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Construction of *N*-Alkyl- and *N*-Arylaziridines from Unprotected Amines via C−H Oxidative Amination Strategy

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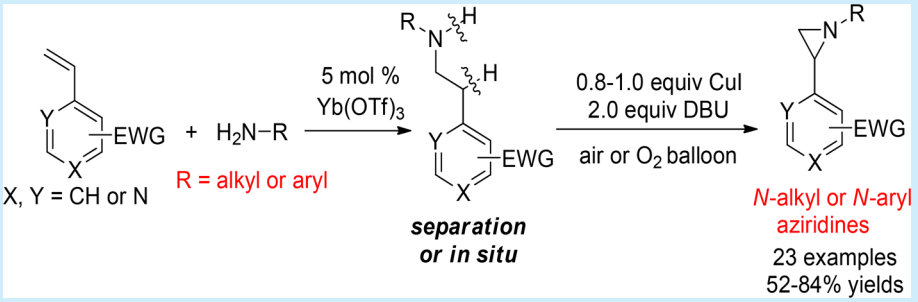
Supporting Information

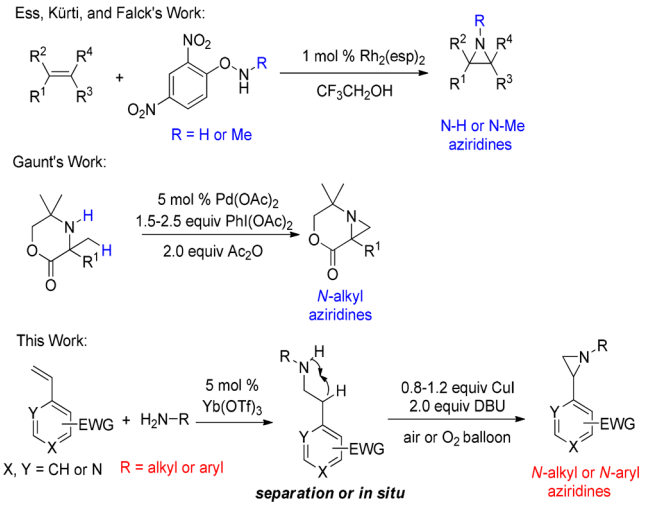


ABSTRACT: A copper-promoted intramolecular C−H oxidative amination reaction between secondary amine (N−H) and C(sp^3)−H at the benzylic position of azaarenes or α-position of ketones for the synthesis of aziridine derivatives has been developed. Moreover, a practical annulation of electron-deficient vinylarenes with an unprotected primary alkyl amine by a Yb(OTf)$_3$−CuI relay system has also been reported. The reactions were carried out with oxygen as the sole oxidant to give the *N*-alkyl- and *N*-arylaziridines in good yields.

Aziridines and their derivatives are important classes of *N*-containing heterocycles occurring in numerous natural products,[1] biologically active compounds,[2] and intermediates in organic synthesis.[3] Thus, aziridines, the smallest ring motifs, have been of great interest to chemists for years.[4] The main approaches for the construction of aziridines[5] involve nitrogen−olefin additions,[6] carbon−imine cyclizations,[7] intramolecular substitutions,[8] and other reactions.[9] The nitrogen−olefin addition strategy requires stable *N*-protecting groups such as *p*-toluenesulfonyl (Ts) or *p*-nitrophenylsulfonyl (Ns), which are often troublesome to remove and lead to a reduced overall yield.[10] Furthermore, the other methods require prefunctionalization of the reaction substrates with a leaving group such as OTs, OAc, halogen, ylide, and azide, which is not atom-economic. Therefore, the development of practical methods for the straightforward synthesis of aziridines by using non-functionalized reagents remains a challenge.

