

Total Syntheses of (+)-Vigulariol and (-)-Sclerophytin A

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Supporting Information

¹H and ¹³C Spectra

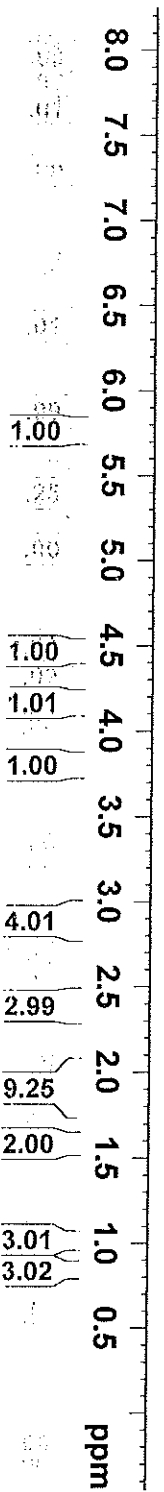
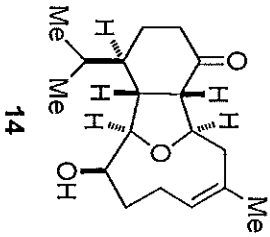
CSS ¹H NMR, 400 MHz, CDCl₃)



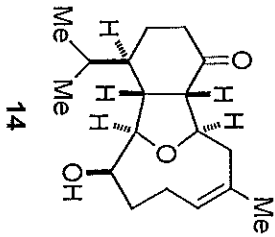
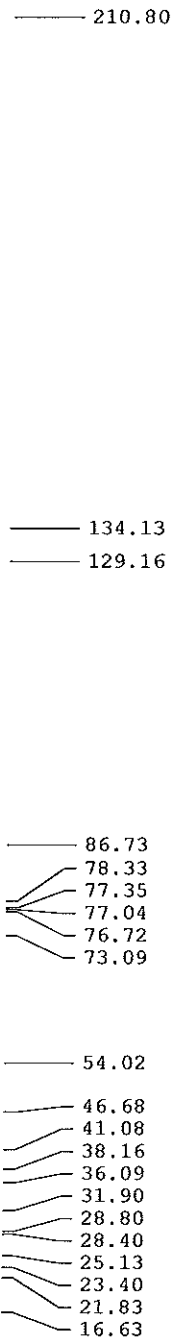
Current Data Parameters
 NAME CSS II-18-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070910
 Time 10.25
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 11
 DS 2
 SWH 6218.905 Hz
 FIDRES 0.094893 Hz
 AQ 5.2691445 sec
 RG 45.3
 DW 80.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

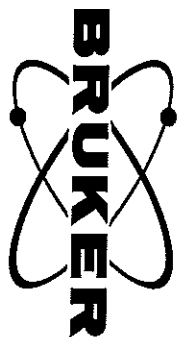
==== CHANNEL F1 =====
 NUC1 ¹H
 P1 9.30 usec
 PL1 -3.00 dB
 SFO1 399.8424924 MHz
 F2 - Processing parameters
 SI 32768
 SF 399.8400000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



CSS : ¹³C NMR, 100 MHz, CDCl₃



Current Data Parameters
 NAME CSS IT-18-1
 EXPNO 2
 PROCNO 1

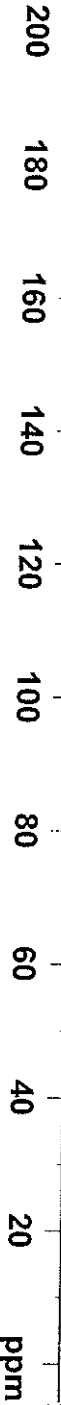


F2 - Acquisition Parameters
 Date_ 20070910
 Time_ 10.33
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 134
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 406.4
 DW 20.850 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

==== CHANNEL F1 =====
 NUC1 13C
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 100.5499020 MHz

==== CHANNEL F2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -3.00 dB
 PL12 16.72 dB
 PL13 20.00 dB
 SFO2 399.8415994 MHz

F2 - Processing Parameters
 SI 32768
 SF 100.5398480 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



