Supporting Information

for

Asymmetric Synthesis of Highly-Substituted β -Nitroalcohols and Enantiomerically-Enriched 4,4,5-Trisubstituted Oxazolidinones

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General procedure for the Henry Reaction. 2-Nitropropane (20 equiv.) and aldehyde were dissolved in a 1/1 mixture of acetonitrile and *tert*-butanol to a concentration of 0.1M in aldehyde. After cooling the mixture to 0 °C, potassium *tert*-butoxide (1.0 equiv.) was added and the reaction mixture stirred for 24h with gradual warming to room temperature. Saturated aqueous ammonium chloride (50 mL) and ethyl acetate (50 mL) were then added, the organic layer was separated and the aqueous layer extracted with ethyl acetate. The combined organic layer was washed with water, brine, dried (Na₂SO₄) and the solvent removed under reduced pressure. Purification by silica gel chromatography (eluent: ethyl acetate/hexanes, 1:5) afforded the respective nitroaldol products.

(±)-3-Methyl-3-nitro-2-butanol (10). Following the general procedure, acetaldehyde (4.97 g, 0.12 mol) afforded **10**¹ (10.5g, 70 %) as a colorless oil. IR (film, cm⁻¹) γ: 3448,

1539, 1462; ^1H NMR δ 4.22 (q, J = 6.6 Hz, 1H), 2.55 (br, s, 1H), 1.53 (s, 3H), 1.50 (s, 3H), 1.17 (d, J = 6.6 Hz, 3H); ^{13}C NMR δ 92.3, 71.9, 23.7, 19.7, 17.8.

($\pm$)-4-Methyl-4-nitro-3-pentanol (11). Following the general procedure, propionaldehyde (1.17 g, 20 mmol) afforded **11**¹ (2.57 g, 87%) as a colorless oil. IR (film, cm^{-1}) γ : 3450, 1540, 1462; ^1H NMR δ 3.90-3.84 (br. m, 1H), 2.65 (br. d, J = 6.3 Hz, 1H), 1.51-1.43 (m, 7H), 1.31-1.20 (m, 1H), 1.01 (t, J = 7.2 Hz, 3H); ^{13}C NMR δ 92.3, 77.6, 24.6, 23.7, 20.3, 11.1.

($\pm$)-4-Methyl-4-nitro-1-phenyl-3-pentanol (12). Following the general procedure, 3-phenylpropionaldehyde (1.56 g, 11.6 mmol) afforded **12**² (1.90 g, 73%) as a colorless oil. IR (film, cm^{-1}) γ 3467, 1541; ^1H NMR δ 7.34-7.20 (m, 5H), 4.05-4.00 (m, 1H), 2.97-2.92 (m, 1H), 2.74-2.67 (m, 1H), 2.52-2.49 (d, J = 6.9 Hz, 1H), 1.80-1.63 (m, 2H), 1.56 (s, 3H), 1.52 (s, 3H); ^{13}C NMR δ 141.2, 128.7, 128.6, 126.3, 92.3, 75.3, 33.4, 32.6, 23.9, 20.3. ESIHRMS: Calcd for $\text{C}_{12}\text{H}_{17}\text{NO}_3$ [$\text{M}+\text{Na}$] 246.1098. Found: 246.1106.

($\pm$)-Cyclohexyl-2-methyl-2-nitro-1-propanol (13). 2-Nitropropane (4.76 g, 53.5 mmol) was first treated with *tert*-BuOK (1.2 g, 10.7 mmol) for 5 minutes followed by the addition of cyclohexane carboxaldehyde (2.0 g, 17.8 mmol) dropwise over 15min. The contents were then stirred for 24h at room temperature after which the work up and purification in the general Henry procedure were followed to afford **13** (0.63 g, 18%) as a colorless oil. IR (film, cm^{-1}) γ 3467, 1540; ^1H NMR δ 3.77 (d, J = 3.6 Hz, 1H), 2.97 (br. s, 1H), 1.74-1.15 (m, 11H), 1.58 (s, 3H), 1.57 (s, 3H); ^{13}C NMR δ 92.0, 79.8, 39.8, 32.3, 28.9, 27.0, 26.6, 26.1, 23.8, 22.4. ESIHRMS: Calcd for $\text{C}_{10}\text{H}_{19}\text{NO}_3$ [$\text{M}+\text{Na}$] 224.1264. Found: 224.1263.

