Synthetic Studies of Carbapenem and Penem Antibiotics. II. Synthesis of 3-Acetyl-2-azetidinones by (2+2) Cycloaddition of Diketene and Schiff Bases

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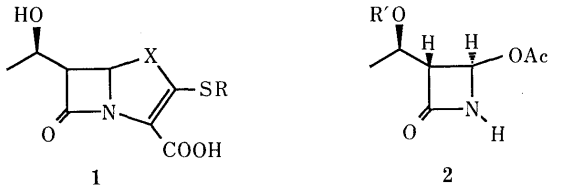
It was found that (2+2) cycloaddition reaction of diketene with Schiff bases was effectively promoted by imidazole as a catalyst to afford 3-acetyl-2-azetidinone derivatives 4. As an application of this new method, a practical asymmetric synthesis of 4 and its conversion into (3*S*,4*S*)-4-carboxy-1-(di-*p*-anisylmethyl)-3-[(*R*)-1-hydroxyethyl]-2-azetidinone, which is a key intermediate for the synthesis of carbapenem and penem antibiotics, were accomplished.

Keywords penem; carbapenem; 3-acetyl-2-azetidinone; (*R*)-1-hydroxyethyl group; diketene; (2+2) cycloaddition; asymmetric synthesis

Over the years, much effort has been directed to the stereocontrolled total synthesis of carbapenems and penems **1**, which exhibit potent, broad-spectrum antimicrobial activity and excellent stability to β-lactamases. These compounds have an (*R*)-1-hydroxyethyl group, which is essential for their antibiotic activity and stability against β-lactamases, at C-6. For the purpose of total synthesis of **1**, (3*R*,4*R*)-4-acetoxy-3-[(*R*)-1-hydroxyethyl]-2-azetidinone **2** has been widely used as a key intermediate[1] (Fig. 1). In the previous paper,[1l] we reported that oxymercuration–reduction of 4-carboxy-3-ethenyl-2-azetidinone gave the (*R*)-1-hydroxyethyl derivative **3** highly stereoselectively, and compound **3** could be easily converted into **2**. 3-Acetyl-2-azetidinone **4** was also considered to be a useful precursor of **3** as well as 3-ethenyl-2-azetidinone, because the highly stereoselective reduction of the 3-acetyl group to an (*R*)-1-hydroxyethyl group was reported.[2] However, a practical and effective synthesis of **4** has not been reported. Therefore, we initiated studies on the practical and high-yield synthesis of **4** by the new method. In this paper, we describe our investigation to develop a new, effective synthetic method for **4** and its application to the asymmetric synthesis of **4**[3] (Chart 1).

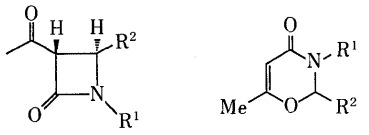
Synthetic Design The (2+2) cycloaddition reaction of ketene with Schiff bases has been widely used for the preparation of the β-lactam ring system.[4] In applying this method to the synthesis of **4**, it was considered that acetylketene should be a reasonable precursor and **4** should be obtained in one step. Acetoacetyl chloride seemed to be the most reasonable substrate to prepare acetylketene. But it is not stable at ambient temperature and its preparation and handling should be done at quite low temperature.[5] Therefore we considered that it was not suitable as a substrate for large-scale production. Thus diketene, an equivalent for acetylketene, was selected as a starting material because its availability and handling seemed to be much better than those of acetoacetyl chloride. After our study was completed and a patent concerning this work was filed,[3a] the preparation of 3-acetyl-2-azetidinone using acetoacetyl chloride or diketene was reported by Kato *et al.*[6a] albeit in low yields. That is, (2+2) cycloaddition reaction of acetylketene, which was prepared by the treatment of acetoacetyl chloride with triethylamine, with *N*-benzylidene-*n*-propylamine **5b** gave 3-acetyl-4-phenyl-1-*n*-propyl-2-azetizinone **4b** in 14% yield accompanied with 3,4-dihydro-6-methyl-2-phenyl-3-*n*-propyl-2*H*-1,3-oxazin-4-one **6b** in 16% yield. It was also reported that treatment of Schiff bases with diketene at room temperature for 1 d



