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Hypervalent Iodine-Mediated Synthesis of Sulfones Using Organozinc Pivalates and Sulfinate Salts

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ABSTRACT

Sulfones are key motifs in pharmaceuticals, agrochemicals, and functional materials, as well as versatile intermediates in synthesis. We report a general and practical protocol for sulfone synthesis that combines the group-transfer capability of hypervalent iodine reagents (HIRs)—particularly in situ-generated sulfonyl-containing HIRs—with organozinc pivalates. The method delivers a broad range of sulfones in high yields under mild conditions, exhibiting excellent functional group tolerance. Importantly, the method enables efficient sulfonylation using sodium bicyclo[1.1.1]pentane sulfinate (BCP–SO_2Na), providing streamlined access to functionalized BCP-sulfones. The protocol further enables in situ sulfinate generation and one-pot transformations. These findings expand the scope of HIR-enabled umpolung reactivity and offer a practical, modular and operationally simple platform for accessing sulfones with broad synthetic and medicinally relevance.

1 | Introduction

Sulfonyl-containing compounds, including sulfones, sulfonamides, and sulfonyl hydrazides, constitute an important class of functional groups in organic, medicinal, and agrochemical chemistry [1–3]. Among these, sulfones are particularly prominent, as they are found in a variety of biologically active molecules and have been shown to display antibacterial, anti-HIV, and antifungal activity (Figure 1) [4–9].

From a synthetic perspective, sulfones are highly versatile intermediates and play a central role in classic transformations such as the Ramberg–Bäcklund reaction and the Julia olefination [10–13]. Traditional methods for sulfone synthesis often rely on hazardous reagents, such as gaseous SO_2, which poses significant challenges in terms of handling safety [14]. As an alternative, sulfonyl chlorides are commonly employed as electrophilic SO_2 sources and are widely used in sulfonylation chemistry; however, their broad application is constrained by limited functional group tolerance [15, 16]. Other strategies involve the use of sulfinate salts, particularly sodium sulfinates, which can participate in reactions with electrophiles or in metal-catalyzed processes [17, 18]. Despite their utility, these reagents suffer from limited commercial availability and are often prepared as sulfonyl chlorides, thereby restricting broader application [19]. Additional approaches to sulfone synthesis include diazo-mediated reactions, Friedel–Crafts-type reactions, or metal-catalyzed transformations [20–23]. More recently, significant advances have been achieved through the development of SO_2 surrogates, such as DABSO, as well as electrochemical and photochemical methodologies, and the use of hypervalent iodine reagents (HIRs) (Figure 2) [24–30]. While these methods represent

[Correction added on 12 March 2026, after first online publication: Correction in dedication line.]

Dedicated to Armin de Meijere (1939–2025) and Henning Hopf for his 85th birthday – two pioneering organic chemists.

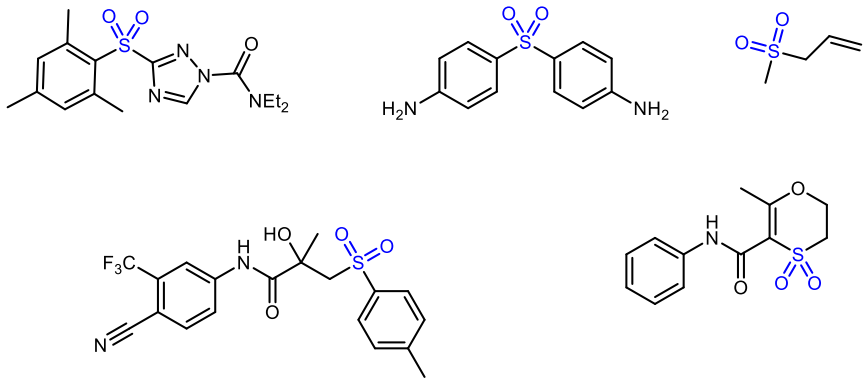


FIGURE 1 | Representative biologically active sulfones [4–9].

