Supporting Information for

Highly efficient construction of spirocyclic chromanone-pyrrolidines *via* Cu(I)/TF-BiphamPhos-catalyzed asymmetric 1,3-dipolar

cycloaddition

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General Remarks.

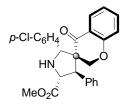
¹H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = single, d = double, t = triple, q = quartet, m = multiple or unresolved, brs = broad single, coupling constant(s) in Hz, integration). ¹³C NMR spectra were recorded on a VARIAN Mercury 75 MHz spectrometer in chloroform-d. 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 excesses were determined by HPLC, using a chiralcel AD-H column, a chiralpak AS-H column with hexane and *i*-PrOH as solvents. Ligands L1 and L2 were prepared according to the literature procedure reported by us.^[1] (E)-3-Alkylidene chroman-4-ones were prepared according to the literature procedure.^[2] The racemic adducts were attained by using AgOAc/ (\pm) -TF-BiphamPhos as the catalyst. The absolute $(2^{\circ}R, 3R, 4^{\circ}R, 5^{\circ}R)$ -**3aa** achieved by AgOAc/(S)-TF-BiphamPhos 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 racemic 1,3-Dipolar Cycloaddition of Azomethine Ylides with (*E*)-3-Alkylidene chroman-4-ones Catalyzed by $AgOAc/(\pm)$ -TF-BiphamPhos Complex.

Under argon atmosphere, (\pm)-TF-BiphamPhos (4.6 mg, 0.0072 mmol) and AgOAc (1.0 mg, 0.006 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.4 mmol), Et₃N (0.03 mmol) and (*E*)-3-alkylidene chroman-4-ones (0.2 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the organic solvent was removed and the residue was purified by column chromatography to give the cycloadduct, which was used as the racemic sample for the chiral HPLC analysis.

General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with (*E*)-3-Alkylidene chroman-4-ones Catalyzed by AgOAc/(*S*)-TF-BiphamPhos Complex.

Under argon atmosphere (*S*)-TF-BiphamPhos **L5** (5.8 mg, 0.0072 mmol) and $Cu(CH_3CN)_4BF_4$ (2.0 mg, 0.006 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.4 mmol), Et₃N (0.03 mmol) and (*E*)-3-alkylidene chroman-4-ones (0.2 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the mixture was filtered through celite and the filtrate was concentrated to dryness. The product purified by column chromatography to give the corresponding cycloaddition product, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess.

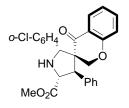


(**3aa**)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 2'-(4-chlorophenyl)-4-oxo-4'-phenylspiro[chroman-3,3'pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. m.p. 60-62 °C; $[\alpha]^{25}_{D} = +91.3$ (*c* 1.08, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.46 (d, *J* = 8.1 Hz, 1H), 7.32-7.26 (m, 6H), 7.10 (d, *J* = 8.7 Hz, 2H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.78 (t, *J* = 7.5 Hz, 1H), 6.66 (d, *J* = 8.7 Hz, 1H), 4.84 (s, 1H), 4.53 (d, *J* = 8.1 Hz, 1H), 4.38 (d, *J* = 8.4 Hz, 1H), 4.16 (d, *J* = 12.0 Hz, 1H), 3.84 (d, *J* = 11.7 Hz, 1H), 3.78 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.59, 173.49, 160.42, 136.97, 136.35, 135.84, 133.67, 129.34, 128.95, 128.74, 127.99, 127.86, 127.38, 121.43, 121.26, 117.17, 73.26, 69.79, 59.66, 53.63, 52.71, 51.93; IR (KBr) v 3677, 3622, 3019, 2977, 2400, 1734, 1606, 1520, 1477, 1415, 1216, 1046, 928, 757, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate

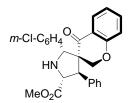
1.0 mL/min, λ = 220 nm); t_r = 21.75 and 38.18 min.



(3ba)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 2'-(2-chlorophenyl)-4-oxo-4'-phenylspiro[chroman-3,3'pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 81% yield. m.p. 99-100 °C; $[\alpha]^{25}_{D} = +33.8$ (*c* 1.24, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.53 (d, J = 7.5 Hz, 1H), 7.37-7.19 (m, 7H), 7.07-6.93 (m, 3H), 6.75 (d, J = 7.5 Hz, 1H), 6.63 (d, J = 8.4 Hz, 1H), 5.51 (s, 1H), 4.52 (d, J = 7.5 Hz, 1H), 4.42 (d, J = 7.5 Hz, 1H), 4.18 (d, J = 12.0 Hz, 1H), 3.87 (d, J = 12.0 Hz, 1H), 3.80 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.83, 173.21, 160.67, 136.55, 135.49, 133.83, 129.05, 128.62, 127.65, 126.81, 126.37, 120.89, 117.06, 72.41, 64.87, 64.50, 59.50, 52.50; IR (KBr) v 3678, 3621, 3020, 2978, 2400, 1733, 1606, 1521, 1478, 1416, 1217, 1046, 928, 757, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 89% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 11.94 and 23.73 min.

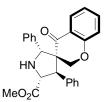


(3ca)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 2'-(3-chlorophenyl)-4-oxo-4'-phenylspiro[chroman-3,3'pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 56-58 °C; $[\alpha]^{25}_{D} = +92.3$ (*c* 0.73, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.48 (dd, *J* = 1.5Hz, 8.1 Hz, 1H), 7.32-7.23 (m, 7H), 7.12-7.07 (m,

2H), 6.96-6.94 (m, 2H), 6.77 (d, J = 7.5 Hz, 1H), 6.68 (d, J = 8.4 Hz, 1H), 4.81 (s, 1H), 4.54 (d, J = 8.1 Hz, 1H), 4.38 (d, J = 8.4 Hz, 1H), 4.16 (d, J = 11.7 Hz, 1H), 3.83 (d, J = 11.7 Hz, 1H), 3.79 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.25, 173.11, 160.15, 140.12, 136.12, 135.65, 133.49, 128.93, 128.73, 128.50, 128.30, 127.81, 127.65, 127.11, 125.71, 121.17, 116.92, 72.96, 69.79, 64.13, 59.37, 52.51, 51.53; IR (KBr) v 3680, 3619, 3018, 2978, 2399, 1734, 1606, 1521, 1478, 1416, 1216, 1047, 928, 757, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 89% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 11.23 and 23.05 min.

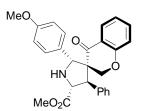


(**3da**)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 4-oxo-2',4'-diphenylspiro[chroman-3,3'-pyrrolidine]-5'carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. m.p. 128-130 °C; $[\alpha]^{25}_{D} = +119.0$ (*c* 1.15, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.44 (d, *J* = 7.5 Hz, 1H), 7.34-7.14 (m, 8H), 7.00-6.99 (m, 3H), 6.74-6.62 (m, 2H), 4.85 (s, 1H), 4.54 (d, *J* = 8.1 Hz, 1H), 4.37 (d, *J* = 8.1 Hz, 1H), 4.18 (d, *J* = 12.0 Hz, 1H), 3.83 (d, *J* = 11.4 Hz, 1H), 3.78 (s, 3H), 2.90 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.74, 173.34, 160.30, 137.89, 136.43, 135.37, 138.71, 128.56, 127.79, 127.71, 127.59, 127.13, 121.17, 120.96, 16.94, 76.32, 73.21, 70.76, 64.34, 59.60, 52.50, 52.26; IR (KBr) v 3682, 3625, 3020, 2980, 2400, 1735, 1610, 1518, 1476, 1420, 1223, 1046, 928, 757, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 16.76 and 49.71 min.

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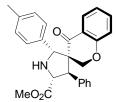


(**3ea**)

(2'R,3R,4'R,5'R)-methyl 2'-(4-methoxyphenyl)-4-oxo-4'-phenylspiro[chroman-

3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 90% yield. m.p. 59-61 °C; $[\alpha]^{25}{}_{D} = +132.9 (c \ 1.04, CHCl_3)$; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.49 (d, J = 7.8 Hz, 1H), 7.36-7.23 (m, 8H), 7.09 (d, J = 8.1 Hz, 1H), 6.77 (d, J = 7.5 Hz, 1H), 6.68 (d, J = 8.1 Hz, 1H), 6.56 (d, J = 8.1 Hz, 1H), 4.85 (s, 1H), 4.55 (d, J = 8.7 Hz, 1H), 4.38 (d, J = 8.4 Hz, 1H), 4.20 (d, J = 11.4 Hz, 1H), 3.89 (d, J = 12.9 Hz, 1H), 3.80 (s, 3H), 3.67 (s, 3H), 2.85 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.44, 172.95, 159.83, 158.55, 135.99, 134.91, 129.51, 128.46, 128.24, 128.12, 126.71, 120.76, 120.54, 116.49, 112.64, 72.74, 69.68, 63.71, 58.97, 54.69, 52.04, 51.73; IR (KBr) v 3682, 3621, 3020, 2976, 2400, 1736, 1688, 1606, 1523, 1478, 1425, 1216, 1047, 929, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 26.92 and 60.81 min.

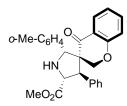


(**3fa**)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 4-oxo-4'-phenyl-2'-*p*-tolylspiro[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 86% yield. m.p. 137-140 °C; $[\alpha]_{D}^{25} = +129.9$ (*c* 1.44, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.44 (d, *J* = 8.1 Hz, 1H), 7.32-7.20 (m, 6H), 7.01 (d, *J* =

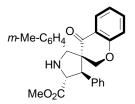
7.8 Hz, 2H), 6.81 (d, J = 7.8 Hz, 2H), 6.73 (d, J = 7.5 Hz, 1H), 6.65 (d, J = 8.1 Hz, 1H), 4.83 (s, 1H), 4.52 (d, J = 8.7 Hz, 1H), 4.37 (d, J = 8.4 Hz, 1H), 4.19 (d, J = 12.0 Hz, 1H), 3.83 (d, J = 12.0 Hz, 1H), 3.77 (s, 3H), 2.13 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.18, 173.32, 160.23, 137.36, 136.33, 135.20, 134.80, 128.59, 128.50, 128.30, 127.52, 127.09, 121.14, 120.83, 116.87, 73.01, 70.28, 64.13, 53.37, 52.40, 52.16, 20.82; IR (KBr) v 3683, 3620, 3020, 2979, 2399, 1735, 1607, 1523, 1479, 1416, 1219, 1047, 929, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 89% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 11.37 and 44.17 min.



(3ga)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 4-oxo-4'-phenyl-2'-*o*-tolylspiro[chroman-3,3'-pyrrolidine]-5'-carboxylate

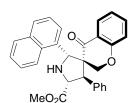
The title compound was prepared according to the general procedure as described above in 95% yield. m.p. 131-134 °C; $[\alpha]^{25}{}_{D} = +64.2$ (*c* 1.28, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.46 (d, *J* = 7.8 Hz, 1H), 7.36-7.26 (m, 4H), 7.20-7.15 (m, 2H), 6.94 (d, *J* = 7.2 Hz, 1H), 6.87 (t, *J* = 7.2 Hz, 1H), 6.79 (t, *J* = 7.5 Hz, 1H), 6.71 (t, *J* = 7.5 Hz, 1H), 6.60 (d, *J* = 8.4 Hz, 1H), 5.16 (s, 1H), 4.59 (d, *J* = 8.4 Hz, 1H), 4.37 (d, *J* = 8.4 Hz, 1H), 4.13 (d, *J* = 12.0 Hz, 1H), 3.81 (d, *J* = 12.0 Hz, 1H), 3.78 (s, 3H), 2.81 (brs, 1H), 2.26 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 173.05, 160.19, 136.49, 136.30, 135.23, 130.08, 128.74, 128.52, 127.62, 127.41, 127.22, 127.09, 125.78, 121.00, 116.63, 73.30, 65.72, 64.32, 59.89, 53.07, 52.52, 19.33; IR (KBr) v 3680, 3619, 3019, 2977, 2400, 1735, 1609, 1524, 1478, 1415, 1216, 1046, 928, 757, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 89% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 10.51 and 12.82 min.



(3ha)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 4-oxo-4'-phenyl-2'-*m*-tolylspiro[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 94% yield. m.p. 141-144 °C; $[\alpha]^{25}{}_{D} = +103.7$ (*c* 1.46, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.43 (dd, J = 1.8 Hz, 8.1 Hz, 1H), 7.33-7.19 (m, 6H), 6.99-6.88 (m, 3H), 6.78 (d, J = 7.5 Hz, 1H), 6.72 (t, J = 7.5 Hz, 1H), 6.64 (d, J = 8.7 Hz, 1H), 4.81 (s, 1H), 4.53 (d, J = 8.7 Hz, 1H), 4.37 (d, J = 8.1 Hz, 1H), 4.17 (d, J = 12.0 Hz, 1H), 3.81 (d, J = 12.0 Hz, 1H), 3.77 (s, 3H), 2.89 (brs, 1H), 2.08 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.56, 173.16, 160.24, 137.60, 137.14, 136.36, 135.18, 128.61, 128.54, 128.39, 127.60, 127.50, 127.04, 124.69, 121.25, 120.80, 116.75, 73.07, 70.66, 64.19, 59.57, 52.38, 52.11, 21.00; IR (KBr) v 3679, 3619, 3021, 2978, 2401, 1735, 1606, 1520, 1477, 1415, 1217, 1046, 928, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 89% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r= 9.83 and 21.72 min.



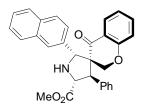
(**3ia**)

(2'R,3R,4'R,5'R)-methyl 2'-(naphthalen-1-yl)-4-oxo-4'-phenylspiro[chroman-

3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 93% yield. m.p. 178-181 °C; $[\alpha]^{25}_{D} = -68.4$ (*c* 1.82, CHCl₃); ¹H NMR

(CDCl₃, TMS, 300 MHz) δ 8.03 (d, J = 8.7 Hz, 1H), 7.56-7.43 (m, 3H), 7.36-7.15 (m, 8H), 7.07 (d, J = 8.1 Hz, 1H), 6.81 (d, J = 7.5 Hz, 1H), 6.37 (t, J = 7.5 Hz, 1H), 6.19 (d, J = 8.1 Hz, 1H), 5.76 (s, 1H), 4.65 (d, J = 9.3 Hz, 1H), 4.41 (d, J = 8.7 Hz, 1H), 4.29 (d, J = 11.7 Hz, 1H), 3.73 (d, J = 12.0 Hz, 1H), 3.66 (s, 3H), 2.84 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.11, 173.16, 160.02, 135.87, 134.67, 134.56, 133.14, 130.94, 128.59, 128.17, 127.52, 126.55, 125.80, 125.26, 124.95, 124.81, 122.75, 120.84, 120.61, 116.41, 73.82, 63.61, 63.46, 60.21, 52.38, 51.76; IR (KBr) v 3683, 3621, 3019, 2980, 2400, 1736, 1686, 1606, 1517, 1478, 1425, 1215, 1040, 928, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 20.91 and 40.67 min.



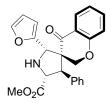
(3ja)

(2'R,3R,4'R,5'R)-methyl 2'-(naphthalen-2-yl)-4-oxo-4'-phenylspiro[chroman-

3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 83% yield. m.p. 107-110 °C; $[\alpha]^{25}_{D} = +73.4$ (*c* 1.21, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.64-7.61 (m, 3H), 7.53 (d, *J* = 15.4 Hz, 1H), 7.39-7.29 (m, 9H), 7.10 (t, *J* = 8.1 Hz, 1H), 6.63 (d, *J* = 8.1 Hz, 1H), 6.57 (d, *J* = 7.5 Hz, 1H), 5.06 (s, 1H), 4.68 (d, *J* = 8.7 Hz, 1H), 4.66 (d, *J* = 8.7 Hz, 1H), 4.29 (d, *J* = 12.0 Hz, 1H), 3.90 (d, *J* = 12.0 Hz, 1H), 3.84 (s, 3H), 2.95 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 188.80, 168.84, 155.69, 131.81, 128.34, 128.10, 124.25, 124.13, 123.34, 123.13, 122.93, 122.75, 122.54, 121.28, 120.95, 116.66, 116.42, 112.31, 68.57, 66.01, 59.74, 55.27, 48.07, 47.51; IR (KBr) v 3681, 3620, 3020, 2979, 2400, 1737, 1680, 1616, 1514, 1478, 1425, 1216, 1046, 928, 759, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 86% ee (Chiralcel AD-H,

i-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 21.06 and 57.83 min.

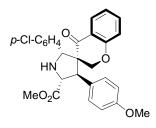


(3ka)

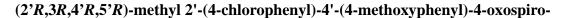
(2'R,3R,4'R,5'R)-methyl 2'-(furan-2-yl)-4-oxo-4'-phenylspiro[chroman-3,3'-

pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 86% yield. m.p. 69-72 °C; $[\alpha]^{25}{}_{D} = +129.0 (c \ 1.15, CHCl_3)$; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.67 (dd, J = 1.5 Hz, 7.8 Hz, 1H), 7.35-7.27 (m, 6H), 7.04 (d, J = 1.6 Hz, 1H), 6.86 (d, J = 6.6 Hz, 1H), 6.76 (d, J = 8.4 Hz, 1H), 6.11 (d, J = 3.0 Hz, 1H), 6.02-6.01 (m, 1H), 4.89 (s, 1H), 4.52 (d, J = 7.5 Hz, 1H), 4.32 (d, J = 8.1 Hz, 1H), 4.20 (d, J = 12.0 Hz, 1H), 3.85 (d, J = 12.0 Hz, 1H), 3.76 (s, 3H), 3.06 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.00, 173.15, 160.57, 150.72, 141.90, 136.41, 135.51, 128.71, 128.58, 127.62, 127.30, 121.13, 120.59, 117.14, 110.04, 108.59, 72.33, 64.39, 63.42, 58.83, 52.55, 51.78; IR (KBr) v 3680, 3621, 3019, 2981, 2400, 1737, 1687, 1609, 1513, 1478, 1425, 1214, 1040, 928, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 13.61 and 57.64 min.

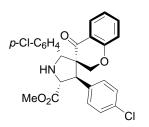


(**3ab**)



[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. $[\alpha]^{25}{}_{\rm D}$ = +58.4 (*c* 1.40, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.45 (d, *J* = 7.5 Hz, 1H), 7.30-7.22 (m, 3H), 7.09 (d, *J* = 8.7 Hz, 2H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.86 (d, *J* = 8.1 Hz, 2H), 6.78 (t, *J* = 7.2 Hz, 1H), 6.67 (d, *J* = 8.7 Hz, 1H), 4.82 (s, 1H), 4.49 (d, *J* = 7.2 Hz, 1H), 4.32 (d, *J* = 6.6 Hz, 1H), 4.19 (d, *J* = 12.0 Hz, 1H), 3.85 (d, *J* = 13.2 Hz, 1H), 3.78 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.41, 173.29, 160.19, 158.89, 136.82, 135.60, 133.40, 130.86, 129.52, 129.06, 127.77, 127.12, 121.17, 121.04, 116.96, 114.01, 72.94, 69.37, 64.11, 59.46, 55.15, 52.50, 50.99; IR (KBr) v 3680, 3621, 3019, 2979, 2400, 1735, 1685, 1606, 1517, 1478, 1426, 1215, 1044, 929, 758, 668 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 22.85 and 39.31 min.

