

**ULTRASONICALLY ASSISTED SYNTHESIS AND CHARACTERIZATION OF  
ETHYL 5-CHLORO-2-OXO-1-((1-SUBSTITUTED-1H-1,2,3-TRIAZOL-4-  
YL)METHYL)-1,2-DIHYDROQUINOLINE-3-CARBOXYLATES**

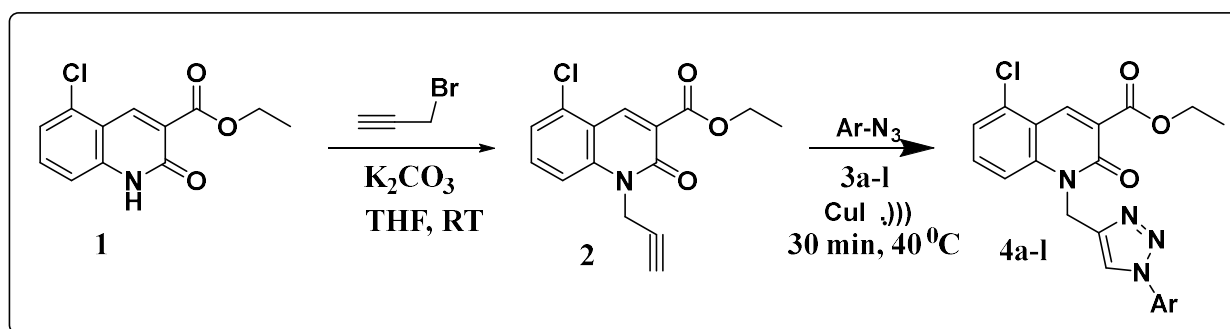
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**Abstract:** Herein, we report the treatment of ethyl 5-chloro-2-oxo-1,2-dihydroquinoline-3-carboxylate **1** with 3-bromoprop-1-yne to give desired ethyl 5-chloro-2-oxo-1-(prop-2-yn-1-yl)-1,2-dihydroquinoline-3-carboxylate **2** on N-H propargylation. It is further treated with series of azides **3a-l**, CuI as key catalyst under ultra sonications to give desired products 1,2,3-triazole fused dihydroquinolines **4a-l** through cycloaddition as design targets.

**Keywords:** propargyl bromide, copper (I) iodide, cyclo addition, 1,2,3-triazoles, quinolines.

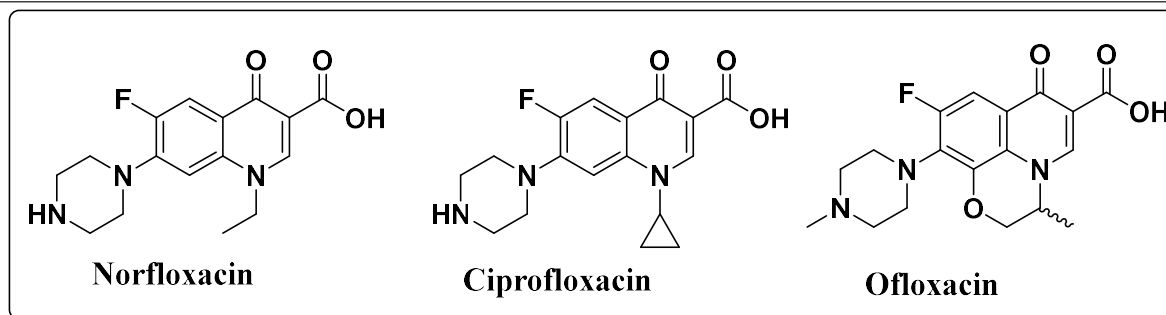


**Scheme I.** Synthetic path way for target compounds **4a-l**

## 1. INTRODUCTION

One of the main classes of heterocycles containing nitrogen is quinolone. They are omnipresent and one of the most essential pharmacophores in contemporary drug design, which is crucial in the development of novel drugs. The extensive variety of chemotherapeutic drugs frequently includes the quinolone-based structural unit [1]. Due to their wide range of biological actions, quinolone-based medicines have received a great deal of attention among heterocyclic compounds [2]. This is the exact reason that medicinal chemists and pharmacists, respectively, devote special attention to the design and development of these agents as well as their pharmacological evaluation.

