Unified Synthesis of 10-Oxygenated *Lycopodium* Alkaloids: Impact of C_{10}-Stereochemistry on Reactivity.

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Electronic Supplementary Information: NMR Spectra

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XL iv-30 sulfone

7.948 7.934 7.930 7.926 7.706 7.687 7.669 7.666 7.617 7.597 7.582 7.579 5.780 5.770 5.763 5.754 5.737 5.728 5.721 5.711 5.695 5.099 5.096 5.092 5.089 5.071 5.069 5.053 5.049 5.046 3.206 3.192 3.186 3.178 3.165 2.515 2.499 2.495 2.490 2.486 2.478 2.475 2.461 2.458

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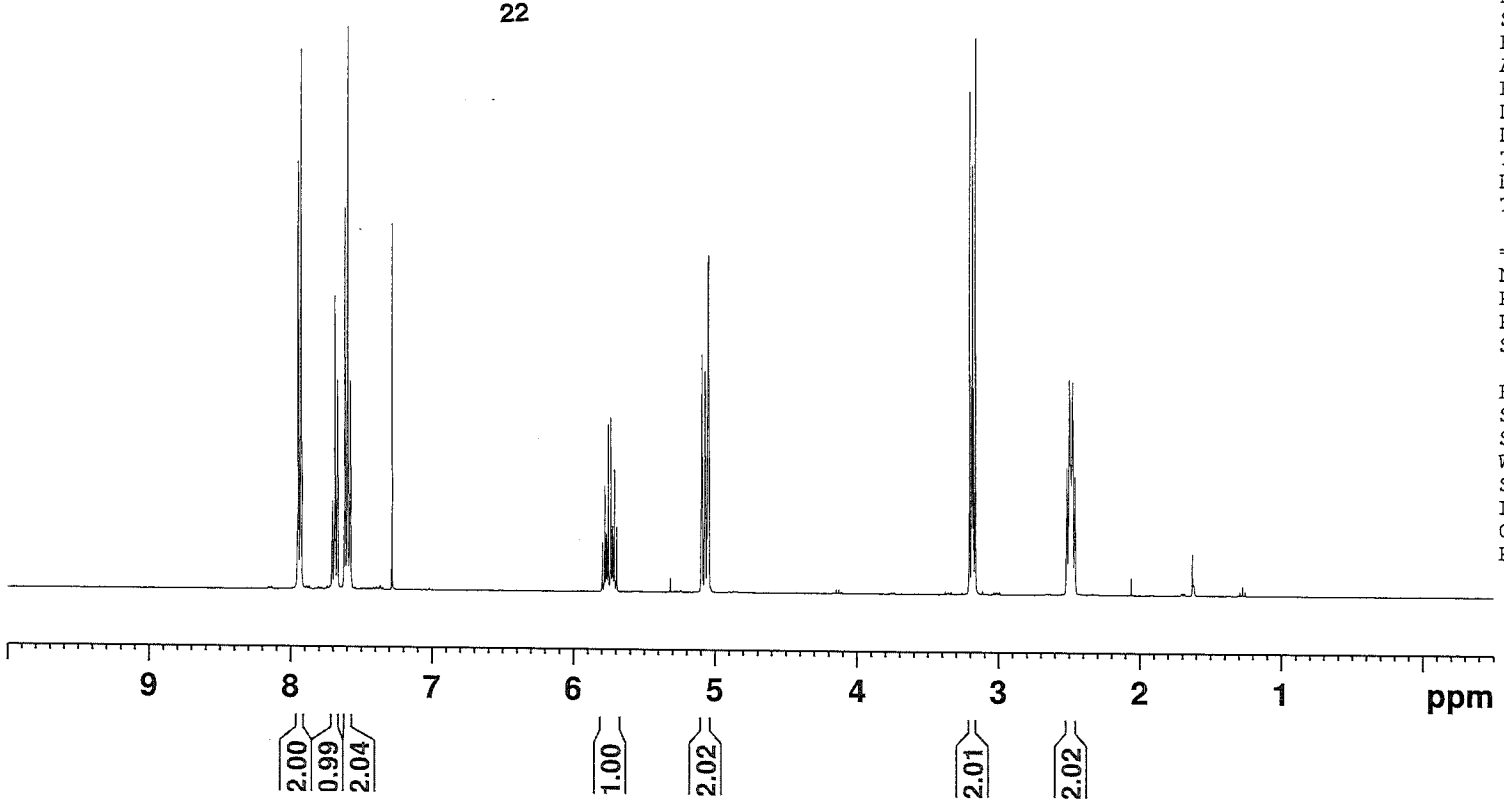


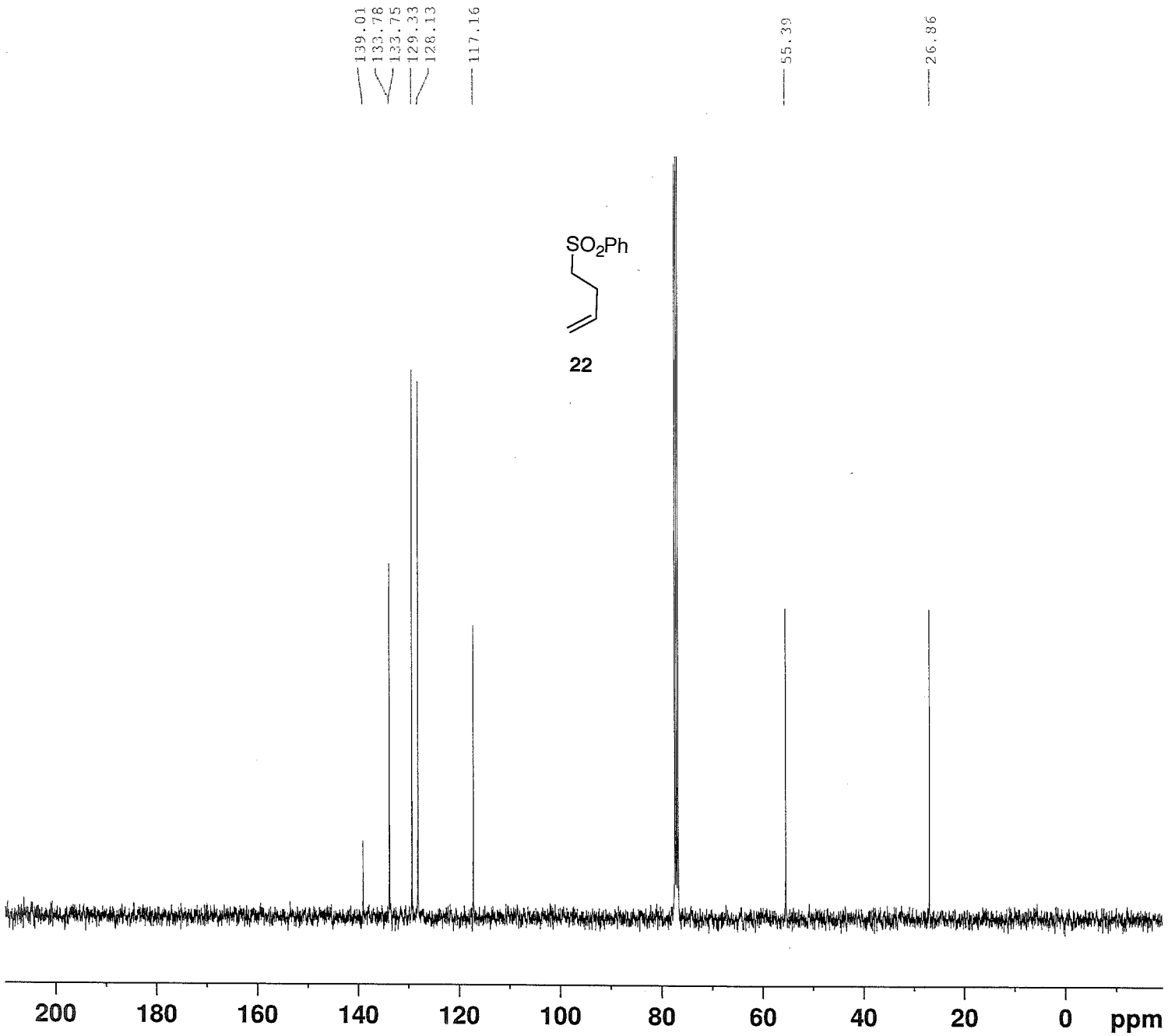
Current Data Parameters
NAME XL iv-30 sulfone
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150209
Time 18.01
INSTRUM robinson
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 17
DS 2
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2806528 sec
RG 114
DW 69.600 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 0 dB
SFO1 400.1428010 MHz

F2 - Processing parameters
SI 16384
SF 400.1400000 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

XL iv-30 sulfone carbon



Current Data Parameters
NAME XL iv-30 sulfone
EXPNO 3
PROCNO 1

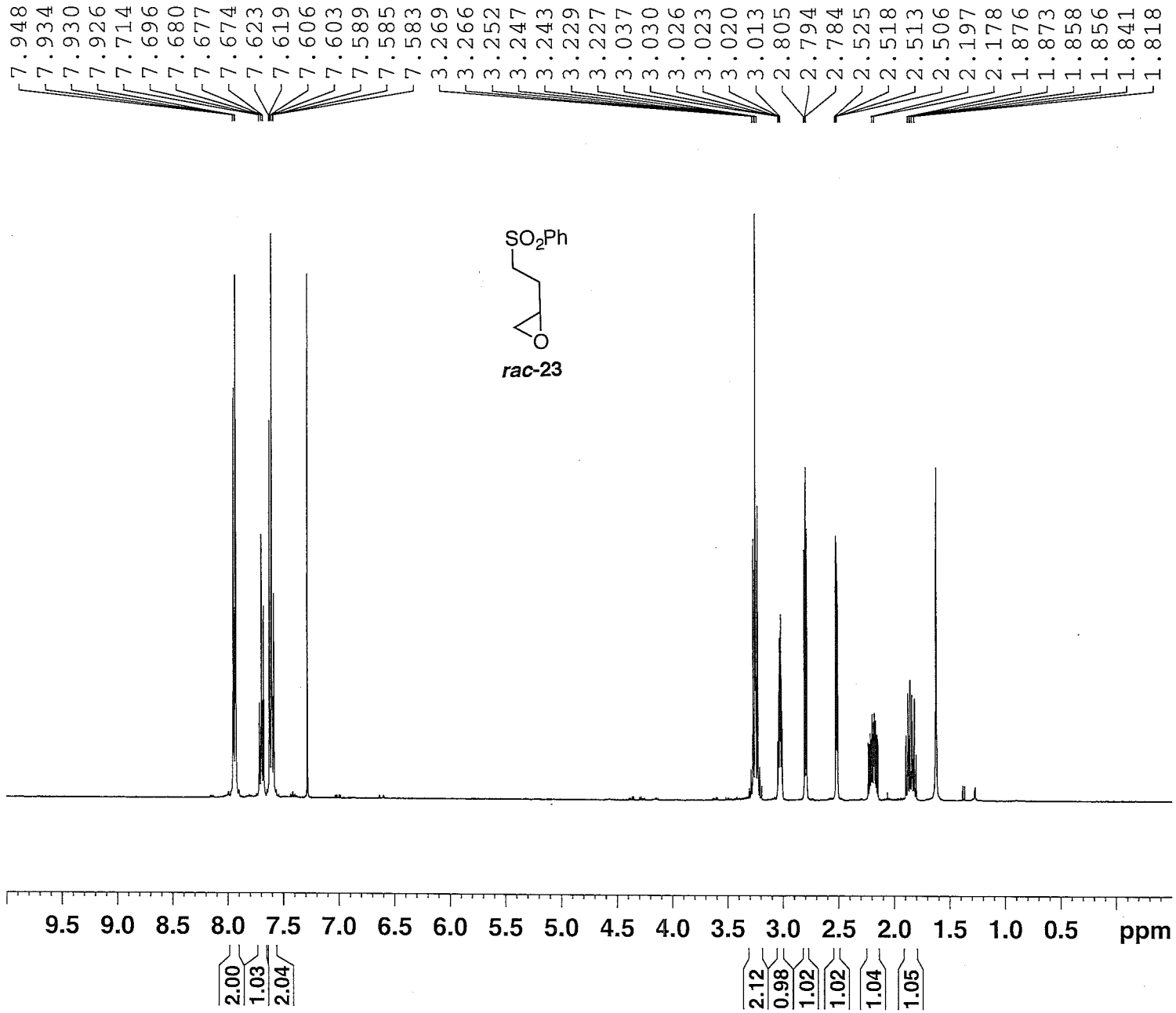
F2 - Acquisition Parameters
Date_ 20150209
Time 18.04
INSTRUM robinson
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 202
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.60 usec
PL1 -2.00 dB
SFO1 100.6253446 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0 dB
PL12 18.10 dB
PL13 17.00 dB
SFO2 400.1416006 MHz

F2 - Processing parameters
SI 32768
SF 100.6152830 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

XL i-07 epoxide




```
NAME        XL i-07 epoxide
EXPNO                     1
PROCNO                    1
Date_            20140113
Time                 10.59
INSTRUM          robinson
PROBHD     5 mm PABBO BB-
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      19
DS                       2
SWH              7183.908 Hz
FIDRES           0.219235 Hz
AQ              2.2807028 se
RG                     128
DW                  69.600 us
DE                    6.50 us
TE                   298.3 K
D1             2.00000000 se
TD0                      1

======== CHANNEL f1 ======
NUC1                    1H
P1                   11.20 us
PL1                   0.00 dB
SFO1          400.1428010 MH
SI                    16384
SF            400.1400000 MH
WDW                      EM
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```

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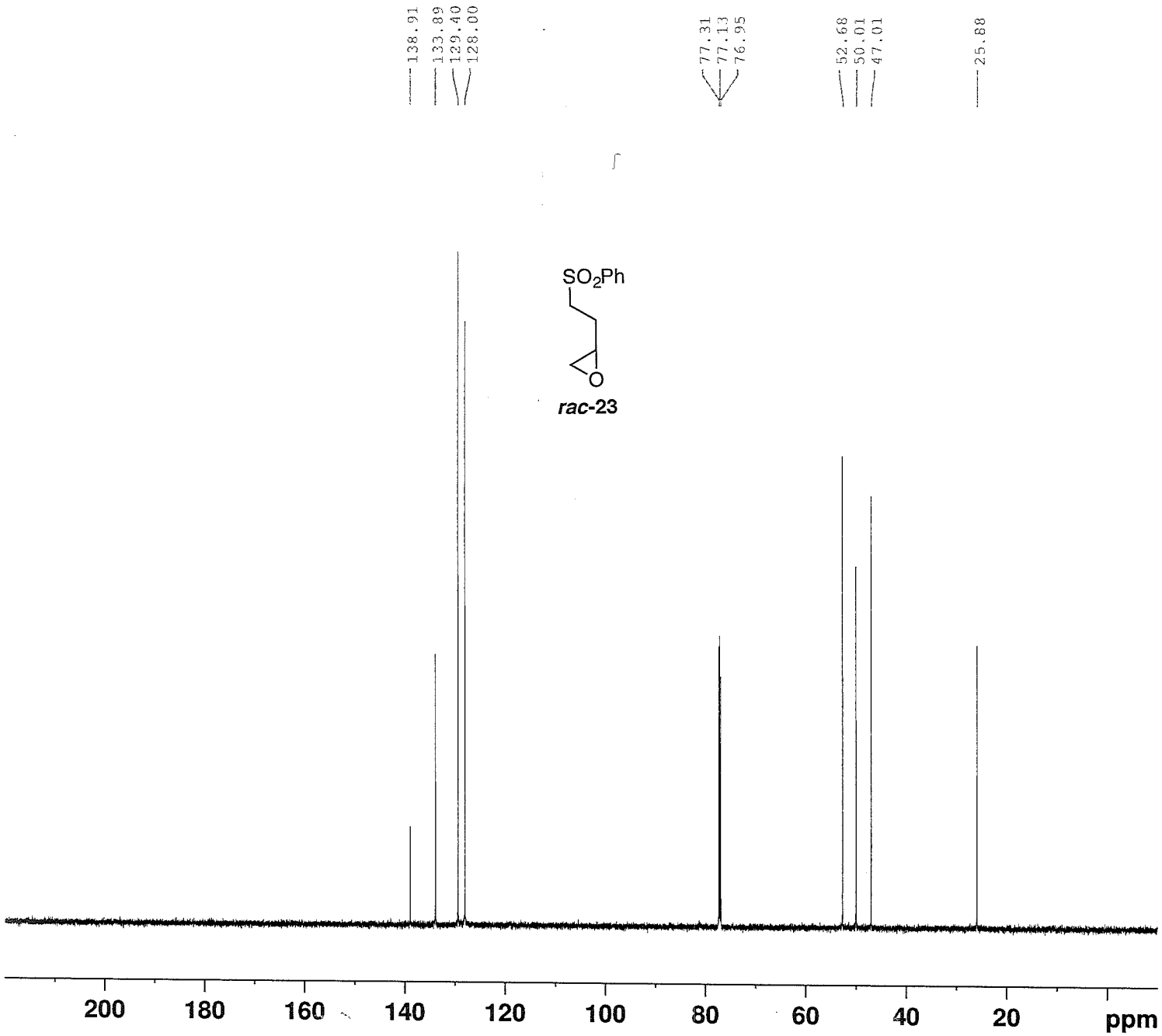
XL-viii-26-epoxide



BRUKER

Current Data Parameters
NAME XL-viii-26-epoxide
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151203
Time 16.18 h
INSTRUM spect
PROBHD Z566601_0014 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 42613.637 Hz
FIDRES 0.650232 Hz
AQ 0.7689557 sec
RG 203
DW 11.733 usec
DE 6.50 usec
TE 303.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.0345018 MHz
NUC1 13C
P1 21.00 usec
PLW1 150.00000000 W
SFO2 700.0098000 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 55.00 usec
PLW2 25.00000000 W
PLW12 0.41427001 W
PLW13 0.14935000 W

F2 - Processing parameters
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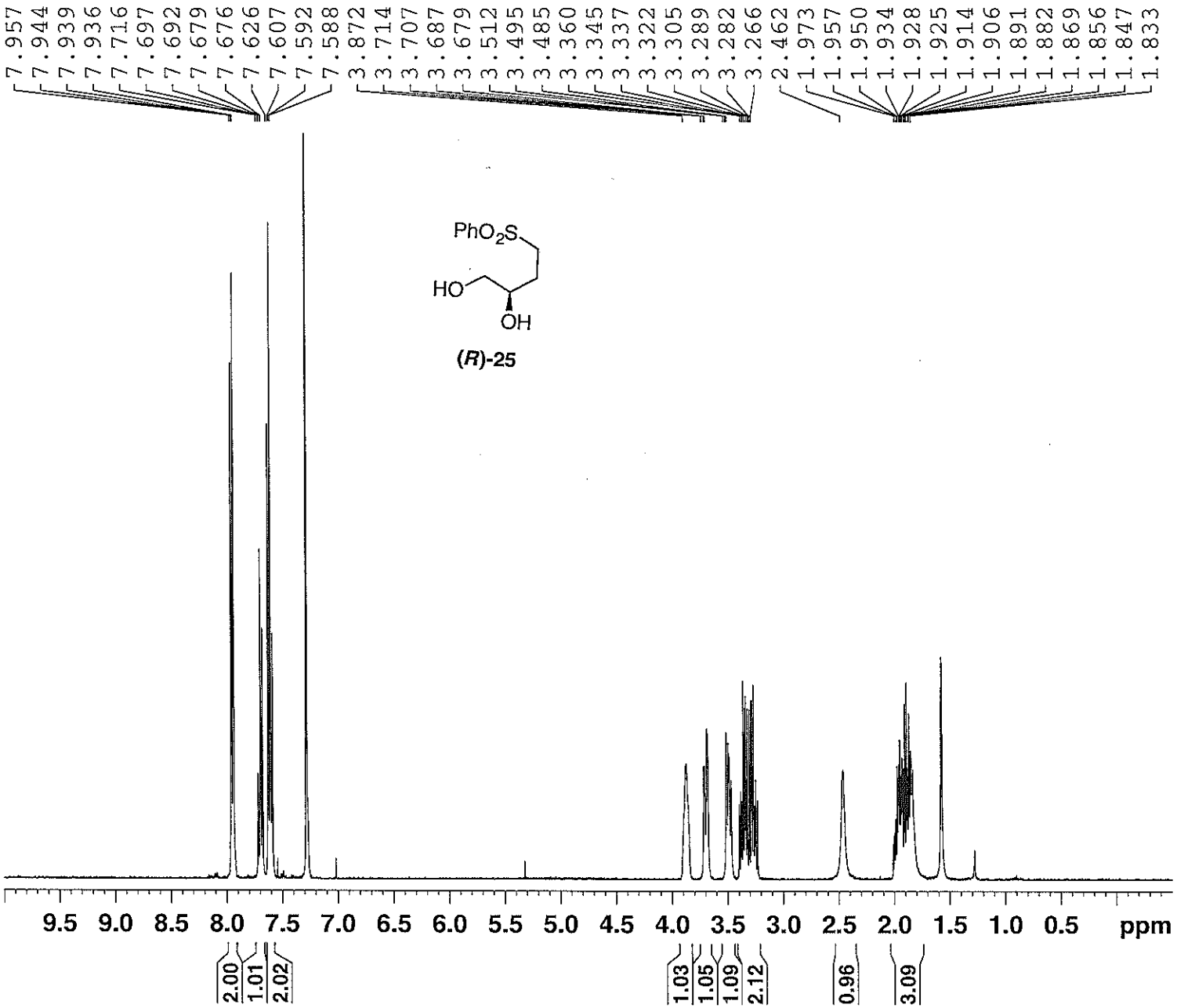
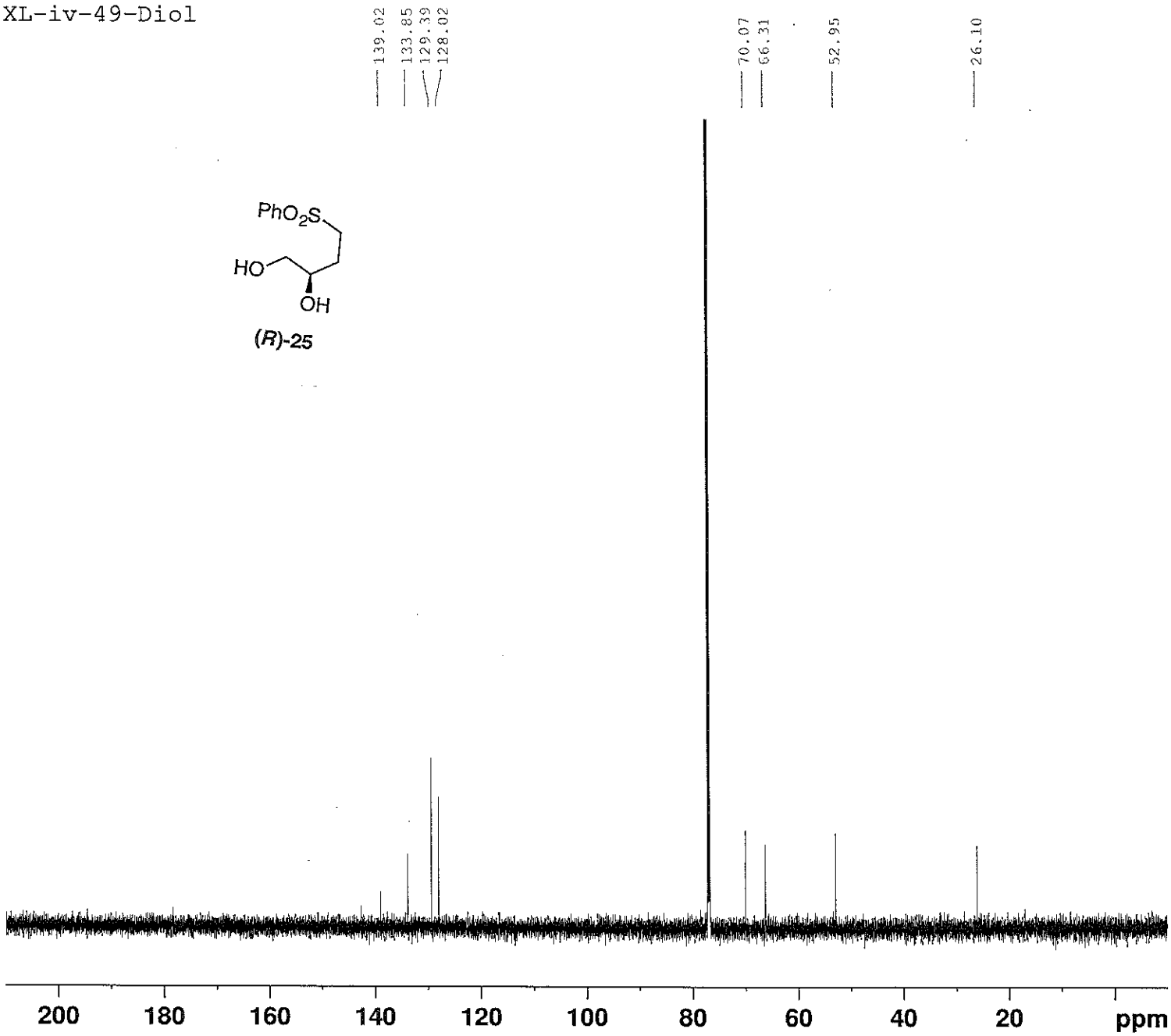
SF 176.0169002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

XL-iv-49-Diol



NAME	XL-iv-49-Diol
EXPNO	2
PROCNO	1
Date_	20150220
Time	23.30
INSTRUM	robinson
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	7183.908 Hz
FIDRES	0.219235 Hz
AQ	2.2807028 se
RG	362
DW	69.600 us
DE	6.50 us
TE	298.2 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	11.20 us
PL1	0.00 dB
SFO1	400.1428010 MH
SI	16384
SF	400.1400000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

XL-iv-49-Diol

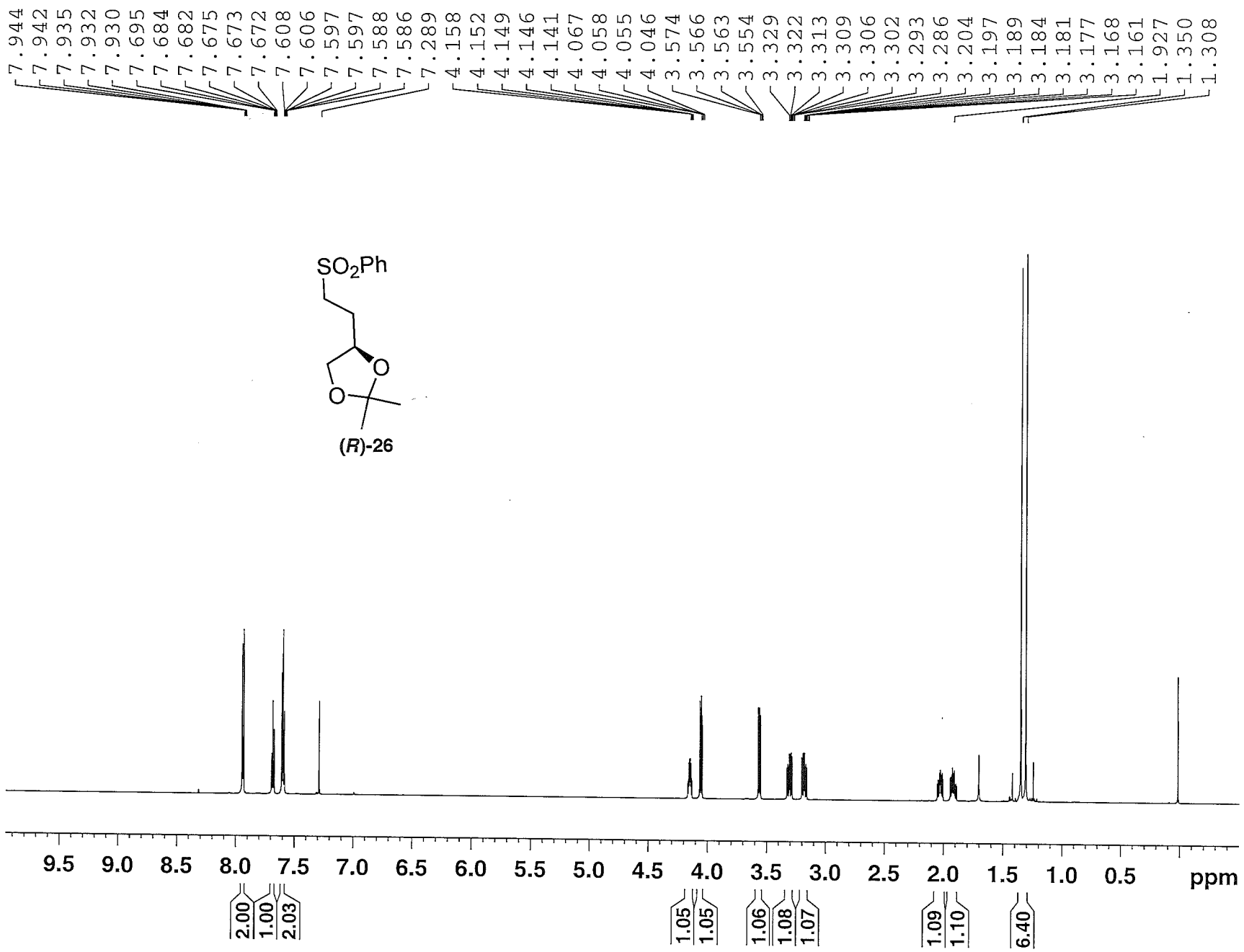


Current Data Parameters
NAME XL-iv-49-Diol
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160606
Time 18.08 h
INSTRUM spect
PROBHD Z44908_0013 (C
PULPROG zgpg30
TD 65356
SOLVENT CDCl3
NS 801
DS 4
SWH 42613.637 Hz
FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 203
DW 11.733 usec
DE 18.00 usec
TE 305.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.0345018 MHz
NUC1 13C
P1 12.80 usec
PLW1 220.00000000 W
SFO2 700.0098000 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 10.00000000 W
PLW12 0.15148000 W
PLW13 0.07627600 W

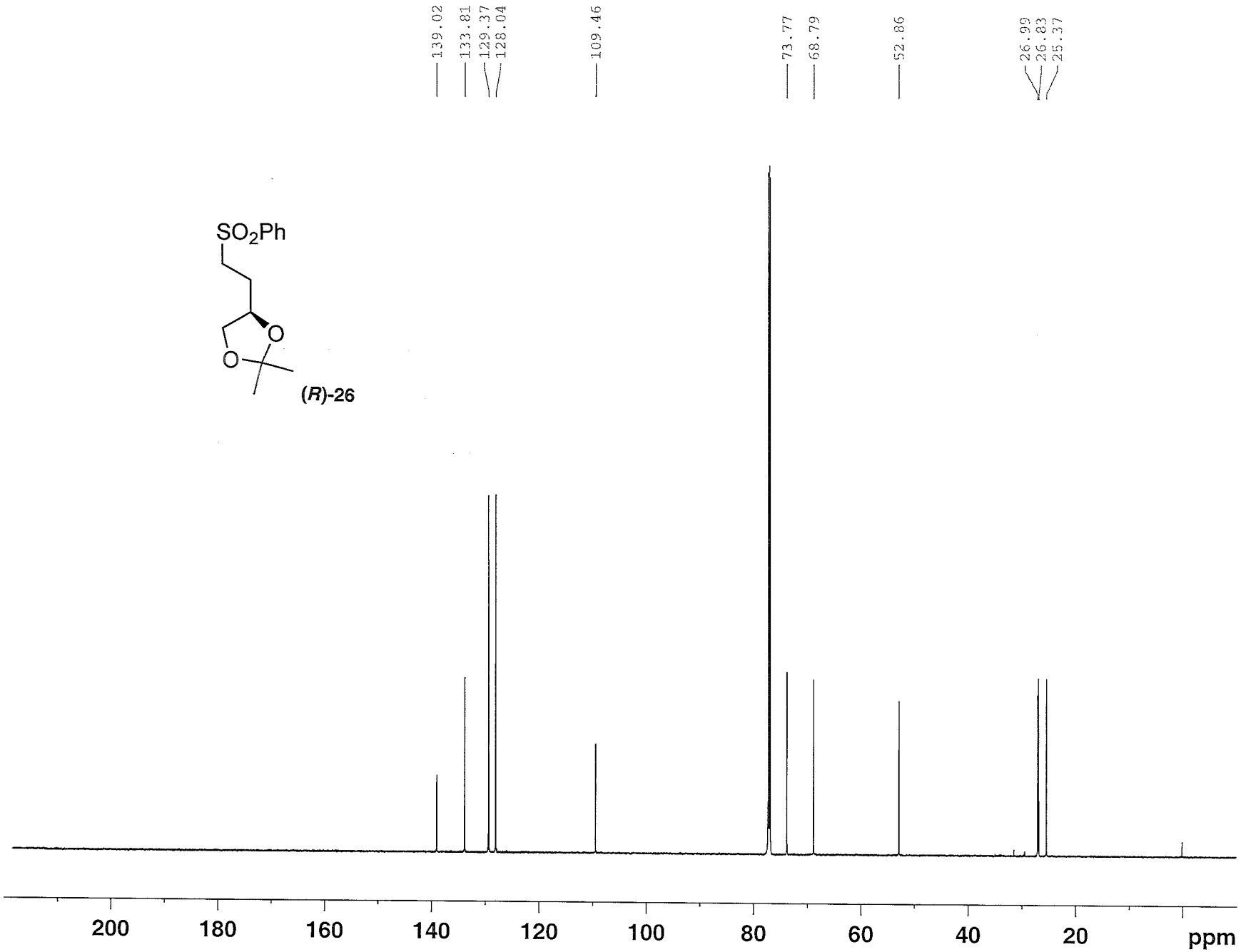
F2 - Processing parameters
SI 32768
SF 176.0169002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Acetonide



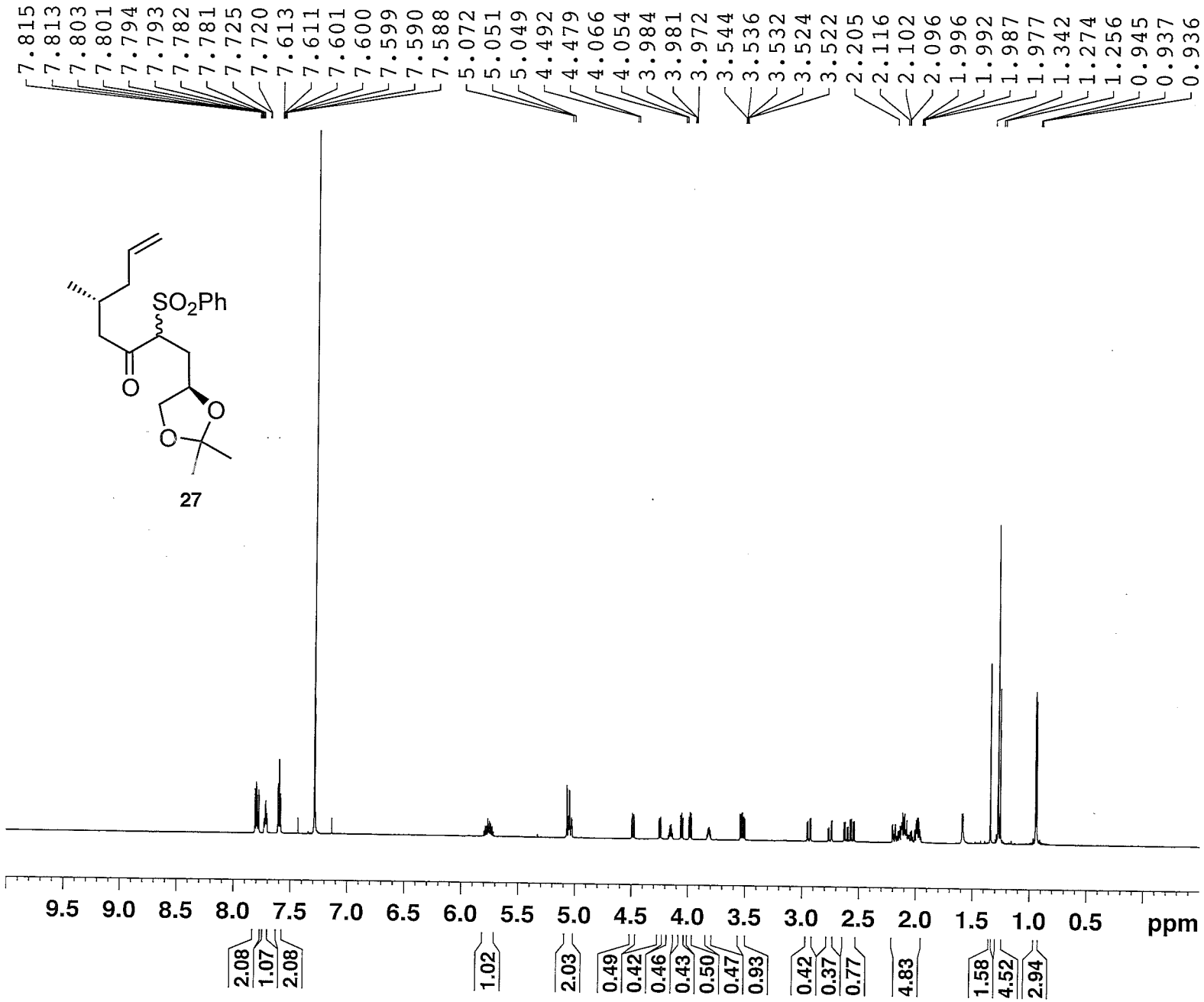

