Efficient Asymmetric Synthesis of (2*R*,3*R*)-3-{(1*R*)-1-[*tert*-Butyl(dimethyl)-siloxy]ethyl}-4-oxoazetidin-2-yl Acetate

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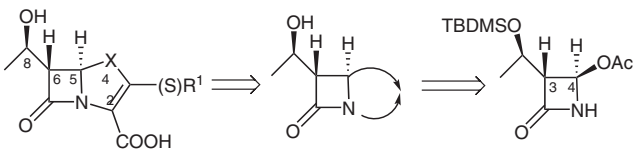
Abstract: (2*R*,3*R*)-3-{(1*R*)-1-[*tert*-Butyl(dimethyl)siloxy]ethyl}-4-oxoazetidin-2-yl acetate was efficiently prepared from L-ascorbic acid. The key steps were the a highly diastereoselective [2 + 2] cycloaddition of diketene with an (*S*)-glyceraldehyde-derived aldimine to give the ketone, stereoselective titanium tetrachloride mediated asymmetric reduction to give the corrsponding *S*-configured alcohol, and Mitsunobu inversion of the latter to give the desired *R*-configured alcohol.

Key words: carbapenems, penem, heterocycles, cycloadditions, cyclizations

Over the last three decades, carbapenems (**1**) and penems (**2**) have been a focus of synthetic attention from academia and industry because of their unique structures and their potent broad-spectrum antibacterial activities and excellent stabilities to β-lactamases.[1] Considerable efforts have been directed at the development of simple and elegant approaches for the synthesis of such β-lactam antibiotics. The most common strategy for synthesizing carbapenems **1** and penems **2** involves the initial construction of an appropriately substituted monocyclic β-lactam **3** with the correct stereochemistry at the C3 and C4 positions of the β-lactam ring, and subsequent establishment of the five-membered heterocyclic framework by chemical manipulations at the N1 and C4 positions (Scheme 1).[2] (2*R*,3*R*)-3-{(1*R*)-1-[*tert*-Butyl(dimethyl)siloxy]ethyl}-4-oxoazetidin-2-yl acetate (**3**), which has three contiguous stereogenic centers corresponding the C5, C6, and C8 carbons of **1** and **2** is recognized as a versatile chiral building block for the asymmetric syntheses of **1** and **2**, because its acetoxy group can be easily replaced by a variety of nucleophiles.[1a]



Scheme 1 Retrosynthetic analysis of **1** and **2**

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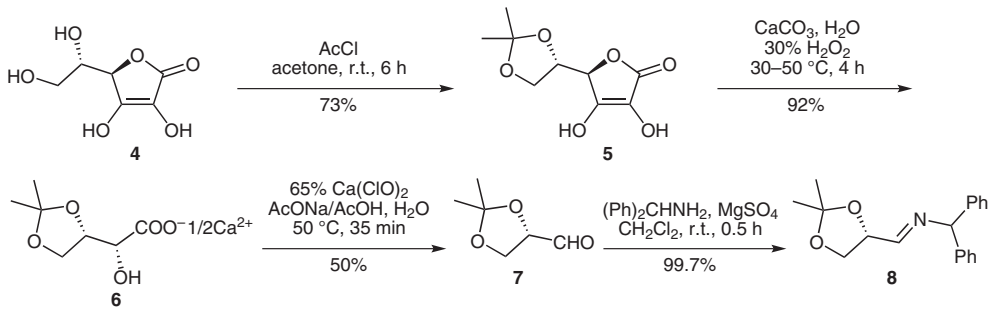
Many literature reports describe asymmetric syntheses of **3** by various chiral techniques.[1a] One of the most convenient approaches that has come to our attention is the asymmetric [2+2] cycloaddition of diketene with various chiral imines derived from various chiral pools, including ethyl (*S*)-lactate,[3] L-menthyl glyoxylate,[4] and D-mannitol.[5] However, the use of these processes for large-scale preparation of the intermediate is limited by problems of unsatisfactory chemical yields or low diastereoselectivities. An asymmetric synthesis of an N-substituted β-lactam with 3-(1-hydroxyethyl) and a 4-acetoxy substituents has been reported in which commercially available L-ascorbic acid (**4**) is used as a chiral pool.[6a] However, when the procedure was repeated with a *p*-methoxyphenyl group as the N-protecting group, a low stereoselectivity was observed in the reduction by potassium borohydride in methanol–tetrahydrofuran of the side-chain acetyl group on the β-lactam ring. Here, we describe a high-yielding and highly stereoselective synthesis of **3** in which the pivotal steps are a [2 + 2] cycloaddition of diketene with the chiral imine **8** derived from **4** and bulky (diphenylmethyl)amine, a highly diastereoselective titanium tetrachloride mediated reduction of the ketone **9a** with borane–pyridine complex as the reducing agent, and a Mitsunobu inversion of alcohol **10b** to give **10a**.

Our asymmetric synthesis began with the preparation of the *R*-configured imine **8** (Scheme 2). The (*S*)-glyceraldehyde acetonide **7** was prepared from commercially available and inexpensive L-ascorbic acid (**4**) by a modified literature procedure.[6b] Condensation of **7** with (diphenylmethyl)amine in the presence of magnesium sulfate in dichloromethane at room temperature for 30 minutes gave the imine **8** in almost quantitative yield.

Having prepared **8**, we turned our attention to the synthesis of the (3*S*,4*S*)-β-lactam **9a** by a strategy involving an asymmetric [2+2] cycloaddition.[4,6a] When the reaction was carried out in the presence of one equivalent of imidazole and five equivalents of diketene in anhydrous tetrahydrofuran at –15 °C for twenty-four hours, a mixture of (3*S*,4*S*)-β-lactam **9a** and (3*R*,4*R*)-β-lactam **9b** was obtained in 78% yield. Analysis by ^{1}H NMR spectroscopy showed the presence of a 4.7:1 ratio of **9a** and **9b**. The single diastereomer **9a** was obtained in 54.3% yield by recrystallization from ethyl acetate–petroleum ether (1:1). To improve the diastereoselectivity of this reaction, we screened a variety of reaction conditions (solvent, cata-



Scheme 2 Scheme for the synthesis of *R*-configured imine **8**

lyst, and temperature); the results are summarized in Table 1. It was evident that, of the solvents tested, tetrahydrofuran was best for this reaction (entries 1–4). A significant increase in diastereoselectivity occurred when 4-methyl-1*H*-imidazole was used as a catalyst instead of imidazole (entries 4 and 6). It is noteworthy that none of the desired product was obtained when 2-methyl-1*H*-imidazole was used as catalyst (entry 5). A decrease in the reaction temperature improved the diastereoselectivity of the cycloaddition reaction (entries 6–9), and a remarkable improvement in diastereoselectivity was observed when the reaction was performed at –78 °C (entry 9). The stable configuration of **9a** was assigned on the basis of two-dimensional (2D) NOESY NMR spectroscopy techniques, which showed a strong NOE interaction between the C4-H atom (δ = 4.16 ppm, dd, *J* = 2.4, 7.0 Hz) and the C7-H atom (δ = 4.01 ppm, m), confirming their *cis* orientation, and a weak NOE correlation between the C3-H atom (δ = 3.88 ppm, d, *J* = 2.4 Hz) and the C4-H atom, implying a *trans* orientation (Figure 1).

