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SUPPORTING INFORMATION

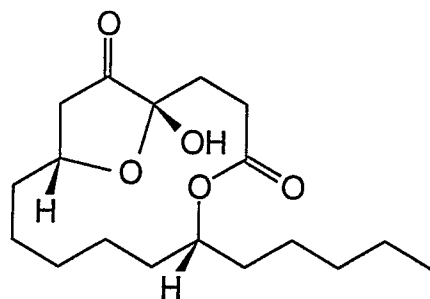
Total Syntheses of (+)-Ricinelaiddic Acid Lactone and of (-)-Gloeosporone Based on Transition Metal Catalyzed C-C-Bond Formations

Alois Fürstner* and Klaus Langemann

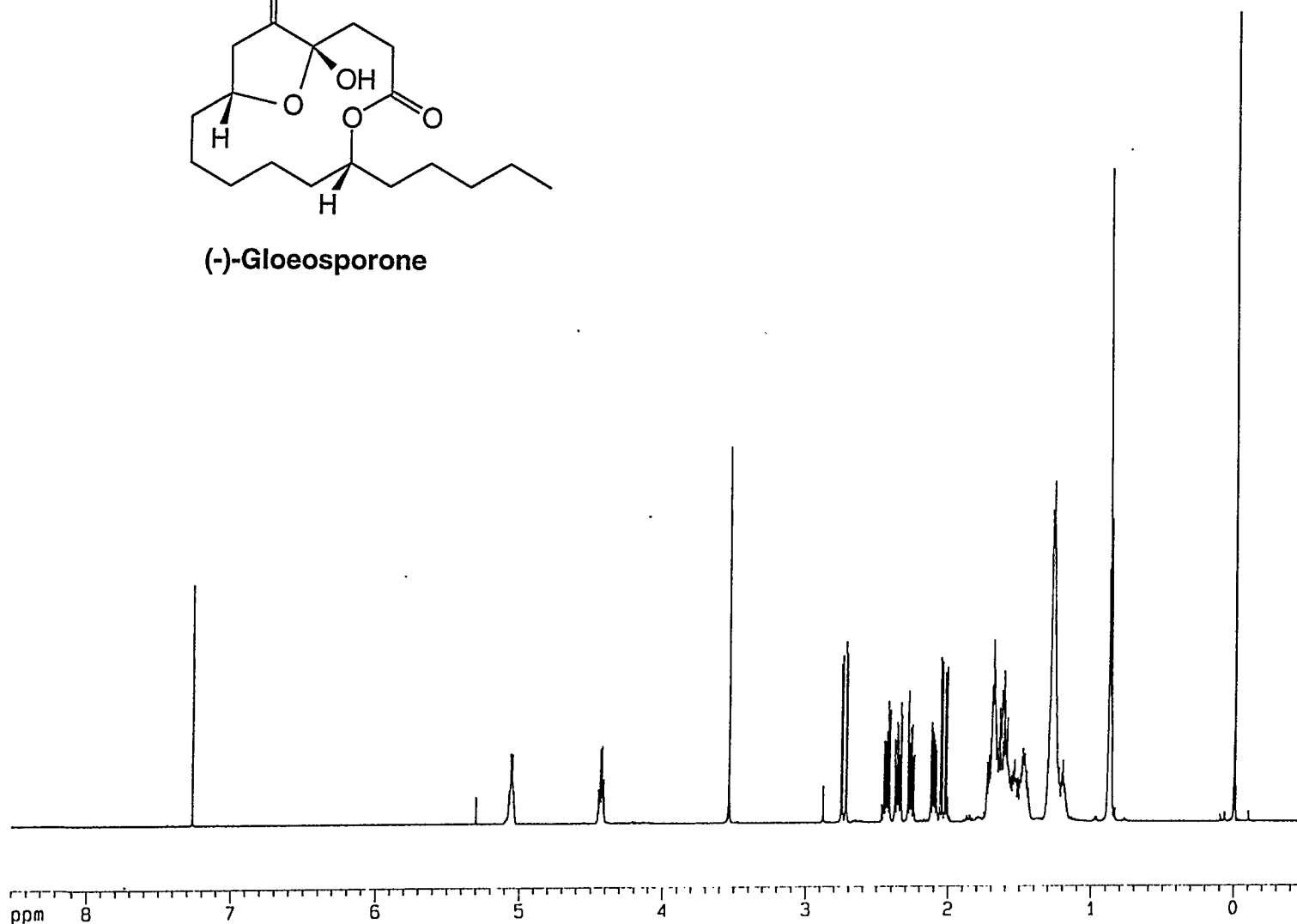
Max-Planck-Institut für Kohlenforschung

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



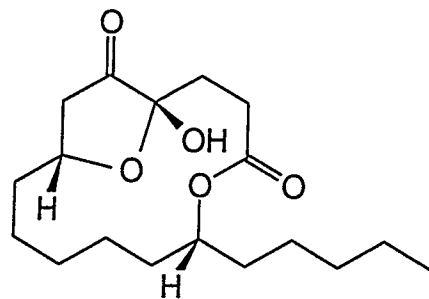
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 DU v
 USER bara

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 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 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 1.00000000 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
 WDW no
 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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 HZCM 270.09900 Hz/cm

LGM-LB-143-01 1
 1H



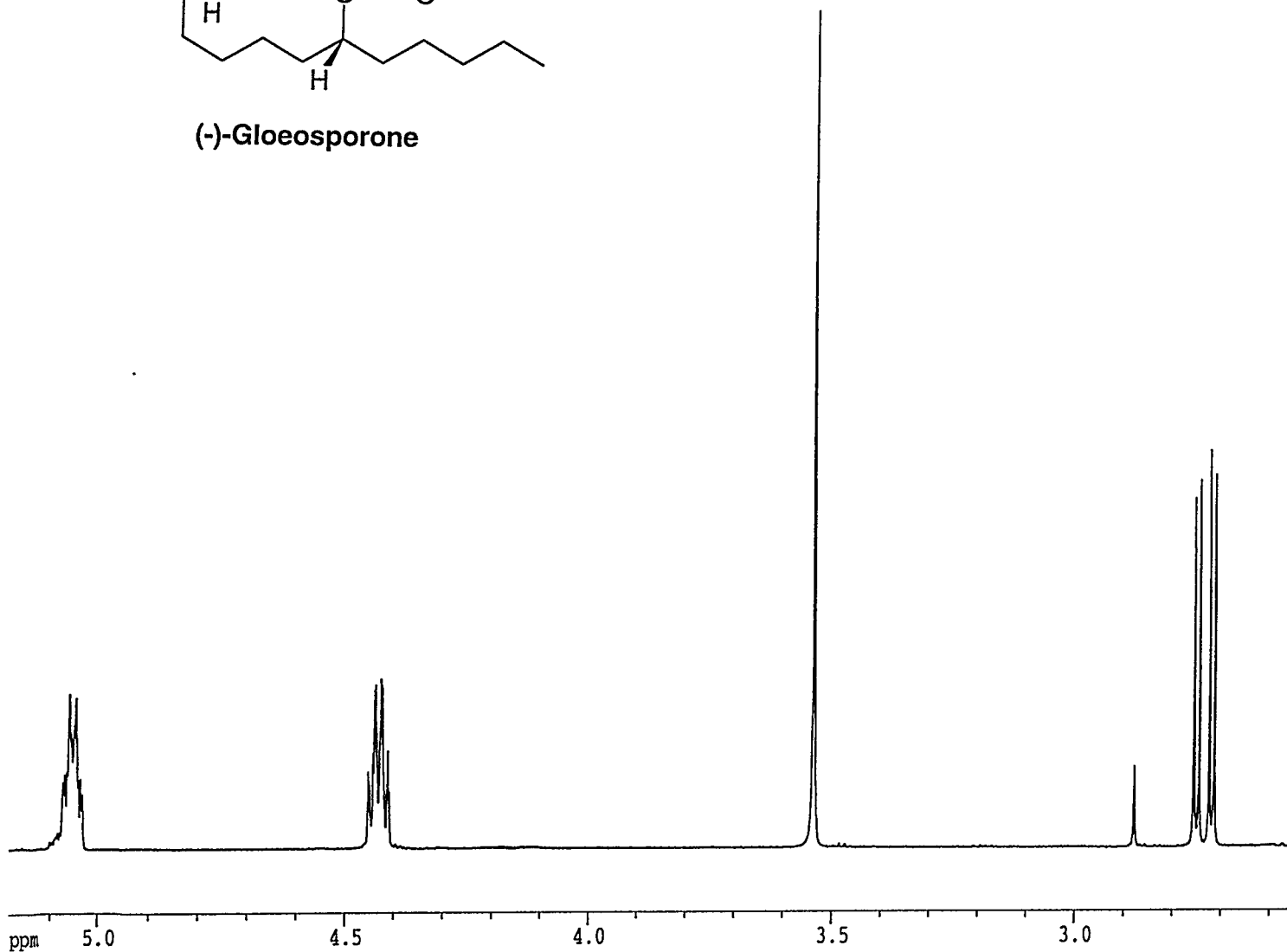
(-)-Gloeosporone

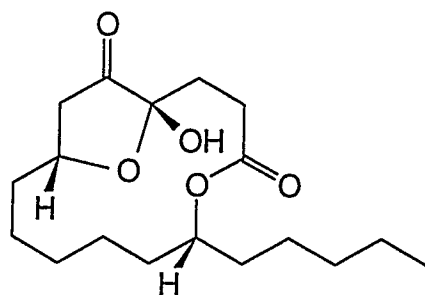
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 SOLVENT CDCl3
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 DS 2
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 AQ 2.7263477 sec
 RG 32
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 NUCLEUS 1H

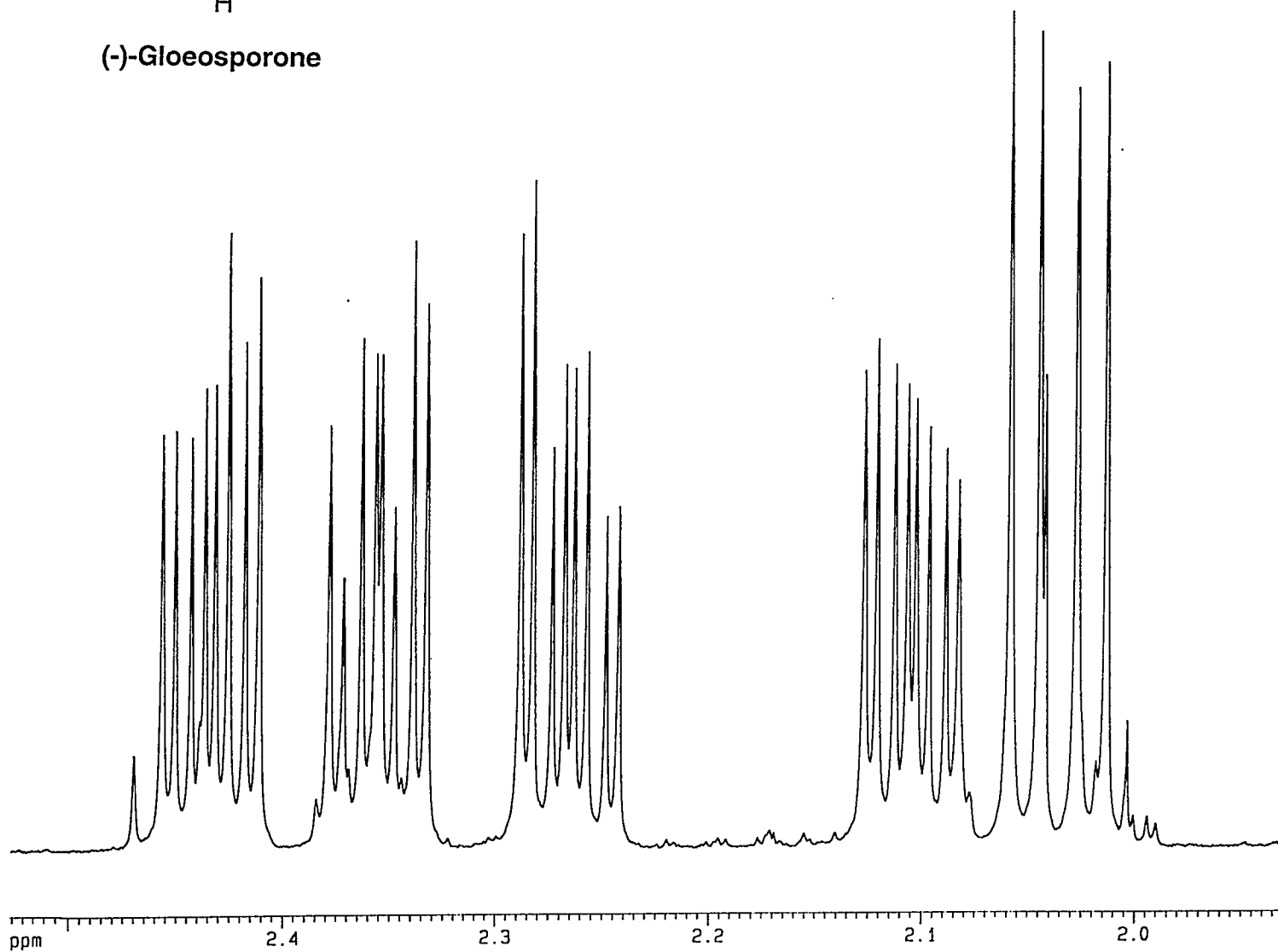
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 F1 3118.50 Hz
 F2P 2.542 ppm
 F2 1525.96 Hz
 PPMCM 0.13266 ppm/cm
 HZCM 79.62740 Hz/cm





(-)-Gloeosporone



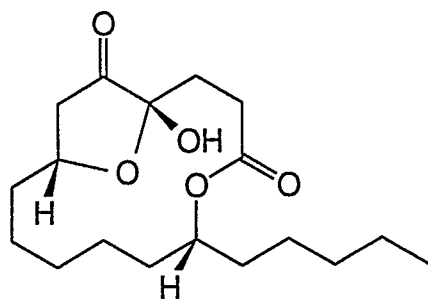
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 NS 32
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 32
 DW 41.600 usec
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 NUCLEUS 1H

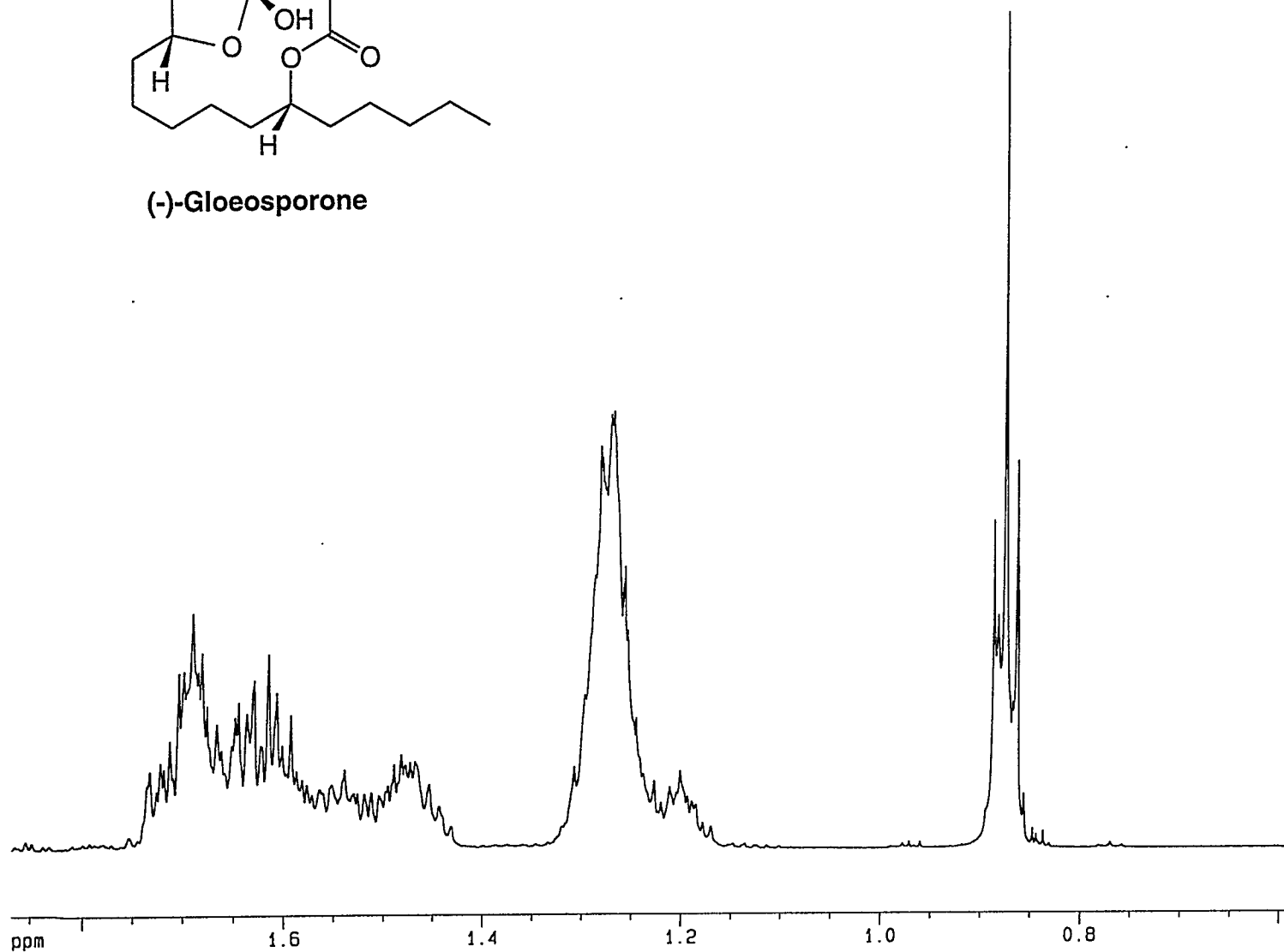
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 F1P 2.526 ppm
 F1 1516.18 Hz
 F2P 1.927 ppm
 F2 1156.82 Hz
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 HZCM 17.96821 Hz/cm

