Electronic Supplementary Information for

Organocatalytic asymmetric desymmetrization: efficient construction of spirocyclic oxindoles bearing a unique all-carbon quaternary stereogenic center *via* sulfa-Michael addition

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I. General Remarks

¹H NMR spectra were recorded on a VARIAN Mercury 300 MHz or Bruker 400 MHz spectrometer in CDCl₃. 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 75 MHz or Bruker 100 MHz spectrometer in CDCl₃ or in DMSO-d₆. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm and with the internal hexadeuterodimethyl sulfoxide signal at 39.5 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Enantiomeric ratios were determined by HPLC, using a chiralpak AS-H column and chiralpak IC column with hexane and *i*-PrOH as solvents. Catalysts I-VI were prepared according to our previous report¹. The spiro cyclohexadienone oxindoles² were prepared according to the literature procedure. The racemic adducts were attained by using DABCO as the catalyst. The absolute configuration of 3aj was determined unequivocally according to the X-ray diffraction analysis of the derived oximes 4E, and those of other adducts were deduced on the basis of these results.

II. General Procedure for Asymmetric *sulfa*-Michael Addition of Thiols to Spiro Cyclohexadienone Oxindoles Catalyzed by Organocatalysts (I-d)

Under argon atmosphere, spiro cyclohexadienone oxindole (0.020 mmol) and the catalyst **I-d** (8.1 mg, 0.012 mmol) were dissolved in 0.5 mL CHCl₃. After the mixture was cooled to -20 °C, thiol (0.22 mmol) was added. The mixture was stirred at this temperature until the consumption of **1** (monitored by TLC analysis). Then, the solvent was removed and the residue was purified by flash chromatography on silica gel to give the corresponding product, which was then directly analyzed by HPLC to determine the enantiomeric excess.



(1*R*,6*R*)-1'-methyl-6-(phenylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4-dione: Yield (92%); Yellow solid; m.p. 47 °C; $[α]^{25}_D$ = +86 (*c* 0.23, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.44 (t, *J* = 7.8 Hz, 1H), 7.29-7.21 (m, 6H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 6.58 (d, *J* = 10.2 Hz, 1H), 6.25 (d, *J* = 10.2 Hz, 1H), 4.22-4.15 (m, 1H), 3.28 (s, 3H), 3.02-2.98 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.6, 176.1, 146.3, 144.3, 133.3, 132.3, 130.1, 129.0, 128.1, 125.3, 125.0, 123.0, 108.9, 54.8, 49.9, 42.0, 26.7; IR (KBr) v 3068, 3017, 1675, 1568, 1315, 1128, 819 765, 696; HRMS Calcd. For C₂₀H₁₇NO₂S + H⁺: 336.1061, found: 336.1053. The product was analyzed by HPLC to determine the enantiomeric excess: 84% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 230 nm); t_r = 9.97 and 13.20 min.



(1*R*,6*R*)-1'-methyl-6-(o-tolylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4-dione: Yield (85%); Light yellow solid; m.p. 53 °C; $[\alpha]^{25}_{D} = +105.7$ (*c* 0.49, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.44 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 7.5 Hz, 1H), 7.28 (d, *J* = 7.5 Hz, 1H), 7.16-7.11 (m, 4H), 6.96 (d, *J* = 7.8 Hz, 1H), 6.55 (d, *J* = 9.9 Hz, 1H), 6.23 (d, *J* = 9.9 Hz, 1H), 4.11 (dd, *J*₁ = 4.8 Hz and *J*₂ = 13.2 Hz, 1H), 3.23 (s, 3H), 3.10-2.90 (m, 2H), 2.30 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.5, 175.9, 146.4, 144.1, 141.1, 130.4, 130.0, 128.5, 126.4, 125.3, 125.0, 123.0, 108.9, 54.6, 48.9, 41.8, 26.5, 20.8; IR (KBr) v 3030, 2945, 1608, 1473, 1428, 1337, 1129, 818, 756, 699; HRMS Calcd. For C₂₁H₁₉NO₂S + H⁺: 350.1218, found: 350.1209. The product was analyzed by HPLC to determine the enantiomeric excess: 83% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 9.48 and 14.13 min.



(1*R*,6*R*)-1'-methyl-6-(p-tolylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4-dione: Yield (85%); Light yellow solid; m.p. 72 °C; $[\alpha]^{25}_{D}$ = +113.4 (*c* 0.52, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.43 (t, *J* = 7.8 Hz, 1H), 7.22-7.16 (m, 3H), 7.11 (d, *J* = 7.5 Hz, 1H), 7.04 (d, *J* = 7.8 Hz, 2H), 6.97 (d, *J* = 7.8 Hz, 1H), 6.56 (d, *J* = 10.2 Hz, 1H), 6.23 (d, *J* = 10.2 Hz, 1H), 4.14-4.10 (m, 1H), 3.28 (s, 3H), 2.99-2.95 (m, 2H), 2.29 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.5, 176.0, 146.2, 144.1, 138.2, 133.7, 130.0, 129.9, 129.6, 128.4, 125.2, 124.9, 122.8, 108.8, 54.7, 50.0, 41.7, 26.5, 21.0; IR (KBr) v 3068, 3013, 1702, 1601, 1319, 1120, 825, 778, 712; HRMS Calcd. For C₂₁H₁₉NO₂S + H⁺: 350.1218, found: 350.1209. The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 10.81 and 17.16 min.



(1*R*,6*R*)-1'-methyl-6-(m-tolylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4-dione: Yield (83%); White solid; m.p. 126 °C; $[\alpha]^{25}_{D} = +109.7$ (*c* 0.55, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.43 (t, *J* = 7.2 Hz, 1H), 7.21-7.10 (m, 6H), 6.97 (d, *J* = 7.8 Hz, 1H), 6.56 (d, *J* = 9.9 Hz, 1H), 6.23 (d, *J* = 9.9 Hz, 1H), 4.17 (t, *J* = 8.1 Hz, 1H), 3.27 (s, 3H), 2.99-2.96 (m, 2H), 2.27 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.6, 176.0, 146.2, 144.2, 138.7, 133.8, 131.6, 130.0, 129.9, 128.9, 128.7, 125.2, 125.0, 122.9, 108.8, 107.1, 54.7, 49.5, 41.8, 26.6, 21.1; IR (KBr) v 3072, 3031, 1698, 1574, 1332, 1173, 809, 747, 687; HRMS Calcd. For C₂₁H₁₉NO₂S + H⁺: 350.1219, found: 350.1209. The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 10.17 and 14.56 min.





Yield (77%); White solid; m.p. 51 °C; $[\alpha]^{25}_{D}$ = +132.4 (*c* 0.54, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.44 (t, *J* = 7.8 Hz, 1H), 7.27-7.21 (m, 3H), 7.14-6.96 (m, 4H), 6.56 (d, *J* = 9.9 Hz, 1H), 6.24 (d, *J* = 9.9 Hz, 1H), 4.21 (t, *J* = 9.3 Hz, 1H), 3.25 (s, 3H), 3.03 (d, *J* = 9.6 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 196.3, 175.7, 162.3 (d, *J* = 24.6 Hz), 146.3, 144.2, 135.8, 132.4, 130.6 (d, *J* = 8.0 Hz), 127.8, 125.0, 124.5(d, *J* = 3.9 Hz), 123.2, 123.0, 119.4, 119.2, 160.0 (d, *J* = 22.9 Hz), 108.9, 54.8, 49.1, 41.8, 26.6; IR (KBr) v 3032, 3013, 1701, 1568, 1437, 1255, 1214, 1108, 818, 754, 699; HRMS Calcd. For C₂₀H₁₆NO₂FS + H⁺: 354.0970, found: 354.0958. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 10.54 and 21.55 min.



(1*R*,6*R*)-6-((4-fluorophenyl)thio)-1'-methylspiro[cyclohex[2]ene-1,3'-indoline]-2', 4-dione:

Yield (85%); White solid; m.p. 42 °C; $[α]^{25}_{D}$ = +115.5 (*c* 0.54, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.44 (t, *J* = 7.5 Hz, 1H), 7.27-7.19 (m, 3H), 7.13-7.08 (m, 1H), 7.00-6.91 (m, 3H), 6.57 (d, *J* = 9.9 Hz, 1H), 6.24 (d, *J* = 9.9 Hz, 1H), 4.09 (dd, *J*₁ = 6.9 Hz and *J*₂ = 11.7 Hz, 1H), 3.30 (m, 3H), 3.00-2.95 (m, 2H); ¹³C NMR (CDCl₃, TMS, 125 MHz) δ 196.3, 176.1, 162.8 (d, *J* = 247.6 Hz), 146.2, 144.2, 136.0 (d, *J* = 8.5 Hz), 130.1, 127.2 (d, *J* = 2.6 Hz), 125.2, 125.0, 116.1 (d, *J* = 21.9 Hz), 109.0, 54.8, 50.4, 41.8, 26.6; IR (KBr) v 3057, 3007, 1687, 1591, 1487, 1136, 1074, 827, 765, 694; HRMS Calcd. For C₂₀H₁₆NO₂FS + H⁺: 354.0968, found: 354.0958. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 9.04 and 12.45 min.



(1*R*,6*R*)-6-((3-fluorophenyl)thio)-1'-methylspiro[cyclohex[2]ene-1,3'-indoline]-2', 4-dione:

Yield (84%); Yellow solid; m.p. 46 °C; $[\alpha]^{25}_{D}$ = +112 (*c* 0.21, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.45 (t, *J* = 7.8 Hz, 1H), 7.23-7.20 (m, 2H), 7.14-7.09 (m, 2H), 7.00-6.92 (m, 3H), 6.59 (d, *J* = 9.9 Hz, 1H), 6.26 (d, *J* = 10.2 Hz, 1H), 4.25-4.19 (m, 1H), 3.28 (s, 3H), 3.03-2.99 (m, 2H); ¹³C NMR (CDCl₃, TMS, 125 MHz) δ 196.2, 176.0, 162.4 (d, *J* = 248.1 Hz), 146.2, 144.3, 134.5, 134.4, 130.2, 128.5, 125.1, 125.0,

123.1, 119.7 (d, J = 21.9 Hz), 115.2 (d, J = 20.6 Hz), 109.0, 54.7, 49.7, 41.9, 26.7; IR (KBr) v 3046, 2981, 1693, 1582, 1502, 1227, 1216, 1113, 830, 767, 685; HRMS Calcd. For C₂₀H₁₆NO₂FS + H⁺: 354.0966, found: 354.0958. The product was analyzed by HPLC to determine the enantiomeric excess: 85% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, $\lambda = 254$ nm); t_r = 9.82 and 11.53 min.





