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Practical synthetic approach to 4-acetoxy-2-azetidinone for the preparation of carbapenem and penem antibiotics

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Abstract A practical synthesis of 4-acetoxy-2-azetidinone useful for the preparation of carbapenem- and penem-type antibiotics is described. The synthesis has advantages such as avoiding the tedious and costly column chromatographic or recrystallized separation steps for diastereomers. The overall yield of the product is greatly improved and the process is also more economical for large-scale production. In addition, the mechanism for oxidative decarboxylation is also present.

Keywords Practical synthesis · 4-Acetoxy-2-azetidinone · Mechanism · Oxidative decarboxylation

Introduction

(3*R*,4*R*)-4-Acetoxy-3-[(1′*R*-*tert*-butyldimethylsilyloxy)ethyl]-2-azetidinone (**4-AA**) (Fig. 1) and its derivatives are well known to be highly versatile intermediates for the synthesis of thienamycin and carbapenem derivatives and other novel antibiotics that might defeat bacterial resistance [1–4]. With possible industrial developments, a tremendous amount of effort has been devoted to the search for efficient preparations of this chiral azetidinone (**4-AA**) and a variety of methods for

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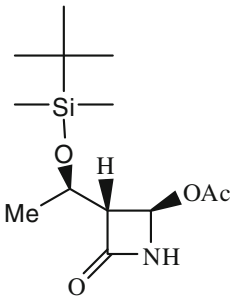


Fig. 1 Structure of (3*R*,4*R*)-4-acetoxy-3-[(1′*R*-*tert*-butyldimethylsilyloxy)ethyl]-2-azetidinone

synthesizing this compound have been performed such as [2 + 2] cycloadditions of ketenes to Schiff bases, using optically active natural sources and so on [5–8].

Though some of these have been industrialized and have succeeded in mass production of the compound, they involve various common problems in that the cost of starting materials is very high, large quantities of solvents and reagents for extensive column chromatographic separation of diastereomers are required, and the overall yield is still low, in spite of using advanced industrial techniques.

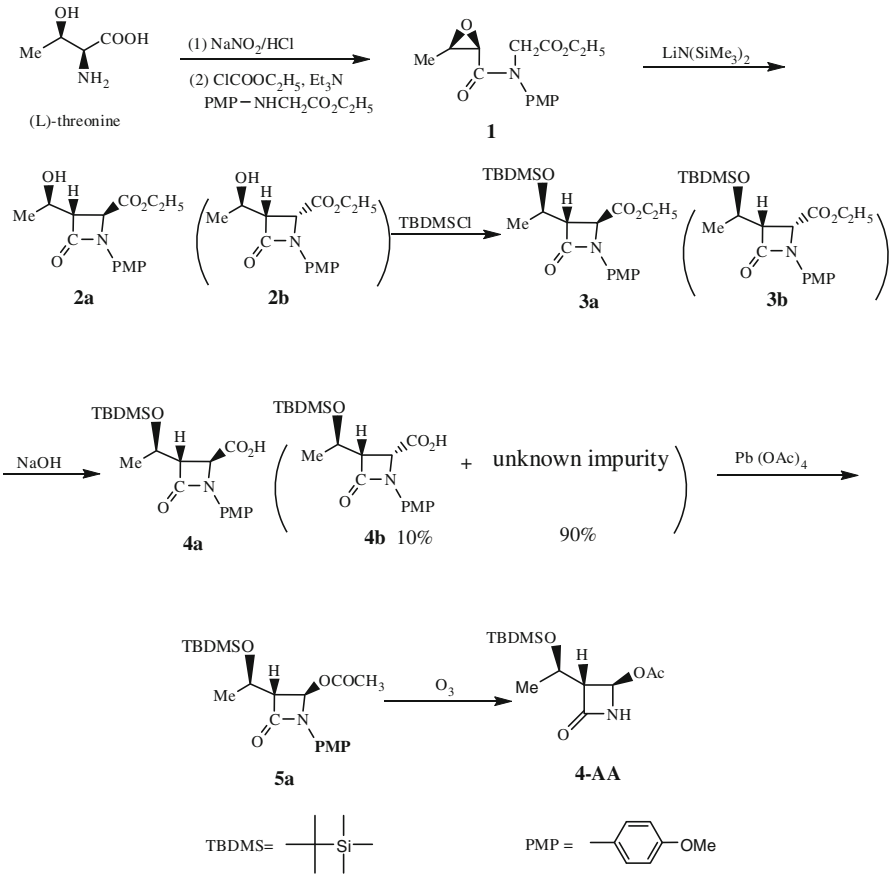
Results and discussion

Our considerable current research interest in the industrial synthesis of (3*R*,4*R*)-4-acetoxy-3-[(1′*R*-*tert*-butyldimethylsilyloxy)ethyl]-2-azetidinone (**4-AA**) is supported by Lee's work, etc. [9, 10] using L-threonine as the optically active source (Scheme 1).

