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New Nickel-Based Catalytic System with Pincer Pyrrole-Functionalized N-Heterocyclic Carbene as Ligand for Suzuki-Miyaura Cross-Coupling Reactions

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ABSTRACT

A new catalytic system with $Ni(NO_3)_2{\cdot}6H_2O$ as the catalyst and a pincer pyrrole-functionalized N-heterocyclic carbene as the ligand was employed in the Suzuki-Miyaura cross-coupling reactions of aryl iodides with arylboronic acids. With 5 mol% catalyst, the catalytic reactions proceeded at 160 °C, giving coupling products in isolated yields of up to 94% in short reaction times (1-4 h). The system worked efficiently with aryl iodides bearing electron-donating or electron-withdrawing groups and arylboronic acids with electron-donating groups. Steric effects were observed for both aryl iodides and arylboronic acids. It is proposed that the reactions underwent a Ni(I)/Ni(III) catalytic cycle.

Introduction

Transition metal-catalyzed cross-coupling reactions have become one of the most important methods for the formation of carbon-carbon and carbon-heteroatom bonds. Recently, nickel catalysis has been applied in a wide variety of cross-coupling reactions, including Suzuki-Miyaura, Kumada, Hiyama, Negishi, and Stille couplings [1–4]. Among them, the Suzuki- Miyaura coupling has become one of the most effective reactions for the construction of carbon−carbon bonds [5–12]. Although the Ni-catalyzed Suzuki cross-coupling reactions have been greatly promoted, there are still limitations on the scope of substrates and relevant functional groups [13]. Development of new ligands might provide solutions to overcome these limitations. N-heterocyclic carbenes (NHCs), which are electron-rich and behave like typical σ-donor ligands, have found broad applications in coordination chemistry and homogeneous catalysis [14,15]. Particularly, the introduction of NHCs in the second-generation Grubbs catalysts has boosted ruthenium-catalyzed metathesis and related reactions [16–20]. Multiple examples have also been reported on their application as ligands in nickel catalysis [21–24]. More recently, nickel-based catalytic systems with pincer ligands have attracted significant attention as efficient catalysts for cross-coupling reactions [25–28]. Herein, we reported Ni(II)-catalyzed Suzuki-Miyaura cross-coupling reactions with a pincer pyrrole-functionalized NHC (**L1** in Scheme 1), which can be formed *in situ* from bis(imidazolium) (**Pre-L1**) in the presence of base, as the ligand. To our best knowledge, this is the first employment of a CNC-type pincer ligand with two NHC donors and one anionic N-donor bridge in nickel catalysis.

Experimental

Materials and methods

Pre-ligand **Pre-L1** was prepared according to literature procedures [29]. Bases were ground into powder and dried under vacuum at 125°C for 4 h prior to use. Toluene was refluxed over Na/benzophenone and distilled under nitrogen. All other reagents and solvents were purchased from commercial suppliers and used as received. All glasswares were dried in an oven at 120°C for 8 h and cooled under nitrogen atmosphere in a desiccator before use. An Agilent 6890N Network Gas Chromatograph, equipped with a capillary column (HP-5) and a TCD detector, was used to assay the reaction yields for the optimization of reaction conditions. Column chromatography was performed with 300–400 mesh silica gel. NMR spectra were recorded on a Bruker Avance 400 MHz spectrometer in $CDCl_3$ and referenced to the residual solvent signals (^{1}H: 7.26 ppm; ^{13}C: 77.16 ppm). Peaks were characterized as singlet (s), doublet (d), triplet (t), quartet (q), doublet of doublet (dd), and multiplet (m). Chemical shifts were expressed in ppm and *J* values were given in Hz.

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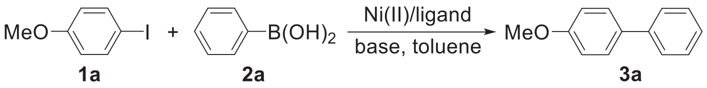
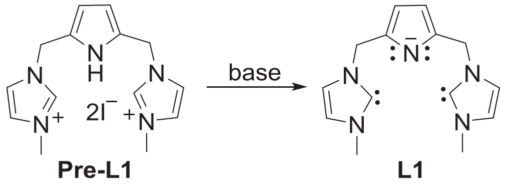
Table 1
Optimization of reaction conditions[a].