Yield (95%); White solid; m.p. 51 °C; $[α]^{25}_{D}$ = +140.3 (*c* 0.80, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.45 (t, *J* = 7.8 Hz, 1H), 7.22 (m, 5H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 8.7 Hz, 1H), 6.58 (d, *J* = 9.9 Hz, 1H), 6.25 (d, *J* = 9.9 Hz, 1H), 4.14 (dd, *J*₁ = 6.6 Hz and *J*₂ = 12.3 Hz, 1H), 3.28 (s, 3H), 3.00-2.96 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.2, 176.0, 146.2, 144.2, 134.7, 134.4, 130.7, 130.1, 129.2, 125.2, 125.1, 123.1, 109.0, 54.8, 50.0, 41.8, 26.7; IR (KBr) v 3048, 2940, 1617, 1484, 1472, 1254, 1103, 1054, 823, 751, 695; HRMS Calcd. For C₂₀H₁₆NO₂SCl + H⁺: 370.0670, found: 370.0670. The product was analyzed by HPLC to determine the enantiomeric excess: 86% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 10.78 and 14.50 min.



(1*R*,6*R*)-1'-methyl-6-(naphthalen-1-ylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4 -dione: Yield (86%); White solid; m.p. 70 °C; $[\alpha]^{25}_{D} = +87.1$ (*c* 0.77, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.23 (d, *J* = 8.1 Hz, 1H), 7.81-7.79 (m, 2H), 7.63 (d, *J* = 6.9 Hz, 1H), 7.55-7.25 (m, 6H), 7.18-7.13 (m, 1H), 6.93 (d, *J* = 7.8Hz, 1H), 6.55 (d, *J* = 9.9 Hz, 1H), 6.21 (d, *J* = 9.9 Hz, 1H), 4.26 (dd, *J*₁ = 4.8 Hz and *J*₂ = 13.5 Hz, 1H), 3.16 (s, 3H), 3.06-2.85 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.3, 175.8, 146.4, 144.1, 134.2 134.1, 133.9, 129.9, 129.6, 129.0, 128.4, 126.8, 126.2, 125.5, 125.4, 125.3, 125.0, 122.9, 109.0, 54.6, 49.5, 41.6, 26.4; IR (KBr) v 3057, 3016, 1698, 1591, 1482, 1351, 1137, 817, 750, 685; HRMS Calcd. For C₂₄H₁₉NO₂S + H⁺: 386.1218, found: 386.1209. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, $\lambda = 254$ nm); t_r = 11.73 and 17.38 min.



(1*R*,6*R*)-1'-methyl-6-(naphthalen-2-ylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4 -dione:

Yield (89%); White solid; m.p. 61 °C; $[\alpha]^{25}_{D}$ = +152.9 (*c* 0.77, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.79-7.72 (m, 4H), 7.49-7.47 (m, 3H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.26-7.22 (m, 1H), 7.15-7.10 (m, 1H), 6.99 (d, *J* = 6.9 Hz, 1H), 6.59 (d, *J* = 9.9 Hz, 1H), 6.25 (d, *J* = 9.9 Hz, 1H), 4.32 (t, *J* = 9.6 Hz, 1H) 3.25 (s, 3H), 3.04-3.01 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.4, 176.1, 146.3, 144.2, 133.2, 132.6, 130.2, 130.1, 130.0, 129.2, 128.7, 127.6, 127.5, 126.6, 125.3, 125.1, 123.0, 109.0, 54.8, 49.3, 41.8, 26.6; IR (KBr) v 3054, 3018, 1710, 1580, 1472, 1352, 1140, 823, 756, 691; HRMS Calcd. For C₂₄H₁₉NO₂S + H⁺: 386.1217, found: 386.1209. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 13.66 and 18.92 min.



(3ak)

(1*R*,6*R*)-1'-methyl-6-(thiophen-2-ylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4-d ione:

Yield (83%); Yellow solid; m.p. 53 °C; $[\alpha]^{25}_{D} = +99$ (*c* 0.18, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.44 (t, J = 7.5 Hz, 1H), 7.33 (d, J = 4.5 Hz, 1H), 7.18-7.06 (m, 2H), 6.99 (d, J = 7.8 Hz, 1H), 6.90-6.86 (m, 2H), 6.56 (d, J = 9.9 Hz, 1H), 6.24 (d, J = 9.9 Hz, 1H), 4.02 (dd, $J_{I} = 5.7$ Hz and $J_{2} = 12.6$ Hz, 1H), 3.34 (s, 3H), 3.10-2.92 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.4, 175.9, 146.1, 144.3, 136.1, 131.1, 130.1, 129.6, 127.6, 125.2, 124.9, 123.0, 109.0, 54.6, 51.6, 41.3, 26.7; IR (KBr) v 3034, 3087, 1614, 1527, 1432, 1325, 1134, 864, 826, 743, 690; HRMS Calcd. For C₁₈H₁₅NO₂S₂ + H⁺: 342.0624, found: 342.0617. The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, $\lambda = 254$ nm); t_r = 12.98 and 18.15 min.



(1*R*,6*R*)-5'-methoxy-1'-methyl-6-(naphthalen-2-ylthio)spiro[cyclohex[2]ene-1,3'-i ndoline]-2',4-dione:

Yield (85%); White solid; m.p. 86 °C; $[\alpha]^{25}{}_{D} = +201.9$ (*c* 0.84, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.79-7.72 (m, 4H), 7.49-7.46 (m, 2H), 7.39-7.36 (m, 1H), 6.97-6.88 (m, 2H), 6.79 (s, 1H), 6.59 (d, *J* = 9.6 Hz, 1H), 6.25 (d, *J* = 9.9 Hz, 1H), 4.30 (t, *J* = 9.3 Hz, 1H), 3.75 (s, 3H), 3.22 (s, 3H), 3.01 (d, *J* = 10.2 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.3, 175.5, 155.9, 146.3, 137.5, 133.1, 132.5,

130.2, 129.9, 129.1, 128.5, 127.5, 127.4, 126.4, 113.8, 112.6, 109.1, 55.6, 55.1, 49.0, 41.6, 26.6; IR (KBr) v 3064, 3012, 1702, 1611, 1452, 1375, 1127, 1070, 820, 743, 690; HRMS Calcd. For $C_{25}H_{21}NO_3S + H^+$: 416.1325, found: 416.1315. The product was analyzed by HPLC to determine the enantiomeric excess: 84% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 17.12 and 25.34 min.



(1*R*,6*R*)-5'-chloro-1'-methyl-6-(naphthalen-2-ylthio)spiro[cyclohex[2]ene-1,3'-ind oline]-2',4-dione:

Yield (80%); White solid; m.p. 98 °C; $[α]^{25}_{D}$ = +223.7 (*c* 0.66, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.81-7.71 (m, 4H), 7.50-7.47 (m, 2H), 7.44-7.41 (m, 1H), 7.38-7.35 (m, 1H), 7.19-7.18 (m, 1H), 6.91 (d, *J* = 8.1 Hz, 1H), 6.55 (d, *J* = 9.9 Hz, 1H), 6.27 (d, *J* = 9.9 Hz, 1H), 4.32-4.26 (m, 1H), 3.21 (s, 3H), 3.02-2.98 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 195.9, 175.6, 145.3, 142.8, 133.2, 132.6, 130.5, 130.1, 129.9, 128.7, 128.3, 127.6, 127.4, 127.0, 126.6, 125.5, 109.8, 54.9, 49.0, 41.6, 26.7; IR (KBr) v 3030, 3014, 1698, 1589, 1491, 1472, 1324, 1132, 1084, 817, 745, 696; HRMS Calcd. For C₂₄H₁₈NO₂SCl + Na⁺: 442.0647, found: 442.0639. The product was analyzed by HPLC to determine the enantiomeric excess: 84% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 12.54 and 17.21 min.



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(1*R*,6*R*)-1',5'-dimethyl-6-(naphthalen-2-ylthio)spiro[cyclohex[2]ene-1,3'-indoline] -2',4-dione:

Yield (81%); White solid; m.p. 72 °C; $[α]^{25}_{D}$ = +191.6 (*c* 0.43, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.79-7.71 (m, 4H), 7.48-7.47 (m, 2H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.26-7.23 (m, 1H), 6.99 (s, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 6.58 (d, *J* = 9.9 Hz, 1H), 6.24 (d, *J* = 9.9 Hz, 1H), 4.33-4.27 (m, 1H), 3.24 (s, 3H), 3.05-3.01 (m, 2H), 2.30 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.6, 176.0, 146.6, 141.8, 133.2, 132.6, 130.3, 130.0, 129.1, 128.5, 127.5, 127.4, 126.5, 125.8, 125.1, 108.7, 54.9, 49.1, 41.7, 26.6, 21.0; IR (KBr) v 3043, 1702, 1596, 1483, 1459, 1231, 1145, 821, 752, 689; HRMS Calcd. For C₂₅H₂₁NO₂S + H⁺: 400.1373, found: 400.1366. The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 14.25 and 21.62 min.



(1*R*,6*R*)-1'-benzyl-6-(naphthalen-2-ylthio)spiro[cyclohex[2]ene-1,3'-indoline]-2',4 -dione:

Yield (82%); White solid; m.p. 75 °C; $[α]^{25}_{D}$ = +177.2 (*c* 0.89, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.79-7.72 (m, 4H), 7.49-7.38 (m, 5H), 7.30-7.23 (m, 3H), 7.06 (t, *J* = 7.2 Hz, 1H), 6.86 (d, *J* = 7.2 Hz, 1H), 6.64 (d, *J* = 9.9 Hz, 1H), 6.27 (d, *J* = 10.2 Hz, 1H), 5.15 (d, *J* = 15.9 Hz, 1H), 4.85 (d, *J* = 15.9 Hz, 1H), 4.36 (dd, *J*₁ = 5.4 Hz, *J*₂ = 12.6 Hz 1H), 3.16-3.07 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 196.4, 176.4, 146.2, 143.3, 135.0, 133.3, 132.5, 130.1, 130.0, 129.3, 128.7, 127.7, 127.6, 127.5, 127.3, 126.5, 125.3, 125.1, 123.0, 110.1, 54.8, 49.3, 44.3, 42.1; IR (KBr) v 3074, 3021, 1715, 1587, 1485, 1457, 1252, 1234, 814, 748, 685; HRMS Calcd. For C₃₀H₂₃NO₂S + H⁺: 462.1527, found: 462.1522. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 11.16 and 14.19 min.



Under argon atmosphere, **3aj** (77 mg, 0.2 mmol, 88% ee) and NaHCO₃ were dissolved in 2 mL C₂H₅OH, and hydroxylammonium chloride (16.8 mg, 0.24 mmol) was added, the mixture was refluxed for 2 h. Then, the solvent was evaporated and the residue was purified by column chromatography followed by recrystallization (in CH₃OH/DCM) to give (**1***R*,**6***R*)-**4**E in 36% yield, which was then directly analyzed by HPLC to determine the enantiomeric excess.

