A simple synthesis of 1,3-di-aryl-quinolone derivatives by palladium catalyzed cross-coupling reaction

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An efficient and novel method for the preparation of N-aryl-quinolone via enaminone synthesis has been described. Subsequent derivatization at 3 position has been accomplished by the palladium catalyzed Suzuki cross coupling reaction. Using this method, various aryl boronic acids as well as hetero aryl boronic acids have been coupled with 3-bromo-quinolone to generate the novel 1,3-di-aryl-quinolone derivatives. The key step quinolone ring formation has been achieved by intramolecular displacement reaction of enaminone with suitably substituted ortho-fluoro group in the aromatic ring.

**Keywords**: Enaminone, Suzuki coupling, natural products, quinolone, displacement reaction

Quinolin-4(1\textsubscript{H})-ones constitute a major class of nitrogen containing heterocycles\textsuperscript{1,2}. It is among the most common frameworks present in the bioactive molecules which play an increasingly important role in drug discovery. While 4-quinolone-3-carboxylic acids are one of the largest classes of antimicrobial agents (Figure 1) used worldwide, variously substituted quinolin-4(1H)-ones (4-quinolone) derivatives have shown a wide range of different pharmacological activities\textsuperscript{3} such as anti-inflammatory\textsuperscript{4}, anti-tumor\textsuperscript{5}, anxiolytic\textsuperscript{6}, antiischemic\textsuperscript{7}, antiviral activity\textsuperscript{8}, etc. These compounds comprise the structural units found in a vast range of natural products\textsuperscript{9} and synthetic molecules\textsuperscript{10}. Due to their synthetic accessibility and possible functionalization at different positions of the molecule, quinolin-4(1H)-ones represent an attractive platform for the design of small molecule libraries\textsuperscript{11}.

Most routes to access 4-quinolones rely on traditional reactions such as the Gould-Jacobs\textsuperscript{12}, Conrad-Limpach\textsuperscript{13}, Niementowski\textsuperscript{14}, or Camps cyclizations\textsuperscript{15}. However, these transformations are limited by elevated reaction temperatures, unsatisfactory yields, and poor regioselectivities. Furthermore, several mild synthetic approaches focusing primarily on 2-substituted 4-quinolones have been developed utilizing transition metal catalysis\textsuperscript{16} as well as base-promoted Camps cyclization of N-(ketoaryl)amides\textsuperscript{17}. Among the entire repertoire of 4-quinolone syntheses\textsuperscript{18}, the Conrad-Limpach cyclization is the most prevalent reaction for the preparation of 3-substituted 4-quinolones involving 2-substituted-β-ketoesters and anilines as starting materials. Nevertheless, the cyclization step using sterically hindered and/or acid-sensitive 2-substituted β-ketoesters commonly generates 3-substituted-4-quinolones in poor yields and requires difficult purification protocols.

**Result and Discussion**

In our continued endeavour to prepare novel hybrid molecules containing variety of natural products\textsuperscript{19}, we developed interest in the synthesis of novel 1,3-diphenylquinolone (1-aryl-quinolone) and herein we report our initial results. There are few reports for the synthesis of 1-substituted quinolone using 2-nitroacetophenone\textsuperscript{20} and 2-fluoro-acetophenone as starting materials\textsuperscript{21}, but the method has not been generalized for the synthesis of 1-aryl-quinolone without any substitution at 3-position. Awasaguchi \textit{et al.} reported the synthesis of 1-aryl-3-fluoro-4-oxoquinoline by fluoro cyclization of N-arylenaminone with Select
Fluor and potassium carbonate in DMF as solvent\textsuperscript{22}. Also the Pd-catalyzed decarboxylative coupling of 1-aryl-3-carboxylic acid quinolone with aryl halides has been reported for the synthesis of 1,3-diaryl-quinolone\textsuperscript{23}. These methods use expensive palladium catalysis and reagents. Thus, there is a need to prepare 1,3-diaryl-quinolone using a simple methodology. Retrosynthetic analysis shows that the key step quinolone ring formation (Scheme I) can be achieved by intramolecular reaction of enaminone with suitably substituted 2-fluoro group in the aromatic ring. The o-fluoro-enaminone derivatives 2 can be prepared in a two step synthetic protocol from 2-fluoro-acetophenone via enaminone. It was envisioned by us that the Suzuki coupling reaction could be used to synthesize 1,3-diaryl quinolone derivatives by using various boronic acids at the final step. The required bromo-derivative could be prepared by suitable placement of bromo group in the quinolone ring.

