Highly Efficient Palladacycle/Dihydroimidazolium Chloride System for the Suzuki-Miyaura Cross-Coupling of Aryl Halides (I, Br, Cl) with Arylboronic Acids

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A combination of a tertiary amine-based palladacycle and an *N*-heterocyclic carbene ligand precursor (**1**, *N*,*N*-bis-mesityl-4,5-dihydroimidazolium chloride) has been applied to catalyze the Suzuki-Miyaura cross-coupling of aryl halides with arylboronic acids. The substrate scope is general: a variety of electron rich and deficient aryl halides (I, Br, Cl) and arylboronic acids were found to undergo the cross-coupling reaction in good to excellent yields at low catalyst loading of 0.01—1 mol%.

Keywords Suzuki-Miyaura reaction, *N*-heterocyclic carbine, palladacycle, aryl halides

Introduction

During the last three decades, the Suzuki-Miyaura reaction has been demonstrated as an efficient and versatile method to synthesize dienes, polyenes, styrenes, and biaryls, which are common structural subunits in complex natural products, pharmaceuticals and functional materials.[1] Traditionally, Pd-phosphine complexes have been proven as powerful catalysts for the Suzuki-Miyaura reaction according to numerous reports.[2] However, the air-sensitivity of phosphine ligands poses significant difficulty to the application of these catalysts to certain synthetic efforts.[3] Therefore, it remains active interest in developing new catalysts that provide improved stability and activity.

Since Herrmann reported the first Palladium/*N*-heterocyclic carbene (NHC) complex catalyzed Heck reaction in 1995,[4] NHCs have been shown as another class of highly effective and widely applicable ligands for Pd-based catalyst systems.[5] NHCs are air-stable, low toxic and readily tunable, therefore, they have attractive advantages of easy storage and handling. On the other hand, palladacycles, with air and moisture-stability and high catalytic activity, are also alternative catalysts (precatalysts) proven to be efficient for cross-coupling reactions.[6] Among them, tertiary amine-based palladacycles developed by Bedford and others including our group have been demonstrated as effective precatalysts for the Heck reaction and the Suzuki-Miyaura reaction.[7] However, the combination of a tertiary amine-based palladacycle and an NHC ligand has been rarely explored and only a limited amount of examples have been reported.[8] Herein, we report a novel combination of a tertiary amine-based palladacycle and an NHC precursor (**1**, *N*,*N*-bis-mesityl-4,5-dihydroimi-dazolium chloride) as a highly efficient catalyst system for the Suzuki-Miyaura cross-coupling of aryl halides with arylboronic acids.

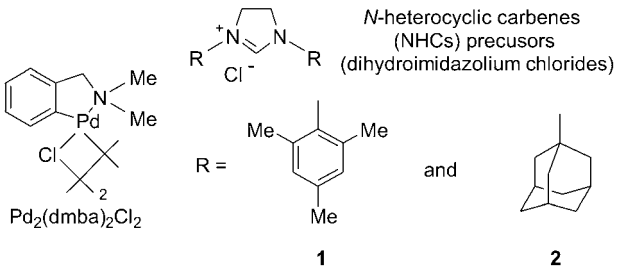

Figure 1 Structures of palladacycle $Pd_2(dmba)_2Cl_2$ and NHC ligands.

Results and discussion

We began our investigation with the cross-coupling of bromobenzene and phenylboronic acid in *N*,*N*-dimethylacetamide (DMA) (Table 1). The easily accessible palladacycle $Pd_2(dmba)_2Cl_2$ and *N*-heterocyclic carbene ligand precursor *N*,*N*-bis-mesityl-4,5-dihydroimidazolium chloride **1** were chosen to *in situ* form the

