Synthesis of Benzo[4,5]imidazo[1,2-c]pyrimidin-1-amines and Their Analogs via Copper-Catalyzed C–N Coupling and Cyclization

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ABSTRACT: 2-(2-Bromovinyl)benzimidazoles and 2-(2-bromophenyl)benzimidazoles react with cyanamide by micro-wave irradiation in dimethylformamide in the presence of a catalytic amount of CuI along with a base to give the corresponding benzo[4,5]imidazo[1,2-c]pyrimidin-1-amines and benzo[4,5]imidazo[1,2-c]quinazolin-6-amines, respectively, in moderate to good yields. 2-(2-Bromophenyl)indoles also react with cyanamide under similar conditions to afford indolo[1,2-c]quinazolin-6-amines. The reaction pathway seems to proceed via a sequence such as intermolecular C–N coupling, C–N formative cyclization, and tautomerization.

INTRODUCTION

Besides a class of homonuclear N-heterocyclic compounds, their N-fused hybrid structures frequently exhibit characteristic biological activities that are not shown in each homonuclear scaffold. Thus, many synthetic methods for such hybrid structures have been developed and tested for biological activities. It is known that benzimidazole-fused pyrimidines, benzo[4,5]imidazo[1,2-a]pyrimidines, also exhibit diverse biological activities and fluorescent properties (Scheme 1, A).−4

However, in contrast to the well-known synthetic methods and biological activities for benzo[4,5]imidazo[1,2-a]pyrimidines, those for benzo[4,5]imidazo[1,2-c]pyrimidines are relatively rare (Scheme 1, B). It is reported that such a scaffold can be synthesized by the initial addition of 2-cyanomethylbenzimidazole to trichloroacetonitrile and the condensation of the resulting adduct with ethyl orthoformate. Goekjian and co-workers have demonstrated that a series of functionalized benzo[4,5]imidazo[1,2-c]pyrimidines can also be prepared by az-Graebe–Ullmann coupling and palladium-catalyzed Buchwald–Hartwig coupling protocols starting from 4,6-dichloropyrimidine and benzotriazole, thus showing activity against anaplastic lymphoma kinase. The course of our continuing studies aiming to develop a new protocol for transition-metal-catalyzed coupling and cyclization reactions led us to seek for a new synthetic method for benzo[4,5]imidazo[1,2-c]-pyrimidines. This report describes copper-catalyzed coupling and cyclization of 2-(2-bromovinyl)benzimidazoles with cyanamide, leading to a class of benzo[4,5]imidazo[1,2-c]pyrimidines under microwave irradiation (Scheme 2). To the best of our knowledge, two reports are found for the synthesis of N-fused hybrid scaffolds using cyanamide. Fu et al. have shown that 2-bromo-N-(2-halophenyl)benzamides and N-(2-haloalkenyl)propioalamides are coupled and cyclized with cyanamide in the presence of a copper catalyst along with a ligand to form benzimidazo[2,1-b]quinazolin-12(6H)-ones and benzo[4,5]imidazo[1,2-a]pyrimidin-4(10H)-ones, respectively.
Scheme 3. Copper-Catalyzed Synthesis of N-Fused Heterocycles Using Cyanamide

