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Nickel-catalyzed direct C–H arylation of unactivated arenes with aryl halides

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ABSTRACT

$Ni(OAc)_2{\cdot}4H_2O$ could catalyze direct C–H arylation of unactivated arenes with aryl halides in presence of 1,10-phenanthroline without using additives.

1. Introduction

The study of metal-catalyzed intermolecular coupling of arenes has received copious attention over the past few decades [1–3]. Particularly, catalytic direct C–H arylation of arenes with haloarenes for biaryl synthesis is fast becoming an area of prominent interest. Although various transition metals, such as Pd [4–8], Rh [9–12], Ru [13–15], Ir [16,17], Ni [18–23], Cu [24,25], and other transition metals [26–31], have been shown as highly effective catalysts for this transformation, direct arylation of the C–H bond in unreactive aromatics using aryl halides has been reported in only a few studies: Pd-catalyzed arylation of benzene [4,5], Ir-catalyzed arylation of benzene [16] and Fe-catalyzed arylation of benzene [28,29].

Recently, Itami has developed a $Ni(OAc)_2$-catalyzed arylation of azoles with aryl halides and triflates using bipy or dppf as the ligand [19]. Independently, Hachiya and Miura described that $NiBr_2$·diglyme/1,10-phenanthroline/LiO*t*-Bu is an efficient catalytic system for arylation of azoles using aryl bromides. However, 0.5 equiv. of Zn powder and high temperature (150 °C) were necessary for the catalytic reaction to proceed [20]. In another study, Yamakawa and co-workers reported that Cp_2Ni could successfully catalyze direct C–H arylation of unfunctionalized aromatic hydrocarbons using aryl halides, but using BEt_3 as the additive [21]. Based on our interest in coupling reactions [32–35], herein, we describe that nickel can catalyze the direct arylation of unfunctionalized simple aromatic hydrocarbons using aryl halides without using any additives.

2. Experimental

All reactions were carried out under an argon atmosphere condition. Solvents were dried and degassed by standard methods and all aryl halides and bases were purchased from Aldrich, Alfa and TCI. All other reagents and solvents were used as is from commercial sources. Column chromatography was performed using silica gel (300–400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 200–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). NMR spectra were measured in $CDCl_3$ on a Varian Inova-400 NMR spectrometer (400 MHz) with TMS as an internal reference.

2.1. General procedure for the direct C–H arylation of unactivated arenes with aryl halides

A schlenk tube was charged with aryl halide (if it is a solid) (0.5 mmol), base (if it is a solid) (1.0 mmol), a nickel compound and ligand. The tube was connected to a vacuum line and filled with argon (3 times), arenes (2 mL), the corresponding halide (if it is a liquid) (1 equiv.) were added. The reaction mixture was stirred at 90–110 °C (as mentioned in the tables) for the time mentioned. At the end of the reaction, the reaction mixture was cooled to room temperature and was diluted with diethyl ether and water was added. The combined organic phase was dried over anhydrous Na_2SO_4. After removal of the solvent, the residue was subjected to column chromatography on silica gel using ethyl acetate and petroleum ether mixtures to afford the desired product in high purity.

