Facialselectivity and Stereospecificity in the (4+3) Cycloaddition of Epoxy Enol Silanes

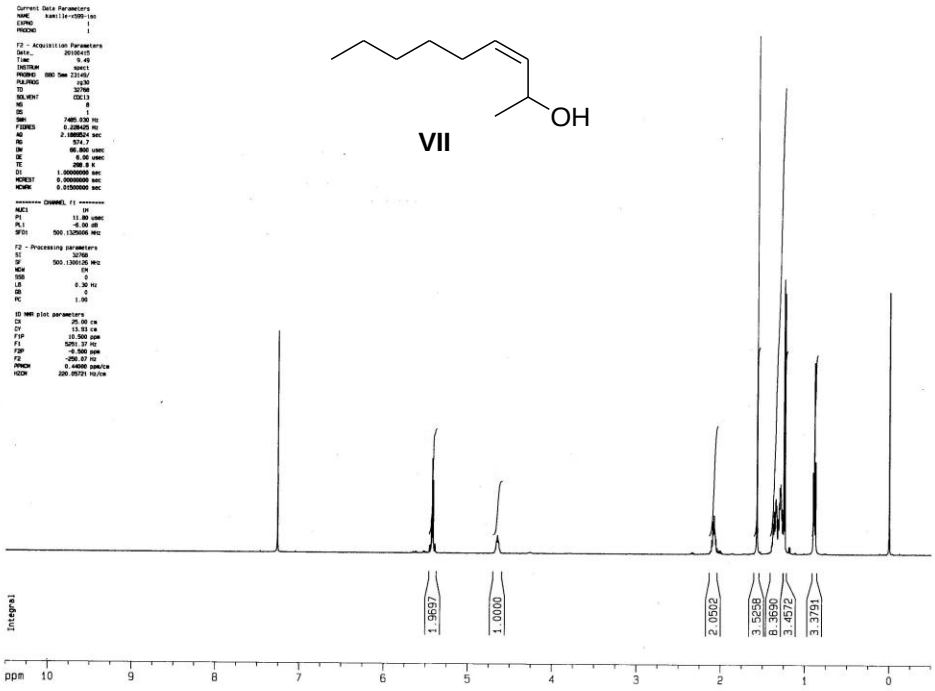
Brian Lo, Pauline Chiu*

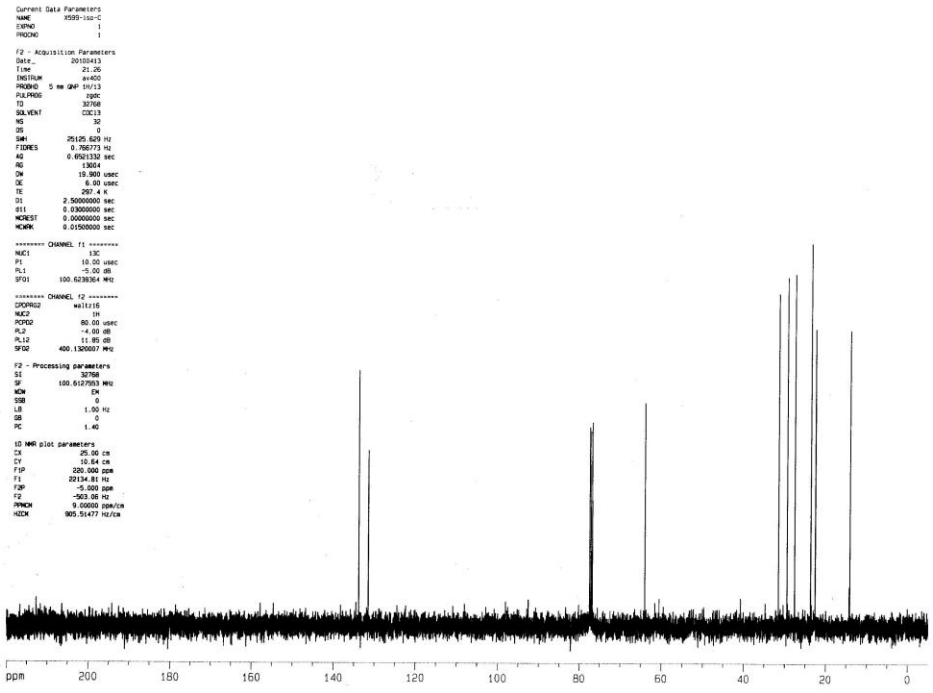
Department of Chemistry, the University of Hong Kong, Pokfulam Road, Hong Kong

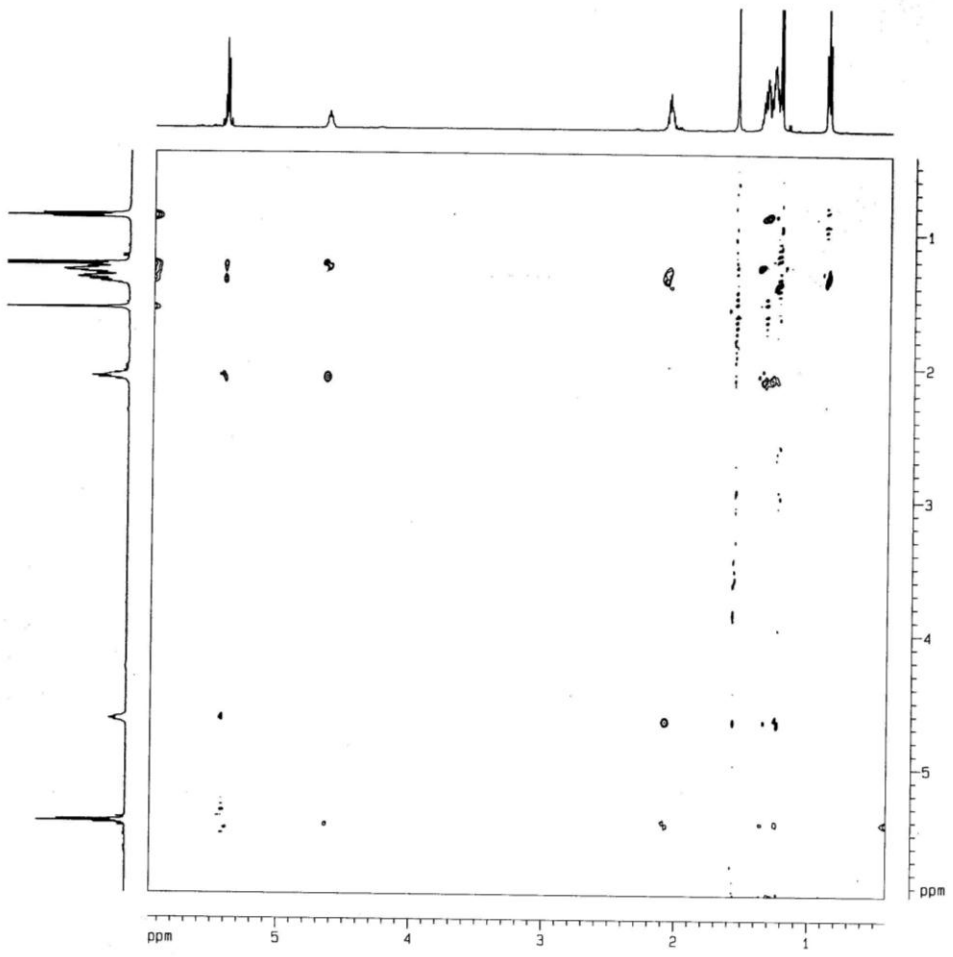
Supporting Information - Spectra

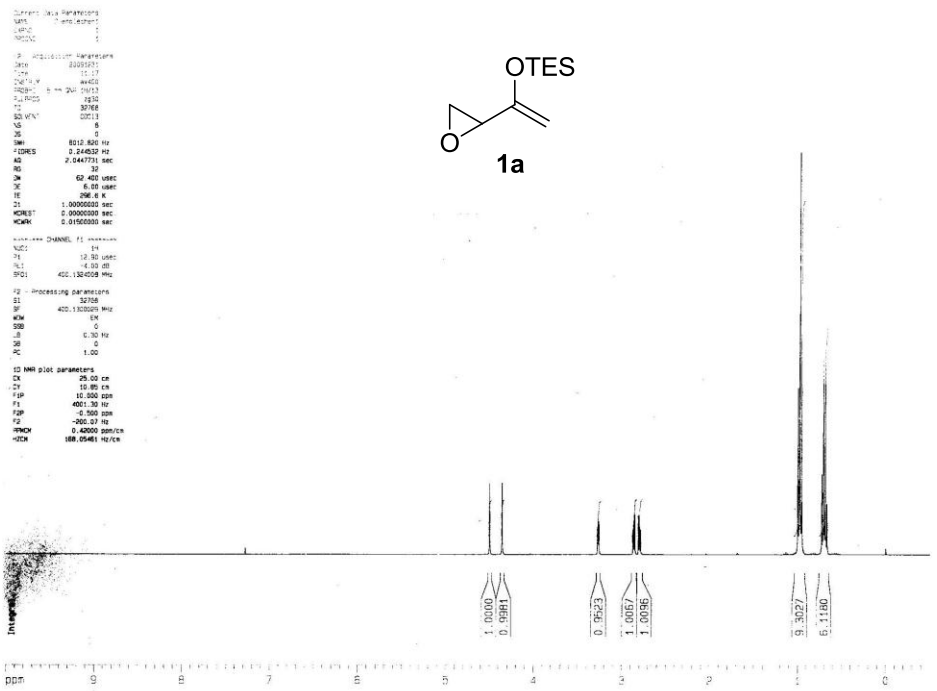
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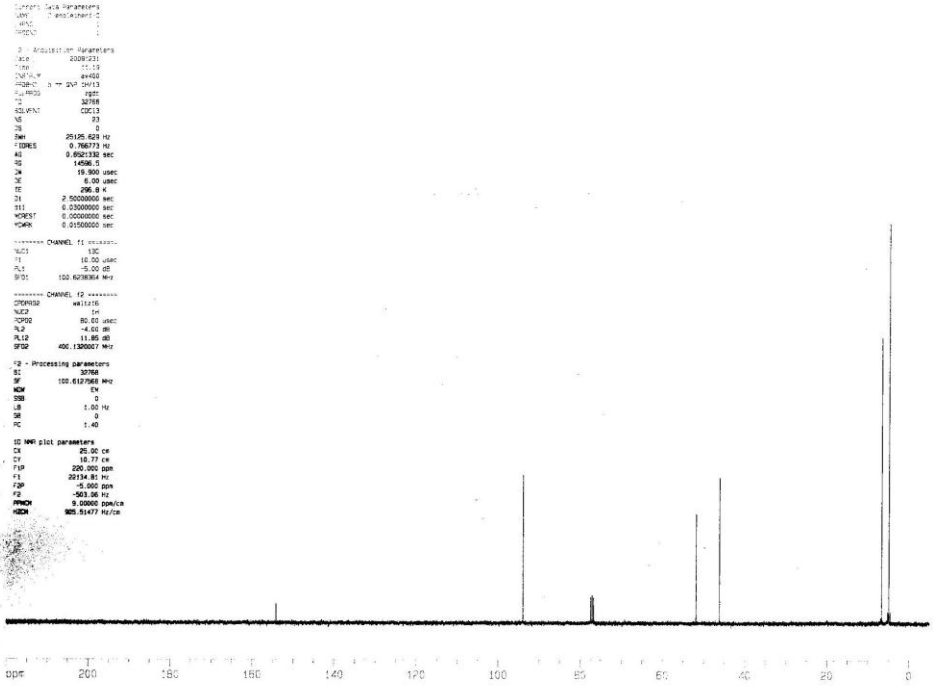
NMR Spectra for Cycloaddition reaction precursors.....S47-S61
NMR Spectra for Cycloaddition reaction products.....S62-S166

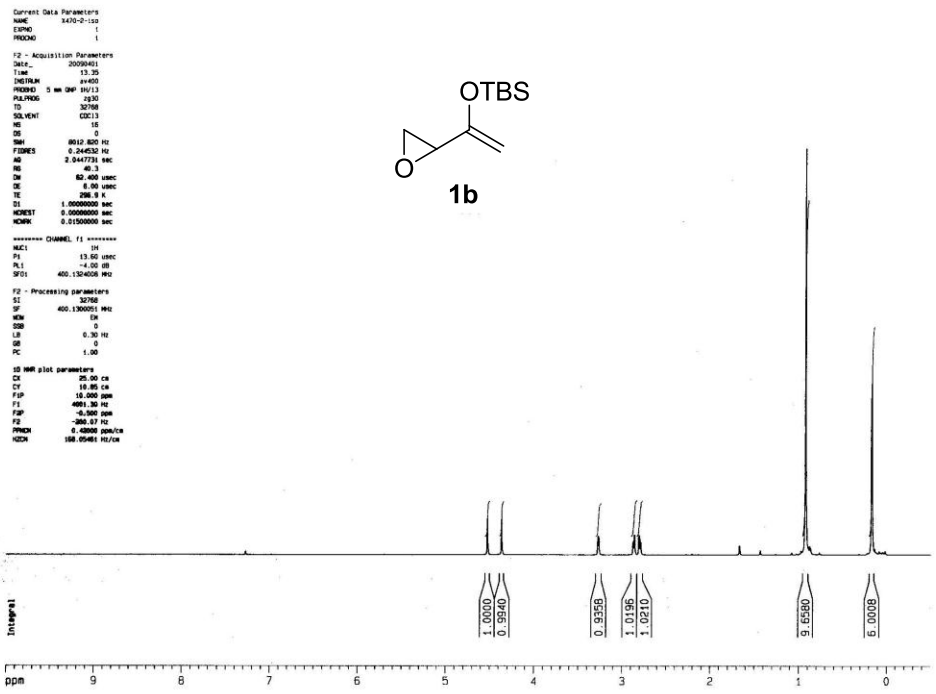

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FIDRES 0.228425 Hz
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MCWRK 0.01500000 sec
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F2 - Processing parameters
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LB 0.30 Hz
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CY 13.93 cm
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F1 5251.37 Hz
F2P -0.500 ppm
F2 -250.07 Hz
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HZCM 220.05721 Hz/cm
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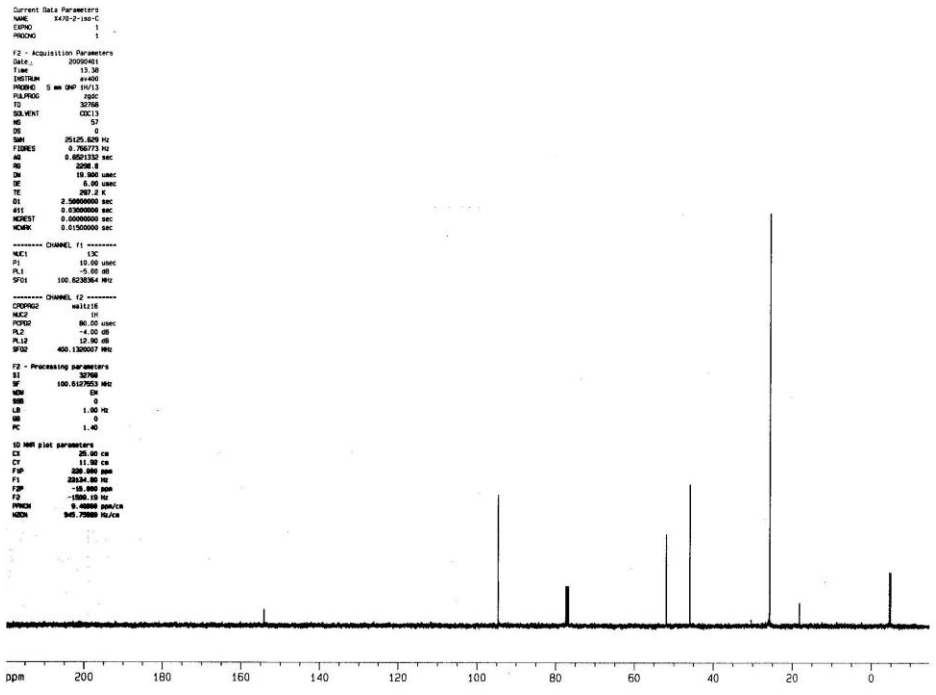

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TD 32768
SOLVENT CDCl3
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SWH 25125.629 Hz
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AQ 0.6521332 sec
RG 13004
DW 19.900 usec
DE 6.00 usec
TE 297.4 K
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D11 0.03000000 sec
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P1 10.00 usec
PL1 -5.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
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NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 11.85 dB
SFO2 400.1320007 MHz
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SF 100.6127953 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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CY 19.64 cm
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F1 22134.81 Hz
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HZCM 905.51477 Hz/cm
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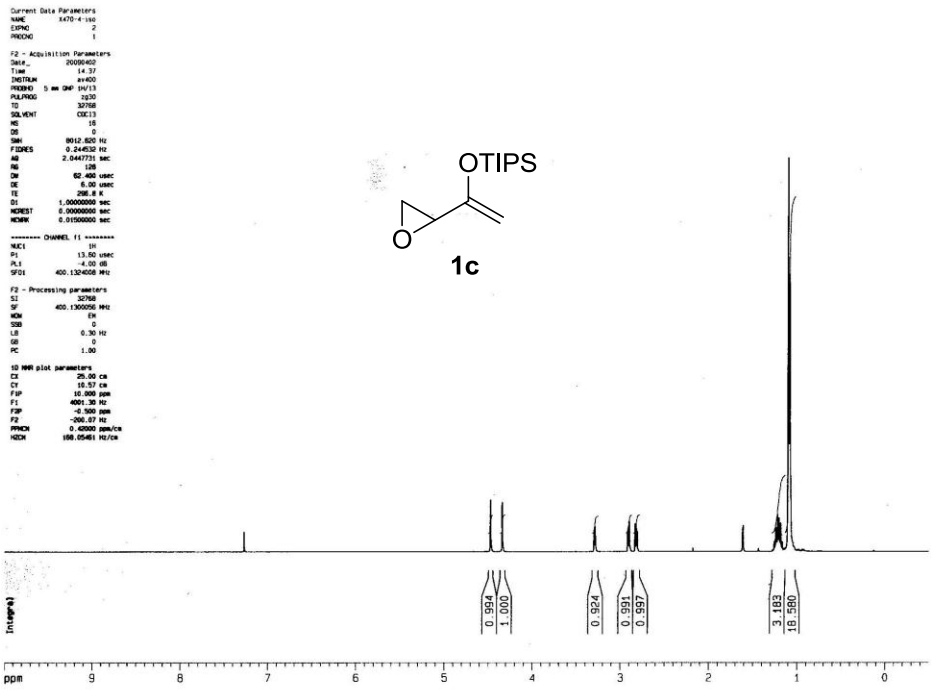

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ppm
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2
3
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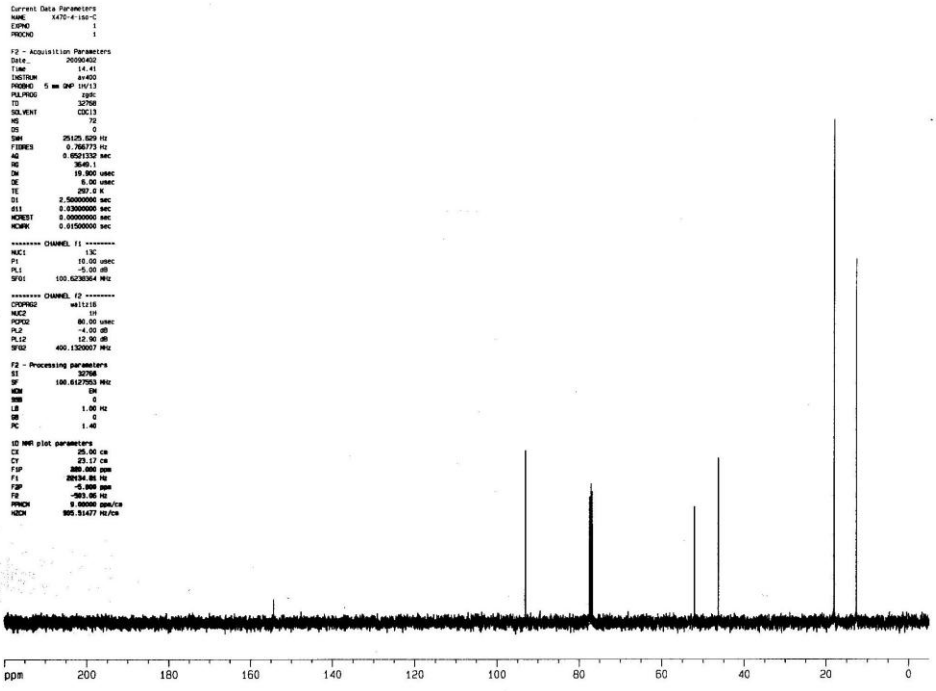

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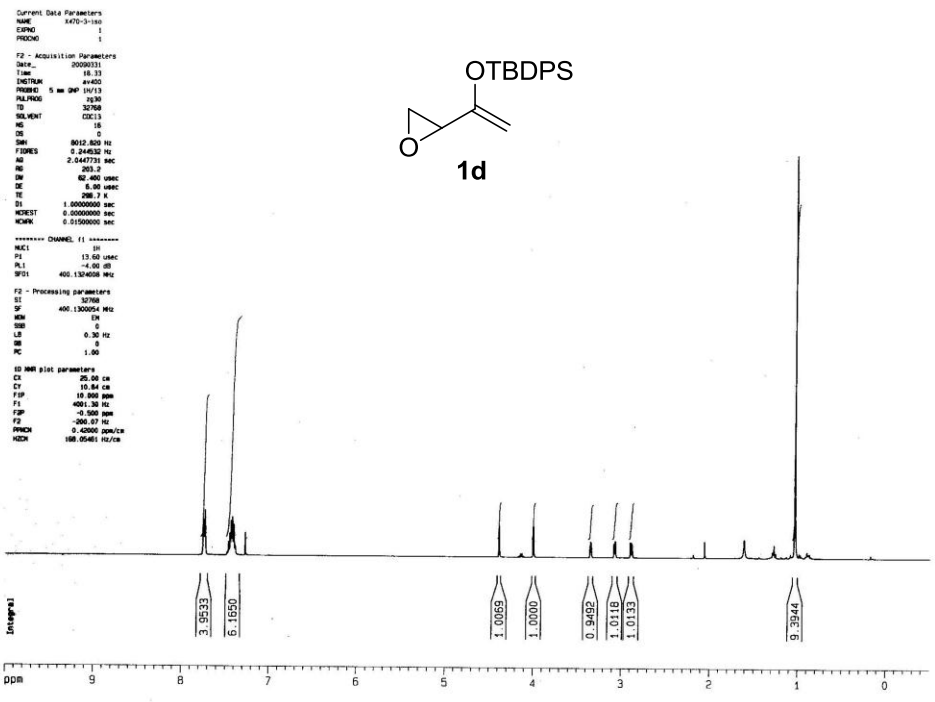

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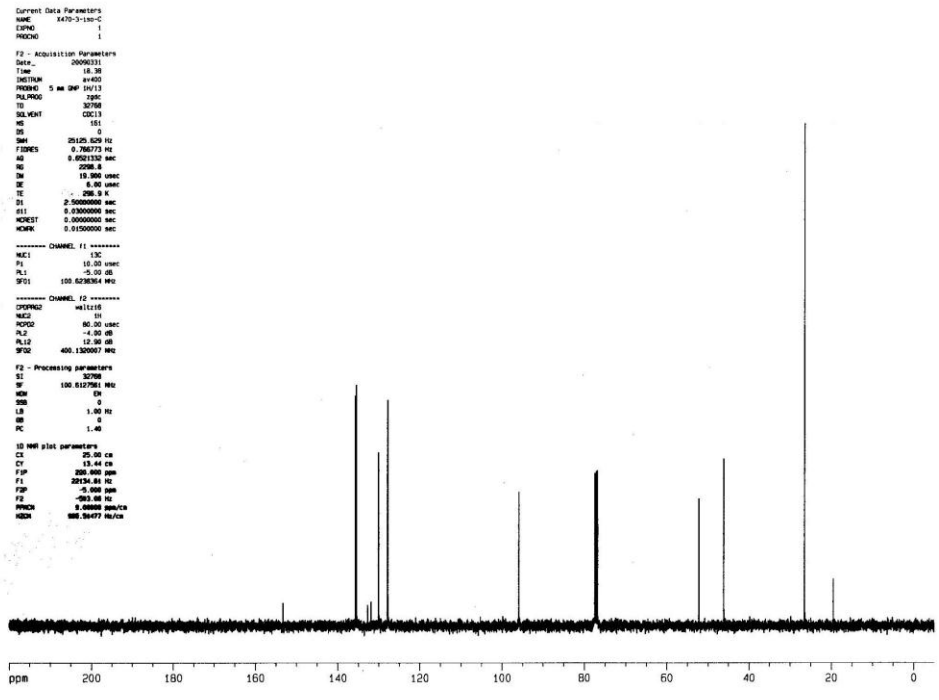

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0

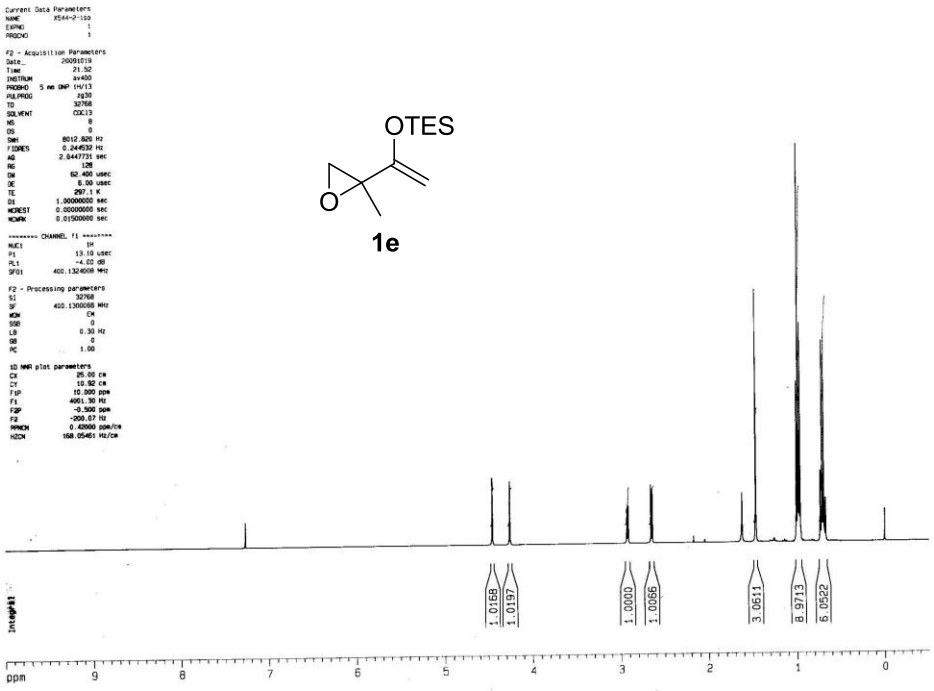

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140
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100
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60
40
20
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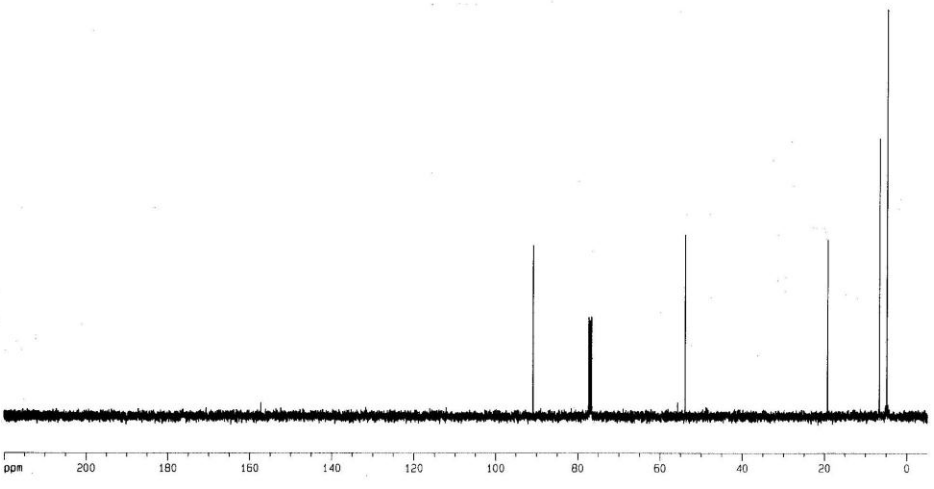

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7
6
5
4
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2
1
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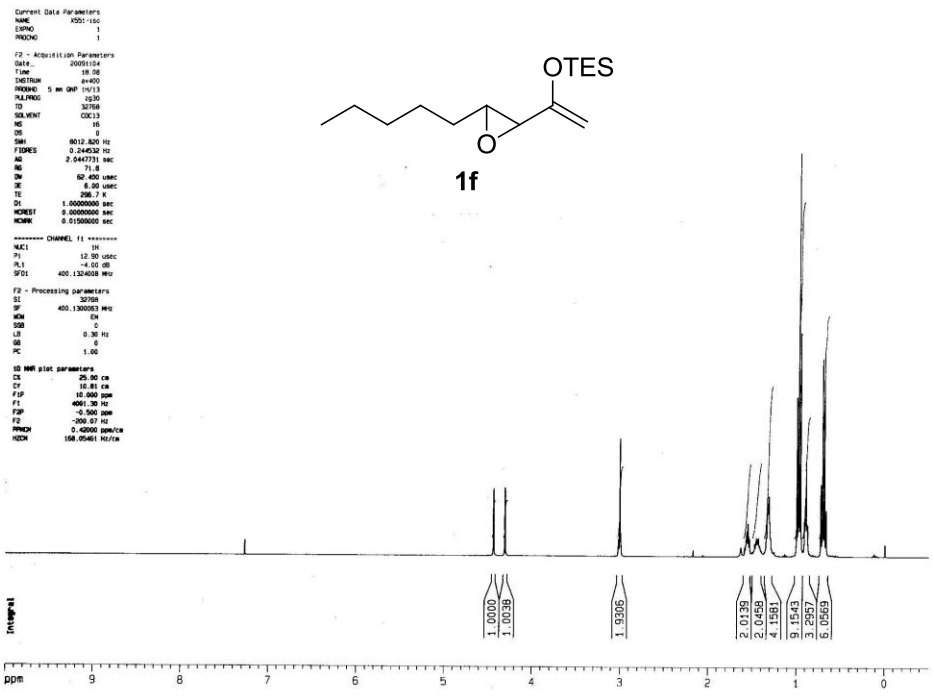

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140
120
100
80
60
40
20
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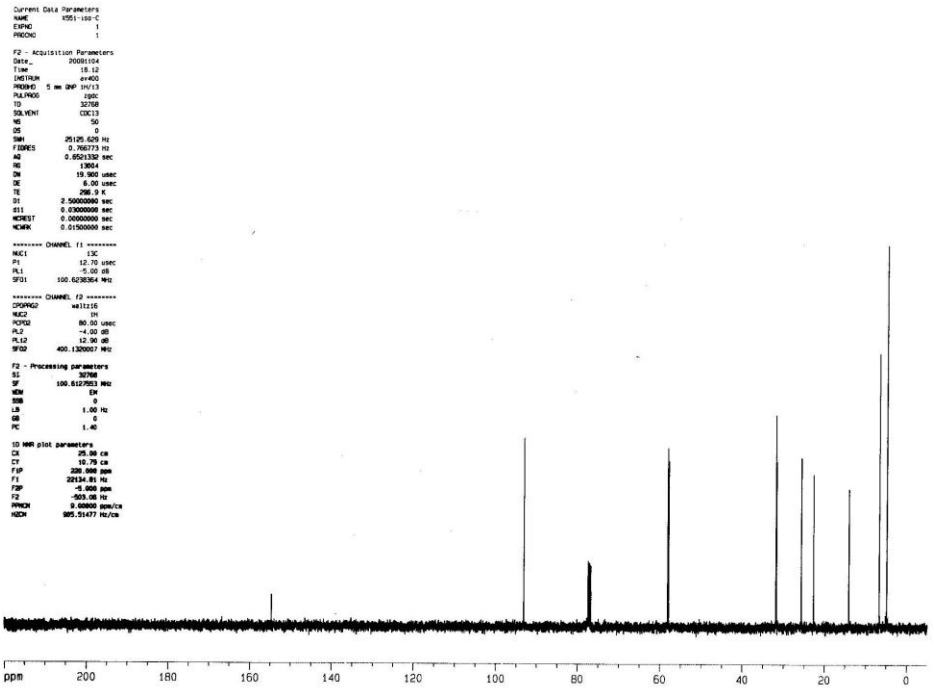

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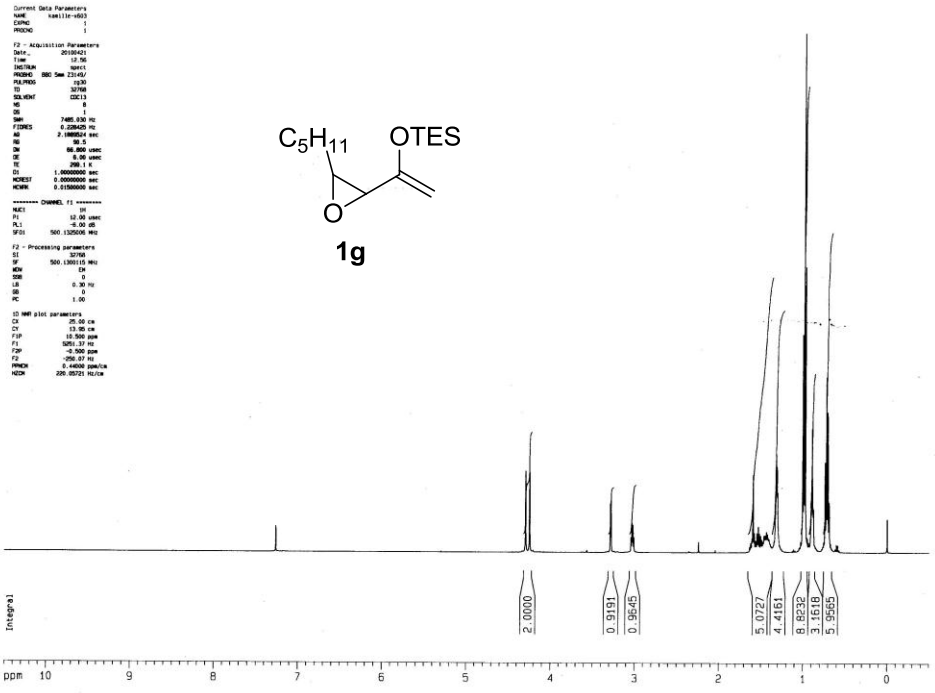

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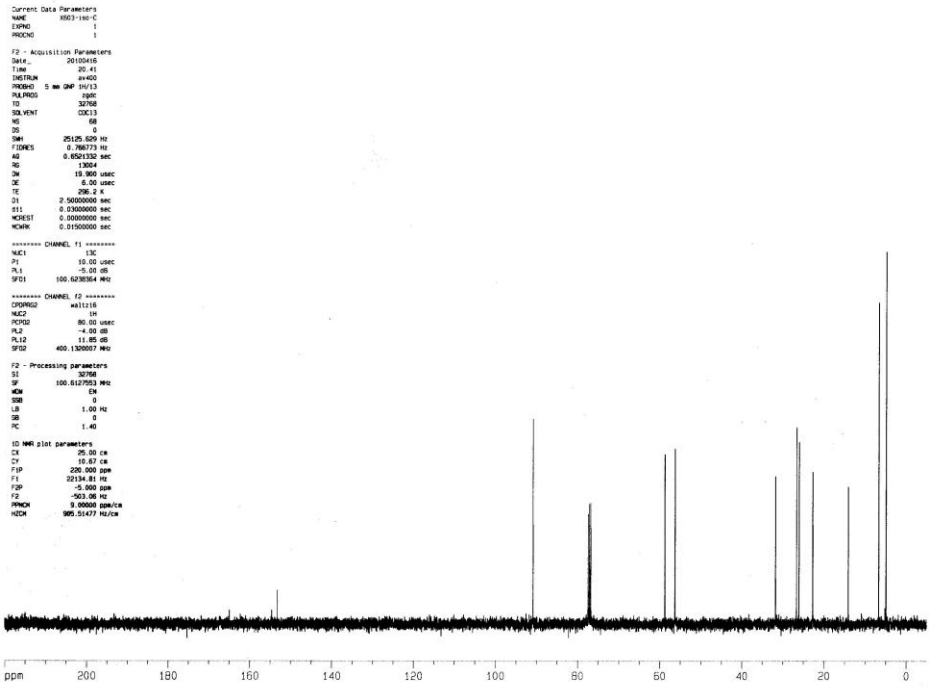

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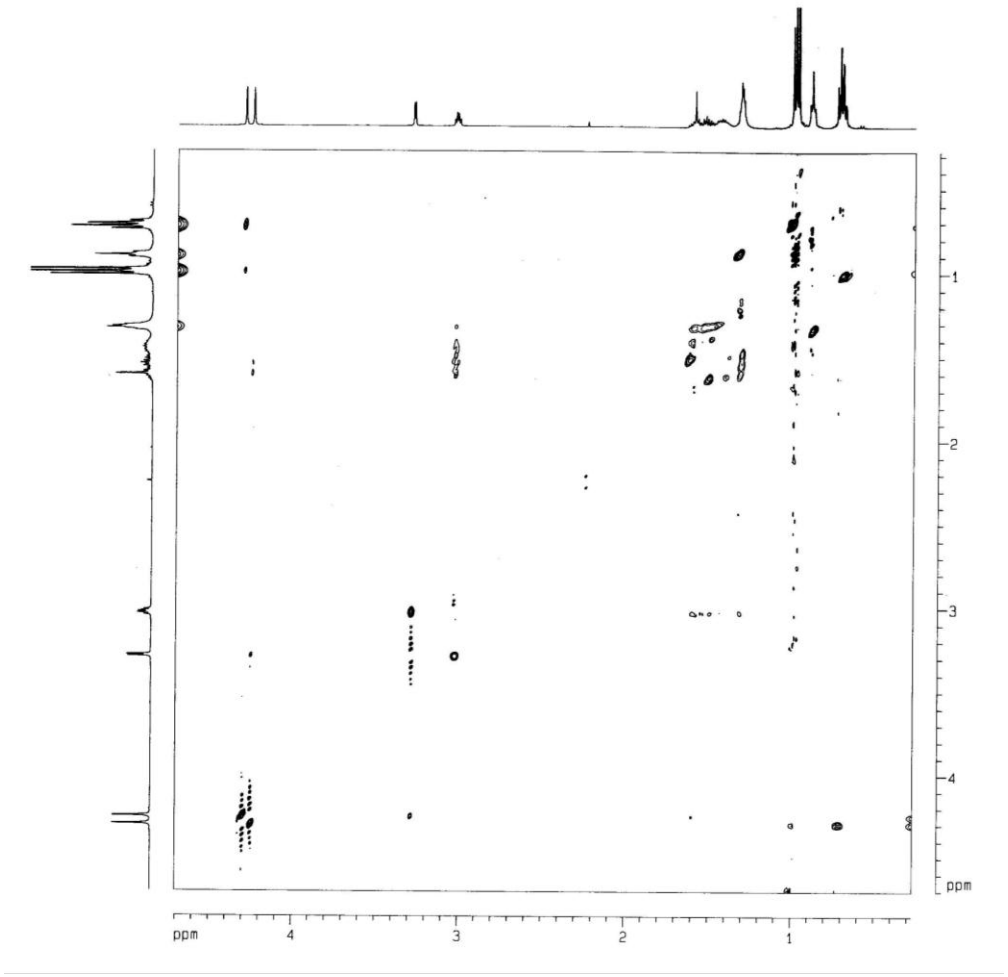

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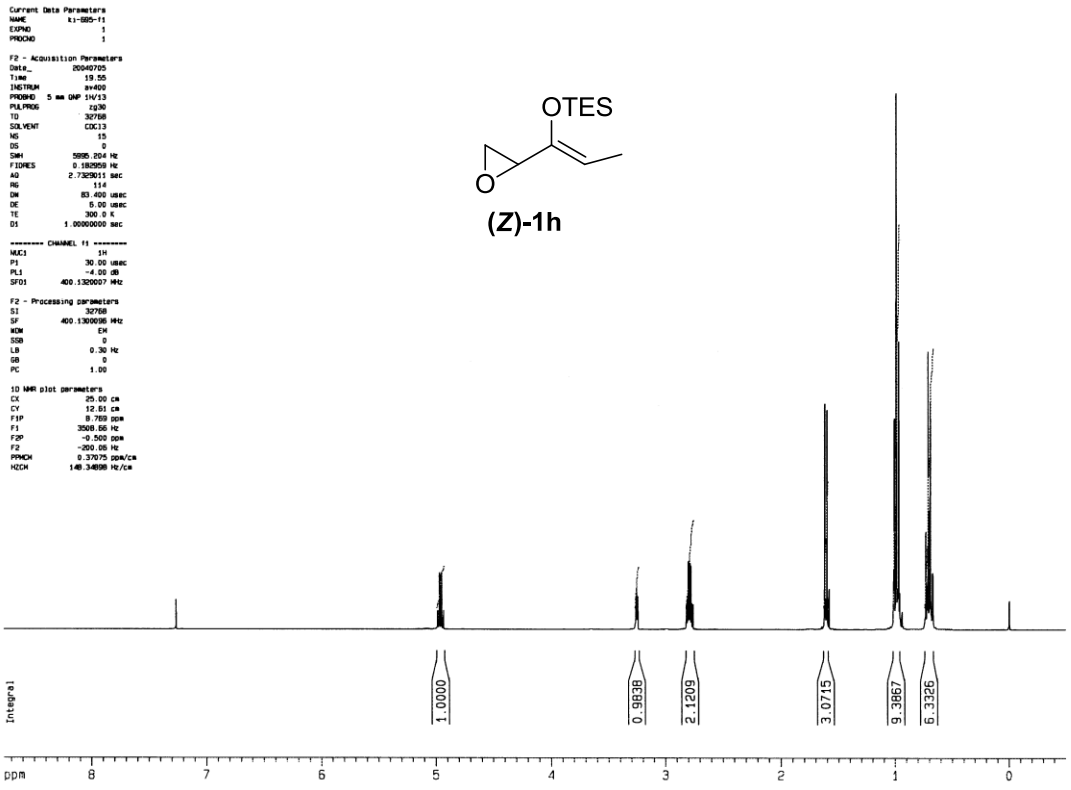

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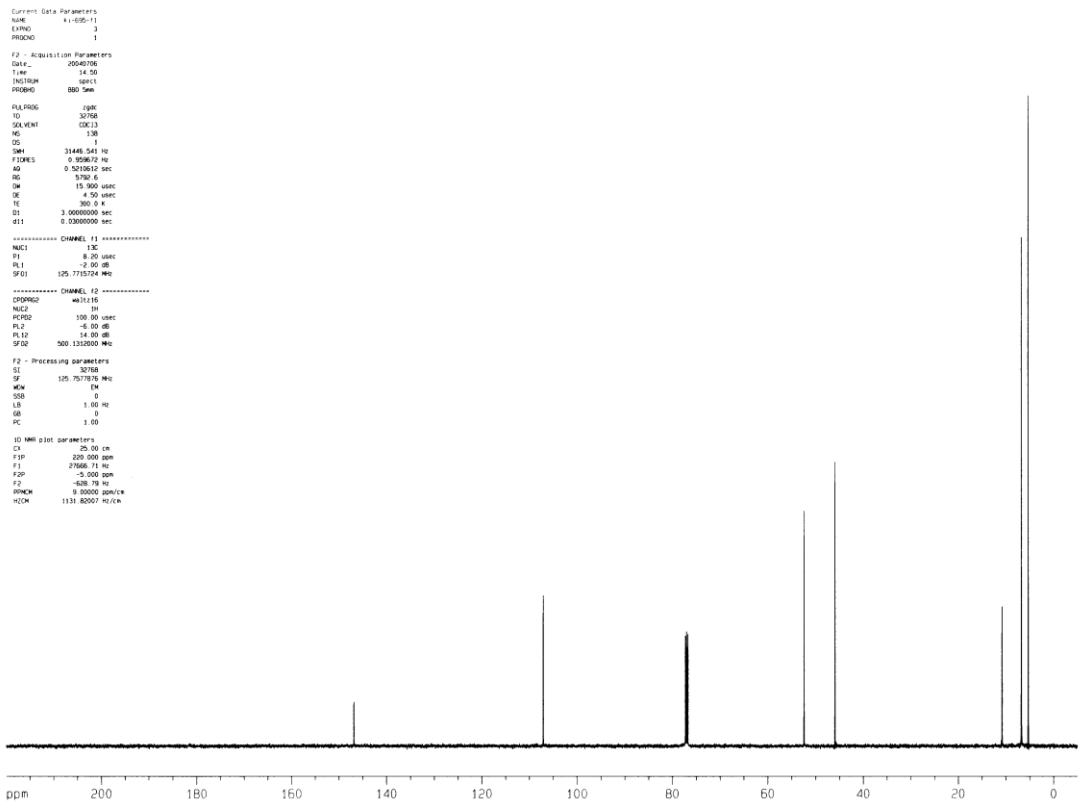

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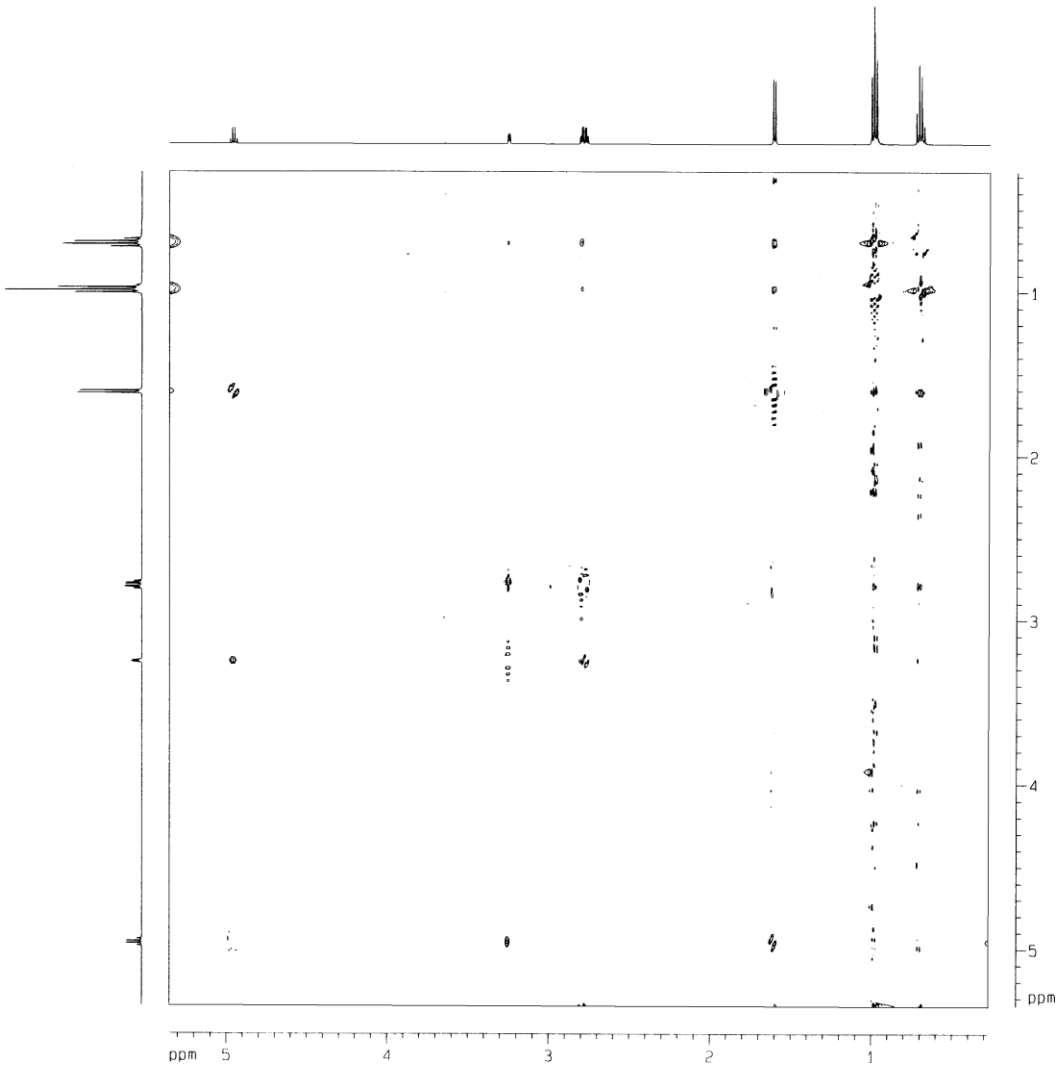

C_5H_{11}
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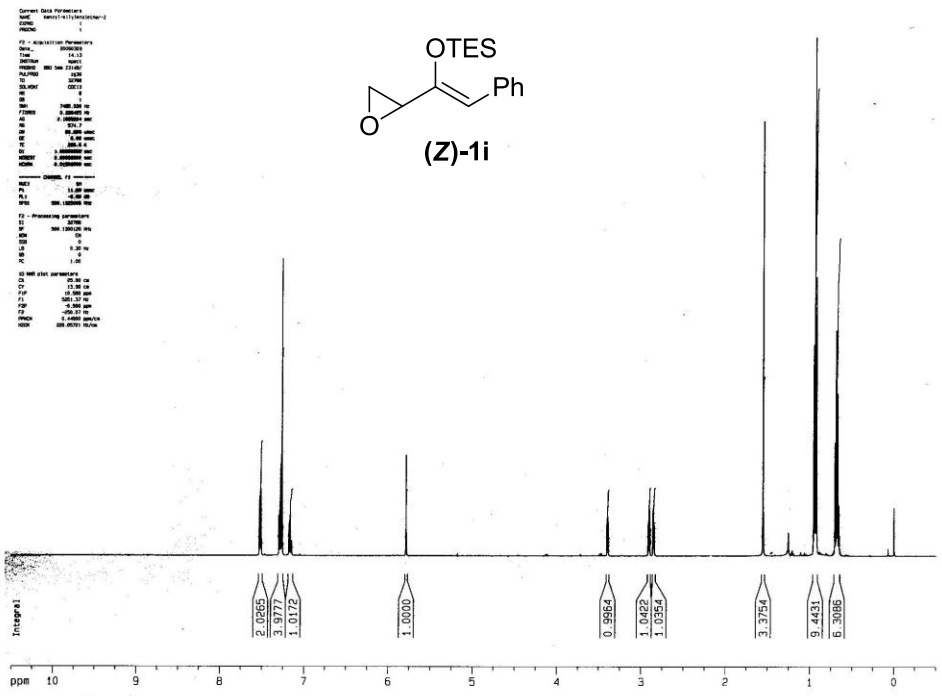

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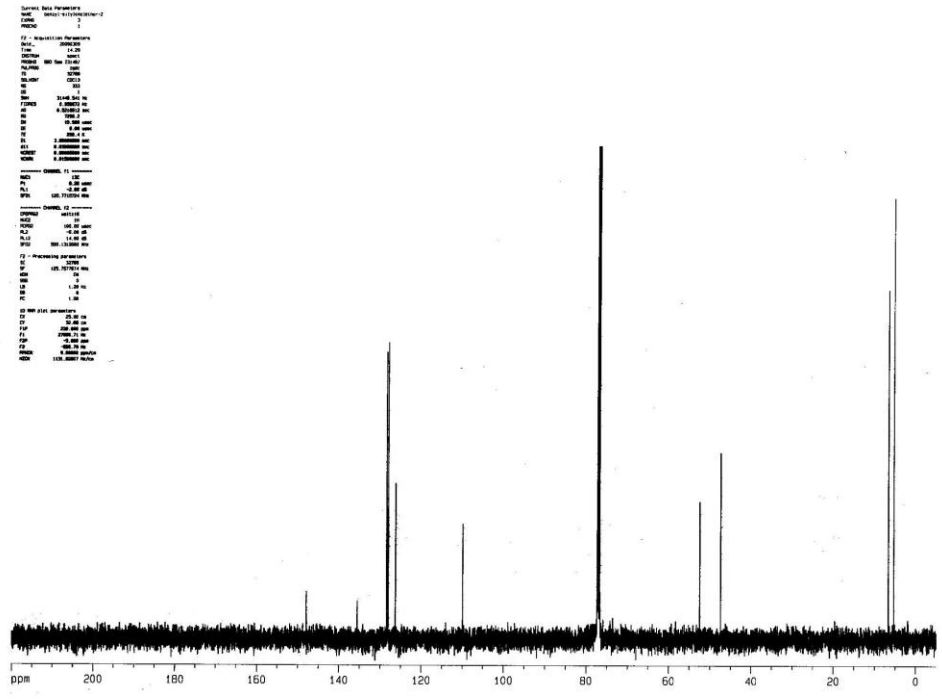

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ppm
1
2
3
4

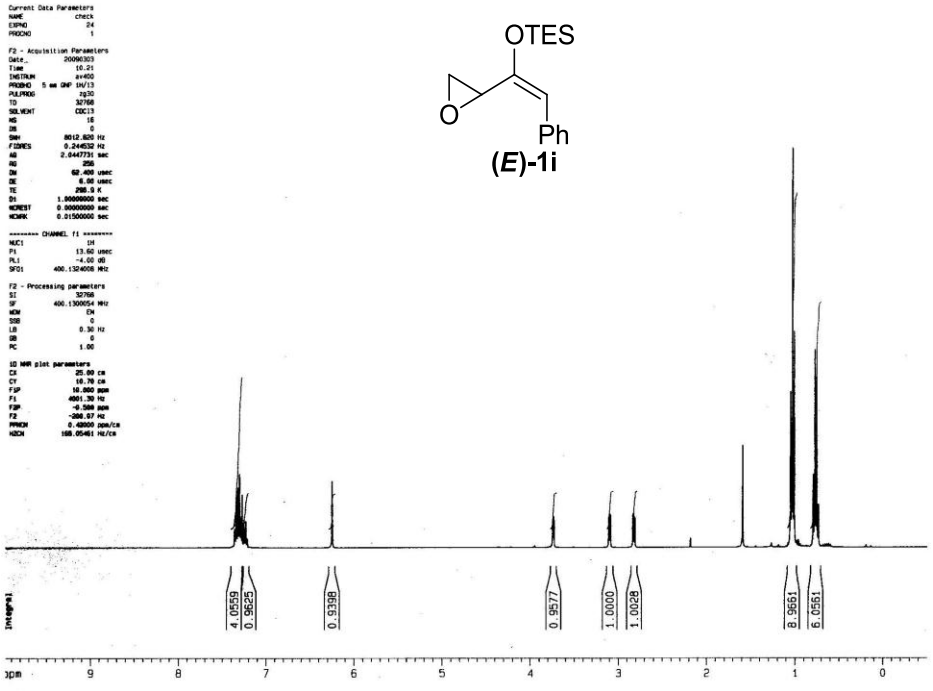

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PULPROG zg30
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SOLVENT CDCl3
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FIDRES 0.182959 Hz
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RG 114
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TE 300.0 K
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F2 - Processing parameters
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SSB 0
LB 0.30 Hz
GB 0
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F2P -0.500 ppm
F2 -200.08 Hz
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3
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1
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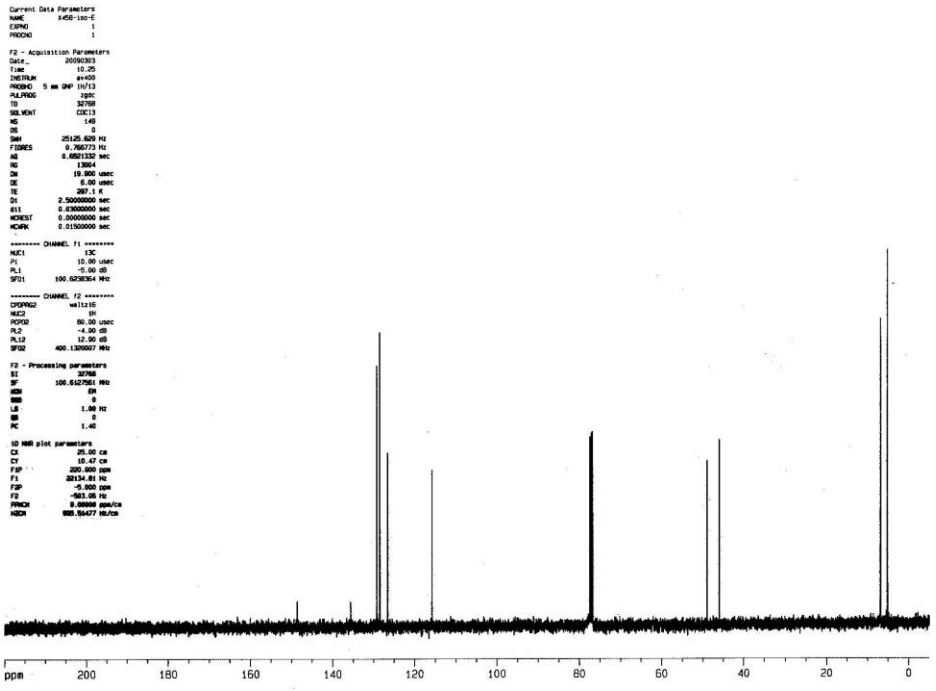

