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Nickel-catalyzed Mizoroki-Heck cross-coupling reaction of aryl halides with styrenes

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Herein is presented an efficient and a simple protocol using 10 mol % of $NiCl_2.6H_2O$, 20 mol % of 2,3-dihydroxy naphthalene, 50 mol % of CTAB, and 2 equiv. of NaOH in 2.5 mL of DMF at 150°C for C-C bond forming Mizoroki-Heck coupling. Use of styrene and 4-methyl styrene with various aryl iodides and bromides having functionalities such as methyl, methoxy, nitro, fluoro, chloro, hydroxyl, trifluoromethyl, methyl, and methoxyaryl halide show smooth conversion with 50 to 93% isolated yield. The electron-withdrawing groups like nitro at *para*-position give a lower yield. The aryl iodides have been found to be more reactive than aryl bromides. The advantages of this protocol are moderate to excellent yield in the absence of an inert atmosphere, and the use of cheap and readily available catalyst.

Keywords: Mizoroki-Heck reaction, $NiCl_2.6H_2O$, 2,3-Dihydroxynaphthalene, CTAB

The Mizoroki-Heck reaction[1] is a powerful carbon–carbon bond forming process. This reaction played an important role in organic synthesis. It involves the use of different solvents, bases, and metal complexes as a catalyst. The catalytic activity depends on the ligands and metals in the complex. The use of other transition metal salts of Ni[4-8], Fe[9-11], Cu[12-15], In[16] and Palladacycles[17-19], carbenes[20-24], Schiff base ligands[25-29] palladium complexes being studied. The most preferred metal is Palladium for the C-C bond-forming reactions with excellent yields[2,3]. The most common catalytic system for C-C bond-forming reactions is Palladium with Phosphine ligands. Nevertheless, both palladium and phosphine ligands are more expensive. Therefore, the study extends to either palladium or phosphine-free catalytic systems.

Nickel-based catalysts are generally said to be less effective but some reports depict that they can replace palladium for Mizoroki-Heck coupling reactions[5-8]. as its low cost and ability to readily undergo oxidative addition reactions with C-X bonds. The Nickel complex such as $[NiCl_2(PPh_3)_2]$ [4,40], different ligands with nickel complexes such as TPPTS[30], pincer[31,32], 1,10-phenanthroline[33], Thiosemicarbazone[34], Triphos[35], $Ni(COD)_2$ with 1,1-bis(diphenylphosphino) ferrocene (DPPF) and Cy_2NMe[36], *in situ* generated Ni(dppf) catalyst[37], $Ni(OAc)_2/(PPh_3)_2$[38], nickel(II) naringenin-oxime complex[39], Ghosh *et al.*, reported reductive Mizoroki-Heck reaction[40]. $NiBr_2$•glyme and (xantphos)[41], also proved as a effective catalysts for Mizoroki-Heck coupling reactions. Recently, in 2019 Leiyang Lv and Dianhu Zhu reported direct dehydrogenative alkyl Heck-coupling in the absence of an oxidant catalyzed by nickel[42]. Ni *et al.*, gave the first report on the cross-electrophile coupling between alkyl pyridinium salts and halides catalyzed by Nickel by C-N bond activation[43]. Despite the massive effort put into the Nickel-ligand complex, there is still a great deal of potential to develop a simple and robust protocol that can be applied on a larger scale experiment.

Furthermore, our research group demonstrated the competence of the $NiCl_2.6H_2O$ with N-aryl ethanol[44] and curcumin with palladium[45] for the Mizoroki-Heck reaction. Herein, in the search of a more cost-effective protocol, explored the efficacy of ligand 2,3-dihydroxynaphthalene with $NiCl_2.6H_2O$ for the Mizoroki-Heck reaction.

Experimental Section

Material and Methods

All reactions were carried out under normal conditions, and no stringent conditions were used. All chemicals were obtained from Aldrich Chemical Co., Alfa Aesar and Spectrochem and used as received without additional purification. Lab reagent grade solvents from Loba Chemicals were used for extraction and for column chromatography.

