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Nickel(II)-Catalyzed Addition of Aryl-, Alkenyl-, and Alkylboronic Acids to Alkenylazaarenes

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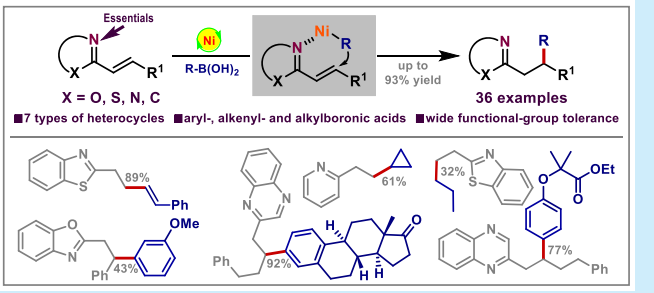
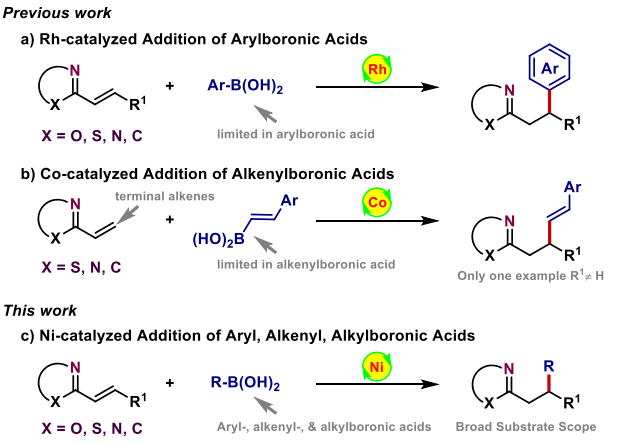
ABSTRACT: A nickel(II)-catalyzed addition of aryl-, alkenyl-, and alkylboronic acids to alkenylazaarenes was presented. This reaction exhibited high efficiency (up to 93% yield), a broad substrate scope (seven types of heterocycles), and good functional group compatibility. The resulting products can be further transformed to many useful building blocks. Finally, the preliminary studies suggested that the adjacent N atom of the heterocycles was essential for the high reactivity.



Nitrogen-containing heteroarenes are privileged scaffolds in pharmaceutical molecules, advanced materials, and natural products.[1] Therefore, significant efforts have been put into developing methods for the functionalization of the heteroarenes and their derivatives. The metal-catalyzed cross-coupling reactions[2] and the recently emerged C−H functionalization reactions including biaryl coupling and insertions of an unsaturated bond initiated by *ortho*-C−H activation have been identified as powerful tools to afford these structures.[3] Apart from these commonly used methods, the catalytic nucleophilic addition to the alkenylheteroarenes, in which the adjacent alkene groups were activated by the electron-withdrawing heteroaromatic moiety, provided an alternative approach to constructing the heteroarene-containing building blocks.[4−7]

However, compared with the addition of common α,β-unsaturated carbonyl derivatives, alkenylheteroarenes are less reactive due to the weak activation from the heteroaromatic environment.[4c,6a,f] In most cases, Grignard reagents,[4,6a,b,g] which have to be carefully handled, were essential for the success of the reaction. Boronic acids are widely used due to their stability, low toxicity, and commercial availability. But as for the addition of boronic acids to the alkenylheteroarenes, it was not until 2001 that an effective Rh-catalyzed addition of arylboronic acid to β-unsubstituted alkenyl *N*-heteroarenes was disclosed by Lautens and coworkers (Scheme 1a).[5a] Then, in 2010, Lam and coworkers made a seminal contribution by developing an enantioselective Rh-catalyzed addition of arylboronic acids to β-substituted alkenyl *N*-heteroarenes.[5d] Unfortunately, as for the alkenylboronic acids, no products were observed in their report. As the alternatives to alkenylboronic acids, the alkenyl *N*-methyliminodiacetic acid (MIDA) boronates were essential to afford the alkenylation product in modest yield with a modest *ee* value. At almost the same time, Yorimitsu, Oshima, and coworkers realized a Co-catalyzed alkenylation of the alkenylazaarenes (Scheme 1b);[5g] however, arylboronic, alkylboronic, and cyclic alkenylboronic acids were incompatible in the reaction system, and only one-type products of the β-substituted alkenyl *N*-heteroarene have been presented. Considering the paucity of the current methodologies for the addition of alkenylheterocyclic aromatic rings with boronic acids and the lack of reports on the Ni-

