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One Pot Synthesis of 6-Phenyl-pyrazolyl-quinzolinone Derivatives using Palladium Catalyst *via* Suzuki-Miyaura Cross-Coupling Reaction

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In present work, a palladium-mediated Suzuki-Miyaura cross-coupling reaction, which enables the efficient synthesis of 6-phenyl-pyrazolyl-quinzolinone derivatives from 6-iodo-pyrazolyl-quinzolinone, aryl boronic acid with potassium carbonate (K_2CO_3) is described.

Keywords: Suzuki-Miyaura, Palladium, Boronic acid.

INTRODUCTION

In recent years, progress has been made on the development of Suzuki-Miyura cross-coupling reaction, which is one of the most important reactions in organic synthesis to bring about catenation in the molecule. It refers to the cross coupling reaction of aryl boronic acid and organohalide with the palladium catalyst. This reaction has attracted extensive attention due to its mild reaction conditions, good tolerance of functional groups and economical availability of aryl boronic acid and high yield of product [1-3]. Suzuki coupling reaction is an important tool for synthesizing biaryl compounds, which have been widely used as building blocks for many drugs, herbicides, natural products, conducting polymers, agrochemicals, liquid crystalline materials and also used as ligand for catalysis [4,5].

The Suzuki coupling exhibit a wide spectrum of biological applications such as antibacterial [6,7], antifolate [8,9], antiviral [10], anti-inflammatory [11,12], antimicrobial [13,14], anticancer agents [15], antihypertensives [16], antileishmanials [17], anticonvulsants [18], antiaggressives [19,20], tyrosine kinase [21], antitumor [22] and antioxidant activities [23]. Antioxidants play a significant role in several important biological processes such as immunity, protect ion against tissue damage, reproduction and growth or development. They preserve adequate function of cells against homeostatic disturbances such as those caused by septic shock, aging and in general, processes involving oxidative stress [24].

EXPERIMENTAL

General procedure for Suzuki coupling of compound 3a-j: To a 6-iodopyrazolyl-quinzolinone (**1**) (1 equiv.), a mixture of 1,4-dioxane and methanol (3:1) and phenyl boronic acid (**2**) (1.5 equiv.) were added followed by K_2CO_3 and $Pd(PPh_3)_4$ catalyst. The mixture is stirred for 4 h at 70 ºC. After the completion of the reaction (monitored by TLC), the reaction mixture was extracted with ethyl acetate, washed with dil. HCl and distilled water. The combined organic layer was dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and crude was purified by silica gel column chromatography to afford pure Suzuki coupled derivatives **3a-g**.

2-(1,3-Diphenyl-1*H*-pyrazol-4-yl)-6-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (3a): Brown powder; 92% yield; R_f (30% EtOAc-hexane): 0.44, m.p.: 184-186 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2983, 1737, 1664, 1486, 1373, 1240; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.91 (s, 1H), 8.43 (s, 1H), 7.98 (t, *J* = 4.7 Hz, 3H), 7.83 (d, *J* = 6.5 Hz, 2H), 7.69-7.63 (m, 1H), 7.61 (d, *J* = 7.5 Hz, 2H), 7.58-7.51 (m, 2H), 7.51-7.46 (m, 2H), 7.42 (dd, *J* = 7.5, 2.2 Hz, 3H), 7.38-7.32 (m, 1H), 7.32-7.28 (m, 1H), 7.26 (s, 1H), 6.90 (dd, *J* = 8.4, 2.8 Hz, 1H), 5.98 (s, 1H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 150.9, 148.0, 139.8, 139.4, 132.5, 131.6, 129.8, 129.7, 129.5, 129.0, 128.5, 128.4, 128.3, 126.7, 126.6, 125.7, 125.4, 120.4, 118.4, 115.8, 115.4, 60.3. HRMS-ESI $[M]^+$ *m/z*: 442.1798.