General procedures

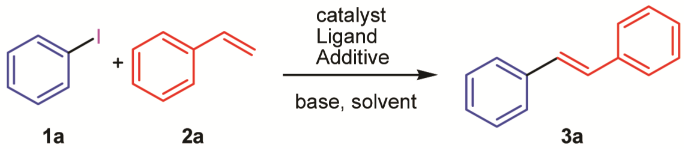
The aryl halide **1** (0.5 g, 1 equiv.) mixed with olefin **2** (2 equiv.), $NiCl_2.6H_2O$ (10 mol %), 2,3-dihydroxynaphthalene (20 mol%), CTAB (50 mol %.) NaOH (2 equiv.), and solvent DMF (2.5 mL, 5 volume) was added. The mixture was heated at 150°C for 24 h and the reaction progress was monitored by TLC. After complete conversion, the reaction mixture was diluted with cold water (10 mL) and the products were isolated by extraction with EtOAc (10 mL). The EtOAc layer was dried over anhydrous sodium sulphate and concentrated under reduced pressure to obtain the mixture of crude Heck coupled product 3 (Table 1). Column purification afforded pure Heck coupled product **3**.

Detection Method

The progress of reaction was monitored by analytical thin-layer chromatography (TLC Silica gel 60 F_{254} plates). The plates were visualized first with short wavelength UV light followed by iodine or ninhydrin staining solution followed by heating. Melting points are uncorrected and determined in an open capillary tube.

All the infrared spectra were recorded on a Shimadzu FTIR MIRacle 10 IR spectrometer. ^1H and

Table 1 — Selected observations during reaction optimization[a]



Entry	Catalyst (mol %)	Base (equiv)	Solvent (mL)	Yield[b] of **3a** (%)
1	10	TEA (2)	DMF (2.5)	–
2	10	Morpholine (2)	DMF (2.5)	–
3	10	Imidazole (2)	DMF (2.5)	–
4	10	NaOAc (2)	DMF (2.5)	20
5	10	t-BuOK (2)	DMF (2.5)	20
6	10	$NaHCO_3$ (2)	DMF (2.5)	10
7	10	Na_2CO_3 (2)	DMF (2.5)	10
8	10	$CsCO_3$ (2)	DMF (2.5)	10
9	10	K_2CO_3 (2)	DMF (2.5)	–
10	10	NaOH (2)	DMF (2.5)	90
11	10	NaOH (2)	MeOH	–
12	10	NaOH (2)	*o*-xylene (2.5)	–
13	10	NaOH (2)	1,4-dioxane (2.5)	–
14	10	NaOH (2)	H_2O (2.5)	–
15[c]	10	NaOH (2)	DMF (2.5)	–
16[d]	10	NaOH (2)	DMF (2.5)	–
17[e]	10	NaOH (2)	DMF (2.5)	60
18[f]	10	NaOH (2)	DMF (2.5)	65.25
19	01	NaOH (2)	DMF (2.5)	–
20	05	NaOH (2)	DMF (2.5)	50
21	20	NaOH (2)	DMF (2.5)	90
22	10	NaOH (3)	DMF (2.5)	90
23[g]	10	NaOH (2)	DMF (2.5)	–
24[h]	10	NaOH (2)	DMF (2.5)	70

[a] All reactions were carried out on approximately a 0.5 g scale using Iodobenzene (1a) (2.45mmol, 1 equiv.), Styrene (2a) (2 equiv.), $NiCl_2.6H_2O$ (10 mol %), 2,3-dihydroxy naphthalene (20 mol %), base (2 equiv.), cetyltrimethylammonium bromide (CTAB) (50 mol %) solvent (2.5-5 mL, 5-10 volume), temperature 150°C for 24 h. All reagent and substrate addition was done at RT (25°C). [b] Isolated yields. [c] Reaction carried without $NiCl_2.6H_2O$. [d] Reaction carried out without 2,3-dihydroxy naphthalene. [e] Reaction carried out without CTAB. [f] Reaction in presence of tetrabutyl ammonium bromide(TBAB). [g] Reaction temperature initially at ambient temperature then increased up to 50 and 80°C. [h] Reaction temperature at 120°C

^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker Avance-III 500 MHz spectrometer using TMS as an internal standard. The residual solvent signals were used as references, and the chemical shifts were converted to the TMS scale ($CDCl_3$: δ_H = 7.25-7.30 ppm).

