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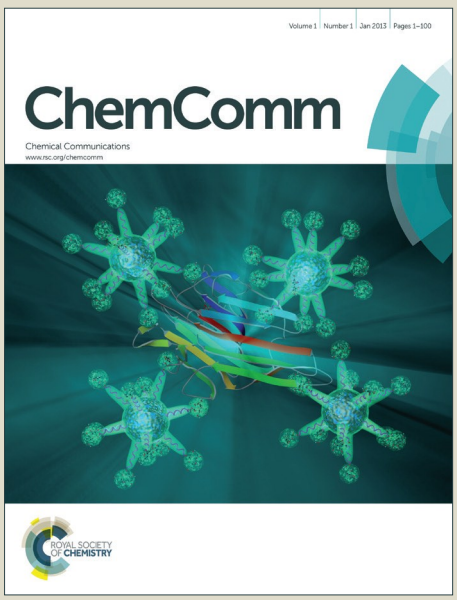

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Direct synthesis of sulfonated dihydroisoquinolinones from *N*-allylbenzamide and arylsulfinic acids *via* TBHP-promoted cascade radical addition and cyclization

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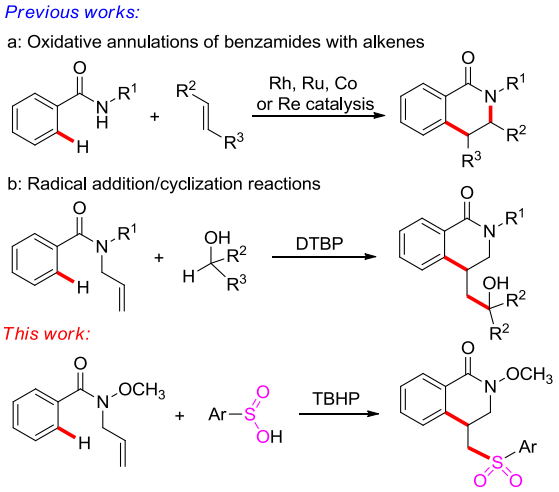
A novel synthesis of sulfonated dihydroisoquinolinones *via* cascade radical addition and cyclization was developed in presence of *tert*-butyl hydroperoxide (TBHP). The reactions generated the desired sulfonated dihydroisoquinolinones in good yields from readily available arylsulfinic acids and *N*-allylbenzamides under metal-free conditions.

Dihydroisoquinolone derivatives, as one of the important class heterocycles have been widely found in complex natural products and drug molecules, which display diverse biological properties such as anti-tumor, anti-inflammatory, anti-allergic and estrogenic behaviour.[1] Due to their fascinating biological and pharmacological activities, the synthesis of dihydroisoquinolone derivatives have received much attention. Traditional synthetic approach to dihydroisoquinolone is based on the condensation of homophthalic anhydride with imines in the presence of acid or base catalyst or under thermal conditions.[2] Recently, transition metal catalyzed constructions of dihydroisoquinolone frameworks have been demonstrated to be the powerful and efficient routes.[3] Especially, Guimond,[3a] Glorius,[3b] Daugulis,[3c] Wang,[3d] and others[3e-g] have elegantly developed the synthesis of dihydroisoquinolones *via* Rh-, Ru-, Co- or Re-catalyzed oxidative annulations of benzamides and alkenes on the basis of C–H activation and functionlization strategy (Scheme 1a). Furthermore, the reactions of *N*-allylbenzamides could provide promising access to substituted dihydroisoquinolones through a free radical process.[4] For example, Han and Pan reported a facile DTBP-promoted radical cyclization of *N*-allylbenzamide with alcohols for synthesis of 4-hydroxyalkyl-substituted 3,4-dihydroisoquinolin-1(2*H*)-one (Scheme 1b).[4a] Meanwhile, The sulfone is a key organic structural motif in a variety of biologically active



Scheme 1. Synthetic strategies for dihydroisoquinolones.

compounds, pharmaceuticals and synthetic intermediates, especially for aryl alkyl sulfones (Figure 1).[5] For example, **I** can be used as antiproliferative activity against cancer cell,[5b] and **II** is an important intermediate for synthesis of isoquinoline-derived target molecules.[5c] In addition, the sulfonyl group is a strong electron-withdrawing group, and can facilitate the deprotonation of a neighboring carbon atom to generate α-carbanion with a base, the corresponding carbanion can be used for various transformations.[6] So, development of new and efficient methodologies for the incorporation of a sulfonyl group into dihydroisoquinolinones from simple, readily available starting materials remains highly desirable in organic chemistry.

