C₂-Symmetric Bisprolinamide as Highly Efficient Catalyst for Direct Aldol Reaction

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Supplementary Material

Procedure for the Preparation of Catalyst 3-5:

Experimental procedure for the synthesis of A-C: To a stirred solution of *N*-carbobenzyloxy-L-proline (500 mg, 2.0 mmol) in CH_2Cl_2 (20 mL) was added the corresponding diamine (1.0 mmol), EDCI (425 mg, 2.2 mmol), HOBT (300 mg, 2.2 mmol) and sodium bicarbonate (8.0 mmol) at 0 °C and the reaction mixture was stirred at room temperature for 24 h. Water (20 mL) was then added to quench the reaction. The mixture was extracted with ethyl acetate (3×20 mL), washed with aqueous NaHCO_3 (10 mL), aqueous HCl (1 N, 20 mL), brine (3×10 mL) and dried over anhydrous MgSO_4 . Afterwards the organic phase was evaporated under reduced pressure to give the crude product, which was purified by column chromatography through silica gel, eluting with 4:1 ethyl acetate/hexane solvent mixture, to give the pure **A-C**.

***N,N'*-Bis[(*S*)-*N*-benzyloxycarbonylpropyl]-1,2-ethylenediamine (A):** Yield 93%, ^1H and ^{13}C NMR spectroscopic data are in agreement with those reported.⁹

(1*R*,2*R*)-*N,N'*-Bis[(*S*)-*N*-benzyloxycarbonylpropyl]-1,2-diphenyl-1,2-ethanediamine

(B): Yield 89%; ^1H NMR (500 MHz, CDCl_3) δ 1.81-2.28 (m, 8H), 3.41-3.62 (m, 4H), 4.18-4.33 (m, 2H), 5.11-5.29 (m, 6H), 6.83-7.38 (m, 20H); ^{13}C NMR (125 MHz, CDCl_3) δ 24.3,* 30.1,* 47.5,* 58.7, 61.1,* 67.6, 127.4, 127.6, 128.0, 128.3, 128.5, 128.8, 136.5,* 138.8,* 155.7,* 172.4.* (* Doblet due to existence of rotomers)

(1*S*,2*S*)-*N,N'*-Bis[(*S*)-*N*-benzyloxycarbonylpropyl]-1,2-diphenyl-1,2-ethanediamine

(C): Yield 91%; ^1H NMR (500 MHz, CDCl_3) δ 1.67-2.35 (m, 8H), 3.26-3.60 (m, 4H), 4.17 -4.43 (m, 2H), 5.01-5.12 (m, 6H), 6.82-7.32 (m, 20H); ^{13}C NMR (125 MHz, CDCl_3) δ 24.3,* 30.5,* 47.4,* 58.7, 60.5,* 67.5 (2C), 127.5, 127.7, 128.0, 128.2, 128.6, 128.8, 136.7, 138.7,* 155.8,* 172.7.* (* Doblet due to existence of rotomers)

Experimental procedure for the synthesis of 3-5: To a stirred solution of **A-C** (1.0 g) in dry methanol (40 mL) was added 10% Pd/C (0.8 g) under hydrogen (2 atm) for 5 h. Then the solution was filtered through short plug celite and washed with methanol (30 mL). The filtrate was evaporated under reduced pressure to give the crude product, which was purified by column chromatography through silica gel (1:1 methanol/ethyl acetate) to afford pure **3-5**.

***N,N'*-Bis[(*S*)-propyl]-1,2-ethylenediamine (3):** Yield 85%, ^1H and ^{13}C NMR spectroscopic data are in agreement with those reported.⁹

(1*R*,2*R*)-*N,N'*-Bis[(*S*)-prolyl]-1,2-diphenylethane-1,2-diamine (4): Yield 83%; mp 168-169 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.67-2.17 (m, 10 H), 2.89-3.01 (m, 4H), 3.64-3.67 (m, 2H), 5.19-5.22 (m, 2H), 7.07-7.19 (m, 10H), 8.39 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 26.5, 31.0, 47.5, 58.3, 60.9, 127.6, 127.7, 128.5, 139.3, 175.5.

(1*S*,2*S*)-*N,N'*-Bis[(*S*)-prolyl]-1,2-diphenylethane-1,2-diamine (5): Yield 79%; mp 68-69 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.60-2.12 (m, 10H), 2.85-3.02 (m, 4H), 3.73-3.76 (m, 2H), 5.20 (dd, *J*= 2.5, 6 Hz, 2H), 7.08-7.22 (m, 10H), 8.54 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 26.2, 30.8, 47.4, 58.9, 60.9, 127.6, 127.7, 128.6, 139.1, 175.3.

