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Communication

Intermolecular [4 + 2] process of *N*-acyliminium ions with simple olefins for construction of functional substituted-1,3-oxazinan-2-ones

Xiaoli Han[a], Xiaodi Nie[a], Yiman Feng[a], Bangguo Wei[a,*], Changmei Si[a,*], Guoqiang Lin[b]

[a] Institutes of Biomedical Sciences and School of Pharmacy, Fudan University, Shanghai 200433, China
[b] Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

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ABSTRACT

An efficient approach to functionalized 4,6-disubstituted-and 4,6,6-trisubstituted-1,3-oxazinan-2-ones skeleton has been developed through the reaction of semicyclic *N,O*-acetals **4a** and **4b** with 1,1-disubstituted ethylenes **5** or **8**. As a result of such a [4 + 2] cycloaddition process, 4,6,6-trisubstituted-1,3-oxazinan-2-ones **6aa, 6af-6au, 7ba, 7bf-7bw** and 6,6-spiro containing 1,3-oxazinan-2-ones **9ad, 9ae, 10ba-10bg** were obtained in 36%-96% yields and with moderate to excellent diastereoselectivities. In addition, the synthesis of (±)-norallosedamine **12** could be conveniently achieved from the cycloadduct **7bf**.

Nitrogen-containing heterocycles are abundant structural motifs in a large number of alkaloids [1], drug molecules [2] and biologically active substances [3]. Although significant accomplishments have been achieved towards the synthesis of various nitrogen heterocyclic compounds in the past few decades [4], modern organic synthesis still demands more efficient and divergent methodologies to access privileged motifs of biological active compounds [5]. As a prime instance, 1,3-oxazinan-2-ones are not only a core scaffold within natural products [6] and pharmacologically interesting molecules [7], but also widely utilized as key intermediates [8] in the synthesis of drugs [8c,8f] and bioactive natural products [7b,8g]. Numerous synthetic approaches were reported to access various substituted 1,3-oxazinan-2-ones, including halonium-mediated [6c,9] or metal-catalyzed cyclization [10], intramolecular Michael addition of functionalized homoallylamines/homoallylic alcohols [11], allylic C-H amination [12], and tethered aminohydroxylation of olefins [13]. However, the effective methods to access 6,6-disubstituted-1,3-oxazinan-2-one **1**, exemplified with the core structural unit of biologically active compounds such as anti-HIV Efavirenz **2** (Merck) [7e] and 11-β-HSD-1 inhibitor **3** [7c], are rare [14] (Fig. 1).

N-Acyliminium ions, acting as important organic synthetic intermediates, are widely used in the formation of C-C and C-heteroatom bonds [15], mostly through intermolecular addition [16] and intramolecular cyclization [17] with various nucleophilic reagents. For examples, the reactions of *N*-acyliminium ions with olefins could undergo Lewis acid-catalyzed intramolecular addition-cyclization to construct a series of heterocyclic skeletons (Fig. 2, Eq. 1) [18]. Intermolecular reactions of *N*-acyliminium ions with olefins were also reported [19]. Kobayashi achieved the ring-opening allylation of semicyclic *N,O*-acetals with allylic silanes (Fig. 2, Eq. 2a) [19a]. Later, Zhang developed the intermolecular coupling reaction of *N*-acyliminium ions with styrene (Fig. 2, Eq. 2b) [19b]. Notably, *N*-acyliminium ions could serve as part of electron-deficient dienes, undergoing [4 + 2] cycloaddition with various dienophiles (alkenes or alkynes) [20,21]. For example, Yoshida established a cycloaddition process of *N*-acyliminium ions connecting with an alkoxycarbonyl group with alkenes to afford substituted 1,3-oxazinan-2-one framework, but the formation of corresponding *N*-acyliminium dienes required anodic oxidation of α-silyl carbamate substrates (Fig. 2, Eq. 3) [20d]. On the basis of our continuous efforts in exploring chemical transformations of semicyclic *N,O*-acetals [22], we envisioned that such [4 + 2] cycloaddition could lead to various important units. Herein we present an efficient synthetic approach to 4,6,6-trisubstituted-1,3-oxazinan-2-ones **6/7/9/10** through TMSOTf-mediated [4 + 2] cycloaddition of semicyclic *N,O*-acetals **4** with 1,1-disubstituted ethylenes **5/8** (Fig. 2, Eq. 4).

