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Silver-Catalyzed Cascade Cyclization Reaction of Isocyanides with Sulfoxonium Ylides: Synthesis of 3-Aminofurans and 4-Aminoquinolines

Yong-Xin Liang, Ming Yang, Bo-Wen He, and Yu-Long Zhao*

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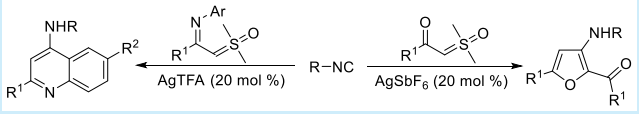
ABSTRACT: A silver-catalyzed cascade cyclization reaction of isocyanides with sulfoxonium ylides has been developed for the first time. This reaction provides a new and efficient method for the construction of highly functionalized 3-aminofurans and 4-aminoquinolines from readily available starting materials in a single step.



The furan scaffold is an important structural unit abundant in many biologically active natural products, pharmaceuticals, and agrochemicals.[1] Thus, considerable efforts have been developed to access polysubstituted furans.[2] As important members of these families, amino-substituted furans not only show potential biological activities[3] but also are useful and important intermediates with significant applications in organic synthesis.[4] Despite their broad utility, the availability of methods for the construction of 3-aminofurans is limited. Several methods have been reported for their synthesis, such as direct coupling reaction of 3-bromofuran with amines,[5] cyclization reactions of dibenzoylethylene or γ-hydroxy α,β-unsaturated acetylenic ketones with amines,[6] base-promoted cyclization reactions of α-cyanoketone with ethyl glyoxylate[7] or enaminones with aldehydes,[8] and base-mediated three-component reaction of thiazolium salts, aldehydes, and dimethyl acetylenedicarboxylate.[9] However, these reactions suffer from limited substrate scope, lack of readily available precursors, tedious synthetic procedures, or requirement for the use of stoichiometric amounts of strong bases. Recently, transition-metal-catalyzed reactions, including the gold-catalyzed three-component reaction of phenylglyoxal derivatives, secondary amines, and terminal alkynes;[10] the rhodium/gold-cocatalyzed domino reaction of *N*-sulfonyl-1,2,3-riazoles with propargyl alcohols;[11] and the zinc-catalyzed [4 + 1] annulation of alkylthio-substituted enaminones with sulfur ylides,[12] have become a convenient and promising method for the synthesis of 3-aminofurans. Despite great progress, exploiting new transition-metal-catalyzed synthesis of 3-aminofurans from readily available precursors is highly desired.

On the other hand, quinoline compounds, as an important heterocyclic skeleton, are widely present in natural products and pharmaceuticals with a broad range of bioactivities. Among them, 4-aminoquinolines have attracted much attention because of their effective drug activity.[13] Especially, 4-aminoquinolines bearing 2-aryl functionalities show promise as non-nucleoside HIV-1 inhibitors.[14] Many methods for preparing 4-aminoquinolines bearing 2-aryl functionalities have been developed.[15] In 2013, Orru and co-workers reported a Pd-catalyzed oxidative cyclization of *tert*-butyl isocyanide with *N*-arylimines to afford the 4-aminoquinolines. However, this method was limited to *tert*-butyl isocyanide and low yields.[15e] Thus, the development of an efficient and simple method for the construction of 4-aminoquinolines bearing 2-aryl functionalities is still in need.

Isocyanides are extraordinarily versatile and powerful building blocks due to their unique reactivity toward electrophiles, nucleophiles, and radicals.[16] In this field, transition-metal-catalyzed coupling reactions of isocyanides with various carbene precursors, such as diazo compounds[17] and organic azides,[18] have become a convenient and promising approach to access various N-containing compounds. In addition, as a kind of alternative carbene analogues, sulfonium/sulfoxonium ylides have been widely applied in transition-metal-catalyzed C−H activation due to their easy accessibility, stability, and diverse reactivity.[19] Aside from these transformations, transition-metal-catalyzed cross-coupling reactions of sulfoxonium ylides via the carbene insertion process were also sporadically reported in recent years.[20] However, to the best of our knowledge, the transition-metal-catalyzed carbene-transfer reaction of sulfoxonium ylides with isocyanides has not been reported. As part of our continuing interest in the applications of diazo compounds[21] and isocyanides,[22] herein we report a novel silver-catalyzed coupling cyclization of isocyanides with sulfoxonium ylides for the first time. This reaction offers a new and efficient strategy for the synthesis of

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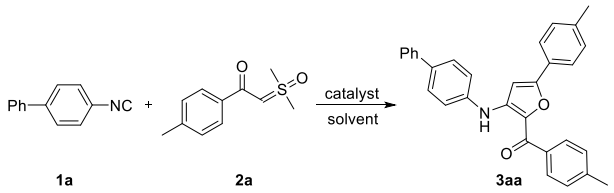
highly functionalized 3-aminofurans and 4-aminoquinolines in a single step from readily available starting materials.

