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Amine-based ionic liquids ($R_3N^+PPh_2$) as a reusable reaction medium and Pd(II) ligand in Heck reactions of aryl halides with styrene and *n*-butyl acrylate

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ABSTRACT

Amine-based ionic liquids ($R_3N^+PPh_2$) are reported as an effective reusable medium, suitable Pd(II) ligand and also reducing agent for the Heck coupling reaction of aryl iodides and bromides with styrene and *n*-butyl acrylate. The ionic liquid, still containing its corresponding Pd(0) complex, was easily recovered and reused in several runs without losing its efficiency.

1. Introduction

Transition-metal catalyzed carbon–carbon bond forming reactions have become one of the most important tools in synthetic chemistry [1]. Among different transition metals [2], Pd based catalysts have been used extensively in these reactions during the last decades. In this regard, the chemists have been interested in improving and development of different aspects of Pd catalysts involving types of new ligands [3] or the use of heterogeneous Pd catalyst which Pd is fixed to a solid support such as activated carbon [4], complex matrices immobilized on silica and functionalized silica [5], polymers [6] alongside techniques such as microwave irradiation [7] or reaction media including ionic liquids [8], PEG [9] and also reaction under solvent-free conditions [5a,10] have also been considered as potential tools for this purpose. The importance of green reactions in organic synthesis has encouraged scientists to use from ionic liquids (ILs) as environmentally friendly reaction media, catalysts, reagents, and also in separation processes [11]. ILs usually possesses unique properties, such as nonvolatility, nonflammability, and remarkable chemical and thermal stability [12]. A variety of reactions [7b,8b–c], including transition-metal catalyzed carbon–carbon coupling reactions such as Mizoroki–Heck reaction have been reported in ILs [7,8c,13]. The Heck reaction as a versatile method provides an efficient route for the synthesis of substituted olefins through cross-coupling of aryl halides and olefins in organic synthesis. In 1984, in a pioneering work Jeffery reported the palladium-catalyzed vinylation of organic iodides under solid liquids phase transfer conditions using tetrabutylammonium chloride. This salt as an additive has been proved to have a crucial effect on the rate and yield of reaction through much probably the role of tetrabutylammonium chloride as the stabilizing agent for the colloidal palladium, which hinders its agglomeration to metallic palladium [13b]. However, the first application of ILs in palladium-catalyzed Heck reaction was reported by Kauffmann et al. in the presence of $Pd(OAc)_2$ or $PdCl_2$ as appropriate catalyst precursors for this reaction without additive ligands in $[C_{16}H_{33}PBu_3]Br$. The use of $[bmim]PF_6$ and other ILs, such as hexylpyridinium salts (C_6PyCl, C_6PyPF_6, C_6PyBF_4), has also been described for phosphine-free Heck reaction. The addition of Ph_3P as ligand promoted the reactions which required longer reaction times and higher temperature to go to completion [14]. Generally, due to the importance of phosphines and phosphinites as phosphorous-based ligands in organometallic chemistry, their metal complexes have been used in many catalytic reactions [5a,6,15]. Common ILs have a tendency to leach dissolved catalysts into the co-solvent used to extract the product(s). To avoid metal catalysts leaching out of the IL system, significant efforts have been made to enhance the solubility of the catalysts in ILs and one of approaches is to insert functional groups

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able to coordinate with metal centers into the ILs [13b]. In this regard, the task-specific ionic liquids are developing in the Heck reaction. An imidazolium-based phosphinite, a room temperature task-specific IL, was reported by Iranpoor et al. as Pd ligand and solvent for the efficient carbon–carbon coupling reactions of aryl halides [8c,16].

2. Experimental

Transmission electron microscopy (TEM) analyses were performed on a Philips model CM 10 instrument. Scanning electron micrograph was obtained by SEM, XL-30 FEG SEM, Philips, at 20 kV. IR spectra were run on a Shimadzu FTIR-8300 spectrometer. The ^{1}H and ^{13}C NMR spectra were recorded on a Brucker Avance DPX-250 MHz spectrometer using tetramethylsilane as internal standard.