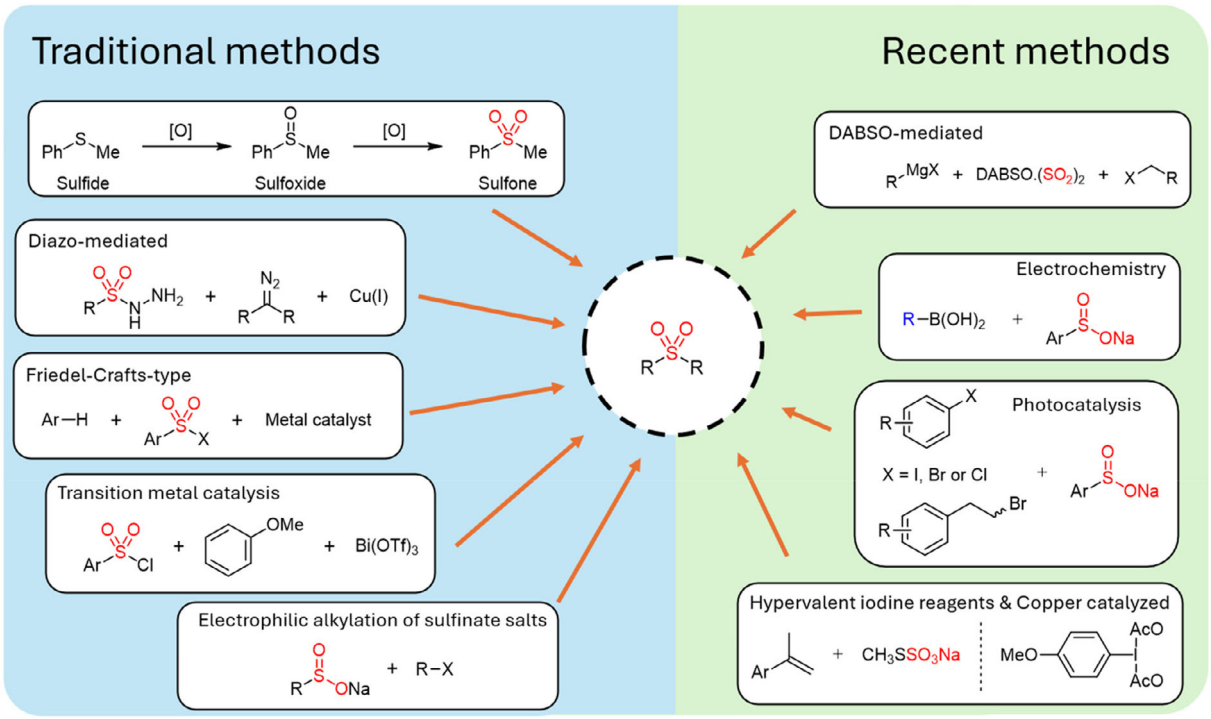
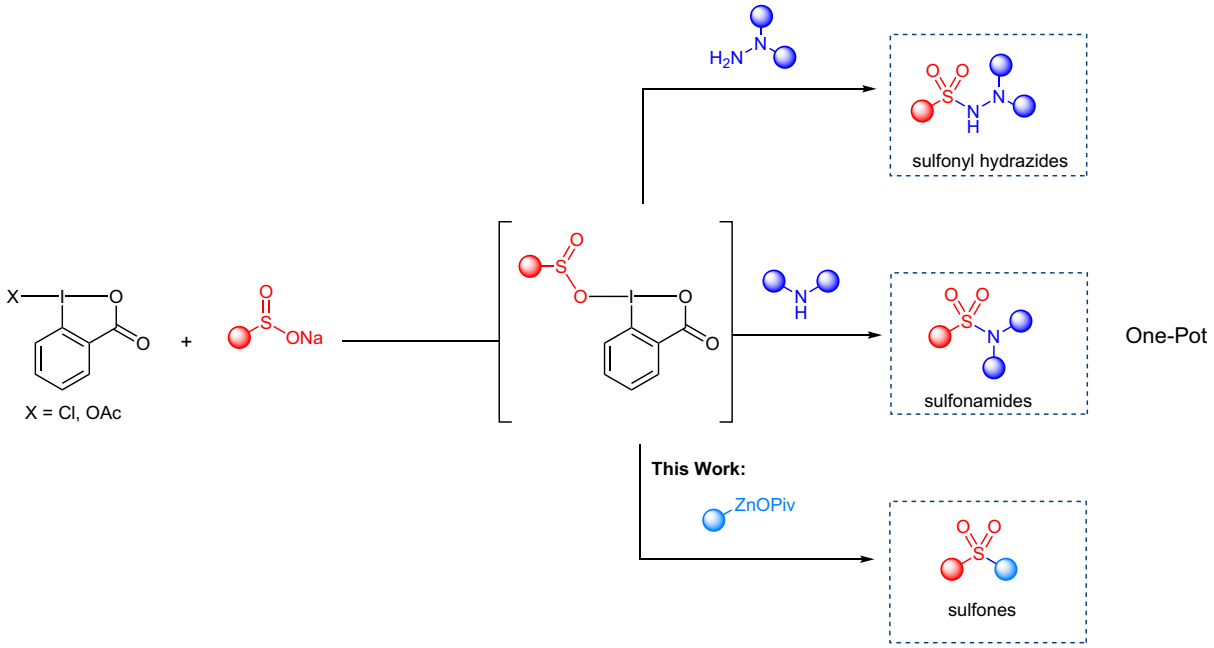