Recently, Falck and co-workers developed a new synthetic method for the formation of unprotected N−H and N-Me aziridines from simple olefins via homogeneous rhodium catalysis (Scheme 1).[11] In addition, Gaunt's group reported palladium-catalyzed direct intramolecular C−H amination for the synthesis of unprotected aziridine derivatives starting from cyclic aliphatic amines (Scheme 1).[12] However, the aziridine scope in this method is very limited. It is highly desirable to

Scheme 1. Transition-Metal-Catalyzed or Promoted Synthesis of *N*-Unprotected Aziridines



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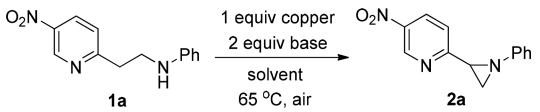
develop a facile and efficient procedure for constructing unprotected *N*-aryl- or *N*-alkylaziridines. Recently, an oxidative cross-coupling reaction between two nucleophiles has been developed as a powerful synthetic methodology to construct functional molecules.[13] As far as we know, the synthesis of aziridines from simple amines by using oxidative cross-coupling has not been reported. Based on our early investigation of functionalization of azaarenes,[14] we want to develop a new strategy that nucleophilic amine reacts with nucleophilic benzylic C−H of azaarenes[15] to produce structure diversified aziridines through an oxidative cross-coupling pathway. Herein, we present an account of our recent work on copper-mediated[16] annulation from secondary amine and C(sp^3) at the benzylic position of azaarenes or α-position of ketones for the synthesis of *N*-alkyl- and *N*-arylaziridines.

In the initial attempt on the formation of aziridines, secondary amine **1a** was synthesized by Michael addition between aniline and 5-nitro-2-vinylpyridine catalyzed by Yb(OTf)$_3$ (see the Supporting Information). The solution of **1a** (0.02 mmol) in the presence of CuBr (0.02 mmol) and 2,3,4,6,7,8,9,10-octahydropyrimido[1,2-*a*]azepine (DBU) (0.04 mmol) in toluene was stirred for 2 h at 65 °C. The desired product aziridine **2a** was obtained in 59% yield (Table 1, entry 1).

Table 1. Optimization of Reaction Conditions.[a]



entry	copper	base	solvent	yield[b] (%)
1	CuBr	DBU	toluene	59
2	$CuBr_2$	DBU	toluene	54
3	CuI	DBU	toluene	72
4[c]	CuI/PIDA	DBU	toluene	33
5[d]	CuI/TBHP	DBU	toluene	trace
6[e]	CuI	DBU	toluene	51
7	CuI	DBU	dioxane	63
8	CuI	DBU	DCE	48
9	CuI	DBU	DMF	7
10	CuI	TBD	toluene	11
11	CuI	DMAP	toluene	trace
12	CuI	DABCO	toluene	trace
13[f]	CuI	DBU	toluene	53
14[g]	CuI	DBU	toluene	35

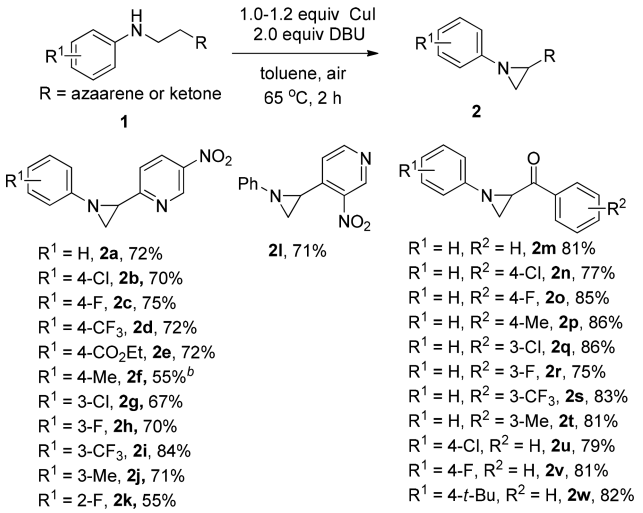
[a]Unless specified, amine **1a** (0.2 mmol), base (0.4 mmol), and copper salt (0.2 mmol) in 1.5 mL of toluene was stirred for 2 h at 65 °C. [b]Isolated yield. [c]0.3 equiv of CuI and 2.0 equiv of PIDA were used under nitrogen. [d]0.3 equiv of CuI and 2.0 equiv of TBHP were used under nitrogen. [e]1.0 equiv of DBU was used. [f]The reaction was carried out at rt. [g]The reaction was carried out at 90 °C.

