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Silica Sulfuric Acid as an Efficient Catalyst for the Friedländer Quinoline Synthesis from Simple Ketones and *ortho*-Aminoaryl Ketones Under Microwave Irradiation

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The synthesis of quinoline derivatives *via* Friedländer method from *ortho*-aminoaryl ketones in the presence of a catalytic amount of silica sulfuric acid (SSA) under solvent-free condition and microwave irradiation was described. A good range of simple ketones such as cyclohexanone and deoxybenzoin were used.

Keywords: Quinolines, Friedlander method, *ortho*-Aminoaryl ketones, Ketones, Silica sulfuric acid

INTRODUCTION

Quinolines are important heterocyclic systems, constituting the structure of many naturally occurring products with interesting pharmacological properties [1]. A large number of quinolines have been widely used as antimalarial [2], antiasthmatic [3], antiinflamatory [4], tyrosine kinase PDGF-RTK inhibitor [5], antitumor [6], and antiviral agent [7]. In addition, quinolines are valuable synthons used for the preparation of nano- and mesostructures with enhanced electronic and photonic properties [8].

Among the various synthetic methods in quinoline synthesis, Friedlander condensation is an extremely useful and versatile method for the direct construction of a quinoline ring [9]. The condensation of an aromatic *o*-aminoketone or aldehyde with an enolizable ketone proceeds directly with the loss of two molecules of water. Friedländer reactions are generally carried out either by refluxing an aqueous or alcoholic solution of reactants in the presence of base or by heating a mixture of the reactants at high temperatures ranging from 150-220 °C in the absence of catalyst [11n-q]. Under thermal or base catalysis conditions; *o*-aminobenzophenone fails to react with simple ketones such as cyclohexanone and deoxybenzoin [11r]. There are some modified procedures for Friedlander annulations at room temperature [10], elevated temperature [11] and microwave irradiations [12].

According to our previous results for the quinoline synthesis from *o*-aminoarylketones with ketones using Lewis acid catalysts, it was found that aryl alkyl ketones undergo the mentioned condensation often with difficulty [13a-c]. This is in close agreement with the reported results by Shaabani *et al.* for the same reactions in the presence of silica sulfuric acid (SSA) as solid protic acid at 100 °C [14].

In continuation of our studies on the quinoline synthesis [13], and preparation and applications of silica supported catalysts [15], we now report SSA as an efficient catalyst for the preparation of quinolines from *o*-aminoarylketones and different ketones (including dialkyl cyclic ketones) under solvent-free conditions and microwave irradiation.

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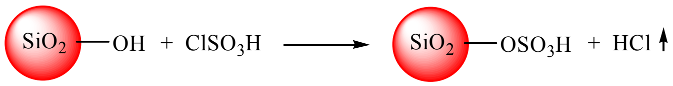


Scheme 1

EXPERIMENTAL

Chemicals and Apparatus

All commercially available chemicals were obtained from Aldrich, Merck and Fluka companies, and used without further purifications. Some reagents were prepared from previous known procedures such as silica sulfuric acid [15b], metal hydrogen sulfates [16], AlPW$_{12}$O$_{40}$ [17].

Melting points were measured on a SMPI apparatus. Nuclear magnetic resonance (NMR) spectra were recorded on Bruker 300 MHz Advance Spectrameters. Infrared (IR) was conducted on a Perkin Elmer GX FT IR spectrometer. Mass spectrometry was carried out using a Micromass Tof Spec 2E spectrometer.

Typical Experimental Procedure

2-Amino-5-chlorobenzophenone (233 mg, 1 mmol), acetophenone (600 mg, 5 mmol) and silica sulfuric acid (40 mg, 0.1 mmol) were mixed together and irradiated under microwave condition (Black & Deaker, 900 W, 80%) for 5 min (5 × 1 min). The mixture was washed with EtOAc (2 × 10 ml) and filtered. After evaporation of the solvent, the desired product was recrystallized in hot ethanol to give **3a** in 91% yield.

