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JOURNAL OF THE AMERICAN CHEMICAL SOCIETY

J. Am. Chem. Soc., 1997, 119(39), 9130-9136, DOI:[10.1021/ja9719945](https://doi.org/10.1021/ja9719945)

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SUPPORTING INFORMATION

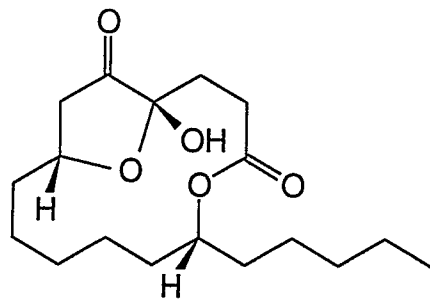
Total Syntheses of (+)-Ricinelaïdic Acid Lactone and of (-)-Gloeosporone Based on Transition Metal Catalyzed C-C-Bond Formations

Alois Fürstner* and Klaus Langemann

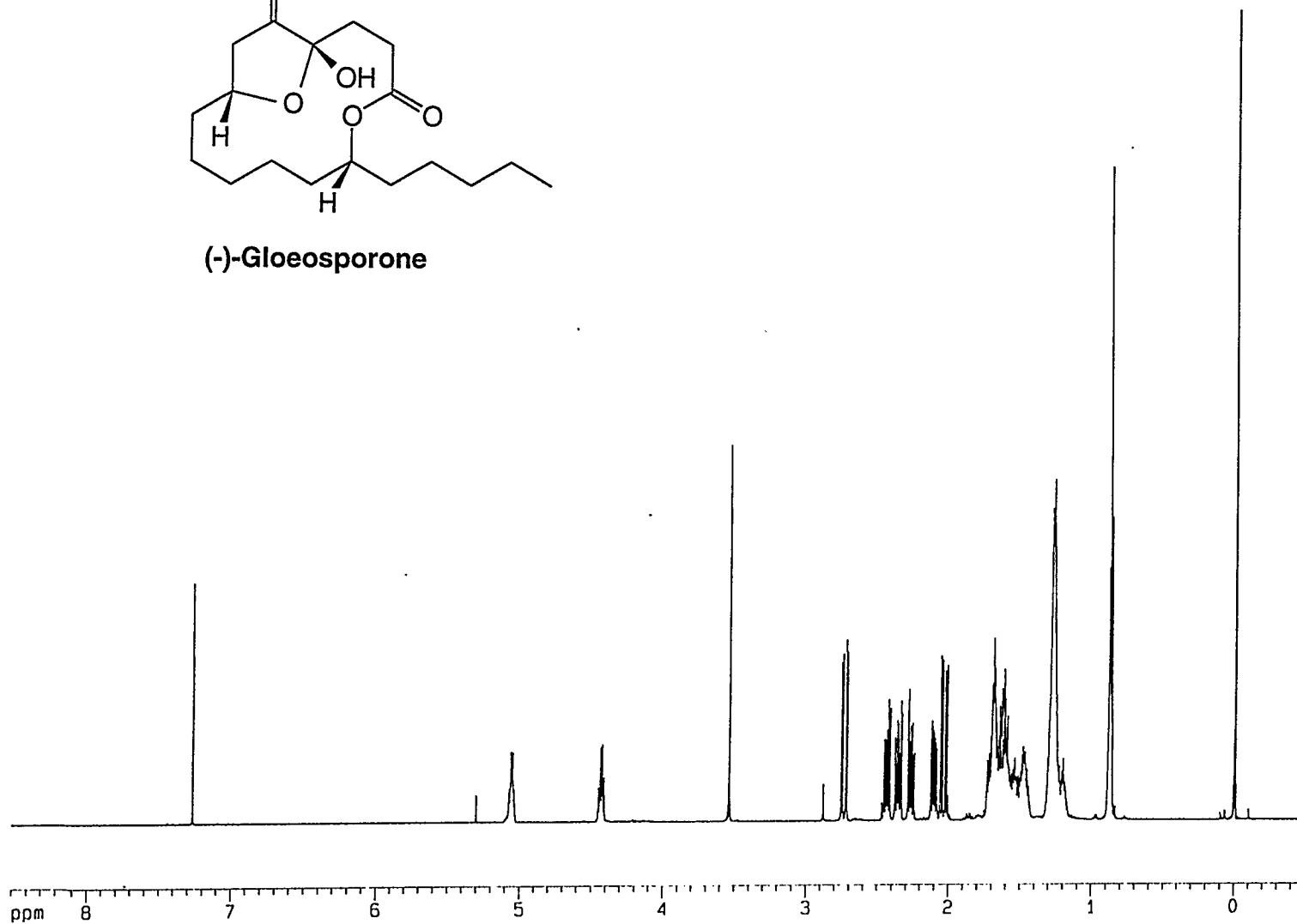
Max-Planck-Institut für Kohlenforschung

Kaiser-Wilhelm-Platz 1, D-45470 Mülheim/Ruhr, Germany

Instrumentation and Spectra Formats. NMR: Spectra were recorded on a Bruker AC 200, AMX 300, AMX 400 or DMX 600 spectrometer in CDCl₃ unless stated otherwise. Chemical shifts (δ) are given in ppm relative to TMS, coupling constants (J) in Hz. IR: Nicolet FT-7199, wavenumbers in cm⁻¹. MS: Varian CH-5 (70 eV); HR-MS: Finnigan MAT SSQ 7000 (70 eV). Melting points were recorded on a Gallenkamp melting point apparatus and are uncorrected. Elemental analyses: Dornis & Kolbe, Mülheim.



(-)-Gloeosporone



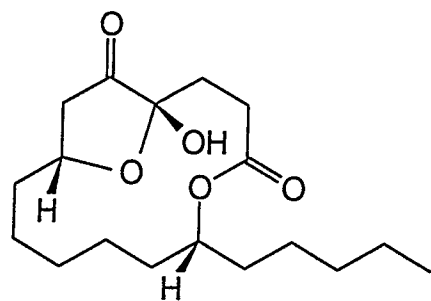
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 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 32
 DS 2
 SMI 12019.230 Hz
 AQ 2.7263477 sec
 RG 32
 DM 41.600 usec
 DE 4.50 usec
 TE 303.0 K
 D1 1.0000000 sec
 P1 9.70 usec
 DE 4.50 usec
 SFO1 600.2242403 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
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 SF 600.2200101 MHz
 SR 10.06 Hz
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 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.50

1D NMR plot parameters
 CX 20.00 cm
 CY 42.00 cm
 F1P 8.500 ppm
 F1 5101.87 Hz
 F2P -0.500 ppm
 F2 -300.11 Hz
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 HZCH 270.09900 Hz/cm

LGM-LB-143-01 1
 1H



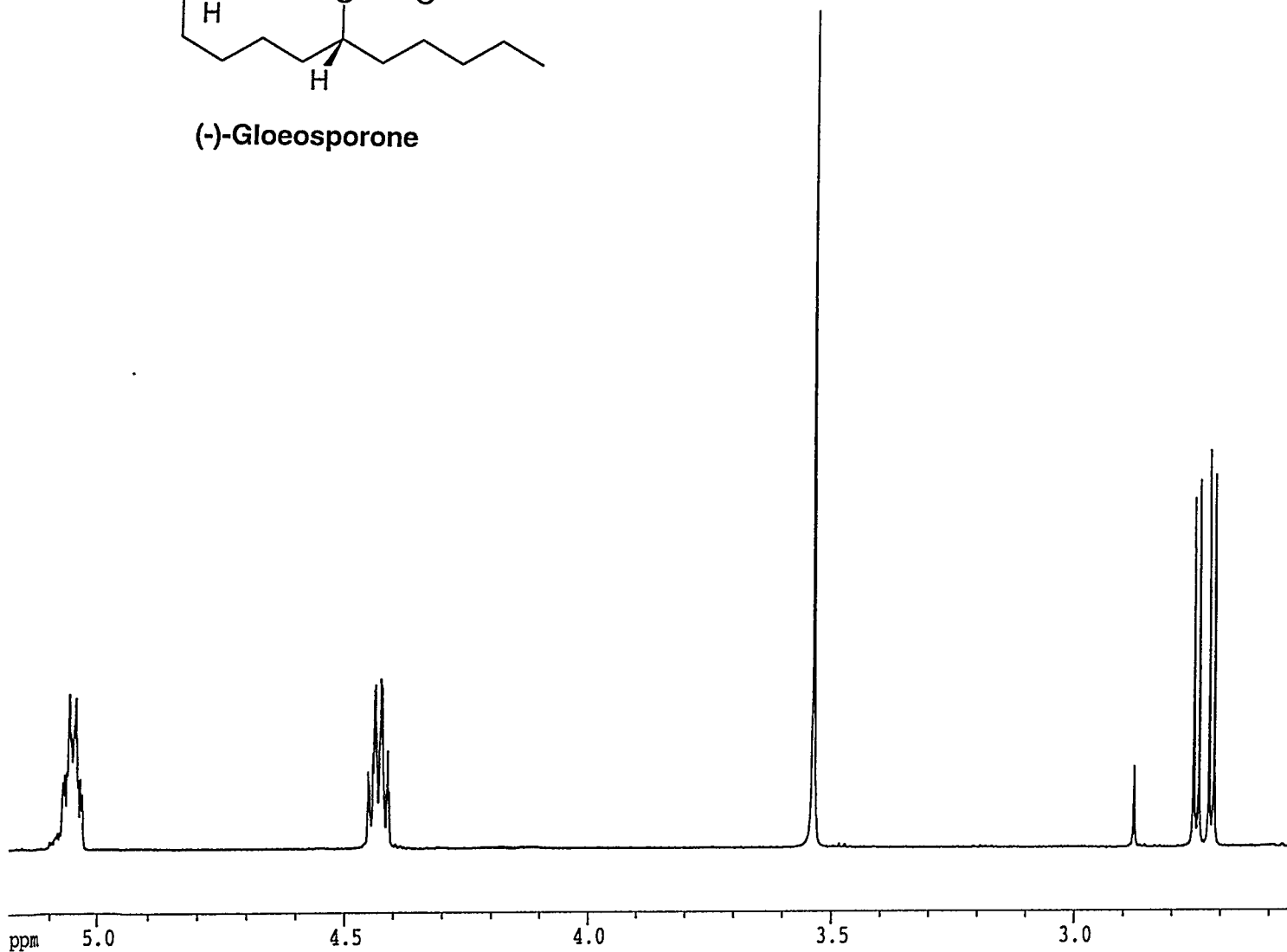
(-)-Gloeosporone

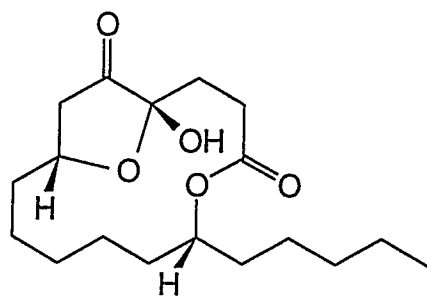
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 PULPROG zg30
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 SOLVENT CDC13
 NS 32
 DS 2
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 AQ 2.7263477 sec
 RG 32
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 DE 4.50 usec
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 NUCLEUS 1H

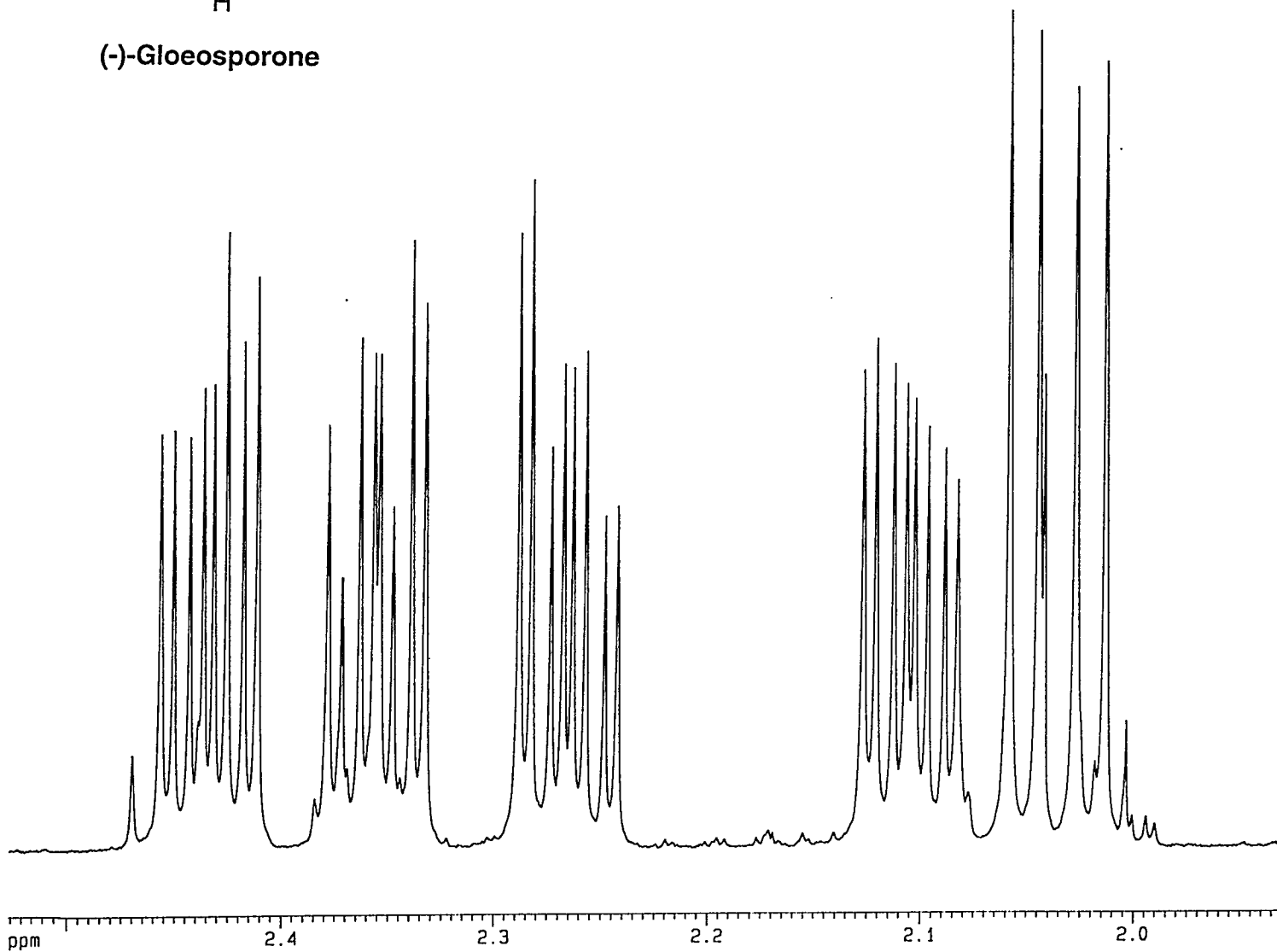
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 F1P 5.196 ppm
 F1 3118.50 Hz
 F2P 2.542 ppm
 F2 1525.96 Hz
 PPMCM 0.13266 ppm/cm
 HZCM 79.62740 Hz/cm





(-)-Gloeosporone



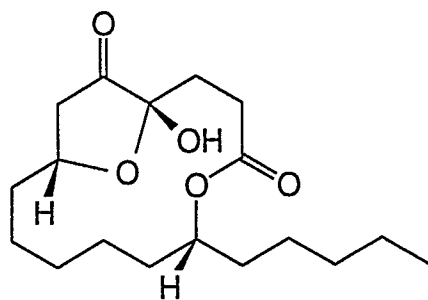
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 USER kl

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 TD 65536
 SOLVENT CDC13
 NS 32
 DS 2
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 AQ 2.7263477 sec
 RG 32
 DW 41.600 usec
 DE 4.50 usec
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 NUCLEUS 1H

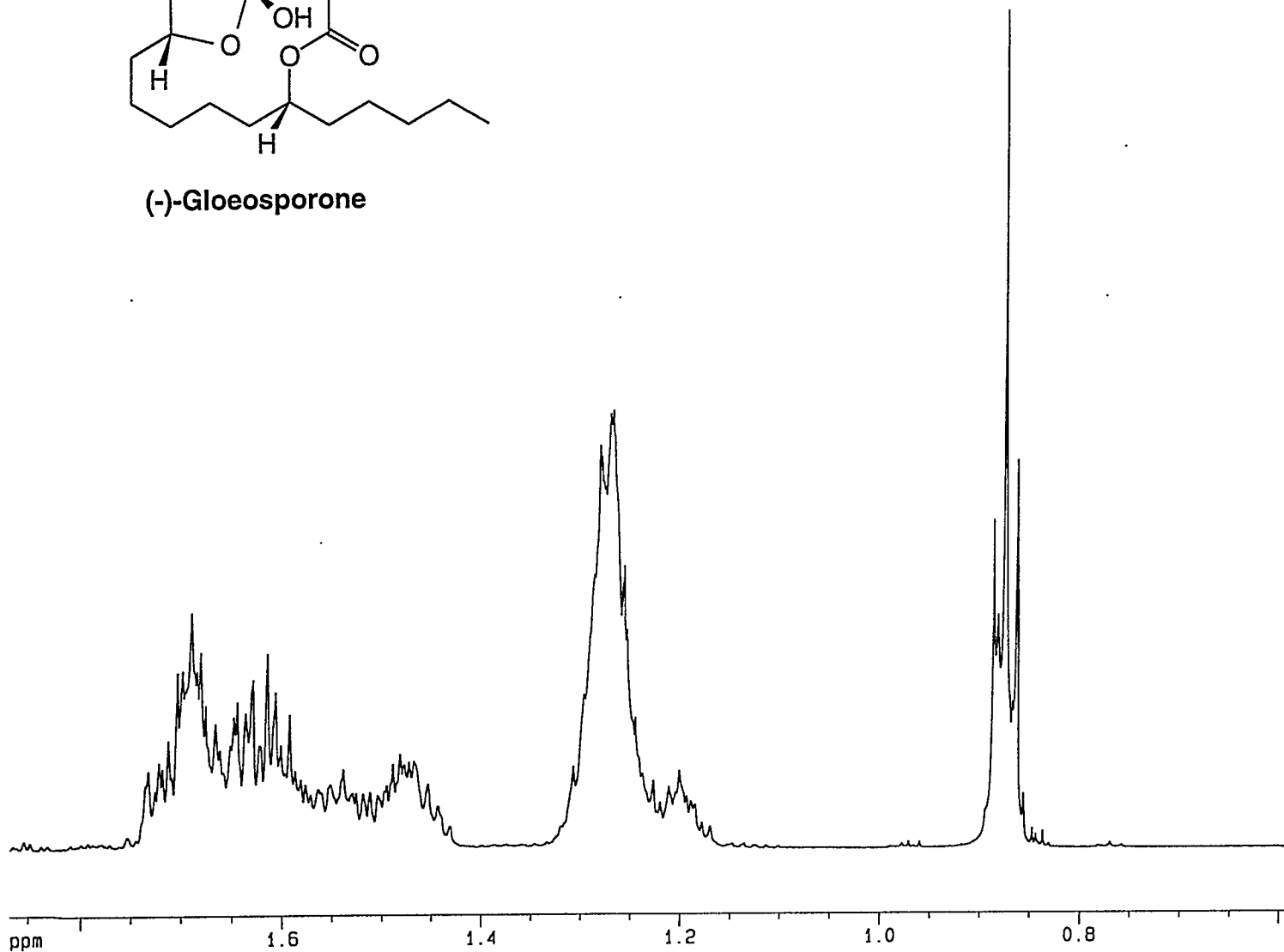
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1D NMR plot parameters
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 F1P 2.526 ppm
 F1 1516.18 Hz
 F2P 1.927 ppm
 F2 1156.82 Hz
 PPMCM 0.02994 ppm/cm
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LGM-LB-143-01 1
 1H



(-)-Gleosporone



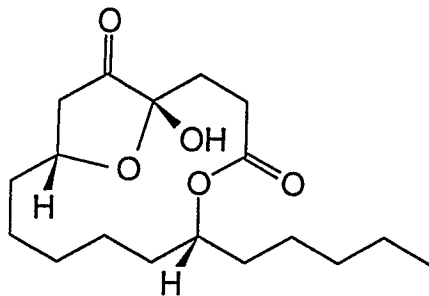
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 DU mpi
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 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 32
 DW 41.600 usec
 DE 4.50 usec
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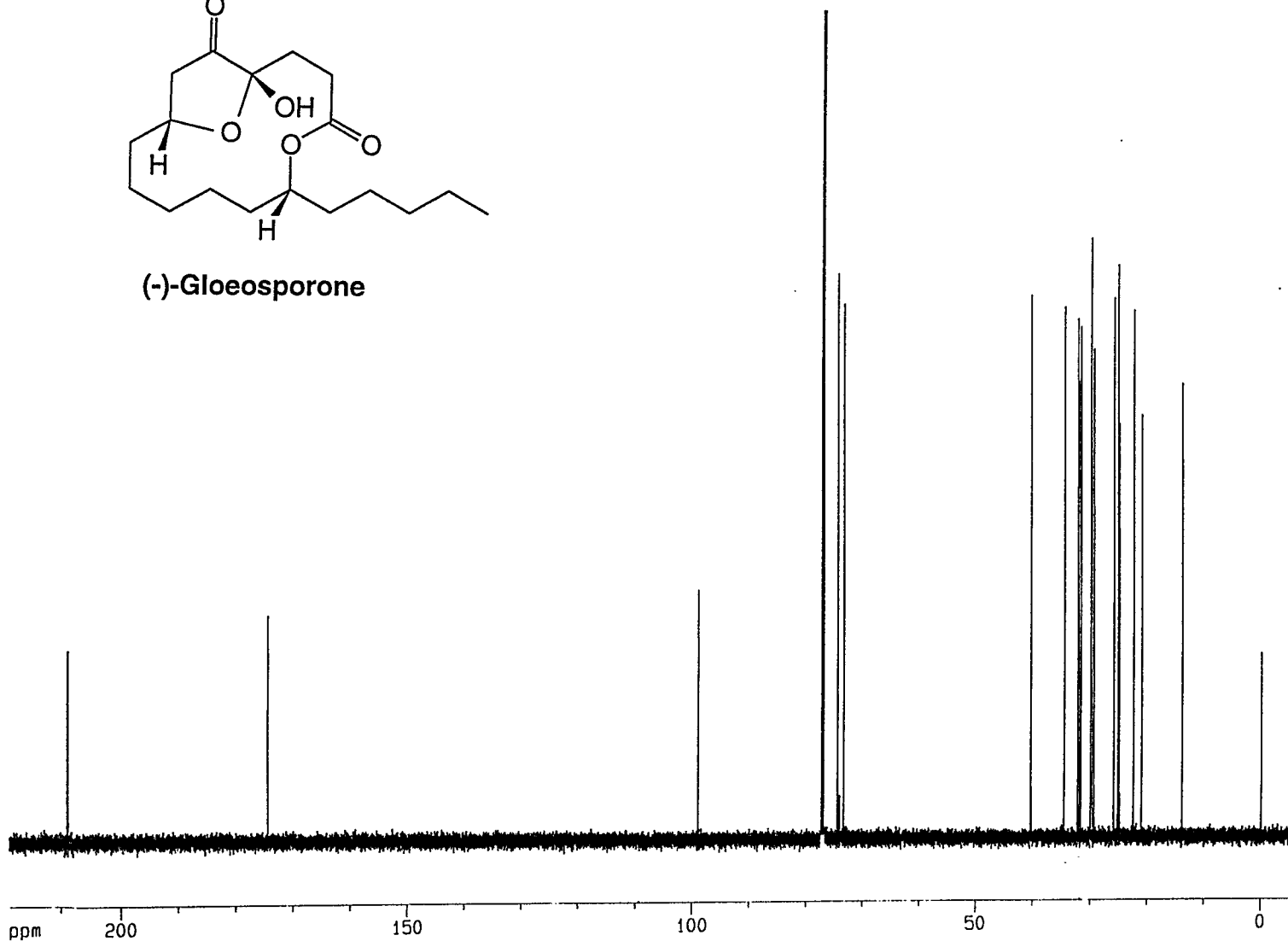
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1D NMR plot parameters
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 F1P 1.868 ppm
 F1 1121.33 Hz
 F2P 0.583 ppm
 F2 349.88 Hz
 PPHCM 0.05426 ppm/cm
 HZCH 38.57263 Hz/cm

