# LiCl-Accelerated Nickel Catalyzed Cross-Coupling of Aryl Tosylates with the Aryl Grignard Reagents

Xiao-Yun He<sup>a,\*</sup>, Zhi-Xun Zhang<sup>a</sup>, Chun-Jing Li<sup>a</sup>, and Yan Li<sup>a,\*\*</sup>

<sup>a</sup> Department of Chemistry and Environmental Engineering, Hebei Chemical and Pharmaceutical College, Shijiazhuang, 050026 China e-mail: \*61027391@qq.com; \*\*hebeihuagongyy@163.com

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Abstract—A mild coupling reaction catalyzed by  $Ni(acac)_2-L4$  has been studied. The catalyst acts efficiently in the reaction of biaryl coupling between various electrophiles and common or functionalized aryl Grignard reagents with high functional group tolerance. The study demonstrates that LiCl acts as an essential component in efficient cross-coupling by accelerating reduction of Ni(II) to Ni(0). The new catalytic system for selective couplings of aryl tosylates with aryl chlorides has been developed.

Keywords: aryl tosylates, C-C coupling, Ni(II)-catalyst, LiCl

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## INTRODUCTION

Cross-coupling reactions catalyzed by transition metals currently are among the most important synthetic methods [1, 2]. Application of inexpensive and abundant nickel reagents instead of those of palladium for the cross-coupling reactions has gained close attention [3]. Traditionally, aryl iodides, aryl bromides, triflates [4–6], and some more are used as organic electrophiles in cross-coupling processes [7–12]. Application of aryl tosylates in cross-coupling reactions became widely used reagents due to their availability, inexpensive synthesis, and stability towards hydrolysis [13]. Nevertheless, this greater stability of the tosylates made those less reactive in nickel-catalyzed processes, particularly in the course of the first step of the catalytic cycle. For this reason the cross-coupling reactions were carried out with the catalysts bearing a bulky electron-rich phosphine ligand or a bulky N-heterocyclic carbene (NHC), that could facilitate formation of coordinatively unsaturated, highly nucleophilic catalytic species [7]. Lately, we have reported application of a variety of tertiary phosphines [14] as excellent phosphine ligands.

Herein, we report the study of the Grignard reagents coupling with aryl tosylates involving the electron-rich phosphine ligand L4, which allowed to make several improvements in the reaction procedure.

## **RESULTS AND DISCUSSION**

To start with, we combined the simple ligand PPh<sub>3</sub> with different nickel complexes in the catalytic system,

and the reaction of methylphenyltosylate with PhMgBr was carried out for optimizing the reaction conditions (Table 1). The effect of the nickel containing components on the process was significant (Table 1).  $Ni(cod)_2$  and Ni(acac)<sub>2</sub> were found to be the most active ones leading to the yields of 64% and 65% respectively without formation of self-coupling products (Table 1, entries 1, 6). Comparison of the experimental results in combination with cost effectiveness singled out Ni(acac)<sub>2</sub> as the most promising system. Evaluation of the sulfonate leaving groups, was screened with a variety of *p*-carbomethoxyphenyl sulfonates. Trifluoromethane sulfonate led to high vield of p-toluenesulfonate (Table 1, entry 10). Benzenesulfonate demonstrated low leaving ability compared to p-toluenesulfonate (Table 1, entry 11), and the low yield was achieved for the methanesulfonate leaving group (Table 1, entry 12).

A variety of phosphine ligands were also screened. The L1 and L2 ligands did not seem to be superior to PPh<sub>3</sub> (Table 2, entry 1, 2), whereas L3, L4 and L5 that contained the tert-butyl and cyclohexyl groups were characterized by high activity, particularly L4 (Table 2, entries 3–5). According to the data presented in Table 2 (entries 6, 7), the yield lowered down significantly upon diminishing the equivalent of PhMgBr.

According to the following step of the study, application of additives, such as LiBr, LiCl or MgCl<sub>2</sub>, gave the cross-coupling products in high yields (Table 3, entries 1–3). Surprisingly, the cross-coupling reaction yield was significantly improved by addition of anhydrous . 1 . 10/