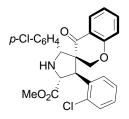


(**3ac**)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 2',4'-bis(4-chlorophenyl)-4-oxospiro[chroman-3,3'pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 95% yield. m.p. 60-62 °C; $[\alpha]^{25}_{D} = +103.2$ (*c* 1.10, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.46 (d, *J* = 7.8 Hz, 1H), 7.28-7.22 (m, 5H), 7.09 (d, *J* = 8.1 Hz, 2H), 6.98 (d, *J* = 8.1 Hz, 2H), 6.86 (d, *J* = 8.1 Hz, 2H), 6.78 (m, 1H), 6.68 (d, *J* = 8.1 Hz, 1H), 4.82 (s, 1H), 4.49 (d, *J* = 8.4 Hz, 1H), 4.35 (m, 1H), 4.19 (d, *J* = 12.0 Hz, 1H), 3.85 (d, *J* = 11.7 Hz, 1H), 3.78 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 191.93, 172.92, 160.20, 136.89, 135.76, 134.45, 133.57, 129.89, 129.12, 128.88, 127.85, 127.24, 121.35, 121.04, 117.00, 72.80, 69.12, 63.63, 52.10, 50.68, 29.84; IR (KBr) v 3683, 3621, 302, 2980, 2400, 1736, 1606, 1517, 1478, 1425, 1215, 1040, 928,

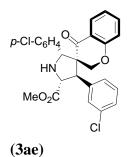
758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 41.15 and 48.18 min.



(3ad)

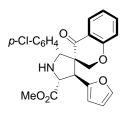
(2'*R*,3*R*,4'*R*,5'*R*)-methyl 4'-(2-chlorophenyl)-2'-(4-chlorophenyl)-4-oxospiro-[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 146-148 °C; $[\alpha]^{25}_{D} = +85.7$ (*c* 1.68, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.62 (t, *J* = 11.1 Hz, 2H), 7.47 (t, *J* = 7.2 Hz, 2H), 7.29 (d, *J* = 7.2 Hz, 1H), 7.27-7.17 (m, 3H), 6.93 (t, *J* = 8.4 Hz, 1H), 6.78 (t, *J* = 7.5 Hz, 1H), 6.45 (d, *J* = 8.1 Hz, 1H), 4.93 (d, *J* = 4.5 Hz, 1H), 4.75 (d, *J* = 6.9 Hz, 1H), 4.23 (s, 1H), 4.12 (d, *J* = 11.7 Hz, 1H), 3.93 (s, 3H), 3.86 (d, *J* = 11.7 Hz, 1H), 3.08 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 193.69, 173.79, 160.35, 136.68, 135.74, 135.35, 134.81, 133.79, 130.24, 129.49, 129.10, 127.89, 127.25, 121.35, 120.84, 117.12, 73.00, 66.90, 58.39, 52.97, 49.70; IR (KBr) v 3679, 3620, 3018, 2983, 2403, 1736, 1683, 1606, 1513 1473, 1425, 1215, 1040, 929, 758, 668 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 11.47 and 43.77 min.



(2'*R*,3*R*,4'*R*,5'*R*)-methyl 4'-(3-chlorophenyl)-2'-(4-chlorophenyl)-4-oxospiro-[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. m.p. 66-68 °C; $[\alpha]^{25}_{D} = +110.3$ (*c* 0.76, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.38 (d, *J* = 7.5 Hz, 1H), 7.24-7.02 (m, 5H), 7.01 (d, *J* = 8.1 Hz, 2H), 6.90 (d, *J* = 8.1 Hz, 2H), 6.72 (d, *J* = 7.5 Hz, 1H), 6.61 (t, *J* = 7.8 Hz, 1H), 4.76 (s, 1H), 4.50 (d, *J* = 8.1 Hz, 1H), 4.26 (d, *J* = 8.4 Hz, 1H), 4.13 (d, *J* = 12.0 Hz, 1H), 3.77 (d, *J* = 12.0 Hz, 1H), 3.75 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.18, 173.06, 160.37, 138.39, 136.81, 135.98, 134.84, 133.77, 130.19, 129.34, 128.83, 128.03, 127.43, 126.96, 121.55, 121.15, 117.20, 73.00, 69.60, 63.98, 59.61, 52.81, 51.23; IR (KBr) v 3680, 3620, 3020, 2981, 2400, 1735, 1685, 1605, 1515, 1477, 1425, 1215, 1039, 929, 759, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 20.98 and 46.34 min.

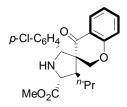


(3af)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 2'-(4-chlorophenyl)-4'-(furan-2-yl)-4-oxospiro[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 89% yield. m.p. 146-148 °C; $[\alpha]^{25}{}_{D} = +115.3$ (*c* 0.66, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.49 (d, *J* = 7.5 Hz, 1H), 7.40 (s, 1H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.06 (d, *J* = 8.1 Hz, 2H), 6.96 (d, *J* = 8.4 Hz, 2H), 6.80 (t, *J* = 7.8 Hz, 1H), 6.69 (d, *J* = 8.7 Hz, 1H), 6.32 (s, 1H), 6.25 (s, 1H), 4.84 (s, 1H), 4.57 (d, *J* = 8.4 Hz, 1H), 4.40-4.35 (m, 2H), 3.98 (d, *J* = 12.0 Hz, 1H), 3.80 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.03, 172.81, 160.54, 150.31, 142.69, 136.64, 135.90, 133.64, 129.22, 127.93, 127.33, 121.47, 121.11, 117.25, 110.68, 109.20, 72.37, 69.55, 62.74, 60.29,

52.71, 45.51; IR (KBr) v 3680, 3620, 3019, 2980, 2399, 1736, 1606, 1519, 1478, 1425, 1215, 1040, 928, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 17.72 and 33.19 min.

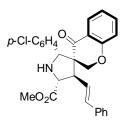


(3ag)

(2'R,3R,4'R,5'R)-methyl 2'-(4-chlorophenyl)-4-oxo-4'-propylspiro[chroman-

3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 75% yield. $[\alpha]^{25}_{D} = +86.7$ (*c* 0.72, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.46 (d, *J* = 8.4 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 6.98 (s, 4H), 6.87-6.83 (m, 2H), 4.74 (s, 1H), 4.48 (d, *J* = 11.1 Hz, 2H), 3.86 (s, 3H), 3.71 (d, *J* = 9.0 Hz, 1H), 3.29 (q, *J* = 8.4 Hz, 1H), 2.24 (brs, 1H), 1.55-1.49 (m, 2H), 1.34-1.21 (m, 1H), 0.89 (t, *J* = 6.6 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 191.90, 173.97, 160.29, 137.93, 135.66, 133.27, 128.99, 127.75, 127.42, 121.41, 121.20, 117.17, 70.93, 67.53, 64.81, 59.15, 52.37, 45.47, 29.79, 21.28, 14.17; IR (KBr) v 3682, 3621, 3020, 2976, 2400, 1736, 1688, 1606, 1523, 1478, 1425, 1216, 1047, 929, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.35 and 11.10 min.

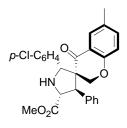




(2'R,3R,4'R,5'R)-methyl 2'-(4-chlorophenyl)-4-oxo-4'-styrylspiro[chroman-

3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. m.p. 133-134 °C; $[\alpha]^{25}{}_{D} = +159.3$ (*c* 1.28, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.45 (dd, J = 1.5 Hz, 7.8 Hz, 1H), 7.36-7.20 (m, 6H), 7.05-6.97 (m, 4H), 6.81-6.78 (m, 2H), 6.62 (d, J = 15.9 Hz, 1H), 6.05 (dd, J = 8.7 Hz, 15.3 Hz, 1H), 4.80 (s, 1H), 4.57 (d, J = 11.4 Hz, 1H), 4.43 (d, J = 12.0 Hz, 1H), 4.10 (t, J = 9.0 Hz, 1H), 3.99 (d, J = 9.3 Hz, 1H), 3.81 (s, 3H), 2.54 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 191.47, 173.04, 160.29, 137.29, 136.37, 135.70, 134.75, 133.46, 128.95, 128.53, 127.86, 127.26, 126.37, 123.23, 121.39, 121.21, 117.11, 71.71, 68.26, 64.20, 60.66, 52.41, 49.64; IR (KBr) v 3681, 3618, 3019, 2979, 2400, 1736, 1686, 1606, 1517, 1478, 1425, 1215, 1040, 928, 758, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); t_r = 17.88 and 34.69 min.

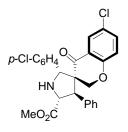


(**3ai**)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 2'-(4-chlorophenyl)-6-methyl-4-oxo-4'-phenylspiro-[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 95% yield. m.p. 206-208 °C; $[\alpha]^{25}{}_{D} = +121.4$ (*c* 1.01, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.31-7.26 (m, 6H), 7.10 (d, *J* = 8.1 Hz, 3H), 6.99 (d, *J* = 8.7 Hz, 2H), 6.57 (d, *J* = 8.1 Hz, 1H), 4.84 (s, 1H), 4.52 (d, *J* = 8.7 Hz, 1H), 4.39 (d, *J* = 8.7 Hz, 1H), 4.15 (d, *J* = 12.0 Hz, 1H), 3.80 (d, *J* = 12.0 Hz, 1H), 3.78 (s, 3H), 2.16 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.57, 173.27, 158.35, 136.74, 136.08, 133.43, 130.71, 129.15, 128.70, 128.50, 127.80, 127.61, 126.70, 120.68, 116.77, 72.99, 69.40, 63.97, 59.59, 52.53, 51.70, 20.19; IR (KBr) v 3681, 3621, 3021, 2977,

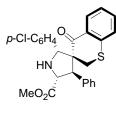
2400, 1735, 1521, 1476, 1424, 1215, 1047, 929, 773, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 15.56 and 21.28 min.



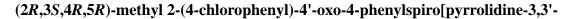
(3aj)

(2'*R*,3*R*,4'*R*,5'*R*)-methyl 6-chloro-2'-(4-chlorophenyl)-4-oxo-4'-phenylspiro-[chroman-3,3'-pyrrolidine]-5'-carboxylate

The title compound was prepared according to the general procedure as described above in 87% yield. $[\alpha]^{25}{}_{\rm D}$ = +105.6 (*c* 1.12, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.45 (d, *J* = 2.4 Hz, 1H), 7.33-7.20 (m, 7H), 7.11-7.05 (m, 4H), 6.63 (d, *J* = 9.0 Hz, 1H), 4.87 (s, 1H), 4.52-4.45 (m, 2H), 4.20 (d, *J* = 12.0 Hz, 1H), 3.82 (d, *J* = 14.4 Hz, 1H), 3.79 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 191.45, 172.77, 158.74, 135.65, 134.01, 129.12, 128.86, 128.48, 128.06, 127.89, 126.86, 126.36, 118.80, 73.06, 69.50, 64.03, 59.20, 52.76, 51.76; IR (KBr) v 3682, 3620, 3020, 2978, 2429, 2400, 1736, 1523, 1479, 1428, 1219, 1043, 929, 773, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 14.90 and 17.95 min.



(3ak)



thiochroman]-5-carboxylate

The title compound was prepared according to the general procedure as described above in 93% yield. $[\alpha]^{25}{}_{D} = +124.3$ (*c* 1.06, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.58 (d, *J* = 8.1 Hz, 1H), 7.36-7.16 (m, 9H), 7.01-6.93 (m, 3H), 5.17 (s, 1H), 4.78 (d, *J* = 7.2 Hz, 1H), 4.40 (d, *J* = 12.0 Hz, 1H), 3.71 (s, 3H), 3.04 (d, *J* = 13.8 Hz, 1H), 2.85 (d, *J* = 14.7 Hz, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 192.76, 173.19, 139.76, 138.06, 135.37, 133.42, 132.90, 130.62, 130.40, 129.39, 129.06, 128.45, 127.78, 127.53, 126.30, 124.67, 66.53, 62.12, 60.15, 53.48, 52.51, 34.28; IR (KBr) v 3683, 3621, 3019, 2980, 2400, 1736, 1686, 1606, 1517, 1478, 1425, 1215, 1040, 928, 758, 669 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, $\lambda = 220$ nm); t_r = 12.40 and 22.69 min.

X-ray Crystal Structures of (2'R,3R,4'R,5'R)-3aa

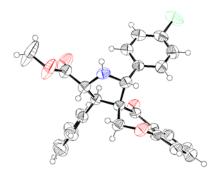
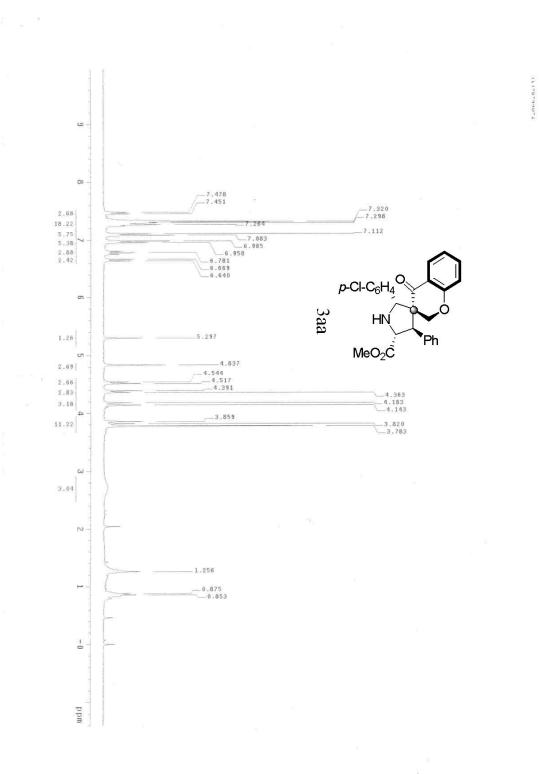


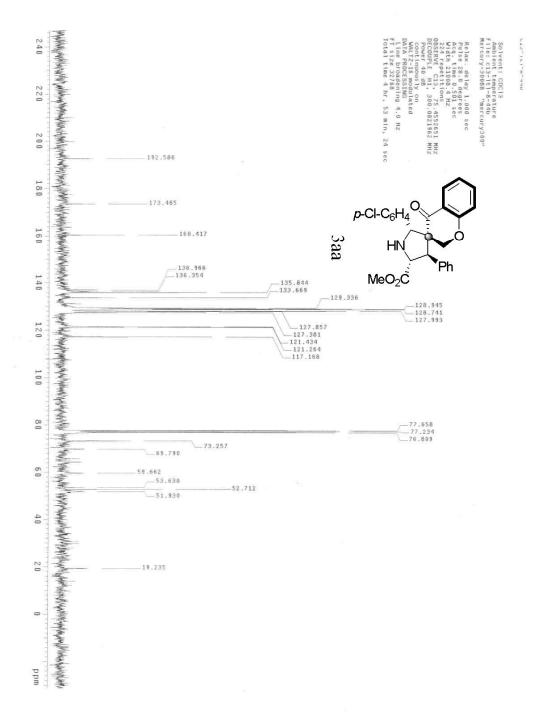
Figure 2. X-ray structure of (2'R,3R,4'R,5'R)-3aa.

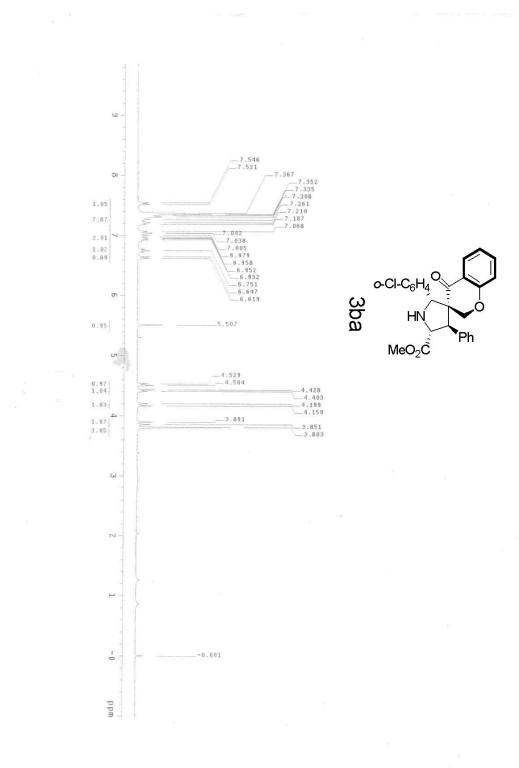
For (2'R, 3R, 4'R, 5'R)-**3aa**: C₂₆H₂₂ClNO₄, $M_r = 447.90$, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 9.960(9), b = 12.124(11), c = 18.738(16) Å, V = 2263(3) Å³, Z = 4, 4436 reflections measured, 2920 unique ($R_{int} = 0.0503$) which were used in all caclculations. The final $wR_2 = 0.1037$ (all data), Flack $\chi = -0.10(10)$. CCDC 828929 (**3aa**).

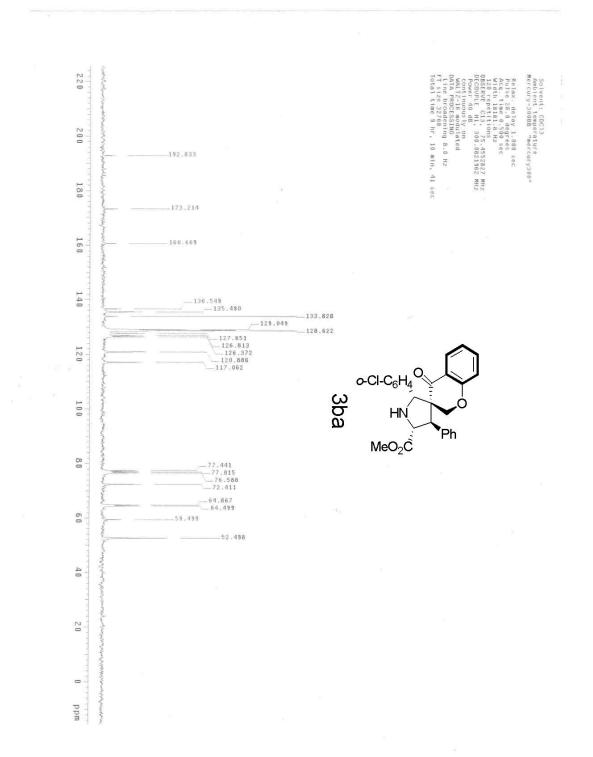
References

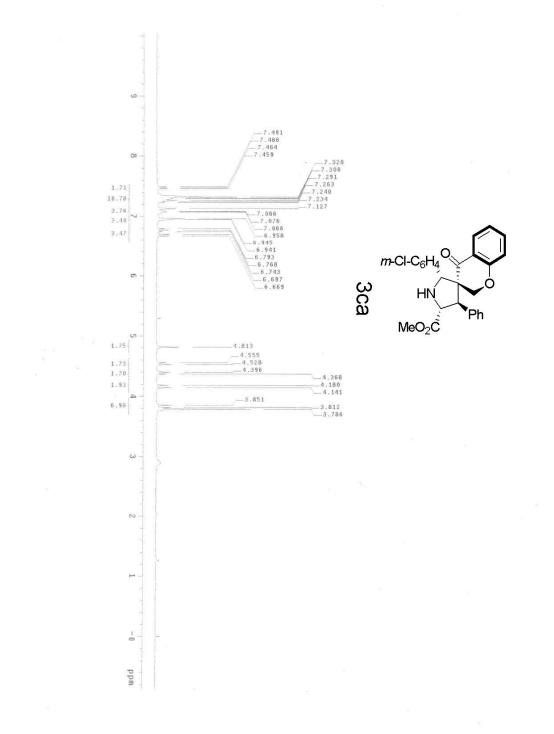
- [1] C.-J. Wang, G. Liang, Z-Y. Xue, F. Gao, J. Am. Chem. Soc. 2008, 130, 17250.
- [2] (a) K. M.Dawood, T. Fuchigami, J. Org. Chem., 2001, 66, 7691. (b) D. Basavaiah,
- M. Bakthadoss, S. Pandiaraju, chem.commun., 1998, 1639. (c) S. H. Kim, S. H. Kim,
- J. N. Kim, Bull. Korean Chem. Soc. 2008, 29, 2039.