Entry	Ligand	Catalyst	Base	Time (h)	Yield (%)[b]
1	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	K_3PO_4	4	99
2	**L2**	$Ni(NO_3)_2{\cdot}6H_2O$	K_3PO_4	4	5
3	**L3**	$Ni(NO_3)_2{\cdot}6H_2O$	K_3PO_4	4	4
4	**L1**	$Ni(Ph_3P)_2Cl_2$	K_3PO_4	4	42
5	**L1**	$Ni(OAc)_2$	K_3PO_4	4	57
6	**L1**	NiI_2	K_3PO_4	4	98
7	**L1**	$NiBr_2$	K_3PO_4	4	92
8	**L1**	$NiCl_2{\cdot}6H_2O$	K_3PO_4	4	94
9	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	K_3PO_4	4	65[c]
10	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	K_3PO_4	1	99
11	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	Cs_2CO_3	1	11
12	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	CsF	1	30
13	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	K_2CO_3	1	67
14	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	K_3PO_4	2	81[d]
15	**L1**	$Ni(NO_3)_2{\cdot}6H_2O$	K_3PO_4	2	3[e]

[a] Reaction conditions: **1a** (1.0 mmol), **2a** (2.0 mmol), Ni(II) (0.05 mmol), ligand (0.05 mmol; **Pre-L1** was used to form **L1** *in situ*), base (3.0 mmol), toluene (3 mL), 160 °C.
[b] GC yield using hexadecane as the internal standard.
[c] **2a** (1.2 mmol).
[d] 1-Bromo-4-methoxybenzene.
[e] 1-Chloro-4-methoxybenzene.



Scheme 1. *In situ* formation of pincer NHC ligand (**L1**) from bis(imidazolium) (**Pre-L1**)

General procedure for the Suzuki-Miyaura cross-coupling reactions

Aryl iodide (1.0 mmol), arylboronic acid (2.0 mmol), $Ni(NO_3)_2{\cdot}6H_2O$ (0.05 mmol), pre-ligand **Pre-L1** (0.05 mmol), base (3.0 mmol), and toluene (3 mL) were added to a glass tube, which was then sealed with a PTFE cap. After the reaction mixture was stirred vigorously at room temperature for 5 min, the sealed glass tube with the reaction mixture was placed in a Radleys Carousel 12 Plus Reaction Station, which was preheated to the described temperature. After the reaction was stirred for the required time and then cooled down to room temperature, water (10 mL) was added to the reaction mixture. The resulting mixture was extracted with ethyl acetate (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated to dryness. The remaining residue was analyzed by GC or purified by flash chromatography on silica gel with ethyl acetate-hexanes (0-20% ethyl acetate in hexanes).

Results and Discussion

Optimization of reaction conditions

As shown in Scheme 1, ligand **L1** can be generated *in situ* by deprotonation of the corresponding bis(imidazolium) (**Pre-L1**). **Pre-L1** was prepared according to literature procedures [29]. With the coupling of 4-iodoanisole (**1a**) with phenylboronic acid (**2a**) as the model reaction (Table 1), we first compared the efficiency of **L1** with 2,2'-bipyridine (**L2**) and 1,10-phenanthroline (**L3**), both of which are commercially available and often used as ligand in Ni-catalyzed coupling reactions [30]. The results show that under the same reaction conditions, ligand **L1** is much more efficient than **L2** and **L3** (Table 1, entries 1-3). Among the six nickel (II) salts that were screened, $Ni(Ph_3P)_2Cl_2$ and $Ni(OAc)_2$ gave moderate yields (entries 4 and 5), and all other four gave excellent yields (>90%, entries 1 and 6-8). Salt $Ni(NO_3)_2{\cdot}6H_2O$ was chosen for further study due to its highest yield and lower cost. A decrease of the amount of **2a** from 2 eq. to 1.2 eq. resulted in much lower yield (entries 1 and 9). Using TLC to monitor the reaction, it was found that the same reaction was complete in 1 h, resulting in a shorter reaction time (entry 10 vs. entry 1). The studies on the effect of base on the coupling reaction showed that K_3PO_4 was the most efficient base (entry 10) while other bases such as Cs_2CO_3, CsF, and K_2CO_3 gave poor to moderate yields (entries 11-13). The efficiency of the base in the reaction is determined by multiple factors including its basicity, its solubility in the solvent, and the affinity of its metal cation for the iodide ion. The decrease in yields using Cs_2CO_3, CsF, or K_2CO_3 as base is probably due to one or more of these factors. Furthermore, the results showed that under the same reaction conditions, the reactivity of aryl halides in this coupling reaction follows the sequence of iodides > bromides >> chlorides (entries 10, 14 and 15), which is consistent with the recently reported results that were obtained with a PCP-type Ni(II) pincer complex [25]. The higher reactivity of this catalytic system towards aryl iodides than aryl chlorides is probably due to its different catalytic cycle from those we previously reported [31–33]. Finally, the optimal reaction conditions for the coupling reaction were set as entry 10 in Table 1.

