HETEROCYCLES, Vol. 62, 2004, pp. 839 - 846
Received, 16th September, 2003, Accepted, 6th November, 2003, Published online, 14th November, 2003

NEW ACCESS TO CHIRAL CYCLIC ω-OXYGENATED β-ENAMINO ESTERS BY INTRAMOLECULAR AMINOCYCLISATION REACTIONS‡

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Abstract – The preparation of chiral 4-methoxycarbonylmethylidene-oxazolidines and oxazolidinone from (*S*)-1-phenylethylamine and hydroxy alkynoates is described. The extension of this strategy to 6- and 7-membered heterocyclic homologues is also reported.

INTRODUCTION

Optically active 1,2-amino alcohols are important building blocks in asymmetric synthesis.[1] They are usually used as chiral synthons or as precursors of chiral auxiliaries.[2] We have recently reported the preparation of chiral methoxycarbonylmethylidenemorpholinone **(1)** (Figure 1) as a precursor of functionalized 1,2-amino alcohols.[3] However, this synthon presented the disadvantages to be prone to racemisation during purification and to possess two ester functions of similar reactivity. In order to overcome these limitations and to develop a new, general access to chiral functionalized 1,ω-amino alcohols, we envisioned that chiral heterocyclic β-enamino esters **(2)** and **(3)** would be attractive precursors. These heterocycles bear an exocyclic, stereochemically stable *N*-substituent and display a joint protection of the amine and alcohol functions as an hemiaminal or as a carbamate respectively (Figure 1). The choice of the hemiaminal and the carbamate protecting groups was dictated by their

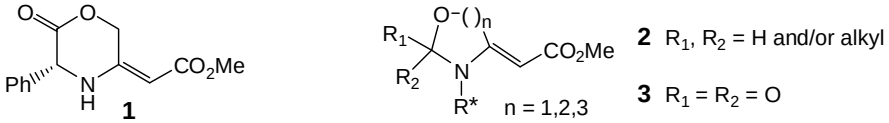


Figure 1

‡ Dedicated to Professor Leo A. Paquette on the occasion of his 70th birthday

orthogonality compared to the ester function and by their different pattern of reactivity-stability. In the projected strategy, the key step to access to the targeted 1,ω-amino alcohols from **2** or **3** was designed as a reduction of the double bond to generate a new chiral center α to the nitrogen atom. This reduction was expected to be highly stereoselective owing to the close vicinity of the chiral substituent attached to the nitrogen atom.[4]

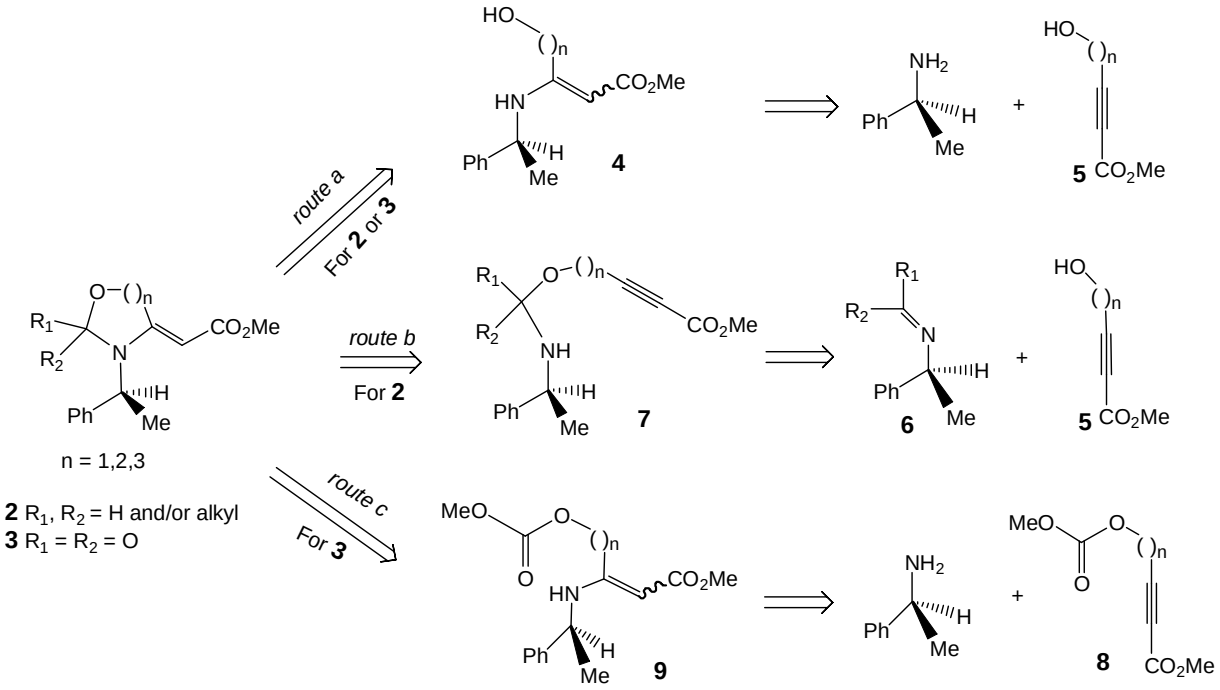
We also realized that, beside their potential as precursors of amino alcohols, these heterocycles would constitute valuable intermediates toward the synthesis of various natural products as suggested by the case of 4-alkylidene-2-oxazolidinone (**3**) (n = 1), whose related reduction products have been reported as useful synthons in the synthesis of alkaloids[5] or of compounds of biological interest.[6]

