Copper-catalyzed C—N Coupling and Cyclization of 2-(2-Bromophenyl)-1*H*-indoles with Primary Amides Leading to Indolo[1,2-*c*]quinazolines

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It is well-known that indole-fused quinazolines, indolo[1,2-*c*]quinazolines exhibit biological activities such as antibacterial and antifungal properties (Scheme 1).[1,2] Since the synthesis of indolo[1,2-*c*]quinazolines by thermal cyclization of 2-(2-aminophenyl)indoles with acyl cyanides followed by HCl treatment, many synthetic methods for such a hybrid scaffold are well documented.[3,4] Several groups have shown that indolo[1,2-*c*]quinazolines can be formed by condensation of 2-(2-aminophenyl)indoles with aromatic aldehydes and subsequent treatment with powdered $KMnO_4$ for oxidative cyclization.[1,2,5] Such a hybrid scaffold formation was also exemplified by palladium-catalyzed cyclocarbonylation of bis(*o*-trifluoroacetamidophenyl)-acetylene with aryl or vinyl halides and triflates[6] and cyclization of 1-(N-arylimino)indoles formed by addition-elimination between indoles and N-aryltrifluoroacetimidoyl chlorides.[7] It is also reported that 2-(2-halophenyl)indoles are coupled and cyclized with (aryl)methaneamines, amidine hydrochlorides, and amino acids in the presence of a copper catalyst to give indolo[1,2-*c*]quinazolines.[8–10] Wang *et al*. recently reported on synthesis of indolo[1,2-*c*]quinazolines from N-{2-[(2-aminophenyl)ethynyl]phenyl}amides by $ZnBr_2$-promoted domino hydroamination-cyclization.[11] During the course of our continuing studies directed towards cyclization reactions,[12] we have identified several new synthetic methods for N-fused hybrid scaffolds.[13] Under these circumstances, this report describes copper-catalyzed coupling and cyclization of 2-(2-bromophenyl)-1*H*-indoles with primary amides leading to indole-fused quinazolines, indolo[1,2-*c*]quinazolines.

Treatment of 2-(2-bromophenyl)-1*H*-indole (**1a**) with an equimolar amount of formamide (**2a**) in DMF at 130°C for 24 h in the presence of a catalytic amount of CuI (10 mol %) along with Cs_2CO_3 afforded indolo[1,2-*c*]quinazoline (**3a**) in 10% isolated yield with several unidentifiable products (Table 1, entry 1). Higher reaction rate and yield were observed with further addition of L-proline as a ligand (entry 2). It is known that the catalytic system of CuI combined with L-proline effectively catalyzes coupling and cyclization of 2-bromobenzamides with terminal alkynes leading to 3-alkylideneisoindolin-1-ones.[14] The molar ratio of [**2a**]/[**1a**] affected the yield of **3a** and the yield increased with the increase of the molar ratio up to 2.0 along with complete conversion of **1a** (entry 3). Lower reaction temperature resulted in a decreased yield of **3a** (entry 4). The reaction proceeded using other amino acids such as glycine, L-phenylalanine, *N*,*N*-dimethylglycine, and 4-hydroxy-L-proline in place of L-proline, but the yield of **3a** was generally lower than that by the use of L-proline except for 4-hydroxy-L-proline, which exhibited similar activity as L-proline (entries 5–8).

Among bases examined under the employed conditions, Cs_2CO_3 was shown to be the base of choice (entries 3 and 9–11). When used with L-proline and Cs_2CO_3, copper catalysts such as CuCl, CuBr, and copper powder showed lower catalytic activity than CuI (entries 12–14). As a result, even though the exact roles of copper catalysts, ligands and bases are still obscure, the best result in terms of both product yield and complete conversion of **1a** is obtained using the standard set of reaction conditions shown in entry 3 of Table 1.

Based on optimized reaction conditions, various 2-(2-bromophenyl)-1*H*-indoles **1** were subjected to the reaction with primary amides **2** in order to investigate the reaction scope, and several representative results are summarized in Table 2. 2-(2-Bromophenyl)-1*H*-indole (**1a**) was coupled and cyclized with aliphatic, aromatic and heteroaromatic primary amides (**2b-e**) to give the corresponding indolo[1,2-*c*]quinazolines (**3b-e**) in the range of 40–59% isolated yields. However, unlike the reaction with formamide (**2a**), higher reaction temperature was needed for the reaction with **2b-e** to get similar yields of **3b-e** as that of **3a**. For example, treatment of **1a** with **2d** under the employed conditions (130°C) afforded in only 21% isolated yield of **3d**, giving 52% yield of **3d** at higher reaction temperature (150°C). The reaction of 2-(2-bromophenyl)-1*H*-indoles (**1b-d**) having electron-donating and –withdrawing substituents (R^2 and R^3) on indole moiety with primary amides (**2a** and **2d**) also afforded the corresponding coupled and cyclized indolo[1,2-*c*]quinazolines **3f-j** and the