Spectral Data

The spectral data for the model compounds shown in Table 1 are as follows:

3a. FT-IR (KBr): υ 3058, 1588, 1541, 1483, 1358, 1152, 1076, 884, 780, 699 cm^{-1}. ^{1}H NMR (90 MHz, CDCl$_3$) δ (ppm): 8.33 (m, 3H), 7.73-7.44 (m, 11H). ^{13}C NMR (22.5 MHz, CDCl$_3$) δ (ppm): 157.08, 148.52, 147.34, 139.25, 137.88, 132.30, 131.83, 130.48, 129.53, 128.90, 127.61, 126.58, 124.54, 120.01.

3b. FT-IR (KBr): υ 3066, 2956, 2930, 2933, 1602, 1587, 1541, 1515, 1483, 1356, 1171, 1026, 889, 825, 777, 704 cm^{-1}. ^{1}H NMR (90 MHz, CDCl$_3$) δ (ppm): 3.85 (s, 3H), 6.99 (d, 2H), 7.52-7.80 (m, 8H), 8.15 (m, 3H).

3c. FT-IR (KBr): υ 3440, 3059, 1588, 1546, 1490, 1353, 1297, 1076, 875, 748, 702 cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 6.95 (t, 1H), 7.11 (d, *J* = 8.21 Hz, 1H), 7.39 (t, 1H), 7.53 (m, 2H), 7.60 (m, 3H), 7.69 (q, 1H), 7.855 (d, *J* = 2.14 Hz, 1H), 7.95 (d, *J* = 8.05 Hz, 1H), 7.98 (s, 1H), 8.04 (d, *J* = 8.937 Hz, 1H), 14.909 (br, 1H). ^{13}C NMR (75 MHz, CDCl$_3$) δ (ppm): 118.48, 118.76, 118.97, 124.80, 126.07, 127.07, 129.00, 129.12, 129.40, 129.52, 131.26, 132.38, 132.64, 137.27, 143.72, 149.45, 157.66, 161.01. MS: m/z (%) = 331 (M^{+}, 100), 254 (31), 147 (25), 133 (21), 121(19).

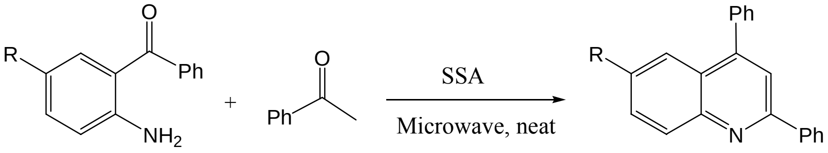
3d. FT-IR (KBr): υ 3046, 2924, 1589, 1541, 1483, 1355, 1154, 1076, 888, 829, 753, 699 cm^{-1}. ^{1}H NMR (90 MHz, CDCl$_3$) δ (ppm): 7.17 (s, 1H), 7.46-7.82 (m, 9H), 8.02 (d, 3H).

3e. FT-IR (KBr): υ 3078, 1598, 1588, 1510, 1348, 1153, 852, 698 cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 7.58 (m, 5H), 7.743 (q, 1H), 7.90 (s, 1H), 7.925 (d, *J* = 2.09 Hz, 1H), 8.253 (d, *J* = 8.97 Hz, 1H), 8.39 (s, 4H). ^{13}C NMR (75 MHz, CDCl$_3$) δ (ppm): 119.92, 124.12, 124.67, 126.90, 128.43, 129.01, 129.13, 129.40, 131.26, 131.67, 133.48, 137.14, 144.63, 146.87, 148.51, 149.51, 154.17. MS: m/z (%) = 360 (M^{+}, 100), 314 (38), 278 (29), 139 (31).