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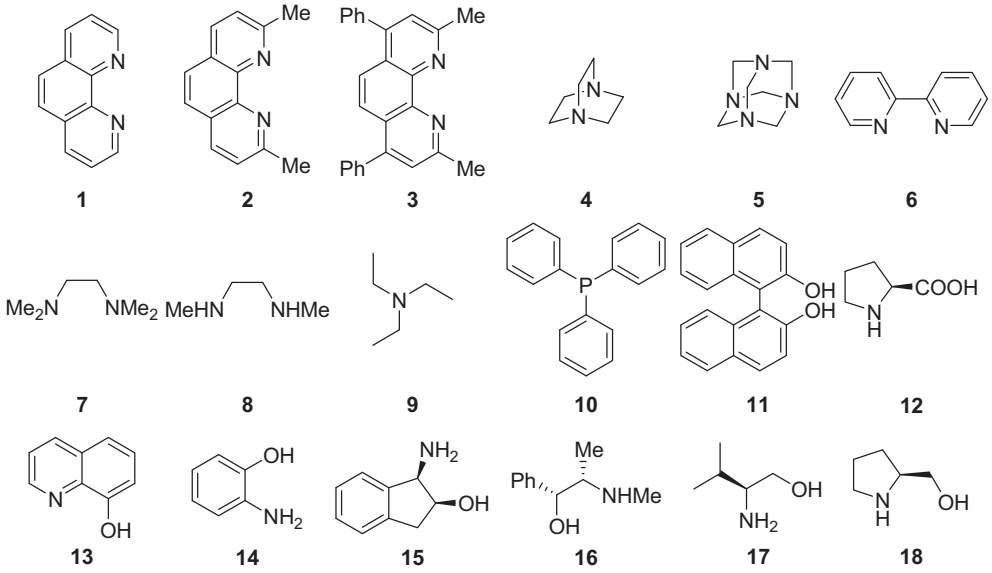


Fig. 1. The ligands evaluated in this study.

2.2. Characterization of the desired product

4-Methoxylbiphenyl: ^{1}H NMR ($CDCl_3$, 300 MHz) (δ, ppm) 3.86 (s, 3H, CH_3O), 6.96–7.00 (m, 2H, ArH), 7.26 (s, 1H, ArH), 7.40–7.44 (m, 2H, ArH), 7.52–7.57 (m, 4H, ArH); ^{13}C NMR ($CDCl_3$, 100 MHz) (δ, ppm) 129.2, 128.6, 127.2, 127.1, 114.7, 55.8; TOF MS (EI): calcd for $[C_{13}H_{12}O]^+$ requires *m*/*z* 184.0888, found 184.0888.

4-Methylbiphenyl: ^{1}H NMR ($CDCl_3$, 300 MHz) (δ, ppm) 2.37 (s, 3H, CH_3), 7.22 (d, *J* = 7.5 Hz, 2H, ArH), 7.30 (t, *J* = 7.2 Hz, 1H, ArH), 7.40 (t, *J* = 7.5 Hz, 2H, ArH), 7.48 (d, *J* = 7.8 Hz, 2H, ArH), 7.56 (d, *J* = 7.5 Hz, 2H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 141.6, 138.8, 137.4, 129.9, 129.2, 127.4, 127.4, 21.6; TOF MS (EI): calcd for $[C_{13}H_{12}]^+$ requires *m*/*z* 168.0939, found 168.0938.

Biphenyl: ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.36 (d, *J* = 7.2 Hz, 2H, ArH), 7.45 (t, *J* = 7.2 Hz, 4H, ArH), 7.60 (t, *J* = 7.2 Hz, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 141.67, 129.22, 127.72, 127.64; TOF MS (EI): calcd for $[C_{12}H_{10}]^+$ requires *m*/*z* 154.0783, found 154.0783.

4-Fluorobiphenyl: ^{1}H NMR ($CDCl_3$, 300 MHz) (δ, ppm) 7.12 (t, *J* = 8.7 Hz, 2H, ArH), 7.34 (t, *J* = 7.1 Hz, 1H, ArH), 7.43 (t, *J* = 7.2 Hz, 2H, ArH), 7.51–7.56 (m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 164.2, 161.7, 140.7, 137.8, 137.8, 129.3, 129.2, 129.1, 127.7, 127.5, 116.2, 116.0; TOF MS (EI): calcd for $[C_{12}H_9F]^+$ requires *m*/*z* 172.0688, found 172.0686.

4-Chlorobiphenyl: ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.33–7.45 (m, 5H, ArH), 7.49–7.55 (m, 4H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 140.4, 140.1, 133.8, 129.5, 129.4, 130.0, 129.4, 128.9, 128.1, 127.4, 110.2; TOF MS (EI): calcd for $[C_{12}H_9Cl]^+$ requires *m*/*z* 188.0393, found 188.0392.

