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Multicomponent Assembly of α,α-Bis-Sulfonyl Arylketones and Multiple Substituted Conjugated Dienes Induced by Visible-Light Irradiation without Additives and Photocatalysts

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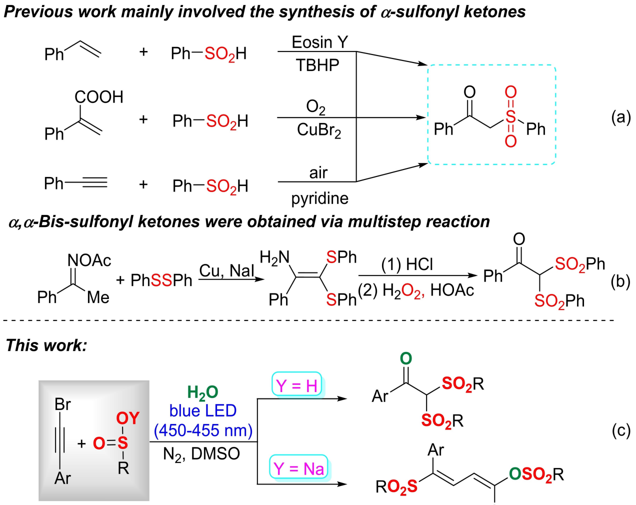
Abstract: A synthetic method is described to produce α,α-bis-sulfonyl arylketones from bromoalkynes and sulfinic acids under visible-light irradiation, this visible-light-promoted multicomponent cascade processes are used without additives or photocatalysts. Replacing sulfinic acids with sodium arylsulfinates results in another multicomponent reaction for synthesizing multisubstituted conjugated dienes with 26%–54% yields via construction of a C–C bond and without the use of transition metals and other additives.

Keywords: Visible-light; bromoalkynes; multicomponent reactions; α,α-bis-sulfonyl arylketones; conjugated dienes

One-pot multicomponent reactions are environmentally friendly synthetic schemes that can be used to prepare complicated organic compounds from simple, commercially available or easily prepared synthetic precursors.[1] However, most of these efficient and atomic economic transformations require catalysts or additives.[2] Therefore, the development of a simple and convenient multicomponent synthetic strategy remains an important challenge to chemists, particularly for obtaining products without using mature synthetic methods. Introduction of sulfone units into organic molecules is an important goal in organic chemistry because of the widespread synthetic applications of sulfones.[3] Among different sulfone derivatives, sulfone-substituted ketones have been extensively studied by synthetic chemists, because these compounds are widely applied in biological and pharmaceutical science.[4] Most pioneering studies have mainly pursued preparing α-sulfonyl ketones from alkenes,[5] alkynes[6] or their derivatives[7] (Scheme 1a). Few studies have developed synthetic schemes to produce bisulfonyl compounds,[8] especially α,α-bis-sulfonyl arylketones, by linking two sulfone groups at one carbon atom (Scheme 1b).[9] Consequently, the development of a direct, green and convenient synthetic platform to produce α,α-bis-sulfonyl arylketones is a challenging problem with considerable significance in the field.

In recent years, significant advantages have become associated with using visible light to promote chemical transformations, such as easy availability, matching green chemistry concepts with sustainable energy sources, as an alternative to conventional synthetic methods. Among visible-light-promoted reactions, visible-light photoredox catalysis has been the most actively investigated, and a variety of photocatalysts have been used in these reaction systems.[10] Chemists have also performed in-depth research on photocatalyst-free photoreactions because of unique characteristics and convenient operability; however, additives are required to carry out these phototransformations efficiently.[11] Recently, our group reported a photoinduced oxidative amidation of bromoalkynes with anilines and a controllable chemoselectivity in coupling bromoalkynes with alcohols, in which the corresponding products of α-ketoamides, propargyl alcohols and α-ketoesters were generated without use of additional reagents (e.g. catalysts, oxidants and other additives).[12] This reaction mode appears to be more effective and practical than many reported photoredox reactions. Furthermore, we discovered other novel visible-light-initiated transformations of bromoalkynes using sulfinic acids as substrates. Unex-

