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Radical dehydroxymethylative fluorination of aliphatic primary alcohols and diverse functionalization of α-fluoroimides *via* $BF_3{\cdot}OEt_2$-catalyzed C–F bond activation

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ABSTRACT

Ag_2CO_3-promoted dehydroxymethylative fluorination of aliphatic alcohols has been achieved with Selectfluor as both oxidant and fluorine source. The reaction involves β-fragmentation of primary alkoxy radicals, followed by the fluorination of the resulting C-centered radical intermediates. The transformation proceeds under mild reaction conditions and exhibits a broad substrate scope, thus opening up a new entrance to the synthesis of fluorinated constructs including α-fluoroimides and 1-fluoroalkyl benzoates as well as secondary and tertiary alkyl fluorides like versatile 2-fluoro-2-alkyl 1,3-propandiol derivatives. The divergent functionalization of the obtained α-fluoroimides enables an efficient access to amine derivatives through C–F bond activation under the action of $BF_3{\cdot}OEt_2$.

The incorporation of fluorine atom in an organic molecule has shown potential in regulating the pK_a, increasing the lipophilicity, improving metabolic stability and bioavailability, and exerting the conformation control of the molecule [1]. As a consequence, the fluorinated settings increasingly attract attention in pharmaceuticals, agrochemicals, and materials science [2,3]. The development of novel synthetic methods for fluorine atom incorporation has become a hot topic in organic synthesis [4]. Besides conventional nucleophilic and electrophilic fluorination [5,6], radical fluorination reactions have emerged as powerful strategies for efficiently constructing C–F bonds [7]. The recent significant advances include decarboxylative fluorination and C–H fluorination (Scheme 1a) under transition-metal catalysis [8], photocatlysis [9], and electrocatalysis [10]. These transformations involve either a radical reaction mechanism or a radical-polar crossover reaction pathway.

Alkoxy radicals [11] have proven to be versatile reactive intermediates and usually engage in three elementary reactions, namely 1,*n*-hydrogen atom transfer (1,*n*-HAT, $n = 5, 6, 8$, *etc.*), β-scission, and addition to alkenes [12–14]. It is well-recognized that compared to tertiary and strained cyclic alkoxy radicals, primary alkoxy radicals show the relatively smaller tendency to undergo β-cleavage. However, such processes have been designed and applied in a plethora of organic transformations over the past years [15]. Photocatalyzed generation of primary alkoxy radicals and the following β-fragmentation have been achieved by the Zuo [16], Chen [17], and Martin groups [18] using the corresponding alcohols or *N*-alkoxyphthalimides as the starting materials (Scheme 1b). These processes have been employed in the formation of various carbon-carbon bonds by means of the Giese reactions and the nickel-catalyzed cross-coupling reactions with alkyl and aryl bromides, providing elegant routes to convert readily available alcohols into high-valued chemicals. The Minisci reactions based on β-fragmentation of primary alkoxy radicals have also been independently documented by Liu, Chen, and their co-workers [19,20]. These reactions used hypervalent iodine reagents as oxidants with resort to visible light irradiation (Scheme 1b). In continuation of our interest in β-scission of alkoxy radicals [21], we herein report radical dehydroxymethylative fluorination of aliphatic alcohols. The reaction accommodates various alcohols and enables access to α-fluoroimides and 1-fluoroalkyl benzoates as well as secondary and tertiary alkyl fluorides. Divergent functionalization of α-fluoroimides is also established through $BF_3{\cdot}OEt_2$-catalyzed C–F bond activation, providing a novel route to amine derivatives.

α-Amino C-centered radicals that are stabilized by the adjacent *N*-substituents are considered reactive intermediates. These species have been successfully applied to construction of carbon-

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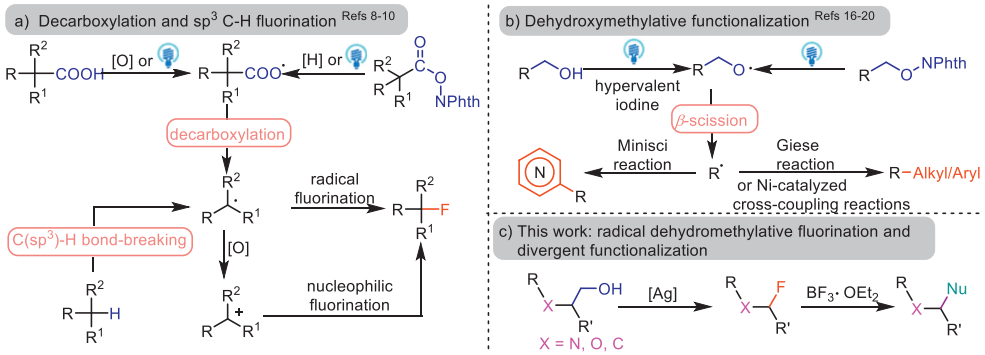


