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# Synthesis of (*E*)-iodo vinylsulfones via oxidative addition of thiol into alkyne under metal free condition

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ABSTRACT

An efficient, transition-metal free and molecular iodine promoted protocol for the construction of (*E*)-β-iodovinyl sulfone derivatives via oxidative C−S coupling of thiol and alkyne has been demonstrated. Both aryl and alkyl terminal acetylenes were found to be an excellent substrate for the present reaction which provides a wide range of β-iodovinyl sulfone derivatives with very good yield and excellent regio and stereo-selectivities. Diaryldisulfide is also found to be an equally efficient sulfonyl group surrogate under identical reaction conditions.



## 1. Introduction

Direct difunctionlization on adjacent C-atom of an alkyne is considered to be a powerful synthetic tool [1] to introduce two functional group in a single step to access polyfunctionalized alkene derivatives. Among various literature known methods [2] for direct alkyne difunctionalization, halosulfonylation [1b-c] is of particular interest as it directly furnishes functionally rich β-halovinyl sulfone derivatives. Vinyl sulfones and halogenated vinyl sulfones are the compounds of contemporary interest because of their prevalence in biologically functional molecules [3] and their versatility as starting material in organic synthesis [4]. Additionally, sulfone functionality can play multiple role as an activating agent, a good leaving group or as an electron withdrawing group which made this class of compounds more appealing. Due to their above mentioned importance, synthetic chemists showed their long-standing interest towards this class of molecules. As a result, various elegant methods have been developed to access halogenated vinyl sulfone derivatives.

A variety of transition metal catalysts have been employed for direct difunctionalization of alkyne leading to halovinyl sulfone derivatives [5]. Inexpensive iron catalysts were used by Nakamura [5a] and Li [5b] to synthesize (*E*)-β-vinylsulfones from terminal alkyne with complete regio- and stereoselectivity. Copper catalyzed addition of sulfonyl chloride to acetylene offering β-chlorovinyl sulfone was first reported in 1971 by Amiel [5c]. Recently, Liu et al. reported another copper(I)halide catalyzed halosulfonylation of terminal alkyne using sulfonohydrazides as sulfonylating agent to β-halovinyl sulfone derivatives with *Z*-selectivity [5d]. Apart from halosulfonylation of alkyne, various transition metal catalyzed sulfonylation of olefin offering vinyl sulfone are also well established synthetic methods [5e-g]. Despite their synthetic efficiency, very often, metal catalyzed reactions are being considered as environmentally unfavorable due to toxic metal byproducts. Therefore, development of environmentally benign and synthetically more efficient methods for difunctionalization of alkyne is an issue of urgent significance. As a consequence, a sizeable number of metal free halosulfonylation of alkyne have been reported in the recent literature [6]. In this regard, iodine mediated β-halosulfonylation of alkyne via C−S coupling is of great importance. In general, iodine promoted organic transformations constitute a large area in organic research [7].

A diverse range of important heterocyclic compounds such as pyridine [7a], indole [7b], pyrrole [7c], furan [7d], thiophene [7e], thiazole [7f] and many more have been successfully synthesized via iodine mediated reactions. Similarly, many molecular iodine promoted difuctionalization reaction of alkyne offering β-halovinyl sulfone derivatives have been reported recently. Wang et al. [6a]

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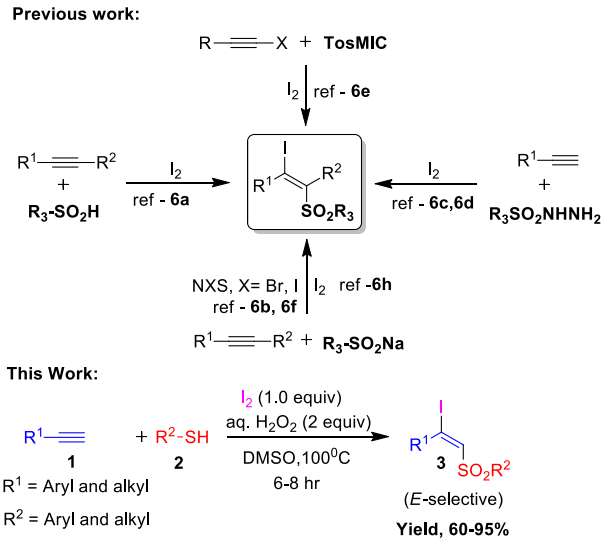



**Scheme 1.** Cascade iodination and sulfonylation into alkyne.

diclosed an efficient approach to access (*E*)-β-iodovinyl sulfones via direct difunctionalization of alkynes with sulfinic acid and iodine (Scheme 1). Liu and coworkers accomplished the same transformation using sodium sulfinate as sulfonating agent and molecular iodine in aqueous medium (Scheme 1) [6b]. Again, sulfonyl hydrazide was used as sulfonating reagent in presence of iodine by Gong et al. [6d] to synthesize the title compound in aqueous medium (Scheme 1). In a different instance, TosMIC was explored as potential sulfonylating agent in association with molecular iodine as catalyst by Yallapragada group (Scheme 1) [6e]. Jiang and coworkers [6f] demonstrated a NBS or NIS promoted sulfonylation of terminal alkyne using sodium sulfinate to access bromo or iodovinyl sulfone derivative with high regio- and stereoselectivity (Scheme 1). Even DMSO-$H_2O$ system in presence of iodine is successfully used as sulfonylating agent furnishing halovinyl sulfone from alkyne [6g]. Though a number of synthetic methods are available to get β-halovinyl sulfone derivatives, development of more efficient, metal free process to access various β-halovinyl sulfones from commercially cheap materials is still highly desirable. Owing to our continuing interest in developing molecular iodine mediated organic transformations [8], herein, we report a iodine promoted β-halovinyl sulfonylation of terminal alkyne using thiol as sulfonyl precurssor and $H_2O_2$ as oxidant to access (*E*)- β-iodovinyl sulfone derivatives with good to excellent yield and complete selectivity.