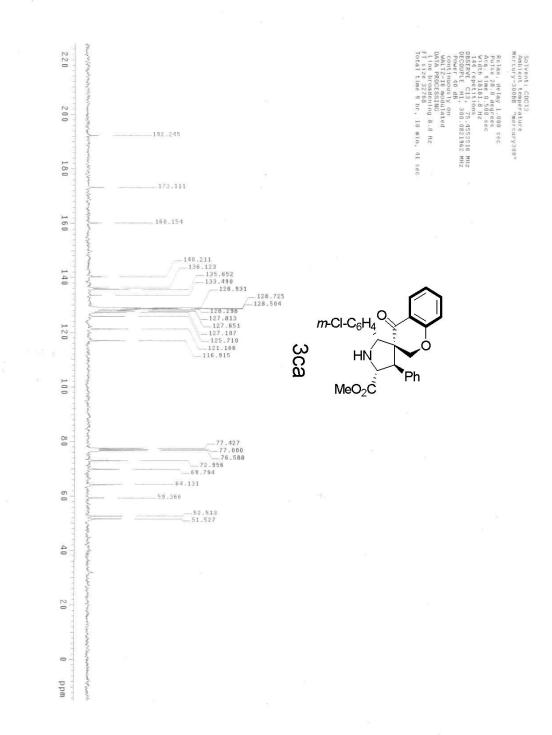


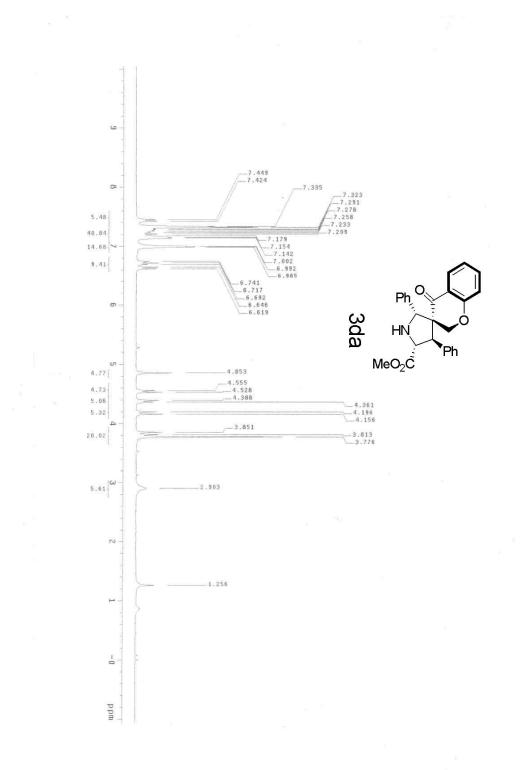


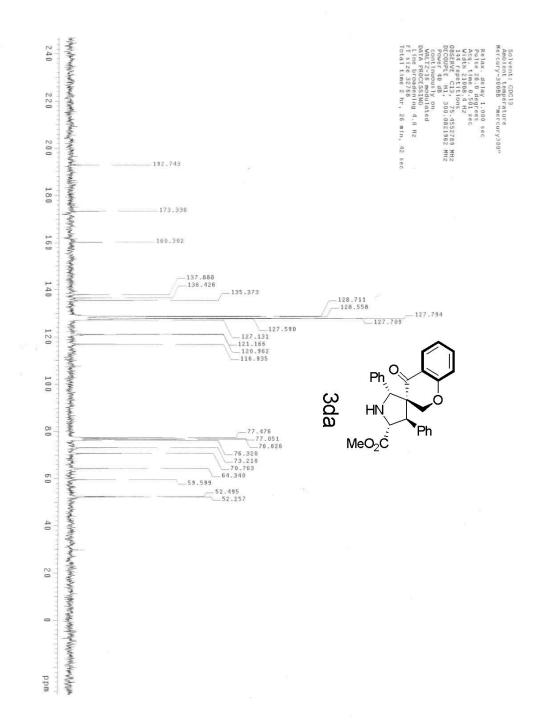


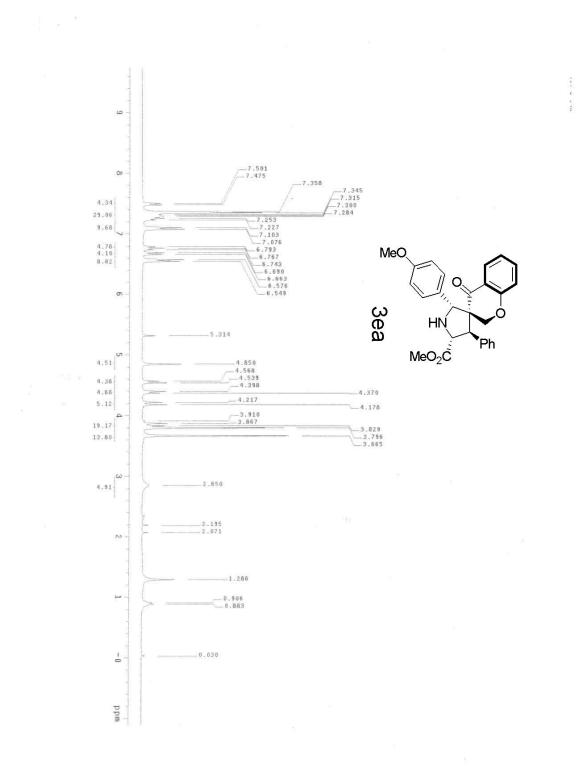


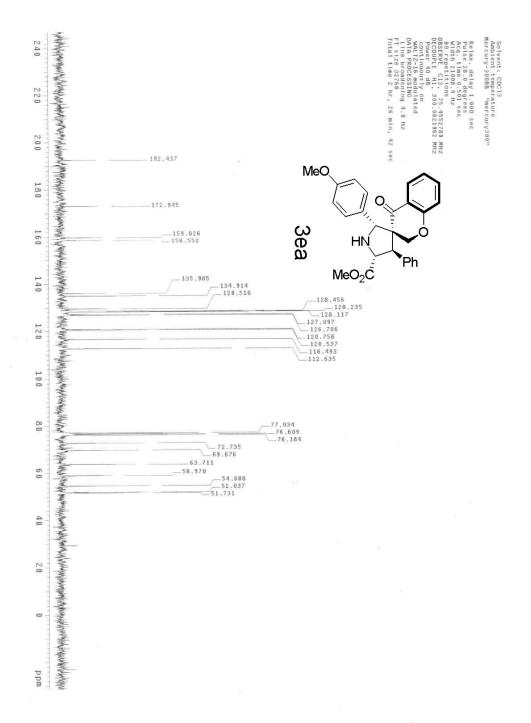


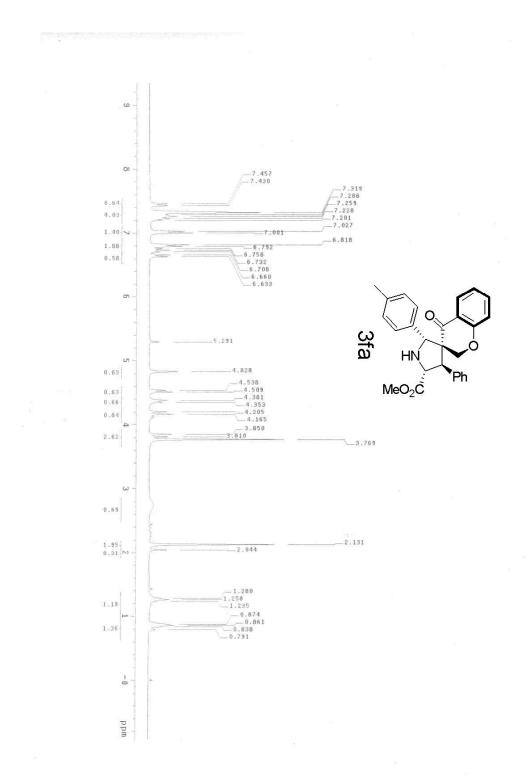


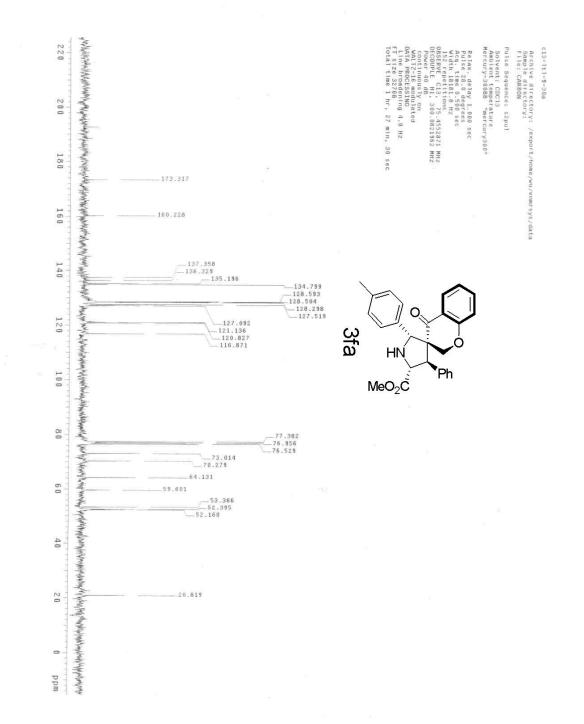


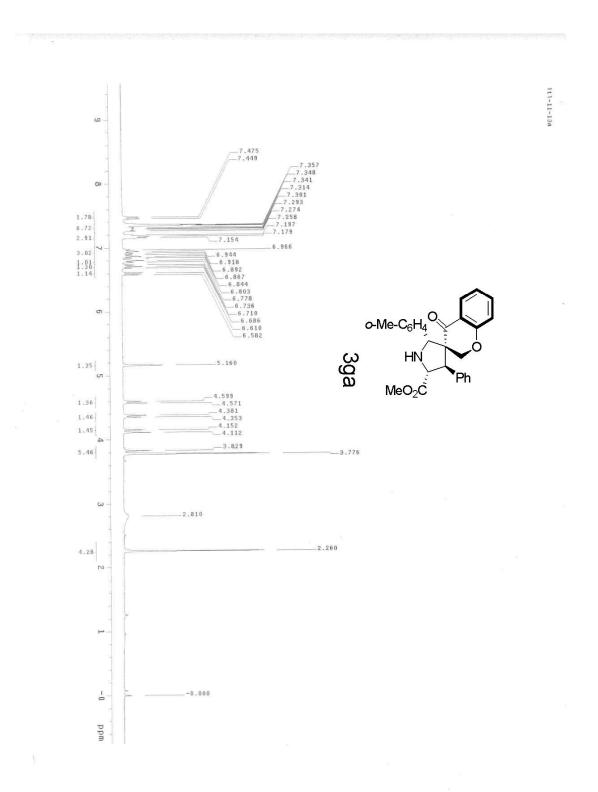


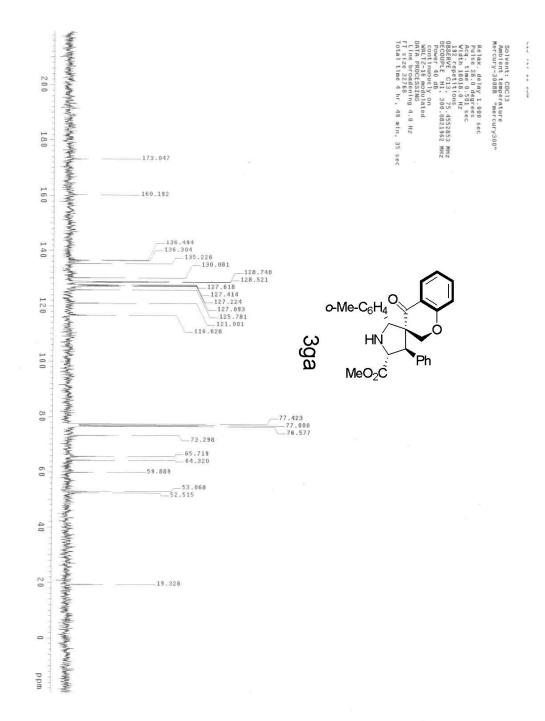


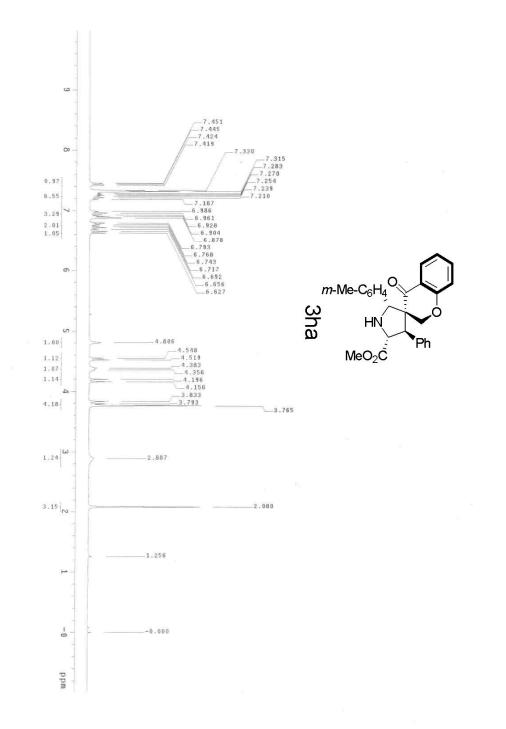




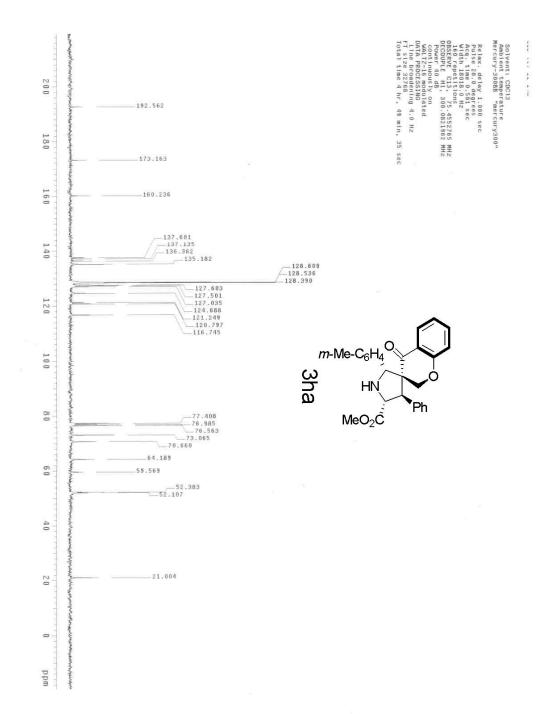


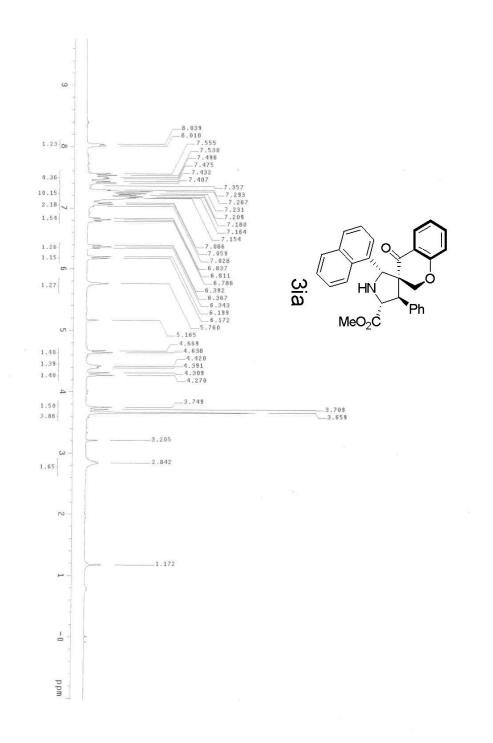




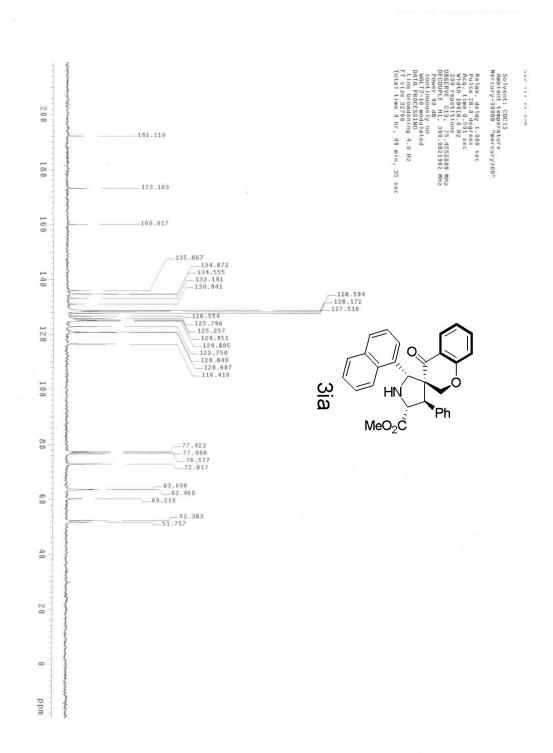


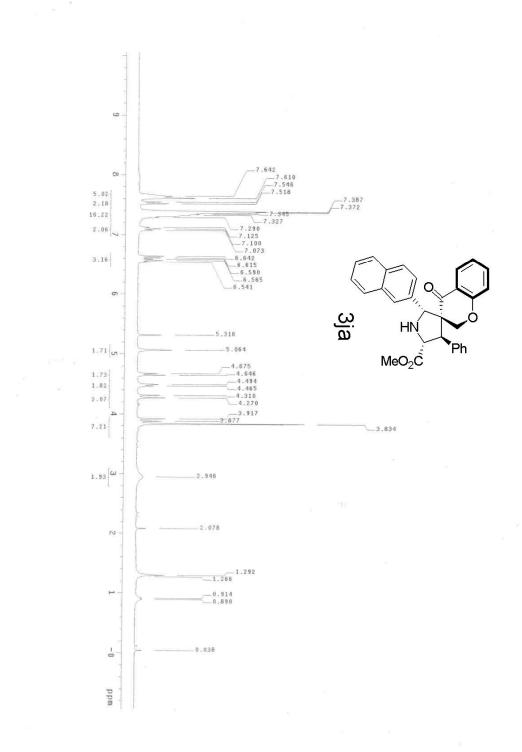
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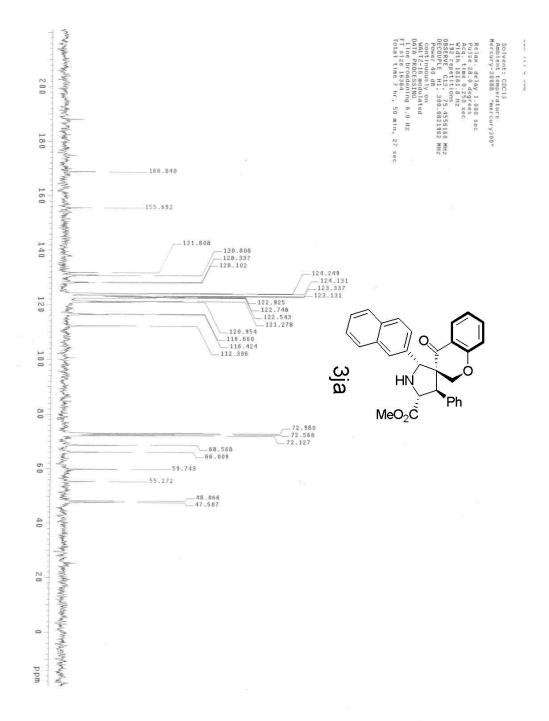


