**NH₄I-mediated sp³ C-H cross-dehydrogenative coupling of benzylamines with 2-methylquinoline for the synthesis of E-2-styrylquinolines**

Xue Li¹, Bin Huang¹, Jiangwei Wang², YuanYuan Zhang¹ and WeiBo Liao¹

**Abstract**

Without any metal catalyst, a simple and efficient method for the synthesis of E-2-styrylquinolines through sp³ C-H cross-dehydrogenative coupling of benzylamines with 2-methylquinolines mediated by NH₄I under air is successfully developed. The oxidative olefination proceeded through deamination and sp³ C–H bond activation. A plausible mechanism is proposed for the construction of E-2-styrylquinolines.

**Keywords**

2-methylquinolines, benzylamines, E-2-styrylquinolines, NH₄I, sp³ C-H cross-dehydrogenative coupling

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**Introduction**

On account of their unique structures and reactivities, quinoline and its derivatives are widely utilized to prepare various biologically important compounds.¹,² Among quinoline derivatives, E-2-styrylquinolines are typical bioactive compounds acting as potent HIV-1 integrase inhibitors,³–⁵ leukotriene receptor antagonists,⁶,⁷ and antiallergic drugs.⁸ The wide applications of E-2-styrylquinolines have attracted considerable attention in synthetic chemistry. In the past 10 years, widely used methods for the synthesis of E-2-styrylquinolines have mainly involved the reactions of 2-methylquinolines with benzyl alcohols,⁹–¹² benzyl amines,⁹,¹³–¹⁵ aldehydes,¹⁶–¹⁹ and N-benzylidene-4-methylbenzenesulfonamides²⁰ (Scheme 1). In 2011, Qian et al.²⁰ disclosed that Fe(OAc)₂ promoted the reactions of 2-methylquinolines with benzyl amines,⁹,¹³–¹⁵ benzyl alcohols,⁹–¹² and aldehydes,¹⁶–¹⁹ and N-benzylidene-4-methylbenzenesulfonamides by removing a molecule of p-toluenesulfonamide to give E-2-styrylquinolines. In 2019, Liang et al.¹⁶ reported that various 2-alkenylquinolines could be produced from 2-methylquinolines and aldehydes under the synergistic organocatalysis of 1,3-dimethylbarbituric acid/HOAc for 24 h. In 2020, Susanta et al.⁹ reported that E-2-styrylquinolines could be prepared from 2-methylquinolines by reactions with primary alcohols or primary amines using the NaCl/TBHP oxidative system. In the same year, Zhang et al.¹⁰ showed that reactions of 2-methylquinolines and primary alcohols catalyzed by MnO₂ in the presence of KOH eliminated molecule of H₂O and were transformed into E-2-styrylquinolines. Nevertheless, these methods suffer from environmental and economic concerns as they utilize strong oxidants and transition-metal catalysts, which impede the applicability of these methods. To avoid these drawbacks, we successfully developed a method that involves deamination and sp³ C-H cross-dehydrogenative coupling of 2-methylquinolines and benzylamines to give various E-2-styrylquinolines mediated by NH₄I in moderate to good yields (30%–93%) with NMP (N-methyl-2-pyrrolidone) as the solvent, without any other metal catalyst at 160 °C for 10 h (Scheme 1).

¹Drug Research Center, Traditional Chinese Medicine Institute of Jiangxi, Nanchang, P.R. China
²Jiangxi Provincial Hospital of Traditional Chinese Medicine, Nanchang, P.R. China

**Corresponding author:**

Xue Li, Drug Research Center, Traditional Chinese Medicine Institute of Jiangxi, Nanchang 330046, P.R. China.

