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# Asymmetric Synthesis of Quaternary α-Trifluoromethyl α-Amino Acids by Ir-catalyzed Allylation Followed by Kinetic Resolution

Xi-Shang Sun, Qiu Ou-Yang, Shi-Ming Xu, Xing-Heng Wang, Hai-Yan Tao, Lung Wa Chung,\*and Chun-Jiang Wang\*

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#### 1. General remarks

<sup>1</sup>H NMR spectra were recorded on a Bruker 400 MHz spectrometer in CDCl<sub>3</sub>. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. <sup>13</sup>C NMR spectra were recorded on a Bruker 100 MHz spectrometer in CDCl<sub>3</sub>. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. <sup>19</sup>F NMR spectra were recorded on a Bruker 376 MHz spectrometer in CDCl<sub>3</sub>. Chemical shifts are reported in ppm with the internal CF<sub>3</sub>COOH signal at -76.55 ppm. The data are reported as (s = single, d = double, t = triple, q = quartet, m = multiple or unresolved, brs = broad single, coupling constant(s) in Hz, integration). 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 AD-H, chiralpak IE, chiralpak AS, chiralcel OJ, and chiralcel OD column with hexane and *i*-PrOH. Compounds 1<sup>1</sup> and Allylic carbonates 2<sup>2</sup> were prepared according to the literature procedure. Chiral ligands (*R*,*R*<sub>*p*</sub>)-L1, (*S*,*S*<sub>*p*</sub>)-L1, (*S*,*S*,*S*)-L2 and (*R*,*R*,*R*)-L2 were prepared according to the literature procedure.<sup>3,4</sup> The absolute configurations of **31** and **41** were determined based on X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.

# 2. General Procedure for Sequential Ir-Catalyzed Allylation of α-Trifluoromethyl Aldimine Esters Followed by Kinetic Resolution via 2-aza-Cope Rearrangement



In a 25 mL nitrogen-filled dry Schlenk tube,  $[Ir(COD)Cl]_2$  (6.7 mg, 0.01 mmol), phosphoramidite ligand (*S*,*S*,*S*)-**L2** (10.8 mg, 0.02 mmol), degassed THF (0.5 mL) and degassed propylamine (0.5 mL) were added. After stirring at 50 °C for 30 mins, the reaction was concentrated via rotary evaporation under reduced pressure to give the iridium complex as a pale yellow solid.<sup>5</sup> Then,  $\alpha$ -Trifluoromethyl aldimine esters **1** (0.5 mmol), allylic carbonates **2** 

(0.4 mmol), Cs<sub>2</sub>CO<sub>3</sub> (0.5 mol) and dry benzene (2 mL) was added into the nitrogen-refilled Schlenk tube. Once starting material was consumed (monitored by TLC), the reaction mixture was concentrated via rotary evaporation under reduced pressure, and the base was removed through a short silica gel plug (PE / EA = 50:1), then the crude product was diluted in 1 ml benzene and heated to 50 °C. Once one of the diastereomers was consumed (monitored by <sup>19</sup>F NMR), a solution of NH<sub>2</sub>OH•AcOH in methanol [1 M, 20 mL, prepared from NH<sub>2</sub>OH•HCl, NaOH (solid, 1 equiv.), and AcOH (1 equiv.) in methanol] was added to the reaction mixture. After stirring at 15 °C for 2 h, the reaction mixture was concentrated via rotary evaporation under reduced pressure, and then purified by flash chromatography on silica gel (PE / EA = 10:1) to give the allylation products **3** and rearranged products **5**.

3a

#### Ethyl (2*R*,3*S*)-2-amino-3-phenyl-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 56% yield.  $[\alpha]^{25}_{D} = 8.7$  (*c* 0.93, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.39 – 7.14 (m, 5H), 6.30 – 6.05 (m, 1H), 5.21 – 4.98 (m, 2H), 4.32 – 4.12 (m, 2H), 4.04 (d, *J* = 9.4 Hz, 1H), 1.83 (brs, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.9, 136.5, 134.1, 128.1, 127.5, 126.6, 123.6 (q, *J* = 288.3 Hz), 117.6, 67.2 (q, *J* = 25.6 Hz), 61.7, 51.2, 12.9. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.66 (s). HRMS Calcd. For C<sub>14</sub>H<sub>17</sub>F<sub>3</sub>NO<sub>2</sub> ([M+H]<sup>+</sup>): 288.1206, found: 288.1193. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 5.11 and 5.64 min.

#### (*S*,*E*)-1,4-diphenylbut-3-en-1-amine (Known compound):

White solid; 35% yield, m.p. 70-72 °C;  $[\alpha]^{25}_{D} = -26.1$  (*c* 0.9, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 – 7.12 (m, 10H), 6.48 (d, *J* = 15.8 Hz, 1H), 6.27 – 6.00 (m, 1H), 4.08 (dd, *J* = 7.9, 5.3 Hz, 1H), 2.70 – 2.42 (m, 2H), 1.68 (s, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.8, 137.4, 132.9, 128.5, 128.4, 127.2, 127.1, 127.0, 126.4, 126.1, 55.8, 43.5. HRMS Calcd. For C<sub>16</sub>H<sub>18</sub>N

([M+H]<sup>+</sup>): 224.1434, found: 224.1426. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 6.76 and 9.07 min.



#### Ethyl (2*R*,3*S*)-2-amino-3-(*p*-tolyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 59% yield.  $[\alpha]^{25}_{D} = 27.5$  (*c* 1.2, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.30 – 7.09 (m, 4H), 6.25 (ddd, *J* = 17.2, 10.0, 9.6 Hz, 1H), 5.23 – 5.08 (m, 2H), 4.38 – 4.19 (m, 2H), 4.08 (d, *J* = 9.4 Hz, 1H), 2.32 (s, 3H), 1.89 (brs, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.0, 136.3, 134.3, 133.4, 128.3, 127.9, 123.6 (q, *J* = 288.4 Hz), 117.4, 67.2 (q, *J* = 25.4 Hz), 61.7, 50.8, 20.0, 12.3. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.61 (s). HRMS Calcd. For C<sub>15</sub>H<sub>19</sub>F<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 302.1362, found: 302.1352. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 4.94 and 5.21 min.



#### (*S*,*E*)-1-phenyl-4-(*p*-tolyl)but-3-en-1-amine:

White solid; 30% yield, m.p. 65-67 °C;  $[\alpha]^{25}_{D} = -23.6$  (*c* 0.56, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 – 7.30 (m, 4H), 7.29 – 7.17 (m, 3H), 7.10 (d, *J* = 7.9 Hz, 2H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.18 – 6.01 (m, 1H), 4.07 (dd, *J* = 8.0, 5.2 Hz, 1H), 2.68 – 2.42 (m, 2H), 2.32 (s, 3H), 1.74 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.7, 137.0, 134.6, 132.8, 129.2, 128.5, 127.1, 126.4, 126.0, 125. 9, 55.8, 43.4, 21.2. HRMS Calcd. For C<sub>17</sub>H<sub>20</sub>N ([M+H]<sup>+</sup>): 238.1590, found: 238.1584. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 6.37 and 8.43 min.



#### Ethyl (2*R*,3*S*)-2-amino-3-(*m*-tolyl)-2-(trifluoromethyl)pent-4-enoate:

Colourless oil; 53% yield.  $[\alpha]^{25}_{D} = 16.8 (c \ 1.02, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.36 – 6.99 (m, 4H), 6.26 (ddd, J = 16.8, 9.6, 9.6 Hz, 1H), 5.24 – 5.08 (m, 2H), 4.39 – 4.18 (m, 2H), 4.07 (d, J = 9.5 Hz, 1H), 2.34 (s, 3H), 1.91 (brs, 2H), 1.33 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  168.0, 138.2, 137.5, 135.3, 129.8, 128.4, 128.3, 126.2, 124.6(q, J = 288.3 Hz), 118.5, 68.2(q, J = 25.5 Hz), 62.7, 52.2, 21.5, 14.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.66 (s). HRMS Calcd. For C<sub>15</sub>H<sub>19</sub>F<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 302.1362, found: 302.1352. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 4.71 and 5.35 min.



#### (*S*,*E*)-1-phenyl-4-(*m*-tolyl)but-3-en-1-amine:

Thick oil; 35% yield.  $[\alpha]^{25}_{D} = -20.1$  (*c* 1.21, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.42 – 7.10 (m, 8H), 7.02 (d, *J* = 7.2 Hz, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.22 – 6.05 (m, 1H), 4.06 (dd, *J* = 8.0, 5.2 Hz, 1H), 2.72 – 2.42 (m, 2H), 2.32 (s, 3H), 1.70 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.8, 138.1, 137.3, 133.0, 128.5, 128.4, 128.0, 127.1, 126.9, 126.8, 126.4, 123.29, 55.8, 43.5, 21.4. HRMS Calcd. For C<sub>17</sub>H<sub>20</sub>N ([M+H]<sup>+</sup>): 238.1590, found: 238.1586. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 7.01 and 8.80 min.



3d



Colorless oil; 66% yield.  $[\alpha]^{25}_{D} = 83.3$  (*c* 0.33, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.34 – 7.19 (m, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 6.23 (ddd, *J* = 16.8, 9.6, 9.6 Hz, 1H), 5.15 (dd, *J* = 9.5, 8.2 Hz, 2H), 4.40 – 4.18 (m, 2H), 4.08 (d, *J* = 9.4 Hz, 1H), 3.79 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H).<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  168.1, 159.0, 135.3, 130.1, 129.4, 124.63 (q, *J* = 287.2 Hz), 118.3, 114.0, 68.23 (q, *J* = 25.1 Hz, 1H), 62.7, 55.2, 51.4, 14.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.61 (s). HRMS Calcd. For C<sub>15</sub>H<sub>19</sub>F<sub>3</sub>NO<sub>3</sub>([M+H]<sup>+</sup>): 318.1312, found: 318.1304. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 7.00 and 7.46 min.



#### (*S*,*E*)-4-(4-methoxyphenyl)-1-phenylbut-3-en-1-amine:

White solid; 24% yield, m.p. 70-72 °C;  $[\alpha]^{25}_{D} = -15.4$  (*c* 0.72, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.42 – 7.30 (m, 4H), 7.26 (m, 4H), 6.83 (d, *J* = 8.7 Hz, 2H), 6.42 (d, *J* = 15.8 Hz, 1H), 6.10 – 5.90 (m, 1H), 4.06 (dd, *J* = 7.9, 5.4 Hz, 1H), 3.79 (s, 3H), 2.69 – 2.46 (m, 4H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  158.9, 145.3, 132.4, 130.2, 128.5, 127.3, 127.2, 126.4, 124.5, 113.9, 55.8, 55.3, 43.1. HRMS Calcd. For C<sub>17</sub>H<sub>20</sub>NO ([M+H]<sup>+</sup>): 254.1539, found: 254.1534. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 7.36 and 8.79 min.



#### Ethyl (2R,3S)-2-amino-3-(3-methoxyphenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 53% yield.  $[\alpha]^{25}_{D} = 9.7$  (*c* 0.63, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 – 7.17 (m, 1H), 6.92 (dd, *J* = 11.6, 5.0 Hz, 2H), 6.86 – 6.79 (m, 1H), 6.25 (ddd, *J* = 16.8, 9.6, 9.6 Hz, 1H), 5.26 – 5.07 (m, 2H), 4.37 – 4.20 (m, 2H), 4.07 (d, *J* = 9.5 Hz, 1H), 3.80 (s, 3H), 1.93 (brs, 2H), 1.34 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.9, 159.6, 139.1, 135.1, 129.5, 124.6 (q, *J* = 288.5 Hz), 121.4, 118.6, 115.1, 112.8, 68.3 (q, *J* = 25.7 Hz), 62.8, 55.2,

52.2, 14.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.71 (s). HRMS Calcd. For C<sub>15</sub>H<sub>19</sub>F<sub>3</sub>NO<sub>3</sub>([M+H]<sup>+</sup>): 318.1312, found: 318.1305. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 7.00 and 7.46 min.



#### (*S*,*E*)-4-(3-methoxyphenyl)-1-phenylbut-3-en-1-amine:

Thick oil; 36% yield.  $[\alpha]^{25}_{D} = -17.1$  (*c* 0.61, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.44 – 7.30 (m, 4H), 7.30 – 7.16 (m, 2H), 6.93 (d, *J* = 7.7 Hz, 1H), 6.87 (d, *J* = 2.1 Hz, 1H), 6.77 (dd, *J* = 8.1, 2.1 Hz, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.24 – 6.07 (m, 1H), 4.08 (dd, *J* = 8.0, 5.3 Hz, 1H), 3.80 (s, 3H), 2.67 – 2.42 (m, 2H), 1.91 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  159. 8, 145.7, 138.8, 132.8, 129.5, 128.5, 127.4, 127.1, 126.3, 118.8, 112. 8, 111.5, 55. 8, 55.2, 43.4. HRMS Calcd. For C<sub>17</sub>H<sub>20</sub>NO ([M+H]<sup>+</sup>): 254.1539, found: 254.1534. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 9.10 and 12.21 min.



3f

# Ethyl (2*R*,3*S*)-2-amino-3-(benzo[d][1,3]dioxol-5-yl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 73% yield.  $[\alpha]^{25}_{D} = 20.2 (c 1.11, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.87 (d, J = 1.2 Hz, 1H), 6.78 (dd, J = 9.8, 4.7 Hz, 2H), 6.19 (ddd, J = 16.8, 10.0, 10.0 Hz, 1H), 5.94 (s, 2H), 5.16 (dd, J = 9.6, 8.2 Hz, 2H), 4.37 – 4.20 (m, 2H), 4.04 (d, J = 9.3 Hz, 1H), 1.90 (brs, 2H), 1.33 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.0, 147.7, 147.0, 135.2, 131.2, 124.59 (q, J = 288.5 Hz), 122.5, 118.4, 109.3, 108.4, 101.1, 68.2 (q, J = 25.4 Hz), 62.8, 51.8, 14.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.71 (s). HRMS Calcd. For C<sub>19</sub>H<sub>17</sub>F<sub>3</sub>NO<sub>4</sub>([M+H]<sup>+</sup>): 332.1104, found: 332.1095. The product was analyzed by HPLC to determine the enantiomeric

excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 8.20 and 8.80 min.



#### (*S*,*E*)-4-(benzo[d][1,3]dioxol-5-yl)-1-phenylbut-3-en-1-amine:

Thick oil; 22% yield.  $[\alpha]^{25}_{D} = -13.2$  (*c* 0.62, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 – 7.31 (m, 4H), 7.29 – 7.22 (m, 1H), 6.87 (s, 1H), 6.81 – 6.68 (m, 2H), 6.39 (d, *J* = 15.7 Hz, 1H), 6.06 – 5.88 (m, 3H), 4.06 (dd, *J* = 8.0, 5.3 Hz, 1H), 2.68 – 2.39 (m, 2H), 1.62 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  148.0, 146.9, 145.8, 132.4, 131.9, 128.5, 127.1, 126.3, 125.2, 120.6, 108.3, 105.5, 101.0, 55.8, 43.4. HRMS Calcd. For C<sub>17</sub>H<sub>18</sub>NO<sub>2</sub> ([M+H]<sup>+</sup>): 268.1332, found: 268.1326. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 8.67 and 10.42 min.



3g

#### Ethyl (2R,3S)-2-amino-3-(4-chlorophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 44% yield.  $[\alpha]^{25}{}_{D} = 35.9 \ (c \ 1.28, CH_2Cl_2).$  <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.30 (s, 4H), 6.31 – 6.12 (m, 1H), 5.25 – 5.10 (m, 2H), 4.39 – 4.20 (m, 2H), 4.09 (d, J = 9.3 Hz, 1H), 1.89 (brs, 2H), 1.33 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.8, 135.1, 133.8, 132.5, 129.5, 127.7, 123.5 (q, J = 288.4 Hz), 118.0, 67.1 (q, J = 25.7 Hz), 61.9, 50.4, 12.9. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.64 (s). HRMS Calcd. For C<sub>14</sub>H<sub>16</sub>ClF<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 322.0816, found: 328.0806. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 5.48 and 5.83 min.



#### (*S*,*E*)-4-(4-chlorophenyl)-1-phenylbut-3-en-1-amine:

White solid; 30% yield m.p. 85-88 °C;  $[\alpha]^{25}_{D} = -19.1$  (*c* 0.35, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 – 7.36 (m, 4H), 7.29 – 7.20 (m, 5H), 6.42 (d, *J* = 15.8 Hz, 1H), 6.23 – 6.05 (m, 1H), 4.08 (dd, *J* = 7.9, 5.4 Hz, 1H), 2.67 – 2.43 (m, 2H), 1.65 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.7, 135.9, 132.8, 131.6, 128.7, 128.5, 127.8, 127.3, 127.1, 126.3, 55.8, 43.4. HRMS Calcd. For C<sub>16</sub>H<sub>17</sub>ClN([M+H]<sup>+</sup>): 258.1044, found: 258.1038. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 6.70 and 7.48 min.



#### Ethyl (2*R*,3*S*)-2-amino-3-(3-chlorophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil;41% yield.  $[\alpha]^{25}_{D} = 26.2 \ (c \ 1.15, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.37 (s, 1H), 7.30 – 7.21 (m, 3H), 6.31 – 6.13 (m, 1H), 5.18 (dd, *J* = 13.5, 2.1 Hz, 2H), 4.38 – 4.21 (m, 2H), 4.08 (d, *J* = 9.4 Hz, 1H), 1.91 (brs, 2H), 1.34 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.7, 138.7, 133.6, 133.2, 128.7, 128.2, 126. 8, 126.4, 123.4 (q, *J* = 288.5 Hz), 118.1, 67.1 (q, *J* = 25.8 Hz), 61.9, 50.8, 12.9. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.70 (s). HRMS Calcd. For C<sub>14</sub>H<sub>16</sub>ClF<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 322.0816, found: 328.0806. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 4.66 and 5.22 min.



#### (S,E)-4-(3-chlorophenyl)-1-phenylbut-3-en-1-amine:

White solid; 29% yield, m.p. 60-62 °C;  $[\alpha]^{25}_{D} = -12.5$  (*c* 0.67, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.41 – 7.14 (m, 9H), 6.40 (d, *J* = 15.8 Hz, 1H), 6.28 – 6.05 (m, 1H), 4.08 (dd, *J* = 7.8,

5.5 Hz, 1H), 2.68 – 2.46 (m, 2H), 1.80 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.4, 139.2, 134.5, 131.6, 129.7, 128.6, 128.5, 127.2, 127.1, 126.3, 126.0, 124.4, 55.8, 43.3. HRMS Calcd. For C<sub>16</sub>H<sub>17</sub>ClN([M+H]<sup>+</sup>): 258.1044, found: 258.1034. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 7.05 and 8.46 min.



