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# Unsymmetric-1,3-disubstituted imidazolium salt for palladium-catalyzed Suzuki–Miyaura cross-coupling reactions of aryl bromides

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**Abstract**

A series of 1-(ferrocenylethyl)-3-substituted-imidazolium salts [3-substitute = 2,6-di(*iso*-propyl)phenyl (**1a**), 2,4,6-trimethylphenyl (**1b**), *tert*-butyl (**1c**), 1-Ad (**1d**), cyclohexyl (**1e**)] have been synthesized from a racemic ferrocenylethyl acetate and the corresponding N-substituted imidazole in high yields (70–94%). A combination of $Pd(OAc)_2$ and **1a–d** was found to form an excellent catalyst system for the Suzuki–Miyaura cross-coupling reactions of aryl bromides with phenylboronic acid in the presence of $Cs_2CO_3$.


*Keywords:* Palladium; Imidazol-2-ylidene; Suzuki–Miyaura reaction; Aryl bromides; Catalysis

## 1. Introduction

Transition metal-catalyzed reactions dominate a fundamental roll in the modern organic chemistry [1]. Among them, palladium-catalyzed cross-coupling reactions represent an extremely versatile tool in organic synthesis [2]. Coupling between organoboron reagents and aryl halides (or pseudo halides), named as the Suzuki–Miyaura reaction [3], is one of the most successful strategies for forming C–C bonds. Many of organoboron reagents used for the reaction are air- and moisture-stable, and many of them are commercially available. Furthermore, the by-products produced from boron reagents, unlike the tin-containing by-products of the Stille process [4], are nontoxic and easily separated from the desired products.

The importance of the Suzuki–Miyaura reaction has attracted much attention to elevate the efficiency of the palladium catalysts [5–12]. Recently, employing sterically demanding and electron-rich monodentate tertiary phosphines as supporting ligands has resulted in the activation of inexpensive aryl chlorides as coupling partners in the Suzuki–Miyaura reaction at room temperature [12]. Despite tertiary phosphines are effective in controlling reactivity and selectivity in organometallic chemistry and homogeneous catalysis, they require of air-free handling to prevent their oxidation. Besides, they are subject to P–C bond degradation at elevated temperatures [7], leading to that much excess of phosphine is often practiced in such catalytic processes to overcome these problems.

Since the discovery of stable N-heterocyclic carbene (NHC) by Arduengo et al. [13], increasing attention has been focused on using them as ancillary ligands; they are alternatives to phosphines, and have been used for a variety of catalytic reactions, including for palladium-catalyzed cross-coupling reactions [7,14–17].

NHCs are characteristics of electron-rich nature, leading to favoring oxidative addition and coordinating more tightly to the Pd. Importantly, their steric properties are easily tunable through the size of the substituents linking to the N atoms. Steric hindrance resulted from the size of the substituents to the N atoms plays a crucial role for the catalytic performance of the palladium catalyst coordinated by NHCs [7], and the couplings of deactivated aryl chlorides with various arylboronic acids are realized [7,16,17]. For 1,3-bis(2,6-dialkylphenyl)-imidazol-2-ylidene ligating palladium catalysts, the catalytic performance with *iso*-propyl substituent is much better than that with methyl for the Suzuki–Miyaura cross-coupling reaction [7]. However, the rigid steric bulkiness may disfavor the couplings between the substrates with *ortho* substituents. Recently, Glorius and coworkers introduced the notion of the *flexible* steric bulkiness,

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of which could vary in a range: to be large to meet steric hindrance required for the high activity of catalyst; to be small to favor the couplings with sterically hindered substrates [14,15]. The exciting result with the *flexible* steric bulkiness is to be able to realize to couple terically hindered aryl chlorides with arylboronic acids. Along with the flexible notion, we were interested in unsymmetric NHCs which may possess flexible steric hindrance produced by the different size of the two substituents on the two sides of NHC. Here we report the catalytic performance of some unsymmetric NHCs with different steric bulkiness at one side for the Suzuki–Miyaura cross-coupling reactions.