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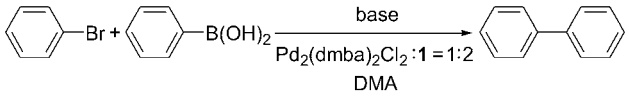
active catalyst under the same reaction condition. The impact of the base on this reaction was first investigated (Table 1, Entries 1—7). In the presence of 0.1 mol% catalyst, the reaction turned out to be very sluggish while Et_3N was used as the base (Table 1, Entry 1). However, to our delight, the reactions employing inorganic bases performed much better even with significantly reduced catalyst loading (0.01 mol%) (Table 1, Entry 2—7). In general, the yield improved with the increasing strength of the base and K_3PO_4•$3H_2O$ gave the best result (76%, Table 1, Entry 7). Although the reaction tolerates moisture, addition of extra amount of water exhibited deleterious effect on the yield (Table 1, Entry 8 vs. 7). Further increasing the steric hindrance of the ligand by replacing the mesityl group with a bulkier adamantly group dramatically decreased the yield (Table 1, Entry 9 vs. 7). Most importantly, excellent yield is obtained for cross-coupling of bromobenzene with phenylboronic acid catalyzed by $Pd_2(dmba)_2Cl_2$/**1** (0.01 mol%) at 120 ℃ (Table 1, Entry 10). Furthermore, the reaction could proceed at room temperature without a significantly negative impact on the yield, although a higher catalyst loading was required (Table 1, Entry 11).

Table 1 Preliminary reaction condition survey for the Suzuki-Miyaura cross-coupling of bromobenzene with phenylboronic acid[a]

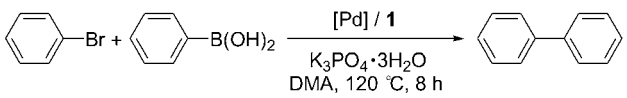


Entry	Base	Catalyst/mol%	T/℃	Time	Yield[b]/%
1	Et_3N	0.1	140	60 min	34
2	NaOAc	0.01	140	70 min	36
3	Na_2CO_3	0.01	140	70 min	54
4	K_2CO_3	0.01	140	70 min	65
5	Cs_2CO_3	0.01	140	70 min	61
6	KF	0.01	140	70 min	60
7	K_3PO_4•$3H_2O$	0.01	140	70 min	76
8[c]	K_3PO_4/H_2O	0.01	140	70 min	63
9[d]	K_3PO_4•$3H_2O$	0.01	140	70 min	47
10	K_3PO_4•$3H_2O$	0.01	120	8 h	89
11	K_3PO_4•$3H_2O$	0.1	r.t.	48 h	76

[a] General reaction condition: PhBr (1 mmol), $PhB(OH)_2$ (1.2 mmol), DMA (2 mL), base (1.5 mmol), $Pd_2(dmba)_2Cl_2$/**1**＝1∶2. [b] Isolated yield, average of two runs. [c] H_2O (0.044 mmol) was added. [d] 1,3-Bis(1-adamantyl)-4,5-dihydroimidazolium chloride (**2**) was used as the ligand.

We conjectured that both the palladacycle $Pd_2(dmba)_2Cl_2$ and *N*-heterocyclic carbene ligand precursor *N*,*N*-bis-mesityl-4,5-dihydroimidazolium chloride **1** might be critical for this cross-coupling process. In order to test our hypothesis, we initially chose an alternative Pd source, $PdCl_2$, to replace $Pd_2(dmba)_2Cl_2$ and indeed observed a marked yield decreasing (Table 2, Entry 2 vs. 1). Lack of the NHC ligand precursor **1** also led to a dramatically reduced yield, although the palladacycle $Pd_2(dmba)_2Cl_2$ itself has been known as an effective catalyst for a variety of cross-coupling reactions employing aryl iodides as coupling partners.[7c,7e-7g]

Table 2 Effect of Pd sources and ligand on catalytic efficiency[a]



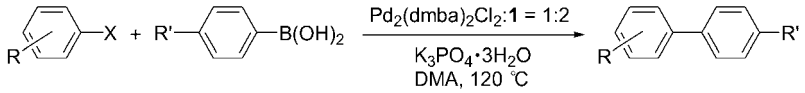
Entry	[Pd]/mol%	**1**/mol%	Yield[b]/%
1	$Pd_2(dmba)_2Cl_2$ (0.01)	0.02	89
2	$PdCl_2$ (0.02)	0.02	32
3	$Pd_2(dmba)_2Cl_2$ (0.01)	None	21

[a] General reaction condition: PhBr (1 mmol), $PhB(OH)_2$ (1.2 mmol), DMA (2 mL), K_3PO_4•$3H_2O$ (1.5 mmol). [b] Isolated yield, average of two runs.

