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Friedlander Synthesis of Quinolines Promoted By Polymer-bound Sulfonic Acid

Behrooz Maleki^a, Esmail Rezaei Seresht^a & Zahra Ebrahimi^a ^a Department of Chemistry, Hakim Sabzevari University, Sabzevar, Khorasan, Iran Published online: 18 Mar 2015.

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Friedlander Synthesis of Quinolines Promoted By Polymer-bound Sulfonic Acid

Behrooz Maleki, Esmail Rezaei Seresht, and Zahra Ebrahimi

Department of Chemistry, Hakim Sabzevari University, Sabzevar, Khorasan, Iran

Quinolines and their derivatives are very important biologically and occur widely in natural products. Members of this family have displayed interesting physiological activities and found major applications in medicinal chemistry, as anti-malarial,^{1–2} anti-bacterial,^{3–4} anti-inflammatory,^{5–6} anti-hypertensive,^{7–8} anti-platelet, and tyrosine kinase inhibiting agents.^{9–13} They are also useful for the preparation of nano- and meso-structures having enhanced electronic and photonic properties.^{14–15} Moreover, quinolines are also employed in the fields of bioorganic,¹⁶ bioorganometallic processes¹⁷ and industrial organic chemistriy.¹⁸ Therefore, the exploration of efficient synthetic methods to construct the quinoline framework has continually drawn great attention for many decades.

Among these methods, the Friedlander annulations is one of the simplest and most direct approaches for the synthesis of quinolines. It involves the acid-, base-catalyzed or thermal condensation between o-aminoarylketones and aldehydes with another carbonyl compound possessing a reactive α -methylene group followed by cyclodehydration.^{19–23} It has been shown that acid catalysis is more effective than base catalysts for the Friedlander annulation. In recent years, Bronsted and Lewis acids such as ionic liquids,24-26 sulfamic acid,²⁷ hydrochloric acid,²⁸ zirconium tetrakisdodecyl sulfate [Zr(DS)₄],²⁹ silica sulfuric acid,³⁰⁻³¹ silica supported phosphomolybdic acid [PMA.SiO₂],³² nanocrystalline aluminium oxide,³³ dodecylphosphonic acid (DPA),³⁴ poly(N-bromoethylbenzene-1,3-disulfonamide) [PBBS] or N,N,N'-tetrabromobenzene-1,3-disulfonamide [TBBDA],³⁵ zinc triflate,³⁶ nano-flake ZnO,³⁷ Cs_xH_{3-x}PW₁₂O₄₀ heteropoly salts,³⁸ Yb(OTf)₃,³⁹ nanosized MCM-41 supported ionic liquid [(BSPY)HSO4/MCM-41],⁴⁰ o-benzendisulfonimide⁴¹ and nanocrystalline sulfated zirconia⁴² have been utilized. However, many of these methods suffer from harsh reaction conditions, long reaction times, low yields, expensive reagents, tedious work-up procedures. The main disadvantage of nearly all existing methods is that the catalysts cannot be recovered and reused. For these reasons, the use of solid and heterogeneous catalysts has received considerable attention in organic synthesis. This extensive application of heterogeneous catalysts can make the process more efficient from both the environmental and economic points of view when the catalyst can be easily recycled.⁴³⁻⁴⁵

There are no previous report describing the use of Poly(AMPS-co-AA) in organic transformations.^{46–48} and it has been shown to possess several advantages such as low toxicity and cost, ease of handling and high catalytic activity as a potential green catalyst.

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Address correspondence to Behrooz Maleki, Department of Chemistry, Hakim Sabzevari University, Sabzevar, Khorasan, Iran. E-mail: malekibehrooz@gmail.com





We now disclose a novel, green, facile and convenient method for the preparation of quinoline from o-aminoaryl ketones or aldehydes and various ketones catalyzed by poly (AMPS-co-AA) under solvent-free conditions at 110°C (Scheme 1).

In order to determine the optimum conditions, the effect of temperature on the rate of the reaction was studied first for the preparation of **3a** from the two-component condensation of ethyl acetoacetate with o-aminobenzophenone under solvent-free conditions (Table 1, Entries 1–4). At 110° C, the reaction proceeded smoothly with near complete conversion to product. Further increase in temperature to 120 and 130°C did not accelerate rate or improve the yield. The study to determine optimal amount of Poly (AMPS-co-AA), was carried out by varying the amount of the catalyst (Entries 5, 6). The maximum yield was obtained with 0.06 g. Further increases in amount of Poly (AMPS-co-AA) did not have any significant effect on the yield. In absence of the catalyst, no products could be detected even after 1h.

