Enantioselective Addition of Activated Terminal Alkynes to 1-Acylpyridinium Salts Catalyzed by Cu-Bis(oxazoline) Complexes

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Experimental

General methods: All the reactions were carried out under an argon atmosphere with dry solvents under anhydrous conditions unless otherwise indicated. Optical rotations were measured by used a Perkin–Elmer 241MC polarimeter in the solvent indicated. IR spectra were recorded on an AVATAR-360 spectrophotometer. ^1H and ^{13}C NMR spectra were recorded on MERCURY300, Bruker DRX-400, and Bruker AV-500 spectrometers with TMS as the internal standard. HRMS were recorded by using either FTMS-7 or IonSpec 4.7 spectrometers. Silica gel plate GF254 were used for thin layer chromatography (TLC) and silica gel H or 300-400 mesh were used for flash column chromatography. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise indicated. Enantiomeric excesses were determined by HPLC using a Daicel Chiralpak AD-H or OD-H column (wavelength = 214, 220 or 230 nm) with hexane/*i*-PrOH as the eluent.

Material. All solvents and CuI were purified according to *Purification of Laboratory Chemicals (4th edition)*, W. L. F. Armarego & D. D. Perrin, Butterworth Heinemann.

General procedure for addition of alkynes to 1-acylpyridium salts: CuI (10 mg, 0.05 mmol) and ligand **L5** (20 mg, 0.05 mmol) were dissolved in 1 mL of CH_2Cl_2 and the solution was cooled to $-78\text{ }^\circ\text{C}$. 1-Alkyne (2.5 mmol) and DIPPA (0.6 mL, 2.5 mmol) were then added, and the solution was stirred at this temperature for 30 min before it was transferred to a flask containing the 1-acylpyridinium salt (generated from 0.5 mmol of pyridine and 2.5 mmol of methyl chloroformate) at -78

°C by a syringe. The resultant mixture was stirred for 15 h at the same temperature and then was directly purified by chromatography to afford the addition products.

3a

(R)-Methyl 2-(2-(ethoxycarbonyl)ethynyl)pyridine-1(2H)-carboxylate (3a):

72% yield. $[\alpha]_D^{29} +683$ (*c* 1.1, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 6.78 & 6.67 (due to rotamers, both d, *J* = 7.8 Hz, 1H), 6.02 (m, 1H), 5.75 & 5.62 (due to rotamers, both d, *J* = 6.3 Hz, 1H), 5.50 (m, 1H), 5.34 (m, 1H), 4.18 (q, *J* = 7.5 Hz, 2H), 3.8 (s, 3H), 1.26 (t, *J* = 6.6 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 153.3, 153.2, 124.6, 123.6, 116.2, 105.4, 82.0, 75.0, 62.1, 53.7, 43.6, 13.9; MS (ESI) *m/z* 258.1 (M + Na)⁺; HRMS *m/z* calcd for C₁₂H₁₃NO₄Na (M + Na)⁺ 258.0737, found 258.0737; HPLC DAICEL CHIRALPAK AD-H, hexane/2-propanol = 80/20, flow rate: 0.8 mL/min, detector: 214 nm, *t*_R(minor) = 8.22 min, *t*_R(major) = 9.70 min, ee = 94%.

3b

(R)-Methyl 2-(2-(methoxycarbonyl)ethynyl)pyridine-1(2H)-carboxylate

(3b): 74% yield. $[\alpha]_D^{29} +637$ (*c* 1.1, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 6.75 & 6.64 (due to rotamers, both d, *J* = 7.5 Hz, 1H), 5.98 (m, 1H), 5.71 & 5.60 (due to rotamers, both d, *J* = 6.3 Hz, 1H), 5.50 (m, 1H), 5.28 (m, 1H), 3.78 (s, 3H), 3.69 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 153.4, 153.1, 125.2, 123.9, 116.0, 105.3, 84.2, 74.3, 53.6, 52.6, 43.4; MS (ESI) *m/z* 244.1 (M + Na)⁺; HRMS *m/z* calcd for C₁₁H₁₁NO₄Na (M + Na)⁺ 244.0580, found 244.0589; HPLC DAICEL CHIRALPAK

AD-H, hexane/2-propanol = 80/20, flow rate: 0.8 mL/min, detector: 214 nm, t_R (minor) = 8.1 min, t_R (major) = 9.5 min, ee = 95%.

3c

(*R*)-Methyl 2-(2-((benzyloxy)carbonyl)ethynyl)pyridine-1(2*H*)-carboxylate

(3c): 81% yield. $[\alpha]_D^{29} +549$ (c 0.27, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.33 (m, 5H), 6.81 & 6.67 (due to rotamers, both d, $J = 7.8$ Hz, 1H), 6.03 (m, 1H), 5.77 & 5.64 (due to rotamers, both d, $J = 6.0$ Hz, 1H), 5.55 (m, 1H), 5.38 (m, 1H), 5.16 (s, 2H), 3.83 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 153.1, 153.0, 134.5, 128.7, 128.6, 125.0, 123.9, 116.0, 109.7, 105.5, 84.5, 74.6, 67.7, 53.7, 43.5; MS (ESI) m/z 320.1 ($\text{M} + \text{Na}$) $^+$; HRMS m/z calcd for $\text{C}_{17}\text{H}_{15}\text{NO}_4\text{Na}$ ($\text{M} + \text{Na}$) $^+$ 320.0893, found 320.0891; HPLC DAICEL CHIRALPAK OD-H, hexane/2-propanol = 95/5, flow rate: 0.8 mL/min, detector: 214 nm, t_R (minor) = 22.0 min, t_R (major) = 27.0 min, ee = 86%.

3d

(*R*)-Methyl 2-(3-oxobut-1-ynyl)pyridine-1(2*H*)-carboxylate (3d): 69% yield.