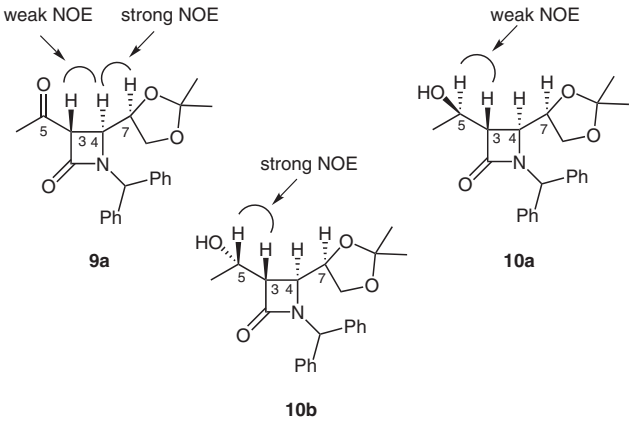
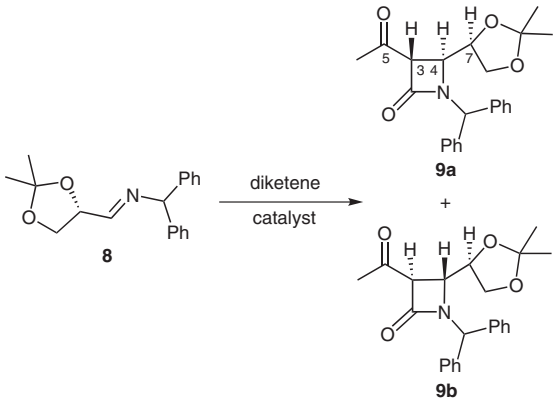


Figure 1 NOE correlations for **9a**, **10a**, and **10b**

In attempts to prepare azetidinone **10a**, three sets of reducing systems (potassium borohydride in methanol–tetrahydrofuran, potassium tris(*sec*-butyl)borohydride and potassium iodide in tetrahydrofuran, and potassium triethyborohydride in tetrahydrofuran)[6a,7] were examined for the stereoselective reduction of **9a**. A diastereoisomeric mixture of **10a** and its epimer **10b** was obtained with low diastereoselectivity (Table 2, entries 1–3). Several

Table 1 Optimization of Conditions for the [2+2] Cycloaddition of Imine **8** with Diketene

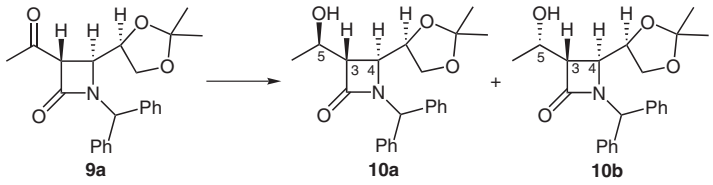


Entry	Conditions[a]	Yield (%)[b]	Ratio **9a/9b**[c]
1	imidazole, toluene, –15 °C, 24 h	71	2.5:1
2	imidazole, CH_2Cl_2, –15 °C, 24 h	82	3.3:1
3	imidazole, MeCN, –15 °C, 24 h	84	4.1:1
4	imidazole, THF, –15 °C, 24 h	78	4.7:1
5	2-methyl-1*H*-imidazole, THF, –15 °C, 24 h	–	–
6	4-methyl-1*H*-imidazole, THF, –15 °C, 24 h	78	8.0:1
7	4-methyl-1*H*-imidazole, THF, –35 °C, 72 h	82	12.8:1
8	4-methyl-1*H*-imidazole,, THF, –55 °C, 120 h	87	18.1:1
9	4-methyl-1*H*-imidazole,, THF, –78 °C, 168 h	85	23:1

[a] General reaction conditions: the reaction was carried out in the presence of catalyst (1 equiv) and diketene (5 equiv) in anhyd solvent.
[b] Yield of crude products.
[c] Determined by ^{1}H NMR spectroscopy by integration of the signals for the acetyl protons, which appeared as two singlets in the isomeric mixture.

highly diastereoselective titanium tetrachloride mediated reductions of α-chiral ketones using borane–pyridine as a reducing reagent have been reported,[8] and, interestingly, the reduction of **9a** also proceeded well upon treatment with titanium tetrachloride in tetrahydrofuran at –78 °C for 10 minutes followed by diastereoselective reduction with borane–pyridine at –78 °C for 15 minutes; **10b** and

Table 2 Optimization of Diastereoselective Reduction of β-Lactam **9a**



Entry	Reaction system	Temp (°C)	Time (min)	Yield (%)	Ratio **10a/10b**[a]
1	KBH_4, THF–MeOH	–20	40	99	1.5:1
2	$K(s\text{-}Bu)_3BH$, KI, THF	0	15	99	2.5:1
3	KEt_3BH, THF	–78	25	99	5.9:1
4	$TiCl_4$, $Py{\cdot}BH_3$, THF	–78	25	98	1:21
5	$TiCl_4$,$(i\text{-}Pr)_2NH{\cdot}BH_3$, THF	–78	25	99	1:13.6
6	$TiCl_4$, $BH_3{\cdot}THF$	–78	25	98	1:6.3
7	$TiCl_4$, $KBHEt_3$, THF	–78	25	99	1:4.8

[a] Determined by ^1H NMR of the mixture of **10a** and **10b**.

its epimer **10a** were obtained in 98% yield with excellent diastereoselectivity (dr 21:1; Table 2, entry 4). A 75% yield of pure **10b** was obtained by recrystallization from ethyl acetate. Note that a low diastereoselectivity was observed when borane–pyridine was replaced with other reducing agents under the same conditions (Table 2, entries 5–7).

The stable configuration of **10b** was characterized by means of a 2D NOESY experiment, which showed a strong NOE correlation between the C3-H proton (δ = 2.83 ppm, dd, J = 2.4, 6.4 Hz) and the C5-H proton (δ = 4.03 ppm, m), suggesting that the hydroxy group at the C5 position in **10b** adopts an *S*-configuration (Figure 1). This absolute configuration was confirmed by means of X-ray diffraction (Figure 2).[9] Furthermore, the single-crystal configuration of **10b** established that the framework of **9a** had been correctly assigned.