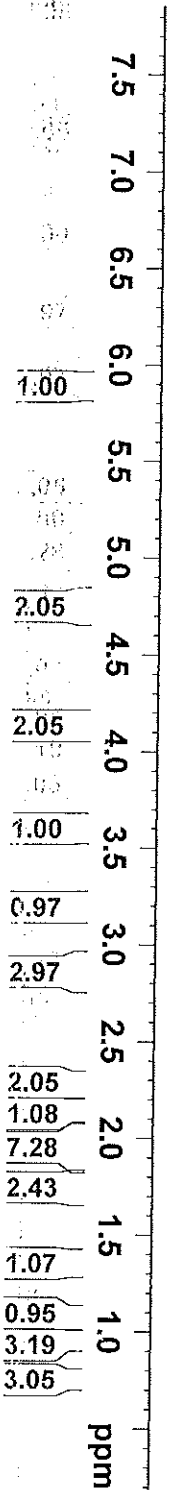
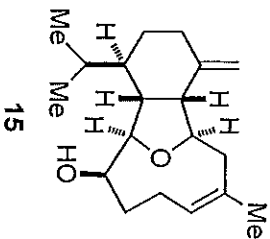
CSS ¹H NMR, 400 MHz, CDCl₃



Current Data Parameters
 NAME CSS-IT-20
 EXPNO 1
 PROCNO 1

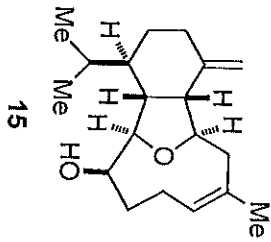
F2 - Acquisition Parameters
 Date_ 20070912
 Time_ 11.37
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 13
 DS 2
 SWH 5341.880 Hz
 FIDRES 0.163021 Hz
 AQ 3.0672283 sec
 RG 71.8
 DW 93.600 usec
 DE 6.00 usec
 TE 293.6 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 ¹H
 P1 12.00 usec
 PL1 -3.00 dB
 SFO1 400.0923618 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.0900000 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



¹³C NMR, 100 MHz, CDCl₃

- 146.92
- 135.60
- 129.25
- 110.53
- 87.79
- 79.77
- 77.35
- 77.03
- 76.72
- 74.19
- 48.47
- 47.16
- 42.36
- 34.75
- 31.87
- 31.60
- 29.17
- 29.10
- 26.88
- 25.57
- 21.97
- 15.87



Current Data Parameters
 NAME CSS-IT-20
 EXPNO 2
 PROCNO 1

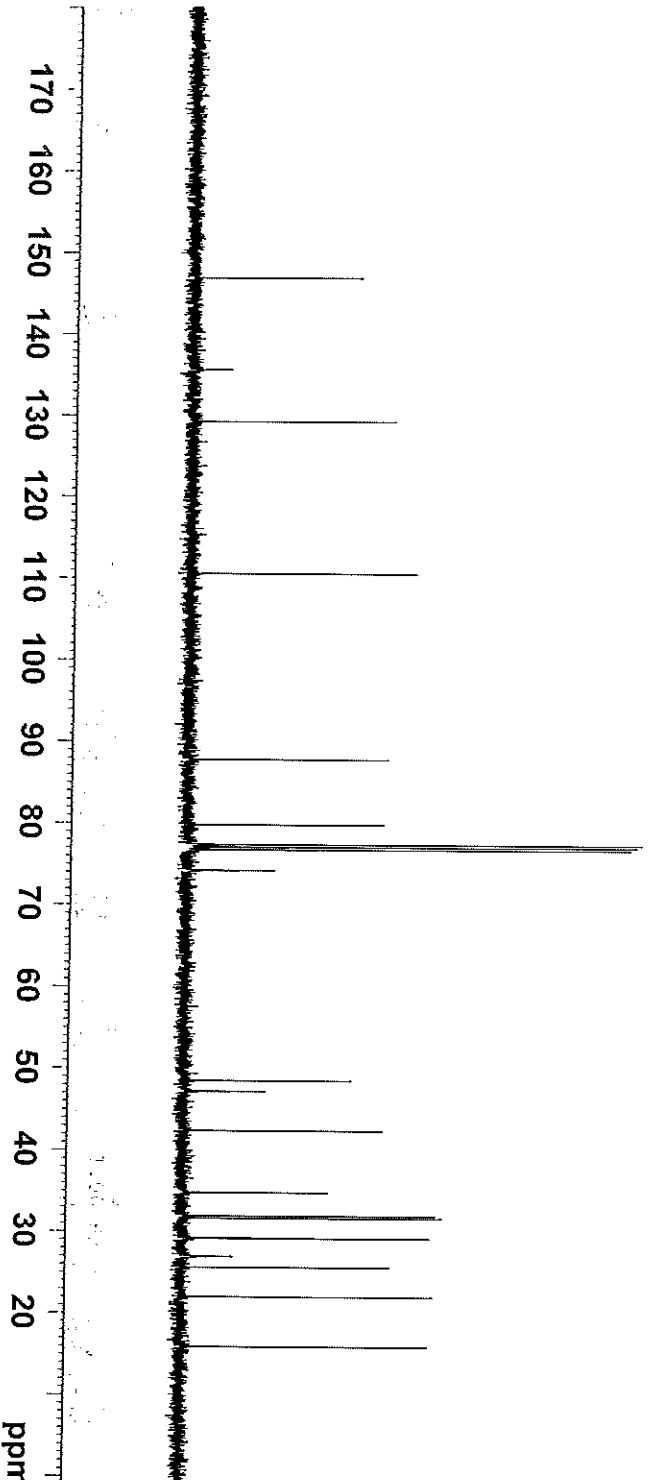
F2 - Acquisition Parameters
 Date_ 20070912
 Time 11.41
 INSTRUM spect
 PROBD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 105
 DS 4
 SMH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 3251
 DW 20.800 usec
 DE 6.00 usec
 TE 293.8 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 6.00 usec
 PL1 0.00 dB
 SFO1 100.6137773 MHz

