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# $Sc(OTf)_3$-catalyzed intramolecular diastereoselective cyclization from *tert*-butoxycarbonyl to acyliminium ion

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ABSTRACT

An effective approach to access dyadic 1,3-oxazinan-2-ones **8a**-**8c** and 4,4a,5,6-tetrahydro-[1,3]oxazino [3,4-a]quinolin-1(3H)-ones **8d-8h** was developed through $Sc(OTf)_3$-catalyzed intramolecular cyclization from *tert*-butoxycarbonyl to acyliminium ion **7a-7j**. A variety of substituted *N,O*-acetals, with different ring size, proved to be suitable substrates for this transformation, and a series of (4a*S*,6*S*,7*R*)-6-OTBS-7-substituted-hexahydropyrrolo[1,2-c][1,3]oxazin-1-ones **11a-11j** and other chiral dyadic 1,3-oxazinan-2-ones **8i**, **8j**, **11k** were synthesized in moderate yields with excellent diastereoselectivities (*dr* > 99:1). Moreover, 2,5-*trans*-products **11a-11j** were obtained through this interesting Lewis acid-catalyzed intramolecular cyclization process.



## 1. Introduction

Substituted 2,5-*cis* or 2,5-*trans* 3-hydroxyl-pyrrolidin-3-ol skeleton **1** (Fig. 1) is a valuable structural unit existing in a variety of natural products [1] and numerous biologically active pharmaceuticals [2]. Particularly, these heterocyclic natural products featuring a 2,5-*cis* or 2,5-*trans* substituted chiral pyrrolidine [3] usually exhibit potent biological and medicinal activities. For example, (+)-preussin [4] (**2**), a pyrrolidinol alkaloid containing 2,5-*cis* chiral pyrrolidine unit, can induce apoptosis in human tumor cells. A considerable number of natural products contain 2,5-*trans* pyrrolidine unit. Australine [5] (**3**) and casuarine [6] (**4**) are not only effective inhibitors of glucosidase I, but also display a variety of biological activities including antiviral and anti-HIV. Another example is epohelmin A [7] (**5**), which demonstrated inhibition of recombinant lanosterol synthase ($IC_{50}$ = 10 μM). In the past decades, tremendous efforts have been devoted to the development of stereospecific approach to 2,5- disubstituted pyrrolidine skeleton. Some important approaches include the nucleophilic substitution of *N,O*-acetals with organoboron [8], organosilicone [9] and organometallic reagents [10], as well as the addition-cyclization process [11], mainly forming 2,5-*cis* disubstituted pyrrolidines. The examples to generate 2,5-*trans* products are relatively uncommon. Huang's group developed a $SmI_2$-mediated radical cross-coupling reaction of azahemiacetals with ethyl acrylate [12], and our group recently achieved gold-catalyzed intermolecular addition cyclization of *N,O*-acetals with ynamides [13]. Therefore, a direct process to 2,5-*trans* pyrrolidine unit is still in demand.

*N*-Acyliminium ions (NAIs) [14], a type of extremely important reactive intermediates, are widely used in chemical transformations in past decades [15]. From the reaction point of view, NAIs can act as highly electron-deficient carbocations to react with weak nucleophiles [16], forming carbon-carbon (C—C) and carbon-heteroatom bond through both intermolecular and intramolecular reactions [17]. Therefore, NAIs chemistry is highly recognized in the divergent synthesis and synthetic applications [18]. The reaction of NAIs with $sp^3$ hybridized carbon is very rare [19]. For examples, Sugihara's group [20] observed such a by-product while studying Lewis acid-catalyzed allylation of *N,N*-acetal with allyl trimethylsilane, with isolated yields of 16—38% (Fig. 2a). Mancheño also observed a similar cyclization by-product during the oxidative

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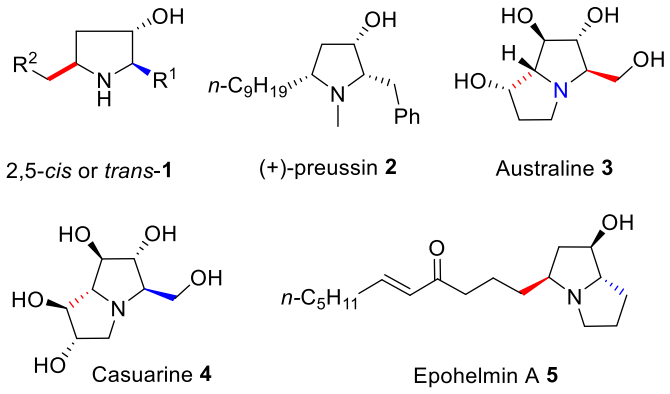



**Fig. 1.** Some 2,5- disubstituted pyrrolidines.

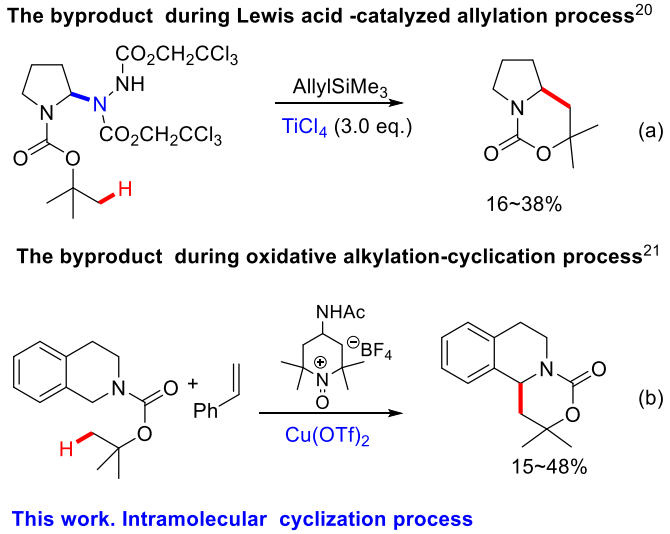



**Fig. 2.** The intramolecular cyclization of NAIs.

alkylation-cyclization process [21] (Fig. 2b). On the basis of our continuous efforts in exploring chemical transformations of NAIs [11e,13,22a,22b], we envisioned that such intramolecular cyclization from methyl of *tert*-butoxycarbonyl group [23] could be achieved. Herein we present this interesting Lewis acid-catalyzed intramolecular cyclization from *tert*- butoxycarbonyl group to NAIs (Fig. 2c).

