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Palladium(II) chalcogenolate complexes as catalysts for C–C cross-coupling and carbonylative Suzuki coupling reactions

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ABSTRACT

The catalytic activity of palladium chalcogenolate complexes, $[\mathrm{PdCl(ECH_2CH_2NMe_2)}]_3$ (E = S or Se), $[\mathrm{PdCl(ECH_2CH_2CH_2NMe_2)}]_2$ and $[\mathrm{PdCl(SeCH_2CH_2NMe_2)(PPh_3)}]$ has been investigated in carbonylative Suzuki coupling reactions. The phosphine complex has also been used as catalyst in C-C cross coupling reactions involving an aryl halide and either triaryl bismuth or aryl boronic acid (Suzuki coupling). The catalytic system was also explored for other carbonylation reactions including carbonylative Sonogashira coupling and amino carbonylation reaction. The phosphine complex showed better activity in carbonylative Suzuki coupling reactions than dimeric and trimeric complexes. All the complexes showed comparable activity in Suzuki coupling reactions.

Palladium catalyzed coupling reactions have emerged as one of the most powerful and versatile tools for the synthesis of pharmaceuticals, agrochemicals and advanced materials, both on laboratory and industrial scales.[1-3] Among the different catalytic reactions, the carbon-carbon cross-coupling of aryl halides with arylboronic acids, i.e. Suzuki reaction, and the same reaction in the presence of carbon monoxide (CO), i.e. carbonylative Suzuki reaction, are efficient strategies for the synthesis of biaryl[1,4,5] and heteroaryl carbonyl compounds,[2,3,6-8] respectively. Non-toxic and stable boronic acids, mild reaction conditions with a wide range of functional group compatibility make these coupling reactions of choice for the syntheses of various functional materials.[3,5,9-13] Since the discovery of Suzuki cross-coupling reaction in 1979[14] and carbonylative Suzuki reaction in 1993,[15] numerous modifications have been made in reaction conditions as well as catalyst systems. For instance, initially utilized palladium catalyst has been modified by using bulky or electron rich phosphine ligands,[16-18] nitrogen containing ligands,[19] *N*-heterocyclic carbenes,[20-22] oximes[23,24] and imines.[19,25] In general, these catalysts are air and moisture sensitive and are often difficult to prepare or recover. There have been consistent efforts to develop stable and efficient Pd catalyst for these reactions. Recently, palladium complexes derived from chalcogen ligands (S, Se, Te) have emerged as highly effective catalysts and are air and moisture stable. Palladium complexes containing S[26,27] and Se[28,29] in palladacycles, carbenes,[30,31] Schiff bases[32,33] and ether type[34,35] ligands have been developed as highly effective phosphine-free palladium catalysts in Suzuki and Heck reactions. In fact, the cyclopalladated selenium complex, $[\mathrm{Pd}(\mu\text{-OAc})(\mathrm{C_6H_4CH_2SeBu}^t)]_2$[28] has outperformed not only the sulfur counterpart,[36] but also the well known Hermann's palladacycle, $[\mathrm{Pd_2}(\mu\text{-OAc})_2(o\text{-tol}_2\mathrm{PC_6H_4CH_2}\text{-}2)_2]$[37] and Milstein pincer complex, $[\mathrm{Pd_2\{C_6H_3(1,3\text{-}CH_2PPr}^i{}_2)_2\}(\mathrm{O_2CCF_3})]$.[38]

The chalcogenolate palladium complexes containing "Pd–ER" (E = S, Se, Te) linkages are little explored as catalysts for C–C coupling reactions[26,30] although their utility in C–S/Se bond formation is well documented.[39,40] We have recently described catalytic activity of selenolate complexes, $[\mathrm{PdCl(4\text{-}Sepy)(PPh_3)_2}]$, $[\mathrm{PdCl(4\text{-}Sepy)(PPh_3)}]_n$[41] and $[\mathrm{PdX(E^{\cap}N)}]_n$ (X = Cl, OAc; $\mathrm{E^{\cap}N} = \mathrm{ECH_2CH_2CH_2NMe_2}$, $\mathrm{ECH_2CH_2NMe_2}$; n = 2, 3) in Suzuki coupling and Sonogashira reactions.[42,43] The latter series of complexes $[\mathrm{PdCl(E^{\cap}N)}]_n$ ($\mathrm{E^{\cap}N} = \mathrm{ECH_2CH_2NMe_2}$, E = S (**1**), Se (**2**); $\mathrm{E^{\cap}N} = \mathrm{ECH_2CH_2CH_2NMe_2}$, E = S (**3**), Se (**4**)) together with $[\mathrm{PdCl(SeCH_2CH_2NMe_2)(PPh_3)}]$ (**5**) have been chosen to assess their suitability as catalysts in C–C coupling and different carbonylative coupling reactions like carbonylative Suzuki coupling, carbonylative Sonogashira coupling and amino carbonylation reaction. Results of this work are reported herein.