1 2

X=CH_2, CH–Me, S R′ : protecting group

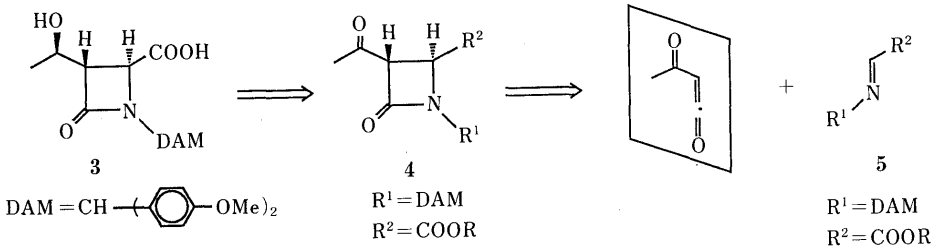
Fig. 1



4a–d **6a–d**

a : R^1=CH_2Ph, R^2=Ph
b : R^1=*n*-Pr, R^2=Ph
c : R^1=iso-Pr, R^2=Ph
d : R^1=*tert*-Bu, R^2=Ph

Fig. 2



3 **4** **5**

DAM=CH–⟨⟩–$(OMe)_2$ R^1=DAM, R^2=COOR R^1=DAM, R^2=COOR

Chart 1

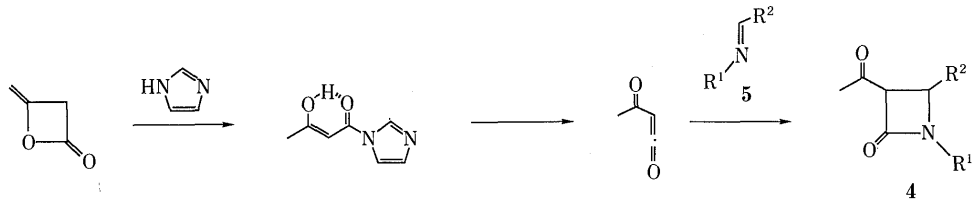
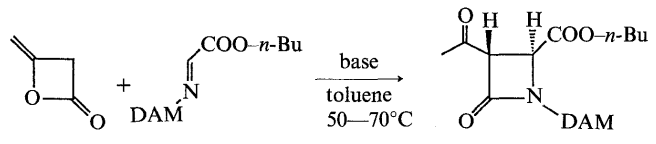


Chart 2. Working Hypothesis

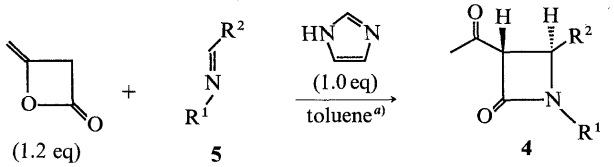
TABLE I. Studies of Bases



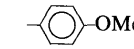
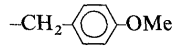
(1.2 eq) **5e** **4e**

Entry	Base (eq)	Yield (%)
1	Triethylamine (1.0)	0
2	Pyridine (1.5)	0
3	Imidazole (1.0)	88
4	Imidazole (0.5)	81
5	Imidazole (0.1)	60
6	4-Methylimidazole (1.0)	55
7	2-Methylimidazole (1.0)	5
8	1,2,3-Triazole (1.0)	0

TABLE II. Synthesis of **4**



(1.2 eq) **5** **4**

Product	R^1	R^2	Yield (%)
4f	–DAM	$-COOCH_2Ph$	72
4g		–COO–*n*-Bu	80
4h		–COO–*n*-Bu	76
4i	$-CH_2Ph$	–COO–*n*-Bu	81
4j	–Ph	–Ph	30
4k	–DAM	$-(CH_2)_2-Ph$	17

a) All reactions except that to give **4k** were carried out at 50°C. **4k** was obtained at 25°C.

in the presence of triethylamine afforded β-lactam **4a—d** (4—19%) and 1,3-oxazin-4-one **6a—d** (4—39%)[6a] (Fig. 2).

In order to establish a simple and high-yield method to prepare **4** using (2+2) cycloaddition of diketene and Schiff bases **5**, we focused our attention on how to convert diketene into acetylketene effectively. We presumed that imidazole might react with diketene to form 1-acetoacetylimidazole, which could be transformed into acetylketene by imidazole because of the high acidity of the α-proton in 1-acetoacetylimidazole (Chart 2).

