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Palladium-catalyzed multi components oxy-aminofluorination and aminofluorination of *gem*-difluoroalkenes

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ABSTRACT

Organofluorine compounds are widely used in the realm of drug discovery and material science. Herein, we developed palladium catalyzed intermolecular aminofluorination and oxy-aminofluorination of *gem*–difluoroalkenes with *N*-fluorobenzenesulfonimide (NFSI), in which NFSI was used as the nitrogen source and oxidant. The reaction provides an efficient and straightforward synthesis route of a series of α-trifluoromethyl benzylic amines. Notably, three/four components oxy-aminofluorination processes were realized to give α-trifluoromethyl benzylic ether with a terminal amino group, which proceed through C(sp^3)–O bond cleavage of easily available ether and simultaneous introduced a fluorine, an amino and an oxy substituent in one pot with excellent regioselectivity. The divergent reactivity not only included the incorporation of one ether molecular, but also much more challenged two ether insertion with excellent selectivity through succession C(sp^3)–O bonds cleavage. This protocol allows for concise synthesis of high value amines with fluoroalkyl-substituents and selectively transformation of easily available ethers by high-valent palladium catalysis.

The incorporation of fluoroalkyl group into organic molecules has been widely applied in pharmaceuticals and agrochemicals development [1–5]. In this context, the synthesis of bioactive molecules with C(sp^3)–CF_3 scaffold is particularly sought after [6–15]. The amine or ether bearing α-CF_3 group is highly attractive for lead candidate study in medicinal chemistry. While, existing methods to give these compounds mainly focus on the nucleophilic addition of trifluoromethyl reagent or transformation of CF_3-substituted imines (ketones) [16–19]. These methods usually need multistep transformations or expensive reagents. An efficient and direct synthetic route is still highly desirable.

In the last two decades, the *gem*–difluoroalkenes have been used as one type of ideal substrates for the synthesis of high-value-added organofluorine compounds [20–23]. However, these reports usually gave the monofluoroalkenes and derivatives *via* β-F elimination by transition-metal catalyst [20–30]. Recently, the fluorine retentive reaction to access functional α-fluoroalkyl containing products has been disclosed [31–43]. The nucleophilic addition of fluoride ion to *gem*–difluoroalkenes is a novel strategy to generate α-CF_3-containing products [35–39], which was applied in the palladium-catalyzed allylation and arylation [40–42]. In 2019, the Malcolmson group developed the synthesis of α-trifluoromethyl benzylic amines by using N-containing *gem*–difluoroalkenes through palladium catalyzed arylation (Scheme 1A) [43]. All of these reactions proceeded through C–C bond formation from α-CF_3 organopalladium intermediate, the C–N or C–O bond formation is still unexplored.

On the other hand, the aminofluorination of simple olefins with transition metal catalysis is a powerful strategy for the synthesis of fluorine-containing amines [44–52]. Liu group developed pioneer and attractive palladium-catalyzed aminofluorination of simple styrenes [53]. The more steric hindrance trifluoromethyl products and the cleavage of C(sp^3)–O bond by high valent Pd species has not been discovered. Later, Zhang group developed the radical aminofluorination by copper catalysis with regioselectivities opposite to that of palladium catalysis (Scheme 1B) [54]. Inspired by these remarkable works and our continuous interest in the synthesis of α-fluoroalkyl containing compounds, herein, we report the palladium catalyzed aminofluorination and multi

