Asymmetric Synthesis of *trans*-2,5- Disubstituted Pyrrolidines from Enantiopure Homoallylic Amines. Synthesis of Pyrrolidine (-)-197B

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Supporting Information Available

Contents:

^1H and ^{13}C spectra for compounds **1** to **22** and for the *N*-benzoyl derivative of **22**.

General Procedures. All reagents were used as received unless otherwise noted. Tetrahydrofuran (THF), diethyl ether, dichloromethane, and toluene were purified by filtration on a solvent purification system. Column chromatography was performed on silica gel, grade 60 (230–400 mesh). TLC plates were visualized with UV, in an iodine chamber, or with phosphomolybdic acid, unless otherwise noted. ^1H NMR and ^{13}C NMR spectra were recorded on 400 and 300 MHz NMR spectrometers.

Archive directory: /home/temple/vnmrsys/data
Sample directory: minsoo/MS-I-119A_10Feb2005
File: PROTON

Solvent: CDC13
Ambient temperature
INOVA-300 "nmr300"

Pulse 45.0 degrees

Acq. time 2.500 sec
Width 4799.6 Hz

8 repetitions

BSERVE H1, 29

ATA PROCESSING

Resol. enhanced
Gauss. anodizat.

T size 32768

total time 0 min

(*S*, 4*R*)-(+)-*N*-(*p*-Toluenesulfinyl)-4-amino-1,4-diphenylbut-1-en (3).

MS-I-119Acarbon

Archive directory: /home/temple/vnmrSYS/data
Sample directory: minsoo/MS-I-119Acarbon_10Feb2005
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
INNOVA-300 "nmr300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18859.0 Hz

256 repetitions

OBSERVE C13, 75.4311280 MHz

DECOUPLE H1, 299.9858744 MHz

Power 38 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 12 min, 2 sec

(*S*,4*R*)-(+)-*N*-(*p*-Toluenesulfinyl)-4-amino-1,4-diphenylbut-1-en (3).

(R)-(+)-N-(1,4-Diphenylbut-3-enyl)-4-methylbenzenesulfonamide (4).

Current Data Parameters
 NAME minsoo
 EXPNO 150
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050212
 Time 11.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 143.7
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.80 usec
 PL1 0.00 dB
 SFO1 400.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0200326 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 ppm
 F1 4400.22 Hz
 F2P -1.000 ppm
 F2 -400.02 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 240.01201 Hz/cm

F2 - Acquisition Parameters

Year	Population (millions)
1995	124.837
1996	126.614
1997	126.880
1998	126.979
1999	127.376
2000	127.462
2001	127.504
2002	127.826
2003	127.907
2004	128.660
2005	128.852
2006	129.043
2007	129.698
2008	129.763
2009	132.724
2010	134.446
2011	137.133
2012	140.994
2013	143.471

Date_Time		INSTRUM		PULPROG		TD		SOLVENT		NS		DS		SWH		FIDRES		AQ		RG		DW		DE		TE		D1		d11		d12	
20050212	12.09	spec	5 mm PABBO BB-	zpgpg60	65536	CDCl3	400	2	25125.629	Hz	1.3843387	Hz	1.3042164	sec	18390.4	19.900	usec	6.00	usec	300.0	K	2.00000000	sec	0.03000000	sec	0.00002000	sec						

```
===== CHANNEL f1 =====
NUC1      13C
P1        5.70 usec
PL1       2.00 dB
SF01      100.5951275 MHz
```

```
===== CHANNEL F2 =====
waltz16
1H
92.00 usec
0.00 dB
PL2
11.00 dB
PL12
120.00 dB
PL13
400.0216001 MHz
SF02
```

F2 - Processing parameters	
SI	32768
SF	100.5850690 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters	
CX	22.00 cm
CY	6.00 cm
F1P	210.000 ppm
F1	21122.86 Hz
F2P	-10.000 ppm
F2	-1005.85 Hz
PPMCM	10.00000 ppm/cm
HZCM	1005.85071 Hz/cm

(2*S*,3*R*,5*R*)-(-)-3-Iodo-2,5-diphenyl-1-tosylpyrrolidine (5a).

