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Anionic amido/carbocyclic carbene ligated palladium (II) complex for room temperature Suzuki reaction

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ABSTRACT

The synthesis of anionic amido/carbocyclic carbene ligated Pd (II) complex has been reported. The palladium complex is an active catalyst for the cross-coupling reaction between aryl bromides and phenylboronic acids under phosphine-free conditions at room temperature.

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1. Introduction

Palladium catalyzed cross-coupling reactions represent an extremely versatile tool in organic synthesis [1–3]. The formation of new bonds between two carbon atoms are often key steps in a wide range of organic processes ranging from supramolecular chemistry [4,5] to natural product synthesis [6–9] and therefore various research groups around the world are involved in broadening the new methodologies that are able to improve the reaction conditions. The great significance of palladium-catalyzed C-C coupling reactions has been recognized by the 2010 Nobel laureates Heck, Negishi, and Suzuki. Among these, the Suzuki reaction involving the coupling of an aryl halide with an organoboron reagent is one of the most important reactions in academic and industry as well. This is because of several advantages associated with Suzuki reactions such as air/moisture stable and availability of boron reagents, functional group tolerance, low toxicity and high thermal stability. However, for the success of coupling reactions, the selection of highly active and stable palladium catalyst is primary requirement/highly desirable. In this context the design of new ligands and their Pd-complexes that can catalyze the Suzuki-Miyaura reaction of less reactive aryl bromides and aryl chlorides with both high activity and high efficiency is a current important topic of research. Phosphine-based ligands are well documented as highly efficient and active ligands to catalyze Suzuki–Miyaura reaction. However they are often toxic, air sensitive or quite expensive. Therefore, the development of phosphine-free Pd catalysis has become another equally important topic of research [10–12]. In recent years, *N*-heterocyclic carbenes (NHCs) [13,14] have become a paradigmatically new generation of strong sigma donor phosphine-free ligands in palladium catalyzed C–C coupling reactions. Among the class of *N*heterocyclic carbene ligands, nucleophilic carbocyclic carbenes are less known and recently used in catalytic reactions. We herein report the synthesis of new anionic amido/carbocyclic carbene ligated Pd (II) complex and its catalytic activity in Suzuki–Miyaura reaction at room temperature.

2. Results and discussion

N-Heterocyclic carbene (NHC) ligands have important advantages over the tertiary phosphine ligands because of their ability to tune the electronic properties of metals, straightforward and convenient synthesis, less toxic and stable to air/moisture. Among the diaminocarbenes and other heterocyclic carbenes [15], nucleophilic carbocyclic carbene ligands are less explored in the catalysis [16– 19]. Recently Yao et al. reported the applications of nucleophilic carbocyclic carbene ligands in palladium catalyzed Heck reaction of aryl halides [20]. In our previous study, it has been observed that





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Scheme 1. Design of multi-functional Pd (II) complex.

the anionic amide (deprotonated amide) ligands strongly donate electrons to the metal center, thus stabilizing the various oxidation states of the metals [21-25]. We envisaged the possibility by designing the anionic amide group appended to nucleophilic carbocyclic carbene ligand, as a multi-functional and multi-donor ligand for the palladium catalyzed room temperature Suzuki reaction. As depicted in Scheme 1, we assumed that the presence of anionic carboxylates and alkylamine could enhance the activity of Pd toward oxidative addition and carbocyclic anionic carbene ligands derived from aromatic nitrones stabilized with cationic group such as an iminium group could serve as a neutral donor to palladium center [20] to afford additional stability to the resultant palladium complex. Thus, we report here our efforts toward the realization of this concept using simple and high yielding reaction of 3-formyl benzoic acid and amino alcohol, and subsequent reaction with hydroxyl amine acetate in dichloromethane (DCM) (Scheme 2).