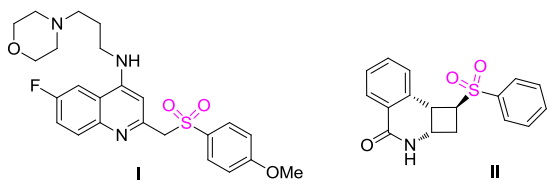


Figure 1. Selected examples of important aryl alkyl sulfones.

Arylsulfinic acids (salts) are relatively stable and versatile intermediates in organic synthesis.[7] In the past decades, considerable efforts have been devoted to the synthesis of

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organic sulfones using arylsulfinic acids (salts) as sulfonylation reagents. As a result, remarkable achievements in cross-coupling of aromatic sulfinic acids (salts) with arylhalides, arylborons and allylic amines have been accomplished by using Pd, Cu and Fe catalysts, and arylsulfinic salts as the precursor of sulfonyl anion reacted with metal catalysts to form a key sulfonyl-metal intermediates.[8] However, the use of transition metals limits their applicability. Most recently, addition of sulfonyl radical to carbon-carbon multiple bonds provided an important alternative for the synthesis of functionalized sulfones under metal-free conditions.[9] For example, Lei and co-workers described oxysulfonylation of alkenes or alkynes employing arylsulfinic acids as the sulfonyl radical precursor using molecular oxygen as oxidant.[9a,b] Subsequently, our group developed visible-light photocatalytic difunctionalization of arylacrylamides and arylpropiolates with arylsulfinic acids to offer sulfone-containing oxindoles and coumarins through a tandem C–S/C–C bond formation.[9e,f] With our continuing interest in application of arylsulfinic acids as sulfonylation agents, herein, we wish to disclose a novel and efficient arylsulfonylation of *N*-allylbenzamides with arylsulfinic acids using TBHP as an oxidant under metal-free condition, which undergoes smoothly through a sequence radical addition and intramolecular cyclization to sulfonated dihydroisoquinolones in high atom-economy and excellent functional group tolerance (Scheme 1).

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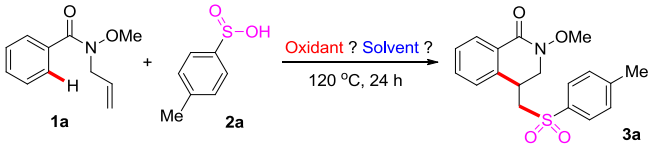
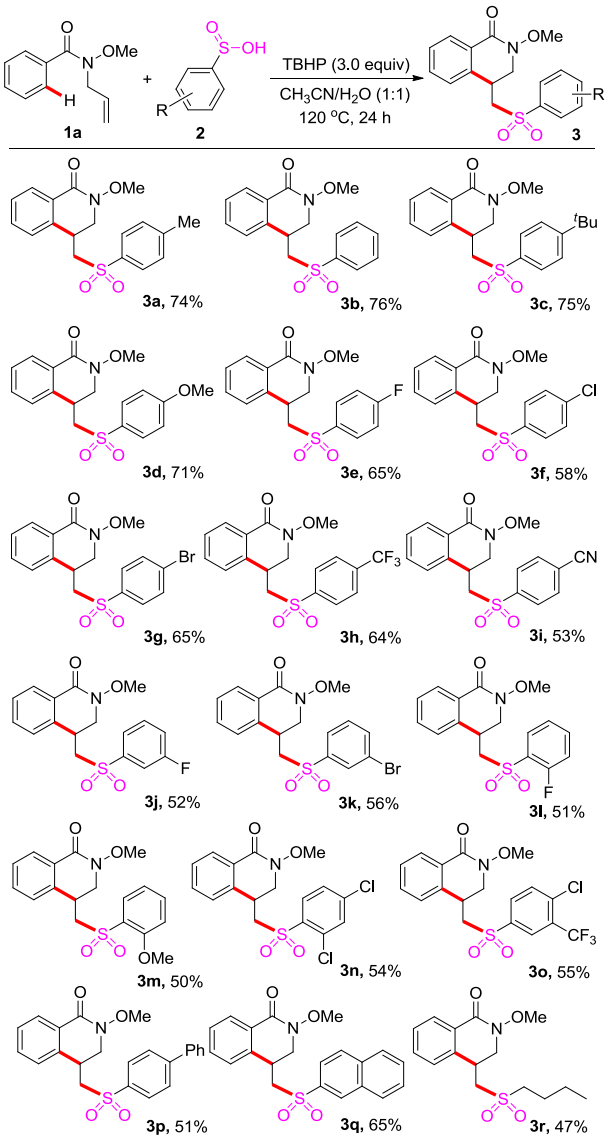
In our initial study, *N*-allyl-*N*-methyloxybenzamide (**1a**) and *p*-tolylsulfinic acid (**2a**) were chosen as the model substrates to optimize the reaction conditions with different solvent and oxidant. When CH_3CN was used as a solvent with TBHP as an oxidant, the model reaction of **1a** with **2a** generated the corresponding sulfonated dihydroisoquinolinone **3a** in 45% yield (Table 1, entry 1). However, other solvents including DMSO (dimethyl sulphoxide), 1,4-dioxane, DMF (*N*,*N*-dimethylformamide), THF (tetrahydrofuran), methanol, toluene, DCE (1,2-dichloroethane) and H_2O were inferior and afforded desired **3a** in 10–42% yields (Table 1, entries 2–9). Much to our pleasure, 74% yield of **3a** was achieved when a mixed solvent of acetonitrile and H_2O in 1:1 (V/V) was used as reaction medium

