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Photoinduced synthesis of 2-sulfonylacetonitriles with the insertion of sulfur dioxide under ultraviolet irradiation

Kaida Zhou,^a Jin-Biao Liu,^b Wenlin Xie,^c Shengqing Ye,*^a and Jie Wu*^{a,d}

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Metal-free insertion of sulfur dioxide with aryl iodides and 3-azido-2-methylbut-3-en-2-ol under ultraviolet irradiation at room temperature is achieved, giving rise to 2-(arylsulfonyl)acetonitriles in moderate to good yields. Alkyl iodide is also workable under the conditions. This transformation proceeds smoothly under mild conditions with broad substrate scope. Various functional groups are compatible including amino, ester, halo, and trifluoromethyl groups. No metal catalyst or additive is needed during the reaction process. Mechanistic studies show that under ultraviolet irradiation, aryl radical is generated in situ from aryl iodide, which undergoes subsequent sulfonylation via insertion of sulfur dioxide leading to arylsulfonyl radical intermediate. Then arylsulfonyl radical reacts with 3-azido-2-methylbut-3-en-2-ol giving rise to the corresponding 2-(arylsulfonyl)acetonitrile.

In the past few years, focus on the preparation of sulfonyl compounds has been centred on the insertion of sulfur dioxide by using the sulfur dioxide surrogates of DABCO (SO2)2 (1,4diazabicyclo[2.2.2]octane-sulfur dioxide)^{1,2} or potassium/sodium metabisulfite.³ Many approaches have been appeared in the presence of metal catalysis or under photoinduced conditions.⁴ Due to the importance of sulfonyl-containing drugs and related compounds,⁵ we are also involved in this area and have developed several strategies with the insertion of sulfur dioxide via radical process.⁶ For instance, we reported an example by using Katritzky salts as alkyl radical precursors with the insertion of sulfur dioxide under photoredox catalysis, leading to diverse dialkyl sulfones.^{6g} Recently, the photoinduced reactions of aryl iodides under visible light or ultraviolet irradiation have been developed rapidly.^{7,8} It is noteworthy that the cleavage of C-I bond can occur under catalystfree conditions via ultraviolet irradiation, with the generation of aryl radical intermediate. For example, arylboronic acids could be prepared through a catalyst- and additive-free, photoinduced borylation of haloarenes under ultraviolet irradiation.9a

2-(Arylsulfonyl)acetonitriles have a wide range of biological activities and are broadly used in the synthesis of pharmaceuticals natural products intermediates. For example, sulfanylacrylonitril derivatives are inhibitors of the diadenosine tetraphosphate phosphorylase Rv2613c of mycobacterium tuberculosis.¹⁰

Triazolothienopyrimidine UT-B inhibitors synthesized from 2-(arylsulfonyl)acetonitriles can selectively and reversibly inhibit urea transport and reduced urinary concentration in mice.¹¹ Additionally, arylsulfonylacetonitriles can be used to synthesize β -aminonitriles and β -keto sulfones, which are used to synthesize pesticides, food, medicine and feed additives.¹² Therefore, continuous efforts have been given for the generation of arylsulfonylacetonitriles. Traditional synthesis of arylsulfonylacetonitriles includes the use of toxic sulfonyl chlorides,¹³ sulfonic acids or salts,¹⁴ haloacetonitriles,¹⁵ or harsh reaction conditions that require additional catalysts, oxidants and additional reaction steps.¹⁶



 $\label{eq:Scheme 1. A proposed route to 2-(arylsulfonyl) acetonitriles through the insertion of sulfur dioxide under photoinduced metal-free conditions$

Prompted by the recent advance of photoinduced reactions of aryl halides under ultraviolet irradiation and the results of sulfur dioxide chemistry, we envisioned that the photoinduced insertion of sulfur dioxide into aryl iodides might be feasible for

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^{a.} School of Pharmaceutical and Materials Engineering & Institute for Advanced Studies, Taizhou University, 1139 Shifu Avenue, Taizhou 318000, China. Email: ysq0607@gmail.com; jie_wu@fudan.edu.cn

^{b.} School of Metallurgical and Chemical Engineering, Jiangxi University of Science and Technology, Ganzhou 341000, China

