Supporting Information

**Enantioselective Organocatalytic Tandem Michael-Aldol Reactions:
One-Pot Synthesis of Chiral Thiochromenes**

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General Information: Commercial reagents were used as received, unless otherwise stated. Merck 60 silica gel was used for chromatography, and Whatman silica gel plates with fluorescence F₂₅₄ were used for thin-layer chromatography (TLC) analysis. ¹H and ¹³C NMR spectra were recorded on Broker Avance 500, and tetramethylsilane (TMS) was used as a reference. Data for ¹H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Data for ¹³C NMR are reported as ppm. HR Mass Spectra were obtained from University of New Mexico Mass Spectral facility.

General Procedure for addition of 2-Mercapto-benzaldehydes to α,β -unsaturated aldehydes: To a solution of an α,β -unsaturated aldehyde (0.1 mmol) in the presence of 10 mol % catalyst **VI**, benzoic acid (0.1 equiv) and 4Å MS (50 mg) in toluene (0.5 mL) was added a 2-mercapto-benzaldehyde (0.15 mmol) and the resulting solution was stirred for 12-16 h at rt or 0 °C. The reaction mixture was directly purified by silica gel chromatography without work-up and fractions were collected and concentrated *in vacuo* to provide the pure desired products.

2-Phenyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 1): Yield: 85%; ¹H NMR (500 MHz, CDCl₃): δ 9.65 (s, 1H), 7.45 (s, 1H), 7.39 (d, 1H, J = 7.5 Hz), 7.24-7.29 (m, 2H), 7.17-7.19 (m, 6H), 5.21 (s, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 191.1, 145.1, 141.6, 134.7, 134.0, 131.7, 131.0, 129.8, 128.6, 127.8, 127.6, 126.4, 125.8, 38.3; $[\alpha]_D^{23}$ = + 574.9 (c = 1.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): 94% ee, t_R = 29.45 (major), 33.93 (minor) min. HRMS (EI) exact mass calcd for $[M+H]^+$ 255.0949, found 255.0946.

2-(4-Methoxy-phenyl)-2H-thiochromene-3-carbaldehyde (Table 2, entry 2): Yield: 82%; ¹H NMR (500 MHz, CDCl₃): δ 9.64 (s, 1H), 7.43 (s, 1H), 7.39 (d, 1H, J = 8.0 Hz), 7.24-7.29 (m, 2H), 7.16-7.19 (m, 1H), 7.11 (d, 2H, J = 9.0 Hz), 6.70 (d, 2H, J = 9.0 Hz), 5.16 (s, 1H), 3.69 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 191.2, 159.2, 144.8, 135.0, 134.1, 134.0, 131.6, 130.9, 129.8, 127.6, 125.7, 114.0, 55.1, 37.8; $[\alpha]_D^{23}$ = + 315.4 (c = 1.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 80:20, flow rate 0.5 mL/min, λ = 254 nm): 85% ee, t_R = 28.28 (minor), 29.90 (major) min. HRMS (EI) exact mass calcd for $[M+H]^+$ 283.0793, found 283.0786.

2-(2-Methoxy-phenyl)-2H-thiochromene-3-carbaldehyde (Table 2, entry 3): Yield: 96%;

¹H NMR (500 MHz, CDCl₃): δ 9.65 (s, 1H), 7.59 (s, 1H), 7.40 (d, 1H, *J* = 7.0 Hz), 7.20 (t, 1H, *J* = 7.0 Hz), 7.14 (t, 3H, *J* = 7.0 Hz), 6.86 (d, 1H, *J* = 8.0 Hz), 6.80 (d, 1H, *J* = 6.5 Hz), 6.67 (t, 1H, *J* = 7.5 Hz), 5.66 (s, 1H), 3.89 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 191.1, 155.4, 146.5, 134.7, 133.7, 131.5, 130.8, 129.9, 128.8, 127.8, 126.8, 125.5, 120.2, 110.9, 55.6, 31.7; [α]_D²³ = + 754.1 (*c* = 1.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 80:20, flow rate 0.5 mL/min, λ = 254 nm): 94% ee, *t*_R = 27.88 (major), 36.03 (minor) min. HRMS (EI) exact mass calcd for [M+H]⁺ 283.0793, found 283.0792.