(±)-1-(2-Furanyl)-2-methyl-2-nitro-1-propanol (14). Following the general procedure, furfuraldehyde (4.4 g, 45.8 mmol) afforded **14**³ (6.80 g, 81%) as a pale yellow oil. IR (film, cm⁻¹) γ 3380, 1543; ¹H NMR δ 7.38 (s, 1H), 6.36 (s, 2H), 5.25 (d, J = 5.1 Hz, 1H), 2.93 (d, J = 4.5 Hz, 1H), 1.63 (s, 3H), 1.49 (s, 3H); ¹³C NMR δ 151.3, 142.9, 110.6, 109.3, 91.3, 72.6, 24.1, 20.0.

(±)-2-Methyl-2-nitro-1-(2-thienyl)-1-propanol (15). To a solution of 2-thiophene carboxyaldehyde (6.95 g, 62.0 mmol) and 2-nitropropane (17 mL, 0.19 mol) in methanol (70mL) was added sodium methoxide (8.08 g, 0.15 mol) in one portion at room temperature and the reaction mixture stirred overnight. Acetic acid was then added to bring the reaction mixture to pH 4. The resulting brown suspension was filtered through Celite and washed thoroughly with ethyl acetate. Concentration of the filtrate under reduced pressure afforded a residue, to which was added a saturated solution of sodium bisulfite (25 mL) and ethyl acetate (100 mL). The two-phase mixture was stirred vigorously for 30 min and the layers separated. The organic layer was washed well with water, brine and dried (Na₂SO₄) and the solvent removed under reduced pressure. Recrystallization of the crude reaction mixture from cyclohexane afforded **15**³ (4.42 g, 35%) as white crystals. M.p. 63 °C (lit³ m.p. 59-60 °C); IR (film, cm⁻¹) γ 3517, 1539; ¹H NMR δ 7.30 (dd, J = 5.1, 1.2 Hz, 1H), 7.03-6.98 (m, 2H), 5.53 (s, 1H), 3.00 (br. s, 1H), 1.64 (s, 3H), 1.49 (s, 3H); ¹³C NMR δ 141.4, 126.9, 126.7, 126.1, 92.0, 74.9, 24.2, 19.7.

(±)-1-(1-Nitrocyclopentyl)-3-phenyl-1-propanol (16). Following the general procedure, reaction of 3-phenylpropanal (0.97 g, 7.52 mmol) with nitrocyclopentane⁴ (4.5 equiv, 3.7 g, 32.9 mmol) afforded **16** (0.92 g, 50%) as a colorless oil. IR (film, cm⁻¹) γ : 3538, 2958, 1535, 1453; ¹H NMR δ 7.34-7.2 (m, 5H), 3.89 (br. d, J = 9.3 Hz, 1H), 3.01-

2.92 (m, 2H), 2.76-2.39 (m, 3H), 2.15-2.05 (m, 1H), 1.86-1.55 (m, 7H); ^{13}C NMR δ 141.4, 128.7, 128.6, 126.3, 103.7, 75.1, 35.6, 34.4, 33.8, 32.5, 24.9, 24.7. ESIHRMS: Calcd for $\text{C}_{14}\text{H}_{19}\text{NO}_3\text{Na}$ $[\text{M}+\text{Na}]$ 272.1263. Found: 272.1254.

General procedure for the Formation of Mosher Esters. To a solution of nitroalcohol in ethyl ether (0.04 M in substrate) was added (*R*)-(+)- or (*S*)-(-)- α -methoxy- α -(trifluoromethyl)phenylacetic acid (3.0 equiv.) followed by DMAP (0.5 equiv.) and DCC (3.75 equiv.). The reaction mixture was stirred at room temperature for 24 h then diluted with ethyl acetate (30 mL). The organic layer was washed with a saturated solution of sodium bicarbonate, water, and brine and dried (Na_2SO_4) and the solvent removed under reduced pressure. The crude reaction mixture was purified by silica gel column chromatography (eluent: ethyl acetate/hexanes, 1:10) to afford the corresponding Mosher esters.

