Supporting Information for “A Modular Synthesis of Amine Functionalized Oxazolines”

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General Methods:

Unless otherwise noted all reactions were performed under an argon atmosphere with stirring. CH_2Cl_2 was freshly distilled from CaH_2 . Fmoc amino acids were obtained from Advanced Chemtech and rigorously dried by azeotroping toluene before use. Amino alcohols were prepared by methods reported in literature.¹ DIPEA (Lancaster) was distilled from CaH_2 and stored under nitrogen in the dark. Flash chromatography was performed using EM Reagent silica 60 (230-400 mesh). Analytical thin layer chromatography was performed with Whatman K6F Silica gel 60 Å plates. Pyridyloxazoline **14** has been previously reported.² All melting points are uncorrected and were recorded on a Electrothermal Melting Point apparatus. Optical rotations were recorded on a Perkin Elmer Model 343 Polarimeter. IR spectra were recorded using a Mattson Satellite FTIR instrument. NMR spectra³ were recorded using one of the following: (i) Varian Unity-300 Spectrometer, (ii) Varian XL-300 Spectrometer, (iii) Varian VXR-500 Spectrometer. HRMS were recorded using a Finnigan MAT 95 spectrometer.

Purification of PPh_3 :

PPh_3 was purified by recrystallization from hexane after a hot filtration. The crystals are washed with hexane and repeatedly azeotroped with dry toluene followed by drying under high vacuum.

Purification of CCl_4 :

CCl_4 is stirred overnight with one fifth its volume of satd. KOH. After washing with water, nitrogen is bubbled through for two hours. It is then percolated through a 2,4-dinitrophenylhydrazine column,⁴ distilled from CaH_2 and stored under Nitrogen.

Synthesis of protected oxazolines:

General Procedure for synthesis of F-moc protected oxazolines:

0.2 mmol of amino alcohol, 0.2 mmol of Fmoc protected amino acid and 0.6 mmol of PPh_3 are weighed into a flame-dried 10 ml Schlenk tube. The tube is flushed with argon and 3.9 ml of CH_2Cl_2 is added followed by 0.6 mmol of DIPEA. The tube is cooled down to 0 °C with an ice bath. 1 mmol of CCl_4 is added over three hours, during

which time the tube is allowed to warm up to ambient temperature. The reaction is stirred for 24h.

Purification:

~ 30 ml of silica is made into a slurry with 10% EtOAc/hexanes and packed into a column. The reaction mixture is loaded onto the column without any reduction in volume. The tube is rinsed with ~0.5ml of CH₂Cl₂ and ~2 ml of 10% EtOAc/hexanes. The column is first eluted with ~150 ml of 10% EtOAc/hexanes followed by ~200 ml of 30% EtOAc/hexanes. The fractions containing product are collected and the solvents are removed under reduced pressure.

(1): Yield: 92%; white solid; mp: 126.1-127.6 °C (dec); $[\alpha]_D^{20} = -33.2^\circ$ (c = 0.25, CHCl₃); IR (KBr) 3400, 3306, 3033, 2962, 2937, 1690, 1666, 1533, 1274, 1263 cm⁻¹; ¹HNMR (500 MHz, CDCl₃) δ 1.27 (d, *J* = 6.8 Hz, 3H), 3.04 (dd, *J* = 5.9 Hz, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 6.4 Hz, *J* = 13.7 Hz, 1H), 4.0 (dq, *J* = 7.1 Hz, *J* = 7.1 Hz, 1H), 4.21 (t, *J* = 7.1 Hz, 1H), 4.33 (dd, *J* = 7.3 Hz, *J* = 10.8 Hz, 1H), 4.46 (dd, *J* = 7.3 Hz, *J* = 10.3 Hz, 1H), 4.86 (dd, *J* = 6.3 Hz, *J* = 6.3 Hz, 1H), 4.96 (d, *J* = 8.3 Hz, 1H), δ 5.13 (d, *J* = 8.3 Hz, 1H), 7.12 (t, *J* = 7.3 Hz, 2H), δ 7.25-7.42 (m, 12H), 7.58 (dd, *J* = 7.3 Hz, *J* = 10.3 Hz, 2H), 7.77 (d, *J* = 7.8 Hz, 2H); ¹³CNMR {¹H} (75 MHz, C₆D₆) δ 21.1, 39.4, 47.9, 51.1, 67.1, 70.5, 89.3, 120.1, 125.4, 125.5, 126.0, 126.9, 127.3, 127.8, 128.5, 128.9, 130.0, 137.1, 140.5, 141.9, 144.6, 144.7, 155.7, 165.2; HRMS (E.I.) *m/z* (M)⁺ calcd. 502.2256 obsd. 502.2235.