2-(1,3-Diphenyl-1*H*-pyrazol-4-yl)-6-(*p*-tolyl)-2,3-dihydroquinazolin-4(1*H*)-one (3b): Yellow powder; 87%

yield; R_f (40% EtOAc-hexane): 0.47, m.p.: 182-184 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2984, 2363, 1737, 1665, 1617, 1497, 1373, 1241, 1104; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.24 (s, 1H), 8.10 (s, 1H), 7.64 (dd, *J* = 12.4, 7.6 Hz, 4H), 7.52-7.45 (m, 1H), 7.41-7.26 (m, 7H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 7.8 Hz, 2H), 6.64 (d, *J* = 8.3 Hz, 1H), 6.47 (s, 1H), 6.06 (s, 1H), 4.55 (s, 1H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.3, 151.4, 146.3, 139.7, 137.2, 136.8, 133.2, 132.6, 132.2, 129.7, 129.6, 129.0, 128.9, 128.3, 128.2, 127.2, 126.8, 126.4, 120.0, 119.3, 116.5, 115.7, 61.1, 21.2. HRMS-ESI $[M]^+$ *m/z*: 456.1999.

2-(1,3-Diphenyl-1*H*-pyrazol-4-yl)-6-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3c): Yellow powder; 90% yield; R_f (40% EtOAc-hexane): 0.55, m.p.: 240-242 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 33329, 3122, 3064, 2921, 2851, 1672, 1613, 1492, 1358, 1240, 1176; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.93 (s, 1H), 8.39 (s, 1H), 8.00-7.96 (m, 2H), 7.90 (d, *J* = 2.3 Hz, 1H), 7.82 (d, *J* = 7.0 Hz, 2H), 7.59 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.53 (d, *J* = 8.7 Hz, 4H), 7.48 (dd, *J* = 8.2, 6.6 Hz, 2H), 7.44-7.39 (m, 1H), 7.38-7.33 (m, 1H), 7.17 (s, 1H), 7.00 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 1H), 5.94 (s, 1H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 158.3, 150.9, 147.5, 139.4, 132.4, 132.3, 131.2, 129.7, 129.6, 129.5, 128.5, 128.4, 128.3, 126.8, 126.6, 124.8, 120.3, 118.3, 115.9, 115.4, 114.4, 60.3, 55.1. HRMS-ESI $[M]^+$ *m/z*: 472.1899.

2-(1,3-Diphenyl-1*H*-pyrazol-4-yl)-6-(4-isocyanophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3d): White powder; 89% yield; R_f (40% EtOAc-hexane): 0.45, m.p.: 242-244 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2984, 1738, 1455, 1374, 1238, 1101; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.94 (s, 1H), 8.46 (s, 1H), 8.05 (d, *J* = 2.3 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 2H), 7.90-7.78 (m, 6H), 7.75 (dd, *J* = 8.5, 2.4 Hz, 1H), 7.54 (dd, *J* = 8.6, 7.4 Hz, 2H), 7.51-7.45 (m, 3H), 7.44-7.39 (m, 1H), 7.38-7.33 (m, 1H), 6.90 (d, *J* = 8.5 Hz, 1H), 6.00 (s, 1H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.7, 150.9, 148.9, 144.2, 139.3, 132.9, 132.4, 131.9 129.7, 129.6, 128.5, 128.4, 128.3, 127.3, 126.6, 126.3, 126.0, 120.3, 118.3, 115.7, 115.5, 108.8, 60.1. HRMS-ESI $[M]^+$ *m/z*: 467.1697.

6-(4-Chlorophenyl)-2-(1,3-diphenyl-1*H*-pyrazol-4-yl)-2,3-dihydroquinazolin-4(1*H*)-one (3e): White powder; 88% yield; R_f (40% EtOAc-hexane): 0.50, m.p.: 216-218 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2936, 1739, 1677, 1373, 1240, 1098; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.93 (s, 1H), 8.43 (s, 1H), 7.98 (dd, *J* = 8.7, 1.2 Hz, 2H), 7.96-7.94 (m, 1H), 7.83-7.80 (m, 2H), 7.64 (d, *J* = 8.7 Hz, 2H), 7.54 (dd, *J* = 8.7, 7.3 Hz, 2H), 7.47 (d, *J* = 8.7 Hz, 4H), 7.43-7.33 (m, 3H), 7.31 (s, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 5.96 (s, 1H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.9, 150.9, 148.2, 139.3, 138.6, 132.4, 131.5, 131.2, 129.7, 129.6, 128.9, 128.5, 128.4, 128.2, 127.4, 126.6, 125.4, 120.3, 118.3, 115.8, 115.4, 60.2. HRMS-ESI $[M]^+$ *m/z*: 476.1398.