The complex $[\mathrm{PdCl(SeCH_2CH_2NMe_2)(PPh_3)}]$ (**5**) has been used as a catalyst in C–C coupling reactions involving an aryl halide and either an aryl boronic acid (Suzuki reaction)[44] or triaryl bismuth.[45] Initial optimization of reaction conditions was

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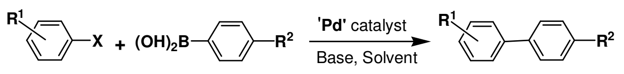
performed. It was noted that dioxane and K_2CO_3 were the preferred combination over other solvents (DMF, toluene) and base (Na_2CO_3). The coupling reactions of aryl halide and aryl boronic acid were carried out using catalyst [PdCl(SeCH$_2$CH$_2$NMe$_2$)(PPh$_3$)] (**5**) in dioxane in the presence of K_2CO_3 as a base and yields of the products were calculated from ^{1}H NMR integration. The catalyst can be applied to a wide range of aryl iodides and bromides. As expected the coupling of aryl iodides was excellent and afforded the desired biaryls in high yields. The yields (≥ 85%, Table 1 entry nos. 5 and 6) obtained within 6 h of reaction is comparable with the yields obtained from the coupling reaction of aryl iodide and aryl boronic acid catalyzed by **1**-**4**, recently reported by us.[42]

Cross-coupling of electron-poor aryl bromides also gave quantitative yields (≥ 92%) of the corresponding biaryls (Table 1, entry nos. 8 and 9). Reactions involving electron-rich aryl bromides led to low conversions (Table 1, entry no. 15). A longer reaction time however gave a slightly higher conversion. For example, the coupling of tolyl bromide and phenyl boronic acid catalyzed by **3** yielded 18% of biaryl in 18 h.

Table 1. Catalytic activity of palladium chalcogenolate complexes in Suzuki coupling reaction[a]



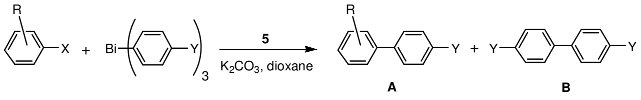
Entry	R^1	R^2	X	'Pd' catalyst	Yield (%)[c]
1.	4-CH_3	H	I	**1**	87[d]
2.	4-CH_3	H	I	**2**	63[d]
3.	4-CH_3	H	I	**3**	96[d]
4.	4-CH_3	H	I	**4**	93[d]
5.	4-CH_3	H	I	**5**	88
6.	H	CH_3	I	**5**	85
7.	2-CHO	H	Br	**3**	95[d]
8.	2-CHO	H	Br	**5**	92
9.	2-CHO	CH_3	Br	**5**	97
10.	2-CHO	NMe_2	Br	**5**	43
11.	4-CN	CH_3	Br	**5**	30
12.	4-NO_2	H	Br	**3**	97[d]
13.	4-NO_2	H	Br	**5**	58
14.	4-NO_2	CH_3	Br	**5**	45
15.	4-CH_3	H	Br	**5**	10[b]

[a] Reaction conditions: Aryl halide (1 mmol), aryl boronic acid (1.3 mmol), Catalyst (0.1 mol% of Pd) K_2CO_3 (2 mmol), dioxane (3 mL), $T = 100$ °C.
[b] The refluxing time was 8 h; the same coupling reaction yielded 18% of biaryls within 21 h when the catalyst **3** was used.
[c] Isolated yield.
[d] Taken from Ref. 42.