Preparation of 4 To test our hypothesis, the imine **5e** was treated with 1.2 eq of diketene in the presence of 1 eq of imidazole at 50 °C in toluene and the expected cycloaddition reaction took place smoothly to give **4e** in 88% yield (Table I). The stereochemistry of **4e** was assigned as 3,4-*trans* on the basis of the coupling constant between H-3 and H-4 in the proton nuclear magnetic resonance (^{1}H-NMR) spectrum ($J_{3,4}$ = 2.3 Hz), whereas the cycloaddition of ketene with a Schiff base affords generally the *cis* isomer.[4] The outcome was considered to be due to epimerization at C-3, that is, the predominantly formed *cis* isomer was transformed into the thermodynamically more stable *trans* isomer in the presence of imidazole.

On the other hand, the desired product **4e** was not obtained by the treatment of the imine **5e** with 1.2 eq of diketene in the presence of 1 eq of triethylamine at 50 °C or 1.5 eq of pyridine at 70 °C in toluene.

In order to confirm our working hypothesis and to generalize this new method, we further studied the most suitable promoter in this reaction. The results are shown in Table I. Imidazole derivatives such as 2- and 4-methylimidazole were also effective and gave **4e** in 5% and 55% yields, respectively, under the same reaction conditions as described above, while 1,2,3-triazole did not afford **4e**. When 0.5 and 0.1 eq molar ratio of imidazole were used under the same reaction conditions, **4e** was obtained in 81% and 60% yields, respectively. Thus, imidazole appears to be the most effective catalyst, supporting our presumed reaction mechanism. The reactions using various types of imines **5f—k** were also investigated. Treatment of **5f—j** with 1.2 eq of diketene in the presence of 1 eq of imidazole in toluene at 50 °C gave **4f—j** in fair to good yields. In the case of **5k**, **4k** was formed in a trace amount under the same reaction conditions, but the yield improved to 17% when the reaction was carried out at 25 °C (Table II). Further studies on the modification of reaction conditions seemed to be necessary to improve the yield of the (2+2) cycloaddition product in the case of Schiff bases derived from the aliphatic aldehydes.[3c,d]

Asymmetric Synthesis of 4 and Preparation of Optically Active 3 As metioned above, we found a new, effective method to prepare 3-acetyl-2-azetidinones **4**. So we aimed to apply this procedure to the asymmetric synthesis of **4** and preparation of optically pure **3**. We considered that Schiff base **5l** derived from *l*-menthyl glyoxylate[7] would be a useful and practical chiral source, because we presumed that it would cause asymmetric induction, and that optical resolution of the diastereomers by fractional recrystallization would be possible, based on a study of 3-ethenyl-2-azetidinone.[1l] The Schiff base **5l** was treated with diketene in the presence of 1 eq of imidazole at 50 °C, and the (2+2) cycloaddition reaction took place smoothly to give a diastereomeric mixture of (−)-**4l** and (−)-**4m** in a ratio of 3:2 (by ^{1}H-NMR in C_6D_6) in 82% yield. Fractional recrystallization of the mixture from *n*-hexane and carbon tetrachloride gave the major product (−)-**4l** as colorless crystals (mp 99—100 °C). The minor product (−)-**4m** was also obtained as colorless crystals (mp 123—125 °C) by recrystallization of the concentrated filtrate from *n*-hexane