We next established the generality of these conditions using different types of α -methylene carbonyl compounds with *o*-aminoaryl ketones to produce the corresponding quinolines under solvent-free conditions (Table 2).

	Screening of the Reaction	on Conditions for	itions for the Synthesis of 3a				
	COPh NH ₂ + EtO	poly(AMPS-co-AA) Solvent-Free	Ph CO ₂ Et N Me				
	1 2		3a				
Entry	Poly (AMPS-co-AA) (g)	Temp. (°C)	Times (min)	Yield (%)			
1	0.06	100	15	68			
2	0.06	110	10	84			
3	0.06	120	10	68			
4	0.06	130	10	50			
5	0.08	110	10	80			
6	0.03	110	20	72			
7	_	110	60	a			

Table 1

^a No reaction.

a Aminoard					mp	(°C)
ketones or aldehydes	α -Methylene ketones	Quinoline	Yield (%)	Time (min)	Found	Lit.
COPh NH ₂	Me o EtO	$\begin{array}{c} Ph \\ CO_2Et \\ N \\ Me \end{array}$	84	10	100–101	100–101 ⁴⁰
COPh NH ₂	Me O Me	Ph CO ₂ Me N Bb	80	10	108–110	108–109 ⁴⁰
COPh NH ₂	$\overset{\texttt{l}}{\bigcirc}$	Ph N 3c	80	10	134–135	133–135 ³⁷
COPh NH ₂		Ph N 3d	90	60	140–142	138–141 ³⁷
COPh NH ₂	ů,	$ \begin{array}{c} $	95	10	156–158	159 ²⁶
COPh NH ₂	Me Ne	Ph O N Me 3f	90	10	190–192	189–191 ³⁷

 Table 2

 The Heterogeneous Synthesis of Quinolines using Poly(AMPS-co-AA)

a Aminoaryl					mp	o (°C)
ketones or aldehydes	α -Methylene ketones	Quinoline	Yield (%)	Time (min)	Found	Lit.
COPh NH ₂	COCH3	Ph N N Bh Sg	97	70	107–109	107–109 ³¹
COPh NH2	COCH ₃	Ph N NO ₂ 3h	86	60	160–161	_
COPh NH2	COCH ₃	Ph N F 3i	86	70	93–94	_
COPh NH ₂	Et ₂ CO	Ph Me N Bt	84	20	96–97	95–96 ³⁸

Table 2 The Heterogeneous Synthesis of Quinolines using Poly(AMPS-co-AA) (Continued)

a Aminoarul					mp	(°C)
ketones or aldehydes	α -Methylene ketones	Quinoline	Yield (%)	Time (min)	Found	Lit.
COPh NH ₂	EtMeCO	Ph Me N 3k	80	20	145–146	144 ³⁸
COPh NH ₂		Ph O N 31	76	60	176–177	174–175 ³⁴
COPh NH ₂		$Ph \qquad O \qquad $	89	30	174–176	172 ³⁸
CL COPh NH ₂	Me o EtO	Cl Cl CO_2Et N Me $3n$	86	10	102–104	102–104 ⁴⁰
CICOPh NH2	Me O Me	Ph CO ₂ Me N 30	70	20	154–155	154 ⁴⁰
CI NH ₂		CI PhN $3p$	89	15	97–98	97–98 ⁴⁰

 Table 2

 The Heterogeneous Synthesis of Quinolines using Poly(AMPS-co-AA) (Continued)

				mp	o (°C)
α -Methylene ketones	Quinoline	Yield (%)	Time (min)	Found	Lit.
	CL N 3q	70	25	161–162	162–163 ⁴⁰
° Co	Cl Ph O N 3r	90	10	185–186	185 ²⁹
Me Me	Cl Ph O Me 3s	95	15	208–209	209–211 ³⁰
COCH3	Cl Ph N Ph 3t	65	45	126–127	128–130 ³¹
F COCH3	Cl Ph	94	50	137–139	_
	α -Methylene ketones	$\begin{array}{c} \begin{array}{c} \begin{array}{c} \alpha \text{-Methylene} \\ ketones \end{array} & Quinoline \end{array} \\ \hline \\ \begin{array}{c} \begin{array}{c} \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$	$\begin{array}{c c} \mathbf{a} \cdot \mathrm{Methylene} \\ \mathrm{ketones} & \mathrm{Quinoline} & \mathrm{Yield} \\ \mathrm{Quinoline} & \begin{pmatrix} & & & \\ &$	$\begin{array}{c cccc} \mathbf{a} & & & & & & & & & & & & & & & & & & &$	α -Methylene ketonesQuinolineYield Yield (%)Time Time Found $\begin{tabular}{lllllllllllllllllllllllllllllllllll$

