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Supporting Information

A Practical Ruthenium-Catalyzed Cleavage of Allyl Protecting Group in Amides, Lactams, Imides and Congeners

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General Procedure for the Preparation of Allylic Compounds 1a–d, 13a, 13b, and 16b. The appropriate *NH*-amide-like compound (2.35 mmol) in THF or DMF (5 mL) was added dropwise to a stirred suspension of sodium hydride (3.52 mmol) in THF or DMF (20 mL) cooled at 0 ^{o}C. After 30 min, allyl bromide (0.30 mL, 3.52 mmol) was slowly added and the mixture was stirred at room temperature. until dissapearance of the starting material (TLC). Then, saturated aqueous sodium hydrogen carbonate (1 mL) was added at 0 ^{o}C, and the mixture was allowed to warm to room temperature, before being extracted with ethyl acetate (3 x 10 mL). The organic extract was washed with brine, dried ($MgSO_4$) and concentrated under reduced pressure. Chromatography of the residue eluting with hexanes/ethyl acetate mixtures gave analytically pure compounds **1a–d**, **13a**, **13b**, and **16a–d**. Spectroscopic and analytical data for some representative forms of **1a–d**, **13a**, **13b**, and **16a–d** follow.

1-Allyl-pyrrolidin-2-one 1a. From 200 mg (2.35 mmol) of pyrrolidin-2-one, compound **1a** (290 mg, 98%) was obtained as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 5.67 (m, 1H), 5.16 (m, 2H), 3.81 (d, *J* = 6.0 Hz, 2H), 3.29 (dd, *J*= 7.1, 6.9 Hz, 2H), 2.34 (dd, *J* = 8.3, 7.8 Hz, 2H), 2.01 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 174.4, 132.2, 117.4, 46.4, 44.9, 30.7, 17.5; IR ($CHCl_3$): ν = 1724 cm^{-1}; MS (EI): *m/z* (%): 126 (7) $[M + H]^+$, 125 (100) $[M]^+$; elemental analysis calcd (%) for $C_7H_{11}NO$ (125.2): C 67.17, H 8.86, N 11.19; found C 67.29, H 8.83, N 11.15.

(*S*)-1-Allyl-5-oxo-pyrrolidine-2-carboxylic acid ethyl ester (+)-1b. From 400 mg (2.55 mmol) of 5-oxo-pyrrolidine-

2-carboxylic acid ethyl ester, and after chromatography of the residue using hexanes/ethyl acetate (3:1) as eluent, compound (+)-**1b** (403 mg, 80%) was obtained as a yellow oil; $[\alpha]_D$ = +18.5 (*c* = 1.0 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 5.67 (m, 1H), 5.11 (m, 2H), 4.18 (m, 4H), 3.40 (dd, *J*= 15.3, 7.5 Hz, 1H), 2.20 (m, 4H), 1.18 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 174.9, 171.8, 131.9, 118.7, 61.4, 59.0, 44.4, 29.5, 22.9, 14.1; IR ($CHCl_3$): ν = 1740, 1722 cm^{-1}; MS (ES): *m/z* (%): 198 (100) $[M + H]^+$, 197 (9) $[M]^+$; elemental analysis calcd (%) for $C_{10}H_{15}NO_3$ (197.2): C 60.90, H 7.67, N 7.10; found C 60.77, H 7.70, N 7.07.

1-Allyl-piperidin-2-one 1c. From 200 mg (2.02 mmol) of piperidin-2-one, compound **1c** (232 mg, 92%) was obtained as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 5.69 (m, 1H), 5.05 (m, 2H), 3.92 (d, *J* = 6.0 Hz, 2H), 3.15 (m, 2H), 2.30 (m, 2H), 1.73 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 169.3, 132.8, 116.9, 49.2, 47.2, 32.2, 23.1, 20.9; IR ($CHCl_3$): ν = 1654 cm^{-1}; MS (EI): *m/z* (%): 140 (5) $[M + H]^+$, 139 (100) $[M]^+$; elemental analysis calcd (%) for $C_8H_{13}NO$ (139.2): C 69.03, H 9.41, N 10.06; found C 69.14, H 9.37, N 10.10.

1-Allyl-azepan-2-one 1d. From 100 mg (0.88 mmol) of azepan-2-one, compound **1d** (124 mg, 92%) was obtained as a yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 5.53 (m, 1H), 4.99 (m, 2H), 3.82 (d, *J* = 4.0 Hz, 2H), 3.09 (m, 2H), 2.38 (m, 2H), 1.45 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 175.6, 133.7, 117.1, 50.4, 48.7, 37.1, 29.9, 28.3, 23.2; IR ($CHCl_3$): ν = 1650 cm^{-1}; MS (ES): *m/z* (%): 154 (100) $[M + H]^+$, 153 (12) $[M]^+$; elemental analysis calcd (%) for $C_9H_{15}NO$

(153.2): C 70.55, H 9.87, N 9.14; found C 70.68, H 9.82, N 9.10.

***N*-Allyl-*N*-(4-methoxyphenyl)-acetamide 13a.** From 165 mg (1.0 mmol) of **15a**, compound **13a** (164 mg, 80%) was obtained as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.01 (m, 2H), 6.81 (m, 2H), 5.79 (m, 1H), 5.03 (m, 2H), 4.19 (d, *J* = 6.3 Hz, 2H), 3.75 (s, 3H), 1.77 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 170.7, 159.0, 135.8, 133.3, 129.2, 117.8, 114.7, 55.5, 52.2, 22.7; IR ($CHCl_3$): ν = 1652 cm^{-1}; MS (EI): *m/z* (%): 206 (3) [*M* + H]$^{+}$, 205 (100) [*M*]$^{+}$; elemental analysis calcd (%) for $C_{12}H_{15}NO_2$ (205.2): C 70.22, H 7.37, N 6.82; found C 70.35, H 7.33, N 6.85.

***N*-Allyl-*N*-benzyl-acetamide 13b.** From 149 mg (1.0 mmol) of **15b**, compound **13b** (142 mg, 75%) was obtained as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.32 (m, 5H), 5.70 (m, 1H), 5.10 (m, 2H), 4.52 (s, 2H), 3.85 (m, 2H), 2.08 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 171.2, 137.7, 132.6, 128.7, 128.4, 127.5, 116.9, 50.1, 48.3, 21.5; IR ($CHCl_3$): ν = 1650 cm^{-1}; MS (ES): *m/z* (%): 190 (100) [*M* + H]$^{+}$, 189 (11) [*M*]$^{+}$; elemental analysis calcd (%) for $C_{12}H_{15}NO$ (189.2): C 76.16, H 7.99, N 7.40; found C 76.30, H 7.96, N 7.44.

