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Photoredox-Catalyzed Sulfonylation of *O*-Acyl Oximes via Iminyl Radicals with the Insertion of Sulfur Dioxide

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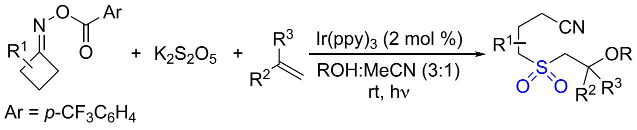
Supporting Information

ABSTRACT: A multicomponent sulfonylation of *O*-acyl oximes via iminyl radicals with the insertion of sulfur dioxide under photoredox catalysis is achieved. This multicomponent reaction of *O*-acyl oximes, potassium metabisulfite, alkenes, and nucleophiles under visible-light irradiation is efficient, giving rise to a range of sulfones in moderate to good yields. A broad reaction scope is presented with good functional group compatibility.



- iminyl radical initiated transformation
- insertion of sulfur dioxide
- photoinduced multicomponent reaction

Currently, sulfonylation with the insertion of sulfur dioxide via a radical process under mild conditions has attracted continuous interest.[1,2] Many approaches have appeared that use the sulfur dioxide surrogates of DABCO·$(SO_2)_2$[3] and inorganic sulfites[4] instead of gaseous sulfur dioxide. Different radical precursors including aryldiazonium tetrafluoroborates, aryl halides, diaryliodonium salts, and potassium alkyltrifluoroborates have been reported for the generation of carbon radicals, which would initiate the reaction with the capture of sulfur dioxide to produce sulfonyl radical intermediates. Thus, diverse sulfonyl compounds can be efficiently prepared with the insertion of sulfur dioxide, especially from the cheap and easily available inorganic sulfites.

The core *β*-substituted sulfone such as *β*-alkoxy sulfone can be found in many biologically active molecules.[5] In general, these sulfones are synthesized through the oxidation of *β*-alkoxy sulfides or the alkoxylation of *α*,*β*-unsaturated sulfones.[6] However, a stoichiometric amount of exogenous additives/oxidants is required, which limits their applicability in organic synthesis. Difunctionalization of alkenes through sulfonylation for the preparation of *β*-substituted sulfones provides an alternative route. For example, Lei and co-workers reported the synthesis of *β*-alkoxy sulfones through an electrochemical oxidative alkoxysulfonylation of alkenes starting from sulfonyl hydrazines and alcohols.[7] In their approaches, the preinstalled sulfonyl compounds or odorous thiol substrates had to be utilized.[8] As part of our continuous interest in the sulfonylation reaction with the insertion of sulfur dioxide, we considered that the preparation of *β*-substituted sulfones from the sulfur dioxide surrogate of inorganic sulfite would be ideal.

In the past few years, reactions of iminyl radicals have attracted much attention.[9,10] For instance, Yu and co-workers described the intermolecular remote C(sp^3)−H and C−C vinylation of *O*-acyl oximes with vinyl boronic acids via iminyl radicals.[10a] The iminyl radicals were generated via a photoreductive strategy from *O*-acyl oximes under irradiation by visible light. Guo and co-workers reported the synthesis of cyanoalkylated heteroarenes through an iron-catalyzed C−H cyanoalkylation of heteroarenes via C−C bond cleavage of cyclobutanone oxime esters.[10b] Inspired by these results and recent advances with the insertion of sulfur dioxide,[1] we envisioned that sulfur dioxide might react with iminyl radicals under photoredox catalysis as well.

We hypothesized that a multicomponent sulfonylation of *O*-acyl oximes via iminyl radicals with the insertion of sulfur dioxide under photoredox catalysis would be feasible for the synthesis of *β*-substituted sulfones. This proposed multicomponent reaction of *O*-acyl oximes **1**, sulfur dioxide, alkenes **2**, and nucleophiles under visible-light irradiation is presented in Scheme 1. We reasoned that in the presence of photocatalyst under visible-light irradiation *O*-acyl oxime **1** would undergo the N−O bond dissociation to provide iminyl radical intermediate **A**, which would go through the intramolecular C−C bond cleavage leading to a carbon radical **B**. This carbon radical **B** would be trapped by sulfur dioxide to produce sulfonyl radical **C**, which would then attack the double bond of alkene **2** to afford another carbon radical intermediate **D**. A subsequent oxidative single-electron transfer (SET) by the oxidized form of photocatalyst would give rise to a cation intermediate **E**. In the presence of base, the nucleophile would be involved and react with cation **E** leading to the corresponding product **3**. With this consideration in mind, we thus started to explore the practicability of this transformation.

