



A simple and novel amide ligand based on quinoline derivative used for palladium-catalyzed Suzuki coupling reaction

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ABSTRACT

This paper discusses the synthesis of the amide ligand N,N-diisopropyl quinoline-2-carboxamide from quinoline-2-carboxylic acid and diisopropylamine. The prepared ligand was utilized in the palladium catalyzed Suzuki cross-coupling reaction in ethanol-water 1:1 (v/v). The reaction exhibited high catalytic efficiency with low Pd loading (0.05 mol %) under mild reaction conditions (at 60–90 °C under air atmosphere). In this study, the catalyst was successfully used in coupling reactions between various aryl halides with phenylboronic acid to obtain desired products in excellent yields. Spectrometric methods of ¹H NMR, ¹³C NMR, and HR-MS were used to characterize this amide ligand, and its binding properties toward the Pd center were also investigated in detail via HR-MS measurements.

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1. Introduction

Palladium-catalyzed coupling reactions have played an important role in organic synthesis in recent decades [1–5]. Among these reactions, the Suzuki reaction stands out as one of the most powerful, convenient, and versatile methods for cross-coupling aryl halides with arylboronic acids [6–13]. This method has been identified as a reliable platform for carbon–carbon bond formation and has extensive use in the synthesis of natural products, pharmaceuticals, and advanced materials [14–19]. Usually, the most common ligands used for the Suzuki cross-coupling reaction are phosphines [20–26]. However, most of them are sensitive to air and moisture and are expensive and toxic, which significantly limits their industrial applications [27–30]. Recently, significant progress has been made toward the Suzuki coupling reaction through the use of phosphine-free ligands, such as N-heterocyclic carbene, N,N,O-tridentate, N,N,N-tridentate, N,O-bidentate, N,S-bidentate, and N,N-bidentate ligands (including diimine and

diamino) [2,31–47]. The latter-mentioned heteroatoms act as chelating sites toward a center metal in a proper chemical environment. Few studies practiced the use of amide ligands for Suzuki cross-coupling reaction, even though it has been verified that heteroatoms of the amide carbonyl can act as chelating sites for transition metal [48–51].

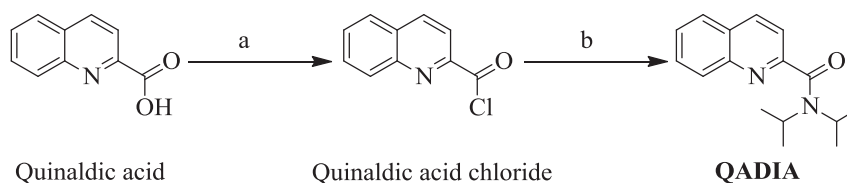
In 1993, Minisci's group obtained N,N-diisopropyl quinoline-2-carboxamide in order to study the introduction of a carbamoyl group into heteroaromatic bases via the homolytic carbamoylation of monoamides of oxalic acid [52]. It should be noted that Minisci et al. did not report the application of this amide compound. Herein, we report a palladium-based catalytic system for the Suzuki reaction using this novel, simple, and efficient amide ligand (the product of quinoline-2-carboxylic acid chloride reacting with diisopropylamine, QADIA) in aqueous media (H₂O–EtOH, 1:1) to obtain the desired coupling products in good yields under mild reaction conditions. The synthetic route of this amide ligand is illustrated in Scheme 1. The new ligand was characterized via ¹H NMR (Fig. S1), ¹³C NMR (Fig. S2), and HR-MS (Fig. S3) spectrometry.

2. Results and discussion

We initially tested the reaction of phenylboronic acid with 4-

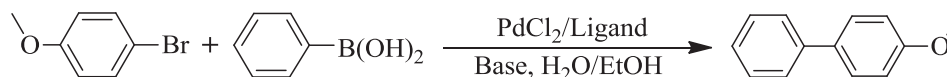
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Scheme 1. The synthetic route of QADIA. (a) CH_2Cl_2 , oxalyl chloride, reflux, 4 h; (b) CH_2Cl_2 , diisopropylamine, RT, 2 h.