(1*R*,6*R*,*E*)-4-(hydroxyimino)-1'-methyl-6-(naphthalen-2-ylthio)spiro[cyclohex[2]e ne-1,3'-indolin]-2'-one: White solid; m.p. 193 °C; $[\alpha]^{25}_{D}$ = +103.1 (*c* 0.37, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.78-7.72 (m, 4H), 7.45-7.42 (m, 4H), 7.18-7.10 (m, 2H), 6.97-6.94 (m, 1H), 6.41 (d, *J* = 9.6 Hz, 1H), 5.92 (d, *J* = 9. Hz, 1H), 4.06-4.02 (m, 1H), 3.68-3.62 (m, 1H), 3.24 (s, 3H), 2.79 (t, *J* = 16.5Hz, 1H); ¹³C NMR (DMSO-*d*₆, TMS, 75 MHz) δ 176.4, 151.6, 144.0, 133.1, 131.9, 131.7, 130.7, 130.0, 129.3, 129.0, 128.6, 127.6, 127.5, 127.3, 126.8, 126.4, 126.1, 124.7, 122.6, 109.0, 54.1, 48.2, 27.1, 26.4; IR (KBr) v 3407, 3034, 2935, 1701, 1684, 1611, 1482, 1452, 1341, 1129, 813, 742, 685; The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 17.50 and 25.10 min.

III. Synthetic Application of the Michael Adduct 3aj.



3aj, 88%ee

5, 92% yield, >20:1 dr, 88%ee

To a solution of **3aj** (77 mg, 0.2 mmol, 88% ee) in 2 mL CH₃OH was added $Pd(OH)_2/C$ (7.7 mg, 10% w/w). The mixture was stirred under H₂ (30 atm) at room temperature for 24 h. Then, the solvent was evaporated and the residue was purified by column chromatography to give **5** in 92% yield, which was then directly analyzed by HPLC to determine the enantiomeric excess.

(1*R*,2*R*)-1'-methyl-2-(naphthalen-2-ylthio)spiro[cyclohexane-1,3'-indoline]-2',4-di one: White solid; m.p. 61 °C; $[α]^{25}_{D}$ = +43.7 (*c* 0.35, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.79-7.69 (m, 4H), 7.49-7.43 (m, 4H), 7.37 (d, *J* = 8.7 Hz, 1H), 7.13 (t, *J* = 7.8 Hz, 1H), 7.02 (d, *J* = 7.8 Hz, 1H), 3.97 (dd, *J*₁ = 6.9 Hz, *J*₂ = 11.4 Hz 1H), 3.27 (s, 3H), 3.01-2.97 (m, 2H), 2.85-2.74 (m, 1H), 2.61-2.56 (m, 1H), 2.43-2.32 (m, 1H), 2.01-1.95 (m, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 206.9, 177.7, 144.0, 133.3, 132.6, 130.3, 129.6, 129.2, 128.9, 128.6, 127.6, 127.5, 126.5, 124.6, 122.5, 108.9, 51.5, 51.3, 45.7, 36.8, 32.8, 26.6; IR (KBr) v 3057, 1721, 1603, 1583, 1317, 1115, 819, 736, 678; HRMS Calcd. For C₂₄H₂₁NO₂S + H⁺: 388.1379, found: 388.1366. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 254 nm); t_r = 13.93 and 21.18 min.



3aj, 88%ee

6, 99% yield, 88%ee

To a solution of 3aj (77 mg, 0.2 mmol, 88% ee) in 2 mL MeOH was added NaBH₄ (8.4 mg, 1.1 eq.), the mixture was stirred at this temperature for 1 h. Then, the solvent was evaporated and the residue was purified by column chromatography to give **6** in 99% yield, which was then directly analyzed by HPLC to determine the enantiomeric excess.

(1*R*,4*S*,6*R*)-4-hydroxy-1'-methyl-6-(naphthalen-2-ylthio)spiro[cyclohex[2]ene-1,3' -indolin]-2'-one: White solid; m.p. 78 °C; $[α]^{25}_{D} = +95.1$ (*c* 0.84, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.74-7.71 (m, 4H), 7.48-7.39 (m, 5H), 7.14 (t, *J* = 7.5 Hz, 1H), 6.90 (d, *J* = 7.2 Hz, 1H), 6.05 (d, *J* = 9.9 Hz, 1H), 5.37 (d, *J* = 9.9 Hz, 1H), 4.49 (s, 1H), 3.93 (d, *J* = 12.6 Hz, 1H), 3.17 (s, 3H), 2.58-2.53 (m, 1H), 2.16 (q, *J* = 13.2 Hz, 1H), 1.93 (s, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 177.9, 143.5, 134.4, 133.1, 132.2, 131.4, 130.6, 129.9, 128.9, 128.8, 128.3, 127.4, 127.2, 126.3, 126.1, 125.9, 125.3, 122.6, 108.2, 66.2, 54.3, 48.8, 36.3, 26.3; IR (KBr) v 3407, 3052, 2930, 1695, 1609, 1491, 1469, 1351, 1132, 816, 747, 695; HRMS Calcd. For C₂₄H₂₁NO₂S + Na⁺: 410.1192, found: 410.11852. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 254$ nm); t_r = 32.63 and 42.00 min.





3aj, 88%ee

7, 81% yield, 88%ee

Under argon atmosphere, **3aj** (77.1 mg, 0.2 mmol, 88% ee) and K_2CO_3 (2.7 mg, 0.02 mmol) were dissolved in 2 mL DCM, and 4-Chlorothiophenol (31.8 mg, 0.22 mmol) was added, the mixture was stirred at room temperature overnight. Then, the solvent was evaporated and the residue was purified by column chromatography to give 7 in 81% yield, which was then directly analyzed by HPLC to determine the enantiomeric excess.

(1*R*,2*R*,6*R*)-2-((4-chlorophenyl)thio)-1'-methyl-6-(naphthalen-2-ylthio)spiro[cyclo hexane-1,3'-indoline]-2',4-dione: White solid; m.p. 83 °C; $[α]^{25}_{D}$ = +10.5 (*c* 0.44, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.76-7.69 (m, 4H), 7.48-7.34 (m, 4H), 7.26-7.24 (m, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 6.96-6.91 (m, 4H), 4.02 (dd, *J_I* = 5.1 Hz and *J₂* = 12.6 Hz, 1H), 3.85 (t, *J* = 5.1 Hz, 1H), 3.57 (dd, *J_I* = 4.2 Hz and *J₂* = 15.6 Hz, 1H), 3.46-3.36 (m, 1H), 3.28 (s, 3H), 2.88 (dd, *J_I* = 4.5 Hz and *J₂* = 15.9 Hz, 1H), 2.63 (dd, *J_I* = 5.1 Hz and *J₂* = 15.9 Hz, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 205.9, 175.3, 143.9, 134.8, 134.3, 133.4, 132.8, 132.6, 131.7, 130.0, 129.2, 129.0, 128.9, 128.5, 127.6, 127.5, 126.7, 126.5, 122.2, 108.2, 54.8, 51.1, 50.5, 44.3, 42.1, 26.4; IR (KBr) v 3046, 1711, 1618, 1482, 1347, 1123, 827, 745, 697; HRMS Calcd. For C₃₀H₂₄NO₂S₂Cl + Na⁺: 552.0842, found: 552.0829. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 254 nm); t_r = 17.50 and 25.10 min.



3ah, 86%ee

epi-7, 85% yield, 86%ee

Under argon atmosphere, **3ah** (73.8 mg, 0.2 mmol, 88% ee) and K₂CO₃ (2.7 mg, 0.02 mmol) were dissolved in 2 mL DCM, and 4-Chlorothiophenol (31.8 mg, 0.22 mmol) was added, the mixture was stirred at room temperature overnight. Then, the solvent was evaporated and the residue was purified by column chromatography to give epi-7 in 85% yield, which was then directly analyzed by HPLC to determine the enantiomeric excess.

(1S,2R,6R)-2-((4-chlorophenyl)thio)-1'-methyl-6-(naphthalen-2-ylthio)spiro[cyclo hexane-1,3'-indoline]-2',4-dione: White solid; m.p. 81 °C; $[\alpha]^{25}_{D} = +11.2$ (c 0.37, CHCl₃): ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.77-7.75 (m, 1H), 7.65-7.62 (m, 2H), 7.48-7.43 (m, 3H), 7.32-7.26 (m, 2H), 7.20-7.14 (m, 5H), 6.94 (d, J = 7.5 Hz, 1H), 6.79-6.75 (m, 1H), 4.08-4.04 (m, 1H), 3.74 (m, 1H), 3.59-3.40 (m, 2H), 3.34 (s, 3H), 2.95- 2.90 (m, 1H), 2.60-2.54 (m, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 206.1, 134.8, 133.2, 132.6, 130.3, 129.4, 128.6, 127.6, 126.6, 126.4, 122.1, 108.3, 51.5, 50.0, 44.5, 42.1, 26.5, 14.2; IR (KBr) v 3061, 2935, 1715, 1514, 1472, 1341, 1126, 817, 750, 691; The product was analyzed by HPLC to determine the enantiomeric excess: 86% ee (Chiralpak IC, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 254$ nm); $t_r = 19.78$ and 32.49 min.





8,70% yield, 88%ee

Under argon atmosphere, **3aj** (77 mg, 0.2 mmol, 88% ee) and DABCO (2.2 mg, 0.02 mmol) were dissolved in 2 mL DCM, and 2-Naphthalenethiol (35.3 mg, 0.22 mmol) was added, the mixture was stirred at room temperature overnight. Then, the solvent was evaporated and the residue was purified by column chromatography to give **8** in 70% yield, which was then directly analyzed by HPLC to determine the enantiomeric excess. This control experiment demonstrates that the two substituted groups at 2,6-position in the cyclohexanone are in *trans* configuration, otherwise *meso*-compound should be generated.

(2*R*,6*R*)-1'-methyl-2,6-bis(naphthalen-2-ylthio)spiro[cyclohexane-1,3'-indoline]-2 ',4-dione: White solid; mp 79 °C; $[α]^{25}_{D} = +10$ (*c* 0.27, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.76-7.64 (m, 6H), 7.46-7.27 (m, 9H), 7.17 (d, *J* = 8.1 Hz, 1H), 6.95 (d, *J* = 6.6 Hz, 1H), 6.80 (t, *J* = 7.2 Hz, 1H), 4.15-4.11 (m, 1H), 3.89 (m, 1H), 3.61-3.44 (m, 2H), 3.32 (s, 3H), 2.97-2.92 (m, 1H), 2.67-2.61 (m, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 206.3, 175.5, 144.0, 133.1, 132.7, 132.5, 130.3, 130.0, 129.3, 129.1, 128.8, 128.5, 127.6, 126.6, 126.4, 122.1, 108.2, 54.8, 51.1, 50.0, 44.6, 42.1, 26.4; IR (KBr) v 3057, 1696, 1607, 1484, 1312, 1023, 857, 762, 662; HRMS Calcd. For C₃₄H₂₇NO₂S₂ + Na⁺: 568.1370, found: 568.1375. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralpak IC, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 254 nm); t_r = 22.00 and 38.87 min.

IV. X-ray Crystal Structures of (1R,6R)-4E



Crystal data for (1*R*,6*R*)-4E: C₂₄H₂₀N₂O₂S, $M_r = 400.48$, T = 296 K, Orthorhombic, space group $P2_12_12_1$, a = 8.8678(9), b = 9.3161(9), c = 24.972(3) Å, V = 2063.0(4) Å³, Z = 4, 4277 reflections measured, 3493 unique ($R_{int} = 0.0363$) which were used in all caclculations. The final $wR_2 = 0.0851$ (all data), Flack $\chi = 0.02(7)$. CCDC 932106 contains the supplementary crystallographic data, which 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).

