FULL PAPER

Synthesis of α-Sulfonyl Ketones through a Salicylic Acid-Catalyzed Multicomponent Reaction Involving Arylsulfonation and Oxidation

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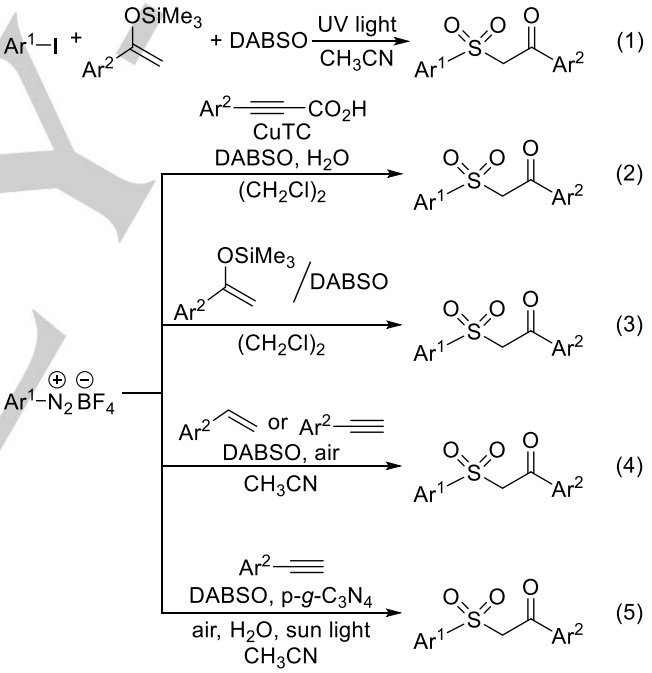
Abstract: The direct synthesis of α-sulfonyl ketones was accomplished by a multicomponent reaction of styrene derivatives, anilines, *t*-butyl nitrite, 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide (DABSO), and oxygen catalyzed by salicylic acid. The aryl radicals generated from aniline derivatives and *t*-butyl nitrite under the catalysis of salicylic acid was sulfonylated by DABSO to generate the arylsulfonyl radicals, which reacted further with styrenes, and then oxidized by oxygen to give the title compounds. Under the optimized conditions, the title compounds were obtained in good yields at ambient temperature within 1.5-2 h.

Introduction

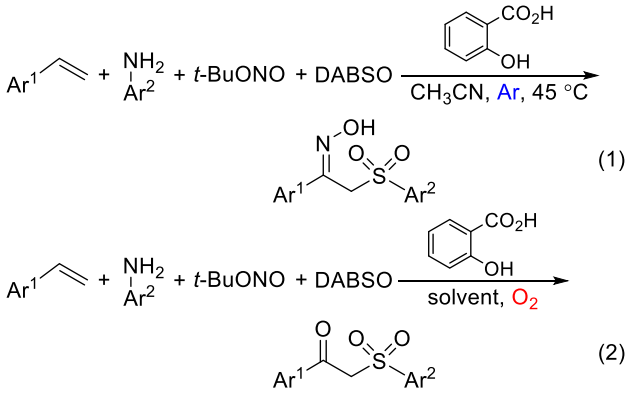
α-Sulfonyl ketones, or β-keto sulfones, are sulfone-containing compounds, and as such, some of these compounds exhibit interesting biological activities, such as antifungal[1] and anti-bacterial[2] activities. These compounds have also been used as the antagonists for bacterial quorum sensing[3] and the inhibitors of hydroxysteroid dehydrogenase type I.[4] In chemistry, α-sulfonyl ketones are versatile synthons in organic synthesis.[5-7] For examples, they have found applications in the synthesis of substituted alkenes,[8] alkynes,[9,10] and allenes,[11] 4*H*-pyrans,[12] β-hydroxysulfones,[13,14] vinylsulfones,[14] ketones,[15] and carboxylic acids.[16] Because of their broad utility in synthetic and medicinal chemistry, many synthetic methods have been developed for the preparation of α-sulfonyl ketones. Traditionally, α-sulfonyl ketones can be synthesized through the nucleophilic substitution of α-halo ketones[17,18] or α-tosyloxy ketones[19] with sodium sulfinates, or acyl chlorides with (methylsulfonyl)arenes in the presence of a strong base,[20] oxidation of the corresponding β-keto sulfide derivatives,[21] and the coupling between diazo sulfones and aldehydes.[22] Nonetheless, these methods demand the use of preformed substrates with special functional groups and/or drastic reaction conditions. In the last two decades, radical reactions were developed as new approaches for the synthesis of α-sulfonyl ketones. Most of these methods rely on the reaction of arylsulfonyl radicals with alkenes,[23-33[alkynes,[34-37] silyl enol ethers,[38] enol acetates,[39] enol phosphates,[40] enamides,[41] vinyl azide,[42-43] atropic acids,[44] or hydrazones.[45] As the source of the arylsulfonyl radicals, sulfonyl chlorides,[23-25, 34, 38-39] sodium sulfinates,[26-27,35-36,43,45] arylsulfinic acid,[28-29,37,40] sulfonyl hydrazides[30-32,41,44] or the combination of sulfonyl chlorides and hydrazine,[42] and *S*-phenyl benzenesulfonothioate[33] have been employed. Depending on the reagents used, the reactions can be either photocatalyzed,[23-24,29,34,39,44] metal-catalyzed,[27,32-33,35, 38,42,45] or completely metal-free.[25-26,28,30-31,36-37,40-41,43]



