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Journal of Molecular Catalysis A: Chemical 206 (2003) 77–82

JOURNAL OF MOLECULAR CATALYSIS A: CHEMICAL

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Novel palladium imidazole catalysts for Suzuki cross-coupling reactions

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Received 28 March 2003; received in revised form 28 March 2003; accepted 10 June 2003

Abstract

Novel palladium imidazole catalytic systems for the Suzuki cross-coupling reaction have been developed from commercially available and inexpensive imidazoles and palladium sources, and exhibit high activity and no *homo*-coupling.

Keywords: Palladium; Cross-coupling reaction; N-donor ligands; Imidazoles; Suzuki; Aqueous catalysis

1. Introduction

In recent years there has been a renaissance in palladium catalysed coupling reactions. This, in part, has been due to the development of highly active and efficient catalytic systems that have enabled activation of aryl chlorides [1], room temperature catalysis [2] and previously inhibited coupling reactions [3]. Significant innovations have been made in the discoveries of novel phosphine systems and in the modification of traditional phosphines. The replacement of the commonly used triarylphosphine ligands with sterically-hindered and electron-rich trialkylphosphines [4], phosphites [5], and phospha-palladacycles [6] have all given rise to highly active systems in palladium catalysed coupling reactions.

There are, however, a number of problems that are frequently encountered in the use of phosphine ligands in catalysis. For example, the degradation of P–C bonds is notorious and can result in deactivation of the catalyst as well as the scrambling of the coupling partners with the phosphine substituents [7]. This, in addition to their sensitivity to moisture and aerial oxidation, their high toxicity, often laborious synthesis and loss during product extraction, has driven research into alternatives to phosphines. One area where little has been reported to date is the application of *N*-coordinated ligands in palladium catalysed coupling reactions. Of those reported most involve cyclometallated palladium complexes incorporating either the imine [8,9], amine [10], oxime [11], or oxazoline [12] moieties. The remainder include either chelating bi- or terdentate *N*-coordinating ligands such as diazabutanes [13,14], bis(oxazolinyl)pyrrole [15] or dipyridyl [16] ligands, respectively.

A series of methyl palladium(II) complexes have been reported, incorporating *N*-coordinated bis- and tris-imidazole chelates that were found to be catalytically active in the Heck reaction of 4-bromoacetophenone with *N*-butyl acrylate [17]. More recently, an example of a mixed imidazolylidene-imidazole

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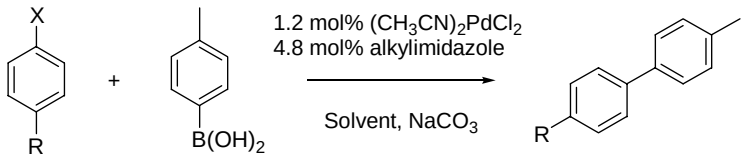
doi:10.1016/S1381-1169(03)00447-3



Scheme 1. The Suzuki coupling reaction of a halogenoarene with tolylboronic acid.

palladium complex and its application in Sonogashira coupling reaction has been reported [18].

Although the Suzuki cross-coupling reaction (Scheme 1) is one of the most powerful methodologies for the generation of new carbon–carbon bonds, particularly in the synthesis of biaryls, it suffers from a number of disadvantages that limit its application [19]. The traditional triarylphosphine catalysts employed in the Suzuki reaction often suffer from low activities, poor catalyst solubility and catalyst decomposition. Further to this, the ubiquitous presence of *homo*-coupled by-products that originate from either the halogenoarene or the arylboronic acid coupling with itself, necessitates additional purification steps. We chose to investigate the use of simple, commercially available imidazoles as ligands for palladium catalysts for Suzuki cross-coupling reactions.

2. Results and discussion

The catalysts were prepared in situ from $(CH_3CN)_2PdCl_2$ (1.2 mol.%) and the appropriate imidazole (4.8 mol.%) in the solvent used. After heating to give a solution, an aqueous sodium carbonate solution, the halogenoarene and tolylboronic acid were added and the reaction heated to 110 °C for 20 min. The products were isolated by the addition of water and subsequent extraction with hexane.

The reaction of bromobenzene with tolylboronic acid was investigated in the first instance. This reaction has the advantage that the product of the competitive *homo*-coupling reaction (4,4′-dimethylbiphenyl) can be readily identified in the product mixture. Initial investigation of the reaction conditions (Table 1) using $(CH_3CN)_2PdCl_2$ and 1-methylimidazole as the ligand and dioxane as the solvent showed that the reaction could be conducted at a wide range of temperatures. At 80 °C it afforded a modest yield of 45%, which increased to 98.3% after 3 h. A similar yield (95.7%, TON h^{-1} = 239) could be achieved within 20 min at 110 °C. This compares very favourably to other *N*-donor ligands that have been used. For instance, under similar conditions, but with a higher concentration of catalyst, a series of diazabutadienes gave yields of between 60 and 99% (TON h^{-1} between 60 and 99) for the closely related reaction of 4-bromotoluene and phenylboronic acid [13].