### Ethyl (2R,3S)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 49% yield.  $[\alpha]^{25}{}_{D} = 31.5 (c \ 1.17, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 (d, J = 1.9 Hz, 1H), 7.40 (d, J = 8.3 Hz, 1H), 7.21 (dd, J = 8.3, 1.8 Hz, 1H), 6.16 (ddd, J = 16.8, 10.0, 10.0 Hz, 1H), 5.19 (dd, J = 13.8, 5.1 Hz, 2H), 4.39 – 4.20 (m, 2H), 4.06 (d, J = 9.3 Hz, 1H), 1.91 (brs, 2H), 1.34 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.6, 138.0, 134.4, 132.5, 131.8, 131.0, 130.4, 128.6, 124.4 (q, J = 288.4 Hz), 119.5, 68.0 (q, J = 25.7 Hz), 63.1, 51.2, 14.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.65 (s). HRMS Calcd. For C<sub>14</sub>H<sub>15</sub>Cl<sub>2</sub>F<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 356.0426, found: 352.0415. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 4.86 and 5.23 min.



#### (*S*,*E*)-4-(3,4-dichlorophenyl)-1-phenylbut-3-en-1-amine:

White solid; 31% yield, m.p. 66-68 °C;  $[\alpha]^{25}_{D} = -6.2$  (*c* 0.77, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.47 – 7.31 (m, 6H), 7.29 – 7.25 (m, 1H), 7.12 (dd, *J* = 8.3, 1.9 Hz, 1H), 6.36 (d, *J* = 15.8 Hz, 1H), 6.22 – 6.08 (m, 1H), 4.08 (dd, *J* = 7.8, 5.6 Hz, 1H), 2.49 – 2.63 (m, 2H), 1.71 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.4, 137.5, 132.6, 130.8, 130.5, 130.4, 129.3, 128.6, 127.8, 127.2, 126.3, 125.4, 55.8, 43.3. HRMS Calcd. For C<sub>17</sub>H<sub>18</sub>Cl<sub>2</sub>N([M+H]<sup>+</sup>): 275.0371, found: 275.0378. The product was analyzed by HPLC to determine the enantiomeric excess:

99% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 6.87 and 7.91 min.



#### Ethyl (2*R*,3*S*)-2-amino-3-(4-bromophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 48% yield.  $[\alpha]^{25}_{D} = 24.7 (c \ 1.42, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.46 (d, J = 8.4 Hz, 2H), 7.32 – 7.16 (m, 2H), 6.31 – 6.12 (m, 1H), 5.26 – 5.07 (m, 2H), 4.40 – 4.20 (m, 2H), 4.08 (d, J = 9.3 Hz, 1H), 1.91 (brs, 2H), 1.33 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.8, 136.7, 134.8, 131.6, 130.9, 124.5 (q, J = 288.5 Hz), 121.7, 119.1, 68.0 (q, J = 25.6 Hz), 62.9, 51.5, 14.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.63 (s). HRMS Calcd. For C<sub>14</sub>H<sub>16</sub>BrF<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 366.0311, found: 366.0299. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206 \text{ nm}$ ); t<sub>r</sub> = 13.66 and 15.26 min.



#### (*S*,*E*)-4-(4-bromophenyl)-1-phenylbut-3-en-1-amine:

White solid; 32% yield, m.p. 72-74 °C;  $[\alpha]^{25}_{D} = -3.5$  (*c* 0.37, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.46 – 7.30 (m, 6H), 7.30 – 7.23 (m, 1H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.40 (d, *J* = 15.8 Hz, 1H), 6.21 – 6.06 (m, 1H), 4.08 (dd, *J* = 7.7, 5.5 Hz, 1H), 2.68 – 2.44 (m, 2H), 1.71 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.5, 136.3, 131.7, 131.6, 128.6, 127.9, 127.7, 127.2, 126.3, 120.9, 55.8, 43.3. HRMS Calcd. For C<sub>16</sub>H<sub>17</sub>BrN([M+H]<sup>+</sup>): 302.0539, found: 302.0532. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 6.70 and 7.48 min.



# Ethyl (2*R*,3*S*)-2-amino-2-(trifluoromethyl)-3-(4-(trifluoromethyl)phenyl)pent-4-enoate: Colorless oil; 49% yield. $[\alpha]^{25}_{D} = 9.3$ (*c* 1.01, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.59 (d, J = 8.2 Hz, 2H), 7.50 (d, J = 8.1 Hz, 2H), 6.25 (ddd, J = 18.0, 10.8, 9.6 Hz, 1H), 5.31 – 5.09 (m, 2H), 4.45 – 4.23 (m, 2H), 4.17 (d, J = 9.3 Hz, 1H), 1.92 (brs, 2H), 1.34 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.66, 141.88, 134.59, 129.86 (q, J = 32.7 Hz), 129.62, 125.39 (q, J = 3.8 Hz), 124.48 (q, J = 288.3 Hz),124.09 (q, J = 273.1 Hz), 119.41, 68.14 (q, J = 25.8Hz), 63.02, 51.93, 13.96. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.64 (s), -72.70 (s). HRMS Calcd. For C<sub>15</sub>H<sub>16</sub>F<sub>6</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 356.1072, found: 352.1080. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, $\lambda = 206$ nm); t<sub>r</sub> = 4.67 and 5.01 min.

#### (S,E)-1-phenyl-4-(4-(trifluoromethyl)phenyl)but-3-en-1-amine:

White solid; 41% yield, m.p. 80-82 °C;  $[\alpha]^{25}_{D} = -10.9$  (*c* 1.06, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, MeOD)  $\delta$  7.79 – 7.18 (m, 9H), 6.53 (d, *J* = 15.8 Hz, 1H), 6.42 – 6.16 (m, 1H), 4.29 – 4.09 (m, 1H), 2.75 (s, 2H), 1.29 (brs, 2H). <sup>13</sup>C NMR (101 MHz, MeOD)  $\delta$  141.44, 141.03, 131.87, 128.99 (q, *J* = 32.8 Hz), 128.49, 128.37, 127.71, 126.44, 126.21, 125.03 (q, *J* = 3.8 Hz), 124.35 (q, *J* = 271.9 Hz), 55.38, 40.66. <sup>19</sup>F NMR (376 MHz, MeOD)  $\delta$  -63.98 (s). HRMS Calcd. For C<sub>17</sub>H<sub>17</sub>F<sub>3</sub>N([M+H]<sup>+</sup>): 292.1304, found: 293.1308. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 5.72 and 6.24 min.



#### Ethyl (2R,3S)-2-amino-3-(4-nitrophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 60% yield.  $[\alpha]^{25}{}_{D} = 34.4 (c \ 0.97, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.19 (d, J = 8.6 Hz, 2H), 7.57 (d, J = 8.5 Hz, 2H), 6.23 (ddd, J = 17.2, 9.6, 9.2 Hz, 1H), 5.22 (dd, J = 13.4, 8.2 Hz, 2H), 4.58 – 4.27 (m, 2H), 4.22 (d, J = 9.2 Hz, 1H), 1.95 (brs, 2H), 1.35 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.41, 147.36, 145.37, 134.08, 130.16, 124.35 (q, J = 288.5 Hz), 123.55, 119.95, 68.10 (q, J = 25.9 Hz), 63.19, 51.87, 13.94. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.68 (s). HRMS Calcd. For C<sub>14</sub>H<sub>16</sub>F<sub>3</sub>N<sub>2</sub>O<sub>4</sub>([M+H]<sup>+</sup>): 333.1046, found: 333.1057. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 10.29 and 11.05 min.



#### Ethyl (2R,3S)-2-amino-3-(naphthalen-2-yl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 50% yield.  $[\alpha]^{25}_{D} = 40.0 (c \ 0.13, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.83 - 7.79 (m, 4H), 7.50 – 7.46 (m, 3H), 6.38 (ddd, J = 17.2, 9.6, 8.4 Hz, 1H), 5.21 (dd, J = 13.6, 6.9 Hz, 2H), 4.38 – 4.25 (m, 3H), 1.97 (brs, 2H), 1.36 (t, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.0, 134.2, 134.1, 132.3, 131.7, 127.2, 127.1, 126.9, 126.6, 126.1, 125.2, 125.0, 123.6 (q, J = 288.6 Hz), 117.8, 67.3 (q, J = 25.6 Hz), 61.8, 51.2, 13.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.57 (s). HRMS Calcd. For C<sub>18</sub>H<sub>19</sub>F<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 338.1362, found: 338.1351. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak ID, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206 \text{ nm}$ ; t<sub>r</sub> = 6.26 and 7.36 min.



### (S,E)-4-(naphthalen-2-yl)-1-phenylbut-3-en-1-amine:

White solid; 35% yield, m.p. 80-84 °C;  $[\alpha]^{25}_{D} = -11.8$  (*c* 0.85, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 – 7.74 (m, 3H), 7.67 (s, 1H), 7.55 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.49 – 7.33 (m, 6H), 7.29 – 7.25 (m, 1H), 6.64 (d, *J* = 15.8 Hz, 1H), 6.39 – 6.20 (m, 1H), 4.12 (dd, *J* = 8.0, 5.3 Hz, 1H), 2.77 – 2.49 (m, 2H), 1.77 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.8, 134.8, 133.6, 133.0, 132.8, 128.5, 128.1, 127.9, 127.7, 127.5, 127.1, 126.4, 126.2, 125.8, 125.7, 123.5, 55.9, 43.6. HRMS Calcd. For C<sub>20</sub>H<sub>20</sub>N([M+H]<sup>+</sup>): 274.1590, found: 274.1582. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 11.73 and 13.80 min.



3n

#### Ethyl (2R,3R)-2-amino-3-(furan-2-yl)-2-(trifluoromethyl)pent-4-enoate:

Brown oil; 44% yield.  $[\alpha]^{25}{}_{D} = 92.3$  (*c* 1.28, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.40 (s, 1H), 6.47 – 6.29 (m, 1H), 6.24 (d, *J* = 3.1 Hz, 1H), 6.18 – 5.94 (m, 1H), 5.26 – 5.11 (m, 1H), 4.41 – 4.19 (m, 3H), 2.14 (brs, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.5, 150.6, 142.5, 132.4, 124.3 (q, *J* = 287.9 Hz), 119. 5, 110.5, 108.5, 68.0 (q, *J* = 26.0 Hz), 62.9, 46.4, 14.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -74.60 (s). HRMS Calcd. For C<sub>12</sub>H<sub>15</sub>F<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 278.0999, found: 278.0987. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 4.87 and 5.05 min.



#### (*S*,*E*)-4-(furan-2-yl)-1-phenylbut-3-en-1-amine:

Brown thick oil; 30% yield.  $[\alpha]^{25}_{D} = -9.0$  (c 1.64, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.47

-7.11 (m, 6H), 6.46 - 6.21 (m, 2H), 6.21 - 5.96 (m, 2H), 4.06 (dd, J = 8.0, 5.2 Hz, 1H), 2.58 - 2.43 (m, 2H), 1.90 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 152.8, 145.6, 141.6, 128.5, 127.1, 126.3, 125.9, 121.4, 111.2, 106.8, 55.8, 43.3. HRMS Calcd. For C<sub>14</sub>H<sub>16</sub>NO([M+H]<sup>+</sup>): 214.1226, found: 214.1219. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 5.90 and 7.64 min.



#### Ethyl (2R,3R)-2-amino-3-(thiophen-2-yl)-2-(trifluoromethyl)pent-4-enoate:

Brown oil; 57% yield.  $[\alpha]^{25}_{D} = 92.7 (c \ 1.41 \ CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 – 7.21 (m, 1H), 7.00 – 6.97 (m, 2H), 6.18 – 6.09 (m, 1H), 5.31 – 5.08 (m, 2H), 4.46 (d,  $J = 9.3 \ Hz$ , 1H), 4.39 – 4.17 (m, 2H), 2.05 (brs, 2H), 1.33 (t,  $J = 7.1 \ Hz$ , 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  166.6, 138.1, 134.1, 125.7, 125.5, 124.3, 123.3 (q,  $J = 288.4 \ Hz$ ), 117.6, 67.1 (q,  $J = 25.6 \ Hz$ ), 61.9, 47.6, 12.9. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -73.00 (s). HRMS Calcd. For C<sub>12</sub>H<sub>15</sub>F<sub>3</sub>NO<sub>2</sub>S([M+H]<sup>+</sup>): 294.0770, found: 294.0758. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206 \ nm$ ); t<sub>r</sub> = 5.40 and 5.85 min.

#### (*S*,*E*)-1-phenyl-4-(thiophen-2-yl)but-3-en-1-amine:

Brown thick oil; 29% yield.  $[\alpha]^{25}_{D} = -24.2$  (*c* 1.23, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 – 7.30 (m, 4H), 7.26 (d, *J* = 7.0 Hz, 1H), 7.09 (d, *J* = 4.9 Hz, 1H), 6.96 – 6.90 (m, 1H), 6.88 (d, *J* = 2.9 Hz, 1H), 6.60 (d, *J* = 15.6 Hz, 1H), 6.16 – 5.88 (m, *J* = 15.2, 7.5 Hz, 1H), 4.06 (dd, *J* = 8.0, 5.2 Hz, 1H), 2.66 – 2.40 (m, 2H), 1.70 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.7, 142.6, 128.5, 127.3, 127.1, 126.9, 126.3, 126.0, 124.9, 123.6, 55.7, 43.3. HRMS Calcd. For C<sub>14</sub>H<sub>16</sub>NS([M+H]<sup>+</sup>): 230.0998, found: 230.0990. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow

rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 6.75 and 9.06 min.



#### Methyl (2R,3S)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-enoate

Colorless oil; 58% yield.  $[\alpha]^{25}_{D} = 16.9 (c \ 1.39, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 (d, J = 1.9 Hz, 1H), 7.40 (d, J = 8.3 Hz, 1H), 7.21 (dd, J = 8.3, 1.8 Hz, 1H), 6.30 – 6.05 (m, 1H), 5.27 – 5.11 (m, 2H), 4.06 (d, J = 9.3 Hz, 1H), 3.84 (s, 3H), 1.91 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  168.1, 137.8, 134.5, 132.5, 131.9, 131.0, 130.4, 128.6, 124.4 (q, J = 288.3 Hz), 119.4, 68.3 (q, J = 25.8 Hz) 53.6, 51.3. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.66 (s). HRMS Calcd. For C<sub>13</sub>H<sub>13</sub>Cl<sub>2</sub>F<sub>3</sub>NO<sub>2</sub>([M+H]<sup>+</sup>): 342.0270, found: 342.0260. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak IE, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 206 \text{ nm}$ ; t<sub>r</sub> = 5.92 and 6.63 min



#### (*S*,*E*)-1-(4-chlorophenyl)-4-phenylbut-3-en-1-amine:

White solid; 33% yield, m.p. 82-84 °C;  $[\alpha]^{25}_{D} = -10.4$  (*c* 1.63, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.41 – 7.12 (m, 9H), 6.47 (d, *J* = 15.8 Hz, 1H), 6.24 – 6.01 (m, 1H), 4.07 (dd, *J* = 7.9, 5.3 Hz, 1H), 2.63 – 2.41 (m, 2H), 1.57 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  144.2, 137.2, 133.2, 132.6, 128.6, 128.5, 127.8, 127.3, 126.5, 126.1, 55.2, 43.5. HRMS Calcd. For C<sub>16</sub>H<sub>17</sub>ClN ([M+H]<sup>+</sup>): 258.1044, found: 258.1036. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda = 256$  nm); t<sub>r</sub> = 8.11 and 10.60 min.



#### (*S*,*E*)-4-phenyl-1-(*p*-tolyl)but-3-en-1-amine:

Thick oil; 39% yield.  $[\alpha]^{25}_{D} = -15.7$  (*c* 0.96, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.45 – 7.08 (m, 9H), 6.48 (d, *J* = 15.8 Hz, 1H), 6.24 – 6.04 (m, 1H), 4.04 (dd, *J* = 7.7, 5.5 Hz, 1H), 2.69 – 2.45 (m, 2H), 2.34 (s, 3H), 2.13 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  142.6, 137.4, 136.7, 132.8, 129.2, 128.5, 127.2, 127.1, 126.2, 126.1, 55.5, 43.4, 21.1. HRMS Calcd. For C<sub>17</sub>H<sub>20</sub>N([M+H]<sup>+</sup>): 238.1590, found: 238.1585. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak OD, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 6.84 and 8.78 min.

#### 3. Divergent Synthesis of α-quaternary α-CF<sub>3</sub> AAs.



To a 25 mL nitrogen-filled dry Schlenk tube,  $[Ir(COD)Cl]_2$  (6.7 mg, 0.01 mmol), phosphoramidite ligand (*S,S,S*)-L2 or (*R,R,R*)-L2 (10.8 mg, 0.02 mmol), degassed THF (0.5 mL) and degassed propylamine (0.5 mL) were added. After stirring at 50 °C for 30 mins, the reaction was concentrated via rotary evaporation under reduced pressure to give the iridium complex as a pale yellow solid.<sup>5</sup> Then,  $\alpha$ -Trifluoromethyl aldimine esters 1 (0.5 mmol), allylic carbonates 2 (0.4 mmol), Cs<sub>2</sub>CO<sub>3</sub> (0.5 mol) and dry benzene (2 mL) was added into the nitrogenrefilled Schlenk tube. Once starting material was consumed (monitored by TLC), the reaction mixture was concentrated via rotary evaporation under reduced pressure, and then purified by flash chromatography on silica gel (PE / EA = 50:1), then the crude product was diluted in 1ml MeOH and NH<sub>2</sub>OH•OAc was added. After stirring at rt for 2 h, the reaction mixture was concentrated via rotary evaporation under reduced pressure, and then purified by preparative HPLC or chromatography on silica gel (PE / EA = 20:1) to give diastereoisomers **3** and **4**.

The separation of diastereomers (2R,3S)-**3a** and (2S,3S)-**4a** was conducted by prep-HPLC (Chiralpak IA-H, 0.10 cm I.D. ×25 cm L, MeCH / H<sub>2</sub>O = 45%, flow rate 3 mL/min,  $\lambda$  = 206 nm); t<sub>major</sub> = 11.38 min and t<sub>minor</sub> = 14.14 min.

(2R,3S)-3a

#### Ethyl (2R,3S)-2-amino-3-phenyl-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 56% yield. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 4.56 and 5.22 min.

(2S,3S)-4a

#### Ethyl (2S,3S)-2-amino-3-phenyl-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 35% yield.  $[\alpha]^{25}_{D} = 30.2$  (*c* 0.88, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.44 – 7.33 (m, 2H), 7.34 – 7.20 (m, 3H), 6.40 – 6.01 (m, 1H), 5.38 – 5.09 (m, 2H), 4.13 (d, *J* = 9.0 Hz, 1H), 4.11 – 3.89 (m, 2H), 1.97 (brs, 2H), 1.10 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.9, 138.1, 134.3, 129.0, 128.4, 127.6, 124.8 (q, *J* = 288.5 Hz), 119.2, 68.4 (q, *J* = 25.4 Hz), 62.4, 52.1, 13.7. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.78 (s). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda$  = 206 nm); t<sub>r</sub> = 4.37 and 4.84 min.

The separation of diastereomers (2S,3R)-**3a** and (2R,3R)-**4a** was conducted by prep-HPLC (Chiralcel OJ-H, 0.46 cm I.D. ×15 cm L, MeOH = 100%, flow rate 0.5 mL/min,  $\lambda$  = 214 nm);  $t_{major} = 5.53$  min and  $t_{minor} = 6.40$  min.

#### Ethyl (2S,3R)-2-amino-3-phenyl-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 55% yield.  $[\alpha]^{25}_{D} = -8.5$  (*c* 0.37, CH<sub>2</sub>Cl<sub>2</sub>). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 4.56 and 5.22 min.