LGM-LB-143-01 1
 1H



(-)-Gloeosporone



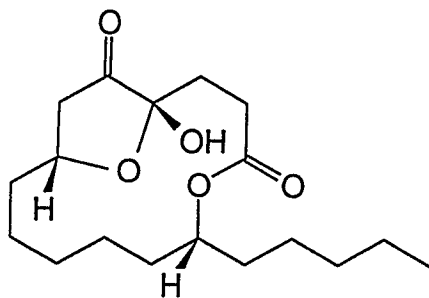
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 SOLVENT CDCl3
 NS 32
 DS 2
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 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 32
 DW 41.600 usec
 DE 4.50 usec
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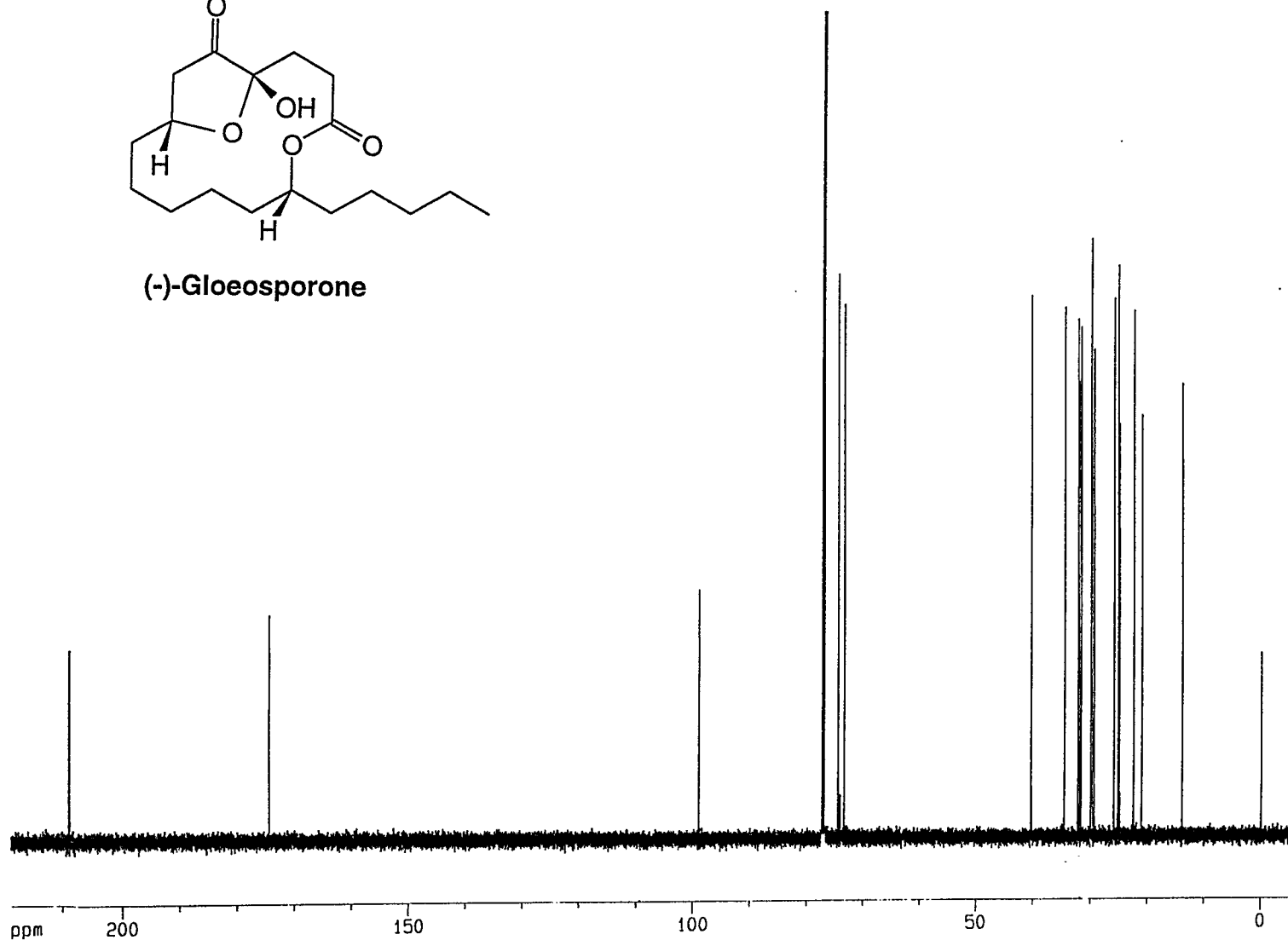
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1D NMR plot parameters
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 F1P 1.868 ppm
 F1 1121.33 Hz
 F2P 0.583 ppm
 F2 349.88 Hz
 PPHCM 0.05426 ppm/cm
 HZCM 38.57263 Hz/cm

LGM-LB-143-01 1
 1H



(-)-Gloeosporone



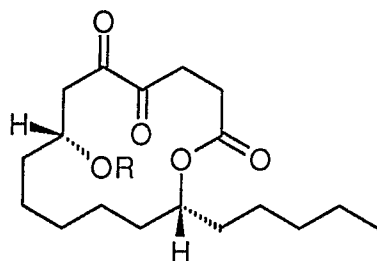
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 USER k1

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 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
 WDM no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.50

1D NMR plot parameters
 CX 20.00 cm
 F1P 219.151 ppm
 F1 33075.50 Hz
 F2P -5.317 ppm
 F2 -802.52 Hz
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 HZCM 1693.90100 Hz/cm

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



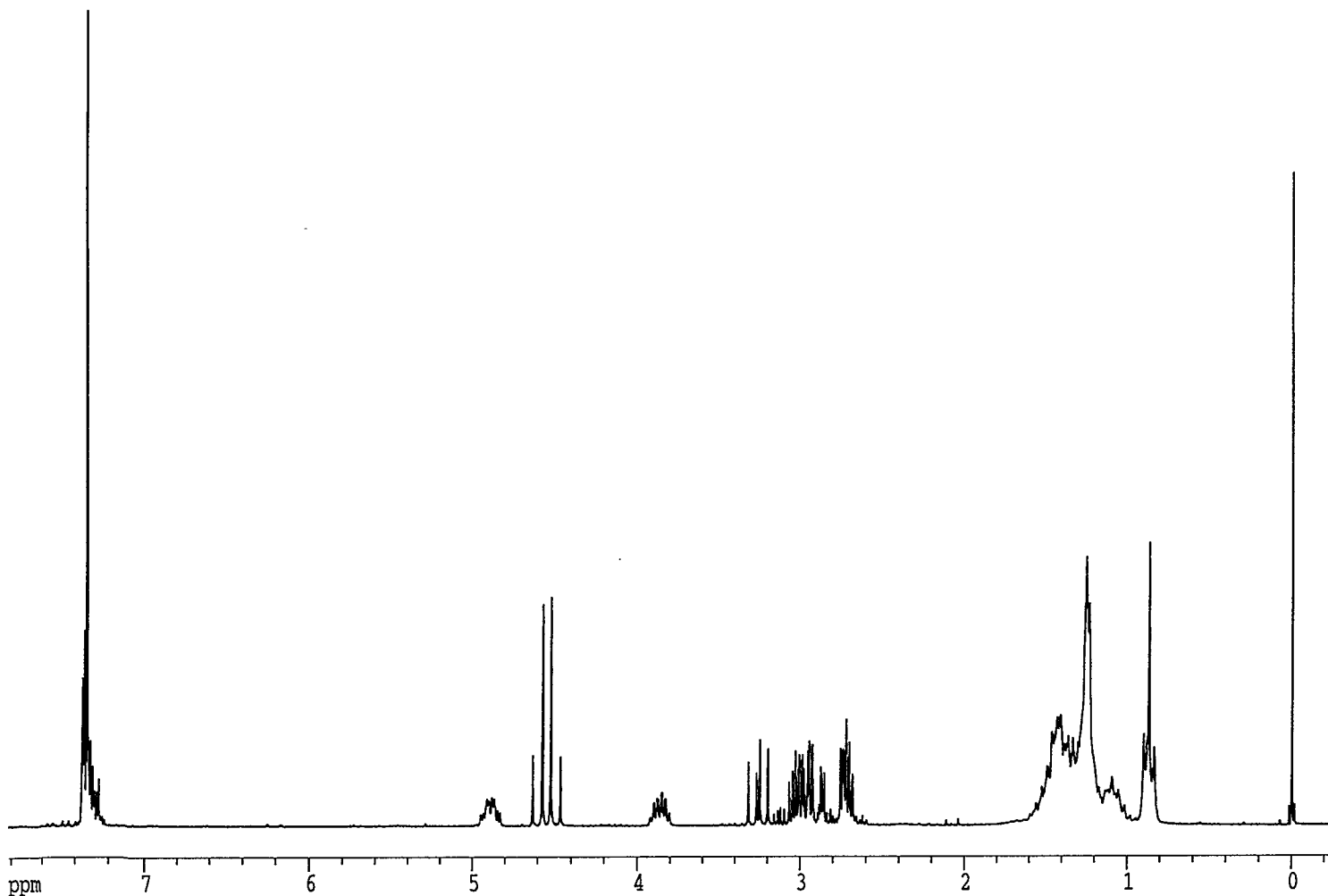
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 USER kl

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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
 HLL 83 dB
 D1 1.00000000 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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 HZCM 81.21971 Hz/cm

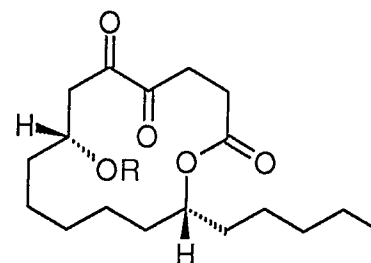


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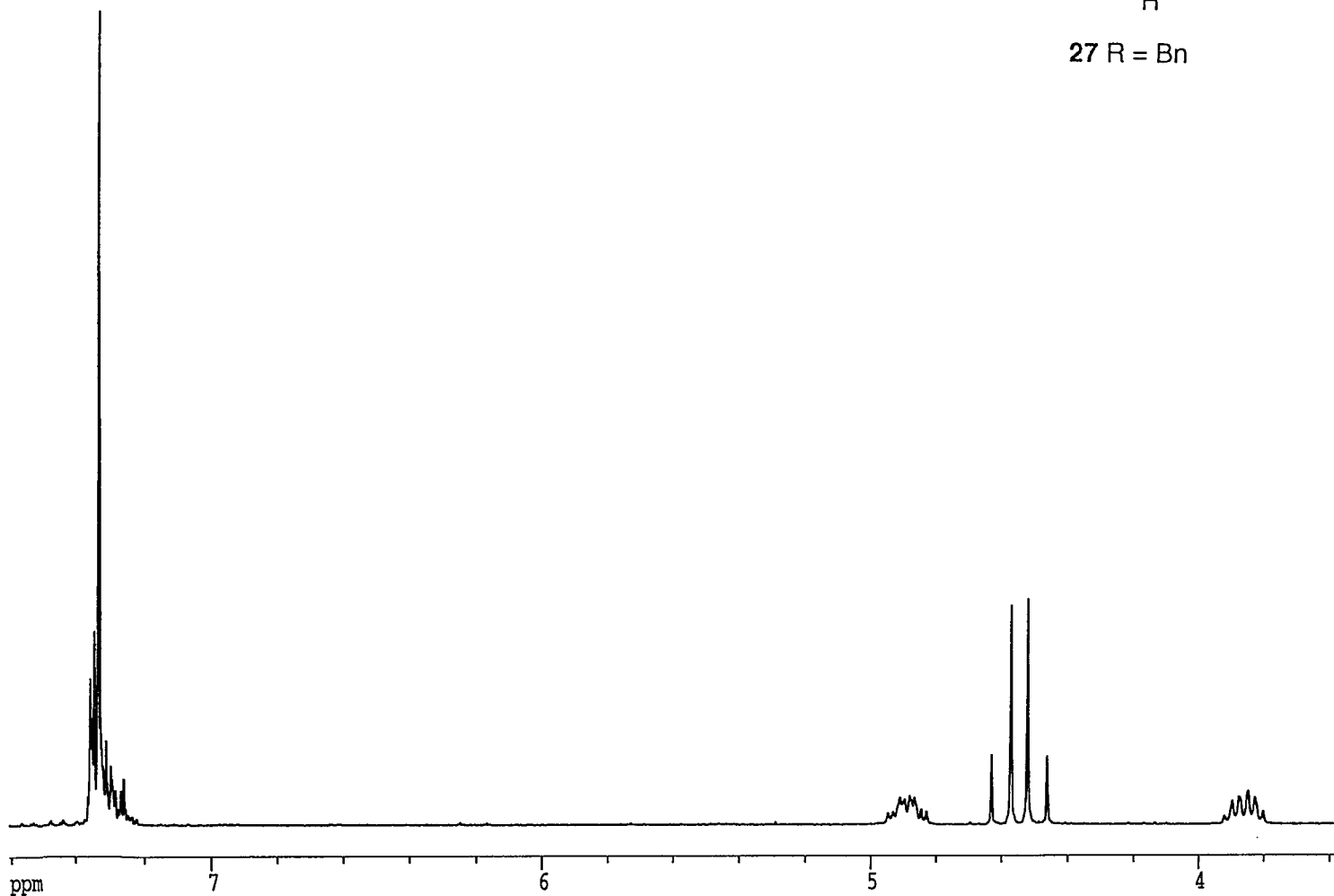
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 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 16
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
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 DE 155.00 usec
 SF01 200.1332390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

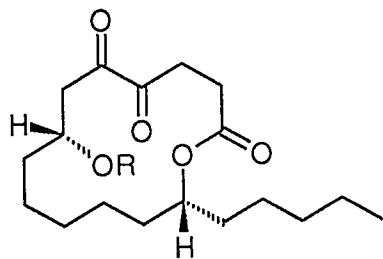
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 PC 4.00

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



27 R = Bn





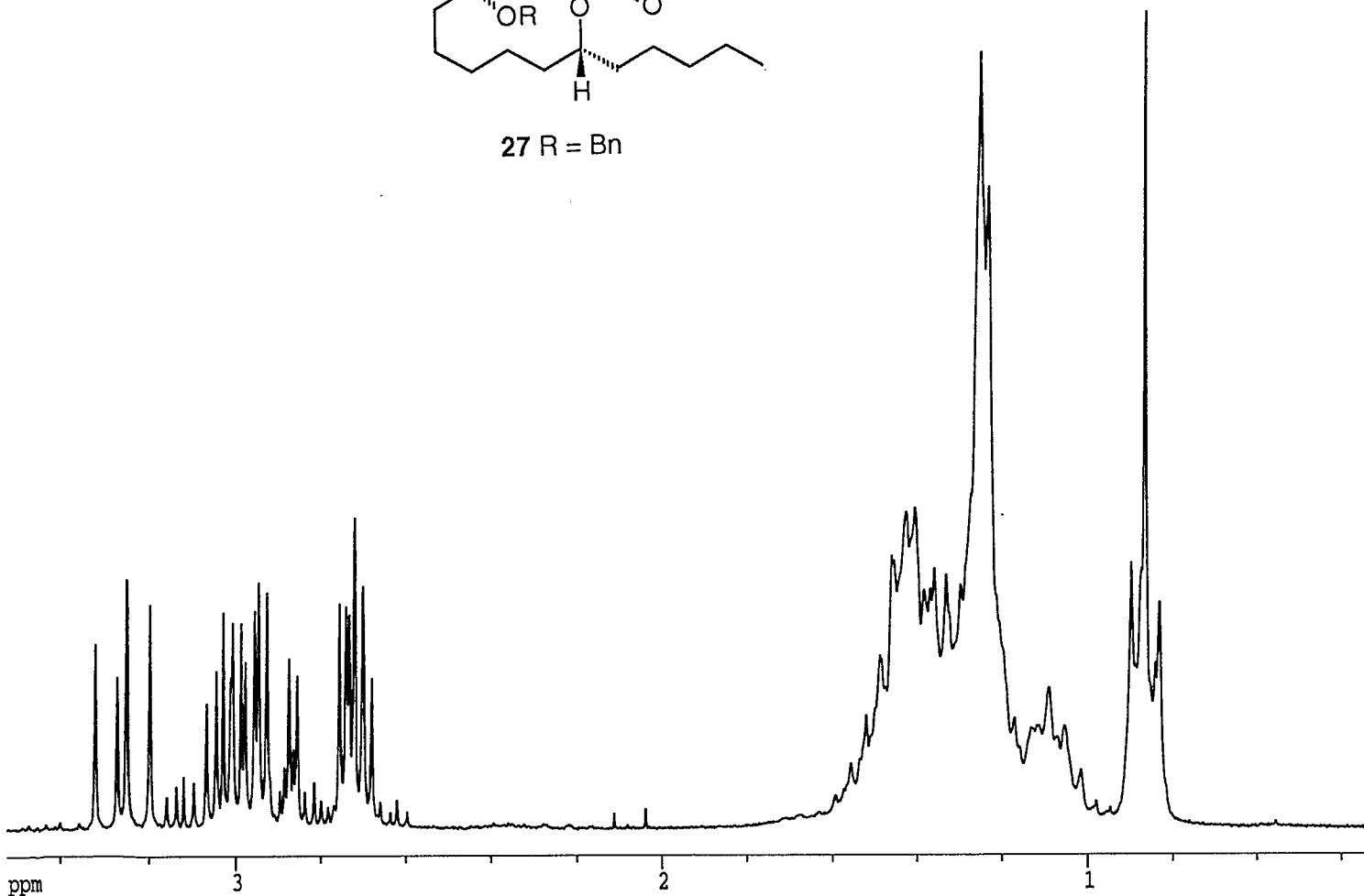
27 R = Bn

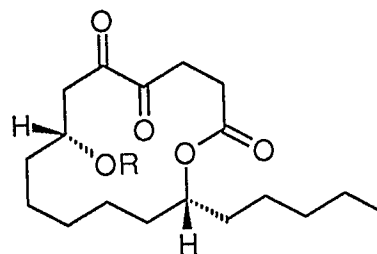
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 EXPNO 134
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date_ 970418
 Time 5.39
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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 1.00000000 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
 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





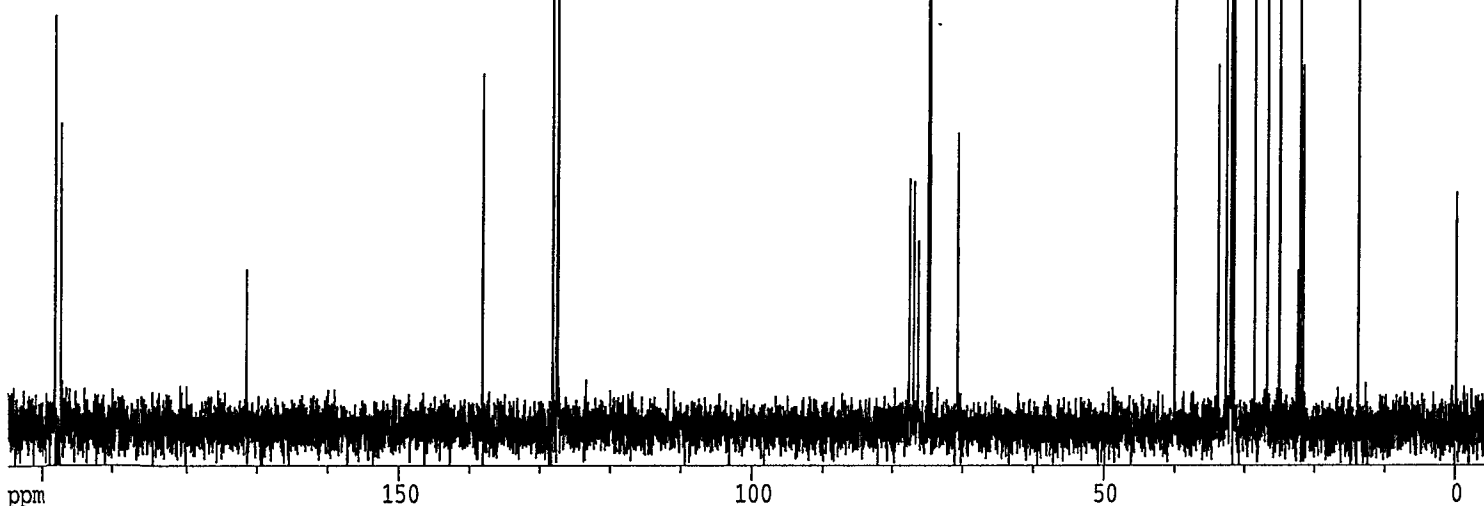
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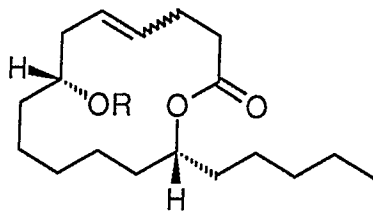
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 EXPNO 134
 PROCNO 1
 DU mpi
 USER kl

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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
 P1 15.50 usec
 HLL 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 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