The synthesis starts with the commercially available 2-fluoro-acetophenone 3 which was converted to enaminone 4 by heating the 2-fluoro-acetophenone 3 with dimethylformamide-dimethylacetal (DMF-DMA) in the presence of catalytic amount of p-TSA in toluene at 100°C (Scheme II). The above condensation reaction using DMF as solvent gave similar yield of the compound 4. The reaction went to completion in 16 h as shown by TLC. The solvent was evaporated and the reaction was quenched with water to give the enaminone 4. At this juncture we thought of displacing the labile diamino group with aniline to get the N-aryl-enamino 2 which can be cyclized taking the advantage of 2-F-position in the aromatic ring. Thus, the enaminone 4 was heated with aniline in toluene to give N-aryl-enamino 2 in good yield. The compound 2 was well characterized by \textsuperscript{1}HNMR and MS data. No other by product formation could be detected in the reaction as determined by LC-MS analysis. \textsuperscript{1}HNMR showed peaks at δ 6.17 and 6.96 which correspond to quinolone C-2 and C-3 protons and the absence of NH proton validates the 4-quinolone formation. The IR spectrum of compound 6 showed the carbonyl peak at 1631 cm\textsuperscript{-1}. MS analysis showed M+1 peak at \textit{m/z} 222.34 which confirmed the formation of compound 6.

After obtaining the key intermediate 6, it was desired to explore the bromination reaction of N-aryl-
enaminone 6 to quickly derivatize it. The bromination reaction of compound 6 was tried using NBS in acetonitrile to give the 3-bromo-quinolone derivative 7 in good yield. To confirm the position of bromine in the quinolone ring, $^1$HNMR studies were conducted. The presence of characteristic proton at $\delta$ 6.8 and disappearance of peak at $\delta$ 6.9 in $^1$HNMR indicated the formation of the compound 7. The structure of the compound 7 was further confirmed by HSQC (Heteronuclear Single Quantum Coherence spectroscopy) and HMBC (Heteronuclear Multiple-Bond Correlation) studies (Figure 2). H-8 gave HMBC correlation with C-4 and C-11. Also, in HSQC experiment, CH-8 signal appeared at $\delta$ 143.8 which confirmed that Br is attached to C-9 of quinolone.

After synthesizing the key building block 7, it was subjected to the Suzuki reaction with phenyl boronic acid using PdCl$_2$(dppf)/Na$_2$CO$_3$/DME/EtOH/H$_2$O condition to give compound 1a in good yield. The Suzuki reaction was tried using microwave condition [PdCl$_2$(dppf)/Na$_2$CO$_3$/DME/EtOH/H$_2$O]. The reaction went to completion in 20 min and it was possible to isolate the cross-coupling product in good yield. Once the Suzuki reaction condition was standardized, various boronic acids were subjected to cross-coupling reaction to give the compounds 1a-j (Table I). All compounds 1a-j were well characterized by $^1$H and $^{13}$C NMR and HRMS data (for unknown compounds). As indicated in Table I, both the electron withdrawing (entry 2) as well as electron donating groups (entry 7) on aryl boronic acid gave similar yield of the cross-coupling product. The heteroaryl (entry 4 and 6) boronic acids also gave good yield of the coupling products.

