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**Authors:** Jongwoo Son, Tyler W Reidl, Ki Hwan Kim, Donald J Wink, and Laura L Anderson



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COMMUNICATION

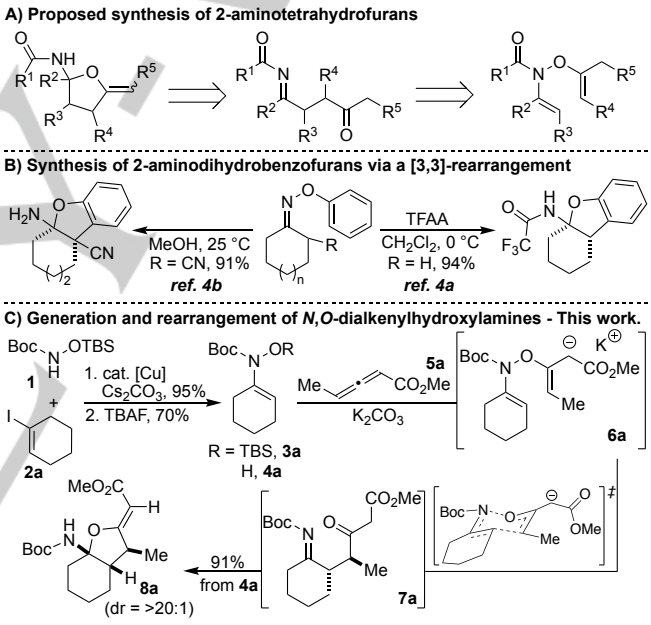
# Generation and Rearrangement of *N,O*-Dialkenylhydroxylamines for the Synthesis of 2-Aminotetrahydrofurans

Jongwoo Son,[+] Tyler W. Reidl,[+] Ki Hwan Kim, Donald J. Wink, and Laura L. Anderson*[a]

Dedication ((optional))

**Abstract:** A new diastereoselective route to 2-aminotetrahydrofurans has been developed from *N*,*O*-dialkenylhydroxylamines. These intermediates undergo a spontaneous C–C bond-forming [3,3]-sigmatropic rearrangement followed by a C–O bond-forming cyclization. A copper-catalyzed *N*-alkenylation of an *N*-Boc-hydroxylamine with alkenyl iodides, and a base-promoted addition of the resulting *N*-hydroxyenamines to an electron-deficient allene, provide modular access to these novel rearrangement precursors. The scope of this de novo synthesis of simple nucleoside analogues has been explored to reveal trends in diastereoselectivity and reactivity. In addition, a base-promoted ring-opening and Mannich reaction has been discovered to covert 2-aminotetrahydrofurans to cyclopentyl β-aminoacid derivatives or cyclopentenones.

Tetrahydrofurans are found in a variety of natural products and biologically active molecules.[1] Due to the privileged nature of these heterocycles, many methods have been developed to access these important scaffolds.[2] One strategy that has not yet been explored for the synthesis of tetrahydrofurans is the [3,3]-sigmatropic rearrangement of *N*,*O*-dialkenylhydroxylamines followed by cyclization of the initial rearrangement product (Scheme 1A).[3] This approach is appealing because of the potential to simultaneously set several contiguous stereocenters in the tetrahydrofuran ring and the latent modularity of the precursor. Analogous transformations have been reported for the synthesis of dihydrobenzofurans from *O*-aryloxime ethers, which are easily prepared via oxime arylation or condensation of the corresponding *O*-arylhydroxylamine with the appropriate ketone.[4,5] In contrast, the tetrahydrofuran synthesis has remained elusive because of the difficulty in generating and controlling the reactivity of *N*,*O*-dialkenylhydroxylamines (Scheme 1B). Due to our interest in advancing the reactivity of unsaturated hydroxylamines and nitrones, we wondered if we could leverage our understanding of oxime and hydroxylamine alkenylation to design a modular route to *N*,*O*-dialkenylhydroxylamines.[6] Herein we describe the synthesis of 2-aminotetrahydrofurans such as **8a** with three contiguous carbon stereocenters through a copper-catalyzed *N*-alkenylation of *N*-Boc-siloxyamine **1**, followed by generation of *N*,*O*-dialkenyl-hydroxylamine **6a** via deprotection of **3a** and addition of **4a** to allene **5a**, which precipitates a spontaneous [3,3]-rearrangement and cyclization sequence (Scheme 1C). The 2-amino-tetrahydrofuran products that are accessible by this method are highly-substituted and deoxygenated nucleoside analogues with a simple carbamate base. Since nucleoside analogues are known to be potent bioactive reagents, the development of this new approach to these types of structures will assist in expanding the chemical space around these important compounds, while further advancing de novo strategies for the synthesis of furanosides.[7-9]




**Scheme 1.** Synthesis of 2-Aminodihydrobenzofurans and 2-Aminotetrahydrofurans via [3,3]-Sigmatropic Rearrangements. TFAA = trifluoroacetic anhydride, TBS = *t*-butyldimethylsilyl, Boc = *t*-butyloxycarbonyl, TBAF = tetrabutylammonium fluoride.