2.1. Typical procedure for the preparation of *N-(diphenylphosphino)triethylammonium chloride (**IL$_1$**):*

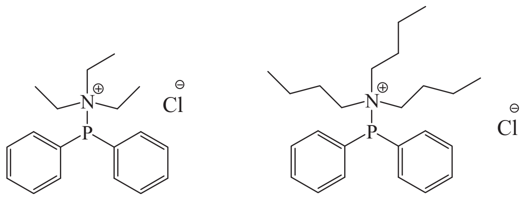
Under argon atmosphere, to a two-necked flask containing triethylamine (15.0 mmol, 2.0 mL), chlorodiphenylphosphine (10.0 mmol, 1.8 mL) was added drop wise with vigorous stirring. Stirring was continued for 5 min at 50 °C to obtain the product as a white solid. The produced *N*-(diphenylphosphino)triethylammonium chloride (**IL$_1$**) was washed with diethyl ether (3 × 5 mL) to remove the remaining substrates, then filtered, dried under vacuum, and was stored in a capped bottle without any change for months (3.0 g, 93%). m.p.: 120–122 °C; IR (KBr): 3055 (=CH), 2974 (C–H), 2937 (C–H), 1590 (C=C), 1475 (C=C), 1178 (C–N), 1128 (P-Ph), 961 (P-N) cm^{-1}; δ_H (250 MHz, CDCl$_3$) 7.30–6.87 (10H, m, Ph), 2.64 (6H, m, C$\underline{H_2}$), 0.88 (9H, t, *J* 7.3 Hz, $\underline{Me}$); δ_C (62.9 MHz, CDCl$_3$) 131.78, 131.74, 131.15, 130.99, 130.64, 130.46, 128.38, 128.16, 45.88, 8.63.

2.2. Typical procedure for the preparation of *N-(diphenylphosphino)tributylammonium chloride (**IL$_2$**):*

Under argon atmosphere, to a flask containing tributylamine (15.0 mmol, 3.6 mL) was added ClPPh$_2$ (10.0 mmol, 1.8 mL) drop wise at room temperature. The mixture was stirred with a mechanical stirrer. After 30 min, the reaction mixture was cooled in ice bath for 1 h and then washed with diethyl ether (3 × 5 mL), filtered and dried under vacuum. **IL$_2$** was obtained as a room temperature ionic liquid in 85% yield (3.4 g), which was stored in a capped bottle without any change for months. IR (KBr): 3054 (=CH), 2973 (C–H), 2936 (C–H), 1590 (C=C), 1474 (C=C), 1178 (C–N), 1127 (P-Ph), 959 (P-N) cm^{-1}; δ_H (250 MHz, CDCl$_3$) 7.62–7.20 (10H, m, Ph), 2.96 (6H, m, NC$\underline{H_2}$), 1.28 (12H, m, C$\underline{H_2}$C$\underline{H_2}$), 0.86 (9H, m, $\underline{Me}$); δ_C (62.9 MHz, CDCl$_3$) 131.86, 131.82, 131.26, 128.41, 128.20, 52.23, 25.13, 20.11, 13.58.

2.3. *General procedure for the Mizoroki–Heck reaction in the presence of PdCl$_2$/**IL$_1$***

PdCl$_2$ (3 mol%, 0.0053 g), **IL$_1$** (0.5 mmol, 0.16 g), NaOH (2.0 mmol, 0.08 g), olefin (2.0 mmol), and aryl halide (1.0 mmol) were placed in a 25 mL flask equipped with a magnetic stirring bar and heated at 120 °C. The completion of the reaction was monitored by TLC analysis. After completion of the reaction, the reaction mixture was cooled to room temperature and the products were extracted with diethyl ether (3 × 3 mL). The solvent was then evaporated to leave the crude products, which were purified by column chromatography over silica gel using *n*-hexane/ethyl acetate (4:1) as eluent to afford the highly pure products. The products were identified by their spectral data and comparison with authentic samples.



(**IL$_1$**) (**IL$_2$**)

Scheme 1. Structures of *N*-(diphenylphosphino)trietylammonium chloride (**IL$_1$**) and *N*-(diphenylphosphino)tributylammonium chloride (**IL$_2$**).

