Supporting Information

Chiral binaphthylthiophosphoramidate-Cu(I) catalyzed asymmetric addition of diethylzinc to N-sulfonylimines

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w10-8-4c

Archive directory: /export/home/ASM/vnmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: szput

Solvent: CDCl₃

Ambient temperature

Mercury-300DB "QNP300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. 1.500 sec

Width 19011.4 Hz

92 repetitions

OBSERVE C13, 75.4421044 MHz

DECOUPLE H1, 300.0294698 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

FT size 32768

Total time 9 hr, 31 min, 48 sec

142.609
140.440
137.664
137.641
129.012
128.089
127.743
127.251
126.920
123.567

77.408
76.985
76.562

59.796

30.402

21.304
21.081

10.453

w10-8-2c
 Archive directory: /export/home/ASH/vnmr/sys/data
 Sample directory: /export/home/ASH/vnmr/sys/data
 File: CARBON
 Pulse Sequence: szpul
 Solvent: CDCl3
 Sample temperature: 40.0
 Mercury-500B8 "OMC300"
 Relax delay 1.000 sec
 P1 45.000000 sec
 Acq time 1.500 sec
 Width 19011.4 Hz
 92 Repetitions
 OBSERVE C13, 75.4421044 MHz
 DECOUPLE H1, 300.0294698 MHz
 Power 41 dB
 Continuity on
 Continuity off
 Data processing
 Line broadening 1.0 Hz
 FT size 65536
 Total time 9 hr, 31 min, 48 sec

155.552
 142.617
 137.695
 132.803
 129.104
 127.651
 126.928
 113.500
 77.423
 76.992
 76.569
 59.265
 55.082
 30.410
 21.320
 10.437

W10-14-2C

Archive directory: /export/home/wang-qx/vnmrSYS/data
Sample directory:
File: CARBON

Pulse sequence: zgpg30
Solvent: CDCl3
Acquisition temperature:
Mercury-3000B "QNP300"

Relax. delay 1.000 sec
Pulse delay 0.040000000000000000
Acq. time 1.720 sec
Width 19011.4 Hz
176 repetitions
OBSERVE C13, 75.4421038 MHz
DECOUPLE H1, 300.0294658 MHz
Power 36 dB
continuously on
WALTZ16 modulated
Data processing
Time broadening 1.0 Hz
F1 size 65536
Total time 10 hr, 23 min, 53 sec

143.063
142.717
137.264
133.911
129.496
129.189
127.089
126.851
126.828
124.774

77.423
77.000
76.577

59.296

30.287

21.335

10.360

W9-982-C

Archive directory: /export/home/wang-qx/vnmr sys/data
Sample directory:

Pulse Sequence: szpul

Solvent: CDCl3
Sample temperature
F1 frequency 101.625 MHz
Mercury-300BB "OMC300"

Relax delay 1.000 sec
Pulse 45.0 degrees
Acq time 1.700 sec
Width 19011.4 Hz
156 repetitions
OBSERVE C13, 75.4420992 MHz
DECUPLE H1, 300.0294698 MHz
Power 36 dB
Continuously on
101.625 MHz
Data processing
Line broadening 1.0 Hz
FT size 65536
Total time 10 hr, 23 min, 53 sec

143.186
139.664
137.372
131.273
129.266
128.381
126.951
121.014

77.423
77.000
76.577

59.227

30.325

21.435

10.360

W10-14-1-C

Archive directory: /export/home/wang-qx/vnmr/sys/data
Sample directory:

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: w10-14-1c-shi-C
Mercury-300BB "QMC300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 19011.4 Hz
520 repetitions
OBSERVE G13, 75.4420992 MHz
DECOUPLE D1, 300.0234636 MHz
Continuous on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10 hr, 23 min, 53 sec

W10-8-3C
 Archive directory: /export/home/ASM/vnmrSYS/data
 Sample directory: /export/home/ASM/vnmrSYS/data
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 Mercury-300B8 "OMC300"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.500 sec
 V1 pulse 1301.4 Hz
 V2 pulse 1301.4 Hz
 OBSERVE C13 on 75.4421015 MHz
 DECOUPLE H1, 300.0294638 MHz
 Power 41 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 F1 time 8.8558
 F2 time 1.0 Hz
 Total time 9 hr, 31 min, 48 sec

w10-11-2c

Archive directory: /export/home/wang-qx/vnmr/sys/da
Sample directory:
File: CARBON