## 2. Results and discussion

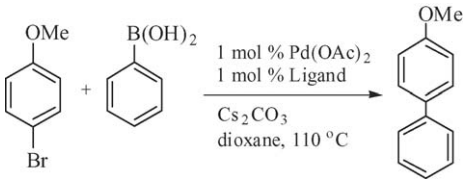
### 2.1. Synthesis of unsymmetric 1,3-disubstituted-imidazolium iodides

1,3-Disubstituted-imidazolium salts are conventional precursors of NHCs. Alkylation of N-substituted imidazole is a convenient route to produce unsymmetric 1,3-disubstituted-imidazolium salts [18]. With readily accessible racemic ferrocenylethyl acetate (**2**) as alkylation reagent [19], five of unsymmetric 1,3-disubstituted-imidazolium salts (**1**) with different steric hindrance were obtained in 70–94% of yields (Scheme 1). The structures of **1** are consistent with their characteristic spectroscopic data.

### 2.2. Influence of the imidazolium salts on Suzuki–Miyaura cross-coupling reaction

We first compared the reaction between 4-bromoanisole and phenylboronic acid using $Pd(OAc)_2$ in dioxane with $Cs_2CO_3$ as base at 110 °C to test the effect of the imidazolium salts (**1**) with different steric hindrance; the combination of palladium source, base, and solvent is a classic reaction condition for the Suzuki–Miyaura cross-coupling reaction using imidazolium salts as a precursor of NHC ligands [7]. The NHC supported palladium catalysts could be produced in situ in the above reaction condition; $Cs_2CO_3$ is an effective base to deprotonate imidazolium salts to generate NHCs, which subsequently coordinate to palladium. The results were summarized in Table 1. The imidazolium salts (**1a**–**d**) are comparable effective ligands in the above reaction condition, although **1d** is a little better (Table 1, entry 5), achieving >90% of conversions of 4-bromoanisole, an deactivated aryl bromide substrate, within 3 h. However, the imidazolium salt **1e** with cyclohexyl substituent only achieved 67% of conversion of 4-bromoanisole even after 9 h (Table 1, entry 6). This trend of the catalytic reactivities of the synthesized unsymmetric imidazolium salts is in agreement with the larger hindrance of N-substituents enhancing activity [7]. The influence of ligand-to-palladium ratio was also investigated, found that the 1:1 ratio of imidazolium salt to palladium is better than 2:1 in catalytic activity (Table 1, entry 2), which is consistent with the observation by Nolan [7].

Table 1
Effect of imidazolium salts on $Pd(OAc)_2$ catalyzed Suzuki–Miyaura cross-coupling of 4-bromoanisole with phenylboronic acid[a]

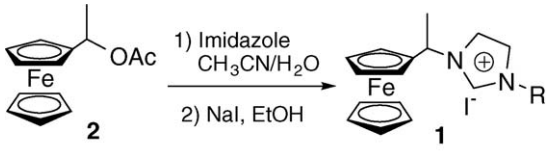



| Entry | Ligand | Time (h) | Yield[b] (%) |
|---|---|---|---|
| 1 | **1a** | 3 | 94[c] |
| 2[d] | **1a** | 3 | 91[c] |
| 3 | **1b** | 3 | 93[c] |
| 4 | **1c** | 3 | 95[c] |
| 5 | **1d** | 3 | 96 (91)[c] |
| 6 | **1e** | 9 | 67[e] |

[a] Reaction conditions: 1.0 mmol of 4-bromoanisole; 1.5 mmol of phenylboronic acid; 2.0 mmol of $Cs_2CO_3$; 3.0 mL of dioxane.
[b] GC yield against a calibrated internal standard (undecane). Isolated yields in parentheses.
[c] 1–2% of biphenyl was observed.
[d] Pd/L = 1/2.
[e] 3% of biphenyl was observed.

### 2.3. Effect of the base on Suzuki–Miyaura cross-coupling of 4-bromoanosole with phenylboronic acid

An brief investigation of the influence of a variety of bases on the $Pd(OAc)_2$/**1d**-catalyzed Suzuki–Miyaura cross-coupling suggested that $Cs_2CO_3$ was the reagent of choice (Table 2,




R = 2,6-di(*iso*-propyl)phenyl (**1a**);
2,4,6-trimethylphenyl (**1b**);
*t*-Bu (**1c**);
1-Ad(**1d**);
cyclohexyl (**1e**);

Scheme 1. Synthesis of unsymmetric 1,3-disubstituted-imidazolium salts.