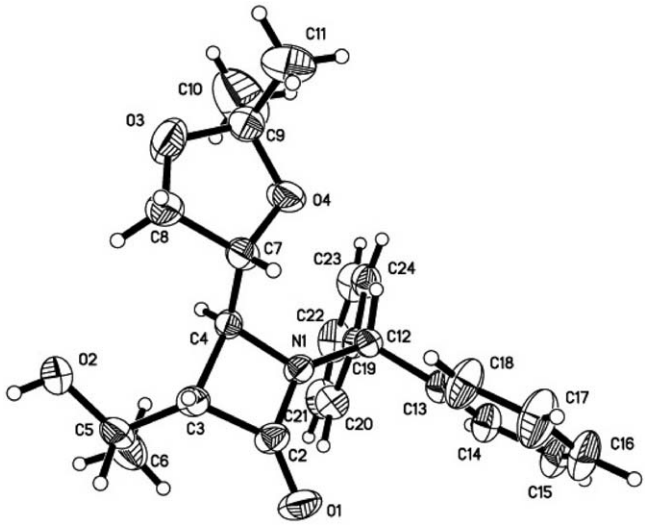


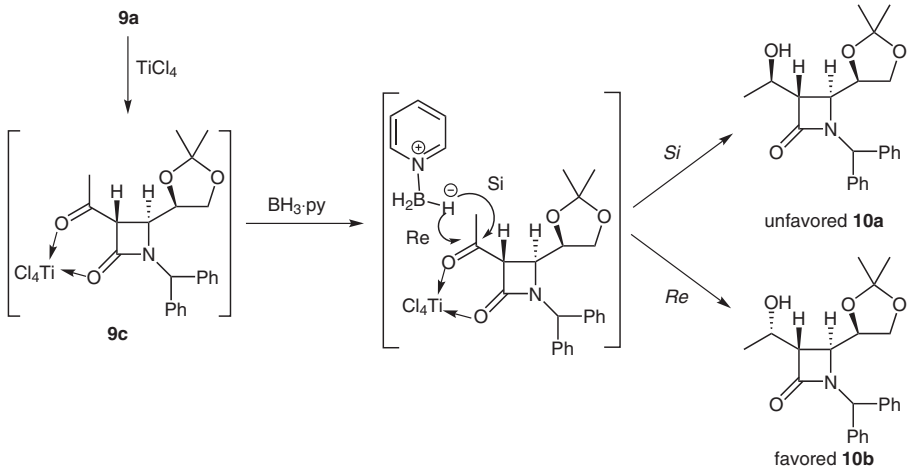
Figure 2 X-ray crystal structure of **10b**

The diastereoselectivity of this reduction can be explained in terms of the formation of a chelate of **9a** with titanium tetrachloride to give intermediate **9c**, and attack by pyridine–borane at the less hindered *re*-face of the carbonyl group of **9c** to give diastereomer **10b** predominantly (Scheme 3).[8]

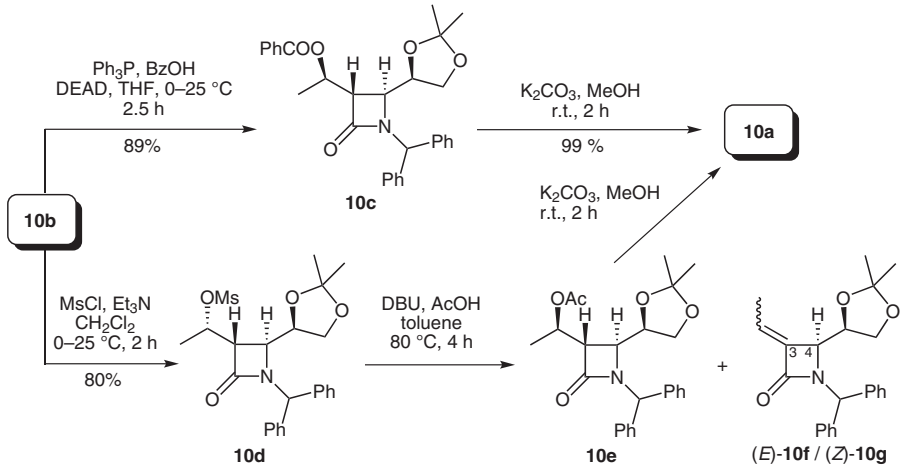
Inversion of the azetidinone **10b** to the desired diastereoisomer **10a** was performed by Mitsunobu reaction with diethyl azodicarboxylate, triphenylphosphine, and benzoic acid at room temperature for 2.5 hours, and subsequent alkaline hydrolysis of the resulting *R*-configured formate **10c** to provide the desired azetidinone **10a** in 88% overall yield (Scheme 4).[10] 2D NOESY experiments showed that the hydroxy group at the C5 position in **10a** adopts an *R*-configuration, resulting in a weak NOE correlation between the C3-H proton (δ = 2.79 ppm, dd, J = 2.4, 6.8 Hz) and the C5-H proton (δ = 3.72 ppm, m) (Figure 1).

Another attempt at inversion of **10b** into **10a** was made by mesylation of **10b** with mesyl chloride and triethylamine, followed by S_N2 neucleophilic replacement with 1,8-diazabicyclo[5.4.0]undec-7-ene and acetic acid in dichloromethane. This gave a separable mixture of **10e** and two byproducts **10f** and **10g** in a ratio of 60:35:5.[11] **10e**, isolated by chromatography, was smoothly hydrolyzed to **10a** by treatment with potassium carbonate in methanol.

Acetonide **10a** was deprotected in the presence of 1 M aqueous sulfuric acid at room temperature for 24 hours to give the vicinal diol **11** in 80% yield (Scheme 5). Oxidation of the diol **11** by treatment with silica gel supported sodium metaperiodate (Shing's protocol)[12] gave aldehyde **12** in 92% yield. Potassium permanganate mediated selective oxidation of **12** under alkaline condition in aqueous tetrahydrofuran at room temperature gave the acid **13** in 86% yield.[13] Acid **13** was converted into the corresponding acetate **14** in 72% yield by treatment with lead tetraacetate in anhydrous *N*,*N*-dimethylformamide.[13]



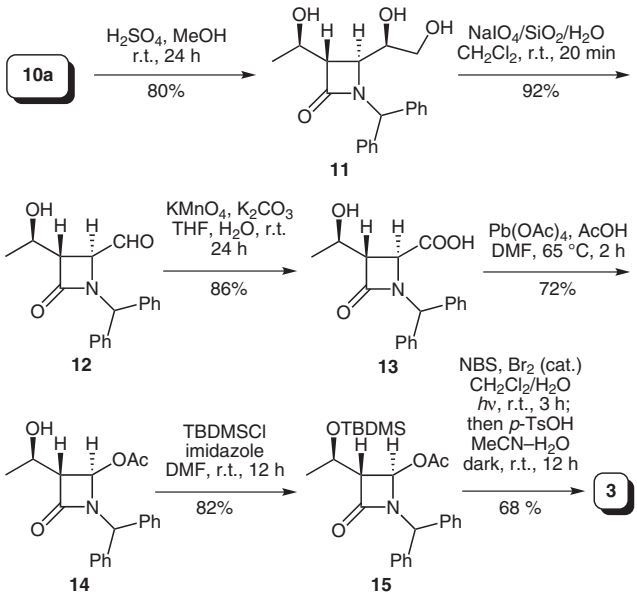
Scheme 3 Proposed mechanism for the titanium-mediated reduction of azetidinone **9a**



Scheme 4 Synthetic scheme for the preparation of **10a**

Subsequently, alcohol **14** was protected by treatment with chloro(*tert*-butyl)(dimethyl)silane in the presence of imidazole in anhydrous *N*,*N*-dimethylformamide at room temperature for 12 h to give ester **15** in 82% yield. The *N*-benzhydryl group of ester **15** was cleaved by treatment with a stoichiometric amount of *N*-bromosuccinimide and a catalytic amount of bromine under UV irradiation in dichloromethane/water. Hydrolysis with 4-toluenesulfonic acid in aqueous acetone provided the β-lactam **3** in 68% overall yield.[14]

In conclusion, we have developed a convenient asymmetric synthesis of the azetidinone **3** starting from L-ascorbic acid. Further efforts toward the asymmetric syntheses of carbapenems (**1**) and penems (**2**) by utilizing this pivotal intermediate are underway, and will be reported in due course.



