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In situ generated 1-alkylbenzimidazole–palladium catalyst for the Suzuki coupling of aryl chlorides

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Abstract

Six 1-alkylbenzimidazole have been prepared and characterized by conventional spectroscopic methods and elemental analyses. Novel in situ generated palladium–benzimidazole complexes, tested in the Suzuki coupling reaction between phenylboronic acid and several aryl chlorides showed excellent catalytic activity.

Keywords: Palladium; Benzimidazole ligands; C—C coupling; Suzuki reaction

1. Introduction

Biaryls constitute important building blocks for the synthesis of biologically active substances, e.g. pharmaceuticals and herbicides [1]. Palladium-catalyzed cross-coupling reaction between aryl halides or triflates and organometallic reagents (Sn, Mg, B, Li, Zn, etc.) have been developed as a versatile and efficient method for a variety of synthetic transformations [2]. Among these reactions, palladium catalyzed cross-coupling reaction between aryl halides and aryl boronic acids, known as Suzuki–Miyaura reaction, is highly valuable for the synthesis of symmetrical and unsymmetrical biaryls [3]. Its popularity is based on its wide functional-group tolerance and the ready availability and low toxicity of the required boronic acids. The ability to use aryl chlorides as substrates in Suzuki biaryl coupling reactions, rather than the far more commonly employed aryl bromides, is advantageous for two reasons. Firstly there are many more commercially available aryl chlorides than bromides and secondly they are much cheaper. These considerations are particularly important for industrial applications. Consequently the search for catalysts that can active these substrates is a highly topical field of study. Notable catalyst developments for the use of aryl chlorides [4] have been reported by Buchwald [5], Fu [6], Bedford [7], Beller [8], us [9] and others [10]. In this respect the most active ligands for Suzuki reactions which are composed of sterically demanding basic phosphines, still have significant drawback such as the high sensitivity towards oxygen and/or the availability of the ligand. Therefore the development of more efficient ligands, which lead to highly active catalyst systems and can easily be prepared and modified on a larger scale is still an important topic in this area.

Transition metal complexes with nitrogen-containing ligands have recently shown their potential to perform selective catalytic transformations of molecules with atom economy [11]. Especially, a variety of RuX_2(arene)(L) complexes are promoting catalytic reactions such as nucleophilic addition to triple bonds to form furans (L = imidazoline, tetrahydropyrimidine [12], benzimidazole [13]), hydrogen transfer (L = diamine [14]), or Diels-Alder cycloaddition and Claisen rearrangement (L = bisoxazoline [15,16]). Palladium catalysts bearing nitrogen-donor ligands (imidazoline) have proved to be effective for CO/styrene copolymerization [17]. 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 cyclometalled palladium complexes incorporating either the

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imine [18], oxazoline moieties [19]. Recently, *N*-coordinated palladium–imidazole and imidazoline complexes active catalysts for Suzuki coupling [20], and *N*-coordinated bis- and tris-imidazole chelates palladium complexes that were found to be catalytically active in the Heck reactions [21].

However, the development of new ligands or the application of existing ligands in Suzuki reaction, particularly involving aryl chlorides as substrates, is still of considerable importance. In order to find more efficient palladium catalysts we have prepared a series of new 1-alkylbenzimidazole (**1**–**6**) and we reported here in situ Pd-*N*-coordinated 1-alkylbenzimidazole based catalytic system for the Suzuki coupling reaction.

2. Experimental

^{1}H NMR and ^{13}C NMR spectra were recorded using a Bruker AC300P FT spectrometer operating at 300.13 MHz (^{1}H), 75.47 MHz (^{13}C). Chemical shifts (δ) are given in ppm relative to TMS, coupling constants (*J*) in Hz. FT-IR spectra were recorded on a Mattson 1000 spectrophotometer, wave numbers in cm^{-1}. Melting points were measures in open capillary tubes with an Electrothermal-9200 melting point apparatus and uncorrected. Elemental analyses were performed by TUBITAK (Ankara, Turkey) Microlab.

