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Nickel(II)/*N*-Heterocyclic Carbene Catalyzed Desulfinylative Arylation by C-S Cleavage of Aryl Sulfoxides with Phenylboronic Acids

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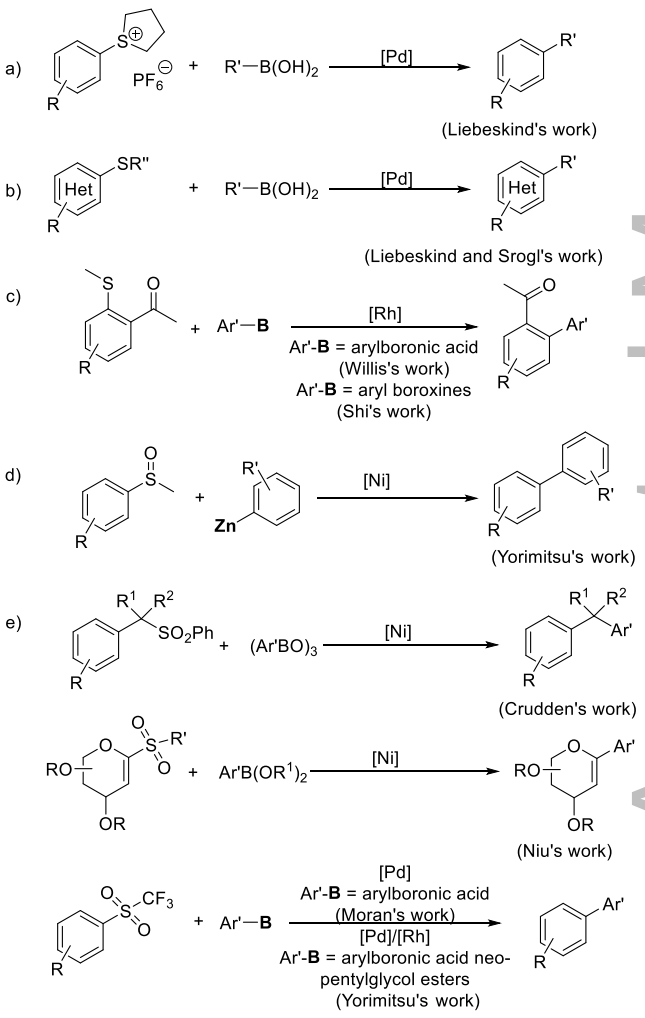
Abstract. Suzuki-Miyaura coupling of haloarenes is the most widely used protocol for the synthesis of biphenyls. Organosulfur compounds are promising electrophiles since they are abundant in nature and versatile in organic synthesis. We report here the desulfinylative Suzuki-Miyaura coupling of aryl sulfoxides with phenylboronic acids using bench-stable nickel/5-(2,4,6-triisopropylphenyl)imidazolylidene[1,5-*a*]pyridines as the catalyst. The ligands are readily prepared from common commercial chemicals. The method is applicable to both symmetric and unsymmetric aryl sulfoxides, and a range of biphenyls bearing various functional groups were obtained in up to 94% yield.

Keywords: *N*-heterocyclic carbene; nickel; Suzuki-Miyaura coupling; phenylboronic acids; sulfoxide

Biphenyls are a quite privileged structural scaffold. Biphenyl units have been found to exist in many important pharmaceuticals, agrochemicals, and materials.[1] One of the most practical and efficient approaches to construct biphenyls is Suzuki-Miyaura cross-coupling, which frequently uses organic halides as the electrophilic component. However, due to the undesirable halogen-based contamination and relatively high cost,[2] significant efforts have been devoted to developing alternatives to organic halides, such as aryl ethers,[3] aryl esters,[4] aryl amides,[5] nitroarenes,[6] dimethyl aryl amines,[7] aryl nitriles,[8] and simple ketones.[9]

Organosulfur compounds are promising electrophiles in cross-coupling reactions due to their abundance in nature and versatility in organic synthesis.[10] The employment of organosulfur compounds including even unactivated aryl sulfides to construct C-C bonds has attracted attention in the recent decade.[11]



Scheme 1. Formation of C-C bonds via C-S Bonds Cleavage.