Table 1. Optimization of Conditions for the Reaction of 1a and 2

| entry | Cu catalyst | base | solvent | temp (°C) | yield (%) |
|-------|-------------|------|---------|-----------|-----------|
| 1     | Cu          | K₂CO₃| DMF     | 100       | 51        |
| 2     | Cu          | K₂CO₃| DMF     | 100       | 77        |
| 3     | Cu          | K₂CO₃| DMF     | 80        | 62        |
| 4     | Cu          | K₂CO₃| DMF     | 80        | 76        |
| 5     | CuCl        | K₂CO₃| DMF     | 100       | 65        |
| 6     | CuBr        | K₂CO₃| DMF     | 100       | 57        |
| 7     | Cu(OAc)₂·H₂O| K₂CO₃| DMF     | 100       | 65        |
| 8     | Cu powder   | K₂CO₃| DMF     | 100       | 47        |
| 9     | Cu powder   | K₂CO₃| DMF     | 100       | 49        |
| 10    | Cu          | NaOAc| DMF     | 100       | 54        |
| 11    | Cu          | KOH  | DMF     | 100       | 63        |
| 12    | Cu          | NaOBut| DMF    | 100       | 71        |
| 13    | Cu          | Cs₂CO₃| DMF      | 100       | 44        |
| 14    | Cu          | K₂PO₄| DMF     | 100       | 79        |
| 15    | Cu          | K₂PO₄| DMF     | 100       | 79        |
| 16    | Cu          | K₂PO₄| DMSO    | 100       | 73        |
| 17    | Cu          | K₂PO₄| 1,4-dioxane| 100     | 79        |
| 18    | Cu          | K₂PO₄| toluene | 100       | 38        |
| 19    | Cu          | K₂PO₄| DMF     | 100       | 0         |
| 20    | Cu          | K₂PO₄| DMF     | 100       | 0         |
| 21    | Cu          | K₂PO₄| DMF     | 100       | 0         |
| 22    | Cu          | KOH  | DMF     | 100       | 0         |
| 23    | Cu          | NaOBut| DMF   | 100       | 0         |

*Reaction conditions: 1a (0.3 mmol), 2a (0.6 mmol), Cu catalyst (0.03 mmol), base (0.6 mmol), solvent (3 mL), under microwave irradiation (100 W of initial power), 1 h, unless otherwise stated.

**Isolated yield.** Reaction time: 30 min. In the presence of l-proline (0.12 mmol).
The coupling and cyclization with 1d having a dimethyl substituent on the benzimidazole moiety also proceeded to give the corresponding benzo[4,5]imidazo[1,2-c]pyrimidin-1-amine 3d. With cyclic 2-(2-bromovinyl)benzimidazoles 1e–h having various ring sizes, the corresponding coupled and cyclized products, benzo[4,5]imidazo[1,2-c]pyrimidin-1-amines 3e–h, were also formed, as expected, in the range of 57–83% yields. Lower reaction yield, not yet explained, was observed with 1e.

For testing the effect of the position of bromide and benzimidazole groups on benzo-fused 2-(2-bromovinyl)benzimidazoles 1i and 1j were employed. The coupling and cyclization took place irrespective of the position, and both cases are accompanied by the dehydrogenation of benzo[4,5]imidazo[1,2-c]pyrimidin-1-amines initially formed by the coupling and cyclization of 1i and 1j with 2 under the employed conditions. The coupling and cyclization product 3i formed from 1i was partially dehydrogenated to 3i′, whereas the product from 1j was completely dehydrogenated to 3j. Such a similar dehydrogenation was observed by our recent reports on copper-catalyzed coupling and cyclization of β-bromo-α,β-unsaturated carboxylic acids with terminal alkenes and palladium-catalyzed carbynolative cyclization of 2-(2-bromovinyl)benzimidazoles.12 The reaction of acyclic 2-(2-bromovinyl)benzimidazole 1k with 2 also afforded the coupled and cyclized product 3k; however, the yield was lower than that when cyclic 2-(2-bromovinyl)benzimidazoles were used. Similar treatment of 2-(2-bromoaryl)benzimidazoles 1l–n and 2-(2-bromoaryl)imidazole 1o with 2 under the employed conditions also afforded the coupling and cyclization products 3l–o in 69–85% yields. It is known that benzimidazole- and imidazole-fused quinazolines, benzo[4,5]imidazo[1,2-c]quinazolines and imidazo[1,2-c]quinazolines, have a wide spectrum of biological activities and act as commercial drugs.13

The present protocol can be extended to the reaction with 2-(2-bromophenyl)-1H-indoles 4 (Table 2). Similar treatment of 2-(2-bromophenyl)-1H-indole (4a) with 2 under the optimized conditions shown in Table 1 produced indolo[1,2-c]quinazolin-6-amine (5a) in 47% yield. However, further tuning with the reaction temperature resulted in an increased yield of 5a (73% yield at 130 °C). From the reaction of several 2-(2-bromophenyl)-1H-indoles 4b–d with 2, the corresponding coupled and cyclized indolo[1,2-c]quinazolin-6-amines 5b–d were also invariably produced irrespective of straight and branched alkyl chains on the indole moiety. It is also known that indole-fused quinazolines, indolo[1,2-c]quinazolines, exhibit biological activities such as antibacterial and antifungal properties.14

