Experimental Section

General Methods: NMR spectra were recorded using Varian Unity Plus 500 MHz, Bruker AMX-400 MHz, and GE QE Plus 300MHz spectrometers and chemical shift values are reported in parts per million (ppm) downfield from tetramethylsilane. The infrared spectra were recorded using a Perkin Elmer Spectrum 1000 FT-IR spectrometer. The mass spectral data were obtained using a Finnigan SSQ700 DCI/MS and a Finnigan SSQ7000 APCI/MS and ESI/MS. All reactions were run under an atmosphere of N_2. All reagents and solvents were used as purchased from commercial suppliers. All reactions were monitored by TLC (250μm Merck Silica Gel 60 F_{254}) and HPLC (Hewlett-Packard 1100 series, Zorbax SB C8 4.6 mm x 7.5 mm column, Flow rate 1.5 mL/min., UV detection, gradient elution 0.1v/v% H_3PO_4 in H_2O:CH_3CN). Elemental analysis and optical rotations were performed by Robertson Microlit Laboratories, Inc., 29 Samson Avenue, PO Box 927, Madison, NJ 07940.

3*S*,4*S*)-4-[(*tert*-Butyloxycarbonyl)-amino]-6-methyl-1-hepten-3-ol (7) To a solution of *N*-Boc-L-leucinol, **5**, (2.00 Kg, 9.126 moles) and 2,2,6,6-tetramethyl-1-piperidinyloxy, free radical (14.5 g, 92.7 mmol) in CH_2Cl_2 (36.6 Kg) and H_2O (5.54 Kg) at 0 °C was added 7% $NaHCO_3$ solution (28.9 Kg) and 12.6% NaOCl solution (6.62 Kg) over 2 h with vigorous stirring. The biphasic mixture was stirred for 20 min. then the layers were separated. The organic layer was washed sequentially with 10% $NaHSO_4$ solution (25.7 Kg) containing NaI (151 g), 10% $Na_2S_2O_3$ (28.7 Kg), 7% $NaHCO_3$ (28.5 Kg) and brine (21.7 Kg). The organic layer was dried over Na_2SO_4, filtered, and concentrated to 3.80 Kg of **6** in CH_2Cl_2 (a small portion was concentrated to dryness for characterization $[\alpha]^{25}_D$ -32.4 (c 1.0, MeOH); IR (neat) 3346, 2955, 1731, 1713, 1694, 1515, 1363, 1249, 1166, 1054, 1017cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.43 (s, 1H), 7.22 (d, *J*=7.8 Hz, 1H), 3.86 (ddd, *J*=11.0, *J*=7.8, *J*=4.7 Hz, 1H), 1.65 (sept., *J*=6.8 Hz, 1H), 1.34-1.48 (m, 2H), 1.39 (s, 9H), 0.89 (d, *J*=6.8 Hz, 3H), 0.86 (d, *J*=6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 202.0, 155.7, 78.2, 57.8, 36.5, 28.1, 24.0, 22.9, 21.3; Analytical calcd for $C_{11}H_{21}NO_3$: C, 61.37; H, 9.83; N, 6.51 Found: C, 61.16; H, 9.77; N, 6.43. The ee of **6** was determined see below in supplementary addendum.

To a solution of vinyl magnesium chloride in THF (17.5 wt%, 13.8 Kg, 27.9 moles) was added CH_2Cl_2 (18.8 Kg). The resulting solution was cooled to 10 °C and treated with *N*-Boc-L-leucinal **6 /** CH_2Cl_2 (3.80 Kg) while maintaining an internal temperature < 30^{o}C. The reaction was stirred for 25 min. then poured into a stirring solution of 15% NH_4Cl (51.9 Kg) and diluted with MTBE (25.6 Kg). Layers were separated and the organic layer washed with brine (26.9 Kg). The organic layer was concentrated to approximately 6 liters, whereupon heptane 20 Kg was added and concentrated to 3.65 kg, cooled to -20 °C (overnight), filtered and washed with cold heptane (1.2 Kg) to afford 1.48 Kg of off-white solid as a mixture of **7** (1.28 Kg 58 % overall) and the undesired isomer **13** (0.20 Kg, 9.0%). **(3*S*,4*S*)-4-[(*tert*-Butyloxycarbonyl)-amino]-6-methyl-1-hepten-3-ol (7).** TLC: 2:1 heptane:EtOAc; R_f=0.42; $[\alpha]^{25}_D$ -0.27.3 (c 1.01, MeOH); IR (neat) 3383, 2955, 2868, 1688, 1506, 1367, 1169, 919 cm-1; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.89 (ddd, *J*=17.3, 10.7, 5.9 Hz, 1H), 5.29 (ddd, *J*=17.3, 1.5, 1.5 Hz, 1 H), 5.18 (ddd, *J*=10.7, 1.5, 1.5 Hz, 1H), 4.68 (br d, 1H), 4.06 (br s, 1H), 3.66 (br m, 1H), 2.60 (br s, 1H), 1.68 (hept, *J*=6.6 Hz, 1H), 1.43 (s, 9H), 1.71-1.35 (m, 2H), 0.92 (d, *J*=6.6 Hz, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.3, 138.2, 116.1, 75.1, 52.9, 40.7, 28.3, 24.8, 23.3, 21.9; MS $C_{13}H_{25}NO_3$ m/z 243, DCI/NH3$^+$ [M+1] 244, ESI$^+$ [M+1] 244, ESI$^-$ [M-1] 242; Analytical calcd for $C_{13}H_{25}NO_3$: C, 64.16; H, 10.36; N, 5.76 Found: C,64.00; H,10.25; N,5.65.
For the determination of the ee% of **7** see the supplementary material addendum.

(3*R*,4*S*)-4-[(*tert*-Butyloxycarbonyl)-amino]-6-methyl-1-hepten-3-ol (8). TLC: 2:1 heptane:EtOAc; R_f=0.33; $[\alpha]^{25}_D$ -37.9 (c 9.54, MeOH); IR (neat) 3358, 2949, 1682, 1527 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.82 (ddd, *J*=17.3, 10.6, 5.5 Hz, 1H), 5.31 (ddd, *J*=17.3, 1.5,1.5 Hz, 1 H), 5.21 (ddd, *J*=10.6, 1.5, 1.5 Hz, 1H), 4.53-4.58 (br m, 1H), 4.17 (br s, 1H), 3.85-3.74 (br m, 1H), 3.14 (br s, 1H), 1.70-1.60 (m, 1H), 1.43 (s, 9H), 1.27-1.21 (m, 2H), 0.91 (d, *J*=5.5 Hz, 3H), 0.89 (d, *J*=5.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.8, 136.7, 116.5, 75.9, 53.5, 39.1, 28.3, 24.7, 23.4, 21.7; MS (APCI$^+$) $C_{13}H_{25}NO_3$ m/z 243 (relative intensity), 244 (M+1, 85) 244, (100) 188, (47) 170, 144 (90); Analytical calcd for $C_{13}H_{25}NO_3$: C, 64.16; H, 10.36; N, 5.76 Found: C, 63.91; H, 10.36; N, 5.85.

(4*S*,5*S*)-3-(*tert*-Butyloxycarbonyl)-2,2-dimethyl-5-ethenyl-4-(2-methylpropyl)oxazolidine (9). To a solution of allylic alcohols **12** and **13** (20.5 g, 84.4 mmol, **7**:**8**, 6:1; Assay for **7**, 17.6 g, 72.3 mmol) in