Scheme 1. Catalytic Nucleophilic Addition of Boronic Acids to Alkenylazaarenes



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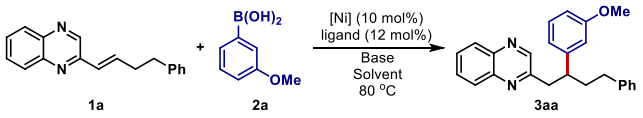
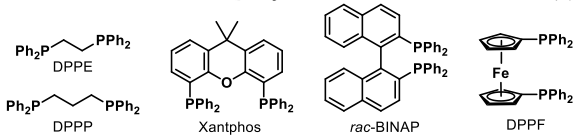
catalyzed 1,4-addition of the organoboron reagents,[8] herein, we present a highly efficient nickel(II)-catalyzed addition of aryl-, alkenyl-, and alkylboronic acids to alkenylazaarenes.

We commenced our studies by examining the arylation of alkenylquinoxaline **1a** with (3-methoxyphenyl)boronic acid (**2a**) in the presence of $Ni(acac)_2$, DPPE, and K_2CO_3. To our delight, the desired product **3aa** was observed with 45% yield accompanied by a trace reduction product (Table 1, entry 1).[5d]

Table 1. Selected Optimization Studies[a]



entry	ligand	base	solvent	yield (%)[b]
1	DPPE	K_2CO_3	toluene	45(2)[c]
2	DPPE	Na_2CO_3	toluene	10(0)[c]
3	DPPE	Cs_2CO_3	toluene	0
4	DPPE	KOH	toluene	12(0)[c]
5	PPh_3	K_2CO_3	toluene	0
6	DPPP	K_2CO_3	toluene	40(4)[c]
7	Xantphos	K_2CO_3	toluene	44(0)[c]
8	DPPF	K_2CO_3	toluene	62(9)[c]
9	*rac*-BINAP	K_2CO_3	toluene	58(0)[c]
10	*rac*-BINAP	K_2CO_3	DCE	0
11	*rac*-BINAP	K_2CO_3	TFE	0
12	*rac*-BINAP	K_2CO_3	dioxane	61(0)[c]
13	*rac*-BINAP	K_2CO_3	dioxane/H_2O[h]	0
14[d]	*rac*-BINAP	K_2CO_3	dioxane	0
15[e]	*rac*-BINAP	K_2CO_3	dioxane	84(0)[c]
16[f]	*rac*-BINAP	K_2CO_3	dioxane	64(0)[c]
17[g]	*rac*-BINAP	K_2CO_3	dioxane	52(0)[c]
18	-	K_2CO_3	dioxane	69(0)[c]



[a]Reactions were carried out with **1a** (0.2 mmol), **2a** (0.6 mmol), $Ni(acac)_2$ (10 mol %), ligand (12 mol %), base (0.4 mmol), and solvent (2.0 mL) under an Ar atmosphere, 6 h. [b]Determined by ^1H NMR analysis of unpurified mixtures using CH_2Br_2 as an internal standard. [c]Number in parentheses indicates the yield of reduction product. [d]$BF_3{\cdot}OEt_2$ (40 mol %) was added. [e]Under 120 °C. [f]**2a** (0.3 mmol) was used instead. [g]$Ni(acac)_2$ (5 mol %) and *rac*-BINAP (6 mol %) were used instead. [h]Dioxane/H_2O = 9/1 (v/v).