Following the optimized reaction condition, the scope of the Suzuki-Miyaura reaction catalyzed by $Pd_2(dmba)_2Cl_2$/**1** system was investigated. A wide range of aryl halides bearing substituents of various electronic properties were found to undergo the cross-coupling reaction with phenylboronic acid in consistently excellent yields with a catalyst loading of 0.01 mol% (Table 3, Entry 1—8). More challenging aryl halide such as *p*-methoxyphenyl bromide (Table 3, Entry 10 vs. 9) also coupled well with higher catalyst loading (1 mol%). Most importantly, the coupling of aryl chlorides with phenylboronic acid catalyzed by the $Pd_2(dmba)_2Cl_2$/**1** system proceeded smoothly in good to excellent yields (Table 3, Entry 11—14), mostly within 1 h. It is noteworthy that aryl chlorides are usually challenging "unreactive" cross-coupling partners although they are readily available, versatile substrates with relatively low cost.[9] Attempts to facilitate a cross-coupling of a deactivated aryl chloride with phenylboronic acid had, unfortunately, limited success (Table 3, Entry 15). To further demonstrate the scope of this reaction, a series of arylboronic acid with various electronic properties as the other coupling partner were tested in the cross-coupling with bromobenzene. In general, these arylboronic acids underwent the cross-coupling reaction successfully to give high yields with a negligible difference among them (Table 3, Entries 16—18).

In conclusion, we have demonstrated a novel and highly efficient catalyst system with a combination of a tertiary amine-based palladacycle $Pd_2(dmba)_2Cl_2$ and an *N*-heterocyclic carbene ligand precursor (**1**, *N*,*N*-bis-mesityl-4,5-dihydroimidazolium chloride) for the Suzuki-Miyaura cross-coupling of aryl halides with arylboronic acids. A variety of aryl halides (I, Br, Cl) and arylboronic acids bearing various electronic properties were found to undergo the cross-coupling reaction in good to excellent yields at low catalyst loading of 0.01—1 mol%. Furthermore, both of the palladacycle $Pd_2(dmba)_2Cl_2$

Table 3 The Suzuki-Miyaura cross-coupling of aryl halides with arylboronic acids[a]



Entry	R	X	R'	Time	Catalyst/mol%	Yield[b]/%
1	H	I	H	1.5 h	0.01	97
2	*p*-Me	I	H	3 h	0.01	99
3	*p*-MeO	I	H	12 h	0.01	94
4	*o*-Me	I	H	5 h	0.01	98
5	H	Br	H	8 h	0.01	89
6	*p*-CH_3CO	Br	H	4 h	0.01	99
7	*p*-NO_2	Br	H	4 h	0.01	99
8	*p*-Me	Br	H	24 h	0.01	85
9	*p*-MeO	Br	H	32 h	0.01	54
10	*p*-MeO	Br	H	50 min	1.00	99
11	*p*-CH_3CO	Cl	H	50 min	1.00	98
12	2,4-diNO_2	Cl	H	45 min	1.00	99
13	*p*-CF_3	Cl	H	45 min	1.00	76
14	H	Cl	H	5 h	1.00	62
15	*p*-Me	Cl	H	10 h	1.00	42
16	H	Br	MeO	8h	0.01	90
17	H	Br	Me	8h	0.01	88
18	H	Br	CH_3CO	8h	0.01	85

[a] General reaction condition: aryl halides (1.0 mmol), arylboronic acid (1.2 mmol), DMA (2 mL), K_3PO_4•$3H_2O$ (1.5 mmol), $Pd_2(dmba)_2Cl_2$/1＝1 : 2. [b] Isolated yield, average of two runs.

and the dihydroimidazolium chloride **1** are readily accessible, insensitive to air, moisture and heat, and suitable for long-term storage.

Experimental

All reagents and solvents were purchased from commercial sources and were used without further purification. The melting points are uncorrected. ^{1}H NMR spectra were measured at 500 MHz with TMS as the internal standard.