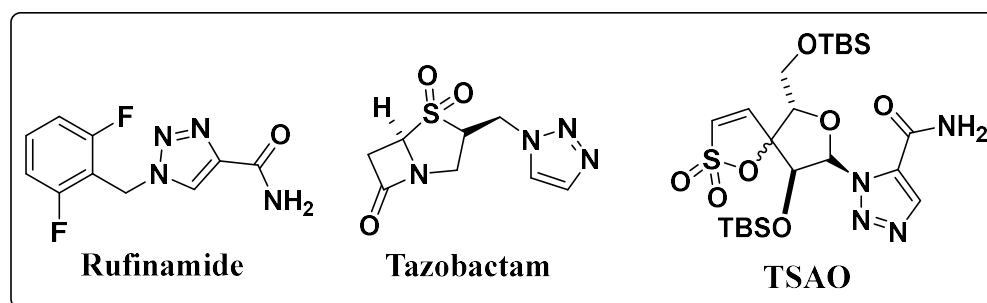
Unfortunately, the rising incidence of resistance, which has been observed in every species that is treated by this drug family, poses a threat to the use of quinolones. [3–6] The bacterial type II topoisomerases, gyrase, and topoisomerase IV are the targets for quinolones in cells. [7–9]



**Fig. 1** Norfloxacin, ciprofloxacin, and ofloxacin are the second-generation quinolones

Quinolones take advantage of this latter, potentially lethal, characteristic and kill cells by increasing the concentration of enzyme-DNA cleavage complexes. [10] Thus, these drugs are termed “topoisomerase poisons” because they convert gyrase and topoisomerase IV into cellular toxins. [11]

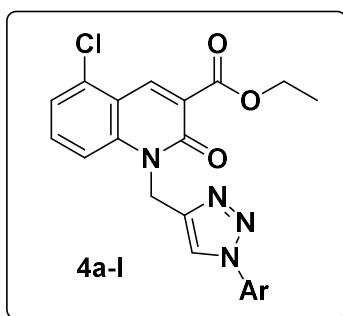
Quinolines and 1,2,3-triazoles are two important categories of small heterocyclic compounds, which are among the numerous N-heterocycles and have a variety of chemotherapeutic implications. Surprisingly, even though 1,2,3-triazoles, which are chemically inert chemicals, have not been found in naturally occurring products [13–15], quinoline molecules frequently occur in living organisms as significant secondary metabolites [12]. However, the quinoline and 1,2,3-triazole skeletons are each present in a wide variety of conjugated hybrids, which are important biomolecules for drug discovery.



**Fig. 2** 1,2,3- triazole based drugs

The modified Huisgen reaction between quinoline with a terminal alkyne group and an organoazide via a catalytic 1, 3-dipolar cycloaddition process is one of the most significant and useful methods for the 4-(quinolinyl)-1, 2,3-triazoles [16]. Noteworthy is that the Huisgen reaction, which occurs without any catalysts, gives poorly only a regioisomeric mixture of products that reduces its practical application [17], while Meldal and Fokin-Sharpless modified protocols [18, 19] that use copper catalysts (CuI, CuBr, CuSO<sub>4</sub>) allow obtaining exclusively 1,4-disubstituted 1,2,3-triazoles.

Herein, we report the synthesis of Ethyl 5-chloro-2-oxo-1-((1-substituted-1*H*-1,2,3-triazol-4-yl)methyl)-1,2-dihydroquinoline-3-carboxylates are shown below.



**Fig: 3** 1,2,3-triazole fused dihydroquinoline

## 2. EXPERIMENTAL SECTION

### 2.1 General information

All reactions were carried out in a round bottom flask under room temperature. Copper (I) iodide, heterocyclic compounds, and propargyl bromide were purchased from Aldrich chemical company. All the reagents and solvents were purchased from S.D. Fine chemicals limited and used without further purification. Thin-layer chromatography (TLC) was performed using Merck silica gel 60F254 precoated plates (0.25 mm) and silica gel (particle size 60-120 mesh) was used for column chromatography. Ultrasoni Sonicator 20 kHz of 300 W, 40% wave amplitude is used.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a 400 MHz instrument. Chemical shift values are given in  $\delta$  (ppm) with tetramethyl silane as an internal standard and the coupling constant values ( $J$ ) are in Hz. Signals are expressed as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and brs (broad singlet). Mass spectral measurements were carried out by EI method on a Jeol JMC-300 spectrometer at 70eV. Elemental analyses were performed on a Carlo Erba 106 and Perkin Elmer model 240 analyzers. Mass spectra were recorded on ESI-MS.

