Supplementary Information-2

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

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

Jin-ah Kim,$^a$ Yeon Ji Seo,$^{a,b}$ Soyeong Kang,$^{a,b}$ Juae Han$^{a,b}$ and Hyeon-Kyu Lee$^{a,b,*}$

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e-mail: leehk@kRICT.re.kr
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$^1$H-NMR and $^{13}$C-NMR spectra
H NMR (500 MHz, CDCl3)

\[
\begin{align*}
\text{OH} & \quad \text{O} \\
\text{O} & \quad \text{O} \\
\text{O} & \quad \text{O} \\
\text{O} & \quad \text{O}
\end{align*}
\]
$^1$H NMR (500 MHz, CDCl$_3$)

$\text{KSY prop OH}$
HIAJπimine
The image contains a spectrum graph with chemical shift values ranging from -0.1 to 10 ppm. There is a molecule structure labeled as 61 with chemical formulas and annotations indicating the presence of S, N, O, and other elements. The spectrum appears to be for a compound labeled LHK 120305 ACI dimine in DCM. The text on the page includes technical details and notations related to the experiment and measurement conditions.
$^1H$ NMR (500 MHz, CDCl$_3$)
1H NMR (500MHz, CDCl3)

6k

LHK-4 F Imine
69
1H NMR (500 MHz, CDCl3)

KIA-4-Ome-car
1H NMR (500MHz, CDCl3)
N-Boc-(5S,5R)-71

\[
\text{H NMR (500 MHz, CDCl}_3)\n\]

LR-158-Boc
\[(2R,3S)-8a\]

\[
\begin{align*}
\text{OCH} & \\
\text{OCH} & \\
\text{OCH} & \\
\text{OCH} & \\
\end{align*}
\]

H NMR (500 MHz, CDCl₃)
(2R,3S)-8R

Boehn

\text{NMR}(500\text{MHz}, \text{CDCl}_3)
(2R,3S)-9a

Benzo C12

3J NMR(500MHz, CDCl3)

Kra ph carbonate Boc OH 0401
$^{1}H$ NMR (500 MHz, CDCl₃)
$^{1}H$ NMR (500MHz, CDCl$_3$)

$^{2}A_{35.8}$
$^{1}H$ NMR (500 MHz, CDCl$_3$)

$\text{KIA-4-O-Me-Carbop-N-BOC-OH}$

$\text{(2R,3S)-9}$
Chiral HPLC Chromatograms
Chiral HPLC Chromatograms of ATH-DKR products

- **Sample name:** (S,S)-7a

- **Analysis condition:** Chiralpak IB, 20% EtOH/n-hexane, 1.0 ml/min, 215nm

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ee=98%
Sample name: (S,S)-N-Boc-7a

Analysis condition: Chiralpak AD-H, 10% iPrOH/n-hexane, 1.0 ml/min, 215nm

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ee=98%
Sample name: \((R,R)\)-N-Boc-7a

Analysis condition: Chiralpak AD-H, 10% iPrOH/n-hexane, 1.0 ml/min, 215nm

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\[ \text{ee} = 98\% \]
Sample name: (S,S)-N-Boc-7b

Analysis condition: Chiralpak AD-H, 10% iPrOH/n-hexane, 1.0 ml/min, 215nm

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\[ \text{ee} = 97.2\% \]
Sample name: (S,S)-N-Boc-7c

Analysis condition: Chiralpak AD-H, 10% iPrOH/n-hexane, 1.0 ml/min, 215nm

\[
\begin{array}{c}
\text{Racemic-}N\text{-Boc-7c} \\
\end{array}
\]

\[
\begin{array}{c}
\text{N-Boc- (S,S)-7c} \\
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Result Report

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\[ee=97.9\%\]
Sample name: (S,S)-7d
Analysis condition: Chiralpak AD-H, 5% iPrOH/n-hexane, 1.0 ml/min, 215nm

Racemic-7d

(S,S)-7d

Result Report

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ee => 99%
Sample name: \((S,S)-7e\)

Analysis condition: Chiralpak AD-H, 20% iPrOH/n-hexane, 1.0 ml/min, 215nm

**Result Report**

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\(\text{ee}=92.1\%\)
Sample name: (S,S)-7f

Analysis condition: Chiralpak AD-H, 10% iPrOH/n-hexane, 1.3 ml/min, 215 nm

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ee=98.7%
**Sample name:** (S,S) -7g

**Analysis condition:** Chiralpak AD-H, 20% iPrOH/n-hexane, 1.0 ml/min, 215nm

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**Result Report**
Sample name: \((S,S)\)-7h

Analysis condition: Chiralpak AD-H, 10% iPrOH/n-hexane, 1.2 ml/min, 215nm

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\[
\text{ee}=96.7\%
\]
Sample name: (S,S)-7i

Analysis condition: Chiralpak AD-H, 10% EtOH/n-hexane, 1.5 ml/min, 215nm

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$ee = 97.3\%$
- **Sample name:** (S,S)-7j

- **Analysis condition:** Chiralpak AD-H, 20% iPrOH/n-hexane, 1.0 ml/min, 215nm

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\[ ee = >99\% \]
Sample name: \((S,S)-7k\)

