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Synthesis of Quinoxaline Derivatives via Tandem Oxidative Azidation/Cyclization Reaction of *N*-Arylenamines

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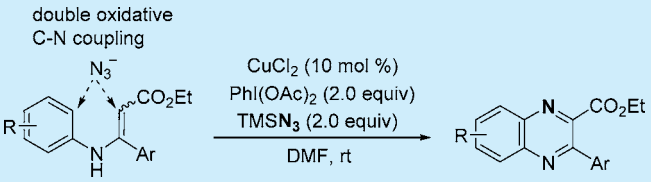
Supporting Information

ABSTRACT: A new method was developed for the synthesis of quinoxalines. This method employs *N*-arylenamines and $TMSN_3$ as the starting materials and implements two oxidative C−N bond-forming processes in a tandem pattern by using (diacetoxyiodo)benzene as the common oxidant. The present reaction conditions are mild and simple and thus are useful in practical synthesis.



Azide-involved C−N-forming reactions are of great importance in the synthesis of nitrogen-containing compounds.[1] On the one hand, organic azides can be conveniently prepared by various types of ionic[1,2] and radical azidation reactions[3,4] as well as transition-metal-catalyzed coupling reactions.[5] On the other hand, organic azides can react under a variety of conditions to form a new C−N bond.[1,6] Recent studies show that organic azides are also reliable precursors to aminyl and iminyl radicals.[7,8] From the viewpoint of synthetic efficiency, it is highly desirable to combine azidation reactions with the azidyl-derived C−N-forming process in one synthetic operation, in a way that two C−N bonds can be constructed in a tandem or one-pot pattern.[9]

Quinoxaline is an important heterocyclic skeleton that appears in many biologically and pharmaceutically significant compounds (Figure 1). The commonly used methods to gain access to quinoxaline derivatives comprise the Hinsberg−Körner reaction and other relevant reactions that use 1,2-disubstituted benzenes, such as 1,2-diaminobenzenes, *ortho*-nitroanilines, or 2-fluoro-1-nitrobenzenes, as the substrates,[10] whereas those starting from simpler monosubstituted anilines were still lacking until now.[11]

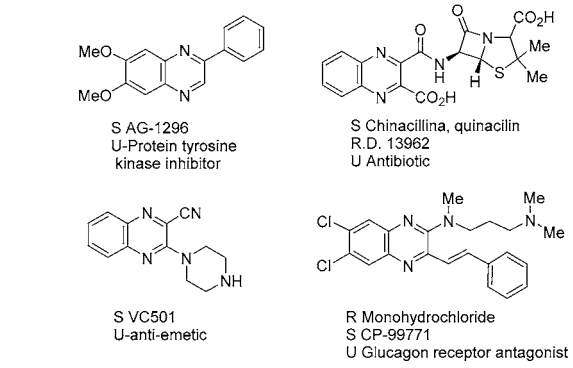
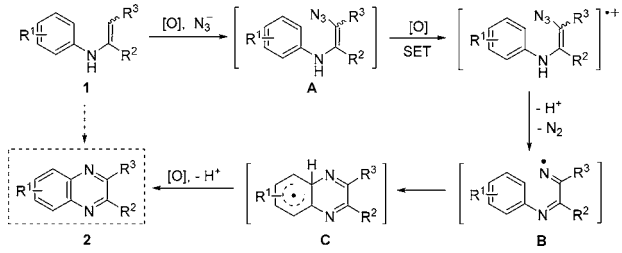


Figure 1. Several quinoxaline-containing drugs.

Recently, Spagnolo et al.,[12] Zhang et al.,[13] and our group[14] found that the cyclization of iminyl radicals provides an effective means for the synthesis of a quinoxalin-2-one ring system, a sister structure of quinoxaline. We envisioned that the quinoxaline motif might also be prepared through a similar process. Our literature survey shows that the quinoxaline-forming radical cyclization was reported decades ago by McNab[15] and Nanni et al.,[16] but the synthetic value of this reaction has not been demonstrated by further study. To develop a method of practical usefulness, and also to expand the azide-based C−N-forming reactions, we designed a new tandem strategy for the synthesis of a quinoxaline ring system. This strategy uses structurally simple enamines as the starting material and features two consecutive C−N-forming reactions with an azide ion as the nitrogen source.