Although no additional experimental efforts for the reaction pathway were performed, the reaction seems to proceed via an initial formation of intermediate 6 by copper-catalyzed Ullmann-type coupling between 1 (or 4) and 2 (Scheme 4).15 This is followed by the intramolecular addition of N–H to CN to form 7, which triggers tautomerization to give product 3.
The following experimental observation is worth noting as evidence for the formation of intermediate 6. We have found that a similar treatment of 2-(2-bromophenyl)-1-methyl-1H-benzo[d']imidazole (8) with 2 under the employed conditions afforded C–N-coupled product 9 in 78% yield (Scheme S, see the Supporting Information). A similar pathway has already been proposed in copper-catalyzed coupling and cyclization reactions using cyanamide.\(^\text{10,16}\)

### Scheme 5. Experiment for Mechanism Study

![Scheme 5. Experiment for Mechanism Study](image_url)

**CONCLUSIONS**

It has been shown that 2-(2-bromovinyl)benzimidazoles and their analogs are coupled and cyclized with cyanamide under microwave irradiation in the presence of CuI to give a class of N-fused hybrid scaffolds, benzo[4,5]imidazo[1,2-c]pyrimidin-1-amines and their analogs. The present reaction provides a new method for synthesizing several N-fused hybrid heterocycles from readily available starting compounds.

### EXPERIMENTAL SECTION

**General Information.** \(^{\text{1H}}\) and \(^{\text{13C}}\) NMR spectra were recorded at 500 and 125 MHz, respectively, in DMSO-\(d_6\) or CDCl\(_3\). Melting points were determined on a microscopic melting point apparatus. High-resolution mass data were recorded using electron ionization (HRMS-EL) magnetic sector-electric sector double-focusing mass analyzer) at the Korea Basic Science Center, Daegu, Korea. All microwave reactions (CEM, Discover LabMate) were carried out in a sealed tube (5 mL), and the reaction temperature was maintained by an external infrared sensor. The isolation of pure products was carried out via thin-layer (a glass plate coated with Kieselgel 60 GF254, Merck) chromatography (TLC). The starting 2-(2-bromovinyl)benzimidazoles and their analogs were prepared by literature procedures.\(^\text{17–19}\)

**Commerically available organic and inorganic compounds were used without further purification.**

**General Procedure for the Synthesis of 3.** A 10 mL microwave reaction tube was charged with 1 (0.3 mmol) and 2 (0.025 g, 0.6 mmol) together with CuI (0.006 g, 0.03 mmol), K\(_2\)PO\(_4\) (0.127 g, 0.6 mmol), and DMF (3 mL). The reaction mixture was heated to 100 °C for 1 h by microwave irradiation at 100 W initial power. The mixture was then cooled to room temperature and filtered through a short silica gel column (ethyl acetate) to remove inorganic components. Removal of the solvent left a crude mixture that was separated by TLC (dichloromethane:MeOH = 19:1) to give 3. Except for known \(^{31}\) new all products were characterized spectroscopically.

\[^{1,2,3,4,5,6}\text{Tetrahydrobenzo}[4,5]\text{imidazo}[1,2-c]\text{quinazolin-6-amine (3a).}^\] White solid (56 mg, 79%). mp 251–253 °C. \(^{\text{1H}}\) NMR (500 MHz, DMSO-\(d_6\)): \(\delta\) 1.78–1.84 (m, 4H), 2.61–2.63 (m, 2H), 2.76–2.78 (m, 2H), 2.73–2.70 (m, 3H), 7.44–7.47 (m, 1H), 7.73 (d, \(J = 8.0\) Hz, 1H), 8.33 (d, \(J = 8.3\) Hz, 1H). \(^{\text{13C}}\) NMR (125 MHz, DMSO-\(d_6\)): \(\delta\) 21.9, 22.5, 30.9, 106.8, 114.3, 118.1, 120.5, 125.2, 127.2, 144.8, 147.0, 150.7, 151.5. HRMS (EI) anal. calcd for C\(_{16}\)H\(_{13}\)N\(_4\) (M\(^+\)): 258.1375. Found: 258.1374.

