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Generation of (*E*)-β-trifluoromethyl vinylsulfonohydrazides under photocatalysis and their anti-bacteria activity

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ABSTRACT

Trifluoromethylation/sulfonylation of alkynes from trifluoromethyl thianthrenium triflate and sulfur dioxide under extremely mild reaction conditions provides a facile access to trifluoromethyl-substituted vinyl sulfonohydrazides in moderate to good yields. This multicomponent reaction of trifluoromethyl thianthrenium triflate, alkynes, sulfur dioxide and hydrazines proceeds efficiently under visible light irradiation in the presence of photocatalyst at room temperature with broad substrate scope and excellent functional group compatibility. This reaction is highly stereoselective, and only (*E*)-isomers are obtained. Additionally, these trifluoromethyl-substituted vinyl sulfonohydrazides are further evaluated for anti-bacteria activity. *In vitro* activities of these compounds against *Staphylococcus aureus* (G^+) and *Escherichia coli* (G^-) are examined.

It is well established that compounds with substitution of fluorine might have a higher stability against metabolic enzymes and a better membranous permeability [1–10]. Therefore, introducing a trifluoromethyl group into small molecule is highly desirable. Recently, trifluoromethyl thianthrenium triflate (TT-$CF_3^+OTf^-$) developed by Ritter and co-workers as a novel trifluoromethylating reagent has attracted much attention [11]. This trifluoromethylating reagent can be easily synthesized *via* a single step from thianthrene and triflic anhydride. So far, the application of TT-$CF_3^+OTf^-$ in electrophilic, radical, and nucleophilic trifluoromethylation reactions has been demonstrated [12,13]. For instance, synthesis of C^α-tetrasubstituted α- and β-amino acid analogues *via* a radical conjugate addition of a trifluoromethyl thianthrenium salt to Michael acceptors with acetonitrile could be achieved [14]. During the transformation, α-thianthrenium carbonyl species as the equivalent of an α-carbonyl carbocation was the key intermediate. As part of a program for the generation of trifluoromethyl-substituted small molecules for biological evaluations, we are interested in methodologies development by using trifluoromethyl thianthrenium triflate (TT-$CF_3^+OTf^-$) as the trifluoromethylating reagent.

It is known that natural products and pharmaceuticals containing sulfonyl group exhibit remarkable biological activities [15–20]. For example, vinyl sulfones have been identified as anti-Gram-positive bacteria as SrtA transpeptidase inhibitors [21], antiparasitic as cysteine protease inhibitors [22], and anti-virus as potent inhibitors of HIV-1 integrase [23]. Additionally, many sulfonyl compounds are marketed drugs. In the past decade, generation of sulfones and sulfonamides by using sulfur dioxide surrogates as the source of sulfonyl group has attracted much attention [24–51]. We also focus on the chemistry of sulfur dioxide insertion for the preparation of diverse sulfonyl compounds from the sulfur dioxide surrogates of DABCO·$(SO_2)_2$ and inorganic sulfites [52–66]. Encouraged by the recent advance of trifluoromethyl thianthrenium triflate (TT-$CF_3^+OTf^-$) [1–13], we envisioned that trifluoromethyl group and sulfonyl group could be incorporated in one molecule by using trifluoromethyl thianthrenium triflate (TT-$CF_3^+OTf^-$) as the trifluoromethylating reagent and sulfur dioxide as the sulfonyl source. It was reported that compounds containing an α,β-unsaturated vinyl sulfone moiety exhibited modest inhibitory potencies in inflammation as a novel class toward Parkinson's [67] and arthritis [68] disease therapy by depressing the expression of endothelial cells of adhesion molecules. Additionally, antitumor activity of trifluoromethyl-substituted vinyl sul-

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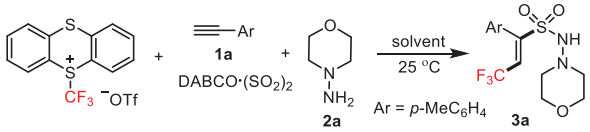
Table 1
Initial studies for the reaction of trifluoromethyl thianthrenium triflate, 4-methylphenylacetylene **1a**, DABCO·$(SO_2)_2$ and morpholin-4-amine **2a**. [a]