3-Methoxylbiphenyl: ^{1}H NMR ($CDCl_3$, 400 MHz) (δ, ppm) 3.85 (s, 3H, CH_3), 6.88–6.90 (m, 1H, ArH), 7.12 (s, 1H, ArH), 7.18 (d, *J* = 7.6 Hz, 1H, ArH), 7.33–7.37 (m, 2H, ArH), 7.43 (t, *J* = 7.6 Hz, 2H, ArH), 7.58 (d, *J* = 8.0 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 161.6, 144.4, 142.8, 131.4, 130.4, 129.1, 128.9, 121.3, 114.6, 114.3, 56.9.

3-Methylbiphenyl: 1H NMR ($CDCl_3$, 400 MHz) (δ, ppm) 2.42 (s, 3H, CH_3), 7.22 (d, *J* = 7.5 Hz, 2H, ArH), 7.30 (t, *J* = 7.2 Hz, 1H, ArH), 7.40 (t, *J* = 7.5 Hz, 2H, ArH), 7.48 (d, *J* = 7.8 Hz, 2H, ArH), 7.56 (d, *J* = 7.5 Hz, 2H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 141.59, 138.79, 137.44, 129.93, 129.16, 127.44, 127.42, 21.56; TOF MS (EI): calcd for $[C_{13}H_{12}]^+$ requires *m*/*z* 168.0939, found 168.0950.

2,5-Bimethyl-4'-methoxybiphenyl: ^{1}H NMR ($CDCl_3$, 400 MHz) (δ, ppm) 2.23 (s, 3H, CH_3), 2.34 (s, 3H, CH_3), 3.85 (s, 3H, CH_3), 6.94 (d, *J* = 8.4 Hz, 2H, ArH), 7.05 (d, *J* = 6.8 Hz, 2H, ArH), 7.14 (d, *J* = 8.0 Hz, 1H, ArH), 7.23–7.25 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 158.6, 141.6, 135.4, 134.7, 132.5, 130.9, 130.5, 127.9, 113.7, 55.5, 21.2, 20.3; TOF MS (EI): calcd for $[C_{15}H_{16}O]^+$ requires *m*/*z* 212.1201, found 212.1206.

3. Results and discussions

Initially, we examined arylation of toluene with 4-iodoanisole using a range of commonly used ligands: a combination of 4-iodoanisole (1 equiv.), toluene (2 mL), $Ni(OAc)_2{\cdot}4H_2O$ (20 mol%), K^{t}OBu (2 equiv.) and 40 mol% of ligand (Fig. 1), at 110 °C was found to provide the desired product with several ligands. The best initial result was achieved using 1,10-phenanthroline (**1**) with 54% yield. However, PPh_3 (**10**) and *rac*-BINOL (**11**), did not yield any the desired coupling product. When DABCO (**4**), bipyridine (**6**), Et_3N (**9**), L-proline (**12**), 8-hydroxyquinoline (**13**), 2-aminophenol (**14**) were employed, only a trace amount of the direct arylation product was observed. 2,9-Dimethyl-1,10-phenanthroline (**2**), 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (**3**), TMEDA (**7**), (1*R*, 2*S*)-1-amino-2-indanol (**15**), pseudoephedrine (**16**), L-valinol (**17**), L-prolinol (**18**), were also efficient ligands for catalysing this direct arylation. However, none of them gave better result than 1,10-phenanthroline as shown in Fig. 2.