Experimental procedure for the synthesis of catalyst 6

(1*S*,2*S*)-*N*-[(*S*)-prolyl]-*N*-acetyl-1,2-diphenylethane-1,2-diamine (6): To a stirred solution of *N*-carbobenzyloxy-L-proline (500 mg, 2.0 mmol) in CH₂Cl₂ (20 mL) was added (1*S*,2*S*)-*N*-acetyl-1,2-diphenyl-1,2-ethylenediamine¹¹ (2.0 mmol), EDCI (425 mg, 2.2 mmol), HOBT (300 mg, 2.2 mmol) and triethyl amine (8.0 mmol) at 0 °C and then reaction mixture was stirred at room temperature for 24 h. The reaction mixture was quenched with water and the reaction mixture was extracted with ethyl acetate (3 × 20 mL), washed with aqueous NaHCO₃, aqueous HCl (1 N 20 mL), brine (3 × 10 mL) and dried over anhydrous MgSO₄. Afterward the organic phase was evaporated under reduced pressure to give the crude product.

The obtained *N*-protected compound (485 mg, 1 mmol) was hydrogenated (3 atm) by 10 mol % Pd/C (30 mg) in dry methanol (30 mL) for 5 h. After that the reaction mixture was filtered on Celite to remove the Pd/C, and then evaporated to dryness to give a crude

product, which was purified by column chromatography (silica gel), eluting with EtOAc/Hexane (4:1) solvent mixture, to give the pure catalyst **6** (Yield: 89%).

¹H NMR (500 MHz, CDCl₃) δ 1.57-1.69 (m, 2H), 1.95 (s, 3H), 2.08-2.19 (m, 4H), 2.82-2.86 (m, 1H), 2.99-3.03 (m, 1H), 3.89-3.92 (m, 1H) 5.18 (t, *J* = 9 Hz, 1H), 5.29 (2d, *J*₁ = 9.00 Hz, *J*₂ = 8.00 Hz, 1H), 7.12-7.33 (m, 10H), 8.64 (d, *J* = 8.00 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 23.6, 26.0, 30.7, 47.3, 59.0, 59.7, 60.6, 127.5 (4C), 127.6 (2C), 127.7, 128.0, 128.6 (2C), 128.7 (2C), 138.5, 139.4, 170.4, 175.0.

General experimental procedure for the aldol reaction: To a stirred solution of catalyst **5** (13.4 mg, 0.025 mmol, 10 mol%) in anhydrous acetone (0.5 mL) was added the aldehyde (0.25 mmol) at -35 °C. The stirring was continued for 24 h (for Table 2, entries 11 and 12, 48 h). The reaction mixture was quenched with saturated aqueous ammonium chloride solution, extracted with ethyl acetate (3 × 10 mL) [for Table 2, entries 11 and 12, with diethyl ether (3 × 10 mL)]. The organic layer was washed with water and dried over MgSO₄. After evaporation of the solvent under reduced pressure, the crude product was purified by column chromatography over silica gel using ethyl 1:4 acetate/hexane as the eluent to provide the pure aldol product. All aldol products are known compounds and their spectroscopic data are identical with those reported.^{6,7}

The ee values of the aldol products were determined by HPLC analysis on a chiral HPLC column. The HPLC conditions and retention times were collected in Table 3.

Table 3. HPLC conditions and Retention Times for the Enantiomers^a

Compound	Eluent <i>i</i> -PrOH/Hexane	Column	UV Detection (nm)	T _R (min)	T _S (min)
	15/85	AS	220	23.3	34.3
	20/80	OJ-H	220	17.4	19.7
	30/70	AS	220	9.6	7.8
	30/70	AS	220	10.8	22.0
	10/90	AS	220	10.5	13.8
	10/90	AS	220	13.8	19.0
	20/80	AS	220	8.3	10.3
	10/90	AS	220	12.0	10.5

To be continued.

