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TMSOTf-mediated approach to 1,3-oxazin-2-one skeleton through one-pot successive reduction-[4 + 2] cyclization process of imides with ynamides

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ABSTRACT

A one-pot approach to access functionalized 1,3-oxazin-2-one skeleton has been developed through successive reduction and subsequent [4 + 2] cyclization process of *N*-Boc lactams with ynamides by TMSOTf. As a result, a number of five to seven membered ring fused bicyclic [1,2-c][1,3]oxazin-1-ones **12a-m** and tricyclic derivatives **13a-f** were obtained in moderate to excellent yields with excellent regioselectivities. Moreover, linear *N*-Boc amides **9a-e** were also amenable to this transformation, and the desired 3,4-dihydro-1,3-oxazin-2-ones **14a-m** were readily achieved in moderate yields with excellent regioselectivities.

Introduction

The discovery of efficient synthetic methodology to access divergent skeletons is one of the most important tasks in the field of organic and pharmaceutical chemistry [1]. Nitrogen-containing heterocyclic small molecules not only widely exist in nature [2], but also serve as important subunits of many clinical drugs discovered in the past decades [3]. As a prime instance (Fig. 1), functionalized 3,4-dihydro-1,3-oxazin-2-one skeletons **1**–**3** are the key framework of several pharmacologically interesting molecules and clinical drugs [4]. Moreover, these skeletons could be easily converted to functionalized 1,3-amino alcohol fragment **4**, an important unit of many natural products and pharmacologically interesting molecules [5]. One example is geissolosimine **5**, which was isolated from the geissospermum vellosii in the Amazonian forest [6], showed antiparasitic activities and it may be a lead molecular structure for possible antimalarial drug development [7]. Another is PD172938 **6** which was identified as a potent dopamine D_4 ligand and indicated possible antipsychotic activity [8]. Due to the important application of 3,4-dihydro-1,3-oxazin-2-one skeleton in natural products and potential clinical drugs, the related intermediates have attracted great interest in the past decades and thereby several synthetic methods have been developed [9]. However, efficient synthetic approach to access functionalized 3,4-dihydro-1,3-oxazin-2-one skeleton is still in great demand [10].

N-Acyliminium ions [11], an important class of organic synthetic intermediates, are widely used in the formation of C—C and C-heteroatom bonds [12] through intermolecular addition [13] and intramolecular cyclization [14] with various nucleophilic reagents. As a prime instance (Fig. 2), *N*-acyliminium ions have been successfully used to synthesize functionalized 1,3-oxazin-2-ones. In 1992, Hiemstra and Speckamp's group reported the synthesis of oxazinone skeleton through intermolecular reactions of *N*-alkoxycarbonyliminium ions with aryl acetylenes (Fig. 2, a) [4c]. Maruoka's group achieved various 1,3-oxazin-2-ones through the $BF_3{\cdot}OEt_2$-mediated cyclization process of Boc-protected aminals with different alkynes (Fig. 2, b) [4d]. Very recently, Wei and Si successfully converted *N*-acyliminium ions to pyrido or pyrrolo[1,2-c][1,3]oxazin-1-ones[9a-c] and 3,4-dihydro-1,3-oxazin-2-ones (Fig. 2, c) [10b]. To our best knowledge, all these important transformations to 1,3-oxazin-2-ones require the use of isolated precursors *N, O*- acetals or *N*-Boc aminals, which are relatively unstable in most cases. Consequently, the application of these methods is limited in the preparation of functionalized 3,4-dihydro-1,3-oxazin-2-one skeleton[4d-e]. Ynamides, a class of popular nucleophiles, have been widely used in organic synthesis [15]. On the basis of our con-