2-Allyl-4-methyl-1-phenyl-pyrazolidin-3-one 16b. From 200 mg (1.13 mmol) of **18b**, compound **16b** (217 mg, 89%) was obtained as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.20 (m, 2H), 6.91 (m, 3H), 5.73 (m, 1H), 5.10 (m, 2H), 4.32 (dd, *J*= 15.4, 5.6 Hz, 1H), 3.77 (dd, *J* = 11.2, 7.7 Hz, 1H), 3.56 (dd, *J* = 15.4, 6.4 Hz, 1H), 3.40 (t, *J* = 11.2 Hz, 1H), 2.30 (m, 1H), 1.03 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz,

$CDCl_3$, 25 °C): δ = 175.0, 149.9, 131.8, 129.1, 123.4, 118.4, 117.9, 63.6, 45.9, 34.1, 13.0; IR ($CHCl_3$): ν = 1720 cm^{-1}; MS (ES): *m/z* (%):217 (100) $[M + H]^+$, 216 (15) $[M]^+$; elemental analysis calcd (%) for $C_{13}H_{16}N_2O$ (216.3): C 72.19, H 7.46, N 12.95; found C 72.31, H 7.42, N 12.90.

General Procedure for the Preparation of *N*-Allylic Compounds 16a, 16c, and 16d. Allyl bromide (0.52 mL, 6.06 mmol) was added dropwise to a stirred suspension of the corresponding lactam-like compound **18** (2.02 mmol) and potassium carbonate (2.80 g, 20.20 mmol) in acetonitrile (45 mL), and the mixture was stirred for 30 min at reflux temperature. After cooling at RT, the solid was removed by filtration, and the filtrate was concentrated under reduced pressure to give analytically pure compounds **16a, 16c, and 16d**. Spectroscopic and analytical data for some representative pure forms follow.

1-Allyl-pyrrolidine-2,5-dione 16a. From 200 mg (2.02 mmol) of pyrrolidine-2,5-dione **18a**, compound **16a** (272 mg, 97%) was obtained as a colorless oil; ^1H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 5.72 (m, 1H), 5.09 (m, 2H), 3.96 (d, *J* = 5.8 Hz, 2H), 2.62 (s, 4H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 °C): δ = 176.5, 130.6, 117.9, 40.6, 27.9; IR ($CHCl_3$): ν = 1750 cm^{-1}; MS (EI): *m/z* (%):140 (3) $[M + H]^+$, 139 (100) $[M]^+$; elemental analysis calcd (%) for $C_7H_9NO_2$ (139.1): C 60.42, H 6.52, N 10.07; found C 60.54, H 6.49, N 10.03.

3-Allyl-1-methyl-imidazolidine-2,4-dione 16c. From 200 mg (1.75 mmol) of **18c**, compound **16c** (265 mg, 98%) was obtained as a colorless oil; ^1H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 5.70 (m, 1H), 5.07 (m, 2H), 3.99 (m, 2H), 3.78 (s, 2H),

2.88 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 169.5, 156.5, 131.3, 117.9, 51.7, 40.9, 29.6; IR ($CHCl_3$): ν = 1745 cm^{-1}; MS (ES): *m/z* (%):155 (100) [*M* + H]$^{+}$, 154 (9) [*M*]$^{+}$; elemental analysis calcd (%) for $C_7H_{10}N_2O_2$ (154.2): C 54.54, H 6.54, N 18.17; found C 54.65, H 6.51, N 18.11.

3-Allyl-oxazolidin-2-one 16d. From 200 mg (2.20 mmol) of **18d**, compound **16d** (161 mg, 58%) was obtained as a colorless oil after chromatography of the residue using hexanes/ethyl acetate (1:1) as eluent; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 5.72 (m, 1H), 5.21 (m, 2H), 4.26 (m, 2H), 3.81 (m, 2H), 3.48 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 158.4, 132.1, 118.7, 61.9, 47.1; IR ($CHCl_3$): ν = 1725 cm^{-1}; MS (ES): *m/z* (%):128 (100) [*M* + H]$^{+}$, 127 (11) [*M*]$^{+}$; elemental analysis calcd (%) for $C_6H_9NO_2$ (127.1): C 56.68, H 7.13, N 11.02; found C 56.58, H 7.16, N 11.06.

General Procedure for the Synthesis of Compounds 4. The corresponding alkoxyacetylchloride (38 mmol) in anhydrous dichloromethane (25 mL) was added dropwise via syringe to a solution of the corresponding imine (25 mmol) and Et_3N (75 mmol) in dichloromethane (160 mL), at 0 ^{o}C under argon. The resulting mixture was allowed to warm to room temperature, and was stirred for 16 h. The crude mixture was diluted with CH_2Cl_2 (100 mL) and washed with saturated $NaHCO_3$ (2 x 20 mL) and brine (40 mL). The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. Chromatography of the residue eluting with ethyl acetate/hexanes gave analytically pure compounds **4**. Spectroscopic and analytical data for some representative forms of **4** follow.

1-Allyl-4-(2,5-dimethoxyphenyl)-3-methoxy-azetidin-2-one (±)-4a. From 822 mg (4.01 mmol) of imine, compound (±)-**4a** (1.09 mg, 99%) was obtained as a pale yellow oil after chromatography of the residue using hexanes/ethyl acetate (1:1) as eluent; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.76 (m, 3H), 5.67 (m, 1H), 5.12 (d, *J* = 4.3 Hz, 1H), 5.05 (m, 2H), 4.65 (d, *J* = 4.5 Hz, 1H), 4.13 (dd, *J* = 14.4, 7.0 Hz, 1H), 3.69 and 3.72 (s, each 3H), 3.42 (dd, *J* = 14.4, 7.0 Hz, 1H), 3.08 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 167.3, 153.5, 151.7, 131.0, 123.4, 118.8, 114.2, 113.5, 111.3, 85.7, 59.1, 55.9, 55.7, 55.6, 42.9; IR ($CHCl_3$): ν = 1745 cm^{-1}; MS (ES): *m/z* (%):278 (100) [*M* + H]$^{+}$, 277 (9) [*M*]$^{+}$; elemental analysis calcd (%) for $C_{15}H_{19}NO_4$ (277.3): C 64.97, H 6.91, N 5.05; found C 64.84, H 6.95, N 5.03.

(+)-(3*R*,4*S*)-4-[(*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-3-methoxy-1-(2-propenyl)-2-azetidinone (+)-4b. From 4.0 g (23.60 mmol) of imine, compound (+)-**4b** (3.20 mg, 55%) was obtained as a colorless solid after chromatography of the residue using hexanes/ethyl acetate (2:1) as eluent; m.p. 60–61 ^{o}C (hexanes/ethyl acetate); $[\alpha]_D$ = +49.4 (*c* = 1.2 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 5.85 (m, 1H), 5.15 (m, 2H), 4.37 (d, *J* = 5.1 Hz, 1H), 4.35 (dd, *J* = 14.0, 7.0 Hz, 1H), 4.06 (dd, *J* = 8.8, 6.6 Hz, 1H), 3.65 (m, 3H), 3.46 (s, 3H), 1.27 and 1.35 (s, each 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 167.3, 131.5, 118.6, 109.6, 82.9, 77.2, 66.8, 59.8, 59.2, 43.7, 26.9, 25.2; IR (KBr): ν = 1750 cm^{-1}; MS (ES): *m/z* (%): 242 (100) [*M* + H]$^{+}$, 241 (12) [*M*]$^{+}$; elemental analysis calcd (%) for $C_{12}H_{19}NO_4$ (241.3): C 59.73, H 7.94, N 5.81; found C 59.84, H 7.90, N 5.84.