During our research, we found some industrial synthetic problems in the above process: (**1**) In the C3–C4 β-lactam ring formation, a significant amount of stereoisomer (**2b**) is produced, which needs to be separated by the tedious and costly column chromatographic or recrystallized separation step. (**2**) The stereoisomer **2b** had to be discarded because of an unknown impurity, which was produced when **3b** was hydrolyzed. (**3**) The overall yield of the product is still low.

In order to overcome the industrial synthetic problems mentioned above, we developed a practical, scalable, and economical procedure for synthesis of **4-AA**. Our synthetic strategy is shown in Scheme 2.

As shown in Scheme 2, ethyl 2-((2*R*,3*R*)-*N*-(4-methoxyphenyl)-3-methyloxirane-2-carboxamido)acetate (**1**) was derived in high yield from L-threonine according to a known method [11]. This epoxy amide (**1**) was then cyclized with 1 N lithium hexamethyldisilazide (LiHMDS) in THF at −20 °C to give ethyl 3-((*R*)-1-hydroxyethyl)-1-(4-methoxyphenyl)-4-oxoazetidine-2-carboxylate (**2a** and **2b**) in 89% yield, in which the diastereomeric ratio is about 6:1. Hydrolyzation of (**2a** and **2b**) with 1 N NaOH in ethanol to afford 3-((*R*)-1-hydroxyethyl)-1-(4-methoxyphenyl)-4-oxoazetidine-2-carboxylic acid (**3a**′ and **3b**′) in quantitative yield, which was protected with *tert*-butyldimethylsilyl chloride in DMF at 35 °C for 10 h to give 3-((*R*)-1-(*tert*-butyldimethylsilyloxy)ethyl)-1-(4-methoxyphenyl)-4-oxoazetidine-2-carboxylic acid (**4a** and **4b**) in 95% yield. Acetoxylation of (**4a** and **4b**) was accomplished with