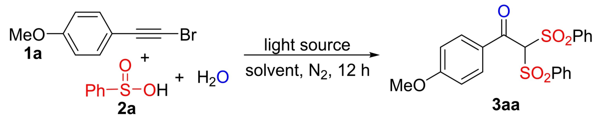


Scheme 1. Various sulfonyl reactions.

pected α,α-bis-sulfonyl arylketones were smoothly generated by carrying out the reaction in DMSO with the addition of a few amount of water under blue LED irradiation in the presence of N_2, whereas the chemoselectivity of the photoreaction with sodium arylsulfinates as substrates resulted in a novel multicomponent reaction that generated sulfonyl-substituted conjugated dienes (Scheme 1c). Herein, we report this adjustable and controllable synthetic strategy for the synthesis of α,α-bis-sulfonyl arylketones and sulfonyl-substituted conjugated dienes from bromoalkynes with sulfinic acids or sodium arylsulfinates under visible-light irradiation without photocatalysts or other additives.

We initially selected 1-(bromoethynyl)-4-methoxybenzene (**1 a**) and benzenesulfonic acid (**2 a**) as model substrates to assess the feasibility of the reaction under visible-light irradiation. As shown in Table 1, a trace amount of the target product of α,α-bis-sulfonyl arylketone **3 aa** was observed in dry DMSO with a 450–455 nm blue LED light source (Table 1, entry 1). Satisfyingly, the yield of **3 aa** was considerably increased to 53% by adding 0.1 mL of H_2O to the reaction mixture (Table 1, entry 2). As the experimental results showed that the reaction efficiency depended on the loading of water in DMSO, water was added to improve the yield (Table 1, entries 3–5). An acceptable yield of 78% was obtained upon addition of 0.4 mL of H_2O (Table 1, entry 4). Decreasing the quantity of benzenesulfonic acid (**2 a**) further to 0.4 mmol resulted in sharp decrease in the yield (Table 1, entry 6). However, no further improvement in the yield was observed upon increasing the quantity of benzenesulfonic acid (**2 a**) (Table 1, entry 7). Unfortunately, Lower yields of the desired product **3 aa** was obtained when the reaction was irradiated using other visible-light sources, such as blue LED (420–425 nm), purple, green, yellow or red LED (Table 1, entries 8–12). Extensive screening showed negative effects were produced by using other solvents, such as H_2O, DMF, EtOH, THF, toluene, EtOAc, DCE ($ClCH_2CH_2Cl$), and acetone (Table 1, entries 13–20).

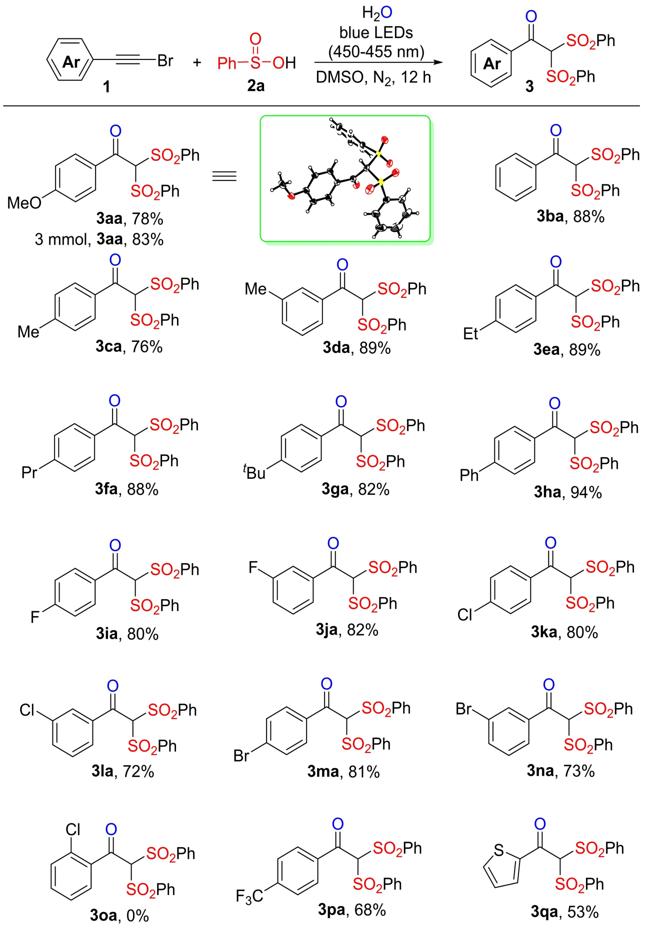
Table 1. Optimization of the reaction conditions.[a]



