

Benzimidazolin-2-ylidene–palladium-catalysed coupling reactions of aryl halides

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The *in situ* prepared three-component system Pd(OAc)₂–1,3-dialkylbenzimidazolium chlorides (2a–f) and Cs₂CO₃ catalyses, quantitatively, the Suzuki cross-coupling of deactivated aryl chlorides and Heck coupling reactions of aryl bromide and iodide substrates. The 1,3-dialkylbenzimidazolium salts (2a–f) were characterized by conventional spectroscopic methods and elemental analysis. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: Heck; Suzuki; carbene; palladium; aryl halides; dialkylbenzimidazolium

INTRODUCTION

An important goal in organic chemistry is the development of green processes that use fewer raw materials and less energy, maximize the use of renewable resources, and minimize or eliminate the use of dangerous chemicals. Clearly, none of this is possible without catalysis. In general, as the science of accelerating chemical reactions, catalysis is about using value-added transformations to convert simple raw materials to more complex molecules with versatile application characteristics. In this regard, palladium catalysts have emerged as extremely powerful tools for the construction of carbon–carbon (Scheme 1) and carbon–heteroatom bonds.^{1,2}

Numerous review articles have documented the increasing frequency with which palladium-catalysed coupling processes are applied to a wide array of endeavours, which range from synthetic organic chemistry to materials science. Their popularity stems in part from their tolerance for many functional groups, which allows them to be employed in the synthesis of highly complex molecules.^{3–6} The palladium-catalysed reaction of aryl chlorides with arylboronic acid (the Suzuki reaction) or with alkenes (the Heck reaction) is one of the most common methods

for C–C bond formation and has attracted much current interest.^{7–11} The reactions are usually carried out homogeneously in the presence of a base under inert atmosphere.

Lately, various bulky and electron-rich phosphines have been developed as ligands to promote the cross-coupling reaction.^{12,13} The ligands may afford coordinatively unsaturated monophosphine-ligated complexes and accelerate the catalytic steps, i.e. oxidative addition, transmetalation, and reductive elimination. As a result, even relatively less-reactive aryl chlorides can be used effectively in these coupling reactions.¹⁴

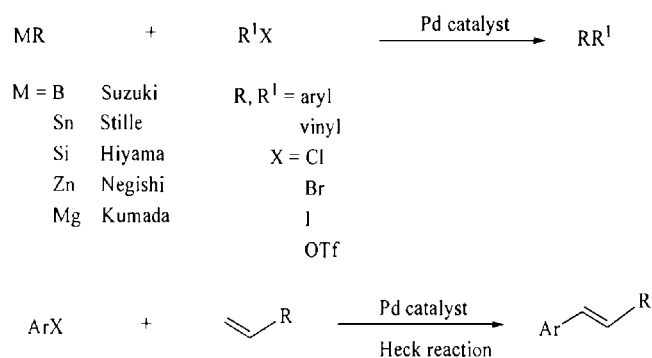
Recently, it has been shown that palladium complexes of *N*-heterocyclic carbene (NHC) ligands offer distinct advantages as possible alternatives to palladium–phosphine systems in C–C coupling reactions.^{15–21} Some highly active palladium systems with monodentate carbene ligands for the activation of aryl chlorides have been developed.^{22–24} It is worth noting that ‘*in situ*’ formation of the NHC complex by deprotonation of the imidazol(in)ium salt led to significantly better results than the use of the preformed complex.

In order to find more efficient palladium catalysts, we have prepared a series of new bulky or functional 1,3-dialkylbenzimidazolium chlorides (2a–f) (Scheme 2) containing a benzimidazole ring. Herein, we report a mild, practical *in situ* generated catalytic system composed of commercially available and stable reagents, using Pd(OAc)₂ as the palladium source, 1,3-dialkylbenzimidazolium chlorides (2a–f) as carbene precursors and Cs₂CO₃ as a base for Suzuki and Heck cross-coupling of aryl halides.

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Scheme 1.