3f. FT-IR (KBr): υ 3037, 2969, 1601, 1545, 1476, 1349, 1072, 849, 699 cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 0.78 (t, 3H), 2.615 (q, 2H), 7.28-7.35 (m, 3H), 7.49-7.60 (m, 9H), 8.105 (d, *J* = 8.934 Hz, 1H). ^{13}C NMR (75 MHz, CDCl$_3$): 14.97, 23.49, 124.92, 128.15, 128.26, 128.35, 128.58, 128.66, 129.33, 129.58, 131.01, 132.11, 134.17, 136.58, 141.19, 144.34, 146.73, 161.31. MS: m/z (%) = 342 (M^{+}, 100), 327 (33), 291 (16), 146 (28).

3g. FT-IR (KBr): υ 3059, 3028, 2953, 1602, 1564, 1494, 1475, 1370, 1080, 1011, 834, 702, 695 cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 4.26 (s, 2H), 6.870 (m, 2H), 6.96 (m, 2H), 7.065 (m, 2H), 7.11-7.20 (m, 6H), 7.26 (m, 3H), 7.51 (d, *J* = 2.17 Hz, 1H), 7.68 (q, 1H), 8.185 (d, *J* = 8.94 Hz, 1H). ^{13}C NMR (75 MHz, CDCl$_3$) δ (ppm): 43.69, 125.43, 126.05, 127.05, 127.32, 127.58, 127.69, 127.93, 128.08, 129.03, 130.03, 130.13, 130.45, 130.88, 132.07, 135.17, 136.11, 137.56, 139.01, 145.57, 159.89. MS: m/z (%) = 404 (M^{+}, 100), 328 (24), 294 (91), 146 (14), 91 (15).

3h. FT-IR (KBr): υ: 3060, 2945, 2858, 1600, 1569, 1477, 1440, 1349, 1266, 1165, 1075, 937, 831, 818, 756, 708, 617



1a; R= H
1b; R= Cl

Scheme 2

cm^{-1}. ^{1}H NMR (90 MHz, CDCl$_3$) δ (ppm): 1.89 (m, 4H), 2.58 (t, 2H), 3.17 (t, 2H), 7.27-7.89 (m, 8H).

3i. FT-IR (KBr): υ 3041, 2954, 1603, 1564, 1486, 1384, 1341, 1162, 1076, 949, 829, 706 cm^{-1}. ^{1}H NMR (90 MHz, CDCl$_3$) δ (ppm): 2.12 (q, 2H), 2.81 (t, 2H), 3.15 (q, 3H), 7.30-7.71 (m, 7H), 7.91 (d, 1H). ^{13}C NMR (22.5 MHz, CDCl$_3$) δ (ppm): 23.26, 30.25, 35.01, 124.35, 126.91, 128.60, 128.80, 129.04, 130.36, 131.21, 134.49, 135.98, 141.71, 146.37, 167.67.

3j. FT-IR (KBr): υ 3062, 1602, 1572, 1476, 1345, 1076, 944, 836, 701 cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 2.17 (s, 3H), 7.315 (q, 2H), 7.379 (d, J = 2.16 Hz, 1H), 7.44-764 (m, 9H), 8.135 (d, J = 8.93 Hz, 1H). ^{13}C NMR (75 MHz, CDCl$_3$) δ (ppm): 18.78, 124.82, 127.85, 127.94, 128.25, 128.41, 128.45, 128.94, 129.26, 129.59, 131.07, 132.18, 136.98, 140.98, 144.55, 147.23, 161.05. MS: m/z (%) = 328 (M$^+$, 100), 293 (19), 146 (21), 105 (23).

3k. FT-IR (KBr): 3054, 1592, 1545, 1486, 1416, 1355, 1094, 1016, 831, 770 υ cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 7.52 (m, 3H), 7.57 (s, 5H), 7.755 (m, 1H), 7.80 (s, 1H), 7.937 (d, J = 8.36 Hz, 1H), 8.17 (d, J = 8.59 Hz, 2H), 8.275 (d, J = 8.38 Hz, 1H). ^{13}C NMR (75 MHz, CDCl$_3$) δ (ppm): 119.00, 125.74, 125.82, 126.67, 128.60, 128.69, 128.91, 129.06, 129.56, 129.88, 135.68, 137.78, 138.16, 148.53, 149.66, 155.47. MS: m/z (%) = 314 (M$^+$, 100), 278 (13), 202 (23), 139 (24), 84 (22).

