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Catalytic Method for the Synthesis of C−N-Linked Bi(heteroaryl)s Using Heteroaryl Ethers and *N*-Benzoyl Heteroarenes

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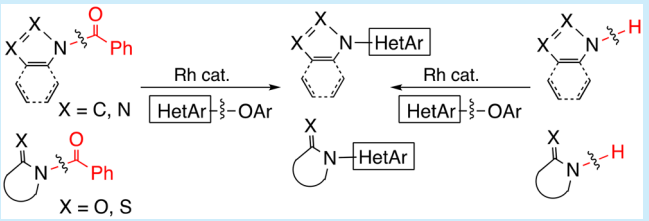
Supporting Information

ABSTRACT: C−N-linked bi(heteroaryl)s are synthesized by a rhodium-catalyzed *N*-heteroarylation reaction of *N*-benzoyl heteroarenes including azoles/azolones, pyridones, cyclic ureas, and cyclic imides using heteroaryl aryl ethers. The reaction involves the covalent bond-exchange reaction of N−CO and HetAr−O bonds without using metal bases and exhibits a broad applicability, giving diverse C−N-linked bi(heteroaryl)s containing five- and six-membered heteroarenes. The *N*-heteroarylation of N−H azoles/azolones and pyridone proceeds at higher reaction temperatures.



The diaryl is a privileged structure for drugs,[1] and the bi(heteroaryl) is an attractive structure because of structural diversity involving various heteroaryls. Such compounds contain several heteroatoms in the diaryl structure capable of interacting with proteins and nucleic acids and likely to exhibit biological activities. In such compounds, along with the carbon−carbon (C−C) linked mode, the carbon−nitrogen (C−N) linked mode is conceivable (Figure 1), which occurs in drugs such as epirizole and zopiclone.[2]

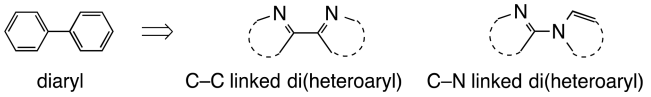


Figure 1. C−C-linked mode and C−N-linked mode.

With regard to the synthesis of C−N-linked bi(heteroaryl)s, a substitution reaction of the N−H-hydrogen of azoles/azolones, pyridones, cyclic ureas, and cyclic imides with halogenated heteroarenes is generally employed in the presence of a metal base or a metal reagent.[3] Catalysis by copper,[4,7−11] palladium,[5] and nickel[6] complexes is also used. Recently, the synthesis of C−N-linked bi(heteroaryl)s by oxidative coupling between the N−H nitrogen in azoles and the C−H carbon in heteroarenes has also been developed in the presence of oxidants.[12] The scope of these conventional syntheses of C−N-linked bi(heteroaryl)s, however, is rather limited, and it is necessary to employ different catalysts, ligands, bases, and reaction conditions depending on the substrates. For example, copper complexes such as CuI, CuCl, Cu_2), and CuO were employed in copper-catalyzed reactions in the presence of stoichiometric amounts of different bases such as K_2CO_3, $CsCO_3$, K_3PO_4, Et_3N, and NaOMe. With regard to the ligand effect, *N*,*N*′-(phenylmethylene)bisacetamide was used for the *N*-heteroarylation reaction of benzotriazoles;[7] salicylaldoxime (salox) for the reaction of pyrazoles;[8] (1*R*,2*R*)-cyclohexanediamine for the reaction of benzimidazoles;[9] 1-methylimidazole for the reaction of carbazoles;[10] and *trans*-*N*,*N*′-dimethylcyclohexane-1,2-diamine for the reaction of 2- and 4-pyridones.[11]

The limited scope was considered to be due to significant differences in the reactivity of metalated *N*-heterocyclic reagents depending on the heteroaryl structure. Heteroarenes have N−H hydrogens with diverse acidities, as shown by examples of N−H protons of triazole derivatives (pK_a 14−15), 2,5-pyrrolidinedione (pK_a 14.7), 4-pyridone (pK_a 14.8), 2-pyridone (pK_a 17.0), 1,3-dihydro-2*H*-indol-2-one (pK_a 18.5), diazoles (pK_a 18−20), indole (pK_a 21.0), and pyrrole (pK_a 23.0).[13] The reactivity of the conjugate bases differs, and controlling the reactivity by designing the metal complex and ligands is necessary depending on the *N*-metalated substrates. In addition, metalated *N*-heteroarenes are often not soluble in organic solvents, and the resulting heterogeneous conditions makes the reaction complex. It is desirable to develop a versatile synthetic method for C−N-linked bi(heteroaryl)s.