Table 3 continued

Compound	Eluent <i>i</i> -PrOH/Hexane	Column	UV Detection (nm)	T _R (min)	T _S (min)
	15/85	AS	220	8.4	10.5
	15/85	AS	220	9.8	12.2
	20/80	AS	220	15.4	18.0
	3/97	AD-H	280	7.7	8.9
	10/90	AD-H	280	7.0	8.0
 <i>rac</i> -	30/70	OJ-H	220	9.6 (<i>R,R</i>)	11.2 (<i>S,S</i>)
	30/70	AS	220	13.1	18.6
 <i>rac</i> -	30/70	AD-H	220	6.3 (<i>R,R</i>) ^b	5.8 (<i>S,S</i>) ^b
 <i>rac</i> -	30/70	AD-H	220	7.0 ^c	8.5 ^c

To Be Continued.

Table 3 continued

Compound	Eluent <i>i</i> -PrOH/Hexane	Column	UV Detection (nm)	T _R (min)	T _S (min)
 <chem>C[C@H](O)[C@H](C1CCCC1=O)c2ccc([N+](=O)[O-])cc2</chem> <i>rac</i> -	15/85	AS	220	28.2 (<i>R,R</i>)	23.0 (<i>S,S</i>)
 <chem>C[C@H](O)[C@@H](C1CCCC1=O)c2ccc([N+](=O)[O-])cc2</chem> <i>rac</i> -	15/85	As	220	61.1 (<i>R,S</i>)	19.4 (<i>S,R</i>)

^a Flow rate was 1.0 mL/min for all cases, except for the last two entries, where the flow rate was 0.8 mL/min. Absolute configuration of the enantiomers was determined by comparing the measured optical rotation with the reported data. ^bTentatively assigned.

^cAbsolute configuration not determined.

Additional Reference:

11. Isobu, T.; Fukuda, K.; Hosogai, A. Jpn. Kokai Tokkyo Koho **1998**, 6 pp. JP 10045721 A2 19980217 Heisei. Application: JP 96-198683 19960729; CA: **1998**, 128:204889, AN 1998:112694.

Nucleus
 Scans 1D
 Scan Count
 SW +/-
 Dwell 1D
 Points 1D
 Acq. Points
 F1 freq
 F2 freq
 RXVR gain
 TX Power
 DEC Power
 DEC Scheme
 DEC BW
 PW01
 Last Delay
 Dummy scan
 FILTER
 Sequence
 Field
 Acq. Time
 LB 1D
 Phase 0
 Phase 1
 GN set
 lock freq
 Solvent
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 250u
 16384
 16384
 300.6500030
 0.0000000
 1170
 62
 2850
 WALTZ 16
 256
 3u
 1s
 0
 2000
 7.0615070
 4.096s
 0.30
 -138.03
 -17.59
 0
 0.000
 Chloroform

16 peaks found in IL-SS-106
 peak ppm freq amp
 1 7.308 2197.14 4070.20
 2 7.202 2165.16 3101.96
 3 7.197 2163.70 2157.59
 4 7.116 2139.28 3138.98
 5 7.009 2107.30 2679.37
 6 5.206 1565.06 1377.62
 7 5.130 1542.36 971.64
 8 5.072 1524.78 1719.46
 9 5.033 1513.31 1317.14
 10 4.793 1441.04 328.98
 11 4.790 1440.06 335.32
 12 4.754 1429.32 232.51
 13 4.187 1258.67 783.75
 14 3.340 1004.27 901.57
 15 1.986 597.05 1210.77
 16 1.756 527.95 1862.59

STANDARD CARBON PARAMETERS

II-SS-100carbon1
 Archive directory: /home/samanta/vnmrSYS/data
 Sample directory: /home/samanta/vnmrSYS/data
 Sample name: II-SS-100carbon1

File: CARBON

Pulse Sequence: szpul

PRG: zgpg30

Acquire: 1000000000

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STANDARD PROTON PARAMETERS
II-SS-98a

Archive directory: /home/samanta/vmr/sys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
INOVA-500 "Varian500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.0 Hz
64 repetitions

OBSERVE H1, 499.7485607 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 5 sec

STANDARD CARBON PARAMETERS
II-SS-98carbon

Archive directory: /home/samanta/vnmrSYS/data
Sample directory:
File: CARBON

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
User: 1-14-87
INOVA-500 "Varian500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
22272 repetitions
OBSERVE C13, 125.6618455 MHz
DECOUPLE H1, 499.7510595 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FI size 131072
Total time 16 hr, 2 min, 14 sec

STANDARD CARBON PARAMETERS
11-SS-10carbon

Archive directory: /home/samanta/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
User: 1-14-87
INQVA-500 "Varian500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
3000 repetitions
OBSERVE C13, 125.6602075 MHz
DECOUPLE H1, 499.7445451 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 1 hr, 55 min, 28 sec

