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Synthesis of Novel -2-(benzo[d][1,3] dioxol-5-yl)-5-fluoro-4-Phenylquinolines as Antibacterial Agents

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Abstract

A solution of 2-(benzo[d][1.3]dioxol-5-yl)-8-bromo-5-fluoro-4-phenylquinoline **(4)** react with boronic acid **(5a-j)** was heated at 100 ^{0}C for 12 hours in presence of Tetrakis(triphenylphosphine) palladium catalyst and K_2CO_3 as base in toluene:ethanol by the Suzuki-Miyaura cross-coupling reaction to give 2-(benzo[d][1,3] dioxol-5-yl)-5-fluoro-4-phenylquinolines derivatives**(6a-j)** in good yields.

Keywords: 2-(benzo[d][1.3]dioxol-5-yl)-8-bromo-5-fluoro-4-phenylquinoline, $SnCl_2.2H_2O$, Tetrakis(triphenylphosphine)palladium, Antibacterial activity.

Introduction

8-Arylquinolines are pharmaceutically important scaffolds, broadly present in many molecules with a wide array of biological activity.[1-5] In addition they have also been designed and synthesized as key structural elements in materials science.[6-9] A series of 4-substituted 8-aryl-2-methylquinolines was designed and synthesized as highly potent antagonists for the human CRF1 receptor. Dwight Macdonald et al have prepared an emetic, efficacious, and competitive PDE4 inhibitor capable of covalently tagging its biological targets upon photoactivation.[10-14] This provides the possibility of identifying the emesis and efficacy targets of PDE4 inhibitors.

Taking into consideration the biological activity of 8- arylquinolines, we started the investigation for the synthesis of their derivatives.[15-20] Over the past four decades, transition-metal-catalyzed coupling reactions have evolved into an indispensable tool in academia and industry.[21] Among the many known reactions of this type, Suzuki-Miyaura couplings are particularly popular because of their significant advantages including reaction efficiency, mild conditions, high functional group tolerance, and the ease of handling and separating byproducts from reaction mixtures. So, we found Suzuki-Miyaura coupling reaction the best option for the introduction of aryl group at C-8 position of quinoline.[22] The most of the starting materials used for the methodology development were ortho-halo nitrobenzene. The idea behind this was to synthesis 8-halo-quinolines followed by subsequent Suzuki-Miyaura reaction for the elaboration of 8-arylquinoline analogs.

Results and Discussion

Synthesis of 2-(benzo[d][1,3] dioxol-5-yl)-5-fluoro-4-phenylquinolines(6a-j)

The starting material for the synthesisi of target molecules 8-bromo-4-phenyl quinoline, this can be prepared by 2-bromo-5-flurobenzenamine **(1)**, phenylacetylene **(2)** and dioxole-5-carbaldehyde **(3)** in reduction of nitro group with $SnCl_2.2H_2O$ or decomposition of azido group resulted into the formation of amino group which reacts with aldehyde insitu for the formation of imine bond, there is a cycloaddition [4+2] reaction between adduct and two terminal carbons of alkyne to form dihydroquinoline, which undergoes intramolecular oxidation to obtain corresponding quinoline **(4)**. Further the solution of 2-(benzo[d][l,3]dioxol-5-yl)-8-bromo-5-fluoro-4-phenylquinoline**(4)** and 4-methoxy boronic acid **(5a)** was heated at 100 ^{o}C for 12 hrs in presence of Tetrakis (triphenyl phosphine)palladium catalyst and K_2CO_3 as base in toluene : ethanol in the ratio of 3:1 to obtain 2-(benzo[d][1,3] dioxol-5-yl)-5-fluoro-4-phenylquinolines derivatives**(6a-j).** The formation of these compounds was

