Organic LETTERS

Letter

Cite This: Org. Lett. XXXX, XXX, XXX−XXX

pubs.acs.org/OrgLett

Copper-Catalyzed Decarboxylative Disulfonylation of Alkynyl Carboxylic Acids with Sulfinic Acids

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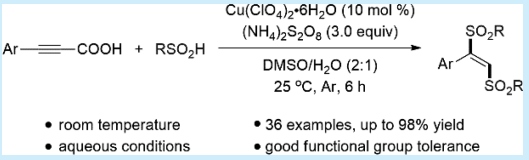
Supporting Information

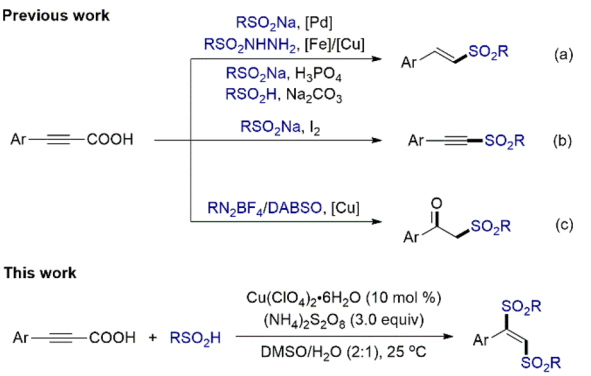
ABSTRACT: A copper-catalyzed decarboxylative disulfonylation of alkynyl carboxylic acids with sulfinic acids in aqueous solution has been developed. The reaction provides a straightforward and practical access to (*E*)-1,2-disulfonylethenes, which are important building blocks in synthetic organic chemistry, and exhibits a good functional group tolerance and excellent stereoselectivity. A possible mechanism for the transformation is proposed.



Sulfone-containing molecules have broad applications in the fields of biochemistry, medicinal chemistry, materials, and organic synthesis.[1] Among them, 1,2-disulfonylethenes have attracted significant attention because of their usefulness as synthetic intermediates.[2] In light of their importance, the development of an efficient approach to 1,2-disulfonylethenes is of considerable interest. Generally, the preparation of 1,2-disulfonylethenes relies on several classic methods, the oxidation of 1,2-dithioethenes or 1-thio-2-sulfonylethenes,[3] the condensation of 1-sulfonyl-2,2-dichloroethanes with sodium sulfonates,[4] the nucleophilic substitution of alkenyliodonium salts with sodium sulfonates,[5] and cycloaddition or Michael-type addition of bis(sulfonyl)acetylene.[6] However, these approaches always involve complex substrates and/or multiple synthetic steps. Thus, developing a simple and efficient method to construct 1,2-disulfonylethenes is necessary.

Decarboxylative coupling is one of the most powerful methods for the construction of C−C and C−heteroatom bonds due to the easy storage and operability of carboxylic acids, the high selectivity, and the release of a nontoxic byproduct, CO_2.[7,8] Recently, the use of alkynyl carboxylic acids as terminal alkyne surrogates has received increased attention.[9−14] Since Lee and co-workers first demonstrated a palladium-catalyzed decarboxylative coupling reaction of alkynyl carboxylic acids with aryl halides to afford unsymmetric diarylalkynes,[9a] numerous decarboxylative coupling reactions of alkynyl carboxylic acids have been developed to construct C−C,[9] C−N,[10] C−P,[11] C−Si,[12] C−B,[13] and C−S[14] bonds. In this context, much effort has been made to create C−S bonds by decarboxylative sulfonylation of alkynyl carboxylic acids (Scheme 1). Jiang et al. reported a palladium-catalyzed coupling reaction for the formation of internal alkynes and vinyl sulfones from alkynyl carboxylic acids and sodium sulfonates.[14a] Mao, Zhang, and co-workers subsequently developed Cu/Fe-cocatalyzed sulfonylation of aromatic propiolic acids with sulfonyl hydrazides to construct vinyl sulfones.[14b] Brønsted acid[14c] and base[14d] promoted decarboxylative sulfonylations were also achieved (Scheme 1a). Kuhakarn et al. reported an I_2-catalyzed decarboxylative coupling

