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Synthesis of 2-Bromoimidazoles from Alkynes, *N*-Sulfonylazides, and Bromocyanides

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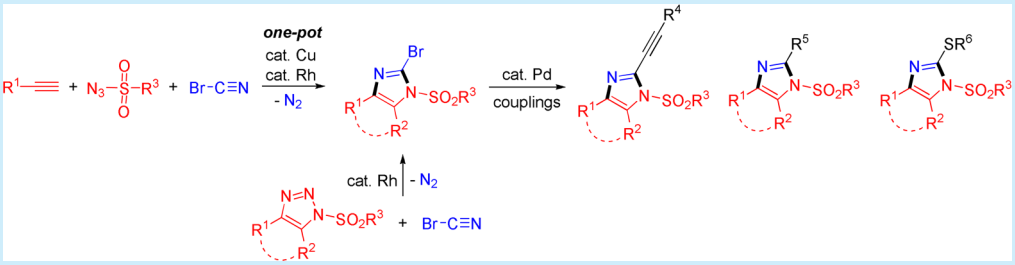
Supporting Information

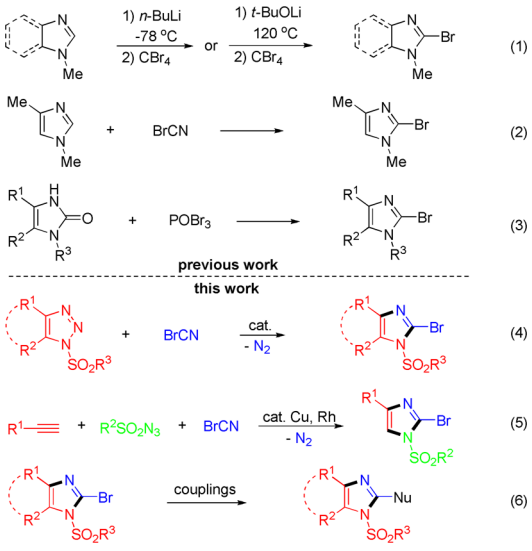


ABSTRACT: A synthetic method for 2-bromoimidazoles is developed from Rh-catalyzed cyclization of *N*-sulfonyl-1,2,3-triazoles with bromocyanides. Cu-catalyzed [3 + 2] cycloaddition followed by Rh-catalyzed cyclization starting from alkynes, *N*-sulfonylazides, and bromocyanides is also demonstrated for *de novo* synthesis of 2-bromoimidazoles in one pot. Moreover, this work was successfully employed to introduce diverse functional groups to the 2-position of imidazoles *via* cross-coupling reaction.

Imidazoles are a significant scaffold of azaheterocyclic compounds, which are extensively used in biologically active compounds,[1] in ionic liquids,[2] and as precursors of stable carbene ligands.[3] Consequently, development of synthetic routes of highly functionalized imidazoles from easily accessible starting materials has been a continuing challenge in modern organic synthesis, regardless of the many reported methods.[4] In particular, because 2-bromoimidazoles can be applied in the introduction of diverse functional groups to imidazole ring *via* cross-coupling reactions, the discovery of novel synthetic approaches to such heterocycles remains a formidable challenge. To date, a myriad of 2-bromoimidazoles could be synthesized by functionalization of a *preformed* imidazole nucleus or 1,3-diazacyclopentane derivatives possessing two nitrogen atoms at the 1,3-position (Scheme 1): reaction of tetrabromomethane with 2-lithioimidazole derivatives obtained from 1-alkylimidazoles or 1-alkylbenzimidazoles with a strong base such as *n*-BuLi at low temperature and *t*-BuOLi at high temperature (eq 1),[5] reaction of bromocyanide with *N*-alkylimidazoles in acetonitrile (eq 2),[6] and treatment of imidazolones with phosphorus oxybromide (eq 3).[7] However, some of these synthetic methods are limited by their low yields, severe conditions (low or high temperature), formation of polybromide, and use of strong bases under anhydrous conditions. In addition, we are not aware of any reports describing the synthesis of 2-bromoimidazoles a *de novo* procedure.