Initially, the reaction of 4-isocyanobiphenyl **1a** with sulfoxonium ylide **2a** was investigated to optimize the reaction conditions (Table 1). We found that, under the catalysis of

Table 1. Optimization of Reaction Conditions[a,b]



1a 2a 3aa

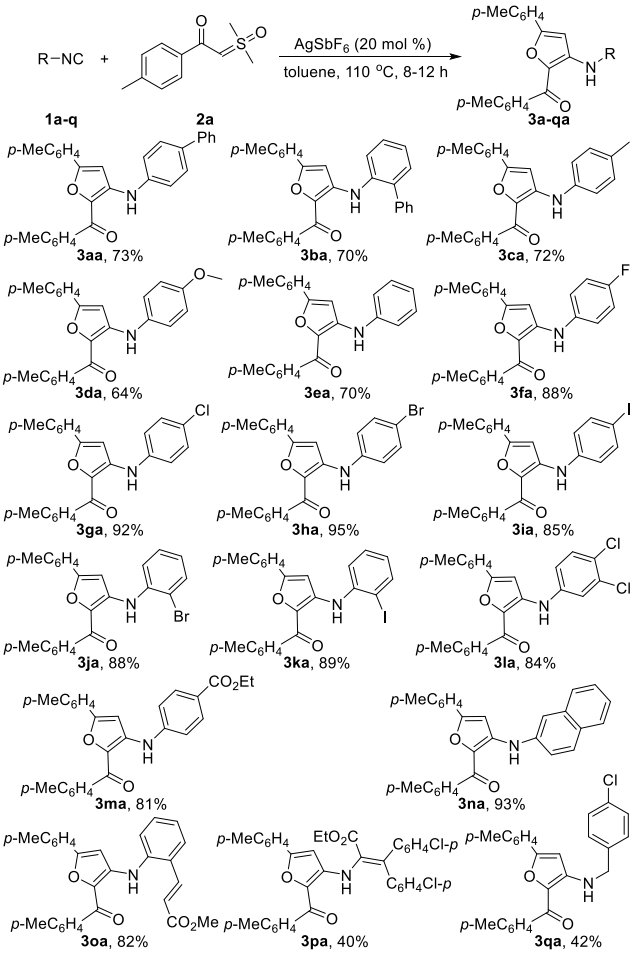
entry	catalyst (mol %)	1a:2a	solvent	t/h	yield (%)[b]
1	$AgSbF_6$ (10)	1:2	toluene	10	42
2	$AgSbF_6$ (20)	1:2	toluene	10	73[c]
3	$AgSbF_6$ (30)	1:2	toluene	10	54
4	$AgSbF_6$ (20)	1:1	toluene	10	27
5	$AgSbF_6$ (20)	1:3	toluene	10	48
6	Ag(TFA) (20)	1:2	toluene	10	11
7	AgOTf (20)	1:2	toluene	10	24
8	Ag_2CO_3 (20)	1:2	toluene	10	---
9	AgOAc (20)	1:2	toluene	10	---
10	$AgNO_3$ (20)	1:2	toluene	10	10
11	$AgSbF_6$ (20)	1:2	*m*-xylene	10	30
12	$AgSbF_6$ (20)	1:2	PhCl	12	20
13	$AgSbF_6$ (20)	1:2	DCE	12	21
14	$AgSbF_6$ (20)	1:2	DMF	12	---

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), catalyst (0.02−0.06 mmol), solvent (2.0 mL), at 110 °C for 10−12 h. [b]Estimated by ^{1}H NMR spectroscopy using 1,3,5-trimethoxybenzene as an internal standard. [c]Isolated yield.