Our investigation started with the reaction of semicyclic *N,O*-acetal **4b** with 1,1-diphenylethene **5a**. The reaction could not take place in the absence of Lewis acid (Table 1, entry 1). Several types of iron Lewis acids could lead to only faint products (Table 1, entries 2-6). When $Ni(OTf)_2$, $Cu(OTf)_2$ and $Sc(OTf)_3$ were examined,

* Corresponding authors.
E-mail addresses: bgwei1974@fudan.edu.cn (B. Wei), sicm@fudan.edu.cn (C. Si).

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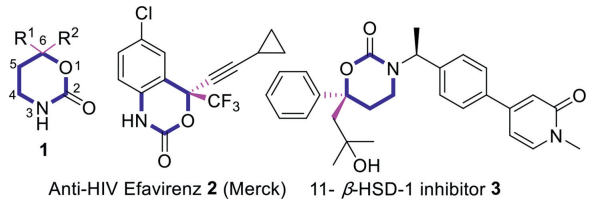


Fig. 1. 6,6-Disubstituted-1,3-oxazinan-2-one scaffold and active compounds.

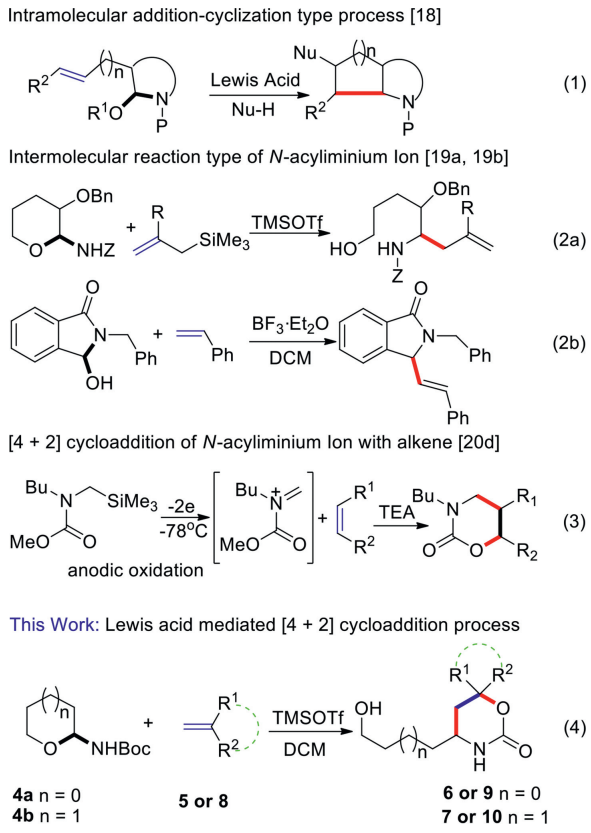


Fig. 2. The intra- or intermolecular reactions of *N*-acyliminium ions with olefins.

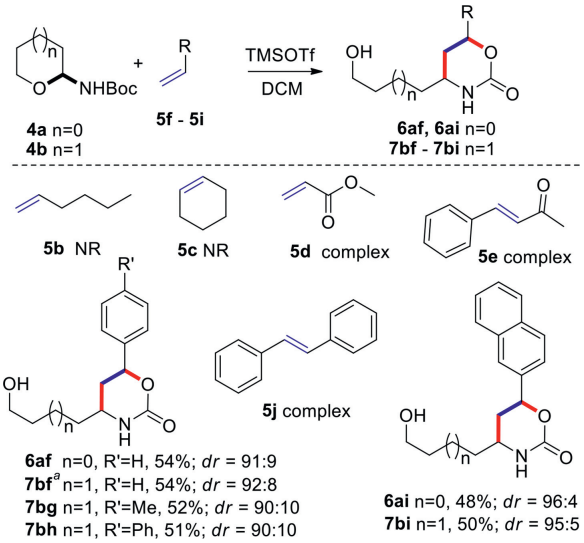
no product was observed (Table 1, entries 7-9). $SnCl_4$ could afford the desired product **7ba** in 34% yield (Table 1, entry 10). Slight improvements in yields were achieved when $TiCl_4$ and $BF_3{\cdot}Et_2O$ were applied, and the desired product **7ba** could be obtained in moderate yield (48% and 66% respectively, Table 1, entries 11 and 12). Delightfully, TMSOTf could significantly increase the yield of **7ba** to 81% (Table 1, entry 13). It was worth noting that the reaction was conducted at -78 °C. Either increasing or decreasing the loading of TMSOTf resulted in slight drop of the reaction yield (Table 1, entries 14 and 15). The reaction could also afford the desired product using THF and PhMe as solvents, but the corresponding yields were lower compared with that in dichloromethane (Table 1, entries 16 and 17).

