

Synthesis of 2,4-diarylquinolines: nickel-catalysed ligand-free cross-couplings of 4-chloro-2-arylquinolines with arylmagnesium halides in 2-methyltetrahydrofuran

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A ligand-free and room temperature protocol for the synthesis of 2,4-diarylquinolines is described. Treatment of 4-chloro-2-arylquinolines with arylmagnesium halides in the presence of a catalytic amount of nickel(II) chloride without ligands in 2-methyltetrahydrofuran (2-MeTHF) afforded the corresponding cross-coupling products in good yields.

Keywords: nickel-catalysis, cross-coupling, Grignard reagents, organomagnesium reagents, 2,4-diarylquinolines

Compounds containing the quinoline scaffold in the structural framework are expected to have wide-spectrum biological activity. Among their various applications,^{1–3} functionalised quinolines are in widespread use as a result of their anti-malarial,^{4–6} anti-inflammatory,^{7,8} anti-asthmatic,^{9,10} anti-bacterial^{11,12} and anti-hypersensitive activities.^{13,14}

Transition metal-catalysed cross-coupling has proved to be efficient in forming new C–C bonds. This methodology plays an important role in the synthesis of 4-substituted quinolines. Usually, palladium-¹⁵, iron-^{16,17} or cobalt-¹⁸ catalysed cross-coupling required the use of phosphine or other ligands. Korn *et al.*¹⁹ and Rueping and Teawsuwan²⁰ later independently discovered that Kumada-type coupling also could generate 4-substituted quinolines by cobalt and manganese catalysis without any ligands. However, the majority of the reactions required low temperatures and, after reactions were quenched, a large amount of toluene or other solvent was needed to extract the products. With 2-MeTHF, which provides very clean organic-water phase separations, the product can be conveniently isolated without adding another solvent. Furthermore, 2-MeTHF has proved to be a greener and more environmentally friendly solvent due to its specific characteristics such as high temperature for the preparation of chloro-Grignard reagents, ready availability from renewable resources, and being readily recoverable.²¹ We now report a practical, ligand-free arylation of 4-chloro-2-arylquinolines by treatment with organomagnesium reagents and catalytic amounts of nickel(II) chloride in 2-MeTHF with convenient extraction at room temperature in good to excellent yields (Scheme 1).

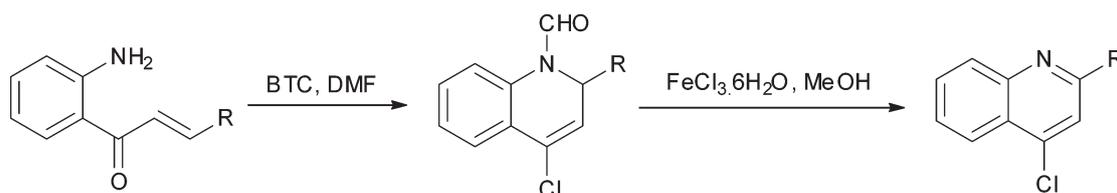


Scheme 1

Firstly, the starting materials, 4-chloro-2-arylquinolines **1**, were prepared by treatment of the 2'-aminochalcones with the novel Vilsmeier-type reagent derived from bis(trichloromethyl) carbonate (BTC) and DMF, followed by oxidative deformylation with FeCl₃·6H₂O in refluxing methanol (Scheme 2).²² We initially chose 4-chloro-2-phenylquinoline **1a** as a model to test this reaction. Treatment of **1a** (1.0 mmol) with *p*-tolylmagnesium chloride (4.0 mmol) in 2-MeTHF in the presence of nickel(II) chloride (0.1 mmol) for 1 h at room temperature afforded 2-phenyl-4-(*p*-tolyl)quinoline **2a** in 81% yield. We re-examined the reaction conditions in order to improve the yield, and the results are summarised in Table 1. It was found that inexpensive NiCl₂ was a good coupling catalyst to give 2-phenyl-4-(*p*-tolyl)quinoline **2a** in good yield at room temperature (entry **1**). Other metal salts such as CoCl₂, FeCl₃ and MnCl₂ (entries **2–4**) also catalysed the reaction, but the yields were lower under the same conditions. When 0.05 mmol catalyst was used, the reaction gave 71% yield of **2a** (entry **5**). Under the catalyst-free conditions, the yield of **2a** was sharply reduced to 15% (entry **6**). Parallel experiments using 3.0, 4.0 and 5.0 mmol of *p*-tolylmagnesium chloride indicated that 4.0 mmol of Grignard reagent was optimal for the conversion of **1a** into **2a** and that more Grignard reagent did not afford the product in significantly increased yield (entries **1**, **7** and **8**). Neither lower nor higher temperatures improved the yield of **2a** (entries **9–11**).

