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Activation of C—X (=Cl, Br) bond in aryl halides toward the palladium-catalyzed Heck reaction using 2,6-bis(diphenylphosphino)pyridine

Ali Ataei[a], Shirin Nadri[a], Ezzat Rafiee[a], Sirous Jamali[b], Mohammad Joshaghani[a,*]

[a] *Faculty of Chemistry, Razi University, Kermanshah 67149, Iran*
[b] *Chemistry Department, Sharif University of Technology, P.O. Box 11155-3615, Tehran, Iran*

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ABSTRACT

The 2,6-bis(diphenylphosphino)pyridine/palladium catalytic system successfully catalyzes the Heck coupling reaction of less reactive aryl chlorides as well as aryl bromides with styrene to give the corresponding olefins in reasonable yields. TBAB (tetrabutylammoniumbromide) as an additive was found to be essential for these reactions. The results of Heck reaction exhibited a high selectivity (>99/1) favoring the *trans* product.

1. Introduction

Palladium-catalyzed Heck coupling has been recognized as one of the most important tools for building up of carbon—carbon bond and represents the best technology for the synthesis of the arylated olefins [1–5]. In recent years, numerous papers have been reported concerning improvements to the Heck reaction, but it still is an attractive synthetic goal in academia as well as in industry [6–9].

In Heck reaction, depending on the reaction conditions including the properties of the aryl substrate, olefin, solvent and catalytic system a mixture of arylated products, *trans*/*cis* isomers (β-arylation), and *gem* isomer (α-arylation) can be formed (Scheme 1).

On the other hand, the readily accessible and cheaper aryl chlorides have not been employed much in cross-coupling reactions, due to their reluctance to oxidatively add to Pd(0) center. Therefore, coupling aryl chlorides as industrially important compounds by Heck reaction has become another equally important topic of research [10–12].

On the basis of these considerations, it is important to develop rewarding catalytic system that meets the goals of high selectivity and high activity to aryl chlorides. It is clear that the nature of the ligand plays an important role in determining the catalytic activity. The use of multifunctional ligands gives advantage as high capacity chelating molecules for the stabilization of catalytic active species during the catalytic process, which manipulate the activity of the catalyst. Also, increasing the rigidity of the supporting ligand decreases the number of the possible conformations of the metal–ligand assembly, thus leading to improved selectivity [13,14].

The presence of both soft and hard donor sites (e.g. P and N) in phosphorus–nitrogen containing ligands forms an ideal acceptor–donor pair for catalytic uses [15–17]. Indeed, the π-acceptor ability of the phosphorous atom, together with σ-donor nature of nitrogen, synergistically induce an alteration of the catalyst reactivity, though the stabilization of the low-valent transition metals (by phosphine), and promote the oxidative addition of the aryl halide on metal center (by nitrogen).

The juxtaposition of a pyridine and two phosphine moieties, presenting a P–N–P framework, in a 2,6-relationship of type shown in Fig. 1, has found considerable attention in the field of organometallic chemistry [18–22].

Of special interest in this context is 2,6-bis(diphenylphosphino)pyridine, $(Ph_2P)_2py$ (**1**), first synthesized by Newkome and Hager [23], owing to its rigid backbone. Absence of the methylene groups connecting the phosphorus atoms and the pyridine ring make $(Ph_2P)_2py$ (**1**), more rigid than other phosphine–pyridine ligands presented in Fig. 1. The coordination chemistry of $(Ph_2P)_2py$ has been well studied [24–28], but relatively scarce attention has been given to its application as a supporting ligand in catalysis.

Because of rigidity and multi-donor nature of $(Ph_2P)_2py$, and also based on our experience in C—C coupling reaction [29–35], herein, we thought to use this ligand in Heck reaction of aryl

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[*] Corresponding author. Tel.: +98 831 4274559; fax: +98 831 4274559.
E-mail addresses: mjoshaghani@razi.ac.ir, joshaghanim@yahoo.com (M. Joshaghani).