| $- OR + MgBr \xrightarrow{\text{PPh}_3, 2\%} + $ |                     |  |            |                       |    |  |  |  |  |
|--|---------------------|--|------------|-----------------------|----|--|--|--|--|
|  | 1a                  | 2a   | <b>3</b> a |                       | 4a |  |  |  |  |
| Entry  | R                   | Ni $astabust (1 m al 0/)$                          | Time h     | Yield, % <sup>b</sup> |    |  |  |  |  |
|  |                     | Ni catalyst (1 mol %)                              | 11me, n    | <b>3</b> a            | 4a |  |  |  |  |
| 1  | OTs                 | Ni(cod) <sub>2</sub>                               | 1.5        | 64                    | 0  |  |  |  |  |
| 2  | OTs                 | NiCl <sub>2</sub>                                  | 1.5        | 18                    | 4  |  |  |  |  |
| 3  | OTs                 | NiCO <sub>3</sub> .H <sub>2</sub> O                | 1.5        | 6                     | 0  |  |  |  |  |
| 4  | OTs                 | NiCl.6H <sub>2</sub> O                             | 1.5        | 13                    | 2  |  |  |  |  |
| 5  | OTs                 | Ni(OAc) <sub>2</sub> ·4H <sub>2</sub> O            | 1.5        | 21                    | 5  |  |  |  |  |
| 6  | OTs                 | Ni(acac) <sub>2</sub>                              | 1.5        | 65                    | 0  |  |  |  |  |
| 7  | OTs                 | NiCl <sub>2</sub> (dme)                            | 1.5        | 48                    | 25 |  |  |  |  |
| 8  | OTs                 | Ni(Pph <sub>3</sub> ) <sub>2</sub> Cl <sub>2</sub> | 1.5        | 35                    | 30 |  |  |  |  |
| 9  | OTs                 | NiCl <sub>2</sub> (dppp)                           | 1.5        | 47                    | 18 |  |  |  |  |
| 10   | OTf                 | Ni(acac) <sub>2</sub>                              | 1.5        | 68                    | 1  |  |  |  |  |
| 11   | OSO <sub>2</sub> ph | Ni(acac) <sub>2</sub>                              | 1.5        | 61                    | 2  |  |  |  |  |
| 12   | OMs                 | Ni(acac) <sub>2</sub>                              | 1.5        | 47                    | 3  |  |  |  |  |

Table 1. Effect of Ni(II)-catalysts on the cross-coupling reaction<sup>a</sup>

<sup>a</sup> Reaction conditions: sulfonate (1.0 equiv), the Grignard reagent (1.3 equiv), Ni(cod)<sub>2</sub> 1%, L4 (2%), LiCl (1.3 equiv), THF (3 mL). <sup>b</sup> Determined by GC using *n*-dodecane as an internal standard.

LiCl [15]. The solvents used were also important in the present method. Medium yield was achieved in ether medium (Table 3, entry 4). Application of toluene gave almost no products (Table 3, entry 5), as well as aprotic polar solvents, such as dioxane and 1,2-dimethoxyethane (DME) (Table 3, entries 6, 7). Obviously, the amount of LiCl (Table 3, entries 8, 9) influenced upon the yield of the process, though not significantly. Higher temperature supported formation of self-coupling products (Table 3, entry 10). Longer process time elevated the reaction outcome (Table 3, entry 11).

A wide range of (hetero)aryl functionalized tosylates was coupled with several representative (hetero)aryl functionalized Grignard reagents under the optimized conditions (Table 4). Various aryl electrophiles were tested with different aryl Grignard reagents **3a–3h** (Table 4). The tested tosylates coupled well furnishing the corresponding biaryl products in high yields. Steric hindrance of the ortho substituted aryl Grignard reagents affected the reaction progress, the coupling reaction of 2,6-dimethylphenyl Grignard reagent proceeded with relatively low yields (**3i**, **3j**). The reaction of functionalized tosylates coupled with common aryl Grignard reagents completed within 5–6 h (**3k**, **3m**, **3o**). The functionalized Grignard reagents with an electron-withdrawing group demonstrated low reactivity (**3n**, **3p–3s**). On the other hand, the coupling process of heteroaryl tosylates proceeded quite efficiently, second only to normal aryl electrophiles (**3t–3z**).

The accumulated data indicated that diarylation or polyarylation could be achieved upon catalysis by nickel containing compounds. As outlined in Scheme 1, the terphenyl intermediate **6** was synthesized on a multigram scale by selective biaryl coupling. The biaryl synthesis based on 4-ClC<sub>6</sub>H<sub>4</sub>OTs and the aryl Grignard reagent proceeded selectively at the C–Cl bond providing compound **5** (the compound had been reported in literature) in 89% yield (Scheme 1).