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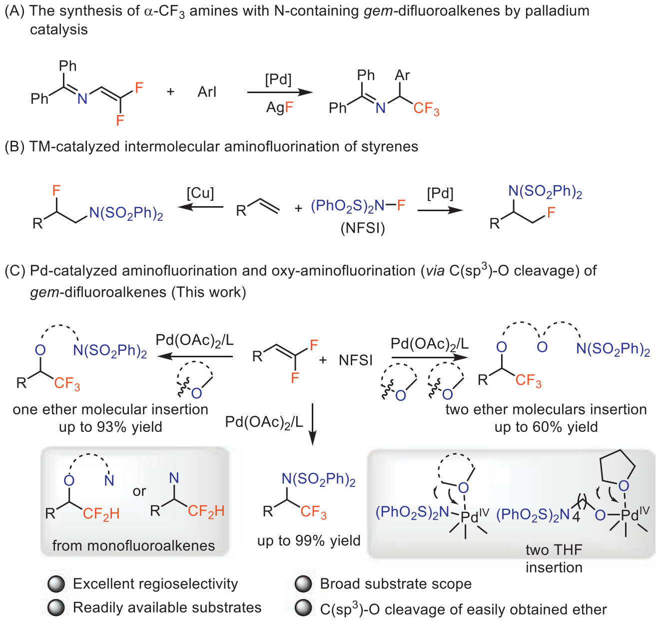
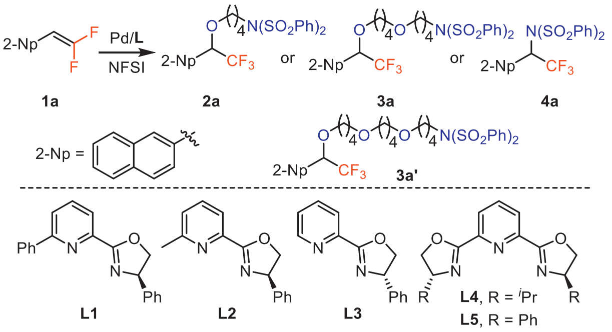


Scheme 1. The synthesis of α-fluoroalkyl containing amines with transition-metal catalyst.

components oxy-aminofluorination of *gem*–difluoroalkenes with *N*-fluorobenzenesulfonimide (NFSI), which was used as the nitrogen source as well as the oxidant, affording various functional α-CF_3 containing amines (Scheme 1C). Noteworthy, the three components oxy-aminofluorination reaction involved the cleavage of C(sp^3)–O bonds in easily available alkyl ethers (such as THF, dioxane, DME), providing the α-CF_3 benzylic ethers bearing terminal amino groups with excellent regioselectivity. We also realized the succession C(sp^3)–O bond cleavage, affording two ethers insertion products with excellent selectivity by addition of Lewis acids, which might be benefit for the coordination of ether to the Pd(IV) center. We suggested that the C(sp^3)–O bond cleavage initiated by the coordination of ether to the Pd(IV) center would be one attractive strategy for the transformation of ethers [55–58]. Additionally, this reaction could access to α-CF_2H containing amines from monofluoroalkenes. The challenge of this catalytic system does not just include the steric hindrance of trifluoromethyl group and β-fluorine elimination, the one pot incorporation of a fluorine, an amino and an oxy substituent with high regioselectivity is also an important synthetic challenge.

We began our studies by examining the reaction parameters of *gem*–difluoroalkene **1a** and NFSI in the presence of Pd$(OAc)_2$ catalysis (Table 1). We found that the ligand **L1** was efficient to give the oxy-aminofluorination product **2a** in 47% yield with high chemoselectivity through C(sp^3)–O cleavage in THF (entry 1). The yield of **2a** could be increased to 75% when NaF was added (entry 2). Very interesting, the more challenged two THF insertion product **3a** could be obtained in 60% yield when $AgSbF_6$ was used as an additive (entry 3, for more details see Table S1 in Supporting information). The addition of $AgSbF_6$ might be favored for the coordination of THF to Palladium center and accelerated the succession cleavage of C(sp^3)–O bond [59–61]. Trace amount of three THF insertion products observed under the reaction conditions of entry 3 (**3a**:**3a'** = 93:7). We next examined the solvent effect and found that the aminofluorination product **4a** was obtained (entries 4–8, for more details see Table S2 in Supporting information). The product **4a** could be obtained in 99% yield when MeO^{t}Bu was used as solvent at 80 °C (entry 4). The DCE gave the comparative result, while DMF and ^{i}PrOH gave no desired product (entries 5–8).