***O*-(2-Methyl-2-nitro-3-pentyl) 3,3,3-Trifluoro-2-methoxy-2-phenylpropionate (**18**).** Following the general procedure, ($\pm$)-**11** (0.08 g, 0.53 mmol) and (*R*)- α -methoxy- α -(trifluoromethyl)phenylacetic acid afforded **18** (0.17 g, 85 %) as a 1/1 mixture of diastereomers. IR (film, cm^{-1}) γ : 2949, 1746, 1545, 1454; ^1H NMR δ 7.52-7.40 (m, 10H), 5.73-5.58 (m, 2H), 3.49 (s, 3H), 3.47 (s, 3H), 1.72-1.51 (m, 16H), 0.97 (t, $J = 7.4$ Hz, 3H), 0.92 (t, $J = 7.43$ Hz, 3H); ^{13}C NMR δ 166.1, 166.0, 131.8, 131.7, 130.1, 129.9, 129.0, 128.9, 127.9, 127.8, 124.8, 122.5, 90.2, 90.1, 80.5, 80.4, 55.7, 55.6, 24.0, 23.9, 21.7, 21.3, 11.1, 10.9; ^{19}F NMR δ 1.50, 1.16. ESIHRMS: Calcd for $\text{C}_{16}\text{H}_{20}\text{F}_3\text{NO}_5\text{Na}$ $[\text{M}+\text{Na}]$ 386.1191. Found: 386.1208. When the reaction was repeated with (*R*)-**11**, ester **18** was obtained in 90% yield as a 13.5/1 mixture of diastereomers (based on ^{19}F NMR studies). ^1H NMR (major isomer) δ 7.53-7.40 (m, 5H), 5.71 (dd, $J = 9.9$ Hz, 2.8 Hz, 1H),

3.50 (s, 3H), 1.69-1.53 (m, 8H), 0.97 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (major isomer) δ 166.0, 131.8, 130.2, 128.9, 127.8, 127.1, 124.8, 122.5, 90.1, 80.4, 55.8, 24.0, 23.5, 21.8, 11.1; ^{19}F NMR δ 1.50 (minor), 1.16 (major).

***O*-(4-Methyl-4-nitro-1-phenyl-3-pentyl) 3,3,3-Trifluoro-2-methoxy-2-phenylpropionate (19).** Following the general procedure, ($\pm$)-**12** (0.05 g, 0.22 mmol) and (*R*)- α -methoxy- α -(trifluoromethyl)phenylacetic acid afforded **19** (0.78 g, 81%) as 1/1 mixture of diastereomers (based on ^{19}F NMR studies). IR (film, cm^{-1}) γ 1750, 1541; ^1H NMR δ 7.60-7.07 (m, 10H), 5.85-5.79 (m, 1H), 3.53 (s, 3H), 2.65 (m, 2H), 1.91-1.87 (m, 2H), 1.55 (s, 3H), 1.52 (s, 3H); ^{13}C NMR δ 165.7, 140.4, 140.3, 130.0, 128.8, 128.7, 128.4, 128.4, 127.6, 127.5, 127.5, 126.7, 126.6, 89.8, 89.7, 78.3, 78.3, 55.5, 55.4, 33.0, 32.4, 32.3, 23.7, 23.2, 21.4, 20.9; ^{19}F NMR δ 1.58, 1.29. ESIHRMS: Calcd for $\text{C}_{22}\text{H}_{24}\text{F}_3\text{NO}_5$ [M+K] 478.1260. Found: 478.1244. When the reaction was repeated with (*R*)-**12**, ester **19** was obtained in 84% yield as a predominant diastereomer in the ratio of 33.3/1 (94% ee) based on the ^{19}F NMR studies. ^1H NMR (major isomer) δ 7.55-7.08 (m, 10H), 5.85-5.82 (m, 1H), 3.52 (s, 3H), 2.61 (t, $J = 8.1$ Hz, 2H), 1.84-1.81 (m, 2H), 1.52 (s, 3H), 1.51 (s, 3H); ^{13}C NMR (major isomer) δ 165.9, 140.4, 131.4, 130.0, 128.7, 128.4, 127.6, 127.5, 126.7, 126.6, 125.3, 89.8, 78.4, 55.4, 33.0, 32.3, 23.7, 20.9; ^{19}F NMR (C_6D_6) δ 1.57 (major), 1.28 (minor).