## EXPERIMENTAL

Most of the reagents used were purchased from Aldrich and Alfa Aesar. Some common reagents were available commercially elsewhere, and used without





<sup>a</sup> The list of ligands: 1-[bis(4-methoxyphenyl)phosphanyl]naphthalene (L1); biphenyl-2-yl-bis(4-methoxyphenyl)phosphane (L2), *tert*-butylcyclohexylphenylphosphane (L3), *tert*-butylcyclohexyl-(3,5-dimethylphenyl)phosphane (L4), *tert*-butylcyclohexyl-(4-triffluoromethylphenyl)phosphane (L5). <sup>b</sup> Determined by GC using *n*-dodecane as an internal standard. <sup>c</sup> 1.1 equiv of PhMgBr, 1.5 equiv of PhMgBr.

further purification, unless otherwise indicated. All reactions were carried out under the atmosphere of Ar in dry solvents under anhydrous conditions, unless otherwise noted. THF was dried over alumina under the atmosphere of  $N_2$  using a Grubbs-type solvent purification system. All arylmagnesium bromides were prepared from the corresponding aryl bromides and magnesium (turnings). All aryl tosylates were prepared according to the common procedures. L1–L5 were prepared according to literature procedure [15]. The products were purified by column chromatography on silica gel 300–400 mesh under the atmosphere of argon. Spectroscopy data of the known compounds matched the data reported in the corresponding references. Reactions were monitored by an Agilent GC Series 6890N and a GCMS 7890A. <sup>1</sup>H and <sup>13</sup>C NMR spectra were measured on a Bruker 400 M spectrometer using TMS as an internal standard and CDCl<sub>3</sub> as a solvent.

Synthesis of the Grignard reagents. The aryl Grignard reagents such as phenylmagnesium or 4-methyl-





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| $-OTs + MgBr \xrightarrow{N(acac)_2, 1\%} L4, 2\%$ EtMgBr, 1.0 equiv<br>additives Solvent, t°, time + ( |                         |                   |          |                      |            |  |  |  |  |  |
|---|-------------------------|-------------------|----------|----------------------|------------|--|--|--|--|--|
| 1a 2a 3a 4a   |                         |                   |          |                      |            |  |  |  |  |  |
| Entry   | Additives (equiv)       | Solvent           | Time h   | Yield, %a            |            |  |  |  |  |  |
|   | Additives (equiv)       | Solvent           | Time, ii | <b>3</b> a           | <b>4</b> a |  |  |  |  |  |
| 1   | LiBr (1.3)              | THF               | 1.5      | 89                   | 0          |  |  |  |  |  |
| 2   | LiCl (1.3)              | THF               | 1.5      | 95 (93) <sup>b</sup> | 0          |  |  |  |  |  |
| 3   | MgCl <sub>2</sub> (1.3) | THF               | 1.5      | 86                   | 0          |  |  |  |  |  |
| 4   | LiCl (1.3)              | Et <sub>2</sub> O | 1.5      | 45                   | 0          |  |  |  |  |  |
| 5   | LiCl (1.3)              | Toluene           | 1.5      | 5                    | 0          |  |  |  |  |  |
| 6   | LiCl (1.3)              | DME               | 1.5      | 23                   | 0          |  |  |  |  |  |
| 7   | LiCl (1.3)              | 1,4-Dioxane       | 1.5      | 53                   | 0          |  |  |  |  |  |
| 8c  | LiCl (1.1)              | THF               | 1.5      | 78                   | 8          |  |  |  |  |  |
| 9d  | LiCl (1.5)              | THF               | 1.5      | 96                   | 0          |  |  |  |  |  |
| 10e   | LiCl (1.3)              | THF               | 1.5      | 81                   | 12         |  |  |  |  |  |
| 11f   | LiCl (1.3)              | THF               | 2.0      | 96                   | 0          |  |  |  |  |  |

Table 3. Effect of ligands on the cross-coupling reaction

<sup>a</sup> Determined by GC using *n*-dodecane as an internal standard. <sup>b</sup> Isolated yield is given in parentheses. <sup>c</sup> 1.1 equiv LiCl. <sup>d</sup> 1.5 equiv LiCl. <sup>e</sup> The reaction mixture was refluxed. <sup>f</sup>The reaction was run over 2.0 h.

phenyl magnesium were prepared according to the standard procedures. 2-Methoxylpyridinyl Grignard reagent was prepared via bromine-magnesium exchange using *i*-PrMgCl. The functionalized aryl Grignard reagents, such as 2-carbomethoxyphenyl magnesium chloride, were prepared via iodine- magnesium exchange using *i*-PrMgCl. All Grignard reagents were titrated before use.

