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Sulfonylation

Silver(I)-Promoted Radical Sulfonylation of Allyl/Propargyl Alcohols: Efficient Synthesis of γ-Keto Sulfones

Guichun Fang[+],[a] Jianquan Liu[+],[a] Weidong Shang,[a] Qun Liu,[a] and Xihe Bi*[a, b]

Abstract: An efficient Ag_2CO_3-promoted sulfonylation of allyl/propargyl alcohols with sodium sulfinates has been developed. The reaction tolerates a wide range of functional groups to deliver γ-keto sulfones in high yields (up to 93%). Propargyl alcohols furnished trimerization product 1,3,5-triaroylbenzenes in the presence of sodium methanesulfinate under the standard conditions. A mechanism involving a sulfonyl radical was suggested.

Keto sulfones are well known to be a highly valuable class of compounds which are versatile building blocks and valuable intermediates in organic synthesis[1] and pharmaceutical chemistry.[2] The addition of sulfonyl radicals to unsaturated systems such as alkenes and alkynes represents a particularly useful approach to such compounds.[3] With this strategy, various sulfonyl radicals, generated from sulfonyl hydrazides,[4] dimethyl sulfoxide,[5] sulfinates,[6] sulfinic acids,[7] and thiophenols,[8] etc. have been successfully employed in these radical reactions to furnish keto sulfones. Among these, the synthetic methodologies of β-keto sulfones have been widely explored.[9] However, compared to the rapid development of β-keto sulfones, the direct synthesis of γ-keto sulfones in terms of sulfonyl radical addition is not well documented. Recently, a number of metal-catalyzed or metal-free protocols to construct sulfonylated oxindoles from *N*-arylacrylamide derivatives via a radical cascade process have been reported, which effectively delivered heterocycles bearing a γ-keto sulfone moiety in high yields (Figure 1a). However, in these cases the access to the γ-keto sulfone moiety is highly limited to structurally specialized radical acceptors. Most recently, a hydrosulfonylation of chalcones with arylsulfonyl hydrazides leading to γ-keto sulfones in good to excellent yields was reported, using 20 mol% of tetra-*n*-butylammonium iodide (TBAI), along with stoichiometric amounts of benzoyl peroxide (BPO) and *tert*-butyl hydroperoxide (TBHP) as co-oxidants (Figure 1b).[10] However, in comparison with the high efficiency of arylsulfonyl hydrazides, alkylsulfonyl hydrazides were inapplicable under the employed conditions. Therefore, this definitely calls for the development of a more efficient, general catalytic approach for the synthesis of γ-keto sulfones because of their potential applicability in organic synthesis and their diverse biological properties.[11] Prompted by these facts, we report herein the first example of an efficient Ag_2CO_3-promoted sulfonylation of terminal allyl/propargyl alcohols with sodium sulfinates to access γ-keto sulfones in good to excellent yields, in which both aryl- and alkyl-substituted sodium sulfinates resulted in a successful transformation (Figure 1c).[12] The sulfonylation offered an access to the synthesis of γ-keto sulfones via a radical route through a one-step procedure.[13,14]

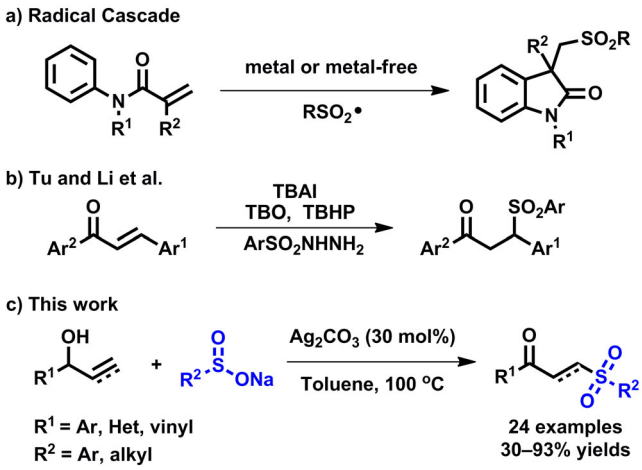

Figure 1. Synthesis of γ-keto sulfones via radical processes.