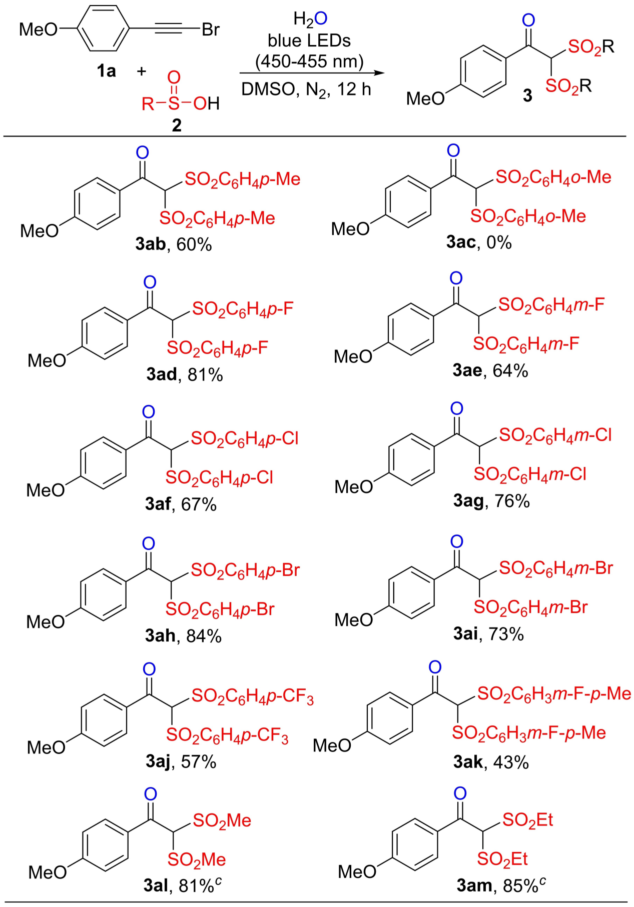
Entry	Light source	Solvent	Yield (%)[b]
1	blue LED (450–455 nm)	dry DMSO	< 5
2	blue LED (450–455 nm)	DMSO	53[c]
3	blue LED (450–455 nm)	DMSO	68[d]
4	blue LED (450–455 nm)	DMSO	78
5	blue LED (450–455 nm)	DMSO	80[e]
6	blue LED (450–455 nm)	DMSO	49[f]
7	blue LED (450–455 nm)	DMSO	80[g]
8	blue LED (420–425 nm)	DMSO	45
9	purple LED (380–385 nm)	DMSO	ND[h]
10	green LED (480–570 nm)	DMSO	< 5
11	yellow LED (570–610 nm)	DMSO	ND
12	red LED (610–650 nm)	DMSO	ND
13	blue LED (450–455 nm)	H_2O	ND
14	blue LED (450–455 nm)	DMF	ND
15	blue LED (450–455 nm)	EtOH	ND
16	blue LED (450–455 nm)	THF	ND
17	blue LED (450–455 nm)	toluene	< 5
18	blue LED (450–455 nm)	EtOAc	ND
19	blue LED (450–455 nm)	DCE	< 5
20	blue LED (450–455 nm)	acetone	ND