Scheme 1. Reported methods for the synthesis of α-sulfonyl ketones using DABSO as the sulfonylation reagent.

Through the initial work of Willis, Wu, and others,[46-51] 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide (DABSO)[52] has been demonstrated as a convenient and safe surrogate for sulfur dioxide in radical-mediated arylsulfonylation reactions.[46-48,53-58] In this regard, Wu and co-workers have developed several methods for the synthesis of α-sulfonyl ketones using DABSO as the sulfonylation reagent, starting from either the reaction of iodoarenes and silyl enol ethers under the activation of UV light (Scheme 1, Eq. 1),[59] the reaction of aryldiazonium salts and 3-arylpropiolic acids under copper catalysis (Scheme 1, Eq. 2),[60] or the reaction of aryldiazonium salts and silyl enol ethers directly

(Scheme 1, Eq. 3).[61] Most recently, Singh and co-workers reported the synthesis of α-sulfonyl ketones from the reaction of aryldiazonium salts, DABSO, and arylacetylenes or styrenes in the presence of air (Scheme 1, Eq. 4).[62] A similar synthesis using aryldiazonium salts, DABSO, arylacetylenes, oxygen, and water was also achieved by Niu and co-workers with carbon nitrides photocatalysis (Scheme 1, Eq. 5).[63]



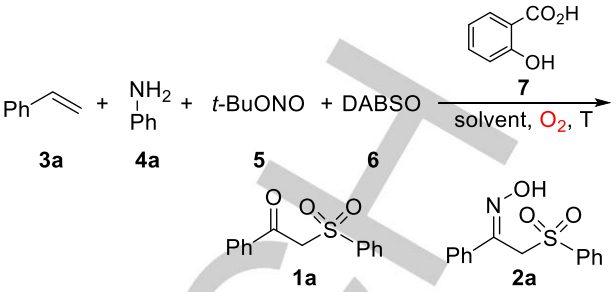
Scheme 2. Organocatalyzed synthesis of α-sulfonyl ketoximes (Eq. 1) and α-sulfonyl ketones (Eq. 2) using DABSO as the sulfonylation reagent.

As summarized above, while numerous methods have been developed for the synthesis of α-sulfonyl ketones, organocatalyzed synthetic methods are very rare. To the best of our knowledge, only tetra-*n*-butylammonium bromide[31] and tetra-*n*-butylammonium iodide[41] have been used as the catalysts together with TBHP (*tert*-butylhydroperoxide) and arylsulfonylhydrazides for the synthesis of these compounds. Moreover, an organocatalytic method that uses the readily available DABSO as the sulfonylation reagent is still lacking. Most recently, we reported the first organocatalytic synthesis of α-sulfonyl ketoximes using a four-component reaction involving DABSO (Scheme 2, Eq. 1),[64] which was developed on the basis of the salicylic acid-catalyzed aryl radical formation reaction from arylamines and *t*-butylnitrite reported originally by Gonzalez-Gomez and co-workers.[65-68] Inspired by the work of Singh and Niu,[62-63] we envisioned that this organocatalytic method could be modified for the synthesis of α-sulfonyl ketones if the reaction was conducted under an oxygen atmosphere (Scheme 2, Eq. 2). Herein we wish to disclose a novel method for the synthesis of these interesting compounds from readily available starting materials using an organocatalyzed multicomponent reaction.