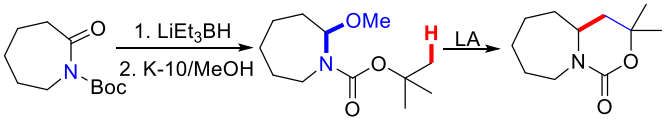
## 2. Results and discussion

Our investigation started with the seven-membered cyclic imide **6a**, which underwent the reduction with lithium triethylborohydride ($LiEt_3BH$) and subsequent treatment with montmorillonite K-10 in methanol to give the cyclization precursor, *N,O*-acetal **7a**. To avoid potential elimination, the newly prepared crude **7a** was directly employed in next step without further purification. When $BF_3Et_2O$ was used for this transformation, the desired cyclization product **8a** was obtained in 21% yield (Table 1, entry 1). A variety of metallic Lewis acids were examined. $Ni(OTf)_2$, $In(OTf)_3$ and $Er(OTf)_3$ led to no desired product (Table 1, entries 2–4). A small amount of **8a** was obtained when $AgSbF_6$ was used (10%, Table 1, entry 5). Fortunately, $Zn(OTf)_2$, $Cu(OTf)_2$ and $Y(OTf)_3$ could significantly increase the yield of **8a** (up to 78%, Table 1, entries 6–8). It was worth noting that $Sc(OTf)_3$ could lead to the desired **8a** in 90% yield (Table 1, entry 9). In this case, when the reaction was conducted at room temperature or with lower loading of $Sc(OTf)_3$, the desired product **8a** could still be produced, albeit in moderate yields (51% and 77%, Table 1, entries 10–11). Different solvents were also screened for this transformation. The reaction could also afford the desired product in DCM, but the corresponding yield of **8a** was only 30% (Table 1, entry 12). Other solvents, such as THF, PhMe and MeCN, resulted in complex reaction mixtures (Table 1, entries 13–15), and no desired **8a** was isolated.

Next, we turned to investigate the scope and limitation of such intramolecular addition cyclization from *tert*-butoxycarbonyl group to acyliminium ion (Scheme 1). A variety of *N*-Boc lactams **6** were prepared according to typical procedure of reduction and subsequent etherification, and the resulting cyclization precursors **7** were used in the following step without further purification. When the crude pyrrolidine substrate **7b** was subjected to the optimized $Sc(OTf)_3$ conditions, the yield of **8b** was much lower compared with **8a**. However, 4,4-disubstituted pyrrolidine substrate **7c** could afford the desired cyclization product **8c** in 87% yield. Several tetrahydroquinoline substrates were also surveyed under the optimized conditions. As a result, the desired cyclization products **8d-8h** were obtained in moderate to excellent yields. Notably, chiral substrates **7i** and **7j** led to the desired products **8i** and **8j** with excellent stereoselectivities and yields. The chemical structures and relative stereochemistry of **8i** and **8j** were unambiguously confirmed based on the X-ray crystallographic analysis of compound **8j** (see the Supporting Information).

Next, we extend this method to synthesize 2,5-disubstituted pyrrolidines and piperidines (Scheme 2). Due to the potential

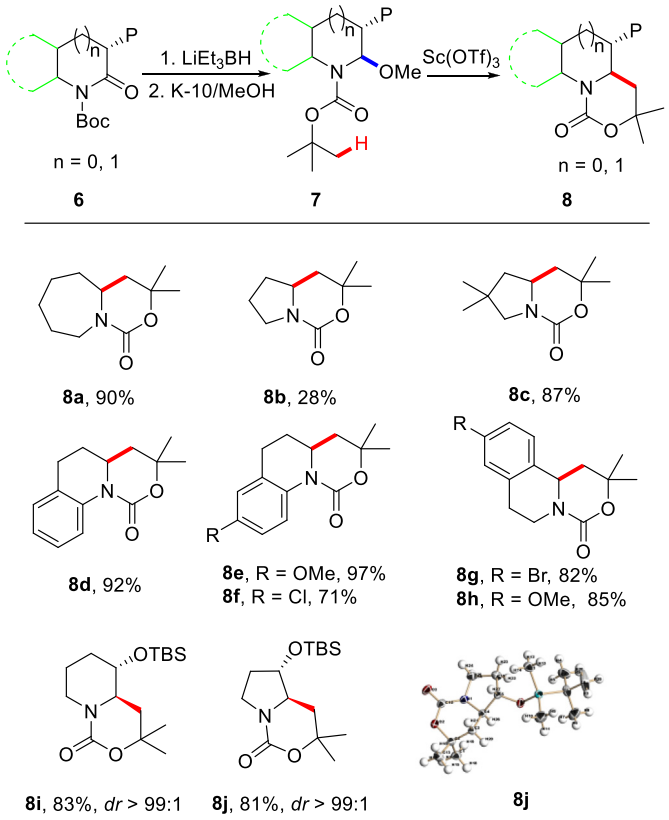
**Table 1**
Optimization of the reaction conditions.



