Supporting Information for the Paper

**Lewis Acid-Promoted Intermolecular Carbonyl-Ene Reaction of
Enantiopure 4-Oxoazetidine-2-carbaldehydes. Rapid Entry to Novel Fused
Polycyclic β -Lactams**

Benito Alcaide,^{*a} Pedro Almendros,^b Carmen Pardo,^a Carolina Rodríguez-Ranera,^a and Alberto Rodríguez-Vicente^a

^a*Departamento de Química Orgánica I. Facultad de Química. Universidad Complutense 28040-Madrid. Spain*

^b*Instituto de Química Orgánica General, CSIC, Juan de la Cierva 3, 28006-Madrid, Spain*

E-mail: alcaideb@quim.ucm.es

Table of Contents

Preparation and Characterization of Homoallylic Alcohol Derivatives 2–4	S2
Preparation and Characterization of the <i>O</i> -Acetylmandelates of Alcohol (+)-2b	S6
Preparation and Characterization of Methanesulfonates (+)-5b and (+)-5b	S7
Preparation and Characterization of Diels-Alder Cycloadduct (+)-6c	S8
Preparation and Characterization of tricyclic β -lactam (+)-7a	S8
Preparation and Characterization of bicyclic amide ($\pm$)-8b	S8
Preparation and Characterization of bicyclic β -lactam (+)-11b	S9

(3R,4S)-4-[(R)-1-Hydroxy-2-(1-cyclopentenyl)-ethyl]-3-methoxy-1-(p-methoxyphenyl)-2-azetidinone, (+)-2a. From 100 mg (0.43 mmol) of aldehyde (+)-**1a**, 88 mg (65%) of compound (+)-**2a** was obtained as a colorless solid. Mp: 140–142 °C (hexanes/ethyl acetate). $[\alpha]_D = +127.8$ (*c* 1.0, CHCl₃). ¹H-NMR: δ 1.86 (m, 2H), 2.31 (m, 6H), 2.49 (d, 1H, *J* = 3.4 Hz), 3.69 (s, 3H), 3.80 (s, 3H), 4.20 (m, 1H), 4.27 (t, 1H, *J* = 5.0 Hz), 4.63 (d, 1H, *J* = 5.1 Hz), 5.49 (br s, 1H), 6.86 and 7.44 (m, each 2H). ¹³C-NMR: δ 165.1, 156.7, 140.3, 139.7, 126.9, 120.4, 114.1, 82.6, 69.1, 60.4, 59.6, 55.4, 35.6, 35.0, 32.5, 23.3. IR (KBr, cm⁻¹): ν 3408, 1720. MS (EI), *m/z* : 317 (M⁺, 32), 87 (100). Anal. Calcd for C₁₈H₂₃NO₄: C, 68.12; H, 7.30; N, 4.41. Found: C, 68.22; H, 7.33; N, 4.39.

(3R,4S)-4-[(R)-1-Hydroxy-2-(1-cyclopentenyl)]-1-(p-methoxyphenyl)-3-phenoxy-2-azetidinone, (+)-2b. From 100 mg (0.34 mmol) of aldehyde (+)-**1b**, 75 mg (60%) of compound (+)-**2b** was obtained as a colorless solid. Mp: 149–151 °C (hexanes/ethyl acetate). $[\alpha]_D = +157.5$ (*c* 1.0, CHCl₃). ¹H-NMR: δ 1.91 (m, 2H), 2.40 (m, 7H), 3.81 (s, 3H), 4.33 (m, 1H), 4.66 (t, 1H, *J* = 5.6 Hz), 5.39 (d, 1H, *J* = 5.4 Hz), 5.54 (br s, 1H), 6.89 (m, 2H), 7.11 (m, 3H), 7.35 (m, 2H), 7.52 (m, 2H). ¹³C-NMR: δ 163.8, 157.4, 156.8, 140.0, 130.8, 129.7, 127.7, 122.7, 120.4, 115.9, 114.1, 79.6, 69.5, 60.9, 55.4, 35.9, 35.0, 32.5, 23.3. IR (KBr, cm⁻¹): ν 3529, 1743. MS (EI), *m/z* : 380 (M⁺ + 1, 11), 379 (M⁺, 39), 149 (100). Anal. Calcd for C₂₃H₂₅NO₄: C, 72.80; H, 6.64; N, 3.69. Found: C, 72.93; H, 6.57; N, 3.72.

