Organic LETTERS
Letter
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Copper-Assisted Oxidative Trifluoromethylthiolation of 2,3-Allenoic Acids with $AgSCF_3$

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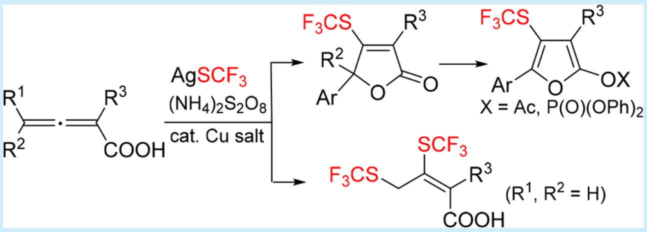
Supporting Information

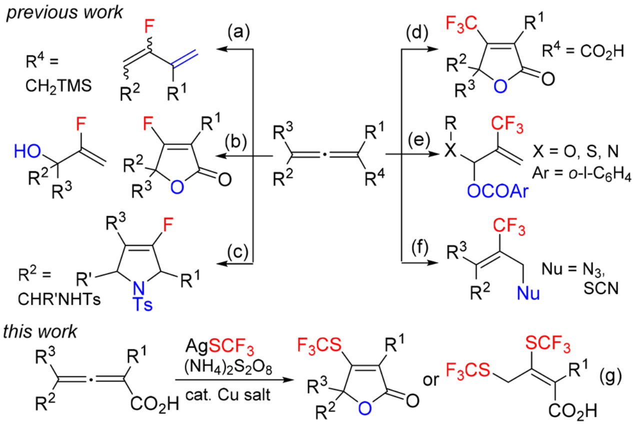
ABSTRACT: The oxidative trifluoromethylthiolation of 2,3-allenoic acids with $AgSCF_3$ in the presence of $(NH_4)_2S_2O_8$ and catalytic copper salt was investigated. A series of 4-aryl-2,3-allenoic acids underwent radical trifluoromethylthiolation/intramolecular cyclization to afford β-trifluoromethylthiolated butenolides, which were conveniently transformed into trifluoromethylthiolated furan derivatives. In contrast, 2-monosubstituted 2,3-allenoic acids were converted into the corresponding 3,4-bis(trifluoromethylthio)but-2-enoic-acids under similar reaction conditions.



The incorporation of a SCF_3 moiety into organic molecules can improve the physical, chemical, and biological properties of parent compounds.[1] Consequently, the development of efficient approaches to SCF_3-containing compounds has attracted extensive attention.[2] Traditional synthetic methods of these compounds mainly rely on halogen−fluorine exchange of halomethyl sulfides or the trifluoromethylation of thiols.[3] Recently, the direct construction of C−SCF_3 bonds has emerged as an attractive research area in organofluorine chemistry. Strategies for trifluoromethylthiolation through C−X (Cl, Br, I) bond transformation,[4] oxidative cross-coupling,[5] C−H bond activation,[6] and reduction of SO_2CF_3[7] have been reported. On the other hand, the vicinal difunctionalization of alkenes and alkynes has also been developed for the introduction of a SCF_3 group and another functional group.[8] However, a strategy based on functionalization of allenes to incorporate a SCF_3 group has not been established.

In recent years, the reactions of allenes have been investigated intensively due to their unique structures and diverse reactivities.[9] Among them, the fluorination and trifluoromethylation of allenes have been disclosed.[10,11] For example, Gouverneur reported the electrophilic fluorodesilylation of allenylmethylsilanes for the synthesis of 2-fluoro-1,3-dienes (Scheme 1a).[10a] Ma and co-workers described the inter- and intramolecular oxyfluorination of allenes, respectively (Scheme 1b).[10b,c] Liu developed a silver-catalyzed intramolecular aminofluorination of the activated allenes for the synthesis of fluorinated dihydropyrroles (Scheme 1c).[10d] In the cases of trifluoromethylation of allenes, Ma's group developed an efficient copper-catalyzed cyclic oxytrifluoromethylation of 2,3-allenoic acids in 2013 (Scheme 1d).[11a] Recently, the regioselective oxytrifluoromethylation, trifluoromethylazidation, and trifluoromethylthiocyanation of allenes with Togni reagent have also been reported (Scheme 1e[11b] and 1f[11c]). Herein, we describe an efficient and practical oxidative cyclic oxytrifluoromethylthiolation and regioselective bis-trifluoromethylthiolation of 2,3-allenoic acids with $AgSCF_3$ (Scheme 1g). The resulting β-trifluoromethythiolated butenolides and 3,4-bis(trifluoromethylthio)but-2-enoic-acids are previously unknown and potentially useful in different research areas.

