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Copper-mediated formal [5+1] annulation of 2-vinylanilines and glyoxylic acid: A facile approach for the synthesis of 4-arylated quinolines

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ABSTRACT

A copper-mediated formal [5 + 1] oxidative annulation of 2-vinylanilines and glyoxylic acid to 4-arylated quinolines was developed. A series of 4-arylated quinoline derivatives were obtained in good to excellent yields. This protocol could be carried out efficiently on gram scale. The transformation probably underwent nucleophilic addition/6π electrocyclization/oxidative aromatization and the elimination of CO_2 cascade processes.

1. Introduction

Quinolines are privileged structural motifs, which can be found in many pharmaceuticals and biologically active natural products (Fig. 1) [1,2], such as Quinine, Aablaquine, Amodiaquine, Primaquine, Mefloquine, Pitavastain, Piperaquine and so on. Many quinoline-containing molecules possess diverse activities, such as antimalarial, anticancer, anti-inflammatory, anti-HIV, antibacterial, and antituberculosis activities [1e,2]. Owing to the great importance of quinoline and its derivatives, great efforts have been made on the development of efficient methods for the synthesis of them. The general protocols for the construction of quinoline ring including Skraup reaction [3], Gould–Jacobs [4], Doebner–von Miller [5], Niementowski [6], Knorr [7], Conrad–Limpach [8], Pfitzinger [9], Friedländer reaction [10], Povarov Reaction [11], and some other protocols [12,13]. Although great progresses have been made, the development of new strategies for more efficiently construction of quinoline scaffolds is still desirable.

Recently, decarboxylative coupling reaction is a valuable alternative to forming carbon-carbon or carbon-heteroatom bond [14], which has been widely used in organic synthesis. Readily available glyoxylic acid and its derivatives can serve as decarboxylative coupling reagent [15]. In 2012, Gooβen reported the decarboxylative allylation of glyoxylic acids with diallyl carbonate [16]. In 2017, Wang and co-works reported the formylation reactions using glyoxylic acid and diethoxyacetic acid as formyl equivalents [17]. Xu group developed an Ir-photoredox-catalyzed decarboxylative Michael addition [18]. In 2018, Tao group reported the Pd-catalyzed hydroformylation of terminal arylacetylenes using glyoxylic acid as formyl source [19]. Wu reported the 3-formylation of indoles via Ni-catalyzed dehydrogenative-decarboxylative coupling with glyoxylic acid [20]. Recently, Huang also reported the formylation of amines through electrochemical decarboxylation of glyoxylic acid [21].

2-Vinylanilines have been evidenced as versatile synthetic reagents in modern organic syntheses. A range of functionalized *N*-heterocyles, such as quinolones [13b,22], indoles [23], quinolone-2-thiones [24], cinnolines [25], indazoles [26], and 2-quinolinones

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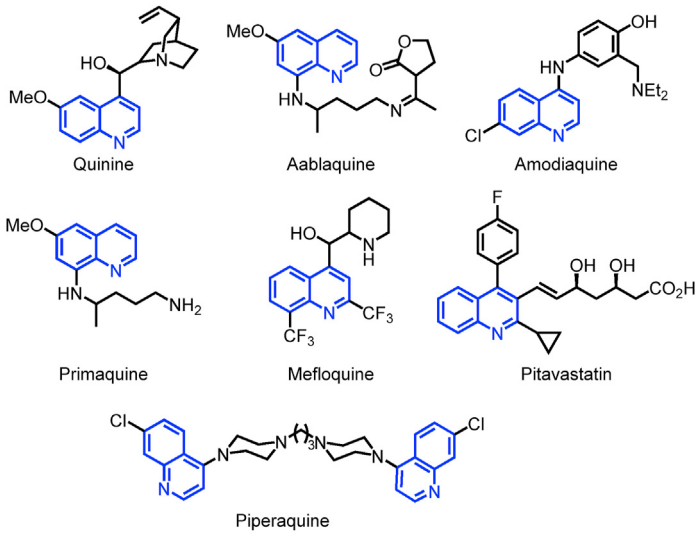


Fig. 1. Some quinoline-containing bioactive molecules.

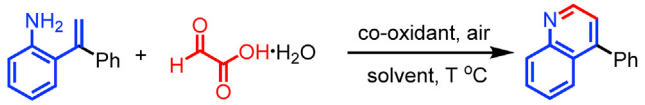
[27], have been prepared via cascade C(sp^2)-H activation/cyclization of 2-vinylanilines. Such transformations could proceed well, with or without transition-metal. For instance, a number of useful *N*-heterocycles have been obtained efficiently promoted by several kinds of transition-metal, such as Rh, Pd, Ru, and Cu catalysts. Recently, the group of Ma [22e], Cheng [13b], Yan [22c], Zeng [22a,b], and us [22d] described the transition-metal-catalyzed C(sp^2)-H activation/annulation of 2-vinylanilines to give functionalized quinolines. Meanwhile, the synthesis of some indoles, quinolone-2-thiones, cinnolines, and 2-quinolinones were reported under transition-metal-free conditions promoted by Brønsted acid or base. In 2018, Quan and co-workers reported the synthesis of quinoline-2-thiones via base-catalyzed thio-lactamization of 2-vinylanilines with CS_2 [24].