LGM-LB-143-01 1
 1H



(-)-Gloeosporone



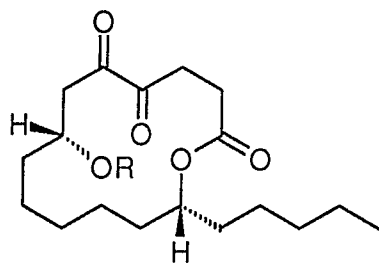
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 EXPNO 11
 PROCNO 1
 DU mpi
 USER k1

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 Time 10.17
 INSTRUM spect
 PROBHD 5 mm TXI 13C
 PULPROG zgdc30
 TD 65536
 SOLVENT CDC13
 NS 4500
 DS 320
 SWH 37593.984 Hz
 FIDRES 0.573639 Hz
 AQ 0.8716788 sec
 RG 16384
 DW 13.300 usec
 DE 4.50 usec
 TE 303.0 K
 D11 0.03000000 sec
 CPDPRG waltz16
 P31 88.00 usec
 S2 83 dB
 HL1 90 dB
 D1 0.03000000 sec
 P1 11.70 usec
 DE 4.50 usec
 SF01 150.9419346 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 65536
 SF 150.9254361 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.50

ID NMR plot parameters
 CX 20.00 cm
 F1P 219.151 ppm
 F1 33075.50 Hz
 F2P -5.317 ppm
 F2 -802.52 Hz
 PPMCH 11.22343 ppm/cm
 HZCH 1693.90100 Hz/cm

LGM-LB-143-01 1
 13C {1H}



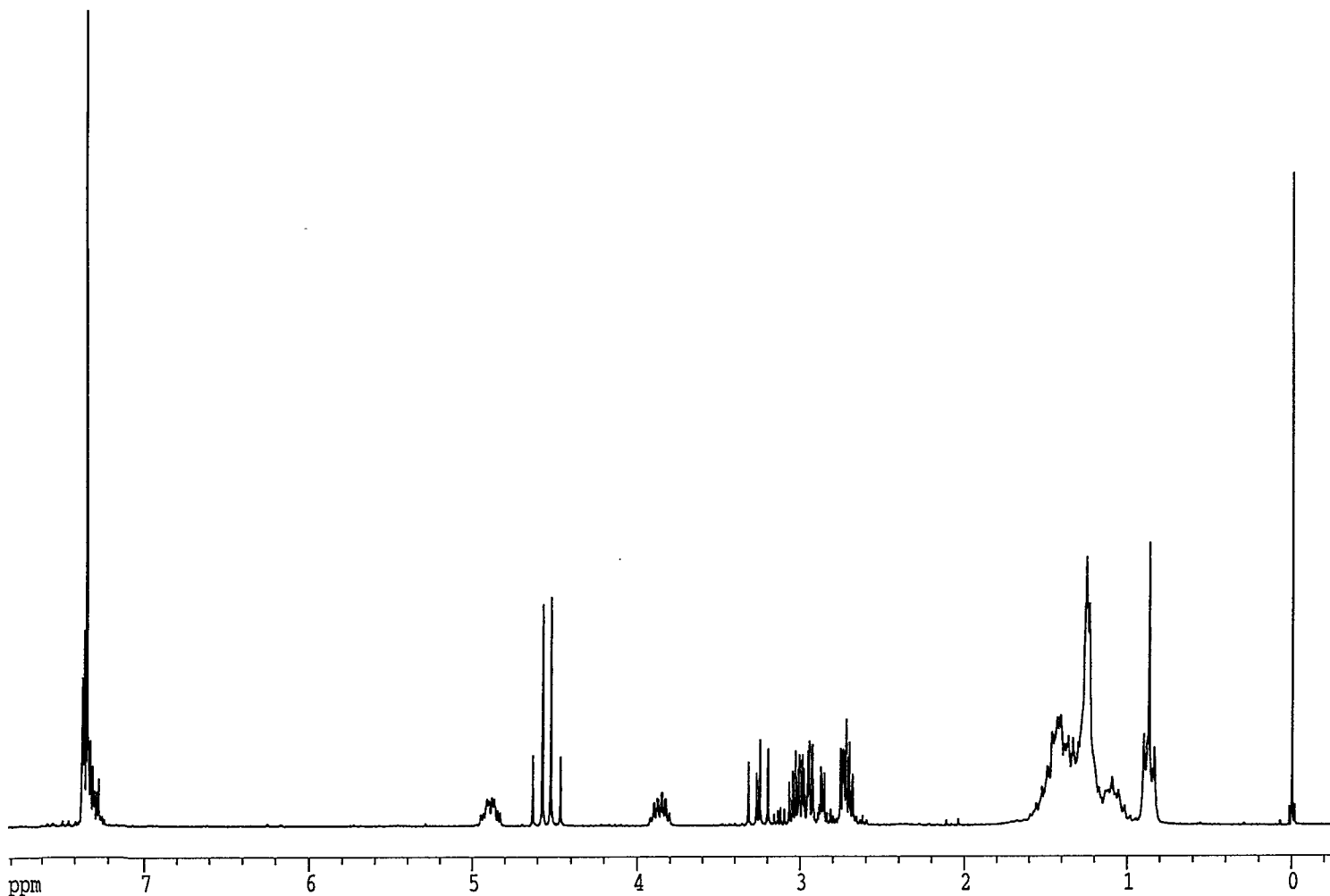
27 R = Bn

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 EXPNO 134
 PROCNO 1
 DU mpi
 USER kl

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 INSTRUM ac200
 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 16
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HLL 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 200.1323407 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
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 F2P -0.287 ppm
 F2 -57.53 Hz
 PPMCM 0.40583 ppm/cm
 HZCM 81.21971 Hz/cm

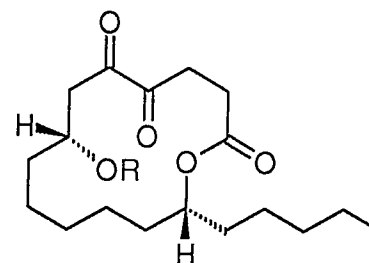


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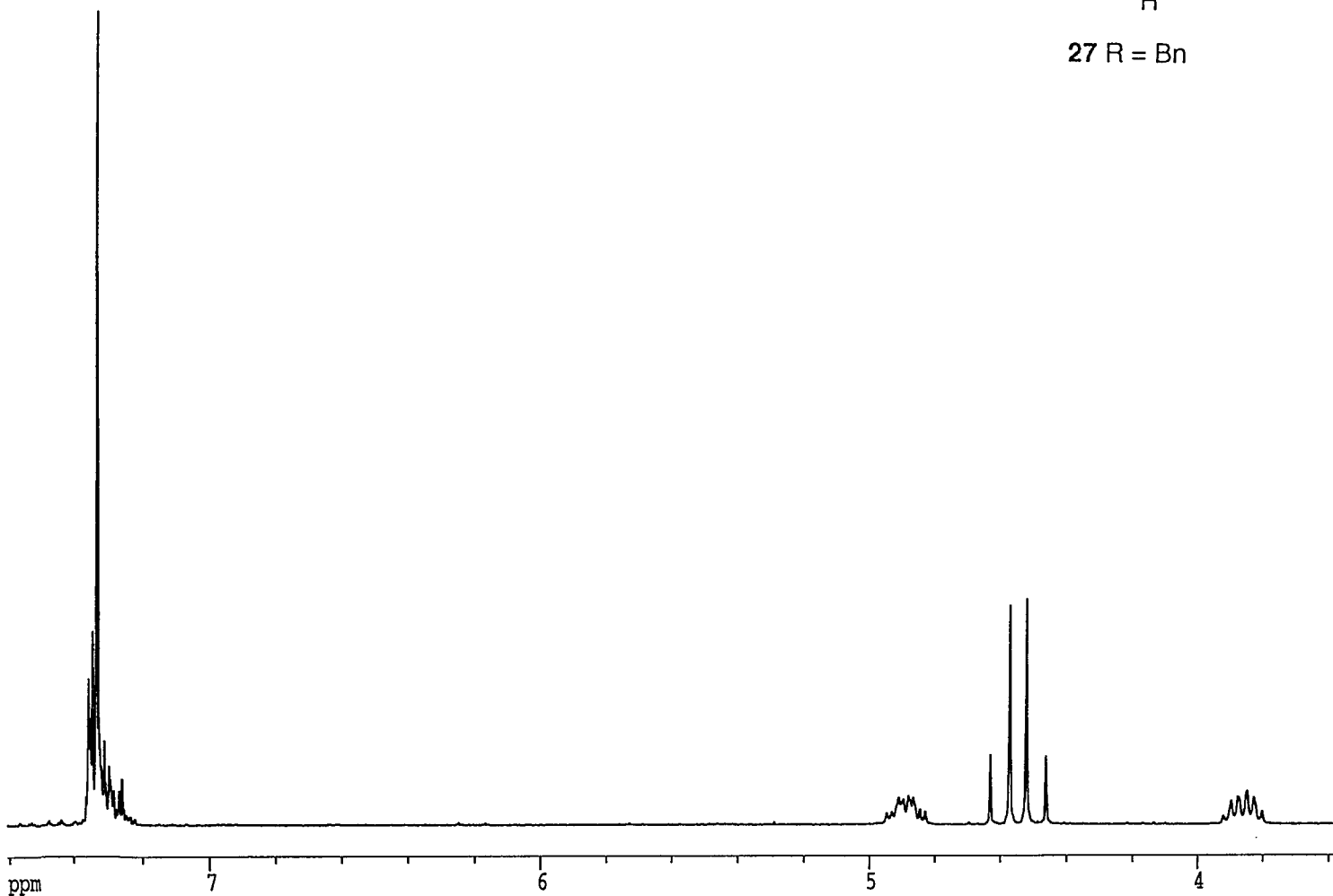
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 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 16
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HL1 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SF01 200.1332390 MHz
 NUCLEUS unknown

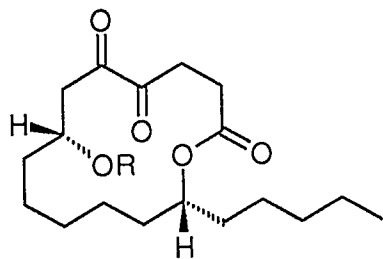
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1D NMR plot parameters
 CX 20.00 cm
 F1P 7.611 ppm
 F1 1523.30 Hz
 F2P 3.557 ppm
 F2 711.81 Hz
 PPMCM 0.20274 ppm/cm
 HZCM 40.57460 Hz/cm



27 R = Bn





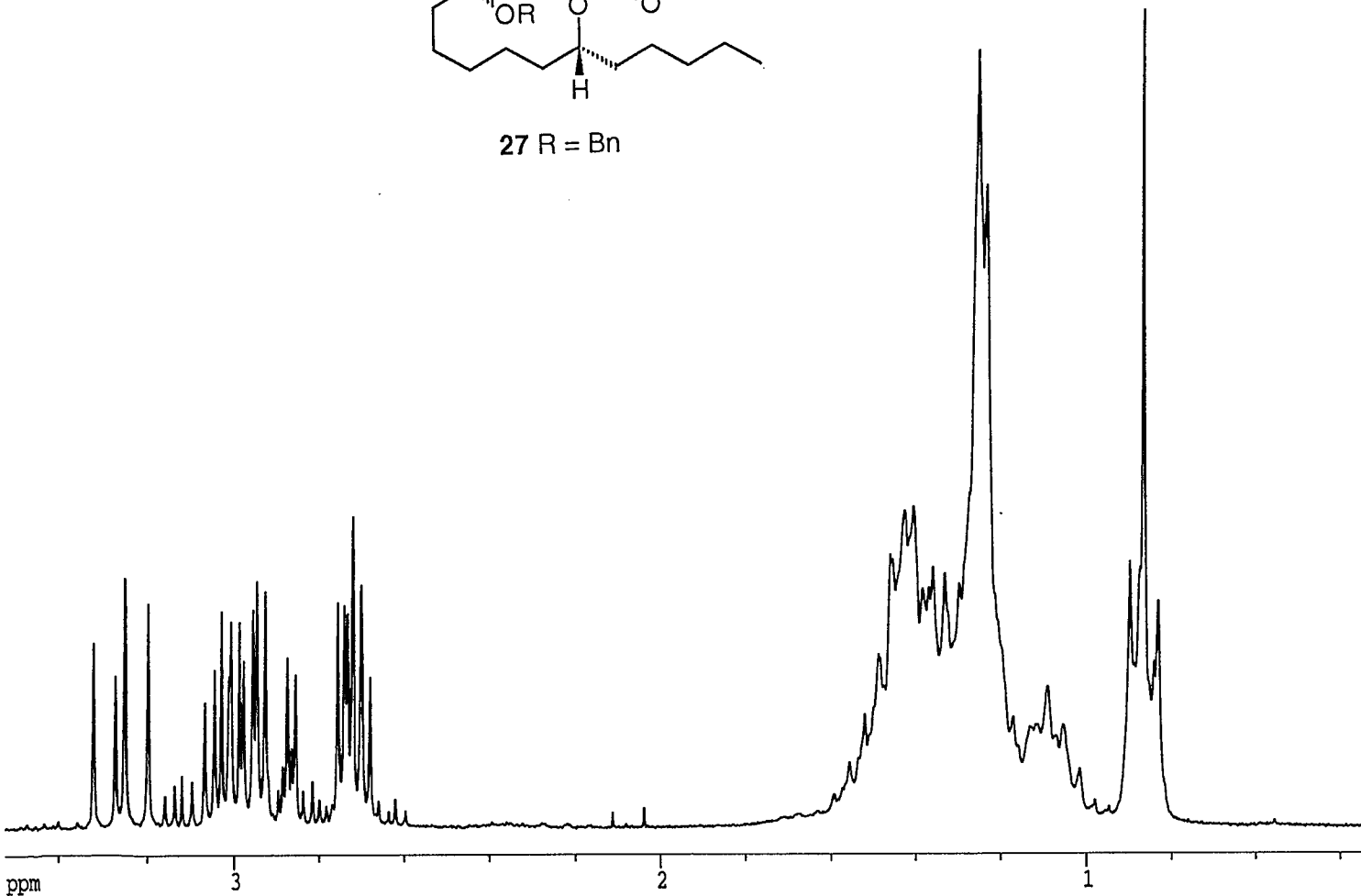
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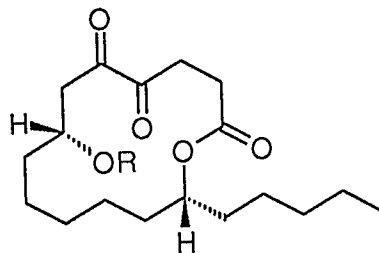
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 PROCNO 1
 DU mpi
 USER kl

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 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 16
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HL1 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SF01 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 200.1323407 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 3.532 ppm
 F1 706.77 Hz
 F2P 0.333 ppm
 F2 66.65 Hz
 PPMCM 0.15992 ppm/cm
 HZCM 32.00605 Hz/cm





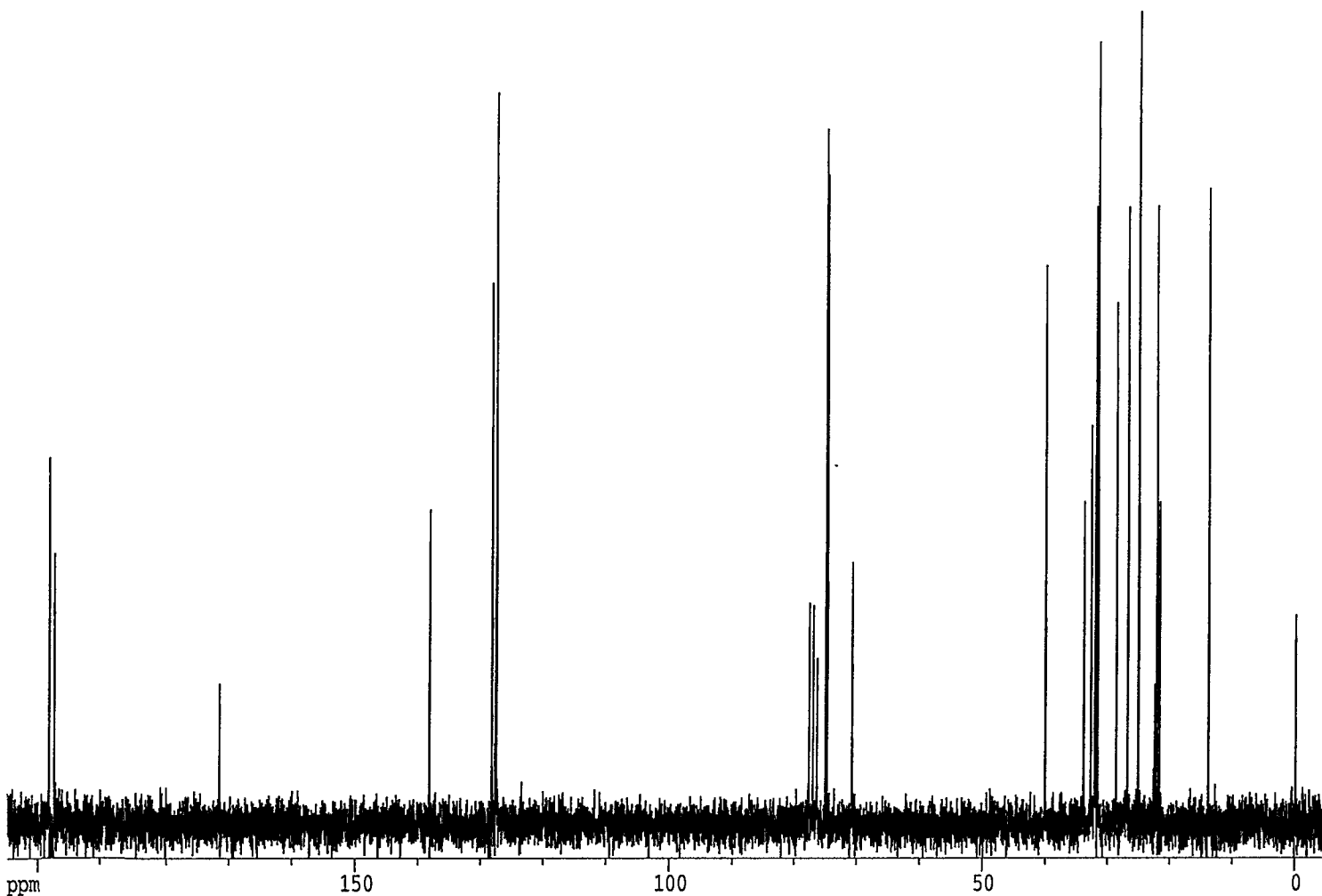
27 R = Bn

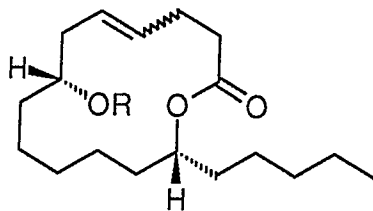
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 EXPNO 134
 PROCNO 1
 DU mpi
 USER kl

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 Time 6.12
 INSTRUM ac200
 PROBHD
 PULPROG X53.AU
 TD 32768
 SOLVENT unknown
 NS 1568
 DS 0
 SWH 14285.714 Hz
 FIDRES 0.435965 Hz
 AQ 1.1469300 sec
 RG 400
 DW 35.000 usec
 DE 46.30 usec
 TE 300.0 K
 PI 15.50 usec
 HLL 20 dB
 D1 0.00100000 sec
 DE 46.30 usec
 SF01 50.3287650 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 50.3233171 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 205.335 ppm
 F1 10333.13 Hz
 F2P -5.736 ppm
 F2 -288.63 Hz
 PPMCM 10.55352 ppm/cm
 HZCM 531.08813 Hz/cm