Recently, we have reported a synthetic method for unsymmetric bi(heteroaryl) compounds, HetAr-O-HetAr′,[14] HetAr-S-HetAr′,[15] and HetAr-CH_2-HetAr′[16] using a rhodium-catalyzed heteroaryl-exchange reaction between heteroaryl aryl ethers and heteroaryl benzoates/heteroaryl thioesters/heteroarylmethylene ketones.[17] Metalated heteroaryl alcohols and heteroaryl thiols are not used in rhodium-catalyzed reactions, and neutral organic substrates are used such as esters and thioesters. These reactions have a broad applicability and provide various novel bi(heteroaryl) compounds because of the use of bond exchange reactions between C−O bonds and C−S/C−O/C−C bonds in neutral organic substrates.

Described here is the rhodium-catalyzed synthesis of C−N-linked bi(heteroaryl)s by an *N*-heteroarylation reaction using

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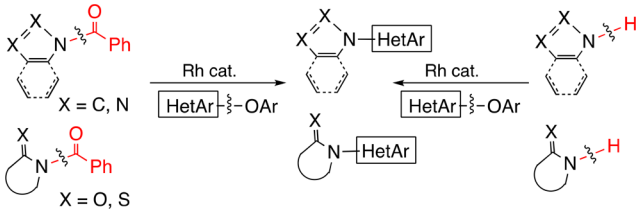
heteroaryl aryl ethers and *N*-benzoyl heteroarenes (Scheme 1). Various *N*-benzoyl azoles containing benzotriazolyl, benzimidazolyl, indolyl, carbazolyl, and pyrrolyl groups, and *N*-benzoyl cyclic amides containing 2-pyridonyl, 4-pyridonyl, 1,3-benzothiazolonyl, 1,3-benzoxazolonyl, imidazolonyl, imidathiazolonyl, 1*H*-isoindole-1,3(2*H*)-dionyl, and 2,5-pyrrolidinedionyl groups were efficiently reacted. In addition, a wide range of electron-deficient/electron-rich five/six-membered heteroaryl aryl ethers such as 2-benzoxazolyl, 2-benzothiazolyl, 2-oxazolyl, 2-furyl, 2-pyridyl, 2-quinazolinyl, and 2-triazyl derivatives were employed. The rhodium-catalyzed *N*-heteroarylation of *N*-benzoyl heteroarenes proceeds without using metal bases, unlike conventional methods, and exhibits a broad scope to provide various C−N-linked bi(heteroaryl)s. *N*-Heteroarylation of *N*-benzoyl heteroarenes by heteroaryl aryl ethers provided various C−N-linked bi(heteroaryl)s with concomitant C−N and C−O bond cleavage and exchange.[18] The *N*-heteroarylation of N−H azoles/azolones and pyridone proceeded at a high reaction temperature and also showed broad applicability.

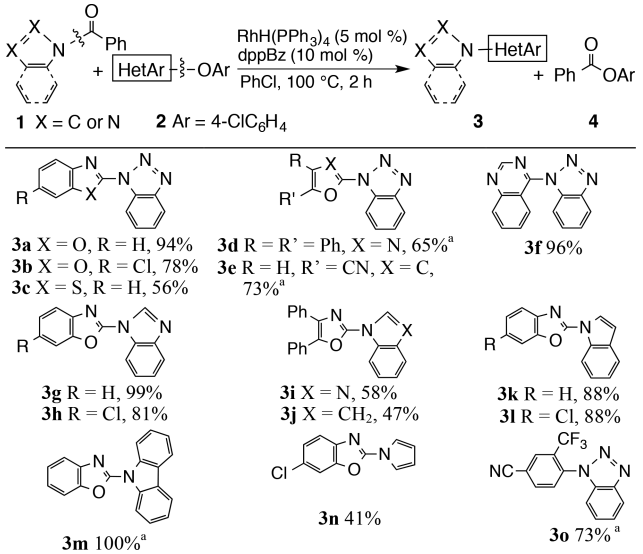
Scheme 1. Rh-Catalyzed *N*-Heteroarylation Reaction