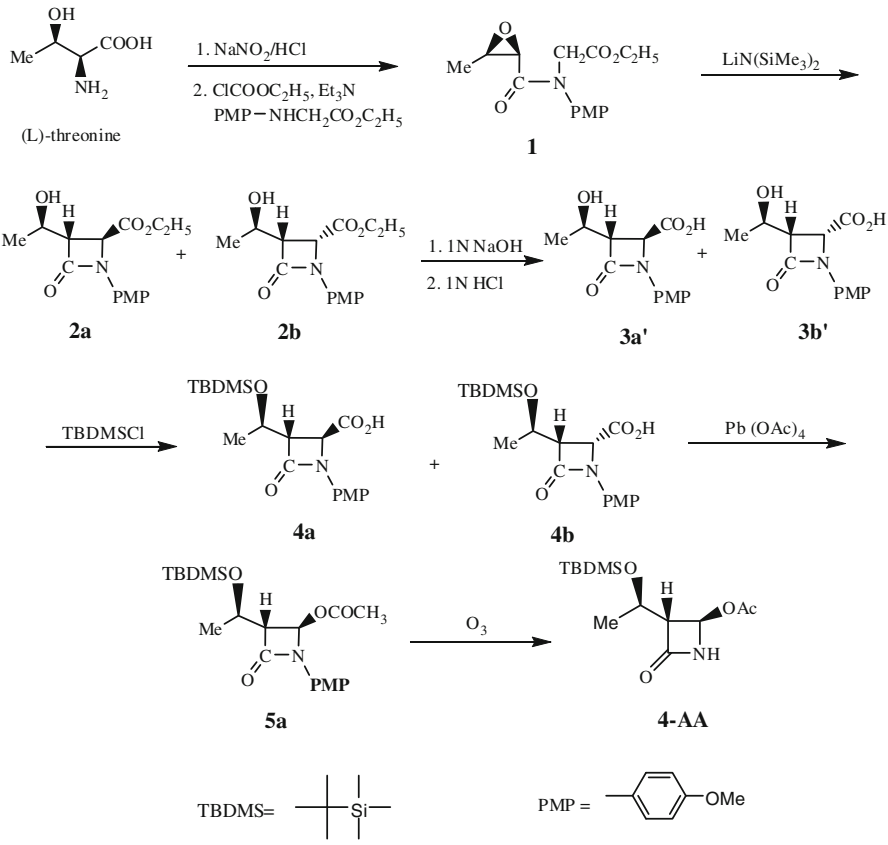


Scheme 1 Synthesis of (3*R*,4*R*)-4-acetoxy-3-[(1′*R*-*tert*-butyldimethylsilyloxy)ethyl]-2-azetidin-one (**4-AA**)

Pb_3O_4 in acetic acid to stereoselectively afford (2*R*,3*R*)-3-((*R*)-1-(*tert*-butyldimethylsilyloxy)ethyl)-1-(4-methoxyphenyl)-4-oxoazetidin-2-yl acetate (**5a**) in 96% yield [12]. At last, the product (**5a**) reacted with ozone in methanol at −20 °C followed by reductive work-up to give **4-AA** in 95% yield [13, 14]. It is noteworthy that separation of stereoisomers (**2a** and **2b**) is not needed, diastereomers **2b**, **3b**′, and **4b** all can be converted into target compound without being discarded, and the overall yield of the product is greatly improved in this process.

Acetoxylation reaction is a key step for stereoselective synthesis of **4-AA**. In our study, the acetoxylation reaction of **4a**, **4b**, and the mixture of **4a** and **4b** were examined with Pd_3O_4 in acetic acid at 50 °C. The results are shown in Table 1.

When **4a**, **4b** or the mixture of **4a** and **4b** was treated with Pd_3O_4 in acetic acid at 50 °C respectively, an unexpected process took place and all gave only one product of *trans*-isomer **5a**, without containing any of the *cis*-isomer **5b**. Therefore, the possible mechanism we deduced is shown in Scheme 3 [15–19].



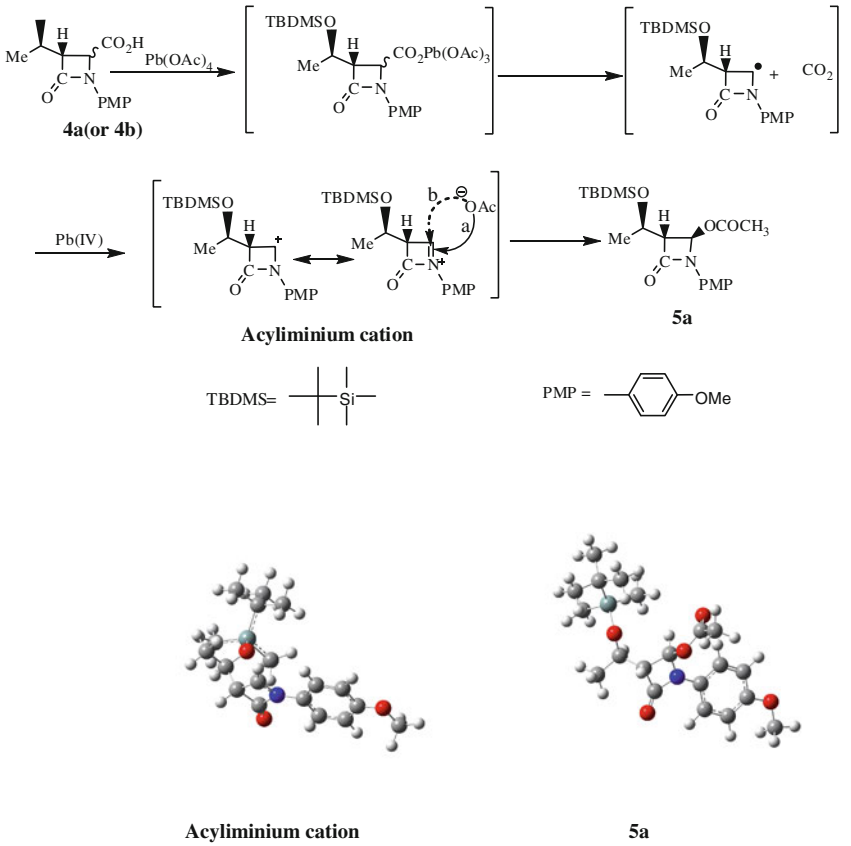
Scheme 2 Practical synthesis of (3*R*,4*R*)-4-acetoxy-3-[(1′*R*-*tert*-butyldimethylsilyloxy)ethyl]-2-azetidinone (**4-AA**)