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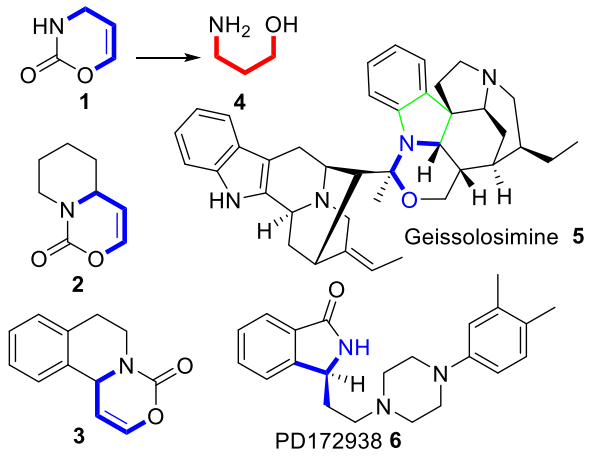


Fig. 1. Natural products containing [1,2-c][1,3]oxazine skeleton.

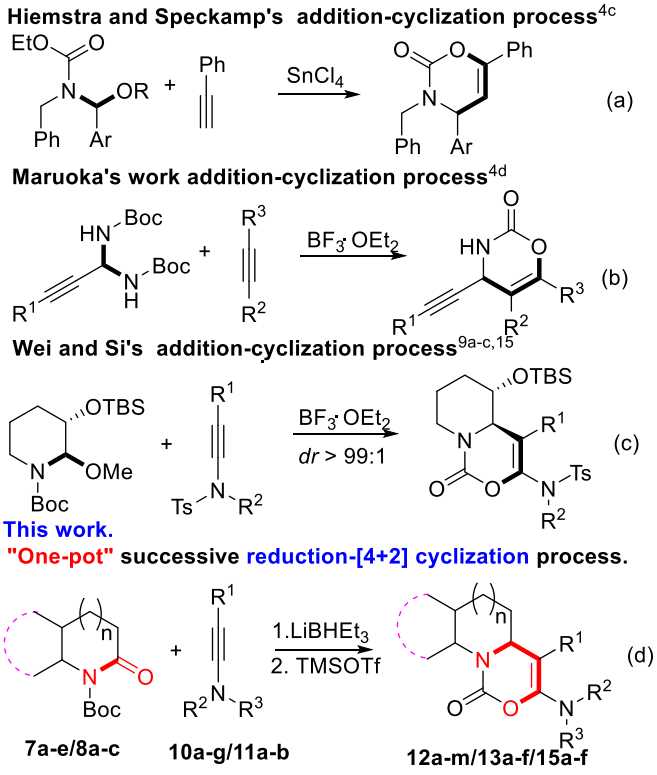


Fig. 2. The reactions of *N*-acyliminium ions.

tinuous efforts in exploring novel heterocyclic compounds as potential innovative drugs [16], we envisioned that functionalized 1,3-oxazin-2-one skeletons **1**, **2** and **3** could be conveniently prepared in a one-pot fashion from *N*-Boc protected lactams. Herein we present our one-pot approach to access 1,3-oxazin-2-ones through TMSOTf-mediated successive reduction-[4 + 2] cyclization process (Fig. 2, d).

Our investigation started with the reaction of imide **7a** with ynamide **10a**. The general one-pot transformation was conducted as followed: After the imide **7a** reacted with lithium triethylborohydride [17] (1.1 equiv.) at −78 °C for 0.5 h, the resulting mixture, without quenching, was treated with the ynamide **10a** and Lewis acid in turn. A variety of Lewis acids were screened for this process. Initially, AgOTf and $AgNTf_2$ both afforded a lot of by-products, and trace amount of desired product which was hardly isolated by silica gel column (Table 1, entries 1 and 2). When $Cu(OTf)_2$ was used, the desired product **12a** was obtained in 26% yield[15e] (Table 1 entry 3). Other triflates, such as $Sc(OTf)_3$, $Zn(OTf)_2$, $Yb(OTf)_3$, $In(OTf)_3$ and $Ce(OTf)_3$, could also afford the desired product **12a** with 36%-51% yields (Table 1, entries 4–8) because of incomplete reaction. Similarly, strong Lewis acid $TiCl_4$ could only afford trace amount of desired product **12a** (Table 1 entry 9). Delightfully, BF_3-OEt_2 (1.0 equiv.) and TMSOTf (1.0 equiv.) could lead to the desired **12a** in moderate yields (Table 1, entries 10–11). When the TMSOTf increased to 2 equiv., the desired product **12a** was achieved in 80% yield (Table 1, entry 12). Further warming the reaction mixture to room temperature could not improve the yield of **12a** (Table 1, entry 13). In addition, other solvents like toluene, THF and Hexanes were also screened, and none of them led to the better yields (Table 1, entries 14–16). Especially in Hexanes, the reaction became complex with great amount of side reaction.