(+)-(3*R*,4*S*)-4-[(*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-3-(*tert*-butyldimethylsilanyloxy)-1-(2-propenyl)-2-azetidinone (+)-4c. Compound (+)-**4c** was obtained (92%) as a colorless oil after chromatography of the residue using hexanes/ethyl acetate (2:1) as eluent; $[\alpha]_D$ = +10.7 (*c* = 1.0 in $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$, 25 ^oC): δ = 5.60 (m, 1H), 5.03 (m, 2H), 4.61 (d, *J* = 4.9 Hz, 1H), 4.02 (m, 3H), 3.45 (m, 3H), 3.65 (m, 3H), 1.23 and 1.28 (s, each 3H), 0.77 (s, 9H), 0.08 and 0.03 (s, each 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^oC): δ = 167.8, 131.6, 118.4, 109.5, 77.8, 75.9, 66.9, 60.6, 43.7, 26.9, 25.7, 25.1, 18.0; IR ($CHCl_3$): ν = 1750 cm^{-1}; MS (ES): *m/z* (%):342 (100) $[M + H]^+$, 341 (18) $[M]^+$; elemental analysis calcd (%) for $C_{17}H_{31}NO_4Si$ (341.5): C 59.79, H 9.15, N 4.10; found C 59.67, H 9.19, N 4.12.

1-Allyl-4-furan-2-yl-3-methoxy-2-azetidinone (±)-4d. Compound (±)-**4d** was obtained (36%) as a colorless solid after chromatography of the residue using hexanes/ethyl acetate (3:1) as eluent; m.p. 86–87 ^oC (hexanes/ethyl acetate); ^1H NMR (300 MHz, $CDCl_3$, 25 ^oC): δ = 7.37 (m, 3H), 6.82 (m, 3H), 6.41 and 6.29 (d, *J* = 3.2 Hz, each 1H), 5.77 (m, 1H), 5.45 (d, *J* = 4.3 Hz, 1H), 5.65 (dd, *J* = 8.6, 1.3 Hz, 2H), 5.03 (d, *J* = 4.3 Hz, 1H), 4.12 and 3.50 (dd, *J* = 15.4, 7.0 Hz, each 2H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^oC): δ = 165.5, 157.6, 148.0, 143.8, 131.3, 129.9, 122.8, 119.8, 116.1, 111.3, 82.6, 56.0, 43.8; IR (KBr): ν = 1752 cm^{-1}; MS (ES): *m/z* (%):208 (100) $[M + H]^+$, 207 (11) $[M]^+$; elemental analysis calcd (%) for $C_{11}H_{13}NO_3$ (207.2): C 63.76, H 6.32, N 6.76; found C 63.88, H 6.35, N 6.72.

(+)-(3*R*,4*S*)-4-[(*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl]-3-phenoxy-1-(2-propenyl)-2-azetidinone (+)-4e. From 1.69 g (10.0 mmol) of imine, compound (+)-**4e** (2.24 mg, 74%) was obtained as a colorless solid after chromatography of the residue using hexanes/ethyl acetate (5:1) as eluent; m.p. 63–65 ^{o}C (hexanes/ethyl acetate); $[\alpha]_D$ = +100.9 (*c* = 1.1 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.30 (m, 2H), 7.02 (m, 3H), 5.75 (m, 1H), 5.20 (d, *J* = 5.2 Hz, 1H), 4.45 (m, 1H), 4.18 (m, 2H), 3.82 (m, 2H), 3.38 (dd, *J* = 8.8, 6.6 Hz, 1H), 1.37 and 1.44 (s, each 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 165.6, 157.3, 131.2, 129.6, 129.5, 122.5, 118.9, 109.7, 79.9, 77.1, 66.9, 59.8, 44.0, 26.8; IR (KBr): ν = 1755 cm^{-1}; MS (EI): *m/z* (%):304 (11) $[M + H]^+$, 303 (100) $[M]^+$; elemental analysis calcd (%) for $C_{17}H_{21}NO_4$ (303.3): C 67.31, H 6.98, N 4.62; found C 67.18, H 7.04, N 4.60.

Procedure for the Preparation of 4-Oxoazetidine-2-carbaldehyde (+)-7. Phenoxyacetylchloride (38 mmol) in anhydrous dichloromethane (25 mL) was added dropwise via syringe to a solution of the 2,3-*O*-(isopropylidene)-D-glyceraldehyde imine (25 mmol) and Et_3N (75 mmol) in dichloromethane (160 mL), at 0 ^{o}C under argon. The resulting mixture was allowed to warm to room temperature, and was stirred for 16h. The crude mixture was diluted with CH_2Cl_2 (100 mL) and washed with saturated $NaHCO_3$ (2 x 20 mL) and brine (40 mL). The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. Chromatography of the residue eluting with EtOAc/hexanes (1:2) give analytically pure (3*R*,4*S*)-4-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]-3-phenoxy-1-(*p*-methoxyphenyl)-2-azetidinone.

To a solution of the above acetonide β-lactam (15 mmol) in THF/water (1:1, 300 mL) was added solid *p*-TsOH·H_2O (18 mmol) in a single portion. The resulting clear solution was heated under reflux for 2h. The reaction mixture was allowed to cool to room temperature, and then was neutralized with solid $NaHCO_3$. The mixture was extracted with ethyl acetate (3 x 60 mL), the organic layer was dried ($MgSO_4$) and the solvent was removed under reduced pressure to give (3*R*,4*S*)-4-[(*S*)-1,2-dihydroxyethylen]-3-phenoxy-1-(*p*-methoxyphenyl)-2-azetidinone. Further purification was not necessary.

Saturated aqueous sodium hydrogen carbonate (1.5 mL) was added to a solution of the above diol (15.0 mmol) in dichloromethane (95 mL), maintaining the temperature below 25 ^oC. Solid sodium periodate (30.0 mmol) was added over a 10 min period with vigorous stirring and the reaction was allowed to proceed for 2h, while the temperature was maintained below 25 ^oC. The solid was removed by filtration, the filtrate was dried ($MgSO_4$) and the solvent was removed under reduced pressure to give 4.68 g (63% overall yield) of compound (+)-**7**. The crude product was used for next step without any further purification.

(+)-(3*R*,4*S*)-1-(*p*-Methoxyphenyl)-3-phenoxy-4-oxoazetidine-2-carbaldehyde (+)-7. Colorless solid; m.p. 127–129 ^oC (hexanes/ethyl acetate); $[\alpha]_D$ = +160.4 (*c* = 1.0 in $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$, 25 ^oC): δ = 9.08 (d, *J* = 3.7 Hz, 1H), 7.36 (m, 5H), 7.05 (m, 2H), 6.89 (m, 3H), 5.56 (d, *J* = 5.3 Hz, 1H), 4.72 (dd, *J* = 5.3, 3.7 Hz, 1H), 3.78 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^oC): δ = 197.7, 161.5, 157.3, 156.9, 130.4, 129.9, 123.3, 118.3, 115.8, 114.8,

81.7, 63.2, 55.7; IR (KBr): ν = 1765, 1750 cm^{-1}; MS (EI): *m/z* (%):297 (100) $[M]^+$; elemental analysis calcd (%) for $C_{17}H_{15}NO_4$ (297.3): C 68.68, H 5.09, N 4.71; found C 68.80, H 5.05, N 4.74.

