Morita-Baylis-Hillman Adducts as Effective Dipolarophiles in Cu(I)-Catalyzed 1,3-Dipolar Cycloaddition with Azomethine Ylides: Asymmetric Construction of Pyrrolidine Derivatives Containing Quaternary Stereogenic Center

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General Remarks.

¹H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = single, d = double, t = triple, q = quarte, m = multiple or unresolved, brs = broad single, coupling constant(s) in Hz, integration). ¹³C NMR spectra were recorded on a VARIAN Mercury 100 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Diastereomeric ratios were determined from crude ¹H NMR or HPLC analysis. Enantiomeric ratios were determined by HPLC, using a chiralcel AD-H column, a chiralpak AS-H column with hexane and *i*-PrOH as solvents. Ligands **1a-e** were prepared according to the literature procedure reported by us.¹ Morita-Baylis-Hillman Adducts were prepared according to the literature procedure.² The racemic adducts were attained by using racemic AgOAc/PPh₃ as the catalyst. The absolute configuration of 5 was determined unequivocally according to the X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.

General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with Morita-Baylis-Hillman Adducts Catalyzed by Cu(I)/(S)-TF-BiphamPhos Complex in the Presence of Et₃N as Base

Under argon atmosphere (*S*)-TF-BiphamPhos **1e** (5.3 mg, 0.0066 mmol) and $Cu(CH_3CN)_4BF_4$ (1.9 mg, 0.006 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h at room temperature. Then, imine substrate (0.40 mmol), Et₃N (0.03 mmol) were added sequentially, the misture was dropped to -20°C and Morita-Baylis-Hillman Adducts (0.20 mmol) was added. Once starting material was consumed (monitored by TLC), the mixture was filtered through celite and the filtrate was concentrated to dryness, the residue was purified by column chromatography to give the corresponding cycloaddition product, which was then

directly analyzed by chiral HPLC to determine the enantiomeric excess.

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 5-(4-chlorophenyl)-4-(hydroxymethyl)-

pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}_{D} = +1.8$ (*c* 2.10, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.36 (d, *J* = 7.2 Hz, 2H), 7.28 (d, *J* = 7.2 Hz, 2H), 4.31 (s, 1H), 3.98 (t, *J* = 8.4 and 10.2 Hz, 1H), 3.89 (d, *J* = 11.4 Hz, 1H), 3.82 (s, 3H), 3.77 (d, *J* = 11.4 Hz, 1H), 2.64 (br, 1H), 2.58 (m, 2H), 2.32 (m, 1H) ,1.09 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.38, 37.02, 52.24, 58.56, 60.56, 66.00, 66.90, 81.91, 128.11, 129.01, 133.27, 138.02, 172.31, 173.58; IR (KBr) v 3675, 3616, 3019, 2977, 2400, 1737, 1216, 757 cm⁻¹. HRMS Calcd. For C₁₈H₂₄ClNO₅ + H⁺: 370.1416, found 370.1411. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm); t_r = 18.76 and 32.26 min.

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 5-(2-chlorophenyl)-4-(hydroxymethyl)pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{D} = -26.2 \ (c \ 1.21, \ CHCl_3); \ ^1H \ NMR \ (CDCl_3, \ TMS, \ 300 \ MHz) \ \delta \ 7.70 \ (d, \ J = 7.8 \ Hz, \ 1H), \ 7.15-7.35 \ (m, \ 3H), \ 4.86 \ (s, \ 1H), \ 4.12-4.00 \ (m, \ 2H), \ 3.80 \ (s, \ 3H), \ 3.75 \ (d, \ J = 11.1 \ Hz, \ 1H), \ 2.62 \ (t, \ J = 11.4 \ Hz, \ 1H), \ 2.38-2.46 \ (m, \ 2H), \ 1.06 \ (s, \ 9H); \ ^{13}C \ NMR \ (CDCl_3, \ TMS, \ 100 \ MHz) \ \delta \ 27.28, \ 35.58, \ 52.20, \ 58.45, \ 61.94, \ 65.91, \ 81.59, \ 126.90, \ 128.60, \ 129.09, \ 129.84, \ 133.65, \ 138.50, \ 171.31, \ 173.91; \ IR \ (KBr) \ v \ 3683, \ 3621, \ 128.60, \ 129.09, \ 129.84, \ 133.65, \ 138.50, \ 171.31, \ 173.91; \ IR \ (KBr) \ v \ 3683, \ 3621, \ 128.60, \ 129.09, \ 129.84, \ 133.65, \ 138.50, \ 171.31, \ 173.91; \ IR \ (KBr) \ v \ 3683, \ 3621, \ 128.60, \ 129.09, \ 129.84, \ 133.65, \ 138.50, \ 171.31, \ 173.91; \ IR \ (KBr) \ v \ 3683, \ 3621, \ 128.60, \ 129$

3019, 2977, 2400, 1736, 1216, 757 cm⁻¹. HRMS Calcd. For $C_{18}H_{24}CINO_5 + H^+$: 370.1416, found 370.1416. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 11.40 and 20.42 min.

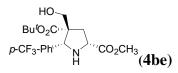
(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 5-(4-fluorophenyl)-4-(hydroxymethyl)pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 72% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{\rm D} = +1.2$ (*c* 0.79, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.36 (m, 2H), 6.99 (t, *J* = 8.7 Hz and 8.4 Hz, 2H), 4.28 (s, 1H), 3.97-3.86 (m, 2H), 3.80 (s, 3H), 3.74 (d, *J* = 11.7 Hz, 1H), 2.68 (br, 1H), 2.54 (m, 1H), 2.32 (m, 1H), 1.08 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.75, 37.43, 52.57, 58.93, 60.86, 66.66, 67.38, 82.28, 115.05, 115.33, 129.53, 129.64, 172.79, 173.88; IR (KBr) v 3684, 3620, 3019, 2896, 2400, 1737, 1217, 758 cm⁻¹. HRMS Calcd. For C₁₈H₂₄FNO₅ + H⁺: 354.1711, found 354.1713. The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 11.29 and 15.97 min.

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 5-(4-bromophenyl)-4-(hydroxymethyl)pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 80% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{D} = -3.0 \ (c \ 1.24, \text{CHCl}_3); {}^{1}\text{H} \text{ NMR} \ (\text{CDCl}_3, \text{TMS}, 300 \text{ MHz}) \delta 7.43 \ (d, J = 8.1 \text{ Hz} 2\text{H}), 7.29 \ (d, J = 8.7 \text{ Hz}, 2\text{H}), 4.27 \ (s, 1\text{H}), 3.96 \ (m, 1\text{H}), 3.88 \ (d, J = 11.1 \text{ Hz}, 1\text{H}), 3.81 \ (s, 3\text{H}), 3.75 \ (d, J = 11.1 \text{ Hz}, 1\text{H}), 2.73 \ (br, 1\text{H}), 2.54 \ (m, 1\text{H}), 2.32 \ (m, 1\text{H}), 1.09$

(s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.53, 37.20, 52.40, 58.72, 60.78, 66.34, 67.15, 82.15, 121.54, 129.51, 131.21, 138.70, 172.50, 173.68; IR (KBr) v 3684, 3620, 3019, 2976, 2400, 1737, 1217, 741 cm⁻¹. HRMS Calcd. For C₁₈H₂₄BrNO₅ + H⁺: 414.0911, found 414.0912. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 11.27 and 16.96 min.



(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 4-(hydroxymethyl)-5-(4-(trifluoromethyl)phenyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{D} = +2.5 \ (c \ 1.05, CHCl_3); {}^{1}H \ NMR \ (CDCl_3, TMS, 300 \ MHz) \ \delta \ 7.57 \ (s, 4H), 4.42 \ (s, 1H), 4.01 \ (t, J = 9.0 \ Hz \ and 8.7Hz, 1H), 3.90 \ (d, J = 11.7 \ Hz, 1H), 3.82 \ (s, 3H), 3.80 \ (d, J = 11.7 \ Hz, 1H), 2.90 \ (br, 1H), 2.57 \ (m, 1H), 2.32 \ (m, 1H), 1.04 \ (s, 9H); {}^{13}C \ NMR \ (CDCl_3, TMS, 100 \ MHz) \ \delta \ 27.78, 37.39, 52.80, 59.16, 61.09, 66.69, 67.46, 82.61, 125.41, 128.60, 144.40, 172.77, 174.01; IR \ (KBr) \ v \ 3684, 3620, 3019, 2977, 2400, 1738, 1216, 757 \ cm^{-1}. \ HRMS \ Calcd. For \ C_{19}H_{24}F_3NO_5 + H^+: 404.1679, found 404.1682. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralcel AS-H,$ *i* $-propanol/hexane = 20/80, flow rate 0.5 mL/min, <math>\lambda = 210$ nm); t_r = 9.21 and 12.88 min.

HO Bu'O₂C¹/ *p*-CN-Ph¹/¹/_N CO₂CH₃ H (4bf)

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 5-(4-cyanophenyl)-4-(hydroxymethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 65% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]_{D}^{25} = +6.4 (c \ 0.57, CHCl_3); {}^{1}H \ NMR \ (CDCl_3, TMS, 300 \ MHz) \ \delta \ 7.56 \ (s, 4H), 4.42$

(s, 1H), 4.00 (m, 1H), 3.86 (d, J = 11.7 Hz, 1H), 3.82 (s, 3H), 3.79 (d, J = 11.7 Hz, 1H), 2.73 (br, 1H), 2.55 (m, 1H), 2.32 (m, 1H), 1.06 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.69, 36.87, 52.63, 58.85, 60.77, 66.38, 67.08, 82.53, 111.45, 119.01, 128.97, 132.07, 145.96, 172.43, 173.77; IR (KBr) v 3423, 3055, 2985, 1720, 1265, 739 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralcel AD-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 230 nm); t_r = 25.38 and 29.42 min.

HO Bu^fO₂C^{··} Ph^{····} N^{··} (CO₂CH₃ H (4bg)

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 4-(hydroxymethyl)-5-phenyl pyrrolidine-2,4dicarboxylate

The title compound was prepared according to the general procedure as described above in 65% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{D} = +6.4$ (*c* 0.57, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.39-7.24 (m, 5H), 4.28 (s, 1H), 4.02-3.94 (m, 2H), 3.82 (s, 3H), 3.75 (d, *J* = 11.7 Hz ,1H), 2.86 (br, 1H), 2.55 (m, 1H), 2.36 (m, 1H), 1.06 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.07, 37.25, 51.97, 58.51, 60.54, 66.11, 67.57, 81.43, 127.24, 127.35, 127.85, 138.88, 172.24, 173.33; IR (KBr) v 3683, 3621, 3019, 2976, 1710, 1216, 776 cm⁻¹. HRMS Calcd. For C₁₉H₂₄F₃NO₅ + H⁺: 336.1806, found 336.1815. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 10.46 and 16.22min.

p-MeO-Ph^{ut}, NH^U, CO₂CH₃(4bh)

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 4-(hydroxymethyl)-5-(4-methoxyphenyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 70% yield. It was purified by flash chromatography to afford colorless oil.

[α]²⁵_D = +6.3 (*c* 0.51, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.29 (d, *J* = 9.0 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 4.22 (s, 1H), 4.00-3.91 (m, 2H), 3.81 (s, 3H), 3.78 (s, 3H), 3.73 (d, *J* = 11.1 Hz, 1H), 2.82 (br, 1H), 2.54 (m, 1H), 2.35(m, 1H), 1.10 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.32, 37.34, 52.09, 55.10, 58.56, 60.64, 66.26, 67.38, 81.57, 108.63, 113.30, 128.50, 131.06, 158.92, 172.48; IR (KBr) v 3684, 3620, 3019, 2976, 2400, 1737, 1516, 1217, 743 cm⁻¹. HRMS Calcd. For C₁₉H₂₄F₃NO₅ + H⁺: 366.1911, found 366.1915. The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralcel OD-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 19.42 and 26.06 min.

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 4-(hydroxymethyl)-5-(2-methoxyphenyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 68% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}_{D} = -32.1$ (*c* 0.55, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.45 (d, *J* = 7.5 Hz, 1H), 7.22 (t, *J* = 12 Hz and 7.8 Hz, 1H), 6.94 (t, *J* = 7.2 Hz and 7.5 Hz, 1H), 6.82 (d, *J* = 12.9 Hz, 1H), 4.55 (s, 1H), 4.02-3.90 (m, 2H), 3.79 (s, 6H), 3.65 (d, *J* = 11.1 Hz, 1H), 2.66-2.46 (m, 3H), 1.08 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.28, 37.38, 51.98, 54.95, 58.60, 61.14, 65.45, 80.99, 110.01, 120.36, 126.80, 128.41, 156.68, 172.23, 173.59; IR (KBr) v 3423, 3055, 2985, 1720, 1265, 739 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 86% ee (Chiralcel OD-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 13.83 and 21.54 min.