FIGURE 2 | Traditional and recent methods used for sulfone synthesis.

important progress, many still require metal catalysts and/or prefunctionalized substrates, highlighting the continued need for efficient, broadly applicable, and operationally simple strategies for the synthesis of sulfonyl derivatives.

HIRs have attracted considerable attention due to their powerful oxidative properties and unique ability to enable umpolung reactivity [31]. In particular, iodine(III) compounds exhibit reactivity patterns reminiscent of transition-metal complexes, serving as electrophilic synthons of intrinsically nucleophilic functional groups. Among these, cyclic benziodoxolones are especially attractive due to their enhanced stability and have found widespread application in electrophilic and oxidative atom-transfer reactions. Prominent examples of this reagent class include Togni's reagent, ethynylbenziodoxolone (EBX), and azidobenziodoxolone (ABX), which enable the efficient transfer of trifluoromethyl, alkynyl, and azide groups, respectively [32, 33]. Our group has previously investigated the umpolung reactivity of HIRs, particularly benziodoxolones, in the synthesis of sulfonamides [34] and sulfonyl hydrazides [35], via formation of a sulfonyl-benziodoxolone as key intermediate (Scheme 1).

In this work, we report a new and efficient approach to sulfone synthesis. By leveraging our expertise in situ generation of sulfonyl-containing HIR intermediates, we developed a method that enables the formation of a broad range of sulfones in high to quantitative yields using stable sulfinate salts in combination with organozinc pivalates. A highlight of this work is the successful sulfonylation of structurally diverse substrates employing the rigid bicyclo[1.1.1]pentane sulfinate salt (BCP–SO_2Na), providing streamlined access to functionalized BCP-sulfones. Notably, this innovative approach allows for the in situ generation of the sulfinate salt and exhibits excellent compatibility with subsequent transformations, rendering it particularly attractive for one-pot sulfone synthesis.

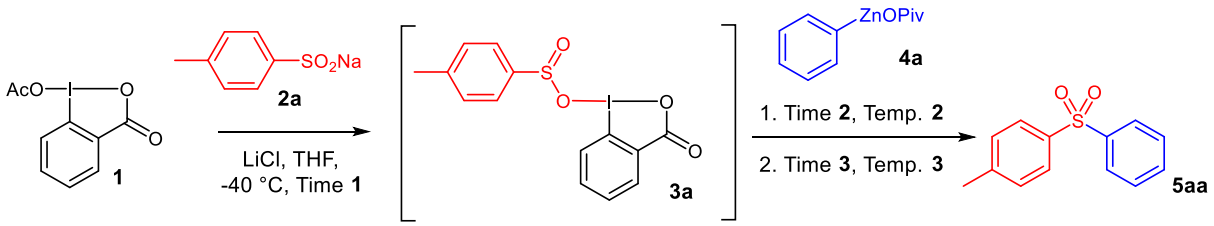


SCHEME 1 | Our previous work on hypervalent iodine-mediated sulfonamide and sulfonyl hydrazides synthesis and this work.