Our synthetic strategy is outlined in Scheme 1. It was expected that *N*-arylenamine (**1**) would undergo oxidative azidation to

Scheme 1. Synthetic Design toward a Quinoxaline Ring in This Work



afford vinyl azide intermediate **A**; further oxidation of **A** would result in the formation of iminyl radical **B**, from which quinoxaline **2** could be formed via cyclization. Thus, the realization of this plan hinged on two key steps: azidation of enamine **1** and conversion of **A** to iminyl radical **B**.

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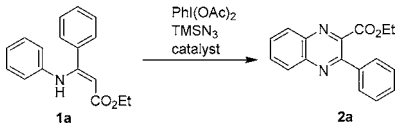
Studies by Chiba et al.[17] and others[18,19] show that vinyl azides constitute a reliable source of iminyl radicals. Considering the electron richness of 2-azidyl enamine **A**, we assumed that it would be readily oxidized to iminyl radical **B** via a single electron transfer process. The transformation from enamine **1** to **A** would also be achieved using an oxidant in combination with an azidating reagent. Previous studies demonstrate that the azidation of olefins can be readily achieved under oxidative conditions.[3b,20] Thus, we anticipated that these two oxidative steps could be combined in a tandem fashion by using one oxidant. Hypervalent iodine reagents were believed to be suitable oxidants to meet this requirement because they are not only capable of oxidizing enamines[21,22] but also can effect the oxidative azidation of olefins including enamines.[20h]

Previous investigations by Zhao[21] and our group[22] show that enamine esters can be readily oxidized by (diacetoxyiodo)-benzene (DIB). Thus, at the initial stage of this study, we chose compound ethyl-3-amino-3-phenyl acrylate (**1a**) as the substrate and subjected it to $TMSN_3$ and DIB. The desired reaction took place readily at room temperature in DCM or CH_3CN, giving rise to quinoxaline product **2a** in moderate yield (Table 1, entries 1−5). Comparable yields were obtained when (ditrifluoroacetoxyiodo)benzene was used instead of DIB (not shown in Table 1). Subsequent experiment indicated that using $CuCl_2$ as catalyst would improve the yield of **2a**. Thus, when the reaction was carried out in DMF with 10 mol % of $CuCl_2$ as catalyst, **2a** was obtained in a yield of 69% (Table 1, entry 10). Several other copper salts were also tested for their effectiveness, but they were found to be inferior to $CuCl_2$ under the current circumstance (Table 1, entries 13−15).

Table 1. Screening of the Reaction Conditions with 1a as the Substrate[a]



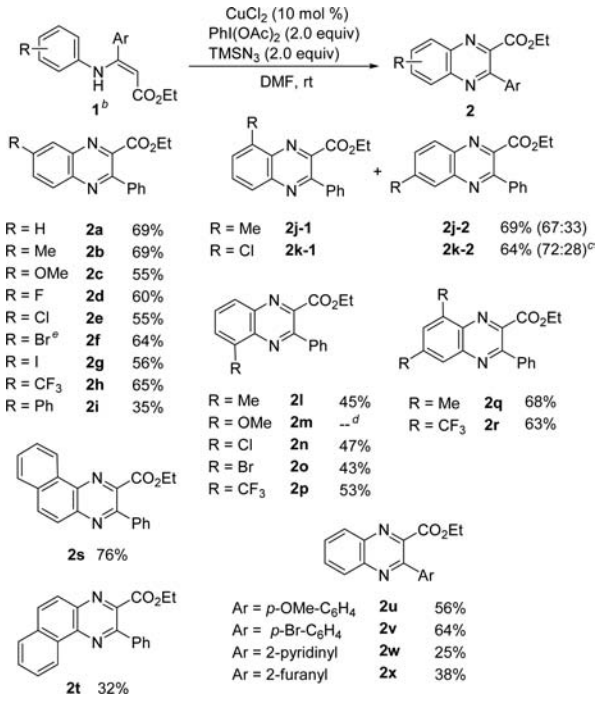
entry	equiv of DIB	equiv of $TMSN_3$	cat.[b]	solv.	yield (%)[c]
1	2.0	2.0	none	DCM	28
2	3.0	2.0	none	DCM	32
3	3.0	3.0	none	DCM	37
4	3.0	3.0	none	CH_3CN	29
5	2.0	1.0	none	DCM	14[d]
6	2.0	3.0	$CuCl_2$	DCM	53
7	2.0	3.0	$CuCl_2$	CH_3CN	53
8	2.0	3.0	$CuCl_2$	DMF	37
9	2.0	3.0	$CuCl_2$[e]	DMF	47
10	***2.0***	***2.0***	***$CuCl_2$***	***DMF***	***69***
11	2.0	2.0	$CuCl_2$	DCM	42
12	2.0	2.0	$CuCl_2$	CH_3CN	50
13	2.0	2.0	$CuBr_2$	DMF	41
14	2.0	2.0	$Cu(OAc)_2$	DMF	49
15	2.0	2.0	$CuPF_6$	DMF	56

