Organocatalytic Highly Enantioselective Nitroaldol Reaction of α -Ketophosphonates and Nitromethane

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Supporting Information

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Experimental Procedures

General

^1H and ^{13}C NMR spectra were obtained on a Varian INOVA 500 MHz spectrometer. HPLC analysis was performed on a Shimadzu instrument with LC-10AT pump and SPD-10AV UV-Vis detector. Chiralcel and Chiralpak HPLC columns were purchased from Daicel Chemical Industry, Ltd. Except for ethyl benzoylphosphonate (**4a**), which was purchased from Aldrich and used as received, α -ketophosphonates,¹³ as well as the catalysts, cupreine (**2**)^{11a,12} and 9-*O*-benzylcupreine (**3**),^{11a,12} were prepared by using the reported procedures. THF was freshly distilled over potassium. Other reagents and solvents were purchased from either Aldrich or Acros and used as received.

General experimental procedure for the enantioselective nitroaldol reaction of α -ketophosphonates and nitromethane

To a stirred solution of the α -ketophosphonate (0.25 mmol) and nitromethane (0.2 mL) in dry THF (0.2 mL) was added catalyst **2** (3.9 mg, 0.0125 mmol, 5 mol %) at 0 °C and the reaction mixture was allowed to stir for the time as specified in Table 2 (monitored by TLC). Afterwards the reaction mixture was quenched by adding a few drops of saturated ammonium chloride solution. The reaction mixture was then extracted with ethyl acetate (2 x 10 mL), washed with brine (2 mL) and dried over MgSO₄. The crude product, obtained after evaporation of the solvent, was purified by column chromatography (eluting with EtOAc/hexane) to provide the pure desired product.

In the case of catalyst **3**, all the reactions were carried out in a similar manner; except for the reaction temperature was room temperature.

Most of the products are known compounds,⁷ except for the following:

Diethyl [1-(4-Bromophenyl)-1-hydroxy-2-nitroethyl]phosphonate (**5k**)

White needles, mp 110-112 °C; IR (neat): ν_{max} 3173, 1558, 1486, 1420 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.20 (t, J = 6.5 Hz, 3H), 1.30 (t, J = 6.5 Hz, 3H), 3.85-3.90 (m, 1H), 3.98-4.01 (m, 1H), 4.14-4.18 (m, 2H), 4.52-4.55 (br s, 1H), 5.02 (dd, J_1 = 4.0, J_2 = 13.5 Hz, 1H), 5.12 (dd, J_1 = 7.0, J_2 = 12.5 Hz, 1H), 7.48-7.54 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 16.5 (t), 64.7 (d), 64.9 (d), 75.40 (d, J_{CP} = 163.6 Hz), 79.9 (d), 123.3, 128.1 (d), 131.9 (d), 134.6. Anal. Calcd. for C₁₂H₁₇BrNO₆P: C, 37.72; H, 4.48; N, 3.67. Found: C, 37.85; H, 4.60; N, 3.68.

Diethyl [1-Hydroxy-1-(4-methoxyphenyl)-2-nitroethyl]phosphonate (**5n**)

White needles, mp 92-94 °C; IR (neat): ν_{max} 3200, 1604, 1550, 1510, 1443, 1421, 1392 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.17 (t, J = 7.00 Hz, 3H), 1.31 (t, J = 7.00 Hz, 3H), 3.76-3.86 (m, 4H), 3.92-4.00 (m, 1H), 4.11-4.19 (m, 2H), 4.35 & 4.38 (2s, 1H), 5.017 (dd, J_1 = 4.0, J_2 = 13.5 Hz, 1H), 5.15 (dd, J_1 = 7.5, J_2 = 13.5 Hz, 1H), 1H), 6.92 (d, J = 8.5 Hz, 2H), 7.52 (dd, J_1 = 2.5, J_2 = 8.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 16.5 (t),

55.5, 64.3 (d), 64.8 (d), 75.4 (d, $J_{CP} = 166.0$ Hz), 80.1 (d), 114.2 (d), 127.0, 127.6 (d), 160.1 (d). Anal. Calcd. for $C_{13}H_{20}NO_7P$: C, 46.85; H, 6.05; N, 4.20. Found: C, 47.10; H, 6.21; N, 4.20.