Email: m18702635626@163.com
Results and discussion

The 2-Methylquinoline (1a) and benzylamine (2a) were selected as model substrates to optimize the reaction conditions for the synthesis of E-2-styrylquinoline (3a). The effects of different catalysts, solvents, and temperatures were investigated. The results are summarized in Table 1. First, different additives such as NBS (N-bromosuccinimide), NIS (N-iodosuccinimide), TBAI (tetra-n-butylammonium iodide), NH₄I, and KI were investigated and NH₄I was found to be the best, affording a 52% yield of 3a (Table 1, entries 1–5). No product was formed in the absence of a catalyst, even on increasing the reaction temperature to 160 °C (Table 1, entry 6). Next, the solvent was optimized and NMP was found to be the best (Table 1, entries 4 and 7–9). With NMP as the solvent, the reaction temperature and time were further optimized. When the temperature was increased from 120–160 °C, the yield of 3a increased from 52% to 85% (Table 1, entries 4, 10, and 11), which indicated that the reaction proceeded best at 160 °C. To further improve the yield of 3a, increased reaction times of up to 24 h at 160 °C were tested; however, the yield was not improved (Table 1, entries 11–14). The optimum reaction time was therefore about 10 h (Table 1, entry 11). When the reaction was carried out under N₂, the yield of 3a decreased dramatically (Table 1, entry 15), which indicated that the mixture required the presence of O₂ or air. Subsequently, the reaction was carried out under an O₂ atmosphere and the yield of target product 3a was found to be almost equal to the yield in air (Table 1, entries 11 and 16). So the optimal reaction conditions were established as follows: using NH₄I as the additive, NMP as the solvent, 160 °C, 10 h.

Table 1. Optimization of the reaction conditions for the synthesis of 3a.¹

| Entry | Additive | Solvent | Temp (°C) | Yield of 3a (%)³ |
|-------|----------|---------|-----------|-----------------|
| 1     | NBS      | NMP     | 120       | Trace           |
| 2     | NIS      | NMP     | 120       | Trace           |
| 3     | TBAI     | NMP     | 120       | 10              |
| 4     | NH₄I     | NMP     | 120       | 52              |
| 5     | KI       | NMP     | 120       | Trace           |
| 6     | None     | NMP     | 160       | 0               |
| 7     | NH₄I     | CH₃CN   | 80        | Trace           |
| 8     | NH₄I     | Toluene | 110       | Trace           |
| 9     | NH₄I     | DMSO    | 160       | Trace           |
| 10    | NH₄I     | NMP     | 140       | 64              |
| 11    | NH₄I     | NMP     | 160       | 85              |
| 12    | NH₄I     | NMP     | 160       | 85              |
| 13    | NH₄I     | NMP     | 160       | 86              |
| 14    | NH₄I     | NMP     | 160       | 85              |
| 15    | NH₄I     | NMP     | 160       | 18              |
| 16    | NH₄I     | NMP     | 160       | 86              |

NBS: N-bromosuccinimide; NIS: N-iodosuccinimide; TBAI: tetra-n-butylammonium iodide; DMSO: dimethyl sulfoxide.

¹Reaction conditions: 1a (1.0 mmol), 2a (3.0 mmol), catalyst (1.2 equiv), solvent (4 mL), in air, 10 h.
²Isolated yield.
³Reaction time: 14 h.
⁴Reaction time: 18 h.
⁵Reaction time: 24 h.
⁶Under N₂.
⁷Under O₂.
With optimized conditions in hand, we set out to explore the substrate scope of various quinolines having sp3 carbons for oxidative cross-dehydrogenative coupling reactions. The target products 3 were obtained in moderate to good yields ranging from 51% to 93% by reacting quinolines 1 with benzylamine (2a) in NMP under air at 160 °C for 10 h. The results are shown in Table 2. The nature of the substituents on substrates 1 affected the reaction yields to some degree. Both halogen-substituted and methyl-substituted 2-methylquinoline smoothly afforded the corresponding products in 54%–93% yields (Table 2, entries 2–12). Among them, when substituents were attached to C-3, C-4, C-6, C-7, or C-8, the yields of the halogen-substituted products were slightly higher than those of the methyl-substituted products. Notably, dimethyl-substituted quinolines only offered the products of olefination at the 2-methyl position, the methyls attached at other positions were unreactive (Table 2, entries 2, 4, 6, 9, and 12). Besides, 2-methylquinoxaline and 1-methylisoquinoline also exhibited excellent reactivity with benzylamine (2a) under the standard conditions and gave the olefination products 3n in 60% and 3o in 51% yields (Table 2, entries 14 and 15).

