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Ni-Catalyzed C–F Bond Functionalization of Unactivated Aryl Fluorides and Corresponding Coupling with Oxazoles

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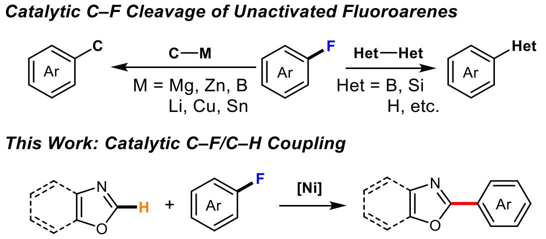
Abstract. A Ni-catalyzed C–F bond functionalization of unactivated aryl fluorides with oxazoles as coupling partners was developed. Various arylated oxazoles could be obtained in moderate to good yields in the presence of Ni(cod)$_2$/IMes catalytic system. A rapid synthesis of natural product texaline was also demonstrated using this protocol. This transformation could be potentially utilized in regioselective introduction of an oxazole group in late-stage synthetic applications.

Keywords: nickel catalysis; C–F functionalization; C–H functionalization; oxazoles; cross-coupling

Fluorine-containing organic compounds are widely found in a broad range of areas such as medicine, biochemistry, catalysis, material science, etc.[1] In the past decade, great efforts have been devoted to developing protocols that could efficiently construct carbon-fluorine (C–F) bond.[2] In light of the growing importance of C–F bond formation, C–F bond functionalization has also attracted a great deal of interest.[3] The availability of fluoroarenes has been significantly expanded with the recent advances in fluorination methods while the inert nature of C–F bond makes C–F bond functionalization a useful protocol in the late-stage synthesis of complex organic compounds.

Owing to the fact that C–F bond is a strong and inert chemical bond in organic compounds, carbon-carbon and carbon-heteroatom bond formation via C–F bond cleavage is always challenging. Since the pioneering work on the Kumada-Corriu cross-coupling involving aryl fluorides,[4] various transition-metal-catalyzed cross-coupling reactions involving aryl fluorides and stoichiometric organometallic reagents have been developed (Scheme 1).[5] Additionally, catalytic hydrodefluorination,[6] borylation,[7] silylation,[8] and other carbon-heteroatom bond formations[9] have been reported as well (Scheme 1). Although significant progress has been made in the development of aromatic C–H bond arylation,[10] rare examples have been reported on directly coupling both C–F and C–H bonds.[11] Herein, we communicate a Ni-catalyzed C–F/C–H cross-coupling involving the C–F bond cleavage of unactivated aryl fluorides and C–H bond functionalization of oxazoles (Scheme 1). This novel C–F/C–H coupling would offer a new strategy for the synthesis of complex 2-aryl oxazole derivatives wherein the fluoro group could be utilized as an oxazole equivalent.[12]



Scheme 1. Catalytic C–F/C–H coupling.

We began by investigating the arylation of benzoxazole **1a** with 4-fluoro-1,1'-biphenyl **2a** in the presence of nickel catalyst. After extensive screening, the best result was obtained when the reaction was conducted under the conditions consisting of Ni(cod)$_2$, IMes•HCl (1,3-bis(2,4,6-trimethylphenyl) imidazolium chloride) and NaO^{t}Bu in ^{c}hexane at 160 °C for 16 h affording the arylated product **3a** in 87% GC yield and 80% isolated yield (Table 1).

The effect of alteration to the "standard conditions" is listed in Table 1. Nickel catalyst is essential for this reaction as no arylation occurred without Ni(cod)$_2$. The use of Ni(acac)$_2$ and NiCl$_2$ as catalyst precursors resulted in few or no products indicating that Ni(0)

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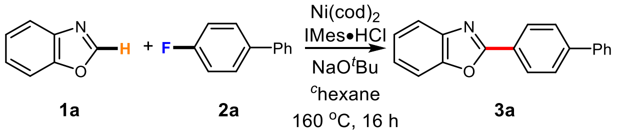
species might be required to initiate the reaction. Low yield of **3a** was obtained with the rest **2a** recovered when 10 mol% Ni(cod)$_2$ was employed. Elongation of reaction time gave similar result suggesting the deactivation of nickel catalyst. Both N-heterocyclic carbene ligands and phosphine ligands showed efficiency for this transformation, although these ligands (IPr•HCl, ICy•HCl, PPh$_3$, PCy$_3$) were found to be less effective. As to the base effect, other alkaline metal alkoxides like KO^{t}Bu led to low yield while K$_3$PO$_4$ and Cs$_2$CO$_3$ afforded no product. This arylation occurred smoothly in non-polar solvents such as toluene (70% yield) while no product was obtained using other solvents like DMF or DME. Due to the strong and inert nature of C–F bond, lowering the reaction temperature led to decreased yield.