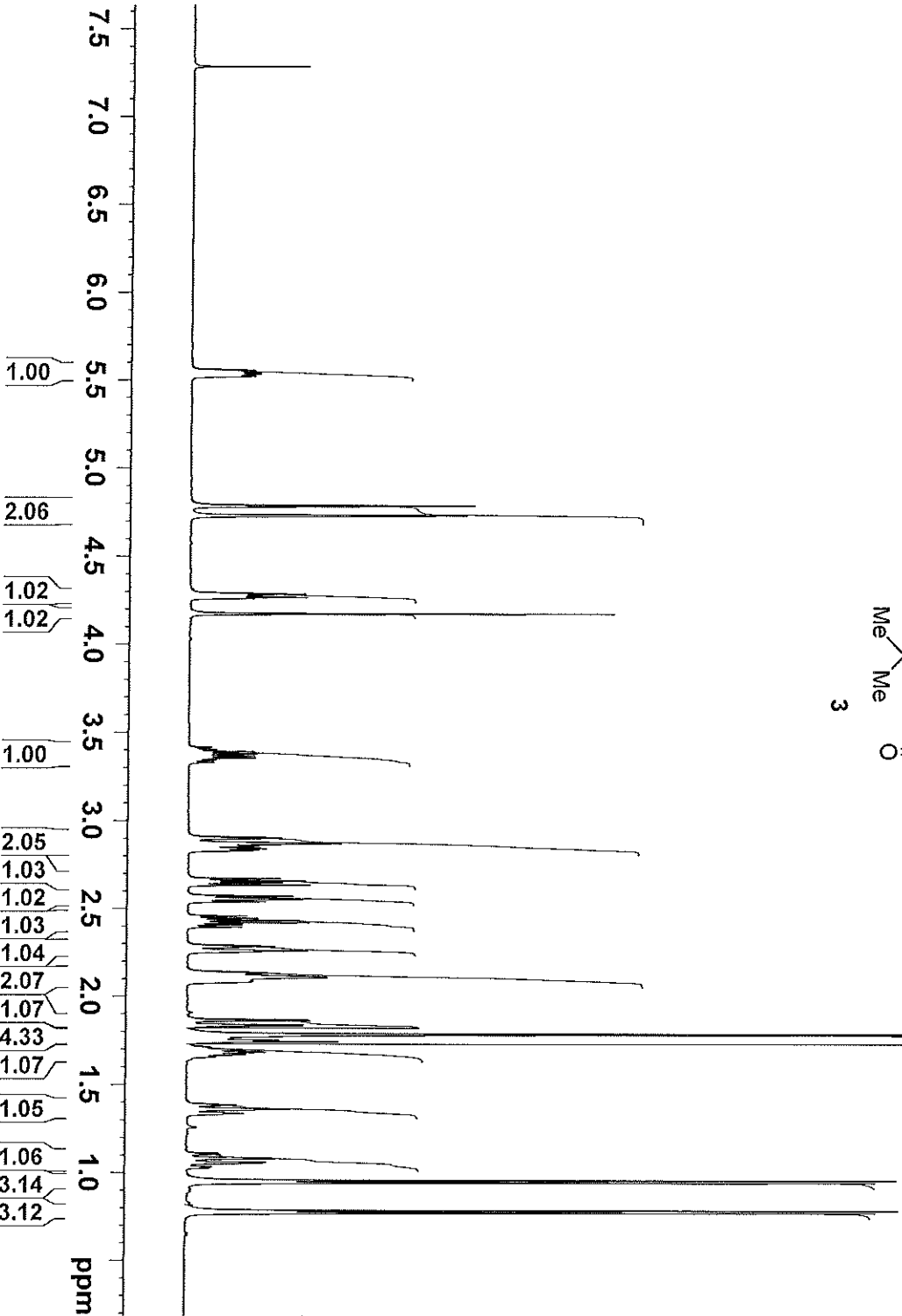
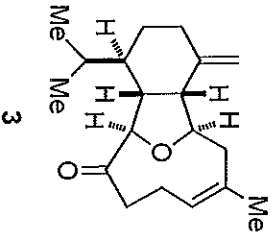
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 10.39 dB
 PL13 11.39 dB
 SFO2 400.0916004 MHz