56

alex/aa60p_01Jun2005	USER: -- DATE: Jun 1 2005		PTS1d: 8192	W/mNuts - \$PROTON.fid	
F1: 299.986	F2: 299.986	SW1: 3000	OF1: 1308.4	LB: 0.0	
EX: s2pul		PW: 5.8 usec	PD: 1.0 sec	NA: 16

(2*S*,3*R*,5*R*)-(-)-3-Iodo-2,5-diphenyl-1-tosylpyrrolidine (5a).

58

(*R,Z*)-(+)-*N*-(1,4-Diphenylbut-3-enyl)-4-methylbenzenesulfonamide (6a).

alex/aa34zstrange_31Mar2005		USER: -- DATE: Mar 31 2005	
F1: 299.988	F2: 299.987	PTS1d: 16384	W/mNuts - \$PROTON.fid
EX: s2pul		OF1: 1659.4	LB: 0.0
		NA: 32	
		PD: 1.0 sec	
		PW: 5.8 usec	
		SW1: 4800

(R,Z)-(+)-N-(1,4-Diphenylbut-3-enyl)-4-methylbenzenesulfonamide (6a).

Current Data Parameters
NAME alex
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20051129
Time 17.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg60
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 322.5
DW 21.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 5.70 usec
PL1 2.00 dB
SFO1 100.6387191 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 11.00 dB
PL13 120.00 dB
SFO2 400.1927165 MHz

F2 - Processing parameters

SI 32768
SF 100.6280960 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 22.00 cm
CY 6.00 cm
F1 220.000 ppm
F1 22138.18 Hz
F2P -10.000 ppm
F2 -1006.28 Hz
PPMCM 10.45455 ppm/cm
HZCM 1052.02112 Hz/cm

(2S,3R,5R)-(+)-2-Ethyl-3-iodo-5-phenyl-1-tosylpyrrolidine (5b).

Current Data Parameters
 NAME minsoo
 EXPNO 194
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050420
 Time 9.34
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 161.3
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.80 usec
 PL1 0.00 dB
 SF01 400.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0200291 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 ppm
 F1 4400.22 Hz
 F2P -1.000 ppm
 F2 -400.02 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 240.01201 Hz/cm

015

MS-I-166A
carbon30MIN.atm

Current Data Parameters
NAME minsoo
EXPNO 195
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050420
Time 9.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 400
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 18390.4
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.70 usec
PL1 2.00 dB
SF01 100.5951275 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 11.00 dB
PL13 120.00 dB
SF02 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 6.00 cm
F1P 210.000 ppm
F1 21122.86 Hz
F2P -10.000 ppm
F2 -1005.85 Hz
PPMCM 10.00000 ppm/cm
HZCM 1005.85071 Hz/cm

142.694
139.218

115

(2S,3R,5S)-(+)-2-Ethyl-3-iodo-5-methyl-1-tosylpyrrolidine (5c).

F1: 500.130		F2: 500.130		SW1: 6006		OF1: 2500.6		PTS1d: 16384		USER: -- DATE: Fri Dec 1 13:24:19	
EX: 1 puls		PW: 8.0 usec		PD: 1.0 sec		NA: 60		LB: 0.0		WinNuts - \$expMS-I-80B	

512

513

(2S,3R,5S)-(+)-2-Ethyl-3-iodo-5-methyl-1-tosylpyrrolidine (5c).

F1: 125.758		F2: 125.758	SW1: 33333	PD: 1.0 sec	OF1: 13204.5	PTSId: 16384	USER: -- DATE: Fri Dec 1 13:30:55	
EX: 1puls			PW: 8.0 usec		NA: 3000		WinNuts - \$expMS-I-80Bcarbon	
					LB: 0.0

(*R,Z*)-(+)-4-methyl-*N*-(1-phenylhex-3-enyl)benzenesulfonamide (6b).

(S,Z)-(-)-N-(Hept-4-en-2-yl)-4-methylbenzenesulfonamide (6c).

