

## A simple and green synthesis of highly functionalized quinoline derivatives using zinc oxide nanoparticles

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A simple and one pot method has been designed for the synthesis of highly substituted quinoline derivatives using ZnO nanoparticles in aqueous medium by means of three component condensation reaction of aromatic aldehydes, malononitrile and substituted anilines *via* Knoevenagel condensation followed by Michael addition in water. This new method has many advantages over reported methods like easy work-up, operational simplicity and excellent yields with short reaction time.

**Keywords:** Three component reaction, quinoline, ZnO nanoparticles

The quinoline ring occurs in a number of synthetic as well as in some natural products having interesting physical, biological and medicinal properties<sup>1</sup>. Since many years, a number of quinoline derivatives have been utilized for treating of a variety of diseases<sup>2</sup>. The quinoline compounds are also used in different fields such as polymer chemistry, optoelectronics, catalysts, corrosion inhibitors and in electronic media<sup>3</sup>. They constitute an important class of natural and non-natural products and also exhibit various chemical and biological activities<sup>4</sup>. Currently, a remarkable range of new quinoline alkaloids have been reported and considerable spectroscopic proof has been presented for most of the new structures<sup>5</sup>.

On the prime basis, the different quinoline derivatives have been synthesized by well known named reactions like Knorr synthesis<sup>6</sup>, Friedlander synthesis<sup>7</sup>, Skraup synthesis<sup>8</sup>, Miller synthesis<sup>9</sup>, Conard-Limpach synthesis<sup>10</sup> and Combes synthesis<sup>11</sup>.

Although there have been tremendous developments to obtain various kinds of quinoline derivatives as mentioned in the literature<sup>12-14</sup>, many of these reported methods suffer from one or more disadvantages like hazardous reaction conditions, tedious work-up procedures for isolation of desired products, long-duration and high temperature heating, use of volatile organic solvents and several of them are also reported as multistep syntheses with poor yield of the product.

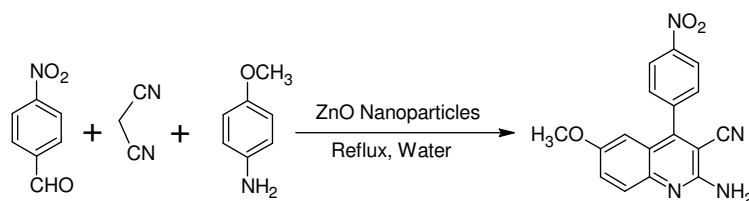
To overcome all the above mentioned difficulties, presently many chemists have successfully presented their work through multi-component reactions using water mediated MCRs in presence of metal oxide

nanoparticles<sup>15</sup>. MCRs play a very vital role in today's organic chemistry due to their ability to construct a complex molecule<sup>16</sup> in a single step from three or more components. MCRs obey most of the green chemistry principles like atom economy, no formation of side products<sup>17</sup>, mild reaction conditions<sup>18</sup>, and most of the time they take place in aqueous medium. So they are generally attempted in present synthetic organic chemistry for the synthesis of many heterocycles including a variety of quinoline derivatives<sup>19</sup>. MCRs when carried out in water are more advantageous as water is cheap, green, easily available and an economical solvent<sup>20</sup>. Moreover, if MCRs are carried out in water using metal oxide nanoparticles, it is interesting to study such reactions and it is today's hot topic of discussion for future chemistry<sup>21</sup>.

In view of this and in continuation with our previous success<sup>22</sup>, we wish to report synthesis of highly substituted quinoline derivatives using ZnO nanoparticles in aqueous medium. It is a facile, simple and rapid protocol for the synthesis of highly substituted quinoline derivatives by means of three component condensation reactions of aromatic aldehydes, malononitrile and substituted anilines *via* Knoevenagel condensation followed by Michael addition in water (Scheme I).

### Results and Discussion

In a model reaction, 4-nitrobenzaldehyde (1 mmol) and malononitrile (1 mmol) kept in a 50 mL round bottom flask containing ZnO nanoparticles as a



Scheme I — Synthesis of 2-amino-6-methoxy-4-(4-nitrophenyl)quinoline-3-carbonitrile

catalyst in 10 mL water and 4-methylaniline (1 mmol) in 10 mL water was refluxed using ZnO nanoparticles as a heterogeneous catalyst. The reaction mixture was stirred at RT for 3-5 min up to formation of the Knoevenagel product. After the completion of Knoevenagel condensation, aromatic amine (1 mmol) was added into the resulting reaction mixture and it was refluxed at 80°C till completion. The progress of the reaction was checked by TLC. After completion, the reaction mixture was filtered and washed thoroughly with 50 mL water to remove all water soluble impurities. The formation of 2-amino-6-methyl-4-(4-nitrophenyl)quinoline-3-carbonitrile was confirmed from the relevant spectroscopic data and physical constants from the reported literature. To optimize the reaction conditions, we tried out several combinations of catalyst and solvent and results are summarized in Table I.

