Supporting Information

Synthesis and properties of MIDA boronate containing aromatic amino acids: New peptide building blocks

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Part 1  \(^1\)H and \(^{13}\)C NMR Spectra
Part 2  Chiral HPLC Analysis for Compounds 9-11
$^{13}$C expansion: compound 1a
**13C expansion: compound 2a**

[Graph showing the 13C expansion spectrum of compound 2a with labels for ppm on the x-axis and intensity on the y-axis.]
Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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$^{13}$C expansion: compound 3a
$^{13}$C expansion: compound 12
$^{13}\text{C}$ expansion: compound 13
\(^{13}\)C expansion: compound 15
Chiral HPLC Test mix for 9a and 9b
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Data File: C:\CHEM32\DATA\ONY_0510170510000025.D
Sample Name: NCI11

===============================================================================
Acq. Operator : Katie Poole  Seq. Line : 3
Acq. Instrument : AS LC3  Location : Vial 13
Injection Date : 17/05/2010 18:00:47  Inj : 2
Different Inj Volume from Sequence : Actual Inj Volume : 10 µl
Acq. Method : C:\CHEM32\2\METHODS\WIDA06.M
Analysis Method : C:\CHEM32\2\METHODS\WIDA06.M
Last changed : 17/05/2010 18:22:47 by Katie Poole
Last changed : 18/05/2010 09:39:07 by Katie Poole
(modified after loading)
Method Info : Chiral purity by HPLC
Chiralcel IA 250 x 4.6mm, Sum P8 (Daicel; #80325)

Sample Info : (S)-Enantiomer

![Chiral HPLC chromatogram with structure of compound](image)

Area Percent Report

Sorted By : Signal
Calib. Data Modified : 10/05/2010 09:39:08
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD A, Wavelength=220 nm

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Area %</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.384</td>
<td>MM</td>
<td>0.179</td>
<td>45.30048</td>
<td>0.5238</td>
<td>(R)-Enantiomer</td>
</tr>
<tr>
<td>2</td>
<td>6.267</td>
<td>MM</td>
<td>0.2116</td>
<td>8603.71582</td>
<td>99.4762</td>
<td>(S)-Enantiomer</td>
</tr>
</tbody>
</table>

Totals : 8649.01630

1 Warnings or Errors:

AS LC3 18/05/2010 09:40:27 Katie Poole
Sample Name: NC197

Acq. Instrument: AS_LC1  
Location: Vial 14
Injection Date: 17/05/2010 18:16:51
Inj: 1

Different Inj Volume from Sequence | Actual Inj Volume: 5 μl

Acq. Method: C:\CHEM32\METHODS\MIDA60.M
Last changed: 17/05/2010 16:22:47 by Katie Poole
Analysis Method: C:\CHEM32\METHODS\MIDA60.M
Last changed: 18/05/2010 09:39:07 by Katie Poole
(modified after loading)

Method Info: Chiral purity by HPLC
Chiralcel IA 250 x 4.6mm, Sum PS (Daicel; #80325)

Sample Info: (R)-Enantiomer

Area Percent Report

Signal 1: VWD1 A, Wavelength=220 nm

Peak Ret Time Type Width Area % Area Name
0.1773 1.86628e4 98.7106 (R)-Enantiomer
0.2088 246.40179 1.2894 (S)-Enantiomer

Totals: 1.91093e4

AS_LC1 18/05/2010 09:41:07 Katie Poole
Chiral HPLC Test mix for 10a and 10b

Area Percent Report

Signal 1: VND1 A, Wavelength=220 nm

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type Width</th>
<th>Area</th>
<th>Area Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.586 min</td>
<td>0.1901 8502.86426</td>
<td>49.6346 (R)-Enantiomer</td>
<td></td>
</tr>
<tr>
<td>6.585 min</td>
<td>0.2506 8628.07422</td>
<td>50.3654 (S)-Enantiomer</td>
<td></td>
</tr>
</tbody>
</table>

Totals: 1.71309s4
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Data File C:\CHEM32\2\DATA\ONY_0510\170510000033.D
Sample Name: WC138

========================================================================================================
Acq. Instrument : A8_LC3          Location : Vial 17
Acq. Operator : Katie Poole      Inj : 2 
Injection Date : 17/05/2010 20:09:33  Inj Volume : 10 µl
Different Inj Volume from Sequence : Actual Inj Volume : 5 µl
Acq. Method : C:\CHEM32\2\METHODS\MIDA60.M
Last changed : 17/05/2010 16:22:47 by Katie Poole
Analysis Method : C:\CHEM32\2\METHODS\MIDA60.M
Last changed : 18/05/2010 11:47:40 by Katie Poole
(modified after loading)
Method Info : Chiral purity by HPLC
Chiralcel IA 250 x 4.6mm, Sum PS [Daicel; #80325]
Sample Info : (R)-Enantiomer

========================================================================================================            