## 2. Results and discussion

For our initial investigation of vicinal difunctionalization of alkyne we choose ethynylbenzene **1a** and thiophenol **2a** as our model compounds. Accordingly, phenylacetylene **1a** (1.0 equiv) and thiophenol **2a** (1.5 equiv) were allowed to react in presence of iodine (0.5 equiv) and aqueous $H_2O_2$ (2.0 equiv) in DMSO as solvent at room temperature. To our disappointment, the desired β-iodovinyl sulfone **3a** was not detected, rather, the isolated product was found to be the disulfide **4a** as exclusive product (Table 1, Entry 1). Next, the reaction was carried out at 60 °C keeping the other reaction parameters unchanged (Table 1, Entry 2). This time, the desired product **3a** was detected with moderate yield but the formation of disulfide **4a** was not fully suppressed. Being encouraged by this preliminary result, we further increased the reaction temperature to 100 °C without altering any other reaction conditions (Table 1, Entry 3). To our delight, the desired product **3a** was obtained with 74% yield and with no detectable amount of disulfide **4a**. We also examined the feasibility of other oxidant such as TBHP, DTBP, $K_2S_2O_8$ and Oxone (Table 1, Entry 4–7). TBHP and DTBP were able to furnish desired product **3a** though with slightly inferior yield whereas oxone failed to initiate the reaction. On the other hand, disulfide **4a** was formed exclusively when $K_2S_2O_8$ was used as oxidant (Table 1, Entry 6). Therefore, $H_2O_2$ was proved to be the best oxidant among all the oxidant examined in this reaction. To improve the yield, we planned to increase the amount of iodine up to 100 mol% using $H_2O_2$ as oxidant. The best result was obtained when the reaction was carried out with 100 mol% of iodine and $H_2O_2$ as oxidant in DMSO at 100 °C (Table 1, Entry 8). Under this optimized condition, formation of disulfide **4a** was completely suppressed. We also investigated the effect of oxygen by performing the reaction under oxygen (Table 1, Entry 9) atmosphere and we isolated the desired product **3a** with marginally inferior yield compared to the best result. Different iodine sources including NIS, IBX, KI and TBAI were systematically examined (Table 1, Entry 10–13). NIS and IBX were able to produce the desired product though with much inferior yield while both KI and TBAI failed to produce any trace of β-iodovinyl sulfone derivative. Among all the solvents investigated, DMSO was found to be the best one for the current reaction. Other solvents like acetonitrile, DMF and THF (Table 1, Entry 14–16) were also examined under standard reaction condition but in none of the cases the yield was found to be satisfactory.

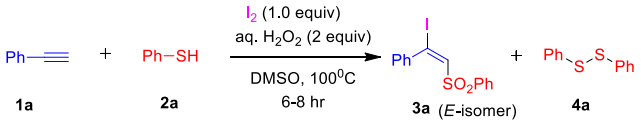
Having the optimized reaction condition in hand, we focused our attention to explore the scope and limitation of our newly developed synthetic method. First, we investigated the substrate scope of the reaction with various alkynes and the results are summarized in Table 2. Terminal aryl acetylenes with different alkyl substitution (H, Me, Et, *n*Bu) at *para*-position were treated with thiophenol **2a** under optimized reaction condition and desired β-iodovinyl sulfone derivatives were isolated with excellent yield (Table 2, Entry **3a**-**3e**) and complete regio and stereoselectivity. It is worth mentioning that in all the cases the product was found to be (*E*)-selective. Terminal acetylene with polyaryl appendage such as 4-ethynylbiphenyl and 1-ethynylnaphthalene were successfully employed as substrate to afford the title compound with excellent yield and complete selectivity (Table 2, Entry **3f**-**3g**). We anticipated that aliphatic acetylenes could also be equally potential candidates for our current investigation. Towards this end, we examined the potential of 1-pentyne, 1-hexyne and cyclopropylacetylene as substrate under the optimized reaction condition. It was gratifying that in each case, the desired product iodovinyl sulfone was obtained though with moderate yield (Table 2, Entry **3h**-**3j**).

Internal alkyne such as diphenylacetylene was tested as substrate under optimized reaction condition but all our efforts were proved to be futile and no trace of desired product **3k** was identified (Table 2, Entry **3k**).

At this juncture, it was important to confirm the stereochemical orientation of the iodovinyl sulfone derivatives. Therefore, compound **3b** was crystallized from dichloromethane and ethyl acetate (20:80) solvent system at room temperature to get a pale yellow crystal suitable for X-ray analysis. Structure determination of **3b** confirmed unambiguously the stereochemistry across the olefinic double bond.

Substrate scope was further extended with various thiophenol derivatives under standard reaction condition. Differently substituted thiophenol including *p*-thiocresol, 4-methoxythiophenol and 4-chlorothiophenol reacted with phenylacetylene **1a** under optimum reaction condition to afford

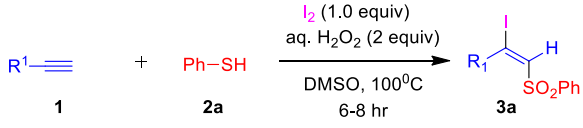
**Table 1**
Preliminary screening of oxidative addition of thiol into alkyne[a].