```
NAME          MS III-08
EXPNO                 1
PROCNO
Date_          20100915
Time             15.2
INSTRUM          spec
PROBHD   5 mm CPDCH 13C
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    2
SWH           11904.762 H
FIDRES         0.181652 H
AQ            2.7525620 s
RG                 40.3
DW                42.000 u
DE                 16.50 u
TE                 297.9 K
D1          2.00000000 s
TD0                   1

======== CHANNEL f1 =====
NUC1                 1H
P1                9.40 u
PL1              -3.20 d
PL1W        33.59817505 W
SFO1        700.1245508 M
SI                65536
SF          700.1200000 M
WDW                  EM
SSB                   0
LB                 0.30 H
GB                    0
PC                 1.00
```

Acetonide carbon




```
NAME          MS III-08
EXPNO                 2
PROCNO
Date_          20100916
Time              15.28
INSTRUM           spect
PROBHD   5 mm CPDCH 1H
PULPROG         zgpg30
TD              98304
SOLVENT         CDCl3
NS                 60
DS                  4
SWH           41666.668 H
FIDRES         0.423855 H
AQ            1.1796980 s
RG                203
DW               12.000 u
DE               15.00 u
TE               298.0 K
D1          2.00000000 s
D11         0.03000000 s
TD0                 1

======== CHANNEL f1 =====
NUC1                13C
P1                 9.00 u
PL1                4.50 d
PL1W       38.14553833 W
SFO1       176.0629186 M

======== CHANNEL f2 =====
CPDPRG2          waltz16
NUC2                1H
PCPD2             65.00 u
PL2               -3.20 d
PL12              13.60 d
PL13             120.00 d
PL2W       33.59817505 W
PL12W       0.70196527 W
PL13W       0.00000000 W
SFO2       700.1228005 M
SI              131072
SF         176.0453140 M
WDW                EM
SSB                 0
LB               3.00 H
GB                  0
PC               1.40
```

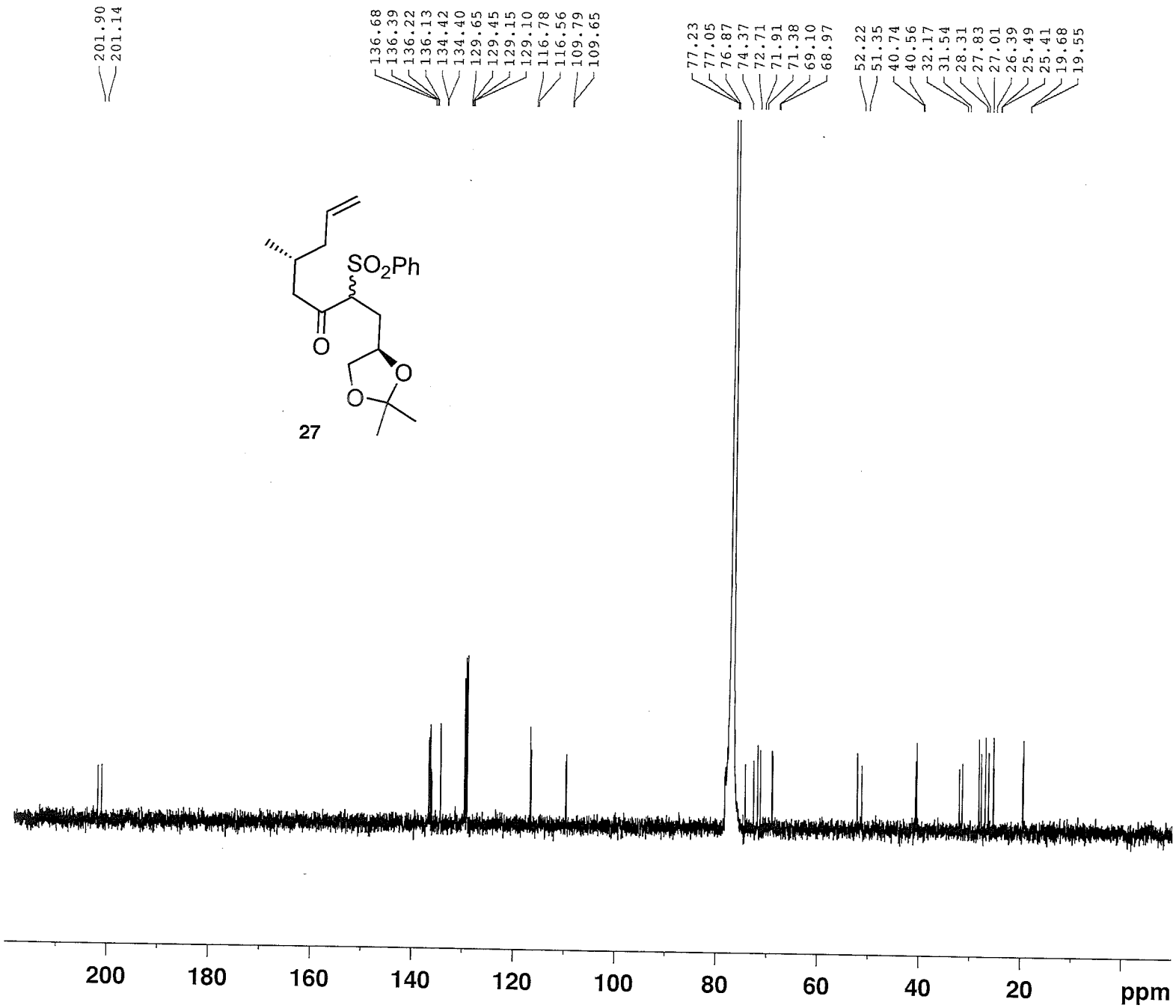
Julia coupling



NAME	MS III-84	
EXPNO	1	
PROCNO	1	
Date_	20110127	
Time	16.44	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	19	
DS	2	
SWH	11904.762	Hz
FIDRES	0.181652	Hz
AQ	2.7525620	se
RG	101	
DW	42.000	us
DE	16.50	us
TE	298.2	K
D1	2.00000000	se
TD0	1	
======== CHANNEL f1 ======		
NUC1	1H	
P1	9.40	us
PL1	-3.20	dB
PL1W	33.59817505	W
SFO1	700.1245508	MH
SI	65536	
SF	700.1200000	MH
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

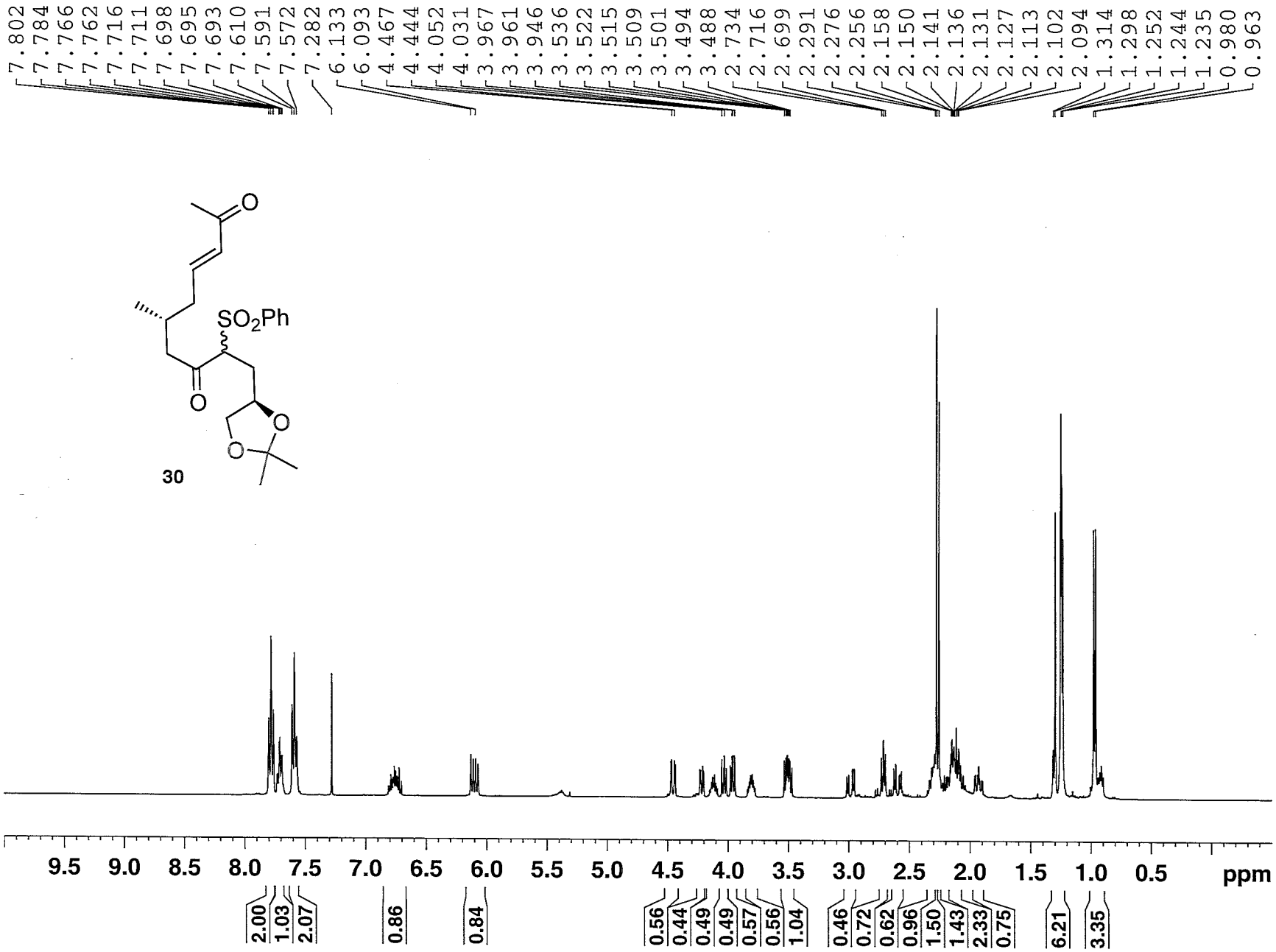
S 11

Julia coupling carbon



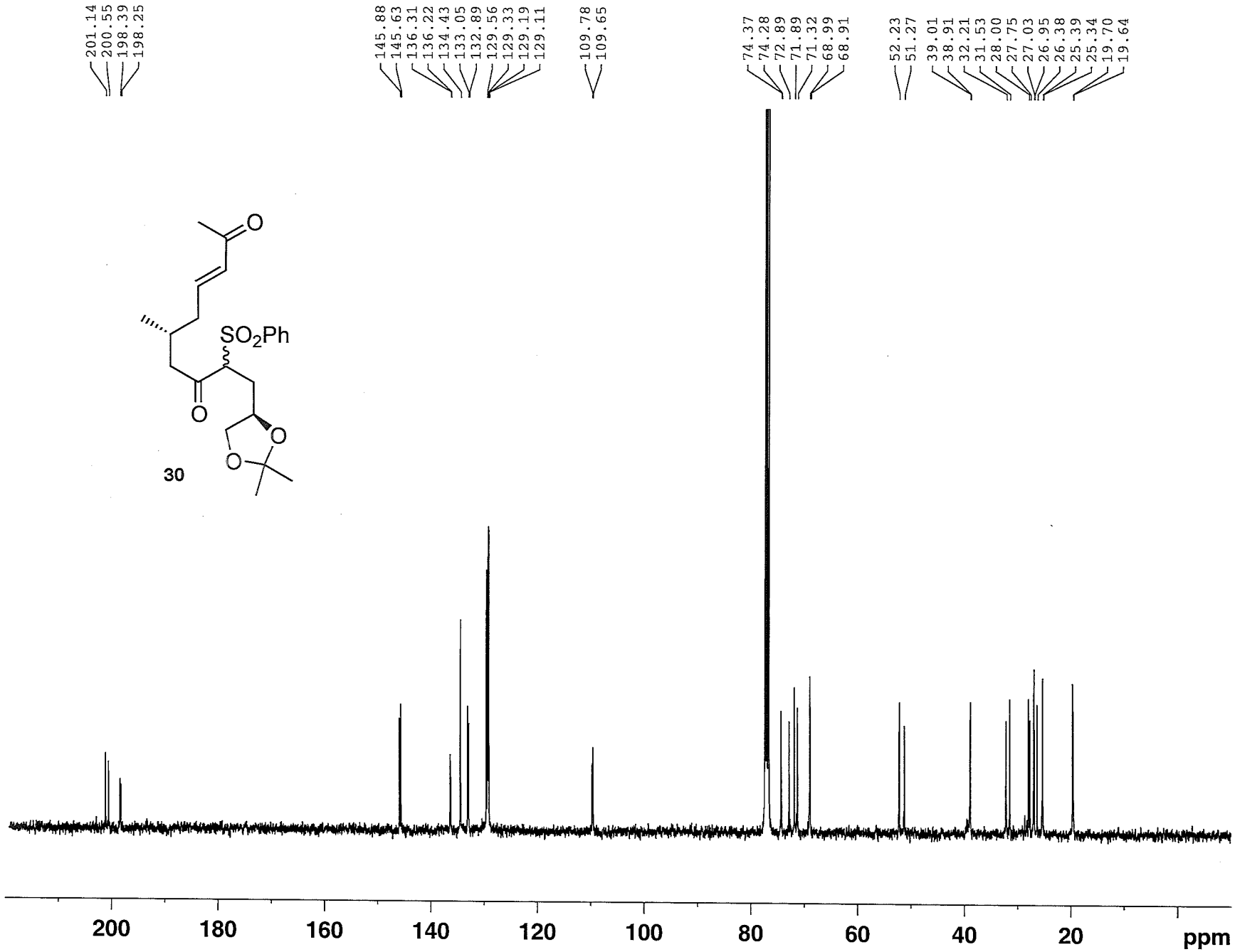
NAME	MS III-84
EXPNO	2
PROCNO	1
Date_	20110127
Time	17.43
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	98304
SOLVENT	CDCl3
NS	2048
DS	4
SWH	41666.668 Hz
FIDRES	0.423855 Hz
AQ	1.1796980 se
RG	203
DW	12.000 us
DE	15.00 us
TE	298.2 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.00 us
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0629186 MH
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 us
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1228005 MH
SI	131072
SF	176.0453140 MH
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Enone check



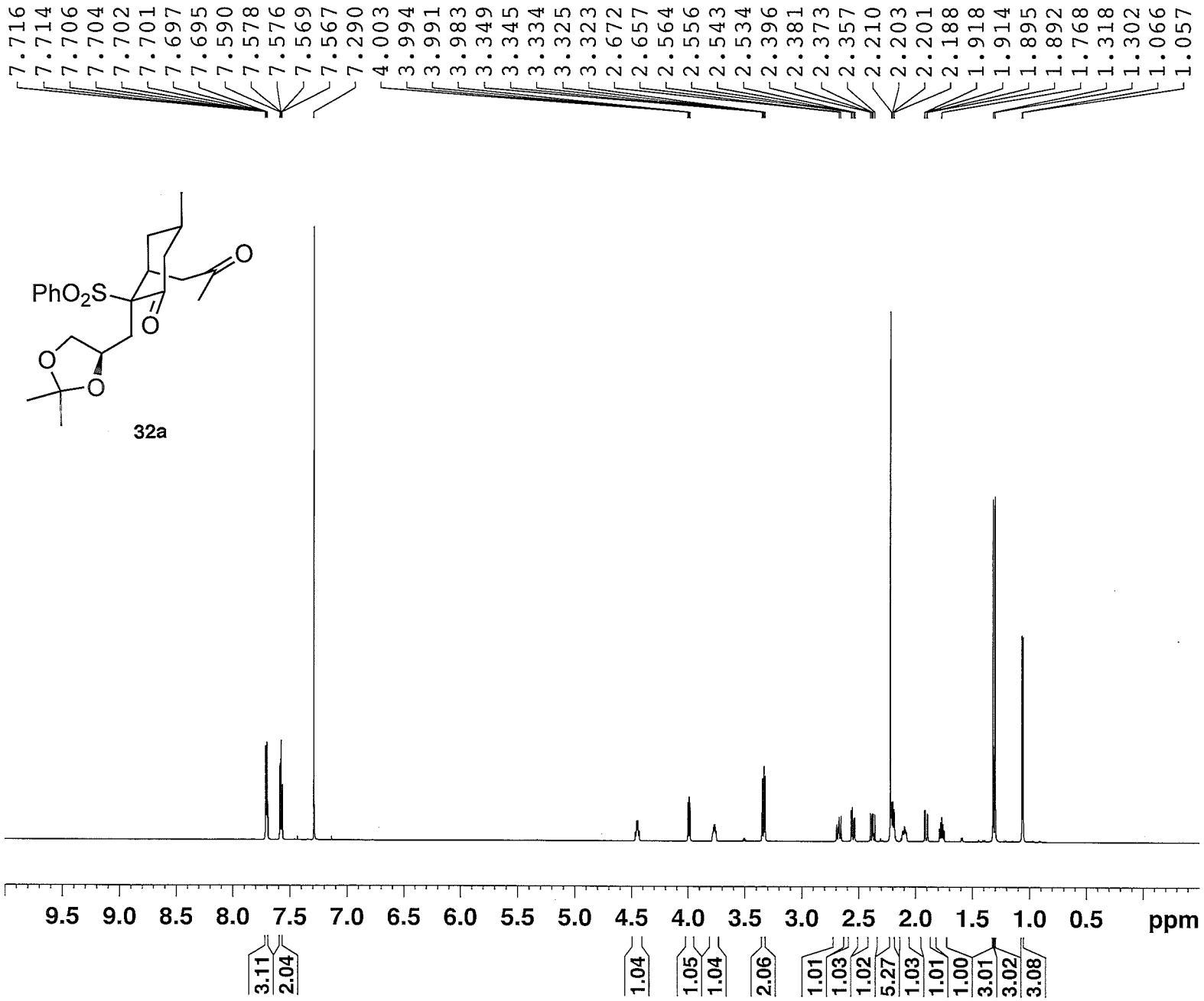