Characterization data for the isolated products

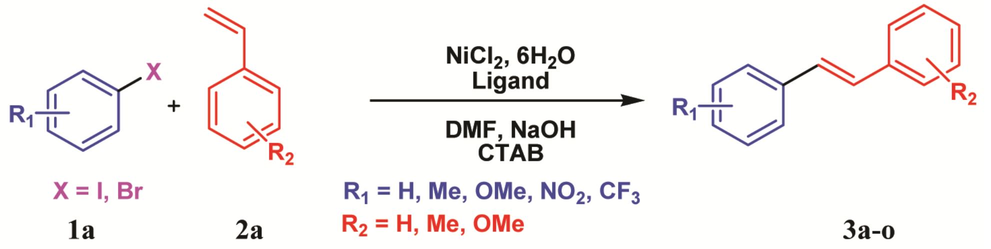
(E)- 1,2-Diphenylethene, 3a (Table 2)

The product was obtained as white solid. Yield 90% (0.3975 g). m.p.124°C. FTIR: 3020, 2358, 1494, 960, 688 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 – 7.41 (m, 4H), 7.28 (dd, *J* = 10.6, 4.8 Hz, 4H), 7.20 – 7.14 (m, 2H), 7.03 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 137.39 (s), 128.76 (d, *J* = 1.0 Hz), 127.71 (s), 126.60 (s); GC-MS (EI, 70 eV): *m/z* Found: 180 ($C_{14}H_{12}$). Calcd: 180.25 ($C_{14}H_{12}$).

1-(4-Methylstyryl)benzene, 3b (Table 2)

The product was obtained as white solid. Yield 93% (0.4142 g) . m.p.118°C. FTIR: 3022, 1803, 1508, 1072, 806, 688 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$); δ 7.50 (dd, *J* = 8.1, 1.0 Hz, 2H), 7.41 (d, *J* = 8.1 Hz, 2H), 7.35 (t, *J* = 7.7 Hz, 2H), 7.26 – 7.22 (m, 1H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.11 – 7.03 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 137.54 (d, *J* = 3.1 Hz), 134.55 (s), 129.41 (s), 128.65 (d, *J* = 5.5 Hz), 127.71 (s), 127.42 (s), 126.42

Table 2 — Scope of aryl halides with olefins for Heck coupling



Entry	1a	2a	Product	Isolated Yield (%)
1	Iodobenzene	Styrene	**3a**	90
2	Bromobenzene	Styrene	**3a**	50
3	Chlorobenzene	Styrene	**3a**	–
4	Flourobenzene	Styrene	**3a**	–
5	4-Iodotoluene	Styrene	**3b**	93
6	4-Iodoanisole	Styrene	**3c**	93
7	4-Iodonitrobenzene	Styrene	**3d**	48
8	4-CF_3 bromobenzene	Styrene	**3e**	50
9	2-Methyl-4-nitroiodobenzene	Styrene	**3f**	75
10	4-Iodophenol	Styrene	**3g**	80
11	3,5-Dimethoxybromobenzene	Styrene	**3h**	85
12	1-Bromonaphthalene	Styrene	**3i**	80
13	2-Amino-5-bromopyridine	Styrene	**3j**	90
14	5-Bromo-1,3-dichloro-2-fluorobenzene	Styrene	**3k**	91
15	Iodobenzene	4-Methyl styrene	**3b**	92
16	Bromobenzene	4-Methyl styrene	**3b**	50
17	Chlorobenzene	4-Methyl styrene	**3a**	–
18	Flourobenzene	4-Methyl styrene	**3a**	–
19	4-Iodotoluene	4-Methyl styrene	**3l**	92
20	4-Iodoanisole	4-Methyl styrene	**3m**	93
21	4-Iodo nitrobenzene	4-Methyl styrene	**3n**	50
22	4-CF_3 bromobenzene	4-Methyl styrene	**3o**	80
23	2-Methyl-4-nitroiodobenzene	4-Methyl styrene	**3p**	75
24	Iodobenzene	4-Methoxy styrene	**3c**	90
25	3,5-Dimethoxybromobenzene	4-Methoxy styrene	**3q**	50