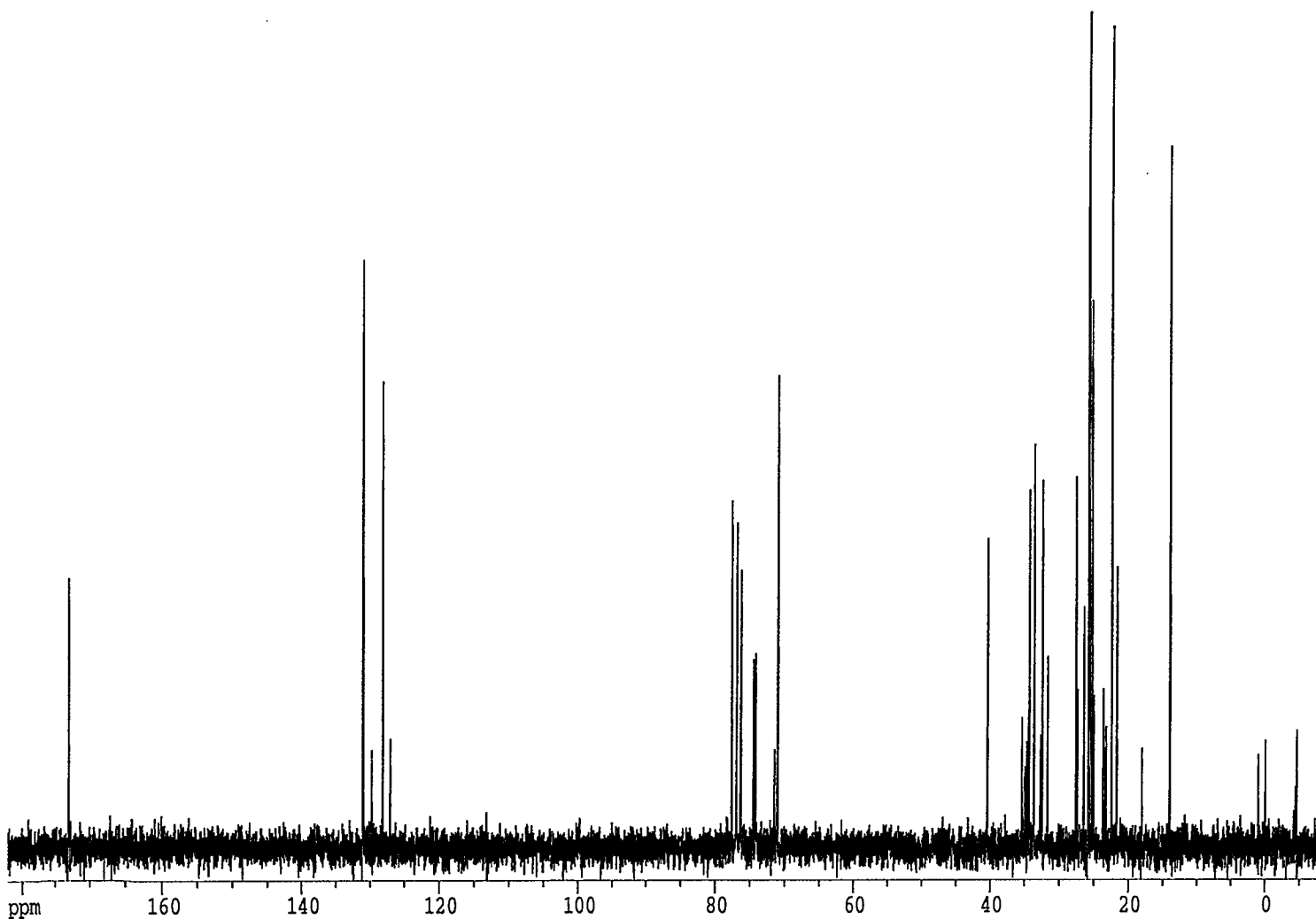
26 R = SiMe₂tBu (*E* : *Z* = 2.7 : 1)

Current Data Parameters
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 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
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 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
 SF01 50.3287650 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 SF 50.3233163 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
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 F1 9180.62 Hz
 F2P -8.520 ppm
 F2 -428.77 Hz
 PPMCM 9.54765 ppm/cm
 HZCM 480.46945 Hz/cm

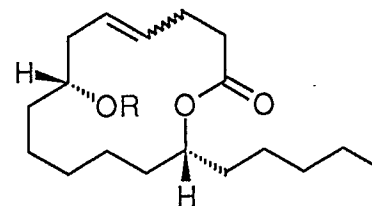


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 DU mpi
 USER kl

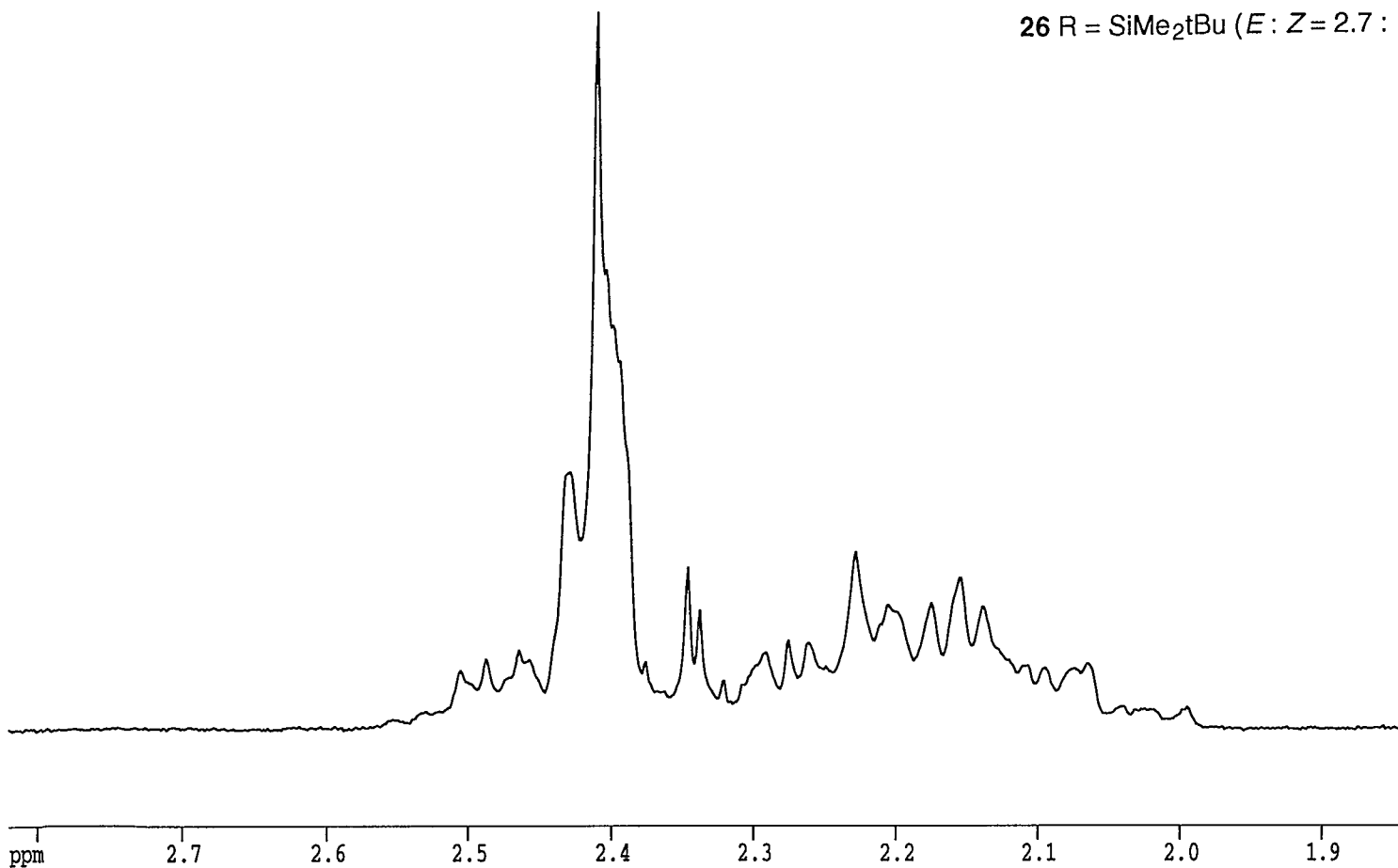
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 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 10
 DW 124.000 usec
 DE 155.00 usec
 TE 300.0 K
 P1 10.10 usec
 HL1 83 dB
 D1 1.00000000 sec
 DE 155.00 usec
 SFO1 200.1332390 MHz
 NUCLEUS unknown

F2 - Processing parameters
 SI 16384
 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
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26 R = SiMe₂tBu (E : Z = 2.7 : 1)

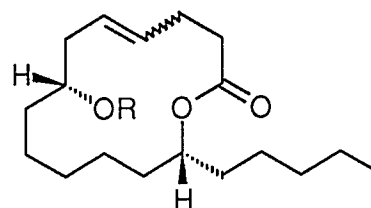


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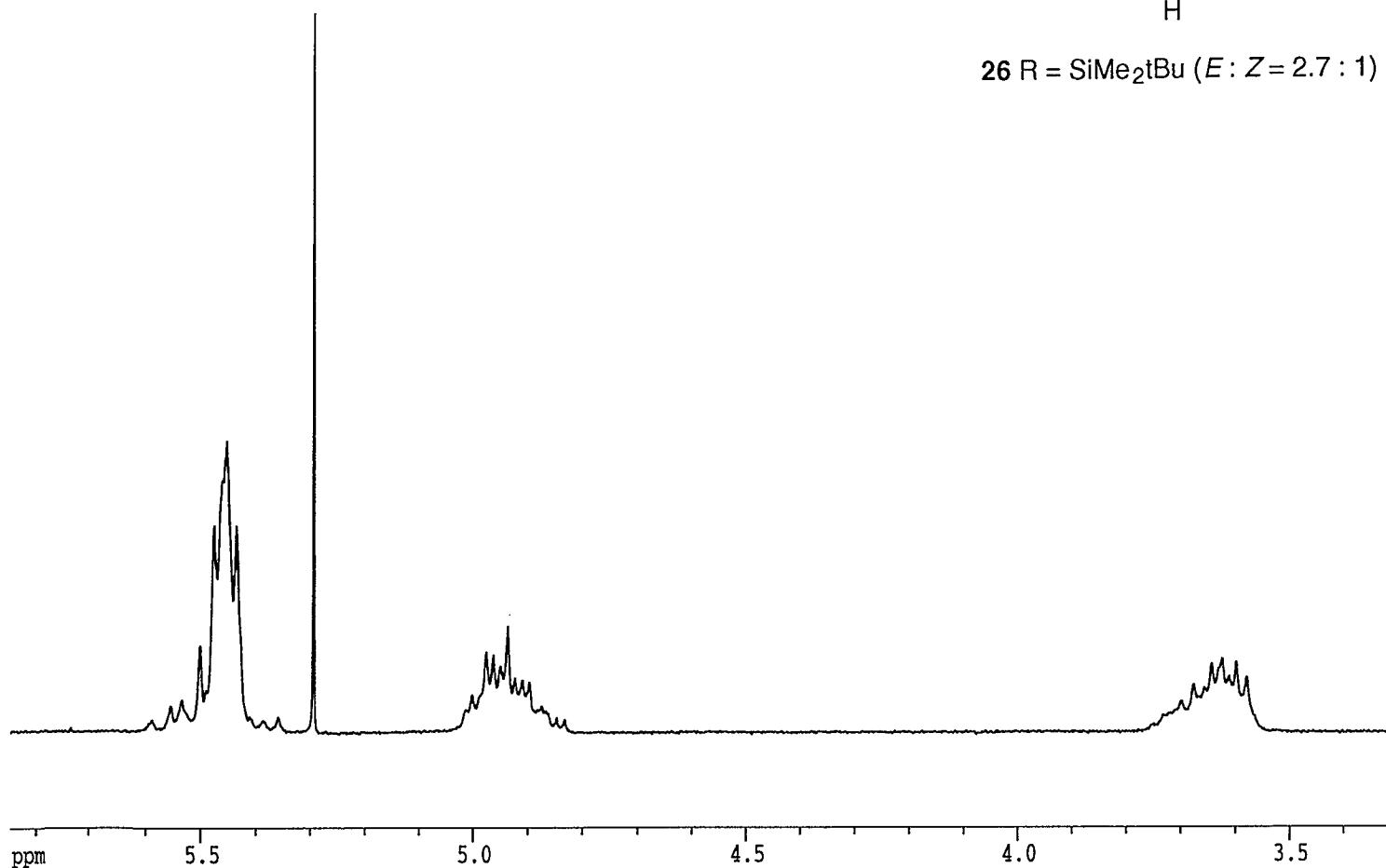
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 TD 32768
 SOLVENT unknown
 NS 32
 DS 0
 SWH 4032.258 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 10
 DW 124.000 usec
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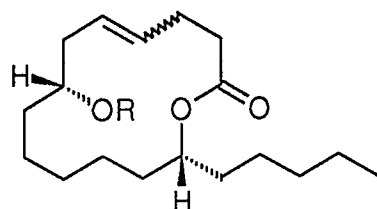
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 F1 1170.65 Hz
 F2P 3.306 ppm
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 HZCM 25.45363 Hz/cm



26 R = SiMe₂tBu (E : Z = 2.7 : 1)





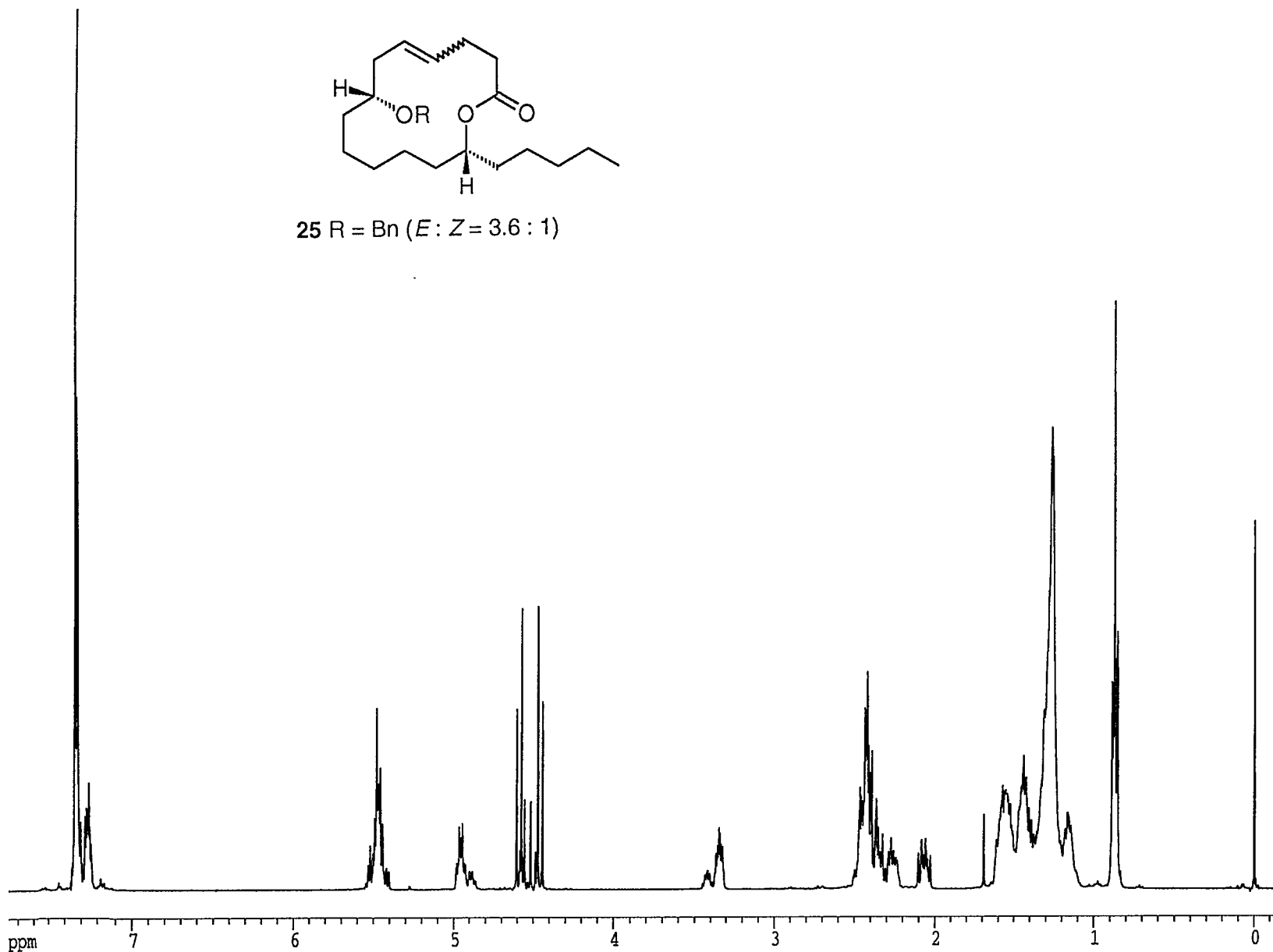
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 USER kl

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 PROBHD 5 mm Multinu
 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 2.00000000 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
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 F1 3111.06 Hz
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 F2 -75.04 Hz
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 HZCM 144.82246 Hz/cm

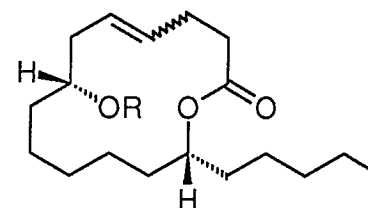


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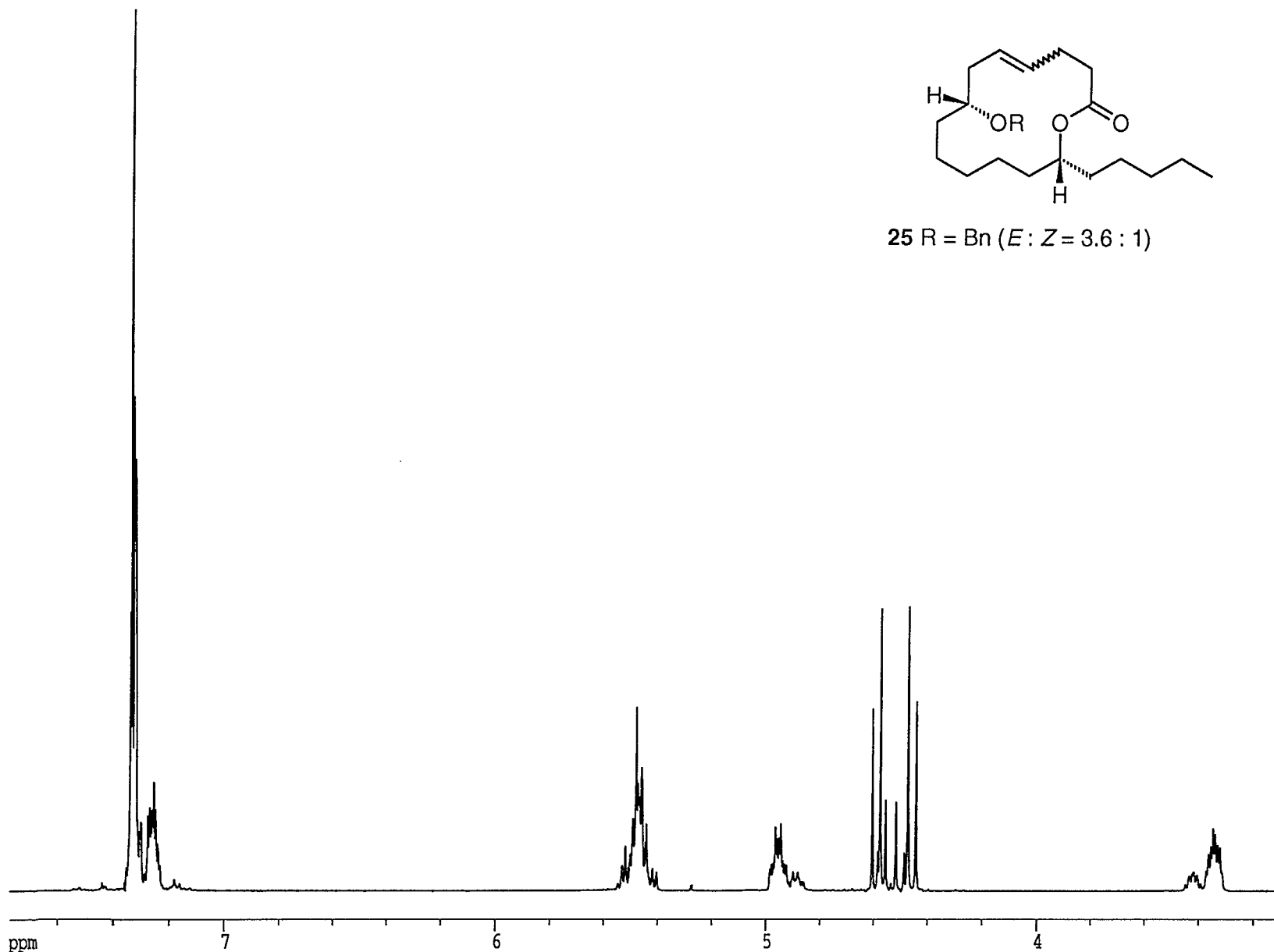
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 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 8064.474 Hz
 FIDRES 0.246108 Hz
 AQ 2.0316660 sec
 RG 32
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 HL1 1 dB
 D1 2.00000000 sec
 P1 5.60 usec
 DE 88.57 usec
 SFO1 400.1368403 MHz
 NUCLEUS 1H