CH_2Cl_2 (200 mL) was added pTSA (0.64 g, 3.4 mmol). The resulting mixture was cooled to -20 °C then added 2,2-dimethoxypropane (35.4 g, 340 mmol). The reaction was stirred at -20 °C for 4 h. While maintaining temperature at -20 °C, Et_3N (30.4 g, 300 mmol), succinic anhydride (25.1 g, 251 mmol), and DMAP (15.8 g, 129 mmol) were added to the reaction mixture. The reaction was then allowed to warm to ambient temperature. The reaction was quenched with MeOH (10.8 mL) and stirred for 30 min. The volatiles were removed by concentration and then diluted with heptane (210 mL), MTBE (55 mL), and a 5% NaH_2PO_4 solution (500 mL). The layers were separated and the organic layer was washed with a mixture of 5% $NaHCO_3$ solution (450 mL) and methanol (75 mL). The layers were separated and the remaining organic layer was washed with brine (50 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated to give **9** (18.6 g, 91% yield based on starting alcohol **7**) as a viscous oil. TLC: 2:1 heptane:EtOAc; **9**, R_f=0.56; $[\alpha]^{25}_D$ +17.8 (c 6.09, MeOH); IR (neat) 2950, 1702, 1460, 1385, 1178 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz) δ 5.97 (ddd, *J*=17.3, 10.3, 7.4 Hz, 1H), 5.32 (dt, *J*=17.3, 1.1 Hz, 1H), 5.19 (dt, *J*=10.3, 1.1 Hz, 1H), 4.29 (dd, *J*=7.4, 3.6 Hz, 1H), 3.66 (br d, *J*=8.5 Hz, 1H), 1.68-1.53 (m, 2H), 1.50 (s, 3H), 1.42 (s, 3H), 1.41 (s, 9H), 1.40-1.35 (m, 1H), 0.88 (dd, *J*=9.7, 6.1 Hz, 6H); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 150.8, 138.8, 117.2, 93.7, 81.2, 79.3, 59.8, 28.3, 27.5, 24.8, 23.9, 21.4; MS (APCI$^+$) $C_{16}H_{29}NO_3$ m/z (relative intensity) 284 (M+1, 11), 184 (100); Analytical calcd for $C_{16}H_{29}NO_3$: C, 67.90; H, 10.33; N, 4.95. Found: C, 67.66; H, 10.33; N, 4.83.

(4*S*,5*R*)-3-(*tert*-Butyloxycarbonyl)-2,2-dimethyl-4-(2-methylpropyl)- oxazolidine-5-carboxylic acid (4). To a suspension of RuO_2xH_2O (0.42 g, 3.1 mmol), acetone (17 mL) and water (17 mL) at 0 °C was added $NaIO_4$ (2.90 g, 13.6 mmol) in H_2O (20 mL). After 5 min. the vinyl oxazolidine **9** (12.0 g, 41.6 mmol) in acetone (375 mL) was added. Next $NaIO_4$ (55.2 g, 256 mmol) in H_2O (355 mL) was added dropwise over 3 h keeping the temperature below 15 °C. After 1 h of additional stirring the reaction was cautiously quenched with *iso*-propanol (21 mL) and stirred for 30 minutes while the reaction warmed to ambient temperature. The reaction was then filtered though Celite and the resulting filter cake washed with a 1:1 acetone:water solution (300 mL). NaCl (20 g, 342 mmol) was added to the combined filtrates and then extracted with MTBE (500 mL). The organic extract was washed with a 1:1 mixture of 1 M $NaHSO_4$ and 1 M $NaHSO_3$ (190 mL total). The organic layer was then extracted with 0.5 M K_2CO_3 aq. solution (380 mL). The alkaline aqueous layer was pH adjusted to pH 5 with portion wise addition of a concentrated citric acid solution (47 g in 100 mL H_2O). The acidic aqueous layer was extracted with MTBE (360 mL) and the organic layer was washed with brine (90 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated to give **4** (10.6 g, 85%). TLC: 2:1 heptane:EtOAc; R_f=0. 12; mp 91-93.5 ^{o}C, $[\alpha]^{25}_D$ -7.4 (c 10.61, $CHCl_3$); IR (neat) 3500 (br), 1704, 1381 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz) δ 13.0 (br s, 1H), 4.35 (d, *J*=1.5 Hz, 1H), 4.22-4.06 (br m, 1H), 1.80-1.55 (m, 2H), 1.51 (s, 3H), 1.47 (s, 3H), 1.41 (s, 9H), 1.40-1.32 (m, 1H), 0.96 (d, *J*=6.7 Hz, 6H); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 172.9, 150.3, 94.8, 79.2, 77.9, 58.2, 43.4, 28.1, 25.9, 25.0, 23.6, 21.1; MS (ESI$^-$) $C_{15}H_{27}NO_5$ m/z 301 (relative intensity) 301 (M, 15), 300 (M-1, 100); Analytical calcd for $C_{15}H_{27}NO_5$: C, 59.77; H, 9.03; N, 4.65. Found: C, 59.46; H, 8.99; N, 4.66.

***Iso*-propyl (2*R*,3*S*)-3-amino-2-hydroxy-5-methylhexanoate (13).** To a solution of the oxazolidine acid **16** (16.0 g, 53 mmol) in *iso*-propanol (95 mL) was added $MeSO_3H$ (6.5 g, 68 mmol) and the resulting solution was heated to 73 °C for 4 h. The reaction was then cooled to ambient temperature and stirred for 1 h. Water (27 mL) was added to the reaction which was stirred for an additional 3 h. The reaction mixture was concentrated to a low volume then diluted with isopropyl acetate (350 mL). The isopropyl acetate solution was washed with 2 M K_2CO_3 solution (45 mL). The organic extract was dried over Na_2SO_4, filtered, and concentrated to give **13** (10.3 g, 96%). mp 54.6-55.8 ^{o}C; $[\alpha]^{25}_D$ -13.6 (c 5.27, $CHCl_3$); IR (neat) 3085 (br), 1731, 1592, 1462, 1378, 1208; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.20 (hept, *J*=6.3 Hz, 1H), 4.07 (d, *J*=2.6 Hz, 1H), 3.22 (td, *J*=7.0, 2.6 Hz, 1H), 2.33 (br s, 3H), 1.81 (hept, *J*=6.6 Hz, 1H), 1.46 (t, *J*=7.0 Hz, 2H), 1.37 (s, 3H), 1.35 (s, 3H), 1.02 (d, *J*=6.6 Hz, 3H), 0.99 (d, *J*=6.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 175.3, 75.1, 70.8, 52.9, 44.9, 26.1, 24.5, 23.5, 23.2; MS (APCI$^+$) $C_{10}H_{21}NO_5$ m/z 245 (M+42, 32) 204 (M+1, 100); Analytical calcd for $C_{10}H_{21}NO_5$: C, 59.08; H, 10.41; N, 6.89. Found: C, 58.70; H, 10.32; N, 6.80.

(3R, 4S)-bis-N,O-triethylsilyl-4-(2-methylpropyl)-3-hydroxy-azetidin-2-one (15b). To a solution of isopropyl (*2R,3S*)-3-amino-2-hydroxy-5-methylhexanoate (**13**) (10.0 g, 49.3 mmol) in MTBE (200 mL) was added NEt_3 (13.3 g, 131 mmol). The mixture then was cooled to 0 °C and treated with TESCl (17.1

g, 113 mmol). The resulting white slurry was stirred at 23 °C for 1.5 h. The mixture was cooled to 0 °C and treated dropwise with *t*-butylmagnesium chloride (197 mL, 1 M solution in THF, 197 mmol). The reaction was stirred at 23 °C for 1 h during which it became thick slurry. The reaction mixture was poured into 15% NH_4Cl solution (735 mL) and diluted with MTBE (375 mL). The layers were separated and the organic layer washed with brine (212 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated to afford the crude bis-TES lactam **15b** as a brown oil (23.4 g, 78% pure by HPLC which indicates 18.3 g, 49.3 mmol). The crude **15b** was used directly in the next step. TLC: EtOAc/hexane (1:4), R_f=0.82; $[\alpha]^{25}_D$ +83.14 (c 10.021, $CHCl_3$); IR (neat) 2956, 1749, 1194, 1017 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.82 (d, *J*= 5.1 Hz, 1H), 3.62 (ddd, *J*= 10.5, 5.3, 3.3 Hz, 1 H), 1.77 (ddd, *J*= 13.2, 10.2, 4.5 Hz, 1H), 1.67-1.57 (m, 1H), 1.28 (ddd, *J*= 13.2, 9.0, 3.0 Hz, 1H), 0.98-0.91 (m, 18 H), 0.90 (d, *J*= 6.6 Hz, 3H), 0.88 (d, *J*= 6.6 Hz, 3H), 0.76-0.61 (m, 12 H); ^{13}C NMR (CDCl3, 75 MHz) δ 176.8, 79.0, 57.1, 41.8, 27.2, 25.6, 23.9, 8.64, 8.56, 6.7, 5.6; MS (ESI^+) $C_{19}H_{41}NO_2Si_2$ m/z (relative intensity) 372 (MH^+, 100), 342 (5); Analytical calcd for $C_{19}H_{41}NO_2Si_2$: C, 61.39, H, 11.12; Found: C, 61.27, H, 11.28.