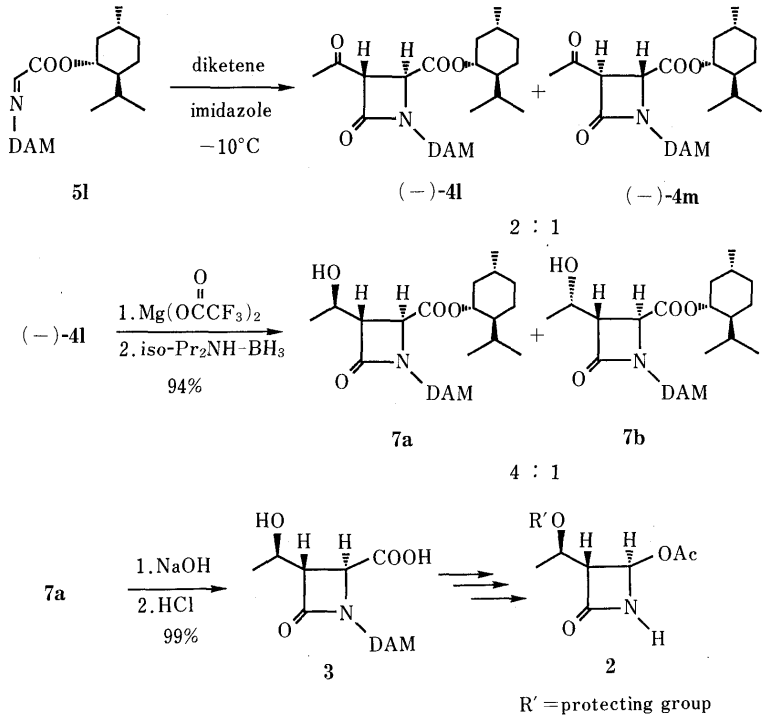


Chart 3

and carbon tetrachloride. It is well known that the optical yield is dependent on the reaction temperature. When this reaction was carried out at −10 to −12 °C, the diastereomeric ratio of (−)-**4l** and (−)-**4m** was improved to 2:1 (by ^{1}H-NMR in C_6D_6) and the chemical yield was also improved to 90%. Conversion of (−)-**4l**, which was the major isomer in the (2+2) cycloaddition reaction, into **3** was performed as shown in Chart 3. The stereoselective reduction of (−)-**4l** by di-isopropylamine–borane complex in the presence of magnesium trifluoroacetate[2c] gave a mixture of **7a** and **7b** (**7a** : **7b** = 4 : 1) in 94% yield. Compound **7a** could be separated by crystallization from *n*-hexane and diethyl ether. Alkaline hydrolysis of **7a** gave the carboxylic acid **3** in 99% yield. The spectral data (infrared (IR) and ^{1}H-NMR) and physical data (mp and $[\alpha]_D$) were identical with those of an authentic sample.[1l]

Conclusion

In summary, we have developed new methodology to synthesize **4** using the (2+2) cycloaddition reaction of diketene with Schiff bases and have succeeded in establishing an effective and practical procedure to provide **4**. We also applied this reaction to obtain optically pure **3**, which is a useful intermediate for the preparation of penem and carbapenem antibiotics. Concerning the reaction mechanism, our hypothesis is that the reaction is a (2+2) cycloaddition involving acetylketene that is formed from diketene by imidazole as a catalyst. It was confirmed that imidazole activated diketene catalytically, but we have no definite evidence for the formation of acetylketene and there is no reasonable explanation for the different stereoselectivity between the following two (2+2) cycloaddition reactions. That is, (2+2) cycloaddition reaction of diketene with **5l**, which contained an *l*-menthyl moiety, gave the (4*S*)-isomer as the major product as mentioned above, while the asymmetric reaction of crotonyl chloride with **5l** in the presence of triethylamine afforded predominantly the (4*R*)-isomer.[1l] Further studies on the reaction mechanism are under-way in our laboratory.

Experimental

Melting points were measured using a Thomas–Hoover capillary melting point apparatus and were not corrected. IR spectral measurements were carried out with a Hitachi 260-10 IR spectrometer. ^{1}H-NMR spectra were measured with JEOL FX-90Q (90 MHz) and GX-270 (270 MHz) spectrometers. Chemical shift values are expressed as ppm downfield from tetramethylsilane used as an internal standard (δ-value). Measurements of optical rotation were performed with JASCO DIP-181 and DIP-370 digital polarimeters. Silica gel 60 (70—230 mesh, E. Merck) was used as an adsorbent for column chromatography. Preparative thin layer chromatography (preparative TLC) was performed on Silica gel 60 F_{254} TLC plates (E. Merck).