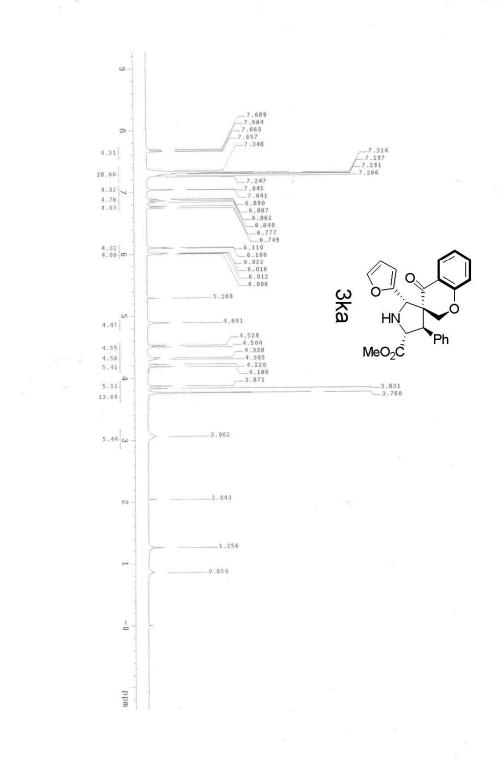


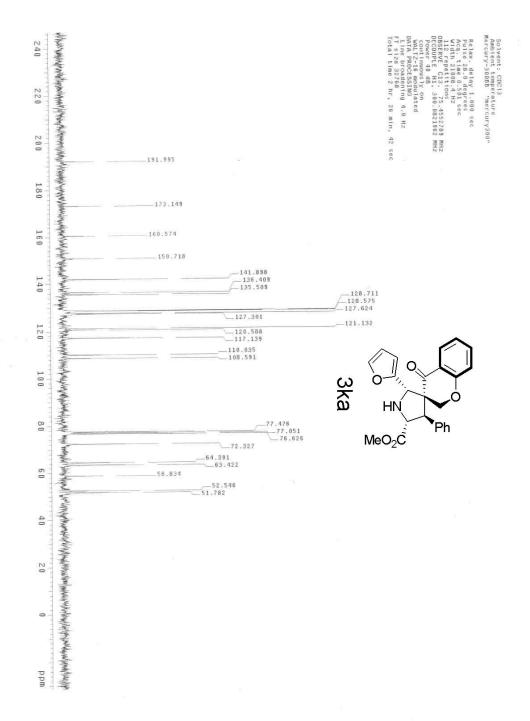
101 22 200



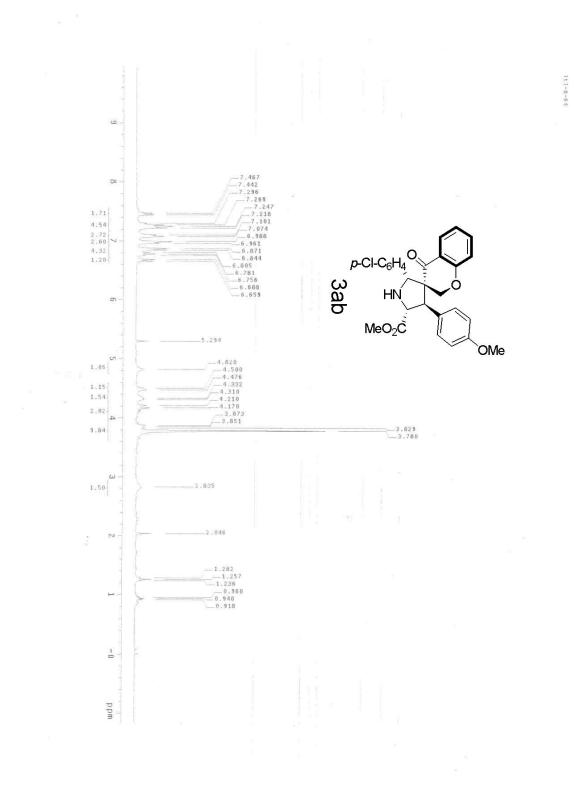


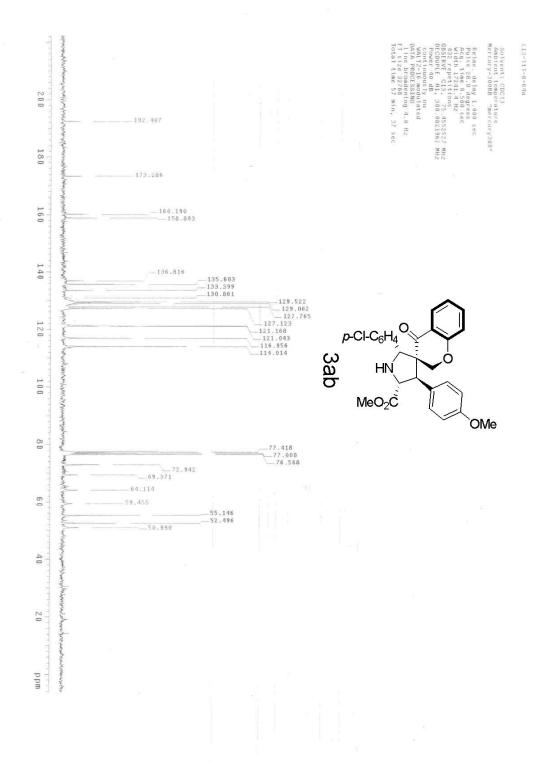


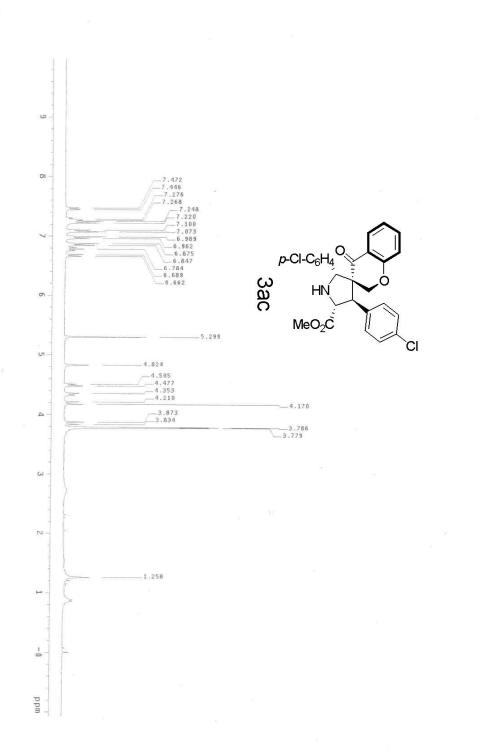


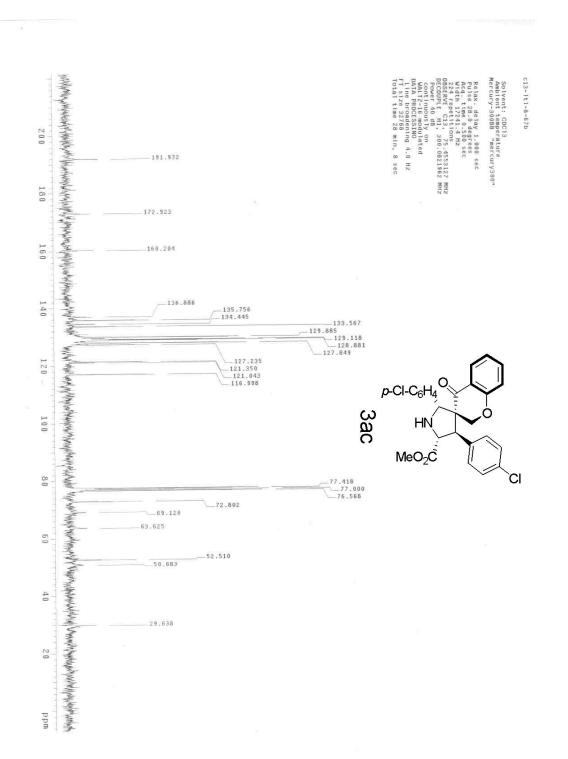


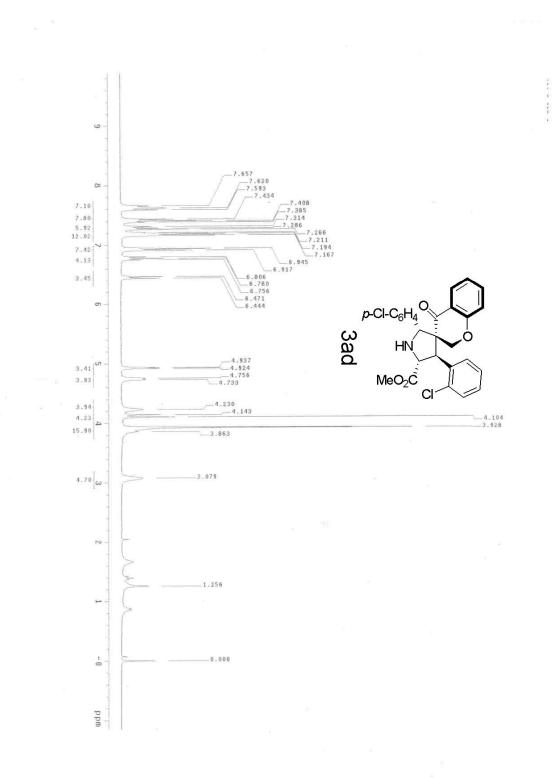
Electronic Supplementary Material (ESI) for Chemical Communications This journal is The Royal Society of Chemistry 2011