(3R, 4S)-3-triethylsilyloxy-4-(2-methylpropyl)-azetidin-2-one (16b). To a solution of crude bis-TES lactam **15b** (23.4 g crude at 78% purity gives 18.3 g, 49.3 mmol) in EtOAc (250 mL) at -10 °C was added 0.7 % KF in EtOH (16 mL) then the reaction mixture was stirred at -10 °C for 4 h. The reaction mixture was diluted with 2% NaCl solution (100 mL) and the layers separated. The organic layer was diluted with hexane (130 mL) and washed sequentially with 5% NaH_2PO_4 (50 mL), 7% $NaHCO_3$ (50 mL) and brine (50 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated to afford 21.0 g of the crude mono-TES lactam **16b** (21.0 g, 60% pure by HPLC (contaminated with ethoxytriethylsilane) which indicates 12.7 g, 49.3 mmol). The crude **16b** was used directly in the next step. TLC EtOAc/hexane (1:4), R_f=0.15; $[\alpha]^{25}_D$ +33.60 (c 10.93, $CHCl_3$); IR (neat) 3228, 2957, 1760, 1182 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.77 (bs, 1H), 4.81 (dd, *J*= 4.8, 2.7 Hz, 1H), 3.71 (ddd, *J*=7.6, 5.7, 5.0 Hz, 1H), 1.70-1.57 (m, 1H), 1.37-1.34 (m, 2H), 0.95 (t, *J*= 7.8 Hz, 9H), 0.92 (d, *J*= 6.3 Hz, 3H), 0.89 (d, *J*= 6.6 Hz, 3H), 0.68-0.59 (m, 6H); ^{13}C NMR (CDCl3, 75 MHz) δ 170.3, 77.2, 54.1, 38.7, 25.3, 23.0, 22.3, 6.5, 4.6; MS (ESI^-) $C_{13}H_{27}NO_2Si$ m/z (relative intensity) 256 (M^+ - H, 100); Analytical calcd for $C_{13}H_{27}NO_2Si$: C, 60.65, H, 10.57; Found: C, 60.45, H, 10.59.

(3*R*,4*S*)-1-(*tert*-Butyloxycarbonyl)-3-triethylsilyloxy-4-(2-methylpropyl)azetidin-2-one (2b). To a solution of mono-TES lactam **16b** (21.0 g, 60% pure by HPLC indicates 12.7 g, 49.3 mmol) in CH_2Cl_2 (80 mL) at 23 °C was added NEt_3 (14.6 g, 144 mmol), a solution of di-*tert*-butyl dicarbonate (16.1 g, 73.9 mmol) in CH_2Cl_2 (15 mL) and DMAP (3.31 g, 27.1 mmol). The reaction mixture was stirred at 23 °C for 1.5 h. The reaction was diluted with hexane (90 mL) and washed sequentially with 5% NaH_2PO_4 (3 x 255 mL), 7% $NaHCO_3$ (120 mL) and brine (180 mL). The organic layer was dried over Na_2SO_4 and concentrated to give the crude Boc lactam **2b** (22.0 g crude oil, 74% pure by HPLC indicates 16.3 g, 91% yield over 3 steps). The major impurity (~ 20 wt. %) is the by-product ethoxytriethylsilane carried over from the previous desilylation step. TLC EtOAc/hexane (1:4), R_f=0.67; $[\alpha]^{25}_D$ +79.19 (c 10.07, $CHCl_3$); IR (neat) 2957, 1805, 1731, 1330 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.83 (d, *J*= 5.7 Hz, 1H), 4.05 (q, *J*= 6.1 Hz, 1H), 1.78-1.64 (m, 3H), 1.49 (s, 9H), 0.96 (t, *J*= 7.8 Hz, 9H), 0.94 (d, *J*= 6.3 Hz, 3H), 0.93 (d, *J*= 5.9 Hz, 3H), 0.70-0.62 (m, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 166.4, 148.4, 83.1, 75.7, 57.0, 36.5, 28.3, 28.0, 25.1, 23.0, 6.5, 4.6; MS (ESI^+) $C_{18}H_{35}NO_4Si$ m/z (relative intensity) 375 (M^+ + NH_4, 100); Analytical calcd for $C_{18}H_{35}NO_4Si$: C, 60.46, H, 9.87; Found: C, 60.57, H, 10.10.

SUPPLEMENTAL MATERIAL ADDENDUM:

Determination of ee% of **6**. To a solution of aldehyde **6** (100 mg, 0.465 mmol) in THF (2 mL) at 0 °C was added dropwise a solution of lithium aluminum hydride in THF (1M, 0.51 mL, 0.51 mmol). The reaction mixture was stirred at room temperature for 1 h, then quenched slowly with MeOH followed by the addition of 15% NH_4Cl solution and the dilution with Et_2O. The layers were separated and the organic layer washed with brine and dried over Na_2SO_4. Concentration of the organic layer *in vacuo* gave 95 mg of the corresponding alcohol. The alcohol (95 mg, 0.438 mmol) was dissolved in CH_2Cl_2 (2 mL). Pyridine (0.071 mL, 0.876 mmol) was added followed by 3,5-dinitrobenzoyl chloride (121 mg, 0.525 mmol). The mixture was stirred at room temperature for 3 h, then quenched with unsym. dimethylethylenediamine (0.2 mL) and diluted with Et_2O (4 mL). The mixture was washed with 10% $KHSO_4$ (3 mL), 7% $NaHCO_3$ (3 mL) and brine (3 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo* to give an oil which was purified using column chromatography on silica gel eluting with EtOAc/hexane (1:9) solvent system to give 142 mg of pure 3,5-dinitrobenzoate. Chiral HPLC assay of the dinitrobenzoate indicates >99% ee.

Chiral HPLC assay: Pirkle Covalent D-naphthylalanine HiChrom HPLC Column, 25 cm x 4.6 mm; UV detection at 210 nm, 35 ^{o}C; Solvents: 90%hexane/10%IPA, 1.5 mL/min. run time: 17.5 min, Retention time: desired enantiomer 15.3 min, undesired enantiomer 12.5 min.

