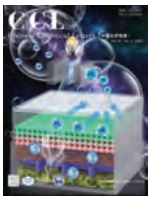
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A highly efficient approach to Z-monofluoroolefin-dehydroamino acid derivatives from *gem*-difluoroalkenes

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ABSTRACT

Amino acids are the building blocks of proteins and various bioactive molecules. Fluorination is a key strategy in medicinal chemistry, making the development of new and efficient methods for fluorinating amino acids highly desirable. While many fluorination reagents have been developed, their application on amino acid frameworks remains limited. In this study, we demonstrate that ethyl 3–bromo-2-((diphenylmethylene)amino)-3,3-difluoropropanoate is an effective *gem*–difluoroalkenes precursor for constructing a library of monofluoroolefin amino acids *via* a Pd-catalyzed cross-coupling reaction.

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Organic fluorides have important applications in the pharmaceutical industry due to the ability of C-F bonds to alter the physical, chemical, and biological properties of small organic compounds, including solubility, metabolic stability, hydrogenbonding ability, lipophilicity, and chemical reactivity [1–4]. As a result, an increasing number of products of the pharmaceutical, agrochemical, and material science industries contain fluorine atoms as key components [5]. Nearly 20 % of the top 200 drugs in 2023 contain a fluorine atom in their structure [6]. Monofluoroolefins are ideal amide-bond mimics in medicinal chemistry and hold great potential in isomer-based drug design [7], creating a growing need for efficient methods to synthesize various classes of monofluoroolefins. The alkylation [8], arylation [9–11], hydrogenation [12–14], alkynylation [15], borylation [16,17], silylation [18–20], carbonylation [21–23] and carboxylation [24] of C-F bonds in difluoroalkenes can be achieved through free radical [25–30] and metal catalysis strategies [31–48] (Fig. 1b). Although important progress has been achieved in the conversion of C-F bond to other functional groups, the application such strategy to monofluoro amino acid derivatives has not been disclosed. Here, we report a newly developed glycine containing fluoroalkylation reagent, which is directly applied to the synthesis of monofluorocarbon amino acids *via* a Pd-catalyzed cross-coupling reaction. A variety of arylboronic acids were well tolerated, yielding products with well-defined stereochemistry in moderate to high yields ($Z{:}E > 20{:}1$). Additionally, late-stage functionalization of bioactive compounds was also achieved. Furthermore, this new glycine-containing fluoroalkylation reagent was applied in Heck coupling reactions (Fig. 1c).

Amino acids are essential for the human body and each amino acid has unique functional properties. Among them, glycine is the smallest amino acid, which can be used as a raw material for the production of cephalosporins, intermediates of thiamphenicol, and intermediates for the synthesis of imidazoleacetic acid. The addition of fluorine atoms to amino acids has become a key synthetic strategy for the development of new drugs and pesticides, such as eflornithine, bicalutamide (Casodex) and (*S*)-3-fluorothalidomide (Fig. 1a). However, the approach of combining monofluoroolefins with small amino acids has not yet been reported till now. Therefore, it would be great importance and attractive to design a new *gem*–difluoroolefin precursor, using glycine as the backbone to construct the bank of α,β-dehydroamino acids which are non-natural amino acid with unique properties, such as planar conformation and the ability to resist degradation by peptidases [49]. The fluorinated -CF_2Br block serves as a key reacting center for forming fluorinated products, a strategy that has been extensively explored by the groups of Zhang [50–53], Ackermann [54–56], and