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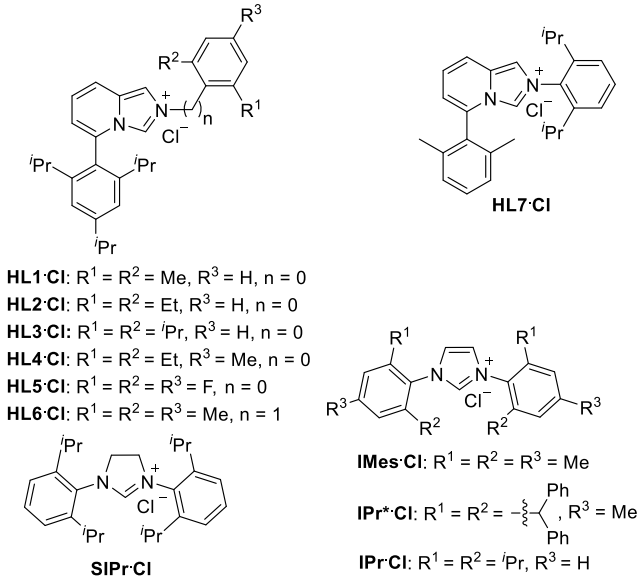
In this context, palladium catalyzed Suzuki-coupling of arylsulfonium salts containing activated C-S bond were firstly reported by Liebeskind and co-workers (Scheme 1a).[11a] Later, palladium catalyzed arylation of activated heteroaromatic thioethers was described by the same group (Scheme 1b).[11b] Although the protocol is highly effective, the scope of thioethers was restricted to electron-deficient heteroaromatic thioethers. Thioesters, thioalkynes, and sulfonyl chlorides were also applied to react with boronic acids to construct C-C bonds.[11c-f] The earlier progress in the metal-catalyzed C-S activation and application of organosulfur compounds as electrophilic reagents in C-C formation reactions were summarized.[11g-k] In 2013, Willis and Shi et al. independently reported a rhodium-catalyzed Suzuki-Miyaura coupling of aryl sulfides (Scheme 1c).[12] The C-S bond cleavage was achieved via an acetyl group as a directing group. Several challenges are associated with this transformation such as the inertness of C-S bond of aryl sulfides toward oxidative addition and the ease of thiolate poisoning of transition metal catalyst. Aryl sulfoxides and sulfones as alternatives are more electron-deficient and thus more reactive.[13] Recently, Yorimitsu et al. reported a nickel-catalyzed Negishi cross-coupling of aryl sulfoxides with arylzinc halides giving biphenyls in good yields (Scheme 1d).[14] More recently, several groups described the Suzuki-Miyaura coupling of sulfones via C-SO_2 bond cleavage of aryl and alkenyl sulfones using nickel,[15a,b] palladium[15c] or bimetallic palladium/rhodium catalysts[15d] (Scheme 1e).



Scheme 2. NHC Ligand Precursors.

To the best of our knowledge, there are no reports on desulfinylative coupling of aryl sulfoxides with phenylboronic acids thus far. Sterically hindered *N*-heterocyclic carbenes (NHCs) have shown their efficiencies in the activation and functionalization of inert C-O, C-N, and C-Cl bonds.[4,7,16] We have recently shown that the NHC ligands having a imidazolylidene[1,5-*a*]pyridine skeleton promote palladium-catalyzed denitrative coupling of nitroarenes.[6b-c,17] We envisioned that the well-designed *N*-heterocyclic carbenes which show strong σ-donating ability and suitable steric hindrance could also promote the desulfinylative coupling of C-S bond of aryl sulfoxides. Herein, we disclose the NHC/Ni catalyzed Suzuki-Miyaura coupling of aryl sulfoxides leading to a number of biphenyls in moderate to excellent yields.