[a] Reaction conditions: **1 a** (0.2 mmol), **2 a** (0.6 mmol), H_2O (0.4 mL), light source, solvent (2.0 mL), rt, N_2 atmosphere, 12 h.
[b] Isolated yield.
[c] 0.1 mL of H_2O added.
[d] 0.2 mL of H_2O added.
[e] 0.6 mL of H_2O added.
[f] 0.4 mmol of **2 a** was added.
[g] 0.8 mmol of **2 a** was added.
[h] No desired product was detected.

After optimizing the reaction conditions, we investigated the scope of bromoalkynes (**1**), as detailed in Scheme 2. Generally, aromatic bromoalkynes with electron-rich groups, such as *p*-Me, *m*-Me, *p*-Et, *p*-Pr, and *p*-^{t}Bu, successfully reacted with **2 a** to generate 76%–89% yields of the corresponding products (**3 ba**–**3 ga**). Subjecting the phenyl substituent at the *para*-position on the benzene ring of bromoalkynes resulted in an excellent yield of 94% (**3 ha**). Bromoalkynes containing halogen-substitutent at the *para*- or *meta*-position were well tolerated, generating 72%–82% yields of the products (**3 ia**–**3 na**). However, substrates bearing chloride atom at the *ortho*-position could not react with benzenesulfonic acid (**2 a**) to generate a



Scheme 2. The scope of bromoalkynes. a) Reaction conditions: **1** (0.2 mmol), **2a** (0.6 mmol), H_2O (0.4 mL), blue LED (450–455 nm), DMSO (2.0 mL), rt, N_2 atmosphere, 12 h. b) Isolated yield.

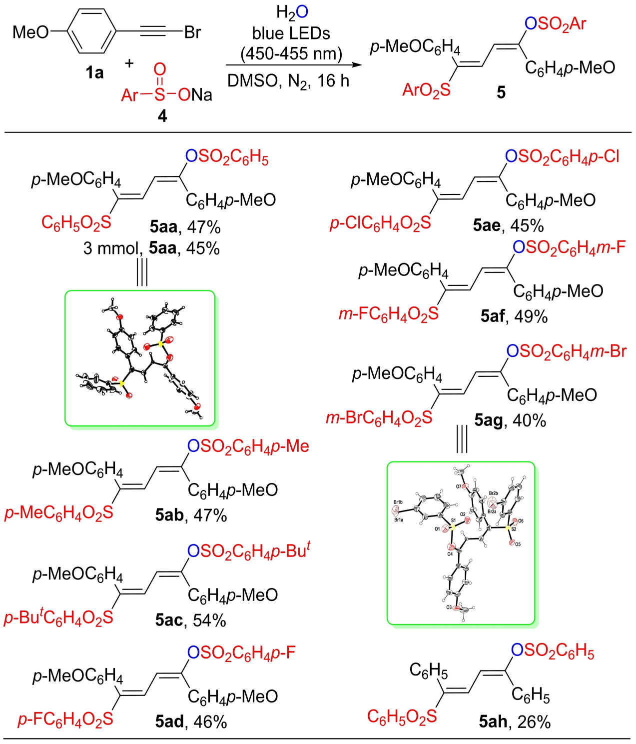


Scheme 3. The scope of sulfinic acids. a) Reaction conditions: **1a** (0.2 mmol), **2** (0.6 mmol), H_2O (0.4 mL), blue LED (450–455 nm), DMSO (2.0 mL), rt, N_2 atmosphere, 12 h. b) Isolated yield. c) **1a** (0.2 mmol); $MeSO_2Na$ or $EtSO_2Na$ (0.6 mmol), HCl (0.6 mmol), H_2O (0.4 mL), DMSO (2.0 mL), rt, in N_2, 24 h.