**Materials and Methods**

Dry solvents were purchased from chemical suppliers and used without further purification. All melting points were taken in open capillaries and are uncorrected. Analytical thin-layer chromatography (TLC) was performed on commercially available Merck TLC Silica gel 60 F$_{254}$. Silica gel column chromatography was performed on silica gel 60...
FTIR spectra were recorded on Perkin-Elmer FT/IR-4000 spectrophotometer and only the characteristic peaks are reported. Mass spectra were scanned on a Shimadzu LCMS 2010 spectrometer. $^1$H NMR spectra were recorded on Varian 400 (400 MHz) spectrometer. Chemical shifts of $^1$H NMR spectra were reported relative to tetramethylsilane. $^{13}$C NMR spectra were recorded on Varian 400 (100 MHz) spectrometer. Chemical shifts of $^{13}$C NMR spectra were reported relative to CDCl$_3$ (77.0). Splitting patterns were reported as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, double doublet; br, broad.

### Table I — List of 1,3-Diaryl Quinolone derivatives

<table>
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<tr>
<th>Entry</th>
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<th>Yield (%)</th>
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</table>

To a stirred solution of compound 3 (1 g, 7.246 mmol) in toluene (5 mL), p-TSA.H$_2$O (0.13 g, 0.724 mmol) and DMF-DMA (5 mL) was added. The reaction mixture was heated at 120°C for 16 h. TLC analysis (50% ethyl acetate/ pet ether) showed completion of reaction. The solvent was evaporated and the reaction mixture was quenched with ice and extracted with ethyl acetate. The organic phase was washed with water, brine and dried over anhydrous Na$_2$SO$_4$. The solvent was evaporated and the crude...
product was charged on silica gel column. Elution of the column with 30% ethyl acetate/pet ether gave the compound 4 (1.2 g, 85%) as thick yellow liquid.

$^1$H NMR (400 MHz, DMSO-$d_6$): δ 7.43-7.57 (m, 3H), 7.19-7.25 (m, 2H), 5.43 (d, $J = 12.0$ Hz, 1H), 3.11 (bs, 3H), 2.84 (bs, 3H); LC-MS: $m/z$ 194 (99.6%, M+H$^+$).

**Experimental procedure for the preparation of 1-(2-fluorophenyl)-3-(phenylamino)prop-2-en-1-one, 2**

To a stirred solution of compound 4 (10 g, 51.81 mmol) in toluene (100 mL), aniline (5.30 g, 56.99 mmol) was added. The reaction mixture was heated at 120°C for 16 h. TLC analysis (50% ethyl acetate/pet ether) showed completion of reaction. The solvent was evaporated and the residue charged on silica gel column. Elution of the column with 30% ethyl acetate/pet ether gave the compound 2 (10 g, 80%) as yellow crystals. m.p.111-115°C. IR (KBr): 3234, 3064, 2363, 1660, 1603, 1470, 1299, 1275, 984, 884, 742 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): δ 12.12 (bs, 1H), 7.86 (tt, $J = 12.0$ Hz, 1H), 3.11 (bs, 3H); LC-MS: $m/z$ 242.10 (99.5%, M+H$^+$).

**Experimental procedure for the preparation of 1-phenylquinolin-4(1H)-one, 6**

To a stirred solution of compound 2 (6 g, 24.89 mmol) in DMF (40 mL), 60% NaH (1.49 g, 37.34 mmol) was added at 0°C and stirred at RT for 2 h. The reaction mixture was quenched with cold water and extracted with ethyl acetate. The organic phase was washed with water, brine and dried over anhydrous Na$_2$SO$_4$. The solvent was evaporated and the crude product was charged on silica gel column. Elution of the column with 30% ethyl acetate/pet ether gave the compound 6 (2.5 g, 11.31 mmol) as white solid. m.p.122-126°C. IR (KBr): 3037, 1631, 1583, 1485, 1318, 1315, 1285, 1277, 1269, 1256, 1247, 1237, 1173, 1127, 1119, 1026, 750, 691 cm$^{-1}$; $^1$H NMR (400 MHz, DMSO-$d_6$): δ 8.58 (s, 1H), 8.26 (dd, $J = 1.6$ Hz, 1H), 7.69-7.60 (m, 6H), 7.47 (t, $J = 10.0$ Hz, 1H), 6.97 (d, $J = 11.2$ Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 171.1 (C-4), 143.8 (C-2), 140.4 (C-1), 132.2 (C-7), 130.1 (C-10), 129.6 (C-3'), 125.8 (C-2'), 124.4 (C-5), 117.6 (C-4'), 104.6 (C-3); LC-MS: $m/z$ 298.99 (99.2%, M+H$^+$).