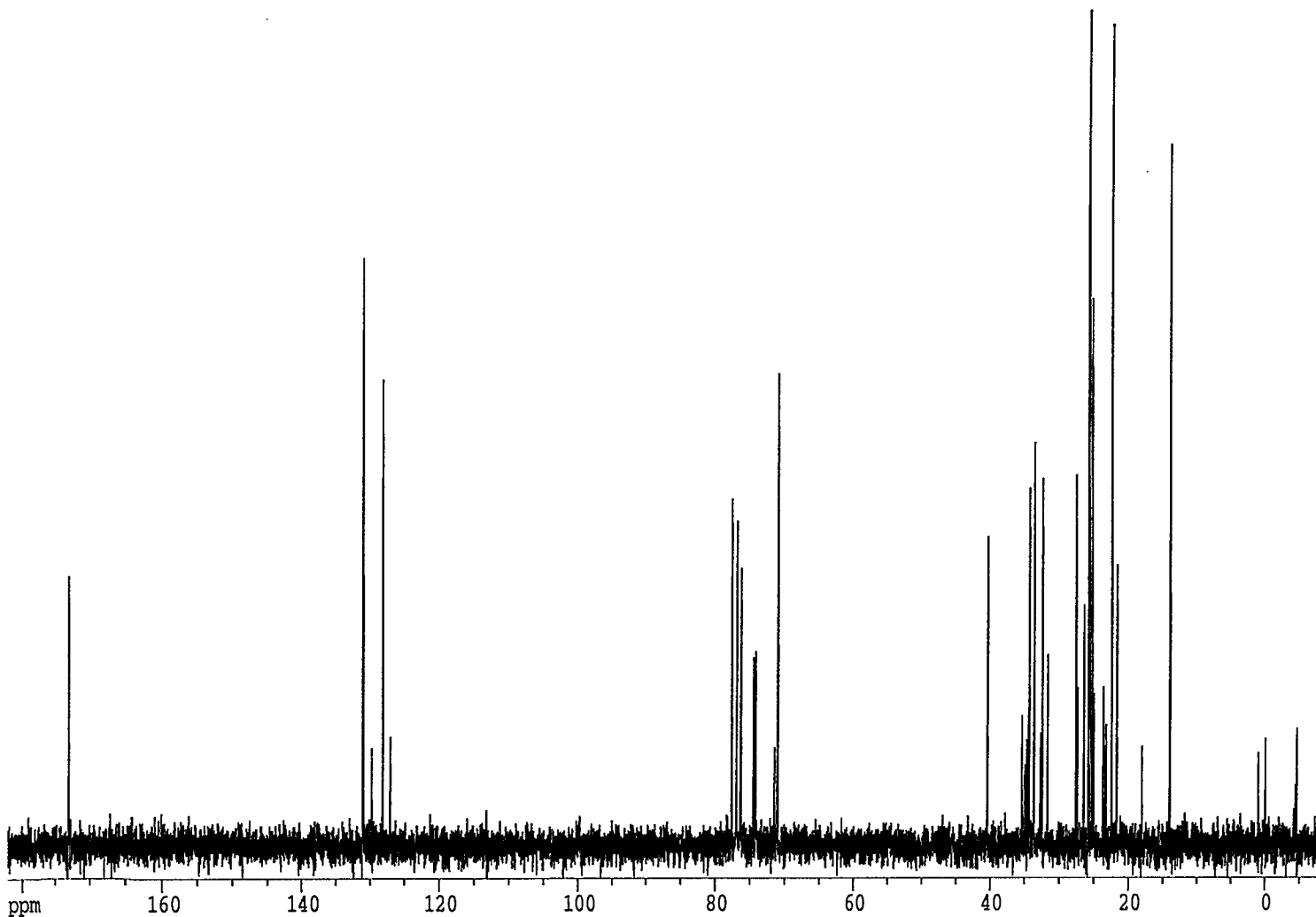
26 R = SiMe₂tBu (*E* : *Z* = 2.7 : 1)

Current Data Parameters
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 EXPNO 104
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
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 Time 12.45
 INSTRUM ac200
 PROBHD
 PULPROG X53.AU
 TD 32768
 SOLVENT unknown
 NS 1568
 DS 0
 SWH 14285.714 Hz
 FIDRES 0.435965 Hz
 AQ 1.1469300 sec
 RG 640
 DW 35.000 usec
 DE 46.30 usec
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 SF01 50.3287650 MHz
 NUCLEUS unknown

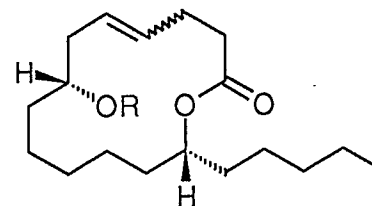
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 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
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 F1P 182.433 ppm
 F1 9180.62 Hz
 F2P -8.520 ppm
 F2 -428.77 Hz
 PPMCM 9.54765 ppm/cm
 HZCM 480.46945 Hz/cm



Current Data Parameters
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 EXPNO 104
 PROCNO 1
 DU mpi
 USER kl

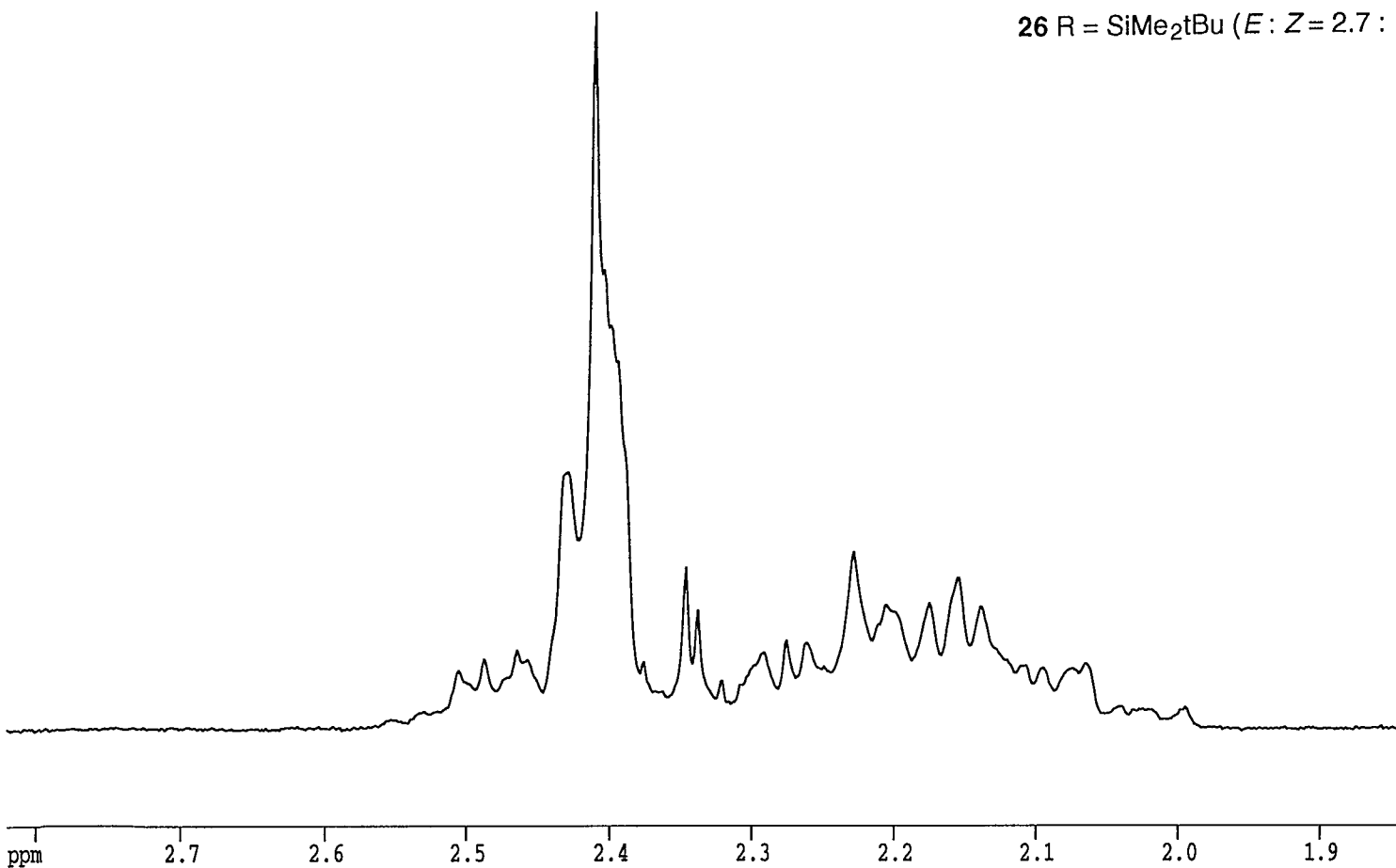
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 SOLVENT unknown
 NS 32
 DS 0
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 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 10
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HL1 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown



26 R = SiMe₂tBu (E : Z = 2.7 : 1)

F2 - Processing parameters
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 SF 200.1323405 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 2.821 ppm
 F1 564.61 Hz
 F2P 1.838 ppm
 F2 367.92 Hz
 PPMCM 0.04914 ppm/cm
 HZCM 9.83477 Hz/cm

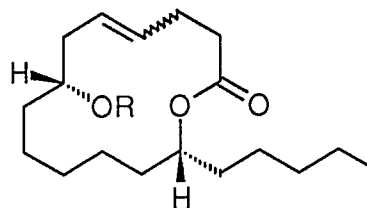


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 USER kl

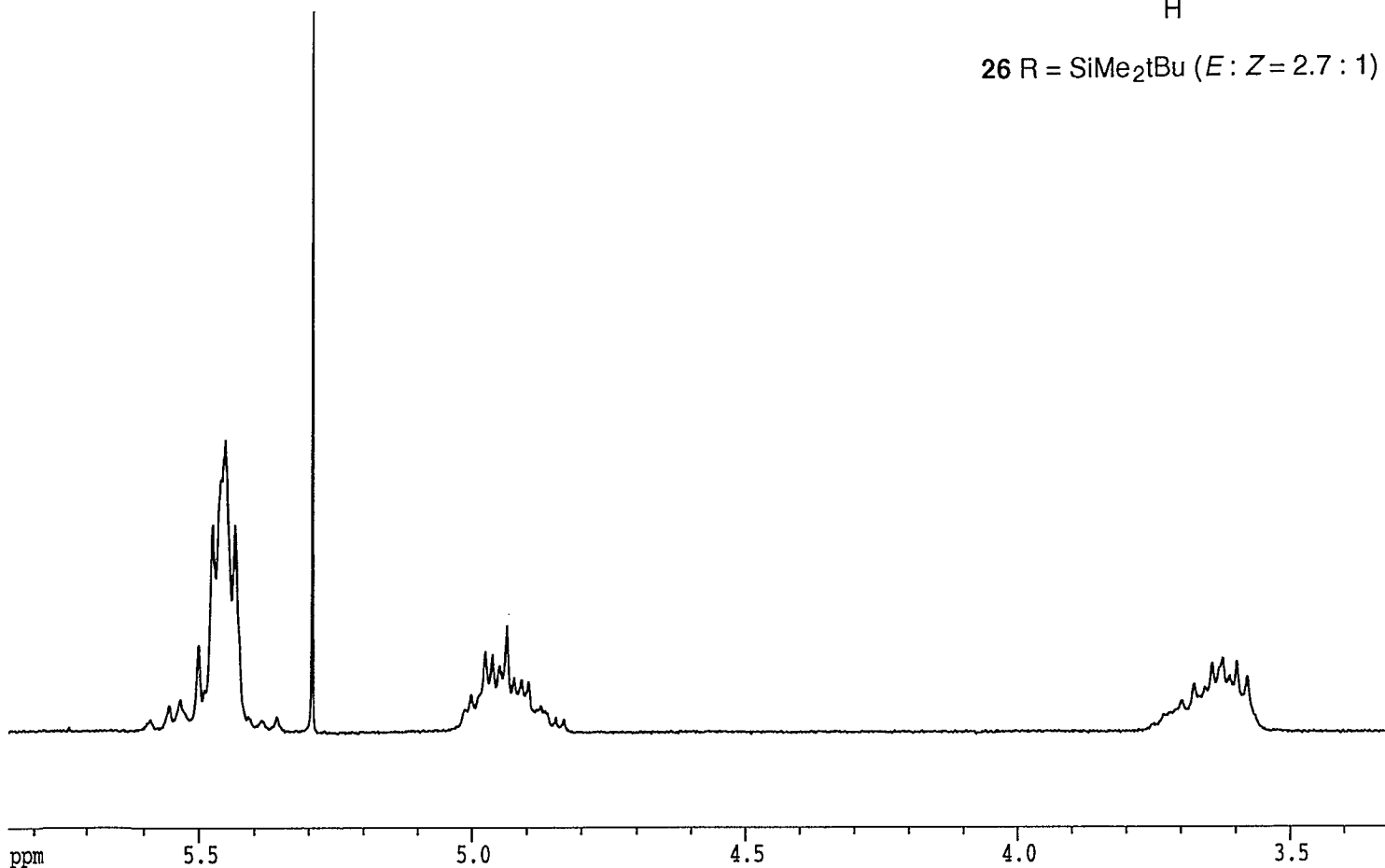
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 DS 0
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 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 10
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 PI 10.10 usec
 HL1 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
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 NUCLEUS unknown

F2 - Processing parameters
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 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 5.849 ppm
 F1 1170.65 Hz
 F2P 3.306 ppm
 F2 661.58 Hz
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 HZCM 25.45363 Hz/cm



26 R = SiMe₂tBu (E : Z = 2.7 : 1)

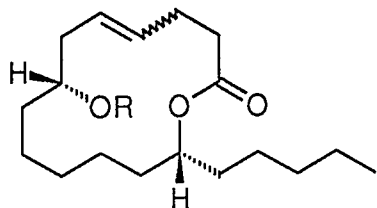


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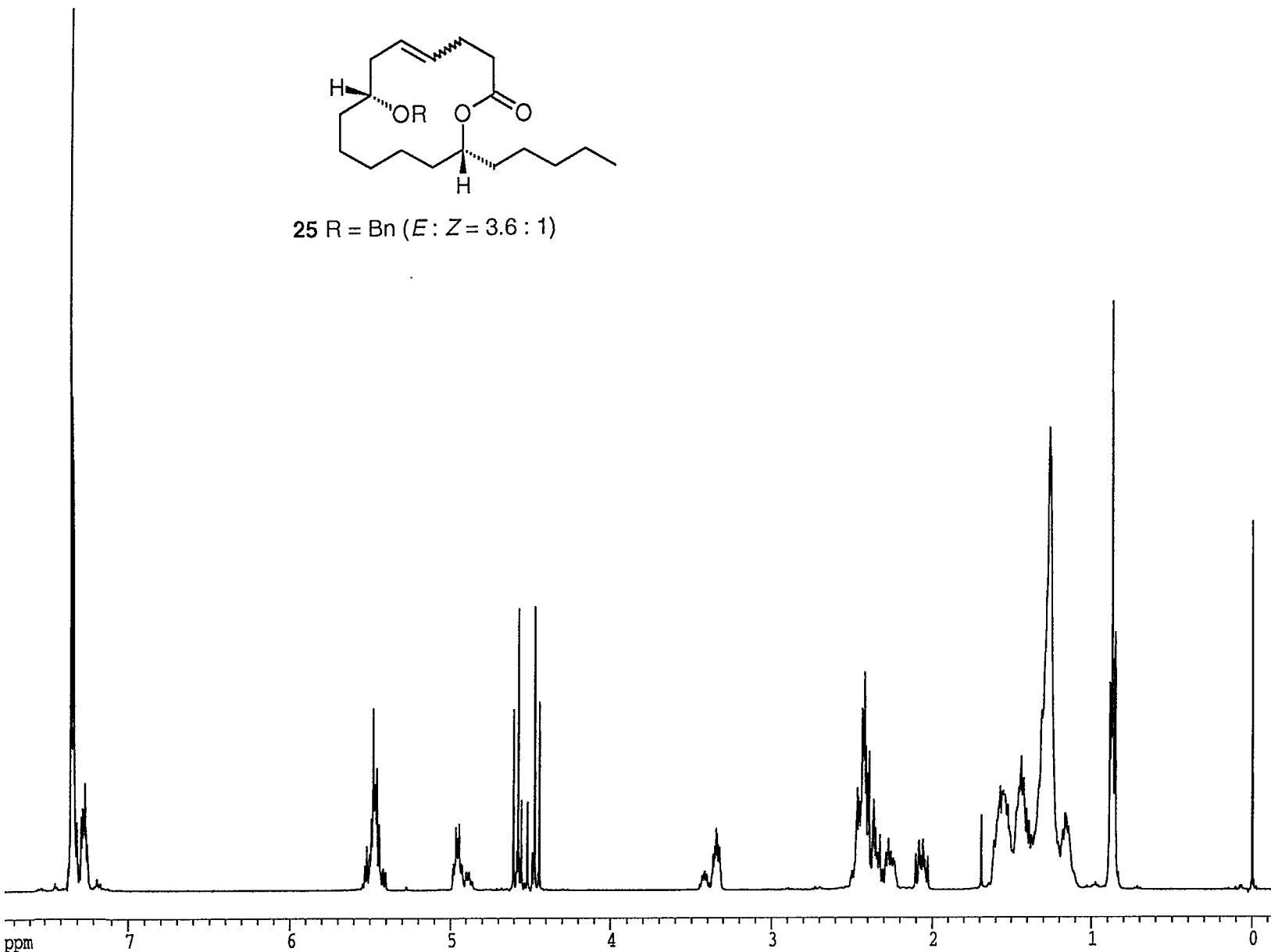
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 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 32
 DS 2
 SWH 8064.474 Hz
 FIDRES 0.246108 Hz
 AQ 2.0316660 sec
 RG 32
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 HL1 1 dB
 D1 2.0000000 sec
 P1 5.60 usec
 DE 88.57 usec
 SF01 400.1368403 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 400.1343978 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

1D NMR plot parameters
 CX 22.00 cm
 F1P 7.775 ppm
 F1 3111.06 Hz
 F2P -0.188 ppm
 F2 -75.04 Hz
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 HZCM 144.82246 Hz/cm



25 R = Bn (E : Z = 3.6 : 1)

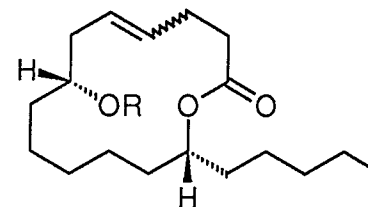


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 USER kl

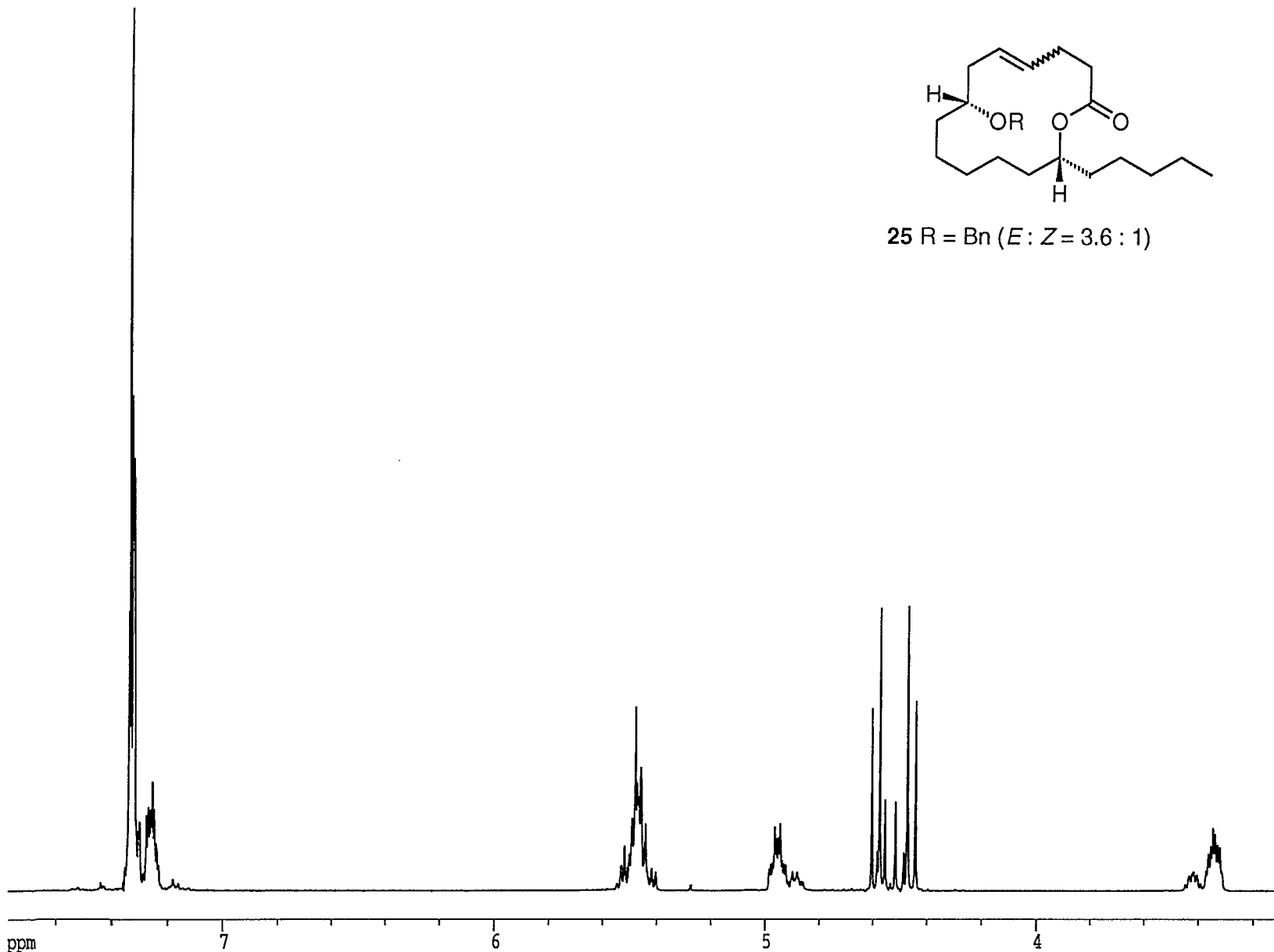
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 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 8064.474 Hz
 FIDRES 0.246108 Hz
 AQ 2.0316660 sec
 RG 32
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 HL1 1 dB
 D1 2.0000000 sec
 P1 5.60 usec
 DE 88.57 usec
 SFO1 400.1368403 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 400.1343978 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

1D NMR plot parameters
 CX 22.00 cm
 F1P 7.792 ppm
 F1 3117.90 Hz
 F2P 3.081 ppm
 F2 1232.83 Hz
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 HZCM 85.68504 Hz/cm