V. Proposed transition-state model for our catalytic system



VI. Reference

- [1] a) T. Okino, Y. Hoashi, Y. Takemoto, J. Am. Chem. Soc. 2003, 125, 12672; b) B. Vakulya, S. Varga, A. Csampai, T. Soos, Org. Lett. 2005, 7, 1967; c) T. Marcelli, J. H. van Maarseveen, H. Hiemstra, Angew. Chem. Int. Ed. 2006, 45, 7496; d) S. J. Connon, Chem. Commun. 2008, 2499; e) C. J. Wang, Z. H. Zhang, X. Q. Dong, X. J. Wu, Chem. Commun. 2008, 0, 1431.
- [2] a) G. O'Mahony, A. K. Pitts, Org. Lett. 2010, 12, 2024; b) W. Yu, Z. Yu, X. Ju, J.
 Wang, Synthesis 2011, 2011, 860.

VII. ¹H NMR and ¹³C NMR Spectra
































































50





















VIII. HPLC Chromatograms

Data File D:\LC\DATA\YL\YL-2-58B-S\YL-2-58B-S 2012-05-23 16-55-05\022-0101.D Sample Wame: YL-2-58B-S

Acq. Operator :	THL Seq. Line : 1
Acq. Instrument :	Instrument 1 Location : Vial 22
Injection Date :	5/23/2012 4:57:07 PM Inj: 1
	Inj Volume : 5 µl
Acq. Method :	D:\LC\DATA\YL\YL-2-58B-S\YL-2-58B-S 2012-05-23 16-55-05\ASH-50-50-1ML-
	230 NM.M
Last changed :	5/23/2012 4:54:08 PM by THL
Analysis Method :	D:\LC\DATA\YL-YL-2-58B-S\YL-2-58B-S 2012-05-23 16-55-05\022-0101.D\DA.M (
	ASH-50-50-1ML-230MM.M)
Last changed :	1/30/2013 9:56:22 AM by FX
	(modified after loading)
VM/D1 A, Wave	length=230 nm (DALC/DATAXYLYL-2-58B-SYYL-2-58B-S 2012-05-23 16-55-05'022-0101.D)
mAU -	a O
	a 1/
250	
] 330]	
300 -	
250	
2007	
200-	
150	
100-	
1 007	
0-	
8	9 10 11 12 13 14 15 16 mm
	Area Percent Report
Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000
Use Multiplier s	Dilution Factor with ISTDs
Signal 1: VWD1 A,	Wavelength=230 nm
Peak RetTime Type	Width Area Height Area
# [min]	[min] mAU *s [mAU] %
1 10.236 VB	0.5690 1.46745e4 388.35678 50.3719
2 13.384 BB	0.8274 1.44578e4 262.54214 49.6281
Totals :	2.91323e4 650.89893

Instrument 1 1/30/2013 9:56:43 AM FX

Data File D:\LC\DATA\YL\YL-3-43\YL-3-43 2012-09-25 20-22-38\011-0101.D Sample Name: YL-3-43 Acq. Operator : YL Seq. Line : 1 Location : Vial 11 Acq. Instrument : Instrument 1 Injection Date : 9/25/2012 8:23:52 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-43\YL-3-43 2012-09-25 20-22-38\ASH-50-50-1ML-230NM-Acq. Method 20MIN.M Last changed : 5/23/2012 8:48:56 PM by THL Analysis Method : D:\LC\DATA\YL\YL-3-43\YL-3-43 2012-09-25 20-22-38\011-0101.D\DA.M (ASH-50-50-1ML-230NM-20MIN.M) Last changed : 1/29/2013 7:57:57 PM by FX (modified after loading) W/DIA, Wavelength=230 nm (D\LC\DATA\YL\YL-343\YL-3-43 2012-09-25 20-22-38\011-0101.D) mAU 1000 (3aa) 800 O 600 400 13 2 0 3 200 ٥ 14 10 16 12 т'n Area Percent Report _____ Sorted By Signal : : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=230 nm Peak RetTime Type Width Area Height Area [min] mAU *s [mAU] # [min] 5 1 9.973 BB 0.5938 4.46753e4 1140.58459 91.8316 2 13.203 BB 0.7807 3973.87573 74.18596 8.1684 Totals : 4.86492e4 1214.77055

**** End of Report ***

Instrument 1 1/29/2013 7:58:46 PM FX

Sample Name: YL-3-47-oCH3 Acq. Operator : YL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/5/2012 8:26:39 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-0CH3\YL-3-47-0CH3 2012-10-05 20-25-14\ICH-50-50ML-Acq. Method 254NM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-0CH3\YL-3-47-0CH3 2012-10-05 20-25-14\001-0101.D\DA. M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 9:10:01 PM by FX (modified after loading) W/D1A Wavelength=264nm(DXLCVDATAXYLVL347-0CH3VL-347-0CH32012-10-0520-25-14001-0101.D) mAU 99 O 120 (3ab) 100 6 LO 🕯 റ 80 -60 40 20 -۵ 10 12 14 16 min Area Percent Report _____ Sorted By . Simul ; Multiplier 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area 1 9.456 BB 0.2894 2577.26636 136.04590 51.6613 2 14.079 BB 0.4259 2411.50513 87.61938 48.3387 4988.77148 223.66528 Totals : _____

Data File D:\LC\DATA\YL\YL-3-47-0CH3\YL-3-47-0CH3 2012-10-05 20-25-14\001-0101.D

Instrument 1 1/29/2013 9:10:07 PM FX

Sample Name: YL-3-47-0CH3 Acq. Operator : YDC Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/11/2012 3:38:26 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-0CH3\YL-3-47-0CH3 2012-10-11 15-36-35\ICH-50-50ML-Acq. Method 254NM-20MIN.M Last changed : 10/8/2012 2:44:55 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-0CH3\YL-3-47-0CH3 2012-10-11 15-36-35\001-0101.D\DA. M (ICH-50-50ML-254NM-20MIN.M) Last changed : 1/29/2013 9:10:53 PM by FX (modified after loading) W/DIA, Wavelergth=254nm(DALC/DATA/YL/347-0CH3/YL-347-0CH3 2012-10-11 15:36:35001-0101.D) mAU 2 \cap 400 (3ab) 300 \cap 200 100 127 ÷ ٥ 11 12 13 14 15 16 ģ 10 min Area Percent Report _____ Sorted By . Simul : Multiplier 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width 1 9.478 BV 0.2788 8948.98633 496.22672 91.6117 2 14.127 BB 0.4309 819.40564 29.58645 8.3883 9768.39197 525.81316 Totals : _____

Data File D:\LC\DATA\YL\YL-3-47-0CH3\YL-3-47-0CH3 2012-10-11 15-36-35\001-0101.D

Instrument 1 1/29/2013 9:10:58 PM FX

Sample Name: YL-3-47-PCH3 Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/12/2012 3:30:49 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-PCH3\YL-3-47-PCH3 2012-10-12 15-29-25\ICH-50-50ML-Acq. Method 254NM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-PCH3\YL-3-47-PCH3 2012-10-12 15-29-25\001-0101.D\DA. M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 9:14:09 PM by FX (modified after loading) WWD1 A. Wavelength=254 nm (DXLC/DATA/YL/YL-347-PCH3/YL-347-PCH3/2012-10-12/15-29-25/001-0101.D) mAU 8 70 -60 -7.141 (3ac) 50 Ω 40 -30 -20 -10 ٥ 18 10 12 14 16 min Area Percent Report _____ Sorted By . Simul : Multiplier 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width 1 10.830 BB 0.3303 1636.98621 76.32961 50.2297 2 17.141 BB 0.5339 1622.01636 46.97461 49.7703 1 10.830 BB 3259.00256 123.30422 Totals : _____

Data File D:\LC\DATA\YL\YL-3-47-PCH3\YL-3-47-PCH3 2012-10-12 15-29-25\001-0101.D

Instrument 1 1/29/2013 9:14:15 PM FX

Sample Name: YL-3-47-PCH3 Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/15/2012 5:38:34 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-PCH3\YL-3-47-PCH3 2012-10-15 17-37-11\ICH-50-50ML-Acq. Method 254NM-25MIN.M Last changed : 10/6/2012 3:15:19 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-PCH3\YL-3-47-PCH3 2012-10-15 17-37-11\001-0101.D\DA. M (ICH-50-50ML-254NM-25MIN.M) Last changed : 1/29/2013 9:14:56 PM by FX (modified after loading) W/D1 A. Wavelergth=254nm(DALC/DATA/YL/VL-347-PCH3/VL-347-PCH3 2012-10-15 17-37-11/001-0101.D) mAU 8 Į 300 250 200 -(3ac) 150 100 -50 -15 E D 18 1Ô 12 14 18 min Area Percent Report _____ Sorted By . Simul Multiplier . 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width 1 10.806 VB 0.3272 6937.76758 327.56784 90.8713 2 17.157 BB 0.5403 696.95325 20.01100 9.1287 1 10.806 VB 7634.72083 347.57884 Totals : _____

Data File D:\LC\DATA\YL\YL-3-47-PCH3\YL-3-47-PCH3 2012-10-15 17-37-11\001-0101.D

Instrument 1 1/29/2013 9:15:03 PM FX

Sample Name: YL-3-47-MCH3 Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/12/2012 2:56:48 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-MCH3\YL-3-47-MCH3 2012-10-12 14-55-33\ICH-50-50ML-Acq. Method 254NM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-MCH3\YL-3-47-MCH3 2012-10-12 14-55-33\001-0101.D\DA. M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 8:06:01 PM by FX (modified after loading) WWDIA Wavelergth=254nm(DXLCVDATAVYLVI-3-47-MCH3VYL3-47-MCH3 2012-10-12 1455-33001-0101.D) mAU 8 Į 80 -14.500 60 -(3ad) 0: 40 20 -٥ 12 14 16 10 min Area Percent Report Sorted By : Signal 1.0000 Multiplier 1 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area الاست المراجع المراجع المراجع المراجع المراجع المراجع المراجع (المراجع المراجع ا 1 10.163 BB 0.3088 1852.73242 92.15969 50.1461 2 14.500 BB 0.4437 1841.93372 64.52709 49.8539 Totals : 3694.66614 156.68678

Data File D:\LC\DATA\YL\YL-3-47-MCH3\YL-3-47-MCH3 2012-10-12 14-55-33\001-0101.D

**** End of Report ***

Instrument 1 1/29/2013 8:06:08 PM FX

Sample Name: YL-3-47-MCH3

Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/15/2012 2:47:58 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-MCH3\YL-3-47-MCH3 2012-10-15 14-46-24\ICH-50-50ML-Acq. Method 254NM-20MIN.M Last changed : 10/8/2012 2:44:55 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-MCH3\YL-3-47-MCH3 2012-10-15 14-46-24\001-0101.D\DA. M (ICH-50-50ML-254NM-20MIN.M) Last changed : 1/29/2013 8:08:41 PM by FX (modified after loading) WWDIA Wavelergth=254nm(DXLCVDATAVYLYL-3-47-MCH3VYL-3-47-MCH3 2012-10-15 1448-24001-0101.D) 88 日 日 mAU 300 250 (3ad) 200 150 O 100 1558 50 · ٥ 15 11 12 13 14 16 10 min Area Percent Report Sorted By Signal : Multiplier 1 1.0000 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area [min] mAU *s [mAU] # [min] 8 1 10.166 BB 0.3099 6843.88770 340.94449 90.9717 2 14.558 BB 0.4411 679.20776 23.66611 9.0283

Data File D:\LC\DATA\YL\YL-3-47-MCH3\YL-3-47-MCH3 2012-10-15 14-46-24\001-0101.D

*** End of Report ***

7523.09546 364.61060

... Fug of Keboir ...