Our initial investigation commenced with the exploratory reaction between 1-phenylprop-2-en-1-ol (**1a**) and sulfinate **2a** in the presence of 30 mol% of Ag_2CO_3 in 1,4-dioxane at 100 °C in air.[15] Delightfully, the reaction proceeded well, furnishing the desired product, γ-keto sulfone **3a**, in 76% yield (Table 1, entry 1). Compared to Ag_2CO_3, other silver salts (Ag_3PO_4, $AgNO_3$, AgOTf, and AgOAc) were far less effective or even failed in this transformation (entries 2–5). It is worth mention-

[a] G. Fang,[+] J. Liu,[+] W. Shang, Q. Liu, Prof. X. Bi
Jilin Province Key Laboratory of Organic Functional Molecular Design & Synthesis
Department of Chemistry
Northeast Normal University
Renmin Str. 5268#, Changchun 130024 (China)
E-mail: bixh507@nenu.edu.cn
Homepage: http://www.bigroup.com.cn/

[b] Prof. X. Bi
State Key Laboratory of Elemento-Organic Chemistry
Nankai University
Tianjin 300071 (China)

[+] These authors contributed equally to this work.

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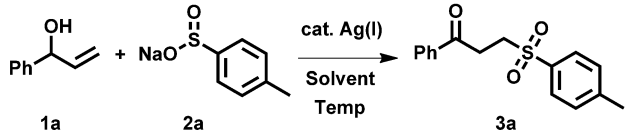
Table 1. Optimization of the reaction conditions.[a]



1a 2a 3a

Entry	Cat.	Solvent	T [°C]	Yield[b] [%]
1	Ag_2CO_3	1,4-Dioxane	100	76
2	Ag_3PO_4	1,4-Dioxane	100	trace
3	$AgNO_3$	1,4-Dioxane	100	0
4	AgOTf	1,4-Dioxane	100	0
5	AgOAc	1,4-Dioxane	100	trace
6	–	1,4-Dioxane	100	0
7	Ag_2CO_3	DMF	100	0
8	**Ag_2CO_3**	**Toluene**	**100**	**91**
9	Ag_2CO_3	Toluene	50	42
10[c]	Ag_2CO_3	Toluene	100	20

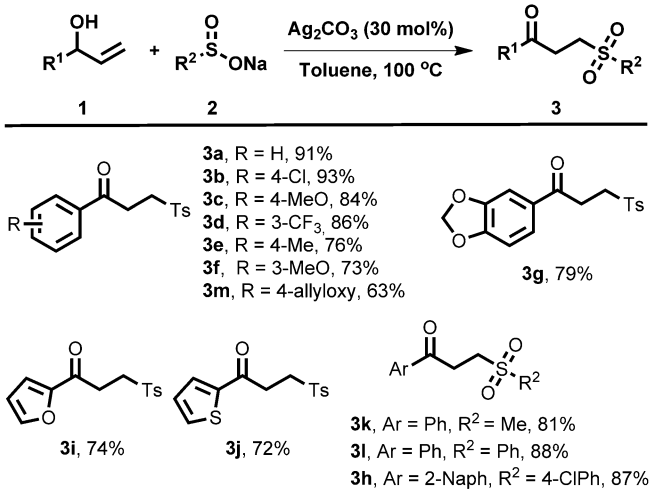
[a] Reaction conditions: **1a** (0.5 mmol), **2a** (1.5 mmol) and the catalyst (30 mol%) in solvent (2.0 mL) at 100 °C under air atmosphere for 6 h. [b] Isolated yield of **3a**. [c] Under N_2 atmosphere.

ing that no reaction was observed in the absence of silver salts (entry 6). Further screening of solvents identified toluene as the most suitable solvent (entries 7–8). Decreasing the reaction temperature to 50 °C resulted in a significant drop in the yield of **3a** (entry 9). Interestingly, the yield of **3a** considerably decreased to 20% under a nitrogen atmosphere (entry 10). This observation demonstrates that dioxygen has a significant impact on the efficiency of the process.