Promoted by the aforementioned information and our recent interest in the synthesis of functionalized quinoline derivatives [28], we herein report the copper-mediated oxidative annulation of 2-vinylanilines and glyoxylic acid to 4-arylated quinolines.

2. Results and discussion

2-(1-Phenylvinyl)aniline **1a** was chosen as a model substrate to optimize the reaction conditions using various co-oxidants and solvents. Firstly, we found that the desired product **3a** was obtained in 51% yield when the reaction was stirred in the presence of 1.5 equiv of Cu_2O in DMSO under air atmosphere for 1.5 h at 130 °C (Table 1, entry 1). Encouraged by this discovering, we next examined the effects of other Cu oxidants such as CuCl, CuI, $CuSO_4$, $CuCO_3{\cdot}Cu(OH)_2$, $Cu(NO_3)_2{\cdot}3H_2O$ and $Cu(OAc)_2$ (Table 1, entries 2–7). We found that $Cu(OAc)_2$ can provide quinoline **3a** in 90% yield (Table 1, entry 7). However, lower yields were obtained in the presence of other oxidants such as AgOAc and $K_2S_2O_8$ (Table 1, entries 8 and 9). Controlled experiment confirmed that it's necessity to obtain satisfied results in the presence of copper-oxidant (Table 1, entries 10 and 11). In addition, air also played important role in this transformation (Table 1, entry 12). Then, the evaluation of various other solvents, such as DMF, CH_3CN, 1,4-dioxane, EtOH, toluene, DCE and THF, showed that DMSO still was the best solvent for this reaction (Table, entries 13–19). Lowering the loading of $Cu(OAc)_2$ (50 mol%) produced inferior yield (59%, Table 1, entry 20). Finally, the reaction temperature was examined, and the yield could maintain a high level when the temperature was reduced to 80 °C (Table 1, entry 21, 90%). However, the yield was obviously decreased to 76% when the reaction was carried out at 50 °C (Table 1, entry 22).

Table 1
Optimization of the reaction conditions[a].



