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SYNTHESIS OF QUINOLYL- AND ISOQUINOLYL HETEROARYLAMINES USING PALLADIUM CATALYZED SUZUKI CROSS-COUPLING REACTION

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Abstract – Quinolyl- and isoquinolylboronic acids are prepared from 5-bromoquinoline and 5-bromoisoquinoline, respectively, *via* halogen-metal exchange. Suzuki cross-coupling of these compounds with haloheteroarylamine leading to the corresponding heteroarylamine derivatives is described.

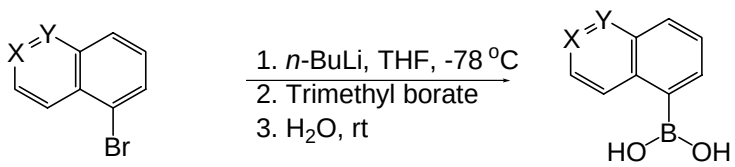
INTRODUCTION

The palladium catalyzed Suzuki cross-coupling reaction of aryl halides and heteroaryl halides with boronic acids to construct biaryls and heterobiaryls is well known as one of the most versatile and utilized reactions in organic synthesis.[1] Synthesis of 6-aryl-2,4-diaminopyrimidines and triazines using palladium catalyzed Suzuki cross-coupling was reported by Cooke *et al.* in 2001.[2] Several groups have described the palladium-catalyzed Suzuki coupling of quinoline halides or isoquinoline halide with phenylboronic acid and quinolylboronic acid with aryl halides.[3] There are a few examples to the synthesis of quinolylboronic acid or quinolylboronic ester in the literature,[4] but study of isoquinolylboronic acid has not been published so far.

RESULTS AND DISCUSSION

In our ongoing project to synthesize biologically active compounds as potential therapeutic agent for inhibition of the sodium hydrogen exchanger (NHE),[5] we needed to introduce heteroaryl with amino group onto 5-quinolylboronic acids or 5-isoquinolylboronic acids. Following this strategy, first of all, bromination of quinoline or isoquinoline gave 5-bromoquinoline (**1a**) or 5-bromoisoquinoline (**1b**) as described in the literature.[6] The subsequent reaction was the one-pot reaction composed of lithiation by halo-metal exchange reaction and addition of trialkyl borate. In our reactions for the preparations of 5-quinolylboronic acid or 5-isoquinolylboronic acids, we found the standard addition of triisopropyl borate gave poor yields. This difficulty was succumbed by the addition of trimethyl borate to the lithiated intermediate resulted from the reaction of *n*-BuLi with bromide (**1a-b**) in THF at -78 ^{o}C. The yields from **1a-b** to **2a-b** were 45% and 56%, respectively.[7]

Scheme 1



1a: X=C, Y=N
b: X=N, Y=C

2a: X=C, Y=N
b: X=N, Y=C

Cross-coupling reactions of **2a-b** with amino heteroaryl halides were investigated under the standard conditions using sodium carbonate as a base and (tetrakistriphenylphosphino)palladium as a catalyst. The choice of solvent was important in this reaction. The experimental results were summarized in Table 1. The coupling reaction seems to be dependent on solvent. At first, DME was our choice of the solvent for Suzuki coupling reaction. In the case of Entries 1, 2, 7, and 8 , Suzuki coupling did not provide any good yields due to poor solubility of 6-halo-2,4-diaminopyrimidine or triazine and quinolylboronic acid or isoquinolylboronic acid in DME. When DME was switched to dioxane, better yields of products were observed.[8]

CONCLUSION

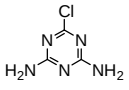
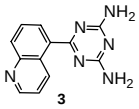
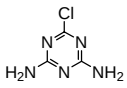
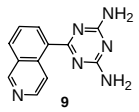
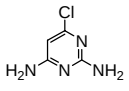
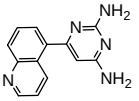
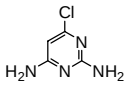
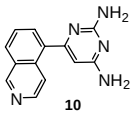
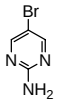
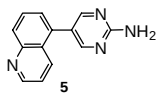
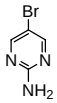
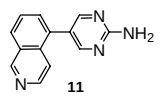
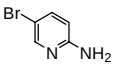
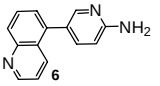
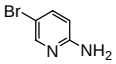
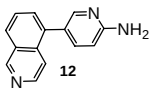
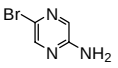
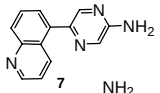
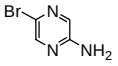
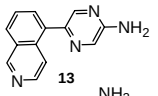
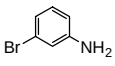
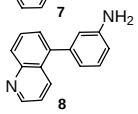
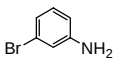
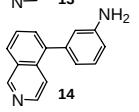
Preparation of quinolyl- and isoquinolylboronic acid and their Suzuki cross-coupling reaction with haloheteroarylamine to get corresponding heteroarylamines in good yields are achieved.