(3R,4S)-3-Benzoyloxy-4-[(R)-1-hydroxy-2-(1-cyclopentenyl)-ethyl]-1-(2-propenyl)-2-azetidinone, (+)-2c. From 100 mg (0.41 mmol) of aldehyde (+)-**1c**, 81 mg (60%) of compound (+)-**2c** was obtained as a colorless solid. Mp: 65–67 °C (hexanes/ethyl acetate). $[\alpha]_D = +41.1$ (*c* 1.0, CHCl₃). ¹H-NMR: δ 1.86 (m, 2H), 2.34 (m, 7H), 3.67 (t, 1H, *J* = 5.0 Hz), 3.82 (ddt, 1H, *J* = 15.6, 5.1, 1.6 Hz), 4.67 (d, 1H, *J* = 4.9 Hz), 4.70 and 4.98 (d, each 1H, *J* = 11.7 Hz), 5.24 (m, 2H), 5.48 (br s, 1H), 5.80 (m, 1H), 7.35 (m, 5H). ¹³C-NMR: δ 167.6, 140.1, 136.8, 132.0, 128.5, 128.1, 127.9, 127.4, 118.3, 80.8, 73.1, 68.9, 60.5, 44.2, 35.9, 34.9, 32.4, 23.3. IR (KBr, cm⁻¹): ν 3427, 1747. MS (CI), *m/z* : 328 (M⁺ + 1, 100), 327 (M⁺, 22). Anal. Calcd for C₂₀H₂₅NO₃: C, 73.37; H, 7.70; N, 4.28. Found: C, 73.26; H, 7.74; N, 4.31.

(3R,4S)-3-Benzoyloxy-4-[(R)-1-hydroxy-2-(1-cyclopentenyl)-ethyl]-1-(3-butenyl)-2-azetidinone, (+)-2d. From 87 mg (0.34 mmol) of aldehyde (+)-**1d**, 70 mg (61%) of compound (+)-

2d was obtained as a colorless solid. Mp: 57–58 °C (hexanes/ethyl acetate). $[\alpha]_D = +55.0$ (*c* 1.0, CHCl₃). ¹H-NMR: δ 1.88 (m, 2H), 2.31 (m, 9H), 3.26 (m, 1H), 3.63 (m, 1H), 3.67 (t, 1H, *J* = 5.1 Hz), 4.00 (m, 1H), 4.63 (d, 1H, *J* = 4.9 Hz), 4.69 and 4.97 (d, each 1H, *J* = 11.7 Hz), 5.08 (m, 2H), 5.51 (br s, 1H), 5.76 (m, 1H), 7.35 (m, 5H). ¹³C-NMR: δ 167.8, 140.1, 136.9, 135.0, 128.4, 128.0, 127.9, 127.8, 127.6, 80.6, 73.0, 69.0, 60.4, 40.9, 36.0, 34.9, 32.4, 31.8, 23.3. IR (KBr, cm⁻¹): ν 1743. MS (CI), *m/z* : 342 (*M*⁺ + 1, 100), 341 (*M*⁺, 19). Anal. Calcd for C₂₁H₂₇NO₃: C, 73.87; H, 7.97; N, 4.10. Found: C, 73.97; H, 7.96; N, 4.08.

(3*R*,4*S*)-4-[(*R*)-1-Hydroxy-2-(1-cyclopentenyl)-ethyl]-3-methoxy-1-(2-propynyl)-2-azetidinone, (+)-2e. From 90 mg (0.54 mmol) of aldehyde (+)-**1e**, 68 mg (51%) of compound (+)-**2e** was obtained as a colorless solid. Mp: 87–89 °C (hexanes/ethyl acetate). $[\alpha]_D = +60.0$ (*c* 1.0, CHCl₃). ¹H-NMR: δ 1.88 (m, 2H), 2.35 (m, 8H), 3.60 (s, 3H), 3.81 (t, 1H, *J* = 4.9 Hz), 3.93 (dd, 1H, *J* = 17.6, 2.7 Hz), 4.02 (m, 1H), 4.40 (dd, 1H, *J* = 17.6, 2.7 Hz), 4.48 (d, 1H, *J* = 4.9 Hz), 5.55 (br s, 1H). ¹³C-NMR: δ 166.8, 139.9, 127.2, 83.3, 72.4, 68.5, 59.9, 59.3, 35.8, 34.9, 32.3, 30.8, 23.2. IR (KBr, cm⁻¹): ν 3423, 1754. MS (CI), *m/z* : 250 (*M*⁺ + 1, 100), 249 (*M*⁺, 15). Anal. Calcd for C₁₄H₁₉NO₃: C, 67.45; H, 7.68; N, 5.62. Found: C, 67.34; H, 7.72; N, 5.65.