Table 1
Effect of water in the palladium-catalyzed Suzuki–Miyaura reaction^a.



Entry	Ligand	[Pd] source	Solvent, V/V	Temp. (°C)/time (h)	Yield ^b (%)
1	QADIA	PdCl_2	Only EtOH	80/2	78
2	QADIA	PdCl_2	EtOH/ H_2O , 9/1	80/2	83
3	QADIA	PdCl_2	EtOH/ H_2O , 4/1	80/2	89
4	QADIA	PdCl_2	EtOH/ H_2O , 2/1	80/2	94
5	QADIA	PdCl_2	EtOH/ H_2O , 1/1	80/2	99
6	QADIA	PdCl_2	EtOH/ H_2O , 1/1	60/4	98
7	QADIA	PdCl_2	EtOH/ H_2O , 1/1	25/24	19
8	QADIA	PdCl_2	EtOH/ H_2O , 1/2	80/2	95
9	QADIA	PdCl_2	EtOH/ H_2O , 1/4	80/2	76
10	QADIA	PdCl_2	EtOH/ H_2O , 1/9	80/2	46
11	QADIA	PdCl_2	Only H_2O	80/2	23

^a Reaction conditions: n (ligand)/n (Pd) = 2,4-bromo anisole (1 mmol), phenylboronic acid (1.25 mmol), K_3PO_4 (2.0 mmol).

^b Isolated yield.

bromo anisole in 6 mL water as a model reaction to establish the best conditions for cross-coupling in the presence of 0.1 mol% of PdCl_2 and 0.2 mol% of ligand. The effect of various bases (LiOH , NaOH , NaHCO_3 , Na_2CO_3 , KOH , KHCO_3 , K_2CO_3 , K_3PO_4 , Cs_2CO_3 , and Et_3N) on the Suzuki reaction was investigated in the experiment. It was discovered that reactions with KOH , K_3PO_4 , and Cs_2CO_3 produced the desired product in good yields (Table S1). With the consideration of practical application, K_3PO_4 was chosen as the optimal base for enhancing the efficiency of this protocol.

Simultaneously, commonly used solvents (H_2O , DMF, THF, Dioxane, MeOH, EtOH, *i*-PrOH and *t*-BuOH) were also tested in the reaction. It was determined that EtOH performed best; the reaction in EtOH afforded the desired product in 78% yield after 2 h at 80 °C oil bath in the presence of 0.1 mol% PdCl_2 and 0.2 mol% ligand (Table S2). The addition of appropriate amounts water to EtOH promoted activity of the catalysis system and resulted in the desired products in better yields [53] (Table 1, entries 2–10), may be). Reasoning for this occurrence may be attributed to the possibility that the proper polarity of the media is conducive to the Suzuki coupling reaction [5,43,53]. These results suggest that the proper ratio of water and ethanol plays a key role in the model reaction with good solubility of two substrates (aryl halides and phenylboronic acid) in these mixed solvents. Ultimately, the mixture of EtOH and H_2O (1:1, v/v) was chosen as the best medium for this catalyst system.

In this study, two different catalytic precursors (PdCl_2 and $\text{Pd}(\text{OAc})_2$) were tested. From Fig. 1, it can be seen that PdCl_2 performed better than $\text{Pd}(\text{OAc})_2$ in the model reaction. In order to compare the catalytic effect of the system (QADIA/ PdCl_2 in EtOH and H_2O), diisopropylamine (DIPA), quinoline-2-carboxylic acid (QLCA), and a simple combination of QLCA and DIPA were also tested in the model reaction. It was found that QADIA performed best and could stabilize the $\text{Pd}(0)$ species more efficiently than quinoline-2-carboxylic acid and diisopropylamin (Fig. 1). It is

pertinent to note that DIPA, as a ligand, can also coordinate the metal center and promote the model reaction with the same Pd-loading to some extent, which was similar to Qiu's research [54].