The coupling reaction of iodo benzene and tritolyl bismuth in dioxane and K_2CO_3 as a base in the presence of **5** gave moderate yields of cross-coupled product (Table 2, entry nos. 1 and 2) with significant amount of homo coupled product. In the case of aryl bromide with electron withdrawing group conversion to cross-coupled product was observed within 6 h, the longer reaction time significantly increased the yield of cross-coupled product with some amount of homo coupled product (Table 2, entry nos. 3 and 4). This indicates that the catalyst is still active even after 20 h of reaction. The formation of homo coupled biaryls was reported in the coupling reaction of aryl halide and triaryl bismuth, and the amount of product is varied depending on cross-coupling conversion.[46,47] However, a homocoupled product arising from an arylhalide, has not been observed as has also been noted earlier.[47b]

The reaction between iodobenzene and phenyl boronic acid in the presence of [PdCl(SeCH$_2$CH$_2$NMe$_2$)(PPh$_3$)] as a palladium catalyst and K_2CO_3 base under carbon monoxide pressure (200 psi) results in carbonylative coupling[48] (Table 3). In general better yields of benzophenone was noted in toluene as a solvent and K_2CO_3 as a base. Depending on the nature of palladium catalyst yield varied in the range 40-70%, the **5** being the most active. The formation of any dehalogenation product/homocoupled derivative was not observed as these products are expected to be formed above 120 °C / below 50 psi CO pressure, respectively. The general trend of the activity in the present case is **5** > binuclear (**3**, **4**) > trinuclear (**1**, **2**) (Supplementary material). The observed activity may reflect deoligomerization of high nuclearity complexes to generate mononuclear complex as an active catalytic species.[30,49]

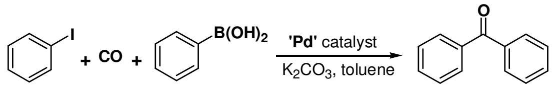
Table 2. Catalytic activity of [PdCl(SeCH$_2$CH$_2$NMe$_2$)(PPh$_3$)] in C–C coupling reaction[a] between an aryl halide and triaryl bismuth



Entry	R	X	Y	Time (h)	Yield of A[b]	Yield of B[c]
1.	H	I	Me	8	28	66
2.	H	I	Me	16	40	44
3.	2-CHO	Br	Me	6	9	22
4.	2-CHO	Br	Me	20	42	28
5.	4-NO_2	Br	Me	6	3	5
6.	4-NO_2	Br	Me	16	6	5
7.	4-Me	Br	H	6	2	48

[a] Reaction conditions: Aryl halide (0.92 mmol), triarylbismuth (0.28 mmol), catalyst (0.002 mmol of Pd), K_2CO_3 (1.2 mmol), dioxane (3 mL), $T = 100$ °C.
[b] % Yield of cross-coupled biaryl.
[c] % Yield of homo-coupled biaryl.

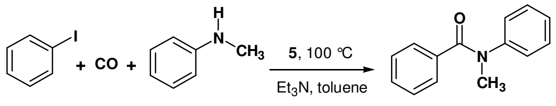
Table 3. Carbonylative Suzuki coupling reaction of iodobenzene with phenyl boronic acid[a]



Entry	**Pd** catalyst	Yield (%)[b]
1.	[PdCl(SCH$_2$CH$_2$NMe$_2$)]$_3$ (**1**)	40
2.	[PdCl(SeCH$_2$CH$_2$NMe$_2$)]$_3$ (**2**)	44
3.	[PdCl(SCH$_2$CH$_2$CH$_2$NMe$_2$)]$_2$ (**3**)	52
4.	[PdCl(SeCH$_2$CH$_2$CH$_2$NMe$_2$)]$_2$ (**4**)	41
5.	[PdCl(SeCH$_2$CH$_2$NMe$_2$)(PPh$_3$)] (**5**)	70

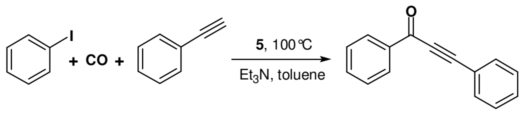
[a] Reaction conditions: Iodobenzene (1 mmol), phenyl boronic acid (1.2 mmol), Pd catalyst (1 mol%), K_2CO_3 (3 mmol), toluene (10 mL), Time = 8 h, $T = 100$ °C, CO pressure = 200 psi.
[b] Isolated yield.

The high activity of **5** has prompted us to examine its activity in other carbonylative reactions. Thus amino carbonylation of secondary amine was studied. The reaction between iodobenzene and *N*-methyl aniline in toluene in the presence of Et_3N and **5** under CO at 200 psi gave 70% yield of N-methylphenyl benzamide (Scheme 1).



Scheme 1. Aminocarbonylation reaction.

Carbonylative Sonogashira coupling reaction was also studied. Thus the reaction between iodo benzene and phenyl acetylene in toluene in the presence of Et_3N and **5** under CO at 250 psi gave a carbonylated product in 50% yield (Scheme 2).