[a]Reaction was carried out on a 0.2 mmol scale in 2 mL of solvent at room temperature. Reaction time was 2−4 h unless otherwise specified. [b]With 10 mol % of copper salt used unless otherwise specified. [c]Isolated yield. [d]Reaction time was 10 h. [e]With 0.5 equiv of $CuCl_2$. DCM: dichloromethane. $CuPF_6$ is in the form of $Cu(CH_3CN)_4PF_6$.

The optimized reaction conditions (Table 1, entry 10) were then applied to variously substituted enamine esters (**1b−1x**), and the results are illustrated in Scheme 2. Under the indicated conditions, all tested substrates except ethyl 3-((2-methoxyphenyl)amino)-3-phenyl acrylate (**1m**) were converted to the corresponding quinoxalines **2**, albeit with yields that varied according to the substitution patterns. It is interesting to see that compounds **1l**, **1n**, **1o**, and **1p**, which incorporate an *ortho*-substituent at the *N*-phenyl ring, also reacted to give quinoxalines. This result is in contrast to that of our previous studies on the cyclization of *α*-(aminocarbonyl)iminyl radicals, where an *ortho*-substituent at the *N*-phenyl ring would prevent the formation of a quinoxalin-2-one ring.[14] This discrepancy can be attributed to the relief of steric hindrance in **1l−1p**, in which it is the small hydrogen atom other than an alkyl group that is attached to the nitrogen atom. The steric repulsion between the *ortho*-substituent and the *N*-alkyl group is believed to be the reason why the corresponding *α*-(aminocarbonyl)iminyl radicals failed to cyclize to afford the quinoxalin-2-one ring. When *meta*-substituted **1j** and **1k** were used as the substrates, the products were the mixture of two regioisomers, with the sterically hindered **2j-1** and **2k-1** being the major products. This selectivity is in accordance with our previous observations.[14]

Cyclization of aryliminoiminyl radicals to a quinoxaline ring can be realized in two patterns: direct cyclization and initial formation of a spirodienyl radical followed by 1,2-iminyl migration (Scheme 3). Cyclization via the second pathway would lead to the formation of rearrangement products,[15] as both iminyl groups in the spirodienyl radical intermediate could migrate. In the present reactions, however, no rearrangement

Scheme 2. Preparation of Quinoxalines from Variously Substituted Enamine Esters[a]



[a]Reaction was conducted on a 0.5 mmol scale. [b]Substrates are of *Z* configuration. [c]Ratio was determined by ^1H NMR. [d]Complex mixture was generated. [e]Structure of **2f** was confirmed by single-crystal X-ray analysis. See ref 23.

