Triflic Anhydride Mediated Cyclization of 5-Hydroxy Substituted Pyrrolidinones for the Preparation of α -Trifluoromethyl-sulfonamido Furans

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Supporting Information Available: ^1H and ^{13}C -NMR spectra for new compounds lacking elemental analyses together with an Ortep drawing for compounds **19** and **36**. This material is available free of charge *via* the Internet at <http://pubs.acs.org>.

General Experimental Section Paragraph

Melting points are uncorrected. Mass spectra were determined at an ionizing voltage of 70eV. Unless otherwise noted, all reactions were performed in flame dried glassware under an atmosphere of dry argon. Solutions were evaporated under reduced pressure with a rotary evaporator and the residue was chromatographed on a silica gel column using a 5% ethyl acetate/hexane mixture as the eluent unless specified otherwise. All solids were recrystallized from 3% ethyl acetate/hexane for analytical data.

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Ortep of 1-Benzyl-5-(3,3,3-trifluoro-2-oxo-propylidene)-pyrrolidin-2-one (19)

Ortep of 3-[5-Methyl-furan-2-yl)-trifluoromethanesulfonylamino]propionic Acid
(36)

Supplemental Experimental Section

5-Hydroxy-5-methyl-1-phenethyl-pyrrolidin-2-one (9) was prepared in 61% yield from lactone **5** and phenethylamine: IR (neat) 1682, 1496, 1455, 1359, and 1142 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 1.38 (s, 3H), 2.00-2.12 (m, 2H), 2.27 (ddd, 1H, J = 14.6, 8.9, and 6.0 Hz), 2.50 (ddd, 1H, J = 16.8, 9.2, and 7.3), 2.81 (ddd, 1H, J = 13.3, 9.8, and 5.8 Hz), 2.94 (ddd, 1H, J = 13.3, 10.2, and 6.0 Hz), 3.31-3.50 (m, 2H), and 7.16-7.28 (m, 5H); ^{13}C -NMR (75 MHz, CDCl_3) δ 26.4, 29.3, 35.2, 35.3, 41.1, 90.2, 126.6, 128.6, 129.0, 139.4, and 175.0; HRMS Calcd. for $\text{C}_{13}\text{H}_{17}\text{NO}_2$: 219.1259. Found: 219.1258.

1-[2-(3,4-Dimethoxyphenyl)ethyl]-5-hydroxy-5-methyl-pyrrolidin-2-one (10) was prepared in 80% yield from lactone **5** and phenethylamine: ^1H -NMR (300 MHz, CDCl_3) δ 1.39 (s, 3H), 1.95-2.13 (m, 2H), 2.20-2.38 (m, 1H), 2.40-2.56 (m, 1H), 2.67-2.95 (m, 3H), 3.29-3.50 (m, 2H), 3.81 (s, 3H), 3.83 (s, 3H), and 6.68-6.80 (m, 3H). The spectral data of this compound is identical to that reported in the literature.¹

3-(2-Hydroxy-2-methyl-5-oxo-pyrrolidin-1-yl)-propionic Acid *tert*-Butyl Ester (11) was prepared in 89% from lactone **5** and β -alanine *tert*-butyl ester; IR (neat) 1726, 1671, 1417, and 1157 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 1.37 (s, 9H), 1.46 (s, 3H), 1.98-2.09 (m, 2H), 2.22 (ddd, 1H, J = 13.4, 7.9, and 5.4 Hz), 2.38-2.52 (m, 2H), 2.69 (dtd, 1H, J = 11.1, 7.6, and 1.9 Hz), 3.30 (dt, 1H, J = 14.3 and 7.3 Hz), 3.54 (dt, 1H, J = 14.3 and 7.3 Hz), and 4.66 (brs, 1H); ^{13}C -NMR (75 MHz, CDCl_3) δ 26.4, 28.1, 29.3, 34.7, 35.3, 81.6, 90.0, 173.0 and 170.5; HRMS Calcd. for $\text{C}_{12}\text{H}_{21}\text{NO}_4$ ($\text{M}-\text{H}^+$): 242.1392. Found: 242.1392.

4-Oxo-pentanoic Acid But-3-enyl-amide (14) was prepared in 63% yield from lactone **5** and 4-butenylamine²: IR (neat) 1716, 1650, 1437, 1360, and 1164 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 2.18 (s, 3H), 2.24 (qt, 2H, J = 6.7 and 1.3 Hz), 2.40 (t, 2H, J = 6.7 Hz), 2.79 (t, 2H, J = 6.7 Hz), 3.30 (q, 2H, J = 6.7 Hz), 5.05-5.12 (m, 2H), 5.69-5.80 (m, 1H), and 5.70 (brs, 1H); ^{13}C -NMR (100 MHz, CDCl_3) δ 29.9, 30.0, 33.8, 38.6, 38.7, 117.2, 135.4, 172.0, and 208.0; HRMS Calcd. for $\text{C}_9\text{H}_{15}\text{NO}_2$: 169.1103. Found: 169.1108.