The ligand **1** was prepared by coupling of 3-formyl benzoic acid and 2-amino-3-phenylpropan-1-ol in dry dichloromethane in presence of 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI. HCl), 1-hydroxybenzotriazole hydrate (HOBT) and ethyldiisopropylamine (DIPEA) (Scheme 2). The ligand **2** was prepared in quantitative yield by simple addition of *N*-tertiary butyl hydroxyl amine acetate and triethylamine to the solution of ligand **1** in dry dichloromethane at room temperature. The complexation of palladium acetate with the ligand **2** was observed in the presence of LiOH·H₂O in THF at room temperature to afford air and moisture stable yellow color complex **3** in 70% yield.

The solubility of complex **3** was tested in various solvents however the reasonable solubility was observed only in highly polar solvents such as CH₃OH, DMSO, and DMF. Complex **3** was fully Table 1

Suzuki reaction between bromoanisole and phenylboronic acid.^a



Entry	Base	Solvent	Time (h)	Yield (%) ^b
1	K ₂ CO ₃	MeOH	10	35
2	Cs ₂ Co ₃	MeOH	10	73
3	LiOH.H ₂ O	MeOH	10	35
4	Ba(OH) ₂ 8H ₂ O	MeOH	10	25
5	DIPEA	MeOH	15	_
6	Na ₂ CO ₃	MeOH	15	30
7	Cs ₂ CO ₃	DMF	15	_
8	Cs ₂ CO ₃	DMSO	15	_
9	Cs ₂ CO ₃	DME	15	_
10	Cs ₂ CO ₃	MeOH	10	82 ^c
11	Cs ₂ CO ₃	MeOH	8	92 ^d
12	Cs ₂ CO ₃	MeOH	10	90 ^e
13	Cs ₂ CO ₃	MeOH	15	55 ^f

^a Reaction conditions: bromoanisole (1 mmol), phenylboronic acid (1.5 mmol), Base (1 mmol), catalyst **3** (0.1 mol%) and Solvent (2 mL) at room temperature.

^b Yield of isolated product.

^c With 0.5 mol% of catalyst **3**

^d With 1 mol% of catalyst **3**.

^e With 0.1 mol% of catalyst **3** at 60 °C.

^f With 0.01 mol% of catalyst **3**.

characterized by various spectroscopic tools (NMR, MS, HRMS, FT-IR). The ¹H NMR and ¹³C NMR study of **2** and **3** clearly distinguished the coordination of carbene and anionic amide to the palladium center in presence of base. Further, high resolution mass confirmed the loss of two mass units from benzene ring and amide group of **2** during the complexation between palladium acetate and **2** to **3** (see Supporting information).

A preliminary testing of complex **3** revealed that this palladium complex is highly efficient catalyst for the Suzuki coupling reaction. In a subsequent optimization using various bases, solvents and reaction parameters (Table 1, entries 1–9), it was observed that the coupling of bromoanisole with phenylboronic acid could be achieved with 73% yield of 4-methoxy-biphenyl (entry 2) in presence of 0.1 mol% of **3**, cesium carbonate as a base



Scheme 2. Synthesis of amido/carbocyclic carbine ligated Pd (II) complex.

Table 2

Suzuki reaction of aryl halides and arylboronic acids.^a



$$X = I, Br$$

Entry	Aryl halide	Arylboronic acid	Product	Time (h)	Yield (%) ^b
1	⟨ − Br	HO ^B H		10	60
2	0- Br	HO ² B	<u> </u>	10	73
3	⟨}−Br	HO ^B		10	62
4	⟨	HO ^B O	-0	10	61
5	⟨ − Br	HO ^B OH	© – Č	10	89 ^c
6	0- Br	HO ^B		10	60
7	O- Br	HO'B	o- <o< td=""><td>10</td><td>55</td></o<>	10	55
8	——————————————————————————————————————	HO' ^B		10	57
9	∑_I	HO ^B		10	75
10	_0−√−I	HO'B	,o-<	10	74
11	∑ı	HO ^{.B}		10	80
12	∑_I	HO'B		10 (contin	65 uued on next page)

Table 2 (continued)

Entry	Aryl halide	Arylboronic acid	Product	Time (h)	Yield (%) ^b
13		HO'B		10	90 ^c
14	`o−(I	HO'B		10	95
15	`0-∕I	HO ^{-B}	_0-	10	90
16	CI	HO'B		10	35 ^c
17	Cl	HO'B		10	30 ^c

^a Reaction conditions: aryl halide (1 mmol), arylboronic acid (1.5 mmol), Cs₂CO₃ (1 mmol), catalyst **3** 0.1 mol% and methanol (2 mL) at room temperature.