Scheme 5 Scheme for the synthesis of azetidinone **3**

Anhydrous (anhyd) THF was distilled from sodium/benzophenone before use. Anhyd CH_2Cl_2 and toluene were distilled from CaH_2. Anhyd DMF was distilled from CaH_2 under reduced pressure. Other reagents were obtained from commercial sources and were used as received. All melting points were measured on a WRS-1B digital melting-point apparatus and are uncorrected. ^1H (400 MHz) and ^{13}C

(100 MHz) NMR spectra were recorded on a Bruker Avance 400 spectrometer in $CDCl_3$, CD_3OD, or D_2O using TMS and $CDCl_3$ (^{13}C, δ = 77.0) or CD_3OD (^{13}C, δ = 49.0) as internal standards. Coupling constants (*J* values) are given in hertz. Mass spectra were recorded on a Waters Quattro Micromass instrument using electrospray ionization techniques. IR spectra were recorded on a Jasco FT/IR-4200 spectrometer. Optical rotations were measured on a Jasco P1020 digital polarimeter.

5,6-*O*-Isopropylidene-L-ascorbic Acid (5)

AcCl (88 mL, 1.24 mol) was added dropwise to a stirred soln of L-ascorbic acid (**4**, 200 g, 1.136 mol) in acetone (500 mL) and the mixture was stirred at 40 °C for 2 h. The mixture was then cooled to 0 °C and filtered. The residue was washed with cold acetone (200 mL) and dried to give a white solid; yield: 179 g (73%); mp 208.3–208.4 °C (Lit.[15] 218–219 °C).

IR (KBr): 3241, 3080, 1754, 1663 cm^{-1}.

^1H NMR (400 MHz, CD_3OD): δ = 1.34 (s, 3 H, CH_3), 1.35 (s, 3 H, CH_3), 4.05 (dd, *J* = 6.6, 8.8 Hz, 1 H, OCH_2), 4.19 (dd, *J* = 6.6, 8.0 Hz, 1 H, OCH_2), 4.34 (m, 1 H, OC*H*CH_2), 4.68 (d, *J* = 4.4, 1 H, C*H*COH), 4.90 (br, 2 H, OH).

^{13}C NMR (100 MHz, CD_3OD): δ = 153.32, 118.61, 109.73, 75.49, 69.23, 65.06, 62.16, 24.85, 24.30.

Calcium Bis[(2*R*)-[(4*S*)-2,2-dimethyl-1,3-dioxolan-4-yl](hydroxy)acetate] (6)

30% aq H_2O_2 (315 mL) was added to a vigorously stirred suspension of lactone **5** (170 g, 0.787 mol), $CaCO_3$ (157.4 g, 1.574 mol), and antifoaming agent (2 drops) in H_2O (1 L) while the temperature of the mixture was kept at 30–35 °C. The mixture was stirred at r.t. for 3 h and then at 50 °C for 1 h. Excess H_2O_2 was destroyed by addition of activated MnO_2 (1.7 g, 19.5 mmol), and stirring was continued at 50 °C for 30 min. The mixture was filtered and the filtrate was concentrated to ~240 mL in vacuo. Acetone (800 mL) was added, and the mixture was cooled to below 10 °C and kept overnight. The suspension was filtered and the residue was dried to give a white solid; yield: 141 g (92%); mp 258.3–260.1 °C (dec.) (Lit.[16] 257–261 °C); $[\alpha]_D^{25}$ +23.8 (*c* 0.9, H_2O) [Lit.[16] +23.6 (*c* 0.974, H_2O)].

IR (KBr): 3391, 1611 cm^{-1}.

^1H NMR (400 MHz, D_2O): δ = 1.29 (s, 3 H, CH_3), 1.37 (s, 3 H, CH_3), 3.88 (dd, *J* = 6.8, 8.4 Hz, 1 H, OCH_2), 3.96 (d, *J* = 4.9 Hz, 1 H, C*H*OH), 4.07 (dd, *J* = 6.8, 8.4 Hz 1 H, OCH_2), 4.33–4.38 (m, 1 H, OCH).

^{13}C NMR (100 MHz, D_2O): δ = 109.85, 77.06, 72.22, 65.52, 25.20, 24.12.

MS (ESI): *m*/*z* = 175.2 $[M + Ca]^+$.

(4*S*)-2,2-Dimethyl-1,3-dioxolane-4-carbaldehyde (7)

NaOAc (171 g, 2.09 mol) and AcOH (165 mL, 2.90 mol) were added to a stirred suspension of calcium salt **6** (97.2 g, 0.50 mol) in H_2O (1.25 L) at r.t., and the mixture was heated to 50 °C. 65% solid $Ca(OCl)_2$ (65.7 g) was added portionwise over 35 minute. The heater was then removed and the pH of the mixture was adjusted to 8 with Na_2CO_3. The suspension was filtered and the residue was washed with CH_2Cl_2 (3 × 200 mL). The organic phase was separated and the aqueous layer was extracted with CH_2Cl_2 (6 × 50 mL). The combined organic phases were collected, dried ($MgSO_4$), filtered, and concentrated in vacuo to give a colorless oil; yield: 32.4 g (50%); $[\alpha]_D^{25}$ –63.7 (*c* 1.0, $CHCl_3$) (Lit.[15] –63.5).

IR ($CHCl_3$): 2988, 1735 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.32 (s, 3 H, CH_3), 1.39 (s, 3 H, CH_3), 4.00–4.10 (m, 2 H, OCH_2), 4.29–4.32 (m, 1 H, OC*H*CH_2), 9.61 (d, *J* = 2.0 Hz, 1 H, CHO).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 201.54, 111.07, 79.73, 65.36, 26.09, 24.99.

MS (EI): *m*/*z* = 130 $[M]^+$.

[(4*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]methylene}-1,1-diphenylmethanamine (8)

A mixture of aldehyde **7** (26.2 g, 0.2 mol), Ph_2CHNH_2 (36.6 g, 0.2 mol), CH_2Cl_2 (130 mL), and anhyd $MgSO_4$ (26 g) was stirred at r.t. for 30 min. The suspension was filtered and the filtrate was concentrated under reduced pressure to give a colorless oil; yield: 59 g (99.7%). This was used directly in the next step without any further purification.