2.1. General procedure for the preparation of the 1-alkylbenzimidazole

Potassium hydroxide (1 mmol) was added to a solution of benzimidazole (1 mmol) in ethanol (20 mL), the mixture was stirred for 1 h at room temperature, and the corresponding alkyl halides was added dropwise and heated for 8 h at 76 °C. The mixture was diluted with 30 mL of water and extracted with chloroform (3 × 10 mL), the combined extracts were washed with water and dried over $MgSO_4$, the solvent was distilled off, and the residue was either distilled under reduced pressure or recrystallized from dichloromethane/hexane.

2.2. 1-(2-Methoxyethyl)benzimidazole, ***1***

Yield: 85%, b.p: 105–106 °C/0.1 mmHg, AnalCalfor $C_{10}H_{12}N_2O$; C: 68.16, H: 6.86, N: 15.89; found C: 68.17, H: 6.85, N: ^{1}H NMR (δ, $CDCl_3$): [s, 3H, $CH_2CH_2OCH_3$]; [t, *J* = 4.8 Hz, 2H, $CH_2CH_2OCH_3$]; [t, *J* = 4.8 Hz, 2H, $CH_2CH_2OCH_3$]; 7.21-[m, 4H, Ar-*H*]; [s, 1H, 2-C*H*]^{13}C {H}NMR (δ, $CDCl_3$): 45.2, 59.2, [$CH_2CH_2OCH_3$], 109.7, 120.5, 122.3, 123.1, [Ar-*C*], [2-*C*H].

2.3. 1-(2-Diethylaminoethyl)benzimidazole, ***2***

Yield: 78%, b.p.: 109–110 °C/0.1 mmHg, Anal. Cal. for $C_{13}H_{19}N_3$; C: 71.85, H: 8.81, N: 19.34; found C: 71.87, H: 8.82, N: 19.34. ^{1}H NMR (δ, $CDCl_3$): 0.95 [t, *J* = 7.2 Hz, 6H, $CH_2CH_2N(CH_2CH_3)_2$]; 2.54 [quar., *J* = 7.2 Hz, 4H, $CH_2CH_2N(CH_2CH_3)_2$]; 2.82 [t, *J* = 6.8 Hz, 2H, $CH_2CH_2N(CH_2CH_3)_2$]; 4.21 [(t, *J* = 6.8 Hz, 2H, $CH_2CH_2N(CH_2CH_3)_2$]; 7.24–7.80 [m, 4H, Ar-*H*]; 7.98 [s, 1H, 2-C*H*]. ^{13}C {H} NMR (δ, $CDCl_3$): 12.2, 44.3, 47.7, 52.8 [$CH_2CH_2N(CH_2CH_3)_2$]; 109.7, 120.4, 122.2, 122.9, 133.9, 141.3 [Ar-*C*]; 143.8 [2-*C*H].

2.4. 1-(3,4,5-Trimethoxybenzyl)benzimidazole, ***3***

Yield: 93%, m.p.: 135–136 °C Anal. Cal. for $C_{17}H_{18}N_2O_3$; C: 68.44, H: 6.08, N: 9.39; found C: 68.43, H: 6.09, N: 9.39. ^{1}H NMR (δ, $CDCl_3$): 3.73 and 3.80 [s, 9H, 3,4,5-$CH_2C_6H_2(OCH_3)_3$]; 5.24 [s, 2H, 3,4,5-$CH_2C_6H_2(OCH_3)_3$]; 6.37 [s, 2H, 3,4,5-$CH_2C_6H_2(OCH_3)_3$]; 7.24–7.82 [m, 4H, Ar-*H*]; 7.93 [s, 1H, 2-C*H*]. ^{13}C {H} NMR (δ, $CDCl_3$): 49.3 and 56.4 [3,4,5-$CH_2C_6H_2(OCH_3)_3$]; 61.1 [3,4,5-$CH_2C_6H_2(CH_3)_3$]; 104.6, 110.2, 120.6, 122.6, 123.3, 131.2, 134.2, 134.3, 138.2, 144.1 [Ar-*C*]; 153.9 [2-CH].