product (**3oa**). Electron-withdrawing group (–CF_3) on the phenyl ring or the thiophene substituent on the alkynyl bromide were well tolerated, generating 68% and 53% yields of the desired products (**3pa** and **3qa**), respectively. Conversely, aliphatic bromoalkyne (e.g. 1-bromohex-1-yne) was not suitable for the transformation, and no corresponding product was obtained under optimal conditions. When the model reaction was carried out on a gram-scale (3 mmol), an 83% yield of **3aa** was isolated. The structure of **3aa** was further determined by X-ray analysis.[13]

We further explored the scope of the reaction by investigating the reaction of 1-(bromoethynyl)-4-methoxybenzene (**1a**) with different benzenesulfonic acids (**2**). As shown in Scheme 3, toluenesulfonic acid was used in the reaction, and moderate yield of the desired product **3ab** was obtained. By contrast, no reaction occurred when *o*-toluenesulfonic acid (**2c**) was employed as the substrate. Sulfonic acids with halogen substituents, including *p*-F, *m*-F, *p*-Cl, *m*-Cl, *p*-Br and *m*-Br, on the benzene ring produced 64%–84% yields of **3ad–3ai**. The substrate with a strong electron-withdrawing group (CF_3) on the benzene ring underwent a smooth reaction to generate a 57% yield of the product (**3aj**). Moreover, benzenesulfonic acid containing disubstituted groups (e.g. F and Me) also afforded the corresponding product (**3ak**) in 43% yield. Interestingly, alkyl-substituted sulfonic acids, such as methylsulfonic and ethanesulfonic acid, were well tolerated and generated 81% and 85% yields of the corresponding products **3al** and **3am**, respectively.

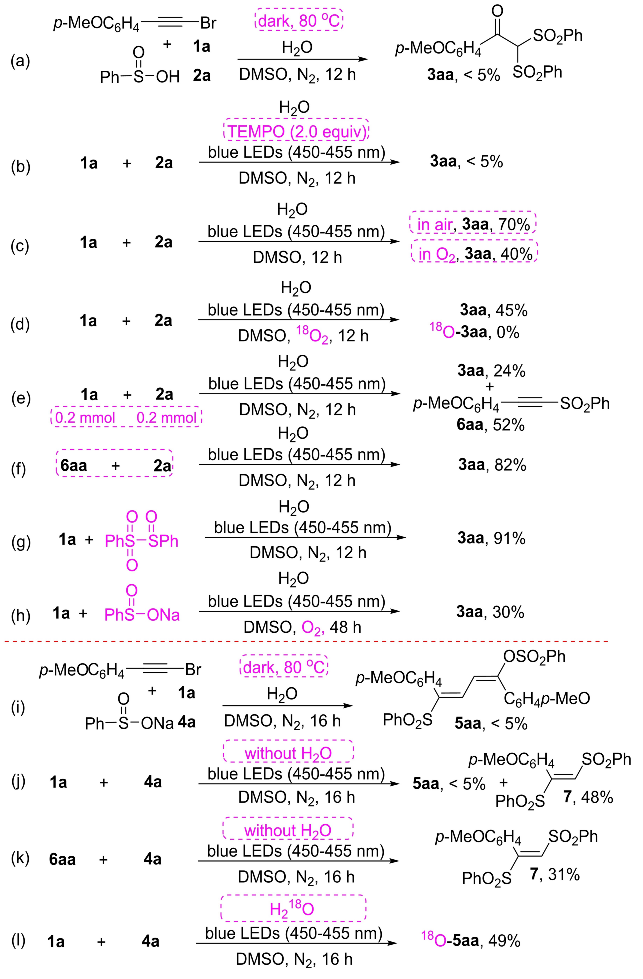
Surprisingly, replacing phenylsulfonic acid with sodium phenylsulfinates in the reaction produced modest yield of multiply substituted conjugated dienes (**5aa**) (see ESI for details on the optimization of this cascade transformation), and increasing the quantities of the starting materials did not decrease the reaction efficiency. Encouraged by these results, we next explored substrate generality. As shown in Scheme 4, reacting sodium *p*-toluenesulfinates and *p*-*tert*-butyl phenylsulfinates with **1a** produced 47% and 54%