2-(3-(4-Methoxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl)-6-(*p*-tolyl)-2,3-dihydroquinazolin-4(1*H*)-one (3f): White powder; 91% yield; R_f (40% EtOAc-hexane): 0.60, m.p.: 224-226 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2984, 1730, 1664, 1498, 1371, 1244; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.89 (s, 1H), 8.39 (s, 1H), 7.99-7.93 (m, 3H), 7.79-7.74 (m, 2H), 7.62 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.56-7.48 (m, 4H), 7.37-7.31 (m, 1H), 7.26-7.20 (m, 3H), 7.07-7.02 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 1H), 5.93 (s, 1H), 3.78 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.2, 159.4, 150.8, 147.9, 139.4, 136.9, 135.7, 131.4, 129.7, 129.6, 129.5, 129.4, 126.5, 125.6, 125.0, 124.9, 119.9, 118.2, 115.9, 115.4, 113.9, 60.4, 55.2, 20.6. HRMS-ESI $[M]^+$ *m/z*: 486.2099.

6-(4-Methoxyphenyl)-2-(3-(4-nitrophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-2,3-dihydroquinazolin-4(1*H*)-one (3g): Yellow powder; 91% yield; R_f (40% EtOAc-hexane): 0.45, m.p.: 210-212 °C. FTIR (KBr, ν_{max}, cm^{-1}): 2984, 1738, 1446, 1373, 1233, 1098; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.98 (s, 1H), 8.41 (s, 1H), 8.35-8.31 (m, 3H), 8.18 (d, *J* = 8.9 Hz, 2H), 7.90 (d, *J* = 2.3 Hz, 1H), 7.59-7.53 (m, 5H), 7.52 (s, 1H), 7.40 (d, *J* = 7.4 Hz, 1H), 7.19 (s, 1H), 7.00 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 1H), 6.08 (s, 1H), 3.79 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 158.3, 148.7, 147.4, 147.0, 139.1, 132.2, 131.3, 130.5, 129.8, 129.4, 127.1, 126.8, 126.7, 124.7, 123.7, 121.1, 118.5, 115.9, 115.5, 114.4, 114.1, 60.2, 55.1. HRMS-ESI $[M]^+$ *m/z*: 517.1798.

2-(3-(4-Bromophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-6-(*p*-tolyl)-2,3-dihydroquinazolin-4(1*H*)-one (3h): White powder; 86% yield; R_f (40% EtOAc-hexane): 0.56, m.p.: 226-228 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2984, 1738, 1446, 1372, 1232, 1098; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.91 (s, 1H), 8.37 (s, 1H), 7.98-7.93 (m, 3H), 7.83-7.80 (m, 2H), 7.67 (dd, *J* = 8.6, 2.5 Hz, 2H), 7.57-7.53 (m, 2H), 7.53-7.47 (m, 3H), 7.43-7.30 (m, 2H), 7.24 (d, *J* = 7.9 Hz, 2H), 6.87 (dd, *J* = 8.4, 2.5 Hz, 1H), 5.97 (d, *J* = 11.6 Hz, 1H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 150.9, 149.7, 148.6, 147.8, 139.2, 136.9, 135.7, 132.4, 131.8, 131.4, 130.4, 129.7, 129.5, 128.5, 128.4, 126.7, 125.7, 125.0, 121.7, 120.5, 120.4, 118.4, 115.8, 115.7, 114.6, 60.2, 20.6. HRMS-ESI $[M]^+$ *m/z*: 534.1099.