25 R = Bn (*E* : *Z* = 3.6 : 1)

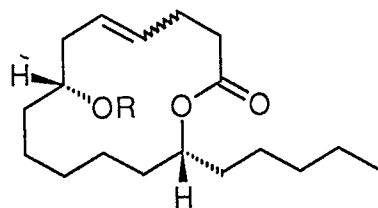


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 USER kl

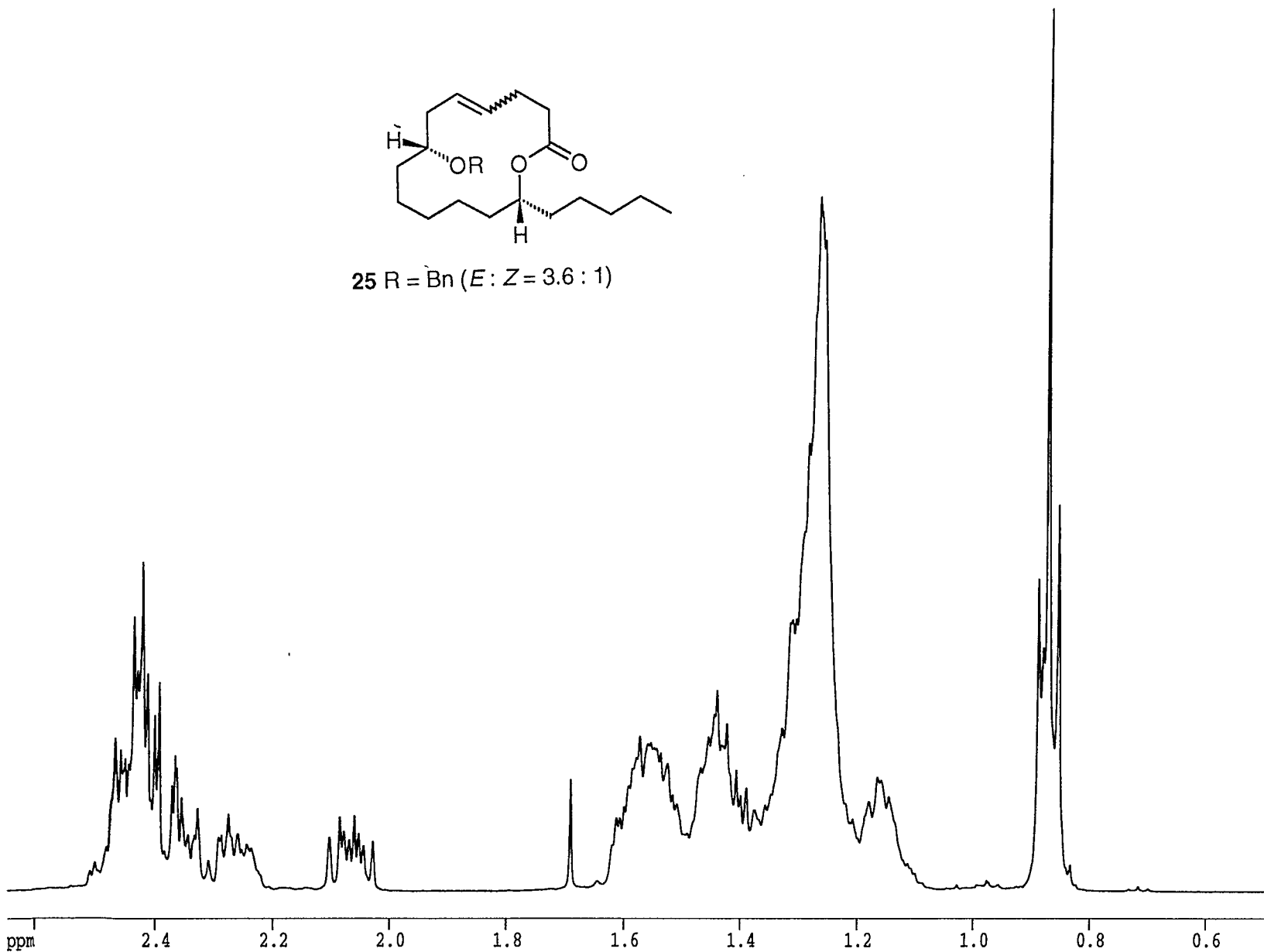
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 SOLVENT CDCl3
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 DS 2
 SWH 8064.474 Hz
 FIDRES 0.246108 Hz
 AQ 2.0316660 sec
 RG 32
 DW 62.000 usec
 DE 88.57 usec
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 SFO1 400.1368403 MHz
 NUCLEUS 1H

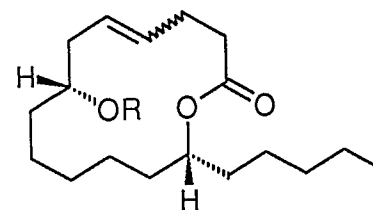
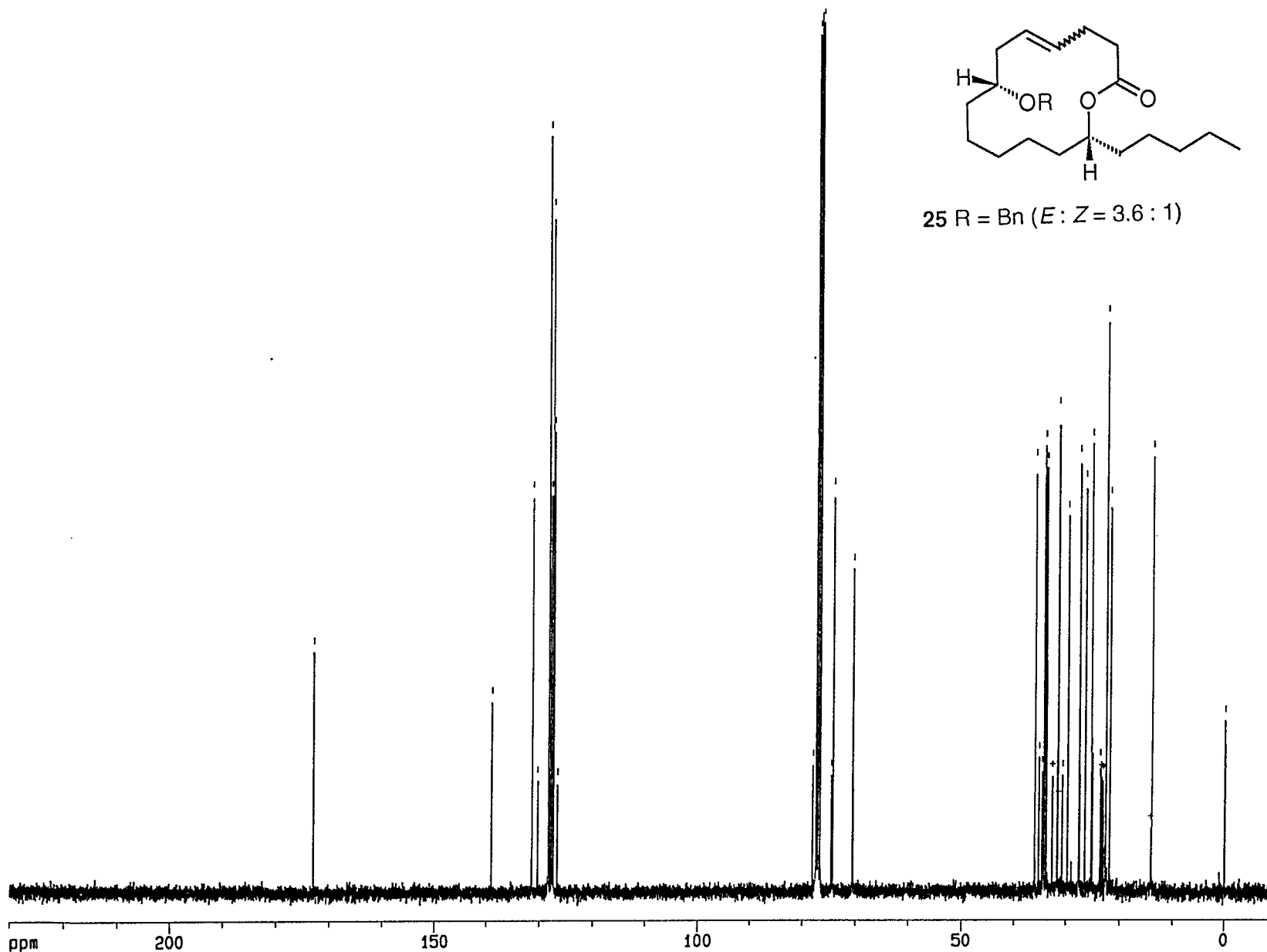
F2 - Processing parameters
 SI 32768
 SF 400.1343978 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

1D NMR plot parameters
 CX 22.00 cm
 F1P 2.653 ppm
 F1 1061.46 Hz
 F2P 0.486 ppm
 F2 194.53 Hz
 PPMCM 0.09848 ppm/cm
 HZCM 39.40596 Hz/cm



25 R = Bn (*E*:*Z* = 3.6 : 1)



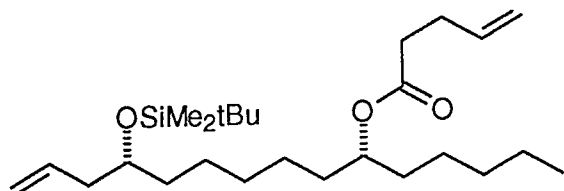


Current Data Parameters
 NAME LGM09102
 EXPNO 11
 PROCNO 1
 DU mpi
 USER wisno

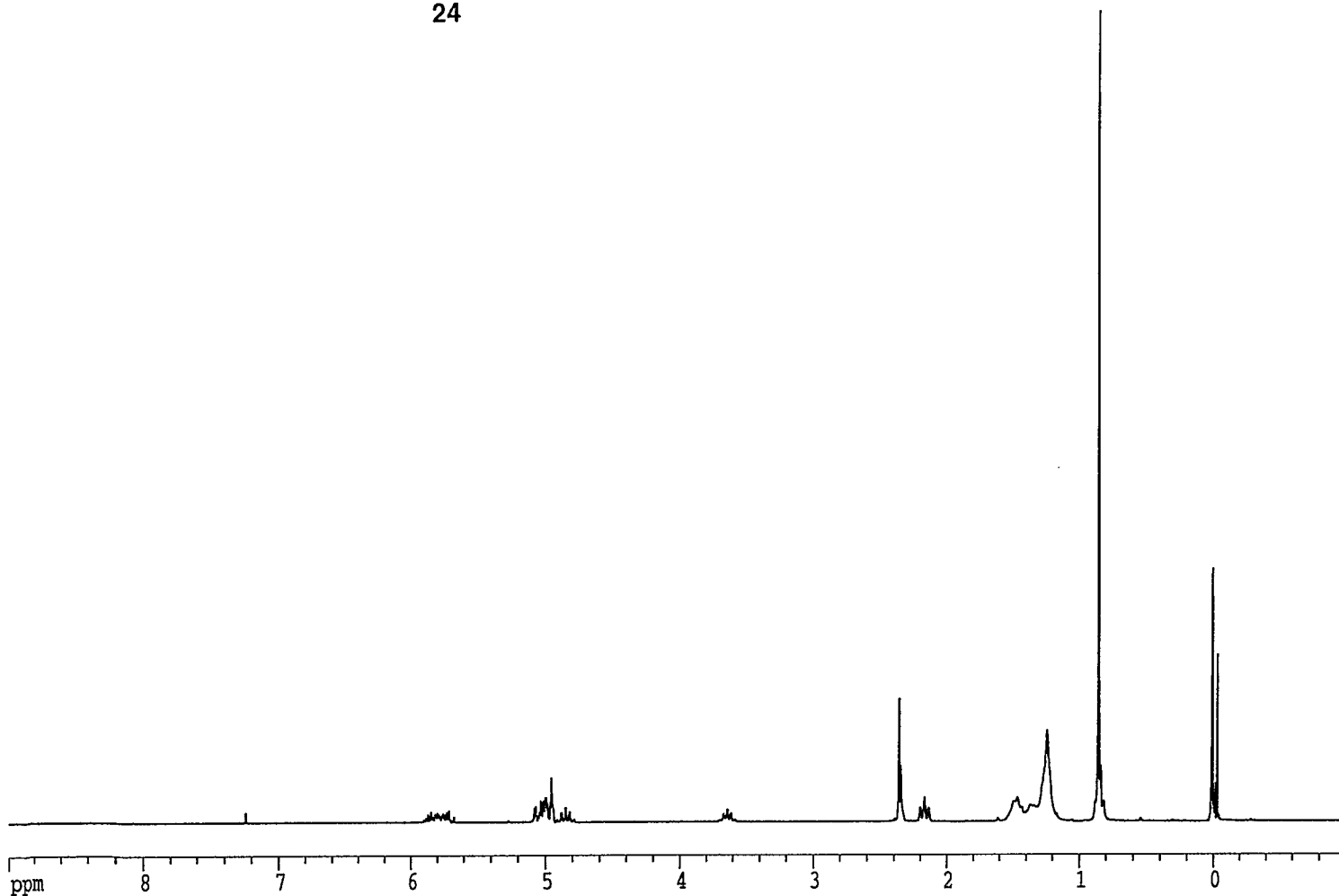
F2 - Acquisition Parameters
 Date_ 970129
 Time 3.33
 INSTRUM amx400
 PROBHD 5 mm Multinu
 PULPROG zgdc30
 TD 65536
 SOLVENT CDC13
 NS 43518 20000
 DS 300
 SWH 31249.838 Hz
 FIDRES 0.476835 Hz
 AQ 1.0486259 sec
 RG 16384
 DW 16.000 usec
 DE 22.86 usec
 TE 300.0 K
 O1 6399.74 Hz
 O2 6840.29 Hz
 D11 0.03000000 sec
 CPDPRG waltz16
 P31 100.00 usec
 S2 28 dB
 HL1 1 dB
 D1 0.01000000 sec
 P1 14.40 usec
 DE 22.86 usec
 SF01 100.6263997 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 65536
 SF 100.6138685 MHz
 HZpPT 0.476835 Hz
 SR -6131.55 Hz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.00 cm
 CY 15.00 cm
 F1P 230.000 ppm
 F1 23141.19 Hz
 F2P -10.000 ppm
 F2 -1006.14 Hz
 PPMCM 10.90909 ppm/cm
 HZCM 1097.60583 Hz/cm



24

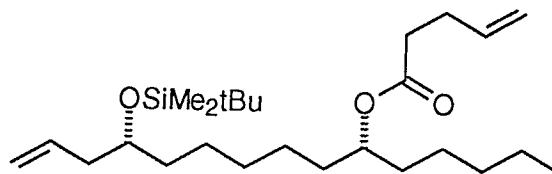


Current Data Parameters
 NAME AP240F
 EXPNO 137
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970425
 Time 11.09
 INSTRUM ac200
 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 8
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HL1 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SF01 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 200.1323448 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 1801.19 Hz
 F2P -1.000 ppm
 F2 -200.13 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 100.06617 Hz/cm



24

Current Data Parameters

NAME AP240F
 EXPNO 137
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters

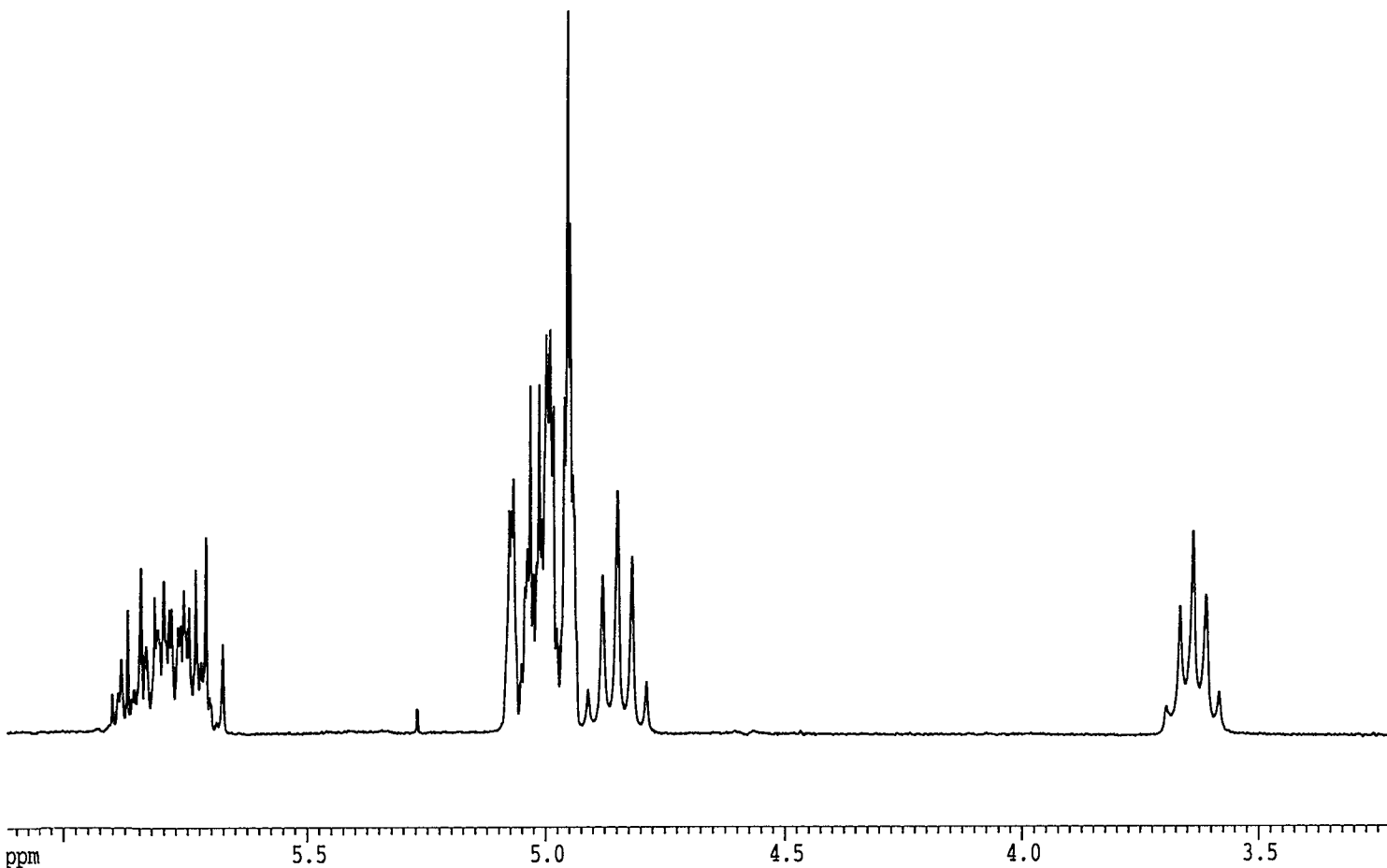
Date_ 970425
 Time 11.09
 INSTRUM ac200
 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 8
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HLL 83 dB
 D1 1.00000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown

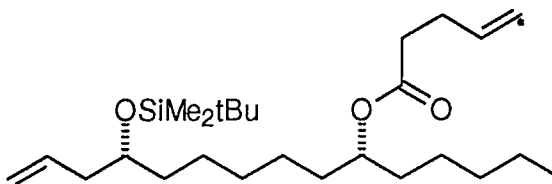
F2 - Processing parameters

SI 16384
 SF 200.1323448 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters

CX 20.00 cm
 F1P 6.130 ppm
 F1 1226.89 Hz
 F2P 3.209 ppm
 F2 642.21 Hz
 PPMCM 0.14607 ppm/cm
 HZCM 29.23387 Hz/cm





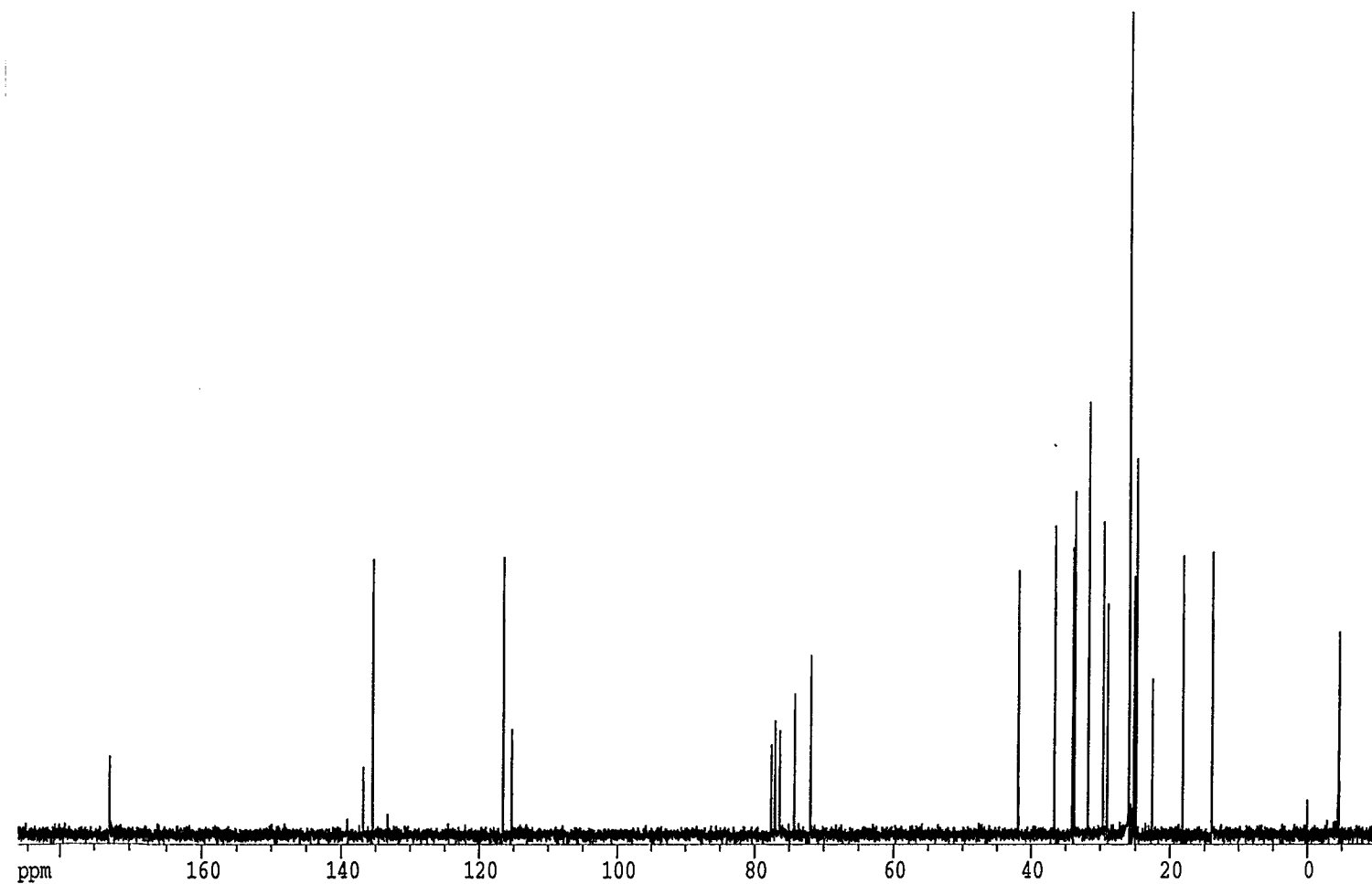
24

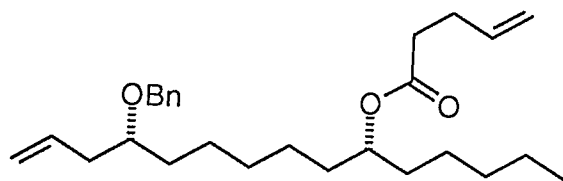
Current Data Parameters
 NAME AP241F
 EXPNO 137
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970425
 Time 11.42
 INSTRUM ac200
 PROBHD
 PULPROG X53.AU
 TD 32768
 SOLVENT unknown
 NS 1568
 DS 0
 SWH 14285.714 Hz
 FIDRES 0.435965 Hz
 AQ 1.1469300 sec
 RG 640
 DW 35.000 usec
 DE 46.30 usec
 TE 300.0 K
 P1 15.50 usec
 HL1 20 dB
 D1 0.0010000 sec
 DE 46.30 usec
 SFO1 50.3287650 MHz
 NUCLEUS unknown