### 2.2 General Procedure for the Synthesis of 1,4- Disubstituted 1,2,3-triazole fused dihydroquinolines 4a-l:

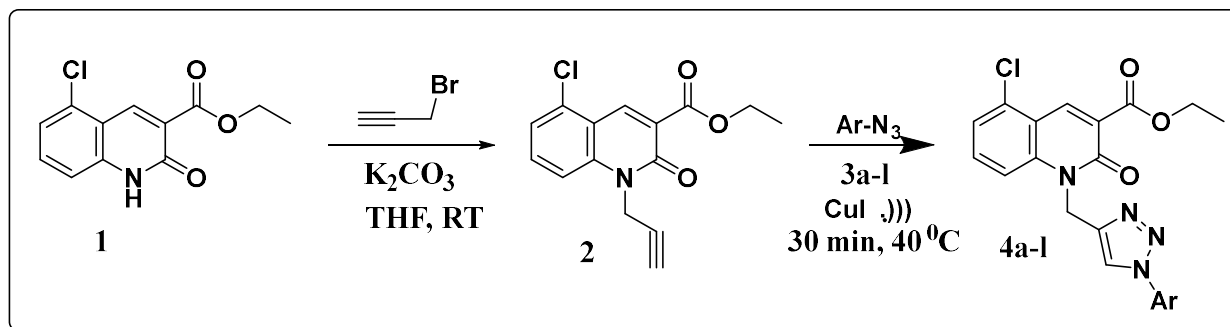
To a solution of ethyl 5-chloro-2-oxo-1,2-dihydroquinoline-3-carboxylate **1** (24.03 mmol) in THF (48.06 mmol) and  $\text{K}_2\text{CO}_3$  was added. After 5 minutes, propargyl bromide or 3-bromoprop-1-yne (31.25 mmol) was added to reaction mass and the reaction was stirred at room temperature for 4 h. After the completion of reaction by TLC analysis, ice-cold water (100 mL) was added and extracted with ethyl acetate (3X 50 mL). The organic layer was dried out over  $\text{Na}_2\text{SO}_4$  and evaporated to afford desired compound ethyl 5-chloro-2-oxo-1-(prop-2-yn-1-yl)-1,2-dihydroquinoline-3-carboxylate **2**.

Furthermore ethyl-5-chloro-2-oxo-1-(prop-2-yn-1-yl)-1,2-dihydroquinoline-3-carboxylate **2** (0.50 mmol), series of azides **3a-l** (0.50 mmol), and  $\text{CuI}$  (1 mol %) in distilled water (2.0 mL) was placed in a 25 mL two-neck flask. The mixture was directly irradiated with an ultrasonic probe at

60 °C and maintained at this temperature for 30 min. The ultrasound probe was placed in one neck, and the temperature sensor was placed in the other. The resulting precipitate was filtered, washed with H<sub>2</sub>O, and then purified by flash chromatography on silica gel (eluent/ eluent: CH<sub>2</sub>Cl<sub>2</sub>/CH<sub>3</sub>OH 50:1 to 100:1 v/v) to afford the pure 1,2,3-triazole fused dihydroquinolines **4a–l** through cycloaddition.

### 3. RESULTS AND DISCUSSIONS

A different series of 1,4- Disubstituted 1,2,3-triazole fused dihydroquinolines through Ultrasonically assisted synthesis led to promising yields described in Scheme I.