1a **2** **3a**

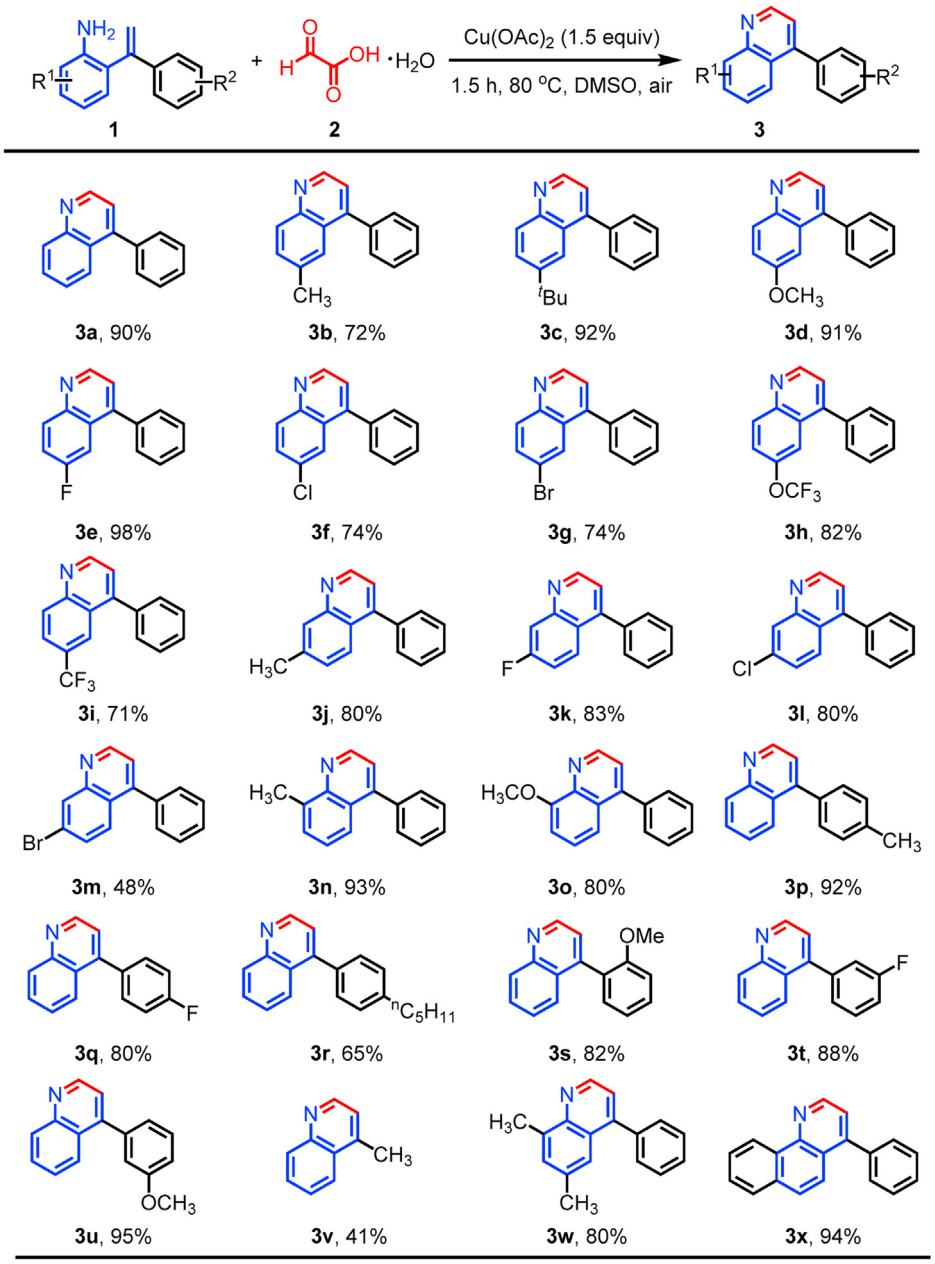
entry	co-oxidant (1.5 equiv)	solvent	yield %[b]
1	Cu_2O	DMSO	51
2	CuCl	DMSO	80
3	CuI	DMSO	trace
4	$CuSO_4$	DMSO	71
5	$CuCO_3{\cdot}Cu(OH)_2$	DMSO	53
6	$Cu(NO_3)_2{\cdot}3H_2O$	DMSO	47
7	$Cu(OAc)_2$	DMSO	90
8	AgOAc	DMSO	36
9	$K_2S_2O_8$	DMSO	31
10	–	DMSO	trace
11[c]	–	DMSO	N.R.
12[d]	$Cu(OAc)_2$	DMSO	74
13	$Cu(OAc)_2$	DMF	72
14	$Cu(OAc)_2$	CH_3CN	36
15	$Cu(OAc)_2$	1,4-dioxane	35
16	$Cu(OAc)_2$	EtOH	18
17	$Cu(OAc)_2$	toluene	28
18	$Cu(OAc)_2$	DCE	75
19	$Cu(OAc)_2$	THF	trace
20	$Cu(OAc)_2$ (0.5 equiv)	DMSO	59
21	$Cu(OAc)_2$	DMSO (80 °C)	90
22	$Cu(OAc)_2$	DMSO (50 °C)	76

[a] Reaction conditions: **1a** (0.2 mmol), glyoxylic acid **2** (0.4 mmol, 2.0 equiv), co-oxidant (1.5 equiv), and solvent (2 mL) with stirring at 130 °C for 1.5 h under air atmosphere.
[b] Isolated yields of **3a**.
[c] Under oxygen (O_2) condition.
[d] Under nitrogen (N_2) condition.

With the optimized reaction conditions in hand, we investigated the scope and limitation of 2-vinylanilines **1** (Table 2). Generally, the results showed that the method was successfully applied to a wide range of 2-vinylanilines **1**. Substrates bearing both electron-donating (R^1 = Me, ^{t}Bu, OCH_3) and electron-withdrawing (R^1 = F, Cl, Br, CF_3, OCF_3) groups on the anilines all worked well to afford the desired products in good to excellent yields. For example, products **3c** and **3d** with tertiary butyl and methoxy group were obtained in 92% and 91%, respectively. Substrates containing fluoro and trifluoromethoxy also provided the desired products **3e** and **3h** both in high yields. Substitutes (R^1) at the *meta* and *ortho* positions of the anilines showed little effect on the efficiency, leading to the corresponding quinolines (**3j-3o**) in moderate to good yields. Next, we investigated the substitute effect of R^2 under the same reaction conditions. A range of functional group such as methyl, fluoro and methoxy were tolerated, providing the desired products (**3p-3u**) in good yields. While the yield of 4-(4-pentylphenyl) quinolone (**3r**) is relative low 65%. It is worthwhile to note that 2-(prop-1-en-2-yl) aniline also carried out smoothly to afford the desired products (**3v**) in moderate yield (41%). Furthermore, 2, 4-dimethyl-6-(1-phenylvinyl) aniline was amenable for the present protocol, affording the product **3w** in 80% yield. 4-Phenylbenzo[*h*]quinoline (**3x**) was obtained in 94% yield from corresponding substrate.