EtO<sub>2</sub>C 
$$CF_3$$
  
H<sub>2</sub>N  $Ph$   
(2*R*,3*R*)-4a

#### Ethyl (2R,3R)-2-amino-3-phenyl-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 35% yield.  $[\alpha]^{25}_{D} = -30.5$  (*c* 1.00, CH<sub>2</sub>Cl<sub>2</sub>). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 4.37 and 4.84 min.



(2*R*,3*S*)-3m

#### Ethyl (2R,3S)-2-amino-3-(naphthalen-2-yl)-2-(trifluoromethyl)pent-4-enoate:

White solid; 50% yield. HRMS Calcd. For  $C_{18}H_{19}F_3NO_2([M+H]^+)$ : 338.1362, found: 338.1351. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak ID, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 11.54 and 13.71 min.



(2S,3S)-4m

#### Ethyl (2S,3S)-2-amino-3-(naphthalen-2-yl)-2-(trifluoromethyl)pent-4-enoate:

White solid; 35% yield, m.p. 58-60 °C;  $[\alpha]^{25}_{D} = 89.5$  (*c* 1.02, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.09 – 7.64 (m, 4H), 7.68 – 7.34 (m, 3H), 6.50 – 6.08 (m, 1H), 5.25 (dd, *J* = 13.5, 7.4 Hz, 2H), 4.31 (d, *J* = 8.9 Hz, 1H), 4.14 – 3.75 (m, 2H), 2.02 (brs, 2H), 1.03 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  167.9, 135.7, 134.3, 133.3, 132.7, 128.1, 128.0, 127.9, 127.6, 127.1, 126.1, 126.0, 124.9 (q, *J* = 288.7 Hz), 119.5, 68.5 (q, J = 25.3 Hz), 62.5, 52.1, 13.7. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.77 (s). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak ID, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 11.98 and 12.32 min.



(2S,3R)-3m

#### Ethyl (2S,3R)-2-amino-3-(naphthalen-2-yl)-2-(trifluoromethyl)pent-4-enoate:

White solid; 54% yield.  $[\alpha]^{25}_{D} = -39.1$  (*c* 1.03, CH<sub>2</sub>Cl<sub>2</sub>). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak ID, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 11.54 and 13.71 min.



(2*R*,3*R*)-4m

#### Ethyl (2R,3R)-2-amino-3-(naphthalen-2-yl)-2-(trifluoromethyl)pent-4-enoate:

White solid; 36% yield.  $[\alpha]^{25}_{D} = -88.0$  (*c* 0.82, CH<sub>2</sub>Cl<sub>2</sub>). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak ID, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min,  $\lambda = 206$  nm); t<sub>r</sub> = 11.98 and 12.32 min.



#### Methyl (2R,3S)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 58% yield. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda$  = 220 nm); t<sub>r</sub> = 5.90 and 6.59 min.



Methyl (2*S*,3*S*)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-enoate: Colorless oil; 31% yield. [α]<sup>25</sup><sub>D</sub> = 80.2 (*c* 1.08, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.54 (d, J = 2.0 Hz, 1H), 7.36 (d, J = 8.3 Hz, 1H), 7.28 (d, J = 2.0 Hz, 1H), 6.10 (ddd, J = 17.6, 9.6, 8.8 Hz, 1H), 5.23 (dd, J = 21.8, 13.6 Hz, 2H), 4.07 (d, J = 9.0 Hz, 1H), 3.63 (s, 3H), 1.98 (brs, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.9, 138.4, 133.3, 132.3, 131.7, 131.1, 130.3, 128.4, 127.4 (q, J = 289.1 Hz), 120.1, 68.2 (q, J = 25.8 Hz), 53.3, 51.2. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -73.04 (s). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 4.59 and 7.17 min.



#### Methyl (2S,3R)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 53% yield.  $[\alpha]^{25}_{D} = -17.1$  (*c* 1.52, CH<sub>2</sub>Cl<sub>2</sub>). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 5.90 and 6.59 min.



(2R,3R)-4p

#### Methyl (2S,3S)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-enoate:

Colorless oil; 28% yield.  $[\alpha]^{25}_{D} = -79.5$  (*c* 1.02, CH<sub>2</sub>Cl<sub>2</sub>). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 220$  nm); t<sub>r</sub> = 4.59 and 7.17 min.

## 4. Gram Scale and Synthetic Applications



In a 50 mL nitrogen-filled dry Schlenk tube,  $[Ir(COD)Cl]_2$  (67.0 mg, 0.1 mmol), phosphoramidite ligand (*S*,*S*,*S*)-**L2** (108.0 mg, 0.2 mmol), degassed THF (5 mL) and degassed propylamine (5 mL) were added. After stirring at 50 °C for 30 mins, the reaction was

concentrated via rotary evaporation under reduced pressure to give the iridium complex as a pale yellow solid. Then under the nitrogen-refilled dry Schlenk tube, **1b** (1.2 g, 5 mmol), allylic carbonates **2p** (1.0 g, 4 mmol) and dry benzene (20 mL) was added. After stirring at room temperature for 12 hours, the reaction mixture was concentrated via rotary evaporation under reduced pressure, and purified by flash chromatography on silica gel (PE / EA = 20 : 1) to remove the base. The crude product was diluted in 10 ml MeOH, then NH<sub>2</sub>OH•OAc was added. After stirring for 3 hours, the solvent was removed by rotary evaporation, diastereoisomers **3p** (66 % yield and 99 % ee) and **4p** (34 % yield and 99 % ee) were obtained by chromatography on silica gel (PE / EA = 20 : 1) as colorless oil.



**3p** (0.36 g, 1 mmol) was added to a 25 ml nitrogen-filled dry shlenk tube, followed by THF. Under -78 °C, DIBAL-H (1.70 ml, 2.50 mmol)was added dropwised. After stirring for 2 hours at room temprature, the reaction was quenched by coled NaOH aq (1 M, 10 ml), and the aqueous layer was extracted with additional portions of CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layers were combined, concentrated and purified by flash chromatography on silica gel (PE / EA = 5 : 1) to give the inseparable products **6** (83% yield and 99 % ee) as colorless oil.



(2S,3S)-6

#### (2S,3S)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-en-1-ol

Colorless oil; 85% yield.  $[\alpha]^{25}_{D} = 102.3 (c \ 1.11, CH_2Cl_2).^{1}H \ NMR (400 \ MHz, CDCl_3) \delta 7.51 (d, <math>J = 2.0 \ Hz, 1H$ ), 7.39 (d,  $J = 8.3 \ Hz, 1H$ ), 7.23 (dd,  $J = 8.3, 2.0 \ Hz, 1H$ ), 6.46 – 6.07 (m, 1H), 5.32 – 5.10 (m, 1H), 3.75 (d,  $J = 9.7 \ Hz, 1H$ ), 3.57 (dd,  $J = 56.2, 11.6 \ Hz, 2H$ ), 1.81 (brs, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  138.9, 135.5, 134.6, 132.4, 131.5, 130.3, 129.0, 127.1 (q,

J = 289.4 Hz), 125.71, 119.4, 62.8, 61.8 (q, J = 23.0 Hz), 51.3. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$ -73.23 (s). HRMS Calcd. For C<sub>14</sub>H<sub>13</sub>Cl<sub>2</sub>F<sub>3</sub>NO([M+H]<sup>+</sup>): 314.0321, found: 314.0307. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 209$  nm); t<sub>r</sub> = 12.25 and 14.80 min.



(2R,3S)-6

#### (2R,3S)-2-amino-3-(3,4-dichlorophenyl)-2-(trifluoromethyl)pent-4-en-1-ol

Colorless oil; 83% yield.  $[\alpha]^{25}_{D} = 103.2 (c \ 0.73, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 (d, J = 1.9 Hz, 1H), 7.38 (d, J = 8.3 Hz, 1H), 7.17 (dd, J = 8.3, 1.9 Hz, 1H), 6.56 – 6.10 (m, 1H), 5.46 – 5.08 (m, 2H), 3.72 (d, J = 10.3 Hz, 1H), 3.69 – 3.62 (m, 2H), 1.59 (brs, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  139.0, 134.8, 132.4, 131.4, 131.1, 130.3, 128.6, 127.2 (q, J = 289.3 Hz), 120.0, 62.3, 62.3, 61.6 (q, J = 23.2 Hz), 50.7. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -72.75 (s). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak OJ, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 210$  nm); t<sub>r</sub> = 14.92 and 16.19 min.



To a 25-mL vial, stirrer bar, **6** (50.0 mg, 0.2 mmol), CH<sub>2</sub>Cl<sub>2</sub> (2 mL) and Et<sub>3</sub>N (32.4 mg, 0.3 mmol) were added. After stirring for 5 mins under 0 °C, MsCl (27.4 mg, 0.2 mmol) was added via a microsyringe, then the reaction was allowed to react for overnight at rt. Once the starting material was consumed (monitored by TLC), the reaction mixture was concentrated via rotary evaporation under reduced pressure, and then purified by flash chromatography on Al<sub>2</sub>O<sub>3</sub> (PE / EA = 10 : 1) to give 7 (95 % yield and 99 % ee)as colorless oil.



#### (S)-2-((S)-1-(3,4-dichlorophenyl)allyl)-2-(trifluoromethyl)aziridine:

Colorless oil; 92% yield.  $[\alpha]^{25}{}_{D} = 184.0 (c \ 1.59, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, C<sub>6</sub>D<sub>6</sub>)  $\delta$  7.13 (s, 1H), 6.95 (d, J = 8.3 Hz, 1H), 6.53 (s, 1H), 6.04 – 5.21 (m, 1H), 5.12 – 4.44 (m, 2H), 3.50 (d, J = 8.4 Hz, 1H), 1.48 (s, 1H), 0.94 (s, 1H). <sup>13</sup>C NMR (101 MHz, C<sub>6</sub>D<sub>6</sub>)  $\delta$  139.5, 134.4, 132.5, 131.3, 130.3, 128.2, 128.1, 125.6 (q, J = 278.8 Hz), 119.4, 47.1, 40.1 (q, J = 32.0 Hz), 24.8. <sup>19</sup>F NMR (376 MHz, C<sub>6</sub>D<sub>6</sub>)  $\delta$  -70.60 (s). HRMS Calcd. For C<sub>12</sub>H<sub>11</sub>Cl<sub>2</sub>F<sub>3</sub>N ([M+H]<sup>+</sup>): 296.0215, found: 296.0206. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak OJ, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 210$  nm); t<sub>r</sub> = 6.50 and 6.90 min.



(2R,3S)-**7** 

#### (R)-2-((S)-1-(3,4-dichlorophenyl)allyl)-2-(trifluoromethyl)aziridine:

Colorless oil; 95% yield.  $[\alpha]^{25}_{D} = 137.6 (c 2.02, CH_2Cl_2)$ . <sup>1</sup>H NMR (400 MHz, C<sub>6</sub>D<sub>6</sub>)  $\delta$  7.50 – 7.20 (m, 1H), 6.99 (d, J = 8.3 Hz, 1H), 6.82 – 6.57 (m, 1H), 5.80 – 5.25 (m, 1H), 5.00 – 4.66 (m, 2H), 3.49 (d, J = 8.8 Hz, 1H), 1.54 (s, 1H), 1.25 (s, 2H). <sup>13</sup>C NMR (101 MHz, C<sub>6</sub>D<sub>6</sub>)  $\delta$  139.9, 135.0, 132.6, 131.4, 130.2, 128.1, 127.7, 125.6 (q, J = 278.9 Hz), 118.1, 47.2, 40.4 (q, J = 31.7 Hz), 24.9. <sup>19</sup>F NMR (376 MHz, C<sub>6</sub>D<sub>6</sub>)  $\delta$  -70.54 (s). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak OJ, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min,  $\lambda = 210$  nm); t<sub>r</sub> = 6.09 and 6.45 min.

# 5. References

- (1) (a) Ohkura, H.; Berbasov, D. O.; Soloshonok, V. A. *Tetrahedron*, **2003**, *59*, 1647. (b) Soloshonok, V. A.; Kukhar, V. P. *Tetrahedron*, **1997**, *53*, 8307.
- (2) Stanley, L. M.; Hartwig, J. F. Angew. Chem., Int. Ed. 2009, 48, 7841.
- (3) Richards, C. J.; Mulvaney, A. W. Tetrahedron: Asymmetry 1996, 7, 1419.
- (4) Smith, C. R.; Mans, D. J.; Rajanbabu, T. V. Org. Synth. 2008, 85, 238.
- (5) Stanley, L. M.; Hartwig, J. F. J. Am. Chem. Soc. 2009, 131, 8971.

7. NMR spectra











 $\begin{array}{c} 7.385\\ 7.386\\ 7.3355\\ 7.236\\ 7.236\\ 7.281\\ 7.281\\ 7.282\\ 7.2255\\ 7.2255\\ 7.2255\\ 7.2255\\ 7.2255\\ 6.2100\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.190\\ 6.100\\ 6.190\\ 6.100\\ 6.$ 















S34



(2R,3S)-**3c** 

7.253 7.1233 7.1245 7.1245 7.1245 6.218 7.127 7.127 7.127 6.218 6.238 7.238 7.238 7.238 7.238 7.238 7.238 7.238 7.238 7.238 7.238 7.238 7.






## 



90 80 f1 (ppm) 



















S42











6.869 6.787 6.787 6.787 6.787 6.787 6.787 6.744 6.142 6.142 6.142 6.142 6.133 6.157 6.157 6.157 6.157 6.157 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.203 6.133 7.236 6.133 7.236 6.133 7.236 6.133 7.236 6.133 7.236 6.133 7.2367 7.2367 7.236 7.2367 7.236 7.236 7.236 7.236 7.236 7.236 7.236 7.2



















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7.370 7.2565 7.72565 7.72565 7.72565 6.232 6.232 6.1265 6.1289 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.189 6.165 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 6.165 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.239 6.189 7.2397 7.239 7.239 7.239 7.239 7.239 7.239 7.2397 7





S51





















 $\begin{array}{c} 7.467\\ 7.446\\ 7.242\\ 7.226\\ 6.244\\ 6.220\\ 6.153\\ 6.153\\ 6.153\\ 6.153\\ 6.153\\ 6.149\\ 4.321\\ 4.321\\ 4.321\\ 4.322\\ 4.321\\ 4.228\\ 4.321\\ 4.228\\ 4.321\\ 4.228\\ 4.321\\ 4.262\\ 4.321\\ 4.362\\ 4.362\\ 1.352\\ 1.$ 







-140 -160 -180 -200







 $\begin{array}{c} 7.597\\ 7.567\\ 7.567\\ 7.567\\ 7.567\\ 6.290\\ 6.249\\ 6.249\\ 6.249\\ 6.243\\ 6.172\\ 6.172\\ 6.172\\ 6.172\\ 6.172\\ 6.172\\ 6.172\\ 4.303\\ 4.303\\ 4.289\\ 4.4162\\ 4.289\\ 4.162\\ 4.216\\ 4.289\\ 4.162\\ 4.216\\ 4.289\\ 4.162\\ 4.216\\ 4.232\\ 4.216\\ 4.2322\\ 4.232\\ 4.2322\\ 4.232\\ 4.232\\ 4.232\\ 4.232\\ 4.232\\ 4.232\\ 4.232\\$ 













< 8.199 < 8.178 < 7.584 < 7.563</pre> -1.951 -1.368 -1.350 -1.332











 $\begin{array}{c} 7.854\\ 7.771\\ 7.750\\ 7.771\\ 7.750\\ 7.756\\ 7.7551\\ 7.435\\ 6.326\\ 6.326\\ 6.326\\ 6.326\\ 6.326\\ 6.326\\ 6.326\\ 6.326\\ 6.326\\ 5.268\\ 6.326\\ 5.268\\ 5$ 









## 





## $\begin{array}{c} -7.398 \\ 6.242 \\ 6.053 \\ 6.057 \\ 6.057 \\ 6.057 \\ 6.057 \\ 6.057 \\ 6.053$



90 80 f1 (ppm) 







100 90 80 f1 (ppm)


(2R,3R)-**30** 

















20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 f1 (ppm)









 $\begin{array}{c} 7.337\\ 7.337\\ 7.7395\\ 7.7395\\ 7.7395\\ 7.7395\\ 7.7182\\ 7.7182\\ 7.7182\\ 7.7182\\ 7.7146\\ 6.460\\ 6.460\\ 6.165\\ 6.145\\ 6.1465\\ 6.1466\\ 6.1466\\ 6.1466\\ 6.1466\\ 6.1466\\ 6.1466\\ 6.1466\\ 6.1466\\ 6.1466\\ 6.128\\ 6.12$ 

















---70.596

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200 f1 (ppm)





## 8. HPLC spectra



Data File E:\DATA\SXS\SXS-7-73-B-OPTICAL 2019-05-20 10-53-44\SXS-7-73-B-OPTICAL.D Sample Name: SXS-7-77-B-RAC

Acg. Operator : S	SYSTEM	Seg. Line : 1	
Acq. Instrument : 1	1260	Location: 34	
Injection Date : 5	5/20/2019 10:55:21 AM	Tnj: l	
		Inj Volume : 4.000 ul	
Acg. Method : E	C:\DATA\SXS\SXS-7-73-B-OPTICAL	, 2019-05-20 10-53-44\	ADH-95-5-10MIN-1.0ML.M
Last changed : 5	5/20/2019 11:02:36 AM by SYSTE	стана и по на	
, ,	(modified after loading)		
Analysis Method : E	2:\DATA\SXS\SXS-7-73-B-OPTICAL	, 2019-05-20 10-53-44\	ADH-95-5-10MIN-1.0ML.M (
- 2	Sequence Method)		
Last changed : 6	5/10/2019 11:05:26 AM by SYSTE	m	
- (	(modified after loading)		
Additional Info : P	Peak(s) manually integrated		
DAD1 A, Sig=206,4	4 Ref=360,100 (E:\DATA\SXS\SXS-7-73-B-0 PTIC)	AL 2019-05-20 10-53-44\SXS-7-73-B	- OPTICAL.D)
mAU			Josh Andrew Statish
	~		
		4 5	6 7 min
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	Area Percent Report		
Sorted By	: Signal		
Multiplier	: 1.0000		
Dilution	: 1.0000	-	
Do not use Multipli	ler & Dilution Factor with 151	Ds	
Sigmal 1: DAD1 A, S	šig=206,4 Ref=360,100		
Peak RetTime Type	Width Area Height	Area	
# [min]	[min] [mAU*s] [mAU]	*	
-		1	
1 5.286 FM	0.2914 2.03269e4 1162.49976	46.6007	
2 5.864 FM	0.3495 2.32924e4 1110.64587	53.3993	
Totals :	4.36193e4 2273.14563		
	*** End of Report ***		

1260 6/10/2019 11:05:29 AM SYSTEM

Data File E:\DATA\SXS\SXS-6-139-2 2018-07-26 18-18-30\SXS-6-139-21.D Sample Name: SXS-6-169-2



1260 6/10/2019 9:44:35 AM SYSTEM



Data File E:\DATA\SXS\SXS-6-139-3 2018-07-26 18-54-31\SXS-6-139-3.D Sample Name: SXS-6-115