Thorough understanding of the binding mechanism between the center metal and the ligand is critical in the Pd-catalyzed Suzuki coupling system. Eseola and Plass et al. confirmed that their 2-(oxazol-2-yl) pyridine compounds (N,N-bidentate ligands) coordinated with the Pd center in a 1:1 bonding model [43]. Amadio and Scrivanti et al. verified that their thioether-triazole compound (N,S-bidentate ligand) coordinated with the Pd center in a 1:1 bonding model [39]. Li et al. confirmed that their β -oxo amide ligand coordinated with Pd center in a 2:1 bonding model [48]. In order to have more insight into the binding properties of QADIA toward center

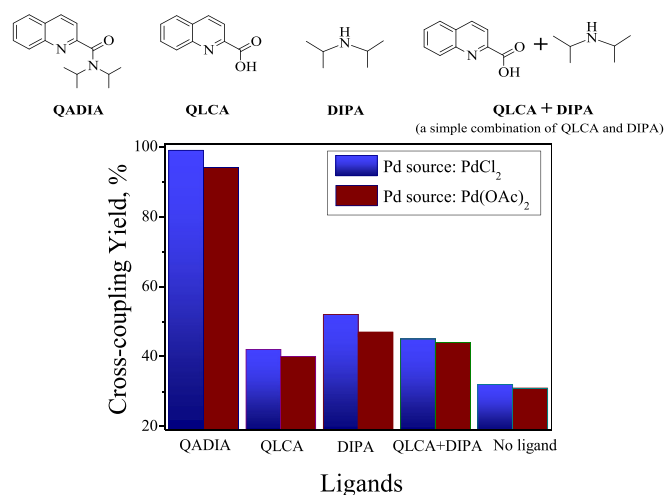


Fig. 1. Effect of different ligands and Pd sources in the model reaction (Above: QLCA, DIPA and “QLCA + DIPA” are the potential ligands comparing to QADIA).

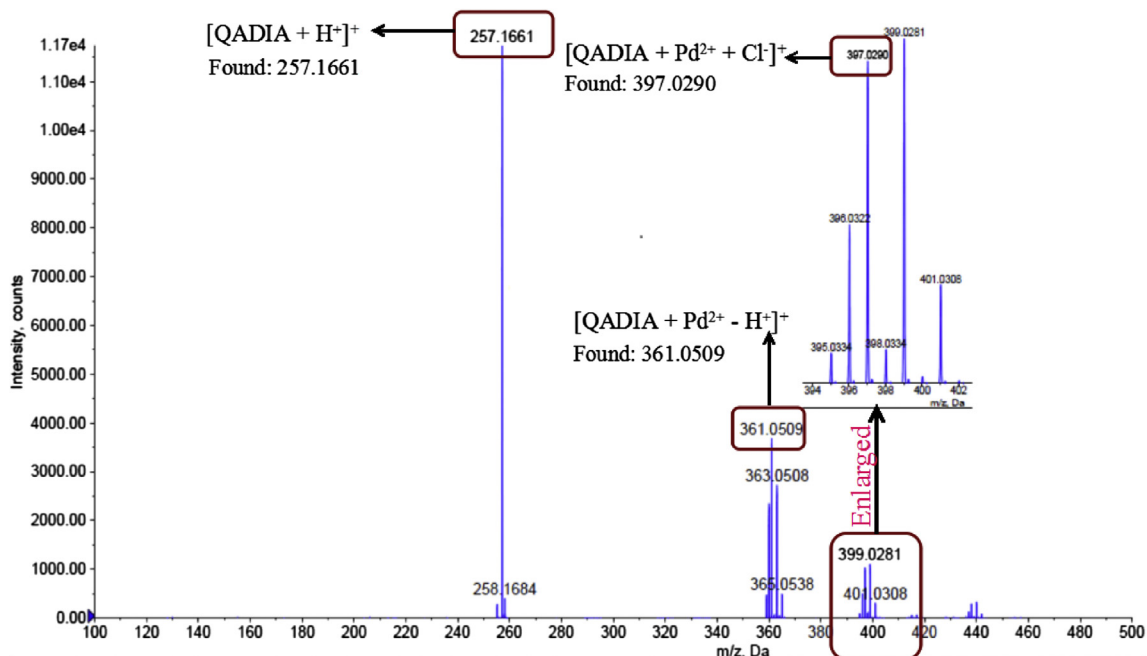
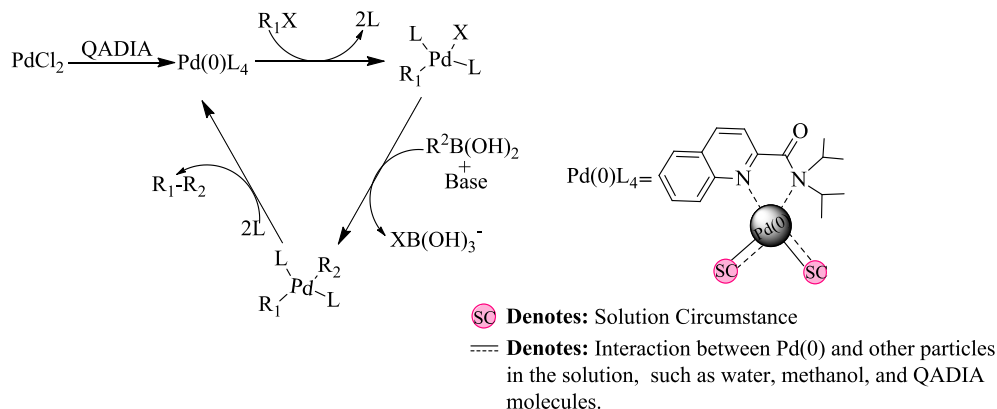