**General procedure for the preparation of compounds, 1a-j**

A microwave vial was charged with 7 (0.50 mmol), the appropriate arylboronic acid (1.00 mmol), PdCl$_2$(dpf) (0.10 mmol), 2M Na$_2$CO$_3$ (1.50 mmol), DME (5 mL), EtOH (1 mL) and exposed to microwave irradiation at 110°C for 20 min. After cooling, the reaction mixture was diluted with dichloromethane and filtered through celine. The organic layer was washed with brine, dried and concentrated to a residue, which was purified by flash chromatography [silica gel, ethyl acetate/pet ether] to provide the title compounds 1a-j.

1,3-Diphenylquinolin-4(1H)-one, 1a: White solid. m.p.203-207°C. IR (KBr): 3043, 1613, 1579, 1488, 1318, 1251, 1027, 916, 757, 694 cm$^{-1}$; $^1$H NMR (400 MHz, DMSO-$d_6$): δ 8.35 (dd, $J = 5.6$ Hz, 1H, Ar-H), 8.14 (s, 1H, H-2), 7.76 (dd, $J = 1.6$ Hz, 2H, Ar-H), 7.62-7.75 (m, 7H, Ar-H), 7.37-7.45 (m, 3H, Ar-H), 7.31 (t, $J = 4.8$ Hz, 1H, Ar-H), 7.01 (d, $J = 6.4$ Hz, 1H, Ar-H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 174.4 (C-1), 142.2 (C-2), 140.8 (C-9), 140.2 (C-1'), 135.2 (C-2), 131.9 (C-7), 130.1 (C-10), 129.3 (C-3', 5'), 127.6 (C-8), 125.9 (C-2', 6'), 125.5 (C-5'), 123.6 (C-6), 117.3 (C-4'), 109.2 (C-3); LC-MS: $m/z$ 298.99 (99.2%, M+H$^+$).

3-(4-Methoxyphenyl)-1-phenylquinolin-4(1H)-one, 1b: White solid. m.p.181-185°C. IR (KBr): 3051, 1623, 1593, 1487, 1282, 1119, 1026, 750, 691 cm$^{-1}$; $^1$H NMR (400 MHz, DMSO-$d_6$): δ 8.35 (d, $J = 7.6$ Hz, 1H, Ar-H), 7.97 (d, $J = 8.0$ Hz, 1H), 7.68-7.75 (m, 6H), 7.39 (t, $J = 7.2$ Hz, 1H), 6.96 (d, $J = 8.4$ Hz, 1H), 6.17 (d, $J = 7.6$ Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 171.1 (C-4), 143.8 (C-2), 141.0 (C-1'), 140.8 (C-9), 132.0 (C-7), 130.2 (C-10), 129.3 (C-3', 5'), 127.6 (C-8), 125.9 (C-2', 6'), 125.5 (C-5'), 123.6 (C-6), 117.3 (C-4'), 109.2 (C-3); LC-MS: $m/z$ 298.99 (99.2%, M+H$^+$).
7.58-7.72 (m, 8H, Ar-H), 7.42 (t, J = 1.6 Hz, 1H, Ar-H), 6.93-7.02 (m, 3H, Ar-H), 3.80 (s, 3H, OCH₃); ¹³C NMR (100 MHz, DMSO-d₆): δ 174.5 (C-4), 158.2 (C-4'), 141.5 (C-1), 140.8 (C-9), 140.2 (C-2), 131.8 (C-7), 130.1 (C-10), 129.6 (C-2''', 6''), 129.3 (C-1''), 127.7 (C-3', 5'), 127.4 (C-8), 126.1 (C-2', 6'), 125.9 (C-5), 123.6 (C-6), 120.0 (C-4), 117.2 (C-3), 113.3 (C-3', 5''), 55.0 (-OCH₃); ESI-HRMS: m/z Calcd for C₂₃H₂₃NO₂ [M+H]⁺: 328.1338. Found: 328.1334.