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PULPROG zgdc
TD 32768
SOLVENT CDCl3
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FIDRES 0.959672 Hz
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RG 5792.6
DW 15.900 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec
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PL1 -2.00 dB
SFO1 125.7715724 MHz
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CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
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PL12 14.00 dB
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LB 1.00 Hz
GB 0
PC 1.00
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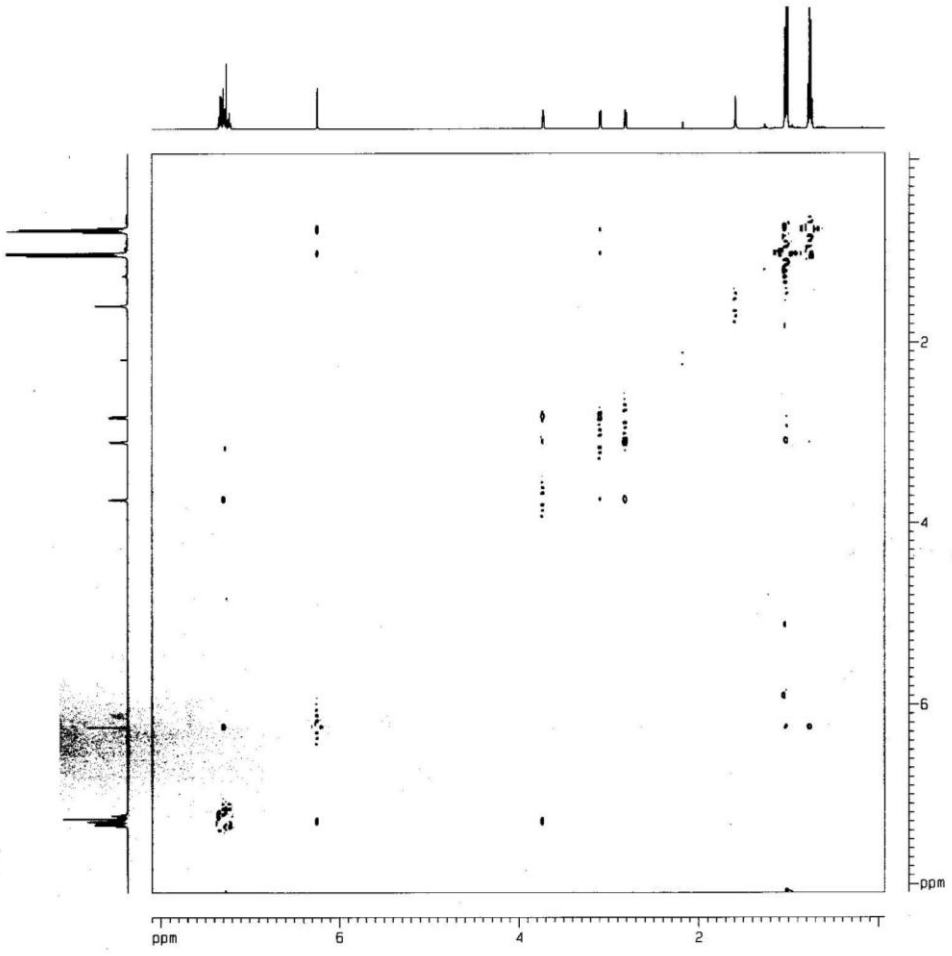

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ppm
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2
3
4
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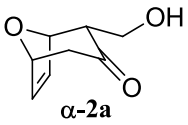

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6.3086
ppm 10 9 8 7 6 5 4 3 2 1 0


ppm 200 180 160 140 120 100 80 60 40 20 0


OTES
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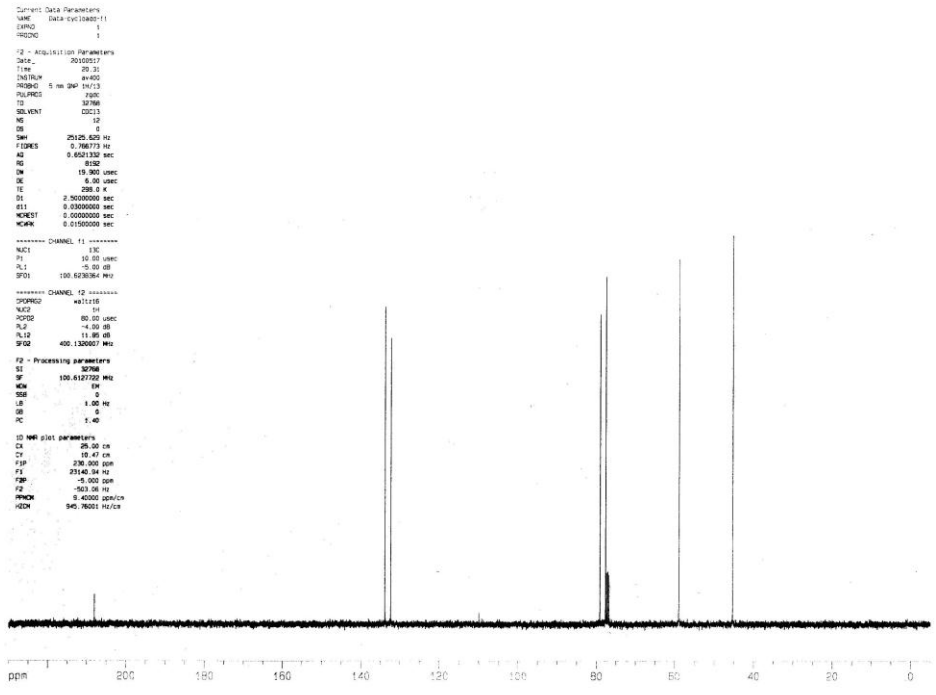

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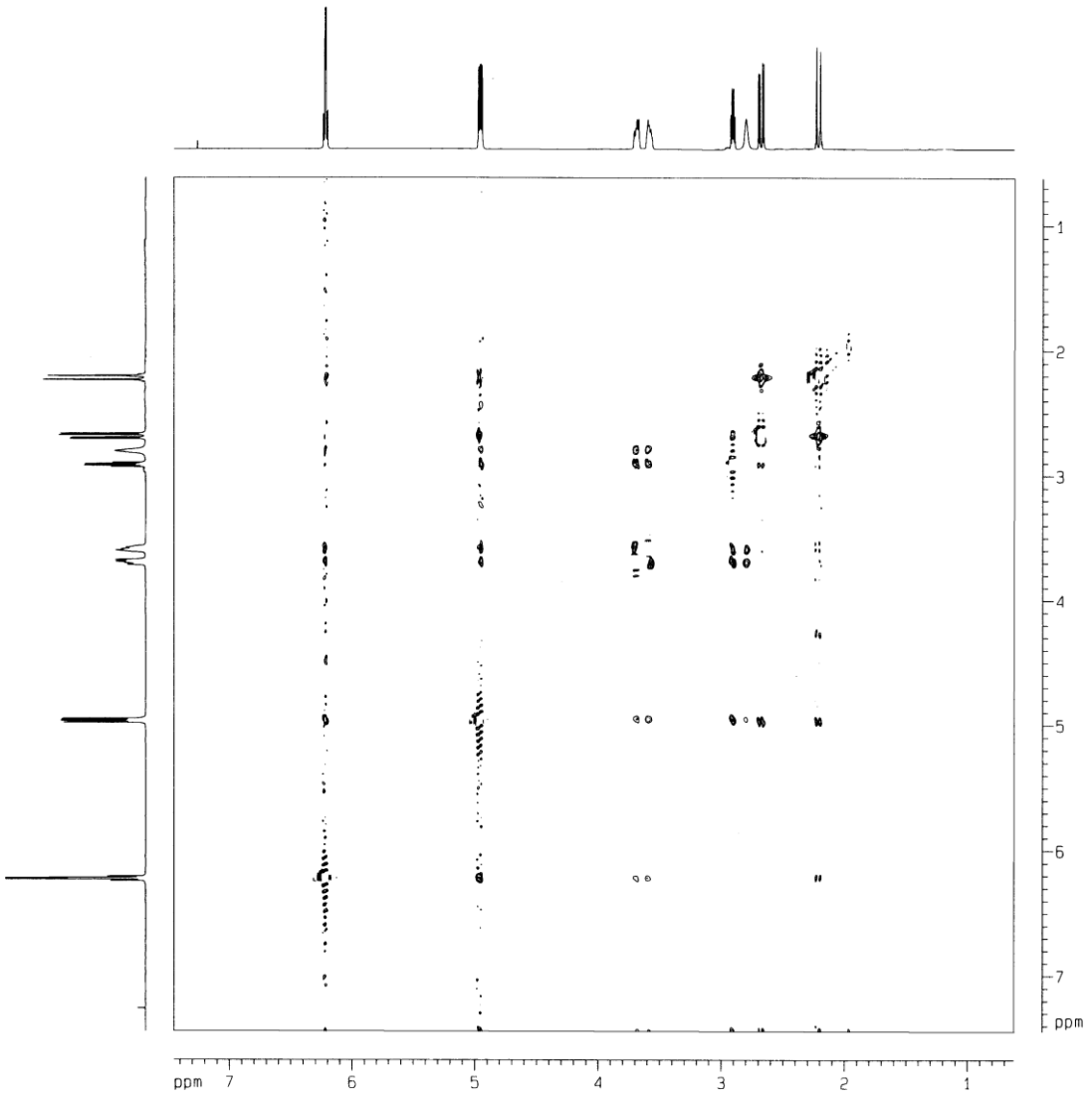

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ppm
ppm
6
4
2

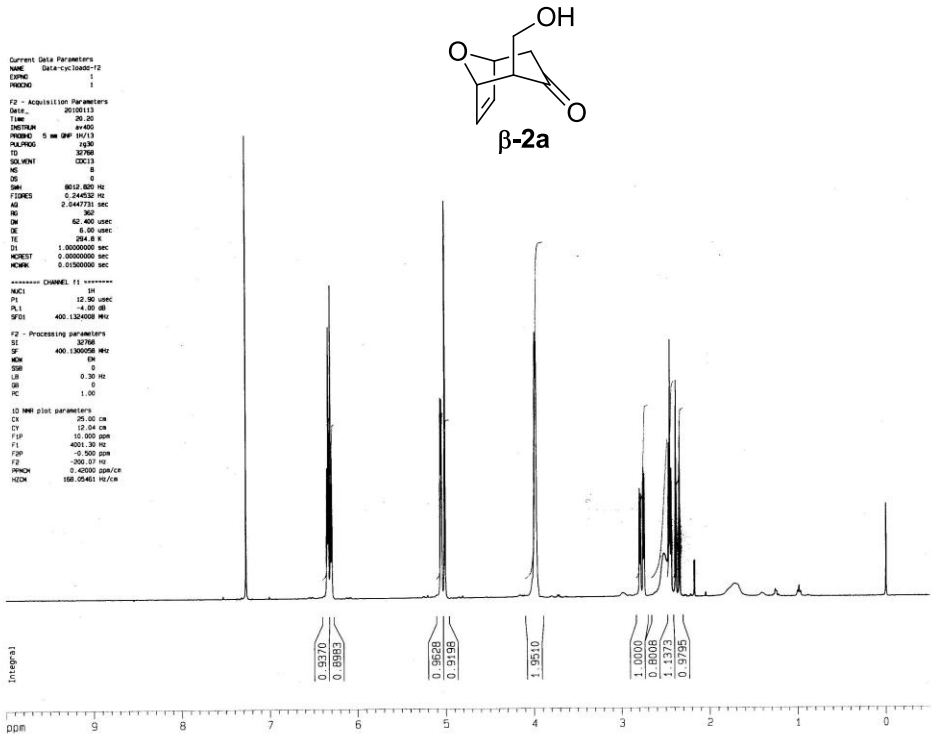

O
OH
O

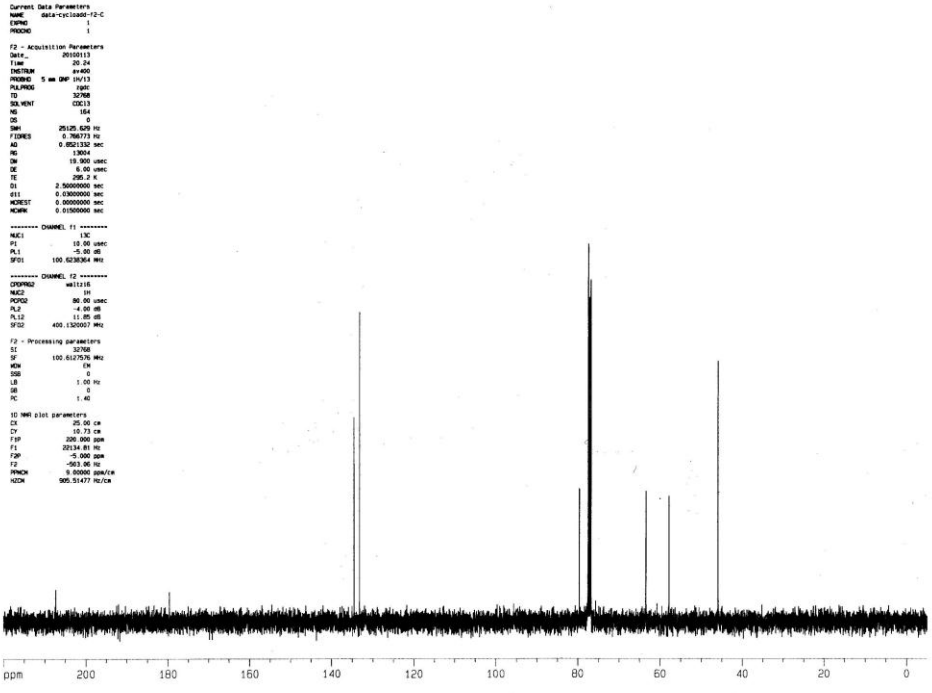
α-2a

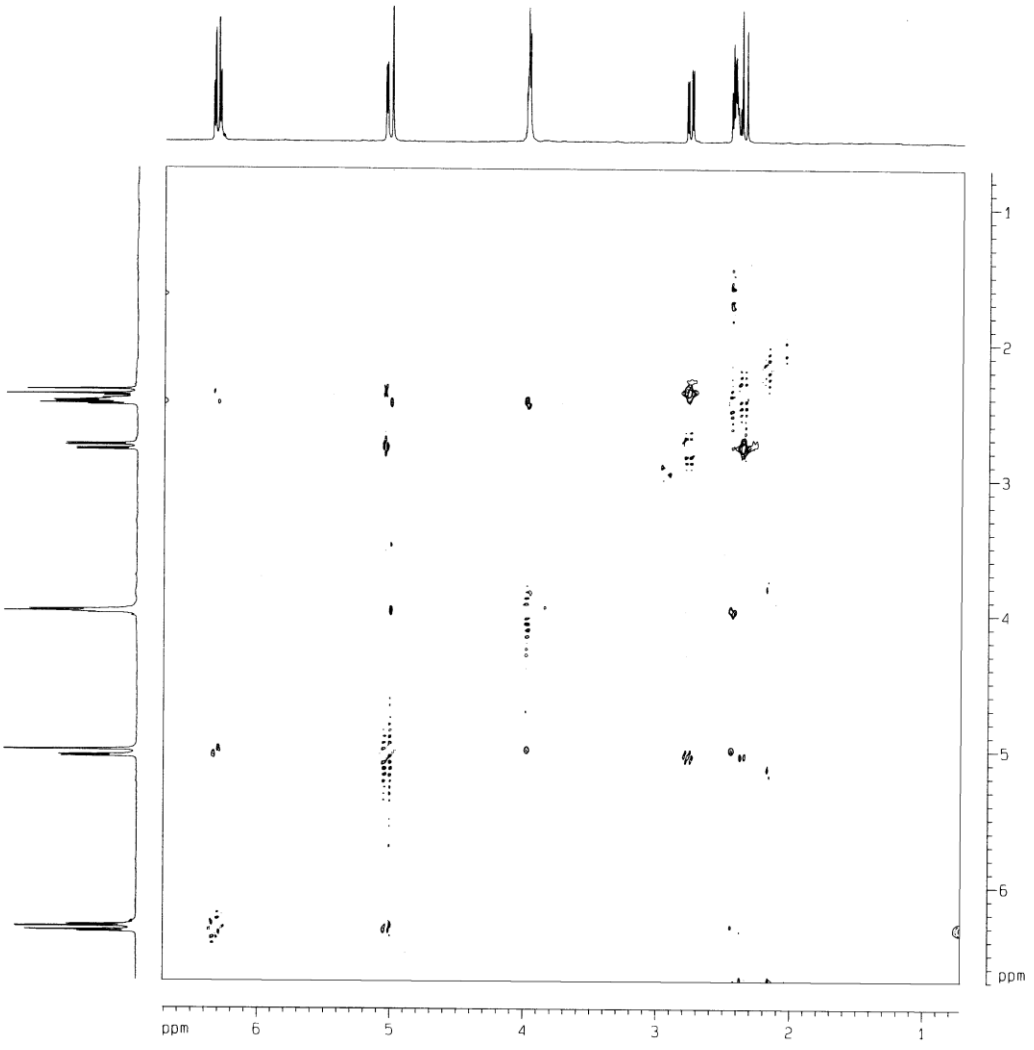
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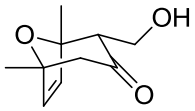

ppm


ppm
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2
1
ppm
1
2
3
4
5
6
7

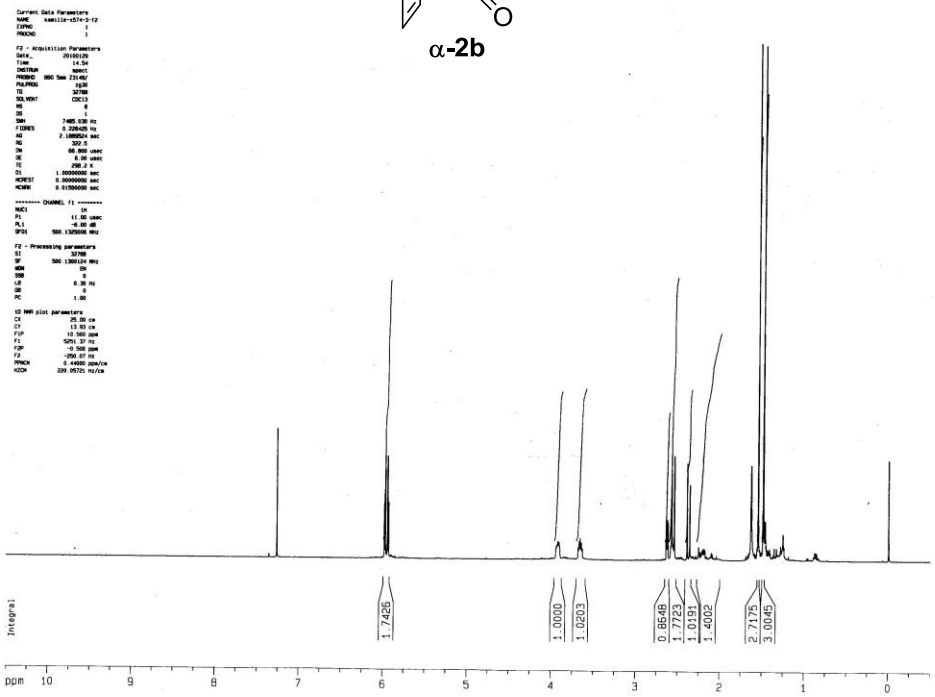

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O
β-2a
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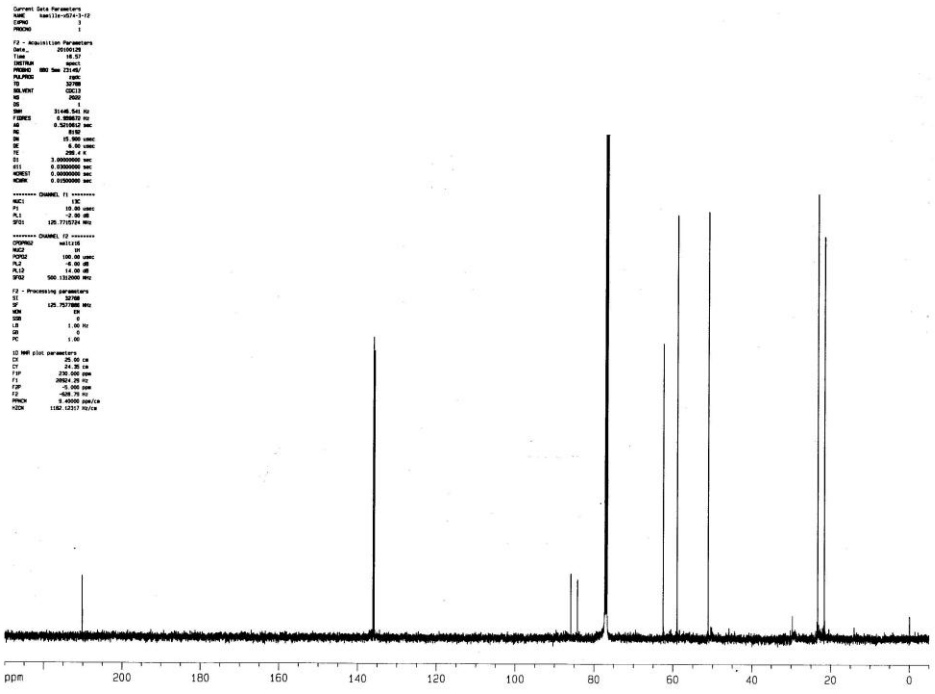

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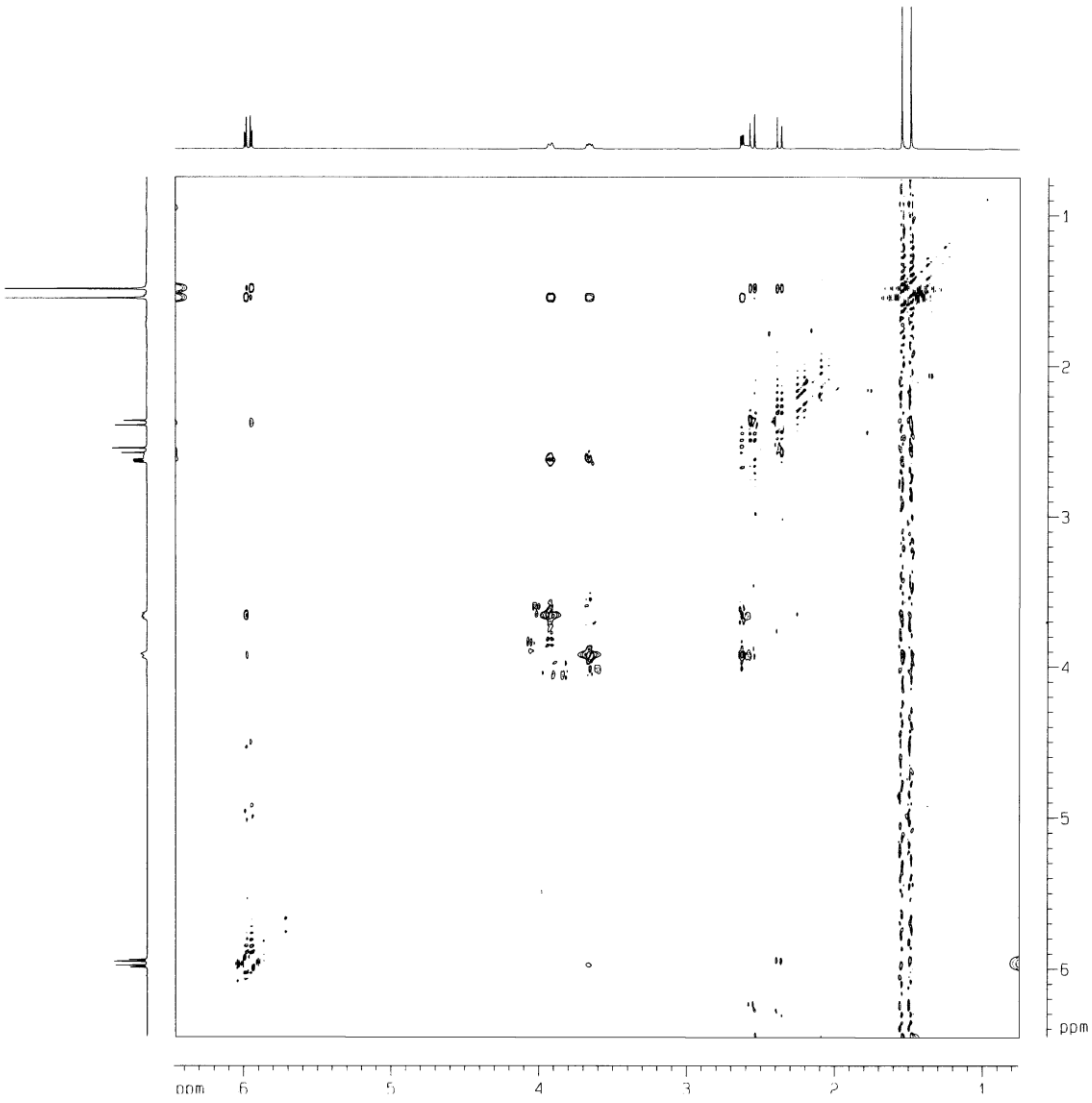

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ppm
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4
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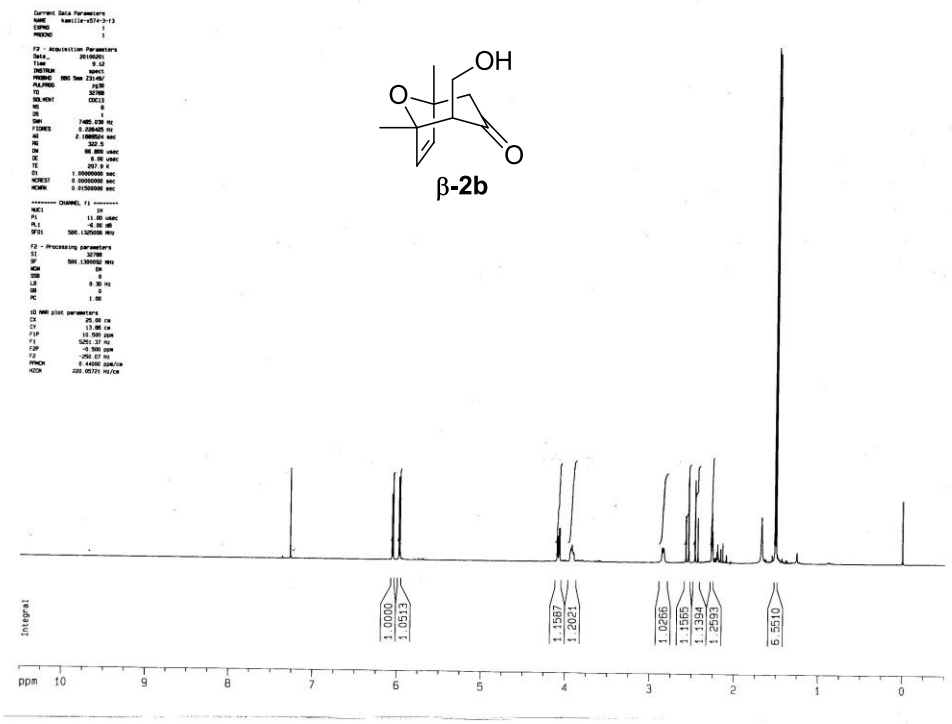

OH
O
O

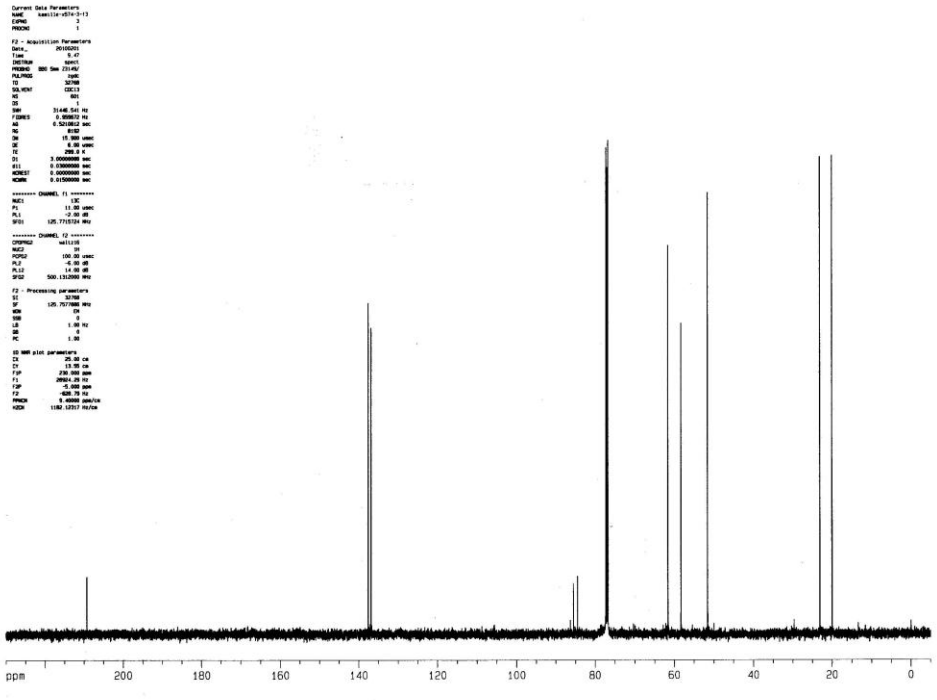
α-**2b**

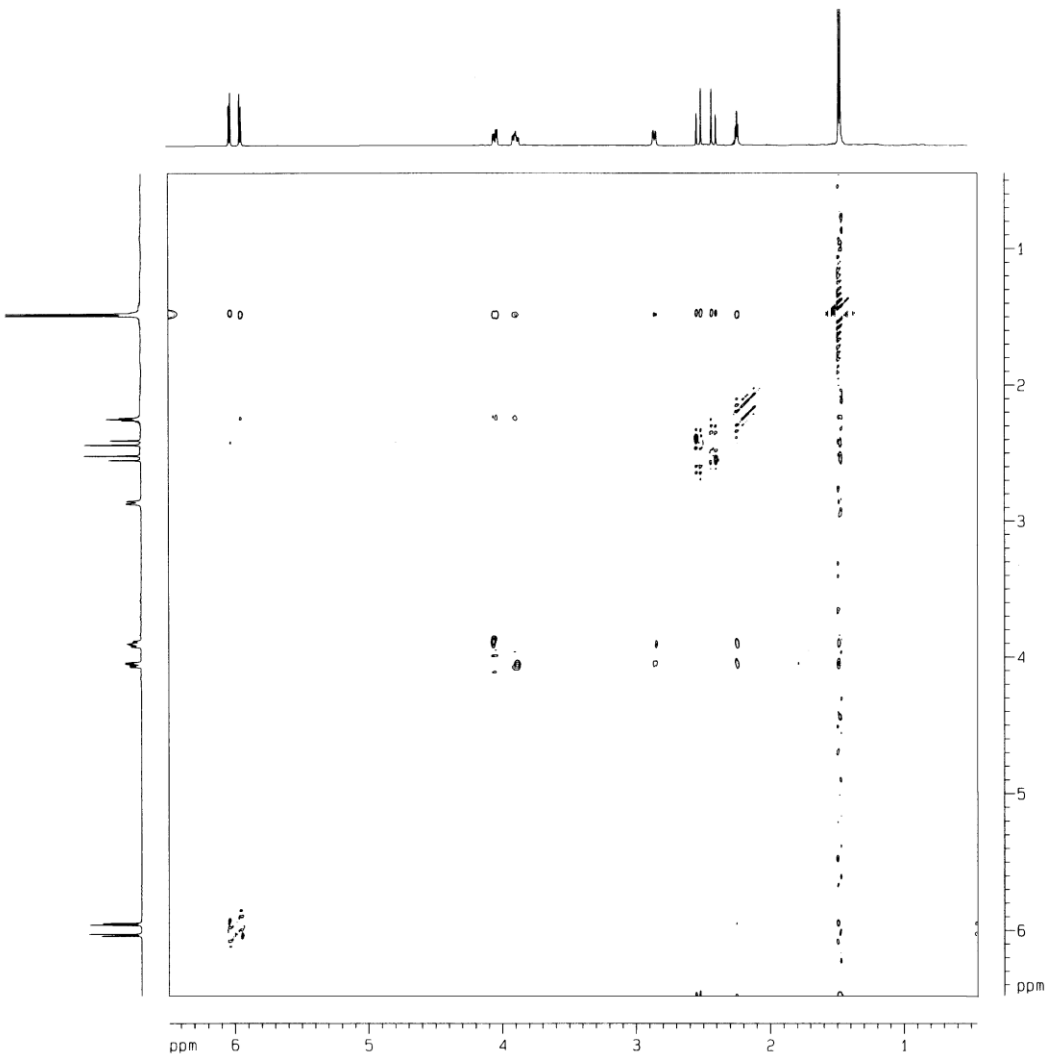

Integral
1.7426
1.0000
1.0203
0.8648
1.7723
1.0191
1.4002
2.7175
3.0045
ppm
10
9
8
7
6
5
4
3
2
1
0

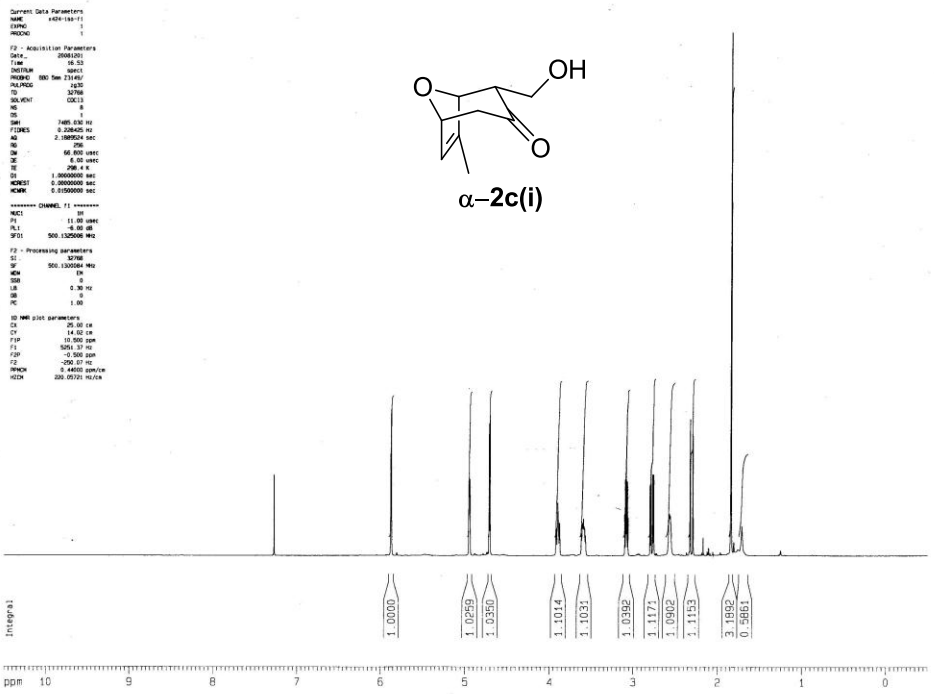

ppm
200
180
160
140
120
100
80
60
40
20
0

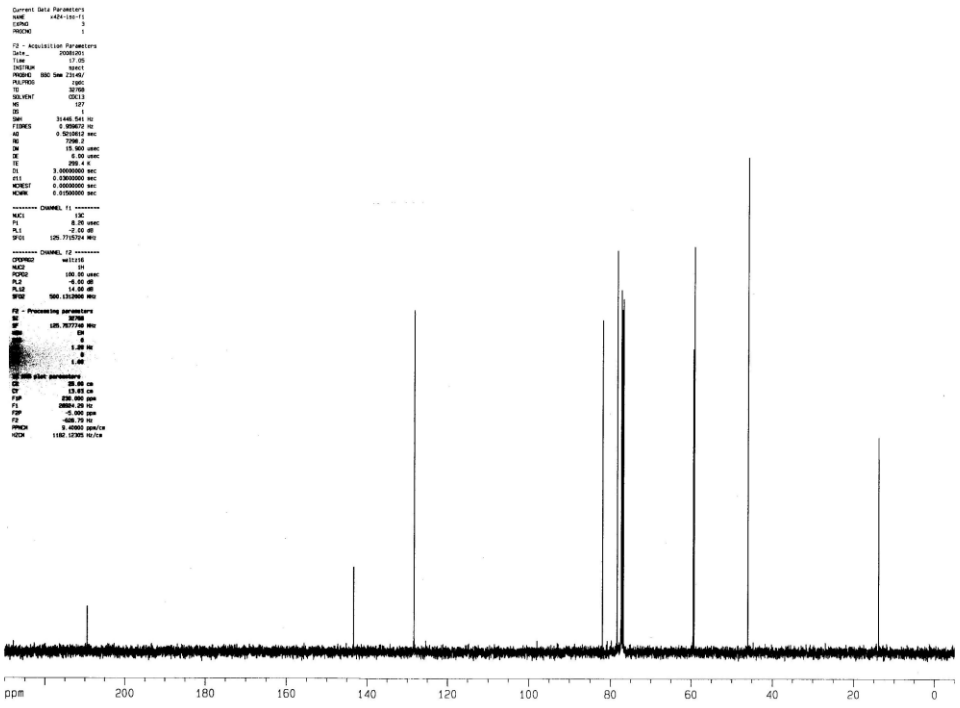

ppm
6
5
4
3
2
1
ppm

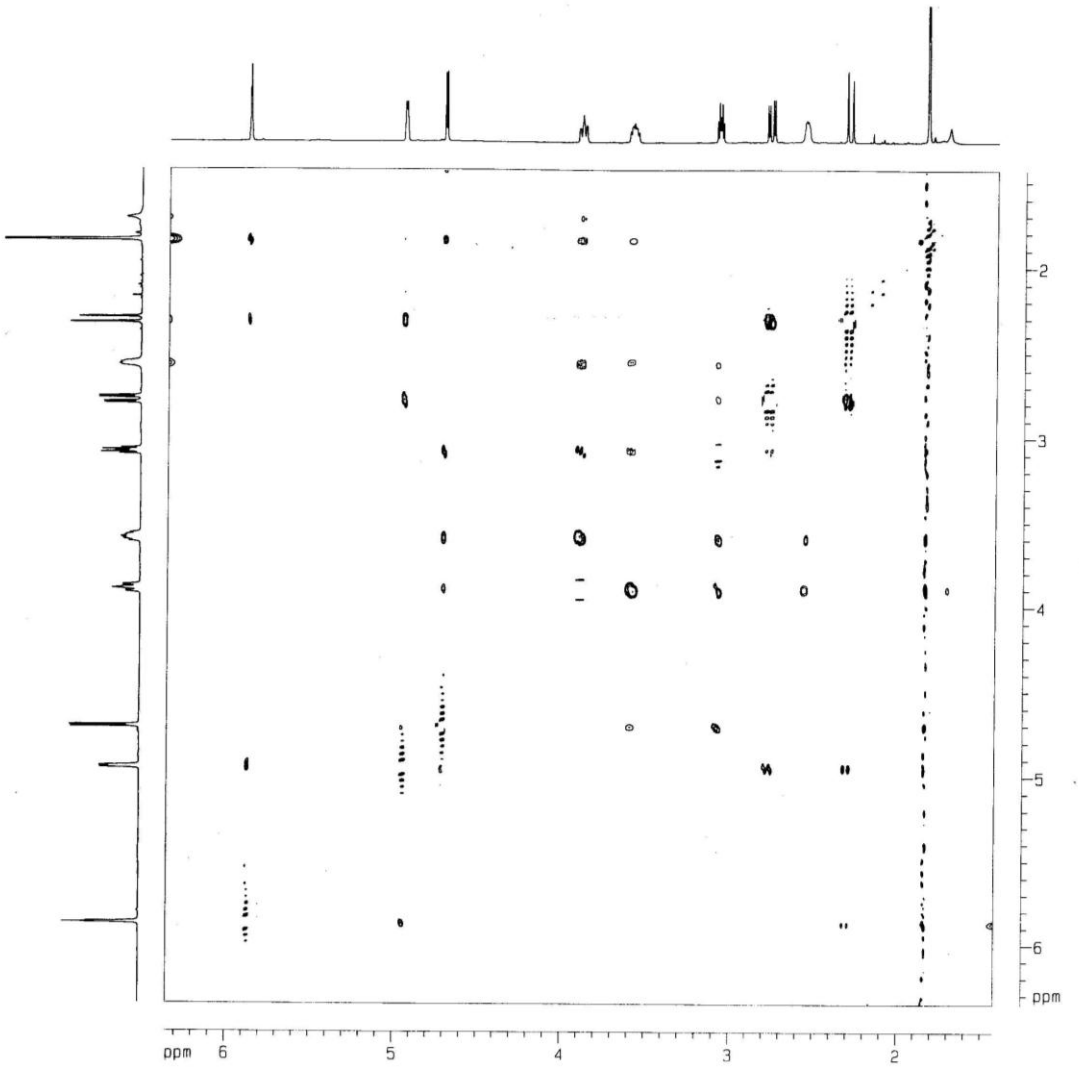

OH
O
O
β-2b
Integral
1.0000
1.0513
1.1567
1.2021
1.0266
1.1565
1.1394
1.2593
6.5510
ppm

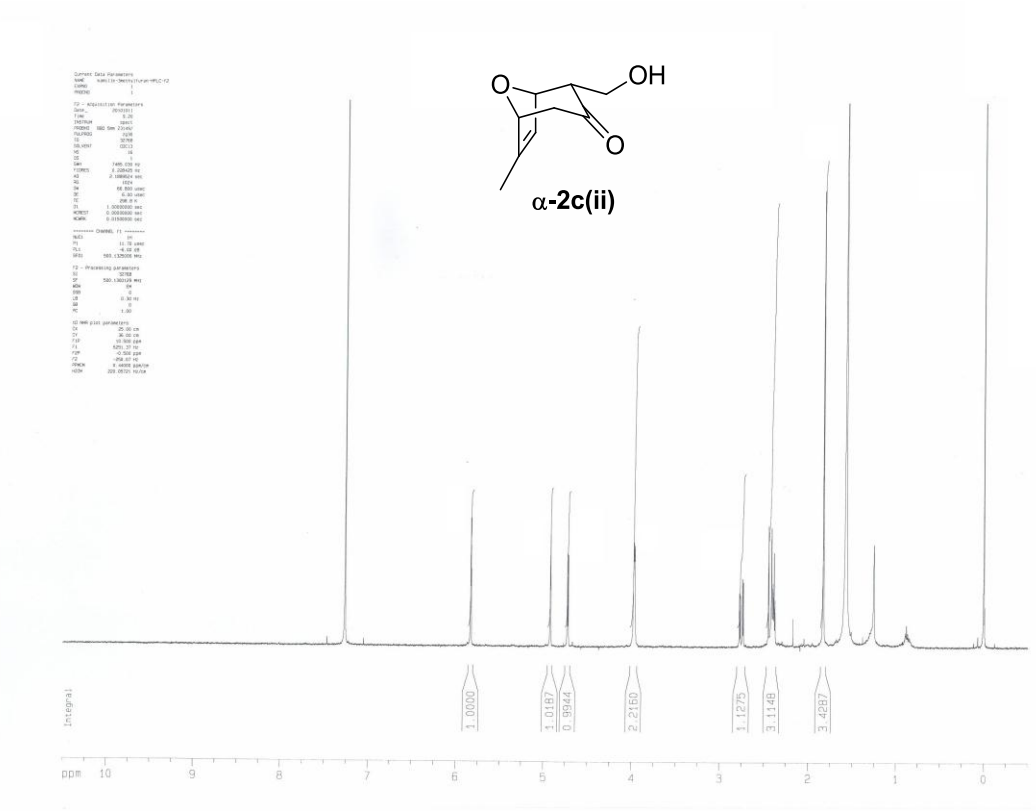

ppm

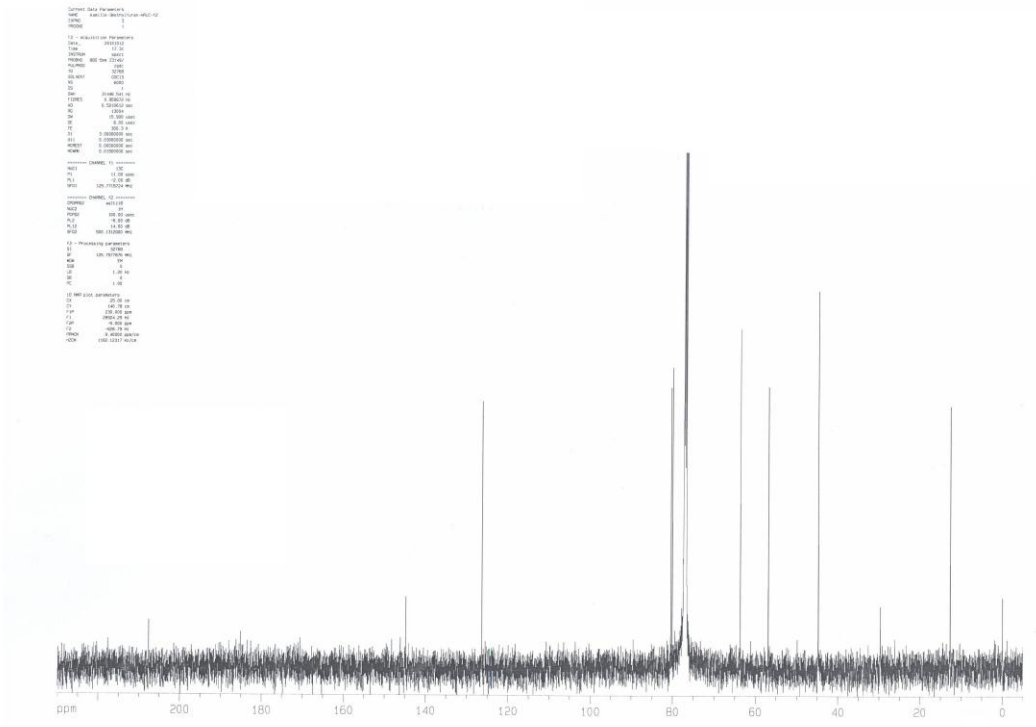

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

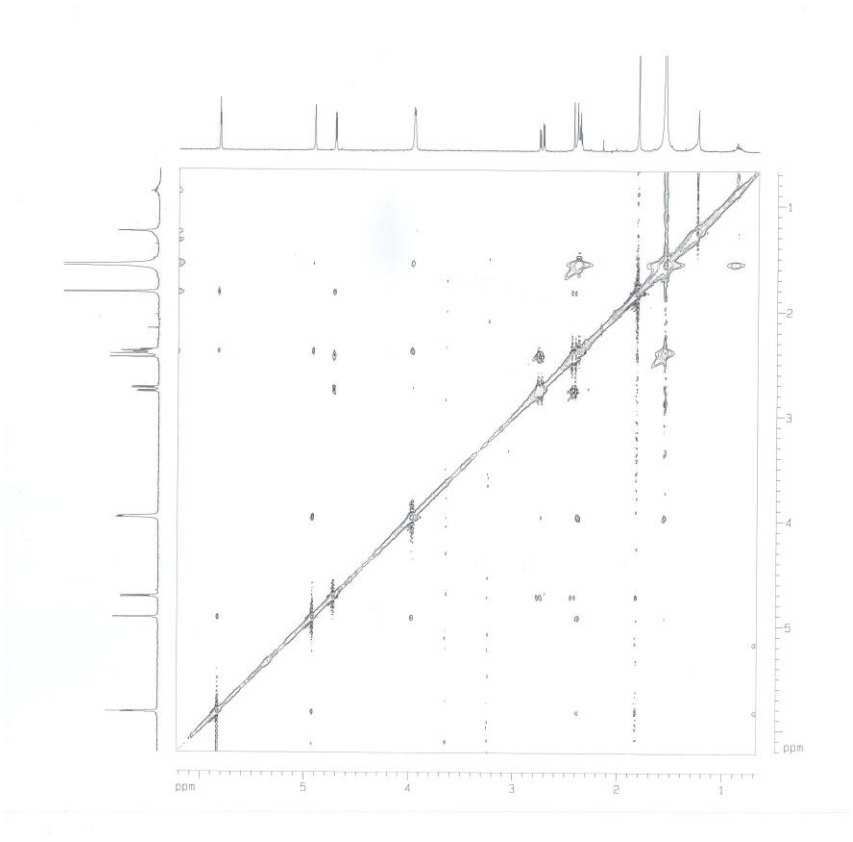

OH
O
O
α–2c(i)
Integral
1.0000
1.0259
1.0350
1.1014
1.1031
1.0392
1.1171
1.0902
1.1153
3.1892
0.5861
ppm 10 9 8 7 6 5 4 3 2 1 0

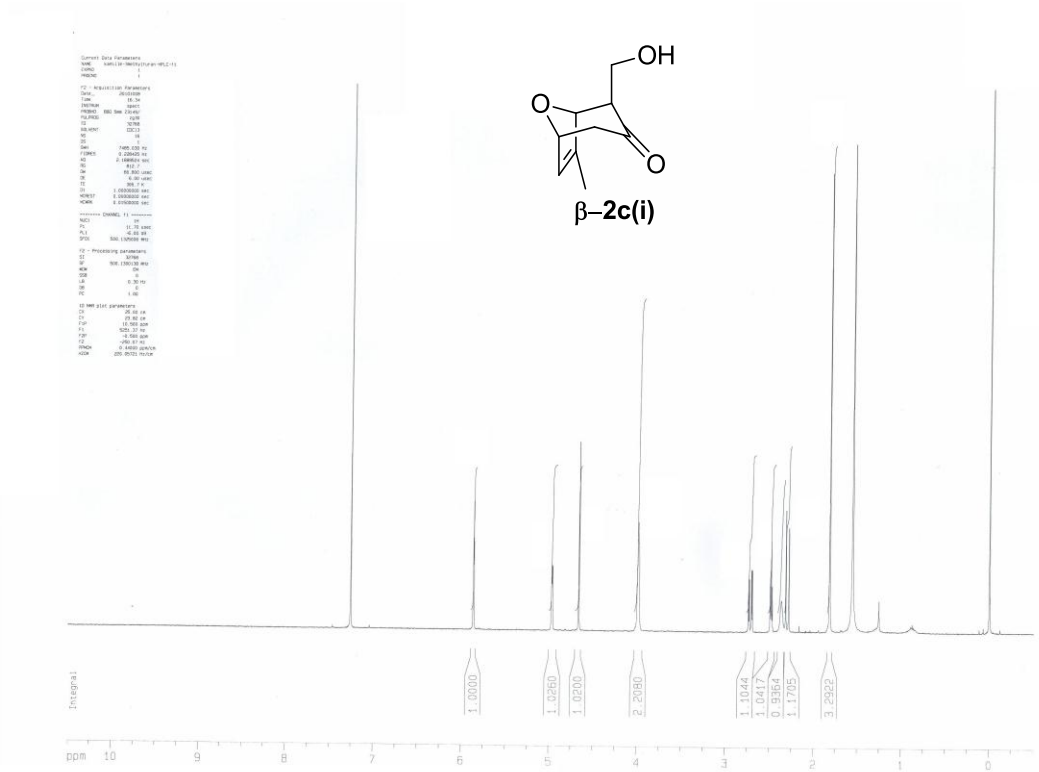

ppm 200 180 160 140 120 100 80 60 40 20 0

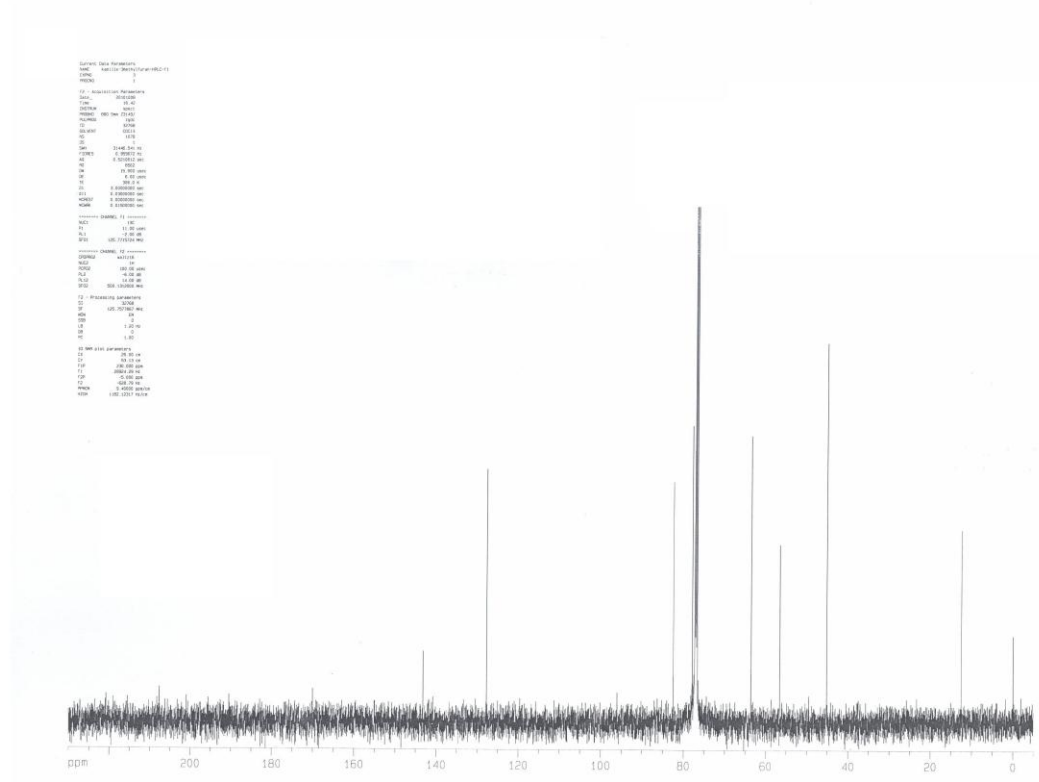

2
3
4
5
6
ppm
ppm 6
5
4
3
2

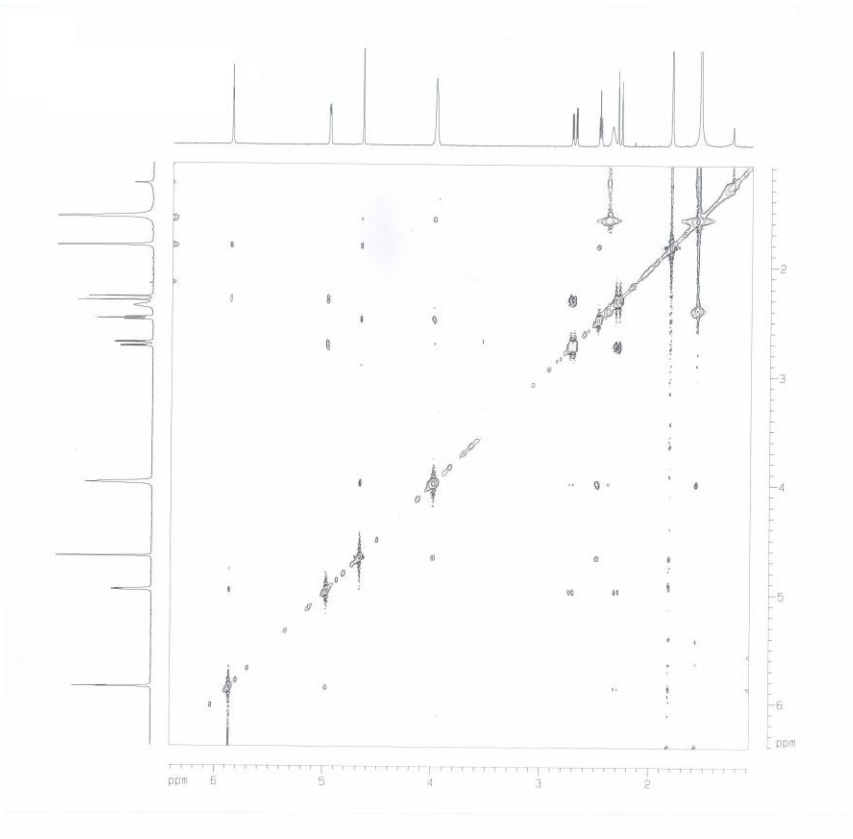

OH
O
O
α-2c(ii)
Integral
1.0000
1.0187
0.9944
2.2160
1.1275
3.1148
3.4287
ppm 10 9 8 7 6 5 4 3 2 1 0