Data File E:\DATA\SXS\SXS-6-139-3 2018-07-26 18-54-31\SXS-6-139-31.D Sample Name: SXS-6-139-3 Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 74 Injection Date : 7/26/2018 7:09:19 PM Inj: 1 Inj Volume : 1.000 µl Acq. Method : E:\DATA\SXS\SXS-6-139-3 2018-07-26 18-54-31\ODH-60-40-1ML-1uL-20MIN.M Last changed : 7/26/2018 7:05:52 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-6-139-3 2018-07-26 18-54-31\0DH-60-40-1ML-1uL-20MIN.M ( Sequence Method) Last changed : 6/10/2019 9:45:55 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\SXS\SXS-6-139-3 2018-07-26 18-54-31\SXS-6-139-31.D) mAU T 175 -150 -125 -100 -75 -50 -25 -20 0 10 é. ś -----Area Percent Report Sorted By : Signal Multiplier 1.0000 . 1.0000 Dilution : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* 1 6.760 BB 0.1397 34.21788 3.07704 0.9943 2 9.070 BB 0.2772 3407.19702 190.05014 99.0057 Totals : 3441.41490 193.12718 \*\*\* End of Report \*\*\*

1260 6/10/2019 9:46:55 AM SYSTEM



(27(,00) 0

Data File E:\DATA\SXS\SXS-7-4-B 2018-09-06 16-08-27\SXS-7-4-B.D Sample Name: SXS-7-3-RAC ------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 73 Injection Date : 9/6/2018 4:09:53 PM Inj : 1 Inj Volume : 2.000 µl : E:\DATA\SXS\SXS-7-4-B 2018-09-06 16-08-27\ADH-95-5-10MIN-1.0ML.M Acq. Method : 9/6/2018 4:18:07 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-4-B 2018-09-06 16-08-27\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 9:49:34 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DADI A Sig=206,4 Ref=360,100 (E:DATA:SXS\SXS:7:4-B 2018-09-06 16-08-27\SXS:7-4-B.D) mAU -200 150 100 50 0 6 Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* 
 1
 4.944
 BV
 0.1397
 2285.96436
 243.04341
 44.3915

 2
 5.217
 VB
 0.1668
 2863.59229
 242.52235
 55.6085
Totals : 5149.55664 485.56577 \*\*\* End of Report \*\*\*

Data File E:\DATA\SXS\SXS-7-4-B 2018-09-06 16-08-27\SXS-7-4-B1.D Sample Name: SXS-7-4-0PTICAL

Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 74 Injection Date : 9/6/2018 4:20:15 PM Inj: 1 Inj Volume : 2.000 µl Acq. Method : E:\DATA\SXS\SXS-7-4-B 2018-09-06 16-08-27\ADH-95-5-10MIN-1.0ML.M Last changed : 9/6/2018 4:27:12 PM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-4-B 2018-09-06 16-08-27\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 9:49:34 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:DATA\SXS\SXS-7-4-B 2018-09-06 16-08-27\SXS-7-4-B1.D) mAU ] 800 -700 -600 -500 -400 -300 -200 -100 -D 4 5 mir \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier : 1.0000 Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Height Peak RetTime Type Width Area Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----|-----|-----| 1 4.941 VV R 0.1540 9157.38672 841.19965 100.0000 Totals : 9157.38672 841.19965 \*\*\* End of Report \*\*\*

1260 6/10/2019 9:50:59 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-4-L 2018-09-06 16-38-57\SXS-7-4-L.D Sample Name: SXS-7-3-L-RAC



Data File E:\DATA\SXS\SXS-7-4-L 2018-09-06 16-38-57\SXS-7-4-L1.D Sample Name: SXS-7-4--L-OPTICAL



1260 6/10/2019 9:53:41 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-24-B 2018-10-10 10-14-31\SXS-7-24-B.D Sample Name: SXS-7-20-B-RAC

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Acq. Operator : S	YSTEM		Seg. Line :	1			
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injection bace . I	10/10/2010 10:13:30		···· · · · · · · · · · · · · · · · · ·	1 000 1			
		L 0. 0.00 G V	nj volume :				
Acq. Method : H	: \DATA\ 5X5\5X5-7-2	24-B 2018-10-	·10 10-14-31	\ADH-95-5-IOMIN-1.OML.M			
Last changed : 1	LU/IU/ZUI8 IU:ZZ:U.	L AM DY SYSTE	.m				
(	(modified after loa	ading)					
Analysis Method : E	C:\DATA\SXS\SXS-7-2	24-B 2018-10-	10 10-14-31	\ADH-95-5-10MIN-1.0ML.M (Sequence			
Ľ	lethod)						
Last changed : 6	5/10/2019 9:54:53 A	AM by SYSTEM					
(	(modified after log	ading)					
Additional Info : F	eak(s) manually in	ntegrated					
DAD1 A, Sig=206,4	DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\\$X\$\\$X.\$.7-24-B 2018-10-10 10-14-31\\$X.\$-7-24-B.D)						
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Do not use Multipli	ter « Difucion Pac)	LOE WICH ISH	18				
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Peak RetTime Type	Width Area	Height	Area				
# [min]	[min] [mAU*s]	[mAU]	*				
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1 4.723 MF	0.1892 8682.55664	764.78741	49.3047				
2 5.372 FM	0.2083 8927.43945	714.26990	50.6953				
Totals :	1.76100e4	1479.05731					
Totals :	1.76100e4	1479.05731					
Totals :	1.76100e4	1479.05731					
Totals :	1.76100e4	1479.05731					
Totals :	1.76100e4	1479.05731					
Totals :	1.76100e4 *** End of	1479.05731 Report ***					

1260 6/10/2019 9:54:54 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-24-B 2018-10-10 10-14-31\SXS-7-24-B1.D Sample Name: SXS-7-24-OPTICAL

Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 2 Injection Date : 10/10/2018 10:25:23 AM Inj: 1 Inj Volume : 4.000 µl Acq. Method : E:\DATA\SXS\SXS-7-24-B 2018-10-10 10-14-31\ADH-95-5-10MIN-1.0ML.M Last changed : 10/10/2018 10:22:01 AM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-24-B 2018-10-10 10-14-31\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 9:54:53 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\SXS\SXS-7-24-B 2018-10-10 10-14-31\SXS-7-24-B1.D) baitstn A mAU 🗋 1600 -1400 -1200 -1000 -800 -600 -400 200 4.714 D 4 6 ś Area Percent Report Sorted Bv Sional : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] ÷ ---|-----|----|-----| 1 4.714 VB 0.1526 121.07980 10.90072 0.5026 2 5.352 MF 0.2268 2.39714e4 1761.61389 99.4974 Totals : 2.40925e4 1772.51461 \*\*\* End of Report \*\*\*

1260 6/10/2019 9:56:15 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-24-L 2018-10-10 10-42-03\SXS-7-24-L.D Sample Name: SXS-7-20-L-RAC

Acg. Operator :	SYSTEM	Seq	Line : l			
Acq. Instrument :	1260	Loc	ation: 3			
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,coolon bace .	10,10,0010 10.10.	Tni S	Tolume : 3 000 ul			
Acq. Method	F.\DATA\ 373\ 373-7	-24-T. 2018-10-10	0-42-03\0DH-60-40-	IML - 111L - 20MTN M		
Last changed :	10/10/2018 10:52:	46 AM by SVSTEM				
	(modified after 1	oeding)				
Amelwaia Mathod .	F.)DATAL GVGLGVG_7	.0auing) /_2/_T_2018_10_10_1	0-42-031008-60-40-1	INI - 111 - 20MIN M (		
Analysis Mechod :	C: (DATA) SAS(SAS- /	-24-6 2010-10-10 .	10-42-03\000-60-40	ING-IUG-ZONIN.N (		
	Sequence Method)					
Last changed :	6/10/2019 9:57:20	AM BY SYSTEM				
(modified after loading)						
Additional Info :	Peak(s) manually	integrated				
DAD1 A, Sig=206	3,4 Ref=360,100 (E:\DATA\SX	S\SXS-7-24-L 2018-10-10 10-4	12-D3\SXS-7-24-L.D)			
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o Sorted By Multiplier Dilution Do not use Multipl	Area Perce  : Signal : 1.0000 : 1.0000 Lier & Dilution Fe	4 6 ent Report		     		
0 Sorted By Multiplier Dilution Do not use Multipl	Area Perce : Signal : 1.0000 : 1.0000 Lier & Dilution Fe	4 6 ent Report		     		
o Sorted By Multiplier Dilution Do not use Multipl	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa	A 6 4 6 ent Report cont with ISTDs		     		
o  Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A,	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa	A 6 4 6 ent Report cont state of the stat				
o Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A,	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa	4 6 ent Report				
0 Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area	4 6 ent Report cor with ISTDs 0,100 Height Are				
O Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min]	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fe Sig=206,4 Ref=360 Width Area [min] [mAU*s]	4 6 ant Report ctor with ISTDs ,100 Height Are [mAU] 2				
O Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	Area Perce : Signal : 1.0000 : 1.0000 Lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s]	4 6 ant Report contor with ISTDs (mAU] 3 	<u>, / , , , , , , , , , , , , , , , , , ,</u>			
O Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min]     1 7.085 VV R	Area Perce : Signal : 1.0000 : 1.0000 Lier & Dilution Fe Sig=206,4 Ref=360 Width Area [min] [mAU*s]  0.2192 1.62521e4	4 6 4 6 ent Report cont with ISTDs 0,100 Height Ara [mAU] 2 	2a 3 3 3 3 3 3 3 3 3 3 3 3 3			
Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	 Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s] 	4 6 4 6 4 6 9 9 9 9 9 9 100 100 100 100	a 			
Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s] 	4 6 4 6 ent Report 0,100 Height Are [mAU] 2 1138.95215 49.3 4 858.67590 50.4	a 			
sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s] 0.2192 1.62521e4 0.2944 1.65179e4 3.27700e4	4 6 4 6 4 6 4 6 4 6 4 6 4 6 6 10 10 10 100 Height Ard [mAU] 5 1138.95215 49.3 4 858.67590 50.4 1997.62805	a 			
o Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s] 0.2192 1.62521e4 0.2944 1.65179e4 3.27700e4	4 6 4 6 4 6 4 6 4 6 4 6 4 6 6 10 10 10 10 10 10 10 10 10 10	a 			
o Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s] 0.2192 1.62521e4 0.2944 1.65179e4 3.27700e4	4 6 4 6 4 6 4 6 4 6 4 6 4 6 6 10 10 10 10 10 10 10 10 10 10	a 3943 4057			
o Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s] 0.2192 1.62521e4 0.2944 1.65179e4 3.27700e4	4 6 4 6 4 6 4 6 4 6 4 6 4 6 6 10 10 10 10 10 10 10 10 10 10	24 3 3 3 3 3 4 3 4 3 4 3 4 3 4 4 5 5 5 5 5 5 5 5 5 5 5 5 5			
o Sorted By Multiplier Dilution Do not use Multipl Signal 1: DAD1 A, Peak RetTime Type # [min] 	Area Perce : Signal : 1.0000 : 1.0000 lier & Dilution Fa Sig=206,4 Ref=360 Width Area [min] [mAU*s] 0.2192 1.62521e4 0.2944 1.65179e4 3.27700e4	4 6 4 6 int Report       	a 30 30 30 30 30 30 30 30 30 30			

Data File E:\DATA\SXS\SXS-7-24-L 2018-10-10 10-42-03\SXS-7-24-L1.D Sample Name: SXS-7-24-L-OPTICAL





Data File E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\SXS-7-35-B.D Sample Name: SXS-7-34-B-RAC ------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 1 Injection Date : 10/22/2018 2:45:50 PM Inj : 1 Inj Volume : 6.000 µl Acq. Method : E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\ADH-95-5-10MIN-1.0ML.M : 10/22/2018 2:54:20 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 10:03:56 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E-DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\SXS-7-35-B.D) mAU 800 600 400 200 ٥ 7 Area Percent Report \_\_\_\_\_ Sorted By : Signal 1.0000 Multiplier : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] ÷ ----|-----|----|-----|-----| 
 1
 6.877 BV
 0.2268
 1.45137e4
 952.38239
 46.2913

 2
 7.318 VB
 0.2577
 1.68393e4
 955.09454
 53.7087
2 7.318 VB

\*\*\* End of Report \*\*\*

3.13530e4 1907.47693

Totals :

Data File E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\SXS-7-35-B1.D Sample Name: SXS-7-35-B-OPTICAL



S103



Data File E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\SXS-7-12-13-L.D Sample Name: SXS-7-11-L-RAC



1260 6/10/2019 9:59:58 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\SXS-7-12-13-L1.D Sample Name: SXS-7-12-L-OPTICAL





Data File E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\SXS-7-35-B.D Sample Name: SXS-7-34-B-RAC

------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 1 Injection Date : 10/22/2018 2:45:50 PM Inj : 1 Inj Volume : 6.000 µl Acq. Method : E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\ADH-95-5-10MIN-1.0ML.M : 10/22/2018 2:54:20 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 10:03:56 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E-DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\SXS-7-35-B.D) mAU 800 600 400 200 ٥ 7 Area Percent Report \_\_\_\_\_ Sorted By : Signal 1.0000 Multiplier : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----| 1 6.877 BV 0.2268 1.45137e4 952.38239 46.2913 2 7.318 VB 0.2577 1.68393e4 955.09454 53.7087 Totals : 3.13530e4 1907.47693 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:03:58 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-35-B 2018-10-22 14-44-24\SXS-7-35-B1.D Sample Name: SXS-7-35-B-OPTICAL



S107



Data File E:\DATA\SXS\SXS-7-35-L 2018-10-22 15-19-33\SXS-7-35-L.D Sample Name: SXS-7-34-L-RAC

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 3 Injection Date : 10/22/2018 3:21:00 PM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-7-35-L 2018-10-22 15-19-33\ODH-60-40-1ML-1uL-20MIN.M Last changed : 10/22/2018 3:33:06 PM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-35-L 2018-10-22 15-19-33\ODH-60-40-1ML-1uL-20MIN.M ( Sequence Method) : 6/10/2019 10:06:16 AM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\SXS\SXS-7-35-L 2018-10-22 15-19-33\SXS-7-35-L.D) mAU -800 -830 700 -600 -500 -400 -300 -200 100 0 ś 10 12 14 \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----|-----|-----| 1 8.799 VV R 0.2887 1.72969e4 894.06628 49.4862 2 11.930 VV R 0.4234 1.76560e4 633.06915 50.5138 Totals : 3.49529e4 1527.13544 \_\_\_\_\_ \*\*\* End of Report \*\*\*

1260 6/10/2019 10:06:19 AM SYSTEM
Data File E:\DATA\SXS\SXS-7-35-L 2018-10-22 15-19-33\SXS-7-35-L1.D Sample Name: SXS-7-35-L-OPTICAL



1260 6/10/2019 10:07:08 AM SYSTEM

EtO<sub>2</sub>C, CF<sub>3</sub> H<sub>2</sub>N (2*R*,3*S*)-**3**f

Data File E:\DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\SXS-7-40-B.D Sample Name: SXS-7-42-B-RAC ------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 61 Injection Date : 11/5/2018 3:36:23 PM Inj : 1 Inj Volume : 2.000 µl : E:\DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\ADH-95-5-10MIN-1.0ML.M Acq. Method : 11/5/2018 3:46:41 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 10:08:47 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\SXS-7-40-B.D) mAU 400 300 200 -100 ٥ 10 ś Area Percent Report \_\_\_\_\_ Sorted By : Signal 1.0000 Multiplier : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] ÷ ----|-----|----|-----|-----| 
 1
 8.278
 FM
 0.2710
 7487.49854
 460.40265
 47.3637

 2
 8.924
 VB
 0.2583
 8321.03125
 463.89944
 52.6363
 Totals : 1.58085e4 924.30209 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:08:51 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\SXS-7-40-B1.D Sample Name: SXS-7-40-B-OPTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 62 Injection Date : 11/5/2018 3:49:55 PM Inj: 1 Inj Volume : 2.000 µl Acq. Method : E:\DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\ADH-95-5-10MIN-1.0ML.M Last changed : 11/5/2018 3:46:41 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 10:08:47 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\SXS\SXS-7-40-B 2018-11-05 15-34-54\SXS-7-40-B1.D) mAU 1200 -1000 -800 -600 -400 -200 -8 2 ٥. 4 10 6 ś Area Percent Report Sorted Bv Sional . : 1.0000 Multiplier Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Area Height # [min] [min] [mAU\*s] [mAU] \* ----|-----|-----|-----| 1 8.195 BV E 0.2226 145.58292 7.87915 0.6213 2 8.803 VB R 0.2619 2.32856e4 1281.84119 99.3787 Totals : 2.34312e4 1289.72034 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:09:45 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-59-L-OPTICAL 2018-11-30 14-50-35\SXS-7-59-L-OPTICAL.D Sample Name: SXS-7-65-L-RAC

Acq. Operator :	SYSTEM		Seq. Line : 1	
Acq. Instrument :	1260		Location : 93	
Injection Date :	11/30/2018 2:52:02	PM	Inj: 1	
			Inj Volume : 3.000 μl	
Acq. Method :	E:\DATA\SXS\SXS-7-3 M	59-L-OPTICAL	2018-11-30 14-50-35\ODH-6	0-40-1ML-1uL-20MIN
Last changed :	11/30/2018 3:09:00	PM by SYSTE	И	
	(modified after log	ading)		
Analysis Method :	E:\DATA\SXS\SXS-7-	59-L-OPTICAL	2018-11-30 14-50-35\0DH-6	0-40-1ML-1uL-20MIN
	M (Sequence Method)	)		
Last changed :	6/10/2019 10:10:55	AM by SYSTE	м	
	(modified after loa	ading)		
Additional Info :	Peak(s) manually in	ntegrated		
DAD1 A, Sig=206	,4 Ref=360,100 (E:\DATA\SXS\:	SX S-7-59-L-O PTIC/	L 2018-11-30 14-50-35\SXS-7-59-L-OPTICA	L.D)
mAU -			16 66 17 15	
400			11 11	
250			A (1	
300-				
250-				
200				
150 -				
100-				
	l.			
50-	<u></u> д.			
	_ MUN			
			· · · · · · · · · · · · · · · · · · ·	
2	4	6	8 10 12	14 16
	Area Percent	t Report		
Sorted By	: Signal			
Multiplier	: 1.0000			
Dilution	: 1.0000		_	
Do not use Multipl	ier & Dilution Fact	tor with IST	)s	
Circul Is DADI A	64	100		
argnar I: DADI A,	JIG=200,4 KEI=360,	100		
Peak RetTime Type	Width Area	Height	Area	
# [min]	[min] [mAU*s]	[mAU]	\$	
1 8.573 BV R	0.2771 7942.19678	415.27612	43.9369	
2 10.345 BV R	0.3359 1.01342e4	419.37051	56.0631	
Totals :	1.80764e4	834.64664		
	*** End of	Report ***		
				Dome 1 of 1
6/10/2019 10:10:5	7 AM SYSTEM			rage I OI I