STANDARD PROTON PARAMETERS II-SS-100

Archive directory: /home/.../sys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "Varian500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.0 Hz
10 repetitions
OBSERVE H1, 499.7485607 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 29 sec

STANDARD CARBON PARAMETERS

Archive directory: /home/samanta/vnmrSYS/data
 Sample directory:
 File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

INNOVA-500 "Varian500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

5248 repetitions

OBSERVE C13, 125.6618455 MHz

DECOUPLE H1, 499.7510595 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 3 hr, 50 min, 56 sec

STANDARD PROTON PARAMETERS

Archive directory: /home/samanta/vnmrSYS/data
 Sample directory:
 File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3
 Ambient temperature
 INOVA-500 "Varian500"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 7996.0 Hz
 160 repetitions
 OBSERVE H1, 499.7420463 MHz
 DATA PROCESSING
 FT size 32768
 Total time 9 min, 40 sec

STANDARD CARBON PARAMETERS

II-SS149carbon

Archive directory: /home/samanta/vnmrSYS/data

Sample directory:

File: CARBON

Pulse Sequence: s2pu1

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

INOVA-500 "Varian500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

5056 repetitions

OBSERVE C13, 125.6602075 MHz

DECOUPLE H1, 499.7445451 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 3 hr, 50 min, 56 sec

Area % Report

Page 1 of 1

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\4-NITROBENZALDEHYDERACEMIC
User: System
Acquired: 7/23/2005 3:58:18 PM
Printed: 7/23/2005 4:44:15 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
23.358	58903795	49.94	494817	63.80
34.308	59046273	50.06	280819	36.20
Totals	117950068	100.00	775636	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\01-JY-28G
User: System
Acquired: 7/25/2005 11:11:10 AM
Printed: 7/25/2005 11:59:45 AM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
23.200	164428445	99.14	1144174	98.61
33.758	1422777	0.86	16169	1.39
Totals	165851222	100.00	1160343	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\II-SS-154A
 User: System
 Acquired: 8/16/2005 3:52:44 PM
 Printed: 8/16/2005 4:43:50 PM

Table 1, Entry 6

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
25.467	39956374	98.99	307183	99.07
41.417	407893	1.01	2881	0.93

Totals	40364267	100.00	310064	100.00

racemic

Page 1 of 1

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\II-3-NITROBENZALDEHYDERACEMIC1
 User: System
 Acquired: 7/15/2005 1:27:03 PM
 Printed: 7/22/2005 5:17:29 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
17.392	122153243	49.71	2715448	51.89
19.692	123565693	50.29	2518094	48.11
Totals	245718936	100.00	5233542	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\01-JY-29B
User: System
Acquired: 6/28/2005 5:01:54 PM
Printed: 7/22/2005 5:19:01 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
0.000	0	0.00	0	0.00
15.275	70547178	94.15	2064243	93.20
17.600	4383749	5.85	150564	6.80
Totals	74930927	100.00	2214807	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\II-SS-2-NITRORACEMIC-2
User: System
Acquired: 7/9/2005 3:42:19 PM
Printed: 7/22/2005 5:13:46 PM

racemic

SPD-10AVvp
Ch1-257nm
Results

Retention Time	Area	Area %	Height	Height %
7.783	33265187	49.96	1026401	58.76
9.608	33319783	50.04	720441	41.24

Totals	66584970	100.00	1746842	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\II-SS-2-NITRO
 User: System
 Acquired: 7/8/2005 3:17:39 PM
 Printed: 7/8/2005 3:44:02 PM

SPD-10AVvp
 Ch1-257nm
 Results

Retention Time	Area	Area %	Height	Height %
7.617	4798605	3.52	192383	6.41
8.800	131644307	96.48	2808817	93.59