We have shown that NHCs with a imidazolylidene[1,5-*a*]pyridine skeleton are efficient for palladium-catalyzed C-N activation of nitroarenes and successive coupling.[6b-c,17] A series of NHC ligand precursors listed in Scheme 2 were prepared,[6b] and their performance for nickel-catalyzed arylation of aryl sulfoxides were tested. Initially, to screen the reaction conditions, the reaction of diphenyl sulfoxide and 4-methoxyphenylboronic acid was chosen as the model reaction using Ni(acac)$_2$/**HL1·Cl** as the catalyst in 1,4-dioxane at 130 °C. To our delight, **HL1·Cl** bearing a *N*-2,6-dimethylphenyl substituent (R^1 = R^2 = Me) was a suitable ligand and the desulfinylative Suzuki-Miyaura coupling successfully gave 4-methoxybiphenyl **3aa** in 49% yield (Table 1, entry 1). Inspired by this result, we next examined other NHC ligands. When the two *ortho* methyl were replaced by two bulkier ethyl groups or isopropyl groups (R^1 = R^2 = Et, ^{i}Pr), the yields were sharply decreased to below 20% (Table 1, entries 2-4). The use of less sterically hindered ligand, **HL6·Cl**, also gave low yield of **3aa** (Table 1, entry 6). We speculate that the bulkiness of **HL1·Cl** just meets the requirement of C-S oxidative addition and C-C reductive elimination. **HL5·Cl** bearing fluoro substituents at two *ortho* positions (R^1 = R^2 = F) also resulted in low conversion (Table 1, entry 5). These results illustrate that two *ortho* methyl groups are needed to reach satisfactory yield, and the *para*-methyl plays a negative role (Table 1, entry 2 *vs* entry 4). **HL7·Cl** containing a less bulky 2,6-dimethylphenyl group at 5-position showed lower activity, and the desired compound was obtained in diminished yield (Table 1, entry 7). Obviously, the 5-(2,4,6-triisopropylphenyl) moiety plays an important role on the stabilization of active nickel species, and promotes the reductive elimination process. For comparison, the commonly used bulky NHC ligands such as **SIPr**, **IPr**, **IMes**, and **IPr*** were explored, and they were found to be less efficient (Table 1, entries 8-11). The influences of bases were tested. In the presence of K_3PO_4 and CsF, no desired product was observed. The arylation proceeded in 28% yield when KOH was used (Table 1, entries 12-14). Screening of solvents illustrated that the etheric solvents are more appropriate for the reaction (Table 1, entries 15-17). Ni(OAc)$_2$ and $NiBr_2$ were also applied to the coupling, but both of them were almost inactive (Table 1, entries 18-19). We found that

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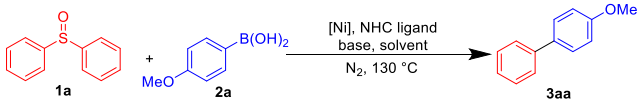
increasing the loading of $Ni(acac)_2$ to 7.5 mol% from 5.0 mol% can further improve the reaction yield to 89% (Table 1, entry 20). The $Ni(acac)_2$/**HL1·Cl** catalyst was not much sensitive to air. When the reaction was performed in air, the desired product **3aa** could also be obtained in 45% yield. Finally, when the reaction time was extended to 36 hours, the yield could be further increased to 94% with a trace amount of diphenyl sulfide detected. The detailed optimizations were listed in Tables S1-S5 (supporting information). In these reactions, a small amount of homocoupling product of arylboronic acids in less than 10% yield was often observed.

Table 1. Optimization of Reaction Conditions[a]



Entry	NHC ligand	Base	Solvent	Yield[b] %
1	**HL1·Cl**	KO^{t}Bu	dioxane	49
2	**HL2·Cl**	KO^{t}Bu	dioxane	19
3	**HL3·Cl**	KO^{t}Bu	dioxane	11
4	**HL4·Cl**	KO^{t}Bu	dioxane	7
5	**HL5·Cl**	KO^{t}Bu	dioxane	13
6	**HL6·Cl**	KO^{t}Bu	dioxane	10
7	**HL7·Cl**	KO^{t}Bu	dioxane	32
8	**SIPr·Cl**	KO^{t}Bu	dioxane	6
9	**IMes·Cl**	KO^{t}Bu	dioxane	12
10	**IPr*·Cl**	KO^{t}Bu	dioxane	<5
11	**IPr·Cl**	KO^{t}Bu	dioxane	16
12	**HL1·Cl**	K_3PO_4	dioxane	0
13	**HL1·Cl**	CsF	dioxane	0
14	**HL1·Cl**	KOH	dioxane	28
15	**HL1·Cl**	KO^{t}Bu	toluene	5
16	**HL1·Cl**	KO^{t}Bu	THF	35
17	**HL1·Cl**	KO^{t}Bu	^{t}BuOH	<5
18	**IPr·Cl**	KO^{t}Bu	dioxane	<5[c]
19	**IPr·Cl**	KO^{t}Bu	dioxane	<5[d]
20	**HL1·Cl**	KO^{t}Bu	dioxane	89[e] 45[f]
21	**HL1·Cl**	KO^{t}Bu	dioxane	94[g]