(2): Yield: 89%; white solid; mp: 135.6-138.1 °C (dec); $[\alpha]_D^{20} = +36^\circ$ (c = 0.175, CHCl₃); IR (KBr) 3312, 3062, 3029, 2960, 2924, 1693, 1664, 1545, 1450 cm⁻¹; ¹HNMR (500 MHz, CDCl₃) δ 1.60 (d, *J* = 6.8 Hz, 3H), 3.17 (dd, *J* = 5.4 Hz, *J* = 13.7 Hz, 1H), 3.24 (dd, *J* = 4.9 Hz, *J* = 13.7 Hz, 1H), 4.00 (dq, *J* = 6.6 Hz, *J* = 6.6 Hz, 1H), 4.23 (t, *J* = 6.8 Hz, 1H), 4.36 (dd, *J* = 6.8 Hz, *J* = 10.3 Hz, 1H), 4.54 (dd, *J* = 7.3 Hz, *J* = 10.7 Hz, 1H), 4.87 (dd, *J* = 5.7 Hz, *J* = 12.2 Hz, 1H), 5.47 (d, *J* = 7.8 Hz, 1H), 7.17 (d, *J* = 6.8 Hz, 2H), 7.27-7.44 (m, 12H), 7.61 (t, *J* = 8.3 Hz, 2H), 7.79 (d, *J* = 7.3 Hz, 2H); ¹³CNMR {¹H} (75 MHz, CDCl₃) δ 21.4, 38.7, 47.5, 50.2, 67.1, 70.4, 89.4, 120.2, 125.3, 125.4, 125.9, 127.3, 127.9, 128.6, 128.8, 129.1, 130.0, 136.0, 139.0, 141.6, 144.0, 144.2, 155.7, 164.9; HRMS (LSIMS, Cs⁺ in glycerol matrix) *m/z* (M+H)⁺ calcd. 503.2335 obsd. 503.2328.

(3): Yield: 78%; white solid; mp: 117.1-117.3 °C (dec); $[\alpha]_D^{20} = -35^\circ$ (c = 0.22, CHCl₃); IR (KBr) 3403, 2968, 2954, 2869, 1726, 1662, 1514, 1449, 1218 cm⁻¹; ¹HNMR (500 MHz, CDCl₃) δ 1.07 (s, 9H), 1.41 (d, *J* = 6.8 Hz, 3H), 4.09 (dq, *J* = 6.8 Hz, *J* = 6.8 Hz, 1H), 4.27 (t, *J* = 7.1 Hz, 1H), 4.40-4.43 (m, 3H), 4.97 (d, *J* = 8.2 Hz, 1H), 5.49 (d, *J* = 9.8 Hz, 1H), δ 7.29-7.41 (m, 1H), 7.62 (d, *J* = 7.3 Hz, 1H), 7.78 (d, *J* = 67.3 Hz, 2H); ¹³CNMR {¹H} (75 MHz, CDCl₃) δ 21.6, 26.8, 35.4, 47.4, 58.0, 67.2, 70.2, 89.0, 120.2, 125.4, 126.1, 127.3, 127.9, 128.7, 129.1, 140.0, 141.5, 141.0, 144.2, 156.3, 165.1; HRMS (LSIMS, Cs⁺ in glycerol matrix) *m/z* (M+H)⁺ calcd. 469.24863 obsd. 469.24912.