4-Oxo-pentanoic Acid (3-Methyl-but-3-enyl)amide (15) was prepared in 82% yield from lactone **5** and 3-methyl-but-3-enylamine: IR (neat) 1715, 1653, 1545, 1360, and 1164 cm^{-1} ;

$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.62 (s, 3H), 2.08 (s, 3H), 2.10 (t, 2H, $J = 7.0$ Hz), 2.31 (t, 2H, $J = 6.7$ Hz), 2.69 (t, 2H, $J = 6.7$ Hz), 3.24 (q, 2H, $J = 7.0$ Hz), 4.62 (s, 1H), 4.70 (s, 1H), and 5.98 (brs, 1H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 22.2, 30.0, 30.1, 37.4, 37.5, 38.8, 112.3, 142.8, 172.0, and 208.0; HRMS Calcd. for $\text{C}_{10}\text{H}_{17}\text{NO}_2$: 183.1259. Found: 183.1264.

4-Oxo-pentanoic Acid Phenethylamide (16) was prepared in 91% yield from lactone **5** and phenethylamine: $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 2.17 (s, 3H), 2.37 (t, 2H, $J = 6.5$ Hz); 2.54-2.83 (m, 4H), 3.49 (q, 2H, $J = 6.7$ Hz); 5.64 (brs, 1H), and 7.18-7.34 (m, 5H). The spectral data of this compound is identical to that reported in the literature.¹

C,C,C-Trifluoro-N-(5-methyl-furan-2-yl)-N-phenethylmethanesulfonamide (33) was prepared in 79% yield from **9** (or in 84% yield from **16**): IR (neat) 1617, 1406, 1229, 1190, 1141, and 1068 cm^{-1} ; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 2.29 (s, 3H), 2.93 (dd, 2H, $J = 8.0$ and 8.0 Hz), 3.93 (dd, 2H, $J = 8.0$ and 8.0 Hz), 6.00 (dd, 1H, $J = 3.2$ and 1.2 Hz), 6.19 (d, 1H, $J = 3.2$ Hz), and 7.15-7.34 (m, 5H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 13.9, 35.5, 54.1, 107.5, 109.7, 120.1 (q, $J = 321.5$ Hz), 127.1, 128.8, 129.0, 137.0, 140.2, and 152.0; Anal. Calcd. for $\text{C}_{14}\text{H}_{14}\text{F}_3\text{NO}_3\text{S}$: C, 50.45; H, 4.23; N, 4.20. Found: C, 50.66; H, 4.34; N, 4.33.

3-[(5-Methyl-furan-2-yl)-trifluoromethanesulfonylamino]propionic Acid *tert*-Butyl Ester (35) was prepared in 72% yield from **11**: IR (neat) 1730, 1618, 1559, 1407, 1370, 1190, 1089, and 1060 cm^{-1} ; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.41 (s, 9H), 2.24 (s, 3H), 2.53 (t, 2H, $J = 7.3$ Hz), 3.94 (t, 2H, $J = 7.3$ Hz), 5.96 (dd, 1H, $J = 3.2$ and 1.0 Hz), and 6.19 (d, 1H, $J = 3.2$ Hz); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 13.9, 28.1, 35.0, 48.6, 81.7, 107.5, 110.1, 120.0 (q, $J = 321.7$ Hz), 139.6, 152.2, and 169.3; Anal. Calcd. for $\text{C}_{13}\text{H}_{18}\text{F}_3\text{NO}_5\text{S}$: C, 43.69; H, 5.08; N, 3.92. Found: C, 43.87; H, 5.07; N, 4.02.

