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Directing-Group-Assisted C(sp^2)−H Arylsulfonylation from Sulfur Dioxide

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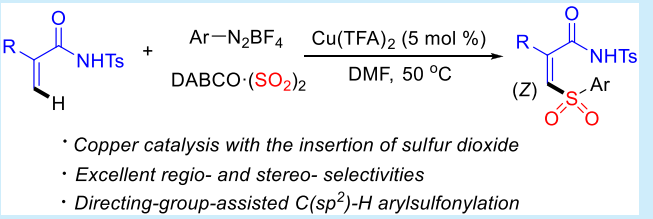
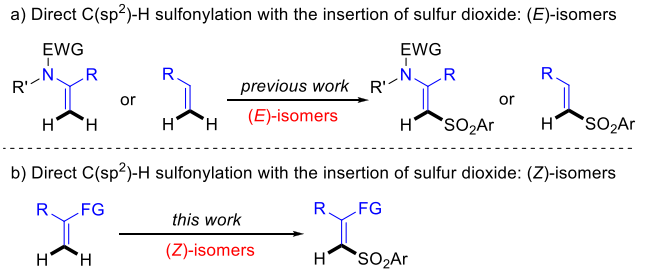
ABSTRACT: A straightforward and stereoselective preparation of (*Z*)-*β*-alkenyl sulfones through a reaction of *N*-tosyl acrylamides, 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide, and aryldiazonium tetrafluoroborates under copper catalysis is accomplished. The direct C(sp^2)−H arylsulfonylation of acrylamides using sulfur dioxide as the sulfonyl source in the presence of copper trifluoroacetate proceeds smoothly, giving rise to (*Z*)-*β*-alkenyl sulfones in good yields. During the reaction process, excellent regio- and stereoselectivities are observed.



Because of its excellent atom economy and step economy, direct C−H functionalization is an efficient strategy for the generation of carbon−carbon or carbon−heteroatom bonds.[1] Among these transformation, the C(sp^2)−H sulfonylation of alkenes is an important approach for the formation of alkenyl sulfones, benefiting from the importance of alkenyl sulfones in pharmaceuticals and natural products.[2] Up to now, continuous methods have appeared and been developed in this field.[3] However, in some cases, the approaches for the direct C(sp^2)−H sulfonylation of olefins still have some limitations in terms of the employment of expensive catalysts and the stoichiometric amount of oxidants or additives. Thus the development of efficient, green, and straightforward methods for the preparation of alkenyl sulfones is highly desirable.

Recently, utilizing 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide (DABCO·(SO$_2$)$_2$) or potassium/sodium metabisulfite as the sulfur dioxide surrogates for the construction of sulfonyl derivatives has attracted much attention.[4] In particular, with this strategy, efforts have been made for the preparation of alkenyl sulfones via C(sp^2)−H sulfonylation. For instance, the C(sp^2)−H sulfonylation of alkenes was accomplished via a reaction of aryldiazonium tetrafluoroborates, sulfur dioxide, and alkenes using copper salt as a catalyst or via a iodide-promoted radical-type insertion of sulfur dioxide.[5a,b] Recently, an iron-chloride-mediated C(sp^2)−H methylsulfonylation of alkenes from sulfur dioxide was achieved.[5c] Loh and coworkers described the C(sp^2)−H sulfonylation of electron-rich olefins (enamides) with sulfur dioxide in the presence of copper(II) triflate or photocatalysis.[5d,e] Despite these achievements, the thermodynamically stable (*E*)-alkenyl sulfones are obtained as the major products (Scheme 1, eq a). So far, there are no examples of the stereoselective preparation of (*Z*)-alkenyl sulfones using sulfur dioxide as the sulfonyl source. Inspired by the recent advances in C(sp^2)−H bond functionalization assisted by directing groups,[6] we envisioned that *N*-tosyl acrylamides would be the choice of substrate for the method development, which would utilize the weakly coordinating tosyl-imide as the directing group.[7,8] Herein we report a straightforward and stereoselective preparation of (*Z*)-*β*-alkenyl sulfones through a reaction of *N*-tosyl acrylamides, 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide, and aryldiazonium tetrafluoroborates under copper catalysis. A radical process involving arylsulfonyl radicals is proposed. With the assistance of tosyl-imide as the directing group, (*Z*)-*β*-alkenyl sulfones are stereoselectively formed (Scheme 1, eq b).