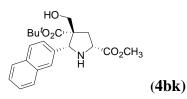
HO Bu^tO₂C¹ *m*-MeO-Ph¹,¹,¹</sup> H

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl

4-(hydroxymethyl)-5-(3-methoxyphenyl)

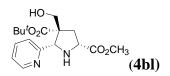
pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 72% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{\rm D}$ = +5.6 (*c* 0.55, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.27-7.18 (m, 1H), 6.94 (m, 1H), 6.79 (d, *J* = 7.8 Hz, 1H), 4.26 (s, 1H), 3.97-3.92 (m, 2H), 3.80 (s, 3H), 3.79 (s, 3H), 3.74 (d, *J* = 11.4 Hz, 1H), 2.64-2.35 (m, 3H), 1.08 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.90, 37.99, 52.77, 55.70, 59.26, 61.29, 66.92, 68.27, 82.24, 113.62, 113.70, 120.29, 129.60, 141.49, 159.86, 173.11, 174.22; IR (KBr) v 3423, 3055, 2985, 1720, 1265, 739 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 86% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 15.10 and 19.92 min.



(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 4-(hydroxymethyl)-5-(3-methoxyphenyl) pyrrolidine-2,4-dicarboxylate

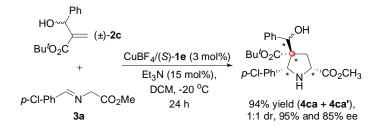
The title compound was prepared according to the general procedure as described above in 72% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{D} = +3.3$ (*c* 1.12, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.84-7.75 (m, 4H), 7.46 (m, 3H), 4.53 (s, 1H), 4.11 (t, *J* = 8.7 Hz and 8.7 Hz, 1H), 4.00 (d, *J* = 11.4 Hz, 1H), 3.84 (d, *J* = 11.4 Hz, 1H), 3.83 (s, 3H), 2.69-2.60 (m, 2H), 2.41-2.48 (m, 1H), 0.91 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.27, 37.60, 52.23, 58.77, 60.84, 66.29, 67.94, 80.70, 125.69, 125.78, 126.04, 126.22, 127.43, 127.64, 127.82, 132.80, 132.95, 136.55, 172.56, 173.65; IR (KBr) v 3683, 3620, 3019, 2976, 2400, 1736, 1522, 1216, 751 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 254 nm); t_r = 11.89 and 21.25 min.



(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 4-(hydroxymethyl)-5-(pyridin-2-yl)

pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 41% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}{}_{\rm D} = +0.1$ (*c* 0.55, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.65 (s, 1H), 8.51 (d, *J* = 3.6 Hz, 1H), 7.85 (d, *J* = 7.8 Hz, 1H), 7.27 (s, 1H), 4.42 (s, 1H), 4.02 (m, 1H), 3.95-3.82 (m, 5H), 2.76 (br, 1H), 2.56 (m, 1H), 2.35 (m, 1H), 1.08 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.64, 36.81, 52.50, 58.88, 60.88, 65.23, 65.91, 82.27, 123.44, 135.71, 148.80, 149.38, 172.32, 173.80; IR (KBr) v 3684, 3620, 3019, 2976, 2400, 1736, 1521, 1218, 1046, 743 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 72% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, $\lambda = 254$ nm); t_r = 15.30 and 18.62 min.



(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 5-(4-chlorophenyl)-4-((*S*)-hydroxy(phenyl) methyl)pyrrolidine-2,4-dicarboxylate 4ca and (2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 5-(4-chlorophenyl)-4-((*R*)-hydroxy(phenyl)methyl) pyrrolidine-2,4-dicarboxylate 4ca'

The title compounds **4ca** and **4ca**'were prepared according to the general procedure as described above and separated by silica gel column as colorless oil, and the absolute configuration of the stereogenic center bearing hydroxyl group has not been determined. For one diastereomer: $[\alpha]^{25}_{D} = -4.2$ (*c* 0.46, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.46-7.26 (m, 9H), 4.88 (s, 1H), 4.85 (s, 1H), 3.85 (t, *J*=9.3 Hz and

8.1 Hz, 1H), 3.78 (s, 3H), 2.54 (m, 1H), 2.45 (m, 1H), 1.04 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.37, 35.24, 52.32, 58.36, 63.98, 67.89, 76.70, 82.95, 127.38, 128.15, 128.40, 129.74, 133.32, 139.69, 140.44, 172.68, 174.19; IR (KBr) v 3683, 3620, 3019, 2976, 2400, 1736, 1521, 1423, 1217, 759 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 210 nm); t_r = 7.06 and 20.49 min. For the other diastereomer: [α]²⁵_D = -2.9 (*c* 1.01, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.39-7.26 (m, 9H), 5.21 (s, 1H), 4.84 (s, 1H), 3.83 (m, 1H), 3.76 (s, 3H), 2.72-2.52 (m, 2H), 0.97 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.34, 33.21, 46.20, 52.24, 58.30, 64.23, 66.89, 74.85, 82.02, 89.19, 127.66, 128.23, 129.20, 133.40, 138.03, 140.71, 143.50, 170.91, 174.09; IR (KBr) v 3684, 3620, 3019, 2975, 2400, 1736, 1520, 1422, 1216, 1046, 762 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 85% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm); t_r = 16.39 and 30.48 min.

PhOCO Bu⁴O₂C¹/_{*} *p*-CI-Ph¹¹*N^{*} CO₂CH₃ OPh (5)

(2*R*,4*R*,5*R*)-4-*tert*-butyl 2-methyl 1-benzoyl-4-(benzoyloxymethyl)-5-(4chlorophenyl)pyrrolidine-2,4-dicarboxylate

Under argon atmosphere **4ba** (369 mg, 1 mmol) was dissolved in 5 mL DCM, and PhCOCl (352 mg, 2.5 mmol), TEA (252 mg, 2.5 mmol) were added sequentially. The mixture was stirred at room temperature for 4h. Once starting material was consumed (monitored by TLC), the residue was purified by column chromatography to give **5** in 94% yield, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess. $[\alpha]^{25}_{D} = -10.6$ (*c* 1.00, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.98 (d, *J* = 7.8 Hz, 1H), 7.62-7.01 (m, 13H), 4.74 (m, 2H), 4.55 (s, 1H), 4.43 (m, 1H), 3.88 (s, 3H), 2.88 (t, *J* = 12.9 Hz and 12.0 Hz, 1H), 2.62 (s, 1H), 1.06 (s, 9H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 27.42, 30.52, 52.61, 58.29, 59.66, 66.30, 67.54, 82.61, 126.65, 128.13, 128.44, 128.62, 129.06, 129.63, 129.96, 130.07, 133.62, 134.13, 135.28, 137.31, 165.84, 167.77, 171.50, 172.15; IR (KBr) v 3683, 3620, 3019, 2977, 2400, 1737, 1436, 1370, 1216, 1046, 758 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralcel AD-H, *i*-propanol/hexane = 20/80, flow rate 0.5 mL/min, λ = 210 nm); t_r = 19.86 and 24.18 min.

The absolute configuration of the cycloadduct (2R,4R,5R)-5 was determined by X-ray diffraction analysis

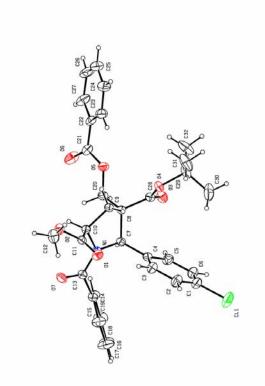


Figure 1. X-ray structure of (2*R*,4*R*,5*R*)-**5**

Crystal data for (2R,4R,5R)-**5**: C₃₂H₃₂ClNO₇, M_r = 578.04, T = 293 K, Orthorhombic, space group P2(1)2(1)2, a = 19.401(3), b = 24.000(6), c = 6.6143(9) Å, V = 3079.8(7) Å³, Z = 4, 6359 reflections measured, 4817 unique ($R_{int} = 0.0412$) which were used in all caclculations. The final $wR_2 = 0.0844$ (all data). Flack $\chi = 0.03(8)$. CCDC 813952 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the

Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

Proposed transition state of the *endo*-selectivity for asymmetric 1,3-dipolar cycloaddition of imino esters with Morita-Baylis-Hillman adduct

Based on the relative and absolute configuration of (2R,4R,5R)-5 and previous studies,^[1,3] a plausible transition state accounting for the observed *endo*-selectivity of the 1,3-DC addition of imino esters with Morita-Baylis-Hillman adduct in the presence of Cu(CH₃CN)₄BF₄/(*S*)-TF-BiphamPhos (**1a**) is shown in Figure 2. The *in situ*-formed azomethine ylide is coordinated to the metallic center and oriented in such way because of the steric repulsion between the phenyl group in the ylide and the phenyl ring on the phosphorus atom of the chiral ligand, and the MBH adduct **2** approached the *Si* (C=N) face of the azomethine ylide and forms the *endo*-cycloadduct, which is compatible with the experimental results. The carbonyl group of Morita-Baylis-Hillman adduct could coordinate with the Cu(I) center, which can stabilize the negatively charged oxygen atom in the proposed transition states.^[4] It could not rule out the possible hydrogen bond interaction between the carbonyl group of dipolarophile (**2**) and the NH₂ group of the chiral (*S*)-TF-BiphamPhos ligand (**1a**), which also facilitates stabilizing the proposed transition states.^[3b,3c] Nevertheless, the real catalytic mechanism still needs further investigation.

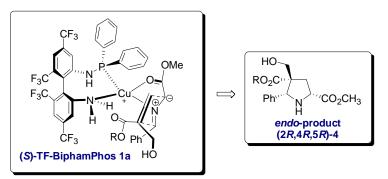


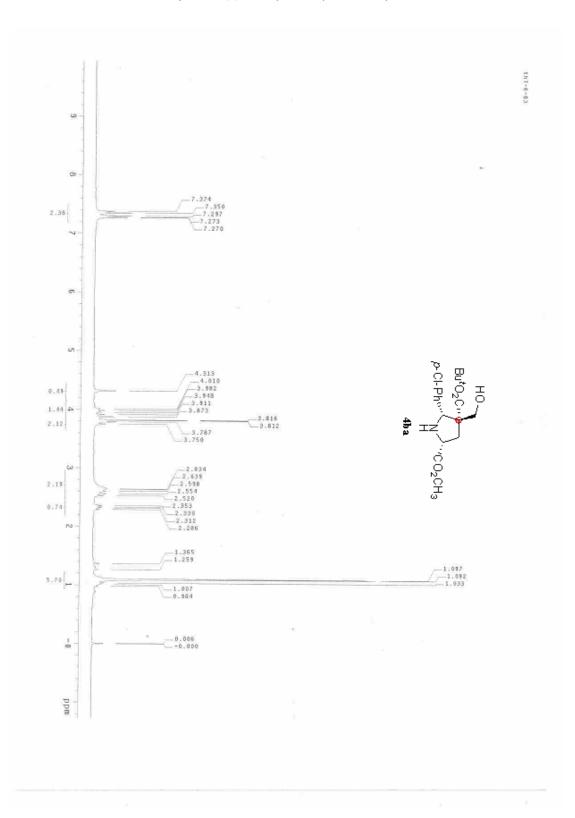
Figure 2. Proposed transition states leading to *endo-*(2*R*,4*R*,5*R*)-cycloadduct while using Morita-Baylis-Hillman adducts as the dipolarophiles.

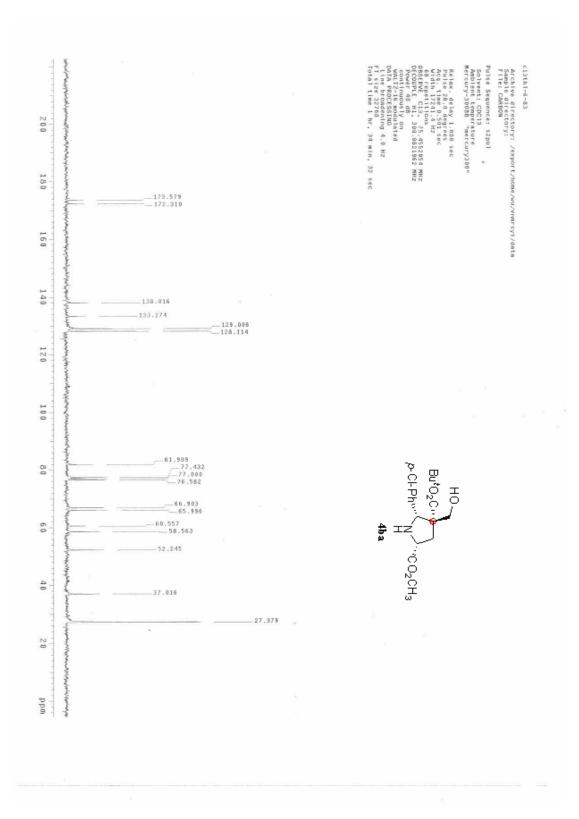
References

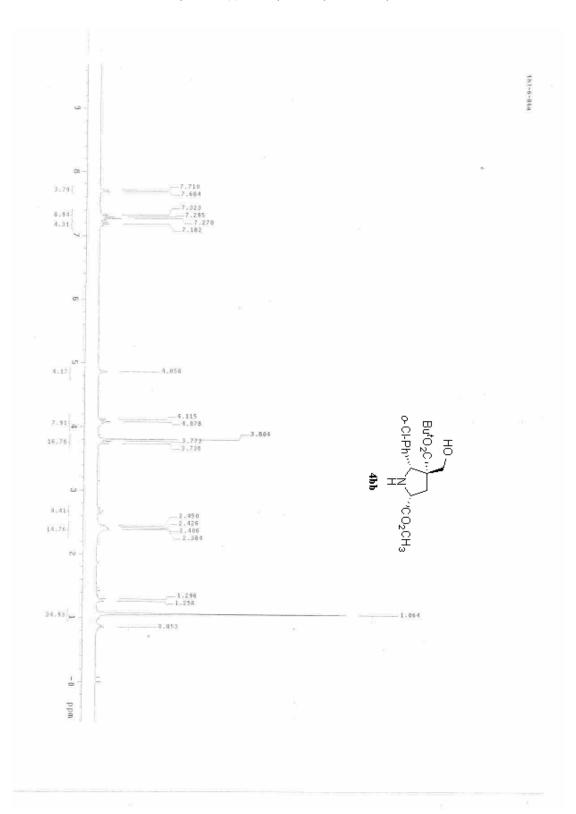
a) C.-J. Wang, G. Liang, Z.-Y. Xue, F. Gao, J. Am. Chem. Soc. 2008, 130, 17250; b)
 C.-J. Wang, Z.-Y. Xue, G. Liang, Z. Lu, Chem. Commun. 2009, 2905; c) G. Liang,
 M.-C. Tong, C.-J. Wang, Adv. Synth. Catal. 2009, 351, 3101; d) Z.-Y. Xue, T.-L. Liu,
 Z. Lu,; H. Huang, H.-Y. Tao, C.-J. Wang, Chem. Commun. 2010, 46, 1727.