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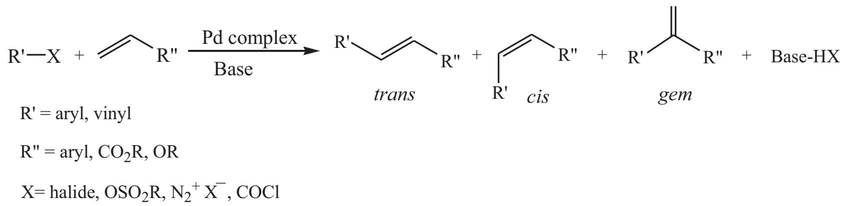


Scheme 1. The Heck coupling reaction, principle and possible isomers.

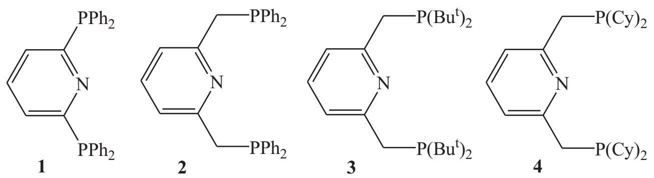


Fig. 1. Selected examples of 2,6-disubstituted phosphine-pyridine ligands.

chlorides as well as aryl bromides. To the best of our knowledge, this is the first report for the use of $(Ph_2P)_2py$ for palladium-catalyzed C—C coupling reactions with high efficiency.

2. Experimental

2.1. General remarks

All reactions were performed under an atmosphere of dry nitrogen. All chemicals purchased from Fluka and/or Merck companies were used without further purification. 2,6-bis(diphenylphosphino)pyridine was commercially available.

^1H (200 MHz), ^{13}C (100 MHz) NMR spectra were recorded on a Bruker Avance Spectrometer. Elemental analysis was performed using CHN Herause rapid model. Thin layer chromatography on precoated silica gel Fluorescent 254 nm (0.2 mm) on aluminum plates were used for monitoring the reactions. The cross coupling products were characterized by their ^1H NMR spectra.

2.2. General procedure for Heck coupling of styrene with aryl halides

A reaction tube was charged with chlorobenzene (4 mmol), styrene (4 mmol), TBAB (3 mmol), and K_2CO_3 (4 mmol) under a dry nitrogen atmosphere. A solution of $(Ph_2P)_2py$ (0.05 mol% in 2 mL of DMAc) and prepared solution of palladium acetate (0.025 mol% in 2 mL) was added through a rubber septum, and the resulting mixture was refluxed at 135 °C for appropriate time. Upon completion of the reaction, the reaction mixture was then cooled to room temperature. After extraction with CH_2Cl_2 (3 × 20 mL), the combined organic layer was dried over $MgSO_4$. The solvent was evaporated and the crude product was characterized by ^1H NMR spectroscopy. Isolated yield of coupling products of chlorobenzene, and 4-bromotoluene were 82% and 86%, respectively.

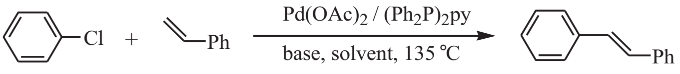
3. Results and discussion

3.1. Initial optimization

By employing a reaction of chlorobenzene and styrene as a probe, the reaction conditions were optimized. We were pleased to find that the reaction proceeded smoothly with only 0.05 mol% $Pd(OAc)_2$/0.1 mol% $(Ph_2P)_2py$ when K_2CO_3 and DMAc were used as the base and solvent respectively. Thus, after 24 h at 135 °C, the product *trans*-stilbene was obtained in 75% yield, corresponding to a turnover number (TON) of 1500 (Table 1, entry 1). It should be noted that in the absence of $(Ph_2P)_2py$ (ligand free condition) no amount of the desired product was formed (not shown in the table). When the palladium loading was reduced to 0.025 mol% the process was less efficient (Table 1, entry 2). Significantly lower yield (20%, 24 h) was obtained in the presence of triethylamine as a representative organic base (Table 1, entry 3). One possible interpretation of this result is blocking of free coordination sites on the palladium center by amine [36,37].