We further examined the efficiency of this transformation under the same conditions, but varying the bases. Only KO^{t}Bu produced the corresponding arylation product in 54% yield (Table 1, entry 1), although LiO^{t}Bu and LiHMDS gave a small amount of product (<10%) (Table 1, entries 2 and 7). All other tested bases, such as KOH, K_2CO_3, KOAc and K_3PO_4, were ineffective in promoting the direct arylation of toluene with 4-iodoanisole (Table 1, entries 3–6) and a mixture of KO^{t}Bu/LiO^{t}Bu also did not favor the reaction (Table 1, entry 9) [25]. We found that neither varying the nature nor the amount of base improved yields (Table 1, entries 10 and 11). The use of DMAc as a solvent also did not afford higher yields (Table 1, entry 12). Changing the ratios of the nickel source and ligand, $Ni(OAc)_2{\cdot}4H_2O$ (10%) and 1,10-phenanthroline (20 mol%) was to provide the desired product in the same yield (Table 1, entry 17). Other nickel sources such as $Ni(OAc)_2$, $NiCl_2$ or $Ni(acac)_2$ also showed promising catalytic activities (Table 1, entries 17–19). Comparatively, of all the conditions tested, readily available $Ni(OAc)_2{\cdot}4H_2O$ gave the best result. Control experiments revealed that only 1,10-phenanthroline can afford the desired

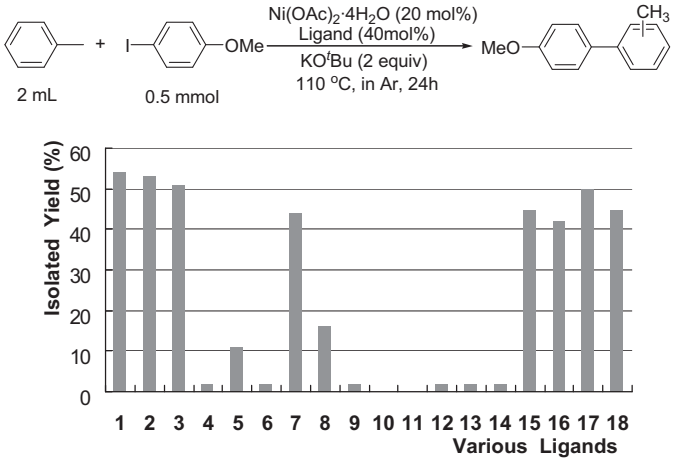


Fig. 2. Various ligands investigated in the direct C–H arylation of toluene using 4-iodoanisole.

product in reduced yield (37%) (Table 1, entry 20) [36–39] and no reaction was observed only in the presence of $Ni(OAc)_2{\cdot}4H_2O$ (Table 1, entry 21).

Benzene was also arylated by a $Ni(OAc)_2{\cdot}4H_2O$-KO^{t}Bu-1,10-phenanthroline catalyst system. Our optimization studies determined that a mixture of 4-iodoanisole (1 equiv.), benzene (2 mL), KO^{t}Bu (4 equiv.), $Ni(OAc)_2{\cdot}4H_2O$ (10 mol%), and 1,10-phenanthroline (20 mol%) at a relatively mild 90 °C provided the desired 4-methoxybiphenyl in 76% yield (Table 2, entry 2). With the optimized conditions in hand, we explored the scope of our method using commercially available aryl halides as coupling partners for benzene. Aryl iodides containing electron-donating and electron-withdrawing groups reacted well with benzene to provide corresponding products with yields ranging from 66% to 76% (Table 2, entries 3 and 5–8). Higher yields were observed with aryl iodides than with the equivalent aryl bromides (compare Table 2, entries 2, 4, 5, 8 with entries 9–12).

Next, the coupling of different arene derivatives was examined as shown in Table 3. Transformation of *p*-xylene (Table 3, entry 4) and pyridine (Table 3, entry 5) gave modest yields (55% and 32%, respectively). In contrast, direct arylation of naphthalene with aryl iodides provided up to 90% yield (Table 3, entries 1 and 2). In this case, the product was a mixture of 1-arylnaphthalene and 2-arylnaphthalene, favoring the α-position over the *β*-position. Consistent with the results of benzene arylation, the use of 4-iodoanisole led to better yields than 4-bromoanisole (Table 3, compare entries 1 and 3). A similar preference for α-position was also observed for 4-bromoanisole.

