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DDQ-mediated regioselective C–S bond formation: efficient access to allylic sulfides†

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A protocol for the synthesis of allylic sulfides from simple allylic hydrocarbons with thiophenol derivatives *via* a DDQ-mediated oxidative dehydrogenation strategy is described. This reaction possesses good functional group compatibility, broad substrate scope, and high atom- and step-economy. Moreover, the synthetic utility of this method can be highlighted by its application in the synthesis of versatile organo-sulfur compounds.

Sulfur-containing compounds play a significant role in biological[1] and pharmaceutical research.[2] Allylic sulfur-containing compounds (such as allylic sulfones and allylic sulfides) represent the outstanding and versatile building blocks among them in organic synthesis.[3,4] For example, allylic sulfones are an excellent precursor for regio- and diastereoselective diene synthesis through the Julia olefination procedure.[5] Moreover, numerous allylic thioethers have been found to be bioactive[6] and crucial reaction intermediates.[7] Therefore, more and more attention has been paid to develop an efficient approach for constructing allylic C–S bonds in recent years.[8,9] The facile synthesis of allylic sulfones has been well developed, which can be divided into two major methods: (i) sulfonyl radical addition[10] and (ii) nucleophilic addition.[11] However, the preparation of highly valuable functionalized allylic sulfides remains a highly desirable goal and long-term challenge.

The general strategies for the preparation of allylic sulfides are transition-metal allylation of sulfur nucleophiles, especially under palladium- or iridium-catalyzed nucleophilic addition to metal π-allyl intermediates.[12] These transformations feature overall broad reaction scope and good functional group tolerance. In 2010, Zhao and co-workers reported the application of sodium thiophenoxide instead of thiophenol as a sulfur nucleophile which avoided the deactivation of metal catalysis due to the strong coordination of sulfur containing compounds.[13] Under iridium catalytic conditions, allyl sulfides were generated in modest to good yields with excellent enantioselectivities. However, the major drawback of this process was the pre-functionalization of the reactants. As part of our ongoing studies towards the development of new methods for the oxidative functionalization of allylic sp^3 C–H bonds,[14] we herein present an atom- and step-economical strategy for the synthesis of allylic sulfides *via* oxidative dehydrogenation of olefins and thiophenol derivatives. Notably, this reaction not only delivered allylic sulfides in moderate to high yields but also performed under metal-free conditions, highlighting the efficiency and practicality of this protocol.

Initially, the reaction between α-methylstyrene (**1a**) and 4-methylbenzenethiol (**2a**) was tested under various conditions (Table 1). When we utilized our previously reported reaction conditions,[14g] no desired product was detected. Next, we aimed at investigating the effect of solvents for the allylic oxidative dehydrocoupling reaction, as shown in Table 1. The screening of different solvents indicated that the solvent played a vital role in this transformation (Table 1, entries 1–5). Pleasingly, a trace amount of **3a** was generated when DMA was adopted (Table 1, entry 4). Considering the important role of the oxidant in this dehydrocoupling process, other oxidants were surveyed. To our delight, the addition of mixed oxidants (DDQ : BQ = 4 : 1) increased the yield of **3a** distinctly (Table 1, entry 4). Therefore, different mixed oxidants were then tested in this reaction. The mixture of DDQ and NQ (1 : 1, 2 equiv.) provided the best yield of **3a** (Table 1, entries 8–12). Surprisingly, this oxidative dehydrocoupling reaction still proceeded smoothly without palladium catalysis (Table 1, entry 13). Controlled experiments revealed that DDQ dominated this reaction (Table 1, entries 14 and 15). Thus, the optimal conditions were determined as DDQ (2.0 equiv.) and NQ (2.0 equiv.) in anhydrous DMA at 100 °C with stirring for 24 h.