$AgSbF_6$ (10 mol %) in toluene at 110 °C for 10 h, the reaction of **1a** (0.2 mmol) with **2a** (0.4 mmol) resulted in 3-aminofuran **3aa** in 42% yield (entry 1). To our delight, the yield of **3aa** was raised to 73% in the presence of $AgSbF_6$ (20 mol %) (entry 2). Decreasing the ratio of **2a**/**1a** to 1/1, the product **3aa** was obtained in 27% yield along with the recovery of 4-isocyanobiphenyl **1a** in 38% yield (entry 4). Interestingly, increasing the amount of $AgSbF_6$ and the ratio of **2a**/**1a** led to lower yields of **3aa** (entries 3 and 5). Other silver catalysts such as Ag(TFA), AgOTf, Ag_2CO_3, AgOAc, and $AgNO_3$ were less effective (entries 6, 7, and 10) or ineffective (entries 8 and 9). In addition, solvent screening revealed that solvents have a great effect on the reaction. Among the solvents tested, toluene seemed to be the best choice. Other solvents, such as *m*-xylene, PhCl, and DCE, gave lower product yields (entries 11−13). No desired product **3aa** was observed (TLC) when the reaction was performed in DMF (entry 14).

With the optimal reaction condition in hand, the scope of the [3 + 1 + 1] cyclization reaction was examined with respect to isocyanides **1**, and the results are summarized in Scheme 1. All selected aryl isocyanides **1a−o**, bearing phenyl, electron-rich, electron-deficient aryl, biphenyl, and 2-naphthyl R groups, reacted smoothly with sulfoxonium ylide **2a** to give the corresponding 3-aminofurans **3a−oa** in good to high yields. In contrast, aryl isocyanides **1f−m** and **1o** with electron-withdrawing groups on the phenyl ring and naphthalenyl isocyanide **1n** gave relatively higher yields. It is noteworthy that the vinyl isocyanide **1p** can be tolerated, with the

Scheme 1. Scope of the Reaction with Respect to the Isocyanides 1a−q[a,b]



[a]Reaction conditions: **1** (0.2 mmol), **2a** (0.4 mmol), $AgSbF_6$ (0.04 mmol), toluene (2.0 mL), at 110 °C for 8−12 h. [b]Isolated yield.

generation of **3pa** in 40% yield. More importantly, benzyl isocyanide **1q** was found to be a suitable coupling partner, generating the desired 3-aminofuran **3qa** in 42% yield. However, no desired product was observed when *tert*-butyl isocyanide was employed as the partner (Scheme 1). In addition, we found that the [3 + 1 + 1] annulation reaction is easy to scale-up. A scale-up reaction of **1g** (4.0 mmol) and **2a** (8.0 mmol) was carried out for 10 h under otherwise identical conditions as above, furnishing 1.14 g of the desired product **3ga** in 71% isolated yield.

Next, the scope of sulfoxonium ylides **2** was explored under the optimal conditions. As shown in Scheme 2, various benzoyl-substituted sulfoxonium ylides **2b−j** bearing a variety of functional groups, such as electron-donating groups (CH_3, *t*-Bu, OMe) and electron-withdrawing groups (Cl, Br, F, CF_3) at the para, ortho, and meta positions of the phenyl ring, reacted smoothly with **1a** to produce the corresponding 3-aminofurans **3ab−j** in good to high yields. Similarly, thienyl-substituted sulfoxonium ylide **2k** was also compatible in this reaction and produced the desired product **3ak** in 53% yield. Notably, in the case of alkanoyl sulfoxonium ylide **2l**, the [3 + 1 + 1] cyclization reaction also worked well, affording the desired product **3al** in 65% yield (Scheme 2). In addition, we found that, when two different sulfoxonium ylides **2a** (0.2 mmol) and

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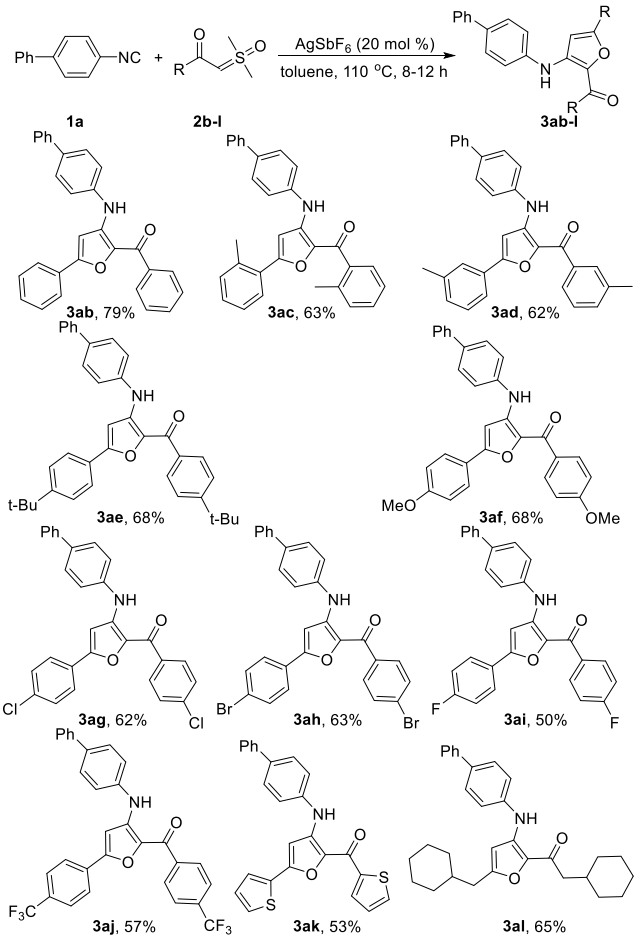
Org. Lett. XXXX, XXX, XXX−XXX