$[\alpha]_D^{29} +594$ (c 0.57, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 6.81 & 6.69 (due to rotamers, both d, $J = 7.5$ Hz, 1H), 6.03 (m, 1H), 5.79 & 5.67 (due to rotamers, both d, $J = 6.3$ Hz, 1H), 5.55 (m, 1H), 5.36 (m, 1H), 3.83 (s, 3H), 2.3 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 184.1, 153.3, 125.3, 123.9, 116.3, 105.3, 87.9, 82.2, 53.6, 43.6, 32.5; MS (ESI) m/z 228.1 ($\text{M} + \text{Na}$) $^+$; HRMS m/z calcd for $\text{C}_{11}\text{H}_{11}\text{NO}_3\text{Na}$ ($\text{M} + \text{Na}$) $^+$

228.0631, found 228.0628; HPLC DAICEL CHIRALPAK AD-H, hexane/2-propanol = 98/2, flow rate: 0.8 mL/min, detector: 214 nm, t_R (minor) = 17.0 min, t_R (major) = 19.6 min, ee = 93%.

(R)-Methyl 2-(3-oxopent-1-ynyl)pyridine-1(2H)-carboxylate (3e): 65% yield. $[\alpha]_D^{29} +602$ (c 1.17, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 6.81 & 6.69 (due to rotamers, both d, $J = 7.5$ Hz, 1H), 6.02 (m, 1H), 5.79 & 5.67 (due to rotamers, both d, $J = 6.3$ Hz, 1H), 5.55 (m, 1H), 5.36 (m, 1H), 3.83 (s, 3H), 2.56 (q, $J = 7.8$ Hz, 2H), 1.10 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 188.1, 153.3, 125.3, 123.9, 116.5, 105.4, 88.3, 82.0, 53.7, 43.8, 38.6, 7.76; MS (ESI) m/z 242.1 ($\text{M} + \text{Na}$) $^+$; HRMS m/z calcd for $\text{C}_{12}\text{H}_{13}\text{NO}_3\text{Na}$ ($\text{M} + \text{Na}$) $^+$ 242.0788, found 242.0789; HPLC DAICEL CHIRALPAK AD-H, hexane/2-propanol = 100/1, flow rate: 0.8 mL/min, detector: 220 nm, t_R (minor) = 27.9 min, t_R (major) = 33.4 min, ee = 99%.

(R)-Methyl 2-(3-oxohept-1-ynyl)pyridine-1(2H)-carboxylate (3f): 70% yield. $[\alpha]_D^{29} +563$ (c 0.4, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 6.81 & 6.68 (due to rotamers, both d, $J = 7.5$ Hz, 1H), 6.02 (m, 1H), 5.79 & 5.67 (due to rotamers, both d, $J = 6.0$ Hz, 1H), 5.55 (m, 1H), 5.34 (m, 1H), 3.83 (s, 3H), 2.53 (t, $J = 7.5$ Hz, 2H), 1.60 (m, 2H), 1.34 (m, 2H), 0.9 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 187.7, 153.4, 125.5, 123.9, 116.5, 105.5, 88.3, 81.9, 53.7, 45.0, 43.9, 25.9, 22.0, 13.7;

MS (ESI) m/z 270.1 ($M + Na$)⁺; HRMS m/z calcd for C₁₄H₁₇NO₃Na ($M + Na$)⁺ 270.1101, found 270.1101; HPLC DAICEL CHIRALPAK OD-H, hexane/2-propanol = 98/2, flow rate: 0.5 mL/min, detector: 214 nm, t_R (minor) = 13.6 min, t_R (major) = 14.6 min, ee = 91%.

(R)-Methyl 2-(3-oxooct-1-ynyl)pyridine-1(2H)-carboxylate (3g): 68% yield. $[\alpha]_D^{29} +558$ (c 1.75, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 6.78 & 6.66 (due to rotamers, both d, J = 7.2 Hz, 1H), 5.98 (m, 1H), 5.76 & 5.64 (due to rotamers, both d, J = 6.0 Hz, 1H), 5.56 (m, 1H), 5.34 (m, 1H), 3.83 (s, 3H), 2.48 (t, J = 7.8 Hz, 2H), 1.60 (m, 2H), 1.30 (m, 4H), 0.84 (t, J = 6.6 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 188.0, 153.0, 125.0, 123.0, 116.0, 105.0, 88.1, 81.1, 53.6, 45.2, 43.5, 30.9, 23.3, 22.2, 13.7; MS (ESI) m/z 284.1 ($M + Na$)⁺; HRMS m/z calcd for C₁₅H₁₉NO₃Na ($M + Na$)⁺ 284.1257, found 284.1252; HPLC DAICEL CHIRALPAK OD-H, hexane/2-propanol = 98/2, flow rate: 0.3 mL/min, detector: 214 nm, t_R (minor) = 28.3 min, t_R (major) = 30.3 min, ee = 90%.

(R)-Methyl 2-(6-(benzyloxy)-3-oxohex-1-ynyl)pyridine-1(2H)-carboxylate (3h): 70% yield. $[\alpha]_D^{29} +427$ (c 0.8, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.31 (m, 5H), 6.81 & 6.68 (due to rotamers, both d, J = 7.5 Hz, 1H), 6.03 (m, 1H), 5.79 & 5.66

(due to rotamers, both d, $J = 5.4$ Hz, 1H), 5.55 (m, 1H), 5.34 (m, 1H), 4.47 (s, 2H), 3.83 (s, 3H), 3.47 (t, $J = 6.0$ Hz, 2H), 2.67 (t, $J = 7.5$ Hz, 2H), 1.93 (m, 2H); MS (ESI) m/z 362.2 ($M + Na$)⁺; HRMS m/z calcd for C₂₀H₂₁NO₄Na 362.1363 ($M + Na$)⁺, found 362.1375; HPLC DAICEL CHIRALPAK AD-H, hexane/2-propanol = 90/10, flow rate: 0.8 mL/min, detector: 214 nm, t_R (minor) = 16.0 min, t_R (major) = 17.6 min, ee = 77%.

