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Synthesis of Furoxans and Isoxazoles via Divergent [2 + 1 + 1 + 1] Annulations of Sulfoxonium Ylides and ^{t}BuONO

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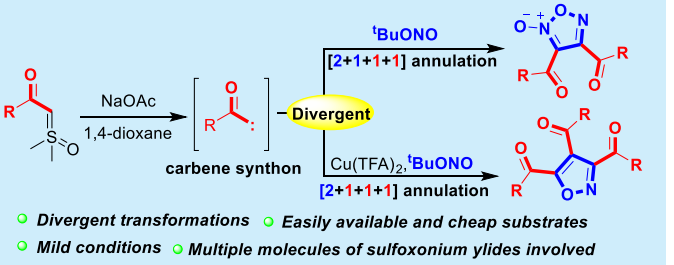
Supporting Information

ABSTRACT: We have presented a simple and new method for the divergent assembly of furoxans and isoxazoles in which the [2 + 1 + 1 + 1] annulation reaction of sulfoxonium ylides is reported for the first time. When the reaction was performed using ^{t}BuONO as the nitrogen source without metal catalyst, the desired furoxans were obtained in decent yields with wide substrate scope. Isoxazoles bearing three carbonyl groups were achieved when the reaction was conducted using $Cu(TFA)_2$ as catalyst.



Heterocyclic compounds are ubiquitously embedded in many pharmaceuticals, materials, and bioactive molecules. Among myriad heterocycles, furoxans and isoxazoles are two subclasses of privileged pharmacophores (Figure 1).

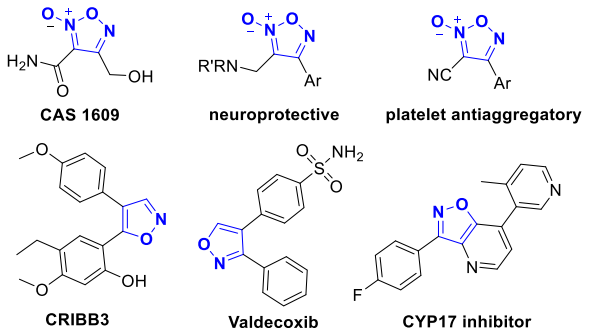


Figure 1. Examples of biologically active molecules containing furoxan and isoxazole rings.

Furoxan derivatives have loomed as important NO donors[1] and feature bioactivities such as anticancer,[2] antibacterial,[3] anti-inflammatory,[4] and so on.[5] In addition, a round of promising furoxan-based derivatives has been investigated as the core framework of melt-cast eutectic materials and blasting materials.[6,7] As for isoxazoles, the isoxazole ring ranks 33rd among the 351 ring systems in commercially available drugs,[8] which unarguably represents one of the most important skeletons in pharmaceuticals and agrochemicals. Given the particular physiological activity of furoxans and isoxazoles, the development of simple and mild routes to assemble the valuable furoxans and isoxazoles is of great interest in organic chemistry and pharmaceutical chemistry.

Since sulfoxonium ylides could serve as carbene surrogates of diazo compounds, considerable growth in the field of sulfoxonium ylides has been witnessed. In the past years, sulfoxonium ylides have been proven to be versatile building blocks in organic synthesis to forge intricate molecules (Scheme 1a).[9] In particular, sulfoxonium ylides were prominent coupling partners in transition-metal-catalyzed C−H activation in which diverse ketone-functionalized (hetero)-arenes and heterocycles were achieved varied from different directing groups.[10] Given the bipolarity of sulfoxonium ylides, they could also be utilized as ketone precursors to afford various ketones.[11] Aside from these transformations, the C−H cross-coupling of sulfoxonium ylides and the application of sulfoxonium ylides as weak directing group for the synthesis of substituted sulfoxonium ylides were also sporadically reported.[12] Despite this undisputed advance of sulfoxonium ylides, only one molecule of sulfoxonium ylide was involved in most of these excellent transformations. To our knowledge, the transformation involving multiple molecules of sulfoxonium ylides has yet to be explored, and the [2 + 1 + 1 + 1] annulation of sulfoxonium ylides has never been documented so far.