Scheme 1. Dehydroxymethylative functionalization of primary alkoxy radicals *via* β-fragmentation.

carbon bonds and carbon-heteroatom bonds [22,23]. Based on this knowledge, we chosen L-*N*-phthaloyl-leucinol **1a** as a model substrate to determine the optimal reaction parameters for dehydroxymethylative fluorination of aliphatic primary alcohols. Since Ag(II) species are recognized to be capable of converting alcohols into the corresponding alkoxyl radicals by single electron transfer [24], and the ensuing β-scission is highly expedited by hydrogen bond-donating solvents [25], we thereby conducted silver salt-promoted oxidative radical dehydroxymethylation of **1a** using Selectfluor as fluorine source in aqueous acetone. After scrutinizing various reaction parameters, including composition of solvent, reaction concentration, reaction temperature, type and equivalent of silver salts and fluorinating source, and additives HF·H_2O or HF·pyridine (Table S1 in Supporting information), we found that treatment of alcohol **1a** with 0.5 equiv. of Ag_2CO_3 and 5.0 equiv. of Selectfluor in acetone/H_2O (v/v = 4:1) delivered monofluoride **2a** and difluoride **2a'** in 89% overall yield and at the highest ratio of **2a**/**2a'** = 4:1 after stirring for 30 min at 28 °C (Scheme 2). Based on these observations, the control experiments, and the precedents in the literature [16–21], a radical reaction mechanism involving the formation of intermediate alkoxy radical and the following β-fragmentation was proposed (Scheme S1 in Supporting information).

