Total Synthesis of (-)-Normalindine via Addition of Metallated 4-Methyl-3- cyanopyridine to an Enantiopure Sulfinimine

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Supporting Information Available

Contents:

^1H and ^{13}C spectra for compounds **13**, **14**, **16-21**, **23**, **24** and (-)-**9**.

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.

jeff/044-1dmso_26Jan2004

Archive directory: /home/temple/vnmrsys/data
Sample directory: jeff/044-1dmso_26Jan2004
File: PROTON

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
INOVA-300 "nmr300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 3599.9 Hz

32 repetitions

OBSERVE H1 299.9858004 MHz

DATA PROCESSING

Resol. enhancement 0.4 Hz

Gauss apodization 0.240 sec

FT size 32768

Total time 1 min, 59 sec

14

14

steve/15p_01Jun2004

Archive directory: /home/temple/vnmrSYS/data
Sample directory: steve/15p_01Jun2004
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
32 repetitions
OBSERVE H1, 299.9843628 MHz
DATA PROCESSING
Resol. enhancement 0.4 Hz
Gauss apodization 0.240 sec
F1 size 32768
Total time 1 min, 59 sec

16a

steve/15pc-13_01Jun2004

Archive directory: /home/temple/vnmrsys/data
Sample directory: steve/15pc-13_01Jun2004
File: CARBON

Pulse Sequence: szpul

Solvent: CDCl3
Ambient temperature
INNOVA-300 "nmr300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 20741.5 Hz

1536 repetitions

OBSERVE C13, 75.4311280 MHz

DECOUPLE H1, 299.9858744 MHz

Power 34 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 9 hr, 24 min, 47 sec

16a

steve/p35_25May2004

May 25 2004

USER:

Experiment = s2pul

Pulse length = 4.40 usec

Recycle delay = 1.00 sec

NA = 32

PTS1d = 16384

F1 = 299.986176 MHz

F2 = 75.438675 MHz

SW1 = 4799.62 Hz

AT1 = 3.41 sec

Hz per Pt 1stD = 0.29 Hz

SW2 = 1.00 Hz

Hz per Pt 2ndD = 1.00 Hz

O1 = 1799.95 Hz

O2 = -0.50 Hz

LB1 = 0.00 Hz

TP A = 122.16

B = 39.53

166

steve/p35_25May2004

F1: 299.986

F2: 75.439

EX: s2pul

SW1: 4800

PW: 4.4 usec

PD: 1.0 sec

OF1: 1800.0

NA: 32

LB: 0.0

PTS1d: 16384

W/nNuts: \$PROTON.fid

USER: -- DATE: May 25 2004

166

steve/p35c13_25May2004	OF1: 8297.5	PTSD: 65536	USER: -- DATE: May 25 2004
F1: 75.439	NA: 1024	LB: 0.0	WinNmr - \$CARBON.fid
EX: s2pul	PD: 1.0 sec	SW1: 18859	
	PW: 3.9 usec		

steve/17_03Jun2004

Jun 3 2004

USER:

Experiment = s2pul

Pulse length = 4.40 usec

Recycle delay = 1.00 sec

NA = 32

PTS1d = 16384

F1 = 299.986176 MHz

F2 = 75.438675 MHz

SW1 = 4799.62 Hz

AT1 = 3.41 sec

Hz per Pt 1stD = 0.29 Hz

SW2 = 1.00 Hz

Hz per Pt 2ndD = 1.00 Hz

O1 = 1799.95 Hz

O2 = -0.50 Hz

LB1 = 0.00 Hz

TP A = 151.69

B = -13.33

17a

steve/17_03Jun2004

F1: 299.986

F2: 75.439

SW1: 4800

PW: 4.4 usec

PD: 1.0 sec

OF1: 1800.0

NA: 32

LB: 0.0

PTS1d: 16384

USER: -- DATE: Jun 3 2004

WinNmr - SPROTON.fid

EX: s2pul

C:\MY DOC NTS\NUTSIDATA\SCARBON.fid

steve/17c-13_03Jun2004

Jun 3 2004

USER:

Experiment = s2pul

Pulse length = 3.90 usec

Recycle delay = 1.00 sec

NA = 640

PTS1d = 65536

F1 = 75.439613 MHz

F2 = 299.985870 MHz

SW1 = 20741.51 Hz

AT1 = 3.16 sec

Hz per Pt 1stD = 0.32 Hz

SW2 = 1.00 Hz

Hz per Pt 2ndD = 1.00 Hz

O1 = 8486.07 Hz

O2 = -0.50 Hz

LB1 = 0.00 Hz

TP A = -155.93

B = 199.76

176

steve/17c-13_03Jun2004

USER: -- DATE: Jun 3 200

F1: 75.440

F2: 299.986

SW1: 20742

OF1: 8486.1

PTS1d: 65536

EX: s2pul

PW: 3.9 usec

PD: 1.0 sec

NA: 640

LB: 0.0

WmNuts - \$CARBON.f

176

USER: -- DATE: May 25 2004

PTS1d: 16384

OF1: 1800.0

NA: 32

SW1: 4800

PW: 4.4 usec

PD: 1.0 sec

steve/37p_25May2004

F1: 299.986

F2: 75.439

EX: s2pul

LB: 0.0

WinNmr

*PROTON.fid

rent Data Parameters
 IE jeff
 NO 2251
 CNO 1

- Acquisition Parameters
 e_ 20030929
 ae 11.44

YTRUM spect
 BHD 5 mm QNP 1H/13

PULPROG zgpg60
 TD 65536
 SOLVENT CDC13

NS 600
 DS 0

SWH 25125.629 Hz
 FIDRES 0.383387 Hz

AQ 1.3042164 sec
 RG 9195.2

DW 19.900 usec
 DE 6.00 usec

TE 300.0 K
 DI 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
 F1P 215.000 ppm
 F1 21625.79 Hz
 F2P -5.000 ppm
 F2 -502.92 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.43579 Hz/cm

steve/37by_08Jun2004

Jun 8 2004

USER:

Experiment = s2pul

Pulse length = 4.40 usec

Recycle delay = 1.00 sec

NA = 32

PTS1d = 16384

F1 = 299.986176 MHz

F2 = 75.438675 MHz

SW1 = 4799.62 Hz

AT1 = 3.41 sec

Hz per Pt1sID = 0.29 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = 1799.95 Hz

O2 = -0.50 Hz

LB1 = 0.00 Hz

TP A = 130.01

B = 14.79

512

steve/37by_08Jun2004

F1: 299.986 F2: 75.439

EX: s2pul SW1: 4800 PW: 4.4 usec

PD: 1.0 sec

NA: 32

LB: 0.0

C:\MY DOCUMENTS\11\OUTSIDATA\CARBON.fid

steve/37byc-13_08Jun2004

Jun 8 2004

USER:

Experiment = s2pul

Pulse length = 3.90 use

Recycle delay = 1.00 se

NA = 384

PTS1d = 65536

F1 = 75.439613 MHz

F2 = 299.985870 MHz

SW1 = 20741.51 Hz

AT1 = 3.16 sec

Hz per Pt 1stD = 0.32 Hz

SW2 = 1.00 Hz

Hz per Pt 2ndD = 1.00 Hz

O1 = 8486.07 Hz

O2 = -0.50 Hz

LB1 = 0.00 Hz

TP A = -106.64

B = 188.13

steve/37byc-13_08Jun2004	OF1: 8486.1	PTS1d: 65536	USER: -- DATE: Jun 8 2004
F1: 75.440	NA: 384	LB: 0.0	WinNutz: \$CARBON.fid
EX: s2pul	SW1: 20742	PD: 1.0 sec	
	PW: 3.9 usec

jeff/314proyon_15Jul2004		USER: -- DATE: Jul 15 2004	
F1: 299.986	F2: 75.439	PTS1d: 16384	
	SW1: 3600	OF1: 1500.0	
	PW: 4.4 usec	NA: 8	LB: 0.0
EX: s2pul	PD: 1.0 sec	WinNuts - \$PROTON.fid	

515

(+)-20a

jeff314-c-13_15Jul2004	OF1: 7166.0	PTS1d: 32768	USER: -- DATE: Jul 15 2004
F1: 75.438	NA: 64	LB: 0.0	
EX: s2pul	PD: 1.0 sec		WinNuts - \$CARBON.fid
	SW1: 15842		
	PW: 3.9 usec		
	F2: 299.986