Table 1 Results of acetoxylation reaction of **4a**, **4b**, and the mixture of **4a** and **4b**

Entry	Reactant	Product	Yields[a] (%)
1	**4a**	**5a**	97
2	**4b**	**5a**	96.5
3	**4a** and **4b**(**4a:4b** = 6:1)	**5a**	96

[a] Isolated yields

The reaction of **4a** (or **4b**) with the oxidizing agent was expected to go through the lead(IV)carboxylate intermediate, which would decompose to an alkyl radical. The acyliminium cation was formed after the free radical has undergone a one-electron oxidation, which can smoothly react with nucleophiles because it was stabilized by adjacent nitrogen. In order to confirm the formation of a four-membered acyliminium ion, the oxidation of **4a** in the mixture of AcOH and water was carried out and we obtained the expected products **5a** and (*R*)-3-(1-(*tert*-butyldimethylsilyl-oxy)ethyl)-1-(4-methoxyphenyl)azetidine-2,4-dione (Fig. 2).



Scheme 3 The possible mechanism of acetoxylation reaction

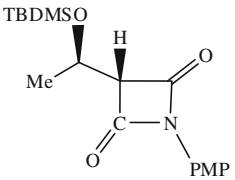


Fig. 2 Structure of (*R*)-3-(1-(*tert*-butyldimethylsilyl-oxy)ethyl)-1-(4-methoxyphenyl)azetidine-2,4-dione

According to the key transition state, the acyliminium cation is preferably attacked by acetate anion from the less sterically hindered side (from the side opposite the (*tert*-butyldimethylsilyloxy)ethyl) to produce **5a** in high stereoselectivity.

Conclusions

In conclusion, we have developed a practical, scalable, and economical procedure for the synthesis of **4AA**, which has advantages such as avoiding the tedious

and costly column chromatographic or recrystallized separation steps for diastereomers, diastereomers (**4a**, **4b**) all can be stereoselectively converted into product (**5a**), the overall yield of the product (**4-AA**) is greatly improved, and the process is more economical for large-scale production. Further work is currently going on to extend our synthetic protocol.

Experimental section

General commercially available chemicals were all reagent-grade. Melting points (mp) were determined on a Buchi 535 capillary melting apparatus. Optical rotations were obtained using a JASCO DIP-1000 polarimeter at room temperature using the sodium D line. The [1]H-NMR and [13]C-NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on a Mercury Plus Varian 400-MHz FT-NMR spectrometer, using TMS as the internal standard. ESI/MS were acquired on a Thermo Scientific LCQ spectrometer. IR spectra were determined on a Nicolet NEXUS-470 FT-IR spectrometer as KBr pellets. Analytical TLC was performed on a Merck precoated TLC (silica gel 60 F254) plate.

Preparation of ethyl 2-((2*R*,3*R*)-*N*-(4-methoxyphenyl)-3-methyloxirane-2-carboxamido)acetate 1

This compound was obtained as yellow oil according to the known procedures [20–23]. Yield: 80%, $[\alpha]_D^{20} = +20.7°$ (c = 1.0, $CHCl_3$). [1]H-NMR: 1.25 (*t*, *J* = 4.0, 3 H), 1.41 (*d*, *J* = 5.6, 3 H), 3.04 (*m*, 1 H), 3.29 (*d*, *J* = 4.8, 1 H), 3.82 (*s*, 3 H), 4.15 (*m*, 3 H), 4.62 (*d*, *J* = 16.8, 1 H), 6.93 (*m*, 2 H), 7.27 (*m*, 2 H). [13]C-NMR: 168.37, 166.84, 159.24, 133.12, 128.89, 114.74, 61.23, 55.42, 54.12, 53.59, 51.34, 14.13, 13.14. *m/z* (ESI): 316.3 $[M + Na]^+$. IR (KBr): *v* 3,485, 2,980, 2,937, 2,839, 1,747, 1,681, 1,606, 1,583, 1,512, 1,433, 1,396, 1,373, 1,300, 1,249, 1,199, 1,107, 1,026, 970, 839 cm^{-1}.