516

F1: 500.130	F2: 500.130	SW1: 6006	PD: 1.0 sec	NA: 16	OF1: 2500.6	PTSId: 16384	USER: -- DATE: Fri Dec 11 13:16:02
EX: 1puls		PW: 8.0 usec					WinNuts - \$expMS-I-80SM

517

(S,Z)-(-)-N-(Hept-4-en-2-yl)-4-methylbenzenesulfonamide (6c).

F1: 125.758	F2: 125.758	SW1: 33333	OF1: 13204.5	PTSId: 16384	USER: -- DATE: Tue Dec 5 07:26:27
EX: 1puls		PW: 8.0 usec	PD: 1.0 sec	LB: 0.0	WinNuts - \$expMS-182SMcarbon

alex/aa67_13Sep2005

Archive directory: /home/temple/vnmrSYS/data
Sample directory: alex/aa67_13Sep2005
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
INNOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 2999.9 Hz

32 repetitions

OBSERVE H1, 299.9843628 MHz

DATA PROCESSING

Resol. enhancement 0.4 Hz

Gauss apodization 0.240 sec

FT size 16384

Total time 1 min, 59 sec

alex/aa67c_12Aug2005

USER: -- DATE: Aug 12 2005

F1: 75.438

F2: 299.986

SW1: 15842

PW: 5.0 usec

PD: 1.0 sec

OF1: 7166.0

NA: 256

PTS1d: 32768

LB: 0.0

EX: s2pul

WinNuts - \$CARBON.fid

alex/aa69_02Aug2005

USER: -- DATE: Aug 2 2005

F1: 299.986

SW1: 3600

OF1: 1500.0

PTS1d: 16384

EX: s2pul

PW: 5.8 usec

PD: 2.0 sec

NA: 8

LB: 0.0

WinNuts - \$PROTON.fid

MS-I-234A

Archive directory: /home/temple/vnmrsys/data
Sample directory: minsoo/MS-I-234A_12Aug2005
File: PROTON

Pulse Sequence: szpul

Solvent: CDCl3
Ambient temperature
INNOVA-300 "nmr-300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 4799.6 Hz

32 repetitions

OBSERVE H1 299.9843628 MHz

DATA PROCESSING

Resol. enhancement 0.4 Hz

Gauss apodization 0.240 sec

FT size 32768

Total time 1 min, 59 sec

(S)-(-)-4-Methyl N-(4-oxobutan-2-yl)benzenesulfonamide (9b).

MS-I-234Acarbon

Archive directory: /home/temple/vnmrsys/data
Sample directory: minsoo/MS-I-234Acarbon_12Aug2005
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
INNOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec

Width 18859.0 Hz
64 repetitions

OBSERVE C13, 75.4311280 MHz
DECOUPLE H1, 299.9858744 MHz

Power 38 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 48 min, 11 sec

(S)-(-)-4-Methyl N-(4-oxobutan-2-yl)benzenesulfonamide (9b).

523

MS-I-165A

Archive directory: /home/temple/vnmr/sys/data
Sample directory: minsoo/MS-I-165A_16Apr2005
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
INOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 4799.6 Hz
8 repetitions

OBSERVE H1, 299.9843628 MHz

DATA PROCESSING

Resol. enhancement 0.4 Hz

Gauss apodization 0.240 sec

FT size 32768

Total time 0 min, 35 sec

(R)-(+)-4-Methyl N-(1-phenylhex-3-enyl)benzenesulfonamide (10).

143.384
141.185
140.907
137.909
137.645
136.164
129.661
128.671
128.635
127.685
127.528
126.983
126.952
123.658
123.246
77.708
77.393
77.072
58.106
57.778
41.178
35.568
25.865
21.792
20.998
14.379
13.898

(R)-(+)-4-Methyl N-(1-phenylhex-3-enyl)benzenesulfonamide (10).