IR ($CHCl_3$): 3061, 3026, 2986, 2935, 2870, 1671, 1493, 1453, 1371 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.30 (s, 3 H, CH_3), 1.33 (s, 3 H, CH_3), 3.91 (dd, *J* = 6.0, 8.4 Hz, 1 H, OCH_2), 4.12 (dd, *J* = 6.8, 8.4 Hz, 1 H, OCH_2), 4.62 (td, *J* = 4.8, 6.0, 6.8 Hz, 1 H, C*H*CH_2O), 5.34 (s, 1 H, C*H*Ph), 7.10–7.23 (m, 10 H, ArH), 7.72 (d, *J* = 4.8 Hz, 1 H, CH=N).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.56, 142.99, 142.89, 128.49, 127.58, 127.46, 127.14, 127.11, 110.19, 67.38, 26.49, 25.41.

MS (EI): *m*/*z* = 295.4 $[M]^+$.

Anal. Calcd for $C_{19}H_{21}NO_2$: C, 77.26; H, 7.17; N, 4.74. Found: C, 77.18; H, 7.15; N, 4.75.

(3*S*,4*S*)-3-Acetyl-4-[(4*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)azetidin-2-one (9a)

Diketene (30 mL) was added dropwise to a soln of amine **8** (22 g, 74.5 mmol) and 4-methyl-1*H*-imidazole (6.35 g, 77.4 mmol) in anhyd THF (150 mL) at –78 °C, and the mixture was stirred at –78 °C for 7 d. The solvent was evaporated and CH_2Cl_2 (300 mL) was added to the residue. The resulting mixture was washed successively with 1 M H_2SO_4 (2 × 40 mL), 2 M NaOH (20 mL), and H_2O. The organic layer was dried ($MgSO_4$) and concentrated in vacuo to give a 23:1 mixture of **9a** and **9b**; yield: 24 g (85%). The residue was crystallized from EtOAc–PE (1:1) to give the single isomer **9a** as a white solid; yield: 19.2 g (68%); mp 101.1–101.9 °C; $[\alpha]_D^{23}$ +50.8 (*c* 1, $CHCl_3$).

IR (KBr): 3025, 2979, 2967, 2935, 2885, 1761, 1709 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.23 (s, 3 H, CH_3), 1.33 (s, 3 H, CH_3), 2.29 (s, 3 H, CH_3), 3.61 (dd, *J* = 6.0, 9.0 Hz, 1 H, OCH_2), 3.81 (dd, *J* = 6.8, 9.0 Hz, 1 H, OCH_2), 3.88 (d, *J* = 2.4 Hz 1 H, C_3H), 4.01 (m, 1 H, OCH), 4.16 (dd, *J* = 2.4, 7.0 Hz, 1 H, C_4H), 5.90 (s, 1 H, C*H*Ph), 7.24–7.36 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.35, 162.13, 138.80, 138.74, 128.60, 128.52, 128.43, 127.99, 127.77, 127.74, 110.33, 76.47, 65.47, 63.57, 62.67, 55.24, 29.78, 26.55, 24.78.

MS (EI): *m*/*z* = 379 $[M]^+$.

Anal. Calcd for $C_{23}H_{25}NO_4$: C, 72.80; H, 6.64; N, 3.69. Found: C, 72.71; H, 6.66; N, 3.70.

(3*S*,4*S*)-4-[(4*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)-3-[(1*R*)-1-hydroxyethyl]azetidin-2-one (10a) and -3-[(1*S*)-1-hydroxyethyl]azetidin-2-one (10b)

$TiCl_4$ (0.88 mL) was added to a soln of ketone **9a** (2.68 g, 7.05 mmol) in anhyd CH_2Cl_2 at –78 °C, and the mixture was stirred for 10 min. BH_3·py (0.66 g, 7.1 mmol) was slowly added and the mixture was stirred at –78 °C for 15 min. 1 N HCl (5 mL) was added, and the temperature of the mixture rose to 25 °C. The organic phase was washed successively with 1 M H_2SO_4 (2 × 5 mL) and H_2O (2 × 5 mL), dried ($MgSO_4$), filtered, and concentrated in vacuo to give 1:21 epimeric mixture of **10a** and **10b**; yield: 2.64 g (98%). This

was recrystallized (EtOAc) to give pure **10b** as a white solid; yield: 2.02 g (75%); mp 142.5–142.7 °C.

IR (KBr): 3463, 1717 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.22 (s, 3 H, CH_3), 1.26 (d, *J* = 6.4 Hz, 3 H, C*H*$_3$CH), 1.32 (s, 3 H, CH_3), 2.83 (dd, *J* = 2.4, 6.4 Hz, 1 H, C_3H), 3.57 (dd, *J* = 2.4, 7.6 Hz, 1 H, C_4H), 3.62 (dd, *J* = 6.0, 8.8 Hz, 1 H, OCH_2), 3.82 (dd, *J* = 6.8, 8.8 Hz, 1 H, OCH_2), 4.03 (m, 2 H, C*H*OH, OC*H*), 5.93 (s, 1 H, C*H*Ph), 7.27–7.35 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.33, 24.82, 26.61, 56.42, 57.34, 62.21, 65.55, 66.14, 77.13, 110.18, 127.67, 127.71, 128.20, 128.45, 128.55, 128.72, 138.66, 139.34, 167.82.

MS (EI): *m*/*z* = 381 $[M]^+$.

Anal. Calcd for $C_{23}H_{27}NO_4$: C, 72.47; H, 7.13; N, 3.67. Found: C, 72.57; H, 7.14; N, 3.66.

Crystallographic data for compound **10b** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC 654872; copies can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: +44(1223)336033 or email: deposit@ccdc.cam.ac.uk].

(1*R*)-1-[(2*S*,3*S*)-2-[(4*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)-4-oxoazetidin-3-yl]ethyl Benzoate (10c)

A soln of DEAD (1.43 mL) in anhyd THF (5 mL) was added dropwise to a soln of **10b** (2.0 g, 5.24 mmol), Ph_3P (5.34 g, 20.4 mmol), and BzOH (0.64 g, 5.24 mmol) in anhyd THF (55 mL) at r.t., and the mixture was stirred at the r.t. for 2.5 h. The mixture was then poured into phosphate buffer (pH 5; 100 mL) and extracted with EtOAc (3 × 20 mL). The solvent was evaporated and the residue was washed with hot H_2O to give a white solid; yield: 2.27 g (89%); mp 91.6–93.2 °C.

IR (KBr): 1733, 1716 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.21 (s, 3 H, CH_3), 1.31 (s, 3 H, CH_3), 1.47 (d, *J* = 6 Hz 3 H, C*H*$_3$CH), 3.06 (dd, *J* = 2.4, 7.6 Hz, 1 H, C_3H), 3.57 (dd, *J* = 6.8, 8.4 Hz, 1 H, OCH_2), 3.80 (dd, *J* = 2.4, 7.6 Hz, 1 H,C_4H), 3.85 (dd, *J* = 6.4, 8.4 Hz, 1 H, OCH_2), 4.09 (m, 1 H, C*H*CH_2O), 5.43–5.50 (m, 1 H, OC*H*CH_3), 5.94 (s, 1 H, C*H*Ph), 7.29–7.58 (m, 15 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 165.59, 165.24, 139.36, 138.79, 133.16, 129.61, 128.78, 128.58, 128.50, 128.48, 128.08, 127.74, 127.66, 110.27, 68.86, 65.90, 62.35, 57.84, 56.29, 26.56, 24.89, 18.88.