2 | Results and Discussion

Acetoxybenziodoxolone (**1**) was used in combination with sodium *p*-tolylsulfinate salt (**2a**) and phenylzinc pivalate (**4a**), the latter generated by transmetallation [36]. The study was initiated by applying conditions previously developed in our laboratory for the synthesis of sulfonyl hydrazides [35], together with insights gained from our studies on the electrophilic amination of arylzinc pivalates (Table 1, entry 1) [37]. Accordingly, acetoxybenziodoxolone (**1**) was used as

TABLE 1 | Optimization studies.



	SO_2Na, equiv	1, equiv	4a, equiv	TBAI, equiv	LiCl, equiv	Catalyst	Time 1, h	Temp. 2, °C	Time 2, h	Temp. 3, °C	Time 3, h	Yield, %
1	1	1	2	0.2	—	—	0.5	−40	1	—	—	trace
2	1	1	2	0.2	1	——	0.5	−15	4	—	—	37
3	1	1	2	0.2	1	TEA	0.5	−15	4	—	—	25
4	1	1	2	0.2	1	—	0.5	−15	0.5	—	—	46
5	1.5	1	2.5	0.2	1	—	0.5	−40	0.5	—	—	trace
6	1.5	1	2.5	0,2	1	$Cu(OAc)_2$	0.5	−15	0.5	—	—	trace
7	1.5	1	2.5	0.2	1	—	0.5	−15	0.5	—		57
8	1.5	1	2.5	0.2	1	—	0.5	−15	0.5	40	2	61
9	1.5	1	2.5	0.2	1	—	0.5	−15	0.5	40	12	21
10	2.5	1	5	0.2	1	—	0.5	−15	0.5	40	2	55
11	**1.5**	**1**	**2.5**	**—**	**1**	**—**	**0.5**	**−15**	**0.5**	**40**	**2**	**65**
12[a]	1.5	1	2.5	—	1	—	0.5	−15	0.5	40	2	38
13	1.5	1	2.5	—	1	$Zn(OTf)_2$	0.5	−15	0.5	40	2	66
14	1.5	1	2.5	—	1	CuCl	0.5	−15	0.5	40	2	32

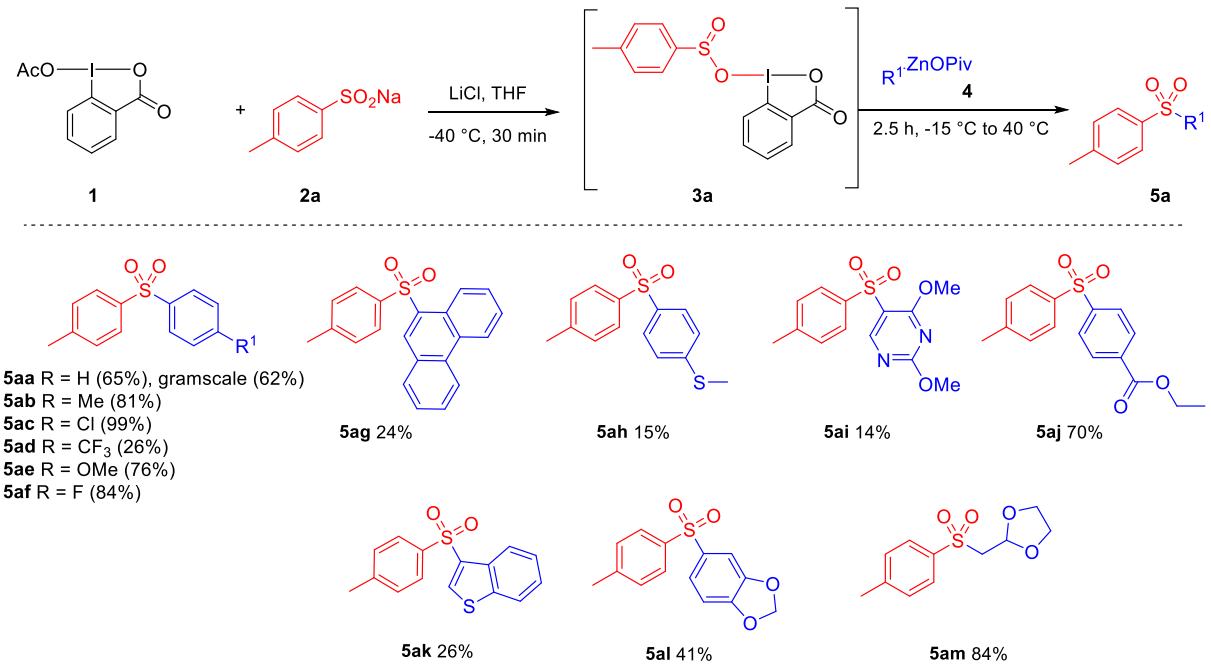
[a]PhZnCl was used instead of PhZnOPiv.