It has been observed that in presence of synthesized ZnO nanoparticles, reaction was completed within short time giving higher yield of the product in water as compared with the other reaction conditions. The resultant trial reaction product was confirmed on the basis of spectroscopic data. The IR spectrum of synthesized 2-amino-6-methoxy-4-(4-nitrophenyl)quinoline-3-carbonitrile (Table II, Entry 5e) showed a distinguishing absorption peak at 2232  $\text{cm}^{-1}$  and 3360  $\text{cm}^{-1}$  which confirm the presence of  $-\text{CN}$  and  $-\text{NH}_2$  groups in the synthesized compound. The  $^1\text{H}$  NMR spectrum of 2-amino-6-methoxy-4-(4-nitrophenyl)quinoline-3-carbonitrile exhibited singlet at  $\delta$  3.86 for  $-\text{OCH}_3$  group, the two doublets of doublet appeared at  $\delta$  8.04-8.08 ( $J = 8.0$  Hz) and  $\delta$  8.30-8.34 ( $J = 8.0$  Hz) are due to the *ortho*-coupled aromatic four protons, while two doublet at  $\delta$  6.94-6.99 and at  $\delta$  7.29-7.34 are for two aromatic and two  $-\text{NH}_2$  protons respectively. The remaining uncoupled single aromatic proton appeared as singlet at  $\delta$  8.58.

This inspiring trial result promoted us to check the catalytic efficiency, reactivity and applicability of this method. To find out the scope and limitations of this protocol, in this study, we have worked out a wide

Table I — Screening of catalysts and solvents

Sr. No.	Catalyst	Solvent	Yield (%)
1	No catalyst	Water	No reaction
4	Bulk ZnO nanoparticles	Water	60
5	CuO nanoparticles	Water	70
6	ZnO nanoparticles	Water	90
7	ZnO nanoparticles	H <sub>2</sub> O:Ethanol	80
8	ZnO nanoparticles	Ethanol	76

range of highly substituted aromatic aldehydes, malononitrile and different substituted anilines. It has been observed that the reaction worked smoothly with electron-withdrawing as well as electron-releasing groups. All the variations give excellent yields of the products in shorter time durations using ZnO nanoparticles in water, as compared with other combinations of catalyst and solvent that were tried. All these results are put forth in Table II.

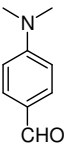
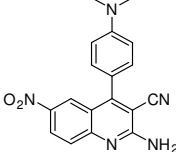
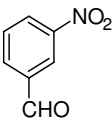
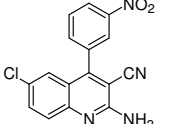
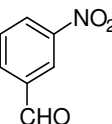
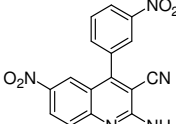
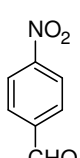
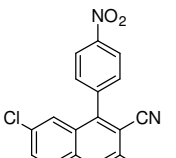
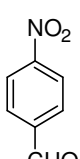
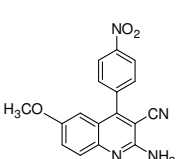
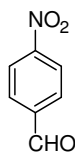
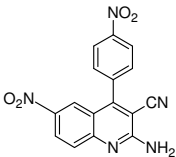
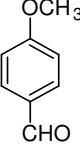
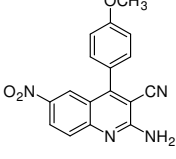
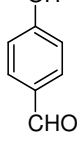
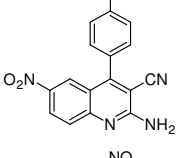
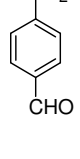
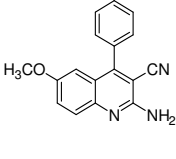
The proposed mechanism for this reaction is represented in Scheme II. The ZnO nanoparticles enable the Knoevenagel condensation type coupling between aromatic aldehydes and malononitrile through Lewis acid sites ( $\text{Zn}^{2+}$ ) coordinated to the oxygen of carbonyl groups.