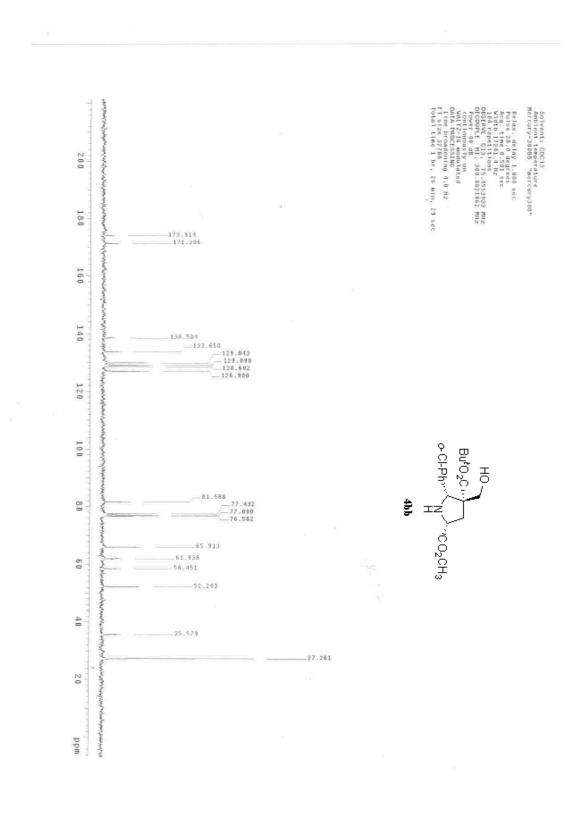
2. a) H. Huang, X. Liu, J. Deng, M. Qiu and Z. Zheng, *Organic. Letters.*, **2006**, *8*, 3359; b) W. -D. Lee, K. -S. Yang and K. Chen, *Chem. Commun.*, 2001, 1612.

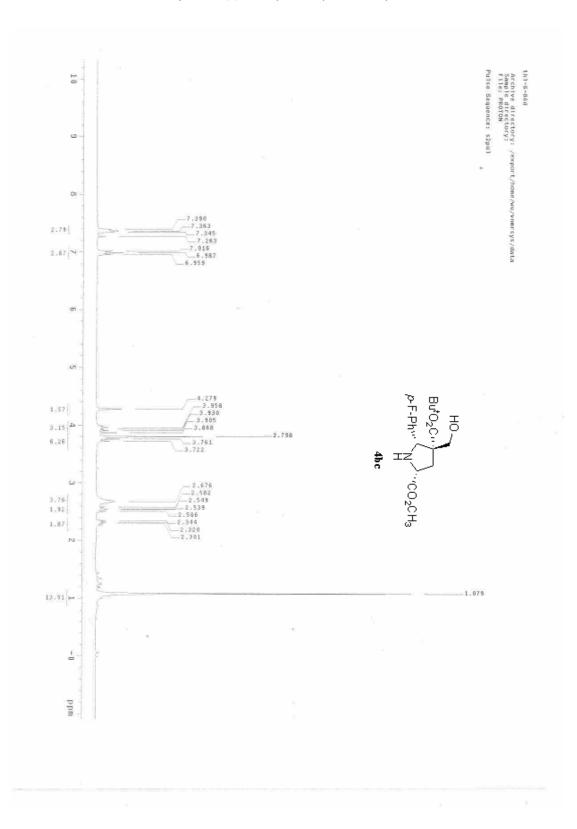
3 a) S. Cabrera, R. G. Arrayás, B. Martín-Matute, F. P. Cossío, J. C. Carretero, *Tetrahedron* 2007, 63, 6587; b) W. Zeng, G.-Y. Chen, Y.-G. Zhou, Y.-X. Li, J. Am. Chem. Soc. 2007, 129, 750; c) H. Y. Kim, H.-J. Shih, W. E. Knabe, K. Oh, Angew. Chem., Int. Ed. 2009, 48, 7420; d) J. M. Longmire, B. Wang, X. Zhang, J. Am. Chem. Soc. 2002, 124, 13400; e) W. Gao, X. Zhang, M. Raghunath, Org. Lett. 2005, 7, 4241.
4 X.-X. Yan, Q. Peng, Y. Zhang, K. Zhang, W. Hong, X.-L. Hou, Y.-D. Wu, Angew. Chem., Int. Ed. 2006, 45, 1979.

