#### Supporting Information

# Chiral Binaphthyl-Derived Amine-Thiourea Organocatalyst Promoted Asymmetric Morita-Baylis-Hillman Reaction

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<sup>‡</sup>Department of Medicinal Chemistry, College of Pharmaceutical Engineer, East China University of Science & Technology, Shanghai 200237, P. R. China **General.** 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. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on Bruker Advance 500, and tetramethylsilane (TMS) was used as a reference. Data for <sup>1</sup>H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). High resolution mass spectra (HRMS) are obtained from Department of Chemistry, Ohio State University MS facility.

#### Procedures for preparation of binapthyl-based thiourea catalyst II.



Figure. Synthesis of binapthal-based thiourea catalyst II.



(*R*)-*N*-(1-(2-Aminonaphthalen-1-yl)naphthalen-2-yl)acetamide (1).<sup>1</sup> To a solution of (*R*)-(+)-1,1'-Binaphthyl-2,2'-diamine (284 mg, 1.0 mmol) and AcOH (0.6 mL, 10 mmol) in 10 mL of dried CH<sub>2</sub>Cl<sub>2</sub> was added acetic anhydride (104  $\mu$ L, 1.0 mmol) at 0 °C under N<sub>2</sub>. The resulting solution was stirred for overnight at room temperature, then 2*N* NaOH aqueous solution was added until pH  $\approx$  7. The reaction mixture was extracted by CH<sub>2</sub>Cl<sub>2</sub> (3 × 50 mL) and the combined organic phases were washed with saturated brine and dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography (ethyl acetate/hexane = 2/1) to afford a colorless oil in 77% yield (0.25 g, 0.77 mmol).



# (R)-N-(1-(2-(Dimethylamino)naphthalen-1-yl)naphthalen-2-yl)acetamide(2). $^2$ N-(1-(2-aminonaphthalen-1-yl)naphthalen-2-yl)acetamide1(0.25 g, 0.77 mmol) and aqueousformaldehyde(37%, 0.75ml, 9.0 mmol) were combined in 10 mL of THF and stirred for 15 min.

NaBH<sub>3</sub>CN (200 mg, 5.3 mmol) was added, followed 15 min later by AcOH (1.0 ml). The resulting solution was stirred for 4 h at room temperature, then 1*N* NaOH aqueous solution was added until pH  $\approx$  7. The reaction mixture was extracted by CH<sub>2</sub>Cl<sub>2</sub> (3 × 50 mL) and the combined organic phases were washed with saturated brine and dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography (ethyl acetate/hexane = 1/5) to afford a brown powder in quantitative yield (272 mg, 0.77 mmol).



(*R*)-1-(2-(Dimethylamino)naphthalen-1-yl)naphthalen-2-amine (3).<sup>2</sup> To a solution of *N*-(1-(2-(dimethylamino)naphthalen-1-yl)naphthalen-2-yl)acetamide 2 (0.18 g, 0.51 mmol) in 15 mL of EtOH was added 4*M* HCl (6 mL). The resulting solution was stirred for overnight at room temperature, then 1*N* NaOH aqueous solution was added until pH  $\approx$  7. The reaction mixture was extracted by CH<sub>2</sub>Cl<sub>2</sub> (3 × 50 mL) and the combined organic phases were washed with saturated brine and dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography (ethyl acetate/hexane = 1/10) to afford a colorless oil in 93% yield (148 mg, 0.47 mmol).