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Scheme 1. Proposed Multicomponent Reaction of *O*-Acyl Oximes, Sulfur Dioxide, Alkenes, and Nucleophiles under Visible-Light Irradiation

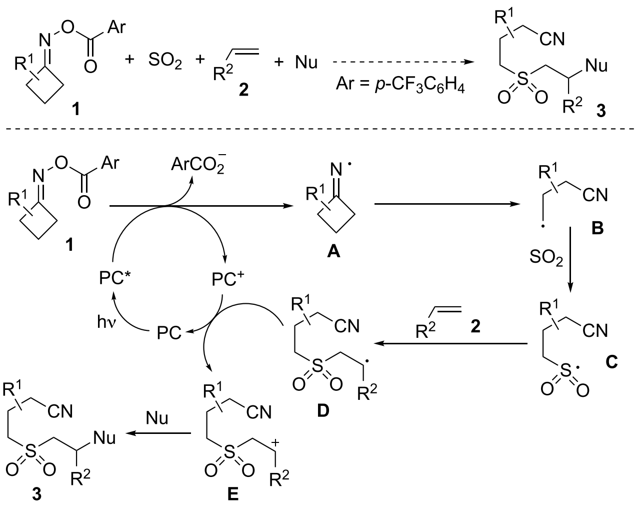
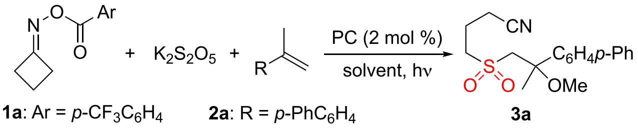


Table 1. Initial Studies for the Reaction of Cyclobutanone *O*-(4-(Trifluoromethyl)benzoyl) Oxime 1a, Potassium Metabisulfite, 4-(Prop-1-en-2-yl)-1,1′-biphenyl 2a, and Methanol under Visible-Light Irradiation[a]



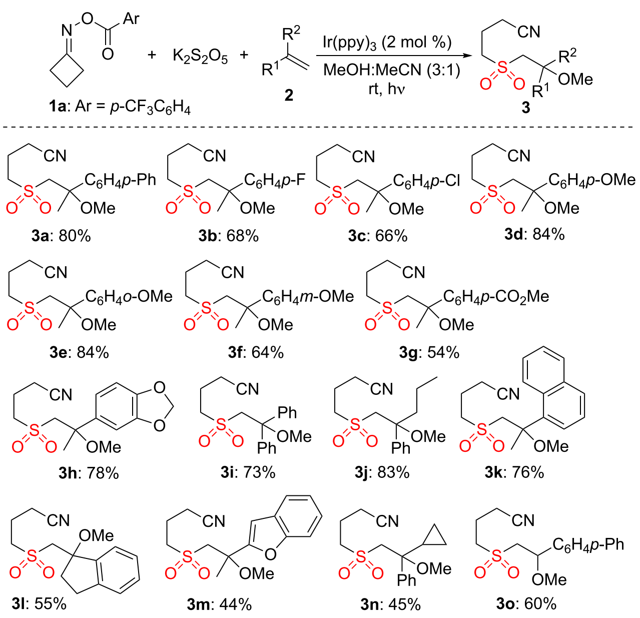
entry	PC	solvent	light	yield[b] (%)
1	$Ru(bpy)_3Cl_2 \cdot 6H_2O$	MeOH	35 W white	nr
2	Eosin Y	MeOH	35 W white	nr
3	Fluorescein	MeOH	35 W white	trace
4	$Ir(ppy)_3$	MeOH	35 W white	42
5	$Ir(ppy)_3$	MeOH	15 W blue	50
6	$Ir(ppy)_3$	MeOH/MeCN (3:1)	15 W blue	80
7	$Ir(ppy)_3$	MeOH/DCE (3:1)	15 W blue	66
8	$Ir(ppy)_3$	MeOH/PhMe (3:1)	15 W blue	66
9	$Ir(ppy)_3$	MeOH/1,4-dioxane (3:1)	15 W blue	65
10[c]	$Ir(ppy)_3$	MeCN	15 W blue	29