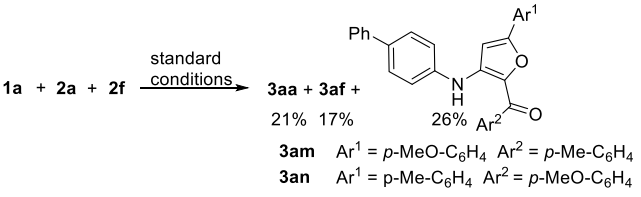
Scheme 2. Scope of the Reaction with Respect to the Sulfoxonium Ylides 2b−l[a,b]



[a]Reaction conditions: **1a** (0.2 mmol), **2b−l** (0.4 mmol), $AgSbF_6$ (0.04 mmol), toluene (2.0 mL), at 110 °C for 8−12 h. [b]Isolated yield.

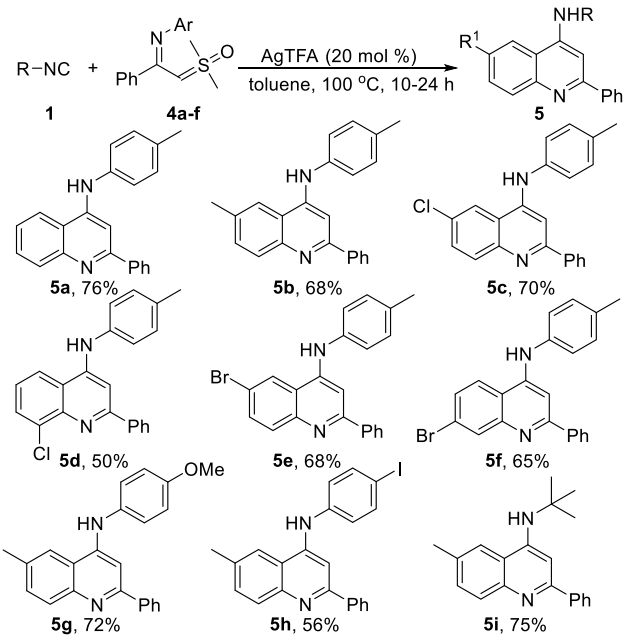
2f (0.2 mmol) were treated with **1a** (0.2 mmol) under the optimized reaction conditions, 3-aminofurans **3aa** and **3af** were produced in 21% and 17% yields along with the mixture of **3am** and **3an** in 26% yield, respectively (Scheme 3).

Scheme 3. Reaction of 1a with Two Different Sulfoxonium Ylides 2a and 2f



On the basis of the above experimental results, along with the consideration of the structural feature of *N*-aryl imidoyl sulfoxonium ylides,[23] we reasoned that the ketenimine intermediate generated from the reaction of *N*-aryl imidoyl sulfoxonium ylides **4a−f** and isocyanides **1** can undergo an intramolecular cyclization to afford 4-aminoquinolines. As expected, when the reaction of **1c** (0.2 mmol) and *N*-phenyl imidoyl sulfoxonium ylide **4a** (0.2 mmol) was carried out under identical conditions as above, the 4-aminoquinoline **5a** was obtained in 43% yield (Scheme 4). To our delight, after a series of optimization steps, we found that, under the catalysis

Scheme 4. Synthesis of 4-Aminoquinolines 5[a,b]



[a]Reaction conditions: **1** (0.2 mmol), **4a−f** (0.2 mmol), AgTFA (0.04 mmol), toluene (2.0 mL), at 100 °C for 10−24 h. [b]Isolated yield.