2.4. *General procedure for the Mizoroki–Heck reaction in the presence of PdCl$_2$/**IL$_2$***

Aryl halides (1.0 mmol) were added to a flask containing **IL$_2$** (0.5 mmol, 0.20 g), PdCl$_2$ (3 mol%, 0.0053 g), NaOH (2.0 mmol, 0.08 g) and olefin (2.0 mmol) and the mixture was stirred at 120 °C. After completion of the reaction, which was detected by TLC analyses, the mixture was cooled to room temperature and the coupled products were extracted with diethyl ether (3 × 3 mL). The solvent was then evaporated to leave the crude products, which were purified by column chromatography over silica gel using *n*-hexane/ethyl acetate (4:1) as the eluent to give pure products. The products were identified by their spectral data and comparison with authentic samples.

3. Results and discussion

3.1. *Synthesis and characterization*

In this work we report a very easy method for the synthesis of new amine-based ionic liquids of *N*-(diphenylphosphino)trietylammonium chloride (**IL$_1$**) and *N*-(diphenylphosphino)tributylammonium chloride (**IL$_2$**) (Scheme 1). Triethylamine and tributylamine were functionalized by treatment with ClPPh$_2$ under argon atmosphere at 50 °C and room temperature, respectively. Then, their successful application has been studied both as reaction media and as a potential complexing agent through the phosphine group, in conjunction with Pd(II) salts for Heck reaction of aryl iodides and bromides with styrene and *n*-butyl acrylate.

In order to have more information about the reducing property of these ILs, We probed the ultraviolet (UV) spectrum of Pd(II) in aqueous solution of **IL$_1$**. The white color of the IL changed almost immediately to gray upon the addition of PdCl$_2$ at room temperature; this demonstrated the efficiency of the generation of zero-valent palladium by this IL. The UV spectrum of palladium chloride solution is shown in curve **A** of Fig. 1. The peak at around 450 nm shows the presence of Pd(II). Curve **B** belongs to the aqueous solution of **IL$_1$** including PdCl$_2$. The disappearance of the peak around 450 nm confirms that Pd(II) has been reduced to the Pd(0) species in the presence of **IL$_1$**.

The simplest of the spectrophotometric techniques that have been used for the study of the composition of complexes is the mole ratio method which the amount of one reactant, usually the moles of metal, is held constant, which the amount of the other reactant is varied. In order to find out the nature of the catalyst, initially the solution of PdCl$_2$ (0.002 M), base (0.13 M), and **IL$_1$** (0.03 M) in DMSO were prepared, separately. Then, the solution of Pd(II) and base were mixed and the absorbance of the solution was measured by UV spectra. It was in the wavelength of 281 nm in related to

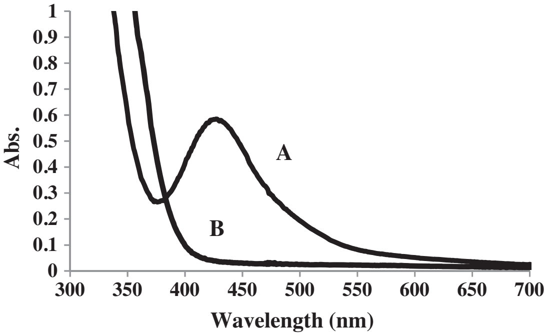


Fig. 1. UV spectra of the palladium catalyst. (A) $PdCl_2$, (B) $\mathbf{IL}_1 + PdCl_2$.

the complexation of Pd(II) and DMSO (Fig. 2). In the next step, a series of solutions were prepared which contain equal formal concentrations of metal ion (Pd^{2+}) but different formal concentrations of the ligand. The ratio of these concentrations varied from 0.5 to 5. The absorbance of each solution was then measured. A plot of the absorbance against the ratio of the number of moles of ligand to the number of moles of the metal ion showed the stochiometric ratio of ligand to metal (Fig. 2). As shown in this figure, the absorbance reduced by adding ligand to the solution of metal ion gradually due to the replacement of ligand with DMSO in complex and finally it remained constant. The use of the mole ratio method [17] for the complex formation between this IL and $PdCl_2$ is in good agreement with the ML_2 structure.