Then, to illustrate the synthetic utility of this transformation, a gram-scale synthesis of quinoline **3a** was performed (Scheme 1). The result showed that the approach could be carried out on gram

Table 2
Substrate scope[a,b].



[a]The reactions were carried out with **1** (0.2 mmol), $Cu(OAc)_2$ (0.3 mmol), and $HCOCO_2H{\cdot}H_2O$ **2** (0.4 mmol) in DMSO (2 mL) at 80 °C for 1.5 h. [b]Isolated yields.

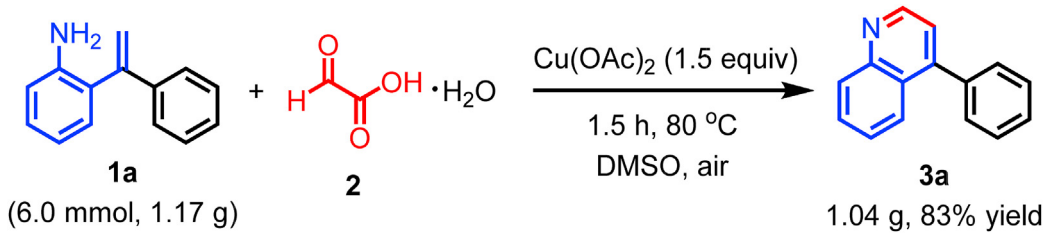
[a] The reactions were carried out with **1** (0.2 mmol), $Cu(OAc)_2$ (0.3 mmol), and $HCOCO_2H{\cdot}H_2O$ **2** (0.4 mmol) in DMSO (2 mL) at 80 °C for 1.5 h.
[b] Isolated yields.

scale (6.0 mmol) smoothly to give the desired product **3a** in 83% yield.

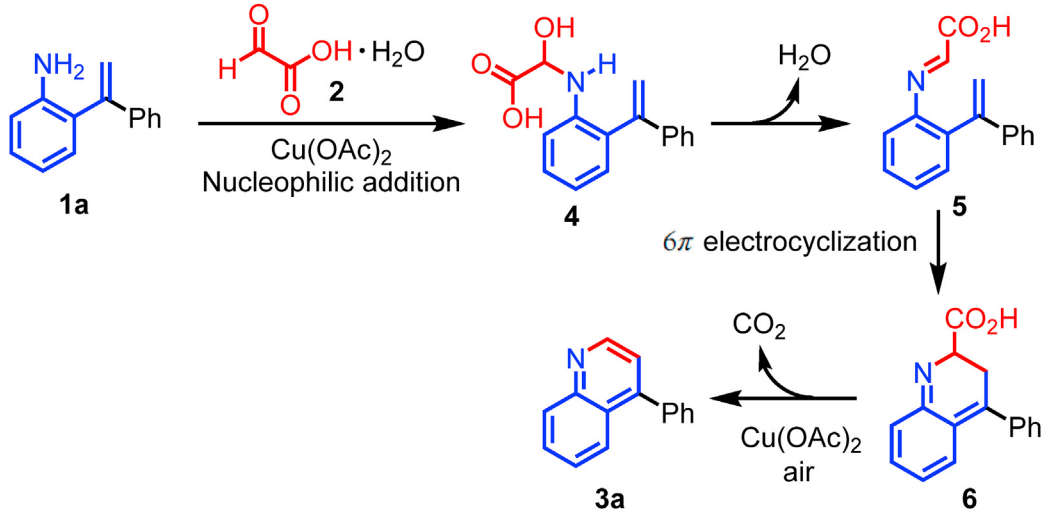
Based on previous reports [15–22] and the results of our control experiments, a plausible mechanism for the synthesis of the 4-phenylquinoline is illustrated in Scheme 2. Initially, the Lewis acid $Cu(OAc)_2$-catalyzed nucleophilic addition of 2-vinylanilines **1a** and glyoxylic acid **2** led to intermediate **4**, followed by the release of H_2O and formed the intermediate imine **5**, which was observed by HRMS. Subsequently, imine **5** can undergo a 6π electrocyclization to provide 2,3-dihydroquinoline **6**. Finally, the desired product **3a** is formed from **6** via copper salt-mediated oxidative aromatization and elimination of CO_2 under air atmosphere.

3. Conclusion

In summary, we have developed a facile approach for the construction of 4-aryl- and 4-methyl-quinolines from 2-vinylanilines and commercially available glyoxylic acid. A broad range of 2-



Scheme 1. Gram-scale reaction.



Scheme 2. Proposed mechanism for the synthesis of 4-arylated quinoline.

vinylanilines were served as effective substrates, affording to the corresponding quinolines in good to excellent yields. The transformation could be carried out efficiently on gram scale. Further investigations on bio-activities of these 4-arylated quinoline derivatives are currently underway in our laboratory.

4. Experimental section

Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra were recorded on a Bruker AV-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II instrument.