Scheme 1. Fluorination, Trifluoromethylation, and Trifluoromethylthiolation of Allenes

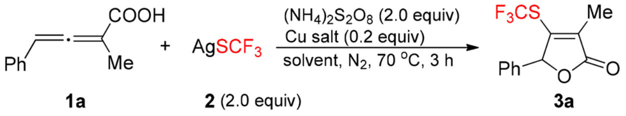


Our study commenced with the trifluoromethylthiolation of 2-methy-4-phenylbuta-2,3-dienoic acid (**1a**) with $AgSCF_3$ (**2**) (Table 1). The reaction in the presence of $(NH_4)_2S_2O_8$ in

Received: July 21, 2017

DOI: 10.1021/acs.orglett.7b02249

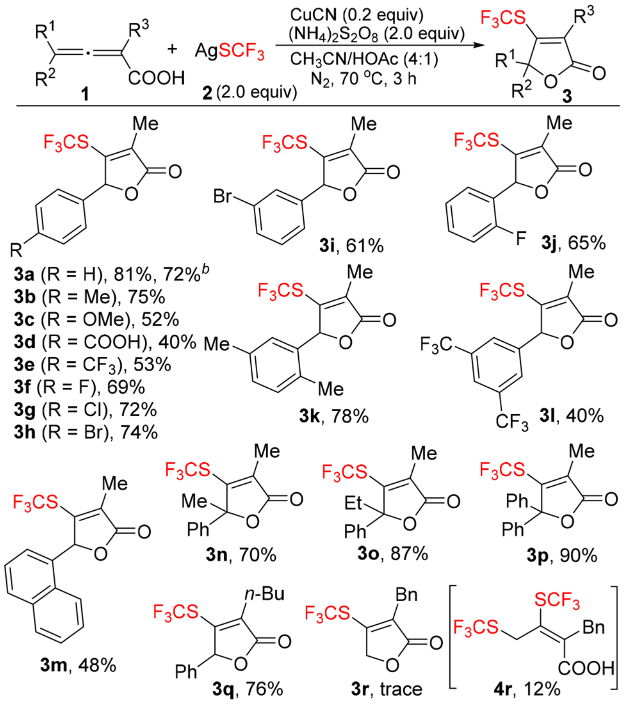
Table 1. Optimization of Reaction Conditions[a]



entry	Cu salt	solvent	yield (%)[b]
1	–	MeCN	22
2	–	DMF	trace
3	–	THF	NR
4	–	MeCN/HCO_2H (1:1)	15
5	–	MeCN/HOAc (1:1)	38
6	–	MeCN/HOAc (2:1)	60
7	–	MeCN/HOAc (4:1)	65
8	CuI	MeCN/HOAc (4:1)	67
9	$Cu(OAc)_2$	MeCN/HOAc (4:1)	77
10	CuCN	MeCN/HOAc (4:1)	85

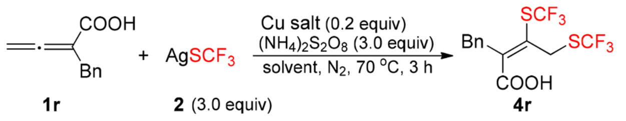
[a] Reaction conditions: **1a** (0.1 mmol), $AgSCF_3$ (0.2 mmol), Cu salt (0.02 mmol), $(NH_4)_2S_2O_8$ (0.2 mmol), solvent (2.0 mL), under N_2, 70 °C, 3 h. [b] Yields determined by ^{19}F NMR spectroscopy using trifluoromethylbenzene as an internal standard.