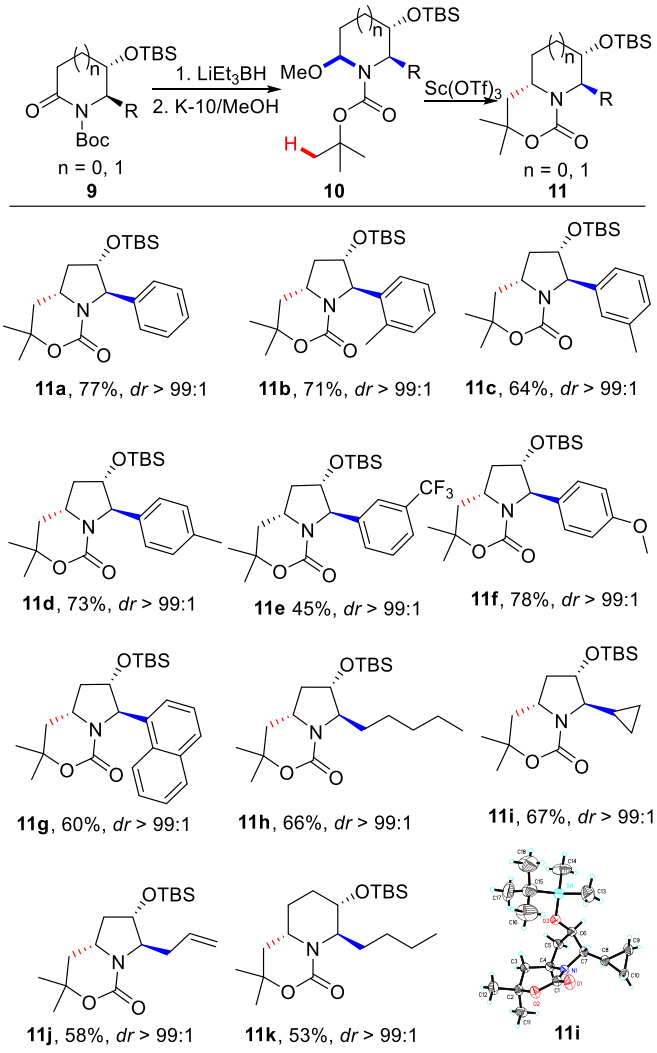

**6a** **7a** (crude) **8a**

| Entry[a] | Lewis Acid | Solvent | T (°C) | Yield %[b] |
|---|---|---|---|---|
| 1 | $BF_3.Et_2O$ (2.0 eq.) | DCE | r.t.-60 | 21 |
| 2 | $Ni(OTf)_2$ (0.5 eq.) | DCE | 60 | – |
| 3 | $In(OTf)_3$ (0.5 eq.) | DCE | 60 | – |
| 4 | $Er(OTf)_3$ (0.5 eq.) | DCE | 60 | – |
| 5 | $AgSbF_6$ (0.5 eq.) | DCE | 60 | 10 |
| 6 | $Zn(OTf)_2$ (0.5 eq.) | DCE | 60 | 50 |
| 7 | $Cu(OTf)_2$ (0.5 eq.) | DCE | 60 | 67 |
| 8 | $Y(OTf)_3$ (0.5 eq.) | DCE | 60 | 78 |
| 9 | $Sc(OTf)_3$ (0.5 eq.) | DCE | 60 | 90 |
| 10 | $Sc(OTf)_3$ (0.5 eq.) | DCE | r.t. | 51 |
| 11 | $Sc(OTf)_3$ (0.2 eq.) | DCE | 60 | 77 |
| 12 | $Sc(OTf)_3$ (0.5 eq.) | DCM | 40 | 30 |
| 13 | $Sc(OTf)_3$ (0.5 eq.) | THF | 60 | Complex |
| 14 | $Sc(OTf)_3$ (0.5 eq.) | PhMe | 60 | Complex |
| 15 | $Sc(OTf)_3$ (0.5 eq.) | MeCN | 60 | Complex |

[a] The reactions were performed with **6a** (2.0 mmol), $LiEt_3BH$ (2.1 mmol) in THF (8 mL) at −78 °C for 30 min, the crude product was stirred in MeOH (8 mL) with K-10 (200 mg) at rt overnight. Then crude **7a** (1.0 mmol) was treated with Lewis acid in solvent (4 mL)
[b] Isolated yield.




**Scheme 1.** [a] The reactions were performed with **6** (2.0 mmol), $LiEt_3BH$ (2.1 mmol) in THF (8 mL) at −78 °C for 30 min, crude product was stirred in MeOH (8 mL) with K-10 (200 mg) at rt overnight. Then, the crude **7** (1.0 mmol) was treated with $Sc(OTf)_3$ in DCE (4 mL) at 60 °C for 1–2 h; [b] Isolated yield for 3 steps, [c] *dr* were determined by $^1H$ NMR of crude products.




**Scheme 2.** [a] The reactions were performed with **9** (2.0 mmol), $LiEt_3BH$ (2.1 mmol) in THF (8 mL) at −78 °C for 30 min, crude product was stirred in MeOH (8 mL) with K-10 (200 mg) at rt overnight. Then, the crude **10** (1.0 mmol) was treated with $Sc(OTf)_3$ in DCE (4 mL) at 60 °C for 1–2 h; [b] Isolated yield for 3 steps; [c] *dr* were determined by $^1H$ NMR of crude products.

elimination issue during silica gel column chromatography, the newly prepared *N*,*O*-acetals **10a-10k** were used immediately in next step without further purification. All of them afforded the desired cyclization products with excellent diastereoselectivities (*dr* > 99:1). Substrates with different substituted phenyl groups led to 2,5-*trans*-disubstituted pyrrolidines **11a-11f** in 45–78% yields. When α-naphthyl substituted *N*,*O*-acetal **10g** was investigated, the desired 2,5-*trans*-**11g** was obtained in 60% yield with excellent diastereoselectivities (*dr* > 99:1). Aliphatic substituted substrates also worked well under the optimal conditions. *n*-Pentyl, cyclopropyl and allyl *N*,*O*-acetals **10h-10j** could afford 2,5-*trans*-**11h-11j** in moderate yields, along with outstanding diastereoselectivities (*dr* > 99:1). When *n*-butyl substituted piperidine *N*,*O*-acetal **10k** was examined, the yield of 2,6-*trans* -**11k** slightly reduced, but the diastereoselectivity was still excellent (*dr* > 99:1). The stereochemistry of the products **11a-11k** were unambiguously confirmed based on X-ray crystallographic analysis of compound **11i** (see Supporting Information).

