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Copper-Catalyzed Difluoroalkylation-Thiolation of Alkenes Promoted by $Na_2S_2O_5$

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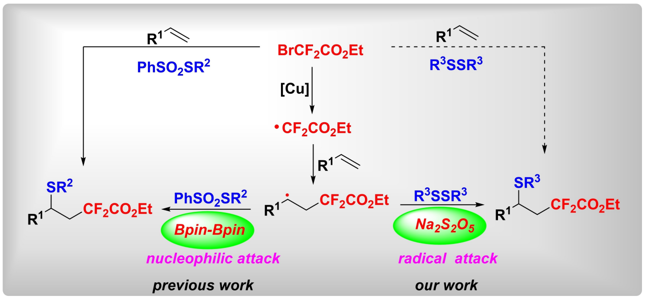
A novel and convenient protocol for the difluoroalkylation-thiolation of alkenes catalyzed by Cu/$Na_2S_2O_5$ system has been developed. This reaction was carried out using readily available starting materials under mild conditions, wherein the C–C and C–S bonds simultaneously were constructed smoothly. The reaction features broad substrate scope of alkenes and disulfides, good functional group tolerance, and good to excellent yields with high selectivity.

Multicomponent reactions, defined as synthetic protocols that join together three or more coupling partners have emerged as strategically efficient and synthetically useful methods for constructing complex carbon frameworks from relatively simple precursors, thus meeting diverse transformation of easily available substrates in all fields of organic synthesis.[1] Among these reactions, transition-metal-catalyzed alkene difunctionalization reactions that involve the installation of two vicinal functional groups in one step have been studied as powerful tools for the construction of substituted alkanes, and valuable building blocks for natural and biologically active compounds from simple starting materials.[2]

Compared with the common molecules, fluorine-containing compounds generally show completely different physical and chemical properties and the introduction of fluorine can generally enhance the lipophilicity, bioavailability, and metabolic stability of its parent molecules.[3] In view of the ubiquity of organofluorine groups in materials science and biologically active molecules,[4] the difunctionalization of unsaturated bonds is a direct way to construct complex molecular frameworks that otherwise multistep synthesis is required. Among these fluorinated moieties, the bromodifluoroalkyl compounds are extremely appealing reagents, which can be utilized to make valuable molecules with a different function, such as the difluoroalkyl radical,[5] difluorocarbene precursors,[6] and C1 and C2 synthons.[7] The transition-metal-catalyzed difluoroacetylation of alkenes has emerged as a powerful method for the construction of these compounds, such as difluoroacetylation with arylation,[8] oxylation,[9] halogenation reactions.[10] Moreover, the visible-light-mediated difluoroacetylation of alkenes also have been developed as a useful alternative for the synthesis of α,α-difluoroacetates.[11]

The sulfur-containing molecules are prevalent in a broad range of materials science, pharmaceuticals, and natural products, and often display interesting various biological activities.[12] With this in the background, the simultaneous construction of C–C and C–S bonds via the C=C bond has successfully been disclosed by the groups of Song[13] and Cai.[14] Very recently, Song has reported commercially available halodifluoroalkyl reagents displaying versatile reactivities, represent one of the significant synthetic building blocks and key subunits of pharmaceutically active molecules. Moreover, Cai et al. have developed a new protocol for difluoroalkylation-thiolation of alkenes catalyzed by iron facilitated photoredox, in which the C–C and C–S bonds simultaneously were constructed smoothly under mild conditions.

Based on the background above, we assumed if we could use directly disulfides as a sulfur source to achieve difluoroalkylation-thiolation of alkenes, which can efficiently expand the reaction efficiency from economic performance. In view of this, it is reasonable to hypothesize that if the alkyl radical generated by the addition of difluoroalkyl radicals to alkenes is directly captured by disulfide reagent, which could accomplish a direct difluoroalkylation-thiolation of alkenes from the perspective of atomic economy (Scheme 1). Although, in Cai's work,[14] disulfide was utilized as sulfur source to give desired product **3 aa** in their control experiment, only 18 % yield was obtained. Due to few reports and great potential in this area, the development of concise approaches to introduce the difluoroalkyl group



Scheme 1. Copper-catalyzed three-component difluoroalkylation-thiolation of alkenes.