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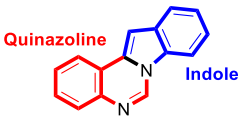


Scheme 1. Indolo[1,2-*c*]quinazoline.

product yield was not affected by the position and electronic nature of the substituent on **1b-d**. With 2-(2-bromophenyl)-1*H*-indoles **1e-g** having straight alkyl chains at position 3 (R^1) of indole moiety, the corresponding coupled and cyclized indolo[1,2-*c*]quinazolines **3k-o** were also invariably formed irrespective of the chain length of **1e-g**. As is the case for the reaction with **1e-g**, the coupling and cyclization also similarly proceeded with 2-(2-bromophenyl)-1*H*-indole **1h** having branched alkyl chain at position 3 (R^1).

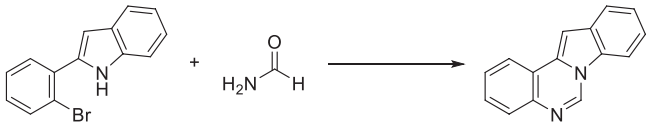
The present reaction seems to proceed via an initial formation of C—N coupled intermediate **4** by copper-catalyzed Ullmann-type coupling between **1a** and **2**, which triggers cyclization and dehydration to give 2-(2-bromophenyl)-1*H*-indole **3** (Scheme 2).[15,16] It is reported that N-substituted *o*-bromobenzamides and 2-(2-bromoaryl)-1*H*-imidazoles are found to be coupled and cyclized with primary amides in the presence of CuI to give quinazolinones and imidazo[1,2-*c*]quinazolines.[17,18] We confirmed in a separate experiment that C—N coupled intermediate **6** is formed in 56% yield from 2-(2-bromophenyl)-1-methyl-1*H*-indole (**5**) and **2d** under the employed conditions (Scheme 3).

In summary, it has been shown that 2-(2-bromophenyl)-1*H*-indoles react with primary amides in the presence of CuI and L-proline along with a base to give indolo[1,2-*c*]quinazolines via C—N coupling and cyclization process. The present reaction provides a new method for the synthesis of indole-fused quinazolines, indolo[1,2-*c*]quinazolines from readily available starting compounds. Further studies on the synthesis of N-fused hybrid scaffolds using copper-catalyzed coupling and cyclization protocol are underway.

Experimental

General. ^{1}H (400 and 500 MHz) and ^{13}C NMR (100 and 125 MHz) spectra were recorded on Bruker Avance (Billerica, MA, USA) spectrometers using TMS as an internal standard. Melting points were determined on a Stanford Research Inc. (Sunnyvale, CA, USA). MPA100 automated melting point apparatus. High-resolution mass data were recorded using electronic ionization (HRMS-EI, magnetic sector-electric sector double focusing mass analyzer) at the Korea Basic Science Center (Daegu, Korea). The isolation of pure products was carried out via thin layer (silica gel 60 GF_{254}, Merck, Darmstadt, Germany) chromatography. The starting 2-(2-bromophenyl)-1*H*-indoles were prepared from the corresponding 1-(2-bromophenyl)alkanones and arylhydrazines according to literature procedures.[19,20] Commercially available organic and inorganic compounds were used without further purification.

Table 1. Optimization of conditions for the reaction of **1a** with **2a**.[a]