The information about the surface of catalyst was obtained by the scanning electron microscopy (SEM) image as presented in Fig. 3.

Transmission electron microscopy (TEM) image of the catalyst shows that Pd nanoparticles are formed and dispersed on the $\mathbf{IL_1}$ with a size ~20 nm (Fig. 4).

3.2. Catalytic activity of the catalyst in C–C coupling reactions

We initially studied the efficiency of *N*-(diphenylphosphino) triethylammonium chloride both as solvent and as ligand for the coupling of bromobenzene with styrene as a model reaction in the presence of catalytic amounts of $PdCl_2$ and different bases at 120 °C (Table 1). Among applied organic and inorganic bases, sodium hydroxide (NaOH) showed the most suitable one for this reaction (Table 1, entry 5). In the absence of base, the starting material remained unchanged under the same reaction condition after 24 h (Table 1, entry 1).

The use of this ionic liquid also allows to be avoided the use of organic solvents such as DMF or DMSO, and toluene widely employed in Heck reactions. Under our optimized reaction conditions (1.0 mmol of aryl halide, 2.0 mmol of NaOH, 2.0 mmol of styrene, 0.03 mmol (3 mol%) $PdCl_2$, 0.5 mmol of ILs), the desired products were obtained in good to excellent yields for a wide array of aryl iodides and bromides both in $\mathbf{IL_1}$ and in $\mathbf{IL_2}$ at 120 °C. The results are shown in Table 2. The cross-coupling reaction proceeded smoothly for iodobenzene and *p*-iodotoluene as benzene bearing electron-donating group at the para position with styrene and *n*-butyl acrylate (Table 2, entries 1, 3, 9, 11). The *ortho*-methyl

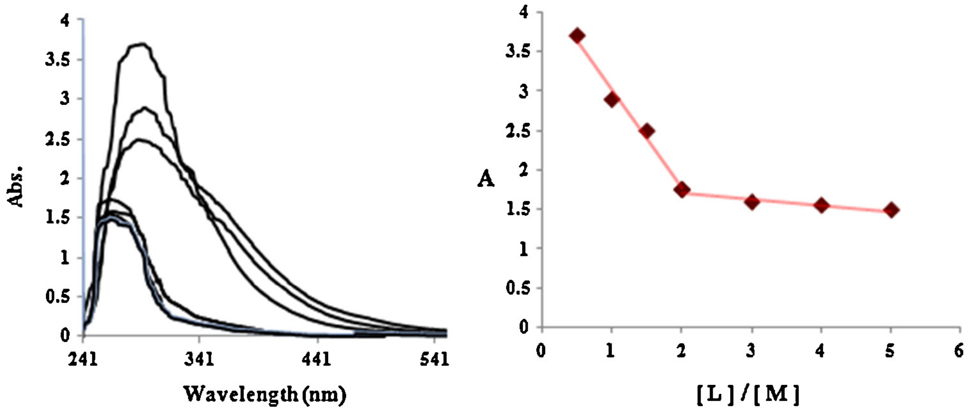
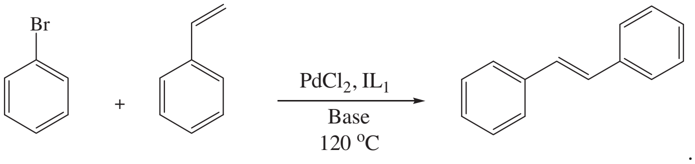


Fig. 2. Mole ratio plot for complex formation between $PdCl_2$ and $\mathbf{IL}_1$.

Table 1
Effect of different bases on Heck reaction of bromobenzene and styrene.[a]



Entry	Base	Time (h)	Yield of trans stylbene (%)[b]
1	–	24	–
2	Na_2CO_3	24	–
3	$CaCO_3$	24	–
4	DBU	25 min	90
5	NaOH	8 min	93
6	Et_3N	24	60
7	Bu_3N	24	50

[a] Reaction conditions: 0.03 mmol of $PdCl_2$, 0.5 mmol of $\mathbf{IL_1}$, 1.0 mmol of bromobenzene, 2.0 mmol of styrene and 2.0 mmol of base at 120 °C.
[b] Isolated yield.