3-(3-Isopropoxy-5-(trifluoromethoxy)phenyl)-1-phenylquinolin-4(1H)-one, 1c: Off White solid. m.p. 108-112°C. IR (KBr): 3057, 2980, 1624, 1587, 1378, 1319 (C=O), 1296 (C-2'', 6''), 129.3 (C-1''), 127.7 (C-3', 5'), 127.4 (C-8), 126.1 (C-2', 6'), 125.9 (C-5), 123.6 (C-6), 120.0 (C-4), 117.2 (C-3), 113.3 (C-3', 5''), 55.0 (-OCH₃); ESI-HRMS: m/z Calcd for C₂₃H₂₃NO₂ [M+H]⁺: 328.1338. Found: 328.1334.

1-Phenyl-3-(thiopen-3-yl)quinolin-4(1H)-one, 1f: Light yellow solid. m.p. 196-200°C. IR (KBr): 3430, 3074, 3040, 1616, 1580, 1478, 1301, 1165, 838, 750 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆); δ 8.49 (s, 1H, H-2), 8.36-8.39 (m, 2H), 7.73 (dd, J = 1.6 Hz, 1H, Ar-H), 7.40-7.76 (m, 8H, Ar-H), 6.98 (d, J = 3.2 Hz, 1H, Ar-H); ¹³C NMR (100 MHz, DMSO-d₆); δ 174.2 (C-4), 141.5 (C-1'), 140.8 (C-2), 139.8 (C-9), 135.2 (C-7), 131.8 (C-3''), 130.1 (C-10), 129.4 (C-3', 5'), 127.8 (C-8), 126.7 (C-2', 6'), 126.1 (C-4'), 125.8 (C-5), 124.7 (C-5''), 123.7 (C-6), 122.1 (C-4'), 117.3 (C-3'), 115.6 (C-3); ESI-HRMS: m/z Calcd for C₁₈H₁₅N₂O₂ [M+H]⁺: 304.0796. Found: 304.0792.

1-(4-Nitrophenyl)-1-phenylquinolin-4(1H)-one, 1g: Yellow solid. m.p. 230-233°C. IR (KBr): 3065, 2965, 1624, 1592, 1501, 1480, 1336, 1253, 1110, 846, 753, 693 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆); δ 8.54 (s, 1H, H-2), 8.39 (dd, J = 1.2 Hz, 1H, Ar-H), 8.25 (d, J = 9.2 Hz, 2H, Ar-H), 8.16 (d, J = 8.8 Hz, 2H, Ar-H), 7.62-7.70 (m, 6H, Ar-H), 7.49 (t, J = 8.0 Hz, 1H, Ar-H), 7.04 (d, J = 8.8 Hz, 1H, Ar-H); ¹³C NMR (100 MHz, DMSO-d₆); δ 174.2 (C-4), 145.6 (C-4''), 143.8 (C-1'), 142.5 (C-2), 140.6 (C-9), 140.3 (C-2), 132.4 (C-7), 130.1 (C-10), 129.5 (C-3', 5'), 129.1 (C-8'), 127.8 (C-2', 6'), 126.2 (C-5'), 124.4 (C-6, 3', 5'), 122.9 (C-4'), 117.5 (C-3); ESI-HRMS: m/z Calcd for C₁₉H₁₄N₂O₃ [M+H]⁺: 343.1083. Found: 343.1079.