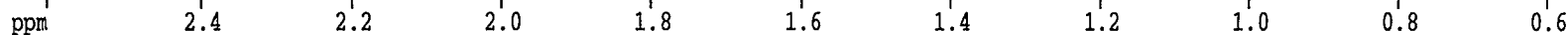
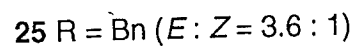
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1D NMR plot parameters
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 F2P 3.081 ppm
 F2 1232.83 Hz
 PPMCM 0.21414 ppm/cm
 HZCM 85.68504 Hz/cm

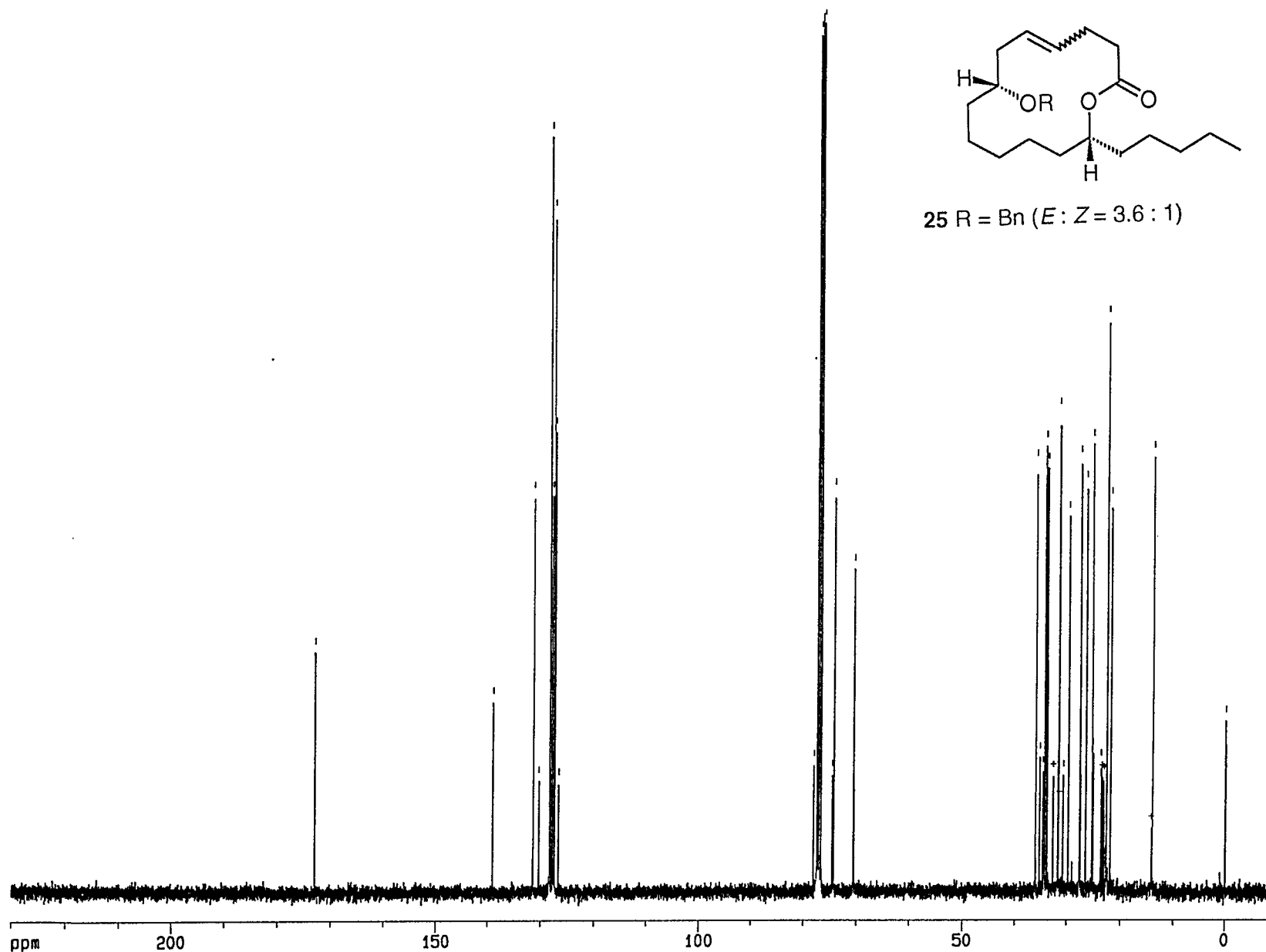


25 R = Bn (*E* : *Z* = 3.6 : 1)





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

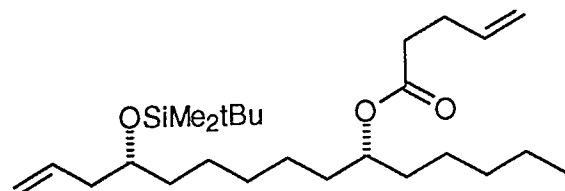


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 DU mpi
 USER wisno

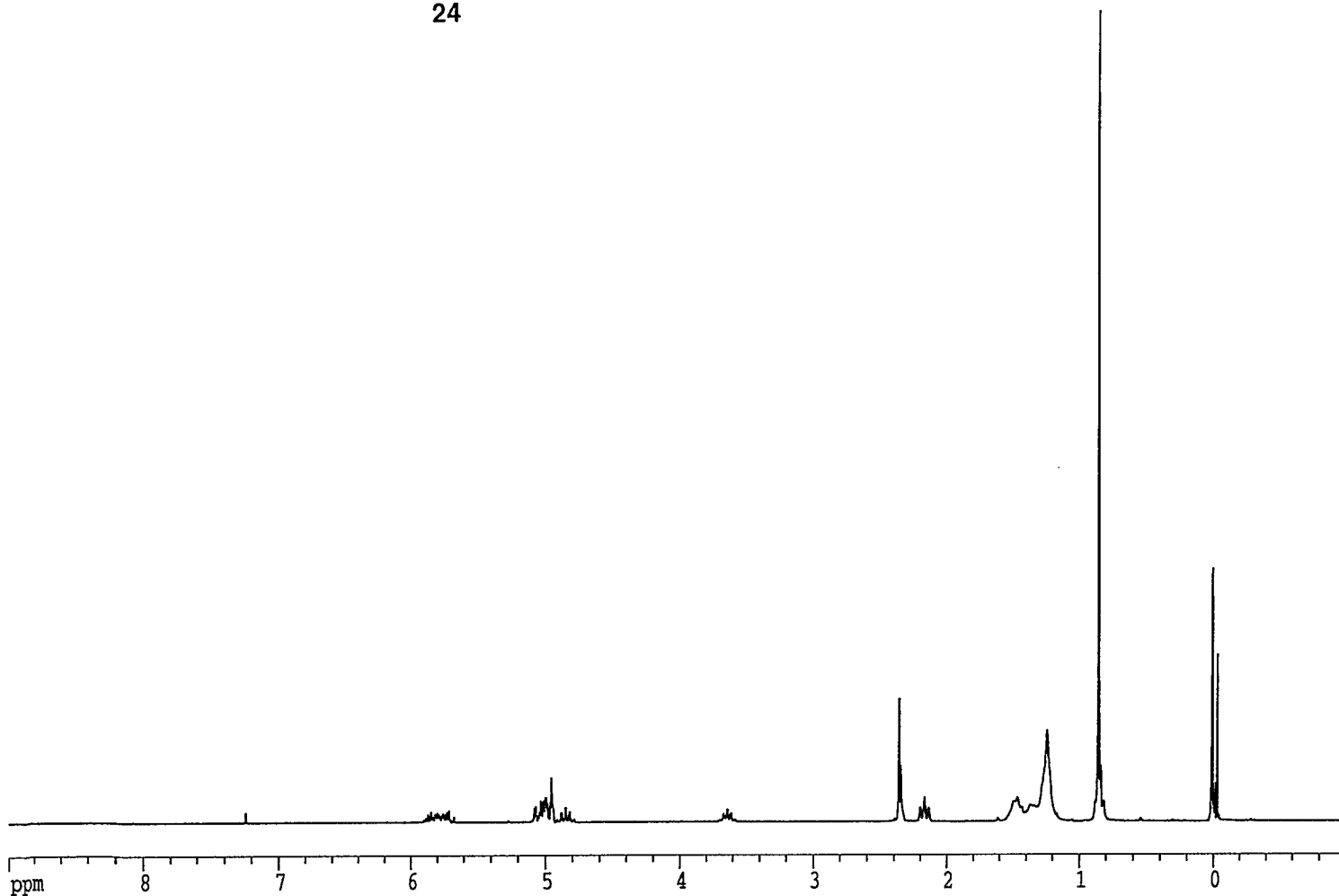
F2 - Acquisition Parameters
 Date_ 970129
 Time 3.33
 INSTRUM amx400
 PROBHD 5 mm Multinu
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 43518 20000
 DS 300
 SWH 31249.838 Hz
 FIDRES 0.476835 Hz
 AQ 1.0486259 sec
 RG 16384
 DW 16.000 usec
 DE 22.86 usec
 TE 300.0 K
 O1 6399.74 Hz
 O2 6840.29 Hz
 D11 0.03000000 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

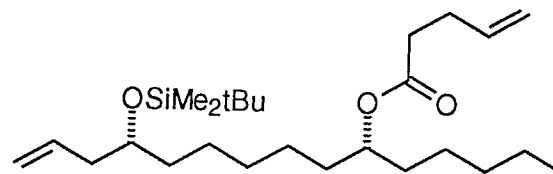


Current Data Parameters
 NAME AP240F
 EXPNO 137
 PROCNO 1
 DU mpi
 USER kl

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 1.00000000 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



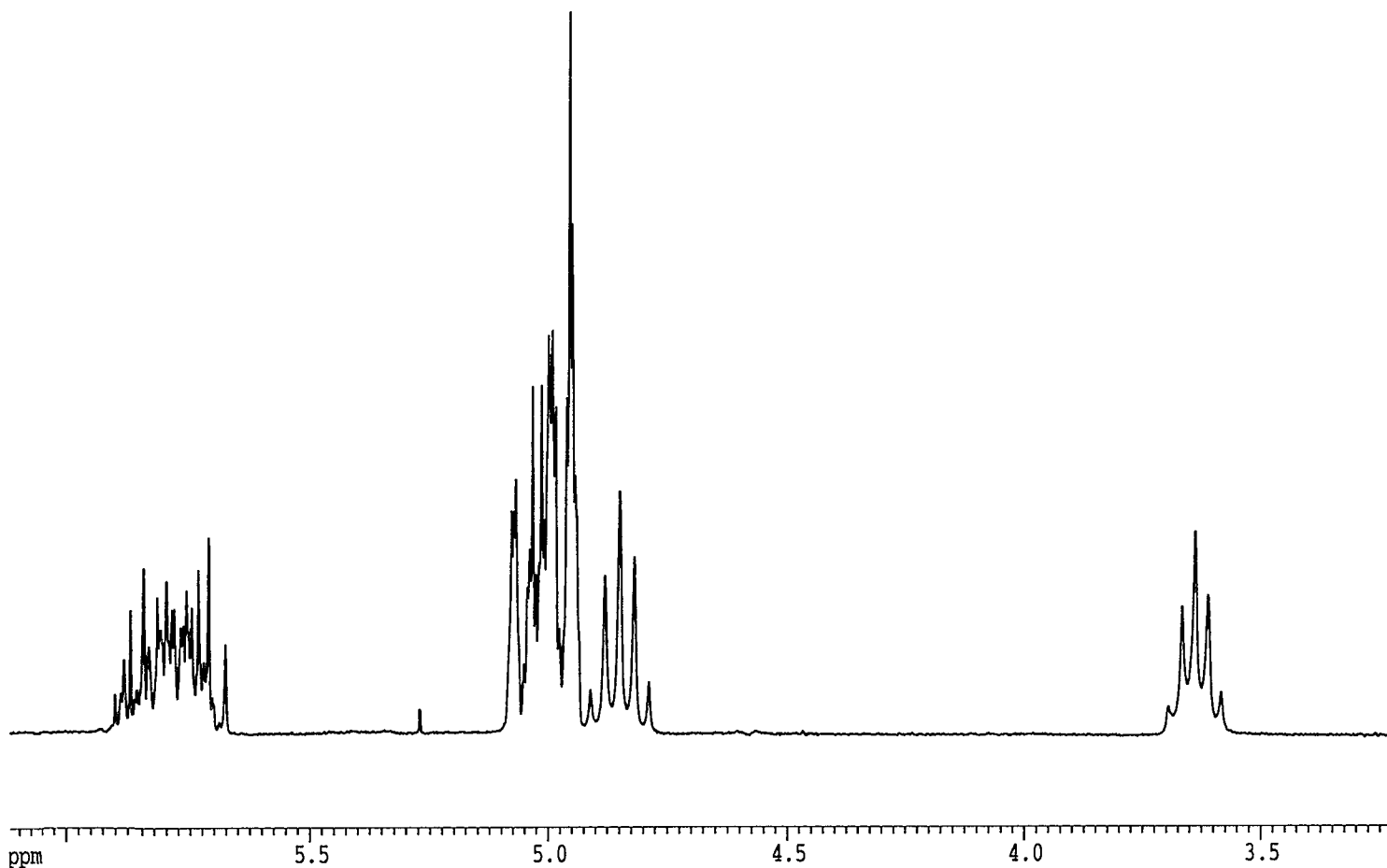
24

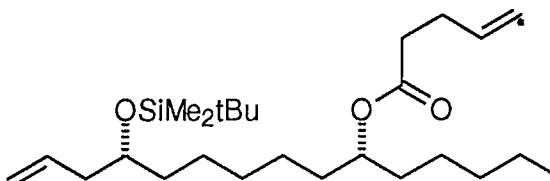
Current Data Parameters
 NAME AP240F
 EXPNO 137
 PROCNO 1
 DU mpi
 USER kl

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





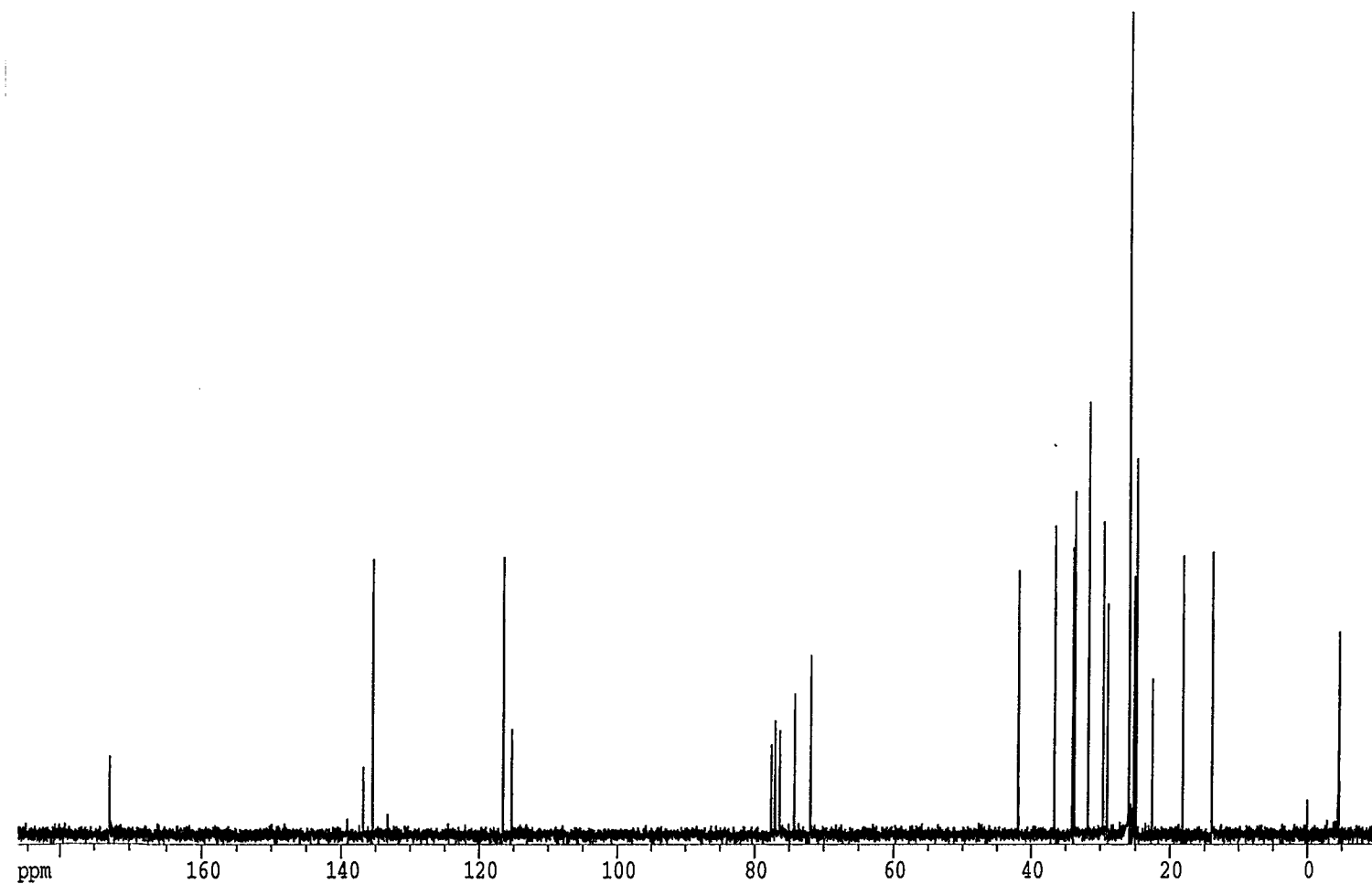
24

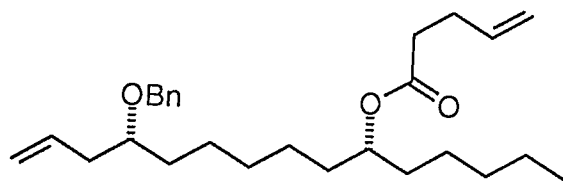
Current Data Parameters
 NAME AP241P
 EXPNO 137
 PROCNO 1
 DU mpi
 USER kl

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 0.00100000 sec
 DE 46.30 usec
 SFO1 50.3287650 MHz
 NUCLEUS unknown