2.5. 1-(2,4,6-Trimethylbenzyl)benzimidazole, ***4***

Yield: 89%, m.p.: 103–104 °C Anal. Cal. for $C_{17}H_{18}N_2$; C: 81.56, H: 7.25, N: 11.19; found C: 81.55, H: 7.25, N: 11.18. ^{1}H NMR (δ, $CDCl_3$): 2.23 and 2.33 [s, 9H, 2,4,6-$CH_2C_6H_2(CH_3)_3$]; 5.23 [s, 2H, 2,4,6-$CH_2C_6H_2(CH_3)_3$]; 6.96 [s, 2H, 2,4,6-$CH_2C_6H_2(CH_3)_3$]; 7.29–7.83 [m, 4H, Ar-*H*]; 7.44 [s, 1H, 2-C*H*]. ^{13}C {H}NMR (δ, $CDCl_3$): 19.8 and 21.3 [2,4,6-$CH_2C_6H_2(CH_3)_3$]; 43.4 [2,4,6-$CH_2C_6H_2(CH_3)_3$]; 109.8, 120.6, 122.5, 123.1, 127.4, 129.9, 134.5, 138, 138.9 [Ar-*C*]; 144.3 [2-*C*H].

2.6. 1-(1-methyl-2-Dimethylaminoethyl)benzimidazole, ***5***

Yield: 84%, m.p.: 123–124 °C Anal. Cal. for $C_{12}H_{17}N_3$; C: 70.90, H: 8.43, N: 20.67; found C: 70.93, H: 8.45, N: 20.68. ^{1}H NMR (δ, $CDCl_3$): 0.82 [d, *J* = 6.8, 3H, $CH(CH_3)CH_2N(CH_3)_2$]; 2.17 [s, 6H, $CH(CH_3)CH_2N(CH_3)_2$]; 2.91 [hex., *J* = 6.8, 1H, $CH(CH_3)CH_2N(CH_3)_2$]; 3.77 and 3.83 [m, 2H, $CH(CH_3)CH_2N(CH_3)_2$]; 7.13–7.73 [m, 4H, Ar-*H*]; 7.89 [s, 1H, 2-C*H*]. ^{13}C {H} NMR (δ, $CDCl_3$): 11.4, 40.8, 48.1, 58.6 [$CH(CH_3)CH_2N(CH_3)_2$]; 109.8, 120.3, 122.1, 122.7, 134.2 [Ar-*C*]; 143.8 [2-*C*H].

2.7. 1-(2-Morpholinoethyl)benzimidazole, ***6***

Yield: 82%, m.p.: 67–68 °C. Anal. Cal. for $C_{13}H_{17}N_3O$; C: 67.51, H: 7.41, N: 18.17; found C: 67.49, H: 7.40, N: 18.20. ^{1}H NMR (δ, $CDCl_3$): 2.57 and 2.64 [t, *J* = 7.2 Hz, 4H, NCH_2CH_2N]; 3.57[t, *J* = 6.4 Hz, 4H, $\overline{N(CH_2CH_2)_2O}$]; 4.13[t, *J* = 6.4 Hz, 4H, $\overline{N(CH_2CH_2)_2O}$]; 57.8, 67.1[$\overline{NCH_2CH_2)_2O}$]; 7.15–7.71 [m, 4H, Ar-*H*]; 7.93 [s, 1H, 2-C*H*]. ^{13}C {H}NMR (δ, $CDCl_3$): 42.6, [53.9

NCH_2CH_2N]; 57.8, 67.1[$(N(CH_2CH_2)_2O$]; 109.8, 120.3, 122.1, 122.7, 134.2 [Ar-*C*]; 143.8 [2-*C*H].

2.8. General procedure for the Suzuki type coupling reactions

$PdCl_2(CH_3CN)_2$ (1.5% mmol), 1-alkylbenzimidazole, **1**–**6** (3% mmol), aryl chloride (1.0 mmol), phenylboronic acid (1.5 mmol), Cs_2CO_3 (2 mmol) toluene (3 mL) were added in a small Schlenk tube under argon and the mixture was heated at 80 °C for 6 h. At the conclusion of the reaction mixture was cooled then volatiles were removed under vacuum. The product extracted with Et_2O, filtered through a pad of silicagel with copious washings, concentrated and purified by flash chromatography on silicagel. Purity of compounds was checked by NMR and yields are based on arylchloride.