4-(4-Oxo-1-phenyl-1,4-dihydroquinolin-3-yl)benzene nitrile, 1h: Off White solid. m.p.176-180°C. IR (KBr): 3057, 2222, 1624, 1596, 1480, 1328, 1250, 908, 841, 751, 694 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆); δ 8.38 (s, 1H, H-2), 8.35 (d, J = 1.6 Hz, 1H, Ar-H), 8.03 (d, J = 11.2 Hz, 2H, Ar-H), 7.83 (d, J = 11.2 Hz, 2H, Ar-H), 7.63-7.67 (m, 6H, Ar-H), 7.47 (t, J = 10.4 Hz, 1H, Ar-H), 7.02 (d, J = 11.2 Hz, 1H, Ar-H); ¹³C NMR (100 MHz, DMSO-d₆); δ 174.2 (C-4), 143.4 (C-1', 1''), 140.6 (C-9), 140.3 (C-2, 7), 7.88 (dt, J = 1.2 Hz, 4.0 Hz, 1H, Ar-H), 7.63-7.69 (m, 6H, Ar-H), 7.54 (t, J = 2.4 Hz, 1H, Ar-H), 7.46 (t, J = 0.8 Hz, 1H, Ar-H), 7.20 (d, J = 6.4 Hz, 1H, Ar-H), 2.61 (s, 3H, -CH₃); ¹³C NMR (100 MHz, DMSO-d₆); δ 197.9 ([C=O]), 174.4 (C-4), 142.6 (C-1), 140.7 (C-9), 140.3 (C-3''), 136.6 (C-2), 135.6 (C-7), 133.3 (C-6''), 132.1 (C-10), 130.1 (C-1''), 129.4 (C-3', 5'), 128.3 (C-8), 128.1 (C-5''), 127.8 (C-4'), 126.4 (C-2', 6'), 126.1 (C-5), 126.0 (C-2''), 123.9 (C-6), 119.3 (C-4'), 117.3 (C-3), 26.8 (-CH₃); ESI-HRMS: m/z Calcd for C₂₇H₂₂N₂O₃ [M+H]⁺: 399.1381. Found: 399.1378.
3. (4-Fluorophenyl)-1-phenylquinolin-4(1H)-one, \( \text{Ii} \): Off White solid. m.p.200-203°C. IR (KB): 3046, 1617, 1577, 1483, 1326, 1221, 1157, 837, 755, 695 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 8.58 (dd, \( J = 1.6 \) Hz, 1H, Ar-H), 7.80 (s, 1H, H-2), 7.38-7.53 (m, 9H, Ar-H), 7.02-7.12 (m, 3H, Ar-H), 7.02 (d, \( J = 8.4 \) Hz, 1H, Ar-H), 6.89 (d, \( J = 8.4 \) Hz, 1H, Ar-H), 4.26 (s, 4H, OCH\(_2\)CH\(_3\)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 175.9 (C-6'), 161.7, 157.9 (C-4'), 150.1 (C-8), 142.9 (C-5'), 141.4 (C-1'), 141.0 (C-9), 140.5 (C-2'), 131.5 (C-7), 130.2 (C-10), 129.4 (C-3', 5'), 128.5 (C-8), 127.5 (C-5'), 127.1 (C-2'), 126.6 (C-6'), 126.6 (C-7'), 126.3 (C-1'), 121.9 (C-2'), 121.5 (C-3'), 117.6 (C-6''), 117.0 (C-3''), 64.4 (-OCH\(_3\)), 64.3 (-OCH\(_3\)). ESI-HRMS: \( m/z \) Caled for C\(_{23}\)H\(_{18}\)N\(_3\)O\(_3\) [M+H]: 356.1287. Found: 356.1292.

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