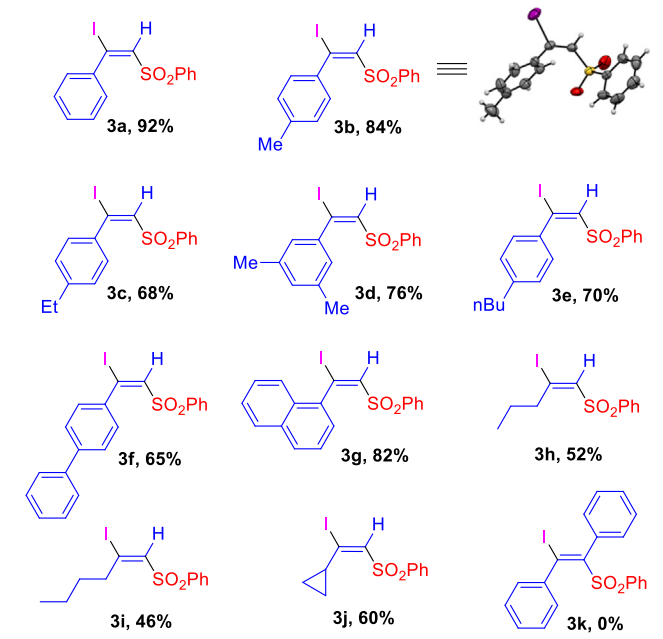



| Entry | Iodine source (equiv) | Oxidant | Solvent | Temprature ($^0$C) | Product[b] ratio(3a/4a) |
|---|---|---|---|---|---|
| 1 | $I_2$ (0.5) | aq. $H_2O_2$ | DMSO | RT | 0/84 |
| 2 | $I_2$ (0.5) | aq. $H_2O_2$ | DMSO | 60 | 54/28 |
| 3 | $I_2$ (0.5) | aq. $H_2O_2$ | DMSO | 100 | 74/0 |
| 4 | $I_2$ (0.5) | TBHP | DMSO | 100 | 68/0 |
| 5 | $I_2$ (0.5) | DTBP | DMSO | 100 | 72/0 |
| 6 | $I_2$ (0.5) | $K_2S_2O_8$ | DMSO | 100 | 0/64 |
| 7 | $I_2$ (0.5) | Oxone | DMSO | 100 | 0/0 |
| **8** | **$I_2$ (1.0)** | **aq. $H_2O_2$** | DMSO | **100** | **92/0** |
| 9[c] | $I_2$ (1.0) | aq. $H_2O_2$ | DMSO | 100 | 88/0 |
| 10 | NIS (1.0) | aq. $H_2O_2$ | DMSO | 100 | 62/0 |
| 11 | IBX | aq. $H_2O_2$ | DMSO | 100 | 36/0 |
| 12 | KI | aq. $H_2O_2$ | DMSO | 100 | ND |
| 13 | TBAI | aq. $H_2O_2$ | DMSO | 100 | ND |
| 14 | $I_2$ (1.0) | aq. $H_2O_2$ | $CH_3CN$ | 100 | 43/0 |
| 15 | $I_2$ (1.0) | aq. $H_2O_2$ | DMF | 100 | 25/0 |
| 16 | $I_2$ (1.0) | aq. $H_2O_2$ | THF | 100 | 52/0 |

[a] Reaction condition: **1a** (1.0 equiv), **2a** (1.5 equiv), $I_2$ (100 mol%), DMSO (2.0 mL), reflux at 100 °C (8–10 h).
[b] Isolated yield.
[c] Reaction was carried out at oxygen atmosphere.

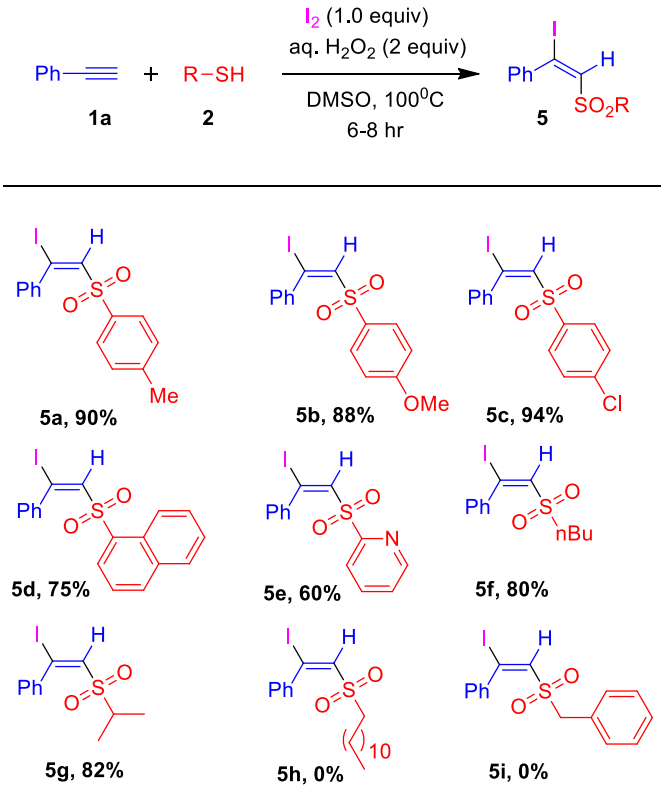
**Table 2**
Substrate scope of the protocol with alkyne[a,b].




[a] Reaction condition: **1** (1.0 equiv), **2** (1.5 equiv), $I_2$ (100 mol%), DMSO (2.0 mL), reflux at 100$^0$C (8-10 hr) [b] Isolated yield

**Table 3**
Substrate scope of the protocol with thiol[a,b].




[a] Reaction condition: **1** (1.0 equiv), **2** (1.5 equiv), $I_2$ (100 mol%), DMSO (2.0 mL), reflux at 100°C (8-10 hr) [b] Isolated yield

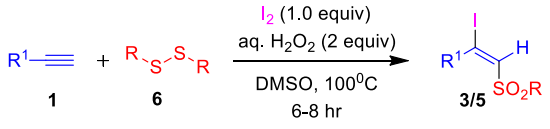
corresponding iodovinyl sulfone **5a**-**5c** with comparable yields (Table 3, Entry **5a**-**5c**) and complete selectivity. Polyaromatic thiol such as 1-naphthalene thiol was proved to be an excellent substrate for our newly developed method to furnish corresponding iodovinyl sulfone **5d** (Table 3, Entry **5d**).