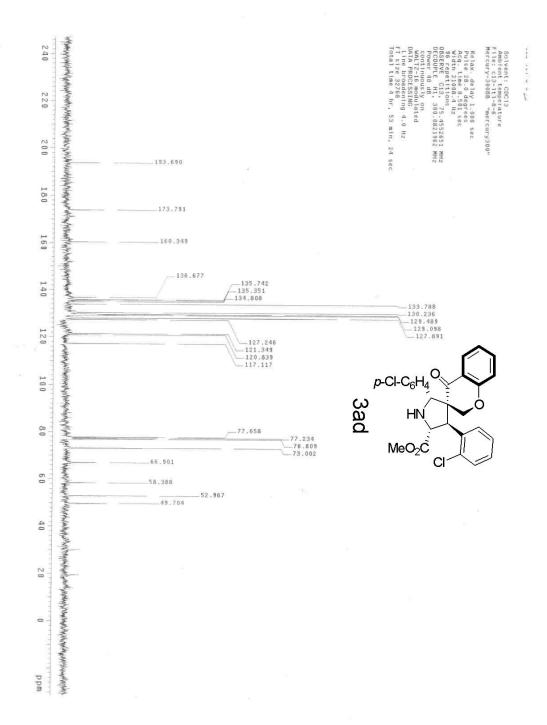


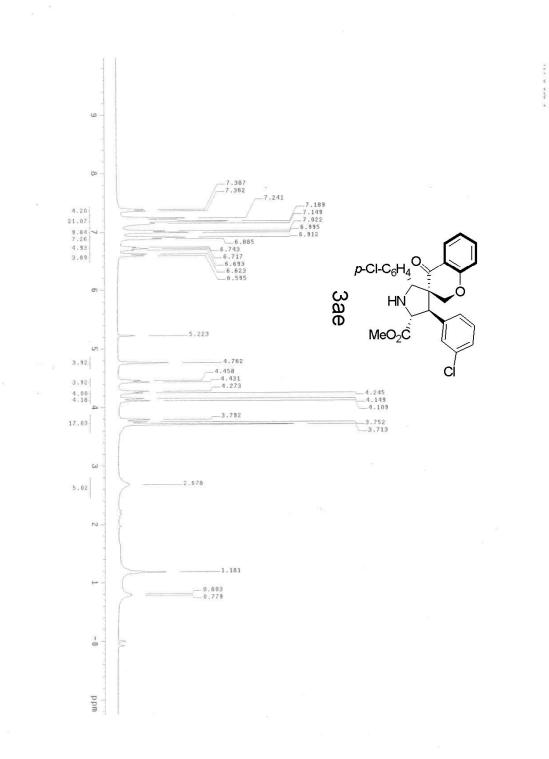


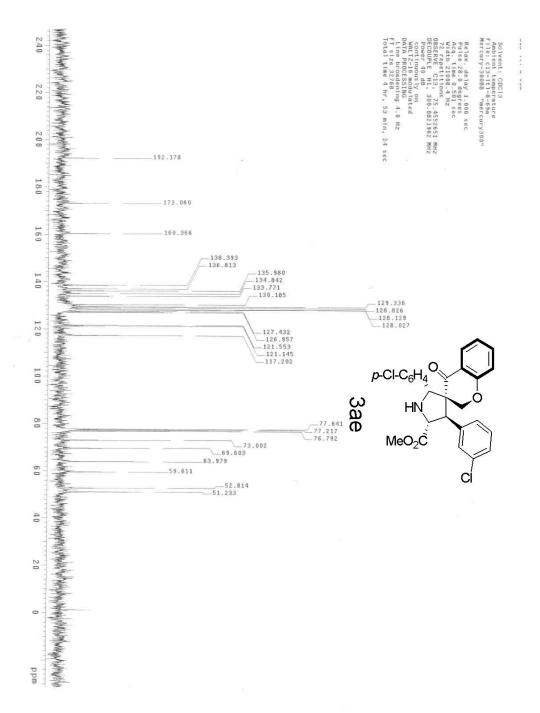


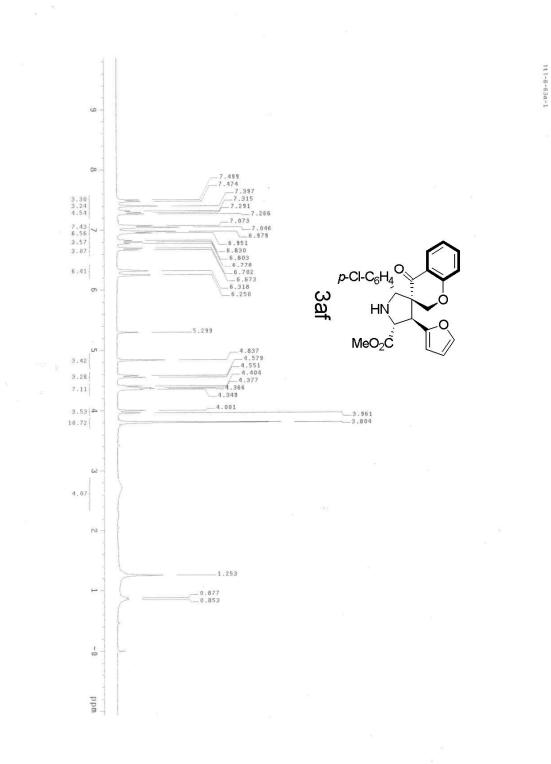


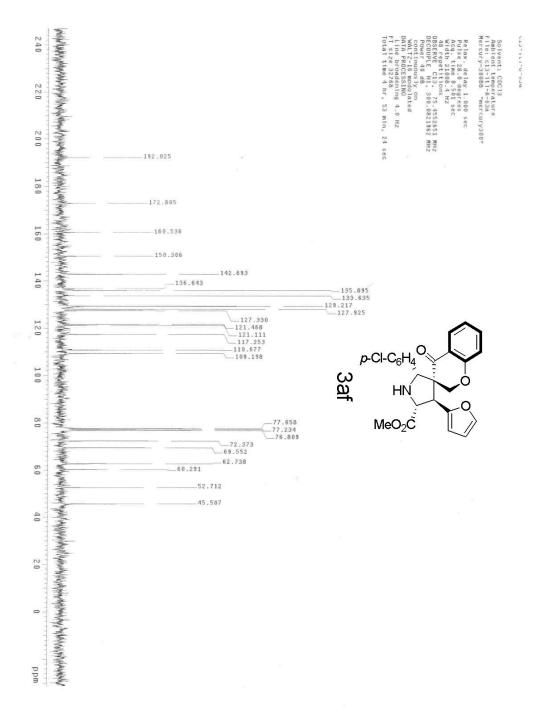


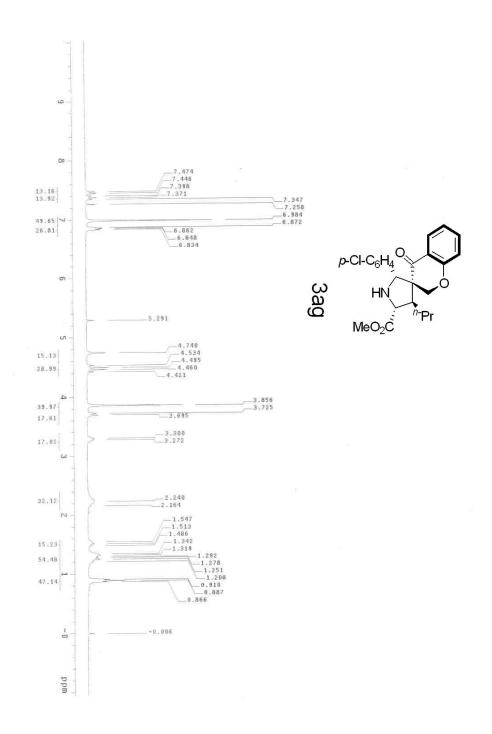




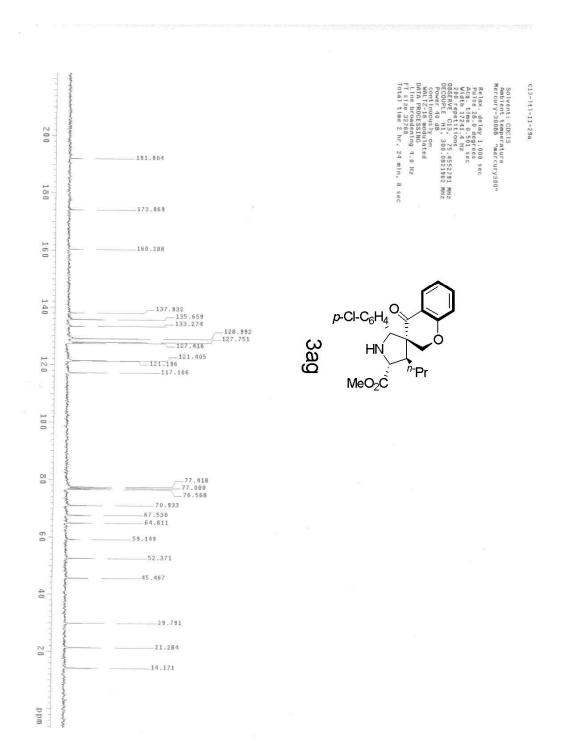


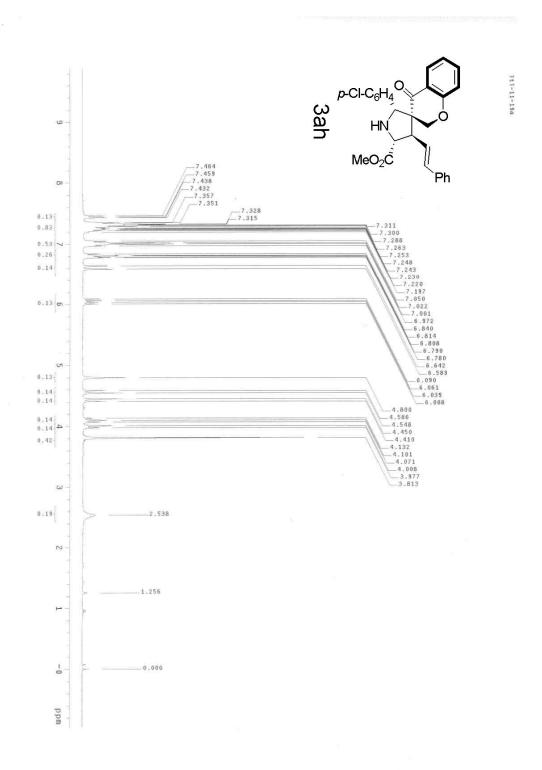


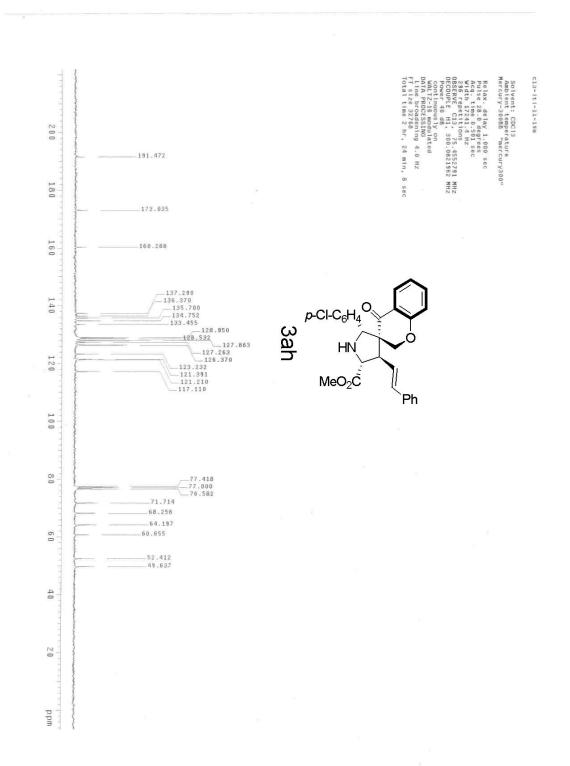


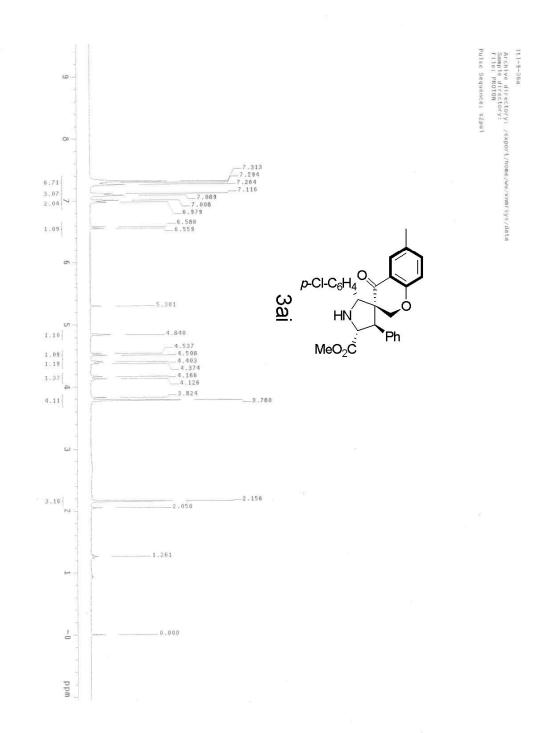


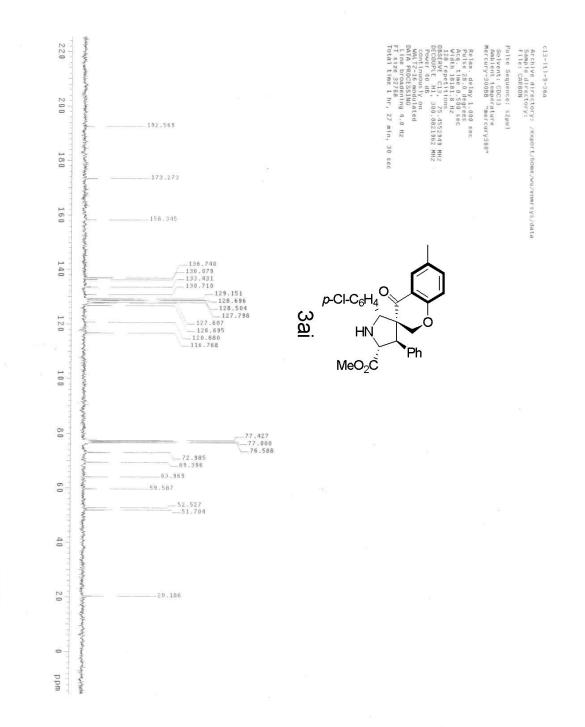
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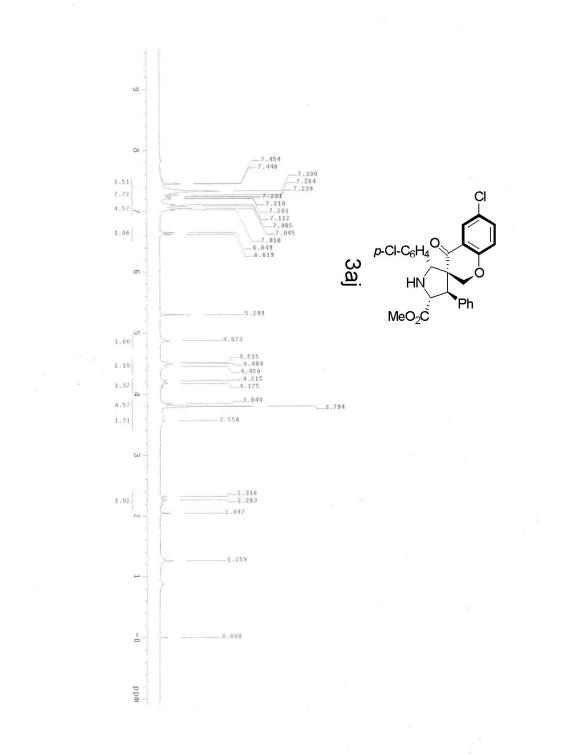


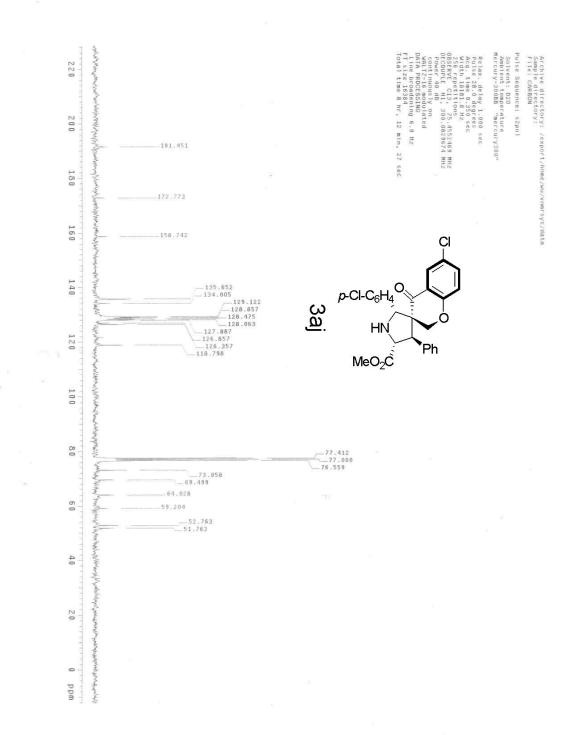








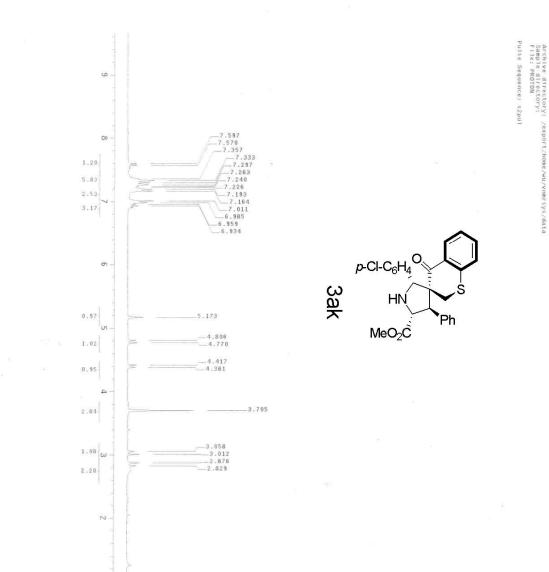


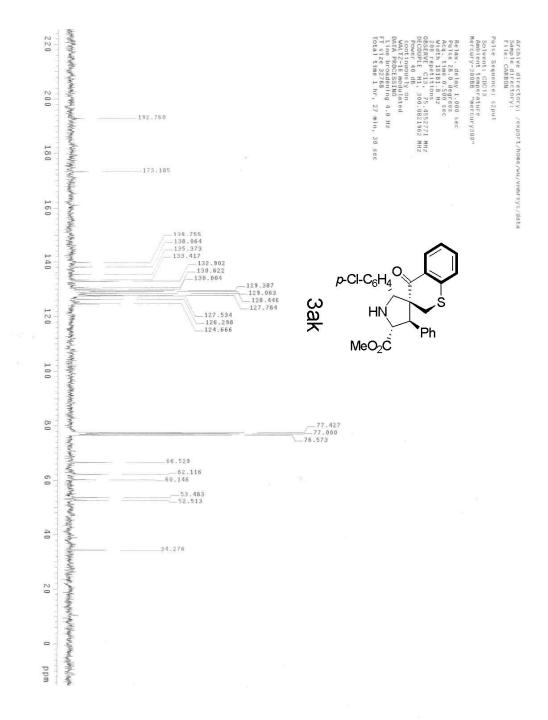


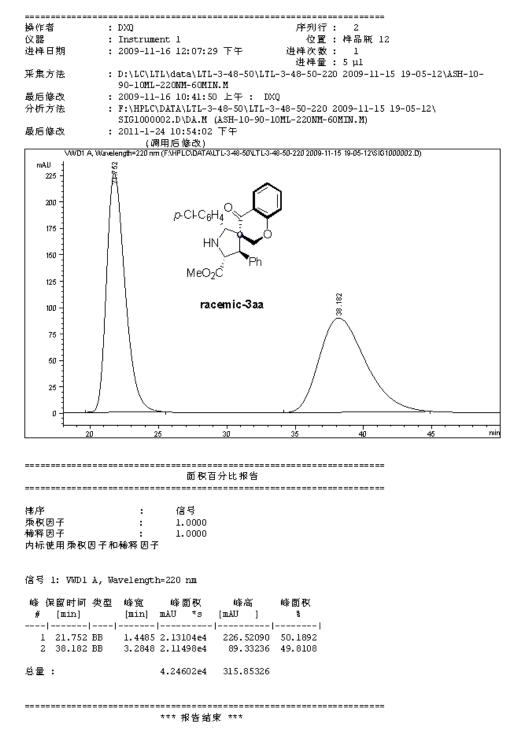
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bbu







数据文件: F:\HPLC\DATA\LTL-3-48-50\LTL-3-48-50-220 2009-11-15 19-05-12\SIG1000002.D 样品名称: LTL-3-48

仪器 1 2011-1-24 10:54:14 下午

页1/1

Data File D:\LC\LTL\DATE\LTL-8-44\LTL-8-44 2010-11-15 16-27-54\070-0301.D Sample Name: LTL-8-44B Seq. Line : 3 Acq. Operator : DXQ Location : Vial 70 Acq. Instrument : Instrument 1 Inj: 1 Injection Date : 11/15/2010 5:32:04 PM Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-8-44\LTL-8-44 2010-11-15 16-27-54\ASH-10-90-1ML-220NM-Acq. Method 50MIN.M Last changed : 8/30/2010 8:44:32 AM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-8-44\LTL-8-44 2010-11-15 16-27-54\070-0301.D\DA.M (ASH-10-90-1ML-220NM-50MIN.N) : 11/19/2010 2:37:26 PM by THL Last changed (modified after loading) W/D1 A Wavelergt=220 nm (D/LC/TL/DATELTL-844/LTL-844/2010-11-15 16-27-54070-0301.D) 0000 mAU 400 p-CI-C₆H 300 MeO₂C 3aa 200 .001301 458 100 0 45 min 30 25 Area Percent Report Signal Sorted By : 1.0000 : Multiplier 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VUD1 &, Wavelength=220 nm Height Area Peak RetTime Type Width Area [min] mAU *s [mAU] a. [min] # -1--1--1 -- | ------ | -83.28065 6.9752 1 22.458 MM 1.6156 8073.06641 2 37.534 MM 3.8536 1.07667e5 465.64847 93.0248 2 37.534 MM 1.15740e5 548.92912 Totals :

Instrument 1 11/19/2010 2:37:31 PM THL

Data File D:\LC\LTL\DATE\LTL-9-42\LTL-9-42& 2011-02-15 20-11-47\071-0201.D Sample Name: LTL-9-42A Acq. Operator : LTL Acq. Instrument : Instrument 1 Injection Date : 2/15/2011 8:24:04 PM Seq. Line : 2 Location : Vial 71 Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-9-42\LTL-9-42& 2011-02-15 20-11-47\ADH-30-70-1ML-220NM-Acq. Method 70MIN.M Last changed : 2/15/2011 10:56:36 AM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-9-42\LTL-9-42& 2011-02-15 20-11-47\071-0201.D\DA.M (ADH-30-70-1ML-220NM-70MIN.M) : 2/16/2011 10:27:14 AM by LTL Last changed (modified after loading) W/DIA Wavelength=220 nm (DALCUTU/DATEXITU-942A1TU-942A2011-02-15 20-11-47071-0201.D) Jas. Ashing 12121 mAU 1600 1400 0-CI-CaH HN 1200 e. MeO₂C 1000 racemic-3ba 78 800 600 400 200 0 20 18 22 24 14 18 Area Percent Report _____ Sorted By Signal Multiplier : 1.0000 1.0000 Dilution 120 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area [min] mAU *s [mAU] 970 # [min] -11 12.127 MM 0.5095 4.98426e4 1630.31836 49.6966 2 23.778 MM 1.0098 5.04512e4 832.65930 50.3034 1.00294e5 2462.97766 Totals :

Instrument 1 2/16/2011 10:27:18 AM LTL

Electronic Supplementary Material (ESI) for Chemical Communications This journal is C The Royal Society of Chemistry 2011

Sample Name: LTL-9-42B Acq. Operator : LTL Acq. Instrument : Instrument 1 Seq. Line : 3 Location : Vial 62 Injection Date : 2/16/2011 11:31:21 AM Inj : 1 Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-9-41\LTL-9-41B 2011-02-16 10-32-03\ADH-30-70-1ML-220MM-Acq. Method 30MIN.M Last changed : 1/22/2011 10:59:12 AM by THL-7-95-97 Analysis Method : D:\LC\LTL\DATE\LTL-9-41\LTL-9-41B 2011-02-16 10-32-03\062-0301.D\DA.M (ADH-30-70-1ML-220NM-30MIN.M) Last changed : 2/16/2011 2:45:55 PM by LTL (modified after loading) weigh=220 nm(DALCUTLVDATELTL-941VLTL-9418 2011-02-16 10-32-03062-0301.D) V0/D1 & V05 m,AU 8 1100 A o2 3000 2500 o-Cl-Cat 2000 MeO2Č 3ba 1500 1000 500 0 14 16 24 min 26 Area Percent Report Sorted By Signal : Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] 5 -- | ---1 -----1 ------1 0.5926 1.17681e5 3309.99829 94.4588 0.9442 6903.51270 121.86088 5.5412 1 11.935 MM 2 23.727 MM Totals : 1.24584e5 3431.85917

Data File D:\LC\LTL\DATE\LTL-9-41\LTL-9-41B 2011-02-16 10-32-03\062-0301.D

Instrument 1 2/16/2011 2:45:59 PM LTL

Data File D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\061-0201.D Sample Name: LTL-9-41A

	: LTL Seq. Line : 2
Acq. Instrument	: Instrument 1 Location : Vial 61
	: 6/9/2011 12:22:48 PM Inj: 1
2	Inj Volume : 5 µl
Acq. Method	: D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\ADH-30-70-1ML-
042	220NM-50MIN-NOT.M
Last changed	: 6/9/2011 12:11:29 PM by hz1
Analysis Method	1 : D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\061-0201.D\DA.M (
	ADH-30-70-1ML-220NM-50MIN-NOT.M)
Last changed	: 6/13/2011 9:51:14 PM by LTL
	(modified after loading)
200107 W	avelength=220 nm (DALCALTIVDATEATL-11-28/LTL-11-28/2011-06-09/12-10-08/061-0201.D)
mAU -	で 第
1600 -	
1.400	
1400 -	m-CI-C ₆ H ₄
1200 -	
1200	MeO2 ^C Ph
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	Area Percent Report
o +	Area Percent Report : Signal
0	Area Percent Report : Signal
Sorted By Multiplier Dilution	۸rea Percent Report : Signal : 1.0000 : 1.0000
Sorted By Multiplier Dilution	Area Percent Report : Signal : 1.0000
Sorted By Multiplier Dilution	۸rea Percent Report : Signal : 1.0000 : 1.0000
Sorted By Multiplier Dilution Use Multiplier	۸rea Percent Report : Signal : 1.0000 : 1.0000
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1	Area Percent Report : Signal : 1.0000 : 1.0000 6 Dilution Factor with ISTDs A, Wavelength=220 nm
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime Ty	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs A, Wavelength=220 nm mpe Width Area Height Area
o 10 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=220 nm pe Width Area Height Area [min] mAU *s [mAU] %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 6 Dilution Factor with ISTDs A, Wavelength=220 nm pe Width Area Height Area [min] mAU *s [mAU] %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 6 Dilution Factor with ISTDs Å, Wavelength=220 nm mpe Width Area [min] mAU %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 6 Dilution Factor with ISTDs A, Wavelength=220 nm pe Width Area Height Area [min] mAU *s [mAU] %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs Å, Wavelength=220 nm mpe Width Årea [min] mÅU * [mÅU * [mÅU * [mÅU * [mÅU * (mÅU * (mÅU </td
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 6 Dilution Factor with ISTDs Å, Wavelength=220 nm mpe Width Area [min] mAU %
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime Ty # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs Å, Wavelength=220 nm mpe Width Årea [min] mÅU * [mÅU * [mÅU * [mÅU * [mÅU * (mÅU * (mÅU </td
o 10 Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 1 11.974 WV 2 23.240 EB Totals :	Area Percent Report : Signal : 1.0000 : 1.0000 6 Dilution Factor with ISTDs Å, Wavelength=220 nm Tpe Width Årea [min] mÅU * [mÅU] *
o 10 Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD1 Peak RetTime Ty # [min] 1 11.974 WV 2 23.240 EB Totals :	Area Percent Report : Signal : 1.0000 : 1.0000 & Dilution Factor with ISTDs Å, Wavelength=220 nm mpe Width Årea [min] mÅU * [mÅU * [mÅU * [mÅU * [mÅU * (mÅU * (mÅU </td