Scheme 1. Decarboxylative Sulfonylation of Alkynyl Carboxylic Acids



of arylacetylenic acids for the synthesis of arylacetylenic sulfones (Scheme 1b).[14e] Very recently, copper(I)-catalyzed decarboxylative sulfonylation of arylpropiolic acids have also been developed to construct *β*-keto sulfones by the Wu group (Scheme 1c).[14f] Despite these advances, to the best of our knowledge, the decarboxylative disulfonylation of alkynyl carboxylic acids has not been well developed up to now. On the basis of our continuing interest in decarboxylative coupling and sulfonylation,[15] herein we report a novel, efficient, and practical copper-catalyzed decarboxylative disulfonylation of alkynyl carboxylic acids with sulfinic acids to give (*E*)-1,2-disulfonylethenes.

At the outset of our investigation, phenylpropiolic acid (**1a**) and benzenesulfinic acid (**2a**) were chosen as the model substrates to optimize reaction conditions for the decarboxylative disulfonylation. When phenylpropiolic acid was treated with 3.0 equiv of benzenesulfinic acid in the presence of 10 mol % of $Cu(ClO_4)_2·6H_2O$ and 3.0 equiv of $K_2S_2O_8$ in CH_3CN/H_2O (2/

Received: December 17, 2017

DOI: 10.1021/acs.orglett.7b03922
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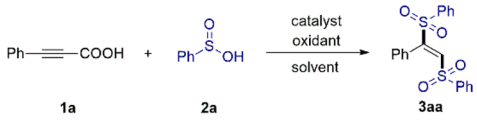
1 (v/v)) at 80 °C for 12 h under an argon atmosphere, to our delight, the reaction proceeded smoothly and afforded the desired disulfonylated product **3aa** in 52% yield with 98/2 *E*/*Z* selectivity. Subsequently, various oxidants were screened to improve the reaction efficiency, and $(NH_4)_2S_2O_8$ proved to be better than the other oxidants (see the Supporting Information). In addition to $Cu(ClO_4)_2{\cdot}6H_2O$, other Cu catalysts including CuCl, CuO, $Cu(OAc)_2$, and $CuSO_4{\cdot}5H_2O$ were also tested, and the results indicate that $Cu(ClO_4)_2{\cdot}6H_2O$ was still the best choice (Table 1, entries 1−5). Different solvents were

Table 1. Screening of Reaction Conditions[a]



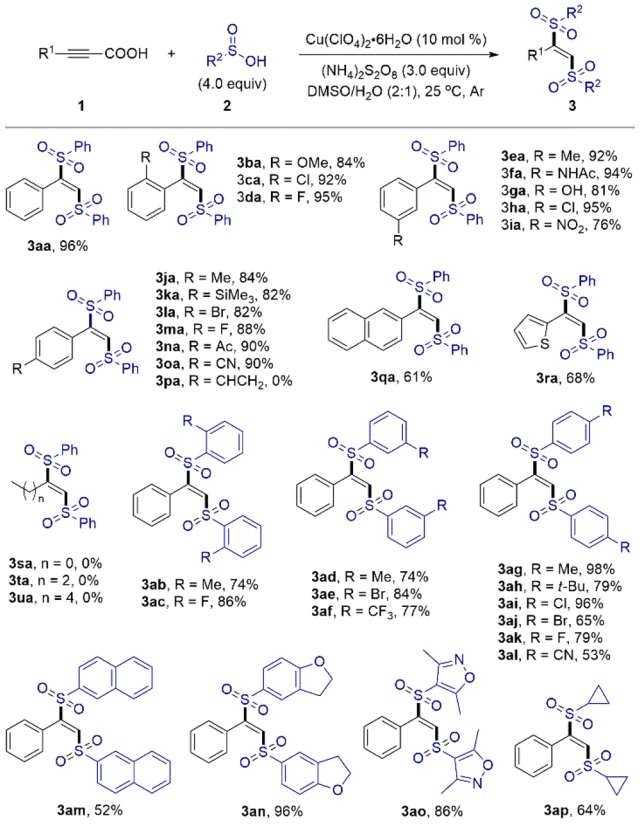
entry	catalyst	solvent (v/v)	yield (%)	*E*/*Z*
1	$Cu(ClO_4)_2{\cdot}6H_2O$	CH_3CN/H_2O (2/1)	55	99/1
2	CuCl	CH_3CN/H_2O (2/1)	48	>99/1
3	CuO	CH_3CN/H_2O (2/1)	53	98/2
4	$Cu(OAc)_2$	CH_3CN/H_2O (2/1)	trace	
5	$CuSO_4{\cdot}5H_2O$	CH_3CN/H_2O (2/1)	50	99/1
6	$Cu(ClO_4)_2{\cdot}6H_2O$	acetone/H_2O (2/1)	39	99/1
7	$Cu(ClO_4)_2{\cdot}6H_2O$	DMF/H_2O (2/1)	50	>99/1
8	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (2/1)	77	>99/1
9	$Cu(ClO_4)_2{\cdot}6H_2O$	toluene/H_2O (2/1)	35	94/6
10[b]	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (2/1)	93	>99/1
11[b]	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (3/1)	88	>99:1
12[b]	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (1/1)	84	>99:1
13[b,c]	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (2/1)	90	>99:1
14[b−d]	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (2/1)	96	>99/1
15[b−e]	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (2/1)	trace	
16[b,c,d]		DMSO/H_2O (2/1)	0	
17[b−d,f]	$Cu(ClO_4)_2{\cdot}6H_2O$	DMSO/H_2O (2/1)	0	