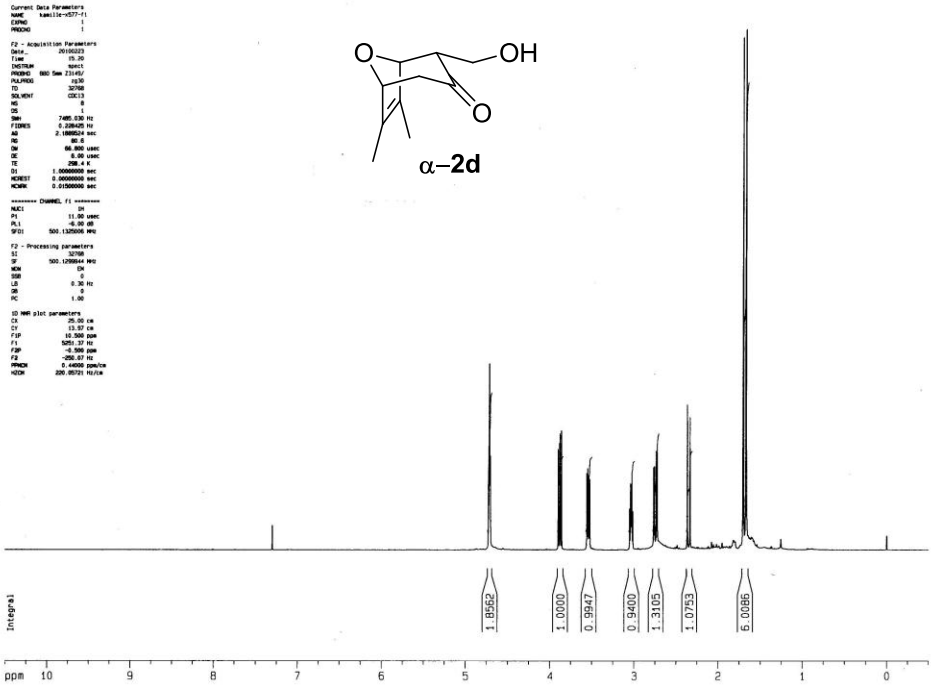

ppm 200 180 160 140 120 100 80 60 40 20 0

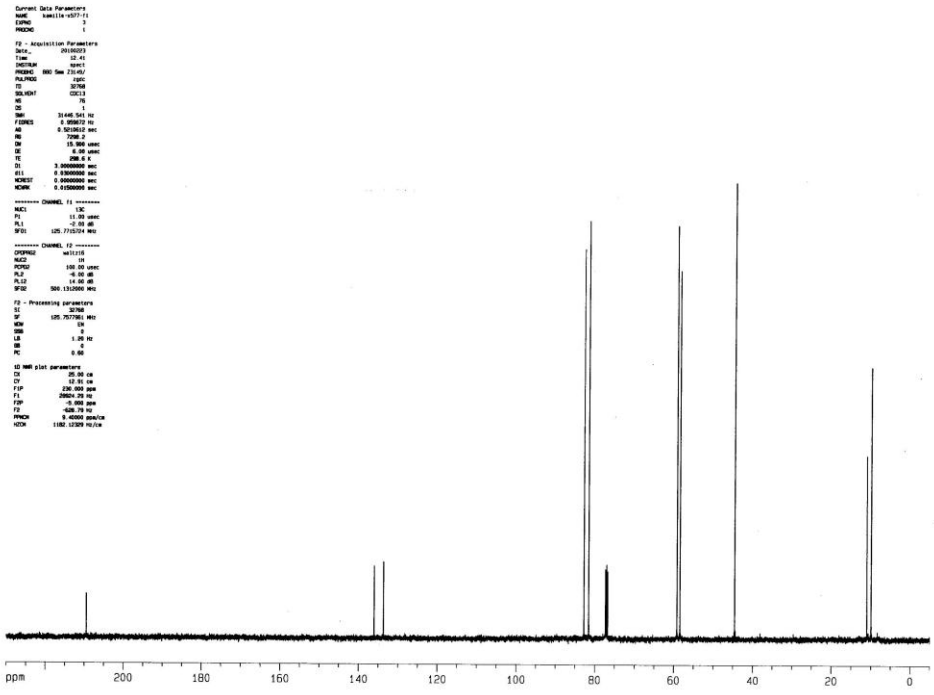

ppm
5
4
3
2
1
ppm
1
2
3
4
5

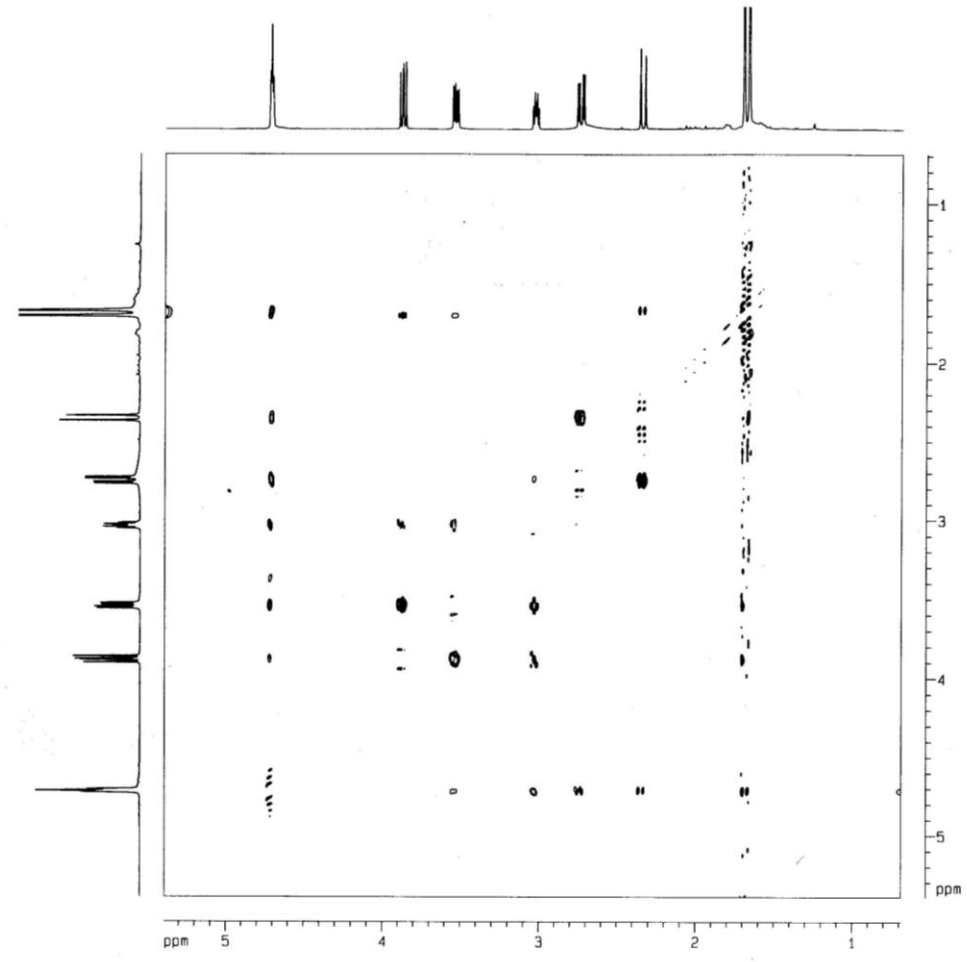

OH
O
O
β–2c(i)
Integral
1.0000
1.0260
1.0200
2.2080
1.1044
1.0417
0.9364
1.1705
3.2922
ppm 10 9 8 7 6 5 4 3 2 1 0

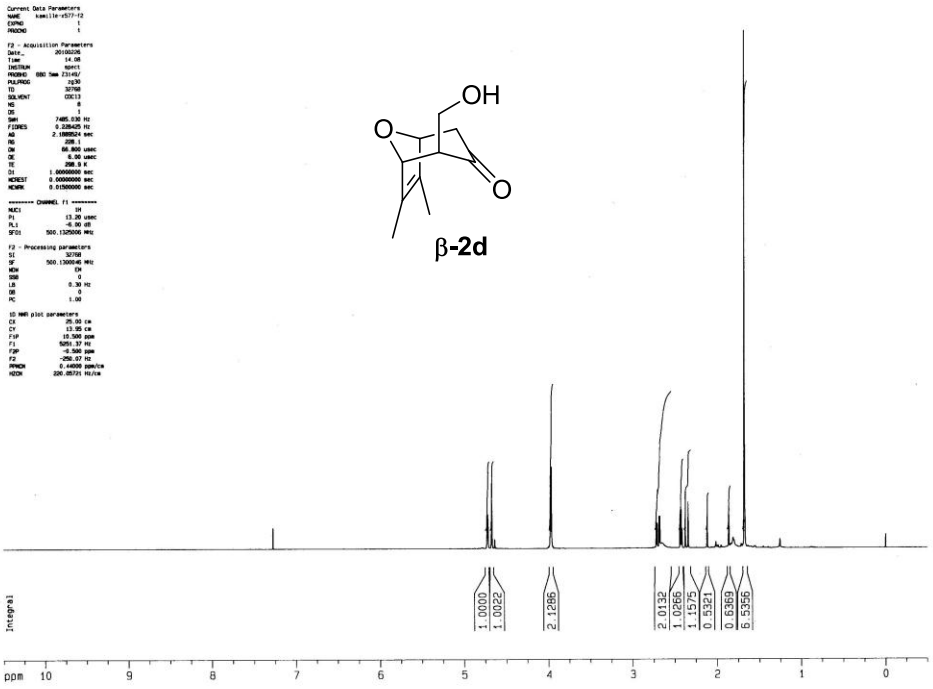

ppm 200 180 160 140 120 100 80 60 40 20 0

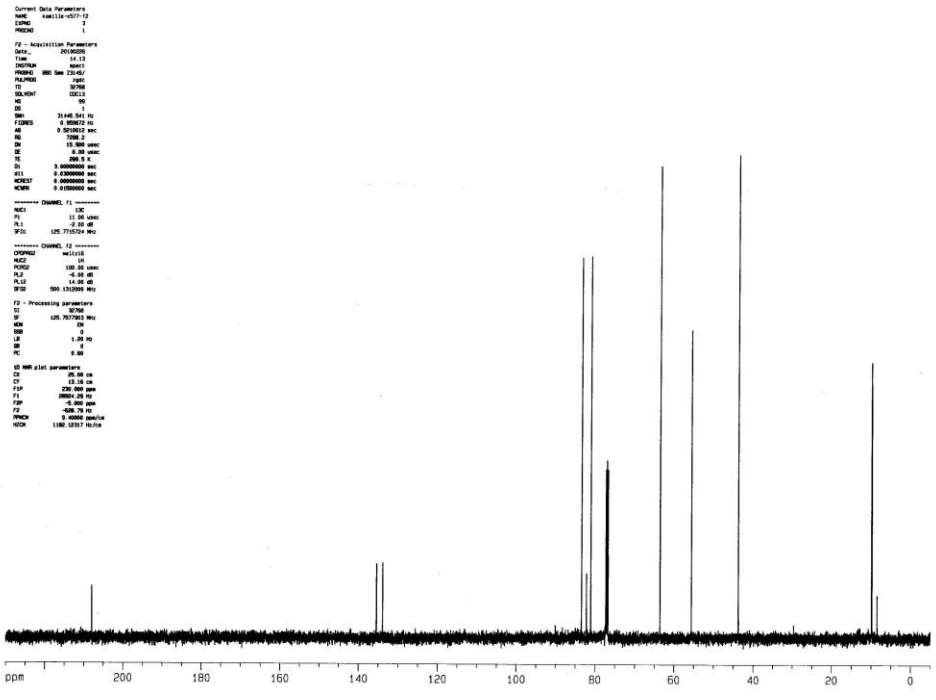

ppm
6
5
4
3
2
ppm

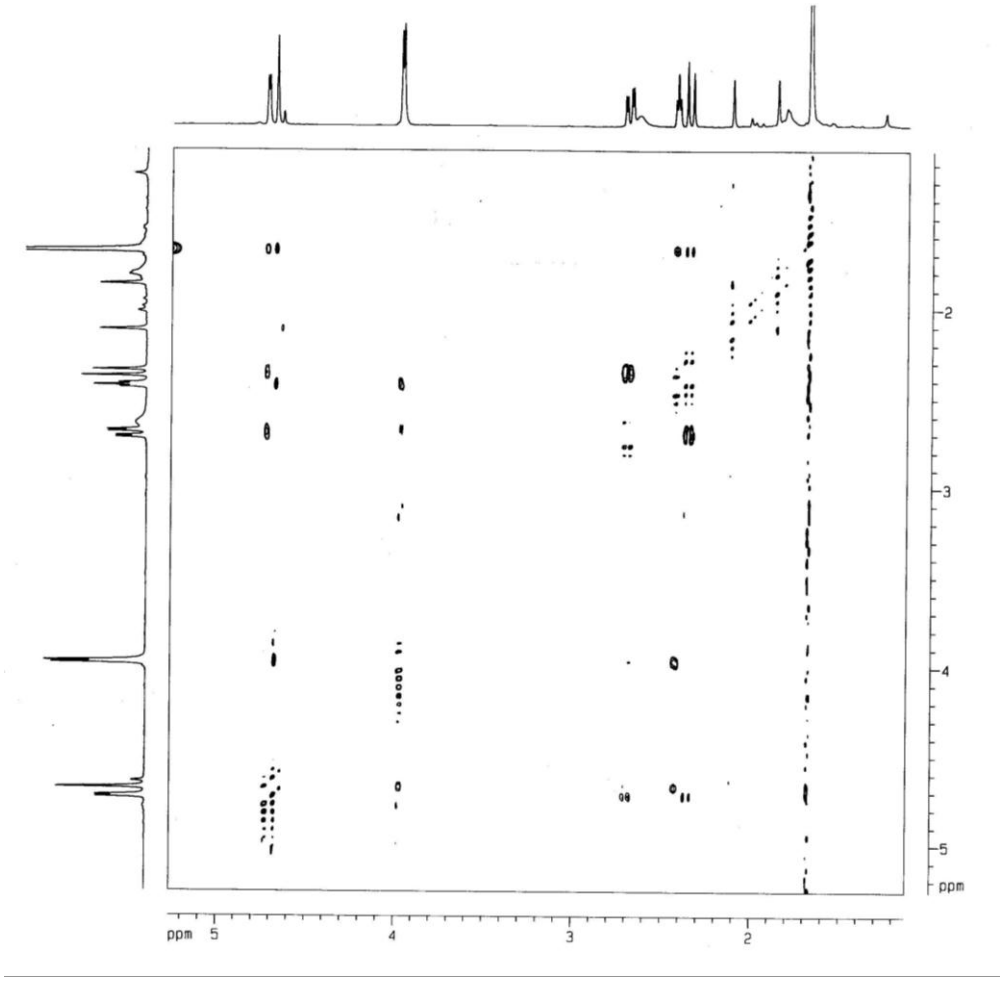

O
OH
O
α–2d
Integral
1.8562
1.0000
0.9947
0.9400
1.3105
1.0753
6.0086
ppm 10 9 8 7 6 5 4 3 2 1 0

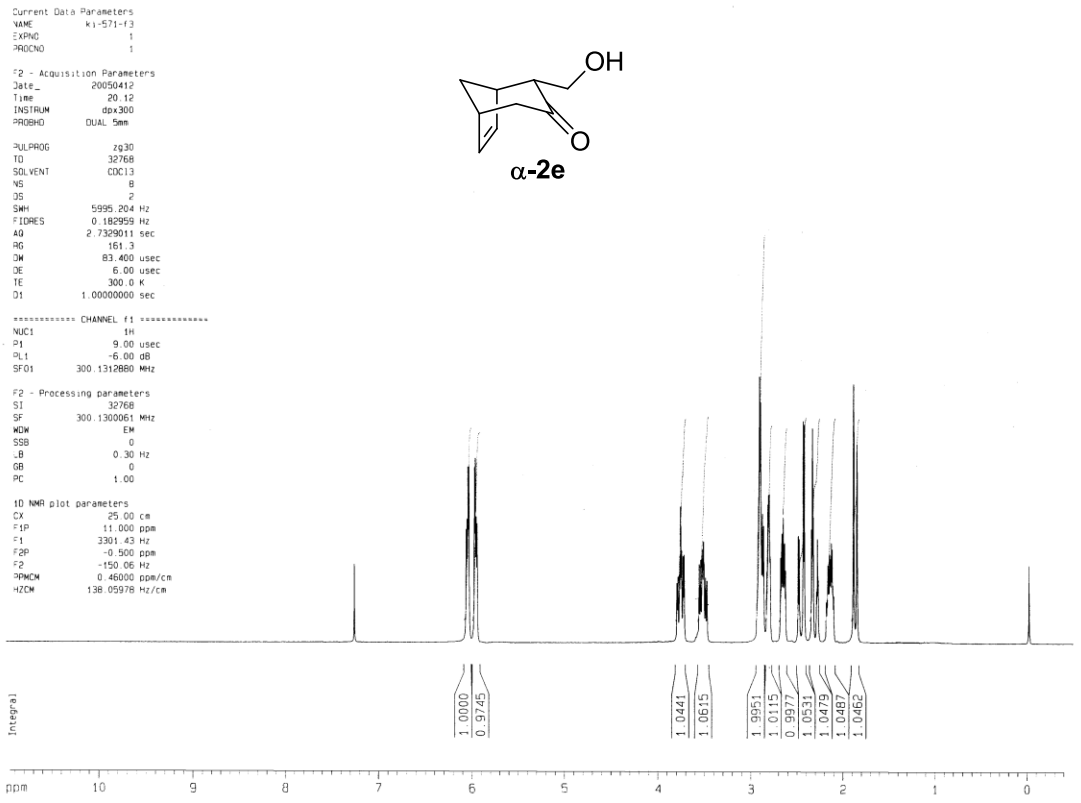

ppm 200 180 160 140 120 100 80 60 40 20 0

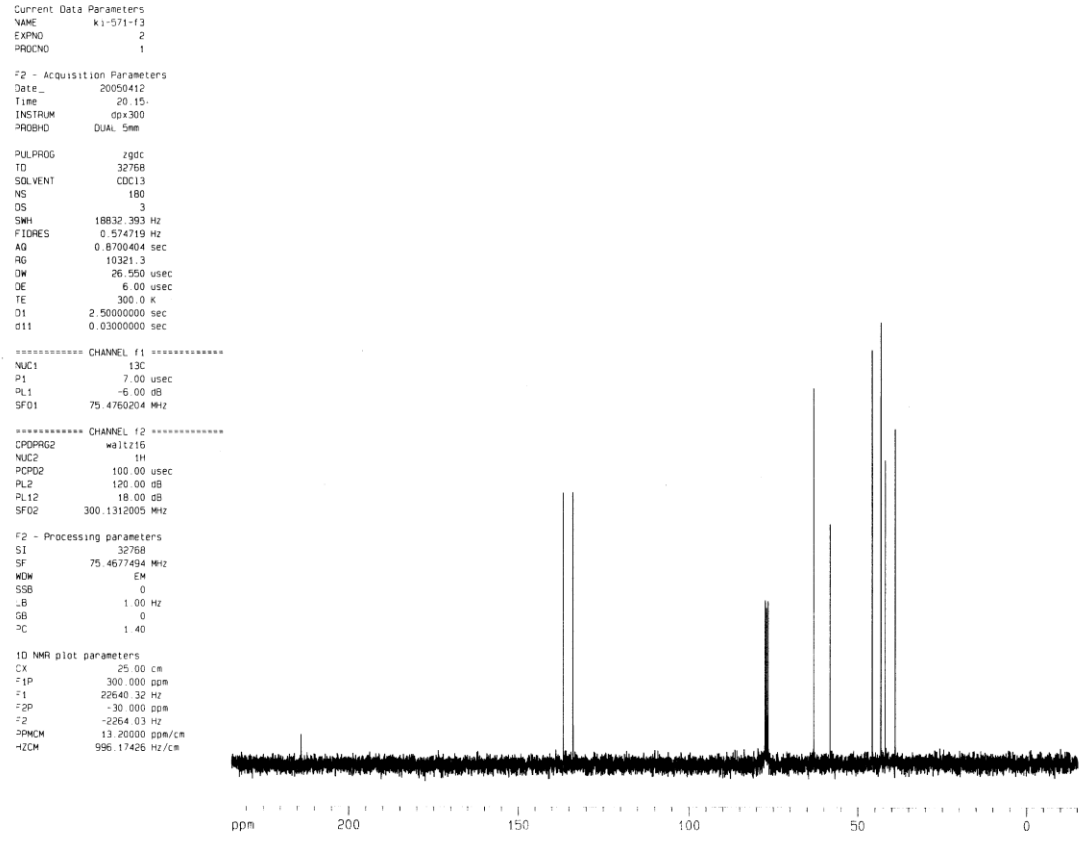

1
2
3
4
5
ppm
ppm 5
4
3
2
1

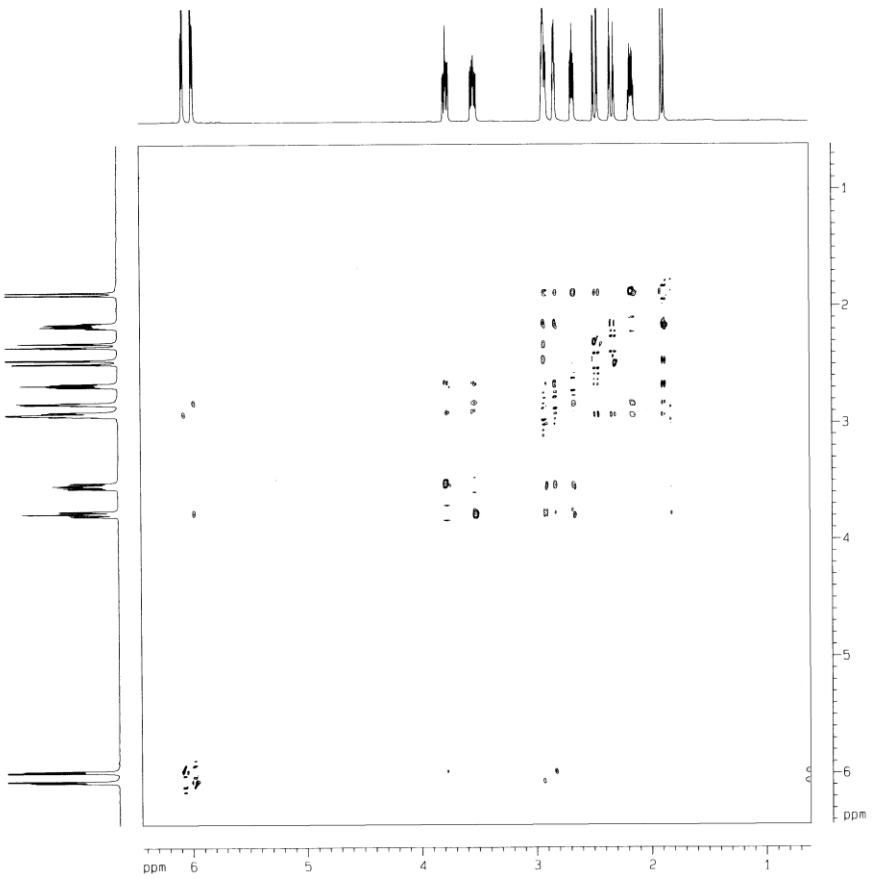

OH
O
O
β-2d
Integral
1.0000
1.0022
2.1286
2.0132
1.0266
1.1575
0.5321
0.6369
6.5356
ppm 10 9 8 7 6 5 4 3 2 1 0

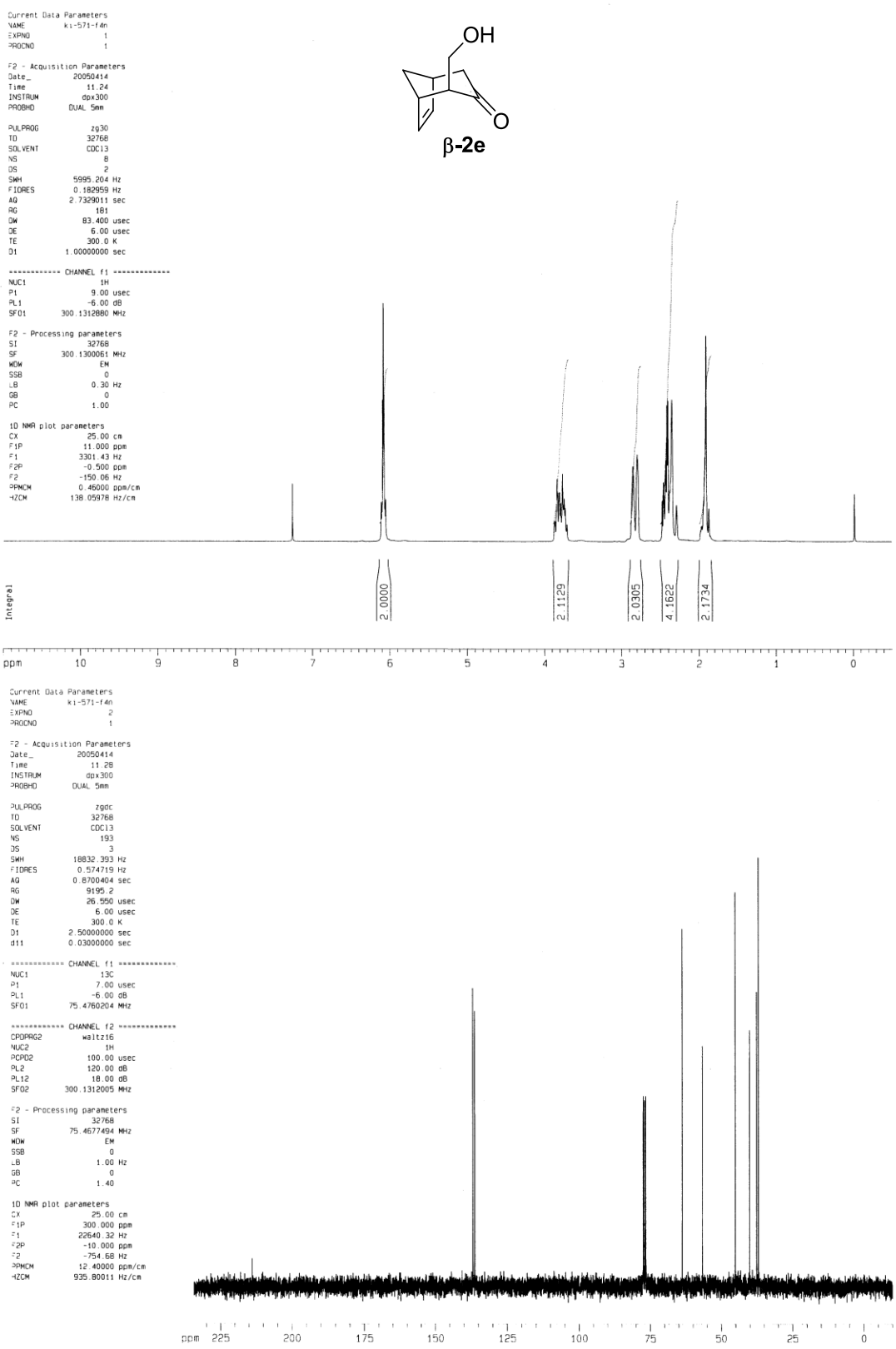

ppm 200 180 160 140 120 100 80 60 40 20 0

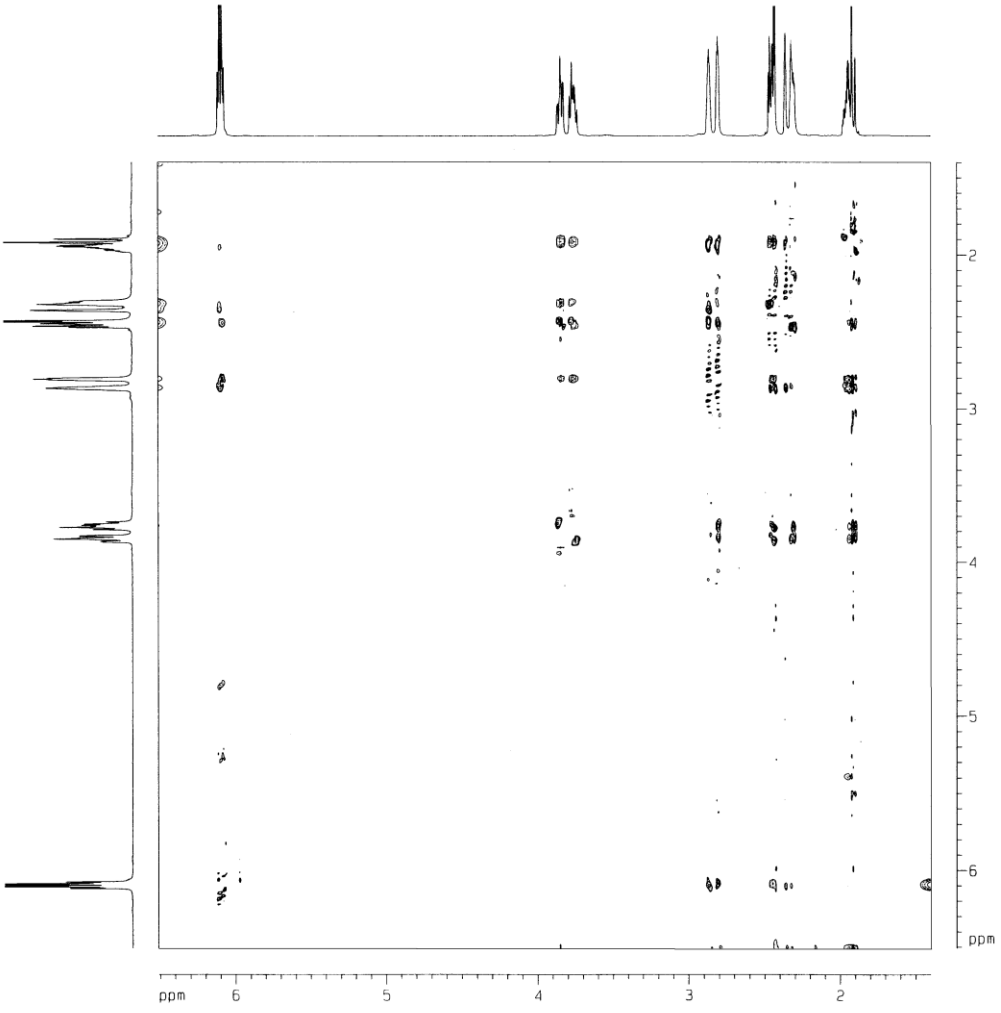

2
3
4
5
ppm
ppm 5
4
3
2

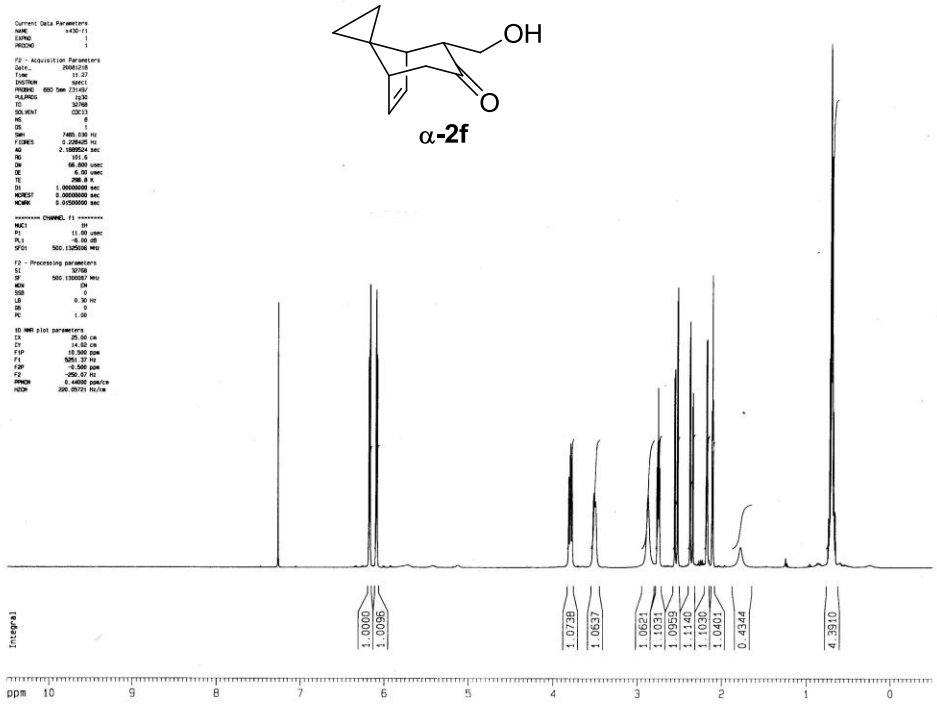

Current Data Parameters
NAME ki-571-f3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050412
Time 20.12
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 161.3
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1312880 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.46000 ppm/cm
HZCM 138.05978 Hz/cm
OH
O
α-2e
Integral
1.0000
0.9745
1.0441
1.0615
1.9951
1.0115
0.9977
1.0531
1.0479
1.0487
1.0462
ppm
10
9
8
7
6
5
4
3
2
1
0

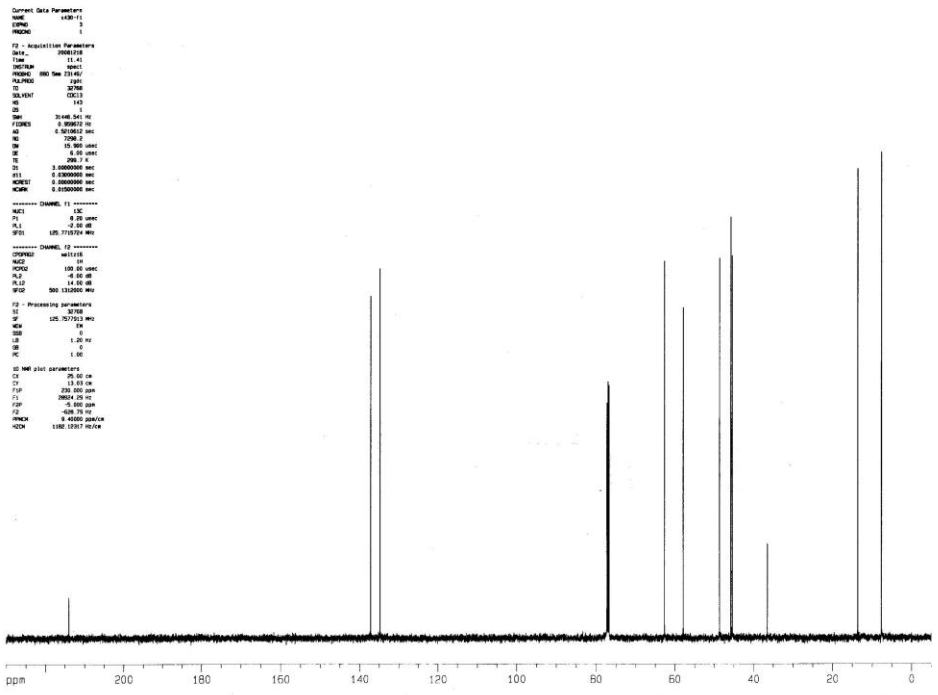

Current Data Parameters
NAME ki-571-f3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050412
Time 20.15
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zgdc
TD 32768
SOLVENT CDCl3
NS 180
DS 3
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 10321.3
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.4760204 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1P 300.000 ppm
F1 22640.32 Hz
F2P -30.000 ppm
F2 -2264.03 Hz
PPMCM 13.20000 ppm/cm
HZCM 996.17426 Hz/cm
ppm
200
150
100
50
0

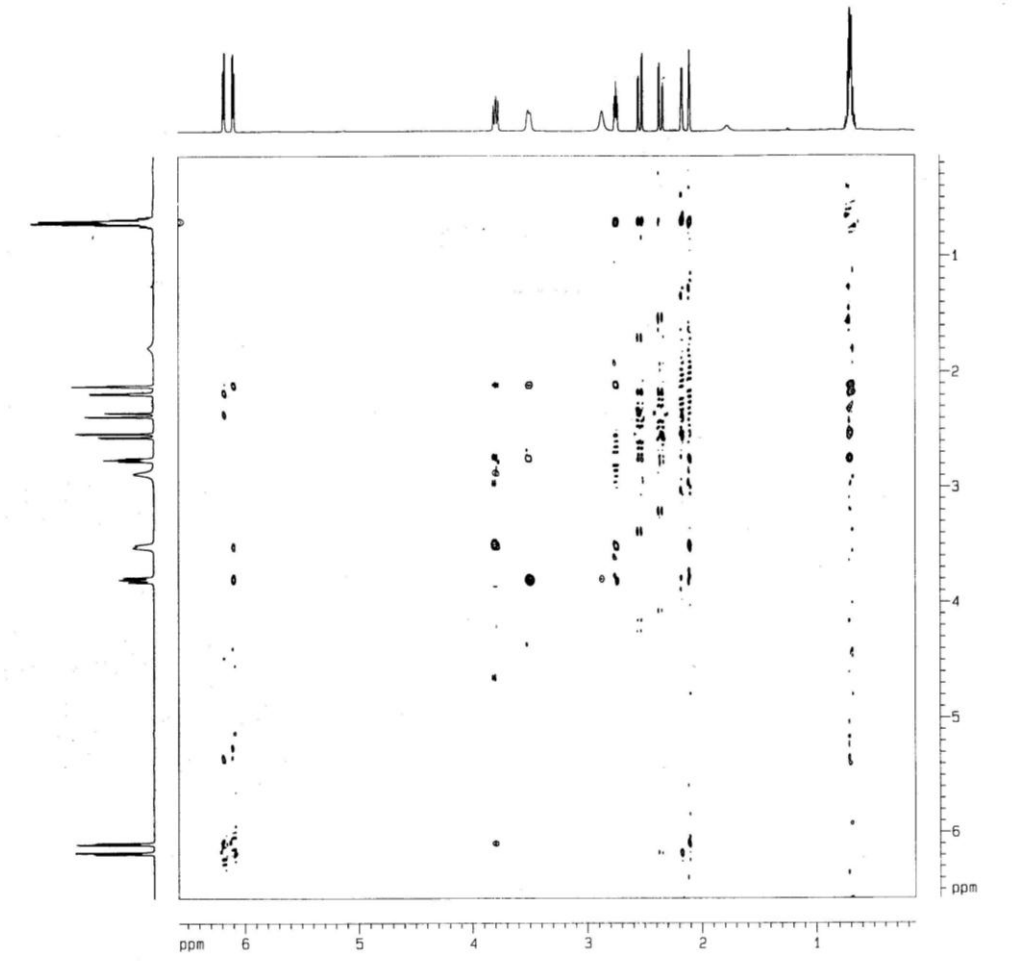

1
2
3
4
5
6
ppm
ppm 6
5
4
3
2
1

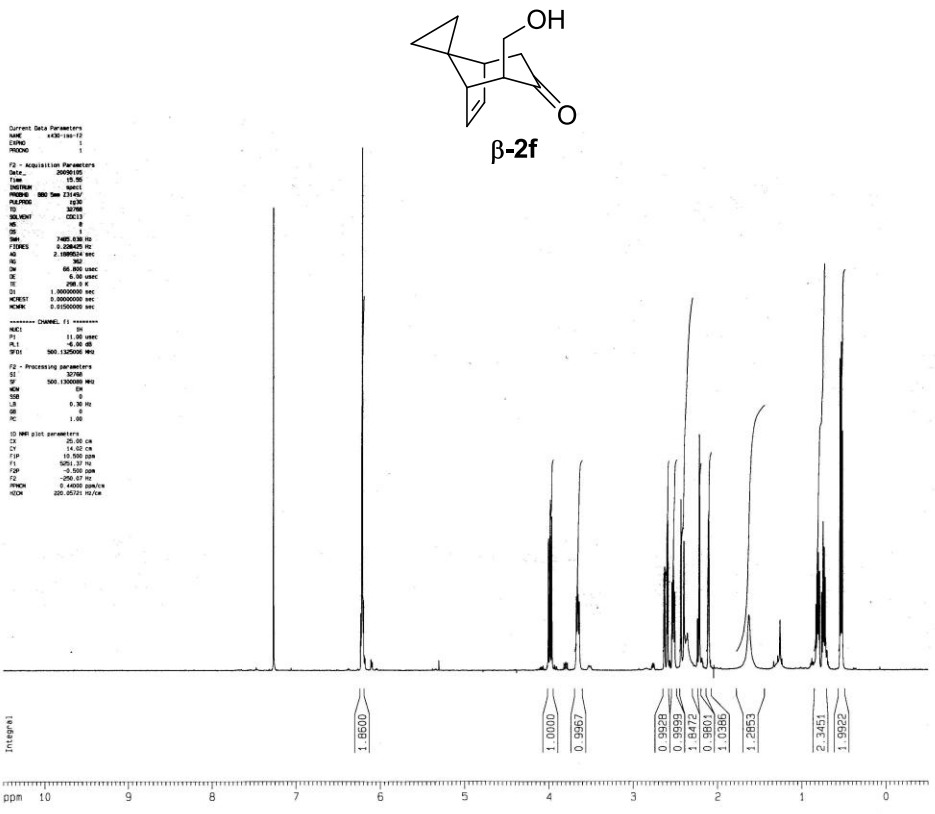

Current Data Parameters
NAME ki-571-f4n
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050414
Time 11.24
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 181
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SF01 300.1312880 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.46000 ppm/cm
HZCM 138.05978 Hz/cm
OH
O
β-2e
Integral
2.0000
2.1129
2.0305
4.1622
2.1734
ppm
Current Data Parameters
NAME ki-571-f4n
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050414
Time 11.28
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zgdc
TD 32768
SOLVENT CDCl3
NS 193
DS 3
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 9195.2
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SF01 75.4760204 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1P 300.000 ppm
F1 22640.32 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 12.40000 ppm/cm
HZCM 935.80011 Hz/cm
ppm

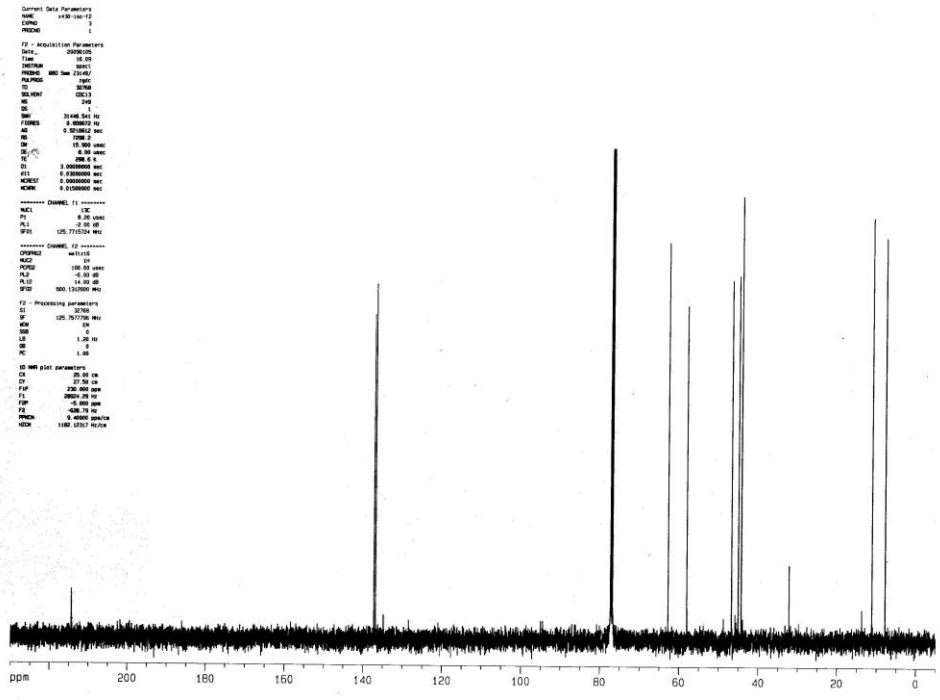

ppm
6
5
4
3
2
2
3
4
5
6
ppm

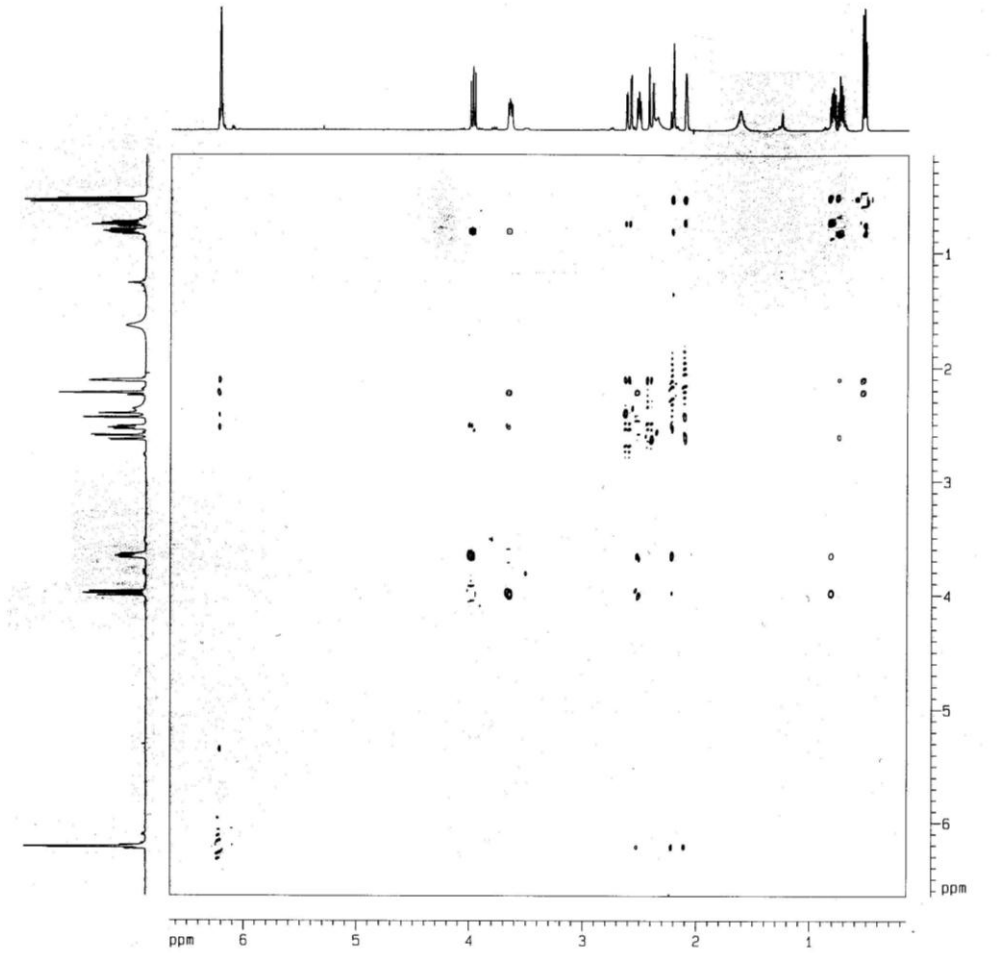

OH
O
α-2f
Integral
1.0000
1.0096
1.0738
1.0637
1.0621
1.1031
1.0959
1.1140
1.1030
1.0401
0.4344
4.3910
ppm 10 9 8 7 6 5 4 3 2 1 0

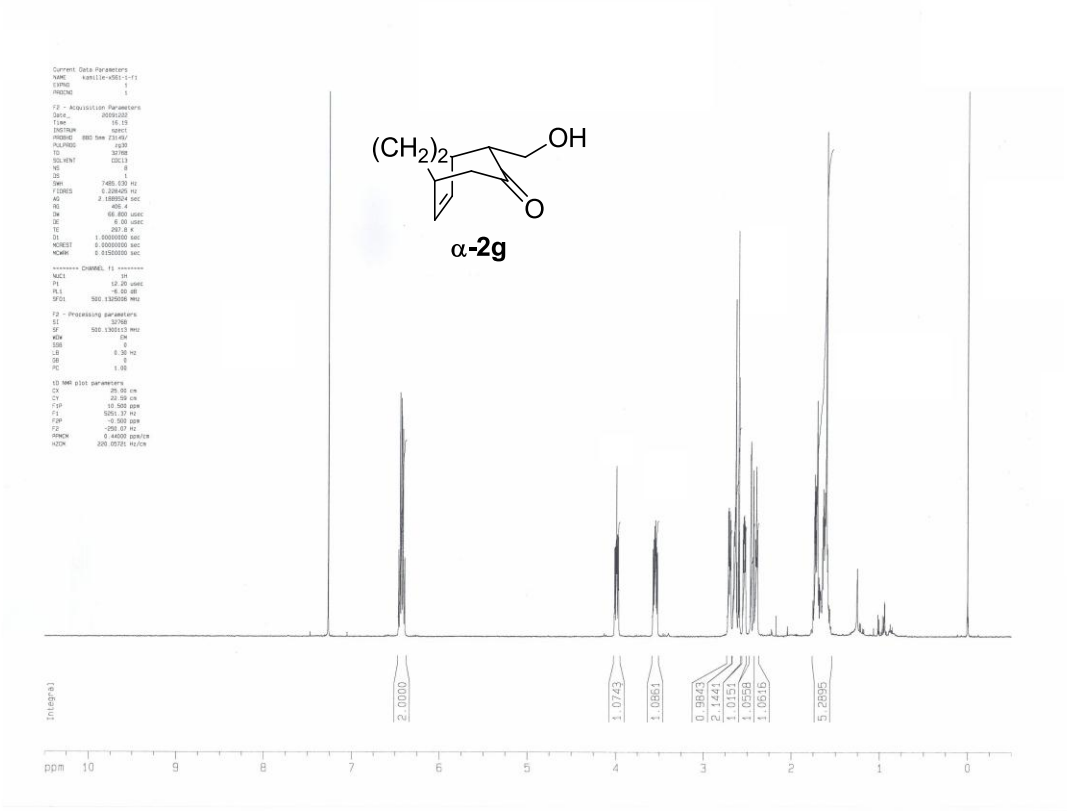

ppm 200 180 160 140 120 100 80 60 40 20 0

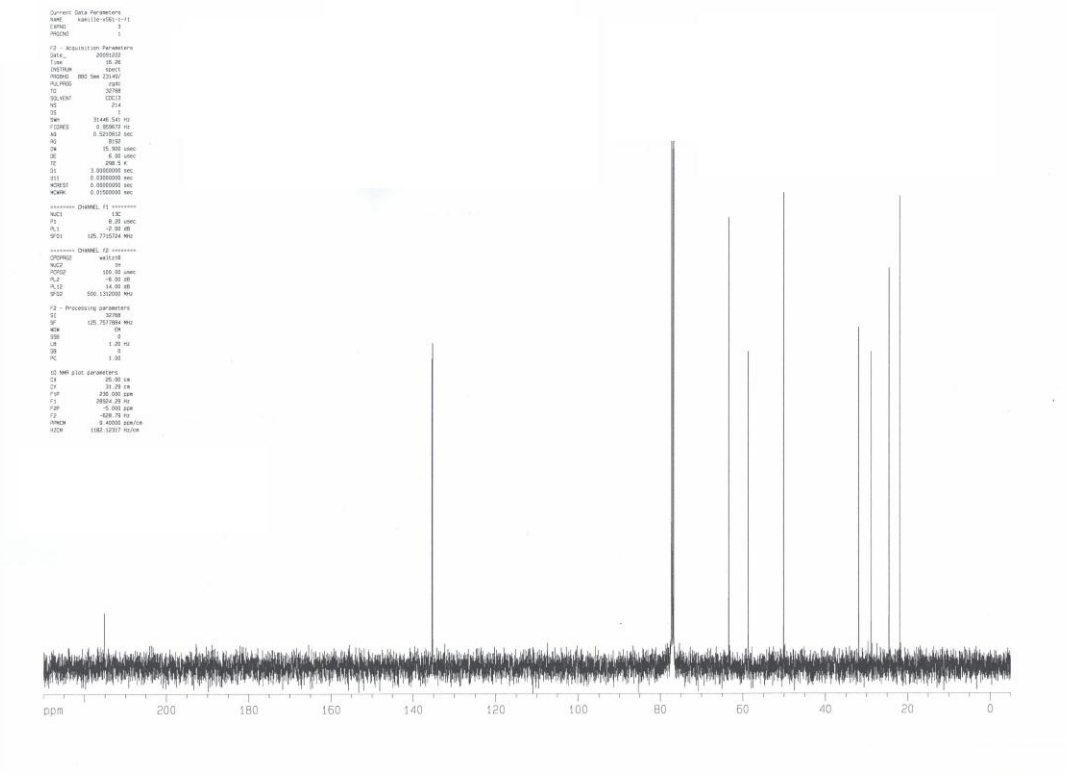

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

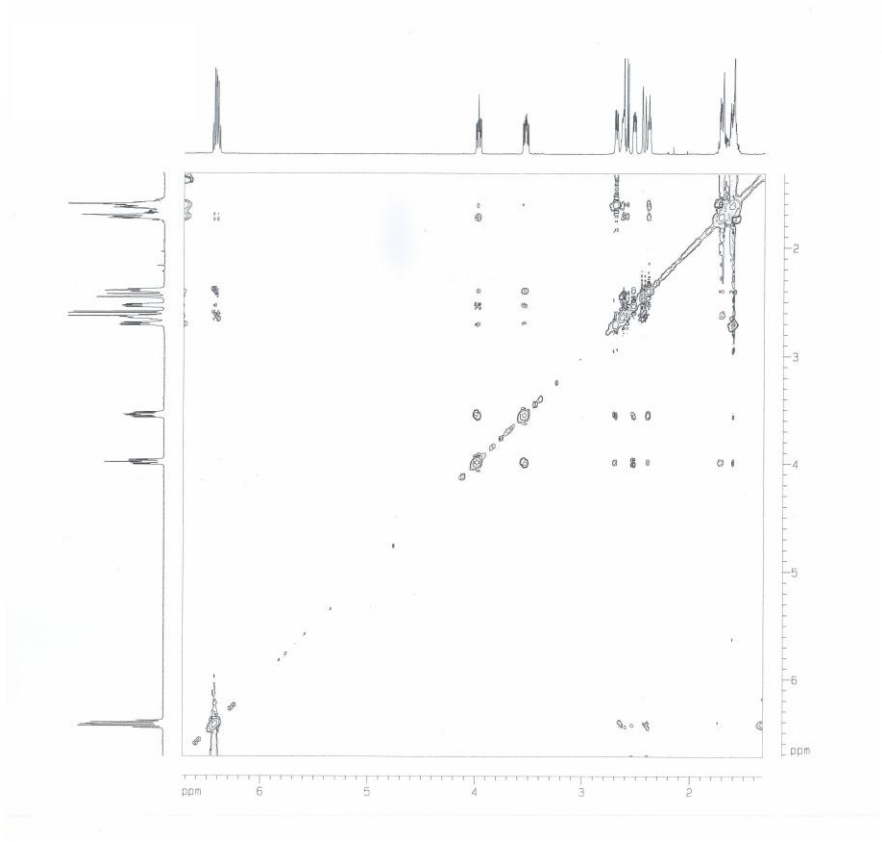

OH
O
β-2f
Integral
1.8600
1.0000
0.9967
0.9928
0.9999
1.6472
0.9801
1.0386
1.2853
2.3451
1.9922
ppm 10 9 8 7 6 5 4 3 2 1 0