Instrument 1 6/13/2011 9:51:18 PM LTL

Sample Name: LTL-11-28A Acq. Operator : LTL Seq. Line : 3 Acq. Instrument : Instrument 1 Injection Date : 6/9/2011 1:14:36 PM Location : Vial 62 Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\ADH-30-70-1ML-220NM-50MIN-NOT.M : 6/9/2011 12:11:29 PM by hzl Last changed Analysis Method : D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\062-0301.D\DA.M (ADH-30-70-1ML-220NM-50MIN-NOT.M) : 6/13/2011 9:51:37 PM by LTL Last changed (modified after loading) engb=220 nm(DALCLTUDATELTL-11-28/LTL-11-28 2011-08-09 12-10-08/062-0301.D) MO/DI A Wavele mAU 60 2000 -1750 mCLCeH 1500 -MeO₂C 1250 -3ca 1000 750 500 049 250 23. 0 14 16 18 20 24 26 min Area Percent Report Sorted By Signal 2 Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area [min] mAU *s # [min] [mAU] 2 - ---------------1--1 1 11.809 BB 0.4324 6.21266e4 2136.55786 95.2202 2 23.049 EB 0.7556 3118.56201 61.55494 4.7798 Totals : 6.52451e4 2198.11280

Data File D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\062-0301.D

*** End of Report ***

Instrument 1 6/13/2011 9:51:43 PM LTL

Sample Name: LTL-8-53A

Acq. Operator : THL Acq. Instrument : Instrument 1 Injection Date : 12/7/2010 10:40:48 AM Seq. Line : 2 Location : Vial 55 : 12/7/2010 10:40:48 AM Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-8-53\LTL-8-53A 2010-12-07 10-28-20\ADH-30-70-1ML-220NM-Acq. Method SOMIN.M Last changed : 11/27/2010 11:06:07 AM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-8-53\LTL-8-53& 2010-12-07 10-28-20\055-0201.D\DA.M (ADH-30-70-1ML-220NM-80MIN.M) : 12/7/2010 7:05:30 PM by THL Last changed (modified after loading) WVD1 A Wavelength=220 nm(DNLCLTL/DATELTL-8-53NLTL-8-53A 2010-12-07 10-28-20055-0201.D) mAU 0-0.02 800 700 600 MeO₂Č racemic-3da 500 45.899 400 300 200 100 ñ 50 55 45 30 i mi 16 20 25 ____ _____ Area Percent Report Signal Sorted By : Multiplier 1.0000 : . 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area [min] mAU *s [mAU] 10 * [min] -----|------| 1 16.062 VB 0.6031 3.56487e4 873.00519 49.9184 2 45.899 BB 1.7280 3.57653e4 307.55499 50.0816 7.14140e4 1180.56018 Totals :

Data File D:\LC\LTL\DATE\LTL-8-53\LTL-8-53A 2010-12-07 10-28-20\055-0201.D

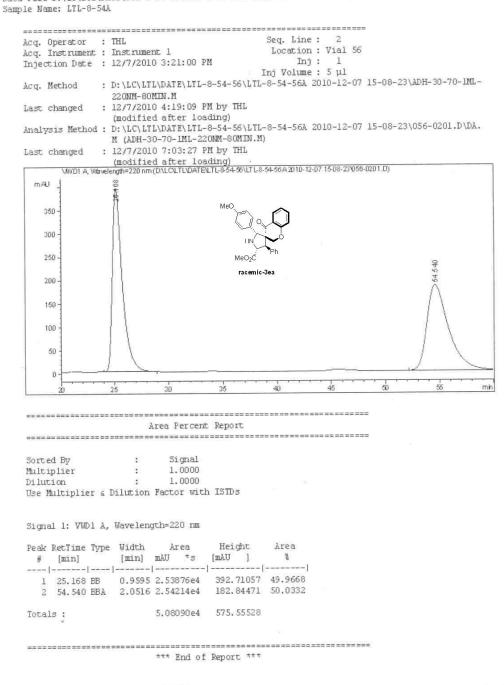
Instrument 1 12/7/2010 7:05:34 PM THL

Sample Name: LTL-9-29A

Seq. Line : 2 Acq. Operator : thl Location : Vial 63 Acq. Instrument : Instrument 1 Injection Date : 1/12/2011 10:35:10 AM Inj: 1 Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29A 2011-01-12 10-22-43\ADH-30-70-1ML-220MM-Acq. Method 60MIN.M Last changed : 12/7/2010 3:06:41 PM by THL Analysis Method : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29A 2011-01-12 10-22-43\063-0201.D\DA.M (ADH-30-70-1ML-220MM-60MIN.M) : 1/24/2011 4:53:15 PM by LTL Last changed (modified after loading) ergth=220 nm(D%LCUTUDATENTL-9-29N1-01-01-1210-22-43063-0201.0) W/D1 A Wavele mAU 82 1750 1500 1250 3da 1000 750 500 129.707 250 Ď 50 15 20 30 Area Percent Report Signal Sorted By . 1.0000 Multiplier . 1.0000 : Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Height Area Peak RetTime Type Width Area # [min] [min] mAU *s [mAU] 5 -1-------1 16.762 EB 0.6723 8.82917e4 1955.83447 95.7878 2 49.707 MM 1.9543 3882.52197 33.11037 4.2122 1 16.762 BB 9.21742e4 1988.94485 Totals :

Data File D:\LC\LTL\DATE\LTL-9-29\LTL-9-29A 2011-01-12 10-22-43\063-0201.D

Instrument 1 1/24/2011 4:53:21 PM LTL



Data File D:\LC\LTL\DATE\LTL-8-54-56\LTL-8-54-56A 2010-12-07 15-08-23\056-0201.D Sample Name: LTL-8-54A

Instrument 1 12/7/2010 7:03:31 PM THL

Sample Name: LTL-9-29B Seq. Line : 1 Acq. Operator : DXQ Acq. Instrument : Instrument 1 Injection Date : 1/12/2011 3:47:39 PM Location : Vial 64 Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29B 2011-01-12 15-46-16\ADH-30-70-1ML-220MM-Acq. Method 60MIN.M : 1/12/2011 4:27:17 PM by DXQ Last changed (modified after loading) Analysis Method : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29B 2011-01-12 15-46-16\064-0101.D\DA.M (ADH-30-70-1ML-220NM-60MIN.M) : 1/24/2011 5:23:26 PM by LTL Last changed (modified after loading) VWD1 A Wavelength=220 nm (D\LCLTL\DATELTL-9-29\LTL-9-298 2011-01-12 15-48-16064-0101.D) mAU では山田 1200 MeC 1000 800 MeO₂ 36 600 400 200 808 60 D 55 60 min 50 30 4) 35 Area Percent Report ______ Signal Sorted By ×. 1.0000 Multiplier : 1.0000 Dilution 3 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] \$ d b # 1 26.921 EB 1.0181 8.55990e4 1251.12134 96.9249 2 60.809 EB 1.8770 2715.79858 19.20364 3.0751 8.83148e4 1270.32498 Totals_: *** End of Report ***

Data File D:\LC\LTL\DATE\LTL-9-29\LTL-9-29B 2011-01-12 15-46-16\064-0101.D

Instrument 1 1/24/2011 5:23:30 PM LTL

Sample Name: LTL-9-30A Seq. Line : 2 Acq. Operator : LYY Acq. Instrument : Instrument 1 Injection Date : 1/17/2011 7:28:35 PM Location : Vial 5 Inj : 1 Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-9-30\LTL-9-30A 2011-01-17 19-16-11\ADH-30-70-1ML-220MM-Acq. Method SOMIN.M : 1/17/2011 8:15:20 PM by LYY Last changed (modified after loading) Analysis Method : D:\LC\LTL\DATE\LTL-9-30\LTL-9-30A 2011-01-17 19-16-11\005-0201.D\DA.M (ADH-30-70-1ML-220NM-80MIN.M) : 1/24/2011 5:06:58 PM by LTL Last, changed (modified after loading) \W/DIA Wavelergth=220 nm(D\LCLTLUDATELTL-9-30\LTL-9-30A2011-01-17 19-16-11/005-0201.0) ese esta 10-658 mAU 1600 1400 1200 1000 MeO₂C 630 racemic-3fa 800 \$2 600 -400 200 n. 45 35 40 min 20 25 30 Area Percent Report ______ Signal Sorted By . : 1.0000 Multiplier 1.0000 : Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] m&U *s [m&U] % d'D -1 16.556 MM 0.7480 7.93428e4 1767.99084 49.4192 2 43.630 BB 1.7742 8.12076e4 676.02759 50.5808 1.60550e5 2444.01843 Totals : *** End of Report ***

Data File D:\LC\LTL\DATE\LTL-9-30\LTL-9-30A 2011-01-17 19-16-11\005-0201.D

Instrument 1 1/24/2011 5:07:02 PM LTL

TC BCHICE NTW -	\DATE\LTL-9-30\LTL-9-30B 2011-01-22 09-47-32\069-0201.D 30B
le Name: LTL-9-	
Acq. Operator	: THL-7-95-97 Seq. Line : 2
log Instrument	: Instrument 1 Location : Vial 69
Injection Date	: 1/22/2011 10:05:00 AM INJ : 1
	In Volume : a ul
Acq. Method	: D:\LC\LTL\DATE\LTL-9-30\LTL-9-30B 2011-01-22 09-47-32\ADH-30-70-1ML-220MM 60MIN.M
Last changed	: 1/22/2011 10:53:50 AM by THL-7-95-97
Analysis Method	d: D:\LC\LTL\DATE\LTL-9-30\LTL-9-30B 2011-01-22 09-47-32\069-0201.D\DA.M (ADH-30-70-1ML-220NM-60MIN.M)
Last changed	: 1/24/2011 5:08:31 PM by LTL
10/D1 A 3/	(modified after loading) Novelength=220 nm(DMLCLTL/DATEXTL-9-301/LL-9-308 2011-01-22 09-47-32069-0201.D)
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	Area Percent Report
Sorted By	Area Percent Report : Signal
Sorted By Multiplier	Area Percent Report : Signal : 1.0000
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 Use Multiplier	Area Percent Report : Signal : 1.0000 : 1.0000 r & 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: VWD	<pre>Area Percent Report : Signal : 1.0000 : 1.0000 r 4 Dilution Factor with ISTDs 1 A, Wavelength=220 nm</pre>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD1 Peak RetTime 1	<pre>Area Percent Report : Signal : 1.0000 : 1.0000 r 4 Dilution Factor with ISTDs l A, Wavelength=220 nm Type Width Area Height Area</pre>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWD Peak RetTime 1 # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 r & Dilution Factor with ISTDs 1 Å, Wavelength=220 nm Type Width Årea Height Årea [min] mAU *s [mAU]
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMDJ Peak RetTime 7 # [min]	<pre>krea Percent Report : Signal : 1.0000 : 1.0000 r & Dilution Factor with ISTDs 1 Å, Wavelength=220 nm Type Width Årea Height Årea [min] mAU *s [mAU] %</pre>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMDJ Peak RetTime 2 # [min] 	Area Percent Report : Signal : 1.0000
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMDJ Peak RetTime 2 # [min] 	<pre>krea Percent Report : Signal : 1.0000 : 1.0000 r & Dilution Factor with ISTDs 1 Å, Wavelength=220 nm Type Width Årea Height Årea [min] mAU *s [mAU] %</pre>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD Peak RetTime 7 # [min] 	<pre>krea Percent Report : Signal : 1.0000 : 1.0000 r 4 Dilution Factor with ISTDs l A, Wavelength=220 nm Type Width Area Height Area [min] mAU *s [mAU] %</pre>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMDJ Peak RetTime 2 # [min] 	Area Percent Report : Signal : 1.0000
Sorted By Multiplier Dilution Use Multiplier Signal 1: VMD Peak RetTime 7 # [min] 	<pre>krea Percent Report : Signal : 1.0000 : 1.0000 r 4 Dilution Factor with ISTDs l A, Wavelength=220 nm Type Width Area Height Area [min] mAU *s [mAU] %</pre>
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWDJ Peak RetTime 7 # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 r 4 Dilution Factor with ISTDs 1 Å, Wavelength=220 nm Type Width Årea Height Årea [min] mÅU *s [mÅU] * 1.8286 1.98583e5 3459.97070 94.5218 MM 1.8286 1.15092e4 104.90173 5.4782 2.10092e5 3564.87243
Sorted By Multiplier Dilution Use Multiplier Signal 1: VWDJ Peak RetTime 7 # [min] 	<pre>krea Percent Report : Signal : 1.0000 : 1.0000 r 4 Dilution Factor with ISTDs l A, Wavelength=220 nm Type Width Area Height Area [min] mAU *s [mAU] %</pre>

Instrument 1 1/24/2011 5:08:35 FM LTL

Data File D:\LC\LTL\DATE\LTL-11-13\LTL-11-13& 2011-06-01 08-29-22\034-0201.D Sample Name: LTL-11-13A Acq. Operator : LTL Seq. Line : 2 Acq. Instrument : Instrument 1 Location : Vial 34 Injection Date : 6/1/2011 8:41:57 AM Inj: 1 Inj Volume: 5 µl : D: \LC\LTL\DATE\LTL-11-13\LTL-11-13& 2011-06-01 08-29-22\ADH-30-70-1ML-Acq. Method Last changed : D:\LC\LILDATE\LIL-11-13\LIL-11-13A 2011-06-01 08-29-22\ADH-30-70-1ML-Last changed : 11/27/2010 11:06:07 AM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-11-13\LTL-11-13A 2011-06-01 08-29-22\034-0201.D\DA.M (: 6/1/2011 3:20:14 PM by LTL (modified after loading) ngm=220 nm(DALCUTUDATEUTL-11-13/LTL-11-13A2011-06-01.08-29-22/0340201.0) 10/D1 A 10/D01 mAU 22 12.879 600 C₆H HN 500 MeO₂C 400 racemic-3ga 300 200 100 D 10 11 12 14 min Area Percent Report Sorted By : Signal Multiplier 5 1,0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VUD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] 1 ------1 1 10.555 VB 0.3760 1.73337e4 686.65393 50.0591 2 12.879 BB 0.4580 1.72928e4 566.58771 49.9409 Totals : 3.46265e4 1253.24164 *** End of Report ***

istrument 1 6/1/2011 3:20:18 PM LTL

Data File D:\LC\LTL\DATE\LTL-11-13\LTL-11-13B 2011-06-01 15-01-45\035-0101.D Sample Name: LTL-11-13B Acq. Operator : LTL Acq. Instrument : Instrument 1 Seq. Line : 1 Location : Vial 35 Injection Date : 6/1/2011 3:03:17 PM Inj: 1 Inj Volume: 5 µ1 : D:\LC\LTL\DATE\LTL-11-13\LTL-11-13B 2011-06-01 15-01-45\ADH-30-70-1ML-Acq. Method 220NM-25MIN.M : 6/1/2011 3:16:55 PM by LTL Last changed (modified after loading) Analysis Method : D: \LC\LTL\DATE\LTL-11-13\LTL-11-13B 2011-06-01 15-01-45\035-0101.D\DA.M (ADH-30-70-1ML-220NM-25MIN.M) : 6/1/2011 3:19:45 PM by LTL Last changed (modified after loading) WWD1 A Wavelength=220 nm (DALCULTLVDATEWITL-11-13WITL-11-138 2011-06-01 15-01460035-0101.D) mAU 10 KOSP A A 600 o-Me-CeH 500 HN MeO₂C 400 3da 300 200 100 0 10 11 12 14 min Area Percent Report _____ Sorted By : Signal Multiplier : 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Area Height [min] mAU *s # [min] [mAU] 뭅 ---- |----- |----- |----------1 10.510 MM 0.4163 1.70504e4 682.63660 94.6384 2 12.816 MM 0.4792 965.96259 33.59390 5.3616 Totals : 1.80164e4 716.23050

*** End of Report ***

strument 1 6/1/2011 3:19:50 PM LTL

Data File D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\063-0401.D Sample Name: LTL-11-14A Acq. Instrument : Instrument 1 Acq. Operator : LTL Seq. Line : 1 Location : Vial 63 Injection Date : 6/9/2011 2:06:07 PM Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\ADH-30-70-1ML-Acg. Method 220NM-40MIN-NOT.M : 6/9/2011 2:30:27 PM by LTL (modified after loading) Last changed Analysis Method : D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\063-0401,D\DA.M (ADH-30-70-1ML-220NM-40MIN-NOT.M) : 6/13/2011 9:52:05 PM by LTL Last changed (modified after loading) end=220 nm(D)LCLTLDATENTL-11-282LTL-11-282011-06-09 12-10-08063-0401.D) VIII/D1 A. Wavelength mAU 99 1000 800 m-Me-C₆H 21.748 600 MeO₂C racemic-3h 400 200 0 22 24 20 10 12 14 18 min 16 Area Percent Report Sorted By : Signal Multiplier : 1,0000 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area (min) mAU *s [mAU] # [min] 2 -1--1 1 9.860 VB 0.3486 2.67065e4 1135.79907 49.7768 2 21.748 BB 0.7475 2.69460e4 536.53448 50.2232 Totals : 5.36525e4 1672.33356

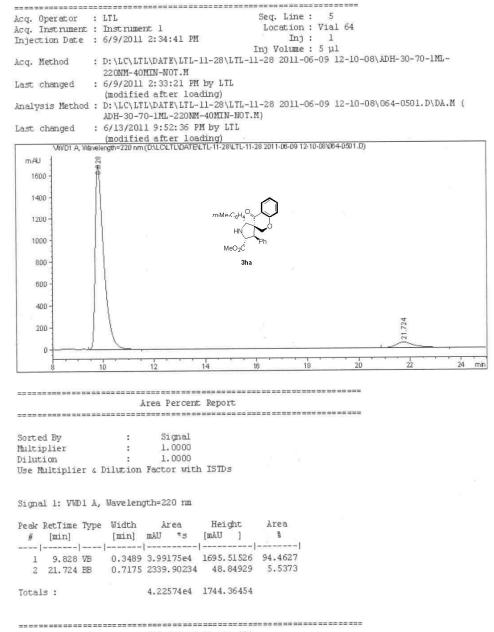
*** End of Report ***

Instrument 1 6/13/2011 9:52:09 PM LTL

Page 1 of 1

75

Data File D:\LC\LTL\DATE\LTL-11-28\LTL-11-28 2011-06-09 12-10-08\064-0501.D Sample Name: LTL-11-28B



*** End of Report ***

Instrument 1 6/13/2011 9:52:40 PM LTL

Sample Name: LTL-11-15A Acq. Operator : LTL Acq. Instrument : Instrument 1 Injection Date : 6/1/2011 9:50:41 AM Seq. Line : 1 Location : Vial 32 Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-11-15\LTL-11-15A-AD 2011-06-01 09-49-02\ADH-30-70-1ML-Acq. Method 220NM-80MIN.M : 6/1/2011 10:36:50 AM by LTL Last changed (modified after loading) Analysis Method : D:\LC\LTL\DATE\LTL-11-15\LTL-11-15A-AD 2011-06-01 09-49-02\032-0101.D\DA.M (ADH-30-70-1ML-220NM-80MIN.M) Last changed : 6/1/2011 3:21:14 PM by LTL (modified after loading) WWDIA Wavelength=220 nm(D/LCNTLVDATEVIT-11-15/LTL-11-15A-AD 2011-06-01 09-49-02/032-0101.D) mAU 69 800 700 600 740 500 4 MeO₂Ċ 400 racemic-3ia 300 200 100 0 m 25 30 45 35 40 min Area Percent Report Sorted By Signal 1 Multiplier 1.0000 Dilution 2 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VUD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] 10 ---|-----1 21.159 EB 0.7944 4.38642e4 821.92499 50.1692 2 40.740 EB 1.5097 4.35683e4 430.54077 49.8308 Totals : 8.74325e4 1252.46576