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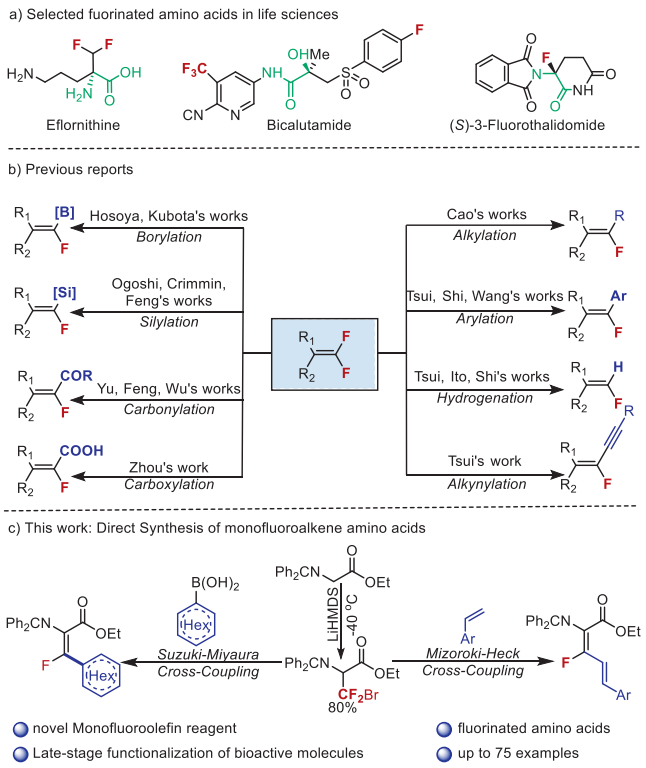


Fig. 1. Background and novel monofluoroalkene reagent.

our group [57–63]. Introducing the -CF_2Br group directly onto protected glycine may offer a novel approach to fluoroalkylation, allowing for the preparation of new framework compounds through well-established catalytic systems such as cross-coupling, C–H activation, photocatalytic radical processes, and electrocatalytic reactions (Fig. 1c).

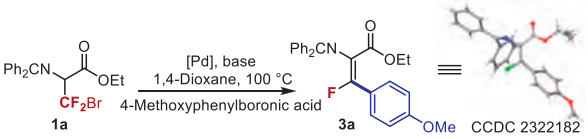
At the beginning of the study, the 3–bromo-3,3-difluoroalanine Schiff base (**1a**) was easily prepared from protected glycine in good yield (8 g, 80 %). We then explored its application in the Pd-catalyzed cross-coupling reaction to synthesize fluorinated amino acid derivatives. When **1a** was treated with *p*-methoxyphenylboronic acid (**2a**), potassium carbonate, and water (150 µL) in the presence of $PdCl_2$ (5 mol%) and PPh_3 (10 mol%) in 1,4-dioxane at 100 °C for 2 h, the fluorinated dehydroamino acid **3a** was obtained in 65 % yield (Table 1, entry 1). Subsequently, we screened some phosphine ligands, including Davephos, Xantphos, dppf, and dppe, among which the **3a** yield of the dppf ligand was slightly higher than that of the other ligands (Table 1, entries 1–5). We next employed Pd(dppf)Cl_2 directly as the catalyst, giving the product **3a** in 78 % yield. We also screened different bases, including K_3PO_4, $KHCO_3$, KOH, Et_3N, and Cs_2CO_3, and obtained excellent yield of **3a** when using Cs_2CO_3. In a control experiment using only Cs_2CO_3 no product was obtained, confirming that Pd(dppf)Cl_2 was essential for this reaction (Table 1, entry 12).

After optimizing the reaction conditions, the applicability of this reaction was further examined by evaluating the substrate scope of aryl boronic acids, as presented in Fig. 2. The reaction showed good tolerance to various functional groups, including electron-donating groups such as methoxy, methylthio, and *tert*–butyl, as well as electron-withdrawing groups like -F, -Cl, ester, formyl, and -CF_3. For electron-donating substituents in the *para* position, such as -OMe, -SMe and -^{t}Bu, the target products were obtained in 93 %, 85 %, and 90 % yields, respectively (**3a-3c**). With chlorine (**3f**) and fluorine (**3e**) substituents at the *para* position, the target products were obtained in 88 % and 87 % yields, respectively. The reaction yield was slightly decreased when electron-withdrawing substituents such as ester, formyl, and -CF_3 were introduced on the aromatic ring (**3g-3i**). Notably, when cyano group was introduced in the *para* position, the target product was obtained in a lower yield (**3j**, 57 %). The yields for *meta* and *para*-substituted phenylboronic acid were similar (**3k-3o**), but due to steric hindrance, the yield for *ortho*-substituted phenylboronic acid was lower compared with the *para* and *meta*-substituted ones (**3p-3t**). Polysubstituted phenylboronic acids were also examined, and they all provided the desired products in good to excellent yields (**3u-3ac**), except for *ortho*-disubstituted phenylboronic acids, which resulted in lower yields (**3x**). When naphthylboronic acids and biphenylboronic acidwere used, the corresponding products were isolated in excellent yields (**3ad-3ag**). The yield remained consistent when the ethyl ester was replaced with methyl ester, *tert*–butyl ester, or benzyl ester (**3ah-3aj**). Given the widespread presence of heterocycles in bioactive molecules, we further investigated heteroarylboronic acids. The yield of 3-thiopheneboronic acid was higher than that of 2-thiopheneboronic acid, with the target products obtained in 88 % and 65 % yields, respectively (**3ak**, **3al**). The reaction also proceeded well with 2-benzothiopheneboronic acid, yielding 62 % of the target product (**3am**). When other *N*-heterocyclic boric acids such as pyridine, quinoline, isoquinoline, and indole were used, the obtained yields were relatively poor (**3ao-3av**).