(4): Yield: 90%; white solid; mp: 141-142.5 °C (dec); $[\alpha]_D^{20} = +39.1^\circ$ (c = 0.115, CHCl₃); IR (KBr) 3414, 3033, 1719, 1700,

1507, 1497, 1332 cm^{-1} ; ^1H NMR(500 MHz, CDCl_3) δ 1.43 (d, J = 6.8 Hz, 3H), 4.00 (dq, J = 6.71 Hz, J = 6.71 Hz, 1H), 4.23 (t, J = 6.8 Hz, 1H), 4.40 (d, J = 7.3 Hz, 2H), 5.03 (d, J = 7.3 Hz, 1H), 5.67 (d, J = 6.8 Hz, 1H) 6.26 (d, J = 7.3 Hz, 1H), 6.92 (d, J = 5.8 Hz, 2H), 7.497-7.219 (m, 12H), 7.61 (d, J = 7.8 Hz, 2H), 7.76 (d, J = 7.3 Hz, 2H); ^{13}C NMR $\{^1\text{H}\}$ (75 MHz, CDCl_3) δ 21.7, 47.4, 53.7, 67.3, 70.8, 89.1, 120.2, 125.24, 125.4, 127.3, 127.4, 127.9, 128.4, 128.7, 128.9, 129.1, 138.1, 140.3, 141.5, 144.1, 144.2, 155.6, 165.1; HRMS (LSIMS, Cs^+ in glycerol matrix) m/z (M+H) $^+$ calcd. 489.2178 obsd. 489.2172.

(5): Yield: 88%; white solid; mp: 54-54.6 $^{\circ}\text{C}$ (dec); $[\alpha]_{\text{D}}^{20}$ = +31.8 $^{\circ}$ (c = 0.11, CHCl_3); IR (KBr) 3395, 3361, 2963, 2928, 1726, 1672, 1530, 1450, 1234 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.0 (d, J = 6.8 Hz, 3H), 1.04 (d, J = 6.8 Hz, 3H), 1.4 (d, J = 6.3 Hz, 3H), 2.20- 2.26 (m, 1H), 4.05 (dq, J = 7 Hz, J = 7 Hz, 1H), 4.26 (t, J = 7.1 Hz, 1H), 4.41 (d, J = 2.9 Hz, 1H), 4.42 (d, J = 3.4 Hz, 1H), 4.77 (dd, J = 4.9 Hz, J = 8.8 Hz, 1H), 4.98 (d, J = 8.3 Hz, 1H), 5.44 (d, J = 8.8 Hz, 1H), 7.26-7.42 (m, 9H), 7.72 (dd, J = 4.9 Hz, J = 7.3 Hz, 2H), 7.77 (d, J = 7.3 Hz, 2H); ^{13}C NMR $\{^1\text{H}\}$ (75 MHz, CDCl_3) δ 17.7, 19.3, 21.6, 31.7, 47.5, 54.8, 67.2, 70.4, 89.1, 120.2, 125.4, 125.9, 127.3, 127.9, 128.7, 129.1, 140.2, 141.6, 144.1, 144.3, 156.4, 165.4; HRMS (LSIMS, Cs^+ in glycerol matrix) m/z (M+H) $^+$ calcd. 455.2335 obsd. 455.23274.