Scheme 4. Photo-promoted tandem reaction of bromoalkynes with sodium arylsulfinates [a] Reaction conditions: **1a** (0.2 mmol), **4** (0.4 mmol), H_2O (0.4 mL), blue LED, (450–455 nm), DMSO (2.0 mL), rt, N_2 atmosphere, 16 h. [b] Isolated yield.

yields yields of the desired products (**5ab** and **5ac**), respectively. Sodium arylsulfinates with halogen substituents, such as *p*-F, *p*-Cl, *m*-F and m-Br, also produced in 40%–49% yields of the corresponding products (**5ad**–**5ag**) under optimal conditions. The reaction efficiency decreased when (bromoethynyl) benzene was used as a reactant, generating a 26% yield of the desired product (**5ah**). The configuration of product **5aa** and **5ag** were further confirmed by X-ray diffraction.[13]

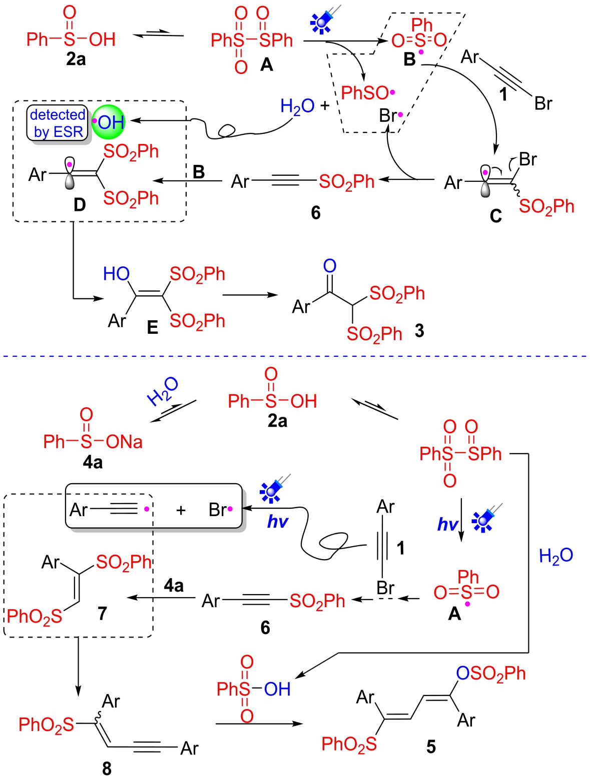
Scheme 5 shows several control experiments that were conducted to elucidate the reaction mechanism. Only a trace amount of **3aa** was detected when the model reaction between **1a** and **2a** was performed in the dark, even at high temperature or in the presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (Schemes 5a and 5b), which implied that this transformation was almost halted without visible-light irradiation and accomplished via a free-radical process. Furthermore, the presence of O_2 decreased the reaction efficiency (Scheme 5c). When the model reaction was stirred in the presence of $^{18}O_2$, but no ^{18}O-**3aa** was obtained after HRMS analysis (Scheme 4d), which further confirmed that the oxygen element in **3aa** is from H_2O (not from O_2). Schemes 5e and 5f confirmed that **3aa** was formed via the intermediate acetylenic sulfone product



Scheme 5. Control experiments.

6aa. Considering the conversion of sulfinic acids to sulfinyl sulfone[14] in conjunction with the results of Schemes 5g and 5h showed that this photo-promoted tandem process was initiated by sulfonyl radicals derived from sulfinyl sulfones in the presence of visible-light irradiation. On the other hand, the possibility that the generation of product **3aa** underwent the cationic process through **6aa** and **2a** was also ruled out by other designed control experiments (see the ESI for details). Performing similar control experiments (Schemes 5i–5l) to investigate another multi-molecular reaction mechanism produced the following results: (1) the substituted conjugated diene product **5aa** cannot be formed without visible-light irradiation (Scheme 5i); (2) assembly of multiple substituted conjugated dienes may require the presence of sulfinyl sulfone, acetylenic sulfone, and bis(arylsulfonyl) ethylene intermediate products (Scheme 5j and 5k);[14] and (3) the O-atom of the sulfonate group in the product **5aa** derives from H_2O (Scheme 5l).

