

Stereoselective Oxidative Cyclization of *N*-Allyl Benzamides to Oxaz(ol)ines

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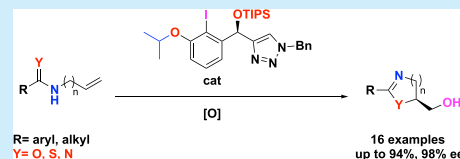


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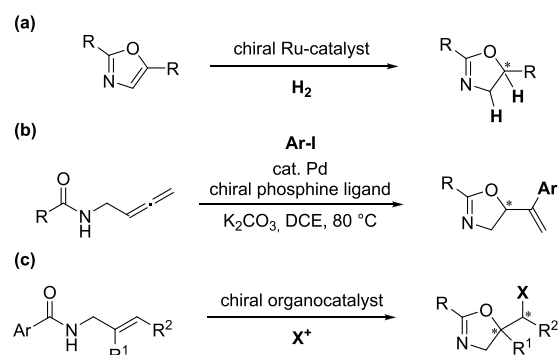
ABSTRACT: This study presents an enantioselective oxidative cyclization of *N*-allyl carboxamides via a chiral triazole-substituted iodoarene catalyst. The method allows the synthesis of highly enantioenriched oxazolines and oxazines, with yields of up to 94% and enantioselectivities of up to 98% ee. Quaternary stereocenters can be constructed and, besides *N*-allyl amides, the corresponding thioamides and imideamides are well tolerated as substrates, giving rise to a plethora of chiral 5-membered *N*-heterocycles. By applying a multitude of further functionalizations, we finally demonstrate the high value of the observed chiral heterocycles as strategic intermediates for the synthesis of other enantioenriched target structures.



Partially hydrogenated *N*-heterocycles (azolines), in particular 2-oxazolines, 2-thiazolines, and 2-imidazolines, are important synthetic targets, not only due to their abundance in biologically active compounds^{1,2} but also due to their high value as useful synthetic building blocks.^{3–5} Enantiopure derivatives substituted at C4, at C5, or at both saturated carbons are of particular importance due to the stereochemical requirements of the desired products or the chiral building blocks made by them. Enantiopure 4-oxazolines are also widely applied as core structural motifs, for example in chiral ligands for enantioselective transition-metal-catalyzed reactions.⁶

Enantiopure 5-oxazolines are found in biologically active compounds such as shahidine—the parent compound of the strong antioxidant aegilin,⁷ nagelamide alkaloids,⁸ and the tubulin-binder A289099 (Figure 1).² While synthetic approaches for 4- and 4,5-disubstituted chiral 2-azolines are well established,^{4,5,9} the synthesis of enantiopure monosubstituted 5-azolines is underdeveloped (Scheme 1).^{10,11} The latter can

Scheme 1. Known Approaches for the Synthesis of 5-Oxazolines



be synthesized by Ru-catalyzed hydrogenations of oxazoles (Scheme 1a)¹² or by Pd-catalyzed cyclizations of allenyl-substituted aryl amides (Scheme 1b).¹³ Organocatalytic approaches have also been established whereby a hydrogen-bonding donor in the presence of a halonium source results in enantioselective halocyclizations of *N*-allyl amides (NAAs, Scheme 1c).¹⁴

A more general approach for the cyclization of NAAs involves their treatment with a chiral hypervalent iodine compound. This induces an oxidative cyclization via a π -complexed iodane **A**, similar to halonium sources. Iodine(III) activates π -complexed NAA to produce **B** through an intramolecular attack by the amide oxygen (Scheme 2). The

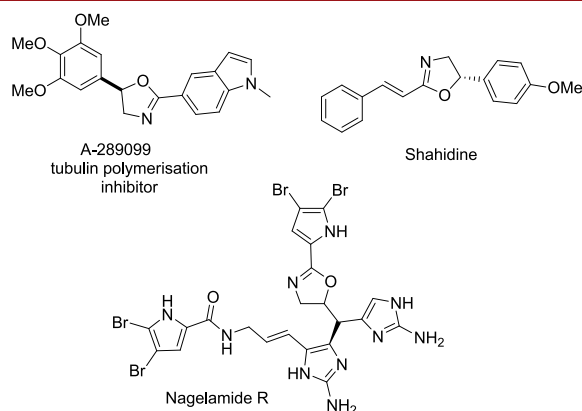
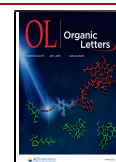
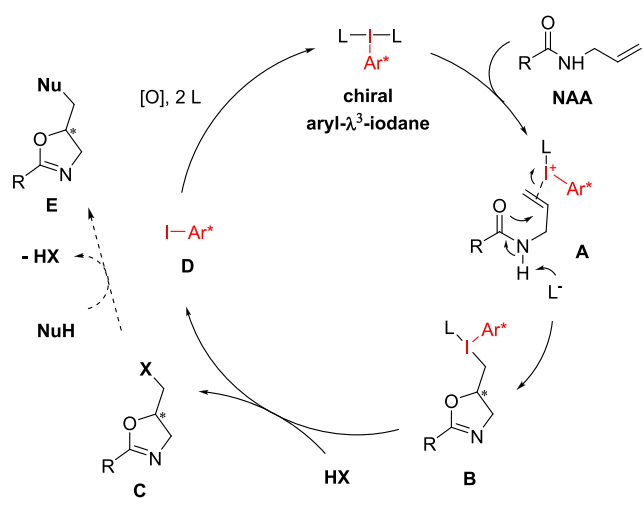