Subsequently, we found that this new glycine-containing fluoroalkylation reagent (**1a**) not only performed well in the Suzuki Cross-Coupling but also underwent Heck Cross-Coupling. Under optimal conditions, the scope of styrenes was investigated (Fig. 3, **5a-5o**). Styrenes substituted with electron-donating and electron-withdrawing groups were all compatible, resulting in the corresponding products in moderate to good yields. Moreover, the stereoselectivity was excellent, favoring the (2*Z*,4*E*)-isomer regardless of the substituents. The product structure and the configuration of the two double bonds were confirmed by NMR and the X-ray crystal structure of compound (**5b**).

Moreover, to further demonstrate the practical aspects of the present method, several transformations involving the amino and fluoride group of **3a** were conducted (Fig. 4). Treatment of **3a** with a catalytic amount of acid in water led to a deamination product, which could be further modified with acyl chloride or benzoyl chloride, yielding amide products (**6a**, **6b**) in good yields. Fluorinated olefins can be diversified on the C(sp^2)-F bonds through var-

Table 1
Optimization of reaction conditions.[a]



Entry	[Pd]	Ligand	Base	Yield (%)[b]
1	$PdCl_2$	PPh_3	K_2CO_3	65
2	$PdCl_2$	Davephos	K_2CO_3	70
3	$PdCl_2$	Xantphos	K_2CO_3	69
4	$PdCl_2$	dppf	K_2CO_3	76
5	$PdCl_2$	dppe	K_2CO_3	65
6	Pd(dppf)Cl_2	None	K_2CO_3	78
7	Pd(dppf)Cl_2	None	K_3PO_4	60
8	Pd(dppf)Cl_2	None	$KHCO_3$	65
9	Pd(dppf)Cl_2	None	KOH	35
10	Pd(dppf)Cl_2	None	Et_3N	43
11	Pd(dppf)Cl_2	None	Cs_2CO_3	89
12	None	None	Cs_2CO_3	0

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), base (3.0 equiv.), 150 µL H_2O, [Pd] (5 mol%), 1,4-dioxane (0.5 mL), 100 °C, under argon.
[b] Solated yield.