Results and Discussion

Using styrene (**3a**), aniline (**4a**, 1.5 equiv.), *t*-butyl nitrite (**5**, 1.2 equiv.), and DABSO (**6**, 1.5 equiv.) as the model substrates, we first attempted the reaction in acetonitrile under oxygen with salicylic acid (**7**, 10 mol %) as the catalyst at room temperature. As shown in Table 1, 74% yield of the desired α-sulfonyl ketone product **1a** was obtained in 1.5 h (entry 1). In a control reaction without adding **7** as the catalyst, the yield of **1a** dropped dramatically to 21% (entry 2). In contrast, when the same reaction was carried out under argon, no formation of any product was observed, only a complex mixture was obtained (entry 3). These results clearly show salicylic acid **7** is indeed catalyzing this

Table 1. Optimization of the reaction conditions for the salicylic acid-catalyzed multicomponent reaction for the synthesis of **1a**[a]

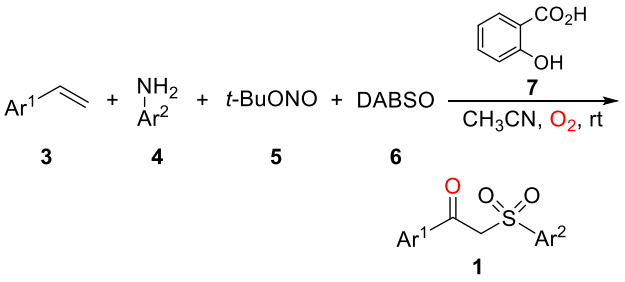


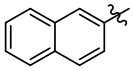
Entry	**5** (equiv.)	**6** (equiv.)	Time (h)	T (°C)	Solvent	Yield (%)[b]
1	1.2	1.5	1.5	rt	CH_3CN	74
2[c]	1.2	1.5	1.5	rt	CH_3CN	21
3[d]	1.2	1.5	1.5	rt	CH_3CN	--[e]
4	3.0	1.5	1.5	rt	CH_3CN	56[f]
5	1.0	1.5	1.5	rt	CH_3CN	69
6	1.2	1.0	1.5	rt	CH_3CN	66
7	1.2	2.0	1.5	rt	CH_3CN	58
8[g]	1.2	1.5	1.5	rt	CH_3CN	45
9[h]	1.2	1.5	1.5	rt	CH_3CN	74
10	1.2	1.5	3.0	rt	CH_3CN	69
11	1.2	1.5	1.5	rt	CH_2Cl_2	48
12	1.2	1.5	1.5	rt	$CHCl_3$	53
13	1.2	1.5	1.5	rt	CCl_4	20
14	1.2	1.5	1.5	rt	$(CH_2Cl)_2$	50
15	1.2	1.5	1.5	rt	Et_2O	23
16	1.2	1.5	1.5	rt	THF	68
17	1.2	1.5	1.5	rt	1,4-dioxane	46
18	1.2	1.5	1.5	rt	EtOAc	40
19	1.2	1.5	1.5	rt	toluene	63
20	1.2	1.5	1.5	45	CH_3CN	65
21	1.2	1.5	1.5	rt	CH_3CN	40
22[i]	1.2	1.5	1.5	rt	CH_3CN	28
23[j]	1.2	1.5	1.5	rt	CH_3CN	43
24[k]	1.2	1.5	1.5	rt	CH_3CN	10