To extend the scope of this reaction, a wide range of substituted and structurally diverse 4-chloro-2-arylquinolines were subjected to this reaction and the corresponding substituted 2,4-diarylquinolines were obtained. The results are summarised in Table 2.

As can be seen from Table 2, with some variations in detail this method was successful in all cases except 4-chloro-2-(4-nitrophenyl)quinoline (entry **1m**). To our delight, we found that using 9-chloro-1,2,3,4-tetrahydroacridine **1n** also afforded the corresponding product in 65% yield (Scheme 3). The electronic effect of substituted groups in the 4-chloro-2-arylquinolines was obvious and the substrates with electron-donating groups (entries **1e–f**, **1j**) were more active and



Scheme 2

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Table 1 Addition of *p*-tolylMgCl to 4-chloro-2-phenylquinoline **1a** to afford 2-phenyl-4-(*p*-tolyl)quinoline **2a**

Entry	Cat. /mol%	Ratio : 1a: <i>p</i> -tolylMgCl	Temp. /°C	Yield ^a /%
1	NiCl ₂ (10)	1:4	r.t.	81
2	CoCl ₂ (10)	1:4	r.t.	73
3	FeCl ₃ (10)	1:4	r.t.	35
4	MnCl ₂ (10)	1:4	r.t.	76
5	NiCl ₂ (5)	1:4	r.t.	71
6	NiCl ₂ (0)	1:4	r.t.	15
7	NiCl ₂ (10)	1:3	r.t.	68
8	NiCl ₂ (10)	1:5	r.t.	82
9	NiCl ₂ (10)	1:4	0	43
10	NiCl ₂ (10)	1:4	-10	39
11	NiCl ₂ (10)	1:4	40	68

^aIsolated yields based on 4-chloro-2-phenylquinoline **1a**

efficient in providing the products in higher yields than those with the electron-withdrawing groups (entries **1g**, **1m**). On the other hand, while the use of electron-rich substituted aryl magnesium halides (entry 1) was successful, the use of electron deficient substituents or of heterocyclic magnesium halides gave the corresponding desired products in rather diminished yields (entries **1k**, **1l**).

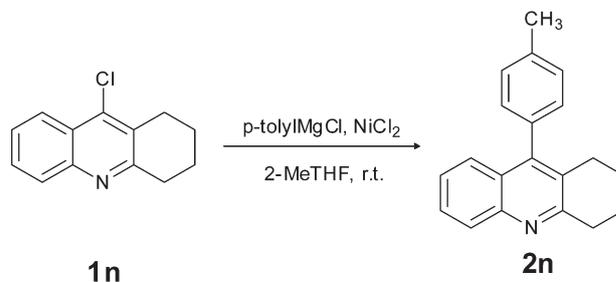
In summary, ligand-free nickel catalysed cross-coupling of 4-chloro-2-arylquinolines with arylmagnesium halides to prepare a range of 2,4-diarylquinolines in 2-MeTHF was developed. Furthermore, the reaction products can be directly extracted and no additional solvents were needed. We believe that this protocol will be a useful alternative for the synthesis of 2,4-disubstituted quinolines from readily available precursors.