[a] All reactions were carried out on a 0.5 g scale using Iodobenzene (1a) (2.45mmol, 1 equiv.), Styrene (2a) (2 equiv.), $NiCl_2.6H_2O$ (10 mol %), 2,3-dihydroxy naphthalene (20 mol %), base (2 equiv.), Cetyltrimethylammonium bromide (CTAB) (50 mol %) solvent (2.5-5 mL, 5-10 volume), temperature 150°C for 24 h. Addition of all reagent and substrate was done at RT (25°C). [b] Isolated yields.

(d, *J* = 4.1 Hz), 21.28 (s); GC-MS (EI, 70 eV): *m/z* Found: 194 ($C_{15}H_{14}$). Calcd: 194.27 ($C_{15}H_{14}$).

1-(4-Methoxystyryl)benzene, 3c (Table 2)

The product was obtained as off-white solid. Yield 93% (0.4178 g) . m.p.136°C. FTIR: 1508, 1247, 966, 688 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.52 – 7.43 (m,4H), 7.34 (t, *J* = 7.7 Hz, 2H), 7.23 (t, *J* = 7.4 Hz, 1H), 7.07 (d, *J* = 16.3 Hz, 1H), 6.97 (d, *J* = 16.3 Hz, 1H), 6.90 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 159.33 (s), 137.67 (s), 130.17 (s), 128.67 (s), 128.23 (s), 127.75 (s), 127.24 (s), 126.64 (s), 126.28 (s), 114.16 (s), 55.35 (s); GC-MS (EI, 70 eV): *m/z* Found: 210 ($C_{15}H_{14}O$). Calcd: 210.27 ($C_{15}H_{14}O$).

1-(4-Nitrostyryl)benzene, 3d (Table 2)

The yellow coloured solid was obtained with Yield 48% (0.2170 g) . m.p.158°C. FTIR: 1504, 1388, 970, 692 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 8.22 (d, *J* = 8.8 Hz, 2H), 7.64 (d, *J* = 8.8 Hz, 2H), 7.59 – 7.52 (m, 2H), 7.41 (dd, *J* = 10.3, 4.7 Hz, 2H), 7.36 – 7.31 (m, 1H), 7.27 (d, *J* = 15.4 Hz, 1H), 7.15 (d, *J* = 16.3 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.78 (s), 143.87 (s), 136.19 (s), 133.33 (s), 128.90 (d, *J* = 6.7 Hz), 128.36 (s), 127.05 (s), 126.89 (s), 126.30 (s), 124.42 (s), 124.18 (s); GC-MS (EI, 70 eV): *m/z* Found: 225 ($C_{14}H_{11}NO_2$). Calcd: 225.24 ($C_{14}H_{11}NO_2$).

1-(4-(Trifluoromethyl)styryl)benzene, 3e (Table 2)

The product was obtained as white solid. Yield 50% (0.2785 g). m.p.170°C. FTIR: 3026, 1612, 1321, 1107, 825, 694 cm^{-1}; ^1H NMR (500 MHz $CDCl_3$): δ 7.60 (s, 4H), 7.54 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.31 (d, *J* = 7.3 Hz, 1H), 7.18 (s, 1H), 7.14 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 140.81 (s), 136.64 (s), 131.22 (s), 129.65 (s), 129.39 (s), 129.14 (s), 128.85 (d, *J* = 6.8 Hz), 128.32 (s), 127.48 (s), 127.14 (s), 126.79 (s), 126.59 (s), 125.65 (q, *J* = 3.8 Hz), 125.32 (s), 123.16 (s); GCMS (EI, 70 eV): *m/z* Found: 248 ($C_{15}H_{11}F_3$). Calcd: 248.24 ($C_{15}H_{11}F_3$).