Fig. 2. ESI mass spectra of QADIA in the presence of PdCl_2 (2 equiv).

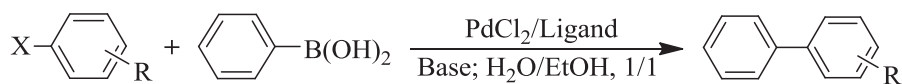
Pd , ESI mass spectrometry was used to measure of $\text{QADIA}/\text{PdCl}_2$. As shown in Fig. 2, the cluster peak at $m/z = 361.0509$ (calcd = 361.0527) corresponds to $[\text{QADIA} + \text{Pd}^{2+} - \text{H}]^+$, and $m/z = 397.0290$ (calcd = 397.0299) corresponds to $[\text{QADIA} + \text{Pd}^{2+} + \text{Cl}]^+$, both of which were clearly observed when 2 equiv of PdCl_2 was added to QADIA. This indicates the formation of a 1:1 metal-ligand catalyst. According to these results, a working mechanism was proposed on the basis of the previously proposed mechanism as outlined in Scheme 2 [12,31,40,43–45]. This proposed catalytic cycle involves oxidative addition of $\text{Pd}(0)\text{L}_4$ to the halide (R_1X) to form the organopalladium intermediate $\text{Pd}(\text{L}_2)\text{R}_1\text{X}$, transmetalation reaction of $\text{Pd}(\text{L}_2)\text{R}_1\text{X}$ with the boronic acid to form a diarylpalladium species $\text{Pd}(\text{L}_2)\text{R}_1\text{R}_2$, and reductive elimination of $\text{Pd}(\text{L}_2)\text{R}_1\text{R}_2$ to release the desired coupling product (R_1-R_2) [1,12].

In the above Pd -catalyzed Suzuki cross-coupling system, all the substrates ran smoothly in the procedure, and the desired products were isolated in excellent yields (Table 2). For the model reaction of phenylboronic acid with 4-bromo anisole, a yield of 97% was obtained only at 60°C in 5 h in the presence of 0.05 mol% PdCl_2 and 0.1 mol% QADIA (Table 2, Entry 3). When Pd -loading was decreased