Preparation of ethyl 3-((*R*)-1-hydroxyethyl)-1-(4-methoxyphenyl)-4-oxoazetidine-2-carboxylate 2a and 2b

To a solution of **1** (58.6 g, 0.2 mol) in THF under nitrogen, 1 N lithium hexamethyldisilazide $LiN(SiMe_3)_2$ solution (240 mL) was added dropwise at −40 °C. After 3 h of stirring, the solution was allowed to slowly warm to −20 °C. The reaction was monitored by TLC. After this was accomplished, the reaction was quenched with excess AcOH, and diluted with EtOAc, washed with sat. $NaHCO_3$ and brine, dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by recrystallization from toluene/*n*-hexane to give 52.0 g of **2a** and **2b** as light yellow powder. Analytical samples were further purified by column chromatography.

2a: m.p. 93.5–95.0°. $[\alpha]_D^{20} = -115.2°$ (c = 1.0, $CHCl_3$). [1]H-NMR: 1.19 (*m*, 6 H), 3.38 (*t*, *J* = 2.8, 1 H), 3.71(*s*, 3 H), 4.08 (*m*, 1 H), 4.18 (*m*, 2 H), 4.63 (*d*, *J* = 2.8, 1 H), 5.20 (*d*, *J* = 4.8, 1 H), 6.93 (*d*, *J* = 8.8, 2 H), 7.21 (*d*, *J* = 8.8, 2 H). [13]C-NMR: 169.94, 163.69, 155.36, 130.75, 117.25, 114.20, 62.57, 62.11, 61.17, 55.23, 52.00, 21.83, 14.04. m/z (ESI): 294.2 $[M + H]^+$. IR (KBr): *v* 3,425, 2,964, 2,956, 2,935, 2,908, 1,724, 1,587, 1,518, 1,447, 1,411, 1,375, 1,338, 1,300, 1,255, 1,209, 1,165, 1,132, 1,118, 1,030, 877, 833, 804 cm^{-1}.

2b: m.p. 164.0–165.5°. $[\alpha]_D^{20} = +71.0°$ (c = 1.0, CH_3OH). [1]H-NMR: 1.21 (*m*, 6 H), 3.57 (*m*, 1 H), 3.70 (*s*, 3 H), 3.93 (*m*, 1 H), 4.15 (*m*, 2 H), 4.73 (*d*, *J* = 5.6, 1 H), 4.91 (*d*, *J* = 6.0 Hz, 1 H), 6.91 (*d*, *J* = 8.8, 2 H), 7.15 (*d*, *J* = 8.8, 2 H). [13]C-NMR: 169.68, 163.47, 155.41, 130.65, 117.16, 114.28, 62.52, 60.99, 59.97, 55.22, 53.81, 22.65, 13.98. *m/z* (ESI): 294.1 $[M + H]^+$. IR (KBr): *v* 3,508, 2,974, 2,937, 1,739, 1,716, 1,581, 1,514, 1,444, 1,404, 1,363, 1,301, 1,251, 1,220, 1,168, 1,114, 1,091, 1,066, 1,030, 898, 833, 815 cm^{-1}.

Preparation of 3-((*R*)-1-hydroxyethyl)-1-(4-methoxyphenyl)-4-oxoazetidine-2-carboxylic acid 3a′ and 3b′

To a stirred solution of **2a** and **2b** (45.8 g, 0.156 mol) in EtOH (100 mL) was added gradually 1 N-NaOH (187 mL) at room temperature, then the solution was allowed to warm to 50 °C. After stirring for 30 min, the reaction mixture was concentrated in vacuo and diluted with H_2O, washed with ethyl acetate. The aqueous phase was acidified to pH = 2 with 1 N-HCl, filtered and washed with *n*-hexane to give 39.7 g of **3a′** and **3b′** as a white solid which was employed for the next reaction. Analytical samples were further purified by recrystallization.

