

Synthesis of 3-Acetyl-2-azetidiones by [2+2] Cycloaddition of Diketene and Imines

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The 2-azetidione derivatives, which can be prepared by [2+2] cycloaddition reaction of ketenes and imines, have been extensively employed for the synthesis of carbapenems and other β -lactam antibiotics.¹ The [2+2] cycloaddition of chiral imines to ketenes appeared to be the most amenable to large scale synthesis of chiral 2-azetidiones.² Actually in many cases asymmetric inductions have been achieved by employing the strategy in the synthesis of chiral 2-azetidione derivatives, which are then further converted to thienamycin analogs.³ It has been proved that the adjacent chiral centers existing either in ketenes or imines induce asymmetric cycloaddition. Especially, many chiral 2-azetidione derivatives have been obtained from cycloaddition of ketenes with chiral imines. For the preparation of 3-(2-hydroxyethyl)-2-azetidione synthons for the synthesis of thienamycin analogs, it is necessary to control the chiral center at the 2-hydroxyethyl group as well as those at the 3- and 4-position on the β -lactam ring. (*S*)-3-Hydroxybutanoate that can be prepared from acetoacetate by reduction with yeast has been employed for the construction of chiral β -lactam rings.⁴ But in this case, the 2-azetidione derivatives can be employed for the synthesis of thienamycin skeleton after the hydroxy group of their 3-hydroxyethyl side chain is inverted.

During the course of synthesis of thienamycin analogs, we examined formation of 3-acetyl-2-azetidione derivatives and reduction of the acetyl group to (*R*)-1-hydroxyethyl group. Acetylketene was not suitable for the reaction with imines due to its instability at ambient temperature. But diketene, an acetylketene equivalent, has been examined for the synthesis of 3-acetyl-2-azetidiones⁵ and it has been reported that the cycloaddition reaction can proceed in a highly stereoselective manner to afford 3,4-*trans*-3-acetyl-2-azetidiones. The reaction has been applied for the synthesis of (3*R*,4*R*)-4-acetoxy-3-[(*R*)-1-(*t*-butyldimethylsilyloxy)ethyl]-2-azetidione⁶ and 3,4-*trans*-1-(*p*-methoxyphenyl)-3-acetyl-4-ethynyl-2-azetidione,⁷ which are chiral intermediates of 1 β -methylcarbapenem antibiotics.⁸ The 3-acetyl group at the 2-azetidione ring has been stereoselectively reduced to the 3-[(*R*)-1-hydroxyethyl]-2-azetidione derivatives with potassium tri-*sec*-butylborohydride in the presence of potassium iodide,⁶ or by employing microorganisms.⁷ However, application of the cycloaddition reaction of diketene with imines to the construction of 2-azetidione derivatives is limited because diketene favors dimerization to a pyridone derivative. In the present study, we would like to report our results on the [2+2] cycloaddition reactions of diketene with various imines and the possibilities for the preparation of chiral



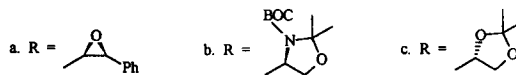
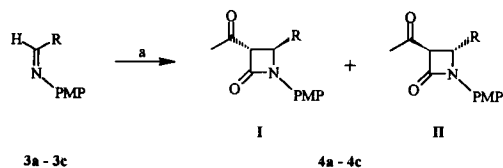
a) imidazole, diketene/CH₂Cl₂, -30 °C, 12h

Scheme 1.

Table 1. Yield of 3,4-*trans*-3-acetyl-2-azetidiones prepared

Product	R	R'	Yield
2a	Ph	CH ₂ CH(OCH ₃) ₂	41
2b	Ph	CH ₂ CO ₂ Et	43
2c	Ph	CH(CH ₂ Ph)CO ₂ CH ₃	49
2d	COPh	PMP	60
2e	CO ₂ CH ₃	PMP	86
2f	(CH ₂) ₂ OCH ₂ Ph	PMP	10

PMP = *p*-methoxyphenyl



a) imidazole, diketene / CH₂Cl₂, -30 °C, 12h

Scheme 2.

