Tandem Enantioselective Conjugate Addition- Cyclopropanation. Formal Synthesis of (-)-(S,S)-Clavukerin A and (+)-(R,S)-Isoclavukerin

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Supporting Information

Description of compound 5a :	S2
Description of compound 5b :	S4
Description of compound 5c :	S6
Description of compound 5d :	S8
Description of compound 6a :	S10
Description of compound 6b :	S12
Description of compound 6c :	S14
Description of compound 6d :	S16
Description of compound 8a :	S18
Description of compound 9a :	S22
Description of compound 10a :	S26
Description of compound 10b :	S30
Description of compound 10c :	S34
Description of compound 10d :	S38

File : C:\HPCHEM\2\DATA\SM250AL2.D
Operator : Sébastien
Acquired : 1-14-02 15:38:59 using AcqMethod B60-30.M
Instrument : HP G1530A
Sample Name:
Misc Info : B60-30-LipodexE
Vial Number: 1

File : C:\HPCHEM\2\DATA\SM258A1T.D
Operator : Sébastien
Acquired : 2-6-02 9:39:25 using AcqMethod B60-40.M
Instrument : HP G1530A
Sample Name: SM 258 A11
Misc Info : 50-90-15-170-0
Vial Number: 1

File : C:\HPCHEM\2\DATA\SMCYC7.D
Operator : Sébastien
Acquired : 7-19-02 21:19:48 using AcqMethod B60-40.M
Instrument : HP G1530A
Sample Name: racémate cyclopropanol à 7C
Misc Info : Lip E, 50-90-15-170-0
Vial Number: 1

File : C:\HPCHEM\2\DATA\SM298AL1.D
Operator : Sébastien
Acquired : 7-22-02 23:01:59 using AcqMethod B601-70.M
Instrument : HP G1530A
Sample Name: idem
Misc Info : ChiralDEX B-TA, 60-0-1-80-10-15-170-0
Vial Number: 1

File : C:\HPCHEM\2\DATA\SM298RAC.D
Operator : Sébastien
Acquired : 7-23-02 00:02:03 using AcqMethod B601-70.M
Instrument : HP G1530A
Sample Name: idem
Misc Info : ChiralDEX B-TA, 60-0-1-80-10-15-170-0
Vial Number: 1

File : C:\HPCHEM\2\DATA\SM257AL1.D
Operator : Sébastien
Acquired : 2-3-02 12:14:57 using AcqMethod DEFAULT.M
Instrument : HP G1530A
Sample Name: SM257 Al1
Misc Info : 60-50-1-70-0-15-270-0 (lipodex E)
Vial Number: 1

File : C:\HPCHEM\2\DATA\RACBZ.D
Operator : Sébastien
Acquired : 7-26-02 12:11:24 using AcqMethod DEFAULT.M
Instrument : HP G1530A
Sample Name: Et-Benzalacetone env Rac
Misc Info : Lip E, 60-50-1-70-0-15-170-0
Vial Number: 8

¹H Spectrum

*** Current Data Parameters ***
 NAME : sm216fr2
 EXPNO : 10
 PROCNO : 1
 *** Processing Parameters ***
 SF : 400.1300170 MHz
 *** 1D NMR Plot Parameters ***
 Hz_cm : 113.50

¹³C CPD-Spectrum

*** Current Data Parameters ***

NAME : sm216fr2

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127786 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 1013.95

¹H Spectrum

*** Current Data Parameters ***

NAME : xr19fr5b

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300051 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 123.49

¹³C CPD-Spectrum

*** Current Data Parameters ***

NAME : *xr19fr5b*

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127706 MHz

*** 1D NMR Plot Parameters ***

Hz_{cm} : 1013.95

¹H Spectrum

*** Current Data Parameters ***

NAME : sm284f7

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 121.08

¹³C CPD-Spectrum

*** Current Data Parameters ***

NAME : sm284f7

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127700 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 976.79

¹H Spectrum

7.3108
7.2931
7.2262
7.2067

*** Current Data Parameters ***

NAME : xr22puri

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300052 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 118.67

6d

2.3136
2.2889
1.7718
1.7131
1.4277
1.2895
0.8785
0.8595
0.8412
0.7800
0.7642
0.6922
0.5072
0.3191
0.3045
0.2894
0.1821
0.1095

