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Nickel-Catalyzed Domino Heck Cyclization/Suzuki Coupling for the Synthesis of 3,3-Disubstituted Oxindoles

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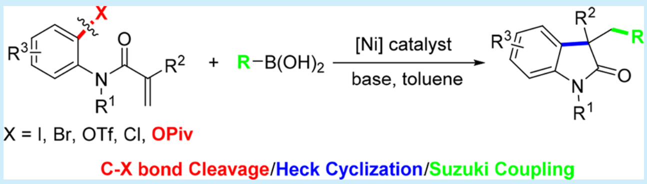
Supporting Information

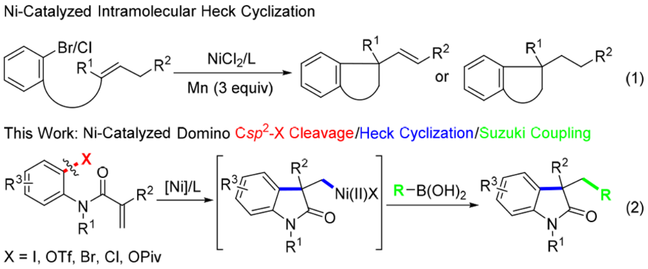
ABSTRACT: The first nickel-catalyzed domino Heck cyclization/Suzuki coupling reaction for the synthesis of 3,3-disubstituted oxindoles bearing quaternary all-carbon centers is reported. A wide range of electrophiles, such as aryl iodides, bromides, triflates, and chlorides, are all compatible with the reaction conditions. Moreover, cheap aryl esters, which undergo catalytic C−O bond cleavage, could also be employed as electrophiles. The approach shows good yields and broad scope, complementing a more practical and sustainable alternative to the conventional palladium-based analogues.



The intramolecular Heck cyclization has been intensely investigated for decades and has been utilized for the synthesis of a range of natural products and pharmaceutically active compounds bearing quaternary carbon centers.[1] Although palladium-based catalysts are commonly used in this reaction,[2,3] considerable efforts have been made to replace this highly expensive and scarce metal with nickel, which is a low-cost, nontoxic, abundant, and sustainable element.[4] In addition, nickel is a facile oxidative addition on account of its smaller size, allowing for the use of much less reactive electrophiles, such as aryl chlorides, carbamates, esters, ethers, and so on.[5] Nickel catalyzed Heck reaction was known about as early as the 1980s;[6] however, Heck reactions with Ni are still relatively rare and challenging.[7,8]

Recently, nickel-catalyzed intramolecular Heck cyclization of aryl halides for construction of quaternary carbon centers has been reported (Scheme 1, eq 1).[9] In connection with our interest in transition-metal-catalyzed domino processes,[10] we envisaged that the in situ generated σ-alkyl-Ni(II) intermediate could directly undergo Suzuki coupling with boronic acids without an external stoichiometric amount of reducing agent.[11] None have yet achieved the generality, economy, and efficiency of this domino process with catalytic amount of nickel.[12]

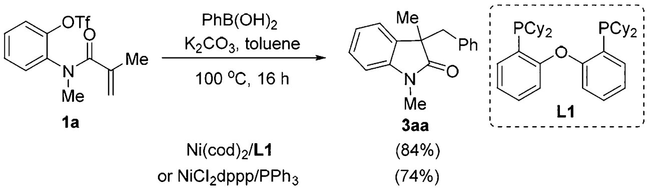
Scheme 1. Nickel-Catalyzed Domino Heck Cyclization Reaction



Herein, we report our investigations of the first example of nickel-catalyzed cyclizative cross coupling of aryl halides and pivalates with boronic acids for the synthesis of 3,3-disubstituted oxindoles through a domino Csp^2−X cleavage/Heck cyclization/Suzuki coupling process (Scheme 1, eq 2).

Our study commenced with the reaction of 2-(*N*-methylmethacrylamido)phenyl trifluoromethanesulfonate **1a** and phenylboronic acid **2a** (for the optimization of reaction conditions, see Table S1). In the presence of Ni(cod)$_2$ (10 mol %), **L1** (20 mol %), and K$_2$CO$_3$ (2 equiv), the desired oxindole **3aa** could be isolated in 84% yield in toluene at 100 °C. When an air-stable NiCl$_2$dppp (10 mol %) complex and PPh$_3$ (20 mol %) were employed in place of Ni(cod)$_2$ and the ligand, 74% isolated yield of **3aa** could also be obtained (Scheme 2).

