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Nickel-catalyzed reductive cross-coupling of polyfluoroarenes with alkyl electrophiles by site-selective C–F bond activation

Longlong Xi[a], Liting Du[b], Zhuangzhi Shi[a,*]

[a] *State Key Laboratory of Coordination Chemistry, Chemistry and Biomedicine Innovation Center (ChemBIC), School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, China*

[b] *Advanced Analysis and Testing Center, Nanjing Forestry University, Nanjing 210037, China*

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ABSTRACT

A nickel-catalyzed reductive cross-coupling reactions between polyfluoroarenes and alkyl electrophiles is reported to access substituted fluoroarenes through chelation-assisted C–F activation. Diverse primary and secondary alkyl (*pseudo*)halides can be employed to couple with polyfluoroarenes, showing excellent regioselectivity. Furthermore, the nickel-catalyzed asymmetric cross-coupling of polyfluoroarenes with racemic alkyl halides is preliminarily explored. In addition, the practicability of the title transformation is also demonstrated by total synthesis of losmapimod and an analog as key steps. The developed method exhibits many advantages, including economic catalytic systems, commercially available alkyl electrophiles, and lack of sensitive organometallic reagents.

Aryl fluoride is among the most versatile structural motifs in pharmaceuticals and biologically active molecules [1–5]. During the past decades, extensive efforts have been made by chemists toward the exploration of more general and practical methods to build them. Compared to the common fluorination process [6–12], defluorinative functionalization of polyfluoroarenes has emerged as a promising way to generate unique fluorinated compounds [13–38]. An illustrative case is described in Fig. 1a, wherein losmapimod **(I)**, as a selective MAPK (mitogen-activated protein kinases) inhibitor, was accessed by de novo synthesis from compound **II** through a palladium-catalyzed Suzuki-Miyaura coupling reaction [39]. We envisioned that late-stage defluoroalkylation of a difluoroarene III might provide an alternative route to molecule I, especially simplifying the route to a series of analogs bearing diverse alkyl substituents, for high-throughput screening in drug discovery.

Typically, preformed alkyl organometallic reagents (Zn, Mg, Li) need to be employed in the defluoroalkylation of polyfluoroarenes, leading to inconvenient operation and limited substrate scope [40–43]. Recent progress has provided new defluoroalkylative methods from sustainable chemical feedstocks [44,45]. For example, Xiong *et al.* developed Cu-catalyzed defluoroalkylation of polyfluoroarenes using alkenes and silanes (Fig. 1b, left) [46]. The Ritter group also uncovered the related defluorinative alkylation by decarboxylation of aliphatic carboxylic acids under photoredox conditions (Fig. 1b, right) [47]. Controlled by electronic and steric effects, such reactions exhibit good para-selectivity in symmetric polyfluoroarenes, but the use of unsymmetrical substrates leads to low regioselectivity.

Transition metal-catalyzed reductive cross-coupling reactions, which employ two electrophiles with a terminal reductant, have emerged as inspiring methods for the construction of carbon−carbon bonds [48–55]. Aryl [56–64] or vinyl [65–67] halides, including Cl, Br, I and pseudohalides such as OTs and OTf, have been used for the synthesis of alkylated arenes with alkyl electrophiles in the presence of nickel catalysis (Fig. 1c) [68–74]. Given the high value of fluorinated arenes, the direct use of polyfluoroarenes in reductive cross-coupling through C–F activation is appealing [75–77]. Here, we showcase the first example of site-selective defluoroalkylation of polyfluoroarenes with bench-stable and readily available alkyl (*pseudo*)halides, avoiding the pregeneration of air- and moisture-sensitive organometallic reagents (Fig. 1d). Controlled by the chelation effect, diverse polyfluoroarenes with coordinating functional groups direct nickel to activate proximal C–F bonds *via* cyclometallated intermediates, showing an excellent level of ortho-selectivity [78–83].

We began our work by monitoring the reactivity of polyfluoroarene **1a** and alkyl bromide **2a** (Table 1). After systematic screening, the reaction was found to be facile with $NiBr_2$(dme) (10 mol%), X-phos (12 mol%), Zn^0 (2.0 equiv.) as the stoichiometric reductant, NaI (2.0 equiv.) as the additive in DMA after 2 h at 50 °C, affording desired defluoroalkylation product **3aa** in 80% yield (en-

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[*] Corresponding author.
E-mail address: shiz@nju.edu.cn (Z. Shi).