In a recent publication,[7] we described a convenient preparation of chiral cyclic β-enamino esters by a tandem Michael addition-alkylation reaction from a primary chiral amine in the presence of ω-halogeno activated alkynes, while a similar approach has been reported by others[8] for the synthesis of achiral 4-alkylidene-2-oxazolidinone starting from acetylenic carbamates. Based on these two reports, we planed to extend these approaches toward the synthesis of the target compounds **(2)** and **(3)** by reacting ω-oxygenated alkynoates with (*S*)-1-phenylethylamine (or its derivatives). We report hereafter the results of this study and in particular we will describe the variations required to successfully prepare the different targeted heterocycles.

RESULTS AND DISCUSSION

Our initial goal was the obtention of chiral amino alcohol intermediates **(4)** by reacting (*S*)-1-phenylethylamine with ω-hydroxy alkynoates **(5)** followed by a joint protection of the amino and hydroxyl groups leading to a cyclic enamino ester (Scheme 1, *route a*). However, preliminary results showed that, even if the formation as an inseparable (*E*)- and (*Z*)- mixture of the expected hydroxy enamino esters **(4)** occurred in good yields, the subsequent cyclisation step upon reaction with aldehydes, ketones (to afford **2**) or carbonates (to afford **3**) proved unsuccessful, leading either to unreacted material or to intractable mixtures. Consequently, we considered alternative strategies in which ring closure would be achieved through an intramolecular process.

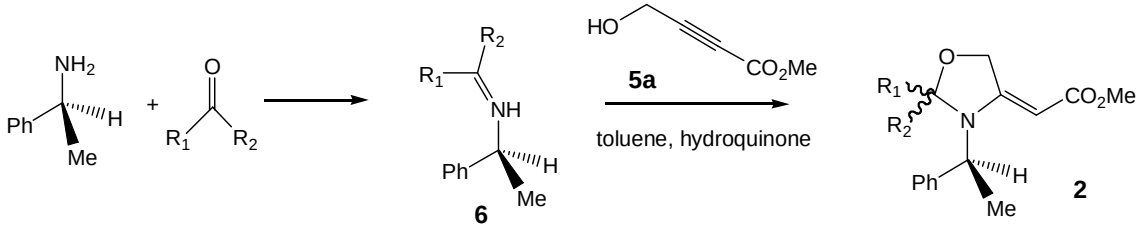
Alcohols are known to add onto imines to afford amino ethers. As far as the preparation of hemiaminals **(2)** was concerned, we envisaged that we could apply this reaction to chiral imines **(6)** and ω-hydroxy alkynoates **(5)**. The amino group of the resulting intermediates **(7)** could subsequently add to the activated triple bond in an intramolecular process to lead to the expected cyclic enamino esters **(2)** (Scheme 1, *route b*).