*4.1. Typical procedure for the synthesis of 4-arylated quinolines from 2-vinylanilines **1** and glyoxylic acid **2***

To a 25 mL Schlenk tube equipped with a stir bar, 2-vinylaniline **1** (0.2 mmol), glyoxylic acid **2** (0.4 mmol) and $Cu(OAc)_2$ (0.3 mmol) in DMSO (2 mL) were added, the mixture was stirred at 80 °C for 1.5 h. After the completion of the reaction, monitored by TLC, the mixture was cooled to room temperature. The residue was diluted with H_2O solution (10 mL) and extracted with dichloromethane (3 × 10 mL). The solvent concentrated via reduced pressure, and the residue was purified by silica gel chromatography with petroleum ether/ethyl acetate (1/5) as the eluent to obtain the product **3**.

*4.2. 4-Phenylquinoline (**3a**). Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow liquid (36.1 mg, 88% yield)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.95 (s, 1H), 8.18 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.4 Hz, 1H), 7.72 (t, *J* = 7.2 Hz, 1H), 7.56–7.46 (m, 6H), 7.32 (d, *J* = 4.0 Hz, 1H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 150.0, 148.7, 148.5, 138.0, 129.9, 129.6, 129.3, 128.6, 128.4, 126.6, 125.9, 121.4. HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{15}H_{12}N^+$: 206.0964, found: 206.0969.

*4.3. 6-Methyl-4-phenylquinoline (**3b**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (31.5 mg, 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (d, *J* = 4.4 Hz, 1H), 8.07 (d, *J* = 8.6 Hz, 1H), 7.66 (s, 1H), 7.57–7.48 (m, 6H), 7.28 (d, *J* = 4.4 Hz, 1H), 2.46 (s, 3H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 149.1, 147.8, 147.3, 138.3, 136.5, 131.6, 129.6, 129.5, 128.6, 128.3, 126.7, 124.6, 121.4, 21.8. HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{16}H_{14}N^+$: 220.1121, found: 220.1125.

*4.4. 6-(tert-butyl)-4-phenylquinoline (**3c**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (48.2 mg, 92.3% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.88 (d, *J* = 4.4 Hz, 1H), 8.12 (d, *J* = 8.8 Hz, 1H), 7.88 (s, 1H), 7.83 (d, *J* = 8.8 Hz, 1H), 7.55–7.45 (m, 5H), 7.30 (d, *J* = 4.4 Hz, 1H), 1.34 (s, 9H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 149.4, 149.3, 148.3, 147.2, 138.3, 129.5, 129.3, 128.5, 128.4, 128.2, 126.3, 121.4, 120.7, 35.1, 31.1. HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{19}H_{20}N^+$: 262.1590, found: 262.1581.

4.5. 6-Methoxy-4-phenylquinoline (**3d**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow solid (42.4 mg, 91% yield); mp: 62–64 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.79 (d, J = 4.4 Hz, 1H), 8.07 (d, J = 9.2 Hz, 1H), 7.55–7.47 (m, 5H), 7.38 (dd, J = 9.2, 2.8 Hz, 1H), 7.27 (d, J = 4.4 Hz, 1H), 7.19 (d, J = 2.8 Hz, 1H), 3.78 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 157.9, 147.5, 147.1, 144.8, 138.4, 131.3, 129.3, 128.7, 128.4, 127.7, 121.8, 121.7, 103.7, 55.4. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{16}H_{14}NO^+$: 236.1070, found: 236.1071.

4.6. 6-Fluoro-4-phenylquinoline (**3e**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow liquid (43.5 mg, 98% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.91 (d, J = 4.4 Hz, 1H), 8.17 (dd, J = 9.2, 5.6 Hz, 1H), 7.59–7.43 (m, 7H), 7.35 (d, J = 4.4 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 160.7 (d, $^1J_{C\text{-}F}$ = 246 Hz), 149.3, 148.0, 145.9, 137.6, 132.4 (d,$^3J_{C\text{-}F}$ = 9.2 Hz), 129.3, 128.8, 128.7, 127.6 (d, $^3J_{C\text{-}F}$ = 9.4 Hz), 121.8, 119.7 (d, $^2J_{C\text{-}F}$ = 25.7 Hz), 109.2 (d, $^2J_{C\text{-}F}$ = 23.0 Hz). HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{11}FN^+$: 224.0870, found: 224.0873.

4.7. 6-Chloro-4-phenylquinoline (**3f**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a white solid (35.5 mg, 74% yield); mp: 62–64 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92 (d, J = 4.4 Hz, 1H), 8.11 (d, J = 9.0 Hz, 1H), 7.88 (d, J = 2.4 Hz, 1H), 7.65 (dd, J = 9.0, 2.0 Hz, 1H), 7.57–7.50 (m, 3H), 7.49–7.45 (m, 2H), 7.34 (d, J = 4.4 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.1, 147.8, 147.1, 137.3, 132.6, 131.5, 130.3, 129.4, 128.8, 128.7, 127.5, 124.7, 122.1. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{11}ClN^+$: 240.0575, found: 240.0579.