^{t}BuONO has emerged as a brilliant nitrogen source to streamline synthesis of N-heterocycles in synthetic chemistry.[13] As part of our ongoing interest in ^{t}BuONO[14] and carbene chemistry,[15] herein we report a novel [2 + 1 + 1 + 1]

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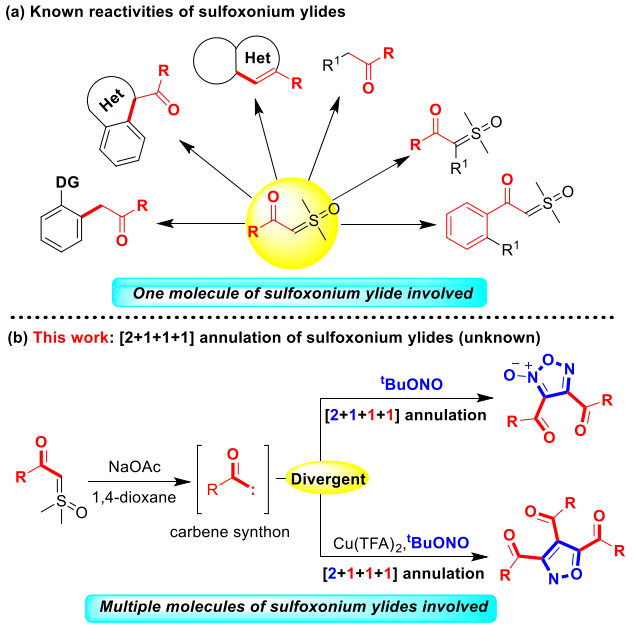
Scheme 1. Reactions of sulfoxonium ylides

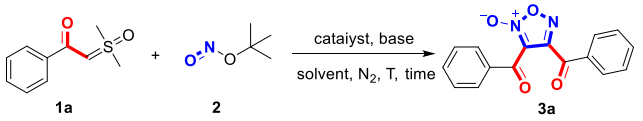


annulation reaction involved of multiple molecules of sulfoxonium ylides for the divergent synthesis of furoxans and isoxazoles using sulfoxonium ylides as carbene precursors and ^{t}BuONO as the nitrogen source (Scheme 1b).

To commence our study, sulfoxonium ylide **1a** and ^{t}BuONO (**2**) were selected as benchmark substrates to test our assumption. When the reaction was performed in the presence of CuI and NaOAc in dioxane under nitrogen atmosphere at 80 °C for 12 h, the isolated yield of the furoxan **3a** was found to be 84% (Table 1, entry 1). We also screened some other metals. The yields of furoxan **3a** using $Cu(OTf)_2$, $Pd(OAc)_2$, and $FeCl_3$ as catalysts were 83%, 85%, and 82%, respectively (Table 1, entries 2−4). Since the yields of **3a** when different metals were used were similar, we considered whether the metal had any effect on this transformation. To our surprise and delight, when the reaction was conducted without the metal, **3a** could be achieved with 86% isolated yield as well (Table 1, entry 5), which indicated that metal was unnecessary for the annulation reaction to provide furoxan. Subsequently, a number of bases were examined. However, the tested bases, such as K_2CO_3, DABCO, and DBU all failed to promote this transformation (Table 1, entries 5−8). We also inspected a range of solvents (Table 1, entries 9−12); only marginal improvements were acquired, and it was proven that dioxane was the best reaction medium to deliver the furoxan **3a**. As a further investigation, we screened the effect of reaction temperature for this annulation reaction (Table 1, entries 13−16). It was found that 40 °C was the best reaction temperature, affording the targeted furoxan **3a** in 90% yield (Table 1, entry 15). The attempt to shorten the reaction time was triumphant (Table 1 entries 17−20), and the desired furoxan **3a** could be readily obtained in high yield when we shortened the reaction time to 2 h.