3l. ^{1}H NMR (90 MHz, CDCl$_3$) δ (ppm): 1.86 (m, 4H), 2.59 (t, 2H), 3.20 (t, 2H), 7.26-7.98 (m, 9H). ^{13}C NMR (22.5 MHz, CDCl$_3$) δ (ppm): 22.64, 22.76, 27.74, 33.99, 125.09, 125.47, 126.40, 127.44, 128.01, 128.32, 128.85, 136.88, 146.13, 158.71.

3m. FT-IR (KBr): 3053, 3024, 1589, 1544, 1488, 1444, 1406, 1356, 1028, 889, 770, 699 υ cm^{-1}. ^{1}H NMR (90 MHz, CDCl$_3$) δ (ppm): 7.49 (s, 1H), 7.79-8.02 (m, 10H), 8.44 (t, 3H).

3n. FT-IR (KBr): υ 3048, 3989, 1570, 1553, 1483, 1347, 1070, 764, 699 cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 2.20 (s, 3H), 7.345 (m, 2H), 7.43 (m, 2H), 7.45-7.60 (m, 6H), 7.68 (m, 3H), 8.231 (d, J = 8.475 Hz, 1H). ^{13}C NMR (75 MHz, CDCl$_3$) δ (ppm): 18.70, 126.04, 126.32, 126.78, 127.10, 127.89, 128.16, 128.38, 128.61, 128.70, 128.99, 129.36, 129.47, 137.74, 141.50, 146.25, 147.84, 160.85. MS: m/z (%) = 294 (M$^+$, 100), 217 (16), 189 (13), 139 (14), 91 (14).

3o. mp.: 190 ºC. FT-IR (KBr): υ 3058, 1604, 1562, 1473, 1442, 1370, 1079, 830, 695 cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$) δ (ppm): 6.91 (m, 2H), 7.04 (m, 3H), 7.145 (m, 2H), 7.24 (m, 3H), 7.32 (m, 3H), 7.39 (m, 2H), 7.592 (d, J = 2.20 Hz, 1H), 7.69 (q, 1H), 8.226 (d, J = 8.937 Hz, 1H). ^{13}C NMR (75 MHz, CDCl$_3$) δ (ppm): 125.39, 126.58, 127.48, 127.64, 127.78, 127.86, 128.07, 129.90, 130.21, 130.37, 131.25, 131.35, 132.48, 133.82, 136.24, 137.94, 140.73, 145.69, 147.04, 159.27. MS: m/z (%) = 390 (M$^+$, 100), 354 (12), 314 (10), 176 (17), 57 (10).

RESULTS AND DISCUSSION

For the optimization of reaction conditions, 2-amino-5-chlorobenzophenone (**1b**) and acetophenone were selected as model substrates. It was found that, in the absence of solvent, the reaction is completed in the presence of 0.1 equivalents of SSA as catalyst and irradiation by microwave for 5 min. 6-Chloro-2,4-diphenylquinoline was obtained in 91% yield (Scheme 2) (Table 1, entry 1).

To demonstrate the generality of the method, we next extended the scope of this reaction to other model compounds, the results of which are summarized in Tables 1 and 2. As is obvious from Table 1, this method is equally effective for both cyclic and acyclic ketones. A controlled reaction was run in