Results and Discussion

In preliminary Heck reactions between iodobenzene **1a** and styrene **2a** with 10 mol % of $NiCl_2.6H_2O$, 20 mol % of 2,3-dihydroxynaphthalene, 50 mol % of CTAB, 2 equiv. of organic bases like triethylamine, morpholine, and imidazole in 2.5 mL DMF solvent at 150°C for 24 h did not result in the coupled products **3a** (Table 1, entries **1−3**). With the use of inorganic bases like NaOAc, t-BuOK, $NaHCO_3$, Na_2CO_3 $CsCO_3$ and K_2CO_3 yields were observed up to 20% (Table 1, entries **4−9**). On reaction with 10 mol % of $NiCl_2.6H_2O$ and DMF (2.5 mL) with 2 equiv. of NaOH, yields were enhanced up to 90% at 150°C temperature (Table 1, entry **10**). Hence the reactions were carried out with 2 equiv. of NaOH. The experiment were carried out with methanol, *o*-xylene, 1-4-dioxane and water to know the effect of solvents but unsuccessful with no conversion (Table 1, entry **11-14**). To understand the effect of catalyst, ligand, and additives, an experiment was performed in absence of $NiCl_2.6H_2O$, 2,3-dihydroxy naphthalene (Table 1, entry **15-16**) and in absence of CTAB yield was 60% (Table 1, entry **17**). The reaction under the same conditions with tetrabutylammonium bromide (TBAB) gave 65.25% yield (Table 1, entry **18**). The control experiments were carried out to fix quantity of $NiCl_2.6H_2O$ where 1 mol % of $NiCl_2.6H_2O$ showed no conversion (Table 1, entry **19**), 5 mol % of $NiCl_2.6H_2O$ showed lower yield (50%) and (Table 1, entry **20**) and 20 mol% of $NiCl_2.6H_2O$ delivered 90% yield (Table 1, entry **21**). The experiment with 3 equiv. of NaOH showed no impact on the yield (Table 1, entry **22**). These control experiments suggested that both catalyst and ligand are effective for the formation of the product. While optimizing the temperature of the reaction, experiments carried out at different temperature and noted that (Table 1, entry **23**) 120°C was crucial to get good yield (70%) (Table 1, entry **24**).

Finally, screening experiments concluded that the reaction could deliver 90% yield within 24h by using 10 mol % of $NiCl_2.6H_2O$, 20 mol % of 2,3-dihydroxy naphthalene and 50 mol %. of CTAB, 2 equiv. of NaOH in 2.5 mL of DMF at 150°C (Table 1, entry **10**).

After optimization the Heck coupling was carried out successfully with different substituted aryl halides. The aryl iodides are more reactive than aryl bromides, whereas aryl chlorides and flourides did not show any reactivity. Styrene and 4-methyl styrene with various aryl iodides and bromides having functionalities such as methyl, methoxy, nitro, flouro, chloro, hydroxyl, triflouromethyl, methyl and methoxy aryl halide showed smooth conversion with excellent yields. The electron-withdrawing groups like nitro at *para*-position gave a lower yield. 4-methoxy styrene also show good conversion with iodobenzene and 3,5-dimethoxybromobenzene. As mentioned above, the reaction of styrene and 4-methyl styrene with chlorobenzene, flourobenzene was unsuccessful.

As per the results presented (Table 2) 2,3-dihydroxynaphthalene acts as bidentate-ligand. It forms a complex with Nickel and converts Ni(II) to

Ni(0) to start the coupling process. It was checked by recording the UV spectrum of $NiCl_2.6H_2O$ in DMF and Complex in DMF as shown in Fig. 1. It is observed that the peaks for Ni(II) at 620 and 700 nm disappear in the spectrum of complex implying the formation of Ni(0) from Ni(II).