Inspired by this result, the reaction conditions were optimized to improve the reaction yield. $CuBr_2$ gave a slightly lower reaction yield than CuBr (entry 2). The yield was improved to 72% when CuI was used (entry 3).

The effect of oxidants was also examined in this reaction. The product **2a** was obtained in 33% yield in the presence of CuI (0.3 equiv) and oxidant (diacetoxyiodo)benzene (PIDA). In comparison, no product **2a** was observed when PIDA was replaced with 2-hydroperoxy-2-methylpropane (TBHP) (entry 5). Decreasing the amount of DBU to 1 equiv reduced the reaction yield to 51% (entry 6). The solvent screening revealed that more polar solvents, such as dioxane, dichloroethane (DCE), and DMF, dioxane, and DCE gave slightly lower yields (entries 7 and 8), and DMF gave very poor yield (entry 9). Compared to DBU, three other common organic bases, 2,3,4,6,7,8-hexahydro-1*H*-pyrimido[1,2-*a*]pyrimidine (TBD), *N*,*N*-dimethylpyridin-4-amine (DMAP), and 1,4-diazabicyclo[2.2.2]octane (DABCO), were proven to be less efficient (entries 10−12). Moreover, the results showed that temperature has an influence on the yields, the yield was significantly lower at room temperature or 90 °C (entries 13 and 14). Thus, the optimal reaction condition was determined as follows: 1 equiv of CuI and 2 equiv of DBU in toluene for 2 h at 65 °C.

With the optimal reaction conditions in hand, we then examined the substrate scope of CuI-promoted C(sp^3)−N coupling for the formation of aziridines **2** from amines **1** with azaarenes. As shown in Scheme 2, almost all of the tested

Scheme 2. Scope of *N*-Arylamines for the Synthesis of *N*-Arylaziridines[a]



[a]Reaction conditions: a solution of **1a** (0.2 mmol) and CuI (0.2 mmol) with DBU (0.4 mmol) in toluene (1.5 mL) was stirred for 2 h at 65 °C. 0.24 mmol of CuI was used for **2m**−**w**. Isolated yield. [b]The reaction was run for 2 h at 45 °C.

combinations produced the corresponding products in moderate to high yields (**2a**−**l**). Electron-withdrawing groups (EWGs) on the benzene rings such as halogen, CF_3, and CO_2Et in the *para*-position on the phenyl ring had a slight impact on the yields (**2a**−**e**, 70−75%). Even for the amine-bearing electron-donating group (EDG) on the *para*-position of phenyl ring, the reaction proceeded smoothly in 55% yield (**2f**). Furthermore, the application of substrates bearing a *meta*-EWG substituent on the phenyl ring provided the desired products in good yields as well (**2g**−**i**, 67−84%). *m*-Me- and *o*-F-substituted amines also transformed to the corresponding products **2j** and **2k** in good yields. Moreover, aniline **1l** with benzylic C−H at the *para*-position of pyridine can also be suitable for this protocol to give the corresponding product **2l** in 71% yield. To further expand the scope of the reaction, β-secondary amino ketones were investigated (**1m**−**w**). In general, good yields were achieved across a range of substituted α-aryl ketones. The simple 1-phenyl-3-(phenylamino)propan-1-one **1m**, promoted by 1.2 equiv of CuI, generated the aziridine product **2m** in 81% yield. The results showed that there was no significant influence