Table 2
Effect of the base on $Pd(OAc)_2$/**1d** catalyzed Suzuki–Miyaura cross-coupling reaction of 4-bromoanisole with phenylboronic acid

| Entry | Base | Time (h) | Yield[a,b] (%) |
|---|---|---|---|
| 1 | $Cs_2CO_3$ | 3 | 96 |
| 2 | $K_2CO_3$ | 3 | 93 |
| 3 | NaO$^t$Bu | 12 | 67 |
| 4 | KF | 3 | <10 |

Reaction conditions: 1.0 mmol of 4-bromoanisole; 1.5 mmol of phenylboronic acid; 2.0 mmol of bases ; 2.0 mol% Pd $(OAc)_2$; 3.0 mL of dioxane.
[a] GC yields against a calibrated internal standard (undecane).
[b] 1–2% of biphenyl was observed.

entry 1). $K_2CO_3$ [20] was also a comparably effective base as $Cs_2CO_3$, and 93% of conversion for 4-bromoanisole was achieved. As a generally used base for palladium-catalyzed C–N formation, NaO$^t$Bu [21] only exhibited moderate effect for palladium-catalyzed Suzuki–Miyaura reaction with **1d** as precursor of NHC. KF, a very effective base for $Pd_2(dba)_3$/phosphine Suzuki–Miyaura reactions [22,23], in turn, proved not efficient in the present system with 10% of conversion for 4-bromoanisole. Since $Pd(OAc)_2$ and imidazolium salts could form NHC coordinating palladium complexes without base in DMSO at 50 °C [24], the poor performance of KF for $Pd(OAc)_2$/**1d** system may not the reason that the weak base cannot deprotonate imidazolium salts to form NHC.

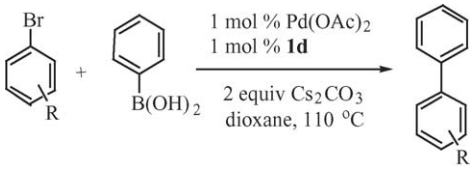
### 2.4. Substitution patterns and functional group tolerance on aryl bromides

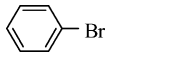
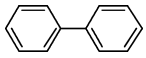
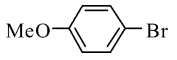
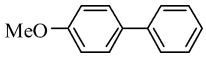
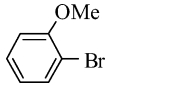
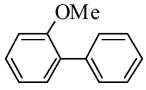
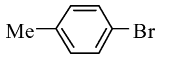
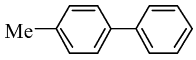
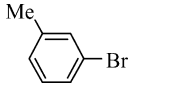
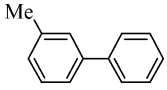
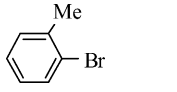
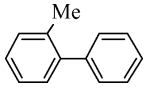
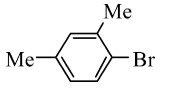
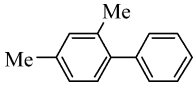
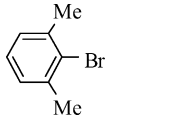
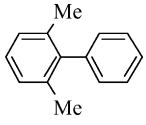
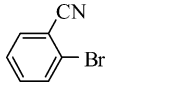
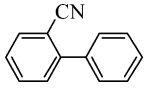
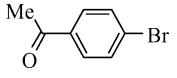
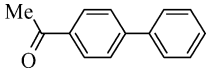
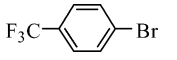
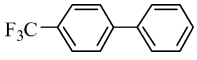
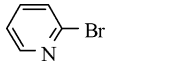
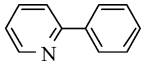
Since the $Pd(OAc)_2$/**1d** system in the presence $Cs_2CO_3$ as a base proves optimum for the coupling of the deactivated 4-bromoanisole with phenylboronic acid, it is not surprising that the catalyst system is very effective for the nonactivated and activated aryl bromides, as shown in Table 3. Compared to MeO group at *para*-position, the aryl bromides with the H, Me, and $CF_3$ substituents are more easily converted with high isolated yields of desired products (Table 3, entries 1, 4, and 11). However, the extension of reaction time was necessary for the sterically congested substrates to achieve high yields. For example, for the aryl bromides with one *ortho*-methyl substitute, 6 h was consumed for achieving 98–99% of conversions (entries 6 and 7); even for the activated aryl bromide with withdrawing substitutes at *ortho*-position, 9 h was needed for reaching 99% of conversion of 2-bromobenzonitrile (entry 9). To our delight, 95% of conversion could be achieved with the unsymmetric NHC supported palladium catalyst, even for the aryl bromide with two *ortho*-methyl substituents [14], which is a very sterically congested substrate. As expected, nitrilo and keto functional groups were found to be compatible under these reaction conditions (entries 9 and 10). In all cases examined, only traces of homocoupling and dehalogenation product were found (1–2%). Unfortunately, attempts to couple 4-chlorotoluene with phenylboronic acid were not successful.