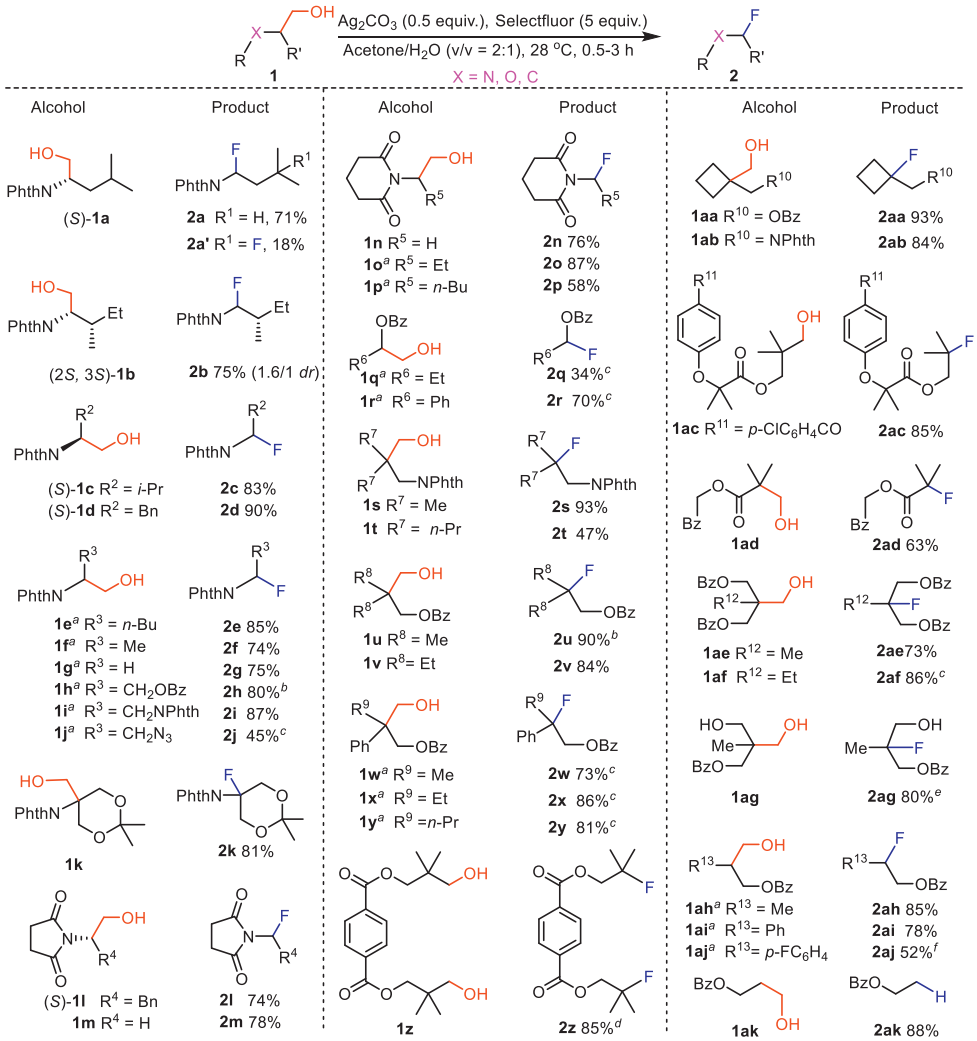
With the optimized condition established, we then examined the reaction scope and limitations with respect to structurally diverse primary alcohols (Scheme 2). A variety of *N*-phthaloyl-β-amino alcohols were first examined, and it was found that alcohols **1b**–**1g** were amenable to dehydroxymethylative fluorination, affording the desired α-fluoroimides **2b**–**2g** in 74%–90% yields. These transformations deserve further comments. Distinct from leucinol derivative **1a**, dehydroxymethylative fluorination of *N*-phthaloyl-L-isoleucinol **1b** and 2-phthalimido-hexanol **1e** produced α-fluoroimides **2b** and **2e** in 75% and 85% yields as the sole product, leaving the secondary C–H moiety at the δ position untouched. We ascribe these differences to the lower bond dissociation energy of the δ tertiary C–H bond in **1a** than that of δ the secondary C–H bonds in **2b** and **2e** [26]. Ready access to **2c** and **2d** demonstrates the tolerance of γ tertiary C–H and benzyl C–H next to hydroxy group to the radical dehydroxymethylative fluorination. *N*-Phthaloyl-glycinol **1g** was uneventfully converted into phthalimidomethyl fluoride **2g** in 75% yield. This result highlights that phthalimidomethyl radical is easily generated and enables fluorine abstraction from Selectfluor although it is a primary alkyl radical.

Taking serinol derivatives **1h**–**1j** as examples, we evaluated the influence of the functional group on the side chain to dehydroxymethylative fluorination. 3-*O*-Benzoyl-2-*N*-phthaloyl serinol **1h** and its 3-deoxy-3-phthalimido congener **1i** underwent smoothly dehydroxymethylative fluorination to afford fluorides **2h** and **2i** in 80% and 87% yields, respectively. 3-Azido-3-deoxy-*N*-phthaloyl serinol **1j**, however, furnished the expected fluoride **2j** in much lower yield of 45%. This observation might be attributed to the stronger electron-withdrawing nature of azido group than benzoyloxy and phthalimido groups [27]. This property of azido group disfavors the formation of α-phthalimido alkyl radicals. It is worth noting that the high-yielding transformation of alcohols **1h** into **2h** was carried out on one mmol scale, demonstrating the feasibility of scale-up for dehydroxymethylative fluorination.

N-Phthaloyl-tris(hydroxymethyl)aminomethane derivative **1k** was subjected to dehydroxymethylative fluorination, supplying fluoride **2k** in 81% yield with the quaternary C–F bond formation. It should be noted that the acetonide group to protect 1,3-diol is compatible with the reaction conditions although the reaction medium is found to be acidic [28]. Besides *N*-phthaloyl-β-amino alcohols, *N*-succinyl-β-amino alcohols **1l** and **1m** as well as *N*-glutaryl congeners **1n**–**1p** are all competent substrates, enabling the production of fluorides **2l**–**2p** in 58%–87% yields.

Glycosyl fluorides and reverse glycosyl fluorides are typical representatives of α-fluoroethers [21,29]. These architectures have been fruitfully applied in carbohydrate chemistry as glycosylating agents. Inspired by these advances, we attempted to make 1-fluoroalkyl carboxylates through dehydroxymethylative fluorination. Pleasingly, β-benzoyloxy-propanol **1q** and β-benzoyloxy-β-phenyl ethanol **1r** participated in dehydroxymethylative fluorination reaction, and yielded 1-fluoroalkyl benzoates **2q** and **2r** in 34% and 70% yields. The higher yield for **2r** compared with that for **2q** might result from the stabilizing effect of phenyl on the adjacent C-centered radicals.