F2 - Processing parameters

SI 32768
 SF 100.6027110 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



¹H NMR, 500 MHz, CDCl₃



Current Data Parameters
 NAME css2-21
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

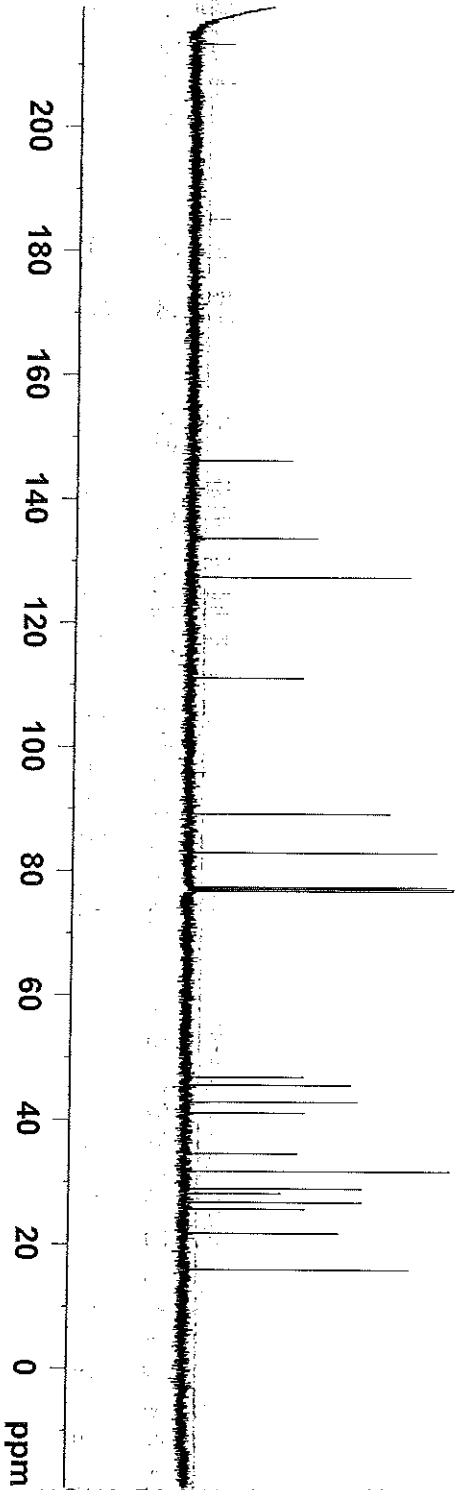
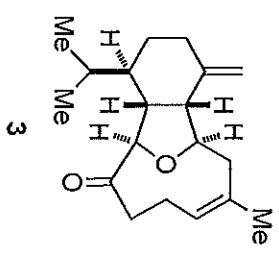
Date_ 20070913
 Time_ 10.07
 INSTRUM spect
 PROBHD 5 mm SEF 1H-19
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 7507.507 Hz
 FIDRES 0.114555 Hz
 AQ 4.3648143 sec
 RG 28.5
 DE 66.600 usec
 TE 291.8 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL F1 =====
 NUCL1 1H
 P1 9.65 usec
 PL1 -3.00 dB
 SFO1 500.1330008 MHz