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
Mercury-300B8 "OMC300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
VH1 100.625 MHz
128 repetitions
OBSERVE C13 75.4621060 MHz
DECUPLE H1 300.0294698 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
File broadening 2.0 Hz
F2 100.625000 MHz
Total time 11 hr, 41 min, 23 sec

W10-8-1c

Archive directory: /export/home/ASW/vnmrSYS/data
Sample directory:
File: CARBON

Pulse Sequence: szpul

Solvent: CDCl₃
Amplitude: 16.000 V
Frequency: 300.000 MHz

Relax: delay 1.000 sec

Acq: time 1.500 sec

Width 19011.4 Hz

100 repetitions

OBSERVE C13, 75.442097 MHz

DECOUPLE H1, 300.0294638 MHz

Power 41.48 W
SFO: 300.135 MHz
WALTZ16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 31 min, 48 sec

***** MODEL 1022 RUNLOG for run: CH-6__70 *****
Run terminated manually on Channel A.

Chiralcel OD

hexane/i-PrOH = 9/1

0.7 mL/min, 230 nm 1

File : CH-6__70.D01

w9-951 racemic

Run : 01

Path : C:\CH-5

Type : Sample
Inst : 1022 LC Plus

Collection : 13:47:10 Feb 11 2003 Method : LCTEST

[09:43:49 Feb 11 2003]

Integration: 13:47:10 Feb 11 2003 Method : LCTEST

[09:43:49 Feb 11 2003]

Report : 14:27:47 Feb 11 2003 Method : LCTEST

[09:43:49 Feb 11 2003]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	6.117	1380136	5.6799	T	0.3422	0.7464
2	7.377	332212	1.3591		0.0824	0.1786
3	10.443	71096	0.3733		0.0176	0.0491
4	20.043	200013616	419.7807		49.5943	55.1649
5	25.910	199216032	330.2724		49.3966	43.4023
6	39.057	2286272	3.4913		0.5669	0.4588

6 Peaks > Area Reject

403299360 Total Area

6 Peaks > Height Reject

760.957 Total Height

(CH-6__70.D01) mV

authentic racemic

***** MODEL 1022 RUNLOG for run: CH-6__71 *****

Run terminated manually on Channel A.

Chiralcel OD
hexane/i-PrOH = 9/1
0.7 mL/min, 230nm

File : CH-6__71.D01

w9-951

Run : 02

Type : Sample

Path : C:\CH-5

Inst : 1022 LC Plus

Collection : 14:29:48 Feb 11 2003

Method : LCTEST

[09:43:49 Feb 11 2003]

Integration: 14:29:48 Feb 11 2003

Method : LCTEST

[09:43:49 Feb 11 2003]

Report : 15:01:38 Feb 11 2003

Method : LCTEST

[09:43:49 Feb 11 2003]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.943	416301	2.4798		0.2296	0.7908
2	7.123	49707	0.2033		0.0274	0.0648
3	20.157	12341638	28.9753		6.8073	9.2399
4	23.623	270436	0.5979		0.1492	0.1907
5	25.743	168221296	281.3337		92.7865	89.7139

5 Peaks > Area Reject

181299376 Total Area

5 Peaks > Height Reject

313.590 Total Height

(CH-6__71.D01) mV

Software Version: 4.1<2F12>

Date: 02-11-27 13:29

Sample Name : W9-951

Data File : D:\TC4\DATA\CAO\CA0_338Y.RAW Date: 02-11-26 14:35

Sequence File: D:\TC4\DATA\CAO\CA0.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Dilution Factor : 1.00

Chiralpak AS

Hexane / i-PrOH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	20.342	110631.50	2048.33	6.91	0.00	BB	54.0105
2	27.346	1490827.50	17526.30	93.09	0.00	BB	85.0623
		1601459.00	19574.64	100.00	0.00		

Software Version: 4.1<2F12>

Date: 02-11-26 10:45

Sample Name : W-9-952RAC

Data File : D:\TC4\DATA\CAO\CA0_338U.RAW Date: 02-11-26 10:08

Sequence File: D:\TC4\DATA\CAO\CA0.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Dilution Factor : 1.00