See 63176-142

```
Data file   :                        D:\HPCHEM\1\DATA\MIXVINOH.D
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mixture enantiomers of N-bocleucinol (as 3,5-dinitroben
zoates)                                                        ->
Injection Date   : Tue, 12. Jan. 1999          Inj. Vol.   :
Sample Name      : mixvinylalcohols            (modified after loading)
Acq. Method      : CHIRAL.M
Analysis Method : D:\HPCHEM\1\METHODS\CHIRAL.M
90/10 hex / ipa 1.5 ml min chiral assay
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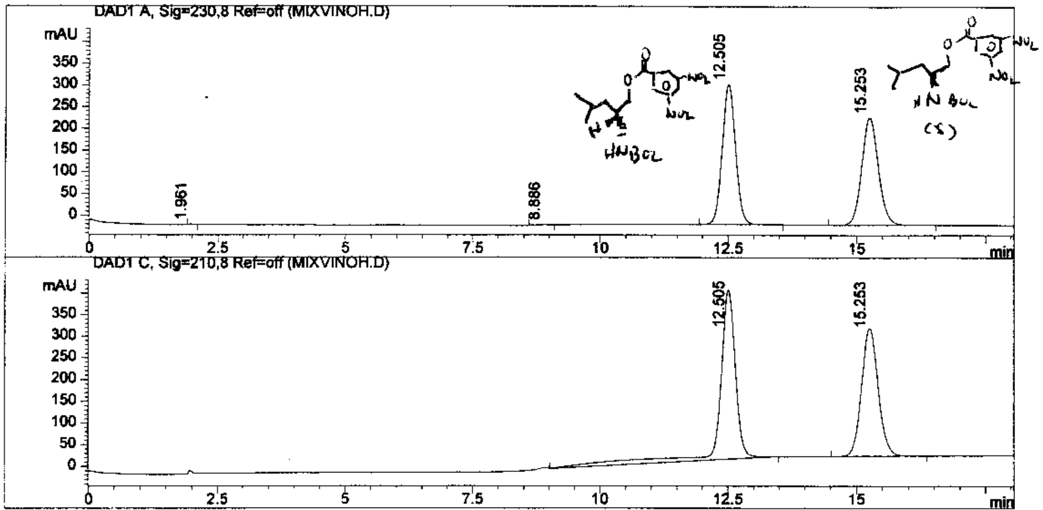

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Customized Report:


 Signal   1: DAD1 A, Sig=230,8 Ref=off
       Compound           Peak  RT     Height   Width  Amount    Area     Area %
  -------------------- ---- ------- ------- ------- ------- --------- -------
  -------------------- ---- ------- ------- ------- ------- --------- -------
                           1   1.961   1.222   0.066   0.000     5.410   0.048
                           2   8.886   2.037   0.182   0.000    24.471   0.215
                           3  12.505 323.739   0.275   0.000  5.754e3   50.516
                           4  15.253 248.232   0.349   0.000  5.606e3   49.222

 Signal   2: DAD1 C, Sig=210,8 Ref=off
       Compound           Peak  RT     Height   Width  Amount    Area     Area %
  -------------------- ---- ------- ------- ------- ------- --------- -------
  -------------------- ---- ------- ------- ------- ------- --------- -------
                           1  12.505 391.857   0.327   0.000  8.680e3   56.461
                           2  15.253 296.463   0.347   0.000  6.694e3   43.539

                        *** End of Report ***
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Column D-Napthalamine Flow = 1.5ml/min

λ = 210/230

Col temp = 35.0°C

90/10 hexane / ipA

1mg/ml injection @ 5ul injection

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Data file  :                      D:\HPCHEM\1\DATA\RALDNB.D
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chiral chrom 1 mg/ml boc leu (r) dinitrobenzoate std

Injection Date  : Mon, 11. Jan. 1999          Inj. Vol.  :
Sample Name     : ralcohol                    (modified after loading)
Acq. Method     : CHIRAL.M
Analysis Method : D:\HPCHEM\1\METHODS\CHIRAL.M
90/10 hex / ipa 1.5 ml min chiral assay
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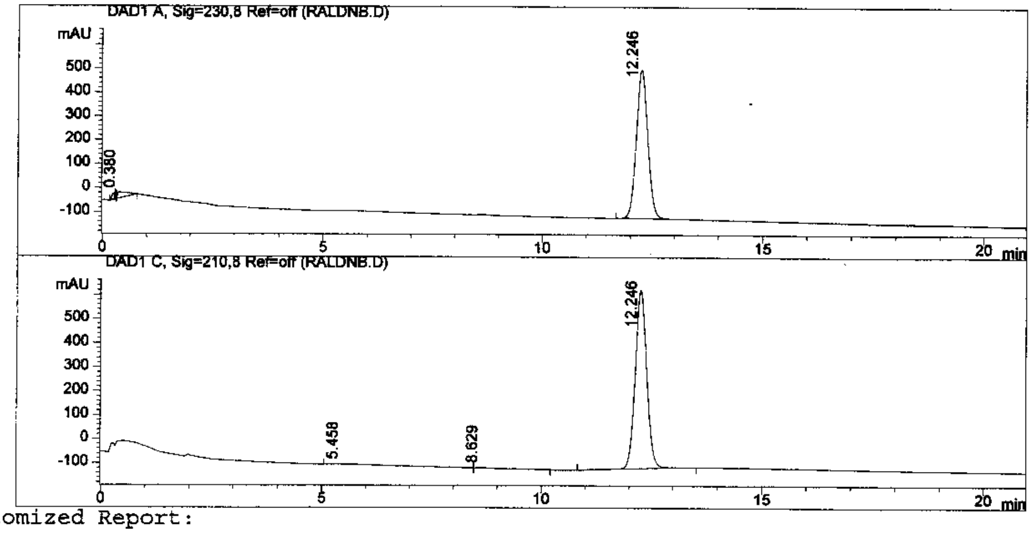

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Customized Report:

 Signal   1: DAD1 A, Sig=230,8 Ref=off
      Compound            Peak   RT    Height   Width  Amount    Area     Area %
 -------------------- ---- ------- ------- ------- ------- --------- -------
 -------------------- ---- ------- ------- ------- ------- --------- -------
                         1   0.256  27.275   0.063   0.000   110.487   0.948
                         2   0.291  22.248   0.036   0.000    48.651   0.418
                         3   0.380  25.702   0.194   0.000   398.417   3.420
                         4  12.246 623.701   0.276   0.000  1.109e4   95.215

 Signal   2: DAD1 C, Sig=210,8 Ref=off
      Compound            Peak   RT    Height   Width  Amount    Area     Area %
 -------------------- ---- ------- ------- ------- ------- --------- -------
 -------------------- ---- ------- ------- ------- ------- --------- -------
                         1   5.458   3.557   0.927   0.000   271.864   1.972
                         2   8.629   3.061   0.102   0.000    16.336   0.119
                         3  12.246 745.066   0.279   0.000  1.350e4   97.909
                     *** End of Report ***
```

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Data file  :                          D:\HPCHEM\1\DATA\SALDNB.D
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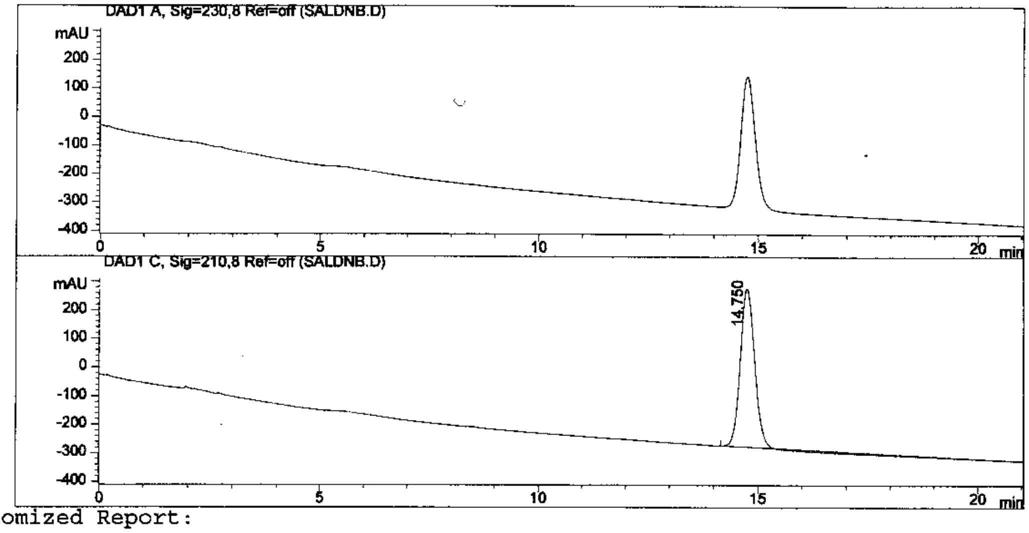
chiral chrom 1 mg/ml boc leu (s) dinitrobenzoate std

Injection Date  : Mon, 11. Jan. 1999          Inj. Vol.  :
Sample Name     : salcohol                    (modified after loading)
Acq. Method     : CHIRAL.M
Analysis Method : D:\HPCHEM\1\METHODS\CHIRAL.M
90/10 hex / ipa 1.5 ml min chiral assay
```



```
Customized Report:


 Signal  1: DAD1 A, Sig=230,8 Ref=off
        Compound          Peak   RT    Height   Width  Amount    Area     Area %
  -------------------- ---- ------- ------- ------- ------- --------- -------
  -------------------- ---- ------- ------- ------- ------- --------- -------


 Signal  2: DAD1 C, Sig=210,8 Ref=off
        Compound          Peak   RT    Height   Width  Amount    Area     Area %
  -------------------- ---- ------- ------- ------- ------- --------- -------
  -------------------- ---- ------- ------- ------- ------- --------- -------
                            1  14.750 552.795   0.327   0.000 1.147e4 100.000

