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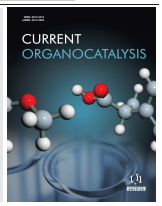
LETTER ARTICLE

Microwave-assisted, [Bmim]HSO_4-catalyzed the Friedländer Quinoline Synthesis of Quinoline Under Solvent-free Conditions



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ARTICLE HISTORY

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Abstract: An efficient and green method for the Friedländer quinoline synthesis has been described. The synthesis was performed under microwave irradiation using ionic liquid [Bmim]HSO_4 as a catalyst. A diverse range of quinoline derivatives was obtained in high yields from 2-aminoaryl aldehydes and ketones under solvent-free conditions.

Keywords: 2-aminoaryl aldehydes, ketones, condensation, electron-donating, irradiation, ionic liquid.

1. INTRODUCTION

Quinoline-containing compounds appearing in both natural and synthetic products are one of the most important classes of heterocyclic compounds. Quinoline derivatives have a broad spectrum of bioactivities, such as antimalarial, anti-bacterial, anthelmintic, anticonvulsant, antiviral, anti-inflammatory and analgesic activity [1, 2]. Some substituted quinolines have been marketed as drugs for treatment of various diseases, such as quinine, chloroquine, primaquine, santoquine, pentaquine, chloroxin, rosoxacin, and lomefloxacin (Fig. **1**). Considering these physiological activities of quinolines, their syntheses have received intensive research interest and various synthesis approaches have been developed.

The Friedländer condensation constitutes one of the most useful and straightforward methods for the synthesis of quinoline derivatives. The reaction usually starts with a 2-aminoaryl aldehyde or ketone and an aldehyde or ketone containing an α- methylene group [3, 4]. Various catalysts have been developed for this reaction including bases, acids, Lewis acids, nanomaterials and transition metal complexes [5, 6]. Ionic liquid, a green solvent for organic synthesis, has also well been applied for the Friedländer condensation reaction [7-9]. Recently, the use of microwave technique for the Friedländer quinoline synthesis has been investigated [10, 11]. The technique usually delivers many advantages, such as high yields, simple work-up, clean reaction pathways, and short reaction time [12]. In many cases, microwave irradiation coupled with solvent-free techniques represents a powerful, eco-friendly, green alternative to conventional synthesis. Herein, we report the Friedländer synthesis of quinolines using [Bmim]HSO_4 as a catalyst under microwave irradiation and solvent-free conditions. This is the first time the use of microwave technique in combination with [Bmim]HSO_4 catalyst is applied for the Friedländer condensation. Advantages of the synthesis include short reaction time, environmentally friendly protocol, high efficiency, and broad substrate scope.

2. MATERIALS AND METHOD

All ketones, 2-aminobenzaldehydes, and the ionic liquid [Bmim]HSO_4 were purchased from Sigma- Aldrich, Merck, and Fluka companies. Microwave reactions were performed in a CEM microwave reactor at 100 °C, 100 W in a 3 mL capped vial. ^{1}H and ^{13}C NMR spectra were recorded on a Brucker Advance-III 500 NMR Spectrometer (^{1}H NMR running at 500 MHz and ^{13}C NMR running at 125 MHz) instrument. $CDCl_3$, was used as the NMR solvent. Data for ^{1}H NMR are reported as follows: Chemical shift (δ ppm), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant and assignment. Data for ^{13}C NMR are reported in terms of chemical shift (δ ppm).

3. EXPERIMENTAL

3.1. General Procedure for the Quinoline Synthesis

A mixture of 2-aminobenzaldehyde (1 mmol) and ketone (1 mmol, 1 equiv.) was placed in a 3 mL microwave vial and [Bmim]HSO_4 (0.4 mmol, 0.4 equiv.) was added. The reaction mixture was left stirring under microwave irradiation (initial setting at 100 °C, 100 W) for 30 minutes. After completion of the reaction, the reaction mixture was cooled to and the residue was purified by column chromatography using solvent system of *n*-hexane/ethyl acetate as the eluent to give the desired product.