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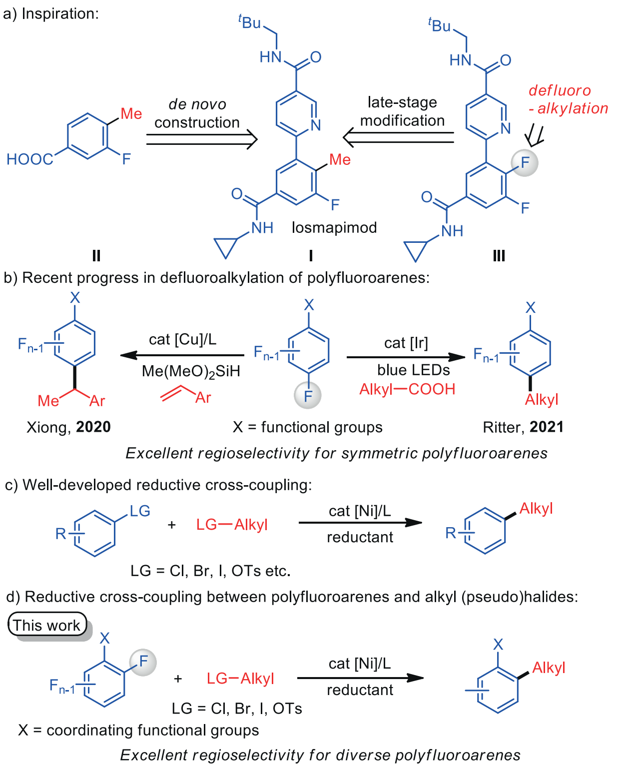
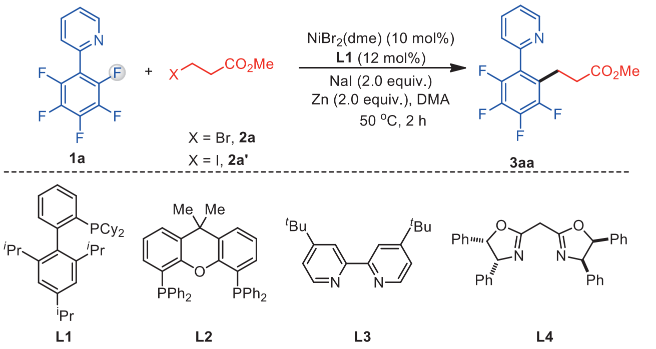


Fig. 1. Nickel-catalyzed defluoroalkylation of polyfluoroarenes with alkyl electrophiles through reductive cross-coupling reactions.

Table 1
Reaction optimization.[a]



Entry	Variation from the standard conditions	Yield of **3aa** (%)[b]
1	none	80 (74)[c]
2	Using **L2** instead of **L1**	7
3	Using **L3** instead of **L1**	60
4	Using **L4** instead of **L1**	71
5	Using Mn instead of Zn	0
6	Shortening the reaction time to 45 min	76
7	Extending the reaction time to 12 h	62
8	At room temperature	73
9	Without NaI	trace
10	Using **2a'** instead of **2a**	82 (75)[c]
11	Using **2a'** instead of **2a**, without NaI	33
12	Without $NiBr_2(dme)$	0

[a] Reaction conditions: 10 mol% $NiBr_2(dme)$, 12 mol% ligand, NaI (0.40 mmol), reductant (0.40 mmol), **1a** (0.20 mmol), **2a** or **2a'** (0.40 mmol), solvent (1.0 mL), 50 °C, 2 h.

[b] Yields were determined by GC analysis.