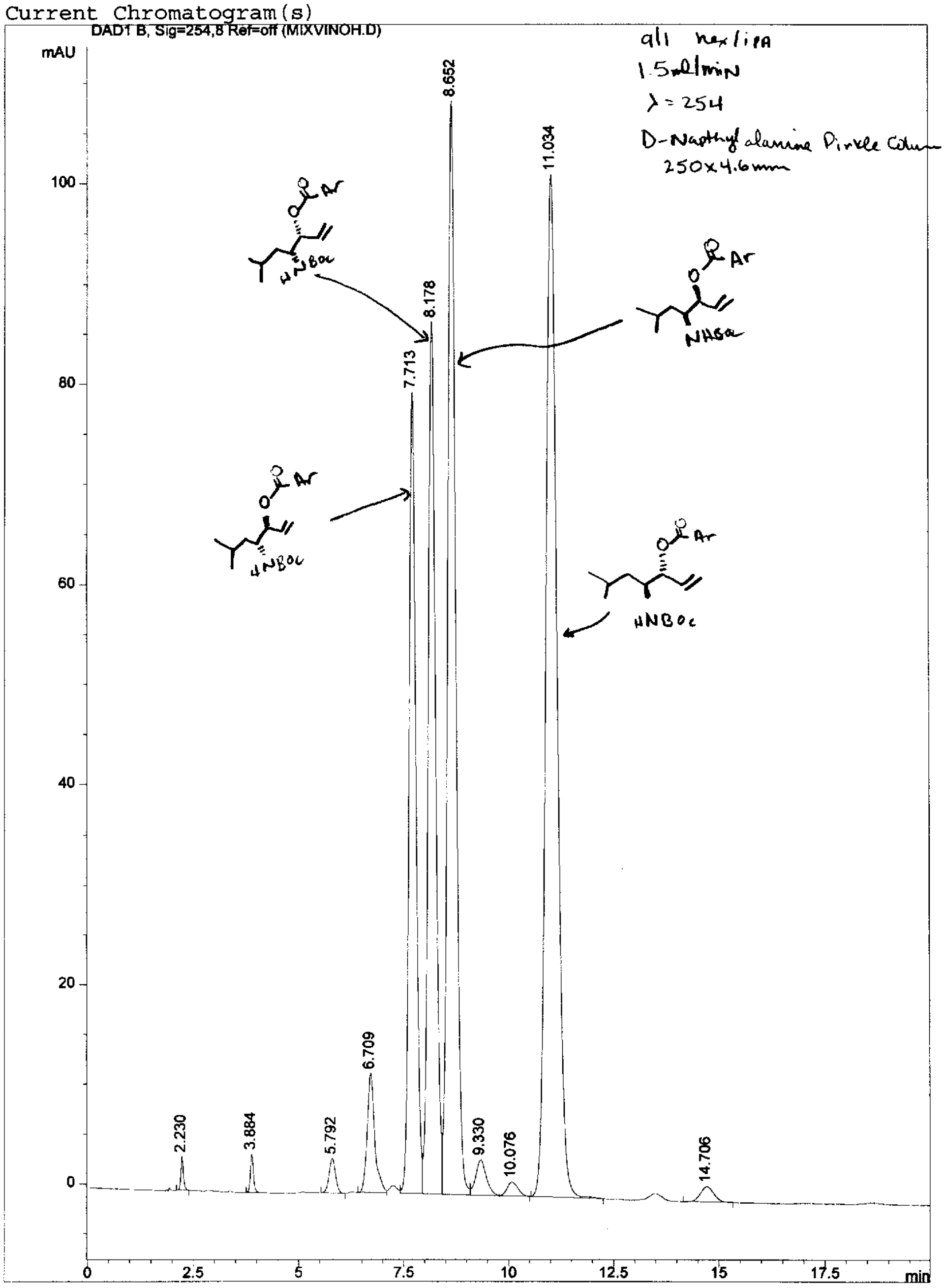
                        *** End of Report ***
```

Determination of ee% of **7**. To a solution of **7** (0.06 g, 0.25 mmol) in CH_2Cl_2 (2 mL) was added pyridine (0.05 mL, 0.62 mmol) , followed by 3,5-dinitrobenzoyl chloride (0.085 g, 0.37 mmol). The mixture was stirred at room temperature for 1 h, then quenched with unsym. dimethylethylenediamine (0.1 mL) and diluted with Et_2O (4 mL). The mixture was washed with 10% $KHSO_4$ (5 mL), followed by 5% KH_2PO_4 (5 mL) and brine (5 mL). The organic layer was dried over Na_2SO_4 and concentrated