We initiated our studies towards the generation of *N*,*O*-dialkenylhydroxylamines **6** for the synthesis of 2-amino-tetrahydrofurans **8** through the development of conditions for the *N*-alkenylation of *N*-Boc-siloxyamine **1**. Using previous studies of hydrazodiformates as a starting point, copper-catalyzed conditions for the *N*-alkenylation of **1** were optimized and the reaction was determined to work best in the presence of 10 mol % CuI and DMEDA (*N*,*N*'-dimethylethylenediamine) (Scheme 2).[10,11] The scope of the synthesis of **3** was then investigated and shown to work well for both cyclic and linear *E*-alkenyliodides **2**. Cyclohexenyl- and cycloheptenyl *N*-siloxyenamines **3a** and **3b** were prepared in excellent yield and acetal-substituted cyclohexenyl enamine **3c** and β-tetralone-derived **3d** were also

[a] J. Son, T. W. Reidl, K. H. Kim, Dr. D. J. Wink, Prof. L. L. Anderson
Department of Chemistry
University of Illinois at Chicago
845 W Taylor St., Chicago, IL USA
E-mail: lauralin@uic.edu

[+] These authors contributed equally to this work.

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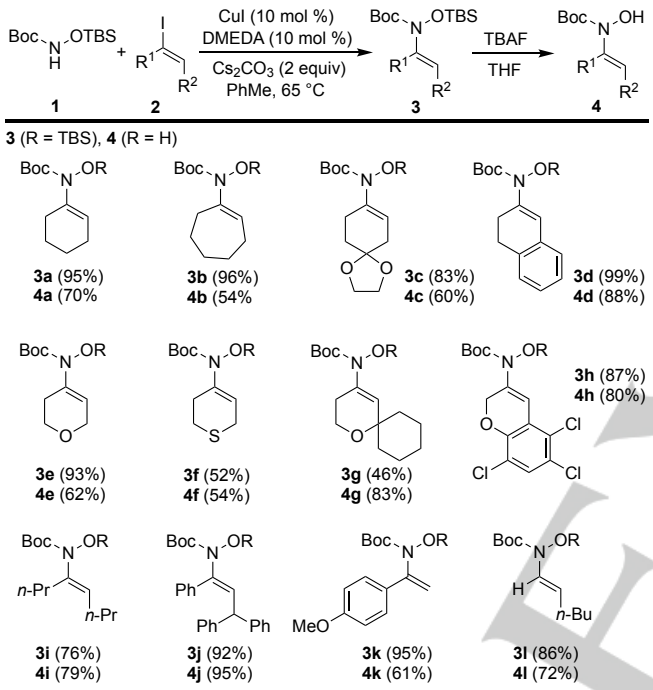
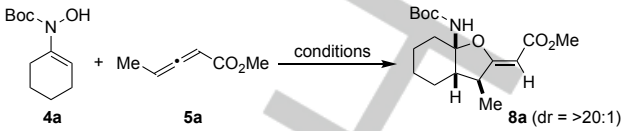
shown to be easily accessible. Heterocyclic alkenyl iodides were tolerated by the transformation for the preparation of *N*-siloxyenamines **3e** and **3f**, as well as **3g** and **3h** with more complicated substitution patterns. *E*-Disubstituted linear alkenyl iodides were used to form **3i** and **3j**; however, *Z*-4-octenyl iodide was unreactive. Monosubstituted alkenyl iodides were also tolerated with substitution at either the α- or β-position to give **3k** and **3l**. Silyl ether deprotection and conversion of **3** to **4** was achieved for all of the substrates shown in Scheme 2 using TBAF. The range of *N*-hydroxyenamines prepared in Scheme 2 was then used to facilitate our further study of *N*,*O*-dialkenylhydroxylamine generation and 2-aminotetrahydrofuran synthesis.