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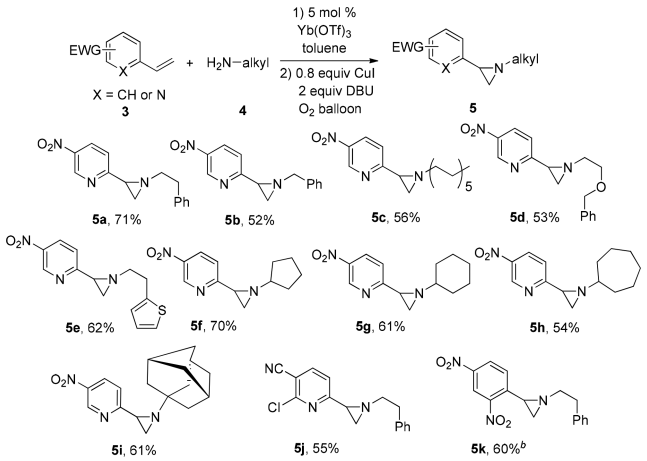
whether R^1 was *para*-EWG (**2n** and **2o**) or *para*-EDG (**2p**). Similar results were also observed in the aromatic anilines with EWGs and EDG at the *meta*-position of the phenyl ring of ketones (**2q**–**t**). Furthermore, the ketones with *p*-Cl, *p*-F, and bulky *p*-*t*-Bu on the phenyl ring also gave high yields (**2u**–**w**). The structure of the products was determined by the single-crystal X-ray diffraction analysis of **2g**.[17]

Besides *N*-arylaziridines, *N*-alkylaziridines were also successfully synthesized via this C(sp^3)–N coupling reaction. A $Yb(OTf)_3$/CuI relay system has been developed via a Michael-oxidative cycloaddition tandem pathway. A solution of 2-vinylpyridine **3** (0.4 mmol), alkylamine **4** (0.8 mmol), and $Yb(OTf)_3$ (0.02 mmol) in toluene (2 mL) was stirred for 0.5 h at 65 °C. Then DBU (0.8 mmol) and CuI (0.32 mmol) were added into the solution for another 2 h under the O_2 atmosphere to afford the aziridine products **5** (Scheme 3). Primary amines with

Scheme 3. Scope of *N*-Alkylamines[a]

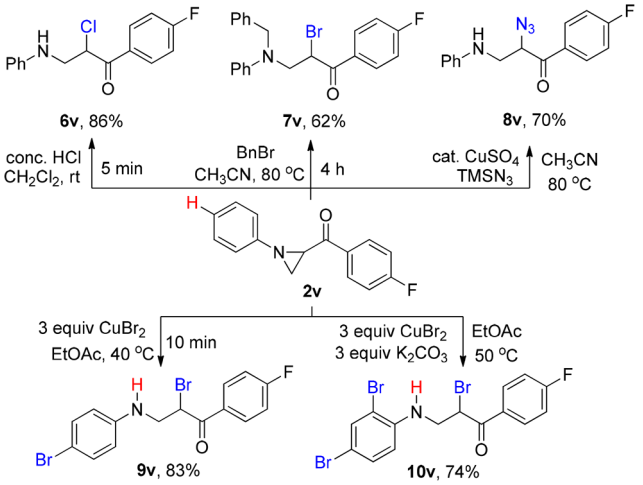


[a]Reaction conditions: a solution of 2-vinylpyridine (0.4 mmol), alkylamine (0.8 mmol), and $Yb(OTf)_3$ (0.02 mmol) in toluene (2 mL) was stirred for 0.5 h at 65 °C. Then DBU (120 μL, 0.8 mmol) and CuI (60 mg, 0.32 mmol) were added into the solution for 2 h under O_2 atmosphere. [b]The first step of the reaction was stirred for 2 h.

mono-, di-, and trisubstituents were all tolerable in this reaction. For example, phenethylamine **4a** formed **5a** in 71% yield. Benzylamine, hexylamine, 2-(benzyloxy)ethanamine, and 2-(thiophene-2-yl)ethanamine rendered the corresponding products in moderate yields (**5b**–**e**). Additionally, reactions with amines bearing five-, six-, or seven-membered rings resulted in moderate yields (**5f**–**h**). It is noteworthy that the reaction of more bulky bioactive amantadine proceeded smoothly as well (**5i**). Disubstituted 2-vinylpyridine 2-chloro-6-vinylnicotinonitrile could afford product **5j** in acceptable yield (55%). Besides azaarenes, 2,4-dinitrobenzene, as an activating moiety, produced the desired product **5k** in moderate yield.

