## Supplementary Information-2

# Copies of ${ }^{\mathbf{1}} \mathbf{H}$-NMR, ${ }^{13} \mathbf{C}$-NMR spectra and chiral HPLC chromatograms 

Stereoselective Synthesis of 4-Substituted-Cyclic Sulfamidate-5Carboxylates By Asymmetric Transfer Hydrogenation Accompanying Dynamic Kinetic Resolution and Its Use in Concise Stereoselective Synthesis of (-)-epi-Cytoxazone and Taxotere Side-Chain.

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## ${ }^{1} \mathrm{H}$-NMR and ${ }^{13} \mathrm{C}$-NMR spectra


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$<\quad 158.5515$

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$-60.4822$
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- 21.0158


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-3.9660
-3.7276


| 7.8738 |
| ---: |
| -7.8600 |
| $=$7.4582 <br> -7.4431 <br> -7.4023 <br> -7.3872 <br> 7.3721 |
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$-193.8153$

- 169.0981
$-53.0466$


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 7.2583



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$-171.4620$
$-163.7952$

- 148.2172
$<_{130.4883}^{130.4999}$
-123.7567


| 8.0604 |
| ---: |
| -7.9779 |
| -7.9624 |
| -7.6204 |
| $\quad 7.4748$ |





(IכOכ'zHWOOS)YWN HI
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8.0770

-8.0601 | 7.2479 |
| :--- |
| $=6.9752$ |
| $=6.9583$ |





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8.2348
$<\quad 8.1229$
$-7.1346$
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$-196.4880$
$-196.4880$
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167.3251
$\mathbf{1} \begin{array}{r}167.2653 \\ 165.2758\end{array}$
$-165.2758$

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| $<$ |
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| 7.8278 |
| 7.8118 |



$\square$
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-4.0925
-3.9840
-3.98
-3.7602



$-192.9496$
$-168.6754$

| —36.2366 |
| :--- |
| $=132.5915$ |
| 129.8500 |







 ${ }^{13} \mathrm{CNMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right)$ 136.3516
$/ \begin{array}{r}135.0399 \\ 134.9676 \\ 134.6841 \\ 130.1221 \\ 130.0596 \\ 129.9641 \\ 129.8562 \\ 129.7789 \\ 129.6941 \\ 129.3367\end{array}$

$\begin{array}{r}53.5935 \\ -53.1561 \\ \hline\end{array}$



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8Lレレ-HO-4deu- $\forall \Gamma$ Y








$-181.8468$
$-169.1197$
-149.7677
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$\mathrm{HO}^{-} \mathrm{n}^{-}$9Z9021 ${ }^{-}$入SY

- 53.3471



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$-40.5937$
-16.9981
-13.5645





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- 27.4910
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$\qquad$
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KSY 120508 3Me_ $-6.1730$



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-55.5696
-53.5128 $\left[\begin{array}{r}29.4033 \\ 29.2493 \\ 29.0954 \\ 28.9415 \\ 28.7875 \\ 28.6335 \\ 28.4792\end{array}\right.$





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-169.3633
-165.8990
-163.5325
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$<{ }_{122.7635}^{122.8053}$
$-117.4815$
${ }^{117.1840}$


| 8.2378 |
| ---: |
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| $\mathbf{7 . 8 5 6 3}$ |


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- 170.5461
$-153.0176$
$\left.\begin{array}{r}137.4866 \\ \text { 137.2209 } \\ 136.9561 \\ 130.7286 \\ 129.4449 \\ \hline 126.5680 \\ 126.5412 \\ 126.5140 \\ 126.4824 \\ 124.0640 \\ 121.8922\end{array}\right)$
$-54.6042$



8.2082
-8.1925
-7.8806
-7.8650




8.2135
-8.1974
-8.1442
$-6.2268$
-3.9717
$-\quad 3.8066$










T - 163.8833

$-54.5317$
$-0.1343$




$-5.9735$


-163.4275
-159.7571
-151.1044
-143.2709 - 124.8902
- 114.5852






$-179.8823$
$-18.9401$
- 13.5539
$-0.1308$




[3.1319
3.1075
3.0921
-3.0774
3.0721
$\mathbf{3} .0774$
-3.0705
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$\left[\begin{array}{r}3.0614 \\ -3.0495 \\ 3.9732\end{array}\right.$
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$-2.9732$