With the above identified optimized reaction conditions, the olefin substrates **5b**-**5j** with different electronic properties were examined and the results were summarized in Scheme 1. First, neither hex-1-ene **5b** nor cyclohexene **5c** could afford any desired product in the presence of TMSOTf. The dienophiles with electron-withdrawing groups, methyl acrylate **5d** and (*E*)-4-phenylbut-3-en-2-one **5e**, led to complicated reaction mixtures. Delightfully, styrene **5f** and its derivatives (**5g** and **5h**) could generate the desired products **6af, 7bf-7bh** in moderate yields and diastereoselectivities. 2-Vinylnaphthalene could also react with **4a** and **4b** to

Table 1
Optimization of reaction conditions.[a]

Entry	Catalyst (equiv.)	T (°C)	Solvents	Yield (%)[b]
1	–	r.t.	DCM	NR
2	$FeCl_3$ (0.2)	0∼r.t.	DCM	trace
3	$FeCl_2$ (0.2)	0∼r.t.	DCM	trace
4	$Fe(OTf)_3$ (0.2)	0∼r.t.	DCM	trace
5	$Fe(OTf)_2$ (0.2)	0∼r.t.	DCM	trace
6	$Fe(BF_4)_3$ (0.2)	0∼r.t.	DCM	trace
7	$Ni(OTf)_2$ (0.2)	r.t.	DCM	NR
8	$Cu(OTf)_2$ (0.2)	r.t.	DCM	NR
9	$Sc(OTf)_3$ (0.2)	r.t.	DCM	NR
10	$SnCl_4$ (2.0)	-78∼r.t.	DCM	34
11	$TiCl_4$ (2.0)	-78∼r.t.	DCM	48
12	$BF_3{\cdot}Et_2O$ (2.0)	-78∼r.t.	DCM	66
13	TMSOTf (2.0)	-78	DCM	81
14	TMSOTf (1.5)	-78	DCM	74
15	TMSOTf (3.0)	-78	DCM	78
16	TMSOTf (2.0)	-78	PhMe	71
17	TMSOTf (2.0)	-78	THF	51

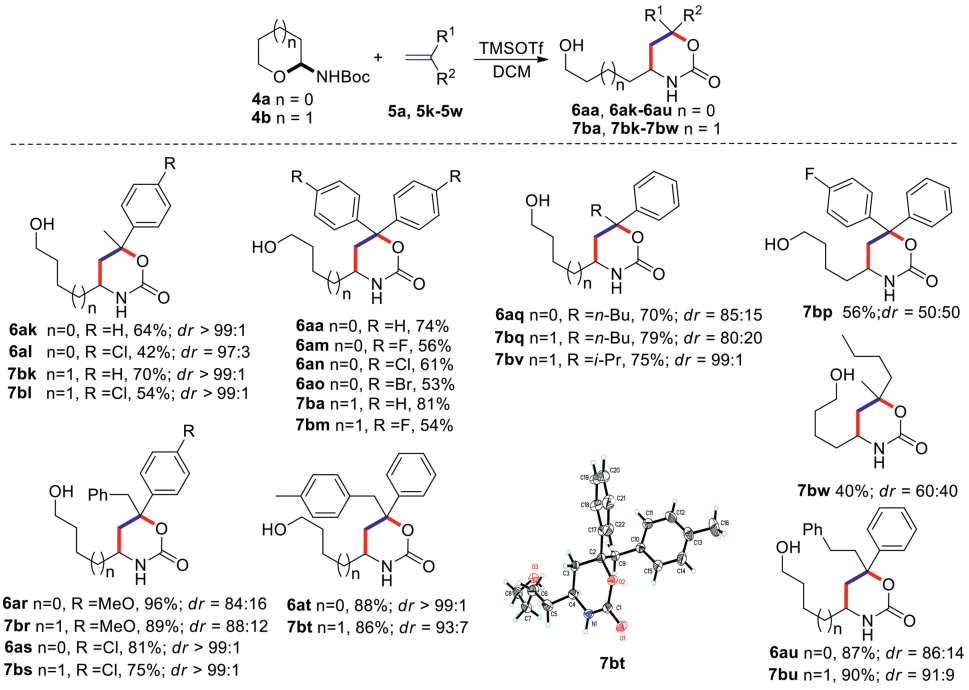
[a] The reaction was performed with **4b** (0.5 mmol), **5a** (1.0 mmol) in solvents (2.0 mL).
[b] Isolated yield.