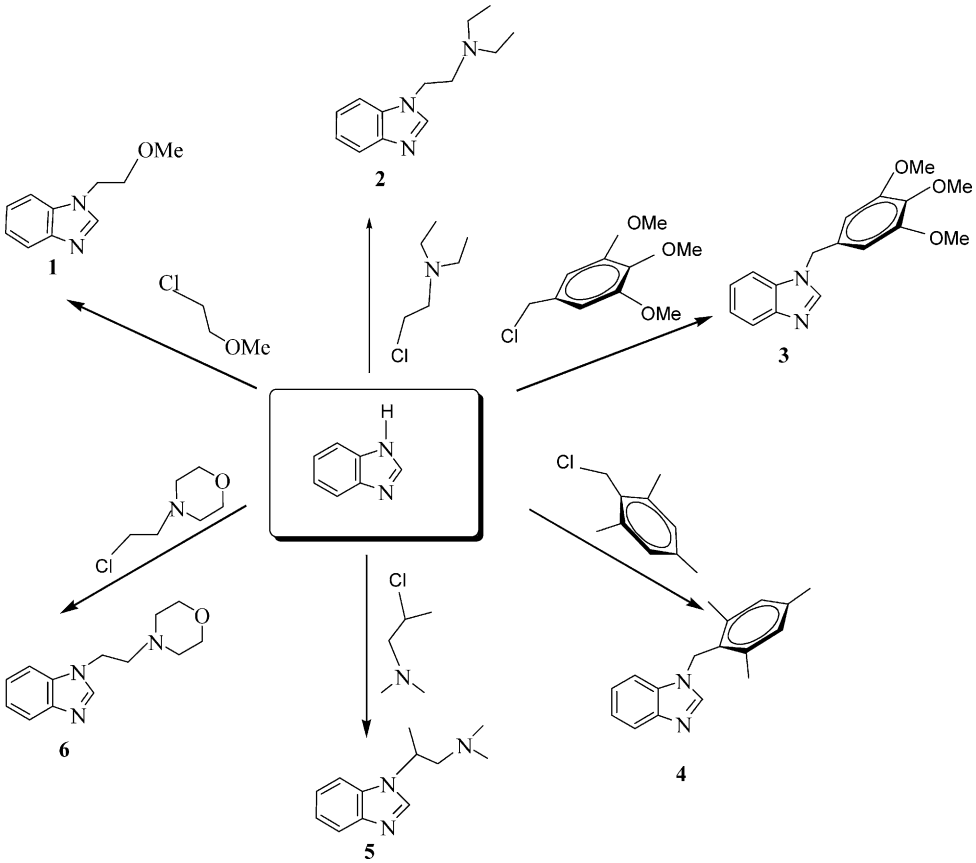
3. Results and discussion

The 1-alkylbenzimidazoles were synthesized according to the steps illustrated in Scheme 1.

1-Alkylbenzimidazoles **1**–**6** have been characterized by analytical and spectroscopic techniques. The ^{1}H NMR spectra clearly exhibit a singlet at 7.44–7.98 ppm typical of the N = C*H*–N fragment. In ^{13}C NMR, the chemical shift of the corresponding C(2) atom was detected in the region 143.8–153.9 ppm.

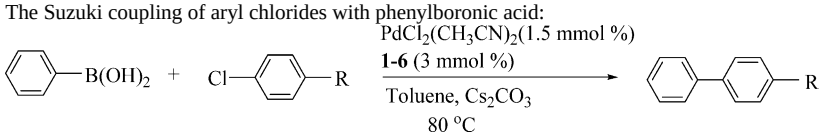
The IR data for benzimidazoles **1**–**6** clearly indicate the presence of the –C=N– group with a *ν*(C=N) vibration at 1466, 1464, 1458, 1460, 1464, and 1452 cm^{-1} respectively for **1**–**6**. These new 1-alkylbenzimidazole show typical spectroscopic signatures which are in line with those recently reported for other alkylbenzimidazole [22].

