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Oxidative Rearrangement via in Situ Generated *N*-Chloroamine: Synthesis of Fused Tetrahydroisoquinolines

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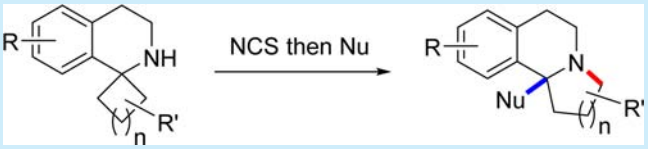
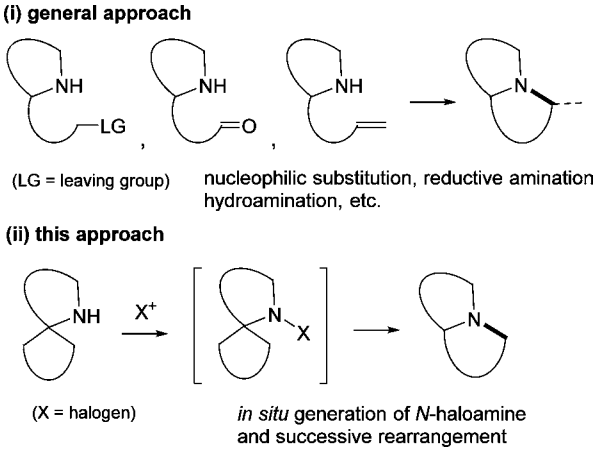
Supporting Information

ABSTRACT: An oxidative rearrangement reaction of spiro tetrahydroisoquinolines has been developed for the synthesis of fused tetrahydroisoquinolines using in situ generated *N*-chloroamines. The reaction proceeds via initial chlorination of an amine, followed by a 1,2-carbon to nitrogen migration, and nucleophilic trapping of a ketiminium ion intermediate in a one-pot operation. The electrophilic nature of *N*-chloroamines allowed for the carbon−nitrogen bond formation in this reation.



Fused aliphatic N-heterocycles can be found in a wide range of biologically active molecules, including natural products and pharmaceutical agents.[1] For the formation of the C−N bonds in aliphatic cyclic amines, the methods including intramolecular cyclization via nucleophilic substitution, reductive amination, and hydroamination, which utilizes nucleophilic nature of amines, are generally used (Scheme 1(i)).[2] Intramolecular C(sp^3)−H amination reactions have recently been developed as an alternative to these approaches.[3] However, the development of new and improved methods and strategies is still highly desired in organic synthesis. In particular, the development of a new method that does not require the prefunctionalization of the amino group or the use of expensive transition-metal reagents would represent a significant step forward in this area. Herein, we describe the development of an oxidative rearrangement reaction of spiro tetrahydroisoquinolines for the synthesis of fused tetrahydroisoquinolines, which can be found in a wide variety of biologically active natural products.[4] This method is characterized by carbon−nitrogen bond formations using in situ generated *N*-chloroamine as an electrophilic nitrogen source and ring expansion reaction via a 1,2-carbon to nitrogen migration (Scheme 1(ii)).

Scheme 1. General Approach and This Approach for Fused Cyclic Amines



N-Haloamines are one of the important classes of electrophilic nitrogen sources.[5] They are well utilized for the synthesis of nitrogen-containing molecules.[6] One of the key features of *N*-haloamines is their ease of preparation: They can be readily prepared by a treatment of an amine with a halogenating reagent.[7] It is therefore expected to use in situ generated *N*-haloamines directly as electrophilic nitrogen sources in a one-pot transformation without the need for the prefunctionalization of amines prior to the reaction.[8,9] However, despite considerable potential of *N*-haloamines, their uses in the 1,2-carbon to nitrogen migration reactions, which are powerful tools for the synthesis of nitrogen-containing molecules, have been less explored compared to the other electrophilic nitrogen sources such as hydroxyamine derivatives[10,11] and azides.[12] Although there have been several reports in the literature concerning the rearrangement reactions of *N*-chloroamines,[13] these transformations generally require the isolation of *N*-chloramines and the use of a stoichiometric amount of a silver salt, such as $AgBF_4$, to induce the rearrangement reaction. On the other hand, we have recently reported the halogen-induced oxidative rearrangements of *N*,*N*- and *N*,*O*-ketals, leading to bicyclic amidines and pyrrolidone derivatives.[14] In these reactions, in situ generated *N*-haloamines underwent rearrangement reaction without the use of silver salts due to the assistance of neighboring heteroatoms. These works demonstrated that suitable substrate selection enables the spontaneous rearrangement of *N*-haloamines under mild conditions.