EXPERIMENTAL

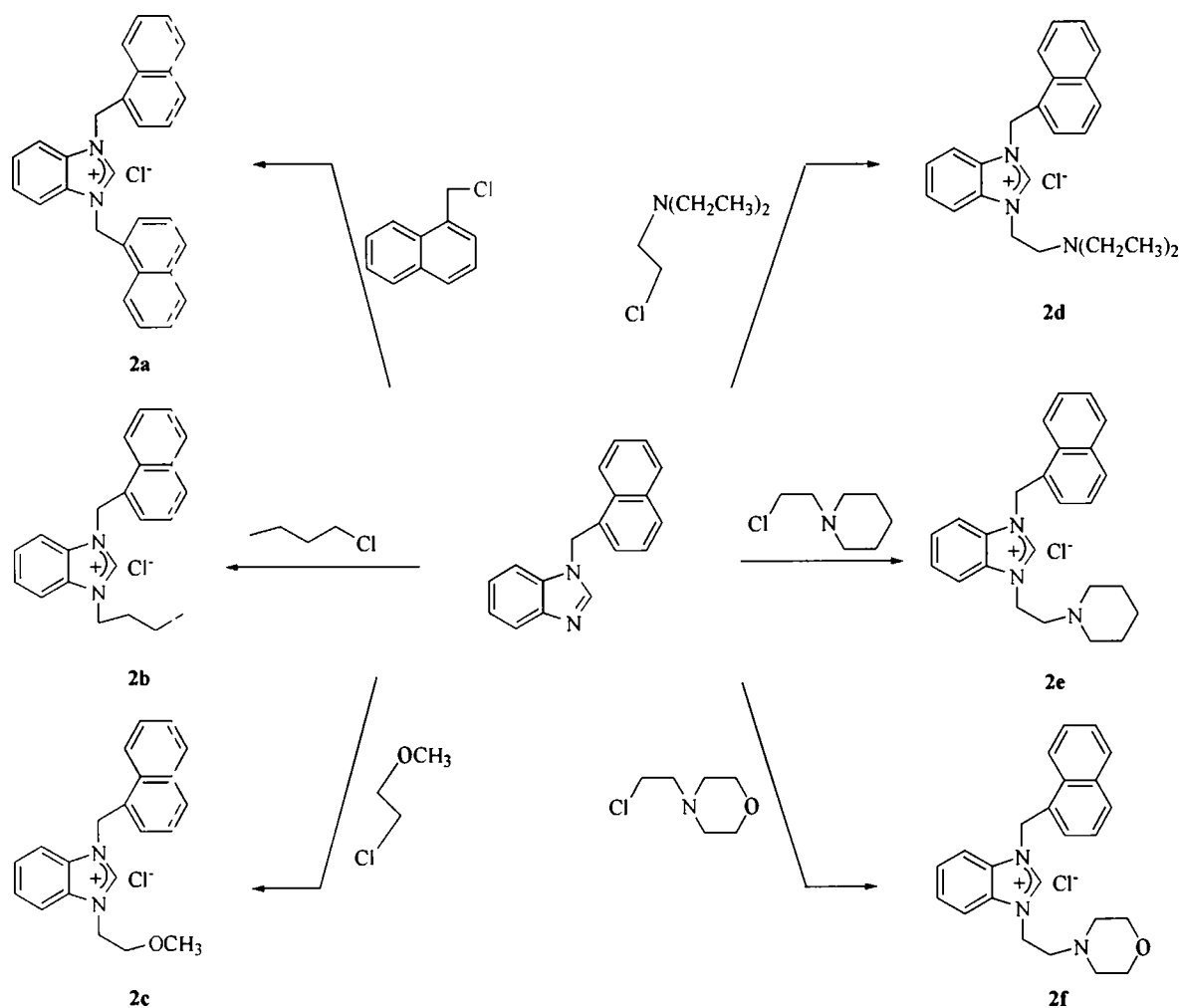
All reactions for the preparation of 1-naphthalenomethylbenzimidazoline²⁵ and 1,3-dialkylbenzimidazolium salts were carried out under argon using standart Schenk-type flasks. Test reactions for the catalytic activity of catalysts in the

Suzuki and Heck cross-coupling reactions were carried out in air. All reagents were purchased from Aldrich Chemical Co. The solvents, Et₂O over Na–K alloy, dimethylformamide (DMF) over BaO, and EtOH over Mg, were distilled prior to use.

All ¹H and ¹³C NMR spectroscopy was performed in CDCl₃. ¹H NMR and ¹³C NMR spectra were recorded using a Bruker AC300P FT spectrometer operating at 300.13 MHz (¹H) or 75.47 MHz (¹³C). Chemical shifts δ are given in parts per million relative to tetramethylsilane, coupling constants *J* are given in hertz. FT-IR spectra were recorded on a Mattson 1000 spectrophotometer, and wavenumbers are in reciprocal centimetres. Melting points were measured in open capillary tubes with an Electrothermal-9200 melting-point apparatus and are uncorrected. Elemental analyses were performed by TUBITAK (Ankara, Turkey) Microlab.