(*R*)-1-(3,5-Bis(trifluoromethyl)phenyl)-3-(1-(2-(dimethylamino)naphthalen-1-yl)naphthalen-2-yl)th iourea (II). To a solution of 1-(2-(dimethylamino)naphthalen-1-yl)naphthalen-2-amine 3 (36 mg, 0.12 mmol) in 2 mL of dried CH<sub>2</sub>Cl<sub>2</sub> was added 3,5-bis(trifluoromethyl)phenyl isothiocyanate (22 mg, 0.132 mmol) at 0 °C under N<sub>2</sub>. The resulting solution was stirred for overnight at room temperature. The reaction was concentrated *in vacuo* and the residue was purified by flash chromatography (ethyl acetate/hexane = 1/10) to afford a slight yellow solid in 91% yield (64 mg, 0.11 mmol).  $[\alpha]^{25}_{D} = -8.3$ (*c* = 0.5 in CHCl<sub>3</sub>); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS):  $\delta = 8.37$  (s, 1H), 8.06 (d, 1H, *J* = 8.5 Hz), 7.98 (d, 2H, J = 9.0 Hz), 7.82 (d, 1H, J = 8.0 Hz), 7.71 (d, 1H, J = 8.5 Hz), 7.56-7.50 (m, 5H), 7.41 (s, 1H), 7.36 (s, 2H), 7.26 (m, 2H), 7.09 (t, 1H, J = 7.5 Hz), 6.90 (d, 1H, J = 7.5 Hz), 2.59 (s, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>, TMS):  $\delta = 179.7$ , 149.9, 139.6, 134.0, 133.3, 133.2, 132.9, 132.0, 131.8, 131.6, 130.5, 130.0, 129.9, 128.5, 128.4, 127.5, 127.2, 126.8, 125.0, 124.6, 124.2, 123.9, 122.9, 121.8, 118.9, 44.0. HRMS (EI) calcd for C<sub>31</sub>H<sub>23</sub>N<sub>3</sub>SF<sub>6</sub> + Na 606.1409, obsd 606.1415.

**General Procedure for Baylis-Hillman Reaction** (Table 2): The catalyst amino-thiourea **IV** (10 mg, 0.019 mmol) was added to a vial containing 2-cyclohexen-1-one **1a** (36  $\mu$ L, 0.374 mmol) in CH<sub>3</sub>CN (1 mL) at 0 °C. The mixture was stirred vigorously for 10 min, and then aldehyde **2** (27  $\mu$ L, 0.187 mmol) was added. After 48-120 h stirring, the reaction mixture was concentrated *in vacuo*. The residue was then purified by flash silica gel chromatography, eluting with EtOAc/hexane (1:10 to 1:2) to afford a clear oil



**2-((***R***)-1-Hydroxy-3-phenylpropyl)cyclohex-2-enone<sup>3</sup>** (Table 2, entry 1): The reaction was carried out following the general procedure to provide a clear oil (35 mg, 80%).  $[\alpha]^{25}_{D} = -49.4$  (c = 0.5, CHCl<sub>3</sub>); HPLC (Daicel CHIRALCEL OD-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/min,  $\lambda = 254$  nm);  $t_{R} = 12.62$  (minor), 9.62 (major) min.



**2-((***R***)-1-Hydroxy-3-methylbutyl)cyclohex-2-enone<sup>3</sup>** (Table 2, entry 2): The reaction was carried out following the general procedure to provide a clear oil (25 mg, 72%).  $[\alpha]^{25}{}_{D} = -25.0$  (c = 0.2, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/ min,  $\lambda = 254$  nm);  $t_{R} = 6.92$  (minor), 8.96 (major) min.



**2-((***R***)-1-Hydroxypentyl)cyclohex-2-enone** (Table 2, entry 3): The reaction was carried out following the general procedure to provide a clear oil (31 mg, 84%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  6.87 (t, 1H, J = 4.0 Hz), 4.28 (brs, 1H), 2.90 (brs, 1H), 2.50-2.38 (m, 4H), 2.05-1.95 (m, 2H), 1.70-1.56 (m, 2H), 1.45-1.22 (m, 4H), 0.91 (t, 3H, J = 6.5 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  200.7, 145.8, 140.9, 71.8, 38.7, 35.9, 28.2, 25.7, 22.6, 22.5, 14.0;  $[\alpha]^{25}{}_{D} = -28.4$  (c = 0.4, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/min,  $\lambda = 254$  nm);  $t_{R} = 7.77$  (minor), 11.25 (major) min. HRMS (EI) calcd for [C<sub>11</sub>H<sub>18</sub>O<sub>2</sub> + Na] 205.1199, obsd 205.1206.