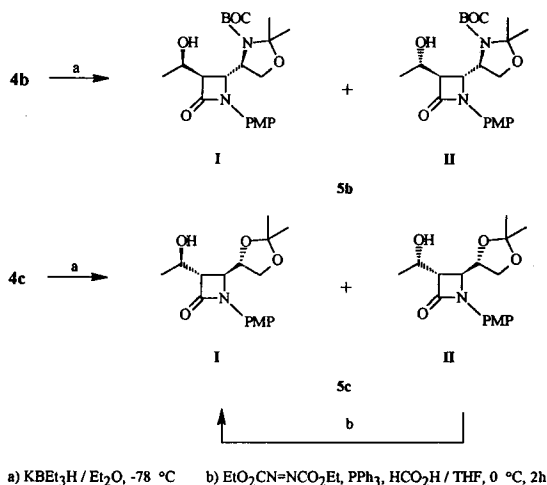
synthons, which can be utilized for the synthesis of 1-hydroxy or 1-aminocarbapenems.

The reaction of imines (**1**), obtained from aldehydes and amines, with diketene gave 3,4-*trans*-3-acetyl-2-azetidiones (**2**) in 10-66% yields (Scheme 1 and Table 1).

We examined the [2+2] cycloaddition reaction of diketene with several chiral imines. The imine (**3a**) prepared from a chiral (2*S*,3*S*)-2,3-epoxy-3-phenylpropanal and *p*-anisidine was reacted with diketene (5 equiv.) in methylene chloride at -30 °C to the presence of imidazole (1.5 equiv.) to give a mixture of (3*S*,4*S*)- and (3*R*,4*R*)-3-acetyl-4-[(1*R*,2*S*)-1,2-epoxyethyl-2-phenyl]-1-(*p*-methoxyphenyl)-2-azetidione (**4a-I** and **4a-II**) in 55% yield. The ratio of **I** and **II** was found to be 1 : 7 from their NMR spectra. The two isomers were actually isolated by silica gel column chromatography with ethyl acetate-hexane-methylene chloride (3 : 10 : 1). The imine (**3b**) prepared from *t*-butyl (*S*)-4-formyl-2,2-dimethyl-1,3-oxazolidine-3-carboxylate and *p*-anisidine, when it was reacted with diketene, gave (3*R*,4*R*)-3-acetyl-4-[(4*R*)-3-(*t*-butoxycarbonyl)-2,2-dimethyl-1,3-oxazolidin-4-yl]-1-(*p*-methoxyphenyl)-2-azetidione (**4b**) exclusively in 51% yield. But the reaction of imine (**3c**) prepared from (-)-acetone-*d*-glyceraldehyde and *p*-anisidine with diketene gave (3*S*,4*S*)-3-acetyl-4-[(4*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]-1-(*p*-methoxyphenyl)-2-azetidione (**4c**) exclusively in 55% yield (Scheme 2).

Table 2. Yield and ratios of isomers of 3,4-*trans*-3-acetyl-2-azetidiones prepared

Product	Yield (%)	Ratio I : II
4a	55	1 : 7
4b	51	0 : 100
4c	55	100 : 0

**Scheme 3.**

Reduction of the acetyl group of compound **4b** with potassium triethylborohydride in ether at -78°C gave **5b**, a mixture of two epimeric alcohols (**I** and **II**) in 95% yield in the ratio of 14 : 5. Reduction of compound **4c** under the same conditions gave **5c**, also as a mixture of two isomers (**I** and **II**) in 90% yield in the ratio of 7 : 1. The isomers of both reduction products were isolated by silica gel column chromatography. The hydroxy group of the epimeric alcohol, **5c-II**, could be converted to the other isomer (**5c-I**) by Mitsunobu reaction in 91% yield (Scheme 3).