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3.2. Supplementary Data

3.2.1. 2-phenylquinoline (3a)

White solid; 189 mg, 92% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.19-8.13 (m, 4H), 7.84-7.78 (m, 2H), 7.74-7.68 (m, 1H), 7.53-7.43 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.3, 148.2, 139.6, 136.7, 129.7, 129.5, 129.3, 128.7, 127.5, 127.3, 127.1, 126.2, 118.9. NMR data are consistent with literature report [13].

3.2.2. 2-(p-tolyl)quinoline (3b)

White solid; 206 mg, 94% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.15 (d, J = 8.5 Hz, 2H), 8.06 (d, J = 8.0 Hz, 2H), 7.84-7.78 (m, 2H), 7.73-7.68 (m, 1H), 7.52-7.46 (m, 1H), 7.31 (d, J = 8.0 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.3, 148.2, 139.3, 136.8, 136.5, 129.7, 129.5, 127.4, 127.0, 126.0, 118.7, 21.3. NMR data are consistent with literature report [14].

3.2.3. 2-(o-tolyl)quinoline (3c)

White solid; 197 mg, 90% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16 (t, J = 8.5 Hz, 2H), 7.82-7.78 (m, 1H), 7.73-7.69 (m, 1H), 7.54-7.49 (m, 3H), 7.33-7.27 (m, 3H), 2.41 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.1, 147.7, 140.6, 135.9, 135.8, 130.7, 129.5, 129.4, 128.3, 127.4, 126.5, 126.2, 125.8, 122.2, 20.2. NMR data are consistent with literature report [15].

3.2.4. 2-(m-tolyl)quinoline (3d)

Yellow oil; 199 mg, 91% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16 (t, J = 8.5 Hz, 2H), 7.99 (s, 1H), 7.91 (d, J = 7.5 Hz, 1H), 7.83-7.76 (m, 2H), 7.73-7.68 (m, 1H), 7.52-7.48 (m, 1H), 7.41 (t, J = 8.5 Hz, 1H), 7.26 (d, J = 7.5 Hz, 1H), 2.46 (s, 3H); 13C NMR (125 MHz, CDCl3) δ: 157.5, 148.1, 139.5, 138.4, 136.6, 130.0, 129.6, 129.5, 128.6, 128.1, 127.3, 127.1, 126.1, 124.6, 119.0, 21.5. NMR data are consistent with literature report [16].

3.2.5. 2-(4-methoxyphenyl)quinoline (3e)

White solid; 232 mg, 95% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16-8.10 (m, 4H), 7.83-7.76 (m, 2H), 7.71-7.66 (m, 1H), 7.51-7.45 (m, 1H), 7.06-7.02 (m, 2H), 3.86 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.7, 156.8, 148.2, 136.5, 132.1, 129.5, 129.46, 128.8, 127.3, 126.8, 125.8, 118.4, 114.1, 55.3. NMR data are consistent with literature report [13].