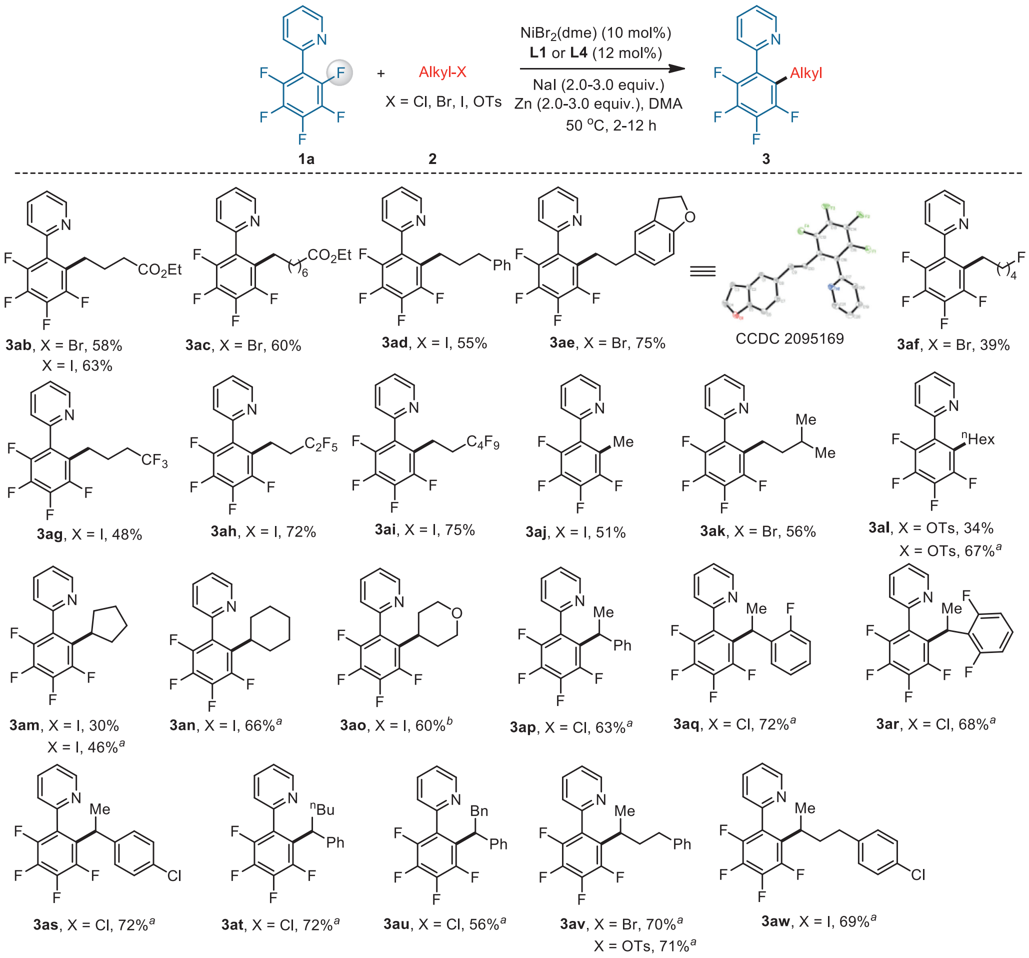
[c] Isolated yield after chromatography.

try 1). Diphosphine ligands such as xantphos (**L2**) greatly lowered the selectivity (entry 2). Several bidentate nitrogen ligands were also investigated; *tert*-butyl bipyridine **L3** delivered product **3aa** in 60% yield (entry 3), and a bis(oxazoline) ligand **L4** could improve the yield to 71% (entry 4). The use of Zn^0 can accelerate the cross-coupling process, and other reductants, such as Mn^0, were not effective (entry 5). Notably, shortening reaction time to 45 min maintained a good reactivity (entry 6), and extending the reaction time to 12 h led to the slow decomposition of product **3aa** (entry 7). In addition, conducting the reaction at room temperature resulted in a slightly lower yield (entry 8). The reaction completely failed in the absence of NaI, which was reported to be a key additive in reductive cross-couplings *via* acceleration of electron transfer between metal reductant and nickel catalyst (entry 9) [84–87]. In addition, treatment of alkyl iodide **2a'** in the reaction showed slightly higher reactivity, affording the product **3aa** in 82% yield (entry 10). Under the above reaction conditions, removal of NaI in the system could detect the formation of the product **3aa**, albeit with low conversion, indicating that NaI may also help in situ formation of organo-iodide electrophiles from alkyl bromides in the system (entry 11). Finally, a control experiment confirmed that the defluoroalkylation failed without the addition of Ni catalyst (entry 12).