Scope of substrates

With the optimal reaction conditions, we investigated the scope of aryl iodides by examining the coupling of phenylboronic acid (**2a**) with a variety of aryl iodides (**1**). As shown in Table 2, substrates with electron-donating groups such as methoxy and methyl groups gave excellent yields (entries 1-3), while substrates with electron-withdrawing groups such as chloro, fluoro, trifluoromethyl, ester, and acyl groups gave moderate to good yields (entries 6-16). Steric effects were observed as ortho-substituted iodides generally gave lower yields than their para-substituted isomers (entries 1 and 5, 2 and 4, respectively). This reaction also

Table 2
Scope of aryl iodides[a].

$$\underset{\mathbf{1}}{\text{Ar–I}} + \underset{\mathbf{2a}}{\text{Ph–B(OH)}_2} \xrightarrow[\text{toluene, 160 }^{o}\text{C}]{\text{Ni(NO}_3)_2\cdot 6\text{H}_2\text{O, }\mathbf{Pre\text{-}L1}\text{, K}_3\text{PO}_4} \underset{\mathbf{3}}{\text{Ar–Ph}}$$

Entry	Ar-I	Product	Time (h)	Yield (%)[b]
1	*p*-MeO-Ph-I (**1a**)	*p*-MeO-Ph-Ph (**3a**)	1	94
2	*p*-Me-Ph-I (**1b**)	*p*-Me-Ph-Ph (**3b**)	2	90
3	*m*-Me-Ph-I (**1c**)	*m*-Me-Ph-Ph (**3c**)	2	90
4	*o*-Me-Ph-I (**1d**)	*o*-Me-Ph-Ph (**3d**)	2	30
5	*o*-MeO-Ph-I (**1e**)	*o*-MeO-Ph-Ph (**3e**)	2	48
6	*p*-Cl-Ph-I (**1f**)	*p*-Cl-Ph-Ph (**3f**)	2	84
7	*p*-F-Ph-I (**1g**)	*p*-F-Ph-Ph (**3g**)	2	70
8	*p*-CF$_3$-Ph-I (**1h**)	*p*-CF$_3$-Ph-Ph (**3h**)	2	84
9	*m*-CF$_3$-Ph-I (**1i**)	*m*-CF$_3$-Ph-Ph (**3i**)	2	78
10	*o*-CF$_3$-Ph-I (**1j**)	*o*-CF$_3$-Ph-Ph (**3j**)	2	32
11	*p*-EtOOC-Ph-I (**1k**)	*p*-EtOOC-Ph-Ph (**3k**)	2	88
12	*o*-MeOOC-Ph-I (**1l**)	*o*-MeOOC-Ph-Ph (**3l**)	2	50
13	*p*-OHC-Ph-I (**1m**)	*p*-OHC-Ph-Ph (**3m**)	4	76
14	*p*-MeOC-Ph-I (**1n**)	*p*-MeOC-Ph-Ph (**3n**)	2	68
15	*p*-PhOC-Ph-I (**1o**)	*p*-PhOC-Ph-Ph (**3o**)	4	88
16	*o*-PhOC-Ph-I (**1p**)	*o*-PhOC-Ph-Ph (**3p**)	4	66
17	4-Iodopyridine (**1q**)	Pyridine-4-Ph-*p*-Me (**3q**)	2	71[c]
18	3-Iodopyridine (**1r**)	Pyridine-3-Ph-*p*-Me (**3r**)	2	38[c]
19	2-Iodopyridine (**1s**)	Pyridine-2-Ph-*p*-Me (**3s**)	2	54[c]
20	*p*-Ph-Ph-I (**1t**)	*p*-Ph-Ph-Ph (**3t**)	1	50
21	*m*-I-Ph-I (**1u**)	*m*-Ph-Ph-Ph (**3u**)	2	41[d]
22	2,6-Bis(I-CH$_2$)pyridine (**1v**)	2,6-Bis(Ph-CH$_2$)pyridine (**3v**)	4	22[d]
23	*m*-MeOOC-Ph-CH$_2$I (**1w**)	*m*-MeOOC-Ph-CH$_2$-Ph-*p*-Me (**3w**)	2	21[c]

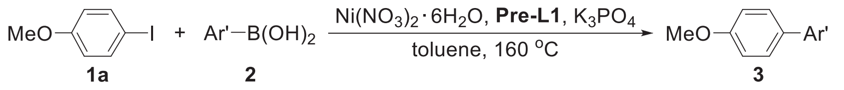
[a] Reaction conditions: **1** (1.0 mmol), **2a** (2.0 mmol), Ni(NO$_3$)$_2$·6H$_2$O (0.05 mmol), **Pre-L1** (0.05 mmol), K$_3$PO$_4$ (3.0 mmol), toluene (3 mL), 160 °C, Ph = C$_6$H$_5$ or C$_6$H$_4$ where appropriate.