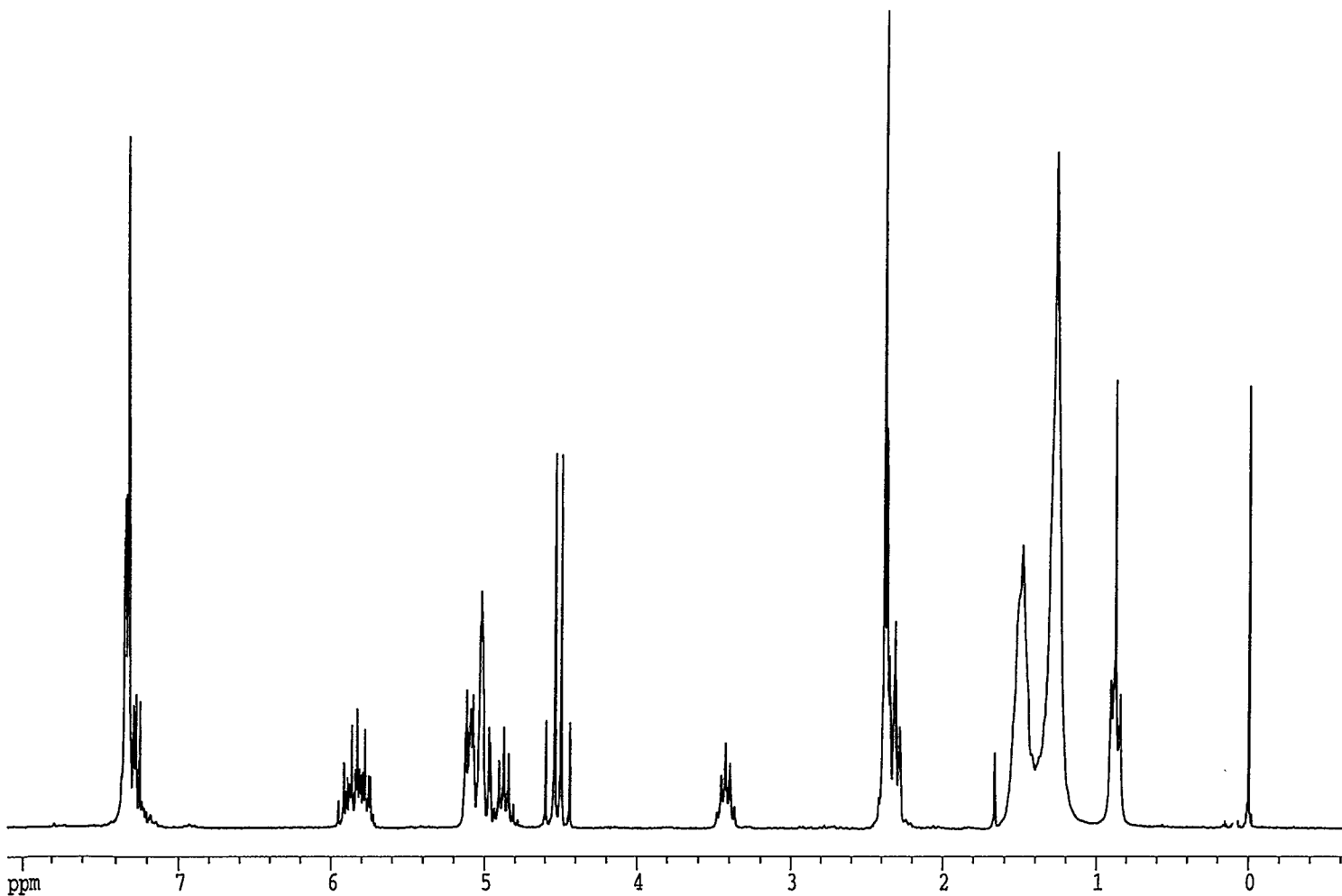
F2 - Processing parameters
 SI 16384
 SF 50.3233171 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 186.788 ppm
 F1 9399.81 Hz
 F2P -10.360 ppm
 F2 -521.33 Hz
 PPMCM 9.85740 ppm/cm
 HZCM 496.05722 Hz/cm





23



Current Data Parameters
 NAME JA220F
 EXPNO 106
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970122
 Time 12.20
 INSTRUM ac200
 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 4
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HLI 83 dB
 D1 1.00000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 200.1323432 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

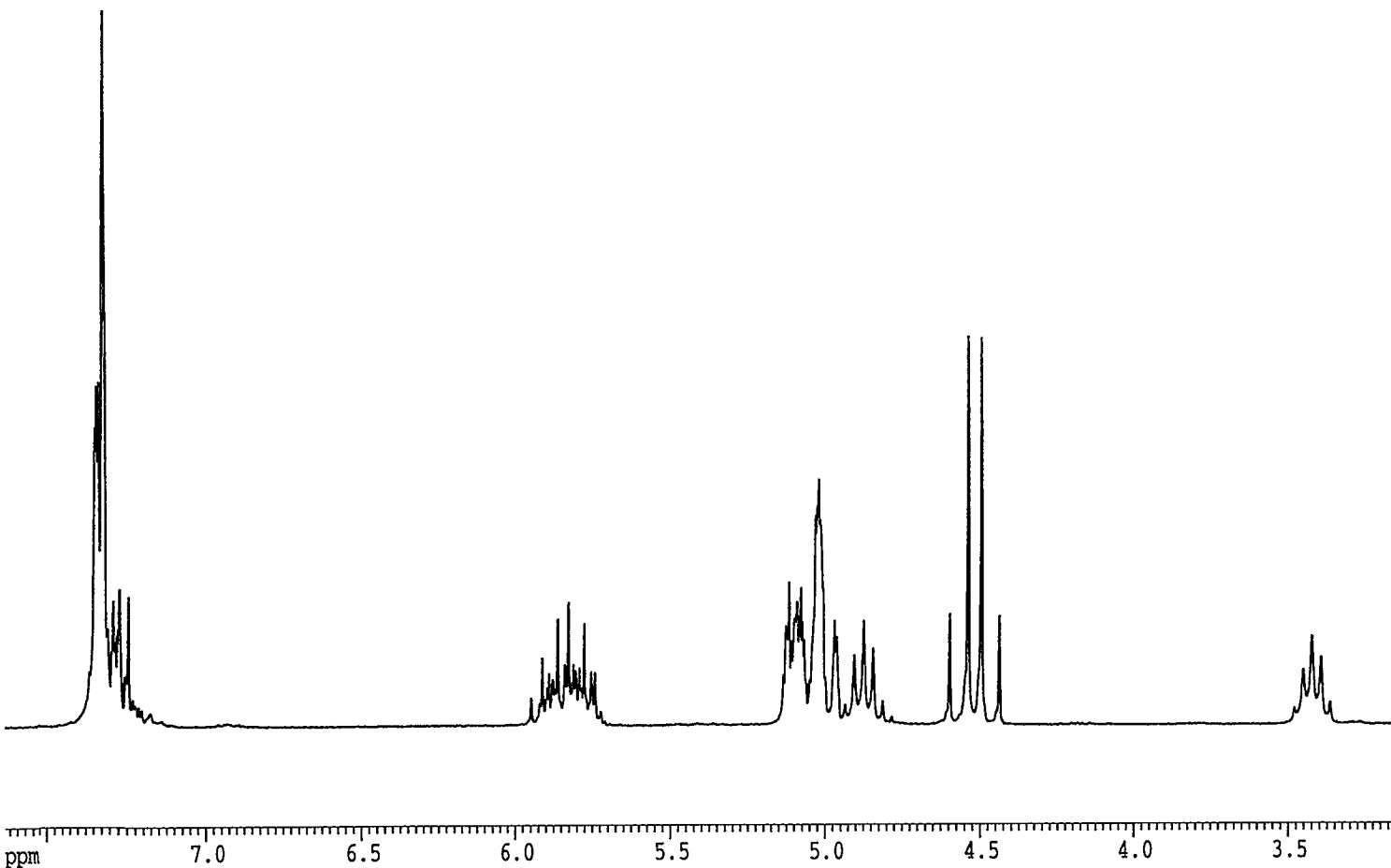
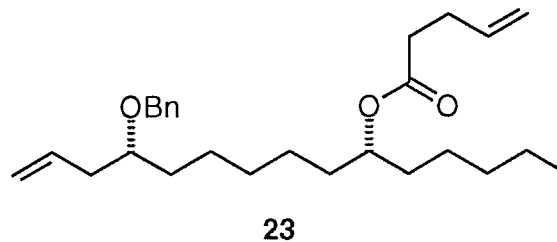
1D NMR plot parameters
 CX 20.00 cm
 F1P 8.110 ppm
 F1 1623.09 Hz
 F2P -0.658 ppm
 F2 -131.64 Hz
 PPMCM 0.43839 ppm/cm
 HZCM 87.73654 Hz/cm

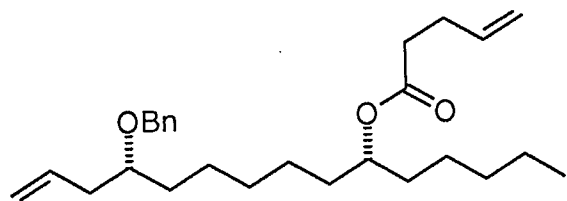
Current Data Parameters
 NAME JA220F
 EXPNO 106
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970122
 Time 12.20
 INSTRUM ac200
 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 4
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HLI 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 200.1323432 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 7.650 ppm
 F1 1530.92 Hz
 F2P 3.141 ppm
 F2 628.70 Hz
 PPMCM 0.22541 ppm/cm
 HZCM 45.11089 Hz/cm





23

Current Data Parameters

NAME JA221F
 EXPNO 106
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters

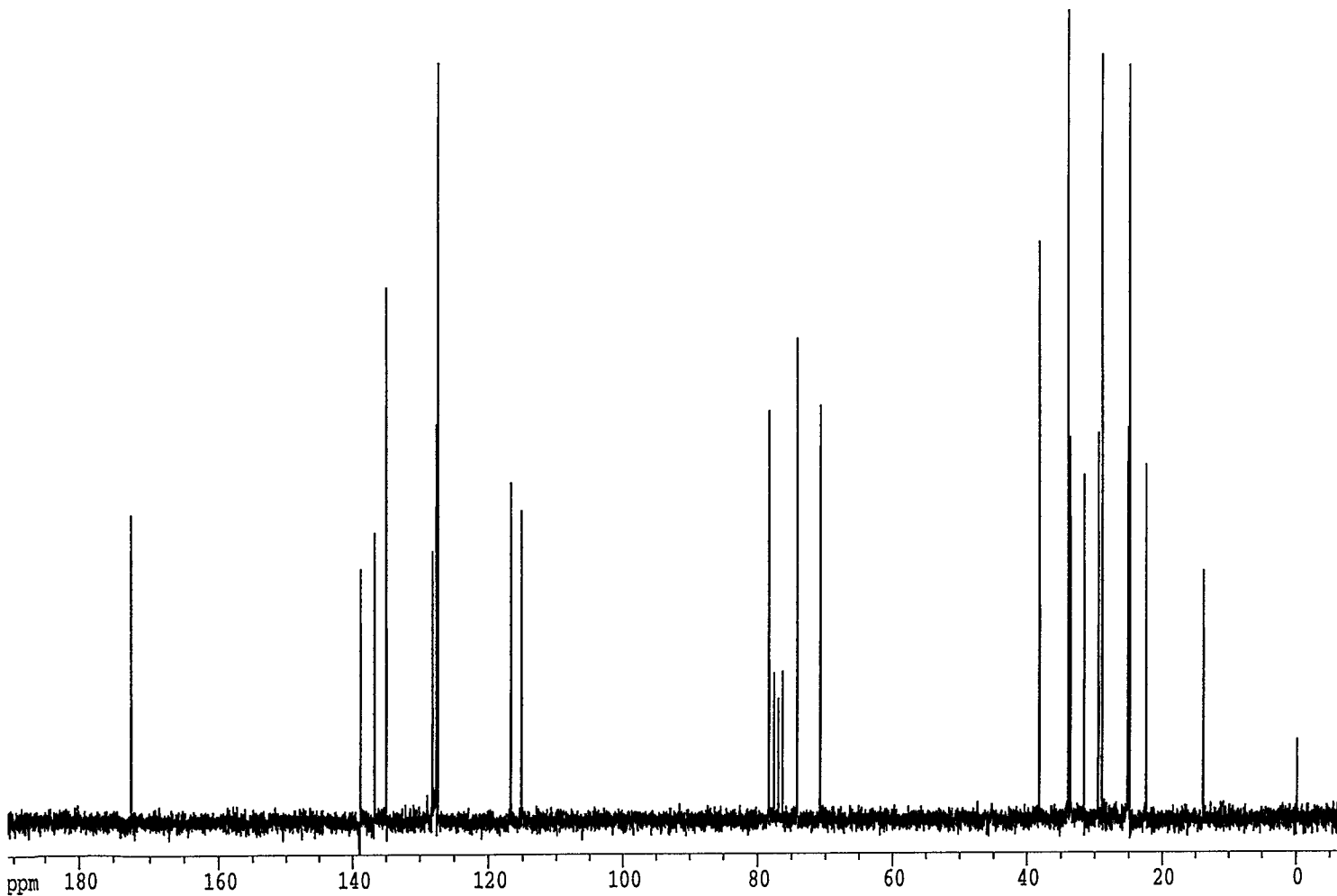
Date_ 970122
 Time 12.54
 INSTRUM ac200
 PROBHD
 PULPROG X53.AU
 TD 32768
 SOLVENT unknown
 NS 1568
 DS 0
 SWH 14285.714 Hz
 FIDRES 0.435965 Hz
 AQ 1.1469300 sec
 RG 640
 DW 35.000 usec
 DE 46.30 usec
 TE 300.0 K
 P1 15.50 usec
 HL1 20 dB
 D1 0.00100000 sec
 DE 46.30 usec
 SFO1 50.3287650 MHz
 NUCLEUS unknown

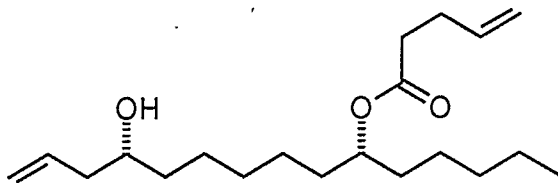
F2 - Processing parameters

SI 16384
 SF 50.3233198 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters

CX 20.00 cm
 F1P 191.474 ppm
 F1 9635.60 Hz
 F2P -7.132 ppm
 F2 -358.90 Hz
 PPMCM 9.93028 ppm/cm
 HZCM 499.72488 Hz/cm





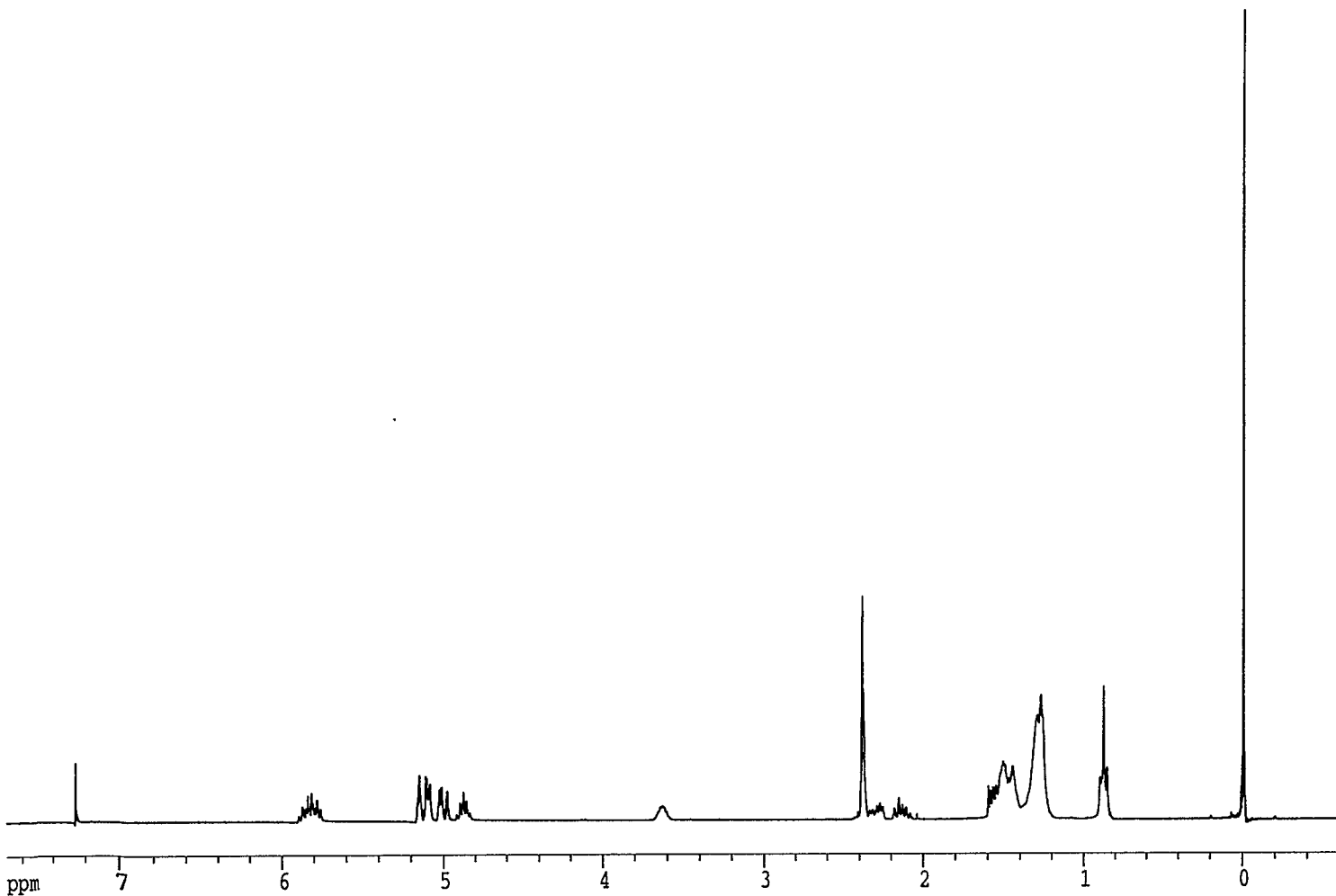
22

Current Data Parameters
 NAME jan07109
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970107
 Time 14.20
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6249.967 Hz
 FIDRES 0.190734 Hz
 AQ 2.6214900 sec
 RG 512
 DW 80.000 usec
 DE 100.00 usec
 TE 302.0 K
 HL1 0 dB
 D1 1.00000000 sec
 P1 6.75 usec
 DE 100.00 usec
 SF01 300.1349393 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1333665 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 7.713 ppm
 F1 2314.94 Hz
 F2P -0.627 ppm
 F2 -188.26 Hz
 PPMCM 0.41701 ppm/cm
 HZCM 125.15981 Hz/cm