[a] Reaction conditions: cyclobutanone *O*-(4-(trifluoromethyl)benzoyl) oxime **1a** (0.2 mmol), $K_2S_2O_5$ (0.22 mmol), 4-(prop-1-en-2-yl)-1,1′-biphenyl **2a** (0.3 mmol), solvent (2.0 mL), N_2, 20 h. [b] Isolated yield based on cyclobutanone *O*-(4-(trifluoromethyl)benzoyl) oxime **1a**. [c] In the presence of MeOH (10 equiv).

Initial studies were carried out with the reaction of cyclobutanone *O*-(4-(trifluoromethyl)benzoyl) oxime **1a**, potassium metabisulfite, 4-(prop-1-en-2-yl)-1,1′-biphenyl **2a**, and methanol under visible-light irradiation (Table 1). At the outset, the reaction was screened with several photocatalysts. No reaction occurred in the presence of $Ru(bpy)_3Cl_2 \cdot 6H_2O$ or eosin Y under visible-light irradiation (Table 1, entries 1 and 2). Only a trace amount of product was observed when fluorescein was used as a replacement (Table 1, entry 3). Gratifyingly, the reaction provided the desired product **3a** in 42% yield when $Ir(ppy)_3$ was utilized as the photocatalyst (Table 1, entry 4). The yield was higher when the light source (35 W white LED) was changed to 15 W blue LED (Table 1, entry 5). The result could be improved when the reaction was performed in a mixed solvent of MeOH/MeCN, leading to the corresponding product **3a** in 80% yield (Table 1, entry 6). The yields were inferior when other solvents were used (Table 1, entries 7−9). The yield was reduced to 29% with 10 equiv of methanol. No further improvement could be obtained when the amount of reactants was changed (data not shown in Table 1).

With the above optimized conditions in hand, we started to explore the substrate scope for the multicomponent reaction of *O*-acyl oxime **1a**, potassium metabisulfite, alkenes **2**, and methanol under visible light irradiation. The result is summarized in Scheme 2. A wide range of alkenes could be employed in the reaction under the standard conditions. This transformation was highly effective, and diverse *β*-methoxy sulfones were generated in moderate to good yields. Additionally, different functionalities were tolerated. For instance, the ester-containing product **3g** was produced in 54% yield. The benzofuranyl-substituted compound **3m** was obtained in 44% yield. The structure of compound **3i** was confirmed by the X-ray diffraction analysis (see the Supporting Information), which showed the excellent chemoselectivity during the reaction process.

Scheme 2. Scope Exploration for the Reaction of *O*-Acyl Oxime 1a, Potassium Metabisulfite, Alkenes 2, and Methanol[a]



[a] Reaction conditions: cyclobutanone *O*-(4-(trifluoromethyl)benzoyl) oxime **1a** (0.2 mmol), $K_2S_2O_5$ (0.22 mmol), alkene **2** (0.3 mmol), MeOH/MeCN (v/v = 3:1, 2 mL), N_2. [b] Isolated yield based on cyclobutanone *O*-(4-(trifluoromethyl)benzoyl) oxime **1a**.

Subsequently, a range of *O*-acyl oximes **1** was examined in the reaction of potassium metabisulfite, 4-(prop-1-en-2-yl)-1,1′-biphenyl **2a**, and methanol under the standard conditions (Scheme 3). As expected, all reactions worked well to provide the corresponding products in good yields. The bromo-, ester-, and nitrogen-containing heterocycle were all compatible during the transformation.