Scheme 2. Cyclic Oxytrifluoromethylthiolation of 2,3-Allenoic Acids[a]



[a] Reaction conditions: **1** (0.2 mmol), $AgSCF_3$ (0.4 mmol), CuCN (0.04 mmol), $(NH_4)_2S_2O_8$ (0.4 mmol), MeCN/HOAc (3.2 mL/0.8 mL), under N_2, 70 °C, 3 h; isolated yields. [b] The reaction was carried out on a 1.0 mmol scale.

Table 2. Optimization of Reaction Conditions[a]



entry	Cu salt	solvent	yield (%)[b]
1	CuCN	MeCN/HOAc (4:1)	16
2	$Cu(OAc)_2$	MeCN/HOAc (4:1)	24
3	$CuCl_2$	MeCN/HOAc (4:1)	19
4	CuI	MeCN/HOAc (4:1)	12
5	$Cu(OAc)_2$	MeCN/HCO_2H (4:1)	28
6	$Cu(OAc)_2$	MeCN/HCO_2H/H_2O (4:1:1)	38
7	$Cu(OAc)_2$	MeCN/HCO_2H/H_2O (4:1:2)	51
8	$Cu(OAc)_2$	MeCN/HCO_2H/H_2O (4:1:3)	45

[a] Reaction conditions: **1r** (0.1 mmol), $AgSCF_3$ (0.3 mmol), Cu salt (0.02 mmol), $(NH_4)_2S_2O_8$ (0.3 mmol), solvent (2.8 mL), under N_2, 70 °C, rt, 3 h. [b] Yields determined by ^{19}F NMR spectroscopy using trifluoromethylbenzene as an internal standard.

MeCN gave the cyclized trifluoromethylthiolated product **3a** in 22% yield (entry 1). THF and DMF were not suitable for the reaction (entries 2 and 3). Based on our experience of bis-trifluoromethylthiolation of propiolic acid derivatives,[8k] HCOOH and HOAc were tested as the co-solvents (entries 4 and 5). It was found that MeCN/HOAc was more effective than MeCN/HCO_2H. A careful survey of solvent ratios and copper catalysts was then conducted (entries 6–10), and the combination of MeCN/HOAc (4:1) and CuCN (0.2 equiv) was optimal, giving the desired product **3a** in 85% yield. Finally, the temperature, oxidant, and ligand were also examined and no better result was realized (for detailed data, see the Supporting Information).

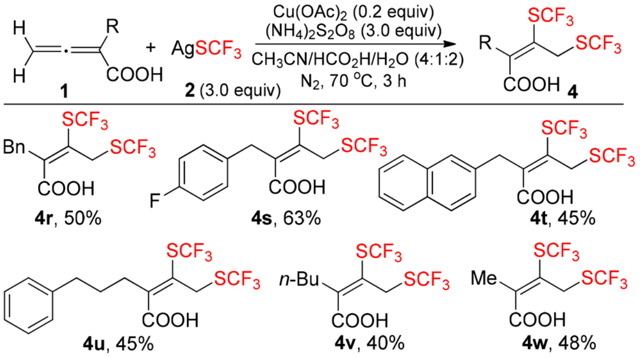
With the optimized reaction conditions in hand, the substrate scope of this cyclic oxytrifluoromethylthiolation was examined (Scheme 2). A series of substrates bearing electron-donating (**3b** and **3c**) and electron-withdrawing (**3d** and **3e**) groups as well as halides (**3f**–**3h**) were all effective. The structure of the product was confirmed by X-ray diffraction studies of **3f** (see the Supporting Information). The position and number of substitutions (**3i**–**3m**) on the benzene ring did not interfere with this transformation. Me, Et, and Ph were all competent substituents for R^2 (**3n**–**3p**), and this protocol could also be applied for the substrate with *n*-butyl as R^3 (**3q**). Notably, when 2-monosubstituted buta-2,3-dienoic acid **1r** (R^1 = R^2 = H) was subjected to this reaction, only a trace of the desired trifluoromethylthiolated butenolide **3r** was detected. Instead, an unexpected bis-trifluoromethylthiolated product **4r** was obtained in 12% yield.