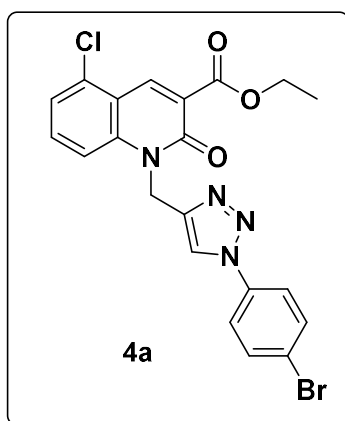


Scheme I

#### Ethyl-1-((1-(4-bromophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-5-chloro-2-oxo-1,2-

**dihydroquinoline-3-carboxylate (4a):** MF: C<sub>21</sub>H<sub>16</sub>BrClN<sub>4</sub>O<sub>3</sub>, Light orange, yield: 90%;

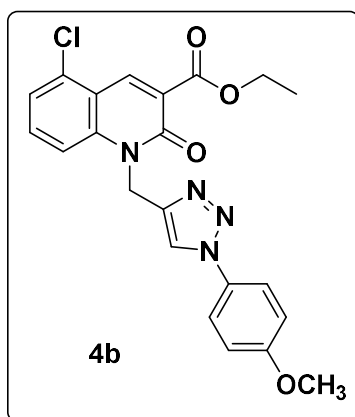
m.p. 149-151 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm): 8.33 (s, 1H), 8.08 (s, 1H), 7.62 – 7.39 (m, 6H), 7.32 (d, *J* = 7.3 Hz, 1H), 5.53 (s, 1H), 5.14 (s, 1H), 4.22 (d, *J* = 17.9 Hz, 2H), 1.41 (s, 3H); <sup>13</sup>CNMR(100 MHz, CDCl<sub>3</sub>) δ(ppm): δ 164.32, 163.61, 155.61, 139.85, 138.14, 135.45, 132.12, 128.31, 124.09, 122.79, 121.80, 120.54, 119.82, 115.43, 61.50, 37.34, 14.69; ESI [M+H]<sup>+</sup>: 486.01; Elemental Analysis (%): Found: C, 51.83; H, 3.32; N, 11.51, (Calcd): (C, 51.71; H, 3.31; N, 11.49).



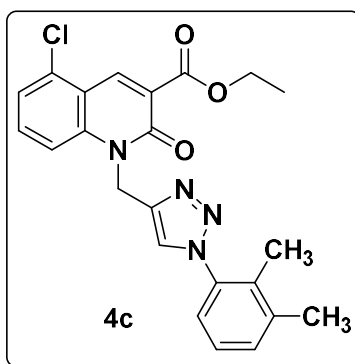
#### Ethyl-5-chloro-1-((1-(4-methoxyphenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-2-oxo-1,2-

**dihydroquinoline-3-carboxylate (4b):** MF: C<sub>22</sub>H<sub>19</sub>ClN<sub>4</sub>O<sub>4</sub>, Colourless solid, yield: 88%;

m.p. 114-116°C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ (ppm): 8.33 (s, 1H), 8.08 (s, 1H), 7.66 – 7.25 (m, 5H), 6.84 (s, 2H), 5.55 (s, 1H), 5.14 (s, 1H), 4.23 (d,  $J = 18.0$  Hz, 2H), 3.81 (s, 3H), 1.39 (s, 3H).  $^{13}\text{CNMR}$ (100 MHz,  $\text{CDCl}_3$ )  $\delta$ (ppm): 164.32, 163.61, 157.83, 155.61, 139.85, 138.14, 132.12, 131.16, 128.31, 124.09, 122.60, 119.82, 115.43, 114.61, 61.50, 56.03, 37.34, 14.69. **ESI**  $[\text{M}+\text{H}]^+$ :438.11. **Elemental Analysis**: (%) Found: C, 60.33; H, 4.37; N, 12.79 (Calcd): (C, 60.21; H, 4.36; N, 12.77).