[a] Unless otherwise indicated, all reactions were carried out using styrene (**3a**, 1.0 mmol), aniline (**4a**, 1.5 mmol), *t*-butyl nitrite (**5**, 1.2 mmol), DABSO (**6**, 1.5 mmol), and salicylic acid (**7**, 0.10 mmol, 10 mol %) in the specified solvent (5 mL) under oxygen at rt for 1.5 h. [b] Yield of the isolated product after column chromatography. [c] Reaction was performed without salicylic acid. [d] Reaction was conducted under argon. [e] A complex mixture was obtained. [f] α-Sulfonyl ketoxime **2a** was also obtained in 32% yield. [g] Reaction was performed with salicylic acid (**7**, 0.050 mmol, 5 mol %). [h] Reaction performed with salicylic

acid (**7**, 0.20 mmol, 20 mol %). [i] Reaction was performed with benzoic acid (0.10 mmol, 10 mol %) as the catalyst. [j] Reaction was performed with 2,5-dihydroxybenzoic acid (0.10 mmol, 10 mol %) as the catalyst. [k] Reaction was performed with anthranilic acid (0.10 mmol, 10 mol %) as the catalyst.

Table 2. Synthesis of α-sulfonyl ketones (**1**)[a]

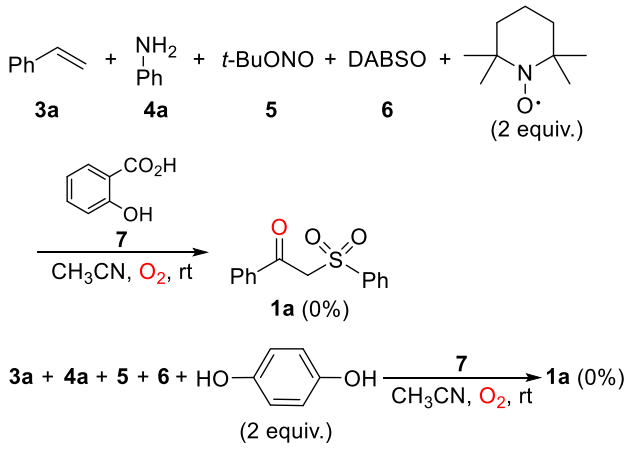


Entry	Ar^1	Ar^2	Time (h)	**1**/Yield (%)[b]
1	C_6H_5	C_6H_5	1.5	**1a**/74
2	C_6H_5	4-MeC_6H_4	1.5	**1b**/72
3	C_6H_5	4-$MeOC_6H_4$	2.0	**1c**/74
4	C_6H_5	4-$AcNHC_6H_4$	2.0	**1d**/66
5	C_6H_5	4-FC_6H_4	1.0	**1e**/66
6	C_6H_5	4-ClC_6H_4	1.0	**1f**/61
7	C_6H_5	4-BrC_6H_4	1.0	**1g**/62
8	C_6H_5	4-IC_6H_4	1.2	**1h**/68
9[c]	C_6H_5	4-CNC_6H_4	2.0	**1i**/61
10[c]	C_6H_5	4-$NO_2C_6H_4$	2.0	**1j**/58
11	C_6H_5	2-ClC_6H_4	1.0	**1k**/57
12	C_6H_5	3-ClC_6H_4	1.2	**1l**/68
13	C_6H_5	3-BrC_6H_4	1.2	**1m**/66
14	4-MeC_6H_4	C_6H_5	1.5	**1n**/66
15	4-$MeOC_6H_4$	C_6H_5	2.0	**1o**/52
16	4-ClC_6H_4	C_6H_5	1.0	**1p**/63
17	4-CNC_6H_4	C_6H_5	1.0	**1q**/54
18	2-$MeOC_6H_4$	C_6H_5	2.0	**1r**/28
19	3-$MeOC_6H_4$	C_6H_5	1.5	**1s**/63
20		C_6H_5	1.5	**1t**/70

