Catalytic Asymmetric Intramolecular Aminopalladation: Improved Palladium(II) Catalysts

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Supporting Information

Copies of ^1H and ^{13}C NMR spectra for 2-oxazolidinones **6**, **8**, **10** and **12**; copies of HPLC traces to determine enantiopurity of **6**, **8**, **10** and **12**.

(10 Pages)

General experimental details: All HPLC analyses used to determine enantiomeric purity were calibrated with samples of the racemate. All reactions were performed under Argon. CH_2Cl_2 was dried according to published procedures.¹ COP-OAc, di- μ -acetatobis[η^5 -(*S*)-(p*R*)-2-(2'-(4'-methylethyl)oxazoliny)cyclopentadienyl,1-C,3'-*N*)(η^4 -tetraphenylcyclobutadiene)cobalt] dipalladium [(+)-**4**] was prepared according to published procedures.²

¹ a) Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.
b) GlassContour Homepage: <http://www.glasscontour.com>.

² (a) Anderson, C. E.; Kirsch, S. F.; Overman, L. E.; Richards, C. J.; Watson, M. P. *Org. Synth.* submitted for publication; available from the senior author upon request. (b) Stevens, A. M.; Richards, C. J. *Organometallics* **1999**, *18*, 1346.

6 (entry 6)

sk238
¹H spectrum

sk259
1H spectrum

sk257
1H spectrum

sk264
1H spectrum

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Injection Date   : 5/19/04 3:53:38 PM      Seq. Line :    1
Sample Name     : sk252g                  Location  : Vial 51
Acq. Operator   : LEO                     Inj       :    1
                                           Inj Volume: 8 µl

Acq. Method     : C:\HPCHEM\1\METHODS\SK2.M
Last changed    : 5/19/04 3:53:23 PM by LEO
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SKXX.M
Last changed    : 12/20/04 5:15:53 PM by Vinh
                  (modified after loading)

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Odh column, 10% iPrOH/90% Hexanes, 1.0 mL/min

Area Percent Report

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Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs

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Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.953	MM	0.8719	130.36264	2.49200	3.6587
2	27.933	MM	1.2300	3432.71387	46.51216	96.3413

Totals : 3563.07651 49.00416

Results obtained with enhanced integrator!

*** End of Report ***

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Injection Date	: 5/20/04 2:56:35 PM	Seq. Line	: 1
Sample Name	: sk256c	Location	: Vial 51
Acq. Operator	: LEO	Inj	: 1
		Inj Volume	: 8 µl
Acq. Method	: C:\HPCHEM\1\METHODS\SK2.M		
Last changed	: 5/20/04 12:49:38 PM by LEO		
Analysis Method	: C:\HPCHEM\1\METHODS\SKXX.M		
Last changed	: 12/20/04 5:17:36 PM by Vinh		
	(modified after loading)		

Odh column, 10% iPrOH/90% Hexanes, 1.0 mL/min

DAD1 B, Sig=254,16 Ref=360,100 (D:\DATA\STEFAN\SK256C01.D)

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.255	MM	0.8574	24.20664	4.70570e-1	2.3271
2	28.340	MM	1.2138	1015.99005	13.95080	97.6729

Totals : 1040.19670 14.42137

Results obtained with enhanced integrator!

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*** End of Report ***

2% IPA in Hex, AS, 1ml/min

Injection Date : 5/27/04 1:28:57 PM
 Sample Name : sk259 Location : Vial 51
 Acq. Operator : stefan Inj Volume : 4 µl
 Acq. Method : C:\HPCHEM\1\METHODS\SK2.M
 Last changed : 5/27/04 1:26:02 PM by stefan
 (modified after loading)
 Analysis Method : C:\HPCHEM\1\METHODS\SKXX.M
 Last changed : 12/20/04 3:33:51 PM by Vinh
 (modified after loading)
 AS column, 2% iPrOH/98% Hexanes, 1.0 mL/min

Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	40.626	MM	3.6593	2.57132e4	117.11204	99.2628
2	53.185	MM	3.6138	190.97217	8.80749e-1	0.7372

Totals : 2.59042e4 117.99279

Results obtained with enhanced integrator!

*** End of Report ***

10% IPA in Hex, OdH, 1ml/min

Injection Date : 5/26/04 4:01:22 PM

Sample Name : sk257

Location : Vial 51

Acq. Operator : stefan

Inj Volume : 8 µl

Acq. Method : C:\HPCHEM\1\METHODS\SK2.M

Last changed : 5/26/04 4:01:21 PM by stefan
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\SK2.M

Last changed : 5/26/04 6:38:59 PM by stefan
(modified after loading)

10% IPA in Hexanes, Odh column, 1.0 ML/MIN

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.114	BB	0.4423	442.58783	15.53955	1.6096
2	12.361	BB	0.5498	2.70538e4	770.24597	98.3904

Totals : 2.74964e4 785.78553

Results obtained with enhanced integrator!

Summed Peaks Report

S9

Injection Date : 6/2/04 5:34:11 PM Seq. Line : 1
Sample Name : SK264 Location : Vial 51
Acq. Operator : CC Inj : 1
Inj Volume : 2 µl
Acq. Method : C:\HPCHEM\1\METHODS\SKXX.M
Last changed : 6/2/04 5:32:36 PM by CC
Analysis Method : C:\HPCHEM\1\METHODS\SKXX.M
Last changed : 12/20/04 3:26:36 PM by Vinh
(modified after loading)

OdH, 10% iPrOH/90% Hexanes, 1.0 mL/min

DAD1 D, Sig=230,16 Ref=360,100 (D:\DATA\STEFAN\SK264002.D)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.427	MM	0.7301	346.99713	5.68137	4.5550
2	30.322	MM	1.6547	7270.86475	73.23663	95.4450

Totals : 7617.86188 78.91800

Results obtained with enhanced integrator!

*** End of Report ***