Diethyl [1-Hydroxy-2-nitro-1-(thiophen-2-yl)ethyl]phosphonate (**5o**)

White needles, mp 76-78 °C; IR (neat): ν_{max} 3210, 1561, 1410, 1390 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$) δ 1.24 (t, $J = 7.00$ Hz, 3H), 1.32 (t, $J = 7.00$ Hz, 3H), 3.92-3.99 (m, 1H), 4.03-4.09 (m, 1H), 4.17-4.22 (m, 2H), 5.02-5.02 (m, 3H), 7.01-7.03 (m, 1H), 7.14-7.15 (m, 1H), 7.33-7.34 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 16.5 (q), 64.8 (d), 65.1 (d), 74.7 (d, $J_{CP} = 169.7$ Hz), 80.3 (d), 125.9 (d), 126.8 (d), 127.6 (d), 139.6 (d). Anal. Calcd. for $C_{10}H_{16}NO_6PS$: C, 38.83; H, 5.21; N, 4.53. Found: C, 39.04; H, 5.31; N, 4.57.

Experimental procedure for the reduction of the nitroaldol product **5c** to a β -amino- α -hydroxyphosphonate

To a solution of diisopropyl [1-hydroxy-2-nitro-1-phenylethyl]phosphonate (**5c**, 331 mg, 1.0 mmol, 98 % ee) in 10 mL of methanol was added 60 mg of palladium over activated charcoal (10 % w/w). The mixture was stirred under hydrogen atmosphere (1 atm) until the completion of the reduction (2.5 h, monitored by TLC). The reaction mixture was passed through a plug of Celite and washed with methanol (10 mL). An oily product was obtained after methanol was evaporated (211 mg, 70 % yield).

To determine the ee value of this product, it was directly protected with a benzoyl group without further purification: the reduced product was dissolved in CH_2Cl_2 (5 mL), and triethyl amine (85 mg, 0.84 mmol) and benzoyl chloride (108 mg, 0.77 mmol) were added at the room temperature with stirring. The reaction completed after 7 min as indicated by TLC analysis. Afterwards the reaction mixture was extracted with ethyl acetate (5 ml $\times$ 3). The combined extracts were washed with brine (5 ml $\times$ 2) and dried

over MgSO₄. Evaporation of the solvent yielded the crude product, which was purified by column chromatography to give the pure compound **6** (270 mg, 95% yield). This product was characterized by its ¹H and ¹³C NMR spectroscopic data: colorless needles solid, mp 125-127 °C; IR (neat) ν 3277, 1687, 1637, 1576, 1559, 1475, 1459, 1318 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 0.91 (d, J = 6.0 Hz, 3H), 1.15 (d, J = 6.00 Hz, 3H), 1.19 (d, J = 6.00 Hz, 3H), 1.24 (d, J = 6.00 Hz, 3H), 4.09-4.20 (m, 2H), 4.39-4.43 (m, 1H), 4.68-4.72 (m, 1H), 5.4 (brs, 1H), 7.27 -7.55 (m, 7H), 7.73-7.78 (m, 4H) ppm; ¹³C NMR (CDCl₃, 125 MHz) δ 23.3 (d), 24.0 (d), 24.1 (d), 24.4 (d), 48.9 (d), 72.5 (d), 72.8 (d), 77.5 (d, J_{cp} = 163.00 Hz), 126.7 (d), 127.5, 127.8 (d), 128.2 (d), 128.6, 131.9, 133.9, 138.7, 169.9 ppm.

The ee value of compound **6** was determined to 97% by HPLC analysis using a Chiralpak AD-H column.

Additional Reference

13. Baccolini, G.; Faggiano, M.; Todesco, P. E. *J. Chem. Soc., Perkin Trans. 1* **1979**, 2329-2333.

STANDARD PROTON PARAMETE
I-TM-benzoylprotection

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

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
8 repetitions
OBSERVE H1, 499.6847200 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec

6

¹H NMR

STANDARD CARBON PARAMETERS

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

Pulse Sequence: s2pul

Solvent: CDC13
Ambient temperature
User: 1-14-87
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
896 repetitions
OBSERVE C13, 125.6457928 MHz
DECOUPLE H1, 499.6872185 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 29 sec

6

¹³C NMR

STANDARD PROTON PARAMETERS
VII-SS-122

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

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
File: VII-SS-122
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
20 repetitions
OBSERVE H1, 499.6847200 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 58 sec

STANDARD CARBON PARAMETERS
VII-SS-122c

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

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
User: 1-14-87
File: VII-SS-122c
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6457928 MHz
DECOUPLE H1, 499.6872185 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 29 sec

STANDARD PROTON PARAMETERS
VII-SS-129

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

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
File: VII-SS-129
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
8 repetitions
OBSERVE H1, 499.6847200 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec

5n

STANDARD CARBON PARAMETERS
VII-SS1-129c

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

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: VII-SS-129c
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6457928 MHz
DECOUPLE H1, 499.6872185 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 29 sec

5n

STANDARD PROTON PARAMETERS
VII-SS-131

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

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
File: VII-SS-131
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
8 repetitions
OBSERVE H1, 499.6847200 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec

50

STANDARD CARBON PARAMETERS
VII-SS-131c

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

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
User: 1-14-87
File: VII-SS-131c
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
2500 repetitions
OBSERVE C13, 125.6457928 MHz
DECOUPLE H1, 499.6872185 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 1 hr, 36 min, 13 sec

50

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-1
 User: System
 Acquired: 10/23/2006 4:00:28 PM
 Printed: 12/13/2006 2:25:50 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
22.325	31039191	49.63	621121	57.94
28.675	31497587	50.37	450847	42.06
Totals	62536778	100.00	1071968	100.00

(5a, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-105OJ
 User: System
 Acquired: 12/4/2006 11:14:52 AM
 Printed: 12/13/2006 10:30:06 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
19.708	14108786	98.79	318799	99.08
25.358	172407	1.21	2950	0.92
Totals	14281193	100.00	321749	100.00

(5a, 98 % ee)
 catalyst **3**

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-35
 User: System
 Acquired: 11/6/2006 5:24:32 PM
 Printed: 12/13/2006 10:35:19 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.458	3305944	50.15	292435	52.32
6.775	3286216	49.85	266503	47.68
Totals	6592160	100.00	558938	100.00

(5b, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-123
 User: System
 Acquired: 12/8/2006 10:15:15 AM
 Printed: 12/13/2006 10:32:14 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
6.283	212464	2.32	21444	2.89
6.617	8961844	97.68	719754	97.11
Totals	9174308	100.00	741198	100.00

(5b, 95 % ee)
 catalyst 3

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-68OJ
 User: System
 Acquired: 11/19/2006 1:09:00 PM
 Printed: 12/13/2006 10:36:03 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
7.900	16276453	49.59	583350	69.25
14.033	16545745	50.41	259003	30.75
Totals	32822198	100.00	842353	100.00

(5c, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-30OJ
 User: System
 Acquired: 11/19/2006 1:42:07 PM
 Printed: 12/13/2006 10:36:29 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
7.942	8306569	98.75	301904	99.38
13.950	104827	1.25	1893	0.62
Totals	8411396	100.00	303797	100.00

(5c, 98 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-39G
 User: System
 Acquired: 11/17/2006 3:55:48 PM
 Printed: 12/13/2006 10:39:48 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.042	3770875	49.18	366197	53.05
6.567	3896317	50.82	324138	46.95
Totals	7667192	100.00	690335	100.00

(5d, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-42P
 User: System
 Acquired: 11/17/2006 4:45:24 PM
 Printed: 12/13/2006 2:47:27 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
6.058	324058	1.90	35896	2.61
6.533	16724650	98.10	1339245	97.39
Totals	17048708	100.00	1375141	100.00

(5d, 96 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-20AD
 User: System
 Acquired: 11/22/2006 3:47:20 PM
 Printed: 12/13/2006 10:41:15 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
8.858	2412593	49.31	151600	55.98
10.292	2475061	50.59	118364	43.71
10.875	5081	0.10	846	0.31
Totals	4892735	100.00	270810	100.00

(5e, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-77AD
 User: System
 Acquired: 11/22/2006 4:46:40 PM
 Printed: 12/13/2006 10:43:50 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
8.775	349131	2.54	22741	3.40
10.133	13370025	97.44	645338	96.53
10.875	1877	0.01	464	0.07

Totals	13721033	100.00	668543	100.00
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(**5e**, 95 % ee)
 catalyst **2**

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-59
 User: System
 Acquired: 11/17/2006 12:59:20 PM
 Printed: 12/13/2006 10:44:20 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
5.250	2232731	49.19	262457	51.14
5.575	2306218	50.81	250720	48.86
Totals	4538949	100.00	513177	100.00

(5f, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-43
 User: System
 Acquired: 11/17/2006 2:00:21 PM
 Printed: 12/13/2006 10:44:45 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
5.242	2324	0.04	472	0.08
5.567	5721612	99.96	609449	99.92

Totals	5723936	100.00	609921	100.00
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(5f, >99 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-63OJ
 User: System
 Acquired: 11/18/2006 3:00:11 PM
 Printed: 12/13/2006 10:45:11 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
15.075	61200500	49.25	1396728	54.04
17.025	63067976	50.75	1187964	45.96
Totals	124268476	100.00	2584692	100.00