1) Preparation of 4. 3-Acetyl-1-(di-*p*-anisylmethyl)-4-*n*-butoxycarbonyl-2-azetidinone (4e) A mixture of di-*p*-anisylmethylamine (2.43 g, 10.0 mmol) and *n*-butyl glyoxylate monohydrate (1.78 g, 12.0 mmol) in toluene (95 ml) was dehydrated azeotropically under reflux to give a solution of the Schiff base **5e**. After addition of imidazole (0.68 g, 10.0 mmol), a solution of diketene (1.01 g, 12.0 mmol) in toluene (30 ml) was added dropwise at 50 °C over 2 h. After being stirred for 15 min at 50 °C, the reaction mixture was cooled to room temperature and washed successively with 2 N HCl, aqueous $NaHCO_3$ and brine. The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel to give **4e** as a colorless solid (3.86 g, 88%). mp 92.5—94.0 °C. IR (Nujol): 1765, 1740, 1715 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 0.89 (3H, t, J=7.4 Hz), 1.2—1.4 (2H, m), 1.4—1.6 (2, m), 2.34 (3H, s), 3.79 (6H, s), 3.88 (2H, dt, J=1.7, 6.6 Hz), 4.24 (1H, d, J=2.3 Hz), 4.47 (1H, d, J=2.3 Hz), 5.85 (1H, s), 6.8—6.9 (4H, m), 7.1—7.3 (4H, m). *Anal.* Calcd for $C_{25}H_{29}NO_6$: C, 68.32; H, 6.65; N, 3.19. Found: C, 67.93; H, 6.69; N, 3.19.

Compounds **4f**—**k** were obtained similarly.

3-Acetyl-1-(di-*p*-anisylmethyl)-4-benzyloxycarbonyl-2-azetidinone (4f) A colorless solid (72% yield). mp 135—138 °C. IR (Nujol): 1762, 1739, 1719 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 2.28 (3H, s), 3.73 (6H, s), 4.19 (1H, d, J=2 Hz), 4.45 (1H, d, J=2 Hz), 4.87 (2H, s), 5.80 (1H, s), 6.5—7.4 (13H, m). *Anal.* Calcd for $C_{28}H_{27}NO_6$: C, 71.02; H, 5.75; N, 2.96. Found: C, 70.90; H, 5.80; N, 2.99.

3-Acetyl-1-*p*-methoxyphenyl-4-*n*-butoxycarbonyl-2-azetidinone (4g) A viscous oil (80% yield). IR (neat): 1735 (br) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ:

0.89 (3H, t, J=7.3 Hz), 1.25—1.50 (2H, m), 1.50—1.75 (2H, m), 2.41 (3H, s), 3.78 (3H, s), 4.19 (2H, t, J=6.6 Hz), 4.39 (1H, d, J=2.3 Hz), 4.94 (1H, d, J=2.3 Hz), 6.70—7.50 (4H, m). *Anal.* Calcd for $C_{17}H_{21}NO_5$: C, 63.93; H, 6.63; N, 4.39. Found: C, 63.60; H, 6.62; N, 4.34.

3-Acetyl-1-*p*-methoxybenzyl-4-*n*-butoxycarbonyl-2-azetidinone (4h) A viscous oil (80% yield). IR (neat): 1760, 1740 (sh), 1710 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 0.93 (3H, t, J=7.3 Hz), 1.25—1.50 (2H, m), 1.50—1.75 (2H, m), 2.34 (3H, s), 3.80 (3H, s), 4.27 (1H, d, J=2.3 Hz), 4.37 (1H, d, J=2.3 Hz), 3.8—4.4 (4H, m), 6.87 (2H, d, J=8.9 Hz), 7.13 (2H, d, J=8.9 Hz). *Anal.* Calcd for $C_{18}H_{23}NO_5$: C, 64.85; H, 6.95; N, 4.20. Found: C, 64.42; H, 6.92; N, 4.22.

3-Acetyl-1-benzyl-4-*n*-butoxycarbonyl-2-azetidinone (4i) A viscous oil (81% yield). IR (neat): 1760, 1740 (sh), 1715 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 0.92 (3H, t, J=7.4 Hz), 1.20—1.50 (2H, m), 1.50—1.70 (2H, m), 2.35 (3H, s), 4.08 (2H, t, J=6.9 Hz), 4.30 (1H, d, J=2.0 Hz), 4.40 (1H, d, J=2.0 Hz), 4.49 (2H, m), 7.23 (5H, s). *Anal.* Calcd for $C_{17}H_{21}NO_4$: C, 67.31; H, 6.98; N, 4.62. Found: C, 67.56; H, 6.96; N, 4.79.