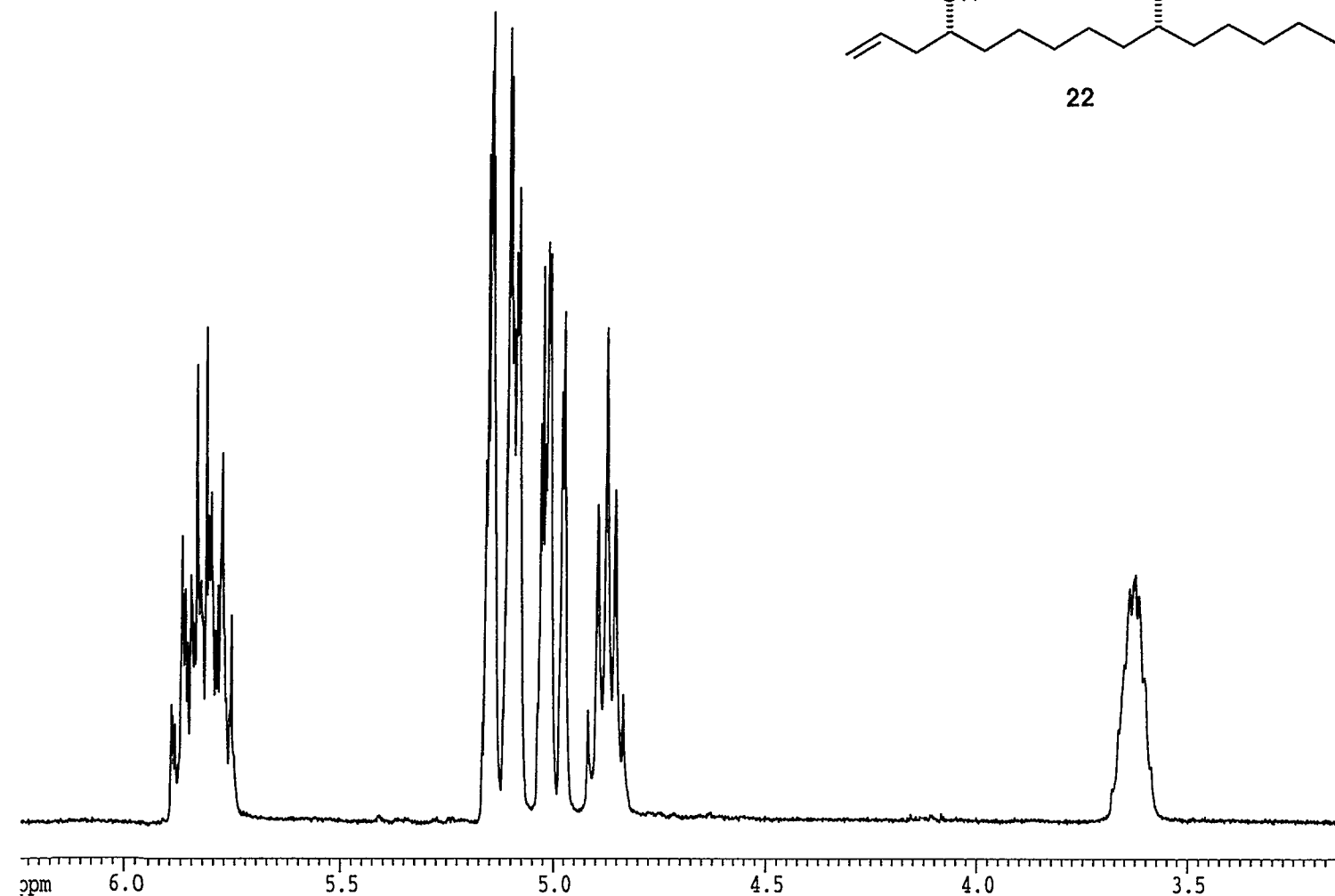
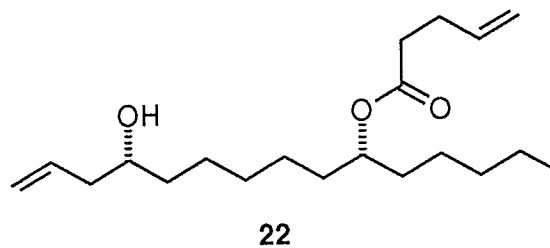


Current Data Parameters
NAME jan07109
EXPNO 10
PROCNO 1
DU mpi
USER kl

F2 - Acquisition Parameters
Date_ 970107
Time 14.20
INSTRUM amx300
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6249.967 Hz
FIDRES 0.190734 Hz
AQ 2.6214900 sec
RG 512
DW 80.000 usec
DE 100.00 usec
TE 302.0 K
HL1 0 dB
D1 1.00000000 sec
P1 6.75 usec
DE 100.00 usec
SF01 300.1349393 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 300.1333665 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 8.00

1D NMR plot parameters
CX 20.00 cm
F1P 6.256 ppm
F1 1877.49 Hz
F2P 3.132 ppm
F2 939.99 Hz
PPMCM 0.15618 ppm/cm
HZCM 46.87476 Hz/cm

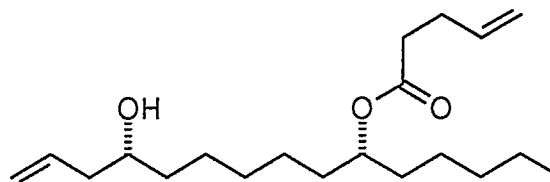


Current Data Parameters

NAME jan07109
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters

Date_ 970107
 Time 14.20
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6249.967 Hz
 FIDRES 0.190734 Hz
 AQ 2.621490 sec
 RG 512
 DW 80.000 usec
 DE 100.00 usec
 TE 302.0 K
 HL1 0 dB
 D1 1.00000000 sec
 P1 6.75 usec
 DE 100.00 usec
 SF01 300.1349393 MHz
 NUCLEUS 1H



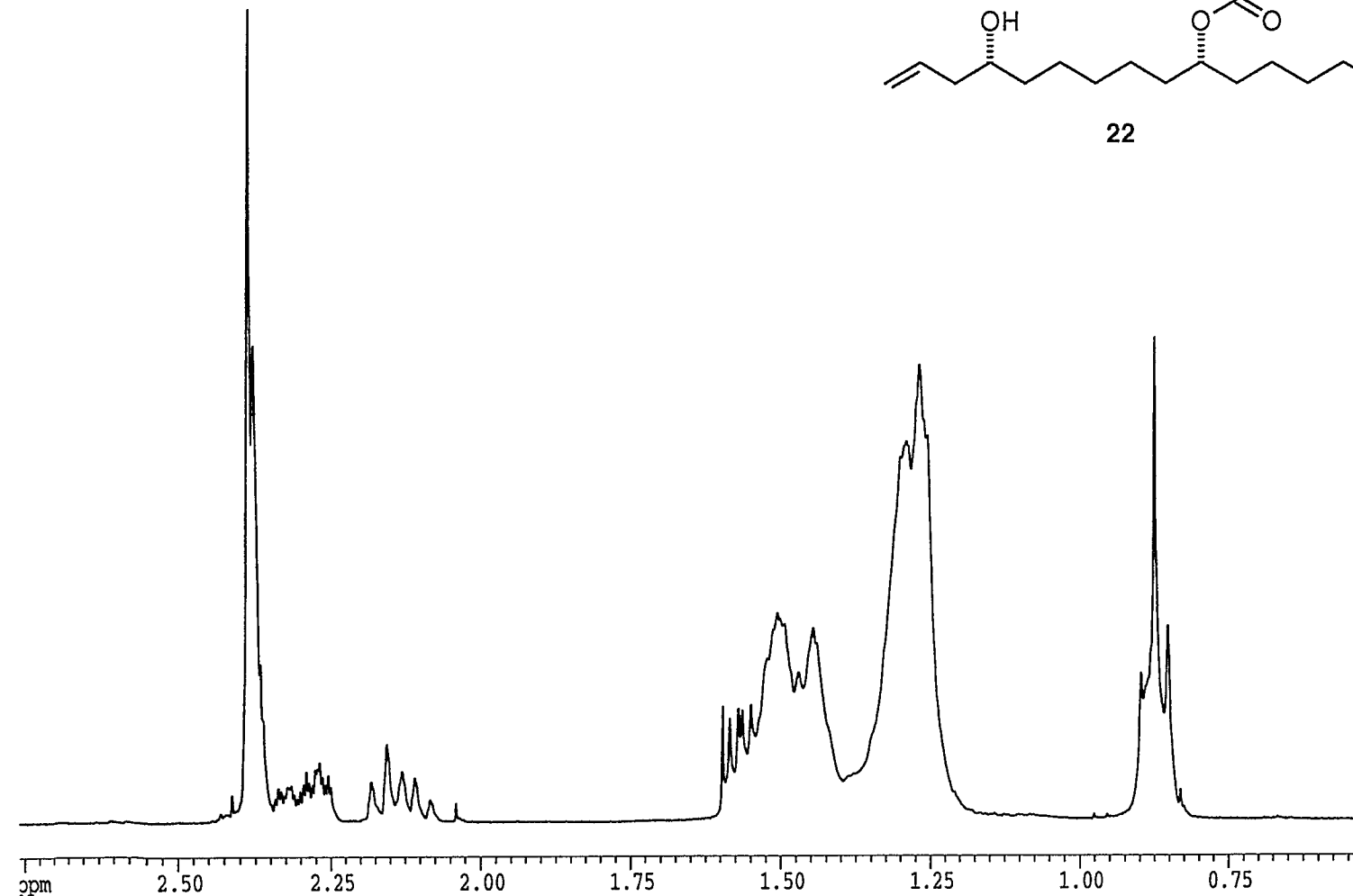
22

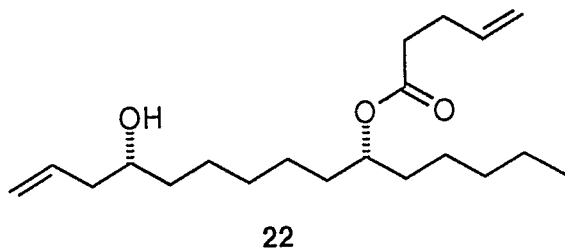
F2 - Processing parameters

SI 32768
 SF 300.1333665 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

1D NMR plot parameters

CX 20.00 cm
 F1P 2.768 ppm
 F1 830.62 Hz
 F2P 0.529 ppm
 F2 158.75 Hz
 PPMCM 0.11193 ppm/cm
 HZCM 33.59358 Hz/cm



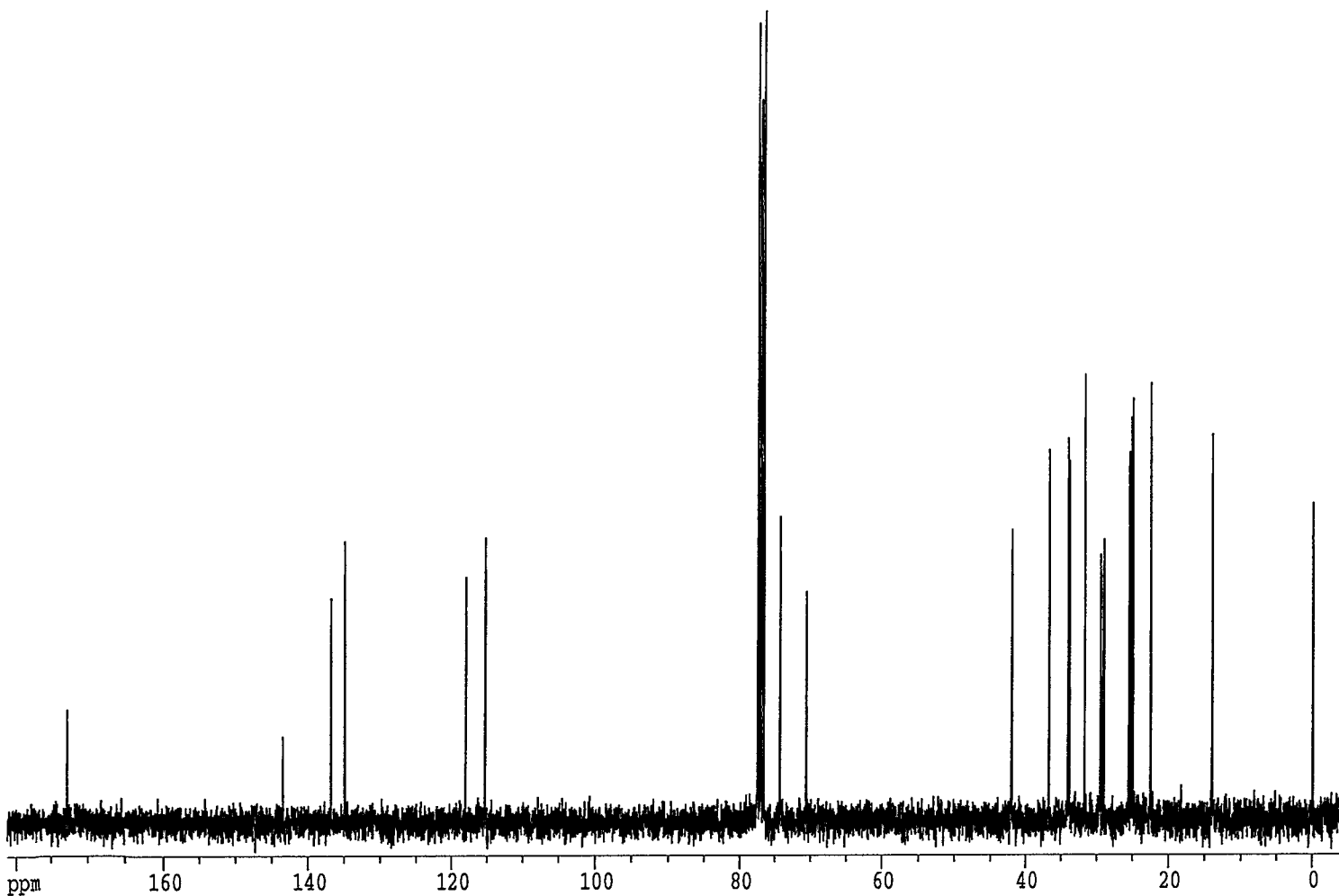


Current Data Parameters
 NAME jan07109
 EXPNO 11
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970107
 Time 14.29
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 2000
 DS 16
 SWH 33333.156 Hz
 FIDRES 0.508624 Hz
 AQ 0.9830900 sec
 RG 16384
 DW 15.000 usec
 DE 21.43 usec
 TE 302.0 K
 D11 0.03000000 sec
 CPDPRG waltz16
 P31 100.00 usec
 S2 27 dB
 HL1 0 dB
 D1 0.03000000 sec
 P1 5.68 usec
 DE 21.43 usec
 SFO1 75.4815977 MHz
 NUCLEUS 13C

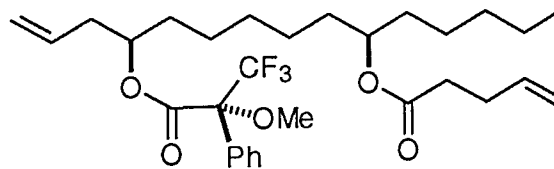
F2 - Processing parameters
 SI 32768
 SF 75.4685893 MHz
 WDW EM
 SSB 0
 LB 0.80 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 181.532 ppm
 F1 13700.00 Hz
 F2P -4.882 ppm
 F2 -368.41 Hz
 PPMCM 9.32071 ppm/cm
 HZCM 703.42047 Hz/cm



ppm

-71.976



¹⁹F NMR: Mosher ester of **22**

Current Data Parameters
 NAME apr04107
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date 500000
 Time 6.33
 PULPROG zgpg30
 SOLVENT CDCl3
 AQ 0.2621640 sec
 DW 4.0 usec
 RG 256
 NUCLEUS 19F
 SFO1 282.3891972 MHz
 SFO2 300.1341790 MHz
 TE 302.0 K
 HL1 0 dB
 D1 1.0000000 sec
 P31 100.0 usec
 S4 27 dB
 D11 0.0300000 sec
 S2 27 dB
 P1 8.0 usec
 DE 5.7 usec
 SFO1 282.3891972 MHz
 SWH 125000.01 Hz
 TD 65536
 NS 2000
 DS 16

F2 - Processing parameters
 SI 32768
 SF 282.4076971 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 5.00

1D NMR plot parameters
 CX 20.00 cm
 F1P -70.975 ppm
 F1 -20043.87 Hz
 F2P -73.069 ppm
 F2 -20635.23 Hz
 PPMCM 0.10470 ppm/cm
 HZCM 29.56800 Hz/cm

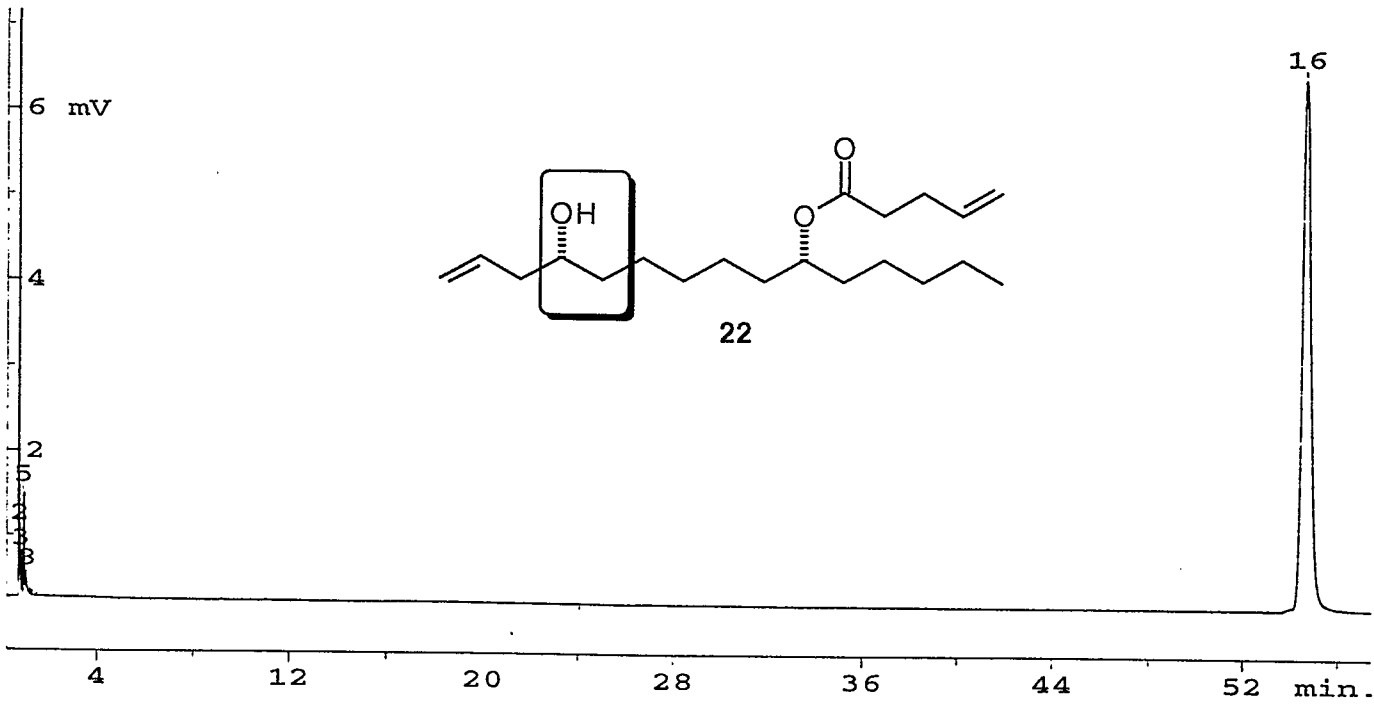
ppm

-71.5

-72.0

-72.5

-73.0



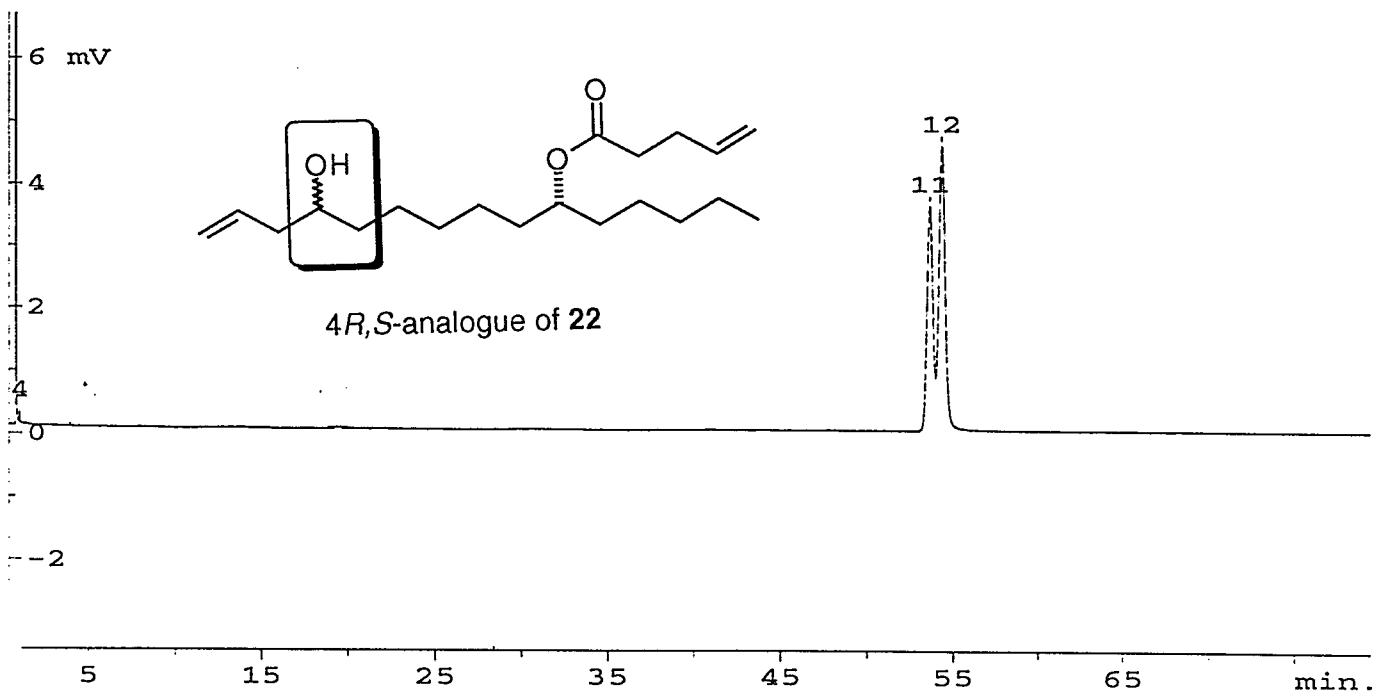
***** r127s844 ***** 23-Apr-97 11:45:36

Sample : LGM-LB-136-01-97-64382-K measured: 23-Apr-97 10:36:51
 A.-no. : 127 processed: 23-Apr-97 11:44:55
 by: GC
 with:

No.	min.	area-%
16	54.66	100.0

15 peaks had been suppressed.