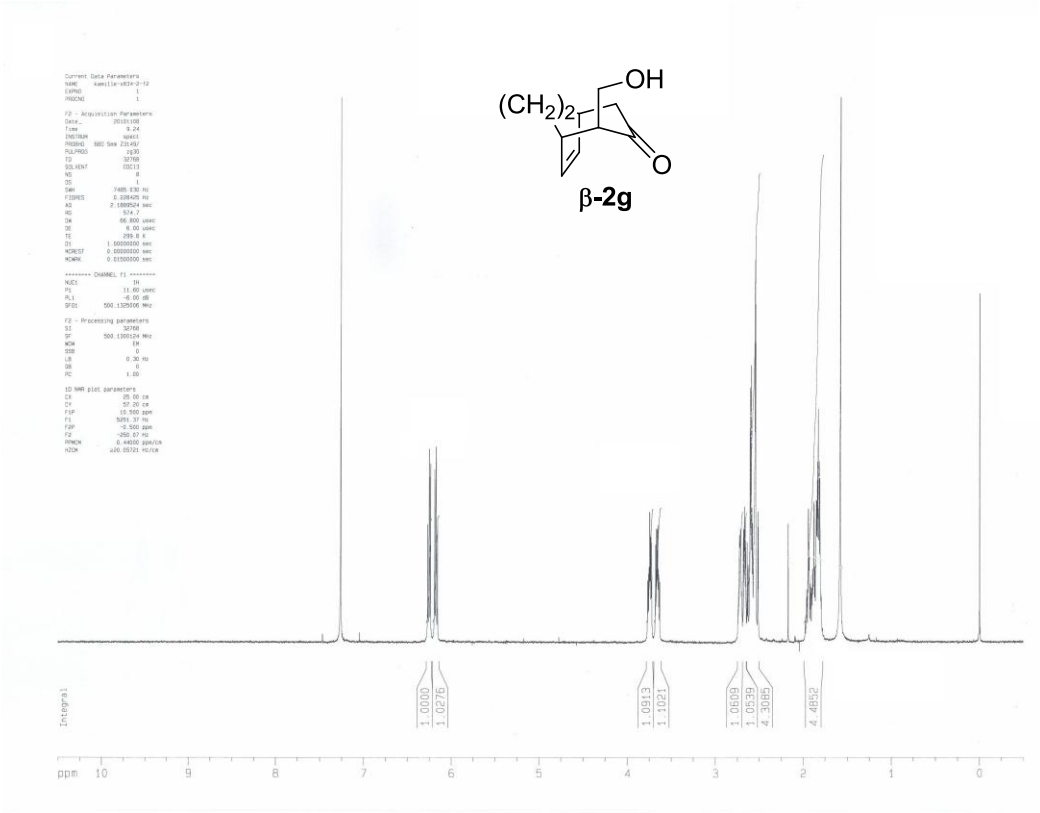

ppm 200 180 160 140 120 100 80 60 40 20 0

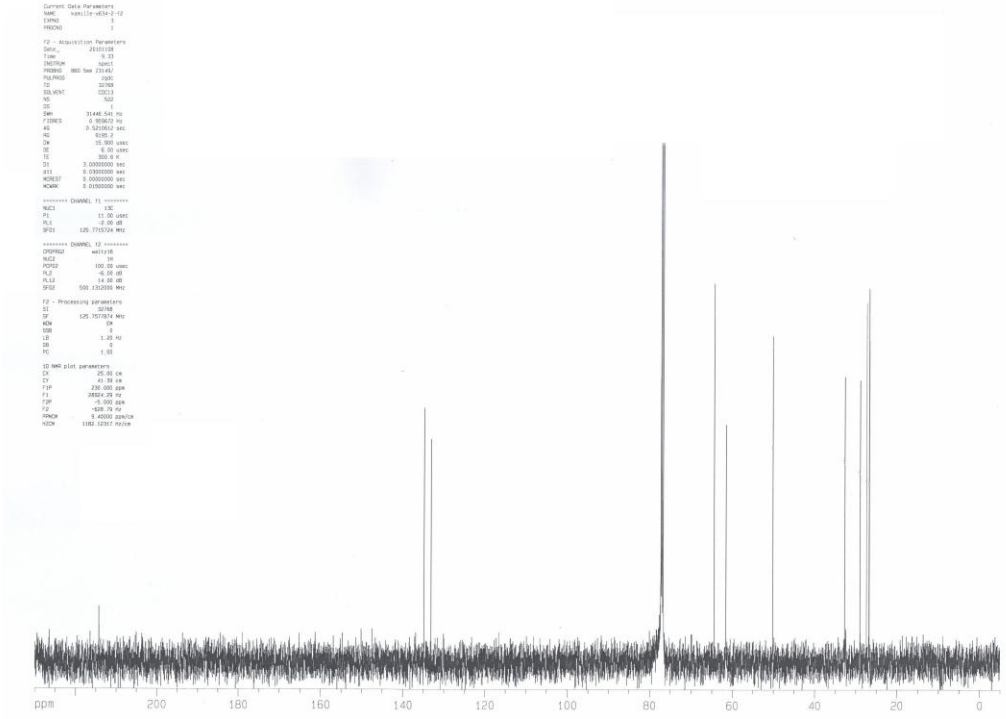

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

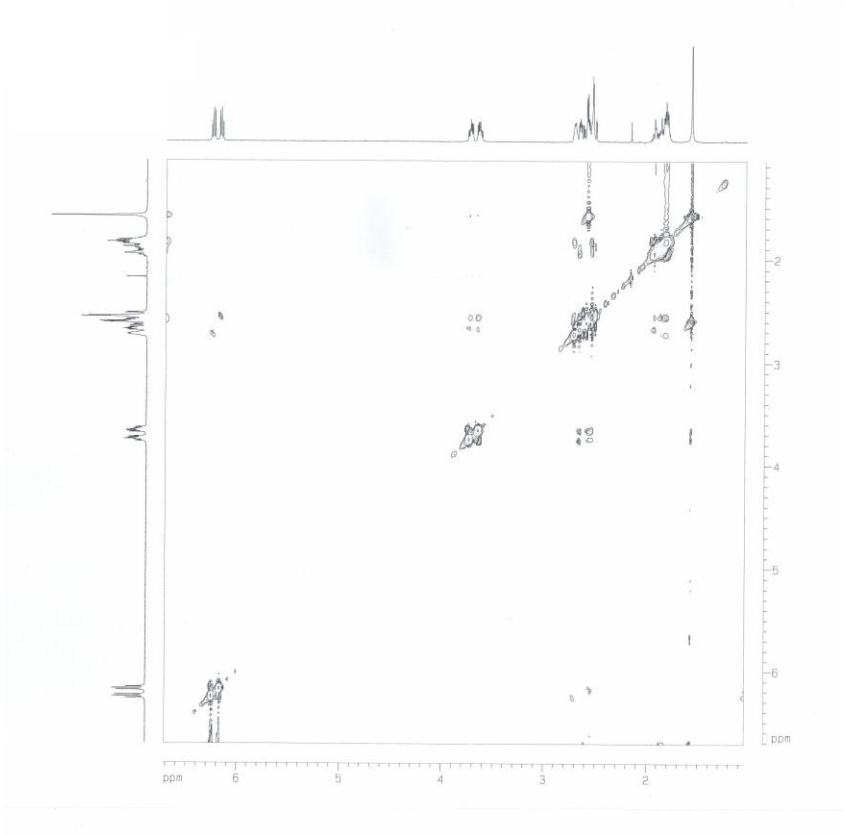

(CH2)2
OH
O
α-2g
Integral
2.0000
1.0743
1.0861
0.9643
2.1441
1.0151
1.0558
1.0616
5.2895
ppm 10 9 8 7 6 5 4 3 2 1 0

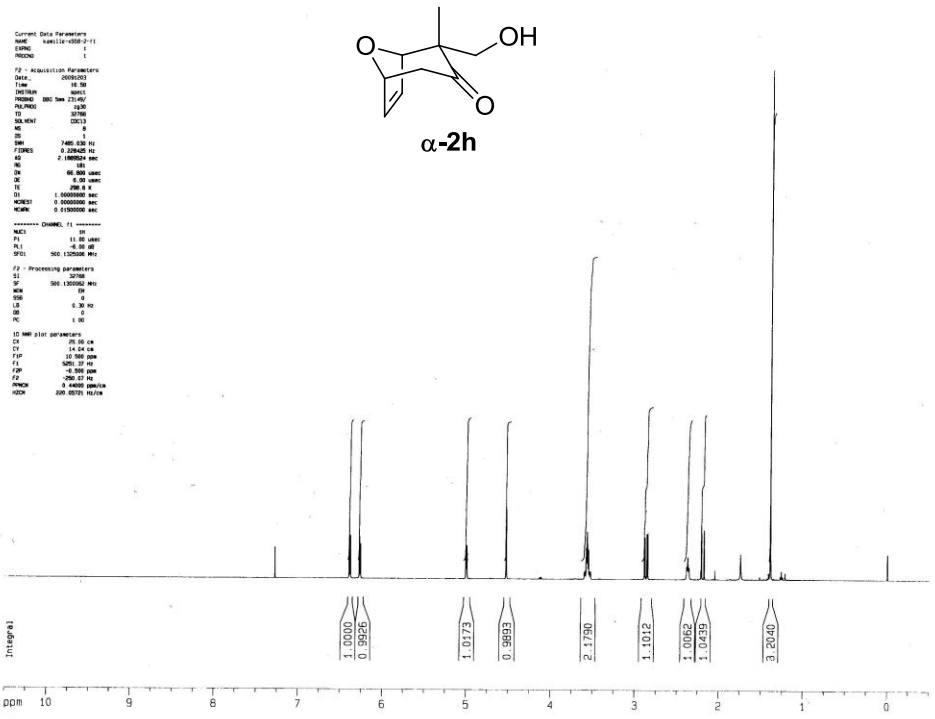

ppm 200 180 160 140 120 100 80 60 40 20 0

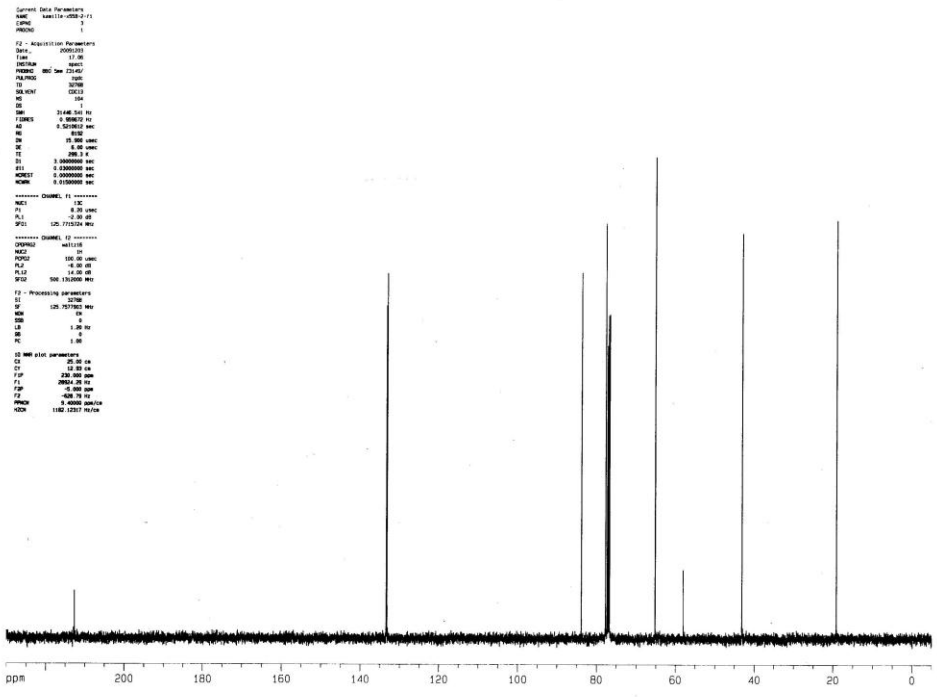

ppm
6
5
4
3
2
ppm
2
3
4
5
6

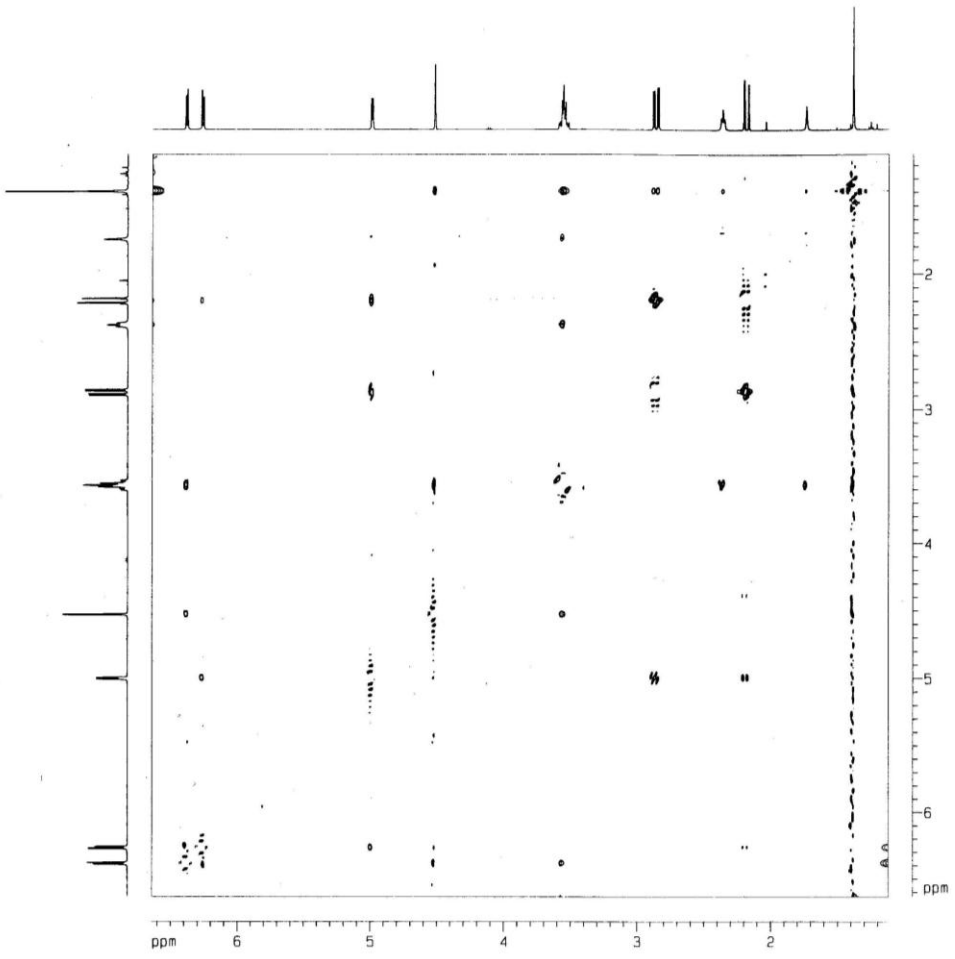

OH
(CH2)2
O
β-2g
Integral
1.0000
1.0276
1.0913
1.1021
1.0609
1.0539
4.3085
4.4852
ppm 10 9 8 7 6 5 4 3 2 1 0

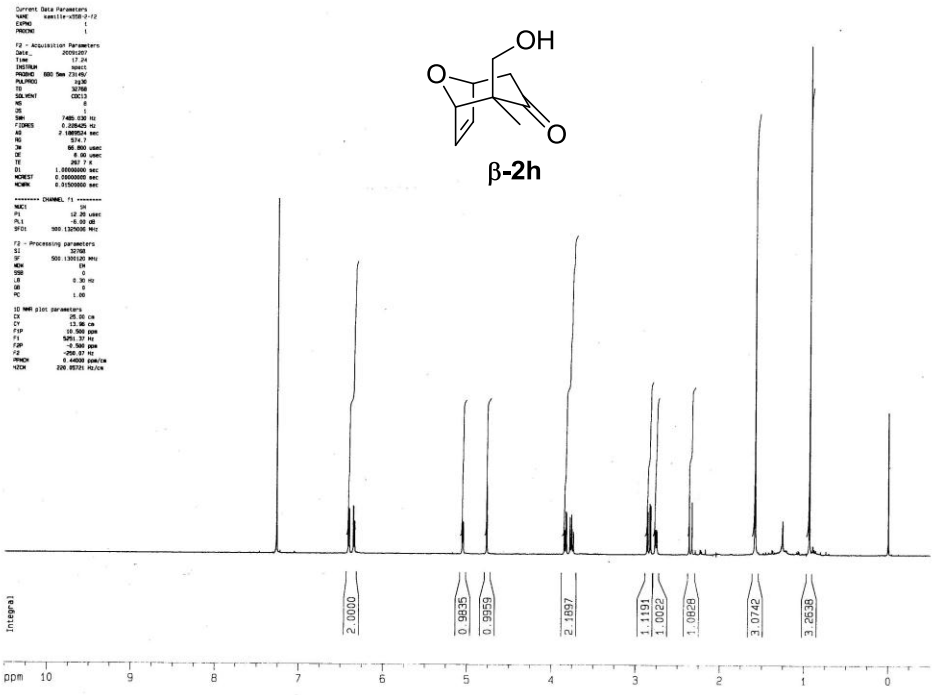

ppm 200 180 160 140 120 100 80 60 40 20 0

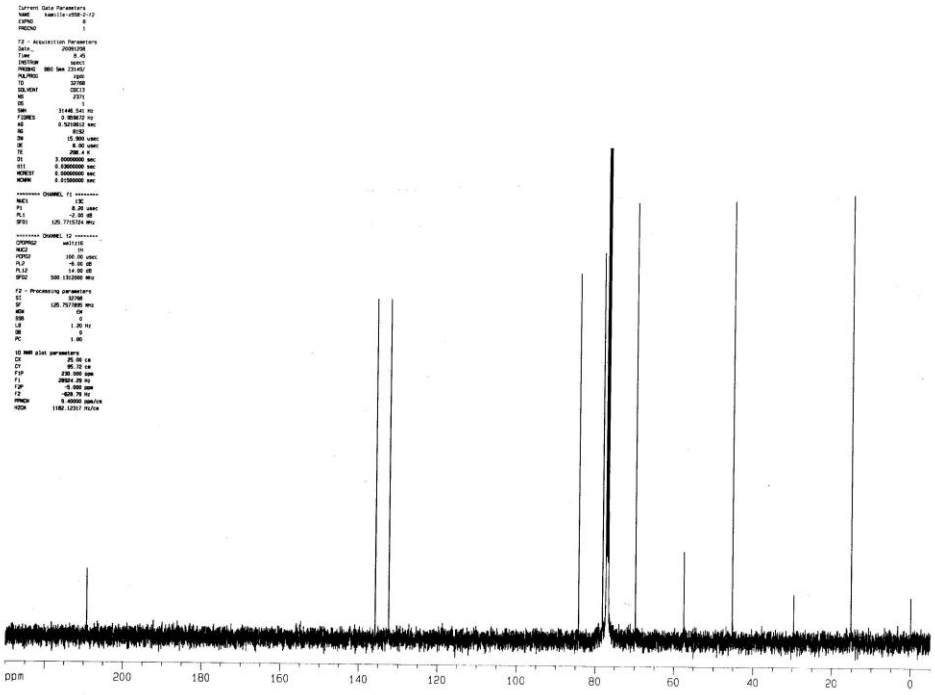

ppm
6
5
4
3
2
ppm
2
3
4
5
6

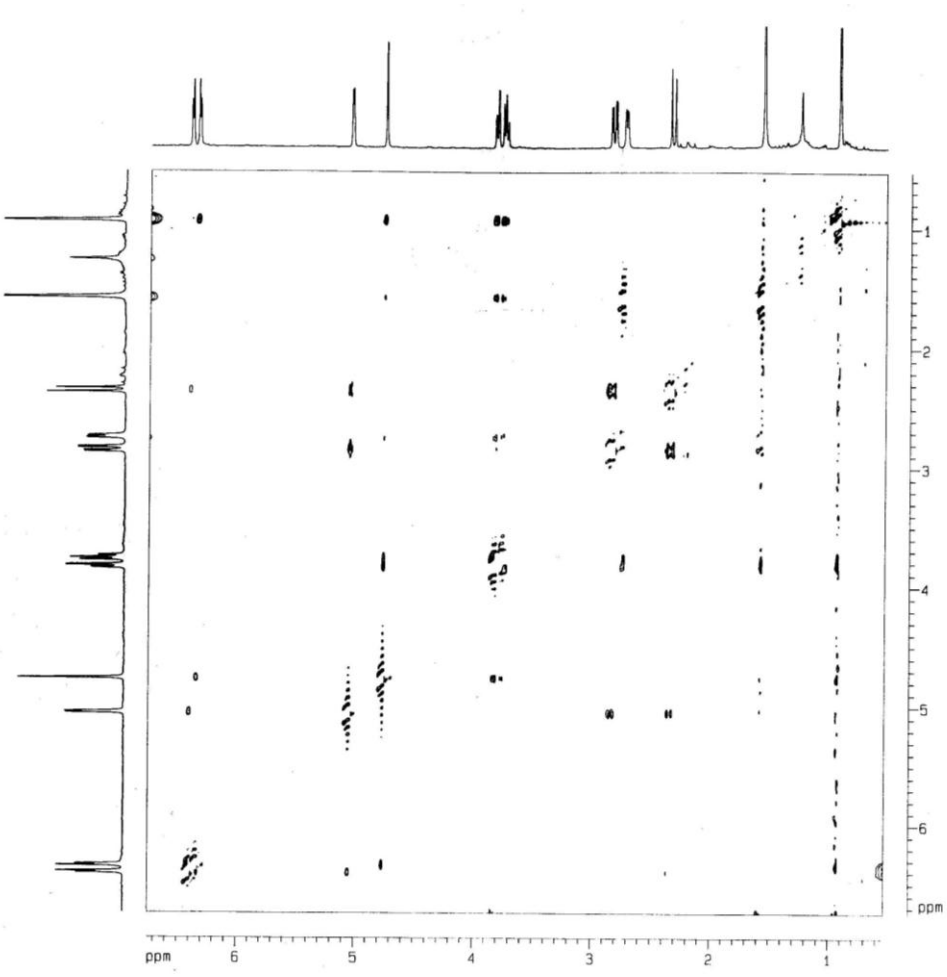

OH
O
O
α-2h
Integral
1.0000
0.9926
1.0173
0.9893
2.1790
1.1012
1.0062
1.0439
3.2040
ppm

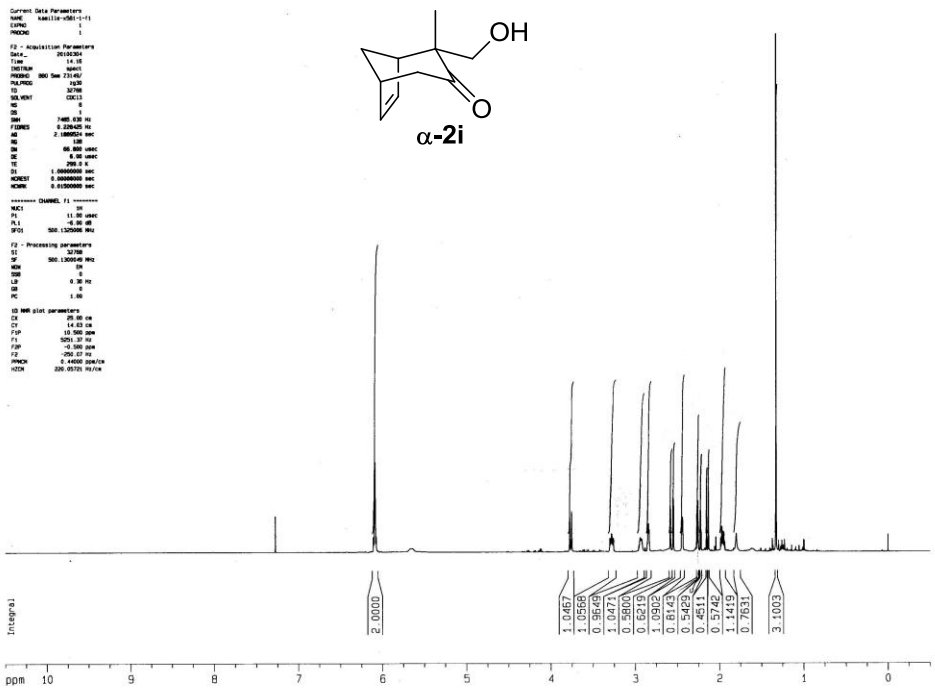

ppm

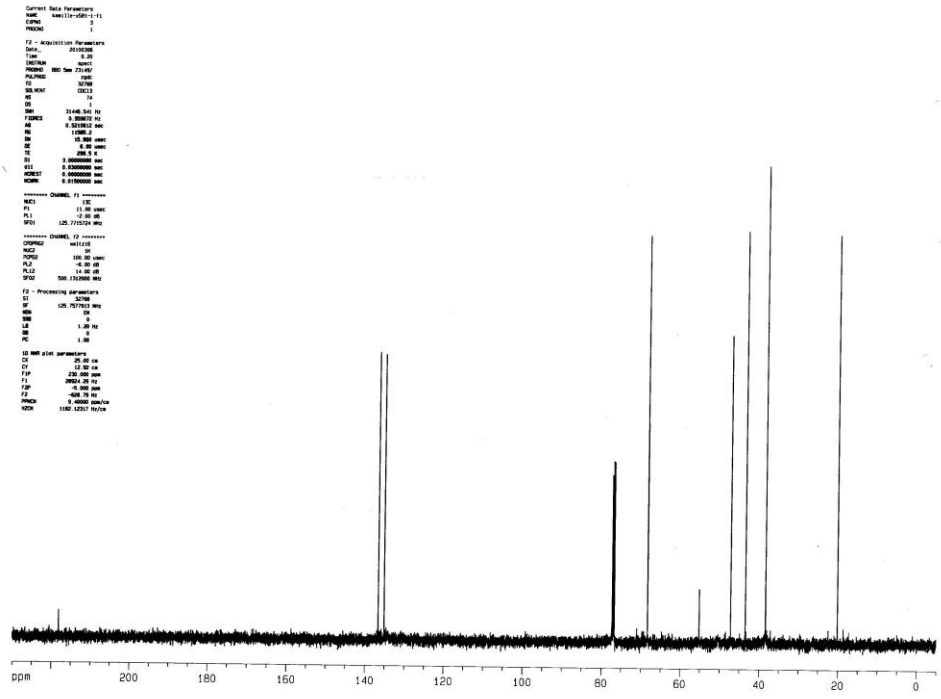

ppm
6
5
4
3
2
ppm
2
3
4
5
6

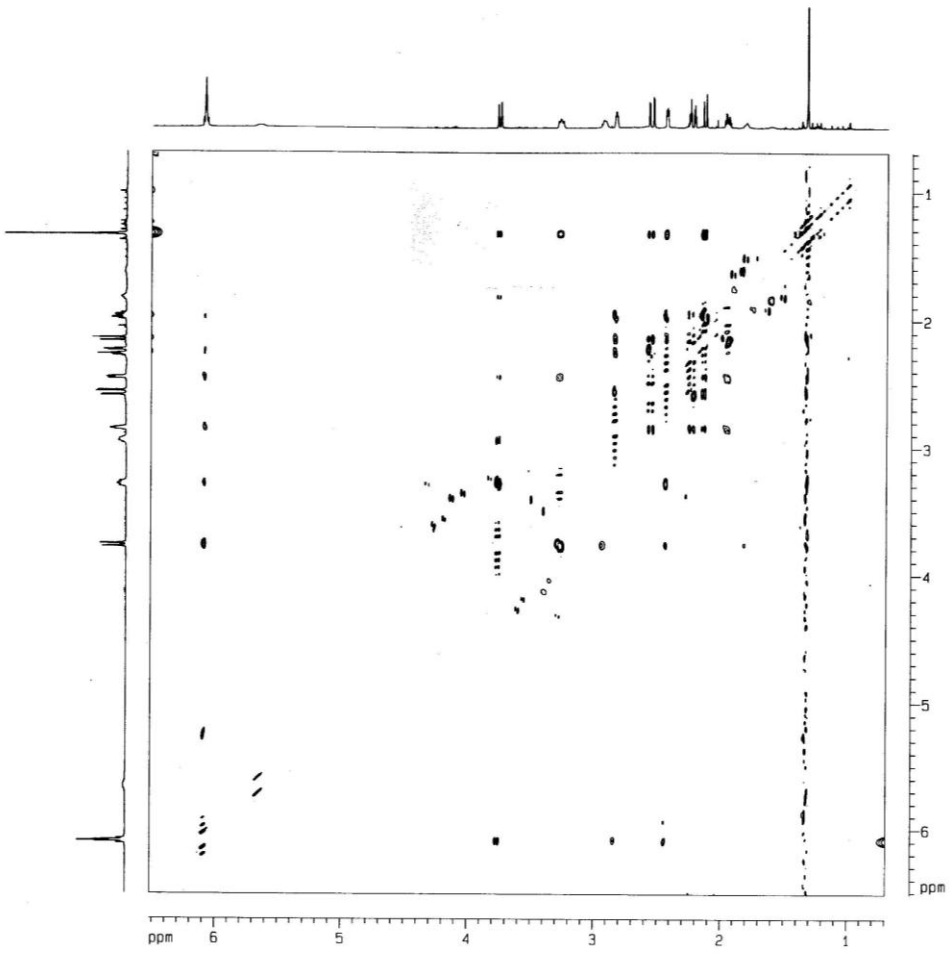

OH
O
O
β-2h
Integral
2.0000
0.9835
0.9959
2.1897
1.1191
1.0022
1.0828
3.0742
3.2638
ppm
10
9
8
7
6
5
4
3
2
1
0

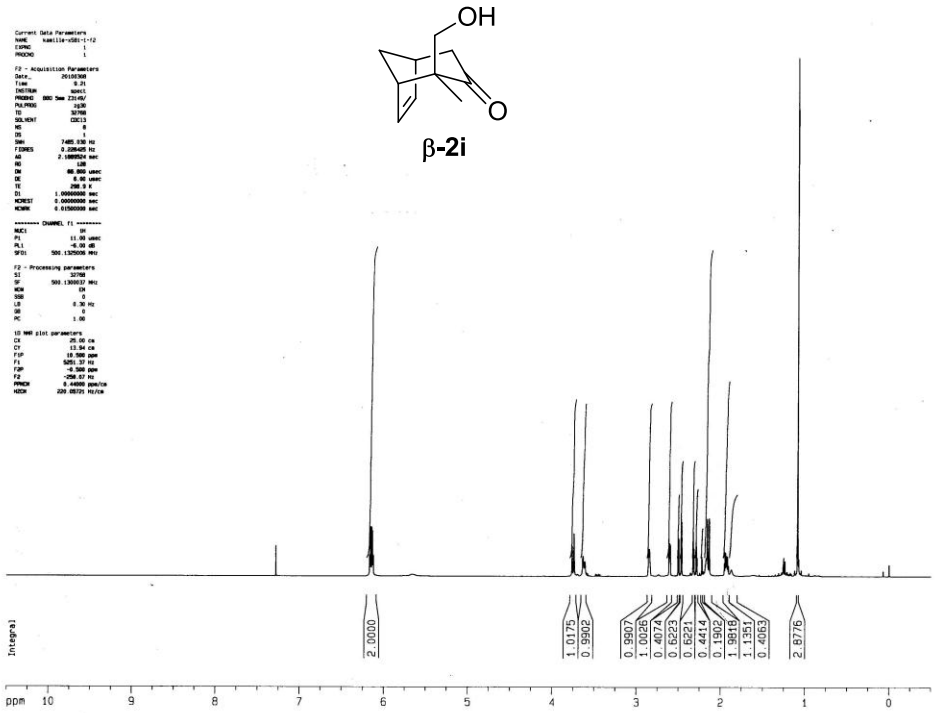

ppm
200
180
160
140
120
100
80
60
40
20
0

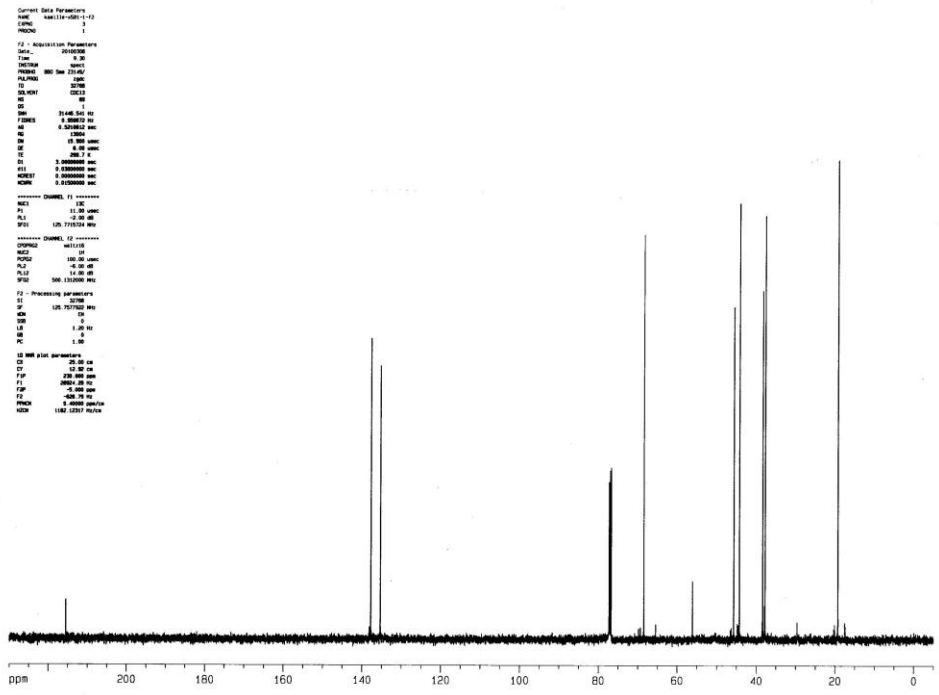

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

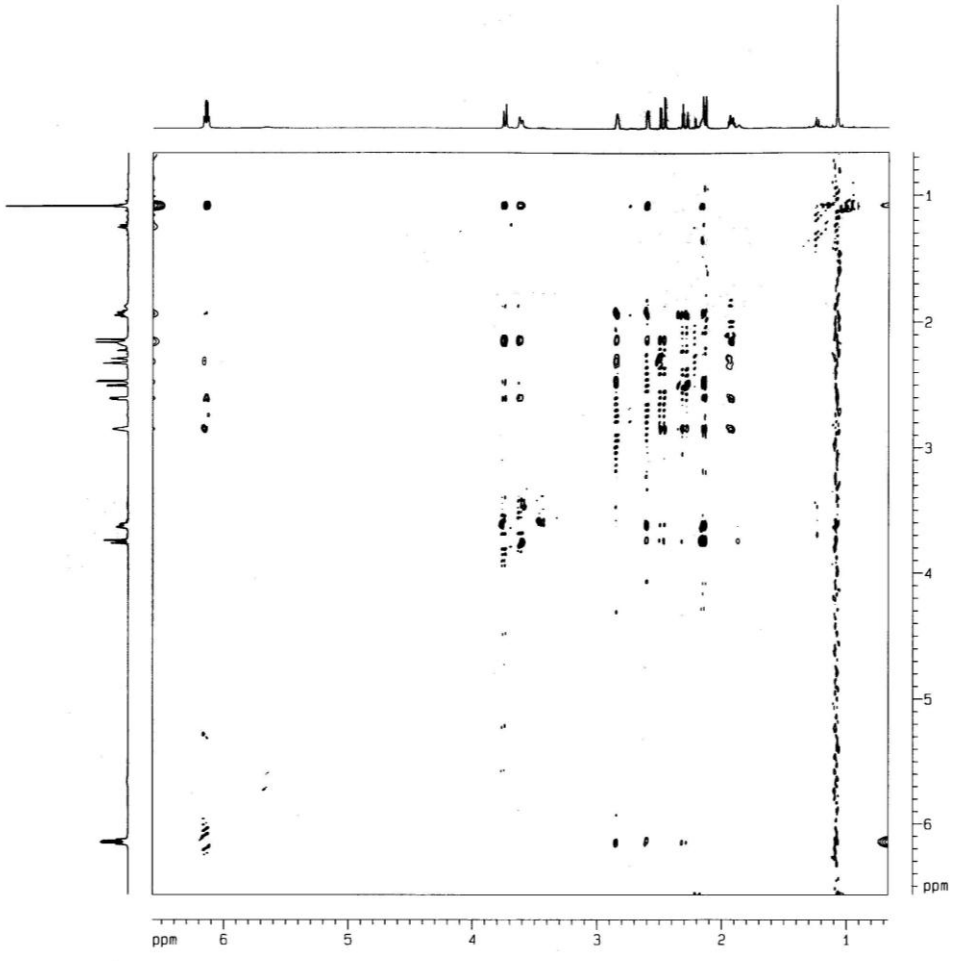

OH
O
α-2i
Integral
2.0000
1.0467
1.0568
0.9649
1.0471
0.5800
0.6219
1.0902
0.8143
0.5429
0.4511
0.5742
1.1419
0.7631
3.1003
ppm
10
9
8
7
6
5
4
3
2
1
0

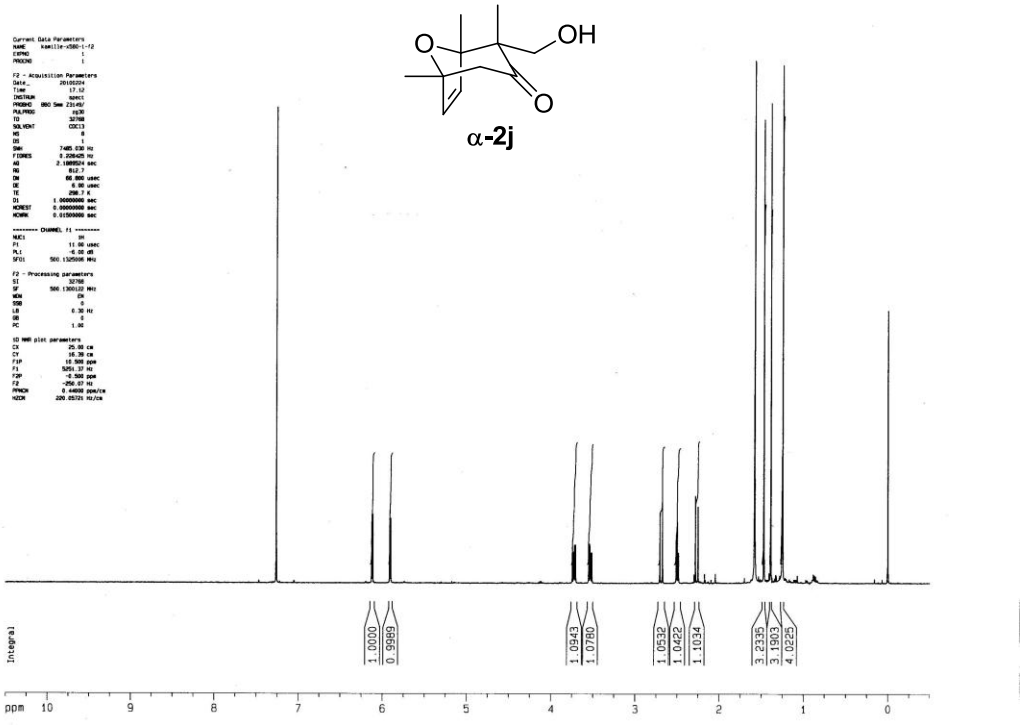

ppm
200
180
160
140
120
100
80
60
40
20
0

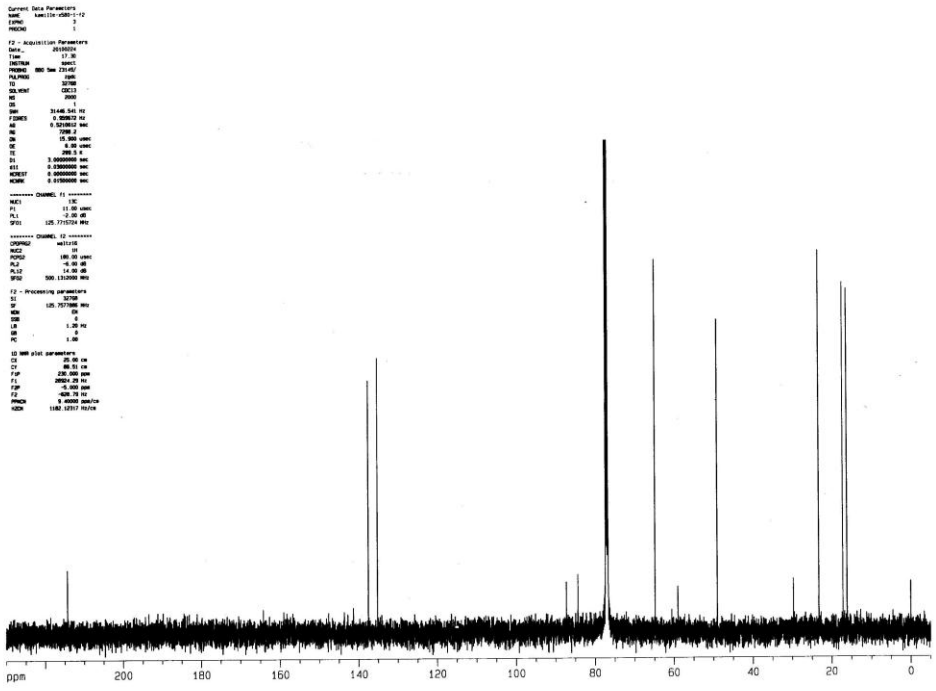

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

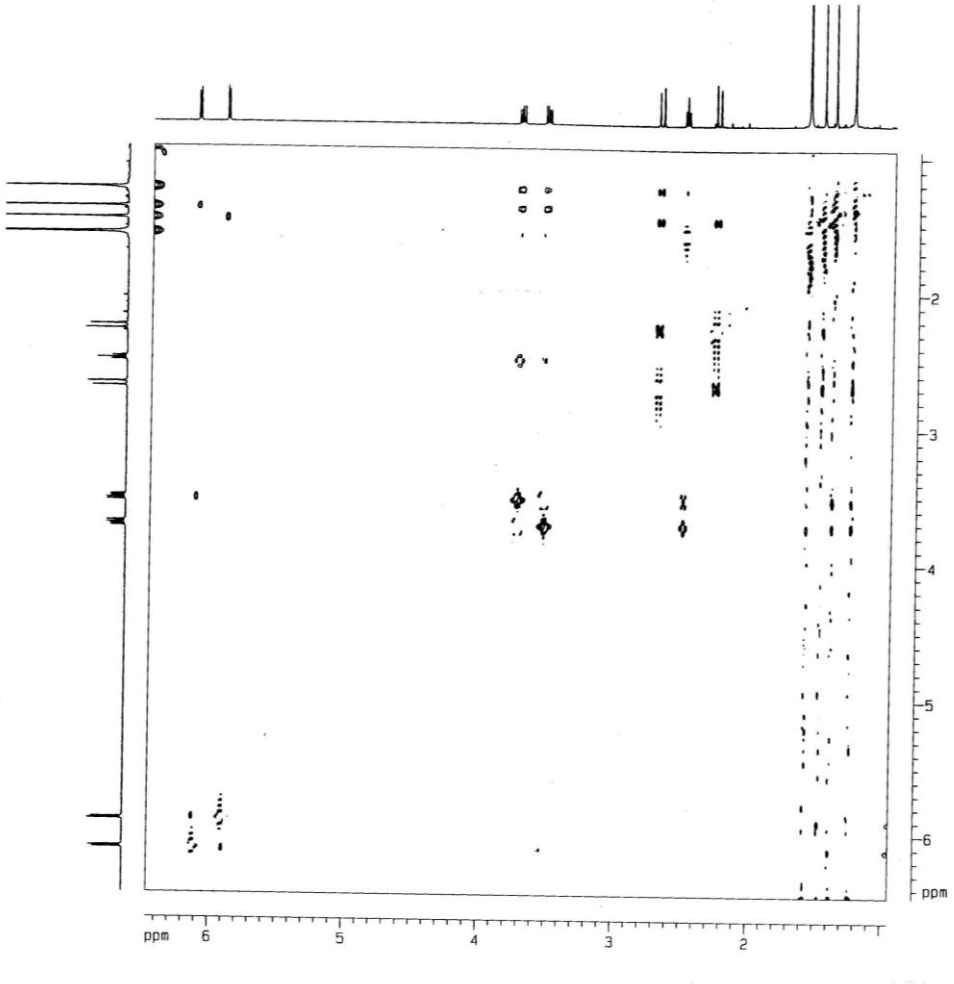

OH
O
β-2i
Integral
2.0000
1.0175
0.9902
0.9907
1.0026
0.4074
0.6223
0.6221
0.4414
0.1902
1.9816
1.1351
0.4063
2.8776
ppm

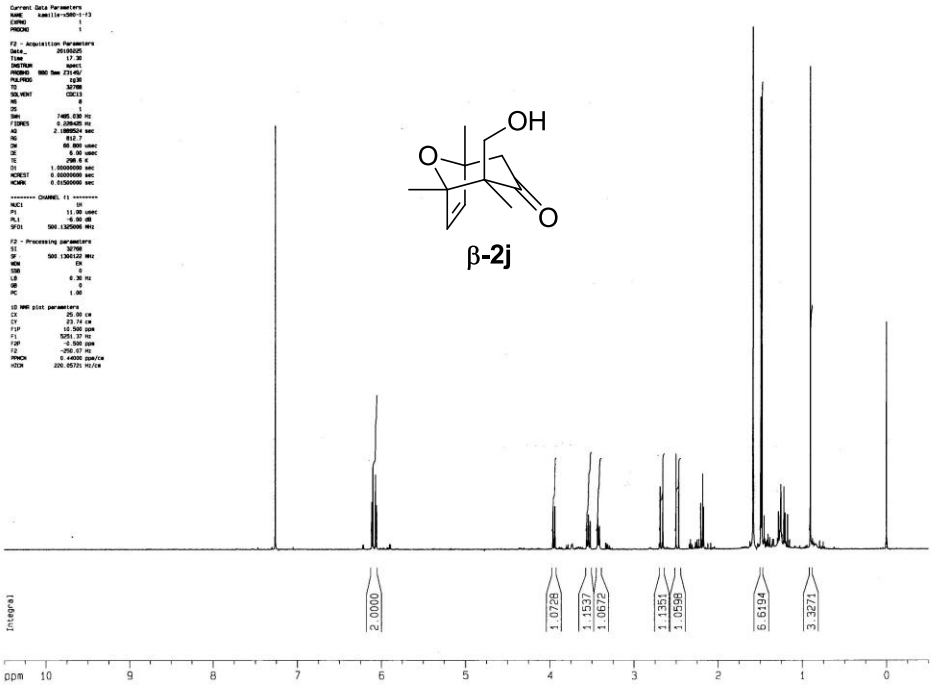

ppm


ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

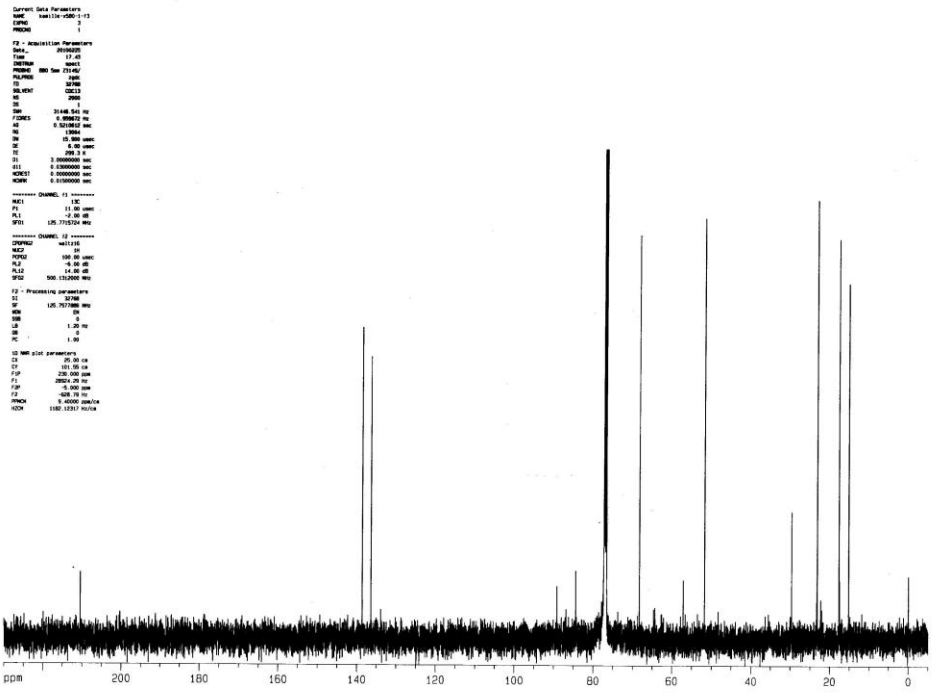

O
OH
O
α-2j
Integral
1.0000
0.9989
1.0943
1.0780
1.0532
1.0422
1.1034
3.2335
3.1903
4.0225
ppm 10 9 8 7 6 5 4 3 2 1 0

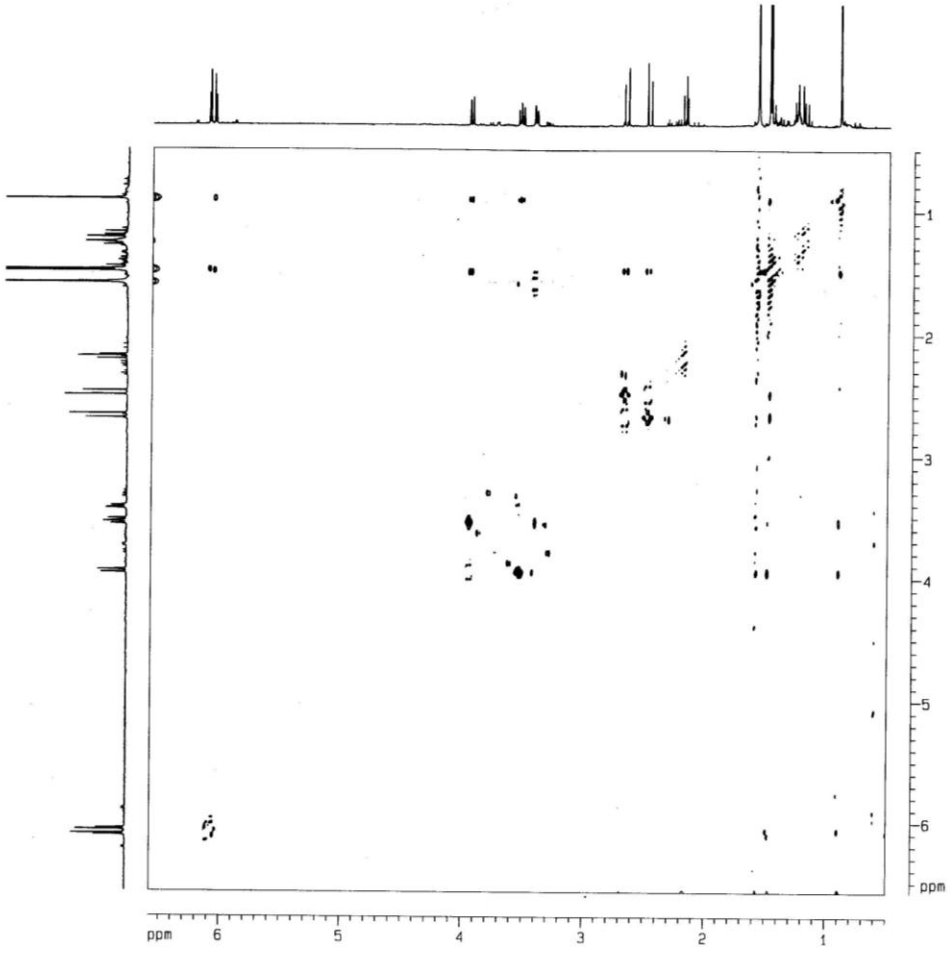

ppm 200 180 160 140 120 100 80 60 40 20 0

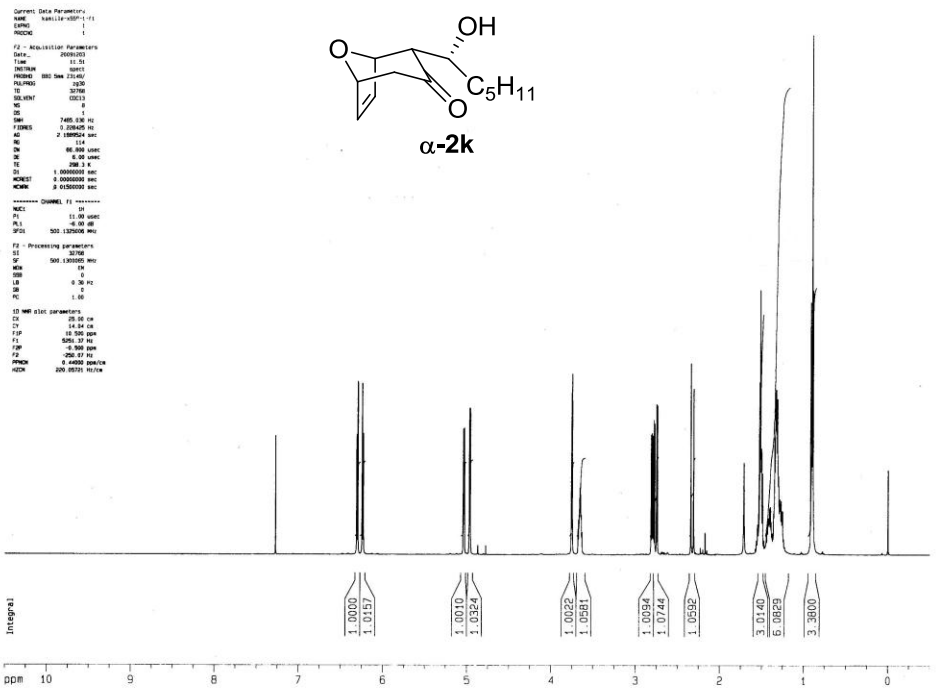

ppm
6
5
4
3
2
2
3
4
5
6
ppm

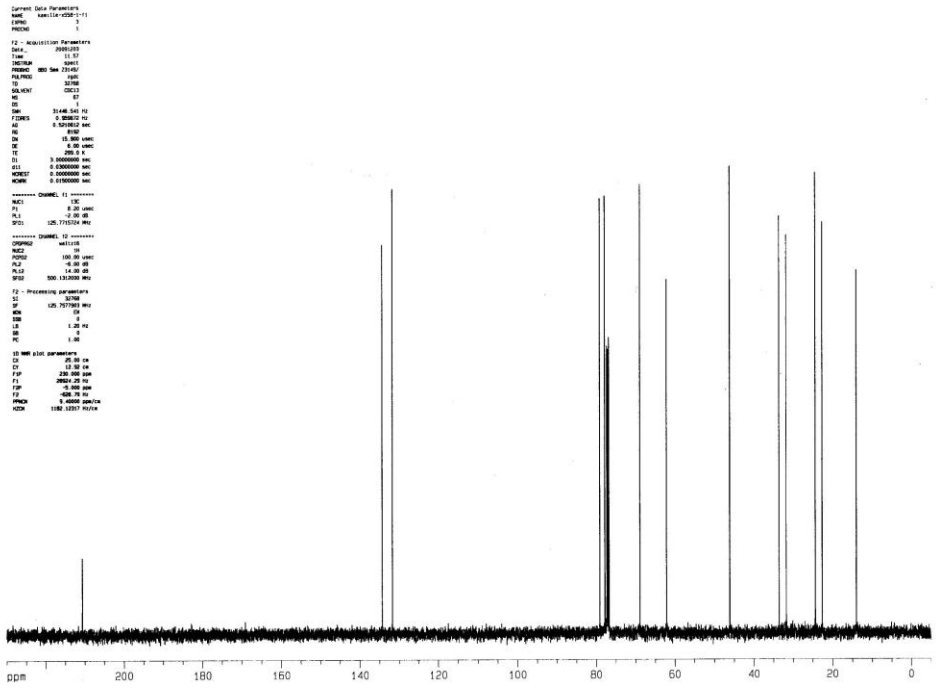

OH
O
O
Integral
2.0000
1.0728
1.1537
1.0672
1.1351
1.0598
6.6194
3.3271
ppm
10
9
8
7
6
5
4
3
2
1
0