(+)-20a

USER: -- DATE: Aug 3 2004

PTSID: 16384

OF1: 1499.9

PD: 1.0 sec

SW1: 3600
PW: 4.4 usec

J4
F2: 75.439

jeff/319-01 03 Aug 2004

WinNuts - \$PROTON.fid

LB: 0.0

NA: 32

PW: 4.4 usec

EX: s2pul

S 16

(+)-20a

jeff/319carbon_03Aug2004	USER: -- DATE: Aug 3 2004
F1: 75.438	PTS1d: 32768
EX: s2pul	WinNuts - \$CARBON.fid
F2: 299.986	LB: 0.0
SW1: 15842	OF1: 7166.0
PW: 3.9 usec	NA: 1216
PD: 1.0 sec	

jeff/185-reprep4_05Feb2004

Archive directory: /home/temple/vnmrSYS/data
Sample directory: jeff/185-reprep4_05Feb2004
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 3599.9 Hz

8 repetitions
OBSERVE H1, 299.9843744 MHz

DATA PROCESSING

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

47-206

jeff/185rep4-C13_05Feb2004

Archive directory: /home/temple/vnmrSYS/data
Sample directory: jeff/185rep4-C13_05Feb2004
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 15841.6 Hz

2688 repetitions

OBSERVE C13, 75.4311129 MHz

DECOUPLE H1, 299.9858744 MHz

Power 34 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 85536

Total time 9 hr, 24 min, 47 sec

(+/-) 206

(+)-20c

(+)-20c

Current Data Parameters
 NAME jeff
 EXPNO 650
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030307
 Time 8.53
 INSTRUM spect
 PROHD 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 456.1
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 400.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0200147 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
 FLIP 10.000 ppm
 F1 4000.20 Hz
 F2 -0.500 ppm
 F2 -200.01 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.01051 Hz/cm

278

jeff/319carbon 03Aug2004

F1: 75.438

F2: 299.986

EX: s2pul

SW1: 15842

SW1: 1584Z
PW: 3.9 usec

PD: 1.0 sec

OF1: 7166.0

NA: 1216

LB: 0.0

USER: -- DATE: Aug 3 2004

PTS1d: 32768

WinNuts - \$CARBON.fid

523

(+)-21b

525

(+)-23a

F1: 400.022	F2: 1.000	SW1: 8278	OF1: 2000.1	USER: temple -- DATE: Mon Apr 07 12:55:39 2003
EX:		PW: 10.0 usec	NA: 8	PTS1d: 32768
		PD: 1.0 sec	LB: 0.0	WinNuts - \$jeff.820

1-7-23a

F1: 100.596	F2: 1.000	SW1: 25126		OF1: 11063.8	PTSId: 32768	USER: temple --- DATE: Mon Apr 07 13:47:26 2003
EX:		PW: 12.0 usec	PD: 2.0 sec	NA: 600	LB: 0.0	
					WinNuts - \$jeff.910

15223b

528

jeff/205-1_11Nov2003	USER: -- DATE: Nov 11 2003
F1: 299.986	PTS1d: 16384
EX: s2pul	LB: 0.0
SW1: 4800	OF1: 1790.8
PW: 4.4 usec	NA: 32
PD: 1.0 sec	WinNuts - \$PROTON.fid

(-)-23b

jeff/351-2_140ct2004

Archive directory: /home/temple/vnmrsys/data
Sample directory: jeff/351-2_140ct2004
File: PROTON

Pulse Sequence: s2pul

Solvent: CD3OD
Ambient temperature
INNOVA-300 "nmr300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 3599.9 Hz
64 repetitions
OBSERVE H1, 299.9855448 MHz
DATA PROCESSING
Resol. enhancement 0.4 Hz
Gauss apodization 0.240 sec
FT size 32768
Total time 3 min, 51 sec

(-)-24

1-24

531

jeff/224-3longe-13_21Feb2004	OF1: 8297.5	LB: 0.0	PTS1d: 65536	USER: -- DATE: Feb 21 2004
F1: 75.440	NA: 48000			
EX: s2pul	PD: 1.0 sec			
	SW1: 18859			
	PW: 3.9 usec			
				WinNuts - \$CARBON.fid

jeff/233-1_09Jan2004

Archive directory: /home/temple/vnmrsys/data
Sample directory: jeff/233-1_09Jan2004
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 3599.9 Hz

64 repetitions

OBSERVE H1, 299.9843744 MHz

DATA PROCESSING

Resol. enhancement 0.4 Hz

Gauss apodization 0.240 sec

FT size 32768

Total time 3 min, 51 sec

(-)-9

532

Jeff/356-1c13_260ct2004

Archive directory: /home/temple/vnmrSYS/data
Sample directory: Jeff/356-1c13_260ct2004
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
8320 repetitions
OBSERVE C13, 75.4311280 MHz
DECOUPLE H1, 299.9858744 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 9 hr, 24 min, 47 sec

(-)-9

533