Scheme 1. The reactions of semicyclic *N,O*-acetals **4a**/**4b** with olefins **5b-5j**. The reactions were performed with *N,O*-acetals **4** (0.5 mmol), olefins **5b-5j** (1.0 mmol), and TMSOTf (1.0 mmol) in dry DCM (2 mL) at -78 °C for 5-10 h, isolated yield. *dr* was determined by HPLC or NMR of crude products. [a] **7bf** (2.80 g, 51% yield) was obtained with **4b** (22.0 mmol), **5f** (44.0 mmol), and TMSOTf (44.0 mmol) in dry DCM (50 mL) at −78 °C for 8 h.

give the corresponding products **6ai** and **7bi** in moderate yields and diastereoselectivities. It was worth mentioning that a complex result was obtained when (*E*)-1, 2-diphenylethene **5j** was investigated, probably due to the steric effect during the intermolecular [4+2] cycloaddition process.

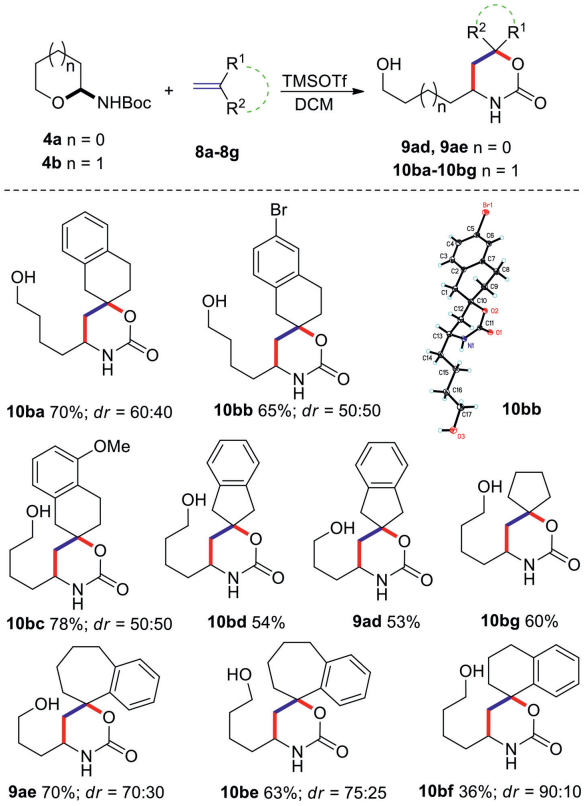
Next, we turned to investigate the scope and limitation of such addition-cyclization of semicyclic *N,O*-acetal (**4a** or **4b**) with 1,1-disubstituted ethylenes **5a, 5k-5v** (Scheme 2). When prop-1-en-2-ylbenzene **5k** was explored, desired products **6ak, 7bk** were obtained in moderate yields and excellent diastereoselectivities. 4-Chloro substitution at phenyl ring (**5l**) led to slight decrease in yields of **6al, 7bl**, but with excellent diastereoselectivities (*dr* up to 99:1). Replacement of the methyl group of **5k** with other alkyl