(3*R*,4*S*)-3-Allyloxy-4-[(*R*)-1-hydroxy-2-(1-cyclopentenyl)-ethyl]-1-(*p*-methoxyphenyl)-2-azetidinone, (+)-2g. From 100 mg (0.38 mmol) of aldehyde (+)-**1g**, 109 mg (83%) of compound (+)-**2g** was obtained as a colorless solid. Mp: 67–69 °C (hexanes/ethyl acetate). $[\alpha]_D = +105.5$ (*c* 1.2, CHCl₃). ¹H-NMR: δ 1.87 (m, 2H), 2.32 (m, 6H), 2.53 (d, 1H, *J* = 2.9 Hz), 3.79 (s, 3H), 4.22 (m, 1H), 4.29 (t, 1H, *J* = 5.0 Hz), 4.46 (ddt, 1H, *J* = 12.7, 5.1, 1.3 Hz), 4.76 (d, 1H, *J* = 5.1 Hz), 5.33 (m, 2H), 5.49 (br s, 1H), 5.96 (m, 1H), 6.86 (m, 2H), 7.43 (m, 2H). ¹³C-NMR: δ 165.3, 156.7, 140.3, 133.2, 130.9, 127.1, 120.4, 118.2, 114.1, 80.4, 72.5, 69.3, 60.5, 55.4, 35.6, 35.0, 32.5, 23.3. IR (KBr, cm⁻¹): ν 3522, 1745. MS (EI), *m/z* : 344 (*M*⁺ + 1, 9), 343 (*M*⁺, 100). Anal. Calcd for C₂₀H₂₅NO₄: C, 69.95; H, 7.34; N, 4.08. Found: C, 70.05; H, 7.37; N, 4.10.

(3*R*,4*S*)-4-[(*R*)-1-Hydroxy-2-(1-cyclopentenyl)-ethyl]-1-(3-butenyl)-3-phthalimido-2-azetidinone, (–)-2i. From 186 mg (0.62 mmol) of aldehyde (–)-**1i**, 121 mg (51%) of compound (–)-**2i** was obtained as a colorless oil. $[\alpha]_D = -22.1$ (*c* 1.0, CHCl₃). ¹H-NMR: δ 1.69 (m, 2H), 1.95 (m, 2H), 2.15 (m, 4H), 2.26 (d, 1H, *J* = 3.2 Hz), 2.47 (m, 2H), 3.46 (m, 1H), 3.69 (m, 1H), 3.84 (dd, 1H, *J* = 8.8, 5.1 Hz), 3.99 (tt, 1H, *J* = 8.8, 2.9 Hz), 5.15 (m, 2H), 5.33 (d, 1H, *J* = 5.1 Hz), 5.37 (br s, 1H),

5.85 (m, 1H), 7.78 (m, 2H), 7.86 (m, 2H). ^{13}C -NMR: δ 167.4, 163.8, 138.8, 135.2, 134.6, 131.4, 128.5, 123.7, 117.1, 69.1, 61.3, 54.7, 41.7, 36.4, 34.7, 32.4, 31.9, 23.1. IR (CHCl_3 , cm^{-1}): ν 3402, 1757. MS (CI), m/z : 381 ($\text{M}^+ + 1$, 100), 380 (M^+ , 11). Anal. Calcd for $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_4$: C, 69.46; H, 6.36; N, 7.36. Found: C, 69.38; H, 6.33; N, 7.40.

(3R,4S)-3-Benzoyloxy-4-[(R)-1-hydroxy-2-(1-cyclohexenyl)-ethyl]-1-(2-propenyl)-2-azetidinone, (+)-3b. From 100 mg (0.41 mmol) of aldehyde (+)-**1c**, 51 mg (37%) of compound (+)-**3b** was obtained as a colorless solid. Mp: 76–78 °C (hexanes/ethyl acetate). $[\alpha]_{\text{D}} = +31.6$ (c 1.0, CHCl_3). ^1H -NMR: δ 1.58 (m, 5H), 1.96 (m, 4H), 2.29 (m, 2H), 3.66 (t, 1H, $J = 5.4$ Hz), 3.83 (ddt, 1H, $J = 15.7, 6.8, 1.2$ Hz), 3.95 (m, 1H), 4.17 (ddt, 1H, $J = 15.7, 5.1, 1.6$ Hz), 4.66 (d, 1H, $J = 5.1$ Hz), 4.70 and 4.99 (d, each 1H, $J = 11.7$ Hz), 5.23 (m, 2H), 5.52 (br s, 1H), 5.77 (m, 1H), 7.35 (m, 5H). ^{13}C -NMR: δ 167.8, 137.0, 133.9, 132.2, 128.6, 128.2, 128.0, 125.4, 118.4, 80.9, 73.3, 68.8, 60.8, 44.3, 42.9, 28.3, 25.4, 23.0, 22.4. IR (KBr, cm^{-1}): ν 3443, 1726. MS (CI), m/z : 342 ($\text{M}^+ + 1$, 100), 341 (M^+ , 15). Anal. Calcd for $\text{C}_{21}\text{H}_{27}\text{NO}_3$: C, 73.87; H, 7.97; N, 4.10. Found: C, 73.96; H, 7.92; N, 4.12.