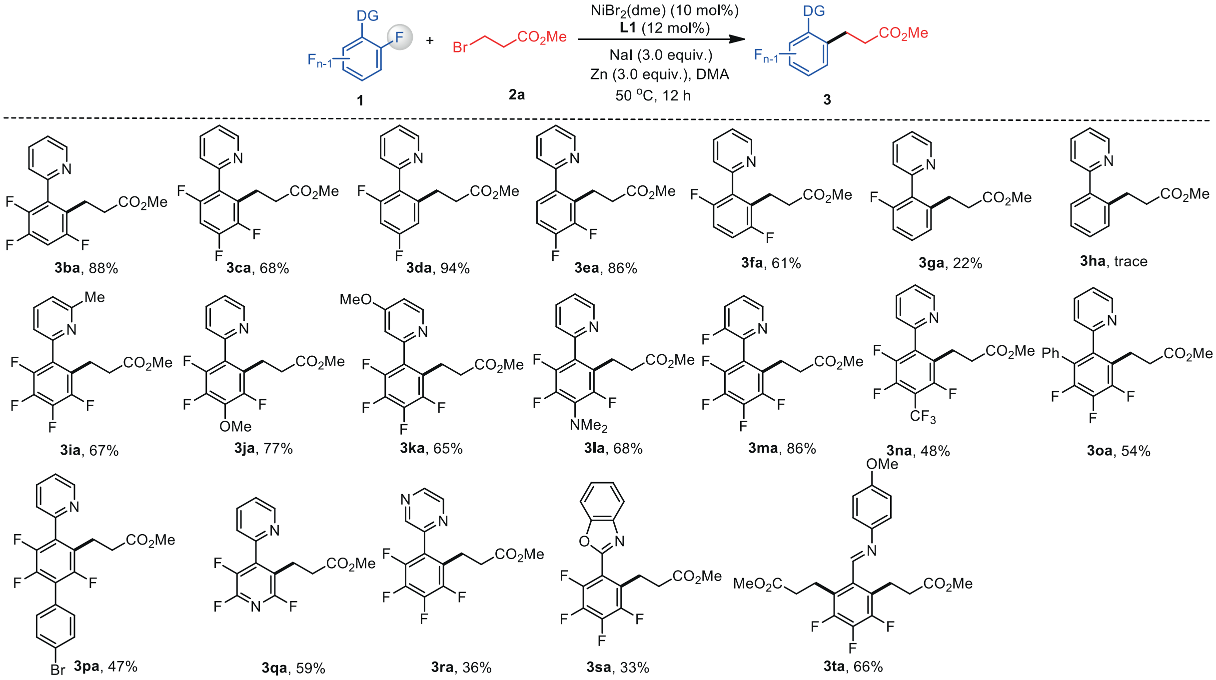
With the optimized reaction conditions in hand, we first examined the scope of alkyl electrophiles (Scheme 1). A range of primary alkyl bromides and iodides were efficiently transformed into the corresponding products, including those possessing ester (**3ab** and **3ac**), aryl (**3ad** and **3ae**), and fluoroalkyl (**3af-3ai**). Among these compounds, the structure of **3ae** was further confirmed by X-ray diffraction. Moreover, selective defluoromethylation of polyfluoroarene **1a** using CH_3I (**2j**) provided product **3aj** in 51% yield [88]. Alkyl tosylate **2l** and secondary alkyl iodide **2m** were also tolerated, in which the selection of ligand **L4** exhibited much higher reactivity. Under slightly modified reaction conditions, cyclic compounds **2n** and **2o** underwent facile coupling. Furthermore, a variety of benzyl chlorides **2p-2s** and β-substituted derivatives **2t** and **2u** could also maintain good reactivity. We were pleased to find that alkyl electrophiles **2v** and **2w** (I, Br, OTs) bearing a remote aryl group were also compatible with this reaction, and a reported chain walking process to the benzylic position was inhibited in our system [89,90].