3a′: m.p. 179.5–181.5°. $[\alpha]_D^{20} = -105.8°$ (c = 1.0, DMF). [1]H-NMR: 1.18 (*m*, 3 H), 3.31 (*q*, *J* = 2.4, 1 H), 3.71 (*s*, 3 H), 4.05 (*m*, 1 H), 4.50 (*m*, 1 H), 5.14 (*s*, 1 H), 6.91 (*m*, 2 H), 7.15 (*m*, 2 H). [13]C-NMR: 171.54, 163.81, 155.25, 130.90, 117.16, 114.21, 62.65, 61.94, 55.25, 52.08, 21.83. *m/z* (ESI): 266.2 $[M + H]^+$. IR (KBr): *v* 3,400, 2,974, 2,843, 2,729, 2,615, 2,536, 1,743, 1,712, 1,583, 1,514, 1,442, 1,375, 1,352, 1,300, 1,247, 1,178, 1,128, 1,082, 1,024, 968, 927, 871, 835, 812 cm^{-1}.

3b′: m.p. 194.5–196.5°. $[\alpha]_D^{20} = +85.5°$ (c = 1.0, DMF). [1]H-NMR: 1.24 (*m*, 3 H), 3.52 (*q*, *J* = 5.6, 1 H), 3.70 (*s*, 3 H), 3.97 (*q*, *J* = 6.0, 1 H), 4.60 (*d*, *J* = 6.0, 1 H), 6.91 (*m*, 2 H), 7.15 (*m*, 2 H). [13]C-NMR: 170.08, 163.56, 155.31, 130.83, 117.14, 114.27, 62.57, 59.76, 55.26, 54.09, 22.58. *m/z* (ESI): 266.1 $[M + H]^+$. IR (KBr): *v* 3,412, 2,937, 2,630, 2,538, 1,739, 1,701, 1,581, 1,516, 1,458, 1,444, 1,400, 1,301, 1,246, 1,174, 1,136, 1,114, 1,084, 1,030, 891, 831 cm^{-1}.

Preparation of 3-((*R*)-1-(*tert*-butyldimethylsilyloxy)ethyl)-1-(4-methoxyphenyl)-4-oxoazetidine-2-carboxylic acid 4a and 4b

A mixture of **3a′** and **3b′** (31.8 g, 0.12 mol), *tert*-butylmethylsilyl chloride (47.2 g, 0.288 mol), triethylamine (29.0 g 0.288 mol) in DMF (300 mL) was allowed to

stand at 35 °C for 10 h. The reaction mixture was diluted with ethyl acetate, washed with 1 N HCl, sat. $NaHCO_3$ and brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by recrystallization from $MeOH/H_2O$ to give 44.8 g of **4a** and **4b** as a white solid. Analytical samples were further purified by column chromatography.

4a: m.p. 189.0–191.0°. $[\alpha]_D^{20} = -121.1°$ (c = 1.0, $CHCl_3$). [1]H-NMR 0.00 (*s*, 3 H), 0.07 (*s*, 3 H), 0.74 (*s*, 9 H), 1.27 (*d*, $J = 6.0$ Hz, 3 H), 3.40 (*s*, 1 H), 3.77 (*s*, 3 H), 4.36 (*q*, $J = 2.4$, 1 H), 4.63 (*d*, $J = 2.0$, 1 H), 6.83 (*d*, $J = 9.2$, 2 H), 7.25 (*d*, $J = 9.2$, 2 H), 11.53 (*s*, 1 H). [13]C-NMR: 175.13, 164.20, 156.17, 130.47, 117.87, 114.20, 64.16, 62.82, 55.43, 51.99, 25.58, 22.39, 17.78, −4.20, −5.02. *m/z* (ESI): 380.2 $[M + H]^+$. IR (KBr): *v* 2,964, 2,931, 2,858, 2,615, 1,751, 1,716, 1,589, 1,512, 1,444, 1,394, 1,361, 1,334, 1,294, 1,246, 1,211, 1,153, 1,139, 1,116, 1,070, 1,039, 976, 829 cm^{-1}.