^b Yield of isolated product.

^c With 1 mol% of catalyst **3**.

and methanol as a solvent at room temperature. The catalytic activity of complex **3** was tested at different catalyst loadings (entries 10–12) and at high temperature (entry 13). The high catalytic efficiency of the catalyst **3** may be attributed to the anionic nature of carbene stabilized by anionic amide group. The catalytic activity of complex 3 was then extended to various aryl halides and different phenylboronic acids using the entry 2 as the standard reaction conditions and the results are shown in Table 2. In the room temperature Suzuki reaction, aryl bromides and aryl iodides with both electron-rich and electron-deficient substrates were allowed to coupled with various boronic acids using catalyst loading as low as 0.1 mol % (entries 1–15). The resulting coupling products were obtained in moderate to good yields. Interestingly, excellent yields of coupling products are obtained with the reactions of deactivated aryl iodides with different arylboronic acids (entries 8,10,13 and 15). However, the catalyst has limited activity in the reaction of chlorobenzene with phenyl and 4-methylphenylboronic acids even at 1 mol% of catalyst loading (entries16 and 17).

3. Conclusion

We have synthesized anionic amido/carbocyclic carbene ligated Pd (II) complex in high yields from the easily available substrates. The multi-functional and highly stable palladium catalyst was tested for the C–C coupling reaction of deactivated aryl halides with various boronic acids. Low loading palladium catalyst in Suzuki reaction at room temperature affords coupling products in good yields which were accomplished previously either by using complicated phosphine or heterocyclic carbene ligands [26,27], and with relatively high loading of Pd (i.e., >1 mol%) [28,29]. Unlike previously reported catalytic systems for C–C cross-coupling reactions, additional co-catalysts such as [NBu4] Br or [PPh4]Cl are not required to achieve high catalytic activity [30–34].

4. Experimental section

4.1. Synthesis of ligand (1)

A two necked round bottom flask was charged with 3-formyl benzoic acid (1 g, 6.66 mmol) in dry DCM (3 mL), was then added 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (1.91 g, 9.99 mmol) and 1-hydroxybenzotriazole hydrate (1.35 g, 9.99 mmol) under nitrogen atmosphere at 0 °C. The reaction mixture was allowed to stir for 10 min and was added amino alcohol (1.2 g 8.00 mmol), ethyldiisopropylamine (DIPEA) (2.8 mL, 16.65 mmol) at the same temperature. After stirring for 2 h at room temperature, the crude product was obtained and then was washed with water, brine and extracted with ethyl acetate. The organic layer was dried over sodium sulfate, concentrated under vacuum, purified by column chromatography.

¹H NMR (300 MHz, CDCl₃, TMS) δ 2.99 (d, J = 6.798 Hz, 2H), 3.31 (br, 1H), 3.65–3.70 (dd, J = 3.022 Hz, 1H), 3.75–3.80 (dd, J = 5.288 Hz, 1H), 4.35–4.43 (m, 1H, CH), 6.98 (d, J = 8.309 Hz, 1H, CONH), 7.18–7.31 (m, 5H), 7.47–7.52 (t, J = 7.554 Hz, 1H), 7.89–7.96 (m, 2H), 8.129 (s, 1H), 9.924 (s, 1H); ¹³C NMR (300 MHz, CDCl₃) δ 36.67, 53.18, 62.92, 126.21, 127.79, 128.18, 128.94, 129.20, 131.89, 132.76, 134.91, 135.78, 137.59, 166.51, 191.62; IR (KBr): ν bar = 3334 cm⁻¹ (br, OH), 3063, 3028 cm⁻¹ (aromatic C–H), 1698, 1641 cm⁻¹ (C=O), 1541 cm⁻¹ (C=C), 1208 cm⁻¹ (C–N); ESI-MS: (M + H)⁺ = 284. HRMS (ESI) calcd for C₁₇H₁₇NO₃Na (M + Na)⁺: 306.1106; found: 306.1105.