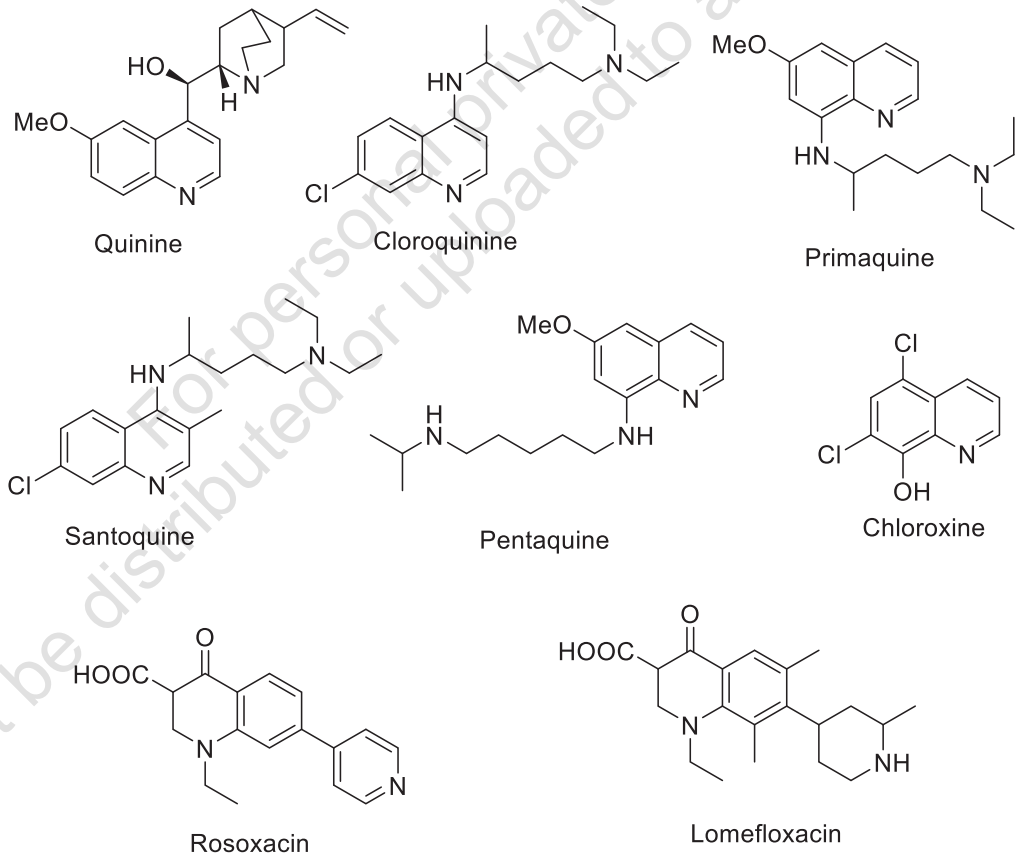


Fig (1). Representative quinoline-based compounds as pharmaceuticals.

3.2.6. 2-(4-chlorophenyl)quinoline (3f)

White solid; 213 mg, 80% yield. ^{1}H NMR (500 MHz, $CDCl^3$) δ: 8.17-8.09 (m, 4H), 7.79 (d, *J* = 8.5 Hz, 2H), 7.74-7.69 (m, 1H), 7.54-7.45 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.8, 148.1, 138.0, 136.8, 135.4, 129.7, 129.6, 128.9, 128.7, 127.4, 127.1, 126.4, 118.4. NMR data are consistent with literature report [14].

3.2.7. 2-(4-bromophenyl)quinoline (3g)

White solid; 249 mg, 88% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.18-8.15 (m, 2H), 8.06-8.02 (m, 2H), 7.80 (d, *J* = 8.5 Hz, 2H), 7.74-7.69 (m, 1H), 7.66-7.61 (m, 2H), 7.54-7.49 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.9, 148.1, 138.4, 136.9, 131.9, 129.8, 129.6, 129.0, 127.4, 127.1, 126.4, 123.8, 118.4. NMR data are consistent with literature report [14].

3.2.8. 2-(4-nitrophenyl)quinoline (3h)

Yellow solid; 215 mg, 86%. ^{1}H NMR (400 MHz, CDCl3) δ: 8.40-8.25 (m, 5H), 8.19-8.19 (m, 1H), 7.91-7.85 (m, 2H), 7.80-7.76 (m, 1H), 7.61-7.57 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.4, 148.22, 148.18, 145.3, 137.2, 130.1, 129.8, 128.2, 127.5, 127.2, 123.9, 118.6. NMR data are consistent with literature report [15].

3.2.9. 3-methyl-2-phenylquinoline (3i)

Pale-yellow oil; 43.3 mg, 84% yield. 1H NMR (500 MHz, $CDCl_3$) δ: 8.12 (d, *J* = 8.5 Hz, 1H), 7.98 (s, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.68-7.62 (m, 1H), 7.59-7.56 (m, 2H), 7.51-7.38 (m, 4H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.4, 146.5, 140.8, 136.6, 129.2, 129.1, 128.7, 128.6, 128.2, 128.1, 127.5, 126.6, 126.3, 20.5. NMR data are consistent with literature report [16].

3.2.10. 2-(tert-butyl)-3-methylquinoline (3j)

Pale-yellow oil; 27.8 mg, 78% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.08-8.04 (m, 2H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.69-7.63 (m, 1H), 7.52 (d, *J* = 8.5 Hz, 1H), 7.50-7.44 (m, 1H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.2, 147.4, 135.8, 129.4, 128.9, 127.1, 126.4, 125.5, 118.2, 38.1, 30.1. NMR data are consistent with literature report [15].