Other bases, including Na_2CO_3, Cs_2CO_3, and KOH, were then tested, respectively, and led to a big erosion of the reaction yield without exception (Table 1, entries 2−4). Next, several representative phosphine ligands were carefully screened. The monophosphine ligand PPh_3 showed inefficiency for this reaction (Table 1, entry 5). The usage of DPPP and Xantphos ligands resulted in a slight decrease in the yields (Table 1, entries 6 and 7). When the DPPF ligand was checked, the reaction yield was dramatically improved; however, a certain amount of reduction product as a side product was also produced, which made purification difficult (Table 1, entry 8). The commonly used *rac*-BINAP ligand gave a similar yield, and to our pleasure, no reduction product was detected (Table 1, entry 9). Therefore, *rac*-BINAP was chosen as the privileged ligand for further optimization. Other solvents, including 1,2-dichloroethane (DCE), 2,2,2-trifluoroethanol (TFE), 1,4-dioxane, and 1,4-dioxane/H_2O (9:1), were then screened, but only dioxane afforded the desired product with a slight increase in yield (Table 1, entries 10−13). The Lewis acid additive ($BF_3{\cdot}OEt_2$) was proved to promote the Cu-catalyzed addition of alkenylazaarenes;[6a] however, it was detrimental in our case (Table 1, entry 14). Increasing the reaction temperature could significantly enhance the yield to 84% (Table 1, entry 15), whereas reducing the loading of boronic acid **2a** or the catalyst caused a decrease in the yield (Table 1, entries 16 and 17). Finally, this reaction showed lower efficiency without the ligand (Table 1, entry 18).

With the optimized condition in hand, various substrates were screened, and the results are summarized in Scheme 2. A variety of *meta*-substituted and *para*-substituted arylboronic acids, regardless of the electron-donating or electron-withdrawing property of the substituent at the benzene ring, were amenable in this reaction (Scheme 2, **3aa**−**ah**), and a wide array of functional groups, including ether (**3aa**), ester (**3ab**), halides (**3ac** and **3ad**), and trifluoromethyl (**3ae**) and silyl groups (**3ag**), were comfortably tolerated under the condition. It should be mentioned that the sterically hindered boronic acid, that is, 2-methylphenyl boronic acid, could also afford the addition product in excellent yield (**3ai**). As for the 1-naphthyl-, 2-naphthyl-, and di- and trisubstituted aryl boronic acids, the reaction proceeded equally well (Scheme 2, **3aj**−**am**). Other functionalized alkyl groups at the β-position of the 2-alkenylquinoxaline had no influence on the efficiency of the reaction (Scheme 2, **3ba** and **3ca**). The 1-dibenzothiophenyl group can also be present in our reaction (Scheme 2, **3an**). Additionally, other alkenylazaarenes, including 2-alkenylquinoline and 2-alkenylbenzoxazole, afforded the desired product in moderate yield by modifying the ligand from *rac*-BINAP to DPPF (Scheme 2, **3da** and **3ea**). When the readily available phenylboronic acids **2o** and **2p** derived from estrone and clofibrate were subjected to this transformation, the addition products were obtained in high to excellent yields (Scheme 2, **3ao** and **3ap**). As for the internal alkenes, alkenyl- and alkylboronic acids failed to afford the desired addition products, and only small amounts of homocoupling products were observed.

As for the 2-vinyl *N*-heteroarenes, a broader scope of boronic acids was applied to this reaction. Not only the arylboronic acids but also the alkenylboronic acids and even the alkylboronic acids could afford the addition product in moderate to high yields (Scheme 2, **3fa**−**nx**). It is noteworthy that 2-vinylpyridine **1h** bearing a methyl group at the six position still provided the product **3hr**, albeit in relatively low yield. Considering that the heteroarenes play an important role in the pharmaceuticals and natural products, different heteroarenes including quinoxaline (**3iw** and **3ir**), triazine (**3jr**), quinoline (**3kr**), pyrimidine (**3lr** and **3mr**), and benzothiazole (**3nr** and **3nx**) were tested under the same reaction condition, and all of them afforded the desired addition products.