 $\cap$ OH *n*-C<sub>5</sub>H<sub>11</sub>

**2-((***R***)-1-Hydroxyhexyl)cyclohex-2-enone** (Table 2, entry 4): The reaction was carried out following the general procedure to provide a clear oil (25 mg, 75%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  6.87 (t, 1H, *J* = 4.0 Hz), 4.29 (brs, 1H), 2.90 (brs, 1H), 2.49-2.38 (m, 4H), 2.04-1.95 (m, 2H), 1.70-1.56 (m, 2H), 1.48-1.36 (m, 1H), 1.35-1.23 (m, 5H), 0.88 (t, 3H, *J* = 6.0 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  200.7, 145.8, 140.9, 71.8, 38.7, 36.2, 31.6, 25.7, 25.6, 22.6, 14.0;  $[\alpha]^{25}_{D} = -44.0$  (*c* = 0.5, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/min,  $\lambda$  = 254 nm); *t*<sub>R</sub> = 7.42 (minor), 10.10 (major) min. HRMS (EI) calcd for [C<sub>12</sub>H<sub>20</sub>O<sub>2</sub> + Na] 219.1355, obsd 219.1353.



**2-((***R***)-1-Hydroxyheptyl)cyclohex-2-enone** (Table 2, entry 5): The reaction was carried out following the general procedure to provide a clear oil (28 mg, 71%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  6.87 (t, 1H, *J* = 4.0 Hz), 4.28 (dd, 1H, *J* = 13.0 Hz, 6.0 Hz), 2.88 (d, 1H, 7.0 Hz), 2.48-2.38 (m, 4H), 2.04-1.95 (m, 2H), 1.70-1.56 (m, 2H), 1.45-1.36 (m, 1H), 1.35-1.23 (m, 7H), 0.88 (t, 3H, *J* = 7.0 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  200.7, 145.8, 140.9, 71.9, 38.7, 36.2, 31.8, 29.1, 26.0, 25.7, 25.6, 14.1;  $[\alpha]^{25}_{D} = -33.0$  (*c* = 0.1, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/ min,  $\lambda$  = 254 nm); *t*<sub>R</sub> = 6.71 (minor), 8.79 (major) min. HRMS (EI) calcd for [C<sub>13</sub>H<sub>22</sub>O<sub>2</sub> + Na] 233.1512, obsd 233.1515.



**2-((***R***)-1-Hydroxyoctyl)cyclohex-2-enone** (Table 2, entry 6): The reaction was carried out following the general procedure to provide a clear oil (31 mg, 74%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  6.87 (t, 1H, *J* = 4.0 Hz), 4.28 (dd, 1H, *J* = 12.0 Hz, 6.0 Hz), 2.89 (d, 1H, 6.5 Hz), 2.47-2.38 (m, 4H), 2.04-1.95 (m, 2H), 1.70-1.56 (m, 2H), 1.45-1.36 (m, 1H), 1.35-1.23 (m, 9H), 0.88 (t, 3H, *J* = 6.5 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  200.7, 145.8, 140.9, 71.8, 38.7, 36.2, 31.8, 29.4, 29.2, 26.0, 25.7, 22.6, 22.5, 14.1;  $[\alpha]^{25}_{D} = -28.2 (c = 0.5, CHCl_3)$ ; HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/min,  $\lambda = 254$  nm);  $t_R = 6.24$  (minor), 8.13 (major) min. HRMS (EI) calcd for  $[C_{14}H_{24}O_2 + Na]$  247.1668, obsd 247.1674.



**2-((***R***,***Z***)-1-Hydroxyhept-4-enyl)cyclohex-2-enone<sup>3</sup>** (Table 2, entry 7): The reaction was carried out following the general procedure to provide a clear oil (32 mg, 82%).  $[\alpha]^{25}_{D} = -26.5$  (c = 0.4, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/ min,  $\lambda = 254$  nm);  $t_{R} = 7.72$  (minor), 10.67 (major) min.



**2-((***R***)-1-Hydroxy-2-methylpropyl)cyclohex-2-enone<sup>3</sup>** (Table 2, entry 8): The reaction was carried out following the general procedure to provide a clear oil (20 mg, 63%).  $[\alpha]^{25}_{D} = -48.0$  (c = 0.1, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/ min,  $\lambda = 254$  nm);  $t_{R} = 9.11$  (minor), 15.11 (major) min.



**2-((***R***)-Cyclopentyl(hydroxy)methyl)cyclohex-2-enone<sup>4</sup>** (Table 2, entry 9): The reaction was carried out following the general procedure to provide a clear oil (26 mg, 76%).  $[\alpha]^{25}{}_{\rm D} = -57.0$  (c = 0.7, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/ min,  $\lambda = 254$  nm);  $t_{\rm R} = 10.01$  (minor), 15.45 (major) min.