2-(4-Fluoro-phenyl)-2H-thiochromene-3-carbaldehyde (Table 2, entry 4): Yield: 93%; ¹H NMR (500 MHz, CDCl₃): δ 9.65 (s, 1H), 7.45 (s, 1H), 7.40 (d, 1H, *J* = 7.0 Hz), 7.28 (m, 2H), 7.12 (m, 3H), 6.86 (t, 2H, *J* = 8.5 Hz), 5.18 (s, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 191.0, 163.2, 161.3, 145.0, 137.5, 134.7, 133.8, 131.9, 131.0, 129.7, 128.2, 128.1, 127.6, 126.0, 115.5, 115.4, 37.6; [α]_D²³ = + 339.6 (*c* = 1.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): 86% ee, *t*_R = 24.32 (major), 27.10 (minor) min. HRMS (EI) exact mass calcd for [M+H]⁺ 271.0591, found 271.0592.

2-(4-Nitro-phenyl)-2H-thiochromene-3-carbaldehyde (Table 2, entry 5): Yield: 95%; ¹H NMR (500 MHz, CDCl₃): δ 9.69 (s, 1H), 8.04 (d, 2H, *J* = 9.0 Hz), 7.55 (s, 1H), 7.44 (d, 1H, *J* = 7.5 Hz), 7.36 (d, 2H, *J* = 8.5 Hz), 7.33 (m, 1H), 7.22-7.28 (m, 2H), 5.27 (s, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 190.8, 148.4, 147.3, 145.7, 133.8, 133.0, 132.2, 131.3, 129.5, 127.7, 127.5, 126.4, 123.9, 37.7; [α]_D²³ = + 404.7 (*c* = 1.0, CHCl₃); HPLC (Daicel CHIRALCEL OD-H, Hexane/2-PrOH = 70:30, flow rate 0.7 mL/min, λ = 254 nm): 92% ee, *t*_R = 21.87 (major), 25.67 (minor) min. HRMS (EI) exact mass calcd for [M+H]⁺ 298.0538, found 298.0535.

2-Methyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 6): Yield: 90%; ¹H NMR (500 MHz, CDCl₃): δ 9.61 (s, 1H), 7.34 (t, 2H, *J* = 6.5 Hz), 7.29 (m, 1H), 7.22 (s, 1H), 7.18 (m, 1H), 4.08 (q, 1H, *J* = 7.0 Hz), 1.28 (d, 3H, *J* = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 191.1, 144.1, 137.8, 134.0, 131.3, 130.8, 129.8, 128.2, 125.6, 30.2, 22.1; [α]_D²³ = + 74.2 (*c* = 1.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): 92% ee, *t*_R = 13.50 (minor), 15.32 (major) min. HRMS (EI) exact mass calcd for [M+H]⁺ 191.0531, found 191.0525.

2-Ethyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 7): Yield: 81%; ¹H NMR (500

MHz, CDCl₃): δ 9.62 (s, 1H), 7.33 (d, 2H, J = 8.0 Hz), 7.25-7.29 (m, 2H), 7.16 (t, 3H, J = 7.5 Hz), 3.88 (q, 1H, J_1 = 6.0 Hz, J_2 = 7.5 Hz), 1.56 (m, 2H), 0.92 (t, 3H, J = 7.5 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 191.4, 144.7, 136.7, 134.5, 131.2, 130.8, 130.3, 127.9, 125.6, 37.2, 29.7, 28.2, 10.4; $[\alpha]_D^{23}$ = + 84.9 (c = 1.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): 95% ee, t_R = 20.26 (major), 23.11 (minor) min. HRMS (EI) exact mass calcd for [M+H]⁺ 205.0687, found 205.0680.