3-[(5-Methyl-furan-2-yl)-trifluoromethanesulfonylamino]propionic Acid (36). To a solution of furan **35** (0.18 g, 0.5 mmol) in 1 mL of CH_2Cl_2 was added CF_3COOH (0.2 mL) and the mixture was stirred at room temperature for 12 h. The solvent was removed under reduced pressure and the residue was purified by recrystallization from ether/hexane. Carboxylic acid **36** (0.13 g, 87%) was obtained as a colorless solid; mp 82-83 $^\circ\text{C}$; IR (KBr) 1717, 1617, 1560, 1404, 1229, 1206, and 1140 cm^{-1} ; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 2.27 (s, 3H), 2.72 (t, 2H, $J =$

7.3 Hz), 4.01 (t, 2H, $J = 7.3$ Hz), 5.99 (dd, 1H, $J = 3.2$ and 1.0 Hz), and 6.23 (d, 1H, $J = 3.2$ Hz); ^{13}C -NMR (100 MHz, CDCl_3) δ 13.9, 33.8, 48.1, 107.6, 110.3, 120.0 (q, $J = 321.7$ Hz), 139.4, 152.5, and 176.5; Anal. Calcd. for $\text{C}_9\text{H}_{10}\text{F}_3\text{NO}_5\text{S}$: C, 35.88; H, 3.35; N, 4.65. Found: C, 36.13; H, 3.44; N, 4.66.

***N*-(5-Butyl-furan-2-yl)-C,C,C-trifluoro-*N*-methyl-methanesulfonamide (37).** To an ice cold solution of *N*-methyl succinimide (0.2 g, 1.8 mmol) in THF (2.0 ml) under argon was added 1.2 M solution of *n*-BuMgCl in THF (1.6 mL, 1.9 mmol) dropwise over 90 min. The mixture was stirred for another 10 min and was quenched with NH_4Cl (3 mL) in an ice bath. The aqueous layer was extracted with CHCl_3 , dried over MgSO_4 and concentrated under reduced pressure to afford a clear oil; ^1H -NMR (400 MHz, CDCl_3) δ 0.77-0.89 (m, 3H), 0.95-2.60 (m, 6H), 2.62 (d, 3H, $J = 2.4$ Hz), 2.79 (s, 2H), 2.87 (s, 2H), and 5.10 (s, 1H). Cyclic carbinol **24** was used in the next step without further purification.

To a solution of the above oil (0.1 g, 0.6 mmol) in CH_2Cl_2 (6 mL) at -78°C was slowly added pyridine (0.2 mL, 2.9 mmol) followed by triflic anhydride (0.2 mL, 1.2 mmol). The reaction was allowed to warm to room temperature and was quenched with H_2O . The mixture was extracted with CHCl_3 , dried over MgSO_4 and concentrated under reduced pressure. Purification by flash chromatography on silica gel using 20% ethyl acetate/hexane as the eluent gave **37** in 49% yield as a yellow oil: IR (neat) 1616, 1561, 1402, 1234, 1192, 1129, and 970 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 0.93 (t, 3H, $J = 7.6$ Hz), 1.30-1.42 (m, 2H), 1.56-1.66 (m, 2H), 2.58 (t, 2H, $J = 7.6$ Hz), 3.42 (s, 3H), 5.97 (d, 1H, $J = 3.1$ Hz), and 6.18 (d, 1H, $J = 3.1$ Hz); ^{13}C -NMR (75 MHz, CDCl_3) δ 13.9, 22.3, 28.0, 29.9, 40.0, 106.5, 107.4, 120.2 (q, $J = 322.4$ Hz), 141.9, and 155.9; Anal. Calcd. for $\text{C}_{10}\text{H}_{14}\text{F}_3\text{NO}_3\text{S}$: C, 42.10; H, 4.95; N, 4.91. Found: C, 42.47; H, 5.07; N, 4.11.

***N*-(5-But-3-enyl-furan-2-yl)-C,C,C-trifluoro-*N*-methyl-methanesulfonamide (38).** To a suspension of 0.49 g (20 mmol) of magnesium turnings in 13 mL of ether at room temperature was added dropwise a solution of 4-bromo-1-butene (0.4 mL, 4.4 mmol) in 7 mL of ether. The mixture was heated under reflux for 1 h. A solution of *N*-methyl succinimide (0.5 g, 4.4 mmol) in ether was added dropwise and the mixture was stirred for 1 h. The mixture was

quenched slowly by the addition of cold H₂O, extracted with EtOAc, dried over MgSO₄ and concentrated under reduced pressure to leave behind a clear oil: ¹H-NMR (300 MHz, CDCl₃) δ 1.60-2.54 (m, 4H), 2.66 (s, 2H), 2.69 (s, 3H), 2.92 (s, 2H), 4.78 (s, 1H), and 4.88-5.06 (m, 2H), 5.64-5.86 (m, 1H). Cyclic carbinol **25** was used in the next step without further purification.