Recently, a synthetic application of *N*-sulfonyl-1,2,3-triazoles as precursors of α-imino Rh carbenoid has been intensively investigated.[8] In particular, Fokin and co-workers reported that Rh-catalyzed transannulation of 1,2,3-triazoles with nitriles

Scheme 1. Approaches for the Synthesis of 2-Bromoimidazoles



produced imidazoles.[9] In continuation of our ongoing program related to the synthesis of azaheterocyclic compounds using *N*-sulfonyl-1,2,3-triazoles,[10] we envisioned that the use of bromocyanide would give 2-bromoimidazoles, and additional valuable transformation is possible despite the risk of

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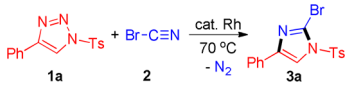
debromination. Herein, we report a synthetic method of 2-bromoimidazoles from Rh-catalyzed cyclization of *N*-sulfonyl-1,2,3-triazoles with bromocyanides (eq 4). Tandem Cu-catalyzed [3 + 2] cycloaddition and Rh-catalyzed cyclization from alkynes, *N*-sulfonylazides, and bromocyanides is also described for *de novo* synthesis of 2-bromoimidazoles in one pot (eq 5). Moreover, this work was extended to introduction of various functional groups to the 2-position of imidazole derivatives *via* a coupling reaction (eq 6).

We initiated our studies with *N*-tosyl-4-phenyl-1,2,3-triazole (**1a**) generated from [3 + 2] cycloaddition of phenylacetylene with tosyl azide in the presence of Cu(I) thiophene-2-carboxylate (Table 1).[11] Although 2.0 mol % of $Rh_2(OAc)_4$, $Rh_2(Oct)_4$, and

Table 1. Reaction Optimization[a]



1a 2 3a

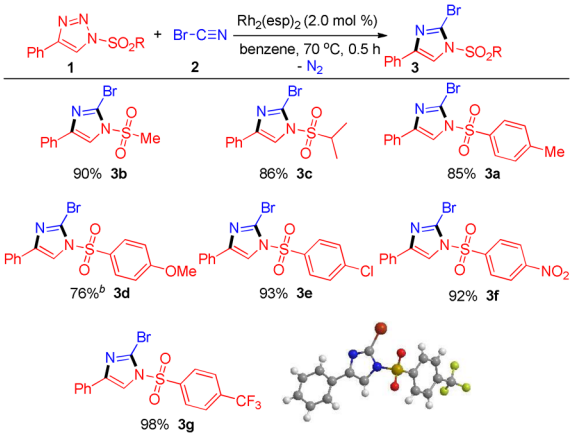
entry	cat. (mol %)	solvent	time (h)	yield[b] (%)
1	$Rh_2(OAc)_4$	DCE	12	(44)
2	$Rh_2(Oct)_4$	DCE	12	(16)
3	Rh_2(*S*-DOSP)$_4$	DCE	12	0
4	$Rh_2(esp)_2$	DCE	1	43
5	$Rh_2(esp)_2$	$CHCl_3$	12	18 (8)
6	$Rh_2(esp)_2$	THF	12	(68)
7	$Rh_2(esp)_2$	1,4-dioxane	12	(34)
8	$Rh_2(esp)_2$	EtOAc	12	(16)
9	$Rh_2(esp)_2$	cyclohexane	12	0
10	$Rh_2(esp)_2$	toluene	1	50
11	$Rh_2(esp)_2$	benzene	1	57
12[c]	$Rh_2(esp)_2$	benzene	1	64
13[c,d]	$Rh_2(esp)_2$	benzene	0.5	86 (85)[e]

[a]Reactions were carried out with **1a** (0.2 mmol) and **2** (3.0 equiv) in the presence of Rh catalyst (2.0 mol %) in solvent (0.8 mL, 0.25 M) at 70 °C. [b]NMR yield using CH_2Br_2 as an internal standard. Numbers in parentheses are NMR yield of **1a**. [c]**2** (5.0 equiv) was used. [d]Solvent (2.0 mL, 0.1 M) was used. [e]Isolated yield of **3a**.