**Scheme 2.** *N*-Boc-Hydroxylamine Silyl Ether *N*-Alkenylation and Deprotection.

Conditions for the generation of *N*,*O*-dialkenylhydroxylamines **6** and their conversion to 2-aminotetrahydrofurans **8** were initially tested with *N*-hydroxyenamine **4a** and allenoate **5a**. As shown in Table 1, treatment of **4a** with **5a** in the absence of a base resulted in no reaction but when these reagents were combined in the presence of $Cs_2CO_3$, rapid formation of **8a** was observed with high diastereoselectivity (Table 1, entries 1 – 2). The favored diastereomer was identified as the *cis*-fused ring system through X-ray crystallography.[12] A solvent screen indicated that $CH_2Cl_2$ was the optimal solvent for this transformation and subsequent screening of similar carbonate and phosphate bases showed that $K_2CO_3$ gave the highest yield of **8a** (Table 1, entries 2 – 9).[13] The use of 20 mol % $K_2CO_3$ gave the desired product in attenuated yield and the addition and rearrangement reaction was shown to be sensitive to reaction time and temperature with the optimal conditions determined to be 30 min at 0 °C (Table 1, entries 6 and 10 – 12).[14] These conditions were used to investigate the scope of this transformation using *N*-hydroxyenamines **4**.

**Table 1.** Optimization of Addition and Rearrangement Reaction




| Entry[a] | Base | Solvent | T (°C) | t (h) | % Yield[b] |
|---|---|---|---|---|---|
| 1 | none | $CH_2Cl_2$ | 0 | 1 | nr |
| 2 | $Cs_2CO_3$ (1 equiv) | $CH_2Cl_2$ | 0 | 1 | 57 |
| 3 | $Cs_2CO_3$ (1 equiv) | PhMe | 0 | 1 | 55 |
| 4 | $Cs_2CO_3$ (1 equiv) | MeCN | 0 | 1 | 38 |
| 5 | $Cs_2CO_3$ (1 equiv) | THF | 0 | 1 | 50 |
| 6 | $Cs_2CO_3$ (1 equiv) | $CH_2Cl_2$ | 25 | 1 | 29 |
| 7 | $K_2CO_3$ (1 equiv) | $CH_2Cl_2$ | 0 | 1 | 83 |
| 8 | $Li_2CO_3$ (1 equiv) | $CH_2Cl_2$ | 0 | 1 | 54 |
| 9 | $K_3PO_4$ | $CH_2Cl_2$ | 0 | 1 | nr |
| 10 | $K_2CO_3$ (20 mol %) | $CH_2Cl_2$ | 0 | 1 | 69 |
| 11 | $K_2CO_3$ (1 equiv) | $CH_2Cl_2$ | 0 | 2 | 80 |
| 12 | $K_2CO_3$ (1 equiv) | $CH_2Cl_2$ | 0 | 0.5 | 93 (91)[c] |

[a] Conditions: **4a** (1 equiv), **5a** (2 equiv), 0.1 M in solvent. [b] % Yield determined by $^1$H NMR spectroscopy using $CH_2Br_2$ as a reference. [c] Isolated yield.

*N*-Hydroxyenamines **4** were paired with electron-deficient allenes **5** to explore the scope of the synthesis of 2-aminotetrahydrofurans **8**. As shown in Scheme 3, *N*-hydroxyenamines **4** with cycloalkenyl or heterocyclic *N*-substituents underwent the addition and rearrangement to give **8a** – **8c** and **8e** – **8g** in good to excellent yield with high diastereoselectivity. In comparison, the facial selectivity for the formation of **8d** and **8h** were attenuated, which may be caused by the increased planarity of the starting materials. Substrates **4i** – **4l** with linear alkenyl substituents gave good yields of the corresponding 2-aminotetrahydrofurans, but low to moderate diastereoselectivity for the formation of **8i** – **8l**. Minor diastereomers of **8d** and **8h** – **8l** were determined to have inverted stereochemistry at the hemiaminal ether.[15] Within this set of substrates, it was observed that changing the size and shape of the *N*-alkenyl group from **4i** to **4j** resulted in an increase in diastereomeric ratio between **8i** and **8j**. For monosubstituted *N*-alkenyl substrates **4k** and **4l**, the substituent at the β-position had a greater influence on the diastereoselectivity of the 2-aminotetrahydrofuran synthesis. Further investigation of the method involved varying the electron-withdrawing and alkyl substituents on the allene. As shown for **8m** – **8p**, several alkylated allenoates are tolerated for the transformation. All