(R)-Methyl 2-(2-phenylethynyl)pyridine-1(2H)-carboxylate (3i): 75% yield.

¹H NMR (300 MHz, CDCl₃) δ 7.39 (m, 2H), 7.25 (m, 3H), 6.84 & 6.70 (due to rotamers, both d, $J = 7.2$ Hz, 1H), 5.99 (m, 1H), 5.83 & 5.68 (due to rotamers, both d, $J = 6.0$ Hz, 1H), 5.62 (m, 1H), 5.35 (m, 1H), 3.38 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 153.4, 131.7, 131.4, 128.1, 127.8, 125.1, 122.3, 118.6, 105.3, 86.3, 82.8, 53.3, 44.1; MS (ESI) m/z 262.1 ($M + Na$)⁺; HRMS m/z calcd for C₁₅H₁₃NO₂Na ($M + Na$)⁺ 262.0838, found 262.0841; HPLC DAICEL CHIRALPAK AD-H, hexane/2-propanol = 80/20, flow rate: 0.8 mL/min, detector: 214 nm, t_R (minor) = 6.93 min, t_R (major) = 7.34 min, ee = 1%.

(R)-Methyl 2-(hex-1-ynyl)pyridine-1(2H)-carboxylate (3j): 63% yield. ¹H

NMR (300 MHz, CDCl₃) δ 6.70 (m, 1H), 5.86 (m, 1H), 5.53 (m, 1H), 5.43 (m, 1H), 5.27 (m, 1H), 3.73 (s, 3H), 2.11 (t, $J = 7.2$ Hz, 2H), 1.36 (m, 2H), 1.27 (m, 2H), 0.81 (t,

$J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 153.0, 124.8, 121.4, 119.8, 105.4, 83.9, 73.3, 53.4, 43.6, 30.0, 21.8, 18.5, 13.6; MS (ESI) m/z 242.1 ($\text{M} + \text{Na}$) $^+$; HRMS m/z calcd for $\text{C}_{13}\text{H}_{17}\text{NO}_2\text{Na}$ ($\text{M} + \text{Na}$) $^+$ 242.1151, found 242.1164; HPLC DAICEL CHIRALPAK AD-H, hexane/2-propanol = 90/10, flow rate: 0.8 mL/min, detector: 214 nm, $t_{\text{R}}(\text{minor}) = 5.96$ min, $t_{\text{R}}(\text{major}) = 6.26$ min, ee = 11%.

(R)-Methyl 2-(3-acetoxyprop-1-ynyl)pyridine-1(2H)-carboxylate (3k): 77% yield. ^1H NMR (300 MHz, CDCl_3) δ 6.75 (m, 1H), 5.90 (m, 1H), 5.63 (m, 1H), 5.55 (m, 1H), 5.33 (m, 1H), 4.68 (s, 2H), 3.83 (s, 3H), 2.10 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 170.2, 153.0, 124.6, 122.4, 118.3, 105.4, 84.0, 74.1, 53.6, 52.2, 43.5, 20.7; MS (ESI) m/z 258.1 ($\text{M} + \text{Na}$) $^+$; HRMS m/z calcd for $\text{C}_{12}\text{H}_{13}\text{NO}_4\text{Na}$ ($\text{M} + \text{Na}$) $^+$ 258.0737, found 258.0749; HPLC DAICEL CHIRALPAK AD-H, hexane/2-propanol = 90/10, flow rate: 0.8 mL/min, detector: 214 nm, $t_{\text{R}}(\text{minor}) = 11.9$ min, $t_{\text{R}}(\text{major}) = 13.2$ min, ee = 3%.

(R)-Butyl 2-(2-(ethoxycarbonyl)ethynyl)pyridine-1(2H)-carboxylate (4): 72% yield. ^1H NMR (300 MHz, CDCl_3) δ 6.82 & 6.73 (due to rotamers, both d, $J = 6.0$ Hz, 1H), 6.06 (m, 1H), 5.79 & 5.68 (due to rotamers, both d, $J = 5.4$ Hz, 1H), 5.57 (m, 1H), 5.35 (m, 1H), 4.22 (t, $J = 7.2$ Hz, 2H), 4.04 (m, 2H), 2.04 (m, 1H), 1.28 (t, $J = 7.2$ Hz, 3H), 0.96 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 153.2,

152.9, 125.5, 124.0, 116.2, 105.2, 83.9, 75.7, 72.9, 62.0, 43.8, 43.2, 28.0, 19.1, 13.9;
 MS (ESI) m/z 300.1 ($M + Na$)⁺; HRMS m/z calcd for C₁₅H₁₉NO₄Na ($M + Na$)⁺
 300.1206, found 300.1217; HPLC DAICEL CHIRALPAK OD-H, hexane/2-propanol
 = 98/2, flow rate: 0.7 mL/min, detector: 214 nm, t_R (minor) = 12.6 min, t_R (major) =
 14.6 min, ee = 86%.