```
NAME        MS IX-17 Enone
EXPNO                    3
PROCNO                   1
Date_           20120905
Time                 1.05
INSTRUM         robinson
PROBED   5 mm PABBO BB-
PULPROG             zg30
TD                  32768
SOLVENT             CDCl3
NS                     10
DS                      2
SWH              7183.908 H
FIDRES           0.219235 H
AQ             2.2807028 s
RG                     64
DW                69.600 u
DE                  6.50 u
TE                 298.3 K
D1             2.00000000 s
TD0                     1

======== CHANNEL f1 =====
NUC1                   1H
P1                 14.00 u
PL1                 0.00 d
SFO1          400.1428010 M
SI                  32768
SF            400.1400000 M
WDW                    EM
SSB                     0
LB                   0.30 H
GB                      0
PC                   1.00
```

Enone carbon



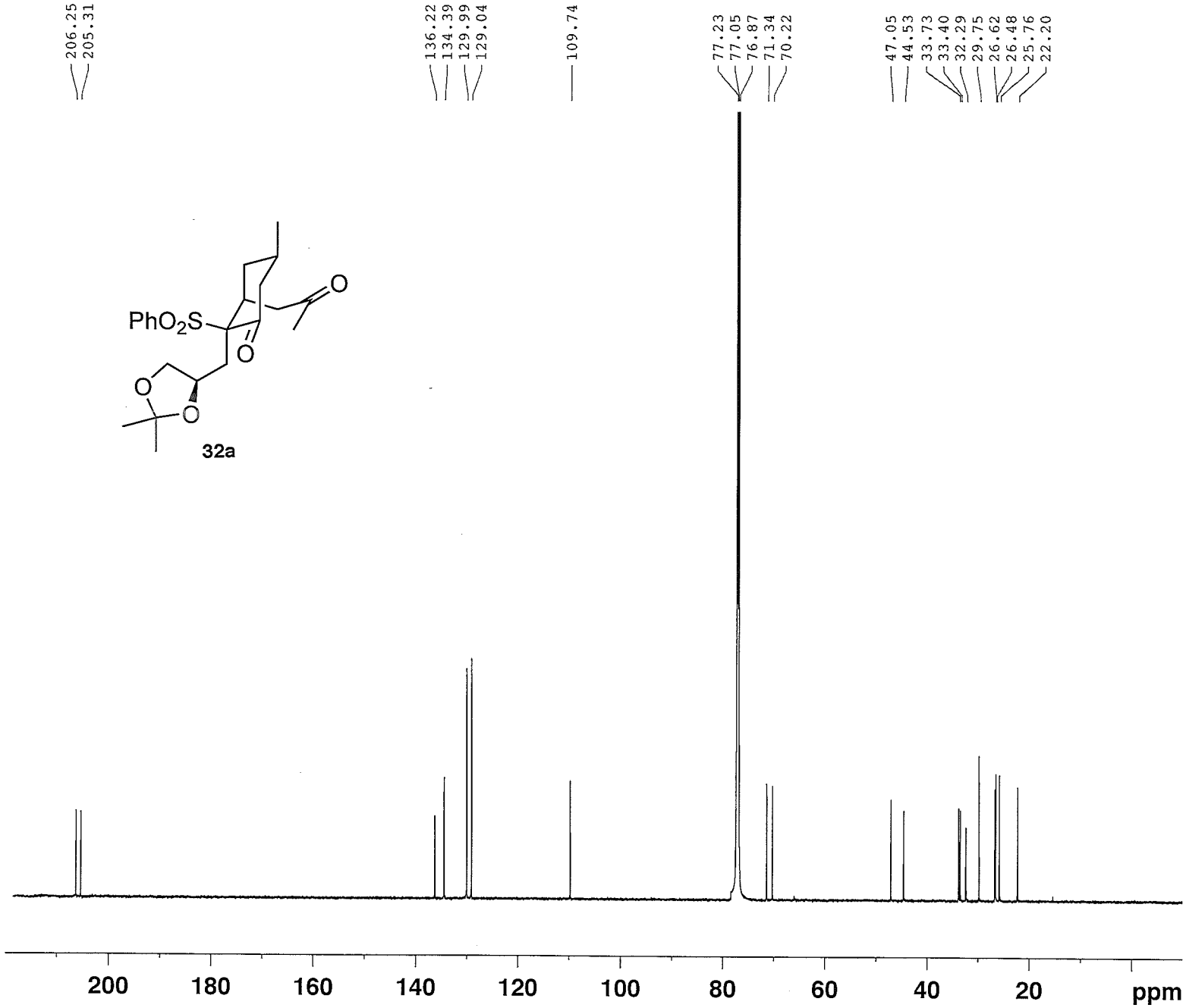

```
NAME        MS IX-17 Enone
EXPNO                    4
PROCNO                   1
Date_             20120909
Time                  1.09
INSTRUM          robinson
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1500
DS                       4
SWH              23980.814 Hz
FIDRES            0.365918 Hz
AQ               1.3664756 se
RG                   16384
DW                  20.850 us
DE                    6.50 us
TE                   298.8 K
D1             1.00000000 se
D11            0.03000000 se
TD0                      1

======== CHANNEL f1 ======
NUC1                   13C
P1                    9.00 us
PL1                  -2.00 dB
SFO1          100.6253446 MH

======== CHANNEL f2 ======
CPDPRG2             waltz16
NUC2                    1H
PCPD2                90.00 us
PL2                   0.00 dB
PL12                 16.16 dB
PL13                 17.00 dB
SFO2          400.1416006 MH
SI                   32768
SF            100.6152830 MH
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40
```

Michael Reaction



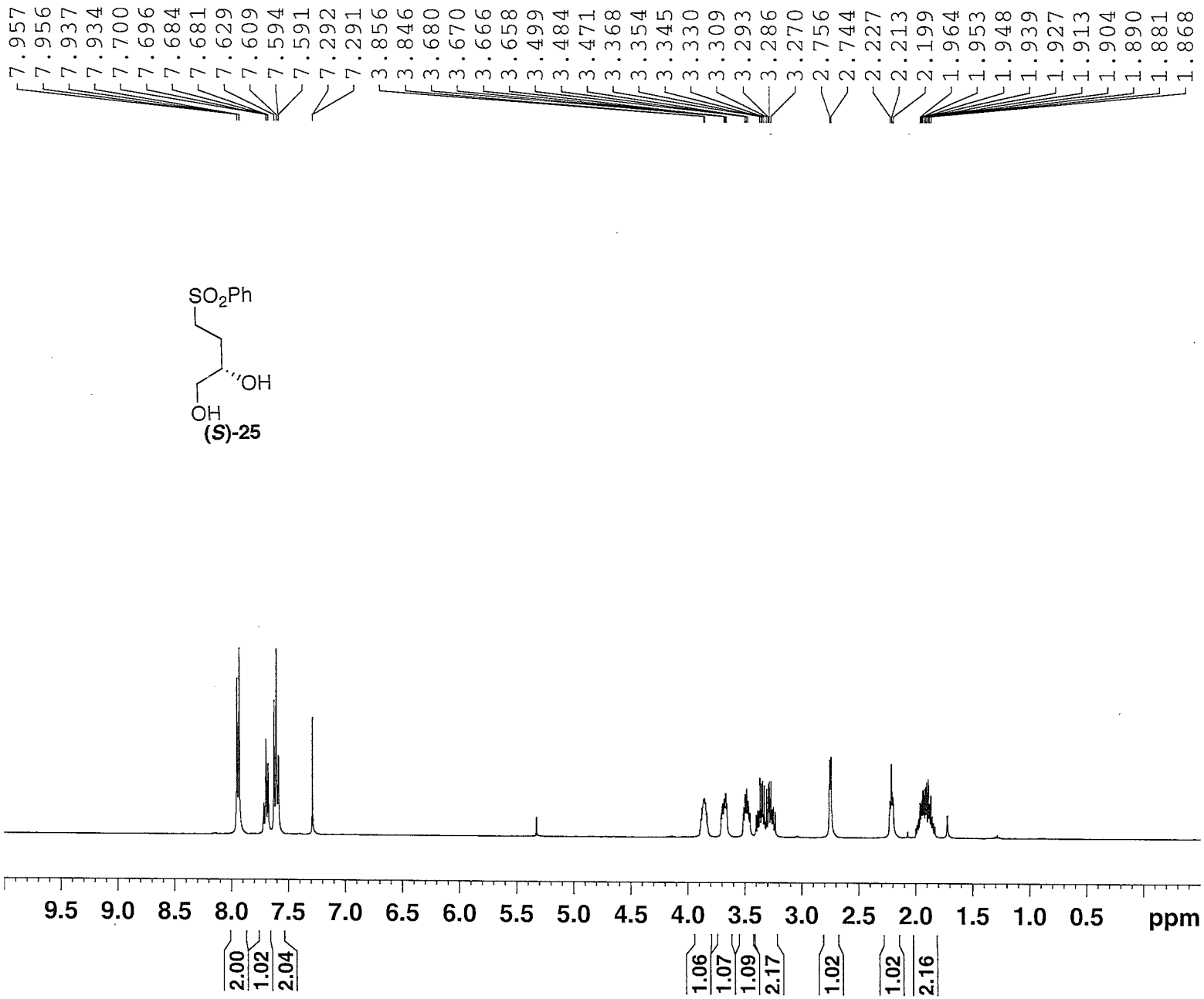
NAME	MS IV-08-L-HC cyclizati
EXPNO	1
PROCNO	1
Date_	20110129
Time	18.11
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	31
DS	2
SWH	11904.762 Hz
FIDRES	0.181652 Hz
AQ	2.7525620 sec
RG	64
DW	42.000 usec
DE	16.50 usec
TE	298.2 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1245508 MHz
SI	65536
SF	700.1200000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Michael reaction carbon




```
NAME        MS IV-08-L-HC cyclizati
EXPNO                          2
PROCNO                         1
Date_                   20110129
Time                       18.15
INSTRUM                    spect
PROBHD      5 mm CPDCH 13C
PULPROG                   zgpg30
TD                         98304
SOLVENT                    CDCl3
NS                          4000
DS                             4
SWH                    41666.668 Hz
FIDRES                  0.423855 Hz
AQ                     1.1796980 sec
RG                           203
DW                        12.000 usec
DE                         15.00 usec
TE                         298.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                          9.00 usec
PL1                         4.50 dB
PL1W                 38.14553833 W
SFO1                176.0629186 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      65.00 usec
PL2                        -3.20 dB
PL12                       13.60 dB
PL13                      120.00 dB
PL2W                 33.59817505 W
PL12W                 0.70196527 W
PL13W                 0.00000000 W
SFO2                700.1228005 MHz
SI                        131072
SF                  176.0453140 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.40
```

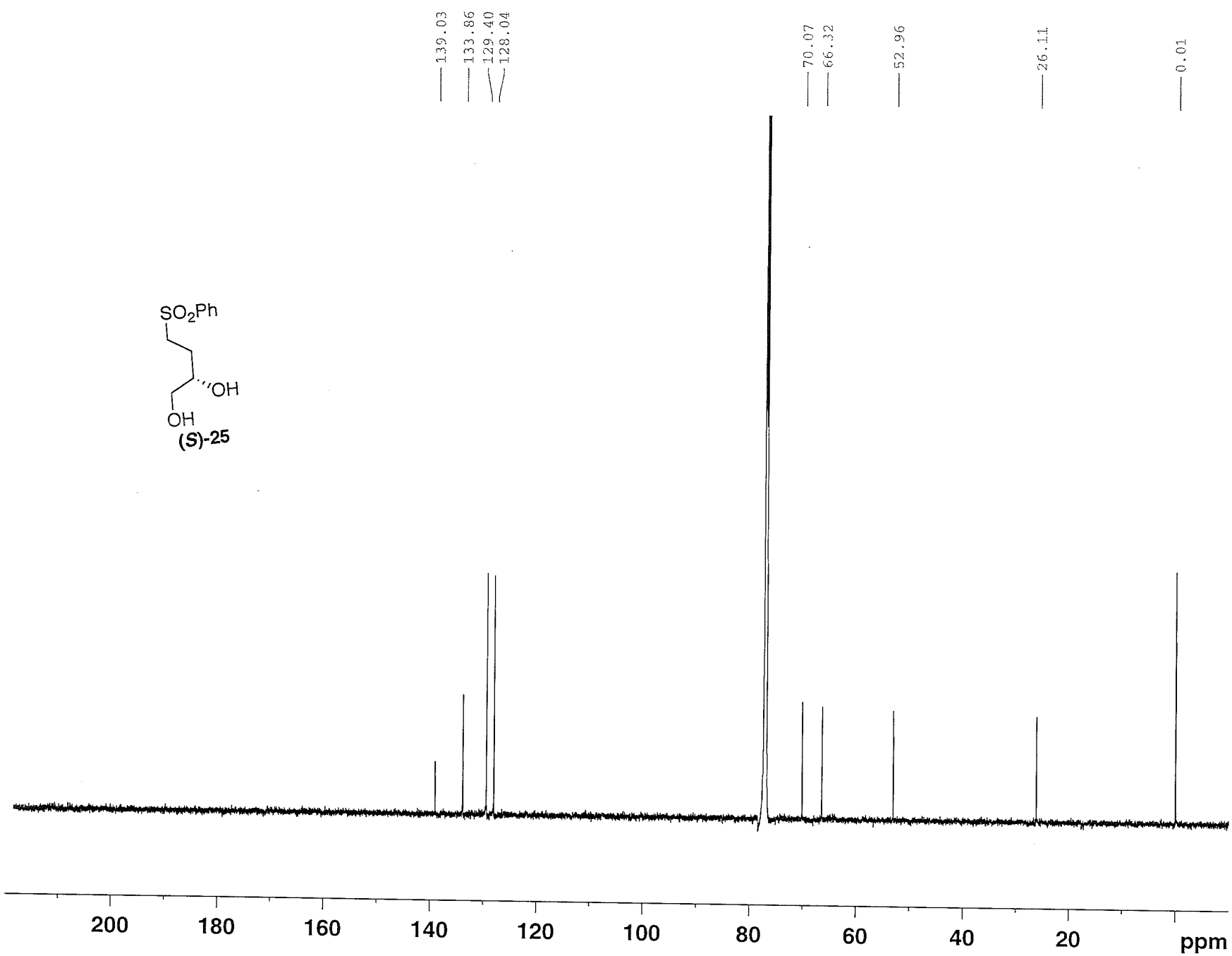
(S)-C10-Diol



NAME	MSII13
EXPNO	1
PROCNO	1
Date_	20100208
Time	11.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 se
RG	181
DW	78.000 us
DE	6.50 us
TE	299.4 K
D1	1.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.00 us
PL1	0.00 dB
PL1W	10.27361584 W
SFO1	400.1378009 MH
SI	32768
SF	400.1350000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

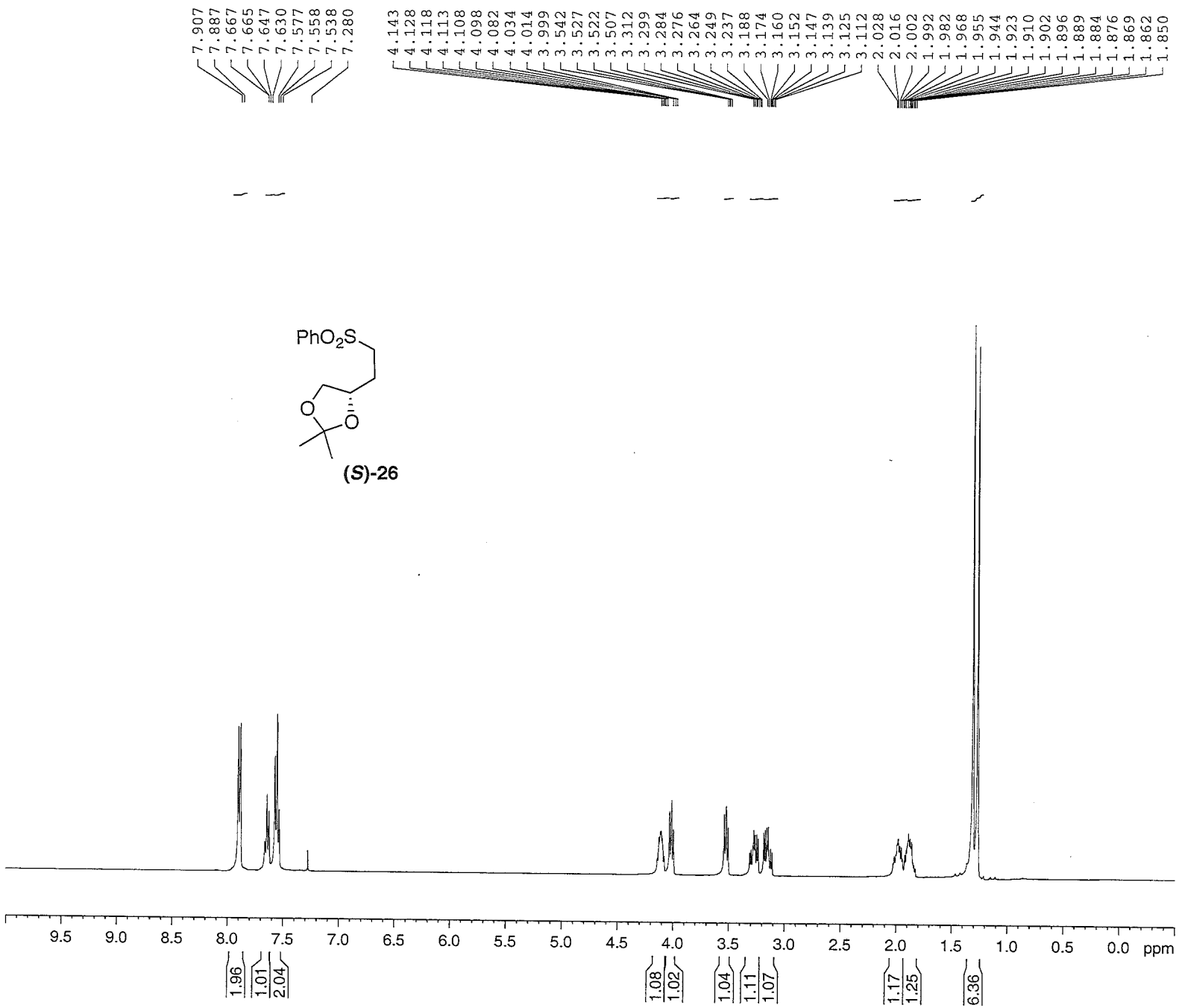
S 17

HKR carbon



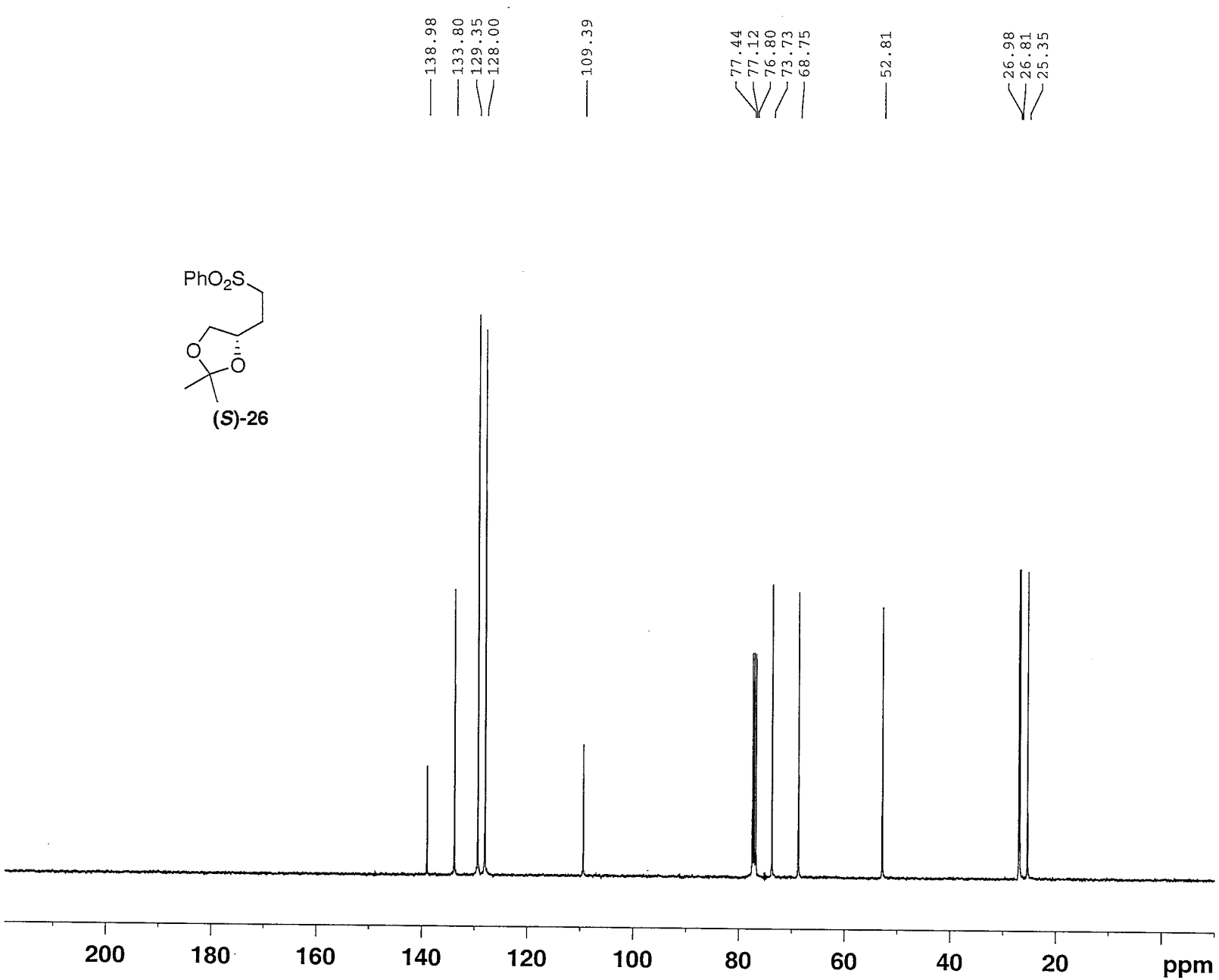

```
NAME            MSII13
EXPNO                 1
PROCNO                1
Date_        20100915
Time             1 s
INSTRUM          spec
PROBHD   5 mm CPDCH 13C
PULPROG        zgpg30
TD             98304
SOLVENT        CDCl3
NS               901
DS                 4
SWH        41666.668 H
FIDRES      0.423855 H
AQ         1.1796980 s
RG               203
DW            12.000 u
DE             15.00 u
TE             298.1 K
D1        2.00000000 s
D11       0.03000000 s
TD0                1

======== CHANNEL f1 =====
NUC1              13C
P1               9.00 u
PL1              4.50 d
PL1W       38.14553833 W
SFO1       176.0629186 M

======== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2               1H
PCPD2           65.00 u
PL2             -3.20 d
PL12            13.60 d
PL13           120.00 d
PL2W       33.59817505 W
PL12W       0.70196527 W
PL13W       0.00000000 W
SFO2       700.1228005 M
SI              131072
SF         176.0453140 M
WDW                EM
SSB                 0
LB               3.00 H
GB                  0
PC               1.40
```

Acetonide



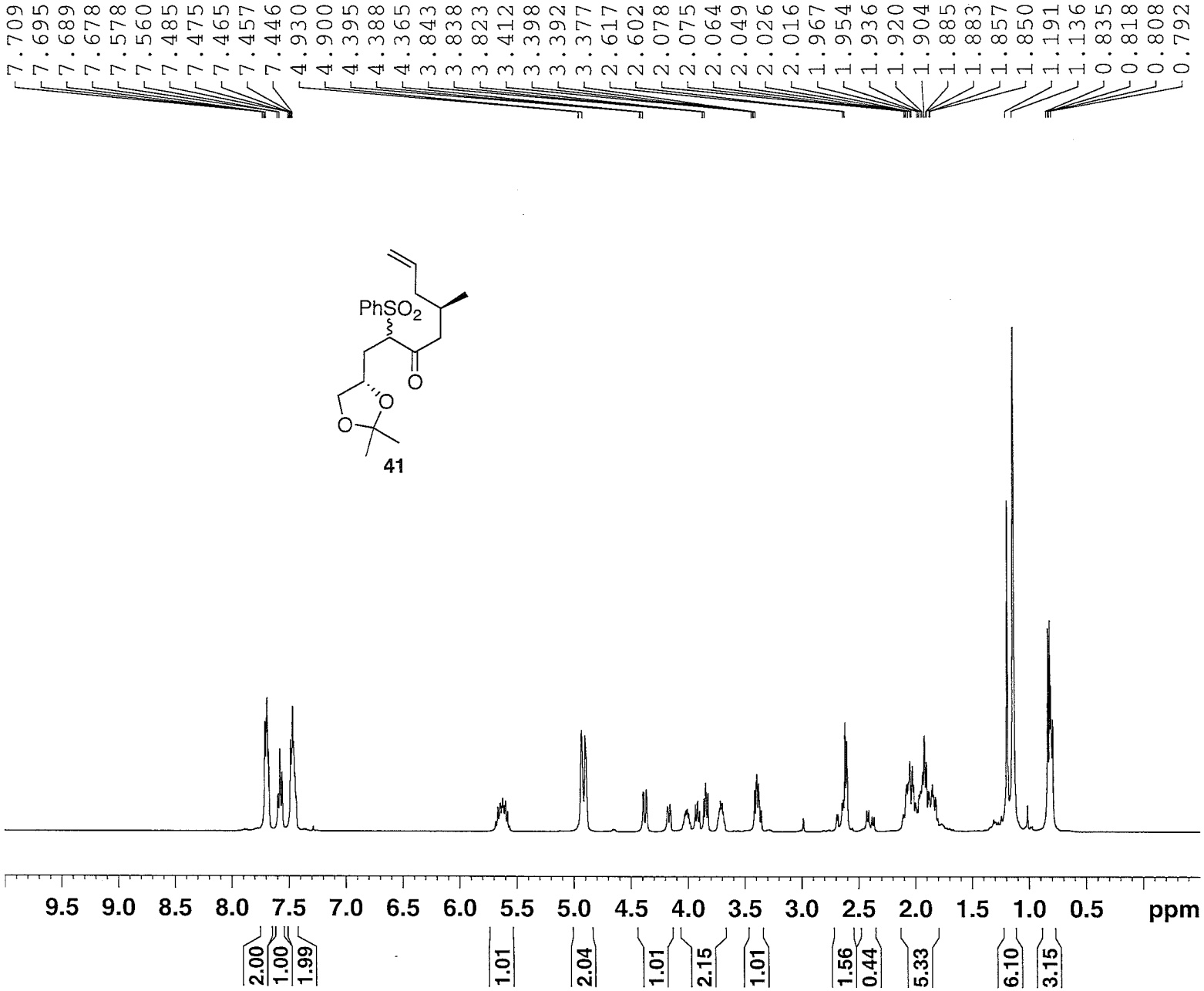

```
Current Data Parameters
NAME                MSII24
EXPNO                    1
PROCNO                   1
DU                      /m
USER              Mrinmoy

F2 - Acquisition Parameters
Date_             20100211
Time                 10.55
INSTRUM             DPX400
PROBHD    5 mm BBO BB-1H
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       2
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ               2.5559540 sec
RG                    40.3
DW                  78.000 usec
DE                    6.00 usec
TE                   298.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                  -3.00 dB
SFO1           400.2478017 MHz

F2 - Processing parameters
SI                   32768
SF            400.2450000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

Acetonide 13C



NAME	MSII24
EXPNO	3
PROCNO	20
Date_	20100211
Time	11.23
INSTRUM	DPX400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	23980.814
FIDRES	0.365918
AQ	1.3664756
RG	8192
DW	20.850
DE	6.00
TE	298.2
D1	2.00000000
d11	0.03000000
DELTA	1.89999998
TD0	1
======== CHANNEL f1 ====	
NUC1	13C
P1	8.30
PL1	-3.00
SFO1	100.6517495
======== CHANNEL f2 ====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00
PL2	-3.00
PL12	15.00
PL13	15.00
SFO2	400.2466010
SI	32768
SF	100.6416850
WDW	EM
SSB	0
LB	3.00
GB	0
PC	1.40