The $(CH_3CN)_2PdCl_2$/4mim (where mim = 1-methylimidazole) catalysed Suzuki reaction could also be performed at room temperature, affording a 70.2% yield in 24 h. Although slower than the reaction at higher temperatures, this can be useful for thermally sensitive substrates. For the subsequent investigations in this project, we chose to perform the reactions at 110 °C for 20 min.

Having established the reaction conditions, we conducted an initial study of the effect of changing the imidazole ligands on the reaction in dioxane (Table 2). For comparison, the use of 'ligand-free' palladium sources, the traditional phosphine based catalyst $Pd(PPh_3)_4$, and the independently prepared chlorotris(1-methylimidazole)palladium chloride $\{[(mim)_3PdCl]Cl\}$, have been included.

Imidazoles with an N–H bond did not afford any coupled products and unreacted bromobenzene was detected at the end of the reaction period (Table 2, entries 3 and 5). All other imidazoles tested showed

Table 1
Investigation of Suzuki reaction conditions in dioxane

Entry	Time	Temperature (°C)	Yield (%)[a]	TON h^{-1} [b]
1	20 min	110	95.7 (94.2)	239 (236)
2	20 min	80	45.0 (47.2)	113 (118)
3	3 h	80	98.3 (96.7)	27 (27)
4	24 h	25	70.2 (73.9)	2 (3)

Results of repeat reactions in parentheses.
[a] Isolated yield of 4-methylbiphenyl, based on bromobenzene.
[b] TON h^{-1}: number of moles of desired product per mole of catalyst used per hour.

Table 2
Effect of changing imidazoles in dioxane

Entry	Catalyst/ligand	Yield (%)[a]	TON h^{-1} [b]	*Homo* (%)[c]
1	$(CH_3CN)_2PdCl_2$/imidazole	0 (0)	–	0 (0)
2	$(CH_3CN)_2PdCl_2$/1-methylimidazole	95.7 (93.7)	239 (234)	0 (0)
3	$(CH_3CN)_2PdCl_2$/2-methylimidazole	0 (0)	–	0 (0)
4	$(CH_3CN)_2PdCl_2$/1,2-dimethylimidazole	95.5 (96.7)	239 (242)	0 (0)
5	$(CH_3CN)_2PdCl_2$/1,2,4,5-tetramethylimidazole	95.9 (94.4)	240 (236)	0 (0)
6	$(CH_3CN)_2PdCl_2$/1-phenylimidazole	63.5 (59.0)	159 (128)	0 (0)
7	$(CH_3CN)_2PdCl_2$/none	43.2 (45.1)	108 (113)	1.9 (2.8)
8	$PdCl_2$/none	54.9	137	3.5
9	$PdCl_2$/1-methylimidazole	94.0 (97.3)	235 (243)	0 (0)
10	$[(mim)_3PdCl]Cl$	96.5 (95.8)	241 (240)	0 (0)
11	$Pd(PPh_3)_4$	28.6	29	5.23

Results of repeat reactions in parentheses. Extensive catalyst decomposition observed during reaction, except those which did not afford any product.

[a] Isolated yields of 4-methylbiphenyl, based on bromobenzene.

[b] TON h^{-1}: number of moles of desired product per mole of catalyst used per hour.

[c] *Homo*-coupled product, 4,4′-dimethylbiphenyl, yield.

considerably greater reactivities than either the 'ligand-free' systems or the traditional $Pd(PPh_3)_4$ catalyst under similar reaction conditions. Little difference was seen in the reactivity of the various multiply methyl substituted imidazoles and 1-methylimidazole itself (Table 2, entries 2, 4 and 5). However, a much reduced activity was observed for the 1-phenylimidazole (Table 2, entry 6), so *N*-substitution can greatly effect the efficacy of the catalyst. We are continuing to investigate this. Although *homo*-coupling was clearly seen for the 'ligand-free' and $Pd(PPh_3)_4$ catalyst systems, it was not found in any of the reactions using imidazole ligands.

One of the primary drawbacks of the Suzuki reaction in synthesis is the production of a *homo*-coupled by-product that can be difficult to separate from the primary reaction product (Table 2, entries 7, 8 and 11) [19]. It is, therefore, of great interest that no *homo*-coupling was observed with any of the imidazole-based catalysts, under any of the reaction conditions used. This suggests that the catalysts formed in this system do not catalyse the *homo*-coupling reaction. This increased selectivity for the Suzuki product is an important potential advantage of these systems.