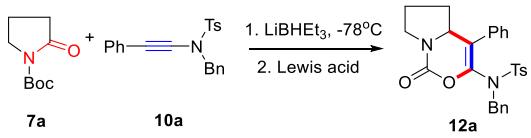
With the above optimized reaction conditions in hand, different *N*-Boc lactams **7a-e** and ynamides **10a-f** were examined for this one-pot transformation, and the results were summarized in Scheme 1. *N*-Boc γ-lactam **7a** could react with different ynamides **10a-e**, affording the desired **12a-e** in moderate yields (68%-80%). *N*-Boc δ-lactam **7b** also worked well, albeit leading to the desired products **12f** and **12g** in slightly lower yields (56% and 61%, respectively). Notably, the reactions of *N*-Boc ε-lactam **7c** with *N*-(Ts) alkyl or Bn ynamides could afford the desired products **12h**, **12i** and **12j** in excellent yields (70%–87%). Importantly, **7c** could react with oxazolidone-substituted ynamide **10f** to afford **12k** in 45% yield. Substituted *N*-Boc γ-lactams were also investigated. The reactions of 2-OTBS substituted **7d** with ynamides **10a-b** could afford the desired **12l** and **12m** with excellent diastereoselectivities (*dr* > 99:1) and in moderate yields [9a]. It was worth mentioning that *N*-Boc β-lactam **7f** could not work for this transformation, probably due to the small ring tension effect during the reduction-[4 + 2] cycloaddition process.

Next, the benzoimides **8a-c** were examined under optimal reaction conditions, and the results are shown in Scheme 2. *tert*-Butyl 2-oxo-3,4-dihydroquinoline-1(2*H*)-carboxylate **8a** (n = 1) could react with ynamides **10a-d** to give the desired product **13a-d** in moderate yields (43%-70%), whereas the reaction of *tert*-butyl 2-oxoindoline-1-carboxylate **8b** (n = 0) and ynamide **10a** could not lead to any desired product. Notably, *tert*-butyl 2-oxo-2,3,4,5-tetrahydrobenzo[*b*]azepine-1-carboxylate **8c** (n = 2) could react with ynamides **10a** and **10b** to afford the desired **13e** and **13f** in 54% and 67% yields respectively. The structures of **13a-f** were unambiguously confirmed by the X-ray crystallographic analysis of compound **13a** (see Supporting Information).

Then we turned our attention to investigate the application of linear imides for this one-pot transformation, and the results are summarized in Scheme 3. The imide substrate methylamine (*tert*-butyl acetyl(methyl)carbamate) **9a** worked well with ynamides **10a-c, 10e, 10g** under the optimized reaction conditions, giving the desired 3,4-dihydro-1,3-oxazin-2-ones skeleton **14a-e** in moderate yields (57%–80%). Similar results were obtained for the imide substrates **9b** and **9c**, and the desired products **14f-m** were afforded in moderate yields (57%-87%). Unfortunately, the imide substrates **9d** and **9e** with phenyl group for either R^1 or R^2 led to no desired products.

Finally, two chiral ynamides **11a** and **11b** were selected to investigate the potential stereochemical control of this one-pot transformation in Scheme 4. The achiral oxazolidone-substituted ynamide **10f** could react with linear imide **9a** to give the desired **15a** in 44% yield. When chiral oxazolidone-substituted ynamide **11a** (R^4 = Bn, R^5 = H) was used, the desired **15b** was produced in 59% yield and with moderate diastereoselectivity (*dr* = 78:22). Similar diastereoselectivity (*dr* = 75:25) was observed for the desired product **15c** when another chiral oxazolidone-substituted ynamide

Table 1
Optimization of reaction conditions.