The IR spectrum of 2,3-dihydroxynaphthalene and its complex with $NiCl_2.6H_2O$ under the same condition in which reactions are performed as shown in Fig. 2. The disappearance of peaks at 3244 and 3441 cm^{-1} for –OH in the spectrum of complex indicates the complexation of 2,3-dihydroxynaphthalene with nickel.

The complex of $NiCl_2.6H_2O$ was synthesized with 2,3-dihydroxynaphthalene in presence of NaOH as a base and solvent ethanol under stirring overnight. The prepared complex when subjected to Heck reaction of styrene and iodobenzene under the same reaction conditions affords 90% yield of product stilbene. The prepared complex was characterized by UV-DRS, and IR spectroscopy as shown in Fig. 3. There are reports on tetraalkylammonium salt additives used to stabilize the

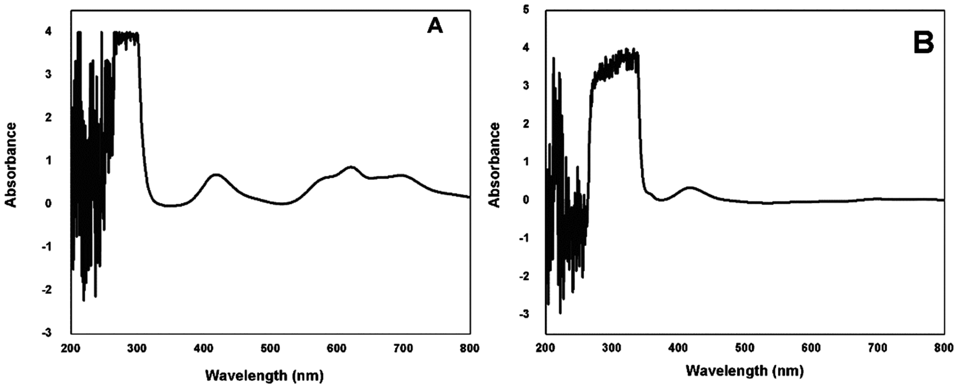


Fig. 1 — UV-Visible Spectrum of $NiCl_2.6H_2O$ (A) and $NiCl_2.6H_2O$ with 2,3-Dihydroxynaphthalene (B)

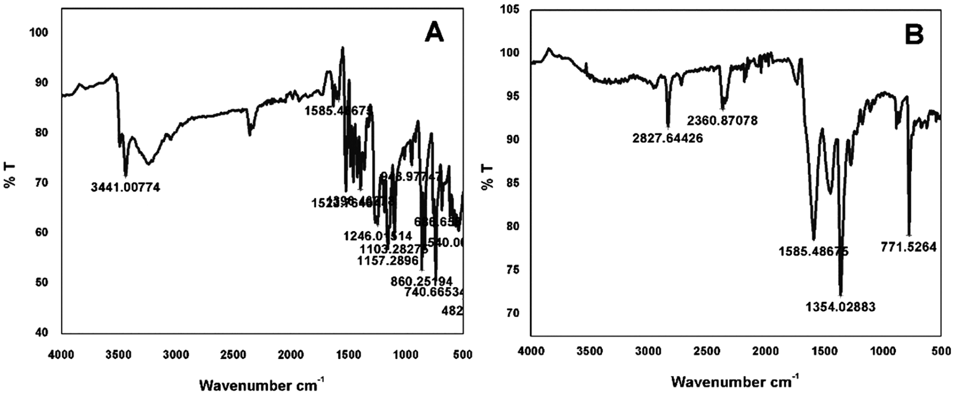


Fig. 2 — IR spectrum of 2,3-dihydroxynaphthalene (A) and 2,3-dihydroxynaphthalene Ni complex (B)