Instrument 1 1/29/2013 8:08:49 PM FX

Totals :

Data File D:\LC\DATA\YL\YL-3-47-0F\YL-3-47-0F 2012-10-05 19-30-02\001-0101.D

Sample Name: YL-3-47-0F Acq. Operator : YL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/5/2012 7:31:18 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-0F\YL-3-47-0F 2012-10-05 19-30-02\ICH-50-50ML-254MM. Acq. Method М Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-0F\YL-3-47-0F 2012-10-05 19-30-02\001-0101.D\DA.M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 9:12:09 PM by FX (modified after loading) W/D1 A. Wavelength=254nm (DXLCXDATAXYLX1-347-0FX1-347-0F2012-10-0519-30-02001-0101.D) 0-298 mAU 500 - \cap 400 (3ae) 300 562Ο 200 100 -٥ 12 16 18 20 ź 24 min 10 14 _____ Area Percent Report _____ Sorted By . Simul : Multiplier 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width 1 10.598 BB 0.3092 1.01775e4 508.62097 50.0895 2 21.562 BB 0.6595 1.01411e4 239.02881 49.9105 2.03185e4 747.64978 Totals : _____

Instrument 1 1/29/2013 9:12:14 PM FX

Data File D:\LC\DATA\YL\YL-3-47-0F\YL-3-47-0F 2012-10-11 14-59-20\001-0101.D

Sample Name: YL-3-47-0F Acq. Operator : YDC Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/11/2012 3:00:37 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-0F\YL-3-47-0F 2012-10-11 14-59-20\ICH-50-50ML-254MM-Acq. Method 30MIN.M Last changed : 10/6/2012 3:59:52 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-0F\YL-3-47-0F 2012-10-11 14-59-20\001-0101.D\DA.M (ICH-50-50ML-254MM-30MIN.M) Last changed : 1/29/2013 9:13:02 PM by FX (modified after loading) W/D1A Wavelength=254nm(DXLCXDATAXYLX1-347-0FX1-347-0F2012-10-111459-20001-0101.D) mAU #0-537 300 250 (3ae) 200 C) 150 -100 -50 · 1.550 2 ٥ 24 min 12 18 ź 10 14 16 20 _____ Area Percent Report _____ Sorted By 1 Simul Multiplier . 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width 1 10.537 BB 0.3122 7131.78906 354.02740 93.9355 2 21.550 BB 0.6631 460.43182 10.74324 6.0645 7592.22089 364.77065 Totals :

Instrument 1 1/29/2013 9:13:07 PM FX

Data File D:\LC\DATA\YL\YL-3-47-PF\YL-3-47-PF 2012-10-05 20-03-27\001-0101.D Sample Name: YL-3-47-PF Acq. Operator : YL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/5/2012 8:04:48 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-PF\YL-3-47-PF 2012-10-05 20-03-27\ICH-50-50ML-254MM. Acq. Method М Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-PF\YL-3-47-PF 2012-10-05 20-03-27\001-0101.D\DA.M (ICH-50-50ML-254MM.M) Last changed : 1/29/2013 8:28:31 PM by FX (modified after loading) WWDIA, Wavelength=254nm(DALC/DATAYL/NL-3-47-PF/YL-3-47-PF2012-10-05 20-03-27/001-0101.D) mAU ß 120 -12.458 100 С 80 -(3af) 60 -Ο 40 20 -٥ ģ 10 11 13 12 min Area Percent Report Sorted By : Signal 1.0000 : Multiplier 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 9.058 VB 0.2617 2108.23682 124.42626 50.1061 2 12.458 BB 0.3670 2099.30688 88.98952 49.8939 Totals : 4207.54370 213.41578

**** End of Report ***

Instrument 1 1/29/2013 8:28:37 PM FX

Sample Name: YL-3-47-PF Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/8/2012 5:02:27 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-PF\YL-3-47-PF 2012-10-08 17-01-01\ICH-50-50ML-254MM-Acq. Method 20MIN.M Last changed : 10/8/2012 2:44:55 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-PF\YL-3-47-PF 2012-10-08 17-01-01\001-0101.D\DA.M (ICH-50-50ML-254MM-20MIN.M) Last changed : 1/29/2013 8:29:48 PM by FX (modified after loading) W/D1A, Wavelength=264nm (DALCADATAYLAVL-347-PFVL-347-PF2012-10-08 17-01-01001-0101.D) mAU 88 350 300 250 (3af) 200 \cap 150 -100 -**†**52 5D · ŝ ٥ 13 ģ 12 14 10 11 min Area Percent Report Sorted By Signal 1 Multiplier 1 1.0000 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area [min] mAU *s [mAU] # [min] 5 1 9.038 VB 0.2631 6710.80664 393.38159 93.8005 2 12.452 BB 0.3780 443.53589 18.07591 6.1995

Data File D:\LC\DATA\YL\YL-3-47-PF\YL-3-47-PF 2012-10-08 17-01-01\001-0101.D

Totals : 7154.34253 411.45750

**** End of Report ***

Instrument 1 1/29/2013 8:29:53 PM FX
Data File D:\LC\DATA\YL\YL-3-47-MF\YL-3-47-MF 2012-10-05 17-39-09\001-0101.D Sample Name: YL-3-47-mF Acq. Operator : YL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/5/2012 5:40:37 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-MF\YL-3-47-MF 2012-10-05 17-39-09\ICH-50-50ML-254MM. Acq. Method М Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-MF\YL-3-47-MF 2012-10-05 17-39-09\001-0101.D\DA.M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 9:04:26 PM by FX (modified after loading) W/D1 A. Wavelength=264 nm (DALCADATAXYL/YL-3-47-MFYL-3-47-MF 2012-10-05 17-39-09001-0101.D) mAU 11.572 250 200 (3ag) 150 O: 100 -50 ٥ 12 13 ģ 10 11 14 min Area Percent Report Sorted By Signal : 1.0000 : Multiplier 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *5 [mAU] % 1 9.851 BB 0.2853 5466.89453 298.07571 50.0353 2 11.572 BB 0.3384 5459.16992 250.83772 49.9647 Totals : 1.09261e4 548.91344 *** End of Report ***

Instrument 1 1/29/2013 9:04:31 PM FX

Sample Name: YL-3-47-MF Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/8/2012 2:53:26 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-MF\YL-3-47-MF 2012-10-08 14-51-58\ICH-50-50ML-254MM-Acq. Method 20MIN.M Last changed : 10/8/2012 2:44:55 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-MF\YL-3-47-MF 2012-10-08 14-51-58\001-0101.D\DA.M (ICH-50-50ML-254MM-20MIN.M) Last changed : 1/29/2013 9:05:32 PM by FX (modified after loading) W/D1A, Wavelength=264nm (DALCADATAXYLVL-3-47-MF2012-10-08 14-51-58/001-0101.D) mAU 2 700 -600 500 (3ag) 400 -O300 200 1534 100 -٥ 11.5 12 9.5 10 10.5 11 12.5 min Area Percent Report Sorted By : Signal Multiplier 1 1.0000 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Area [mAU [min] mAU *s # [min] 1

Data File D:\LC\DATA\YL\YL-3-47-MF\YL-3-47-MF 2012-10-08 14-51-58\001-0101.D

	[mmir]		[mmir]	10220		Lunzo	1	•	
1	9.820	VB	0.2843	1.37896	e4	750.	30682	92.3717	
2	11.534	BB	0.3386	1138.78	308	52.	26485	7.6283	
Total	5 :			1.49284	le4	802.	57167		

**** End of Report ***

Instrument 1 1/29/2013 9:05:37 PM FX

Sample Name: YL-3-47-PC1 Acq. Operator : YL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 9/28/2012 7:29:32 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-PCL\YL-3-47-PCL 2012-09-28 19-28-05\ICH-50-50ML-Acq. Method 254NM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-PCL\YL-3-47-PCL 2012-09-28 19-28-05\001-0101.D\DA.M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 9:16:24 PM by FX (modified after loading) WWD1 A. Wavelergth=254 nm (D:\LC\DATA\YL\YL-347-PCL\YL-347-PCL 2012-09-28 19:28-05'001-0101.D) mAU ž CI 140 ŝ 0 120 \sim 100 -(3ah) 80 -O60 -40 20 -٥ -11 13 14 15 ģ 10 12 min Area Percent Report _____ Sorted By . Simul : Multiplier 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width 1 9.621 BB 0.2843 2797.49121 152.18671 50.1040 2 12.694 BB 0.3792 2785.87280 113.62158 49.8960 5583.36401 265.80829 Totals : _____

Data File D:\LC\DATA\YL\YL-3-47-PCL\YL-3-47-PCL 2012-09-28 19-28-05\001-0101.D

Instrument 1 1/29/2013 9:16:31 PM FX

Data File D:\LC\DATA\YL\YL-4-5\YL-4-5 2013-02-22 20-21-59\001-0101.D

Sample Name: YL-4-5 Acq. Operator : LQH Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 2/22/2013 8:23:14 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-4-5\YL-4-5 2013-02-22 20-21-59\ICH-50-50-1ML-254MM-20MIN. Acq. Method М Last changed : 1/12/2013 11:01:51 AM by FX Analysis Method : D:\LC\DATA\YL\YL-4-5\YL-4-5 2013-02-22 20-21-59\001-0101.D\DA.M (ICH-50-50-1ML-254NM-20MIN.M) Last changed : 4/1/2013 3:00:06 PM by FX (modified after loading) WDIA, Wavelength=254nm (D:LC/DATAYL/YL-45/YL4-5 2013-02-22 20-21-59/001-0101.D) 1.4. 1.4.1 1 85 mAU 300 晋 250 \cap 200 (3ah) 150 Ο 100 -Ini Ini Ini 50 ŝ₽ ٥ 11 14 15 10 12 13 min Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 10.785 MM 0.3230 5880.54932 303.38959 92.7120 2 14.497 MM 0.4447 462.26587 17.32384 7.2880 6342.81519 320.71342 Totals :

Instrument 1 4/1/2013 3:00:11 PM FX

Sample Name: YL-3-47-1NAP Seq. Line : Acq. Operator : YDC 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/20/2012 4:13:56 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-1NAP\YL-3-47-1NAP 2012-10-20 16-12-27\ICH-50-50ML-Acq. Method 254NM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-1MAP\YL-3-47-1MAP 2012-10-20 16-12-27\001-0101.D\DA. M (ICH-50-50ML-254MM.M) 1/29/2013 8:57:10 -----Last changed : 1/29/2013 8:57:13 PM by FX WWD1 A. Wavelength=254 nm (DALCXDATAXYLYL-3-47-1NAPYL-3-47-1NAP 2012-10-20 16-12-27001-0101.D) mAU g Ο 50 17.425 40 (3ai) Ο S 30 20 -10 -D 10 12 14 16 18 min _____ Area Percent Report _____ e . . .