In continuation of our research on bis-trifluoromethylthiolation of propiolic acid derivatives,[8k] we then focused on the bis-trifluoromethylthiolation of 2-monosubstituted buta-2,3-dienoic acids. The optimization of reaction conditions of **1r** was undertaken to improve the yield of **4r** (Table 2). Increasing the amounts of $AgSCF_3$ and $(NH_4)_2S_2O_8$ to 3.0 equiv led to a slightly higher yield (entry 1). The screening of different copper salts demonstrated that $Cu(OAc)_2$ was more effective than other Cu salts including CuCN, $CuCl_2$, and CuI (entries 1–4). It was found that the solvent was crucial for this reaction (entries 5–8). The yield of **4r** was improved by adding H_2O as the co-solvent, and the highest yield of **4r** was achieved in MeCN/HCO_2H/H_2O (4:1:2). Other attempts to promote this transformation proved to be ineffective (see the Supporting Information).

Next, the scope of the bis-trifluoromethylthiolation reaction was evaluated under the modified conditions, and the results are listed in Scheme 3. A series of 2-monosubstituted buta-2,3-dienoic acids **1r**–**1w** reacted smoothly to afford the bis-trifluoromethylthiolated products **4r**–**4w** in moderate yields. In all cases, the cyclic oxytrifluoromethylthiolated products were also formed in very low yields.

DOI: 10.1021/acs.orglett.7b02249
Org. Lett. XXXX, XXX, XXX–XXX

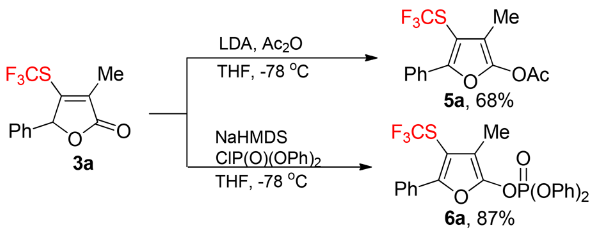
Scheme 3. Bis-trifluoromethylthiolation of 2-Monosubstituted Buta-2,3-dienoic Acids[a]



[a]Reaction conditions: **1** (0.2 mmol), $AgSCF_3$ (0.6 mmol), $Cu(OAc)_2$ (0.04 mmol), $(NH_4)_2S_2O_8$ (0.6 mmol), $MeCN/HCO_2H/H_2O$ (3.2 mL/0.8 mL/1.6 mL), under N_2, 70 °C, 3 h; isolated yields.

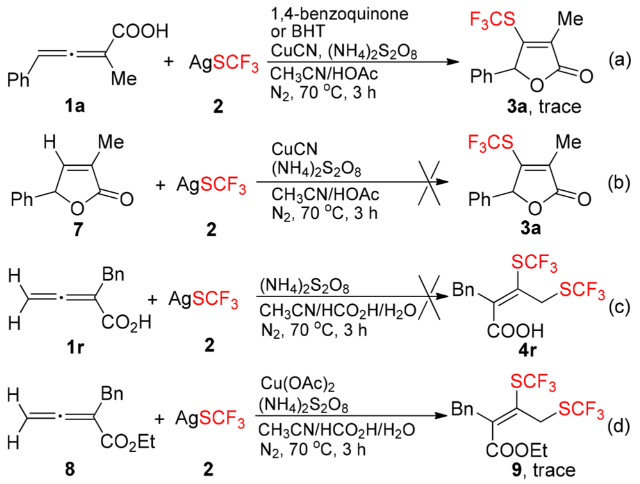
Subsequently, the transformation of trifluoromethylthiolated butenolide **3a** was investigated. As shown in Scheme 4, treatment of **3a** with LDA/Ac_2O or NaHMDS/ClP(O)$(OPh)_2$ afforded trifluoromethylthiolated furan derivatives **5a** or **6a** in good yields, respectively.