(3R,4S)-4-[(R)-1-Hydroxy-3-phenyl-3-butenyl]-3-methoxy-1-(p-methoxy-phenyl)-2-azetidinone, (+)-4a. From 100 mg (0.43 mmol) of aldehyde (+)-**1a**, 100 mg (66%) of compound (+)-**4a** was obtained as a colorless oil. $[\alpha]_{\text{D}} = +68.9$ (c 1.0, CHCl_3). ^1H -NMR: δ 2.35 (d, 1H, $J = 4.0$ Hz), 2.63 (dd, 1H, $J = 10.3, 9.6$ Hz), 2.97 (ddd, 1H, $J = 14.1, 3.7, 1.0$ Hz), 3.69 (s, 3H), 3.79 (s, 3H), 4.12 (m, 1H), 4.30 (t, 1H, $J = 5.5$ Hz), 4.62 (d, 1H, $J = 5.4$ Hz), 5.16 (br s, 1H), 5.44 (d, 1H, $J = 1.0$ Hz), 6.86 (m, 2H), 7.30 (m, 2H), 7.39 (m, 5H). ^{13}C -NMR: δ 165.4, 157.0, 144.6, 140.1, 130.9, 128.6, 128.0, 126.4, 120.7, 115.8, 114.3, 82.8, 69.7, 60.7, 60.0, 55.7, 39.7. IR (CHCl_3 , cm^{-1}): ν 3475, 1747. MS (CI), m/z : 354 (M^+ , 100), 353 (11). Anal. Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_4$: C, 71.37; H, 6.56; N, 3.96. Found: C, 71.29; H, 6.52; N, 3.99.

(3R,4S)-3-Benzoyloxy-4-[(R)-1-hydroxy-3-phenyl-3-butenyl]-1-(3-butenyl)-2-azetidinone, (+)-4b. From 75 mg (0.29 mmol) of aldehyde (+)-**1d**, 65 mg (60%) of compound (+)-**4b** was obtained as a colorless oil. $[\alpha]_{\text{D}} = +4.6$ (c 0.8, CHCl_3). ^1H -NMR: δ 2.21 (d, 1H, $J = 2.4$ Hz), 2.37 (m, 2H), 2.47 (dd, 1H, $J = 13.9, 9.8$ Hz), 3.08 (ddd, 1H, $J = 13.9, 3.4, 1.1$ Hz), 3.27 (m, 1H), 3.57 (m, 1H), 3.67 (dd, 1H, $J = 7.1, 5.1$ Hz), 3.94 (m, 1H), 4.64 (d, 1H, $J = 4.9$ Hz), 4.68 and 5.0 (d, each 1H, $J = 11.2$ Hz), 5.08 (m, 2H), 5.18 (br s, 1H), 5.50 (br s, 1H), 5.76 (m, 1H), 7.22 (m, 5H), 7.38 (m,

5H). $^{13}\text{C-NMR}$: δ 167.8, 143.9, 139.1, 136.8, 135.0, 128.5, 128.4, 128.3, 128.1, 127.8, 126.1, 116.9, 115.8, 80.5, 73.2, 69.6, 60.6, 40.8, 40.0, 31.8. IR (CHCl_3 , cm^{-1}): ν 1743. MS (EI), m/z : 378 ($\text{M}^+ + 1$, 7), 377 (M^+ , 100). Anal. Calcd for $\text{C}_{24}\text{H}_{27}\text{NO}_3$: C, 76.36; H, 7.21; N, 3.71. Found: C, 76.46; H, 7.25; N, 3.69.