The ring opening of aziridines played an important role toward the preparation of a large variety of *N*-containing compounds. However, the ring-opening studies of aziridine-2-ketones and their derivatives was very limited. To test the applicability of our methodology, the initially obtained aziridine **2v** was converted into a variety of highly functionalized ketones with high yield and regioselectivity. As depicted in Scheme 4, treating **2v** with HCl or benzyl bromide gave products **6v** and **7v** via C–N bond cleavage between nitrogen and carbon at the α-position of ketone. Additionally, azide **8v** was generated in good

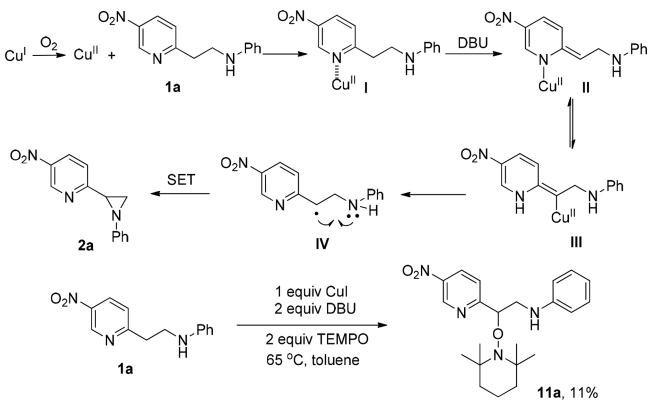
Scheme 4. Highly Regioselective Ring-Opening Reactions of 2v



yield via $CuSO_4$-catalyzed azidation. Interestingly, a tandem reaction occurred rapidly (10 min) to provide dibrominated **9v** in 83% yield. Bromination of arene may take place first to give 1 equiv of HBr, which then opened the aziridine ring. Moreover, the polybrominated product **10v** was obtained by prolonging the reaction time and adding K_2CO_3 as base.

Based on the above results, a possible reaction mechanism is proposed in Scheme 5. Initially, Cu^I was oxidized by oxygen to

Scheme 5. Proposed Mechanism and Mechanistic Experiments



Cu^{II}, which chelated with the substrate **1a** to generate the intermediate **I**. The Cu^{II} intermediate **II** was formed under basic conditions and converted to the intermediate **III**[18] and **IV**[19] in sequence. Finally, the radical intermediate **IV** underwent cyclization reaction via a single-electron transfer process affording the aziridine product **2a**. To validate the reaction mechanism, 2 equiv of 2,2,6,6-tetramethylpiperidine 1-oxyl was added into the reaction solution. No desired aziridine **2a** was found, and the coupling product **11a** was obtained in 11% yield. This result indicated that the radical intermediate **IV** was generated during the reaction.

In summary, an efficient copper-promoted C–H oxidative amination approach providing *N*-alkyl- and *N*-arylaziridines has been developed. The secondary amines containing azaarenes or ketones tolerated the reaction to give the aziridine products in moderate to good yields, and a practical $Yb(OTf)_3$ combined with CuI-promoted annulation of electron-deficient vinylarenes

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and alkylamines for the synthesis of *N*-alkylaziridines has been developed as well. This procedure proceeds via Michael addition and C−N direct dehydrogenative coupling with oxygen as the sole oxidant under mild conditions. We anticipate this new facile C−H oxidative amination for constructing aziridines will find a broad range of application in the synthesis of biologically active compounds.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b03799.

Experimental details and spectroscopic data (PDF)

Accession Codes

CCDC 1867799 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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(17) See the Supporting Information crystallographic data for compound **2g**.

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