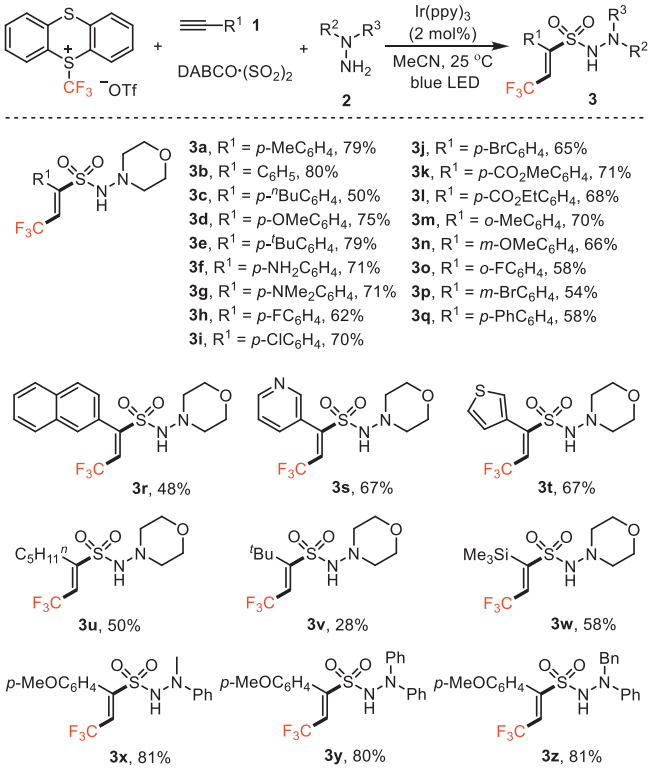


Entry	PC	Solvent	Yield (%)[b]
1	-	MeCN	40
2[c]	-	MeCN	10
3	$Ir(ppy)_3$	MeCN	92 (79)[d]
4	$Ru(bpy)Cl_2·6H_2O$	MeCN	72
5	4CzIPN	MeCN	85
6	EosinY	MeCN	75
7	$Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$	MeCN	84
8	$Ir(ppy)_3$	DCE	65
9	$Ir(ppy)_3$	DMF	34
10	$Ir(ppy)_3$	DMSO	46
11	$Ir(ppy)_3$	THF	49
12	$Ir(ppy)_3$	1,4-Dioxane	68
13	$Ir(ppy)_3$	EtOH	26

[a] Unless otherwise noted, reaction conditions are as follows: alkyne **1a** (0.2 mmol), TT-$CF_3^+OTf^-$ (0.3 mmol), morpholin-4-amine **2a** (0.4 mmol), DABCO·$(SO_2)_2$ (0.16 mmol), photocatalyst (0.004 mmol), solvent (4 mL), 36 W blue LEDs, under a N_2 atmosphere.
[b] Yield determined by ^1H NMR analysis using dibromomethane an internal standard.
[c] In the dark.
[d] Isolated yield.

fones was discovered through activation of autophagy by suppression PI3K/Akt/mTOR signal pathway [13]. Therefore, we conceived that a small library of trifluoromethyl-substituted vinyl sulfonyl compounds may be constructed, which is beneficial for their further biological evaluations. Herein, we report the synthesis of trifluoromethyl-substituted vinyl sulfonohydrazides through a four-component reaction of alkynes, trifluoromethyl thianthrenium triflate (TT-$CF_3^+OTf^-$), DABCO·$(SO_2)_2$ and hydrazines under extremely mild conditions. This transformation proceeds smoothly under visible light irradiation in the presence of photocatalyst at room temperature. Additionally, *in vitro* activities of these trifluoromethyl-substituted vinyl sulfonohydrazides against *Staphylococcus aureus* (G^+) and *Escherichia coli* (G^-) are further evaluated.