2-(3-(4-Chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-6-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3i): White powder; 87% yield; R_f (40% EtOAc-hexane): 0.48, m.p.: 180-182 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2982, 2933, 1739, 1499, 1446, 1373, 1240, 1094; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.93 (s, 1H), 8.39 (d, *J* = 1.4 Hz, 1H), 8.00-7.94 (m, 3H), 7.91 (d, *J* = 2.4 Hz, 1H), 7.89 (s, 1H), 7.88-7.85 (m, 2H), 7.61 (d, *J* = 2.3 Hz, 1H), 7.56-7.53 (m, 5H), 7.19-7.16 (m, 1H), 7.00 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 1H), 5.98 (s, 1H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.2, 158.3, 149.7, 148.6, 147.5, 139.3, 132.3, 131.4, 131.2, 130.1, 129.9, 129.7, 128.5, 126.8, 126.7, 124.8, 120.4, 120.4, 118.4, 115.9, 115.4, 114.4, 60.3, 55.1. HRMS-ESI $[M]^+$ *m/z*: 506.1499.

2-(3-(4-Nitrophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-6-(*p*-tolyl)-2,3-dihydroquinazolin-4(1*H*)-one (3j): Brown powder; 86% yield; R_f (40% EtOAc-hexane): 0.56, m.p.: 228-230 ºC. FTIR (KBr, ν_{max}, cm^{-1}): 2984, 2928, 1738, 1664, 1618, 1556, 1499, 1448, 1373, 1240, 1154, 1107; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.98 (s, 1H), 8.42 (d, *J* = 1.5 Hz, 1H), 8.33 (d, *J* = 8.9 Hz, 2H), 8.17 (d, *J* = 8.9 Hz, 2H), 8.01-7.97 (m, 2H), 7.94 (d, *J* = 2.3 Hz, 1H), 7.63 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.56 (dd, *J* = 8.6, 7.4 Hz, 2H), 7.49 (d, *J* = 8.2 Hz, 2H), 7.42-7.36 (m, 1H), 7.27-7.21 (m, 3H), 6.87 (d, *J* = 8.4 Hz, 1H), 6.09 (s, 1H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 148.6, 147.6, 146.9, 139.0, 136.8, 135.7, 131.3, 130.4, 129.8,

129.7, 129.4, 129.3, 127.0, 126.5, 125.5, 124.9, 123.6, 121.0, 118.5, 115.8, 115.4, 60.1, 20.5. HRMS-ESI $[M]^+$ *m/z*: 501.1799.

RESULTS AND DISCUSSION

For the synthesis of biphenyl from 6-iodopyrazolyl quinazolinone and boronic acid, we initially examined the effect of various catalysts and found that $Pd(PPh_3)_4$ was the catalyst of choice after proper systematic screening (Table-1).

TABLE-1
SCREENING OF CATALYST

Entry	Catalyst (10 mol %)	Time (h)	Yield[a] (%)
1	$NiCl_2(PPh_3)_2$	48	–
2	$Pd(OAc)_2$	24	20
3	$Pd(dppf)Cl_2$	24	25
4	$Pd(PPh_3)_4$	4	92
5	$Pd(dppb)Cl_2$	12	30
6	$Pd(PPh_3)_2Cl_2$	6	65
7	$Pd(dba)_3$	8	50
8	$Pd(dppp)Cl_2$	12	35

[a]Isolated yield.

To make the reaction greener, preliminary screening with various solvents was performed. The best result was obtained when the reaction was carried out with 1,4-dioxane-methanol mixture (3:1) (Table-2).