Scheme 4. Transformation of Compound 3a



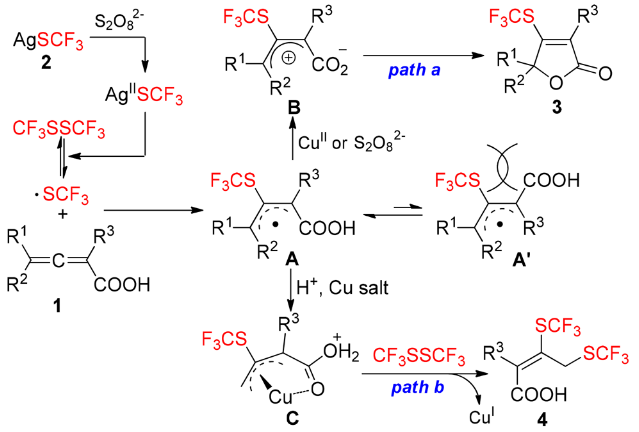
To gain mechanistic insights into this reaction process, some preliminary studies were performed. Only a trace of the desired product **3a** was detected when a radical scavenger, 1,4-benzoquinone or 2,6-di-*tert*-butyl-4-methylphenol (BHT), was added to the standard reaction of **1a**, which indicated this transformation probably proceeded through a radical pathway (Scheme 5a). Moreover, 3-methyl-5-phenylfuran-2(5*H*)-one (**7**) was synthesized and reacted with $AgSCF_3$ under the standard reaction conditions. However, the target product **3a** was not formed (Scheme 5b). On the other hand, the bis-trifluoromethylthiolation reaction could not take place without a copper catalyst (Scheme 5c), which revealed the important role of the copper salt in this reaction. It was noteworthy that buta-2,3-dienoate ester (**8**) was not a suitable substrate for the bis-trifluoromethylthiolation reaction (Scheme 5d), proving that the carboxylic acid of 2-monosubstituted buta-2,3-dienoic acids was a key directing group for promoting bis-trifluoromethylthiolation reaction.

Scheme 5. Mechanistic Investigation



On the basis of the above results, a plausible reaction mechanism was proposed (Scheme 6). First, oxidation of $AgSCF_3$ by $(NH_4)_2S_2O_8$ generates $Ag^{II}SCF_3$, which could be further transformed to the CF_3S radical or CF_3SSCF_3.[5g,h,12] The addition of the SCF_3 radical to allenes **1** would generate allyl radical species **A** and **A′**.[11] Species **A′** would isomerize to the thermodynamically more favored **A**, because of the steric hindrance between COOH and SCF_3 groups.[11c] Then, the reaction of intermediate **A** could proceed in two different ways depending on the substrate. Intermediate **A** with aromatic substitutions was easily oxidized by Cu^{II} or $(NH_4)_2S_2O_8$ to yield the allyl cation **B**. Finally, the intramolecular attack of intermediate **B** delivered the cyclized products **3** (path a).[11a] In the case of substrates with R^1 and R^2 as H atoms, we speculated that trapping species **A** by a Cu salt formed SCF_3-allyl-Cu complex **C**.[8k,10b,11a] Complex **C** was converted to the bis-trifluoromethylthiolated products **4** by copper-assisted trifluoromethylthiolation with CF_3SSCF_3 (path b).[8k] However, the exact mechanism of this transformation remains unclear at the present stage.

Scheme 6. Proposed Reaction Mechanism



In conclusion, we have disclosed a copper-assisted oxidetive cyclic oxytrifluoromethylthiolation and bis-trifluoromethylthiolation of 2,3-allenoics with $AgSCF_3$. A series of previously unknown β-trifluoromethylthiolated butenolides, trifluoromethylthiolated furans, and bis-trifluoromethylthiolated allyllic acids were obtained in good yields. Further investigation of the reaction mechanism and the applications of this protocol are currently in progress.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b02249.

Optimization of reaction conditions; experimental procedures; characterization data; mechanistic study data; copies of ^{1}H, ^{19}F, and ^{13}C NMR spectra; X-ray crystal structure of **3f** (PDF)

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

National Natural Science Foundation of China (21332010, 21421002), the Strategic Priority Research Program of the Chinese Academy of Sciences (XDB20000000), Youth Innovation Promotion Association CAS (No. 2016234), and Shanghai Pujiang Program (No. 15PJ1410300) are greatly acknowledged for funding this work.

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