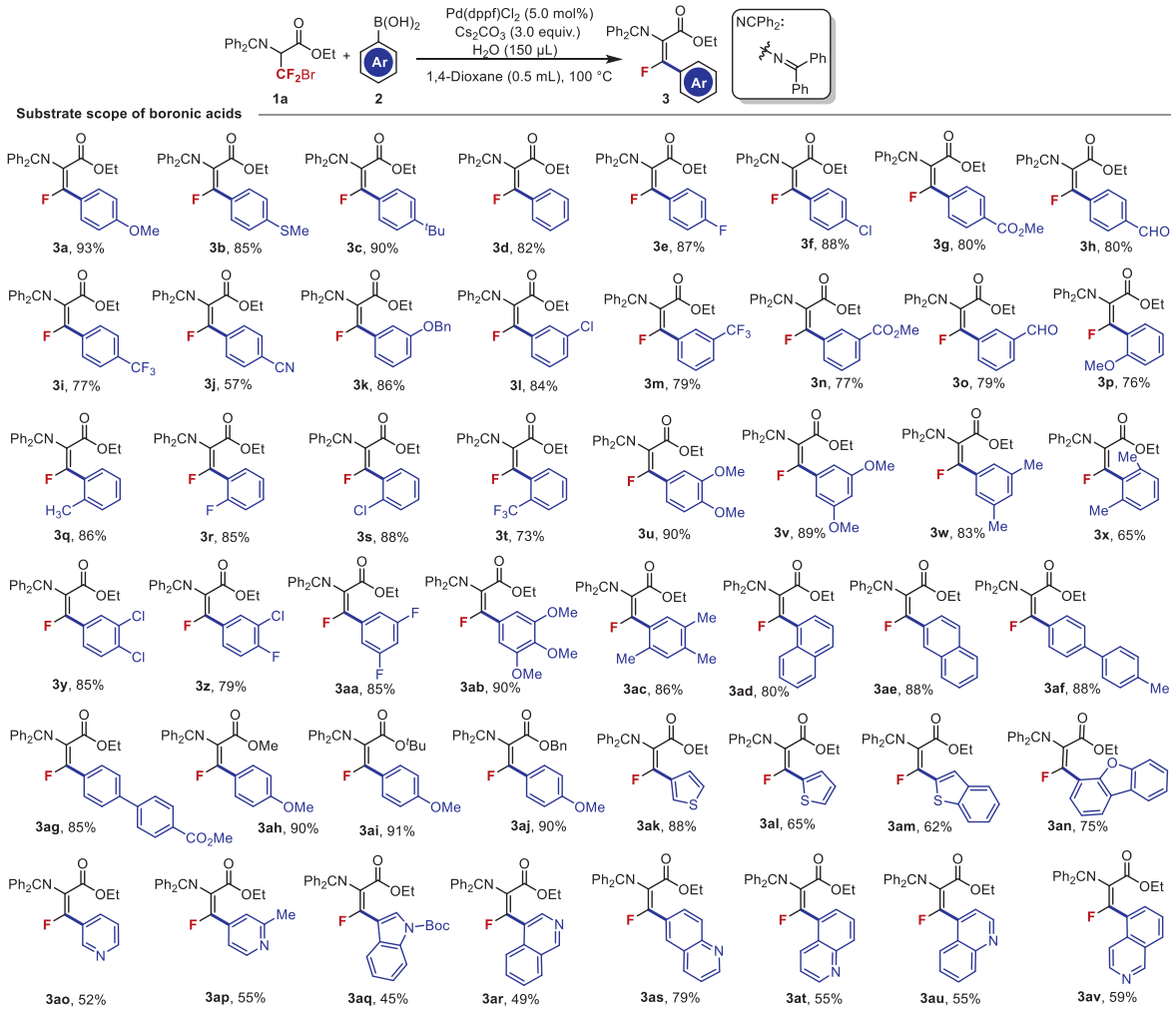


Fig. 2. Substrate scope of Suzuki-Miyaura cross coupling. Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), Cs_2CO_3 (3.0 equiv.), 150 μL H_2O, $Pd(dppf)_2Cl_2$ (5 mol%), 1,4-dioxane (0.5 mL), 100 °C, under argon, isolated yield.

ious nucleophilic substitution reactions. To this end, we performed a series of follow-up transformations with different nucleophiles. For instance, **3d** or **3a** reacted directly with trimethylsilyl cyanide, morpholine, and trifluoroethanol under mild conditions, yielding the corresponding products in good to excellent yields (**7a-7c**).