[b] Isolated yields.

[c] *p*-MePhB(OH)$_2$ (2.0 mmol).

[d] **2a** (4.0 mmol), Ni(NO$_3$)$_2$·6H$_2$O (1.0 mmol), **Pre-L1** (1.0 mmol), K$_3$PO$_4$ (6.0 mmol), toluene (6 mL).

Table 3
Scope of arylboronic acids[a].



Entry	Ar'B(OH)$_2$	Product	Time (h)	Yield (%)[b]
1	*p*-MeO-PhB(OH)$_2$ (2b)	*p*-MeO-Ph-Ph-*p*-OMe (3x)	1	92
2	*p*-Me-PhB(OH)$_2$ (2c)	*p*-Me-Ph-Ph-*p*-OMe (3y)	1	92
3	*m*-Me-PhB(OH)$_2$ (2d)	*m*-Me-Ph-Ph-*p*-OMe (3z)	2	84
4	*o*-Me-PhB(OH)$_2$ (2e)	*o*-Me-Ph-Ph-*p*-OMe (3a’)	2	45
5	1-B(OH)$_2$-naphthalene (2f)	1-(*p*-MeO-Ph)-naphthalene (3b’)	1	92
6	2-B(OH)$_2$-naphthalene (2g)	2-(*p*-MeO-Ph)-naphthalene (3c’)	1	87
7	*p*-Cl-PhB(OH)$_2$ (2h)	*p*-Cl-Ph-Ph-*p*-OMe (3d’)	3	51
8	*p*-F-PhB(OH)$_2$ (2i)	*p*-F-Ph-Ph-*p*-OMe (3e’)	3	50
9	*p*-MeOOC-PhB(OH)$_2$ (2j)	*p*-MeOOC-Ph-Ph-*p*-OMe (3f’)	4	5
10	*m*-MeOOC-PhB(OH)$_2$ (2k)	*m*-MeOOC-Ph-Ph-*p*-OMe (3g’)	4	8
11	2-B(OH)$_2$-furan (2l)	2-(*p*-MeO-Ph)-furan (3h’)	4	21
12	2-B(OH)$_2$-benzofuran (2m)	2-(*p*-MeO-Ph)-benzofuran (3i’)	4	23
13	(*E*)-Styrylboronic acid (2n)	(*E*)-1-Methoxy-4-styrylbenzene (3j’)	4	17

[a] Reaction conditions: **1a** (1.0 mmol), **2** (2.0 mmol), Ni(NO$_3$)$_2$·6H$_2$O (0.05 mmol), **Pre-L1** (0.05 mmol), K$_3$PO$_4$ (3.0 mmol), toluene (3 mL), 160 °C, Ph = C$_6$H$_5$ or C$_6$H$_4$ where appropriate.

[b] Isolated yields.

works for heteroaromatic iodides (entries 17-19), giving moderate yields. The formation of *p*-terphenyl (**3t**) and *m*-terphenyl (**3u**) indicates that this catalytic protocol is efficient for the preparation of polyaryls (entries 20 and 21). Pleasingly, the coupling reaction occurred on benzylic sp^3-hybridized carbons as well, though the yields were low (entries 22 and 23).

Next, we examined the scope of arylboronic acids. As shown in Table 3, phenylboronic acids bearing electron-donating groups (entries 1-3) and naphthylboronic acids (entries 5 and 6) successfully coupled with 4-iodoanisole (**1a**) in good to excellent yields. The presence of electron-withdrawing groups generally deactivates arylboronic acids and resulted in low to moderate yields (entries 7-10). Steric effect was observed as the yields followed the sequence of para > meta > ortho (entries 2-4). This protocol also works for heteroarylboronic acids (entries 11 and 12) and a vinylboronic acid (entry 13) with low yet acceptable yields.