On the basis of literature reports and our experimental results, a mechanism for this reaction is tentatively proposed (Fig. 3). First, $Sc(OTf)_3$ led to partial *N*-Boc deprotection of *N*,*O*-acetal to release isobutene. Upon the formation of acyliminium ion **A** through the elimination of MeOH assisted by $Sc(OTf)_3$, isobutene could add to it to give a stable tertiary carbon cation **B**. Further intramolecular attack by carbonyl group could give the product **8** or **11**, and regenerate isobutene. In the half-chair conformation of acyliminium ion **A**, both OTBS and R groups is positioned in the pseudo equatorial orientation. Presumably due to the steric effect of axial hydrogen at 3-position, the nucleophilic isobutene would attack the iminium **A** from the bottom to form a *trans*-product.

## 3. Conclusions

In summary, we established a novel and interesting approach for the synthesis of dyadic 1,3-oxazinan-2-ones **8a-8c**, 4,4a,5,6-tetrahydro-[1,3]oxazino[3,4-a]quinolin-1(3H)-ones **8d–8h**, chiral piperidine, chiral dyadic 1,3-oxazinan-2-ones **8i**, **8j**, **11k** and (4*aS*,6*S*,7*R*)-6-OTBS-7-substituted-hexahydropyrrolo[1,2-c][1,3] oxazin-1-ones **11a-11j**. The Lewis acid $Sc(OTf)_3$ could catalyze the intramolecular cyclization from *tert*-butoxycarbonyl to acyliminium ion **7** and **10**, and the corresponding products **8** and **11** were obtained in moderate to excellent yields with excellent diastereoselectivities (*dr* > 99:1). In addition, 2,5-*trans*-products **11a-11j**

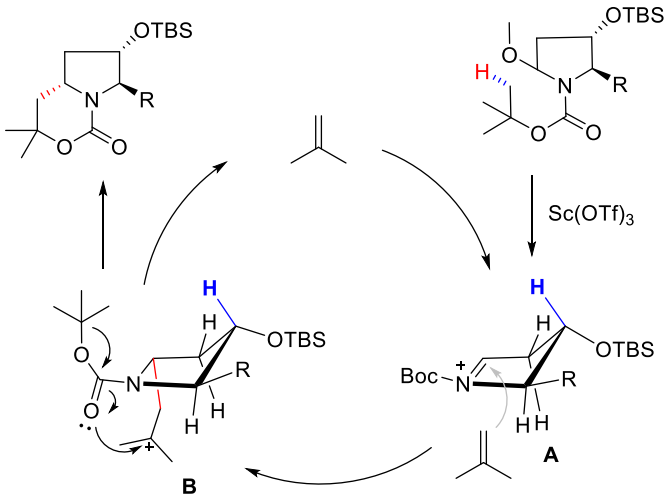



**Fig. 3.** Proposed mechanism.

were obtained using this interesting Lewis acid-catalyzed intramolecular cyclization process.

## 4. Experimental section

### 4.1. General methods

THF was distilled from sodium/benzophenone. Reactions were monitored by thin layer chromatography (TLC) on glass plates coated with silica gel with fluorescent indicator. Flash chromatography was performed on silica gel (300–400) with PE/EA as eluent. Optical rotations were measured on a polarimeter with a sodium lamp. HRMS were measured on LTQ-Orbitrap. IR spectra were recorded using film on a Fourier Transform Infrared Spectrometer. NMR spectra were recorded at 400 or 600 MHz, and chemical shifts are reported in (ppm) referenced to an internal TMS standard for $^{1}$H NMR and $CDCl_3$ (77.16 ppm) for $^{13}$C NMR.

General procedure for synthesis of **8** and **11**. To a solution of **6** or **9** (2.0 mmol) in dry THF (8 mL) at −78 °C was added $LiEt_3BH$ (2.1 mL, 2.1 mmol, 1M in THF). After being stirred at −78 °C for 0.5 h, the reaction was quenched with saturated sodium bicarbonate aqueous solution then the mixture was extracted with EtOAc (30 mL*3). The combined organic layers were washed with brine. Dried, filtrated and concentrated to give the crude product without further purification. The residue was dissolved in MeOH (8 mL) and montmorillonite K-10 (200 mg) was added, after being stirred at rt for overnight, the mixture was filtrated through a short silica gel and concentrated to give the *N,O*-acetal **7** or **10**.

To a solution of **7** or **10** (1.0 mmol) in DCE (4 mL) was added $Sc(OTf)_3$ (0.5 mmol), then the mixture was heated at 60 °C for 1–2 h. After being cooled to rt, the reaction was quenched with saturated sodium bicarbonate aqueous solution then the mixture was extracted with EtOAc (30 mL*3). The combined organic layers were washed with brine. Dried, filtrated and concentrated. The residue was purified by flash chromatography on silica gel to give **8** or **11**.

### 4.2. 3,3-Dimethyloctahydro-1H-[1,3]oxazino[3,4-a]azepin-1-one (8a)

White solid (177 mg, 90%), m.p. 93–95 °C, IR (film): $\nu_{max}$ 2919, 1684, 1504, 1431, 1370, 1288 $cm^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 4.01-3.93 (m, 1H), 3.80-3.72 (m, 1H), 2.98-2.89 (m, 1H), 1.92-1.80 (m, 2H), 1.80-1.75 (m, 2H), 1.75-1.62 (m, 4H), 1.50-1.41 (m, 1H), 1.38 (s, 3H), 1.37 (s, 3H), 1.35-1.25 (m, 1H) ppm; $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$) $\delta$ 153.9, 76.3, 51.7, 46.4, 40.1, 34.4, 29.8, 29.7, 28.2, 25.2, 22.4 ppm. HRMS (ESI) *m/z* calcd for $C_{11}H_{20}NO_2^+$ $(M + H)^+$ 198.1489, found 198.1489.