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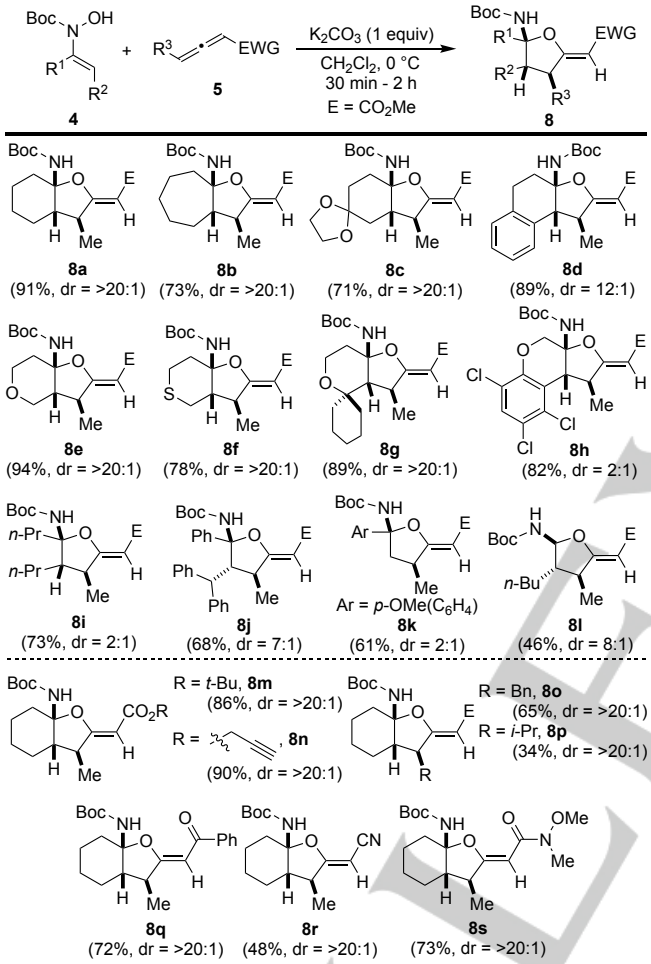
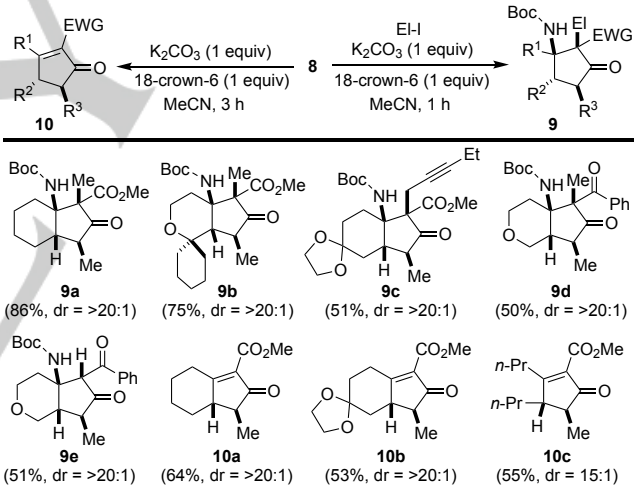
methyl-substituted allenoate addition and rearrangement products were obtained in high yield (see **8m** – **8n**); however, as the size of the alkyl substituent increased, the yield of the addition and rearrangement reaction decreased (see **8o** – **8p**).[16] The use of cyano-substituted allene **5g** resulted in a moderate yield of **8r**, but ketone- and Weinreb amide-substituted allenes **5f** and **5h** were well-tolerated for the formation of **8q** and **8s**, respectively. These studies illustrate the scope of the addition and rearrangement reaction to provide a small library of new 2-aminotetrahydrofurans **8** for reactivity studies.