Rh_2(*S*-DOSP)$_4$ as a catalyst are totally ineffective for cyclization of **1a** with bromocyanide **2** (3.0 equiv) (entries 1−3), $Rh_2(esp)_2$ (2.0 mol %) gratifyingly gave the desired product, 2-bromo-4-phenyl-1-tosylimidazole (**3a**), in 43% yield in DCE (0.25 M) at 70 °C for 1 h (entry 4). Electron-deficient dirhodium complexes such as $Rh_2(tfa)_4$ and $Rh_2(pfb)_4$ as catalyst are ineffective. Next, a variety of solvents such as DCE, $CHCl_3$, THF, dioxane, ethyl acetate, cyclohexane, toluene, and benzene were examined. Gratifyingly, the reaction in benzene produced **3a** in 57% yield *via* the cyclization (entry 11). Because of the low boiling point, excessive use of **2** (5.0 equiv) increased the product yield up to 64% (entry 12). Dilution of concentration of the reaction solution from 0.25 to 0.1 M gave superior results (entry 13).

Next, triazoles **1** bearing a number of sulfonyl groups at N1 were investigated in the reaction with **2** (Scheme 2). A modification of the sulfonyl groups at N1 of triazoles **1** did not largely effect on the efficiency of the cyclization. Methane- and isopropanesulfonyl triazoles provided the 2-bromoimidazoles **3b** and **3c** in 90% and 86% yields, respectively. *N*-[(4-Methoxybenzene)sulfonyl]-1,2,3-triazole **1d** is slightly less reactive. In contrast, (benzenesulfonyl)triazoles possessing electron-withdrawing groups such as chloro, nitro, and trifluoromethyl afforded the desired 2-bromoimidazoles **3e**, **3f**, and **3g** in excellent yields. The optimal result was achieved from

Scheme 2. Synthesis of 2-Bromoimidazoles Using *N*-Sulfonyl-1,2,3-Triazoles and Bromocyanides[a]

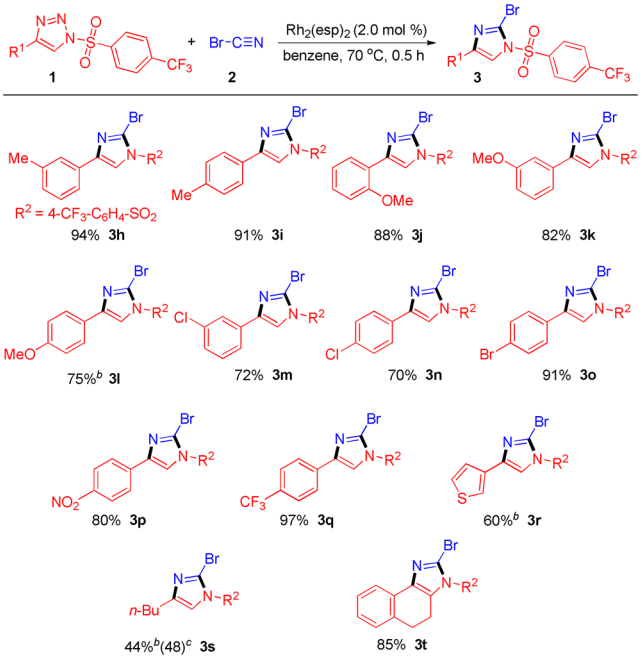


[a]Reactions were carried out with **1** (0.2 mmol) and **2** (5.0 equiv) in the presence of $Rh_2(esp)_2$ (2.0 mol %) in benzene (2.0 mL, 0.1 M) at 70 °C. [b]1 h.

the cyclization of **1g** (0.2 mmol, 1.0 equiv) with **2** (5.0 equiv) using $Rh_2(esp)_2$ (2.0 mol %) in benzene (2.0 mL, 0.1 M) at 70 °C for 0.5 h, providing **3g** in 98% yield. The structure of **3g** was confirmed by X-ray crystallography.

