Asymmetric Synthesis of Six-Membered Cyclic Sulfamides via Palladium-Catalyzed Alkene Carboamination Reactions

Zachary J. Garlets and John P. Wolfe*

Department of Chemistry, University of Michigan, 930 N. University Avenue, Ann Arbor, Michigan 48109-1055

Supporting Information
Experimental procedures and characterization data for new compounds.

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Determination of Absolute Configuration S1
Copies of $^1$H, $^{13}$C NMR spectra, and HPLC traces S2

Determination of Absolute Configuration of 6d

Colorless blocks of 6d were grown by vapor diffusion of diethyl ether into a dichloromethane solution of the compound at 22 °C. The structure analysis of 6d indicated this material had an S stereochemical configuration.
$5a$
$CDCl_3$

$5a$
$CDCl_3$
==== Shimadzu LCsolution Analysis Report ==== 

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S12
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S17
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![Chromatogram Image]

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mAU

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$\text{CDCl}_3$

$\text{6e}$
Shimadzu LCsolution Analysis Report

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mAU

PeakTable

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S28
Contains 5-10% of Unidentified Side Product
Shimadzu LCsolution Analysis Report

Acquired by: Admin
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PDA Multi

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Sample Name: RAC-ZJG-4-113-Bn-XPhos-4-oMe-me-4.00%IPA-1.0mL_min-ADH
Sample ID: Chiral-DW_7_63_hex-IPA_98to2-PR
Tray#: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: RAC-ZJG-4-113-Bn-XPhos-4-oMe-me-4.00%IPA-1.0mL_min-ADH4.tcd
Method File Name: Cyclic Urea Method.lcm
Report File Name: Default.lcr
Data Acquired: 4/10/2017 10:08:14 AM
Data Processed: 4/10/2017 10:47:40 AM

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C:\\...\Membered Sulfonamide Mono\RAC-ZJG-4-113-Bn-XPhos-4-oMe-me-4.00%IPA-1.0mL_min-ADH4.tcd

mAU

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16.0
17.5
20.0
22.5
25.0
min

1 PDA Multi 1/210nm 4mm

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==== Shimadzu LCsolution Analysis Report ==== 

Acquired by: Admin  
Sample Name: CHIRAL-ZJG-4-113-Bn-S-Siphos-PE-4-oMe-me-4.00%IPA-1.0mL_min-ADH  
Sample ID:  
Tray #: 1  
Vial #: 1  
Injection Volume: 1 μL  
Data File Name: CHIRAL-ZJG-4-113-Bn-S-Siphos-PE-4-oMe-me-4.00%IPA-1.0mL_min-ADH4.lcd  
Method File Name: Cyclic Urea Method.lcm  
Batch File Name:  
Report File Name: Default.lcr  
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<Chromatogram> 

6h  

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Shimadzu LC solution Analysis Report

Acquired by: Admin
Sample Name: RAC-ZJG-5-92-cyclohexyl-Bn-XPhos-mphenol4F-6.00%IPA-1.0mL_min-ADH6.lcd
Sample ID: 1
Tray#: 1
Val#: 1
Injection Volume: 1 μL
Data File Name: RAC-ZJG-5-92-cyclohexyl-Bn-XPhos-mphenol4F-6.00%IPA-1.0mL_min-ADH6.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name: 
Report File Name: Default.icr
Data Acquired: 7/22/2016 2:32:28 PM
Data Processed: 7/22/2016 3:10:32 PM

<Chromatogram>

C:\\RAC-ZJG-5-92-cyclohexyl-Bn-XPhos-mphenol4F-6.00%IPA-1.0mL_min-ADH6.lcd

PDA Multi 1

1 PDA Multi 1/210nm 4nm

PeaksTable

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C:\LabSolutions\Data\Zac G6 Membered Sulfonamide
Mono\RAC-ZJG-5-92-cyclohexyl-Bn-XPhos-mphenol4F-6.00%IPA-1.0mL_min-ADH6.lcd