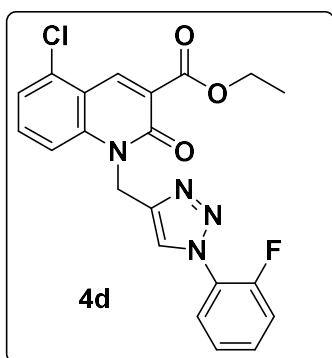


**Ethyl-5-chloro-1-((1-(2,3-dimethylphenyl)-1H-1,2,3-triazol-4-yl)methyl)-2-oxo-1,2-dihydroquinoline-3-carboxylate (4c)**: **MF**:  $\text{C}_{23}\text{H}_{21}\text{ClN}_4\text{O}_3$ ; Pale white solid, yield: 80%; m.p. 104-106°C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ (ppm): 8.29 (s, 1H), 8.08 (s, 1H), 7.45 (dd,  $J = 7.4$  Hz, 4H), 7.04 (d,  $J = 7.5$  Hz, 1H), 6.85 (d,  $J = 8.9$  Hz, 1H), 5.78 (s, 1H), 5.21 (s, 1H), 4.26 (s, 2H), 2.37 (d,  $J = 30.6$  Hz, 6H), 1.38 (s, 3H);  $^{13}\text{CNMR}$ (100 MHz,  $\text{CDCl}_3$ )  $\delta$ (ppm): 164.32, 163.61, 155.61, 139.90, 138.86, 138.14, 132.12, 131.04, 128.31, 126.78, 124.14, 122.93, 119.82, 117.30, 115.43, 61.50, 37.34, 19.77, 15.33, 14.69; **ESI**  $[\text{M}+\text{H}]^+$ : 436.13; **Elemental Analysis** (%): Found: C, 63.14; H, 4.86; N, 12.84, (Calcd): (C, 63.23; H, 4.85; N, 12.82).

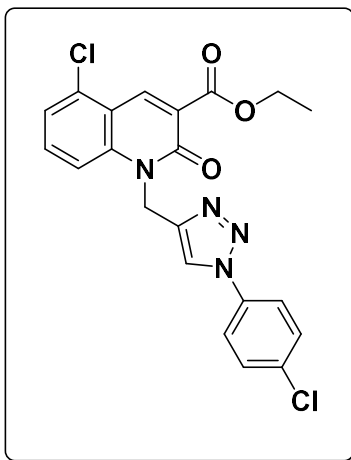


**Ethyl-5-chloro-1-((1-(2-fluorophenyl)-1H-1,2,3-triazol-4-yl)methyl)-2-oxo-1,2-dihydroquinoline-3-carboxylate (4d)**: **MF**:  $\text{C}_{21}\text{H}_{16}\text{ClFN}_4\text{O}_3$ ; Off-white solid, yield: 90%; m.p. 124-126°C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ (ppm): 8.33 (s, 1H), 8.08 (s, 1H), 7.74 (d,  $J = 5.6$  Hz, 1H), 7.66 – 7.20 (m, 3H), 7.17 – 6.96 (m, 3H), 5.53 (s, 1H), 5.14 (s, 1H), 4.38 – 4.05 (m, 2H), 1.40 (t,  $J = 6.0$  Hz, 3H);  $^{13}\text{CNMR}$ (100 MHz,  $\text{CDCl}_3$ )  $\delta$ (ppm): 164.32, 163.61, 159.57, 157.47, 155.61, 139.90, 138.14, 132.12, 128.31, 128.03, 127.76, 127.54, 125.71, 124.09, 122.85, 119.82,

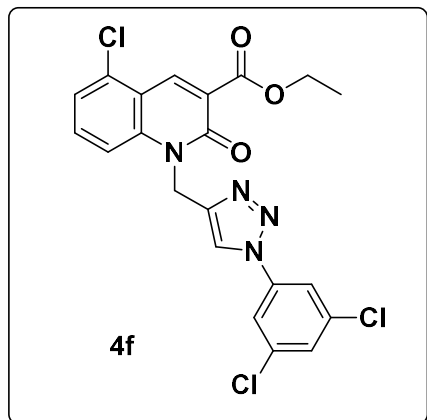
118.01, 117.80, 115.43, 61.50, 37.34, 14.69; **ESI [M+H]**: 426.09; **Elemental Analysis (%)**: Found: C, 59.23; H, 3.79; N, 13.15, (Calcd): (C, 59.09; H, 3.78; N, 13.13).