When 1-benzoyl-1*H*-benzotriazole **1a** was reacted with 2-(4-chlorophenoxy)benzoxazole **2a** (1 equiv) in the presence of $RhH(PPh_3)_4$ (5 mol %) and dppBz (10 mol %) in chlorobenzene at 100 °C for 2 h, 1-(2-benzoxazolyl)-1*H*-benzotriazole **3a** (94%), and 4-chlorophenyl benzoate **4** (99%) were obtained (Table 1, **3a**). No reaction occurred in the absence of $RhH(PPh_3)_4$ or dppBz. Various *N*-heteroaryl azoles were synthesized by the heteroaryl exchange reaction of heteroaryl aryl ethers and *N*-benzoyl azoles. Heteroaryl 4-chlorophenyl ethers containing five- and six-membered heteroaryls, such as 1,3-benzothiazolyl, 1,3-benzoxazolyl, 1,3-oxazolyl, 2-furyl, and 4-quinazolinyl groups, reacted with 1-benzoyl-1*H*-benzotriazole **1a**, and the corresponding 1-heteroaryl-1*H*-benzotriazoles **3a−f** were obtained. The reaction of *N*-benzoyl 1*H*-benzimidazole, 1*H*-indole, 9*H*-carbazole, and pyrrole provided C−N-linked bi(heteroaryl)s **3g−n**. This reaction is a novel catalytic method for synthesizing diverse C−N-linked bi(heteroaryl)s from heteroaryl aryl ethers and *N*-benzoyl azoles. Conventional methods for *N*-heteroarylation of azoles employed different catalysts, ligands, and bases depending on substrates. The reaction of **1a** and 3-trifluoromethyl-4-(4-chlorophenoxy)benzonitrile (1 equiv) gave *N*-arylated product **3o** (73%). The ester **4** was isolated as coproducts in all cases.

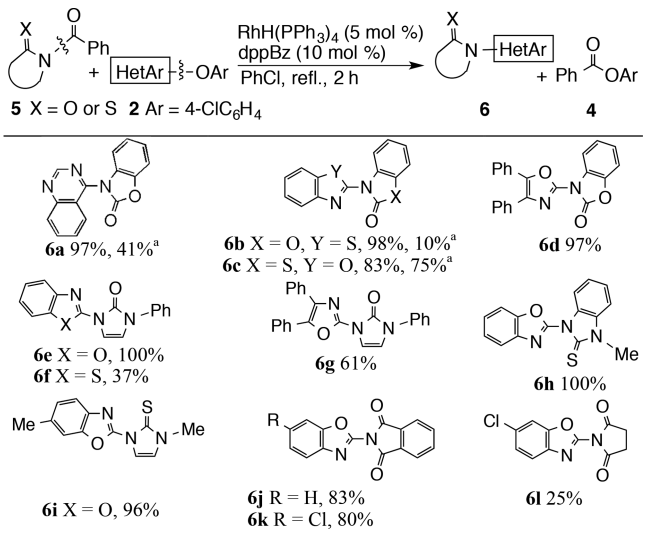
The rhodium-catalyzed heteroaryl exchange reaction was applied to the *N*-heteroarylation of *N*-benzoyl lactams. When 2-(4-chlorophenoxy)quinazoline was reacted with 3-benzoyl-3*H*-benzoxazol-2-one **5a** (1 equiv) in the presence of $RhH(PPh_3)_4$ (5 mol %) and dppBz (10 mol %) in refluxing chlorobenzene (bp 132 °C) for 2 h, 3-(4-quinazolinyl)-3*H*-benzoxazol-2-one **6a** (97%) and 4-chlorophenyl benzoate **4** (98%) were obtained (Table 2, **6a**). The yield of **6a** at 100 °C decreased to 41% yield. No reaction occurred in the absence of $RhH(PPh_3)_4$ or dppBz. Various *N*-heteroaryl azolones, cyclic ureas, and cyclic imides were synthesized by the heteroaryl exchange reaction of heteroaryl aryl ethers and *N*-benzoyl lactams (Table 2). Five- and six-membered heteroaryl 4-chlorophenyl ethers with 1,3-benzothiazolyl, 1,3-benzoxazolyl, 1,3-oxazolyl, and 4-quinazolinyl groups were reacted with **5a**, and the corresponding *N*-heteroaryl benzoxazolones **6a−d** were obtained. The reaction of 1-benzoyl-1,3-dihydro-3-phenyl-2*H*-imidazol-2-one provided *N*-heteroaryl ureas **6e−g**. This reaction was also applied to *N*-heteroarylation of thioamides giving *N*-heteroaryl thiamides **6h** and **6i**. *N*-Heteroarylation of *N*-benzoyl phthalimide and succinimide gave the corresponding *N*-benzoxazolyl compounds **6j**, **6k**, and **6l**. *N*-Heteroarylation of phthalimide and succinimide derivatives has not been reported before. The

Table 1. Rh-Catalyzed *N*-Heteroarylation of *N*-Benzoyl Azoles



[a]Under refluxing PhCl.