Table 1. Ni-catalyzed C–H arylation of **1a** with **2a**: effects of reaction parameters.

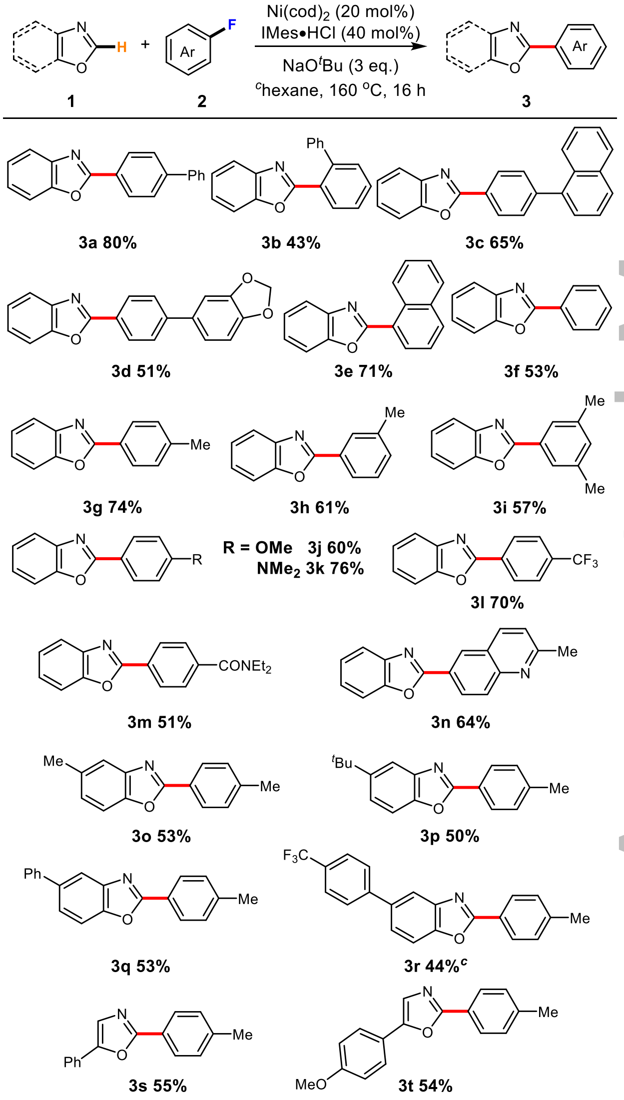


Entry	Variation from “standard conditions”[a]	Yield[%][b]
1	none	87(80)[c]
2	No Ni(cod)$_2$	0
3	Ni(acac)$_2$, instead of Ni(cod)$_2$	18
4	NiCl$_2$, instead of Ni(cod)$_2$	0
5[d]	10 mol% Ni(cod)$_2$	39
6[e]	10 mol% Ni(cod)$_2$	42
7	IPr•HCl, instead of IMes•HCl	64
8	ICy•HCl, instead of IMes•HCl	9
9	PPh$_3$, instead of IMes•HCl	12
10	PCy$_3$, instead of IMes•HCl	57
11	KO^{t}Bu, instead of NaO^{t}Bu	29
12	K$_3$PO$_4$, instead of NaO^{t}Bu	0
13	Cs$_2$CO$_3$, instead of NaO^{t}Bu	0
14	Toluene, instead of ^{c}hexane	70
15	DME, instead of ^{c}hexane	0
16	DMF, instead of ^{c}hexane	0
17	140 °C, instead of 160 °C	73

[a] Standard reaction conditions: **1a** (0.4 mmol), **2a** (0.2 mmol), Ni(cod)$_2$ (0.04 mmol), IMes•HCl (0.08 mmol), NaO^{t}Bu (0.6 mmol), ^{c}hexane (1.0 mL), 160 °C, 16 h. [b] The yield was determined by GC, calibrated using naphthalene as internal standard. [c] Isolated yield. [d] 20 mol% IMes•HCl. [e] 20 mol% IMes•HCl, 36 h.

With the optimized reaction conditions in hand, a variety of aryl fluorides **1** were subjected to this C–F/C–H coupling reaction (Table 2). Biaryl fluorides worked well under the standard conditions affording the desired product in moderate to good yields (**3a**, **3b**). In a similar manner, 1-fluoronaphthalene also gave the desired product (**3c**) in good yield. Remarkably, the reactivity of regular monofluoroarenes was comparable to π-extended systems. Fluorobenzene afforded the product in a satisfying yield (**3f**). Aryl fluorides substituted with a methyl group in meta and para position afforded the corresponding arylated products **3g** and **3h** in 74% and 61% yield, respectively. In general, substrates bearing both electron-donating substituents (**3j**, **3k**) and electron-withdrawing substituents (**3l**) worked well under the standard conditions. Amide group could also be tolerated under standard conditions. Unfortunately, halogen groups like chloride and base sensitive groups like ester and aldehyde could not be tolerated. To our delight, heteroaryl fluoride **2n** could also react well to form the biaryl product in 64% yield. Furthermore, other heteroarenes were tested for this C–H arylation as well. Substituted benzoxazoles and oxazoles were also suitable substrates giving the desired products in moderate yields. Other azoles and acidic arenes like polyfluoroarenes were tested, in which only N-Me benzimidazole afforded trace product while other azoles and polyfluoroarenes gave no product.