Procedure for the Synthesis of C4,C4'-bis-β-Lactam (+)-8. A suspension of 4-oxoazetidine-2-carbaldehyde (+)-**7** (630 mg, 2.12 mmol), allyl amine (0.24 mL, 3.18 mmol), and $MgSO_4$ (2.03 g, 16.9 mmol) in anhydrous dichloromethane (30 mL) was stirred at room temperature overnight. Then, the mixture was filtered and the solvent was removed under reduced pressure. Further purification was not necessary, and the resulting imino β-lactam (712 mg, 100%) was used as such. Methoxyacetyl chloride (0.31 mL, 3.17 mmol) in anhydrous dichloromethane (5 mL) was added dropwise via syringe to a solution of the above imine and Et_3N (0.88 mL, 6.33 mmol) in dichloromethane (30 mL), at 0 ^oC under argon. The resulting mixture was allowed to warm to room temperature, and was stirred for 16 h. The crude mixture was diluted with CH_2Cl_2 (20 mL) and washed with saturated $NaHCO_3$ (2 x 5 mL) and brine (5 mL). The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. Chromatography of the residue eluting with ethyl acetate/hexanes (1:1) gave 766 mg (89%) of analytically pure compound (+)-**8** as a yellow oil.

C4,C4'-bis-2-azetidinone (+)-8. $[\alpha]_D$ = +67.0 (*c* = 0.4 in $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$, 25 ^oC): δ = 7.30 (m, 5H), 7.10 (m, 2H), 6.80 (m, 2H), 5.40 (m, 1H), 5.38 (d, *J* = 5.3 Hz, 1H), 4.99 (m, 1H), 4.62 (m, 2H), 4.46 (d, *J* = 5.2 Hz, 1H), 4.13 (dd, *J* = 7.7, 5.2 Hz, 1H), 3.91 (m, 1H), 3.73 and 3.40 (s, each 3H), 2.76 (dd, *J* = 15.2, 7.9 Hz, 1H); ^{13}C NMR (75

MHz, $CDCl_3$, 25 °C): δ = 168.3, 163.7, 157.4, 130.5, 130.0, 129.8, 122.8, 120.5, 119.9, 115.9, 114.6, 83.8, 80.3, 59.6, 57.5, 56.6, 55.6, 44.6; IR ($CHCl_3$): ν = 1762, 1754 cm^{-1}; MS (ES): *m/z* (%):409 (100) $[M + H]^+$, 408 (10) $[M]^+$; elemental analysis calcd (%) for $C_{23}H_{24}N_2O_5$ (408.4): C 67.63, H 5.92, N 6.86; found C 67.76, H 5.89, N 6.83.

Procedure for the Synthesis of Homopyroglutamic Derivative (–)-10. Sodium methoxide (386 mg, 7.16 mmol) was added in portions at RT to a solution of the C4,C4'-bis-2-azetidinone (+)-**8** (730 mg, 1.79 mmol) in methanol (40 mL). The reaction was stirred at room temperature for 70 h and then water was added (5 mL). The methanol was concentrated under reduced pressure, the aqueous residue was extracted with ethyl acetate (15 x 3 mL), the organic layer was dried over $MgSO_4$, and the solvent was removed under reduced pressure. Chromatography of the residue eluting with ethyl acetate/hexanes (1:2) gave 511 mg (65%) of analytically pure compound (–)-**10** as a yellow oil.

[1-Allyl-3-(*p*-methoxyphenylamino)-5-oxo-4-phenoxy-pyrrolidin-2-yl]-methoxy-acetic acid methyl ester (–)-10. $[\alpha]_D$ = –81.4 (*c* = 0.9 in $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.30 (m, 5H), 7.10 (m, 2H), 6.80 (m, 2H), 6.71 (m, 2H), 5.85 (m, 1H), 5.26 (m, 2H), 4.56 (m, 2H), 4.27 (m, 3H), 3.87 and 3.72 (s, each 3H), 3.60 (dd, *J* = 15.4, 7.6 Hz, 1H), 3.41 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 °C): δ = 169.6, 168.9, 157.5, 152.5, 140.3, 131.6, 129.4, 122.1, 118.5, 115.2, 115.1, 114.7, 82.4, 75.9, 58.8, 58.1, 56.9, 55.6, 51.9, 43.5; IR ($CHCl_3$): ν = 1742, 1725 cm^{-1}; MS (ES): *m/z* (%):441 (100) $[M + H]^+$, 440 (16) $[M]^+$; elemental analysis

calcd (%) for $C_{24}H_{28}N_2O_6$ (440.5): C 65.44, H 6.41, N 6.36; found C 65.57, H 6.45, N 6.33.

Procedure for the Synthesis of *N*-Allyl-bis-g-lactam (+)-11. A solution of homopyroglutamic derivative (–)-**10** (100 mg, 0.23 mmol) in toluene (10 mL) was heated for 3 h at reflux temperature under PTSA catalysis using a Dean-Stark apparatus. After cooling to RT, the solvent was removed under reduced pressure to give 92 mg (100%) of compound (+)-**11** as a yellow oil.

***N*-Allyl-bis-g-lactam (+)-11.** $[\alpha]_D$ = +42.0 (*c* = 0.6 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.30 (m, 2H), 7.10 (m, 2H), 6.78 (m, 5H), 5.85 (m, 1H), 5.75 (m, 1H), 5.22 (m, 2H), 4.67 (m, 2H), 4.25 (m, 2H), 3.88 (d, *J* = 6.8 Hz, 1H), 3.71 (m, 1H), 3.62 and 3.48 (s, each 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 169.4, 168.9, 158.1, 157.3, 130.8, 129.5, 128.9, 124.5, 122.6, 119.6, 116.7, 114.6, 80.3, 78.7, 61.3, 58.9, 55.5, 55.4, 44.5; IR ($CHCl_3$): ν = 1725, 1720 cm^{-1}; MS (ES): *m/z* (%):409 (100) $[M + H]^+$, 408 (15) $[M]^+$; elemental analysis calcd (%) for $C_{23}H_{24}N_2O_5$ (408.4): C 67.63, H 5.92, N 6.86; found C 67.49, H 5.88, N 6.90.

Spectroscopic and analytical data for some representative pure forms of enamide derivatives **2**, **5**, **14,** and **17** follow.

Preparation of Compound 2a. From 70 mg (0.56 mmol) of 1-allyl-pyrrolidin-2-one **1a** and after column chromatography eluting with hexanes/ethyl acetate (3:1), 36 mg (49%) of the less polar compound *E*-**2a** and 29 mg (41%) of the more polar compound, its *Z*-isomer were obtained.