Analysis condition: Chiralpak IA, 20% EtOH/n-hexane, 1.0 ml/min, 215nm

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\(ee=97.3\%\)
Sample name: (S,S)-7l

Analysis condition: Chiralpak IA, 20% EtOH/n-hexane, 1.5 ml/min, 215nm

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Sample name: (S,S)-7m

Analysis condition: Chiralpak IA, 30% EtOH/n-hexane, 1.3 ml/min, 215nm

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<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.3833</td>
<td>50.0070</td>
</tr>
<tr>
<td>2</td>
<td>14.7167</td>
<td>49.9930</td>
</tr>
</tbody>
</table>

Result Report

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Time[min]</th>
<th>Area[mV·s]</th>
<th>BL</th>
<th>width[sec]</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.2187</td>
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<td>FF</td>
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<td>1.8410</td>
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<td>81.0000</td>
<td>98.1590</td>
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<td>Total</td>
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<td>19092.1758</td>
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</tbody>
</table>

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

Analysis condition: Chiralpak IA, 20% EtOH/n-hexane, 1.5 ml/min, 215nm

Result Report

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Time [min]</th>
<th>Area [mV*sec]</th>
<th>BL</th>
<th>Width [sec]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12.0000</td>
<td>129.7953</td>
<td>FF</td>
<td>41.0000</td>
<td>1.8847</td>
</tr>
<tr>
<td>2</td>
<td>13.9833</td>
<td>7888.9291</td>
<td>EB</td>
<td>97.0000</td>
<td>98.3353</td>
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<td>Total</td>
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<td>7796.7246</td>
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</tbody>
</table>

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

Analysis condition: Chiralpak AD-H, 20% iprOH/n-hexane, 1.0 ml/min, 215nm

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Time [min]</th>
<th>Area [mV*s]</th>
<th>BL</th>
<th>width [sec]</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
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<td>11.1500</td>
<td>49.9947</td>
<td>FF</td>
<td>69.0000</td>
<td>96.3557</td>
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<tr>
<td>2</td>
<td>13.0000</td>
<td>50.0053</td>
<td>FF</td>
<td>41.0000</td>
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<td>Total</td>
<td></td>
<td>24804.5996</td>
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</tbody>
</table>

ee=96.7%
Sample name: (S,S)-7p
Analysis condition: Chiralpak IA, 20% EtOH/n-hexane, 1.5 ml/min, 215nm

<table>
<thead>
<tr>
<th></th>
<th>N</th>
<th>RT[min]</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.7167</td>
<td>50.9298</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>14.4500</td>
<td>49.0702</td>
<td></td>
</tr>
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Result Report

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Time[min]</th>
<th>Area[mV*s]</th>
<th>BL</th>
<th>width[sec]</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.6833</td>
<td>1230.3090</td>
<td>RF</td>
<td>60.0000</td>
<td>97.4679</td>
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<td>31.9625</td>
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</tbody>
</table>

ee=94.9%
Sample name: (R,R)-7p

Analysis condition: Chiralpak IA, 20% EtOH/n-hexane, 1.5 ml/min, 215nm

<table>
<thead>
<tr>
<th>N</th>
<th>RT[min]</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>50.9298</td>
</tr>
<tr>
<td>2</td>
<td>14.4500</td>
<td>49.0702</td>
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</tbody>
</table>

Result Report

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Time[min]</th>
<th>Area[mV*s]</th>
<th>BL</th>
<th>width[sec]</th>
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</tr>
</thead>
<tbody>
<tr>
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</table>

ee=95.3%
Sample name: \((S,S)-7q\)

Analysis condition: Chiralpak IA, 20% EtOH/n-hexane, 1.5 ml/min, 215nm

<table>
<thead>
<tr>
<th>N</th>
<th>RT [min]</th>
<th>Area %</th>
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<tbody>
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<td>49.1765</td>
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</table>

Result Report

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Time [min]</th>
<th>Area [mV*s]</th>
<th>BL</th>
<th>Width [sec]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
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<td>FF</td>
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<td>0.8703</td>
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<tr>
<td>2</td>
<td>14.2167</td>
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</tbody>
</table>

\(\text{ee}=98.7\%\)
Sample name: (S,S)-7s

Analysis condition: Chiralpak IA, 20% EtOH/n-hexane, 1.5 ml/min, 215nm

<table>
<thead>
<tr>
<th>Peak</th>
<th>Time[min]</th>
<th>Area[mV*s]</th>
<th>BL</th>
<th>width[sec]</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>FF</td>
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<td>11.9138</td>
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</table>

ee=76.1%
Sample name: (2R,3S)-8r

Analysis condition: Chiralpak IC, 10% iPrOH/n-hexane, 0.7 ml/min, 254nm
Sample name: (2R,3S)-8t

Analysis condition: Chiralpak IC, 10% iPrOH/n-hexane, 0.7 ml/min, 254nm

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Time [min]</th>
<th>Area [mV*s]</th>
<th>BL</th>
<th>Width [sec]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>BB</td>
<td>45.0000</td>
<td>28.2840</td>
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</table>

ee=47.4%