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Priority Communication

A novel zinc-catalyzed Suzuki-type cross-coupling reaction of aryl boronic acids with alkynyl bromides



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ABSTRACT

A novel Suzuki-type cross-coupling reaction of organoboron reagents with alkynyl bromides has been developed in the presence of catalytic $Et_2Zn/DMEDA$ system. The reaction afforded a variety of internal alkynes in moderate to excellent yields under mild reaction conditions without the formation of any homo-coupling products. The resulting internal alkynes have valuable applications in pharmaceutical and industrial areas. The use of relatively non-toxic zinc, chelating amine ligand and low reaction temperature make this protocol an alternative for the synthesis of internal alkynes. The scope and limitations of this protocol are also investigated.

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1. Introduction

Suzuki-Miyaura coupling reaction involves the coupling of an organic halide or pseudohalide with organoboron reagents in the presence of palladium catalyst and a base. This is a very useful tool for the construction of C-C bonds. The traditional Suzuki coupling was employed with the use of palladium catalysts along with phosphine ligands [1]. However, most of the phosphine ligands are toxic and sensitive to air and moisture. In addition to that palladium is too costly. Thus, the development of a non-palladium and phosphine-free, less expensive transition metal catalytic system is a promising area for modern research.

The Suzuki reaction exhibits a wide range of application for natural product synthesis, synthesis of polymeric materials etc. It is broadly categorized into three types depending upon the nature of the organohalide involved, (1) Coupling of organoboron reagents with C_{sp}^3 -X bonds (2) Coupling of organoboron reagents with C_{sp}^2 -X bonds (3) Coupling of organoboron reagents with C_{sp}^3 -X bonds. The couplings of organoboron reagents with C_{sp}^3 -X are most common. These have become a mainstay of modern organic chemistry for the preparation of biaryl as well as bialkyl compounds [2]. It has been widely applied for the synthesis of agrochemicals, natural products, pharmaceuticals and other materials [3]. In contrast, organic halides with C_{sp} -X bonds are quite rare to use as organo electrophiles due to its propensity to form homo-coupling products along with the cross-coupling product [4]. To the best of our knowledge, only a very few reports are available for the C_{sp} -X coupling with organoboron reagents, and which use either copper or palladium as catalysts [5]. So, to extend the efforts towards the zinc-catalyzed coupling reactions and due to the non-toxic and eco-friendly nature of zinc, we decided to explore the scope of zinc catalyst for the Suzuki type cross-coupling reaction of organoboron reagents with C_{sp} -X bonds under moderate reaction conditions.

2. Results and discussion

Based on some previous reports [6], and in continuation of our persistent interest in transition metal catalysis [7], particularly zinc catalysis [8] we initiated coupling using 1-bromotolyl acetylene and phenyl boronic acid as the model substrates. The reaction temperature was kept at 80 °C since the presence of elevated temperature predominantly gave the homo-coupled product of 1a. We used Et₂Zn/DMEDA as the catalytic system in THF solvent in the presence of molecular sieves (3 Å). In the absence of molecular sieves, we could observe phenol as the major byproduct due to the hydrolysis of phenylboronic acid. In order to minimize the phenol formation, we used molecular sieves as the drying agent to avoid any trace of moisture. A slight excess of phenyl boronic acid and K₃PO₄base were used in this reaction (3 and 4 equivalents respectively) due to a previous report which suggests that higher concentration of boronic acid and base accelerate the transmetallation step [9]. After a time period of 48 h under nitrogen atmosphere, the coupling between 1-bromotolyl acetylene 1a and



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phenyl boronic acid **2a** gave the desired cross-coupled product **3a** in 92% yield (Scheme 1). The structure of the product was established by analyzing NMR and mass spectrometric methods. All the spectral data were in good agreement with the reported values 6.

We first examined the effect of other zinc sources in this reaction and observed that Et_2Zn possesses good catalytic ability in this coupling strategy. Anhydrous $ZnCl_2$ gave traces of the desired product while other Zn salts were catalytically inactive under this reaction condition and the results are shown in Table 1.