EXPERIMENTAL

General. ^{1}H and ^{13}C NMR spectra were recorded on Bruker AM-300 (300 MHz) and Bruker AM-500 (500 MHz) spectrometer. HRMS spectra were recorded on a Fision 800 Series Gas Chromatograph/Micromass AutoSpec mass spectrometer. Elemental analyses were obtained using a CE instrument EA-1110 elemental analyzer. THF was distilled from sodium and benzophenone prior to use. Unless otherwise

indicated in a specific experiment, all of the chemicals used were reagent grade and no other further purification has been done. All reactions were carried out in oven dried and flame dried glassware under nitrogen atmosphere. Analytical chromatography was performed on silica gel 60 F_{254} plates (0.25 mm). Flash chromatography was performed on E. Merck silica gel 230-400 mesh.

Table 1. Suzuki Reaction of 2a -b with Amino Hetero or Aromatic Halides

Entry	Boronic acid	Halide	Product	Yield (%)	Entry	Boronic acid	Halide	Product	Yield (%)
1	**2a**		 **3**	90[a]	7	**2b**		 **9**	49[a]
2			 **4**	60[a]	8			 **10**	66[a]
3			 **5**	72[b]	9			 **11**	81[b]
4			 **6**	70[b]	10			 **12**	76[b]
5			 **7**	58[b]	11			 **13**	75[b]
6			 **8**	88[b]	12			 **14**	99[b]

a: Dioxnae. b: DME

General Procedure for the Preparation of Quinolylboronic acids

To a solution of 5-bromoquinoline (2.00 g, 9.66 mmol) in THF (50 mL) was added *n*-butyllithium (1.6 M in hexane, 12.0 mL, 19.2 mmol) dropwise at -78 ^{o}C. The reaction mixture was stirred for 45 min at -78 ^{o}C, and then trimethyl borate (3.30 mL, 28.9 mmol) was added quickly. After stirring for 5 min at -78 ^{o}C, the reaction mixture was allowed to warm rt and left to react for an additional hour. The mixture was quenched by slow addition of 4% NaOH aqueous solution. THF was removed *in vacuo* and the aqueous layer was acidified to pH 5-6 by dropwise addition of 3N HCl, keeping the internal temperature below 5 ^{o}C. Extraction with ethyl acetate, dryness with Na_2SO_4, and concentration gave crude crystals, which were washed with 20% ethyl acetate in hexane to remove nonpolar materials. Crude crystals were recrystallized with solvents.

5-Quinolylboronic acid (2a) 748 mg (45% yield). White solid (10% methanol in ethyl acetate) mp 183-184 ^{o}C**;** ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.87 (d, J = 4.3 Hz, 1H), 8.85 (d, J = 8.3 Hz, 1H), 8.48 (s,

2H), 8.03 (d, *J* = 8.3 Hz, 1H), 7.89 (dd, *J* = 7.0, 1.1 Hz, 1H), 7.74 (dd, *J* = 8.3, 7.0 Hz, 1H), 7.52 (dd, *J* = 8.3, 4.3 Hz, 1H); ^{13}C NMR (125 MHZ, CD_3CO_2D) δ 150.50, 148.43, 139.11, 130.32, 122.17; Anal. Calcd for $C_9H_8NO_2B$: C, 62.49 ; H, 4.66 ; N, 8.10. Found: C, 62.12; H, 4.64; N, 7.87.

5-Isoquinolylboronic acid (2b): 931 mg (56% yield). White solid (methanol), mp 205 °C (decomp); ^{1}H NMR (300 MHz, DMSO-d_6) δ 9.13 (s, 1H), 8.31-8.29 (m, 3H), 8.14-8.12 (m, 1H), 7.93 (d, *J* = 8.0 Hz, 1H), 7.88 (dd, *J* = 7.0, 1.1 Hz,1H), 7.47 (dd, *J* = 8.0, 7.0 Hz, 1H); ^{13}C NMR (125 MHz, CD_3CO_2D) δ 147.9, 144.4, 143.9, 132.2, 131.7, 131.1, 128.9, 127.5; Anal. Calcd for $C_9H_8NO_2B$: C, 62.49 ; H, 4.66 ; N, 8.10. Found: C, 62.45 ; H, 4.74; N, 8.21.