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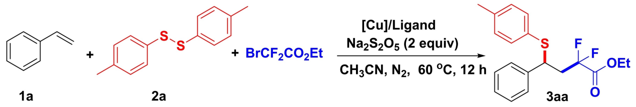
Supporting information for this article is available on the WWW under https://doi.org/10.1002/ejoc.202100091 synchronously with sulfur-containing groups into alkenes remains a meaningful task. Herein, we disclosed an efficient and practical approach to accomplish difluoroalkylation-thiolation of alkenes using disulfide reagents as sulfur source and $Na_2S_2O_5$ as a reducing agent in the presence of copper catalyst under mild reaction condition.

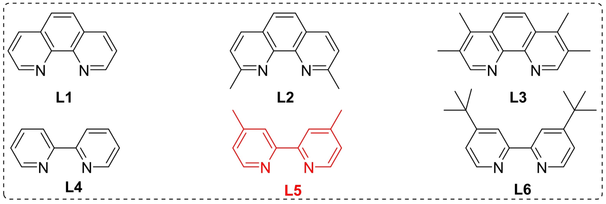
At the outset of the project, we examined the feasibility of the difluoroalkylation-thiolation of styrene with disulfide reagent **2a** and ethyl (bromodifluoroacetate) $BrCF_2CO_2Et$ in the presence of a copper catalyst. First of all, a variety of reductants were screened with 20 mol% of CuTc as a catalyst, 2 equiv. of CsF as an additive, and CH_3CN as solvent under nitrogen atmosphere at 60 °C for 12 h, which showed that $Na_2S_2O_5$ was most efficient for this transformation to furnish product **3aa** in 63 % yield (Table 1, entry 4).

Encouraged by these preliminary results, we considered that solvent maybe actually make a difference and then examined the effects of different other solvents on the reaction. The results showed that most common solvents, such as Toluene, 1,4-dioxane, DMSO, THF *et al*, were completely ineffective, which means the CH_3CN is crucial to promote this reaction (see Supporting Information). Next, we focused on the copper catalysts, and a series of copper catalysts were investigated and the results showed that the reaction performed well with CuCl as catalyst (72 % yield) (Table 1, entry 8), which was better than other copper catalysts. Thus, CuCl was determined to be the most appropriate catalyst for this transformation. Subsequently, a variety of nitrogen-based ligands were screened, and the results were shown in Table 1. Among the ligands examined, the **L5** demonstrated the best efficiency for giving the desired product **3aa** in 74 % yield in the presence of CuCl (Table 1, entry 15). Further experiments conducted for searching proper base found that CsF is still most efficient for this transformation. Moreover, the amount of CsF and $Na_2S_2O_5$ were also examined and no further improvements were found (see Supporting Information). Lastly, 85 % yield was furnished when the reaction time was prolonged to 24 hours (Table 1, entry 20). A control experiment was also conducted in the absence of $CuBr_2$ or **L5**, and showed that no desired product was obtained.

Table 1. Optimization of reaction conditions.[a]