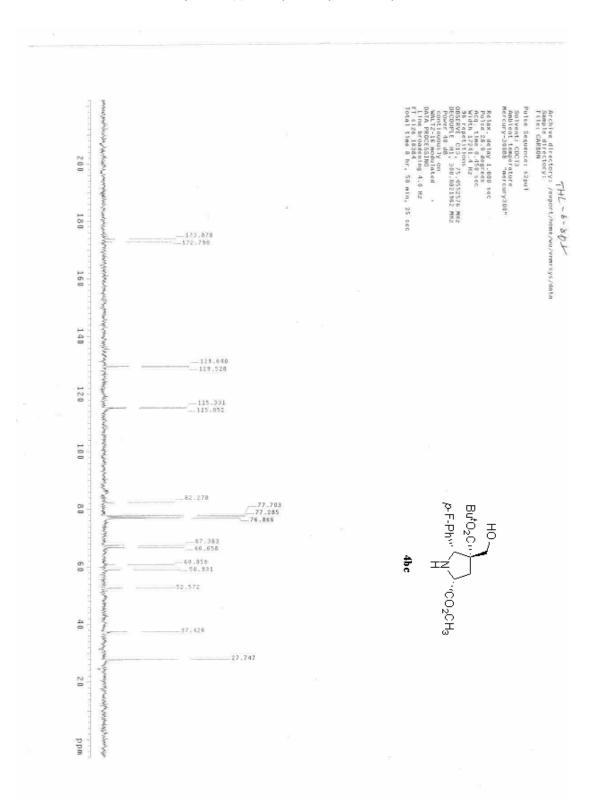


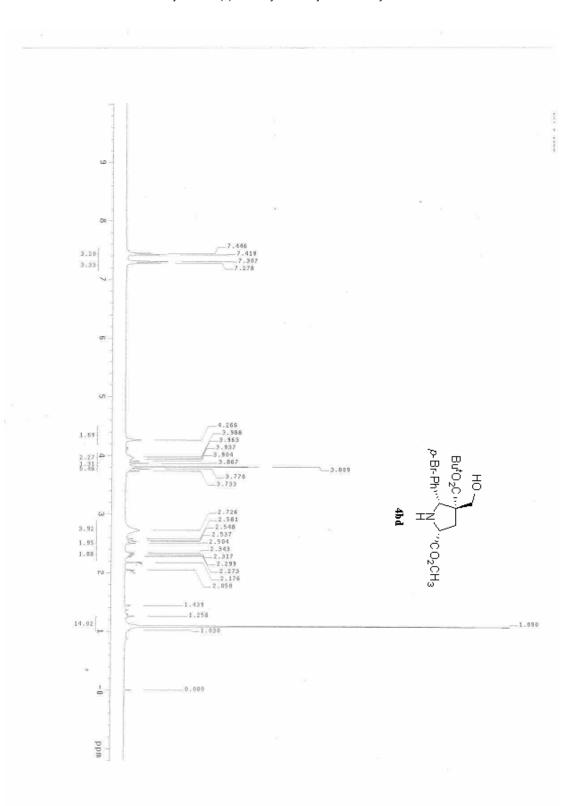


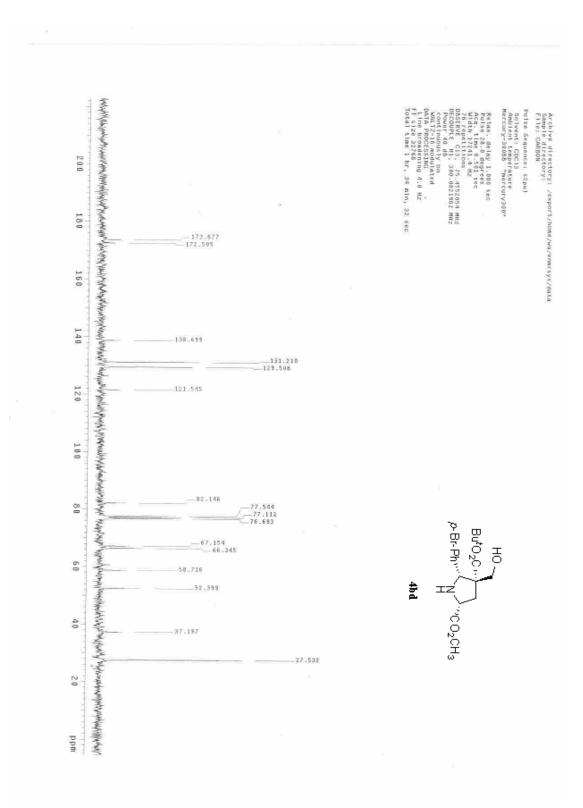


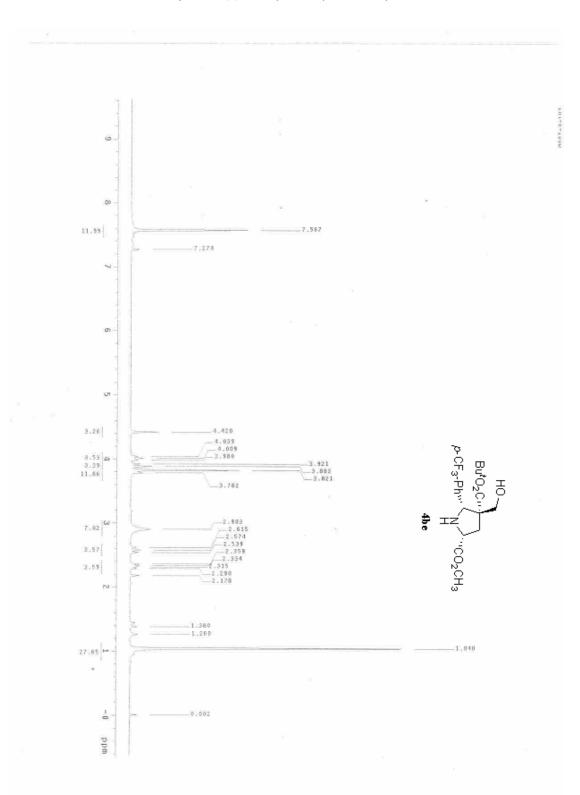


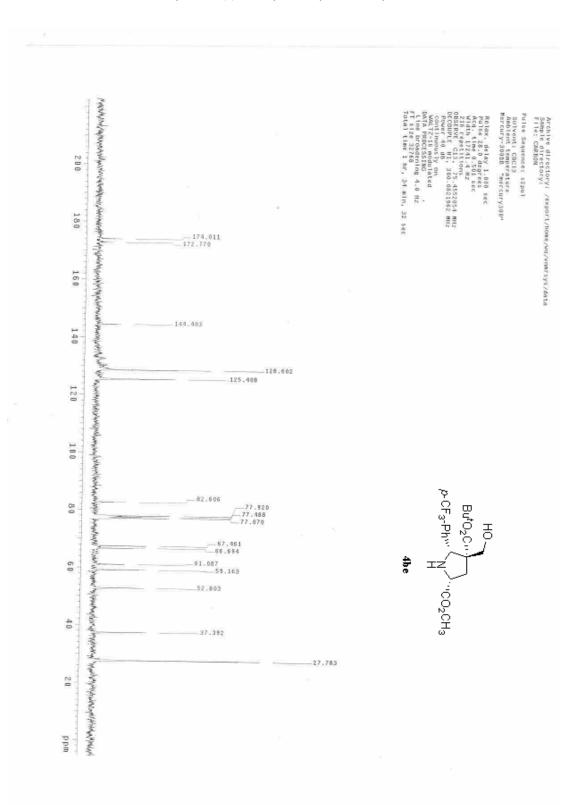


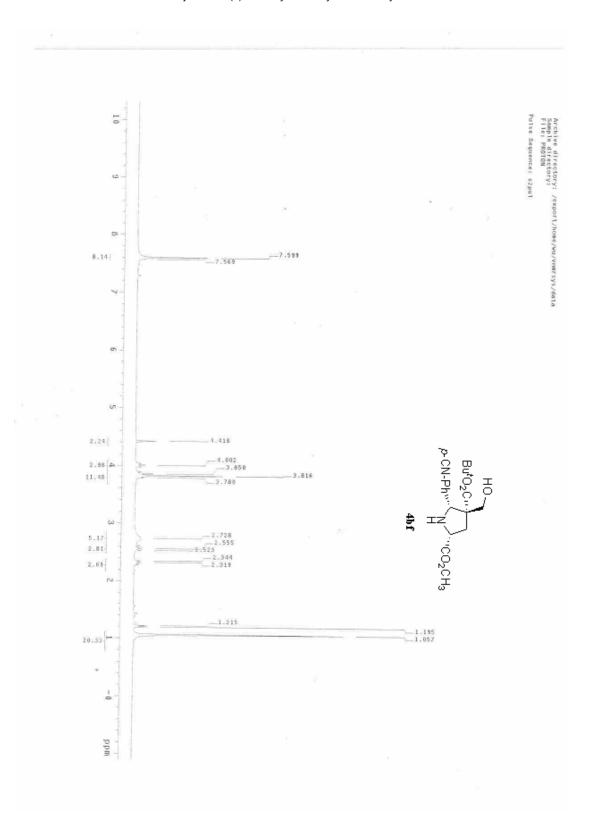


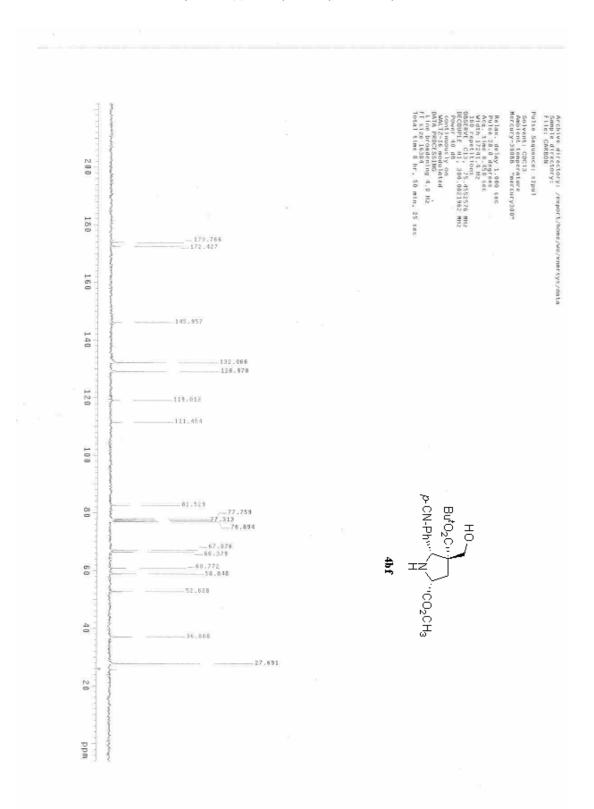


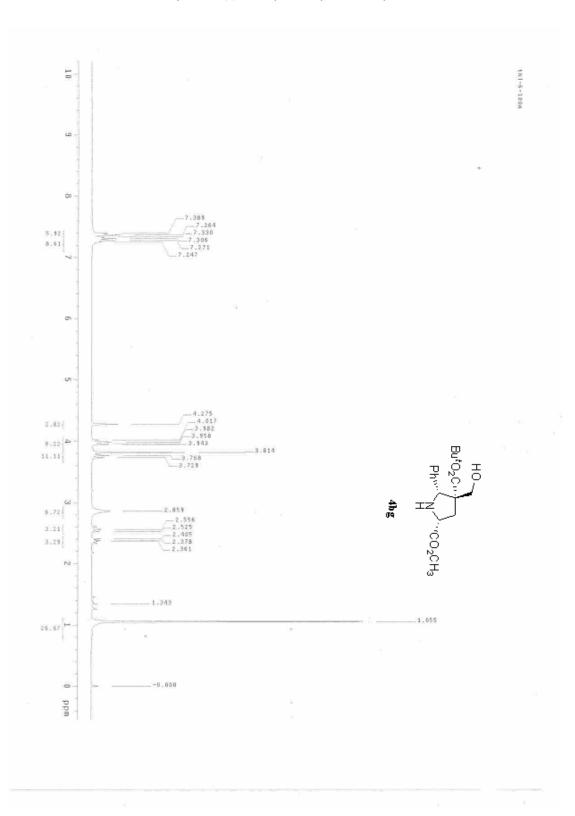




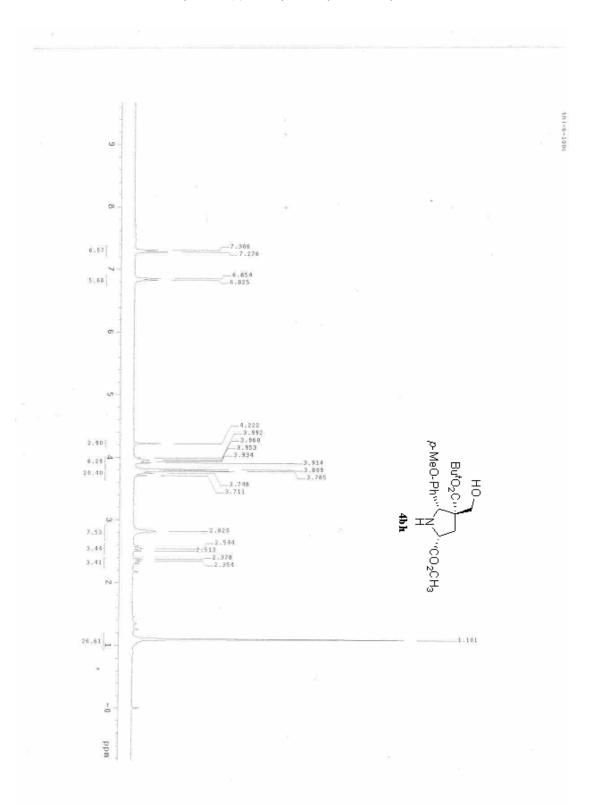


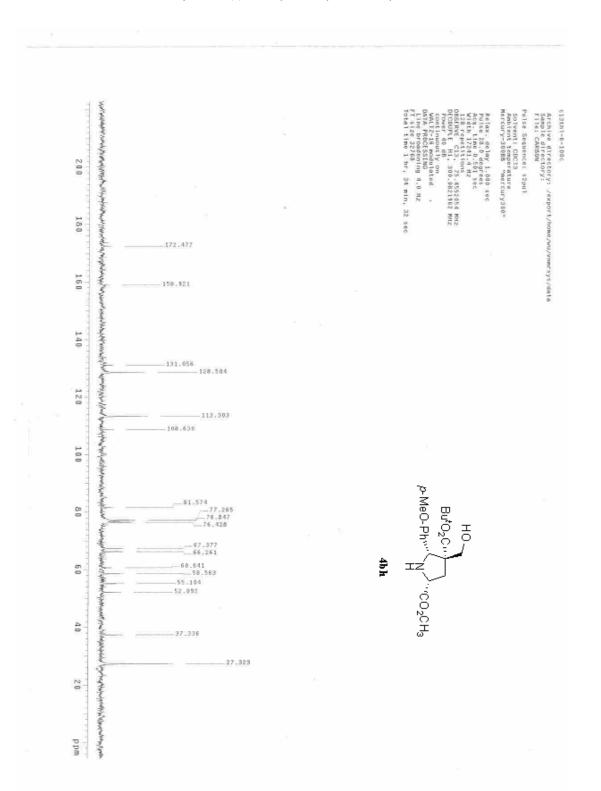


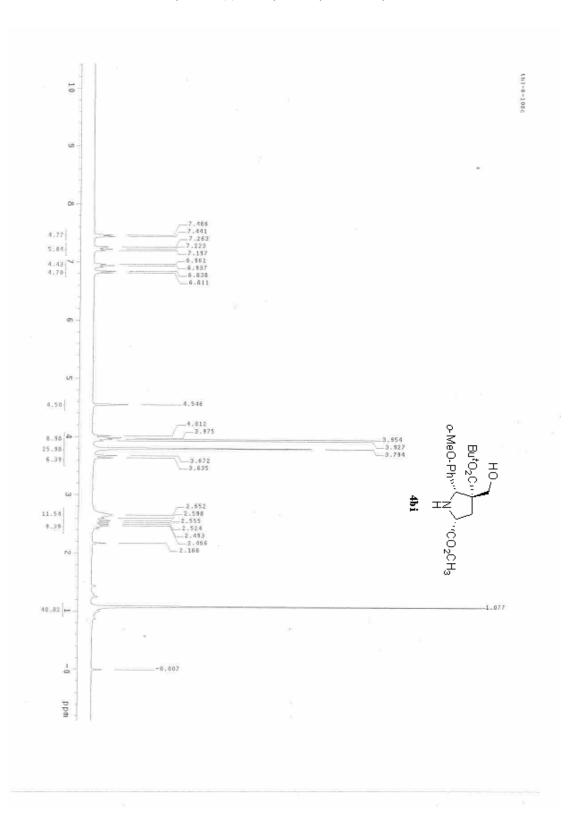


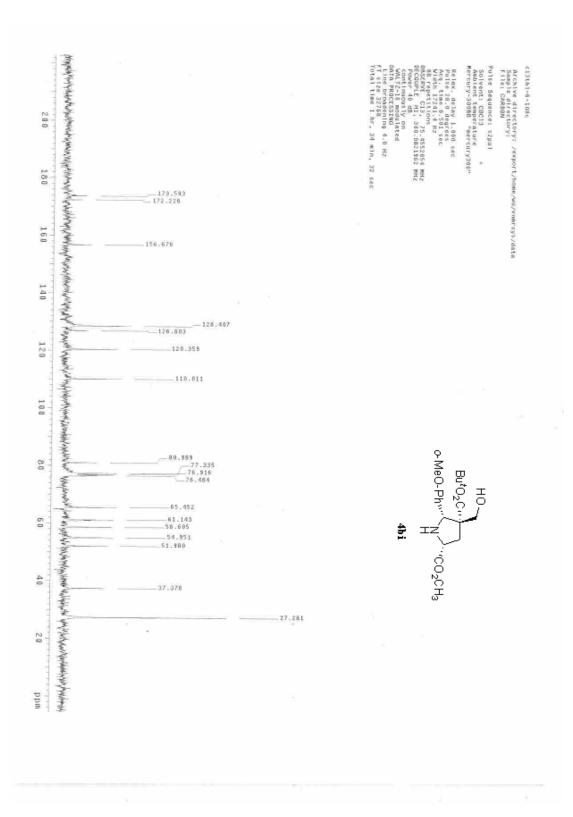


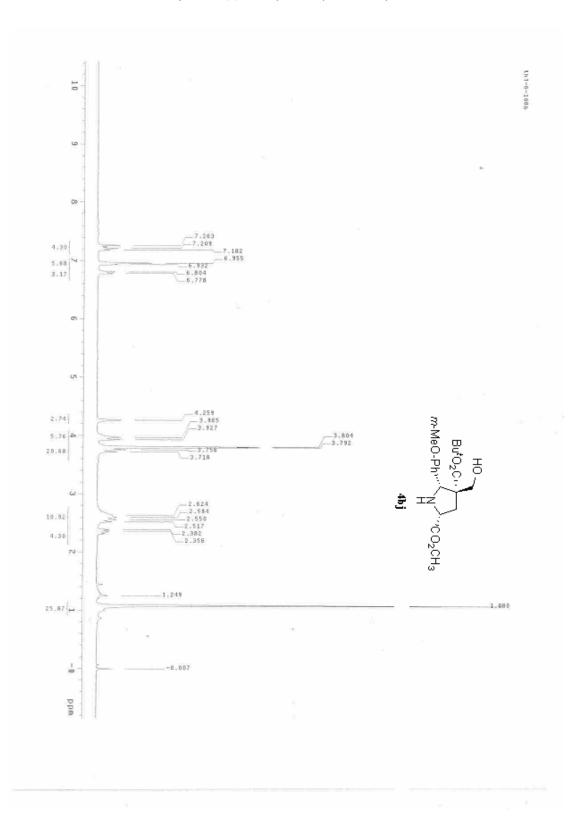


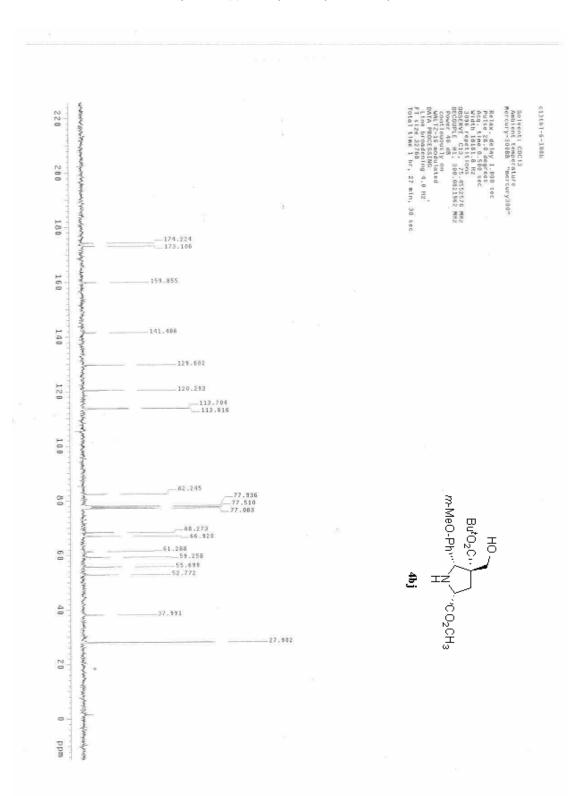


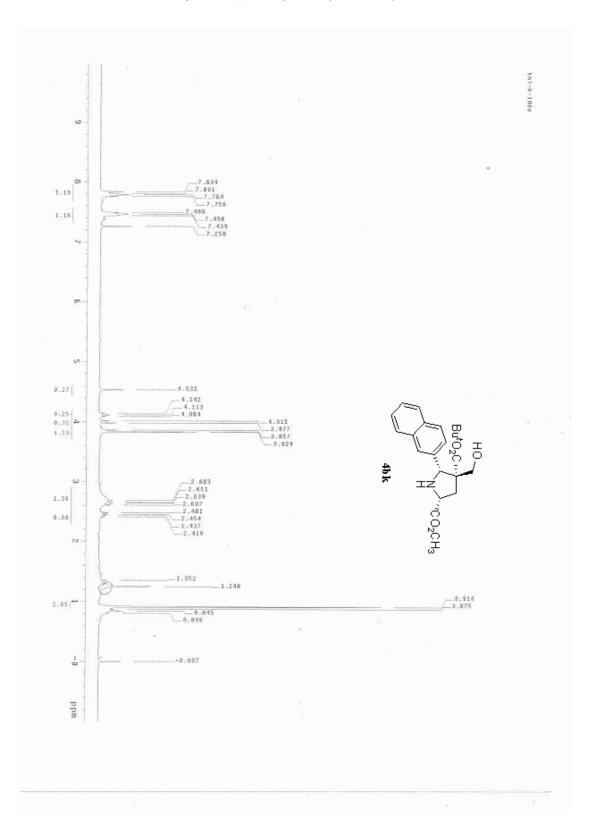


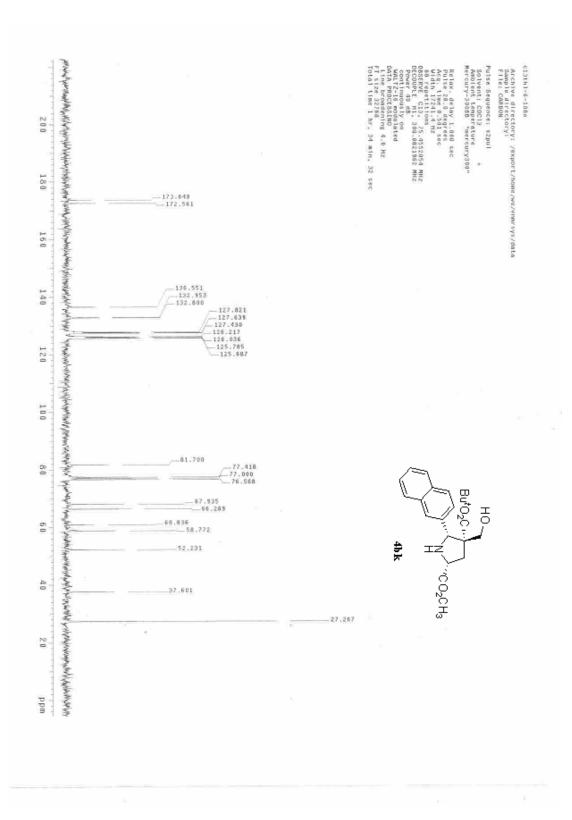


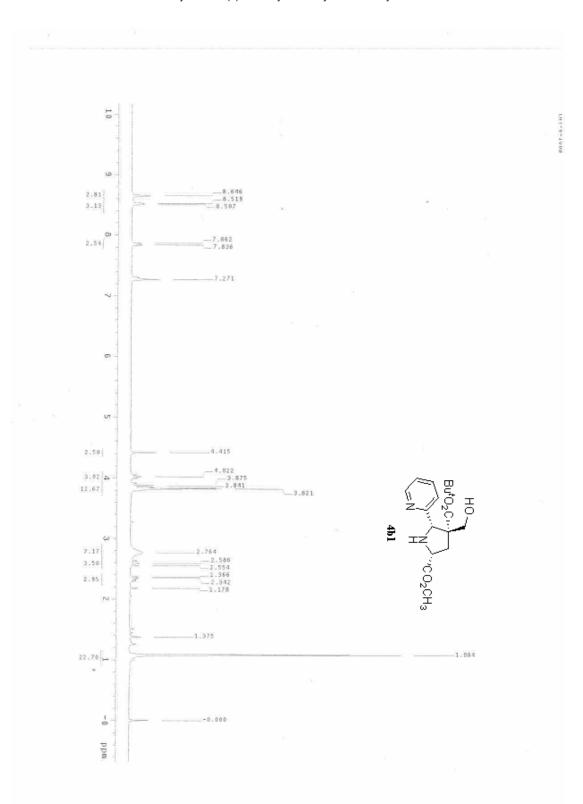


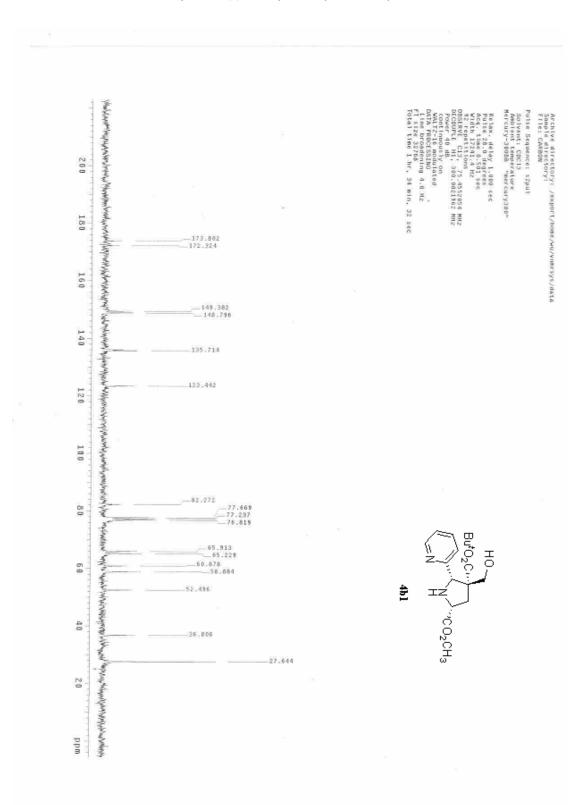


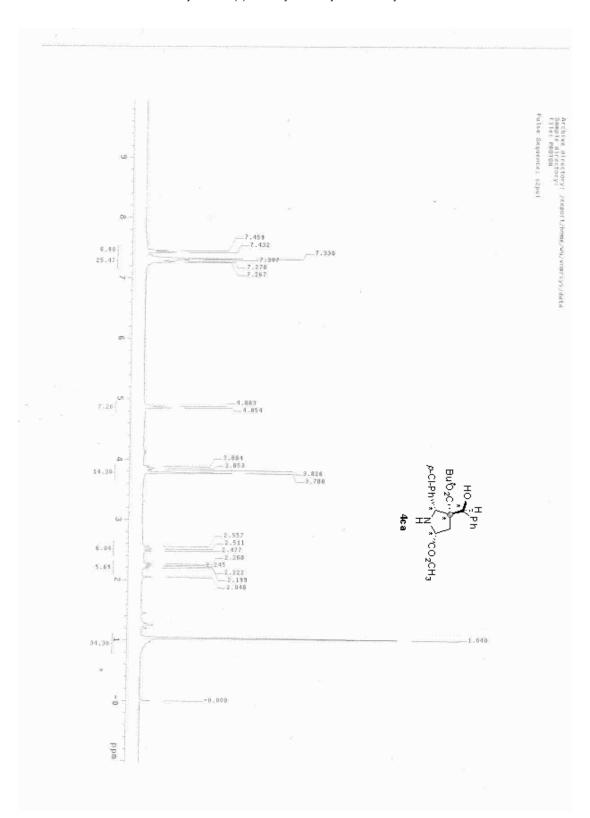




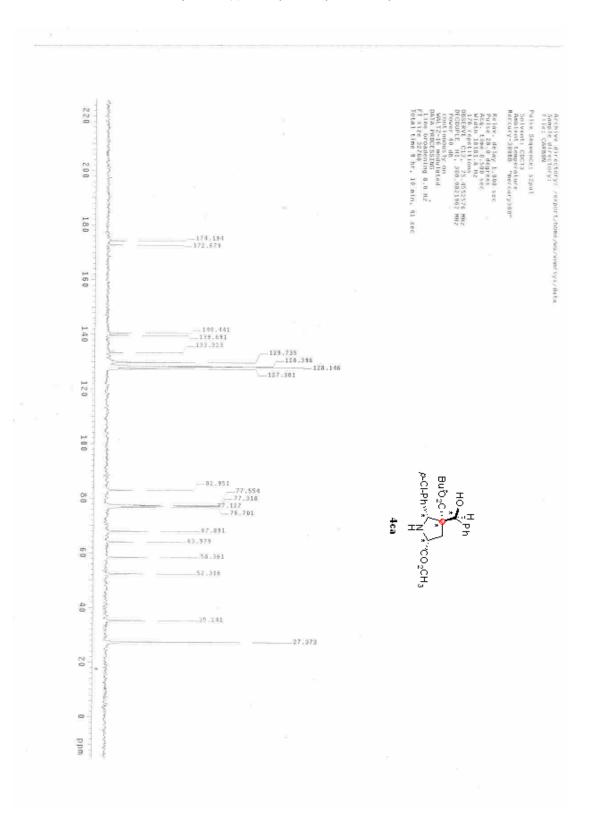


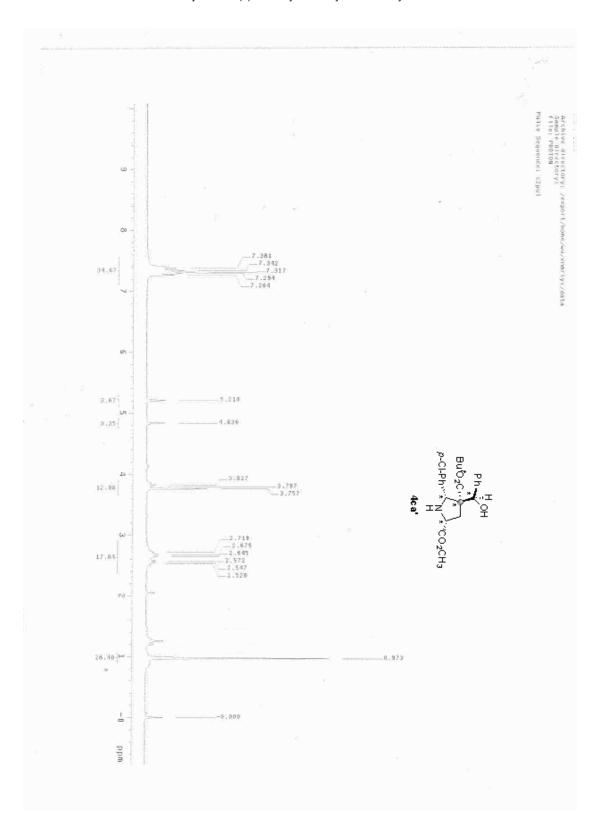


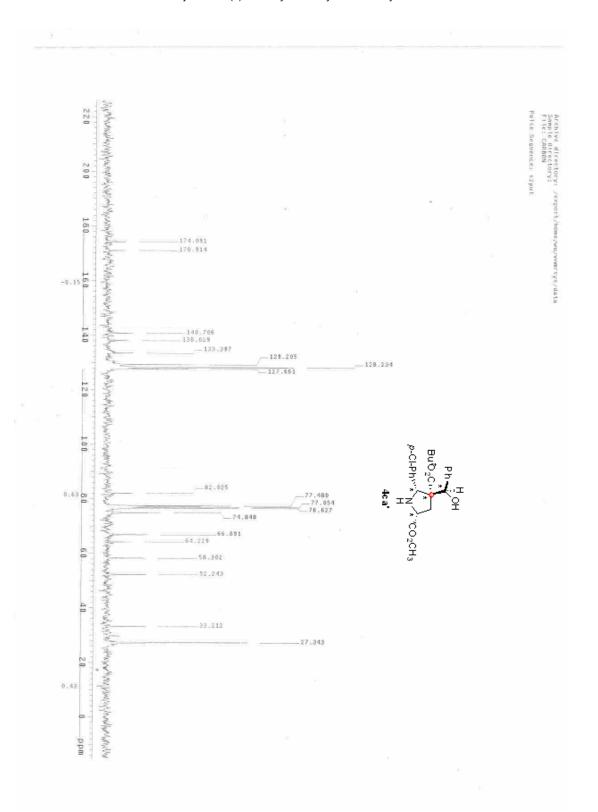


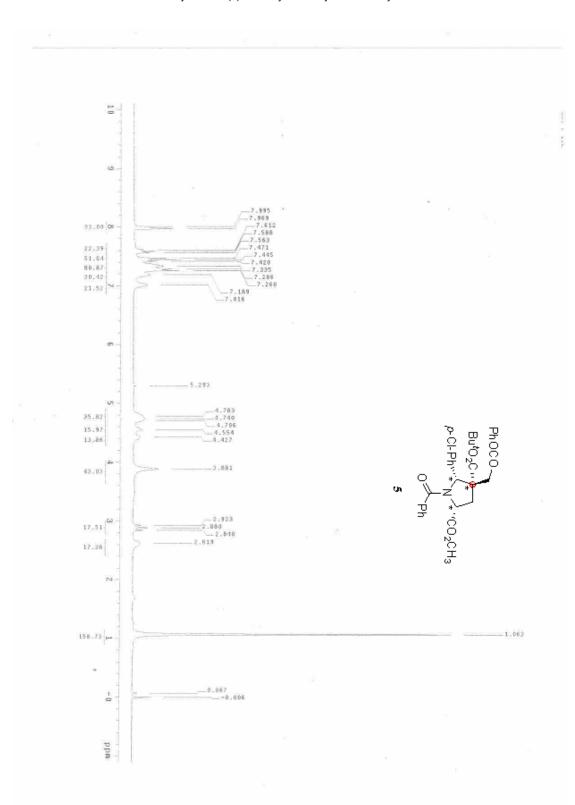


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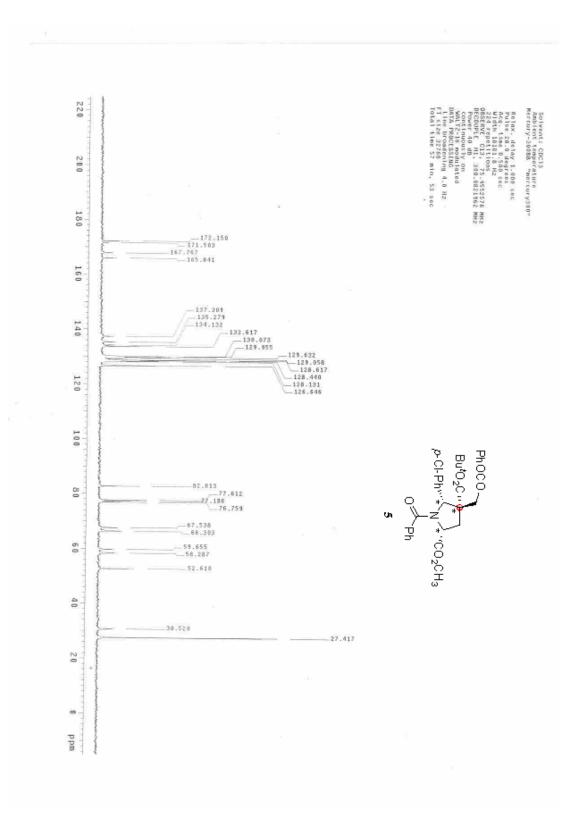




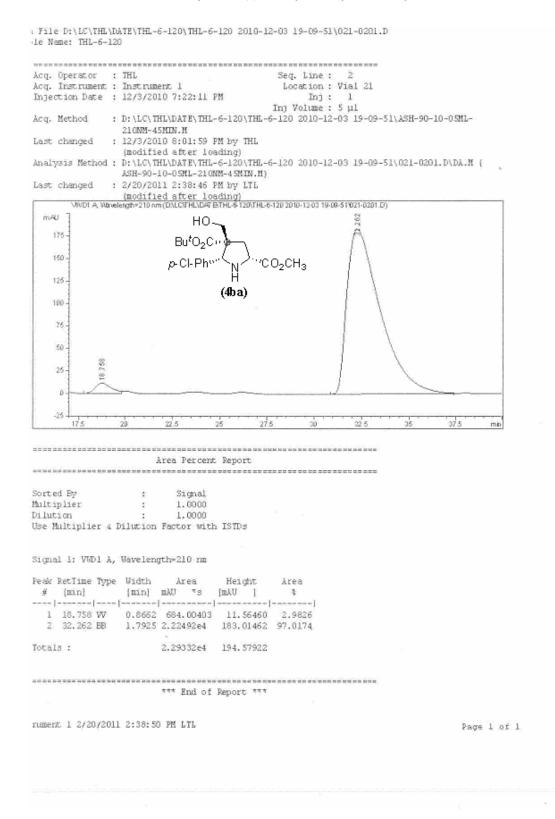




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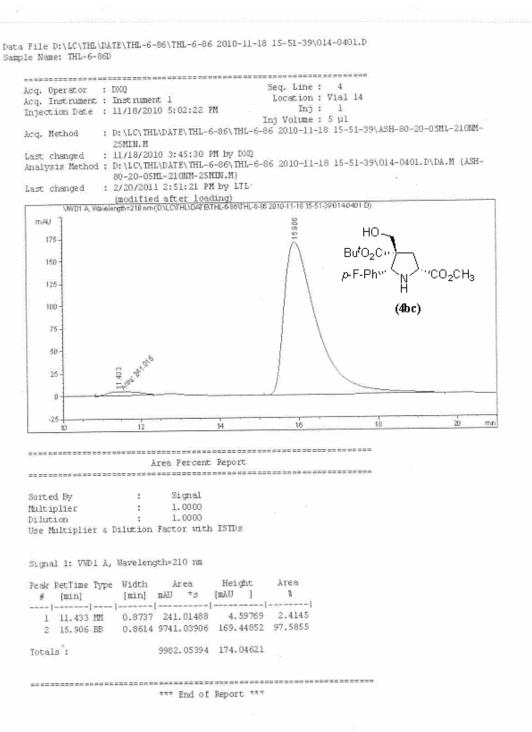
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Instrument 1 2/20/2011 2:51:30 PM LTL

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Last changed	: 11/6/2010 10:09	:43 AM by TMC		
Analysis Method	: D:\LC\THL\DATE\ DA.M (ASH-80-20		E-6-86DRAC 2010	11-17 17-55-53\005-0101.D\
Last changed	: 2/20/2011 2:49: (modified after	and the second sec		
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10 Sorted By Multiplier Dilution Use Multiplier of Signal 1: VUD1) Peek RetTime Typ # [min]	Area Per : Sign : 1.00 : 1.00 : Dilution Factor L, Wavelength=210 be Width Area [min] mAU *	cent Report al 000 with ISTDs nm Height s [mAU]	Årea 8	
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Instrument 1 2/20/2011 2:49:58 PM LTL

Data File D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\061-0201.D Sample Name: THL-6-108A

le Name: THL-6-10	88
Acq. Operator :	THL Seq. Line : 2
Acq. Instrument :	Instrument 1 Location : Vial 61
Injection Date :	11/29/2010 4:12:24 PM In In 1: 4
	Ini Volume : S ul
Acq. Method :	D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\ASH-80-20-05ML-
	210NM-25HIN.M
Last changed :	11 (18/2010 3.45.30 DM by DVD
Inalysis Method :	D: \LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\061-0201.D\DA.M (