We used our assays and previous studies to propose a possible mechanism, which is shown in Scheme 6.



Scheme 6. Possible mechanism.

Initially, a condensate of sulfinic acid **2a** underwent a reaction to produce sulfinyl sulfones **A**, which could be decomposed to the sulfonyl radical **B**. Then, the intermediate **B** was added to bromoalkyne **1** to produce the intermediate **C**, which was followed by a radical elimination reaction to yield the sulfone product **6** and concomitantly removed the bromide radical (Br•). Next, the sulfone product **6** might have reacted with the surplus sulfonyl radical **B** to form the intermediate **D** via conjugate addition. Hydroxyl radicals (•OH) might have been generated by the reaction of possible radical intermediates (Br•, sulfonyl or sulfoxide radicals) with H_2O, which could be indirectly confirmed by electron spin resonance (ESR) experiments (see the ESI for details).[15] Subsequently, the intermediate **D** coupled with •OH to produce the sulfone substituted enol **E**, which was tautomerized to the desired product **3**. A similar procedure was followed for generating **6** with the sodium arylsulfinates substrates, except for an initial hydrolysis reaction, and 1,4-addition reaction of **6** with extra **4a** to yield another intermediate product **7**.[16] Meanwhile, an alkynyl radical and a Br• were produced by the homolytic cleavage of bromoalkynes **1**, where ESR experiments indirectly proved that this homolytic cleavage process could occur; see the ESI for details. Then, the intermediate **7** might couple with the alkynyl radical to produce conjugated 1,3-enynes **8** via desulfonylation, and this compound might be unstable under this model reaction conditions and was failure to isolated. Finally, sulfonic acid was produced by the hydrolysis of sulfinyl sulfones, and condensed with conjugated 1,3-enynes **8** via electrophilic addition to yield the desired product **5**. However, we still could not give reasonable explain for the real internal reason for transformation of **7** to the single geometrical isomer **5**.

In conclusion, we have developed a convenient and simple synthetic method to synthesize α,α-bis-sulfonyl arylketones via a visible-light-promoted multicomponent reaction of bromoalkynes with sulfinic acids in the absence of photocatalysts and other additives. A more complex multicomponent reaction was observed using sodium arylsulfinates as a substrate, which reacted with bromoalkynes to produce moderate yields of multisubstituted conjugated dienes. In addition, a possible reaction mechanism was given by the results of control experiments.

Experimental Section

General procedure for synthesis of α, α-bis-sulfonyl arylketones: To a solution in 2 mL of DMSO was added sulfinic acids (0.6 mmol), bromoalkynes (0.2 mmol) and H_2O (0.4 mL). The mixture was stirred in nitrogen atmosphere under the blue LED (450–455 nm) irradiation for 12 h. Then the reaction mixture was added brine (6 mL), and extracted with ethyl acetate (3 × 6 mL). The residue was then purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) to give the pure product.

General procedure for synthesis of multiply substituted conjugated dienes: To a solution in 2 mL of DMSO was added sodium sulfinates (0.4 mmol), bromoalkynes (0.2 mmol) and H_2O (0.4 mL). The mixture was stirred in nitrogen atmosphere under the blue LED (450–455 nm) irradiation for 16 h. Then the reaction mixture was added brine (6 mL), and extracted with ethyl acetate (3 × 6 mL). The residue was then purified by column chromatography on silica gel (petroleum ether/EtOAc = 3:1) to give the crude product, which was further recrystallized (petroleum ether/dichloromethane) to give the pure product.

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