(R)-Methyl 2-(3-ethoxy-3-oxopropyl)-3,4-dihydropyridine-1(2H)-carboxylate (6a): To a solution of **3a** (77 mg, 0.33 mmol) in MeOH (5 mL) was added 10% Pd/C (7 mg). The reaction mixture was stirred under H₂ (1 atm) for 2 h. The catalyst was filtered off and flash chromatography (hexanes/EtOAc 20/1 to 15/1) gave **6a** (55 mg, 70%) as a colorless oil. $[\alpha]_D^{23} +93.8$ (c 0.95, CHCl₃); IR (film) 2956, 2850, 1735, 1707, 1654, 1445, 1413, 1362, 1237, 1190, 1117, 1076, 945, 769, 720 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 6.78 & 6.64 (due to rotamers, both d, J = 8.4 Hz, 1H), 4.98-4.90 & 4.90-4.81 (due to rotamers, both m, 1H), 4.40-4.31 & 4.31-4.21 (due to rotamers, both m, 1H), 4.12 (q, J = 6.9 Hz, 2H), 3.73 (s, 3H), 2.41-2.25 (m, 2H), 2.10-1.96 (m, 2H), 1.96-1.82 (m, 1H), 1.81-1.68 (m, 3H), 1.24 (t, J = 6.9 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 173.3, 153.9, 123.8 & 123.4 (due to rotamers), 106.3 & 106.0 (due to rotamers), 60.3, 52.8 & 52.7 (due to rotamers), 49.7, 31.2 & 30.8 (due to rotamers), 26.1, 24.8, 17.5 & 17.4 (due to rotamers), 14.2; MS (ESI) m/z 242.1 ($M + H$)⁺, 264.0 ($M + Na$)⁺; HRMS m/z calcd for C₁₂H₁₉NO₄Na ($M + Na$)⁺ 264.1206, found 264.1210.

(2*S*,6*R*)-Methyl 2-allyl-6-(3-ethoxy-3-oxopropyl)piperidine-1-carboxylate

(7a): To a solution of **6a** (51 mg, 0.21 mmol) in CH₂Cl₂ (2 mL) at -20 °C was added allyltrimethylsilane (0.20 mL, 1.26 mmol) followed by TFA (0.065 mL, 0.86 mmol). After 2 h at -20 °C, another portion of allyltrimethylsilane (0.10 mL, 0.63 mmol) and TFA (0.033 mL, 0.43 mmol) was added. The mixture was stirred at -20 °C for another 2 h and then warmed to 0 °C. The reaction was quenched with saturated NaHCO₃ and extracted with CH₂Cl₂. The combined organic extracts were washed with saturated brine, dried over Na₂SO₄ and concentrated. Flash chromatography (hexanes/EtOAc 20/1 to 15/1) gave **7a** (52 mg, 87%) as a colorless oil. $[\alpha]_D^{23} +7.0$ (*c* 1.0, CH₂Cl₂); IR (film) 2943, 2870, 1736, 1662, 1642, 1444, 1409, 1365, 1324, 1272, 1187, 1103, 1026, 916, 773 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 5.84-5.68 (m, 1H), 5.11-5.00 (m, 2H), 4.31-4.08 (m, 4H), 3.67 (s, 3H), 2.41-2.24 (m, 4H), 2.00-1.78 (m, 2H), 1.76-1.40 (m, 6H), 1.26 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 173.3, 156.7, 136.0, 117.0, 60.3, 52.4, 50.1, 49.8, 39.1, 32.0, 29.8, 28.1, 26.7, 14.2, 14.0; MS (ESI) *m/z* 284.2 (M + H)⁺, 306.1 (M + Na)⁺; HRMS *m/z* calcd for C₁₅H₂₅NO₄Na (M + Na)⁺ 306.1676, found 306.1683.

(5*R*,8*aR*)-5-Propylhexahydroindolizin-3(5*H*)-one (8): To a solution of **7a** (62 mg, 0.21 mmol) in MeOH (3 mL) was added 10% Pd/C (7 mg). The reaction

mixture was stirred under H₂ (1 atm) for 1 h. The catalyst was filtered off and concentration gave a colorless oil, which was dissolved in 5 mL of MeCN. To this solution was added NaI (157 mg, 1.05 mmol) and TMSCl (0.14 mL, 1.07 mmol). The mixture was refluxed for 1 h under darkness and then cooled to room temperature. To the reaction mixture was added MeOH (1 mL), then stirred for 30 min. The mixture was partitioned between brine and CH₂Cl₂. The aqueous phase was extracted with CH₂Cl₂. The combined organic extracts were washed with saturated Na₂S₂O₃, dried over Na₂SO₄ and concentrated.

To a solution of the above residue in toluene (6 mL) was added AlMe₃ (2.0 M in toluene, 0.15 mL, 0.30 mmol). The mixture was stirred at 80 °C overnight, cooled to room temperature and poured into 1 N HCl (25 mL). The mixture was extracted with CH₂Cl₂, washed with brine and concentrated. Flash chromatography (EtOAc) gave **8** (25 mg, 65% for 3 steps) as a colorless oil. $[\alpha]_D^{23}$ -27.2 (*c* 1.1, CH₂Cl₂); ¹H NMR (300 MHz, CDCl₃) δ 3.44-3.33 (m, 1H), 3.23-3.13 (m, 1H), 2.40-2.27 (m, 3H), 2.16-2.03 (m, 1H), 1.90- 1.60 (m, 4H), 1.59-1.22 (m, 6H), 0.93 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 174.3, 59.7, 57.3, 34.6, 32.0, 31.8, 29.5, 25.1, 22.7, 20.1, 14.1; MS (ESI) *m/z* 182.1 (M + H)⁺, 204.1 (M + Na)⁺.

(-)-167B (9)

(-)-167B (9): To a solution of **8** (25 mg, 0.14 mmol) in dry ether (3 mL) at 0 °C was added LiAlH₄ (21 mg, 0.54 mmol). The suspension was refluxed for 3 h and cooled to 0 °C. Water (0.025 mL), 15% NaOH (0.025 mL) and water (0.075 mL) were sequentially added. The resulting suspension was stirred for 30 min, filtered through

celite and dried over Na₂SO₄. Concentration gave (-)-167B (**9**) (19 mg, 82%) as a yellow oil. $[\alpha]_D^{23}$ -102.8 (*c* 0.88, CH₂Cl₂); ¹H NMR (500 MHz, CDCl₃) δ 3.25 (dt, *J* = 8.8, 2.2 Hz, 1H), 1.95 (t, *J* = 8.9 Hz, 1H), 1.88-1.52 (m, 8H), 1.50-1.06 (m, 8H), 0.90 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 65.0, 63.7, 51.6, 36.9, 31.0, 30.9, 24.7, 20.4, 19.1, 14.5; MS (ESI) *m/z* 168.1 (M + H)⁺.

