Asymmetric Synthesis of β -Amino Carbonyl Compounds using *N*-Sulfinyl β -Amino

Weinreb Amides

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Supporting Information Available

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Experimental

General Procedures. Column chromatography was performed on silica gel, Merck grade 60 (230-400 mesh). TLC plates were visualized with UV, in an iodine chamber, or with phosphomolybdic acid, unless noted otherwise. Unless stated otherwise, all reagents were purchased from commercial sources and used without additional purification.

F2: 500.130
 SW1: 6006
 OF1: 2500.6
 NA: 16
 LB: 0.0
 PTSid: 16384
 WinNuts - \$179_pure

10
 8
 6
 4
 2
 0 PPM

S₂

54

EL: 500.130	F2: 500.130	SW: 6006	PD: 1.0 sec	OF: 2500.6	LB: 0.0	PTSD: 16384	USBR: - DATE: Tue Aug 11 14:53:31
Ex: 1 pm18		PW: 8.0 usec		NA: 16			WinNMR - 560.9K

55

200
 150
 100
 50
 PPM

7.5439
 F2: 299.986
 SW: 18859
 PW: 3.9 usec
 PD: 1.0 sec
 NA: 1024
 LB: 0.2
 PTSId: 65536
 USER: -- DATE: Jul 21 2004
 WinNuis - SCARBON.fid

50

dan-4-2
h1.gx

Current Data Parameters

NAME	Value
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Parameter	Value
Date_	20030709
Time	19.06
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	128
DM	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

Parameter	Value
NUC1	1H
P1	10.00 usec
PL1	0.00 dB
SFO1	400.0220001 MHz

F2 - Processing parameters

Parameter	Value
SI	32768
SF	400.0200350 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

Parameter	Value
CX	20.00 cm
CY	12.50 cm
FLP	10.000 ppm
F1	4000.20 Hz
F2	-0.500 ppm
PRNCH	0.52500 ppm/cm
HZCM	210.01053 Hz/cm

S11

dan-4-c13-2
c13.gk

Current Data Parameters
NAME: danyang
EXTNO: 6
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20030710
Time: 9.58
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 900
DS: 0
SWH: 25115.629 Hz
FIDRES: 0.383387 Hz
AQ: 1.3042164 sec
RG: 5160.6
RW: 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: 12.00 usec
PL1: 2.00 dB
SFO1: 100.5961334 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 100.00 usec
PL2: 0.00 dB
PL12: 20.00 dB
PL13: 25.00 dB
SFO2: 400.0216001 MHz

F2 - Processing parameters
SI: 32768
SF: 100.5850690 MHz
WDW: EM
SSB: 0
LB: 4.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
CX: 20.00 cm
CY: 12.50 cm
FLP: 215.000 ppm
F1: 21625.79 Hz
F2P: -5.000 ppm
F2: -502.92 Hz
PRACK: 11.000000 ppm/cm
HZCM: 1106.43579 Hz/cm

S12

215

39

S14

3.86
100.585
F2: 1.000
SW: 25126
PW: 12.0 usec
PD: 2.0 sec
OF: 11063.0
NA: 300
LB: 4.0
USER: temple - DATE: Sep 24 18:23:53 2003 EDT (UT-4h) temple
PTSID: 32768
WinNMR - Sandrewu.1

100
150
100
50
PPM

323pure
carbonOVENTE.atm

Current Data Parameters
NAME Brad
EXPNO 1200
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040513
Time 4.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12000
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 181
DM 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 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 224.885 ppm
F1 22620.06 Hz
F2P -24.910 ppm
F2 -2505.56 Hz
PPMCM 12.46974 ppm/cm
HZCM 1256.28137 Hz/cm

517

11-141	172: 299.986	SWH: 18859	PD: 1.0 sec	OP1: 8297.5	NA: 128	LB: 0.2	PTSLd: 65536	USER: - DATE: Mar 23 2004
31: 75.439		PW: 3.9 usec						WinnuS - SCARBON.fid
3X: 82pul								

215

S19

S 2p

324-2
proton.atm

```

Current Data Parameters
NAME      Brad
EXPNO     1220
PROCNO    1

F2 - Acquisition Parameters
Date_     20040518
Time      18.27
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         812.7
DE         60.400 usec
TE         6.00 usec
D1         300.0 K
           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         400.0200277 MHz
WDW        EM
SSB        0
LB         0.10 Hz
GB         0
PC         1.00

ID 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