TABLE-2
SCREENING OF SOLVENT

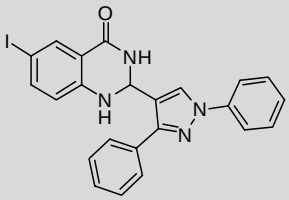
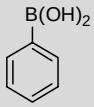
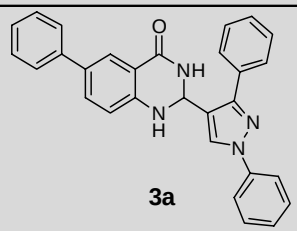
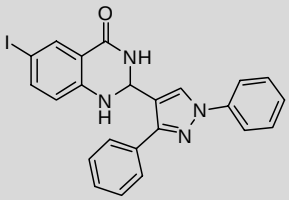
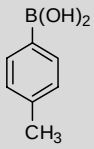
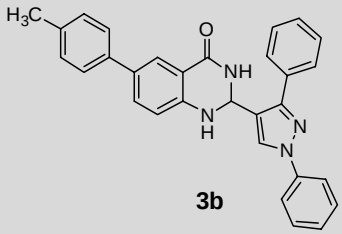
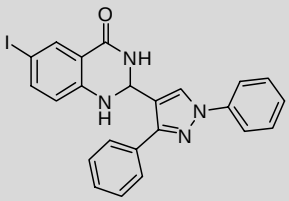
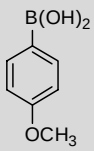
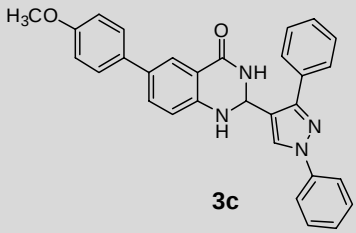
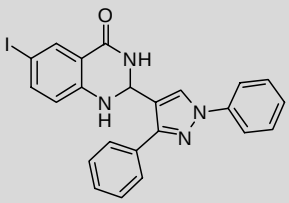
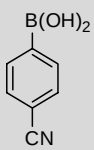
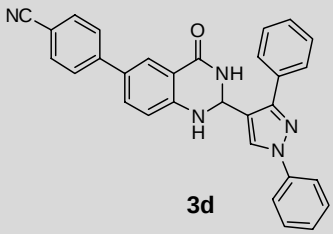
Entry	Solvent	Yield[a] (%)
1	Water	Trace
2	Ethanol	Trace
3	Methanol	30
4	1,4-Dioxane	75
5	1,4-Dioxane-methanol mixture	92

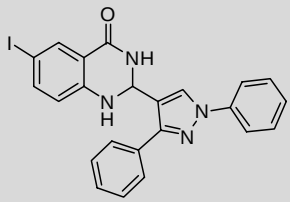
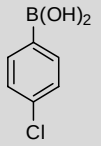
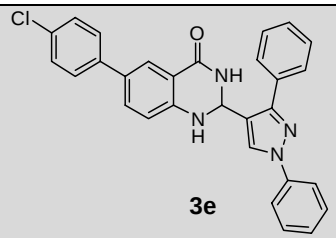
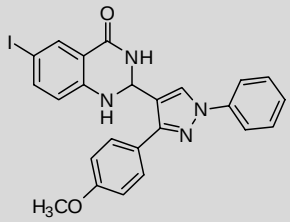
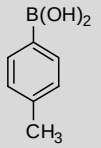
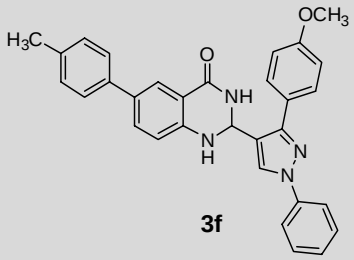
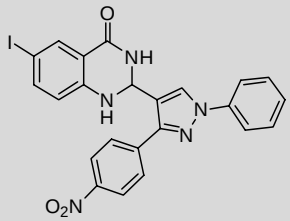
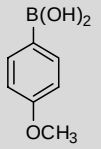
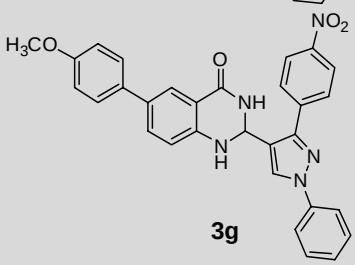
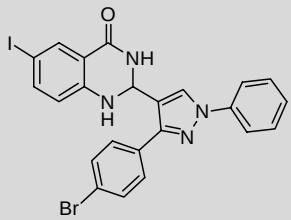
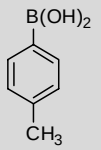
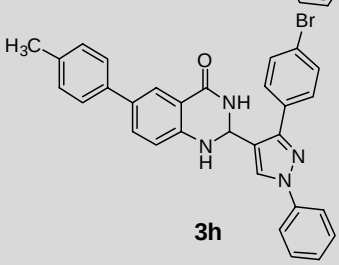
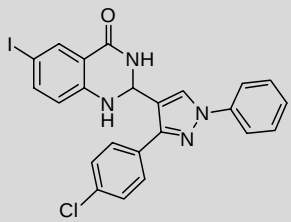
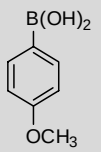
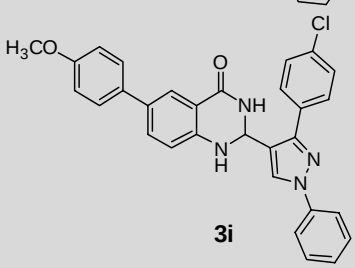
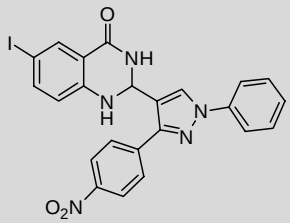
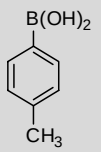
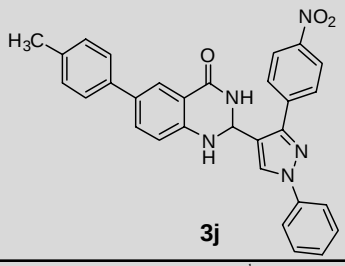
[a]Isolated yield.