The importance of the adjacent N atom in the heterocycles was elaborated by several control experiments. Only trace product was observed for the alkene substrate **1p**, **1r**, or **1s** without the nitrogen atom or with it at another position of the heterocycles. This result clearly indicated that the coordination between the metal catalyst and the N atom would activate the β-carbon of heteroaryl alkenes and increase their reactivities (Scheme 3).[9]

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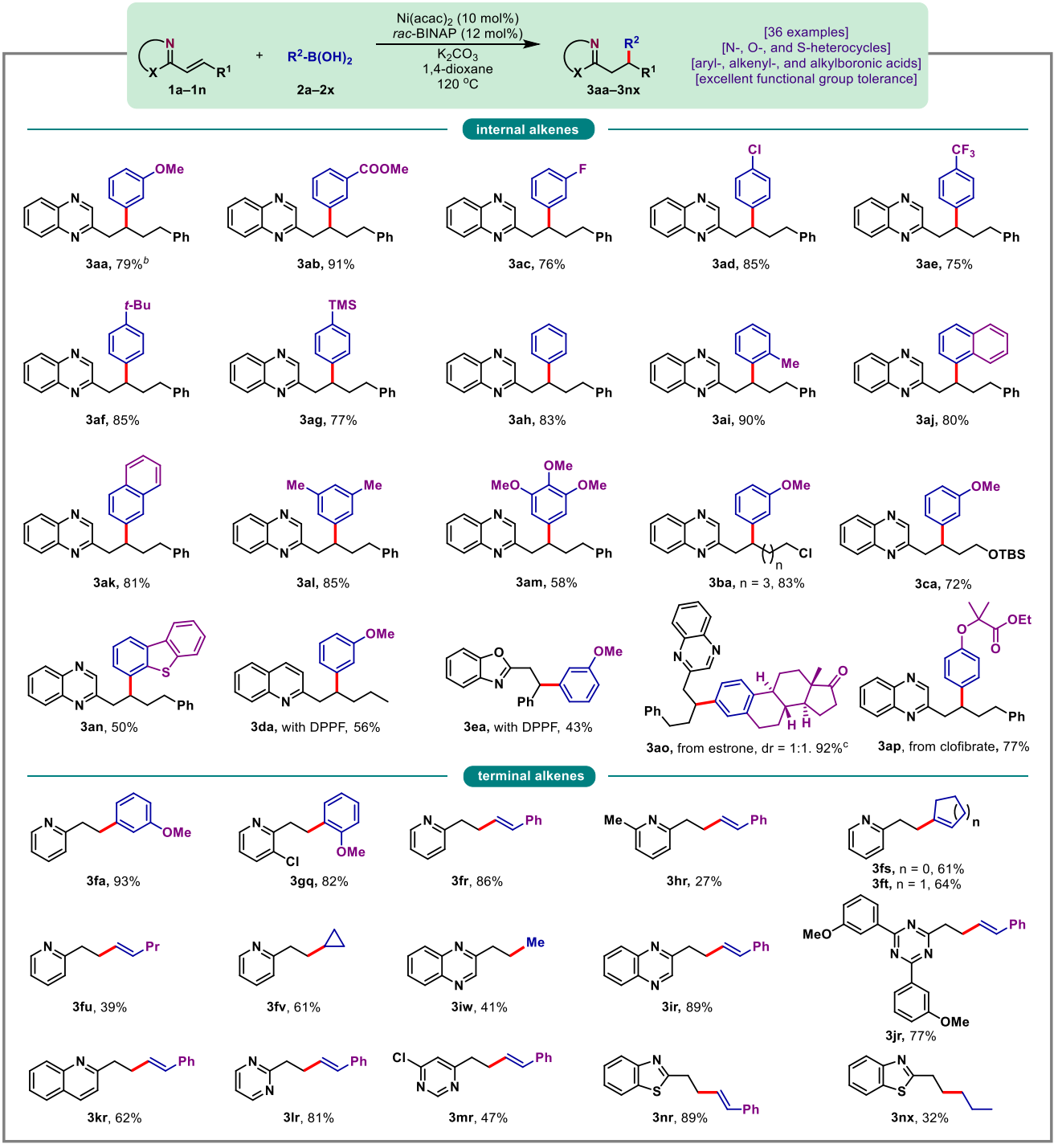
Scheme 2. Substrate Scope of Boronic Acids and Alkenylazaarenes[a]