4.8. 6-Bromo-4-phenylquinoline (**3g**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a white solid (41.6 mg, 74% yield); mp: 59–61 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.94 (d, J = 4.4 Hz, 1H), 8.05 (s, 1H), 8.04 (d, J = 10.8 Hz, 1H), 7.79 (dd, J = 9.0, 2.2 Hz, 1H), 7.56–7.50 (m, 3H), 7.49–7.45 (m, 2H), 7.35 (d, J = 4.4 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.3, 147.7, 147.3, 137.3, 132.9, 131.6, 129.4, 128.8, 128.7, 128.0, 122.1, 120.9. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{11}BrN^+$: 284.0069, found: 284.0070.

4.9. 4-Phenyl-6-(trifluoromethoxy)quinoline (**3h**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow solid (47.3 mg, 82% yield); mp: 64–66 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.97 (d, J = 3.0 Hz, 1H), 8.22 (d, J = 9.2 Hz, 1H), 7.75 (s, 1H), 7.61–7.48 (m, 6H), 7.40 (d, J = 4.3 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.4, 148.5, 147.2, 147.0, 137.2, 132.1, 129.4, 128.9, 127.1, 123.3, 122.1, 120.5 (q, $^1J_{C\text{-}F}$ = 256.3 Hz), 116.6. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{16}H_{11}F_3NO^+$: 290.0787, found: 290.0780.

4.10. 4-Phenyl-6-(trifluoromethyl)quinoline (**3i**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow solid (38.3 mg, 71% yield); mp: 61–63 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.04 (d, J = 4.4 Hz, 1H), 8.29 (d, J = 8.8 Hz, 1H), 8.24 (s, H), 7.90 (d, J = 8.8 Hz, 1H), 7.59–7.48 (m, 5H), 7.43 (d, J = 4.4 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 151.9, 149.7, 149.5, 137.0, 131.2, 129.4, 129.0, 128.9, 128.5 (q, $^2J_{C\text{-}F}$ = 32.3 Hz), 125.9, 125.0 (q, $^3J_{C\text{-}F}$ = 3.0 Hz), 124.0 (q, $^1J_{C\text{-}F}$ = 270.8 Hz), 123.9 (q, $^3J_{C\text{-}F}$ = 4.5 Hz), 122.4. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{16}H_{11}F_3N^+$: 274.0838, found: 274.0840.

4.11. 7-Methyl-4-phenylquinoline (**3j**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow solid (34.9 mg, 80% yield); mp: 66–68 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84 (d, J = 4.4 Hz, 1H), 8.06 (d, J = 8.4 Hz, 1H), 7.59 (dd, J = 8.2, 7.2 Hz, 1H), 7.47–7.39 (m, 3H), 7.32–7.20 (m, 3H), 7.19 (d, J = 4.0 Hz, 1H), 2.01 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 149.8, 149.0, 148.6, 142.4, 135.7, 129.9, 128.9, 128.7, 127.9, 127.8, 126.3, 123.5, 24.5. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{16}H_{14}N^+$: 220.1121, found: 220.1120.

4.12. 7-Fluoro-4-phenylquinoline (**3k**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (36.8 mg, 83% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.91 (s, 1H), 8.01 (d, J = 8.4 Hz, 1H), 7.68–7.60 (m, 1H), 7.45–7.37 (m, 5H), 7.26 (s, 1H), 7.14 (t, J = 8.4 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.3 (d, $^1J_{C\text{-}F}$ = 257.3 Hz), 150.3, 150.0, 146.2, 140.4, 129.0 (d, $^2J_{C\text{-}F}$ = 9.4 Hz), 128.5 (d, $^3J_{C\text{-}F}$ = 3.7 Hz), 128.0, 127.7, 126.1 (d, $^3J_{C\text{-}F}$ = 4.1 Hz), 123.6, 117.5, 111.9 (d, $^2J_{C\text{-}F}$ = 21.7 Hz). HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{11}FN^+$: 224.0870, found: 224.0875.

4.13. 7-Chloro-4-phenylquinoline (**3l**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (38 mg, 80% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.90 (d, J = 4.4 Hz, 1H), 8.13 (d, J = 8.4 Hz, 1H), 7.63–7.54 (m, 2H), 7.44–7.37 (m, 3H), 7.34–7.27 (m, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.1, 149.6, 148.3, 140.8, 130.8, 129.7, 129.6, 128.9, 127.8, 127.7, 124.9, 124.6. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{11}ClN^+$: 240.0575, found: 240.0580.