Based on our previous observation, we herein developed a strategy for fused tetrahydroisoquinolines, as depicted in Scheme 2. It was envisaged that the reaction of the spiro cyclobutane tetrahydroisoquinoline **1** with halonium ion would lead to the formation of *N*-haloamine **i**. Based on the electrophilic nature of

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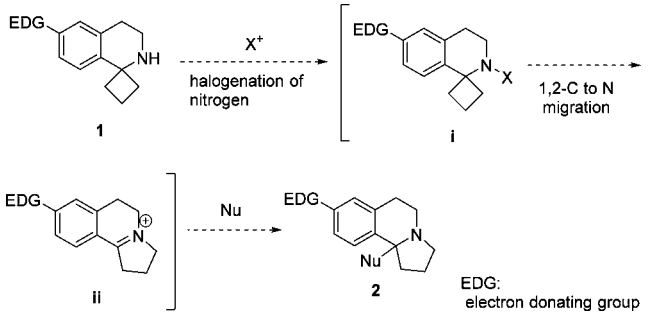
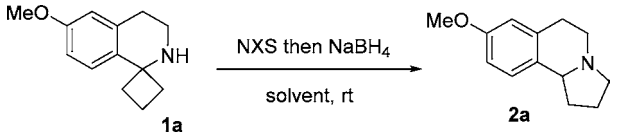
Scheme 2. Strategy for Fused Tetrahydroisoquinolines



the *N*-haloamine, it is expected that the 1,2-carbon to nitrogen migration would occur via the cleavage of a C−C bond to form a C−N bond. Also, in the reaction using *N*,*N*- and *N*,*O*-ketals, it is envisaged that the assistance of an electron-donating group on the aromatic ring would enhance the reaction. The resulting ketiminium ion intermediates **ii** could then be trapped by an appropriate nucleophile to provide the tricyclic fused tetrahydroisoquinoline **2**. Silver-salt-free conditions could allow for the use of various nucleophiles and consequently offer access to a diverse range of 2-substituted tetrahydroisoquinolines in a one-pot operation.

We first studied the reaction of **1a** (Table 1). After the screening of reaction conditions, we found that the N-

Table 1. Study of Reaction Conditions[a]



entry	NXS	solvent	result
1	NCS	MeOH	98%
2	NBS	MeOH	81%
3	NIS	MeOH	complex[b]
4	NCS	THF	no rearrangement[b]
5	NCS	CH_2Cl_2	no rearrangement[b]
6	NCS	CH_3CN	no reduction[b]

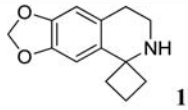
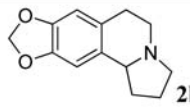
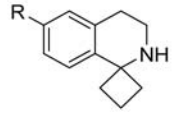
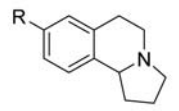
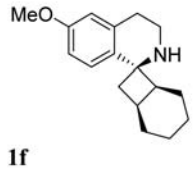
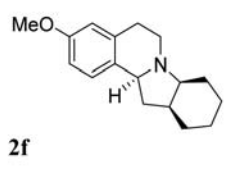
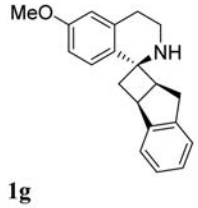
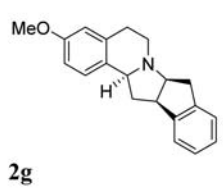
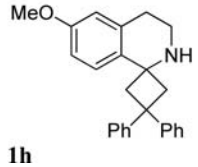
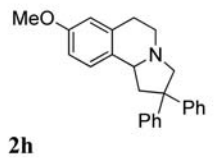
[a]Reaction conditions: NXS (1.1 equiv) in solvent (0.1 M) at 0 °C to rt then $NaBH_4$ (3.0 equiv) at rt. [b]See text for details.