Given the essential role of amino acids in biological systems, site-selective transformations of amino acids, peptides, and proteins have become a prominent topic in organic synthesis, medicinal chemistry, and chemical biology. Among various methods developed, the selective introduction of fluorinated functional groups into amino acids and peptides has emerged as an effective strategy for modifying their physicochemical and biological properties. However, the synthesis of fluorinated reported to date typically involves multiple synthetic steps. In a previous study, the synthesis of peptides containing 2-amino-3-fluoroacrylic acid required 7 steps, with a yield of only 16% [49]. By using our strategy with glycine, 60% of the target product (**8a**) was obtained in just 2 steps (Fig. 5a). The late-stage functionalization of well-known bioactive organic molecules provide a novel, highly efficient approach in this field, which can facilitate the discovery of new drugs. Till now, there were no studies on the direct introduction of the 2-amino-3-fluoroacrylic acid moiety into complex molecules. In this work, we demonstrated that estrone (a female hormone), clofibrate (a lipid-lowering agent), and paclitaxel (an anticancer agent) were successfully coupled with 2-amino-3-fluoropropenoic acid in excellent yields (83%–86%) (Fig. 5b, **8b-8d**). Moreover, our method enabled the synthesis of fluorodehydroborated L-phenylalanine (**8e**), a highly effective anti-cancer drug, as well as derivatives of the tryptophan framework (**8f**). Notably, when **1a** reacted with hydroxyphenylboronic acid, an F-aminocoumarin derivative (**8g**) is obtained under standard reaction conditions with a 69% yield. This derivative was then treated with 1 mol/L hydrochloric acid, resulting in F-aminocoumarin (**8h**) in a good yield (50%) (Fig. 5c).

Following the successful small-scale reactions, the reaction was scaled up to evaluate its applicability at the gram scale (Fig. 6). Reactions between 5 mmol of 3–bromo-3,3-difluoropropanoic acidethyl ester Schiff base and **15** mmol of 4-methoxyphenylboronic acid or phenylboronic acid yielded the target products **3a** (80%) and **3d** (70%) with excellent yields. Additionally, scale-up experiments for the antitumor drug derivative (F-BPA) (**8e**) were conducted, achieving a 65% yield of the target product. It is worth mentioning that these reactions only require a low catalytic amount of palladium (1 mol%).

Several control experiments were conducted to explore the reaction pathway and the origin of stereoselectivity (Scheme 1). Initially, substrate **9** was synthesized and used instead of **1a**. However, the desired product **9a** was not observed, along with **9** being recovered. Based on these results, we believe that direct oxidative addition between Pd(0) and RCF_2Br does not occur in the catalytic cycle (Scheme 1a). According to previous reports [64,65], we spec-

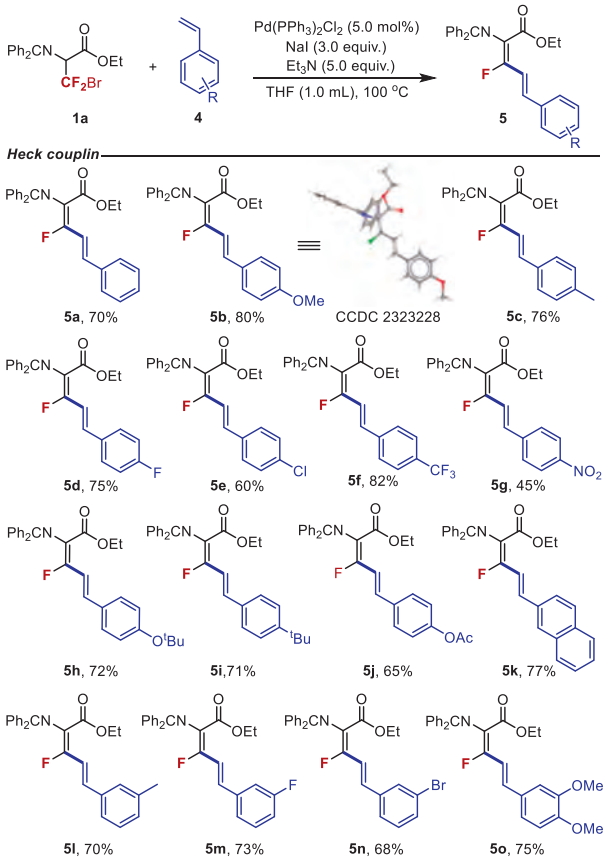


Fig. 3. Substrate scope of Mizoroki-Heck cross coupling. Reaction conditions: **1a** (0.1 mmol), **4** (0.3 mmol), Et_3N (5.0 equiv.), NaI (3.0 equiv.), $Pd(PPh_3)_2Cl_2$ (5.0 mol%), THF (1.0 mL), at 100 °C for 2 h, under argon, isolated yield.