The efficiency of cross-coupling reaction mainly depends on the choice of ligand, and therefore we studied the effect of some easily available bidentate N-N, N-O and O-O ligands for this reaction using 1a and 2a as the model substrates and catalytically active Et_2Zn as the metal source in the presence of K_3PO_4 as the base in THF solvent at 80°C under inert condition (Scheme 2). Surprisingly. the bidentate N-N ligand, N.N-dimethyl ethylenediamine (DMEDA) (L_a) gave excellent results. This may be due to the good complexing ability of this ligand with Et₂Zn [10] under the reaction conditions. Other bidentate N-N ligands like ethylene diamine (L_b), 1,10phenanthroline (L_c) and *trans*-1,2-diaminocyclohexane (L_d) were not effective for this reaction. The use of N-O ligands also gave the product, but with moderate yields (Le, Lf). Bidentate O-O ligands such as ethylene glycol (Lg) and trans-1,2-cyclohexane diol (L_h) gave lower amount of the products. Thus, we fixed our optimal ligand as DMEDA (L_a) and carried out further optimization studies in detail (Table 2).

We tried the reaction using different inorganic bases like phosphate, hydroxide, butoxide, carbonate, hydride as well as one organic base, triethyl amine (Table 2, entries 1–7). Among these we could observe that the inorganic phosphate base, K_3PO_4 showed the optimal result in which the desired cross-coupled product was generated in 92% yield (entry 1). The butoxide and triethyl amine bases were not effective for this reaction (entries 2, 7). The base KOH gave moderate yield of 52% of the product (entry 4). Other bases such as Cs_2CO_3 , K_2CO_3 and NaH afforded low yields of the products (entries 3, 5, 6).With K_3PO_4 as the optimal base, we studied the effect of different solvents using this protocol, which

revealed that THF offered the best result of the desired product 3a in 92% yield (Table 2, entry 1). Comparatively better yields of the products were obtained when polar aprotic solvents like CH₃-CN and 1,4-dioxane were used (entries 8, 9). Moderate yield was obtained when DME was used as the solvent (entry 10). Other polar aprotic solvents like DMF and DMSO were found to be less effective for this coupling reaction (entries 13, 14). No product was observed when toluene was used as the solvent (entry 15). The polar protic solvents like EtOH gave moderate result (entry 11), whereas MeOH gave only low conversion (entry 12). Further, we increased the reaction temperature to 130 C which resulted in the formation of debrominatively coupled product as the major along with traces of the desired cross-coupled product (entry 16). Later, we decreased the boronic acid equivalents to 2 and 1.5, which showed decrease in yields to 52% and 46% respectively (entries 17, 18). Decreasing the base loading resulted in lower yield of **3a** (entry 19). The decrease of reaction time to 24 h also afforded a low yield of the product (entry 20) Absence of either catalyst or ligand and absence of both catalyst and ligand resulted in traces of the required product (Table 2, entries 21–23). When the reaction was conducted in the absence of N₂ atmosphere, trace amount of the desired product was obtained (Table 2, entry 24).

Finally, we checked the effect of the amount of catalyst loading in the present coupling reaction. We conducted the reaction either by decreasing or increasing the metal–ligand ratio and arrived at a conclusion that 15 mol% of Et_2Zn and 30 mol% of DMEDA gave the excellent result (Table 3, entry 4). Further increase or decrease of metal-ligand ratio resulted in decreasing the yield of the product **3a** (Table 3, entry 1–3 and 5). Overall the optimum condition for the desired zinc-catalyzed cross-coupling reaction was found to be 15 mol% of Et_2Zn , 30 mol% of DMEDA and 4 equivalents of K₃PO₄ at 80 °C in THF solvent to obtain 96% of the desired product **3a** (Table 3, entry 4).

Finally, in order to ensure the absence of other metal contaminants in Et_2Zn , we analyzed the metal impurities in the sample by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES), which showed that other trace metals were absent in our sample (Table 4).



Scheme 1. Preliminary experiment on Zn(II)-catalyzed cross-coupling reaction of 1-bromotolylacetylene with phenyl boronicacid. (^aReaction conditions: 1-bromotolylacetylene (1 equiv.), Phenylboronic acid (3 equiv.), K₃PO₄ (4 equiv.), Et₂Zn (20 mol%), DMEDA (40 mol%), THF (3 mL), 80 °C, 48 h, under nitrogen atmosphere.)

Table 1

Screening of different zinc sources in the Zn-catalyzed cross-coupling reaction of 1-bromotolylacetylene with phenylboronic acid^a.