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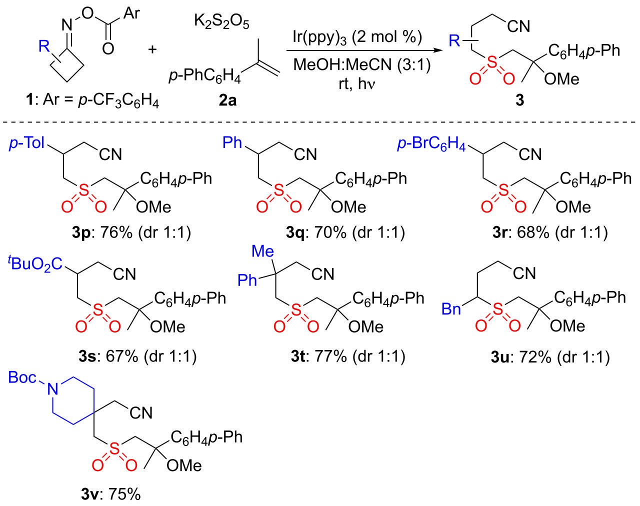
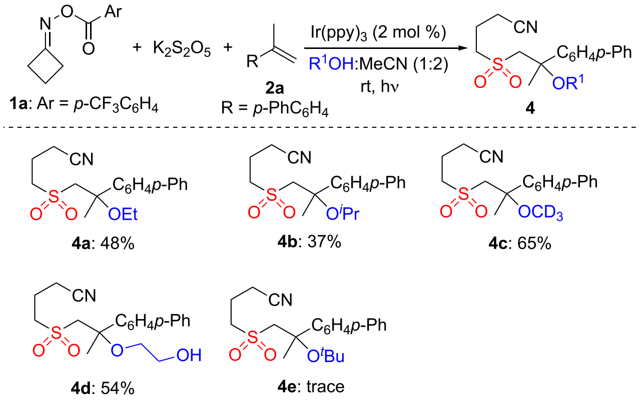
Scheme 3. Reaction of *O*-Acyl Oximes 1, Potassium Metabisulfite, Alkene 2a, and Methanol[a]



[a]Reaction conditions: *O*-acyl oxime **1** (0.2 mmol), $K_2S_2O_5$ (0.22 mmol), alkene **2a** (0.3 mmol), MeOH/MeCN (v/v = 3:1, 2.0 mL), N_2. [b]Isolated yield based on cyclobutanone *O*-acyl oxime **1**.

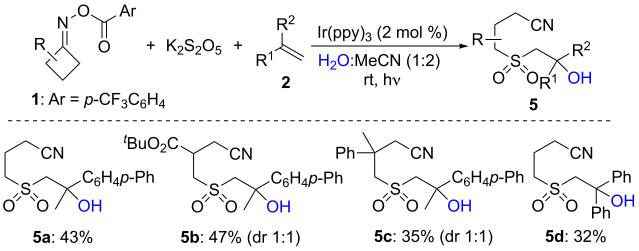
This method was further extended to other alcohols (Scheme 4). When methanol was replaced by ethanol or ethane-1,2-diol in the reaction of oxime **1a**, the corresponding compounds **4a** and **4d** were afforded in moderate yields. We also examined the reactions of secondary alcohol and tertiary alcohol. For example, compound **4b** was obtained in 37% yield when propan-2-ol was used as the nucleophilic component. However, only a trace amount of product was detected with 2-methylpropan-2-ol as the solvent. The results showed that secondary and tertiary alcohols were not good solvents in this transformation. The reaction in methan-d_3-ol-d instead of methanol gave rise to the desired product **4c** in 65% yield. Additionally, β-hydroxy sulfones **5** were generated in 32−47% yields when the reactions were performed in a mixture of water and MeCN (Scheme 5).