Scheme 1

The required chiral imines **(6a-d)** were readily obtained by addition of (*S*)-1-phenylethylamine to the appropriate aldehydes or ketones (Scheme 2). Compound **(6a)** was prepared by a standard method[9] whereas **6b-c** were synthesized according to previously described procedures[10-12] (Table 1).

After a preliminary study aimed at optimizing reaction conditions, oxazolidines **(2)** (n = 1) were obtained by reacting crude imines **(6a-d)** with methyl hydroxybutynoate **(5a)** in refluxing toluene in the presence of hydroquinone (Scheme 2). In all cases, the crude yields of the expected oxazolidines **(2)** (*E*-isomer)[13] exceeded 90 % and these products were pure enough to allow their further utilization as they were. However, they turned out to be very acid sensitive and underwent partial deprotection during chromatography on silica gel, which, in some instances, lowered very significantly isolated yields (see Entries b, c, d of Table 1). Also noteworthy was the low diastereoselectivity observed for compounds **(2c)** and **(2d)** (Table 1, Entries c and d).



Scheme 2

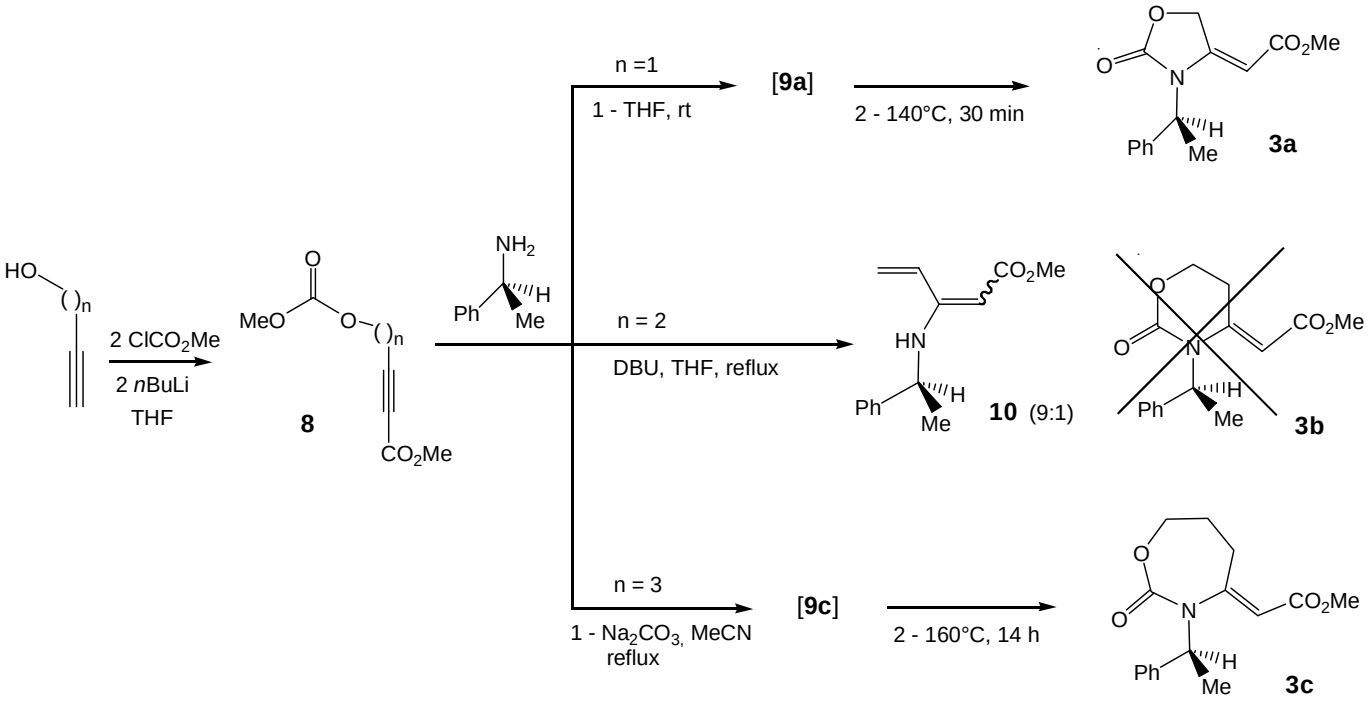
Table 1 - Preparation of oxazolidines **(2)**