Experimental

¹H NMR and ¹³C NMR spectra were recorded on a Varian 400 MHz or Bruker Avance III (500 MHz) instrument in CDCl₃ as the solvent, and chemical shifts were expressed in parts per million (ppm) using TMS as an internal standard. IR spectra (KBr) were recorded on an AVATAR-370 instrument. Mass spectra were measured with a Trace Finnigan DSQ instrument. Melting points were measured on a Büchi B-540 apparatus and are uncorrected.

Preparation of 4-methylphenylmagnesium chloride

A solution of 4-chlorotoluene (4.0 mmol) in 2-MeTHF (5.0 mL) was added dropwise to a three-necked flask, charged with activated magnesium (4.8 mmol), 2-MeTHF (1.0 mL) and nitrogen atmosphere. The mixtures were refluxed for 6 h. The flask was then cooled to room temperature and the Grignard reagent was used directly in the following experiment.

**Scheme 3**

Preparation of 2,4-diarylquinolines

A 25-mL Schlenk tube, equipped with a magnetic stirring bar and a septum, was charged with the appropriate 4-chloro-2-arylquinoline (1.0 mmol), NiCl₂ (0.10 mmol) and 2-MeTHF (5.0 mL). The corresponding arylmagnesium halide (4.0 mmol) was added dropwise at room temperature. The reaction mixture was stirred at the temperature stated in each experiment until the cross-coupling reaction was complete (0.5–1 h, monitored by TLC). The suspension was quenched with HCl (5.0 mL) and the organic phase was dried with anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure to yield the corresponding crude product, which was purified by silica gel chromatography using 2% ethyl acetate/petroleum ether as eluent.

2,4-Diphenylquinoline (2a): White solid; m.p. 113–114 °C (lit.²³ 111–112 °C); ¹H NMR δ: 8.23 (1H, d, *J* = 8.4 Hz), 8.19–8.14 (2H, m), 7.89 (1H, d, *J* = 8.4 Hz), 7.80 (1H, s), 7.72 (1H, td, *J* = 8.4, 1.4 Hz), 7.58–7.44 (9H, m); ¹³C NMR δ: 119.3, 124.5, 125.5, 125.6, 126.2, 127.5 (2C), 128.3, 128.5 (2C), 128.7 (2C), 129.2, 129.4 (2C), 129.9, 138.2, 139.5, 148.6, 149.0, 156.7; MS (EI): *m/z* (%) = 281 (M⁺, 75), 204 (30), 77 (20).

2-Phenyl-4-(*p*-tolyl)quinoline (2b): White solid; m.p. 101–102 °C (lit.²⁴ 100–100.5 °C); ¹H NMR δ: 8.21 (1H, d, *J* = 8.4 Hz), 8.16 (2H, d, *J* = 7.2 Hz), 7.90 (1H, d, *J* = 8.4 Hz), 7.77 (1H, s), 7.69 (1H, t, *J* = 7.6 Hz), 7.49 (2H, t, *J* = 6.8 Hz), 7.43 (4H, d, *J* = 8.0 Hz), 7.32 (2H, d, *J* = 7.6 Hz), 2.46 (3H, s); ¹³C NMR δ: 21.8, 119.6, 125.9, 126.0, 126.4, 127.8 (2C), 129.0 (2C), 129.5 (3C), 129.6 (3C), 130.2, 135.6, 138.5, 139.8, 148.8, 149.4, 157.0; MS (EI): *m/z* (%) = 295 (M⁺, 35), 280 (20), 205 (100).

2,4-Di-*p*-tolylquinoline (2c): Pale yellow solid; m.p. 107–108 °C; IR: ν_{\max} 2920, 1592, 1543, 1497, 1357, 818, 764 cm⁻¹; ¹H NMR δ: 8.22 (1H, d, *J* = 8.2 Hz), 8.08 (2H, d, *J* = 8.0 Hz), 7.90 (1H, dd, *J* = 8.4, 0.8 Hz), 7.77 (1H, s), 7.70 (1H, td, *J* = 8.2, 1.2 Hz), 7.45–7.41 (3H, m), 7.35–7.30 (4H, m), 2.47 (3H, s), 2.42 (3H, s); ¹³C NMR δ: 19.8, 20.1, 119.2, 124.9, 125.5, 125.6, 125.9, 128.6, 129.1 (3C), 129.3 (3C), 129.7, 130.0, 135.4, 136.9, 138.0, 138.1, 148.4, 149.0, 156.8; HRMS (ESI): Calcd for C₂₃H₂₀N 310.1596 (M+H) found 310.1582.