We next subjected various polyfluoroarenes to the system with alkyl bromide **2a** (Scheme 2). A broad scope of polyfluoroarenes bearing four (**1b** and **1c**), three (**1d-1f**), and two (**1g**) fluorine atoms underwent cross-coupling to furnish products **3ba-3ga** in 22%-94% yields with excellent regioselectivities. It's found that the lower the number of substituted fluorine atoms was, the lower the yield of products, and monofluoroarene **1h** gave only trace amounts of product **3ha**. A series of polyfluoroarenes with substituents including Me (**1i**), OMe (**1j** and **1k**), NMe_2 (**1l**), CF_3 (**1n**) and Ph (**1o** and **1p**) moieties were competent coupling partners. The reductive cross-coupling can be extended to heteroaromatic systems; *e.g.*, polyfluoropyridine **1q** was converted to **3qa** in 59% yield. Other heteroarenes, such as pyrazine (**1r**) and benzo[d]oxazole (**1s**) could also be utilized as directing groups. In addition, the reaction of substrate **1t** containing a removable imine-directing group [40,41], can selectively activate two C–F bonds, affording dual alkylation product **3ta** in 66% yield.

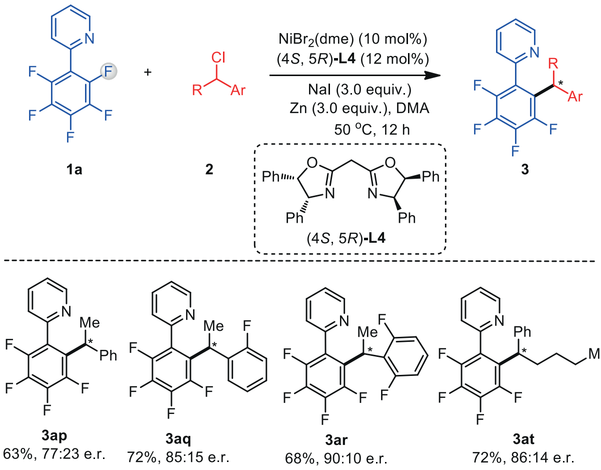
Our preliminary screening demonstrated the feasibility of the enantioselective reductive cross-coupling of polyfluoroarenes using a chiral nickel catalyst (Scheme 3). Thus, asymmetric induction in the reaction of polyfluoroarene **1a** and benzyl chloride **2p** was observed using a host of chiral bis(oxazoline) ligands having different backbones, among which (4*S*,5*R*)-**L4** displayed the highest enantiomeric ratio (er) value of 73:28 with 75% yield. Under the current reaction conditions, a variety of benzyl chlorides including **2q**, **2r** and **2t** could be coupled with **1a** to afford chiral products **3aq**, **3ar** and **3at** in moderate to good er values. While further screening of



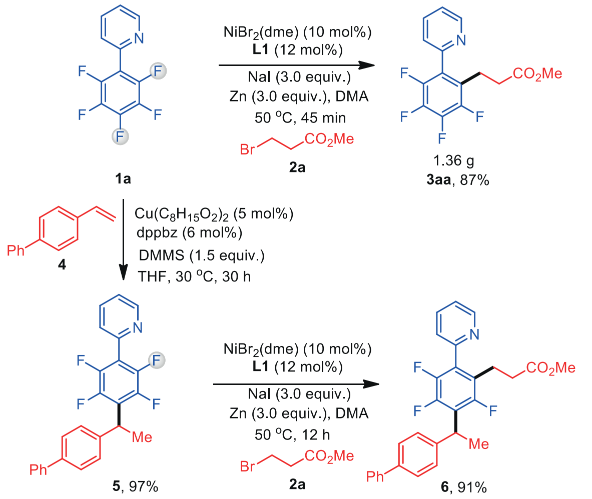
Scheme 1. Substrate scope of alkyl electrophiles. Reaction conditions: 10 mol% $NiBr_2$(dme), 12 mol% **L1**, **1a** (0.20 mmol), **2** (0.40 mmol), NaI (0.40 mmol), Zn (0.40 mmol), DMA (1 mL), 50 °C, 2 h; isolated yields. ^{a}12 mol% **L4**, 2 (0.60 mmol), NaI (0.60 mmol), Zn (0.60 mmol), 12 h.



Scheme 2. Substrate scope of polyfluoroarenes. Reaction conditions: 10 mol% $NiBr_2$(dme), 12 mol% **L1**, **1a** (0.20 mmol), **2** (0.60 mmol), NaI (0.60 mmol), Zn (0.60 mmol), DMA (1 mL), 50 °C, 2 h; isolated yields.