chlorination of the nitrogen with *N*-chlorosuccinimide (NCS), followed by a reduction with $NaBH_4$ in MeOH, gave tricyclic compound **2a** in excellent yield (98%, entry 1). This reaction proceeded smoothly under mild conditions at room temperature, with the rearrangement reaction occurring in the absence of a silver salt. For comparison, we also investigated several conditions with other halosuccinimides (NXS) and solvents. With regard to the halogen source, *N*-bromosuccinimide (NBS) also mediated the reaction effectively but provided a lower yield compared with NCS (entry 2). In contrast, the use of *N*-iodosuccinimide (NIS) resulted in a complex mixture (entry 3). For the solvents, the use of THF or CH_2Cl_2 resulted in the failure of the rearrangement reaction, despite the formation of the *N*-chloroamine precursor (entries 4 and 5). CH_3CN was also found to be ineffective: In this case, the rearrangement reaction of the *N*-chloroamine was very slow and did not reach completion. In addition, the reduction of the ketiminium ion intermediate with $NaBH_4$ could not proceed to give the desired product (entry 6). Overall, these results show that the use of NCS with MeOH as a solvent provided the optimal conditions for conducting three reactions, including the halogenation of nitrogen, the rearrangement reaction, and the reduction of the ketiminium ion intermediate in a one-pot operation.

To gain insight into this transformation, we conducted the reaction in CD_3OD and monitored its progress by ^1H NMR spectroscopy. The results revealed that **1a** was completely converted to *N*-chloroamine **ia** within 15 min. Intermediate **ia** was then gradually converted to the ketiminium ion **iia** over 6 h. These observations suggest that between the N-chlorination and the migration, the later step was the rate-determining step of the oxidative rearrangement reaction. The structure of **iia** was also confirmed by ^{13}C NMR spectroscopy (for ^1H and ^{13}C NMR, see Supporting Information).

With the optimized conditions in hand, we investigated the scope of this transformation using **1b**−**1h** (Table 2). Similar to **1a**, substrate **1b** bearing a methylenedioxy group gave high yields of **2b** (entry 1). Even substrates **1c** and **1d** without an alkoxy group on the aromatic ring reacted smoothly to give the corresponding products **2c** and **2d** (entries 2 and 3). Substrate **1e**, bearing a free phenol group, was also tolerated to give **2e**

Table 2. Generality of Oxidative Rearrangement[a]

entry	substrate	product	yield (%)
1	 **1b**	 **2b**	96
2	 **1c** (R = Me)	 **2c** (R = Me)	91
3	**1d** (R = H)	**2d** (R = H)	59 (82)[c]
4	**1e** (R = OH)	**2e** (R = OH)	57
5[b]	 **1f**	 **2f**	77
6	 **1g**	 **2g**	75
7[c]	 **1h**	 **2h**	quant

[a]Reaction conditions: NCS (1.1 equiv) in MeOH (0.1 M) at 0 °C to rt then $NaBH_4$ (3.0 equiv) at rt. [b]At −78 °C. [c]Reaction conditions: NCS (1.1 equiv) in CF_3CH_2OH at rt, replacement of solvent (CF_3CH_2OH to MeOH), $NaBH_4$ (3.0 equiv) at rt.