Heterocyclic thiol such as 2-Pyridine thiol was also found to be a potential candidate to react smoothly with phenylacetylene **1a** producing corresponding sulfone derivatives **5e** with very good yield (Table 3, Entry **5e**). The potential of various aliphatic thiols as substrate was systematically investigated under standard reaction condition. 1-Butanethiol and 2-propanethiol were subjected to react with phenylacetylene **1a** to afford respective iodo sulfone derivatives **5f** and **5g** with satisfactory yield (Table 3, Entry **5f**-**5g**). Dodecane thiol and aliphatic thiol with aromatic appendage such as benzyl mercaptan was failed to react with phenylacetylene under standard reaction condition for some unknown reason (Table 3, Entry **5h-5i**).

We anticipated that disulfides might be useful as sulfonylating agent under our optimized reaction condition. To check this possibility, diphenyldisulfide and p-tolyldisulfide were allowed to react with different terminal alkynes under standard condition. All the attempts were gratifyingly successful and, the desired products were obtained with excellent yield and selectivity (Scheme 2). Since most of the disulfides are solid, it is easy to handle and, the use of disulfide as sulfonylating agent certainly broadens the scope of the new reaction.

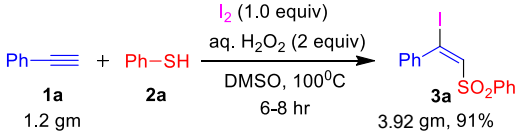
At this point, we were curious to examine whether our current method is useful for gram scale preparation. Therefore, the reaction between our model compounds phenyl acetylene **1a** and thiophenol **2a** was executed in gram scale quantities under standard reaction condition and the reaction was gratifyingly successful in terms of both yield and selectivity (Scheme 3).

Alkenes are known to react with different sulfonylating agents to produce vinyl sulfones under various reaction conditions [1b-c,9]. We were interested to examine whether our newly

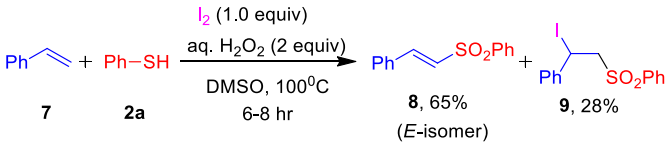



| | | |
|---|---|---|
| $R^1$ = Ph | R = Ph | **(3a)** yield 87% |
| $R^1$ = Ph | R = 4 Me-Ph | **(5a)** yield 82% |
| $R^1$ = 4 Me-Ph | R = Ph | **(3b)** yield 86% |
| $R^1$ = 4(1-butyl)-Ph | R = 4Me-Ph | **(3e)** yield 78% |

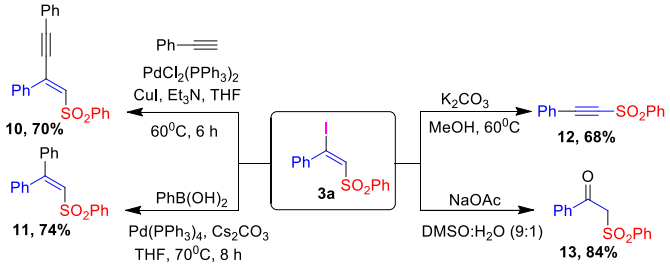
**Scheme 2.** Scope of the protocol with disulfides.




**Scheme 3.** Gram scale preparation of iodo-vinyl sulfone.




**Scheme 4.** Scope of the protocol with Alkene.

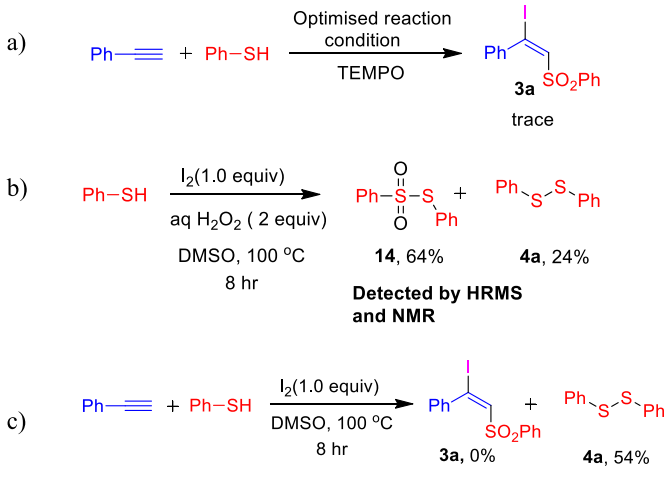



**Scheme 5.** Synthetic application of iodovinyl sulfone.

discovered condition is compatible with alkenes to deliver vinylsulfone derivatives. Therefore, the reaction was carried out between styrene **7** and thiophenol **2a** employing our optimized reaction condition. The expected (*E*)-vinylsulfone **8** was obtained with moderate yield along with the formation of β-iodosulfone **9**, Scheme 4. Probably, incomplete elimination of HI leads to the isolation of **9** as a minor product.

It is needless to mention that Iodovinyl sulfones are invaluable starting material for numerous organic transformation. Iodovinyl motif is an obvious functionality for transition metal catalyzed C–C cross coupling reactions such as Sonogashira [10] and Suzuki [11] reactions. To demonstrate the synthetic utility, iodovinyl sulfone **3a** was treated with phenylacetylene **1a** under typical Sonogashira condition to furnish (*E*)-enyne **10** with very good yield (Scheme 5). Again, **3a** was allowed to react with phenylboronic acid in presence of base and Pd-catalyst to afford Suzuki product **11** with satisfactory yield. Alkynyl sulfone can readily be accessed from (*E*)-β-iodovinyl sulfone under basic condition [6b]. Therefore, treatment of **3a** with $K_2CO_3$ in methanol under heating condition smoothly furnished alkenylsulfone **12** with good yield (Scheme 5). In addition, NaOAc promoted C ($sp^2$)-I hydroxylation of **3a** could efficiently offer α-tosylacetophenone **13** (Scheme 5) [4d].