Data File E:\DATA\SXS\SXS-7-59-L-OPTICAL 2018-11-30 14-50-35\SXS-7-59-L-OPTICAL1.D Sample Name: SXS-7-59-L-OPTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 94 Injection Date : 11/30/2018 3:10:24 PM Inj: l Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-59-L-OPTICAL 2018-11-30 14-50-35\0DH-60-40-1ML-1uL-20MIN. Acq. Method M Last changed : 11/30/2018 3:09:00 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-59-L-OPTICAL 2018-11-30 14-50-35\ODH-60-40-1ML-1uL-20MIN. M (Sequence Method) Last changed : 6/10/2019 10:10:55 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\\$X\$\\$X\$-7-59-L-O PTICAL 2018-11-30 14-50-35\\$X\$-7-59-L-O PTICAL1.D) mAU 1000 800 -600 400 200 8.670 D 10 12 14 min \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : 1.0000 : Multiplier Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----|-----| 1 8.670 VV R 0.2361 583.33868 29.57875 2.0653 2 10.419 BV R 0.3505 2.76619e4 1059.86865 97.9347 Totals : 2.82453e4 1089.44740 \_\_\_\_\_ \*\*\* End of Report \*\*\*

1260 6/10/2019 10:11:49 AM SYSTEM



Data File E:\DATA\SXS\SXS-6-151-B 2018-08-28 11-29-50\SXS-6-151-B.D Sample Name: SXS-6-150-B-RAC

------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 71 Injection Date : 8/28/2018 11:31:15 AM Inj : 1 inj Volume : 2.000 µl Acq. Method : E:\DATA\SXS\SXS-6-151-B 2018-08-28 11-29-50\ADH-95-5-10MIN-1.0ML.M Last changed : 8/28/2018 11:29:50 AM htt «У«тты Inj Volume : 2.000 µl Analysis Method : E:\DATA\SXS\SXS-6-151-B 2018-08-28 11-29-50\ADH-95-5-10MIN-1.0ML.M ( Sequence Method) : 6/10/2019 10:13:04 AM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:/DATA(\$X\$\\$X\$\6-151-B 2018-08-28 11-29-50\\$X\$-6-151-B.D) mAU 250 200 -150 -100 -50 D ė \_\_\_\_\_ Area Percent Report Sorted By : Sional Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area [mAU] # [min] [min] [mAU\*s] ÷ 1 5.480 BV 0.1614 3113.80371 280.72852 46.3869 2 5.829 VV R 0.1848 3598.88062 276.56448 53.6131 Totals : 6712.68433 557.29300 ------

\*\*\* End of Report \*\*\*

1260 6/10/2019 10:13:06 AM SYSTEM

Data File E:\DATA\SXS\SXS-6-151-B 2018-08-28 11-29-50\SXS-6-151-B1.D Sample Name: SXS-6-151-B-0PTICAL



1260 6/10/2019 10:14:14 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\SXS-7-10-L.D Sample Name: SXS-7-9-L-RAC



1260 6/10/2019 10:33:53 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\SXS-7-10-L1.D Sample Name: SXS-7-10-L-OPTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 35 Injection Date : 9/13/2018 3:09:26 PM Inj: 1 Inj Volume : 1.000 µl Acq. Method : E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\0DH-60-40-1ML-1uL-20MIN.M Last changed : 9/13/2018 3:06:30 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\ODH-60-40-1ML-1uL-20MIN.M ( Sequence Method) Last changed : 6/10/2019 10:33:51 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:/DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\SXS-7-10-L1.D) mAU 1 500 -400 -300 -200 -100 -6.69.9 D 6 8 10 4 min Area Percent Report Sorted Bv Sional . : 1.0000 Multiplier Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Area Height # [min] [min] [mAU\*s] [mAU] \* ----|------|-----|------|------| 1 6.699 BB 0.1629 76.10007 5.92357 0.8378 2 7.483 VV R 0.2284 9007.08984 595.54346 99.1622 Totals : 9083.18991 601.46703 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:34:41 AM SYSTEM

EtO<sub>2</sub>C CF<sub>3</sub> H<sub>2</sub>N CI (2R,3S)-**3h** 

Data File E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\SXS-7-36--37-B.D Sample Name: SXS-7-38-B-RAC ------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 92 Injection Date : 10/26/2018 3:20:53 PM Inj : 1 Inj Volume : 4.000 µl : E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\ADH-95-5-10MIN-1.0ML.M Acq. Method : 10/26/2018 3:26:46 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\ADH-95-5-10MIN-1.0ML.M ( Sequence Method) Last changed : 6/10/2019 10:19:14 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206,4 Ref=360,100 (E:DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\SXS-7-36--37-B.D) mAU · 2 800 -600 400 -200 ٥ Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] ÷ ----|-----|----|-----|-----| 
 1
 4.582
 BV
 0.1615
 1.10375e4
 957.22467
 49.6246

 2
 5.215
 VB
 0.1903
 1.12045e4
 858.08838
 50.3754
 Totals : 2.22421e4 1815.31305 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:19:16 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\SXS-7-36--37-Bl.D Sample Name: SXS-7-36-B-0PTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 2 Location : 93 Acq. Instrument : 1260 Injection Date : 10/26/2018 3:30:12 PM Inj: l Inj Volume : 4.000 µl Acq. Method : E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\ADH-95-5-10MIN-1.0ML.M Last changed : 10/26/2018 3:26:46 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\ADH-95-5-10MIN-1.0ML.M ( Sequence Method) Last changed : 9/27/2019 7:25:25 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Ref=360.100 (E-DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\SXS-7-36-37-B1.D) . And a mAU 1 1600 -Ā چ 1400 -1200 -1000 -800 -600 -400 -NG AR 200 -1997 1997 ۵ \_\_\_\_\_ Area Percent Report ------Sorted By Signal : لەسو. 1.0000 . Multiplier : Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* 
 1
 4.657 MF
 0.1844
 122.34775
 11.05521
 0.5105

 2
 5.217 FM
 0.2439
 2.38420e4
 1629.07947
 99.4895
 2.39643e4 1640.13468 Totals : \*\*\* End of Report \*\*\*

1260 9/27/2019 7:25:29 PM SYSTEM



Data File E:\DATA\SXS\SXS-7-36-37-L 2018-10-26 16-08-43\SXS-7-36--37-L.D Sample Name: SXS-7-38-L-RAC



1260 6/10/2019 10:24:32 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-36-37-L 2018-10-26 16-08-43\SXS-7-36--37-L1.D Sample Name: SXS-7-36-L-OPTICAL



EtO<sub>2</sub>C CF<sub>3</sub> H<sub>2</sub>N C Cl (2*R*,3S)-**3**i

Data File E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\SXS-7-36--37-B2.D Sample Name: SXS-7-39-B-RAC

-----Acq. Operator : SYSTEM Seq. Line : 3 Acq. Instrument : 1260 Location : 96 Inj: 1 Injection Date : 10/26/2018 3:39:40 PM Inj Volume : 4.000 µl Acq. Method : E:\DATA\SX\$\\$X\$-7-36-37-B 2018-10-26 15-19-34\ADH-95-5-10MIN-1.0ML.M Last changed : 10/26/2018 3:26:46 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\ADH-95-5-10MIN-1.0ML.M ( Sequence Method) Last changed : 6/10/2019 10:19:14 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Re=360.100 (E-WDATA/SXS/SXS.7-36-37-8 2018-10-26 15-19-34/SXS-7-36-37-82.D) mAU J ю. 8 1400 -1200 -1000 -800 -600 -400 -200 D \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By : Signal : Multiplier 1.0000 1.0000 Dilution Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* 1 4.915 VV R 0.1712 1.76970e4 1464.74426 47.6106 2 5.281 VB 0.1938 1.94732e4 1430.36621 52.3894 Totals : 3.71702e4 2895.11047 \*\*\* End of Report \*\*\*

Data File E:\DATA\SXS\SXS-7-36-37-B 2018-10-26 15-19-34\SXS-7-36--37-B3.D Sample Name: SXS-7-37-B-0PTICAL



1260 6/10/2019 10:23:28 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-36-37-L 2018-10-26 16-08-43\SXS-7-36--37-L2.D Sample Name: SXS-7-39-L-RAC

------Acq. Operator : SYSTEM Seq. Line : 3 Acq. Instrument : 1260 Location : 98 Injection Date : 10/26/2018 4:36:58 PM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-7-36-37-L 2018-10-26 16-08-43\ODH-60-40-1ML-1uL-20MIN.M Last changed : 10/26/2018 4:19:37 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-36-37-L 2018-10-26 16-08-43\0DH-60-40-1ML-1uL-20MIN.M ( Sequence Method) Last changed : 6/10/2019 10:24:29 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Re=360.100 (E:DATAX5X5X57-36-37-L 2018-10-26 16-08-43\5X5-7-36--37-L2.D) mALL 175 -794 150 -125 -100 -75 -50 · 25 D 10 min \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Signal Sorted By : Multiplier : 1.0000 1.0000 Dilution : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* 1 6.759 BV 0.2108 2638.76685 189.15868 50.6568 2 7.794 VV R 0.2475 2570.33789 154.18562 49.3432 Totals : 5209.10474 343.34430 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:26:01 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-36-37-L 2018-10-26 16-08-43\SXS-7-36--37-L3.D Sample Name: SXS-7-37-L-OPTICAL



S125

EtO<sub>2</sub>C CF<sub>3</sub> H<sub>2</sub>N

(2R,3S)-**3j** 

Data File E:\DATA\SXS\SXS-7-10-B 2018-09-14 13-59-21\SXS-7-10-B.D Sample Name: SXS-7-9-B-RAC

-----Acq. Operator : SYSTEM Seq. Line : l Location : Acq. Instrument : 1260 32 Inj: 1 Injection Date : 9/14/2018 2:00:48 PM Inj Volume : 2.000 µl Acq. Method : E:\DATA\SXS\SXS-7-10-B 2018-09-14 13-59-21\ADH-98-2-20MIN-0.5ML.M Last changed : 9/14/2018 1:59:21 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-10-B 2018-09-14 13-59-21\ADH-98-2-20MIN-0.5ML.M (Sequence Method) Last changed : 6/10/2019 10:31:09 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated
DAD1 A Sig=206.4 Ref=360.100 (E-VDATA\SXS\SXS-7-10-B 2018-09-1413-59-21\SXS-7-10-B.D) 1919° mAU 1 2 28 350 -300 -250 -200 -150 -100 -50 -Û - 50 -16 12 14 18 10 \_\_\_\_\_ Area Percent Report -----Sorted By : Signal : Multiplier 1.0000 Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* 1 13.112 FM 0.5089 1.30183e4 426.32828 50.0325 2 14.780 BB 0.4851 1.30014e4 379.13654 49.9675 Totals : 2.60198e4 805.46481 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:31:12 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-10-B 2018-09-14 13-59-21\SXS-7-10-B1.D Sample Name: SXS-7-10-0PTICAL



S127



Data File E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\SXS-7-10-L.D Sample Name: SXS-7-9-L-RAC



Data File E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\SXS-7-10-L1.D Sample Name: SXS-7-10-L-OPTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 35 Injection Date : 9/13/2018 3:09:26 PM Inj: 1 Inj Volume : 1.000 µl Acq. Method : E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\0DH-60-40-1ML-1uL-20MIN.M Last changed : 9/13/2018 3:06:30 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\ODH-60-40-1ML-1uL-20MIN.M ( Sequence Method) Last changed : 6/10/2019 10:33:51 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:/DATA\SXS\SXS-7-10-L 2018-09-13 14-55-28\SXS-7-10-L1.D) mAU 1 500 -400 -300 -200 -100 -6.69.9 D 6 8 10 4 min Area Percent Report Sorted Bv Sional . : 1.0000 Multiplier Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Area Height # [min] [min] [mAU\*s] [mAU] \* ----|------|-----|------|------| 1 6.699 BB 0.1629 76.10007 5.92357 0.8378 2 7.483 VV R 0.2284 9007.08984 595.54346 99.1622 Totals : 9083.18991 601.46703 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:34:41 AM SYSTEM

EtO<sub>2</sub>C CF<sub>3</sub> H<sub>2</sub>N CF<sub>3</sub>

(2R,3S)-**3k** 

Data File E:\DATA\SXS\SXS-9-40-B-0P 2019-11-16 10-48-14\SXS-9-40-B-0P.D Sample Name: SXS-9-41-B-RAC \_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location: 11 Injection Date : 11/16/2019 10:49:41 AM Inj : 1 Inj Volume : 4.000 µl : E:\DATA\SXS\SXS-9-40-B-OP 2019-11-16 10-48-14\ADH-95-5-10MIN-1.0ML.M Acq. Method : 11/16/2019 11:04:59 AM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-9-40-B-0P 2019-11-16 10-48-14\ADH-95-5-10MIN-1.0ML.M ( Sequence Method) : 11/18/2019 7:39:56 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A. Sig=206,4 Re←360,100 (E:\DATA\SXS\SXS-9-40-B-OP 2019-11-16 10-48-14\SXS-9-40-B-OP.D) . mAU 400 300 200 100 ٥ 12 14 10 Area Percent Report Sorted By : Signal Multiplier 1.0000 : : 1.0000 Dilution Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area [min] [mAU\*s] [mAU] \* # [min] - | - - - - - - - | 
 1
 4.673 MF
 0.1138 3208.22437 470.04437 47.8499

 2
 5.024 FM
 0.1210 3496.54639 481.81531 52.1501
 Totals : 6704.77075 951.85968 ------\*\*\* End of Report \*\*\*

1260 11/18/2019 7:40:01 PM SYSTEM

Data File E:\DATA\SXS\SXS-9-40-B-OP 2019-11-16 10-48-14\SXS-9-40-B-OP2.D Sample Name: SXS-9-40-B-OP



## Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000
Do not use Multinlier	s.	Dilution Factor with ISTDs

## Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.671	MF	0.1168	3705.70386	528.98901	97.7545
2	5.008	FM	0.1515	85.12424	9.36646	2.2455

Totals: 3790.82809 538.35547

\*\*\* End of Report \*\*\*

1260 11/18/2019 7:47:31 PM SYSTEM



Data File E:\DATA\SXS\SXS-9-40-L 2019-11-16 16-53-28\SXS-9-40-L.D Sample Name: SXS-9-41-L-RAC

Acq. Operator :	SYSTEM	Seq. Line : 1
Acq. Instrument :	1260	Location : 13
Injection Date :	11/16/2019 4:54:59 PM	Inj: 1
		Inj Volume : 6.000 µl
Acq. Method :	E:\DATA\SXS\SXS-9-40-L	2019-11-16 16-53-28\0DH-60-40-1ML-3uL-100MIN-264NM.M
Last changed :	11/16/2019 4:55:11 PM b	DY SYSTEM
	(modified after loading	1)
Analysis Method :	E:\DATA\SXS\SXS-9-40-L	2019-11-16 16-53-28\0DH-60-40-1ML-3uL-100MIN-264NM.M
	(Sequence Method)	
Last changed :	11/18/2019 7:50:03 PM b	DY SYSTEM
	(modified after loading	1)
Additional Info :	Peak(s) manually integr	ated
DAD1 A, Sig=264	4,4 Ref=360,100 (E:\DATA\SXS\SXS-9-4	40-L 2019-11-16 16-53-28\SX S-9-40-L.D)
mAU 1		u u u u u u u u u u u u u u u u u u u
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	Area Percent Rep	oort
Sorted By	: Signal	
Multiplier	: 1.0000	
Dilution	: 1.0000	
Do not use Multipl	lier & Dilution Factor w	vith ISTDs
Signal 1: DAD1 A,	Sig=264,4 Ref=360,100	
Peak RetTime Type	Width Area He	ight Area
# [min]	[min] [mAU*s] [m	DAU] %
1 5 500 80		
I 3.390 BV	 0.1523 1913.27295 182	2.45921 50.5135
2 6.197 VB	 0.1523 1913.27295 182 0.1761 1874.37561 154	2.45921 50.5135 1.94633 49.4865
2 6.197 VB	 0.1523 1913.27295 182 0.1761 1874.37561 154	2.45921 50.5135 1.94633 49.4865
2 6.197 VB	 0.1523 1913.27295 182 0.1761 1874.37561 154 3787.64856 337	2.45921 50.5135 1.94633 49.4865 7.40555
2 6.197 VB	 0.1523 1913.27295 182 0.1761 1874.37561 154 3787.64856 337	2.45921 50.5135 1.94633 49.4865 7.40555
1 5.390 BV 2 6.197 VB Totals :	 0.1523 1913.27295 182 0.1761 1874.37561 154 3787.64856 337	2.45921 50.5135 1.94633 49.4865 7.40555
1 5.390 BV 2 6.197 VB Totals :	 0.1523 1913.27295 182 0.1761 1874.37561 154 3787.64856 337	2.45921 50.5135 1.94633 49.4865 7.40555
2 6.197 VB	 0.1523 1913.27295 182 0.1761 1874.37561 154 3787.64856 337 *** End of Repo	2.45921 50.5135 1.94633 49.4865 7.40555
1 5.390 BV 2 6.197 VB Totals :	 0.1523 1913.27295 182 0.1761 1874.37561 154 3787.64856 337 *** End of Repo	2.45921 50.5135 1.94633 49.4865 2.40555
1 5.390 BV 2 6.197 VB Totals :	 0.1523 1913.27295 182 0.1761 1874.37561 154 3787.64856 337 *** End of Repo	2.45921 50.5135 1.94633 49.4865 2.40555

Data File E:\DATA\SXS\SXS-9-40-L 2019-11-16 16-53-28\SXS-9-40-L1.D Sample Name: SXS-9-40-L-OP

Acq. Operator : SYSTEM Seq. Line : 2 Location : 14 Acq. Instrument : 1260 Injection Date : 11/16/2019 5:06:24 PM Inj: l Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-9-40-L 2019-11-16 16-53-28\0DH-60-40-1ML-3uL-100MIN-264NM.M Last changed : 11/16/2019 4:55:11 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-9-40-L 2019-11-16 16-53-28\0DH-60-40-1ML-3uL-100MIN-264NM.M (Sequence Method) Last changed : 11/18/2019 7:50:03 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=264.4 Ref=360.100 (E:/DATA\SXS\SXS-9-40-L 2019-11-16 16-53-28\SXS-9-40-L1.D) mAU 500 -400 -300 -200 100 5.715 D 6 \_\_\_\_\_ Area Percent Report -----Sorted By Signal : 1.0000 Multiplier : Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=264,4 Ref=360,100 Peak RetTime Type Width Area Height Area [mAU] # [min] [min] [mAU\*s] \* 1 5.715 BV E 0.1933 88.97283 6.59959 1.5075 2 6.241 VB R 0.1660 5813.20215 525.91736 98.4925 5902.17498 532.51695 Totals : \*\*\* End of Report \*\*\*