To a solution of the above compound (0.19 g, 1.1 mmol) in CH₂Cl₂ (11 mL) at -78°C was slowly added pyridine (0.46 mL, 5.7 mmol) followed by Tf₂O (0.4 mL, 2.4 mmol) respectively. The reaction mixture was allowed to warm to room temperature and was quenched with H₂O. The solution was extracted with CHCl₃, dried over MgSO₄ and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel using 20% ether/hexane as the eluent to give furan **38** in 43% yield as a pale yellow oil: IR (neat) 1644, 1618, 1398, 1234, 1203, 1132, and 973 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 2.34-2.42 (m, 2H), 2.68 (t, 2H, J = 7.6 Hz), 3.40 (d, 3H, J = 0.8 Hz), 4.97-5.10 (m, 2H), 5.81 (qt, 1H, J = 6.8 and 3.6 Hz), 6.00 (d, 1H, J = 3.2 Hz) and 6.18 (d, 1H, J = 3.2 Hz); ¹³C-NMR (75 MHz, CDCl₃) δ 27.8, 31.8, 40.0, 106.9, 107.4, 115.8, 120.2 (q, J = 322 Hz), 137.1, 142.1, and 154.8; Anal. Calcd. for C₁₀H₁₂F₃NO₃S: C, 42.40; H, 4.27; N, 4.94. Found: C, 42.66; H, 4.37; N, 4.90.

C,C,C-Trifluoro-N-methyl-N-(5-pent-4-enyl-furan-2-yl)-methanesulfonamido (39). To a suspension of 0.6 g (25 mmol) of magnesium turnings under argon in ether (16 mL) was added dropwise a solution of 5-bromo-1-pentene (0.6 mL, 5.0 mmol) in 8 mL of ether. The mixture was heated at reflux for 1 h. A solution of *N*-methyl succinimide (0.62 g, 5.5 mmol) in ether was added dropwise and the mixture was stirred for 1 h. The solution was quenched with cold H₂O, extracted with EtOAc, dried over MgSO₄ and concentrated under reduced pressure to leave a clear oil: ¹H-NMR (300 MHz, CDCl₃) δ 1.10-2.44 (m, 6H), 2.58 (d, 3H, J = 1.2 Hz), 2.76 (s, 2H), 2.84 (s, 2H), 4.78- 4.94 (m, 2H), 5.08 (s, 1H), and 5.54-5.76 (m, 1H). Cyclic carbinol **26** was used in the next step without further purification.

To a solution of the above compound (0.3 g, 1.7 mmol) in CH₂Cl₂ (17 mL) at -78°C was slowly added pyridine (0.7 mL, 8.5 mmol) followed by triflic anhydride (0.6 mL, 3.6 mmol). The reaction mixture was allowed to warm to room temperature and was quenched

with H₂O. The solution was extracted with CHCl₃, dried over MgSO₄ and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel using 20% ether/hexane as the eluent to give furan **39** in 36% yield as a pale yellow oil: IR (neat) 1762, 1642, 1401, 1233, 1196, 1130, and 990 cm⁻¹; ¹H-NMR (300 MHz, CDCl₃) δ 1.66-1.79 (m, 2H), 2.09 (q, 2H, J = 7.3 Hz), 2.59 (t, 2H, J = 7.3 Hz), 3.41 (d, 3H, J = 0.9 Hz), 4.96-5.08 (m, 1H), 5.72-5.87 (m, 1H), 5.98 (d, 1H, J = 3.1 Hz), and 6.19 (d, 1H, J = 3.1 Hz); ¹³C-NMR (75 MHz, CDCl₃) δ 27.0, 27.6, 33.1, 40.0, 106.7, 107.4, 115.4, 120.2 (q, J = 322.4 Hz), 138.1, 142.0, and 155.4; Anal. Calcd. for C₁₁H₁₄F₃NO₃S: C, 44.44; H, 4.75; N, 4.71. Found: C, 44.30; H, 4.65; N, 4.51.

C,C,C-Trifluoro-N-methyl-N-(5-phenyl-furan-2-yl)methanesulfonamide (40). To an ice cold solution of *N*-methyl succinimide (0.3 g, 2.7 mmol) in THF (3.0 mL) was added a 1.0 M solution of PhMgBr in THF (3 mL, 2.9 mmol) dropwise over 90 min. The mixture was stirred for another 10 min and was quenched with aqueous NH₄Cl (5 mL). The aqueous layer was extracted with CHCl₃, dried over MgSO₄ and concentrated under reduced pressure. The crude product was purified by recrystallization from ethyl acetate-hexane to give 0.4 g (79%) of cyclic carbinol **27** as a white solid; ¹H-NMR (300 MHz, CDCl₃) δ 2.34 (dd, 2H, J = 7.2 Hz and J = 8.2 Hz), 2.44-2.74 (m, 2H), 3.00 (s, 3H), and 7.30-7.44 (m, 5H).