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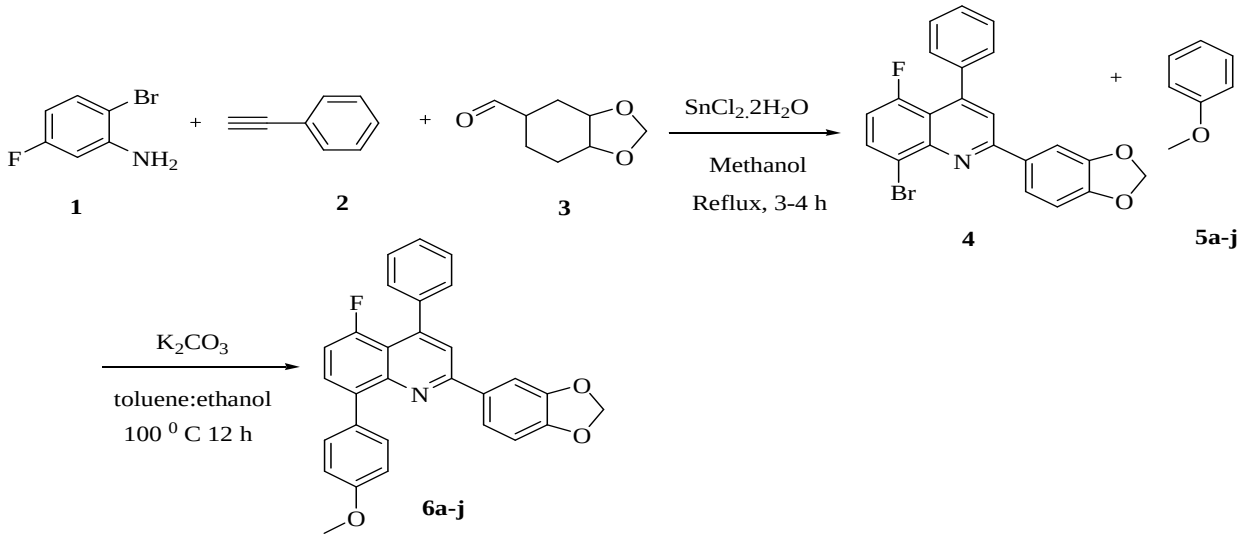
confirmed by their spectral and elemental analysis. In the ^{1}H NMR spectrum of compound **6a** δ 3.92 singlet OCH_3, δ 6.01 singlet (2H, O-CH_2-O). Tha mass spectrum of compound **6a** *m/z* 450 $(M+H)^+$.

Scheme-1



a = anisole **b** = 1,2-difluorobenzene **c** = 1-fluoro-2-(trifluoromethyl)benzene **d** = quinoline
e = 1-chlorobenzene **f** = 1-cyclohexylbenzene **g** = benzonitrile **h** = 1,2,3-trimethoxybenzene
i = benzaldehyde **j** = benzo[*d*][1,3]dioxole

Antibacterial Activity

All the newly prepared compounds **(6a-j)** were screened for the antibacterial activity is done by the paper disc method. Organisms used: Escherichia coli (Gram-positive bacteria) Pseudomonas putida (Gram-negative bacteria)

After solidification of media, petriplates inoculated with actively growing culture of Escherichia coli and Bacillus rumulis separately as follows. Filter paper discs of 5 mm diameter were dipped in the test solution of different concentrations. After drying the disc, it was kept on Antibiotic med-3 agar in petriplates seeded with 1 ml bacterial culture of Escherichia coli and Bacillus rumulis and incubated for 24 hrs at 37° C.

After 24 hours the petridishes were checked for growth inhibition zone. The presence of clear zone of growth inhibition around the paper disc indicated the inhibition of growth of organism. The compound was considered to be active (+). If no clear zone or inhibition around the disc was observed in the petridish, it indicated inactiveness of the sample (-). If partial zone of inhibition was observed, it indicated the partial inhibition of growth (±). The antibacterial activity of the compounds tested is given in Table 1.