to 0.005 mol%, and the reaction time was extended to 20 h, it also afforded the corresponding substituted biphenyl in a yield of 82% (Table 2, entry 4). In the QADIA/ PdCl_2 catalysis system, electron-rich, as well as electron-deficient aryl iodides and aryl bromides coupled efficiently with phenylboronic acid under mild conditions (Table 2, entries 1–12). Due to the steric hindrance of 2-bromotoluene and 2-bromoanisole, the desired products were obtained in medium yields (Table 2, entries 13, 15). In particular, the target products of 2-bromotoluene and 2-bromoanisole with phenylboronic were obtained in good yields when the reaction time was increased to 10 h in the presence of 0.1 mol% PdCl_2 and 0.2 mol% QADIA (Table 2, entries 14 (85%), 16 (92%)). This phenomenon was similar to those mentioned in previous reports concerning the use of nitrogen-based ligands in the Suzuki coupling reaction [55]. Furthermore, it was reported by Nolan's group that 3-bromopyridine and 3-bromoquinoline were not as reactive as other aromatic substrates in the Suzuki coupling reaction due to their strong coordination ability towards the center metal [56]. Both yields of the target products were normally lower than those of other coupling products under the same reaction conditions [54–56]. In this QADIA/



Scheme 2. A possible mechanism for QADIA coordinating toward center metal.

Table 2Suzuki coupling of phenylboronic acid and various aryl halides^a.

X Denotes: I, Br, Cl

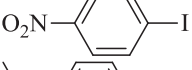
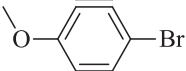
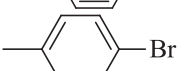
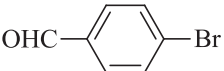
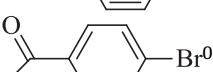
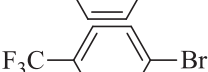
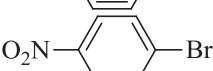
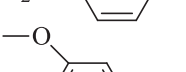
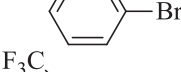
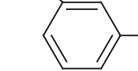
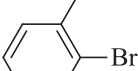
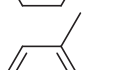
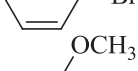
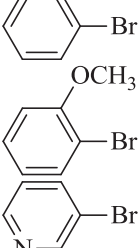
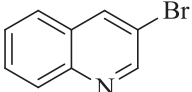
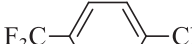
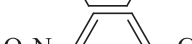
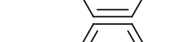
Entry	Ar-X	Pd loading (mol%)	Time (h)	Yield ^b (%)
1		0.05	5	98
2		0.05	4	98
3		0.05	5	97
4		0.005	20	82 ^c
5		0.05	5	96
6		0.05	5	97
7		0.05	5	96
8		0.05	5	97
9		0.05	5	98
10		0.05	5	98
11		0.05	5	96
12		0.05	5	97
13		0.05	5	53
14		0.1	10	85
15		0.05	5	61
16		0.1	10	92
17		0.1	10	89 ^c

Table 2 (continued)

Entry	Ar-X	Pd loading (mol%)	Time (h)	Yield ^b (%)
18		0.1	10	95 ^c
19		1	5	14 ^c
20		1	20	38 ^d
21		1	5	23 ^c
22		1	20	44 ^d

^a Reaction conditions: n (QADIA)/n (Pd) = 2, aryl halides (1 mmol), phenylboronic acid (1.25 mmol), K₃PO₃ (2.0 mmol), H₂O (3.0 mL), EtOH (3.0 mL), 60 °C.

^b Isolated yield.

^c 80 °C.

^d 90 °C, with addition of 0.25 mmol TBAB in the system.

PdCl₂ system, the reaction was found to produce good to excellent yields for the desired product with 3-bromopyridine and 3-bromoquinoline when we extended the reaction time to 10 h at 80 °C in the presence of 0.1 mol % PdCl₂ (Table 2, entries 17, 18). Encouraged by the obvious efficiency of this optimized catalytic system, we also investigated the coupling of phenylboronic acid with activated aryl chloride, which included 4-chloronitrobenzene and 4-chlorobenzotrifluoride. It is well known that the Suzuki cross-coupling of aryl chlorides is not as easy as it is for aryl iodides and aryl bromides. In order to obtain the desired coupling products, harsh reaction conditions and higher dosages of catalyst are typically required in the Suzuki reaction [47,57]. When we raised the Pd loading up to 1.0 mol% and prolonged the reaction time to 20 h by setting the reaction temperature to 90 °C (in the presence of 25 mol % TBAB, simultaneously), moderate coupling yields of 4-chlorobenzotrifluoride (38%) and 4-chloronitrobenzene (44%) with phenylboronic acid were obtained (Table 2, entries 20, 22).