Preparation of 1,3-di(naphthalenomethyl)benzimidazolium chloride (2a)

To a solution of 1-naphthalenomethylbenzimidazoline²⁵ (2 g; 7.75 mmol) in DMF (3 ml) was added slowly



Scheme 2. Synthesis of 1,3-dialkylbenzimidazolium chlorides.

1-chloromethylnaphthalene (1.38 g; 7.81 mmol) at 25 °C. The resulting mixture was stirred at room temperature for 4 h and then heated for 12 h at 80 °C. Diethyl ether (10 ml) was added to obtain a white crystalline solid, which was filtered off. The solid was washed with diethyl ether (3 × 10 ml), dried under vacuum and recrystallized from EtOH/Et₂O (1:2) at room temperature. M.p. 227–228 °C; yield: 2.4 g, 72%, ν_{CN} = 1558 cm⁻¹.

Anal. Found: C, 80.18; H, 5.30; N, 6.52. Calc. for C₂₉H₂₃ClN₂: C, 80.08; H, 5.33; N, 6.44%.

¹H NMR (δ , CDCl₃): 6.39 [s, 4H, CH₂C₁₀H₇], 7.27 and 8.22 [m, 18H, Ar-H], 12.15 [s, 1H, 2-CH]. ¹³C{H} NMR (δ , CDCl₃): 51.4 [CH₂C₁₀H₇]; 111.2, 123.0, 135.9, 123.8, 125.7, 126.5, 127.1, 128.5, 129.3, 130.9, 132.1, 133.6, 133.6 [Ar-C]; 144.3 [2-CH].

Preparation of 1-naphthalenomethyl-3-butylbenzimidazolium chloride (2b)

Compound **2b** was prepared in the same way as **2a**, from 1-naphthalenomethylbenzimidazoline (2.31 g; 13.27 mmol) and butyl chloride (2.35 g; 13.30 mmol), to give white crystals (4.00 g, 86% yield); m.p. 122–123 °C; ν_{CN} = 1560 cm⁻¹.

Anal. Found: C, 75.43; H, 6.68; N, 8.01. Calc. for C₂₂H₂₃ClN₂: C, 75.31; H, 6.61; N, 7.98%.

¹H NMR (δ , CDCl₃): 0.96 [t, 3H, *J* = 7.6 Hz, CH₂CH₂CH₂CH₃]; 1.42 [hep, 2H, *J* = 8 Hz, CH₂CH₂CH₂CH₃]; 2.01 [quin, 2H, *J* = 7.6 Hz, CH₂CH₂CH₂CH₃]; 4.62 [t, 2H, *J* = 7.6 Hz, CH₂CH₂CH₂CH₃]; 6.39 [s, 2H, CH₂C₁₀H₇]; 7.39 and 8.26 [m, 11H, Ar-H]; 11.85 [s, 1H, 2-CH]. ¹³C{H} NMR (δ , CDCl₃): 13.7, 19.9, 31.4, 47.7 [CH₂CH₂CH₂CH₃]; 49.7 [CH₂C₁₀H₇]; 114.4, 123, 125.4, 126.6, 126.9, 127, 127.6, 127.7, 128.7, 129.2, 130, 130.9, 131.6, 131.7, 133.7 [Ar-C]; 144.2 [2-CH].

Preparation of 1-naphthalenomethyl-3-(2-methoxyethyl)benzimidazolium chloride (2c)

Compound **2c** was prepared in the same way as **2a**, from 1-naphthalenomethylbenzimidazoline (2.10 g; 11.93 mmol) and 2-methoxyethyl chloride (1.78 ml; 11.95 mmol), to give white crystals (4.00 g, 95% yield); m.p. 208–209 °C; ν_{CN} = 1562 cm⁻¹.

Anal. Found: C, 71.57; H, 5.71; N, 7.93. Calc. for C₂₁H₂₁ClN₂O: C, 71.48; H, 5.60; N, 7.94%.

¹H NMR (δ , CDCl₃): 3.34 [s, 3H, OCH₃]; 3.98 [t, 2H, *J* = 4.8 Hz, CH₂CH₂OCH₃]; 4.88 [t, 2H, *J* = 4.4 Hz, CH₂CH₂OCH₃]; 6.33 [s, 2H, CH₂C₁₀H₇]; 7.44 and 8.23 [m, 11H, Ar-H], 11.84 [s, 1H, 2-CH]. ¹³C{H} NMR (δ , CDCl₃): 48.1, 49.7, 70.5 [CH₂CH₂OCH₃]; 59.2 [CH₂C₁₀H₇]; 113.8, 114.4, 122.9, 125.5, 126.7, 126.9, 127, 127.4, 127.8, 128.4, 129.3, 130.2, 130.9, 131.5, 132.5, 134 [Ar-C]; 144.5 [2-CH].