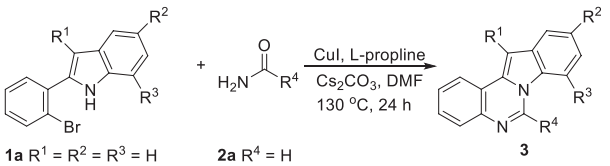


1a **2a** **3a**

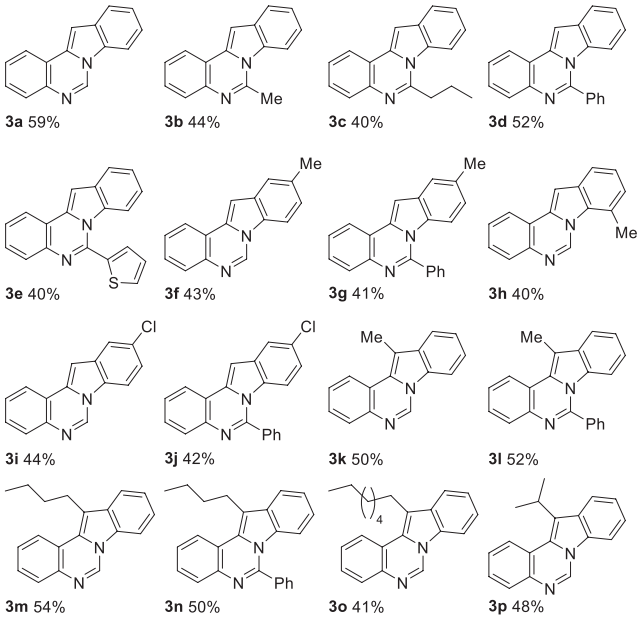
Entry	[**2a**]/[**1a**]	Cu cat.	Ligand	Base	Yield (%)
1	1.0	CuI	—	Cs_2CO_3	10
2	1.0	CuI	L-proline	Cs_2CO_3	27
3	2.0	CuI	L-proline	Cs_2CO_3	59
4[b]	2.0	CuI	L-proline	Cs_2CO_3	36
5	2.0	CuI	Glycine	Cs_2CO_3	36
6	2.0	CuI	L-phenylalanine	Cs_2CO_3	43
7	2.0	CuI	*N*,*N*-Dimethylglycine	Cs_2CO_3	15
8	2.0	CuI	4-hydroxy-L-proline	Cs_2CO_3	58
9	2.0	CuI	L-proline	K_2CO_3	40
10	2.0	CuI	L-proline	NaO^{t}Bu	49
11	2.0	CuI	L-proline	K_3PO_4	35
12	2.0	CuCl	L-proline	Cs_2CO_3	12
13	2.0	CuBr	L-proline	Cs_2CO_3	43
14	2.0	Cu powder	L-proline	Cs_2CO_3	6

[a] Except as noted, all reactions were carried out with **1a** (0.5 mmol), Cu catalyst (0.05 mmol), L-proline (0.15 mmol), base (1.5 mmol), and DMF (5 mL) at 130°C for 24 h.
[b] At 100°C.

Table 2. Copper-catalyzed coupling and cyclization of **1** with primary amides **2** leading to indolo[1,2-*c*]quinazolines **3**.[a]

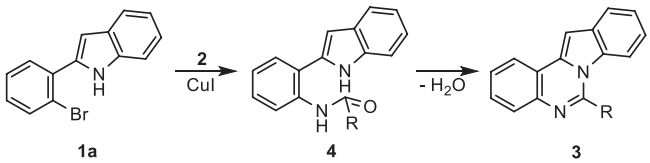


1a $R^1 = R^2 = R^3 = H$ **2a** $R^4 = H$
1b $R^1 = R^3 = H$, $R^2 = Me$ **2b** $R^4 = Me$
1c $R^1 = R^2 = H$, $R^3 = Me$ **2c** $R^4 = Pr$
1d $R^1 = R^3 = H$, $R^2 = Cl$ **2d** $R^4 = Ph$
1e $R^1 = Me$, $R^2 = R^3 = H$ **2e** R^4 = 2-thiophenyl
1f $R^1 = Bu$, $R^2 = R^3 = H$
1g R^1 = heptyl, $R^2 = R^3 = H$
1h R^1 = isopropyl, $R^2 = R^3 = H$



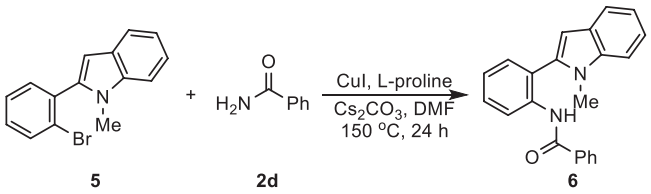
[a] All reactions were carried out with **1** (0.5 mmol), **2** (1 mmol), CuI (0.05 mmol), L-proline (0.15 mmol), Cs_2CO_3 (1.5 mmol), and DMF (5 mL) at 150°C (130°C with **2a**) for 24 h.

General Procedure for CuI-catalyzed Coupling and Cyclization of 2-(2-Bromophenyl)-1*H*-Indoles 1 with Primary Amides 2. To a 25 mL round-bottom flask was added **1** (0.5 mmol), **2** (1.0 mmol), CuI (0.010 g, 0.05 mmol), L-proline (0.018 g, 0.15 mmol), Cs_2CO_3 (0.489 g, 1.5 mmol), and DMF (5 mL). The mixture was stirred at 130–150°C for 24 h. The mixture was then cooled to room temperature and filtered through a short silica gel column (CH_2Cl_2-MeOH) to remove inorganic salts. Removal of the solvent left a crude mixture, which was separated by TLC [silica gel 60 GF_{254} (Merck), CH_2Cl_2-MeOH] to give desired products **3**. Except for known **3a-e**,



1a **4** **3**

Scheme 2. A reaction pathway.