Table 2 The Heterogeneous Synthesis of Quinolines using Poly(AMPS-co-AA) (Continued)

3u

o Aminoaryl					mp	• (°C)
ketones or aldehydes	α -Methylene ketones	Quinoline	Yield (%)	Time (min)	Found	Lit.
CHO NH2	° L	$\left(\begin{array}{c} \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$	82	40	93–95	91–93 ³⁹
CHO NH ₂	o s	N 3w	86	40	98–99	96–99 ²⁰
CHO NH ₂	O NH3	NCH ₃ 3x	88	40	78–79	76–77 ²⁰
O CHO NH ₂	o s	N 3y	92	40	206–207	203–205 ²⁰
O CHO NH ₂	O C C H ₃	N N N N N N N N N N	90	40	191–192	190–192 ²⁰

 Table 2

 The Heterogeneous Synthesis of Quinolines using Poly(AMPS-co-AA) (Continued)

Entry	Quinolines 3	Conditions	Time (min)	Yield (%)
3a	Ph	Zr(DS) ₄ /EtOH:H ₂ O(1:2)/Reflux ²⁹	180	72
		Silica sulfuric acid/100°C/ Solvent-Free ³⁰	55	87
		PMA.SiO ₂ /EtOH/Reflux ³²	60	94
		Nanocrystalline Al ₂ O ₃ /CHCl ₃ /Reflux ³³	180	98
	✓ N Me	DPA/90°C/Solvent-Free ³⁴	30	90
		Zinc triflate /MWI/Solvent-Free ³⁶	4/5	90
		Nano-flake ZnO /100°C/Solvent-Free ³⁷	210	85
		(BSPY)HSO ₄ /MCM-41/100°C/Solvent-Free ⁴⁰	70	93
		o-Benzendisulfonimide/80°C/Solvent-Free ⁴¹	180	93
		Nano-crystalline SZ/EtOH/Reflux ⁴²	110	84
		This work	10	84
3d	Ph	PMA.SiO ₂ /EtOH/Reflux ³²	70	80
		Nanocrystalline Al ₂ O ₃ /CHCl ₃ /Reflux ³³	180	98
		PBBS/100°C/Solvent-Free ³⁵	390	84
		Nano-flake ZnO /100°C/Solvent-Free ³⁷	240	63
	• N •	(BSPY)HSO ₄ /MCM-41/100°C/Solvent-Free ⁴⁰	90	87
		Nano-crystalline SZ/EtOH/Reflux ⁴²	110	78
		This work	60	90
3e	Ph O	SO_3H -functionalized ionic liquid/70°C/ H_2O^{26}	300	90
		Sulfamic acid/60°C/Solvent-Free ²⁷	75	92
		Zr(DS) ₄ /EtOH:H ₂ O(1:2)/Reflux ²⁹	360	90
		Nanocrystalline Al ₂ O ₃ /CHCl ₃ /Reflux ³³	180	98
	✓ N ✓	Cs _x H _{3-x} PW ₁₂ O ₄₀ /100°C/Solvent-Free ³⁸	30	82
		(BSPY)HSO ₄ /MCM-41/100°C/Solvent-Free ⁴⁰	40	87
		This work	10	95

 Table 3

 Comparison of Methods for the Synthesis of Quinolines.

The insolubility of the Poly (AMPS-co-AA) catalyst in ethanol provided an easy method for its separation from the product. After the addition of 96% ethanol to the thick reaction mixture, the insoluble catalyst was filtered off (suction), dried at 110°C and was re-used three times without substantial decrease in yields (84–75%) of **3a**. The products were isolated by simple evaporation of the filtrate and purified by crystallization from ethanol. It is important to note that acid-sensitive functional groups in the substrates (*Table 2*, **3v–3z**) remain unaffected. Unlike previous procedures, our method does not require the use of either hazardous acids or harsh reaction conditions; it has several advantages such as higher yields, shorter reaction times, and simple experimental and work-up procedures. In addition, polymer-like condensation products resulting from the self-condensation of *o*-aminoaryl ketones and aldehydes were not found. ^{14–17}

In order to demonstrate the efficiency of our method, we compared our results with some selected catalyst that have been reported in the recent literature. *Table 3* indicates that it is more efficient as a green, heterogeneous catalyst in terms of reaction times and yields.

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Experimental Section

IR spectra were recorded as KBr pellets on a Shimadzu 435-U-04 spectrophotometer. ¹H and ¹³C NMR spectra were obtained using Bruker DRX-300 Avance spectrometer in DMSO-d₆ or CDCl₃ using TMS as an internal reference. Melting points were determined in open capillary tubes in a Stuart BI Branstead Electrothermal Cat No: IA9200 apparatus and are uncorrected.