Preparation of *N*,*N*-bis-mesityl-4,5-dihydroimidazolium chloride

A mixture of *N*,*N'*-bis-(2,4,6-trimethylphenylamino)ethane dihydrochloride (1.85 g, 5.0 mmol), 16 mL of triethyl orthoformate, and two drops of formic acid was refluxed for 3 h. The ethanol was then distilled. The temperature of the reaction mixture reached 130 ℃ for 30 min. Upon cooling to room temperature a colorless solid precipitated which was collected by filtration. Recrystallization of the crude product with acetonitrile/ether afforded 1.26 g of *N*,*N*-bis-mesityl-4,5-dihydroimidazolium chloride. Yield 74%, m.p.＞260 ℃; ^{1}H NMR (D_2O, 500 MHz) δ: 2.29 (s, 6H), 2.36 (s, 12H), 4.47 (s, 4H), 7.09 (s, 4H), 9.14 (s, 1H).

General procedure for synthesis of biaryls

A mixture of aryl halide (1 mmol), arylboronic acid (1.2 mmol), K_3PO_4•$3H_2O$ (399 mg, 1.5 mmol), $Pd_2(dmba)_2Cl_2$ (0.055 mg, 110^{-4} mmol or 5.5 mg, 0.01 mmol) and *N*,*N*-bis-mesityl-4,5-dihydroimidazolium chloride **1**, (0.069 mg, 2×10^{-4} mmol or 6.9 mg, 0.02 mmol) in DMA (2 mL) was stirred under N_2 at 120 ℃ for an indicated time (Table 3), cooled to room temperature. The solution was diluted with water (5 mL) and extracted with diethyl ether (10 mL×3). The combined organic layer was dried with anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography (silica gel, using mixture of hexane/ethyl acetate as eluent) to afford the biphenyl product.

Biphenyl White crystal, m.p. 69—70 ℃ [Lit.[10] 69—70 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.34 (t, *J*＝7.0 Hz, 2H), 7.42—7.45 (m, 4H), 7.59 (d, *J*＝7.0 Hz, 4H).

4-Methylbiphenyl White powder, m.p. 48—50 ℃ [Lit.[10] 46—47 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 2.43 (s, 3H), 7.27—7.29 (m, 2H), 7.36 (t, *J*＝7.0 Hz, 1H), 7.44—7.47 (m, 2H), 7.53 (d, *J*＝7.0 Hz, 2H), 7.62 (dd, *J*＝7.0, 2.0 Hz, 2H).

4-Methoxybiphenyl White crystal, m.p. 87—88 ℃ [Lit.[10] 87 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 3.84

(s, 3H), 6.97 (d, J=7.0 Hz, 2H), 7.29—7.31 (m, 1H), 7.35—7.42 (m, 2H), 7.52—7.56 (m, 4H).

2-Methylbiphenyl White crystal, m.p. 254—255 ℃ [Lit.[11] 255 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 2.25 (s, 3H), 7.22—7.25 (m, 4H), 7.30—7.32 (m, 3H), 7.38—7.39 (m, 2H).

4-Acetylbiphenyl Colorless crystal, m.p. 121—123 ℃ [Lit.[12] 120—121 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 2.64 (s, 3H), 7.42 (d, J=7.0 Hz, 1H), 7.46 (t, J=7.0 Hz, 2H), 7.63 (dd, J=7.0, 2.0 Hz, 2H), 7.68—7.70 (m, 2H), 8.04 (dd, J=7.0, 2.0 Hz, 2H).

4-Nitrobiphenyl Pale yellow crystal, m.p. 112—114 ℃ [Lit.[13] 113—115 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.39—7.46 (m, 3H), 7.56—7.57 (m, 2H), 7.67 (dd, J=7.5, 2.0 Hz, 2H), 8.24 (dd, J=7.5, 2.0 Hz, 2H).

2,4-Dinitrobiphenyl Yellow crystal, m.p. 107—108 ℃ [Lit.[14] 108 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.32—7.34 (m, 2H), 7.46—7.48 (m, 3H), 7.68 (d, J=7.5 Hz, 1H), 8.42 (dd, J=7.5, 2.0 Hz, 1H), 8.69 (d, J=2.0 Hz, 1H).

4-Trifluoromethylbiphenyl White solid, m.p. 66—67 ℃ [Lit.[10] 67—68 ℃]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.42—7.43 (m, 3H), 7.56—7.58 (m, 2H), 7.65 (m, 4H).

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