MS (ESI): *m*/*z* = 486.5 $[M + H]^+$.

Anal. Calcd for $C_{30}H_{31}NO_5$: C, 74.21; H, 6.43; N, 2.88. Found: C, 74.31; H, 6.42; N, 2.87.

(3*S*,4*S*)-4-[(4*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)-3-[(1*R*)-1-hydroxyethyl]azetidin-2-one (10a)

Benzoate **10c** (2.27 g, 4.67 mmol) was dissolved in MeOH (30 mL). K_2CO_3 (2 g, 14.4 mmol) was added and the mixture was stirred at r.t. for 30 min, then concentrated in vacuo. The residue was mixed with CH_2Cl_2 (30 mL) and H_2O (30 mL), and the organic phase was dried ($MgSO_4$) and concentrated under reduced pressure to give a white solid; yield: 1.76 g (99%); mp 102.5–103.5 °C; $[\alpha]_D^{22}$ +38.8 (*c* 1, MeOH).

IR (KBr): 3427, 1720 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.23 (s, 3 H, CH_3), 1.27 (d, *J* = 2.4 Hz, 3 H, CH_3), 1.35 (s, 3 H, CH_3), 2.79 (dd, *J* = 2.4, 6.8 Hz, 1 H, C_3H), 3.69–3.75 (m, 2 H, HOC*H*, OCH_2), 3.78 (dd, *J* = 6.4, 8.8 Hz, 1 H, OCH_2), 4.03 (dd, *J* = 6.8, 12.6 Hz, 1 H, C_4H), 4.08–4.12 (m, 1 H, CHOH), 5.95 (s, 1 H, C*H*Ph), 7.27–7.36 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.39, 24.29, 26.06, 56.53, 57.84, 61.69, 64.94, 65.20, 109.63, 127.14, 127.74, 127.92, 128.02, 128.22, 138.32, 138.98, 166.54.

MS (ESI): *m*/*z* = 382 $[M + H]^+$.

Anal. Calcd for $C_{23}H_{27}NO_4$: C, 72.47; H, 7.13; N, 3.67. Found: C, 72.54; H, 7.14; N, 3.66.

(1*R*)-1-[(2*S*,3*S*)-2-[(4*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)-4-oxoazetidin-3-yl]ethyl Methanesulfonate (10d)

MsCl (0.30 g, 2.62 mmol) was added dropwise to a soln of **10b** (0.5 g, 1.31 mmol) and Et_3N (0.40 g, 3.93 mmol) in CH_2Cl_2 (10 mL) cooled to 0 °C in an ice bath. When the addition was complete, the mixture was stirred at r.t. for 2 h. The reaction was then quenched with 1 M aq HCl (6 mL). The organic phase was separated, washed with 5% aq $NaHCO_3$ (10 mL), dried ($MgSO_4$), and concentrated under reduced pressure to give a crude product that was purified by chromatography [silica gel, EtOAc–PE (1:5)] to give a colorless oil; yield: 0.48 g (80%).

IR ($CHCl_3$): 1644 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.24 (s, 3 H, CH_3), 1.37 (s, 3 H, CH_3), 1.52 (d, *J* = 6.4 Hz, 3 H, C*H*$_3$CH), 2.89 (s, 3 H, CH_3SO), 3.10 (dd, *J* = 2.4, 3.6 Hz, 1 H, C_3H), 3.65 (dd, *J* = 6.0, 8.4 Hz, 1 H, OCH_2), 3.73 (dd, *J* = 2.4, 7.2 Hz, 1 H, C_4H), 3.86 (dd, *J* = 6.4, 8.4 Hz, 1 H, OCH_2), 4.09 (m, 1 H, C*H*CH_2), 4.99–5.05 (m, 1 H, CHOS, 5.94 (s, 1 H, C*H*Ph), 7.28–7.38 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 162.69, 137.73, 137.40, 126.87, 126.57, 126.35, 126.10, 125.53, 107.62, 75.62, 73.38, 63.70, 60.51, 54.08, 52.97, 36.13, 24.69, 23.37, 16.76.

Anal. Calcd for $C_{24}H_{29}NO_6S$: C, 62.73; H, 6.36; N, 3.05. Found: C, 62.80; H, 6.34; N, 3.04.

(1*R*)-1-[(2*R*,3*R*)-2-[(4*R*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)-4-oxoazetidin-3-yl]ethyl Acetate (10e) and (3*E*,4*S*)- and (3*Z*,4*S*)-4-[(4*R*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)-3-ethylideneazetidin-2-one (10f and 10g)

AcOH (0.11 g, 1.76 mmol) was added to a soln of DBU (0.13 g, 0.88 mmol) in toluene (5 mL) and the mixture was stirred at r.t. for 30 min. Mesylate **10d** (0.20 g, 0.44 mmol) was then added and the mixture was heated at 80 °C with stirring for 4 h. The mixture was then cooled to r.t., diluted with toluene (20 mL), and washed successively with 1 M HCl (5 mL), 5% aq $NaHCO_3$ (10 mL), and brine (10 mL). The organic phase was dried ($MgSO_4$) and concentrated under reduced pressure to give a crude mixture that was separated by flash chromatography [EtOAc–PE (1:8)] to give **10e** as a colorless oil; yield 0.09 g (52%). The elimination byproducts **10f** and **10g** were also obtained as colorless oils; yield: **10f**: 0.053 g (29%); **10g**: 0.007 g (4%).

10e

^1H NMR (400 MHz, $CDCl_3$): δ = 1.24 (s, 3 H, CH_3), 1.34 (d, *J* = 6.4 Hz, 3 H, C*H*$_3$CH), 1.36 (s, 3 H, CH_3), 1.99 (s, 3 H, CH_3CO), 2.92 (dd, *J* = 2.4, 7.2 Hz, 1 H, C_3H), 3.63 (dd, *J* = 6.4, 8.8 Hz, 1 H, OCH_2), 3.68 (dd, *J* = 2.4, 7.2 Hz, 1 H, C_4H), 3.84 (dd, *J* = 6.4, 8.4 Hz, 1 H, OCH_2), 4.05 (m, 1 H, C*H*CH_2O), 5.17 (m, 1 H, CH_3C*H*), 5.93 (s, 1 H, C*H*Ph), 7.28–7.38 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 18.75, 21.15, 24.86, 26.61, 56.07, 57.48, 62.30, 65.70, 68.22, 77.23, 110.28, 127.71, 128.18, 128.45, 128.58, 128.70, 138.79, 139.35, 165.46, 169.94.