2-(4-Methoxyphenyl)-4-(*p*-tolyl)quinoline (2d): Pale yellow solid; m.p. 108–109 °C (lit.²⁵ 108 °C); ¹H NMR δ: 8.21 (1H, d, *J* = 8.6 Hz), 8.14 (2H, d, *J* = 8.8 Hz), 7.88 (1H, dd, *J* = 8.4, 0.8 Hz), 7.74 (1H, s),

Table 2 Nickel(II)-catalysed cross-coupling between 4-chloro-2-arylquinolines and aryl magnesium halides

Entry 1	Quinoline 2-substituent (R)	Arylmagnesium halide (Ar)	Product 2	Time /h	Yield/% ^a
1a	Phenyl	<i>p</i> -Tolyl	2a	1	81
1b	Phenyl	Phenyl	2b	1	77
1c	<i>p</i> -Tolyl	<i>p</i> -Tolyl	2c	0.5	85
1d	4-Methoxyphenyl	<i>p</i> -Tolyl	2d	0.5	93
1e	3,4-Dimethylphenyl	<i>p</i> -Tolyl	2e	0.5	88
1f	3,4-Dimethoxyphenyl	<i>p</i> -Tolyl	2f	0.5	95
1g	4-Chlorophenyl	<i>p</i> -Tolyl	2g	1	50 ^b
1h	2-Furyl	Phenyl	2h	1	65
1i	2-Thiophenyl	<i>p</i> -Tolyl	2i	1	70
1j	<i>p</i> -Tolyl	1-Naphthyl	2j	0.5	80
1k	Phenyl	3,4,5-Trifluorophenyl	2k	1	42
1l	Phenyl	1-Thiophenyl	2l	1	Trace
1m	4-Nitrophenyl	<i>p</i> -Tolyl	2m	1	None

^aIsolated yields based on heteroaryl chloride.^bRatio of **1g** : *p*-tolylMgCl = 1 : 2.5

7.69 (1H, td, $J = 8.4, 1.2$ Hz), 7.46–7.39 (3H, m), 7.34 (2H, d, $J = 8.0$ Hz), 7.03 (2H, d, $J = 8.6$ Hz), 3.88 (3H, s), 2.48 (3H, s); ^{13}C NMR δ : 21.4, 55.4, 114.1, 118.8, 125.5, 125.6, 125.8, 128.8, 129.1, 129.3, 129.6, 129.8, 131.9, 135.3, 138.2, 148.4, 149.0, 156.2, 160.6; MS (EI): m/z (%) = 325 (M^+ , 100), 310 (41), 281 (22).

2-(3,4-Dimethylphenyl)-4-(p-tolyl)quinoline (2e): Pale yellow solid; m.p. 115–116 °C; IR: ν_{max} 2917, 1589, 1543, 1494, 823, 761 cm^{-1} ; ^1H NMR δ : 8.23 (1H, d, $J = 8.4$ Hz), 7.98 (1H, s), 7.88 (2H, t, $J = 8.4$ Hz), 7.77 (1H, s), 7.69 (1H, td, $J = 8.4, 1.2$ Hz), 7.48–7.39 (3H, m), 7.34 (2H, d, $J = 7.6$ Hz), 7.25 (1H, t, $J = 7.6$ Hz), 2.47 (3H, s), 2.38 (3H, s), 2.33 (3H, s); ^{13}C NMR δ : 19.8, 20.1, 21.4, 29.8, 119.2, 124.9, 125.5, 125.6, 125.9, 128.6, 129.1 (2C), 129.3 (3C), 129.7, 130.0, 135.3, 136.9, 138.0, 138.1, 148.4, 149.0, 156.8; HRMS (ESI): Calcd for $\text{C}_{24}\text{H}_{22}\text{N}$ 324.1752 (M+H) found 324.1740.