Table 1
Investigation of reaction conditions.[a]

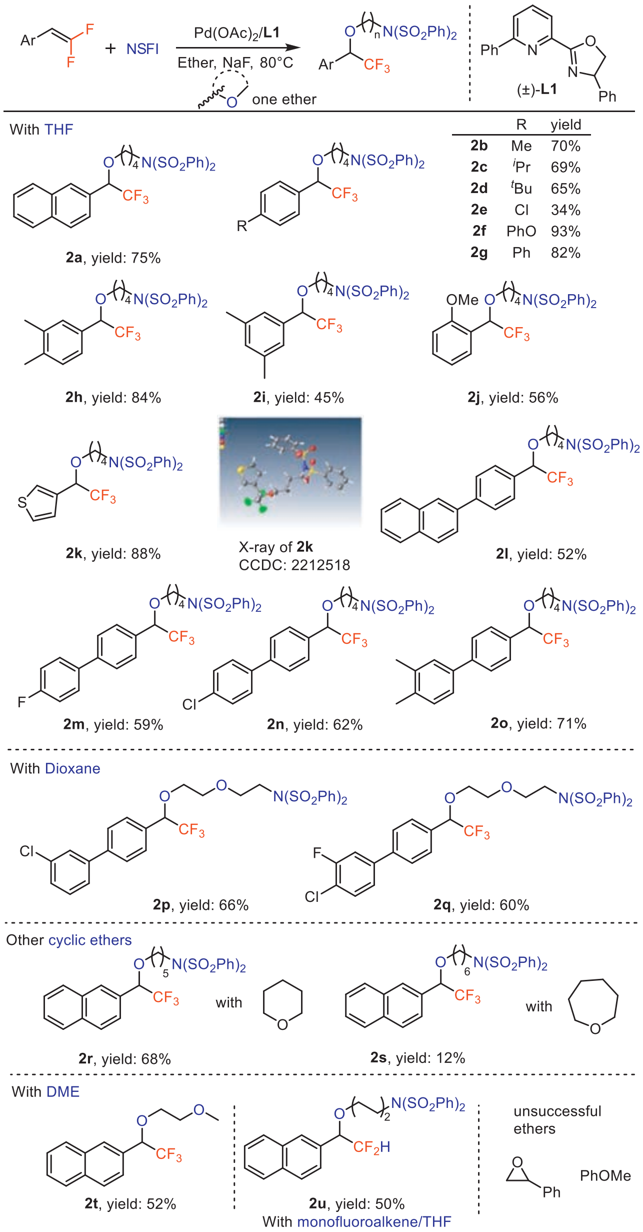


Entry	[Pd]	L	Solvent	Yield (%)		
				2a	3a	4a
1	Pd$(OAc)_2$	**L1**	THF	47	–	3
2[b]	Pd$(OAc)_2$	**L1**	THF	75	–	3
3[c]	Pd$(OAc)_2$	**L1**	THF	3	60	<1
4	Pd$(OAc)_2$	**L1**	MeO^{t}Bu	–	–	99
5	Pd$(OAc)_2$	**L1**	DCE	–	–	98
6	Pd$(OAc)_2$	**L1**	Toluene	–	–	50
7	Pd$(OAc)_2$	**L1**	^{i}PrOH	–	–	–
8	Pd$(OAc)_2$	**L1**	DMF	–	–	–
9	Pd$(OAc)_2$	**L2**	MeO^{t}Bu	–	–	55
10	Pd$(OAc)_2$	**L3**	MeO^{t}Bu	–	–	37
11	Pd$(OAc)_2$	**L4**	MeO^{t}Bu	–	–	25
12	Pd$(OAc)_2$	**L5**	MeO^{t}Bu	–	–	45
13	Pd$(OAc)_2$	PPh_3	MeO^{t}Bu	–	–	2
14	Pd$(OAc)_2$	*rac*-BINAP	MeO^{t}Bu	–	–	9
15	Pd$(OAc)_2$	Bpy	MeO^{t}Bu	–	–	18
16	Pd$(OAc)_2$	1,10-phen	MeO^{t}Bu	–	–	14
17	$PdCl_2$	**L1**	MeO^{t}Bu	–	–	99
18	Pd$(dba)_2$	**L1**	MeO^{t}Bu	–	–	92
19[d]	Pd$(OAc)_2$	**L1**	MeO^{t}Bu	–	–	63
20[e]	Pd$(OAc)_2$	**L1**	MeO^{t}Bu	–	–	99