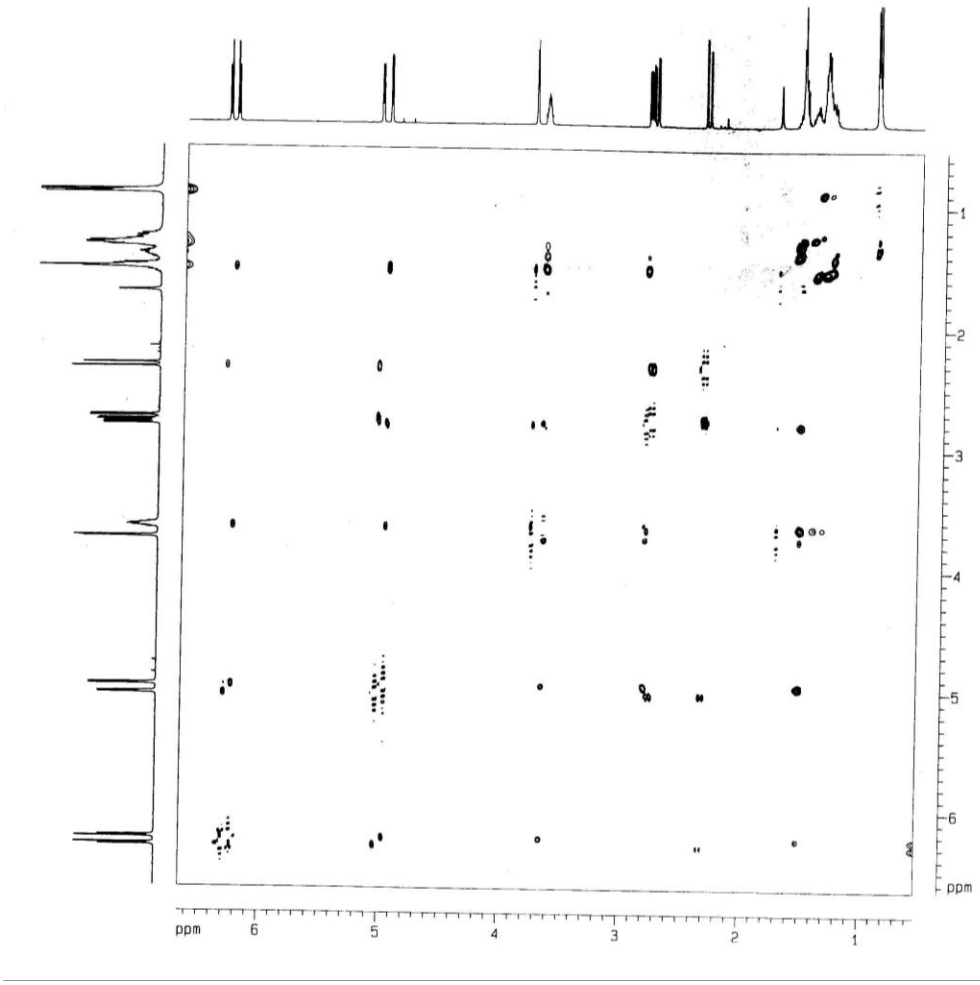
β-2j

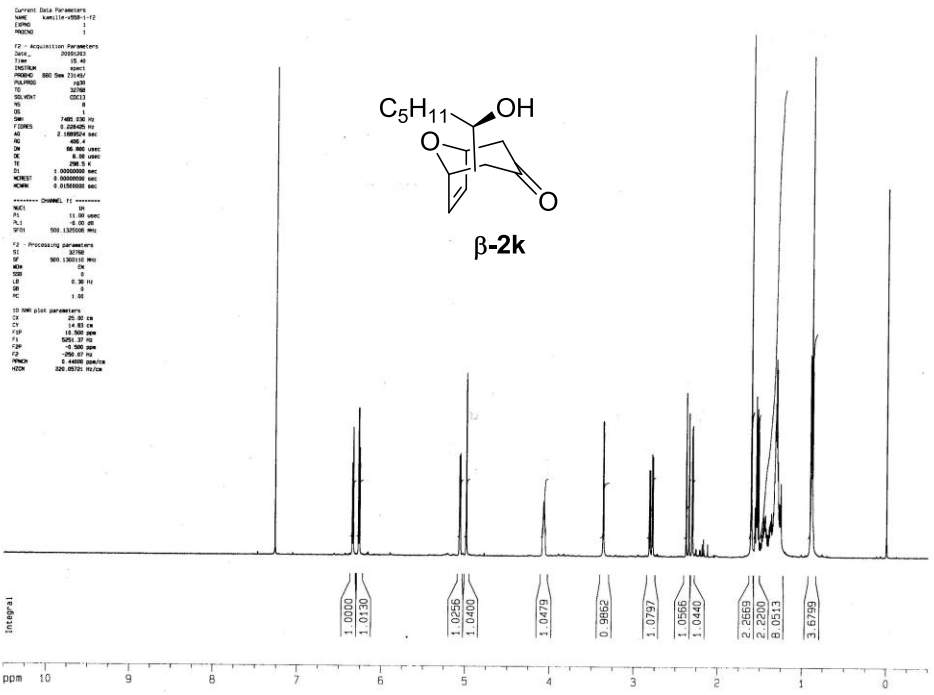

ppm
200
180
160
140
120
100
80
60
40
20
0

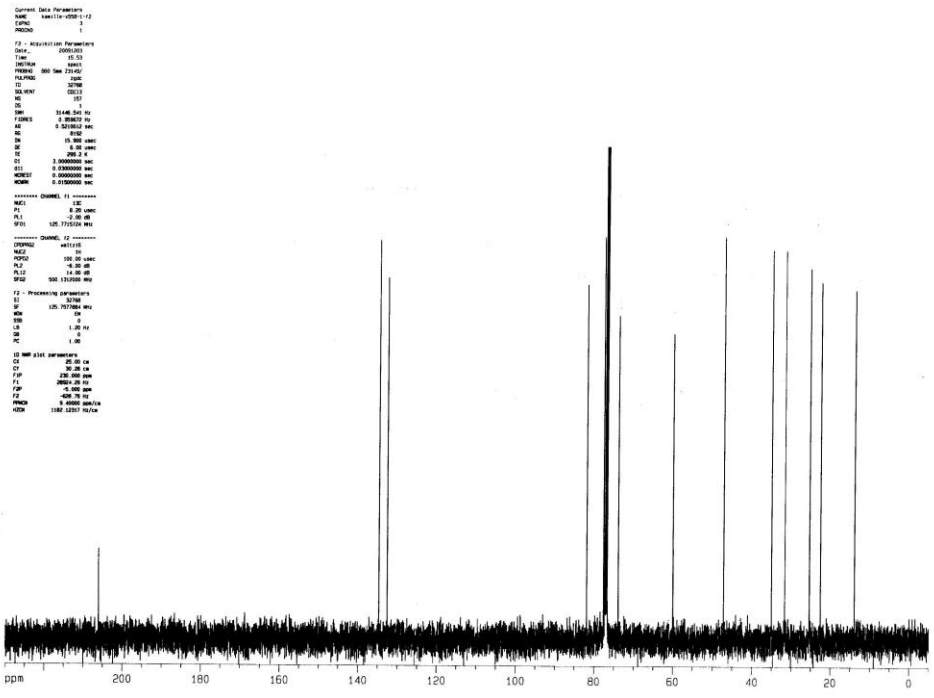

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

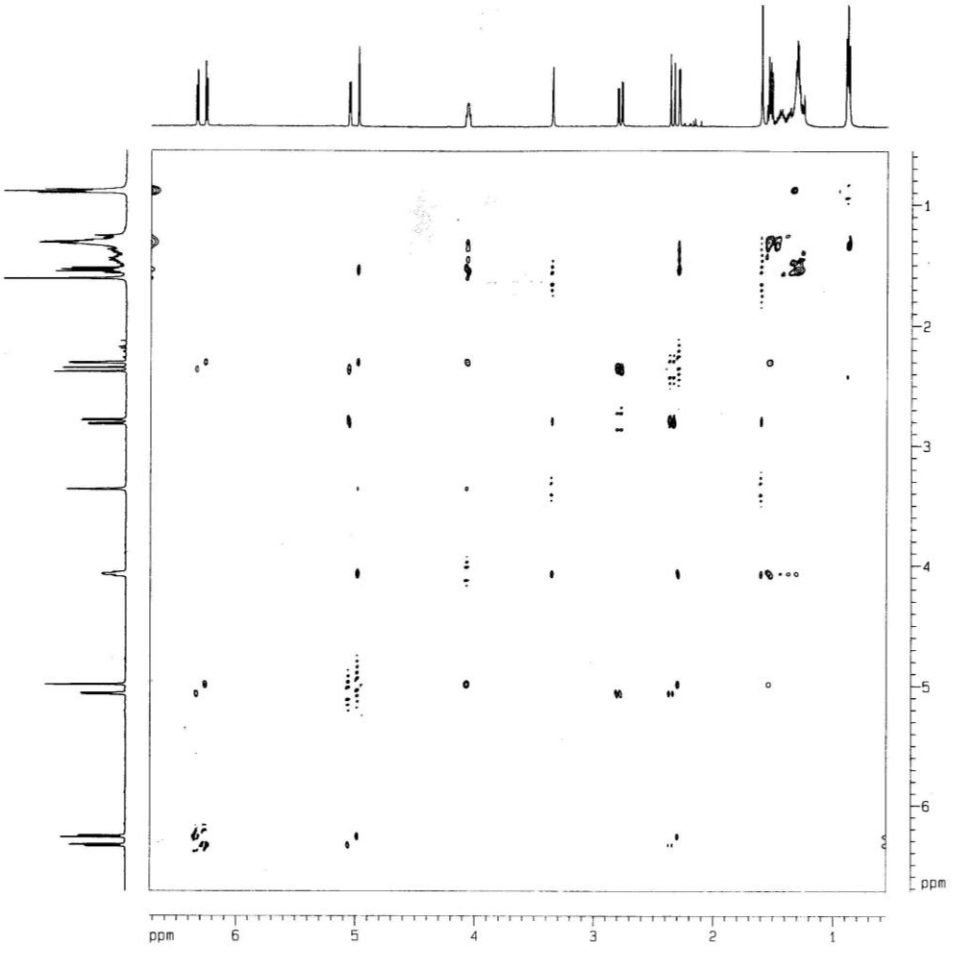

OH
O
C_5H_{11}
O
α-2k
Integral
1.0000
1.0157
1.0010
1.0324
1.0022
1.0581
1.0094
1.0744
1.0592
3.0140
6.0829
3.3800
ppm 10 9 8 7 6 5 4 3 2 1 0

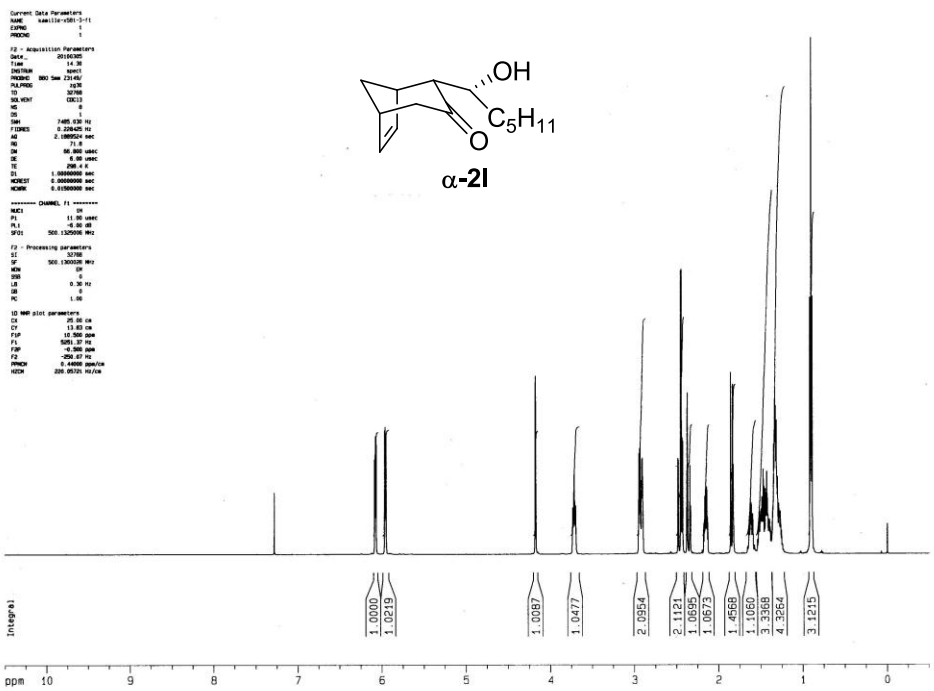

ppm 200 180 160 140 120 100 80 60 40 20 0

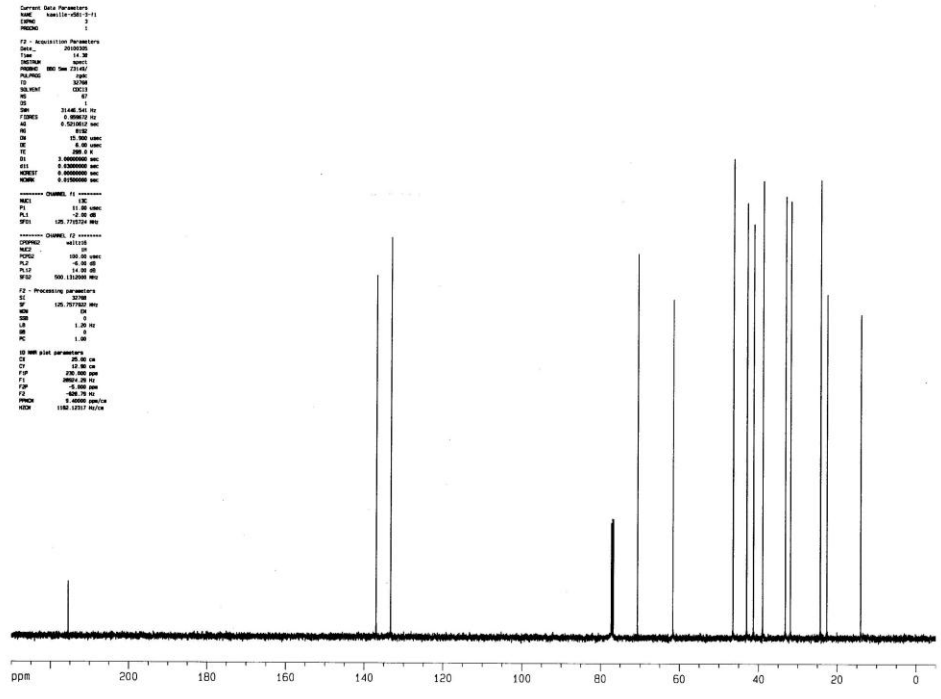

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

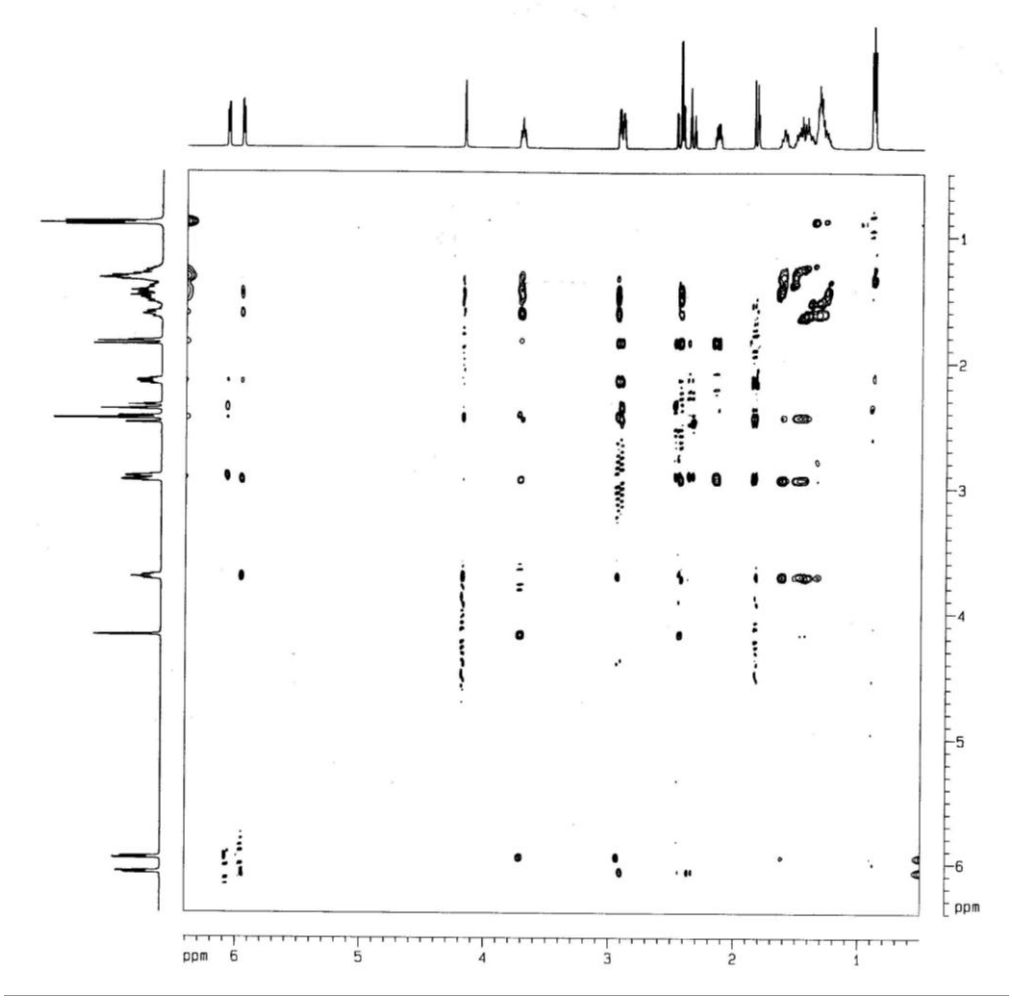

C_5H_{11}
OH
O
O
β-2k
Integral
1.0000
1.0130
1.0256
1.0400
1.0479
0.9862
1.0797
1.0566
1.0440
2.2669
2.2200
8.0513
3.6798
ppm 10 9 8 7 6 5 4 3 2 1 0

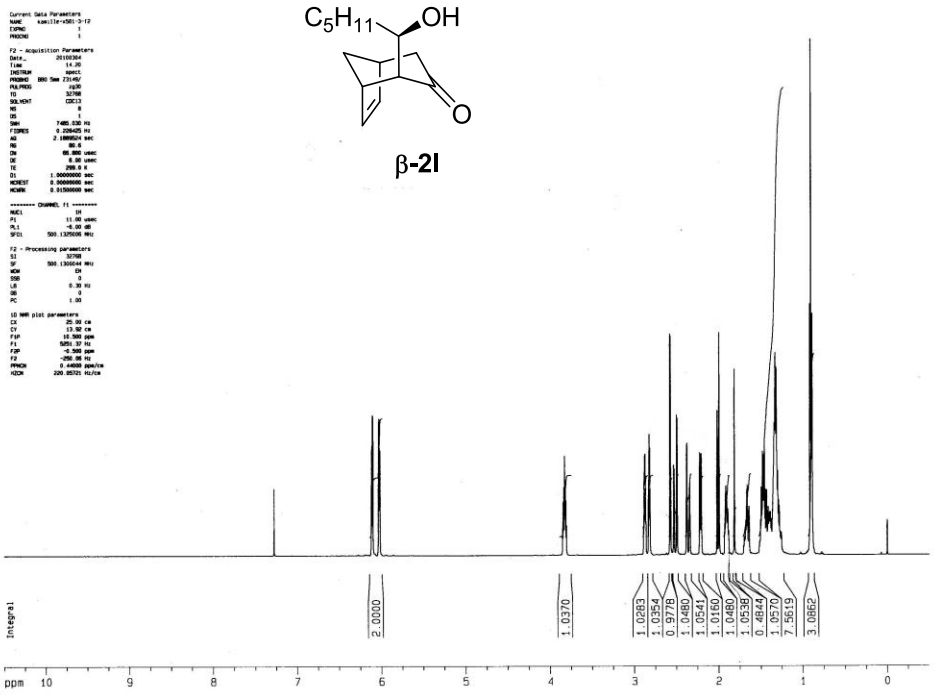

ppm 200 180 160 140 120 100 80 60 40 20 0

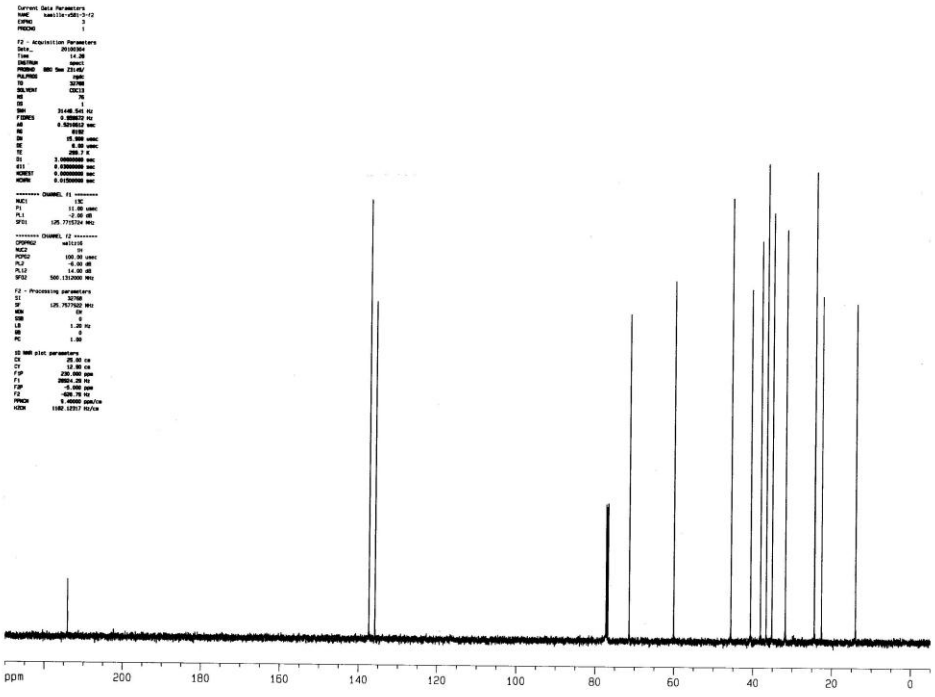

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

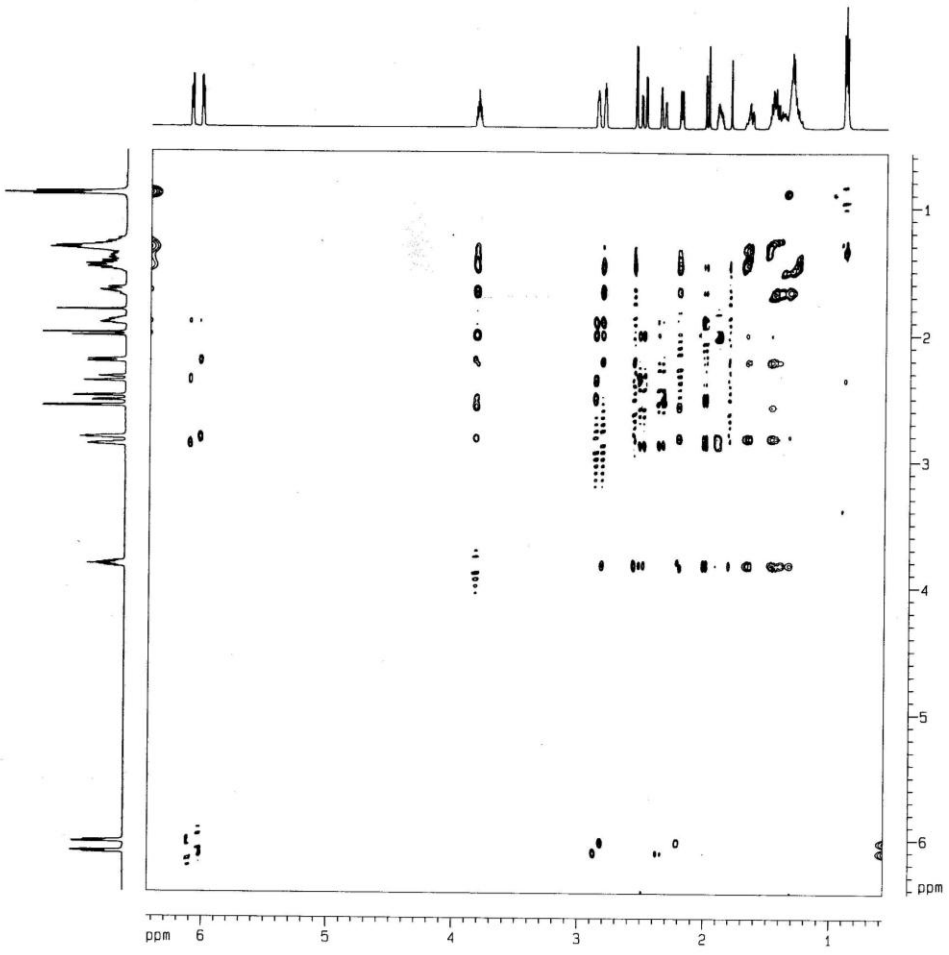

OH
C_5H_{11}
O
α-2l
Integral
1.0000
1.0219
1.0087
1.0477
2.0954
2.1121
1.0695
1.0673
1.4568
1.1060
3.3368
4.3264
3.1215
ppm 10 9 8 7 6 5 4 3 2 1 0

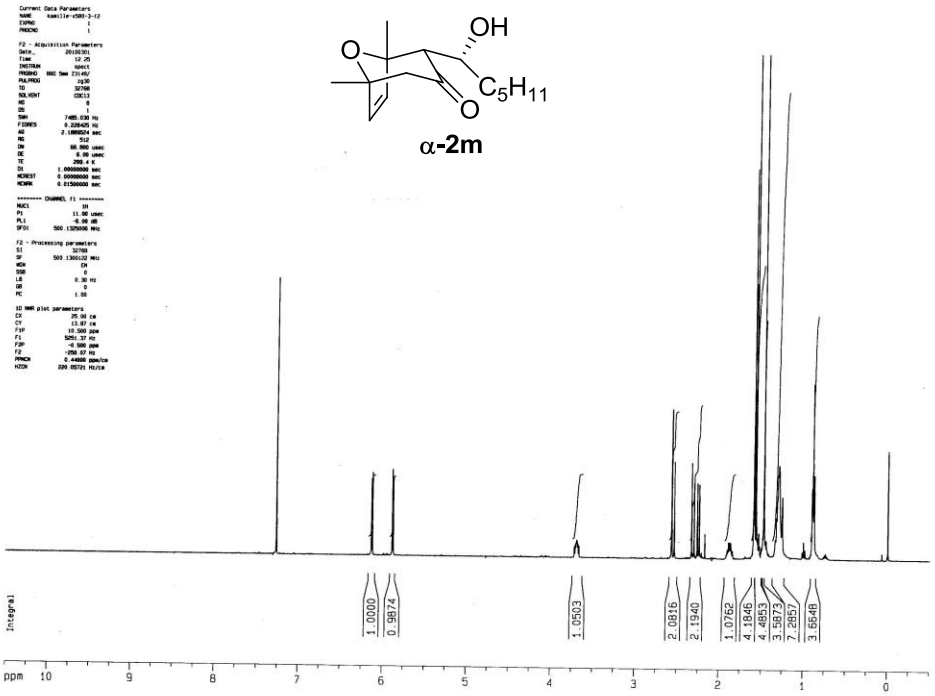

ppm 200 180 160 140 120 100 80 60 40 20 0

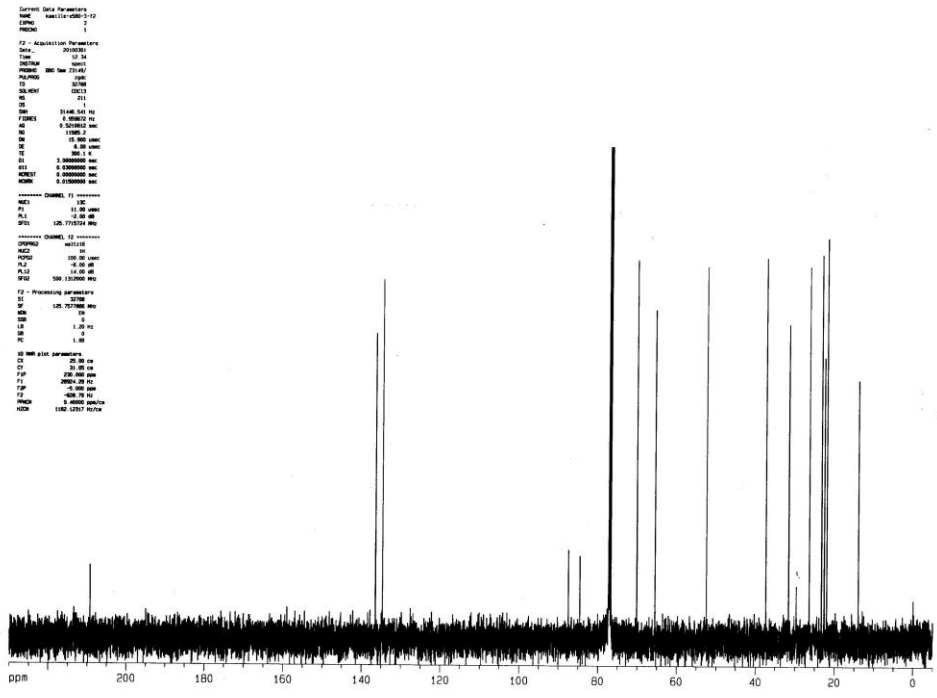

ppm 6
5
4
3
2
1
1
2
3
4
5
6
ppm

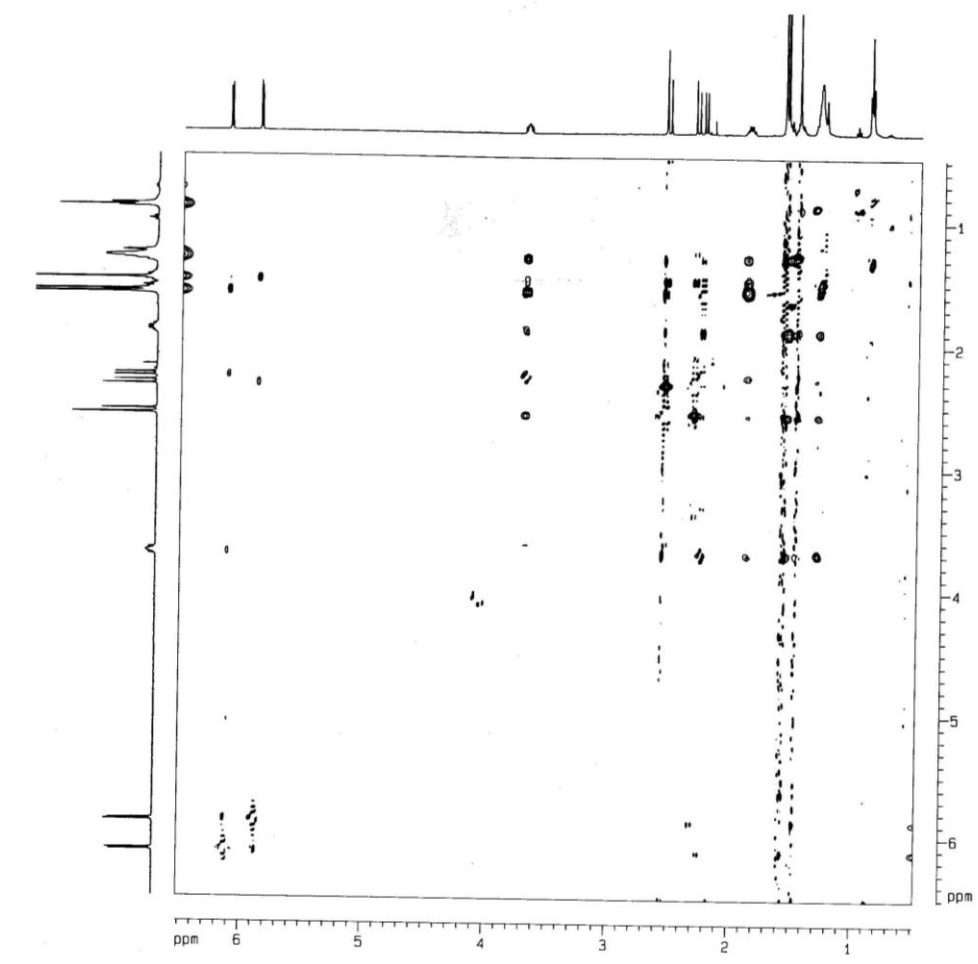

C_5H_{11}
OH
O
β-2l
Integral
2.0000
1.0370
1.0283
1.0354
0.9778
1.0480
1.0541
1.0160
1.0480
1.0538
0.4844
1.0570
7.5619
3.0862
ppm
10
9
8
7
6
5
4
3
2
1
0

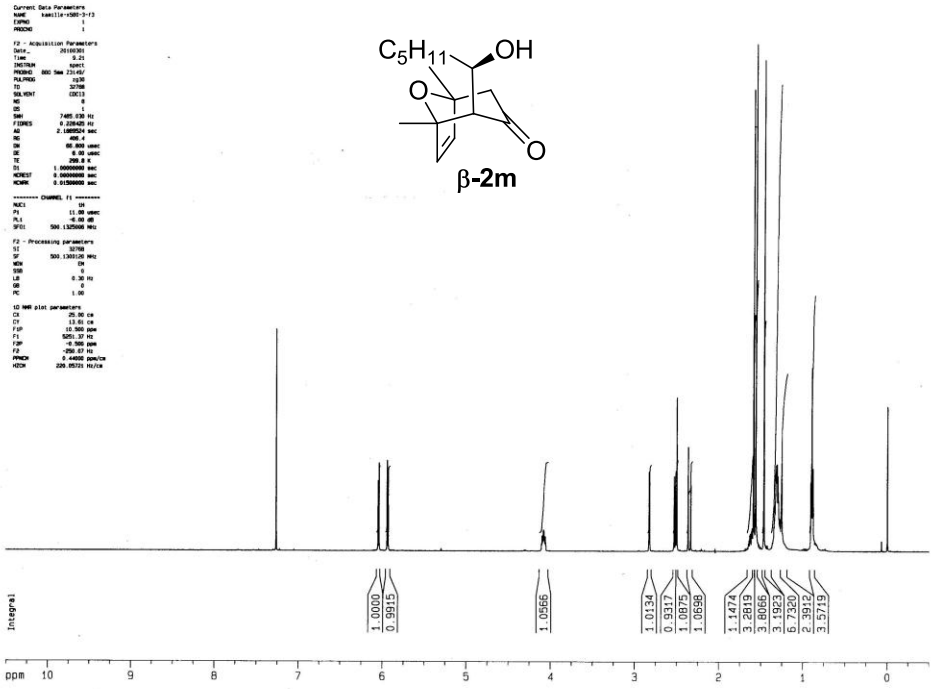

ppm
200
180
160
140
120
100
80
60
40
20
0

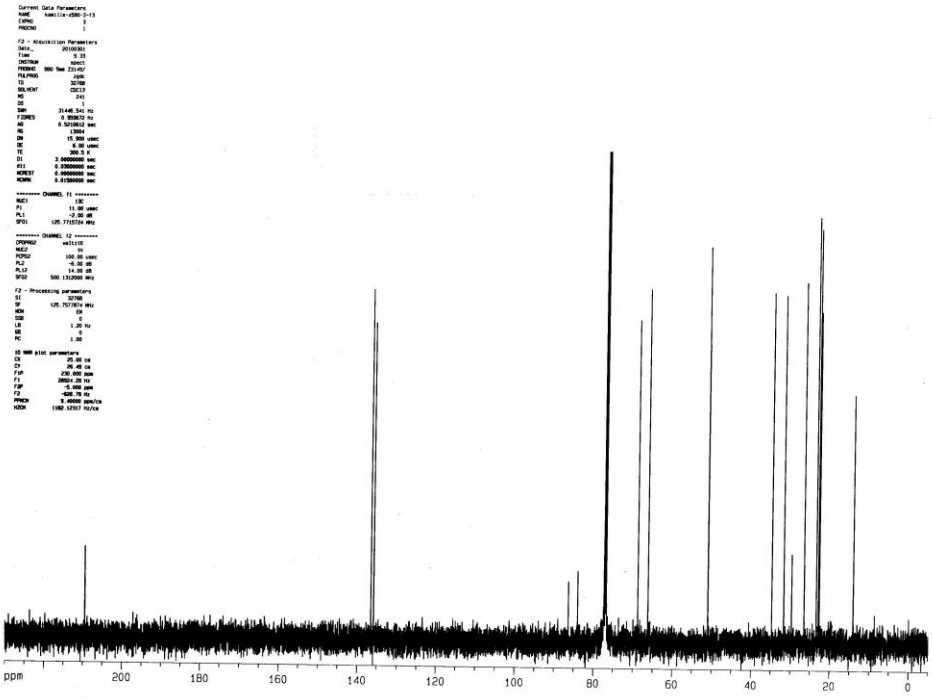

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

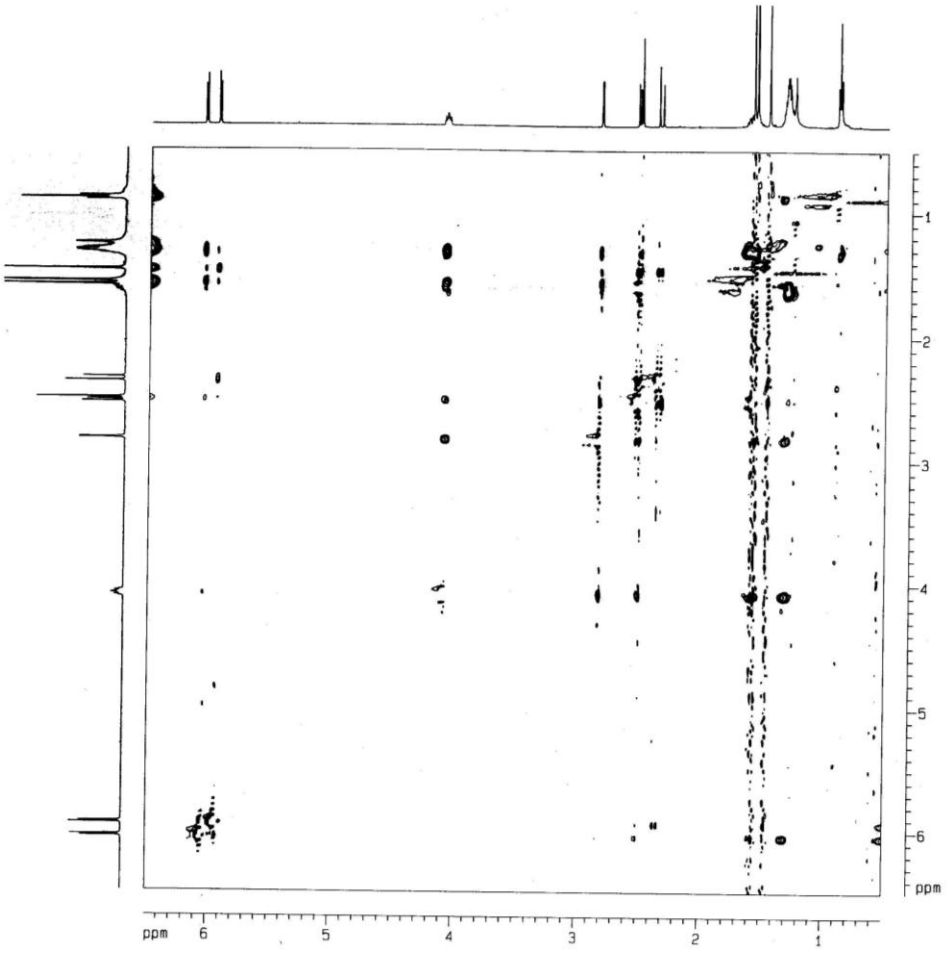

OH
O
O
C_5H_{11}
α-2m
Integral
1.0000
0.9874
1.0503
2.0816
2.1940
1.0762
4.1846
4.4853
3.5973
7.2857
3.6548
ppm
10
9
8
7
6
5
4
3
2
1
0

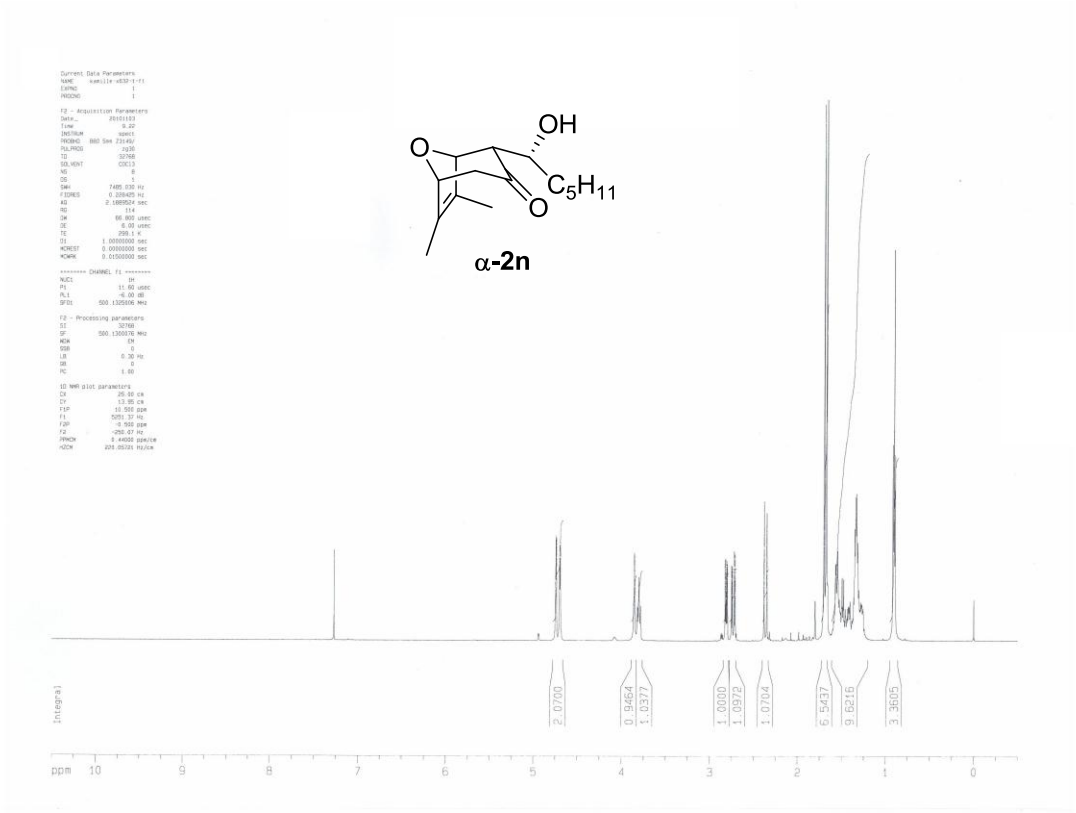

ppm
200
180
160
140
120
100
80
60
40
20
0

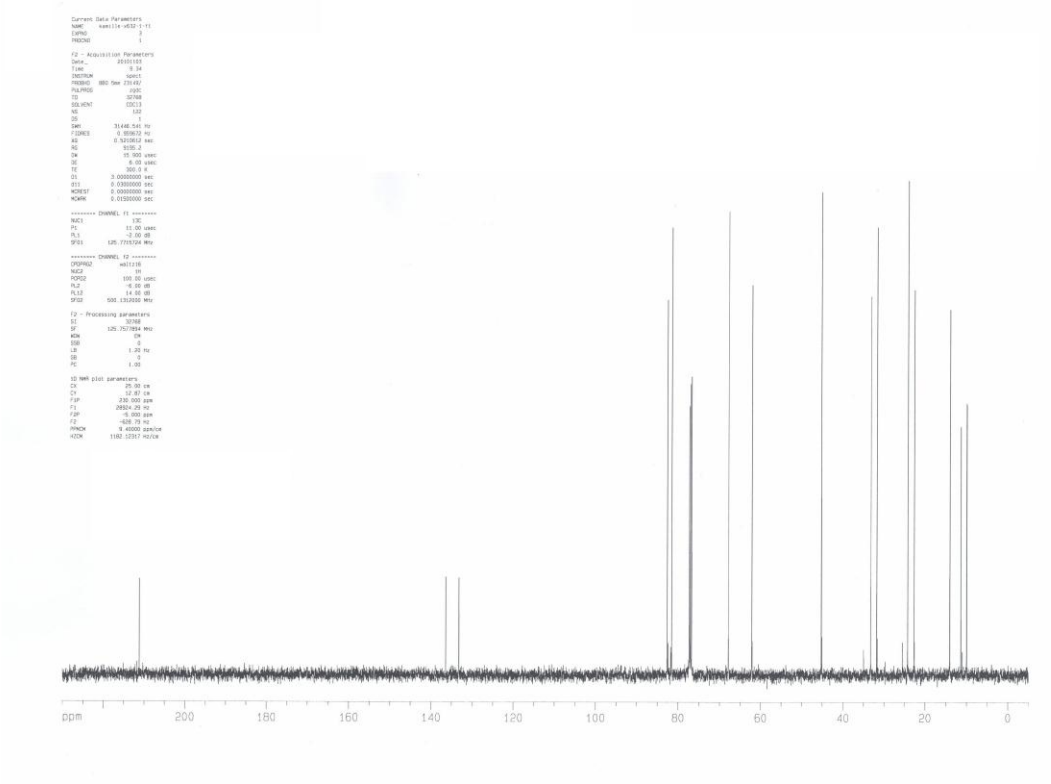

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

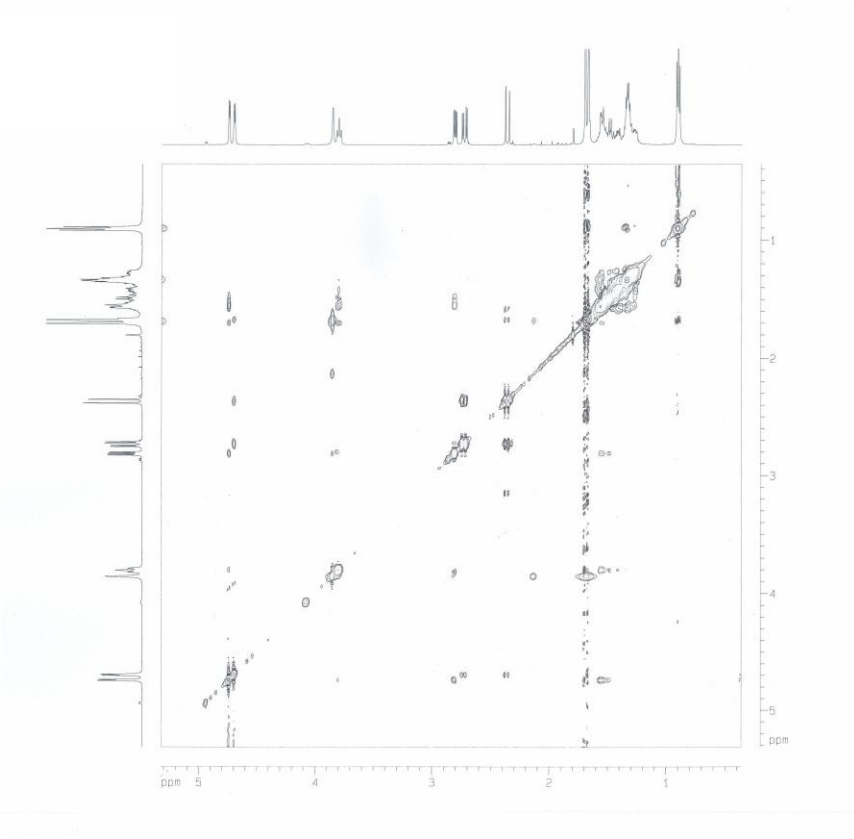

C5H11
OH
O
O
β-2m
Integral
1.0000
0.9915
1.0566
1.0134
0.9317
1.0875
1.0698
1.1474
3.2819
3.8066
3.1923
6.7320
2.3912
3.5719
ppm 10 9 8 7 6 5 4 3 2 1 0

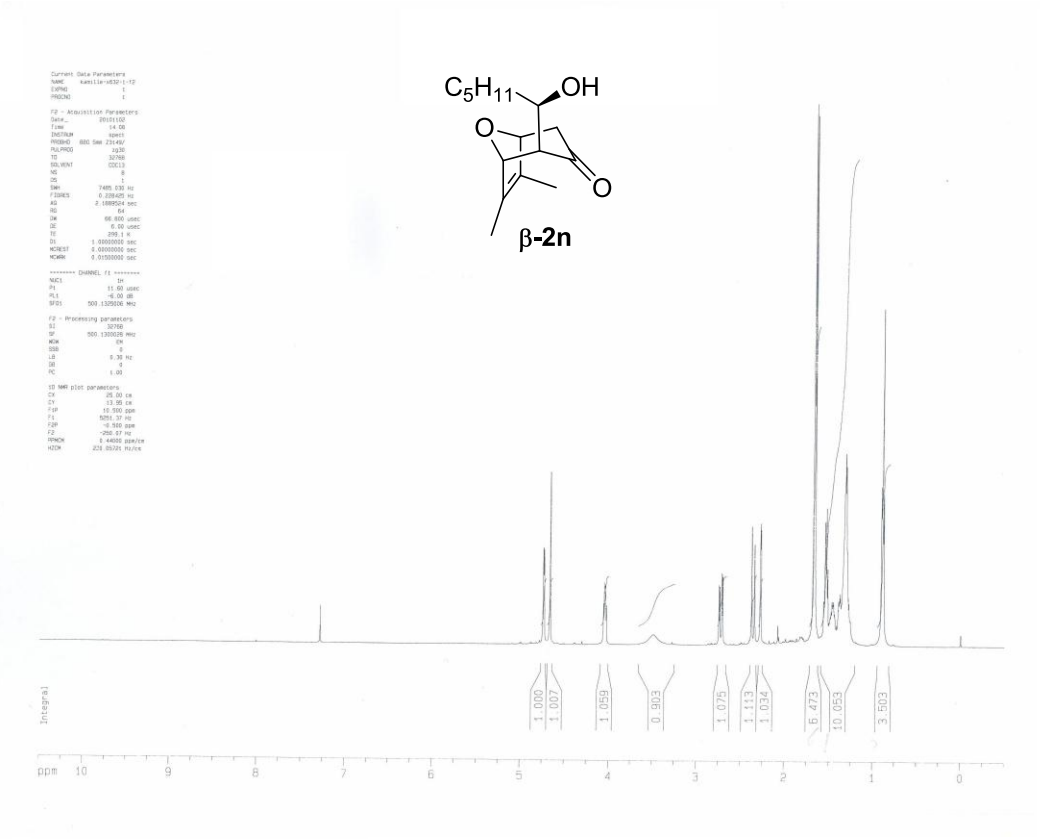

ppm 200 180 160 140 120 100 80 60 40 20 0

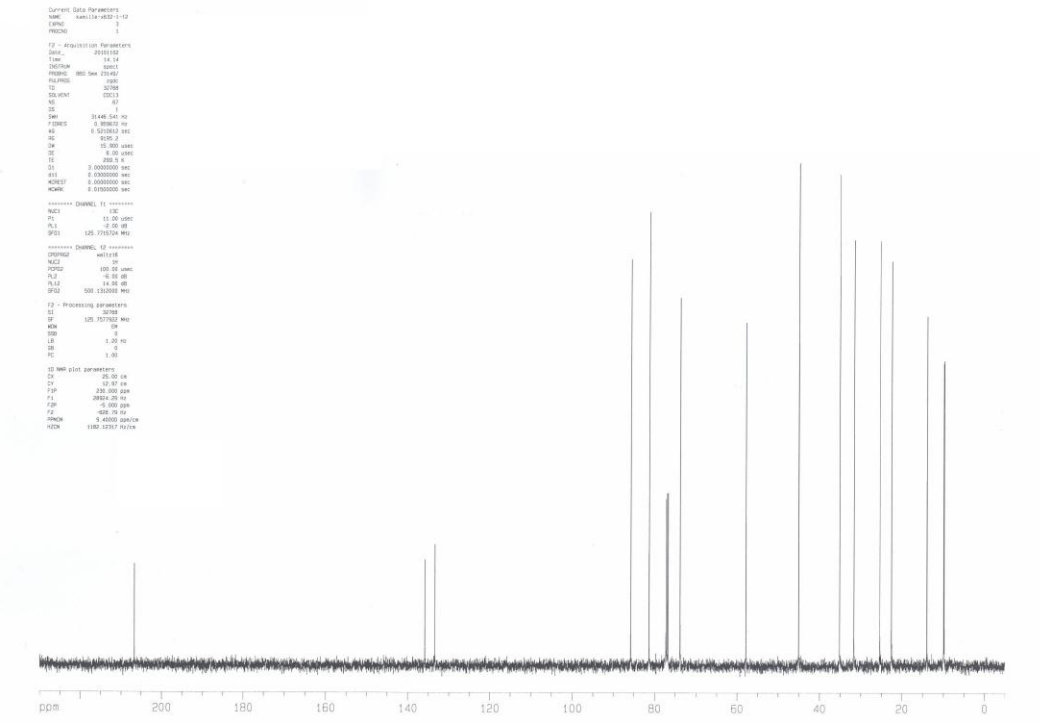

ppm
6
5
4
3
2
1
ppm

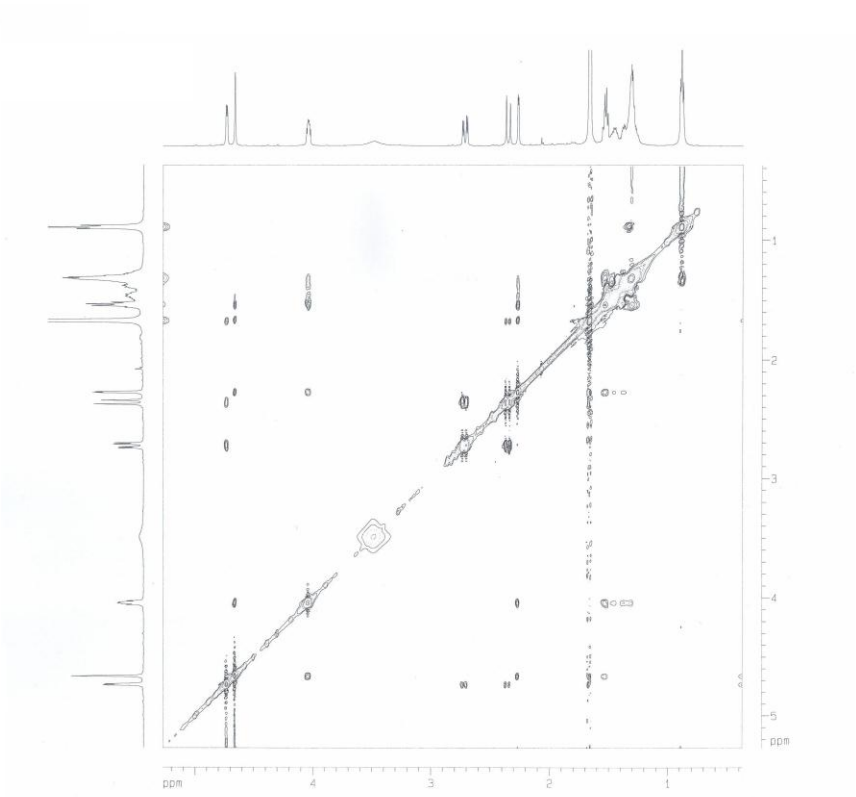

OH
O
O
C_5H_{11}
α-2n
Integral
2.0700
0.9464
1.0377
1.0000
1.0972
1.0704
6.5437
9.6216
3.3605
ppm 10 9 8 7 6 5 4 3 2 1 0

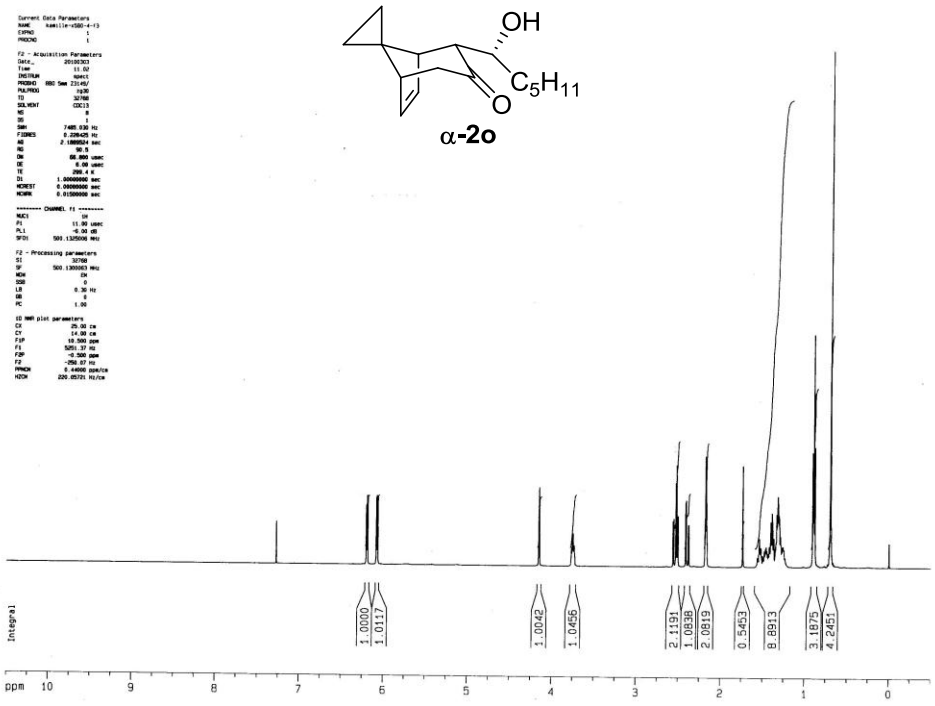

ppm 200 180 160 140 120 100 80 60 40 20 0

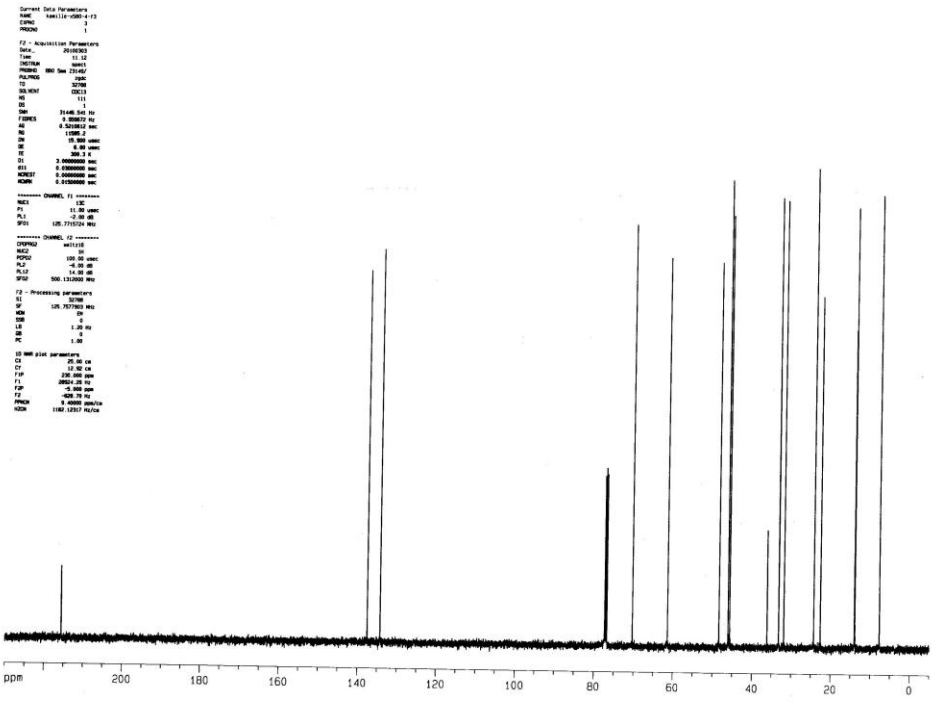

ppm
5
4
3
2
1
ppm

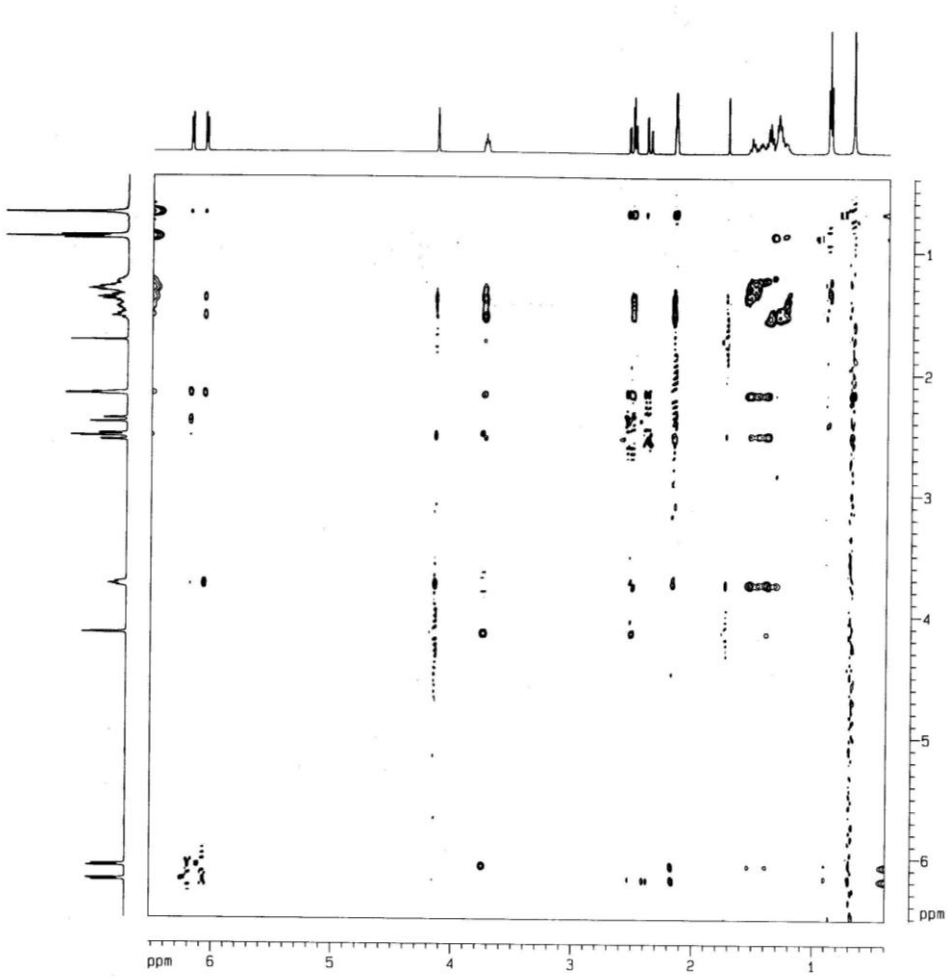

C5H11
OH
O
O
β-2n
Integral
1.000
1.007
1.059
0.903
1.075
1.113
1.034
6.473
10.053
3.503
ppm