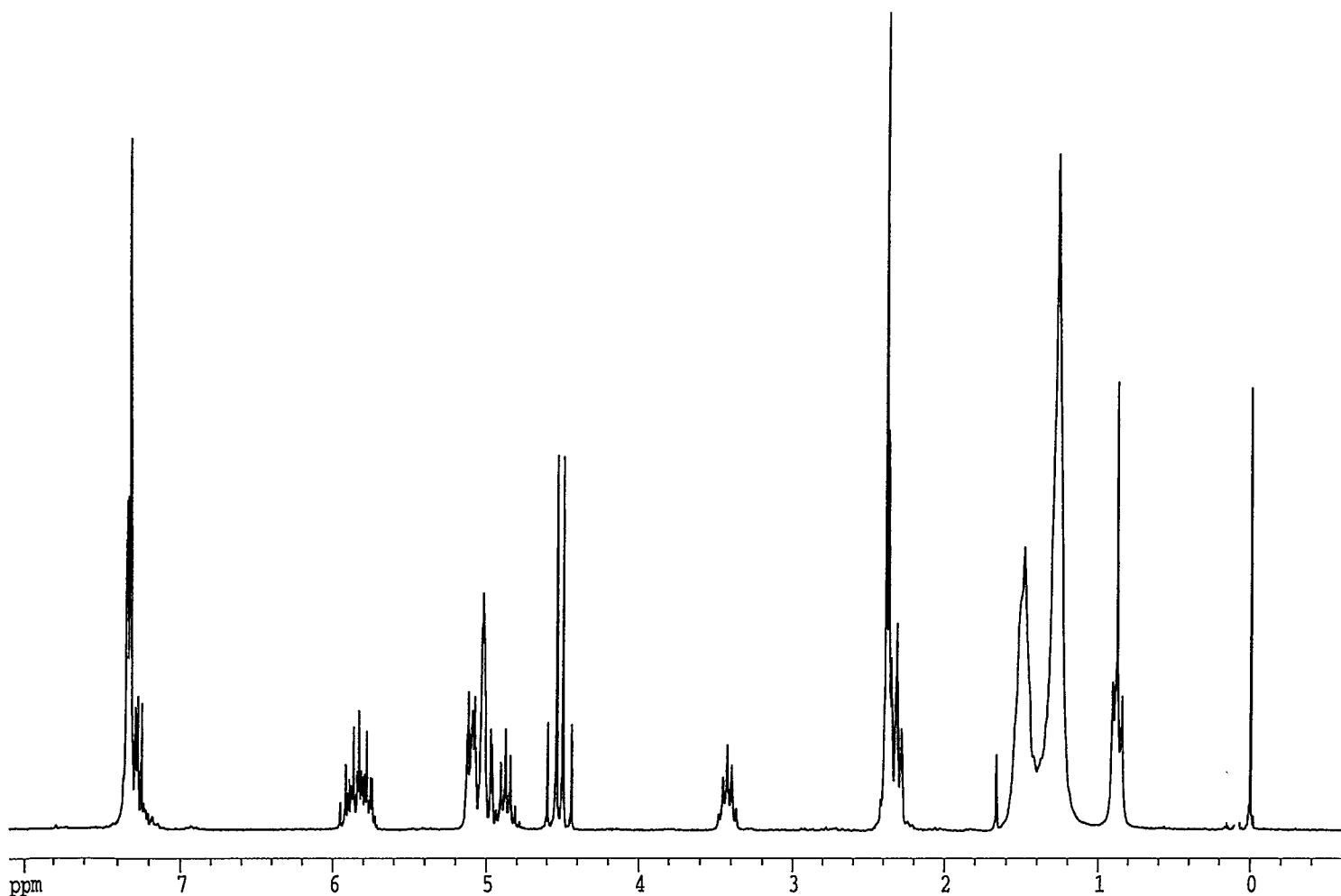
F2 - Processing parameters
 SI 16384
 SF 50.3233171 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 186.788 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
HL1 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

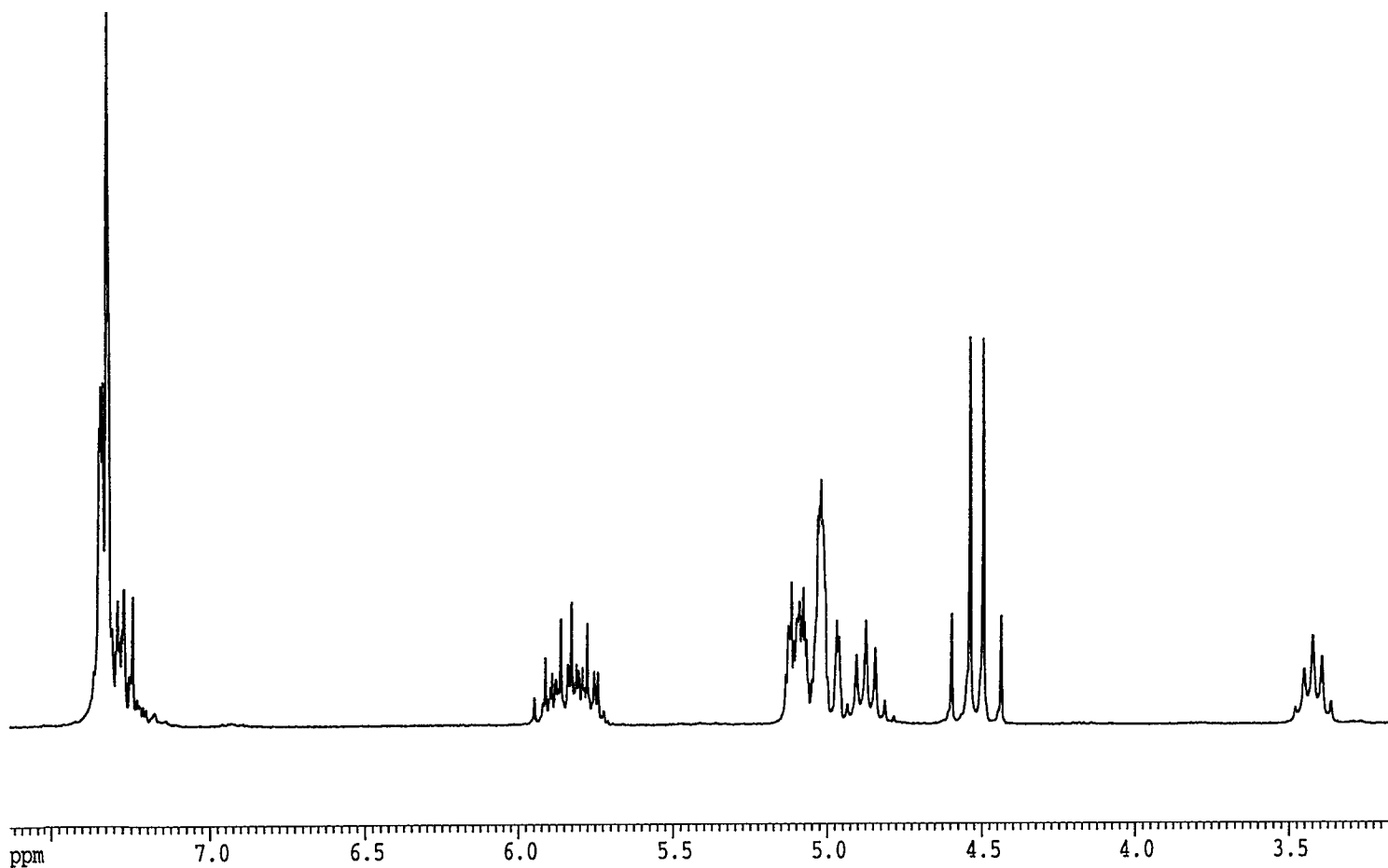
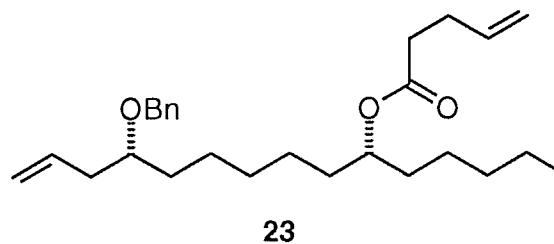
1D NMR plot parameters
CX 20.00 cm
F1P 8.110 ppm
F1 1623.09 Hz
F2P -0.658 ppm
F2 -131.64 Hz
PPMCM 0.43839 ppm/cm
HZCM 87.73654 Hz/cm

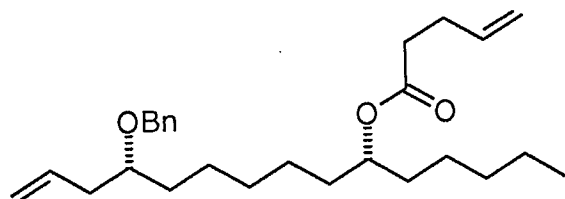
Current Data Parameters
 NAME JA220F
 EXPNO 106
 PROCNO 1
 DU mpi
 USER kl

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
 HL1 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 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

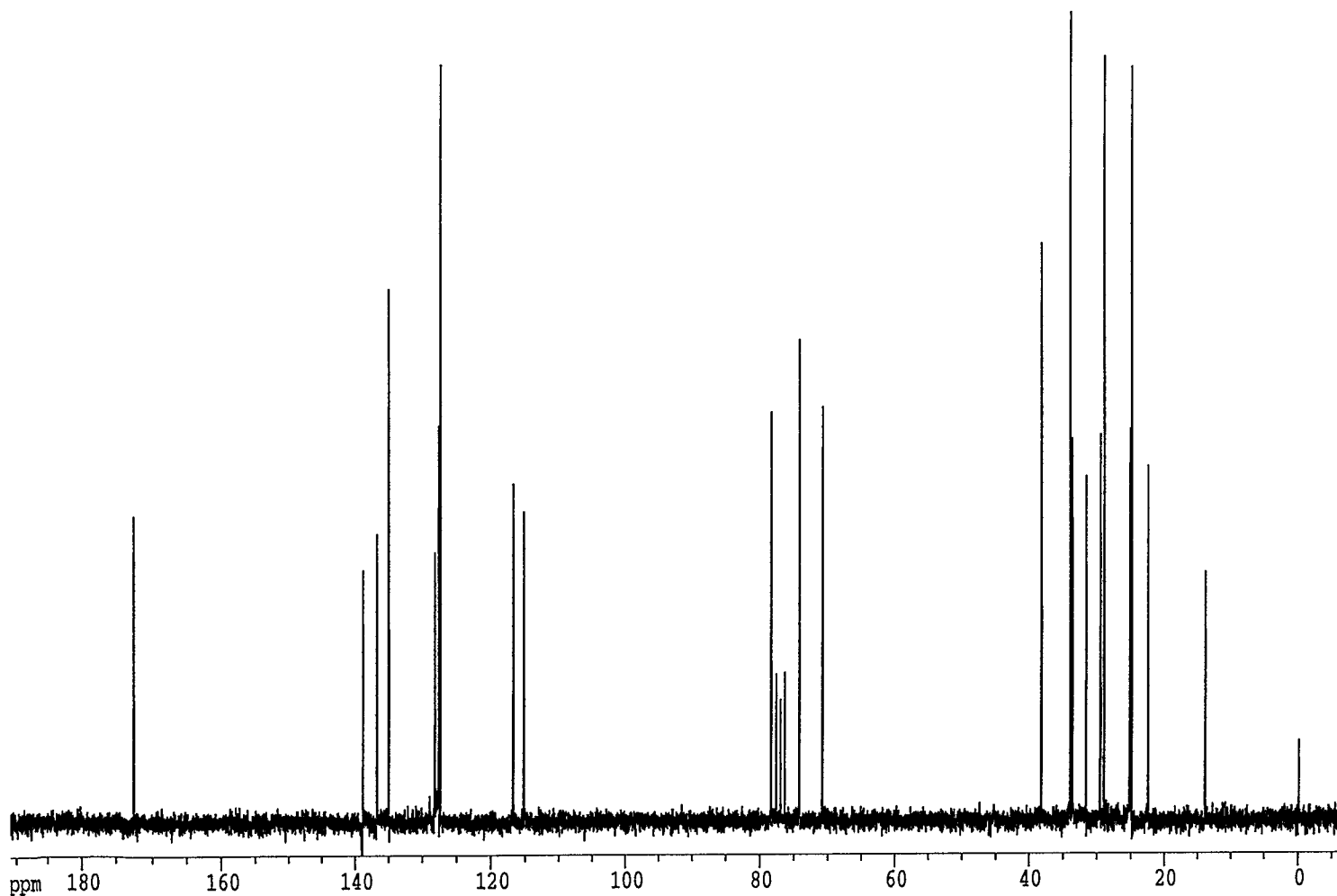
Date_ 970122
Time 12.54
INSTRUM ac200
PROBHD
PULPROG X53.AU
TD 32768
SOLVENT unknown
NS 1568
DS 0
SWH 14285.714 Hz
FIDRES 0.435965 Hz
AQ 1.1469300 sec
RG 640
DW 35.000 usec
DE 46.30 usec
TE 300.0 K
P1 15.50 usec
HL1 20 dB
D1 0.00100000 sec
DE 46.30 usec
SFO1 50.3287650 MHz
NUCLEUS unknown

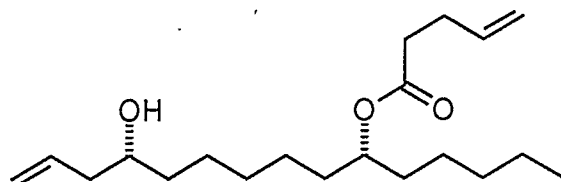
F2 - Processing parameters

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





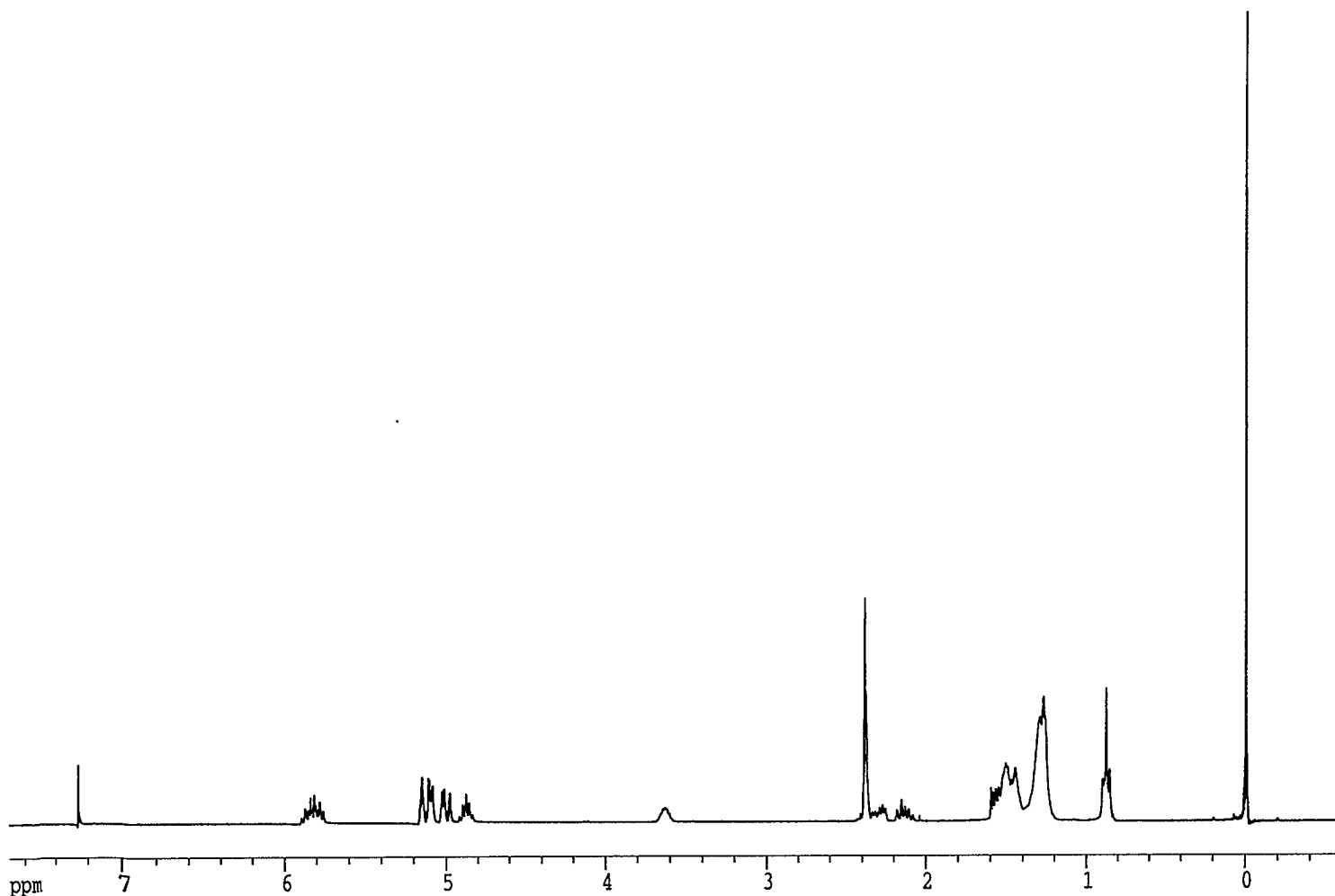
22

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 CDC13
 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
 SFO1 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

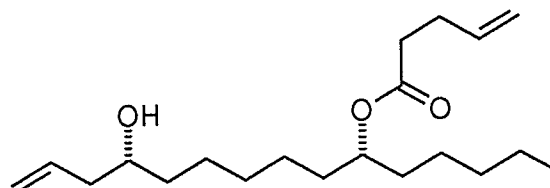


Current Data Parameters
 NAME jan07109
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

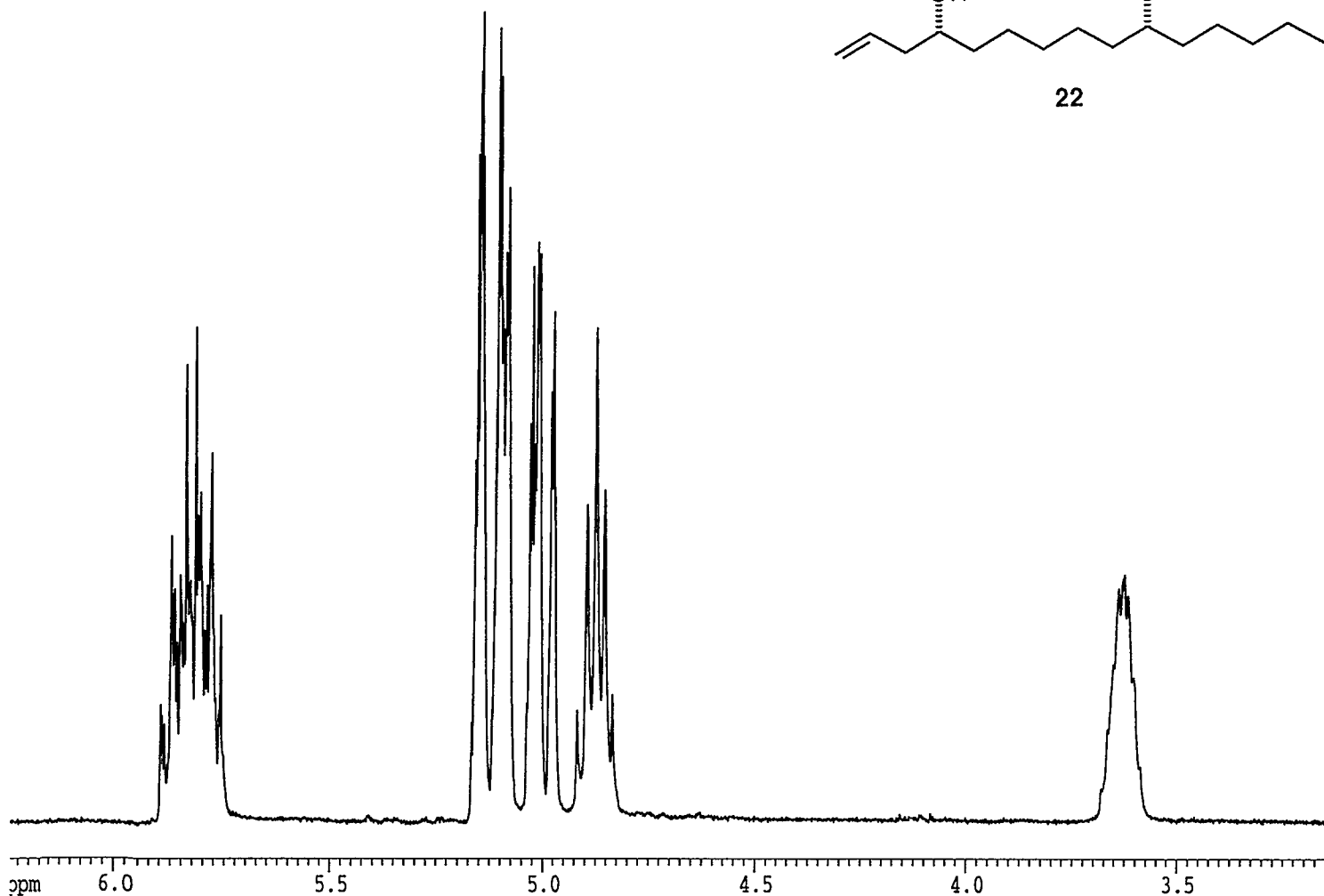
F2 - Acquisition Parameters
 Date_ 970107
 Time 14.20
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6249.967 Hz
 FIDRES 0.190734 Hz
 AQ 2.6214900 sec
 RG 512
 DW 80.000 usec
 DE 100.00 usec
 TE 302.0 K
 HL1 0 dB
 D1 1.00000000 sec
 P1 6.75 usec
 DE 100.00 usec
 SFO1 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