Compound *E*-2a. Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.79 (dd, *J* = 14.4, 1.5 Hz, 1H), 4.89 (m, 1H), 3.42 (t, *J* = 7.1 Hz, 2H), 2.41 (dd, *J*= 8.4, 7.8 Hz, 2H), 1.98 (m, 2H), 1.65 (dd, *J* = 6.7, 1.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 172.0, 124.5, 106.8, 45.2, 31.2, 17.4, 15.2; IR ($CHCl_3$): ν = 1725 cm^{-1}; MS (EI): *m/z* (%): 126 (3) $[M + H]^+$, 125 (100) $[M]^+$; elemental analysis calcd (%) for $C_7H_{11}NO$ (125.2): C 67.17, H 8.86, N 11.19; found C 67.04, H 8.82, N 11.23.

Compound *Z*-2a. Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.31 (dd, *J* = 9.5, 1.7 Hz, 1H), 4.93 (m, 1H), 3.72 (t, *J* = 7.1 Hz, 2H), 2.35 (dd, *J*= 8.2, 7.8 Hz, 2H), 2.02 (m, 2H), 1.67 (dd, *J* = 7.3, 1.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 171.8, 124.6, 106.9, 45.3, 31.3, 17.6, 15.3; IR ($CHCl_3$): ν = 1725 cm^{-1}; MS (EI): *m/z* (%): 126 (5) $[M + H]^+$, 125 (100) $[M]^+$; elemental analysis calcd (%) for $C_7H_{11}NO$ (125.2): C 67.17, H 8.86, N 11.19; found C 67.30, H 8.90, N 11.15.

Preparation of Compound (+)-2b. From 190 mg (0.96 mmol) of (*S*)-1-allyl-5-oxo-pyrrolidine-2-carboxylic acid ethyl ester (+)-**1b** and after column chromatography eluting with hexanes/ethyl acetate (3:1), 28 mg (15%) of the less polar compound *E*-(+)-**2b** and 114 mg (60%) of the more polar compound, its *Z*-isomer were obtained.

Compound *Z*-(+)-2b. Yellow oil; $[\alpha]_D$ = +65.0 (*c* = 0.6 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.76 (dd, *J*= 14.6, 1.5 Hz, 1H), 4.80 (m, 1H), 4.17 (m, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.31 (m, 4H), 1.62 (dd, *J*= 6.6, 1.5 Hz, 3H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ =

172.6, 171.4, 123.4, 107.0, 61.4, 58.5, 29.6, 23.2, 15.1, 13.9; IR ($CHCl_3$): ν = 1724, 1708 cm^{-1}; MS (ES): *m/z* (%): 198 (100) [*M* + H]$^+$, 197 (11) [*M*]$^+$; elemental analysis calcd (%) for $C_{10}H_{15}NO_3$ (197.2): C 60.90, H 7.67, N 7.10; found C 61.02, H 7.71, N 7.14.

Compound *E*-(+)-2b. Yellow oil; $[\alpha]_D$ = +27.0 (*c* = 0.8 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.03 (d, *J*= 7.2 Hz, 1H), 5.20 (m, 1H), 4.42 (m, 1H), 4.15 (q, *J* = 7.1 Hz, 2H), 2.27 (m, 4H), 1.61 (dd, *J*= 7.2, 0.9 Hz, 3H), 1.18 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 174.2, 171.6, 123.4, 118.5, 61.4, 60.8, 58.9, 29.4, 23.2, 13.9; IR ($CHCl_3$): ν = 1724, 1708 cm^{-1}; MS (ES): *m/z* (%): 198 (100) [*M* + H]$^+$, 197 (7) [*M*]$^+$; elemental analysis calcd (%) for $C_{10}H_{15}NO_3$ (197.2): C 60.90, H 7.67, N 7.10; found C 60.77, H 7.70, N 7.07.

Preparation of Compound 2c. From 280 mg (2.01 mmol) of 1-allyl-piperidin-2-one **1c** and after column chromatography eluting with hexanes/ethyl acetate (3:1), 108 mg (38%) of the less polar compound *E*-**2c** and 92 mg (33%) of the more polar compound, its *Z*-isomer were obtained.

Compound *E*-2c. Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.27 (dd, *J* = 14.6, 1.5 Hz, 1H), 4.98 (m, 1H), 3.31 (t, *J* = 6.2 Hz, 2H), 2.39 (t, *J*= 6.2 Hz, 2H), 1.75 (m, 4H), 1.68 (dd, *J* = 6.6, 1.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 167.9, 127.4, 105.8, 45.0, 32.7, 22.6, 20.5, 15.3; IR ($CHCl_3$): ν = 1655 cm^{-1}; MS (ES): *m/z* (%): 140 (100) [*M* + H]$^+$, 139 (11) [*M*]$^+$; elemental analysis calcd (%) for $C_8H_{13}NO$ (139.2): C 69.03, H 9.41, N 10.06; found C 68.91, H 9.38, N 10.02.

Compound *Z*-2c. Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.16 (d, *J* = 8.5 Hz, 1H), 5.30 (m, 1H), 3.37 (m, 2H), 2.37 (m, 2H), 1.76 (m, 4H), 1.57 (dd, *J* = 6.6, 1.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 169.7, 129.1, 119.7, 49.6, 32.3, 23.1, 21.4, 15.9; IR ($CHCl_3$): ν = 1653 cm^{-1}; MS (ES): *m/z* (%): 140 (100) $[M + H]^+$, 139 (9) $[M]^+$; elemental analysis calcd (%) for $C_8H_{13}NO$ (139.2): C 69.03, H 9.41, N 10.06; found C 68.90, H 9.44, N 10.11.

Compound (±)-5a. From 250 mg (0.90 mmol) of 1-allyl-β-lactam (±)-**4a** and after column chromatography eluting with hexanes/ethyl acetate (3:1), 137 mg (62%) of compound (±)-**5a** was obtained as a mixture of isomers *E/Z* (76:24); colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.75 (m, 3H), 6.42 (dd, *J* = 14.3, 1.7 Hz, 0.8H), 6.18 (dd, *J* = 9.3, 1.6 Hz, 0.2H), 5.44 (d, *J* = 4.7 Hz, 0.2H), 5.21 (d, *J* = 4.8 Hz, 0.8 Hz), 4.82 (m, 1H), 4.69 (d, *J* = 4.7 Hz, 0.2H), 4.60 (d, *J* = 4.8 Hz, 0.8H), 3.72 (s, 3H), 3.67 (s, 3H), 3.12 (s, 3H), 1.55 (dd, *J* = 6.8, 1.6 Hz, 2.4H), 1.41 (dd, *J* = 7.3, 1.6 Hz, 0.6H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 165.0 (m), 163.6 (M), 153.7 (m), 153.6 (M), 151.4 (m), 151.1 (M), 124.1 (m), 122.5 (m), 120.9 (M), 119.5 (m), 114.3 (M), 114.0 (m), 113.7 (m), 113.6 (M), 111.3 (m), 111.2 (M), 110.3 (M), 86.1 (m), 85.1 (M), 60.4 (m), 59.1 (m), 58.6 (M), 58.5 (M), 56.9 (M), 55.9 (M), 55.7 (M), 21.0 (m), 14.9 (M), 14.2 (m), 12.5 (m); IR ($CHCl_3$): ν = 1745 cm^{-1}; MS (ES): *m/z* (%):278 (100) $[M + H]^+$, 277 (7) $[M]^+$; elemental analysis calcd (%) for $C_{15}H_{19}NO_4$ (277.3): C 64.97, H 6.91, N 5.05; found C 65.09, H 6.87, N 5.02.