### 4.3. 3,3-Dimethylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one (8b)

White solid (48 mg, 28%), m.p. 73–75 °C, IR (film): $\nu_{max}$ 2971, 1647, 1430, 1369, 1624 $cm^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 3.63-3.52 (m, 2H), 3.51-3.44 (m, 1H), 2.19-2.11 (m, 1H), 2.06 (dd, $J$ = 13.6, 4.4 Hz, 1H), 2.03-1.97 (m, 1H), 1.89-1.76 (m, 1H), 1.54-1.43 (m, 2H), 1.43 (s, 3H), 1.38 (s, 3H) ppm; $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$) $\delta$ 153.0, 78.6, 53.7, 46.4, 39.2, 33.4, 29.8, 25.8, 22.9 ppm. HRMS (ESI) *m/z* calcd for $C_9H_{16}NO_2^+$ $(M + H)^+$ 170.1176, found 170.1176.

### 4.4. 3,3,6,6-Tetramethylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one (8c)

White solid (172 mg, 87%), m.p. 99–101 °C, IR (film): $\nu_{max}$ 2969, 2081, 1637, 1453, 1369, 1646 $cm^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 3.87-3.77 (m, 1H), 3.37 (d, $J$ = 11.2 Hz, 1H), 3.22 (d, $J$ = 11.2 Hz, 1H), 1.52-1.45 (m, 1H), 1.43 (s, 3H), 1.39 (s, 3H), 1.16 (s, 3H), 1.15 (s, 3H) ppm; $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$) $\delta$ 153.0, 78.9, 59.9, 52.7, 47.5, 39.7, 36.5, 30.0, 27.7, 27.6, 26.1 ppm. HRMS (ESI) *m/z* calcd for $C_{11}H_{20}NO_2^+$ $(M + H)^+$ 198.1489, found 198.1489.

### 4.5. 3,3-Dimethyl-4,4a,5,6-tetrahydro-1H,3H-[1,3]oxazino[3,4-a]quinolin-1-one (8d)

White solid (213 mg, 92%), m.p. 113–115 °C, IR (film): $\nu_{max}$ 2955, 2932, 1692, 1418, 1329, 1126, 872, 838 $cm^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.52-7.48 (m, 2H), 7.47-7.40 (m, 2H), 4.82 (d, $J$ = 6.4 Hz, 1H), 4.16-4.10 (m, 1H), 4.05-3.96 (m, 1H), 2.36-2.28 (m, 1H), 2.09 (dd, $J$ = 13.6, 4.8 Hz, 1H), 1.78-1.70 (m, 2H), 1.49 (s, 3H), 1.46 (s, 3H), 0.86 (s, 9H), −0.07 (s, 3H), −0.14 (s, 3H) ppm; $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$) $\delta$ 152.7, 142.2, 129.2, 128.9, 124.3, 124.3, 122.6, 122.6, 79.0, 78.7, 69.3, 53.1, 41.6, 39.5, 29.9, 25.7, 25.5, 17.9, −4.7, −5.1 ppm. HRMS (ESI) *m/z* calcd for $C_{14}H_{18}NO_2^+$ $(M + H)^+$ 232.1332, found 232.1334.

### 4.6. 8-Methoxy-3,3-dimethyl-4,4a,5,6-tetrahydro-1H,3H-[1,3]oxazino[3,4-a]quinolin-1-one (8e)

White solid (253 mg, 97%), m.p. 110–112 °C, IR (film): $\nu_{max}$ 3473, 2920, 1690, 1500, 1404, 1311, 1250 $cm^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.80-7.76 (m, 1H), 6.78-6.73 (m, 1H), 6.63-6.60 (m, 1H), 3.77 (s, 3H), 3.72-3.66 (m, 1H), 2.89-2.84 (m, 2H), 2.15-2.08 (m, 2H), 1.89-1.78 (m, 2H), 1.47 (s, 3H), 1.46 (s, 3H) ppm; $^{13}$C{$^{1}$H} NMR (125 MHz, $CDCl_3$) $\delta$ 155.8, 148.7, 131.0, 129.4, 125.0, 113.5, 112.4, 55.6, 51.3, 46.9, 41.1, 30.7, 29.7, 26.6, 24.6 ppm. HRMS (ESI) *m/z* calcd for $C_{15}H_{20}NO_3^+$ $(M + H)^+$ 262.1438, found 262.1438.

### 4.7. 8-Chloro-3,3-dimethyl-4,4a,5,6-tetrahydro-1H,3H-[1,3]oxazino[3,4-a]quinolin-1-one (8f)

White solid (188 mg, 71%), m.p. 100–102 °C, IR (film): $\nu_{max}$ 3473, 2920, 1690, 1500, 1404, 1311, 1250 $cm^{-1}$; $^{1}$H NMR (600 MHz, $CDCl_3$) $\delta$ 7.83-7.81 (m, 1H), 7.14-7.11 (m, 1H), 7.09-7.07 (m, 1H), 3.71-3.65 (m, 1H), 2.88-2.83 (m, 2H), 2.15-2.09 (m, 2H), 1.89-1.83 (m, 1H), 1.81-1.75 (m, 1H), 1.47 (s, 3H), 1.46 (s, 3H) ppm; $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$) $\delta$ 151.7, 136.3, 129.8, 128.8, 126.5, 125.2, 51.4, 40.9, 30.2, 29.7, 26.3, 24.6 ppm. HRMS (ESI) *m/z* calcd for $C_{14}H_{16}ClNO_2^+$ $(M + H)^+$ 266.0948, found 266.0949.

*4.8. 9-Bromo-2,2-dimethyl-1,6,7,11b-tetrahydro-2H,4H-[1,3] oxazino[4,3-a]isoquinolin-4-one **(8g)***

White solid (253 mg, 82%), m.p. 107–109 °C, IR (film): $\nu_{max}$ 3473, 2920, 1690, 1500, 1404, 1311, 1250 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.80-7.74 (m, 1H), 7.29-7.22 (m, 2H), 3.72-3.64 (m, 1H), 2.90-2.83 (m, 2H), 2.17-2.07 (m, 2H), 1.91-1.83 (m, 1H), 1.82-1.72 (m, 1H), 1.47 (s, 3H), 1.46 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 1151.7, 136.9, 131.8, 130.2, 129.3, 125.5, 116.9, 51.4, 40.9, 30.1, 29.7, 26.2, 24.7 ppm. HRMS (ESI) *m/z* calcd for $C_{14}H_{17}BrNO_2^+$ $(M + H)^+$ 310.0437, found 310.0437.