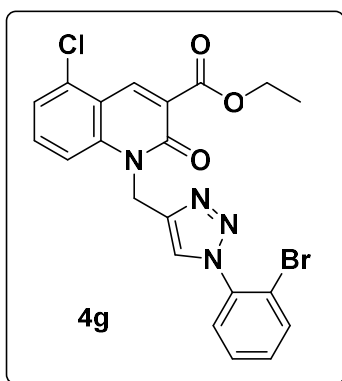
**Ethyl-5-chloro-1-((1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)methyl)-2-oxo-1,2-dihydroquinoline-3-carboxylate (4e)**: **MF**: C<sub>21</sub>H<sub>16</sub>Cl<sub>2</sub>N<sub>4</sub>O<sub>3</sub>; Pale-white solid, yield: 78%; m.p. 149-151°C; **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm)**: 8.32 (s, 1H), 8.08 (s, 1H), 7.64 – 7.18 (m, 7H), 5.53 (s, 1H), 5.14 (s, 1H), 4.22 (q, *J* = 6.0 Hz, 2H), 1.40 (t, *J* = 6.0 Hz, 3H); **<sup>13</sup>CNMR(100 MHz, CDCl<sub>3</sub>) δ(ppm)**: 164.32, 163.61, 155.61, 139.85, 138.14, 136.01, 132.65, 132.12, 129.01, 128.31, 124.09, 122.79, 119.82, 115.43, 61.50, 37.34, 14.69; **ESI [M+H]<sup>+</sup>**: 442.06; **Elemental Analysis (%)**: Found: C, 60.03; H, 3.65; N, 12.66, (Calcd): (C, 56.90; H, 3.64; N, 12.64).



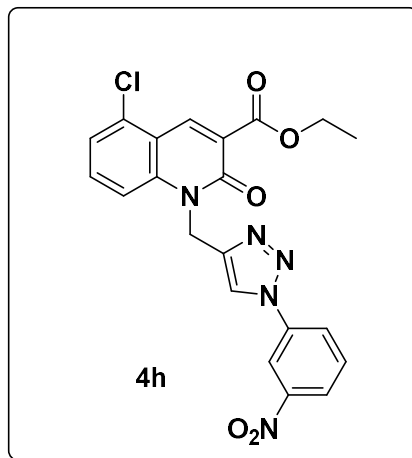
**Ethyl-5-chloro-1-((1-(3,5-dichlorophenyl)-1H-1,2,3-triazol-4-yl)methyl)-2-oxo-1,2-dihydroquinoline-3-carboxylate (4f)**: **MF**: C<sub>21</sub>H<sub>15</sub>Cl<sub>3</sub>N<sub>4</sub>O<sub>3</sub>; White solid, yield: 76%; m.p. 169-171°C; **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm)**: 8.32 (s, 1H), 8.08 (s, 1H), 7.64 (d, *J* = 1.4 Hz, 2H), 7.41 (dd, *J* = 109.6, 33.0, 23.5 Hz, 4H), 5.53 (s, 1H), 5.14 (s, 1H), 4.22 (q, *J* = 6.0 Hz, 2H), 1.41 (s, 3H); **<sup>13</sup>CNMR(100 MHz, CDCl<sub>3</sub>) δ(ppm)**: 164.32, 163.61, 155.61, 139.85, 138.14, 135.29, 132.12, 128.31, 124.09, 122.60, 119.82, 118.71, 115.43, 61.50, 37.34, 14.69; **ESI [M+H]<sup>+</sup>**: **476.02**; **Elemental Analysis (%)**: Found: C, 52.92; H, 3.17; N, 11.75; (Calcd): (C, 52.80; H, 3.16; N, 11.73).



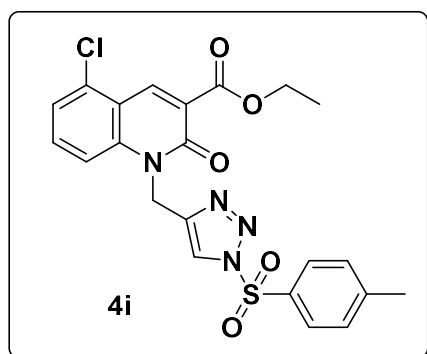
**Ethyl-1-((1-(2-bromophenyl)-1H-1,2,3-triazol-4-yl)methyl)-5-chloro-2-oxo-1,2-dihydroquinoline-3-carboxylate (4g):** MF: C<sub>21</sub>H<sub>16</sub>BrClN<sub>4</sub>O<sub>3</sub>; Off-Yellow solid, yield: 76%; m.p. 119-121°C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm): 8.33 (s, 1H), 8.08 (s, 1H), 7.74 (d, *J* = 7.5 Hz, 1H), 7.61 – 7.41 (m, 3H), 7.39 – 7.18 (m, 2H), 7.04 (t, *J* = 8.2 Hz, 1H), 5.54 (s, 1H), 5.14 (s, 1H), 4.22 (q, *J* = 6.0 Hz, 2H), 1.39 (d, *J* = 12.0 Hz, 3H); <sup>13</sup>CNMR(100 MHz, CDCl<sub>3</sub>) δ(ppm): 164.32, 163.61, 155.61, 139.90, 137.93, 132.10, 129.03, 128.31, 126.24, 124.71, 124.09, 122.86, 119.82, 115.43, 114.90, 61.50, 37.34, 14.69; ESI [M+H]<sup>+</sup>: 486.01; Elemental Analysis (%): Found: C, 51.83; H, 3.32; N, 11.51 ; (Calcd): (C, 51.71; H, 3.31; N, 11.49).