of AgTFA (20 mol %) in toluene at 100 °C for 24 h, the cascade cyclization reaction of **1c** with **4a** resulted in 4-aminoquinoline **5a** in 76% yield. Similarly, *N*-aryl imidoyl sulfoxonium ylides **4b−e** bearing either electron-donating or electron-withdrawing R^1 groups on the aromatic rings could react efficiently with isocyanide **1c** to produce 4-aminoquinolines **5b−e** in good yields. When the *N*-aryl imidoyl sulfoxonium ylide **4f** bearing the Br group at the meta position of the aromatic ring was employed as a coupling partner, the product **5f** was produced in 65% yield. In addition, aryl isocyanides **1d** and **1i** with electron-rich and electron-deficient substituents also participated efficiently in this transformation, giving the 4-aminoquinolines **5g** and **5h** in 72% and 56% yields, respectively. More importantly, *tert*-butyl isocyanide **1r** was also tolerant, with the generation of 4-aminoquinolines **5i** in 75% yield (Scheme 4).

On the basis of the above experimental results together with related reports,[16−20,24] a possible mechanism for the formation of **3** and **5** is proposed in Scheme 5. Initially, isocyanides **1** coordinate with $AgSbF_6$ or AgTFA to form intermediate **A**, which reacts with sulfoxonium ylides **2** or **4** to give intermediate **B**. Subsequently, intermediate **C**, generated by the elimination of DMSO and silver, was trapped by sulfoxonium ylides **2a−l** to produce intermediate **D**. Intermediate **D** undergoes intramolecular nucleophilic substitution reaction to generate intermediate **E** along with the elimination of DMSO. Finally, intermediate **E** undergoes intramolecular cyclization to form **F**, followed by the 1,3-proton shift to give the corresponding 3-aminofurans **3**. In the case of *N*-aryl imidoyl sulfoxonium ylides **4a−f**, the ketenimine intermediate **G** undergoes an intramolecular Friedel−Crafts cyclization or electrocyclization, followed by the protonation to produce the 4-aminoquinolines **5** (Scheme 5).

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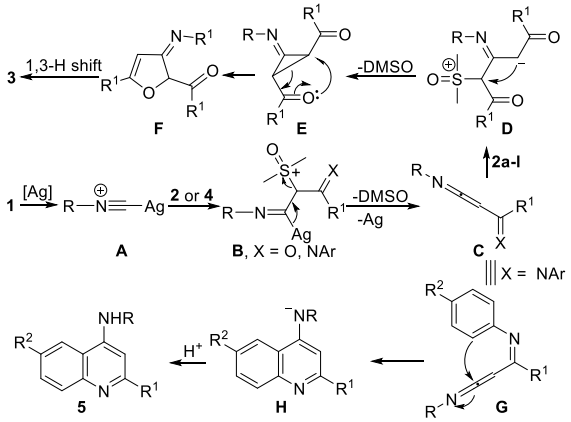
Org. Lett. XXXX, XXX, XXX−XXX

Scheme 5. Proposed Mechanism for the Formation of 3 and 5



In summary, we have developed a silver-catalyzed cascade cyclization reaction of isocyanides with sulfoxonium ylides for the first time. This new reaction provides a novel and efficient method for the synthesis of 3-aminofurans and 4-aminoquinolines in a single operation. This protocol features readily available starting materials, broad substrate scope, good to high yields, and good functional group tolerance. Further studies on the coupling reactions of isocyanides with sulfoxonium ylides are in progress.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c02835.

Experimental procedures, full spectroscopic data, and ^{1}H and ^{13}C NMR spectra of all compounds (PDF)

Accession Codes

CCDC 1994946 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.

■ AUTHOR INFORMATION

Corresponding Author

Yu-Long Zhao − *Jilin Province Key Laboratory of Organic Functional Molecular Design & Synthesis, Faculty of Chemistry, Northeast Normal University, Changchun 130024, China;* orcid.org/0000-0001-6577-1074; Email: zhaoyl351@nenu.edu.cn

Authors

Yong-Xin Liang − *Jilin Province Key Laboratory of Organic Functional Molecular Design & Synthesis, Faculty of Chemistry, Northeast Normal University, Changchun 130024, China*

Ming Yang − *Jilin Province Key Laboratory of Organic Functional Molecular Design & Synthesis, Faculty of Chemistry, Northeast Normal University, Changchun 130024, China*

Bo-Wen He − *Jilin Province Key Laboratory of Organic Functional Molecular Design & Synthesis, Faculty of Chemistry, Northeast Normal University, Changchun 130024, China*

Complete contact information is available at: https://pubs.acs.org/10.1021/acs.orglett.0c02835

Notes

The authors declare no competing financial interest.

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