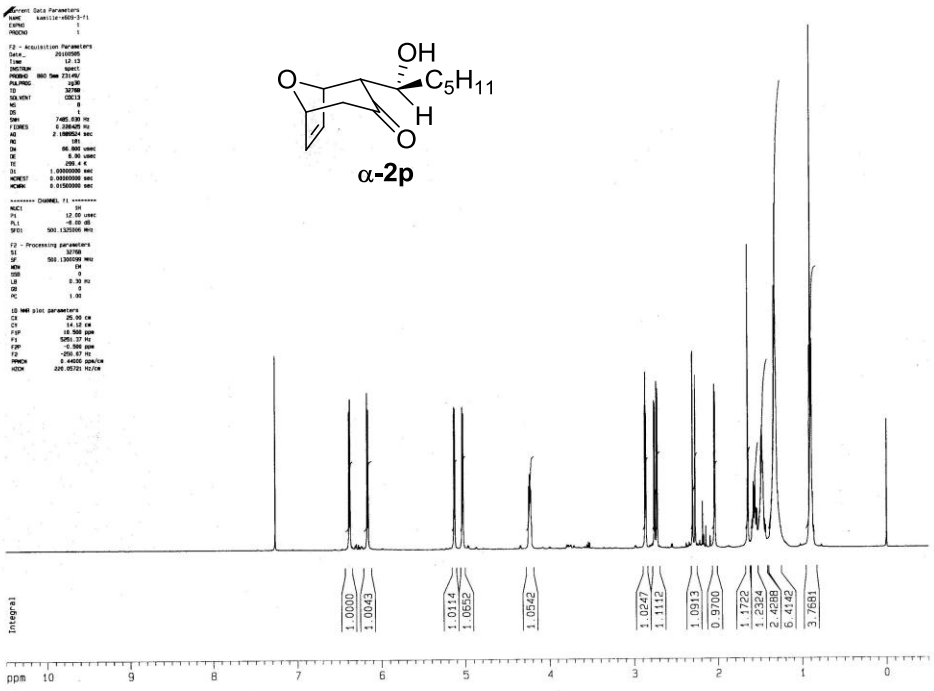

ppm

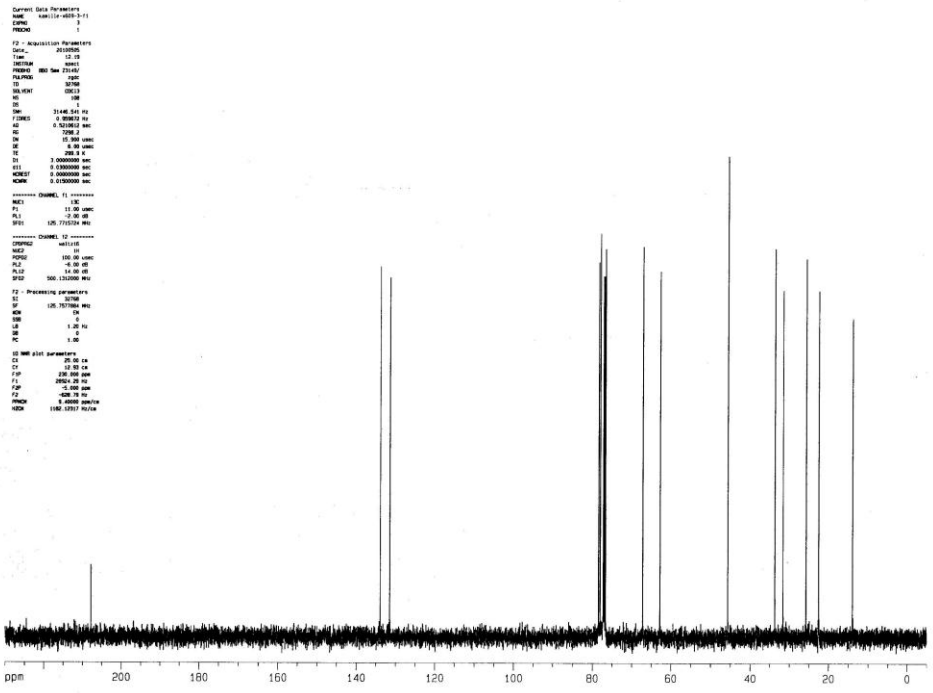

ppm
4
3
2
1
ppm

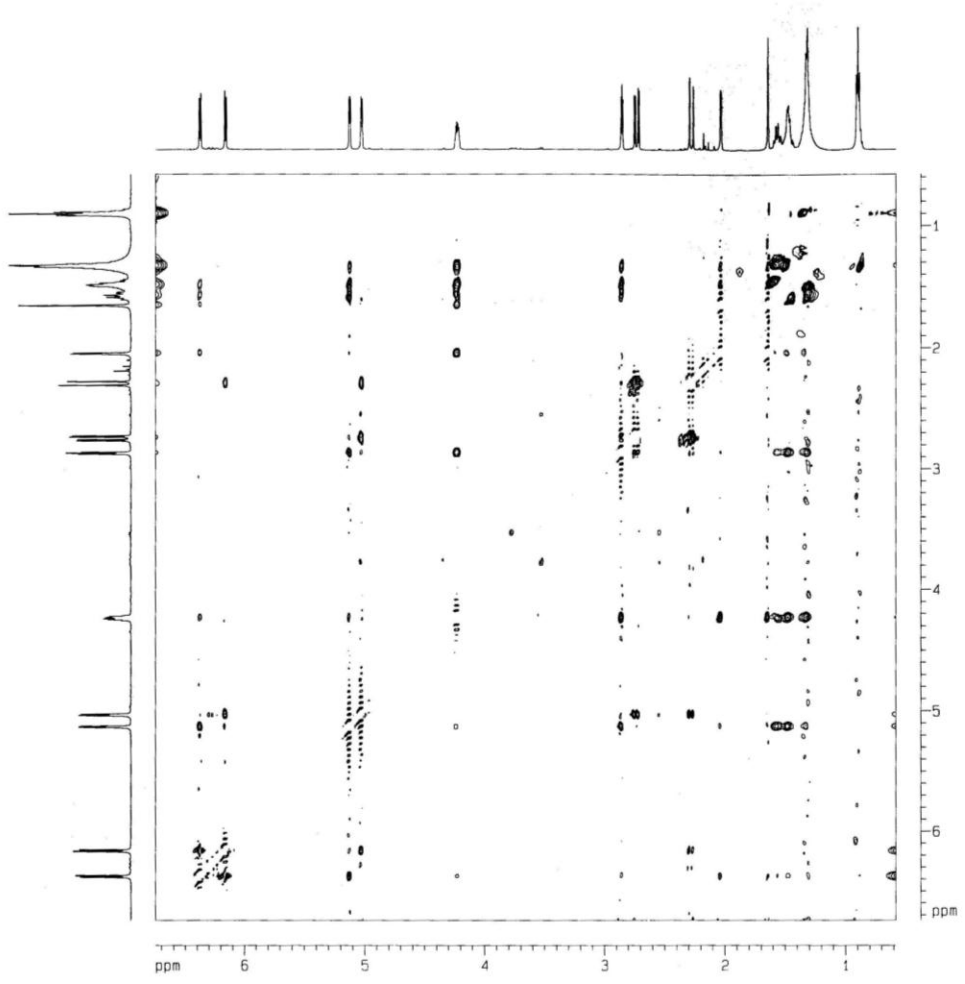

OH
C_5H_{11}
O
α-2o
Integral
1.0000
1.0117
1.0042
1.0456
2.1191
1.0838
2.0819
0.5453
8.8913
3.1875
4.2451
ppm

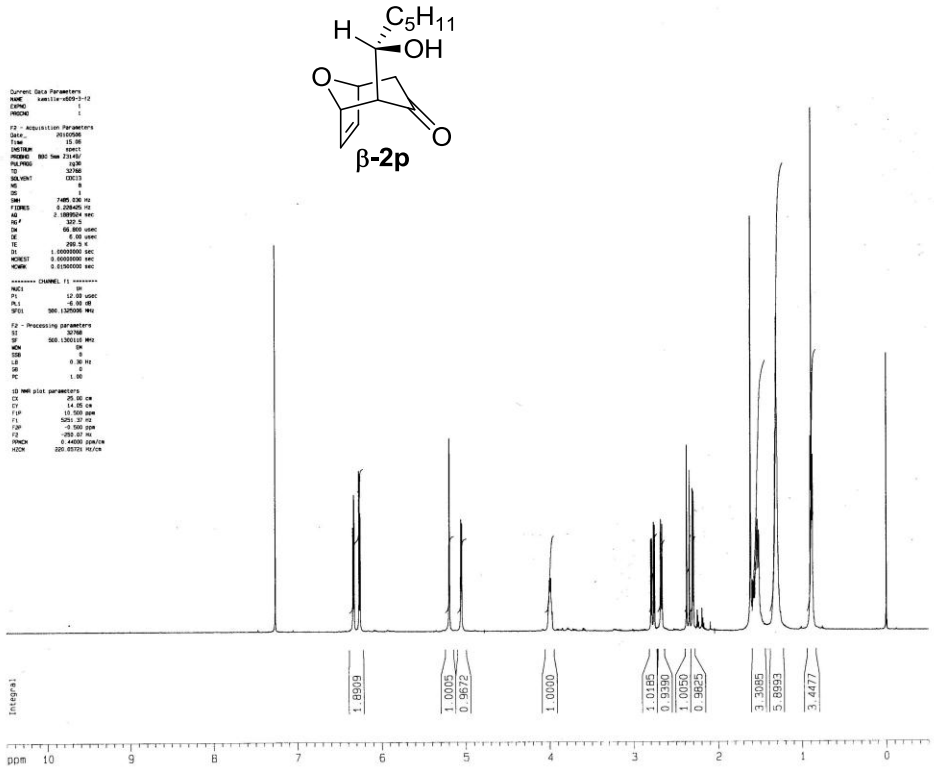

ppm

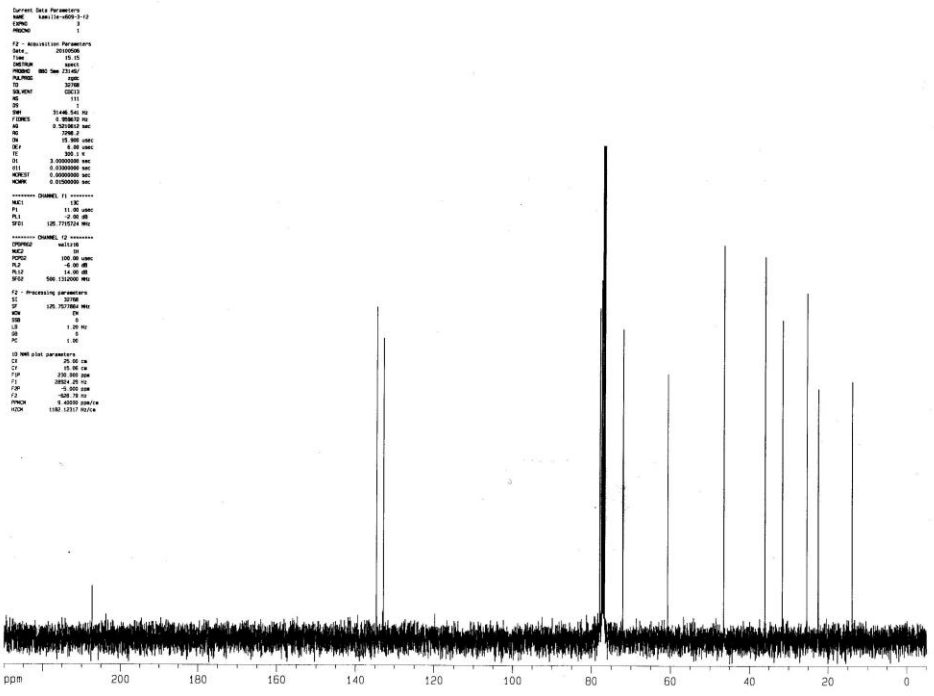

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

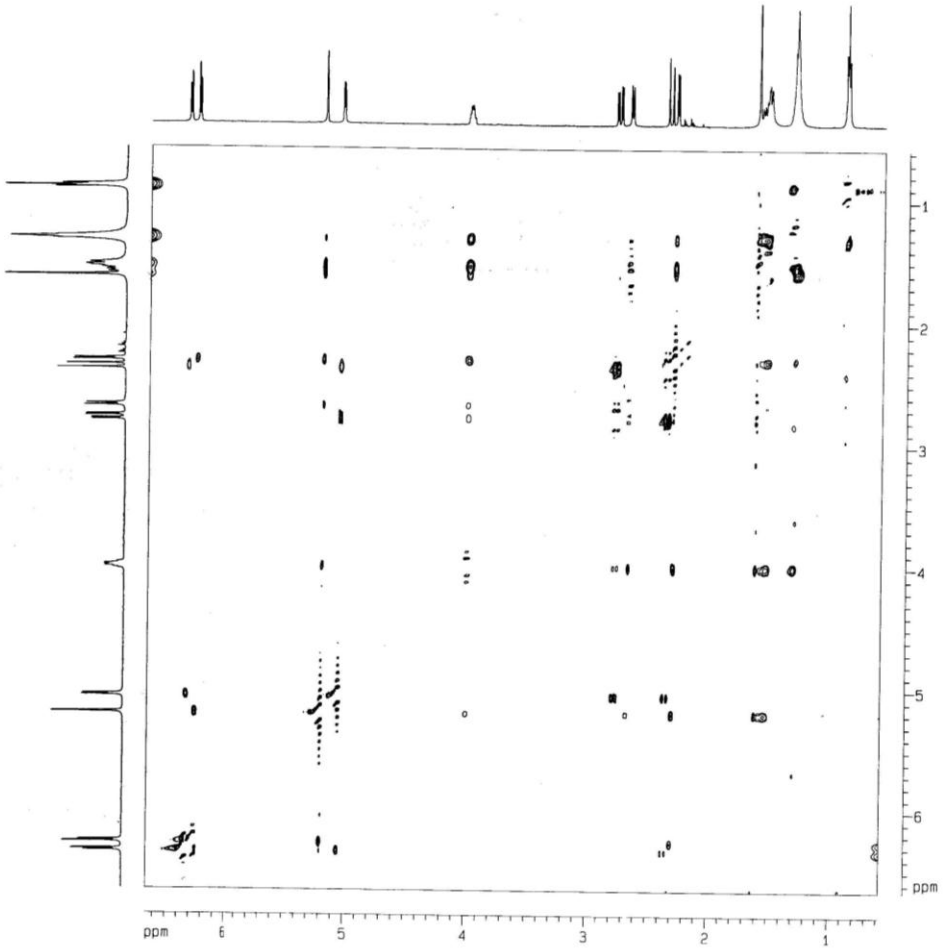

OH
C_5H_{11}
O
H
O
α-2p
Integral
1.0000
1.0043
1.0114
1.0652
1.0542
1.0247
1.1112
1.0913
0.9700
1.1722
1.2324
2.4288
6.4142
3.7681
ppm

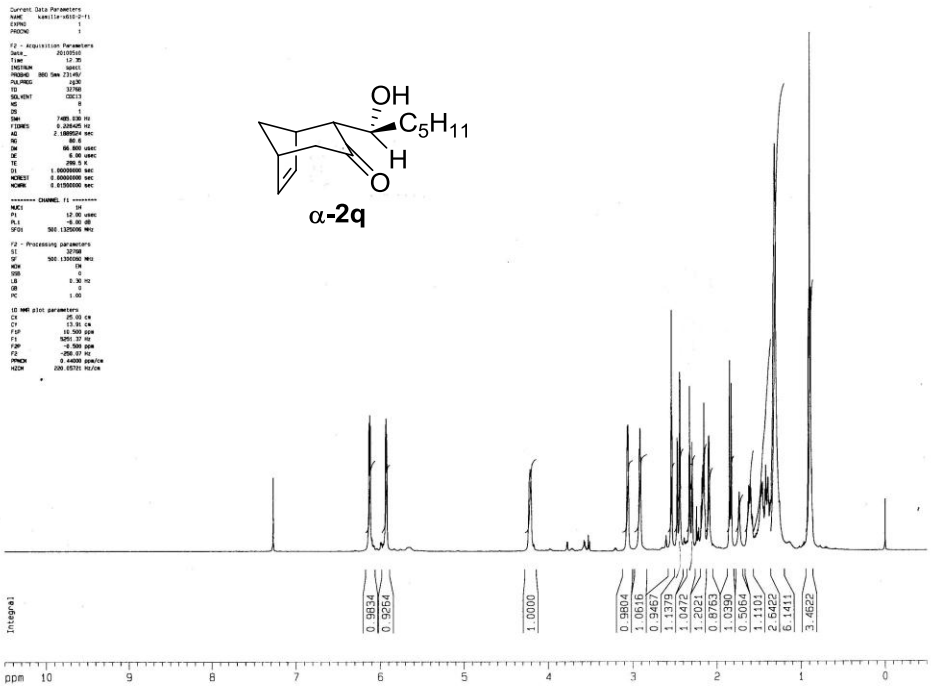

ppm

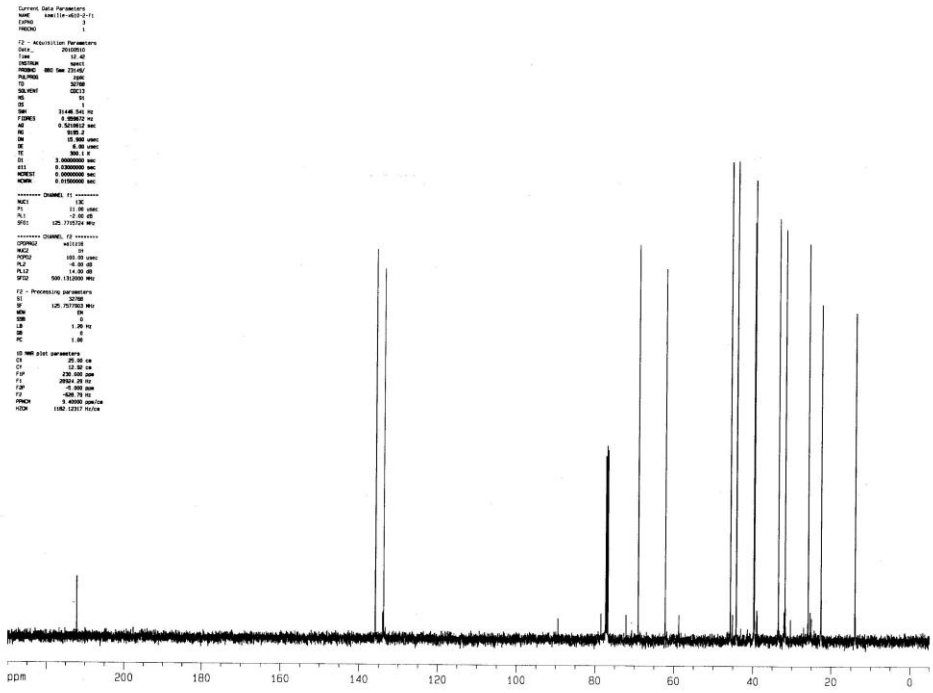

ppm
6
5
4
3
2
1
1
2
3
4
5
6
ppm

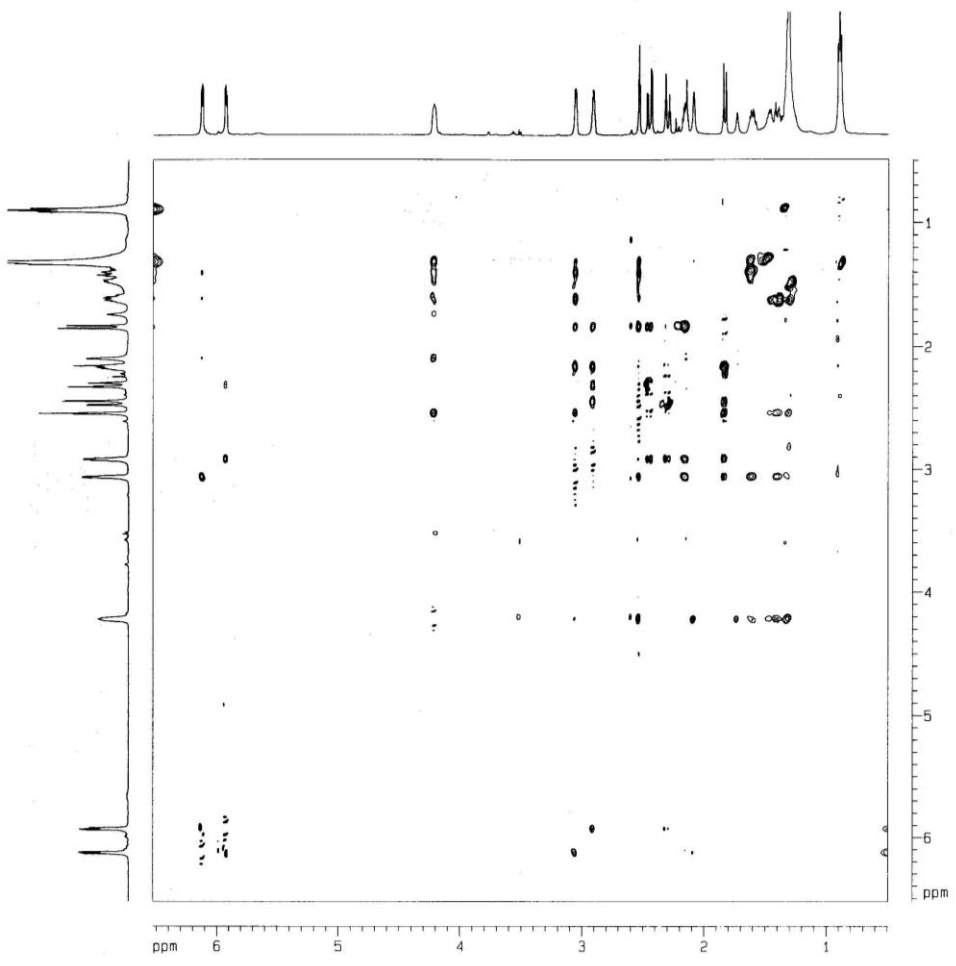

H
C_5H_{11}
OH
O
O
β-2p
1.8909
1.0005
0.9672
1.0000
1.0185
0.9390
1.0050
0.9825
3.3085
5.8993
3.4477
ppm

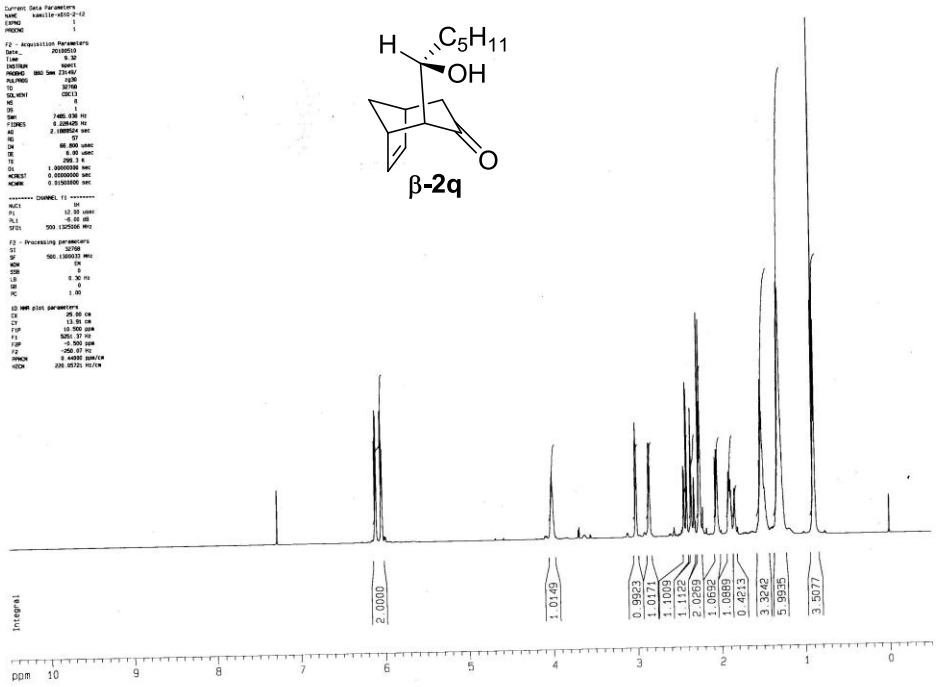

ppm

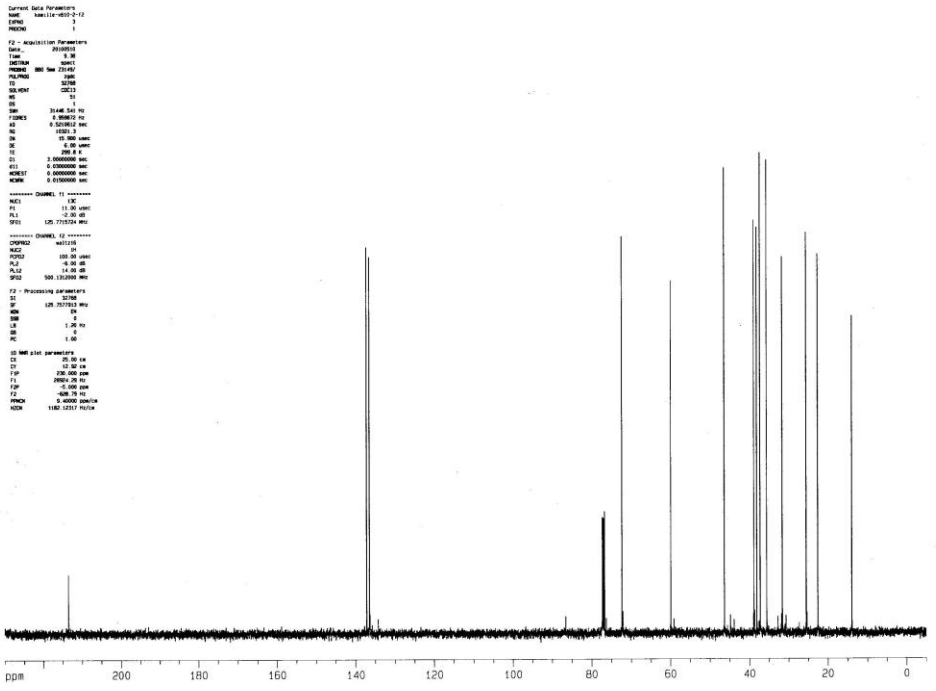

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

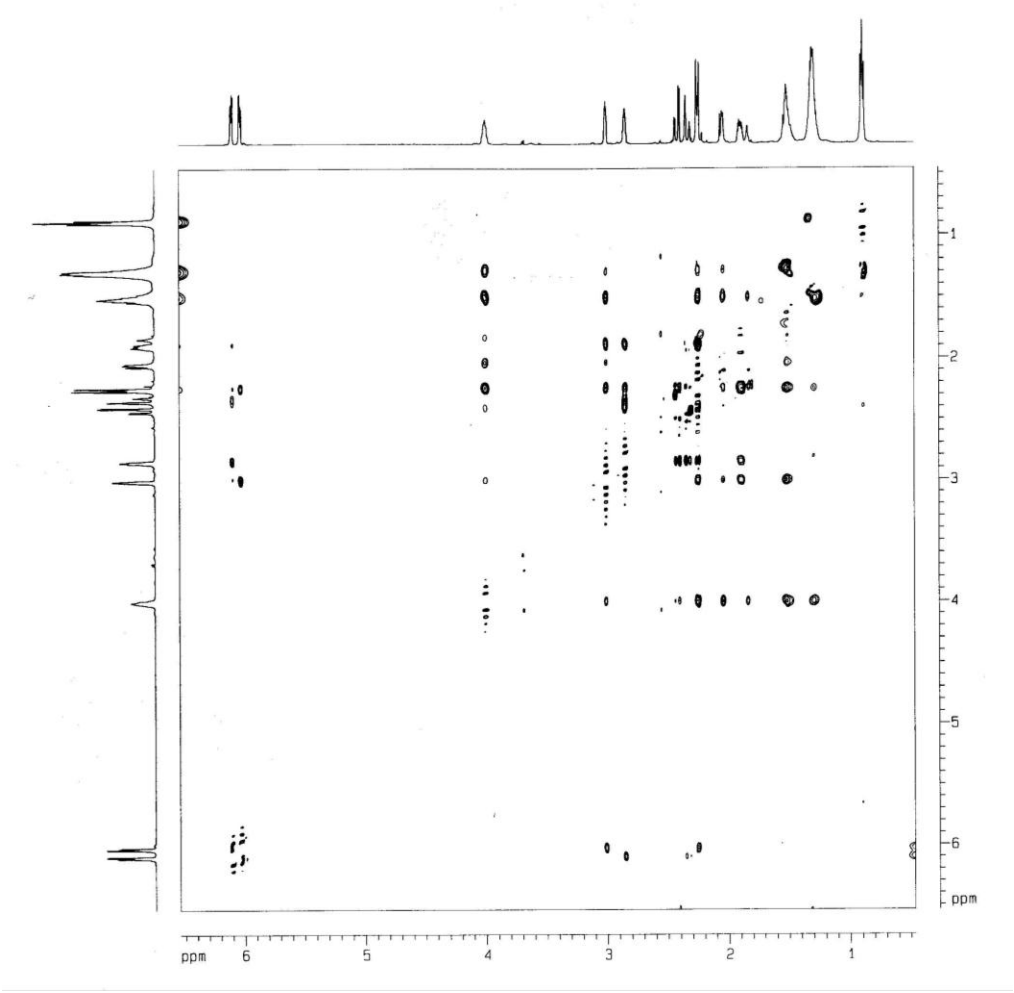

OH
C_5H_{11}
H
O
α-2q
Integral
0.9834
0.9264
1.0000
0.9804
1.0616
0.9467
1.1379
1.0472
1.2021
0.8763
1.0390
0.5064
1.1101
2.6422
6.1411
3.4622
ppm

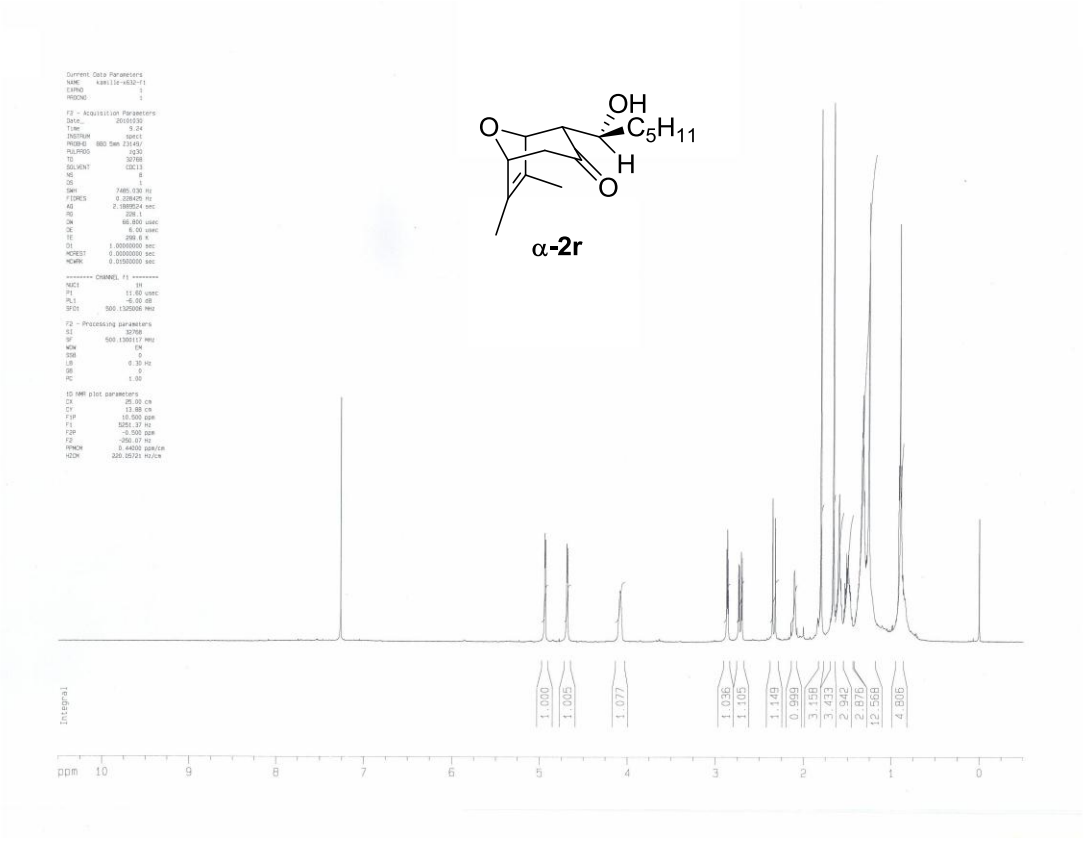

ppm

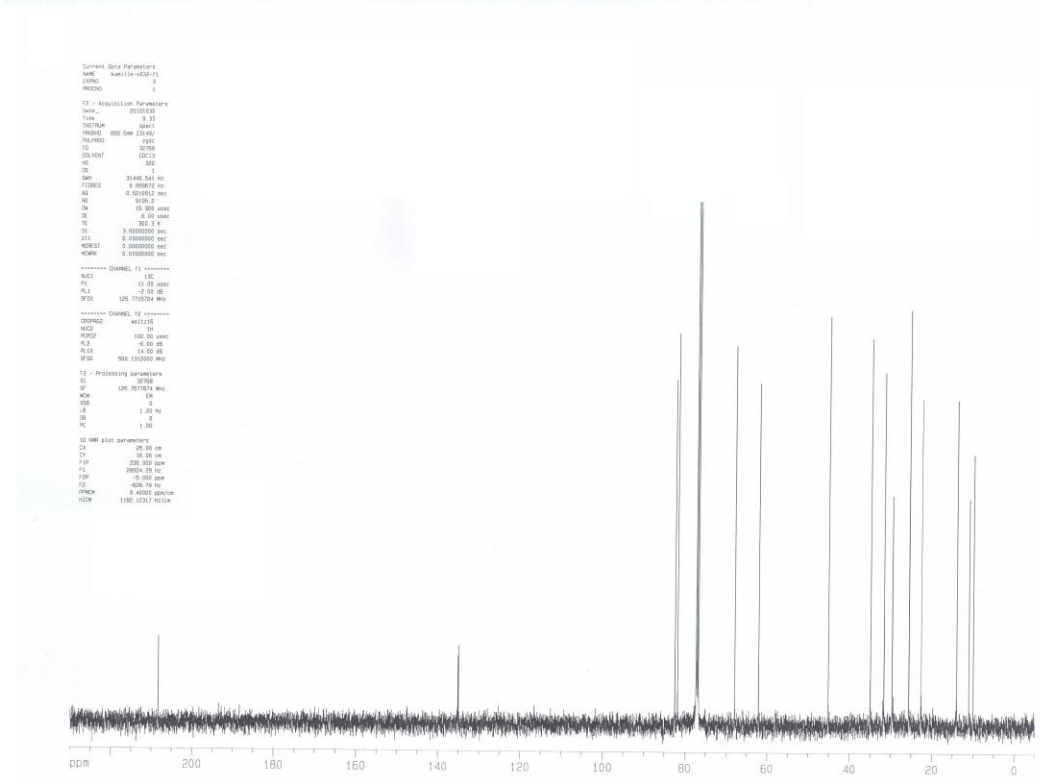

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

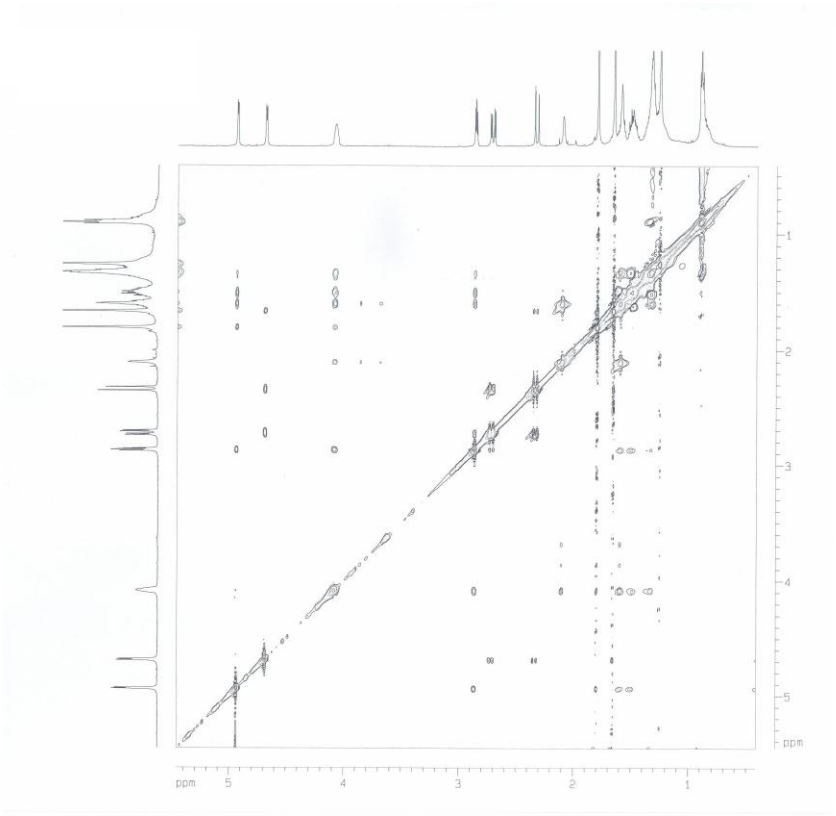

H
C5H11
OH
O
β-2q
Integral
2.0000
1.0149
0.9923
1.0171
1.1009
1.1122
2.0269
1.0652
1.0889
0.4213
3.3242
5.9935
3.5077
ppm

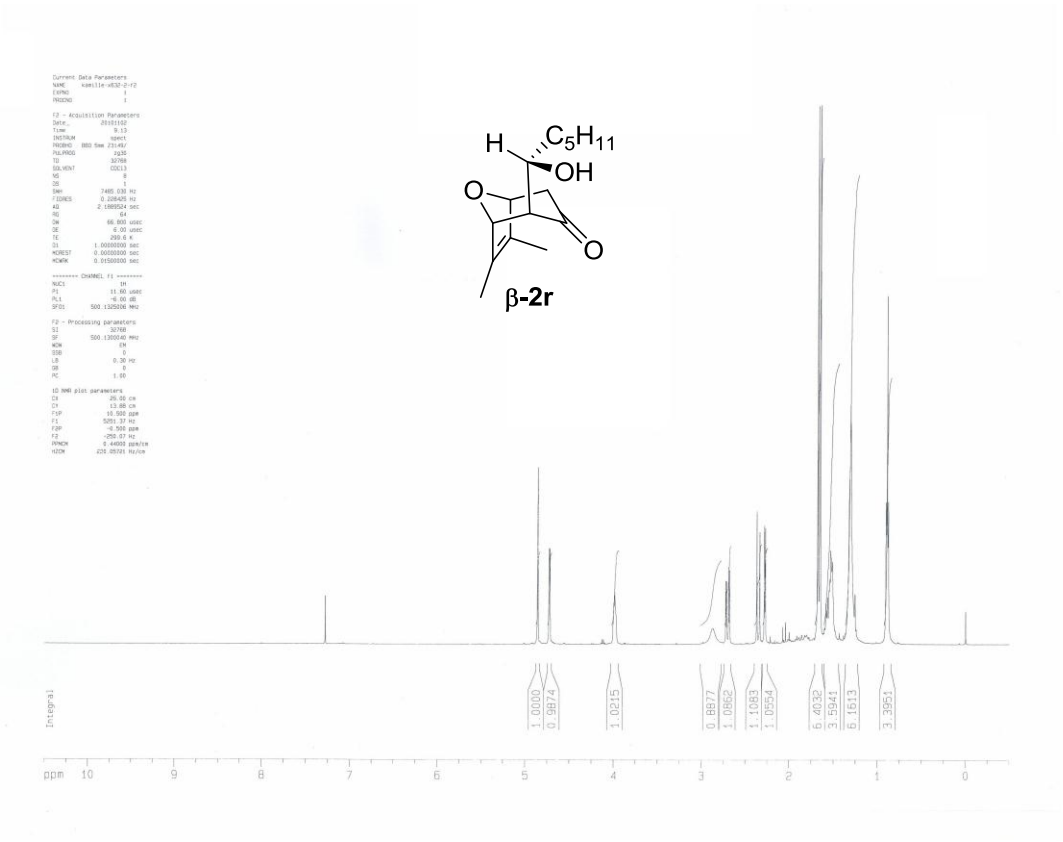

ppm

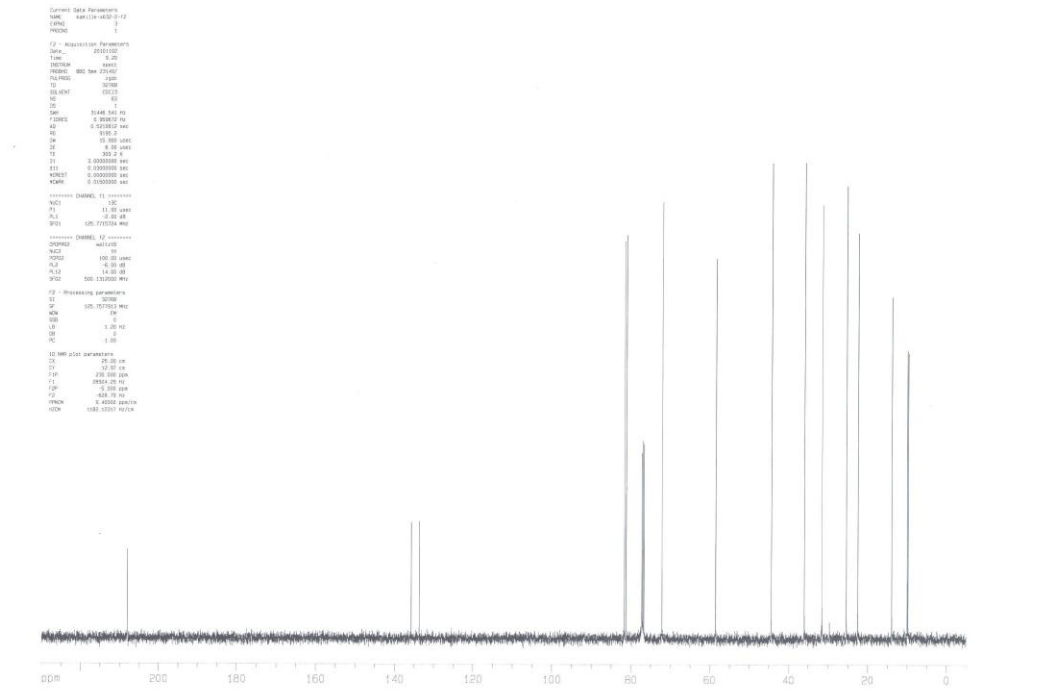

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

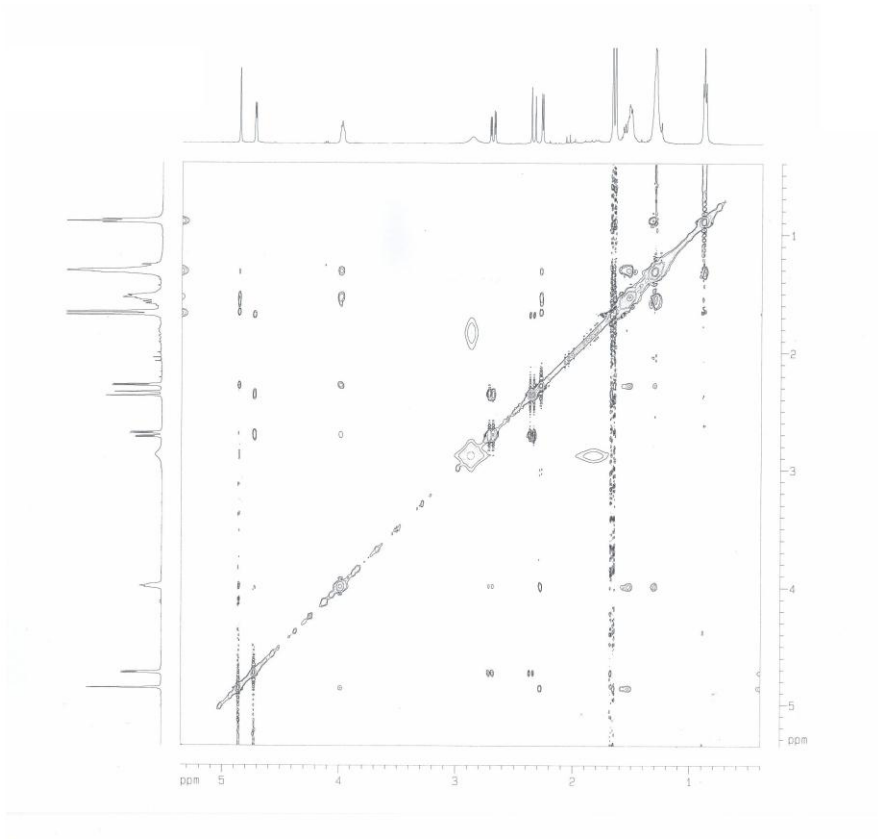

OH
C_5H_{11}
O
H
O
α-2r
Integral
1.000
1.005
1.077
1.036
1.105
1.149
0.999
3.158
3.433
2.942
2.876
12.568
4.806
ppm 10 9 8 7 6 5 4 3 2 1 0

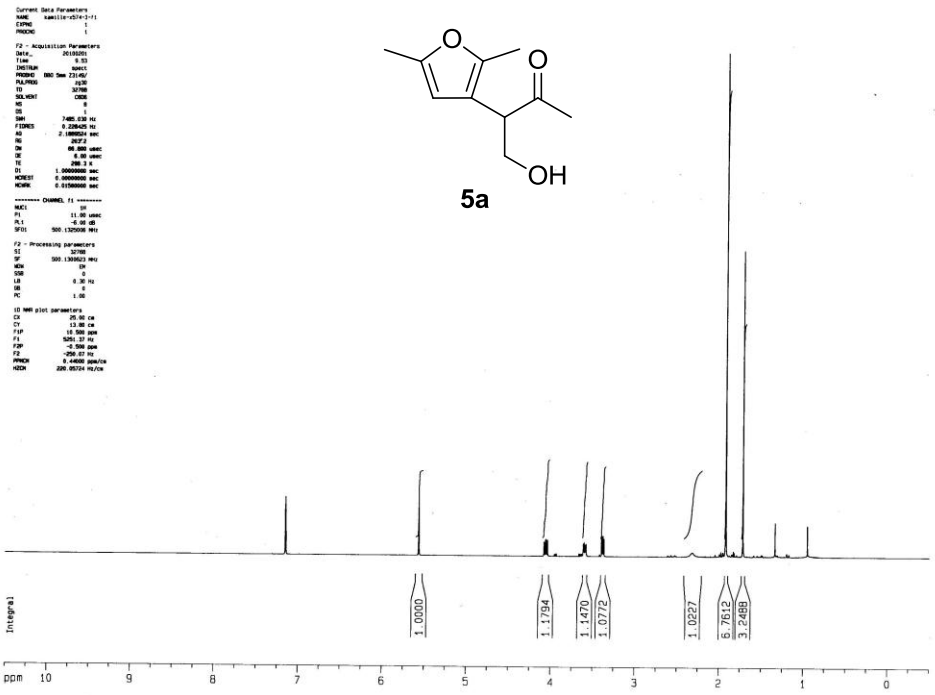

ppm 200 180 160 140 120 100 80 60 40 20 0

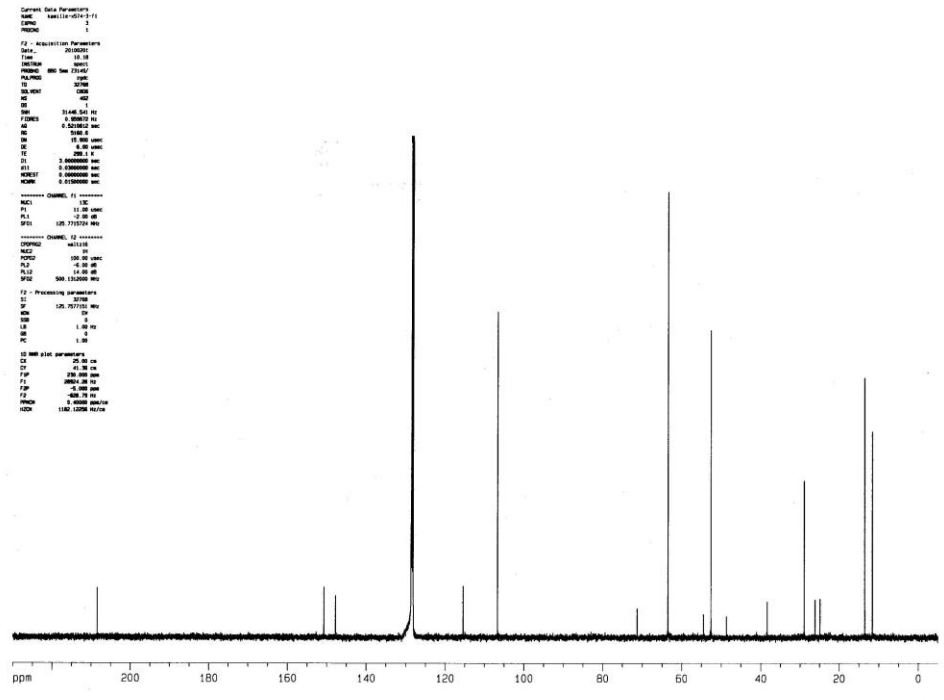

ppm
5
4
3
2
1
ppm

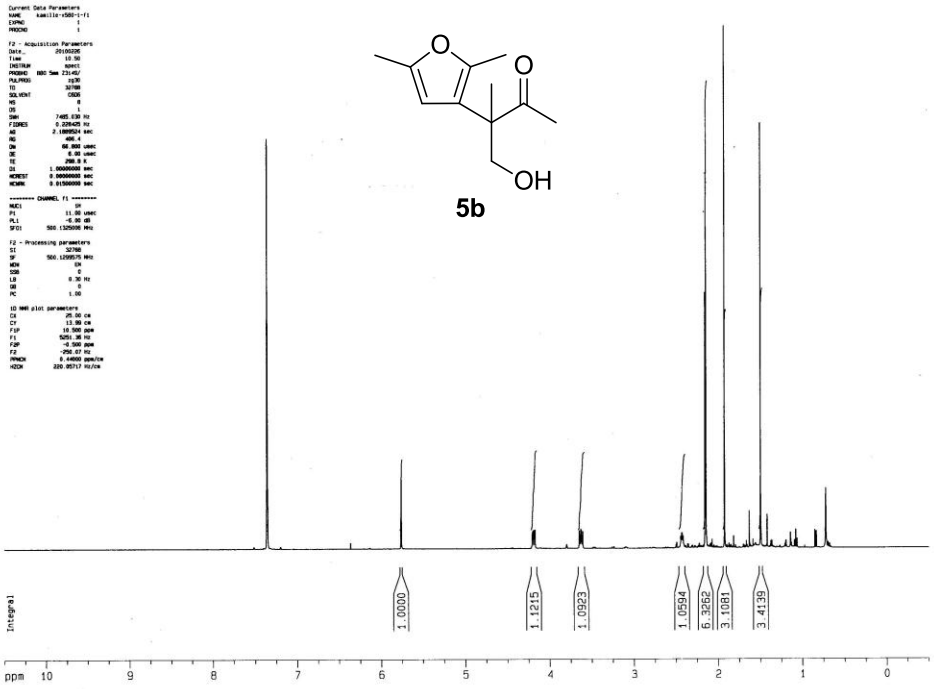

H
C_5H_{11}
OH
O
O
β-2r
Integral
1.0000
0.9874
1.0215
0.8877
1.0862
1.1083
1.0554
6.4032
3.5941
6.1613
3.3951
ppm 10 9 8 7 6 5 4 3 2 1 0