Next, we moved our focus to dehydroxymethylative fluorination of β,β-disubstituted 1,3-diols and derivatives. To our delight, primary alcohols **1s**–**1y** were uneventfully converted into fluorides **2s**–**2y** in the yields ranging from 47% to 93%. Compound **1z** bearing two primary hydroxy groups smoothly underwent dehydroxymethylative fluorination to give rise to difluorides **2z** in 85% yield. The suitability of the present method for scale-up reaction was again demonstrated by 90% conversion of **1u** into **2u**. Both 1,1-cyclobutanedimethanol monobenzoate **1aa** and its phthalimido analogue **1ab** were exposed to dehydroxymethylative fluorination, efficiently giving rise to fluorides **2aa** and **2ab** in 84% and 93% yields. Fenofibrate is an anti-hyperlipidemic drug with a wide range of uses in clinic [30]. Recently, this compound was found to reveal potential in reducing the risk of SARS-CoV-2 infection [31]. Notably, subjection of monoester **1ac**, obtained by reacting β,β-dimethyl 1,3-diol with fenofibric acid, to dehydroxymethylative fluorination reaction afforded 85% yield of **2ac** as a structurally intriguing fluorinated congener of fenofibrate. α-Fluoro pivalate **2ad** was successfully made by treating **1ad** under the established conditions. This result demonstrates that a tertiary C-centered radical next to an ester group is an enabling species for fluorine abstraction from Selectfluor.



Scheme 2. Scope of dehydroxymethylative fluorination. [a] Racemic substrate was used. [b] 1 mmol scale; [c] Acetone/H_2O (v/v = 4:1); [d] Ag_2CO_3 (1.0 equiv.), Selectfluor (10.0 equiv.); [e] Selectfluor II as fluorinating reagent; [f]Acetone/H_2O (2.1 mL, v/v = 6:1).

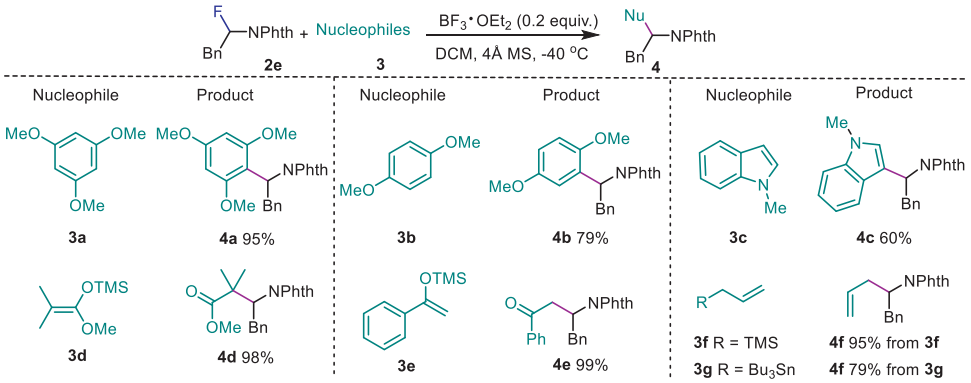
1,1,1-Tris(hydroxymethyl)ethane dibenzoates **1ae** and its propane congener **1af** as well as 1,1,1-tris(hydroxymethyl)ethane monobenzoate **1ag** were suitable for dehydroxymethylative fluorination, uneventfully furnishing fluorides **2ae**, **2af**, and **2ag** in satisfactory yields. Radical decarboxylative fluorination of α,α-disubstituted malonic acids has proven to be an efficient approach to *gem*–difluoro constructs [32]. However, we failed to convert diol **1ag** into the corresponding *gem*–difluoropropyl benzoate by concomitant dehydroxymethylative fluorination of the two hydroxymethyl units. These results could be rationalized by the observations that radical decarboxylation is easier and faster than the corresponding radical dehydroxymethylation [33]. Since 2-alkyl 1,3-diols serve as readily modifiable building blocks in synthetic chemistry [34,35], and 2-alkyl 1,3-diol-based moieties are frequently found in bioactive compounds [36,37], ready access to **2ae**–**2ag** lays a solid foundation for preparing 2-fluoro-2-methyl/ethyl 1,3-diol-based derivatives like fluorinated drug candidates [38,39] and functionalized polymer [40,41].