Experimental

IR spectra were recorded with Perkin-Elmer 735-B IR or Jasco J-0068 FT IR spectrophotometer. ^1H NMR spectra were obtained with Varian EM-360 (60 MHz), Bruker AC-80 (80 MHz) or Varian VXR-200S (200 MHz) NMR spectrometer with tetramethylsilane (TMS) as an internal standard. Chemical shifts are expressed as δ (ppm). Melting points were obtained with digital melting point measurement instrument made by Electrochemical Co. without correction. THF was distilled in the presence of sodium and benzophenone. Other solvents are 1st grade and distilled before use. All the chemicals were purchased from Aldrich Chemical Co. or Merck Co.

General Procedure for Imine-Diketene Condensation. Anhydrous magnesium sulfate (0.72 g, 6.0 mmol) and amine (3.0 mmol) were added to the solution of aldehyde (3.0 mmol) in methylene chloride (20 mL) at -30°C . The mixture was stirred for 1 h at the same temperature. After filtration of magnesium sulfate, imidazole (0.31 g, 4.5 mmol) and diketene (1.26 g, 15 mmol) were added slowly to the

solution and the mixture was stirred for 12 h at -30°C . After the reaction mixture was washed with 1.0 M hydrochloric acid, water, and 1.0 M aqueous sodium hydroxide solution, it was dried over anhydrous magnesium sulfate and evaporated to give residues. Column chromatography of the residue over silica gel with ethyl acetate-hexane gave desired products.

3-Acetyl-1-(2,2-dimethoxy)ethyl-4-phenyl-2-azetidione (2a). Yield 41%; ^1H NMR (CDCl_3) δ 2.36 (s, 3H, COCH_3), 3.00 (dd, $J=15.0, 6.0$ Hz, 1H, NCH), 3.32 (s, 3H, OCH_3), 3.38 (s, 3H, OCH_3), 3.69 (dd, $J=15.0, 5.0$ Hz, 1H, NCH), 4.11 (d, $J=2.6$ Hz, 1H, H-3), 4.52 (dd, $J=6.0, 5.0$ Hz, 1H, N-C-CH), 5.15 (d, $J=2.6$ Hz, 1H, H-4), 7.45 (s, 5H, Ph); IR (neat) 1765 (C=O, β -lactam), 1720 (C=O, ketone) cm^{-1} .

3-Acetyl-1-carbomethoxymethyl-4-phenyl-2-azetidione (2b). Yield 43%; ^1H NMR (CDCl_3) δ 1.31 (t, $J=7.0$ Hz, CH_3), 2.42 (s, 3H, COCH_3), 3.56 (d, $J=18.0$ Hz, 1H, NCH), 4.17 (d, $J=2.6$ Hz, 1H, H-3), 4.20 (d, $J=2.6$ Hz, 1H, NCH), 4.23 (q, $J=7.0$ Hz, 2H, CH_2), 5.33 (d, $J=2.6$ Hz, 1H, H-4), 7.50 (s, 5H, Ph); IR (neat) 1755 (C=O, β -lactam), 1755 (C=O, ester), 1720 (C=O, ketone) cm^{-1} .

3-Acetyl-1-(benzyl)(carbomethoxy)methyl-4-phenyl-2-azetidione (2c). Yield 49%; ^1H NMR (CDCl_3) δ 2.40 (s, 3H, COCH_3), 4.01 (d, $J=2.6$ Hz, 1H, H-3), 3.60 (s, 3H, OCH_3), 3.50-3.80 (dd, $J=15.0, 6.0$ Hz, 1H, NCH), 3.80 (d, $J=6.0$ Hz, 2H, CH_2), 5.13 (d, $J=2.6$ Hz, 1H, H-4), 7.00-7.79 (m, 10H, Ph); IR (neat) 1765 (C=O, β -lactam), 1745 (C=O, ester), 1720 (C=O, ketone) cm^{-1} .