Table 1. Preparation of Quinoline Derivatives

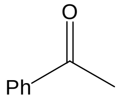
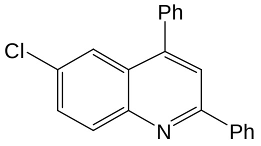
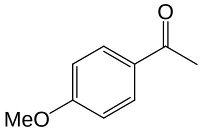
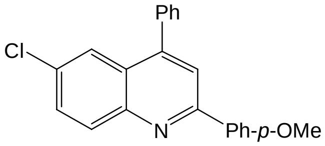
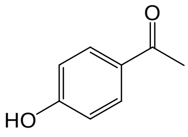
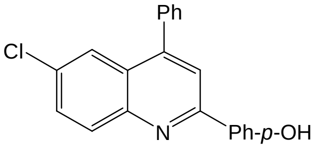
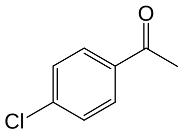
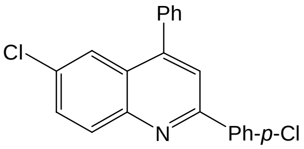
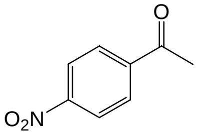
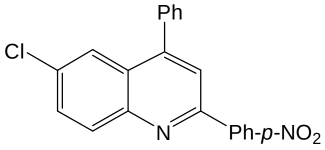
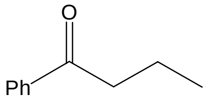
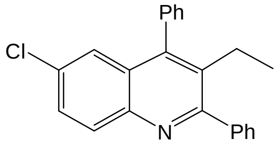
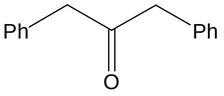
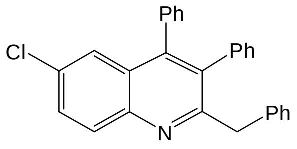
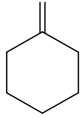
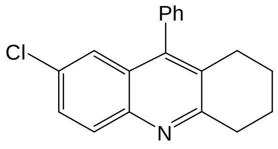
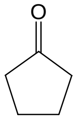
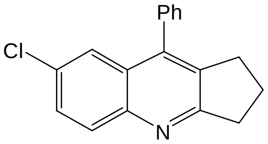
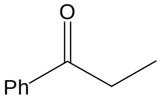
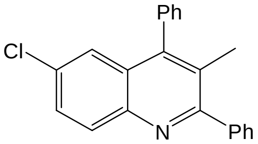
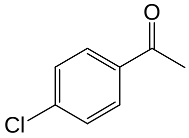
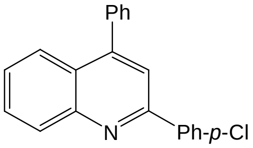
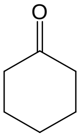
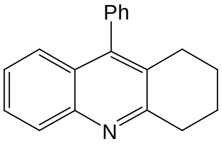
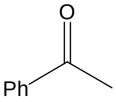
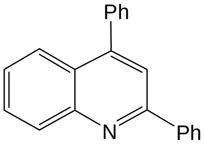
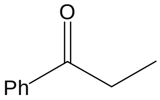
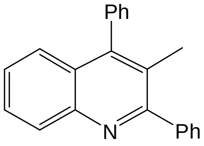
Entry	Substrate **1**	Ketone **2**	Product **3**	Isolated yields (%)	M.p. °C (Lit)
1	**1b**			**3a,** 91 trace[a,b]	128-130 (129) [11a]
2	**1b**			**3b**, 90	137-139
3	**1b**			**3c**, 77[b]	159-161
4	**1b**			**3d**, 88[b]	159-161 (161) [11c]
5	**1b**			**3e**, 91	216-219
6	**1b**			**3f**, 80	155-157
7	**1b**			**3g**, 75	161-163
8	**1b**			**3h**, 92	161-163 (162) [11a]

Table 1. Continued

9	**1b**			**3i**, 94	95-97 (95) [11a]
10	**1b**			**3j**, 90	150-152
11	**1a**			**3k**, 89	103-105
12	**1a**			**3l**, 93	139-141 (137) [11a]
13	**1a**			**3m**, 87	107-109
14	**1a**			**3n**, 88	138-140

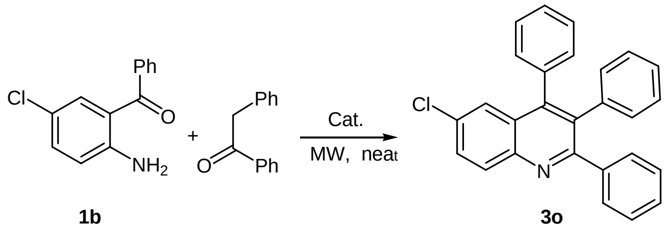
[a]Only silica gel (1 equiv., mesh 60) was used as the catalyst. [b]After 10 min.

the presence of silica gel alone as the catalyst and it was found that the reaction does not yield any product even after long reaction time (Table 1, entry 1).