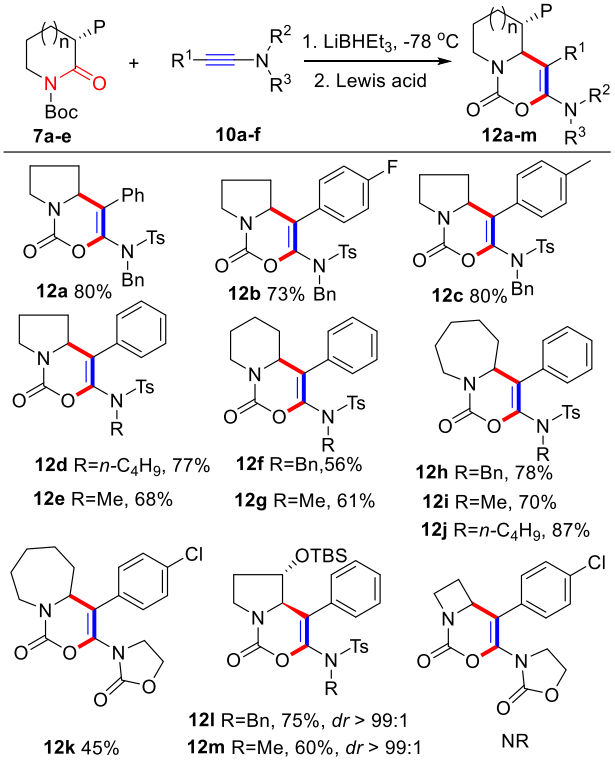


Entry[a]	Lewis acid (equiv.)	T(°C)	Solvent	Yield(%)[b]
1	AgOTf (0.2)	rt ~ 45	DCM	trace
2	$AgNTf_2$ (0.2)	rt ~ 45	DCM	trace
3	$Cu(OTf)_2$ (0.2)	−78 ~ rt	DCM	26
4	$Sc(OTf)_3$ (0.2)	−78 ~ rt	DCM	36
5	$Zn(OTf)_2$ (0.2)	−78 ~ rt	DCM	51
6	$Yb(OTf)_3$ (0.2)	−78 ~ 45	DCM	45
7	$In(OTf)_3$ (0.2)	rt ~ 45	DCM	38
8	$Ce(OTf)_3$ (0.2)	rt ~ 45	DCM	46
9	$TiCl_4$ (1.0)	−78 ~ -45	DCM	trace
10	BF_3OEt_2 (1.0)	−78 ~ -45	DCM	53
11	TMSOTf (1.0)	−78 ~ -45	DCM	58
12	**TMSOTf (2.0)**	**−78 ~ -45**	**DCM**	**80**
13	TMSOTf (2.0)	−78 ~ rt	DCM	78
14	TMSOTf (2.0)	−78 ~ -45	Toluene	31
15	TMSOTf (2.0)	−78 ~ -45	THF	24
16	TMSOTf (2.0)	−78 ~ -45	Hexane	complex

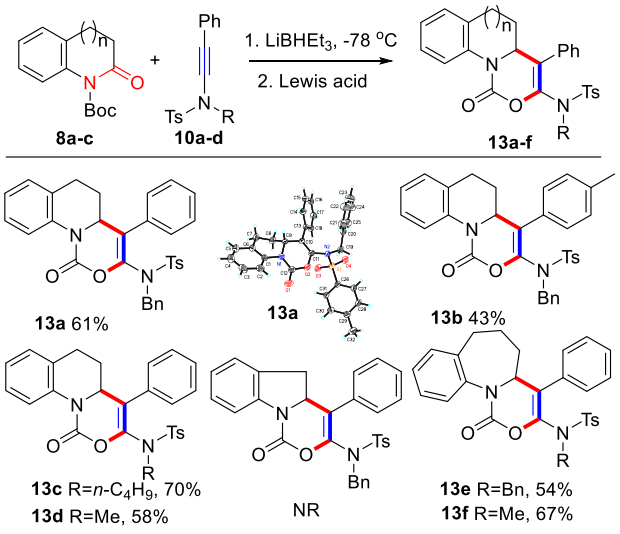
[a] **7a** (0.5 mmol) reacted with $LiBHEt_3$ (0.55 mmol) in dry DCM (2 mL) under N_2 atmosphere at −78 °C for 0.5 h, then the mixture was treated with the **10a** (0.6 mmol) and Lewis acid.
[b] Isolated yield.