Chiralpak AS

hexane / i-PrOH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	20.043	2272094.00	35343.61	50.50	0.00	BB	64.2859
2	25.742	2227480.75	25502.59	49.50	0.00	BB	87.3433
		4499574.75	60846.20	100.00	0.00		

Software Version: 4.1<2F12>

Date: 02-11-26 11:18

Sample Name : W9-952

Data File : D:\TC4\DATA\CAO\CA0_338V.RAW Date: 02-11-26 10:42

Sequence File: D:\TC4\DATA\CAO\CA0.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Dilution Factor : 1.00

Chiralpak AS

hexane/i-PrOH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	20.352	257907.50	4271.62	7.02	0.00	BB	60.3770
2	25.527	3413856.00	38817.08	92.98	0.00	BB	87.9473
		3671763.50	43088.69	100.00	0.00		

***** MODEL 1022 RUNLOG for run: CH-3_14_*****
 Run terminated manually on Channel A.

Chiralpak AD

hexane/i-PrOH = 85/15
 0.7 mL/min, 254 nm

File : CH-3_14.D01 W10-5A-RAC Type : Sample
 Run : 01
 Path : C:\CH-5 Inst : 1022 LC Plus
 Collection : 13:54:36 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]
 Integration: 13:54:36 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]
 Report : 14:18:43 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.077	116807	0.3587		0.2432	0.1721
2	11.677	23559244	108.4872	T	49.0441	52.0542
3	13.123	23666948	97.4627		49.2683	46.7644
4	17.717	693827	2.1034		1.4444	1.0092

4 Peaks > Area Reject 48036824 Total Area
 4 Peaks > Height Reject 208.412 Total Height

(CH-3_14.D01) mU

***** MODEL 1022 RUNLOG for run: CH-3_15 *****
 Run terminated manually on Channel A.

Chiralpak AD
hexane / i-PrOH = 95 / 5
0.7 ml/min, 254 nm

File : CH-3_15.D01 W10-5A
 Run : 01
 Path : C:\CH-5
 Collection : 14:22:38 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]
 Integration : 14:22:38 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]
 Report : 14:38:37 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	4.927	87934	0.3458		0.5421	0.5019
2	6.393	156785	1.0534		0.9665	1.5319
3	9.700	69417	0.2865		0.4279	0.4159
4	10.880	154973	0.8349	T	0.9553	1.2118
5	11.513	113727	5.3389		7.0106	7.7491
6	12.920	14615300	61.0355		90.0976	88.5894

6 Peaks > Area Reject 16221636 Total Area
 6 Peaks > Height Reject 68.897 Total Height

(CH-3_15.D01) AU
 from addition

Software Version: 4.1<2F12>

Date: 02-12-4 16:07

Sample Name : yzq-13-49rac W10-1A-RAL

Data File : D:\TC4\DATA\CAO\CAP_001N.RAW Date: 02-12-4 12:17

Sequence File: D:\TC4\DATA\CAO\CAP.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Chiralpak AS

hexane/i-PrOH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	30.121	1329989.00	12068.40	49.70	0.00	BB	110.2043
2	43.611	1346011.00	8953.73	50.30	0.00	BB	150.3297
		2676000.00	21022.12	100.00	0.00		

Software Version: 4.1<2F12>

Date: 02-12-4 16:12

Sample Name : w10-1a

Data File : D:\TC4\DATA\CAO\CAP_0010.RAW Date: 02-12-4 13:35

Sequence File: D:\TC4\DATA\CAO\CAP.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Chiralpak AS

hexane/i-PrOH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	14.424	274829.00	4493.46	2.42	0.00	BB	61.1620
2	31.502	601913.75	5262.09	5.30	0.00	BB	114.3867
3	43.851	10483064.00	56357.31	92.28	0.00	BB	186.0107
		11359806.75	66112.86	100.00	0.00		

Software Version: 4.1<2F12>

Date: 02-11-26 14:31

Sample Name : W9-933RAC

Data File : D:\TC4\DATA\CAO\CA0_338W.RAW Date: 02-11-26 13:16

Sequence File: D:\TC4\DATA\CAO\CA0_SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Dilution Factor : 1.00