Anal. Calcd for $C_{25}H_{29}NO_5$: C, 70.90; H, 6.90; N, 3.31. Found: C, 70.84; H, 7.00; N, 3.32.

10f

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.24 (s, 3 H, CH_3), 1.35 (s, 3 H, CH_3), 2.04 (d, *J* = 7.2 Hz, 3 H, C*H*$_3$CH), 3.66–3.71 (m, 1 H, OCH_2), 3.86–3.91 (m, 1 H, OCH_2), 4.05 (d, *J* = 3.6 Hz, 1 H, C_4H), 4.03–4.09 (m, 1 H, C*H*CH_2O), 5.63 (q, *J* = 7.2 Hz, 1 H, CH_3C*H*), 6.04 (s, 1 H, C*H*Ph), 7.27–7.37 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.74, 24.95, 26.58, 61.15, 62.13, 65.81, 109.72, 126.68, 127.55, 127.66, 128.19, 128.43, 128.48, 128.87, 136.86, 138.92, 139.73, 163.86.

Anal. Calcd for $C_{23}H_{25}NO_3$: C, 76.01; H, 6.93; N, 3.85. Found: C, 76.04; H, 6.92; N, 3.86.

10g

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.21 (s, 3 H, CH_3), 1.36 (s, 3 H, CH_3), 1.79 (d, *J* = 7.6 Hz, 3 H, C*H*$_3$CH), 3.69 (m, 1 H, OCH_2), 3.77 (m, 1 H, OCH_2), 4.01–4.06 (m, 1 H, C*H*CH_2O), 4.34 (d, *J* = 5.6 Hz, 1 H, C_4H), 6.04 (s, 1 H, C*H*Ph), 6.29 (dq, *J* = 1.2, 7.6 Hz, 1 H, CH_3C*H*), 7.27–7.37 (m, 10 H, ArH).

Anal. Calcd for $C_{23}H_{25}NO_3$: C, 76.01; H, 6.93; N, 3.85. Found: C, 76.03; H, 6.94; N, 3.86.

(3*R*,4*R*)-4-[(4*R*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-1-(diphenylmethyl)-3-[(1*S*)-1-hydroxyethyl]azetidin-2-one (10a) (Alternative Route)

K_2CO_3 (0.06 g, 0.46 mmol) was added to a soln of **10e** (0.09 g, 0.23 mmol) in MeOH (5 mL) at r.t., and the mixture was stirred for 30 min then concentrated in vacuo. The residue was mixed with CH_2Cl_2 (5 mL) and H_2O (5 mL). The organic phase was dried ($MgSO_4$) and concentrated under reduced pressure to give a white solid; yield: 0.086 g (98%).

(3*R*,4*R*)-4-[(1*R*)-1,2-Dihydroxyethyl]-1-(diphenylmethyl)-3-[(1*S*)-1-hydroxyethyl]azetidin-2-one (11)

1 M H_2SO_4 (78 mL) was added to a soln of **10a** (10 g, 26.2 mmol) in MeOH (100 mL), and the mixture was stirred at r.t. for 24 h. The pH of the mixture was the adjusted to 7 with sat. aq $NaHCO_3$. The MeOH was evaporated off, and the aq layer was extracted with EtOAc. The combined organic phase was dried ($MgSO_4$) and concentrated in vacuo to give a white solid; yield 7.1 g (80%). This was used directly in the next step without further purification.

IR (KBr): 3394, 1731 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.21 (d, *J* = 6.4 Hz, 3 H, CH_3), 2.80 (dd, *J* = 2.4, 8.0 Hz 1 H, C_3H), 3.45–3.55 (m, 2 H, O*C*H_2), 3.61–3.65 (m, 2 H, CH_3C*H*OH, C_4H), 3.85–3.92 (m, 1 H, C*H*$OHCH_2$), 5.94 (s, 1 H, C*H*Ph), 7.23–7.34 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 58.16, 59.08, 62.83, 64.25, 66.28, 74.48, 127.73, 127.76, 128.05, 128.54, 128.66, 128.75, 138.89, 139.45, 167.41.

MS (ESI): *m*/*z* = 363.4 $[M + Na]^+$.

Anal. Calcd for $C_{20}H_{23}NO_4$: C, 70.36; H, 6.79; N, 4.10. Found: C, 70.33; H, 6.80; N, 4.09.

(2*R*,3*R*)-1-(Diphenylmethyl)-3-[(1*R*)-1-hydroxyethyl]-4-oxoazetidine-2-carbaldehyde (12)

$NaIO_4$ supported on silica gel (21.5 g, $m_{NaIO4}/m_{SiO2}/m_{H2O}$ 1:4:2) was added to a soln of **11** (5 g, 14.7 mmol) in CH_2Cl_2 (50 mL), and the mixture was stirred at r.t. for 3 h then filtered. The filtrate was concentrated in vacuo to give a white solid; yield: 4.18 g (92%). This was used directly in the next step without further purification.

IR (KBr): 3420, 1733, 1715 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.20 (d, *J* = 6.4 Hz, 3 H, CH_3), 3.08 (dd, *J* = 2.4, 3.6 Hz, 1 H, C_3H), 4.22 (dd, *J* = 2.4, 4.4 Hz, 1 H, C_4H), 4.27–4.29 (m, 1 H), 6.12 (s, 1 H, C*H*Ph), 7.22–7.34 (m, 10 H, ArH), 8.98 (d, *J* = 4.4 Hz, 1 H, CHO).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.07, 59.10, 59.46, 60.48, 60.94, 127.75, 128.72, 128.88, 129.08, 128.62, 138.12, 138.37, 167.16, 198.27.

MS (ESI): *m*/*z* = 310 $[M + H]^+$.

Anal. Calcd for $C_{19}H_{19}NO_3$: C, 73.77; H, 6.19; N, 4.53. Found: C, 73.75; H, 6.20; N, 4.54.

(2*R*,3*R*)-1-(Diphenylmethyl)-3-[(1*R*)-1-hydroxyethyl]-4-oxoazetidine-2-carboxylic Acid (13)

A mixture of $KMnO_4$ (3.8 g, 24.3 mmol) and K_2CO_3 (5.0 g, 36.5 mmol) in H_2O (80 mL) was added to a soln of **12** (5 g, 16.2 mmol) in THF (120 mL), and the mixture was stirred at r.t. for 12 h. The precipitated inorganic materials were filtered off, and the THF was evaporated in vacuo. The aqueous layer was washed with EtOAc (2 × 10) to remove neutral compounds and then acidified to pH 4 with 6 M HCl and extracted again with EtOAc (3 × 20). The organic layer was dried ($MgSO_4$) and concentrated in vacuo to give white crystals; yield: 4.52 g (86%); mp 172.3–172.8 °C; $[\alpha]_D^{21.6}$ +0.144 (*c* 1.46, MeOH).

IR (KBr): 3528, 3480, 1707, 1735 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.27 (d, *J* = 6.4 Hz, 3 H, CH_3), 3.24 (dd, *J* = 2.4, 3.2 Hz, 1 H, C_3H), 4.19 (d, *J* = 2.4 Hz, 1 H, C_4H), 4.24–4.30 (m, 1 H), 5.92 (s, 1 H, C*H*Ph), 7.14–7.25 (m, 10 H, ArH).