^a Reaction conditions: 1-bromotolylacetylene (1 equiv.), phenylboronic acid (3 equiv.), K₃PO₄ (4 equiv.), Et₂Zn (20 mol%), DMEDA (40 mol%), THF (3 mL), 80 °C, 48 h, under nitrogen atmosphere;

^b Isolated yield.

^c nd = Not detected.



Scheme 2. Ligand screening for the Zn-catalyzed cross-coupling of 1-bromotolylacetylene with phenylboronic acid. (^aReaction conditions: 1-bromotolylacetylene (1 equiv.), Phenylboronic acid (3 equiv.), K₃PO₄ (4 equiv.), Et₂Zn (20 mol%), DMEDA (40 mol%), THF (3 mL), 80 C, 48 h, under nitrogen atmosphere; ^b Isolated yield of the product is shown in parenthesis corresponding to each ligand).

Table 2

Effect of base, solvent, temperature and reaction time on the Zn-catalyzed cross-coupling reaction of 1-bromotolylacetylene with phenylboronic acid^a.



Entry	Solvent	Base	Yield 3a $(\%)^{b}$
1	THF	K ₃ PO ₄	92
2	THF	t-BuOK	traces
3	THF	CS ₂ CO ₃	17
4	THF	КОН	52
5	THF	K ₂ CO ₃	24
6	THF	NaH	29
7	THF	Et ₃ N	traces
8	CH ₃ CN	K ₃ PO ₄	83
9	1,4-Dioxane	K ₃ PO ₄	69
10	DME	K ₃ PO ₄	27
11	EtOH	K ₃ PO ₄	33
12	MeOH	K ₃ PO ₄	traces
13	DMF	K ₃ PO ₄	nd ^c
14	DMSO	K ₃ PO ₄	traces
15	Toluene	K ₃ PO ₄	nd
16 ^d	THF	K ₃ PO ₄	traces
17 ^e	THF	K ₃ PO ₄	52
18 ^f	THF	K ₃ PO ₄	46
19 ^g	THF	K ₃ PO ₄	31
20 ^h	THF	K ₃ PO ₄	77
21 ⁱ	THF	K ₃ PO ₄	traces
22 ^j	THF	K ₃ PO ₄	traces
23 ^k	THF	K ₃ PO ₄	traces
24 ¹	THF	K ₃ PO ₄	traces

^a Reaction conditions: 1-bromotolylacetylene (1 equiv.), phenylboronic acid (3 equiv.), K₃PO₄ (4 equiv.), Et₂Zn (20 mol%), DMEDA (40 mol%), THF (3 mL), 80 °C, 48 h, under nitrogen atmosphere;

^b Isolated yield;

^c nd = Not detected;

^d Reaction temp. 130 °C;

^e phenylboronic acid (2 equiv.);

^f phenylboronic acid (1.5 equiv.);

^g K₃PO₄ (2 equiv.);

^h Reaction time 24 h;

i Absence of Et₂Zn;

^j Absence of DMEDA;

^k Absence of both Et₂Zn and DMEDA;

¹ Absence of nitrogen atmosphere.

Table 3

Effect of the amount of catalyst loading in the Zn-catalyzed cross-coupling reaction of 1-bromotolylacetylene with phenylboronic acid^a.



^a Reaction conditions: 1-bromotolylacetylene (1 equiv.), phenylboronic acid (3 equiv.), K₃PO₄ (4 equiv.), THF (3 mL), 80 C, 48 h, under nitrogen atmosphere; ^b Isolated yield.

Table 4

ICP-OES analysis of Et₂Zn.

	Zn (ppm)	Cr (ppm)	Mn (ppm)	Co (ppm)	Ni (ppm)	Pd (ppm)	Cu (ppm)	Cd (ppm)	Pb (ppm)
Et ₂ Zn	66.22	nd							

nd = Not detected





Scheme 3. Substrate scope of the novel Zn-catalyzed cross-coupling reaction of 1-bromoaryl acetylenes with arylboronic acids. (^aReaction conditions: 1-bromotolylacetylene (1 equiv.), phenylboronic acid (3 equiv.), K₃PO₄ (4 equiv.), Et₂Zn (15 mol%), DMEDA (30 mol%), THF (3 mL), 80 °C, 48 h, under nitrogen atmosphere. ^b 4-fluoro phenylboronic acid neopentyl glycol ester was used; ^c (Z) 3-hexenyl-3-boronic acid catechol ester was used; nd = Not detected).