3.2.11. 1,2,3,4-tetrahydroacridine (3k)

Colorless oil; 146 mg, 80% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 8.5 Hz, 1H), 7.79 (s, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.63-7.57 (m, 1H), 7.46-7.42 (m, 1H), 3.12 (t, *J* = 7.0 Hz, 2H), 2.98 (t, *J* = 6.5 Hz, 2H), 2.02-1.96 (m, 2H), 1.91-1.85 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.2, 146.5, 134.9, 130.9, 128.4, 128.2, 127.1, 126.8, 125.4, 33.5, 29.2, 23.2, 22.8. NMR data are consistent with literature report [17].

3.2.12. 6-methyl-2-phenylquinoline (3l)

White solid; 197 mg, 90% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16-8.11 (m, 2H), 8.09-8.05 (m, 2H), 7.80 (d, *J* = 8.5 Hz, 1H), 7.55-7.48 (m, 4H), 7.45-7.40 (m, 1H), 2.52 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.4, 146.8, 139.7, 136.1, 136.0, 131.8, 129.3, 129.0, 128.7, 127.4, 127.1, 126.2, 118.9, 21.5. NMR data are consistent with literature report [18].

3.2.13. 6-methoxy-2-phenylquinoline (3m)

Pale-yellow solid; 221 mg, 94% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.14-8.03 (m, 4H), 7.78 (d, *J* = 8.5 Hz, 1H), 7.53-7.47 (m, 2H), 7.45-7.35 (m, 2H), 7.04 (d, *J* = 3.5 Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.5, 154.9, 144.3, 139.7, 135.4, 131.1, 128.8, 128.7, 128.0, 127.2, 122.3, 119.1, 104.9, 55.4. NMR data are consistent with literature report [19].

3.2.14. 6-chloro-2-phenylquinoline (3n)

White solid; 215 mg, 90% yield. ^{1}H NMR (500 MHz, $CDCl^3$) δ: 8.14-8.02 (m, 4H), 7.84-7.80 (m, 1H), 7.73 (d, *J* = 2.5 Hz, 1H), 7.62 (dd, *J* = 8.5, 2.5 Hz, 1H), 7.54-7.42 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.4, 146.5, 139.1, 135.7, 131.8, 131.2, 130.4, 129.4, 128.8, 127.6, 127.4, 126.0, 119.6. NMR data are consistent with literature report [19].

3.2.15. 6-Bromo-2-phenylquinoline (3o)

White solid; 258 mg, 91% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.15-8.10 (m, 2H), 8.08-7.99 (m, 2H), 7.95-7.91 (m, 1H), 7.84 (d, *J* = 8.5 Hz, 1H), 7.78-7.74 (m, 1H), 7.54-7.43 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.5, 146.7, 139.1, 135.6, 133.0, 131.3, 129.5, 129.4, 128.8, 128.1, 127.4, 119.9, 119.6. NMR data are consistent with literature report [20].

3.2.16. 2-Phenyl-6-(trifluoromethyl)quinoline (3p)

White solid; 221 mg, 81% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.26 (d, *J* = 8.5 Hz, 2H), 8.20- 8.11 (m, 2H), 7.94 (d, *J* = 8.5 Hz, 1H), 7.89-7.85 (m, 1H), 7.57-7.47 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.3, 149.2, 138.8, 137.4, 130.8, 129.9, 128.9, 128.1, 127.7, 127.6, 126.0, 125.6-125.2 (m), 122.7, 120.0. NMR data are consistent with literature report [21].

3.2.17. 7-Chloro-2-phenylquinoline (3q)

White solid; 213 mg, 89% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16-8.08 (m, 4H), 7.80 (d, *J* = 8.5 Hz, 1H), 7.67 (d, *J* = 8.5 Hz, 1H), 7.54-7.40 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.1, 148.5, 139.0, 136.4, 135.3, 129.5, 128.8, 128.6, 128.5, 127.4, 127.1, 125.4, 118.9. NMR data are consistent with literature report [16].