Data File D:\LC\LTL\DATE\LTL-11-15\LTL-11-15A-AD 2011-06-01 09-49-02\032-0101.D

*** End of Report ***

Instrument 1 6/1/2011 3:21:18 PM LTL

Sample Name: LTL-11-15B Acq. Operator : LTL Acq. Instrument : Instrument 1 Injection Date : 6/1/2011 4:01:38 PM Seq. Line : 3 Location : Vial 37 Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-11-13\LTL-11-13B 2011-06-01 15-01-45\ADH-30-70-1ML-Acq. Method 220NM-50MIN.M Last changed : 11/29/2010 10:38:15 AM by THL Analysis Method : D:\LC\LTL\DATE\LTL-11-13\LTL-11-13B 2011-06-01 15-01-45\037-0301.D\DA.M (ADH-30-70-1ML-220NM-50MIN.M) Last changed : 6/1/2011 4:52:29 PM by LTL (modified after loading) WVD1 A Wavelength=220 nm (DALCVITUDATELTL-11-13%ITL-11-13% 2011-06-01 15-0146/037-0301.D) mAll 04 ŝ 1600 1400 1200 1000 MeObC 800 3ia 600 400 6.07 200 40 θ 25 30 35 40 min Area Percent Report Sorted By 5 Signal Multiplier 5 1.0000 Dilution 8 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VMD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area [mAU] # [min] [min] mAU *s 5 ----| 1 20.912 BB 0.8020 9.24083e4 1710.70728 94.9949 2 40.667 BB 1.4488 4868.79541 50.66391 5.0051 Totals : 9.72771e4 1761.37119 *** End of Report ***

Data File D:\LC\LTL\DATE\LTL-11-13\LTL-11-13B 2011-06-01 15-01-45\037-0301.D

Instrument 1 6/1/2011 4:52:33 PM LTL

Seq. Line : 3 Acq. Operator : THL Location : Vial 57 Acq. Instrument : Instrument 1 Injection Date : 12/7/2010 4:22:53 PM Inj: 1 Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-8-54-56\LTL-8-54-2010-12-07 15-08-23\ADH-30-70-1ML-Acq. Method 220NM-60MIN.M Last changed : 12/7/2010 3:06:41 PM by THL Analysis Method : D:\LC\LTL\DATE\LTL-8-54-56\LTL-8-54-56\ 2010-12-07 15-08-23\057-0301.D\DA. M (ADH-30-70-1ML-220MM-60MIN.M) : 12/7/2010 7:02:41 PM by THL Last changed (modified after loading) WWD1A Wavelergt=220 nm(D/LC/LTU/DATELTL-8-54-66/LTL-8-54-66A.2010-12-07 15-08-23/057-0301.0) 02 mAU 2000 1750 1500 MeO2C 1250 racemic-3ja 56.218 1000 750 500 250 ñ 55 50 mit 40 30 Area Percent Report Signal : Sorted By : 1.0000 Multiplier : 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Area Area Height Peak RetTime Type Width [mAU] d b [min] mAU *s # [min] - | ----- | ------ | ------- | ------------ 1 20.648 VB 0.8054 1.20859e5 2230.42065 50.2322 2 56.218 EBA 2.1804 1.19742e5 814.13837 49.7678 2.40601e5 3044.55902 Totals :

Data File D:\LC\LTL\DATE\LTL-8-54-56\LTL-8-54-56& 2010-12-07 15-08-23\057-0301.D Sample Name: LTL-8-56&

Instrument 1 12/7/2010 7:02:45 PM THL

Data File D:\LC\LTL\DATE\LTL-9-29\LTL-9-29D 2011-01-12 18-44-26\066-0101.D Sample Name: LTL-9-29D Seq. Line : 1 Acq. Operator : DXQ Location : Vial 66 Acq. Instrument : Instrument 1 Inj: 1 Inj Volume: 5 µl Injection Date : 1/12/2011 6:46:04 PM : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29D 2011-01-12 18-44-26\ADH-30-70-1ML-220NM-Acq. Method SOMIN.M Last changed : 11/27/2010 11:06:07 AM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29D 2011-01-12 18-44-26\066-0101.D\DA.M (ADH-30-70-1ML-220NM-80MIN.M) : 1/24/2011 4:55:59 PM by LTL Last changed (modified after loading) WWD1 A Wawelergth=220 nm(D/LCVITUDATEVITU-9-29VITU-9-29D 2011-01-12 18-44-26066-0101.D) -000 -100 35 mAU 1750 1500 1250 MeO₂O 1000 3ia 750 500 18190000000 250 ñ 60 ά 20 30 40 ============ Area Percent Report Signal Sorted By : 1.0000 Multiplier . 1.0000 : Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Height Area Peak RetTime Type Width Area 1 # [min] [min] mAU *s [mAU] -1---- | ------ | ---------1 14.125 MM 0.5796 6.93170e4 1993.36377 93.0956 2 61.931 MM 2.4289 5140.88037 35.27583 6.9044 7.44578e4 2028.63960 Totals :

Instrument 1 1/24/2011 4:56:03 PM LTL

Data File D:\LC\LTL\DATE\LTL-8-54-56\LTL-8-54-56A 2010-12-07 15-08-23\058-0401.D Sample Name: LTL-8-55A Acq. Operator : THL Acq. Instrument : Instrument 1 Injection Date : 12/7/2010 5:24:08 PM Seq. Line: 4 Location : Vial 58 Inj: 1 Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-8-54-56\LTL-8-54-56A 2010-12-07 15-08-23\ADH-30-70-1ML-Acg. Method 220NM-60MIN.M : 12/7/2010 5:29:53 PM by THL Last changed (modified after loading) Analysis Method : D:\LC\LTL\DATE\LTL-8-54-56\LTL-8-54-56\ 2010-12-07 15-08-23\058-0401.D\DA. M (ADH-30-70-1ML-220NM-60MIN.M) : 12/7/2010 7:04:36 PM by THL Last changed (modified after loading) WWD1A, Wavelergth=220 nm(DALCALTLDATELTL-864-66ALTL8-54-66A2010-12-07 15-08-23068-0401.D) mAU 9 700 600 500 020 400 racemic-3ka 300 862 200 100 Û 50 60 min ÷2 40 20 Area Percent Report Signal Sorted By 120 1.0000 Multiplier 1 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm -1 1 13.348 EB 0.4798 2.60438e4 801.07672 49.6298 2 56.862 EB 2.0802 2.64323e4 187.64384 50.3702 5.24761e4 988.72057 Totals : *** End of Report *** Instrument 1 12/7/2010 7:04:39 PM THL

Sample Name: LTL-9-29C Seq. Line : 1 Acq. Operator : DXQ Location : Vial 65 Acq. Instrument : Instrument 1 Inj: 1 Injection Date : 1/12/2011 4:55:36 PM Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29C 2011-01-12 16-54-15\ADH-30-70-1ML-220MM-Acq. Method 60MTN, M : 1/12/2011 5:28:48 PM by DXQ Last changed (modified after loading) Analysis Method : D:\LC\LTL\DATE\LTL-9-29\LTL-9-29C 2011-01-12 16-54-15\065-0101.D\DA.M (ADH-30-70-1ML-220NM-60MIN.M) : 1/24/2011 5:24:14 PM by LTL Last changed (modified after loading) (MWD) A. Wavelength=220 nm(D)LCVLTVDATEVIL-9-29VLTV-9-29C2011-01-12 16-54-15065-0101.D) mAU 12435 3500 3000 2500 2000 3ka 1500 1000 624 500 5 0 60 min 30 4 50 Ň Area Percent Report Signal Sorted By . 1.0000 Multiplier . 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VUD1 &, Wavelength=220 nm Peak RetTime Type Width Area Height Area # [min] [min] mAU *s [mAU] % 1 21.435 VB 1.0236 3.18022e5 3682.40796 88.8348 2 61.624 BB 2.3637 3.99707e4 250.91745 11.1652 3.57993e5 3933.32541

Data File D:\LC\LTL\DATE\LTL-9-29\LTL-9-29C 2011-01-12 16-54-15\065-0101.D

Totals ;

*** End of Report ***

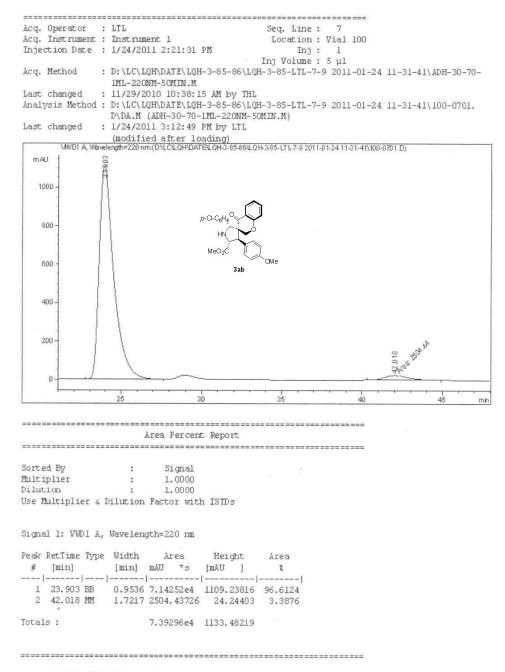
Instrument 1 1/24/2011 5:24:17 PM LTL

Data File D:\LC\LTL\DATE\LTL-8-64-67\LTL-8-64-67A-1 2010-11-27 11-08-15\050-0201.D Sample Name: LTL-8-64A

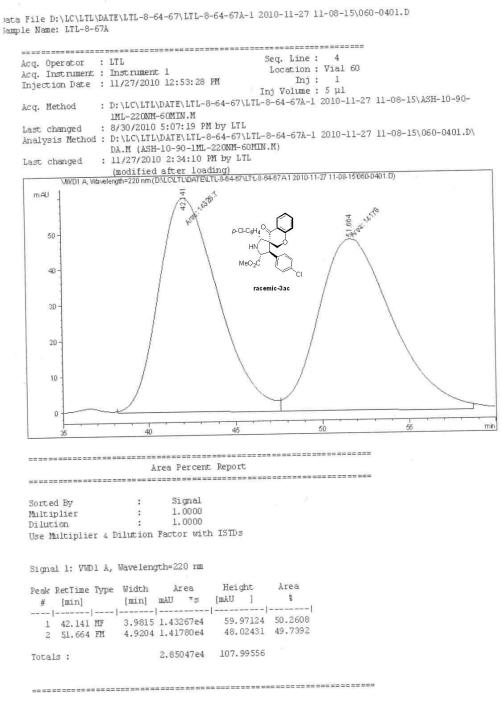
Acq. Operator :	
	: LTL Seq. Line : 2
Acq. Instrument :	
Injection Date :	: 11/27/2010 11:20:46 AM Inj: 1
	Inj Volume : 5 µl
Acq. Method :	: D:\LC\LTL\DATE\LTL-8-64-67\LTL-8-64-67A-1 2010-11-27 11-08-15\ADH-30-70-
	LML-220NM-80MIN.M
Last changed :	: 11/27/2010 11:06:07 AM by LTL
Analysis Method :	: D:\LC\LTL\DATE\LTL-8-64-67\LTL-8-64-67A-1 2010-11-27 11-08-15\050-0201.D\
	DA.M (ADH-30-70-1ML-220NM-80MIN.M)
Last changed :	: 11/27/2010 2:34:39 PM by LTL
	(modified after loading)
VII/D1 A, Vitave	elength=220 nm (DALCALTUDATENTL-864-67ATL-8-64-67A 1 2010-11-27 11-08-15050-0201.D)
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jorted By fultiplier	Area Percent Report : Signal : 1.0000
2 30 Sorted By fultiplier Silution	Area Percent Report : Signal : 1.0000 : 1.0000
20 Sorted By fultiplier Vilution Jse Multiplier 4	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs
20 Sorted By Multiplier Dilution Jse Multiplier 4	Area Percent Report : Signal : 1.0000 : 1.0000
20 Sorted By Multiplier Dilution Jse Multiplier 4 Signal 1: VMD1 Å,	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=220 mm
5orted By Multiplier Multiplier Jac Multiplier & Signal 1: VWD1 A, Peak RetTime Type	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=220 nm e Width Area Height Area
20 Sorted By fultiplier Dilution Jse Multiplier & Signal 1: VWD1 Å, Peak RetTime Type # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=220 nm : Width Area Height Area [min] mAU *s [mAU] %
20 Sorted By Multiplier Dilution Jse Multiplier & Signal 1: VWD1 Å, Peak RetTime Type # [min]	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=220 nm e Width Area Height Area [min] mAU *s [mAU] %
20 Sorted By fultiplier Signal 1: VWD1 A, Peak RetTime Type # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=220 nm : Width Area Height Area [min] mAU *s [mAU] :
20 Sorted By Multiplier Dilution Jse Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=220 nm e Width Area Height Area [min] mAU *s [mAU] %
30 Sorted By Multiplier Jolution Jse Multiplier & Signal 1: VWD1 A, Peak RetTime Type # [min] 1 22.988 EV 2 39.349 EB	Area Percent Report : 1.0000 : 1.0000 Dilution Factor with ISTDs / Wavelength=220 nm e Width Area Height Area [min] mAU *s (mAU) %
20 Sorted By fultiplier Dilution Jse Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type # [min] 	Area Percent Report : Signal : 1.0000 : 1.0000 Dilution Factor with ISTDs , Wavelength=220 nm : Width Area Height Area [min] mAU *s [mAU] :

Instrument 1 11/27/2010 2:34:43 PM LTL

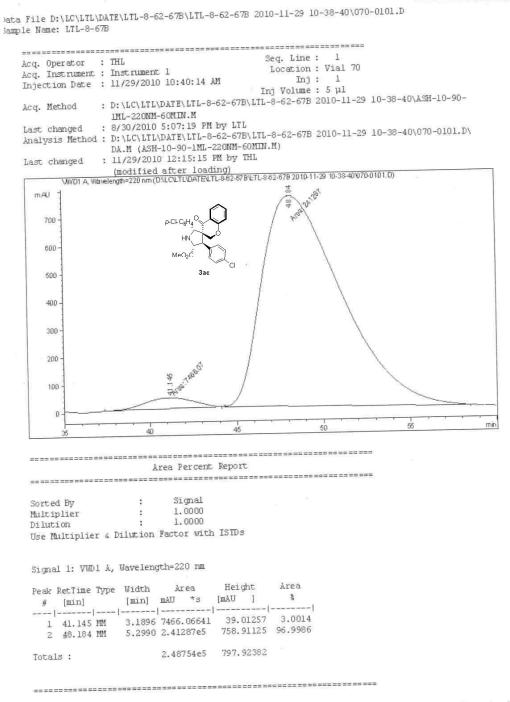
Data File D:\LC\LQH\DATE\LQH-3-85-86\LQH-3-85-LTL-7-9 2011-01-24 11-31-41\100-0701.D Sample Name: LTL-8-64B



Instrument 1 1/24/2011 3:12:53 FM LTL



Instrument 1 11/27/2010 2:34:15 PM LTL



Instrument 1 11/29/2010 12:15:19 PM THL

Data File D:\LC\LTL\DATE\LTL-8-62\LTL-8-62& 2010-11-26 20-04-25\049-0101.D Sample Name: LTL-8-62&

	: LTL Seq. Line : l
Acq. Operator	
Injection Date	: 11/26/2010 8:06:11 PM Inj : 1 Inj Volume : 5 µl
Acq. Method	: D:\LC\LTL\DATE\LTL-8-62\LTL-8-62& 2010-11-26 20-04-25\&SH-10-90-1ML-220NM.
Last changed	n : 6/25/2010 8:30:46 AM by LTL
Analysis Method	: D:\LC\LTL\DATE\LTL-8-62\LTL-8-62A 2010-11-26 20-04-25\049-0101.D\DA.M (ASH-10-90-1ML-220NM.M)
Last changed	: 11/29/2010 10:32:58 AM by THL
Dage changes	(modified after loading)
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Signal 1: VWD1	A, Wavelength=220 nm
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	[min] max b [min]
1 11.591 B	
2 44.676 M	1 3.0207 2.0035564 04.02033 37.7756
m	5 75505pd 621 65675
Totals :	5.75695e4 621.65675

Instrument 1 11/29/2010 10:33:02 AM THL

Data File D:\LC\LTL\DATE\LTL-8-62-67B\LTL-8-62-67B 2010-11-29 10-38-40\068-0301.D Sample Name: LTL-8-62B

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Acq. Instrument :	: Instrument 1	Location	
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	1ML-220NM-60MIN.M	0121010 0 02 01D 1	2010 11-25 10-30-40/R3H-10-90-
Last changed :	: 8/30/2010 5:07:19 PM by	1 7 1	
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istrument 1 11/29/2010 3:00:07 PM THL

mple Name: LTL-8-69A Acq. Operator : dxq Acq. Instrument : Instrument 1 Seq. Line : 2 Location : Vial 33 Inj: 1 Inj Volume: 5 µl Injection Date : 12/1/2010 5:32:38 PM : D:\LC\LTL\DATE\LTL-8-69\LTL-8-69A 2010-12-01 17-20-12\ASH-10-90-1ML-220NM. Acq. Method Μ : 12/1/2010 6:26:02 PM by dxq Last changed : 12/1/2010 6:35:40 PM by dxq (modified after loading) velength=220 nm(DXLCUTUDATELTL-8-69VLTL-8-69A2010-12-01 17-20-12003-0201.D) Last changed VIII/D1 A VIIdvele 55' FL 189" mAU 4 Ê 200 p-CFC₆H, HN 150 MeO₂0 109.66 racemic-3ae 100 50 D 30 55 50 40 min Area Percent Report

a File D:\LC\LTL\DATE\LTL-8-69\LTL-8-69A 2010-12-01 17-20-12\033-0201.D

Son	ted By		:	Sig	nal	
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Dil	ution			1.00	000	
Use	Multiplier	6	Dilution	Factor	with	ISTDS

Signal 1: VWD1 &, Wavelength=220 nm

Peak	RetTime	Type	Width	Ar	ea	Hei	.ght	Area
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1	21.314	MM	1.5798	2.216	594e4	233.	88683	50.2684
2	48.601	MM	4.5950	2.193	827e4	79.	55257	49.7316
Tota.	ls :			4.410)22e4	313.	43939	

*** End of Report ***

trument 1 12/1/2010 6:35:44 PM dxq

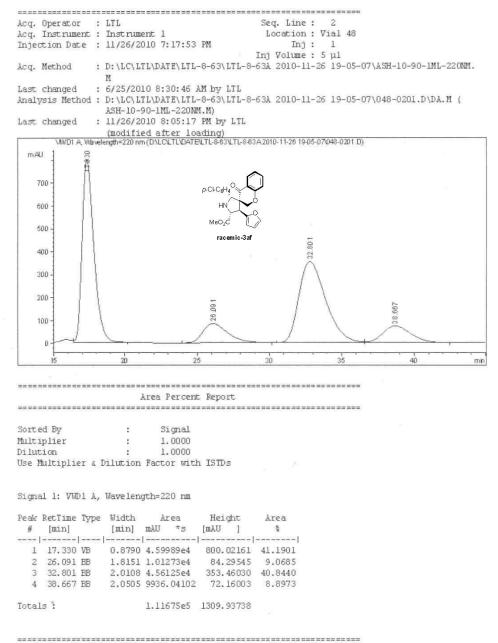
Data File D:\LC\LTL\DATE\LTL-8-69NLTL-8-69B 2010-12-02 10-51-14\034-0201.D Sample Name: LTL-8-69B