***O*-(1-Cyclohexyl-2-methyl-2-nitro-1-propyl) 3,3,3-Trifluoro-2-methoxy-2-phenylpropionate (20).** Following the general procedure, ($\pm$)-**13** (0.07 g, 0.34 mmol) and (*R*)- α -methoxy- α -(trifluoromethyl)phenylacetic acid afforded **20** (0.123 g, 85%) as a 1/1 mixture of diastereomers (based on ^{19}F NMR studies). IR (film, cm^{-1}) γ 1751, 1543; ^1H NMR δ 7.43-7.41 (m, 5H), 5.55-5.53 (m, 1H), 3.48 (s, 3H), 1.65-1.52 (m, 11H), 1.25-

0.96 (m, 6H); ^{13}C NMR δ 166.1, 131.7, 130.2, 130.1, 128.9, 128.8, 128.8, 128.1, 128.0, 127.8, 127.1, 124.8, 90.5, 90.4, 85.2, 85.0, 55.7, 39.8, 39.4, 32.6, 32.4, 30.1, 30.0, 28.2, 27.8, 26.7, 26.7, 26.4, 26.42, 26.1, 26.0, 23.5, 23.3, 23.0, 22.6; ^{19}F NMR (C_6D_6) δ 1.37, 1.18. ESIHRMS: Calcd for $\text{C}_{20}\text{H}_{26}\text{F}_3\text{NO}_5\text{Na}$ [$\text{M} + \text{Na}$] 440.1641. Found: 440.1661. When the reaction was repeated with (*R*)-**13**, ester **20** was obtained in 78% yield as a 4:1 (60% ee) mixture of diastereomers (based on ^{19}F NMR studies). IR (film, cm^{-1}) ν 1750, 1550; ^1H NMR (major isomer) δ 7.53-7.25 (m, 5H), 5.56-5.54 (m, 1H), 3.48 (s, 3H), 1.70-1.60 (m, 7H), 1.57 (s, 3H), 1.53 (s, 3H), 1.25-1.12 (m, 4H); ^{13}C NMR (major isomer) δ 166.1, 131.7, 130.2, 128.9, 128.4, 128.2, 128.1, 128.0, 90.4, 82.3, 55.7, 39.8, 32.4, 28.2, 26.7, 26.42, 26.1, 23.3, 23.0. ^{19}F NMR (C_6D_6): δ 1.38 (minor), 1.20 (major).

***O*-[1-(2-Furanyl)-2-methyl-2-nitro-1-propyl] 3,3,3-Trifluoro-2-methoxy-2-phenylpropionate (21).** Following the general procedure, ($\pm$)-**14** (0.060 g, 0.32 mmol) and (*R*)- α -methoxy- α -(trifluoromethyl)phenylacetic acid afforded **21** (0.098 g, 75%) as 1/1 mixture of diastereomers (based on ^{19}F NMR studies). ^1H NMR δ 7.47-7.33 (m, 6H), 6.73 (s, 1H), 6.56-6.55 (m, 1H), 6.42-6.41 (m, 1H), 3.43 (s, 3H), 1.75 (s, 3H), 1.48 (s, 3H); ^{13}C NMR δ 164.7, 146.5, 143.8, 131.7, 129.6, 128.3, 127.1, 125.0, 112.7, 110.8, 110.6, 89.3, 73.3, 73.3, 55.4, 24.2, 20.2; ^{19}F NMR (C_6D_6) δ 0.96, 0.76. ESIHRMS: Calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_6\text{F}_3$ [$\text{M} + \text{Na}$] 424.0976. Found: 424.0984. When the reaction was repeated with (*S*)-**14**, ester **21** was obtained in 89% yield as a mixture of diastereomers in the ratio of 6.7/1 (based on ^{19}F NMR studies). IR (film, cm^{-1}) ν 1765, 1550; ^1H NMR (major isomer) δ 7.39-7.25 (m, 6H), 6.65 (s, 1H), 6.37 (s, 2H), 3.41 (s, 3H), 1.71 (s, 3H), 1.48 (s, 3H); ^{13}C NMR (major isomer) δ 165.1, 146.9, 144.2, 144.0, 130.0, 128.7, 127.6, 112.4, 111.0, 89.7, 74.4, 55.8, 24.9, 20.0; ^{19}F NMR (C_6D_6) δ 0.95 (major), 0.74 (minor).