Scheme 2. The reactions of semicyclic *N,O*-acetals **4a/4b** with substituted olefins **5a, 5k-5w**. The reactions were performed with *N,O*-acetal **4** (0.5 mmol), olefins (0.75 mmol), and TMSOTf (1.0 mmol) in dry DCM (2 mL) at -78 °C for 5-10 h. Isolated yield. *dr* was determined by HPLC or NMR of crude products.

substitutions (**5q**: *n*-butyl and **5v**: isopropyl) was tolerated, and the desired products **6aq, 7bq, 7bv** were afforded in moderate yields under the optimized conditions. Although the *n*-butyl substituted products **6aq, 7bq** showed moderate diastereoselectivities, the isopropyl substituted product **7bv** was obtained with excellent diastereoselectivities. In addition, a series of diaryl substituted alkenes were surveyed under the optimized conditions. In general, all these substituted alkenes (**5a, 5l-5o**) could react with semicyclic *N,O*-acetals **4a** and **4b**, affording the desired products **6aa, 6am-6ao, 7ba, 7bm, 7bp** in moderate yields. Several benzyl and phenyl olefins **5r-5t** were also screened, most of them could give the desired products **6ar-6at, 7br-7bt** in excellent yields and diastereoselectivities, except for the *p*-methoxyphenyl substituted olefin **5r**. Substituted olefin **5u** containing phenyl and phenethyl could also afford the desired products **6au** and **7bu** in excellent yields with moderate diastereoselectivities. The methyl and butyl substituted ethylene **5w** could also react with *N,O*-acetal **4b** to afford the desired product **7bw** in 40% yield, but the diastereoselectivities was lower than those of aryl olefins. The chemical structures of **6aa, 6ak-6au, 7ba, 7bk-7bw** were unambiguously confirmed based on the X-ray crystallographic analysis of compound **7bt** (see Supporting information for detail).

Next, we turned our attention to investigate the reaction of semicyclic *N,O*-acetal **4a** or **4b** with exocyclic olefins **8a-8g**, aiming for the formation of 1,3-oxazinan-2-ones containing a spiro quaternary carbon (Scheme 3). The reaction of 2-methylene-1,2,3,4-tetrahydronaphthalene **8a** with semicyclic *N,O*-acetal **4b** afforded the desired product **10ba** in 70% yield. The 6-bromo substituted olefin **8b** led to **10bb** in slightly lower yield of 65%, while the 5-methoxy substituted olefin **8c** could generate **10bc** in slightly higher yield of 78%. However, the diastereoselectivities of **10ba-10bc** were low. The symmetric olefin, 2-methylene-2,3-dihydro-1*H*-indene **8d**, also worked well with semicyclic *N,O*-acetals **4a** and **4b**, affording the desired products **9ad** and **10bd** in moderate yields. Notably, a simple exocyclic olefin methylenecyclopentane **8g** also worked well, and the corresponding product **10bg** was obtained in 60% yield. Regarding olefin substrates with



Scheme 3. The reactions of semicyclic *N,O*-acetals **4a/4b** with substituted olefins **8a-8g**. The reactions were performed with *N,O*-acetal **4** (0.5 mmol), olefins (0.75 mmol), and TMSOTf (1.0 mmol) in dry DCM (2 mL) at -78 °C for 5-10 h. Isolated yield. *dr* was determined by HPLC or NMR of crude products.

the *exo*-double bond adjacent to the phenyl ring, 5-methylene-6,7,8,9-tetrahydro-5*H*-benzo[7]annulene **8e** bearing a fused seven-membered ring could lead to the corresponding products in higher