[a]Reaction conditions unless specified otherwise: **1a** (0.3 mmol), **2a** (3.0 equiv), catalyst (10 mol %), and $(NH_4)_2S_2O_8$ (3.0 equiv) in solvent (3.0 mL) at 80 °C for 12 h under Ar. Isolated yield. *E*/Z ratios were determined by ^{1}H NMR of the crude products. [b]Using 4.0 equiv of **2a**. [c]At 25 °C. [d]For 6 h. [e]In air. [f]The reaction was carried out in the absence of $(NH_4)_2S_2O_8$.

investigated, and among them DMSO/H_2O exhibited unmatched efficacy for the decarboxylative disulfonylation (Table 1, entries 6−9). With regard to the amount of benzenesulfinic acid **2a**, when the amount of **2a** was increased to 4.0 equiv, the yield of 1,2-disulfonylethene improved to 93% (Table 1, entry 10). The catalyst loading and effect of oxidant stoichiometry were also examined, and the results indicate that 10 mol % of catalyst with 3.0 equiv of oxidant was the best choice (see the Supporting Information). The role of H_2O might be to improve the solubility of the oxidant. Among the ratios of DMSO and H_2O examined, the 2/1 ratio was preferred (Table 1, entries 10−12). Importantly, it was found that the reaction operated equally well at room temperature (Table 1, entry 13). The effect of reaction time was also investigated, and the best choice was 6 h (Table 1, entry 14). Only a trace amount of **3aa** was observed when the reaction was carried out in an atmosphere of air (Table 1, entry 15). Control experiments showed that no reaction occurred in the absence of either $Cu(ClO_4)_2{\cdot}6H_2O$ or $(NH_4)_2S_2O_8$ (Table 1, entries 16 and 17).

With the optimized reaction conditions, a variety of alkynyl carboxylic acids were subjected to the optimized conditions to evaluate the scope of the decarboxylative disulfonylation, and the results are summarized in Table 2. The electronic properties of

Table 2. Substrate Scope[a,b]



[a]All of the reactions were carried out in the presence of 0.3 mmol of **1**, 4.0 equiv of **2**, 10 mol % of $Cu(ClO_4)_2{\cdot}6H_2O$, and 3.0 equiv of $(NH_4)_2S_2O_8$ in 3.0 mL of DMSO/H_2O (2/1) at room temperature for 6 h under Ar. [b]Isolated yield.

the substituents had no apparent effect on the reaction. Arylpropiolic acids bearing electron-donating (Me, MeO, OH, Me_3Si, and AcNH) or electron-withdrawing groups (F, NO_2, Ac, and CN) were compatible with the reaction conditions, affording the desired products **3aa**−**3oa** in satisfactory yields. Halogen groups on the aromatic ring such as Cl and Br also could be well tolerated in the copper-catalyzed reaction, which provided opportunities for further functionalization. However, vinyl-substituted phenylpropiolic acid was not a suitable substrate. Steric hindrance in the ortho-substituted arylpropiolic acids does not have a considerable influence on the transformation, and good to excellent yields were provided in all cases. In addition, 2-naphthyl-substituted phenylpropiolic acid also underwent the reaction smoothly, giving the product **3qa** in 61% yield. Thienylpropiolic acid, a heteroaromatic substrate, was applicable under the standard conditions as well and afforded the desired product **3ra** in reasonable yield. Notably, alkylpropiolic acids, such as 2-butynoic acid, 2-hexynoic acid, and 2-octynoic acid, were not suitable substrates due to their low reactivity (**3sa**−**3ua**).