### 2.5. Conclusion

A series of unsymmetric 1,3-disubstituted-imidazolium salts derived from ferrocene were prepared and their preliminary behaviors as precursors of NHC ligands for palladium-catalyzed cross-coupling of aryl bromides with phenylboronic acid were examined. The bulkiness of NHC is an important factor for realizing high activity of its supporting palladium catalyst. Excellent yields (>90%) were achieved for a broad of aryl bromides, ranging from steric congested, inactivated, to nitrilo and keto functionalized substrates, at 1 mol% of loading of $Pd(OAc)_2$/**1d** in the presence $Cs_2CO_3$.

Table 3
$Pd(OAc)_2$/**1d**-catalyzed cross-coupling of aryl bromides with phenylboronic acid[a]




| | ArBr | Product | Time | Yield[b,c] (%) |
|---|---|---|---|---|
| 1 |  |  | 2 | 99 (97) |
| 2 |  |  | 3 | 96 (91) |
| 3 |  |  | 9 | 96 (92) |
| 4 |  |  | 2 | 99 (94) |
| 5 |  |  | 2 | 97 (93) |
| 6 |  |  | 6 | 98 (93) |
| 7 |  |  | 6 | 98 (93) |
| 8 |  |  | 16 | 95 (90) |
| 9 |  |  | 9 | 99 (94) |
| 10 |  |  | 1 | 99 (97) |
| 11 |  |  | 1 | 99 (96) |
| 12 |  |  | 9 | 99 (92) |

[a] Reaction conditions: 1.0 mmol of ArBr; 1.5 mmol of phenylboronic acid; 2.0 mmol of $Cs_2CO_3$; 3.0 mL of dioxane; 110 °C (oil bath).
[b] GC yields against a calibrated internal standard (undecane). Isolated yields in parentheses.
[c] 1–2% of biphenyl was observed.

## 3. Experimental

Unless stated otherwise, all reactions were carried out using standard Schlenk-type glassware under an atmosphere of nitrogen. Solvents were dried and distilled according to the standard methods. Reagents were used as received. 1-(2,6-Diisopropylphenyl)imidazole [25], 1-(2,4,6-trimethylphenyl) imidazole [25] 1-*tert*-butylimidazole [25], 1-cyclohexy-

limidazole [26], 1-(1-admantyl)imidazole [27], and ferrocenylethyl acetate [19] were synthesized according to literature methods. Flash column chromatography was performed on silica gel (200–300 mesh). $^1$H NMR and $^{13}$C NMR nuclear magnetic resonance spectra were recorded on a Varian-400 spectrometer in $CDCl_3$.

### 3.1. Synthesis of 1-(ferrocenylethyl)-3-substituted-imidazolium salt iodides

General procedure: ferrocenylethyl acetate (0.28 g, 1.0 mmol) and the corresponding substituted imidazole (1.2 mmol) were stirred in 10 mL of $CH_3CN$ and 5 mL of $H_2O$ for 24 h at 23 °C. Volatiles were evaporated, and a solution of NaI (0.76 g, 5.0 mmol) in 20 mL of absolute ethanol was added. After 24 h, the solvent was evaporated. The residue was dissolved in $CH_2Cl_2$ and filtered through celite, and purified by flash column chromatography ($CH_2Cl_2$/MeOH, 96:4).

#### 3.1.1. Synthesis of 1-(2,6-diisopropylphenyl)-3-ferrocenylethylimidazolium iodide (**1a**)

Yield: 72%. $^1$H NMR ($CDCl_3$, 400 MHz, TMS): $\delta$ 1.00–1.30 (12H, m), 2.08 (3H, d, $J$ = 6.0 Hz), 2.10–2.30 (2H, m), 4.20–4.60 (9H, m), 6.70–6.75 (1H, m), 7.14 (1H, s), 7.30 (2H, d, $J$ = 4.4 Hz), 7.52 (1H, t, $J$ = 7.6 Hz), 7.78 (1H, s), 9.82 (1H, s); $^{13}$C NMR ($CDCl_3$, 100 MHz): $\delta$ 21.3, 24.0, 24.1, 24.3, 24.4, 24.5, 24.6, 28.2, 28.7, 56.9, 65.8, 68.8, 68.9, 69.6, 69.9, 85.0, 121.2, 124.0, 124.2, 124.7, 130.1, 130.5, 131.9, 135.9, 145.2 [19].