Entry	R_1	R_2	Imines **(6)**	Products **(2)** (Isolated yields)	Diastereomeric ratio
a	H	H	**6a**	**2a** (80 %)	_
b	Me	Me	**6b**[10]	**2b** (20 %)	_
c	Ph	H	**6c**[11]	**2c** (56 %)	60:40
d	*t*-Bu	H	**6d**[12]	**2d** (35 %)	55:45

Unfortunately, when extended to methyl hydroxypentynoate **(5b)** and hexynoate **(5c)**, this strategy systematically failed: whatever the conditions used, we obtained complex mixtures, which restricted the scope of this methodology to the synthesis of the sole oxazolidines **(2)**.

We then turned our attention to the preparation of chiral cyclic carbamates **(3)**. Addition of (*S*)-1-phenylethylamine on ω-methoxycarbonyloxy acetylenic esters **(8)** was expected to yield the corresponding enamino esters **(9)** (Scheme 1, *route c*). In this strategy**,** intramolecular acylation of the enamino nitrogen atom by the carbonate moiety should allow the formation of the targeted compounds **(3)**.

Alkynoates **(8)** were readily obtained by reacting methyl chloroformate (2 eq.) with the dianion of the corresponding hydroxyl alkynes in THF at -78°C (Scheme 3). The addition of (*S*)-1-phenylethylamine on crude **8a** (n = 1) at room temperature in THF led indeed to the corresponding acyclic enamino ester **(9a)** which was subsequently heated neat at 140°C to afford the expected (*E*)-oxazolidinone **(3a)** in 85% yield (Scheme 3, n = 1).



Scheme 3

As far as **8b** (n = 2) was concerned, we noted that, under neutral conditions and at room temperature, no reaction occurred in the presence of the chiral amine. However, in preliminary experiments, we had observed that the condensation of the amine onto methyl hydroxypentynoate **(5b)** and hexynoate **(5c)** required heating (refluxing acetonitrile) and basic (Na_2CO_3) conditions to take place. When applied to carbonate **(8b)**, these conditions only led to degradation. However, the use of DBU as the base in refluxing THF led to the formation of unprecedented diene **(10)** as a 9:1 mixture of *E*/*Z* isomers (74%), instead of the expected oxazinone **(3b)** (Scheme 3, n = 2). The origin of compound **(10)** has not been clarified. It may stem through an elimination process from either **8b** or the intermediate enamino ester **(9b)** or the expected compound **(3b)**. Whatever the explanation, this approach constitutes an efficient, new method to prepare such dienes since reaction of an amine with a vinyl alkoxycarbonylmethyl ketone leads to Michael additions[14] rather than condensation onto the carbonyl. Compounds such as **10** could be interesting building blocks for natural product synthesis.

Finally, we succeeded in preparing (*E*)-oxazepanone **(3c)** (n = 3) in 60% yield under above conditions *via* initial formation of the acyclic enamino ester **(9c)** upon reaction of the chiral amine and **8c** in the presence of Na_2CO_3 (refluxing acetonitrile), followed by prolonged heating at 160°C (Scheme 3, n = 3).

In summary, we have developed new synthetic strategies to access efficiently to chiral 4-methoxycarbonylmethylideneoxazolidines and oxazolidinone. Attempted extension of these methodologies toward the synthesis of related oxazines and oxazinone failed. In the latter case, we rather obtained a new, activated diene. Finally, a chiral oxazepanone was successfully obtained in satisfactory yield.