The effect of tetraalkylammonium salts, the so-called Jeffery condition [38], was also considered. Addition of 0.5 mmol

Table 1
Optimizations of reaction condition.[a]



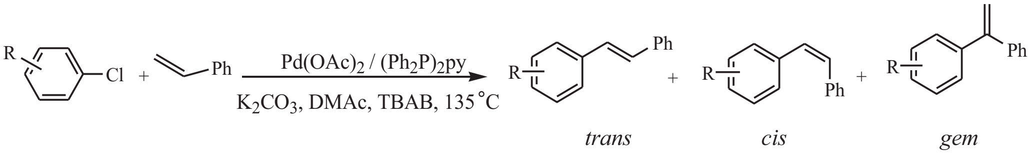
Entry	$Pd(OAc)_2$ (mol%)	Base	Solvent	TBAB (mmol)	Yield (%)[b]	TON
1	0.05	K_2CO_3	DMAc	–	25 (75)	500 (1500)
2	0.025	K_2CO_3	DMAc	–	12 (55)	480 (2200)
3	0.025	NEt_3	DMAc	–	0 (20)	0 (800)
4	0.025	K_2CO_3	DMAc	0.5	30 (55)	1200 (2200)
5	0.025	K_2CO_3	DMAc	1.5	53 (90)	2120 (3600)
6[c]	0.025	K_2CO_3	DMAc	3	57 (90) [82]	2280 (3600)
7	0.025	K_2CO_3	DMAc	5	58 (92)	2280 (3600)
8	0.025	–	DMAc	5	25 (75)	100 (3000)
9	0.025	K_2CO_3	DMSO	3	0 (0)	0 (0)
10	0.025	K_2CO_3	Toluene	3	0 (15)	0 (600)
11	0.025	K_2CO_3	DMF	3	48 (67)	1920 (2680)

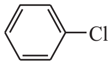
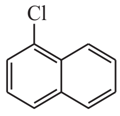
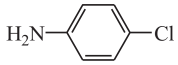
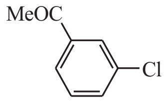
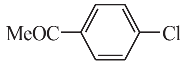
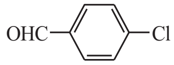
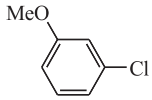
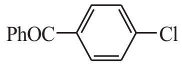
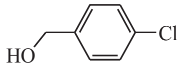
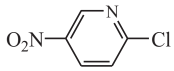
[a] *Reaction conditions*: chlorobenzene (4 mmol), styrene (4 mmol), K_2CO_3 (4 mmol), Pd/$(Ph_2P)_2py$ and DMAc (4 mL), 135 °C, 5 h. Only *trans* isomer was observed in the ^1H NMR spectrum.

[b] NMR yield. The numbers in parentheses are from the reaction time 24 h.

[c] The number in the bracket is isolated yield after 24 h.

Table 2
Heck reaction of aryl chlorides with styrene.[a]



Entry	Aryl chloride	*Trans* (%)[b]	*Cis* (%)[b]	K_{SI} [c]	*gem* (%)[b]	K_{RI} [d]	TON
1[e]		57 (90) [82]	0 (0)	>99/1	0 (0)	>99/1	2280 (3600)
2		75 (93)	0 (0)	>99/1	0 (0)	>99/1	3000 (3720)
3		62 (72)	0 (0)	>99/1	0 (0)	>99/1	3000 (3720)
4		32 (46)	0 (0)	>99/1	0 (0)	>99/1	1280 (1840)
5		28 (81)	0 (0)	>99/1	0 (0)	>99/1	1120 (3240)
6		20 (52)	0 (0)	>99/1	0 (0)	>99/1	800 (2080)
7		52 (82)	0 (0)	>99/1	0 (0)	>99/1	2080 (3280)
8		62 (78)	0 (0)	>99/1	0 (0)	>99/1	2480 (3120)
9		48 (65)	0 (0)	>99/1	0 (0)	>99/1	1920 (2600)
10		67 (80)	0 (0)	>99/1	0 (0)	>99/1	2680 (3200)

[a] *Reaction conditions*: aryl chloride (4 mmol), styrene (4 mmol), K_2CO_3 (4 mmol), $Pd(OAc)_2$ (0.025 mol%), $(Ph_2P)_2py$ (0.05 mol%), TBAB (3 mmol) and DMAc (4 mL), 135 °C, 5 h.