With the optimized conditions in hand, we set out to investigate the reaction scope of various thiophenols **2** in this transformation. As shown in Table 2, various thiophenols bearing the electron-withdrawing or electron-donating groups on the

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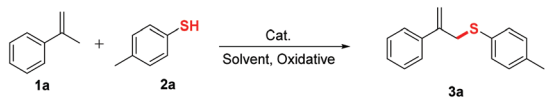
†Electronic supplementary information (ESI) available: Experimental section, characterization of all compounds, and copies of ^{1}H and ^{13}C NMR spectra for all isolated compounds. See DOI: 10.1039/c8qo00799c

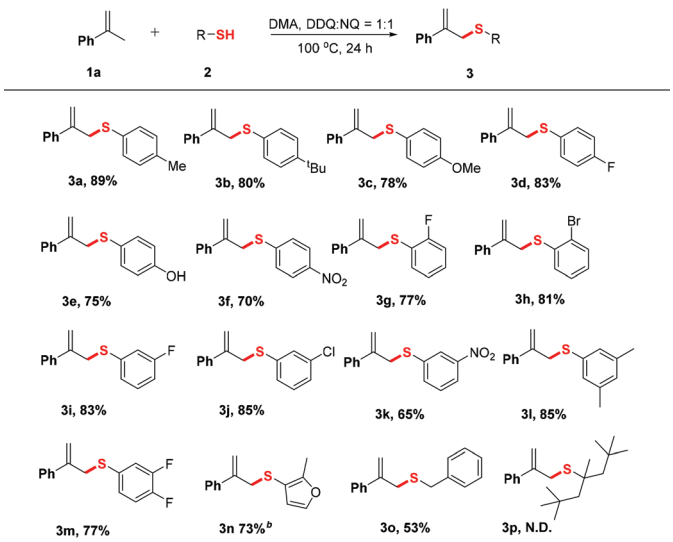
Table 1 Optimization of reaction conditions for allylic sulfides[a]



Entry	Catalyst	Oxidant	Solvent	Yield[b] (%)
1	$Pd(OAc)_2$	NQ	1,4-Dioxane	N.D.
2	$Pd(OAc)_2$	NQ	Toluene	N.D.
3	$Pd(OAc)_2$	NQ	DMF	N.D.
4	$Pd(OAc)_2$	NQ	DMA	Trace
5	$Pd(OAc)_2$	NQ	DMSO	N.D.
6	$Pd(OAc)_2$	BQ	DMA	N.D.
7	$Pd(OAc)_2$	DDQ	DMA	23
8	$Pd(OAc)_2$	DDQ : BQ = (4 : 1)	DMA	52
9	$Pd(OAc)_2$	DDQ : NQ = (4 : 1)	DMA	72
10	$Pd(OAc)_2$	DDQ : NQ = (3 : 1)	DMA	75
11	$Pd(OAc)_2$	DDQ : NQ = (2 : 1)	DMA	80
12	$Pd(OAc)_2$	DDQ : NQ = (1 : 1)	DMA	83
13	—	DDQ : NQ = (1 : 1)	DMA	89
14	—	DDQ : NQ = (0 : 1)	DMA	N.D.
15	—	DDQ : NQ = (1 : 0)	DMA	30

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.1 mmol), oxidant (2 equiv.) in 2 mL of anhydrous solvents at 100 °C for 24 h. NQ: 1,4-naphthoquinone; DDQ: 2,3-dichloro-5,6-dicyano-1,4-benzoquinone; BQ: 1,4-benzoquinone. [b] Determined by GC using dodecane as the internal standard; N.D. = not detected.