**Ethyl-5-chloro-1-((1-(3-nitrophenyl)-1H-1,2,3-triazol-4-yl)methyl)-2-oxo-1,2-dihydroquinoline-3-carboxylate (4h):** MF: C<sub>21</sub>H<sub>16</sub>ClN<sub>5</sub>O<sub>5</sub>; Pale-White solid, yield: 80%; m.p. 116-118°C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm): 8.56 (s, 1H), 8.34 (s, 1H), 8.08 (s, 1H), 7.92 – 7.62 (m, 2H), 7.62 – 7.23 (m, 4H), 5.52 (s, 1H), 5.14 (s, 1H), 4.22 (q, *J* = 6.0 Hz, 2H), 1.39 (s, 3H); <sup>13</sup>CNMR(100 MHz, CDCl<sub>3</sub>) δ(ppm): 164.32, 163.61, 155.61, 147.64, 139.85, 137.68, 132.12, 131.09, 128.31, 127.95, 124.09, 121.48, 119.82, 115.43, 61.50, 37.34, 14.69; ESI [M+H]<sup>+</sup>: 453.08 ; Elemental Analysis (%): Found: C, 55.70; H, 3.56; N, 15.45 ; (Calcd): (C, 55.58; H, 3.55; N, 15.43).

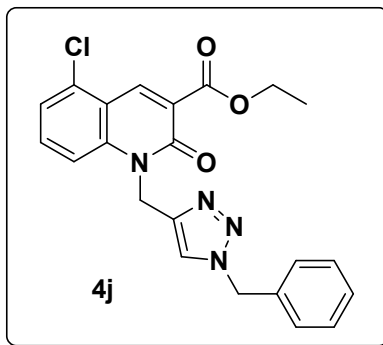


**Ethyl-5-chloro-2-oxo-1-((1-(tosyl-1*H*-1,2,3-triazol-4-yl)methyl)-1,2-dihydroquinoline-3-carboxylate (4i):** MF: C<sub>22</sub>H<sub>19</sub>ClN<sub>4</sub>O<sub>5</sub>S; Colourless solid, yield: 90%; m.p. 134-136°C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm): 9.01 (s, 1H), 8.35 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.62 – 7.23 (m, 5H), 5.61 (s, 1H), 5.14 (s, 1H), 4.23 (q, *J* = 6.0 Hz, 2H), 2.35 (s, 3H), 1.39 (s, 3H); <sup>13</sup>CNMR(100 MHz, CDCl<sub>3</sub>) δ(ppm): 164.32, 163.61, 155.61, 148.92, 143.75, 138.14, 137.76, 132.12, 130.29, 128.01, 126.86, 124.09, 122.79, 119.82, 115.43, 61.50, 37.34, 21.12, 14.69; ESI [M+H]<sup>+</sup>: 486.08; Elemental Analysis (%): Found: C, 54.39; H, 3.94; N, 11.53; (Calcd): (C, 54.27; H, 3.93; N, 11.51).