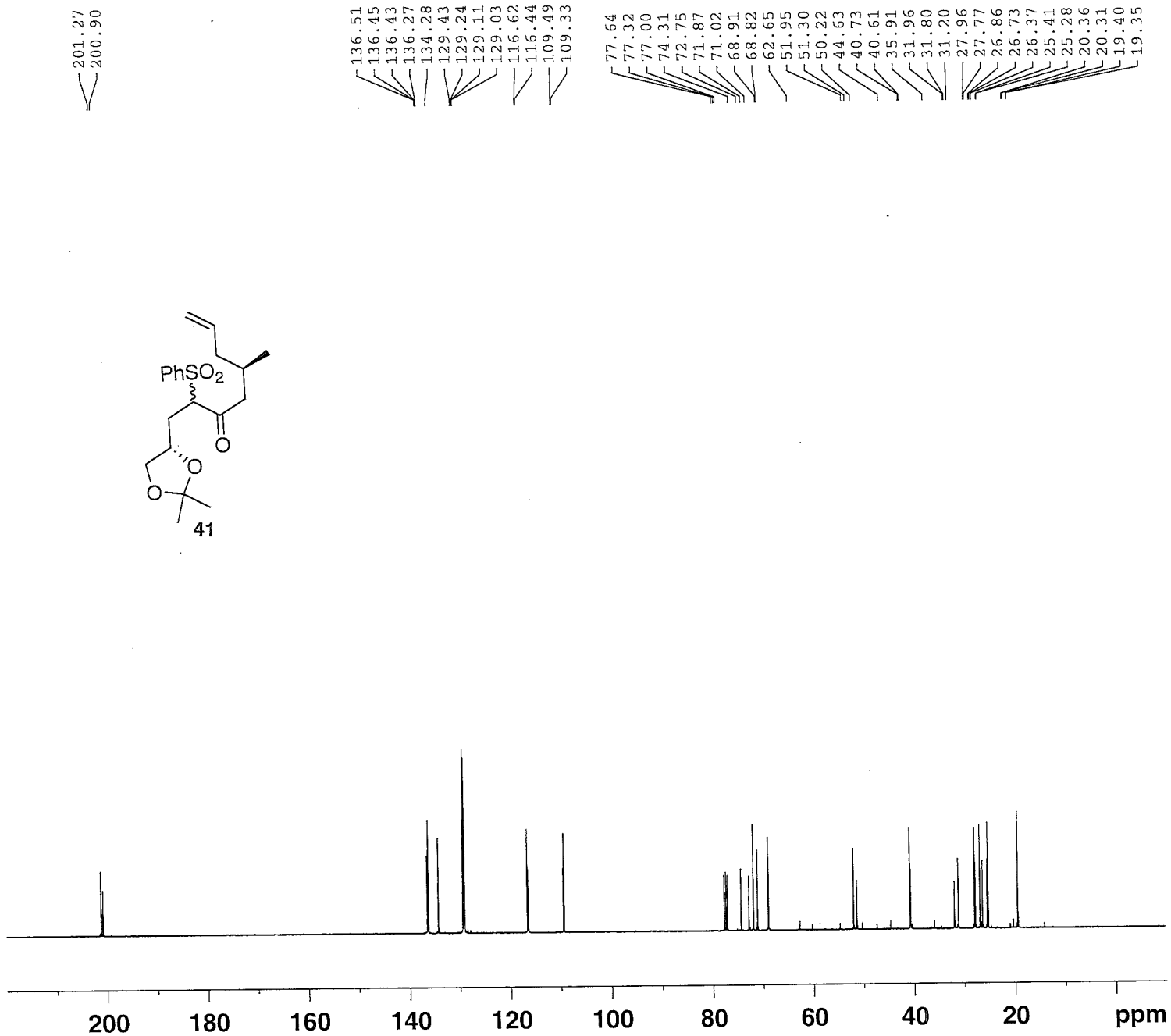
Sulfone-ester coupling



S 21

NAME	MSII45
EXPNO	1
PROCNO	1
Date_	20100316
Time	15.25
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 se
RG	8
DW	78.000 us
DE	6.50 us
TE	298.0 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.00 us
PL1	0.00 dB
PL1W	10.27361584 W
SFO1	400.1378009 MH
SI	32768
SF	400.1350000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

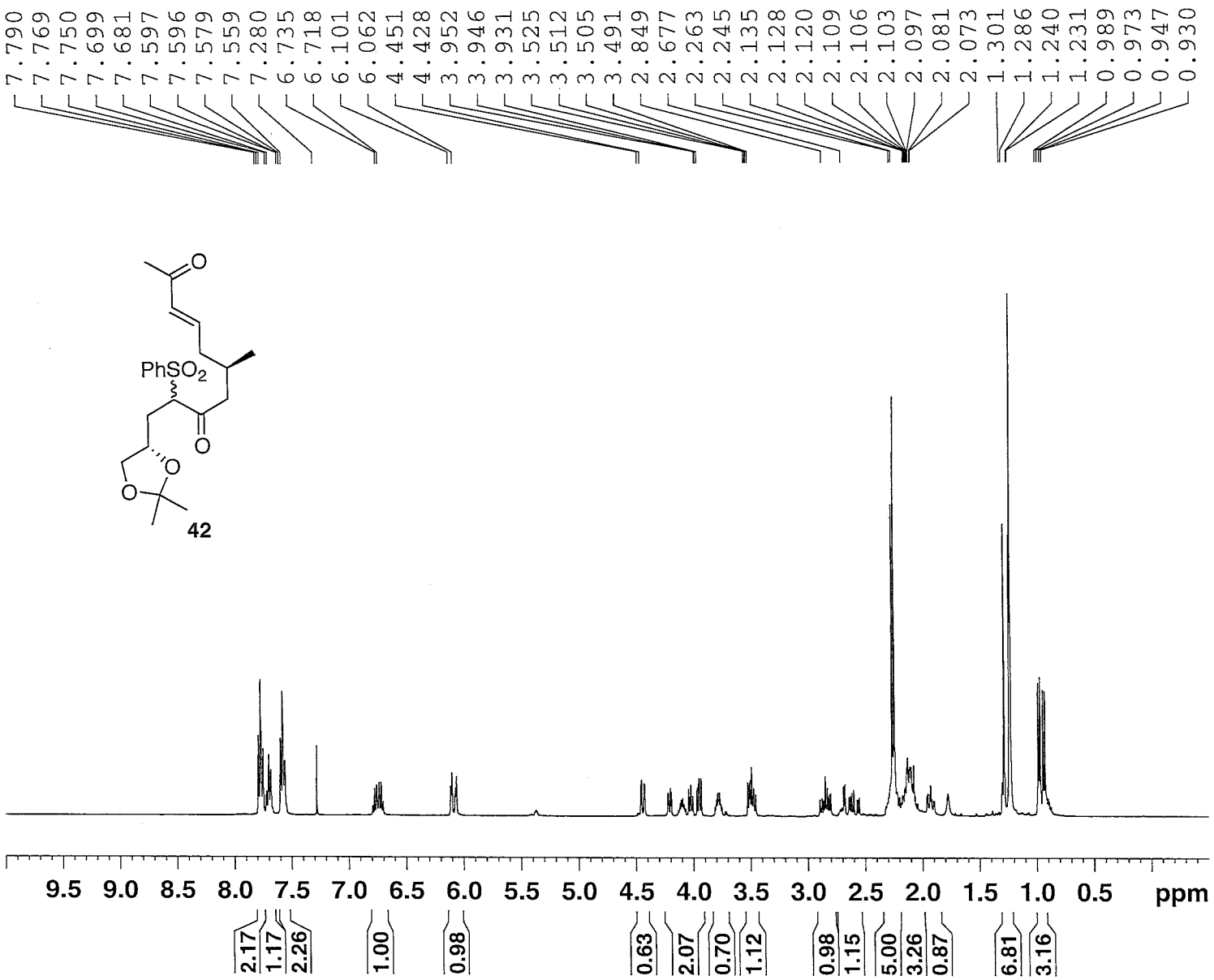
Sulfone-ester coupling carbon



S 22

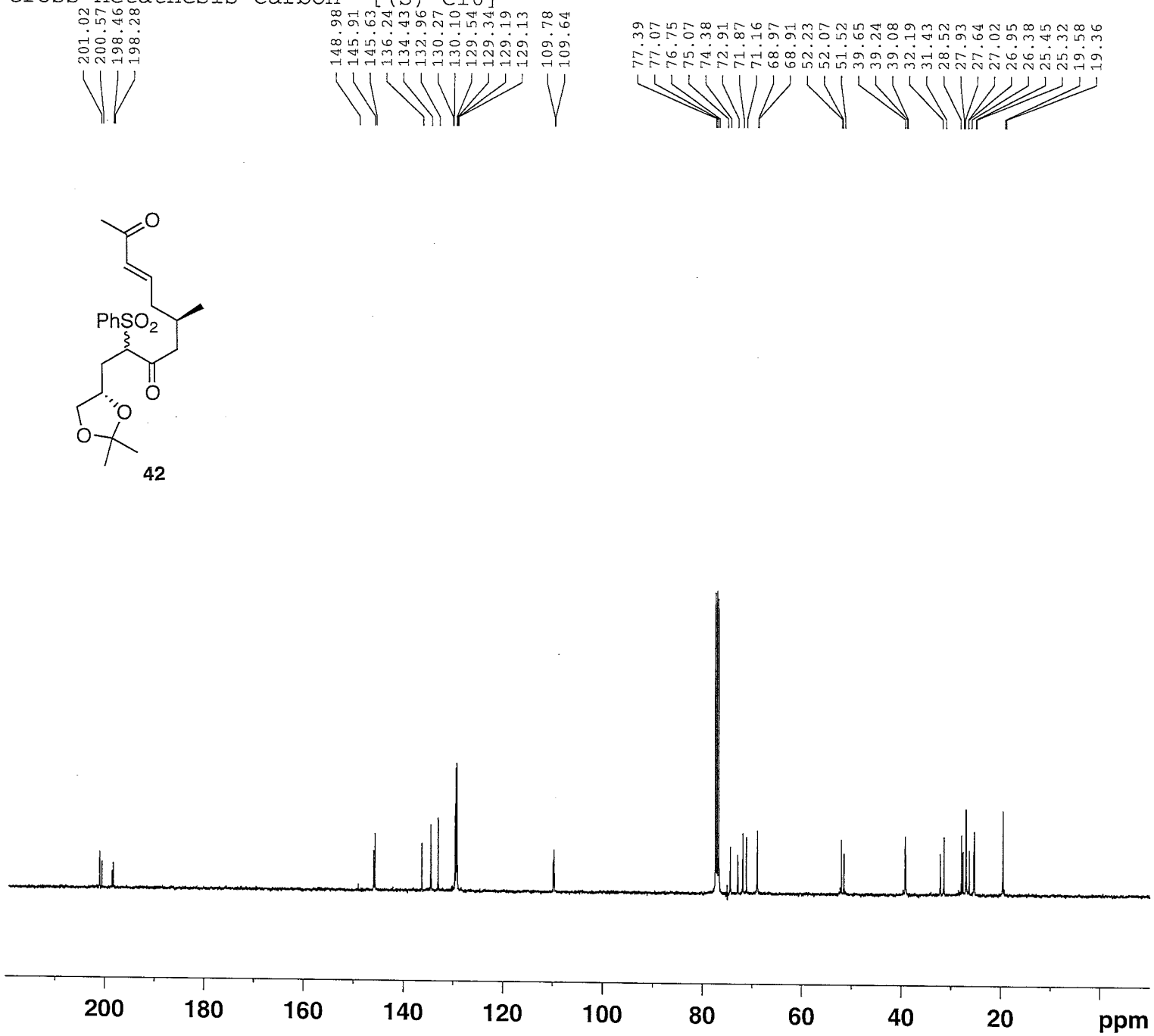
NAME	MSII45
EXPNO	2
PROCNO	1
Date_	20100316
Time	15.28
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	583
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	11585.2
DW	19.900 use
DE	6.50 use
TE	298.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	9.00 use
PL1	-2.00 dB
PL1W	46.89702606 W
SFO1	100.6255966 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 use
PL2	0.00 dB
PL12	16.16 dB
PL13	17.00 dB
PL2W	10.27361584 W
PL12W	0.24872722 W
PL13W	0.20498557 W
SFO2	400.1366005 MHz
SI	32768
SF	100.6140260 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Cross Metathesis [(S)-C10]



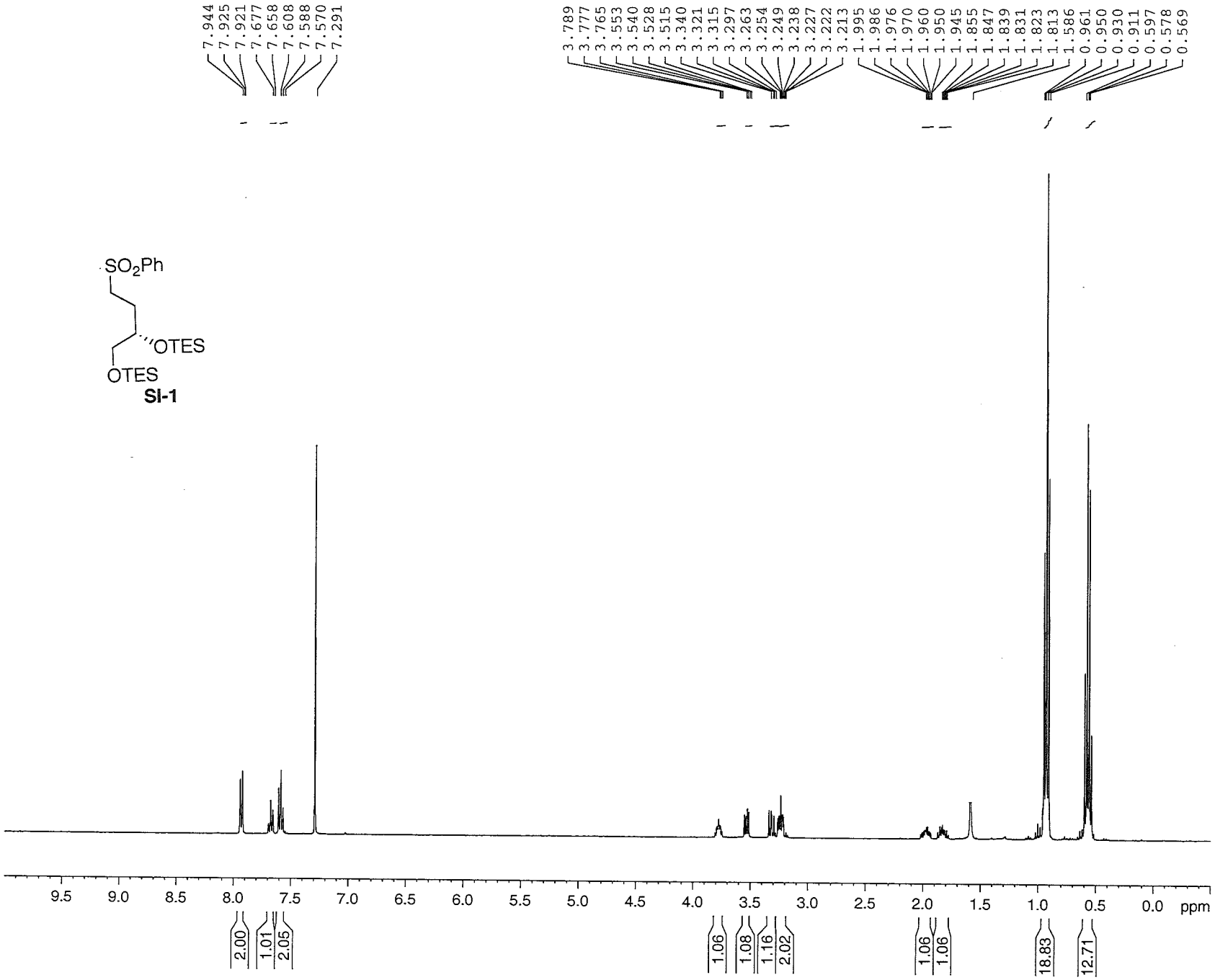

```
Current Data Parameters
NAME            MSII27i
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20100222
Time              12.52
INSTRUM          DPX400
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ            2.5559540 sec
RG                 90.5
DW               78.000 use
DE                 6.00 use
TE                298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                13.50 use
PL1               -3.00 dB
SFO1        400.2478017 MHz

F2 - Processing parameters
SI                32768
SF          400.2450000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

Cross Metathesis carbon [(S)-C10]




```
Current Data Parameters
NAME              MSII27
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameter
Date_           20100222
Time               13.13
INSTRUM          DPX400
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 se
RG               5160.6
DW               20.850 us
DE                6.00 us
TE               298.2 K
D1           2.00000000 se
d11          0.03000000 se
DELTA        1.89999998 se
TD0                   1

======== CHANNEL f1 ======
NUC1                 13C
P1                  8.30 us
PL1                -3.00 dB
SFO1        100.6517495 MH

======== CHANNEL f2 ======
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 us
PL2                -3.00 dB
PL12               15.00 dB
PL13               15.00 dB
SFO2        400.2466010 MH

F2 - Processing parameters
SI                32768
SF        100.6416850 MH
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40
```

tes protection of diol 2nd fraction



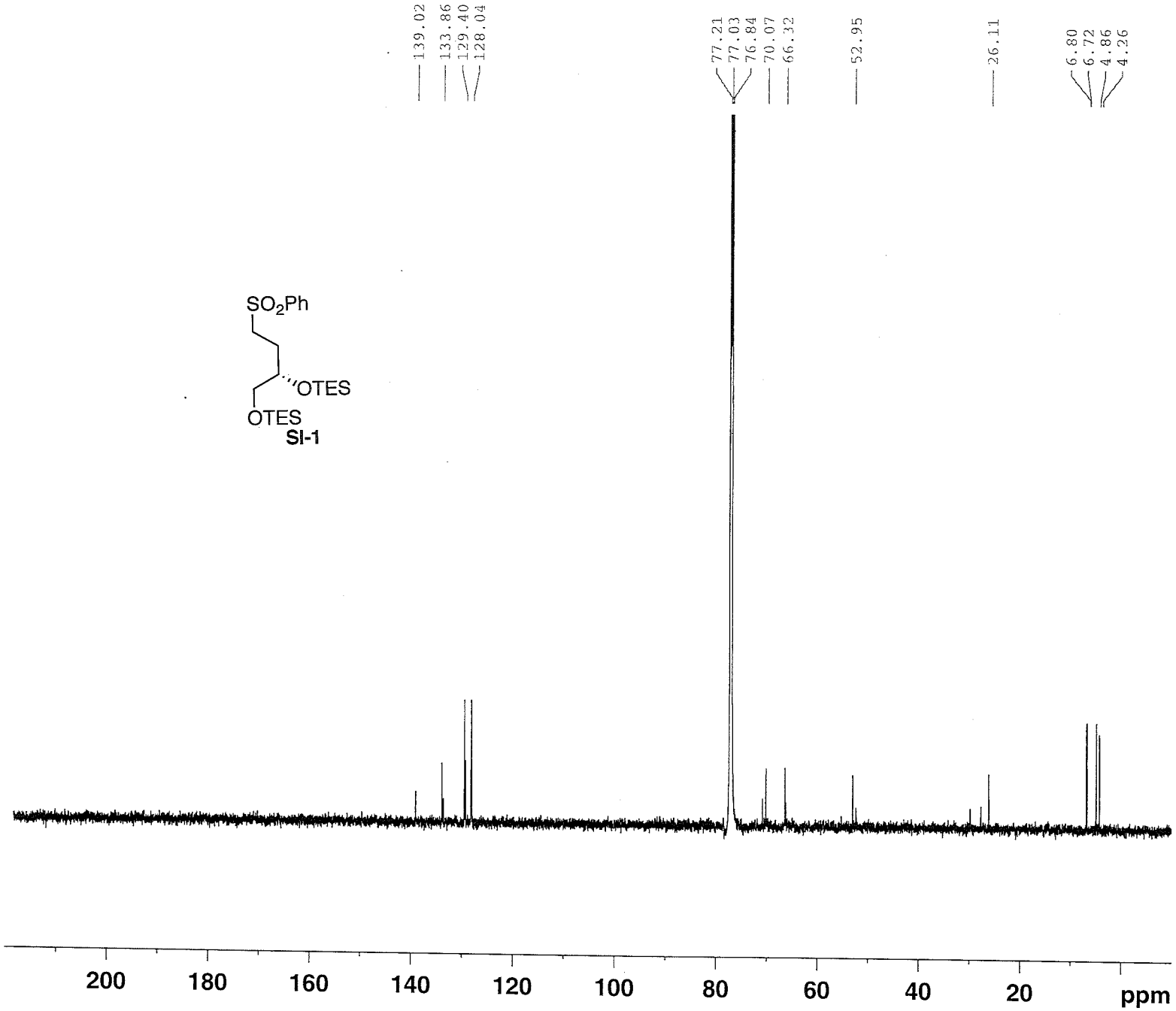
Current Data Parameters
NAME MSII9
EXPNO 2
PROCNO 1
DU /q
USER Mo Gmoy

F2 - Acquisition Parame
Date_ 20100115
Time 17.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6410.256
FIDRES 0.195625
AQ 2.5559540
RG 256
DW 78.000
DE 6.50
TE 300.2
D1 1.00000000
TD0 1

======== CHANNEL f1 ===
NUC1 1H
P1 14.00
PL1 0.00
PL1W 10.27361584
SFO1 400.1378009

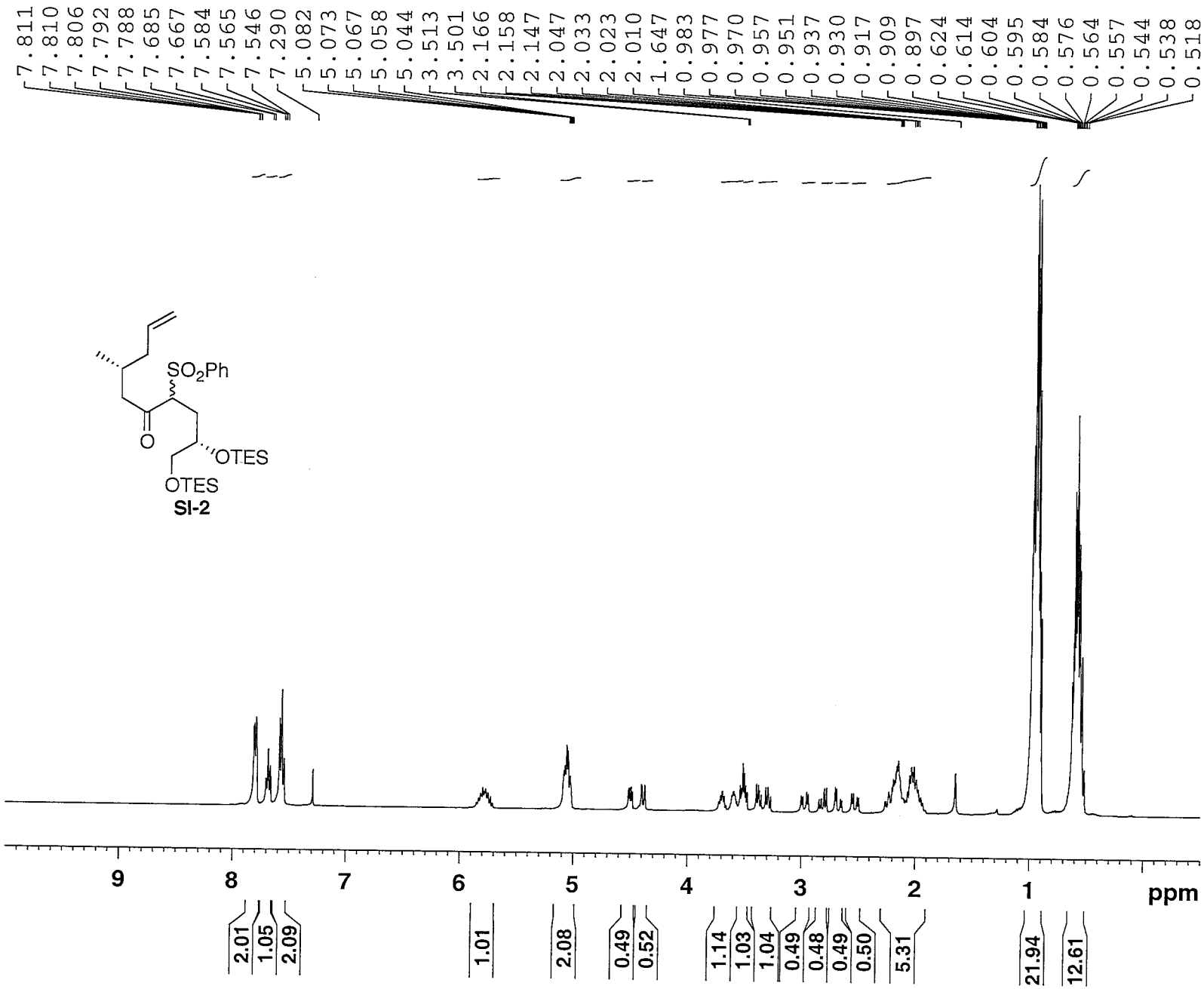
F2 - Processing paramet
SI 32768
SF 400.1350000
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

(S)-di-TES ether



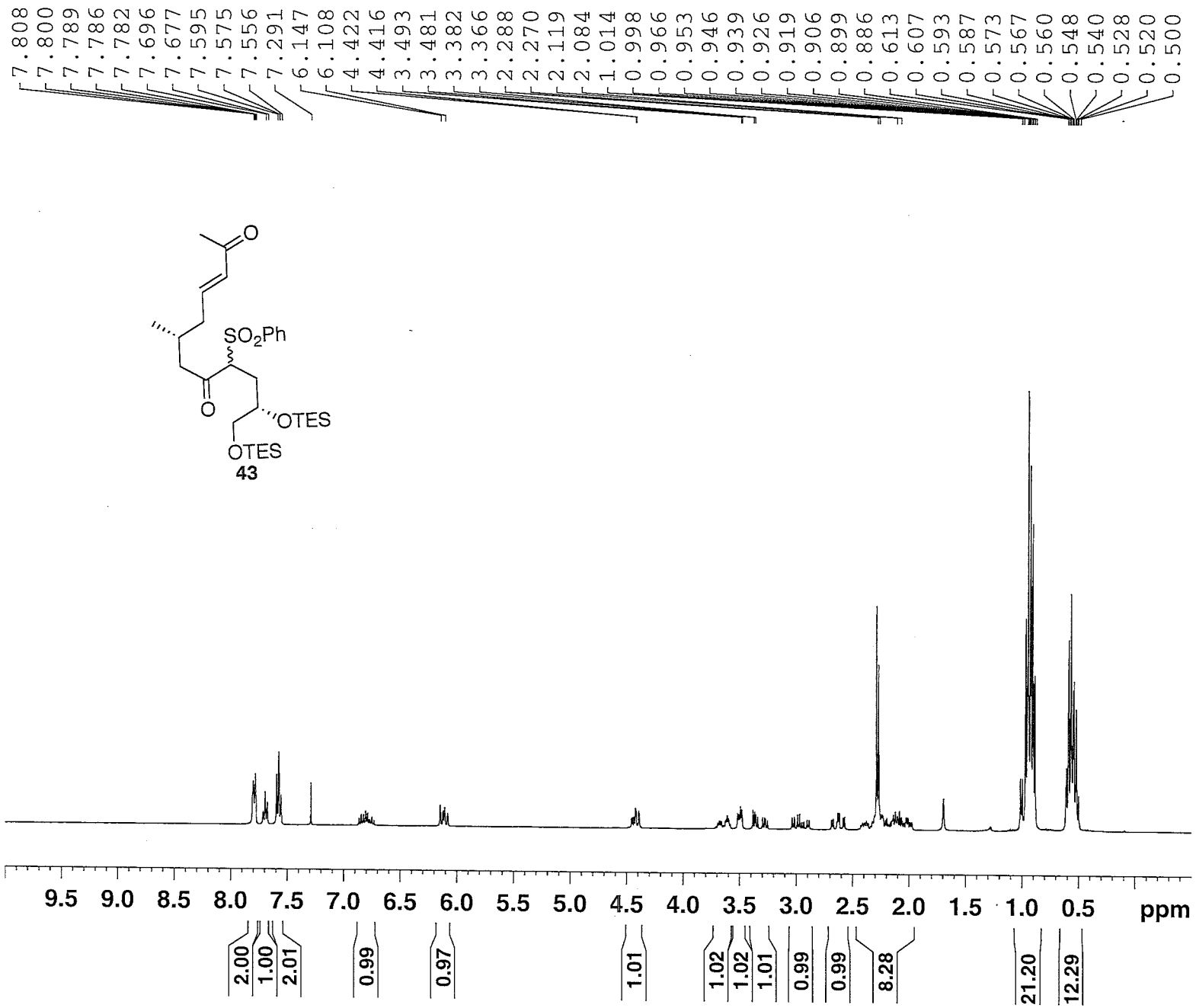
NAME	MS II-09 Di-TES eth
EXPNO	2
PROCNO	1
Date_	20141117
Time	2.27
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Julia Coupling 1st fraction



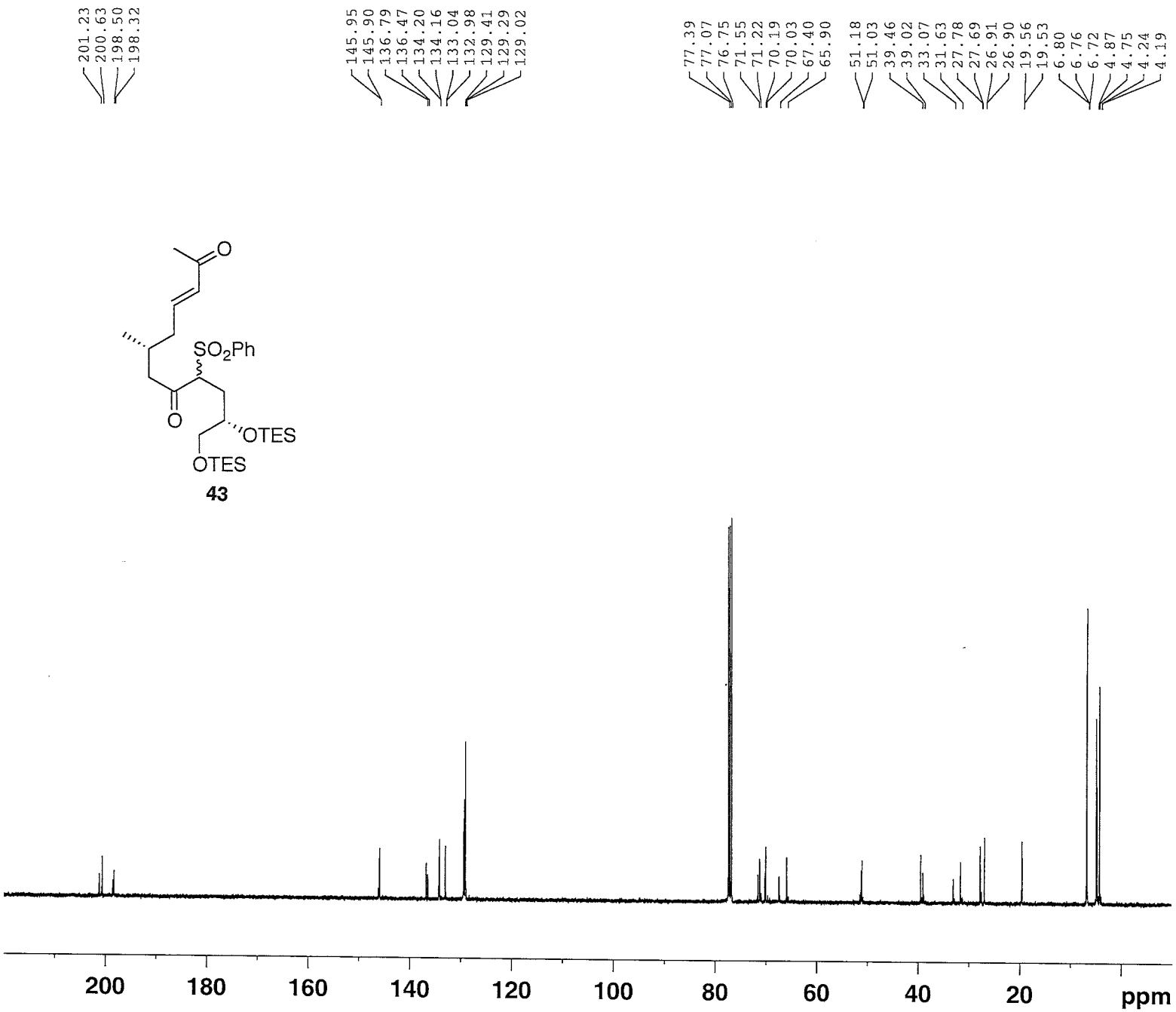