^c School of Chemistry and Chemical Engineering, Hunan University of Science and Technology, Xiangtan 411201, China

^{d.} State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 Lingling Road, Shanghai 200032, China

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the preparation of arylsulfonylacetonitriles under ultraviolet irradiation. А proposed synthetic route to arylsulfonylacetonitriles is shown in Scheme 1. 3-Azido-2methylbut-3-en-2-ol was used as the reaction partner, with an expectation to introduce cyano group in the molecule of arylsulfonylacetonitrile. We conceived that under ultraviolet irradiation, aryl radical would be generated from aryl iodide 1 in the absence of metal- and additive-free conditions. Subsequently, aryl radical would react with $DABCO \cdot (SO_2)_2$ to provide arylsulfonyl radical, with the release of DABCO. Then arylsulfonyl radical would attack the double bond of 3-azido-2methylbut-3-en-2-ol 2, leading to radical intermediate A. A molecule of nitrogen would be released form intermediate A giving rise to nitrogen radical intermediate B, which would undergo C-C bond cleavage to produce arylsulfonylacetonitrile 3. Meanwhile, radical C would be generated, which would combine with iodo radical. Finally, deprotonation by DABCO would afford acetone as byproduct.

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Table 1. Initial studies for the photoinduced synthesis of 2-(arylsulfonyl)acetonitrile with the insertion of sulfur dioxide under ultraviolet irradiation a

\square	/ + SO ₂	+OHN2, rt	/) p-	O_O_C N
1a		2		3a
Entry	Solvent	"SO ₂ "	Time	Yield (%) ^b
1	MeCN	DABCO·(SO ₂) ₂	12 h	30
2	1,4-dioxane	DABCO·(SO ₂) ₂	12 h	trace
3	THF	DABCO·(SO ₂) ₂	12 h	trace
4	DCE	DABCO·(SO ₂) ₂	12 h	6
5	MeOH	$DABCO \cdot (SO_2)_2$	12 h	20
6	MeCN	$K_2S_2O_5$	12 h	26
7	MeCN	$Na_2S_2O_5$	12 h	trace
8	MeCN	$K_2S_2O_5$ (3.0 equiv)	12 h	52
9	MeCN	DABCO· $(SO_2)_2$ (3.0 equiv)	12 h	59
10 ^c	MeCN	DABCO· $(SO_2)_2$ (3.0 equiv)	12 h	62
11 ^c	MeCN	DABCO· $(SO_2)_2$ (3.0 equiv)	24 h	73
12 ^{<i>d</i>}	MeCN	DABCO· $(SO_2)_2$ (3.0 equiv)	24 h	55

^{*a*} Reaction conditions: 1-iodo-4-methylbenzene **1a** (0.3 mmol), DABCO·(SO₂)₂ (0.2 mmol), 3-azido-2-methylbut-3-en-2-ol **2** (0.2 mmol), solvent (4.0 mL), N₂. ^{*b*} Isolated yield based on 3-azido-2-methylbut-3-en-2-ol **2**. ^{*c*} In the presence of 1-iodo-4-methylbenzene **1a** (0.4 mmol). ^{*d*} In the presence of 1-iodo-4-methylbenzene **1a** (0.5 mmol).

With the above consideration in mind, we thus started to explore the practicability of the hypothesis in Scheme 1. Initially, the reaction of 1-iodo-4-methylbenzene **1a**, DABCO· $(SO_2)_2$, and 3-azido-2-methylbut-3-en-2-ol **2** was selected for method development. At the outset, the reaction was performed in MeCN at room temperature under ultraviolet irradiation (600 W, Table 1, entry 1). To our delight, the desired 2-tosylacetonitrile product **3a** was obtained in 30% yield. Only a trace amount of product was detected when the reaction occurred in THF or 1,4-dioxane (Table 1, entries 2 and 3). The

yield was inferior when DCE or MeOH was used as the solvent (Table 1, entries 4 and 5). The result was slightly lower when the sulfur dioxide surrogate of DABCO (SO2)2 was changed to potassium metabisulfite (Table 1, entry 6). However, the reaction was hampered when sodium metabisulfite was used instead (Table 1, entry 7). Interestingly, the yield was increased to 52% when 3.0 equiv of potassium metabisulfite was employed (Table 1, entry 8). The result could be further improved by using 3.0 equiv of DABCO·(SO₂)₂ (Table 1, entry 9). The ratio of reaction partners was subsequently evaluated. A higher yield was afforded when 2.0 equiv of 1-iodo-4methylbenzene 1a was utilized (Table 1, entry 10). The corresponding 2-tosylacetonitrile 3a was produced in 73% yield when the reaction time was extended to 24 h (Table 1, entry 11). No better yield was observed when the amount of substrate 1a was increased (Table 1, entry 12).