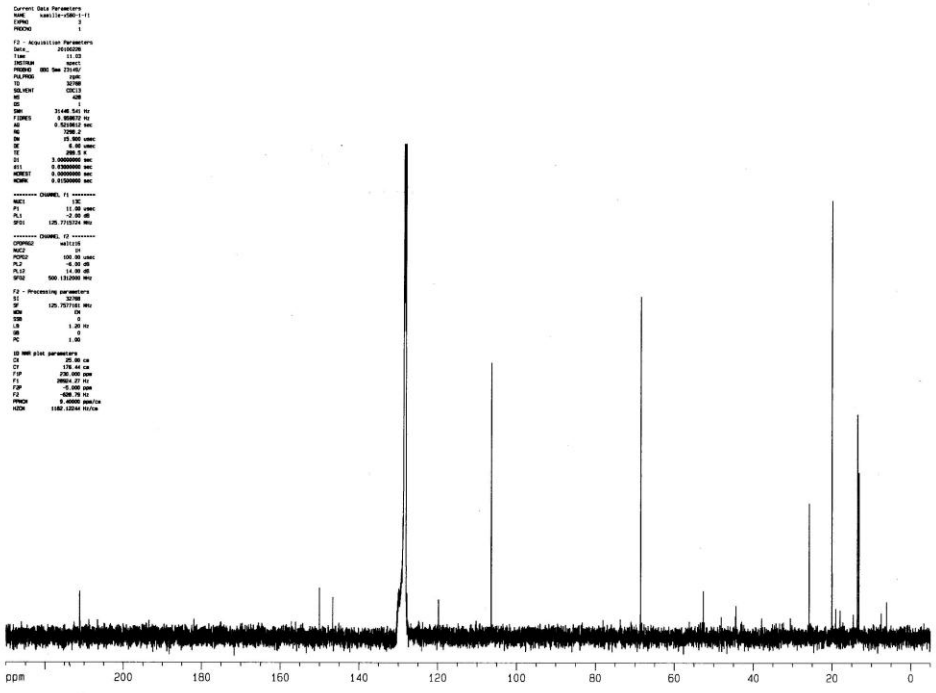

ppm 200 180 160 140 120 100 80 60 40 20 0

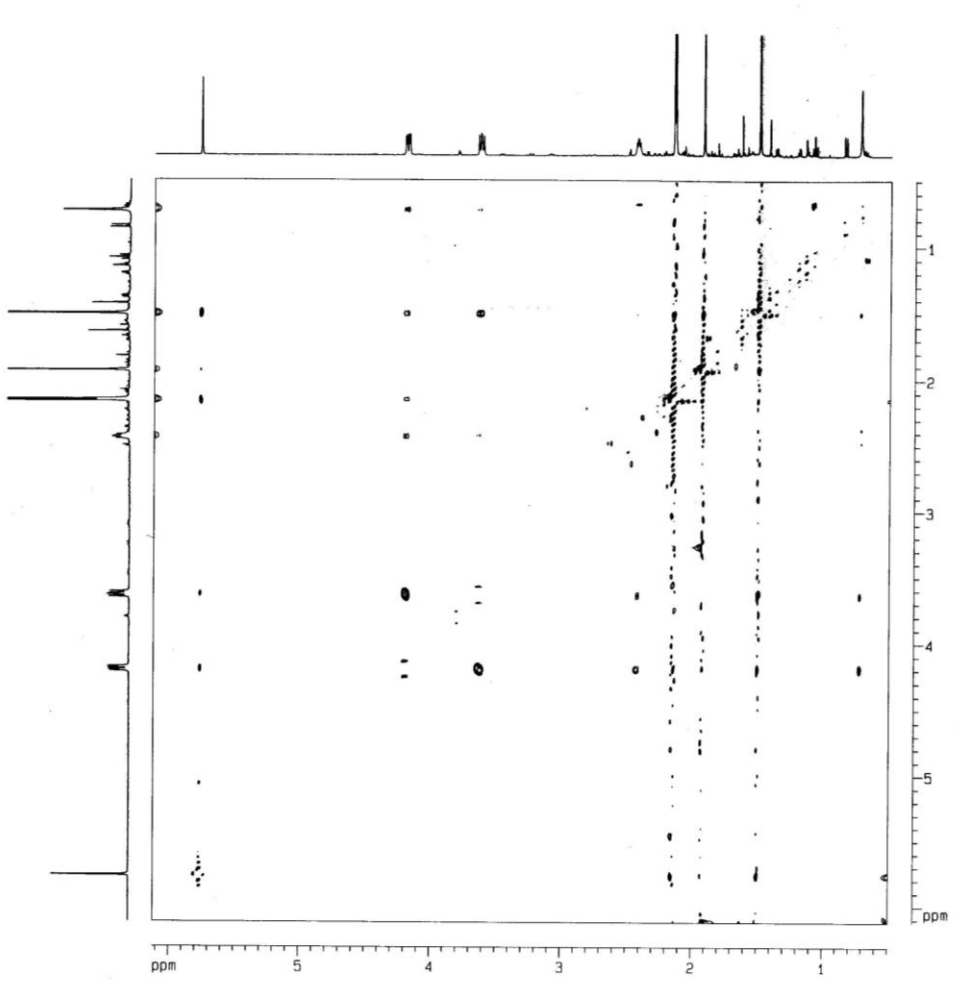

ppm
5
4
3
2
1
ppm

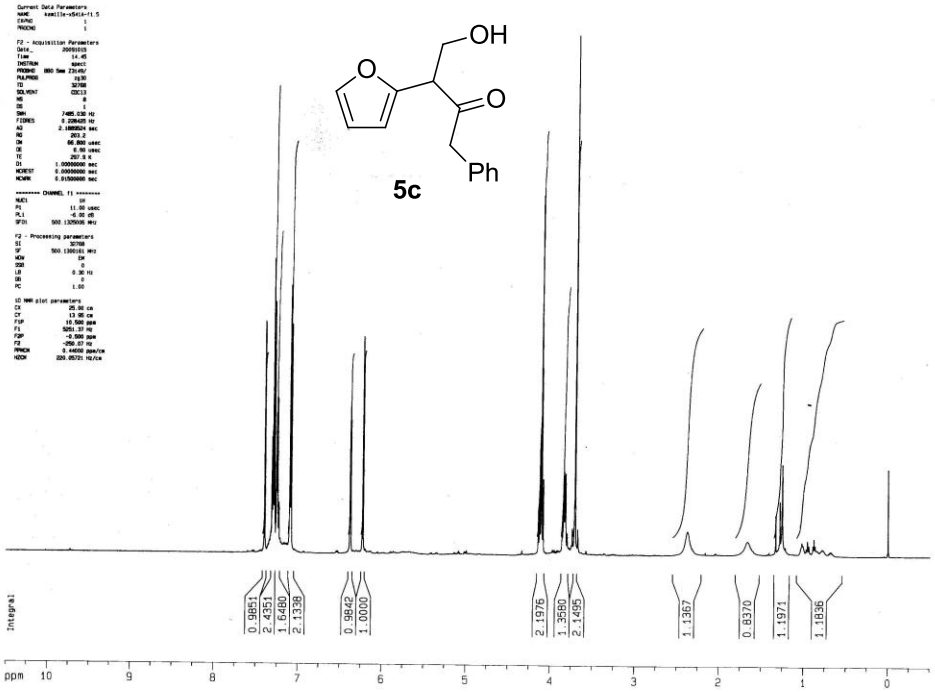

O
O
OH
5a
Integral
1.0000
1.1794
1.1470
1.0772
1.0227
6.7612
3.2488
ppm
10
9
8
7
6
5
4
3
2
1
0

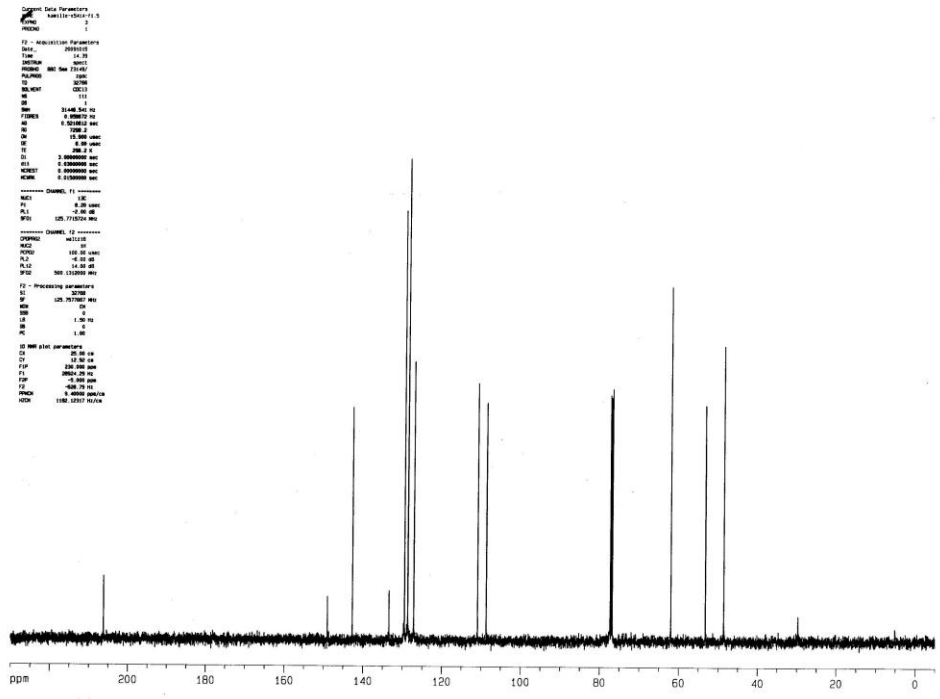

ppm
200
180
160
140
120
100
80
60
40
20
0


O
O
OH
5b
Integral
1.0000
1.1215
1.0923
1.0594
6.3262
3.1081
3.4139
ppm
10
9
8
7
6
5
4
3
2
1
0

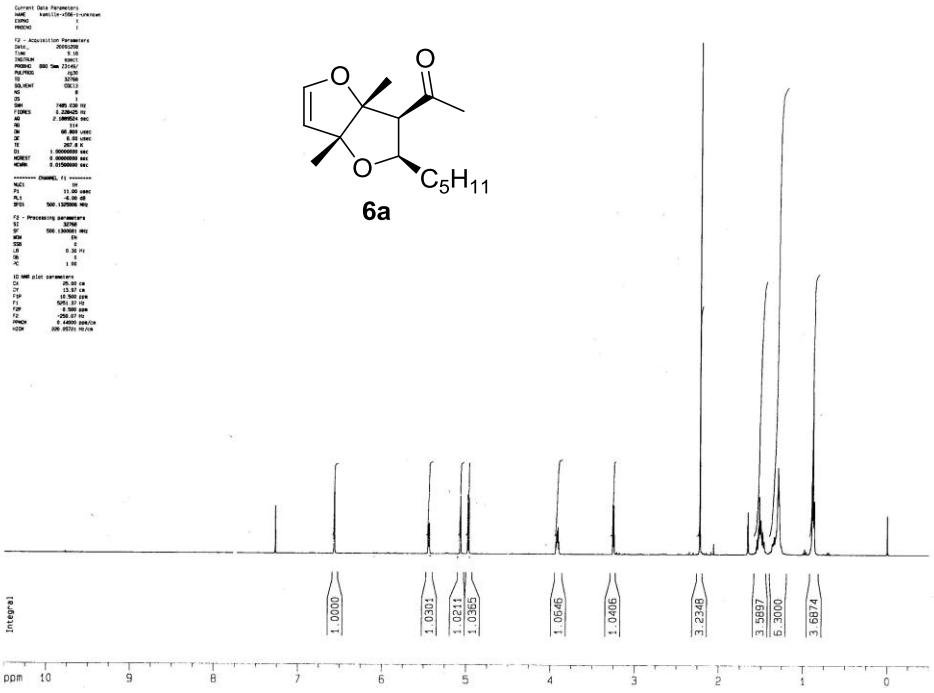

ppm
200
180
160
140
120
100
80
60
40
20
0

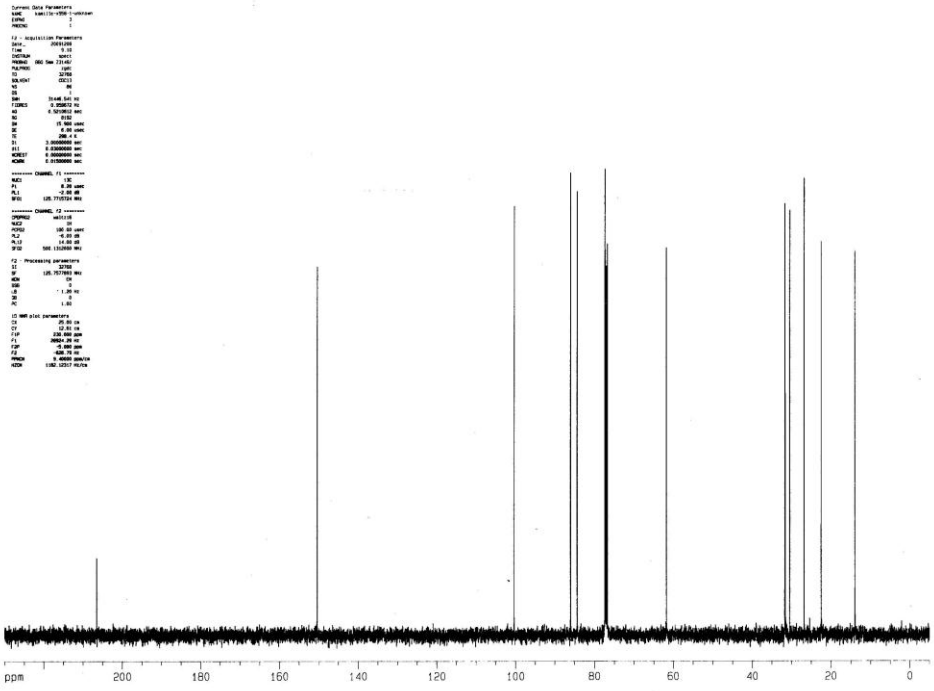

1
2
3
4
5
ppm
ppm
5
4
3
2
1

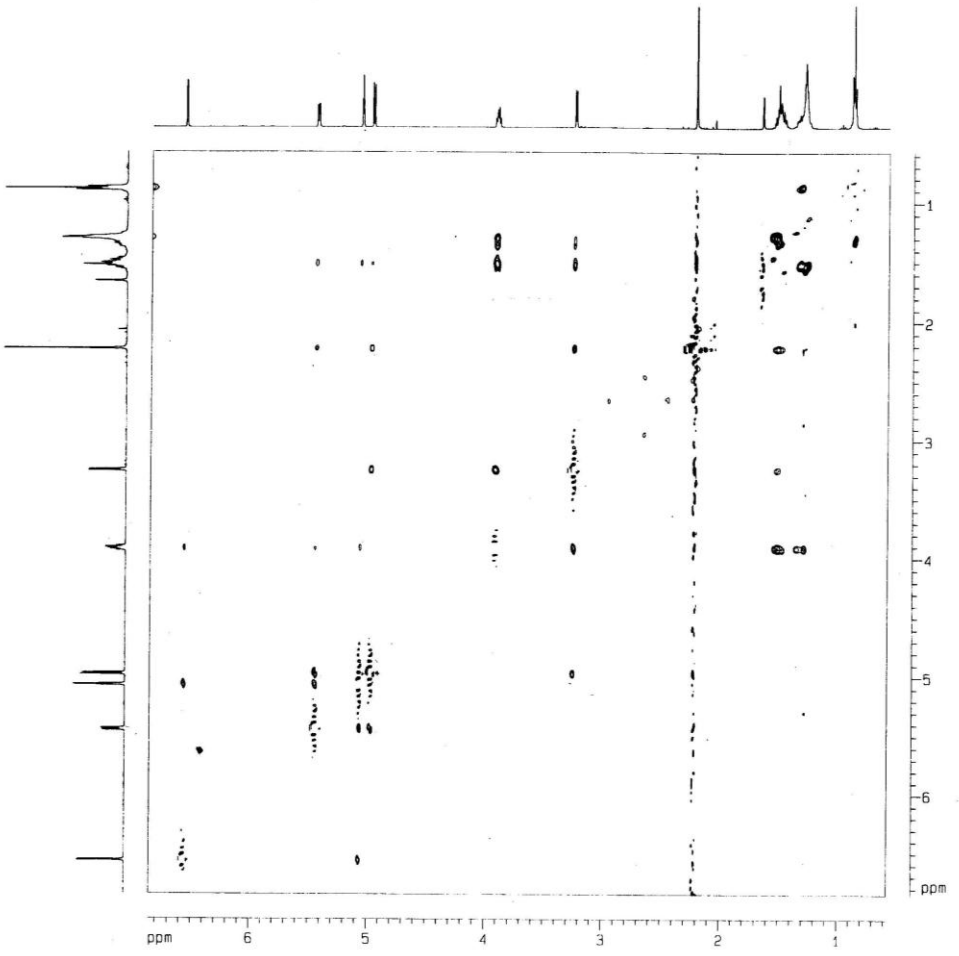

OH
O
O
Ph
5c
integral
0.9851
2.4351
1.6480
2.1338
0.9842
1.0000
2.1976
1.3580
2.1495
1.1367
0.8370
1.1971
1.1936
ppm 10 9 8 7 6 5 4 3 2 1 0

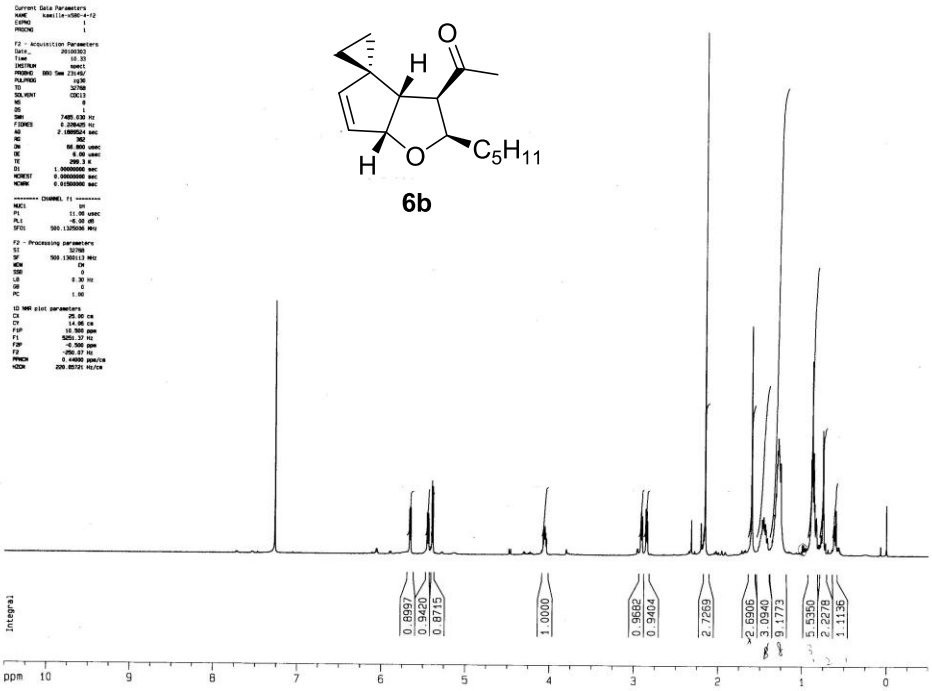

ppm 200 180 160 140 120 100 80 60 40 20 0


4
5
6
7
ppm
ppm
7
6
5
4

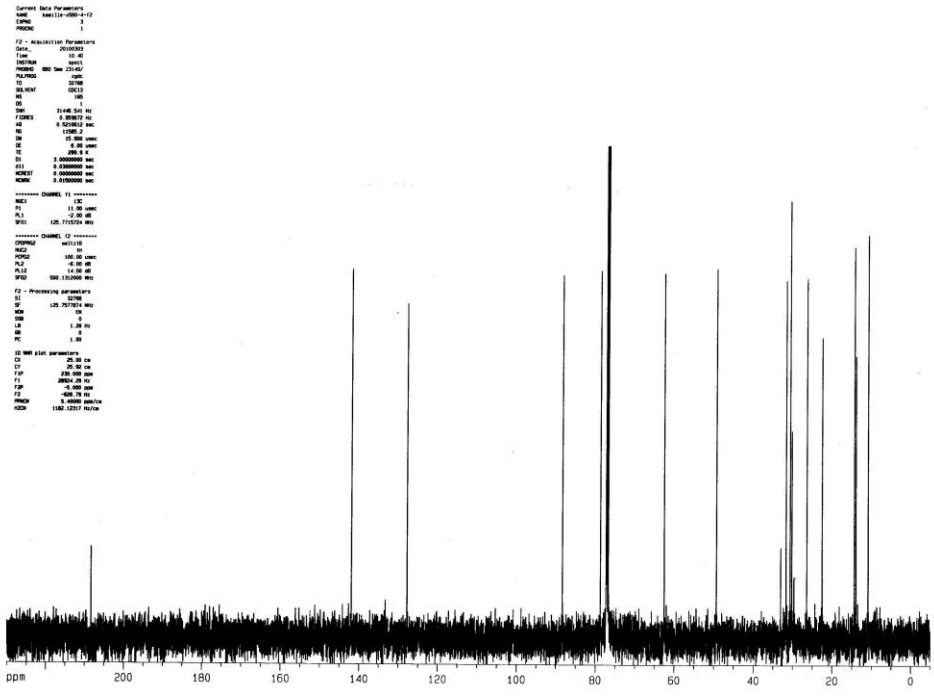

O
O
O
C_5H_{11}
6a
Integral
1.0000
1.0301
1.0211
1.0365
1.0546
1.0406
3.2348
3.5897
5.3000
3.6874
ppm 10 9 8 7 6 5 4 3 2 1 0

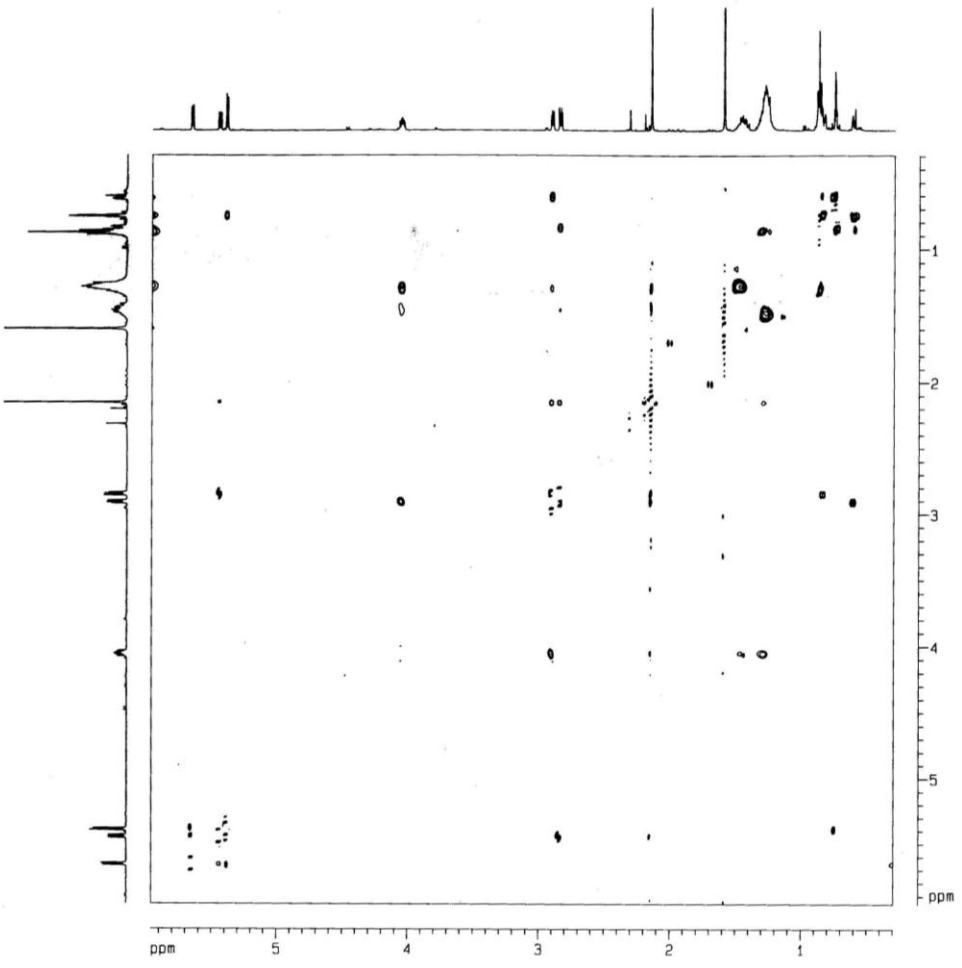

ppm 200 180 160 140 120 100 80 60 40 20 0

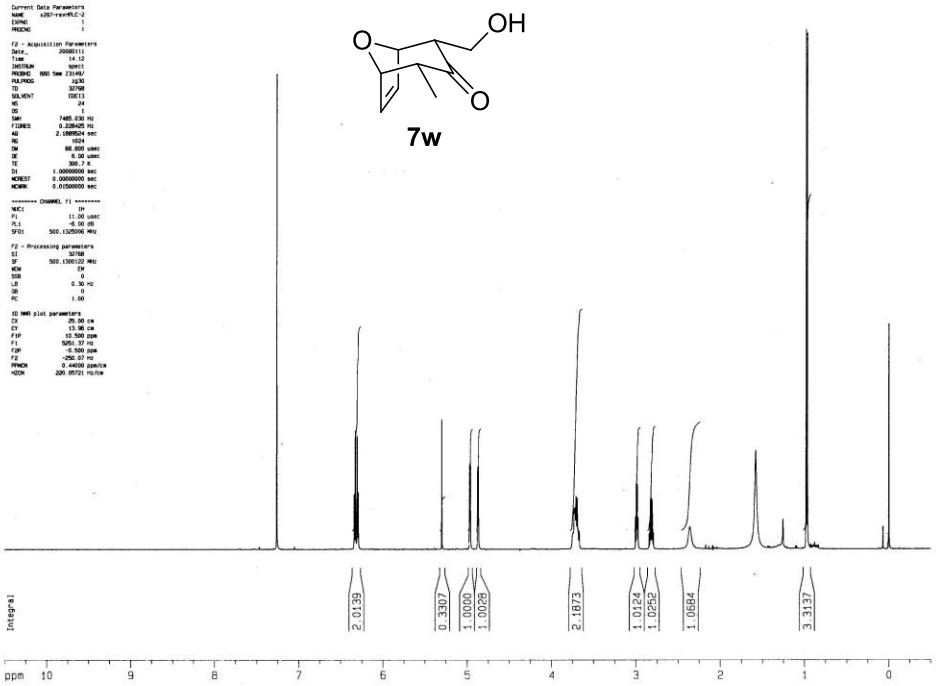

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

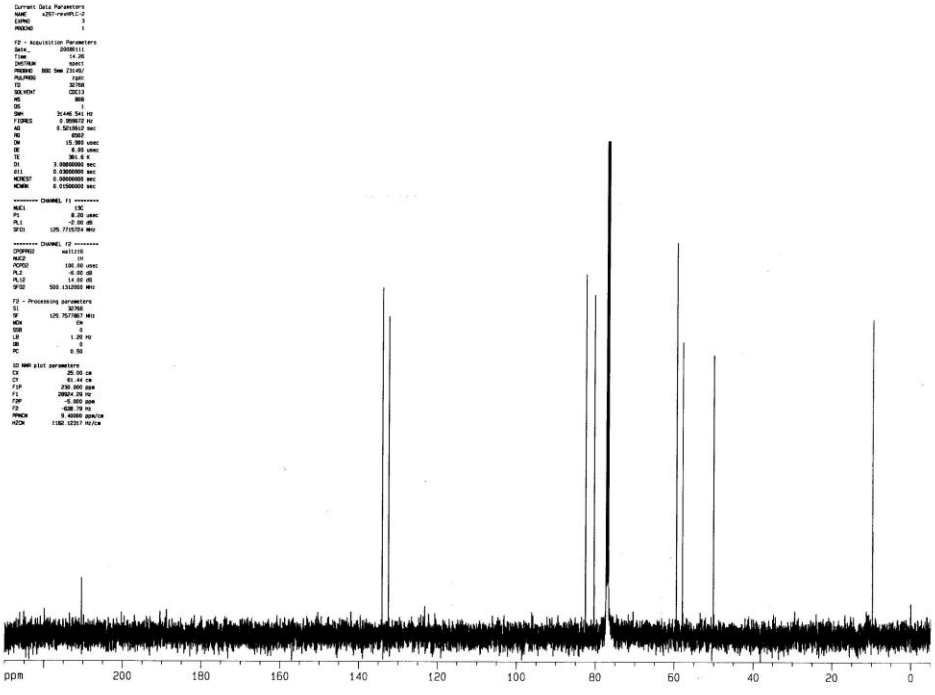

O
H
H
O
C_5H_{11}
Integral
0.8997
0.9420
0.8715
1.0000
0.9682
0.9404
2.7269
2.6906
3.0940
9.1773
5.5350
2.2278
1.1136
ppm 10 9 8 7 6 5 4 3 2 1 0

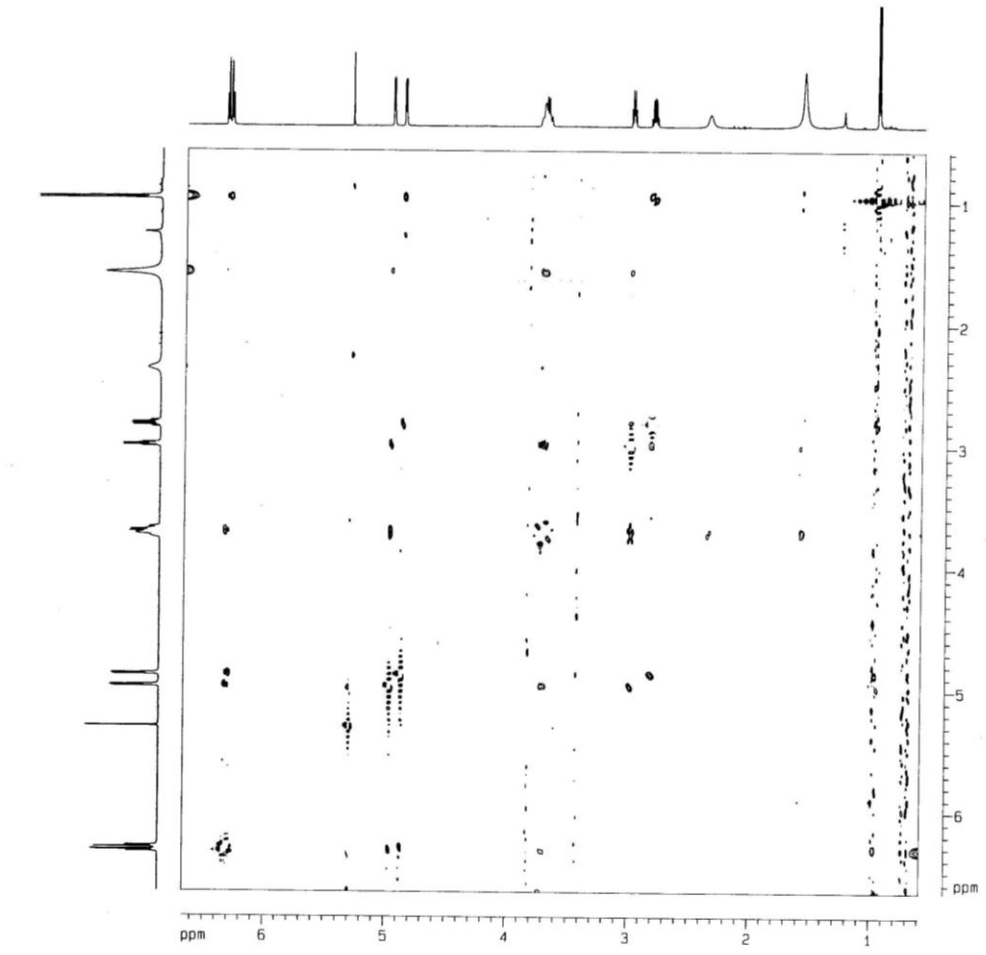
6b

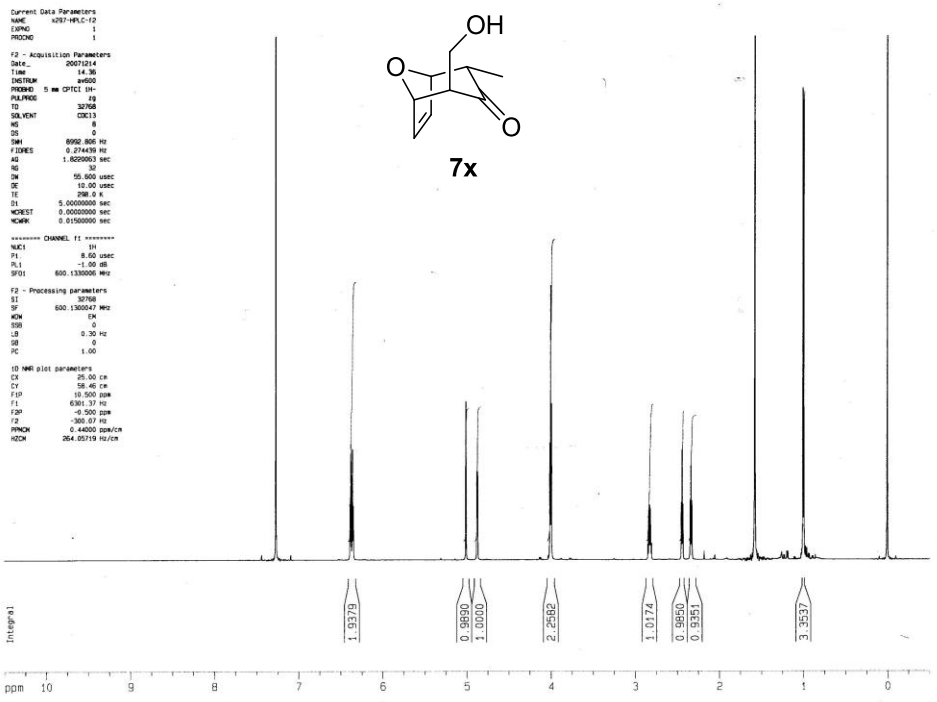

ppm 200 180 160 140 120 100 80 60 40 20 0

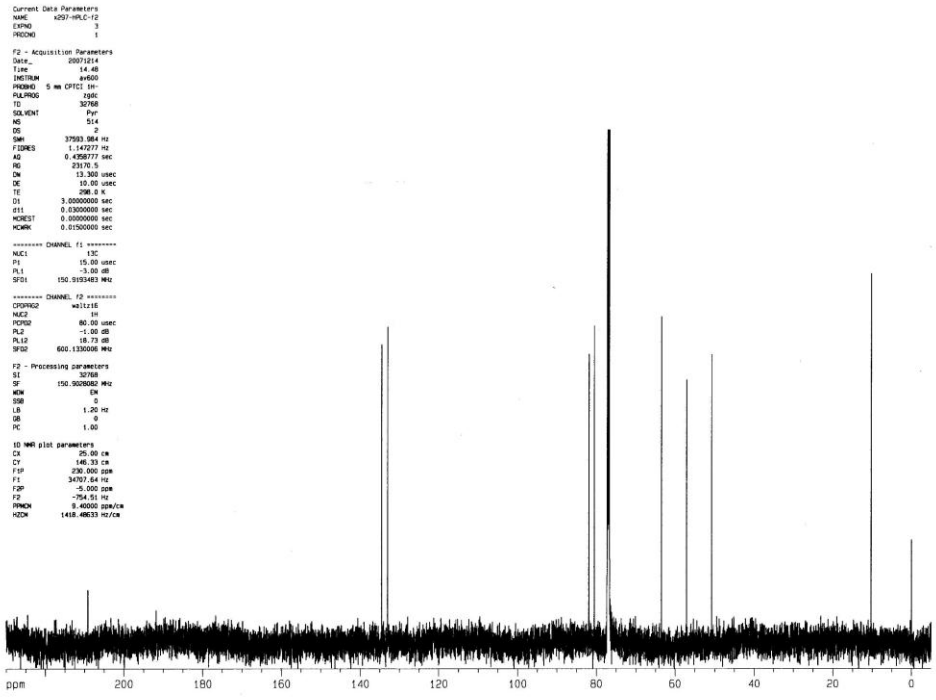

ppm
5
4
3
2
1
ppm
1
2
3
4
5

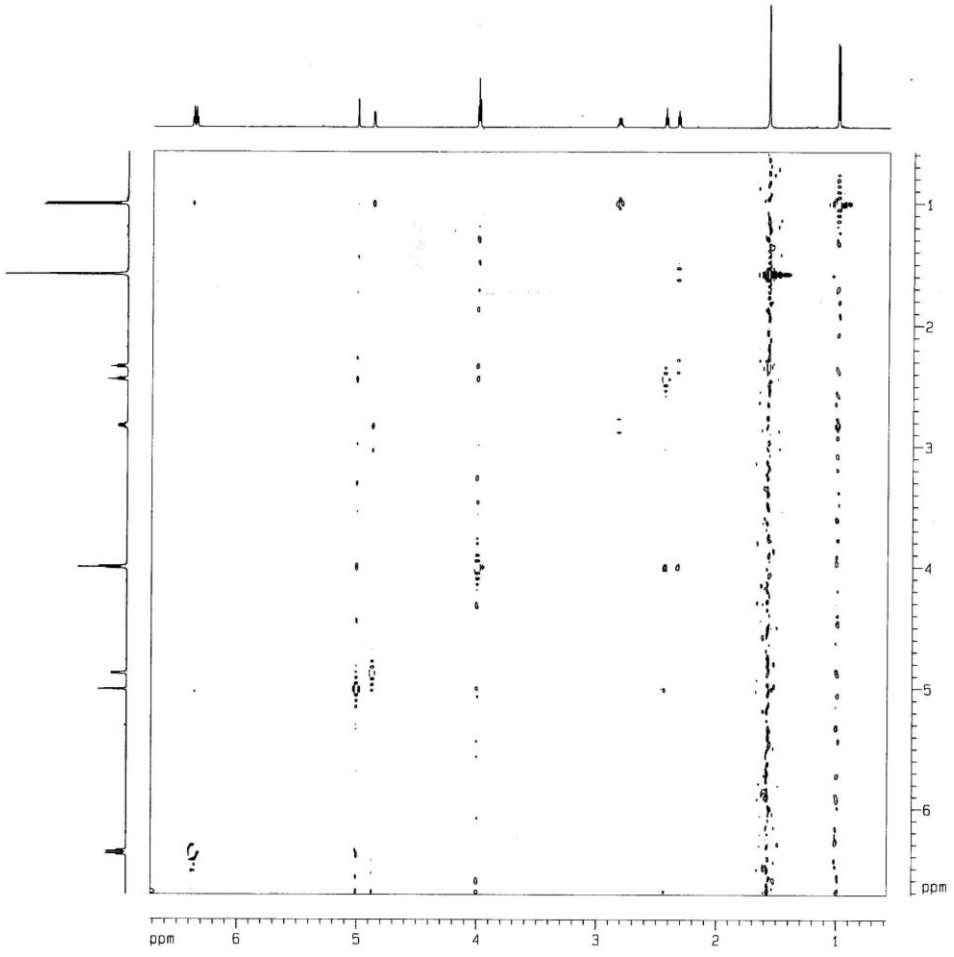

OH
O
O
7w
Integral
2.0139
0.3307
1.0000
1.0028
2.1873
1.0124
1.0252
1.0684
3.3137
ppm 10 9 8 7 6 5 4 3 2 1 0

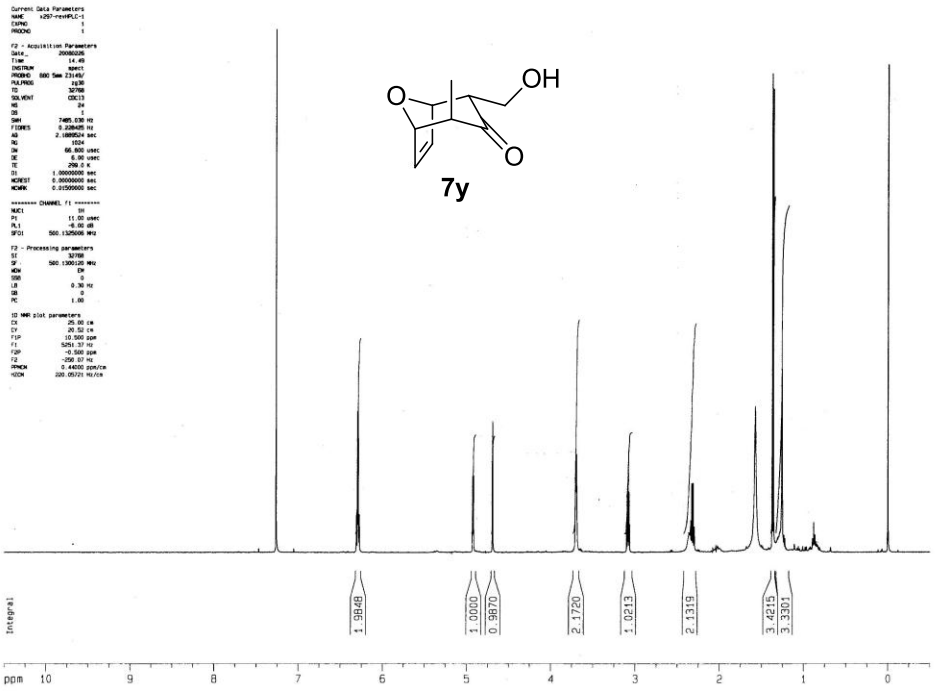

ppm 200 180 160 140 120 100 80 60 40 20 0

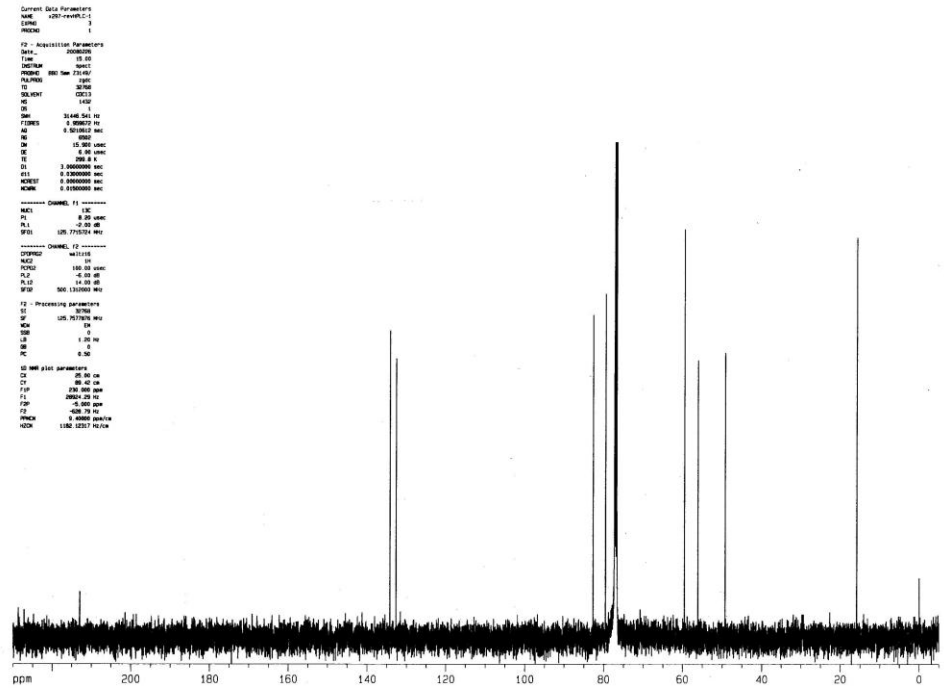

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

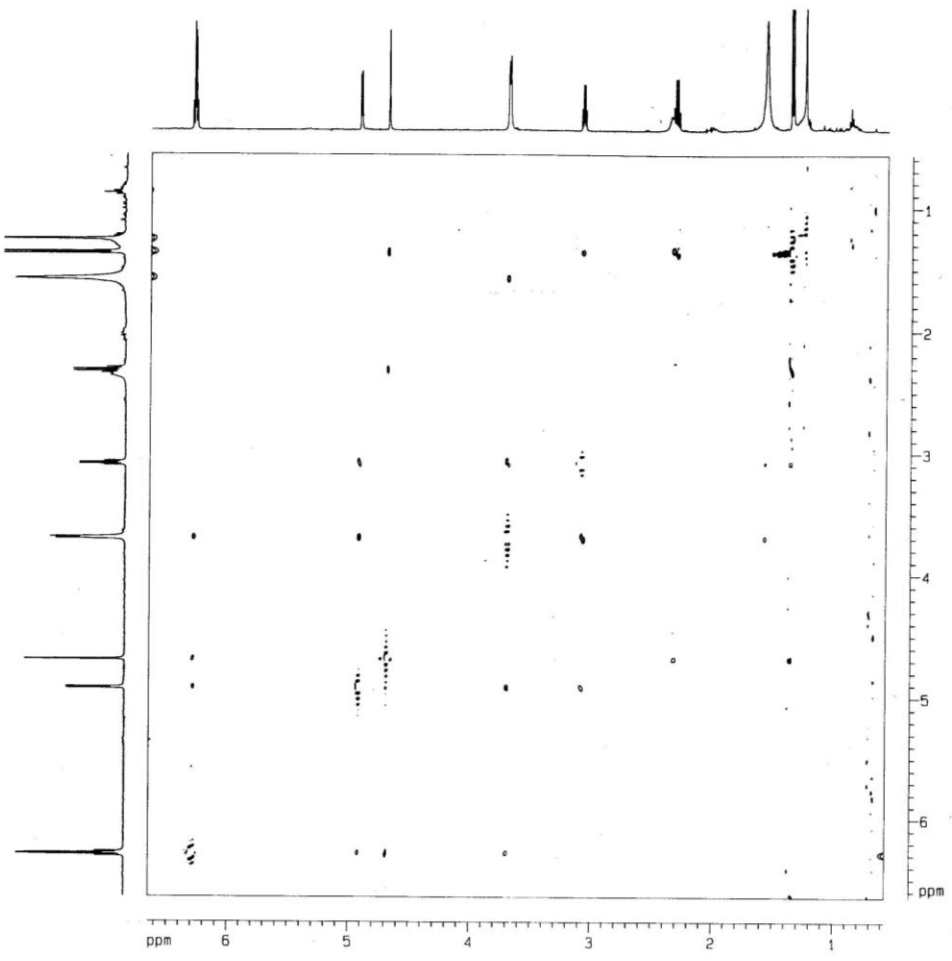

OH
O
O
7x
1.9379
0.9890
1.0000
2.2582
1.0174
0.9856
0.9351
3.3537
Integral
ppm

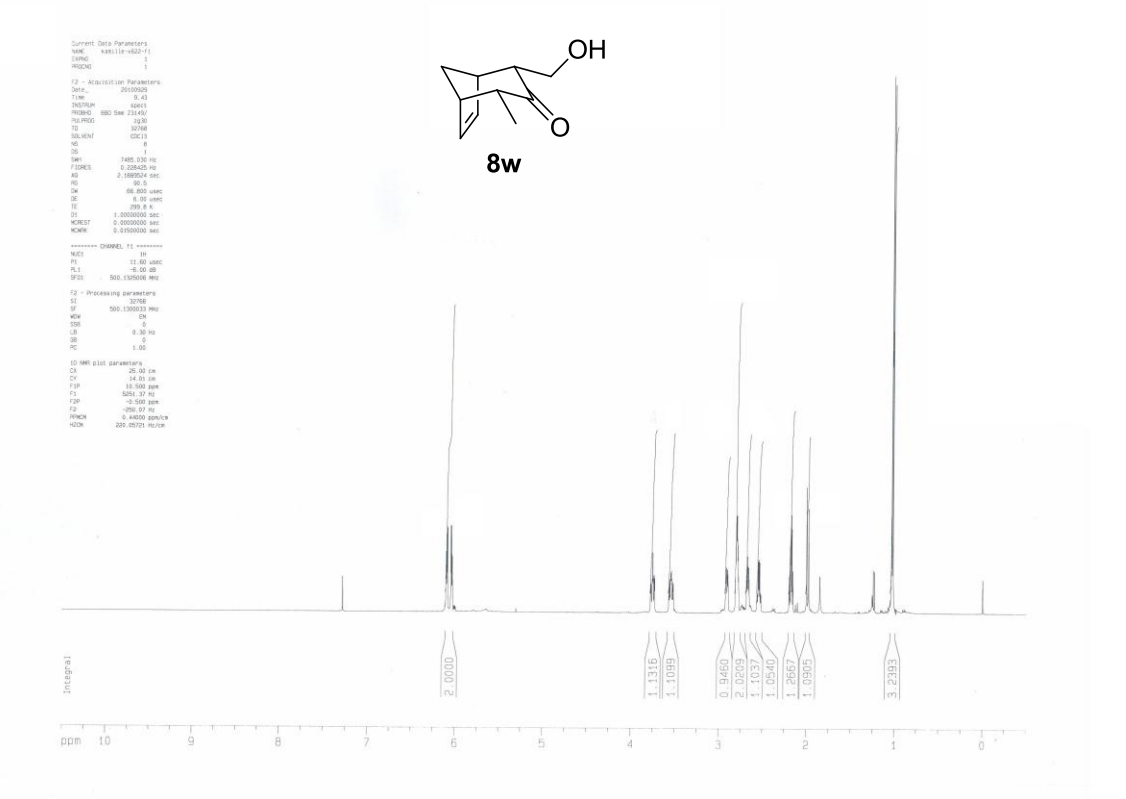

ppm

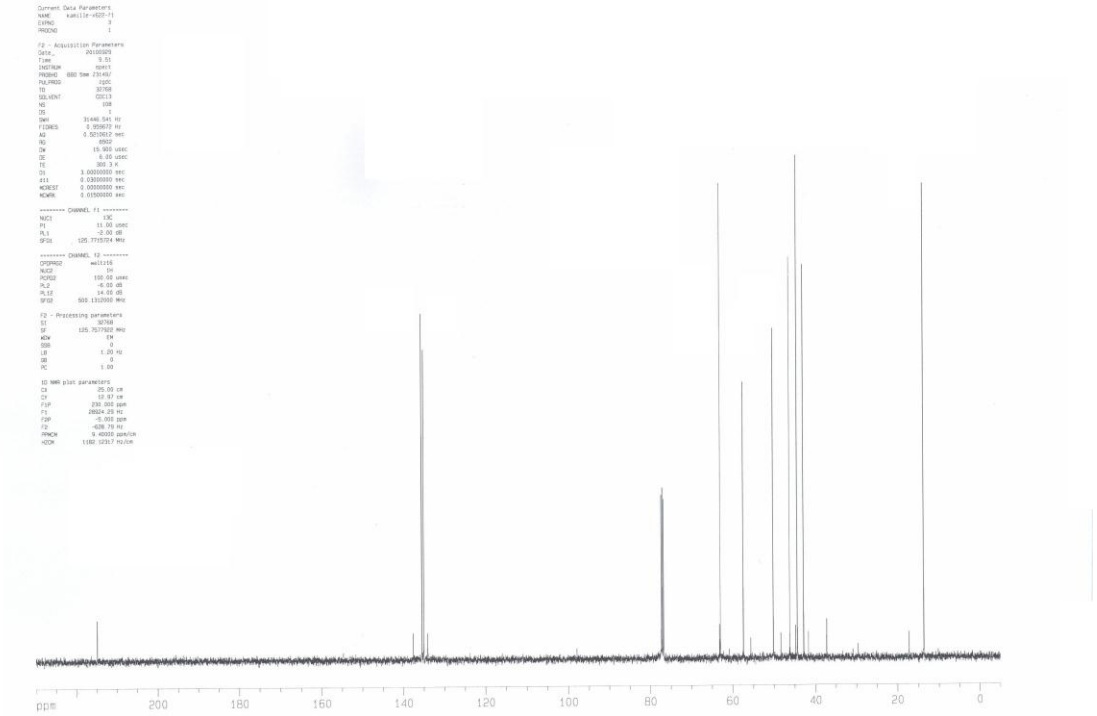

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

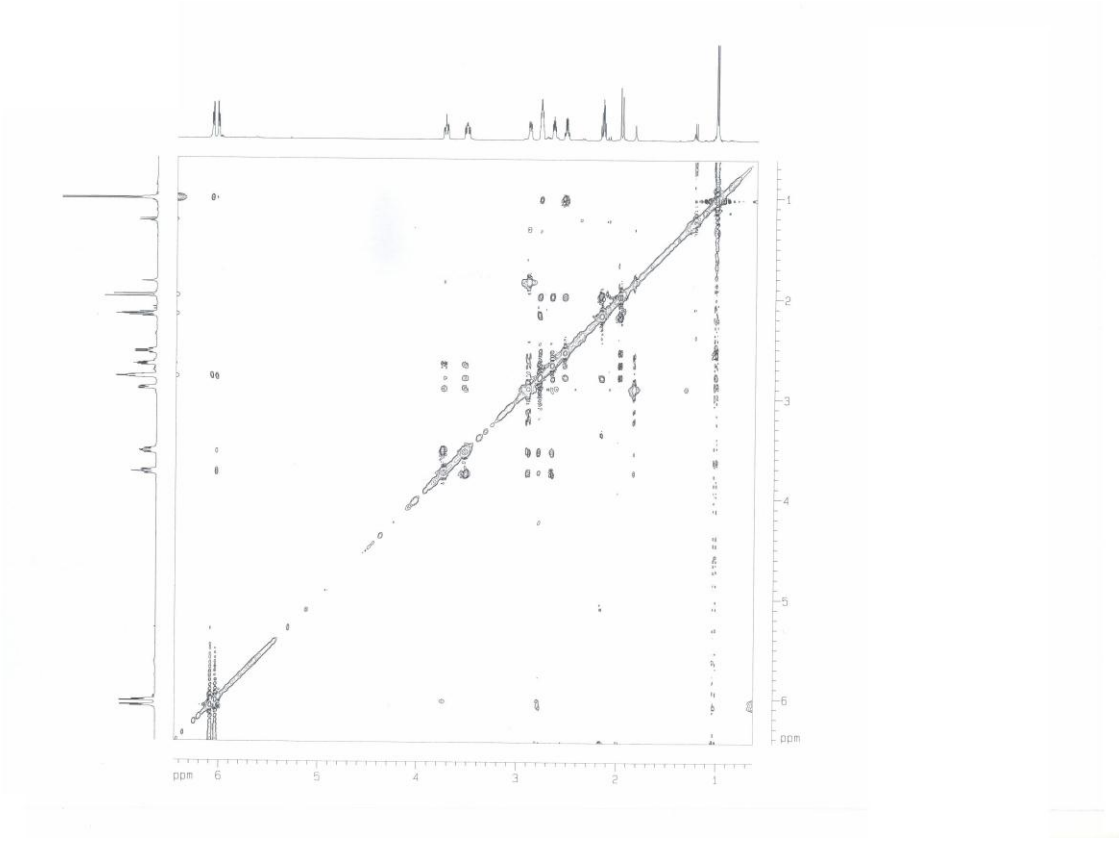

OH
O
O
7y
Integral
1.9848
1.0000
0.9870
2.1720
1.0213
2.1319
3.4215
3.3301
ppm
10
9
8
7
6
5
4
3
2
1
0

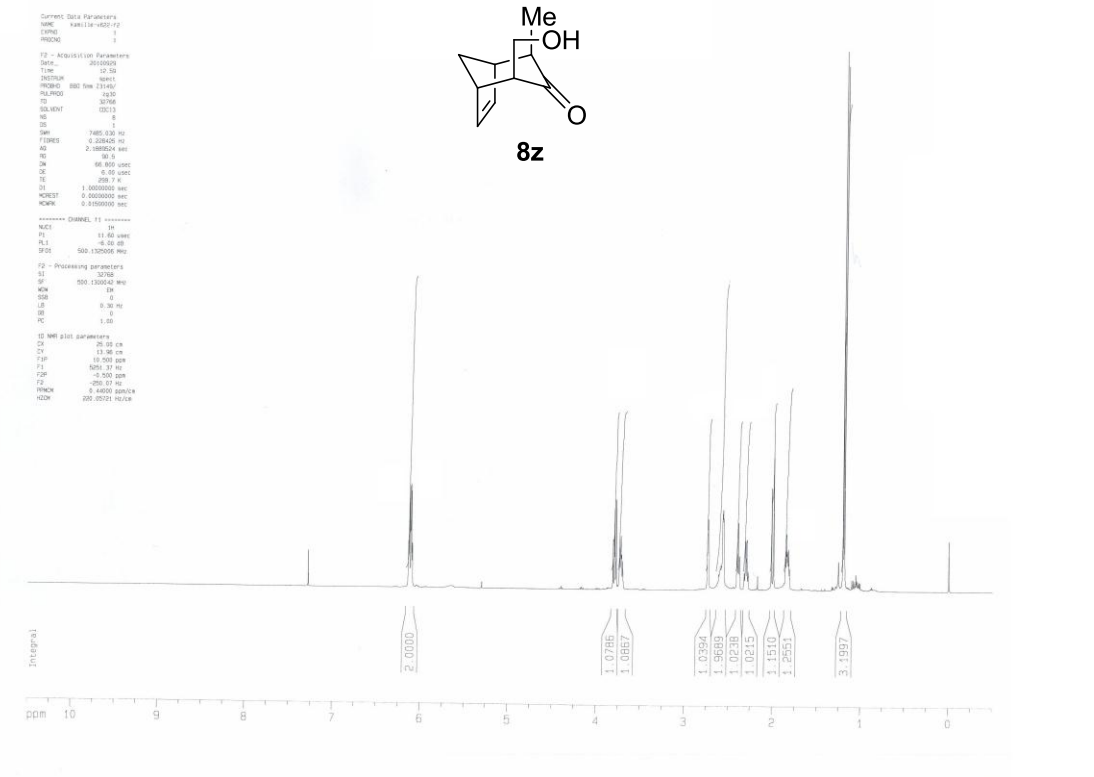

ppm
200
180
160
140
120
100
80
60
40
20
0

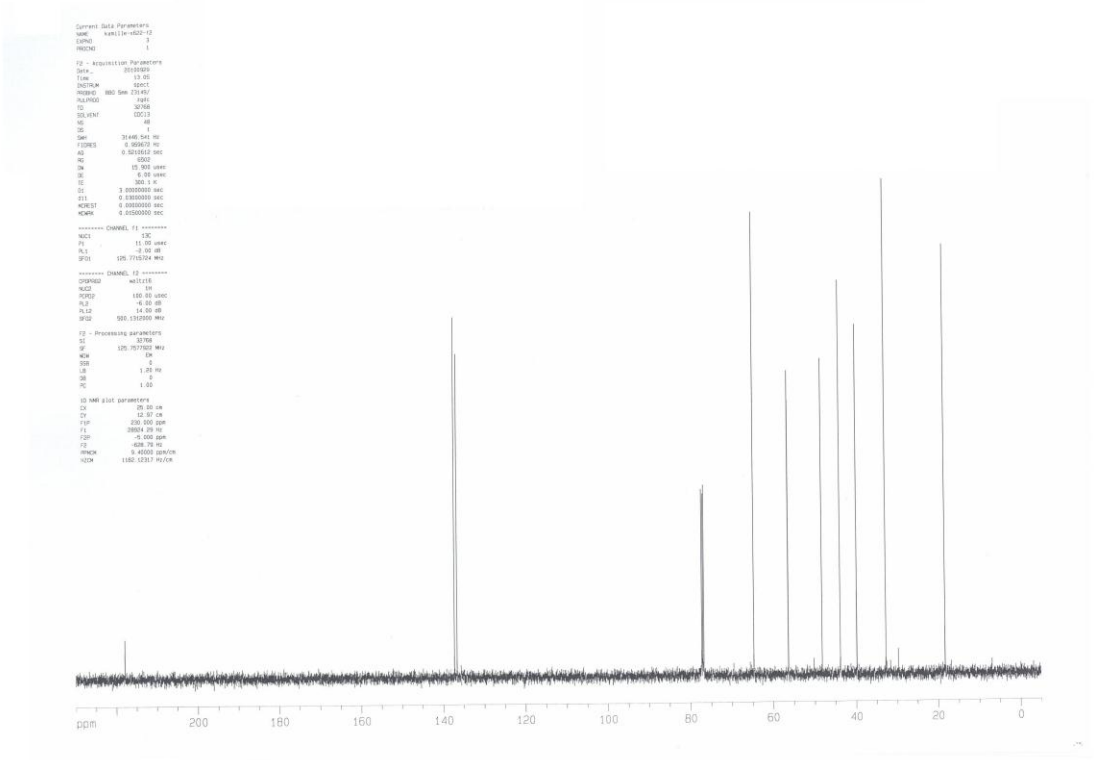

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

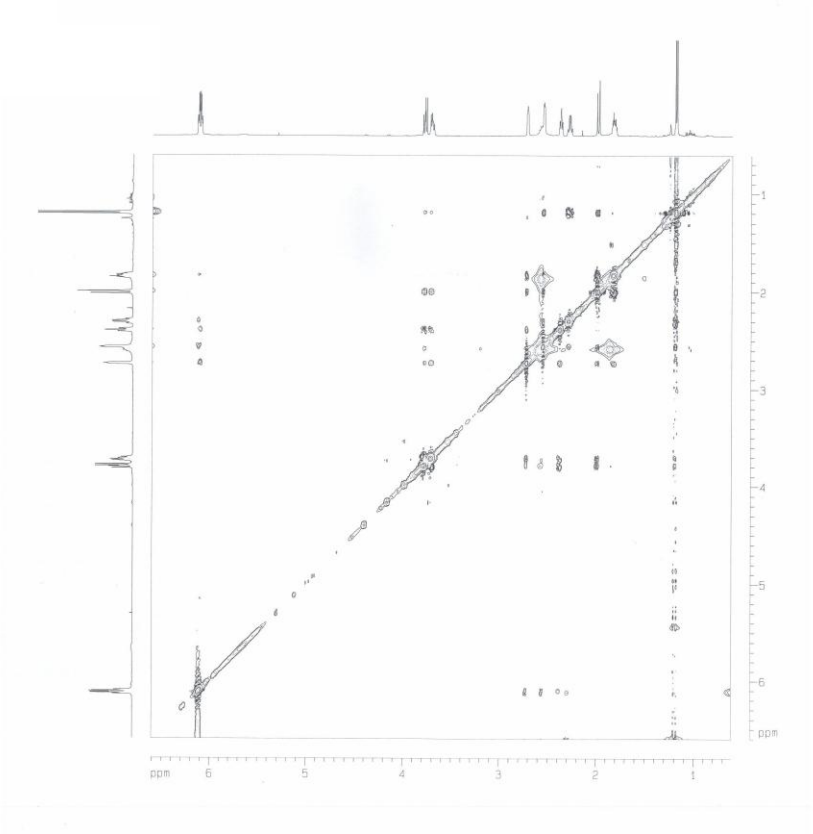

OH
O
8w
Integral
2.0000
1.1316
1.1099
0.9460
2.0269
1.1037
1.0546
1.2667
1.0905
3.2393
ppm