(R)-Methyl 2-(3-oxoheptyl)-3,4-dihydropyridine-1(2H)-carboxylate (6f):

Following the same procedure from **3a** to **6a**, **6f** was obtained from **3f** in 72% yield. $[\alpha]_D^{23}$ +93.4 (*c* 1.2, CHCl₃); IR (film) 2960, 2874, 1712, 1655, 1444, 1413, 1361, 1237, 1191, 1117, 1061, 769, 719 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 6.75 & 6.61 (due to rotamers, both d, *J* = 9.3 & 8.1 Hz, 1H), 4.96-4.94 & 4.88-4.82 (due to rotamers, both m, 1H), 4.28-4.24 & 4.23-4.17 (due to rotamers, both m, 1H), 3.72 (s, 3H), 2.53-2.36 (m, 4H), 2.08-1.90 (m, 2H), 1.83-1.63 (m, 4H), 1.57-1.47 (m, 2H), 1.31-1.23 (m, 2H), 0.87 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.7, 154.0, 123.7 & 123.3 (due to rotamers), 106.5 & 106.2 (due to rotamers), 52.8, 49.8, 42.5, 39.4 & 38.9 (due to rotamers), 25.9, 25.1, 24.9, 22.3, 17.6, 13.8; MS (ESI) *m/z* 254.1 (M + H)⁺, 276.1 (M + Na)⁺; HRMS *m/z* calcd for C₁₄H₂₄NO₃ (M + H)⁺ 254.1751, found 254.1762.

(2S,6R)-Methyl 2-allyl-6-(3-oxoheptyl)piperidine-1-carboxylate (7f):

Following the same procedure from **6a** to **7a**, **7f** was obtained from **6f** in 94% yield. $[\alpha]_D^{26} +12.0$ (*c* 1.1, CHCl₃); IR (film) 2958, 2874, 1695, 1444, 1409, 1365, 1324, 1272, 1191, 1100, 915, 772 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 5.78-5.67 (m, 1H), 5.06-5.00 (m, 2H), 4.26- 4.22 (m, 1H) 4.17-4.13 (m, 1H), 3.67 (s, 3H), 2.49-2.29 (m, 6H), 1.85-1.78 (m, 2H), 1.76-1.41 (m, 8H), 1.36-1.23 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.6, 156.8, 136.1, 116.9, 52.4, 50.2, 49.9, 42.5, 40.3, 39.2, 28.5, 28.3, 26.8, 25.9, 22.3, 14.1, 13.8; MS (ESI) *m/z* 296.2 (M + H)⁺, 318.2 (M + Na)⁺; HRMS *m/z* calcd for C₁₇H₃₀NO₃ (M + H)⁺ 296.2220, found 296.2231.

(-)-5Z, 9Z-223AB (10)

(-)-(5Z, 9Z)-223AB (10): To a solution of **7f** (20 mg, 0.068 mmol) in MeOH (1 mL) was added 10% Pd/C (2 mg). The reaction mixture was stirred under H₂ (1 atm) for 1 h. The catalyst was filtered off and the filtrate was concentrated to give a colorless oil, which was dissolved in 2 mL of MeCN. To this solution was added NaI (51 mg, 0.34 mmol) and TMSCl (0.48 mL, 0.37 mmol). The resultant mixture was refluxed for 1 h under darkness and then cooled to room temperature. To the reaction mixture was added MeOH (0.5 mL), then stirred for 30 min. The mixture was partitioned between brine and CH₂Cl₂. The aqueous phase was extracted with CH₂Cl₂. The combined organic extracts were washed with saturated Na₂S₂O₃, dried over Na₂SO₄ and concentrated.

A solution of the above residue in THF (1.5 mL) and 1N HCl (0.5 mL) was

stirred for 1 d. The mixture was then washed with ether, basified by saturated K_2CO_3 , and extracted with CH_2Cl_2 (10 mL $\times$ 2). The combined organic phase was dried over Na_2SO_4 and concentrated.

To a solution of the above residue in MeOH (1 mL) was added PtO_2 . The mixture was then stirred under H_2 (1 atm) for 2 d. The catalyst was filtered off and the mixture was then acidified by 1 N HCl, washed with ether, basified by saturated K_2CO_3 and extracted with CH_2Cl_2 (10 mL $\times$ 2). The combined organic phase was dried over Na_2SO_4 and concentrated. Flash chromatography on neutral Al_2O_3 eluting with EtOAc gave (-)-(5Z, 9Z)-223AB (**10**) (9 mg, 60% for 4 steps) as a yellow oil. $[\alpha]_{\text{D}}^{23}$ -11.1 (*c* 0.20, MeOH); ^1H NMR (500 MHz, CDCl_3) δ 2.35-2.30 (m, 1H), 2.15-2.06 (m, 2H), 1.80-1.54 (m, 4H), 1.50-1.10 (m, 16H), 0.98-0.86 (m, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 67.6, 65.1, 62.2, 39.9, 38.1 32.0, 31.0, 30.5, 29.9, 29.3, 25.0, 22.9, 19.3, 14.5, 14.2; MS (ESI) m/z 224.2 ($\text{M} + \text{H}$) $^+$.

szp-6-51-2-C13
Archive directory: /export/home/ding-kj/vmr/sys/data
Sample directory:
File: CARBON
Pulse Sequence: szpul