***O*-[1-(2-Thienyl)-2-methyl-2-nitro-1-propyl] 3,3,3-Trifluoro-2-methoxy-2-phenylpropionate (22).** Following the general procedure with CH₂Cl₂, as solvent, (±)-**15** (0.034 g, 0.17 mmol) and (*R*)-α-methoxy-α-(trifluoromethyl)phenylacetic acid afforded **22** (0.064 g, 90%) as 1/1 mixture of diastereomers (based on ¹⁹F NMR studies). IR (film, cm⁻¹) γ 1760, 1549; ¹H NMR δ 7.43-7.30 (m, 12H), 7.18 (dd, 1H, *J* = 3.6, 0.9 Hz), 7.05-6.97 (m, 3H), 6.95 (s, 1H), 6.85 (s, 1H), 3.45 (d, 3H, *J* = 1.2 Hz), 3.43 (d, 3H, *J* = 1.5 Hz), 1.70 (s, 3H), 1.69 (s, 3H), 1.51 (s, 3H), 1.49 (s, 3H); ¹³C NMR δ 164.9, 164.8, 135.1, 135.0, 131.8, 131.7, 129.9, 129.8, 129.7, 129.2, 128.6, 128.5, 127.5, 127.4, 127.3, 127.0, 126.9, 125.2, 125.16, 121.4, 121.3, 90.1, 77.0, 76.6, 55.7, 24.9, 24.4, 20.1, 19.4; ¹⁹F NMR (CDCl₃) δ +1.10, +1.03. ESIHRMS: Calcd for C₁₈H₁₈F₃NO₅SK: 456.0495. Found: 456.0492. When the reaction was repeated with (*S*)-**15**, ester **22** was obtained in 89% yield as a 3/1 mixture of diastereomers (based on ¹⁹F NMR studies). ¹H NMR (major isomer) δ 7.40-7.30 (m, 6H), 7.05-6.97 (m, 2H), 6.85 (s, 1H), 3.43 (d, 3H, *J* = 1.2 Hz), 1.70 (s, 3H), 1.51 (s, 3H); ¹³C NMR (major isomer) δ 164.8, 135.0, 131.7, 129.8, 129.7, 129.2, 128.6, 128.5, 127.5, 127.3, 126.9, 125.1, 121.3, 90.1, 77.0, 55.7, 24.9, 19.4; ¹⁹F NMR δ +1.10 (major), +1.03 (minor).

***O*-[1-(1-Nitrocyclopentyl)-3-phenylpropyl] 3,3,3-Trifluoro-2-methoxy-2-phenylpropionate (23).** Following the general procedure, (±)-**16** (0.056 g, 0.22 mmol) and (*R*)-α-methoxy-α-(trifluoromethyl)phenylacetic acid afforded **23** (0.072 g, 73%) as 1/1 mixture of diastereomers (based on fluorine NMR studies). IR (film, cm⁻¹) γ: 2950, 1764, 1542, 1452; ¹H NMR δ 7.60-7.06 (m, 20H), 5.76-5.68 (m, 2H), 3.56-3.54 (m, 6H), 2.58-2.46 (m, 8H), 1.92-1.56 (m, 16H); ¹³C NMR δ 166.6, 166.4, 140.6, 140.5, 131.8, 130.3, 129.0, 128.7, 127.9, 126.9, 126.8, 124.9, 124.8, 122.6, 122.5, 101.8, 101.6, 77.9, 77.7,

55.9, 35.0, 34.9, 34.8, 34.4, 33.7, 33.6, 32.6, 32.4, 24.6, 24.5; ^{19}F NMR δ 1.97, 1.52. ESIHRMS: Calcd for $\text{C}_{24}\text{H}_{26}\text{NO}_5\text{F}_3\text{Na}$ $[\text{M}+\text{Na}]$ 488.1661. Found: 488.1655. When the reaction was repeated with (*R*)-**16**, ester **23** was obtained in 76% yield as a predominant diastereomer in the ratio 30/1 (based on the fluorine NMR studies). ^1H NMR (major) δ 7.59-7.04 (m, 10H), 5.70-5.67 (m, 1H), 3.55 (s, 3H), 2.51-2.47(m, 4H), 1.87-1.61 (m, 8H); ^{13}C NMR (major) δ 166.6, 140.6, 131.8, 130.3, 129.0, 128.6, 127.8, 126.8, 124.8, 122.6, 101.7, 77.9, 55.9, 34.9, 34.8, 33.6, 32.4, 24.6, 24.5. ^{19}F NMR (CDCl_3) δ 1.97 (major), 1.52 (minor).

References

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16

18 (Racemic MTPA ester)

18 (enantiomerically enriched MTPA ester)

19 (Racemic MTPA ester)

19 (enantiomerically enriched MTPA ester)

20 (racemic MTPA ester)

20 (enantiomerically enriched MTPA ester)

21 (racemic MTPA ester)

21 (enantiomerically enriched MTPA ester)

22 (racemic MTPA ester)

22 (enantiomerically enriched MTPA ester)

23 (racemic MTPA ester)

23 (enantiomerically enriched MTPA ester)

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