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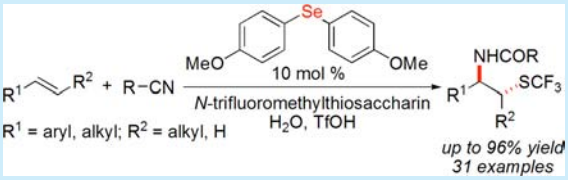
Diaryl Selenide Catalyzed Vicinal Trifluoromethylthioamination of Alkenes

Jie Luo, Zechen Zhu, Yannan Liu, and Xiaodan Zhao*

Institute of Organic Chemistry & MOE Key Laboratory of Bioinorganic and Synthetic Chemistry, School of Chemistry and Chemical Engineering, Sun Yat-Sen University, Guangzhou 510275, P. R. China

Supporting Information

ABSTRACT: An efficient approach to vicinal trifluoromethylthioamination of alkenes with a broad substrate scope catalyzed by electron-rich diaryl selenide has been developed. This intermolecular amination strategy was successfully applied to SCF_3-esterification of alkenes using weak acids as nucleophiles.

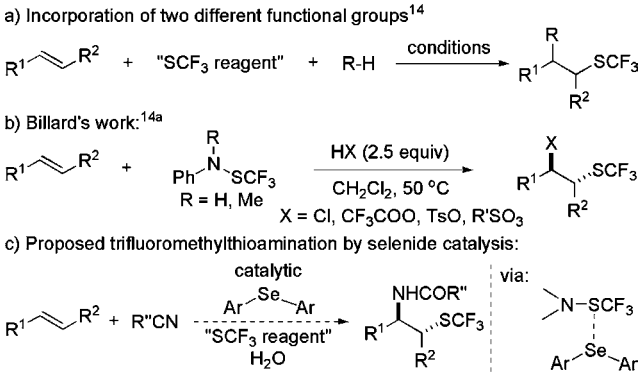


The trifluoromethylthio group ($-SCF_3$) possesses strong electron-withdrawing properties and high lipophilicity (π_R = 1.44).[1] The incorporation of this group into organic molecules can lead to an increase in their bioavailability, which is of great interest to the field of agrochemicals and pharmaceuticals.[2] In the past several years, much effort has been dedicated to the development of efficient approaches to the SCF_3-functionalization of parent substrates, especially with SCF_3 reagents.[3] For instance, transition metal catalyzed or metal-free trifluoromethylthiolation of halides,[4] boronic acids,[5] C−H bonds,[6] alcohols,[7] alkynes,[8] organometallic species,[9] and so on[10] has been documented with stable nucleophilic or electrophilic trifluoromethylthiolating reagents. Recently, the Ruppert-Prakash reagent ($TMSCF_3$) was employed to generate SCF_3 in situ for the trifluoromethylthiolation of alkynes and arene diazonium salts.[11,12] However, these previous examples generally afford only SCF_3-functionalized products.

The difunctionalization of alkenes is an efficient strategy for constructing valuable molecules.[13] When SCF_3 and another group are added onto alkenes simultaneously by multicomponent coupling, SCF_3-containing drug candidates might be easily generated, which could provide a great opportunity for medicinal chemists (Scheme 1a).[14] This way of creating two or more functional group-containing compounds in one step is significantly superior to the methods of multistep synthesis. In 2009, Billard presented an elegant trifluoromethylthiolation of alkenes using SCF_3^+ formed by stoichiometric acid activation of an electrophilic SCF_3 reagent.[14a] Functional groups such as Cl−, CF_3COO−, and sulfonyl could be incorporated into substrates along with the SCF_3 group (Scheme 1b). Unfortunately, SCF_3-amination of such substrates cannot be achieved due to the amine-protonation problem by a strong acid. In view of the clear importance of amine-containing compounds,[15] a solution to the SCF_3-amination of alkenes is an attractive goal.