to give 105 mg of 3,5-dinitrobenzoate of 12. Chiral HPLC assay of the dinitrobenzoate indicates > 99% ee.

Chiral HPLC assay: Pirkle Covalent D-naphthylalanine HiChrom HPLC Column, 25 cm x 4.6 mm; UV detection at 235 nm, 35 ^{o}C; Solvents: 90% heptane/10% IPA, 1.5 mL/min. Retention time: 8.8 min.

Print of window 38: Current Chromatogram(s)

Current Chromatogram(s)

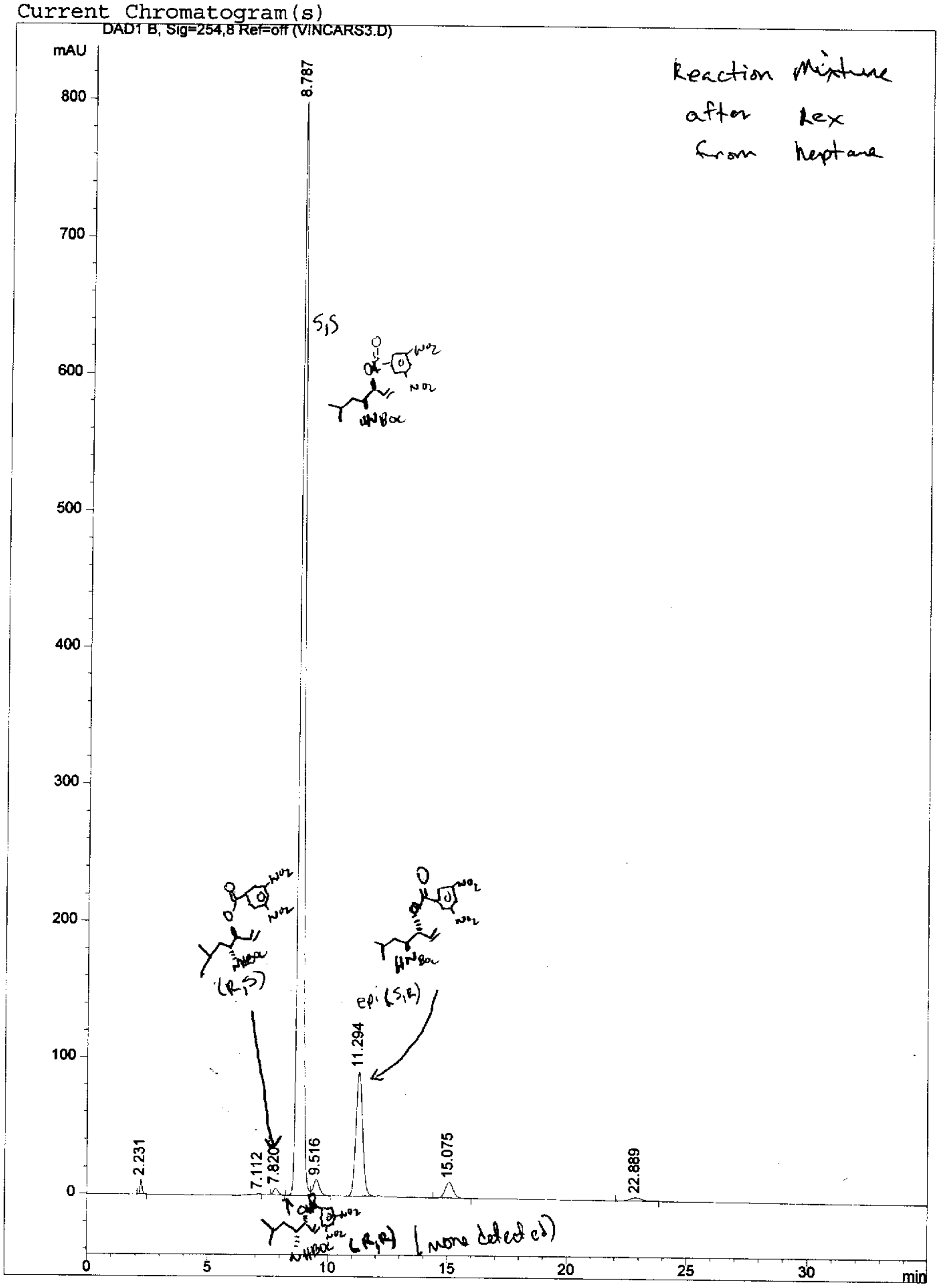


9/1 hex/iPA
1.5 ml/min
λ = 254
D-Napthyl alanine Pirkle Column
250x4.6mm

Instrument 1 1/12/99 12:05:19 PM MRL and Stand-ins

Print of window 38: Current Chromatogram(s)