The reaction of acetophenone derivatives with both electron-donating and electron-withdrawing substituents was investigated in the presence of SSA (Table 1, entries 2-5, 12) and satisfactory results were obtained.

We also attempted to extend the methodology to include more hindered ketones. Surprisingly, dibenzyl ketone reacted with **1b** and produced 2-benzyl-6-chloro-3,4-diphenylquinoline **3g** in good yield (Table 1, entry 7). 6-Chloro-2,3,4-triphenylquinoline **3o** was also prepared through the condensation of **1b** and deoxybenzoin in 85% yield in the presence of 0.1 equivalent of SSA after 5 min (Table 2, entry 1). To show the powerful effect of SSA in the synthesis of quinolines with bulky substituents, the reaction of deoxybenzoin with **1b** was carried out in the presence of other general Lewis acids. As shown in Table 2, only the salts of

Table 2. Synthesis of 6-Chloro-2,3,4-Triphenylquinoline



Entry	Catalyst	Equiv./ **1b**	Time (min)	Yield (%)
1	**SSA**	**0.1**	**5**	**85**
2	$ZrOCl_2.8H_2O$	0.1	10	trace
3	$Al(HSO_4)_3$[a]	0.25	10	80
4	$Zr(HSO_4)_4$[a]	0.2	7	77
5	$Zr(NO_3)_4$	0.15	5	trace
6	$FeCl_3.6H_2O$	0.25	5	trace
7	$Fe(NO_3)_3.9H_2O$	0.25	5	trace
8	$ZnCl_2$	1	5	trace
9	$AlCl_3$	1	5	trace
10	$CoCl_2.6H_2O$	0.50	5	trace
11	$Bi(NO_3)_3$	0.25	6	trace
12	$NaHSO_4$[a]	1	6	79
13	$AlPW_{12}O_{40}$[b]	0.05	6	trace
14	$ZrCl_4$	0.25	6	trace

[a]See Ref. [16]. [b]See Ref. [17].

hydrogen sulfates [16] gave good results, but at higher equivalents than SSA.

In Table 3, a comparison is made for the synthesis of **3l** from cyclohexanone and **1a**, with other literature methods. As seen, the present procedure shows many advantages and is superior with respect to time, reaction condition and yield.

In conclusion, the SSA-promoted reaction reported here is a novel extension of quinoline synthesis *via* condensation of different ketones and *o*-aminoarylketones (Friedlander reaction). This condensation normally requires high-temperature, high-pressure, or long reaction conditions and often hazardous solvents while in our method most of the mentioned disadvantages were overcome.

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Table 3. Comparison of the Results Obtained Using SSA with Other Methods for Synthesis of **31**

Run	Catalyst	Conditions	Solvent	Time (h)	Yield (%)
1	SSA	MW	Neat	5 min	93
2	$Ag_3PW_{12}O_{40}$	Reflux	EtOH	3.5	87 [11a]
3	$Bi(OTf)_3$	rt	EtOH	3.5	86 [10a]
4	$NaAuCl_4$	rt	EtOH	48	62 [10b]
5	HCl	100-200 °C	H_2O	1	68 [11l]
6	NH_2SO_3H	70 °C	Neat	1	90 [11b]
7	$HClO_4/SiO_2$	Reflux	CH_3CN	3	92 [11m]
8	$Zr(DS)_4.xH_2O$[a]	Reflux	H_2O	6	90 [13a]
9	$Zr(HSO_4)_4$	Reflux	H_2O	13	87 [13b]
10	bmimCl-$ZnCl_2$	rt	Ionic Liquids	24	80 [10g]

[a]DS = Dodecyl sulfate.

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