In the past few years, the use of chalcogenides as Lewis base catalysts to activate N−SPh/halo bonds has received attention.[16] Although the N−SCF_3 bond is unique, and quite different from other N−X bonds, its activation by chalcogenides has never been demonstrated. In this connection, selenide was conceived as being capable of activating this bond to generate SCF_3^+ by adjusting its electron-donating ability. Then SCF_3^+ could react with the double bond of alkenes to form a trifluoromethylthiiranium ion, which could be ring-opened by a nitrogen source to give SCF_3-amination products. Nitriles are readily available and could potentially be utilized as such a nitrogen source in Ritter-type reactions.[17] We thus proposed that a SCF_3-amination of alkenes with electrophilic N−SCF_3-containing reagents and nitriles might be attainable by selenide catalysis (Scheme 1c). Herein, we report our discovery of the diaryl selenide-catalyzed SCF_3-amination of unactivated alkenes.

Scheme 1. Construction of SCF_3-Containing Molecules via Multicomponent Reactions



The change of aryl group can adjust the electron-donating ability of selenide.

We initiated our study of SCF_3-amination with styrene (**1a**) as the model substrate. Stable Billard SCF_3 reagents **A** and **B** and Munavalli's *N*-trifluoromethylthiophthalimide (**C**)[18] were first tried as the trifluoromethylthiolating reagents. The multicomponent reaction was carried out in the presence of

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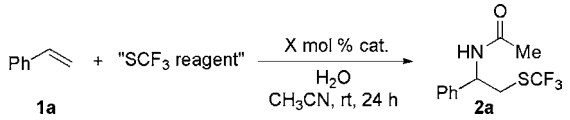
20 mol % of PhSePh, CH_3CN, and water at room temperature for 24 h. The desired product **2a** was not observed (Table 1, entry 1). When the more reactive SCF_3 reagent, Shen's *N*-trifluoromethylthiosaccharin (**D**),[7b] was utilized, trace product was formed, which proved that our proposal worked (Table 1, entry 2). To enhance the catalytic activity of selenide, the electron-donating ability of the selenide was adjusted by making changes to the aryl group. Bulky **C1** and **C2** with an electron-withdrawing CF_3 group were prepared and tested in these reactions, but did not advance the yield (Table 1, entries 3 and 4). To increase the electron density on the selenium atom, a methoxy group was introduced onto the catalysts. Pleasingly, **2a** was formed in 36% yield using 20 mol % of **C3** as the catalyst with reagent **D** (Table 1, entry 5). When more electron-rich selenide **C4** was employed as the catalyst, the yield was improved to 42% (Table 1, entry 6). In contrast, no reaction was observed with electrophilic SCF_3 reagents **A**, **B**, and **C** (Table 1, entry 7). The selenides with three or more methoxy groups were less efficient than **C4**. Acid could activate electrophilic SCF_3 reagents in some cases and was expected to cooperatively activate the N−SCF_3 bond with selenides. When 1 equiv of methanesulfonic acid (MsOH) was added to the reaction, the yield jumped to 56% (Table 1, entry 8). With TfOH, a 91% isolated yield was obtained using 10 mol % of **C4** as the catalyst (Table 1, entry 10). It is worthy to note that the SCF_3-amination proceeded smoothly with a strong acid present. Possibly it could be neutralized by the acetonitrile to diminish the acidity of the reaction system.[19] A lower loading of TfOH resulted in a lower yield of **2a** (Table 1, entry 11). In the absence of selenide, the reaction gave no product (Table 1, entry 12). Under acidic conditions, different catalysts were also tested. PhSePh, PhSeSePh, and **C3** served as catalysts and produced the product in lower yields, while others, i.e., $Ph_3P{=}Se$, sulfide, disulfide, **C1**, and **C2**, resulted in much lower yields (see Supporting Information). The above fact indicates that electron-rich catalysts greatly contribute to the efficiency of the reaction.

In an effort to reduce the amount of nitrile used, we found that **2a** could be obtained in almost the same yield by the reaction of **1a** with 20 equiv of CH_3CN in hexane as the solvent. When the loading of CH_3CN was reduced to 10 equiv and 3 equiv, **2a** was still produced in 81% and 42% yields, respectively. Alkyl and aryl nitriles were examined under similar conditions (Scheme 2). The desired difunctionalized products were formed in 54−94% isolated yields. In general, alkyl nitriles gave the desired products in higher yields than aryl nitriles (**2b**−**2g** vs **2h**−**2m**). Sterically hindered pivalonitrile was still effective at forming the desired product **2b** in 78% yield. Different functional groups, i.e., Cl− and EtO−, on alkyl nitriles were tolerated in the reactions (**2f** and **2g**). Aryl nitrile with an electron-donating group (e.g., MeO−) underwent SCF_3-amination smoothly (**2l**, 76%), but aryl nitrile with an electron-withdrawing group (e.g., F−) led to a moderate yield (**2m**, 54%). When solid nitriles were employed as starting materials, dichloroethane was added to the reactions in order to enhance their solubility in solvent.