Initially, the Knoevenagel condensation between aromatic aldehyde **1** and malononitrile **2** takes place to form an intermediate **3**. The Michael addition of aromatic amines takes place on formed Knoevenagel product **3**. The formed intermediate **4** undergoes further cyclization and aromatization to form the desired product **5**.

### Synthesis and characterization of ZnO (II) nanoparticles

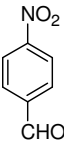
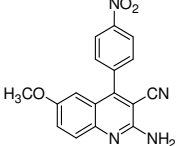
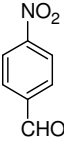
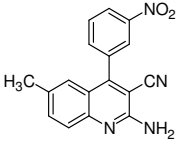
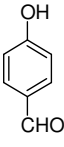
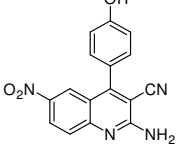
The ZnO nanoparticles were synthesized by reported co-precipitation method<sup>23</sup>. In a 250 mL beaker zinc acetate 0.02 mol was taken and portion-wise addition of 2.0 mol NaOH pellets was carried out until the pH reached to pH 12 and a white coloured precipitate was formed. Vigorous stirring was continued for 2 h. Finally, the solution was filtered and washed with distilled water and ethanol. The

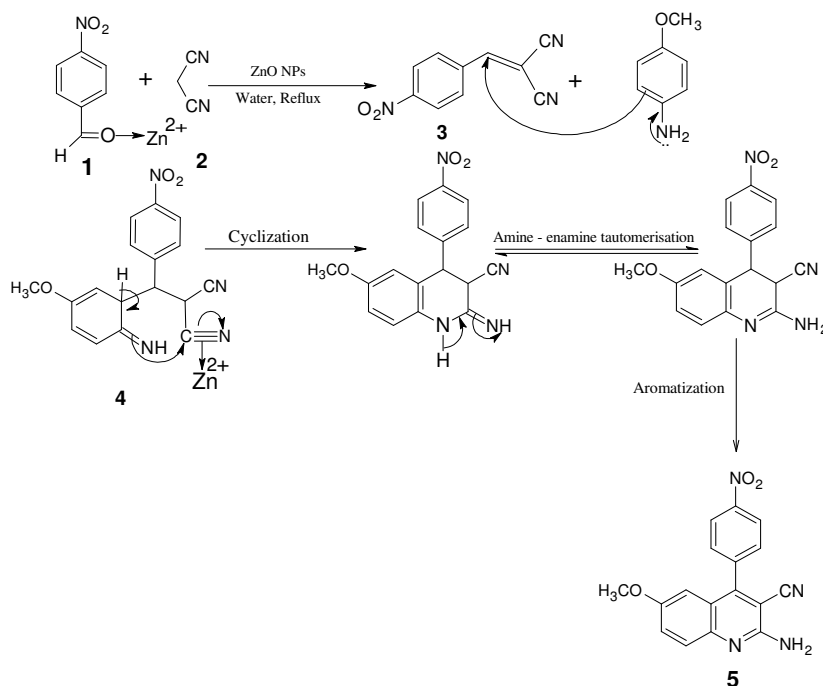
Table II — Synthesis of quinoline derivatives by using ZnO (II) nanoparticles in water

Entry	Aldehyde	Product	Time (minute)	Yield (%)	Catalyst	Solvent
1a			28	90	ZnO NPs	Water
2b			32	62	ZnO NPs	Water
3c			32	93	ZnO NPs	Water
4d			26	60	ZnO NPs	Water
5e			38	94	ZnO NPs	Water
6f			30	88	ZnO NPs	Water
7g			40	87	ZnO NPs	Water
8h			38	90	ZnO NPs	Water
9i			32	92	ZnO NPs	Water

(Contd.)

Table II — Synthesis of quinoline derivatives by using ZnO (II) nanoparticles in water (*Contd.*)

Entry	Aldehyde	Product	Time (minute)	Yield (%)	Catalyst	Solvent
10j			38	80	ZnO NPs	Water: Ethanol
11k			28	76	CuO NPs	Ethanol
12l			38	83	ZnO NPs	Water: Ethanol



Scheme II — Plausible mechanism of 2-amino-6-methoxy-4-(4-nitrophenyl) quinoline-3-carbonitrile

residue obtained was kept for drying in hot air oven at 120°C for about 12 h. The synthesized ZnO nanoparticles were characterized by powder X-ray diffraction spectrum and SEM.