11b (R^4 = Me, R^5 = Ph) was employed. The cyclic imides **7a** and **7c** were subsequently examined for the purpose of futher improving the stereoselectivity. Unfortunately, such efforts turned out to be fruitless, and the desired products **15d-f** were obtained in moderate diastereoselectivities. The stereochemistry of the products **15a-f** was unambiguously confirmed by X-ray crystallographic analysis of compound **2*S*-15d** (see Supporting Information).

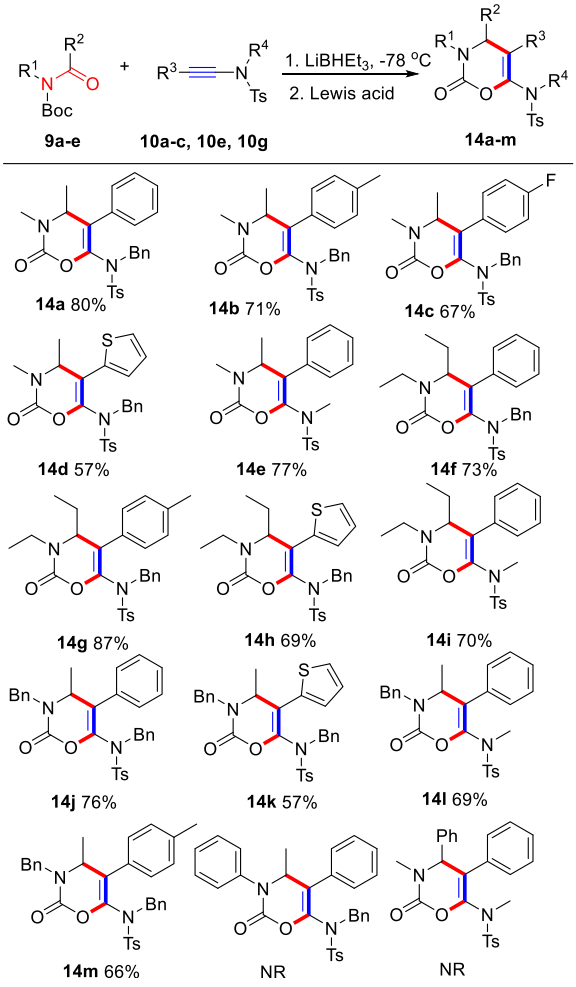
The proposed mechanism of the TMSOTf-mediated [4 + 2] process was illustrated in Fig. 3. Firstly, TMSOTf activated the reduction product **7a'-e'/8a'-c'** to form an iminonium **int-1** [18]. Then, the [4 + 2] cyclization with the triple bond in ynamides **10a-f/11a-b** would take place regioselectively with **int-1** to give a six-membered intermediate **int-2** via the transition state **TS-1**. Finally, the **int-2** was subjected to the cleavage of the *tert*-butyl group to afford the desired product **12a-m/13a-f/15a-f**, together with simultaneous generation of 2-methylprop-1-ene and triflic acid [10a].