Typical Procedure for the Cross Coupling Reactions (Compounds (3-4) and (9-10))

To a mixture of halide (1.0 mmol) and tetrakis(triphenylphosphino)palladium (0.045 mmol relative to boronic acid) in dioxane (35 mL) was added boronic acid (1.5 mmol) followed by aqueous Na_2CO_3 (2 M, 1.0 mL). The reaction mixture was heated under reflux for 48 h. After the removal of dioxane *in vacuo*, THF was added and the suspension was stirred for 1 h. The mixture was filtered, washed thoroughly with THF and the filtrate was evaporated under the reduced pressure. Purification of the residue by column chromatography on silica gel using 10% methanol in dichloromethane provided the corresponding diamine as a white solid.

General Procedure for the Cross Coupling Reactions (Compounds (5-8) and (11-14))

To a mixture of halide (1.0 mmol) and tetrakis(triphenylphosphino)palladium (0.045 mmol) in DME (35 mL) was added boronic acid (1.5 mmol) followed by aqueous Na_2CO_3 (2 M, 1.0 mL). The reaction mixture was heated under reflux for 2-5 h. After the removal of DME *in vacuo*, THF was added, and the suspension was stirred for 1 h. The mixture was filtered, washed thoroughly with THF and the filtrate was evaporated under the reduced pressure. Purification of the residue by column chromatography on silica gel using ethyl acetate provided the corresponding amine as a white solid.

2,4-Diamino-6-quinolyl-1,3,5-triazine (3): White solid (methanol), mp 305 °C (decomp); IR (KBr) 3296, 3111, 1628, 1537, 1454, 796 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6) δ 9.27 (td, *J* = 8.6, 0.8 Hz, 1H), 8.91 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.13 (d, *J* = 2.7 Hz, 1H), 8.12- 8.09 (m, 2H), 7.81 (dd, *J* =8.1, 7.6 Hz, 1H), 7.55 (dd, *J* = 8.6, 4.1 Hz, 1H), 6.90 (br s, 4H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 172.4, 167.5, 150.6, 148.2, 135.9, 135.2, 131.6, 128.8, 128.6, 126.2, 121.8; HRMS (EI) Calcd for $C_{12}H_{10}N_6$ (M^+) 238.0967, found 238.0964.

2,4-Diamino-6-quinolylpyrimidine (4): White solid (methanol), mp 262-264 °C; IR (KBr) 3315, 3147, 2958, 1585, 1423, 1423, 802 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.92 (d, *J* = 4.0 Hz, 1H), 8.68 (d, *J*

= 8.4 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.79 (t, *J* = 7.8 Hz, 1H), 7.64 (d, *J* = 7.1 Hz, 1H), 7.53 (dd, *J* = 8.3, 4.0 Hz, 1H), 6.48 (br s, 2H), 6.08 (br s, 2H), 5.96 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 165.2, 164.0, 163.6, 150.8, 148.2, 138.7, 134.6, 129.9, 129.2, 126.7, 125.9, 121.7, 95.8; HRMS (EI) Calcd for $C_{13}H_{11}N_5$ (M^+) 237.1014, found 237.1014. Anal. Calcd for $C_{13}H_{11}N_5$: C, 65.81 ; H, 4.67 ; N, 29.52. Found: C, 65.70 ; H, 4.54; N, 29.69.

2-Amino-5-quinolylpyrimidine (5): White solid (ethyl acetate), mp 260-261 °C; IR (KBr) 3305, 3137, 1643, 1579, 1546, 1401, 806 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.94 (d, *J* = 4.0 Hz, 1H), 8.61 (d, *J* = 8.3 Hz, 1H), 8.25 (s, 1H), 8.08 (s, 1H), 8.06 (d, *J* = 8.3 Hz, 1H), 7.82 (t, *J* = 7.6 Hz, 1H), 7.70 (d, *J* = 7.0 Hz, 1H), 7.53 (dd, *J* = 8.6, 4.0 Hz, 1H), 6.71 (br s, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 155.2, 150.7, 148.6, 142.8, 140.3, 136.2, 134.5, 131.5, 129.3, 129.2, 127.1, 126.3, 121.8; HRMS (EI) Calcd for $C_{13}H_{10}N_4$ (M^+) 222.0905, found 222.0907.