Table-1: *Antibacterial activity*

Comp.	*Bacillus rumulis* (cone, µg/ml)						*Escherichia coli* (cone. µg/ml)					
	200	100	50	25	12.5	6.25	200	100	50	25	12.5	6.25
6**a**	-	±	±	±	-	±	-	±	±	-	±	-
6b	-	-	-	-	-	-	+	+	+	+	+	±
6c	-	-	±	-	-	-	±	±	±	±	-	-
6d	-	-	-	-	-	-	±	±	±	-	±	-
6e	±	-	±	-	±	±	±	±	±	-	-	-
6f	-	-	±	±	-	-	±	±	±	±	±	-
6g	±	±	±	±	±	±	±	±	±	±	±	±
6h	-	±	±	±	-	±	-	-	-	-	-	-
6i	±	±	±	±	±	±	-	-	-	-	-	-
6j	±	±	±	±	±	±	±	±	±	-	±	±

'+' indicates high activity '±' indicates less activity '-' indicates no activity

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Experimental Section

Chemistry: Melting points were determined on a Buchi-510 instrument. and ^{1}H NMR (400 MHz) and ^{13}C NMR (100.6 MHz) were recorded on spectrometer TMS as internal standard (chemical shifts and ppm). Mass spectra were recorded on a VG micromass70-70H instrument

General procedure for the synthesis of 2-Benzo[d][l,3]dioxol-5-yl)-8-bromo-5-fluoro-4-phenylquinoline (4)

$SnCl_2.2H_2O$ (1 g, 19.86 mmol, 4 equiv.) was added to a stirred solution of 2-bromo-5-fluorobenzenamine (1 g, 4.955 mmol, 1 equiv.), phenylacetylene (1 g, 4.95 mmol, 1 equiv.) and hexahydrobenzo(d)[1,3]dioxole-5-carbaldehyde
(0.686 g, 4.97 mmol, 1 equiv.) in methanol and heated at reflux temperature for 3-4 hours. The completion of the reaction was monitored by TLC. The solvent was evaporated under reduced pressure and the residue was dissolved in ethyl acetate (20 mL) and then treated with saturated solution of $NaHCO_3$. The mixture was stirred for a period of 2 h and the suspension was filtered through a short bed of celite. The organic phase was separated from aqueous layer. The combined organic phases were evaporated under reduced pressure to afford the residue. Purification of the resulting residue by column chromatography (EtOAc) resulted in colorless compound 2-(benzo[d][1.3]dioxol-5-yl)-8-bromo5-fluoro-4-phenylquinoline **(4),** m.p. 175-176 °C. ^{1}H NMR: δ 6.03 (s, 2H, OCH_2), 6.80-7.07 (m, 2H, Ar-H), 7.38-7.56 (m, 5H, Ar-H), 7.72 (s, 1H, Ar-H), 7.74-7.79 (dd, 1H, J_1 = 8.03 Hz & J2 = 8.03 Hz, Ar-H), 7.88-8.04 (m, 2H, Ar-H). ^{13}C NMR : δ 101.458, 108.00, 108.403, 111.786, 117.298, 120.375, 120.432, 121.257, 122.130, 127.781, 128.049, 128.166, 128.356, 128.408, 132.270, 132.395, 132.475, 132.583, 140.157, 140.203, 146.166, 147.318, 147.345, 148.500, 149.554, 156.218, 156.480, 159.657. ESI-MS: m/z 422 $(M+H)^+$.

General procedure for the synthesis of 2-(benzo[d][l,3]dioxol-5-yl)-5-fluoro-8-(4-methoxyphenyl)-4-phenylquinoline (6a-j)