Scheme 1. Synthesis of Alkenyl Sulfones by Using Sulfur Dioxide as the Sulfonyl Source



The studies were initiated from a reaction of *N*-tosyl acrylamide **1a**, DABCO·(SO$_2$)$_2$, and 4-methylphenyldiazonium tetrafluoroborate **2a** in DCE at 80 °C by using copper trifluoroacetate as the catalyst. Fortunately, the desired (*Z*)-2-

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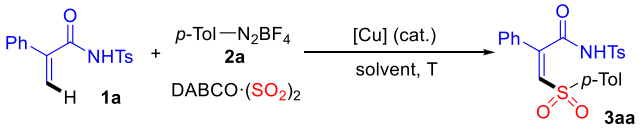
phenyl-*N*,3-ditosylacrylamide **3aa** was observed in 45% yield (Table 1, entry 1). The configuration of compound **3aa** was

Table 1. Initial Studies for the Reaction of *N*-Tosyl Acrylamide 1a, DABCO·$(SO_2)_2$, and 4-Methylphenyldiazonium Tetrafluoroborate 2a[a]



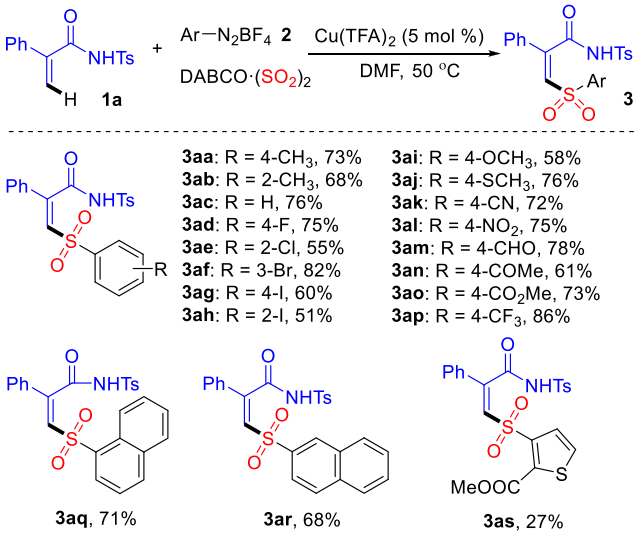
entry	variation of conditions	yield (%)[b]
1	none	45
2	other copper catalysts instead of $Cu(TFA)_2$	21–42
3	DMF instead of DCE	75
4	other solvents instead of DMF	trace–71
5[c]	110 °C	73
6[c]	50 °C	76
7[c]	rt	67
8[c,d]	5 mol % $Cu(TFA)_2$	73
9[c,d]	15 mol % $Cu(TFA)_2$	69
10[c,d]	20 mol % $Cu(TFA)_2$	66
11[c,d]	no $Cu(TFA)_2$	trace

[a]Reaction conditions: *N*-tosyl acrylamide **1a** (0.2 mmol), DABCO·$(SO_2)_2$ (0.3 mmol, 1.5 equiv), 4-methylphenyldiazonium tetrafluoroborate **2a** (0.3 mmol, 1.5 equiv), $Cu(TFA)_2$ (0.02 mmol, 10 mol %), DMF (1.0 mL), 80 °C, 12 h. [b]Isolated yield base on *N*-tosyl acrylamide **1a**. [c]DMF as the solvent. [d]Reaction was performed at 50 °C.

confirmed by X-ray crystallography. Further exploration revealed that other copper catalysts were not as efficient as $Cu(TFA)_2$ (Table 1, entry 2; for details, see the SI). Subsequently, various solvents were examined. It was found that product **3aa** was generated in 75% yield when DMF was employed as the solvent (Table 1, entry 3). However, the results were inferior when other solvents were used instead of DMF (Table 1, entry 4; for details, see the SI). We further evaluated the effect of the reaction temperature. A comparable yield was obtained when the reaction occurred at 110 or 50 °C (Table 1, entries 5 and 6). A lower yield was observed when this transformation was performed at room temperature (67% yield, Table 1, entry 7). Because these results showed that the temperature effect was negligible, we reasoned that the efficiency might be affected by the copper catalyst. Thus the loading of $Cu(TFA)_2$ was examined, which showed that 5 mol % of copper catalyst was optimal (73%, Table 1, entry 8 vs entries 9 and 10). Only a trace amount of compound **3aa** was obtained without the addition of $Cu(TFA)_2$ (Table 1, entry 11). Furthermore, *N*-acetyl acrylamide, *N*-benzoyl acrylamide, and *N*-Boc acrylamide were examined in this transformation. However, it was found that these acrylamides were not suitable under the conditions, and all reaction systems were complex.