(5g, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-640J
 User: System
 Acquired: 11/18/2006 3:36:58 PM
 Printed: 12/13/2006 10:45:30 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
15.308	27935695	97.92	694386	97.95
17.758	592342	2.08	14536	2.05
Totals	28528037	100.00	708922	100.00

(5g, 96 % ee)**catalyst 2**

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-27AD
 User: System
 Acquired: 11/4/2006 3:32:18 PM
 Printed: 12/13/2006 10:36:54 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
6.192	4639156	50.47	473122	53.63
6.717	4552166	49.53	409025	46.37
Totals	9191322	100.00	882147	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-58AD1
 User: System
 Acquired: 11/17/2006 12:06:31 PM
 Printed: 12/13/2006 10:37:42 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.667	305812	3.68	30407	4.51
7.225	7998275	96.32	643608	95.49
Totals	8304087	100.00	674015	100.00

(5h, 93 % ee)

catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-22
 User: System
 Acquired: 11/4/2006 10:06:02 AM
 Printed: 12/13/2006 10:38:08 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
16.483	2557881	4.33	59091	5.37
18.567	27817477	47.05	566013	51.39
19.875	28743548	48.62	476237	43.24
Totals	59118906	100.00	1101341	100.00

(5i, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-24
 User: System
 Acquired: 11/4/2006 10:47:14 AM
 Printed: 12/13/2006 10:38:34 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
18.408	70808517	96.47	1441821	95.63
19.742	2588082	3.53	65870	4.37
Totals	73396599	100.00	1507691	100.00

(5i, 93 % ee)

catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-37
 User: System
 Acquired: 11/7/2006 11:37:35 AM
 Printed: 12/13/2006 10:38:56 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
5.450	22086450	49.84	2318980	51.45
6.083	22227085	50.16	2188501	48.55
Totals	44313535	100.00	4507481	100.00

(5j, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-33P1
 User: System
 Acquired: 11/17/2006 12:32:07 PM
 Printed: 12/13/2006 10:39:20 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
5.808	130995	0.64	15469	0.79
6.525	20193282	99.36	1931561	99.21
Totals	20324277	100.00	1947030	100.00

(5j, 99% ee)

catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\I-TM-720J
User: System
Acquired: 11/21/2006 11:39:54 AM
Printed: 12/13/2006 2:26:45 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
15.858	10804488	48.34	227145	51.55
17.383	11547502	51.66	213490	48.45
Totals	22351990	100.00	440635	100.00

(5k, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-73OJ
 User: System
 Acquired: 11/21/2006 11:15:37 AM
 Printed: 12/13/2006 2:27:06 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
15.517	31137197	97.11	621107	96.83
17.092	927172	2.89	20329	3.17

Totals	32064369	100.00	641436	100.00
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(5k, 94 % ee)

catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-60
 User: System
 Acquired: 11/17/2006 2:33:20 PM
 Printed: 12/13/2006 2:26:09 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.725	4801859	50.42	398061	51.64
7.017	4722059	49.58	372733	48.36
Totals	9523918	100.00	770794	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-129
 User: System
 Acquired: 12/10/2006 12:56:52 PM
 Printed: 12/13/2006 10:31:50 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.567	65031886	98.48	3213717	98.00
6.925	1006916	1.52	65581	2.00
Totals	66038802	100.00	3279298	100.00

(5I, 97 % ee)

catalyst 3

Area % Report

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

Data: C:\EZStart\Projects\Default\Data\I-TM-23

User: System

Acquired: 11/5/2006 11:42:08 AM

Printed: 12/13/2006 2:25:04 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
12.192	18912334	50.50	798230	51.97
12.792	18538828	49.50	737721	48.03
Totals	37451162	100.00	1535951	100.00

(5m, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-26
 User: System
 Acquired: 11/5/2006 1:56:25 PM
 Printed: 12/13/2006 2:24:48 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
12.233	197137	0.69	20783	1.85
12.842	28483741	99.31	1101405	98.15
Totals	28680878	100.00	1122188	100.00

(5m, 99 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-65OJ
 User: System
 Acquired: 11/21/2006 3:30:59 PM
 Printed: 12/13/2006 2:24:24 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
24.167	7130464	50.05	114103	58.55
33.425	7116127	49.95	80765	41.45
Totals	14246591	100.00	194868	100.00