To a solution of the above solid (0.4 g, 2.5 mmol) in CH₂Cl₂ (25 mL) at -78°C was slowly added pyridine (1.0 mL, 12.3 mmol) followed by triflic anhydride (0.9 mL, 5.2 mmol). The reaction mixture was allowed to warm to room temperature and was quenched with H₂O. The mixture was extracted with CHCl₃, dried over MgSO₄ and concentrated under reduced pressure. Purification by flash chromatography on silica gel using 20% ether/hexane as the eluent gave 0.6 g (80%) of **40** as a clear oil: IR (neat) 1600, 1402, 1237, 1192, 1132 cm⁻¹; ¹H-NMR (300 MHz, CDCl₃) δ 3.51 (d, 3 H, J = 0.9 Hz), 6.40 (d, 1H, J = 3.4 Hz), 6.63 (d, 1H, J = 3.4 Hz), and 7.31-7.65 (m, 5H); ¹³C-NMR (75 MHz, CDCl₃) δ 40.8, 106.3, 108.7, 120.2 (q, J = 322.4 Hz), 124.1, 128.5, 129.0, 130.0, 143.2, and 152.7; Anal. Calcd. for C₁₂H₁₀F₃NO₃S: C, 47.21; H, 3.30; N, 4.59. Found: C, 47.59; H, 3.32; N, 4.59.

2-(2-Oxo-cyclohexyl)-N-(2-trimethylsilanyl-ethyl)acetamide (45) was obtained as white solid in 81% yield; mp 79-80 °C; IR (KBr) 1708, 1640, 1554, 1247, and 833 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 0.00 (s, 9H), 0.74-0.80 (m, 2H), 1.36 (dq, 1H, J = 13.0 and 3.8 Hz), 1.55-1.90 (m, 3H), 1.98 (dd, 1H, J = 14.3 and 5.1 Hz), 2.05-2.20 (m, 2H), 2.30-2.43 (m, 2H), 2.57 (dd, 1H, J = 14.3 and 7.6 Hz), 2.84-2.94 (m, 1H), 3.19-3.28 (m, 2H), and 5.79 (brs, 1H); ¹³C-NMR (100 MHz, CDCl₃) δ -1.5, 176.9, 25.5, 28.2, 34.8, 36.1, 37.1, 42.3, 48.1, 171.6, 212.9; Anal. Calcd. for C₁₃H₂₅NO₂Si: C, 61.13; H, 9.87; N, 5.48. Found: C, 61.07; H, 9.94; N, 5.33.

2,2,2-Trifluoro-N-(2-trimethylsilanyl-ethyl)-N-(4,5,6,7-tetrahydrobenzofuran-2-yl)-acetamide (49) was obtained in 85% yield as a colorless oil: IR (neat) 1712, 1579, 1208, 1155, and 861 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 0.02 (s, 9H), 0.85-0.93 (m, 2H), 1.68-1.88 (m, 4H), 2.36-2.43 (m, 2H), 2.50-2.57 (m, 2H), 3.62-3.70 (m, 2H), and 5.97 (s, 1H); ¹³C-NMR (100 MHz, CDCl₃) δ -1.6, 15.5, 22.0, 23.0, 23.1, 47.4, 107.8, 116.2 (q, J = 288.4 Hz), 118.1, 141.5, 149.7, and 157.3 (q, J = 35.1 Hz); Anal. Calcd. for C₁₅H₂₂F₃NO₂Si: C, 54.03; H, 6.65; N, 4.20. Found: C, 54.29; H, 6.79; N, 4.30.

N-(2-Trimethylsilanyl-ethyl)-C,C,C-trifluoro-N-(4,5,6,7-tetrahydrobenzofuran-2-yl)-methanesulfonamide (53) was obtained in 82% yield as a colorless oil: IR (neat) 1399, 1230, 1195, 1137, and 1044 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 0.03 (s, 9H), 0.88-0.95 (m, 2H), 1.68-1.77 (m, 2H), 1.79-1.88 (m, 2H), 2.37-2.43 (m, 2H), 2.51-2.57 (m, 2H), 3.69-3.76 (m, 2H), and 6.10 (s, 1H); ¹³C-NMR (100 MHz, CDCl₃) δ -1.6, 17.5, 22.0, 22.8, 22.9, 23.1, 50.6, 109.8, 118.5, 120.2 (q, J = 324.3 Hz), 139.3, and 150.6; Anal. Calcd. for C₁₄H₂₂F₃NO₃SSi: C, 45.5; H, 6.00; N, 3.79. Found: C, 45.73; H, 6.09; N, 3.92.

References

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