MS-I-165A, carbon30MIN.atm.	USER: temple -- DATE: Apr 16 10:57:08 2005 EDT (UT-4h) templ
F1: 100.585	PTSId: 32768
F2: 1.000	WinNuts - \$Minsoo.1
EX:	File: 193
	LB: 3.0
	PD: 2.0 sec
	NA: 400
	OF1: 10057.2
	SW1: 25126
	PW: 5.7 usec

MS-7-165A

MS-I-88B
proton.atm

Current Data Parameters

NAME	minsoo
EXPNO	55
PROCNO	1

F2 - Acquisition Parameters

Date_	20041215
Time	11.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	32
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

===== CHANNEL f1 =====

NUC1	¹ H
P1	9.80 usec
PL1	0.00 dB
SFO1	400.0220001 MHz

F2 - Processing parameters

SI	32768
SF	400.0200248 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
FIP	11.000 ppm
F1	4400.22 Hz
F2p	-1.000 ppm
F2	-400.02 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.01201 Hz/cm

MS-I-88Bcarbon
carbon30MIN.atm

Current Data Parameters
NAME minsoo
EXPNO 56
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041215
Time 12.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg60
TD 65536
SOLVENT CDCl3
NS 400
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.70 usec
PL1 2.00 dB
SFO1 100.5951275 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 11.00 dB
PL13 120.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 6.00 cm
FIP 210.000 ppm
F1 21122.86 Hz
F2 -10.000 ppm
F2 -1005.85 Hz
PPMCM 10.00000 ppm/cm
HZCM 1005.85071 Hz/cm

(S)-(-)-N-(hept-4-en-2-yl)-4-methylbenzenesulfonamide (11).

143.517

ppm

225

(2R,5R)-(+)-2,5-Diphenyl-1-tosylpyrrolidine (12).

528

F1: 500.130	F2: 500.130	SW1: 12048	PD: 1.0 sec	OF1: 2500.6	LB: 0.0	PTS1d: 16384	USER: -- DATE: Fri Jun 22 05:30:28
EX: 1puls		PW: 8.0 usec		NA: 32			WinNuts - \$exaa72

(2R,5R)-(+)-2,5-Diphenyl-1-tosylpyrrolidine (12).

529

140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140		120		100		80		60		40		20		0		140	
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(2R,5R)-(+)-2,5-Diphenylpyrrolidine (13).

530

alex/aa73p3_01Jul2005	USER: -- DATE: Jul 1 2005
F1: 299.986	PTSId: 8192
F2: 299.986	OF1: 1305.1
EX: s2pul	NA: 32
	PD: 2.0 sec
	SW1: 3000
	PW: 5.8 usec
	LB: 0.0
	WinNuts - \$PROTON.fid

(2R,5R)-(+)-2,5-Diphenylpyrrolidine (13).

alex/aa89c13_22Sep2005				USER: -- DATE: Sep 22 2005	
F1: 75.438	F2: 299.986	SW1: 15842	OF1: 7166.0	PTSId: 32768	WmNuts - \$CARBON.fid
EX: s2pul		PW: 5.0 usec	PD: 1.0 sec	NA: 7424	LB: 3.0

531

MS-I-233A

Archive directory: /home/temple/vnmrSYS/data
Sample directory: minsoo/MS-I-233A_24Aug2005
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
INOVA-300 "nmr300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 4789.6 Hz

8 repetitions

OBSERVE H1, 299.3843628 MHz

DATA PROCESSING

Resol. enhancement 0.4 Hz

Gauss apodization 0.240 sec

FI size 32768

Total time 0 min, 35 sec

(R)-(-)-N-(Pentenylidene)-p-toluenesulfonamide (14).

532

(R)-(-)-N-(Pentenylidene)-p-toluenesulfonamide (14).

MS-I-139B

Archive directory: /home/temple/vnmrsws/data
Sample directory: mnsoc/MS-I-139B_15Mar2005
File: PROTON

Pulse Sequence: s2pu1

Solvent: C6D6
Ambient temperature
INNOVA-300 "nmr-300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.300 sec

Width 4799.6 Hz

8 repetitions

OBSERVE H1, 299.384389

DATA PROCESSING

Resol. enhancement 0.4

Gauss apodization 0.240 sec

FT size 32768

Total time 0 min, 35 sec

(*R,S,R*)-(-)-Methyl-*N*-(*p*-toluenesulfinyl)-3-aminopehtanoate (15).