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$=-52.9023$
- 38.0348
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－ 52.8438
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-7.9043
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$-14.1192$
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$-\quad 53.0069$






$-166.8911$

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ع180-כา d-oogn-4d-s's-rגS
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5.4953
5.4558
5.4432
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$\qquad$ 133.4484
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-128.8305
127.3470





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$-147.7023$

- 62.7622
$-52.7990$
$-27.8238$
$-0.0026$



5.4731
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5.4432
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4.7614
4.7405
4.7196

0.9470
0.9261
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- 162.2545


| 133.6820 |
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| 129.7197 |
| 128.8738 |
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/ $/ \begin{aligned} & 1.6656 \\ & 1.6489\end{aligned}$
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7 $\begin{array}{r}1.5543 \\ -1.5047 \\ -1.4314\end{array}$
1. $\left[\begin{array}{l}-1.4064 \\ -1.3804 \\ 1.3547\end{array}\right.$
 $\qquad$
$-148.4483$

 85.9833
$-59.1924$

- 53.2532
31.8956
$=29.7050$
-27.8765
-18.1601
-13.9067

 $\square$
$-163.9262$







 $-168.2904$ -154.9584

154.9584
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- 31.5997
- 22.6659
- 14.1383



$\begin{array}{r}-52.4724 \\ \hline\end{array}$
$-34.3789$
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- 19.2255
- 13.7186

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- 55.9821
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$-0.0040$







$<{ }_{159.9671}^{160.1476}$
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## Chiral HPLC Chromatograms

Chiral HPLC Chromatograms of ATH-DKR products
Sample name: $(S, S)$-7a

- Analysis condition: Chiralpak IB, $20 \% \mathrm{EtOH} / \mathrm{n}$-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 10.7000 | 550.3260 |  | FF | 40.0000 |
| 2 | 12.8667 | 36285.7819 |  |  | 1.4940 |
| 합계 |  | 36836.1094 |  | 114.0000 | 98.5060 |

-Sample name: $(R, R)-7 \mathbf{a}$

- Analysis condition: Chiralpak IB, 20\% EtOH/n-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.8500 | 22019.3878 | FF | 109.0000 | 99.0959 |
| 2 | 13.7000 | 200.8943 | FF | 42.0000 | 0.9041 |
| 합계 |  | 22220.2832 |  |  |  |

$e e=98 \%$
-Sample name: $(S, S)$ - $N$-Boc-7a
Analysis condition: Chiralpak AD-H, $10 \% \mathrm{iPrOH} / \mathrm{n}-h e x a n e, 1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- ResultReport

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.2333 | 347.1643 | FF | 32.0000 | 1.0986 |
| 2 | 13.4167 | 31252.2745 | BB | 96.0000 | 98.9014 |
| Total |  | 31599.4395 |  |  |  |

[^0]Sample name: $(R, R)$ - $N$-Boc-7a
Analysis condition: Chiralpak AD-H, $10 \% \mathrm{iPrOH} / \mathrm{n}$-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.2833 | 2362.1962 | BB | 62.0000 | 98.8747 |
| 2 | 13.8333 | 26.8841 | FF | 43.0000 | 1.1253 |
| Total |  | 2389.0803 |  |  |  |

-Sample name: $(S, S)$ - $N$-Boc-7b
Analysis condition: Chiralpak AD-H, $10 \% \mathrm{iPrOH} / \mathrm{n}$-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$



- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 9.7000 | 351.3224 | BB | 43.0000 | 1.0484 |
| 2 | 14.2833 | 32805.7090 | BB | 118.0000 | 97.8995 |
| Total |  | 33510.9453 |  |  |  |