Preparation of 1-naphthalenomethyl-3-(2-diethylaminoethyl)benzimidazolium chloride (2d)

Compound **2d** was prepared in the same way as **2a**, from 1-naphthalenomethylbenzimidazoline (1.10 g; 4.26 mmol) and 2-diethylaminoethyl chloride (0.58 g; 4.27 mmol), to give

white crystals (1.32 g, 79% yield); m.p. 228–229 °C; ν_{CN} = 1563 cm⁻¹.

Anal. Found: C, 73.21; H, 7.15; N, 10.72. Calc. for C₂₄H₂₈ClN₃: C, 73.17; H, 7.16; N, 10.67%.

¹H NMR (δ , CDCl₃): 1.00 [t, 6H, *J* = 6.4 Hz, (NCH₂CH₃)₂]; 2.40 [q, 4H, *J* = 6.4 Hz, (NCH₂CH₃)₂]; 2.59 [m, 2H, NCH₂CH₂N]; 3.45 [m, 2H, NCH₂CH₂N]; 6.09 [s, 2H, CH₂C₁₀H₇]; 7.04 and 7.84 [m, 11H, Ar-H]; 11.70 [s, 1H, 2-CH]. ¹³C{H} NMR (δ , CDCl₃): 18.7, 46.4 [(NCH₂CH₃)₂]; 53.4, 52.6 [NCH₂CH₂N]; 57.8 [CH₂C₁₀H₇]; 113.7, 114.0, 122.8, 125.5, 126.6, 126.9, 127, 127.3, 127.7, 128.1, 129.2, 130.2, 130.8, 131.4, 132.5, 134 [Ar-C]; 144.4 [2-CH].

Preparation of 1-naphthalenomethyl-3-(2-piperidinoethyl)benzimidazolium chloride (2e)

Compound **2e** was prepared in the same way as **2a**, from 1-naphthalenomethylbenzimidazoline (1.30 g; 5.03 mmol) and 2-chloromethylpiperidine (0.75 g; 5.04 mmol), to give white crystals (1.79 g, 89% yield); m.p. 233–234 °C; ν_{CN} = 1567 cm⁻¹.

Anal. Found: C, 73.73; H, 6.90; N, 10.38. Calc. for C₂₅H₂₈ClN₃: C, 73.69; H, 6.95; N, 10.35%.

¹H NMR (δ , CDCl₃): 2.07 [m, 2H, NCH₂CH₂CH₂CH₂CH₂N]; 3.13 [m, 4H, NCH₂CH₂CH₂CH₂CH₂N]; 3.47 [m, 4H, NCH₂CH₂CH₂CH₂CH₂N]; 3.66 [m, 2H, NCH₂CH₂N]; 5.53 [t, 2H, *J* = 7.6 Hz, NCH₂CH₂N]; 6.11 [s, 2H, CH₂C₁₀H₇]; 7.41 and 8.61 [m, 11H, Ar-H]; 11.23 [s, 1H, 2-CH]. ¹³C{H} NMR (δ , CDCl₃): 21.9, 22.7, 52.8 [NCH₂CH₂CH₂CH₂CH₂N]; 41.7, 48.8 [NCH₂CH₂N]; 53.8 [CH₂C₁₀H₇]; 114.7, 114.8, 123.8, 126.3, 127.1, 127.5, 127.6, 127.8, 128.2, 129.5, 129.6, 130.3, 131.2, 131.8, 132.2, 134.2 [Ar-C]; 144.3 [2-CH].

Preparation of 1-naphthalenomethyl-3-[2-morpholinoethyl]benzimidazolium chloride (2f)

Compound **2f** was prepared in the same way as **2a**, from 1-naphthalenomethylbenzimidazoline (1.00 g; 3.87 mmol) and 2-chloromethylmorpholine (0.58 g; 3.88 mmol), to give white crystals (1.35 g, 85% yield); m.p. 265–266 °C; ν_{CN} = 1564 cm⁻¹.