3-Acetyl-4-benzyloxy-1-(*p*-methoxyphenyl)-2-azetidione (2d). Yield 60%; ^1H NMR (CDCl_3) δ 2.43 (s, 3H, COCH_3), 3.77 (s, 3H, OCH_3), 4.28 (d, $J=2.6$ Hz, 1H, H-3), 5.97 (d, $J=2.6$ Hz, 1H, H-4), 6.64-8.33 (m, 9H, Ar); IR (neat) 1760 (C=O, β -lactam), 1710 (C=O, ketone), 1690 (C=O, ketone) cm^{-1} .

3-Acetyl-4-methoxycarbonyl-1-(*p*-methoxyphenyl)-2-azetidione (2e). Yield 86%; ^1H NMR (CDCl_3) δ 2.40 (s, 3H, COCH_3), 3.78 (s, 3H, OCH_3), 3.80 (s, 3H, OCH_3), 4.38 (d, $J=2.5$ Hz, 1H, H-3), 4.95 (d, $J=2.5$ Hz, 1H, H-4), 6.80-7.31 (m, 4H, Ar); IR (KBr) 1760 (C=O, β -lactam), 1750 (C=O, ester), 1720 (C=O, ketone) cm^{-1} .

3-Acetyl-4-(2-benzyloxyethyl)-1-(*p*-methoxyphenyl)-2-azetidione (2f). Yield 10%; ^1H NMR (CDCl_3) δ 2.33 (s, 3H, COCH_3), 3.42-3.76 (m, 2H, CH_2), 3.69 (s, 3H, OCH_3), 4.07 (d, $J=2.6$ Hz, 1H, H-3), 4.21-4.50 (m, 3H, H-4, OCH_2), 5.38 (d, 2H, OCH_2), 6.67-7.44 (m, 9H, 2Ar); IR (neat) 1755 (C=O, β -lactam), 1720 (C=O, ketone) cm^{-1} .

(3S,4S)- and (3S,4R)-3-Acetyl-4-[(1R,2S)-1,2-epoxyethyl-2-phenyl]-1-(*p*-methoxyphenyl)-2-azetidione (4a-I, 4a-II). The two isomers were separated by silica gel column chromatography with ethyl acetate-hexane-methylene chloride (3 : 10 : 1). **4a-I:** Yield 7.0%; ^1H NMR (CDCl_3) δ 2.38 (s, 3H, COCH_3), 3.57 (m, 1H, OCH), 3.79 (s, 3H, OCH_3), 3.84 (d, $J=2.1$ Hz, 1H, H-3), 4.22 (d, $J=2.5$ Hz, 1H, CHPh), 4.25 (dd, $J=2.5, 2.1$ Hz, 1H, H-4), 6.84-7.56 (m, 9H, 2Ar); IR (KBr) 1745 (C=O, β -lactam), 1715 (C=O, ketone), 1215 (epoxy) cm^{-1} . **4a-II:** Yield 48%; ^1H NMR (CDCl_3) δ 2.40 (s, 3H, COCH_3), 3.48 (dd, $J=7.0, 2.6$ Hz, 1H, OCH), 3.76 (s, 3H, OCH_3), 3.84 (d, $J=2.6$ Hz, 1H, H-3), 4.25 (dd, $J=7.0, 2.6$ Hz, 1H, H-4), 4.60 (d, $J=2.6$ Hz, 1H, CHPh). 6.80-7.41 (m, 9H, 2Ar); IR (KBr) 1745 (C=O, β -lactam), 1715 (C=O, ketone), 1215 (epoxy) cm^{-1} .