After the optimal reaction conditions were determined, the substrate scope for the assembly of furoxans **3** was then explored. As showcased in Scheme 2, a sequence of sulfoxonium ylides having different electronic properties were proven to be good carbene precursors, enabling the production of furoxans **3a**−**3f** in 54−92% yields. The structure of **3c** was

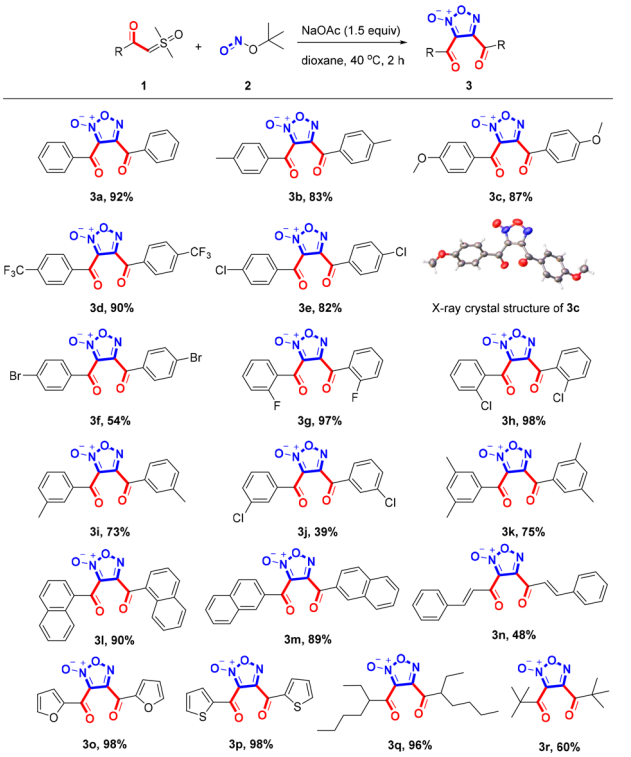
Table 1. Optimization of the Reaction Conditions[a]



entry	catalyst	base	solvent	T (°C)	yield (%)
1	CuI	NaOAc	dioxane	80	84
2	$Cu(OTf)_2$	NaOAc	dioxane	80	83
3	$Pd(OAc)_2$	NaOAc	dioxane	80	85
4	$FeCl_3$	NaOAc	dioxane	80	82
5		NaOAc	dioxane	80	86
6		K_2CO_3	dioxane	80	NR
7		DABCO	dioxane	80	NR
8		DBU	dioxane	80	NR
9		NaOAc	Toluene	80	59
10		NaOAc	CH_3CN	80	39
11		NaOAc	DCE	80	42
12		NaOAc	DMF	80	NR
13		NaOAc	dioxane	100	84
14		NaOAc	dioxane	60	82
15		NaOAc	dioxane	40	90
16		NaOAc	dioxane	rt	73
17[b]		NaOAc	dioxane	40	86
18[c]		NaOAc	dioxane	40	88
19[d]		NaOAc	dioxane	40	90
20[e]		NaOAc	dioxane	40	69

[a]Reaction conditions: **1** (0.3 mmol), **2** (1.5 mmol), and NaOAc (0.45 mmol) were stirred in dioxane (2 mL) at 40 °C for 12 h under N_2. Isolated yields are given. [b]6 h. [c]4 h. [d]2 h. [e]1 h.

Scheme 2. Scope for the Synthesis of Furoxans



assuredly confirmed by X-ray single-crystal diffraction. In addition to *para*-substituted aryl sulfoxonium ylides, *ortho*- and *meta*-substituted aryl sulfoxonium ylides were well compatible in this transformation as well, delivering the targeted furoxans

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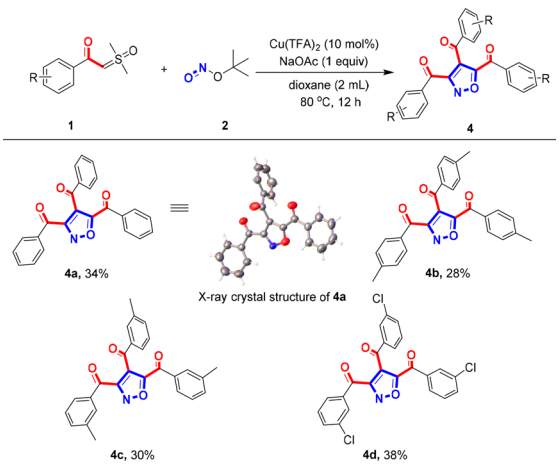
3g−**3k** in decent yields. The sterically demanding naphthalenyl sulfoxonium ylides **1l** and **1m** were also good candidates in this reaction, and the corresponding furoxans (**3l** and **3m**) could be isolated in 90% and 89% yield, respectively. To our delight, alkenyl-derived sulfoxonium ylide **1n** was amenable to this [2 + 1 + 1 + 1] annulation as well, leading to the formation of **3n** in 48% yield. The treatment of heterocyclic sulfoxonium ylides (**1o** and **1p**) with TBN was also successful, in which the desired furoxans **3o** and **3p** were achieved in high yield of 98%. Aside from the aromatic sulfoxonium ylides, aliphatic sulfoxonium ylides were also found to be suitable carbene donors in this reaction, furnishing the expected furoxans **3q** and **3r** in 96% and 60% yields, respectively.