2-(3,4-Dimethoxyphenyl)-4-(p-tolyl)quinoline (2f): Pale yellow solid; m.p. 120–121 °C; IR: ν_{max} 2930, 1590, 1517, 1498, 1263, 1024, 765 cm^{-1} ; ^1H NMR δ : 8.22 (1H, d, $J = 8.4$ Hz), 7.88 (2H, m), 7.75 (1H, s), 7.73–7.65 (2H, m), 7.46–7.41 (3H, m), 7.35 (2H, d, $J = 8.0$ Hz), 6.97 (1H, d, $J = 8.4$ Hz), 4.04 (3H, s), 3.95 (3H, s), 2.48 (3H, s); ^{13}C NMR δ : 21.4, 56.0, 56.1, 110.3, 110.9, 118.8, 120.2, 125.5, 125.8, 129.1 (2C), 129.3 (2C), 129.5, 129.6, 132.2, 135.3, 136.5, 138.2, 148.4, 149.0, 149.1, 150.1, 156.1; HRMS (ESI): Calcd for $\text{C}_{24}\text{H}_{22}\text{NO}_2$ 356.1651 (M+H) found 356.1644.

2-(4-Chlorophenyl)-4-(p-tolyl)quinoline (2g): white solid; m.p. 91–92 °C; IR: ν_{max} 2917, 1592, 1491, 1091, 814, 756 cm^{-1} ; ^1H NMR δ : 8.19 (1H, dd, $J = 8.4, 0.4$ Hz), 8.13–8.07 (2H, m), 7.89 (1H, dd, $J = 8.4, 0.8$ Hz), 7.72 (1H, s), 7.69 (1H, td, $J = 8.4, 1.6$ Hz), 7.47–7.38 (5H, m), 7.32 (2H, d, $J = 8.0$ Hz), 2.46 (3H, s); ^{13}C NMR δ : 30.1, 119.1, 125.9, 126.1, 126.6, 129.0 (2C), 129.2 (2C), 129.5 (2C), 129.6 (2C), 129.8, 130.2, 135.4, 135.7, 138.2, 138.6, 148.9, 149.6, 155.6; HRMS (ESI): Calcd for $\text{C}_{22}\text{H}_{17}^{35}\text{ClN}$ 330.1050 (M+H) found 330.1033.

2-(Furan-2-yl)-4-phenylquinoline (2h): White solid; m.p. 91–92 °C (lit.²⁶ 90–92 °C); ^1H NMR δ : 8.19 (1H, d, $J = 8.4$ Hz), 7.84 (1H, dd, $J = 8.4, 1.2$ Hz), 7.76 (1H, s), 7.69 (1H, ddd, $J = 8.4, 6.8, 1.2$ Hz), 7.60 (1H, dd, $J = 1.6, 0.8$ Hz), 7.56–7.46 (6H, m), 7.43 (1H, td, $J = 8.4, 1.2$ Hz), 6.57 (1H, dd, $J = 3.6, 2.0$ Hz); ^{13}C NMR δ : 110.5, 112.2, 115.3, 117.6, 120.2, 125.6, 126.2, 128.4, 128.5 (2C), 129.3 (3C), 129.4, 129.7, 137.8, 144.1, 148.2, 149.2; MS (EI): m/z (%) = 271 (M^+ , 23), 195 (13), 183 (100), 77 (75).