In order to understand the mechanistic pathway of the iodosulfonylation process, we have conducted a couple of controlled experiments as depicted in Scheme 6. Radical trapping experiment was carried out to know whether any radical intermediate was involved in the reaction pathway. When TEMPO, a well known radical scavenger was employed under optimum reaction condition (Scheme 6, Eqn. a), trace amount of desired product was detected indicating the involvement of radical intermediate in the reaction. Again, when thiophenol **2a** was reacted with our optimal reaction condition in absence of alkyne, benzenesulfonothioate **14** was recovered as a major product along with disulfides which is confirmed by both HRMS and NMR spectroscopy (Scheme 6, eqn. b). This experiment proves the formation of thiosulfonate as a crucial intermediate for the generation of sulfonyl radial. Next, we carried out the reaction in the absence of hydrogen peroxide keeping other reaction parameter unaltered (Scheme 6, eqn. c). This time, we did not get any trace of desired product **3a** rather we ended up with disulfide **4a** as the sole product. This indicates that peroxide is playing a crucial role to generate a sulfonyl radical during the reaction.




**Scheme 6.** Control experiments.

On the basis of the findings of control experiments and literature precedence [12,13] a plausible reaction mechanism of C–S cross coupling between thiol and alkyne has been proposed in Scheme 7. The reaction pathway is presumed to involve an initial oxidative coupling of thiol **2a** into disulfide **4a** under $I_2$/DMSO catalytic oxidant system [12a]. Upon further oxidation with peroxide, disulfide **4a** produced S-phenyl benzenethiosulfonate **14** [13] which on subsequent thermal decomposition generates sulfonyl radical **A** [12c,14]. This in situ generated sulfonyl radical **A** may add to alkyne in two different pathways. In path-a, sulfonyl radial **A** may directly add to alkyne to produced intermediate **B** which on iodination generates product **3a**. Again in path-b, sulfonyl radical **A** may recombine with iodine to produce phenyl sulfonyl iodide **C** which then add to alkyne followed by iodination liberates *E*-iodo vinyl sulfone **3a** (Scheme 7).

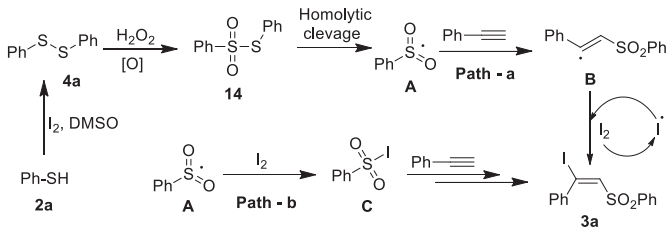
## 3. Conclusion

In summary, we have developed an efficient, molecular iodine promoted and transition metal free method for the synthesis of (*E*)-iodo vinyl sulfones from commercially available, inexpensive starting material via oxidative C–S coupling. A wide range of iodovinyl sulfone derivatives can be readily prepared from milligram to multi-gram scale with excellent yield and complete regio and stereoselectivity. The operational simplicity and environmentally benign nature make the protocol more attractive. Further study and application of iodovinyl sulfones are currently underway in our laboratory.

## 4. Experimental section

General procedure for the synthesis of (*E*)-(1-iodo-2-(phenylsulfonyl)vinyl)benzene (3a) and derivatives:

To a 10 mL round bottom flask was charged with thiophenol **2a**




**Scheme 7.** Postulated reaction pathway.

(0.15 mL, 1.5 mmol), DMSO (2 mL) and iodine (127 mg, 0.5 mmol) and aqueous $H_2O_2$ (0.06 mL, 2 mmol) and the mixture was stirred for half an hour at room temperature. After that phenyl acetylene **1a** (0.11 mL, 1.0 mmol) was added and the resulting reaction mixture was heated to 100 °C for 6–8 h under nitrogen atmosphere. After completion of the reaction as indicated by TLC, reaction flask was cooled for a while. After that 10% aqueous $Na_2S_2O_3$ solution (30 mL) was added to the flask, the whole reaction mixture was extracted with ethyl acetate (3 × 60 mL). The combined organic layer was washed with brine solution and dried over anhydrous $Na_2SO_4$ and concentrated under reduced pressure. The crude product was purified on via column chromatography (100–200 mesh silica gel) with petroleum ether and ethyl acetate (20% of solution) as eluent to yield the desired product **3a** as pale yellow solid (340 mg, yield 92%). Also other derivatives of iodosulfone **3b-3m** were synthesized by the above procedure in smooth yield.

**(*E*)-(1-iodo-2-(phenylsulfonyl)vinyl)benzene (3a):** White solid (340 mg, yield 92%), mp 68–69 °C.**$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$(ppm): 7.56–7.49 (m, 3H), 7.36 (t, $J$ = 8.6 Hz, 3H), 7.29–7.22 (m, 3H), 7.19 (t, $J$ = 6.7 Hz,2H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 141.1, 140.2, 139.6, 133.5, 129.9, 129.1, 128.0, 127.8, 127.7, 114.8. **HRMS ESI (m/z):** Calculated for $C_{14}H_{11}IO_2S$ [M+ Na]$^+$: 392.9422, found: 392.9427.

**(*E*)-1-(1-iodo-2-(phenylsulfonyl)vinyl)-4-methylbenzene(3b):** White solid (323 mg, 84%), mp 113–115 °C. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$(ppm): 7.52 (d, $J$ = 7.7 Hz, 2H), 7.46 (t, $J$ = 7.4 Hz, 1H), 7.31 (t, $J$ = 7.5 Hz, 2H), 7.07–6.99 (m, 4H), 2.26 (s, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$(ppm): 140.5, 140.3, 136.8, 133.5, 129.0, 128.7, 128.6, 127.8, 115.4, 21.5. **HRMS ESI (m/z):** Calculated for $C_{15}H_{13}IO_2S$ [M + Na]$^+$: 406.9579, found: 406.9584.