2-Amino-5-quinolylpyridine (6): White solid (ethyl acetate), mp 156-157 °C; IR (KBr) 3328, 3151, 1656, 1604, 1509, 1396 cm^{-1} ; ^{1}H NMR (300 MHz, DMSO-d_6) δ 9.37(s, 1H), 8.50 (d, *J* = 6.2 Hz, 1H), 8.11 (d, *J* = 7.8 Hz, 1H), 8.06 (d, *J* = 2.4 Hz, 1H), 7.76-7.67 (m, 3H), 7.57 (dd, *J* = 8.6, 2.4 Hz, 1H), 6.64 (d, *J* = 8.6 Hz, 1H), 6.22 (br s, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 159.7, 153.2, 148.4, 143. 6, 138.7, 136.4, 133.8, 130.9, 129.1, 127.6, 127.0, 122.4, 118.2, 107.9; HRMS (EI) Calcd for $C_{14}H_{11}N_3$ (M^+) 221.0953, found 221.0950.

2-Amino-5-quinolylpyrazine (7): White solid (ethyl acetate), mp 250-252 °C; IR (KBr) 3307, 3141, 1546, 1401, 1396 cm^{-1} ; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.93 (d, *J* = 4.0, 1.2 Hz, 1H), 8.61 (d, *J* = 8.6 Hz, 1H), 8.25 (br s, 1H), 8.07 (s, 1H), 8.03 (dd, *J* = 8.5, 0.7 Hz, 1H), 7.79 (td, *J* = 8.4, 0.6 Hz, 1H), 7.67 (d, *J* = 7.1 Hz, 1H), 7.53 (dd, *J* = 8.6, 4.0 Hz, 1H), 6.72 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.2, 150.7, 148.6, 142.8, 140.2, 136.2, 134.6, 131.5, 129.3, 129.2, 127.1, 126.3, 121.8; HRMS (EI) Calcd for $C_{13}H_{10}N_4$ (M^+) 222.0905, found 222.0908.

1-Amino-3-quinolylbenzene (8): White solid (hexane-ethyl acetate), mp 146-147 °C; IR (KBr) 3444, 3313, 3201, 3053, 1589, 1500, 1307, 804 cm^{-1} ;^{1}H NMR (300 MHz, $CDCl_3$) δ 8.91 (dd, *J* = 4.0, 1.7 Hz, 1H), 8.31 (d, *J* = 8.6 Hz, 1H), 8.11 (d, *J* = 8.3 Hz, 1H), 7.73 (dd, *J* = 8.3, 7.0 Hz, 1H), 7.49 (dd, *J* = 7.0, 0.8 Hz, 1H), 7.34 (dd, *J* = 8.6, 4.3 Hz, 1H), 7.27 (dd, *J* = 8.6, 7.5 Hz, 1H), 6.83 (d, *J* = 7.50 Hz, 1H), 6.78-6.76 (m, 2H), 3.77 (br s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 148.8, 146.9,141.1, 140.9, 135.1, 129.7, 129.3, 129.1, 127.3, 127.1, 121.4, 120.8, 117.0, 114.7; HRMS (EI) Calcd for $C_{15}H_{12}N_2$ (M^+) 220.1000, found 220.1001.

2,4-Diamino-6-isoquinolyl-1,3,5-triazine (9): White solid (methanol), mp 275 °C (decomp); IR (KBr) 3405, 3309, 3176, 1623, 1539, 1429, 833 cm^{-1} ; ^{1}H NMR (300 MHz, DMSO- d_6) δ 9.41 (s, 1H), 8.82 (d, *J* = 6.1 Hz, 1H), 8.51 (d, *J* = 6.1 Hz, 1H), 8.36 (d, *J* = 7.1 Hz, 1H), 8.24 (d, *J* = 7.8 Hz, 1H), 7.75 (t, *J* = 7.6 Hz, 1H), 6.90 (br s, 4H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 172.2, 167.5, 153.2, 143.6, 134.2, 133.4, 132.6,

130.6, 129.0, 126.9, 119.4; HRMS (EI) Calcd for $C_{12}H_{10}N_6$ (M^+) 238.0967, found 238.0964. Anal. Calcd for $C_{12}H_{10}N_6$: C, 60.50 ; H, 4.23 ; N, 35.27. Found: C, 60.16 ; H, 4.27 ; N, 34.74 .