Entry	Catalyst	Ligand	Reductant	Additive	Yield [%][a]
1	CuTc	**L1**	Na_2SO_3	CsF	45
2	CuTc	**L1**	$Na_2S_2O_3$	CsF	18
3	CuTc	**L1**	$NaHSO_3$	CsF	43
4	CuTc	**L1**	$Na_2S_2O_5$	CsF	63
5	CuTc	**L1**	Cu	CsF	trace
6	CuO	**L1**	$Na_2S_2O_5$	CsF	trace
7	Cu_2O	**L1**	$Na_2S_2O_5$	CsF	29
8	CuCl	**L1**	$Na_2S_2O_5$	CsF	72
9	$CuCl_2\cdot 2H_2O$	**L1**	$Na_2S_2O_5$	CsF	66
10	CuBr	**L1**	$Na_2S_2O_5$	CsF	62
11	$CuBr_2$	**L1**	$Na_2S_2O_5$	CsF	63
12	CuCl	**L2**	$Na_2S_2O_5$	CsF	nr
13	CuCl	**L3**	$Na_2S_2O_5$	CsF	70
14	CuCl	**L4**	$Na_2S_2O_5$	CsF	27
15	CuCl	**L5**	$Na_2S_2O_5$	CsF	74
16	CuCl	**L6**	$Na_2S_2O_5$	CsF	63
17	CuCl	**L5**	$Na_2S_2O_5$	K_2CO_3	51
18	CuCl	**L5**	$Na_2S_2O_5$	NaOAc	29
19	CuCl	**L5**	$Na_2S_2O_5$	$NaHCO_3$	44
20[b]	CuCl	**L5**	$Na_2S_2O_5$	CsF	85
21	–	**L5**	$Na_2S_2O_5$	CsF	0
22	CuCl	–	$Na_2S_2O_5$	CsF	0
23	CuCl	**L5**	$Na_2S_2O_5$	–	37

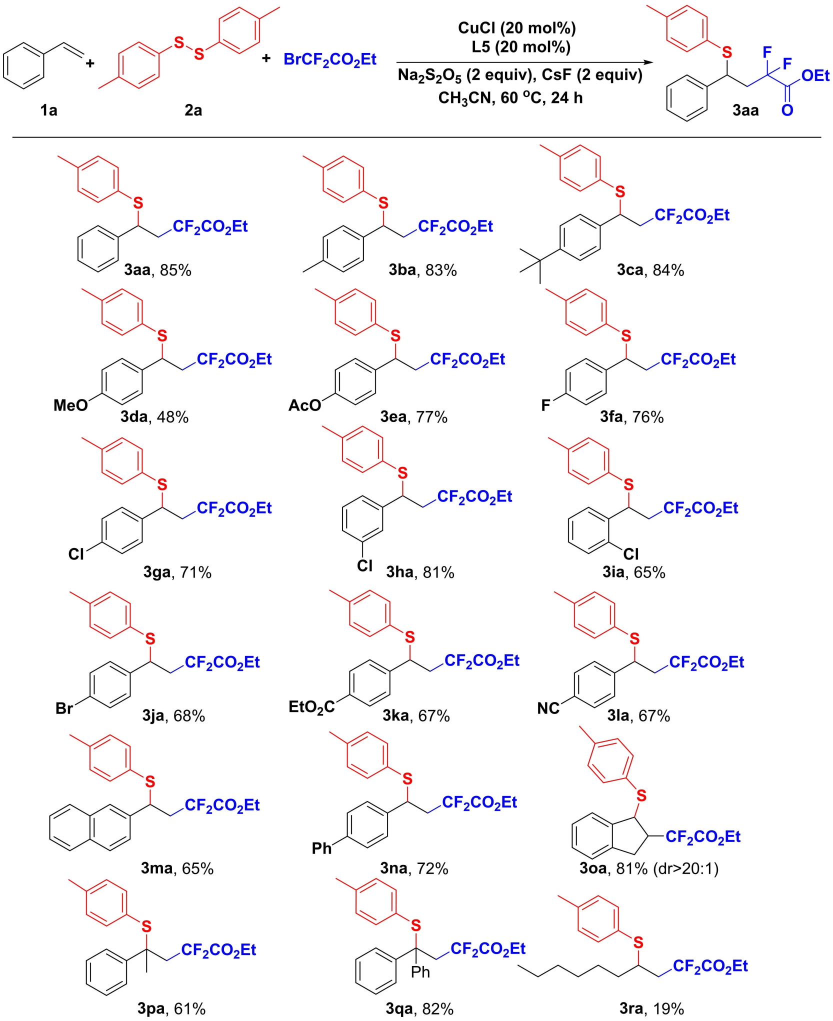


[a] Reaction conditions: styrene **1a** (0.2 mmol), **2a** (0.24 mmol), $BrCF_2CO_2Et$ (0.24 mmol), [Cu] (20 mol%), Liand (20 mol%), Reductant (0.4 mmol), additive (0.4 mmol), CH_3CN (2 mL), 60 °C, 12 h, isolated yield. [b] 24 h.