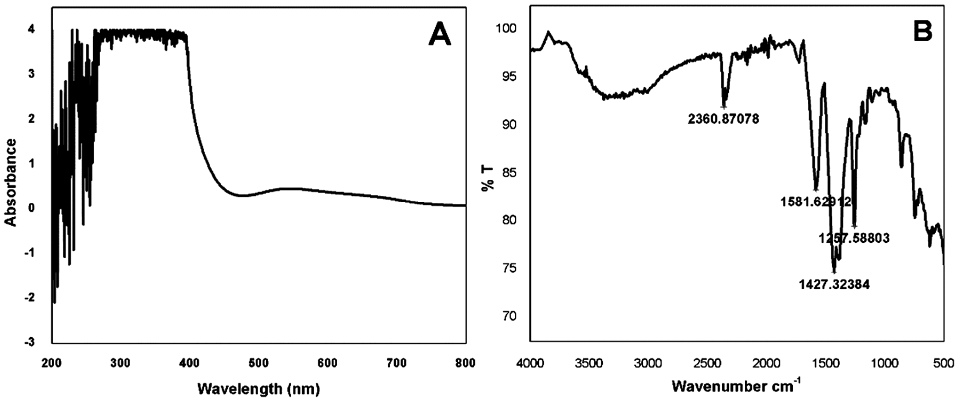
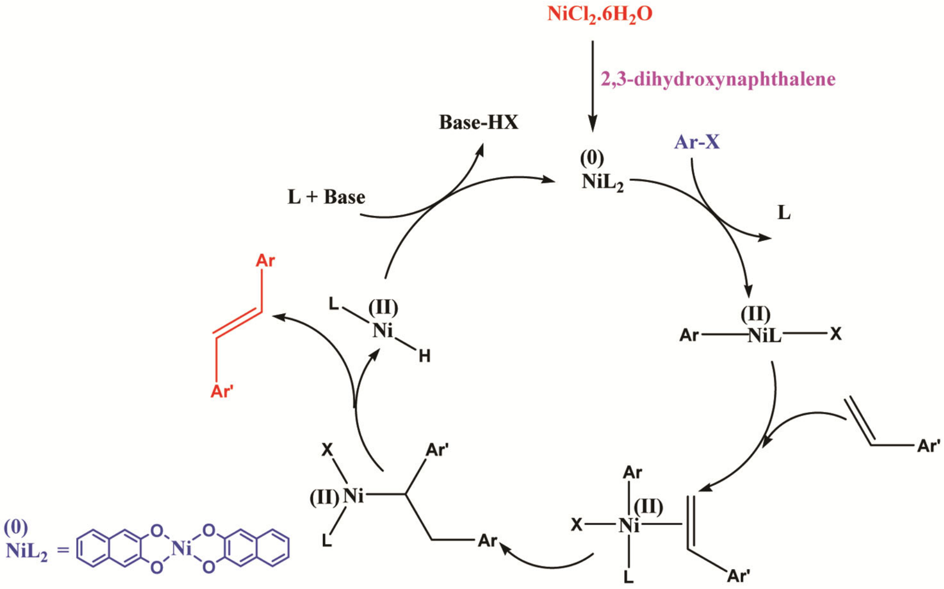


Fig. 3 — UV-Visible spectrum of 2,3-dihydroxynaphthalene Ni complex (A), IR spectrum of 2,3-dihydroxynaphthalene Ni complex (B)



Scheme 1 — Possible mechanism of Heck reaction

catalyst[7,46] cetyltrimethylammonium bromide (CTAB).

The possible mechanism for the formation of alkene derivatives is shown in Scheme 1.

Firstly Nickel chloride forms a complex with 2,3-dihydroxy naphthalene and gets converted from Ni(II) to Ni(0), and undergoes oxidative addition with aryl halide. Insertion and β-hydride elimination give the product stilbene and the Ni(II) complex reductive elimination results in Ni(0).

Conclusions

In conclusion, a highly simple, efficient, and novel protocol has been developed using 10 mol % of $NiCl_2.6H_2O$, 20 mol % of 2,3-dihydroxy naphthalene, and 50 mol % of CTAB, and 2 equiv. of NaOH in 2.5 mL of DMF at 150°C for C-C bond forming Mizoroki-Heck coupling of various aryl iodides and bromides with styrene 4-methyl styrene and 4-methoxy styrene affording yields of products 50 to 93%. The advantages of this protocol are moderate to excellent yields in absence of an inert atmosphere, and the use of cheap and easily available catalyst.

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