Table 2. Rh-Catalyzed *N*-Heteroarylation of *N*-Benzoyl Lactams



[a]Under PhCl at 100 °C.

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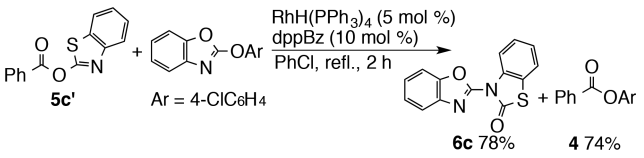
resulting *N*-heteroaryl cyclic ureas and imides except for **6b**, **6c**, **6h**, and **6j** are new compounds.

The above experiments were conducted using *N*-benzoyl azolones, and it was found that *O*-benzoyl derivatives also underwent *N*-heteroarylation. 2-Benzothiazolyl benzoate **5c′**, which was obtained from 2-benzothiazolone and benzoyl chloride using triethylamine at room temperature, was reacted with 2-(4-chlorophenoxy)benzoxazole to give **6c** in 78% yield (Scheme 2). Then both **5c** and **5c′** undergo *N*-heteroarylation

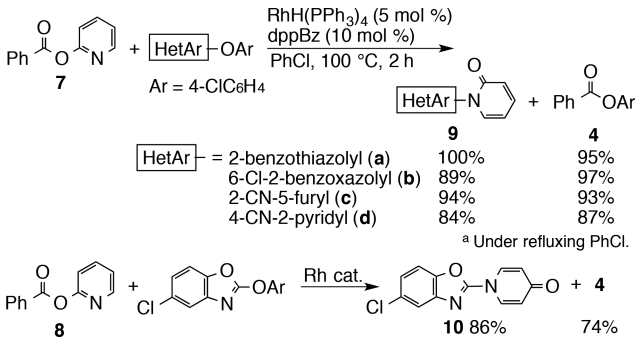
Scheme 2. Rh-catalyzed *N*-Heteroarylation of 5c′



reaction. It is noted that 2-benzothiazolyl benzoate **5c′** quantitatively rearranged to *N*-benzoyl benzothiazolone **5c** in refluxing chlorobenzene for 2 h without the rhodium catalyst.

Pyridyl benzoates were also *N*-heteroarylated. When 2-(4-chlorophenoxyl)benzothiazole was reacted with 2- pyridyl benzoate **7** (1 equiv) in the presence of $RhH(PPh_3)_4$ (5 mol %) and dppBz (10 mol %) in chlorobenzene at 100 °C for 5 h, 1-(2-benzothiazolyl)-1*H*-pyridin-2-one **9a** (100%) and 4-chlorophenyl benzoate **4** (95%) were obtained (Scheme 3).

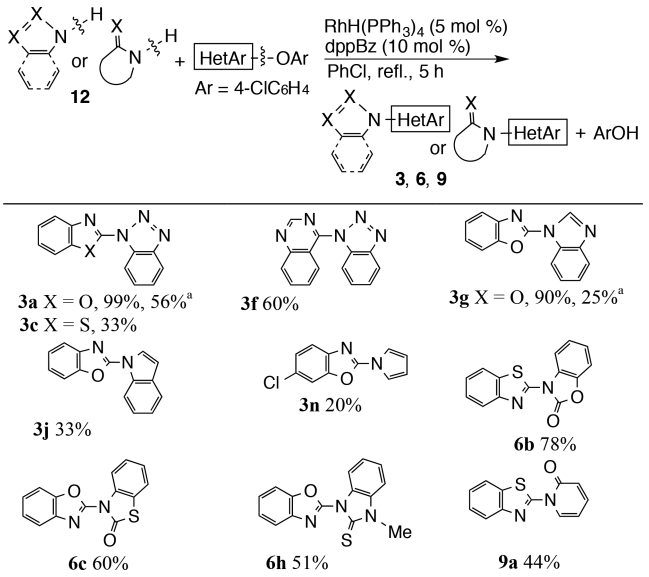
Scheme 3. Rh-Catalyzed *N*-Heteroarylation of 2- and 4-Pyridones



Several heteroaryl (4-chlorophenyl) ethers were reacted with 2- and 4-pyridyl benzoates to give *N*-heteroaryl 2-pyridones and 4-pyridone. It was determined that 2-pyridyl and 4-pyridyl benzoates did not isomerize to 1-benzoyl-1*H*-pyridin-2-one and pyridine-4-one in refluxing chlorobenzene for 2 h in the presence of $RhH(PPh_3)_4$ (5 mol %) and dppBz (10 mol %), which indicated *N*-benzoyl pyridone not being the intermediate of this reaction.