the second s	ASH-80-20-05ML-210NM-25MIN.M)
Last changed	2/20/2011 3:16:59 PM by LTL
	(-dified off or loading)
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Peak Retline Typ	
# [min]	[min] mAU *s [mAU] %
1 11.269 W	0.7109 960.58899 19.92666 4.9559
2 16.959 BB	0.9267 1.84223e4 296.13754 95.0441
	3 00000-4 016 06401
Totals :	1,93829e4 316.06421

	*** End of Report ***

Instrument 1 2/20/2011 3:17:03 PM LTL

Data File D:\LC\THL\DATE\THL-6-108&P&C\THL-6-108&-P&C 2010-11-27 14-48-47\071-0101.D Sample Name: THL-6-108&-P&C

Acq. Operator : T		************	Seq. Line :	1	
kcq. Instrument : I	not rement. 1.		Location :	Vial 71	
Injection Date : 1	1/27/2010 2:50:1	4 FM	Inj:		
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	modified after 1	oading)		8. ALC 25. 11. ALC 21. ALC 2	0101-021-0101
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ast changed : 2	2/20/2011 3:11:25	PM by LTL			
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Data File D:\LC\THL\DATE\THL-6-100\THL-6-100 2010-11-22 15-52-59\001-0201.D Sample Name: THL-6-100B

Acq. Operat	
	ment : Instrument 1 Location : Vial 1
Injection D	Pare : 11/22/2010 4:05:19 PM Inj : 1
	Inj Volume : 5 µl
log. Method	
	210NM-20NIN.H
last change	d : 11/22/2010 3:49:52 FM by tmc
knalysis Me	thod : D:\LC\THL\DATE\THL-6-100\THL-6-100 2010-11-22 15-52-59\001-0201.D\DA.M (
	ASH-80-20-05ML-210MM-20MIN.M)
Last change	
	(modified after loading)
	01 A, Vilavelength=210 nm(DNLCTHLNDAT BTHL-6-100/THL-6-100 2010-11-22 15-52-69/001-0201.0)
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Sorted By Multiplier Dilution	: 1.0000 : 1.0000
Sorted By Multiplier Dilution Use Multipl	: 1.0000 : 1.0000 lier & Dilution Factor with ISTDs
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Sorted By Multiplier Dilution Use Multipl Signal 1: V Peak RetTim # [min]	: 1.0000 : 1.0000 lier & Dilution Factor with ISTDs WWD1 &, Wavelength=210 nm me Type Width Area Height Area [[min] mAU *s [mAU] %
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*** End of Report ***

Instrument 1 2/20/2011 3:01:59 FM LTL

Data File D:\LC\THL\DATE\THL-6-100A-RAC\THL-6-100A-FAC 2010-11-21 15-49-47\008-0201.D Sample Name: thl-6-100b-rac

kcq. Operator :	
Acc. Uperator :	THL Seg. Line : 2
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Acq. Instrument :	
Injection Date :	11/21/2010 4:17:40 PM Inj: 1
	Inj Volume : 5 µl
kcq. Method :	D:\LC\THL\DATE\THL-6-100A-RAC\THL-6-100A-RAC 2010-11-21 15-49-47\ASH-80-