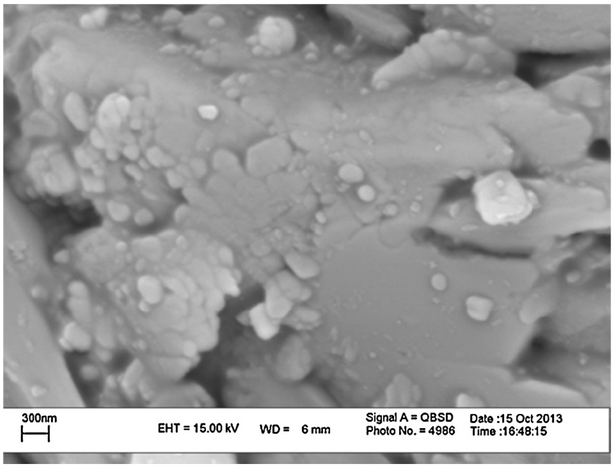


Fig. 3. Scanning electron micrograph (SEM) image of Pd-supported **IL**$_1$.

iodobenzene which has some steric effect also produced the desired coupled products in good to excellent yields (Table 2, entries 2, 10). The catalytic system was equally efficient for aryl bromides: complete conversion had been achieved in the reactions of bromobenzene and 4-bromotoluene with styrene and *n*-butylacrylate in excellent yields (Table 2, entries 5, 6, 13, 14). Also, the Heck reaction of sterically hindered 1-bromonaphthalene afforded moderate and good yields of the corresponding coupled products with styrene and *n*-butylacrylate, respectively (Table 2, entries 8, 16). In general, the reaction of the substrates bearing –NO_2 group proceeded slowly in the same reaction conditions (Table 2, entries 4, 7, 12, 15). Probably the elongation of the reaction time for these aryl halides refers to its less solubility in ILs. Unfortunately, this catalytic system is not efficient for chloro arenes.

The reusability of catalyst is the major area of interest due to environmental concerns, costs of the catalyst and its toxicity. Regarding the property of these ILs as complexing agent with the Pd(II) species, the recovery and reusability of **IL$_1$** and its Pd(0) complex was investigated in the Heck reaction of bromobenzene with styrene for four cycles without any pre-treatment. The results are tabulated in Table 3.

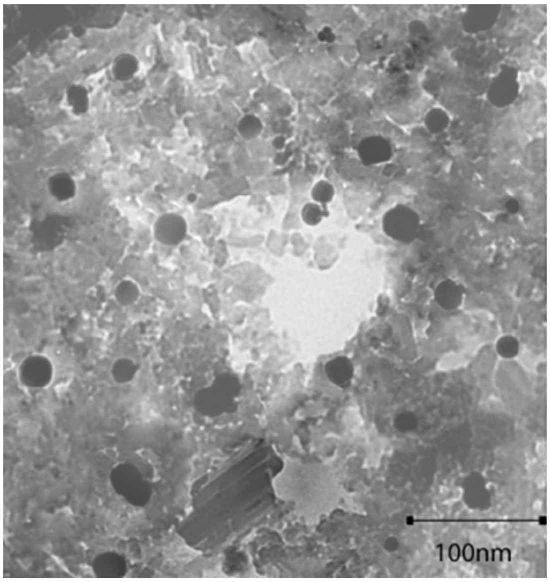
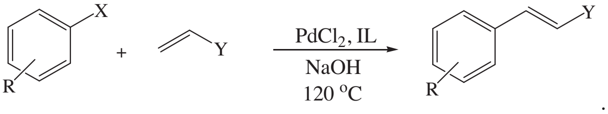


Fig. 4. Transmission electron microscopy (TEM) of Pd-supported **IL**$_1$.