^{13}C NMR (100 MHz,$CDCl_3$): δ = 21.24, 52.75, 61.87, 62.52, 63.83, 127.92, 128.40, 128.48, 128.56, 128.62, 137.98, 138.13, 167.61, 174.86.

MS (ESI): *m*/*z* = 347.4 $[M + Na]^+$.

Anal. Calcd for $C_{19}H_{19}NO_4$: C, 70.14; H, 5.89; N, 4.31. Found: C, 70.18; H, 5.88; N, 4.32.

(2*R*,3*R*)-1-(Diphenylmethyl)-3-[(1*R*)-1-hydroxyethyl]-4-oxoazetidin-2-yl Acetate (14)

AcOH (6 mL) and $Pb(OAc)_4$ (6.5 g, 14.8 mmol) were added successively to a soln of acid **13** (4 g, 12.3 mmol) in anhyd DMF (40 mL), and the suspension was heated at 65 °C for 2 h under N_2. The mixture was then added to H_2O (40 mL) and extracted with EtOAc (2 × 50 mL). The combined organic phases were washed with sat. aq $NaHCO_3$, dried ($MgSO_4$), and concentrated in vacuo to give a white solid; yield: 3.0 g (72%); $[\alpha]_D^{30}$ +24.9 (*c* 1.1, MeOH).

IR (KBr): 3460, 1767, 1748 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.22 (d, *J* = 6.4 Hz, 3 H, C*H*$_3$CH), 1.82 (s, 3 H, $COCH_3$), 2.58 (d, *J* = 3.6 Hz, 1 H, OH), 3.04 (dd, *J* = 0.8, 6.8 Hz, 1 H, C_3H), 4.01–4.05 (m, 1 H, CH_3C*H*), 5.74 (d, *J* = 0.8 Hz, 1 H, C_4H), 5.93 (s, 1 H, C*H*Ph), 7.14–7.27 (m, 10 H, ArH).

^{13}C NMR (100 MHz $CDCl_3$): δ = 20.80, 20.92, 60.89, 64.38, 65.11, 79.29, 127.73, 128.09, 128.13, 128.51, 128.56, 128.78, 137.75, 138.46, 165.59, 170.54.

MS (ESI): *m*/*z* = 362 $[M + Na]^+$.

Anal. Calcd for $C_{20}H_{21}NO_4$: C, 70.78; H, 6.24; N, 4.13. Found: C, 70.83; H, 6.23; N, 4.14.

(2*R*,3*R*)-3-{(1*R*)-1-[*tert*-Butyl(dimethyl)siloxy]ethyl}-1-(diphenylmethyl)-4-oxoazetidin-2-yl Acetate (15)

TBDMSCl (2.5 g, 16.5 mmol) and imidazole (2.8 g, 41.3 mmol) were added successively to a soln of acetate **14** (2 g, 5.9 mmol) in DMF (20 mL), and the mixture was stirred at r.t. for 24 h. The mixture was then diluted with EtOAc (40 mL), and washed with dilute HCl and H_2O. The organic phase was dried ($MgSO_4$) and concen-

trated in vacuo to give an oil; yield: 2.19 g (82%); $[\alpha]_D^{26}$ +45.26 (*c* 0.38, MeOH).

IR ($CHCl_3$): 1775, 1745 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = –0.09 (s, 3 H, CH_3), –0.03 (s, 3 H, CH_3), 0.79 (s, 9 H, CH_3), 1.14 (d, *J* = 6.4 Hz, 3 H, C*H*$_3$CH), 1.72 (s, 3 H, $COCH_3$), 3.06 (dd, *J* = 1.2, 3.6 Hz, 1 H, C_3H), 4.08–4.14 (m, 1 H, CH_3CH), 5.91 (s, 1 H, C*H*Ph), 6.13 (d, *J* = 1.2 Hz, 1 H, C_4-H), 7.16–7.27 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = –4.84, 17.86, 20.61, 22.18, 25.69, 60.48, 64.06, 64.86, 77.52, 127.38, 127.84, 128.06, 128.25, 128.55, 128.58, 137.84, 138.75, 165.93, 169.47.

MS (ESI): *m*/*z* = 476 $[M + Na]^+$.

Anal. Calcd for $C_{26}H_{35}NO_4Si$: C, 68.84; H, 7.78; N, 3.09. Found: C, 68.89; H, 7.77; N, 3.08.

(2*S*,3*R*)-3-((1*S*)-1-[*tert*-Butyl(dimethyl)siloxy]ethyl)-4-oxoazetidin-2-yl Acetate (3)

NBS (1 g, 5.6 mmol) was added to a soln of acetate **15** (2.24 g, 5.1 mmol) and Br_2 [1.3 mL of a soln of Br_2 (0.1 mL) in CH_2Cl_2 (10 mL), 0.25 mmol] in CH_2Cl_2 (70 mL) and H_2O (45 mL). The mixture was stirred and irradiated by UV lamps at r.t. for 24 h, then washed with 5% aq $NaHSO_3$ until it became colorless. The organic phase was then concentrated in vacuo. The residue was dissolved in 1:1 acetone–H_2O (70 mL) containing TsOH (0.94 g, 4.9 mmol), and the mixture was stirred in the dark at r.t. for 24 h. The acetone was evaporated off and the aqueous layer was extracted with EtOAc (3 × 30 mL). The resulting organic phase was dried ($MgSO_4$) and concentrated in vacuo to give a residue that was purified by column chromatography [EtOAc–PE (1:5) to give a white solid; yield: 1.0 g (68%); mp 106.7–107.0 °C (Lit.[17] 106–107 °C; $[\alpha]_D^{20}$ +49.5 (*c* 0.5, $CHCl_3$) [Lit.[17] $[\alpha]_D^{25}$ +51.0 (*c* 0.9, $CHCl_3$)].

IR (KBr): 1777 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = –0.06 (s, 3 H, CH_3), –0.01 (s, 3 H, CH_3), 0.793 (s, 9 H, CH_3), 1.19 (d, *J* = 6.4 Hz, 3 H, C*H*$_3$CH), 2.04 (s, 3 H, CH_3CO), 3.11 (dd, *J* = 1.2, 3.2 Hz 1 H, C_3H), 4.15 (m, 1 H, CH_3CH), 5.76 (d, *J* = 1.2 Hz, 1 H, C_4H), 6.33 (s, 1 H, NH).

^{13}C NMR (100 MHz, $CDCl_3$) δ = –5.19, –4.32, 17.92, 20.89, 22.32, 25.68, 63.86, 65.04, 75.12, 166.53, 171.26.

MS (ESI): *m*/*z* = 310 $[M + Na]^+$.

Anal. Calcd for $C_{13}H_{25}NO_4Si$: C, 54.32; H, 8.77; N, 4.87. Found: C, 54.28; H, 8.78; N, 4.86.

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