(3R,4S)-4-[(R)-1-Hydroxy-3-phenyl-3-butenyl]-3-methoxy-1-(2-propynyl)-2-azetidinone, (+)-4c. From 100 mg (0.60 mmol) of aldehyde (+)-**1e**, 110 mg (64%) of compound (+)-**4c** was obtained as a colorless oil. $[\alpha]_{\text{D}} = +16.3$ (c 1.0, CHCl_3). $^1\text{H-NMR}$: δ 2.30 (t, 1H, $J = 2.5$ Hz), 2.46 (d, 1H, $J = 3.4$ Hz), 2.62 (dd, 1H, $J = 13.8, 9.1$ Hz), 2.99 (ddd, 1H, $J = 14.1, 3.9, 0.8$ Hz), 3.59 (s, 3H), 3.82 (t, 1H, $J = 5.4$ Hz), 3.95 (m, 1H), 3.96 and 4.36 (dd, each 1H, $J = 17.6, 2.5$ Hz), 4.45 (d, 1H, $J = 5.0$ Hz), 5.25 (d, 1H, $J = 1.0$ Hz), 5.49 (d, 1H, $J = 1.3$ Hz), 7.31 (m, 3H), 7.70 (m, 2H). $^{13}\text{C-NMR}$: δ 166.8, 143.8, 139.6, 128.3, 127.8, 126.0, 115.7, 83.1, 72.5, 69.0, 60.0, 59.2, 43.1, 39.9, 30.8. IR (CHCl_3 , cm^{-1}): ν 3462, 1755. MS (CI), m/z : 286 ($\text{M}^+ + 1$, 100), 285 (M^+ , 5). Anal. Calcd for $\text{C}_{17}\text{H}_{19}\text{NO}_3$: C, 71.56; H, 6.71; N, 4.91. Found: C, 71.48; H, 6.68; N, 4.94.

(3R,4S)-3-Allyloxy-4-[(R)-1-hydroxy-3-phenyl-3-butenyl]-1-(p-methoxy-phenyl)-2-azetidinone, (+)-4e. From 82 mg (0.31 mmol) of aldehyde (+)-**1g**, 50 mg (42%) of compound (+)-**4e** was obtained as a colorless oil. $[\alpha]_{\text{D}} = +35.5$ (c 1.1, CHCl_3). $^1\text{H-NMR}$: δ 2.38 (d, 1H, $J = 3.7$ Hz), 2.62 (dd, 1H, $J = 14.2, 9.5$ Hz), 3.01 (dd, 1H, $J = 14.2, 3.7$ Hz), 3.78 (s, 3H), 4.18 (m, 2H), 4.28 (t, 1H, $J = 5.5$ Hz), 4.47 (ddt, 1H, $J = 12.3, 5.5, 1.2$ Hz), 4.75 (d, 1H, $J = 5.1$ Hz), 5.16 (br s, 1H), 5.34 (m, 2H), 5.44 (br s, 1H), 5.97 (m, 1H), 6.84 (m, 2H), 7.43 (m, 7H). $^{13}\text{C-NMR}$: δ 165.2, 156.7, 144.3, 139.7, 133.2, 130.7, 128.3, 127.7, 126.2, 120.4, 118.7, 115.6, 114.0, 80.4, 72.6, 69.6, 60.7, 55.4, 39.5. IR (KBr, cm^{-1}): ν 3528, 1742. MS (CI), m/z : 380 ($\text{M}^+ + 1$, 100), 379 (M^+ , 11). Anal. Calcd for $\text{C}_{23}\text{H}_{25}\text{NO}_4$: C, 72.80; H, 6.64; N, 3.69. Found: C, 72.91; H, 6.60; N, 3.66.

(3R,4S)-4-[(R)-1-Hydroxy-3-phenyl-3-butenyl]-1-(3-butenyl)-3-phthalimido-2-azetidinone, (-)-4g. From 77 mg (0.26 mmol) of aldehyde (-)-**1i**, 50 mg (46%) of compound (-)-**4g** was obtained as a colorless oil. $[\alpha]_{\text{D}} = -41.0$ (c 1.8, CHCl_3). $^1\text{H-NMR}$: δ 2.15 (br s, 1H), 2.41 (m, 4H), 3.45 (m, 1H), 3.67 (m, 1H), 3.85 (m, 2H), 5.14 (m, 2H), 5.29 (s, 1H), 5.33 (d, 1H, $J = 5.1$ Hz), 6.96 (m, 5H), 7.83 (m, 4H). $^{13}\text{C-NMR}$: δ 167.3, 163.7, 143.7, 139.7, 135.2, 134.6, 131.6, 128.2, 127.7, 125.8, 123.9, 117.1, 116.8, 68.9, 61.3, 54.9, 41.8, 41.2, 31.8. IR (CHCl_3 , cm^{-1}): ν 3424, 1751.