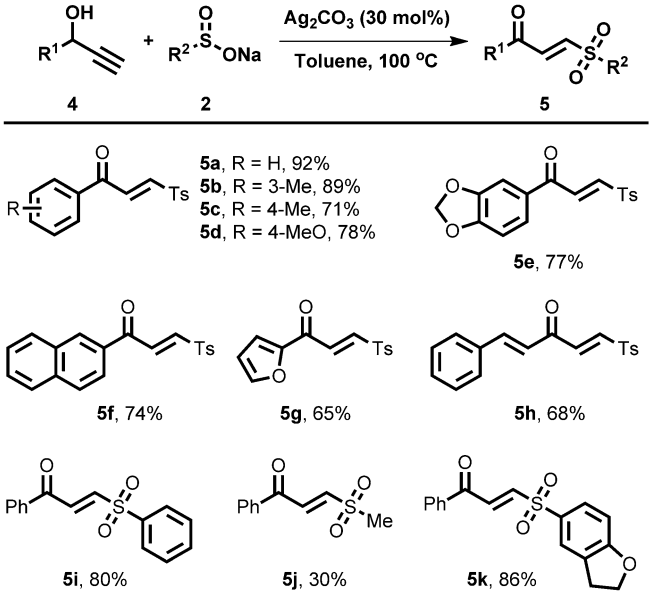
Once the optimal reaction conditions were established, efforts were then focused on the scope of this method (Scheme 1). A series of arylallyl alcohols **1** worked well with various sodium sulfinates **2**. A variety of electron-donating (R = OMe or Me) or -withdrawing groups (R = Cl or F_3C) at the 3- or 4-position of the aromatic ring were all compatible, affording the expected γ-keto sulfones in yields of 73–93% (**3a–g**). Interestingly, allyl alcohols with an allyloxy group on the aromatic ring also furnished sulfone **3m** in a yield of 63%. In addition, allyl alcohols containing fused aromatic groups as well as heteroaromatic groups all reacted smoothly to give the desired products (**3h–j**). Delightfully, the aliphatic sodium sulfinate also possessed high reactivity under the standard conditions, furnishing the desired product **3k** in 81% yield.

In view of these interesting results, we further extended the substrate scope of this strategy to propargyl alcohols (Scheme 2). With sodium *p*-methylphenylsulfinate (**2a**) as the sulfonyl source, a variety of phenyl propargyl alcohols bearing electron-donating or -withdrawing substituents resulted in a smooth conversion to afford the desired products in yields of 71–92% (**5a–e**). Moreover, 2-naphthyl (**4f**) and 2-furyl (**4g**) alkynyl carbinols also furnished good yields of γ-keto sulfones (**5f**, **5g**), thus further expanding the scope of this sulfonylation reaction. It is noteworthy that trimerization was observed when styryl alkynyl carbinol (**4h**) was treated under the standard conditions, although the desired product **5h** was obtained in a high yield (sulfonylation/trimerization = 10:1, 68% combined yield). The reaction was also tested with other sodium arenesulfinates. As illustrated in Scheme 2, the sulfonylation was general with respect to structural variations on the sodium sulfinate. Importantly, when the *S*-substituent on the sodium sulfinate **2** was an aryl group, the reaction appeared to be more effective under the standard conditions with propargyl alcohol **4a** than sodium methanesulfinate ($MeSO_2Na$) (**5i–5k**). However, no desired γ-keto sulfones were obtained when aliphatic alkynyl carbinols were used.

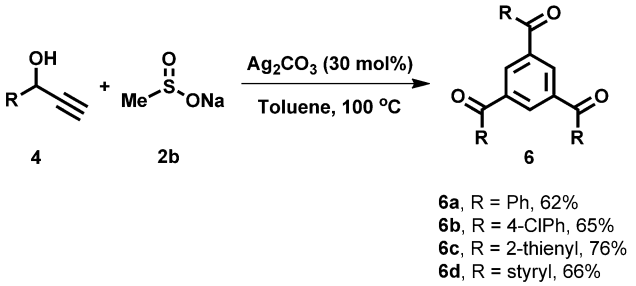
Interestingly, the treatment of the propargyl alcohols **4** with $MeSO_2Na$ (**2b**) under the standard conditions allowed the trimerization of propargyl alcohols, furnishing 1,3,5-triaroylbenzenes as main products (**6a–d**) (Scheme 3). The transition-