Chiralpak AS

hexane/i-PrOH = 75/25

0.7 mL/min, 254 nm

authentic racemic

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	16.752	2538281.50	51339.81	49.15	0.00	BB	49.4408
2	26.764	2626109.00	27893.66	50.85	0.00	BB	94.1472
		5164390.50	79233.47	100.00	0.00		

Software Version: 4.1<2F12>

Date: 02-11-26 14:38

Sample Name : W9-933

Data File : D:\TC4\DATA\CAO\CA0_338X.RAW Date: 02-11-26 13:52

Sequence File: D:\TC4\DATA\CAO\CA0.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Dilution Factor : 1.00

Chiralpak AS

hexane/i-PrOH = 75 / 25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	5.399	106270.00	5277.37	1.92	0.00	BB	20.1369
2	14.110	336074.50	6376.16	6.07	0.00	BB	52.7080
3	17.033	233680.50	4973.39	4.22	0.00	BB	46.9861
4	26.448	4860477.75	48695.98	87.79	0.00	BB	99.8127
		5536502.75	65322.90	100.00	0.00

Chiralpak AD
Hexane/i-PrOH = 85/15
0.7 mL/min, 230 nm

Run terminated manually on Channel A.

File : CH-3_50.D01
Run : 01
Path : C:\CH-5
Collection : 14:16:57 Dec 10 2002 Method : LCTEST
Integration: 14:16:57 Dec 10 2002 Method : LCTEST
Report : 14:41:21 Dec 10 2002 Method : LCTEST
Type : Sample
Inst : 1022 LC Plus
[08:11:29 Dec 10 2002]
[08:11:29 Dec 10 2002]
[08:11:29 Dec 10 2002]

PERCENT (AREA)				
Pk #	RT	Area	Height	Area Percent
1	5.810	211940	0.6220	0.2539
2	11.683	103425	0.5286	0.1239
3	13.890	4150288	160.4795	49.7122
4	15.463	41515904	145.0696	49.7278
5	21.597	152121	0.3510	0.1822

5 Peaks > Area Reject 83486280 Total Area
5 Peaks > Height Reject 307.051 Total Height

64

(CH-3_50.D01) .n

authentic racemic

***** MODEL 1022 RUNLOG for run: CH-3_51 ***** NT3
 Run terminated manually on Channel A.

Chiralpak AD
 hexane / i-PrOH = 85/15
 0.7 mL/min, 230 nm

File : CH-3_51.D01 W110-13B Type : Sample
 Run : 01
 Path : C:\CH-5
 Collection : 14:44:28 Dec 10 2002 Method : LCTEST Inst : 1022 LC Plus
 Integration: 14:44:28 Dec 10 2002 Method : LCTEST [08:11:29 Dec 10 2002]
 Report : 15:03:30 Dec 10 2002 Method : LCTEST [08:11:29 Dec 10 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.460	0	0.1918		0.0000	0.0552
2	9.153	67441	0.3545		0.0673	0.1020
3	11.627	83135	0.4170		0.0830	0.1200
4	13.880	6818160	26.1551	T	6.8043	7.5244
5	15.387	93234224	320.4873		93.0454	92.1985

5 Peaks > Area Reject 100202960 Total Area
 5 Peaks > Height Reject 347.606 Total Height

**** MODEL 1022 RUNLOG for run: CH-3_71 ****

Run terminated manually on Channel A.

Chiralcel OD

hexane/i-PrOH = 9/1

0.7 mL/min, 254 nm

File : CH-3_71.D01

w10-15-rac

Run : 01

Type : Sample

Path : C:\CH-5

Inst : 1022 LC Plus

Collection : 08:38:57 Dec 12 2002 Method : LCTEST

[08:38:42 Dec 12 2002]

Integration: 08:38:57 Dec 12 2002 Method : LCTEST

[08:38:42 Dec 12 2002]

Report : 09:11:48 Dec 12 2002 Method : LCTEST

[08:38:42 Dec 12 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.677	61987	0.7343	T	0.0999	0.7263
2	6.077	140497	1.0228		0.2265	1.0116
3	9.457	90761	0.4342		0.1463	0.4294
4	11.103	162154	0.6148		0.2615	0.6081
5	16.450	292650	0.7447		0.4719	0.7365
6	20.783	30572312	54.7441		49.2937	54.1471
7	26.797	30700360	42.8077		49.5002	42.3408