	20-05ML-210MM-30MIN.M
Last changed :	11/21/2010 4:16:23 PM by THL
	(modified after loading)
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anaryono memoria i	D\DA.M (ASH-80-20-0.5ML-210NM-30MIN.M)
Last changed :	2/20/2011 2:56:20 PM by LTL
ase changed .	(modified after loading)
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Jse Hultiplier a	Dilution Factor with ISTDs
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Jse Multiplier a Signal 1: VWD1 & Peak RetTime Typ	, Wavelength=210 nm e Width Årea Height Årea
Jse Multiplier & Signal 1: VWD1 Å Peak RetTime Typ ∉ [min]	, Wavelength=210 nm g Width Årea Height Årea [min] mAU *s [mAU] %
Jse Multiplier 6 Signal 1: VWD1 & Fesk RetTime Typ # [min]	, Wavelength=210 nm e Width Årea Height Årea [min] mAU *s [mAU] % {
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Jse Multiplier & Signal 1: VUD1 Å Peak RetTime Typ # [min] 	, Wavelength=210 nm e Width Årea Height Årea [min] mAU *s [mAU] % -[
Jse Multiplier a Signal 1: VWD1 A Peak RetTime Typ # [min] 1 9.168 BB 2 12,823 BB	, Wavelength=210 nm e Width Area Height Area [min] mAU *s [mAU] %
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Use Multiplier 6 Signal 1: VWD1 Å Feak RetTime Typ ∉ [min] 1 9.168 EB 2 12.823 EB Totals :	Wavelength=210 nm Width Area Height Area [min] mAU *s [mAU] % 0.3792 943.40753 36.79168 50.7350 0.6295 916.07239 21.75875 49.2650 1859.47992 58.55042
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Page 1 of 1

Instrument 1 2/20/2011 2:56:25 PM LTL

Data File D:\LC\THL\DATE\THL-6-86E-GH\THL-6-86E-GH 2010-11-20 09-02-37\015-0201.D Sample Name: THL-6-86E

Acq. Operator : THL	Seq. Line : 2
lea Instrument : Instrument	1 Location : Vial 15
Injection Date : 11/20/2010	9:14:59 AM Inj. 1 Inj Volume : 5 µl
 J. 107 No. 10, an architecture of 	DATE/THL-6-86E-GH/THL-5-86E-GH 2010-11-20 09-02-37/ADH-80-20-
05ML-230M	0.00.21 JW by 7H
Last changed : 11/20/2010	DATE\THL-6-86E-GH\THL-6-86E-GH 2010-11-20 09-02-37\015-0201.D\
ANALYSIS MECHOD . D. MC(100)	-80-20-05ML-230NM.M)
Last changed : 2/20/2011	2:54:30 PM by LTL
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0- 24	20 20 20 20 20 20 20 20 20 20 20 20 20 2
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0 24 Xr Sorted By : Multiplier : Dilution :	20 28 28 20 20 20 20 20 20 20 20 20 20 20 20 20
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Instrument 1 2/20/2011 2:54:35 FM LTL