The strategy we have developed begins with Suzuki-Miyaura reaction of 6-iodo-pyrazolyl-quinazolinone (**1**), boronic acid (**2**) in mixture of 1,4-dioxane and methanol (3:1) at 70 ºC in the presence of $Pd(PPh_3)_4$ catalyst. The reaction undergoes smoothly within 4 h yielding 92% of products **3** (**Scheme-I**). The solid product was dissolved in ethyl acetate to afford pure product. However, the product was further purified by column chromatography. These products were characterized with various spectroscopic techniques like ^1H & ^{13}C NMR and mass spectrometry (Table-3).

The possible mechanism for the reaction (**Scheme-I**) is discussed below in details (**Scheme-II**). The first step involves formation of organo palladium(II) complex intermediate (**A**) by reaction of 6-iodopyrazolyl-quinzolinone (**1**) with palladium catalyst. During the second step, (**A**) reacts with boronic acid

TABLE-3
SYNTHESIS OF 6-PHENYL-PYRAZOLYL-QUINAZOLINONE

Entry	6-Iodo-pyrazolyl-quinzolinone	Boronic acid (**2a-e**)	Product **3**[a]
1			 **3a**
2			 **3b**
3			 **3c**
4			 **3d**

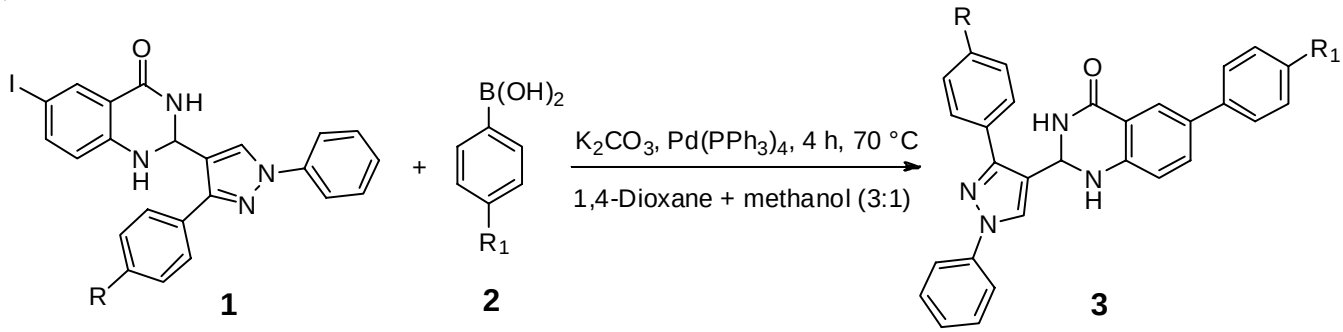
5			 **3e**
6			 **3f**
7			 **3g**
8			 **3h**
9			 **3i**
10			 **3j**

[a]All the reaction carried out by Suzuki coupling reaction and characterized by IR, ^{1}H NMR, ^{13}C NMR and Mass spectroscopy. [b]Isolated yield after column chromatography.