MS (CI), m/z : 417 ($M^+ + 1$, 100), 416 (M^+ , 7). Anal. Calcd for $C_{25}H_{24}N_2O_4$: C, 72.10; H, 5.81; N, 6.73. Found: C, 72.02; H, 5.79; N, 6.77.

General Procedure for the Preparation of the *O*-Acetylmandelates of Homoallylic Alcohol (+)-2b**.** (*R*)– or (*S*)–*O*-Acetylmandelic acid (20 mg, 0.10 mmol) and 4-dimethylaminopyridine (DMAP) (cat.) were added to a solution of the homoallylic alcohol (+)-**2b** (0.09 mmol) in dichloromethane (1.0 mL) followed by a solution of dicyclohexylcarbodiimide (DCC) (37 mg, 0.18 mmol) in dichloromethane (500 μ L) at 0 °C. The reaction mixture was allowed to warm to room temperature and stirred for 16 h. The solvent was removed under reduced pressure and diethyl ether was added. The mixture was then filtered and the filtrate was concentrated under reduced pressure. Chromatography of the residue eluting with hexanes/ethyl acetate mixtures gave analytically pure *O*-acetylmandelates.

(*R*)-*O*-Acetylmandelate of (3*R*,4*S*)-4-[(*R*)-1-hydroxy-2-(1-cyclopentenyl)]-1-(*p*-methoxyphenyl)-3-phenoxy-2-azetidinone. From 30 mg (0.08 mmol) of alcohol (+)-**2b**, 33 mg (75%) of its (*R*)-*O*-acetylmandelate was obtained as a colorless oil. $[\alpha]_D = +79.6$ (c 1.4, $CHCl_3$). 1H -NMR: δ 1.61 (m, 2H), 1.83 (m, 2H), 2.13 (s, 3H), 2.27 (m, 2H), 2.58 (m, 2H), 3.82 (s, 3H), 4.53 (dd, 1H, $J = 5.1, 4.4$ Hz), 5.22 (d, 1H, $J = 5.4$ Hz), 5.44 (br s, 1H), 5.58 (dt, 1H, $J = 6.1, 4.1$ Hz), 5.83 (s, 1H), 7.13 (m, 14H). ^{13}C -NMR: δ 169.8, 167.9, 163.2, 157.4, 157.0, 138.7, 130.2, 129.6, 129.1, 128.7, 128.4, 127.8, 127.6, 122.5, 120.3, 115.9, 114.5, 79.5, 74.6, 71.0, 57.6, 55.6, 35.1, 33.6, 32.6, 23.6, 20.8. IR ($CHCl_3$, cm^{-1}): ν 1743, 1648. MS (CI), m/z : 556 ($M^+ + 1$, 100), 555 (M^+ , 8). Anal. Calcd for $C_{33}H_{33}NO_7$: C, 71.23; H, 5.99; N, 2.52. Found: C, 71.34; H, 5.96; N, 2.50.

(*S*)-*O*-Acetylmandelate of (3*R*,4*S*)-4-[(*R*)-1-hydroxy-2-(1-cyclopentenyl)]-1-(*p*-methoxyphenyl)-3-phenoxy-2-azetidinone. From 30 mg (0.08 mmol) of alcohol (+)-**2b**, 31 mg (71%) of its (*S*)-*O*-acetylmandelate was obtained as a colorless solid. Mp: 125–127 °C (hexanes/ethyl acetate). $[\alpha]_D = +100.2$ (c 2.0, $CHCl_3$). 1H -NMR: δ 1.22 (m, 1H), 1.61 (m, 3H), 1.98 (m, 2H), 2.11 (s, 3H), 2.45 (m, 2H), 3.82 (s, 3H), 4.60 (t, 1H, $J = 5.6$ Hz), 4.87 (br s, 1H), 5.45 (d, 1H, $J = 4.9$ Hz), 5.50 (s, 1H), 5.66 (m, 1H), 7.17 (m, 14H). ^{13}C -NMR: δ 169.8, 167.8, 163.0, 157.4, 157.0, 138.1, 133.7, 130.0, 129.6, 129.0, 128.5, 127.8, 127.7, 122.5, 119.9, 115.6, 114.4, 79.2, 74.1, 71.4, 57.9, 55.5, 34.5, 34.0, 32.3, 23.2, 20.6. IR ($CHCl_3$, cm^{-1}): ν 1745, 1649. MS (CI), m/z : 556 ($M^+ + 1$, 100), 555

(M⁺, 11). Anal. Calcd for C₃₃H₃₃NO₇: C, 71.23; H, 5.99; N, 2.52. Found: C, 71.33; H, 6.03; N, 2.55.