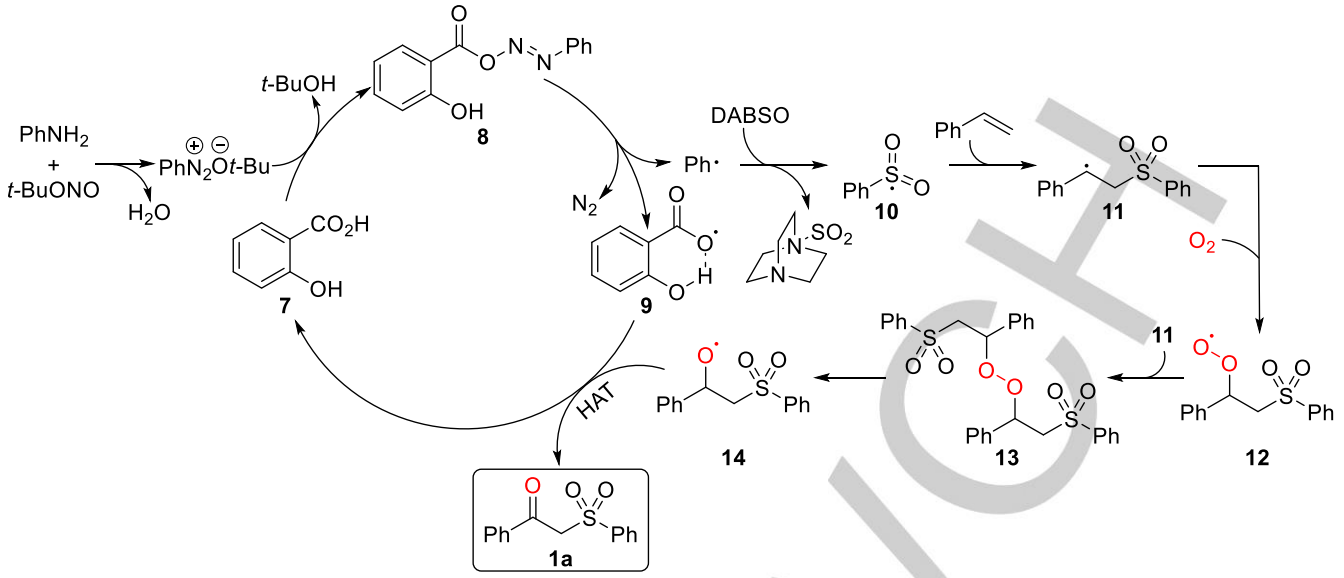
[a] Unless otherwise indicated, all reactions were carried out using **3** (1.0 mmol), **4** (1.5 mmol), **5** (1.2 mmol), **6** (1.5 mmol), and salicylic acid (**7**, 0.10 mmol, 10 mol %) in CH_3CN (5.0 mL) under oxygen at rt. [b] Yield of the isolated product after column chromatography. [c] The reaction was performed at 0 °C.

reaction and the presence of oxygen is crucial for the formation of **1a**. Next the reaction conditions were further optimized, as summarized in Table 1. A higher loading of *t*-butyl nitrite (**5**, 3.0 equiv.) led to a lower yield (56%) of the desired **1a** due to the formation of the oxime product **2a** (entry 4). On the other hand, a lower loading of **5** (1.0 equiv.) also led to a slightly lower yield of **1a** (69%, entry 5). Similarly, changing the loading of DABSO (**6**) was not helpful (entries 6-7), either. While reducing the catalyst loading to 5 mol % led to a reduced yield (45%, entry 8), increasing the catalyst loading to 20 mol % showed no improvement in the yield at all (entry 9). Prolonging the reaction time to 3 h also led to a slightly lower yield (entry 10). Next the solvent effects on this reaction were evaluated, and as the results in Table 1 show, all the many other solvents we screened are all inferior to CH_3CN in terms of the product yield (entries 11-19). Finally, the temperature effects on this reaction were studied, and it was found that lower yields were obtained when the reaction was carried out at both a slightly elevated temperature (45 °C, entry 20) and a subambient temperature (0 °C, entry 21). Lower product yields were also obtained when the reactions were conducted with benzoic acid (entry 22), 2,5-dihydroxybenzoic acid (entry 23), and anthranilic acid as the catalyst (entry 24). In summary, through these optimizations, the optimal reaction conditions identified for this reaction are those listed in entry 1.

Once the reaction conditions were optimized, the substrate scope of this multicomponent reaction was then studied, as summarized in Table 2. Besides aniline (entry 1), substituted anilines are also good substrates for this reaction (entries 2-13). In general, slightly lower yields were obtained for those substrates with an electron-withdrawing group in the *para*-position of the anilines (entries 2-10), which indicates that the electronic effects of the substituent have only minimal effects on this reaction. Similar yields were obtained for substrates with a substituent at the *meta*- or *ortho*-positions (entries 11-13 vs. entries 6-7), which indicates that there are no steric effects for the aniline substrate. In contrast, substituents on the styrene aromatic ring have much higher influence on this reaction (entries 14-19). It seems that both stronger electron-donating and electron-withdrawing groups are leading to lower yields (entries 15 and 17). Moreover, the position of the substituent on the phenyl ring also has major influences on this reaction (entries 15, 18, and 19): While both *para*- and *meta*-methoxy-substituted styrenes gave the same yield (entries 15 and 19), that of the *ortho*-methoxy-substituted styrene was much lower (entry 18), which is most likely due to the steric effects. Besides styrenes, 2-vinylnaphthalene is also a good substrate for this reaction, and the expected product **1t** was obtained in a good yield of 70% (entry 20).