¹³C CPD-Spectrum

*** Current Data Parameters ***

NAME : xr22puri

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127713 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 1013.95

March 11 PROTON CDCl3 u guest 22

H1 spectrum

*** Current Data Parameters ***

NAME : sm272f8

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 300.1300123 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 90.54

9a

C13 Spectrum

*** Current Data Parameters ***

NAME : sm272f8

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 75.4677527 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 731.64

9a

March 11 PROTON CDCl3 u guest 1

March 13 CPD CDCl3 u guest 1

*** Current Data Parameters ***

NAME : sm192pur

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577964 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 1402.76

File : C:\HPCHEM\2\DATA\SM266F18.D
Operator : Sébastien
Acquired : 5-13-02 21:45:42 using AcqMethod B60-40.M
Instrument : HP G1530A
Sample Name:
Misc Info : Lip E, 80-35-15-170-10
Vial Number: 8

File : C:\HPCHEM\2\DATA\S233F12B.D
Operator : Sébastien
Acquired : 5-11-02 5:45:32 using AcqMethod B60-40.M
Instrument : HP G1530A
Sample Name: Racémate
Misc Info : Lip E, 80-50-15-170-10
Vial Number: 8

¹H Spectrum

7.2700
6.1279
6.1253
6.0963
6.0938
6.0919
6.0786
6.0609
6.0477

*** Current Data Parameters ***

NAME : xr24fr10

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 308.89

10b

3.4837
3.4660

2.9300
2.9022
2.8334
2.8176
2.5303
2.5234
2.5139

1.8144
1.8030
1.7058
1.5915
1.5808
1.4753
1.4570
1.2998
1.2701
1.2676
1.2360
0.9778
0.9595
0.9412
0.8774

Integral

1.0000

0.5258

0.5140

0.5169

0.5372

0.5873

1.5425

(ppm)

¹³C CPD-Spectrum

*** Current Data Parameters ***

NAME : xr24fr10

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127700 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 976.79

10b

7.2700
6.3495
6.3400
6.3192
6.3097
5.9479
5.9435
5.9176
5.9126

*** Current Data Parameters ***

NAME : sm288f8

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 308.89

2.6023
2.3138
1.9975
1.9823
1.9697
1.9488
1.9343
1.9217
1.8402
1.8245
1.8087
1.7929
1.7777
1.7178
1.6470
1.5568
1.5151
1.4652
1.1786
1.1609

Sebastien □ C13CPD CDCl3 u guest 1 □

*** Current Data Parameters ***

NAME : sm288f8

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 1013.95

File : C:\HPCHEM\2\DATA\SM300F14.D
Operator :
Acquired : 7-17-02 19:27:36 using AcqMethod F70-801.M
Instrument : GC 6890
Sample Name: hydrodex B3P, 70-40-15-170-5
Misc Info :
Vial Number: 6

File : C:\HPCHEM\2\DATA\S296F14D.D
Operator : Sébastien
Acquired : 6-19-02 4:13:54 using AcqMethod F70-801.M
Instrument : HP G1530A
Sample Name: racémate
Misc Info : Hydrodex B3P, 70-40-15-170-5
Vial Number: 8

¹H Spectrum

*** Current Data Parameters ***

NAME : kc4-350

EXPNO : 20

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300052 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 308.89

10d

¹³C CPD-Spectrum

*** Current Data Parameters ***

NAME : kc4-350

EXPNO : 22

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Hz_cm : 1013.95

10d

CHIRALCEL OD-H

Area Percent Report

Sorted by Signal

Multiplier : 1.000000

Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.484	MM	0.114	11054.94531	1615.12097	93.3946
2	4.881	MM	0.119	781.86926	109.08824	6.6054

Totals : 11836.81445 1724.20923

*** End of Report ***

CHIRALCEL OD-H

Area Percent Report

Sorted by Signal

Multiplier : 1.000000

Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.381	MM	0.114	6883.84863	1006.77881	50.0573
2	4.764	MM	0.123	6868.07471	929.07922	49.9426

Totals : 13751.92383 1935.85803

*** End of Report ***