[a]Reactions were performed using **1** (0.2 mmol, 1.0 equiv), **2** (0.6 mmol, 3.0 equiv), $Ni(acac)_2$ (10 mol %), *rac*-BINAP or DPPF (12 mol %) and dioxane (2.0 mL) under an Ar atmosphere, 120 °C, 6 h. [b]Yield of isolated products **3**. [c]dr value was determined by ^1H NMR analysis.

To demonstrate the practicality and robustness of this method, gram-scale experiments of both internal and terminal alkenes were conducted, respectively. Under standard conditions, an acceptable yield was observed for the internal alkene **1a** (Scheme 4a). To our delight, as for the terminal alkene **1f**, we were able to lower the loading of both the boronic acid and the catalyst with only a slight decrease in the yield (Scheme 4b). Then, several transformations of the products were elaborated. The addition product **3aa** could be oxidized to bis(N-oxide) compound **4aa** by *m*-chloroperbenzoic acid (Scheme 4c)[10] or reduced to hydroquinoxaline **5aa** by BH_3·THF in excellent yield (Scheme 4d).[11] With the assistance of the *n*-BuLi, we realized the aldol-type addition of **3fr** with PhCHO in high yield with moderate diastereoselectivity (Scheme 4e).[12] Next, the oxidative dibromination of the olefin readily occurred to afford **5fr** in the presence of HBr and dimethyl sulfoxide (DMSO) (Scheme 4f).[13] Additionally, the Simmons–Smith reaction was carried out on the addition

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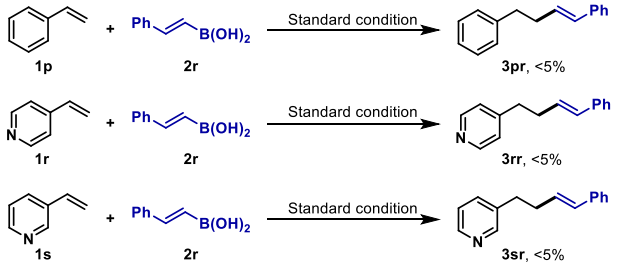
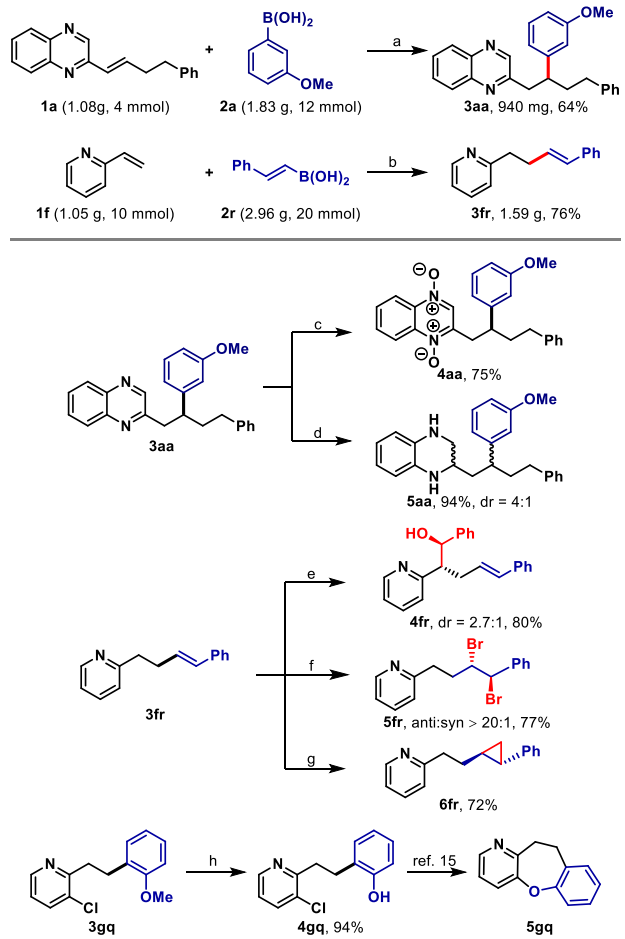
Scheme 3. Investigations of the Function of the N Atom in the Heterocycles