Reaction mechanism

A plausible reaction mechanism is proposed in Fig. 1. Similar to the Suzuki-Miyaura cross-coupling reactions catalyzed by a Ni(II) PCP-type pincer complex [25] and a Ni(II) PNP-type pincer com-

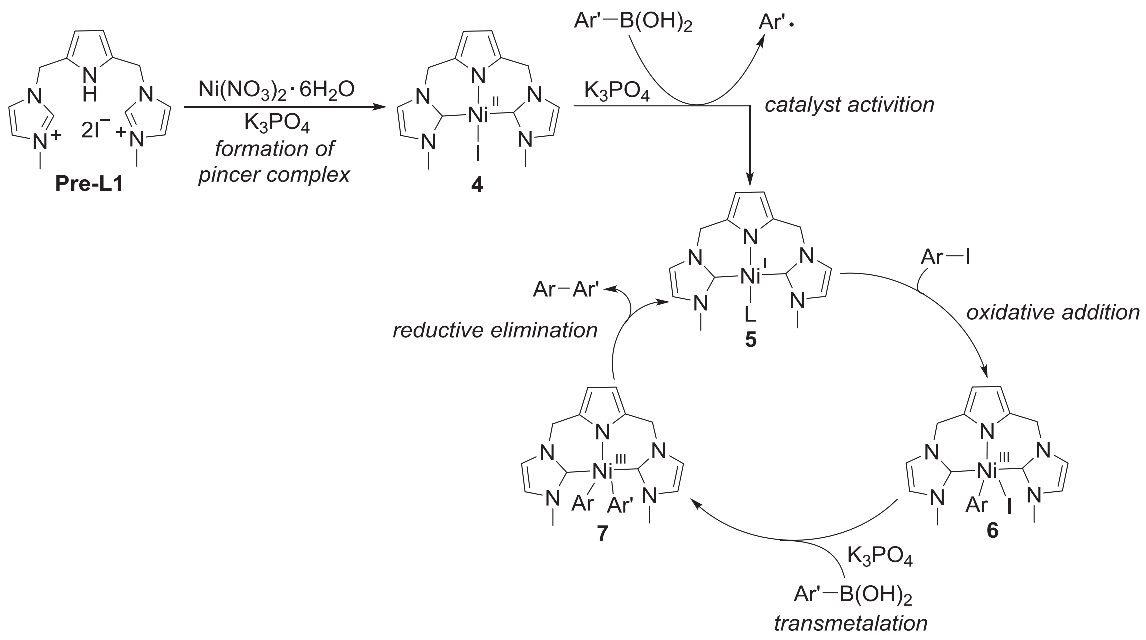


Fig. 1. Proposed reaction mechanism

plex, [26] respectively, it is believed that the reaction system reported here follows a Ni(I)/Ni(III) catalytic cycle. First, in the presence of base under heating, bis(imidazolium) **Pre-L1** reacts via ligand **L1** with $Ni(NO_3)_2$ to form a CNC-type bis(carbene)-containing pincer complex (**4**), which displays a square-planar geometry about nickel [34]. The conversion of complex **4** to the catalytically active Ni(I) species (**5**) is achieved by a single-electron reduction with arylboronic acid (**2**) as the reductant. Oxidative addition of aryl iodide (**1**) to Ni(I) species **5** gives intermediate **6**, which undergoes transmetalation with arylboronic acid (**2**) in the presence of base to produce intermediate **7**. The reductive elimination of intermediate **7** regenerates active Ni(I) species **5** so that the catalytic cycle continues. It is proposed that both the oxidative addition and transmetalation steps also undergo a single-electron pathway. The fact that this reaction system gives best yields for aryl iodides (Table 1, entries 10, 14 and 15) supports a Ni(I)/Ni(III) catalytic cycle because generally, a Ni(0)/Ni(II) catalytic cycle favors aryl chlorides [31–33].

Conclusion

We have developed a new catalytic system with $Ni(NO_3)_2{\cdot}6H_2O$ as the catalyst and a pincer pyrrole-functionalized NHC as the ligand for the Suzuki-Miyaura cross-coupling reactions of aryl iodides with arylboronic acids. For aryl iodides, the catalytic system tolerates both electron-donating and electron-withdrawing groups, while for arylboronic acids, electron-withdrawing groups significantly deactivate substrates, leading to low to moderate yields. Steric effects were observed for both aryl iodides and arylboronic acids. All reactions were complete in a short reaction time of 1-4 h. Similar to two other catalytic systems with nickel(II) pincer complexes, our system is believed to undergo a Ni(I)/Ni(III) catalytic cycle. It is worth noting that all reported reactions were performed without the protection of inert gas.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2021.122068.

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