To a mixture of 2-(benzo[d][l,3]dioxol-5-yl)-8-bromo-5-fluoro-4-phenylquinoline**(4)** (1 gm, 2.375 mmol, 1 equiv.) and 4-methoxyphenylboronic acid**(5a)** (0.433 gm, 2.848 mmol, 1.2 equiv.), catalyst Tetrakis(triphenylphosphine)palladium [10 mol %], K_2CO_3 (0.655 gm, 4.75 mmol, 2 equiv.) was added in toluene : ethanol (3:1) and heated at 100 °C for 12 hours. After the completion of reaction (monitered by TLC), the solvent was evaporated under reduced pressure. The resulting reaction mixture was extracted with ethyl acetate (3x10 mL) and washed with water. The combined ethyl acetate layer was again evaporated in vacuo to give crude product. The purification of the compound was carried out with column chromatograaphy using ethyl acetate : hexane (10 %) to afford pure solid colorless compound **6a**, m.p. 178-179 °C; ^{1}H NMR: δ 3.92 (s, 3H, OCH_3), 6.01(s, 2H, O-CH_2-O), 6.79-6.95 (d, 1H, *J* = 8.30 Hz, Ar-H), 7.10-7.19 (m, 3H, Ar-H), 7.44-7.52 (m, 5H, Ar-H), 7.60-7.79 (m, 6H, Ar-H). ^{13}C NMR:δ 55.32, 101.30, 107.80, 108.31, 110.90, 111.07, 113.19, 116.21, 116.29, 120.16, 121.75, 127.71, 127.78, 127.86, 128.43, 129.53, 131.97, 132.11, 133.28, 136.92, 141.15, 146.78, 147.00, 148.33, 149.06, 154.97, 156.45, 158.51, 158.91. ESI-MS: m/z 450 $(M+H)^+$.

The same procedure was followed for the compounds **(6b-j)**

2-(benzo[d][l,3]dioxol-5-yl)-8-(3,4-difluorophenyI)-5-fluoro-4-phenylquinoline (6b)
M.p. 188-189 °C ^{1}H NMR: δ 6.02 (s, 2H, O-CH_2-O), 6.82-6.93 (d, 1H, *J*= 8.30 Hz, Ar-H), 7.09-7.20 (dd, 1H, *J*= 8.30 Hz & 3.02 Hz, Ar-H), 7.27-7.37 (m, 1H, Ar-H), 7.42-7.54 (m, 6H, Ar-H), 7.57-7.70 (m, 4H, Ar-H), 7.73 (s, 1.H, Ar-H). ^{13}C NMR: δ 101.41, 107.60, 108.39, 110.77, 111.06, 116.26, 116.49, 119.90,120.13, 120.47, 121.80, 126.85,127.76,127.92,128.17, 128.41, 129.87,129.99, 132.86, 135.02,135.07, 136.33, 136.38, 140.81, 146.76, 146.98,148.00,148.16, 148.40,149.26, 151.27, 151.43, 155.36, 156.35, 159.80. ESI-MS: m/z 456 (M+H

2-(benzo[d][l,3]dioxol-5-yl)-5-fluoro-8-(2-fluoro-3-(trifluoromethyl)phenyl)-4-phenylquinoline (6c)
M.p. 173-174 °C ^{1}H NMR 6.00 (s, 2H, O-CH_2-O), 6.78-6.89 (d, 1H, *J*= 8.68 Hz, Ar-H), 7.13-7.23 (m, 1H, Ar-H), 7.33-7.41 (m, 1H, Ar-H), 7.43-7.53 (m, 5H, Ar-H), 7.54-7.63 (m, 2H, Ar-H), 7.66-7.78 (m, 4H, Ar-H). ^{13}C NMR: δ 101.38, 101.74, 108.29, 110.73, 111.02, 120.88, 121.92, 123.38, 123.43, 126.28, 126.34, 127.78, 127.96, 128.44, 128.48, 130.57, 130.69, 132.90, 136.43, 136.47, 140.70, 146.89, 147.12, 148.39, 149.31, 155.78, 157.08, 160.54. ESI-MS: m/z 506 $(M+H)^+$.