```

324-2
carbonOVNTE.atm

- 201.336
- 143.171
- 141.960
- 129.992
- 125.618
- 78.071
- 77.682
- 77.570
- 77.364
- 77.047
- 60.199
- 46.874
- 35.767
- 26.917
- 21.707

Current Data Parameters
NAME Brad
EXPNO 1230
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040519
Time 5.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12000
DS 2
SMH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 181
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 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 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FLP 220.000 ppm
F1 22128.71 Hz
F2 0.000 ppm
PPMCM 11.00000 ppm/cm
HZCM 1106.43579 Hz/cm

525

S23

S24

dan-5-1
hl.gk

Current Data Parameters

NAME	danyang
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20030722
Time	16.06
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	256
DM	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
PL	10.00 usec
PL1	0.00 dB
SFO1	400.0220001 MHz

F2 - Processing parameters

SI	32768
SF	400.0200418 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
FLP	10.000 ppm
F1	4000.20 Hz
F2	-0.500 ppm
F2P	-200.01 Hz
PRICH	0.52500 ppm/cm
HZCN	210.01053 Hz/cm

525

dan-5-cl3
cl3.gk

Current Data Parameters

NAME	danyang
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20030722
Time	16.54
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	600
DS	0
SWH	25125.629 Hz
FIDRES	0.38387 Hz
AQ	1.3042164 sec
RG	8192
DM	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.0002000 sec

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

NUC1	13C
P1	12.00 usec
PL1	2.00 dB
SFO1	100.5961334 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	25.00 dB
SFO2	400.0216001 MHz

F2 - Processing parameters

SI	32768
SF	100.5848821 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CK	20.00 cm
CV	12.50 cm
F1P	215.000 ppm
F1	21625.75 Hz
F2P	-5.000 ppm
F2	-502.92 Hz
PPMCK	11.00000 ppm/cm
MZCK	1106.43372 Hz/cm

S26

S27

225

13

S29

400.020 F2: 1.000 SW1: 8278 PW: 10.0 usec PD: 1.0 sec OF1: 1988.2 NA: 8 LB: 0.3 USER: temple -- DATE: Aug 11 16:32:24 2002 EDT (UT-4h) templ PTSId: 32768 W/inNuis - Sandewwu.1

21.845

531

1 h 1 dec.gk	F2: 1.000	SW1: 75188	PD: 2.0 sec	OF1: -8281.0	LB: 1.0	PTSID: 32768	USER: temple -- DATE: Aug 16:34:26 2002 EDT (UT-4h) templ
161.931				NA: 16			
PW: 12.0 usec							
WinNuts - \$andrewwu.1

14

L: 400.020 F2: 1.000 SWH: 8278 PD: 1.0 sec NA: 8 LB: 0.3
 K: PW: 10.0 usec
 USER: temple - DATE: Sep 24 21:48:39 2003 EDT (UT-4h) templ
 PTS) d: 32768
 WinNuis - Sandrexxm.1

32

S 34

EX: 1 400.020 F2: 1.000 SW1: 8278 PD: 1.0 sec OF1: 1961.7 NA: 8 USER: temple -- DATE: Sep 26 17:41:23 2003 EDT (UT-4h) temp: PTStd: 32768

ppm

8

6

4

2

0

350
h1.gk

Current Data Parameters
NAME Brad
EXPNO 1300
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040709
Time 13.18

INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0

SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
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 400.0200266 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.000 ppm
F1 4000.20 Hz
F2P -0.500 ppm
F2 -200.01 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.01051 Hz/cm

30

350
carbonOVENTE.atm

Current Data Parameters
NAME Brad
EXPNO 1310
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040710
Time 3.47

INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg60

TD 65536
SOLVENT CDCl3
NS 12000

DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz

AQ 1.3042164 sec
RG 181

DE 19.900 usec
TE 6.00 usec

DI 300.0 K
d11 2.00000000 sec
d12 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 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 224.885 ppm
F1 22620.06 Hz
F2P -24.910 ppm
F2 -2505.57 Hz
PRCM 12.48974 ppm/cm
HZCM 1256.28137 Hz/cm

37