Area Percent Report
========================================================================================================
Sorted By : Signal
Calib. Data Modified : 18/05/2010 11:47:40
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Type</th>
<th>Width [min]</th>
<th>Area [mAU]</th>
<th>Area %</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.597</td>
<td>MM</td>
<td>0.1812</td>
<td>7947.11475</td>
<td>99.9843</td>
<td>(R)-Enantiomer</td>
</tr>
<tr>
<td>2</td>
<td>6.596</td>
<td>MM</td>
<td>0.2636</td>
<td>41.19763</td>
<td>0.5157</td>
<td>(S)-Enantiomer</td>
</tr>
</tbody>
</table>

Totals : 7988.31257

1 Warnings or Errors :
AS_LC3 18/05/2010 11:47:59 Katie Poole
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Chiral HPLC Test mix for 11a and 11b

Data File C: \CHEM12\DATA\ONY_0510\170510000035.D
Sample Name: NC REF 6

================================================================================================
Acq. Operator : Katie Poole  Seq. Line : 8
Acq. Instrument : AS LC3  Location : Vial 18
Injection Date : 17/05/2010 20:41:40  Inj : 2
Different Inj Volume from Sequence : Actual Inj Volume : 10 µl
Acq. Method : C:\CHEM12\METHODS\MIDA60.M
Last changed : 17/05/2010 16:22:47 by Katie Poole
Analysis Method : C:\CHEM12\METHODS\MIDA60.M
Last changed : 18/05/2010 11:48:29 by Katie Poole
(modified after loading)
Method Info : Chiral purity by HPLC
Chiralcel IA 250 x 4.6 mm, Sum PG (Daicel; #80325)
Sample Info : NC136 + NC139
Chiralpak IA 250 x 150

Courous of Chiral HPLC Test mix for 11a and 11b

VWD1 A, Wavelength=220 nm (ONY_0510\170510000035.D)

Area Percent Report
================================================================================================
Sorted By : Signal
Calib. Data Modified : 18/05/2010 11:48:29
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type Width Area Area Name
# [min] [min] mAU *s %
--- --- --- --- --- --- --- --- --- --- --- --- --- --- --- ---
1 5.345 MM 0.1961 2.5545e4 70.9981 (R)-Enantiomer
2 6.603 MM 0.2255 1.0435e5 29.0019 (S)-Enantiomer
Totals : 3.59803e5

ASLC3 18/05/2010 11:49:03 Katie Poole

Page 1 of 2
Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Data File C:\CHEM32\2\DATA\ONY_0510\170510000037.D
Sample Name: NC316

====================================================================================================
| Acq. Operator   : Katie Poole | Acq. Instrument : AS LC3 | Location : Vial 19 |
| Injection Date  : 17/05/2010 21:13:51 | Inj : 2 |
| Different Inj Volume from Sequence : | Actual Inj Volume : 5 μL |
| Acq. Method : C:\CHEM32\2\METHODS\MIDA60.M | Last changed : 18/05/2010 11:48:29 by Katie Poole |
| Analysis Method : C:\CHEM32\2\METHODS\MIDA60.M | Last changed : 18/05/2010 11:48:29 by Katie Poole |
| Method Info : Chiral purity by HPLC (modified after loading) |
| Sample Info : (S)-Enantiomer |

Chiralc SIL 250 x 4.6mm, Sum P8 (Daicel; #80325)

VWD1 A, Wavelength=220 nm (ONY_0510/170510000037.D)

-- Area Percent Report --

Sorted By : Signal
Calib. Data Modified : 18/05/2010 11:48:29
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type Width Area % Area Name
# [min] [min] mAUs "s " ------------------- ------------------- ------------------- -------------------
1 5.240 MM 0.1708 60.70354 0.2150 (R)-Enantiomer
2 6.608 MM 0.2455 2.61718s4 99.7850 (S)-Enantiomer
Totals : 2.82325s4

1 Warnings or Errors :

AS LC3 18/05/2010 11:49:47 Katie Poole

Page 1 of 2
Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Data File C:\CHEM32\2\DATA\ONY_0510\1705100000039.D
Sample Name: NC139

Acq. Operator : Katie Poole
Seq. Line : 10
Acq. Instrument : A8_LC3
Location : Vial 20
Injection Date : 17/05/2010 21:46:03
Inj : 2
Inj Volume : 10 μL
Different Inj Volume from Sequence ! Actual Inj Volume : 5 μL

Acq. Method : C:\CHEM32\2\METHODS\MIDA60.M
Last changed : 17/05/2010 16:22:47 by Katie Poole
Analysis Method : C:\CHEM32\2\METHODS\MIDA60.M
Last changed : 18/05/2010 11:48:29 by Katie Poole
(modified after loading)
Method Info : Chiral pury by HPLD

Chiralcel IA 250 x 4.6mm, Sum P5 (Daicel; #80325)
Sample Info : (R)-Enantiomer

VWD1 A, Wavelength=220 nm (ONY_0510\1705100000039.D)

Area Percent Report

Area

Sorted By : Signal
Calib. Data Modified : 10/05/2010 11:48:29
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type Width Area Area % Name
# [min] [min] *μA [μA]

1 5.350 MF 0.1724 1.4019e4 99.7888 (R)-Enantiomer
2 6.601 MM 0.2500 29.66854 0.2112 (S)-Enantiomer

Totals : 1.40495e4

1 Warnings or Errors:

AS_LC3 18/05/2010 11:50:34 Katie Poole