the limiting reagent and first combined with the sodium *p*-tolylsulfinate salt (**2a**) to generate the corresponding sulfonylbenziodoxolone intermediate **3a.** Subsequently, two equivalents of organozinc reagent **4a** were added to react with intermediate **3a**, delivering the desired sulfone **5aa**.

With the reaction conditions for the formation of intermediate **3a** established, we next examined its reactivity toward the organozinc reagent **4a**. To enhance reactivity while preserving the stability of both intermediate **3a** and organozinc **4a** under the reaction conditions, the temperature was slightly increased to −15°C prior to the addition of **4a** (Table 1, entry 2). We then investigated the effect of stoichiometry. When acetoxybenziodoxolone **1** was used as the limiting reagent, in combination with 1.5 equivalents of sulfinate salt **2a** and 2.5 equivalents of organozinc reagent **4a**, the desired sulfone was obtained in 57% yield (Table 1, entry 7). Having established the temperature and stoichiometry, we next evaluated the effect of higher reaction temperatures during the coupling with **4a**. Conducting the reaction at 40°C for 2 h led to a modest increase in yield to 61% and, importantly, resulted in a cleaner reaction profile, which translated into a more straightforward work-up and purification (Table 1, entry 8). Finally, the reaction was carried out in the absence of TBAI, as 1 equivalent of LiCl was already present in the reaction mixture. Under these conditions, a further improvement in yield to 65% was observed (Table 1, entry 11), thereby establishing the optimized reaction conditions.

We next evaluated the effect of using alternative organozinc reagents. Replacing the arylzinc pivalate with zinc chloride led to a decrease in efficiency, with the yield reduced by ≈50% (Table 1, entry 12). The influence of catalysts was also examined, and $Zn(OTf)_2$ had little effect on the reaction outcome, whereas $Cu(OAc)_2$ and CuCl proved highly detrimental, resulting in significantly reduced yields (Table 1, entries 13 and 14). We subsequently investigated the influence of the organozinc reagent concentration. Previous studies by Knochel and co-workers have demonstrated that the concentration of organometallic reagents can be critical for controlling both the reactivity and selectivity [38]. Consistent with these observations, our concentration study revealed a linear correlation between yield and organozinc reagent concentration, indicating that concentrations above 0.86 M are required to achieve optimal yields (Table S1).

To assess the scope and versatility of the reaction, a range of aryl zinc pivalates was examined using *p*-tolylsulfinate salt (**2a**) as a model substrate (Scheme 2). Among the substrates evaluated, 4-chlorophenylzinc pivalate afforded the highest yield (**5ac** 99%), likely reflecting a favorable balance between weak resonance donation and inductive electron-withdrawing effects, which moderately activates the aromatic ring. The 4-fluorophenylzinc pivalate, which is weakly donating by resonance but less polarizable than chlorine, also delivered a high yield (**5af** 84%). In contrast, the electron-donating 4-methoxyphenylzinc pivalate, provided a lower yield (**5ae**, 76%), while the electron-withdrawing 4-carboxyethylphenylzinc pivalate, afforded the sulfone in 70% yield (**5aj**). As expected, the electron-withdrawing 4-trifluoromethylphenylzinc pivalate was significantly less effective, giving **5ad** in 26% yield, consistent with the reduced nucleophilicity of the corresponding arylzinc species under the reaction conditions (Scheme 2). Extension of the methodology to heteroaryl zinc reagents revealed a more pronounce dependence on π-electron density. The 2,6-dimethoxypyrimidine **5ai** was obtained in a modest 14% yield, whereas slightly higher yields were observed for fuzed heteroarenes, such as benzothiophene (**5ak**) and benzodioxole (**5al**), in line with their increase π-electron density. Notably, the reaction proved to be highly scalable, proceeding without loss of efficiency when scaled from milligram to gram quantities (Scheme 2, compound **5aa**). The methodology was successfully extended to a non-aromatic organozinc reagent, with methyl dioxolane-derived sulfone



SCHEME 2 | Scope with optimized conditions.