[a] **1a** (0.3 mmol, 1.0 equiv), **2a** (1.5 equiv), $Ni(acac)_2$ (5 mol%), KO^{t}Bu (3.0 equiv), NHC ligand (10 mol%), dioxane (1.5 mL), N_2, 24 h. [b] Determined by ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard. [c] $Ni(OAc)_2$ instead of $Ni(acac)_2$. [d] $NiBr_2$ instead of $Ni(acac)_2$. [e] $Ni(acac)_2$ (7.5 mol%), NHC ligand (15 mol%), 24 h. [f] $Ni(acac)_2$ (7.5 mol%), NHC ligand (15 mol%), 24 h, in air. [g] $Ni(acac)_2$ (7.5 mol%), NHC ligand (15 mol%), 36 h, isolated yield.

After optimization studies, we set out to explore the substrate scope of the arylation reaction. At first, various symmetric aryl sulfoxides were investigated, and the scope of aryl sulfoxides is presented in Table 2. Under the optimized conditions, a variety of substituted symmetric aryl sulfoxides were transformed to biphenyl derivatives **3aa-3ap** in up to 94% yield. Aryl sulfoxides bearing electron-donating substituents such as alkyl, alkoxy, thioether, trimethylsilyl, and phenyl groups were successfully coupled affording the corresponding biphenyl derivatives **3aa-3ai** in 35-94% yields. Notably, reactive functional groups such as thioether and trimethylsilyl substituents are compatible, giving **3ag** and **3ah** in comparable yields. However, the sterically hindered 2,2'-sulfinylbis(1,3,5-trimethylbenzene) failed to react, and only trace amount of coupling product **3ae** was observed. The result indicates that the desulfinylative coupling is sensitive to steric hindrance of aryl sulfoxides. The reactivities of halo-containing aryl sulfoxides were tested. Ar-F bond maintains in product **3aj**, while Ar-Cl bond was not very compatible due to its competitive reactivity. In the case of 4,4'-sulfinylbis(chlorobenzene), the reaction gave a complicated mixture. **3ak** was afforded in only 10% yield as detected by GC analysis, and about 26% hydrodechlorinated product 1-chloro-4-(phenylsulfinyl)benzene together with 15% 4',4'''-sulfinylbis(4-methoxy-1,1'-biphenyl) which was resulting from the coupling of C-Cl bond were observed. Aryl sulfoxides bearing an electron-withdrawing group such as trifluoromethyl were successfully transformed to **3al** and **3am** in 77% and 85% yields, respectively. These transformations are very important since CF_3 often exists in many pharmaceutically structures. However, the cyano-containing aryl sulfoxide showed low yield possibly because of the high sensitivity of the cyano group toward strong bases. Under the standard conditions, the unsymmetric aryl sulfoxide was applied to the desulfinylative coupling. Unsymmetric 4-methyl-4'-(trifluoromethyl)-1,1'-biphenyl **1q**, was successfully transformed to **3al** and **3ab** in 77% and 12% yields, respectively, which showed that the C-S bond at the electron-deficient side is weaker and its desulfinylative coupling is preferred.