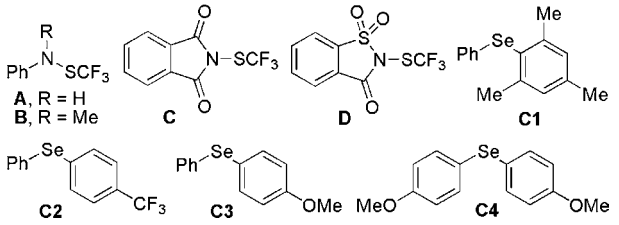
We next turned our attention to exploring the alkene scope with CH_3CN under similar conditions (Scheme 3). The substrate scope is broad. Most alkenes delivered the corresponding products in satisfactory isolated yields. Styrene

Table 1. Condition Optimization[a]

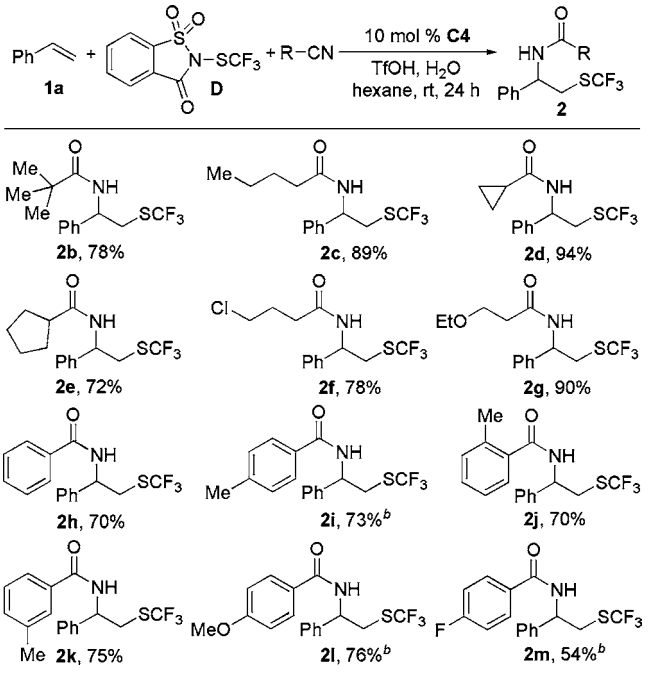


entry	SCF_3 reagent	catalyst/X (mol %)	acid[b]	yield (%)[c]
1	**A**, **B**, or **C**	PhSePh/20	–	0
2	**D**	PhSePh/20	–	trace
3	**D**	**C1**/20	–	trace
4	**D**	**C2**/20	–	0
5	**D**	**C3**/20	–	36
6	**D**	**C4**/20	–	42
7	**A**, **B**, or **C**	**C4**/20	–	0
8	**D**	**C4**/20	MsOH	56
9	**D**	**C4**/20	TfOH	94
10	**D**	**C4**/10	TfOH	94(91)
11[d]	**D**	**C4**/10	TfOH	85
12	**D**	none	TfOH	0

[a]Conditions: styrene (0.05 mmol), SCF_3 reagent (1.3 equiv), H_2O (1.2 equiv), CH_3CN (1.0 mL), room temperature, 24 h. [b]Acid: 1 equiv. [c]Refers to NMR yield using 1,3,5-trimethoxybenzene as the internal standard; isolated yield is in parentheses in 0.1 mmol scale. [d]TfOH: 0.5 equiv.



Scheme 2. Reactions with Alkyl and Aryl Nitriles as the Aminating Reagent[a]



[a]Conditions: styrene (0.1 mmol), **D** (1.3 equiv), **C4** (10 mol %), RCN (20 equiv), TfOH (1.0 equiv), H_2O (1.2 equiv), hexane (1.0 mL), room temperature, 24 h. [b]Mixed solvents containing 0.9 mL of hexane and 0.1 mL of dichloroethane were utilized.