**(*E*)-1-ethyl-4-(1-iodo-2-(phenylsulfonyl)vinyl)benzene(3c):** Gummy solid (270 mg, 68%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$(ppm): 7.61–7.54 (m, 3H), 7.41–7.37 (m, 3H), 7.17 (d, $J$ = 8.3 Hz, 2H), 7.11 (d, $J$ = 8.3 Hz, 2H), 2.69–2.64 (m, 2H), 1.27 (t, $J$ = 7.6 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$(ppm): 146.5, 140.7, 140.2, 136.9, 133.4, 128.9, 127.9, 127.8, 127.4, 115.4,28.7, 15.3. **HRMS ESI (m/z):** Calculated for $C_{16}H_{15}IO_2S$ [M + H]$^+$: 398.9916, found: 398.9918.

**(*E*)-1-(1-iodo-2-(phenylsulfonyl)vinyl)-3,5-dimethylbenzene (3d):** Light brown solid (302 mg, 76%), mp 108–110 °C. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.58–7.54 (m, 3H), 7.42–7.37 (m, 3H), 6.93 (s, 1H), 6.76 (s, 2H), 2.26 (d, $J$ = 1.9 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 140.9, 140.3, 139.4, 137.6, 133.2, 131.5, 128.8, 127.9, 125.1, 115.5, 21.2. **HRMS ESI (m/z):** Calculated for $C_{16}H_{15}IO_2S$ [M+H]$^+$: 398.9916, found: 398.9920.

**(*E*)-1-butyl-4-(1-iodo-2-(phenylsulfonyl)vinyl)benzene (3e):** Brown semi solid (298 mg, 70%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$(ppm): 7.56–7.49 (m, 3H), 7.35 (t, $J$ = 8.7 Hz, 3H), 7.13 (t, $J$ = 6.4 Hz, 2H), 7.06 (d, $J$ = 8 Hz, 2H), 2.59 (t, $J$ = 7.6 Hz, 2H), 1.63–1.56 (m, 2H), 1.42–1.32 (m,2H), 0.95 (t, $J$ = 7.3 Hz, 3H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) $\delta$(ppm): 145.3, 140.8, 140.3, 136.9, 135.3, 129.0, 128.0, 128.0, 127.9, 115.7, 35.6, 33.4, 22.4, 14.1.

**HRMS ESI (m/z):** Calculated for $C_{18}H_{19}IO_2S$ [M+H]$^+$: 427.0229, found: 427.0234.

**(*E*)-4-(1-iodo-2-(phenylsulfonyl)vinyl)-1,1′-biphenyl (3f)**: Gummy solid (290 mg, 65%), **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.59 (t, $J$ = 8.4 Hz, 4H), 7.53 (t, $J$ = 7.5 Hz, 1H), 7.48 (t, $J$ = 8.3 Hz, 4H), 7.41–7.35 (m, 4H), 7.29 (d, $J$ = 8.2 Hz, 2H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) $\delta$ (ppm): 142.7, 141.3, 140.2, 139.9, 138.4, 133.5, 129.0, 128.9, 128.4, 128.0, 127.9, 127.1, 126.6, 114.4. **HRMS ESI (m/z):** Calculated for $C_{20}H_{15}IO_2S$ [M+H]$^+$: 446.9916, found: 446.9921.

**(*E*)-1-(1-iodo-2-(phenylsulfonyl)vinyl)naphthalene (3g):** Brown liquid (345 mg, 82%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.78–7.72 (m,2H), 7.65 (s, 2H), 7.49–7.47 (m, 1H), 7.39–7.32 (m, 4H), 7.27–7.25 (m, 2H), 7.18–7.14 (m, 1H), 7.01–6.97 (m, 2H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) $\delta$ (ppm): 142.6, 138.1, 134.9, 132.3, 132.0, 129.1, 127.4, 127.2, 126.7, 125.5, 125.2, 124.2, 123.9, 123.8, 118.9, 111.3. **HRMS ESI (m/z):** Calculated for $C_{18}H_{13}IO_2S$ [M+H]$^+$: 420.9759, found: 420.9763.

**(*E*)-((2-iodopent-1-en-1-yl)sulfonyl)benzene (3h)**: Oil (174 mg, 52%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm) = 7.93 (d, $J$ = 7.7 Hz, 2H), 7.70–7.67 (m, 1H), 7.59 (t, $J$ = 7.5 Hz, 2H), 7.06 (s, 1H), 3.04 (t, $J$ = 7.7 Hz, 2H), 1.65–1.56 (m, 2H), 0.99–0.95 (m, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$(ppm) = 140.9, 138.8, 133.8, 129.5, 127.5, 16.0, 41.5, 23.3, 12.8. **HRMS ESI (m/z):** Calculated for $C_{11}H_{13}IO_2S$ [M+H]$^+$: 336.9759, found: 336.9763.

**(*E*)-((2-iodohex-1-en-1-yl)sulfonyl)benzene (3i)**: Oil (162 mg, 46%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm) = 7.85–7.83 (m, 2H), 7.61–7.57 (m, 1H), 7.52–7.48 (m, 2H), 6.94 (s, 1H), 2.97 (t, $J$ = 7.6 Hz, 2H), 1.47–1.41 (m, 2H), 1.33–1.27 (m, 2H), 0.93–0.85 (m, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$(ppm) = 141.0, 138.6, 133.7, 129.5, 127.4, 126.2, 39.9, 32.0, 21.7, 13.8. **HRMS ESI (m/z):** Calculated for $C_{12}H_{15}IO_2S$ [M+H]$^+$: 350.9916, found: 350.9920.