(5n, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-67
 User: System
 Acquired: 11/21/2006 2:38:53 PM
 Printed: 12/13/2006 2:23:50 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
23.933	7839829	98.91	129750	99.20
33.433	86266	1.09	1044	0.80
Totals	7926095	100.00	130794	100.00

(5n, 98 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-610J
 User: System
 Acquired: 11/18/2006 10:08:36 AM
 Printed: 12/13/2006 11:03:18 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
12.075	4875704	49.26	179761	56.75
14.558	5022204	50.74	137001	43.25
Totals	9897908	100.00	316762	100.00

(5o, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-510J
 User: System
 Acquired: 11/18/2006 11:12:45 AM
 Printed: 12/13/2006 11:03:34 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
12.142	5055984	96.31	182491	96.91
14.775	193465	3.69	5825	3.09
Totals	5249449	100.00	188316	100.00

(5o, 93 % ee)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-90AD
 User: System
 Acquired: 11/28/2006 9:52:24 AM
 Printed: 12/13/2006 11:03:54 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
8.458	7110395	49.85	409316	52.01
9.050	7151893	50.15	377640	47.99
Totals	14262288	100.00	786956	100.00

(5p, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-91AD5
 User: System
 Acquired: 11/28/2006 10:33:14 AM
 Printed: 12/13/2006 11:04:41 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
8.458	20599117	97.63	1143631	97.72
9.042	500088	2.37	26643	2.28
Totals	21099205	100.00	1170274	100.00

(5p, 95 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\sgo
 User: System
 Acquired: 11/19/2006 3:09:34 PM
 Printed: 12/13/2006 11:05:37 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.900	15035017	48.89	619875	54.02
7.800	15715724	51.11	527595	45.98

Totals	30750741	100.00	1147470	100.00
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(5q, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-46AS
 User: System
 Acquired: 11/19/2006 3:40:47 PM
 Printed: 12/13/2006 11:05:20 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
6.933	82747	0.77	3464	0.99
7.800	10634250	99.23	347036	99.01
Totals	10716997	100.00	350500	100.00

(5q, 98 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-75S
 User: System
 Acquired: 11/22/2006 12:31:59 PM
 Printed: 12/13/2006 11:06:02 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
16.625	23890790	46.90	387897	55.48
18.750	27049306	53.10	311261	44.52
Totals	50940096	100.00	699158	100.00

(5r, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-76
 User: System
 Acquired: 11/22/2006 9:59:15 AM
 Printed: 12/13/2006 11:06:20 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
17.125	301373	4.51	5118	6.50
19.217	6383663	95.49	73566	93.50

Totals	6685036	100.00	78684	100.00
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(5r, 91 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-79P
 User: System
 Acquired: 11/23/2006 2:10:06 PM
 Printed: 12/13/2006 11:07:09 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
5.117	6133951	50.23	339593	61.58
6.583	6077425	49.77	211898	38.42
Totals	12211376	100.00	551491	100.00

(5s, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-80AS
 User: System
 Acquired: 11/23/2006 4:15:52 PM
 Printed: 12/13/2006 11:07:27 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
5.142	1133174	1.54	68250	3.54
6.350	72226910	98.46	1858362	96.46
Totals	73360084	100.00	1926612	100.00

(5s, 97 % ee)
 catalyst 2

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-82OJ
 User: System
 Acquired: 11/24/2006 1:19:45 PM
 Printed: 12/13/2006 11:07:51 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
7.717	7258750	48.55	391955	54.13
8.367	7692932	51.45	332089	45.87
Totals	14951682	100.00	724044	100.00

(5t, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-117OJ
 User: System
 Acquired: 12/7/2006 12:51:11 PM
 Printed: 12/13/2006 10:31:27 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
8.483	336449	2.21	17858	3.10
9.342	14872465	97.79	558277	96.88
10.875	174	0.00	117	0.02
Totals	15209088	100.00	576252	100.00

(5t, 96 % ee)
 catalyst 3

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-126R
 User: System
 Acquired: 12/9/2006 1:29:22 PM
 Printed: 12/13/2006 10:34:24 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.883	10617446	50.05	877564	54.86
8.242	10595901	49.95	722132	45.14
Totals	21213347	100.00	1599696	100.00

(6, Racemic)

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\I-TM-142AD
 User: System
 Acquired: 12/13/2006 10:12:06 AM
 Printed: 12/13/2006 12:53:13 PM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.583	72861	1.31	6532	1.64
7.842	5498925	98.69	392765	98.36

Totals	5571786	100.00	399297	100.00
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(6, 97 % ee)