Subsequently, we started to explore the substrate scope for the stereoselective preparation of (*Z*)-*β*-alkenyl sulfones through a reaction of *N*-tosyl acrylamides **1**, 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide, and aryldiazonium tetrafluoroborates **2** under copper catalysis. The reaction of various aryldiazonium tetrafluoroborates **2** was investigated at the beginning (Scheme 2). As shown in Scheme 2, aryldiazonium tetrafluoroborates **2** bearing different groups on the aromatic ring reacted with *N*-tosyl acrylamide **1a** and DABCO·$(SO_2)_2$, smoothly leading to the corresponding

Scheme 2. Reaction of *N*-Tosyl Acrylamide 1a, DABCO·$(SO_2)_2$, and Aryldiazonium Tetrafluoroborates 2[a,b]



[a]Reaction conditions: *N*-tosyl acrylamide **1a** (0.3 mmol), DABCO·$(SO_2)_2$ (0.45 mmol, 1.5 equiv), aryldiazonium tetrafluoroborates **2a–s** (0.45 mmol, 1.5 equiv), $Cu(TFA)_2$ (0.015 mmol, 5 mol %), DMF (1.5 mL), 50 °C, 12 h. [b]Isolated yield based on *N*-tosyl acrylamide **1a**.

products **3aa–ap** in 51–86% yields. A range of functional groups including CN, NO_2, CHO, COMe, CO_2Me, and CF_3 were compatible under the conditions. Notably, aryldiazonium tetrafluoroborates **2q** and **2r** bearing *α*- and *β*-naphthyl groups were tolerated as well, giving rise to the corresponding products **3aq** and **3ar** in 71 and 68% yields, respectively. A substrate bearing the heterocyclic skeleton (thiophenyl group) was found to be suitable in this transformation, although the yield was not satisfactory (27% yield).

The substrate scope of *N*-tosyl acrylamides was further evaluated (Scheme 3). In general, 4-methylphenyldiazonium tetrafluoroborate **2a** well reacted with DABCO·$(SO_2)_2$ and *N*-tosyl acrylamides **1b–j** containing a variety of substituents, producing the corresponding products **3ba–ja** in moderate to good yields (53–88%). Additionally, *α*- and *β*-naphthyl-derived *N*-tosyl acrylamides were also amenable to the conditions, providing products **3na** and **3oa** in 51 and 67% yields, respectively. To our delight, *α*-alkyl-substituted substrates were also effective, leading to compounds **3ka–ma** in 35–70% yields. However, the reaction failed to afford product **3pa**. Methacrylamide was not workable under the conditions. Additionally, we amplified the model reaction (Table 1) to a 1.0 mmol scale, and (*Z*)-*β*-alkenyl sulfone **3aa** was produced in 77% yield. Moreover, the gram scale showed that compound **3aa** could be formed in 81% yield (1.48 g; see the Supporting Information).

To clarify the possible reaction pathway, several control experiments were performed (Scheme 4). It was found that the addition of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) would hamper the reaction, revealing that this transformation might proceed through a radical process (Scheme 4, eq 1). Additionally, arylsulfonyl radical could be captured by ethene-1,1-diyldibenzene, affording compound **4** in a reasonable yield (Scheme 4, eqs 2 and 3).

On the basis of the previously described control experiments and related reports,[7a,9] we presented a plausible reaction pathway (Scheme 5). We speculated that a Cu^{II} complex **A**