-Sample name: $(S, S)$ - $N$-Boc-7c
Analysis condition: Chiralpak AD-H, $10 \% \mathrm{iPrOH} / \mathrm{n}$-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 18.5667 | 387.5644 | BB | 68.0000 | 1.0268 |
| 2 | 22.1667 | 37040.3066 | BB | 168.0000 | 98.1286 |
| Total |  | 37746.6836 |  |  |  |

ee=97.9\%

- Sample name: $(S, S)$-7d
- Analysis condition: Chiralpak AD-H, 5\% iPrOH/n-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 32.2667 | 28.7343 | BB | 42.0000 | 0.0730 |
| 2 | 36.2500 | 39153.5218 | BB | 208.0000 | 99.4094 |
| Total |  | 39182.2561 |  |  |  |

ee $=>99 \%$

- Sample name: ( $(, S)$-7e

Analysis condition: Chiralpak AD-H, 20\% $\mathrm{iPrOH} / \mathrm{n}$-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 8.4333 | 15569.3232 | BB | 63.0000 | 96.0666 |
| 2 | 10.1000 | 637.4800 | FF | 40.0000 | 3.9334 |
| Total |  | 16206.8037 |  |  |  |

- Sample name: ( $(, S)$-7f
- Analysis condition: Chiralpak AD-H, $10 \% \mathrm{iPrOH} / \mathrm{n}$-hexane, $1.3 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$



## - Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 15.9667 | 196.3357 | FF | 43.0000 | 0.6009 |
| 2 | 17.0000 | 32476.0723 | FF | 93.0000 | 99.3991 |
| Total |  | 32672.4082 |  |  |  |

- Sample name: $(S, S) \mathbf{- 7} \mathbf{g}$
- Analysis condition: Chiralpak AD-H, 20\% iPrOH/n-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- ResultReport

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 10.1167 | 4504.8323 | FF | 67.0000 | 99.5876 |
| 2 | 12.4833 | 18.6563 | FF | 27.0000 | 0.4124 |
| Total |  | 4523.4888 |  |  |  |

- Sample name: $(S, S)$-7h
- Analysis condition: Chiralpak AD-H, $10 \% \mathrm{iPrOH} / \mathrm{n}$-hexane, $1.2 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 13.8333 | 156.0047 | FF | 40.0000 | 1.6030 |
| 2 | 15.0333 | 9576.3127 | FF | 80.0000 | 98.3970 |
| Total |  | 9732.3174 |  |  |  |

ee=96.7\%

- Sample name: ( $S, S$ )-7i
- Analysis condition: Chiralpak AD-H, 10\% EtOH/n-hexane, $1.5 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.2667 | 110.8649 | FF | 33.0000 | 1.3545 |
| 2 | 14.0667 | 8073.8913 | FF | 85.0000 | 98.6455 |
| Total |  | 8184.7563 |  |  |  |

ee=97.3\%

- Sample name: $(S, S)-7 \mathbf{j}$
- Analysis condition: Chiralpak AD-H, $20 \% \mathrm{iPrOH} / \mathrm{n}$-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- Result Report
$\left.\begin{array}{|c|c|c|c|c|c|}\hline \text { Peak \# } & \text { Time[min] } & \text { Area[mV*s] } & \text { BL } & \text { wide[sec] } & \text { Area\% } \\ \hline 1 & 14.0000 & 2420.6589 & & \text { FF } & 72.0000\end{array}\right] 100.0000$
- Sample name: $(S, S)-7 \mathbf{k}$
- Analysis condition: Chiralpak IA, 20\% EtOH/n-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- ResultReport

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 12.5333 | 402.7184 | FF | 41.0000 | 1.3427 |
| 2 | 16.1500 | 29590.0409 | FF | 100.0000 | 98.6573 |
| Total |  | 29992.7598 |  |  |  |

ee=97.3\%

- Sample name: ( $S, S$ )-71
- Analysis condition: Chiralpak IA, 20\% EtOH/n-hexane, $1.5 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 6.5000 | 16.5963 | FF | 24.0000 | 0.2520 |
| 2 | 10.8333 | 6569.2955 | FF | 74.0000 | 99.7480 |
| Total |  | 6585.8921 |  |  |  |