2-(Thiophen-2-yl)-4-(p-tolyl)quinoline (2i): White solid; m.p. 95–96 °C; IR: ν_{max} 2923, 1592, 1547, 1498, 1428, 1082, 821 cm^{-1} ; ^1H NMR δ : 8.13 (1H, d, $J = 8.4$ Hz), 7.84 (1H, d, $J = 8.4$ Hz), 7.77–7.59 (3H, m), 7.57–7.28 (5H, m), 7.13 (1H, dd, $J = 4.8, 3.6$ Hz), 2.47 (3H, s); ^{13}C NMR δ : 21.1, 117.5, 125.3, 125.4, 125.6, 127.6, 128.0, 128.3, 128.9 (2C), 129.0 (2C), 129.1, 129.2, 134.8, 137.9, 145.0, 148.2, 148.7, 151.4. HRMS (ESI): Calcd for $\text{C}_{20}\text{H}_{16}\text{NS}$ 302.1003 (M+H)⁺ found 302.1004.

4-(Naphthalen-1-yl)-2-(p-tolyl)quinoline (2j): White solid; m.p. 135–136 °C; IR: ν_{max} 2919, 1594, 1542, 782, 767 cm^{-1} ; ^1H NMR δ : 8.33 (1H, s), 8.11 (2H, d, $J = 8.0$ Hz), 7.98 (2H, dd, $J = 15.6, 8.0$ Hz), 7.90 (1H, s), 7.71 (1H, t, $J = 7.6$ Hz), 7.62 (1H, t, $J = 7.2$ Hz), 7.54–7.46 (2H, m), 7.45–7.37 (2H, m), 7.33–7.26 (4H, m), 2.43 (3H, s); ^{13}C NMR δ : 21.5, 120.3, 125.2, 125.9, 126.0 (2C), 126.1, 126.4, 126.8, 127.3, 127.4 (2C), 128.2, 128.6, 129.5 (4C), 129.6, 131.8, 133.3, 135.8, 136.2, 139.5, 147.9, 156.5; HRMS (ESI): Calcd for $\text{C}_{26}\text{H}_{20}\text{N}$ 346.1596 (M+H)⁺ found 346.1596.

2-Phenyl-4-(3,4,5-trifluorophenyl)quinoline (2k): Pale yellow solid; m.p. 146–147 °C; IR: ν_{max} 3060, 1527, 1405, 1043, 772 cm^{-1} ; ^1H

NMR δ : 8.29 (1H, s), 8.17 (2H, d, $J = 7.2$ Hz), 7.83–7.71 (3H, m), 7.58–7.42 (4H, m), 7.23–7.15 (2H, m); ^{13}C NMR δ : 114.1 ($J = 6.0$ Hz), 114.2 ($J = 6.0$ Hz), 119.4, 124.9, 125.1, 127.2, 127.7 (2C), 129.1 (2C), 129.9, 130.2, 130.5, 134.4 ($J = 6.0$ Hz), 139.2 ($J = 35$ Hz), 141.5 ($J = 35$ Hz), 146.1, 148.8, 150.1 ($J = 6.0$ Hz), 152.6 ($J = 6.0$ Hz), 156.9; HRMS (ESI): Calcd for $\text{C}_{21}\text{H}_{13}\text{F}_3\text{N}$ 336.1000 (M+H)⁺ found 336.0990.

9-(p-Tolyl)-1,2,3,4-tetrahydroacridine (2n): Yellow solid; m.p. 135–136 °C (lit.²⁷ 145–146 °C); ^1H NMR δ : 8.01 (1H, d, $J = 8.4$ Hz), 7.62–7.55 (1H, m), 7.36–7.26 (4H, m), 7.10 (2H, d, $J = 8.0$ Hz), 3.20 (2H, t, $J = 6.6$ Hz), 2.61 (2H, t, $J = 6.4$ Hz), 2.46 (3H, s), 2.00–1.92 (2H, m), 1.79 (2H, m). ^{13}C NMR δ : 21.2, 22.7, 22.9, 27.9, 33.9, 125.0, 125.5, 126.5, 127.7, 128.0, 128.2, 128.6 (2C), 128.9 (2C), 133.6, 137.1, 145.5, 146.6, 158.5; MS (ESI): m/z = 274.4 (M+1)⁺.

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