5am obtained in excellent 84% yield, highlighting the broad applicability of the protocol.

When different sulfinate salts were evaluated, we observed that the conditions optimized for sodium *p*-tolylsulfinate (**2a**) were not generally applicable, necessitating further optimization across the broader sulfinate scope (Figure 3).

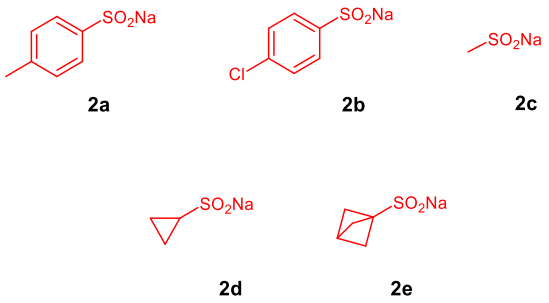
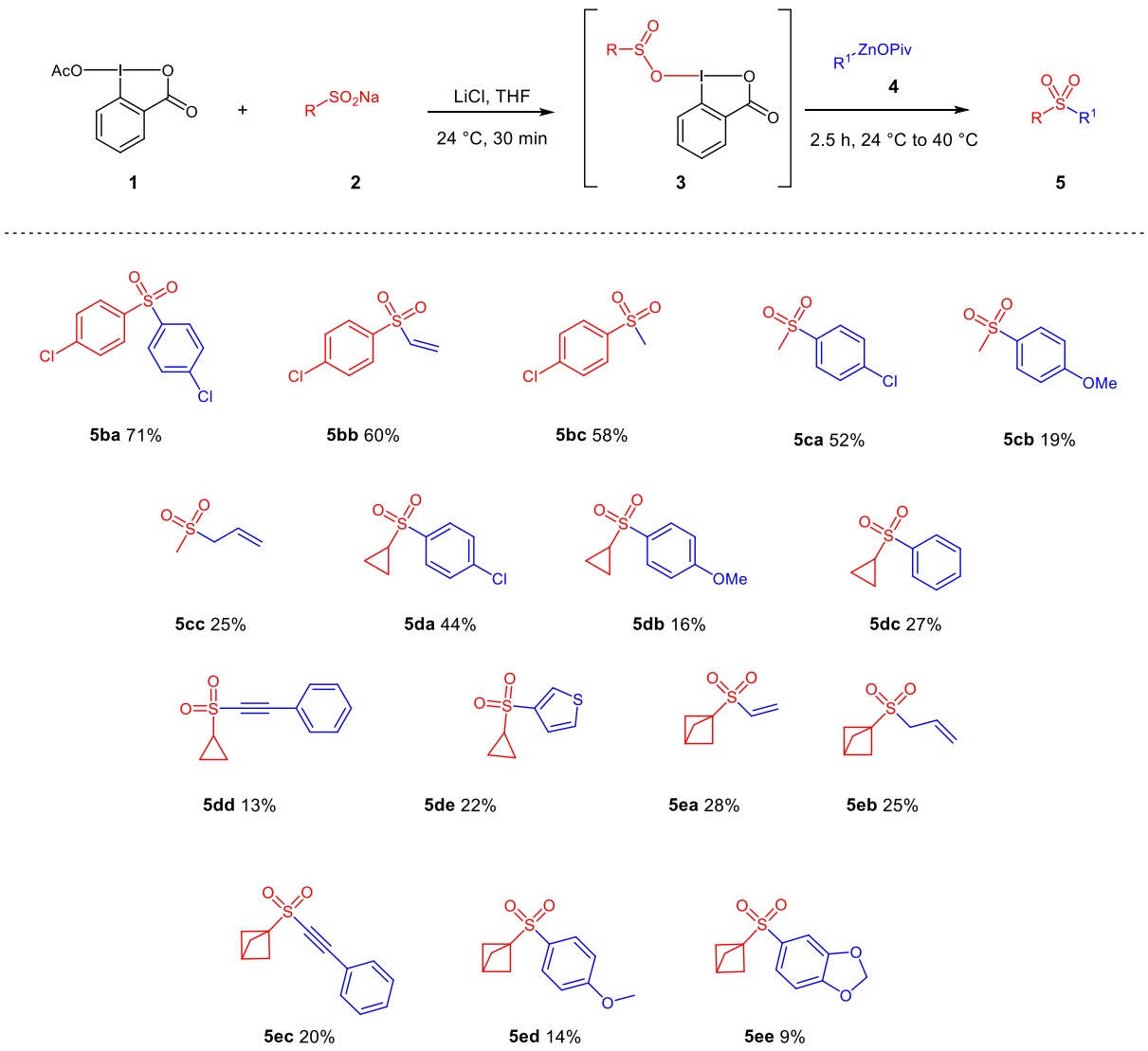
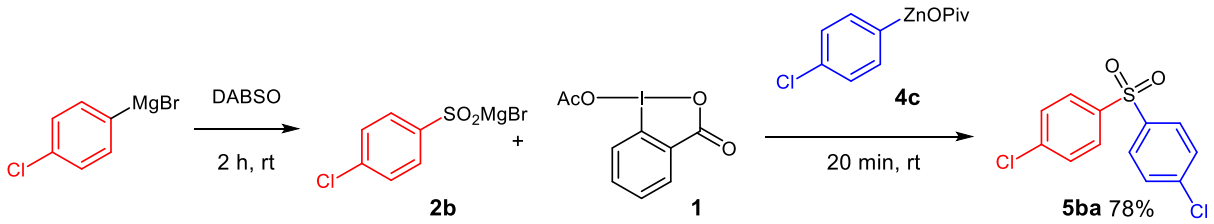