Data File D:\LC\THL\DATE\THL-6-86ERAC\THL-6-86ERAC 2010-11-20 09-54-14\006-0101.D Sample Name: THL-6-85ERAC

Instrument 1 2/20/2011 2:53:19 FM LTL

Data File D:\LC\THL\DATE\THL-6-100\THL-6-100 2010-11-22 15-52-59\016-0601.D Sample Name: THL-6-100Å

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Instrument 1 2/20/2011 2:59:36 FM LTL

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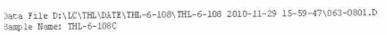
Data File D:\LC\THL\DATE\THL-6-100\THL-6-100 2010-11-22 15-52-59\002-0401.D Sample Name: THL-6-100C

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Sorted By Multiplier Dilution Use Multiplier Signal 1: VUD1 Peak RetTime Ty # [min] 1 19.421 EB	ی : : 4 Dilution I A, Wavelengt pe Width [min] r -1	rea Percent Signal 1,0000 1,0000 Factor with Area AU *s 174,64624 89,90903	Height [mAU] 55,98981	Årea ٩ 			
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VUD1 Peak RetTime Ty # [min] 	۸ : : 4 Dilution 1 ۸, Wavelengt pe Width [min] r 0.8741 : 1.3586	rea Percent Signal 1.0000 1.0000 Vactor with h=210 nm Area Area Area 3174.64624 89.90903 3264.55527	Height (mAU 1 55.98981 1.10300 57.09281	Årea %] 97.2459 2.7541			

58

Data File D:\LC\THL\DATE\THL-6-100C-RAC\THL-6-100C-RAC 2010-11-21 19-45-51\009-0101.D Sample Name: THL-6-108C-RAC

Injection Date : Acq. Method : Last changed : Analysis Method : Last changed :	<pre>: THL Seq. Line : 1 : Instrument 1 Location : Vial 9 : 11/21/2010 7:47:22 PM Inj : 1 Inj Volume : 5 µ1 : D:\LC\THL\DATE\THL-6-100C-RAC\THL-6-100C-RAC 2010-11-21 19-45-51\0DH-80- 20-0 SML-210NM.M : 11/21/2010 8:18:26 PM by THL (modified after loading) : D:\LC\THL\DATE\THL-6-100C-RAC\THL-6-100C-RAC 2010-11-21 19-45-51\009-0101, D\DA.M (0DH-80-20-0 SML-210NM.M) : 2/20/2011 3:03:33 PH by LTL (modified after loading) elergh=210nm(CMLCMHL0ATENTHL-6-100C-RAC 2010-11-21 19-45-510090101.0)</pre>
100 - 30 - 60 -	$ \begin{array}{c} HO \\ Bu'O_2C'' \\ p-MeO-Ph''' \\ H \\ H \end{array} $ (4bh)
40 - 20 - 0 -	
18	20 22 24 26 28 min
Sorted By Multiplier Dilution	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs
Sorted By Multiplier Dilution Use Multiplier 4	: Signal : 1.0000 : 1.0000
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VUD1 4 Peak RetTime Typ # [min]	: Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs ., Wavelength=210 mm
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VUD1 4 Peak RetTime Typ # [min]	: Signal : 1.0000 : 1.0000 : Dilution Factor with ISTDs we Width Area Height Area [min] mAU *s [mAU] % -[]] 0.8868 6220.93457 107.18763 50.1816
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VUD1 & Peak RetTime Typ # [min] 	: Signal : 1.0000 : 1.0000 : Dilution Factor with ISTDs ., Wavelength=210 nm we Width Area Height Area [min] mAU *s [mAU] % -[] 0.8868 6220.93457 107.18763 50.1816
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VUD1 Å Pesk RetTime Typ # [min] [[1 19.301 EB 2 25.615 BB Totals :	: Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=210 nm we Width Area Height Area [min] mAU *s [mAU] % -[] 0.8868 6220.93457 107.18763 50.1816 1.2126 6175.89697 78.07592 49.8184



Acq. Operator	
Acq. Instrument	: Instrument 1 Location : Vial 63
Injection Date	: 11/29/2010 6:51:22 PM In): 1
	Ini Volume : 5 ul
Acq. Method	: D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\0DH-80-20-05ML- 210MM-30MUW.M
Last changed	• 11/29/2010 8•55:30 M by THL
Inglyspic Mathod	: D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\063-0801.D\DA.M (
Mugråsis Hernor	(DH-80-20-05NL-210NM-30NIN.M)
e construire de la construire	: 2/20/2011 3:23:52 PM by LTL
Last changed	(modified after loading)
VA/D1 A, IA5	relength=210 nm (DVLCTHL\DAT EXTHL-6-108/THL-6-108 2010-11-29 15-59-47/063-0801.D)
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min]	Ares Percent Report : Signal : 1.0000 : 1.0000 4 Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Height Area
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 4 Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Height Area [min] mAU *s [mAU] %
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	14 16 10 10 10 10 Ares Percent Report : 1.0000 1.0000 1.0000 : 1.0000 1.0000 1.0000 & Dilution Factor with ISTDs 1.0000 1.0000 k, Wavelength=210 nm 1.0000 1.0000 pe Width Area Height Area
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	14 10 10 10 Area Percent Report : 1.0000 : 1.0000 : 1.0000 4 10 0 10 i 1.0000 :
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	14 16 10 10 10 10 Ares Percent Report : 1.0000 1.0000 1.0000 : 1.0000 1.0000 1.0000 & Dilution Factor with ISTDs 1.0000 1.0000 k, Wavelength=210 nm 1.0000 1.0000 pe Width Area Height Area
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	14 10 10 10 10 10 Ares Percent Report : 1.0000 1.0000 1.0000 : 1.0000 1.0000 1.0000 4. Dilution Factor with ISTDs Å, Wavelength=210 nm pe Width Årea [min] mÅU *s
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Instrument 1 2/20/2011 3:23:57 PM LTL

₽age 1 of 1

)ata File D:\LC\THL\DATE\THL-6-108C-RAC\THL-6-108C-RAC 2010-11-29 08-57-25\001-0201.D 3ample Name: THL-6-108CRAC

land Oner at ar	: THL Seq. Line : 2
kcq. Operator	: Instrument 1 Location : Vial 1
	: 11/29/2010 9:09:48 AM Inj: 1
injection bace	Inj Volume : 5 µl
aw Nathod	: D: \LC\THL\DATE\THL-6-108C-RAC\THL-6-108C-RAC 2010-11-29 08-57-25\0DH-80-
sog. neurou	20-05ML-210MM-30MIN.M
Last changed	: 11/29/2010 8:55:30 AM by THL
Last changed	: D:\LC\THL\DATE\THL-6-108C-RAC\THL-6-108C-RAC 2010-11-29 08-57-25\001-0201.
andrysis neurou	D/DA.M (ODH-80-20-0 5ML-210NM-30HIN.M)
Last changed	: 2/20/2011 3:22:06 PM by LTL
-	(modified after loading)
\M/D1 A, W	avelength=210 nm (DNLCTHL\DAT B.THL-6 108C-RAC\THL-6-108C-RAC 2010 11-29 08-57-25001-0201.D)
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Sorted By Multiplier Dilution	Area Percent Report : Signal : 1.0000
Sorted By Multiplier Dilution	Area Percent Report : Signal : 1.0000 : 1.0000
Sorted By Multiplier Dilution Use Multiplier	Area Percent Report : Signal : 1,0000 : 1.0000 4 Dilution Factor with ISTDs
Sorted By Multiplier Dilution Use Multiplier	Area Percent Report : Signal : 1.0000 : 1.0000
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1	Area Percent Report : Signal : 1.0000 : 1.0000 4 Dilution Factor with ISTDs A, Wavelength=210 nm
Sorted By Multiplier Dilutica Use Multiplier Signal 1: VWD1 Peak RetTime Ty	Area Percent Report : Signal : 1.0000 : 1.0000 4 Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Height Area
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 4 Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Height Area [min] mAU *s [mAU] %
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 4 Dilution Factor with ISTDs A, Wavelength=210 nm. pe Width Area Height Àrea [min] mAU *s [mAU] %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VHD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 4 Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Height Area [min] mAU *s [mAU] %

Instrument 1 2/20/2011 3:22:10 PM LTL

Data File D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\062-0301.D Sample Name: THL-6-108B

Acq. Operator	
	t : Instrument 1 Location : Vial 62
Injection Date	: 11/29/2010 4:38:48 PM Inj: 1
	Inj Volume : 5 µl
Acq. Method	: D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\ASH-80-20-05ML-
	210NH-30MIN.M
last changed	: 11/18/2010 3:45:02 PM by DX0
Analysis Metho	d : D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\062-0301.D\DA.M (
	ASH-80-20-05ML-2100M-30MIN.M)
Last changed	: 2/20/2011 3:18:36 PM by LTL
	(modified after loading)
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Signal 1: VWD1	: 4 Dilution Factor with ISTDs
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Feak RetTime T	L Å, Wavelength=210 nm Vpe Width Årea Height Årea [min] mAU *s [mAU] %

*** End of Report ***

Instrument 1 2/20/2011 3:18:40 PM LTL

)ata Pile D:\LC\THL\DATE\THL-6-108BFAC\THL-6-108B-FAC 2010-11-27 15-17-48\072-0101.D Sample Name: THL-6-108B-FAC

Service Minered Street		
log. Operator	: THL	Seq. Line : 1
	: Instrument 1	Location : Vial 72
Injection Date	: 11/27/2010 3:19:21 PM	Inj: 1
		Inj Volume : 5 µl
kcq. Method	: D:\LC\THL\DATE\THL-6-108BR 05ML-210NM-30MIN.M	AC\THL-6-1088-BAC 2010-11-27 15-17-48\ASH-80-20-
art changed	: 11/18/2010 3:45:02 FM by D	00
nalvzis Method	 D. VIC, THINDATE, THI-6-10888 	LAC\THL-6-108B-RAC 2010-11-27 15-17-48\072-0101.
and parts meeting	D\DA.M (ASH-80-20-05ML-210	NM-30HIN.M)
ast changed	: 2/20/2011 3:19:48 PM by LT	
	(modified after loading)	
W/DI A, W	avelength=210 nm (DNLCYHL\DAT EXTHL-6-1088	BRAC/THL-6-108B-RAC 2010-11-27 15-17-48/072-0101.D)
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Sorted By	Area Percent Report : Signal	
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Sorted By Multiplier Dilution	Area Percent Report : Signal : 1.0000 : 1.0000	
Sorted By Multiplier Dilution	Area Percent Report : Signal : 1.0000	
Sorted By Multiplier Dilution	Area Percent Report : Signal : 1.0000 : 1.0000	
Sorted By Multiplier Dilution Use Multiplier	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs	
Sorted By Multiplier Dilution Use Multiplier	Area Percent Report : Signal : 1.0000 : 1.0000	
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=210 nm	
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Heigh	
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 o Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Heigg (min) mAU *s (mAU	nt Area I \$
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VUD1 Peak RetTime Ty # [min] 1 15.107 FB	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs λ, Wavelength=210 nm pe Width Årea Heigt [min] mÅU *s [mÅU [] 1.1433 1.30940e4	nt Area 1 \$ 7696 49.0227
Sorted By Multiplier Dilution Use Multiplier Signal 1: VUD1 Peak RetTime Ty # [min] 1 15.107 FB	Area Percent Report : Signal : 1,0000 : 1,0000 & Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Heigf [min] mAU *s [mAU 	nt Area 1 \$ 7696 49.0227
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=210 nm pe Width Area Height	nt Area <u>1 \$</u>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 1 15.107 FB	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs λ, Wavelength=210 nm pe Width Årea Heigt [min] mÅU *s [mÅU [] 1.1433 1.30940e4	nt Area 1 \$

*** End of Report ***

Instrument 1 2/20/2011 3:19:52 PM LTL

Data File D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-11-29 15-59-47\065-0501.D Sample Name: THL-6-108E

e Name: THL-6-106	E	
	ти. Seq. Line	
cq. Operator :	1111	: Vial 65
cq. Instrument :	THOUT OTHER TAY A	: 1
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	D: \LC\THL\DATE\THL-6-108\THL-6-108 2010-	11-29 15-59-47\ASH-80-20-05ML-
	254NM-30MIN.H	
ast changed :	11/22/2010 4:59:06 PM by THL D:\LC\THL\DATE\THL-6-108\THL-6-108 2010-	11-29 15-59-47\065-0501.D\DA.M (
halysis Method :	D: \L(\IHL\DATE\IHL=D=100(IHL=0=100 to solo	
	ASH-80-20-05ML-254NH-30MIN.M)	
	2/20/2011 3:25:58 PM by LTL	1000 March 1
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and the second sec	Harry Longether 25.4 MM	
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Peak RetTime Typ	[min] mAU *s [mAU] %	
# [min]	[min] min .2 [min]	Ť.
	1 0069 315,87769 4.18676 4.3444	
1 12.073 BB		
2 21.375 MM	1.7738 6955.00781 65.34890 95.6556	
-4	7270.88550 69.53566	
Totals :	7210.00000 69.0000	
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	AND WAR AT THE SEC. THE	
	*** End of Report ***	

Instrument 1 2/20/2011 3:26:03 PM LTL

Nata File D:\LC\THL\DATE\THL-6-108C-E\THL-6-108C-E-RAC 2010-11-28 13-34-30\003-030L.D Sample Name: THL-6-108E-RAC