(6): Yield: 87%; white solid; mp: 50.8-52.9 $^{\circ}\text{C}$ (dec); $[\alpha]_{\text{D}}^{20}$ = -7.1 $^{\circ}$ (c = 0.406, CHCl_3); IR (KBr) 3227, 3037, 2963, 2929, 1719, 1667, 1542, 1450, 1240 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.02 (d, J = 6.8 Hz, 3H), 1.06 (d, J = 6.8 Hz, 3H), 1.42 (d, J = 6.8 Hz, 3H), 2.02- 2.13 (m, 1H), 4.07 (dq, J = 6.6 Hz, J = 6.6 Hz, 1H), 4.27 (t, J = 6.8 Hz, 1H), 4.4 (dd, J = 6.8 Hz, J = 10.1 Hz, 1H), 4.48 (dd, J = 7.8 Hz, J = 10.7 Hz, 1H), 4.52 (dd, J = 4.9 Hz, J = 8.3 Hz, 1H), 4.99 (d, J = 7.8 Hz, 1H), 5.55 (d, J = 6.3 Hz, 1H), 7.3 – 7.43 (m, 9H), 7.64 (dd, J = 7.8 Hz, J = 8.8 Hz, 2H), 7.78 (d, J = 7.8 Hz, 2H); ^{13}C NMR $\{^1\text{H}\}$ (75 MHz, CDCl_3) δ 17.8, 19.2, 21.7, 31.8, 47.5, 54.7, 67.2, 70.4, 89.2, 120.2, 125.4, 125.9, 127.3, 127.9, 128.7, 129.1, 140.0, 141.6, 144.1, 144.3, 156.5, 165.7 HRMS (LSIMS, Cs^+ in glycerol matrix) m/z (M+H) $^+$ calcd. 455.2335 obsd. 455.23220.

(7): Yield: 57%; clear sticky solid; $[\alpha]_{\text{D}}^{20}$ = -59.2 $^{\circ}$ (c = 0.25, CHCl_3); IR (KBr) 3403, 2962, 1720, 1663, 1524, 1228 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.89 (d, J = 6.4 Hz, 2H), 0.97 (d, J = 6.4 Hz, 2H), 1.02 (s, 9H), 1.73-1.8 (m, 1H), 3.94 (dd, J = 8 Hz, J = 8 Hz, 1H), 4.03 (t, J = 7.8 Hz, 1H), 4.27-4.23 (m, 2H), 4.34 (d, J = 9.8 Hz, 1H), 4.40 (d, J = 6.8 Hz, 2H), 5.49 (d, J = 8.8 Hz, 1H), 7.31- 7.33 (m, 2H), 7.41 (t, J = 7.6 Hz, 2H), 7.62 (d, J = 7.3 Hz, 2H), 7.78 (d, J = 7.3 Hz, 2H); ^{13}C NMR $\{^1\text{H}\}$ (75 MHz, CDCl_3) δ 18.4, 19.0, 32.8, 35.3, 47.5, 57.9, 67.1, 70.4, 71.9, 120.2, 125.4, 127.3, 127.9, 141.5, 144.1, 144.2, 156.4, 165.9; HRMS (CI, isobutane) m/z (M+H) $^+$ calcd. 421.2491 obsd. 421.2472.

(8): Yield: 73%; white solid; mp: 100.5-102.2 $^{\circ}\text{C}$ (dec); $[\alpha]_{\text{D}}^{20}$ = -18.4 $^{\circ}$ (c = 0.38, CHCl_3); IR (KBr) 3356, 2958, 1701, 1662, 1530, 1451, 1246 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.84 (d, J = 6.3 Hz, 3H), 0.91 (d, J = 6.3 Hz, 3H), 1.66- 1.72 (m, 1H), 3.07