EXPERIMENTAL

General. The general experimental procedures were carried out as previously described.[15]

Methylene-(1-(*S*)-phenylethyl)amine (6a). To a solution of (*S*)-1-phenylethylamine (5 g, 41.2 mmol) in dry ethanol (50 mL) was added 35% formaldehyde solution (3.7 g, 45.5 mmol). The reaction mixture was heated at reflux temperature for 12 h. After cooling to rt, the solvent was removed *in vacuo* and the crude oily imine (**6a**) (5.3 g, 97%) was pure enough to be directly engaged in the next step. IR (neat) :1670 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.25 (d, *J* = 6.7 Hz, 3H), 3.35 (br s, 2H), 3.68 (q, *J* = 6.7 Hz, 1H), 7.11-7.34 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 20.0, 59.3, 69.8, 126.7, 127.3, 128.0, 144.1.

General procedure of the preparation of 2.

A mixture of crude imines **(6a-d)** (20 mmol), methyl hydroxybutynoate **(5a)** (3.42 g, 30 mmol) and hydroquinone (2.2 g, 20 mmol) was heated at reflux temperature in anhydrous toluene (20 mL) for 24 h (for **2a** and **2c**) or 72 h (for **2b** and **2d**). The reaction mixture was concentrated *in vacuo* and

chromatographed on silica gel column eluted with CH_2Cl_2 to give the expected compounds.

(–)-Methyl [(*E*)-3-(1-(*S*)-phenylethyl)oxazolidin-4-ylidene]acetate (2a). Pale yellow oil (80 %); $[\alpha]^{20}_D$ -161° (*c* 1.34, CH_2Cl_2); IR (neat) 1640, 1620 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.60 (d, *J* = 7 Hz, 3H), 3.61 (s, 3H), 4.63 (s, 1H), 4.78-4.86 (m, 2H), 4.98 (s, 1H), 5.08-5.23 (m, 2H), 7.23-7.40 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 17.3, 50.3, 52.7, 73.7, 77.5, 81.2, 126.3, 127.9, 128.8, 139.3, 158.0, 169.2. Anal. Calcd for $C_{14}H_{17}NO_3$: C, 67.99; H, 6.93; N, 5.66. Found: C, 67.72; H, 6.84; N, 5.53.

(+)-Methyl [(*E*)-2,2-dimethyl-3-(1-(*S*)-phenylethyl)oxazolidin-4-ylidene]acetate (2b). Pale yellow oil (20 %); $[\alpha]^{20}_D$ +72° (*c* 0.98, CH_2Cl_2); IR (neat) 1640, 1620 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.47 (s, 3H), 1.54 (s, 3H), 1.77 (d, *J* = 7.2 Hz, 3H), 3.52 (s, 3H), 4.18 (m, 1H), 4.68 (q, *J* = 7.2 Hz, 1H), 5.05-5.21 (m, 2H), 7.20-7.36 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 16.0, 25.4, 26.0, 50.2, 51.8, 70.3, 79.7, 98.7, 126.2, 127.2, 128.6, 138.7, 156.3, 169.1. Anal. Calcd for $C_{16}H_{21}NO_3$: C, 69.79; H, 7.68; N, 5.08. Found: C, 69.83; H, 7.75; N, 4.98.