-Sample name: ( $(S, S)$-7m

- Analysis condition: Chiralpak IA, 30\% EtOH/n-hexane, $1.3 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.2167 | 351.4940 | FF | 46.0000 | 1.8410 |
| 2 | 13.7667 | 18740.6826 | FF | 81.0000 | 98.1590 |
| Total |  | 19092.1758 |  |  |  |

ee=96.3\%
-Sample name: ( $(S, S)$-7n

- Analysis condition: Chiralpak IA, 20\% EtOH/n-hexane, $1.5 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 12.0000 | 129.7953 | FF | 41.0000 | 1.6647 |
| 2 | 13.9833 | 7666.9291 | BB | 97.0000 | 98.3353 |
| Total |  | 7796.7246 |  |  |  |

ee=96.7\%

- Sample name: $(S, S)$-7o
- Analysis condition: Chiralpak AD-H, 20\% iprOH/n-hexane, $1.0 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- ResultReport

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 11.5333 | 24396.7379 | FF | 69.0000 | 98.3557 |
| 2 | 13.7333 | 407.8611 | FF | 41.0000 | 1.6443 |
| Total |  | 24804.5996 |  |  |  |

ee=96.7\%
-Sample name: ( $(S, S$ ) -7p

- Analysis condition: Chiralpak IA, 20\% EtOH/n-hexane, $1.5 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 8.6833 | 1230.3090 | FF | 60.0000 | 97.4679 |
| 2 | 14.3333 | 31.9625 | FF | 48.0000 | 2.5321 |
| Total |  | 1262.2714 |  |  |  |

-Sample name: $(R, R)-\mathbf{7 p}$

- Analysis condition: Chiralpak IA, 20\% EtOH/n-hexane, $1.5 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$

- ResultReport

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 8.6167 | 213.4351 | FF | 39.0000 | 2.3101 |
| 2 | 14.2833 | 9025.6053 | BB | 99.0000 | 97.6899 |
| Total |  | 9239.0400 |  |  |  |

- Sample name: ( $S, S$ )-7q
- Analysis condition: Chiralpak IA, 20\% EtOH/n-hexane, $1.5 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 12.5000 | 47.0973 | FF | 36.0000 | 0.6703 |
| 2 | 14.2167 | 6979.5146 |  | FF | 100.0000 |

-Sample name: ( $S, S$ )-7s

- Analysis condition: Chiralpak IA, 20\% EtOH/n-hexane, $1.5 \mathrm{ml} / \mathrm{min}, 215 \mathrm{~nm}$


- Result Report

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 6.0000 | 302.5704 | FF | 25.0000 | 11.9138 |
| 2 | 7.9500 | 2237.1001 | FF | 43.0000 | 88.0862 |
| Total |  | 2539.6707 |  |  |  |

$e e=76.1 \%$

- Sample name: $(2 R, 3 S)-8 \mathbf{r}$
- Analysis condition: Chiralpak IC, $10 \%$ iPrOH/n-hexane, $0.7 \mathrm{ml} / \mathrm{min}$, 254nm



| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 9.2667 | 185.5496 | BB | 38.0000 | 4.3399 |
| 2 | 11.1500 | 4089.8949 | BB | 28.0000 | 95.6601 |
| Total |  | 4275.4443 |  |  |  |

$e e=91.3 \%$

- Sample name: $(2 R, 3 S)-8 \mathbf{t}$

Analysis condition: Chiralpak IC, $10 \%$ iPrOH/n-hexane, $0.7 \mathrm{ml} / \mathrm{min}$, 254 nm

| Peak \# | Time[min] | Area[mV*s] | BL | wide[sec] | Area\% |
| :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 8.3500 | 832.8883 | BB | 46.0000 | 26.2840 |
| 2 | 9.5500 | 2335.9153 | BB | 68.0000 | 73.7160 |
| Total |  | 3168.8035 |  |  |  |

ee=47.4\%


[^0]:    ee=98\%