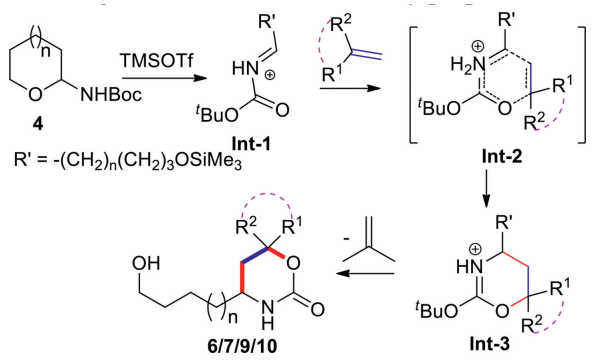
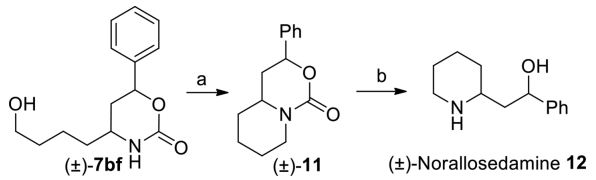


Fig. 3. Possible mechanism for the TMSOTf-mediated [4 + 2] cycloaddition process.



Scheme 4. Synthesis of (±)-Norallosedamine **12**. Reagents and conditions: (a) i, DMP, DCM, r.t., 3 h; ii, Et_3SiH/TMSOTf, MeCN, 0 °C to r.t., 40 min, 75% (2 steps); (b) *t*-BuOH/toluene (v/v = 1/1), KOH, 85 °C, 30 min, 86%.

yields than that of 1-methylene-1,2,3,4-tetrahydronaphthalene **8f** bearing a fused six-membered ring. In detail, the desired products **9ae** and **10be** were obtained in 70% and 63% yields, while the yield of **10bf** was only 36%. The diastereoselectivities of **9ae, 10be** and **10bf** were increased slightly, maybe due to the steric hindrance. The structures of **9ad, 9ae, 10ba-10bg** were unambiguously confirmed based on the X-ray crystallographic analysis of compound **10bb** (see Supporting information for detail).

A possible mechanism for this TMSOTf-mediated [4 + 2] cycloaddition process is presented in Fig. 3 [19a,20d,20f]. When semicyclic *N,O*-acetals **4** reacted with alkenes **5**, diene type of *N*-acyliminium ions **Int-1** was first generated under Lewis acid conditions. The subsequent reaction with alkenes **5** gave a six-membered intermediate **Int-2**, which would define the stereochemical outcome and give **Int-3**. Upon the cleavage of *t*-butyl group, the corresponding cycloadducts **6/7/9/10** were produced, along with the release of 2-methylprop-1-ene.

Finally, we focused on the utility of this intermolecular [4 + 2] process of *N*-acyliminium ions with alkenes in the synthesis of biologically active molecules. Scheme 4 showed a facile synthesis of norallosedamine **12**. As a natural product, norallosedamine **12** was isolated from both the *Sedum* and *Lobelia inflata* plant family, and have attracted great interest in synthetic chemistry [23]. Starting from the cycloadduct **7bf**, Dess-Martin oxidation (DMP) and subsequent reductive amination (Et_3SiH/TMSOTf) could produce bicyclic pyrido [1,2-*c*][1,3]oxazin-1-one **11** in 75% overall yield. Then the ring opening (KOH) of **11** resulted in (±) norallosedamine **12** in 86% yield (*dr* = 94:6). The spectroscopic and physical data of the synthetic (±)-norallosedamine **12** were identical to the reported data [23a]. Norallosedamine **12** could be potentially converted to other alkaloids of its family by known process [23b,23d].

In summary, we established a novel and efficient approach for the synthesis of 4,6-disubstituted- and 4,6,6-trisubstituted-1,3-oxazinan-2-ones **6aa, 6af-6au, 7ba, 7bf-7bw** and 6,6-spiro containing 1,3-oxazinan-2-ones **9ad, 9ae, 10ba-10bg**. The Lewis acid TMSOTf could activate semicyclic *N,O*-acetals (**4a** and **4b**), and the resulting *N*-alkoxycarbonyliminium ions readily underwent a [4 + 2] cycloaddition process with 1,1-disubstituted ethylenes **5a, 5k-5w** and **8a-8g**. The corresponding products were obtained in moderate to excellent yields and diastereoselectivities. In addition, the utility of this methodology was demonstrated by the facile synthesis of natural product (±)-norallosedamine **12** from the cycloadduct **7bf**.

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.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2021.05.003.

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