Table 2 Substrate scope of thiophenols[a]



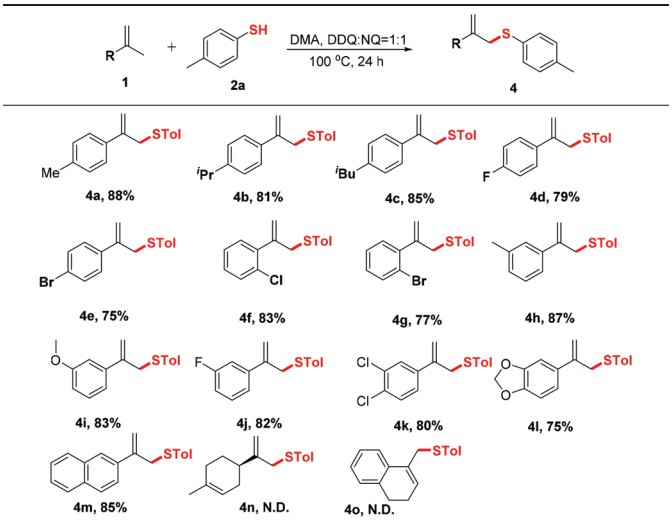
[a] Reaction conditions: α-Methylstyrenes **1a** (0.2 mmol), thiophenols **2** (0.1 mmol), DDQ (2 equiv.), NQ (2 equiv.) in 2 mL anhydrous DMA at 100 °C for 24 h. Yields refer to isolated yields. [b] Bis(2-methyl-3-furyl)disulfide (**2n**) as the substrates.

phenyl ring transformed into the corresponding products smoothly (**3a–3k**). It is noteworthy that this protocol was compatible with a broad range of functional groups such as alkyls (**3a**), halides (F, Cl, and Br; **3d**, **3j**, and **3h**), and hydroxyl (**3e**) and nitro groups (**3k**). These products might be utilized for further synthetic transformations. Moreover, multiple substituents at the phenyl ring did not affect the efficiency (**3l** and **3m**) of this process. Notably, phenylmethanethiol (**2o**) was also tolerated in this reaction, leading to **3o** in a moderate yield. Unfortunately, 2,2,4,6,6-pentamethylheptane-4-thiol (**2p**) was not suitable for this transformation.

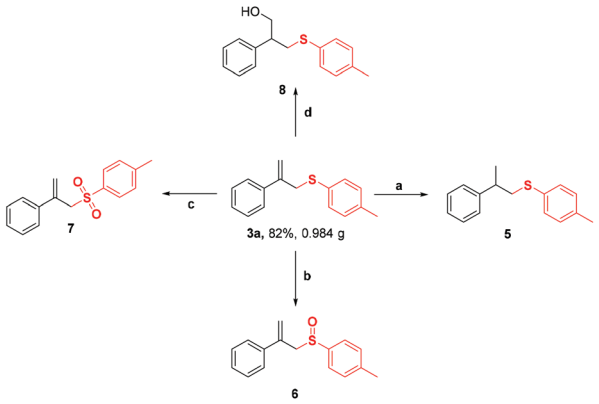
Inspired by the success of thiophenol derivatives, we next explored the generality of a variety of α-methylstyrenes. The representative results are summarized in Table 3. Generally, good yields of the desired products were obtained for additions to a variety of α-methylstyrenes with alkyl-substituents (**4a–4c**). Besides, α-methylstyrenes with halide groups (F, Cl, and Br) were converted into the corresponding products in moderate to good yields (**4d–4g**). Additionally, the multiple-substituted α-methylstyrene (**1k** and **1l**) smoothly transformed into the desired products in good yields. Moreover, the reactions of 2-allylnaphthalene (**1m**) proceeded efficiently under the optimal reaction conditions to deliver product **4m** in 85% yield. Disappointedly, the substrates (*S*)-(−)-4-iso-propenyl-1-methyl-1-cyclohexene (**1n**) and 1-methylene-1,2,3,4-tetrahydronaphthalene (**1o**) could not transform into the corresponding product **4n** or **4o**.