**(*E*)-((2-cyclopropyl-2-iodovinyl)sulfonyl)benzene (3j):** Brown Semi solid (198 mg,60%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.92 (t, $J$ = 7.6 Hz, 2H), 7.67–7.63 (m, 1H), 7.56 (t, $J$ = 7.9 Hz, 2H), 7.06 (s, 1H), 2.47–2.41 (m, 1H), 0.94–0.83 (m, 4H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 141.3, 138.0, 137.2, 134.2, 129.5, 129.2, 127.4, 17.6, 17.4, 12.2. **HRMS ESI (m/z):** Calculated for $C_{11}H_{11}IO_2S$ [M+H]$^+$: 334.9603, found: 334.9612.

**(*E*)-1-((2-iodo-2-phenylvinyl)sulfonyl)-4-methylbenzene (5a):** White solid (345 mg, 90%), mp 80–82 °C. **$^1$H NMR** (500 MHz, $CDCl_3$) $\delta$ (ppm) = 7.44 (d, $J$ = 7.6 Hz, 2H), 7.35 (s,1H), 7.27 (t, $J$ = 7.0 Hz, 3H), 7.21 (d, $J$ = 7.1 Hz, 2H), 7.16 (d, $J$ = 7.3 Hz, 2H), 2.34 (s,3H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) $\delta$ (ppm): 144.6, 141.3, 139.7, 137.3, 129.8, 129.7, 127.9, 127.8, 127.7, 114.2, 21.6. **HRMS ESI (m/z):** Calculated for $C_{15}H_{13}IO_2S$ [M+H]$^+$: 384.9759, found: 384.9764.

**(*E*)-1-((2-iodo-2-phenylvinyl)sulfonyl)-4-methoxybenzene (5b):** White solid (352 mg, 88%), mp 112–114 °C. **$^1$H NMR** (500 MHz, DMSO-$d_6$) $\delta$ (ppm): 7.52–7.49 (m, 2H), 7.39 (s, 1H), 7.33–7.29 (m, 3H), 7.25 (t, $J$ = 5 Hz, 2H), 6.87–6.85 (m, 2H), 3.86 (s, 3H). **$^{13}$C NMR** (125 MHz, DMSO-$d_6$) $\delta$ (ppm): 163.6, 141.6, 139.7, 131.7, 130.1, 129.7, 127.9, 127.7, 114.2, 113.6, 55.7. **HRMS ESI (m/z):** Calculated for $C_{15}H_{13}IO_3S$ [M+Na]$^+$: 422.9528, found: 422.9538.

**(*E*)-1-chloro-4-((2-iodo-2-phenylvinyl)sulfonyl)benzene (5c):** White solid (380 mg, 94%), mp 104–106 °C. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.45 (d, $J$ = 8.3 Hz, 2H), 7.38 (s, 1H), 7.32–7.24 (m, 5H), 7.17 (t, $J$ = 6.8 Hz, 2H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 141.0, 140.2, 139.5, 138.7, 130.0, 129.4, 129.3, 128.0, 127.7, 115.3. **HRMS ESI (m/z):** Calculated for $C_{22}H_{16}O_2S$ [M+H]$^+$: 345.0949, found: 345.0956.

**(*E*)-1-((2-iodo-2-phenylvinyl)sulfonyl)naphthalene (5d):** White solid (316 mg, 75%), mp 78–80 °C. **$^1$H NMR** (400 MHz, DMSO-$d_6$) $\delta$ (ppm): 7.98 (s, 1H), 7.82 (d, $J$ = 8.4 Hz, 2H), 7.73 (d, $J$ = 8.1 Hz, 1H), 7.62–7.51 (m, 3H), 7.45 (s, 1H), 7.20–7.15 (m, 5H), 6.73–6.71 (m, 1H). **$^{13}$C NMR** (100 MHz, DMSO-$d_6$) $\delta$ (ppm): 141.3, 139.5, 136.8, 135.1, 131.9, 130.0, 129.9, 129.4, 129.3, 127.9, 127.7, 127.6, 112.4, 114.9. **HRMS ESI (m/z):** Calculated for $C_{18}H_{13}IO_2S$ [M+Na]$^+$: 442.9579, found: 442.9584.

**(*E*)-2-((2-iodo-2-phenylvinyl)sulfonyl)pyridine (5e):** White solid (223 mg, 60%), brown oil. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 8.67 (d, $J$ = 4.3 Hz, 1H), 7.68 (t, $J$ = 7.7 Hz, 1H), 7.56 (t, $J$ = 9.7 Hz, 1H), 7.43–7.20 (m, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 157.6, 150.3, 139.7, 138.6, 137.9, 129.9, 127.9, 127.7, 127.3, 122.5, 116.1. **HRMS ESI (m/z):** Calculated for $C_{13}H_{10}INO_2S$ [M+H]$^+$: 371.9555, found: 371.9548.

**(*E*)-(2-(butylsulfonyl)-1-iodovinyl)benzene (5f):** Yellowish Green oil (280 mg, 80%), mp 86–88 °C. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.45–7.42 (m, 2H), 7.37–7.34 (m, 3H), 7.21 (s, 1H), 2.69–2.65 (m, 2H), 1.70–1.62 (m, 2H), 1.36–1.26 (m, 2H). 0.85 (t,

$J$ = 7.6 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 139.6, 138.9, 130.3, 128.7, 128.2, 127.9, 115.2, 54.5, 24.0, 21.5, 13.5. **HRMS ESI (m/z):** Calculated for $C_{12}H_{15}IO_2S$ $[M+H]^+$: 350.9916, found: 350.9925.