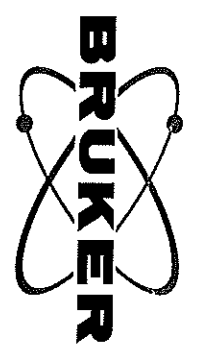
F2 - Processing parameters
 SI 32768
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.25 Hz
 GB 0
 PC 1.00

¹³C NMR, 100 MHz, CDCl₃

- 213.47
- 146.12
- 133.60
- 127.30
- 111.01
- 89.15
- 82.89
- 77.34
- 77.03
- 76.71
- 46.80
- 45.49
- 42.82
- 41.05
- 34.50
- 31.68
- 28.91
- 28.11
- 26.67
- 25.63
- 21.75
- 15.89



Current Data Parameters
 NAME CSS 11-21-1
 EXPNO 2
 PROCNO 1



F2 - Acquisition Parameters
 Date_ 20070913
 Time_ 11.21
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 90
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 101.6
 DE 20.850 usec
 DW 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.899999996 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 100.5499020 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -3.00 dB
 PL12 16.72 dB
 PL13 20.00 dB
 SFO2 399.8415994 MHz

F2 - Processing parameters
 SI 32768
 SE 100.5398480 MHz
 WDW EM
 SSB 0
 GB 0
 PC 1.40

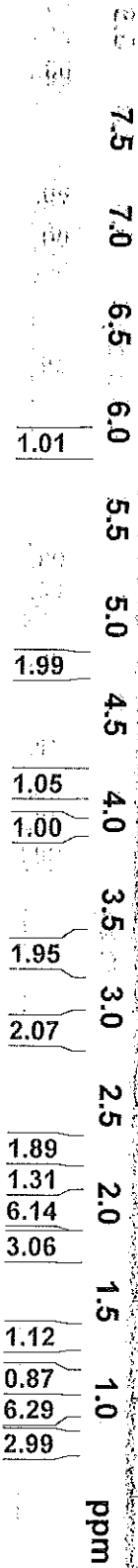
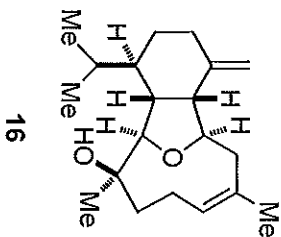
¹H NMR, 400 MHz, CDCl₃



Current Data Parameters
 NAME CSS II-22
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071005
 Time_ 9.30
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6218.905 Hz
 FIDRES 0.094893 Hz
 AQ 5.2691445 sec
 RG 71.8
 DW 80.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