Totals	136442912	100.00	3001200	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\II-SS-123A
User: System
Acquired: 7/11/2005 4:05:20 PM
Printed: 7/22/2005 4:53:30 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
10.517	36698771	49.87	1071377	58.48
13.808	36889762	50.13	760577	41.52
Totals	73588533	100.00	1831954	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\II-SS-108b
User: System
Acquired: 7/1/2005 11:35:06 AM
Printed: 7/22/2005 4:55:27 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
10.842	114915057	96.37	2376665	96.26
14.983	4329203	3.63	92226	3.74
Totals	119244260	100.00	2468891	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\II-SS-124
 User: System
 Acquired: 7/11/2005 5:45:42 PM
 Printed: 7/22/2005 4:48:06 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
13.842	2461975	49.87	48024	55.86
18.983	2475050	50.13	37944	44.14
Totals	4937025	100.00	85968	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\II-SS-109a
User: System
Acquired: 7/1/2005 2:06:24 PM
Printed: 7/22/2005 5:00:30 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
14.192	73281937	97.62	1322925	97.86
20.158	1786154	2.38	28948	2.14
Totals	75068091	100.00	1351873	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\II-SS-125B
User: System
Acquired: 7/13/2005 12:25:53 AM
Printed: 7/22/2005 4:43:23 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
8.300	16890834	47.82	545334	55.16
10.308	18430656	52.18	443250	44.84
Totals	35321490	100.00	988584	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\01-JY-32B
 User: System
 Acquired: 6/30/2005 7:05:40 PM
 Printed: 7/22/2005 4:42:13 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
7.108	122842	0.47	1503	0.19
7.508	24513019	93.69	760035	95.24
9.692	1527648	5.84	36467	4.57
Totals	26163509	100.00	798005	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\01-JY-43RACEMIC-D
 User: System
 Acquired: 7/18/2005 1:58:34 PM
 Printed: 7/18/2005 2:15:04 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
10.492	5196371	49.76	163339	56.38
11.975	5246290	50.24	126387	43.62
Totals	10442661	100.00	289726	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\01-JY-36E
 User: System
 Acquired: 7/18/2005 3:33:46 PM
 Printed: 7/18/2005 3:54:53 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
10.208	3633562	7.36	137360	13.12
11.392	45763722	92.64	909781	86.88
Totals	49397284	100.00	1047141	100.00

racemic

Page 1 of 1

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\01-JY-44RACEMIC-A
 User: System
 Acquired: 7/19/2005 12:45:44 PM
 Printed: 7/22/2005 5:01:06 PM

SPD-10AVvp
 Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
10.833	7632128	49.96	200494	74.34
21.967	7644546	50.04	69214	25.66
Totals	15276674	100.00	269708	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\01-JY-45
User: System
Acquired: 7/19/2005 7:20:50 PM
Printed: 7/22/2005 5:01:51 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
9.783	88565946	90.84	2059393	95.50
19.792	8929806	9.16	96975	4.50
Totals	97495752	100.00	2156368	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\01-JY-46RACEMIC
User: System
Acquired: 7/20/2005 12:55:09 PM
Printed: 7/22/2005 4:03:07 PM

racemic

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
9.750	3164247	49.81	106473	54.67
12.167	3188177	50.19	88266	45.33
Totals	6352424	100.00	194739	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met

Data: C:\EZStart\Projects\Default\Data\01-JY-34

User: System

Acquired: 7/1/2005 6:52:40 PM

Printed: 7/22/2005 4:04:34 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
8.675	36422879	94.83	857646	93.81
10.975	1987026	5.17	56606	6.19
Totals	38409905	100.00	914252	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\II-SS-119B
User: System
Acquired: 7/10/2005 4:08:27 PM
Printed: 7/22/2005 4:14:09 PM

SPD-10AVvp
Ch1-257nm
Results

Retention Time	Area	Area %	Height	Height %
8.425	7752863	50.04	190120	59.47
10.558	7740616	49.96	129555	40.53
Totals	15493479	100.00	319675	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\01-JY-39D
User: System
Acquired: 7/11/2005 1:49:45 PM
Printed: 7/22/2005 4:17:09 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
9.067	3298201	96.47	90285	96.56
11.800	120834	3.53	3216	3.44
Totals	3419035	100.00	93501	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-21
User: System
Acquired: 9/9/2005 1:17:32 PM
Printed: 9/9/2005 2:02:24 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
15.425	1469848	49.23	28710	60.11
18.017	1515690	50.77	19052	39.89
Totals	2985538	100.00	47762	100.00

racemic

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-16
User: System
Acquired: 9/9/2005 11:55:04 AM
Printed: 9/9/2005 3:38:06 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
15.350	15098110	97.88	272732	97.89
18.292	327321	2.12	5870	2.11

Totals	15425431	100.00	278602	100.00

96% ee

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\II-SS-129A
User: System
Acquired: 7/16/2005 3:28:49 PM
Printed: 7/16/2005 3:44:47 PM