2,4-Diamino-6-isoquinolylpyrimidine (10): White solid (methanol), mp 259-260 ^oC; IR (KBr) 3415, 3309, 3168, 1619, 1583, 1421, 806 cm^{-1} ; ^1H NMR (500 MHZ, $CDCl_3$) δ 9.34 (s, 1H), 8.50 (d, *J* = 6.0 Hz, 1H), 8.15 (d, *J* = 8.2 Hz, 1H), 8.12 (dd, *J* = 6.0, 0.8 Hz, 1H), 7.82 (d, *J* = 7.0 Hz, 1H), 7.72 (t, *J* = 7.6 Hz, 1H), 6.45 (s, 2H), 6.06 (br s, 2H), 5.95 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.2, 163.8, 153.1, 143.4, 137.1, 133.1, 130.6, 128.8, 128.7, 118.8, 95.6; HRMS (EI) Calcd for $C_{13}H_{11}N_5$ (M^+) 237.1014, found 237.1010. Anal. Calcd for $C_{13}H_{11}N_5$: C, 65.81 ; H, 4.67 ; N, 29.52. Found: C, 65.72; H, 4.51; N, 29.63.

2-Amino-5-isoquinolylpyrimidine (11): White solid (ethyl acetate), mp 210-212 ^oC; IR (KBr) 3315, 3139, 3031, 1660, 1598, 1504, 1357, 825 cm^{-1} ;^1H NMR (300 MHz, DMSO-d_6) δ 9.39 (s, 1H), 8.53 (d, *J* = 5.9 Hz, 1H), 8.42 (s, 2H), 8.15 (dd, *J* = 5.9, 3.2 Hz, 1H), 7.77-7.71 (m, 3H), 6.96 (br s, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 161.7, 156.9, 151.5, 142.3, 132.0, 131.6, 129.6, 127.3, 126.0, 119.1, 116.2; HRMS (EI) Calcd for $C_{13}H_{10}N_4$ (M^+) 222.0905, found 222.0904.

2-Amino-5-isoquinolylpyridine (12): White solid (ethyl acetate), mp 180-181 ^oC; IR (KBr) 3311, 3111, 1645, 1608, 1506, 1396, 804 cm^{-1} ; ^1H NMR (300 MHz, DMSO-d_6) δ 8.93 (dd, *J* = 4.1, 1.4 Hz, 1H), 8.26 (d, *J* = 8.6 Hz, 1H), 8.05-8.00 (m, 2H), 7.80 (dd, *J* = 8.6, 7.0 Hz, 1H), 7.56-7.50 (m, 1H), 6.63 (d, *J* = 8.6 Hz, 1H), 6.20 (br s, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 159.7, 150.7, 148.6, 148.5, 138.8, 138.0, 133.9, 129.5, 128.4, 127.2, 126.5, 122.7, 121.9, 107.9; HRMS (EI) Calcd for $C_{14}H_{11}N_3$ (M^+) 221.0953, found 221.0954.

2-Amino-5-isoquinolylpyrazine (13): White solid (hexane-ethyl acetate), mp 191-192 ^oC; IR (KBr) 3336, 3160, 3023, 1664, 1581, 1546, 1396, 806 cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 9.32 (s, 1H), 8.54 (d, *J* = 6.2 Hz, 1H), 8.31 (d, *J* = 1.4 Hz, 1H), 8.19 (d, *J* = 1.4 Hz, 1H), 7.68 (d, *J* = 7.6 Hz, 1H), 4.86 (br s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.9, 160.6, 151.3, 150.3, 150.2, 141.9, 141.6, 139.2, 138.6, 136.8, 135.9, 134.5, 125.9; HRMS (EI) Calcd for $C_{13}H_{10}N_4$ (M^+) 222.0905, found 222.0902.

1-Amino-3-isoquinolylbenzene (14): oil. IR (KBr) 3434, 3340, 3214, 3053,1618, 1582, 1484, 1382, 723 cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 9.28 (s, 1H), 8.45 (d, *J* = 6.0 Hz, 1H), 7.94 (dd, *J* = 6.2, 3.3 Hz, 1H), 7.78 (d, *J* = 6.0 Hz, 1H), 7.64-7.61 (m, 2H), 7.30-7.24 (m, 1H), 6.83 (d, *J* = 7.5 Hz, 1H), 6.78- 6.76 (m, 2H), 3.90 (br s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.4, 146.4, 142.8, 139.2, 133.9, 131.7, 130.4, 129.2, 128.6, 126.7, 126.6, 119.9, 118.6, 116.2, 114.2; HRMS (EI) Calcd for $C_{15}H_{12}N_2$ (M^+) 220.1000, found 220.1004.

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