S36
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-ZJG-5-99-cyclohexyl-Bn-S-Siphos-PE-mphenol4F-6.00%IPA-1.0mL_min-ADH7.lcd
Sample ID:
Tray#: 1
Vail #: 1
Injection Volume: 1.0 uL
Data File Name: CHIRAL-ZJG-5-99-cyclohexyl-Bn-S-Siphos-PE-mphenol4F-6.00%IPA-1.0mL_min-ADH7.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 7/23/2016 3:18:11 PM
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PDA Multi 1

PDA Ch1 210nm 4nm

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mAU

0 250 500 750 1000

22.5 25 27.5 30 32.5 35.0 min
$\text{6j}$

$\text{CDCl}_3$
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-ZJG-5-91-cyclohexyl-Bn-XPhos-mCF3-6.00%IPA-1.0mL_min-ADH
Sample ID: 1
Tray #: 1
Well #: 1
Injection Volume: 1 µL
Data File Name: RAC-ZJG-5-91-cyclohexyl-Bn-XPhos-mCF3-6.00%IPA-1.0mL_min-ADH4.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: Default.lcr
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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-ZJG-5-94-cyclohexyl-Bn-XPhos-mCF3-6.00%IPA-1.0mL_min-ADH
Sample ID:
Tray #: 1
Val # : 1
Injection Volume: 1 µL
Data File Name: CHIRAL-ZJG-5-94-cyclohexyl-Bn-XPhos-mCF3-6.00%IPA-1.0mL_min-ADH
Method File Name: Cyclic Urea Method lcr
Batch File Name:
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![Chromatogram Image]

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### Shimadzu LCsolution Analysis Report

C:\... Membered Sulfonamide Mono\RAC-ZJG-5-95-cyclohexyl-Bn-XPhos-mmethoxy-6.00%IPA-1.0mL_min-AD7 ldc

- **Acquired by:** Admin
- **Sample Name:** RAC-ZJG-5-95-cyclohexyl-Bn-XPhos-mmethoxy-6.00%IPA-1.0mL_min-AD
- **Tray #:** 1
- **Vial #:** 1
- **Injection Volume:** 1 uL
- **Data File Name:** RAC-ZJG-5-95-cyclohexyl-Bn-XPhos-mmethoxy-6.00%IPA-1.0mL_min-ADH7 ldc
- **Method File Name:** Cyclic Urea Method ldc
- **Batch File Name:** Default ldc
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- **Data Processed:** 7/22/2016 5:43:42 PM

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![Chromatogram Image]

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**Total**

21528508 | 4165231 | 100.000 | 100.000 |
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-ZJG-5-109-trial2-cyclohexyl-Bn-S-Slphos-PE-mmethoxy-6.00
Sample ID:
Tray#: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: CHIRAL-ZJG-5-109-trial2-cyclohexyl-Bn-S-Slphos-PE-mmethoxy-6.00%IPA-1.0mL_min-ADH9
Method File Name: Cyclic Urea Method.lcm
Batch File Name: Default.lcr
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6k
### Shimadzu LCsolution Analysis Report

**Acquired by:** Admin  
**Sample Name:** RAC-ZJG-5-36-Bn-XPhos-4-1naph-2.00%IPA-0.75mL_min-ODH6.lcd  
**Sample ID:**  
**Tray #:** 1  
**Val #:** 1  
**Injection Volume:** 1 µL  
**Data File Name:** RAC-ZJG-5-36-Bn-XPhos-4-1naph-2.00%IPA-0.75mL_min-ODH6.lcd  
**Method File Name:** Cyclic Urea Method.icm  
**Batch File Name:**  
**Report File Name:** Default.icr  
**Data Acquired:** 6/1/2016 2:48:55 PM  
**Data Processed:** 6/1/2016 3:23:33 PM

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![Chromatogram Image]

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**Output File:** C:\LabSolutions\Data\Zac G\6 Membered Sulfonamide Mono\RAC-ZJG-5-36-Bn-XPhos-4-1naph-2.00%IPA-0.75mL_min-ODH6.lcd