Concise and Efficient Synthesis of Calothrixin B

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General experimental. Melting points were determined using a Kofler block and are uncorrected. IR spectra were recorded neat or as KBr pellets. ¹H NMR spectra were recorded on spectrometers at 300 MHz or at 400 MHz. ¹³C NMR spectra were recorded on spectrometers at 75 MHz or at 100 MHz. All NMR spectra were run in deuteriated solvents with tetramethylsilane as the internal standard. *J* Values are given in Hz. The mass spectra were obtained in GC/MS mode (EI, 70 eV). Thin layer chromatography (TLC) was carried out on Kieselgel 60 F254 fluorescent plates. Flash chromatography was performed with silica gel (40-63 μ). Reactions carried out under an inert atmosphere refer to the use of argon or nitrogen. Diethyl ether, tetrahydrofuran (THF), toluene and benzene were dried by being distilled from sodium and benzophenone. Dichloromethane and acetonitrile were dried by distillation from calcium hydride. All other reagents were purified by distillation, the pressure being reduced if the boiling point of the compound was greater than 110°C at atmospheric pressure.

Current Data Parameters
NAME jfr02
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031212
Time 18.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.833 Hz
FIDRES 0.004300 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
S1 32768
SF 300.1300034 MHz
WDW EM
SSB 0
GB 0
PC 1.00

1.748
1.954
1.974
1.996
2.017
2.038
2.309
2.332
2.343
2.353
2.426
2.445
2.466
3.017
3.047

5.427

7.269
7.561

(¹H, CDCl₃, 300 MHz)

1.m127

NO LITTLE

1m33

Current Data Parameters
 NAME 1m33
 EXPNO 40
 PROCNO 1
 P2 - Acquisition Parameters
 Date_ 20041206
 Time 15.22
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 456.1
 DW 81.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 2.00 dB
 SFO1 300.1318534 MHz
 P2 - Processing parameters
 SI 32768
 SF 300.1300056 MHz
 WDW EM
 SSB 0
 GB 0.30 Hz
 PC 1.00

lm22 pure

15
(¹H, CDCl₃, 300 MHz)

Current Data Parameters
NAME lm22
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041119
Time 12.21
PROBHD 5 mm BBO BE-H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.000444 Hz
AQ 5.3084660 sec
RG 406.4
DW 81.000 usec
DE 655.1 usec
TE 300.0
D1 1.00000000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300091 MHz
WDW EM
SSB 0
GB 0
PC 1.00

No title

15

(^{13}C , CDCl_3 , 75 MHz)

Current Data Parameters

NAME: 5323
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters

Date_: 20070720
Time: 14.36
INSTRUM: spect
PROBHD: 5 mm Wujlino
PULPROG: zgpg30
D1: 2.00000000 sec
SOLVENT: CDCl3
NS: 4
DS: 0
SWH: 4807.692 Hz
FIDRES: 0.146719 Hz
AQ: 3.4070220 sec
RG: 715
RW: 104.000 usec
DE: 148.57 usec
TE: 300.0 K
D1: 2.00000000 sec
P1: 11.25 usec
SF01: 400.1316000 MHz
NUCLEUS: 1H

F2 - Processing parameters

SI: 32768
SF: 400.1300091 MHz
WDW: no
SSB: 0
LB: 0.00 Hz
GB: 0
PC: 1.00

1D NMR plot parameters

CX: 24.00 cm
F1P: 10.000 ppm
F1: 4001.30 Hz
F2P: -0.500 ppm
F2: -200.07 Hz
PPM0H: 0.43750 ppm/cm
HZ0H: 175.05688 Hz/cm

2.047
1.585
1.253

8.319
8.301
7.514
7.464
7.445
7.429
7.409
7.404
7.390
7.374
7.302
7.287
7.269
7.262
7.260
7.253
7.159
7.139
7.120
5.891
5.838
5.818

16
(¹H, CDCl₃, 400 MHz)

0.7618
2.6922
2.8675
2.3102
2.0000

Integral

ppm

pc7m5

```

Current Data Parameters
NAME          sd25a
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20040127
Time          17.07
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            128
DS            2
SWH           6172.839 Hz
FIDRES        0.094130 Hz
AQ            5.3084660 sec
RG            203.2
DW            81.000 usec
DE            1.50 usec
TE            300.0 K
D1            1.00000000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            7.50 usec
PL            0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI            32768
SF            300.1300003 MHz
WDW           EM
SS            0
LB            0.30 Hz
GB            0
PC            1.00
  
```

No title

185.00
180.11

154.44
153.65
147.61
141.49
136.89
135.77
133.21
129.24
129.02
128.11
126.56
125.78
124.51
124.16
123.15
122.99
117.33
114.21
111.73

77.42
77.20
77.00
76.58
49.07

22
(¹³C, CDCl₃, 75 MHz)

```
Current Data Parameters
Date_ 20050326
Time 19.35
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15004
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DM 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.0000000 sec
d11 0.0300000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```

200 180 160 140 120 100 80 60 40 20 ppm

No title

```

Current Data Parameters
NAME          lmi05
EXPNO         10
PROCNO        1
F2 - Acquisition Parameters
Date_         20050322
Time          23.29
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            1000
DS            4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.621258 sec
RG            16384
WDW            27800 usec
SSB            6.50 usec
TE            300.0 K
D1            1.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

===== CHANNEL f1 =====
NUC1          13C
P1            5.50 usec
PL1           -6.00 dB
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPOPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.00 dB
PL12          23.00 dB
PL13          23.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            5.00 Hz
GB            0
PC            1.40
  
```