	An
Acq. Operator	
	: Instrument 1 Location : Vial 3
Injection Date	: 11/28/2010 2:38:48 PM Inj: 1
	Inj Volume : 5 µl
koq. Method	: D:\LC\THL\DATE\THL-6-108C-E\THL-6-108C-E-RAC 2010-11-28 13-34-30\ASH-80-
	20-05ML-254NM-30MIN.M
Last changed	: 11/22/2010 4:59:06 PM by THL
Analysis Method	: D:\LC\THL\DATE\THL-6-108C-E\THL-6-108C-E-RAC 2010-11-28 13-34-30\003-0301.
	D\DA.M (ASH-80-20-05ML-254NM-30MIN.M)
Last changed	: 2/20/2011 3:27:10 PM by LTL
	(modified after loading)
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	Area Percent Report
Covered For	4 Signal
Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000
Use Multiplier	a Dilution Factor with ISTDs
	A, Wavelength=254 nm
Signal 1: VWD1	AF warelenges-67% im
	pe Width Area Height Area
Peak RetTime Ty # [min]	pe Width Area Height Area [min] mAU *s [mAU] %
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Peak RetTime Ty # [min] 1 11.892 VB	pe Width Area Height Area [min] mAU *s [mAU] %
Peak RetTime Ty # [min] 	pe Width Area Height Area [min] mAU *3 [mAU] %
Peak RetTime Ty # [min] 	pe Width Area Height Area [min] mAU *s [mAU] % 0.6554 7713.11719 174.50972 50.2175 1.3726 7646.30957 82.71335 49.7825
Peak RetTime Ty # [min] 	pe Width Area Height Area [min] mAU *s [mAU] %
Peak RetTime Ty # [min] 	pe Width Area Height Area [min] mAU *s [mAU] % 0.6554 7713.11719 174.50972 50.2175 1.3726 7646.30957 82.71335 49.7825
Peak RetTime Ty	pe Width Area Height Area [min] mAU *s [mAU] % 0.6554 7713.11719 174.50972 50.2175 1.3726 7646.30957 82.71335 49.7825 1.53594e4 257.22307
Peak RetTime Ty	pe Width Area Height Area [min] mAU *s [mAU] % 0.6554 7713.11719 174.50972 50.2175 1.3726 7646.30957 82.71335 49.7825 1.53594e4 257.22307
Peak RetTime Ty	pe Width Area Height Area [min] mAU *s [mAU] % 0.6554 7713.11719 174.50972 50.2175 1.3726 7646.30957 82.71335 49.7825 1.53594e4 257.22307

Instrument 1 2/20/2011 3:27:14 PM LTL

Data File D:\LC\THL\D&TE\THL-6-100\THL-6-100 2010-11-22 15-52-59\017-0701.D Sample Name: THL-6-100D Acq. Operator : tmc Acq. Instrument : Instrument 1 Seq. Line : 7 Location : Vial 17 Injection Date : 11/22/2010 5:47:16 PM Inj : 1 Inj Volume : 5 µl : D:\LC\THL\DATE\THL-6-100\THL-6-100 2010-11-22 15-52-59\ASH-80-20-05ML-Acq. Hethod 254NN-30MIN.M Last changed : 11/22/2010 4:59:06 PM by THL Analysis Method : D:\LC\THL\DATE\THL-6-100\THL-6-100 2010-11-22 15-52-59\017-0701.D\DA.M (ASH-80-20-05ML-254NM-30MIN.M) : 2/20/2011 3:09:34 PM by LIL Last changed (modified after loading) WWDTA Wavelength=254 nm(DLLCTHLLDATE/THL-& 100/THL-& 100 2010-11-22 15-52-59017-0701.0) 1.2000 mAU 59.0 HO-60 16 ButO₂C+ 40 ~~CO₂CH₃ 30 (4bl) page 1 20 10 ò -10 24 min 16 18 20 14 Area Percent Report Sorted By Signal ž : 1.0000 Multiplier Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Area Peak RetTime Type Width Height Area [mAU] [min] mAU *s 8. in ana 1 15.885 MF 1.7728 708.80109 6.66365 14.1486 2 18.590 FM 1.5497 4300.87598 46.25590 85.8514 5009.67706 52.91955 Totals :

*** End of Report ***

Instrument 1 2/20/2011 3:09:39 PM LTL

Data File D:\LC\THL\DATE\THL-6-100C-D-RAC\THL-6-100C-D-RAC 2010-11-21 18-03-07\010-0201.D Sample Name: THL-6-00D-rac

Acq. Operator	10777				
A CONTRACTOR OF			Line: 2		
	: Instrument 1		tion : Vial 10		
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ar 22.00 a			lume : 5 µl	an a chur an an an	and the second
Acq. Method		THL-6-100C-D-RAC\THL-	6-100C-D-RAC 20	10-11-21 18-03-07	-HEA/
	80-20-05ML-254M				
	: 11/21/2010 6:13:				
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	0201.D\DA.M (ASH	1-80-20-05ML-254NM.M)		÷	
Last changed	: 2/20/2011 3:07:	LO PM by LTL .			
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1	krea Pero : Signa : 1.000 : 1.000 & Dilution Factor v k, Wavelength=254 r	rent Report al 10 nith ISTDs m Height Årea			
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min]	krea Pero : Signa : 1.000 : 1.001 & Dilution Factor o k, Wavelength=254 n pe Width Area [min] mAU *:	rent Report al 10 nith ISTDs m Height Årea			
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min]	krea Pero : Signo : 1.000 : 1.000 4. Dilution Factor v k, Wavelength=254 r pe Width Area [min] mAU *: 	rent Report 10 10 10 10 10 10 11 11 11 11 11 11 11	[52		
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	krea Pero : Signa : 1.000 : 1.000 4 Dilution Factor of k, Wavelength=254 of pe Width Area [min] mAU *2 0.9920 3269.29 1.2401 3248.93	rent Report 20 20 20 20 20 20 21 21 22 23 24 24 25 20 25 25 25 25 25 25 25 25 25 25	[52		
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	krea Pero : Signa : 1.000 : 1.000 4 Dilution Factor of k, Wavelength=254 of pe Width Area [min] mAU *2 0.9920 3269.29 1.2401 3248.93	rent Report al 10 10 nith ISTDs m Height Area 5 [mAU] %	[52		
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Instrument 1 2/20/2011 3:07:18 PM LTL

)ata File D:\LC\DXQ\DATE\DXQ-9-48\DXQ-9-48AB-ENE 2010-11-30 20-18-33\006-1201.D Sample Name: THL-6-116A

log. Operator				Seq. Line :	12		
	: Instrument 1			Location :	Vial 6		
njection Date	: 12/1/2010 1::	25:13 AM	1	Inj :	1		
			T	nj Volume :	5 µ1		
koq. Method	: D:\LC\DXQ\DA 210NM-25MIN.		9-48\DCCQ-9-4	SAB-ENE 2010	-11-30 20-18	-33\ADH-70-	30-1ML-
Last changed	. 11/20/2010 E	CED (42) 1	PM by THL				
holmois Method	: D:\LC\DXQ\DA	TE\DX0-9	9-48\DX0-9-4	SAB-ENE 2010)-11-30 20-18	-33\006-120	1. D\DA. M
ularysis nechod	(ADH-70-30-1	ML-210M	M-25MIN.M)				
Last changed	: 2/20/2011 3:	43:39 PI	M by LTL			,	
MADI A W	melength=210 nm(D/\LC	OK ONDATE	LDX Q-9-48\LDX(Q-9-	48.48-ENE 2010-11-	30 20-18-33400-140	1.29	
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6 Sorted By Multiplier Dilution Use Multiplier	Àrea : : 4 Dilution Fact	Percent Signal 1.0000 1.0000 tor with	Report				22 m
6 Sorted By Multiplier Dilution Use Multiplier	لند غ Dilution Fact کر Wavelength=	Percent Signal 1.0000 1.0000 tor with 210 nm	Report				22 m
6 Sorted By Multiplier Dilution Use Multiplier	ليت ليت ليت ليت ليت ليت ليت ليت	Percent Signal 1.0000 tor with 210 mm Area	Report	Area			2 1
6 Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime T # [min]	لي ب غ Dilution Fact م, Wavelength= gpe Width (min) mAU	Percent Signal 1.0000 1.0000 tor with 210 nm Area *s	Report n ISTDs Height (mAU)	Area			22 m
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime T # [min] 	Area : : 4 Dilution Fact A, Wavelength= gpe Width [min] mAU	Percent 51gnal 1.0000 1.0000 cor with 210 nm krea *s 9766e4	Report Report 1 ISTDs Height [mAU] 1079,10962	Area 8 1 97.2547			22 m
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime T # [min] 	Area : : 4 Dilution Fact A, Wavelength= ppe Width [min] mAU	Percent 51gnal 1.0000 1.0000 cor with 210 nm krea *s 9766e4	Report Report 1 ISTDs Height [mAU] 1079,10962	Area 8 1 97.2547			22 m
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime T # [min] 1 7.012 V 2 20.710 B	لبند لبن لبن ل ل ل ل ل ل ل ل ل ل ل ل ل	Percent Signal 1.0000 1.0000 tor with 210 nm. Area *s 9766e4 3.88904	Report h ISTDs Height [mAU] 1079,10962 10,88027	Area 8 1 97.2547			22 m
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime T # [min] 	لبند لبن لبن ل ل ل ل ل ل ل ل ل ل ل ل ل	Percent Signal 1.0000 1.0000 tor with 210 nm. Area *s 9766e4 3.88904	Report Report 1 ISTDs Height [mAU] 1079,10962	Area 8 1 97.2547			22 m
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Instrument 1 2/20/2011 3:43:46 PM LTL

)ata File D:\LC\THL\DATE\THL-6-76\THL-6-76 2010-11-12 17-39-05\001-0101.D Sample Name: THL-6-76AA

log. Operator	
	: Instrument 1 Location : Vial 1
Injection Date	: 11/12/2010 5:40:21 PM Inj: 1
22 8 9	Inj Volume : 5 µl
koq. Method	: D:\LC\THL\DATE\THL-6-76\THL-6-76 2010-11-12 17-39-05\ADH-70-30-1ML210NM-
	25MIN. M
last changed	: 11/5/2010 8:32:44 PM by th1
knalysis Method	: D:\LC\THL\DATE\THL-6-76\THL-6-76 2010-11-12 17-39-05\001-0101.D\DA.M (ADH-
	70-30-1ML210MM-25MIN.H)
last changed	: 2/20/2011 3:39:01 PM by LTL
140051 - 101	(modified after loading)
	avelength#210 nm (D)LCTHL\DATEXTHL 6 76/THL 6 78 2010 11 12 17-39 05/001 0101 D)
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1	<pre>Area Percent Report Signal 1.0000 1.0000 Dilution Factor with ISTDs A, Wavelength=210 nm</pre>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1	Area Percent Report : Signal : 1.0000 : 1.0000 4 Dilution Factor with ISTDs Å, Wavelength=210 nm pe Width Årea Height Årea
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VUD1 Peak RetTime Ty # [min]	<pre>Area Percent Report Signal 1.0000 1.0000 2 l.0000 A Dilution Factor with ISTDs Å, Wavelength=210 nm pe Width Area Height Area [min] mAU *s [mAU] %</pre>
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Sorted By Multiplier Dilution Use Multiplier Signal 1: VUD1 Peak RetTime Ty # [min] [Area Percent Report : 1.0000

*** End of Report ***

Instrument 1 2/20/2011 3:39:06 PM LTL

ample Name: thl-116b Acq. Operator : thl Acq. Instrument : Instrument 1 Seq. Line : 3 Location : Vial 2 Inj : 1 Inj Volume : 5 µl Injection Date : 2/21/2011 10:56:27 AM : D:\LC\THL\DATE\THL-6-116\THL-6-116 2011-02-21 10-02-32\ASH-90-10-05ML-Acq. Method 210MM-40MIN.H : 2/21/2011 10:55:14 AM by thl Last changed (modified after loading) Analysis Method : D:\LC\THL\DATE\THL-6-116\THL-6-116 2011-02-21 10-02-32\002-0301.D\DA.M (ASH-90-10-05ML-210MM-40MIN.M) : 2/21/2011 12:04:56 PM by LTL Last changed (nodified after loading) MVD1 A. Wavelergh=210 nm (DXLCITHLNDAT BTHL6-116(THL6-116 2011-02-21 10-02-32002-0301.D) -2⁶⁹⁰ mAU <u>н _{он</u></u>} Ρh 120 BuÔ₂C p-Cl-Ph™ 100 ℃O₂CH₃ 80 4ca' 10,00 60 40 20

ata File D:\LC\THL\DATE\THL-6-116\THL-5-116 2011-02-21 10-02-32\002-0301.D

------Area Percent Report ***************

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Use	Multiplier	6	Dilution	Factor	with	ISTDs

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Signal 1: VWD1 A, Wavelength=210 nm

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Peak	RetTime	Type		Area		Height		Area
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وستعصب								(1,000,000,000,00)
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2	30,080	MH	2.9157	2.393	360e4	136.	82176	92.3606
Totals :			2.59)	158e4	167.	75745		

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Instrument 1 2/21/2011 12:05:02 PM LTL

Page 1 of 1

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