Scheme 4. Reaction of *O*-Acyl Oximes 1a, Potassium Metabisulfite, Alkene 2a, and Other Alcohols[a]



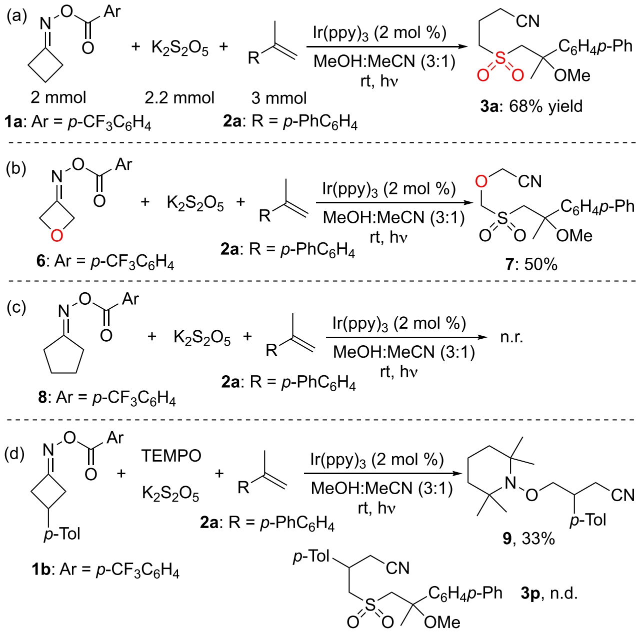
[a]Reaction conditions: *O*-acyl oxime **1a** (0.2 mmol), $K_2S_2O_5$ (0.22 mmol), alkene **2a** (0.3 mmol), R^1OH/MeCN (v/v = 3:1, 2.0 mL), N_2. [b]Isolated yield based on cyclobutanone *O*-acyl oxime **1a**.

Scheme 5. Reaction of *O*-Acyl Oximes 1, Potassium Metabisulfite, Alkenes 2, and H_2O[a]



[a]Reaction conditions: *O*-acyl oxime **1** (0.2 mmol), $K_2S_2O_5$ (0.22 mmol), alkene **2** (0.3 mmol), H_2O/MeCN (v/v = 1:2, 3.0 mL), N_2. [b]Isolated yield based on cyclobutanone *O*-acyl oxime **1**.

Scheme 6. Further Exploration



Further exploration showed that a gram-scale reaction for the synthesis of compound **3a** worked effectively to generate the corresponding product in 68% yield (Scheme 6, eq a). It was also found that the oxygen-containing substrate **6** was suitable under the conditions, which gave rise to the desired product **7** in 50% yield (Scheme 6, eq b). However, no reaction occurred when *O*-acyl oxime **8** was employed in the reaction (Scheme 6, eq c). To gain more insight of the mechanism, 2.0 equiv of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) was added in the reaction of *O*-acyl oxime **1b**, potassium metabisulfite, and 4-(prop-1-en-2-yl)-1,1′-biphenyl **2a** under the standard conditions (Scheme 6, eq d). The result showed that no desired product **3p** was detected. Instead, compound **9** was obtained in 33% yield. This outcome demonstrated that a radical process might be involved. Additionally, the quantum yield for the reaction of *O*-acyl oxime **1a**, $K_2S_2O_5$, and alkene **2a** under the standard condition was determined as 20%, which demonstrated it was a photoredox-catalyzed pathway (see the Supporting Information). These results were consistent with the hypothesis shown in Scheme 1. Furthermore, the *p*-CF_3-benzoyl part in oxime would undergo cleavage via a SET process in the presence of $Ir(ppy)_3$, which would combine with H^+ from nucleophile later affording the corresponding benzoic acid. The formation of

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benzoic acid could be observed in the reaction system and confirmed by ^{19}F NMR.

In conclusion, we have described a photoredox-catalyzed multicomponent sulfonylation of *O*-acyl oximes via iminyl radicals with the insertion of sulfur dioxide. The commercially available potassium metabisulfite as the source of sulfur dioxide works efficiently in this transformation. Not only alcohols but also water can be used as nucleophiles leading to a range of *β*-alkoxy sulfones and *β*-hydroxyl sulfones with good functional group compatibility. A plausible mechanism involving radical process is proposed, which is initiated by the iminyl radical formed in situ from *O*-acyl oxime under irradiation by visible light. The further intramolecular rearrangement with the cleavage of the C−C bond generates a carbon radical, which subsequently undergoes sulfonylation, oxidative single-electron transfer, and nucleophilic attack to produce the final product.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b01323.

Experimental procedures, characterization data, ^{1}H and ^{13}C NMR spectra of products (PDF)

Accession Codes

CCDC 1898774 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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