Conclusively, we successfully developed a novel and simple amide ligand QADIA (N,N-diisopropyl quinoline-2-carboxamide). This compound was proved to be highly effective in the palladium-catalyzed Suzuki coupling reaction of phenylboronic acid with different substituted bromobenzenes, substituted iodo-benzenes, and heteroaryl bromides in EtOH–H₂O (1:1, v/v). In the QADIA/PdCl₂ catalysis system, the reaction can be carried out at low Pd-loading (0.05 mol %) in mild conditions (at 60–90 °C under aerobic conditions), resulting target biphenyl derivatives in good to excellent yields. The ease of preparation of this amide ligand, its low catalyst loading and stability toward air and moisture make it an ideal catalytic system for the Suzuki cross-coupling reaction. In view of the good coordinating performance of this quinoline-based amide compound towards Pd center, this amide ligand may demonstrate useful for other kinds of metal catalyzed transformations. And further effort to develop more simple and efficient amide ligands in the catalytic system and more application of this system are currently under investigation.

3. Experimental

All aryl halides and arylboronic acids were purchased from Energy Chemical. Quinaldic acid and diisopropylamine were purchased from Aladdin Industrial Corporation. Other chemicals were purchased from commercial sources without any process. NMR

spectroscopy was performed on a Bruker advance II 400 spectrometer operating at 400 MHz (¹H NMR) and 100 MHz (¹³C NMR) with CDCl₃ as the solvent and TMS as internal standard. Melting points were determined on a Thomas-Hoover capillary melting point apparatus. The isolation of pure products was carried out via column (Silica gel 200–300 mesh).

3.1. Synthesis of QADIA

Into a 250 mL three-necked flask equipped with a magnetic stirringbar, 1.73 g (0.01 mol) of quinaldic acid and 60 mL anhydrous dichloromethane are added. The mixture was stirred at room temperature for 10 min and then 10 mL oxalyl chloride (0.12 mol) was added. After thermostating at 50 °C in an oil bath and stirring for 4 h, the reaction flask was cooled. After removing all the solvent under reduced pressure, the mid-product quinaldic acid chloride was obtained. The whole of the mid-product was dissolved in 50 mL anhydrous dichloromethane in the same flask, then 1.21 g (0.012 mol) diisopropylamine was added dropwise into the mixture in 10 min under ice-water bath. After the end of addition, the mixture was stirred at room temperature for 6 h. The solvent was removed under reduced pressure and the residue was purified by recrystallization (EtOAc/Cyclohexane = 1:3). Finally, a pale yellow crystal (2.18 g) was obtained, yield: 85%. mp: 135–137 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.23 (d, *J* = 8.4 Hz, 1H), 8.13 (d, *J* = 8.5 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.74 (t, *J* = 8.0, 1.4 Hz, 1H), 7.57 (m, 2H), 3.97 (m, 1H), 3.61 (m, 1H), 1.62 (d, *J* = 6.8 Hz, 6H), 1.22 (d, *J* = 6.7 Hz, 6H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 168.81 (s, 1C), 155.99 (s, 1C), 146.88 (s, 1C), 136.92 (s, 1C), 129.77 (d, 2C), 127.63 (d, 2C), 127.02 (s, 1C), 119.66 (s, 1C), 50.69 (s, 1C), 46.18 (s, 1C), 20.65 (d, 4C) ppm; HRMS calcd for C₁₆H₂₁N₂O [QADIA + H]⁺: 257.1648, found: 257.1661 (Fig. S3). Elem. anal. calcd for C₁₆H₂₀N₂O: C, 74.97; H, 7.86; N, 10.93%. Found: C, 74.81; H, 7.98; N, 10.74%.

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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.2015.06.009>.

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