Supporting Information

Diastereo- and Enantioselective Synthesis of Fluorinated Proline Derivatives via Copper(I)-Catalyzed Asymmetric 1,3-Dipolar Cycloaddition

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University of Vienna, Faculty of Chemistry, Waehringer Strasse 38, A-1090 Wien, Austria

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Table of Contents

1. The ORTEP diagram of X-ray diffraction structure of 3b

2. NMR spectra and HPLC analysis of compounds 3, 4f
1. The ORTEP diagram of X-ray diffraction structure of 3b

2. NMR spectra and HPLC analysis of compounds 3, 4f
F1: 100.598  F2: 400.031  SW1: 24510  OF1: 10563.3  PTS1d: 31875, 32768
EX: s2pul  PW: 7.1 us  PD: 1.0 sec  NA: 13708  LB: 0.0

USER: -- DATE: Jan 5 2011
Nuts - $lq-11-49-c.fid
Std Fluorine
F1: 376.375  F2: 100.597  SW1: 138889  OF1: -28230.3  PTS1d: 138889, 262144
EX: s2pul  PW: 7.2 us  PD: 3.0 sec  NA: 4  LB: 0.0

Nuts - Slq-11-69-F.fid

USER: -- DATE: Dec 15 2010
==== Shimadzu LCsolution Analysis Report ====  

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Sample Name: Iq-11-25  
method: OD-H, 214.70/30.0.7  
Injection Volume: 1 µL  
Data File Name: 11-25b.lcd  
Method File Name: 111.lcm  
Report File Name: 1.lcr  
Data Acquired: 2010-11-25 9:51:36  
Data Processed: 2010-11-25 10:24:17

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1 Det.A Ch1/214nm

Detector A Ch1 214nm

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Peak Table

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S-6

C:\LabSolutions\Data\Project1\iq11-25b.lcd
Shimadzu LC solution Analysis Report

Acquired by: Admin
Sample Name: lq-11-69
Method: OD-H, 214, 70, 30, 1.0
Injection Volume: 1 uL
Data File Name: 11-69.ldc
Method File Name: 111.lcm
Report File Name: 1.lcr
Data Acquired: 2010-12-13 9:51:15
Data Processed: 2010-12-13 10:25:31

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![Chemical Structure](image)

**Parameters:**
- **F1:** 75.450
- **F2:** 300.028
- **SW1:** 18797
- **OF1:** 7750.6
- **PTS1d:** 30075, 32768
- **EX:** s2pul
- **PW:** 4.6 us
- **PD:** 1.0 sec
- **NA:** 12356
- **LB:** 0.0

**USER:** -- **DATE:** Dec 7 2010

**Nuts - $lq-11-50-c.fid**

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**Page:** S.9
**** Shimadzu LCsolution Analysis Report ****

Acquired by: Admin
Sample Name: Ig-11-43racL
Method: OD-H, 214,70/30, 0.7
Injection Volume: 1 uL
Data File Name: 11-43-rac.lcd
Method File Name: 111.lcm
Report File Name: 1.lcr
Data Acquired: 2010-11-26 9:11:36
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Detector A Ch1 214nm

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C:\LabSolutions\Data\Project1\q\11-43-rac.lcd

Det.A Ch1
Shimadzu LCsolution Analysis Report

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Method: OD-H, 214.70/30, 0.7
Injection Volume: 1 µL
Data File Name: 11-43.lcd
Method File Name: 111.lcm
Report File Name: 1.lcr
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Detector A Ch1 214nm

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S-16
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Injection Volume: 1 uL
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Method File Name: 111.lcm
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Data Processed: 2010-11-26 11:39:58

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Detector A Ch1 214nm

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exo-3c
chl8-84C

F1: 75.450  F2: 300.028  SW1: 18797  OF1: 7751.6  PTS1d: 30075  32768
EX: s2pul  PW: 4.6  us  PD: 1.0  sec  NA: 12012  LB: 0.0

USER: -- DATE: Dec 9 2010
Nuts - Slq-11-56-c.fid
Std Fluorine

F1: 376.366  F2: 100.597  SW1: 156250  OF1: -37640.3  PTS1d: 49152 , 65536

EX: s2pul  PW: 7.2 us  PD: 1.0 sec  NA: 12  LB: 0.0

USER: -- DATE: Nov 15 2010  Nuts - Slq-11-56-F.fld
Shimadzu LC solution Analysis Report

Acquired by: Admin
Sample Name: Ig-11-66
Method: OD-H, 214, 80/20, 0.7
Injection Volume: 1 µL
Data File Name: 11-66b.lcd
Method File Name: 111.lcm
Report File Name: 1.lcr
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<Chromatogram>