FIGURE 3 | Sulfinate salts for scope.

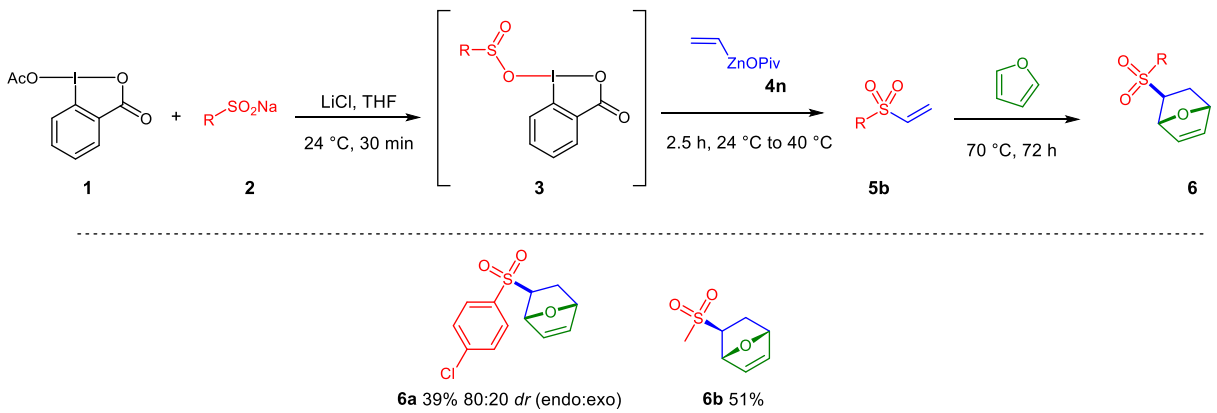
Application of the original conditions to alternative sulfinate salts resulted in prolonged reaction times at the previously optimized temperatures. To overcome this limitation and improve overall efficiency, the formation of the intermediate **3a** was conducted at room temperature. Remarkably, intermediates derived from sulfinate salts **2b–2e** were found considerably more stable than that generated from **2a** (intermediate **3a**), allowing the direct addition of the organozinc reagent immediately after intermediate formation, without the need for temperature adjustment. The modified conditions are summarized in Scheme 3. Under the modified conditions, a broad range of alkylic, aliphatic, vinylic, and aromatic sulfones was obtained in moderate to high yields. Among the sulfinate salts examined, the *p*-chlorosulfinate salt delivered the highest overall yields, reacting smoothly with aromatic, vinylic, and allylic zinc pivalates to afford the corresponding sulfones in 71%, 60%, and 58% yields (**5ba–5bc**), respectively. The methyl sulfinate salt exhibited its highest reactivity with 4-chlorophenylzinc pivalate, affording sulfone **5ca** in 52% yield. Particularly, methyl allyl sulfone (**5cc**) was isolated in 25% yield; this compound is of particular interest as a recently identified anti-inflammatory agent that additionally