```
NAME              MSII10
EXPNO                  2
PROCNO                 1
Date_           20100118
Time                21:16
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      2
SWH             6410.256
FIDRES          0.195625
AQ             2.5559540
RG                  35.9
DW                 78.000
DE                   6.50
TE                  299.9
D1            1.00000000
TD0                     1

======== CHANNEL f1 ====
NUC1                   1H
P1                  14.00
PL1                  0.00
PL1W        10.27361584
SFO1        400.1378009
SI                32768
SF          400.1350000
WDW                   EM
SSB                    0
LB                  0.30
GB                     0
PC                  1.00
```

Cross metathesis with di-TES ether



NAME	MSII12
EXPNO	2
PROCNO	1
Date_	20100124
Time	21.55
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 se
RG	45.3
DW	78.000 us
DE	6.50 us
TE	299.5 K
D1	1.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.00 us
PL1	0.00 dB
PL1W	10.27361584 W
SFO1	400.1378009 MH
SI	32768
SF	400.1350000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Cross Metathesis 13C

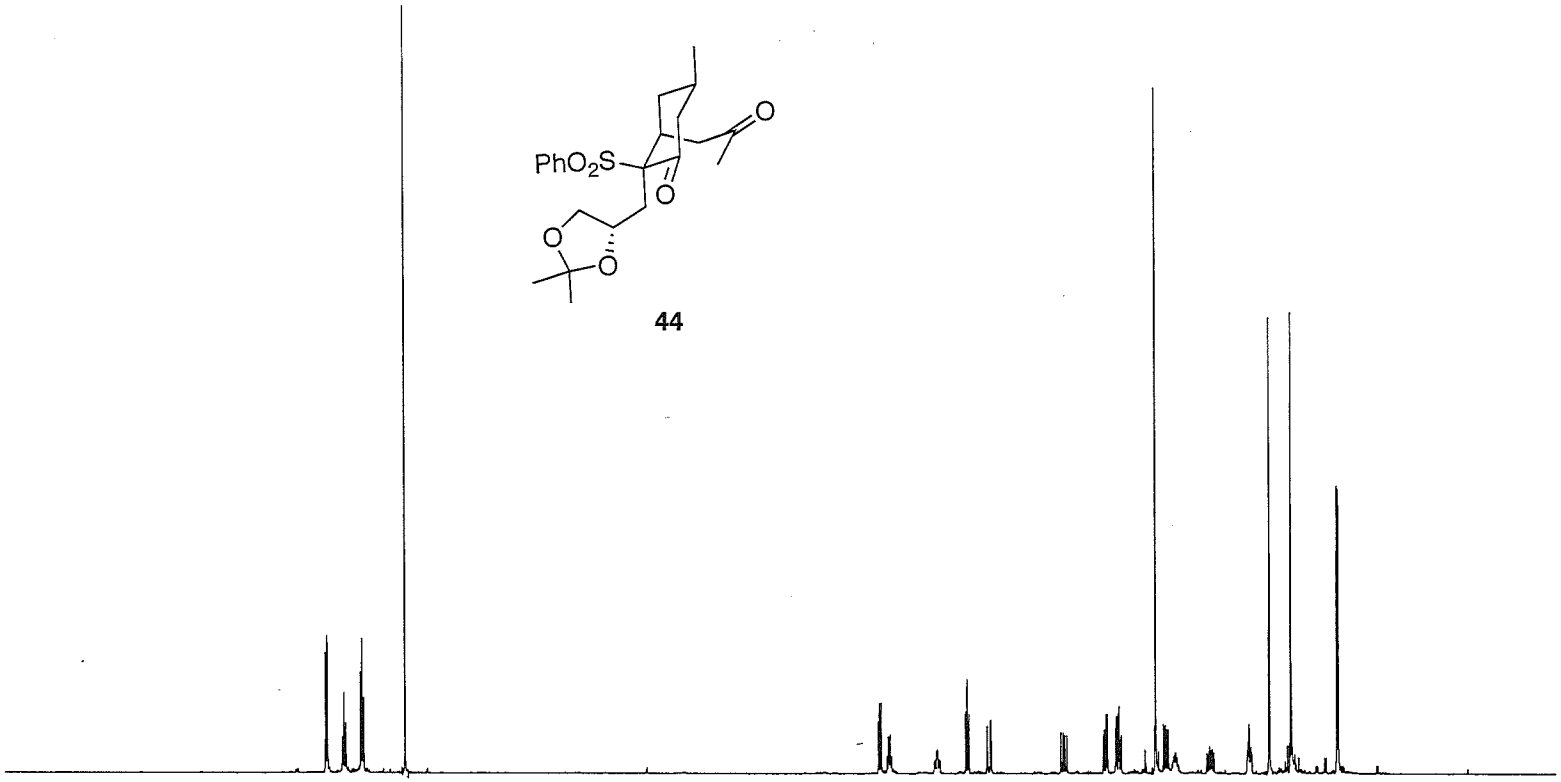


NAME	MSII28
EXPNO	62
PROCNO	1
Date_	20100326
Time	11.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	20642.5
DW	19.900 use
DE	6.50 use
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	9.00 use
PL1	-2.00 dB
PL1W	46.89702606 W
SFO1	100.6255966 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 use
PL2	0.00 dB
PL12	16.16 dB
PL13	17.00 dB
PL2W	10.27361584 W
PL12W	0.24872722 W
PL13W	0.20498557 W
SFO2	400.1366005 MHz
SI	32768
SF	100.6140260 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Micheal product (C10 epimer) check

7.826 7.825 7.814 7.812 7.700 7.689 7.589 7.587 7.579 7.577 7.567 7.288 4.084 4.075 4.072 4.064 3.493 3.482 3.470 3.349 3.347 3.323 3.321 2.851 2.836 2.825 2.563 2.554 2.544 2.534 2.479 2.477 2.474 2.462 2.458 2.455 2.209 2.156 2.144 2.134 2.122 1.576 1.440 1.294 0.985 0.975

44

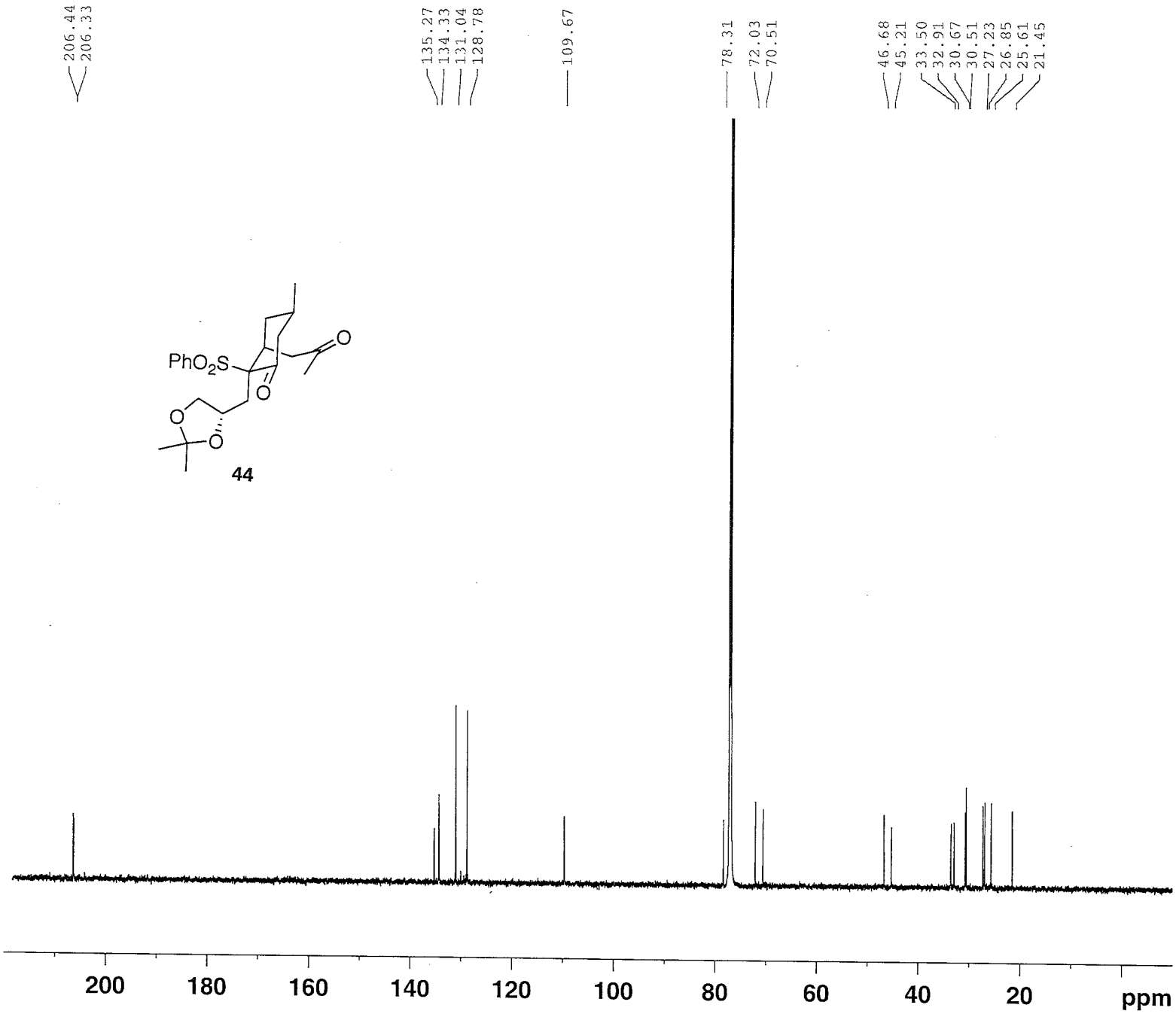


9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

2.13 1.18 2.20 1.00 1.09 1.02 1.02 1.11 1.02 1.07 2.06 3.03 1.14 1.13 1.07 1.48 3.06 3.10 3.07

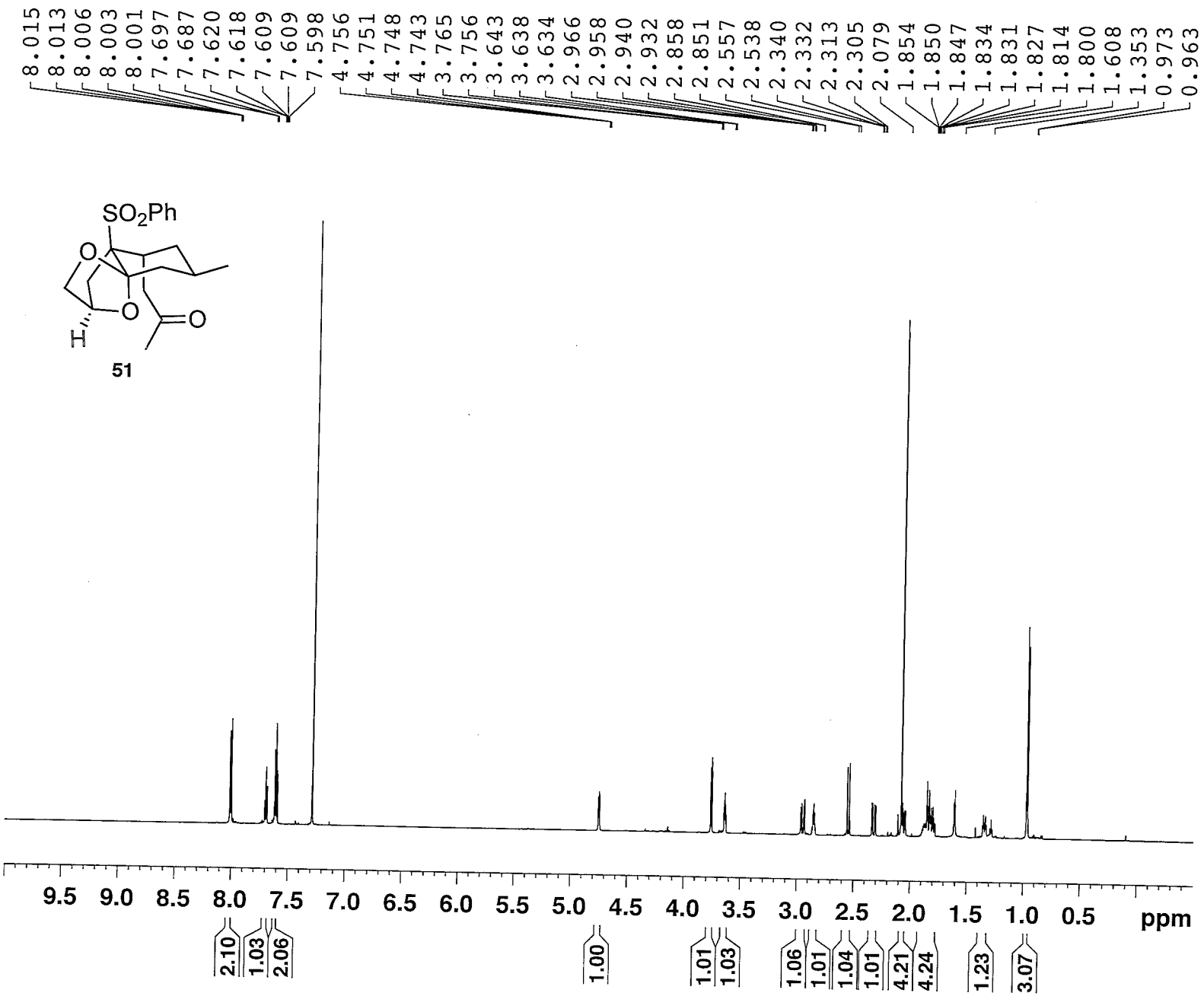
NAME	MS II-49
EXPNO	30
PROCNO	1
Date_	20140122
Time	15.33
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	5
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 se
RG	32
DW	42.000 us
DE	6.50 us
TE	298.2 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	9.40 us
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MH
SI	131072
SF	700.1471400 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Micheal product (C10 epimer) carbon



NAME	MS II-49
EXPNO	1
PROCNO	1
Date_	20140120
Time	15.35
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 se
RG	203
DW	12.000 us
DE	16.50 us
TE	298.2 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.00 us
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MH
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 us
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MH
SI	32768
SF	176.0521380 MH
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Ketal

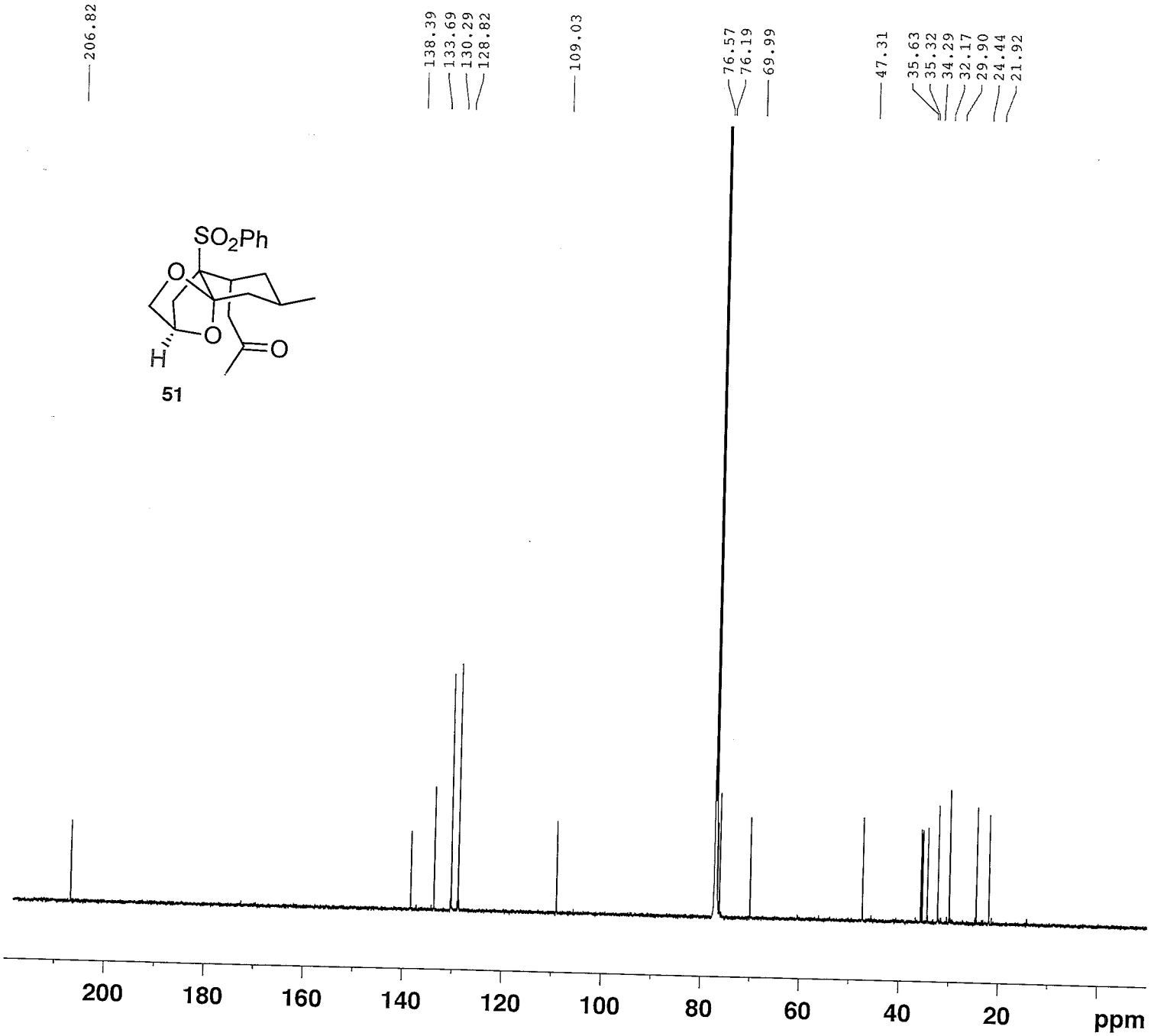


NAME	MS III-1[illegible]
EXPNO	[illegible]
PROCNO	1
Date_	20100726
Time	15.31
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	18
DS	2
SWH	11904.762 Hz
FIDRES	0.181652 Hz
AQ	2.7525620 se
RG	64
DW	42.000 us
DE	16.50 us
TE	298.0 K
D1	2.00000000 se
TD0	1

======== CHANNEL f1 ======

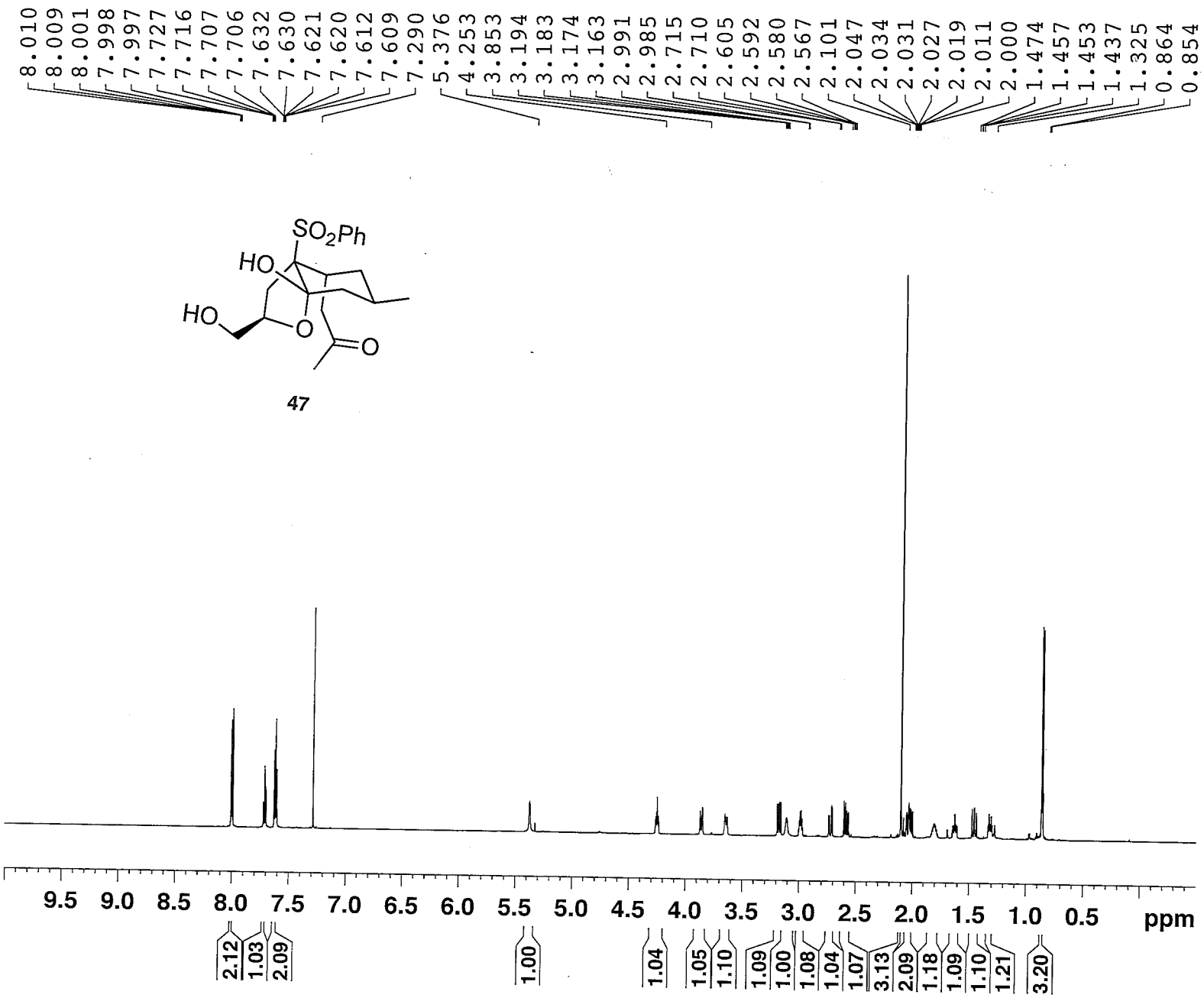
NUC1	1H
P1	9.40 us
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1245508 MH
SI	65536
SF	700.1200000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Ketal carbon



NAME	MS III-193
EXPNO	2
PROCNO	1
Date_	20100726
Time	15.39
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	98304
SOLVENT	CDCl3
NS	296
DS	4
SWH	41666.668 Hz
FIDRES	0.423855 Hz
AQ	1.1796980 se
RG	203
DW	12.000 us
DE	15.00 us
TE	298.0 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.00 us
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0629186 MH
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 us
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1228005 MH
SI	131072
SF	176.0453140 MH
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Hemiketal

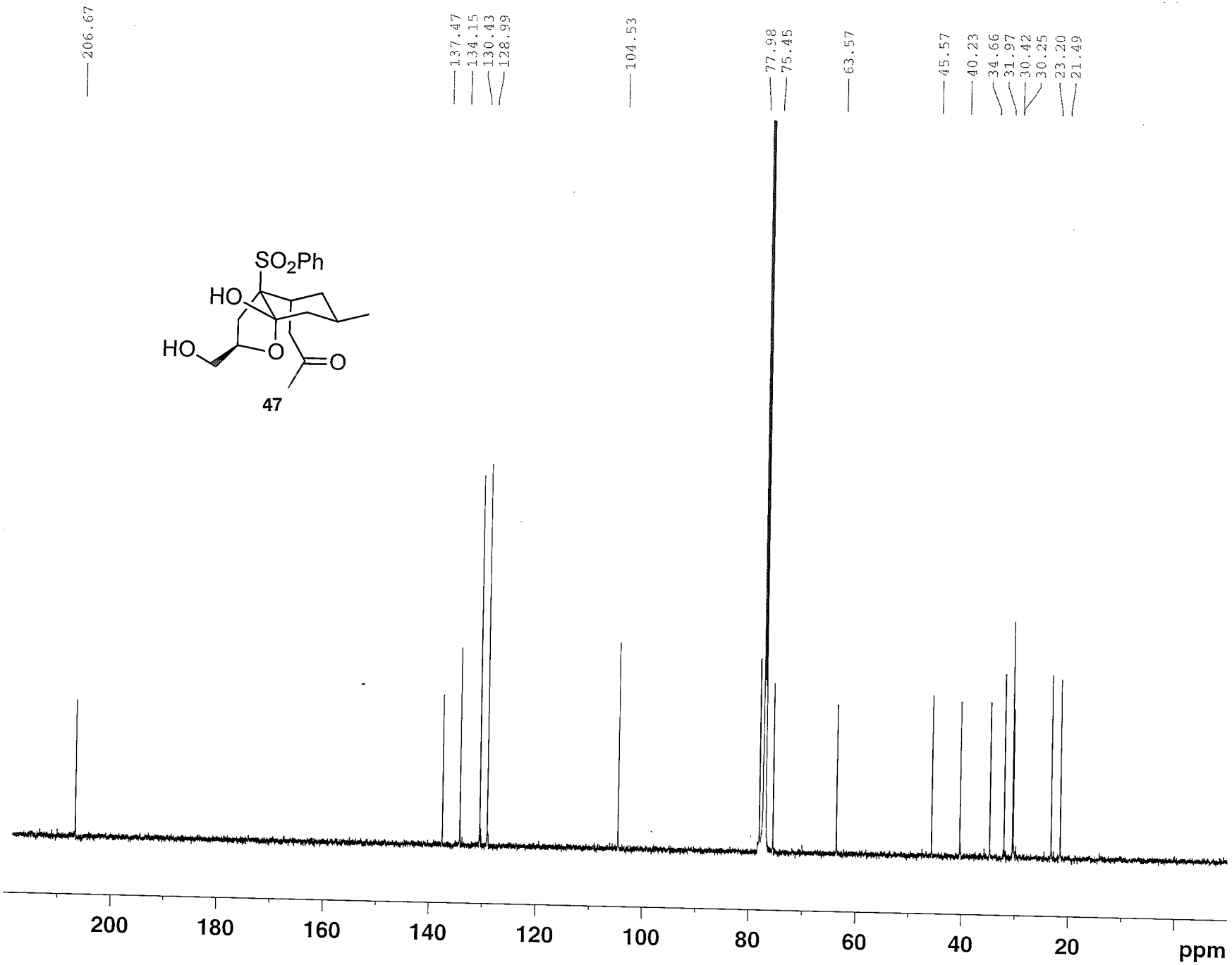


NAME	MS IV-10
EXPNO	1
PROCNO	1
Date_	20110208
Time	0.14
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	10