*4.9. 9-Methoxy-2,2-dimethyl-1,6,7,11b-tetrahydro-2H,4H-[1,3] oxazino[4,3-a]isoquinolin-4-one **(8h)***

White solid (223 mg, 85%), m.p. 110–112 °C, IR (film): $\nu_{max}$ 2919, 1684, 1431, 1288, 1036, 754 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.05-7.00 (m, 1H), 6.81-6.76 (m, 1H), 6.70-6.67 (m, 1H), 4.78-4.72 (m, 1H), 4.59-4.53 (m, 1H), 3.80 (s, 3H), 3.07-2.97 (m, 2H), 2.73-2.67 (m, 1H), 2.40 (dd, $J$ = 13.6, 5.2 Hz, 1H), 1.88-1.80 (m, 1H), 1.50 (s, 3H), 1.43 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 167.9, 158.5, 153.0, 136.3, 125.9, 113.8, 113.2, 55.5, 51.3, 42.1, 41.0, 29.8, 29.2, 25.4 ppm. HRMS (ESI) *m/z* calcd for $C_{15}H_{20}NO_3^+$ $(M + H)^+$ 262.1438, found 262.1438.

*4.10. (4aR,5S)-5-((tert-Butyldimethylsilyl)oxy)-3,3-dimethylhexahydro-1H,3H-pyrido[1,2-c][1,3]oxazin-1-one **(8i)***

White solid (260 mg, 83%), m.p. 78–80 °C, $[\alpha]_D^{21} = -23.3$ (*c* 1.00, $CHCl_3$); IR (film): $\nu_{max}$ 2931, 2856, 1689, 1432, 1132, 871, 832 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 4.25 (d, $J$ = 13.3 Hz, 1H), 3.78-3.73 (m, 1H), 3.48-3.40 (m, 1H), 2.80-2.68 (m, 1H), 2.10-1.87 (m, 2H), 1.85-1.74 (m, 2H), 1.47-1.37 (m, 1H), 1.30 (s, 3H), 1.27 (s, 3H), 0.87 (s, 9H), 0.05 (s, 3H), 0.04 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) $\delta$ 154.0, 75.8, 66.8, 55.4, 44.4, 35.8, 31.9, 29.7, 25.8, 24.9, 18.7, 18.2, −4.2, −5.0 ppm. HRMS (ESI) *m/z* calcd for $C_{16}H_{32}NO_3Si^+$ $(M + H)^+$ 314.2146, found 314.2145.

*4.11. (4aR,5S)-5-((tert-Butyldimethylsilyl)oxy)-3,3-dimethylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one **(8j)***

White solid (242 mg, 81%), m.p. 66–68 °C, $[\alpha]_D^{21} = -23.3$ (*c* 1.00, $CHCl_3$); IR (film): $\nu_{max}$ 2954, 2930, 1703, 1386, 1420, 1131, 862, 838 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 3.82-3.75 (m, 1H), 3.60-3.50 (m, 2H), 3.36-3.29 (m, 1H), 2.21-2.13 (m, 1H), 2.07 (dd, $J$ = 13.2, 4.4 Hz, 1H), 1.84-1.73 (m, 1H), 1.43 (s, 3H), 1.37 (s, 3H), 0.90 (s, 9H), 0.80 (s, 6H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 153.0, 79.0, 58.8, 43.8, 37.5, 32.1, 29.9, 25.8, 25.8, 18.1, −4.4, −4.6 ppm. HRMS (ESI) *m/z* calcd for $C_{15}H_{30}NO_3Si^+$ $(M + H)^+$ 300.1989, found 300.1989.

*4.12. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-3,3-dimethyl-7-phenylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one **(11a)***

White solid (289 mg, 77%); m.p. 89–91 °C, $[\alpha]_D^{21} = +5.9$ (*c* 1.00, $CHCl_3$); IR (film): $\nu_{max}$ 2954, 1692, 1416, 1187, 866, 837 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.34-7.29 (m, 2H), 7.26-7.20 (m, 3H), 4.86 (d, $J$ = 5.2 Hz, 1H), 4.24-4.18 (m, 1H), 4.04-3.94 (m, 1H), 2.34-2.25 (m, 1H), 2.05 (dd, $J$ = 13.6, 4.8 Hz, 1H), 1.78-1.68 (m, 2H), 1.48 (s, 3H), 1.45 (s, 3H), 0.86 (s, 9H), −0.06 (s, 3H), −0.09 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 152.8, 140.9, 128.7, 127.3, 125.7, 78.7, 70.0, 52.7, 41.2, 39.7, 31.5, 30.0, 25.7, 25.7, 17.9, −4.8, −4.9 ppm. HRMS (ESI) *m/z* calcd for $C_{21}H_{34}NO_3Si^+$ $(M + H)^+$ 376.2302, found 376.2302.

*4.13. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-3,3-dimethyl-7-(o-tolyl)hexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one **(11b)***

White solid (276 mg, 71%), m.p. 68–70 °C, $[\alpha]_D^{21} = +30.0$ (*c* 0.10, $CHCl_3$); IR (film): $\nu_{max}$ 2924, 1636, 1417, 1220, 1110, 773 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.15-7.10 (m, 3H), 7.10-7.05 (m, 1H), 5.10 (d, $J$ = 5.2 Hz, 1H), 4.26-4.20 (m, 1H), 4.12-4.04 (m, 1H), 2.36 (s, 3H), 2.35-2.29 (m, 1H), 2.04 (dd, $J$ = 13.6, 4.4 Hz, 1H), 1.77-1.69 (m, 2H), 1.46 (s, 3H), 1.45 (s, 3H), 0.84 (s, 9H), −0.10 (s, 3H), −0.14 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 152.7, 139.7, 136.1, 130.6, 127.2, 126.4, 125.1, 78.9, 78.7, 67.0, 53.4, 41.2, 40.0, 30.1, 29.9, 25.8, 19.9, 18.0, −4.9, −5.0 ppm. HRMS (ESI) *m/z* calcd for $C_{22}H_{36}NO_3Si^+$ $(M + H)^+$ 390.2459, found 390.2460.