[a] Reaction conditions: **1a** (0.20 mmol), NFSI (0.40 mmol), Pd$(OAc)_2$ (10 mol%), **L** (10 mol%), solvent (2.0 mL), 80 °C, 24 h, isolated yields.
[b] NaF (0.40 mmol) was added, 36 h.
[c] $AgSbF_6$ (0.04 mmol), hexane/THF (1.5 mL/0.5 mL).
[d] 40 °C.
[e] Pd$(OAc)_2$ (2 mol%), **L1** (2 mol%).

The ligand structure plays an important role to deliver product **4a** (entries 9–16). The $PdCl_2$ and Pd$(dba)_2$ exhibited similar reactivity to the Pd$(OAc)_2$ catalyst (entries 17 and 18). Low yield of **3a** was observed when the reaction proceeded at 40 °C (entry 19). Furthermore, **4a** could be obtained in 99% yield when the Pd catalysis loading decreased to 2 mol% (entry 20). The use of chiral ligands **L1**-**L5** were found with low levels of enantiocontrol (for more details see Tables S4 and S5 in Supporting information).

With the optimized reaction conditions, we first explored the generality of the three components oxy-aminofluorination reaction. To our delight, the substrate scope was found to be very broad, and gave corresponding α-trifluoromethyl benzylic ethers with terminal amino groups in moderate to high yield. As shown in Scheme 2, the reaction was tolerated with an array of substituents at the *para*-, *meta*- or *ortho*-position and produced the corresponding products **2b**-**2j** in 34%–93% yield. The (hetero)aryl-substituted alkenes and biaryl *gem*–difluoroalkenes were also briefly investigated and yielded the oxy-aminofluorination products **2k**-**2o** in 52%–88% yield. The structure of **2k** was confirmed by X-ray crystallography (CCDC: 2215518). Subsequently, we examined C(sp^3)–O cleavage process of other cyclic ethers. Delightedly, when dioxane was used instead of THF, the corresponding α-trifluoromethyl benzylic ether could be obtained in moderate yield through C(sp^3)–O cleavage in dioxane (**2p**, 66% yield and **2q**, 60% yield). The six number ring pyran was also successfully applied in this oxy-



Scheme 2. Substrate scope of oxy-aminofluorination reaction with one ether insertion. Reaction conditions: **1** (0.20 mmol), NFSI (0.40 mmol), $Pd(OAc)_2$ (10 mol%), **L1** (10 mol%), ether (2.0 mL), 80 °C, 36 h, isolated yields.