### Scanning Electron Microscope (SEM)

The SEM technique is the most important characterization method for the morphological, structural and optical properties of nanoparticles (NPs). The morphological, structural and optical

properties of ZnO nanoparticles (NPs) were studied by using SEM analysis (Figure 1). When carried out at different magnifications it showed the agglomeration nature of ZnO NPs.

### Powder X-ray Diffraction (PXRD)

The crystallinity and crystal phase of the synthesized ZnO nanoparticles was characterized by powder X-ray diffraction (PXRD) (Table III). The high crystallinity of the prepared ZnO nanoparticles

was confirmed from the strong intensity and narrow width of the obtained diffraction peaks. The PXRD of

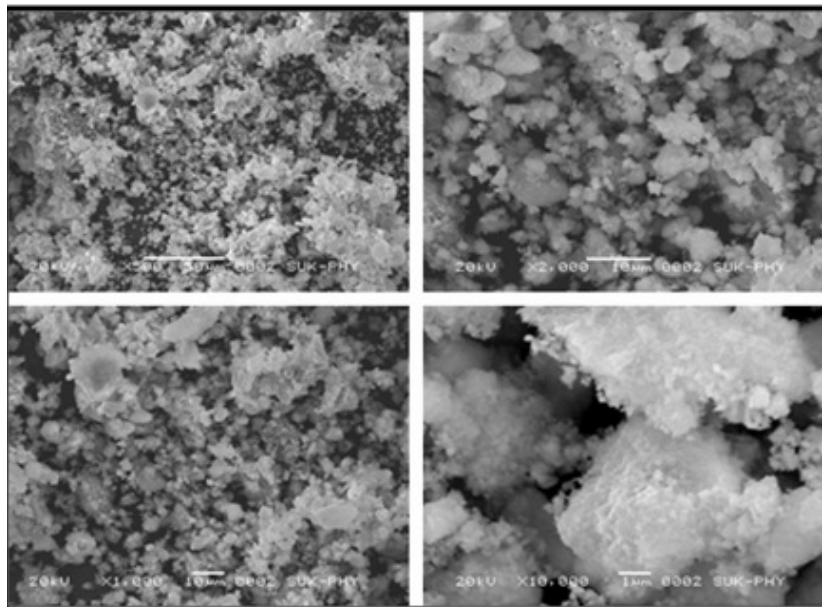


Figure 1 — SEM images of ZnO nanoparticles

Table III — PXRD patterns of ZnO nanoparticles

Sr. No.	Contents	PXRD of ZnO nanoparticles
1	JCPDS cards	JCPDS # 36-4151
2	Space group	P63 mc
3	Symmetry	Hexagonal
4	Diffraction peaks	100, 002, 101, 102, 2-10, 103, 200, 2-12, 201, 004, 202

prepared ZnO nanoparticles gave pure peaks having the hexagonal symmetry with P63mc space group and there was no any indication of any other impurity peaks. This PXRD matched with the standard JCPDS card (JCPDS #36-4151). There was no indication of peaks other than hexagonal ZnO phase hence it can be concluded from PXRD data that the ZnO nanoparticles synthesized are free from any impurities and phases are very pure.

### Experimental Section

The melting points are uncorrected and were determined in an open capillary tube. All chemicals were purchased from Merck and Sigma Aldrich. The  $^1\text{H}$  NMR were recorded on Bruker Spectrospin Avance 200 MHz and 300 MHz spectrometers using  $\text{CDCl}_3$  and  $\text{DMSO}-d_6$  as solvent and tetramethyl silane (TMS) as an internal standard. The  $^{13}\text{C}$  NMR spectra (75 MHz and 50 MHz) were recorded on

Bruker Spectrospin Avance 200 MHz and 300 MHz using  $\text{CDCl}_3$  and  $\text{DMSO}-d_6$  solvent. The homogeneity of the compounds were checked by thin layer chromatography (TLC).

### General experimental procedure for synthesis of quinoline derivatives

A mixture of aromatic aldehyde (1 mmol) and malononitrile (1 mmol) were kept in a 50 mL round bottom flask containing ZnO nanoparticles as a catalyst in 10 mL water. The reaction mixture was stirred at RT for 3-5 min. till the formation Knoevenagel product. After the completion of Knoevenagel condensation, aromatic amine (1 mmol) was added into the resulting reaction mixture and it was refluxed at  $80^\circ\text{C}$  till completion. The reaction was monitored by thin layer chromatography (TLC). After completion of reaction, the reaction mixture was filtered and the crude solid washed with distilled water to afford the final product.