Methyl [(*E*)-2-phenyl-3-(1-(*S*)-phenylethyl)oxazolidin-4-ylidene]acetate (2c). Inseparable 60:40 mixture of diastereomers as a pale yellow oil (56 %); IR (neat) 1640, 1620 cm^{-1}; For the major isomer : ^1H NMR ($CDCl_3$) δ 1.32 (d, *J* = 7 Hz, 3H), 3.63 (s, 3H), 4.75 (q, *J* = 7.0 Hz, 1H), 4.64 (s, 1H), 5.15-5.51 (m, 2H), 5.68 (s, 1H), 7.11-7.37 (m, 10H); ^{13}C NMR ($CDCl_3$) δ 15.9, 50.4, 53.1, 72.7, 78.6, 94.1, 126.6, 127.4, 127.8, 128.6, 129.7, 129.8, 138.4, 138.6, 158.3, 169.2. For the minor isomer : ^1H NMR ($CDCl_3$) δ 1.53 (d, *J* = 7 Hz, 3H), 3.61 (s, 3H), 4.56 (q, *J* = 7.0 Hz, 1H), 4.64 (s, 1H), 5.15-5.51 (m, 2H), 6.02 (s, 1H), 7.11-7.37 (m,10 H); ^{13}C NMR ($CDCl_3$) δ 16.3, 50.4, 53.4, 73.1, 79.1, 95.9, 126.6, 127.0, 127.7, 128.4, 129.7, 129.8, 137.9, 139.0, 157.6, 169.1. Anal. Calcd for $C_{20}H_{21}NO_3$: C, 74.28; H6.54; N, 4.33. Found: C, 74.34; H, 6.49; N, 4.25.

Methyl [(*E*)-2-*tert*-butyl-3-(1-(*S*)-phenylethyl)oxazolidin-4-ylidene]acetate (2d). Inseparable 55:45 mixture of diastereomers as a pale yellow oil (35 %); IR (neat) 1640, 1620 cm^{-1}; For the major isomer : ^1H NMR ($CDCl_3$) δ 0.99 (s, 9H), 1.87 (d, *J* = 7 Hz, 3H), 3.55 (s, 3H), 4.39 (m, 1H), 4.72 -4.79 (m, 1H), 5.02 (m, 2H), 5.20-5.30 (m, 1H), 7.21-7.37 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 16.9, 25.1, 39.0, 50.2, 55.1, 74.0, 80.7, 104.1, 126.1, 127.2, 128.6, 139.2, 159.2, 168.9. For the minor isomer : ^1H NMR ($CDCl_3$) δ 0.97 (s, 9H), 1.67 (d, *J* = 7 Hz, 3H), 3.52 (s, 3H), 4.48 (m, 1H), 4.72 -4.79 (m, 1H), 5.09 (m, 2H), 5.20-5.30 (m, 1H), 7.21-7.37 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 14.8, 24.9, 38.9, 50.3, 57.1, 74.0, 83.8, 103.5, 126.2, 127.0, 128.6, 139.3, 159.1, 168.6. Anal. Calcd for $C_{18}H_{25}NO_3$: C, 71.25; H 8.30; N, 4.61. Found: C, 71.37; H, 8.19; N, 4.65.

General procedure for the preparation of 8.

To a solution of the appropriate ω-hydroxyalkynes (20 mmol) in anhydrous THF (150 mL) cooled at-78°C, was added dropwise a 2.5M solution of *n*-BuLi in hexane (16.8 mL, 42 mmol) under vigorous

stirring. After stirring for 20 min, methyl chloroformate (3.25 mL, 42 mmol) was added and the reaction mixture was allowed to warm to rt. The reaction mixture was washed with water (3×50 mL) and brine (50 mL). The organic layer was dried over Na_2SO_4 and concentrated in vacuo to afford the expected alkynoates **(8)** which were used without further purification in the next step.

Methyl 4-methoxycarbonyloxybut-2-ynoate (8a). Colorless oil (85 %); IR (neat) 2260, 1760, 1720 cm^{-1}, ^1H NMR ($CDCl_3$) δ 3.79 (s, 3H), 3.84 (s, 3H), 4.86 (s, 2H); ^{13} C NMR ($CDCl_3$) δ 52.7, 54.4, 55.2, 77.9, 80.4, 152.9, 154.7.

Methyl 5-methoxycarbonyloxypent-2-ynoate (8b). Colorless oil (95 %); IR (neat) 2260, 1760, 1720 cm^{-1}, ^1H NMR ($CDCl_3$) δ 2.73 (t, *J* =6.7 Hz, 2H), 3.76 (s, 3H), 3.80 (s, 3H), 4.27 (t, *J* = 6.7 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 18.6, 52.0, 54.3, 63.8, 73.5, 84.1, 153.1, 154.9.