7 Peaks > Area Reject

62020724 Total Area

7 Peaks > Height Reject

101.103 Total Height

(CH-3_71.D01) mV

authentic racemic

**** MODEL 1022 RUNLOG for run: CH-3_73 *****
 Run terminated manually on Channel A.

Chiral OD
 hexane/i-PrOH = 9/1
 0.7 mL/min, 254 nm

File : CH-3_73.D01 w10-15a
 Run : 01
 Path : C:\CH-5
 Collection : 09:53:20 Dec 12 2002 Method : LCTEST
 Integration: 09:53:20 Dec 12 2002 Method : LCTEST
 Report : 10:22:31 Dec 12 2002 Method : LCTEST
 Type : Sample
 Inst : 1022 LC Plus
 [09:15:28 Dec 12 2002]
 [09:15:28 Dec 12 2002]
 [09:15:28 Dec 12 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	6.103	99546	0.4303		0.1651	0.4857
2	20.350	2234366	4.9972		3.7048	5.6404
3	25.123	57975600	83.1695		96.1301	93.8739

3 Peaks > Area Reject 60309512 Total Area
 3 Peaks > Height Reject 88.597 Total Height

(CH-3_73.D01) mV

from addition

Software Version: 4.1<2F12>

Date: 02-11-28 15:33

Sample Name : w9-931rac

Data File : D:\TC4\DATA\CAO\CA0_339K.RAW Date: 02-11-28 13:30

Sequence File: D:\TC4\DATA\CAO\CA0.SEQ Cycle: 1 Channel : A

Instrument : 970A_-0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Dilution Factor : 1.00

Chiralpak AS

hexane/i-PrOH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	18.284	1216517.25	20973.62	50.02	0.00	BB	58.0022
2	29.415	1215321.50	11209.87	49.98	0.00	BB	108.4153
		2431838.75	32183.49	100.00	0.00		

Software Version: 4.1<2F12>

Date: 02-11-28 15:27

Sample Name : w9-932

Data File : D:\TC4\DATA\CAO\CA0_339M.RAW Date: 02-11-28 14:48

Sequence File: D:\TC4\DATA\CAO\CA0.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000 Dilution Factor : 1.00

Chiralpak AS

hexane / i-ProH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	18.435	96434.00	1823.29	3.98	0.00	BB	52.8901
2	29.050	2324879.50	21518.87	96.02	0.00	BB	108.0391
		2421313.50	23342.16	100.00	0.00		

***** MODEL 1022 RUNLOG for run: CH-3__76 *****

Run terminated manually on Channel A.

Chiralcel OD

hexane/i-PrOH = 9/1

0.7 mL/min, 254 nm

File : CH-3__76.D01

w10-13a-rac

Run : 01

Path : C:\CH-5

Type : Sample

Inst : 1022 LC Plus

Collection : 11:03:05 Dec 12 2002 Method : LCTEST

[09:15:28 Dec 12 2002]

Integration : 11:03:05 Dec 12 2002 Method : LCTEST

[09:15:28 Dec 12 2002]

Report : 11:34:52 Dec 12 2002 Method : LCTEST

[09:15:28 Dec 12 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.697	75079	0.7103	T	0.0761	0.4340
2	6.090	163287	0.7709		0.1656	0.4710
3	19.270	48752856	93.5623		49.4442	57.1611
4	25.310	573697	1.1365		0.5818	0.6944
5	27.683	49036764	67.5018		49.7322	41.2396

5 Peaks > Area Reject

98601680 Total Area

5 Peaks > Height Reject

163.682 Total Height

(CH-3__76.D01)

UV

**** MODEL 1022 RUNLOG for run: CH-3_77 ****

Run terminated manually on Channel A.