Figure 1. Examples of natural products containing a chiral C5-substituted oxazoline unit.

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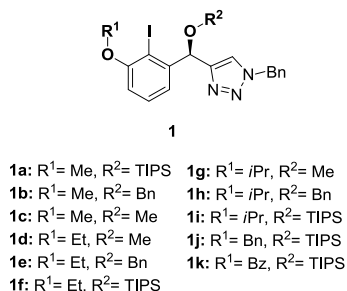
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Scheme 2. General Mechanism for an Iodane-Mediated *N*-Oxidative Cyclization of NAAs

emerging alkyl-substituted iodane **B** then reacts rapidly with additives, usually added Brønsted acids **HX**, to form compound **C** under reduction of the iodane to the iodoarene **D**. Depending on the nature of **C**, rapid substitution often follows with external nucleophiles **NuH**, for example water or hydroxide anions, to generate the substituted oxazoline **E**. The chiral aryl iodide can then be reoxidized by an external oxidant and thus can be used in catalytic amounts.^{15–17} This general method has been applied by Moran and co-workers using a well-established resorcinol-based chiral iodoarene catalyst,^{18–20} but unfortunately with only moderate results, particularly regarding stereoselectivity and substrate scope, with a focus on 6-membered *N*-heterocycles.¹¹

Our group recently established chiral triazole-substituted iodoarenes **1**, where the triazole has a role as a direct stabilizing donor of the hypervalent iodine center through dative N–I interaction, and applied them in a wide range of enantioselective oxidative transformations (Figure 2).^{16,17,21} In this letter we report their further application to the as-yet underexplored oxidative cyclization of *N*-allyl amides.

Figure 2. Structure of the chiral triazole catalysts **1**.

We started our investigations using *N*-allyl benzamide, **2a**, as the model substrate (Table 1). It was established in our early studies that an additional substituent ortho to the central iodine atom is crucial for high reactivity and stereoselectivity in reactions performed with these catalysts. Therefore, we started with the *o*-OMe-substituted iodoarene catalyst **1a**, the most successful to be used to date. During a preliminary optimization we had already established acetonitrile as the best solvent for this reaction, in combination with selectfluor as

Table 1. Optimization of the Reaction Conditions

entry ^a	catalyst	yield [%]	ee [%]
1	1a	67	85
2	1b	61	74
3	1c	62	64
4	1d	72	67
5	1e	71	74
6	1f	75	87
7	1g	83	70
8	1h	81	85
9	1i	91	93
10	1j	68	84
11	1k	80	84
12 ^b	1i	65	93
13 ^c	1i	48	92

^aReaction conditions: **2a** (0.40 mmol, 1.00 equiv), cat (0.04 mmol, 10 mol %), TFA (0.60 mmol, 1.50 equiv), selectfluor (0.40 mmol, 1.00 equiv), CH₃CN (0.12 M). ^b5 mol % of **1i** was used. ^cThe reaction was performed at 0 °C and took 36 h with 70% conversion of **2a**.

a co-oxidant and TFA as an acid additive. While using catalyst **1a**, the 5-substituted oxazoline **3a** was isolated with a 67% yield and 85% ee. Under these conditions, a fluorinated intermediate was not detected via MS. However, formation of the corresponding methyl 2,2,2-trifluoroacetate derivative of **C** (Scheme 2, X = OTFA) is likely since the reaction must be quenched with aq. NaOH, to give **3a** as the final reaction product.