Scheme 4. Gram-Scale Experiments and Several Transformations of the Products[a]



[a](a) **1a** (1.04 g, 4 mmol, 1.0 equiv), **2a** (1.83 g, 12 mmol, 3.0 equiv), $Ni(acac)_2$ (10 mol %), *rac*-BINAP (12 mol %), and dioxane (40.0 mL), 120 °C, 6 h. (b) **1f** (1.05 g, 10 mmol, 1.0 equiv), **2r** (2.96 g, 20 mmol, 3.0 equiv), $Ni(acac)_2$ (5 mol %), *rac*-BINAP (6 mol %), and dioxane (40.0 mL), 120 °C, 6 h. (c) **3aa** (0.2 mmol, 1 equiv), *m*-CPBA (0.6 mmol, 3 equiv), $CHCl_3$, rt, 12 h. (d) **3aa** (0.1 mmol, 1 equiv), BH_3·THF (0.25 mmol, 2.5 equiv), rt. (e) **3fr** (0.2 mmol, 1 equiv), *n*-BuLi (0.25 mmol, 1.25 equiv), PhCHO (0.24 mmol, 1.2 equiv), THF (2 mL), −40 °C, 2 h. (f) **3fr** (0.2 mmol, 1 equiv), DMSO (0.24 mmol, 1.2 equiv), aqueous HBr (48%, 0.72 mmol, 3.6 equiv), ethyl acetate (1 mL), 60 °C, 0.5 h. (g) **3fr** (0.2 mmol, 1 equiv), Et_2Zn (0.8 mmol, 4 equiv), TFA (0.4 mmol, 2 equiv), CH_2I_2 (0.4 mmol, 2 equiv), CH_2Cl_2 (2 mL), 0 °C, 3 h. (h) **3gq** (0.1 mmol, 1 equiv), aqueous HBr (48%, 2 mL), 120 °C, 2 h. *m*-CPBA = *m*-chloroperbenzoic acid, DMSO = dimethyl sulfoxide, TFA = trifluoracetic acid.

product to form the cyclopropane **6fr** in a stereospecific fashion (Scheme 4g).[14] Lastly, the deprotection of phenylmethyl ether **3gp** gave the phenol **4gp** (Scheme 4h), which can be used to prepare the dihydrodibenzoxepine **5gp** through a Pd-catalyzed C−O cross-coupling reaction.[15]

In conclusion, we developed an efficient nickel(II)-catalyzed addition of aryl-, alkenyl-, and alkylboronic acids to alkenylazaarenes. This reaction exhibits high efficiency, a broad substrate scope, and good functional group compatibility and is an excellent complement to the previously reported Rh- and Co-catalyzed system. Then, the products can be further transformed to many useful building blocks. Finally, the preliminary studies suggest that the adjacent N atom of the heterocycles is essential for the high reactivity. Further studies on the Ni(II)-catalyzed enantioselective addition of boronic acids to alkenylazaarenes are ongoing in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c01425.

Experimental procedures and spectra for all new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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