With the optimized reaction conditions in hand, we proceeded to evaluate the scope of this three-component cross-coupling reaction with a variety of substituted alkenes shown in Table 2. The results revealed that the optimized procedure could be applied successfully to a range of different alkenes. The substrates bearing electron-donating and -withdrawing groups on the aromatic ring were converted to the corresponding products in the range of 48%–85% isolated yields. Alkenes bearing electron-donating groups in particular showed higher levels of reactivity towards this reaction. However, only 48% yield was obtained for MeO-substituted substrate **2d** and the detailed reason is still not clear. In addition, the steric hindrance effect was observed. For example, the reactions of the styrene bearing chlorine-substituent at its *o*-position gave the corresponding products in lower yields, which was lower than its *m*- and *p*-position derivatives (Table 2, **3ja**–**3ia**). The substrates **1k** and **1l** with strong electron-withdrawing group also had better reactivity, giving corresponding product **3ka** and **3la** in 67% yield respectively. Interestingly, naphthyl- and 4-Ph-substituted alkene **1m** and **1n** were also compatible with this reaction under the optimized conditions to give the desired products (65–72% yields) (Table 2, **3ma**, **3na**). Interestingly, internal olefin **1o** could participate in this reaction in good yield with high stereoselectivity (dr > 20 : 1) (Table 2, **3oa**). In the case of alkenes bearing a substituent group at α-position of double bond underwent the current difluoroalkylation-thiolation process smoothly in good yield. In addition to the aromatic alkenes, aliphatic alkene **1r** was also examined under the standard reaction conditions, furnishing target product **3ra** smoothly.

Once we had identified that the alkenes were adequate substrates for this reaction system, we aimed to expand the substrate scope to disulfides. As such, substrates containing methyl, methoxy, fluoride, chloride, and bromide on the aromatic ring were all competent reaction partners, providing the corresponding products efficiently in excellent yields (Table 3, **3aa**–**3ah**). In addition, steric hindrance effect was observed. For example, the reactions of the disulfides bearing methyl-substituent at its *o*, *m*, *p*-position gave the correspond-

Table 2. Substrate scope of the reaction for alkenes.[a]



[a] Reaction conditions: alkene **1** (0.2 mmol), **2a** (0.24 mmol), BrCF$_2$CO$_2$Et (0.24 mmol), CuCl (20 mol%), **L5** (20 mol%), Na$_2$S$_2$O$_5$ (0.4 mmol), CsF (0.4 mmol), CH$_3$CN (2 mL), 60 °C, 24 h, isolated yield.

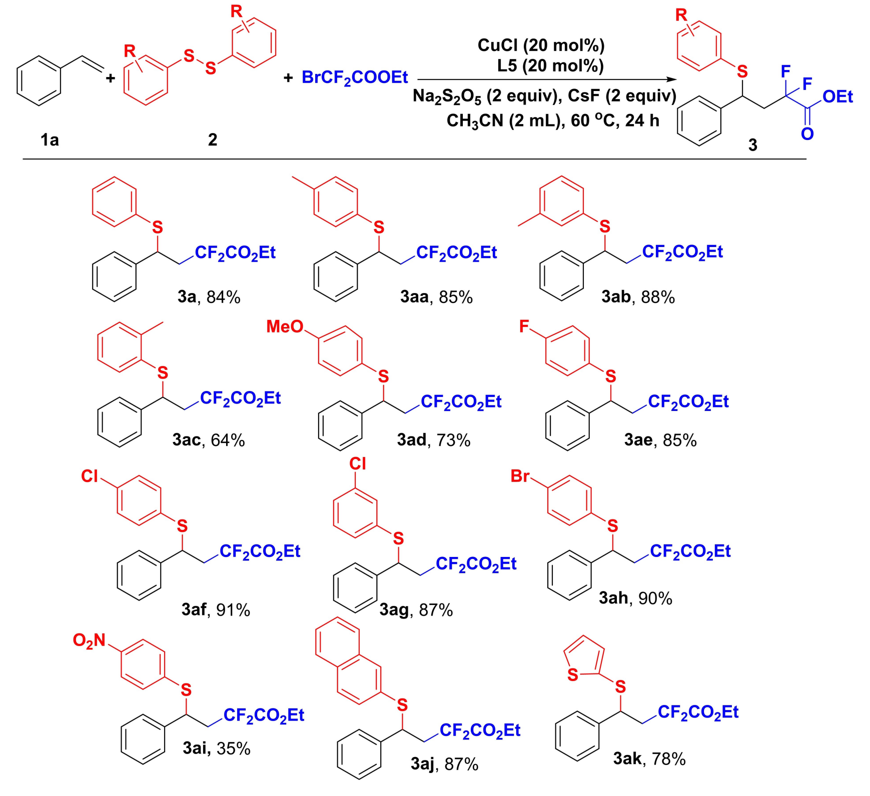
ing products in different yields (Table 3, **3aa**–**3ac**). Moreover, in the case of disulfide containing a strong electron-withdrawing group -NO$_2$ was also a suitable substrate, providing product **3ai** in 35% yield. It should be noted that naphthyl and heteroaryl substrates **2j** and **2k** could also participate in this reaction in excellent yield (Table 3, **3aj**–**3ak**).