*4.14. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-3,3-dimethyl-7-(m-tolyl)hexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one **(11c)***

White solid (249 mg, 64%), m.p. 69–72 °C, $[\alpha]_D^{21} = +9.4$ (*c* 0.50, $CHCl_3$); IR (film): $\nu_{max}$ 2855, 1693, 1415, 1255, 862, 837 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.21-7.18 (m, 1H), 7.06-7.03 (m, 2H), 7.02-6.99 (m, 1H), 4.83 (d, $J$ = 5.2 Hz, 1H), 4.22-4.15 (m, 1H), 4.03-3.94 (m, 1H), 2.33 (s, 3H), 2.05 (dd, $J$ = 13.6, 4.4 Hz, 1H), 1.80-1.74 (m, 1H), 1.73-1.67 (m, 1H), 1.49 (s, 3H), 1.46 (s, 3H), 0.87 (s, 9H), −0.06 (s, 3H), −0.08 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 152.8, 140.8, 138.2, 128.6, 128.1, 126.7, 122.4, 78.8, 78.7, 69.9, 52.9, 43.0, 41.3, 39.8, 30.1, 25.8, 21.7, 18.0, −4.7, −4.9 ppm. HRMS (ESI) *m/z* calcd for $C_{22}H_{36}NO_3Si^+$ $(M + H)^+$ 390.2459, found 390.2458.

*4.15. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-3,3-dimethyl-7-(p-tolyl)hexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one **(11d)***

White solid (284 mg, 73%), m.p. 73–75 °C, $[\alpha]_D^{21} = +41$ (*c* 0.50, $CHCl_3$); IR (film): $\nu_{max}$ 2927, 1613, 1388, 1247, 1109 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.13-7.11 (m, 4H), 4.85 (d, $J$ = 5.2 Hz, 1H), 4.17–4.26 (m, 1H), 4.03-3.95 (m, 1H), 2.33-2.25 (m, 1H), 2.31 (s, 3H), 2.04 (dd, $J$ = 13.2, 4.4 Hz, 1H), 1.79-1.73 (m, 1H), 1.72-1.67 (m, 1H), 1.47 (s, 3H), 1.45 (s, 3H), 0.86 (s, 9H), −0.06 (s, 3H), −0.07 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 152.8, 137.9, 136.9, 129.4, 125.7, 78.7, 78.6, 69.9, 52.9, 41.2, 39.9, 30.1, 25.8, 25.7, 21.2, 18.0, −4.7, −4.8 ppm. HRMS (ESI) *m/z* calcd for $C_{22}H_{36}NO_3Si^+$ $(M + H)^+$ 390.2459, found 390.2459.

*4.16. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-3,3-dimethyl-7-(3-(trifluoromethyl)phenyl)hexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one **(11e)***

White solid (199 mg, 45%), m.p. 80–82 °C, $[\alpha]_D^{21} = +4.8$ (*c* 0.25, $CHCl_3$); IR (film): $\nu_{max}$ 2928, 1690, 1329, 1127, 872, 839 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.53-7.49 (m, 2H), 7.47-7.40 (m, 2H), 4.83 (d, $J$ = 6.0 Hz, 1H), 4.18-4.11 (m, 1H), 4.05-3.97 (m, 1H), 2.37-2.29 (m, 1H), 2.09 (dd, $J$ = 13.2, 4.4 Hz, 1H), 1.78-1.70 (m, 2H), 1.50 (s, 3H), 1.47 (s, 3H), 0.86 (s, 9H), −0.07 (s, 3H), −0.13 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 152.7, 142.3, 129.2, 129.0, 124.4, 122.7, 79.0, 78.7, 69.3, 53.1, 41.6, 39.5, 30.0, 29.9, 25.7, 25.6, 17.9, 0.1, −4.7, −5.1 ppm. HRMS (ESI) *m/z* calcd for $C_{22}H_{33}F_3NO_3Si^+$ $(M + H)^+$ 444.2176, found 444.2178.

*4.17. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-7-(4-methoxyphenyl)-3,3-dimethylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one **(11f)***

White solid (316 mg, 78%), m.p. 80–82 °C, $[\alpha]_D^{21} = +8.2$ (*c* 0.50, $CHCl_3$); IR (film): $\nu_{max}$ 2855, 1692, 1513, 1415, 867, 836 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.19-7.13 (m, 2H), 0.88-0.84 (m, 2H), 4.83-4.79 (d, $J$ = 5.2 Hz, 1H), 4.23-4.17 (m, 1H), 4.02-3.93 (m, 1H), 3.79 (s,

3H), 2.33-2.25 (m, 1H), 2.07-2.00 (dd, $J = 13.2, 4.4$ Hz, 1H), 1.78-1.66 (m, 2H), 1.46 (s, 3H), 1.45 (s, 3H), 0.86 (s, 9H), −0.06 (s, 3H), −0.08 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 156.9, 152.8, 133.1, 127.0, 114.2, 78.7, 78.6, 69.5, 55.4, 52.9, 41.2, 39.8, 30.1, 25.8, 25.7, 18.0, −4.7, −4.8 ppm. HRMS (ESI) $m/z$ calcd for $C_{22}H_{36}NO_4Si^+$ $(M + H)^+$ 406.2408, found 406.2410.