Acq. Operator :	
Acq. Instrument :	
Injection Date :	12/2/2010 11:03:40 AM Ini: 1
	Inj Volume : 5 µl
Acq. Method :	D:\LC\LTL\DATE\LTL-8-69\LTL-8-69B 2010-12-02 10-51-14\ASH-10-90-1ML-220NM-60MIN.M
Last changed :	8/30/2010 5:07:19 PM by LTL
Analysis Method :	D:\LC\LTL\DATE\LTL-8-69\LTL-8-69B 2010-12-02 10-51-14\034-0201.D\DA.M (
	ASH-10-90-1ML-220NM-60MIN.M)
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1000000 A	length=220 nm (D/LC/LTL/DATE/LTL-8-69/LTL-8-698 2010-12-02 10-51-14/034-0201.D)
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Signal I: YWDI A,	Wavelength=220 nm
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1 20 070 107	
2 A6 242 MM	1.4846 7393.44287 83.00124 4.5895
2 40.342 MM	4.9574 1.53702e5 516.74640 95.4105
Totals :	1.61095e5 599.74764
ioonio -	1.0103060 033.14.04

Instrument 1 12/2/2010 2:56:28 PM dxq

Data File D:\LC\LTL\DATE\LTL-8-63\LTL-8-63& 2010-11-26 19-05-07\048-0201.D Sample Name: LTL-8-63&



*** End of Report ***

Instrument 1 11/26/2010 8:05:22 PM LTL

Sample Name: LTL-8-63B

Acq. Operator : THL Acq. Instrument : Instrument 1 Injection Date : 11/29/2010 11:41:40 AM Seq. Line : 2 Location : Vial 69 Inj: 1 Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-8-62-67B\LTL-8-62-67B 2010-11-29 10-38-40\ASH-10-90-Acq. Method 1ML-220NM-60MIN.M Last changed : 8/30/2010 5:07:19 PM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-8-62-67B \LTL-8-62-67B 2010-11-29 10-38-40\069-0201.D\ DA.M (ASH-10-90-1ML-220MM-60MIN.M) Last changed : 11/29/2010 2:56:25 PM by THL (modified after loading) WWD1 A. Wavelength=220 nm (DALCULTUDATEUTL-862-678 ULTL-862-678 2010-11-29 10-38-40/069-0201.D) 951999 S mAU 600 500 400 300 3at 200 10° 100 0 min 30 30 -----Area Percent Report -----Sorted By Signal Multiplier : 1.0000 Dilution 3 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Height Peak RetTime Type Width Area Area [min] mAU *s [mAU] 뭅 # [min] 1 17.115 MM 0.9318 4501.31982 4.3593 80.51328 2 33.193 MF 2.3975 9.87566e4 686.52856 95.6407 1.03258e5 767.04184 Totals :

Data File D:\LC\LTL\DATE\LTL-8-62-67B\LTL-8-62-67B 2010-11-29 10-38-40\069-0201.D

Instrument 1 11/29/2010 2:56:29 PM THL

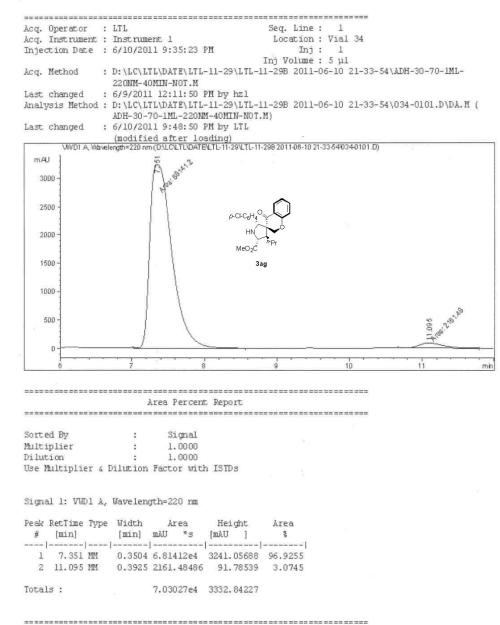
Page 1 of 1

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Data File D:\LC\LTL\DATE\LTL-11-29\LTL-11-29A 2011-06-10 19-40-25\031-0101.D Sample Name: LTL-11-29A Acq. Operator : LTL Seq. Line : 1 Location : Vial 31 Acq. Instrument : Instrument 1 Injection Date : 6/10/2011 7:41:59 PM Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-11-29\LTL-11-29& 2011-06-10 19-40-25\ADH-30-70-1ML-Acq. Method 220MM-50MIN-NOT.M : 6/9/2011 12:11:29 PM by hzl Last changed Analysis Method : D:\LC\LTL\DATE\LTL-11-29\LTL-11-29A 2011-06-10 19-40-25\031-0101.D\DA.M (ADH-30-70-1ML-220NM-50MIN-NOT.M) : 6/10/2011 9:16:06 PM by THL Last changed (modified after loading) ergt=220 nm(D)LCUTUDATELTL-11-29LTL-11-29A2011-06-10 19-40-25031-0101.D) VM/D1 A. Wavele 2400 mAU west and 2500 1.241 n-Cl-C-H 2000 MeO₂C racemic-3ag 1500 1000 500 Ð å 10 mi _____ Area Percent Report Signal Sorted By . 1.0000 Multiplier : 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area [min] mAU *s [mAU] 1 # [min] -1----1----------1 7.460 MM 0.2940 5.05365e4 2864.96631 49.0319 2 11.241 MM 0.4130 5.25321e4 2120.15552 50.9681 1.03069e5 4985.12183 Totals :

Instrument 1 6/10/2011 9:16:10 PM THL

Data File D:\LC\LTL\DATE\LTL-11-29\LTL-11-29B 2011-06-10 21-33-54\034-0101.D Sample Name: LTL-11-29B



*** End of Report ***

Instrument 1 6/10/2011 9:48:54 PM LTL

Data File D:\LC\LTL\DATE\LTL-11-19\LTL-11-19A 2011-06-02 09-41-21\022-0101.D Sample Name: LTL-11-19

Acq. Operator :		Seq. Line : 1
Acq. Instrument :		Location : Vial 22
injection Date :	: 6/2/2011 9:43:03	
a com a second com		Inj Volume : 5 µl
Acq. Method :		L-11-19\LTL-11-19A 2011-06-02 09-41-21\ADH-30-70-1ML-
	220NM-80MIN.M	
	: 11/27/2010 11:06:	
Analysis Method :		L-11-19/LTL-11-19A 2011-06-02 09-41-21/022-0101.D/DA.M
	ADH-30-70-1ML-220	
Last changed :	6/3/2011 4:31:05	PM by THL
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and a second sec	elength=220 nm (DALCALTEAD)	JTELTL-11-19/LTL-11-19A2011-06-02 09-41-21/022-0101.D)
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Sorted By Multiplier Dilution Use Multiplier 4	Area Percent : Signal : 1.0000 : 1.0000 Dilution Factor with	nt Report th ISTDs
Sorted By Multiplier Dilution Use Multiplier 4	Area Perces : Signal : 1.0000 : 1.0000	nt Report th ISTDs
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VWD1 A,	Area Percen : Signal : 1.0000 : 1.0000 Dilution Factor with . Wavelength=220 nm	nt Report
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type	Area Percen : Signal : 1.0000 : 1.0000 Dilution Factor with . Wavelength=220 nm : Width Area	nt Report th ISTDs Height Area
Sorted By Multiplier Dilution Use Multiplier & Signal 1: VWD1 A, Peak RetTime Type # [min]	Area Percen : Signal : 1.0000 : 1.0000 Dilution Factor win . Wavelength=220 nm : Width Area [min] mAU *s	nt Report th ISTDs Height Area [mAU] %
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type # [min]	Area Percen : Signal : 1.0000 : 1.0000 Dilution Factor with Wavelength=220 nm : Width Area [min] mAU *s	th ISTDs Height Area [mAU] %
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type # [min] 	Area Percer : Signal : 1.0000 : 1.0000 Dilution Factor win . Wavelength=220 nm : Width Area [min] mAU *s 	mt Report th ISTDs [mAU] %
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type # [min] 	Area Percen : Signal : 1.0000 : 1.0000 Dilution Factor with Wavelength=220 nm : Width Area [min] mAU *s	mt Report th ISTDs [mAU] %
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type # [min] 	Area Perces : Signal : 1.0000 : 1.0000 Dilution Factor with Wavelength=220 nm e Width Area [min] mAU *s 0.6916 7.16663e4 1.3716 7.24803e4	mt Report th ISTDs Height Area [mAU] % -1
Sorted By Multiplier Dilution Use Multiplier 4 Signal 1: VWD1 A, Peak RetTime Type # [min] 	Area Perces : Signal : 1.0000 : 1.0000 Dilution Factor with Wavelength=220 nm e Width Area [min] mAU *s 0.6916 7.16663e4 1.3716 7.24803e4	mt Report th ISTDs [mAU] %

*** End of Report ***

Instrument 1 6/3/2011 4:31:10 PM THL

Page 1 of 1

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Data File D:\LC\LTL\DATE\LTL-11-19\LTL-11-19B 2011-06-03 15-22-09\072-0101.D Sample Name: LTL-11-19B

Acq. Operator	: LTL		Seq. Line :	1		
Acq. Instrument	: Instrument 1		Location :	Vial 72		
Injection Date	: 6/3/2011 3:23:3	2 PM	Inj :	1		
			Inj Volume :			
Acq. Method	: D:\LC\LTL\DATE\	LTL-11-19\LTL-1	1-19B 2011-06	-03 15-22-0	9\ADH-30-70-1ML-	
	220NM-40MIN.M					
	: 4/22/2011 4:36:					
Analysis Method	: D:\LC\LTL\DATE\		1-19B 2011-06	-03 15-22-0	9\072-0101.D\DA	.М (
	ADH-30-70-1ML-2					
Last changed	: 6/3/2011 4:30:0					
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	avelength=220 nm (DALCALTL	UATEVLTL-11-19/L(L-11	-198 2011-06-03 15-2,	2090/20101.0)		
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		Approximation in the second second				
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Multiplier						
Dilution	: 1.00					
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Signal 1: VWD1	A, Wavelength=220	nm				
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	0.7916 8.05794					
2 34.689 MM	1.3949 2980.48	35.61176	3.5669			
Totals :	8.35599	e4 1732.17902				
	*** 7	l of Report ***				
	2 2 2 P 1 H					

Instrument 1 6/3/2011 4:30:13 PM THL

Acq. Instrument : Instrum Injection Date : 1/20/20		Location : Vial 75 Inj : 1	
		Inj Volume : 5 µl	
Acq. Method : D:\LC\I 80MIN.M		-36A 2011-01-20 10-08-49\ADH-	30-70-1ML-220NM-
Last changed : 11/27/2	2010 11:06:07 AM by LTL		
Analysis Method : D:\LC\I	TL\DATE\LTL-9-36\LTL-9-	-361 2011-01-20 10-08-49\075-	UZUI.D\DA.M (
	-70-1ML-220NM-80MIN.M))11 5:02:35 PM by LTL		
(modifi	ied after loading)		
VIII/D1 A, Wavelength=220 n	m(DALCATIVDATELTL-936/LTL-936	6A,2011-01-20 10-08-49/075-0201.D)	54
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Sorted By :	Signal		
Multiplier :			
Dilution :	1.0000		
Use Multiplier & Dilution	n ractor wrth 15105		
Signal 1: VUD1 A, Wavele	ngth=220 nm		
Peak RetTime Type Width	Area Height	Area	
	mAU *s [mAU]	3	
	9 5.09878e4 1053.68652		
2 24.399 BB 0.935	0 5.08531e4 819.56921	49.9339	
Totals :	1.01841e5 1873.25574		
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Instrument 1 1/24/2011 5:02:39 PM LTL

Data File D:\LC\LQH\DATE\LQH-3-85-86\LQH-3-85-LTL-7-9 2011-01-24 11-31-41\099-0601.D Sample Name: LTL-9-36B

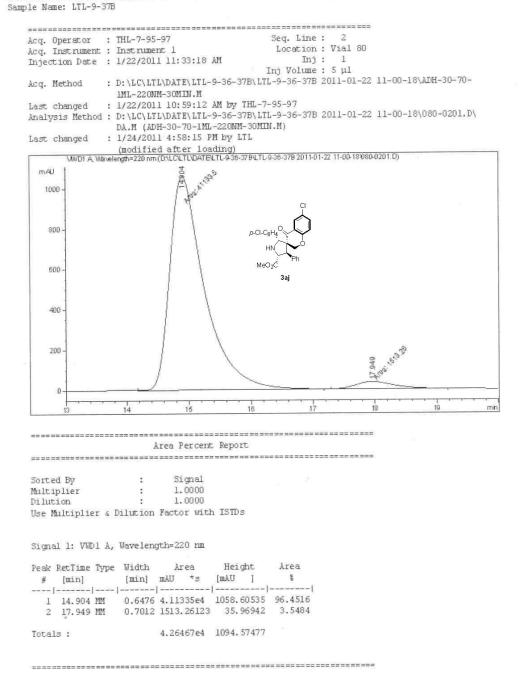
				= = = =		
Acq. Operator	: LTL		eq. Line : 6			
Acq. Instrument		1	Location : Vial	99		
Injection Date	: 1/24/2011 1:49:48 P	M	Inj: 1			
		In	j Volume : 5 µl			
Acq. Method	: D:\LC\LQH\DATE\LQH- 1ML-220NM-30MIN.M	3-85-86\LQH-3	-85-LTL-7-9 201	1-01-24 11-31-4	1\ADH-30-70	0-
Last changed	: 1/22/2011 10:59:12	AM how THL-7-9	5-97			
Instraig Method	: D: \LC\LQH\DATE\LQH-	3-85-86\LOH-3	-85-LTL-7-9 201	1-01-24 11-31-4	1\099-0601.	
MIGL 910 INCOLOG	D\DA.M (ADH-30-70-1					
Last changed	: 1/24/2011 3:13:10 F (modified after los	M by LTL				
V/0/01 A 105	velength=220 nm (DALCALQHADATE	NOH-3-85-864LQH-3-8	5-LTL-7-9 2011-01-24 1	-31-41\099-0601.D)		
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Signal 1: VWD1 .	A, Wavelength=220 nm					
		11-1-2	1			
Peak RetTime Typ		5 · · · ·	Area			
# [min]	[min] mAU *s		1			
1 15.555 BB						
2 21.279 BB	0.7821 1308.20422	24.89078	3.4346			
Totals :	3.80891e4	900.98990				

Instrument 1 1/24/2011 3:13:15 PM LTL

Data File D:\LC\LTL\DATE\LTL-9-37\LTL-9-37A 2011-01-20 10-58-08\076-0101.D Sample Name: LTL-9-37A Acq. Operator : THL Acq. Instrument : Instrument 1 Injection Date : 1/20/2011 10:59:55 AM Seq. Line : 1 Location : Vial 76 Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-9-37\LTL-9-37& 2011-01-20 10-58-08\ADH-30-70-1ML-220MM-Acq. Method SOMIN.M Last changed : 11/27/2010 11:06:07 AM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-9-37\LTL-9-37A 2011-01-20 10-58-08\076-0101.D\DA.M (ADH-30-70-1ML-220NM-80MIN.M) : 1/24/2011 5:04:29 PM by LTL Last changed (modified after loading) WVDIA Wavelength=220 nm(D\LCUTL\DATE\LTL-9-37\LTL-9-37A2011-01-20 10-58-08076-0101.D) 439 00 C 39 mAU .593 M A 88 00,00 350 p-CI-C₆H₄ 300 HN 250 MeO₂C racemic-3aj 200 150 100 50 0 17 18 10 mir 15 16 Area Percent Report Sorted By Signal : Multiplier : 1.0000 . 1,0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Peak RetTime Type Width Height Area Area [mAU] [min] mAU *s d'b # [min] --------1 15.120 MM 0.6737 1.58388e4 391.83405 50.3093 2 18.186 MM 0.7741 1.56441e4 336.82358 49.6907

Totals: 3.14829e4 728.65762

Instrument 1 1/24/2011 5:04:33 FM LTL



Data File D:\LC\LTL\DATE\LTL-9-36-378\LTL-9-36-378 2011-01-22 11-00-18\080-0201.D

Instrument 1 1/24/2011 4:58:18 PM LTL

Sample Name: LTL-9-40A

Acq. Operator : THL-7-95-97 Seq. Line : 1 Location : Vial 67 Acq. Instrument : Instrument 1 Injection Date : 1/21/2011 12:52:36 FM Inj : 1 Inj Volume : 5 µl : D:\LC\LTL\DATE\LTL-9-40\LTL-9-40A 2011-01-21 12-51-05\ADH-30-70-1ML-220MM-Acq. Method SOMIN. M Last changed : 11/27/2010 11:06:07 AM by LTL Analysis Method : D:\LC\LTL\DATE\LTL-9-40\LTL-9-40& 2011-01-21 12-51-05\067-0101.D\DA.M (ADH-30-70-1ML-220NM-80MIN.M) Last changed : 1/24/2011 5:05:26 PM by LTL (modified after loading) W/DIA Wavelength=220 nm(D/LCLTL/DATELTL-9-40/LTL-9-40A2011-01-21 12-51-05067-0101.D) in the second 12429 mAU 1200 1000 p-CI-Cel 800 MeO₂0 racemic-3ak 600 400 200 0 24 min 14 16 18 20 Area Percent Report Signal Sorted By : Multiplier 1.0000 : Dilution 1.0000 : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=220 nm Height Peak RetTime Type Width Area Area [mAU] 뒼 # [min] [min] mAU *s 1 12.429 MM 0.4706 3.54005e4 1253.84875 49.7170 2 22.911 MM 0.9849 3.58035e4 605.87762 50.2830 7.12040e4 1859.72638 Totals :

Data File D:\LC\LTL\DATE\LTL-9-40\LTL-9-40A 2011-01-21 12-51-05\067-0101.D

Instrument 1 1/24/2011 5:05:30 PM LTL

Sample Name: LTL-9-40B

Seq. Line : 3 Acq. Operator : THL-7-95-97 Location : Vial 79 Acq. Instrument : Instrument 1 Injection Date : 1/22/2011 12:04:54 PM Inj: 1 Inj Volume: 5 µl : D:\LC\LTL\DATE\LTL-9-36-37E\LTL-9-36-37E 2011-01-22 11-00-18\ADH-30-70-Acq. Method 1ML-220NM-30MIN.M Last changed : 1/22/2011 10:59:12 AM by THL-7-95-97 Analysis Method : D:\LC\LTL\DATE\LTL-9-36-37B\LTL-9-36-37B 2011-01-22 11-00-18\079-0301.D\ DA.M (ADH-30-70-1ML-220MM-30MIN.M) : 1/24/2011 4:58:58 PM by LTL Last changed (modified after loading) WWD1A Wavelergth=220 nm (DALCUTL/DATELTL-9-36-3784_TL-9-36-378 2011-01-22 11-00-18079-0301.D) ing. Banks mAU +0x.m 2000 1750 p-CI-CaH 1500 MeO₂C 1250 3ak 1000 750 500 22.693 250 0 12 14 16 Area Percent Report Signal Sorted By : Multiplier 1.0000 : : 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VUD1 A, Wavelength=220 nm Peak RetTime Type Width Area Height Area [mAU] 뷥 # [min] [min] mAU *s -1--1 -1--1-----0.5008 6.69852e4 2229.08105 97.4289 1 12.404 MM 2 22.693 MM 0.8975 1767.69250 32.82574 2.5711 6.87529e4 2261.90680 Totals :

Data File D:\LC\LTL\DATE\LTL-9-36-37B\LTL-9-36-37B 2011-01-22 11-00-18\079-0301.D

Instrument 1 1/24/2011 4:59:01 PM LTL