Subsequently, we set out to examine the substrate scope of various methanamines 2 in reactions with 2-methylquinoline (1a). The target products 3 were obtained in good yields ranging from 30% to 90% under the optimized conditions. The results are shown in Table 3. The nature of different R groups of benzylamines 2 affected the reaction yields slightly. Halogen-substituted benzylamines and methyl-substituted benzylamines reacted with 1a to afford the corresponding olefination products in 72%–90% yields (Table 2, entries 1–8). It is worth noting that naphthalen-α-methanamine (2i) provided the desired product 3x in excellent yield 88% (Table 2, entry 9). Besides, a heterocyclic methanamine such as 2-thiophene-methanamine (2j) and an aliphatic amine such as cyclohexyl methanamine (2k) also underwent deamination and were transformed effectively into the corresponding products 3y and 3z in yields of 73% and 30%, respectively (Table 2, entries 10 and 11).

According to the 1H nuclear magnetic resonance (NMR) spectra of olefination products 3 and earlier studies,14–16 we were able to conform the (E)-configurations of the olefination products 3.

To further explore the mechanism of oxidative olefination for the construction of E-2-styrylquinolines, several control experiments were carried out. First, when adding the radical scavenger TEMPO (1.0 equiv) to the standard reaction, 3a could be afforded in 84% yield (Scheme 2(a)), which suggested that the reaction may not proceed through a radical intermediate. Second, when 2a alone was subjected to the standard reaction conditions, 30% yield of phenylmethanimine, 10% yield of N-benzylbenzamide, and a trace amount of benzaldehyde were observed.
Table 3. Substrate scope of various primary amines.a

| Entry | R     | Product | Yield (%) |
|-------|-------|---------|-----------|
| 1     | (2a) 2-CH₃ | 3p       | 89        |
| 2     | (2b) 2-Cl   | 3q       | 72        |
| 3     | (2c) 3-CH₃ | 3r       | 76        |
| 4     | (2d) 3-F    | 3s       | 83        |
| 5     | (2e) 3-Cl   | 3t       | 87        |
| 6     | (2f) 4-CH₃ | 3u       | 90        |
| 7     | (2g) 4-F    | 3v       | 86        |
| 8     | (2h) 4-Cl   | 3w       | 80        |
| 9     | (2i)       | 3x       | 88        |
| 10    | (2j)       | 3y       | 73        |
| 11    | (2k)       | 3z       | 30        |

aReaction condition: 1a (1.0 mmol), 2 (3.0 mmol), NH₄I (1.2 equiv), NMP (4 mL), air, 160 °C, 10 h.
bStructures were confirmed by ¹H and ¹³C nuclear magnetic resonance (NMR) spectroscopy.
cIsolated yield based on 1a.

(Scheme 2(b)). Finally, when 1a reacted with phenylmethanimine under the standard conditions, an 86% yield of 3a was obtained (Scheme 2(c)), suggesting that phenylmethanimine might be an intermediate in this reaction.

Based on these observations and related references, a plausible mechanism is proposed in Scheme 3. Initially, NH₄I is oxidized to a highly active “I⁺” species in air. Second, the benzylamine is transformed into a phenylmethanimine by elimination under the influence of “I⁺.” Finally, phenylmethanimine is attacked by the 2-methylquinoline and is transformed into the corresponding olefination product via elimination of a molecule of NH₃.

Conclusion

In summary, we have developed an efficient approach for the synthesis of a variety of E-2-styrylquinolines through sp³ C-H cross-dehydrogenative coupling of benzylamines with 2-methylquinolines promoted by NH₄I under air without any metal catalyst. The approach provides relatively mild reaction conditions, moderate to good yields, and encompasses a broad substrate scope. A plausible mechanism has been proposed for the oxidative olefination through deamination.

Experimental

Infrared spectra were determined on a Nicolet Avatar-370 spectrometer in KBr (ν in cm⁻¹). Melting points were measured on a Büchi B-540 capillary melting point apparatus and are uncorrected. Mass spectra (ESI-MS) were recorded on a Thermo Finnigan LCQ-Advantage spectrometer. High-resolution mass spectra (ESI-HRMS) were obtained using an Agilent 6210 TOF instrument. ¹H NMR and ¹³C NMR spectra were recorded on a Varian Mercury Plus-400 spectrometer (400 and 100 MHz), δ in parts per million, J in Hertz, using TMS as the internal standard. Signal multiplicities are assigned as singlet (s), doublet (d), multiplet (m). All analytical reagents were commercially available and were used directly without further purification.