Chemical OD

hexane / i-ProH = 9/1

0.7 mL / min, 254 nm 1

File : CH-3_77 w10-13a

Run : 01

Path : C:\CH-3_77

Type : Sample

Inst : 1022 LC Plus

Collection : 11/28/2002 Dec 12 2002 Method : LCTEST [09:15:28 Dec 12 2002]

Integration : 11/28/2002 Dec 12 2002 Method : LCTEST [09:15:28 Dec 12 2002]

Report : 12:28:06 Dec 12 2002 Method : LCTEST [09:15:28 Dec 12 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	19.467	6408061	13.6888		7.5088	11.6781
2	27.200	78932176	103.5294		92.4912	88.3219

2 Peaks > Area Reject 85340240 Total Area
2 Peaks > Height Reject 117.218 Total Height

(CH-3_77.D81) .nU

from addition

***** MODEL 1022 RUNLOG for run: CH-9_44 *****

Run terminated manually on Channel A.

Chiralpak AD
hexane/i-PrOH = 97/3
1 ml/min, 254 nm

File : CH-9_44.D01

W9-9-28A-RAC

Run : 01

Path : C:\CH-4

Type : Sample

Inst : 1022 LC Plus

Collection : 10:03:11 Oct 08 2002 Method : LCTEST [08:53:52 Oct 08 2002]

Integration: 10:03:11 Oct 08 2002 Method : LCTEST [08:53:52 Oct 08 2002]

Report : 11:05:52 Oct 08 2002 Method : LCTEST [08:53:52 Oct 08 2002]

PERCENT (AREA)

PK #	RT	Area	Height	BC	Area Percent	Height Percent
1	3.923	816037	6.6189		0.5706	4.5548
2	5.343	254874	0.8902		0.1782	0.6126
3	6.283	18978	0.2675		0.0133	0.1841
4	8.290	39898	0.0826		0.0279	0.0568
5	42.083	403680	0.2999	T	0.2823	0.2064
6	45.523	70712496	78.1314	T	49.4476	53.7666
7	50.777	70758832	59.0254		49.4800	40.6187

***** MODEL 1022 RUNLOG for run: CH-2__12 *****

Run terminated manually on Channel A.

Chiralpak AD
hexane/i-PrOH = 97/3
1 mL/min, 254 nm

File : CH-2__12.D01

W9-86

Run : 01

Path : C:\CH-5

Collection : 12:31:08 Nov 22 2002 Method : LCTEST [10:19:12 Nov 22 2002]

Integration: 12:31:08 Nov 22 2002 Method : LCTEST [10:19:12 Nov 22 2002]

Report : 13:31:42 Nov 22 2002 Method : LCTEST [10:19:12 Nov 22 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	3.913	465743	5.4774	V	0.9179	9.1950
2	4.383	13974	0.1379		0.0275	0.2315
3	6.480	89728	0.6520		0.1768	1.0946
4	9.420	89803	0.4279		0.1770	0.7182
5	46.880	47462960	50.4372	T	93.5423	84.6695
6	52.800	2617356	2.4371		5.1584	4.0912

File : CH-3_01.D01
 Run : 01
 Path : C:\CH-5
 Collection : 14:41:12 Dec 03 2002 Method : LCTEST
 Integration: 16:41:43 Dec 04 2002 Method : LCTEST
 Report : 16:41:46 Dec 04 2002 Method : LCTEST

W10-1B-RAC
 Chiralpak AD
 Hexane/i-ProH = 80/20
 0.7ml/min, 254 nm
 Type : Sample
 Inst : 1022 LC Plus
 [14:18:58 Dec 03 2002]
 [08:35:00 Dec 04 2002]
 [08:35:00 Dec 04 2002]

PERCENT (AREA)				
PK #	RT	Area	Height	Percent
1	5.850	612770	2.6894	1.5051
2	7.403	216953	1.3939	0.5329
3	8.890	46950	0.2433	0.1153
4	10.937	81943	0.3941	0.2013
5	11.897	96870	0.5668 T	0.2379
6	12.643	19771324	85.6971 T	48.5640
7	13.817	19883110	75.1111	48.8435