3-Acetyl-1-phenyl-4-phenyl-2-azetidinone (4j) A viscous oil (30% yield). IR ($CHCl_3$): 1747, 1717 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 2.39 (3H, s), 4.14 (1H, d, J=2.6 Hz), 5.48 (1H, d, J=2.6 Hz), 7.27 (5H, s), 7.37 (5H, s). *Anal.* Calcd for $C_{17}H_{15}NO_2$: C, 76.96; H, 5.70; N, 5.28. Found: C, 76.64; H, 5.71; N, 5.24.

3-Acetyl-1-(di-*p*-anisylmethyl)-4-(2-phenylethyl)-2-azetidinone (4k) A viscous oil (17% yield). IR (neat): 1742, 1704 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.75—1.95 (2H, m), 2.16 (3H, s), 2.35—2.65 (2H, m), 3.60 (1H, d, J=2.0 Hz), 3.80 (6H, s), 4.03 (1H, m), 5.78 (1H, s), 6.7—7.4 (13H, m). *Anal.* Calcd for $C_{28}H_{29}NO_4$: C, 75.82; H, 6.59; N, 3.16. Found: C, 75.03; H, 6.63; N, 3.23.

2) Asymmetric Synthesis of 4. (3*S*,4*S*)-3-Acetyl-1-(di-*p*-anisylmethyl)-4-(*l*-menthyloxycarbonyl)-2-azetidinone ((−)-41) and (3*R*,4*R*)-3-Acetyl-1-(di-*p*-anisylmethyl)-4-(*l*-menthyloxycarbonyl)-2-azetidinone ((−)-4m) Method A A mixture of di-*p*-anisylmethylamine (1.118 g, 4.60 mmol) and *l*-menthyl glyoxylate monohydrate (1.058 g, 4.60 mmol) in toluene (43 ml) was dehydrated azeotropically under reflux to give a solution of the Schiff base **5l**. After addition of imidazole (313 mg, 4.60 mmol), a solution of diketene (4.646 g, 5.50 mmol) in toluene (14 ml) was added dropwise at 50 °C over 2 h. After being stirred for 15 min at 50 °C, the reaction mixture was cooled to room temperature and washed successively with 2 N HCl, aqueous $NaHCO_3$, and brine. The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel to give a mixture of (−)-**4l** and (−)-**4m** (3:2, by ^{1}H-NMR analysis in C_6D_6) as a colorless solid (1.965 g, 82%). It was fractionally recrystallized from *n*-hexane and CCl_4 to separate (−)-**4l**. The filtrate was concentrated and the residue was crystallized from *n*-hexane and CCl_4 to give (−)-**4m**.

(−)-**4l**: mp 99—100 °C. $[\alpha]_D^{29}$ −1.1° (c=0.53, $CHCl_3$). IR (Nujol): 1764, 1739, 1714 cm^{-1}. ^{1}H-NMR (C_6D_6) δ: 0.5—1.7 (18 H, m), 1.90 (3H, s), 3.27 (3H, s), 3.29 (3H, s), 4.00 (1H, d, J=2 Hz), 4.74 (1H, dt, J=4.6, 10.9 Hz), 4.80 (1H, d, J=2 Hz), 5.93 (1H, s), 6.55—7.50 (8H, m). *Anal.* Calcd for $C_{31}H_{39}NO_6$: C, 71.37; H, 7.54; N, 2.69. Found: C, 71.18; H, 7.50; N, 2.69.

(−)-**4m**: mp 123—125 °C. $[\alpha]_D^{29}$ −42° (c=0.46, $CHCl_3$). IR (Nujol): 1770, 1738, 1713 cm^{-1}. ^{1}H-NMR (C_6D_6) δ: 0.5—1.8 (18H, m), 1.93 (3H, s), 3.29 (3H, s), 3.32 (3H, s), 4.09 (1H, d, J=2 Hz), 4.70 (1H, dt, J=4.3, 10.9 Hz), 4.71 (1H, d, J=2 Hz), 5.92 (1H, s), 6.50—7.50 (8H, m). *Anal.* Calcd for $C_{31}H_{39}NO_6$: C, 71.37; H, 7.54; N, 2.69. Found: C, 71.06; H, 7.62; N, 2.52.