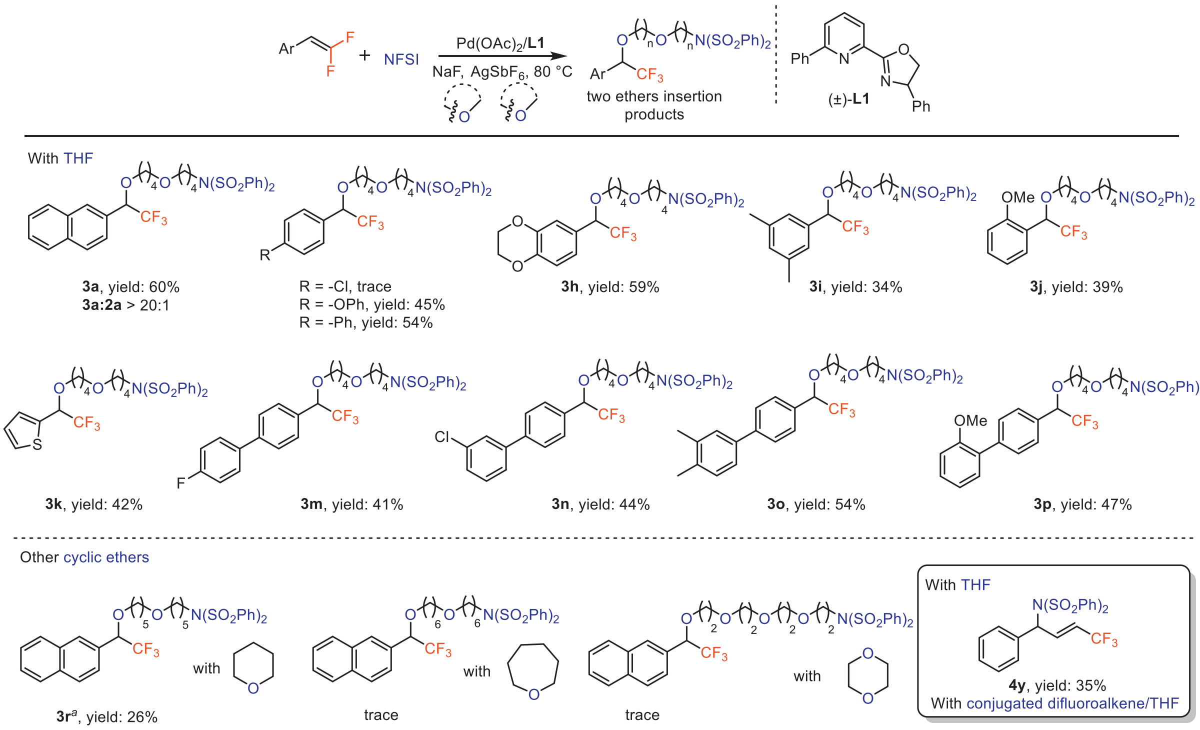
aminofluorination reaction, giving the corresponding product **2r** in 68% yield. Low efficiency for the seven number cyclic ether and gave the corresponding product **2s** in 12% yield. The α-trifluoromethyl benzylic ether without terminal amino group **2t** was obtained in 52% yield when linear ether DME was used instead of cyclic ether. The monofluoroalkene delivered the α-CF_2H benzylic amine **2u** in 50% yield with THF. Other ethers such as 2-phenyloxirane and phenyl methyl ether failed to give desired products.

Then, we investigated the generality of the much more challenged four components oxy-aminofluorination reaction, giving two THF insertion products (Scheme 3). A series of functional *gem*–difluoroalkenes with electron-donating groups were well tolerated to give two THF insertion products with high selectivity through the succession double C(sp^3)–O cleavage (**3a**, 60% yield, **3f**, 45% yield, **3g**, 54% yield and **3h**, 59% yield). Trace amount of desired product observed for the Cl-substituted alkene. The substituents at *meta*- or *ortho*-position and the (hetero)aryl-substituted alkenes were compatible (**3i-3k**, 34%–42% yield). Some biaryl *gem*–difluoroalkenes were briefly investigated (**3m-3p**, 41%–54% yield). Other cyclic ether such as pyran was successfully to deliver the desired two ether insertion product (**3r**, 26% yield). No desired products obtained when dioxane or seven number ether were used. The conjugated *gem*–difluoroalkene only gave the aminofluorination product **4y** in 35% yield.