2-Propyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 8): Yield: 96%; ¹H NMR (500 MHz, CDCl₃): δ 9.62 (s, 1H), 7.32(m, 2H), 7.26 (m, 1H), 7.23 (s, 1H), 7.17 (m, 1H), 3.96 (q, 1H, J_1 = 5.0 Hz, J_2 = 8.5 Hz), 1.50 (m, 3H), 1.31 (m, 1H), 0.84 (t, 3H, J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 191.3, 144.5, 136.9, 134.5, 131.2, 130.7, 130.3, 128.0, 125.6, 37.1, 35.3, 18.9, 13.4; $[\alpha]_D^{23}$ = + 131.3 (c = 2.0, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): 94% ee, t_R = 14.47 (major), 16.31 (minor) min. HRMS (EI) exact mass calcd for [M+H]⁺ 219.0844, found 219.0839.

2-Pentyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 9): Yield: 94%; ¹H NMR (500 MHz, CDCl₃): δ 9.62 (s, 1H), 7.33(d, 2H, J = 8.0 Hz), 7.27 (m, 1H), 7.23 (s, 1H), 7.17 (m, 1H), 3.94 (t, 1H, J_1 = 5.0 Hz, J_2 = 9.0 Hz), 1.50 (m, 3H), 1.23 (m, 5H), 0.84 (t, 3H, J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 191.4, 144.5, 137.0, 134.5, 131.2, 130.8, 130.3, 128.0, 125.6, 35.6, 34.9, 31.1, 25.4, 22.4, 13.9; $[\alpha]_D^{23}$ = + 164.5 (c = 1.6, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, λ = 254 nm): 93% ee, t_R = 11.62 (major), 12.87 (minor) min. HRMS (EI) exact mass calcd for [M+H]⁺ 247.1157, found 247.1146.

6-Chloro-2-methyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 10): Yield: 91%; ¹H NMR (500 MHz, CDCl₃): δ 9.62 (s, 1H), 7.35 (d, 1H, J = 1.5 Hz), 7.27 (m, 2H), 7.16 (s, 1H), 4.09 (q, 1H, J = 7.0 Hz), 1.28 (d, 3H, J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 190.9, 142.6, 138.7, 132.3, 131.1, 131.0, 130.0, 129.3, 30.3, 21.9; $[\alpha]_D^{23}$ = + 50.4 (c = 1.0, CHCl₃); HPLC (Daicel CHIRALCEL OJ-H, Hexane/2-PrOH = 95:5, flow rate 0.5 mL/min, λ = 254 nm): 91% ee, t_R = 18.41 (minor), 21.87 (major) min. HRMS (EI) exact mass calcd for [M+H]⁺ 225.0140, found 225.0131.

6-Methoxy-2-methyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 11): Yield: 80%; ¹H NMR (500 MHz, CDCl₃): δ 9.61 (s, 1H), 7.25 (d, 1H, J = 7.0 Hz), 7.19 (m, 1H), 6.90 (m, 2H), 4.04 (q, 1H, J = 7.0 Hz), 3.83 (s, 3H), 1.27 (d, 3H, J = 7.0 Hz); ¹³C NMR (125 MHz,

CDCl₃): δ 191.2, 157.7, 144.1, 130.8, 129.2, 124.6, 117.9, 115.4, 55.5, 30.3, 21.6; $[\alpha]_D^{23} = +28.3$ ($c = 0.4$, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 85:15, flow rate 0.5 mL/min, $\lambda = 254$ nm): 93% ee, $t_R = 18.03$ (minor), 19.01 (major) min. HRMS (EI) exact mass calcd for $[M+H]^+$ 221.0636, found 221.0630.

2,6-Dimethyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 12): Yield: 97%; ¹H NMR (500 MHz, CDCl₃): δ 9.59 (s, 1H), 7.23 (d, 1H, $J = 8.0$ Hz), 7.18 (s, 1H), 7.17 (s, 1H), 7.11 (d, 1H, $J = 8.0$ Hz), 4.06 (q, 1H, $J = 7.0$ Hz), 2.34 (s, 3H), 1.27 (d, 3H, $J = 7.0$ Hz); ¹³C NMR (125 MHz, CDCl₃): δ 191.2, 144.2, 137.9, 135.4, 132.3, 131.3, 130.4, 129.7, 128.1, 30.3, 21.9, 20.8; $[\alpha]_D^{23} = +115.8$ ($c = 2.0$, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 95:5, flow rate 0.5 mL/min, $\lambda = 254$ nm): 90% ee, $t_R = 15.88$ (minor), 17.02 (major) min. HRMS (EI) exact mass calcd for $[M+H]^+$ 205.0687, found 205.0680.