Data File D:\LC\DATA\YL\YL-3-47-1NAP\YL-3-47-1NAP 2012-10-20 16-12-27\001-0101.D

Sorted By	:	նորու	
Multiplier	:	1.0000	
Dilution	:	1.0000	
Use Multiplier s	Dilution	Factor with	ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak	RetTime	Type	Width	Ar	ea	Heig	nt	Area
#	[min]		[min]	mAU	*s	[mAU]	둽
1	11.782	BB	0.3769	1351.	95154	55.3	1890	50.0910
2	17.425	BB	0.5631	1347.	03809	36.6	2056	49.9090

Totals : 2698.98962 91.93946

**** End of Report ***

Instrument 1 1/29/2013 8:57:33 PM FX

Sample Name: YL-3-47-1NAP Acq. Operator : LQH Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/22/2012 5:30:56 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-1NAP\YL-3-47-1NAP 2012-10-22 17-29-28\ICH-50-50ML-Acq. Method 254NM-25MIN.M Last changed : 10/6/2012 3:15:19 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-1MAP\YL-3-47-1MAP 2012-10-22 17-29-28\001-0101.D\DA. M (ICH-50-50ML-254NM-25MIN.M) Last changed : 1/29/2013 8:59:56 PM by FX (modified after loading) W/D1 A. Wavelergth=254nm(DALC/DATA/YL/VL-347-1NAP/VL-347-1NAP 2012-10-22 17-29-28001-0101.D) mAU 8 74 160 С 140 120 (3ai) 0 S 100 -80 60 -40 · 17 380 20 D 16 18 10 12 14 min Area Percent Report _____ Sorted By . Simul : Multiplier 1.0000 Dilution . 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width 1 11.730 BB 0.3752 4222.47852 173.76492 96.2335 2 17.380 BB 0.5619 165.26472 4.45965 3.7665 4387.74324 178.22457 Totals : _____

Data File D:\LC\DATA\YL\YL-3-47-1NAP\YL-3-47-1NAP 2012-10-22 17-29-28\001-0101.D

Instrument 1 1/29/2013 9:00:01 PM FX

Sample Name: YL-3-47-2NAP Acq. Operator : YL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 10/6/2012 2:38:59 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-47-2NAP\YL-3-47-2NAP 2012-10-06 14-37-37\ICH-50-50ML-Acq. Method 254NM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-2NAP\YL-3-47-2NAP 2012-10-06 14-37-37\001-0101.D\DA. M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 9:00:53 PM by FX (modified after loading) W/DIA, Wavelength=264nm (DXLCVDATAXYLVL-347-2NAP/YL-3-47-2NAP 2012-10-06 14-37-37001-0101.D) mAU 8 O 200 -175 17.557 (3aj) 150 -0 125 -100 -75 -50 -25 -D 18 14 16 10 12 min Area Percent Report Sorted By Signal 1 1.0000 1 Multiplier 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *5 [mAU] %

Data File D:\LC\DATA\YL\YL-3-47-2NAP\YL-3-47-2NAP 2012-10-06 14-37-37\001-0101.D

----|-----| 1 12.750 VB 0.4071 5600.19434 212.04953 50.1211 2 17.557 BB 0.5586 5573.14209 154.17569 49.8789 Totals : 1.11733e4 366.22522

**** End of Report ***

Instrument 1 1/29/2013 9:01:59 PM FX

Data File D:\LC\DATA\YL\YL-3-133\YL-3-133 2012-12-27 16-40-59\012-0101.D Sample Name: YL-3-133 Acq. Operator : FX Seq. Line : 1 Location : Vial 12 Acq. Instrument : Instrument 1 Injection Date : 12/27/2012 4:42:19 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-133\YL-3-133 2012-12-27 16-40-59\ICH-50-50ML-254MM-Acq. Method 25MIN.M Last changed : 10/6/2012 3:15:19 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-133\YL-3-133 2012-12-27 16-40-59\012-0101.D\DA.M (ICH-50-50ML-254NM-25MIN.M) Last changed : 4/1/2013 2:53:35 PM by FX (modified after loading) W/D1 A. Wavelength=264 nm (DALCADATAYLAYLAYLA3-1332012-12-27 16-40-59/012-0101.D) (194. Kr, 93 133860 mAU 500 400 (3aj) 300 200 100 18.921 ٥ ź 14 16 18 min 12 Area Percent Report _____ Sorted By : Signal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area
 # [min]
 [min]
 mAU
 *s
 [mAU
 %

 ---- ----- ----- ----- ----- ----- ----- 1 13.660 MM 0.4981 1.74193e4 582.84009 93.9622 2 18.921 MM 0.6743 1119.33411 27.66591 6.0378 1.85386e4 610.50600 Totals :

Instrument 1 4/1/2013 2:53:42 PM FX

Sample Name: YL-3-47-thienyl Acq. Operator : YL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 1/2/2006 9:15:53 PM Inj : 1 Inj Volume : 5 µl : D: \LC\ DATA\ YL\ YL-3-47-TH IENYL\ YL-3-47-TH IENYL 2006-01-02 21-14-37\ ICH-50-Acq. Method 50ML-254MM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-THIENYL\YL-3-47-THIENYL 2006-01-02 21-14-37\001-0101.D\DA.M (ICH-50-50ML-254MM.M) Last changed : 1/29/2013 8:33:56 PM by FX (modified after loading) WWD1A_Wavelergth=254nm(D:\LCDATAXYL\YL-347-TH/ENYL\YL-347-TH/ENYL2006-01-0221-14-37001-0101.D) mAU 00 175 18.139 150 125 -(3ak) 100 -0 75 -50 25 -٥ 12 14 15 19 min 11 13 17 18 16 Area Percent Report Sorted By . Signal

Data File D:\LC\DATA\YL\YL-3-47-THIENYL\YL-3-47-THIENYL 2006-01-02 21-14-37\001-0101.D

Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Туре	Width [min]	A1 mAll	tea ★s	Heig [mAll	nt 1	Area %
			[]				I	-
1	13.018	BB	0.3876	5081.	63135	202.3	7563	49.5872
2	18.139	VB	0.5594	5166.	23389	143.1	13538	50.4128
Total	5 :			1.024	179e4 -	345.5	51100	

------ **** End of Report ***

Instrument 1 1/29/2013 8:34:01 PM FX

Sample Name: YL-3-47-THIENYL

Seq. Line : Acq. Operator : THL 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 1/8/2006 12:52:08 PM Inj : 1 Inj Volume : 5 µl : D: \LC\DATA\YL\YL-3-47-THIENYL\YL-3-47-THIENYL 2006-01-08 12-50-35\ICH-50-Acq. Method 50ML-254NM-30MIN.M Last changed : 10/6/2012 3:59:52 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-47-THIENYL\YL-3-47-THIENYL 2006-01-08 12-50-35\001-0101.D\DA.M (ICH-50-50ML-254MM-30MIN.M) Last changed : 1/29/2013 8:35:11 PM by FX (modified after loading) WWD1A_Wavelergth=254nm(D:\LCDATAXYL\YL-347-THENYL\YL-347-THENYL2006-01-08 12-50-35001-0101.D) mAU 800 700 -600 -500 -(3ak) 400 - \cap 300 -200 -18.163 100 -٥ 12 14 16 18 min Area Percent Report Sorted By . Signal Multiplier - 1 1.0000 1.0000 Dilution . Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Area

Data File D:\LC\DATA\YL\YL-3-47-THIENYL\YL-3-47-THIENYL 2006-01-08 12-50-35\001-0101.D

T C OR	TCOL THE	-1P-	widdie				,	THE C G
#	[min]		[min]	mAU	*s	[mAU]	5
1	12.981	VB	0.3861	2.038	15e4	815.8	38013	97.4018
2	18.153	BB	0.5507	543.	68622	15.3	11477	2.5982
Total	Ls :			2.092	51e4	830.9	99490	

*** End of Report ***

Instrument 1 1/29/2013 8:35:17 PM FX

Data File D:\LC\DATA\YL\YL-3-63B\YL-3-63A 2006-01-06 12-02-40\053-0101.D Sample Name: YL-3-63A Acq. Operator : THL Seq. Line : 1 Acq. Instrument : Instrument 1 Location : Vial 53 Injection Date : 1/6/2006 12:04:21 PM Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\DATA\YL\YL-3-63B\YL-3-63A 2006-01-06 12-02-40\ICH-50-50ML-254WM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-63B\YL-3-63A 2006-01-06 12-02-40\053-0101.D\DA.M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 8:37:14 PM by FX (modified after loading) W/DIA, Wavelength=254nm(D/LC/DATAYL/YL-3-63BY/L-3-63A2006-01-0612-02-40/053-0101.D) mAU 140 120 25.407 100 -(3bj) OCH₃ 80 0= 60 40 20 ۵ 26 14 16 18 20 24 28 12 22 min _____ Area Percent Report _____ Sorted By : Signal Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Area Peak RetTime Type Width Height Area # [min] [min] mAU *s [mAU] % 1 17.147 BB 0.6178 6080.92090 151.65036 50.1486 2 25.407 BB 0.9220 6044.88525 100.65038 49.8514 Totals : 1.21258e4 252.30074

**** End of Report ***

Instrument 1 1/29/2013 8:37:19 PM FX

Data File D:\LC\DATA\YL\YL-3-63B\YL-3-63A 2006-01-08 11-55-26\001-0101.D Sample Name: YL-3-63A Acq. Operator : THL Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 1/8/2006 11:56:56 AM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-63B\YL-3-63A 2006-01-08 11-55-26\ICH-50-50ML-254MM-Acq. Method 35MIN.M Last changed : 9/29/2012 1:31:31 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-63B\YL-3-63A 2006-01-08 11-55-26\001-0101.D\DA.M (ICH-50-50ML-254NM-35MIN.M) Last changed : 1/29/2013 8:38:05 PM by FX (modified after loading) WDIA, Wavelength=254nm (DALC/DATAYL/YL-363BV/L-363A2006-01-08 11-55-26/001-0101.D) mAU ដ 60 50 40 -(3bj) OCH₃ 30 -0= 20 Ν 25.337 10 n 14 16 18 26 20 28 22 24 min Area Percent Report _____ Sorted By Signal : : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Area [min] mAU *s [mAU] # [min] 8 1 17.122 BB 0.6156 2608.60278 64.75417 92.1757 2 25.337 BB 0.7927 221.43080 3.70056 7.8243 Totals : 2830.03358 68.45474