Dehydroxymethylative fluorination of 2-methyl and phenyl propane-1,3-diol derivatives **1ah** and **1ai** worked well, giving fluorides **2ah** and **2ai** in 85% and 78% yields. However, treatment of 2-*para*-fluorophenyl-propane-1,3-diol monobenzoate **1aj** in aqueous acetone (v/v = 6:1) afforded fluoride **2aj** in 52% yield along with the formation of benzoyloxymethyl aryl ketone **S21** (see Supporting information for details) in 25% yield.

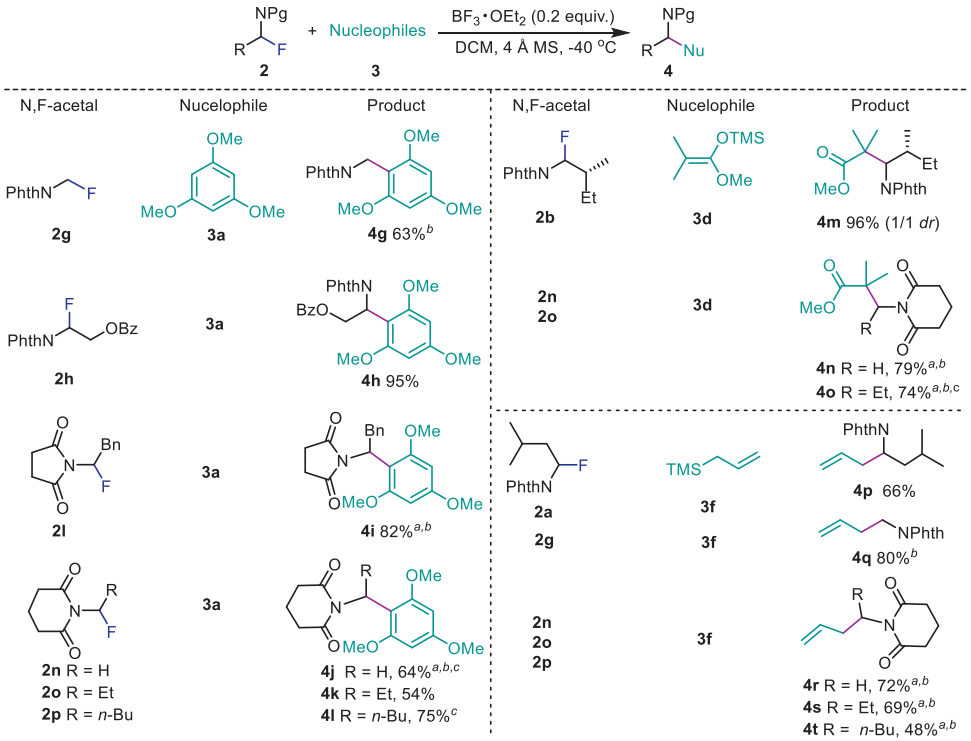
Treatment of propane-1,3-diol monobenzoate **1ak** with the current conditions for dehydroxymethylative fluorination supplied ethyl benzoate **2ak** in 88% yield without the desired fluoride detected. This result implies that 2-benzoyloxyethyl radical, generated by β-fragmentation of 3-benzoyloxypropoxy radical, prefers to abstract hydrogen atom from the medium rather than abstracts fluorination from Selectfluor.

Silver-catalyzed decarboxylative fluorination has proven to be a powerful method to make aliphatic fluorides [28]. For comparison purpose, a competition experiment was set up using each about 0.1 mmol of *N*-phthaloyl L-phenylalanine **S22** and **1e** as the substrates (see Supporting information for details) under the established conditions. The transformation gave **2e** in 49% (based on the quantities of the used **S22** and **1e**) along with 56% of **1e** recovered (based on the quantities of the used **1e**). These results showed a possibility of selective decarboxylative fluorination over the dehydroxymethylative fluorination due to the faster rate the former reaction than the latter. These findings are consistent with the observations in the literature [42].