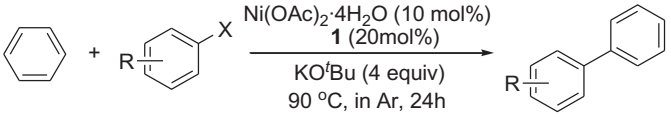
To gain some understanding of the reaction, the isotope effect of the reaction was examined. The reaction was conducted under the standard conditions using 4-iodoanisole coupling with equal amounts of benzene and benzene-d_6. A KIE of 1.22

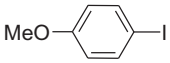
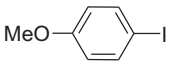
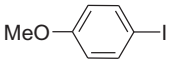
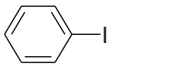
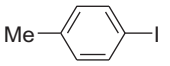
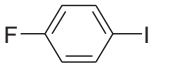
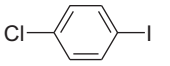
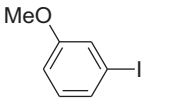
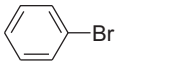
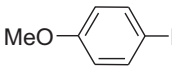
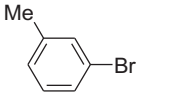
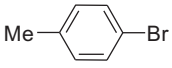
Table 1
Optimization of the reaction conditions for nickel-catalyzed C–H bond activation of toluene using 4-iodoanisole[a].

Entry	Ni cat. (mol%)	**1** (mol%)	Base	Yield (%)[b]
1	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	KO^{t}Bu	54
2	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	LiO^{t}Bu	<10
3	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	Et_3N	NR
4	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	K_3PO_4	NR
5	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	K_2CO_3	NR
6	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	NaOAc	NR
7	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	LiHMDS	<10
8	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	KOH	NR
9[c]	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	MO^{t}Bu	29
10[d]	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	KO^{t}Bu	53
11[e]	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	KO^{t}Bu	39
12[f]	$Ni(OAc)_2{\cdot}4H_2O$ (20)	40	KO^{t}Bu	30
13	$Ni(OAc)_2{\cdot}4H_2O$ (20)	20	KO^{t}Bu	51
14[g]	$Ni(OAc)_2{\cdot}4H_2O$ (10)	20	KO^{t}Bu	54
15	$Ni(OAc)_2{\cdot}4H_2O$ (10)	10	KO^{t}Bu	49
16	$Ni(OAc)_2{\cdot}4H_2O$ (5)	10	KO^{t}Bu	39
17	$Ni(OAc)_2$ (10)	20	KO^{t}Bu	53
18	$NiCl_2$ (10)	20	KO^{t}Bu	50
19	$Ni(acac)_2$ (10)	20	KO^{t}Bu	45
20	–	20	KO^{t}Bu	37
21	$Ni(OAc)_2{\cdot}4H_2O$ (10)	–	KO^{t}Bu	NR

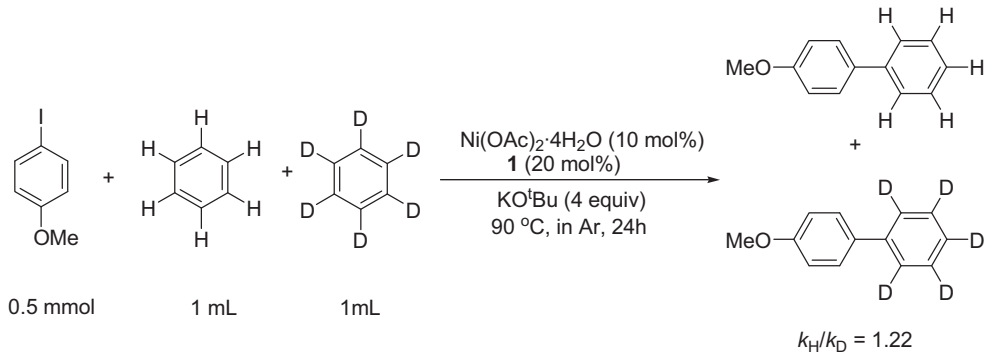
[a] Reactions were carried out with 4-iodoanisole (0.5 mmol) and base (2.0 equiv.) in toluene (2 mL) in Ar at 110 °C for 24 h.
[b] Isolated yield based on the 4-iodoanisole.
[c] LiO^{t}Bu/KO^{t}Bu mixture as the base (1:1).
[d] KO^{t}Bu (3.3 equiv.) was used.
[e] KO^{t}Bu (1.5 equiv.) was used.
[f] 0.5 mL of DMAc was used as solvent.
[g] The ratio of regioisomers (*o*/*m*/*p* = 58:26:16) was determined by ^{1}H NMR analysis.