Table 2. Scope of Aryl Sulfoxides[a]

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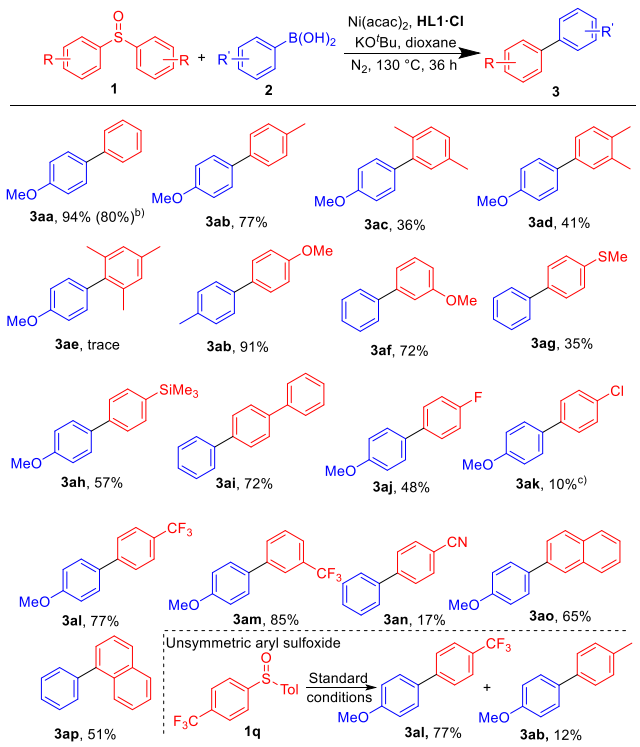
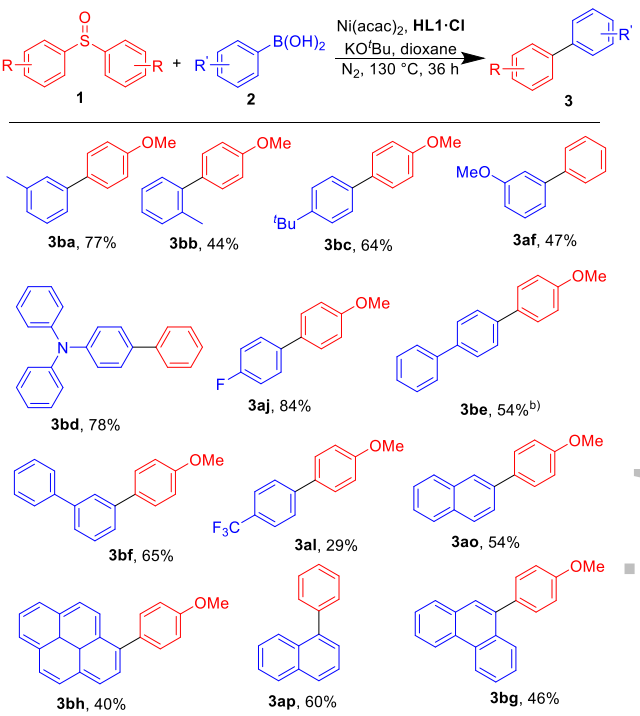


[a] **1** (0.3 mmol, 1.0 equiv), **2** (1.5 equiv), $Ni(acac)_2$ (7.5 mol%), KO^{t}Bu (3.0 equiv), **HL1·Cl** (15 mol%), dioxane (1.5 mL), N_2, 36 h, isolated yields. [b] A gram-scale reaction using 1.01 g of diphenyl sulfoxide, and 1.14 g of 4-methoxyphenylboronic acid under the optimized conditions, isolated yield. [c] Determined by GC analysis.

The reaction of unsymmetric (methylsulfinyl)benzene with 4-methoxyphenylboronic acid was also tested under the same conditions. The desired product 4-methoxybiphenyl was obtained in 14% yield based on ^{1}H NMR analysis. Most (methylsulfinyl)benzene remained unreacted. It was believed that the C(sp^2)-(S=O) bonds of alkyl aryl sulfoxides would be more electron-rich and less reactive toward oxidative addition to electron-rich Ni(0) species.[18] Finally, we examined the possibility of heteroaryl sulfoxides. Unfortunately, the coupling of 3-((2,6-dimethylphenyl)sulfinyl)pyridine and 2,2'-sulfinyldithiophene failed.