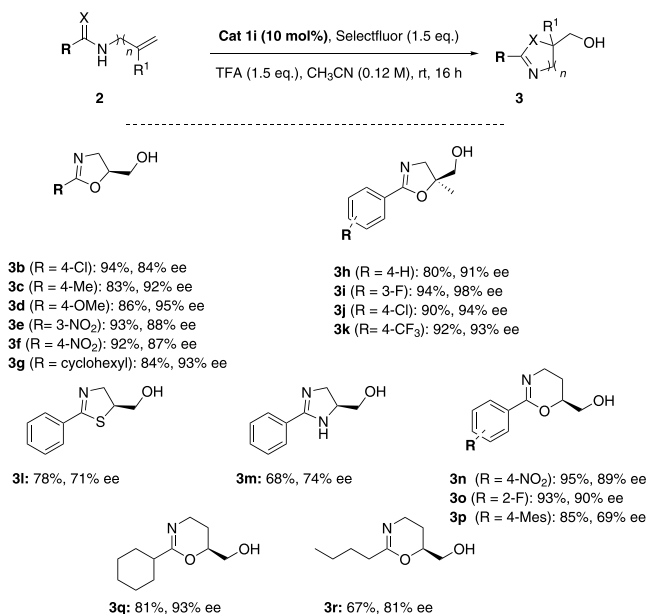
We then systematically investigated the influence of both ether substituents (R¹ and R²) in catalysts **1** on the reaction performance. Switching the TIPS group to other alkyl groups, such as catalysts **1b–1e**, was found to be counterproductive and resulted in diminished enantioselectivities (see entries 2–5). We also varied the alkyl ether at R¹ and decided to introduce greater sterical bulk at this position. With R² being TIPS again, replacement of the methyl with an ethyl ether for R¹ (catalyst **1f**) resulted in a small but less than significant improvement of the enantioselectivity.

Introduction of an isopropyl group (catalyst **1i**) resulted in the most significant improvements, as **3a** was now isolated with a 91% yield and 93% ee. By contrast, the corresponding use of benzyl ether (**1j**) or benzoyl ester (**1k**) was less effective (see entries 10 and 11).

Decreasing the catalyst loading to 5 mol % had a negative effect on the yield. Reduction of the reaction time or performing the reaction at 0 °C did not improve the enantioselectivity but only led to lower reaction rates and incomplete conversions (see entries 12 and 13). Increasing the amount of the additive TFA had no effect on the yield of compound **3a**.

With the optimized conditions in hand, we elaborated the substrate scope of the oxidative cyclization (Scheme 3). The NAA derivatives **2b–f**, with additional substituents at the 2-aryl group, provided the corresponding oxazolines **3b–f** in yields and enantioselectivities comparable to those of the parent compound **3a** (84–95%), regardless of the electronic nature of the substituent. Comparison of the rotary power of the 4-nitro derivative **3f** with a known literature value allowed

Scheme 3. Substrate Scope



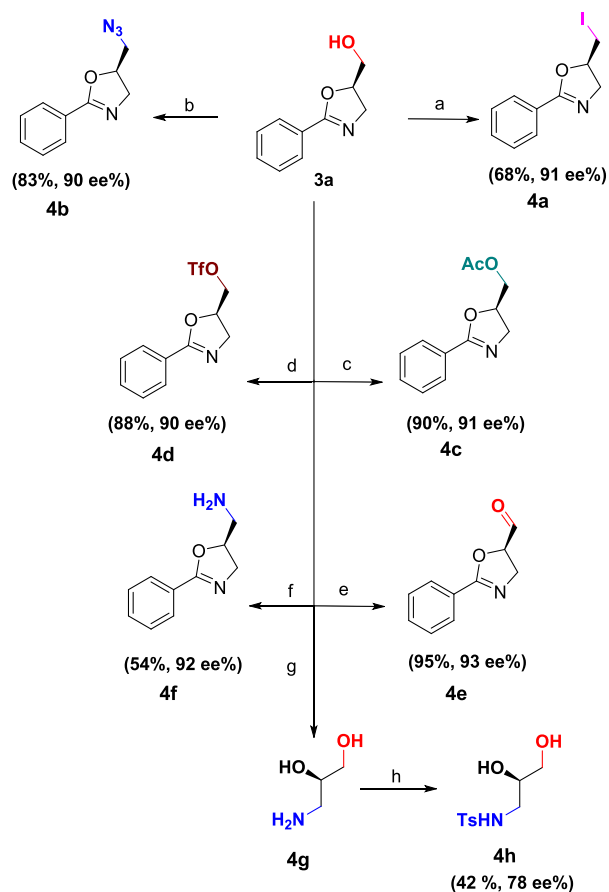
us to determine the absolute configuration of the products to be (S).²² Cyclic aliphatic substituents (R = cyclohexyl) were tolerated as well, giving **3g** with an 84% yield and 93% ee.