Compound (+)-5b. From 50 mg (0.21 mmol) of 1-allyl-β-lactam (+)-**4b** and after column chromatography eluting with hexanes/ethyl acetate (2:1), 34 mg (77%) of compound (+)-**5b** was obtained as a mixture of isomers *E/Z* (80:20); $[\alpha]_D$ = +84.0 (*c* = 1.0 in $CHCl_3$); colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.31 (dd, *J* = 14.3, 1.6 Hz, 0.8H), 5.90 (dd, *J* = 8.8, 1.7 Hz, 0.2H), 5.71 (dd, *J* = 14.3, 6.9 Hz, 0.8H), 5.12 (m, 0.2H), 4.37 (m, 1H), 4.14 (m, 2H), 3.78 (m, 1H), 3.60 (m, 1H), 3.50 (s, 0.6H), 3.48 (s, 2.4H), 1.69 (dd, *J* = 7.1, 1.7 Hz, 0.6H), 1.63 (dd, *J* = 6.8, 1.6 Hz, 2.4H), 1.41 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 161.3 (m), 160.6 (M), 121.8 (M), 118.0 (m), 112.2 (M), 111.7 (m), 82.5 (M + m), 76.4 (M + m), 66.8 (M + m), 61.8 (M), 61.7 (m), 59.2 (M), 58.8 (m), 56.7 (M), 26.6 (m), 25.0 (M + m), 15.3 (M + m), 10.5 (M + m); IR ($CHCl_3$): ν = 1748 cm^{-1}; MS (ES): *m/z* (%): 242 (100) $[M + H]^+$, 241 (9) $[M]^+$; elemental analysis calcd (%) for $C_{12}H_{19}NO_4$ (241.3): C 59.73, H 7.94, N 5.81; found C 59.61, H 7.98, N 5.85.

Compound (+)-5c. From 431 mg (1.26 mmol) of 1-allyl-β-lactam (+)-**4c** and after column chromatography eluting with hexanes/ethyl acetate (10:1), 122 mg (79%) of compound (+)-**5c** was obtained as a mixture of isomers *E/Z* (88:12); colorless oil; $[\alpha]_D$ = +10.7 (*c* = 1.0 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.20 (m, 0.8H), 5.83 (m, 0.2H), 5.60 (m, 0.8H), 5.11 (m, 0.2H), 4.61 (d, *J* = 5.1 Hz, 0.2H), 4.60 (d, *J* = 5.2 Hz, 0.8H), 4.07 (m, 3H), 3.54 (m, 3H), 1.62 (dd, *J* = 7.1, 1.7 Hz, 0.6H), 1.51 (dd, *J* = 6.8, 1.5 Hz, 2.4H), 1.30 (s, 3H), 1.18 (s, 3H), 0.76 (s, 9H), 0.03 (s, 3H), 0.01 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 165.0 (M + m),

112.0 (M + m), 111.8 (M + m), 109.5 (M + m), 77.1 (M + m), 75.4 (M + m), 67.0 (M + m), 62.7 (M + m), 26.9 (M + m), 25.6 (M + m), 24.9 (M + m), 18.1 (M + m), 15.4 (M + m); IR ($CHCl_3$): ν = 1750 cm^{-1}; MS (ES): *m/z* (%):342 (100) [*M* + H]$^+$, 341 (9) [*M*]$^+$; elemental analysis calcd (%) for $C_{17}H_{31}NO_4Si$ (341.5): C 59.79, H 9.15, N 4.10; found C 59.91, H 9.20, N 4.09.

Enamide of *N*-Allyl-b-lactam (+)-8. From 100 mg (0.24 mmol) of *N*-allyl-bis-β-lactam (+)-**8** and after column chromatography eluting with hexanes/ethyl acetate (3:1), 90 mg (90%) of the corresponding enamide was obtained as a mixture of isomers *E/Z* (63:37); colorless oil; $[\alpha]_D$ = +35.0 (*c* = 0.6 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.01 (m, 9H), 5.80 (m, 0.6H), 5.42 (m, 1H), 5.36 (m, 0.4H), 5.04 (m, 0.6H), 4.80 (m, 0.4H), 4.67 (m, 1H), 4.47 (m, 1H), 4.32 (m, 1H), 3.70 (s, 3H), 3.40 (s, 3H), 1.36 (dd, *J* = 7.0, 1.5 Hz, 1.2H), 1.23 (dd, *J* = 7.0, 1.5 Hz, 1.8H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 165.1 (m), 164.7 (M), 163.4 (M + m), 157.2 (M + m), 156.9 (M), 156.9 (m), 130.1 (M), 129.7 (M + m), 122.7 (m), 121.8 (M + m), 121.4 (M + m), 120.0 (M), 119.9 (M + m), 119.6 (m), 117.9 (M + m), 115.7 (M + m), 114.1 (M + m), 113.9 (M), 111.9 (m), 83.4 (m), 83.3 (M), 80.1 (m), 80.0 (M), 59.6 (M), 59.5 (m), 58.7 (M), 57.2 (M), 56.8 (M), 56.7 (m), 55.4 (M + m), 14.7 (M), 13.9 (m); IR ($CHCl_3$): ν = 1762, 1754 cm^{-1}; MS (ES): *m/z* (%):409 (100) [*M* + H]$^+$, 408 (15) [*M*]$^+$; elemental analysis calcd (%) for $C_{23}H_{24}N_2O_5$ (408.4): C 67.63, H 5.92, N 6.86; found C 67.51, H 5.95, N 6.90.

Enamide of *N*-Allyl-g-lactam (+)-11. From 70 mg (0.17 mmol) of *N*-allyl-bis-γ-lactam (+)-**11** and after column

chromatography eluting with hexanes/ethyl acetate (2:1), 57 mg (81%) of the corresponding *E*-enamide was obtained as a yellow oil; $[\alpha]_D$ = +67.1 (*c* = 0.8 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.28 (m, 4H), 6.80 (m, 6H), 5.30 (m, 1H), 4.76 (dd, *J* = 8.0, 2.6 Hz, 1H), 4.65 (d, *J* = 2.6 Hz, 1H), 4.43 (dd, *J* = 8.1, 2.8 Hz, 1H), 3.88 (d, *J* = 2.8 Hz, 1H), 3.68 and 3.60 (s, each 3H), 1.76 (dd, *J* = 6.7, 1.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 169.5, 166.2, 158.1, 157.3, 129.4, 128.9, 124.3, 122.7, 122.6, 116.7, 114.6, 111.4, 80.0, 79.3, 61.0, 59.2, 58.3, 55.5, 15.6; IR ($CHCl_3$): ν = 1724, 1721 cm^{-1}; MS (ES): *m/z* (%): 409 (100) $[M + H]^+$, 408 (11) $[M]^+$; elemental analysis calcd (%) for $C_{23}H_{24}N_2O_5$ (408.4): C 67.63, H 5.92, N 6.86; found C 67.76, H 5.89, N 6.94.