Instrument : HEWLETT PACKARD 5890
 Column : 25MFS/20%TBCD(β)80%SE-54/N 3053 C
 Detector : FID
 Temperature : 230/160-180/0.2/MIN
 Gas : 0.8 BAR H2
 Sample size : 0.1
 Recorder : 0.2CM/MIN



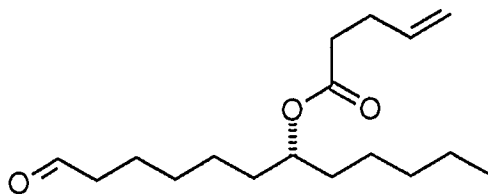
***** r127s449 ***** 24-Jan-97 10:20:08

Sample : LGM-LB-080-01-97-63473-K measured: 24-Jan-97 08:38:31
 A.-no. : 127 processed: 24-Jan-97 10:19:08
 by: GC
 with:

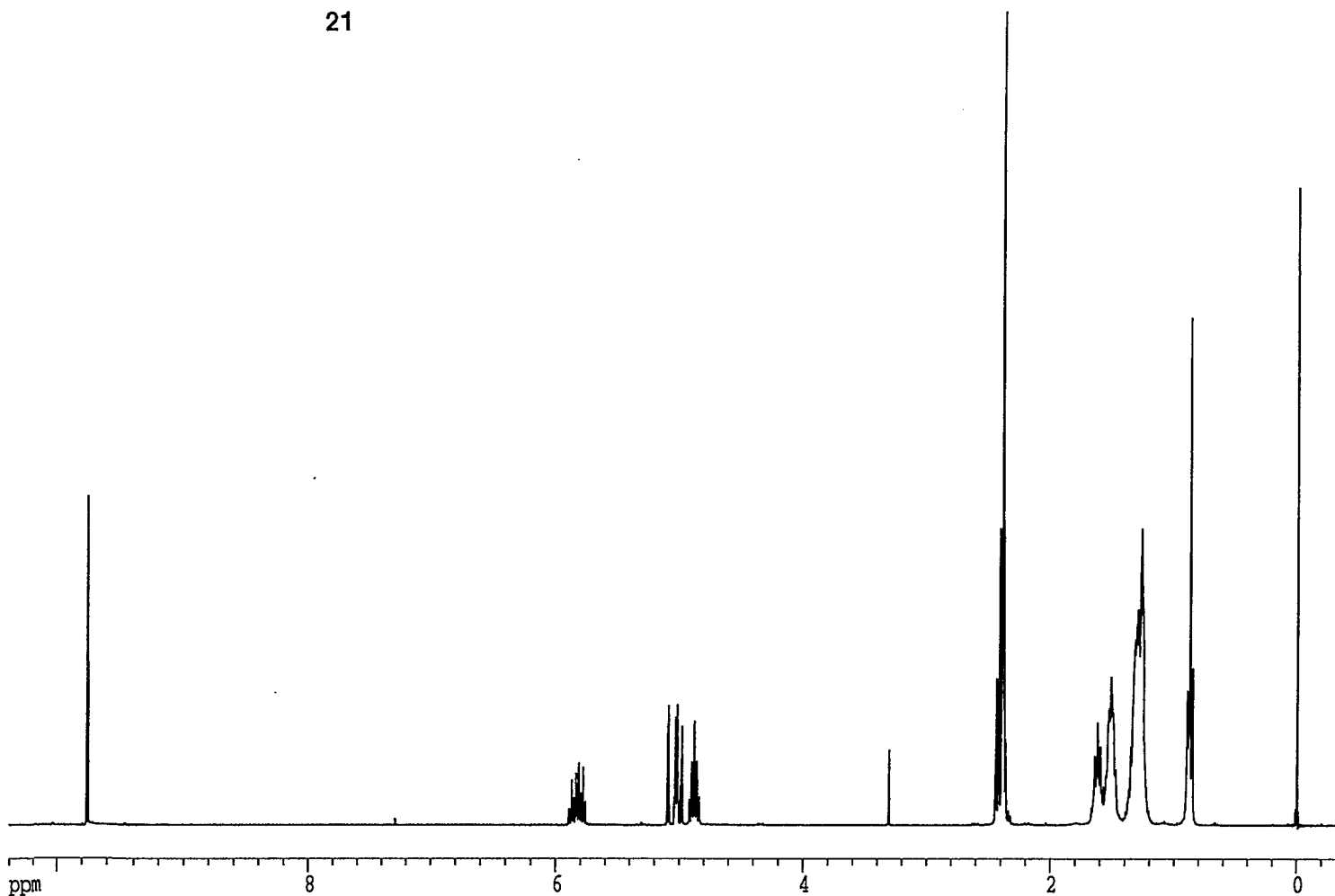
No.	min.	area-%
9	3.91	0.11
10	19.37	1.32
11	53.63	42.6
12	54.31	55.9

8 peaks had been suppressed.

Instrument : HEWLETT PACKARD 5890
 Column : 25MFS/20%TBCD/80%SE-54/N 3053
 Detector : FID
 Temperature : 250/160-180/0.2/MIN
 Gas : 0.8 BAR H2
 Sample size : 0.1
 Recorder : 0.2CM/MIN



21



Current Data Parameters
 NAME jan03101
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970103
 Time 10.21
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6249.967 Hz
 FIDRES 0.190734 Hz
 AQ 2.6214900 sec
 RG 128
 DW 80.000 usec
 DE 100.00 usec
 TE 302.0 K
 HL1 0 dB
 D1 1.00000000 sec
 P1 6.75 usec
 DE 100.00 usec
 SFO1 300.1349393 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1333577 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.416 ppm
 F1 3126.06 Hz
 F2P -0.384 ppm
 F2 -115.26 Hz
 PPMCM 0.53998 ppm/cm
 HZCM 162.06590 Hz/cm

Current Data Parameters

NAME jan03101
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters

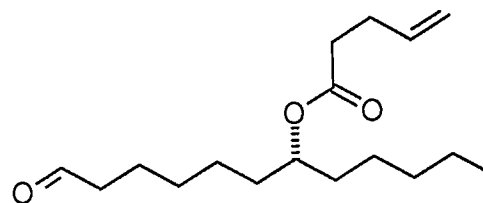
Date_ 970103
 Time 10.21
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 32
 DS 2
 SWH 6249.967 Hz
 FIDRES 0.190734 Hz
 AQ 2.6214900 sec
 RG 128
 DW 80.000 usec
 DE 100.00 usec
 TE 302.0 K
 HL1 0 dB
 D1 1.00000000 sec
 P1 6.75 usec
 DE 100.00 usec
 SF01 300.1349393 MHz
 NUCLEUS 1H

F2 - Processing parameters

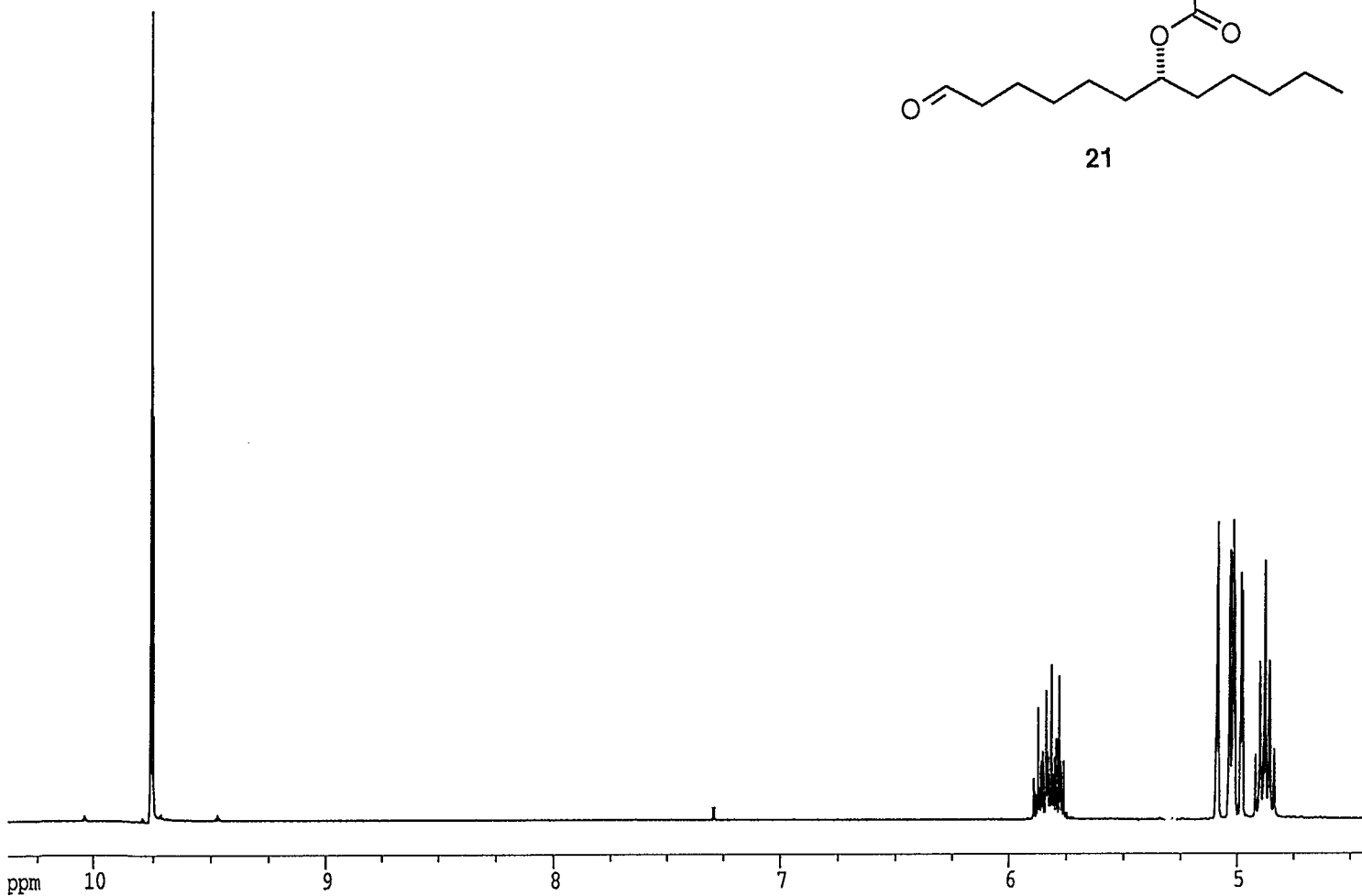
SI 32768
 SF 300.1333577 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

1D NMR plot parameters

CX 20.00 cm
 F1P 10.398 ppm
 F1 3120.67 Hz
 F2P 4.411 ppm
 F2 1323.81 Hz
 PPMCM 0.29934 ppm/cm
 HZCM 89.84328 Hz/cm



21

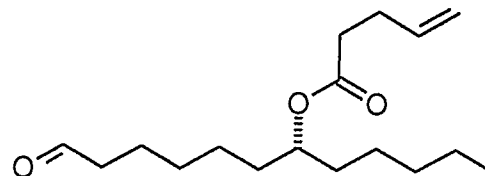


Current Data Parameters
NAME jan03101
EXPNO 10
PROCNO 1
DU mpi
USER kl

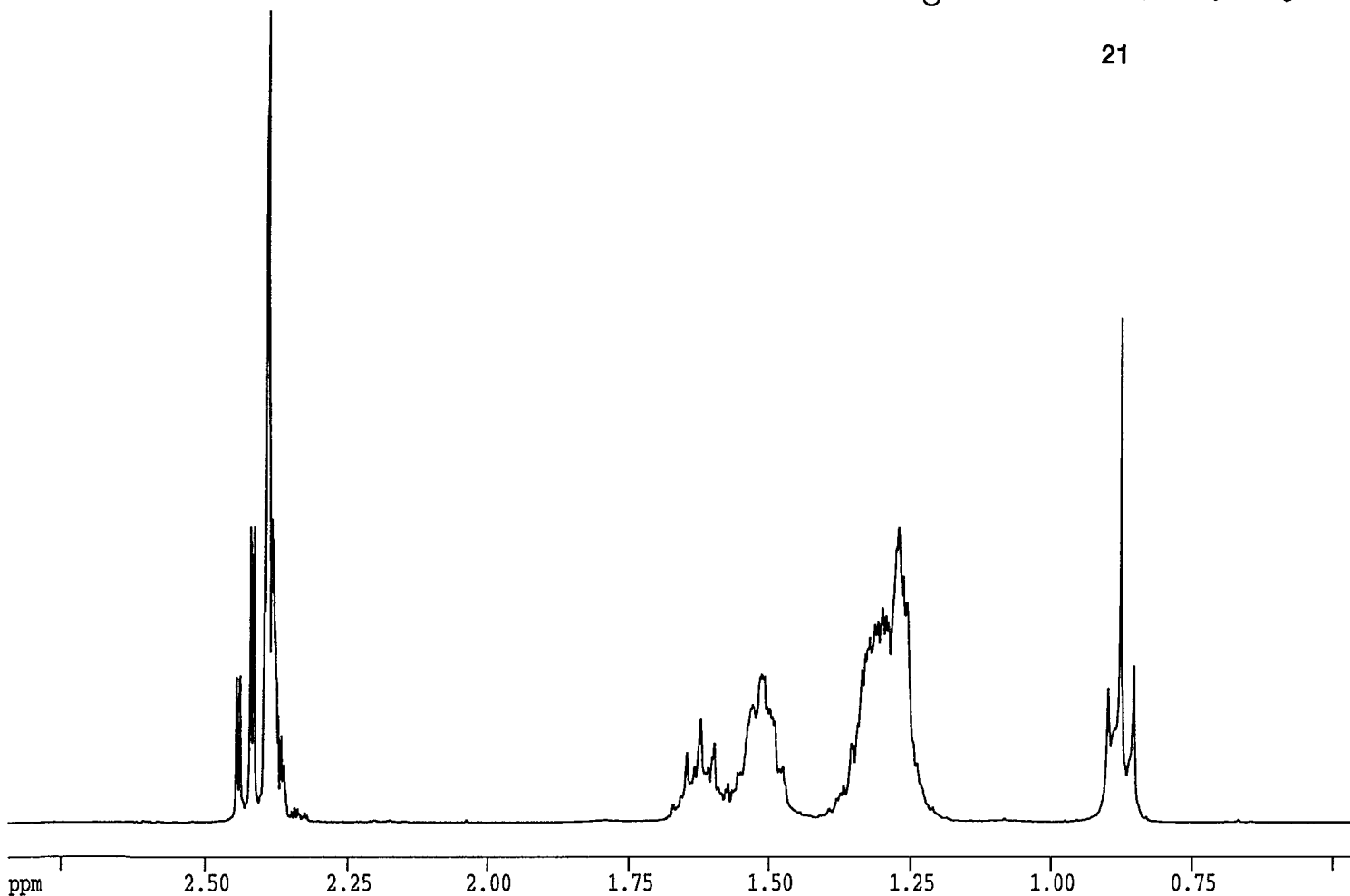
F2 - Acquisition Parameters
Date_ 970103
Time 10.21
INSTRUM amx300
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6249.967 Hz
FIDRES 0.190734 Hz
AQ 2.6214900 sec
RG 128
DW 80.000 usec
DE 100.00 usec
TE 302.0 K
HL1 0 dB
D1 1.00000000 sec
P1 6.75 usec
DE 100.00 usec
SFO1 300.1349393 MHz
NUCLEUS 1H

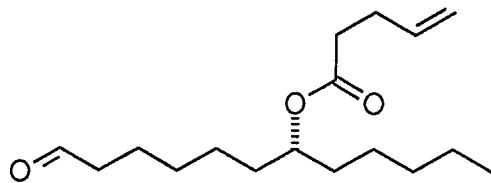
F2 - Processing parameters
SI 32768
SF 300.1333577 MHz
WDM EM
SSB 0
LB 0.00 Hz
GB 0
PC 8.00

1D NMR plot parameters
CX 20.00 cm
F1P 2.849 ppm
F1 855.06 Hz
F2P 0.454 ppm
F2 136.31 Hz
PPMCM 0.11974 ppm/cm
HZCM 35.93731 Hz/cm



21





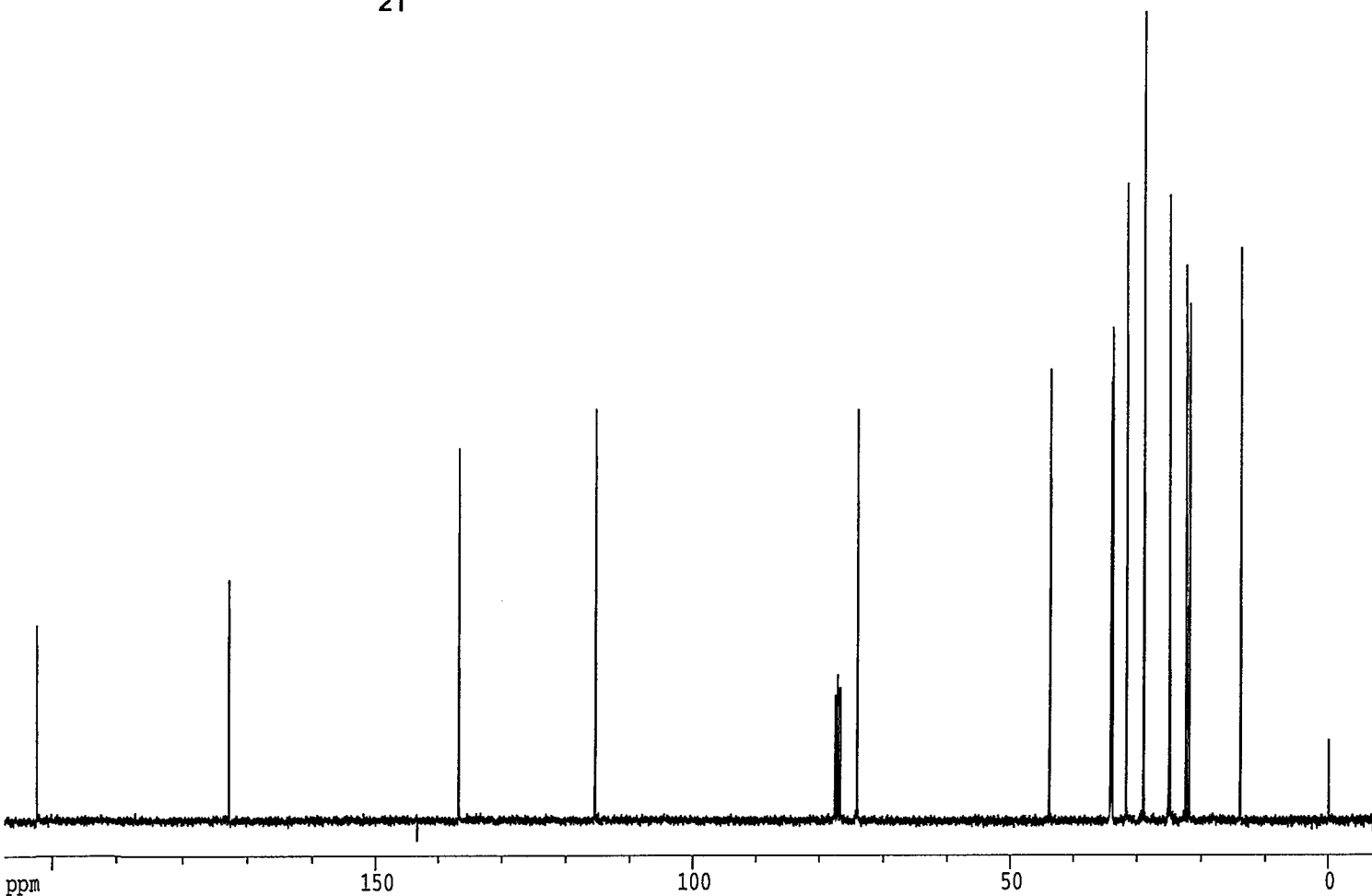
21

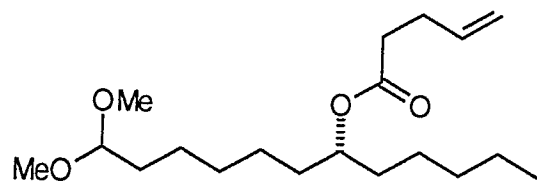
Current Data Parameters
 NAME jan03101
 EXPNO 11
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970103
 Time 10.35
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 2000
 DS 16
 SWH 33333.156 Hz
 FIDRES 0.508624 Hz
 AQ 0.9830900 sec
 RG 32768
 DW 15.000 usec
 DE 21.43 usec
 TE 302.0 K
 D11 0.03000000 sec
 CPDPRG waltz16
 P31 100.00 usec
 S2 27 dB
 HL1 0 dB
 D1 0.03000000 sec
 P1 5.68 usec
 DE 21.43 usec
 SFO1 75.4815977 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 32768
 SF 75.4685862 MHz
 WDW EM
 SSB 0
 LB 0.80 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 207.807 ppm
 F1 15682.93 Hz
 F2P -7.647 ppm
 F2 -577.14 Hz
 PPMCM 10.77275 ppm/cm
 HZCM 813.00385 Hz/cm





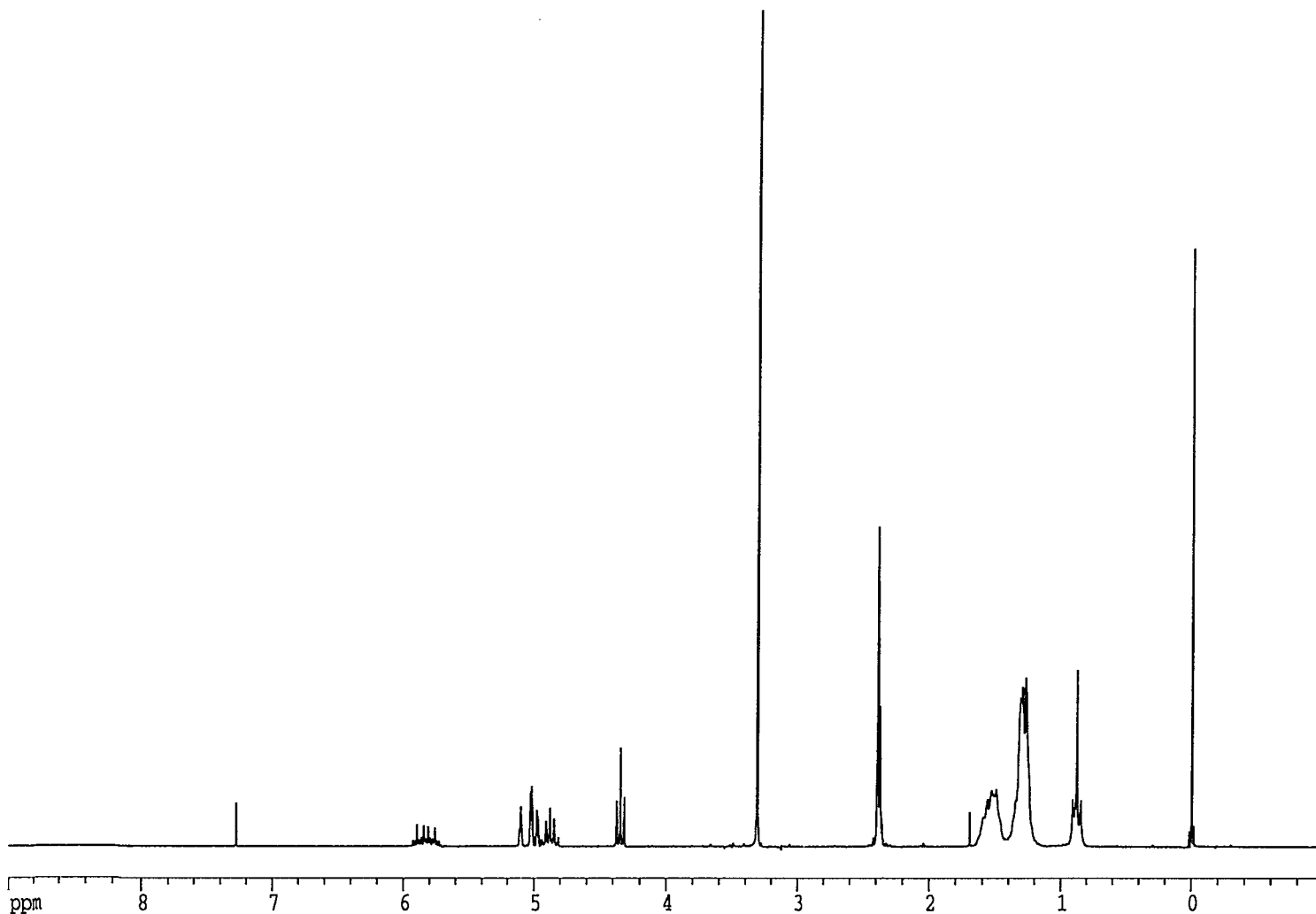
20

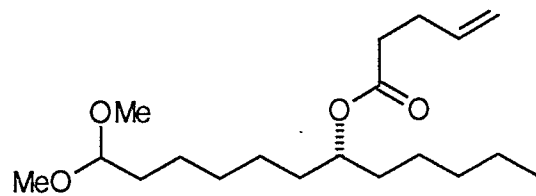
Current Data Parameters
 NAME DE180F
 EXPNO 128
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 961218
 Time 22.07
 INSTRUM ac200
 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 10
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HL1 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 200.1323377 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 1801.19 Hz
 F2P -1.000 ppm
 F2 -200.13 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 100.06617 Hz/cm