ulate that difluoroalkene amino acids (**10**) may be formed during the reaction under basic conditions. As expected, difluoroalkene amino acid (**10**) was easily isolated by treatment with triethylamine (Scheme 1b-1). However, it was unstable and readily transformed into other compounds. When **10** was used as the substrate instead of **1a**, the product **3a** was isolated in 87% yield (Scheme 1b-2), suggesting that **10** is the key precursor in this newly developed Pd-catalyzed cross-coupling reaction. Due to the rapid progression of the Suzuki coupling reaction, we decided to employ the Heck coupling to monitor the formation of the difluorodehydroalanine intermediate. The reaction progress was monitored at 0.5, 1.0, and 2.0 h using ^{19}F NMR spectroscopy.

The results showed that difluoroalkene **10** formed rapidly, followed by its trans formation into the product (Scheme 1c). To further investigate the reaction pathway, substrates **11** and **13** were prepared. When they were used instead of **1a**, product **12** was not obtained, whereas product **14** was isolated in 42% yield (Scheme 1d) [66]. These findings suggest that the oxidative addition step was assisted by the ester group rather than the nitrogen atom in substrate **1a**, which contributed to high selectivity toward Z-isomer.

Based on the above mechanistic verification and previous reports, we propose the following catalytic cycle (Scheme 2): First, 2-amino-3-fluoropropenoic acid undergoes HBr elimination under basic conditions to produce difluorodehydroalanine **10**. $Pd(dppf)Cl_2$ is reduced to Pd(0) by boric acid, and the Pd(0) complex is selectively added to the C-F bond of the difluoroolefin amino acids with the assistance of ester groups to form complex **II**. This is followed by transmetalation and reductive elimination, yielding the product **3a**, with the Pd complex being reduced back to Pd(0).

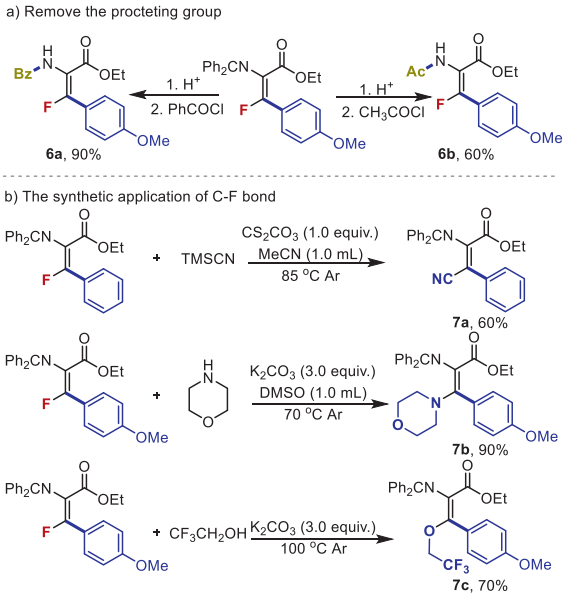


Fig. 4. Structural transformations.