With the optimal reaction conditions, we then explored the scope and limitation of the present method by investigating a wide range of substituents on the aryl group of *N*-[(4-trifluoromethylbenzene)sulfonyl]-4-aryl-1,2,3-triazoles **1** (Scheme 3). Electronic variation of substituents on the aryl ring of **1** did not largely affect the reaction efficiency. For

Scheme 3. Synthesis of 2-Bromoimidazoles Using Triazoles and Bromocyanides[a]



[a]Reactions were carried out with **1** (0.2 mmol) and **2** (5.0 equiv) in the presence of $Rh_2(esp)_2$ (2.0 mol %) in benzene (2.0 mL, 0.1 M) at 70 °C for 30 min. [b]**2** (10.0 equiv) was used. [c]*N*-Sulfonylimine of 1-hexanal.

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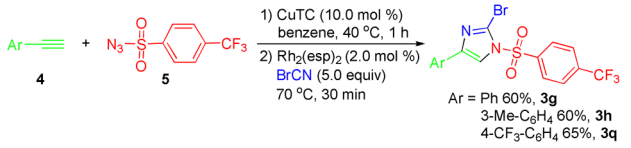
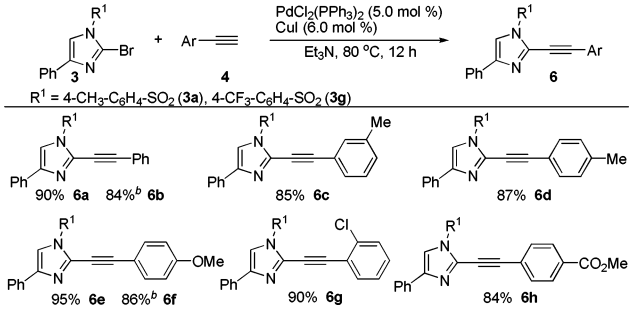
instance, 4-aryl-1,2,3-triazoles having electron-donating methyl and methoxy groups were subjected to the cyclization, affording the corresponding 2-bromoimidazoles in good to excellent yields ranging from 75% to 94%. Also, electron-withdrawing chloro and bromo groups delivered the desired products. 4-Nitro- and 4-trifluoromethyl-substituted 4-aryltriazoles are applicable to the present transformation, leading to 2-bromoimidazoles **3p** and **3q** in 80% and 97% yields, respectively. To our delight, Rh-catalyzed cyclization using thiophene-3-yl substituted triazole **1r** took place to afford **3r** in 60% yield even with use of **2** (10 equiv). However, when *n*-butyltriazole **1s** was subjected to Rh catalyst, the corresponding bromoimidazole **3s** was produced in 44% yield together with the *N*-sulfonylimine of 1-hexanal in 48% yield through *β*-hydride migration.[10b,12] Although 5-phenyltriazole was not totally ineffective, 4,5-disubstituted triazole **1t** turned out to be compatible with the optimal reaction conditions, affording 2-bromoimidazole **3t** in 85% yield.[13]

When a wide range of terminal alkynes was subjected to reaction with bromocyanide to demonstrate the practicability of the one pot process, the corresponding 2-bromoimidazoles were gratifyingly produced in good yields.