The $[(mim)_3PdCl]Cl$ shows very similar reactivity to the in situ generated catalyst (Table 2, entries 2 and 10), suggesting that such palladium imidazole complexes are, at least, the immediate catalyst precursor.

The $(CH_3CN)_2PdCl_2$/4mim catalysed Suzuki reactions of different halogenoarenes were also studied in dioxane (Table 3). The results exhibit no appreciable difference in yields between the activated and deactivated bromo- and iodoarenes, all affording around a 95% yield. This indicates the high activity of this system. However, only the electron-deficient, activated 4-chloroacetophenone cross-coupled to any appreciable extent, affording a 56.2% yield. Addition of the chlorobenzene also resulted in instantaneous catalyst decomposition of the $(CH_3CN)_2PdCl_2$/4mim dioxane

Table 3
Scope of the Suzuki reaction with different halogenoarenes in dioxane

Entry	X	R	Yield (%)[a]	TON h^{-1} [b]
1	I	H	95.5	239
2	Br	H	93.7 (96.3)	234 (241)
3	Cl	H	4.9 (6.4)	12 (16)
4	Br	$COCH_3$	96.2	241
5	Br	CH_3	95.9	240
6	Br	OCH_3	94.6	237
7	Cl	$COCH_3$	56.2 (54.7)	140 (137)
8	Cl	OCH_3	0	–

Repeat reactions in parentheses. Extensive catalyst decomposition observed during reaction.

[a] Isolated yield of 4-methylbiphenyl, based on bromobenzene.

[b] TON h^{-1}: number of moles of desired product per mole of catalyst used per hour.

Table 4
Investigation of Suzuki reaction solvent

Entry	Temperature (°C)	Solvent	Yield (%)[a]	TON h^{-1} [b]
1	110	Dioxane	95.7 (94.2)	239 (236)
2	110	Toluene	89.8 (92.3)	225 (231)
3	65	THF	36.8 (38.0)	92 (95)
4[c]	100	Water	92.8 (94.1)	232 (235)

Repeat reactions in parentheses. Extensive catalyst decomposition observed during reaction.

[a] Isolated yield of 4-methylbiphenyl, based on bromobenzene.

[b] TON h^{-1}: number of moles of desired product per mole of catalyst used per hour.

[c] Palladium added as $[(mim)_3PdCl]Cl$ with no additional 1-methylimidazole.

solution. Extensive catalyst decomposition was also observed at the end of each reaction. Whereas no unreacted substrate was detected for the bromo- and iodoarenes, in all cases unreacted chloroarene was found. No *homo*-coupling was observed in any reaction.

The reaction can also be performed in toluene with a slight reduction in yield. In THF, a considerably lower yield was afforded. This is in a large part due to the lower reflux temperature for THF (65 °C) leading to a lower rate of reaction (cf. Table 1). Extensive catalyst decomposition was observed in all the reactions. No *homo*-coupling was observed in any case.

$[(mim)_3PdCl]Cl$ is soluble in water, leading to the possibility of its use for aqueous Suzuki reactions. The reaction of bromobenzene and tolylboronic acid in water catalysed by $[(mim)_3PdCl]Cl$ gave a 92.8% yield in 20 min (Table 4). In addition to efficient catalyst-product separations, which are particularly important for large-scale industrial processes, aqueous reaction media offer a number of economical and environmental benefits [20]. Recently, there has been extensive research into aqueous palladium catalysed Suzuki reactions [21]. These systems commonly employ charged, water soluble, phosphine ligands, such as sulfonated aryl phosphines or quaternary ammonium salt derivatives of alkylphosphines. In some cases neutral, water soluble, phosphines have been prepared with glycosides or polymer ethylene glycol supports. While all of these phosphines supply water soluble catalysts, the need to prepare specialist ligands adds greatly to the difficulty and expense of the process, and limits the number of ligands that can be used, whereas there are many commercially available imidazoles. The preliminary result of the aqueous mediated Suzuki reaction with the water soluble, phosphine free $[(mim)_3PdCl]Cl$, therefore, represents an exciting new area for aqueous Suzuki catalysis.

3. Conclusions

The novel palladium/imidazole catalyst system was found to be active for Suzuki reactions in dioxane, toluene and water, giving near quantative yields for bromo- and iodoarenes irrespective of their functionality. Similarly high yields were afforded at 25 °C for 24 h. A good yield was achieved for the activated 4-chloroacetophenone, although no significant amount of coupling was observed for chlorobenzene. While the reactivities demonstrated here do not compete with those found for the most active systems that have been recently developed, they have in no way been optimised in terms of the most effective imidazole possible and the number of either commercially available or simply synthetically accessible imidazoles is vast. Hence, the potential for generation of a highly active catalyst is great.