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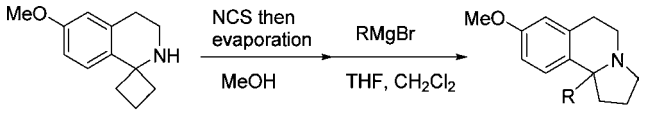
(entry 4). Polycyclic compounds **2f** and **2g** were also obtained from compounds **1f** and **1g** (entries 5 and 6). In these cases, the selective migration of secondary alkyl groups over the primary alkyl groups was expectedly observed. In contrast, substrate **1h** failed to undergo the rearrangement reaction. The lower reactivity of **1h** may be due to the decreased ring strain as a result of the Thorpe−Ingold effect by diphenyl substituents.[15] To overcome this reactivity problem, we investigated the solvent effect in the rearrangement reaction, based on our previous observation that CF_3CH_2OH dramatically accelerated the oxidative rearrangement of *N*,*N*-ketals.[14b] In CF_3CH_2OH, **1h** successfully underwent the oxidative rearrangement reaction to generate a ketiminium intermediate. The following reduction with $NaBH_4$ did not work in CF_3CH_2OH. However, it was found that the solvent can be replaced at this stage because the intermediate was stable even if solvent was removed in vacuo. After the solvent replacement to MeOH, the treatment with $NaBH_4$ then successfully gave **2h** quantitatively in a one-pot operation (entry 7). Identification of the solvent effect and the one-pot procedure was significant because it would broaden the scope of this transformation. Indeed, under the conditions using CF_3CH_2OH, the yield with **1d** was improved (entry 4) and the reaction with a norcamphor derivative **5** was achieved, as shown in Scheme 4.

Given that we had established the exchange of solvent at the ketiminium ion intermediate stage, it would be possible to select the appropriate solvent in accordance with the reagents. We then investigated the use of Grignard reagents as trapping agents for the ketiminium ion intermediate instead of $NaBH_4$ (Table 3).

Table 3. Reaction with Carbon Nucleophiles[a]



1a **3**

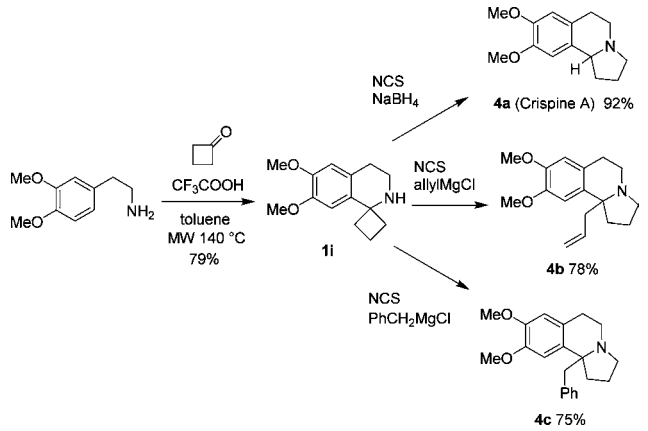
entry	RMgBr	product	yield (%)
1	allylMgBr	**3a** (R = allyl)	86
2	MeMgBr	**3b** (R = methyl)	55
3	EtMgBr	**3c** (R = ethyl)	87
4	*i*PrMgBr	**3d** (R = isopropyl)	84
5	vinylMgBr	**3e** (R = vinyl)	80
6	PhC≡CMgBr	**3f** (R = C≡CPh)	86

[a]Reaction conditions: NCS (1.1 equiv) in MeOH (0.1 M) at 0 °C to rt, replacement of solvent (MeOH to CH_2Cl_2), then RMgBr (10 equiv, THF solution) at 0 °C to rt.