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
SFO1          300.1349393 MHz
NUCLEUS        1H

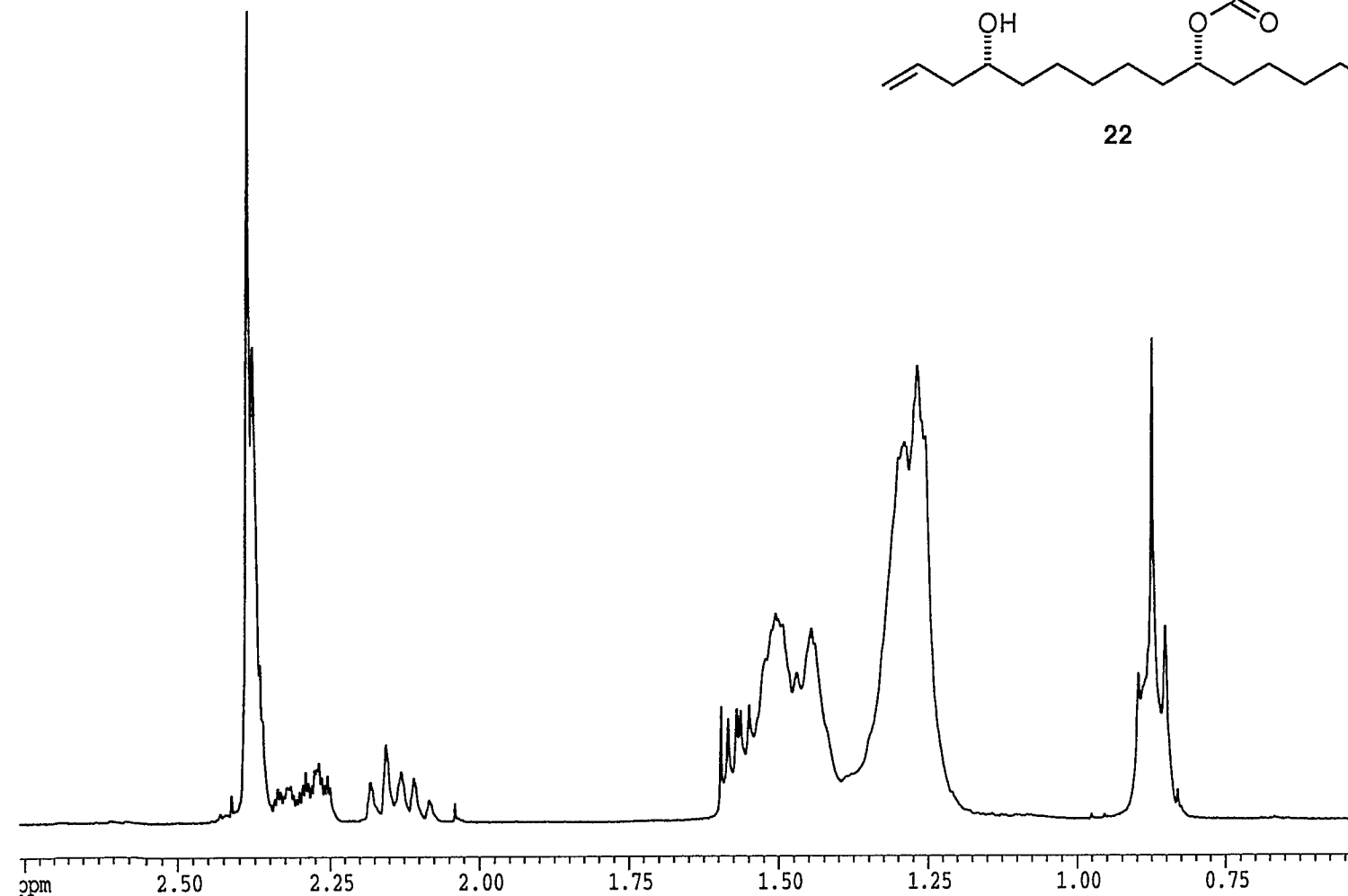
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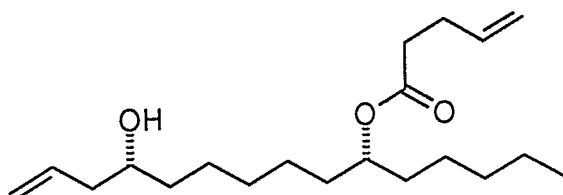
F2 - Processing parameters

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





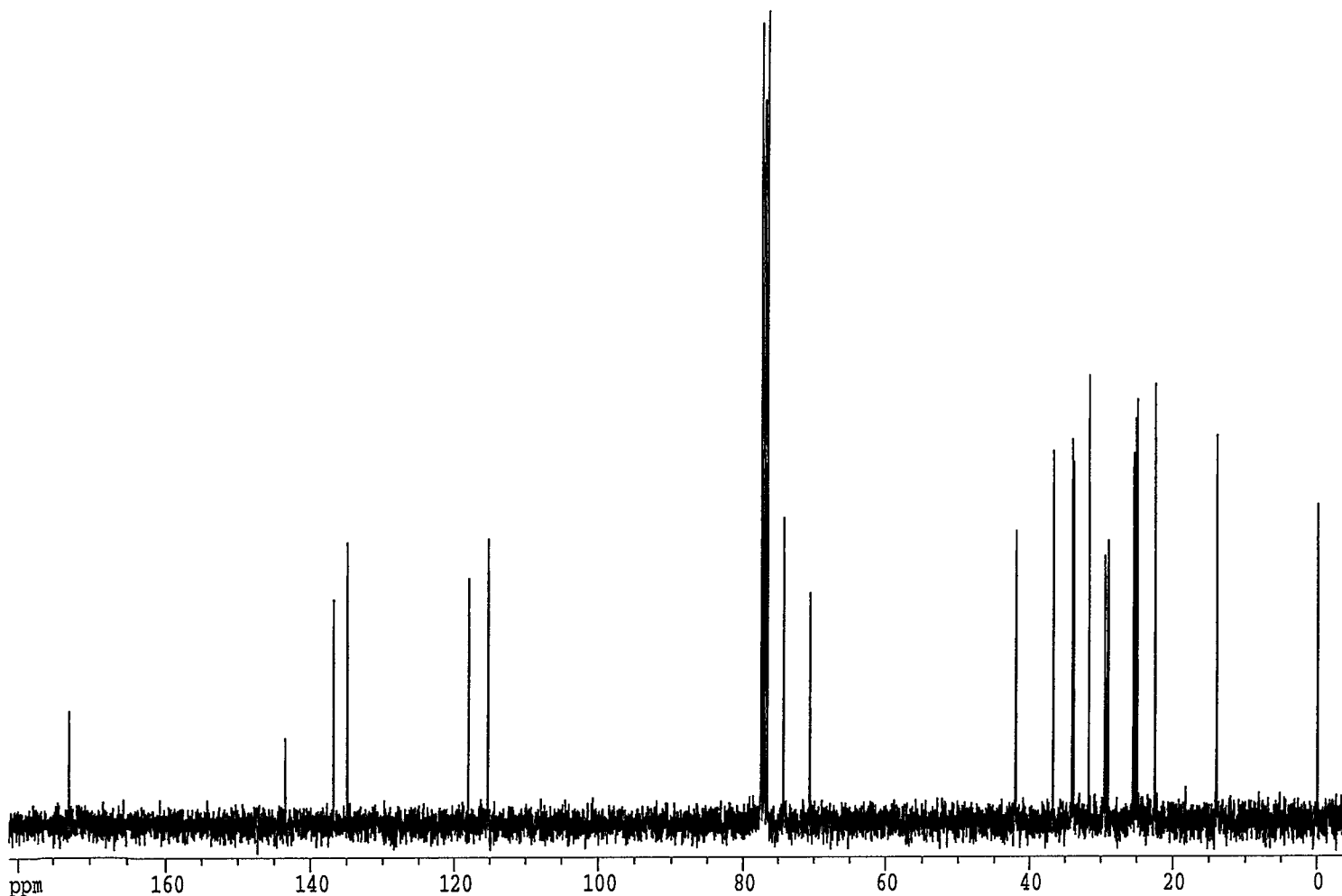
22

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

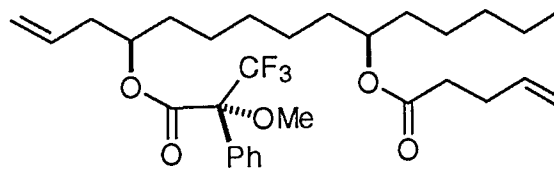
F2 - Processing parameters
 SI 32768
 SF 75.4685893 MHz
 WDW EM
 SSB 0
 LB 0.80 Hz
 GB 0
 PC 2.00

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**

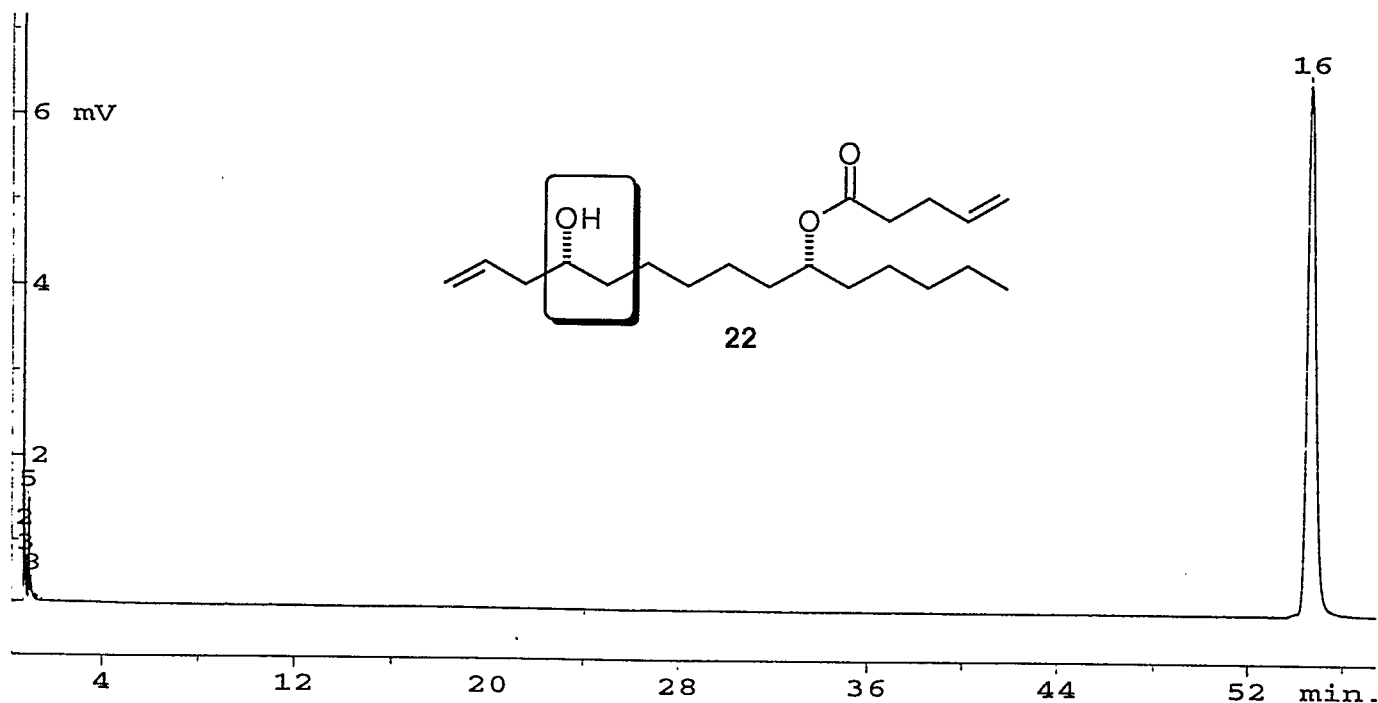
Current Data Parameters
 NAME apr04107
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

F2 - Acquisition Parameters
 Date 500000
 Time 6.33
 PULPROG zgpg30
 SOLVENT CDC13
 AQ 0.2621640 sec
 DW 4.0 usec
 RG 256
 NUCLEUS 19F
 SF01 282.3891972 MHz
 SF02 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
 SF01 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

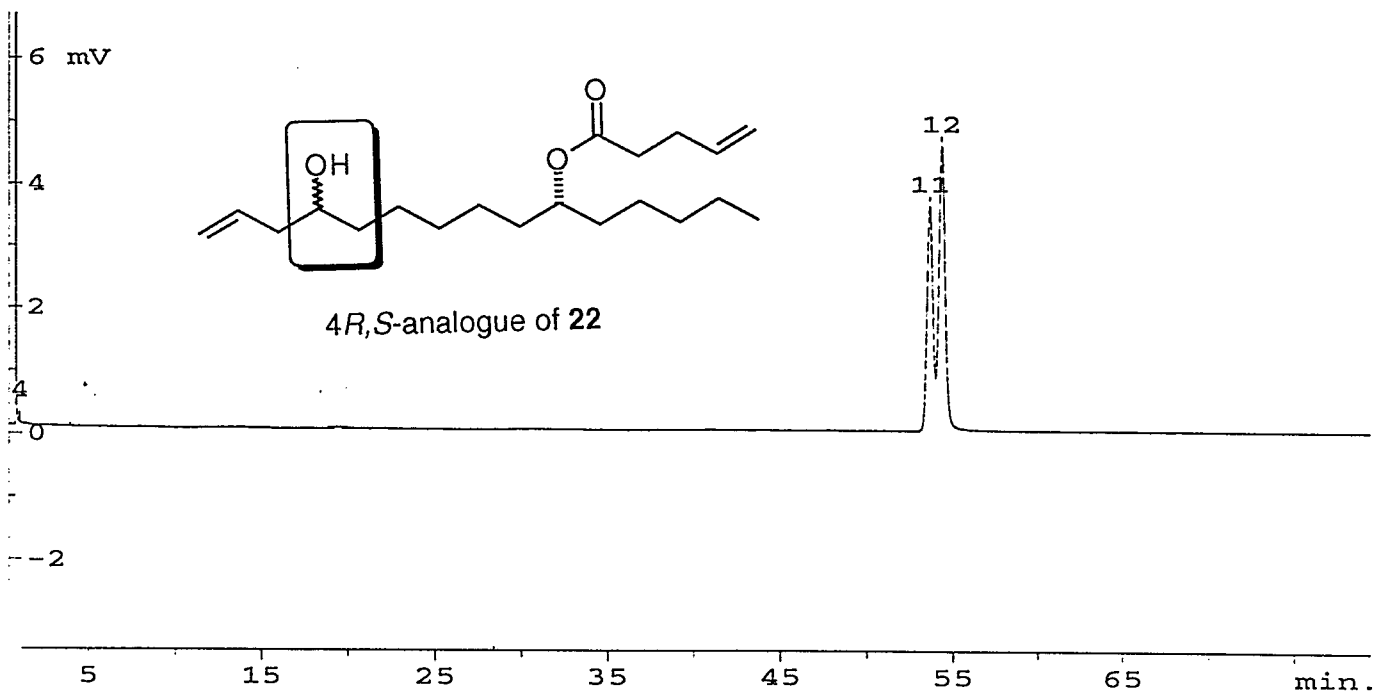


***** r127s844 ***** 23-Apr-97 11:45:36

No.	min.	area-%
16	54.66	100.0

15 peaks had been suppressed.

```
Instrument      : HEWLETT PACKARD 5890
Column         : 25MFS/20%TBCD (P) 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
```



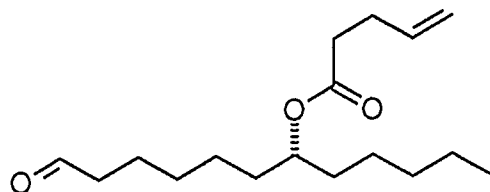
***** r127s449 ***** 24-Jan-97 10:20:08

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:

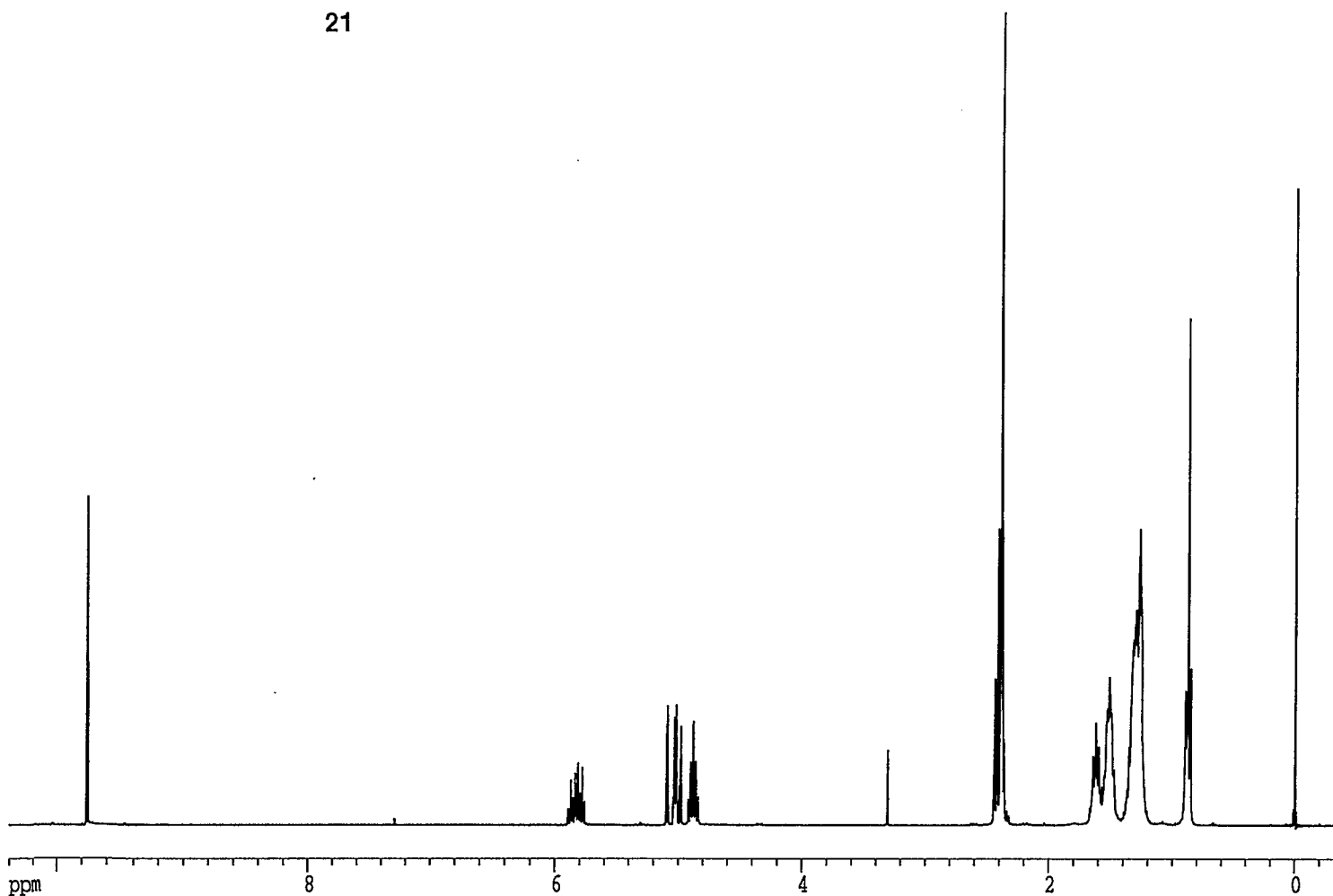
No.	min.	area-%
9	3.91	0.11
10	19.37	1.32
11	53.63	42.6
12	54.31	55.9