Scheme 2. Carbonylative Sonogashira coupling reaction.

In conclusion, palladium chalcogenolate complexes have been used as a catalyst in C–C cross-coupling, Suzuki coupling reactions and carbonylative Suzuki coupling reactions. Furthermore the catalytic system was also tested for other carbonylation reaction including carbonylative Sonogashira coupling reaction and aminocarbonylation reaction. The catalytic activity is influenced by the nuclearity of complexes and follow a trend mononuclear > binuclear > trinuclear.

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44. Experimental procedure for Suzuki reaction: A two-necked flask was charged with dioxane (3 mL), aryl halide (1.0 mmol), arylboronic acid (1.3 mmol), aqueous K_2CO_3 (2.0 mmol, 1 mL) and catalyst (0.1 mol%). The reactants were heated at 100 °C with stirring for a specified time under a nitrogen atmosphere. After cooling the reaction mixture to room temperature, the contents were diluted with water (5 mL), neutralized with dil. HCl and extracted with hexane (3 × 20 mL). The whole organic extract was washed with water (2 × 15 mL), dried over anhydrous Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was characterized by ^1H NMR and $^{13}C\{^1H\}$ NMR spectra. In case of poor yield of the product or its contamination with other impurities, the product was chromatographed on a silica gel column.
45. Experimental procedure for C–C cross coupling reaction between an arylhalide and organobismuth compound: To a dioxane (3 mL) solution of 2-bromobenzaldehyde (170 mg, 0.92 mmol) and tol_3Bi (133 mg, 0.28 mmol) was added powdered K_2CO_3 (152 mg, 1.10 mmol) and $[PdCl(SeCH_2CH_2NMe_2)(PPh_3)]$ (1.2 mg, 0.002 mmol). The whole was heated at 100 °C in an oil bath with stirring for 20 h. The contents were cooled to room temperature and acidified with dilute HCl (10 mL) and extracted with ethylacetate (3 × 15 mL). The combined extracts were washed with water (2 × 15 mL) and brine (15 mL) and dried over anhydrous Na_2SO_4. This was filtered and dried under vacuum to give a yellow oil (148 mg) which was characterized as 2-tolylbenzaldehyde (42%) and 4,4'-dimethylbiphenyl (28%). Similarly, other C–C coupling reactions were carried out and yields were calculated based on aryl groups of Ar_3Bi consumed in the reactions.
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48. Experimental procedure for carbonylative Suzuki reaction: To a 100 mL autoclave were added iodobenzene (1.0 mmol), phenyl boronic acid (1.2 mmol), $[PdCl(SeCH_2CH_2NMe_2)(PPh_3)]$ (1 mol%), toluene (10 mL) and K_2CO_3 (3.0 mmol). The mixture was first stirred for 10 min. and then flushed with 1 atm CO; then 200 psi of CO was taken, and the reaction mixture was heated at 100 °C for 8 h. After completion of the reaction, the reactor was cooled to room temperature and the remaining CO gas was carefully vented and the reactor was opened. The reactor vessel was thoroughly washed with ethyl acetate (2 x 10 mL) to remove any traces of product and catalyst if present. The catalyst

was filtered and the reaction mixture was evaporated under vacuum. The residue obtained was purified by column chromatography (silica gel, 60–120 mesh; petroleum ether (60:80)/ethyl acetate) to afford the desired carbonylated product. The identity of the products was confirmed by GC–MS, ^{1}H NMR, ^{13}C NMR and IR spectra. The purity of the compounds was determined by GC–MS analysis.

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Supplementary Material

Details of base and solvent optimization studies of the complexes **1-5** in carbonylative Suzuki coupling reactions are provided in supplementary material.

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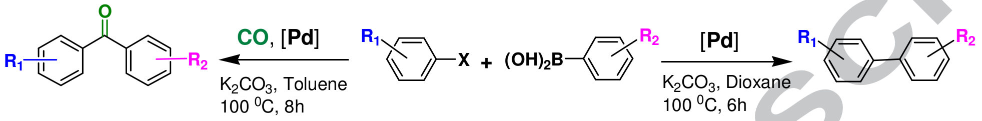
Dilip K. Paluru[a], Sandip Dey[a], Kamal R. Chaudhuri[a], Mayur V. Khedkar[b], Bhalchandra M. Bhanage[b] and Vimal K. Jain[a]



X = Br, I; **[Pd]** = $[PdCl(SeCH_2CH_2NMe_2)(PPh_3)]$