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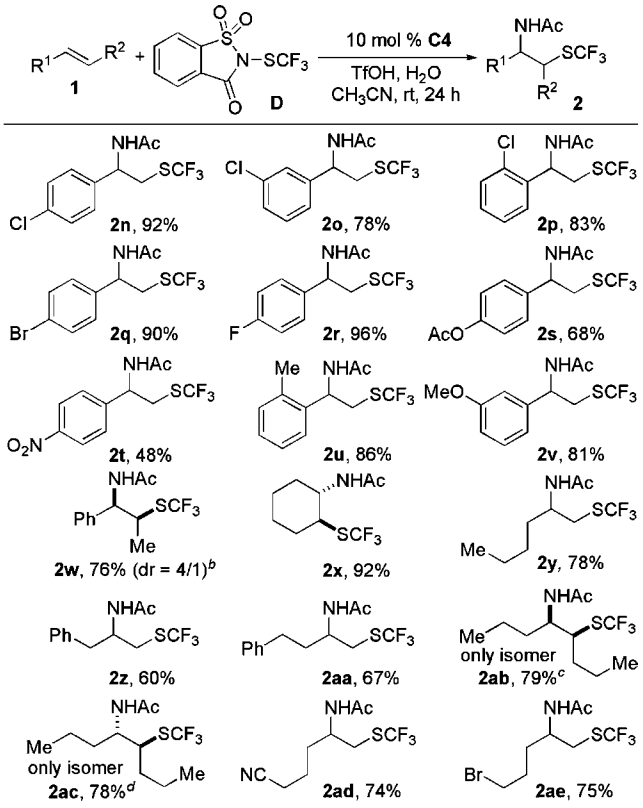
Scheme 3. Reactions with Different Alkenes[a]



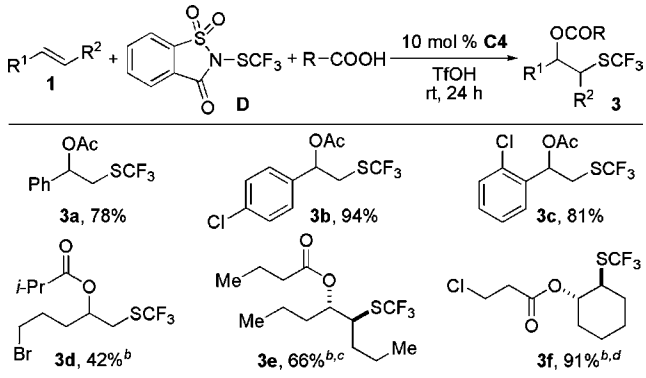
[a]Conditions: alkene (0.1 mmol), **D** (1.3 equiv), **C4** (10 mol %), TfOH (1.0 equiv), H_2O (1.2 equiv), CH_3CN (1.0 mL), room temperature, 24 h. [b]From β-methylstyrene (E/Z = 4:1). [c]From *E*-oct-4-ene. [d]From *Z*-oct-4-ene.

derivatives with various functional groups on aromatic rings, i.e., Cl−, Br−, F−, AcO−, Me−, and MeO−, smoothly led to products (**2n**−**2s**, 68%−96%; **2u**, 86%; **2v**, 81%). When electron-deficient *p*-nitrostyrene was utilized as the substrate, the desired product **2t** was obtained in moderate yield. The reaction of *p*-methyl or *p*-methoxystyrene with **D** generated multiple products. The SCF_3-amination proceeded with excellent stereoselectivity when aliphatic internal alkenes were used. For example, only *trans* **2x** was obtained with cyclohexene, and **2ab**, **2ac** could be formed from the corresponding *E*-oct-4-ene and *Z*-oct-4-ene in what appeared to be a stereospecific transformation. Functional groups, i.e., NC− and Br−, on alkyl alkenes were tolerated under the conditions (**2ad**, 74% and **2ae**, 75%). Regioselective products were observed always in different ratios with alkyl terminal alkenes.

Billard mentioned that weak acids such as AcOH were not capable of activating SCF_3 reagent **A** or **B**. Therefore, no SCF_3-esterification product was observed.[14a] Satisfyingly, when our method was applied to the reaction of alkenes with **D** and weak acids, the corresponding SCF_3-esterification products were obtained in 42−94% yields (Scheme 4). Both aryl and alkyl alkenes worked well under similar conditions. The products, i.e., *trans*-**3e** and *trans*-**3f**, were formed with excellent stereoselectivity. The functional groups, e.g., Br− and Cl−, were tolerated in the reactions. Similar to SCF_3-amination, regioselective products were observed with alkyl terminal alkenes.