The catalysts were prepared in situ from $PdCl_2(CH_3CN)_2$ and the appropriate 1-alkybenzimidazole in the toluene. The palladium-catalyzed cross-coupling of arylboronic acids with aryl halides has been shown to proceed under a variety of conditions: A wide range of bases and solvents, as well as catalysts, have been employed with varying degrees of success according to the substrates[1]. To find optimum conditions a series of experiments has been performed with 4-chloroanisole and phenylboronic acid as model compounds. As a base, Cs_2CO_3 was the best choice and as a solvent toluene was found to be better than other solvents. After having established the optimised coupling reaction conditions, the scope of the reaction and efficiencies of the salts were evaluated by investigating the coupling of $C_6H_5B(OH)_2$ with various *p*-substituted aryl chlorides. The results were summarized in Table 1. Under those conditions, *p*-chlorobenzene, *p*-chlorotoluene, *p*-chlorobenzaldehyde, *p*-chloroacetophenone and *p*-chloroanisole react very cleanly



Scheme 1. Synthesis of 1-alkylbenzimidazole. Reaction conditions: KOH, benzimidazole, RX, EtOH, 76 °C.

Table 1
The Suzuki coupling of aryl chlorides with phenylboronic acid:



Entry	Ligands	R	Yield (%)[a,b,c,d]
1	**1**	H	97
2	**2**	H	90
3	**3**	H	96
4	**4**	H	94
5	**5**	H	88
6	**6**	H	84
7	**1**	CH_3	88
8	**2**	CH_3	75
9	**3**	CH_3	89
10	**4**	CH_3	82
11	**5**	CH_3	70
12	**6**	CH_3	66
13	**1**	CHO	93
14	**2**	CHO	90
15	**3**	CHO	92
16	**4**	CHO	88
17	**5**	CHO	85
18	**6**	CHO	83
19	**1**	$COCH_3$	98
20	**2**	$COCH_3$	95
21	**3**	$COCH_3$	94
22	**4**	$COCH_3$	91
23	**5**	$COCH_3$	84
24	**6**	$COCH_3$	87
25	**1**	OCH_3	93
26	**2**	OCH_3	80
27	**3**	OCH_3	92
28	**4**	OCH_3	85
29	**5**	OCH_3	75
30	**6**	OCH_3	78

[a] Reaction conditions: 1.0 mmol of R-C_6H_4Cl-*p*, 1.5 mmol of phenylboronic acid, 2 mmol Cs_2CO_3, 1.50 mmol% $PdCl_2(CH_3CN)_2$, 3.0 mmol% **1**–**6**, toluene (3 mL).
[b] Purity of compounds is checked by NMR and yields are based on *p*-arylchloride.
[c] All reactions were monitored by GC.
[d] Temperature 80 °C, 6 h.

with phenylboronic acid in good yields (Table 1, entries 3, 9, 13, 20, and 25).

In this catalytic system, when benzimidazole was used as ligand, no coupling was observed as a result. From the results in Table 1, it is evident that the 1-alkylbenzimidazole precursors that contain electron donating methoxyethyl substituent (**1**) are the most effective of the benzimidazole examined. The coordinating ability of the alkoxy group may be an important contributor to the increase in reactivity that has been demonstrated by previous examples [9].

4. Conclusion

The novel palladium/1-alkylbenzimidazole catalyst system was found to be active for Suzuki reactions in toluene, giving quantitative yields for *p*-chloroarenes. A good yield was achieved for the activated *p*-chloroacetophenone, although no significant amount of coupling were observed for other chloroarenes. The new ligand family allows highly efficient coupling reactions of electron rich as well as electron poor aryl chlorides with phenylboronic acid under mild conditions. To further exploit the advantageous properties displayed by the palladium/benzimidazole and imidazoline systems, catalytic investigations focusing on a number of cross-coupling reactions are ongoing in our laboratories.

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