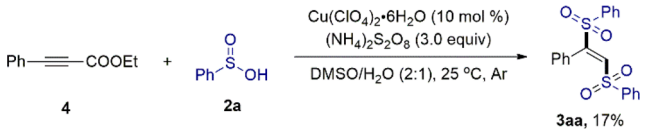
The scope of sulfinic acids was also examined. Both electron-rich and -poor benzenesulfinic acids could be transferred to the 1,2-disulfonylethenes **3ab**−**3al** in good to excellent yields, and a series of functional groups, such as alkyl, halides, nitrile, and trifluoromethyl, were compatible with the reaction conditions.

DOI: 10.1021/acs.orglett.7b03922
Org. Lett. XXXX, XXX, XXX−XXX

Ortho-substituted benzenesulfinic acids also exhibited a high reactivity (**3ab**,**ac**), indicating that steric effects on the phenyl ring are not evident in this transformation. Moreover, 2-naphthylsulfinic acid was also a suitable substrate, albeit in moderate yields. It was pleasant to find that heterocycle-substituted sulfinic acids were compatible with this catalytic system, leading to the expected products **3an**,**ao** in 96% and 86% yields, respectively. Additionally, this transformation is not limited to aromatic sulfinic acids; cyclopropylsulfinic acid also reacted well with phenylpropiolic acid, giving the corresponding disulfonylethene **3ap** in 64% yield.

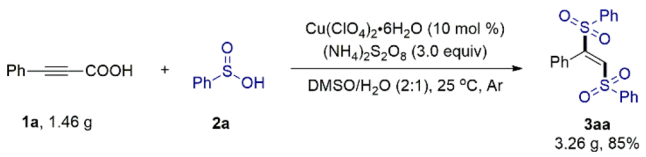
We further explored the deesterification disulfonylation of ethyl 3-phenylpropiolate **4** with benzenesulfinic acid **2a** under the optimal reaction conditions. However, poor conversion was observed, and the disulfonylethene **3aa** was obtained in only 17% yield (Scheme 2). Additionally, cinnamic acid was inert under the standard conditions.

Scheme 2. Deesterification Disulfonylation of Ethyl 3-Phenylpropiolate



To show the potential applications of this protocol, a gram-scale reaction was carried out. As shown in Scheme 3, the

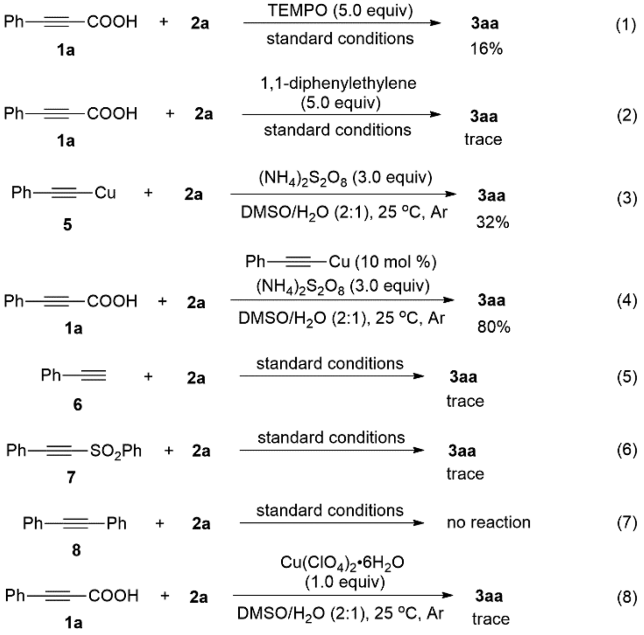
Scheme 3. Gram-Scale Synthesis



reaction of 10.0 mmol of phenylpropiolic acid with benzenesulfinic acid under the standard reaction conditions gave 3.26 g of the desired product in 85% yield. The result indicates that the decarboxylative disulfonylation could be readily scaled up with similar efficiency.