DS	2
SWH	11904.762 Hz
FIDRES	0.181652 Hz
AQ	2.7525620 se
RG	71.8
DW	42.000 us
DE	16.50 us
TE	298.2 K
D1	2.00000000 se
TD0	1

======== CHANNEL f1 ======

NUC1	1H
P1	9.40 us
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1245508 MHz
SI	65536
SF	700.1200000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

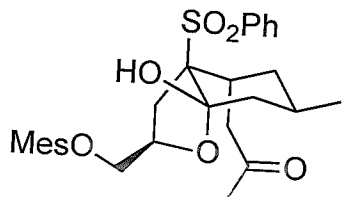
hemiketal carbon



NAME	MS 10
EXPNO	2
PROCNO	1
Date_	2010 08
Time	0.22
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	98304
SOLVENT	CDCl3
NS	202
DS	4
SWH	41666.668 H
FIDRES	0.423855 H
AQ	1.1796980 s
RG	203
DW	12.000 u
DE	15.00 u
TE	298.2 K
D1	2.00000000 s
D11	0.03000000 s
TD0	1
======== CHANNEL f1 =====	
NUC1	13C
P1	9.00 u
PL1	4.50 d
PL1W	38.14553833 W
SFO1	176.0629186 M
======== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 u
PL2	-3.20 d
PL12	13.60 d
PL13	120.00 d
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1228005 M
SI	131072
SF	176.0453140 M
WDW	EM
SSB	0
LB	3.00 H
GB	0
PC	1.40

Mesitylate

7.916 7.914 7.904 7.902 7.704 7.693 7.610 7.599 7.598 7.587 7.288 7.000 4.251 4.249 4.157 4.147 4.143 4.133 4.030 4.022 4.015 4.008 3.879 2.905 2.900 2.787 2.776 2.768 2.757 2.638 2.546 2.538 2.535 2.531 2.333 2.137 2.126 2.118 2.063 1.654 1.517 1.501 1.498 1.481 0.852 0.843



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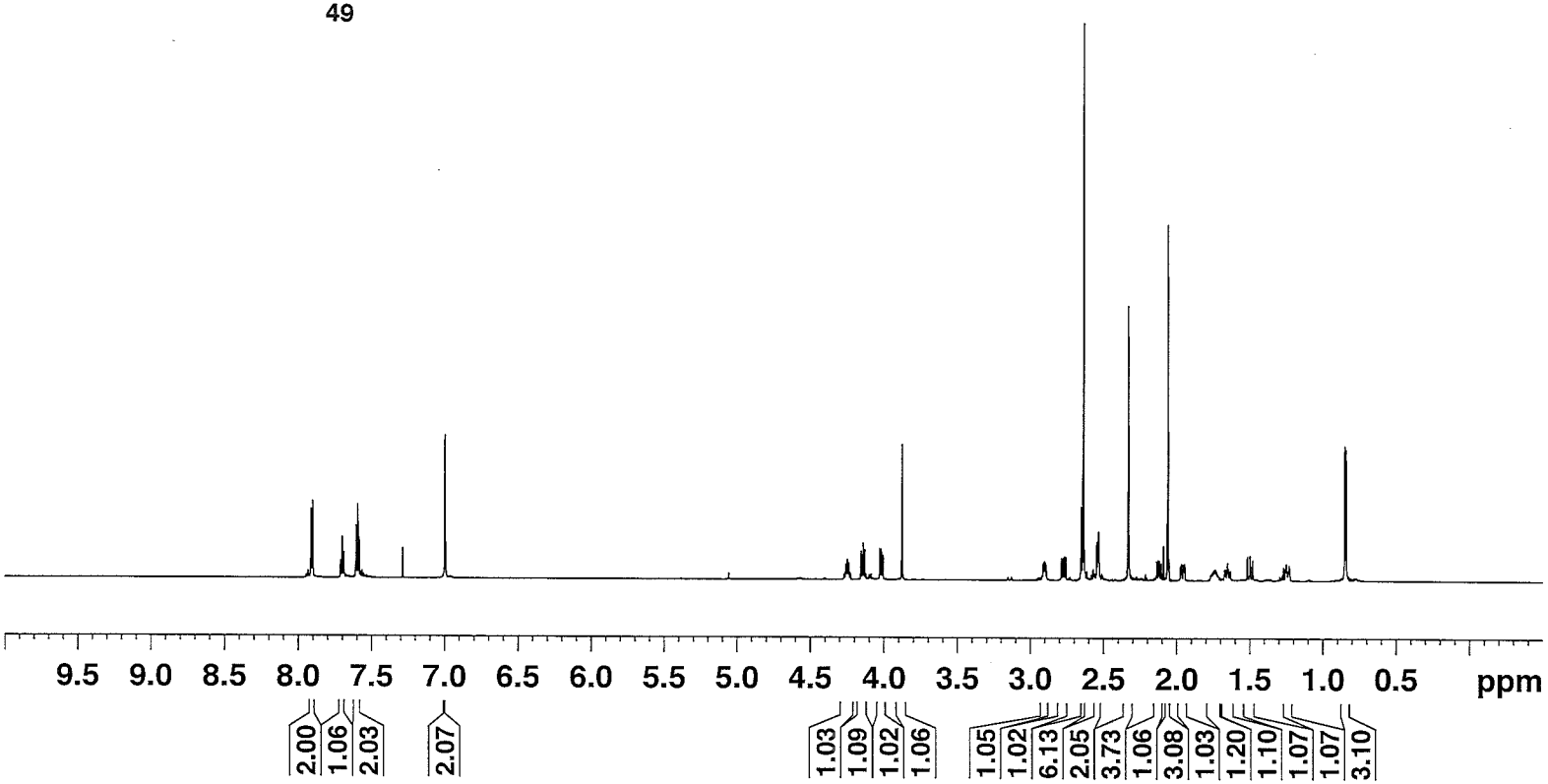
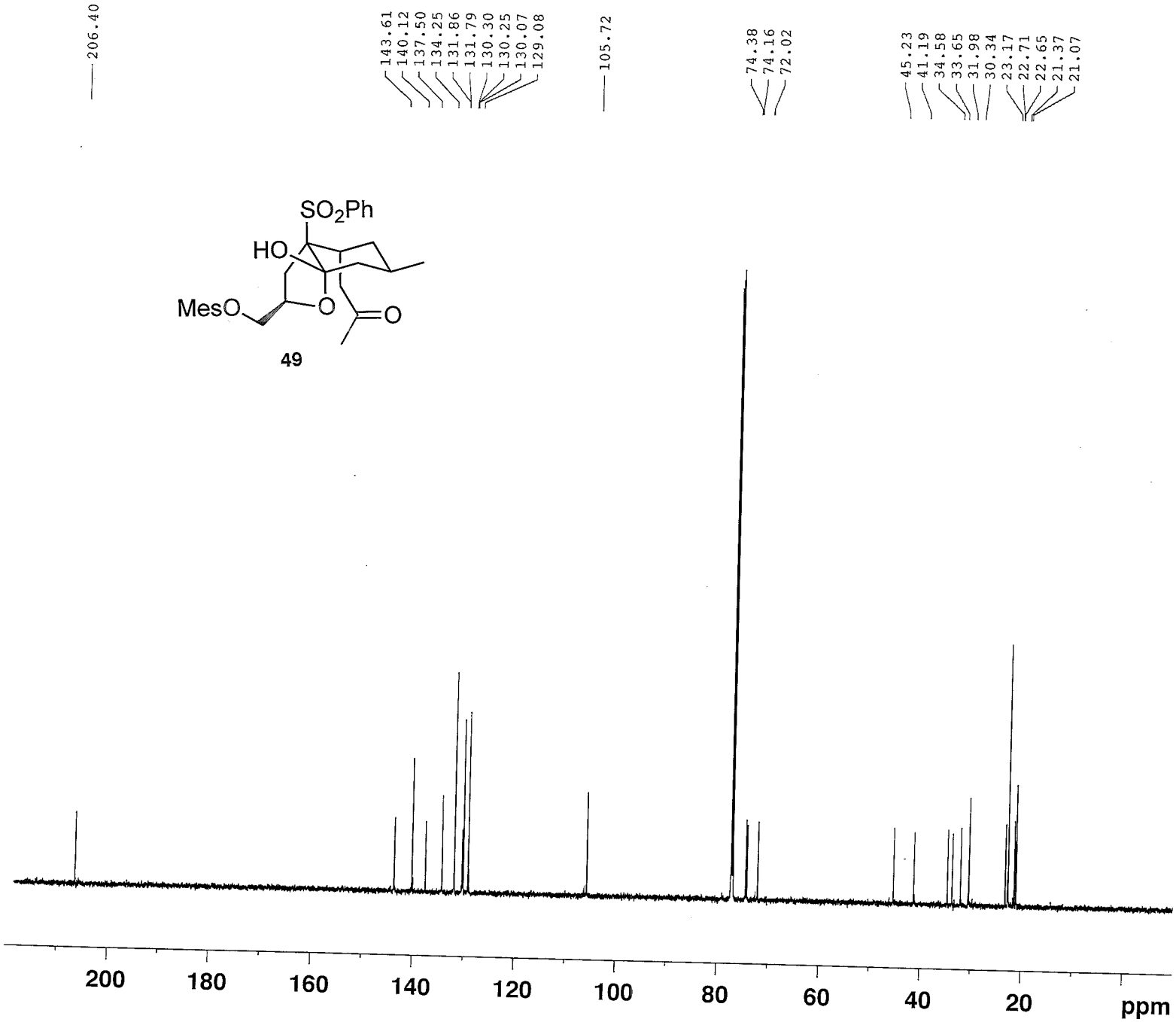


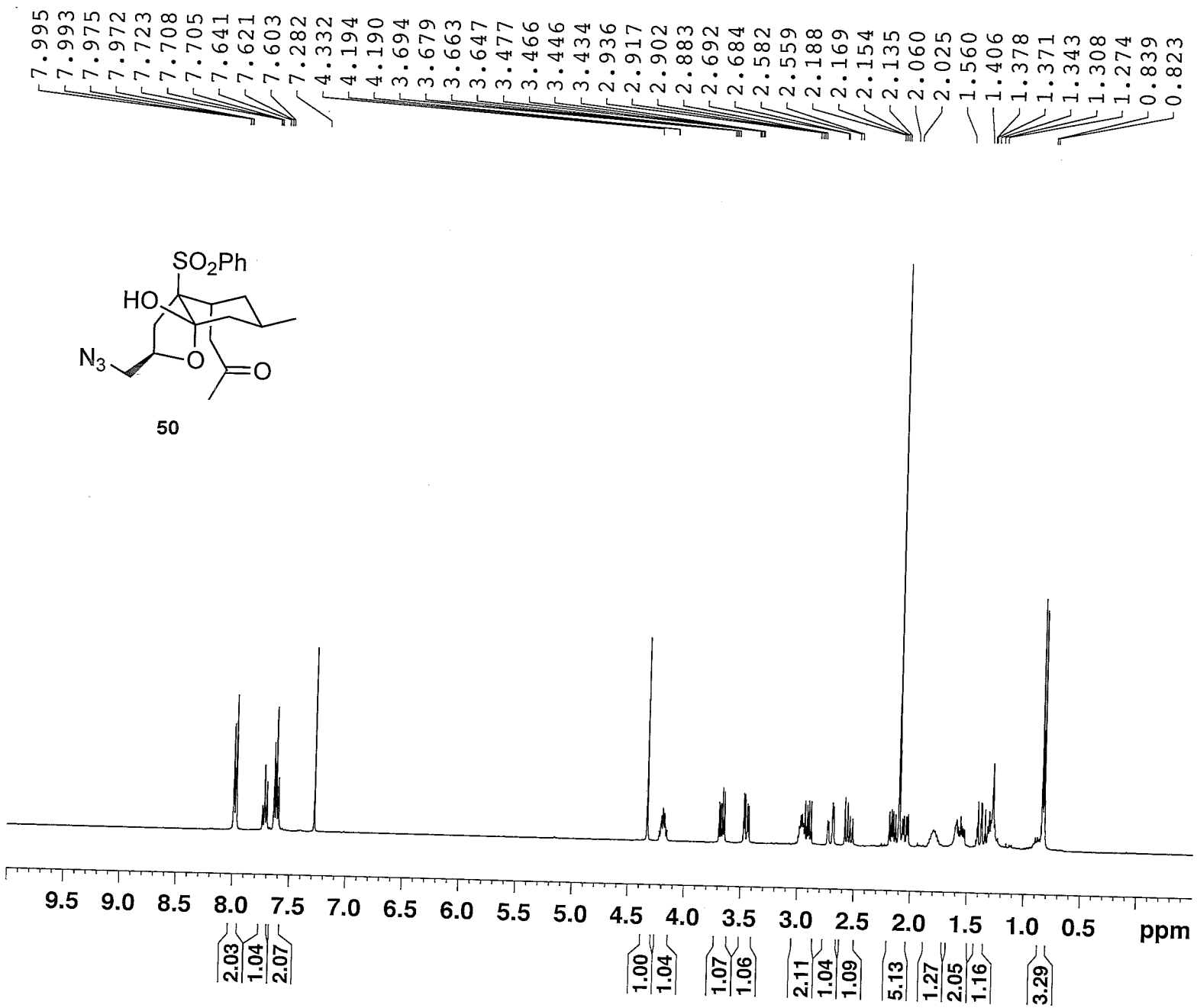
NAME	MS VIII-29 Mesityla
EXPNO	3
PROCNO	1
Date_	20120530
Time	19.21
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	4
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	18
DW	42.000 usec
DE	6.50 usec
TE	298.3 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Mesitylate carbon



NAME	MS VIII-29 Mesityla
EXPNO	5
PROCNO	1
Date_	20120530
Time	19.25
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	6
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Azide

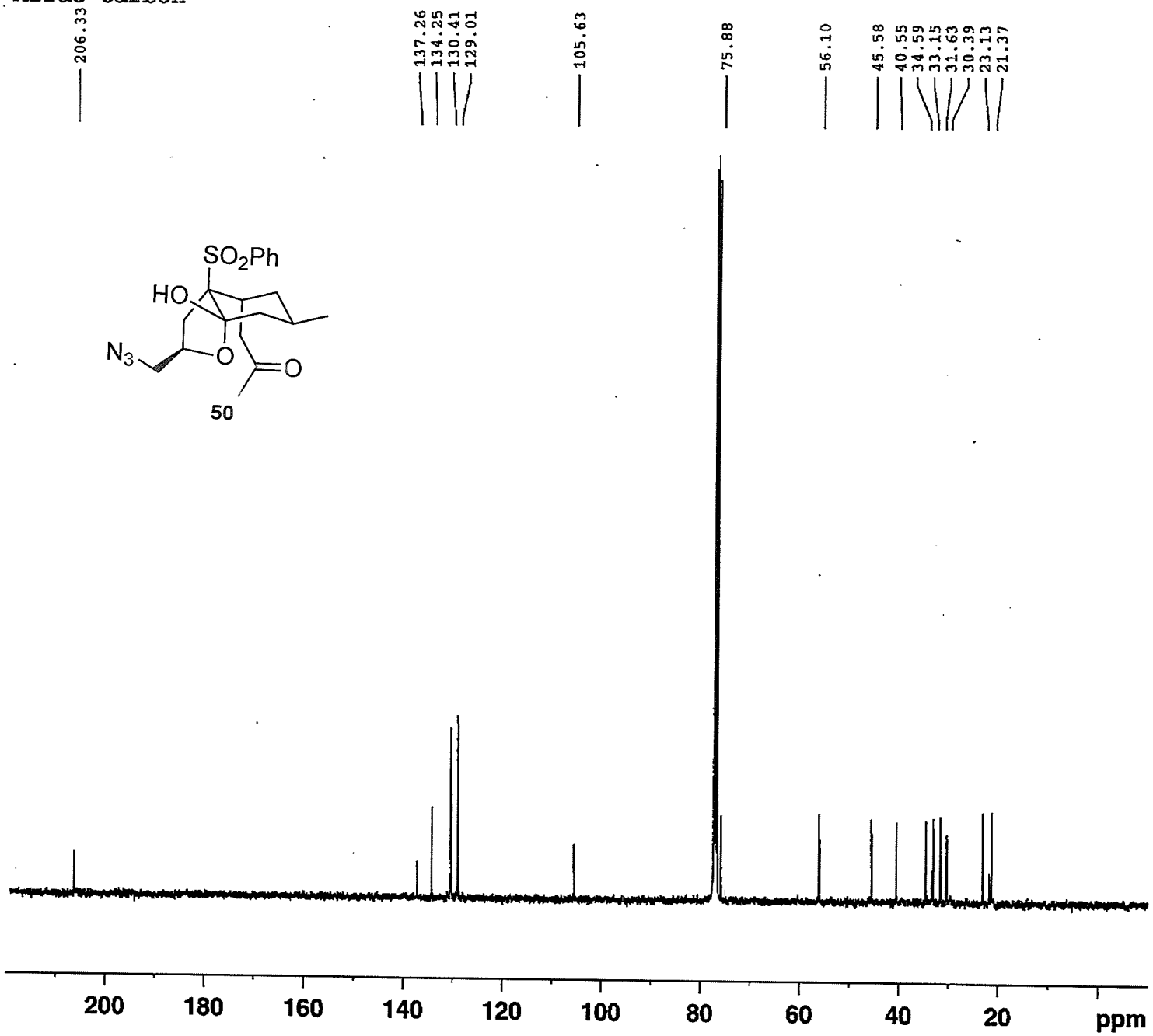


NAME	MS IV-1
EXPNO	1
PROCNO	1
Date_	20110207
Time	12.11
INSTRUM	robinson
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SWH	6793.478 Hz
FIDRES	0.207320 Hz
AQ	2.4117749 se
RG	128
DW	73.600 us
DE	6.50 us
TE	298.2 K
D1	2.00000000 se
TD0	1

======== CHANNEL f1 ======

NUC1	1H
P1	14.00 us
PL1	0.00 dB
SFO1	400.1424008 MH
SI	32768
SF	400.1400000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Azide carbon



Current Data Parameters
NAME MS III-19
EXPNO 3
PROCNO 1

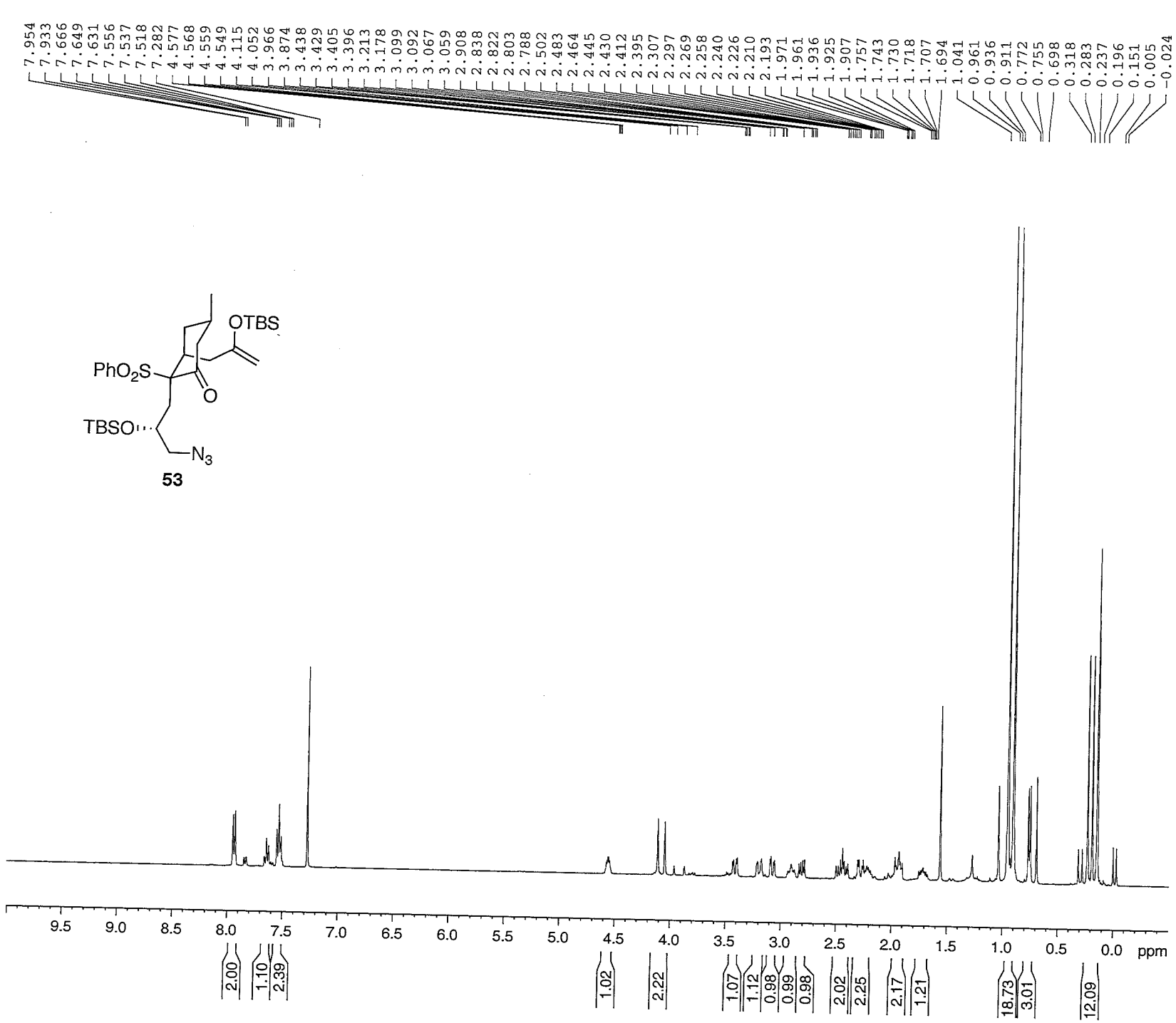
F2 - Acquisition Parameters
Date_ 20100723
Time 17.33
INSTRUM DPX400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 8192
DW 20.850 use
DE 6.00 use
TE 299.2 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1

======== CHANNEL f1 =======
NUC1 13C
P1 8.30 use
PL1 -3.00 dB
SFO1 100.6517495 MH:

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.2466010 MH:

F2 - Processing parameters
SI 32768
SF 100.6416850 MH:
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

TBS enol ether



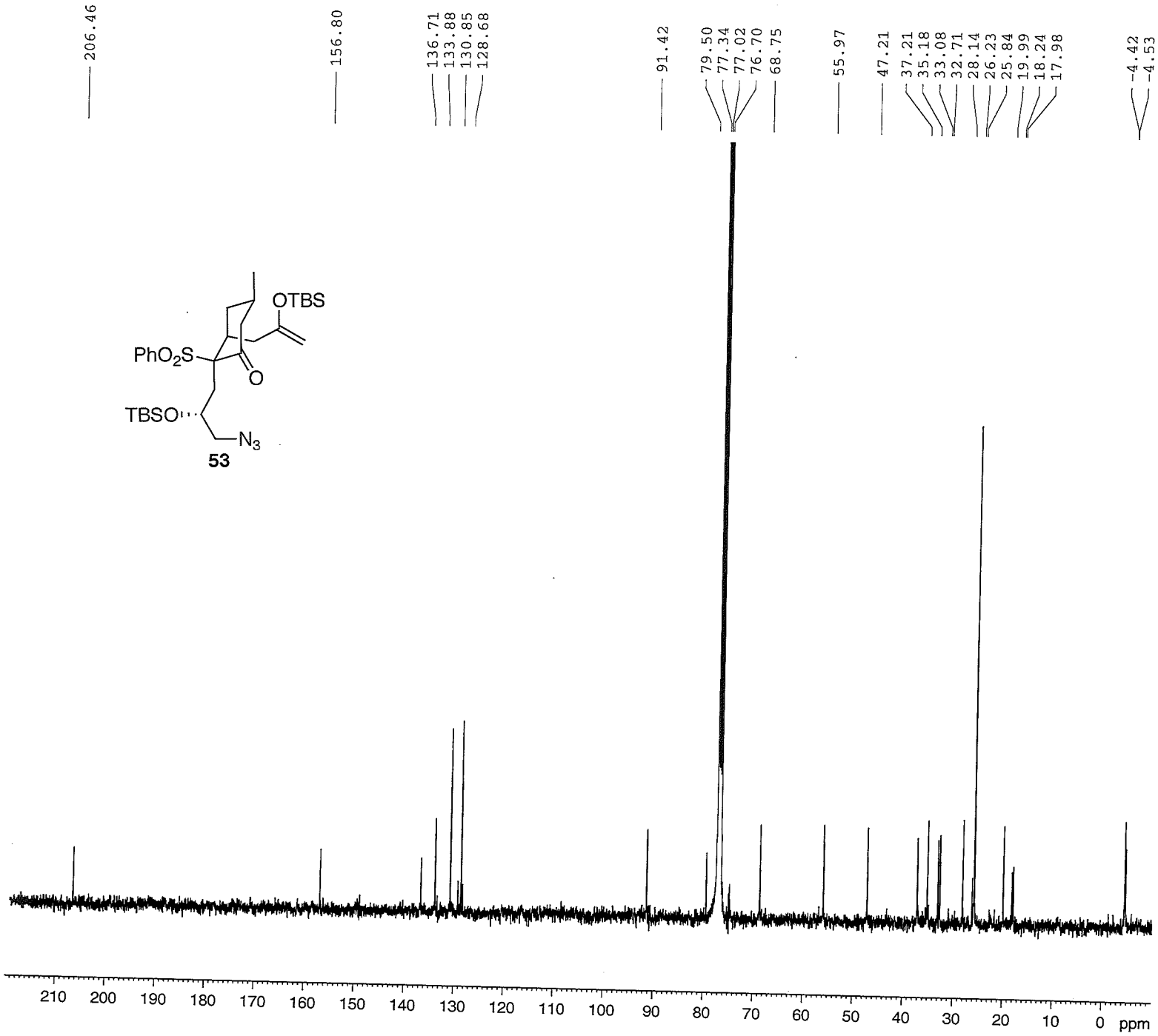
Current Data Parameters
NAME MS IV-15
EXPNO 1
PROCNO 1
DU /m
USER Mrinmoy

F2 - Acquisition Parameters
Date_ 20110209
Time 14.19
INSTRUM DPX400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 574.7
DW 78.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PL1 -3.00 dB
SFO1 400.2478017 MHz

F2 - Processing parameters
SI 32768
SF 400.2450000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

TBS enol ether carbon



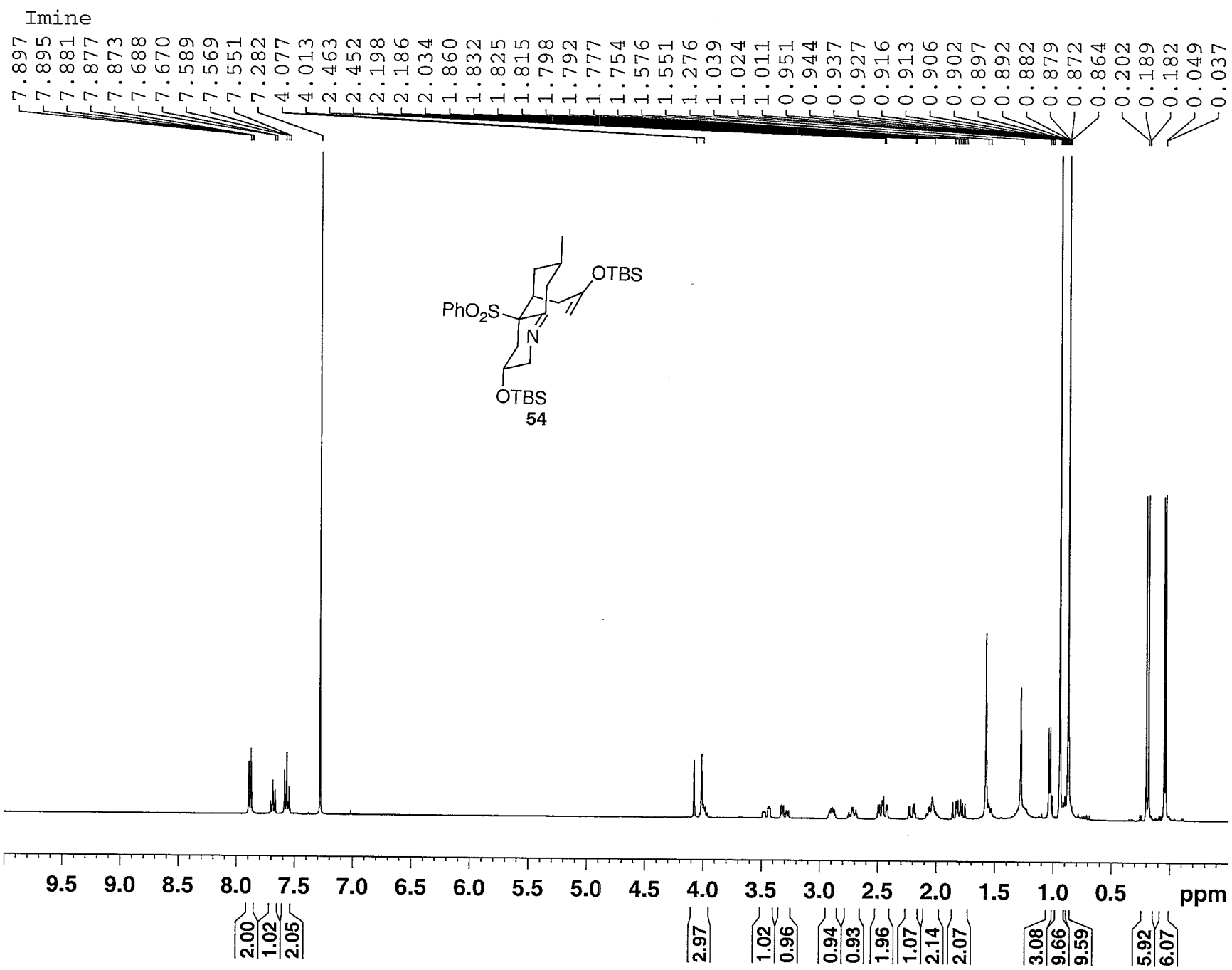
Current Data Parameters
NAME MS IV-15
EXPNO 3
PROCNO 1
DU /m
USER Mrimmoy

F2 - Acquisition Parameters
Date_ 20110209
Time 15.39
INSTRUM DPX400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 6502
DW 20.850 usec
DE 6.00 usec
TE 298.2 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.30 usec
PL1 -3.00 dB
SFO1 100.6517495 MHz

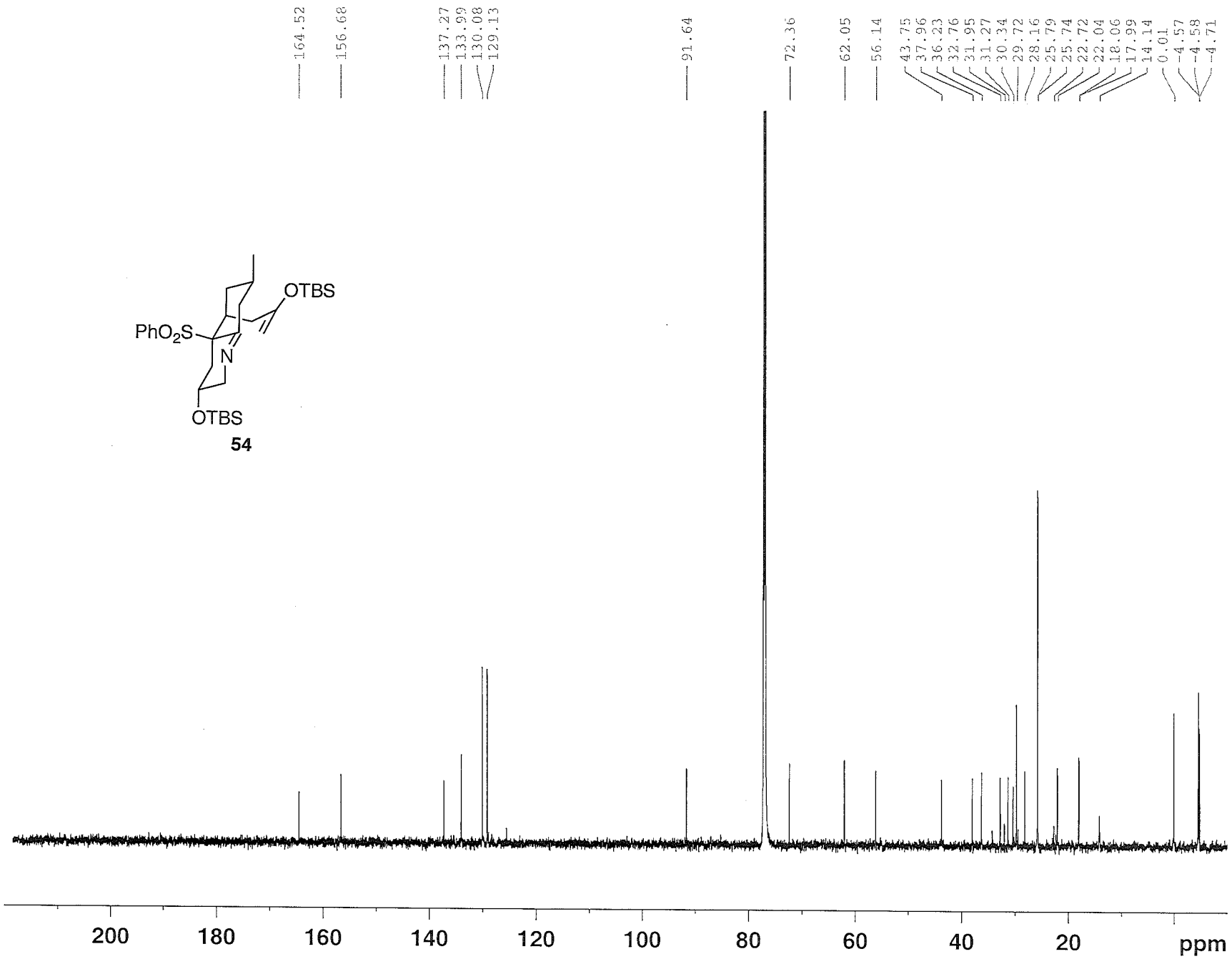
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.2466010 MHz

F2 - Processing parameters
SI 32768
SF 100.6416850 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



NAME	MS IV-23
EXPNO	1
PROCNO	1
Date_	20110216
Time	14:19
INSTRUM	robinson
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6793.478
FIDRES	0.207320
AQ	2.4117749
RG	181
DW	73.600
DE	6.50
TE	298.4
D1	2.00000000
TD0	1
======== CHANNEL f1 ====	
NUC1	1H
P1	14.00
PL1	0.00
SFO1	400.1424008
SI	32768
SF	400.1400000
WDW	EM
SSB	0
LB	0.30
GB	0
PC	1.00

Imine carbon

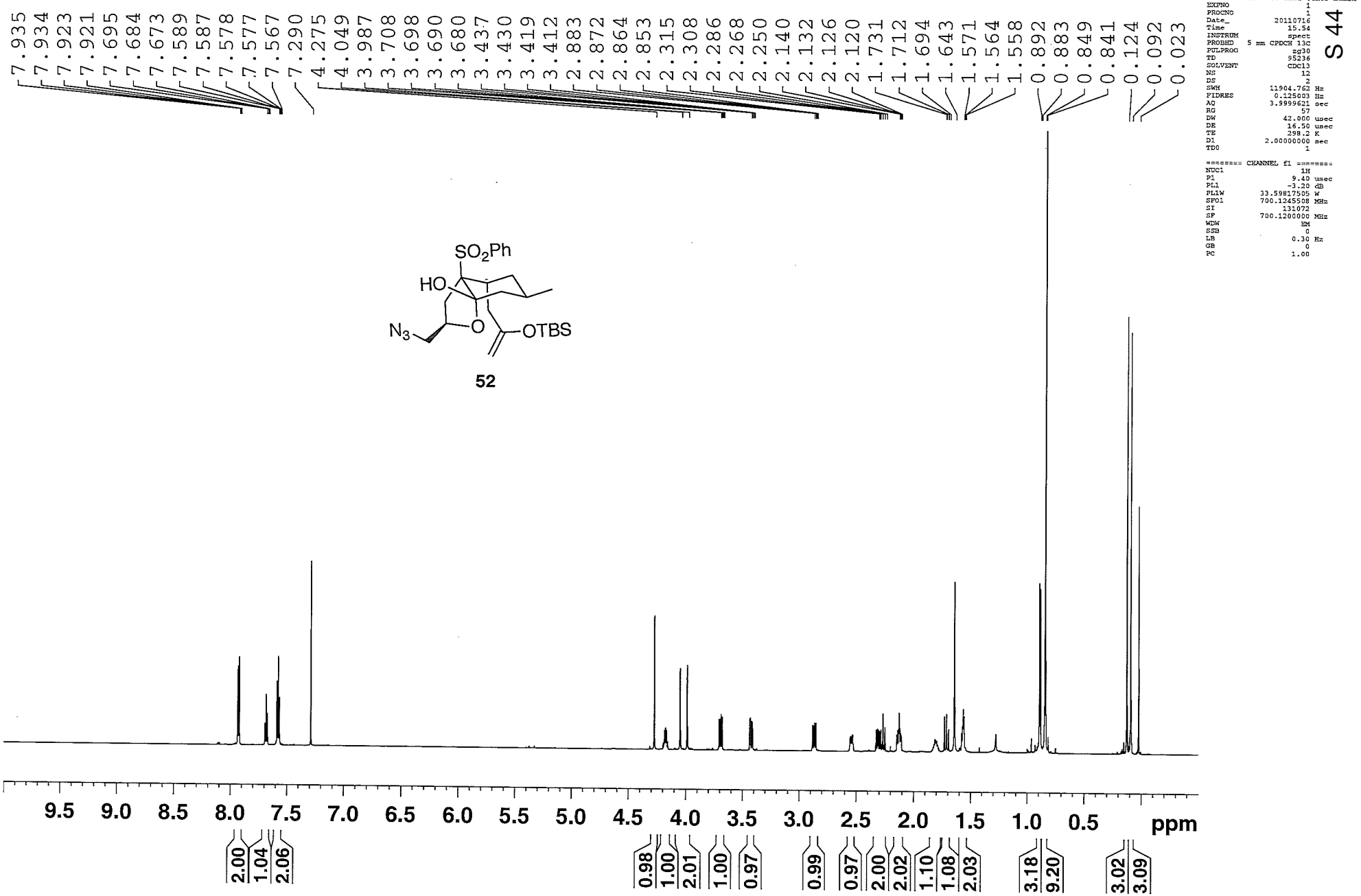