**Scheme 3.** Scope of *N*,*O*-Dialkenylhydroxylamine Generation and 2-Aminotetrahydrofuran Synthesis.

The development of mild conditions for the generation of *N*,*O*-dialkenylhydroxyamine **6**, which initiated a spontaneous rearrangement and cyclization sequence to form 2-aminotetrahydrofurans **8**, provided an opportunity to explore the unknown rearrangement reactivity of these novel heterocycles. As expected, exposure of **8a** to acidic conditions resulted in pyrrole formation.[17] In contrast, we anticipated that mild basic conditions might cleave the tetrahydrofuran C–O bond and convert **8** to highly-substituted cyclopentanone products via a formal aza-Petasis-Ferrier rearrangement.[18] Gratifyingly, treatment of **8a** with MeI in the presence of $K_2CO_3$, resulted in tetrahydrofuran ring-opening, alkylation, and Mannich addition to form β-amino-cyclopentenone **9a** with high diastereoselectivity (Scheme 4). The preferred conformation of this product was confirmed by X-ray crystallography.[12] This transformation was consistent for other 2-aminotetrahydrofurans as shown for **9b** and **9d**, as well as other electrophiles as shown for **9c**.[19] In the absence of an electrophile, an adventitious proton source gave **9e**. Cyclopentyl β-amino acid derivatives such as **9** are important scaffolds in a variety of biologically active molecules and this new route to highly substituted examples from 2-aminotetrahydrofurans **8** will help to broaden the accessibility of these privileged compounds.[20] Under longer reaction times, and in the absence of an electrophile, the base-promoted tetrahydrofuran ring-opening and Mannich cyclization was also observed to occur with an elimination process to give cyclopentenones **10**. These transformations were similarly diastereoselective as shown for **10a** and **10b**. The conversion of **8i** to **10c** also resulted in an increase in the diastereomeric ratio of the product in comparison to the starting material.[21] Cyclopentenones **10** have the opposite electronic substitution pattern to Nazarov rearrangement products and provide an opportunity to continue to expand structural diversity around this structure for synthetic applications.[22]




**Scheme 4.** Ring-Opening of **8** and Mannich Ring-Closure Under Basic Conditions.

In summary, by targeting the generation of *N*,*O*-dialkenylhydroxylamines under mild conditions, we have developed a new modular route to 2-aminotetrahydrofurans. A copper-catalyzed *N*-alkenylation of *N*-Boc-siloxyamine **1** has been optimized for the synthesis of *N*-siloxyenamines **3**. These intermediates have been shown to readily undergo silyl deprotection and deprotonation for the addition to electron-deficient allenes **5** and the generation of *N*,*O*-dialkenyl-hydroxylamines **6**. These intermediates then undergo a spontaneous [3,3]-sigmatropic rearrangement and cyclization to form 2-aminotetrahydrofurans **8**. Taken together, these results provide a new, novel, and diastereoselective route to simple nucleoside analogues from modular precursors. In addition, this

study has also lead to the discovery that 2-aminotetrahydrofurans can be used to access cyclopentyl β-amino acid derivatives and cyclopentenones under basic conditions.

## Acknowledgements

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**Keywords:** sigmatropic rearrangement • allenoate • hydroxylamine • tetrahydrofuran • aza-Petasis-Ferrier

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[12] CCDC 1814362, and CCDC 1814364 contain the supplementary crystallographic data for compounds **8a** and **9a**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

[13] DABCO and DBU were also tested as bases for the addition of **4a** to **5a** but gave **8a** in less than 25% yield. See Supporting Information for an expanded Table 1.

[14] At longer reaction times and higher temperatures, treatment of **4a** with **5a** in the presence of $K_2CO_3$ gave competing formation of a pyrrolidine. Formation of **9a** or **10a** was not observed. See Supporting Information.

[15] See Supporting Information for details.

[16] Treatment of **4a** with methyl 2,3-butadienoate favoured formation of a pyrrolidine. See Supporting Information for details.

[17] See Supporting Information for the conversion of **8a** to a pyrrole.

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[19] Formation of **9** or **10** as byproducts was not observed under the reaction conditions described in Scheme 3 for **4a** – **4l**. One example of **4** with an aryl substituent at the β-position was observed to give **9** when treated with **5a** in low yield. See Supporting Information for details.

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[21] This observation is consistent with the assignment of the minor diastereomer of **4d** and **4h** – **4l** exhibiting inversion at the hemiaminal ether stereocenter. Conversion of **8d** and **8h** to corresponding **9** as single diastereomers was also observed. See Supporting Information.

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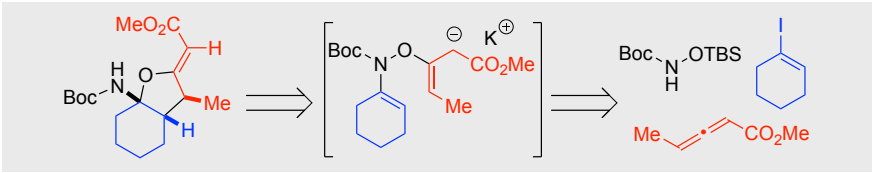


A new approach to the synthesis of 2-aminotetrahydrofurans has been designed using an *N,O*-dialkenylhydroxylamine precursor, which undergoes a [3,3]-rearrangement and cyclization cascade sequence to form the desired heterocycle. This new route provides diastereoselective access to highly-substituted and deoxygenated nucleoside analogues with a simple carbamate base from *N*-Boc-hydroxylamine, alkenyl iodides, and electron-deficient allenes.

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