### Spectroscopic data of representative synthesized quinoline derivatives

**2-Amino-4-[4-(dimethylamino)phenyl]-6-nitroquinoline-3-carbonitrile, 1a** (Table II): Yellow solid. Yield 90%. m.p.  $190-92^\circ\text{C}$  (dec). IR: 2234, 3347, 1560  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.14 (s, 6H,  $-\text{N}(\text{CH}_3)_2$ ), 6.60-6.71 (m, 3H, Ar-H), 7.26 (s, 2H,  $-\text{NH}_2$ ), 7.47 (s, 1H, Ar-H), 7.80-7.84 (dd, 2H, Ar-H),

8.05-8.10 (dd,  $J = 10.0$  Hz, 1H, Ar-H);  $^{13}\text{C}$  NMR (50 MHz,  $\text{CDCl}_3$ ):  $\delta$  40.68, 112.22, 126.95, 134.40, 154.88, 158.71. Anal. Calcd for  $\text{C}_{18}\text{H}_{15}\text{N}_5\text{O}_2$ : C, 64.86; H, 4.54; N, 21.01. Found: C, 64.87; H, 4.52; N, 21.04%.

**2-Amino-6-chloro-4-(3-nitrophenyl)quinoline-3-carbonitrile, 2b** (Table II): Yellow solid. Yield 62%. m.p.150-52°C (dec). IR: 2228, 3328, 1520  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.18-7.22 (dd,  $J = 8.0$  Hz 2H, Ar-H), 7.38 (s, 2H,  $-\text{NH}_2$ ), 7.64-7.72 (m, 1H, Ar-H), 8.23-8.32 (m, 2H, Ar-H), 8.53 (s, 1H, Ar-H), 8.74 (m, 1H, Ar-H);  $^{13}\text{C}$  NMR (50 MHz,  $\text{CDCl}_3$ ):  $\delta$  84.22, 122.51, 124.45, 129.86, 134.86, 148.08, 153.78, 157.36. Anal. Calcd for  $\text{C}_{16}\text{H}_9\text{ClN}_4\text{O}_2$ : C, 59.18; H, 2.79; N, 17.25. Found: C, 59.20; H, 2.77; N, 17.24%.

**2-Amino-6-nitro-4-(3-nitrophenyl)quinoline-3-carbonitrile, 3c** (Table II): Yellow solid, Yield 93%. m.p.176-78°C (dec). IR: 2222, 3367, 1570  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  4.37 (br.s, 2H,  $-\text{NH}_2$ ), 6.61-6.66 (dd, 2H, Ar-H), 7.27 (s, 3H, Ar-H), 8.06-8.11 (dd, 2H, Ar-H);  $^{13}\text{C}$  NMR (50 MHz,  $\text{CDCl}_3$ ):  $\delta$  15.41, 122.63, 128.38, 128.85, 129.66, 134.24, 140.05, 148.82, 157.73. Anal. Calcd For  $\text{C}_{16}\text{H}_9\text{N}_5\text{O}_4$ : C, 57.32; H, 2.71; N, 20.89. Found: C, 57.35; H, 2.75; N, 20.87%.

**2-Amino-6-chloro-4-(4-nitrophenyl)quinoline-3-carbonitrile, 4d** (Table II): Yellow solid. Yield 60%. m.p.242-44°C (Lit.240-45°C<sup>17</sup>) (dec). IR: 2227, 3039, 1579  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.17-7.23 (m, 2H, Ar-H), 7.38-7.42 (d, 2H,  $-\text{NH}_2$ ), 8.05-8.10 (dd,  $J = 10.0$  Hz 2H, Ar-H), 8.32-8.36 (dd,  $J = 10.0$  Hz, 2H, Ar-H), 8.54 (s, 1H, Ar-H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  84.22, 122.51, 124.45, 129.86, 134.86, 148.08, 153.78, 157.36. Anal. Calcd for  $\text{C}_{16}\text{H}_9\text{ClN}_4\text{O}_2$ : C, 59.18; H, 2.79; N, 17.25. Found: C, 59.19; H, 2.78; N, 17.28%.