Methyl 6-methoxycarbonyloxyhex-2-ynoate (8c). Colorless oil (95 %); IR (neat) 2260, 1760, 1720 cm^{-1}, ^1H NMR ($CDCl_3$) δ 1.96 (m, 2H), 2.48 (t, *J* = 6.5 Hz, 2H), 3.76 (s, 3H), 3.79 (s, 3H), 4.24 (t, *J* = 6.5 Hz, 2H); ^{13} C NMR ($CDCl_3$) δ 14.9, 26.4, 52.2, 54.4, 65.7, 73.1, 87.3, 153.5, 155.2.

(−)-Methyl [(*E*)-2-oxo-3-(1-(*S*)-phenylethyl)oxazolidin-4-ylidene]acetate (3a). To a solution of **8a** (262 mg, 1.5 mmol) in anhydrous THF (20 mL) was added (*S*)-1-phenylethylamine (184 mg, 1.5 mmol). After stirring at rt for 3 h, the solvent was removed under reduced pressure and the neat reaction mixture was heated at 140°C under a nitrogen atmosphere for 30 min. After cooling, recrystallization from cyclohexane afforded 333 mg (85%) of the expected compound **(3a)** as a white solid. mp = 104°C; $[\alpha]^{20}_D$ -142° (*c* 1.65, $CHCl_3$); IR (neat) 1700 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.82 (d, *J* = 7.2 Hz, 3H), 3.62 (s, 3H), 4.96 (m, 1H), 5.30 (s, 2H), 5.43 (q, *J* = 7.2 Hz, 1H), 7.01-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 15.3, 50.9, 52.4, 68.5, 89.6, 126.4, 127.9, 128.7, 137.4, 151.9, 156.3, 166.9. Anal. Calcd for $C_{14}H_{15}NO_4$: C, 64.35; H, 5.78; N, 5.36. Found: C, 64.17; H, 5.64; N, 5.29.

Methyl 3-(1-(*S*)-phenylethylamino)pent-2,4-dienoate (10). To a solution of **8b** (1.7 g, 9.13 mmol) in anhydrous THF (25 mL) were added (*S*)-1-phenylethylamine (785 mg, 6.48 mmol) and DBU (98 mg, 0.64 mmol). After stirred at reflux temperature for 2 days, the solvent was removed *in vacuo*. The reaction mixture was chromatographed on silica gel eluted with AcOEt/cyclohexane (4:6) to yield 1.1 g (74%) of a 9:1 diastereomeric mixture of dienes **(10)** as pale yellow oil. IR (neat) 1670, 1630, 1560 cm^{-1}; For the major isomer :^1H NMR ($CDCl_3$) δ 1.51 (d, *J* = 6.7 Hz, 3H), 3.68 (s, 3H), 4.55-4.66 (m, 1H), 4.75 (s, 1H), 5.24 (dd, *J* = 11.0 and 1.5 Hz, 1H), 5.65 (dd, *J* =17.0 and 1.5 Hz, 1H), 6.24 (dd, *J* = 11.0 and 17.0 Hz, 1H), 7.20-7.32 (m, 5H), 8.75 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 24.7, 50.0, 53.0, 81.1, 121.3, 125.4, 127.0, 128.6, 131.2, 144.6, 160.5, 171.0. Anal. Calcd for $C_{14}H_{17}NO_2$: C, 72.70; H, 7.40; N, 6.05. Found: C, 72.77; H, 7.49; N, 6.02.