Synthesis of E-2-styrylquinolines (3a selected as an example; general procedure)

A mixture of 2-methylquinoline (1a) (0.14 g, 1 mmol), benzylamine (0.32 g, 3.0 mmol), and NH₄I (0.17 g, 1.2 mmol) in NMP (4 mL) was stirred at 160 °C for 10 h until the total consumption of 1a. After cooling, the reaction mixture was washed with brine (20 mL) and extracted with CH₂Cl₂ (2 × 20 mL). The combined organic extract was dried over Na₂SO₄ and concentrated under vacuum. The residue was purified by column chromatography (petroleum ether/ EtOAc = 6:1) to afford the target product 3a (pale yellow solid, 85%, 0.20 g).

2-[(1E)-2-phenylethenyl]quinoline (3a). Pale yellow solid; 85%, 0.20 g; m.p. 91–92 °C (Lit.²¹ 91–93 °C)²¹ 1H NMR
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(400 MHz, CDCl 3): δ = 8.14–8.06 (m, 2H), 7.74–7.64 (m, 5H), 7.55–7.46 (m, 1H), 7.43–7.38 (m, 3H), 7.34–7.30 (m, 1H). 13C NMR (100 MHz, CDCl 3): δ = 156.4, 148.2, 136.6, 136.3, 134.5, 129.7, 129.3, 129.0, 128.9, 128.6, 127.6, 127.5, 127.2, 126.3, 118.9. MS (ESI): m/z (%) = 232.1 ([M] +, 100).

HRMS (ESI): m/z [M] + calcd for C 17H14N: 232.1126, found: 232.1133.

3-Methyl-2-[(1E)-2-phenylethenyl]quinoline (3b). Pale yellow solid; 73%, 0.18 g; m.p. 98–100 °C (Lit. 98–100 °C).21 1H NMR (400 MHz, CDCl 3): δ = 8.09 (d, J = 8.4 Hz, 1H), 7.98 (d, J = 15.6 Hz, 1H), 7.82–7.73 (m, 3H), 7.51–7.43 (m, 1H), 7.33–7.25 (m, 1H), 7.22–7.17 (m, 1H), 2.32 (s, 3H). 13C NMR (100 MHz, CDCl 3): δ = 154.9, 146.9, 137.1, 136.2, 135.6, 129.4, 129.1, 128.7, 128.6, 128.5, 127.8, 127.5, 126.7, 125.8, 124.3, 19.6. MS (ESI): m/z (%) = 246.1 ([M] +, 100). HRMS (ESI): m/z [M] + calcd for C 18H16N: 246.1283, found: 246.1291.

3-Chloro-2-[(1E)-2-phenylethenyl]quinoline (3e). Pale yellow solid; 68%, 0.18 g; m.p. 109–111 °C (Lit. 109–111 °C).21 1H NMR (400 MHz, CDCl 3): δ = 8.19–8.16 (m, 1H), 8.09 (d, J = 8.4 Hz, 1H), 7.82–7.73 (m, 2H), 7.73–7.67 (m, 3H), 7.50–7.45 (m, 1H), 7.43–7.37 (m, 2H), 7.36–7.31 (m, 2H). 13C NMR (100 MHz, CDCl 3): δ = 156.1, 149.2, 142.7, 136.3, 135.3, 130.5, 129.8, 128.9, 128.7, 127.9, 127.3, 125.9, 123.7, 119.8, 19.2. MS (ESI): m/z (%) = 266.1 ([M] +, 100). HRMS (ESI): m/z [M] + calcd for C 17H13 ClN: 266.0736, found: 266.0743; C 17H13 ClN: 268.0707, found: 268.0716.