1260 11/18/2019 7:50:31 PM SYSTEM

EtO<sub>2</sub>C CF<sub>3</sub> H<sub>2</sub>N NO<sub>2</sub> (2*R*,3S)-**3**I

Data File E:\DATA\SXS\SXS-9-49-B 2019-12-11 15-22-28\SXS-9-49-B.D

Sample Name: SXS-9-48-B-RAC

-----Acq. Operator : SYSTEM Seq. Line : 1 Location : Acq. Instrument : 1260 71 Inj: 1 Injection Date : 12/11/2019 3:23:54 PM Inj Volume : 4.000 µl Acq. Method : E:\DATA\SXS\SXS-9-49-B 2019-12-11 15-22-28\ADH-95-5-10MIN-1.0ML.M Last changed : 12/11/2019 3:22:28 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-9-49-B 2019-12-11 15-22-28\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 12/11/2019 4:05:00 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Re=360,100 (E-DATA\SX\$\SX\$-9-49-8 2019-12-11 15-22-28\SX\$-9-49-8.D) mAU -ł 180 -160 -140 -120 -100 -80 -6D · 40 -20 10 12 14 mi Area Percent Report \_\_\_\_\_ Sorted By Signal : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Height [mAU] Peak RetTime Type Width Area Area # [min] [min] [mAU\*s] \* ----|-----|----|-----|-----|-----| 1 10.202 BV 0.1988 2357.89307 177.68752 47.7610 2 10.988 VV R 0.2195 2578.96875 177.51172 52.2390 Totals : 4936.86182 355.19923

\*\*\* End of Report \*\*\*

1260 12/11/2019 4:05:04 PM SYSTEM

Data File E:\DATA\SXS\SXS-9-49-B 2019-12-11 15-22-28\SXS-9-49-B1.D Sample Name: SXS-9-49-B-0P

Acq. Operator : SYSTEM Seq. Line : 2 Location : 72 Acq. Instrument : 1260 Injection Date : 12/11/2019 3:40:17 PM Inj: l Inj Volume : 4.000 µl : E:\DATA\SXS\SXS-9-49-B 2019-12-11 15-22-28\ADH-95-5-10MIN-1.0ML.M Acg. Method Last changed : 12/11/2019 3:22:28 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-9-49-B 2019-12-11 15-22-28\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 12/11/2019 4:05:00 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Ref=360.100 (E:0ATA:SXS:SXS-9-49-B 2019-12-11 15-22-28:SXS-9-49-B1.D) AS139 mAU 1 300 Ì 250 -200 -150 -100 -. W. TANA 50 옾 n ł 4 6 8 10 12 14 min Area Percent Report Signal Sorted By : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] \* 1 10.288 MF 0.2230 4332.39453 323.77652 98.1704 2 11.049 FM 0.3774 80.74439 3.56591 1.8296 Totals : 4413.13892 327.34243 \*\*\* End of Report \*\*\*

1260 12/11/2019 4:08:14 PM SYSTEM

EtO<sub>2</sub>C, CF<sub>3</sub> H<sub>2</sub>N

(2R,3S)-3m

Data File E:\DATA\SXS\SXS-7-13-B 2018-09-22 08-42-51\SXS-7-13-B-0PTICAL.D Sample Name: SXS-7-15-B-RAC

-----Acq. Operator : SYSTEM Seq. Line : l Location : Acq. Instrument : 1260 96 Inj: 1 Injection Date : 9/22/2018 8:44:22 AM Inj Volume : 2.000 µl Acq. Method : E:\DATA\SXS\SXS-7-13-B 2018-09-22 08-42-51\ID-95-5-2UL-10MIN.M Last changed : 9/22/2018 8:42:52 AM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-13-B 2018-09-22 08-42-51\ID-95-5-2UL-10MIN.M (Sequence Method) Last changed : 6/10/2019 10:36:25 AM by SYSTEM (modified after loading) DAD1 B, Sig=290,4 Ref=360,100 (E:\DATA\SXS\SXS-7-13-B 2018-09-22 08-42-51\SXS-7-13-B-0 PTICAL.D) mAU -7.358 40 -30 -20 -10 0 7 Ŕ 4 d \_\_\_\_\_ Area Percent Report Signal Sorted By : Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=290,4 Ref=360,100 Height Peak RetTime Type Width Area Area # [min] [min] [mAU\*s] [mAU] ÷ 1 6.264 BB 0.1446 450.28738 47.09322 50.3912 2 7.358 BB 0.1690 443.29584 39.46557 49.6088 Totals : 893.58322 86.55879 \*\*\* End of Report \*\*\*

Data File E:\DATA\SXS\SXS-7-13-B 2018-09-22 08-42-51\SXS-7-13-B-0PTICAL1.D Sample Name: SXS-7-13-B-0PTICAL

Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 97 Injection Date : 9/22/2018 8:55:47 AM Inj: 1 Inj Volume : 2.000 µl Acq. Method : E:\DATA\SX\$\SX\$-7-13-B 2018-09-22 08-42-51\ID-95-5-2UL-10MIN.M Last changed : 9/22/2018 8:42:52 AM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-13-B 2018-09-22 08-42-51\ID-95-5-2UL-10MIN.M (Sequence Method) Last changed : 6/10/2019 10:36:25 AM by SYSTEM (modified after loading) DAD1 B, Sig=290,4 Ref=360,100 (E:/DATA\SXS\SXS-7-13-B 2018-09-22 08-42-51\SXS-7-13-B-0 PTICAL1.D) mAU · 100 80 -60 -40 -20 -٥ 6 7 8 9 5 шi Area Percent Report \_\_\_\_\_ Sorted By Multiplier : Signal 1.0000 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=290,4 Ref=360,100 Peak RetTime Type Width Height Area Area [min] [mAU\*s] [mAU] # [min] \* 1 7.132 BB 0.1711 1251.39746 111.34167 100.0000 Totals : 1251.39746 111.34167 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:37:43 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\SXS-7-12-13-L4.D Sample Name: SXS-7-15-L-RAC

------Acq. Operator : SYSTEM Seq. Line : 5 Acq. Instrument : 1260 Location : 94 Injection Date : 9/21/2018 3:08:25 PM Inj: l Inj Volume : 4.000 µl : E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\0DH-60-40-1ML-1uL-20MIN.M Acg. Method Last changed : 9/21/2018 3:04:01 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\0DH-60-40-1ML-1uL-20MIN.M ( Sequence Method) Last changed : 6/10/2019 10:42:02 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206,4 Ref=360,100 (E:DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\SXS-7-12-13-L4.D) mALL 1 13 804 120 -100 -80 -60 -40 20 0 12 14 10 16 \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Signal Sorted By : Multiplier : 1.0000 1.0000 Dilution : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* 1 11.730 BV R 0.3869 3642.52759 129.58212 50.0880 2 13.804 BV R 0.4246 3629.73145 108.97816 49.9120 Totals : 7272.25903 238.56028 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:42:05 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\SXS-7-12-13-L5.D Sample Name: SXS-7-15-L-0PTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 6 Location : 95 Acq. Instrument : 1260 Injection Date : 9/21/2018 3:27:51 PM Inj: l Inj Volume : 4.000 µl Acq. Method : E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\ODH-60-40-1ML-1uL-20MIN.M Last changed : 9/21/2018 3:04:01 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\0DH-60-40-1ML-1uL-20MIN.M ( Sequence Method) Last changed : 9/27/2019 7:32:48 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Re=360,100 (E-WDATA\SXS\SXS-7-12-13-L 2018-09-21 14-10-29\SXS-7-12-13-L5.D) mAU -829 120 -100 -80 -60 -40 -Los HAND 20 88 ٥ 12 16 10 14 min 8 \_\_\_\_\_ Area Percent Report -----Sorted By Signal Sorted By Multiplier : : 1.0000 . Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* 1 11.893 MM 0.5139 44.81595 1.45343 1.0241 2 13.791 VV R 0.4213 4331.45166 130.12854 98.9759 4376.26761 131.58197 Totals : \*\*\* End of Report \*\*\*

1260 9/27/2019 7:32:53 PM SYSTEM



Data File E:\DATA\SXS\SXS-7-50-B-RAC 2018-11-17 09-28-41\SXS-7-50-B-RAC2.D Sample Name: SXS-7-50-B-RAC



1260 9/21/2019 9:32:53 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-50-B-RAC 2018-11-17 09-28-41\SXS-7-50-B-RAC1.D Sample Name: HYZ-1-41-B-OPTICAL

Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 94 Injection Date : 11/17/2018 9:41:27 AM Inj: 1 Inj Volume : 1.000 µl : E:\DATA\SXS\SXS-7-50-B-RAC 2018-11-17 09-28-41\ASH-95-5-206NM-1.0M.M Acq. Method Last changed : 11/17/2018 9:48:59 AM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-50-B-RAC 2018-11-17 09-28-41\ASH-95-5-206NM-1.0M.M ( Sequence Method) Last changed : 9/21/2019 9:32:32 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\\$X\$\\$X\$-7-50-B-RAC 2018-11-17.09-28-41\\$X\$-7-50-B-RAC1.D) mAU 175 -150 -125 -100 -75 -50 -25 ρ. 6 4 5 Area Percent Report \_\_\_\_\_ Sorted By Signal : : Multiplier 1.0000 Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Height Peak RetTime Type Width Area Area # [min] [min] [mAU\*s] [mAU] ÷ ----|-----|----|-----|-----|-----|-----| 1 5.015 VB R 0.1265 1533.20239 181.60826 100.0000 Totals : 1533.20239 181.60826 \*\*\* End of Report \*\*\*

1260 9/21/2019 9:37:16 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-53-L-HYZ-1-41-L 2018-11-15 21-24-18\SXS-7-53-L-HYZ-1-41-L.D Sample Name: SXS-7-50-L-RAC

Acq. Operator : S	SYSTEM	Seq. Line :	1
Acq. Instrument : ]	1260	Location :	97
Injection Date : 1	11/15/2018 9:25:49 1	M Ini:	1
		Thi Volume : 3	- .000 ul
Acq. Method : E	E:\DATA\SXS\SXS-7-53 20MIN.M	3-L-HYZ-1-41-L 2018-11-15	21-24-18\ODH-60-40-1ML-1uL-
Last changed : 1	L1/15/2018 9:33:54 B	M by SYSTEM	
	(modified after load	ling)	
Analysis Method : H	:\DATA\SXS\SXS-7-53	8-L-HYZ-1-41-L 2018-11-15	21-24-18\0DH-60-40-1ML-1uL-
2	20MIN.M (Sequence Me	ethod)	
Last changed : 6	5/10/2019 10:52:22 <i>4</i>	AM by SYSTEM	
	(modified after load	ling)	
Additional Info : H	Peak(s) manually int	tegrated	
DAD1 A, Sig=206,4	4 Ref=360,100 (E:\DATA\SX53	3-L-H YZ-1-41-L 2018-11-15 21-24-18\SX S-	7-53-L-HYZ-1-41- L.D)
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Sorted By	2 4 Area Percent : Signal	Report	 
Sorted By Multiplier	4 2 4 Area Percent : Signal : 1.0000	Report	 10 12 14 min 
Sorted By Multiplier Dilution	4 2 4 Area Percent : Signal : 1.0000 : 1.0000	Report	 10 12 14 min 
Sorted By Multiplier Dilution Do not use Multipli	 2 4 Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto	Report	
Sorted By Multiplier Dilution Do not use Multipli	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Factor	Report	
Sorted By Multiplier Dilution Do not use Multipli	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Factor	Report	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DAD1 A, S	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10	Report	 10 12 14 min =======
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DAD1 A, S Peak RetTime Type	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10 Width Area	Report or with ISTDs 10 Height Area	 10 12 14 min 
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DAD1 A, S Peak RetTime Type # [min]	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10 Width Area [min] [mAU*s]	Report or with ISTDs 10 Height Area [mAU] %	 10 12 14 min 
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min]	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10 Width Area [min] [mAU*s]	Report or with ISTDs 00 Height Area [mAU] %	 10 12 14 min 
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DAD1 A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 	6       8         Report         or with ISTDs         00         Height Area [mAU] %         293.43491 47.1231	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Factor Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 	6       8         Report         or with ISTDs         00         Height Area         [mAU] %	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 	6       8         Report       8         or with ISTDs       8         00       Height Area         [mAU]       %         293.43491       47.1231         228.68065       52.8769	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 	6       8         Report         or with ISTDs         00         Height Area [mAU] %         293.43491 47.1231 228.68065 52.8769         522.11555	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Facto Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 	6       8         Report         or with ISTDs         00         Height Area [mAU] %         293.43491 47.1231         228.68065 52.8769         522.11555	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Factor Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 	6       8         Report       8         or with ISTDs       8         00       Height Area [mAU] %         293.43491 47.1231       228.68065 52.8769         522.11555       522.11555	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier « Dilution Factor Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 0.1702 2996.99609 0.2116 3362.93652 6359.93262	6       8         Report         or with ISTDs         00         Height Area [mAU] %         293.43491 47.1231 228.68065 52.8769         522.11555	
Sorted By Multiplier Dilution Do not use Multipli Signal 1: DADI A, S Peak RetTime Type # [min] 	Area Percent : Signal : 1.0000 : 1.0000 ier & Dilution Factor Sig=206,4 Ref=360,10 Width Area [min] [mAU*s] 0.1702 2996.99609 0.2116 3362.93652 6359.93262 **** End of F	6       8         Report         or with ISTDs         00         Height Area         [mAU]         293.43491         47.1231         228.68065         522.11555         Report ***	

1260 6/10/2019 10:52:25 AM SYSTEM

Data File E:\DATA\SXS\SXS-7-53-L-HYZ-1-41-L 2018-11-15 21-24-18\SXS-7-53-L-HYZ-1-41-L1.D Sample Name: HYZ-1-41-L-OPTICAL

Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 98 Injection Date : 11/15/2018 9:42:20 PM Inj: 1 Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-53-L-HYZ-1-41-L 2018-11-15 21-24-18\ODH-60-40-1ML-luL-Acq. Method 20MIN.M Last changed : 11/15/2018 9:33:54 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-53-L-HYZ-1-41-L 2018-11-15 21-24-18\ODH-60-40-1ML-1uL-20MIN.M (Sequence Method) Last changed : 6/10/2019 10:52:22 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:/DATA\\$X...53-L-HYZ-1-41-L 2018-11-15 21-24-18\\$X\$-7-53-L-HYZ-1-41-L1.D) mAU 300 -250 -200 -150 -100 -50 902 0 ż 6 ś 10 12 14 min \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : : Multiplier 1.0000 Dilution 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [mAU\*s] [mAU] \* ----|-----|-----|-----|-----| 1 5.902 VV R 0.1331 66.92931 6.21395 1.3541 2 7.643 VV R 0.2233 4875.76465 328.26788 98.6459 Totals : 4942.69396 334.48183 \_\_\_\_\_ \*\*\* End of Report \*\*\*

1260 6/10/2019 10:53:28 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-32-B 2018-10-19 10-52-15\SXS-7-32-B.D Sample Name: SXS-7-29-B-RAC

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Additional Info :	Peak(s)	manually in	tegrated						
DAD1 A, Sig=20	06,4 Ref=360,1	00 (E:\DATA\SXS\S	XS-7-32-8 2018-1	0-19 10-52-15\SX	(S-7-32	-B.D)			
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Sorted By Multiplier Dilution Do not use Multip Signal 1: DAD1 A, Peak RetTime Type # [min]		Area Percent Signal 1.0000 1.0000 ilution Fact ,4 Ref=360,1 Area [mAU*s]	Report cor with IST 00 Height [mAU]	Ds Area	) 		 - 1 - 7	* 1 • • • 8	
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Sorted By Multiplier Dilution Do not use Multip Signal 1: DAD1 A, Peak RetTime Type # [min] 	: ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ;	Area Percent Signal 1.0000 1.0000 ilution Fact ,4 Ref=360,1 Area [mAU*s] 	4 Report or with IS7 00 Height [mAU] 	Area * 47.7083 52.2917			 1 7 7	* 1 * * * * 8	* 1 min
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Sorted By Multiplier Dilution Do not use Multip Signal 1: DAD1 A, Peak RetTime Type # [min] 	: ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ;	2 3 Area Percent Signal 1.0000 1.0000 ilution Fact ,4 Ref=360,1 Area [mAU*s] 1	4 Report or with IST 00 Height [mAU] 269.59311 261.18146 530.77457	Ds Area * 47.7083 52.2917				• 1 • • •	+ 1 min
Sorted By Multiplier Dilution Do not use Multip Signal 1: DADI A, Peak RetTime Type # [min] 	: : : : : : : : : : : : : : : : : : :	Area Percent Signal 1.0000 1.0000 ilution Fact ,4 Ref=360,1 Area [mAU*s] 	4 Report or with IS7 00 Height [mAU] 269.59311 261.18146 530.77457	Area * 47.7083 52.2917	)		 	• 1 • • • • 8	- I min
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1260 6/10/2019 10:54:57 AM SYSTEM
Data File E:\DATA\SXS\SXS-7-32-B 2018-10-19 10-52-15\SXS-7-32-B1.D Sample Name: SXS-7-32-B-OPTICAL

Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 2 Injection Date : 10/19/2018 11:04:16 AM Inj: 1 Inj Volume : 4.000 µl Acq. Method : E:\DATA\SXS\SXS-7-32-B 2018-10-19 10-52-15\ADH-95-5-10MIN-1.0ML.M Last changed : 10/19/2018 11:02:52 AM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-32-B 2018-10-19 10-52-15\ADH-95-5-10MIN-1.0ML.M (Sequence Method) Last changed : 6/10/2019 10:54:54 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:DATA\SXS\SXS-7-32-B 2018-10-19 10-52-15\SXS-7-32-B1.D) mAU -400 -350 -300 -250 -200 -150 -100 -50 -٥ 4 5 6 8 3 Area Percent Report Sorted By Sional : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----|-----| 1 5.831 BB 0.1953 5785.23975 431.73294 100.0000 Totals : 5785.23975 431.73294 \*\*\* End of Report \*\*\*

1260 6/10/2019 10:57:08 AM SYSTEM



Data File E:\DATA\SXS\SXS-7-32-L 2018-10-19 11-22-22\SXS-7-32-L.D Sample Name: SXS-7-29-L-RAC

Acg. Operator : SYSTEM Seg. Line : 1	
Acq. Instrument : 1260 Location : 3	
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Last changed : 6/10/2019 10:58:31 AM by SYSTEM	
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Additional Info : Peak(s) manually integrated	
DAD1 A, Sig=206,4 Ref=360,100 (E:\DATA\SX S\SX S-7-32-L 2018-10-19 11-22-22\SX S-7-32-L.D)	
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Image: constraint of the second se	min

Data File E:\DATA\SXS\SXS-7-32-L 2018-10-19 11-22-22\SXS-7-32-L1.D Sample Name: SXS-7-32-L-OPTICAL



S147



## (2S\*,3R\*)-**3p** + (2R\*,3R\*)-**4p**

Data File E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT.D Sample Name: SXS-8-89-RAC-DIVERGENT

------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 92 Injection Date : 6/8/2019 6:28:10 PM Inj : 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220**NM.M** Last changed : 6/8/2019 6:26:50 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220NM.M (Sequence Method) Last changed : 6/10/2019 3:44:37 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 B, Sig=206,4 Ref=360,100 (E:DATA\SX...9-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT.D) mAU -1400 -1200 -202 1000 -589 800 -626 600 -400 200 D \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [mAU\*s] [mAU] \* 1 4.589 BV R 0.1109 6017.45068 776.55109 15.0179 2 5.918 BV 0.1463 1.53486e4 1540.30920 38.3059 3 6.626 VV 0.1619 6256.44971 575.01453 15.6144 4 7.202 VB 0.1935 1.24460e4 952.11890 31.0619 Totals : 4.00686e4 3843.99371 \_\_\_\_\_ \*\*\* End of Report \*\*\*