SCHEME 3 | Scope of sulfones with various sulfinate salts.



SCHEME 4 | One-pot synthesis of sulfones.



SCHEME 5 | One-pot synthesis of sulfone and Diels-Alder cycloaddition.

reduces oxidative stress [39, 40]. Cyclopropylsulfinate salt **2d** reacted with alkynyl zinc pivalate to afford sulfone **5dd** in 13% yield. Interestingly, similar yields were obtained for both pyridyl and thiophene-derived organozinc pivalates, indicating an enhanced tolerance toward heterocyclic substrates for this of sulfinate class (Scheme 3). Under the optimized conditions, the bicyclo[1.1.1]pentane (BCP) sulfinate salt reacted with vinyl, alkynyl, and aryl zinc pivalates to give BCP-containing sulfones (**5ea–5ee**). Given the growing importance of BCP motifs as bioisosteres in medicinal chemistry, these products represent promising building blocks for the development of novel API candidates.

Owing to the formation of minimal side products and in the presence of LiCl, the reaction provides conditions that are particularly well-suited for one-pot transformations. This operational simplicity enables either sequential downstream functionalization or the direct synthesis of scaffold sulfones without intermediate purification. To showcase this capability, Michael Willis's protocol for the generation of sulfinate salts from Grignard reagents and DABSO was applied [27]. The resulting sulfinate salts were used directly in our reaction sequence: following their formation, acetoxybenziodoxole (**1**) was introduced, and after 30 min at room temperature, the desired zinc pivalate was added, affording the corresponding sulfones in yields of up to 78% (Scheme 4). As a representative example, sulfone **5ba**, a key intermediate in the synthesis of dapsone, was prepared using this one-pot protocol. Dapsone is a clinically important antibiotic widely employed in combination with rifampicin and clofazimine for the treatment of leprosy [4], underscoring the synthetic and medicinal relevance of this methodology.

The versatility of the methodology was further demonstrated through a one-pot follow-up Diels–Alder reaction. Upon direct addition of furan to the crude reaction mixture and subsequent hearing at 70°C for 72 h, the corresponding cycloadducts **6a** and **6b** were obtained in 39% and 51% yield, respectively. The reaction proceeded with a pronounce endo selectivity, affording an endo: exo ratio of 80:20 (Scheme 5).

3 | Conclusion

We report a practical and versatile method for sulfone synthesis using sulfonyl-containing hypervalent iodine reagents and organozinc pivalates. The method delivers diverse sulfones, including challenging bicyclo[1.1.1]pentane derivatives—in high yields under mild conditions with broad functional group tolerance. In situ sulfinate generation and one-pot transformations highlight the efficiency and modularity of the protocol. This strategy expands the scope of HIR-enabled umpolung chemistry, providing a robust platform for accessing synthetically and pharmaceutically valuable sulfones.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The Supporting Information covers detailed material on the conducted experiments and their results including the characterization of the obtained compounds. The data that support the findings of this study are available in the repository Chemotion (https://www.chemotion-repository.net/). All DOIs minted for the data are linked to the specific experiments in the Supporting Information and a summary of all new data obtained in this study can be gained with the collection DOI https://dx.doi.org/10.14272/collection/JFC_2025-05-09.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section. **Supporting Fig. S1:** Correlation between reagent concentration and reaction yield. **Supporting Table S1:** Concentration studies.