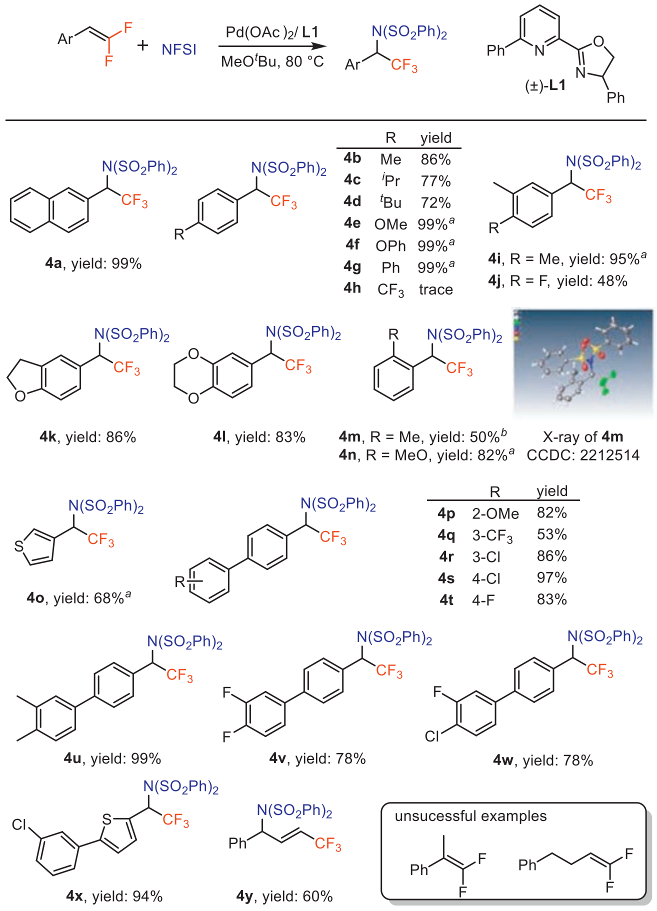
Finally, we turned our attention to study the substrate scope of aminofluorination reaction (Scheme 4). The reaction also tolerated with various functional groups at the *para*-position of the benzene ring of *gem*–difluoroalkenes (**4b-4g**, 72%–99% yield). The reaction showed high efficiency for the electron rich substrates. While the electron-withdrawing substituted alkene (CF_3 group at *para*-position) could not give the desired product (**4h**), only with starting materials recovered. Low reactivity might be due to the coordination of electron-deficient alkene to low-valent palladium, which would stabilize the low oxidation state and not benefit for the generation of high-valent Pd(IV) species [62,63]. The alkenes with substituents at the *meta*-position were also tolerated to yield the α-trifluoromethyl benzylic amines **4i-4l** in 48%–95% yield. The *ortho*-substituted alkenes with methyl group needed higher temperature to produce the amine product **4m**, possibly due to steric effect. While, the electron-rich alkene with MeO-group at the *ortho*-position delivered the product **4n** in 80% yield even with 2 mol% Pd catalysis loading. The structure of **4m** was confirmed by X-ray crystallography (CCDC: 2212514). (Hetero)aryl-substituted alkene gave the corresponding products **4o** in 68% yield. Furthermore, the biaryl *gem*–difluoroalkenes with electron-donating groups, electron-withdrawing groups or halo-groups at the benzene ring worked smoothly to deliver the products **4p-4x** in 53%–99% yield. The conjugated *gem*–difluoroalkene was also compatible to afford the trifluoromethylated allylic amine **4y** in 60% yield with excellent regioselectivity. However, the reaction of (1,1-difluoroprop-1-en-2-yl)benzene or (4,4-difluorobut-3-en-1-yl)benzene all failed to give the desired products, only with starting materials recovered.

The CF_3-containing amine products could undergo several transformations (Scheme 5). These reactions were successfully scaled up to 2.0 mmol without the erosion of yield, giving product **2a** in 74% yield and **4a** in 99% yield (Scheme 5A). The α-trifluoromethyl ether **2a** could be reduced to product **5** in 84% yield. The α-trifluoromethyl benzylic amine **4a** also could be reduced to product **6** in 85% yield. Furthermore, the primary amine **7** was obtained in 65% yield and could be combined with aldehyde *via* reductive amination to obtain the product **8** in 53% yield (Scheme 5B).