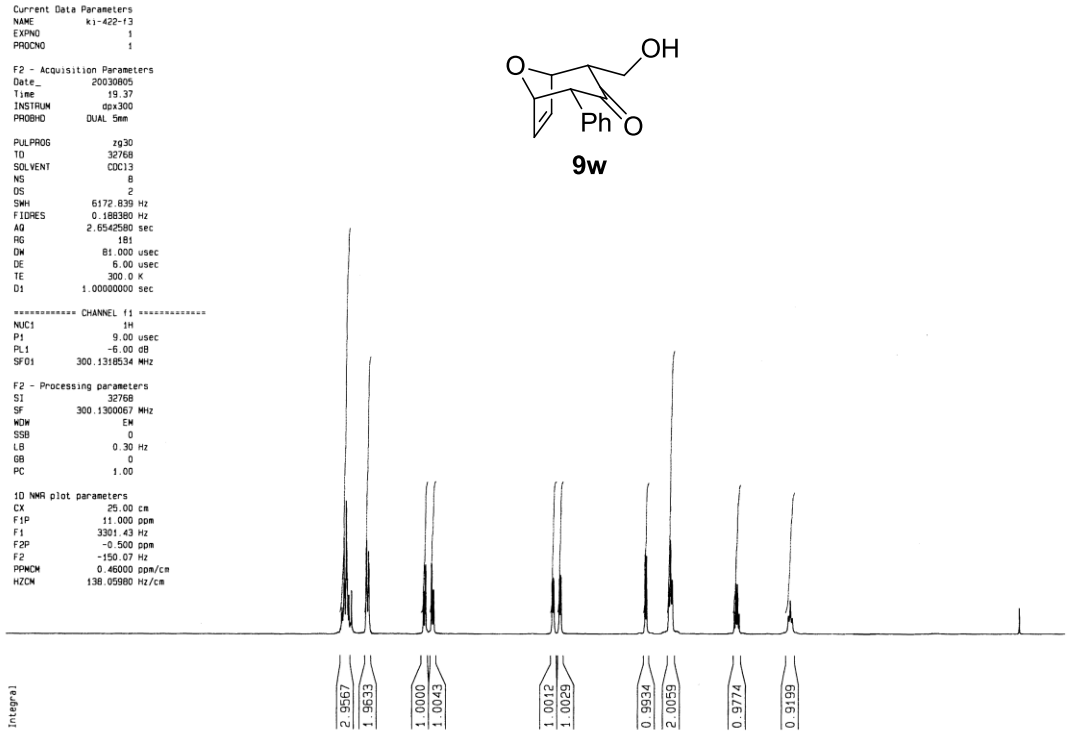

ppm

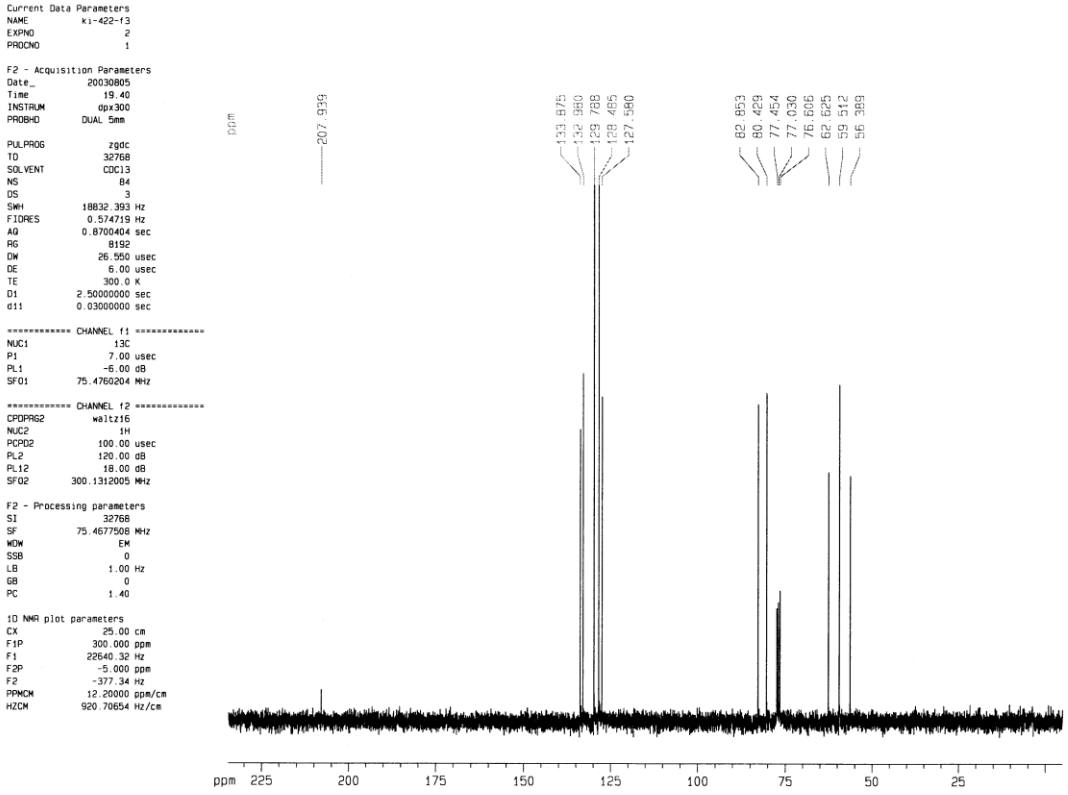

ppm
6
5
4
3
2
1
ppm

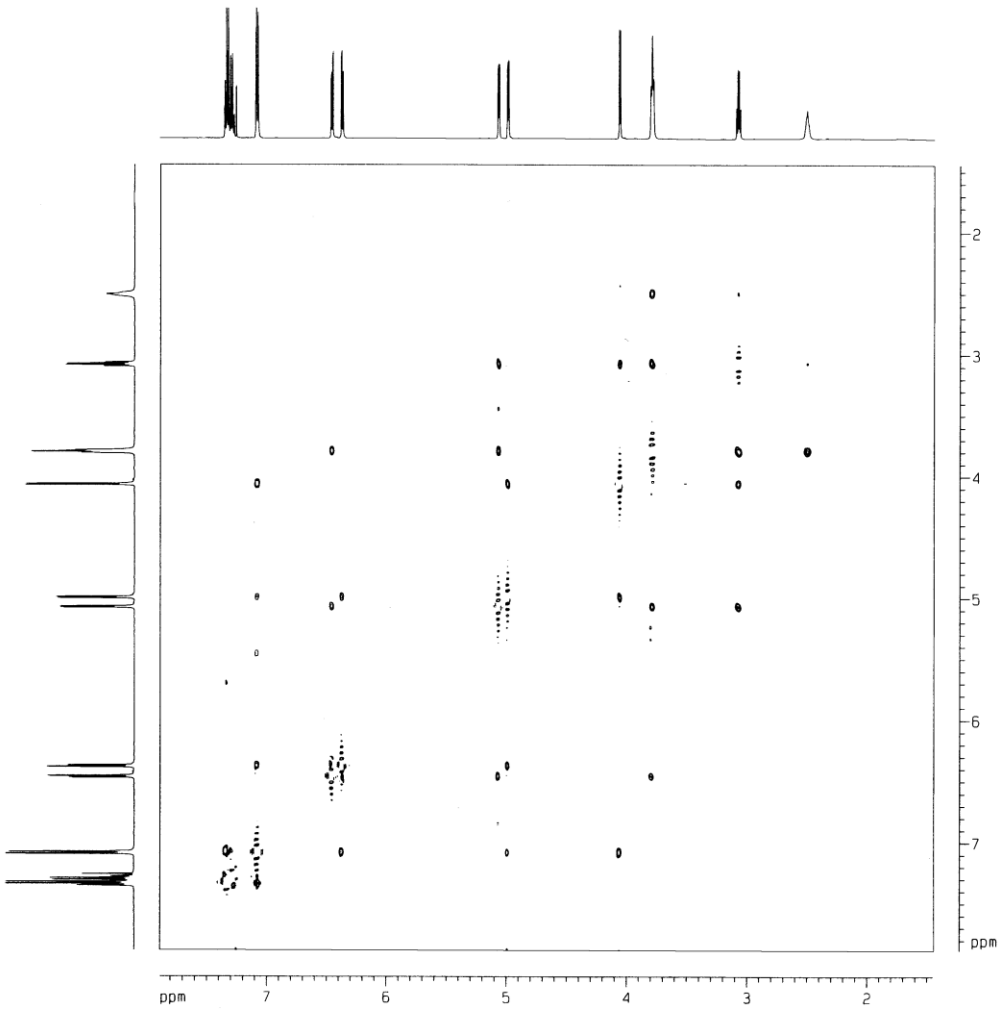

Me
OH
O
8z
Integral
2.0000
1.0786
1.0867
1.0394
1.9689
1.0238
1.0215
1.1510
1.2551
3.1997
ppm

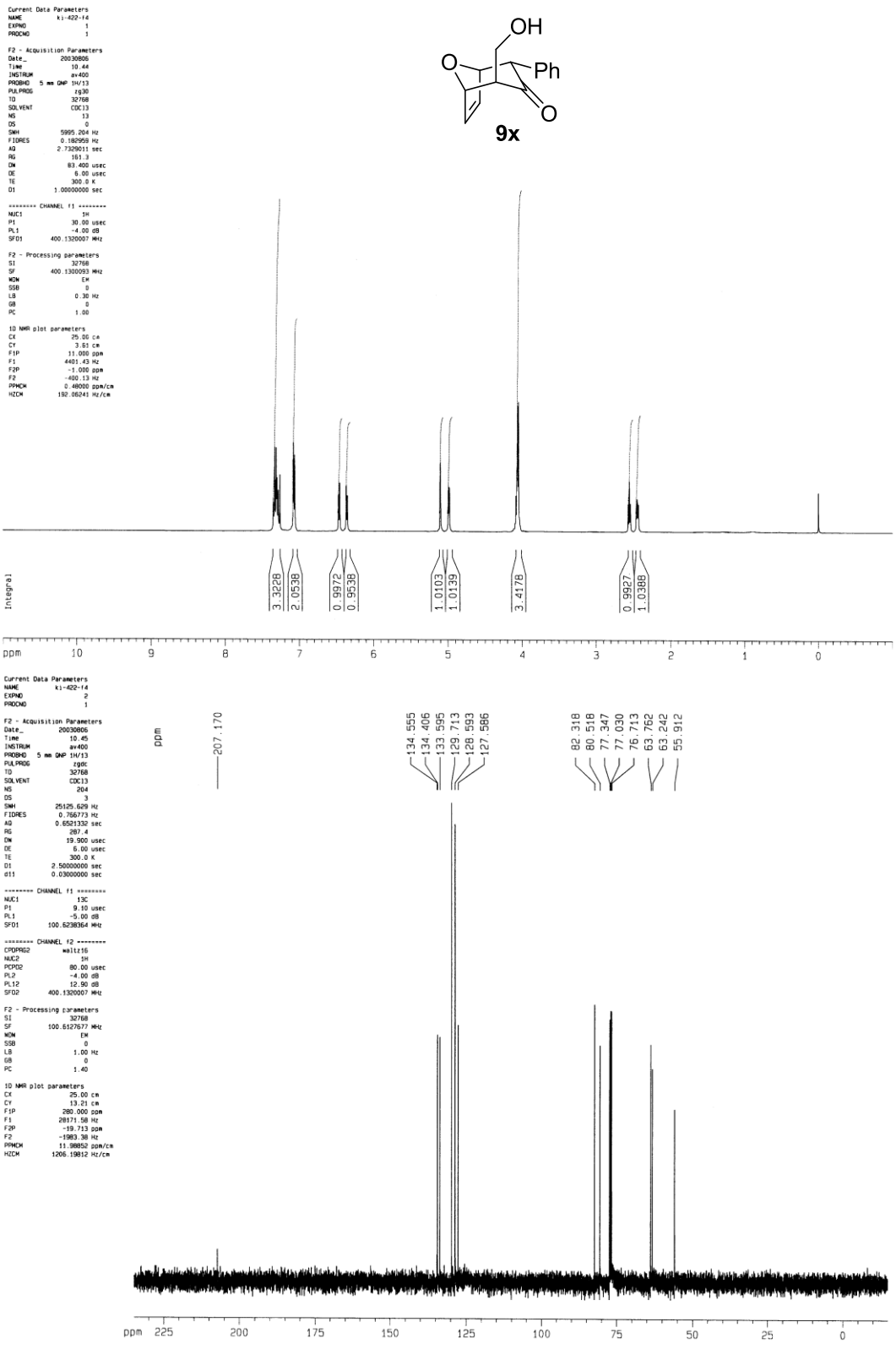

ppm

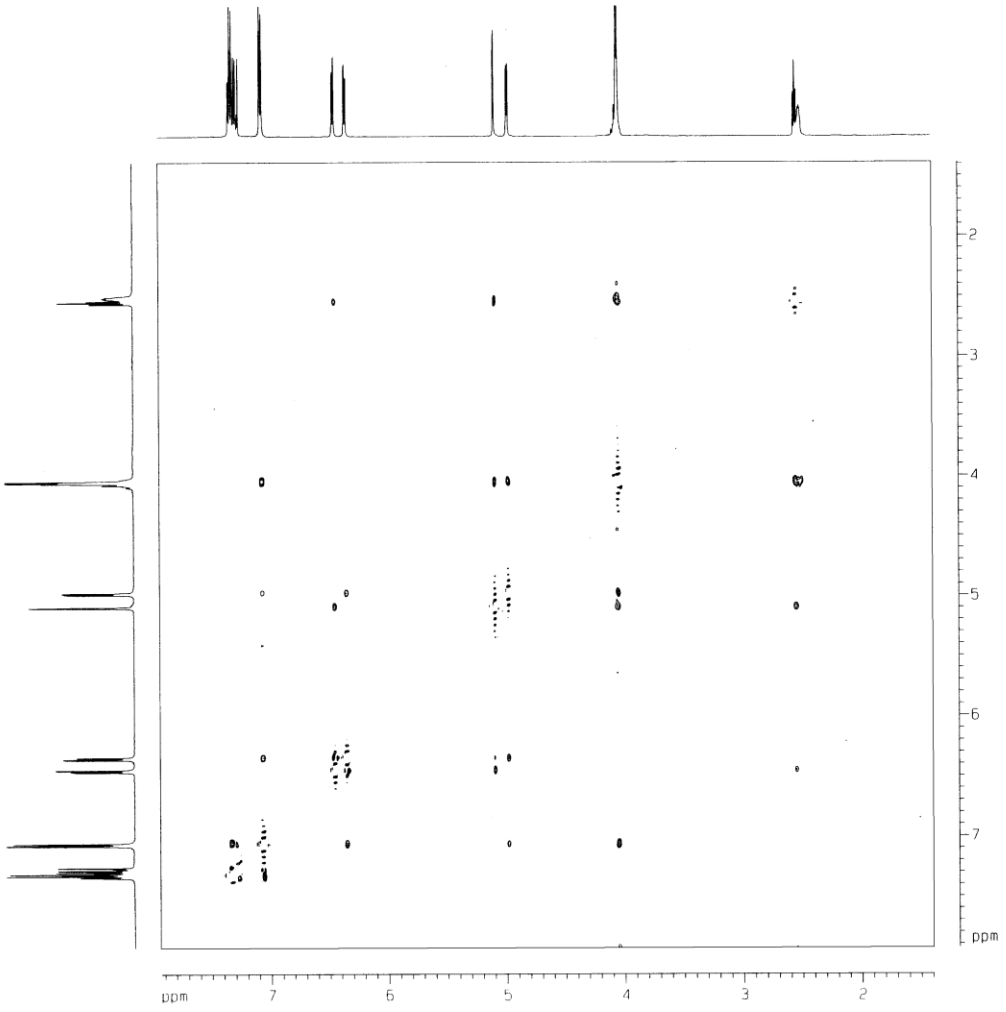

ppm
6
5
4
3
2
1
ppm
1
2
3
4
5
6

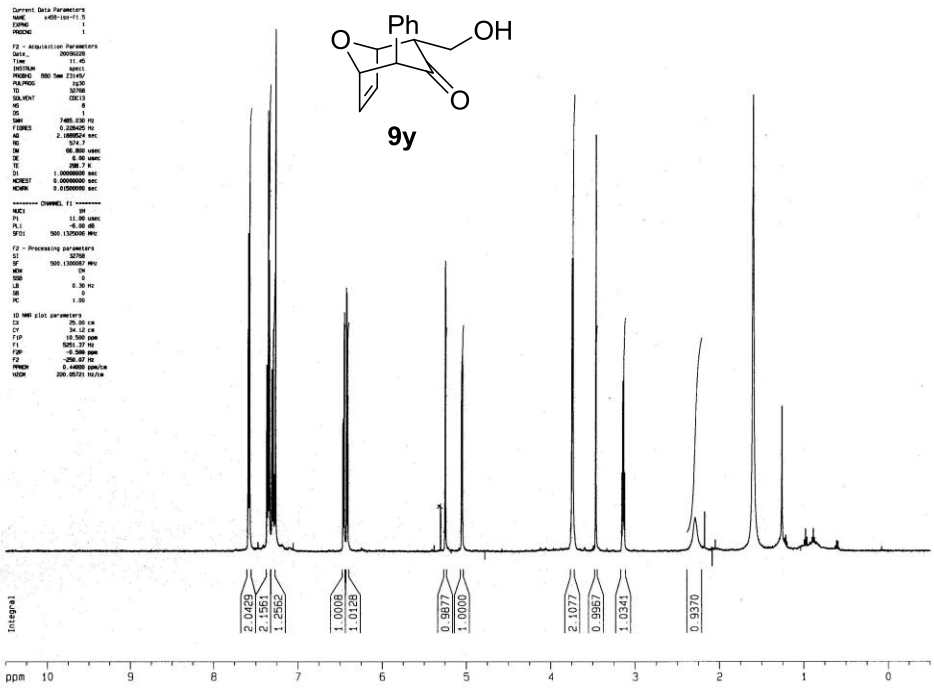

Current Data Parameters
NAME ki-422-f3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030805
Time 19.37
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 181
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.46000 ppm/cm
HZCM 138.05980 Hz/cm
OH
O
Ph
O
9w
Integral
2.9567
1.9633
1.0000
1.0043
1.0012
1.0029
0.9934
2.0059
0.9774
0.9199
ppm
10
9
8
7
6
5
4
3
2
1
0

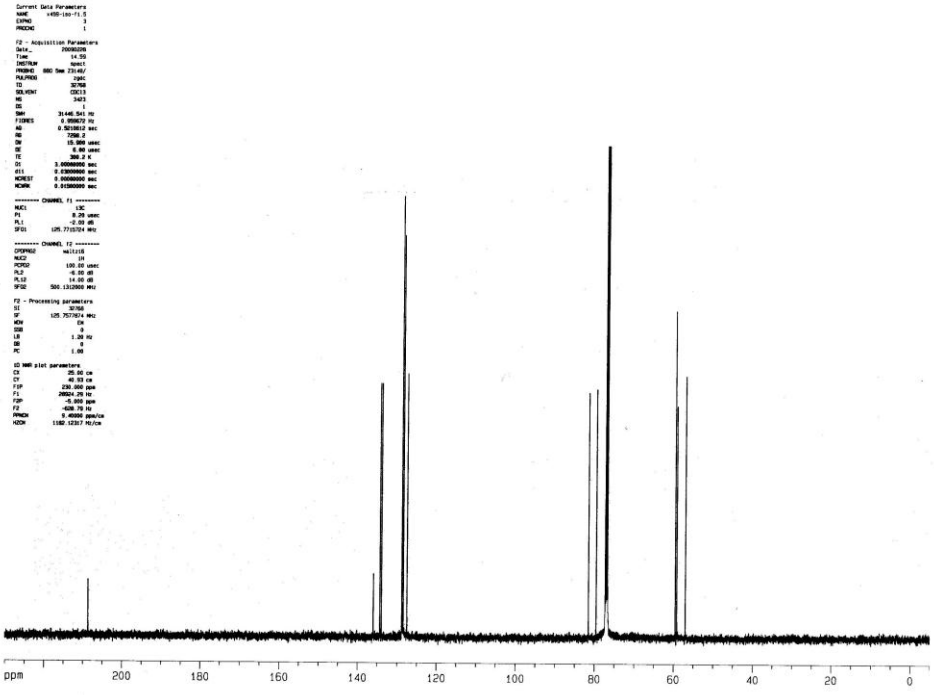

Current Data Parameters
NAME ki-422-f3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030805
Time 19.40
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zgdc
TD 32768
SOLVENT CDCl3
NS 84
DS 3
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 8192
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.4760204 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677508 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1P 300.000 ppm
F1 22640.32 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 12.20000 ppm/cm
HZCM 920.70654 Hz/cm
ppm
207.939
133.875
132.980
129.788
128.485
127.580
82.853
80.429
77.454
77.030
76.606
62.625
59.512
56.389
ppm
225
200
175
150
125
100
75
50
25

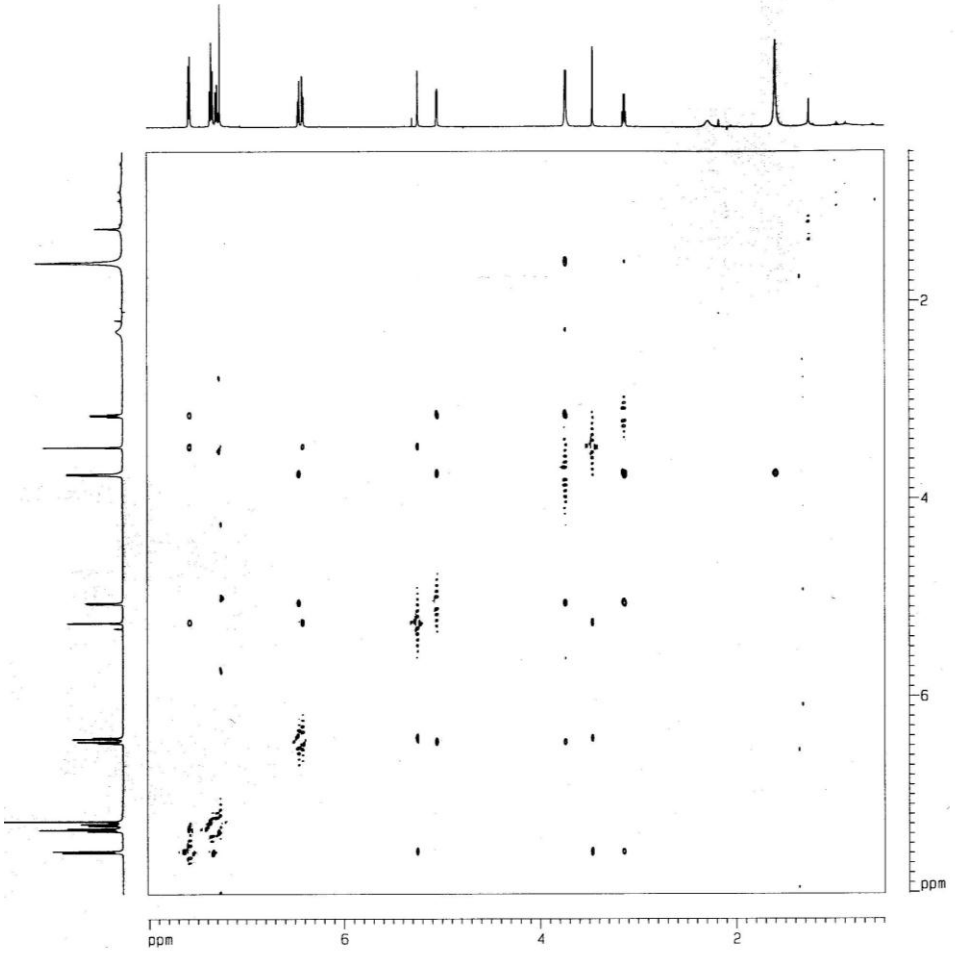

2
3
4
5
6
7
ppm
ppm
7
6
5
4
3
2

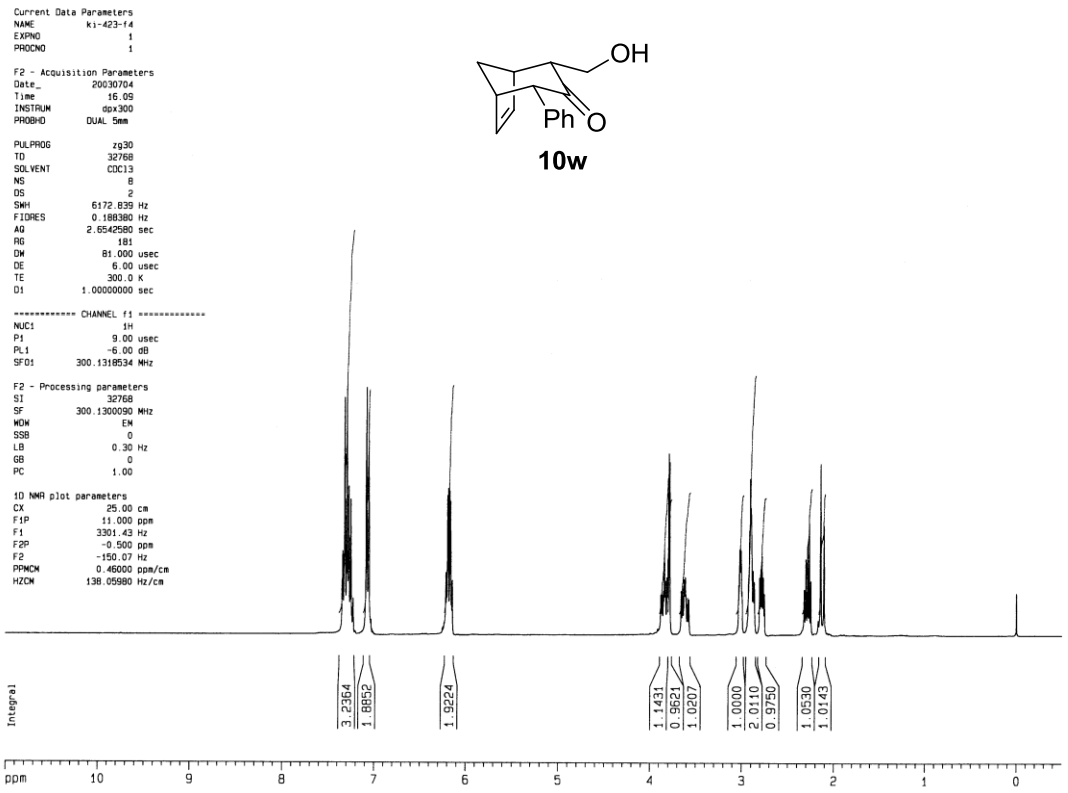

OH
O
Ph
O
9x
Integral
3.3228
2.0538
0.9972
0.9538
1.0103
1.0139
3.4178
0.9927
1.0388
ppm
207.170
134.555
134.406
133.595
129.713
128.593
127.586
82.318
80.518
77.347
77.030
76.713
63.762
63.242
55.912

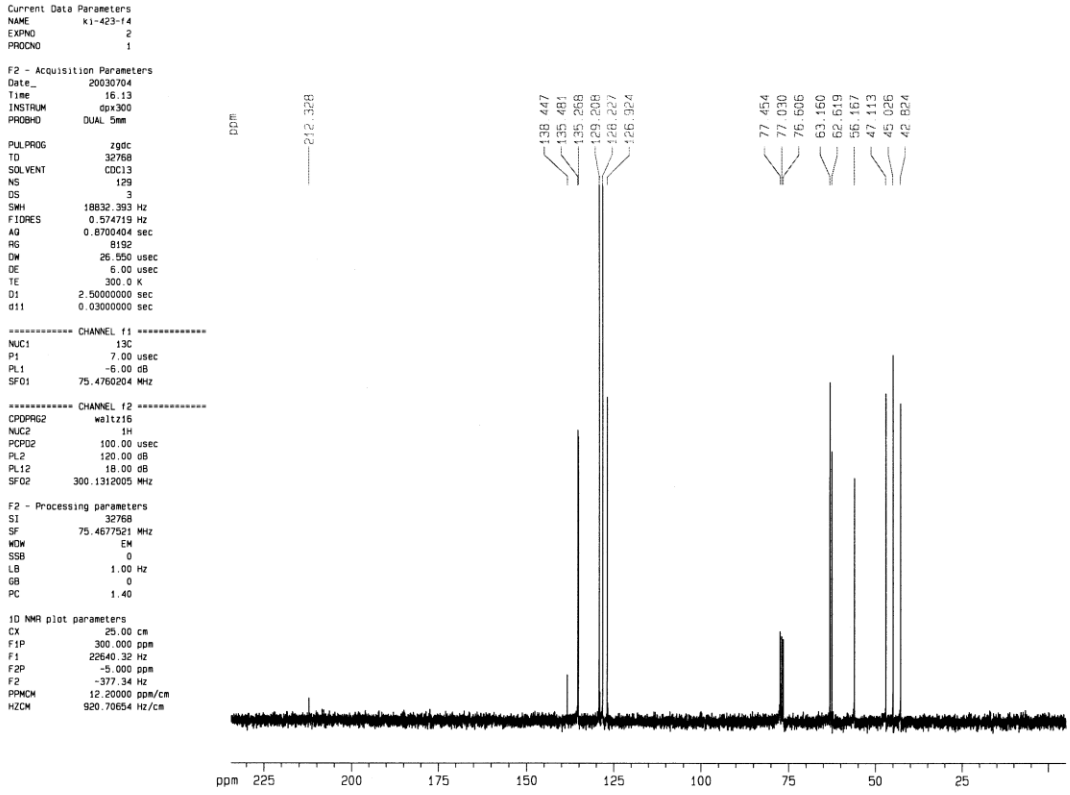

2
3
4
5
6
7
ppm
ppm
7
6
5
4
3
2

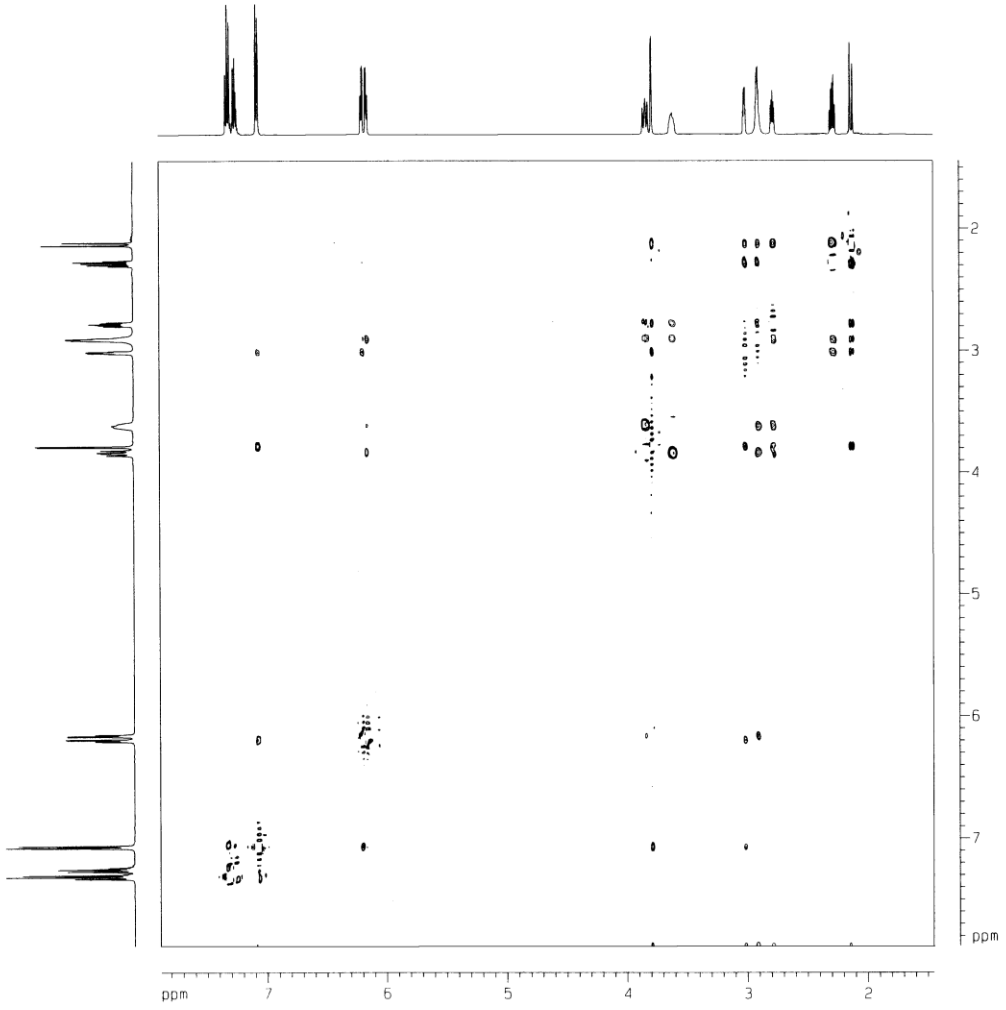

Ph
OH
O
O
9y
Integral
2.0429
2.1561
1.2562
1.0008
1.0128
0.9877
1.0000
2.1077
0.9967
1.0341
0.9370
ppm 10 9 8 7 6 5 4 3 2 1 0

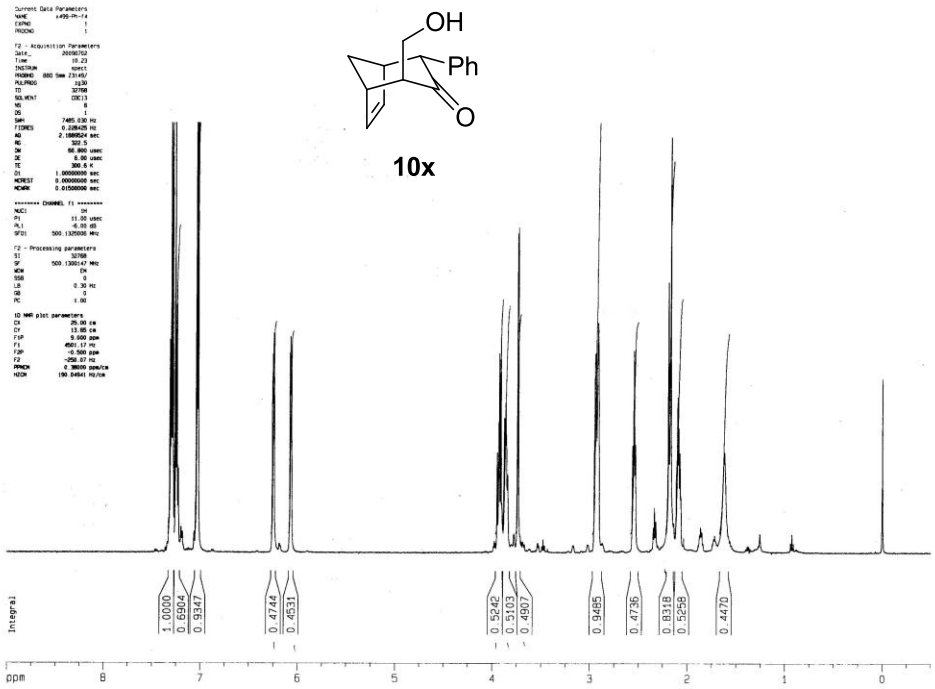

ppm 200 180 160 140 120 100 80 60 40 20 0

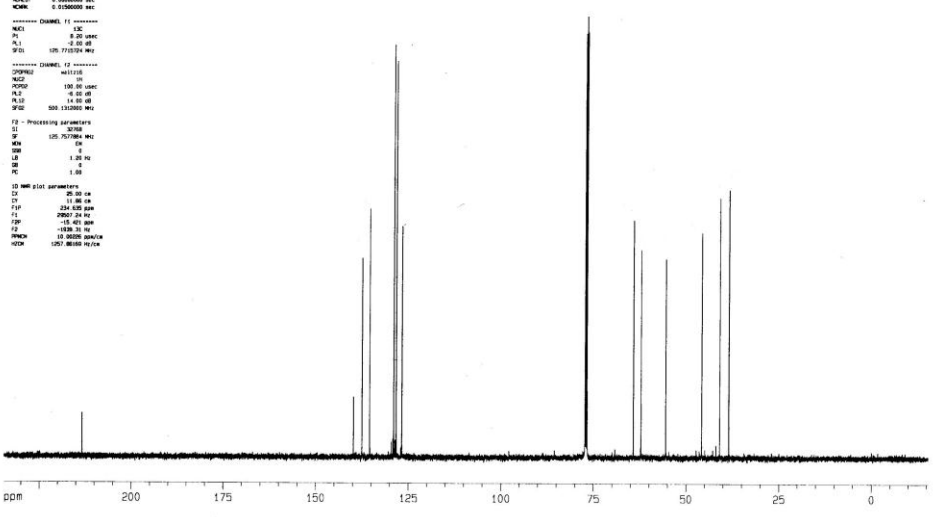

2
4
6
ppm
ppm
6
4
2

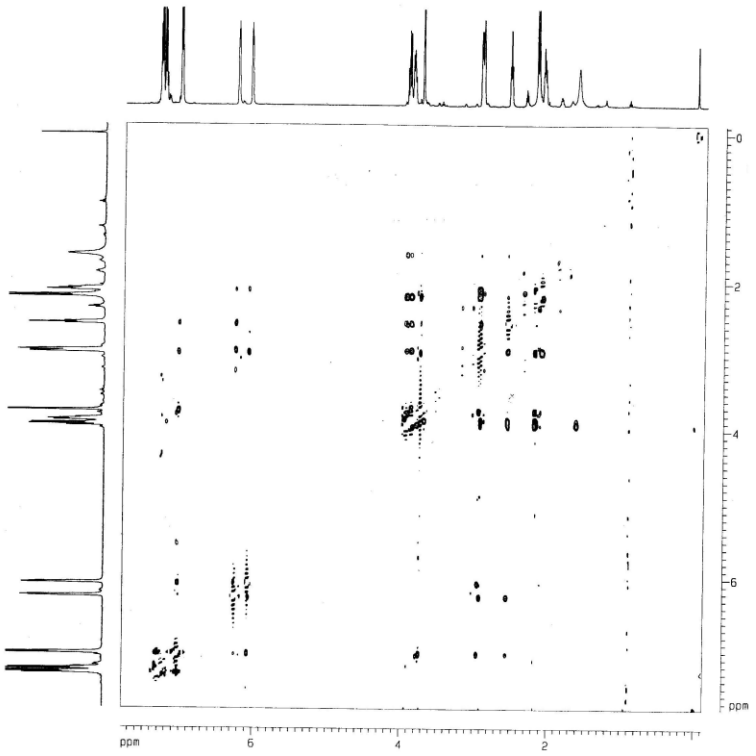

Current Data Parameters
NAME ki-423-f4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030704
Time 16.09
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 181
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.46000 ppm/cm
HZCM 138.05980 Hz/cm
OH
Ph
O
10w
Integral
3.2364
1.8852
1.9224
1.1431
0.9621
1.0207
1.0000
2.0110
0.9750
1.0530
1.0143
ppm
10
9
8
7
6
5
4
3
2
1
0

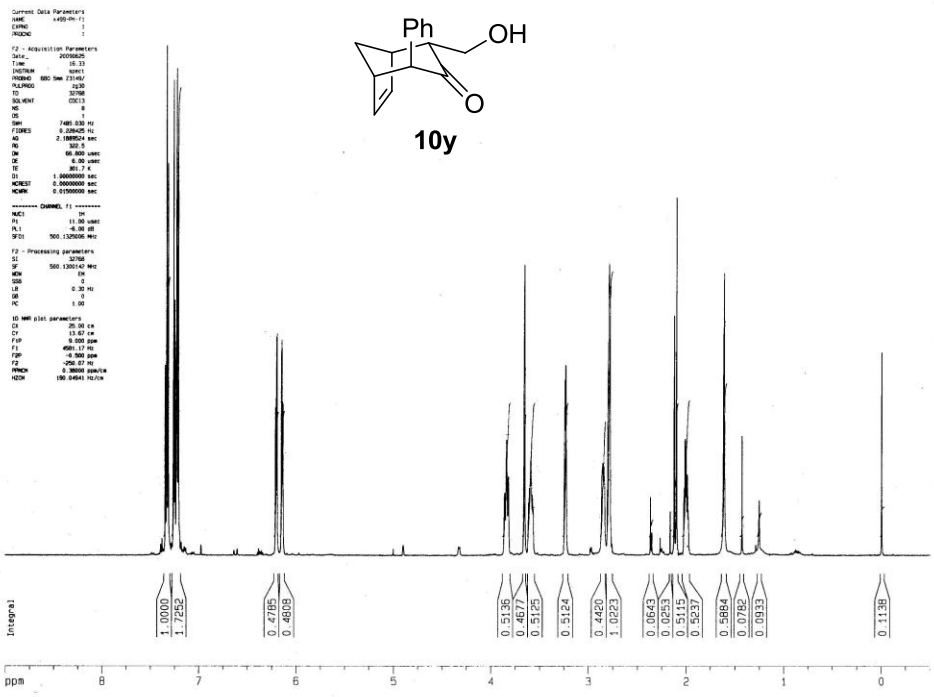

Current Data Parameters
NAME ki-423-f4
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030704
Time 16.13
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zgdc
TD 32768
SOLVENT CDCl3
NS 129
DS 3
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 8192
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.4760204 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677521 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1P 300.000 ppm
F1 22640.32 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 12.20000 ppm/cm
HZCM 920.70654 Hz/cm
ppm
212.328
138.447
135.481
135.268
129.208
128.227
126.924
77.454
77.030
76.606
63.160
62.619
56.167
47.113
45.026
42.824
ppm
225
200
175
150
125
100
75
50
25

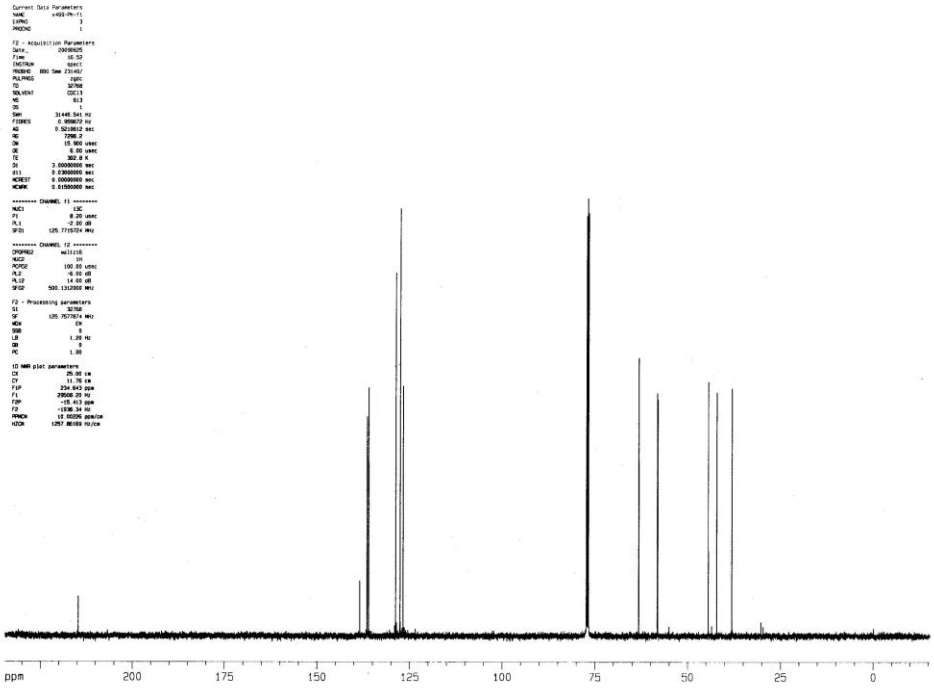

2
3
4
5
6
7
ppm
ppm
7
6
5
4
3
2

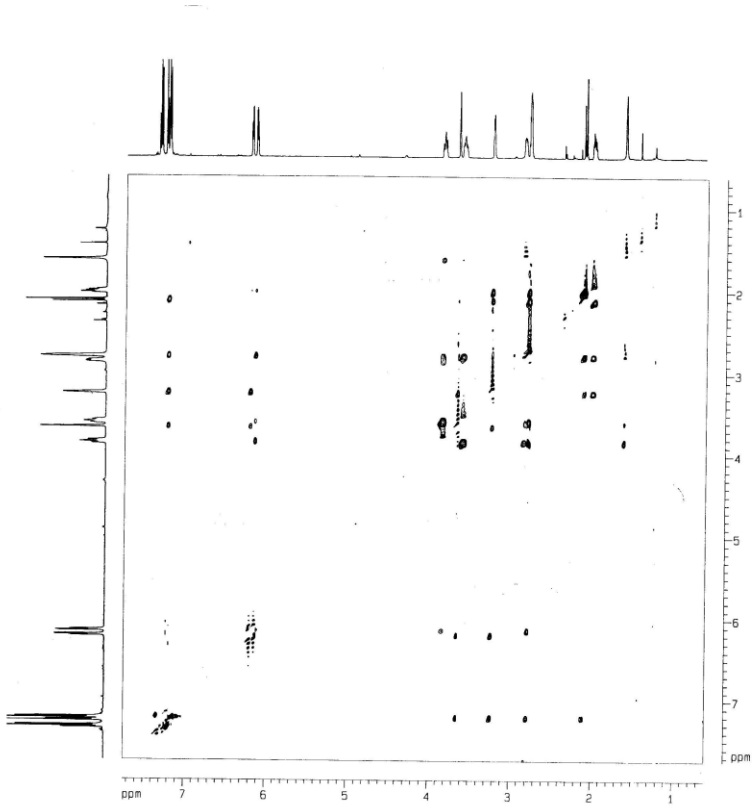

OH
Ph
O
10x
Integral
1.0000
0.6904
0.9347
0.4744
0.4631
0.5242
0.5103
0.4907
0.9485
0.4736
0.8318
0.5258
0.4470
ppm
8
7
6
5
4
3
2
1
0

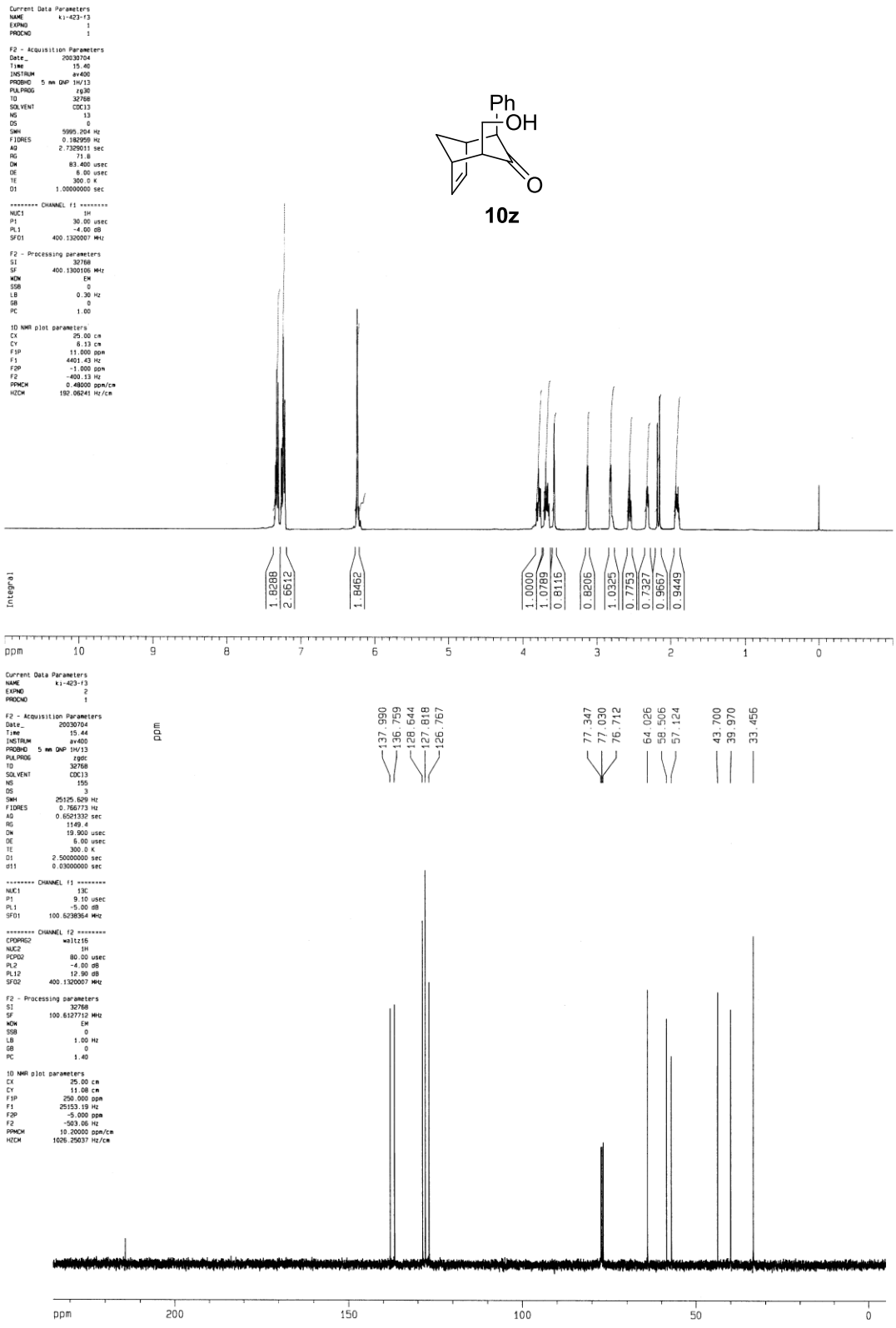

ppm
200
175
150
125
100
75
50
25
0

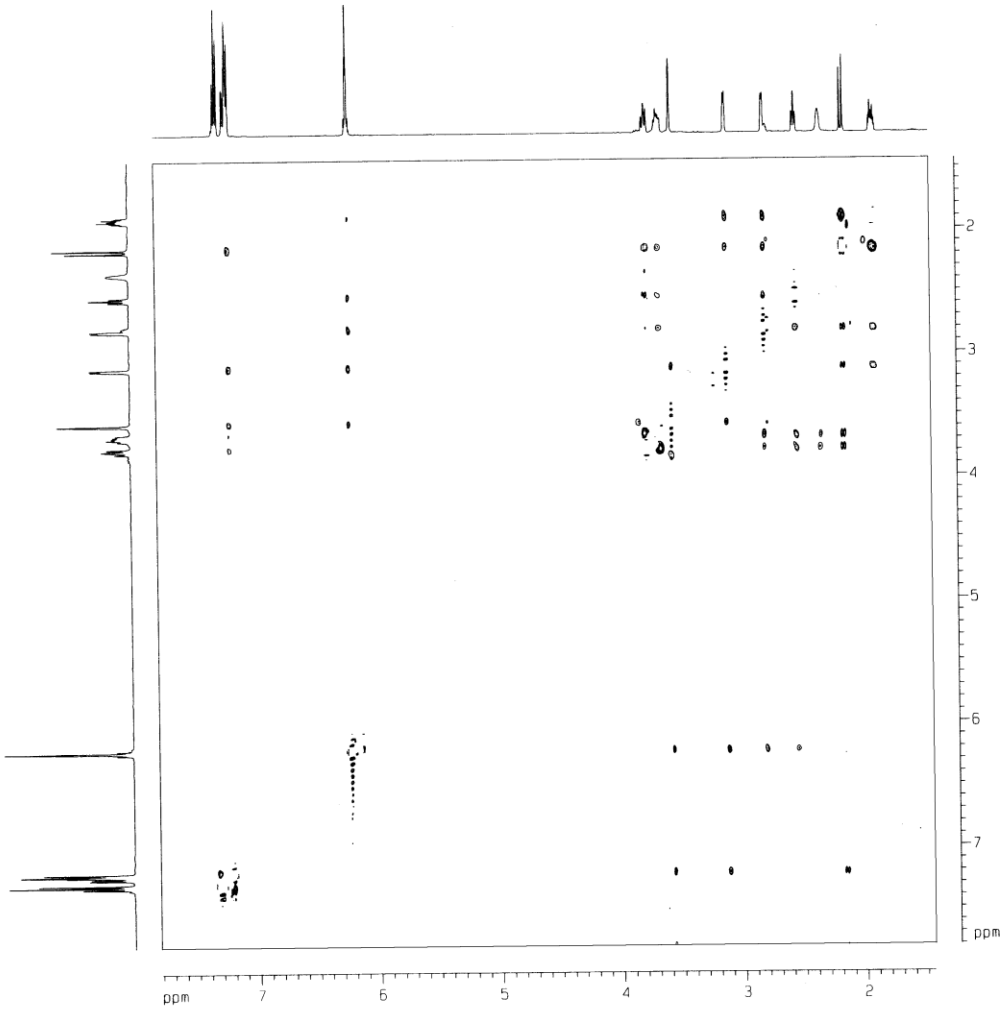

0
2
4
6
ppm
ppm
6
4
2


Ph
OH
O
10y
Integral
1.0000
1.7252
0.4785
0.4809
0.5136
0.4677
0.5125
0.5124
0.4420
1.0223
0.0643
0.0253
0.5115
0.5237
0.5884
0.0782
0.0933
0.1138
ppm
8
7
6
5
4
3
2
1
0


ppm
200
175
150
125
100
75
50
25
0


ppm
7
6
5
4
3
2
1
ppm
1
2
3
4
5
6
7


Ph
OH
O
10z
Integral
1.8288
2.6612
1.8462
1.0000
1.0789
0.8116
0.8206
1.0325
0.7753
0.7327
0.9667
0.9449
ppm
137.990
136.759
128.644
127.818
126.767
77.347
77.030
76.712
64.026
58.506
57.124
43.700
39.970
33.456


ppm
7
6
5
4
3
2
2
3
4
5
6
7
ppm