**2-Amino-6-methoxy-4-(4-nitrophenyl)quinoline-3-carbonitrile, 5e** (Table II): Orange solid. Yield 94%. m.p.134-36°C (Lit.134°C<sup>17</sup>) (dec). IR: 2232, 3120, 3360  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.86 (s, 3H,  $-\text{OCH}_3$ ), 6.94-6.99 (dd, 2H, Ar-H), 7.29-7.34 (d, 2H,  $-\text{NH}_2$ ), 8.04-8.08 (dd,  $J = 8.0$  Hz, 2H, Ar-H), 8.30-8.34 (dd,  $J = 8.0$  Hz, 2H, Ar-H), 8.58 (s, 1H, Ar-H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  55.52, 114.58, 122.58, 123.96, 129.06, 141.99, 143.68, 149.09, 154.72, 159.29. Anal. Calcd for  $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}_3$ : C, 63.75; H, 3.78; N, 17.49. Found: C, 63.74; H, 3.79; N, 17.46%.

**2-Amino-6-nitro-4-(4-nitrophenyl)quinoline-3-carbonitrile, 6f** (Table II): Yellow solid. Yield 88%. m.p.154-56°C (Lit.155°C<sup>17</sup>) (dec). IR: 2221, 3357  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  6.60-6.65 (dd, 2H,  $J = 10.0$  Hz, Ar-H), 7.88 (s, 1H,  $-\text{Ar-H}$ ), 8.05-8.10

(dd, 4H,  $-\text{NH}_2$ , Ar-H), 8.37-8.42 (dd,  $J = 10.0$  Hz, Ar-H). Anal. Calcd for  $\text{C}_{16}\text{H}_9\text{N}_5\text{O}_4$ : C, 57.32; H, 2.71; N, 20.89. Found: C, 57.33; H, 2.74; N, 20.92%.

**2-Amino-4-(4-methoxyphenyl)-6-nitroquinoline-3-carbonitrile, 7g** (Table II): Yellow solid. Yield 87%. m.p.92-94°C (Lit.91°C<sup>17</sup>) (dec);  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.92 (s, 3H,  $-\text{OCH}_3$ ), 6.60-6.65 (dd,  $J = 10.0$  Hz 1H, Ar-H), 6.99-7.04 (dd, 2H, Ar-H), 7.26 (s, 2H,  $-\text{NH}_2$ ), 7.65 (s, 1H, Ar-H) 7.89-7.94 (dd,  $J = 10.0$  Hz, 2H, Ar-H), 8.05-8.10 (dd, 1H, Ar-H). Anal. Calcd for  $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}_3$ : C, 63.75; H, 3.78; N, 17.49%. Found: C, 63.76; H, 3.75; N, 17.52%.

**2-Amino-4-(4-hydroxyphenyl)-6-nitroquinoline-3-carbonitrile, 8h** (Table II): Yellow solid. Yield 90%. m.p.128-30°C (Lit.130°C<sup>17</sup>) (dec). IR: 2228, 3360  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  4.38 (br. s, 2H,  $-\text{NH}_2$ ), 6.13 (br. s, 1H,  $-\text{OH}$ ), 6.63-6.65 (dd, 2H, Ar-H), 6.97-6.99 (dd, 1H, Ar-H), 7.27-7.28 (d, 1H, Ar-H), 7.88-7.90 (dd, 1H, Ar-H), 8.08-8.10 (dd, 2H, Ar-H). Anal. Calcd for  $\text{C}_{16}\text{H}_{10}\text{N}_4\text{O}_3$ : C, 62.74; H, 3.29; N, 18.29. Found: C, 62.71; H, 3.32; N, 18.31%.

**2-Amino-6-methoxy-4-(3-nitrophenyl)quinoline-3-carbonitrile, 9i** (Table II): Yellow solid. Yield 86%. m.p.86-88°C (Lit.83-88°C<sup>17</sup>) (dec). IR: 2228, 3039  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.85 (s, 3H,  $-\text{OCH}_3$ ), 6.94-7.00 (m, 2H, Ar-H), 7.28-7.32 (m, 2H,  $-\text{NH}_2$ ), 7.61-7.69 (m, 1H, Ar-H), 8.22-8.29 (m, 2H, Ar-H), 8.57 (s, 1H, Ar-H), 8.73-8.74 (d, 1H, Ar-H). Anal. Calcd for  $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}_3$ : C, 63.75; H, 3.78; N, 17.49. Found: C, 63.76; H, 3.76; N, 17.52%.

## Conclusion

In summary, we have reported an efficient, simple and three component synthesis of highly substituted quinoline derivatives in excellent yields *via* three component reaction of aromatic aldehydes, malononitrile and aromatic amines. This new method has many advantages over reported methods like easy work-up, operational simplicity, and excellent yields with short reaction time.

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