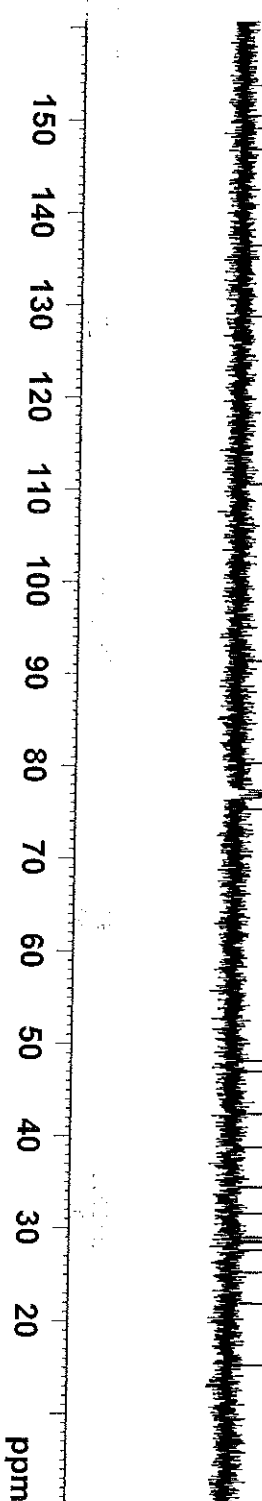
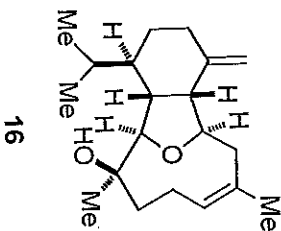
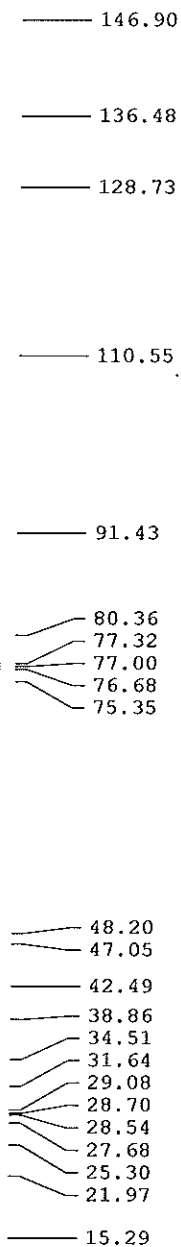
==== CHANNEL f1 =====
 NUC1 1H
 P1 9.30 usec
 P1L -3.00 dB
 SFO1 399.8424924 MHz
 F2 - Processing parameters
 SI 32768
 SF 399.840078 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



ppm

¹³C NMR, 100 MHz, CDCl₃

CSS



Current Data Parameters
 NAME CSS II-22
 EXPNO 2
 PROCNO 1

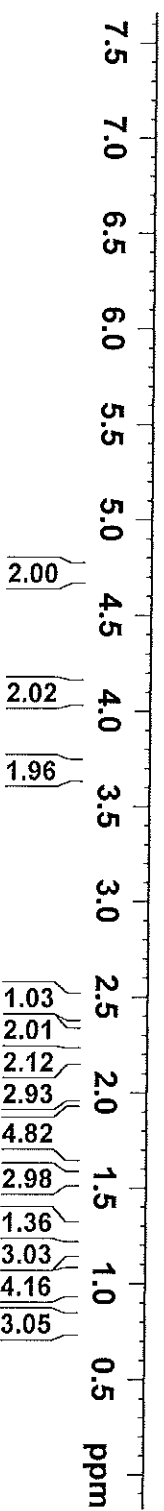
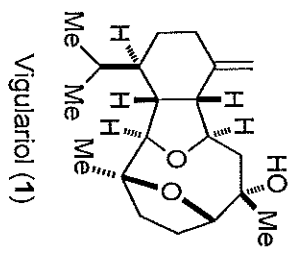
F2 - Acquisition Parameters
 Date_ 20071005
 Time_ 9.40
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 282
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 161.3
 DW 20.850 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

==== CHANNEL F1 =====
 NUC1 13C
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 100.5499020 MHz

==== CHANNEL F2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -3.00 dB
 PL12 16.72 dB
 PL13 20.00 dB
 SFO2 399.8415994 MHz

F2 - Processing parameters
 SI 32768
 SF 100.5398480 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹H NMR, 500 MHz, CDCl₃



Current Data Parameters
 NAME CSS IT-202
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080624
 Time 12.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 2330
 FIDRES 65536
 NS 65536
 SOLVENT CDCl3
 DS 16
 SWH 7507.507 Hz
 FIDRES 0.114555 Hz
 AQ 4.3648143 sec
 RG 181
 DW 66.600 usec
 DE 6.00 usec
 TE 292.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 NUCl 1H
 P1 13.50 usec
 PL1 0.00 dB
 SFO1 500.1330008 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.25 Hz
 GB 0
 PC 1.00