5 **2d** **6**

Scheme 3. Experiment for the formation of C─N coupled intermediate.

3g and **3j**,[10] all new products were characterized spectroscopically as shown below.

10-Methylindolo[1,2-*c*]quinazoline (3f). Solid; mp 215–216°C (from CH_2Cl_2-hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.47 (s, 3H), 6.97 (s, 1H), 7.15 (dd, J = 1.2 and 8.4 Hz, 1H), 7.40–7.48 (m, 2H), 7.52 (s, 1H), 7.70–7.73 (m, 1H), 7.75 (d, J = 8.4 Hz, 1H), 7.95–7.97 (m, 1H), 8.90 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.98, 94.49, 109.67, 120.78, 121.60, 123.29, 124.08, 127.76, 128.30, 128.88, 129.16, 130.20, 133.20, 133.98, 137.33, 139.67; HRMS (EI) anal. calcd for $C_{16}H_{12}N_2$ (M^+): 232.1000. Found: 232.1003.

8-Methylindolo[1,2-*c*]quinazoline (3h). Solid; mp 170–171°C (from CH_2Cl_2-hexane); ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.92 (s, 3H), 7.17–7.18 (m, 2H), 7.33 (t, J = 7.5 Hz, 1H), 7.48–7.51 (m, 1H), 7.52–7.56 (m, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.78 (dd, J = 1.0 and 7.8 Hz, 1H), 8.05 (dd, J = 1.0 and 7.5 Hz, 1H), 9.38 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.75, 95.77, 118.97, 121.63, 122.88, 123.19, 123.97, 125.51, 127.73, 127.94, 129.21, 129.85, 130.44, 133.35, 138.94, 139.53; HRMS (EI) anal. calcd for $C_{16}H_{12}N_2$ (M^+): 232.1000. Found: 232.1000.

10-Chloroindolo[1,2-*c*]quinazoline (3i). Solid; mp 230–231°C (from CH_2Cl_2-hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00 (s, 1H), 7.29 (dd, J = 2.0 and 8.6 Hz, 1H), 7.44–7.53 (m, 2H), 7.72–7.76 (m, 2H), 7.80 (d, J = 8.8 Hz, 1H), 7.97–7.99 (m, 1H), 8.91 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 94.32, 111.08, 120.56, 121.14, 122.75, 123.50, 128.15, 128.52, 128.89, 129.77, 129.99, 130.97, 134.41, 136.90, 139.66; HRMS (EI) anal. calcd for $C_{15}H_9ClN_2$ (M^+): 252.0454. Found: 252.0456.

12-Methylindolo[1,2-*c*]quinazoline (3k). Solid; mp 166–168°C (from CH_2Cl_2-hexane); ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.60 (s, 3H), 7.29–7.32 (m,1H), 7.34–7.42 (m, 3H), 7.65–7.68 (m, 2H), 7.74 (d, J = 8.0 Hz, 1H), 8.03–8.07 (m, 1H), 8.74 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 10.78, 106.21, 109.65, 118.94, 122.54, 123.18, 123.69, 124.00, 127.42, 127.61, 128.12, 128.32, 129.28, 130.69, 137.52, 140.19; HRMS (EI) anal. calcd for $C_{16}H_{12}N_2$ (M^+): 232.1000. Found: 232.0998.

12-Methyl-6-phenylindolo[1,2-*c*]quinazoline (3l). Solid; mp 200–201°C (from CH_2Cl_2-hexane); ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.73 (s, 3H), 6.30 (d, J = 8.5 Hz, 1H), 6.88–6.91 (m, 1H), 7.23–7.26 (m, 1H), 7.41–7.44 (m, 2H), 7.50–7.57 (m, 5H), 7.67 (d, J = 8.0 Hz, 1H), 7.73–7.76 (m, 1H), 8.20–8.23 (m, 1H); ^{13}C NMR

(125 MHz, $CDCl_3$) δ 11.18, 106.99, 114.97, 118.40, 121.88, 122.53, 123.11, 123.82, 127.19, 127.95, 128.35, 128.38, 129.50, 130.13, 130.44, 130.54, 131.43, 136.46, 140.14, 149.79; HRMS (EI) anal. calcd for $C_{22}H_{16}N_2$ (M^+): 308.1313. Found: 308.1311.