7 Peaks > Area Reject 40711920 Total Area
 7 Peaks > Height Reject 166.096 Total Height

File : CH-3_02.D01
 Run : 01
 Path : C:\CH-5
 Collection : 15:02:17 Dec 03 2002 Method : LCTEST
 Integration: 16:41:52 Dec 04 2002 Method : LCTEST
 Report : 16:41:58 Dec 04 2002 Method : LCTEST
 Chiralpak AD
 Hexane / i-PrOH = 80/20
 0.7mL/min type: Sample
 Inst : 1022 LC Plus
 [14:18:58 Dec 03 2002]
 [08:35:00 Dec 04 2002]
 [08:35:00 Dec 04 2002]

Pk #	RT	Area	PERCENT (AREA)		
			Height	BC	Area Percent
1	5.843	250544	0.7459		0.3931
2	11.183	0	0.0923		0.0000
3	12.797	57663008	244.4938	T	90.4838
4	14.077	5813895	21.6415		9.1231

4 Peaks > Area Reject 63727448 Total Area
 4 Peaks > Height Reject 266.973 Total Height

(CH-3_02.D01) mV
 from addition

***** MODEL 1022 RUNLOG for run: CH-3_16 *****
 Run terminated manually on Channel A.

NT's
 Chiralpak AD
 hexane / i-PrOH = 85/15
 0.7 mL/min, 254 nm

File : CH-3_16.D01
 Run : 01
 Path : C:\CH-5
 Collection : 14:39:42 Dec 04 2002 Method : LCTEST Inst : 1022 LC Plus Type : Sample
 Integration: 14:39:42 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]
 Report : 15:04:54 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]

PERCENT (AREA)				
Pk #	RT	Area	Height	BC
1	5.217	80872	0.2149	0.4879
2	15.557	8266256	27.9033	49.8709
3	19.117	8228187	22.9213	49.6412
3 Peaks > Area Reject 16575315 Total Area				
3 Peaks > Height Reject 51.040 Total Height				

(CH-3_16.D01) mV

authentic racemic

***** MODEL 1022 RUNLOG for run: CH-3__17 *****

Run terminated manually on Channel A.

Chiralpak AD
hexane/i-PrOH = 85/15
0.7 mL/min, 254 nm

File : CH-3__17.D01

W10-9B

Run : 01

Path : C:\CH-5

Type : Sample

Inst : 1022 LC Plus

Collection : 15:06:25 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]

Integration : 15:06:25 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]

Report : 15:46:23 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.267	207320	0.4335		0.1781	0.1343
2	9.913	103183	0.5388		0.0886	0.1669
3	11.120	157276	0.8067		0.1351	0.2500
4	15.647	6715589	22.0646		5.7688	6.8366
5	19.247	106166936	292.8971	T	91.1983	90.7529
6	21.687	263846	0.5697		0.2266	0.1765
7	26.687	2799072	5.4310		2.4044	1.6828

7 Peaks > Area Reject

116413224 Total Area

7 Peaks > Height Reject

322.741 Total Height

(CH-3__17.D01) AU

Software Version: 4.1<2F12>

Date: 02-12-20 16:05

Sample Name : w-rac

Data File : D:\TC4\DATA\CAO\CAP_005V.RAW Date: 02-12-20 15:41

Sequence File: D:\TC4\DATA\CAO\CAP.SEQ Cycle: 1 Channel : A

Instrument : 970A _ 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Chiralpak AS

hexane/i-PrOH = 75/25

0.7 mL/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	13.435	446707.76	13345.75	50.25	0.00	BV	33.4719
2	15.104	442319.24	9353.63	49.75	0.00	VB	47.2885
		889027.00	22699.38	100.00	0.00		

Software Version: 4.1<2F12>

Date: 02-12-20 16:30

Sample Name : w10-28

Data File : D:\TC4\DATA\CAO\CAP_005W.RAW Date: 02-12-20 16:02

Sequence File: D:\TC4\DATA\CAO\CAP.SEQ Cycle: 1 Channel : A

Instrument : 970A - 0 Rack/Vial: 0/0 Operator: CAO

Sample Amount : 1.0000

Chiralpak AS

hexane/i-PrOH = 75/25

0.7 ml/min, 254 nm

REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Area BL	Area/Height [sec]
1	13.468	131854.09	4402.00	6.79	0.00	BV	29.9532
2	14.648	1809451.41	31552.73	93.21	0.00	VB	57.3469
		1941305.50	35954.73	100.00	0.00