SPD-10AVvp
Ch1-280nm
Results

Retention Time	Area	Area %	Height	Height %
7.633	4085004	49.85	222162	56.57
8.875	4109081	50.15	170578	43.43
Totals	8194085	100.00	392740	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\II-SS-131A
 User: System
 Acquired: 7/18/2005 10:19:14 AM
 Printed: 7/18/2005 10:32:39 AM

SPD-10AVvp
 Ch1-280nm
 Results

Retention Time	Area	Area %	Height	Height %
7.817	1486853	99.03	98281	99.00
9.333	14593	0.97	993	1.00
Totals	1501446	100.00	99274	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\II-SS-126A
 User: System
 Acquired: 7/13/2005 3:11:08 AM
 Printed: 7/13/2005 3:22:11 AM

SPD-10AVvp
 Ch1-280nm
 Results

Retention Time	Area	Area %	Height	Height %
7.000	584228	50.92	41846	52.28
8.017	563095	49.08	38197	47.72
Totals	1147323	100.00	80043	100.00

Area

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\CYCLOHEXANAL5
User: System
Acquired: 7/20/2005 1:56:53 PM
Printed: 7/20/2005 2:13:49 PM

SPD-10AVvp
Ch1-280nm
Results

Retention Time	Area	Area %	Height	Height %
7.283	419887	95.05	32361	94.95
7.642	16453	3.72	1268	3.72
8.183	5416	1.23	452	1.33
Totals	441756	100.00	34081	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\III-SS-CYCLOPENTANONE1
 User: System
 Acquired: 9/10/2005 10:36:11 AM
 Printed: 9/10/2005 5:00:48 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
19.183	36984377	38.31	467514	58.98
23.033	11675754	12.09	138610	17.49
28.583	11479196	11.89	107626	13.58
58.675	36409956	37.71	78868	9.95
Totals	96549283	100.00	792618	100.00

Syn/Anti (racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\III-DR-15A-2
 User: System
 Acquired: 9/10/2005 1:38:53 PM
 Printed: 9/10/2005 4:49:59 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
19.350	4926337	23.19	74175	39.27
22.950	854893	4.02	12224	6.47
28.150	8389145	39.48	83358	44.13
61.108	7076254	33.31	19144	10.13
Totals	21246629	100.00	188901	100.00

ee anti = 82% ; ee syn = 18%

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-1a
User: System
Acquired: 8/23/2005 4:15:14 PM
Printed: 9/9/2005 6:28:35 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
9.575	13992158	33.25	767794	39.64
10.267	6905242	16.41	314625	16.24
11.133	14413601	34.25	649706	33.54
15.575	6773913	16.10	204965	10.58
Totals	42084914	100.00	1937090	100.00

racemic (anti/syn)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-7B-4
User: System
Acquired: 9/1/2005 2:20:34 PM
Printed: 9/9/2005 3:48:12 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
9.242	77550824	96.52	3133239	95.63
10.867	1921	0.00	1275	0.04
11.142	2790689	3.47	141763	4.33
Totals	80343434	100.00	3276277	100.00

93% ee (anti)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-2b-1
User: System
Acquired: 8/24/2005 4:55:53 PM
Printed: 8/24/2005 5:22:10 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
13.125	7784719	49.72	150432	62.71
18.625	7870876	50.28	89445	37.29
Totals	15655595	100.00	239877	100.00

racemic

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-13B1
User: System
Acquired: 9/5/2005 6:07:30 PM
Printed: 9/9/2005 6:09:28 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
12.575	6860340	98.40	136747	98.81
18.358	111467	1.60	1646	1.19
Totals	6971807	100.00	138393	100.00

97% ee

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-2A-11
User: System
Acquired: 9/8/2005 12:18:56 PM
Printed: 9/8/2005 12:32:08 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
5.767	12150455	29.24	1387789	33.95
6.308	12232501	29.44	1265335	30.95
6.975	8455305	20.35	799329	19.55
8.592	8717943	20.98	635887	15.55
Totals	41556204	100.00	4088340	100.00

racemic (anti/syn)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-DR-13A-3
User: System
Acquired: 9/8/2005 12:02:30 PM
Printed: 9/8/2005 12:15:13 PM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
5.833	1619361	13.07	191873	15.17
6.392	7833935	63.24	819428	64.78
7.067	1414788	11.42	140472	11.10
8.800	1519262	12.26	113186	8.95
Totals	12387346	100.00	1264959	100.00

anti (66% ee)/syn (4%)