Synthesis of aryl tosylates (general procedure). To a solution of phenol (0.941 g, 10 mmol) in anhydrous THF (25 mL) under the atmosphere of argon, KOH (2.24 g, 40 mmol) was added. The mixture was stirred for 5 min at room temperature before the addition of ptoluenesulfonyl chloride (2.29 g, 12 mmol). The reaction mixture was warmed up to 60°C and stirred until phenol was consumed. Upon hydrolysis by water (15 mL), the aqueous layer was extracted with AcOEt (3×15 mL) and the combined organic layers were washed with water (2×15 mL) and brine (15 mL), then dried over anhydrous MgSO<sub>4</sub>, filtered, concentrated in vacuo, and purified to afford the corresponding product. All (hetero)aryl tosylates were prepared according to this synthetic approach reported in literature, besides N,N-diethyl 4-(tosyloxy) benzamide which was the new compound.

*N*,*N*-Diethyl 4-(tosyloxy)benzamide. White solid, yield 83%. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 1.23 (6H, CH<sub>3</sub>), 2.40 (3H, CH<sub>3</sub>), 3.31 (2H, CH<sub>2</sub>), 3.59 (2H, CH<sub>2</sub>), 7.26 (2H, CH, *J* = 8.0 Hz), 7.37 (2H, CH, *J* = 8.3 Hz), 7.45 (2H, CH, *J* = 8.0 Hz), 7.51 (2H, CH, *J* = 8.3 Hz). <sup>13</sup>C NMR spectrum,  $\delta_{\rm C}$ , ppm: 13.3, 14.5, 21.7, 39.8, 42.6, 41.7, 128.3 (CH), 128.5 (CH), 129.1 (CH), 129.9 (CH), 131.0, 133.5, 136.6, 140.7, 168.3.

Nickel-catalyzed biaryl cross-coupling of aryl tosylates with the aryl Grignard reagents. General synthesis of compounds 3. In a glove-box, an aryl tosylate (1 mmol), Ni(acac)<sub>2</sub> (1 mol %), LiCl (1.3 equiv), EtMgBr (1.0 equiv) and L4 (2 mol %) in THF (1 mL) were loaded in a dried two-neck round-bottom flask. The mixture was stirred at room temperature for 15 min before diluting with THF (2 mL). The Grignard reagent (1.3 equiv) was added slowly dropwise, then the flask was sealed, removed from the glove-box and stirred at 25°C for 2 h. The reaction progress was monitored by GC using *n*-dodecane as the internal standard. Once completed, the reaction was quenched with saturated NH<sub>4</sub>Cl followed by extraction with CH<sub>2</sub>Cl<sub>2</sub> several times. The combined organic layers were dried over anhydrous MgSO<sub>4</sub>, concentrated in

| $FG^{1}Ar-OTS + FG^{2}ArMgBr \xrightarrow{N(acac)_{2}, 1\%} FG^{1}Ar-FG^{2}$ |             |             |             |             |                   |             |                 |             |             |             |                 |             |             |
|--|-------------|-------------|-------------|-------------|-------------------|-------------|-----------------|-------------|-------------|-------------|-----------------|-------------|-------------|
| Comp.<br>no  | Yield,<br>% | Comp.<br>no | Yield,<br>% | Comp.<br>no | Yield,<br>%       | Comp.<br>no | Yield,          | Comp.<br>no | Yield,<br>% | Comp.<br>no | Yield,<br>%     | Comp.<br>no | Yield,<br>% |
| <b>3</b> a   | 93          | 3f          | 78          | 3k          | 83 <sup>b,e</sup> | 3p          | 71d             | 3u          | 83          | 3z          | 83              | 30          | 35          |
| <b>3</b> b   | 81          | 3g          | 89          | 31          | 51°               | 3q          | 73 <sup>d</sup> | 3v          | 87          | 3β          | 73 <sup>d</sup> | 3λ          | 71d,f       |
| 3c   | 89          | 3h          | 91          | 3m          | 78                | 3r          | 76 <sup>d</sup> | 3w          | 83          | 3δ          | 48              | 3π          | 45c,f       |
| 3d   | 95          | 3i          | 53          | 3n          | 71d               | 35          | 69 <sup>d</sup> | 3x          | 83          | 3ε          | 78 <sup>d</sup> | 3σ          | 77d,e       |
| 3e   | 90          | 3j          | 51          | 30          | 91 <sup>b</sup>   | 3t          | 81              | 3y          | 81          | 3η          | _               |             |             |

Table 4. Ni(II)-catalyzed cross-coupling reaction of the sulfonate group with (hetero)arylmagnesium bromidea