[b] NMR yield. The numbers in parentheses are from the reaction time 24 h.

[c] Stereoisomer ratio K_{SI} = *trans*/*cis*. The *cis* isomer was not observed in the ^{1}H NMR spectrum.

[d] Regioisomer ratio $K_{RI} = \beta/\alpha$, β = sum of *trans* and *cis* isomers, α = geminal isomer (*gem*). The *gem* isomer was not observed in the ^{1}H NMR spectrum.

[e] The number in the bracket is isolated yield after 24 h.

of *tetra*-*n*-butylammonium bromide (TBAB) had just a moderate effect on the reaction rate (Table 1, entry 4), while it increased markedly in the presence of 1.5 or 3 mmol of TBAB (Table 1, entries 5 and 6). Adding more TBAB (5 mmol) could not further improve the yield (Table 1, entry 7). TBAB is proposed to increase the solubility of the inorganic base K_2CO_3 in the organic reaction medium and strengthened the stabilizations of Pd(0) species [39,40].

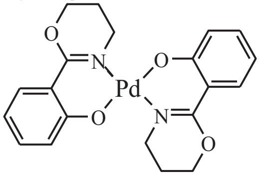
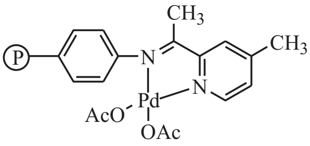
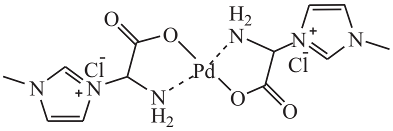
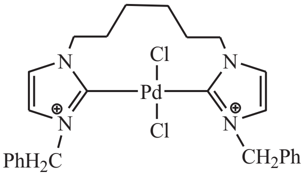
In the next step several solvent systems have been examined. The reaction could not proceed in DMSO and no conversion was observed even when reaction lifted for 24 h (Table 1, entry 9). With toluene as non-polar solvent, the reaction was extremely slow and afforded stilbene in 15% after 24 h (Table 1, entry 10). Using DMF gave acceptable conversion (93%) but a small amount of *cis*-isomer was also formed (Table 1, entry 11). Thus, among the tested solvents, DMAc (dimethylacetamide) in regard to both yield and selectivity was chosen as the preferred solvent for the reactions.

Thus, on comparing results of Table 1, it was seen that the combination of Pd/$(Ph_2P)_2$py and DMAc as the solvent, K_2CO_3 as the base and 3 mmol TBAB as additive gives the best result.

3.2. Heck coupling reactions of various aryl chlorides

To survey the generality of Pd/$(Ph_2P)_2$py catalysis system, we next investigated the reactions of a variety of aryl chlorides with styrene under the identical conditions. Results are listed

Table 3
Comparison of Heck coupling of chlorobenzene and styrene using Pd/$(Ph_2P)_2$py and other catalytic systems.