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



Entry	Oxidant	Solvent	Yield (%) [b]
1	TBHP	CH_3CN	45
2	TBHP	DMSO	22
3	TBHP	DMF	31
4	TBHP	1,4-dioxane	27
5	TBHP	THF	21
6	TBHP	CH_3OH	10
7	TBHP	toluene	15
8	TBHP	DCE	28
9	TBHP	H_2O	42
10	**TBHP**	**H_2O/CH_3CN (1:1)**	**74**
11	DCP	H_2O/CH_3CN (1:1)	61
12	TBPB	H_2O/CH_3CN (1:1)	51
13	DTBP	H_2O/CH_3CN (1:1)	40
14	CHP	H_2O/CH_3CN (1:1)	28
15	H_2O_2	H_2O/CH_3CN (1:1)	27
16	O_2	H_2O/CH_3CN (1:1)	36
17	$K_2S_2O_8$	H_2O/CH_3CN (1:1)	38
18	$Na_2S_2O_8$	H_2O/CH_3CN (1:1)	34
19	TBHP	H_2O/CH_3CN (1:1)	50 [c]
20	TBHP	H_2O/CH_3CN (1:1)	52 [d]

[a] Reaction conditions: **1a** (0.25 mmol), **2a** (0.75 mmol), oxidant (3.0 equiv), solvent (3.0 mL), at 120 °C for 24 h. TBHP = *tert*-butyl hydroperoxide (70% solution in water); DCP = dicumyl peroxide; TBPB = *tert*-butyl peroxybenzoate; DTBP = di-*tert*-butyl peroxide; CHP = cumene hydroperoxide; H_2O_2 (30% aqueous solution). [b] Isolated yield based on **1a**. [c] TBHP (2.0 equiv) was used, [d] **2a** (2.0 equiv) was used.



Scheme 2. Reaction of **1a** with arylsulfinic acids [*Reaction conditions*: **1a** (0.25 mmol), **2** (0.75 mmol), TBHP (3.0 equiv), CH_3CN/H_2O (1:1, 3.0 mL), 120 °C, 24 h; isolated yield of the product based on **1a**.]