Table 2

Heck reaction of *n*-butyl acrylate and styrene with aryl halides.[a]



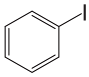
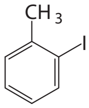
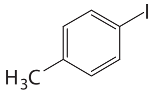
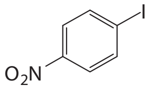
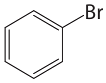
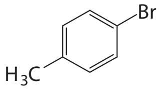
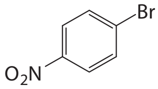
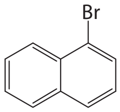
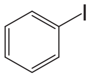
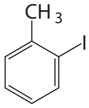
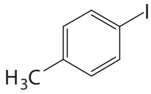
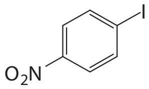
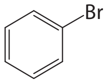
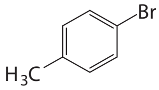
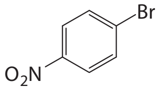
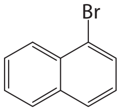
Entry	ArX	Y	Yield (%)[b]	Time (h)
1		-Ph	90 (90)	5 min (35 min)[c]
2		-Ph	70 (73)	1 (2)[c]
3		-Ph	85 (81)	1.5 (2.5)[c]
4		-Ph	60 (56)	24 (24)[c]
5		-Ph	95 (90)	8 min (45 min)[c]
6		-Ph	90 (81)	45 min (1)[c]
7		-Ph	60 (61)	15 (24)[c]
8		-Ph	60 (53)	24 (24)[c]
9		-CO_2Bu-*n*	95 (91)	3 (4)[c]
10		-CO_2Bu-*n*	95 (95)	4 (6)[c]
11		-CO_2Bu-*n*	90 (89)	3 (4)[c]
12		-CO_2Bu-*n*	65 (61)	24 (24)[c]
13		-CO_2Bu-*n*	80 (82)	6 (8)[c]
14		-CO_2Bu-*n*	85 (80)	2 (3)[c]
15		-CO_2Bu-*n*	60 (60)	24 (24)[c]

Table 2 (*Continued*)

Entry	ArX	Y	Yield (%)[b]	Time (h)
16		$-CO_2Bu$-*n*	80 (87)	20 (24)[c]

[a] Reaction conditions: 0.03 mmol of $PdCl_2$, 0.5 mmol of IL, 1.0 mmol of aryl halide, 2.0 mmol of styrene or *n*-butyl acrylate and 2.0 mmol of NaOH at 120 °C.
[b] Isolated yield.
[c] Data in the brackets show isolated yields and reaction time in $\mathbf{IL_2}$.

Table 3
Coupling of bromobenzene with styrene in the presence of recycled ionic liquid ($\mathbf{IL_1}$) and its Pd complex.[a]

Cycle	Time (min)	Yield (%)[b]	Pd-leaching %[c]
1	8	95	1.22
2	40	90	1.12
3	95	80	1.31
4	135	75	1.43

[a] Reaction conditions: 1.0 mmol of bromobenzene, 0.03 mmol of $PdCl_2$, 0.5 mmol of $\mathbf{IL_1}$, 2.0 mmol of styrene or *n*-butyl acrylate and 2.0 mmol of NaOH at 120 °C.
[b] Isolated yield.
[c] The analysis was performed by ICP.

To probe the palladium-leaching in the system, the filtrate of the reaction between bromobenzene with styrene after completion of the reaction was analyzed by ICP in four repeating cycles. Low palladium contamination was observed during this experiment. It showed that the catalyst was highly reusable. The Pd catalyst exhibited only a slight loss in its activity and required a bit longer time to achieve full conversion during four cycles shown in Table 3.

4. Conclusion

In conclusion, we have developed a very easy and cheap method for phosphorylation of trialkylamines and their application in the Heck cross-coupling reaction of aryl halides. The obtained trialkylammonium phosphines can be complexed to $PdCl_2$ in order to reduce Pd(II) to Pd(0) and generate *in situ* Pd(0) nanoparticles. The insolubility of Pd(0) supported trialkylammonium phosphine provides a very simple workup procedure and the catalyst can be simply recovered by filtration. The stability, and also recyclability of the catalyst can be regarded as other advantages of this system.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molcata.2014.01.005.

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