Current Chromatogram(s)

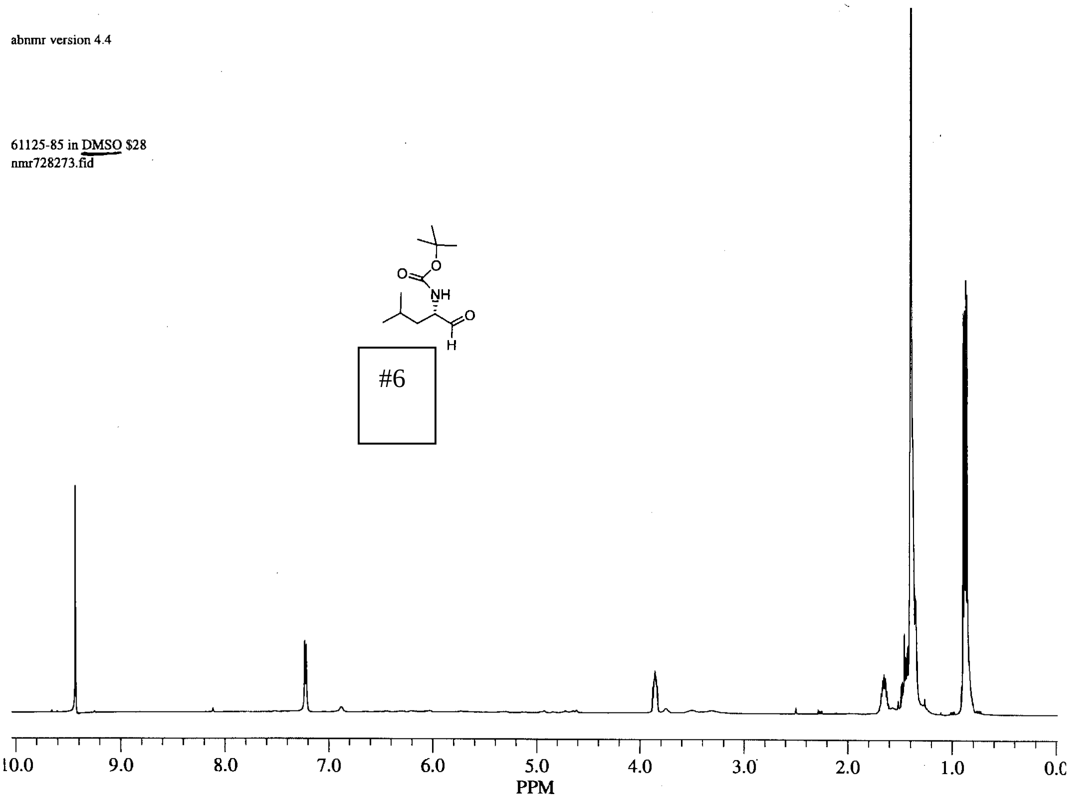


Reaction Mixture
after Rex
from heptane

Instrument 1 1/12/99 10:39:59 AM MRL and Stand-ins

abnmr version 4.4

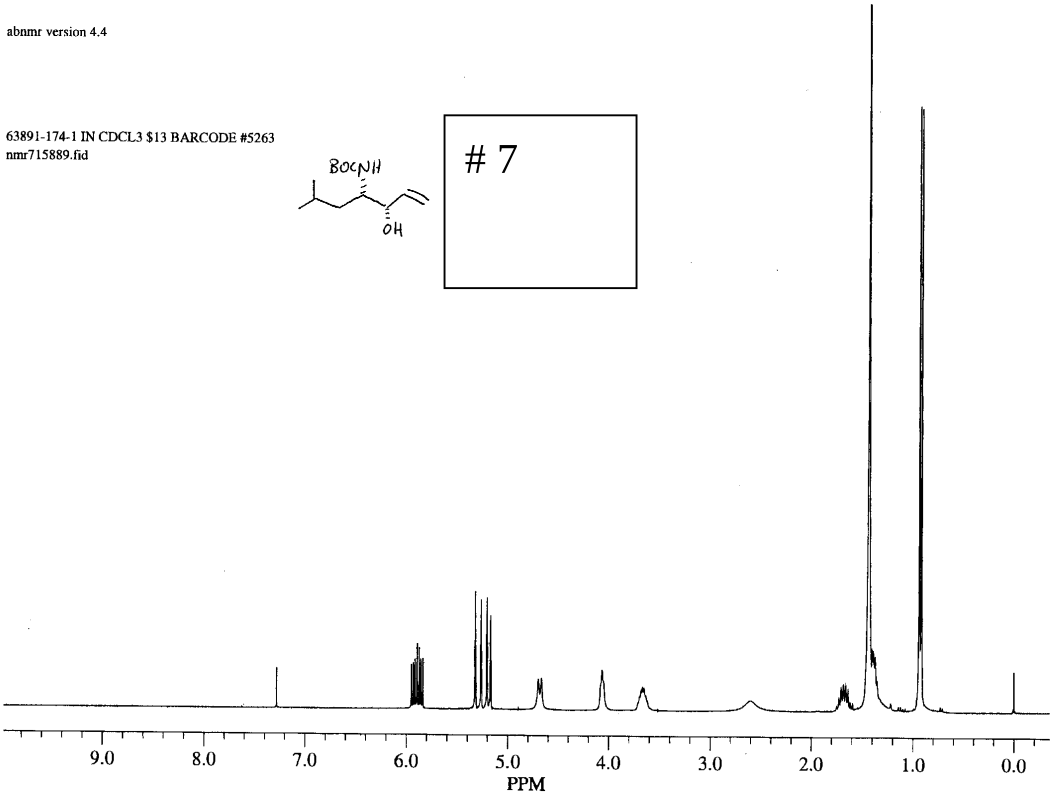
61125-85 in DMSO $28
nmr728273.fid



#6

abnmr version 4.4

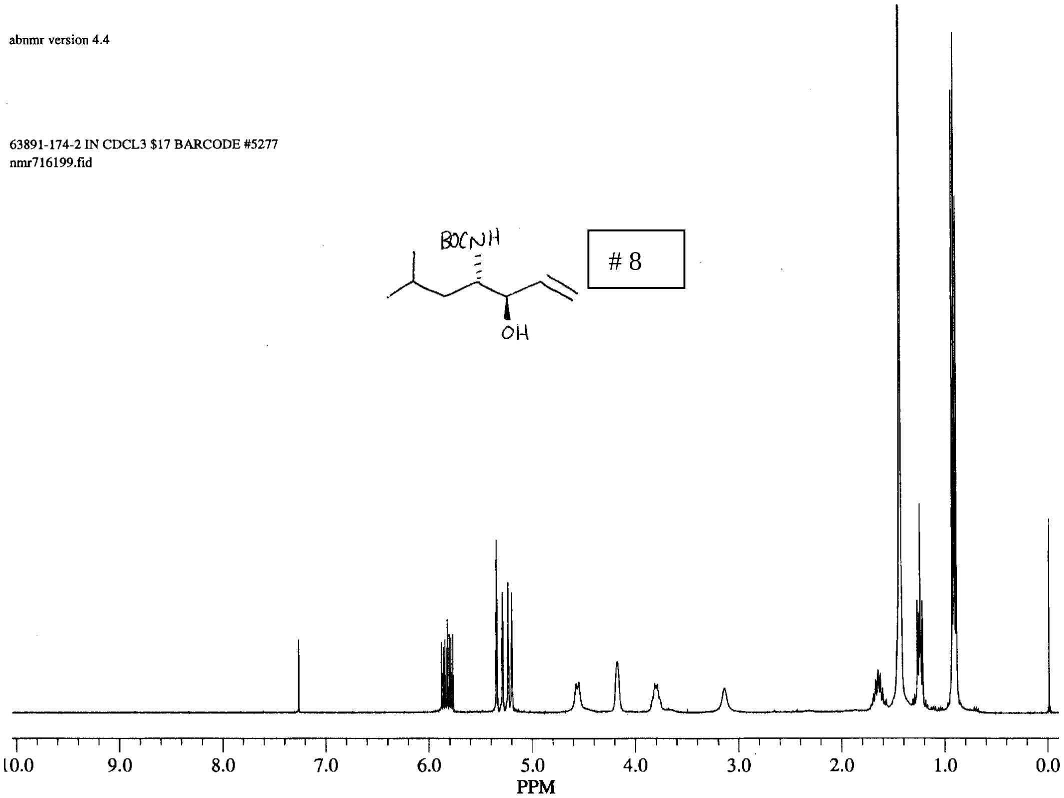
63891-174-1 IN CDCL3 $13 BARCODE #5263
nmr715889.fid



7

abnmr version 4.4

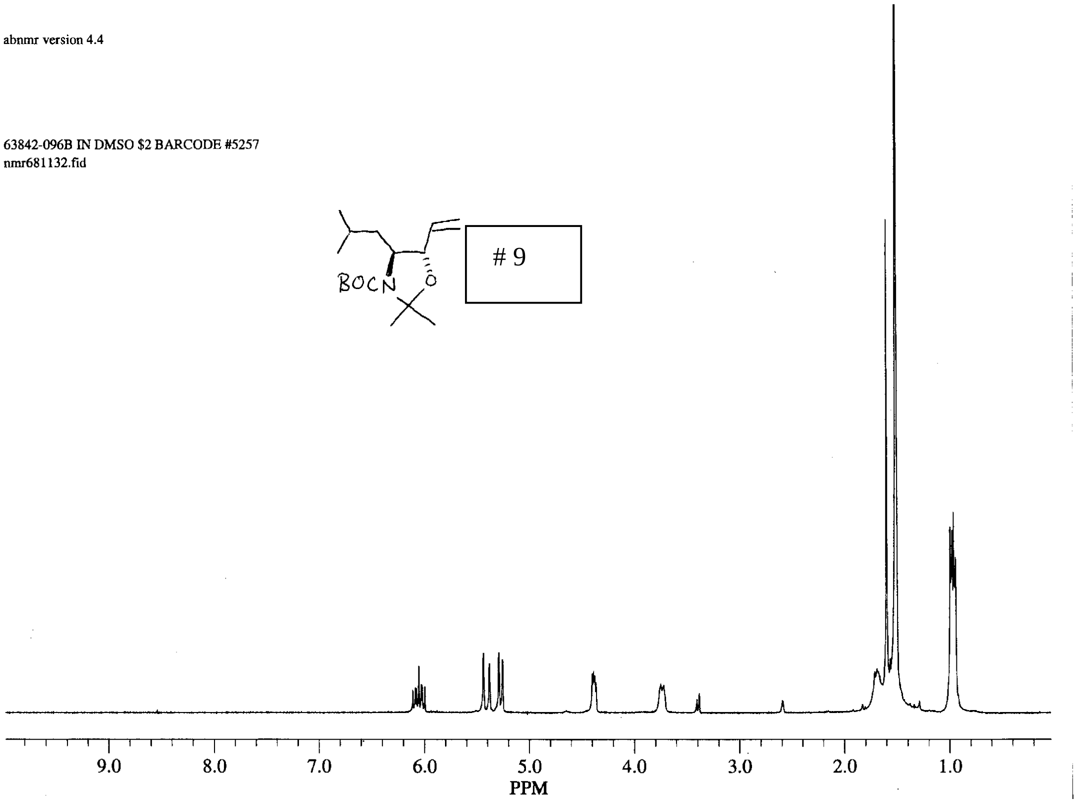
63891-174-2 IN CDCL3 $17 BARCODE #5277
nmr716199.fid



8

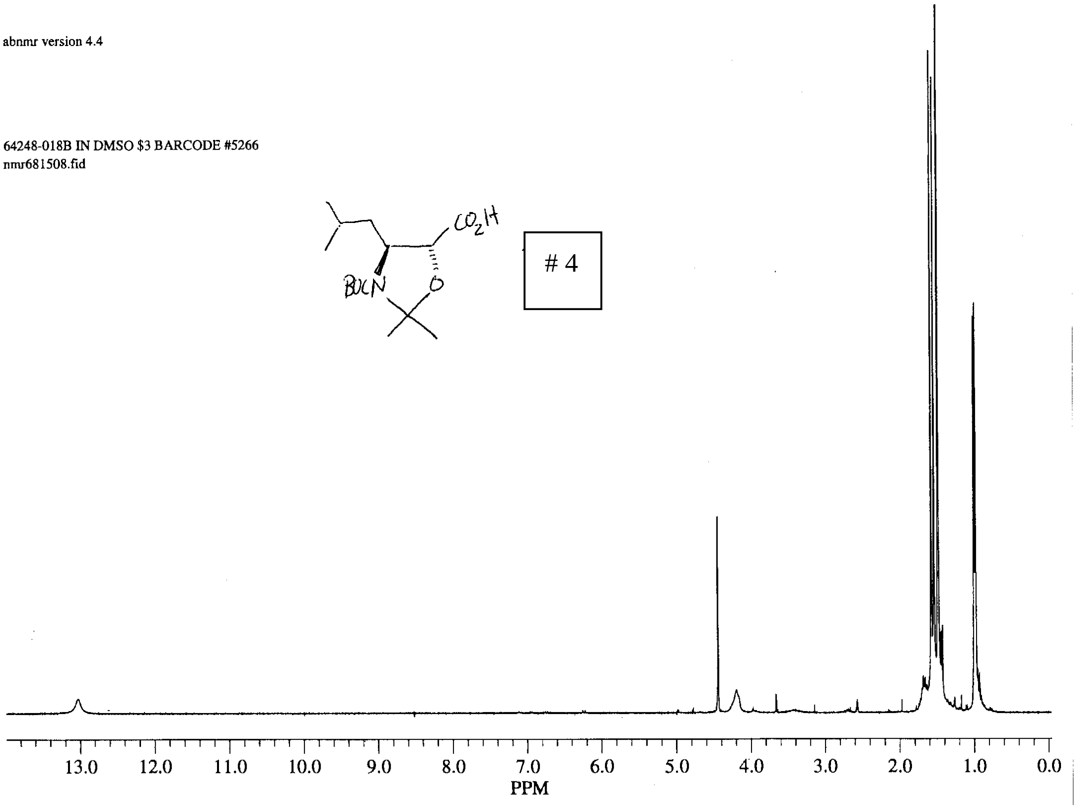
abnmr version 4.4