In order to elucidate the reaction mechanism, several control experiments were carried out (Scheme 4). Initially, when 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) was added to the standard reaction conditions, inhibition of the reaction was observed, and the disulfonylethene **3aa** was obtained in only 16% yield. This reaction was also found to be remarkably suppressed by 1,1-diphenylethylene, and only trace amounts of **3aa** were detected. These two results suggest that the transformation should proceed through a radical pathway. Furthermore, when phenylpropiolic acid was replaced by (phenylethynyl)copper **5**, the desired product **3aa** was observed in 32% yield in the absence of $Cu(ClO_4)_2 \cdot 6H_2O$. Meanwhile, the reaction proceeded smoothly and afforded product **3aa** in 80% yield when (phenylethynyl)copper was employed as the catalyst instead of $Cu(ClO_4)_2 \cdot 6H_2O$. The reaction of benzenesulfinic acid with ethynylbenzene or ((phenylethynyl)sulfonyl)benzene was also performed under the standard conditions, and only trace amounts of **3aa** were detected. When diphenylacetylene was employed in the reaction with benzenesulfinic acid, no reaction was observed. These results indicate that alkynyl copper is probably a key intermediate in this transformation, while

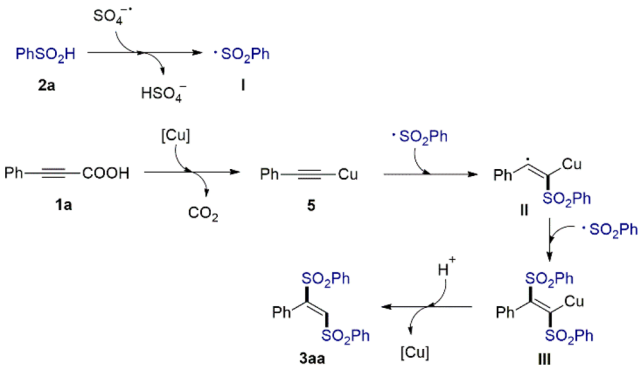
Scheme 4. Control Experiments



terminal alkyne and acetylenic sulfone are not involved in the process of decarboxylative disulfonylation of alkynyl carboxylic acids. Additionally, only trace amounts of disulfonylated product were detected and the starting material phenylpropiolic acid was recovered from the stoichiometric reaction in the absence of an oxidant, thus implying that sulfinic acid is oxidized by $(NH_4)_2S_2O_8$ to generate a sulfonyl radical and not $Cu(ClO_4)_2 \cdot 6H_2O$.

A possible mechanism for this transformation is proposed, as shown in Scheme 5, on the basis of the above experimental

Scheme 5. Proposed Mechanism



results and the precedent literature.[9i,11c,16] Initially, sulfinic acid **2a** is oxidized by ammonium persulfate to generate sulfonyl radical **I**. Meanwhile, the decarboxylation of alkynyl carboxylic acid with the assistance of a copper salt gives the alkynyl copper species **5**. Subsequently, addition of the sulfonyl radical to the alkynyl copper species **5** leads to alkenyl radical **II**, which further interacted with the second sulfonyl radical to afford intermediate **III**. Finally, intermediate **III** was quenched by proton to yield the desired disulfonylethene **3aa**.

In conclusion, we have developed a novel and practical copper-catalyzed decarboxylative disulfonylation of alkynyl carboxylic acids with sulfinic acids in aqueous solution. This transformation is characterized by its wide substrate scope, high stereoselectivity

DOI: 10.1021/acs.orglett.7b03922
Org. Lett. XXXX, XXX, XXX−XXX

for *E* isomers, mild reaction conditions, and utilization of readily available reagents, thus providing an efficient and practical approach to form (*E*)-1,2-disulfonylethenes. Preliminary mechanistic studies revealed that this reaction might involve a radical process. Further mechanistic investigation and the synthetic applications of this reaction are underway.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b03922.

Experimental procedures, characterization data of all new compounds, and ^{1}H and ^{13}C NMR spectra (PDF)

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

This research was supported by the National Natural Science Foundation of China (Nos. 21662021 and 21402071) and the Science and Technology Planning Project of Yunnan Province (No. 2017FB016).

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DOI: 10.1021/acs.orglett.7b03922
Org. Lett. XXXX, XXX, XXX−XXX