*** End of Report ***

Instrument 1 1/29/2013 8:38:09 PM FX

Data File D:\LC\DATA\YL\YL-3-63B\YL-3-63B 2006-01-06 11-30-56\072-0101.D Sample Name: YL-3-63B Acq. Operator : THL Seq. Line : 1 Location : Vial 72 Acq. Instrument : Instrument 1 Injection Date : 1/6/2006 11:32:15 AM Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\DATA\YL\YL-3-63B\YL-3-63B 2006-01-06 11-30-56\ICH-50-50ML-254MM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-63B\YL-3-63B 2006-01-06 11-30-56\072-0101.D\DA.M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 9:30:30 PM by FX (modified after loading) WWD1 A, Wavelength=254nm (D\LC\DATA\YL\YL-363B\YL-363B 2006-01-06 11-30-56072-0101.D) mAU 70 16.933 60 50 -(3cj) CI 40 Ο 30 -20 -10 -۵ 18 12 14 18 1Ò mir _____ Area Percent Report _____ Sorted By : Signal : Multiplier 1.0000 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 12.408 BB 0.4292 2080.08765 74.48940 50.2223 2 16.933 BB 0.5973 2061.67188 53.42269 49.7777 Totals : 4141.75952 127.91208 *** End of Report ***

Instrument 1 1/29/2013 9:30:37 PM FX

Data File D:\LC\DATA\YL\YL-3-63B\YL-3-63B 2006-01-07 12-06-07\001-0101.D Sample Name: YL-3-63b Acq. Operator : LQH Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 1/7/2006 12:07:22 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-63B\YL-3-63B 2006-01-07 12-06-07\ICH-50-50ML-254MM-Acq. Method 25MIN.M Last changed : 10/6/2012 3:15:19 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-63B\YL-3-63B 2006-01-07 12-06-07\001-0101.D\DA.M (ICH-50-50ML-254NM-25MIN.M) Last changed : 1/29/2013 9:22:57 PM by FX (modified after loading) WWD1A, Wavelength=264nm (DALCADATAYLAYL-3638)/1-3638 2006-01-07 12-08-07/001-0101.D) for this? 125645 mAU 500 400 \cap 300 (3cj) 200 0: 100 -17.208 Û 12 14 16 18 min 10 Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 12.545 MM 0.4587 1.41435e4 513.91882 92.1190 2 17.208 BB 0.6006 1210.00098 31.12719 7.8810 1.53535e4 545.04601 Totals :

Instrument 1 1/29/2013 9:23:04 PM FX

Data File D:\LC\DATA\YL\YL-3-63D\YL-3-63D 2012-11-23 15-26-25\052-0101.D Sample Name: YL-3-63D Seq. Line : Acq. Operator : YL 1 Location : Vial 52 Acq. Instrument : Instrument 1 Injection Date : 11/23/2012 3:28:12 PM Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\DATA\YL\YL-3-63D\YL-3-63D 2012-11-23 15-26-25\ICH-50-50ML-254WM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-63D\YL-3-63D 2012-11-23 15-26-25\052-0101.D\DA.M (ICH-50-50ML-254NM.M) Last changed : 4/1/2013 10:53:52 AM by FX (modified after loading) W/D1 A, Wavelength=254nm(D\LC\DATAYLYL-363D\YL-363D2012-11-23 15-26-25'052-0101.D) fer set. mAU Ã 140 Steele Base 120 \cap 100 (3dj) 80 \cap 60 -40 20 ٥· 14 16 18 ź ź 24 тin _____ Area Percent Report _____ Sorted By : Signal Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area . Height Area # [min] [min] mAU *5 [mAU] % 1 14.372 MM 0.5615 5347.10156 158.70834 51.0356 2 21.806 MM 0.8222 5130.08984 103.99667 48.9644 1.04772e4 262.70501 Totals : _____ *** End of Report ***

Instrument 1 4/1/2013 10:53:59 AM FX

Data File D:\LC\DATA\YL\YL-3-63D\YL-3-63D 2012-11-24 19-51-04\023-0101.D Sample Name: YL-3-63D Acq. Operator : YL Seq. Line : 1 Location : Vial 23 Acq. Instrument : Instrument 1 Injection Date : 11/24/2012 7:52:33 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-63D\YL-3-63D 2012-11-24 19-51-04\ICH-50-50ML-254MM-Acq. Method 30MIN.M Last changed : 10/6/2012 3:59:52 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-63D\YL-3-63D 2012-11-24 19-51-04\023-0101.D\DA.M (ICH-50-50ML-254NM-30MIN.M) Last changed : 1/29/2013 9:27:16 PM by FX (modified after loading) W/D1A, Wavelength=264nm (DALCADATAYL\YL-363DXYL3-63D2012-11-2419-51-04/023-0101.D) 192. BUNK mAU 200 175 150 (3dj) 125 100 0: 75 50 625 25 ٥ 14 22 16 18 20 24 min 12 Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 14.253 MM 0.5488 6821.46582 207.17690 91.1403 2 21.625 MM 0.7876 663.10907 14.03206 8.8597 7484.57489 221.20895 Totals :

Instrument 1 1/29/2013 9:28:35 PM FX



Instrument 1 1/29/2013 9:24:12 PM FX

Data File D:\LC\DATA\YL\YL-3-63C\YL-3-63C 2012-11-24 12-26-43\043-0201.D Sample Name: YL-3-63C Acq. Operator : YL Seq. Line : 2 Location : Vial 43 Acq. Instrument : Instrument 1 Injection Date : 11/24/2012 12:49:16 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-63C\YL-3-63C 2012-11-24 12-26-43\ICH-50-50ML-254MM-Acq. Method 20MIN.M Last changed : 10/8/2012 2:44:55 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-63C\YL-3-63C 2012-11-24 12-26-43\043-0201.D\DA.M (ICH-50-50ML-254NM-20MIN.M) Last changed : 1/29/2013 9:25:02 PM by FX (modified after loading) W/DIA Wavelength=264nm (DXLCVDATAXYLYL-363CYL-3-63C 2012-11-24 12-26-431043-0201.D) mAU 5 500 -Ã 400 (3ej) Ο S 300 200 100 -4.187 ٥ 11 12 16 1Ò 13 14 15 min Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] & 1 11.161 VB 0.3833 1.26499e4 508.61636 96.2128 2 14.187 BB 0.5071 497.93774 15.21023 3.7872 1.31478e4 523.82660 Totals :

Instrument 1 1/29/2013 9:25:12 PM FX

Sample Name: YL-3-106A Acq. Operator : hzl Seq. Line : 1 Location : Vial 32 Acq. Instrument : Instrument 1 Injection Date : 12/7/2012 11:01:02 AM Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\DATA\YL-3-106A\YL-3-106A 2012-12-07 10-59-09\ICH-50-50ML-254MM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-106A\YL-3-106A 2012-12-07 10-59-09\032-0101.D\DA.M (ICH-50-50ML-254MM.M) Last changed : 4/1/2013 3:15:45 PM by FX (modified after loading) WWD1 A Wavelength=254nm(DALCDATANYLA-3106A/NL-3-106A/2012-12-07 10-59-09/032-0101.D) mAU 628 100 N-OH 80 60 15.192 O 40 20 4 ۵ 16 1'n 12 14 mi _____ Area Percent Report _____ Sorted By : Signal Multiplier 1.0000 : 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm

Data File D:\LC\DATA\YL\YL-3-106A\YL-3-106A 2012-12-07 10-59-09\032-0101.D

Peak	RetTime	Type	Width	A	rea	Heig	,ht	Area
#	[min]		[min]	mAU	*s	[mAU]	믭
1	6.628	VB	0.2829	2024.	.24878	109.3	39726	49.5729
2	15.192	BB	0.6678	2059.	.13013	47.8	37001	50.4271

Totals : 4083.37891 157.26727

**** End of Report ***

Instrument 1 4/1/2013 3:15:55 PM FX

Data File D:\LC\DATA\YL\YL-3-106B\YL-3-110 2012-12-07 12-20-49\042-0101.D

Sample Name: YL-3-110A Acq. Operator : hzl Seq. Line : 1 Location : Vial 42 Acq. Instrument : Instrument 1 Injection Date : 12/7/2012 12:22:37 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-106B\YL-3-110 2012-12-07 12-20-49\ICH-50-50ML-254MM-Acq. Method 25MIN.M Last changed : 10/6/2012 3:15:19 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-106B\YL-3-110 2012-12-07 12-20-49\042-0101.D\DA.M (ICH-50-50ML-254NM-25MIN.M) Last changed : 4/8/2013 5:30:53 PM by TMC (modified after loading) W/D1 A. Wavelength=264 nm (DXLC/DATAXYL/YL-3106B/YL-3110 2012-12-07 12-20-49/042-0101.D) e^s e^g 6 mAU 400 N-OH 350 300 250 O. 200 150 100 Δ 15.187 50 ٥ 12 14 Ŕ ś. 10 min Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 6.615 MM 0.3090 7929.32568 427.64563 93.9024 2 15.187 MM 0.5797 514.89221 14.80229 6.0976 8444.21790 442.44792 Totals :

Instrument 1 4/8/2013 5:31:06 PM TMC

Data File D:\LC\DATA\YL\YL-3-94\YL-3-94 2012-11-27 10-43-36\081-0101.D Sample Name: YL-3-94 Acq. Operator : THL Seq. Line : 1 Location : Vial 81 Acq. Instrument : Instrument 1 Injection Date : 11/27/2012 10:44:47 AM Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\DATA\YL\YL-3-94\YL-3-94 2012-11-27 10-43-36\ICH-50-50ML-254WM.M Last changed : 9/28/2012 7:27:35 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-94\YL-3-94 2012-11-27 10-43-36\081-0101.D\DA.M (ICH-50-50ML-254NM.M) Last changed : 1/29/2013 8:54:31 PM by FX (modified after loading) W/D1 A. Wavelength=254nm (DXLCVDATAYLYL-3-9442/L-3-942012-11-2710-43-36/081-0101.D) mAU] 50 100 300 a 80 0; 60 40 · 5 20 ٥ 14 16 18 ź 22 тin _____ Area Percent Report _____ Sorted By : Signal Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area . Height Area 1 13.645 BB 0.4782 3745.00952 119.82549 49.8536 2 20.300 W 0.7107 3767.00488 81.33421 50.1464 7512.01440 201.15971 Totals :

**** End of Report ***

Instrument 1 1/29/2013 8:55:00 PM FX

Data File D:\LC\DATA\YL\YL-3-108\YL-3-108 2012-12-04 09-39-52\031-0101.D Sample Name: YL-3-108 Acq. Operator : FX Seq. Line : 1 Location : Vial 31 Acq. Instrument : Instrument 1 Injection Date : 12/4/2012 9:41:10 AM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-108\YL-3-108 2012-12-04 09-39-52\ICH-50-50ML-254MM-Acq. Method 25MIN.M Last changed : 10/6/2012 3:15:19 PM by YL Analysis Method : D:\LC\DATA\YL\YL-3-108\YL-3-108 2012-12-04 09-39-52\031-0101.D\DA.M (ICH-50-50ML-254NM-25MIN.M) Last changed : 1/29/2013 8:51:50 PM by FX (modified after loading) W/D1 A. Wavelergth=264 nm (DALCADATAYLAYL-3-108/VL-3-108 2012-12-04-09-39-52/031-0101.D) mAU 2 300 \cap 250 200 0: 150 -100 178 50 5 2 ٥ 12 14 20 22 1Ĥ 16 18 min Area Percent Report _____ Sorted By : Signal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Area Height Area [min] mAU *s [mAU] # [min] 5