Scheme 2. Optimized Conditions for Domino Heck Cyclization/Suzuki Coupling of Aryl Triflate



With the optimized conditions in hand (Scheme 2), reaction scope with regard to the structure of boronic acids was first explored. Both electron-rich and electron-poor *p*-aryl boronic acids could react with **1a** to give the target products **3aa**−**ag** with good yields (Scheme 3). Notably, functional groups (Cl, OMe) were found to be compatible with this transformation, providing an opportunity to further functionalize these obtained products. The steric hindrance did not affect the

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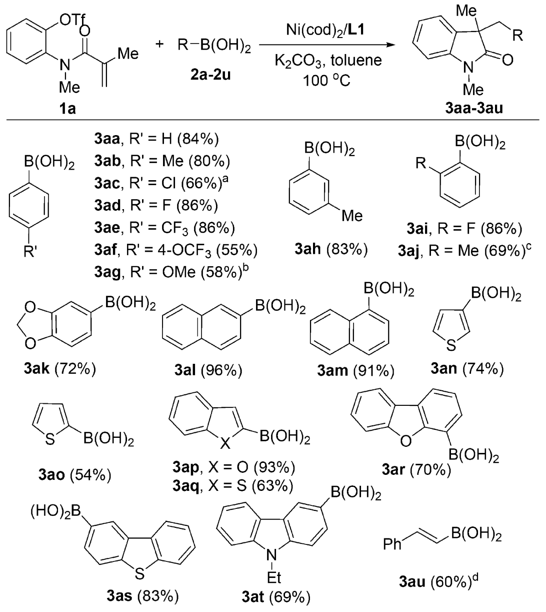
Scheme 3. Scope for Boronic Acids

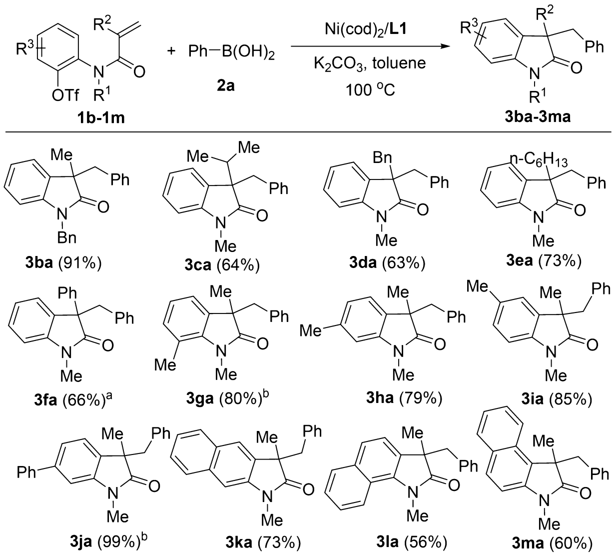


[a]NiCl$_2$dppp (10 mol %), PPh$_3$ (20 mol %), and **2c** (3 equiv) at 90 °C. [b]NiCl$_2$dppp (10 mol %), PPh$_3$ (20 mol %), and **2g** (5 equiv) at 90 °C. [c]NiCl$_2$dppp (10 mol %) and PPh$_3$ (20 mol %). [d]Reaction performed at 120 °C.

efficiency, and all *meta-* and *ortho*-substituted aryl boronic acids (**3ah**−**aj**) showed comparable reactivities. The fluoro (**3ad** and **3ai**), trifluoromethyl (**3ae**), trifluoromethoxyl (**3af**), and ketal (**3ak**) substituents were all tolerated. Both 2- and 1-naphthylboronic acids could be employed to provide **3al** and **3am** in 96% and 91% yield, respectively. Moreover, substrate scope toward (hetero)aryl boronic acids was also screened. As shown by the formation of **3an**−**at**, thiophene, benzofuran, benzothiophene, dibenzofuran, dibenzothiophene, and carbazole heterocycles were transformed into the desired products successfully with satisfied yields. These results demonstrate the potential utility of this methodology for the synthesis of medicinally relevant compounds are feasible.[13] Excitingly, the trans-*β*-styreneboronic acid **2u** could also be efficiently transformed into the desired product **3au** in 60% yield at 120 °C.

Subsequently, the scope of the current method with respect to aryl triflates was evaluated and found to proceed smoothly in all the cases (Scheme 4). The domino reaction between *N*-benzyl triflate **1b** and boronic acid **2a** proceeded efficiently to provide **3ba** in 91% yield. As the *N*-benzyl is easily removed, it constitutes a route to *N*-H oxindoles. The influence of the C*α* substituents (R^2) of the acrylamide double bond on the reaction outcome was tested. Isopropyl, benzyl, and *n*-hexyl substituents were readily transformed into the desired oxindoles **3ca**−**ea** in good yields. The phenyl at C*α* position of the double bond was also found to be effectively leading to the desired oxindole **3fa** in 66% yield. Finally, the influence of substitution pattern in the aniline part was evaluated. *Para-*, *meta-*, or *ortho*-substituted aryl triflates were well tolerated, affording the corresponding oxindoles **3ga**−**ja** in 79−99% yields. More importantly, the reaction was successfully applied to the synthesis of 5,6-benzoxindole **3ka**, 6,7-benzoxindole **3la**, and 4,5-benzoxindole **3ma**.