534

(*R*,*R*)-(-)-Methyl-*N*-(*p*-toluenesulfinyl)-3-aminopentanoate (15).

MS-I-142A

Archive directory: /home
Sample directory: minsoo
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
INOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 4799.6 Hz

8 repetitions
OBSERVE H1, 299.9843628 MHz

DATA PROCESSING

Resol. enhancement 0.4 Hz

Gauss apodization 0.240 sec

FT size 32768

Total time 0 min, 35 sec

(R)-(+)-Methyl 3-(4-methylphenylsulfonamido)heptanoate (16).

MS-I-142Acarbon

Archive directory: /home/temple/vnmr/sys/data
Sample directory: minsoo/MS-I-142Acarbon_22Mar2005
File: CARBON

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
INOVA-300 "nmr300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18859.0 Hz

64 repetitions

OBSERVE C13, 75.4311280 MHz

DECOUPLE H1, 299.9858744 MHz

Power 38 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 12 min, 2 sec

(R)-(+)-Methyl 3-(4-methylphenylsulfonamido)heptanoate (16).

MS-I-145B

Archive directory: /home/temple/vnmrSYS/data
Sample directory: minsoo/MS-I-145B_23Mar2005
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
INOVA-300 "nmr300"

(R)-(+)-4-Methyl N-(1-oxoheptan-3-yl)benzenesulfonamide (17).

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 4799.6 Hz
8 repetitions
OBSERVE H1, 299.9843628
DATA PROCESSING
Resol. enhancement 0.4 Hz
Gauss apodization 0.240 sec
FT size 32768
Total time 0 min, 35 sec

538

MS-I-1458carbon

Archive directory: /home/temple/vnmrsvs/data
Sample directory: minsoo/MS-I-1458carbon_23Mar2005
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18859.0 Hz
64 repetitions

OBSERVE C13, 75.4311280 MHz
DECOUPLE H1, 299.9856744 MHz
Power 38 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec

(R)-(+)-4-Methyl N-(1-oxoheptan-3-yl)benzenesulfonamide (17).

539

MS-I-159A
proton.atm

Current Data Parameters
NAME minsoo
EXPNO 190
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050411
Time 13.11
INSTRUM spect
PROBHD 5 mm PABEO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 0.00 dB
SFO1 400.0220001 MHz

F2 - Processing parameters
SI 32768
SF 400.0200260 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1p 11.000 ppm
F1 4400.22 Hz
F2p -1.000 ppm
F2 -400.02 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.01201 Hz/cm

MS-I-159A
apt15MIN.atm

Current Data Parameters
NAME minsoo
EXPNO 191
PROCNO 1

F2 - Acquisition Parameters

Date 20050411
Time 13.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 2580.3
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
CNS2 145.000000
CNS11 1.000000
D1 5.0000000 sec
d13 0.0000300 sec
d20 0.0068965 sec
DELTA 0.0000726 sec

===== CHANNEL f1 =====

NUC1 13C
P1 5.70 usec
P2 11.40 usec
PL1 2.00 dB
SFO1 100.5951275 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 11.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters

SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 22.00 cm
CT 6.00 cm
F1P 210.000 ppm
F1 21122.86 Hz
F2P -10.000 ppm
F2 -1005.85 Hz
PPMCM 10.00000 ppm/cm
HZCM 1005.85071 Hz/cm

(R)-(+)-4-Methyl N-(tridec-7-en-5-yl)benzenesulfonamide (18).

154

MS-I-150SM

Archive directory: /home/temple/vnmrsys/data
Sample directory: minsoo/MS-
File: PROT0N

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
INNOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 4799.6 Hz
8 repetitions

OBSERVE H1, 299.3843628 MHz
DATA PROCESSING

Resol. enhancement 0.4 Hz
Gauss apodization 0.240 sec
FT size 32768
Total time 0 min, 35 sec

(*R,Z*)-(+)-4-methyl-N-(tridec-7-en-5-yl)benzenesulfonamide (19).

542

(R,Z)-(+)-4-methyl-N-(tridec-7-en-5-yl)benzenesulfonamide (19).