Table 2. Scope exploration for the photoinduced synthesis of 2-(arylsulfonyl)acetonitriles with the insertion of sulfur dioxide under ultraviolet irradiation a



^a Isolated yield based on aryl iodide **1**.

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We next explored the scope generality of this method under the optimized conditions, and the result is shown in Table 2. A range of aryl iodides was examined, and it was found that all reactions proceeded smoothly giving rise to the desired products in moderate to good yields under ultraviolet irradiation. Additionally, this three-component reaction of aryl iodides 1, DABCO·(SO₂)₂, and 3-azido-2-methylbut-3-en-2-ol 2 showed good functional group tolerance, and many sensitive groups were compatible under the standard conditions. For example, the amino-substituted product 3m was afforded in 80 yield, and the ester-containing product 3I was produced in 77% yield. The ortho-substituted aryl iodides were workable as well, although the results were not as good as expected. For instance, ortho-trifluoromethyl substituted product 3u was generated in 61% yield, while 2-((2-(methylthio)phenyl)sulfonyl)acetonitrile 3q was provided in 67% yield. Additionally, the reaction was only effective for aryl iodides. Aryl bromides, chlorides and fluorides were inert under the conditions.



Scheme 2. Further exploration

We further explored the reaction of cyclohexyl iodide **4**, DABCO· $(SO_2)_2$ and 3-azido-2-methylbut-3-en-2-ol **2** under ultraviolet irradiation (600 W) at room temperature (Scheme 2). As expected, the corresponding product **3z** was produced in 40% yield. This result also demonstrated that alkyl iodide was good partner as well in this transformation.



Scheme 3. Investigation of the mechanism

To get more insight for the mechanism, several control experiments were performed (Scheme 3). It was shown that no reaction occurred in the absence of ultraviolet irradiation (Scheme 3, eqn 1). Since this route might be proceeded through a radical process as hypothesized in Scheme 1, we therefore investigated the reaction of 1-iodo-4-methylbenzene **1a**, DABCO·(SO₂)₂, and 3-azido-2-methylbut-3-en-2-ol **2** in the presence of 2.0 equivalents of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) under ultraviolet irradiation (Scheme 3, eqn 2). The outcome showed that no desired product was formed. In Scheme 1, we also proposed that C-C bond cleavage

would take place to produce arylsulfonylacetonitrile and acetone. Thus, we examined the Peaction $3^{\circ}07^{\circ}$ $1005^{\circ}4^{\circ}$ methylbenzene **1a**, DABCO·(SO₂)₂, and 2-azido-1,1-diphenylprop-2-en-1-ol **5**. Consequently, the desired product **3a** was observed and isolated in 57% yield, with the release of benzophenone. All these results demonstrated that the proposed route in Scheme 1 was reasonable.

In conclusion, we have reported a catalyst- and additive-free process for the synthesis of 2-(arylsulfonyl)acetonitriles from aryl iodides, 3-azido-2-methylbut-3-en-2-ol, and sulfur dioxide under ultraviolet irradiation at room temperature. This transformation proceeds smoothly under mild conditions with substrate broad scope. giving rise to 2-(arylsulfonyl)acetonitriles in moderate to good yields. Various functional groups including amino, ester, halo, and trifluoromethyl groups are compatible under the conditions. Alkyl iodide is also workable under the conditions. Mechanistic studies show that under ultraviolet irradiation, aryl radical is generated in situ from aryl iodide, which undergoes subsequent sulfonylation via insertion of sulfur dioxide leading to arylsulfonyl radical intermediate. Then arylsulfonyl radical reacts with 3-azido-2-methylbut-3-en-2-ol giving rise to the corresponding 2-(arylsulfonyl)acetonitrile.

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Conflicts of interest

There are no conflicts to declare.

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