In view of the practicability of this methodology, the reaction between **1a** and **2a** was performed at the 5 mmol scale. The isolated yield of **3a** was 82% (0.984 g, Scheme 1). Next, **3a** was quantitatively converted into the hydrogenated product **5**. Moreover, allylic sulfoxide is a useful intermediate in the preparation of highly functional motifs including natural products (*e.g.*, (−)-agelastin A[15]). The direct oxidation of **3a** to the corresponding sulfoxides was achieved efficiently under mild conditions. Additionally, the transformation of **3a** into allylic sulfone **7** also proceeded smoothly with high chemoselectivity. Notably, with the careful control of the reaction conditions, the hydrolysis of the olefinic moiety was also realized to generate the desired product **8** in a good yield.

Table 3 Substrate scope for α-methylstyrenes[a]

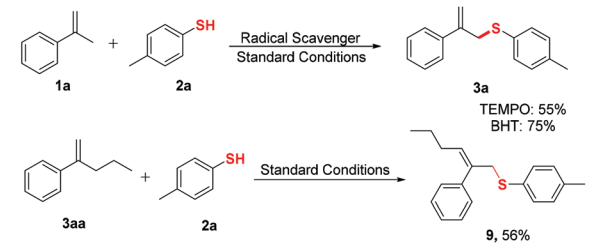


[a] Reaction conditions: α-Methylstyrenes **1** (0.2 mmol), 4-methylbenzenethiol **2a** (0.1 mmol), DDQ (2 equiv.), NQ (2 equiv.) in 2 mL DMA at 100 °C for 24 h. Yields refer to isolated yields.

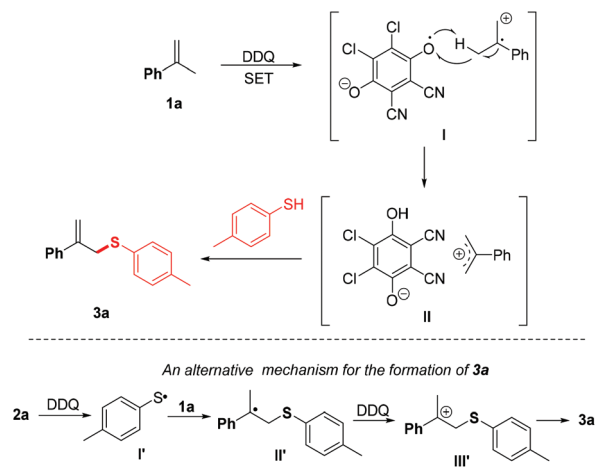


Scheme 1 Further transformation of the product **3a**.

To gain more insight into the mechanism of this reaction, several control experiments were conducted as illustrated in Scheme 2. When 2.0 equiv. of TEMPO or butylated hydroxytoluene (BHT) were added in the reaction mixture, the yields of the corresponding product **3a** were decreased to 55% and 75% respectively, which suggested that a radical process may be involved in this reaction. Moreover, when **3aa** was examined under the optimal conditions, 56% yield of **9** was obtained. Based on the previous reports[16] and our observations, a possible mechanism was proposed (Scheme 3). First, a single-electron transfer (SET) oxidation of **1a** with DDQ generates the radical ion pair intermediates **I**. Next, the ion pair **II** is generated *via* hydrogen atom transfer (HAT). Finally, the nucleophile **2a** attacks the allylic cation to give the dehydrocoupling product **3a**. Another possible pathway involves an initial arylthiol oxidation by DDQ to give thioyl radical **I′**, followed by radical addition to styrene to afford benzylic radical **II′**. In this way, the benzylic radical can be further oxidized to give carbon cation **III′** and lose a beta-proton to give product **3a**.[17]



Scheme 2 Control experiments.



Scheme 3 Proposed mechanism.

In summary, we have successfully developed a novel metal-free oxidative dehydrocoupling of terminal olefins with thiophenol derivatives. Furthermore, this reaction realized the construction of C–S bonds with high regioselectivity. This protocol features high atom- and step-economy and good functional group tolerance, providing an efficient route for the synthesis of organo-sulfur compounds.

Conflicts of interest

There are no conflicts to declare.

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