To elucidate the role of the acid and diaryl selenide in these reactions, ^{19}F NMR studies were conducted with acetonitrile-

Scheme 4. Trifluoromethylthioesterification of Alkenes[a]

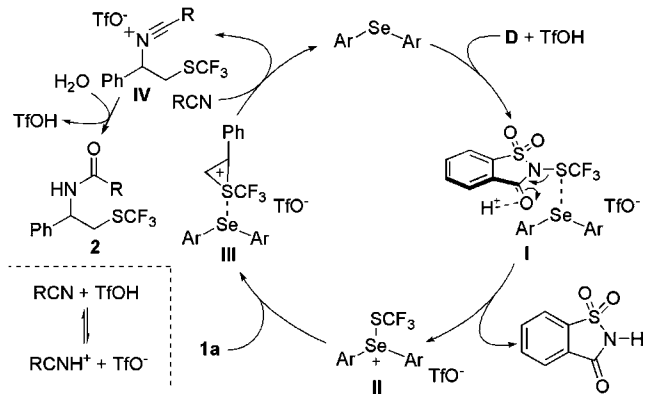


[a]Conditions: alkene (0.1 mmol), **D** (1.3 equiv), **C4** (10 mol %), TfOH (1.0 equiv), AcOH (1.0 mL), room temperature, 24 h. [b]R-COOH (20 equiv); hexane (1.0 mL) was used as the solvent. [c]From *Z*-oct-4-ene. [d]Mixed solvents containing 0.9 mL of hexane and 0.1 mL of dichloroethane were utilized.

d3: (a) when TfOH reacted with 1.3 equiv of **D** for 24 h, 100% of **D** remained; (b) when 1 equiv of PhSePh reacted with 1.3 equiv of **D** for 5 min, some decomposition of **D** was observed (5%). When the reaction time was prolonged to 48 h, more decomposition of **D** was observed (36%). When PhSePh was replaced with electron-rich **C4**, 94% of **D** decomposed in 48 h, which was consistent with the results in Table 1. (c) When 1 equiv of PhSePh and 1 equiv of TfOH were mixed with 1.3 equiv of **D** for 5 min, 30% of **D** decomposed. After 2 h, most of **D** was consumed. Then 1 equiv of **1a** and H_2O were added to the resulting solution, but no SCF_3-amination product was observed, which indicates that SCF_3^+ is highly active leading to easy decomposition without styrene in the reaction. The above control experiments reveal that acid could accelerate the activation of the N−SCF_3 bond resulting in quick decomposition of the SCF_3 reagent.

A reasonable mechanism for this SCF_3-amination is proposed that accords with past literature[16] and our ^{19}F NMR studies (Scheme 5). Diaryl selenide first reacts with **D** in the presence

Scheme 5. Proposed Mechanism



of TfOH to give intermediate **II** and saccharin through transition state **I**. Since an equilibrium is established between the reactants TfOH, CH_3CN, and the adduct, the correct amount of TfOH is required to provide the right acidity. Protonation assists diaryl selenide attack and activation of the N−SCF_3 bond leading to the formation of **II**. The species **II** is

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highly active and quickly reacts with **1a** to generate the trifluoromethylthiiranium ion **III**. It is then attacked by nitrile to give **IV** through an *anti* addition involving the opening of the iranium intermediate, which undergoes hydrolysis to produce the product **2**.

In summary, we have reported the vicinal SCF_3-amination of alkenes by multicomponent coupling. The transformation was efficiently completed by electron-rich diaryl selenide catalysis and has a broad substrate scope. The developed method was successfully applied to the SCF_3-esterification of alkenes. Catalysis by diaryl selenide is mechanistically distinct from that by simple acids and provides differential opportunities for new reactivity. Development of an asymmetric version of this SCF_3-amination of alkenes is ongoing in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

Experimental details, characterization data, and NMR spectra of new compounds. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b01727.

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: zhaoxd3@mail.sysu.edu.cn.

Notes

The authors declare no competing financial interest.

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