N,*N*-Aminals, *N*,*O*-acetals, α-chloroaminals, and α-aminosulfides are popular equivalents of *N*-iminium ions which are an class of



Scheme 3. C–C bond formation with various nucleophiles.



Scheme 4. C–C bond formation with various α-fluoroimides and nucleophiles. [a] $BF_3{\cdot}OEt_2$ (1.2 equiv.); [b] 0 °C; [c] Nucleophile (3.0 equiv.).

important reactive species and play significant roles in organic synthesis [43–46]. With the various α-fluoroimides in hand, we explored the feasibility of these compounds as the masked surrogates of iminium ions through C–F bond activation under the catalysis of Lewis acid (Scheme 3) [47]. It was found that 0.2 equiv. of $BF_3{\cdot}OEt_2$ was capable of catalyzing the Friedel-Crafts alkylation of electron-rich aromatics **3a**-**3c** with α-fluoroimide **2e** through C–F bond activation. The reactions afforded α-aryl imides **4a**-**4c** in 60%–95% yields. With enol silyl ethers as nucleophiles, **2e** was converted into β-imido ester **4d** and β-imido ketone **4e** in excellent yields. Furthermore, the couplings of **2e** with allyltrimethylsilane **3f** and allyltributyltin **3g** furnished homoallylic imide **4f** in 95% and 79% yields, respectively.

Encouraged by these results, we moved to examine the scope of α-fluoroimides as coupling partners using 1,3,5-trimethyloxy benzene **3a**, enol silyl ether **3d**, and allylic trimethylsilane **3f** as nucleophiles. As listed in Scheme 4, all $BF_3{\cdot}OEt_2$-catalyzed transformations performed well, giving the corresponding products **4g**–**4t** in 48%–96% yields. 1,2-Amino alcohols are privileged building blocks prevalent in pharmaceuticals, agrochemicals, natural products, and catalyst architectures [48]. Efficient access to **4h** in 95% yield shows the potential of **2h** as an electrophile in introducing an β-amino alcohol unit. It should be mentioned that the reaction of α-fluorophthalimide **2a** with allyltrimethylsilane **3f** afforded homoallylimide **4p** and enamine **4p'** (see Supporting information for details) in 66% and 13%, respectively. The formation of **4p'** highlights the involvement of iminium ion intermediates during the reaction. In addition, α-fluoroimide **2b** possessing a chiral side chain did not show apparently diastereoselective bias, affording **4m** as a mixture ($dr = 1/1$). Access to benzyl imide **4g** and homoallyl imide **4q** indicates that phthalimidomethyl fluoride **2g** is alternative aminomethylating agent, but it required to be activated at a temperature up to 0 °C for the successful couplings. Activation of α-fluorosuccinimide **2l** and α-fluoroglutarimide **2n**–**2p** entailed

more forcing conditions, and imides **4i**, **4j**, **4n**, **4o**, and **4r**–**4t** were obtained in good yields at 0 °C in the presence of 1.2 equiv. of $BF_3{\cdot}OEt_2$.

Notice that imido moieties are not only the precursors for amines but also versatile building blocks to construct structurally complex and biologically intriguing natural products and pharmaceuticals which contain nitrogen atom [49]. For instance, partial reduction of phthalimidyl, succinimidyl, and glutarimidyl groups with $NaBH_4$ have the potential to be transformed into the corresponding acylaminoacetals which are a class of acyliminium ions precursors of synthetic importance [50]. Consequently, ready accessibility of imides **4a**–**4t** offers promising platform for synthesis of biologically active amines.

In summary, a novel and efficient protocol has been established for radical dehydroxymethylative fluorination of aliphatic primary alcohols with Ag_2CO_3 as promoter and Selectfluor as fluorine source and oxidant. The reaction proceeded under mild reaction conditions, and accommodated a wide range of substrates, efficiently providing various alkyl fluorides of importance. Preliminary mechanistic studies suggest that a β-cleavage of primary alkoxy radicals might be involved. Diverse functionalization of α-fluoroimides was also achieved by $BF_3{\cdot}OEt_2$-catalyzed C–F bond activation. The present work opens up a door for installation of fluorine atom in organic molecules with primary alcohols as the starting materials and adds another tool to the arsenal for the synthesis of structurally diversified amines of biological relevance. With α-fluoroimides as electrophiles, enantioselective construction of carbon-carbon bonds is underway in our laboratory.

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.

Acknowledgments

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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.2024.109748.

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