63842-096B IN DMSO $2 BARCODE #5257
nmr681132.fid



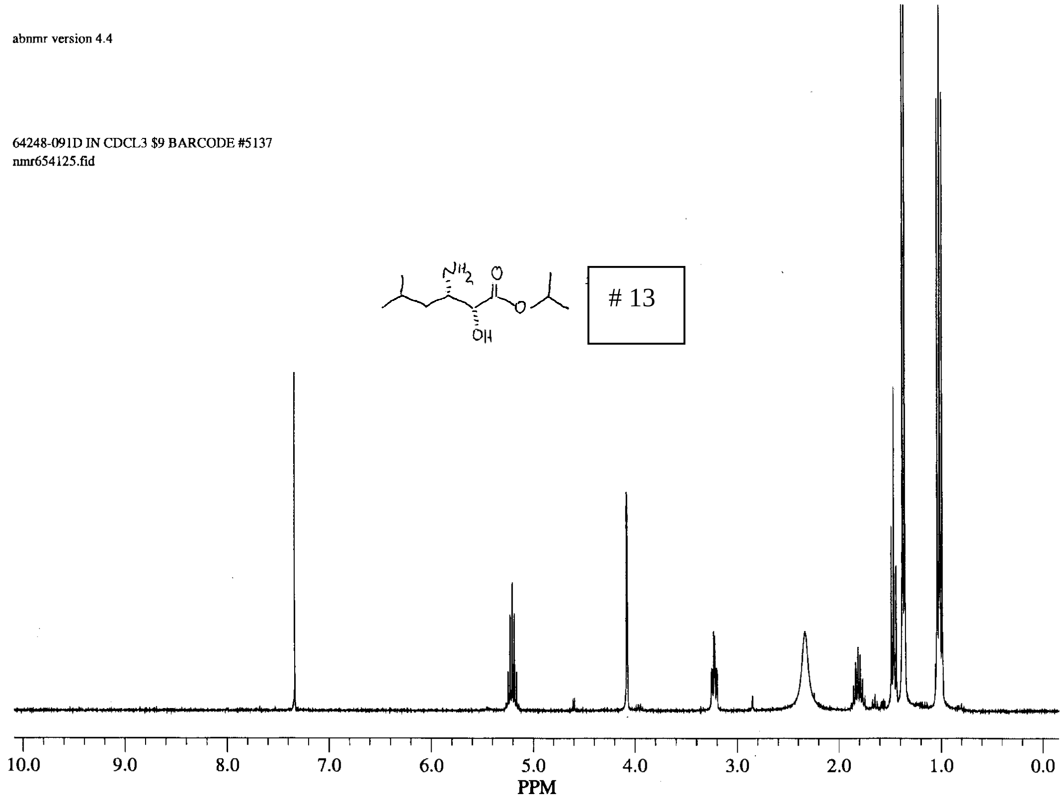
abnmr version 4.4

64248-018B IN DMSO $3 BARCODE #5266
nmr681508.fid



abnmr version 4.4

64248-091D IN CDCL3 $9 BARCODE #5137
nmr654125.fid

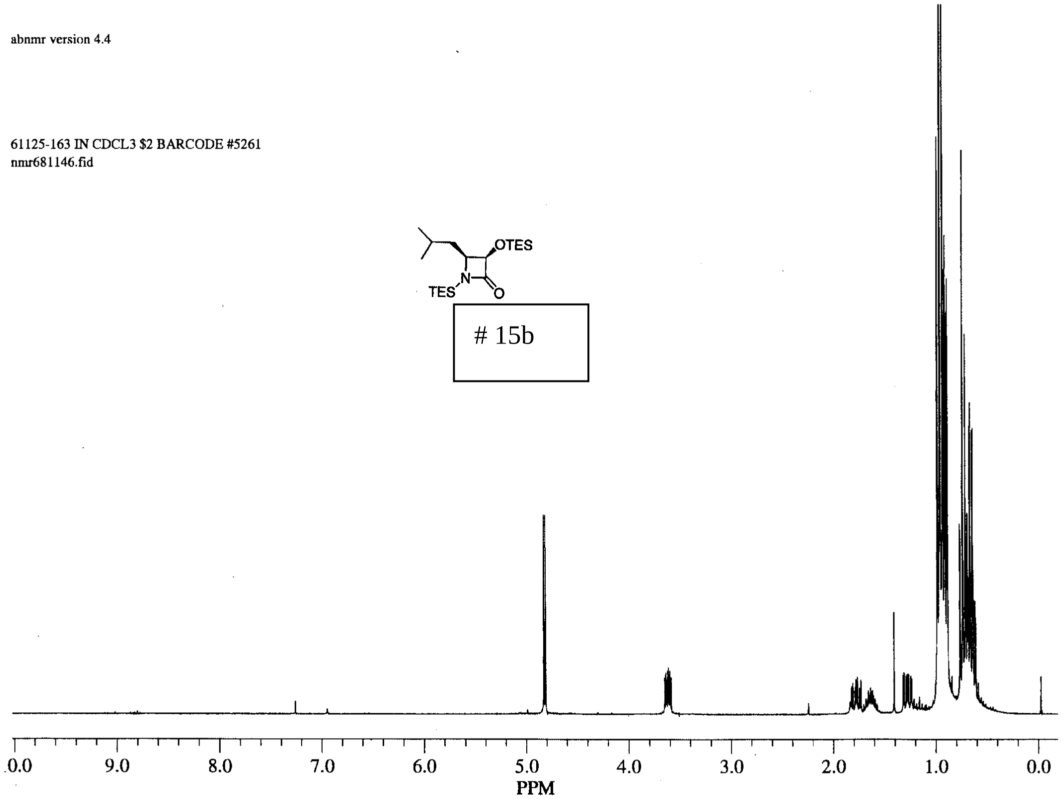


13

#20

abnmr version 4.4

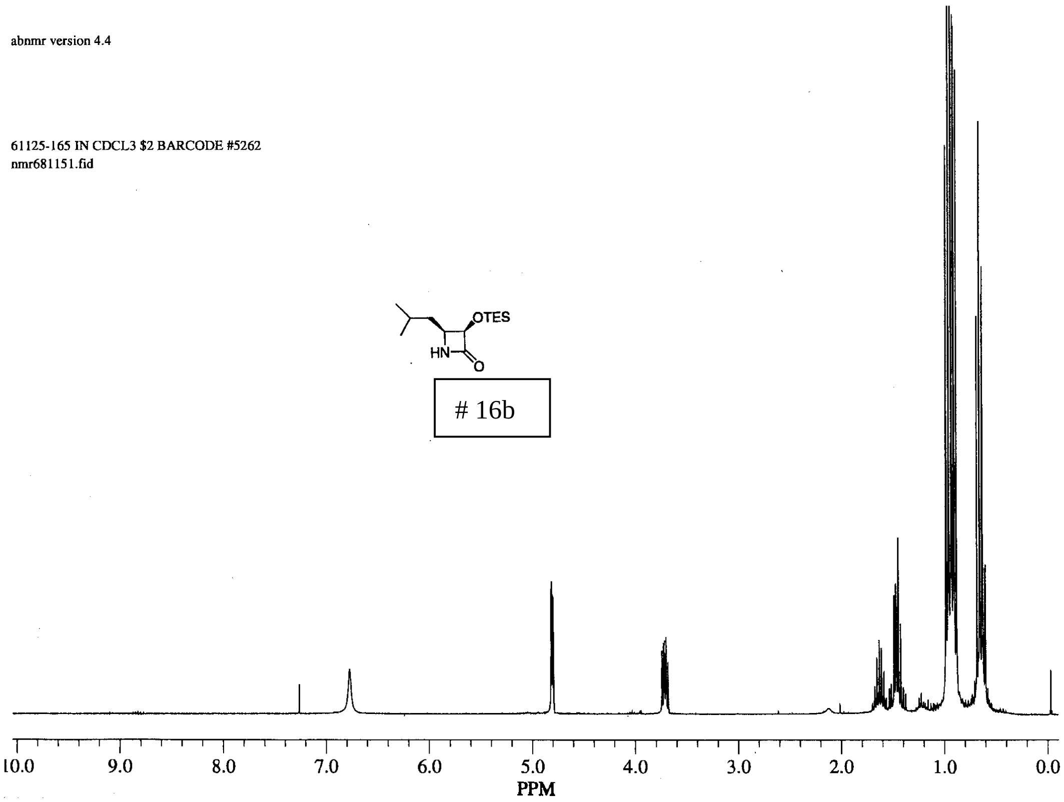
61125-163 IN CDCL3 $2 BARCODE #5261
nmr681146.fid



15b

abnmr version 4.4

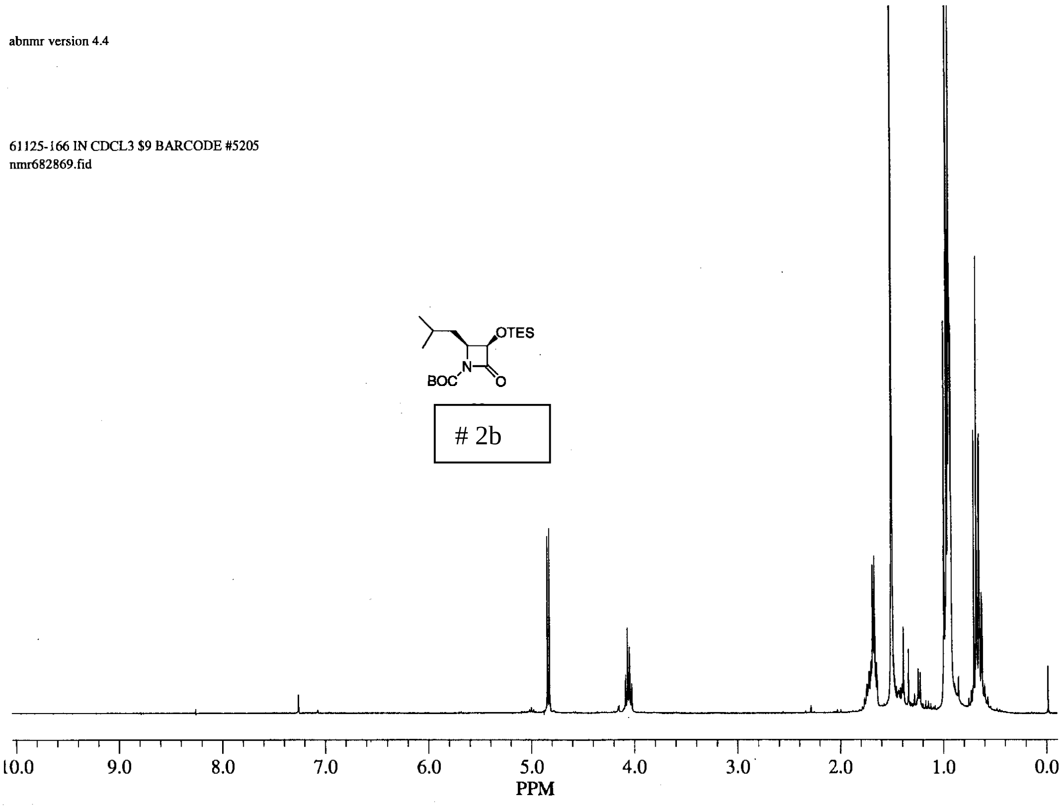
61125-165 IN CDCL3 $2 BARCODE #5262
nmr681151.fid



16b

abnmr version 4.4

61125-166 IN CDCL3 $9 BARCODE #5205
nmr682869.fid



2b