Methanesulfonate of (3*R*,4*S*)-3-propargyloxy-4-[(*R*)-1-hydroxy-2-(1-cyclopentenyl)-ethyl]-1-(*p*-methoxyphenyl)-2-azetidinone, (+)-5b. From 224 mg (0.66 mmol) of alcohol (+)-**2h**, 205 mg (74%) of compound (+)-**5b** was obtained as a colorless oil. [α]_D = +77.2 (*c* 2.4, CHCl₃). ¹H-NMR: δ 1.90 (m, 2H), 2.33 (m, 4H), 2.57 (m, 4H), 2.71 (m, 2H), 3.79 (s, 3H), 4.51 (m, 3H), 5.00 (d, 1H, *J* = 5.4 Hz), 5.15 (ddd, 1H, *J* = 7.6, 5.9, 4.9 Hz), 5.30 (s, 1H), 5.59 (br s, 1H), 6.88 (m, 2H), 7.35 (m, 2H). ¹³C-NMR: δ 164.3, 156.9, 138.1, 130.2, 129.4, 119.8, 114.3, 80.2, 79.4, 78.2, 76.2, 58.8, 57.7, 55.4, 38.1, 35.6, 34.1, 32.5, 23.5. IR (CHCl₃, cm⁻¹): ν 1744. MS (CI), *m/z* : 420 (M⁺ + 1, 100), 419 (M⁺, 9). Anal. Calcd for C₂₁H₂₅NSO₆: C, 60.13; H, 6.01; N, 3.34. Found: C, 60.23; H, 6.04; N, 3.32.

Methanesulfonate of (3*R*,4*S*)-4-[(*R*)-1-hydroxy-3-phenyl-3-butenyl]-3-methoxy-1-(3-butynyl)-2-azetidinone, (+)-5c. From 140 mg (0.47 mmol) of alcohol (+)-**4d**, 199 mg (70%) of compound (+)-**5c** was obtained as a colorless oil. [α]_D = +3.9 (*c* 1.7, CHCl₃). ¹H-NMR: δ 1.96 (t, 1H, *J* = 2.6 Hz), 2.45 (m, 2H), 2.85 (s, 3H), 3.00 (m, 2H), 3.27 (m, 1H), 3.57 (s, 3H), 3.72 (m, 1H), 4.01 (dd, 1H, *J* = 7.8, 5.1 Hz), 4.43 (d, 1H, *J* = 5.1 Hz), 5.03 (td, 1H, *J* = 7.8, 4.1 Hz), 5.27 (br s, 1H), 5.53 (d, 1H, *J* = 1.0 Hz), 7.39 (m, 5H). ¹³C-NMR: δ 167.6, 142.7, 139.5, 128.6, 128.1, 126.1, 117.4, 82.7, 80.8, 70.4, 59.0, 58.5, 39.7, 38.9, 38.0, 31.5, 17.1. IR (CHCl₃, cm⁻¹): ν 1746. MS (CI), *m/z* : 378 (M⁺ + 1, 100), 377 (M⁺, 11). Anal. Calcd for C₁₉H₂₃NSO₅: C, 60.46; H, 6.14; N, 3.71. Found: C, 60.58; H, 6.10; N, 3.68.

Cycloadduct (+)-6c. From 36 mg (0.10 mmol) of methanesulfonate (+)-**5c** and after heating at 140 °C for 44 h, 15 mg (56%) of compound (+)-**6c** was obtained as a colorless oil. [α]_D = +174.8 (*c* 0.5, CHCl₃). ¹H-NMR: δ 2.27 (m, 2H), 2.79 (dddd, 1H, *J* = 13.0, 10.0, 6.7, 1.4 Hz), 3.15 (m, 3H), 3.32 (dd, 1H, *J* = 9.5, 4.1 Hz), 3.66 (s, 3H), 3.96 (ddd, 1H, *J* = 13.0, 5.2, 2.2 Hz), 4.58 (dd, 1H, *J* = 4.1, 1.4 Hz), 5.79 (d, 1H, *J* = 1.1 Hz), 5.97 (t, 1H, *J* = 1.6 Hz), 7.36 (m, 5H). ¹³C-NMR: δ 167.2, 140.6, 135.1, 131.4, 128.3, 127.4, 125.1, 121.1, 121.0, 85.1, 59.4, 59.3, 39.6, 38.0, 33.4, 28.7. IR