```
NAME          MS III-71
EXPNO               3
PROCNO              1
Date_        20101[illegible]
Time            17.[illegible]
INSTRUM         spe[illegible]
PROBHD   5 mm CPDCH [illegible]
PULPROG        zgpg30
TD             98304
SOLVENT        CDCl3
NS               151
DS                 4
SWH        41666.668 H
FIDRES      0.423855 H
AQ        1.1796980 s
RG              203
DW           12.000 u
DE            15.00 u
TE            297.5 K
D1        2.00000000 s
D11       0.03000000 s
TD0                1

======== CHANNEL f1 =====
NUC1             13C
P1              9.00 u
PL1             4.50 d
PL1W      38.14553833 W
SFO1     176.0629186 M

======== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2             1H
PCPD2           65.00 u
PL2             -3.20 d
PL12            13.60 d
PL13           120.00 d
PL2W      33.59817505 W
PL12W      0.70196527 W
PL13W      0.00000000 W
SFO2     700.1228005 M
SI            131072
SF       176.0453140 M
WDW              EM
SSB               0
LB             3.00 H
GB                0
PC             1.40
```

TBS enol ether with hemiketal




```
NAME        MS V-87-enol ether hemik
EXPNO                1
PROCNO               1
Date_         20110716
Time             15.54
INSTRUM          spect
PROBHD   5 mm CPDCH 13c
PULPROG           zg30
TD               95236
SOLVENT          CDCl3
NS                  12
DS                   2
SWH          11904.762 Hz
FIDRES        0.125003 Hz
AQ           3.9999621 sec
RG                  57
DW             42.000 usec
DE             16.50 usec
TE             298.2 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               9.40 usec
PL1             -3.20 dB
PL1W        33.59817505 W
SFO1        700.1245508 MHz
SI               131072
SF          700.1200000 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

S 44

SO_2Ph, HO, N_3, O, OTBS

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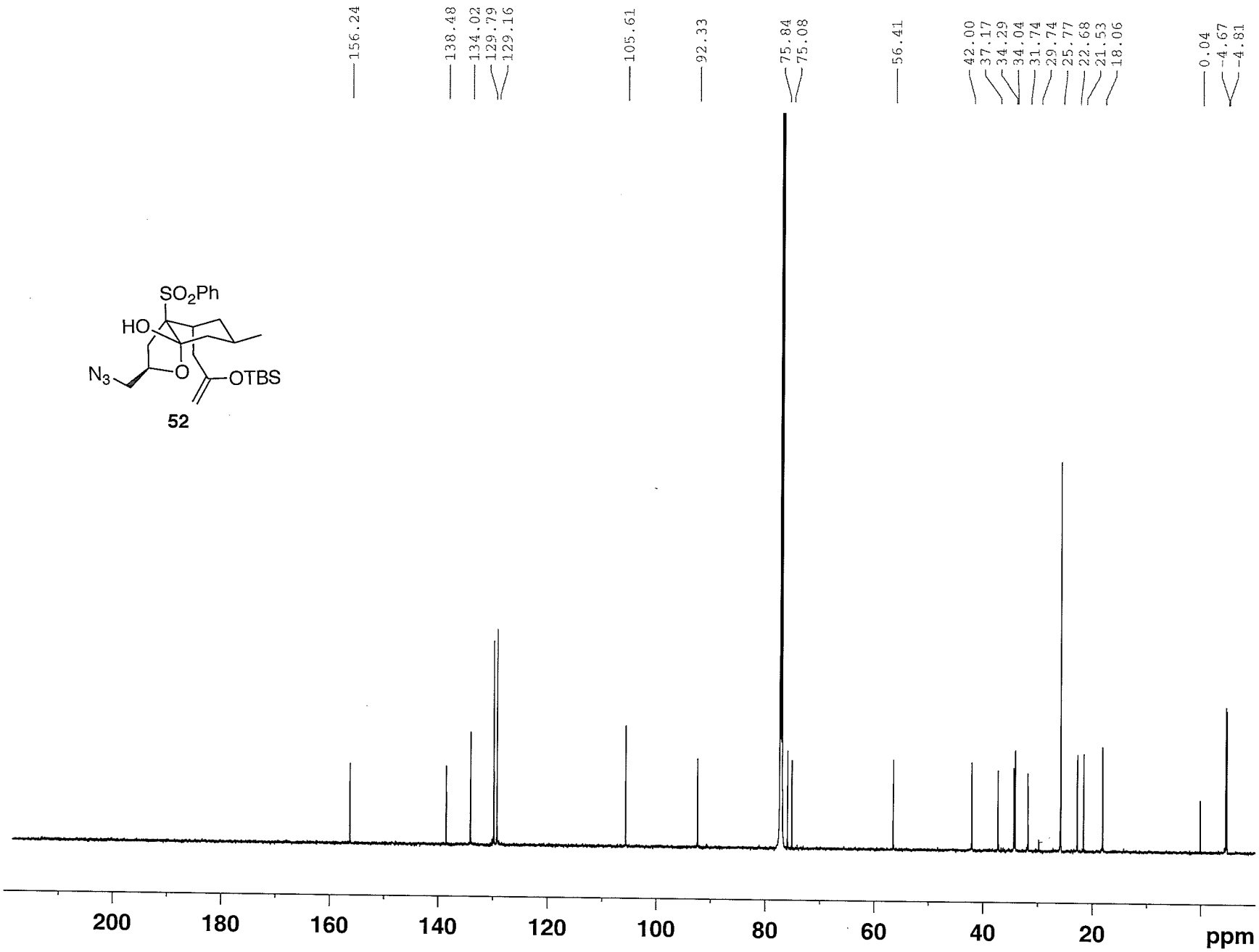
TBS enol ether with hemiketal carbon



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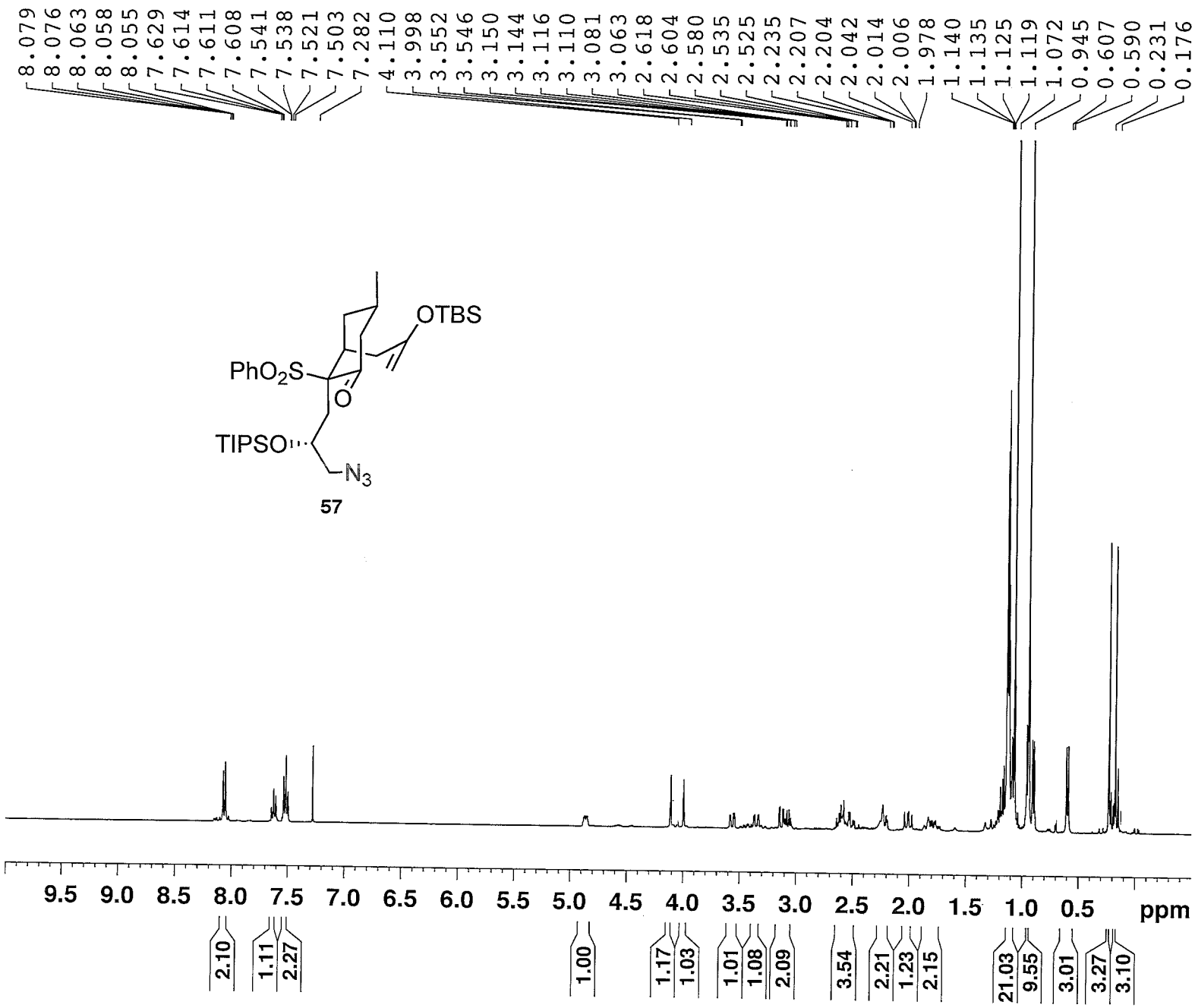
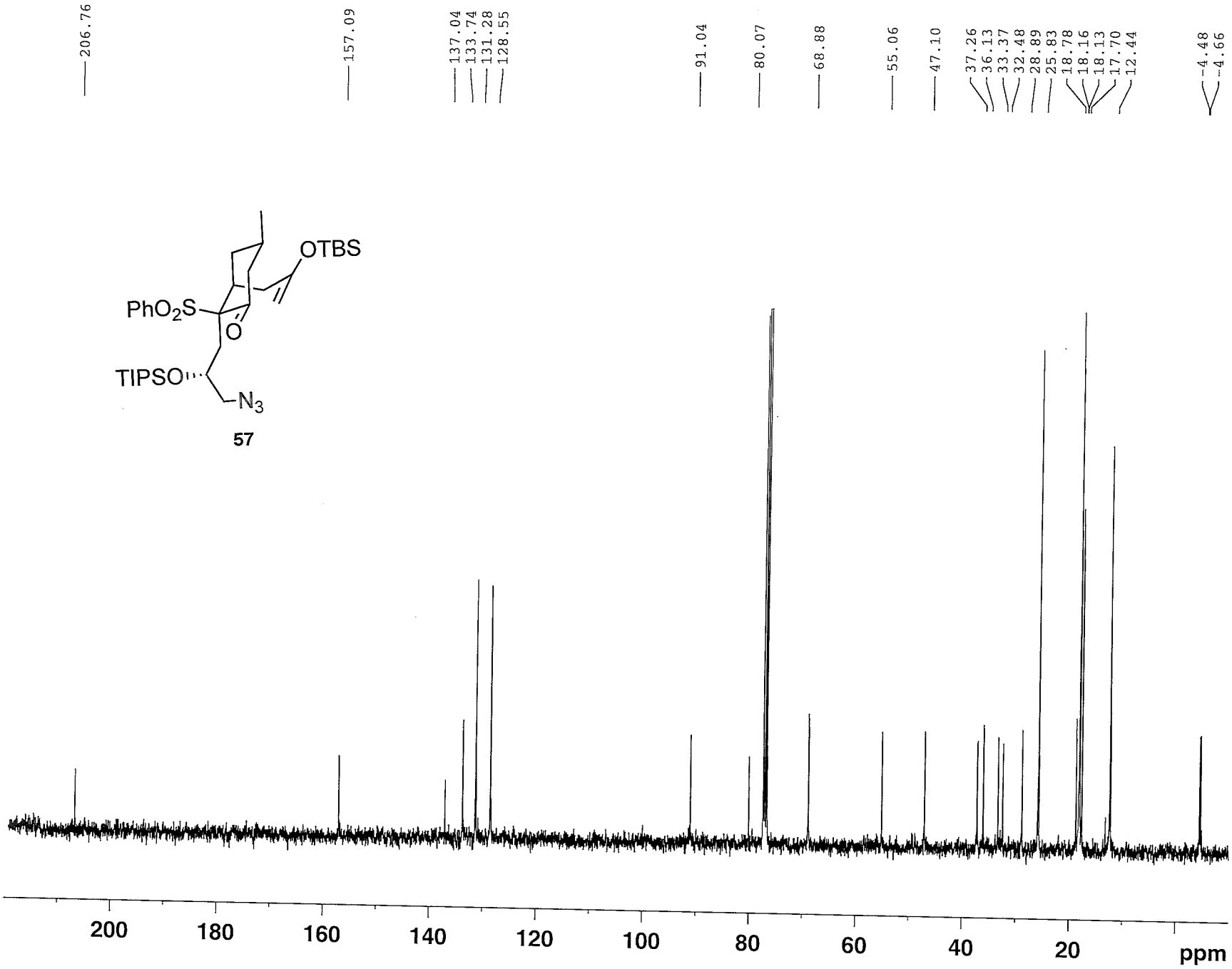
```
NAME        MS V-87-enol ether hemik
EXPNO                              2
PROCNO                             1
Date_                       20110716
Time                            16.04
INSTRUM                         spect
PROBHD     5 mm CPDCH 13C
PULPROG                        zgpg30
TD                              98304
SOLVENT                         CDCl3
NS                                402
DS                                  4
SWH                        41666.668 Hz
FIDRES                      0.423855 Hz
AQ                          1.1796980 sec
RG                                203
DW                             12.000 usec
DE                             15.00 usec
TE                             298.2 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                                 1

======== CHANNEL f1 ========
NUC1                              13C
P1                               9.00 usec
PL1                              4.50 dB
PL1W                     38.14553833 W
SFO1                    176.0629186 MHz

======== CHANNEL f2 ========
CPDPRG2                       waltz16
NUC2                               1H
PCPD2                           65.00 usec
PL2                             -3.20 dB
PL12                            13.60 dB
PL13                           120.00 dB
PL2W                     33.59817505 W
PL12W                     0.70196527 W
PL13W                     0.00000000 W
SFO2                    700.1228005 MHz
SI                             131072
SF                      176.0453140 MHz
WDW                                EM
SSB                                 0
LB                               3.00 Hz
GB                                  0
PC                               1.40
```