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 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
 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

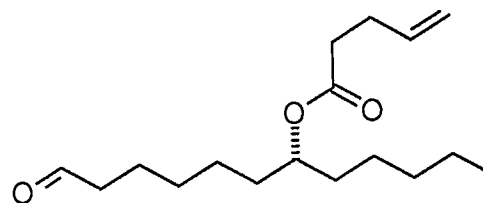
1D NMR plot parameters
 CX 20.00 cm
 F1P 10.416 ppm
 F1 3126.06 Hz
 F2P -0.384 ppm
 F2 -115.26 Hz
 PPMCM 0.53998 ppm/cm
 HZCM 162.06590 Hz/cm

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

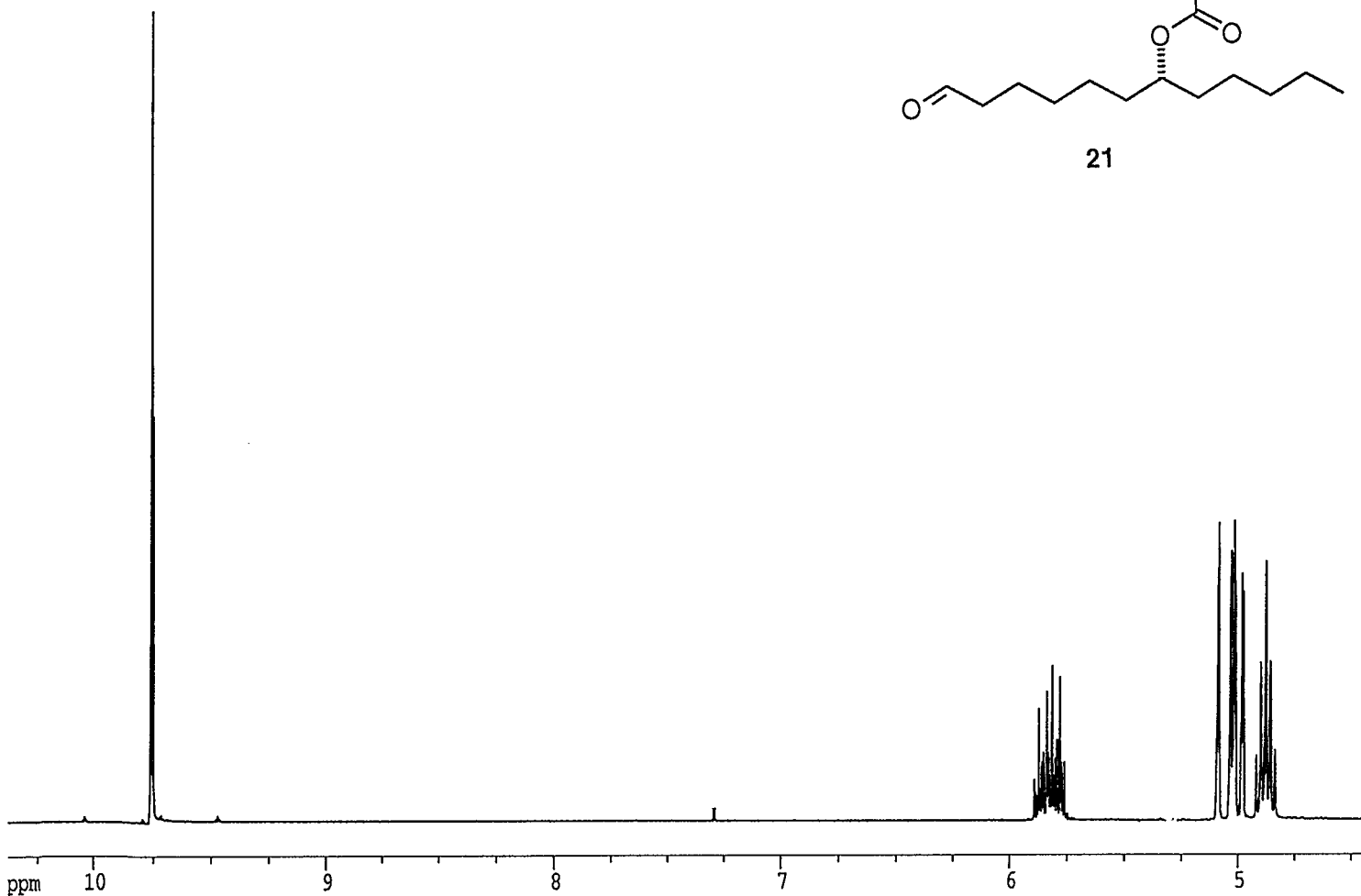
F2 - Acquisition Parameters
 Date_ 970103
 Time 10.21
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6249.967 Hz
 FIDRES 0.190734 Hz
 AQ 2.6214900 sec
 RG 128
 DW 80.000 usec
 DE 100.00 usec
 TE 302.0 K
 HL1 0 dB
 D1 1.00000000 sec
 P1 6.75 usec
 DE 100.00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

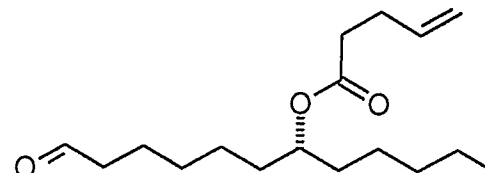


Current Data Parameters
 NAME jan03101
 EXPNO 10
 PROCNO 1
 DU mpi
 USER kl

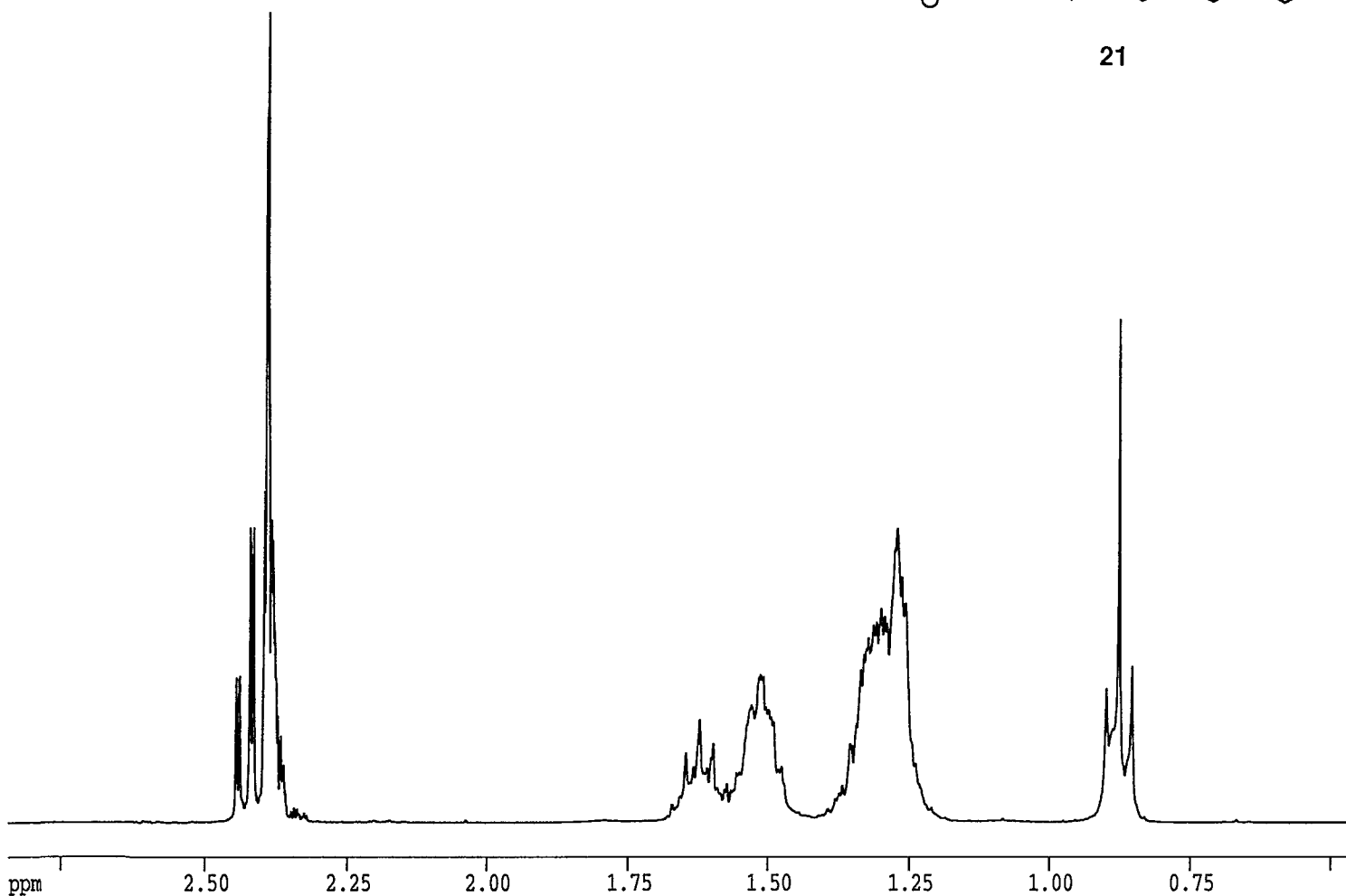
F2 - Acquisition Parameters
 Date_ 970103
 Time 10.21
 INSTRUM amx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6249.967 Hz
 FIDRES 0.190734 Hz
 AQ 2.6214900 sec
 RG 128
 DW 80.000 usec
 DE 100.00 usec
 TE 302.0 K
 HL1 0 dB
 D1 1.00000000 sec
 P1 6.75 usec
 DE 100.00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

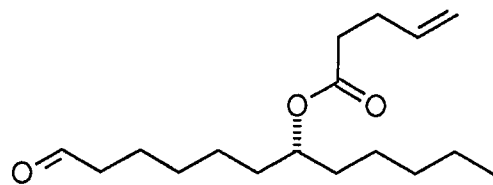
F2 - Processing parameters
 SI 32768
 SF 300.1333577 MHz
 WDM EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 8.00

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





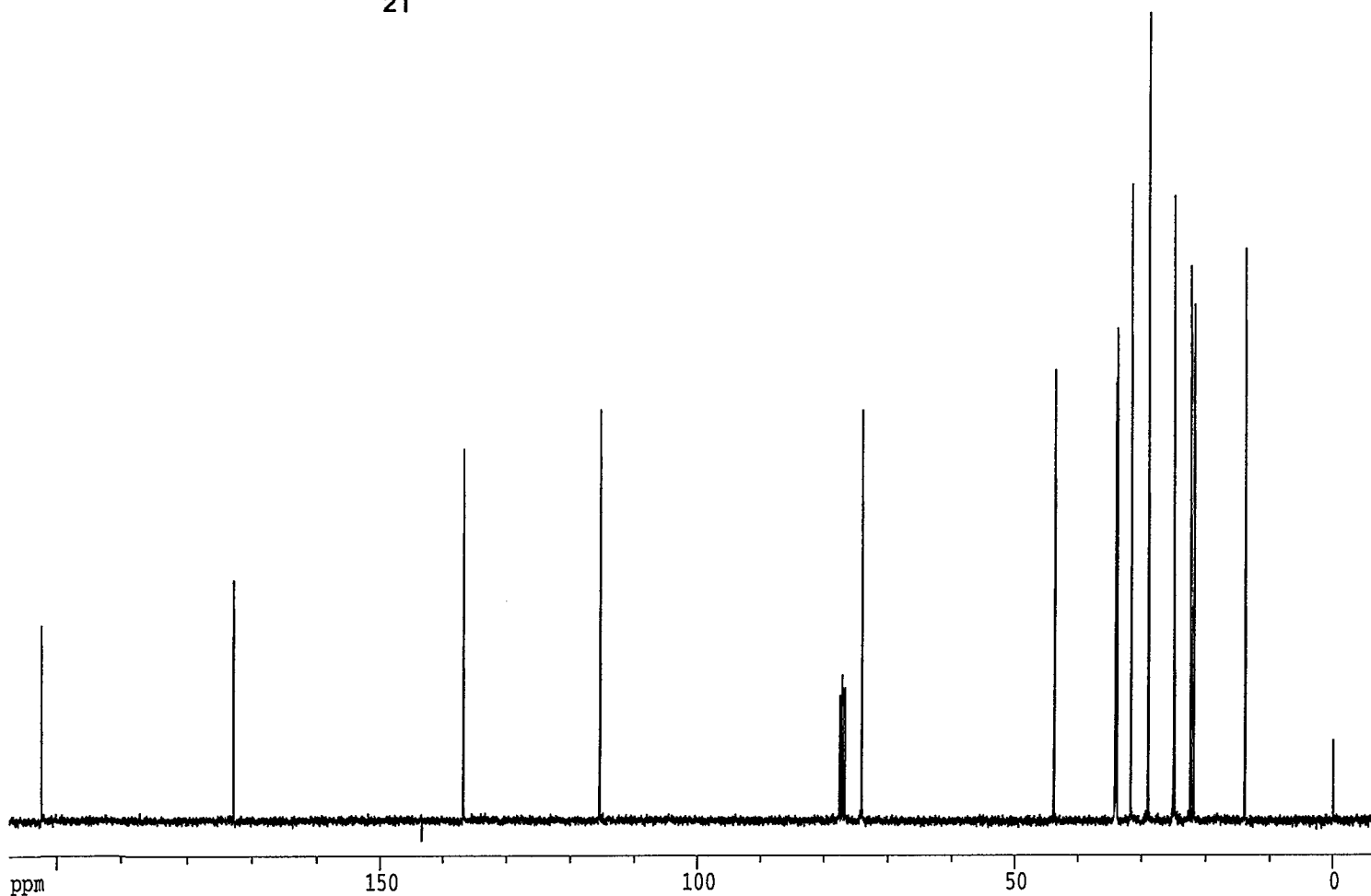
21

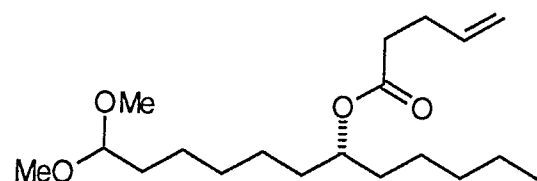
Current Data Parameters
 NAME jan03101
 EXPNO 11
 PROCNO 1
 DU mpi
 USER kl

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

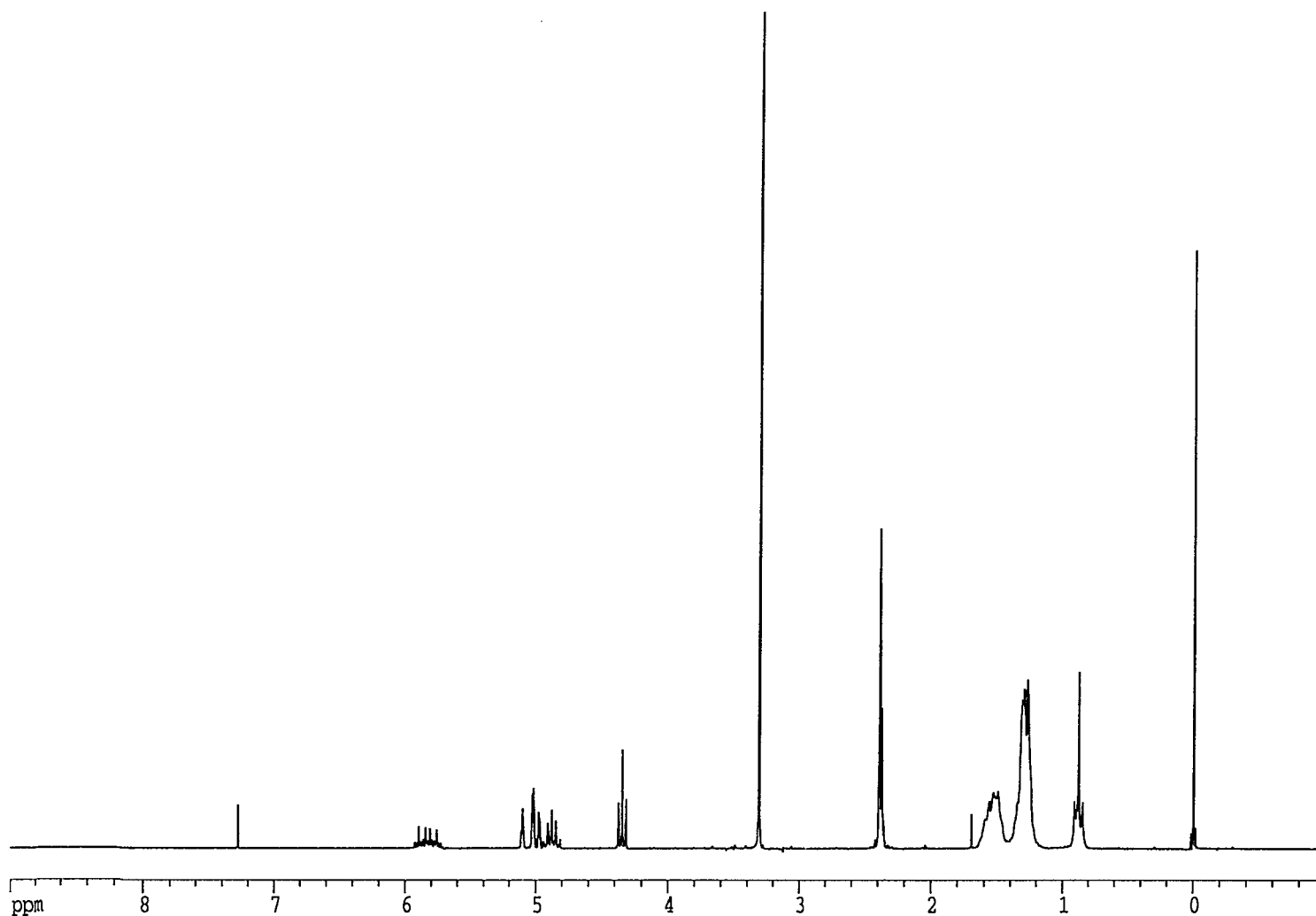
F2 - Processing parameters
 SI 32768
 SF 75.4685862 MHz
 WDW EM
 SSB 0
 LB 0.80 Hz
 GB 0
 PC 2.00

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

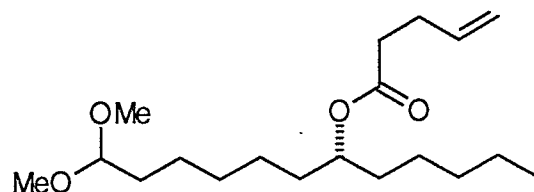


Current Data Parameters
 NAME DE180F
 EXPNO 128
 PROCNO 1
 DU mpi
 USER kl

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 1.00000000 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