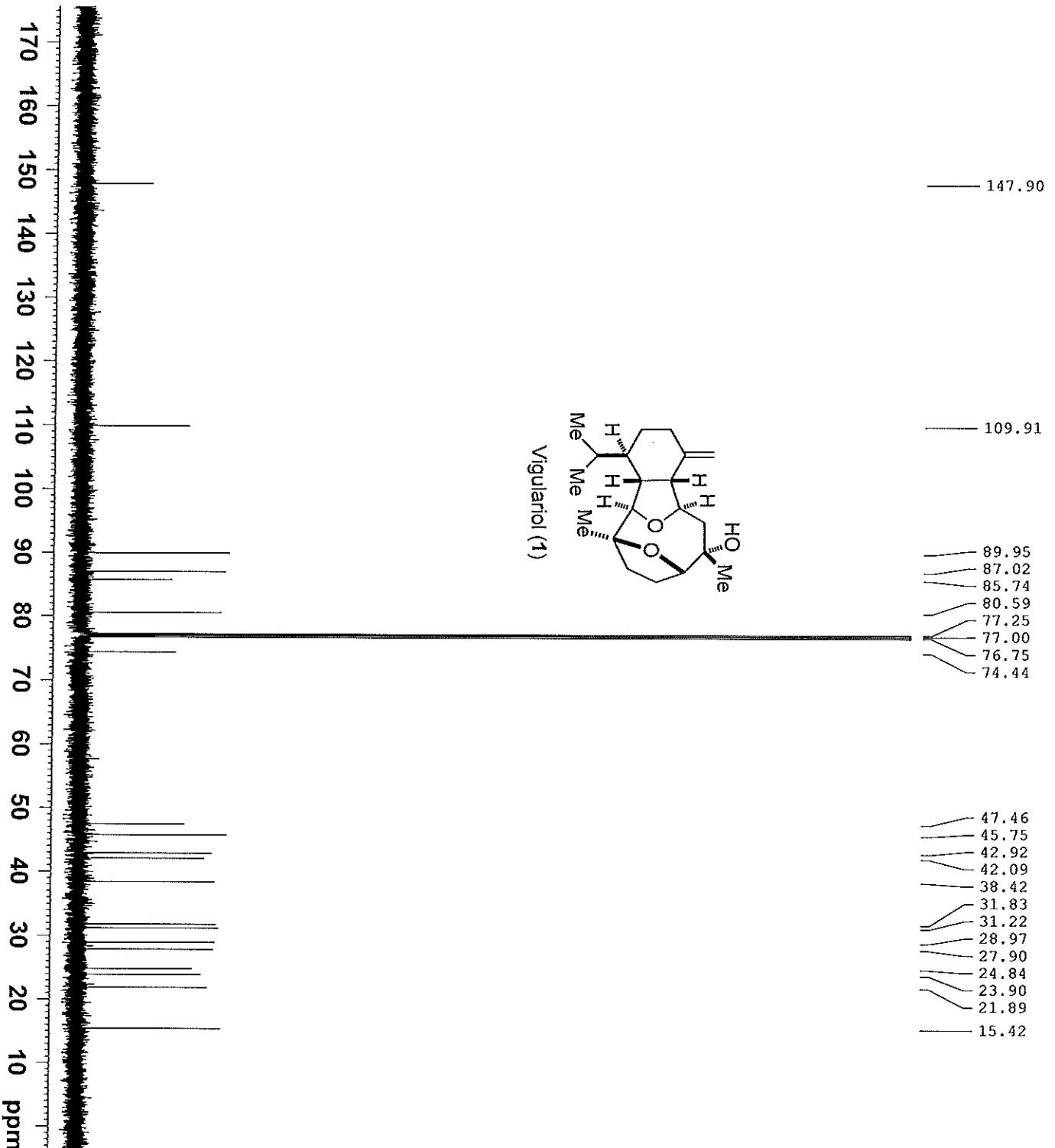
Current Data Parameters
 NAME CSS IT-202-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080624
 Time_ 12.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 94
 DS 2
 SWH 30581.039 Hz
 FIDRES 0.466630 Hz
 AQ 1.0715799 sec
 RG 32768
 DW 16.350 usec
 DE 6.00 usec
 TE 292.5 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TD0 1