543

(2*R*,3*S*,5*R*)-(+)-5-butyl-3-iodo-2-pentyl-1-tosylpyrrolidine (20).

544

Archive directory: /home/temple/vnmrsys/data
Sample directory: minsoo/MS-I-163Bdept_13Jul2005
File: CARBON

Solvent: CDCl₃
Ambient temperature
INOVA-300 "nmr300"

Pulse 45.0 degrees
Acq. time 1.815 sec

512 repetitions

Power 38 dB

WALTZ-16 modulat
DATA PROCESSING

FT size 131072

545

MS-I-154A
proton.atm

Current Data Parameters
NAME minsoo
EXPNO 182
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050404
Time 16.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 ¹H
P1 9.80 usec
PL1 0.00 dB
SF01 400.0220001 MHz

F2 - Processing parameters
SI 32768
SF 400.0200200 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 11.000 ppm
F1 4400.22 Hz
F2P -1.000 ppm
F2 -400.02 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.01201 Hz/cm

MS-I-154A
apt15MIN.atm

Current Data Parameters
NAME minsoo
EXPNO 181
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050404
Time 16.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 25125.629 Hz
FIDRES 0.38387 Hz
AQ 1.3042164 sec
RG 1448.2
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
CNS2 145.0000000
CNS11 1.0000000
D1 5.0000000 sec
d13 0.0000300 sec
d20 0.0068965 sec
DELTA 0.0000726 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.70 usec
P2 11.40 usec
PL1 2.00 dB
SFO1 100.5951275 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 11.00 dB
SFO2 400.0216001 MHz

F2 - Processing parameters
SI 32768
SF 100.5850690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 6.00 cm
F1P 210.000 ppm
F1 21122.86 Hz
F2P -10.000 ppm
F2 -1005.85 Hz
PFCM 10.00000 ppm/cm
HZCM 1005.85071 Hz/cm

13.852
14.304
16.801
21.734
22.914
26.435
26.939
28.427
28.960
29.494
32.002
33.979
34.211
62.056
77.665
77.348
77.030

129.833
140.603
142.761

(2R,5R)-(-)-2-butyl-5-pentyl-1-tosylpyrrolidine (21).

547

MS-I-176A

Archive directory: /home/temple/vnmrsvs/data
Sample directory: minsoo/MS-I-176A_04May2005
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
INNOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec

Width 4799.6 Hz
8 repetitions

OBSERVE H1, 299.9843628 MHz
DATA PROCESSING

Resol. enhancement 0.4 Hz
Gauss apodization 0.240 sec
FT size 32768

Total time 0 min, 35 sec

(-)-Pyrrolidine 197B (22).

(-)-Pyrrolidine 197B (22).

549

MS-I-176A, carbon2HR.atm.			
F1: 100.585	F2: 1.000	SW1: 25126	OF1: 10057.2
EX:		PW: 5.7 usec	NA: 2600
		PD: 2.0 sec	LB: 3.0
			PTSId: 32768
			WinNuts - \$Minsoo.1

USER: temple -- DATE: May 3 14:44:14 2005 EDT (UT-4h) templ

MS-I-176A file No. 209.

(2*R*,5*R*)-(-)-*N*-Benzoyl-2-butyl-5-pentylpyrrolidine.

055

MS-I-296A, proton.atm.		USER: temple -- DATE: Nov 24 12:48:53 2005 EST (UT-5h) templ	
F1: 400.191	F2: 1.000	OF1: 1974.3	PTSId: 32768
EX:	PW: 9.8 usec	PD: 1.0 sec	LB: 0.1
		WinNuts - \$Minsoo.1	

551

(2*R*,5*R*)-(-)-*N*-Benzoyl-2-butyl-5-pentylpyrrolidine.

MS-I-296A, carbonOVNTE.atm.		USEK: temple -- DATE: Nov 25 04:04:49 2005 EST (UT-5h) templ	
F1: 100.628	F2: 1.000	OF1: 10621.8	PTSId: 32768
EX:		PD: 2.0 sec	LB: 3.0
		WinNuts - \$Minsoo.1	