### 4.18. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-3,3-dimethyl-7-(naphthalen-1-yl)hexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one (11g)

White solid (255 mg, 60%), m.p. 100−102 °C, $[\alpha]_D^{21} = +4.4$ ($c$ 1.00, $CHCl_3$); IR (film): $\nu_{max}$ 2929, 2855, 1690, 1416, 1257, 895, 869 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 8.20-8.16 (m, 1H), 7.86-7.82 (m, 1H), 7.76-7.72 (m, 1H), 7.51-7.46 (m,2H), 7.44-7.39 (m, 1H), 7.32-7.27 (m, 1H), 4.35-4.31 (m, 1H), 4.23-4.15 (m, 1H), 2.38-2.31 (m, 1H), 2.10 (dd, $J = 13.6, 4.4$ Hz, 1H), 1.94-1.86 (m, 1H), 1.80-1.74 (m, 1H), 1.57 (s, 3H), 1.50 (s, 3H), 0.89 (s, 9H), −0.10 (s, 3H), −0.16 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 152.9, 136.7, 134.1, 131.0, 128.7, 128.0, 125.9, 125.8, 125.5, 124.5, 121.8, 79.0, 78.3, 68.4, 41.2, 40.4, 30.3, 26.2, 25.8, 17.9, −4.6, −4.9 ppm. HRMS (ESI) $m/z$ calcd for $C_{23}H_{37}NO_3SiH^+$ $(M + H)^+$ 426.2435, found 426.2435.

### 4.19. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-3,3-dimethyl-7-pentylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one (11h)

White solid (244 mg, 66%), m.p. 91−93 °C, $[\alpha]_D^{21} = +15.1$ ($c$ 1.00, $CHCl_3$); IR (film): $\nu_{max}$ 2955, 2929, 1673, 1419, 1113, 882, 838 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 4.40-4.31 (m, 1H), 3.87-3.79 (m,1H), 3.62-3.48 (m, 1H), 2.12-2.02 (m, 1H), 1.98-1.90 (m, 1H), 1.89-1.76 (m, 1H), 1.69-1.62 (m, 1H), 1.61-1.51 (m, 2H), 1.42 (s, 3H), 1.35 (s, 3H), 1.32-1.18 (m, 6H), 0.89 (s, 9H), 0.86-0.80 (m, 3H), 0.07 (s, 6H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 152.2, 79.4, 71.1, 59.8, 49.8, 40.6, 39.1, 32.4, 30.6, 28.8, 27.9, 26.5, 25.9, 22.8, 18.2, 14.2, −4.7, −4.9 ppm. HRMS (ESI) $m/z$ calcd for $C_{20}H_{40}NO_3Si^+$ $(M + H)^+$ 370.2772, found 370.2772.

### 4.20. (4aS,6S,7R)-6-((tert-Butyldimethylsilyl)oxy)-7-cyclopropyl-3,3-dimethylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one (11i)

White solid (227 mg, 67%), m.p. 78−80 °C, $[\alpha]_D^{21} = +0.6$ ($c$ 0.50, $CHCl_3$); IR (film): $\nu_{max}$ 2928, 1691, 1413, 1259, 890, 837 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 4.22-4.18 (m, 1H), 3.78-3.70 (m, 1H), 3.62 (dd, $J = 7.6, 2.8$ Hz, 1H), 2.42-2.34 (m, 1H), 1.93 (dd, $J = 13.2, 4.4$ Hz, 1H), 1.72-1.65 (m, 1H), 1.40 (s, 3H), 1.38 (s, 3H), 0.87 (s, 9H), 0.83-0.77 (m, 1H), 0.60-0.54 (m, 1H), 0.52-0.44 (m, 2H), 0.41-0.35 (m, 1H), 0.08 (s, 3H), 0.06 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 153.4, 78.4, 75.9, 70.5, 51.9, 41.2, 40.1, 30.2, 25.8, 25.6, 18.0, 13.7, 3.2, 2.4, −4.5, −4.8 ppm. HRMS (ESI) $m/z$ calcd for $C_{18}H_{34}NO_3Si^+$ $(M + H)^+$ 340.2303, found 340.2301.

### 4.21. (4aS,6S,7R)-7-Allyl-6-((tert-butyldimethylsilyl)oxy)-3,3-dimethylhexahydro-1H-pyrrolo[1,2-c][1,3]oxazin-1-one (11j)

White solid (197 mg, 58%), m.p. 80−82 °C, $[\alpha]_D^{21} = -24.0$ ($c$ 0.10, $CHCl_3$); IR (film): $\nu_{max}$ 2927, 2855, 1694, 1414, 867, 837 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 5.92-5.80 (m, 1H), 5.13-4.99 (m, 2H), 4.46-4.36 (m, 1H), 4.05-3.96 (m, 1H), 3.63-3.53 (m, 1H), 2.89-2.76 (m, 1H), 2.39-2.29 (m, 1H), 2.12-2.03 (m, 1H), 1.91 (dd, $J = 12.8, 2.8$ Hz, 1H), 1.73-1.60 (m, 2H), 1.41 (s, 3H), 1.35 (s, 3H), 0.92 (s, 9H), 0.09 (s, 3H), 0.01 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 136.4, 117.9, 79.6, 71.0, 60.0, 49.8, 39.9, 39.3, 31.7, 30.6, 29.9, 27.9, 25.9, 18.2, −4.7, −4.9 ppm. HRMS (ESI) $m/z$ calcd for $C_{18}H_{34}NO_3Si^+$ $(M + H)^+$ 340.2303, found 340.2303.

### 4.22. (4aS,7S,8R)-8-Butyl-7-((tert-butyldimethylsilyl)oxy)-3,3-dimethylhexahydro-1H,3H-pyrido[1,2-c][1,3]oxazin-1-one (11k)

White solid (197 mg, 53%), m.p. 84−86 °C, $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 4.16-4.10 (m, 1H), 4.04-3.93 (m, 1H), 3.85-3.76 (m, 1H), 2.01-1.90 (m, 2H), 1.80-1.72 (m, 2H), 1.70-1.54 (m, 3H), 1.38 (s, 3H), 1.36 (s, 3H), 0.87 (s, 9H), 0.07 (s, 3H), 0.06 (s, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) $\delta$ 153.2, 67.8, 59.6, 46.5, 41.2, 33.0, 30.0, 29.8, 29.5, 27.4, 26.3, 25.9, 25.7, 23.1, 17.9, 14.2, −4.6, −4.8 ppm. HRMS (ESI) $m/z$ calcd for $C_{20}H_{40}NO_3Si^+$ $(M + H)^+$ 370.2772, found 370.2772.

## 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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