<sup>a</sup> The reaction was carried out on 1 mmol, the functionalized Grignard reagent was prepared by I/Mg exchange using *i*-prMgCl·LiCl and the concomitant *i*-PrI was removed. <sup>b</sup>Ni(cod)<sub>2</sub> 2%, L4 (4%), 0°C, 5–6 h. °Ni(cod)<sub>2</sub> 2%, L4 (4%), -20°C, 3.5h. <sup>d</sup>Ni(cod)<sub>2</sub> 2%, L4 (4%), -5–0°C, 4–5 h. °3k, 3σ. The yields were obtained with 2.3 equiv of ArMgX. <sup>f</sup> 3λ, 3π are the new compounds.

vacuo, and the crude mixture was purified by silica gel column chromatography.

Most of thus synthesized products had been reported earlier, and their spectral data matched those presented in literature. The products  $3\lambda$  and  $3\pi$  were the newly synthesized compounds.

**4'-***N*,*N*-diethylformamide-1,1'-biphenyl-4-carboxylic acid methyl ester (3λ). Colorless oil. <sup>1</sup>H NMR spectrum, δ, ppm: 1.21–1.35 (6H, CH<sub>3</sub>), 3.46 (2H, CH<sub>2</sub>), 3.53 (2H, CH<sub>2</sub>), 3.95 (3H, CH<sub>3</sub>), 7.49 (2H, CH, *J* = 8.1 Hz), 7.67 (4H, CH, *J* = 8.2, 5.5 Hz), 8.14 (2H, CH, *J* = 8.3 Hz). <sup>13</sup>C NMR spectrum, δ<sub>C</sub>, ppm: 13.5, 14.6, 40.2, 42.6, 51.7, 126.3 (CH), 126.7 (CH), 127.1 (CH), 129.4 (CH),130.0, 136.8, 140.6, 144.5, 166.1, 170.7. HRMS (EI): *m/z*: 311.1527. Calculated for C<sub>19</sub>H<sub>21</sub>NO<sub>3</sub> (311.1521).

**4'-***N*,*N*-diethyl-4-*N*-methyl-Diphenylformamide (3π). Yellow solid. <sup>1</sup>H NMR spectrum, δ, ppm: 1.20–1.33 (6H, CH<sub>3</sub>), 3.09 (3H, CH<sub>3</sub>), 3.48 (2H, CH<sub>2</sub>), 3.57 (2H, CH<sub>2</sub>), 6.01 (1H, NH), 7.35 (2H, CH, *J* = 7.8 Hz), 7.57– 7.65 (4H, CH), 8.01 (2H, CH, *J* = 8.0 Hz). <sup>13</sup>C NMR spectrum,  $\delta_{\rm C}$ , ppm: 13.3, 14.7, 26.1, 40.8, 43.9, 127.8 (CH), 128.7 (CH), 131.0 (CH), 135.8, 138.2 (CH), 141.0, 158.9, 162.1, 165.3, 168.2. HRMS (EI): *m/z*: 310.1687. Calculated for C<sub>19</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub> (310.1681).

**4''-Methyl-[1,1':4',1''-terphenyl]**-*N*-methyl-4-carboxamide (6). White solid. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 2.41(3H, CH<sub>3</sub>), 3.08 (3H, CH<sub>3</sub>), 5.81 (1H, NH), 7.29 (2H, CH, J = 7.2 Hz), 7.33–7.50 (4H, CH), 7.61–7.70 (4H, CH), 7.88 (2H, CH, J = 6.7 Hz). <sup>13</sup>C NMR spectrum,  $\delta_{\rm C}$ , ppm: 21.1, 26.9, 114.2 (CH), 118.9 (CH), 119.7 (CH), 120.3, 122.1 (CH), 126.3, 127.5, 128.1 (CH), 129.1 (CH), 133.5, 138.2, 143.2, 162.7. HRMS (EI): *m/z*: 301.1474. Calculated for C<sub>21</sub>H<sub>19</sub>NO (301.1467).

## CONCLUSIONS

In summary, as part of our ongoing effort, we have developed the general and practically sound method for high-yield cross-coupling of aryl tosylates with aryl Grignard reagents catalyzed by the Ni(acac)<sub>2</sub>–L4 system. The reaction has been supported by LiCl. Such conditions have proven to be highly tolerable for some sensitive functional groups including ester, cyano and amide leading to high yields of the target products. Such advantages of the process as high yield, mild conditions and low cost make this protocol attractive as an alternative and complementary to nickel-catalyzed aryl–aryl cross-coupling reactions.

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#### CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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