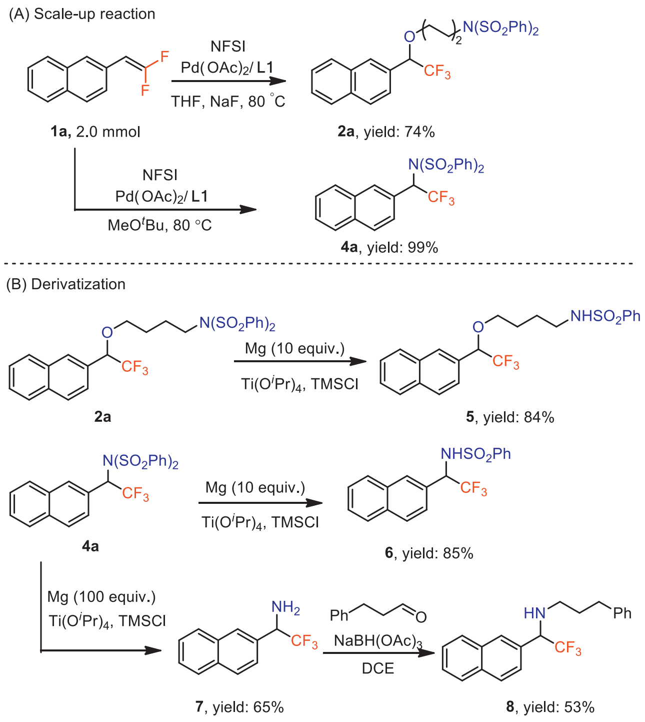
We then performed several experiments to investigate the influence of substituent in the alkene and ether (Scheme 6). When the simple styrene was used, only starting materials were recovered with Pd/**L1** catalysis. The reaction worked smoothly for the monofluoroalkene **(*Z*)–9**, affording the α-difluoromethyl benzylic amine products **10** in 90% yield. The monofluoroalkene **(*E*)–9** was also successfully converted to **10** in 87% yield. These results indicated the importance of fluorine atom at the alkenes, but irrelevant with the alkene configuration. When methyl *n*–butyl ether was used instead of cyclic ether, α-trifluoromethyl benzylic ether **11** was obtained in 24% yield and **12** was obtained in 8% yield. This result suggested that the C(sp^3)–O cleavage prior occurred at the position with small steric hindrance substituent. When different amount of THF was added in MeO^tBu solvent, the THF insertion product was the major product after 50 equiv. THF was added (for more details see Table S3 in Supporting information). Several kinetic studies of the reaction of *gem*–difluoroalkene **1a**, NFSI, [Pd] catalysis and ligand effect by the method of initial rates were per-



Scheme 3. Substrate scope of oxy-aminofluorination reaction with two ether insertion. Reaction conditions: **1** (0.20 mmol), NFSI (0.40 mmol), $Pd(OAc)_2$ (10 mol%), **L1** (10 mol%), $AgSbF_6$ (0.02 mmol), hexane (1.5 mL), ether (0.5 mL), 80 °C, 15 h, isolated yields. [a] $PdCl_2$ (10 mol%) was used instead of $Pd(OAc)_2$.

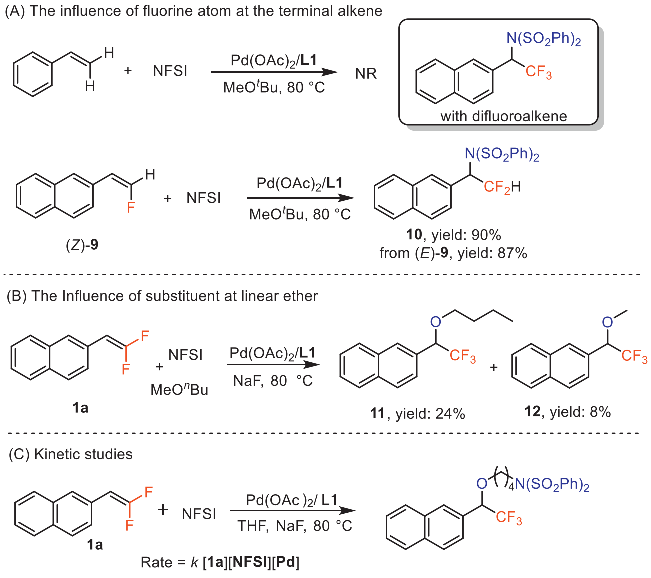


Scheme 4. Substrate scope of aminofluorination reaction. Reaction conditions: **1** (0.20 mmol), NFSI (0.40 mmol), $Pd(OAc)_2$ (10 mol%), **L1** (10 mol%), MeO^tBu (2.0 mL), 80 °C, 24 h, isolated yields. [a] $Pd(OAc)_2$ (2 mol%), **L1** (2 mol%). [b] $Pd(OAc)_2$ (10 mol%), **L1** (10 mol%), 48 h, 120 °C.