Imidazoles having a bromo group at the 2-position provide a chance for further functionalization to access diverse imidazole derivatives (Scheme 4). When 2-bromoimidazole **3g** was reacted

Scheme 4. Sonogashira Reaction of 2-Bromoimidazoles[a]

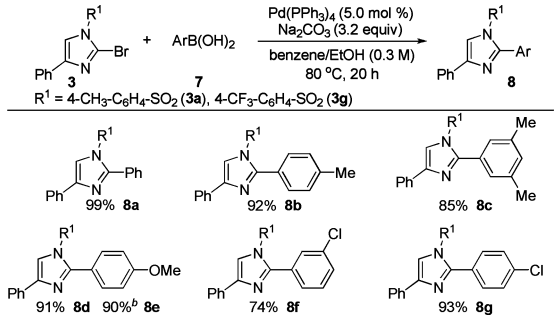


[a]Reaction conditions: 5 mol % of $PdCl_2(PPh_3)_2$, 6 mol % of CuI, **3g**(0.2 mmol), and **4** (1.5 equiv) in Et_3N (0.67 mL, 0.3 M) at 80 °C for 12 h. [b]**3a** (R^1 = 4-$CH_3C_6H_4SO_2$) (0.2 mmol) was used.

with phenylacetylene under Sonogashira reaction conditions [$PdCl_2(PPh_3)_2$, CuI, Et_3N], the alkynylated imidazole **6a** was obtained in 90% yield. *N*-Tosyl-substituted bromoimidazole **3a** was smoothly converted to the coupled product **6b** in 84% yield. The substituents on the aryl group of **4** have no effect on the efficiency of the reaction. Those arylacetylenes **4** with electron-donating and -withdrawing substituents on the aryl ring were treated with 2-bromo-4-phenylimidazoles **3** to furnish the desired products **6c**−**h** in good to excellent yields ranging from 84% to 95%.

The Suzuki reaction of 2-bromoimidazole **3g** with phenylboronic acid produced the corresponding *N*-sulfonyl-2,4-diphenylimidazole **8a** in quantitative yield (Scheme 5). Electronic variation of substituents on the aryl ring of arylboronic acid **7** did not significantly affect the reaction efficiency and

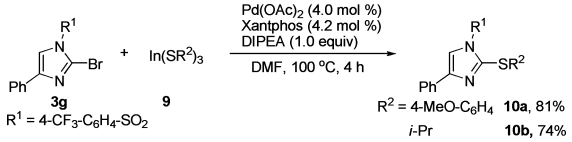
Scheme 5. Suzuki Reaction of 2-Bromoimidazoles[a]



[a]Reaction conditions: 5 mol % of $Pd(PPh_3)_4$, Na_2CO_3 (3.2 equiv) and **3g** (0.2 mmol) and **7** (1.06 equiv) in benzene (0.6 mL) and ethanol (60 μL) at 80 °C for 20 h. [b]**3a** (R^1 = 4-$CH_3C_6H_4SO_2$) (0.2 mmol) was used.

afforded a variety of 2-arylated imidazoles in yields ranging from 74% to 93%.[14]

2-(*p*-Methoxyphenyl)- and 2-isopropylsulfenylated imidazoles **10a** and **10b** were produced in good yields from the reaction of 2-bromoimidazole (**3g**) with indium tris(organothiolates) (**9**).[15]



In summary, Rh-catalyzed cyclization of *N*-sulfonyl-1,2,3-triazoles with bromocyanides was developed for the synthesis of 2-bromoimidazoles. Sequential Cu-catalyzed [3 + 2] cycloaddition and Rh-catalyzed cyclization starting from alkynes, *N*-sulfonylazides, and bromocyanides is also demonstrated for *de novo* synthesis of 2-bromoimidazoles in one pot. Moreover, this work was successfully employed to introduce diverse functional groups to the 2-position of imidazole derivatives *via* a cross-coupling reaction.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures, characterization data, X-ray crystallography data (**3g**), and copies of NMR spectra for all products. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b00977.

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Author Contributions

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Notes

The authors declare no competing financial interest.

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DEDICATION

Dedicated to Professor Sunggak Kim, Ewha Womans University, on the occasion of his 70th birthday.

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(14) The (4-trifluoromethylbenzene)sulfonyl group in **8d** was deprotected with NaOH (15 equiv) in MeOH at 70 °C for 1 h, producing the desired imidazole in 92% yield (see the Supporting Information). Schultz, E. E.; Sarpong, R. *J. Am. Chem. Soc.* **2013**, *135*, 4696.

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