Anal. Found: C, 70.58; H, 6.49; N, 10.38. Calc. for C₂₄H₂₆ClN₃O: C, 70.66; H, 6.42; N, 10.30%.

¹H NMR (δ , CDCl₃): 2.67 [m, 4H, N(CH₂CH₂)₂O]; 3.54 [m, 2H, NCH₂CH₂N]; 3.69 [m, 4H, N(CH₂CH₂)₂O]; 5.24 [t, 2H, *J* = 7.6 Hz, NCH₂CH₂N]; 6.30 [s, 2H, CH₂C₁₀H₇]; 7.42 and 8.60 [m, 11H, Ar-H]; 11.23 [s, 1H, 2-CH]. ¹³C{H} NMR (δ , CDCl₃): 44.1, 56.2 [NCH₂CH₂N]; 53.6, 66.7 [N(CH₂CH₂)₂O]; 53.9 [CH₂C₁₀H₇]; 114.6, 114.7, 123.8, 126.7, 127.2, 127.4, 127.6, 127.9, 128.3, 129.6, 129.8, 130.3, 131.3, 131.8, 132.3, 134.4 [Ar-C]; 144.5 [2-CH].

RESULTS AND DISCUSSION

Dialkylbenzimidazolium salts (**2a–f**) are conventional NHC precursors. The functionalized or bulky benzimidazolium

salts **2a–f** were synthesized by consecutive alkylation of 1-naphthalenomethylbenzimidazole with alkyl halides (Scheme 2).

According to Scheme 2, the salts **2a–f** were obtained in almost quantitative yield by quarternization of 1-naphthalenomethylbenzimidazole in DMF with alkyl halides.^{25–29} The salts are air- and moisture-stable, both in the solid state and in solution. The structures of **2a–f** were determined by their characteristic spectroscopic data and elemental analyses. ¹³C NMR chemical shifts were consistent with the proposed structure; the imino carbon appeared as a typical singlet in the ¹H-decoupled mode at 144.3 ppm, 144.2 ppm, 144.5 ppm, 144.4 ppm, 144.3 ppm and 144.5 ppm respectively for benzimidazolium chlorides **2a–f**. The ¹H NMR spectra of the benzimidazolium salts further supported the assigned structures; the resonances for C(2)–H were observed as sharp singlets at 12.15 ppm, 11.85 ppm, 11.84 ppm, 11.70 ppm, 11.23 ppm and 11.23 ppm respectively for **2a–f**. The IR data for benzimidazolium salts **2a–f** clearly indicate the presence of the –C=N–group with a $\nu(\text{C}=\text{N})$ vibration at 1558, 1560, 1562, 1563, 1567 and 1564 cm^{–1} respectively for **2a–f**. The NMR values are similar to those found for other 1,3-dialkylbenzimidazolium salts.^{28,29}

Palladium–NHC-catalysed Suzuki coupling reactions

To find optimum conditions, a series of experiments was performed with 4-chloroanisole and phenylboronic acid as model compounds. As a base, Cs₂CO₃ was the best choice in water–DMF systems. In addition, the reactions were performed in air and without degassing the water prior to use. 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 C₆H₅B(OH)₂ with various para-substituted aryl chlorides. The results are shown in Table 1.

Under these conditions, *p*-chloroacetophenone, *p*-chlorotoluene, *p*-chlorobenzaldehyde, *p*-chloroanisole, and chlorobenzene react very cleanly with phenylboronic acid in good yields (Table 1, entries 4, 9, 16, 22, and 28).

Table 1 summarizes our results from the screening of six benzimidazolium salts, for the Suzuki cross-coupling reaction. Several trends are readily apparent. First, the use of dialkylbenzimidazolium ligand precursors (**2a–f**) allowed lower reaction temperatures (60 °C), shorter reaction times and lower base loadings in aqueous media. The procedure is simple and does not require induction periods. Second, the scope of this reaction is broad and includes aryl chlorides that are activated or deactivated. Third, all complexes led to good conversions at low catalyst concentration (1 mol%, Pd(OAc)₂, 2 mol% **2**). It is evident that the NHC precursors that contain electron-donating methoxyethyl or diethylaminoethyl substituents (**2c**, **2d**) are the most effective of the salts examined. The coordinating ability of the alkoxy diethylaminoethyl group may be an important contributor