**2-((***R***)-Cyclohexyl(hydroxy)methyl)cyclohex-2-enone<sup>3</sup>** (Table 2, entry 10): The reaction was carried out following the general procedure to provide a clear oil (26 mg, 67%).  $[\alpha]^{25}{}_{D} = -73.7$  (c = 0.3, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 90:10, flow rate 1.0 ml/min,  $\lambda = 254$  nm);  $t_{R} = 10.35$  (minor), 14.07 (major) min.



**2-((***S***)-(2-chlorophenyl)(hydroxy)methyl)cyclohex-2-enone** (Table 2, entry 11): The reaction was carried out following the general procedure to provide a clear oil (24 mg, 55%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  7.33-7.27 (m, 4H), 6.74 (t, 1H, *J* = 4.0 Hz), 5.51 (bs, 1H), 2.48-2.37 (m, 4H), 2.03-1.96 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  200.8, 147.9, 139.3, 138.4, 132.4, 129.3, 128.7, 128.2, 127.0, 68.8, 38.5, 25.8, 24.5; [ $\alpha$ ]<sup>25</sup><sub>D</sub> = -8.5° (*c* = 0.3, CHCl<sub>3</sub>); HPLC (Daicel CHIRALPAK AS-H, Hexane/2-PrOH = 85:15, flow rate 1.0 ml/ min,  $\lambda$  = 254 nm); *t*<sub>R</sub> = 9.91 (minor), 14.98 (major) min. HRMS (EI) calcd for [C<sub>13</sub>H<sub>13</sub>O<sub>2</sub>Cl + Na] 259.0496, obsd 259.0491.

#### **Reference:**

- (1) Pieraccini, S.; Gottarelli, G.; Labruto, R.; Masiero, S.; Pandoli, O.; Spada, G. P. *Chem. Eur. J.* **2004**, *10*, 5632-5639.
- (2) Wang, C. -J.; Shi, M. J. Org. Chem 2003, 68, 6229-6234.
- (3) McDougal, N. T.; Schaus, S. E. J. Am. Chem. Soc. 2005, 125, 12094-12095.
- (4) Yoshihiro, S.; Aya, T.; Yuichi, H.; Kazuo, N. Tetrahedron Lett. 2004, 45, 5589-5592.

























































C:\EZStart\Projects\Default\Method\WeiWang19.met Method Name: C:\EZStart\Projects\WeiWang\jwh87e2.dat Data File: 3/12/2005 7:13:41 PM Date Printed: 07/11/2005 03:13:23 PM Date Acquired: jwh87e Sample ID: OH 150 150 4.060 227969 100 mVolts 100 460 4744665 mVolts 130 4500043 50 50 AL 0 0 0 25 30 20 15 5 10 0 Minutes SPD-10Avp Ch1-254nm Results Area % RT Area Pk # 47.506 4500043 9.130 2 50.088 4744665 11.460 3

Totals	9244708	97.593

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SPD-10Avp	
Ch1-254nm	Results

 Pk #	RT	Area	Area %
2	9.620	4287904	81.416
3	12.620	468488	8.895

Totals	4756392 90.311

Method Name:	C:\EZStart\Projects\D	efault\Method\WeiWang19.met
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Date Acquired: PM	3/17/2005 10:42:10 PM	Date Printed: 07/11/2005 03:10:58
Sample ID:	jwh103e	





SPD-10Avp Ch1-245nm Results			
Pk #	RT	Area	Area %
2	6.810	4965916	32.669
3	8.740	4985126	32.795

	· Totals		9951042	65.464
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SPD-10Avp		,	
Ch1-254nm Results Pk #	RT	Area	Area %
2 3	6.920 8.960	49873 422188	6.396 54.140

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SPD-10Avp Ch1-254nm Results Pk #	RT	Area	Area %
2	7.420	2258560	45.549
. 3	10.420	2270609	45.792

Totals		
	4529169	91.340

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SPD-10Avp	
Ch1-254nm	Resul

Ch1-254nm Res	ults Pk #	RT	Area	Area %
	2	7.770	98330	7.219
	3	-11.250	942332	69.179

	eter.	
Totals	A Commented and the second	
	1040662	76.397

Method Name: C:\EZStart\Projects\Default\Method\WeiWang19.met Data File: C:\EZStart\Projects\WeiWang\jwh104e1.dat Date Acquired: 3/17/2005 11:15:44 PM Date Printed: 07/11/2005 03:17:00 PM Sample ID: jwh104e