Rhodium-catalyzed *N*-heteroarylation can be applied to N−H azoles/azolones and pyridone at a higher temperature without employing *N*-benzoyl heteroarenes (Table 3). When 2-(4-chlorophenyloxy)benzoxazole was reacted with benzotriazole (1 equiv) in the presence of $RhH(PPh_3)_4$ (5 mol %) and dppBz (10 mol %) in refluxing chlorobenzene for 5 h, 1-(2-benzoxazolyl)-1*H*-benzotriazole **3a** (99%) and 4-chlorophenol (96%) were obtained. It is necessary to elevate the reaction temperature to that of refluxing chlorobenzene. For example, the yields of **3a** and **3g** at 100 °C decreased to 56% and 25% from 99% and 90% in refluxing chlorobenzene, respectively. The benzotriazole reacted efficiently with heteroaryl aryl ethers

Table 3. Rh-Catalyzed *N*-Heteroarylation of *N*-Heteroarenes

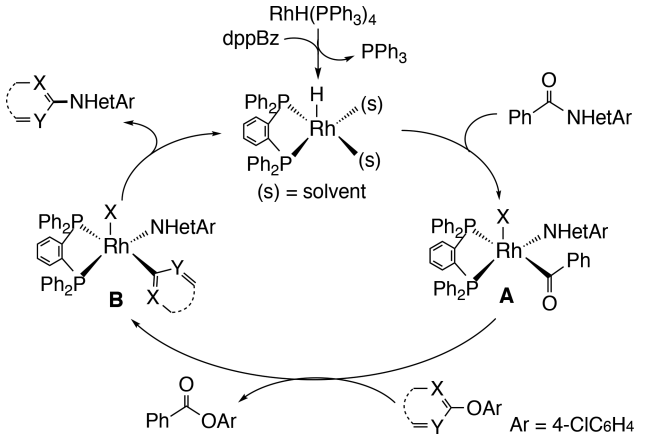


[a]Under PhCl at 100 °C.

containing five- and six-membered heteroarenes giving the corresponding products **3a**, **3c**, and **3f**. *N*-Benzoxazolylation of benzimidazole, indole, and pyrrole provided **3g**, **3j**, and **3n**. *N*-Heteroarylation of azolones and pyridine gave the corresponding *N*-benzoxazolyl/*N*-benzothiazolyl products **6b**, **6c**, **6h**, and **9a**. *N*-Heteroarylation of N−H azoles/azolones and pyridone showed a broad applicability, although the yields decreased for the *N*-benzoyl deriveatives.

A possible mechanism of the reaction is shown in Scheme 4. The phosphine ligand in $RhH(PPh_3)_4$ is exchanged with dppBz,

Scheme 4. Proposed Reaction Mechanism



and the oxidative addition of a *N*-benzoyl heteroarene provides the benzoylrhodium intermediate **A**. The intermediate **A** then undergoes heteroaryl exchange reaction with a heteroaryl aryl ether probably via σ-bond metathesis to form HetAr′−Rh(III)−NHetAr complex **B** and a benzoate ester, and C−N-linked bi(heteroaryl) is liberated by reductive elimination with the regeneration of rhodium catalyst. It is considered that the C−N bond cleavage precedes the C−O bond cleavage. It is because *N*-heteroarylation of *N*-heteroarenes requires more severe reaction conditions than that of *N*-benzoyl heterearenes, and the initial formation of a benzoylrhodium intermediate **A** is

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considered to provide highly reactive species in the rhodium-catalyzed *N*-heteroarylation.

In summary, *N*-heteroaryl azoles/azolones, pyridones, cyclic ureas, and cyclic imides were synthesized from heteroaryl (4-chlorophenyl) ethers and *N*-benzoyl heteroarenes including azoles/azolones, pyridones, cyclic ureas, and cyclic imides using a rhodium-catalyzed heteroaryl exchange reaction. This is a novel method to form the C−N bonds of C−N-linked bi(heteroaryl)s. Rhodium-catalyzed *N*-heteroarylation was also applied to *N*-heteroarylation of N−H azoles/azolones and pyridone. The *N*-heteroarylation reactions have broad applicability and are insensitive to the structures of substrates. It is also worth noting that the byproducts of the reactions, aryl benzoates and phenols, are readily separated and recycled.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b00245.

Experimental procedure, characterization data, and NMR spectra for all products (PDF)

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Notes

The authors declare no competing financial interest.

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