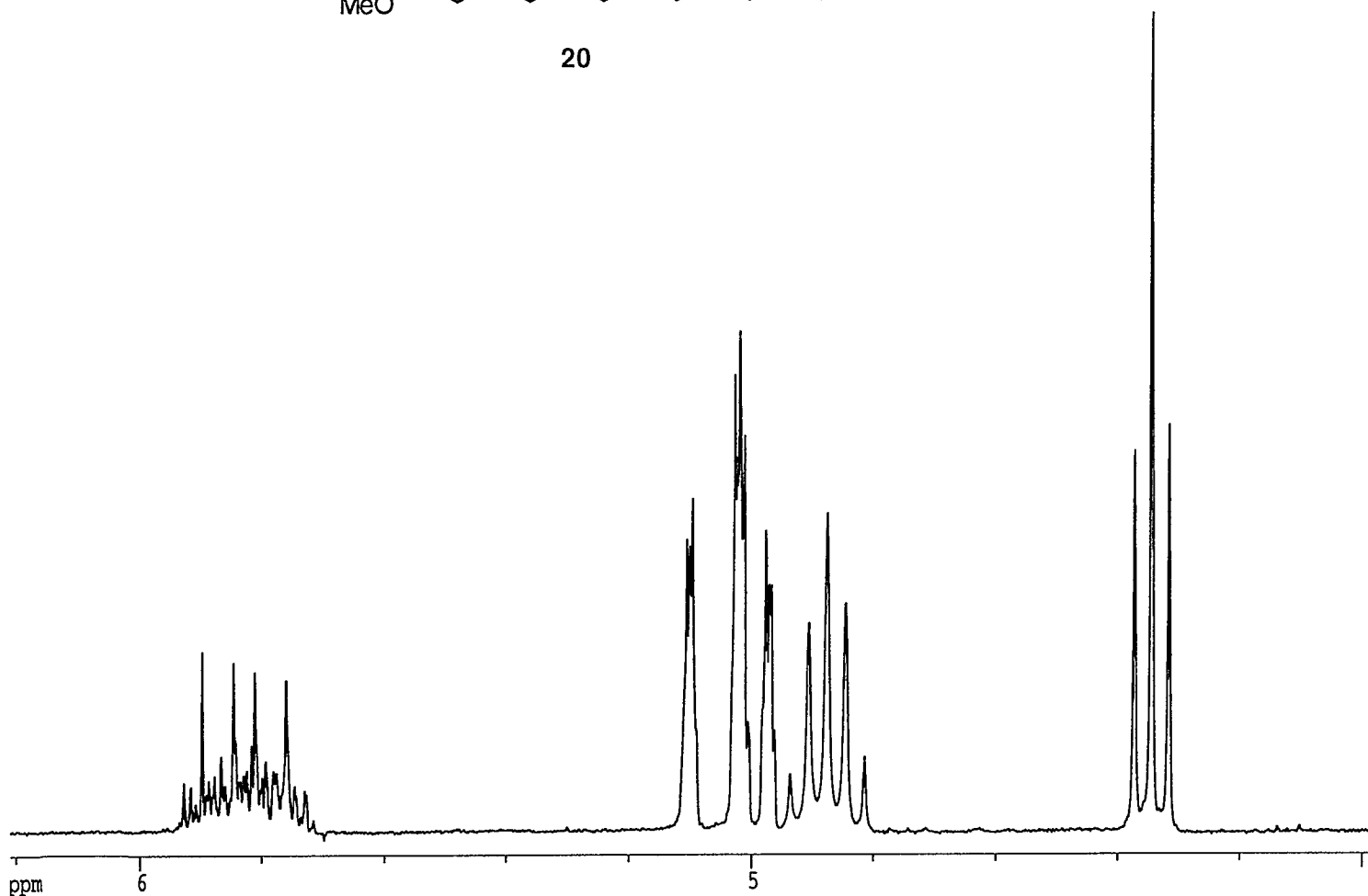
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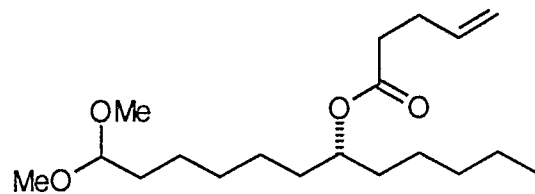
Current Data Parameters
 NAME DE180F
 EXPNO 128
 PROCNO 1
 DU mpi
 USER kl

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 1.00000000 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





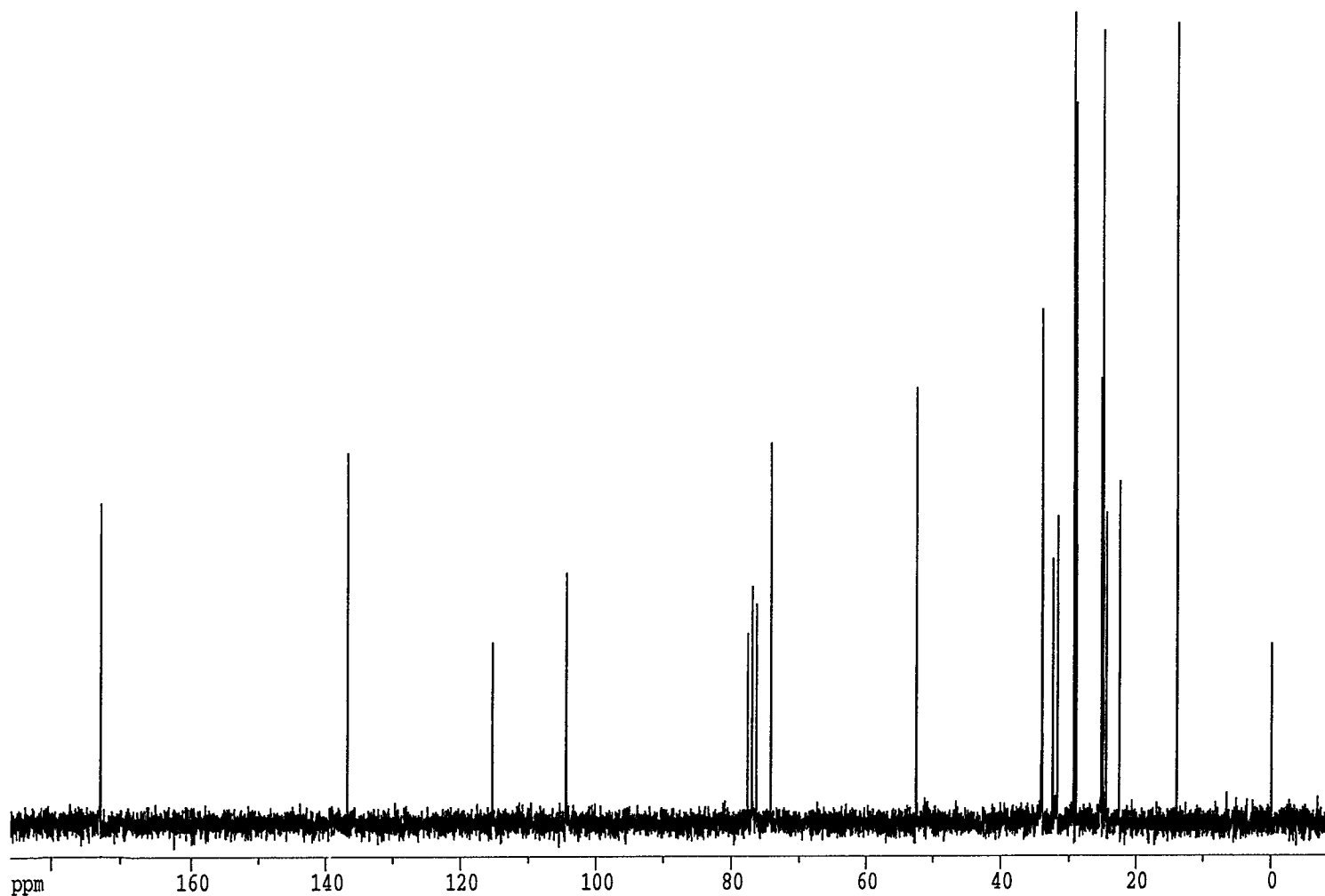
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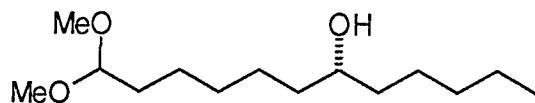
Current Data Parameters
 NAME DE181F
 EXPNO 128
 PROCNO 1
 DU mpi
 USER kl

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

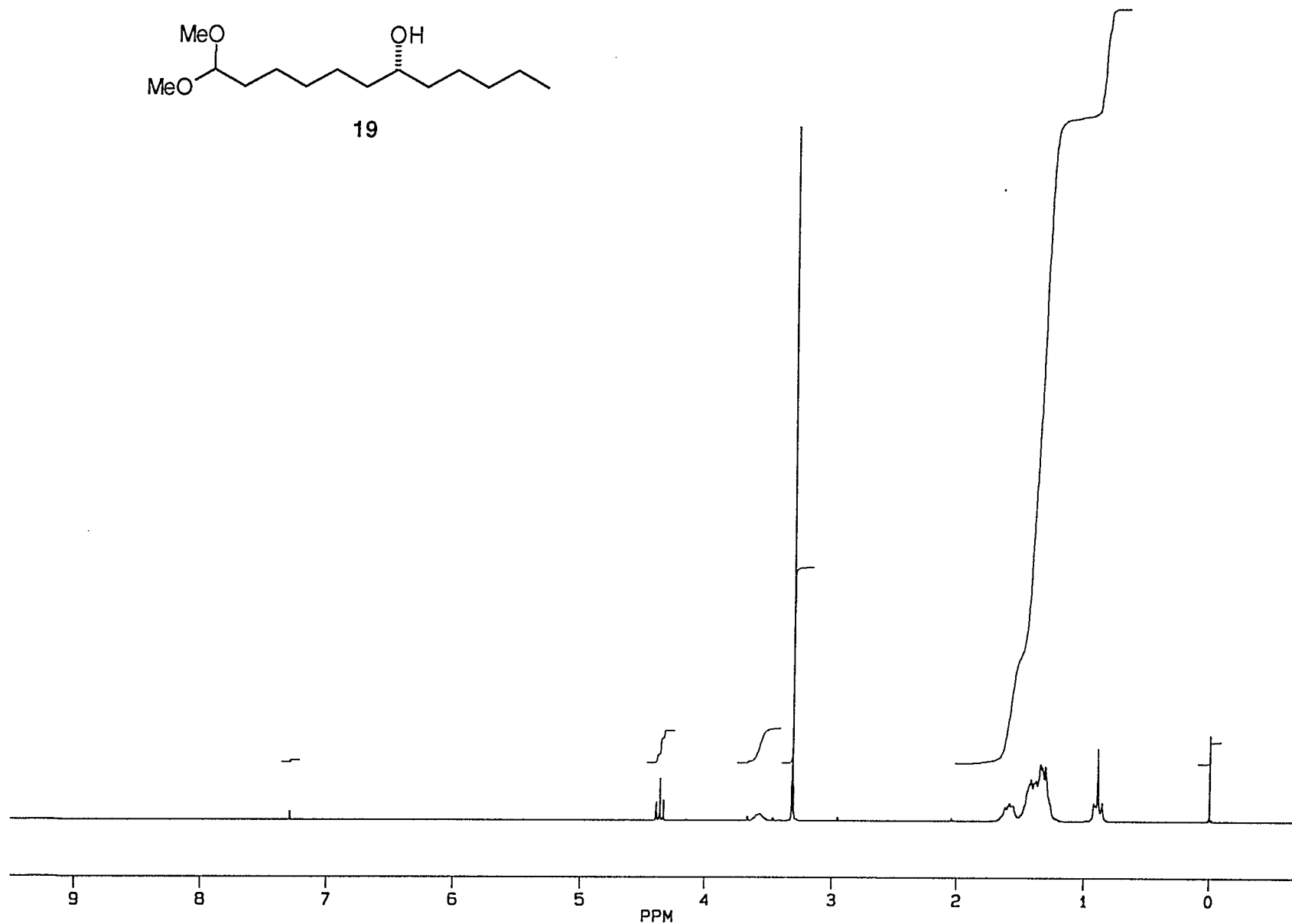
F2 - Processing parameters
 SI 16384
 SF 50.3233171 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

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



DE160F.141
AU PROG:
X51.AU
DATE 17-12-96
TIME 6: 08

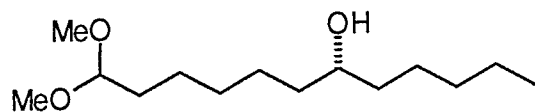
SA.NA LGM07601
SA.NO DE16 141
SOLVENT CDCl3
SF 200.132
SY 80.0
O1 3239.000
SI 32768
TD 32768
SW 4032.258
HZ/PT .246

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

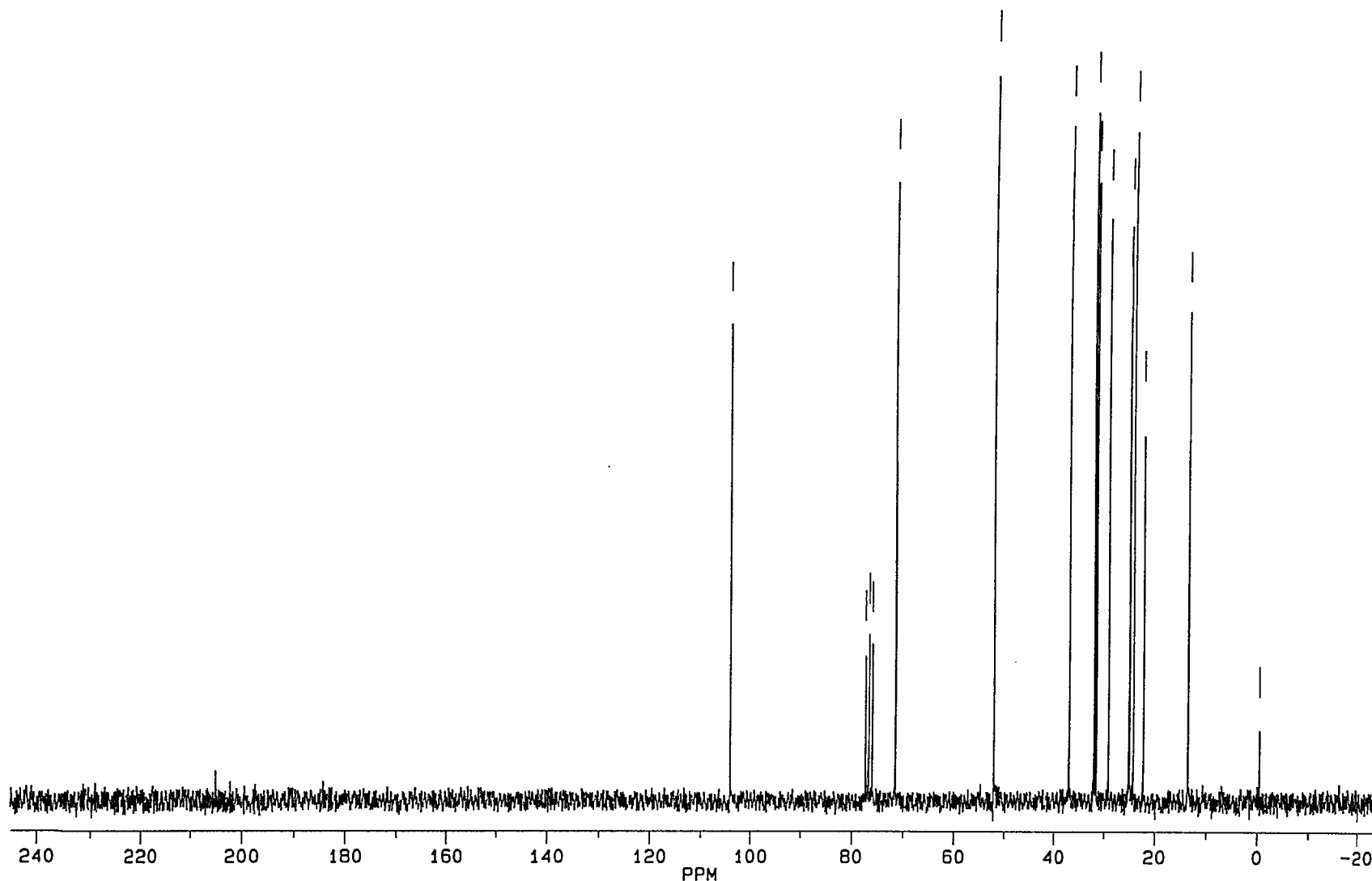
FW 5100
O2 2228.997
DP 63L P0

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



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

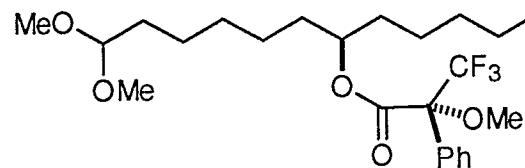
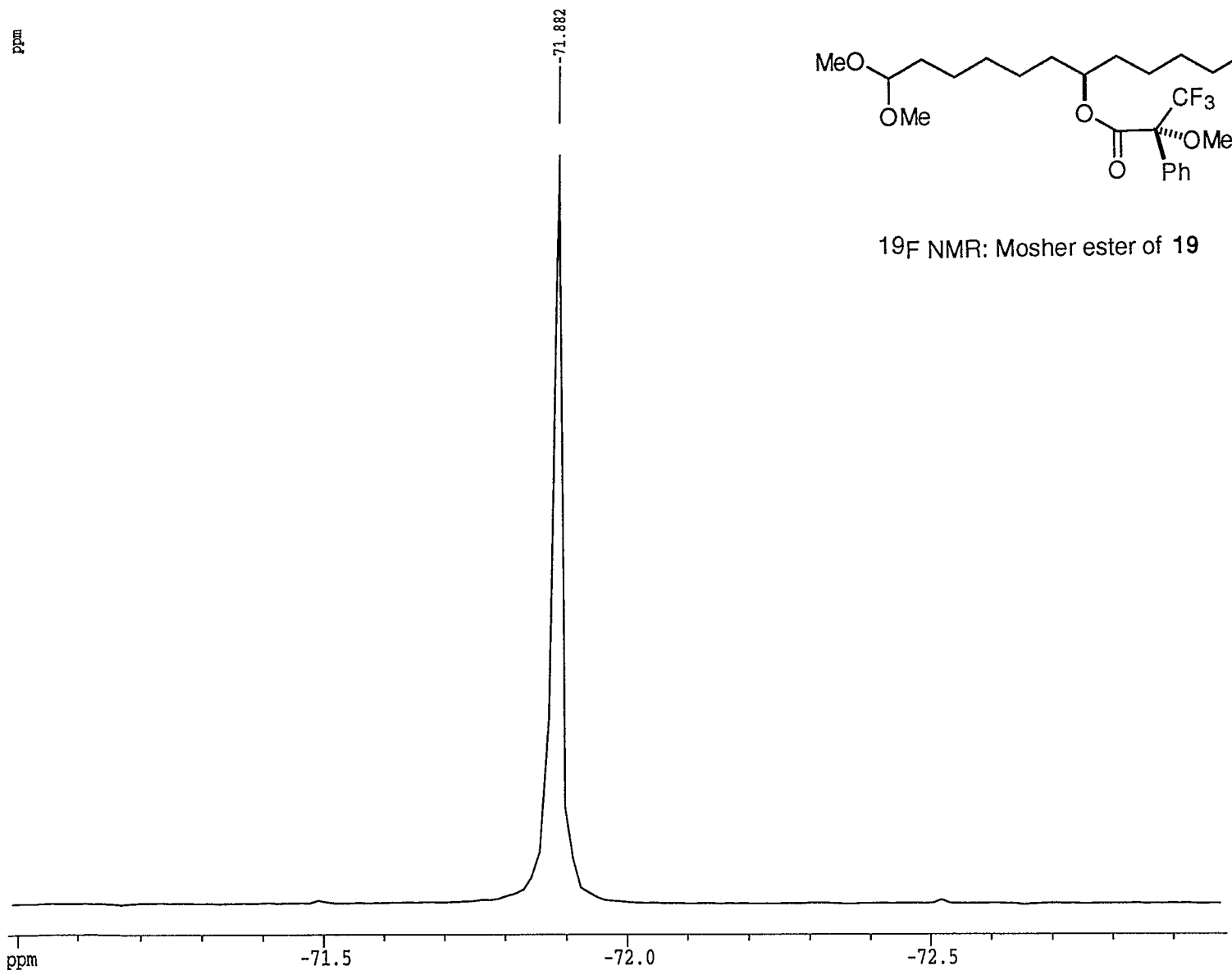
PW 0.0
RD 0.0
AQ 1.147
RG 640
NS 800
TE 300

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 RUN

ppm



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
 SF01 282.3891972 MHz
 SF02 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
 SF01 282.3891972 MHz
 SWH 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