(CHCl₃, cm⁻¹): ν 1751. MS (ES), m/z : 304 (M⁺ + 23, 14), 282 (M⁺ + 1, 100), 281 (M⁺, 7). Anal. Calcd for C₁₈H₁₉NO₂: C, 76.84; H, 6.81; N, 4.98. Found: C, 76.95; H, 6.85; N, 4.94.

Tricyclic β -Lactam (+)-7a. From 15 mg (0.06 mmol) of cycloadduct (+)-**6a**, 100 mg (100%) of tricycle (+)-**7a** was obtained as a colorless oil. $[\alpha]_D = +13.2$ (c 0.4, CHCl₃). ¹H-NMR: δ 2.09 (m, 2H), 2.89 (m, 7H), 3.51 (s, 3H), 4.05 (m, 1H), 4.69 (d, 1H, $J = 4.5$ Hz), 4.79 (d, 1H, $J = 4.5$ Hz), 7.00 (s, 1H), 7.04 (s, 1H). IR (CHCl₃, cm⁻¹): ν 1762. MS (CI), m/z : 244 (M⁺ + 1, 100), 243 (M⁺, 16). Anal. Calcd for C₁₅H₁₇NO₂: C, 74.05; H, 7.04; N, 5.76. Found: C, 73.97; H, 7.00; N, 5.79.

Bicyclic Amide ($\pm$)-8b. From 50 mg (0.11 mmol) of methanesulfonate (+)-**5d**, 27 mg (69%) of bicycle ($\pm$)-**8b** was obtained as a colorless solid. Mp: 186–187 °C (hexanes/ethyl acetate). ¹H-NMR: δ 3.08 (dd, 1H, $J = 11.2, 16.3$ Hz), 3.38 (dd, 1H, $J = 3.8, 16.3$ Hz), 3.82 (s, 3H), 4.36 (dd, 1H, $J = 3.8, 11.2$ Hz), 4.99 (d, 1H, $J = 15.1$ Hz), 5.10 (d, 1H, $J = 14.9$ Hz), 6.90 (m, 2H), 7.13 (d, 1H, $J = 8.1$ Hz), 7.47 (m, 9H), 8.38 (br s, 1H). ¹³C-NMR: δ 168.8, 156.6, 140.6, 140.2, 132.8, 132.6, 130.4, 128.8, 127.7, 127.4, 127.0, 125.4, 124.6, 121.5, 114.2, 74.6, 68.3, 55.5, 31.3. IR (KBr, cm⁻¹): ν 3318, 1665. MS (CI), m/z : 360 (M⁺ + 1, 100), 359 (M⁺, 7). Anal. Calcd for C₂₃H₂₁NO₃: C, 76.86; H, 5.89; N, 3.90. Found: C, 76.97; H, 5.92; N, 3.88.

Bicyclic β -Lactam (+)-11b. From 60 mg (0.20 mmol) of ene adduct (+)-**4d**, 47 mg (36%) of compound (+)-**11b** was obtained as a colorless oil. $[\alpha]_D = +107.1$ (c 1.8, CHCl₃). ¹H-NMR: δ 2.23 (m, 2H), 2.87 (m, 3H), 3.34 (d, 1H, $J = 4.9$ Hz), 3.41 (s, 3H), 3.52 (m, 2H), 3.72 (d, 1H, $J = 4.6$ Hz), 3.80 (m, 1H), 3.97 (m, 1H), 6.19 (s, 1H), 7.43 (m, 20H). ¹³C-NMR: δ 167.4, 158.8, 148.9, 138.4, 137.3, 136.9, 136.6, 129.3, 129.2, 129.1, 128.8, 128.6, 128.5, 128.3, 126.9, 126.0, 84.1, 67.6, 62.4, 59.5, 50.5, 40.7, 39.3, 39.1, 37.8. IR (CHCl₃, cm⁻¹): ν 1743. MS (EI), m/z : 650 (M⁺, 6), 574 (M⁺ – 76, 100). Anal. Calcd for C₃₆H₃₇NO₃Sn: C, 66.48; H, 5.73; N, 2.15. Found: C, 66.62; H, 5.77; N, 2.11.