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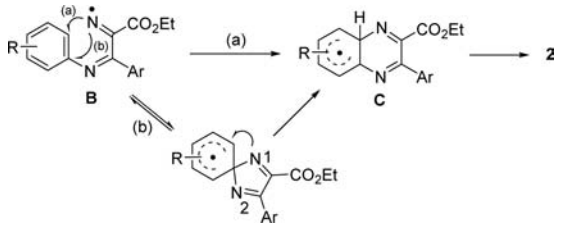
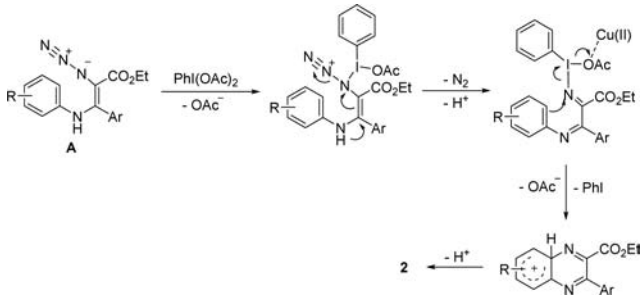
Scheme 3. Proposed Reaction Mechanism



products were obtained. This result is in accordance with that reported by Nanni et al.[16] and thus supports that the cyclization of radical **B** follows exclusively path (a). Apart from this radical mechanism, there is another possibility that transformation from **A** to final product **2** follows the nonradical pathway shown in Scheme 4. Although we cannot determine which mechanism is

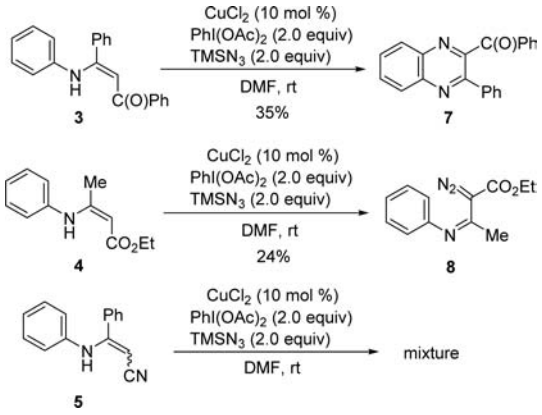
Scheme 4. Alternative Nonradical Mechanism



more plausible at the current stage, one thing is certain: oxidation of compound **A** must be a fast process because under no circumstance could it be isolated from the reaction mixture.

To further examine the scope of this method, we applied it next to compounds **3**–**6** (Schemes 5 and 6). As shown in Scheme

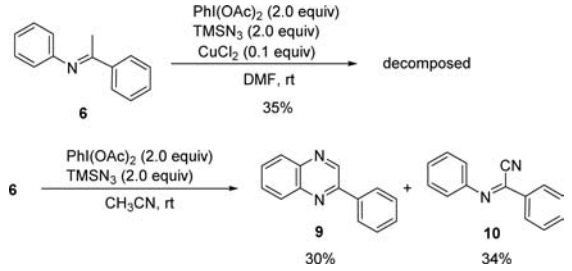
Scheme 5. Test on the Scope of This Method (1)



5, compound **3** reacted under the indicated conditions to afford the desired product **7** in a yield of 35%, but similar reaction did not occur for compounds **4** and **5**. In the case of **4**, the only isolable product was α-diazo ester **6**; the reaction of **5**, on the other hand, produced a complex mixture.

The conditions shown in Scheme 2 also failed when applied to compound **6**. However, when the reaction was performed under copper-free conditions, the quinoxaline product **9** was generated in a moderate yield (Scheme 6), along with compound **10**.

Scheme 6. Test on the Scope of This Method (2)



Compound **10** is obviously derived from further oxidation of the formed azidyl intermediate.[24]

In summary, we have developed a new and efficient method for the synthesis of quinoxaline derivatives. This method uses readily accessible *N*-arylenamines as the precursors and takes advantage of a tandem oxidative azidation/cyclization process to build two C–N bonds consecutively with $TMSN_3$ as the nitrogen source. As the current reaction can take place rapidly under mild and simple conditions, it can be applied to practical synthesis. Despite these advantages, however, the current protocol has some limitation in scope, and further work is being done in our group to explore better reaction conditions to solve this problem.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b00148.

General experimental procedures, characterization data for the substrates and products, ^{1}H NMR and ^{13}C NMR spectra of the substrates and products (PDF)

Crystallographic data for compound **2f** (CIF)

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Notes

The authors declare no competing financial interest.

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(23) The structure of compound **2f** was confirmed by single-crystal X-ray analysis. CCDC 1441580 contains the supplementary crystallographic data. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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