(3R,4R)-3-Acetyl-4-[(4R)-3-(*t*-butoxycarbonyl)-2,2-dimethyl-1,3-oxazolidin-4-yl]-1-(*p*-methoxyphenyl)-2-azetidinone (4b). Yield 51%; $^1\text{H NMR}$ (CDCl_3) δ 1.45 (s, 9H, *t*-Bu), 1.52 (s, 3H, CH_3), 1.63 (s, 3H, CH_3), 2.40 (s, 3H, COCH_3), 3.51 (m, 1H, CH), 3.56-3.62 (m, 2H, CH_2), 3.62 (m, 1H, H-4), 3.85 (s, 3H, OCH_3), 4.15 (d, $J=2.1$ Hz, 1H, H-3), 6.86 (d, $J=8.8$ Hz, 2H, Ar), 7.32 (d, $J=8.8$ Hz, 2H, Ar); IR (neat) 1745 (C=O, β -lactam), 1715 (C=O, ketone) cm^{-1} .

(3S,4S)-3-Acetyl-4-[(4S)-2,2-dimethyl-1,3-dioxolan-4-yl]-1-(*p*-methoxyphenyl)-2-azetidinone (4c). Yield 55%; mp 65.5-66 $^\circ\text{C}$; $^1\text{H NMR}$ (CDCl_3) δ 1.29 (s, 3H, CH_3), 1.42 (s, 3H, CH_3), 2.38 (s, 3H, COCH_3), 3.77 (s, 3H, OCH_3), 3.86 (m, 2H, CH_2), 4.12 (m, 1H, CH), 4.15 (dd, $J=7.0$, 2.1 Hz, 1H, H-4), 4.23 (d, $J=2.1$ Hz, 1H, H-3), 6.86 (d, $J=8.8$ Hz, 2H, Ar), 7.32 (d, $J=8.8$ Hz, 2H, Ar); IR (KBr) 1760 (C=O, β -lactam), 1715 (C=O, ketone) cm^{-1} .

(3R,4R)-3-[(R)-1-Hydroxyethyl]- and (3R,4R)-3-[(S)-1-hydroxyethyl]-4-[(4R)-3-(*t*-butoxycarbonyl)-2,2-dimethyl-1,3-oxazolidin-4-yl]-1-(*p*-methoxyphenyl)-2-azetidinone (5b-I, 5b-II). After potassium triethylborohydride (THF, 1.0 M, 0.36 mL, 0.36 mmol) was added to the solution of **4b** (0.13 g, 0.30 mmol) in THF (10 mL) at -78 $^\circ\text{C}$, the mixture was stirred for 1 h at the same temperature. The reaction mixture was warmed up to room temperature and it was treated with 1.0 M hydrochloric acid (10 mL). The organic layer was separated, washed with water, dried over anhydrous magnesium sulfate, and evaporated to give a pale yellow liquid. Column chromatography of the liquid over silica gel with ethyl acetate-hexane (1:5) gave **5b-I** and **5b-II**. **5b-I**: Yield, 0.83 g (70%); $^1\text{H NMR}$ (CDCl_3) 1.20 (d, $J=5.2$ Hz, 3H, CH_3), 1.27 (br s, 1H, OH), 1.50 (s, 3H, CH_3), 1.62 (s, 3H, CH_3), 1.55 (s, 9H, *t*-Bu), 3.68 (m, 1H, CH), 3.70-4.20 (m, 1H, CHOH), 3.87 (s, 3H, OCH_3), 4.00 (m, 1H, H-3), 4.02 (m, 2H, CH_2), 4.78 (m, 1H, H-4), 6.91 (d, $J=6.9$ Hz, 2H, Ar), 7.72 (d, $J=6.9$ Hz, 2H, Ar); IR (neat) 3450 (OH), 1745 (C=O, β -lactam) cm^{-1} . **5b-II**: Yield, 0.29 g (25%); $^1\text{H NMR}$ (CDCl_3) δ 1.21 (d, $J=5.2$ Hz, 3H, CH_3), 1.28 (br s, 1H, OH), 1.49 (s, 3H, CH_3), 1.54 (s, 9H, *t*-Bu), 1.60 (s, 3H, CH_3), 3.70 (m, 1H, CH), 3.80-4.19 (m, 1H, CHOH), 3.88 (s, 3H, OCH_3), 4.01 (m, 1H, H-3), 4.02 (m, 2H, CH_2), 4.80 (m, 1H, H-4), 6.84 (d, $J=6.9$ Hz, 2H, Ar), 7.74 (d, $J=6.9$ Hz, 2H, Ar); IR (neat) 3450 (OH), 1745 (C=O, β -lactam) cm^{-1} .