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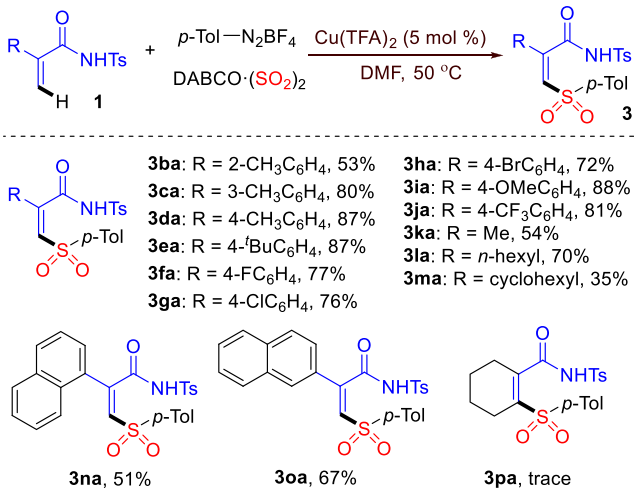
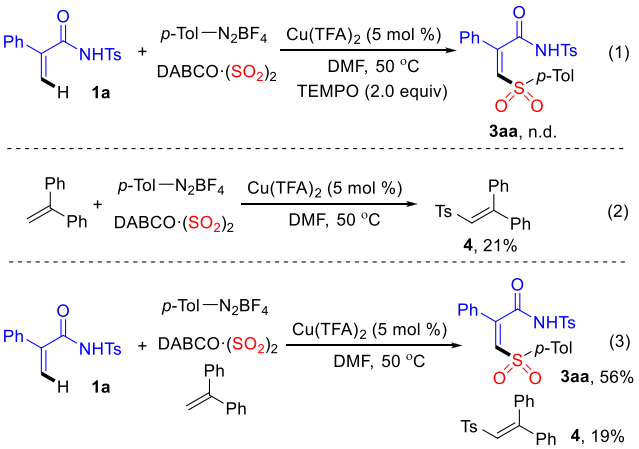
Scheme 3. Reaction of *N*-Tosyl Acrylamides 1, DABCO·$(SO_2)_2$, and 4-Methylphenyldiazonium Tetrafluoroborate 2a[a,b]

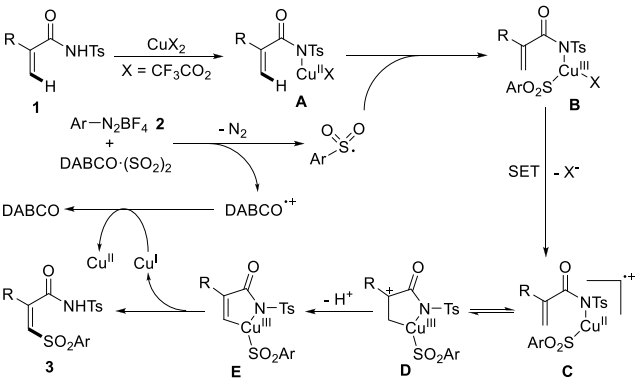


[a]Reaction conditions: *N*-tosyl acrylamides **1b–p** (0.3 mmol), DABCO·$(SO_2)_2$ (0.45 mmol, 1.5 equiv), 4-methylphenyldiazonium tetrafluoroborate **2a** (0.45 mmol, 1.5 equiv), $Cu(TFA)_2$ (0.015 mmol, 5 mol %), DMF (1.5 mL), 50 °C, 12 h. [b]Isolated yield based on *N*-tosyl acrylamide **1**.

Scheme 4. Control Experiments



Scheme 5. Plausible Mechanism



would be formed initially by the treatment of $Cu(TFA)_2$ catalyst with *N*-tosyl acrylamide **1**. Meanwhile, an arylsulfonyl radical and a radical cation DABCO$^{\cdot+}$ would be produced from aryldiazonium tetrafluoroborate **2** and 1,4-diazabicyclo[2.2.2]-octane-sulfur dioxide. Then, Cu^{II} complex **A** would combine with arylsulfonyl radical to furnish Cu^{III} intermediate **B**, which would undergo a single electron transfer, affording intermediate **C**. The interconversion of the cationic alkene radical with the metal center in an intramolecular mode would afford Cu^{III} intermediate **D**. Subsequently, the deprotonation of intermediate **D** would give rise to Cu^{III} intermediate **E**, which would then produce the desired (*Z*)-*β*-alkenyl sulfone **3** via reductive elimination and regenerate the copper catalyst to realize the catalytic cycle.

In conclusion, we have described a straightforward and stereoselective preparation of (*Z*)-*β*-alkenyl sulfones through a reaction of *N*-tosyl acrylamides, 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide, and aryldiazonium tetrafluoroborates under copper catalysis. The direct C(sp^2)–H arylsulfonylation of acrylamides using sulfur dioxide as the sulfonyl source in the presence of copper trifluoroacetate proceeds smoothly, giving rise to (*Z*)-*β*-alkenyl sulfones in good yields. During the reaction process, excellent regio- and stereoselectivities are observed. A radical process involving arylsulfonyl radicals is proposed. With the assistance of tosyl-imide as the directing group, (*Z*)-*β*-alkenyl sulfones are stereoselectively formed. Moreover, a gram-scale synthesis is presented, which demonstrates the synthetic practicability of this method.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c02400.

Detailed experimental procedures, crystallographic data, characterization data, and NMR spectra for all compounds (PDF)

Accession Codes

CCDC 2012716 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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