(dd, $J = 5.9$ Hz $J = 13.7$ Hz, 1H), 4.23 (dd, $J = 13.7$ Hz, $J = 5.4$ Hz, 1H), 3.82 (q, $J = 7.8$ Hz, 1H), 4.03 (t, $J = 8$ Hz, 1H), 4.22 (t, $J = 6.8$ Hz, 1H), 4.27-4.33 (m, 2H), 4.46 (dd, $J = 7.3$ Hz, $J = 10.3$ Hz, 1H), 4.74 (m, 1H), 4.73 (d, $J = 7.8$ Hz, 1H), 7.12 (d, $J = 6.8$ Hz, 2H), 7.26-7.33 (m, 5H), 7.41 (t, $J = 7.6$ Hz, 2H), 7.58 (dd, $J = 7.3$ Hz $J = 11.2$ Hz, 2H), 7.77 (d, $J = 7.3$ Hz, 2H); ^{13}C NMR { ^1H } (75 MHz, CDCl_3) δ 18.4, 19.0, 32.8, 38.9, 47.4, 50.4, 67.1, 71.1, 72.1, 120.2, 125.4, 127.1, 127.3, 127.9, 128.6, 129.8, 136.2, 141.6, 144.1, 144.2, 155.7, 165.2; HRMS (E.I.) m/z (M) $^+$ calcd. 454.2256 obsd. 454.2261.

(9): Yield: 75%; white solid; mp: 117.1-117.3 °C (dec); $[\alpha]_{\text{D}}^{20} = -49.5^\circ$ ($c = 0.22$, CHCl_3); IR (KBr) 3382, 2963, 2927, 1720, 1673, 1530, 1233 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.88- 0.99 (m, 6H), 1.69-1.80 (m, 1H), 2.07-2.18 (m, 1H), 3.9 -4.0 (m, 1H), 4.025 (t, $J = 7.9$ Hz, 1H), 4.23- 4.31 (m, 2H), 4.31- 4.40 (m, 3H), 5.45 (d, $J = 9.6$ Hz, 1H), 4.4 (dd, $J = 6.8$ Hz, $J = 10.1$ Hz, 1H), 7.28-7.43 (m, 1H) 7.62 (d, $J = 6.4$ Hz, 2H), 7.76 (d, $J = 7.6$ Hz, 2H); ^{13}C NMR { ^1H } (75 MHz, CDCl_3) δ 17.9, 18.5, 19.0, 19.1, 31.8, 32.9, 47.4, 54.8, 67.1, 70.9, 72.0, 120.2, 125.4, 127.3, 127.9, 141.5, 144.1, 144.2, 156.4, 166.3; HRMS (CI, isobutane) m/z ($\text{M}+\text{H}$) $^+$ calcd. 407.2335 obsd. 407.2347.

(10): Yield: 58%; white solid; mp: 119.1-123 °C (dec); $[\alpha]_{\text{D}}^{20} = -40^\circ$ ($c = 0.11$, CHCl_3); IR (KBr) 3382, 2963, 1720, 1663, 1543, 1530, 1449, 1247, 1104 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.01 (d, $J = 6.8$ Hz, 3H), 1.04 (d, $J = 6.3$ Hz, 3H), 2.24-2.18 (m, 1H), 4.18 (t, $J = 8.3$ Hz, 1H), 4.26 (t, $J = 7.1$ Hz, 1H), 4.42 (d, $J = 6.8$ Hz, 2H), 4.51 (dd, $J = 5.3$ Hz, 9.28 Hz, 1H), 4.70 (t, $J = 9.5$ Hz, 1H) 5.24 (t, $J = 9.2$ Hz, 1H), 5.51 (d, $J = 9.3$ Hz, 1H), 7.25- 7.42 (m, 9H), 7.62 (dd, $J = 7.32$ Hz, $J = 3.4$ Hz, 2H), 7.77 (d, $J = 7.8$ Hz, 2H); ^{13}C NMR { ^1H } δ 17.9, 19.2, 31.9, 47.4, 54.8, 67.2, 69.5, 75.6, 120.2, 125.38, 125.41, 126.8, 127.3, 127.9, 128.0, 128.6, 129.0, 141.5, 142.0, 144.2, 156.5, 168.0; HRMS (E.I.) m/z (M) $^+$ calcd. 440.2100 obsd. 440.2079.