Entry	Catalytic system	Pd (mol%)	Reaction conditions	Yield (%)[a]	Ref.
1	Pd/$(Ph_2P)_2$py	0.025	K_2CO_3, DMAc, TBAB, 135 °C, 24 h	90 (NMR)	–
2	$[Pd(C_3H_5)Cl]_2$	–	Na_2CO_3, DMAc, TBAB, 120 °C, 24 h	Trace (isolated)	[41]
3[b]	$Pd(OAc)_2$[dpme]	0.1	KOH, DMF, 80 °C, 14 h	4 (isolated)	[42]
4	$PdCl_2$/wool	5.5	NaOAc, aqueous media, PEG-400, 80 °C, 24 h	20 (isolated)	[43]
5	Pd/mesoporous $NiFe_2O_4$	0.4	NEt_3, DMF, 140 °C, 24 h	9 (isolated)	[44]
6		0.1–0.5	NEt_3, DMAc, 120 °C, 8 h	0 (GC, isolated)	[45]
7		0.5	K_2CO_3, DMF, 100 °C, 24 h	12 (GC and GCMS)	[46]
8	$Pd_2(dba)_3/P(t\text{-}Bu)_3$	3	Cs_2CO_3, dioxane, 120 °C, 21 h	83 (isolated)	[47]
9[c]	$Pd_2(dba)_3/P(t\text{-}Bu)_3$	3	Cy_2NM, dioxane, r.t.	78 (isolated)	[48]
10[d]	$[Pd]/[(t\text{-}Bu)_3PH]BF_4$	5	Cy_2NMe, bmimPF_6-dioxane, MWΔ, 180 °C, 60 min	65 (isolated)	[49]
11		0.1 mmol	NEt_3, r.t., 8 h	83 (HPLC)	[50]
12		1	K_2CO_3, DMF/H_2O, CuI, 343 K, 36 h	6 (GCMS)	[51]

[a] *Trans*-isomer exclusively.
[b] Methylacrylate has been used as olefin substrate.
[c] 4-Chloroacetophenone has been used as aryl halide substrate.
[d] Butylacrylate has been used as olefin substrate.

in Table 2. Various types of electronically different aryl chlorides were converted into the corresponding coupling products in good to excellent yields with very low palladium loading (only 0.025 mol%). However, in the cases of 3-chloroacetophenone and 4-chlorobenzaldehyde the yield of corresponding coupled products decreased dramatically to 46% and 52% yield respectively due to dehalogenation reaction (Table 2, entries 4 and 6).

The Heck reaction of sterically hindered 1-chloronaphthalene afforded a good yield of the coupled product (Table 2, entry 2). Also, as reported in Table 2, the heteroaryl chloride, including the 2-chloro-5-nitro-pyridine, gave high yield (80%) (Table 2, entry 10).

As shown in Table 2, the corresponding *trans*-stilbenes were obtained with complete regioselectivity at the β-position ($K_{RI} > 99/1$). In addition, no *cis* olefin products were observed ($K_{SI} > 99/1$).

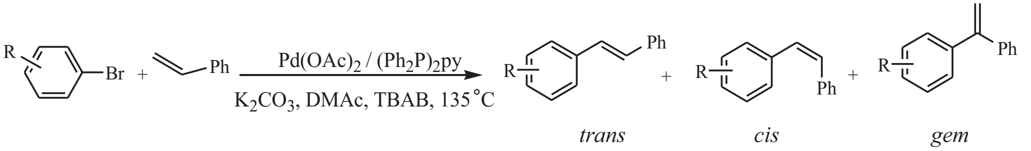
We used the reaction of chlorobenzene with styrene to compare the efficacy of the Pd/$(Ph_2P)_2$py with other reported catalysis systems. Several goals have to be achieved for the utilization of aryl chlorides in Heck reaction, such as the use of stable and inexpensive starting materials and ligands, achievement of high turnover numbers (TONs), use of water and ionic liquids as reaction medium, and easy separation of catalyst. However, the high palladium loadings and long reaction times in some reported systems are not beneficial to the industrial and synthetic applications. In addition, some catalyst systems create practical problems because of high ligand sensitivity toward air and moisture and multistep synthesis procedures. Table 3 illustrates that the Pd/$(Ph_2P)_2$py gives higher yield in very lower catalyst loading in comparison with other systems.