To explore the generality and functional group tolerance of the zinc-catalyzed Suzuki-type cross-coupling reaction, we carried out similar reactions with varying functionality of both 1-bromo arylacetylenes and aryl boronic acids. Initially we conducted a series of reaction with 1-bromo tolylacetylene with both electron withdrawing and electron donating group substituted boronic acids



Scheme 4. Plausible mechanistic pathway for the novel Zn-catalyzed cross-coupling reaction of 1-bromoaryl acetylenes with arylboronic acids.

(Scheme 3, 3a-3f). Here we could observe that the unsubstituted boronic acid gave excellent result (3a). The 4-F substituted boronic ester and 4-Cl substituted boronic acid yielded good results with 1bromo tolylacetylene (**3b**, **3c**). The better result of the former is attributable to its better stability. The acyl, formyl and methoxy substituted boronic acids provided only moderate yields (3d, 3e, 3f). Here we could observe that under the standard reaction conditions, the formation of stable aromatic compounds like acetophenone, benzaldehyde and anisole decrease the formation of the desired cross-coupled products. The same trend was also observed for 4-0CH₃ substituted 1-bromophenyl acetylene and unsubstituted1-bromophenyl acetylene. The unsubstituted boronic acids gave better results compared to EDG substituted boronic acids (3g, 3k). The 4-F-substituted boronic ester and 4-Cl substituted boronic acids gave higher yields compared to the 4-OCH₃ substituted boronic acid (3h, 3i, 3l and 3j, 3m). We also tried to extend this protocol to ortho-substituted and meta-substituted boronic acid. The former gave only low conversion presumably due to steric reason (3n). The latter offered excellent yields compared to the corresponding *para*-substituted ones (**30**, **3p**). Finally we tried the coupling reaction using 3-pyridyl substituted boronic acid and aliphatic (Z) 3-hexenyl-3-boronic acid catechol ester with 1-bromo arylacetylene. Unfortunately, both these cases did not result any trace of the required product (3q, 3r).

Based on the results obtained and previous report [11], we propose a plausible catalytic cycle as shown below (Scheme 4). The reaction between Zn(0) obtained by the thermal decomposition of Et_2Zn [12] and DMEDA results in the *in situ* generation of a zinc complex **I**, which exists in equilibrium with zinc complex **II**. The

zinc complex **II** can undergo oxidative addition with alkynyl bromide generating another zinc complex **III**, which can then undergo transmetallation with aryl carbanion, obtained from aryl boronic acid on treatment with K₃PO₄, forming the complex **IV**. The reductive elimination of the zinc complex **IV** would afford the internal alkyne with the regeneration of the complex **I** and which then continues the catalytic cycle.

In short, we have developed a novel zinc-catalyzed Suzuki-type cross-coupling reaction of aryl boronic acids with alkynyl bromides for the synthesis of symmetrical and unsymmetrical internal alkynes in moderate to excellent yields under mild reaction conditions. The main importance of the study is due the applications of these internal alkynes in pharmaceutical and industrial fields. The reaction is presumed to proceed via an in situ generated Zn-DMEDA complex in THF solvent at 80 °C. The protocol worked well for the coupling of 4-CH₃ and unsubstituted 1-bromo phenyl acetylenes with various electron withdrawing and electron donating group substituted boronic acids. Among them, unsubstituted and electron withdrawing -F and -Cl substituted boronic acids gave excellent results with 1-bromoaryl acetylenes. The methoxy substituted 1-bromo phenyl acetylene gave only moderate yields with aryl boronic acids. Our protocol effectively tolerates the coupling of *m*-Cl substituted boronic acids with 1-bromo aryl acetylenes compared to the corresponding para- substituted boronic acids. The protocol moderately worked for the coupling of o-CH₃ substituted boronic acids with 1-bromo aryl acetylenes possibly due to steric reason. In short, the newly developed Zn/DMEDA catalytic system offers an alternative for the synthesis of various internal alkynes with only 15 mol% of catalytic loading at 80 °C.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jcat.2019.03.005.

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