3.2.18. 7-Bromo-2-phenylquinoline (3r)

White solid; 255 mg, 90% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.34 (d, *J* = 1.5 Hz, 1H), 8.15-8.09 (m, 3H), 7.83 (d, *J* = 8.5 Hz, 1H), 7.63-7.43 (m, 5H); ^{13}C NMR (125 MHz,

$CDCl_3$) δ: 158.0, 148.7, 139.0, 136.5, 131.9, 129.7, 129.6, 128.8, 128.6, 127.5, 125.6, 123.6, 119.1. NMR data are consistent with literature report [21].

3.2.19. 8-Methyl-2-phenylquinoline (3s)

Pale-yellow oil; 195 mg, 89% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.29-8.22 (m, 2H), 8.17 (d, *J* = 8.5 Hz, 1H), 7.89 (d, *J* = 8.5 Hz, 1H), 7.66-7.63 (m, 1H), 7.59-7.50 (m, 3H), 7.48-7,36 (m, 2H), 2.90 (s, 3H); 13C NMR (125 MHz, $CDCl_3$) δ: 155.5, 147.1, 139.8, 137.6, 136.9, 129.6, 129.1, 128.7, 127.4, 127.1, 126.0, 125.4, 118.1, 17.8. NMR data are consistent with literature report [15].

3.2.20. 8-Chloro-2-phenylquinoline (3t)

Pale-yellow oil; 208 mg, 87% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.30-8.25 (m, 2H), 8.21 (d, *J* = 8.5 Hz, 1H), 7.95 (d, *J* = 8.5 Hz, 1H), 7.85-7.81 (m, 1H), 7.74-7.70 (m, 1H), 7.56-7.38 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.4, 144.3, 139.0, 137.1, 133.9, 129.7, 128.8, 128.4, 127.6, 126.4, 126.0, 119.3. NMR data are consistent with literature report [22].

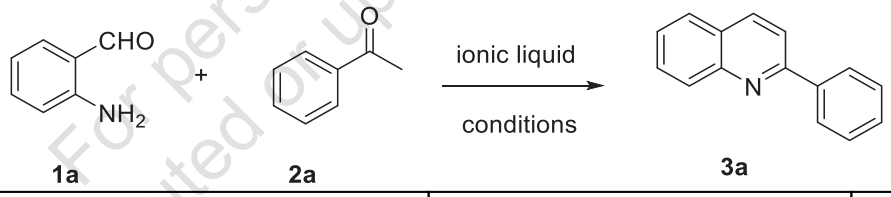
3.2.21. 6,7-Dimethoxy-2-phenylquinoline (3u)

Pale-yellow solid; 225 mg, 70% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.13-8.08 (m, 2H), 8.02 (d, *J* = 8.5 Hz, 1H), 7.71 (d, *J* = 8.5 Hz, 1H), 7.53-7.47 (m, 3H), 7.45-7.39 (m, 1H), 7.03 (s, 1H), 4.04 (s, 3H), 4.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.2, 152.4, 149.5, 145.1, 139.9, 134.8, 128.7, 128.6, 127.1, 122.6, 117.1, 108.3, 104.8, 56.0, 55.9. NMR data are consistent with literature report [22].

4. RESULTS AND DISCUSSION

Initially, 2-aminobenzaldehyde (**1a**) and acetophenone (**2a**) were chosen as model substrates for the Friedländer reaction condition optimization, as shown in Table **1**. We conducted the reaction of **1a** (1 mmol) with **2a** (1 mmol) in the presence of different ionic liquids under various conditions and the progress of the reaction was monitored by TLC. The results were summarized in the Table **1**. Four different ionic liquids (0.8 mmol) were examined as catalysts for the condensation (Table **1**, **Entries 1-4**). The use of [Bmim]Cl catalyst provided desired product in 42% yield (Table **1**, **Entry 1**). Under Et_3NHSO_4 and $[Bmim]PF_6$ catalysts, the reaction proceeded smoothly to give **3a** in 76% and 81 isolated yields, respectively (Table **1**, **Entries 2, 4**). Gratifyingly, excellent yields of product **3a** were achieved when $[Bmim][HSO_4]$ and $[Bmim]BF_4$ were employed (Table **1**, **Entries 3, 5**). In our next attempts, $[Bmim][HSO_4]$ was used as the catalyst for the condensation reaction to enhance the reaction efficiency as well as to find more environmentally benign conditions because the $[Bmim]BF_4$ is detrimental to the environment. Our effort to reduce the amount of $[Bmim][HSO_4]$ catalyst to 0.6 mmol was successful and the reaction yield remained unchanged (Table **1**, **Entry 6**). Unfortunately, when the catalyst loading was decreased to 0.4 mmol, lower yield was observed (Table **1**, **Entry 7**). Our final attempts were to perform the reaction under microwave irradiation. To our delight, 92% yield of product was isolated when mixture of substrates was irradiated at 100 °C, 100 W with 60 mol% of catalyst (Table **1, Entry 8**). The reaction conditions were further improved by reducing the cata