Compound *E*-14b. From 40 mg (0.27 mmol) of 1-allyl-acetamide **13b** and after column chromatography eluting with hexanes/ethyl acetate (4:1), 31 mg (78%) of compound *E*-**14b** was obtained as a yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.71 (m, 5H), 5.93 (dq, *J* = 7.8, 1.8 Hz, 1H), 5.43 (m, 1H), 4.61 (s, 2H), 1.98 (s, 3H), 1.36 (dd, *J* = 6.9, 1.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 169.1, 137.1, 129.0, 128.5 (2C), 128.1 (2C), 127.1, 126.2, 49.9, 21.7, 12.0; IR ($CHCl_3$): ν = 1651 cm^{-1}; MS (EI): *m/z* (%):189 (100) $[M]^+$; elemental analysis calcd (%) for $C_{12}H_{15}NO$ (189.2): C 76.16, H 7.99, N 7.40; found C 76.03, H 7.95, N 7.36.

Compound *E*-17a. From 120 mg (0.86 mmol) of 1-allyl-pyrrolidine-2,5-dione **16a** and after column chromatography eluting with hexanes/ethyl acetate (3:1), 100 mg (83%) of compound *E*-**17a** was obtained as a colorless oil; ^{1}H NMR (300

MHz, $CDCl_3$, 25 ^oC): δ = 6.48 (m, 2H), 2.66 (s, 4H), 1.73 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^oC): δ = 175.5, 120.1, 118.8, 28.2, 27.8; IR ($CHCl_3$): ν = 1748 cm^{-1}; MS (ES): *m/z* (%):140 (100) $[M + H]^+$, 139 (7) $[M]^+$; elemental analysis calcd (%) for $C_7H_9NO_2$ (139.1): C 60.42, H 6.52, N 10.07; found C 60.30, H 6.56, N 10.11.

Compound *E*-17c. From 120 mg (0.78 mmol) of 3-allyl-1-methyl-imidazolidine-2,4-dione **16c** and after column chromatography eluting with hexanes/ethyl acetate (1:1), 78 mg (65%) of compound *E*-**17c** was obtained as a colorless oil; ^1H NMR (300 MHz, $CDCl_3$, 25 ^oC): δ = 6.36 (m, 2H), 3.78 (s, 2H), 2.90 (s, 3H), 1.70 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^oC): δ = 171.3, 165.3, 118.9, 117.8, 60.5, 29.7, 21.1; IR ($CHCl_3$): ν = 1742 cm^{-1}; MS (ES): *m/z* (%):155 (100) $[M + H]^+$, 154 (5) $[M]^+$; elemental analysis calcd (%) for $C_7H_{10}N_2O_2$ (154.2): C 54.54, H 6.54, N 18.17; found C 54.67, H 6.50, N 18.22.

Compound 17d. From 50 mg (0.38 mmol) of 3-allyl-oxazolidin-2-one **16d** and after column chromatography eluting with hexanes/ethyl acetate (3:1), 44 mg (87%) of the corresponding enamide was obtained as a mixture of isomers *E/Z* (90:10); colorless oil; ^1H NMR (300 MHz, $CDCl_3$, 25 ^oC): δ = 6.54 (dd, *J* = 14.3, 1.5 Hz, 0.9H), 6.13 (dd, *J* = 9.4, 1.7 Hz, 0.1H), 4.88 (m, 0.1H), 4.71 (m, 0.9 Hz), 4.35 (m, 2H), 3.61 (m, 2H), 1.62 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^oC): δ = 167.8 (M + m), 124.6 (M + m), 105.7 (M + m), 62.0 (M + m), 42.6 (M + m), 14.8 (M + m); IR ($CHCl_3$): ν = 1722 cm^{-1}; MS (ES): *m/z* (%):128 (100) $[M + H]^+$, 127 (7) $[M]^+$; elemental

analysis calcd (%) for $C_6H_9NO_2$ (127.1): C 56.68, H 7.13, N 11.02; found C 56.80, H 7.09, N 11.06.

Compound 17e. From 200 mg (1.15 mmol) of 3-allyl-2-thioxo-4-thiazolidinone **16e** and after column chromatography eluting with hexanes/ethyl acetate (10:1), 92 mg (46%) of the corresponding enamide was obtained as a mixture of isomers *E/Z* (70:30); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.00 (m, 2H), 4.07 (s, 2H), 1.92 (d, *J* = 7.1 Hz, 0.9H), 1.48 (d, *J* = 5.1 Hz, 2.1H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 199.8 (M + m), 171.7 (M + m), 129.9 (M + m), 121.3 (M + m), 35.8 (M + m), 13.9 (M + m); IR ($CHCl_3$): ν = 1720 cm^{-1}; MS (ES): *m/z* (%):174 (100) $[M + H]^+$, 173 (12) $[M]^+$; elemental analysis calcd (%) for $C_6H_7NS_2O$ (173.3): C 41.59, H 4.07, N 8.08; found C 41.73, H 4.03, N 8.14.

Spectroscopic and analytical data for some representative pure forms of free *NH*-amides, lactams, imides and derivatives **3**, **6**, **9**, **12**, **15,** and **18** follow.

***NH*-γ-Lactam 3a.** From 40 mg (0.30 mmol) of enamide derivative **2a**, 22 mg (87%) of the commercially available pyrrolidin-2-one **3a** was obtained; elemental analysis calcd (%) for C_4H_7NO (85.1): C 56.45, H 8.29, N 16.46; found C 56.57, H 8.25, N 16.41.

***NH*-γ-Lactam (+)-3b.** From 120 mg (0.61 mmol) of enamide derivative (+)-**2b**, 70 mg (73%) of the commercially available (*S*)-5-oxo-pyrrolidine-2-carboxylic acid ethyl ester (+)-**3b** was obtained; $[\alpha]_D$ = +4.3 (*c* = 1.0 in EtOH); elemental analysis calcd (%) for $C_7H_{11}NO_3$ (157.1): C 53.49, H 7.05, N 8.91; found C 53.62, H 7.02, N 8.95.

***NH*-d-Lactam 3c.** From 200 mg (1.44 mmol) of enamide derivative **2c**, 100 mg (70%) of the commercially available piperidin-2-one **3c** was obtained; elemental analysis calcd (%) for C_5H_9NO (99.1): C 60.58, H 9.15, N 14.13; found C 60.45, H 9.11, N 14.18.

***NH*-e-Lactam 3d.** From 50 mg (0.326 mmol) of enamide derivative **2d**, 24 mg (65%) of the commercially available piperidin-2-one **3d** was obtained; elemental analysis calcd (%) for $C_6H_{11}NO$ (113.2): C 63.68, H 9.80, N 12.38; found C 63.82, H 9.76, N 12.33.