Difluoroalkylation-thiolation of estrone derivative alkene bearing a steroid scaffold was also conducted to produce **3sa** in 68% yield with a tolerating ketone group. This transformation indicated that the reaction system is amenable to functionalization and modification of complex alkenes bearing the skeleton of natural products, and show high potential applications in biological evaluation (Scheme 2a). Moreover, the product **3aa** could be reduced to its alcohol derivatives **4** by NaBH$_4$ in 85% yield and be oxidized to its sulfone derivatives **5** by *m*-CPBA in 72% yield, which means these two functional groups are extremely useful and convertible (Scheme 2b).

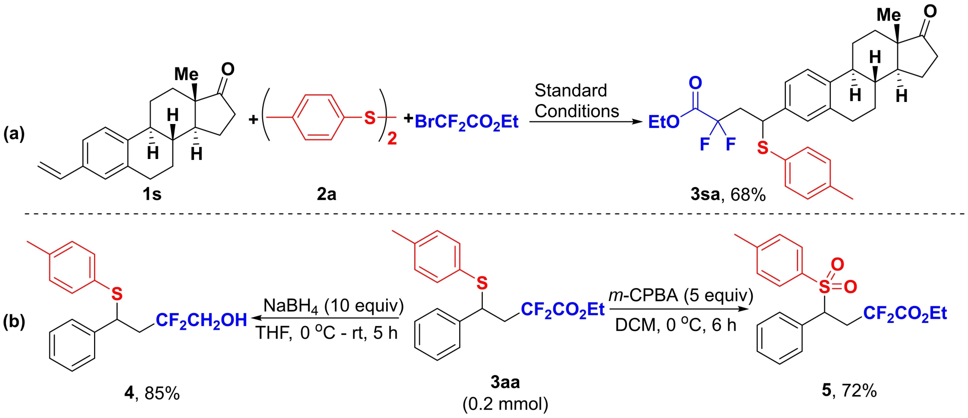
Control experiments were designed to probe the mechanism of this reaction (Scheme 3, see the SI for details). First, the radical scavenger TEMPO completely suppressed the model reaction and 15% yield of the radical coupling adduct **6** was obtained (Scheme 3a), which demonstrated the presence of ethyl difluoroacetate radical in the process. Next, when disulfides **2a** was replaced by *p*-toluenethiol to examine this reaction and the results show that no desired product **3aa** was provided, which indicated the importance of disulfides as sulfur source under our reaction condition. Moreover, the product **3aa** could be obtained in 37% yield in the absence of Na$_2$S$_2$O$_5$, which means Na$_2$S$_2$O$_5$ might be accelerating the reduction of Cu(II) species.[15a]