Software Version: 4.1<2F12>
Date: 06-9-21 10:01
Sample Name : 5-95-1-1+/-
Data File : D:\CS\D-2_499.RAW Date: 06-9-21 9:31
Sequence File: D:\CS\D-2.SEQ Cycle: 1 Channel : A
Instrument : 970A - 0 Rack/Vial: 0/0 Operator: DING
Sample Amount : 1.0000 Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	1.402	29902.88	789.28	1.25	BV
2	2.186	31832.12	641.35	1.33	VB
3	4.050	6293.11	849.46	0.26	BV
4	4.347	16926.39	1282.91	0.70	VB
5	4.869	1564.50	151.60	0.07	BB
6	5.937	1298.07	174.31	0.05	BV
7	6.551	24223.93	545.89	1.01	VV
8	7.533	7238.59	601.21	0.30	VV
9	7.952	32294.93	3178.73	1.34	VV
10	8.222	1108297.11	110761.41	46.15	VE
11	8.984	18489.00	999.43	0.77	EV
12	9.702	1088916.37	92331.81	45.34	VE
13	10.241	23585.00	916.95	0.98	EB
14	12.094	10660.00	315.67	0.44	BB

Software Version: 4.1<2F12>
 Date: 06-9-21 10:09
 Sample Name : 5-95-1-2
 Data File : D:\CS\D-2_500.RAW Date: 06-9-21 9:52
 Sequence File: D:\CS\D-2.SEQ Cycle: 1 Channel : A
 Instrument : 970A -0 Rack/Vial: 0/0 Operator: DING
 Sample Amount : 1.0000 Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	1.608	25405.00	331.94	1.05	BB
2	4.041	31696.32	4811.97	1.31	BV
3	4.290	54577.57	6038.93	2.26	VV
4	4.865	12310.69	1205.45	0.51	VV
5	5.175	4372.42	599.40	0.18	VB
6	5.935	2000.00	188.17	0.08	BB
7	6.581	1045.00	97.96	0.04	BB
8	7.005	2747.38	252.42	0.11	BV
9	7.536	16604.70	1500.93	0.69	VV
10	7.954	37485.88	3987.49	1.55	VV
11	8.220	71631.54	7239.41	2.97	VB
12	9.697	2108857.00	180670.77	87.43	BE
13	10.236	36076.00	2177.26	1.50	EB
14	12.125	7374.00	351.90	0.31	BB

$\frac{2108857}{2748} = 74\%$

szx-6-51-1-c13
Archive directory: /export/home/ding-k1/vnmrSYS/data
Sample directory:
File: CARBON
Pulse Sequence: szpul

Software Version: 4.1<2F12>

Date: 06-9-25 11:43

Sample Name : 5-95-1-1+/-

Data File : D:\CS\D-2_518A.RAW Date: 06-9-25 11:07

Sequence File: D:\CS\D-2_SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: DING

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	0.972	28324.62	690.84	0.25	BV
2	2.944	92027.41	1738.81	0.82	VV
3	4.239	281415.97	5736.11	2.49	VB
4	8.126	5044501.50	500303.82	44.71	BB
5	9.536	5130560.50	428123.35	45.47	BB
6	11.972	64841.10	1398.87	0.57	BV
7	14.186	495648.40	24293.51	4.39	VB
8	16.392	9879.50	387.45	0.09	BB
9	18.742	21634.50	456.19	0.19	BB
10	20.668	3520.50	145.60	0.03	BB
11	21.579	13122.00	366.40	0.12	BB
12	23.635	38242.47	492.60	0.34	BV
13	25.817	16926.72	328.20	0.15	VV
14	27.145	42716.31	597.86	0.38	VB

Software Version: 4.1<2F12>

Date: 06-11-2 13:37

Sample Name : 5-95-1-100

Data File : D:\CS\D-2_679.RAW Date: 06-11-2 10:54

Sequence File: D:\CS\D-2.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: DING-2

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	4.040	57587.99	9270.38	0.67	BV
2	4.285	424428.51	50063.77	4.95	VB
3	5.153	2597.00	480.58	0.03	BB
4	5.665	10710.89	800.24	0.12	BV
5	6.042	1571.78	156.07	0.02	VV
6	6.491	8293.84	756.85	0.10	VB
7	6.900	3587.53	239.35	0.04	BV
8	7.428	6573.35	486.60	0.08	VV
9	8.063	206467.07	20788.09	2.41	VV
10	8.617	30292.33	2403.49	0.35	VV
11	9.049	5784.92	569.34	0.07	VV
12	9.467	7586068.80	649947.25	88.49	VE
13	9.996	200395.00	15580.59	2.34	EB
14	11.704	2350.00	126.61	0.03	BB
15	13.054	16763.22	739.98	0.20	BV

947%

Software Version: 4.1<2F12>

Date: 06-11-9 14:29

Sample Name : 6-17-1-1+/-

Data File : D:\CS\D-2_718.RAW Date: 06-11-9 13:48

Sequence File: D:\CS\D-2.SEQ Cycle: 1 Channel : A

Instrument : 970A-0 Rack/Vial: 0/0 Operator: DIMG-2

Sample Amount : 1.0000 Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	9.119	5524.00	221.59	0.04	BB
2	11.094	525721.00	16722.98	3.99	BB
3	14.992	66057.50	1329.69	0.50	BB
4	18.955	72919.52	1309.21	0.55	BV
5	21.529	6136375.48	95260.31	46.54	VB
6	27.943	6366450.00	74191.32	48.28	BB
7	36.431	12225.00	204.28	0.09	BB
		13185272.50	189239.39	100.00	

色谱分析报告

样品名称:

样品文件名:4-79-1-02-1. che

样品批号:

分析者:

分析日期:2006-04-26

分析时间:13:50

序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		6.277	15823.1	892966.8	1.3664
2	2		6.627	21595.1	419097.1	0.6413
3	3		7.127	51875.1	1753284.1	2.6829
4	4		14.677	1212.5	42197.2	0.0646
5	5		15.977	3852.3	182076.0	0.2786
6	6		17.777	31239.3	1446047.4	2.2127
7	7		19.027	31573.7	1311551.7	2.0069
8	8		21.277	27831.8	1310899.1	2.0059
9	9		22.577	73275.9	3412993.8	5.2226
10	10		26.177	955359.6	54200758.7	82.9382
11	11		29.727	5942.2	329585.3	0.5043
12	12		39.127	609.5	49348.1	0.0755
合计:				1220190.1	65350805.5	100.0000

色谱分析报告

样品名称:

样品文件名:6-21-1+-.che

样品批号:

分析者:

分析日期:2006-12-06

分析时间:13:31

6-21-11

序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		4.077	17247.1	247061.8	0.4593
2	2		5.177	11101.5	649438.9	1.2074
3	3		10.577	1241.6	279118.7	0.5189
4	4		11.777	4079.4	186872.2	0.3474
5	5		13.177	149.3	4729.4	0.0088
6	6		17.077	390972.1	22964785.7	42.6931
7	7		19.627	398132.6	24899566.1	46.2900
8	8		22.227	69438.3	4558777.3	8.4751
合计:				892361.8	53790350.2	100.0000

色谱分析报告

样品名称:

样品文件名:6-271-2.che

样品批号:

分析者:

分析日期:2006-12-06

分析时间:14:36

序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		1.627	557.2	46837.6	0.1274
2	2		4.077	17272.0	255244.3	0.6945
3	3		4.577	6962.1	118726.8	0.3231
4	4		5.277	1823.3	53344.0	0.1451
5	5		6.177	6813.4	196045.9	0.5334
6	6		10.377	580.5	17846.2	0.0486
7	7		12.327	308.4	10922.8	0.0297
8	8		15.677	198.0	9276.6	0.0252
9	9		17.617	34690.7	1324520.4	3.6040
10	10		18.630	1686.9	32844.0	0.0894
11	11		20.333	484999.4	34685605.6	94.3795

合计: 555892.0 36751214.2 100.0000

93%

色谱分析报告

样品名称:

样品文件名:6-18-2-1+-.che

样品批号:

分析者:

分析日期:2006-12-13

分析时间:08:42

序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		4.127	2264.6	39790.2	0.2037
2	2		4.577	5495.6	93362.3	0.4780
3	3		5.227	2195.3	46483.1	0.2380
4	4		5.777	3166.6	101800.1	0.5212
5	5		7.227	854.8	35255.1	0.1805
6	6		12.527	617.6	155789.7	0.7976
7	7		15.727	266.6	8008.7	0.0410
8	8		16.777	214.4	6418.7	0.0329
9	9		17.477	221.0	9238.8	0.0473
10	10		19.977	215.1	7736.5	0.0396
11	11		27.877	112841.2	9252380.7	47.3712
12	12		33.427	70561.0	9641948.8	49.3658
13	13		40.177	1001.2	133439.8	0.6832
合计:				199915.0	19531652.6	100.0000

色谱分析报告

样品名称:

样品文件名:6-18-2-2. che

样品批号:

分析者:

分析日期:2006-12-13

分析时间:09:40

序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		4.177	28432.2	415279.2	0.3217
2	2		4.927	33520.2	978970.5	0.7583
3	3		5.777	7952.9	317095.7	0.2456
4	5		10.327	152.8	5453.8	0.0042
5	6		11.577	248.0	10057.0	0.0078
6	7		12.827	267.8	9004.1	0.0070
7	8		16.777	1051.7	69061.1	0.0535
8	9		19.227	1066.4	52874.1	0.0410
9	10		25.927	201.8	47117.2	0.0365
10	11		29.327	1174498.3	121232472.9	93.9069
11	12		38.977	231.9	31717.9	0.0246
12	13		41.877	42616.2	5929430.9	4.5929

合计: 1290240.0 129098534.5 100.0000

Software Version: 4.1<2F12>

Date: 06-11-9 10:49

Sample Name : 5-61-2-1+/-

Data File : D:\CS\D-2_714.RAW Date: 06-11-9 10:29

Sequence File: D:\CS\D-2.SEQ Cycle: 1 Channel : A

Instrument : 970A -0 Rack/Vial: 0/0 Operator: DIMG-2

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	0.892	332721.80	1621.89	1.26	BV
2	3.539	1659390.20	33403.05	6.28	VB
3	5.849	787026.00	19816.76	2.98	BB
4	7.863	8275.50	541.90	0.03	BB
5	8.912	103940.50	5610.29	0.39	BB
6	11.115	136045.00	3864.59	0.51	BB
7	13.650	11474871.49	480591.68	43.40	BV
8	14.572	11828902.90	456371.42	44.74	VV
9	16.886	70665.77	2165.50	0.27	VV
10	18.108	38374.84	1378.15	0.15	VB

26440214.00 1.01e+06 100.00

Software Version: 4.1<2F12>

Date: 06-11-7 15:51

Sample Name : 5-61-2-2

Data File : D:\CS\D-2_703.RAW Date: 06-11-7 15:22

Sequence File: D:\CS\D-2.SEQ Cycle: 1 Channel : A

Instrument : 970A_0 Rack/Vial: 0/0 Operator: DIMG-2

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	1.996	330004.47	2951.20	2.19	BV
2	4.011	1435744.53	72780.33	9.54	VB
3	5.891	654997.50	26761.30	4.35	BB
4	9.156	91070.77	4737.82	0.61	BV
5	10.019	26233.23	841.14	0.17	VB
6	11.946	17335.00	753.89	0.12	BB
7	14.044	710277.37	28284.50	4.72	BV
8	15.373	11557963.39	436368.72	76.83	VV
9	17.908	98742.36	1993.60	0.66	VV
10	18.954	32974.38	861.74	0.22	VB
11	20.675	7892.00	205.36	0.05	BB
12	28.008	80319.00	1629.33	0.53	BB