Table 2. Ni-catalytic C–F/C–H coupling of unactivated aryl fluorides with oxazoles.[a,b]



[a] Reaction conditions: **1a** (0.4 mmol), **2a** (0.2 mmol), Ni(cod)$_2$ (0.04 mmol), IMes•HCl (0.08 mmol), NaO^{t}Bu (0.6 mmol), ^{c}hexane (1.0 mL), 160 °C, 16 h. [b] Isolated yield. [c] **1r** (0.3 mmol) was used.

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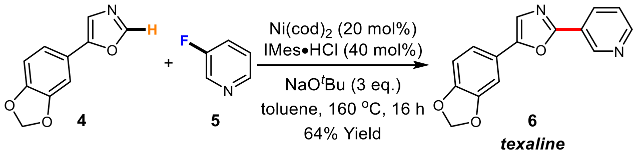
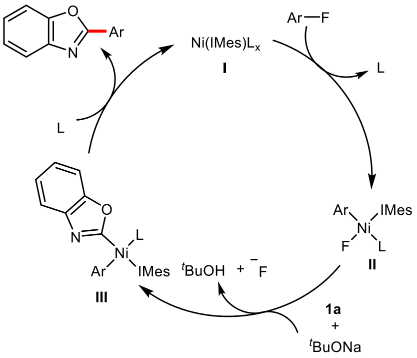
To demonstrate the synthetic utility of the present C–F/C–H coupling, a rapid synthesis of texaline[11i] (important natural product with antitubercular activity) was examined. The coupling reaction of 5-(1,3-benzodioxol-5-yl)-1,3-oxazole **4** with 3-fluoropyridine **5** underwent smoothly furnishing texaline **6** in 64% yield using toluene as solvent (Scheme 2).



Scheme 2. Synthesis of texaline.

On the basis of previous reports,[13] we proposed a mechanism for this C–F/C–H coupling reaction (Scheme 3). Aryl fluoride first coordinates to the Ni(0) species **I** and the subsequent oxidative addition of aryl fluoride to the Ni(0) center affords the Ni(II) intermediate **II**. In the presence of a base, benzoxazole anion is generated by the deprotonation of benzoxazole, which could transmetallate with intermediate **II** to form the diaryl intermediate **III**. Finally, the reductive elimination step furnishes the product and regenerates the active Ni(0) species.



Scheme 3. Proposed mechanism.

In summary, we have developed a Ni-catalyzed C–F bond functionalization of unactivated aryl fluorides and C–H bond functionalization of oxazoles. A variety of functional groups were tolerated and heteroaryl fluorides were suitable substrates. This C–F/C–H cross-coupling transformation could be potentially utilized for regioselective introduction of an oxazole group in a late stage synthesis. Additional exploration of the substrate scope and mechanistic studies are currently underway and will be reported in the due course.

Experimental Section

A 25-mL sealed pressure-resistant tube equipped with a magnetic stirring bar was dried with a heat-gun under reduced pressure and filled with nitrogen after cooling to room temperature. After adding aryl fluorides **1** (0.2 mmol), the tube was introduced inside a nitrogen-atmosphere glovebox. In the glovebox, Ni(cod)$_2$ (11.2 mg, 0.04 mmol), IMes·HCl (27.2 mg 0.08 mmol) and sodium *tert*-butoxide (57.7 mg, 0.6 mmol) were added to the tube, which was sealed with O-ring tap and then taken out of the glovebox. Then, azoles (0.4 mmol) and cyclohexane (1 mL) were added to the vessel under a nitrogen atmosphere. The vessel was heated at 160 °C for 16 h in a heating module with stirring. After cooling the reaction mixture to room temperature, the mixture was concentrated and directly purified by preparative thin-layer chromatography (PTLC; petroleum ether /ethyl acetate as the eluent) to afford the product **3**.

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Ni-Catalyzed C–F Bond Functionalization of Unactivated Aryl Fluorides and Corresponding Coupling with Oxazoles

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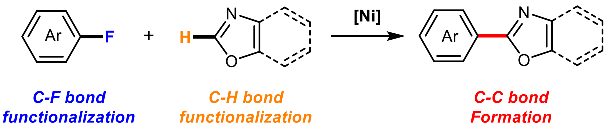

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