6-Methyl-2-[(1E)-2-phenylethenyl]quinoline (3f). Pale yellow solid; 82%, 0.20 g; m.p. 141–143 °C (Lit. 142–144 °C).21 1H NMR (400 MHz, CDCl 3): δ = 8.11 (d, J = 8.4 Hz, 1H), 7.97 (d, J = 8.4 Hz, 1H), 7.67–7.62 (m, 4H), 7.55–7.53 (m, 1H).
8. Methyl-2-[(1E)-2-phenylethenyl]quinoline (3l). Pale yellow solid; 54%, 0.13 g; m.p. 72–73 °C (Lit.21 72 °C).11 1H NMR (400 MHz, CDCl3): δ = 8.05 (d, J = 8.6 Hz, 1H), 7.76 (d, J = 16.2 Hz, 1H), 7.68–7.62 (m, 4H), 7.56 (d, J = 6.7 Hz, 1H), 7.43–7.31 (m, 5H), 2.76 (s, 3H).13C NMR (100 MHz, CDCl3): δ = 154.7, 147.2, 137.1, 136.7, 136.3, 137.7, 129.7, 129.4, 128.7, 128.4, 127.2, 127.1, 125.8, 125.3, 119.2, 18.4. MS (ESI): m/z (%) = 246.1 ([M]+, 100). HRMS (ESI): m/z [M]+ calcd for C18H14NO: 246.1283; found: 246.1289.

8-Chloro-2-[(1E)-2-phenylethenyl]quinoline (3m). Pale yellow solid; 95%, 0.16 g; m.p. 88–90 °C (Lit.16 88–90 °C).16 1H NMR (400 MHz, CDCl3): δ = 8.11 (d, J = 8.3 Hz, 1H), 7.80–7.75 (m, 2H), 7.64–7.68 (m, 4H), 7.47–7.31 (m, 5H).13C NMR (100 MHz, CDCl3): δ = 156.8, 144.3, 136.6, 136.4, 135.2, 133.1, 129.7, 128.8, 128.7, 127.3, 127.1, 126.4, 125.8, 119.6. MS (ESI): m/z (%) = 266.1 ([M]+, 75), 268.1 ([M]+, 25). HRMS (ESI): m/z [M]+ calcd for C18H14NO: 268.0736; found: 268.0745; C18H1535N: 268.0707; found: 268.0715.

2-[(1E)-2-phenylethenyl]quinoloxaline (3n). Pale yellow solid; 60%, 0.14 g; m.p. 101–103 °C (Lit.21 101–103 °C).15 1H NMR (400 MHz, CDCl3): δ = 9.01 (s, 1H), 8.08 (d, J = 7.9 Hz, 2H), 7.88 (d, J = 16.3 Hz, 1H), 7.75–7.62 (m, 4H), 7.44–7.33 (m, 4H).13C NMR (100 MHz, CDCl3): δ = 150.4, 144.7, 142.4, 141.6, 136.6, 135.9, 130.0, 129.4, 129.3, 129.2, 129.1, 128.9, 127.4, 127.3, 126.1, 118.0, 21.9. MS (ESI): m/z (%) = 233.1 ([M]+, 100). HRMS (ESI): m/z [M]+ calcd for C15H12N: 233.1079; found: 233.1086.

1-[(1E)-2-phenylethenyl]isoquinoline (3o). Pale yellow solid; 51%, 0.12 g; m.p. 97–99 °C (Lit.21 97–98 °C).21 1H NMR (400 MHz, CDCl3): δ = 8.58 (d, J = 5.6 Hz, 1H), 8.36 (d, J = 8.5 Hz, 1H), 8.01 (d, J = 1.2 Hz, 2H), 7.83 (d, J = 7.8 Hz, 1H), 7.72–7.66 (m, 3H), 7.65–7.62 (m, 1H), 7.57 (d, J = 5.5 Hz, 1H), 7.44–7.41 (m, 2H), 7.35–7.32 (m, 1H).13C NMR (100 MHz, CDCl3): δ = 154.6, 142.7, 136.9, 136.7, 135.8, 129.9, 128.7, 128.6, 127.4, 127.3, 126.7, 124.5, 122.8, 119.7. MS (ESI): m/z (%) = 232.1 ([M]+, 100). HRMS (ESI): m/z [M]+ calcd for C15H12N: 232.1107; found: 232.1134.