Table 1. Optimization of the reaction conditions.



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

Entries	Ionic liquids (mmol)	Temperature (0 °C)/time (h)	Yield (%)[a]
1	[Bmim]Cl (0.8)	110/8	42
2	Et_3NHSO_4 (0.8)	110/ 8	76
3	$[Bmim][HSO_4]$ (0.8)	100/ 4	90
4	$[Bmim]PF_6$ (0.8)	110/ 6	81
5	$[Bmim]BF_4$ (0.8)	100/ 4	89
6	$[Bmim][HSO_4]$ (0.6)	100/4	90
7	$[Bmim][HSO_4]$ (0.4)	100/4	82
8	$[Bmim][HSO_4]$ (0.6)	100/0.5	92[b]
9	$[Bmim][HSO_4]$ (0.4)	100/0.5	92[b]
10	$[Bmim][HSO_4]$ (0.4)	70/0.5	84[b]
11	$[Bmim][HSO_4]$ (0.2)	100/0.5	83[b]

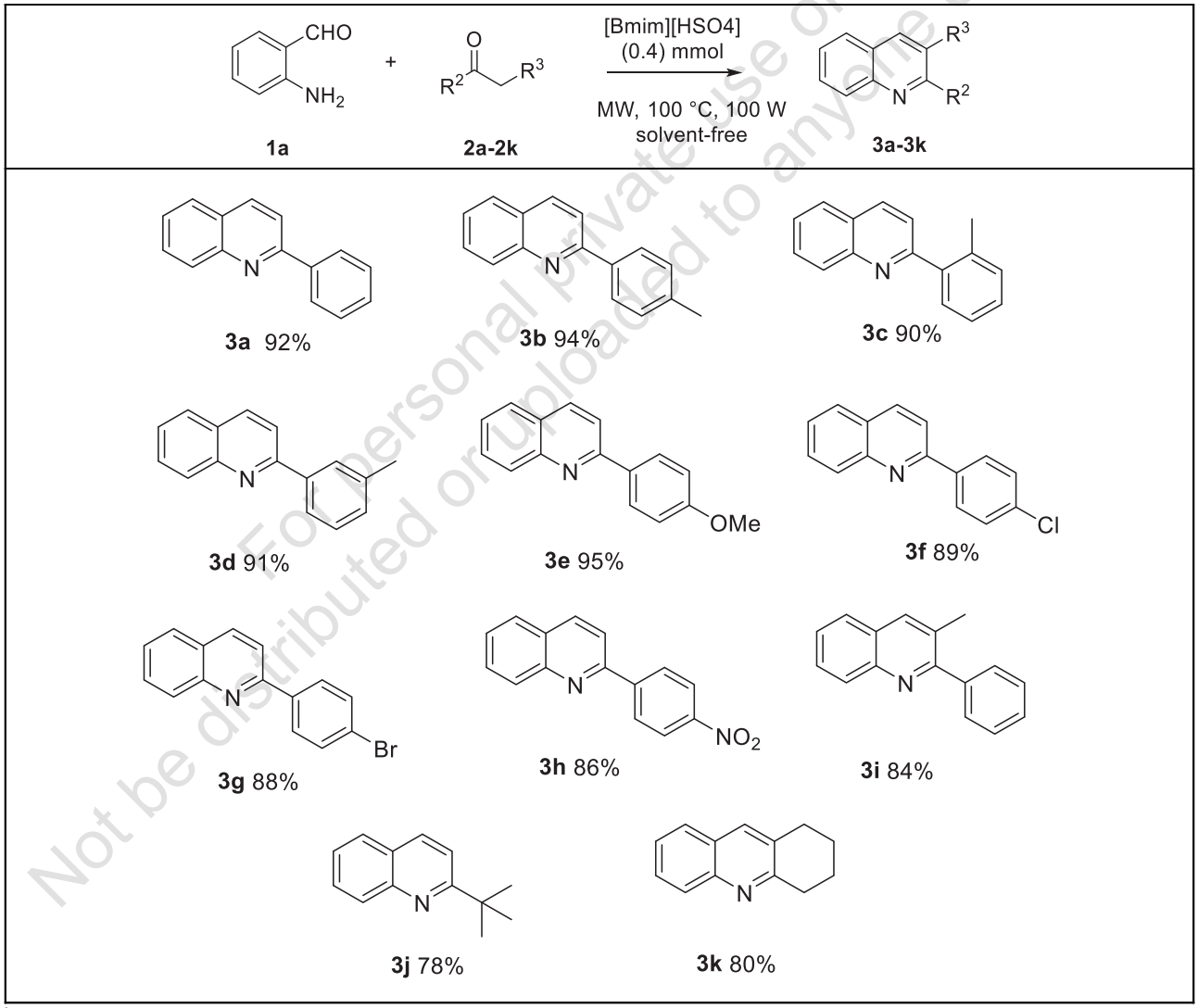
[a]: Isolated yield.
[b]: The reaction was performed at 100 W.