Scheme 3. Radical inhibition experiments.



Scheme 4. Proposed mechanism for the salicylic acid-catalyzed multicomponent reaction.

According to the reported mechanisms of related reactions,[62-68] the current reaction should also proceed through a radical mechanism. To verify this, we conducted the radical inhibition experiments by adding a radical inhibitor (TEMPO or hydroquinone) intentionally to the reaction mixture of styrene (**3a**), aniline (**4a**), *t*-butyl nitrite (**5**), and DABSO (**6**) under the optimized reaction conditions (Scheme 3). As expected, no formation of the desired α-sulfonyl ketone **1a** was observed. These results support the involvement of radical intermediates in this multicomponent reaction. Based on these results and those reported mechanisms for the related reactions,[62,64-68] the following plausible mechanism is proposed for the current multicomponent reaction (Scheme 4). As shown in Scheme 4, aniline (**4a**) and *t*-butyl nitrite (**5**) first react with each other to from a diazonium salt, which in turn reacts with the catalyst salicylic acid (**7**) to give the diazo intermediate **8**. Intermediate **8** decomposes to produce the desired phenyl radical and the salicyloyl radical (**9**), which is stabilized by an intramolecular hydrogen-bond, via a homolytic cleavage that is facilitated by the formation of N_2.[68] The phenyl radical reacts with DABSO (**6**) to produce the phenylsulfonyl radical (**10**). The addition of radical **10** to styrene (**1a**) yields the alkyl radical **11**, which then reacts with oxygen to give the peroxy radical **12**. The reaction of **12** with radical **11** yields the peroxide compound **13**.[62] The homolytic cleavage of the peroxide bond in **13** gives the alkoxy radical **14**.[62] A hydrogen atom transfer (HAT) between the salicyloyl radical (**9**) and radical **14** gives the desired α-sulfonyl ketone product **1a** and completes the catalytic cycle of salicylic acid.

Conclusion

In summary, we have developed a novel organocatalytic method for the direct synthesis of α-sulfonyl ketones using salicylic acid as the catalyst. The multicomponent reaction of styrene derivatives, aniline derivatives, *t*-butyl nitrite, 1,4-diazabicyclo[2.2.2]octane-sulfur dioxide (DABSO), and oxygen yields the desired α-sulfonyl ketones in good yields within short reaction times under mild reaction conditions.

Supporting Information Summary

The general experimental procedure, the characterization data of the reaction products **1a-1t**, and their corresponding ^{1}H and ^{13}C NMR spectra were included in the Supporting Information.

Acknowledgements

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Keywords: multicomponent reaction • organocatalysis • radical reaction • salicylic acid • α-sulfonyl ketone

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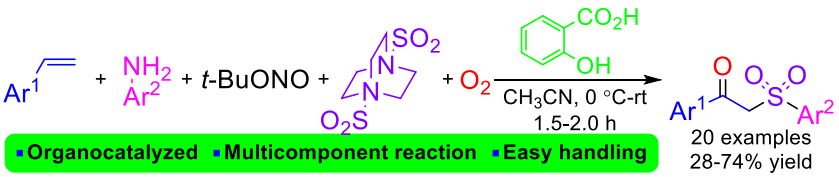

This article presents an organocatalyzed multicomponent reaction between styrene derivatives, anilines, t-butyl nitrite, DABSO, and oxygen at ambient temperature using salicylic acid as the catalyst. The aryl radicals generated from the reaction of aniline derivatives and *t*-butyl nitrite under the catalysis of salicylic acid are sulfonylated by DABSO, and the resulting arylsulfonyl radicals react further with styrenes and oxygen to give the corresponding α-sulfonyl ketones in good yields within 1.5 to 2 h.