(3S,4S)-3-[(R)-1-Hydroxyethyl]- and (3S,4S)-3-[(S)-1-hydroxyethyl]-4-[(4S)-2,2-dimethyl-1,3-dioxolan-4-yl]-1-(*p*-methoxyphenyl)-2-azetidinone (5c-I, 5c-II).

Compound **4c** (0.18 g, 0.56 mmol) was reduced with potassium triethylborohydride (THF, 1.0 M, 0.67 mL, 0.67 mmol) by the same procedure as described for **5b**. **5c-I** and **5c-II** were separated by silica gel column chromatography with ethyl acetate-hexane (1:2). **5c-I**: Yield, 0.20 g (70%); $^1\text{H NMR}$ (CDCl_3) δ 1.26 (d, $J=6.3$ Hz, 3H, CH_3), 1.28 (s, 3H, CH_3), 1.45 (s, 3H, CH_3), 2.15 (br s, 1H, OH), 2.94 (dd, $J=5.9$, 2.2 Hz, 1H, H-4), 3.70-3.90 (m, 1H, CHOH), 3.79 (s, 3H, OCH_3), 3.86 (dd, $J=8.0$, 5.9 Hz, 1H, CH_2), 4.16 (dd, $J=5.9$, 2.2 Hz, 1H, H-3), 4.00-4.50 (m, 1H, CH_2), 4.10-4.40 (m, 1H, CH), 6.82 (d, $J=8.0$ Hz, 2H, Ar), 7.45 (d, $J=8.0$ Hz, 2H, Ar); IR (neat) 3450 (OH), 1755 (C=O, β -lactam) cm^{-1} . **5c-II**: Yield, 0.03 g (20%); $^1\text{H NMR}$ (CDCl_3) δ 1.25 (s, 3H, CH_3), 1.26 (d, $J=6.3$ Hz, 3H, CH_3), 1.28 (s, 3H, CH_3), 2.14 (br s, 1H, OH), 3.25 (dd, $J=6.9$, 2.0 Hz, 1H, H-3), 3.68 (s, 3H, OCH_3), 3.70-3.95

(m, 1H, CHOH), 3.70-4.20 (m, 1H, CH_2), 3.74-3.80 (m, 1H, H-4), 3.86 (dd, $J=8.0$, 6.0 Hz, 1H, CH_2), 4.00-4.20 (m, 1H, CH), 6.84 (d, $J=8.0$ Hz, 2H, Ar), 7.28 (d, $J=8.0$ Hz, 2H, Ar); IR (neat) 3450 (OH), 1755 (C=O, β -lactam) cm^{-1} .

Conversion of 5c-II to 5c-I. To the solution of compound **5c-II** (0.03 g, 0.10 mmol) in THF (2.0 mL) in ice-water bath (0 $^\circ\text{C}$) was added triphenylphosphine (0.11 g, 0.42 mmol), formic acid (0.02 mL, 0.50 mmol) and diethyl azodicarboxylate (0.07 mL, 0.42 mmol) in sequence. After the mixture was stirred for 2 h in the ice-water bath, it was mixed with phosphate buffer solution (pH 5, 5.0 mL). The product was extracted with ethyl acetate (30 mL). The extract was dried over anhydrous magnesium sulfate and evaporated to give a residue. Column chromatography of the residue over silica gel with methylene chloride-ethyl acetate (9:1) gave **5c-I**. Yield, 0.027 g (91%).

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