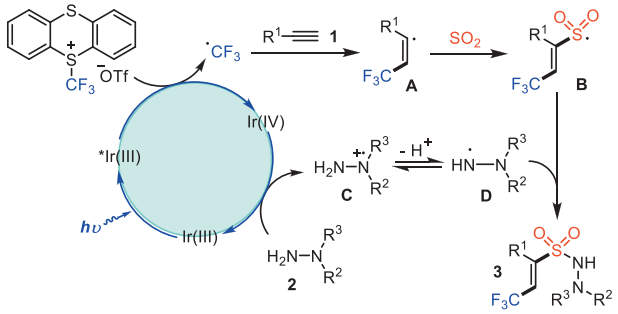
To verify the practicability of our hypothesis, commercially available hydrazine was selected as the partner for reaction development. At the outset, a reaction of trifluoromethyl thianthrenium triflate, 4-methylphenylacetylene **1a**, DABCO·$(SO_2)_2$ and morpholin-4-amine **2a** was explored as the model for condition optimization (Table 1). Initially, the reaction was performed at 25 °C in MeCN. To our delight, the desired trifluoromethyl-substituted vinyl sulfonohydrazide **3a** was generated in 40% yield (Table 1, entry 1). However, the outcome could not be improved when other solvents were used (data not shown in Table 1). The yield was lower when the reaction occurred in the dark (Table 1, entry 2). From this result, we reasoned that the presence of visible light irradiation might facilitate the formation of trifluoromethyl radical [14]. Therefore, we further examined the reaction with the addition of photocatalyst under 36 W blue LEDs irradiation. Interestingly, the desired product **3a** was afforded in 92% yield in the presence of $Ir(ppy)_3$ (Table 1, entry 3). No better result was obtained when other photocatalysts were utilized including $Ru(bpy)Cl_2·6H_2O$, 4CzIPN, EosinY and $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (Table 1, entries 4–7). We further investigated this photocatalytic reaction in various solvents. As shown in Table 1, MeCN was still the best choice in this transformation (entries 8–13). Meanwhile, the structure and configuration of compound **3a** was determined as (*E*)-β-trifluoromethyl vinylsulfonohydrazide by single-



Scheme 1. Multicomponent reaction of alkynes, trifluoromethyl thianthrenium triflate, DABCO·$(SO_2)_2$ and hydrazines under photocatalytic conditions. Conditions: alkyne **1** (0.2 mmol), TT-$CF_3^+OTf^-$ (0.3 mmol), hydrazine **2** (0.4 mmol), DABCO·$(SO_2)_2$ (0.16 mmol), $Ir(ppy)_3$ (0.004 mmol), MeCN (4 mL), 36 W blue LEDs, under a N_2 atmosphere. Isolated yield based on alkyne **1**.

crystal X-ray diffraction analysis (CCDC: 2176532). No reaction occurred when amine, $TsNHNH_2$ or $PhCONHNH_2$ was used as a replacement of hydrazine. For the reaction by employing *O*-phenethylhydroxylamine as the reactant instead of morpholin-4-amine **2a**, only a trace amount of product was detected. Inferior results were obtained when the sulfur dioxide surrogate of DABCO·$(SO_2)_2$ was changed to $NaHSO_3$ (sodium hydrogen sulfite), potassium metabisulfite or sodium metabisulfite (data not shown in Table 1).

With the above promising result in hand, we subsequently explored the substrate scope of this multicomponent reaction of alkynes, trifluoromethyl thianthrenium triflate, sulfur dioxide and hydrazines under photocatalytic conditions. As shown in Scheme 1, it was found that a wide range of arylalkynes bearing electron-donating and electron-withdrawing substituents in the aromatic ring were workable in this transformation. Notably, some sensitive functional groups (such as amino and ester) were compatible. For instance, reaction of trifluoromethyl thianthrenium triflate (TT-$CF_3^+OTf^-$), 4-ethynylaniline, DABCO·$(SO_2)_2$ and morpholin-4-amine **2a** proceeded smoothly, giving rise to the corresponding amino-substituted product **3f** in 71% yield. The ester-containing products **3k** and **3l** could be afforded smoothly as well. Meanwhile, reactions employing other heterocycle-substituted alkynes were explored. Pyridinyl-substituted product **3s** and thiophenyl-substituted product **3t** were produced in 67% yield, respectively. The excellent functional group tolerance supported the practicality of this reaction. Additionally, reactions of alkyl alkynes, trifluoromethyl thianthrenium triflate (TT-$CF_3^+OTf^-$), DABCO·$(SO_2)_2$ and morpholin-4-amine **2a** were examined, leading to the desired products as expected (**3u** and **3v**). Interestingly, trimethylsilylacetylene was a good reactant as well, and the trimethylsilyl-substituted product **3w** was provided in 58% yield. We further



Scheme 2. Proposed mechanism.

examined the reaction by using other hydrazines in the reaction of trifluoromethyl thianthrenium triflate, DABCO·$(SO_2)_2$ and 4-methoxylphenylacetylene under photocatalytic conditions, and the desired products **3x-3z** were generated in good yields. To demonstrate the practicability of this trifluoromethylation/sulfonylation of alkyne, a gram-scale experiment (1.0 mol) was carried out, which provided the corresponding product **3a** in 71% yield.