A significant advantage of these catalysts is the complete removal of *homo*-coupling. Hence, the cross-coupled product could be isolated without any further purification. Also, the catalytic systems were readily prepared in situ from commercially available, inexpensive and air stable imidazoles and palladium sources, circumventing the need for synthesis of ligands and/or palladium complexes. Extensive catalyst decomposition was, however, observed in every reaction and this problem needs to be addressed.

This preliminary investigation highlights the exciting development of novel palladium/imidazole catalytic systems, with potential applications in room temperature and aqueous mediated Suzuki reactions. Further investigations to improve the reactivity and to extend the scope of these catalytic systems are currently in progress.

4. Experimental

4.1. Suzuki reactions with $(CH_3CN)_2PdCl_2$/4mim

In a typical $(CH_3CN)_2PdCl_2$/4mim catalysed Suzuki reaction in dioxane a flask was injected with

$(CH_3CN)_2PdCl_2$ (2.36 mg (0.590 cm^3, 15.43 mM), 1.2 mol.%) and 1-methylimidazole (3.00 mg (0.02050 cm^3, 1.784 M), 4.8 mol.%) under N_2 from CH_3CN standard solutions. The CH_3CN was removed in vacuo for 0.5 h without heating affording an off-white solid. The dioxane was injected and the mixture heated at 110 °C for 1 h with stirring, cooled to room temperature and then stirred for a further 10 min. Na_2CO_3 (161.2 mg, 1.52 mmol, 2 eq.) in deoxygenated water (0.80 ml), bromobenzene (119.3 mg (0.0800 cm^3), 0.760 mmol, 1 eq.) and the tolylboronic acid (113.8 mg, 0.837 mmol, 1.1 eq.) were added to the yellow solution, which was then heated at 110 °C for 20 min. On completion of the reaction, the mixture was rapidly quenched in an acetone-solid CO_2 bath, diluted with water (2 ml) and extracted with hexane (4×15 cm^3). The completely colourless extractions were washed with brine and water, filtered through a pad of silica and evaporated to dryness to afford 4-methylbiphenyl (127.6 mg, 95.7%) as a completely colourless crystalline solid.

4.2. Preparation of [(mim)$_3$PdCl]Cl

A mixture of $PdCl_2$ (558 mg, 3.15 mmol, 1 eq.) and 1-methylimidazole (2.58 g (2.51 cm^3), 31.4 mmol, 10 eq.) was heated at reflux in CH_3CN (50 cm^3) with vigorous stirring for 6 h under N_2. The pale yellow solution was slowly reduced in vacuo to dryness, washed with ethanol and ether, then dried to give [(mim)$_3$PdCl]Cl (971 mg, 72.9%) as an off-white powdery solid; mp 214–215 °C (decomp); ν_{max} (cm^{-1}) (KBr) 3104 and 3054 (CH aromatic), 2922, 2867 and 2780 (CH aliphatic), 1543, 1526, 1121, 837, 764, 660 and 623; δ_H (ppm) (270 MHz, $CDCl_3$) 7.70 (s, N_2CH, 1H), 7.17 and 6.85 (d, NC*H*, 2H), and 3.76 (s, NCH_3, 3H); δ_C (ppm) (270 MHz, D_2O) 139.3 (N_2CH, 1C), 128.2 and 122.5 (NCH, 2C), and 34.50 (NCH_3, 1C); Acc. Mass (FAB) 389.0306 correct for $C_{12}H_{18}N_6Cl_1Pd_1$; *m/z* (FAB) 389 ($[(mim)_3PdCl]^+$, 100%), 270 ($[(mim)_2Pd]^+$, 50), 188 ($[(mim)Pd]^+$, 10) and 83 ($[mimH]^+$, 22).

4.3. Preparation of bis-(acetonitrile)palladium dichloride, $(CH_3CN)_2PdCl_2$

A suspension of $PdCl_2$ (2.00 g, 11.3 mmol) was heated at reflux in CH_3CN (100 cm^3) with vigorous stirring for 18 h under N_2. Hot filtration of the resultant wine-red coloured solution through a ceolite pad into stirred petroleum spirit (40–60 °C) at room temperature afforded a yellow-orange solid. Recrystallisation from CH_3CN (200 cm^3), DCM (300 cm^3) and hexane (100 cm^3) gave $(CH_3CN)_2PdCl_2$ (1.78 g, 65.3%) as a bright yellow powdery solid; mp 129–131 °C (decomp); Found: C, 18.63; H, 2.27; N, 10.77; $C_4H_6N_2Cl_2Pd$ requires C, 18.55; H, 2.33; N, 10.82%; ν_{max} (cm^{-1}) (C≡N) 2305 vs (KBr); δ_H (ppm) (270 MHz, d^6-DMSO) 2.06 (s, CH_3, 3H); δ_C (ppm) (270 MHz, d^6-DMSO) 118.0 (s, CH_3CN, 1C) and 1.09 (s, CH_3CN, 1C).

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