Chiralpak AD

Reserve / λ -P-OH = 80/20
 0.7 ml/min, 230 nm

File : CH-3_03.D01
 Run : 01
 Path : C:\CH-5
 Collection : 15:47:57 Dec 03 2002 Method : LCTEST Type : Sample
 Integration: 16:42:02 Dec 04 2002 Method : LCTEST [15:29:11 Dec 03 2002] Inst : 1022 LC Plus
 Report : 16:42:04 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002] [08:35:00 Dec 04 2002]

W10-5B-RAC

PERCENT (AREA)

PK #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.683	684147	1.5910		1.6822	0.7382
2	7.943	371116	1.1011	T	0.9125	0.5109
3	9.090	19702480	109.1316	T	48.4456	50.6330
4	9.963	19840648	103.4291		48.7854	47.9872
5	11.990	70870	0.2819		0.1743	0.1308

5 Peaks > Area Reject 40669260 Total Area
 5 Peaks > Height Reject 215.535 Total Height

(CH-3_03.D01) AU

authentic racemic

Chiralpak AD
hexane/i-PrOH = 80/20
0.7 mL/min, 230 nm

File : CH-3_05.D01
Run : 02
Path : C:\CH-5
Collection : 16:20:44 Dec 03 2002
Integration: 16:42:12 Dec 04 2002
Report : 16:42:17 Dec 04 2002

Method : LCTEST
Method : LCTEST
Method : LCTEST

Inst : 1022 LC Plus
[15:29:11 Dec 03 2002]
[08:35:00 Dec 04 2002]
[08:35:00 Dec 04 2002]

Type : Sample
1

W10-5B

PERCENT (AREA)				
Pk #	RT	Area	Height	BC
1	5.630	372406	2.1021	T
2	6.117	248640	1.0345	T
3	7.997	2730297	13.3745	T
4	9.210	3964477	22.9658	T
5	10.090	3545360	178.4330	T

5 Peaks > Area Reject 42769180 Total Area
5 Peaks > Height Reject 217.910 Total Height

***** MODEL 1022 RUNLOG for run: CH-3_10 *****
 Run terminated manually on Channel A.

Chiralpak AD
 Hexane/i-PrOH = 85/15
 0.7 mL/min, 230 nm

File : CH-3_10.D01 W10-9A-RAC 1
 Run : 01 Type : Sample
 Path : C:\CH-5 Inst : 1022 LC Plus
 Collection : 11:12:13 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]
 Integration: 11:12:13 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]
 Report : 11:32:55 Dec 04 2002 Method : LCTEST [08:35:00 Dec 04 2002]

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	5.813	639180	1.5863		0.3474	0.2155
2	9.813	134969	0.5650	T	0.0733	0.0767
3	10.560	324038	1.2859	T	0.1761	0.1746
4	11.633	91087272	403.4614	T	49.5011	54.7990
5	13.320	91090040	327.0505		49.5026	44.4207
6	17.060	734970	2.3075		0.3994	0.3134

6 Peaks > Area Reject 184010464 Total Area
 6 Peaks > Height Reject 736.257 Total Height

***** MODEL 1022 RUNLOG for run: CH-3_13 *****

Run terminated manually on Channel A.

Chiralpak AD
hexane/i-PrOH = 85/15
0.7 mL/min, 230 nm
Inst: 1022 LC Plus
Type: Sample
[08:35:00 Dec 04 2002]
[08:35:00 Dec 04 2002]
[08:35:00 Dec 04 2002]

File: CH-3_13.D01 W-10-9A

Run: 03

Path: C:\CH-5

Collection: 13:33:14 Dec 04 2002 Method: LCTEST

Integration: 13:33:14 Dec 04 2002 Method: LCTEST

Report: 13:53:29 Dec 04 2002 Method: LCTEST

PERCENT (AREA)

Pk #	RT	Area	Height	BC	Area Percent	Height Percent
1	6.423	95814	0.2856		0.3683	0.2068
2	10.017	57860	0.3705		0.2224	0.2682
3	11.683	21067984	116.6244		80.9763	84.4395
4	13.250	4795806	20.8355		18.4330	15.0855

4 Peaks > Area Reject

4 Peaks > Height Reject

26017464 Total Area

138.116 Total Height

(CH-3_13.D01) mV