3-Methyl-3H-4-thia-phenanthrene-2-carbaldehyde (Table 2, entry 13): Yield: 72%; ¹H NMR (500 MHz, CDCl₃): δ 9.68 (s, 1H), 8.26 (q, 1H, $J = 3.0$ Hz), 7.81 (q, 1H, $J = 3.0$ Hz), 7.63 (d, 1H, $J = 8.5$ Hz), 7.56 (q, 2H, $J = 3.0$ Hz), 7.40 (d, 1H, $J = 8.5$ Hz), 7.35 (s, 1H), 4.28 (q, 1H, $J = 7.0$ Hz), 1.28 (d, 3H, $J = 7.0$ Hz); ¹³C NMR (125 MHz, CDCl₃): δ 191.0, 144.6, 136.3, 134.7, 133.3, 131.3, 128.5, 128.0, 127.3, 127.0, 126.8, 125.6, 125.3, 30.3, 21.7; $[\alpha]_D^{23} = +442.1$ ($c = 1.0$, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, $\lambda = 254$ nm): 89% ee, $t_R = 18.53$ (minor), 20.49 (major) min.

2,5,7-Trimethyl-2H-thiochromene-3-carbaldehyde (Table 2, entry 14): Yield: 96%; ¹H NMR (500 MHz, CDCl₃): δ 9.60 (s, 1H), 7.40 (s, 1H), 7.03 (s, 1H), 6.84 (s, 1H), 4.04 (q, 1H, $J = 7.0$ Hz), 2.43 (s, 3H), 2.29 (s, 3H), 1.25 (d, 3H, $J = 7.0$ Hz); ¹³C NMR (125 MHz, CDCl₃): δ 191.2, 141.5, 141.0, 138.1, 136.8, 134.3, 128.7, 126.6, 126.1, 29.5, 21.6, 21.4, 19.5; $[\alpha]_D^{23} = +228.2$ ($c = 1.0$, CHCl₃); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 0.5 mL/min, $\lambda = 254$ nm): 89% ee, $t_R = 13.71$ (major), 16.12 (minor) min.

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Sample ID: h1135a

SPD-10AVP Chl-254nm Results				
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1	29.320	11026993	51.797	
2	33.410	10278180	48.243	
Totals		21305173	100.000	

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SPD-10AVP Chl-254nm Results				
PK #	RT	Area	Area %	
1	29.320	21026993	98.705	
2	33.410	6980979	3.190	
Totals		21725092	100.000	

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Sample ID: h1135a

SPD-10AVP Chl-254nm Results				
PK #	RT	Area	Area %	
1	29.320	11394933	47.788	
2	28.260	12345420	52.212	
Totals		23740353	100.000	

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SPD-10AVP Chl-254nm Results				
PK #	RT	Area	Area %	
1	29.320	8137808	7.222	
2	29.360	104238943	92.778	
Totals		112376751	100.000	

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SPD-10AVP Chl-254nm Results				
PK #	RT	Area	Area %	
1	27.150	37014765	49.840	
2	34.630	77301492	50.160	
Totals		114316257	100.000	

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SPD-10AVP Chl-254nm Results				
PK #	RT	Area	Area %	
1	27.880	58197650	96.885	
2	34.030	1880664	3.115	
Totals		60078314	100.000	

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Sample ID: h1k39a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	24.280	109727127	49.247	
2	26.860	113077440	50.753	
TOTALS		222800567	100.000	

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SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	24.320	102807598	32.432	
2	27.100	8344828	7.568	
TOTALS		111252426	100.000	

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SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	21.370	72339104	49.393	
2	25.610	73730141	50.607	
TOTALS		146069245	100.000	