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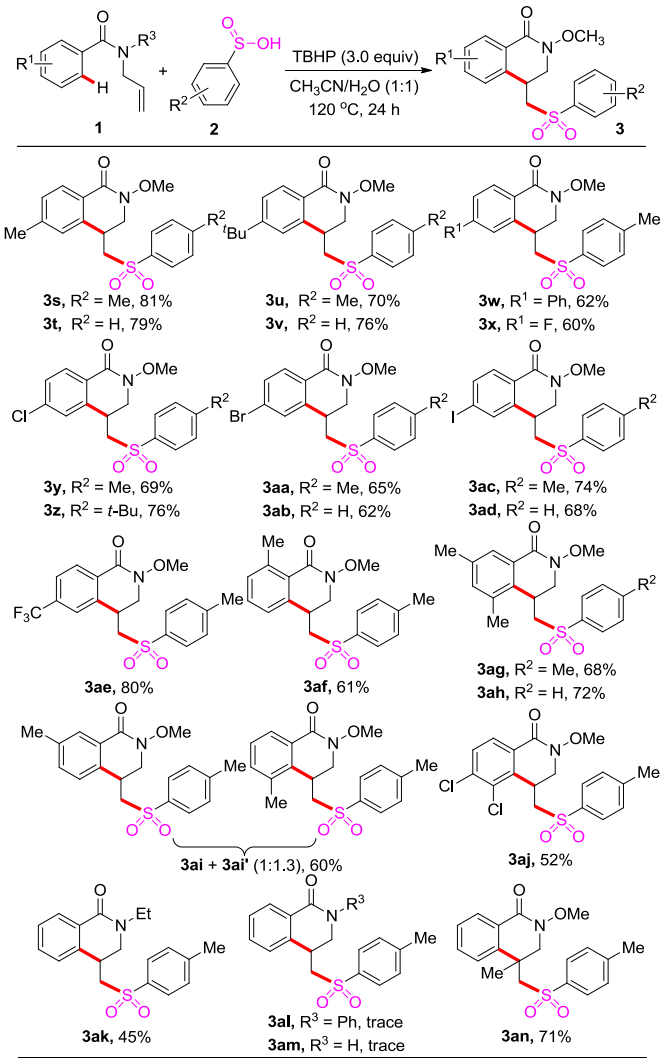
(Table 1, entry 10). Next, the effect of the oxidant on the model reaction was examined. When DCP (dicumyl peroxide) was instead of TBHP, the model reaction generated the desired product **3a** in 61% yield (Table 1, entry 11). Other organic oxidants, such as TBPB, DTBP, CHP, H_2O_2, $K_2S_2O_8$, $Na_2S_2O_8$ and O_2 led to **3a** in 27–51% yields (Table 1, entries 12−18). Reducing amount of TBHP or **2a** from 3.0 equiv to 2.0 equiv, the model reaction produced **3a** in 50% and 52% yields, respectively (Table 1, entries 19 and 20).

With the optimized conditions in hand, we explored the synthetic viability of the reactions using *N*-allyl-*N*-methyloxybenzamide (**1a**) with a variety of arylsulfinic acids, and the results are summarized in Scheme 2. In general, a number of arylsulfinic acids containing both electron-donating and electron-withdrawing groups on the aromatic rings underwent the radical addition and cyclization to provide the corresponding arylsulfonylation products (**3a**–**3q**) in 50−76% yields. In particular, the sterically-hindered substituted arylsulfinic acid, such as *ortho*-fluoro- and *ortho*-methoxybenzenesulfinic acids were suitable substrates in the transformation, leading to the desired products **3l** and **3m** in 51% and 50% yield, respectively. Notably, arylsulfinic acid bearing a trifluoromethyl (CF_3) group, which is a

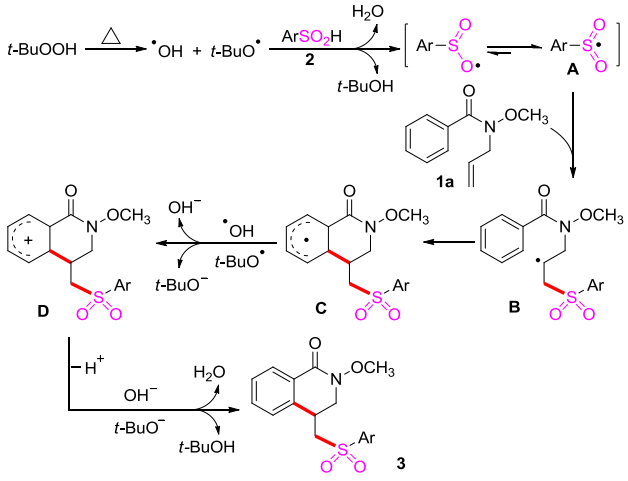


Scheme 3. Reaction of **1** with arylsulfinic acids [*Reaction conditions:* **1** (0.25 mmol), **2** (0.75 mmol), TBHP (3.0 equiv), CH_3CN/H_2O (1:1, 3.0 mL), 120 °C, 24 h; isolated yield based on **1**.]