Detector A Ch 1 214 nm

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Shimadzu LCsolution Analysis Report

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method: OD-H,214,80/20,0.7
Injection Volume: 1 µL
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Detector A Ch1 214nm

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F1: 282.286  F2: 300.028  SW1: 100000  OF1: -21341.7
PW: 5.0 us  PD: 3.0 sec  NA: 4  LB: 0.0

USER: -- DATE: Dec 17 2010
Nuts - Slq-11-63-F.fid
Shimadzu LC solution Analysis Report

Acquired by: Admin
Sample Name: IQ-11-68
Method: OD-H, 214, 80/20, 0.7
Injection Volume: 1 ul
Data File Name: IQ-11-68.lcd
Method File Name: 111.lcm
Report File Name: IQ-11-68.lcr
Data Acquired: 2010-12-2 19:38:40
Data Processed: 2010-12-2 20:32:25

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![Chromatogram Image]

**Peak Table**

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Shimadzu LC solution Analysis Report

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Sample Name: IQ-11-63
Injection Volume: OD-H, 214, 80/20, 0.7
Injection Volume: 1 µL
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Method File Name: 1111.lcm
Report File Name: 1.lcr
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Detector A Ch1 214nm

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EX: s2pul  PW: 5.0 us  PD: 2.0 sec  NA: 8  LB: 0.0

Nuts - Slq-11-45-F.fid

USER: -- DATE: Oct 28 2010
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: iq-11-44
method: Od-H, 214, 70/30, 0.7
Injection Volume: 1 uL
Data File Name: 11-44-70.lcd
Method File Name: 111.lcm
Report File Name: t.lcr
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Data Processed: 2010-11-5 12:30:07

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Detector A Ch1 214 nm

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Det.A Ch1
Shimadzu LCsolution Analysis Report

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Injection Volume: 1 μL
Data File Name: 11-45.lcd
Method File Name: 111.1cm
Report File Name: 1.lcr
Data Acquired: 2010-11-5 12:34:41
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Detector A Ch1 214nm

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EX: s2pul  PW: 7.1 us  PD: 1.0 sec  NA: 14136  LB: 0.0  
USER: -- DATE: Dec 16 2010  
Nuts - S1q-11-64-c.fid
Std Fluorine
F1: 376.375  F2: 100.597  SW1: 138889
EX: s2pul
PW: 7.2 us  PD: 3.0 sec

USER: -- DATE: Dec 16 2010
Nuts - S1q-11-64-F.fid
Shimadzu LC solution Analysis Report

Acquired by: Admin
Sample Name: Ig-11-67
Method: OD-H, 214, 80/20, 0.7
Injection Volume: 1 µL
Data File Name: 11-67.lcd
Method File Name: 111.lcm
Report File Name: 1.lcr
Data Acquired: 2010-12-2 21:36:29
Data Processed: 2010-12-2 22:01:04

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[Image of a chromatogram]

Detector A Ch1 214nm

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C:\LabSolutions\Data\Project1\ig\11-67.lcd

S-36
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: lg-11-58
method: OD-H, 70/30, 0.7, 214
Injection Volume: 10 μL
Data File Name: 11-58b.lcd
Method File Name: 1.lcm
Report File Name: 1.lcr
Data Acquired: 2011-4-25 12:17:26
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Detector A Ch1 214nm

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Shimadzu LC solution Analysis Report

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Injection Volume: 10 uL
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Method File Name: 1.lcm
Report File Name: 1.lcr
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Data Processed: 2011-4-25 12:06:55

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Detector A Ch1 214nm

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Peak Table
Std Fluorine
F1: 376.345  F2: 100.597  SW1: 192308  OF1: -58342.5  PTS1d: 262144 , 262144
EX: s2pul  PW: 6.1 us  PD: 3.0 sec  NA: 8  LB: 0.0

USER: -- DATE: Mar 11 2011
Nuts - $lq-11-90-F.fid
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: Ig-11-93
Method: OD-H, 70/30, 214, 0.7
Injection Volume: 10 μL
Data File Name: 11-93.lcd
Method File Name: cip-1-product-d-rac.lcm
Report File Name: 1.lcr
Data Acquired: 2011-3-23 10:56:57
Data Processed: 2011-3-23 11:53:26

<Chromatogram>

Detector A Ch1 214nm

<table>
<thead>
<tr>
<th>Peak#</th>
<th>Ret. Time</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.690</td>
<td>1687217</td>
<td>91467</td>
<td>49.808</td>
</tr>
<tr>
<td>2</td>
<td>29.093</td>
<td>1700258</td>
<td>30573</td>
<td>50.192</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>3387476</td>
<td>122140</td>
<td>100.000</td>
</tr>
</tbody>
</table>
Shimadzu LC Solution Analysis Report