4.14. 7-Bromo-4-phenylquinoline (**3 m**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow liquid (27.1 mg, 48% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.94 (d, J = 4.4 Hz, 1H), 8.18 (d, J = 8.4 Hz, 1H), 7.92 (d, J = 8.4 Hz, 1H), 7.73 (t, J = 8.0 Hz, 1H), 7.55–7.46 (m, 5H), 7.34 (d, J = 4.4 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 149.9, 148.7, 148.5, 138.0, 129.8, 129.5, 129.3, 128.6, 128.4, 126.8, 126.6, 125.9, 121.3. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{11}BrN^+$: 284.0069, found:284.0070.

4.15. 8-Methyl-4-phenylquinoline (**3n**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow liquid (40.5 mg, 93% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.97 (d, J = 4.4 Hz, 1H), 7.75 (d, J = 8.4 Hz, 1H), 7.57 (d, J = 6.8 Hz, 1H), 7.53–7.46 (m, 5H), 7.37 (dd, J = 8.4, 7.2 Hz, 1H), 7.32 (d, J = 4.4 Hz, 1H), 2.87 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.7, 147.8, 138.5, 137.4, 129.6, 129.5, 128.5, 128.3, 126.8, 126.3, 124.0, 121.2, 18.6. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{16}H_{14}N^+$: 220.1121, found: 220.1123.

4.16. 8-Methoxy-4-phenylquinoline (**3o**)

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:2) as a yellow solid (37.6 mg, 80% yield); mp: 103–105 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 8.95 (d, J = 4.4 Hz, 1H), 7.52–7.42 (m, 6H), 7.39 (t, J = 8.0 Hz, 1H), 7.34 (d, J = 4.4 Hz, 1H), 7.05 (d, J = 7.2 Hz, 1H), 4.10 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 155.6, 148.7, 148.3, 140.7, 138.3, 129.5, 128.5, 128.3, 127.8, 126.6, 122.0, 117.6, 107.4, 56.1. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{16}H_{14}NO^+$: 236.1070, found: 236.1079.

*4.17. 4-(p-tolyl)quinoline (**3p**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (40.1 mg, 92% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92 (d, J = 4.4 Hz, 1H), 8.17 (d, J = 8.4 Hz, 1H), 7.94 (d, J = 8.4 Hz, 1H), 7.71 (t, J = 8.4 Hz, 1H), 7.48 (t, J = 8.4 Hz, 1H), 7.40 (d, J = 8.0 Hz, 2H), 7.35–7.30 (m, 3H), 2.46 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.0, 148.7, 148.6, 138.4, 135.1, 129.8, 129.5, 129.3, 129.2, 126.9, 126.5, 126.0, 121.3, 21.3. HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{16}H_{14}N^+$: 220.1121, found: 220.1126.

*4.18. 4-(4-fluorophenyl)quinoline (**3q**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow liquid (35.6 mg, 80% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.94 (s, 1H), 8.18 (d, J = 8.4 Hz, 1H), 7.87 (d, J = 8.4 Hz, 1H), 7.73 (dd, J = 8.0, 7.2 Hz, 1H), 7.54–7.45 (m, 3H), 7.31 (d, J = 4.2 Hz, 1H), 7.22 (t, J = 8.4 Hz, 2H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 162.5 (d, $^1J_{C-F}$ = 246.8 Hz), 149.9, 148.7, 147.4, 133.9 (d, $^3J_{C-F}$ = 3.2 Hz), 131.3 (d, $^2J_{C-F}$ = 8.2 Hz), 130.0, 129.4, 126.8, 125.6, 121.4, 115.7 (d, $^2J_{C-F}$ = 21.4 Hz). HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{15}H_{11}FN^+$: 224.0870, found: 224.0875.

*4.19. 4-(4-pentylphenyl)quinoline (**3r**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow liquid (35.3 mg, 65% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92 (d, J = 4.4 Hz, 1H), 8.17 (d, J = 8.0 Hz, 1H), 7.96 (dd, J = 8.4, 0.8 Hz, 1H), 7.75–7.67 (m, 1H), 7.60–7.46 (m, 1H), 7.44–7.39 (m, 2H), 7.35–7.32 (m, 3H), 2.74–2.68 (m, 2H), 1.70 (d, J = 7.8 Hz, 2H), 1.44–1.36 (m, 4H), 0.96–0.90 (m, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 149.9, 148.7, 148.6, 143.4, 135.3, 129.8, 129.4, 128.6, 126.9, 126.5, 126.0, 121.3, 35.8, 31.6, 31.1, 22.6, 14.0. HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{20}H_{22}N^+$: 276.1747, found: 276.1750.