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2′-(benzo (d][l,3]dioxol-5-yl)-5'-fluoro-4'-phenyl-3,8'-biquinoline (6d)
M.p. 185-189 °C ^{1}H NMR: δ 5.97 (s, 2H, O-CH_2-O), 6.74-6.90 (d, 1H, *J*= 8.68 Hz, Ar-H), 7.16-7.29 (m, 1H, Ar-H), 7.44-7.56 (m, 5H, Ar-H), 7.57-7.71 (m, 3H, Ar-H), 7.72-7.85 (m, 3H, Ar-H), 7.89-7.97 (d, 1H, *J*= 7.93 Hz, Ar-H), 8.15-8.29 (d, 1H, *J*= 8.30 Hz, Ar-H), 8.47 (s, 1H, Ar-H), 9.28-9.48 (d, 1H, *J* = 1.88 Hz, Ar-H). ^{13}C NMR: δ 101.34, 107.60, 108.45, 111.05, 111.34, 116.29, 116.42, 120.74, 121.96, 126.61, 127.787, 127.911, 127.955, 128.392, 128.434, 129.16,129.28, 130.41, 130.53, 132.688, 132.775, 133.773, 133.829, 136.60, 140.70, 140.74, 146.877, 147.04, 148.34, 149.26, 153.19, 155.756, 156.652, 160.10. ESI-MS: m/z 471 $(M+H)^+$.

2-(benzo[d][l,3]dioxoI-5-yl)-8-(4-chlorophenyl)-5-fluoro-4-phenylquinoline (6e)
M.p. 194-195 °C ^{1}H NMR: δ 6 6.01 (s, 2H, O-CH_2-O), 6.80-6.93 (d, 1H, *J*= 8.68 Hz, Ar-H), 7.06-7.20 (m, 1H, Ar-H), 7.42-7.54 (m, 7H, Ar-H), 7.57-7.76 (m, 6H, Ar-H). ^{13}C NMR: δ 101.36, 107.683, 108.378, 110.88, 111.05, 116.24, 116.31, 120.378, 121.80, 127.75, 127.84, 128.42, 129.86, 129.93, 132.27, 133.02, 133.13, 136.01, 137.94, 140.90, 146.90, 148.36, 149.20, 155.25, 156.92, 158.99. ESI-MS: m/z 454 $(M+H)^+$.

2-(benzo[d][l,3]dioxol-5-yl)-8-(4-cyclohexylphenyl)-5-fluoro-4-phenylquinoline (6f)
M.p. 175-177 °C ^{1}H NMR : δ 1.39 (m, 5H, cyclohexyl), 1.74-1.84 (m, 1H, cyclohexyl), 1.85-1.95 (m, 2H, cyclohexyl), 1.96-2.06 (m, 2H, cyclohexyl), 2.57-2.72 (m, 1H, cyclohexyl), 6.00 (s, 2H, O-CH_2-O), 6.80-6.91 (d, 1H, *J*= 8.01 Hz, Ar-H), 7.10-7.17 (m, 1H, Ar-H) , 7.33-7.40 (m, 2H, Ar-H), 7.43-7.52 (m, 5H, Ar-H), 7.62-7.76 (m, 6H, Ar-H). ^{13}C NMR: δ 26.25, 27.00, 29.67, 34.52, 44.36, 101.289, 107.90, 108.29, 110.84, 111.13, 116.17, 116.30, 120.199, 121.797, 126.175, 127.71, 127.77, 128.43, 128.47, 129.83, 129.95, 130.96, 133.35, 136.87, 137.28, 137.33, 141.20, 146.80, 146.96, 147.06, 148.34, 149.07, 155.05, 155.90, 159.32. ESI-MS: m/z 502 $(M+H)^+$.

4-(2-(benzo[d][l,3]dioxol-5-yl)-5-fluoro-4-phenylquinolin-8-yl)benzonitrile (6g)
M.p. 227-228 °C. ^{1}H NMR: δ 6.02 (s, 2H, O-CH_2-O), 6.81-6.93 (d, 1H, *J*= 8.68 Hz, Ar-H), 7.13-7.22 (m, 1H, Ar-H), 7.42-7.55 (m, 5H, Ar-H), 7.57-7.65 (m, 2H, Ar-H), 7.66-7.72 (m, 1H, Ar-H), 7.74 (s, 1H, Ar-H), 7.78-7.84 (d, 2H, *J*= 7.99 Hz, Ar-H), 7.85-7.90 (d, 2H, *J* = 7.99 Hz, Ar-H). ^{13}C NMR: δ 101.47, 107.62,108.44,110.74, 110.95, 111.13, 116.34, 116.41,119.25, 120.66, 121.86, 127.82, 128.02, 128.41,130.19,130.26, 131.43,131.70,132.80,135.33,135.36, 140.71, 144.42, 146.80, 147.12, 148.48, 149.42, 155.63, 157.52, 159.95. ESI-MS: m/z 445 $(M+H)^+$.