Acquired by: Admin
Sample Name: lq-11-90
Method: OD, 70/30, 214, 0.7
Injection Volume: 10 µL
Data File Name: 11-90.lcd
Method File Name: cjp-1-product-d-rec.lcm
Report File Name: 1.lcr
Data Acquired: 2011-3-23 15:18:38
Data Processed: 2011-3-23 16:02:58

<Chromatogram>

Detector A Ch1 214nm Peak Table

<table>
<thead>
<tr>
<th>Peak#</th>
<th>Ret. Time</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.992</td>
<td>375954</td>
<td>14817</td>
<td>3.193</td>
</tr>
<tr>
<td>2</td>
<td>28.236</td>
<td>11399657</td>
<td>146663</td>
<td>96.807</td>
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<tr>
<td>Total</td>
<td></td>
<td>11774991</td>
<td>161480</td>
<td>100.000</td>
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</table>

Det. A Ch1
S-48
Std Fluorine

F1: 376.345  F2: 100.597  SW1: 192308  OF1: -58342.5  PTS1d: 262144

EX: s2pul  PW: 6.1 us  PD: 3.0 sec  NA: 4  LB: 0.0

USER: -- DATE: Mar 11 2011

Nuts - Slq-11-91-F.fid
### Shimadzu LC Solution Analysis Report

Acquired by: Admin  
Sample Name: Ig-11-94  
Method: OD.70/30,214,0.7  
Injection Volume: 10 uL  
Data File Name: 11-94.lcd  
Method File Name: sjp-1-product-d-rac.lcm  
Report File Name: 1.lcr  
Data Acquired: 2011-3-23 16:07:44  
Data Processed: 2011-3-23 16:42:13

#### Chromatogram

![Chromatogram](C:\LabSolutions\Data\Project1\qj11-94.lcd)

<table>
<thead>
<tr>
<th>Detector A Ch1 214nm</th>
<th>PeakTable</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak#</td>
<td>Ret. Time</td>
</tr>
<tr>
<td>1</td>
<td>12.046</td>
</tr>
<tr>
<td>2</td>
<td>27.478</td>
</tr>
<tr>
<td>Total</td>
<td></td>
</tr>
</tbody>
</table>

2011-3-23 16:43:58 1/1
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: iq-11-91
method: OD,70/30,214,0.7
Injection Volume: 10 uL
Data File Name: 11-91.lcd
Method File Name: cjp-1-product-d-rac.lcm
Report File Name: 1.lcr
Data Acquired: 2011-3-23 16:47:38
Data Processed: 2011-3-23 17:40:29

<Chromatogram>

```
Detector A Ch1 214nm

Peak	 Ret. Time	 Area	 Height	 Area %
---
1	 12.095	 813726	 28942	 3.532
2	 27.481	 22223175	 278196	 96.468
Total	 23036901	 307138	 100.000
```
**HPLC Report**

Sample Name: Data File: LQ-12-14+ PA-2 82 214 0.7..che  
Operator:  
Date: 2011-06-08  
Time: 15:34

![HPLC Graph](image)

<table>
<thead>
<tr>
<th>No.</th>
<th>Peak No</th>
<th>ID. Name</th>
<th>R. Time</th>
<th>Peak Height</th>
<th>Peak Area</th>
<th>Per Cent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td></td>
<td>15.922</td>
<td>56743.5</td>
<td>1791007.5</td>
<td>50.4526</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td></td>
<td>19.177</td>
<td>41037.4</td>
<td>1758870.6</td>
<td>49.5474</td>
</tr>
</tbody>
</table>

**Total**  
97780.9  
3549878.1  
100.0000

![Chemical Structure](image)
HPLC Report

Sample Name: Data File: LQ-12-13.che
Operator: Date: 2011-06-08
Time: 16:11

<table>
<thead>
<tr>
<th>No.</th>
<th>Peak No</th>
<th>ID Name</th>
<th>R. Time</th>
<th>PeakHeight</th>
<th>PeakArea</th>
<th>PerCent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td></td>
<td>15.228</td>
<td>782304.3</td>
<td>24204978.5</td>
<td>95.9377</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td></td>
<td>19.077</td>
<td>21861.2</td>
<td>1024925.3</td>
<td>4.0623</td>
</tr>
</tbody>
</table>

Total 804165.6 25229903.8 100.0000

![Chemical Structure](image)