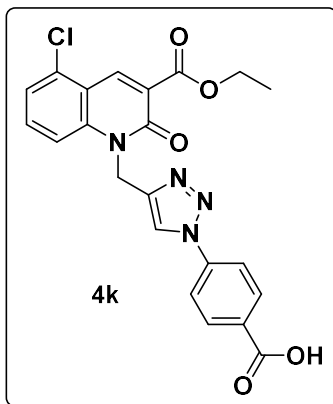


**Ethyl-1-((1-(benzyl-1*H*-1,2,3-triazol-4-yl)methyl)-5-chloro-2-oxo-1,2-dihydroquinoline-3-carboxylate (4j):** MF: C<sub>22</sub>H<sub>19</sub>ClN<sub>4</sub>O<sub>3</sub>; Colourless solid, yield: 80%; m.p. 169-171°C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm): 8.26 (s, 1H), 7.63 (s, 1H), 7.56 – 7.04 (m, 8H), 5.68 (s, 1H), 5.33 – 4.90 (m, 3H), 4.24 (q, *J* = 5.9 Hz, 2H), 1.37 (s, 3H); <sup>13</sup>CNMR(100 MHz, CDCl<sub>3</sub>) δ(ppm): 164.32, 163.61, 155.61, 138.73, 138.14, 136.22, 132.12, 128.87, 128.57, 127.54, 125.90, 124.09, 122.79, 119.82, 115.43, 61.50, 50.17, 37.34, 14.69; ESI [M+H]<sup>+</sup>: 422.11; Elemental Analysis (%): Found: C, 62.51; H, 4.54; N, 13.27; (Calcd): (C, 62.49; H, 4.53; N, 13.25).

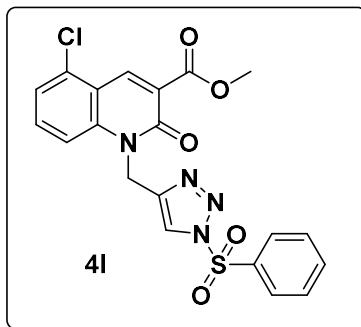




**4-(4-((5-chloro-3-(ethoxycarbonyl)-2-oxoquinolin-1(2H)-yl)methyl)-1H-1,2,3-triazol-1-yl)benzoic acid (4k):** MF:  $C_{22}H_{17}ClN_4O_5$ ; Colourless solid, yield: 90%; m.p. 179-181°C;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$ (ppm): 9.45 (s, 1H), 8.34 (s, 1H), 8.08 (s, 1H), 7.91 (d,  $J = 7.5$  Hz, 2H), 7.76 (d,  $J = 7.5$  Hz, 2H), 7.64 – 7.23 (m, 3H), 5.54 (s, 1H), 5.15 (s, 1H), 4.21 (s, 2H), 1.41 (s, 3H);  $^{13}C$ NMR(100 MHz,  $CDCl_3$ )  $\delta$ (ppm): 168.95, 164.32, 163.61, 140.99, 139.85, 138.14, 132.12, 128.31, 127.07, 124.09, 122.79, 122.60, 121.92, 121.29, 119.82, 115.43, 61.50, 37.34, 14.69; ESI  $[M+H]^+$ : 452.09; Elemental Analysis (%): Found: C, 58.47; H, 3.79; N, 12.39; (Calcd): (C, 58.35; H, 3.78; N, 12.37).



**Methyl-5-chloro-2-oxo-1-((1-(phenylsulfonyl)-1H-1,2,3-triazol-4-yl)methyl)-1,2-dihydroquinoline-3-carboxylate (4l):** MF:  $C_{20}H_{15}ClN_4O_5S$ ; Colourless solid, yield: 76%; m.p. 119-121°C;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$ (ppm): 8.33 (d,  $J = 28.4$  Hz, 2H), 7.97 (t,  $J = 12.2$  Hz, 2H), 7.42 (ddd,  $J = 70.3, 42.4, 9.4$  Hz, 6H), 5.33 (s, 1H), 5.14 (s, 1H), 3.87 (s, 3H);  $^{13}C$ NMR(100 MHz,  $CDCl_3$ )  $\delta$ (ppm): 165.18, 163.61, 156.76, 148.92, 138.12, 135.90, 132.12, 129.72, 129.33, 128.31, 126.86, 124.09, 122.70, 119.82, 115.43, 52.02, 37.34; ESI  $[M+H]^+$ : 458.05; Elemental Analysis (%): Found: C, 52.47; H, 3.31; N, 12.23; (Calcd): (C, 52.35; H, 3.30; N, 12.21).



#### 4. CONCLUSION

In summary, a novel, cost-effective and practical method was developed to synthesize the series of 1,4- Disubstituted 1,2,3-triazole fused dihydroquinolines. The advantages of this method include a simple reaction set-up not requiring specialized equipment's, low-toxicity of the reagent, moderate reaction times, and high product yields with excellent purity.

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