(11): Yield: 78%; white solid; mp: 64.3-65.1 °C (dec); $[\alpha]_{\text{D}}^{20} = -9.6^\circ$ ($c = 0.115$, CHCl_3); IR (CHCl_3 , Salt plate) 3062, 3028, 1722, 1666, 1512, 1451 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 3.16 (dd, $J = 5.8$ Hz, $J = 13.6$ Hz, 1H), 4.23 (dd, $J = 6.0$ Hz, $J = 13.7$ Hz, 1H), 4.16 (t, $J = 8.4$ Hz, 1H), 4.23 (t, $J = 6.9$ Hz, 1H), 4.35 (dd, $J = 6.9$ Hz, $J = 10.4$ Hz, 1H), 4.50 (dd, $J = 7.4$ Hz, $J = 10.4$ Hz, 1H), 4.69 (t, $J = 8.4$ Hz, 1H), 4.90 (dd, $J = 5.9$ Hz, $J = 13.3$ Hz, 1H), 5.12 (t, $J = 9.7$ Hz, 1H), 5.28 (d, $J = 7.96$ Hz, 1H), 7.13-7.44 (m, 14H), 7.59 (t, $J = 6.34$ Hz, 2H), 7.78 (d, $J = 7.1$ Hz, 2H); ^{13}C NMR { ^1H } (75 MHz, CDCl_3) δ 38.9, 47.42, 50.5, 67.1, 69.5, 75.7, 120.2, 125.3, 125.4, 126.8, 127.3, 127.9, 128.7, 129.0, 129.8, 136.2, 141.6, 141.8, 144.0, 144.2, 155.7, 167.2; HRMS (E.I.) m/z (M) $^+$ calcd. 489.2178 obsd. 489.2113.

(12): Yield: 78%; white solid; mp: 141.9-144.8 °C (dec); $[\alpha]_{\text{D}}^{20} = +18.3^\circ$ ($c = 0.12$, CHCl_3); IR (KBr) 3406, 3063, 3031, 1722, 1665, 1496, 1450, 1236 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 4.19-4.24 (m, 2H), 4.40 (d, $J = 6.8$ Hz, 2H), 4.66 (t, $J = 9.0$ Hz, 1H), 5.24 (t, $J = 9.0$ Hz, 1H), 5.61 (d, $J = 7.3$ Hz, 1H), 6.21 (d, $J = 6.8$ Hz, 1H), 7.26-7.50 (m, 14H), 7.59 (d, $J = 7.3$ Hz, 2H), 7.77 (d, $J = 7.6$ Hz, 2H); ^{13}C NMR { ^1H } (75 MHz, CDCl_3) δ 47.4, 53.7, 67.4, 69.5,

76.1, 120.2, 125.4, 127.3, 127.9, 128.1, 128.8, 129.1, 129.2, 129.5, 137.9, 141.5, 141.7, 144.0, 144.1, 155.6, 167.3; HRMS (E.I.) m/z (M)⁺ calcd. 474.1943 obsd. 474.1953.

(13): Workup: After completion of reaction, solvent is removed under reduced pressure and the residue is stirred in 10 ml of ~0.15M K₂CO₃ in methanol. After 12h it is partitioned between 10ml of brine and 10ml of CH₂Cl₂. The aqueous layer is washed thrice with 10 ml of CH₂Cl₂. The combined organic layers are dried with Na₂SO₄ and solvent is removed under reduced pressure. Mobile phase for chromatography: 5% ethylacetate in hexanes. Yield: 68%; clear viscous oil; $[\alpha]_D^{20} = +67.1^\circ$ (c = 0.149, CHCl₃); IR (CHCl₃, Salt plate) 3064, 3032, 2967, 2927, 2869, 1642, 1617, 1492 cm⁻¹; ¹HNMR (300 MHz, CDCl₃) δ 1.5 (d, $J = 6.7$ Hz, 3H), 4.28 (dq, $J = 6.7$ Hz, $J = 6.7$ Hz, 1H), 5.10 (d, $J = 7.7$ Hz, 1H), 6.90 (dt, $J = 1$ Hz, $J = 7.6$ Hz, 1H), 7.06 (dd, $J = 0.7$ Hz, $J = 8.3$ Hz, 1H), 7.35-7.43 (m, 6H), 7.75 (dd, $J = 1.6$ Hz, $J = 7.8$ Hz, 1H), 12.19 (s, 1H); ¹³CNMR {¹H} (75 MHz, CDCl₃) δ 21.7, 69.9, 87.6, 110.9, 117.0, 118.9, 126.0, 128.4, 128.9, 129.2, 133.7, 139.9, 160.3, 164.5; HRMS (CI, isobutane) m/z (M+H)⁺ calcd. 254.1181 obsd. 254.1178.