1260 6/10/2019 3:44:39 PM SYSTEM



Data File E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT4.D Sample Name: 2-C1-S-2-2-OPTICAL ------Acq. Operator : SYSTEM Seq. Line : 5 Acq. Instrument : 1260 Location : 96 Injection Date : 6/8/2019 7:14:06 PM Inj : 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220**NM.M** Last changed : 6/8/2019 6:26:50 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220NM.M (Sequence Method) Last changed : 9/23/2019 5:13:01 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 8, Sig=206,4 Ref=360,100 (E:DATA(SX...-RAC-DIMERGENT 2019-06-08 18-26-49(SXS-8-89-RAC-DIMERGENT4.D) mAU 7 2000 -1750 -1500 -1250 -1000 -750 500 250 5. ٥ -------Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier 1.0000 : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* 1 5.971 MM 0.1685 151.03429 14.93822 0.5177 2 6.587 BV R 0.2100 2.90253e4 2154.02832 99.4823 Totals : 2.91764e4 2168.96654 \*\*\* End of Report \*\*\*

1260 9/23/2019 5:13:03 PM SYSTEM



Data File E:\DATA\SXS\SXS-6-147-RAC 2019-09-21 17-13-32\SXS-6-147.D Sample Name: SXS-6-147-RAC

Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 94 Injection Date : 9/21/2019 5:14:56 PM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-6-147-RAC 2019-09-21 17-13-32\0DH-60-40-1ML-3uL-20MIN-256NM . М : 9/21/2019 5:27:06 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-6-147-RAC 2019-09-21 17-13-32\0DH-60-40-1ML-3uL-20MIN-256NM .M (Sequence Method) Last changed : 9/21/2019 5:46:05 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated
DAD1 A Sig=266.4 Re+360,100 (E-DATA\\$X\$\\$X\$-6-147.RAC 2019-09-21 17-13-32\\$X\$-6-147.D) mAU -300 -690 250 -200 -150 -10.579 100 -50 ٥ 4 6 ś 10 12 mir \_\_\_\_\_ Area Percent Report Sorted By : Signal 1.0000 Multiplier : 1.0000 Dilution : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=256,4 Ref=360,100 Height Peak RetTime Type Width Area Area # [min] [min] [mAU\*s] [mAU] \* 
 1
 8.069 BB
 0.3046
 5753.21045
 277.78970
 66.6445

 2
 10.579 BB
 0.4112
 2879.47070
 102.37064
 33.3555
 Totals : 8632.68115 380.16035 \*\*\* End of Report \*\*\* 1260 9/21/2019 5:46:10 PM SYSTEM Page 1 of 1

Data File E:\DATA\SXS\SXS-7-53-L-HYZ-1-41-L 2018-11-16 14-37-52\SXS-7-53-L-HYZ-1-41-L1.D Sample Name: SXS-7-53-L-OPTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 100 Injection Date : 11/16/2018 3:05:56 PM Inj: 1 Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-53-L-HYZ-1-41-L 2018-11-16 14-37-52\0DH-60-40-1ML-1uL-Acq. Method 20MIN.M Last changed : 11/16/2018 3:21:52 PM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-53-L-HYZ-1-41-L 2018-11-16 14-37-52\0DH-60-40-1ML-1uL-20MIN.M (Sequence Method) Last changed : 9/21/2019 5:49:57 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:/DATA\SX...53-L-HYZ-1-41-L 2018-11-16 14-37-52\SXS-7-53-L-HYZ-1-41-L1.D) mAU 500 -400 -300 -200 -100 -8.114 ٥ 12 14 16 ó. 10 18 Ŕ mi Area Percent Report Sorted By Signal : : Multiplier 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area [min] [mAU\*s] # [min] [mAU] \* ----|-----|----|-----|-----|-----|-----| 1 8.114 BV R 0.1968 261.20016 15.97343 2.0704 2 10.559 BV R 0.3047 1.23548e4 542.43231 97.9296 Totals : 1.26160e4 558.40574 \_\_\_\_\_ \*\*\* End of Report \*\*\* Page 1 of 1 1260 9/21/2019 5:50:00 PM SYSTEM



Data File E:\DATA\SXS\SXS-7-73-L-OPTICAL 2019-05-20 11-23-40\SXS-7-73-L-OPTICAL.D Sample Name: SXS-7-77-L-RAC

------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 34 Injection Date : 5/20/2019 11:25:07 AM Inj: l Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-7-73-L-OPTICAL 2019-05-20 11-23-40\0DH-60-40-1ML-1uL-20MIN. М : 5/20/2019 11:36:06 AM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-73-L-OPTICAL 2019-05-20 11-23-40\ODH-60-40-1ML-1uL-20MIN. M (Sequence Method) Last changed : 6/10/2019 11:08:03 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Ref=360,100 (E-DATA\SXS\SXS-7-73-L-OPTICAL 2019-05-20 11-23-40\SXS-7-73-L-OPTICAL.D) mAU -600 -88 500 -400 -300 -200 -100 -D 6 10 \_\_\_\_\_ Area Percent Report Sorted By : Signal 1.0000 : Multiplier 1.0000 Dilution : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Height Area Peak RetTime Type Width Area # [min] [min] [mAU\*s] [mAU] \* 1 6.635 BB 0.2823 1.30237e4 662.72284 49.9847 2 8.698 BB 0.3693 1.30316e4 506.31525 50.0153 Totals : 2.60553e4 1169.03809 \*\*\* End of Report \*\*\* 1260 6/10/2019 11:08:07 AM SYSTEM Page 1 of 1

Data File E:\DATA\SXS\SXS-7-73-L-OPTICAL 2019-05-20 11-23-40\SXS-7-73-L-OPTICAL1.D Sample Name: SXS-7-73-L-OPTICAL

\_\_\_\_\_ Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 35 Injection Date : 5/20/2019 11:37:36 AM Inj: 1 Inj Volume : 2.000 µl : E:\DATA\SXS\SXS-7-73-L-OPTICAL 2019-05-20 11-23-40\0DH-60-40-1ML-1uL-20MIN. Acq. Method M Last changed : 5/20/2019 11:48:09 AM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-73-L-OPTICAL 2019-05-20 11-23-40\0DH-60-40-1ML-1uL-20MIN. M (Sequence Method) Last changed : 6/10/2019 11:08:03 AM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=206,4 Ref=360,100 (E:/DATA\SXS\SXS-7-73-L-0 PTICAL 2019-05-20 11-23-40\SXS-7-73-L-0 PTICAL1.D) mAU 500 400 -300 -200 -100 -835 ٥ 10 min Area Percent Report Sorted By Signal : : Multiplier 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area [min] [mAU\*s] # [min] [mAU] \* ----|-----|----|-----|-----|-----|-----| 1 6.835 BB 0.2958 163.33990 6.76190 1.0808 2 8.779 BB 0.3789 1.49492e4 572.02917 98.9192 1.51125e4 578.79107 Totals : \_\_\_\_\_ \*\*\* End of Report \*\*\* Page 1 of 1 1260 6/10/2019 11:09:09 AM SYSTEM



(2S\*,3*R*\*)-3a + (2*R*\*,3*R*\*)-4a

Data File E:\DATA\SX...S-6-125-RAC-DIVERGENT 2019-09-26 11-24-49\SXS-6-125-RAC-DIVERGENT.D Sample Name: SXS-6-125-RAC

Aca Operator · «VETEM Sea Line · 1
Acq. Instrument • 1260 Josef on • 95
$\begin{array}{cccccccccccccccccccccccccccccccccccc$
$h_{\text{res}}$ we the $h_{\text{res}}$ . F. (DATA) GVG GVG C 125 DAG DITERCENT 2010 00 25 11 24 40) AGU 00 1 205 W 200
Acq. method : E: \DATA 3555\555-5-125-KAC-DIVERGENI 2019-09-26 11-24-49\A5H-99-1-206NM-30L-
Last changed : 9/26/2019 11:33:17 AM by SYSTEM
(modified after loading)
Analysis Method : E:\DATA\SXS\SXS-6-125-RAC-DIVERGENT 2019-09-26 11-24-49\ASH-99-1-206NM-3UL-
1.0M.M (Sequence Method)
Last changed : 9/26/2019 11:36:25 AM by SYSTEM
(modified after loading)
Additional Info : Peak(s) manually integrated
DAD1 A, Sig=206,4 Ref=360,100 (E:/DATA(SXRAC-DMERGENT 2019-09-26 11-24-49/SXS-6-125-RAC-DI/VERGENT.D)
<sup>4</sup> ₩ ] /\/   <sup>3</sup> / <sub>2</sub> →
1 <sup>300</sup> 1 (1 \ \\ \\
200-] / \ \ \ \ \
100-1 J Y \
<u>1 2 3 4 6 6 mi</u>
Area Davaart Dowart
Sorted By : Signal
Multipiler : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
Signal 1: DAD1 A, Sig=206,4 Ref=360,100
Peak RetTime Type Width Area Height Area
# [min] [mAU*s] [mAU] %
1 4.433 BV 0.1284 3601.51636 435.70584 21.0757
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182 4 5.194 VB 0.1959 3999.95068 299.35025 23.4073
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182 4 5.194 VB 0.1959 3999.95068 299.35025 23.4073
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182 4 5.194 VB 0.1959 3999.95068 299.35025 23.4073 Totals : 1 7088464 1567 34488
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182 4 5.194 VB 0.1959 3999.95068 299.35025 23.4073 Totals : 1.70884e4 1567.34488
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182 4 5.194 VB 0.1959 3999.95068 299.35025 23.4073 Totals : 1.70884e4 1567.34488
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182 4 5.194 VB 0.1959 3999.95068 299.35025 23.4073 Totals : 1.70884e4 1567.34488
2 4.602 VV 0.1687 5844.02393 513.75665 34.1987 3 4.898 VV 0.1714 3642.95142 318.53214 21.3182 4 5.194 VB 0.1959 3999.95068 299.35025 23.4073 Totals : 1.70884e4 1567.34488

1260 9/26/2019 11:36:28 AM SYSTEM



Data File E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\SXS-8-88-RAC-DIVERGENT4.D Sample Name: Ph-S-2-2-0PTICAL

Acq. Operator : SYSTEM Seq. Line : 5 Acq. Instrument : 1260 Location : 96 Injection Date : 6/6/2019 6:58:34 PM Inj: l Inj Volume : 5.000 µl Acq. Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M Last changed : 6/6/2019 6:30:12 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M (Sequence Method) Last changed : 6/10/2019 3:51:14 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Ref=360.100 (E:DATA(SX...-RAC-DIMERGENT 2019-06-06 18-23-02(SXS-8-88-RAC-DIMERGENT4.D) mAU ] 1400 -1200 -1000 -800 -600 -400 -200 ρ. Area Percent Report Sorted By Signal : : Multiplier 1.0000 1.0000 Dilution Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [mAU\*s] [mAU] ÷ 1 4.844 BB 0.1903 1.96154e4 1574.79761 100.0000 Totals : 1.96154e4 1574.79761 \*\*\* End of Report \*\*\*

1260 6/10/2019 3:52:49 PM SYSTEM



Data File E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\SXS-8-88-RAC-DIVERGENT3.D Sample Name: Ph-S-1-2-0PTICAL

Acq. Operator : SYSTEM Seq. Line : 4 Acq. Instrument : 1260 Location : 95 Injection Date : 6/6/2019 6:49:58 PM Inj: l Inj Volume : 5.000 µl Acq. Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M Last changed : 6/6/2019 6:30:12 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M (Sequence Method) Last changed : 6/10/2019 3:51:14 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Ref=360.100 (E:DATA(SX...-RAC-DIMERGENT 2019-06-06 18-23-02(SXS-8-88-RAC-DIMERGENT3.D) mAU . 600 -500 -400 -300 -200 -100 -۵ Area Percent Report Sorted By Signal : : Multiplier 1.0000 1.0000 Dilution Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area ÷ # [min] [mAU\*s] [mAU] 1 5.223 BB 0.2028 8948.10059 674.41937 100.0000 Totals : 8948.10059 674.41937 \*\*\* End of Report \*\*\*

1260 6/10/2019 3:52:23 PM SYSTEM



Data File E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\SXS-8-88-RAC-DIVERGENT2.D Sample Name: Ph-R-2-2-OPTICAL

Acq. Operator : SYSTEM Seq. Line : 3 Acq. Instrument : 1260 Location : 94 Injection Date : 6/6/2019 6:41:26 PM Inj: l Inj Volume : 5.000 µl Acq. Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M Last changed : 6/6/2019 6:30:12 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M (Sequence Method) Last changed : 6/10/2019 3:51:14 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Ref=360.100 (E:DATA(SX...-RAC-DIMERGENT 2019-06-06 18-23-02(SXS-8-88-RAC-DIMERGENT2.D) mAU -1200 -1000 -800 -600 -400 -200 -D Area Percent Report Sorted By Signal : : 1.00001.0000Multiplier Dilution Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area ÷ # [min] [mAU\*s] [mAU] 1 4.370 VB 0.1778 1.52220e4 1317.81311 100.0000 Totals : 1.52220e4 1317.81311 \*\*\* End of Report \*\*\*

1260 6/10/2019 3:51:53 PM SYSTEM



Data File E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\SXS-8-88-RAC-DIVERGENT1.D Sample Name: Ph-R-1-2-0PTICAL

Acq. Operator : SYSTEM Seq. Line : 2 Acq. Instrument : 1260 Location : 93 Injection Date : 6/6/2019 6:32:56 PM Inj: l Inj Volume : 5.000 µl Acq. Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M Last changed : 6/6/2019 6:30:12 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-88-RAC-DIVERGENT 2019-06-06 18-23-02\ASH-99-1-206NM-3UL-1 .OM.M (Sequence Method) Last changed : 6/10/2019 3:51:14 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=206.4 Ref=360.100 (E:DATA(SX...-RAC-DIMERGENT 2019-06-06 18-23-02(SXS-8-88-RAC-DIMERGENT1.D) mAU . 1200 -1000 -800 -600 -400 -200 -0. Area Percent Report Sorted By Signal : : Multiplier 1.0000 1.0000 Dilution Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] ÷ 1 4.561 BB 0.1720 1.49154e4 1317.80725 100.0000 Totals : 1.49154e4 1317.80725 \*\*\* End of Report \*\*\*

1260 6/10/2019 3:51:16 PM SYSTEM



## (2S\*,3R\*)-3m + (2R\*,3R\*)-4m

Data File E:\DATA\SX...S-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\SXS-7-102-RAC-DIVERGENT.D Sample Name: SXS-7-102-RAC

-----Acq. Operator : SYSTEM Seq. Line : 1 Location: 92 Inj: 1 Acq. Instrument : 1260 Injection Date : 6/7/2019 6:27:31 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-Acq. Method 206NM.M Last changed : 6/7/2019 6:45:40 PM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-206NM.M (Sequence Method) Last changed : 6/10/2019 3:38:36 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 B, Sig=206,4 Ref=360,100 (E:DATA\SX...-RAC-DIMERGENT 2019-06-07 18-26-02\SXS-7-102-RAC-DIMERGENT.D) mAU 1 1049.78 300 -250 -200 -150 -100 -50 D 12 4 10 14 16 6 \_\_\_\_\_ Area Percent Report Signal Sorted By : Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----|-----| 1 11.611 MF 0.3806 5134.66748 224.82292 19.7382 2 12.279 FM 0.3033 4761.70557 261.65707 18.3045 
 3
 12.468 FM
 0.4835 9067.67676
 312.54941
 34.8571

 4
 13.929 FM
 0.5439 7049.77979
 216.01880
 27.1001
 Totals : 2.60138e4 1015.04820 -----\*\*\* End of Report \*\*\*



## (2S,3S)-4m

Data File E:\DATA\SX...-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\SXS-7-102-RAC-DIVERGENT3.D Sample Name: 2-Nap-S-1-2-0PTICAL

-----Acq. Operator : SYSTEM Seq. Line : 4 Location : 95 Inj : 1 Acq. Instrument : 1260 Injection Date : 6/7/2019 7:30:02 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-Acq. Method 206NM.M Last changed : 6/7/2019 6:49:35 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-206NM.M (Sequence Method) Last changed : 6/10/2019 3:39:50 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DADI 8, Sig=206,4 Ref=360,100 (E:DATA:SX...RAC-DMERGENT 2019-06-07 18-26-02:SXS-7-102-RAC-DIVERGENT3.D) mAU = 1000 -800 -600 -400 200 Û 14 18 12 16 ŝ. 10 Area Percent Report \_\_\_\_\_ Sorted By Signal : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 e Width Area Height [min] [mAU\*s] [mAU] Peak RetTime Type Width Area \* # [min] 1 12.323 BB 0.4308 3.33712e4 1155.98645 100.0000 Totals : 3.33712e4 1155.98645 \*\*\* End of Report \*\*\*



(2R,3S)-**3m** 

Data File E:\DATA\SX...-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\SXS-7-102-RAC-DIVERGENT1.D Sample Name: 2-Nap-S-2-2-0PTICAL

-----Acq. Operator : SYSTEM Seq. Line : 2 Location : 93 Inj : 1 Acq. Instrument : 1260 Location : Injection Date : 6/7/2019 6:47:03 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-Acq. Method 206NM.M Last changed : 6/7/2019 6:49:35 PM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-206NM.M (Sequence Method) Last changed : 9/27/2019 7:39:08 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 8, Sig=206,4 Ref=360,100 (E:DATA\SX...RAC-DIVERGENT 2019-06-07 18-26-02\SXS-7-102-RAC-DIVERGENT1.D) mAU I 1200 -1000 -800 -600 -400 -200 -٥ 10 12 14 16 18 ------Area Percent Report Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* -----1 13.705 VB 0.4798 4.13981e4 1281.68091 100.0000 Totals : 4.13981e4 1281.68091 ------------\*\*\* End of Report \*\*\*

EtO<sub>2</sub>C CF<sub>3</sub> H<sub>2</sub>N

(2R,3R)-4m

Data File E:\DATA\SX...-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\SXS-7-102-RAC-DIVERGENT4.D Sample Name: 2-Nap-R-1-2-0PTICAL

-----Acq. Operator : SYSTEM Seq. Line : 5 Location : 96 Inj : 1 Acq. Instrument : 1260 Injection Date : 6/7/2019 7:51:29 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-Acq. Method 206NM.M Last changed : 6/7/2019 6:49:35 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-206NM.M (Sequence Method) Last changed : 6/10/2019 3:39:50 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DADI 8, Sig=206,4 Ref=360,100 (E:DATA:SX...RAC-DMERGENT 2019-06-07 18-26-02:SXS-7-102-RAC-DIVERGENT4.D) mAU 1400 -1200 -1000 -800 -600 -400 200 -0 18 12 14 16 ŝ. 10 Area Percent Report \_\_\_\_\_ Sorted By Signal : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 ype Width Area Height [min] [mAU\*s] [mAU] Peak RetTime Type Width Area \* # [min] 1 11.982 BB 0.4172 4.20702e4 1491.12988 100.0000 Totals : 4.20702e4 1491.12988 \*\*\* End of Report \*\*\*

EtO<sub>2</sub>C, CF<sub>3</sub> H<sub>2</sub>N

(2S,2R)-3m

Data File E:\DATA\SX...-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\SXS-7-102-RAC-DIVERGENT2.D Sample Name: 2-Nap-R-2-2-0PTICAL