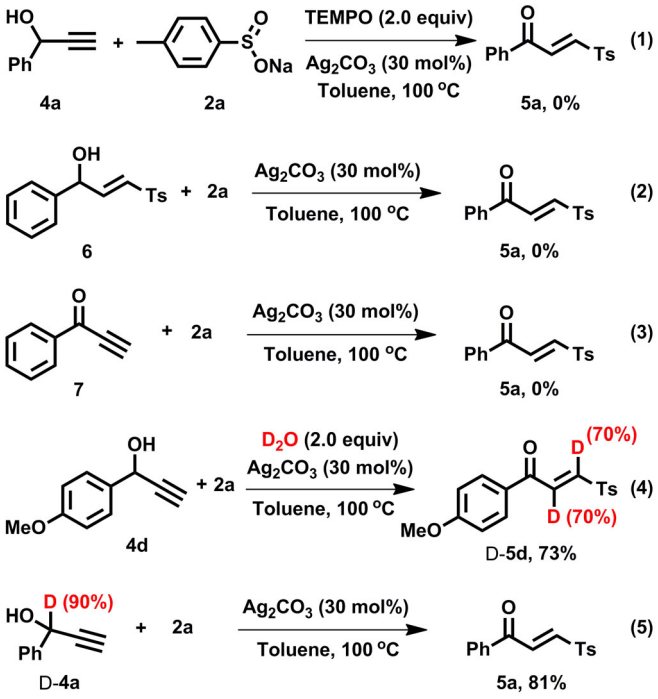
Scheme 1. Sulfonylation reaction of different allyl alcohols with sodium sulfinates.



Scheme 2. Sulfonylation reaction of different propargyl alcohols with sodium sulfinates.



Scheme 3. Trimerization of propargyl alcohols with sodium methanesulfinate to access 1,3,5-triaroylbenzenes.

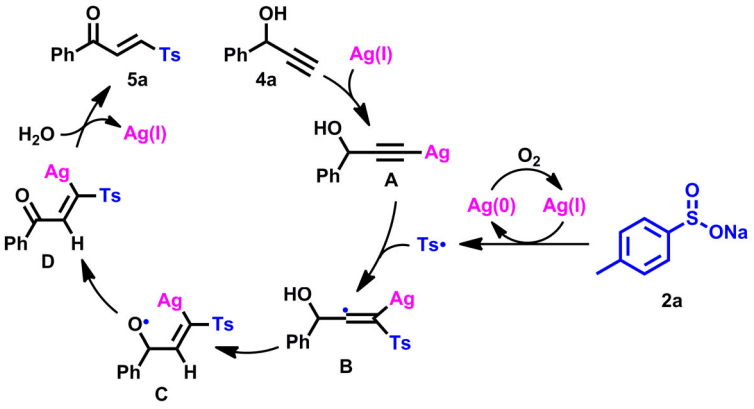


Scheme 4. Mechanism investigations.

metal-catalyzed [2+2+2] cycloaddition of alkynes has been regarded as one of the most classical processes to prepare benzenes and analogous aryl derivatives.[16] Generally, the 1,3,5-triaroylbenzene skeletons are constructed by base-catalyzed Michael-type cyclotrimerization of the corresponding 1-aryl-2-propyn-1-ones.[17] In comparison with the alkynyl ketones, reactions of propargyl alcohols as the starting material require multiple steps.[18] Remarkably, the present strategy appears to be a straightforward and selective route to access 1,3,5-triaroylbenzenes in a single-step operation. The formation of such compounds could be attributed to the sulfonyl radical-induced [2+2+2] cycloaddition of propargyl alcohols **4**.[19] It is worth noting that the reaction proceeded without pre-oxidation of propargyl alcohols.[20] Consequently, the radical protocol described here, starting from propargyl alcohols, constitutes a novel and concise approach to synthesize 1,3,5-triaroylbenzenes.

To probe the plausible mechanistic pathway for this reaction, several control experiments were designed and performed (Scheme 4). Based on literature studies,[3c] a radical trapping experiment was initiated. In the presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (2.0 equiv), under the standard conditions, the formation of γ-keto sulfone **5a** was completely suppressed, which indicates that the reaction probably proceeds through a radical pathway [Eq. (1)]. Additionally, under optimal conditions, β-hydroxy sulfone **6** was not oxidized to form the corresponding γ-keto sulfone **5a**, implying that it was not the intermediate in the transformation [Eq. (2)]. When 1-phenylprop-2-yn-1-one **7** was treated under the standard conditions, no expected sulfonylated product **5a** was observed [Eq. (3)]. This observation demonstrated that the hydroxyl group played a vital role in the success of this transformation. Moreover, a stepwise mechanism, involving oxidation of the hydroxyl group and subsequent Michael addition of the sulfonyl radical to an alkynone intermediate, was excluded. Furthermore, a deuterium-labeling experiment was performed. When D_2O was added into the reaction system of substrate **4d**, the α,β-dideuterated sulfonylation product D-**5d** was obtained in 73% yield, with 70% deuterium incorporation [Eq. (4)]. This result implied that an organosilver intermediate might be generated during the reaction process. Treatment of α-deuterated propargyl alcohol D-**4a** under standard conditions resulted in 81% yield of **5a**, without deuterated product observed [Eq. (5)], which suggested that the α-H of propargyl alcohol **4a** was not the source of the olefinic hydrogen of product **5a**.