Based on the above experimental results and previous literature reports,[15] possible reaction mechanism for the difluoroalkylation-thiolation of alkenes is depicted in Scheme 4. Firstly, the initiation of the catalytic cycle occurs with oxidation of Cu(I) species by BrCF$_2$CO$_2$Et *via* a single-electron transfer process, generating reactive difluoroalkyl radical **I** and Cu(II) concomitantly. Subsequently, the addition of the radical I to styrene gives a transient benzyl radical intermediate **II**, which

Table 3. Substrate scope of the reaction for disulfides.[a]



[a] Reaction conditions: styrene **1a** (0.2 mmol), **2** (0.24 mmol), $BrCF_2CO_2Et$ (0.24 mmol), CuCl (20 mol%), **L5** (20 mol%), $Na_2S_2O_5$ (0.4 mmol), CsF (0.4 mmol), CH_3CN (2 mL), 60 °C, 24 h, isolated yield.



Scheme 2. Synthetic transformations.

reacts directly with disulfides to furnish desired product **3aa**. Finally, recycling Cu(I) species is obtained after the reduction of Cu(II) species by solvent (CH_3CN) or $Na_2S_2O_5$.

In summary, we have developed a new protocol for the difluoroalkylation-thiolation of alkenes catalyzed by Cu/$Na_2S_2O_5$ system with disulfides as a sulfur source. This reaction allows a variety of alkenes and disulfides, giving easy access to difluoroalkylation-thiolation of alkenes. Further investigations on the mechanism and applications of the difluoroalkylation-thiolation of alkenes to other substrates for the synthesis of natural bioactive products are in progress.

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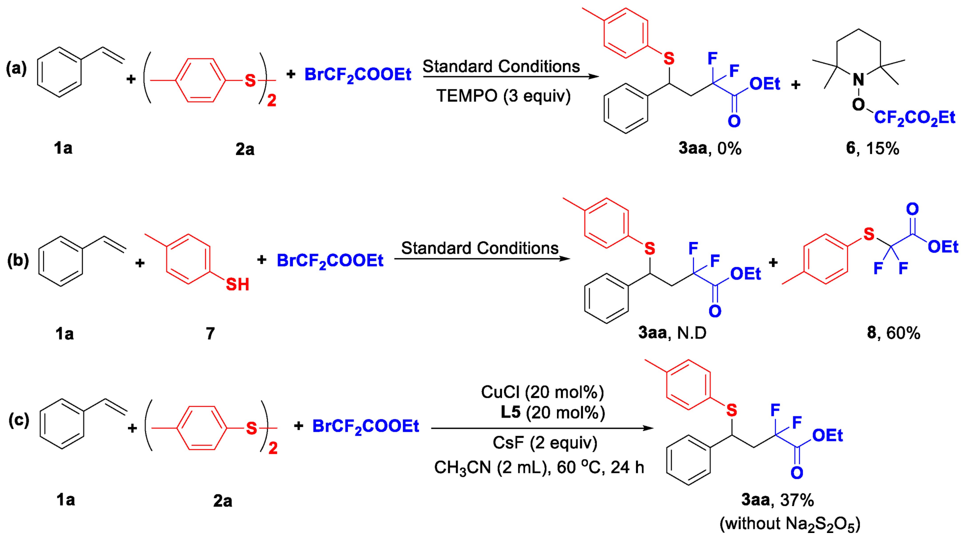
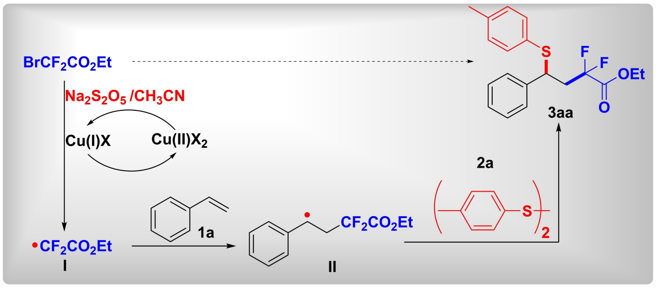


Scheme 3. Control experiments.



Scheme 4. Proposed reaction mechanism.

Conflict of Interest

The authors declare no conflict of interest.

Keywords: Copper · Homogeneous catalysis · Multicomponent reactions · Synthetic methods

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