\[^{2,3,4,5,6}\text{Hexahydrobenzo}[4,5]\text{imidazo}[1,2-c]\text{cyclooctatetra[pyrimidin-8-amine (3g).}^\] White solid (64 mg, 80%). mp 251–252 °C. \(^{\text{1H}}\) NMR (500 MHz, DMSO-\(d_6\)): \(\delta\) 1.33–1.41 (m, 4H), 1.64–1.73 (m, 4H), 2.73–2.75 (m, 2H), 2.97–3.00 (m, 1H), 7.26–7.29 (m, 1H), 7.32 (br s, 2H), 7.44–7.47 (m, 1H), 7.73 (d, \(J = 8.1\) Hz, 1H), 8.34 (d, \(J = 8.3\) Hz, 1H). \(^{\text{13C}}\) NMR (125 MHz, DMSO-\(d_6\)): \(\delta\) 24.4, 25.6, 26.1, 29.5, 29.7, 32.6, 109.6, 114.2, 118.0, 120.0, 125.2, 127.3, 145.0, 147.5, 150.8, 154.3. HRMS (EI) anal. calcd for C\(_{16}\)H\(_{13}\)N\(_4\) (M\(^+\)): 266.1351. Found: 266.1351.
7.30 (m, 1H), 7.44 (m, 1H). 13C NMR (125 MHz, DMSO-d6): δ 21.9, 22.5, 23.7, 23.9, 25.0, 25.4, 26.4, 26.7, 30.0, 110.0, 114.3, 118.1, 120.4, 125.2, 127.0, 149.9, 151.3, 154.1. HRMS (EI) anal. calcld for C18H17N3 (M⁺): 284.1177. Found: 284.1179.

5-Dihydrobenzof[benzo[4,5]imidazo[1,2-c]quinazolin-8-amine (3)]. White solid (48 mg, 56%). mp 227–228 °C. 1H NMR (500 MHz, DMSO-d6): δ 2.85–2.89 (m, 2H), 2.93–2.96 (m, 2H), 7.16–7.19 (m, 1H), 7.26 (d, J = 7.3 Hz, 1H), 7.32–7.37 (m, 2H), 7.51–7.54 (m, 1H), 7.76 (s, 2H), 7.85 (d, J = 7.8 Hz, 1H), 8.41 (d, J = 8.3 Hz, 1H), 9.09 (dd, J = 7.9 and 1.0 Hz, 1H). 13C NMR (125 MHz, DMSO-d6): δ 30.5, 30.7, 105.0, 114.2, 118.4, 121.0, 125.6, 125.8, 126.1, 126.2, 126.4, 127.3, 131.4, 134.5, 144.9, 148.2, 148.3, 154.9. HRMS (EI) anal. calcld for C19H18N4 (M⁺): 322.1577. Found: 322.1581.

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1H and 13C NMR spectra and HRMS data for all new products (PDF)

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Notes
The authors declare no competing financial interest.

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■ ADDITIONAL NOTES
“The yield of 3a was considerably affected by the molar ratio of 2 to 1a. The yield gradually increased from 47% ([2]/[1a] = 1.0), to 60% ([2]/[1a] = 1.5), and to 77% ([2]/[1a] = 2.0). bSimilar treatment of 1a (0.3 mmol) with 2 (0.6 mmol) in DMF (3 mL) in the presence of Cul (0.03 mmol) and K2CO3 (0.6 mmol) under usual heating method (screw-capped vial, 100 °C for 24 h) afforded 3a in 49% yield with 68% conversion of 1a.

It is known that Cu catalyst combined with amino acid effectively catalyzes C═N cross-coupling reactions. See ref 11.

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