SPD-10Avp Ch1-254nm Results			
Pk #	RT	Area	Area %
2	7.050	1830613	44.420
3	- 9.300	1841780	44.691

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IOCAIS	3672393	89.111
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Method Name:C:\EZStart\Projects\Default\Method\jw1.metData File:C:\EZStart\Projects\WeiWang\jwi8e1.datDate Acquired:4/7/2005 4:20:09 PMDate Printed:07/11/2005 03:18:30 PMSample ID:jwi8e



SPD-10Avp Ch1-254nm Results

Pk #	RT	Area	Area %
2	7.420	97688	7.586
3	Ĭ0.100	859542	66.746

Totals	957230	74.332
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SPD-10Avp	
Ch1-254nm	Results

Pk #	RT	Area	Area %
2	6.670	982543	41.457
3	8.610	983708	41.506

	Totals		1966251	82.962
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Method Name:C:\EZStart\Projects\Default\Method\WeiWang19.metData File:C:\EZStart\Projects\WeiWang\jwh112e1.datDate Acquired:3/18/2005 1:36:25 AMDate Printed: 07/11/2005 03:20:59 PMSample ID:jwh112e





SPD-10Avp Ch1-254nm Results			
Pk #	RT	Area	Area 🗞
1	6.170	1690627	49.825
2	7.960	1702481	50.175

Totals	3393108 100.000

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0112 20111	Pk #	RT	Area	Area %
	2	6.240	212890	8.090
	3	8.130	2049132	77.869

Totals	2262022 85.959





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Totals	• 2123333		87.874



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SPD-10Avp Ch1-254nm	Results Pk #	RT	Area	Area %
	2	. 10.010	49831	3.942
	3	15.450	878248	69.470

Totals	928079	73.411

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Method Name: C:\EZStart\Projects\Default\Method\jw4.met Data File: C:\EZStart\Projects\WeiWang\jwi5e1.dat Date Acquired: 4/8/2005 9:53:37 PM Date Printed: 07/11/2005 11:41:13 PM Analysis: System





Ch1-254nm Results Pk #	Name	RT	Area	ESTD conc	Chan
3	Peak @ 9.110	9.110	10272	0.000	SPD-10Avp
	Minutes				Ch1-254nm
4	Peak @ 15.110	15.110	316544	0.000	SPD-10Avp
	Minutes				Ch1-254nm
. Totals					
, 10cars			326816	0.000	

Method Name:C:\EZStart\Projects\Default\Method\WeiWang19.metData File:C:\EZStart\Projects\WeiWang\jwh105e2.datDate Acquired:3/17/2005 11:47:25 PMDate Printed:07/11/2005 03:02:58PMSample ID:jwh105e





SPD-10Avp Ch1-254nm	Results Pk #	RT	Area	Area %
	. 1	9.590	622315	50.064
	2	14.290	620722	49.936
	<b>-</b>			wante new market of the second
•	Totals		1243037	100.000

Method Name:C:\EZStart\Projects\Default\Method\jw5.metData File:C:\EZStart\Projects\WeiWang\jwi12e1.datDate Acquired:4/14/2005 1:05:50 PMDate Printed:07/11/2005 03:05:19 PMSample ID:jwi12e





SPD-10Avp Ch1-254nm	Results Pk #	RT	Area	Area %
	2	. 10.010	49831	3.942
	3	15.450	878248	69.470

Totals	928079	73.411

Method Name:C:\EZStart\Projects\Default\Method\WeiWang19.metData File:C:\EZStart\Projects\WeiWang\jwh91e1.datDate Acquired:3/13/2005 11:26:30 PMDate Printed:PMSample ID:jwh91e





SPD-10Avp	
Ch1-235nm	Results

 Pk #	RT	Area	Area %
1	.9.830	1487491	49.251
2	13.120	1532752	50.749

-		
Totals	3020243	100.000







SPD-10Avp Ch1-245nm Re	esults Pk #	RT	Area	Area %
	2	10.350	257912	2.929
	3	14.070	5140227	58.368

Totals	5398139 61.296