4b: m.p. 119.1–120.5° Yellow crystals $[\alpha]_D^{20} = +32.6°$ (c = 1.0, $CHCl_3$). [1]H NMR: 0.04 (*m*, 6 H), 0.82 (*s*, 9 H), 1.38 (*d*, $J = 6.4$, 3 H), 3.60 (*m*, 1 H), 3.75 (*s*, 3 H), 4.47 (*q*, $J = 5.6$, 1 H), 4.59 (*d*, $J = 5.6$, 1 H), 6.79 (*d*, $J = 9.2$, 2 H), 7.21 (*d*, $J = 9.2$, 2 H). [13]C-NMR: 173.68, 163.73, 156.20, 130.52, 118.117, 114.21, 65.15, 61.40, 55.47, 54.14, 29.77, 25.79, 21.90, 18.06, −4.17, −4.74. *m/z* (ESI): 380.1 $[M + H]^+$. IR (KBr): *v* 3,315, 2,953, 2,931, 2,854, 1,735, 1,708, 1,587, 1,512, 1,440, 1,398, 1,363, 1,301, 1,244, 1,193, 1,153, 1,105, 1,037, 989, 964, 920, 825 cm^{-1}.

Preparation of (2*R*,3*R*)-3-((*R*)-1-(*tert*-butyldimethylsilyloxy)ethyl)-1-(4-methoxyphenyl-4-oxoazetidin-2-yl acetate 5a

Pb_3O_4 (67.5 g. 0.098 mol) was added to the mixture of glacial acetic acid (60 mL) and acetic anhydride (60 mL) by portionwise at 50 °C. After 2 h, the suspension of **4a** and **4b** (30.0 g, 0.079 mol) in glacial acetic acid (60 mL) was added dropwise to the above reaction mixture. The reaction was monitored by TLC, and after being accomplished, the reaction was quenched by the addition of 1 mL of ethyleneglycol and was concentrated in vacuo. The residue was solidified with a large excess of H_2O to give 29.8 g of **5a**. m.p. 76.5–77.5°. $[\alpha]_D^{20} = -72.9°$ (c = 1.0, $CHCl_3$). [1]H-NMR: 0.00 (*s*, 3 H), 0.06 (*s*, 3 H), 0.74 (*s*, 9 H), 1.31 (*d*, $J = 6.4$, 3 H), 2.12 (s, 3 H), 3.19(*d*, $J = 2.4$, 1 H), 3.77 (*s*, 3 H), 4.28 (*m*, 1 H), 6.61 (*s*, 1 H), 6.84 (*m*, 2 H), 7.30 (*q*, $J = 2.0$, 2 H). [13]C-NMR: 169.81, 163.54, 156.25, 129.47, 118.38, 114.20, 76.62, 65.35, 64.13, 55.41, 25.61, 22.34, 21.17, 17.79, −4.18, −5.05. *m/z* (ESI) 394 $(M + H)^+$. IR (KBr): *v* 2,949, 2,931, 2,858, 1,753, 1,512, 1,402, 1,375, 1,247, 1,226, 1,180, 1,151, 1,068, 1,028, 979, 898, 831 cm^{-1}.

Preparation of (3*R*,4*R*)-4-acetoxy-3-[(1′R-*tert*-butyldimethylsilyloxy) ethyl]-2-azetidinone 4-AA

A solution of **5a** (19.6 g, 0.05 mol) in methanol (100 mL) was treated with ozone at −20 °C until the starting material completely disappeared. Then, 10% aqueous

sodium thiosulfate solution (337.5 mL) was added at 8 °C. After 1 h, thiourea (11.4 g) was added portionwise. After stirring 3 h at 40 °C, the organic solvent was removed partly in vacuo, and the residue was solidified with a large excess of H_2O and purified by recrystallization from with *n*-hexane to give **4-AA** as white crystals. m.p. 106–107°. Literature [23]. m.p. 104°. $[\alpha]_D^{20} = +51.5°$ (c = 1.0, $CHCl_3$). [1]H-NMR: 0.01 (*d*, $J = 6.0$, 6 H), 0.80 (*s*, 9 H), 1.19 (*d*, $J = 6.4$, 3 H), 2.04 (s, 3 H), 3.11(*t*, $J = 2.4$, 1 H), 4.14 (*m*, 1 H), 5.76 (*s*, 1 H), 7.04 (*s*, 1 H). [13]C-NMR: 170.7, 166.4, 74.9, 64.8, 63.7, 25.6, 20.8, 20.5, 17.8, −4.4, −5.1. *m/z* (ESI) 310 $(M + Na)^+$. IR (KBr): *v* 3,203, 2,958, 2,929, 2,895, 2,856, 1,782, 1,745, 1,471, 1,377, 1,361, 1,342, 1,300, 1,253, 1,234, 1,163, 1,134, 1,107, 1,078, 1,039, 983, 945, 896, 873 cm^{-1}.

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