Method B A mixture of di-*p*-anisylmethylamine (1.00 g, 4.12 mmol) and *l*-menthyl glyoxylate monohydrate (947 mg, 4.12 mmol) in toluene (100 ml) was dehydrated azeotropically under reflux to give a solution of the Schiff base **5l**. After addition of imidazole (336 mg, 6.18 mmol), a solution of diketene (519 mg, 6.18 mmol) in toluene (5 ml) was added dropwise at −10 to −12 °C over 0.5 h. After being stirred at the same temperature overnight, the reaction mixture was washed successively with brine, dilute HCl, water, aqueous $NaHCO_3$ and water. The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel to give a mixture of (−)-**4l** and (−)-**4m** (2:1, by ^{1}H-NMR analysis in C_6D_6) as a colorless solid (1.93 g, 90%).

3) Preparation of 3. (3*S*,4*S*)-1-(Di-*p*-anisylmethyl)-3-[(*R*)-1-hydroxyethyl]-4-[*l*-menthyloxycarbonyl]-2-azetidinone (7a) Magnesium trifluoroacetate (425 mg, 1.7 mmol) was added to a solution of (−)-**4l** (165 mg, 0.32 mmol) in Et_2O (5 ml) with dry ice–acetone cooling. The mixture was treated with a solution of diisopropylamine–borane complex (about 100 mg, 0.9 mmol) in tetrahydrofuran (THF). After being stirred for 0.5 h, the reaction mixture was diluted with dilute HCl and EtOAc. The organic layer was washed with water, dried over Na_2SO_4, and concentrated *in vacuo* to give a mixture of **7a** and **7b** (158 mg, 94%). The ratio of (5*R*)-isomer **7a** and (5*S*)-isomer **7b** was (4:1) by high performance liquid chromatography (HPLC) analysis. Further, the obtained mixture was fractionally recrystallized with *n*-hexane–Et_2O to separate **7a**. **7a**: mp 105—106 °C. $[\alpha]_D^{29}$ −16° (c=0.48, $CHCl_3$). IR ($CHCl_3$): 3400 (br), 1740 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 0.5—1.9 (18 H, m), 1.27 (3H, d, J=6.3 Hz), 3.15 (1H, dd, J=2.6, 3.0 Hz), 3.78 (3H, s), 3.79 (3H, s), 4.09 (1H, d, J=2.6 Hz), 4.32 (1H, m), 4.62 (1H, dt, J=4.3, 10.9 Hz), 5.83 (1H, s), 6.7—7.4 (8H, m). *Anal.* Calcd for $C_{31}H_{41}NO_6$: C, 71.10; H, 7.89; N, 2.68. Found: C, 70.54; H, 7.86; N, 2.67.

(3*S*,4*S*)-4-Carboxy-1-(di-*p*-anisylmethyl)-3-[(*R*)-1-hydroxyethyl]-2-azetidinone (3) A 1 N NaOH solution (0.06 ml) was added to a solution of **7a** (30 mg, 0.057 mmol) in THF (0.9 ml) and MeOH (0.45 ml) at room temperature. After being stirred for 4 h, the reaction mixture was neutralized with 1 N HCl, concentrated *in vacuo*, and diluted with Et_2O. The mixture was made alkaline with 1 N NaOH (0.1 ml). The aqueous layer was separated, acidified with 1 N HCl (0.12 ml) and extracted with Et_2O. The organic layer was washed with water, dried over Na_2SO_4, and concentrated *in vacuo* to give **3a** (22 mg, quantitative yield). An analytical sample was prepared by crystallization from CH_2Cl_2–CCl_4. mp 86—88 °C (lit.[11] mp 86—88 °C). $[\alpha]_D^{28}$ +12.0° (c=0.21, $CHCl_3$) (lit.[11] $[\alpha]_D^{27}$ +11.0° (c=0.21, $CHCl_3$). The IR and ^{1}H-NMR spectral data were identical with the reported data.[11]

References and Notes

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