useful structural motif in biologically active molecules[10] reacted with **1a** to generate the corresponding product **3h** in 64% yield. In the case of disubstituted arylsulfinic acids, the reaction underwent smoothly, affording products **3n** and **3o** in moderate yields. Furthermore, more bulky 1,1'-biphenyl- and 2-naphthyl-sulfinic acids could participate in the reaction well, furnishing the dihydroisoquinolinones **3p** and **3q** in accepted yields. It should be noted that aliphatic sulfinic acid such as *n*-butylsulfinic acid could also be used in the reaction to provide the desired product **3r** in 47% yield.

After studying the scope of arylsulfinic acids, we turned our attention toward *N*-allylbenzamide derivatives. As shown in Scheme 3, *N*-allylbenzamides have a broad scope and high compatibility with functional groups, such as methyl, *tert*-butyl, phenyl, trifluoromethyl and halogen groups, and gave the corresponding products (**3s**–**3ak**) in moderate to good yields. It is important to note that this domino reaction is compatible with I, Br and Cl at *para*-position of the aromatic rings, which can undergo the further transformation for the synthesis of more complex molecules. Meanwhile, substrate **1** with 3,5-dimethyl groups on phenyl ring could undergo the tandem reaction to afford the **3ag** and **3ah** in good yields. To investigate the regioselectivity of this cyclization, the reaction of **1** with *meta*-substituted group was conducted. As expected, *meta*-substituted substrate **1** gave a mixture of two regioselective products **3ai** and **3ai'** in a ratio of 1:1.3 with total 60% yield. An obviously regioselectivity was obtained when 3,4-dichloro substituted **1** reacted with **2a**, providing **3aj** as sole product in 52% yield. The structure of **3aj** was further confirmed by single-crystal X-ray diffraction analysis (Supporting Information for detail). Furthermore, *N*-ethyl protected *N*-allylbenzamide was also examined, providing product **3ak** in 45% yield. However, the reaction of *N*-phenyl and *N*-H,*N*-allylbenzamides failed to react with *p*-tolylsulfinic acid (**2a**) under the standard reaction conditions (**3al, 3am**). Finally, substrate with substituted double bond *N*-*β*-methyl-allyl-*N*-methyloxybenzamide was found to be suitable for this protocol, and reacted with *p*-tolylsulfinic acid (**2a**) to generate target product **3an** in 71%.

For understanding the mechanism of tandem reaction, the well known radical-trapping reagents, such as TEMPO (2,2,6,6-tetramethylpiperidine-*N*-oxyl) and BHT (2,6-di-*tert*-butyl-4-methyl-phenol) was added in 2.0 equiv to the model reaction respectively,



Scheme 4. Possible mechanism.

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and the reaction was completely inhibited, suggesting this transformation involved a radical process. Based on our observations and the reported investigations, a plausible mechanism is proposed in Scheme 4. Firstly, homolysis of TBHP produced hydroxyl and *tert*-butoxyl radicals, which could abstract hydrogen from arylsulfinic acids to afford an oxygen centered radical resonating with arylsulfonyl radical (**A**). Next, the radical intermediate **A** was added to the double bond of activated alkene (**1a**) to afford the alkyl radical intermediate (**B**), followed by an intramolecular radical cyclization of **B** to form radical intermediate (**C**). Subsequently, radical intermediate (**C**) was oxidized by hydroxyl radical or *tert*-butoxyl radical to generate a cationic intermediate (**D**) and a hydroxide or *tert*-butoxide through a SET process. Finally, a cationic intermediate (**D**) loosed a proton to produce the final sulfonated isoquinolinone **3** and water or *tert*-butanol.

In summary, we have developed a TBHP-promoted arylsulfonylation of *N*-allylbenzamide with readily available arylsulfinic acids *via* radical addition and intramolecular cyclization. A variety of substrates were employed in the reaction and the corresponding sulfonated isoquinolinones were obtained in moderate to good yields under metal-free conditions. This tandem radical reaction provides a simple and environmental friendly access to isoquinolinone derivatives from *N*-allylbenzamide. Further studies towards understanding the mechanistic detail and synthetic application of this kind transformation in the synthesis of other sulfur-containing compounds are currently underway.

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