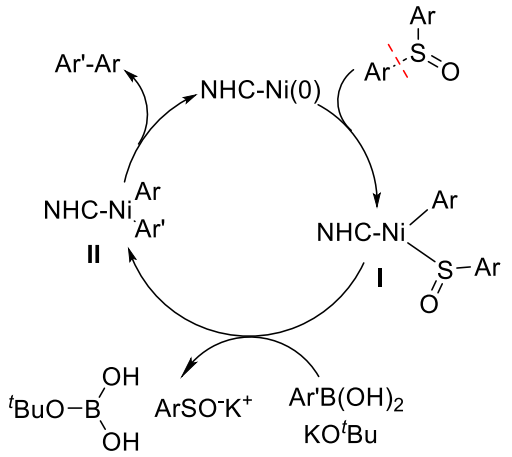
Table 3. Scope of Arylboronic Acids[a]



[a] **1** (0.3 mmol, 1.0 equiv), **2** (1.5 equiv), $Ni(acac)_2$ (7.5 mol%), KO^{t}Bu (3.0 equiv), **HL1·Cl** (15 mol%), dioxane (1.5 mL), N_2, 36 h, isolated yields. [b] Toluene instead of dioxane was used as the solvent.

We next explored the scope of the desulfinylative Suzuki-Miyaura coupling with respect to arylboronic acids. The results for phenyl sulfoxide and 4,4'-dimethoxy diphenylsulfoxide were summarized in Table 3. Arylboronic acids containing different functional groups at *o*-, *m*-, and *p*-positions could undergo the reaction giving the corresponding products in 29-84% yields. Generally, electron-rich arylboronic acids containing alkyl, alkoxyl, amino, and phenyl substituents are favorable to the arylation. The fluoro and amino groups were not affected, and the corresponding products **3aj** and **3bd** were obtained in 84% and 78% yields, respectively. However, electron-deficient arylboronic acid showed diminished yield giving **3al** in 29% yield. As for polycyclic arylboronic acids, biaryl derivatives **3ao**, **3ap**, **3bg**, and **3bh** were produced in moderate yields.

Based on the previous literature reports,[13-14,19] a plausible catalytic cycle in Scheme 3 was proposed. The reaction of $Ni(acac)_2$ and **HL1·Cl** would *in situ* generate a highly electron-rich NHC-Ni(0) species under basic conditions. Oxidative addition of Ar_2SO toward (NHC)Ni(0) would form intermediate **I**. Subsequent transmetalating between the resulting intermediate **I** and arylboronic acid would give **II**. Finally, the target product would be generated through reductive elimination of **II**.



Scheme 3. Plausible catalytic cycle.

In summary, we have developed an efficient nickel/NHC-catalyzed Suzuki-Miyaura coupling of aryl sulfoxides. The key to success is the rational design of NHC ligands which show strong donating ability and suitable steric hindrance. The NHC ligands are generated *in situ* from 5-(2,4,6-triisopropylphenyl)imidazoylidene[1,5-*a*]pyridine and their precursors are readily prepared from inexpensive and common commercial reagents. A number of biphenyls were obtained in moderate to excellent yields. However, heterocyclic coupling partners are still limited. Further improvement of the NHC ligands is needed to expand the scope of heteroaryl sulfoxides as well as heteroarylboronic acids.

Experimental Section

General Procedure for the Suzuki-Miyaura Coupling of Aryl Sulfoxides

To an oven-dried tube equipped with a magnetic stirring bar were added sequentially diaryl sulfoxide **1** (0.3 mmol), boronic acid **2** (0.45 mmol), $Ni(acac)_2$ (5.8 mg, 0.0225 mmol), **HL1·Cl** (20.7 mg, 0.045 mmol), KO^{t}Bu (101 mg, 0.9 mmol), and dioxane (1.5 mL) under N_2 atmosphere. The reaction mixture was stirred at room temperature for 30 minutes, and then heated by a mantle (IKA RCT B S025) at 130 °C for 36 hours. The reaction mixture was cooled to room temperature, and then it was passed through a short pad of celite with CH_2Cl_2. The solution was concentrated in vacuo. The residue was purified by silica gel column chromatography to give the coupling product **3**.

Acknowledgements

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COMMUNICATION

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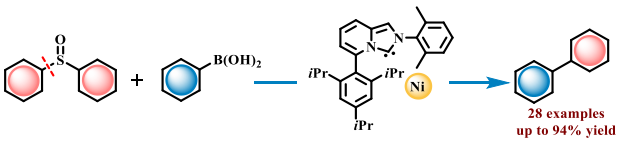

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