Deprotection of oxazolines:

General Procedure for deprotection of Fmoc oxazolines:

0.1 mmol of the protected oxazoline is dissolved in 1 ml of MeOH and cooled down to 0 °C with an ice bath. 1 ml of the required base is added over 10 minutes. After completion of reaction the solvents are removed and the residue purified by a short silica column.

(15): Mobile phase for chromatography: 100 ml of 5% CH₂Cl₂/hexanes followed by 150 ml of 4% MeOH/CH₂Cl₂; Yield: 99%; clear viscous oil; $[\alpha]_D^{20} = +3.1^\circ$ (c = 0.4, CHCl₃); IR (CHCl₃, Salt plate): 2961, 2870, 1659 cm⁻¹; ¹HNMR (300 MHz, CD₃OD) δ 1.04 (s, 9H) 1.38 (d, $J = 6.6$ Hz, 3H), 3.35 (s, 1H), 3.99 (dq, $J = 6.7$ Hz, $J = 6.7$ Hz, 1H), 5.07 (d, $J = 8.0$ Hz, 1H), 7.33- 7.39 (m, 5H); ¹³CNMR {¹H} (75 MHz, CD₃OD) δ 21.5, 27.0, 35.3, 60.3, 70.6, 89.9, 127.0, 129.6, 129.9, 141.2, 169.9; HRMS (CI, isobutane) m/z (M+H)⁺ calcd. 247.1810 obsd. 247.1820.

(16): Mobile phase for chromatography: 5% MeOH/CH₂Cl₂; Yield: 85%; clear viscous oil; $[\alpha]_D^{20} = -15.8^\circ$ (c = 0.645, CHCl₃); IR (CHCl₃, Salt plate) 3371, 3314, 2963, 2928, 2871, 1662 cm⁻¹; ¹HNMR (300 MHz, CDCl₃) δ 1.0 (d, $J = 6.8$ Hz, 3H), 1.04 (d, $J = 6.8$ Hz, 3H), 1.4 (d, $J = 6.7$ Hz, 3H), 2.02-2.13 (m, 1H), 3.44 (d, $J = 5.5$ Hz, 1H), 4.04 (dq, $J = 7.3$ Hz, $J = 7.3$ Hz, 1H), 4.94 (d, $J = 8.1$ Hz, 1H), 7.29- 7.42 (m, 5H); ¹³CNMR {¹H} (75 MHz, CDCl₃) δ 17.6, 19.7, 21.6, 32.3, 55.8, 70.2, 88.8, 125.9, 128.6, 129.0, 140.4, 169.14; HRMS (CI, isobutane) m/z (M+H)⁺ calcd. 233.1654 obsd. 233.1662.

References:

1. Myers, A. I.; McKenon, M. J. *J. Org. Chem.* **1993**, 58, 3568.
2. Brunner, H.; Obermann, U. *Chem. Ber.* **1998**, 122, 499.
3. In compounds 1-6, and 8-10, the quaternary carbons next to the methylene on the Fmoc are diastereotopic and hence show up as different peaks. These two carbons show up as two different peaks in commercially available chiral Fmoc amino acids. In compound 10 an unassigned peak from the minor diastereomer shows in the aromatic region.
4. Armarego, W. L. F.; Perrin, D. D. *Purification of Laboratory Chemicals*; Ed. 4; p136.