-----Acq. Operator : SYSTEM Seq. Line : 3 cation: 94 Inj: 1 Acq. Instrument : 1260 Location : Injection Date : 6/7/2019 7:08:34 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-Acq. Method 206NM.M Last changed : 6/7/2019 6:49:35 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-7-102-RAC-DIVERGENT 2019-06-07 18-26-02\ID-99-1-3UL-0.5ML-206NM.M (Sequence Method) : 9/27/2019 7:40:38 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DADI 8, Sig=206,4 Re+360,100 (E:DATA:SX...RAC-DMERGENT 2019-06-07 18-26-02:SXS-7-102-RAC-DIVERGENT2.D) mAU 1200 1000 -800 -600 -400 -200 ٥ 12 18 á ŝ 10 14 18 min Area Percent Report \_\_\_\_\_ Sorted By Signal : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Height [mAU] Peak RetTime Type Width Area Area [min] [mAU\*s] \* # [min] 1 11.537 BB 0.4220 3.72918e4 1310.60364 100.0000 Totals : 3.72918e4 1310.60364 \*\*\* End of Report \*\*\*



## (2S\*,3R\*)-**3p** + (2R\*,3R\*)-**4p**

Data File E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT.D Sample Name: SXS-8-89-RAC-DIVERGENT

------Acq. Operator : SYSTEM Seq. Line : 1 Acq. Instrument : 1260 Location : 92 Injection Date : 6/8/2019 6:28:10 PM Inj : 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220**NM.M** Last changed : 6/8/2019 6:26:50 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220NM.M (Sequence Method) Last changed : 6/10/2019 3:44:37 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 B, Sig=206,4 Ref=360,100 (E:DATA\SX...9-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT.D) mAU -1400 -1200 -202 1000 -589 800 -626 600 -400 200 D \_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [mAU\*s] [mAU] \* 1 4.589 BV R 0.1109 6017.45068 776.55109 15.0179 2 5.918 BV 0.1463 1.53486e4 1540.30920 38.3059 3 6.626 VV 0.1619 6256.44971 575.01453 15.6144 4 7.202 VB 0.1935 1.24460e4 952.11890 31.0619 Totals : 4.00686e4 3843.99371 \_\_\_\_\_ \*\*\* End of Report \*\*\*

MeO<sub>2</sub>C<sub>2</sub>CF<sub>3</sub> H<sub>2</sub>N C

(2S,3S)-4p

Data File E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT3.D Sample Name: 2-C1-S-1-2-OPTICAL

-----Acq. Operator : SYSTEM Seq. Line : 4 Location : 95 Inj : 1 Acq. Instrument : 1260 Location : Injection Date : 6/8/2019 7:02:39 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-Acq. Method 220NM.M Last changed : 6/8/2019 6:26:50 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220NM.M (Sequence Method) Last changed : 6/10/2019 3:45:35 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 8, Sig=206,4 Ref=360,100 (E:DATA\SX...-RAC-DIMERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIMERGENT3.D) mAU 2000 1500 1000 -500 0 å Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Type Width Area Height [min] [mAU\*s] [mAU] Peak RetTime Type Width Area \* # [min] 1 4.591 BV R 0.1407 2.10075e4 2286.68018 100.0000 Totals : 2.10075e4 2286.68018 \*\*\* End of Report \*\*\*

MeO<sub>2</sub>C<sub>C</sub>F<sub>3</sub> H<sub>2</sub>N C

(2R,3S)-3p

Data File E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT4.D Sample Name: 2-C1-S-2-2-OPTICAL

-----Acq. Operator : SYSTEM Seq. Line : 5 ocation: 96 Inj: 1 Acq. Instrument : 1260 Location : Injection Date : 6/8/2019 7:14:06 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-Acq. Method 220NM.M Last changed : 6/8/2019 6:26:50 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220NM.M (Sequence Method) : 9/26/2019 11:57:15 AM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1 8, Sig=206,4 Ref=360,100 (E:DATA\SX...-RAC-DIMERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIMERGENT4.D) mAU <sup>°</sup> 2000 -1750 -1500 -1250 -1000 -750 -500 250 ٥ mir Area Percent Report \_\_\_\_\_ Sorted By Signal : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Height [mAU] Peak RetTime Type Width Area Area [min] [mAU\*s] \* # [min] 1 6.587 BV R 0.2100 2.90253e4 2154.02832 100.0000 2.90253e4 2154.02832 Totals : \*\*\* End of Report \*\*\*

MeO<sub>2</sub>C<sub>C</sub>F<sub>3</sub> H<sub>2</sub>N C

(2R,3R)-4p

Data File E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT1.D Sample Name: 2-C1-R-1-2-OPTICAL

-----Acq. Operator : SYSTEM Seq. Line : 2 Location : 93 Inj : 1 Acq. Instrument : 1260 Injection Date : 6/8/2019 6:39:38 PM Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-Acq. Method 220NM.M Last changed : 6/8/2019 6:26:50 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220NM.M (Sequence Method) Last changed : 6/10/2019 3:45:35 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 8, Sig=206,4 Ref=360,100 (E:DATA\SX...-RAC-DIMERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIMERGENT1.D) mAU 1750 -1500 -1250 -1000 -750 -500 250 Û. + Ŕ á Area Percent Report \_\_\_\_\_ Sorted By Signal : Multiplier 1.0000 : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Ype Width Area Height [min] [mAU\*s] [mAU] Peak RetTime Type Width Area \* # [min] 1 7.171 BB 0.2259 2.83564e4 1891.10242 100.0000 Totals : 2.83564e4 1891.10242 \*\*\* End of Report \*\*\*



Data File E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIVERGENT2.D Sample Name: 2-C1-R-2-2-OPTICAL

------Acq. Operator : SYSTEM Seq. Line : 3 Acq. Instrument : 1260 Location : 94 Injection Date : 6/8/2019 6:51:10 PM Inj : 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220**NM.M** Last changed : 6/8/2019 6:26:50 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-89-RAC-DIVERGENT 2019-06-08 18-26-49\IE-95-5-3UL-10MIN-220NM.M (Sequence Method) Last changed : 6/10/2019 3:45:35 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 8, Sig=206,4 Ref=360,100 (E:DATA\SX...-RAC-DIMERGENT 2019-06-08 18-26-49\SXS-8-89-RAC-DIMERGENT2.D) mAU \_1 2000 -1750 -1500 -1250 -1000 -750 -500 -250 ٥ ------Area Percent Report \_\_\_\_\_ Sorted By : Signal Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 B, Sig=206,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* ----|-----|----|-----|-----|-----| 1 5.902 BV R 0.1896 2.70806e4 2206.95117 100.0000 Totals : 2.70806e4 2206.95117 \_\_\_\_\_ \*\*\* End of Report \*\*\*

1260 6/10/2019 3:46:10 PM SYSTEM



(2S,3S)-6

Data File E:\DATA\SXS\SXS-8-71-1-2-OPTICAL 2019-05-13 19-37-46\SXS-8-71-1-2-OPTICAL.D Sample Name: SXS-8-67-1-2-RAC

Acq. Operator : SYSTEM Seq. Line : 1 Location: 81 Inj: 1 Acq. Instrument : 1260 Injection Date : 5/13/2019 7:39:12 PM Inj : Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-71-1-2-OPTICAL 2019-05-13 19-37-46\ADH-95-5-15MIN-209NM-1 Acq. Method .OML.M : 5/13/2019 7:57:15 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-8-71-1-2-0PTICAL 2019-05-13 19-37-46\ADH-95-5-15MIN-209NM-1 .OML.M (Sequence Method) : 9/21/2019 4:16:36 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated
DAD1 A. Sig=209.4 Re#360.100 (E:\DATAXSX...8-71-1-2-OPTICAL 2019-05-13 19-37-46\SXS.8-71-1-2-OPTICAL.D) mALI -250 2.305 200 -150 -100 -50 ٥ 14 16 10 12 18 Area Percent Report Sorted By : Signal Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=209,4 Ref=360,100 Peak RetTime Type Width Area Height Area [min] [mAU\*s] ÷ # [min] [mAU] 1 12.305 BB 0.5304 6253.90234 175.63522 35.0817 2 14.779 BB 0.6029 1.15728e4 278.42004 64.9183 Totals : 1.78267e4 454.05527 \*\*\* End of Report \*\*\*

1260 9/21/2019 4:16:39 PM SYSTEM

Data File E:\DATA\SXS\SXS-8-71-1-2-OPTICAL 2019-05-13 19-37-46\SXS-8-71-1-2-OPTICAL1.D Sample Name: SXS-8-71-1-2-OPTICAL

------Seq. Line : 2 Location : 82 Acq. Operator : SYSTEM Acq. Instrument : 1260 Injection Date : 5/13/2019 8:00:34 PM Inj: l Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-8-71-1-2-OPTICAL 2019-05-13 19-37-46\ADH-95-5-15MIN-209NM-1 .OML.M Last changed : 5/13/2019 8:16:16 PM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-8-71-1-2-0PTICAL 2019-05-13 19-37-46\ADH-95-5-15MIN-209NM-1 .OML.M (Sequence Method) Last changed : 9/21/2019 4:16:36 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A Sig=209.4 Ref=360,100 (E:DATA\SX...8-71-1-2-OPTICAL 2019-05-13 19-37-46\SXS-8-71-1-2-OPTICAL1.D) mAU 1 600 -500 -400 -300 -200 -100 -14.799 D 14 10 12 Area Percent Report -----Sorted By Signal : : Multiplier 1.0000 1.0000 Dilution Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=209,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] [mAU] \* 1 12.254 BB 0.5222 2.45543e4 691.60022 99.3029 2 14.799 BB 0.3973 172.36891 5.13915 0.6971 2.47267e4 696.73937 Totals : \_\_\_\_\_ \*\*\* End of Report \*\*\* Page 1 of 1 1260 9/21/2019 4:19:06 PM SYSTEM



(2R,3S)-6

Data File E:\DATA\SXS\SXS-8-68-2-2-RAC 2019-05-16 15-39-46\SXS-8-68-2-2-RAC1.D Sample Name: SXS-8-68-2-2-RAC

Acq. Operator : SYSTEM Seq. Line : 2 Location: 96 Inj: 1 Acq. Instrument : 1260 Injection Date : 5/16/2019 3:58:44 PM Inj : Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-68-2-2-RAC 2019-05-16 15-39-46\0JH-95-5-10MIN-210NM-2UL.M Acq. Method : 5/16/2019 4:16:42 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-8-68-2-2-RAC 2019-05-16 15-39-46\0JH-95-5-10MIN-210NM-2UL.M (Sequence Method) : 9/21/2019 4:23:16 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DADI A. Sig=210.4 Re=5360.100 (E:/DATA%SXS%SXS-8-68-2-2-RAC 2019-05-16 15-38-46\SXS-8-68-2-2-RAC1.D) mAU -300 -250 -8 200 ø 150 -100 -50 -D 14 16 10 12 mir \_\_\_\_\_ Area Percent Report ------Sorted By : Signal Multiplier 1.0000 : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Àrea Area # [min] [min] [mAU\*s] [mAU] \* - | - - - - - - - | 1 14.767 BV 0.5657 1.18371e4 316.93280 64.3781 2 16.133 VB 0.5709 6549.71338 174.50444 35.6219 Totals : 1.83868e4 491.43724 \*\*\* End of Report \*\*\*

1260 9/21/2019 4:23:19 PM SYSTEM

Data File E:\DATA\SXS\SXS-8-68-2-2-RAC 2019-05-16 15-39-46\SXS-8-68-2-2-RAC2.D Sample Name: SXS-8-71-2-2-0PTICAL

Acq. Operator : SYSTEM Seq. Line : 3 Location : 97 Acq. Instrument : 1260 Injection Date : 5/16/2019 4:18:12 PM Inj: l Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-68-2-2-RAC 2019-05-16 15-39-46\0JH-95-5-10MIN-210NM-2UL.M Acg. Method Last changed : 5/16/2019 4:18:29 PM by SYSTEM (modified after loading) Analysis Method : E:\DATA\SXS\SXS-8-68-2-2-RAC 2019-05-16 15-39-46\0JH-95-5-10MIN-210NM-2UL.M (Sequence Method) : 9/21/2019 4:23:30 PM by SYSTEM Last changed (modified after loading) Additional Info : Peak(s) manually integrated DAD1 A, Sig=210,4 Ref=360,100 (E:/DATA\SXS\SXS-8-68-2-2-RAC 2019-05-16 15-39-46\SXS-8-68-2-2-RAC2.D) 18300 B mAU -400 -300 -200 -100 (grind 14920 Û 18 12 14 10 16 mir Area Percent Report Signal Sorted By : 1.0000 Multiplier : Dilution : 1.0000 Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=210,4 Ref=360,100 Peak RetTime Type Width Height Area Area # [min] [min] [mAU\*s] [mAU] \* 1 14.920 FM 0.5230 88.51195 2.82043 0.4813 2 16.187 FM 0.6327 1.83008e4 482.06165 99.5187 Totals : 1.83893e4 484.88208 \*\*\* End of Report \*\*\* Page 1 of 1 1260 9/21/2019 4:23:31 PM SYSTEM

S172



Data File E:\DATA\SXS\SXS-8-72-1-2-OPTICAL 2019-05-16 14-52-00\SXS-8-72-1-2-OPTICAL.D Sample Name: SXS-8-70-1-2-RAC

Acq. Operator : SYSTEM Seq. Line : 1 Location: 92 Inj: 1 Acq. Instrument : 1260 Injection Date : 5/16/2019 2:53:29 PM Inj : Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-72-1-2-OPTICAL 2019-05-16 14-52-00\0JH-95-5-10MIN-210NM-Acq. Method 2UL.M : 5/16/2019 3:01:25 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-8-72-1-2-0PTICAL 2019-05-16 14-52-00\0JH-95-5-10MIN-210NM-2UL.M (Sequence Method) Last changed : 9/21/2019 4:27:07 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated
DAD1 A. Sig=210.4 Re=5360.100 (E:\DATAXSX...8-72-1-2-OPTICAL 2019-05-16 14-52-00\SXS-8-72-1-2-OPTICAL.D) mAU 1 300 -250 -200 -150 -100 -50 -D Area Percent Report Sorted By Signal : Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area [min] [mAU\*s] \* # [min] [mAU] ---|----|---1 6.408 BV 0.1680 3442.43286 311.44089 63.2353 2 6.820 VB 0.1979 2001.40967 150.78511 36.7647 Totals : 5443.84253 462.22600 \*\*\* End of Report \*\*\*

1260 9/21/2019 4:27:10 PM SYSTEM

Data File E:\DATA\SXS\SXS-8-72-1-2-OPTICAL 2019-05-16 14-52-00\SXS-8-72-1-2-OPTICAL1.D Sample Name: SXS-8-72-1-2-OPTICAL

------Seq. Line : 2 Location : 93 Acq. Operator : SYSTEM Acq. Instrument : 1260 Injection Date : 5/16/2019 3:03:25 PM Inj: l Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-8-72-1-2-OPTICAL 2019-05-16 14-52-00\0JH-95-5-10MIN-210NM-2UL.M Last changed : 5/16/2019 3:01:25 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-72-1-2-OPTICAL 2019-05-16 14-52-00\0JH-95-5-10MIN-210NM-2UL.M (Sequence Method) Last changed : 9/21/2019 4:27:25 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated
DAD1 A, Sig=210.4 Ref=360,100 (E-DATA\SX...8-72-1-2-OPTICAL 2019-05-16 14-52-00\SXS-8-72-1-2-OPTICAL1.D) mAU 1 1400 -1200 -1000 -800 -600 -400 -200 -<del>8</del> ٥ ------Area Percent Report Signal Sorted By : Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* 1 6.496 BV E 0.1575 151.13089 14.74959 0.7427 2 6.897 VB R 0.2055 2.01978e4 1495.71680 99.2573 Totals : 2.03489e4 1510.46638 \*\*\* End of Report \*\*\* Page 1 of 1

1260 9/21/2019 4:27:27 PM SYSTEM



Data File E:\DATA\SXS\SXS-8-73-2-2-OPTICAL 2019-05-16 15-14-30\SXS-8-73-2-2-OPTICAL.D Sample Name: SXS-8-69-2-2-RAC

Acq. Operator : SYSTEM Seq. Line : 1 Location: 94 Inj: 1 Acq. Instrument : 1260 Injection Date : 5/16/2019 3:16:01 PM Inj : Inj Volume : 3.000 µl : E:\DATA\SXS\SXS-8-73-2-2-OPTICAL 2019-05-16 15-14-30\0JH-95-5-10MIN-210NM-Acq. Method 2UL.M : 5/16/2019 3:23:22 PM by SYSTEM Last changed (modified after loading) Analysis Method : E:\DATA\SXS\SXS-8-73-2-2-0PTICAL 2019-05-16 15-14-30\0JH-95-5-10MIN-210NM-2UL.M (Sequence Method) Last changed : 9/21/2019 4:30:04 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated
DADIA. Sig=210.4 Re#360.100 (E:DATAXSX...8-73-2-2-OFTICAL 2019-05-16 15-14-30/SXS-8-73-2-2-OFTICAL.D) mALI 800 -600 -400 -200 n Area Percent Report Sorted By Signal : Multiplier : 1.0000 Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [min] [mAU\*s] \* [mAU] 1 6.021 BV 0.1476 9264.98145 951.78058 63.9449 2 6.382 VB 0.1822 5224.02246 428.48306 36.0551 Totals : 1.44890e4 1380.26364 \*\*\* End of Report \*\*\*

1260 9/21/2019 4:30:06 PM SYSTEM

Data File E:\DATA\SXS\SXS-8-73-2-2-OPTICAL 2019-05-16 15-14-30\SXS-8-73-2-2-OPTICAL1.D Sample Name: SXS-8-73-2-2-OPTICAL

------Seq. Line : 2 Location : 95 Acq. Operator : SYSTEM Acq. Instrument : 1260 Injection Date : 5/16/2019 3:25:59 PM Inj: 1 Inj Volume : 3.000 µl Acq. Method : E:\DATA\SXS\SXS-8-73-2-2-OPTICAL 2019-05-16 15-14-30\0JH-95-5-10MIN-210NM-2UL.M Last changed : 5/16/2019 3:23:22 PM by SYSTEM Analysis Method : E:\DATA\SXS\SXS-8-73-2-2-OPTICAL 2019-05-16 15-14-30\0JH-95-5-10MIN-210NM-2UL.M (Sequence Method) Last changed : 9/21/2019 4:30:04 PM by SYSTEM (modified after loading) Additional Info : Peak(s) manually integrated
DAD1 A, Sig=210.4 Ref=360,100 (E-DATA\SX...8-73-2-2-OPTICAL 2019-05-16 15-14-30\SXS-8-73-2-2-OPTICAL1.D) mAU 4 800 -700 -600 -500 -400 -300 -200 -100 -092 ٥. ------Area Percent Report Signal Sorted By . Multiplier : Dilution 1.0000 : Do not use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=210,4 Ref=360,100 Peak RetTime Type Width Area Height Area # [min] [mAU\*s] [mAU] \* 1 6.092 BV E 0.1406 51.21827 5.65977 0.4969 2 6.454 VB R 0.1852 1.02568e4 835.67590 99.5031 Totals : 1.03080e4 841.33567 \*\*\* End of Report \*\*\*

1260 9/21/2019 4:30:15 PM SYSTEM