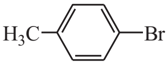
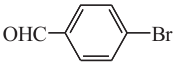
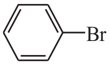
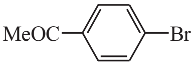
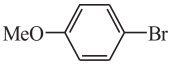
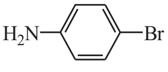
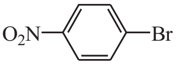
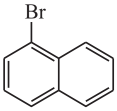
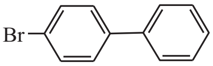
3.3. Heck coupling reactions of various aryl bromides

We also examined the Heck arylation of styrene with various bromides with different electronic and steric effects using Pd/$(Ph_2P)_2$py catalysis system (Table 4). Full conversion of aryl bromides was obtained in all cases when 0.025 mol% Pd was applied. As shown in Table 3, even for the aryl bromides bearing a electron-donor functional groups, such as OMe the reaction was finished within 5 h at a 0.025 mol% Pd loading (Table 4, entry 5). Arylation of styrene with 4-bromobiphenyl was completed within 2 h bringing about a turnover number of 4000 (Table 4, entry 8). It is noteworthy that a high selectivity (>99/1) favoring the *trans* products (K_{RI} and $K_{SI} \gg 99/1$) was also observed in the Heck reaction of aryl bromides with $(Ph_2P)_2$py as same as aryl chlorides.

As aforementioned, rigidity of ligand could improve selectivity of reaction. In agreement with this theory, our experiments showed

Table 4
Heck reaction of aryl bromides with styrene.[a]



Entry	Aryl bromide	*Trans* (%)[b]	*Cis* (%)[b]	K_{SI} [c]	*gem* (%)[b]	K_{RI} [d]	TON
1[e]		72 (99, 6 h) [86]	0 (0)	>99/1	0 (0)	>99/1	2880 (4000)
2		78 (99, 3 h)	0 (0)	>99/1	0 (0)	>99/1	3120 (4000)
3		86 (99, 3 h)	0 (0)	>99/1	0 (0)	>99/1	3440 (4000)
4		50 (98, 10 h)	0 (0)	>99/1	0 (0)	>99/1	2000 (4000)
5		66 (99, 5 h)	0 (0)	>99/1	0 (0)	>99/1	2640 (4000)
6		42 (98, 10 h)	0 (0)	>99/1	0 (0)	>99/1	1680 (4000)
7		51 (99, 10 h)	0 (0)	>99/1	0 (0)	>99/1	2040 (4000)
8		92 (99, 2 h)	0 (0)	>99/1	0 (0)	>99/1	3680 (4000)
9		68 (98, 5 h)	0 (0)	>99/1	0 (0)	>99/1	2720 (4000)

[a] *Reaction conditions*: aryl bromide (4 mmol), styrene (4 mmol), K_2CO_3 (4 mmol), $Pd(OAc)_2$ (0.025 mol%), $(Ph_2P)_2py$(0.05 mol%), TBAB (3 mmol) and DMAc (4 mL), 135 °C, 1 h.

[b] NMR yield.

[c] Stereoisomer ratio K_{SI} = *trans*/*cis*. The *cis* isomer was not observed in the ^{1}H NMR spectrum.

[d] Regioisomer ratio $K_{RI} = \beta/\alpha$, β = sum of *trans* and *cis* isomers, α = geminal isomer (*gem*). The *gem* isomer was not observed in the ^{1}H NMR spectrum.

[e] The number in the bracket is isolated yield after 6 h.

that adding $(Ph_2P)_2py$ dramatically increased the selectivity of the Heck reactions toward the *trans*-isomers.

Rate acceleration were observed in the reactions of aryl bromides with styrene compared to aryl chlorides, primarily because the bond strength of C—Cl is stronger than C—Br, thus the rate of oxidative addition C—Cl to Pd(0) species became slow.

4. Conclusion

In summary, the results presented in this paper show that Pd/$(Ph_2P)_2py$ is capable of catalyzing arylation of styrene by aryl chlorides and bromides containing either electron-rich or electron-withdrawing functional groups with high TONs, and uniformly showed high selectivity for the *trans*-configured product. In fact, the subtle combination of rigidity and strong donor properties of ligand $(Ph_2P)_2py$ results in a highly active and selective catalyst for the Heck reaction at very low Pd loading. Further work to study of the binding $(Ph_2P)_2py$ to $Pd(OAc)_2$ is in hand and the outcome of these studies will be reported in due course.

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