12-Butylindolo[1,2-*c*]quinazoline (3m). Solid; mp 118–120°C (from CH_2Cl_2-hexane); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90 (t, *J* = 7.5 Hz, 3H), 1.39–1.46 (m, 2H), 1.64–1.70 (m, 2H) 3.10 (t, *J* = 8.0 Hz, 2H), 7.29–7.37 (m, 2H), 7.38–7.42 (m, 2H), 7.67–7.69 (m, 2H), 7.78 (d, *J* = 8.0 Hz, 1H), 8.02–8.04 (m, 1H), 8.80 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.28, 23.16, 24.97, 32.12, 109.68, 112.10, 119.10, 122.60, 122.95, 123.73, 123.84, 127.21, 127.55, 128.32, 128.38, 129.41, 130.54, 137.65, 140.32; HRMS (EI) anal. calcd for $C_{19}H_{18}N_2$ (M^+): 274.1470. Found: 274.1472.

12-Butyl-6-Phenylindolo[1,2-*c*]quinazoline (3n). Solid; mp 138–140°C (from CH_2Cl_2-hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.6 Hz, 3H), 1.44–1.53 (m, 2H), 1.69–1.77 (m, 2H), 3.20 (t, *J* = 7.6 Hz, 2H), 6.29 (d, *J* = 8.4 Hz, 1H), 6.87–6.91 (m, 1H), 7.21–7.25 (m, 1H), 7.40–7.44 (m, 2H), 7.48–7.57 (m, 5H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.72–7.76 (m, 1H), 8.12–8.17 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.33, 23.28, 25.06, 31.92, 112.75, 115.03, 118.47, 121.90, 122.18, 123.11, 123.58, 127.31, 128.11, 128.36, 128.39, 129.50, 129.64, 130.41, 130.58, 131.22, 136.51, 140.17, 149.87; HRMS (EI) anal. calcd for $C_{25}H_{22}N_2$ (M^+): 350.1783. Found: 350.1781.

12-Heptylindolo[1,2-*c*]quinazoline (3o). Solid; mp 68–69°C (from CH_2Cl_2-hexane); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.9 Hz, 3H), 1.27–1.42 (m, 6H), 1.48–1.54 (m, 2H), 1.75–1.82 (m, 2H), 3.21 (t, *J* = 7.9 Hz, 2H), 7.40–7.53 (m, 4H), 7.76–7.81 (m, 2H), 7.91 (d, *J* = 8.0 Hz, 1H), 8.12–8.17 (m, 1H), 8.94 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.32, 22.90, 25.30, 29.48, 29.96, 30.07, 32.05, 109.72, 112.22, 119.16, 122.65, 123.01, 123.79, 123.90, 127.26, 127.61, 128.35, 128.44, 129.45, 130.57, 137.73, 140.35; HRMS (EI) anal. calcd for $C_{22}H_{24}N_2$ (M^+): 316.1939. Found: 316.1941.

12-Isopropylindolo[1,2-*c*]quinazoline (3p). Solid; mp 114–115°C (from CH_2Cl_2-hexane); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.65 (d, *J* = 7.5 Hz, 6H), 4.09 (sept, *J* = 7.0 Hz, 1H), 7.38–7.44 (m, 2H), 7.46–7.53 (m, 2H), 7.78–7.80 (m, 1H), 7.90–7.94 (m, 1H), 8.04–8.07 (m, 1H), 8.26–8.30 (m, 1H), 8.94 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 22.65, 27.00, 109.86, 118.13, 121.67, 122.37, 122.89, 123.34, 124.13, 126.15, 127.39, 128.43, 128.47, 128.75, 130.14, 137.62, 140.71; HRMS (EI) anal. calcd for $C_{18}H_{16}N_2$ (M^+): 260.1313. Found: 260.1311.

N-(2-(1-Methyl-1*H*-indol-2-yl)phenyl)benzamide (6). Solid; mp 177–179°C (hexane); ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.89 (s, 3H), 6.93 (s, 1H), 7.12 (dd, *J* = 8.0 and 0.6 Hz, 1H), 7.20 (d, *J* = 8.6 Hz, 1H), 7.61–7.64 (m, 1H), 7.72–7.75 (m, 3H), 7.77–7.89 (m, 5H), 8.00–8.02 (m, 1H), 9.16–9.18 (m, 1H), 10.75 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 22.70, 100.95, 109.17, 111.72, 115.01, 115.92, 117.21, 119.17, 119.35, 119.58, 119.78, 119.87, 120.35, 122.03, 123.86, 124.94, 125.57, 135.50, 143.22, 165.07; HRMS (EI) anal. calcd for $C_{22}H_{18}N_2O$ (M^+): 326.1419. Found: 326.1422.

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