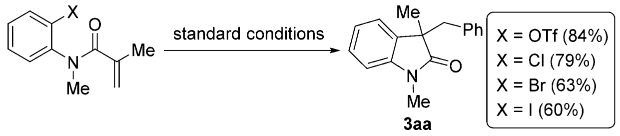
Scheme 4. Scope for Aryl Triflates



[a]**2a** (3 equiv). [b]The reaction was run using NiCl$_2$dppp (10 mol %) and PPh$_3$ (20 mol %).

The effects of electrophiles were investigated subsequently (Scheme 5). The aryl triflate, chloride (**4**), bromide (**5**), and iodide (**6**) are competent electrophiles to afford the desired product **3aa** in 60−84% yields, thus extending the scope compared to Desrosiers' report.[9b]

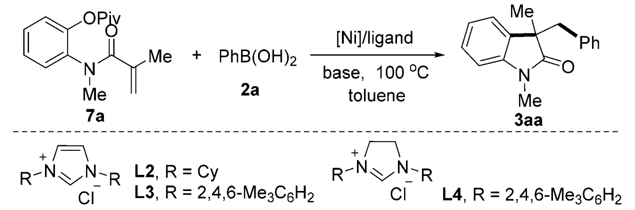
Scheme 5. Effects of the Electrophiles



Encouraged by these results, we decided to further explore the scope of aryl electrophiles. In the past decade, C−O electrophiles have received considerable attention in the cross-coupling reactions due to their lack of toxicity, stability and readily availability compared with commonly used aryl halides. Among the C−O electrophiles, aryl sulfonates have been widely employed due to their high reactivities. Nevertheless, relatively high price and producing of stoichiometric amounts of sulfur-containing waste have limited the application in chemical industry. Use aryl esters as coupling partners could be a synthetically ideal route that is cheaper and more environmentally friendly. However, employing aryl esters as electrophiles via catalytic C−O bond-cleavage is still a formidable challenge due to the requirement of higher activation energy to cleavage the inert Csp^2−O bond.[4,5] Despite the progress made, the overwhelming majority of C−O bond activation reactions are still restricted to the direct cross-coupling for single C−C bond or C-heteroatom bond formation.[4,5,8,14] Indeed, the development of catalytic domino reaction initiated by C−O bond activation, involving multibond formation in one pot, still remains an elusive task.

2-(*N*-methylmethacrylamido)phenyl pivalates **7a** and **2a** were chosen as model substrates to evaluate the feasibility of reaction design (Table 1). Using previously optimized conditions (Scheme 2), however, failed to furnish any of the desired product (Table 1, entries 1 and 2). The choice of a

Table 1. Optimization of the Reaction Conditions[a]



entry	catalyst	ligand	base (equiv)	yield (%) of **3aa**
1	NiCl$_2$dppp	PPh$_3$	K$_2$CO$_3$ (2)	0
2	Ni(cod)$_2$	**L1**	K$_2$CO$_3$ (2)	0
3	Ni(cod)$_2$	**L1**	*t*-BuOLi (4)	0
4	Ni(cod)$_2$	**L1**	*t*-BuONa (4)	15
5	Ni(cod)$_2$	**L1**	*t*-BuOK (4)	0
6	Ni(cod)$_2$	PCy$_3$	*t*-BuONa (4)	11
7	Ni(cod)$_2$	**L2**	*t*-BuONa (4)	trace
8	Ni(cod)$_2$	**L3**	*t*-BuONa (4)	11
9	Ni(cod)$_2$	**L4**	*t*-BuONa (4)	13
10	Ni(cod)$_2$	PPh$_3$	*t*-BuONa (4)	25
11	NiCl$_2$	PPh$_3$	*t*-BuONa (4)	0
12	NiBr$_2$	PPh$_3$	*t*-BuONa (4)	0
13	Ni(acac)$_2$	PPh$_3$	*t*-BuONa (4)	0
14	Ni(PPh$_3$)$_2$Cl$_2$	PPh$_3$	*t*-BuONa (4)	0
15	Ni(PPh$_3$)$_4$	PPh$_3$	*t*-BuONa (4)	41
16	Ni(PPh$_3$)$_4$[b]	PPh$_3$	*t*-BuONa (4)	49
17	Ni(PPh$_3$)$_4$[b]	PPh$_3$	*t*-BuONa (2.5)	56
18	**Ni(PPh$_3$)$_4$**[c]	**PPh$_3$**	***t*-BuONa (2.5)**	**70**