2-[(1E)-2-(2-Methyl)phenylethenyl]quinoline (3p). Pale yellow solid; 89%, 0.22 g; m.p. 69–71 °C (Lit.22 69–71 °C).21 1H NMR (400 MHz, CDCl3): δ = 8.30 (d, J = 8.4 Hz, 1H), 8.06–8.01 (m, 2H), 7.98–7.92 (m, 2H), 7.83–7.77 (m, 2H), 7.60–7.57 (m, 1H), 7.37 (d, J = 16.2 Hz, 1H), 7.27–7.21 (m, 3H), 2.48 (s, 3H).13C NMR (100 MHz, CDCl3): δ = 156.4, 148.2, 136.9, 136.8, 135.2, 131.9, 131.0, 130.4, 130.3, 129.1, 129.0, 128.2, 127.6, 126.9, 126.7, 126.2, 120.5, 20.0. MS (ESI): m/z (%) = 246.1 ([M]+, 100). HRMS (ESI): m/z [M]+ calcd for C18H14NO: 232.1126; found: 232.1126.
2-[(1E)-2-(3-Methyl)-phenyletheny]quinoline (3r). Pale yellow solid; 76%, 0.19 g; m.p. 68–69 °C (Lit. 16, 22 68–69 °C).
1H NMR (400 MHz, CDCl3): δ = 8.12 (d, J = 8.4 Hz, 1H), 8.07–48.0 (m, 1H), 7.72–7.67 (m, 2H), 7.58–7.53 (m, 2H), 7.52–7.46 (m, 1H), 7.41–317 (m, 4H), 7.01 (t, J = 8.0 Hz, 1H).
13C NMR (100 MHz, CDCl3): δ = 163.1 (d, J = 243 Hz), 155.2, 148.3 (d, J = 16.2 Hz, 1H), 136.3, 132.9 (d, J = 3 Hz), 130.2, 130.0, 129.6, 129.1, 129.2, 129.0, 128.3, 127.5, 126.7, 126.5, 126.2, 120.7, 21.1. MS (ESI): m/z (%) = 266.1 ([M]+, 75), 261.8 ([M]+, 25). HRMS (ESI): m/z [M]+ calc for C18H16N: 266.1032, found: 266.1034.

2-[(1E)-2-(4-Chloro)-phenyletheny]quinoline (3w). Yellow solid; 80%, 0.21 g; m.p. 141–143 °C (Lit. 16, 191–193 °C).
1H NMR (400 MHz, CDCl3): δ = 8.12 (d, J = 8.4 Hz, 2H), 8.12 (d, J = 8.4 Hz, 2H), 8.12 (d, J = 8.4 Hz, 2H), 7.86–7.71 (m, 6H), 7.56–7.45 (m, 5H). 13C NMR (100 MHz, CDCl3): δ = 156.2, 148.3, 136.4, 131.4, 131.3, 131.5, 131.3, 131.9, 129.3, 128.8, 128.6, 127.5, 127.4, 126.3, 126.2, 125.8, 125.7, 124.1, 123.8, 119.5. MS (ESI): m/z (%) = 282.1 ([M]+, 100). HRMS (ESI): m/z [M]+ calc for C21H16NCl: 282.1283, found: 282.1283.

2-[(1E)-2-(4-Chloro)-phenyletheny]quinoline (3v). Yellow solid; 86%, 0.21 g; m.p. 120–122 °C (Lit. 16, 120–122 °C).
1H NMR (400 MHz, CDCl3): δ = 8.10–8.06 (m, 2H), 7.78 (d, J = 8.0 Hz, 1H), 7.74–637 (m, 5H), 7.56–7.49 (m, 1H), 7.32 (d, J = 16.2 Hz, 1H), 7.13–77.0 (m, 2H). 13C NMR (100 MHz, CDCl3): δ = 163.0 (d, J = 246 Hz), 159.9, 148.3, 136.3, 133.4, 132.8 (d, J = 3 Hz), 129.8, 129.2, 128.9, 128.6 (d, J = 3 Hz), 127.6, 127.3, 126.4, 119.6, 115.9 (d, J = 22 Hz). MS (ESI): m/z (%) = 210.1 ([M]+, 100). HRMS (ESI): m/z [M]+ calc for C18H14ClN: 210.1032, found: 210.1040.
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ORCID iD
Xue Li https://orcid.org/0000-0002-9499-2033

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