 1
 13.929 BB
 0.4880 1.04882e4
 329.34100 93.8694

 2
 21.178 BB
 0.7126 684.97815 14.38924 6.1306

 Totals :
 1.11732e4 343.73024

Instrument 1 1/29/2013 8:51:55 PM FX

Data File E:\DATA\CJW\YL-3-102-103\YL-3-102-103 2012-12-18 11-48-35\003-0201.D Sample Name: YL-3-102

Acq. Operator :	SYSTEM Seq. Line : 2
Acq. Instrument :	1260HPLC-DAD Location : Vial 3
Injection Date :	12/18/2012 12:00:29 PM Inj: 1
	Inj Volume : 5.000 µl
Acq. Method :	E:\DATA\CJW\YL-3-102-103\YL-3-102-103 2012-12-18 11-48-35\DAD-ICH-10-90-
	1ML-60MIN.M
Last changed :	12/18/2012 11:48:35 AM by SYSTEM
Analysis Method :	E:\DATA\UJW\YL-3-102-103\YL-3-102-103 2012-12-18 11-48-35\DAD-1UH-10-90- 1ML-60MIN.M (Sequence Method)
Last changed :	1/30/2013 12:55:03 PM by SYSTEM
	(modified after loading)
DAD1 F, Sig=25	14,4 Ref=off (E:\DATA\CJ\W\YL-3-102-103\YL-3-102-103 2012-12-18 11-48-35\003-0201.D)
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	34 36 38 40 42 44 min Area Percent Report
Sorted By	34 36 38 40 42 44 min Area Percent Report : Signal
Sorted By Multiplier	34 36 38 40 42 44 min Area Percent Report : Signal : 1.0000
Sorted By Multiplier Dilution	34 36 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip	34 38 38 40 42 44 min Area Percent Report : Signal : 1.0000 : 1.0000 lier 4 Dilution Factor with ISTDs
Sorted By Multiplier Dilution Do not use Multip.	34 38 38 40 42 44 min Area Percent Report : Signal : 1.0000 : 1.0000 lier & Dilution Factor with ISTDs
Sorted By Multiplier Dilution Do not use Multip.	34 36 38 40 42 44 min Area Percent Report : Signal : 1.0000 : 1.0000 lier & Dilution Factor with ISTDs
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F,	34 38 40 42 44 min Area Percent Report : Signal : 1.0000 : 1.0000 i 1.0000 Sig=254,4 Ref=off
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F,	34 36 38 40 42 44 min Area Percent Report : Signal : 1.0000 : 1.0000 lier & Dilution Factor with ISTDs Sig=254,4 Ref=off
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type	Area Percent Report : Signal : 1.0000 : 1.0000 lier & Dilution Factor with ISTDs Sig=254,4 Ref=off Width Area Height Area
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min]	34 36 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min]	34 36 38 40 42 44 min Area Percent Report . Signal : 1.0000 : 1.0000 lier & Dilution Factor with ISTDs Sig=254,4 Ref=off Width Area Height Area [min] [mAU*s] [mAU] *
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min] 	34 36 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min] 	34 36 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min] 	34 38 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min] 	34 38 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min] 	34 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min] 	34 36 38 40 42 44 min Area Percent Report
Sorted By Multiplier Dilution Do not use Multip. Signal 1: DAD1 F, Peak RetTime Type # [min] 	34 36 38 40 42 44 min Area Percent Report

1260HPLC-DAD 1/30/2013 12:55:12 PM SYSTEM

Data File E:\DATA\CJW\YL-3-102-103\YL-3-102-103 2012-12-18 11-48-35\004-0301.D Sample Name: YL-3-103



1260HPLC-DAD 1/30/2013 12:58:16 PM SYSTEM

Data File D:\LC\DATA\YL\YL-3-130\YL-3-130 2013-01-11 16-33-26\001-0101.D

Sample Name: YL-3-130 Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 1/11/2013 4:34:57 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-130\YL-3-130 2013-01-11 16-33-26\ICH-30-70ML-254MM-Acq. Method 35MIN.M Last changed : 1/10/2013 11:09:12 AM by FX Analysis Method : D:\LC\DATA\YL\YL-3-130\YL-3-130 2013-01-11 16-33-26\001-0101.D\DA.M (ICH-30-70ML-254MM-35MIN.M) Last changed : 1/29/2013 9:37:28 PM by FX (modified after loading) W/DIA, Wavelength=254nm (DALC/DATAYL/YL-3-130/VL-3-130 2013-01-11 16-33-26/001-0101.D) mAU 8 A CI 200 D65 8 150 Ο 100 -7 5D · ٥ 18 20 22 24 26 min 16 Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 17.462 BB 0.6139 9910.95996 247.69756 50.6485 2 25.065 BB 0.9117 9657.18066 162.51738 49.3515 1.95681e4 410.21494 Totals :

Instrument 1 1/29/2013 9:37:35 PM FX

Data File D:\LC\DATA\YL\YL-3-146\YL-3-146 2013-01-11 15-56-19\041-0101.D Sample Name: YL-3-146 Acq. Operator : FX Seq. Line : 1 Location : Vial 41 Acq. Instrument : Instrument 1 Injection Date : 1/11/2013 3:57:34 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-146\YL-3-146 2013-01-11 15-56-19\ICH-30-70ML-254MM-Acq. Method 35MIN.M Last changed : 1/10/2013 11:09:12 AM by FX Analysis Method : D:\LC\DATA\YL\YL-3-146\YL-3-146 2013-01-11 15-56-19\041-0101.D\DA.M (ICH-30-70ML-254MM-35MIN.M) Last changed : 4/8/2013 5:17:10 PM by TMC (modified after loading) W/D1 A. Wavelength=264 nm (DALCADATAYLAYL-3-1461/L-3-146 2013-01-11 15-56-19/041-0101.D) mAU 50 С 40 S*** 0 30 20 7 86 10 -٥ 18 24 28 28 32 34 min 20 22 зò 16 Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 17.498 MM 0.6174 222.98653 6.01981 5.9940 2 25.104 MM 1.0285 3497.15308 56.66911 94.0060 3720.13960 62.68892 Totals :

Instrument 1 4/8/2013 5:17:23 PM TMC



Instrument 1 4/1/2013 3:05:53 PM FX

Data File D:\LC\DATA\YL\YL-4-7\YL-4-7 2013-02-23 17-18-19\001-0101.D

Sample Name: YL-4-7 Acq. Operator : LQH Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 2/23/2013 5:19:41 PM Inj : 1 Inj Volume : 5 µl Acq. Method : D:\LC\DATA\YL-YL-4-7\YL-4-7 2013-02-23 17-18-19\ICH-30-70ML-254MM-35MIN.M Last changed : 1/10/2013 11:09:12 AM by FX Analysis Method : D:\LC\DATA\YL-YL-4-7\YL-4-7 2013-02-23 17-18-19\001-0101.D\DA.M (ICH-30-70ML-254MM-35MIN.M) Last changed : 4/1/2013 3:07:55 PM by FX (modified after loading) WWDIA, Wavelength=254nm(D:LC/DATAYL\YL-47\YL-472013-02-2317-18-19'001-0101.D) mAU 70 60 Cl 50 S" 0: 2 40 30 and the state of t 20 epi**-7** 10 n 32 34 20 24 28 зΰ min _____ Area Percent Report _____ Sorted By : Signal Multiplier 1.0000 : 1.0000 Dilution Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] m&U *s [m&U] % 1 19.776 MM 0.6636 432.65024 10.86619 7.0554 2 32.493 MM 1.1823 5699.52539 80.34451 92.9446 Totals : 6132,17563 91,21070 *** End of Report ***

Instrument 1 4/1/2013 3:08:03 PM FX

Data File D:\LC\DATA\YL\YL-3-132\YL-3-132 2013-01-05 09-50-38\013-0101.D Sample Name: YL-3-132 Acq. Operator : FX Seq. Line : 1 Location : Vial 13 Acq. Instrument : Instrument 1 Injection Date : 1/5/2013 9:51:55 AM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-132\YL-3-132 2013-01-05 09-50-38\ICH-30-70ML-254MM-Acq. Method 50MIN.M Last changed : 12/28/2012 3:33:08 PM by FX Analysis Method : D:\LC\DATA\YL\YL-3-132\YL-3-132 2013-01-05 09-50-38\013-0101.D\DA.M (ICH-30-70ML-254MM-50MIN.M) Last changed : 1/29/2013 9:34:04 PM by FX (modified after loading) W/D1 A. Wavelength=264 nm (DALCADATAYLAYL-3-1321/L-3-132 2013-01-05 09-50-38/013-0101.D) and the second s **17**-536 mAU 50 es. Saint 40 S 8 0: 30 -20 -8 10 -٥ 25 άn. ŵ 30 35 min Area Percent Report _____ Sorted By : Simal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 22.236 MF 0.8998 3239.24512 59.99622 50.2536 2 39.586 MM 1.7093 3206.55469 31.26649 49.7464 6445.79980 91.26271 Totals :

Instrument 1 1/29/2013 9:34:53 PM FX

Data File D:\LC\DATA\YL\YL-3-137\YL-3-137 2012-12-31 12-45-26\001-0101.D Sample Name: YL-3-137 Acq. Operator : FX Seq. Line : 1 Location : Vial 1 Acq. Instrument : Instrument 1 Injection Date : 12/31/2012 12:46:39 PM Inj : 1 Inj Volume : 5 µl : D:\LC\DATA\YL\YL-3-137\YL-3-137 2012-12-31 12-45-26\ICH-30-70ML-254MM-Acq. Method 50MIN.M Last changed : 12/28/2012 3:33:08 PM by FX Analysis Method : D:\LC\DATA\YL\YL-3-137\YL-3-137 2012-12-31 12-45-26\001-0101.D\DA.M (ICH-30-70ML-254MM-50MIN.M) Last changed : 4/8/2013 5:26:34 PM by TMC (modified after loading) W/D1A, Wavelength=264nm (DALCADATAYLAYLAYLAY1.3-1372012-12-3112-46-26'001-0101.D) mAU 175 150 125 S١ O: 100 75 50 <u>,</u>19 8 8 25 n 25 30 35 άn. min ŵ Area Percent Report _____ Sorted By : Signal : Multiplier 1.0000 1.0000 Dilution : Use Multiplier & Dilution Factor with ISTDs Signal 1: VWD1 A, Wavelength=254 nm Peak RetTime Type Width Height Area Area # [min] [min] mAU *s [mAU] % 1 22.002 MM 0.8370 1125.99988 22.42122 5.9674 2 38.867 BB 1.4715 1.77433e4 183.06735 94.0326 1.88693e4 205.48857 Totals :

Instrument 1 4/8/2013 5:26:43 PM TMC