(−)-Methyl [(*E*)-2-oxo-3-(1-(*S*)-phenylethyl)oxazepan-4-ylidene]acetate (3c). To a solution of **8c** (400 mg, 1.99 mmol) in anhydrous acetonitrile (15 mL) were added 1-(*S*)-phenylethylamine (230 mg, 1.9

mmol) and Na_2CO_3 (807 mg, 7.6 mmol). After stirring at reflux temperature for 3 days, the reaction mixture was cooled, filtrated and the solvent was removed under reduced pressure. The neat reaction mixture was then heated at 160°C under a nitrogen atmosphere for 14 h. After cooling, column chromatography on silica gel eluted with AcOEt/cyclohexane (2:8) afforded 309 mg (60 %) of the expected compound **(3c)** as a white solid. mp = 69°C; $[\alpha]^{20}_D$ -257° (*c* 1.29, $CHCl_3$); IR (neat) 1670, 1580 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.55 (d, *J* = 7.0 Hz, 3H), 1.82-1.97 (m, 2H), 3.06-3.38 (m, 4H), 3.61 (s, 3H), 4.68 (s, 1H), 4.87 (q, *J* = 7.0 Hz, 1H), 7.21-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 16.8, 20.9, 32.8, 47.2, 49.9, 52.9, 77.9, 126.5, 127.4, 128.6, 140.4, 164.9 (2C), 170.0. Anal. Calcd for $C_{16}H_{19}NO_4$: C, 75.24; H, 7.80; N, 5.16. Found: C, 75.46; H, 5.90; N, 5.05.

REFERENCES

1. G. M. Coppola, H. F. Schuster, « Asymmetric Synthesis : Construction of chiral molecules using amino acids », ed. by Wiley-Interscience, New York, 1986.
2. D. J. Ager, I. Prakash, and D. R. Schaad, *Chem. Rev.*, 1996, **96**, 835.
3. F. Segat-Dioury, O. Lingibé, B. Graffe, M.-C. Sacquet, and G. Lhommet, *Tetrahedron*, 2000, **56**, 233.
4. S. Calvet, O. David, C. Vanucci-Bacqué, M.-C. Fargeau-Bellassoued, and G. Lhommet, *Tetrahedron*, 2003, **59**, 6333.
5. a) D.-C. Ha, K.-E Kil, K.-S. Choi, and H.-S. Park, *Tetrahedron Lett*., 1996, **37**, 5723. b) D.-C. Ha, S.-H. Park, K.-S. Choi, and C.-S. Yun, *Bull. Korean Chem. Soc*., 1998, **19**, 728.
6. a) D. H. Kim, S. J. Chung, E.-J. Kim, and G. R. Tian, *Bioorg. Med. Chem. Lett*., 1998, **8**, 859. b) S. J. Chung, S. Chung, H. S. Lee, E.-J. Kim, K. S. Oh, H. S. Choi, K. S. Kim, Y. J. Kim, J. H. Hahn, and D. H. Kim, *J. Org. Chem*., 2001, **66**, 6462.
7. O. David, M.-C. Fargeau-Bellassoued, and G. Lhommet, *Tetrahedron Lett*., 2002, **43**, 3471.
8. a) Y. Tamaru, M. Kimura, S. Tanaka, S. Kure, and Z. Yoshida, *Bull. Chem. Soc. Jpn,* 1994, **67**, 2838. b) K. Ohe, T. Ishihara, N. Chatani, Y. Kawasaki, and S. Murai, *J. Org. Chem*., 1991, **56**, 2267.
9. S. H. Pine and B. L. Sanchez, *J. Org. Chem*., 1971, **36**, 829.
10. A. Bernadi, C. Gennari, J. M. Goodman, V. Leue, and I. Paterson, *Tetrahedron*, 1995, **51**, 4853.
11. F. A. Davis, J. P. McCauley Jr, S. Chattopadhyay, M. E. Harakal, J. C. Towson, W. H. Watson, and I. Tavanaiepour, *J. Am. Chem. Soc*., 1987, **109**, 3370.
12. R. D. Guthrie, D. A. Jaeger, W. Meister, and D. J. Cram, *J. Am. Chem. Soc*., 1971, **93**, 5137.
13. J. P. Michael and D. Gravestock, *Eur. J. Org. Chem*., 1998, 865.
14. A. Esposito, M. G. Perino, and M. Taddei, *Eur. J. Org. Chem*., 1999, 931.
15. O. David, C. Bellec, M.-C. Fargeau-Bellassoued, and G. Lhommet, *Heterocycles*, 2001, **55**, 1689.