4.2. Synthesis of ligand (2)

A two necked round bottomed flask was charged with **1** (0.8 g, 2.82 mmol) in dry DCM (3 mL), *N*-tertiary butyl hydroxyl amine acetate (3.39 mmol) and Et₃N (5.65 mmol) at room temperature. The reaction mixture was allowed to stir for 12 h at the same temperature. A green color crude product was obtained and it was then washed with water, brine and extracted with ethyl acetate.

The organic layer was dried over anhydrous sodium sulfate, concentrated under vacuum, purified by column chromatography.

¹H NMR (500 MHz, CDCl₃, TMS) δ 1.55 (s, 9H), 2.84–2.89 (dd, 1H), 2.95–2.99 (dd, 1H), 3.57–3.60 (dd, 1H), 3.69–3.71 (dd, 1H), 4.25 (m, 1H), 7.13 (d, 1H, CONH), 7.20–7.31 (m, 5H), 7.40–7.41 (d, *J* = 7.919 Hz, 1H), 7.542 (s, 1H), 7.66–7.67 (d, *J* = 6.929 Hz, 1H), 7.88–7.89 (d, *J* = 6.929 Hz, 1H), 8.91 (s, 1H); ¹³C NMR (300 MHz, CDCl₃) δ 28.15, 37.13, 53.63, 63.32, 71.17, 126.39, 128.46, 128.56, 129.30, 129.47, 130.23, 130.61, 131.89, 134.57, 138.03, 167.15; IR (KBr): ν bar = 3329 cm⁻¹ (br, OH), 3065 cm⁻¹ (aromatic C–H), 1714, 1643 cm⁻¹(C=O), 1538 cm⁻¹(C=C), 1243 cm⁻¹(C-O), 1192 cm⁻¹(C-N); ESI-MS: (M + Na)⁺ = 377; HRMS (ESI) calcd for C₂₁H₂₆N₂O₃Na (M + Na)⁺: 377.1841; found: 377.1838.

4.3. Synthesis of palladium complex (3)

To a round bottom flask containing a stirred suspension of palladium acetate (224.49 mg, 1 mmol) in THF (20 mL) was added **2** (424.8 mg, 1.2 mmol) in one portion followed by LiOH·H₂O (151.2 mg, 3.6 mmol). Upon stirring the solution for 12 h at room temperature, pale yellow precipitation was obtained. The reaction mixture was filtered, crude solid was washed with excess THF, water and finally with methanol to obtain the pure complex in 70% yield.

 ^{1}H NMR (300 MHz, CDCl_3+CD_3OD, TMS) δ 1.650 (s, 9H), 2.95–3.11 (m, 2H), 3.35 (m, 2H), 3.69–3.73 (m, 1H), 7.17–7.47 (m, 8H), 8.019 (s, 1H); ^{13}C NMR (300 MHz, CDCl_3) δ 27.99, 36.81, 57.10, 65.98, 70.84, 72.57, 123.35, 124.74, 125.59, 127.97, 128.64, 129.12, 131.12, 137.18, 139.34, 147.62; IR (KBr): ν bar = 3416 cm $^{-1}$ (br, OH), 1577 cm $^{-1}$ (C=O), 1518 cm $^{-1}$ (C=C), 1119 cm $^{-1}$ (C–N); ESI-MS: (M)+ = 459; HRMS (ESI) calcd for C₂₁H₂₄N₂O₃NaPd (M + Na)+: 481.0719; found: 481.0699.