***NH*-b-Lactam (+)-6b.** From 80 mg (0.33 mmol) of enamide derivative (+)-**5b**, compound (+)-**6b** (57 mg, 85%) was obtained as a colorless oil; $[\alpha]_D$ = +34.1 (*c* = 1.5 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 6.24 (m, 1H), 4.44 (d, *J* = 5.1 Hz, 1H), 4.10 (m, 2H), 3.62 (dd, *J* = 8.8, 5.1 Hz, 1H), 3.57 (dd, *J* = 8.5, 5.5 Hz, 1H), 3.49 (s, 3H), 1.36 and 1.28 (s, each 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 166.3, 112.2, 82.4, 66.8, 61.7, 59.2, 56.9, 26.7, 25.1; IR ($CHCl_3$): ν = 3412, 1745 cm^{-1}; MS (ES): *m/z* (%): 202 (100) $[M + H]^+$, 201 (8) $[M]^+$; elemental analysis calcd (%) for $C_9H_{15}NO_4$ (201.2): C 53.72, H 7.51, N 6.96; found C 53.84, H 7.54, N 6.93.

***NH*-b-Lactam (±)-6d.** From 60 mg (0.22 mmol) of enamide derivative (±)-**5d**, compound (±)-**6d** (29 mg, 57%) was obtained as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.00 (m, 6H), 6.36 (m, 1H), 6.22 (m, 1H), 5.38 and 5.00 (d, *J* = 4.2 Hz, each 1H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 164.1, 142.1, 129.6, 129.2, 122.0, 115.5, 114.4, 110.5, 109.4, 66.0, 53.1; IR ($CHCl_3$): ν = 3406, 1750 cm^{-1}; MS (ES): *m/z* (%):230 (100) $[M + H]^+$, 229 (9) $[M]^+$; elemental analysis

calcd (%) for $C_{13}H_{11}NO_3$ (229.2): C 68.11, H 4.84, N 6.11; found C 67.98, H 4.87, N 6.15.

***NH*-b-Lactam (+)-6e.** From 70 mg (0.23 mmol) of enamide derivative (+)-**5e**, compound (+)-**6e** (47 mg, 78%) was obtained as a colorless oil; $[\alpha]_D$ = +36.0 (*c* = 0.5 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.27 (m, 2H), 7.01 (m, 3H), 6.47 (s, 1H), 5.23 (d, *J* = 5.1 Hz, 1H), 4.08 (m, 2H), 3.83 (dd, *J* = 8.9, 5.1 Hz, 1H), 3.61 (dd, *J* = 8.8, 5.8 Hz, 1H), 1.38 and 1.23 (s, each 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 163.2, 129.7, 122.6, 115.6, 109.1, 81.0, 76.7, 66.6, 56.9, 53.8, 26.8, 25.0; IR ($CHCl_3$): ν = 3410, 1748 cm^{-1}; MS (ES): *m/z* (%):264 (100) [*M* + H]$^+$, 263 (7) [*M*]$^+$; elemental analysis calcd (%) for $C_{14}H_{17}NO_4$ (263.3): C 63.87, H 6.51, N 5.32; found C 64.01, H 6.55, N 5.28.

***NH*-bis-b-Lactam (+)-9.** From 50 mg (0.122 mmol) of the corresponding enamide derivative, compound (+)-**9** (35 mg, 80%) was obtained as a orange oil; $[\alpha]_D$ = +102.9 (*c* = 1.2 in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.23 (m, 5H), 7.01 (m, 2H), 6.72 (m, 2H), 5.90 (s, 1H), 5.39 (d, *J* = 5.2 Hz, 1H), 4.55 (m, 2H), 4.12 (dd, *J* = 8.8, 3.8 Hz, 1H), 3.70 and 3.32 (s, each 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 167.2, 164.1, 157.3, 157.1, 129.9, 129.8, 122.8, 119.9, 116.1, 114.8, 86.0, 80.8, 60.4, 58.1, 55.7, 55.2; IR ($CHCl_3$): ν = 3395, 1760, 1755 cm^{-1}; MS (ES): *m/z* (%):369 (100) [*M* + H]$^+$, 368 (11) [*M*]$^+$; elemental analysis calcd (%) for $C_{20}H_{20}N_2O_5$ (368.4): C 65.21, H 5.47, N 7.60; found C 65.34, H 5.44, N 7.64.

***NH*-Acetamide 15a.** From 20 mg (0.10 mmol) of the enamide derivative **14a**, compound **15a** (19 mg, 97%) was obtained as a

colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.30 (m, 2H), 6.77 (m, 2H), 5.68 (s, 1H), 3.72 (s, 3H), 2.08 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 166.3, 121.9, 121.8, 114.1, 114.0, 55.4, 31.2; IR ($CHCl_3$): ν = 1654 cm^{-1}; MS (EI): *m/z* (%):165 (100) $[M]^{+}$; elemental analysis calcd (%) for $C_9H_{11}NO_2$ (165.2): C 65.44, H 6.71, N 8.48; found C 65.57, H 6.67, N 8.52.

***NH*-Acetamide 15b.** From 16 mg (0.089 mmol) of the enamide derivative **14b**, compound **15b** (9 mg, 69%) was obtained as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.21 (m, 5H), 5.60 (s, 1H), 4.41 (m, 2H), 2.00 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, 25 ^{o}C): δ = 172.3, 128.7, 127.9, 127.8, 127.5, 43.7, 29.2; IR ($CHCl_3$): ν = 1650 cm^{-1}; MS (EI): *m/z* (%):149 (100) $[M]^{+}$; elemental analysis calcd (%) for $C_9H_{11}NO$ (149.2): C 72.46, H 7.43, N 9.39; found C 72.61, H 7.39, N 9.43.

***NH*-imide 18a.** From 65 mg (0.467 mmol) of enamide derivative **17a**, 33 mg (72%) of the commercially available succinimide **18a** was obtained; elemental analysis calcd (%) for $C_4H_5NO_2$ (99.1): C 48.48, H 5.09, N 14.14; found C 48.58, H 5.06, N 14.10.

***NH*-pyrazolidone 18b.** From 61 mg (0.282 mmol) of enamide derivative **17b**, 35 mg (71%) of the commercially available pyrazolidone **18b** was obtained; elemental analysis calcd (%) for $C_{10}H_{12}N_2O$ (176.2): C 68.16, H 6.86, N 15.90; found C 68.28, H 6.83, N 15.85.

***NH*-hydantoin 18c.** From 73 mg (0.474 mmol) of enamide derivative **17c**, 37 mg (67%) of the commercially available hydantoin **18c** was obtained; elemental analysis calcd (%) for

$C_4H_6N_2O_2$ (114.1): C 42.10, H 5.30, N 24.55; found C 42.20, H 5.34, N 24.61.

***NH*-oxazolidinone 18d.** From 30 mg (0.229 mmol) of enamide derivative **17d**, 14 mg (61%) of the commercially available oxazolidinone **18d** was obtained; elemental analysis calcd (%) for $C_3H_5NO_2$ (87.1): C 41.38, H 5.79, N 16.09; found C 41.47, H 5.82, N 16.04.