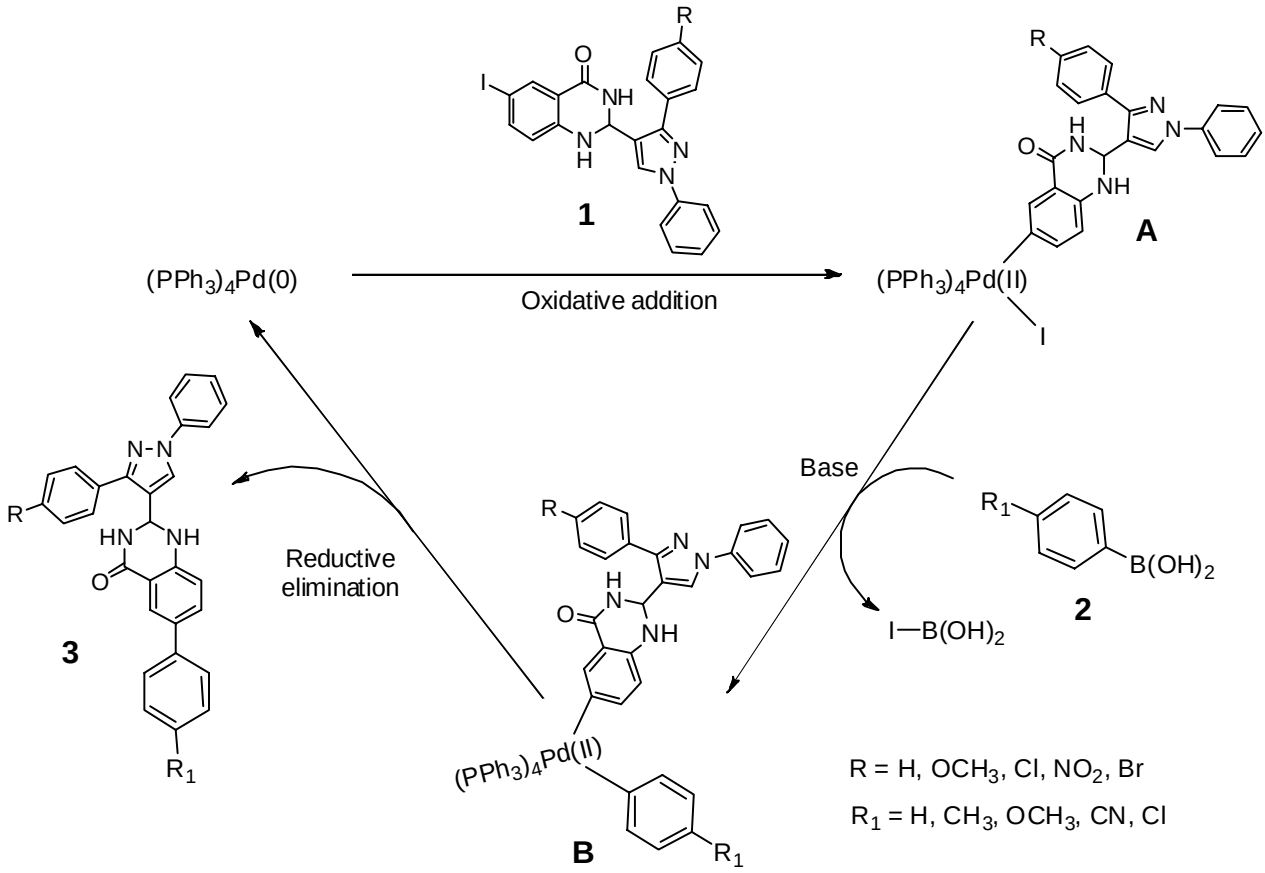
(**2**) in presence of base to give the intermediate (**B**), which further undergoes intramolecular reductive elimination to form desired product **3** and recovered palladium catalyst.

Conclusion

In conclusion, a palladium-mediated Suzuki-Miyaura cross-coupling reaction which enables the efficient synthesis of 6-phenyl-pyrazolyl-quinzolinone derivatives from 6-iodo-pyrazolyl-quinzolinone, aryl boronic acid with K_2CO_3 is developed. The screening studies of solvents and catalyst was carried out. The better result was obtained with 1,4-dioxane and methanol (3:1) mixture and $Pd(PPh_3)_4$ catalyst.



Scheme-I: Synthesis of 6-phenyl-pyrazolyl-quinazolinone



Scheme-II: Plausible mechanism

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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