15043554.00 578168.93 100.00

szk-6-51-9-c13
 Archive directory: /export/home/ding-k1/vnmr-sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: szpul

色谱分析报告

样品名称:

样品文件名:6-41-2-1+-od-h. che

样品批号:

分析者:

分析日期:2007-01-16

分析时间:09:47

序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		10.827	430.9	11858.3	0.1235
2	2		11.677	231.4	13633.4	0.1420
3	3		13.877	312.6	22590.0	0.2353
4	4		15.477	421.3	48813.1	0.5084
5	5		18.627	62.9	1771.7	0.0185
6	6		20.527	141.5	3587.3	0.0374
7	7		21.477	111.4	2585.8	0.0269
8	8		22.427	1405.0	59676.0	0.6215
9	9		25.327	3615.6	184601.0	1.9225
10	10		28.277	86200.1	4400516.3	45.8289
11	11		30.277	82759.0	4612677.8	48.0385
12	12		35.627	131.7	6855.8	0.0714
13	13		37.977	3524.0	232879.2	2.4253
合计:				179347.4	9602045.6	100.0000

色谱分析报告

样品名称:

样品文件名:6-41-1-2-2. che

样品批号:

分析者:

分析日期:2007-01-16

分析时间:11:06

序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		0.527	969.8	33691.0	0.0668
2	2		6.977	25749.9	975095.4	1.9340
3	3		7.977	16300.5	662078.9	1.3131
4	4		9.727	21918.9	1010340.5	2.0039
5	5		10.877	138095.5	10496972.9	20.8194
6	6		12.477	564.2	12870.9	0.0255
7	7		14.127	609.3	17187.9	0.0341
8	8		15.377	1493.2	96928.5	0.1922
9	9		16.627	1122.5	32814.5	0.0651
10	10		17.577	605.1	22315.4	0.0443
11	11		18.977	873.4	43507.0	0.0863
12	12		21.177	332.4	6292.9	0.0125
13	13		22.427	1304.8	47495.3	0.0942
14	14		25.127	39990.4	2100341.2	4.1658
15	15		28.127	33149.8	1659595.7	3.2916
16	16		29.927	616186.2	32999106.3	65.4494
17	17		32.877	3432.9	202630.6	0.4019

合计: 902698.8 50419264.9 100.0000

9.7%

Software Version: 4.1<2F12>

Date: 07-5-15 11:00

Sample Name : 7-11-1-1+/-

Data File : D:\CS\DING175A.RAW Date: 07-5-15 10:36

Sequence File: D:\CS\DING.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: d-1

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	7.566	164206.28	16915.88	1.20	BV
2	8.088	32350.93	1806.91	0.24	VV
3	8.652	38683.65	1719.06	0.28	VV
4	9.783	23626.28	811.27	0.17	VV
5	10.415	23548.57	1292.72	0.17	VV
6	11.076	177206.23	10285.77	1.30	VV
7	12.133	117713.03	7524.60	0.86	VV
8	12.698	75138.86	4293.74	0.55	VV
9	13.161	347796.75	21241.74	2.55	VV
10	14.271	172651.42	6195.78	1.26	VB
11	15.020	49636.50	2656.63	0.36	BB
12	16.005	5744419.50	311431.57	42.08	BB
13	17.581	6121846.06	290403.94	44.85	BE
14	18.875	145122.00	3321.39	1.06	EV
15	19.450	394316.94	16192.52	2.89	VB

Software Version: 4.1<2F12>

Date: 07-5-15 14:57

Sample Name : 7-11-1-3

Data File : D:\CS\DING177A.RAW Date: 07-5-15 14:31

Sequence File: D:\CS\DING.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: d-1

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	8.865	41336.00	2014.31	0.31	BB
2	11.181	122761.00	5221.06	0.93	BB
3	11.919	15801.97	714.68	0.12	BV
4	12.728	26912.03	1851.37	0.20	VB
5	14.441	103309.00	3808.07	0.78	BB
6	16.210	1385181.50	69918.34	10.47	BB
7	17.811	10457548.06	472030.95	79.07	BV
8	19.718	941154.94	37427.84	7.12	VE
9	21.308	5587.00	172.75	0.04	EB
10	22.352	16961.50	715.40	0.13	BB
11	23.838	109516.00	1871.40	0.83	BB

13226069.00 595746.17 100.00

szk-6-51-7-c13
 Archive directory: /export/home/d/ing-kj/vmr/sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: szpul

Software Version: 4.1<2F12>

Date: 06-9-12 9:10

Sample Name : 5-83-1-1+-

Data File : D:\CS\D-2_461.RAW Date: 06-9-12 8:43

Sequence File: D:\CS\D-2_SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: DING

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	9.558	7179.00	162.44	0.04	BB
2	12.584	9067653.88	175064.17	48.36	BV
3	14.558	9253710.98	152289.51	49.35	VV
4	18.079	422327.64	4465.62	2.25	VB

18750871.50 331981.74 100.00

Software Version: 4.1<2F12>

Date: 06-9-12 9:42

Sample Name : 5-83-1-2

Data File : D:\CS\D-2_462.RAW Date: 06-9-12 9:14

Sequence File: D:\CS\D-2.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: DING

Sample Amount : 1.0000

Dilution Factor : 1.00

DEFAULT REPORT

Peak #	Time [min]	Area [uv*sec]	Height [uv]	Area [%]	BL
1	10.997	28920.45	708.51	0.14	BV
2	12.262	1381968.65	27795.24	6.91	VV
3	13.848	17965578.20	309633.95	89.87	VV
4	17.119	558145.53	7884.77	2.79	VB
5	20.542	12987.18	287.49	0.06	BV
6	22.035	25542.06	319.43	0.13	VV
7	24.128	17279.76	288.05	0.09	VB
		19990421.84	346917.44	100.00	

86%

154652