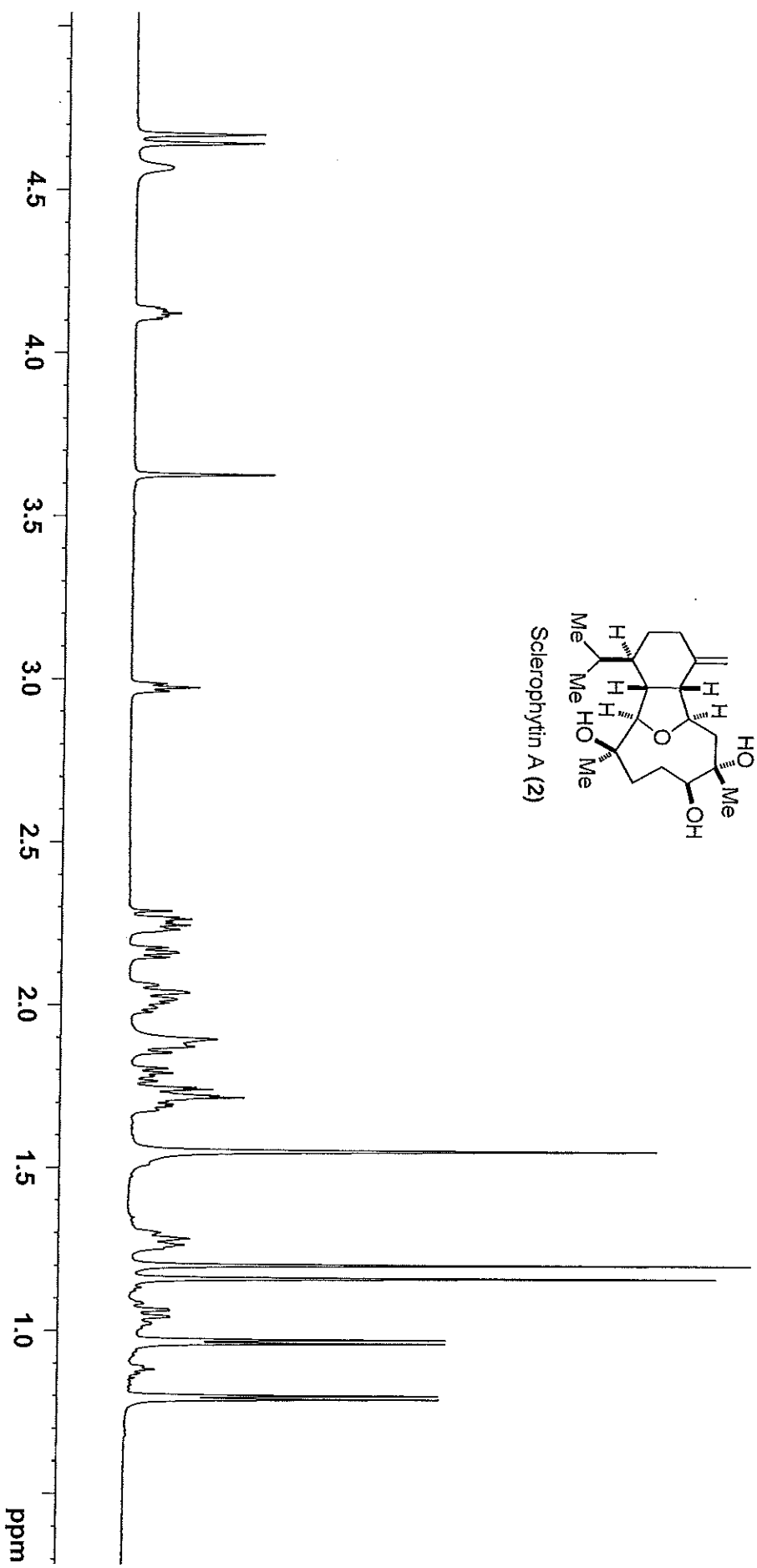
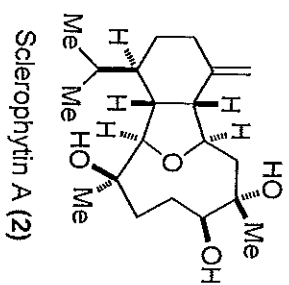
==== CHANNEL f1 =====
 NUC1 13C
 P1 7.00 usec
 PL1 0.00 dB
 SFO1 125.7705462 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 70.00 usec
 PL2 0.00 dB
 PL12 14.30 dB
 PL13 18.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 65536
 SF 125.7577941 MHz
 WMW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.40



^1H NMR, 600 MHz, CDCl_3



^{13}C NMR, 150 MHz, CDCl_3