2-(benzo[d][l,3]dioxol-5-yl)-5-fluoro-4-phenyl-8-(3,4,5-trimethoxyphenyl)quinoline (6h)
M.p. 224-225 °C ^{1}H NMR : δ 3.94 (s, 6H, OCH_3), 3.97 (s, 3H, OCH_3), 6.00 (s, 2H, O-CH_2-O), 6.83-6.95 (d, IE, *J*= 7.99 Hz, Ar-H), 7.00-7.21 (m, 3H, Ar-H), 7.39-7.57 (m, 5H, Ar-H), 7.63-7.8 (m, 4H, Ar-H). ^{13}C NMR: δ 56.13, 56.23, 60.96, 101.40, 104. 51, 107.62, 108.31, 108.31, 108.47, 110.78, 111.06, 120.19, 121.69, 127.73, 127.85, 128.36, 128.40, 129.938, 130.06, 133.03, 135.07, 136.95, 137.00, 140.95, 140.99, 146.93, 148.41, 149.21, 152.58, 155.06, 156.02, 159.45. ESI-MS: m/z 510 $(M+H)^+$.

4-(2-(benzo[d][l,3]dioxol-5-yl)-5-fluoro-4-phenylquinolin-8-yl)benzaldehyde (6i)
M.p. 200-201 °C. ^{1}H NMR: δ 6.00 (s, 2H, O-CH_2-O), 6.84-6.92 (d, 1H, *J*= 7.99 Hz, Ar-H), 7.15-7.23 (m, 1H, Ar-H), 7.45-7.54 (m, 5H, Ar-H), 7.60-7.67 (m, 2H, Ar-H). 7.71-.7.77 (m, 2H. Ar-H), 7.91-7.99 (d, 2H, *J*= 7.99 Hz, Ar-H), 7.99-8.08 (d, 2H, *J*= 7.99 Hz, Ar-H), 10.15 (s, 1H, Ar-H). ^{13}C NMR: δ 101.38, 107.64, 108.42, 110.90, Hi.18, 120.55, 121.83, 127.80, 127.97, 128.37, 129.13, 130.24, 130.35, 131.67, 132.86, 134.99, 140.74, 148.39, 149.30, 155.49, 156.69, 192.35. ESI-MS: m/z 448 $(M+H)^+$.

2,8-di(benzo[d][l,3]dioxol-5-yl)-5-fluoro-4-phenylquinoline (6j)
M.p. 130-131 °C ^{1}H NMR: δ 6.02 (s, 2H, O-CH_2-O), 6.06 (s, 2H, O-CH_2-O), 6.84-6.92 (d, 1H, *J*= 8.30 Hz, Ar-H), 6.96-7.02 (d, 1H, *J*= 8.30 Hz, Ar-H), 7.09-7.18 (m, 1H, Ar-H), 7.19-7.25 (dd, 1H, *J* = 8.30 Hz & 1.51 Hz, Ar-H), 7.29-7.35 (d, 1H, *J*= 2.26 Hz, Ar-H), 7.47-7.51 (m, 5H, Ar-H), 7.62-7.69 (m, 2H, Ar-H), 7.70-7.75 (m, 2H, Ar-H). ^{13}C NMR: δ 101.10, 101.33, 107.80, 108.34, 110.80, 111.08, 111.73, 120.28,121.78, 124.44,127.72,127.81,128.40,128.44,129.69,129.81,133.21, 133.42, 136.84, 136.90, 141.03, 141.07, 146.80, 146.98, 147.12, 148.33, 149.10, 155.08, 155.89, 159.32. ESI-MS: m/z 464 $(M+H)^+$.

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