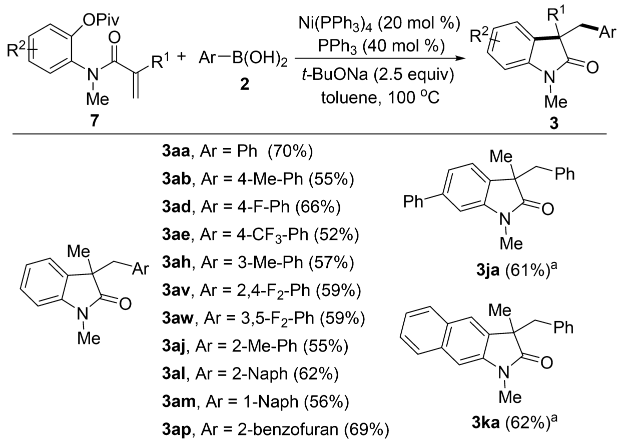
[a] Reaction conditions: **7a** (0.1 mmol), **2a** (0.25 mmol), [Ni] (10 mol %), ligand (20 mol %), toluene (2.0 mL), sealed tube, 100 °C, 16 h. [b] Ni(PPh$_3$)$_4$ (15 mol %) and PPh$_3$ (30 mol %). [c] Ni(PPh$_3$)$_4$ (20 mol %) and PPh$_3$ (40 mol %).

proper base was crucial to this reaction. *t*-BuONa (4 equiv) provided promising results (Table 1, entry 4), and no product took place with *t*-BuOLi or *t*-BuOK. Subsequently, a survey of the ligands was performed (Table 1, entries 6–10). To our delight, the desired product **3aa** could be obtained in 25% yield when PPh$_3$ was employed (Table 1, entry 10). Various nickel complexes were tested (Table 1, entries 11–15), and Ni(PPh$_3$)$_4$ was found to be the most effective, providing **3aa** in 41% yield (Table 1, entry 15). Further screening the reaction conditions (Table 1, entries 16–18) allowed us to define the following optimized conditions: Ni(PPh$_3$)$_4$ (20 mol %)/PPh$_3$ (40 mol %) as catalyst ,and *t*-BuONa (2 equiv) as a base in toluene at 100 °C. Under these conditions, oxindole **3aa** was isolated in 70% yield (Table 1, entry 18).

With the optimized conditions in hand (Table 1, entry 18), the substrate scope and generality of our Ni-catalyzed domino C–O bond activation/Heck cyclization/Suzuki coupling reaction was examined (Scheme 6). Once again, various substituted boronic acids and aryl pivalates all underwent the domino process without event, and the desired oxindoles **3** were obtained in moderate to good yields. These results are noteworthy, taking into account the higher activation energy required for Csp^2–OPiv cleavage in the electron-rich *o*-aminoaryl pivalates **7**. Previous reports on Ni-catalyzed cross-couplings of electron-rich phenyl pivalates usually resulted in a reduced yield.[7j,14] Aryl esters are unreactive with palladium catalysis, which clearly manifests the superiority of the current synthetic method.

Since Ni-catalyzed enantioselective intramolecular (reductive) Heck cyclization reactions were reported very recently,[9c,d] we decided to screen a series of chiral ligands in an attempt to render the reaction asymmetric (for details, see Table S2). Unfortunately, all attempts with aryl triflate **1a** afforded racemic product. When aryl chloride **4** was treated with phosphoramidite ligand, oxindole **3aa** was obtained in 17% yield with 64:36 er. Though the result is far from satisfactory, it might suggest a Ni(0)/Ni(II) or Ni(I)/Ni(III) catalytic cycle instead of a radical-induced pathway.

Scheme 6. Scope for Domino Csp^2–O Bond Cleavage/Heck Cyclization/Suzuki Coupling Reaction



[a] **2a** (3 equiv).

In summary, an efficient strategy for the first nickel-catalyzed domino Heck cyclization/Suzuki coupling for the construction of 3,3-disubstituted oxindoles bearing quaternary carbon centers has been presented. The cheap and traditionally unreactive aryl esters via catalytic Csp^2–O bond cleavage could also be employed in this domino process. This protocol is distinguished by its wide substrate scope and diverse set of substitution patterns, representing a more practical and sustainable alternative to the conventional palladium-catalyzed Heck cyclization reaction. Further investigations on the asymmetric version and detailed mechanistic studies of this transformation are currently underway in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b03713.

Experimental procedures, characterization data, and spectra data (PDF)

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Notes

The authors declare no competing financial interest.

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