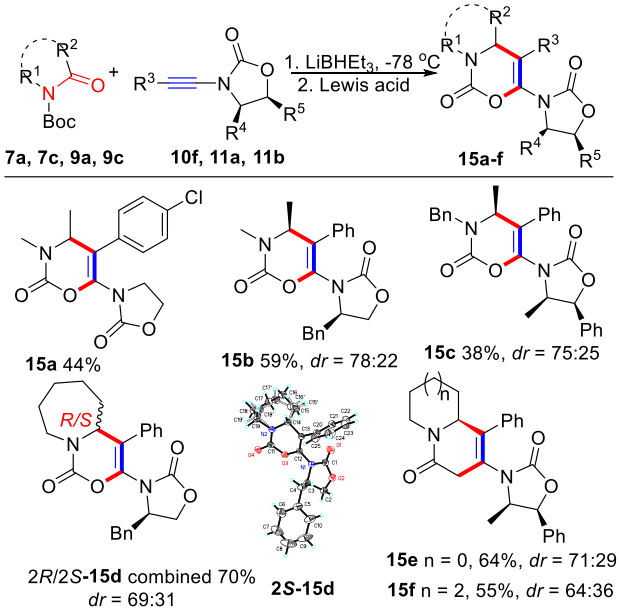
Scheme 1. [a]**7a-e** (0.5 mmol) reacted with $LiBHEt_3$ (0.55 mmol) in dry DCM (2 mL) under N_2 atmosphere at −78 °C for 0.5 h, then the mixture was treated with the **10a-f** (0.6 mmol) and TMSOTf (1.0 mmol); [b]Isolated yield. [c]*dr* values were determined by ^1H NMR of crude products.



Scheme 2. [a]**8a-c** (0.5 mmol) reacted with $LiBHEt_3$ (0.55 mmol) in dry DCM (2 mL) under N_2 atmosphere at −78 °C for 0.5 h, then the mixture was treated with the **10a-d** (0.6 mmol) and TMSOTf (1.0 mmol); [b]Isolated yield.



Scheme 3. [a]**9a-e** (0.5 mmol) reacted with $LiBHEt_3$ (0.55 mmol) in dry DCM (2 mL) under N_2 atmosphere at −78 °C for 0.5 h, then the mixture was treated with the **10a-c, 10e, 10g** (0.6 mmol) and TMSOTf (1.0 mmol); [b]Isolated yield.



Scheme 4. [a]**7a, 7c, 9a, 9c** (0.5 mmol) reacted with $LiBHEt_3$ (0.55 mmol) in dry DCM (2 mL) under N_2 atmosphere at −78 °C for 0.5 h, then the mixture was treated with the **10f, 11a, 11b** (0.6 mmol) and TMSOTf (1.0 mmol); [b]Isolated yield. [c]*dr* values were determined by ^1H NMR of crude products.

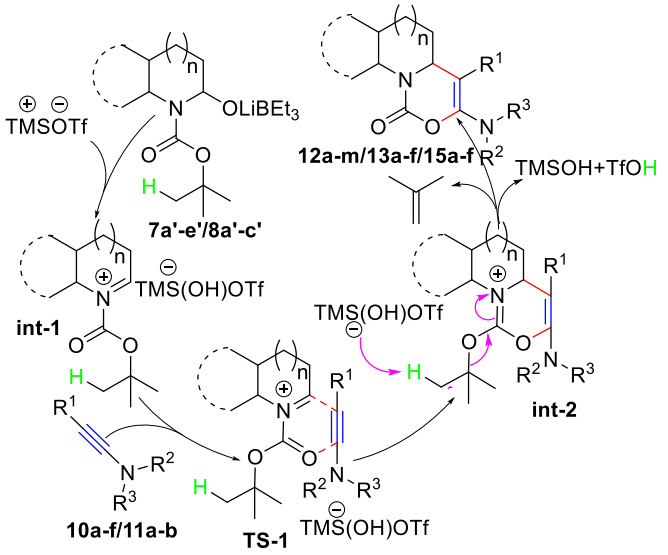


Fig. 3. The proposed mechanism of the TMSOTf-mediated [4 + 2] process.

In summary, we established a one-pot procedure from imides and ynamides to generate a variety of cyclic structures containing [1,3]oxazin-1-one unit. The reactions proceeded through successive reduction and subsequent [4 + 2] cyclization process, and the desired products **12a-m** and **13a-f** were obtained in moderate yields and with excellent regioselectivities. In addition, linear imides **9a-e** were also suitable for this transformation, leading to the desired 3,4-dihydro-1,3-oxazin-2-ones **14a-m** in moderate yields. Chiral oxazolidone-substituted ynamides were also examined, and the desired products **15b-f** were generated with moderate diastereoselectivities.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.152946.

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