Scheme 3. Preliminary investigation of enantioselective variants.

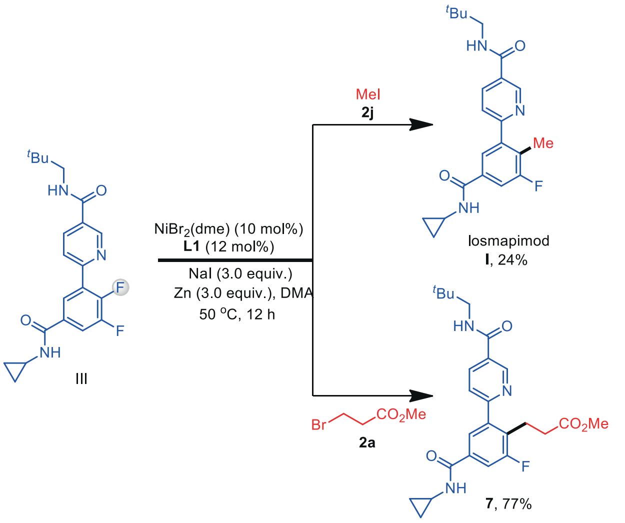


Scheme 4. Gram-scale transformation and successive defluoroalkylation of polyfluoroarene **1a**.

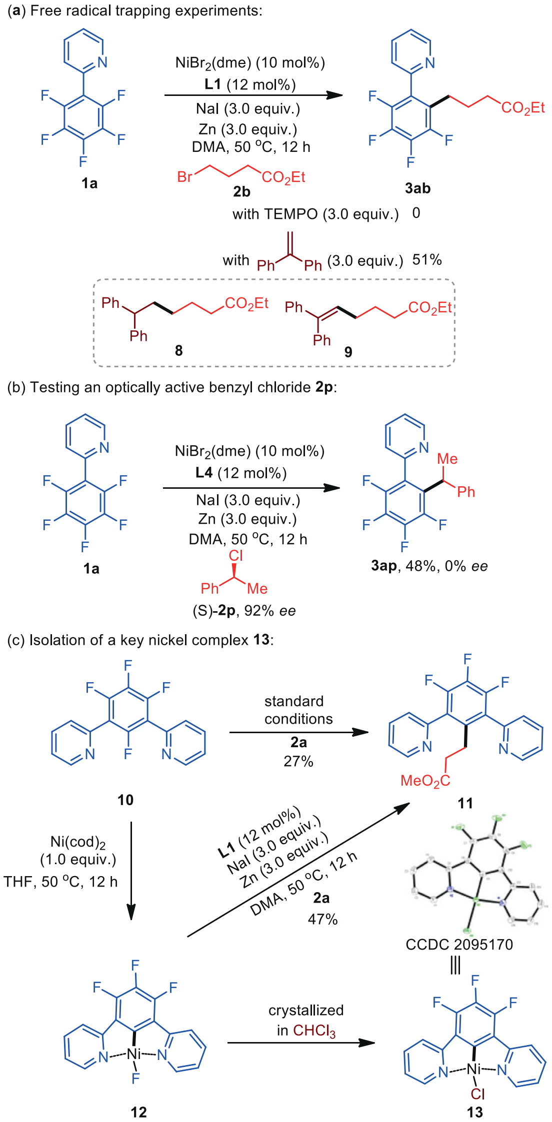
reaction system is currently underway to improve the enantioselectivity, these results would serve as a testament to the reaction mechanism involving an achiral benzyl radical during the coupling process [91–93].

Further investigations were conducted to show the practical utility of the defluoroalkylation process (Scheme 4). The developed system enabled the gram-scale synthesis of **3aa** (1.36 g, 87%) from substrates **1a** and **2a** with higher reactivity compared to the small-scale reaction. Successive defluoroalkylation at different C–F bonds with predictable site selectivity can offer an opportunity to produce complex molecules. For example, polyfluoroarene la has been shown to undergo site-selective defluoroalkylation at the *para*-position with styrene **4** to build product **5** in an excellent yield [46]. Under our reaction conditions, further ortho-selective defluoroalkylation of compound **5** with alkyl bromide **2a** afforded product **6** in 91% yield.