Next, we wondered whether quaternary stereocenters could be constructed.²³ We therefore decided to apply our method for the synthesis of quaternary 2-oxazolines starting from various *N*-(but-3-en-2-yl) benzamides **2h**–**2k** (R¹ = Me, R² = H). The desired products **3h**–**3k** could be observed in high yields of up to 94% and excellent enantioselectivities (91–98% ee). The enantioenriched thiazoline **3l** and the imidazoline **3m** were prepared for the first time by this method, although yields and enantioselectivities were significantly lower in direct comparison with their oxa derivatives (71% ee and 74% ee). It is nonetheless worth mentioning that these *N*-heterocycles are very important core structural motifs found in many biologically active compounds and this method provides a to date undescribed means of accessing them.²⁴

We subsequently focused on the synthesis of 6-membered *N*-heterocycles. Application of *N*-homoallyl benzamide **2n** and **2o** (*n* = 2) provided the oxazines **3n** and **3o** in yields of 95% and 93% and enantioselectivities of 89% ee and 90% ee, respectively. The mesityl-substituted derivative gave the corresponding oxazine **3p** in comparable yields but with a diminished enantioselectivity (69% ee).

Aliphatic homoallyl amides **2q** and **2r** were subsequently investigated, giving the desired 2-cyclohexyl- and 2-*n*-butyl-substituted derivatives **3q** and **3r** in respective yields of 81% and 67% and enantioselectivities of 93% and 81% ee for each. Applications of similar compounds have been reported for the preparation of poly(2-oxazoline) gels for delayed drug delivery systems.²⁵ Again, our method provides unique access to these enantioenriched *N*-heterocycles.

Since 5-oxazolines are potentially useful intermediates for the synthesis of other chiral building blocks, we finally elaborated further synthetic transformations of **3a** (Scheme 4). The OH group could be replaced by iodine under Mitsunobu conditions to afford **4a** in 68% yield without a significant loss of enantiomeric excess.²⁶ Azidation was achieved using a method devised by Kumar and co-workers,

Scheme 4. Derivatization of Oxazoline **3a**^a

^aReaction conditions: (a) I₂ (1.20 equiv), PPh₃ (1.30 equiv), Pyridine (0.95 M), rt, 24 h. (b) NaN₃ (3.00 equiv), BF₃·Et₂O (1.50 equiv) Dioxane, rt, 24 h. (c) Acetic anhydride (2.00 equiv) DCM, rt, 4 h. (d) TF₂O (1.20 equiv), Pyridine (1.10 equiv), DCM, rt, 16 h. (e) MnO₂ (12.0 equiv), CHCl₃, 60 °C, 4 h. (f) 1-Phthalimide (1.10 equiv), PPh₃ (1.10 equiv), DIAD (1.30 equiv), THF (0.42 M), rt, 7 h, 2-Hydrazine (1.50 equiv), EtOH (5 mL), 80 °C, 0.5 h. (g) 1-NaBH₄ (1.20 equiv), I₂ (1.00 equiv), 2- HCl in MeOH, 24 h. (h) TsCl (1.20 equiv) MeCN, rt, 6 h.

treating **3a** with NaN₃ and BF₃·Et₂O to give **4b** in 83% yield and 90% ee.²⁷ Protection of the OH group to the corresponding acetates and triflates (**4c** and **4d**) was successful as well. In addition, the corresponding aldehyde could be obtained by treatment of **3a** with MnO₂, to give **4e** in 95% yield and 93% ee. Interestingly, no overoxidation of the oxazoline was observed. A Mitsunobu reaction was also utilized to prepare the primary amine **4f** in a moderate yield (54%) but sustaining the stereochemistry (92% ee).²⁸ Lastly, we prepared 3-aminopropane-1,2-diol by a reduction of **3a** followed by acid-mediated ring opening to give the amino diol **4g**. Since this compound could not be analyzed by HPLC, it was directly transformed into the *N*-tosylated derivative **4h**. **4h** was isolated in 42% yield but with a diminished selectivity of 78% ee. Racemization of **4h** could occur via intermediate oxirane formation by intramolecular attack of an activated form of the chiral secondary alcohol by the primary alcohol and a subsequent terminal ring opening by water.

In summary, we have established a practical method for the enantioselective oxidative cyclization of *N*-allyl amides by using an improved triazole-substituted iodoarene catalyst.²⁹ This

method is characterized by a broad substrate scope which allows the construction of highly enantioenriched 5-oxazolines, thiazolines, and imidazolines. Quaternary stereocenters can be constructed with high efficiency as well, and the method was further extended to oxazines. Many of the constructed compounds can serve as chiral building blocks for the synthesis of interesting chiral target structures. This was demonstrated in a variety of further functionalizations, in particular of the terminal OH group.

In further investigations, we aim to apply C1-symmetric triazole-based iodoarenes in similar oxidative cyclizations to generate other useful 5- and 6-membered heterocycles. Additionally, cascade reactions in which the reactive hyper-valent iodine intermediate is trapped directly by nucleophiles other than OH will also be part of future investigations.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.orglett.1c01607>.

All experimental procedures and characterization data (PDF)

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Author Contributions

The manuscript was written by B.J.N. and A.H.A. A.H.A. and T.-N.H. performed the experiments.

Notes

The authors declare no competing financial interest.

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