Expectedly, the treatment with allylMgBr to the ketiminium ion intermediate generated from **1a** gave the allylated compound **3a** bearing a quaternary substituted carbon center in good yield (entry 1). In this transformation, C−N and C−C bonds were formed in a one-pot operation. With regard to the reaction solvent, a mixture of THF and CH_2Cl_2 was found to be more effective than THF alone, probably because of the lower solubility of the intermediate to ethereal solvents.[16] In addition to the allylMgBr, the other alkyl Grignard reagents, such as Me, Et, and even *i*PrMgBr, were available to give corresponding products **3b**−**3d** in good yields (entries 2−4). Furthermore, vinyl and phenyl acetylene were also introduced to the intermediate in a similar way (entries 5 and 6).

Oxidative rearrangement reactions of **1i** were also studied to synthesize a natural product, crispine A (**4a**),[17,18] which was isolated from *Carduus crispus* and has a significant cytotoxic activity, and its analogues (Scheme 3). The spiro cyclobutane **1i**

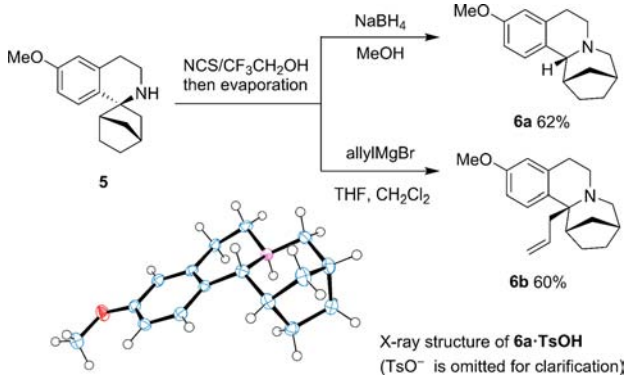
Scheme 3. Concise Synthesis Crispine A and Analogues



was readily prepared from commercially available 2-(3,4-dimethoxyphenyl)ethylamine. Following the procedures described above, the NCS-mediated rearrangement reaction and the treatment of $NaBH_4$ of **1i** expectedly provided **4a** in 92% yield. Similarly, treatment with allylMgCl or $PhCH_2MgCl$ successfully afforded crispine A analogues **4b** and **4c** in good yields. These results show that the developed method is useful for the synthesis of these classes of tetrahydroisoquinoline alkaloids and diverse analogues.

Finally, a norcamphor derivative **5** was applied to the oxidative rearrangement reaction (Scheme 4) because the bridged bicyclic

Scheme 4. Reaction of Norcamphor Derivative 5



structure of **5** could be promising for the rearrangement reaction as well as cyclobutanes and was expected to provide a complex skeleton.[19] Compound **5** was readily prepared from norcamphor in two steps via a Pictet−Spengler reaction.[20] Although the rearrangement reaction did not proceed in MeOH, the reaction in CF_3CH_2OH successfully proceeded to afford compound **6a** in good yield. The structure of **6a** was unambiguously confirmed by X-ray crystal analysis of its TsOH salt.[21] The use of allylMgBr for the trapping of the ketiminium ion gave compound **6b** bearing a quaternary substituted carbon center in a one-pot operation. The easy preparation of structurally unique compound **6b** highlights the effectiveness of this oxidative rearrangement reaction.

In summary, we have developed a novel oxidative rearrangement reaction for the preparation of fused tetrahydroisoquinolines that is triggered by the N-chlorination of spiro

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tetrahydroisoquinoline substrates. This reaction proceeds in three steps, including an N-chlorination, 1,2-carbon to nitrogen migration, and nucleophilic trapping of a generated ketiminium ion intermediate in a one-pot operation. The ketiminium ion intermediates can be trapped with both hydride and carbon nucleophiles. This method will offer efficient access to a wide range of fused tetrahydroisoquinolines, which are important skeletons that can be found in various 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.6b00233.

Experimental procedures and full characterization of all compounds (PDF)

X-ray crystal structural data for the TsOH salt of **6a** (CIF)

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Notes

The authors declare no competing financial interest.

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(21) CCDC 1413456 (**6a**·TsOH) contains the supplementary crystallographic data for this paper. 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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