Table 1. The Suzuki coupling reaction of aryl chlorides with phenylboronic acid

Entry	R	LHX	Yield ^{a,b,c,d} (%)
1	COCH ₃	2a	83
2	COCH ₃	2b	76
3	COCH ₃	2c	91
4	COCH ₃	2d	97
5	COCH ₃	2e	88
6	COCH ₃	2f	86
7	CH ₃	2a	60
8	CH ₃	2b	63
9	CH ₃	2c	74
10	CH ₃	2d	72
11	CH ₃	2e	56
12	CH ₃	2f	57
13	CHO	2a	71
14	CHO	2b	63
15	CHO	2c	85
16	CHO	2d	92
17	CHO	2e	86
18	CHO	2f	85
19	OCH ₃	2a	70
20	OCH ₃	2b	77
21	OCH ₃	2c	81
22	OCH ₃	2d	83
23	OCH ₃	2e	64
24	OCH ₃	2f	67
25	H	2a	77
26	H	2b	71
27	H	2c	88
28	H	2d	90
29	H	2e	71
30	H	2f	68

^a Reaction conditions: 1.0 mmol of *p*-R–C₆H₄Cl, 1.5 mmol of phenylboronic acid, 2 mmol Cs₂CO₃, 1 mmol% Pd(OAc)₂, 2 mmol% **2**, water (3 ml)–DMF (3 ml).

^b Purity of compounds was checked by NMR and yields are based on arylchloride.

^c All reactions were monitored by thin-layer chromatography (TLC).

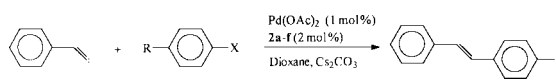
^d Temperature 60 °C, 3 h.

to the increase in reactivity, as has been demonstrated by previous examples.³⁰

Palladium–NHC-catalysed Heck-type reactions

In initial catalytic experiments, the coupling of *p*-bromoacetophenone and styrene was attempted using the *in situ* formed carbene complexes as pre-catalysts (Table 2, entries 3 and 4). All complexes led to good conversions at low catalyst concentration (1.0 mol%).

Following this, the olefination of styrene with the aryl bromides and iodide were tested, and all produced near-quantitative conversions (entries, 10, 15,

Table 2. The Heck coupling reaction of aryl bromides and iodide with styrene


Entry	R	X	LHX	Yield ^{a,b,c,d} (%)
1	COCH ₃	Br	2a	62
2	COCH ₃	Br	2b	71
3	COCH ₃	Br	2c	86
4	COCH ₃	Br	2d	91
5	COCH ₃	Br	2e	83
6	COCH ₃	Br	2f	71
7	CHO	Br	2a	93
8	CHO	Br	2b	90
9	CHO	Br	2c	95
10	CHO	Br	2d	98
11	CHO	Br	2e	58
12	CHO	Br	2f	59
13	H	Br	2a	68
14	H	Br	2b	77
15	H	Br	2c	97
16	H	Br	2d	97
17	H	Br	2e	62
18	H	Br	2f	54
19	CH ₃ O	I	2a	85 ^e
20	CH ₃ O	I	2b	89 ^e
21	CH ₃ O	I	2c	94 ^e
22	CH ₃ O	I	2d	98 ^e
23	CH ₃ O	I	2e	91 ^e
24	CH ₃ O	I	2f	90 ^e

^a Reaction conditions: 1.0 mmol of *p*-R-C₆H₄Br, 1.5 mmol of styrene, 2 mmol Cs₂CO₃, 1 mmol% Pd(OAc)₂, 2 mmol% **2**, dioxane (3 ml).

^b Purity of compounds is checked by NMR and yields are based on aryl halide.

^c All reactions were monitored by TLC.

^d Temperature 80 °C, 2 h.

^e 1 h.

16, 22). In contrast, the Heck coupling reactions of unactivated (electron-neutral) and deactivated (electron-rich) aryl chlorides, under the same catalytic conditions, did not give a detectable amount of product. The reactivity of the aryl halide component decreases drastically in the order X = I > Br > Cl, and electron-withdrawing substituents R are required for the chlorides to react.^{1,7–11}

CONCLUSIONS

In conclusion, bulky, electron-rich NHC ligand precursors with a benzimidazolidine backbone, when combined with Pd(OAc)₂, give highly active catalysts for the Suzuki coupling reaction; only aryl chlorides are reluctant to give the Heck reaction in the presence of Pd(OAc)₂–LHX. The

procedure is simple and efficient towards various aryl halides and does not require induction periods. Detailed investigations are ongoing, focusing on imidazolin-2-ylidene and benzimidazolin-2-ylidene substituent effects, functional group tolerance and catalytic activity in this and other coupling reactions.

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