lyst loading to 40 mol% and the reaction yield was not affected (Table **1, Entry 9**). Unfortunately, attempts to lower temperature were not satisfactory (Table **1, Entry 10**). Further reduction of catalyst use led to a considerable loss in reaction yield (Table **1, Entry 11**). Therefore, the optimized reaction conditions are the following: 40 mol% of [Bmim][HSO_4] catalyst, solvent-free conditions, microwave irradiation at 100 °C and 100 W in 30 minutes.

With optimized conditions in hand, we examined the reaction of various ketones and the results are shown in Table **2**. For the reaction with 2-aminobenzaldehyde, aryl ketones with electron-donating groups and electron-withdrawing groups each participated well in this reaction and moderate to excellent yields of quinoline derivatives were obtained (Table **2**, **3a-3h**). A variety of functional groups, such as methyl, methoxy, chloro, bromo, and nitro group were well tolerated and installed. Moreover, ethylphenyl ketone, *t*-butylmethyl ketone, and cyclohexanone also afforded desired products in good yields (Table **2**, **3i-3k**).

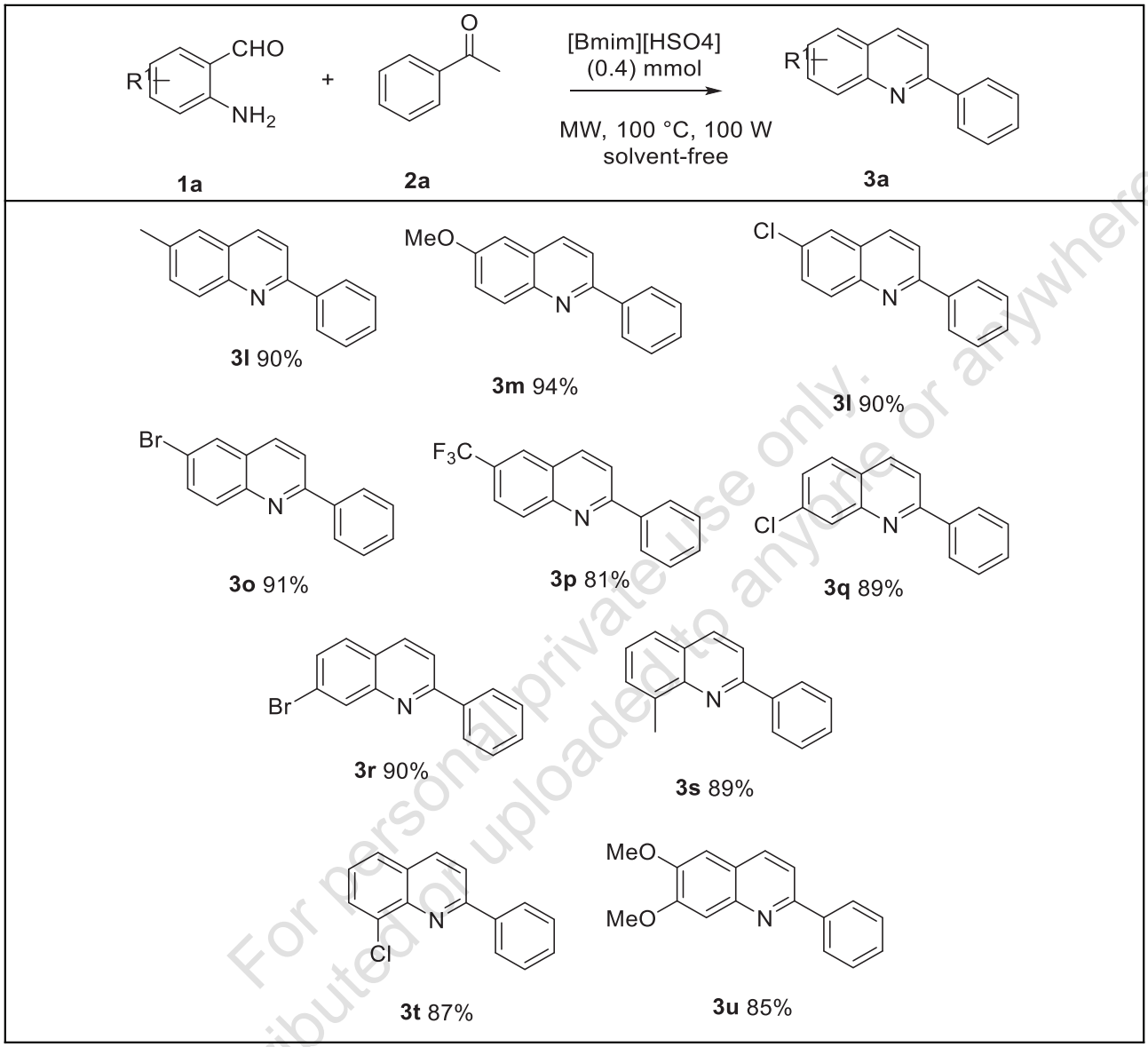
As a further aspect, the reaction of various 2-aminobenzaldehyde derivatives with acetophenone was also examined. The results presented in Table **3** show that 2-bromobenzaldehydes with methyl, methoxy, chloro, fluoro, and trifluoromethyl substituents at different positions could take part in this reaction and high yields of products were also provided in high yields (Table **3**, **3l-3u**). More importantly, both electron-donating and electron-withdrawing groups at the 2-aminobenzaldehyde counterparts gave the products in almost equally excellent yields. Compared to the conventional heating method using [Bmim][HSO_4] catalyst, [9] this microwave-assisted method was performed in a shorter time and required lower catalyst loading.

Table 2. Scope of ketones.



[a]: The reaction was performed in 10 mmol scale

Table 3. Scope of 2-aminobenzaldehydes.



CONCLUSION

In conclusion, we have demonstrated an efficient and straightforward approach for the synthesis of quinolines from 2-aminobenzaldehydes and ketones using [B-mim][HSO_4] as the catalyst. The microwave-assisted Friedländer synthesis featured some advantages such as broad substrate scope, environmentally friendly conditions, short reaction time, readily available starting materials, and high yields of products. In the future, the approach will be expanded for asymmetric aliphatic ketones and acetaldehyde derivatives to examine the versatility and selectivity.

LIST OF ABBREVIATIONS

If abbreviations are used in the text either they should be defined in the text where first used, or a list of abbreviations can be provided.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

Not applicable.

FUNDING

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CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

ACKNOWLEDGEMENTS

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SUPPLEMENTARY MATERIAL

Supplementary Material is available on the publisher's website along with the published article.

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