4.4. General procedure for the Suzuki reaction of aryl halides

The reaction vessel was charged with aryl halide (1 mmol), boronic acid (1.5 mmol), cesium carbonate (1 mmol) and the catalyst **3** (0.1 mol%) in methanol (2 mL). The reaction mixture was stirred at room temperature and the progress of reaction was monitored by TLC. After completion of the reaction, methanol was removed under reduced pressure and the reaction mixture was diluted with EtOAc (20 mL), washed with 1 N aq. HCl and water. The combined organic phase was dried over anhydrous Na₂SO₄. After removal of the solvent, the residue was subjected to column chromatography on silica gel using ethyl acetate and hexane mixtures to afford the Suzuki product.

4.5. Analytical data for the products of the Suzuki reaction

Biphenyl (Table 2, entry 1): ¹H NMR (300 MHz, CDCl₃, TMS) $\delta = 7.29 - 7.31$ (m, 2H), 7.39 (t, J = 7.74 Hz, 4H), 7.54 (d, J = 8.49 Hz, 4H); ¹³C NMR (300 MHz, CDCl₃) $\delta = 96.19$, 127.18, 128.73, 141.30.

4-*Methoxybiphenyl* (Table 2, entry 2): ¹H NMR (300 MHz, CDCl₃ + CD₃OD, TMS) δ = 3.83 (s, 3H), 6.92 (d, *J* = 9.0 Hz, 2H), 7.24 (m, 1H), 7.35 (t *J* = 8.0 Hz, 2H), 7.48–7.50 (m, 4H); ¹³C NMR (300 MHz, CDCl₃) δ = 55.24, 114.14, 126.59, 126.71, 128.10, 128.70, 133.78, 140.86, 159.09.

4-*Methylbiphenyl* (Table 2, entry 3): ¹H NMR (300 MHz, CDCl₃ + CD₃OD, TMS) δ = 2.39 (s, 3H), 7.23–7.26 (m, 2H), 7.29–7.34 (m, 1H), 7.39–7.44 (t, *J* = 8.3 Hz, 2H), 7.50 (d, *J* = 8.31 Hz, 2H), 7.58 (d, *J* = 6.79 Hz, 2H); ¹³C NMR (300 MHz, CDCl₃) δ = 21.07, 126.93, 128.67, 129.44, 136.96, 138.30, 141.1.

4-*Methoxy*-4'-*methyl-biphenyl* (Table 2, entry 6): ¹H NMR (500 MHz, CDCl₃ + CD₃OD, TMS) δ = 2.37 (s, 3H), 3.82 (s, 3H), 6.90 (d *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.38 (d *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (300 MHz, CDCl₃) δ = 21.20, 55.09, 114.15, 126.63, 127.94, 129.42, 133.82, 135.97, 138.18, 158.95. 4,4'-Dimethylbiphenyl (Table 2, entry 8); ¹H NMR (500 MHz, CDCl₃ + CD₃OD, TMS) δ = 2.38 (s, 6H), 7.22 (d, *J* = 8.0 Hz, 4H), 7.46 (d, *J* = 7.0 Hz, 4H); ¹³C NMR (300 MHz, CDCl₃) δ = 21.06, 126.77, 129.40, 136.67, 138.24.

4,4'-Dimethoxybiphenyl (Table 2, entry 7): ¹H NMR (500 MHz, CDCl₃ + CD₃OD, TMS) δ = 3.83 (s, 6H), 6.93 (d, *J* = 8.84 Hz, 4H), 7.44 (d, *J* = 8.84 Hz, 4H); ¹³C NMR (300 MHz, CDCl₃) δ = 55.36, 114.18, 127.73, 133.49, 158.69.

4-Acetylbiphenyl (Table 2, entry 13): ¹H NMR (300 MHz, CDCl₃ + CD₃OD, TMS) δ = 2.61 (s, 3H), 7.34–7.47 (m, 3H), 7.57–7.67 (m, 4H), 8.00 (d, *J* = 8.30 Hz, 2H); ¹³C NMR (300 MHz, CDCl₃) δ = 26.57, 127.16, 127.21, 128.17, 128.85, 128.89, 135.83, 139.83, 145.72, 197.63.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jorganchem.2013.01.013.

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