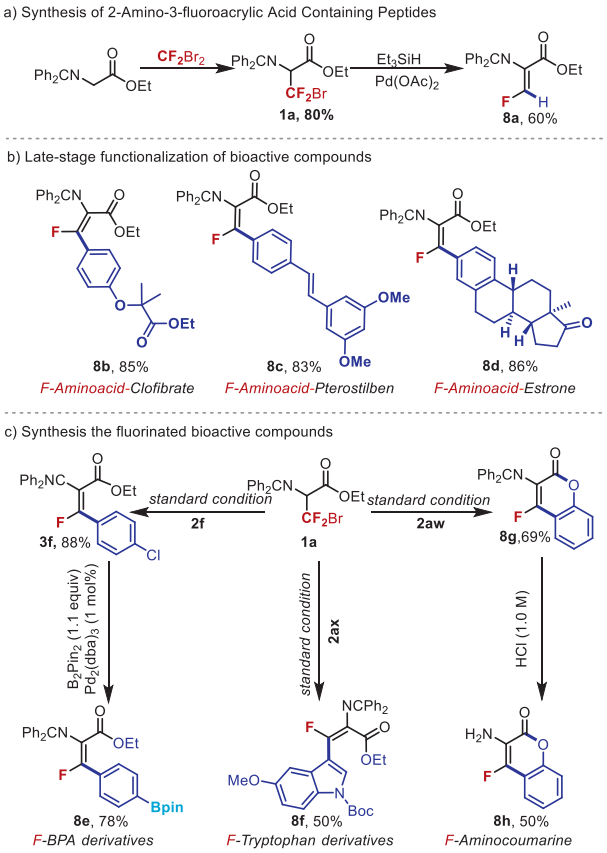


Fig. 5. Synthesis application.

In summary, we have demonstrated that ethyl 3–bromo-2-((diphenylmethylene)amino)-3,3-difluoropropanoate is an effective *gem*–difluoroalkenes precursor, applicable in Suzuki and Heck couplings with palladium catalysts. This reaction provides a straightforward approach for constructing a library of fluorinated amino acids. The novel *gem*–difluoroalkenes precursor not only offer an efficient method for introducing fluorine-containing amino acid

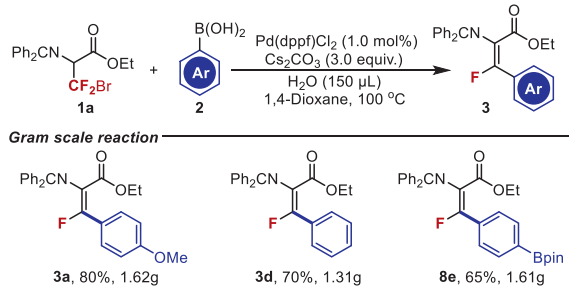
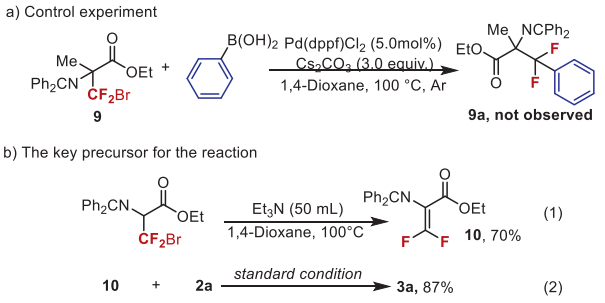
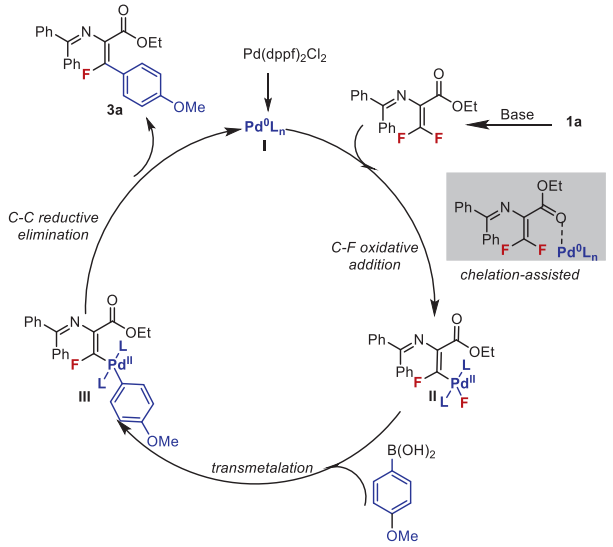


Fig. 6. Synthesis application.



Scheme 1. Mechanistic experiments.



Scheme 2. Plausible catalytic cycle.

moieties into biologically important compounds such as clofibrate, pterostilbene, estrone, and tryptophan but also facilitates the synthesis of fluorinated BPA and aminocoumarin derivatives, further underscoring the synthetic potential of this approach.

Declaration of competing interest

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

CRediT authorship contribution statement

Chuangchuang Liu: Writing – original draft, Validation, Resources, Methodology, Data curation. **Yilian Song:** Validation, Data curation. **Wenjie Ju:** Data curation. **Xin Liu:** Validation. **Min Liang:** Validation. **Yingsheng Zhao:** Writing – review & editing, Funding acquisition, Conceptualization.

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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.2025.111167.

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