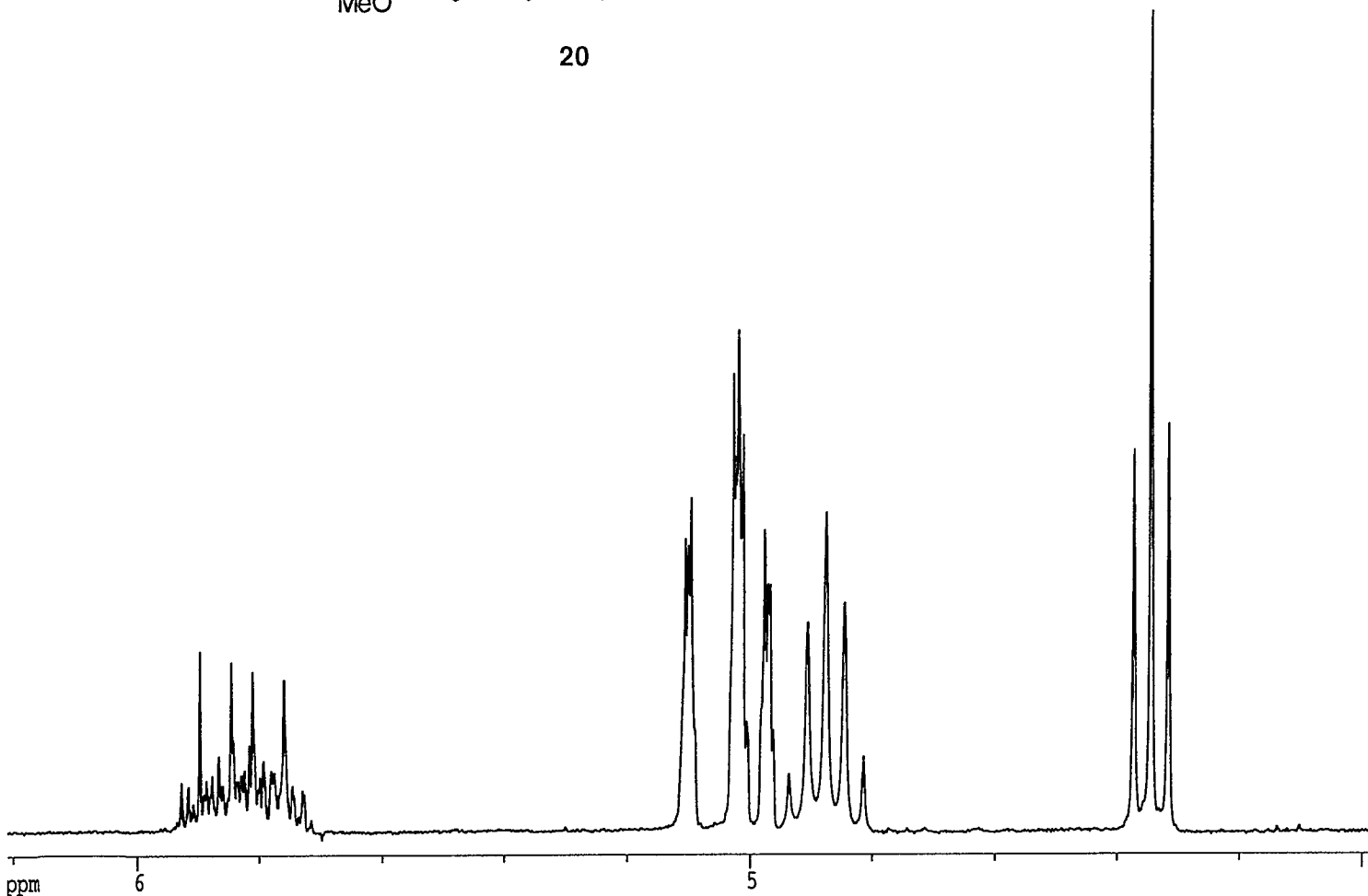
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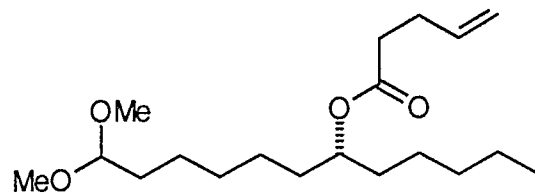
Current Data Parameters
 NAME DE180F
 EXPNO 128
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 961218
 Time 22.07
 INSTRUM ac200
 PROBHD
 PULPROG X51.AU
 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 10
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HL1 83 dB
 D1 1.0000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 200.1323377 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 6.216 ppm
 F1 1244.05 Hz
 F2P 3.971 ppm
 F2 794.79 Hz
 PPMCM 0.11224 ppm/cm
 HZCM 22.46314 Hz/cm





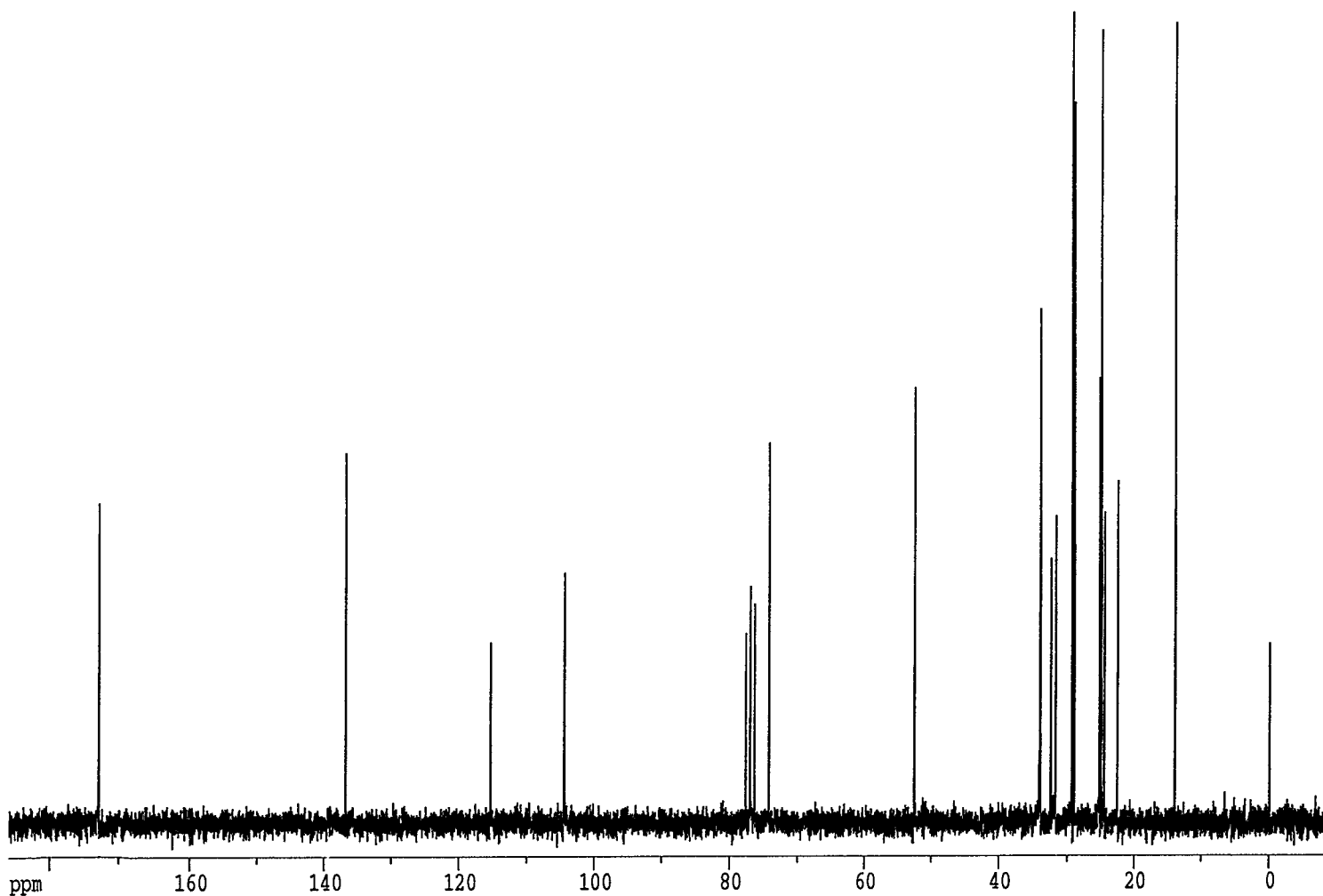
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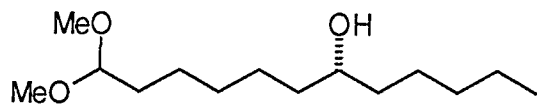
Current Data Parameters
 NAME DE181F
 EXPNO 128
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 961218
 Time 22.40
 INSTRUM ac200
 PROBHD
 PULPROG X53.AU
 TD 32768
 SOLVENT unknown
 NS 1568
 DS 0
 SWH 14285.714 Hz
 FIDRES 0.435965 Hz
 AQ 1.1469300 sec
 RG 640
 DW 35.000 usec
 DE 46.30 usec
 TE 300.0 K
 P1 15.50 usec
 HL1 20 dB
 D1 0.00100000 sec
 DE 46.30 usec
 SFO1 50.3287650 MHz
 NUCLEUS unknown

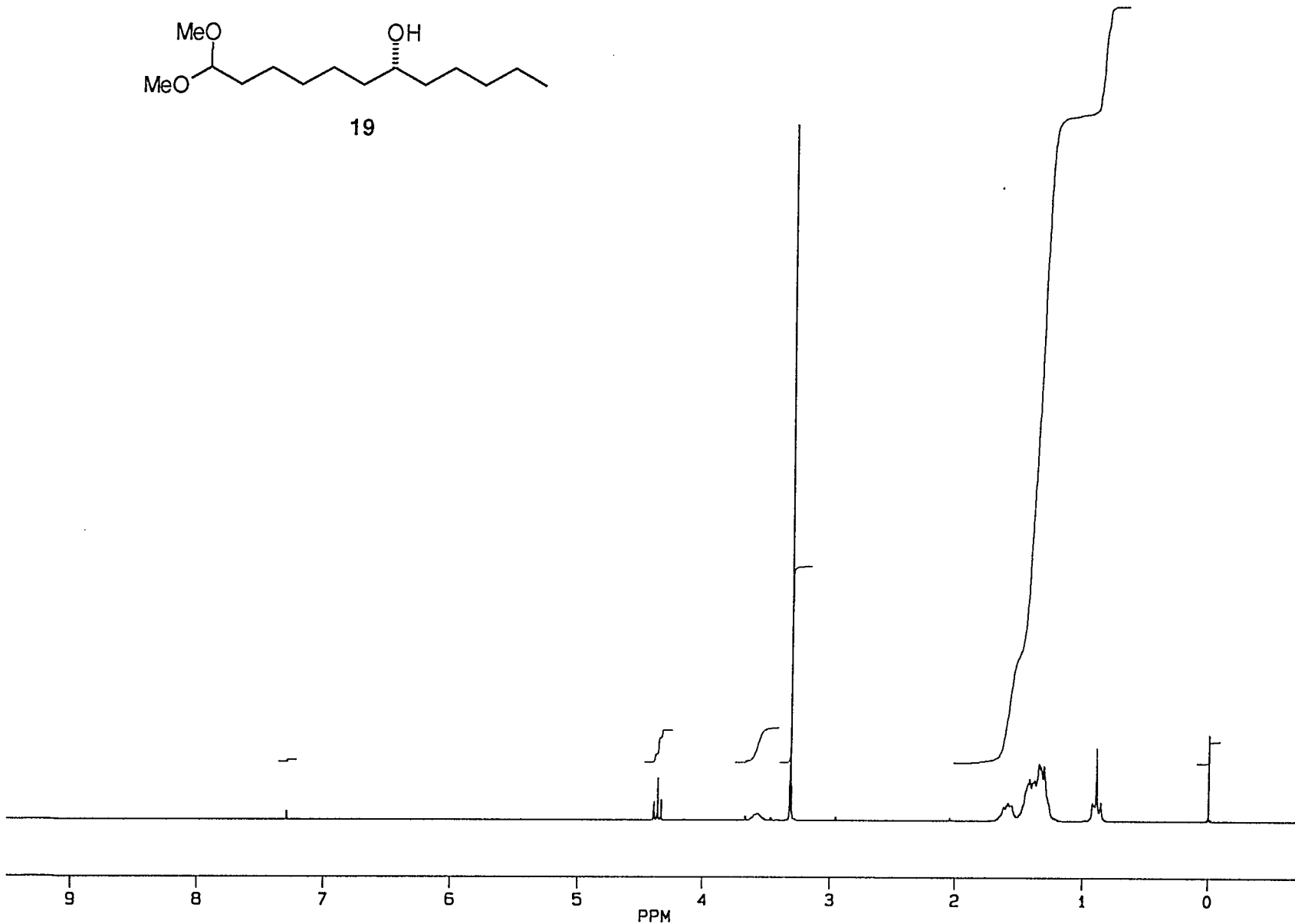
F2 - Processing parameters
 SI 16384
 SF 50.3233171 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 186.424 ppm
 F1 9381.47 Hz
 F2P -9.266 ppm
 F2 -466.32 Hz
 PPMCM 9.78452 ppm/cm
 HZCM 492.38953 Hz/cm





19



U110F.141
 AU PROG:
 X51.AU
 DATE 17-12-96
 TIME 6:08

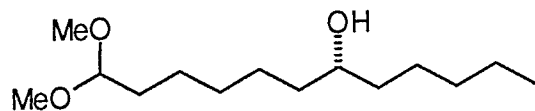
SA.NA LGM07601
 SA.NO DE16 141
 SOLVENT CDC13
 SF 200.132
 SY 80.0
 O1 3239.000
 SI 32768
 TD 32768
 SW 4032.258
 HZ/PT .246

PW 0.0
 RD 0.0
 AQ 4.063
 RG 4
 NS 32
 TE 300

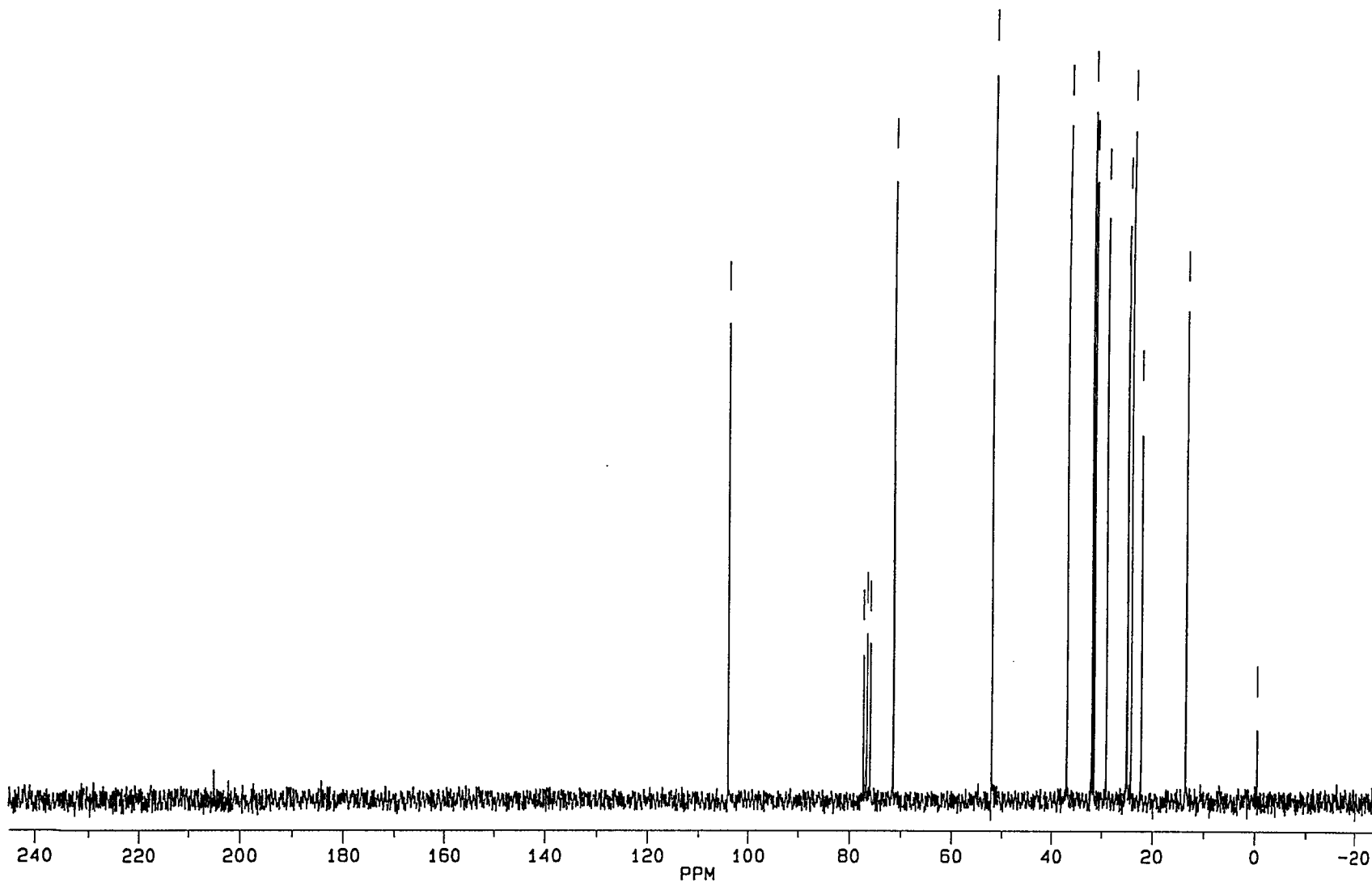
FW 5100
 O2 2228.997
 DP 63L P0

LB 0.0
 GB .180
 CX 22.65
 CY 12.00
 F1 1900.21H
 F2 -149.39H
 HZ/CM 90.490
 PPM/CM .452
 SR 2334.79

D7 .0002000
 D1 1.0000000
 P0 3.40
 RGA
 RD 0.0
 PW 0.0
 DE 155.00
 NS 32
 DS 0



19



3R

DE161F.141
 AU PROG:
 X53.AU
 DATE 17-12-96
 TIME 6: 26

SA.NA LGM07601
 SA.NO DE16 141
 SOLVENT CDC13
 SF 50.323
 SY 50.0
 O1 1765.000
 SI 32768
 TD 32768
 SW 14285.714
 HZ/PT .872

PW 0.0
 RD 0.0
 AQ 1.147
 RG 640
 NS 800
 TE 300

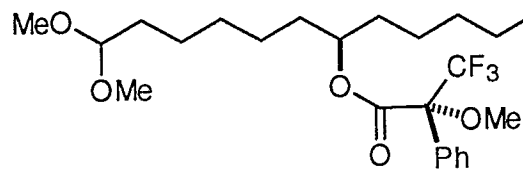
FW 17900
 O2 3239.000
 DP 20H CPD

LB .800
 GB 0.0
 CX 22.65
 CY 12.00
 F1 245.015P
 F2 -24.985P
 HZ/CM 599.881
 PPM/CM 11.921
 SR -3665.39

D7 .0090900
 D1 .0010000
 P9 100.00
 S1 20H
 D5 .0010000
 S2 20H
 P0 3.70
 RGA
 RD 0.0
 PW 0.0
 DE 46.30
 NS RAN

ppm

-71.882



19F NMR: Mosher ester of 19

Current Data Parameters
 NAME dez19102
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date 500000
 Time 4.00
 PULPROG zgpg30
 SOLVENT CDCl3
 AQ 0.2621640 sec
 DW 4.0 usec
 RG 256
 NUCLEUS 19F
 SFO1 282.3891972 MHz
 SFO2 300.1341790 MHz
 TE 302.0 K
 HL1 0 dB
 D1 1.0000000 sec
 P31 100.0 usec
 S4 27 dB
 D11 0.0300000 sec
 S2 27 dB
 P1 8.0 usec
 DE 5.7 usec
 SFO1 282.3891972 MHz
 SMH 125000.01 Hz
 TD 65536
 NS 1000
 DS 16

F2 - Processing parameters
 SI 32768
 SF 282.4076971 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 5.00

1D NMR plot parameters
 CX 20.00 cm
 F1P -70.977 ppm
 F1 -20044.53 Hz
 F2P -72.989 ppm
 F2 -20612.66 Hz
 PPMCM 0.10059 ppm/cm
 HZCM 28.40700 Hz/cm

ppm

-71.5

-72.0

-72.5