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Sample ID: h1k39a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	21.670	9761097	3.810	
2	25.670	145431760	96.190	
TOTALS		155192857	100.000	

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Method Name: C:\ESIStart\Projects\Default\Method\Hs0114.met
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SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	17.350	82010208	49.807	
2	19.380	83692612	50.193	
TOTALS		165662820	100.000	

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SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	13.000	4318788	4.137	
2	15.320	100073760	95.863	
TOTALS		104392548	100.000	

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SPD-10Avp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	19.970	63897888	49.130	
2	21.920	65795060	50.810	
Totals		129692948	100.000	

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Method Name: C:\MSIStart\Projects\Default\Method\Hs0114.met
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PK #	RT	Area	Area %	
1	20.260	59681359	97.442	
2	23.110	1566791	2.558	
Totals		61248150	100.000	

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PK #	RT	Area	Area %	
1	14.007	32449910	50.773	
2	16.270	31463289	49.220	
Totals		63913199	100.000	

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SPD-10Avp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	14.470	118400254	96.032	
2	16.310	4051463	3.308	
Totals		122451717	100.000	

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SPD-10Avp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	11.640	22195522	49.743	
2	12.870	22395322	50.257	
Totals		44590844	100.000	

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Sample ID: hik58e

SPD-10Avp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	11.620	103123172	96.236	
2	12.870	3623962	3.464	
Totals		107557134	100.000	

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Method Name: C:\ESIStart\Projects\Default\Method\Hs0114.met
Data File: C:\ESIStart\Projects\WaiWang\h1k70a.dat
Date Acquired: 1/9/2006 10:22:19 AM Date Printed: 04/20/2006 11:05:15 PM
Sample ID: h1k70a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	18.380	43002996	48.475	
2	22.090	43082905	51.525	
Totals		84685901	100.000	

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Method Name: C:\ESIStart\Projects\Default\Method\Hs0114.met
Data File: C:\ESIStart\Projects\WaiWang\h1k74a1.dat
Date Acquired: 1/9/2006 11:10:30 AM Date Printed: 04/20/2006 11:07:23 PM
Sample ID: h1k74a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	18.410	18134687	4.263	
2	21.870	377336846	90.784	
3	27.300	20881259	4.953	
Totals		398351533	100.000	

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Method Name: C:\ESIStart\Projects\Default\Method\Hs0114.met
Data File: C:\ESIStart\Projects\WaiWang\h1k71a2.dat
Date Acquired: 1/9/2006 9:48:32 PM Date Printed: 04/20/2006 11:05:59 PM
Sample ID: h1k71a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	17.840	23478257	46.581	
2	19.000	26912030	53.419	
Totals		50389987	100.000	

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Method Name: C:\ESIStart\Projects\Default\Method\Hs0114.met
Data File: C:\ESIStart\Projects\WaiWang\h1k71a1.dat
Date Acquired: 1/9/2006 12:14:54 PM Date Printed: 04/20/2006 11:07:58 PM
Sample ID: h1k71a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	18.930	2338143	3.543	
2	19.010	63556870	96.457	
Totals		65895013	100.000	

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Method Name: C:\ESIStart\Projects\Default\Method\Hs0114.met
Data File: C:\ESIStart\Projects\WaiWang\h1k79a2.dat
Date Acquired: 1/11/2006 12:29:34 PM Date Printed: 04/20/2006 11:09:50 PM
Sample ID: h1k79a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	15.880	13010415	48.814	
2	17.270	76565637	51.186	
Totals		149582052	100.000	

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Method Name: C:\ESIStart\Projects\Default\Method\Hs0114.met
Data File: C:\ESIStart\Projects\WaiWang\h1k80a1.dat
Date Acquired: 1/11/2006 1:00:54 PM Date Printed: 04/20/2006 11:10:48 PM
Sample ID: h1k80a

SPD-10Amp Ch1-254nm Results				
PK #	RT	Area	Area %	
1	15.880	8355071	5.105	
2	17.020	155225202	94.895	
Totals		163580273	100.000