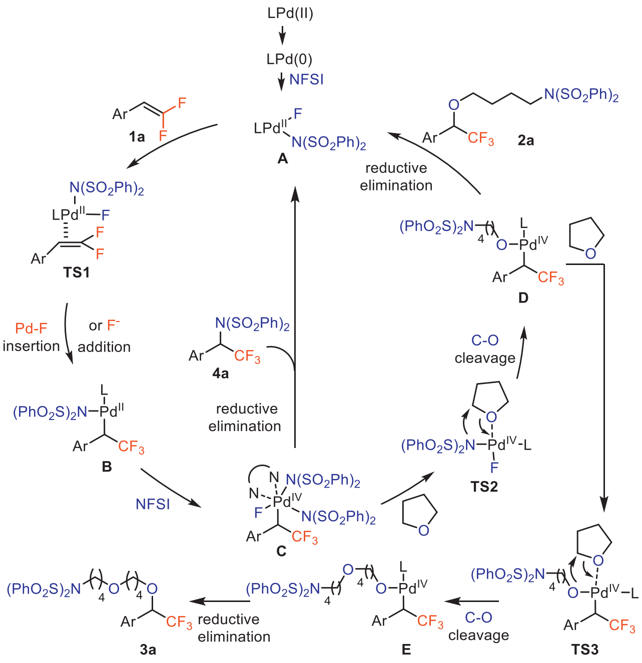


Scheme 5. Scale-up reaction and derivatization.

formed (Scheme 6C). This reaction exhibited a first-order dependence on the concentration of *gem*–difluoroalkene **1a**, [Pd] catalysis and NFSI (for more details in Supporting information). Notably, these kinetic data are consistent with *gem*–difluoroalkene into Pd(II)–F, but are inconsistent with the Pd(IV)–F insertion [53,64–66].



Scheme 6. Mechanistic studies. (a) Influence of substituents at the alkenes. (b) Influence of substituents at the ether. (c) Kinetic studies.



Scheme 7. Proposed mechanism.

Based on these above results and previous studies in palladium-catalyzed aminofluorination of alkenes [44–52,64–72], we proposed a plausible mechanism in Scheme 7. The reaction was initiated by the generation of Pd(II) intermediate **A**. After the coordination of *gem*–difluoroalkene **1a** to the Pd(II)–F species (**TS1**), Pd(II)–F inserted or F$^-$nucleophilic addition to the **1a** and formed the intermediate **B**, which would undergo oxidative addition of intermediate **B** to NFSI and gave the Pd(IV) species **C**. The oxidation of intermediate **B** to give the Pd(IV) intermediate **C** is consistent with our kinetic studies in Scheme 6C. The reductive elimination from the intermediate **C** delivered the α-trifluoromethyl benzylic amine product **4a**. When the cyclic ether was used, the cyclic ether (THF) coordinated to the Pd(IV) center and the C(sp^3)–O bond cleavage occurred to form the Pd(IV)–O species from Pd(IV)–N species intermediate **C** (**TS2**) and gave the intermediate **D**, which would undergo reductive elimination to give the α-trifluoromethyl benzylic ether product **2a**. Alternatively, another THF would coordinated to the Pd(IV) center (**TS3**) and C(sp^3)–O bond cleavage from the Pd(IV)–O species to give the intermediate **E**, which undergo reductive elimination to deliver the two THF insertion product **3a**.

In summary, we have developed a novel palladium-catalyzed aminofluorination and three/four components oxy-aminofluorination of *gem*–difluoroalkenes, in which the NFSI was served not only as the fluorine and nitrogen source but also the oxidant. The reaction proceeded with excellent regioselectivity and atom economy, affording a variety of functionalized α-trifluoromethyl benzylic amines and ethers. Particularly, the intermolecular oxy-aminofluorination simultaneously introduced a fluorine, an amino and an oxy substituent in one pot through C(sp^3)–O bonds cleavage of the easily available ethers. We not only realized one ether insertion process, but also two ether insertion process through succession C(sp^3)–O bonds cleavage. This study contributed to the rapid and divergent synthesis of α-fluoroalkyl containing compounds, and provided insight for further development of functionalization of olefins.

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.2023.109155.

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