A plausible mechanism was devised for the current system, based on literature precedence and the above-mentioned results (Scheme 5).[6b,7a,9b,21] First, a sulfonyl radical is generated by silver catalysis. Subsequently, this radical attacks the triple bond of acetylenic silver species **A**, which is generated from the interaction of propargyl alcohol with silver(I) catalyst, to furnish vinyl radical intermediate **B**. The radical **B** abstracts the hydrogen atom on the adjacent hydroxyl group to generate the oxyl radical **C**, which undergoes hydrogen radical abstraction to generate the C=O bond.[6b,22] Finally, protonation of the



Scheme 5. A plausible reaction mechanism.

vinyl silver(I) **D** affords the desired product **5a**, along with releasing the silver(I) catalyst.

In summary, we have developed a mechanistically novel reaction for the synthesis of γ-keto sulfones by the silver(I)-based sulfonylation of various allyl/propargyl alcohols with sodium sulfinates. The scope of allyl/propargyl alcohols and sodium sulfinates is broad to deliver structurally diverse γ-keto sulfones in good to excellent yields. Mechanistic investigations unraveled that the process plausibly involved sequential sulfonyl radical addition to a C–C triple/double bond and a hydrogen atom transfer. Furthermore, 1,3,5-triaroylbenzene derivatives can be successfully generated from propargyl alcohols by this protocol when sodium methanesulfinate is used as radical source. Further mechanistic details and applications of this sulfonylation protocol are currently ongoing in our laboratory.

Experimental Section

General Procedure for the Synthesis of Compounds 3 and 5 (with **3a** as an example): A screw-capped reaction vial was charged with allyl alcohol **1a** (67 mg, 0.5 mmol) and sodium sulfinate **2a** (133.5 mg, 0.75 mmol) in toluene (2.0 mL) and stirred at room temperature. Subsequently, Ag_2CO_3 (41.4 mg, 0.15 mmol) was added. The reaction mixture was stirred at 100 °C until the substrate **1a** was completely consumed as indicated by thin-layer chromatography (TLC). The resulting mixture was concentrated and the residue was taken up in ethyl acetate. The organic layer was dried over $MgSO_4$ and concentrated. Purification of the crude product by flash column chromatography on silica gel afforded the corresponding γ-keto sulfone **3a** in 91% yield.

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Keywords: allyl/propargyl alcohols · gamma-keto sulfones · radical sulfonylation · reaction mechanisms · silver catalysis

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[12] During the preparation of our manuscript, a palladium(II)-catalyzed sulfonylation of allylic alcohols to access γ-keto sulfones was published, in which the reaction proceeds through one-pot palladium(II)-catalyzed generation of enones followed by water-mediated 1,4-addition of organosulfinates, M. Vellakkaran, M. M. S. Andappan, K. Nagaiah, J. B. Nanubolu, *Eur. J. Org. Chem.* **2016**, 3575–3583 and ref. 20–23.

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COMMUNICATION

Sulfonylation

*Guichun Fang, Jianquan Liu, Weidong Shang, Qun Liu, Xihe Bi**

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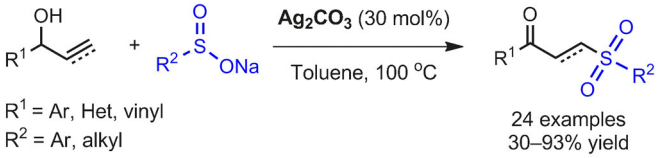

Silver(I)-Promoted Radical Sulfonylation of Allyl/Propargyl Alcohols: Efficient Synthesis of γ-Keto Sulfones

At the terminal: The first example of silver(I)-catalyzed radical sulfonylation of terminal allyl/propargyl alcohols to access γ-keto sulfones with aryl- and alkyl-substituted sodium sulfonates is reported. A mechanism involving a sulfonyl radical insertion is suggested.