On the basis of previous reports [11], a plausible mechanism is proposed, as shown in Scheme 2. It was reasoned that in the presence of photocatalyst under visible light irradiation, trifluoromethyl radical would be formed from trifluoromethyl thianthrenium triflate (TT-$CF_3{}^+OTf^-$) through a single electron transfer. Then, trifluoromethyl radical would be trapped by alkyne **1** giving rise to vinyl radical intermediate **A**, which would react with sulfur dioxide leading to sulfonyl radical intermediate **B**. In the meantime, hydrazine **2** would convert to cation radical intermediate **C** with the assistance of photocatalyst. After deprotonation, radical **D** would be produced, which would subsequently combine with sulfonyl radical intermediate **B** to provide trifluoromethyl-substituted vinyl sulfonohydrazide **3**.

All the prepared trifluoromethyl-substituted vinyl sulfonohydrazide derivatives were evaluated for their antibacterial activities *in vitro* against Gram-positive bacterium (*S. aureus*) and Gram-negative bacterium (*E. coli*), using the standard two folds serial dilution method in 10×150 mm glass tubes. Initially, the compounds were dissolved in dimethyl sulfoxide (DMSO) to prepare the stock solutions (40 mmol/L), then the tested compounds were prepared in LB broth (Tryptone 10 g/L, Yeast extract 5 g/L, NaCl 10 g/L, pH 7.4) to obtain the required concentration of 40 μmol/L (2 mL) in tubes. The bacterial suspension was adjusted with sterile saline to a concentration of 1×10^5 CFU and 100 μL aliquots of the test organisms were added to the appropriate tubes. Inoculated tubes were incubated aerobically at 37 °C, 150 r.p.m. (round per minute) for 18 h. The growth inhibition was determined to be the suppression of optical density at 620 nm.

The results in Table 2 revealed that all of the trifluoromethyl-substituted vinyl sulfonohydrazides displayed no activities against the tested strains. But interestingly, compound **3y** effectively inhibited the growth of *S. aureus* and also displayed weak activity against *E. coli*, and the MIC (the lowest concentration that inhibit visible growth) against *S. aureus* was furtherly detected to be 10 μmol/L, which indicated that the structure of sulfonyl hydrazine might be a novel potential antibacterial pharmacophore, especially for Gram positive bacteria.

In conclusion, we have developed an efficient trifluoromethylation/sulfonylation of alkynes from trifluoromethyl thianthrenium triflate and sulfur dioxide under extremely mild reaction conditions, providing a facile access to trifluoromethyl-substituted vinyl sulfonohydrazides in moderate to good yields. This multicomponent reaction of trifluoromethyl thianthrenium triflate, alkynes, sulfur dioxide and hydrazines proceeds efficiently under visible light irradiation in the presence of photocatalyst at room temperature with broad substrate scope and excellent functional group compatibility. It is noteworthy that this reaction is highly stereoselective, since only (*E*)-isomer is obtained. Additionally, these trifluoromethyl-substituted vinyl sulfonohydrazides are further evaluated for anti-bacteria activity. *In vitro* activities of these compounds against *S. aureus* (G^+) and *E. coli* (G^-) are examined.

Table 2
Antibacterial activities *in vitro* against Gram-positive bacterium (*S. aureus*) and Gram-negative bacterium (*E. coli*).

Compds.	Growth inhibition (O.D. 620 nm, %) *S. aureus*	*E. coli*
3a	5.9	2.5
3b	NA	NA
3c	0.9	0.9
3d	10.9	8.8
3e	NA	4.6
3f	2.3	NA
3g	1.7	NA
3h	NA	NA
3i	0.3	7.1
3j	5.7	NA
3k	NA	NA
3l	NA	NA
3m	NA	0.6
3n	1.1	NA
3o	2.0	3.0
3p	9.4	5.8
3q	4.4	0.6
3y	97.3	13.6

NA: no activity.

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.2022.107984.

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