#### 3.1.2. Synthesis of 1-(2,4,6-trimethylphenyl)-3-ferrocenylethylimidazolium iodide (**1b**)

Yield: 83%. $^1$H NMR ($CDCl_3$, 400 MHz, TMS): $\delta$ 2.03 (9H, s), 2.32 (3H, d, $J$ = 6.0 Hz), 4.25–4.55 (9H, m), 6.54–6.62 (1H, m), 6.97 (2H, s), 7.19 (1H, s), 7.65 (1H, s), 9.88 (1H, s); $^{13}$C NMR ($CDCl_3$, 100 MHz): $\delta$ 17.8, 21.1, 21.4, 56.9, 66.0, 68.8, 68.9, 69.5, 69.9, 85.0, 121.0, 123.4, 129.9, 130.6, 134.1, 135.6, 141.3.

#### 3.1.3. Synthesis of 1-(tert-butyl)-3-ferrocenylethylimidazolium iodide (**1c**)

Yield: 88%. $^1$H NMR ($CDCl_3$, 400 MHz, TMS): $\delta$ 1.74 (9H, s), 2.00 (3H, d, $J$ = 6.4 Hz), 4.25–4.55 (9H, m), 6.0–6.30 (1H, m), 7.17 (1H, s), 7.39 (1H, s), 10.3 (1H, s); $^{13}$C NMR ($CDCl_3$, 100 MHz): $\delta$ 21.6, 30.4, 30.6, 56.5, 60.7, 66.1, 68.8, 69.1, 69.5, 69.7, 85.3, 119.4, 119.8, 134.0.

#### 3.1.4. Synthesis of 1-(1′-admantyl)-3-ferrocenylethylimidazolium iodide (**1d**)

Yield: 94%. $^1$H NMR ($CDCl_3$, 400 MHz, TMS): $\delta$ 1.75–2.35 (16H, m), 1.98 (3H, d, $J$ = 6.8 Hz), 4.20–4.55 (9H, m), 6.30–6.40 (1H, m), 7.16 (1H, s), 7.39 (1H, s), 10.3 (s, 1H); $^{13}$C NMR ($CDCl_3$, 100 MHz): $\delta$ 21.6, 29.4, 35.3, 35.5, 43.0, 56.4, 60.8, 66.1, 68.7, 69.1, 69.5, 69.7, 85.4, 118.4, 119.5, 133.4.

#### 3.1.5. Synthesis of 1-(cyclohexyl)-3-ferrocenylethylimidazolium iodide (**1e**)

Yield: 70%. $^1$H NMR ($CDCl_3$, 400 MHz, TMS): $\delta$ 1.20–2.20 (11H, m), 1.92 (3H, d, $J$ = 6.8 Hz), 4.10–4.40 (9H, m), 5.95–6.05 (1H, m), 7.14 (1H, s), 7.32 (1H, s), 10.1 (1H, s); $^{13}$C NMR ($CDCl_3$, 100 MHz): $\delta$ 21.5, 24.5, 24.8, 33.5, 56.5, 60.0, 66.1, 68.8, 68.9, 69.4, 69.6, 85.2, 119.9, 120.1, 133.8.

### 3.2. General protocol used for Suzuki–Miyaura cross-coupling reaction with $Pd(OAc)_2$

Under an atmosphere of nitrogen, a Schlenk tube was charged with 1,4-dioxane (3.0 mL), 1.0 mol% $Pd(OAc)_2$ (4.6 mg, 0.01 mmol), 1.0 mol% **1** and base (2.0 mmol). After 30 min at 80 °C, the reaction mixture was cooled to room temperature and the aryl halide (1.0 mmol), phenylboronic acid (1.5 mmol) were added in turn. The Schlenk tube was placed in 110 °C oil bath and stirred. The reaction was monitored by GLC. The mixture was filtered through a pad of Celite, and the solvent was removed. The residue was purified by flash chromatography (petroleum ether/ethyl acetate, 15:1).

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