Table 2
Nickel–catalyzed direct arylation of benzene with various aryl halides[a].



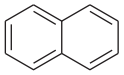
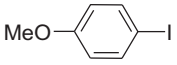
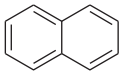
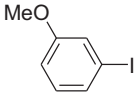
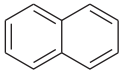
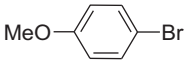
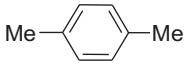
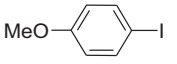
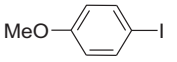
Entry	Aryl halide	KO^{t}Bu (equiv.)	Yield (%)[b]
1		3	70
2		4	76
3		5	75
4		4	73
5		4	74
6		4	66
7		4	72
8		4	68
9		4	51
10		4	61
11		4	42
12		4	58

[a] Reaction conditions: $Ni(OAc)_2{\cdot}4H_2O$ (10 mol%), **1** (20 mol%), aryl halide (0.5 mmol), KO^{t}Bu (4.0 equiv.) in benzene (2 mL) in Ar at 90 °C for 24 h.
[b] Isolated yield based on aryl halide (average of two runs).



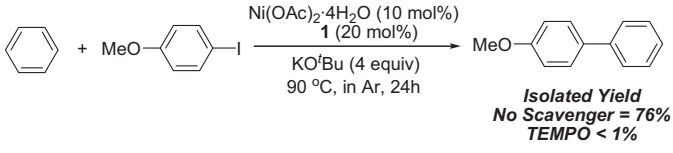
Scheme 1. Kinetic isotope effect experiment.

Table 3
Nickel-catalyzed direct arylation of different arene derivatives with various aryl halides[a].

Entry	Arene	Aryl halide	Total yield (%)[b]
1			90 (α/β = 81/19)
2			87 (α/β = 78/22)
3			39 (α/β = 75/25)
4[c]			55
5[d]			32

[a] Reaction conditions: aryl halide (0.5 mmol), $Ni(OAc)_2{\cdot}4H_2O$ (10 mol%), KO^{t}Bu (4 equiv.), **1** (20 mol%) and arene (40 equiv.), in Ar, at 100 °C for 24 h.
[b] The sum of isolated yield of a mixture of isomers. The ratio of regioisomers was determined by ^{1}H NMR analysis.
[c] *p*-Xylene (2 mL) was used and 145 °C.
[d] Pyridine (2 mL) was used and 120 °C.

was determined through the labeling experiment as shown in Scheme 1, which suggests that the C–H bond breaking event was not rate limiting. The result of this labeling experiment was surprising and prompted us to consider a possible radical pathway, which is known to exhibit such low values in radical aromatic substitutions. Experiment was performed in the presence of radical scavenger: TEMPO (1 equiv.) completely inhibited the reaction (Scheme 2). These results are incompatible with a proton-transfer pathway but are congruent very well with radical processes.



Scheme 2. Effect of radical inhibitors.

4. Conclusion

In summary, we have described a practical direct C–H arylation of unactivated arenes with aryl halides using a nickel catalyst in the absence of any additives, under mild conditions. The catalyst system consisting of $Ni(OAc)_2{\cdot}4H_2O$-KO^{t}Bu-1,10-phenanthroline is low cost and readily available and therefore provides a valuable addition to the nickel-catalyzed direct C–H arylation of arenes. We were able to apply this Ni catalyst system to the direct C–H arylation of pyridine, which suggests its potential for development of efficient and practical applications. Our further efforts are focused on the reaction-mechanism.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molcata.2011.03.007.

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