**(*E*)-(1-iodo-2-(isopropylsulfonyl)vinyl)benzene (5g):** Yellowish Green oil (275 mg, 82%), mp 136–138 °C. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.44–7.39 (m, 3H), 7.33–7.31 (m, 2H), 7.16 (s, 1H), 2.82–2.75 (m, 1H), 1.25 (d, $J$ = 6.8 Hz, 6H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 136.4, 130.1, 128.7, 128.5, 128.0, 127.8, 116.1, 54.5, 15.1. **HRMS ESI (m/z):** Calculated for $C_{11}H_{13}IO_2S$ $[M+H]^+$: 336.9759, found: 336.9764.

**(*E*)-(2-(phenylsulfonyl)vinyl)benzene (8):** Yellowish Green oil (159 mg, 65%), mp 70–72 °C.**$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.87–7.85 (m, 2H), 7.60 (d, $J$ = 15.4 Hz, 1H), 7.51 (t, $J$ = 6.1 Hz, 2H), 7.47–7.43 (m, 2H), 7.40–7.37 (m, 2H), 7.31 (d, $J$ = 5.2 Hz, 3H), 6.78 (d, $J$ = 15.4 Hz, 1H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 41.5, 140.7, 133.4, 132.3, 131.2, 129.4, 129.1, 128.6, 127.7, 127.3. **HRMS ESI (m/z):** Calculated for $C_{14}H_{12}O_2S$ $[M+Na]^+$: 267.0456, found: 267.0458.

**(1-iodo-2-(phenylsulfonyl)ethyl)benzene (9):** Gummy solid (104 mg, 28%). **$^1$H NMR** (500 MHz, $CDCl_3$) $\delta$ (ppm): 7.46 (t, $J$ = 7.3 Hz, 2H), 7.38 (d, $J$ = 4.3 Hz, 4H), 7.37–7.32 (m, 3H), 7.28 (t, $J$ = 5.2 Hz, 1H), 4.77–4.75 (m, 1H), 3.37–3.33 (m, 1H), 3.16–3.11 (m, 1H).

**$^{13}$C NMR** (125 MHz, $CDCl_3$) $\delta$ (ppm): 142.2, 135.0, 130.2, 129.2, 128.7, 128.6, 128.0, 126.8, 12-5.9, 125.8, 71.7, 44.0. **HRMS ESI (m/z):** Calculated for $C_{14}H_{13}IO_2S$ $[M+H]^+$: 372.9759, found: 372.9762.

**(*E*)-(4-(phenylsulfonyl)but-3-en-1-yne-1,3-diyl)dibenzene (10)**[1]: Yellowish Green oil (241 mg, 70%). **$^1$H NMR** (4-00 MHz, $CDCl_3$) $\delta$ (ppm): 7.82–7.80 (m,2H), 7.57–7.51 (m, 4H), 7.44–7.42 (m, 2H), 7.37–7.32 (m, 5H), 7.04 (t, $J$ = 7.1 Hz, 1H), 6.52 (s, 1H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 153.4, 143.5, 135.1, 132.7, 131.7, 131.6, 129.9, 129.7, 129.1, 128.6, 128.4, 127.9, 127.7, 121.7, 99.5, 85.1. **HRMS ESI (m/z):** Calculated for $C_{22}H_{16}O_2S$ $[M+H]^+$: 345.0949, found: 345.0951.

**(2-(phenylsulfonyl)ethene-1,1-diyl)dibenzene (11)**[1]: light brown solid (237 mg, 74%), mp 98–100 °C. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 8.07 (d, $J$ = 6.5 Hz, 1H), 7.77 (s, 1H), 7.59 (d, $J$ = 9.0 Hz, 1H), 7.46 (t, $J$ = 1.5 Hz, 1H), 7.16–7.13 (m, 1H), 6.90 (d, $J$ = 3.5 Hz, 1H), 6.77–6.74 (m, 1H). 6.50–6.49 (m, 1H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 155.3, 141.5, 139.1, 135.5, 132.9, 130.4, 129.8, 129.0, 128.8, 128.7, 128.7, 128.3 **HRMS ESI (m/z):** Calculated for $C_{20}H_{16}O_2S$ $[M+H]^+$: 321.0949, found: 321.0952.

**((phenylethynyl)sulfonyl)benzene (12)**[1]: Gummy liquid (157 mg, 65%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.98 (d, $J$ = 8.3 Hz, 2H), 7.55–7.51 (m, 2H), 7.49–7.47 (m, 1H), 7.42–7.37 (m, 5H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 145.4, 138.9, 132.7, 131.5, 130.0, 128.7, 127.5, 118.0, 92.9, 85.6. **HRMS ESI (m/z):** Calculated for $C_{14}H_{10}O_2S$ $[M+H]^+$: 243.0480, found: 243.0486.

**1-phenyl-2-(phenylsulfonyl)ethanone (13)**[2]: White solid, (218 mg, 84%), mp 100–102 °C. **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.91–7.87 (m, 4H), 7.65–7.56 (m, 2H), 7.51 (t, $J$ = 8.0 Hz, 2H), 7.44 (t, $J$ = 8.0 Hz, 2H), 4.75 (s, 2H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 188.0, 138.8, 135.7, 134.3, 134.2, 129.3, 129.2, 128.8, 128.5, 63.3. **HRMS ESI (m/z):** Calculated for $C_{14}H_{12}O_3S$ $[M+H]^+$: 261.0585, found: 261.0595.

**S-phenyl benzenesulfonothioate (14):** Yellowish liquid (160 mg, 64%). **$^1$H NMR** (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.48–7.43 (m, 3H), 7.38–7.34 (m, 1H), 7.24–7.20 (m, 4H), 7.30 (t, $J$ = 7.9 Hz, 2H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) $\delta$ (ppm): 142.8, 136.6, 133.8, 131.5, 129.5, 128.9, 127.8, 127.5. **HRMS ESI (m/z):** Calculated for $C_{12}H_{10}O_2S_2$ $[M+H]^+$: 251.0200, found: 251.0190.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.tet.2021.132310.

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