In order to prove the feasibility of the aforementioned assumption in Fig. 1a, we next tested the developed method as a key step in the total synthesis of losmapimod (**I**) (Scheme 5). Our route started with difluoroarene **III** obtained through Pd-catalyzed Suzuki-Miyaura coupling [39]. Under the developed reaction conditions, late-stage and site-selective defluoromethylation of compound **III** with CH_3I (**2j**) gave product **I** in 24% yield. Indeed, com-



Scheme 5. Synthesis of losmapimod (**I**) and an analog **7** by late-stage defluoroalkylation of difluoroarene **III**.



Scheme 6. Mechanistic experiments.

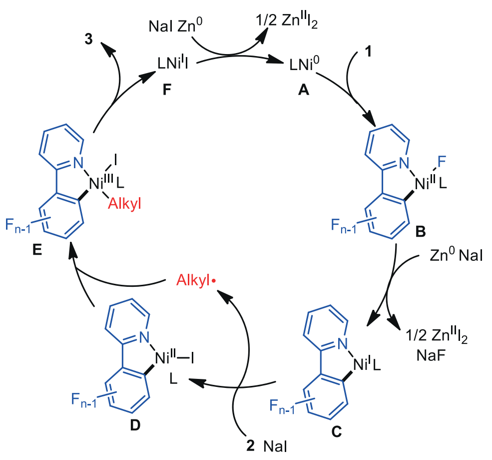


Fig. 2. Proposed mechanism.

pared to the method of *de novo* synthesis, such a strategy can greatly simplify the route to a library of losmapimod analogs for drug discovery. Using alkyl bromide **2a** as a model substrate, late-stage defluoroalkylation of **III** afforded a losmapimod analog **7** in 77% yield.

To establish the mechanism for this transformation, we performed several additional experiments (Scheme 6). When a stoichiometric amount of TEMPO was added to the mixture of substrates **1a** and **2b**, the reaction was completely suppressed (Scheme 6a). Moreover, the addition of 1,1-diphenylethylene to the reaction led to a lower yield of **3ab**, and adducts **8** and **9** were detected by GC-MS. These findings suggest the formation of alkyl radicals during cross-coupling. Furthermore, subjecting enantioenriched benzyl halide **2p** to the optimized reaction conditions with polyfluoroarene **la** only afforded racemic product **3ap**, demonstrating that an achiral benzyl radical was formed in the reaction (Scheme 6b) [94]. When polyfluoroarene **10** bearing two pyridyl groups was allowed to react with alkyl bromide **2a** under the standard reaction conditions, the corresponding product **11** was obtained in 27% yield (Scheme 6c). The reaction of substrate **10** with Ni(cod)$_2$ at 50 °C in THF generated a cyclometalated Ni^{II} complex **12** through oxidative addition of Ni^0 to the C–F bond. Subjecting complex **12** to alkyl halide **2a** under the developed system also afforded the target molecule **11** in 47% yield. These results indicate that complex **12** is a viable intermediate in the catalytic cycle. Finally, dissolving complex **12** in chloroform formed a Ni^{II}-Cl complex **13**, the structure of which was also confirmed by X-ray crystallographic analysis.

Based on the above results and those in previous works [95–98], a possible reaction mechanism is proposed (Fig. 2). Ni^0 species **A** undergoes oxidative addition to the C–F bond of polyfluoroarene 1 chelation-assisted by directing groups, affording Ni^{II} complex **B**, which is further reduced by Zn^0 to generate Ni^I intermediate **C**. Alkyl electrophile **2** reacts with **C** *via* a single electron transfer pathway to form Ni^{II} complex **D**, which further combines with cage-escaped alkyl radicals to generate Ni^{III} species **E**. Subsequent reductive elimination of **E** delivers final product **3** and generates Ni^I species **F**. This species can be further reduced by Zn^0, thus forming Ni^0 species to complete the catalytic cycle.

In summary, we have developed the first defluoroalkylation of polyfluoroarenes with alkyl electrophiles through Ni-catalyzed reductive cross-coupling reactions. Because the coordinating functional groups assist the oxidative addition of the C–F bond, these reactions occur with high reactivity and site selectivity. Further exploration of the enantioselective variant of this defluoroalkylation and examination of detailed mechanistic features are currently ongoing and will be reported in due course.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.01.077.

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