Preparation of Catalyst

Cross-linked *N*,*N*-methylene *bis*-acrylamide (MBA, 2 g) was added to a mixture of 2-acrylamido-2-methylpropanesulfonic acid (AMPS, 10 g) and acrylic acid (AA, 10 ml) in 100 ml distilled water. The mixture was vigorously stirred (magnetic stirrer) at room temperature for 10 min. Then, the solution was added to a three-neck flask equipped with a mechanical stirrer (Heidolph RZR 2021, three blade propeller type). The flask was immersed in a thermo-stated water bath at 70°C and an inert gas (nitrogen) was gently bubbled into the solution to remove any oxygen. After 15 min, an ammonium persulfate solution (APS, 0.2 g in 2 ml H₂O) was added to the mixture, and the mixture was stirred (200 rpm) for 20 min. Methanol (40 mL) was added and the mixture was stirred gently for 72 hrs in order to remove any unreacted monomer. After decantation of the methanol, the product was broken up into small pieces (mortar and pestle), dried in an oven at 110°C for five hours and then powdered (mortar and pestle). The powdered polymer was stored protected from moisture, heat and light.^{46–48}

General Procedure for the Synthesis of Quinolines

To mixture of α -methylene carbonyl compounds (1.2 mmol) and *o*-aminoaryl ketones or aldehydes (1 mmol) was added cross-linked poly(AMPS-co-AA) (0.06 g) [for solid substrates 0.1 ml of ethanol was added] and the mixture was heated on an oil bath at 110°C for the time show in *Table 2*. Upon completion of the reaction as indicated by TLC (hexane:ethyl acetate, 8:2), the appropriate amounts of hot EtOH (96%, 5 ml) was added and the mixture stirred for 10 min. After separation of the catalyst by filtration, the filtrate was concentrated in vacuum to remove the ethanol. The residue was washed with cold water and crystallized from hot ethanol (3 ml) to afford the pure products characterized by comparison between their mp, IR and NMR data with those of authentic samples.

Analytical Data for New Compounds

2-(4-Nitrophenyl)-4-phenylquinoline (3h), IR: 3052, 1600, 1580; ¹H NMR (300 MHz, DMSO-d₆): δ 7.57–7.64 (m, 5H), 7.83–7.91 (m, 2H), 8.18–8.22 (m, 2H), 9.36–9.39 (d, 2H), 9.61–9.64 (d, 2H); ¹³C NMR (75 MHz, DMSO-d₆): δ 119.30, 123.97, 125.38, 125.50, 120,76, 128.52, 128.78, 129.68, 130.00, 130.33, 132.29, 144.45, 148.09, 149.18, 153.47.

Anal. Calcd. for C₂₁H₁₄N₂O₂: C, 77.29; H, 4.33; N, 8.58. Found: C, 77.31; H, 4.32; N, 8.61.

2-(4-Fluorophenyl)-4-phenylquinoline (3i), IR: 3085, 1600, 1560; ¹H NMR (400 MHz, CDCl₃): 7.48–7.55 (m, 7H), 7.74–7.78 (m, 2H), 7.89–7.91 (d, 1H), 8.14–8.16 (d, 2H), 8.21–8.23 (d, 1H); ¹³C NMR (100 MHz, DMSO-d₆): δ 118.92, 125.70, 125.84,

126.56, 128.52, 128.65, 129.02, 129.71, 130.09, 135.58, 138.04, 138.27, 148.78, 149.44, 155.54.

Anal. Calcd. for C₂₁H₁₄FN: C, 84.26; H, 4.72; N, 4.68 Found: C, 84.29; H, 4.70; N, 4.65.

6-Chloro-2-(4-fluorophenyl)-4-phenylquinoline (3u), IR: 3085, 1600, 1560; ¹H-NMR (300 MHz, DMSO-d₆): δ 7.33–7.39 (t, 2H), 7.55–7.63 (m, 5H), 7.75 (s, 1H), 7.79–7.83 (d, 1H), 8.10 (s, 1H), 8.14–8.17 (d, 1H), 8.37–8.42 (t, 2H); ¹³C NMR (100 MHz, DMSO-d₆): δ 120.03, 124.50, 126.51, 127.56, 128.73, 128.84, 128.92, 129.44, 129.62, 130.46, 131.77, 132.17, 137.77, 139.22, 147.25, 148.25, 157.07.

Anal. Calcd. for C₂₁H₁₃ClFN: C, 75.56; H, 3.92; N, 4.28. Found: C, 75.56; H, 3.90; N 4.32.

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