When we screened the metal catalysts for the construction of furoxans (Table 1, entries 1−4), a low yield of isoxazole **4a** was observed. Subsequently, we turned our attention to optimize the reaction conditions for the synthesis of isoxazoles, which are summarized in Table S6−S10. When 0.6 mmol of sulfoxonium ylide **1a** was added, the isoxazole **4a** could be isolated in 34% yield using $Cu(TFA)_2{\cdot}H_2O$ as catalyst and NaOAc as base. To enhance the yield of isoxazole **4a**, we also investigated the effect of other reaction parameters on this transformation (see the SI for details). Disappointingly, only marginal improvements were obtained (Tables S6−S10). Given the multimolecular competitive reactions, it might be challenging to obtain high yield of isoxazole **4a** via this novel [2 + 1+1 + 1] annulation. Therefore, we determined that 34% yield was the optimal reaction yield.

Under the optimal reaction conditions for the assembly of isoxazoles, we also inspected the substrate range of isoxazoles. As demonstrated in Scheme 3, a number of sulfoxonium ylides

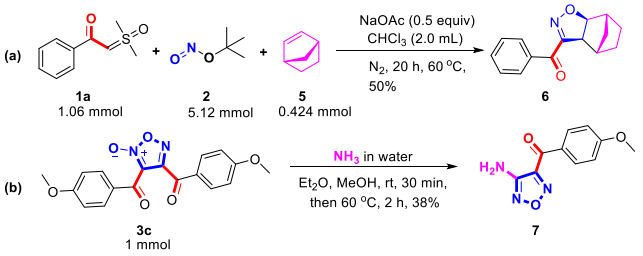
Scheme 3. Scope for the Synthesis of Isoxazoles



could afford the desired isoxazoles **4a**−**4d** in 28%−38% yields. The structure of product **4a** was unambiguously identified by X-ray single-crystal diffraction.

The utility of this protocol is illustrated by its application to transform the furoxans **3** in situ. When the reaction of sulfoxonium ylide **1a**, *tert*-butyl nitrite **2**, and norbornene **5** was conducted in one pot using chloroform as the solvent, the product ((3a*S*,4*S*,7*R*,7a*S*)-3a,4,5,6,7,7a-hexahydro-4,7-methanobenzo[*d*]isoxazol-3-yl)(phenyl)methanone **6** was obtained in moderate yield (Scheme 4a).[16] For further transformation of the furoxans, the treatment of furoxan **3c** with ammonium hydroxide in MeOH was also executed, in which

Scheme 4. Synthetic Applications and Transformations



the (4-amino-1,2,5-oxadiazol-3-yl)(4-methoxyphenyl)-methanone **7** could be achieved in 38% yield (Scheme 4b).[17]

In summary, we have developed the first [2 + 1 + 1 + 1] annulation reaction of sulfoxonium ylides for the divergent synthesis of furoxans and isoxazoles. When the reaction was carried out using NaOAc as base without metal catalyst, two molecules of sulfoxonium ylides and two molecules of ^{t}BuONO were incorporated into the product, in which a range of furoxans were achieved in decent yields with wide substrate scope. When the reaction was conducted using $Cu(TFA)_2$ as catalyst, trimolecular sulfoxonium ylides and one molecule of ^{t}BuONO were incorporated into the product, and isoxazoles bearing three carbonyl groups were constructed in one pot. The current protocol features simple operation, easily available starting materials, and good yields. Further application of sulfoxonium ylides as the carbene precursors is underway in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b01876.

Experimental procedures, characterization data, copies of NMR spectra (PDF)

Accession Codes

CCDC 1904440 and 1904505 contain 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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