*4.20. 4-(2-methoxyphenyl)quinoline (**3s**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a white solid (38.4 mg, 82% yield); mp: 89–92 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (d, J = 4.4 Hz, 1H), 8.15 (d, J = 8.4 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 7.61 (d, J = 8.4 Hz, 1H), 7.47–7.40 (m, 2H), 7.31 (d, J = 4.0 Hz, 1H), 7.25 (d, J = 7.2 Hz, 1H), 7.08 (d, J = 7.6 Hz, 1H), 7.04 (d, J = 8.4 Hz, 1H), 3.68 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 156.8, 149.9, 148.4, 145.9, 131.2, 130.0, 129.6, 129.1, 127.5, 126.8, 126.4, 126.2, 122.2, 120.7, 111.2, 55.5. HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{16}H_{14}NO^+$: 236.1070, found: 236.1072.

*4.21. 4-(3-fluorophenyl)quinoline (**3t**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (39.2 mg, 88% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.94 (d, J = 4.4 Hz, 1H), 8.19 (d, J = 8.4 Hz, 1H), 7.88 (d, J = 8.4 Hz, 1H), 7.73 (t, J = 7.6 Hz, 1H), 7.54–7.45 (m, 2H), 7.31 (d, J = 4.0 Hz, 1H), 7.27 (d, J = 7.6 Hz, 1H), 7.23–7.16 (m, 2H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 162.7 (d, $^1J_{C-F}$ = 245.9 Hz, 1H), 149.9, 148.7, 147.1, 140.1 (d, $^3J_{C-F}$ = 7.6 Hz), 130.2 (d, $^3J_{C-F}$ = 8.4 Hz), 130.0, 129.5, 126.9, 126.4, 125.5, 125.3 (d, $^4J_{C-F}$ = 3.0 Hz, 1H), 121.2, 116.6 (d, $^2J_{C-F}$ = 22 Hz), 115.4 (d, $^2J_{C-F}$ = 20.9 Hz). HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{15}H_{11}FN^+$: 224.0870, found: 224.0874.

*4.22. 4-(3-methoxyphenyl)quinolone (**3u**)*

Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (44.7 mg, 95.1% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (d, J = 4.4 Hz, 1H), 8.18 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 8.4 Hz, 1H), 7.72 (t, J = 8.0 Hz, 1H), 7.50 (t, J = 8.0 Hz, 1H), 7.43 (t, J = 8.0 Hz, 1H), 7.34 (d, J = 4.4 Hz, 1H), 7.08 (d, J = 7.6 Hz, 1H), 7.05–7.01 (m, 2H), 3.86 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 159.7, 149.9, 148.7, 148.4, 139.4, 129.8, 129.7, 129.3, 126.8, 126.6, 126.0, 122.0, 121.2, 115.2, 113.9, 55.4. HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{16}H_{14}NO^+$: 236.1070, found: 236.1072.

*4.23. 4-Methylquinoline (**3v**). Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a colorless liquid (12 mg, 41% yield)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (d, J = 4.4 Hz, 1H), 8.11 (d, J = 8.4 Hz, 1H), 7.98 (d, J = 8.4 Hz, 1H), 7.70 (t, J = 7.6 Hz, 1H), 7.55 (t, J = 7.6 Hz, 1H), 7.21 (d, J = 4.0 Hz, 1H), 2.69 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.1, 148.0, 144.3, 130.0, 129.1, 128.3, 126.3, 123.8, 121.8, 18.6. HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{10}H_{10}N^+$: 144.0808, found: 144.0812.

*4.24. 6,8-Dimethyl-4-phenylquinoline (**3w**). Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a yellow liquid (37.2 mg, 80% yield)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (d, J = 4.4 Hz, 1H), 7.51–7.45 (m, 6H), 7.41 (s, 1H), 7.26 (d, J = 4.4 Hz, 1H), 2.83 (s, 3H), 2.40 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.0, 147.8, 146.4, 138.7, 137.0, 136.0, 131.9, 129.6, 128.5, 128.1, 126.8, 122.6, 121.3, 21.8, 18.5. HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{17}H_{16}N^+$: 234.1277, found: 234.1280.

*4.25. 4-phenylbenzo[h]quinoline (**3x**). Isolated (R_f = 0.4, EtOAc − petroleum ether = 1:5) as a white solid (47.9 mg, 94% yield); mp: 111–113 °C*

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (d, J = 4.4 Hz, 1H), 7.94 (d, J = 9.0 Hz, 1H), 7.86 (d, J = 9.0 Hz, 1H), 7.75 (d, J = 7.6 Hz, 1H), 7.57 (d, J = 8.4 Hz, 1H), 7.41–7.34 (m, 4H), 7.31–7.27 (m, 2H), 7.22 (d, J = 4.8 Hz, 1H), 7.04 (t, J = 8.0 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.6, 147.4, 147.3, 141.6, 131.9, 130.3, 128.7, 128.2, 127.6, 127.5, 127.2, 127.1, 127.0, 125.6, 124.4, 123.3, 122.9. HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{19}H_{14}N^+$: 256.1121, found: 256.1125.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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