TIPS ether



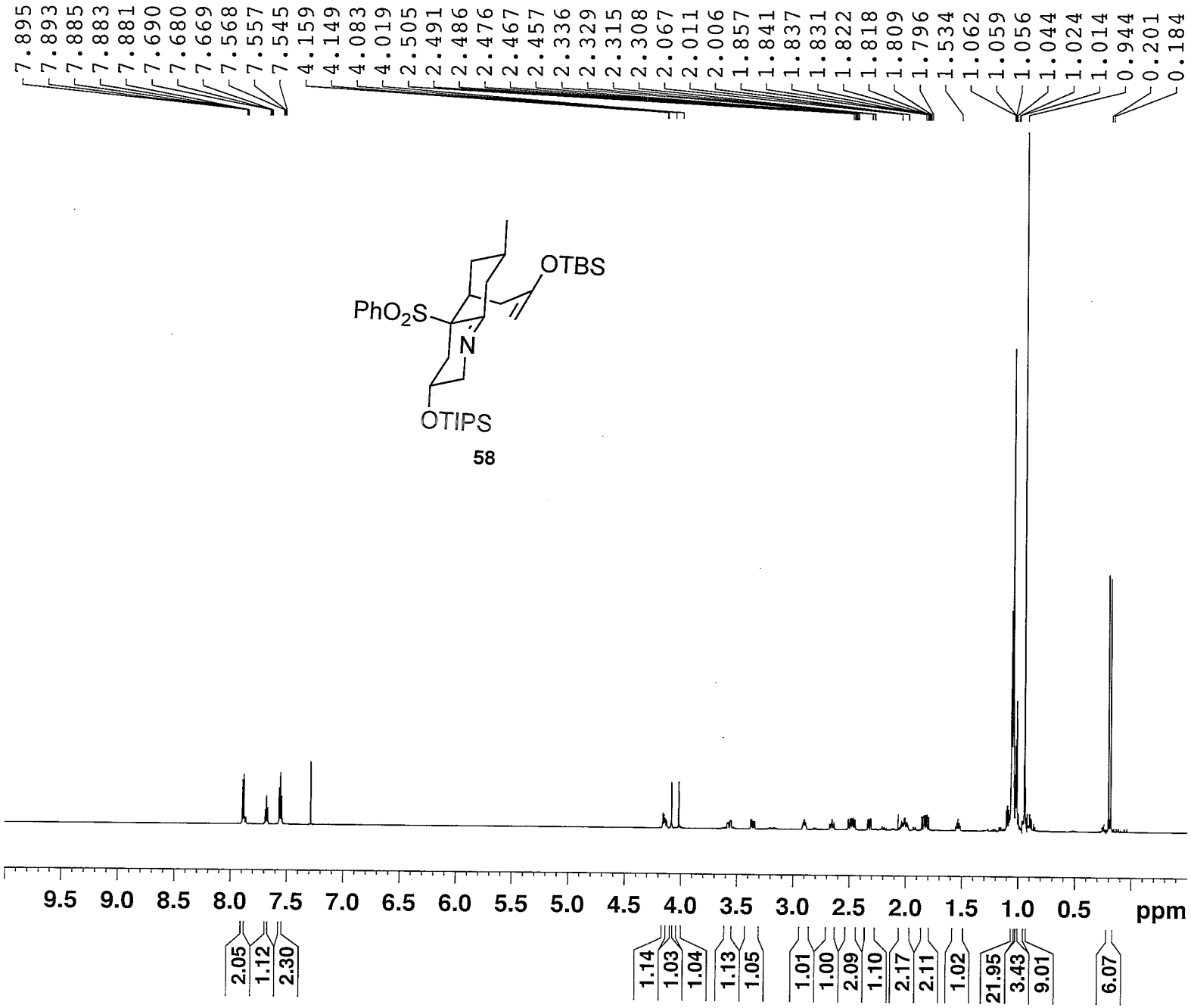

```
NAME        MS VIII-36 TIPS eth
EXPNO                        1
PROCNO                       1
Date_                 20120614
Time                      0.36
INSTRUM                robinson
PROBHD        5 mm PABBO BB-
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          10
DS                           2
SWH                   7183.908 Hz
FIDRES                0.219235 Hz
AQ                   2.2807028 sec
RG                        40.3
DW                      69.600 usec
DE                        6.50 usec
TE                       298.7 K
D1                  2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       11.20 usec
PL1                       0.00 dB
SFO1                400.1428010 MHz
SI                        32768
SF                400.1400000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

TIPS ether carbon



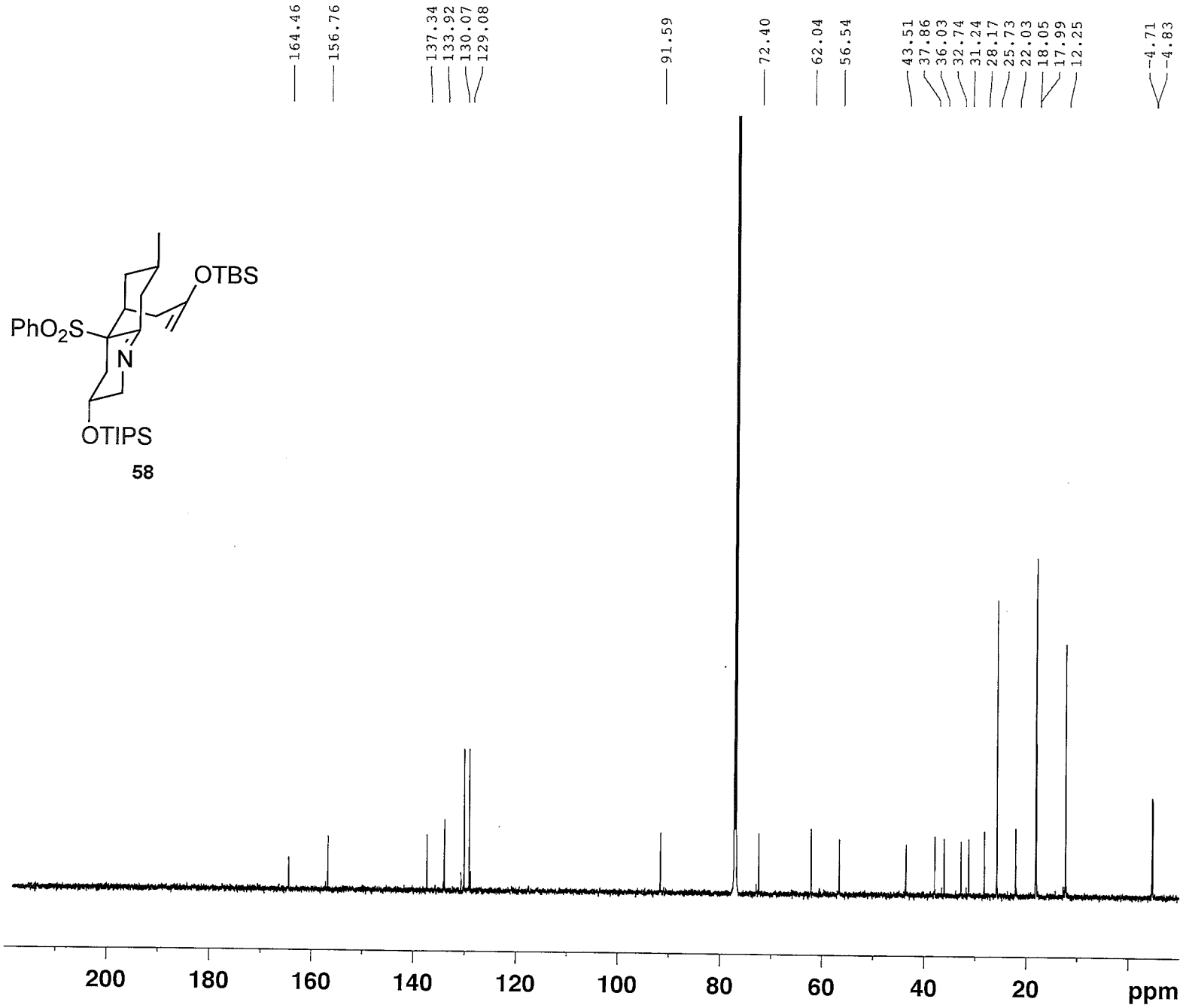

```
NAME        MS VIII-36 TIPS ether
EXPNO                           3
PROCNO                          1
Date_                    20120614
Time                         0.41
INSTRUM                   robinson
PROBHD        5 mm PABBO BB-
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            307
DS                              4
SWH                     23980.814 Hz
FIDRES                   0.365918 Hz
AQ                      1.3664756 sec
RG                          16384
DW                         20.850 usec
DE                          6.50 usec
TE                         299.3 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                           9.00 usec
PL1                         -2.00 dB
SFO1                100.6253446 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                       90.00 usec
PL2                          0.00 dB
PL12                        16.16 dB
PL13                        17.00 dB
SFO2                400.1416006 MHz
SI                          32768
SF                  100.6152830 MHz
WDW                            EM
SSB                             0
LB                           3.00 Hz
GB                              0
PC                           1.40
```

Imine



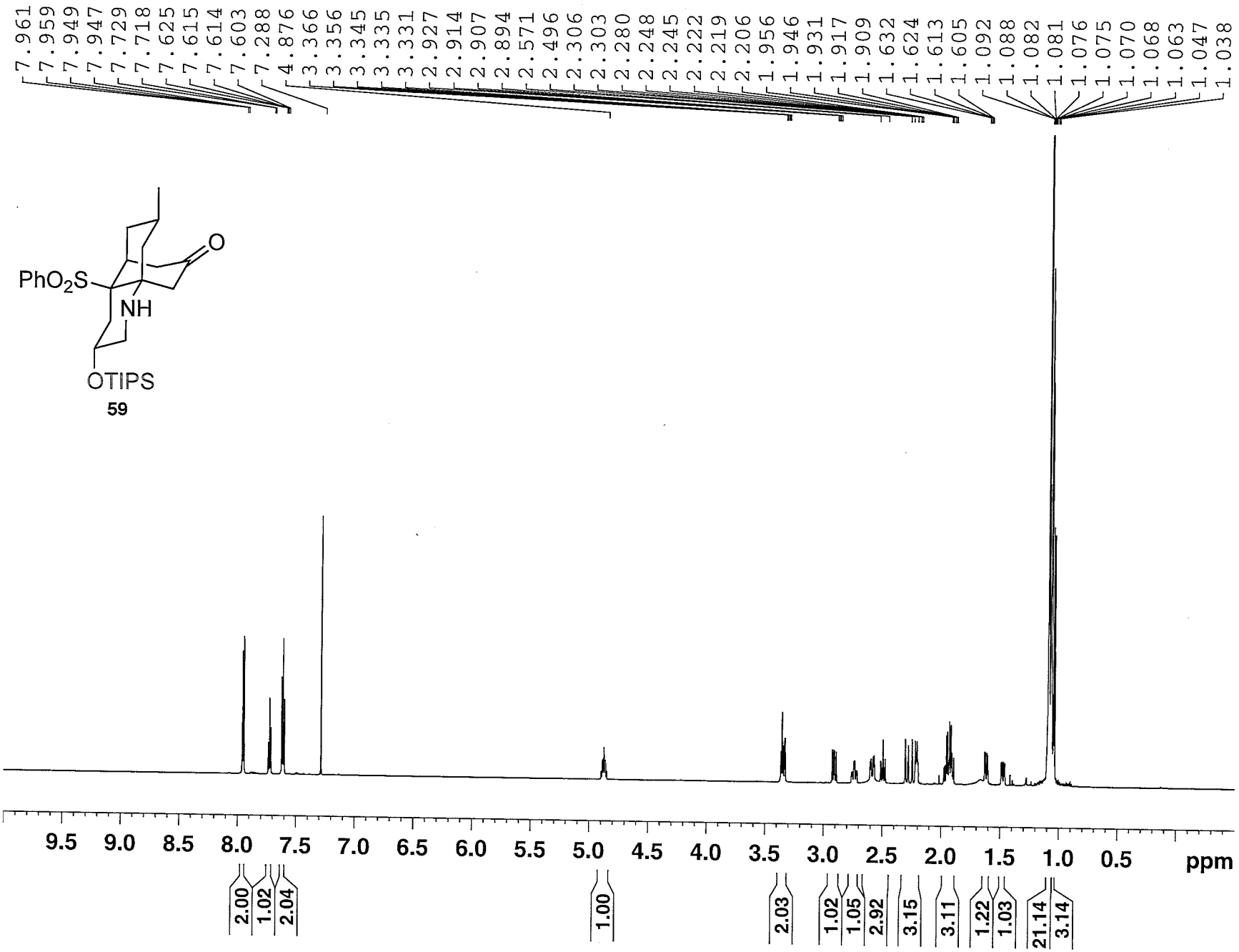
NAME	MS VIII-45 Imine
EXPNO	2
PROCNO	1
Date_	20120618
Time	16.30
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	2
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 se
RG	22.6
DW	42.000 us
DE	6.50 us
TE	298.2 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	9.40 us
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MH
SI	131072
SF	700.1471400 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Imine carbon



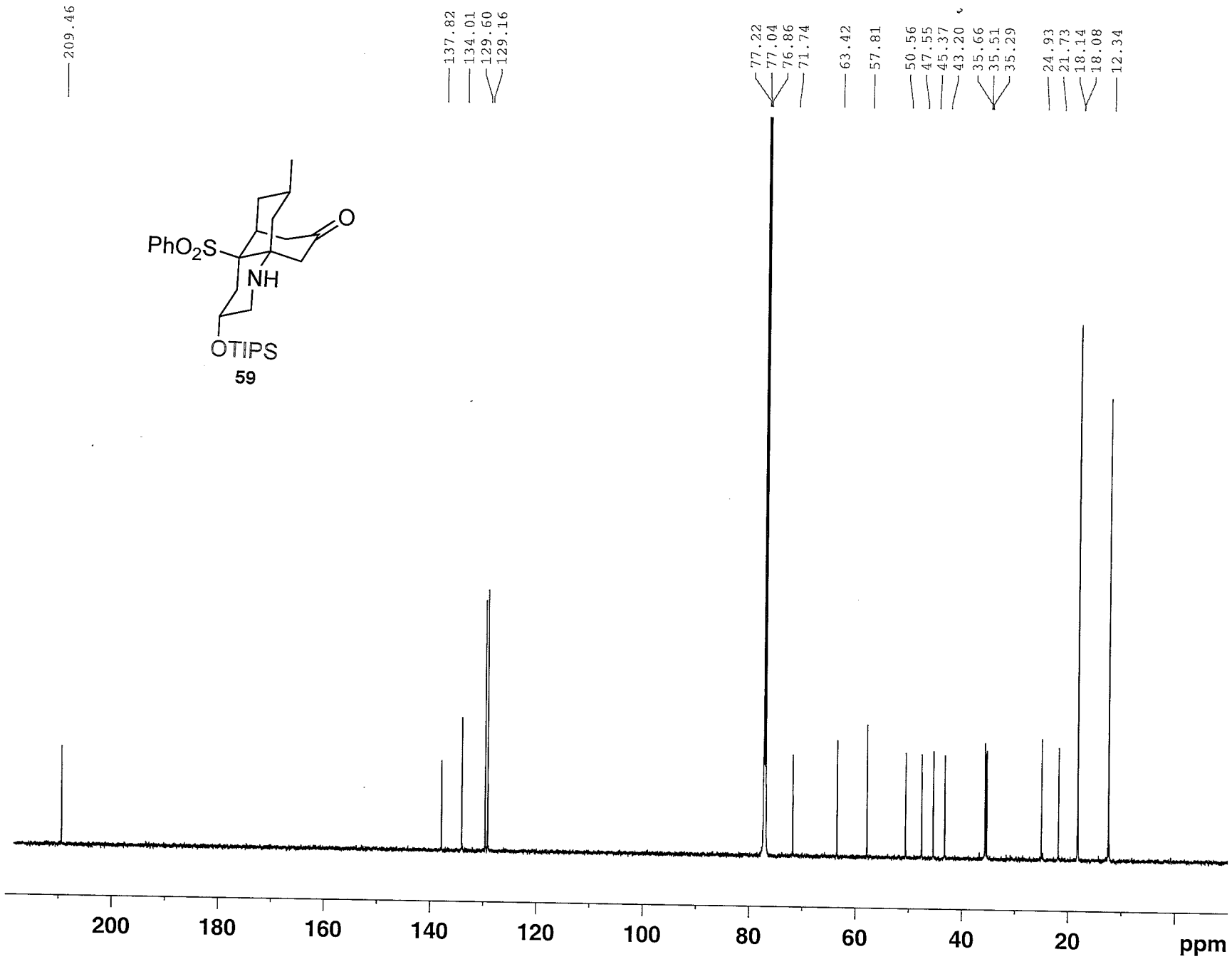
NAME	MS VIII-45 Imine
EXPNO	3
PROCNO	1
Date_	20120618
Time	16.35
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	20
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 se
RG	203
DW	12.000 us
DE	16.50 us
TE	298.2 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.00 us
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MH
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 us
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MH
SI	32768
SF	176.0521380 MH
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Tricycle



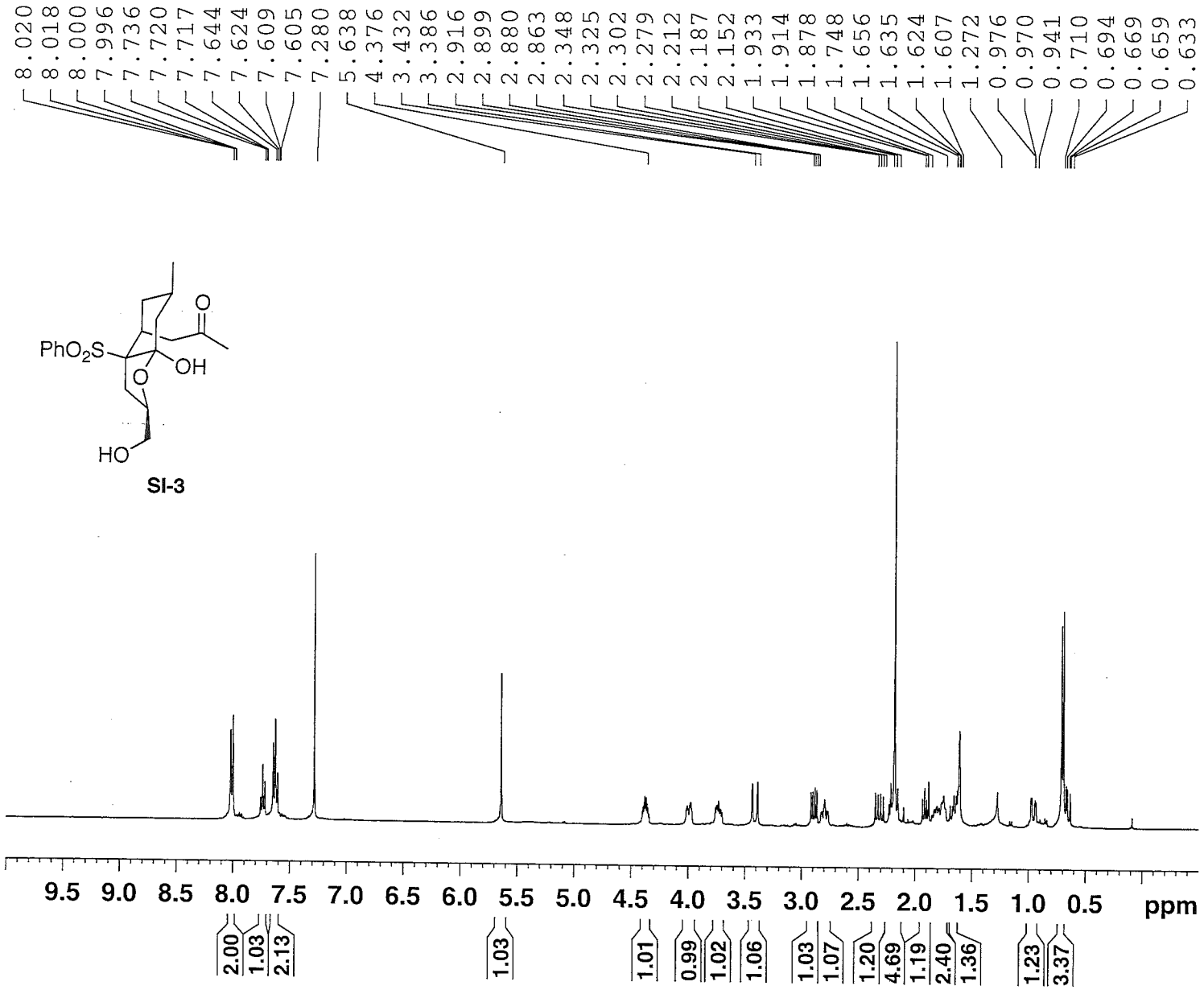

```
NAME        MS VII-86 tri
EXPNO
PROCNO
Date_       20120419
Time        15.
INSTRUM     spect
PROBHD      5 mm CPDCH 13C
PULPROG     zg30
TD          95236
SOLVENT     CDCl3
NS          10
DS          2
SWH         11904.762 H
FIDRES      0.125003 H
AQ          3.9999621 s
RG          32
DW          42.000 u
DE          6.50 u
TE          303.2 K
D1          2.00000000 s
TD0         1

======== CHANNEL f1 =====
NUC1        1H
P1          9.40 u
PL1         -3.20 d
PL1W        33.59817505 W
SFO1        700.1516910 M
SI          131072
SF          700.1471400 M
WDW         EM
SSB         0
LB          0.30 H
GB          0
PC          1.00
```

Tricycle carbon



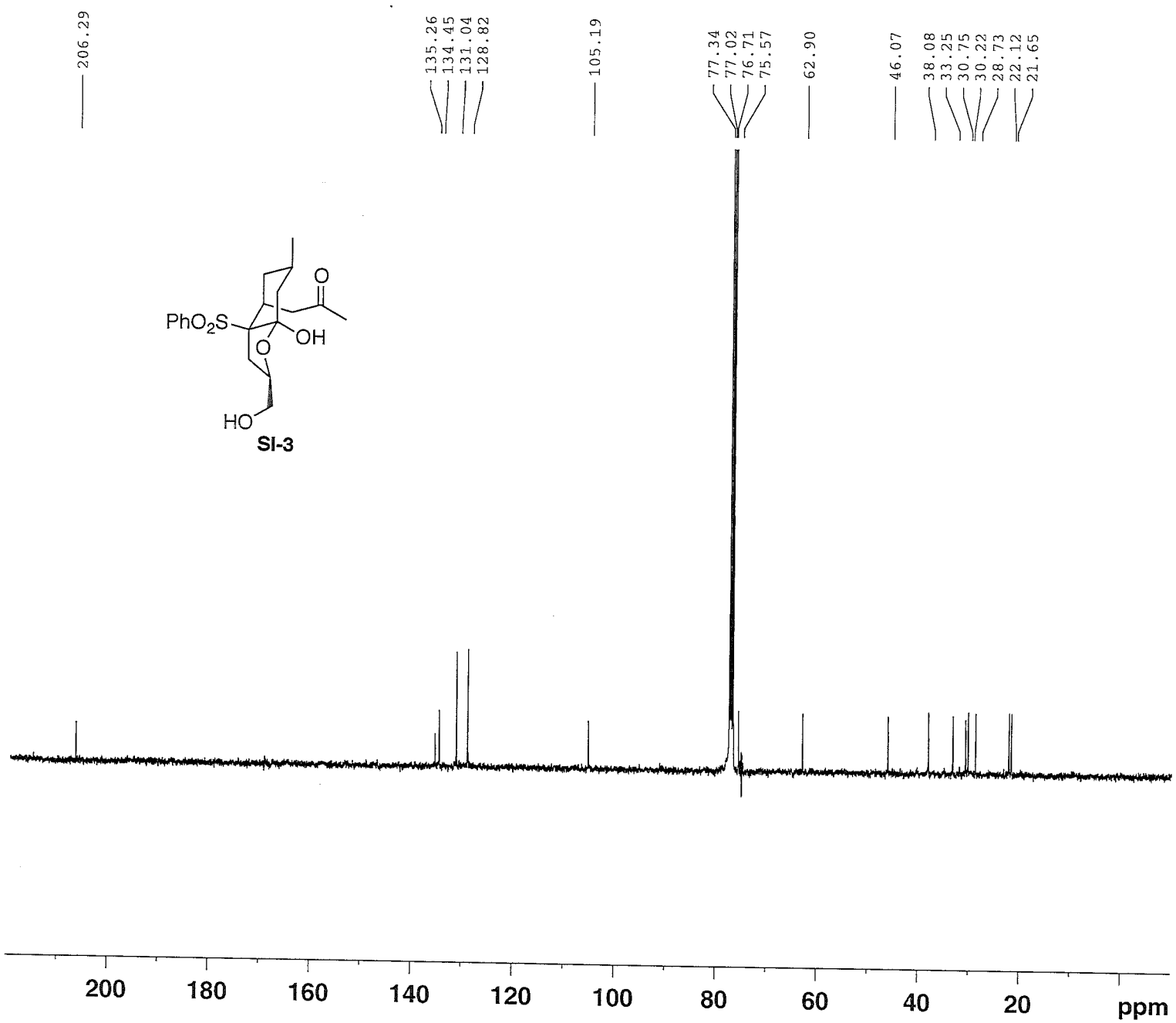
NAME	MS VII-86 tricyc
EXPNO	12
PROCNO	50
Date_	20120415
Time	9.57
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	140
DS	4
SWH	41666.668 H
FIDRES	0.635783 H
AQ	0.7864820 s
RG	203
DW	12.000 u
DE	16.50 u
TE	303.2 K
D1	2.00000000 s
D11	0.03000000 s
TD0	1
======== CHANNEL f1 =====	
NUC1	13C
P1	9.00 u
PL1	4.50 d
PL1W	38.14553833 W
SFO1	176.0697436 M
======== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 u
PL2	-3.20 d
PL12	13.60 d
PL13	120.00 d
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 M
SI	32768
SF	176.0521380 M
WDW	EM
SSB	0
LB	3.00 H
GB	0
PC	1.40

[(S)-C10] Hemiketal




```
Current Data Parameters
NAME              MSII 50
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20100328
Time               18.56
INSTRUM          DPX400
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ            2.5559540 sec
RG                645.1
DW               78.000 usec
DE                 6.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.50 usec
PL1               -3.00 dB
SFO1        400.2478017 MHz

F2 - Processing parameters
SI                 32768
SF          400.2450000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

[(S)-C10] Hemiketal carbon



Current Data Parameters
NAME MSII 50
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100329
Time 17.02
INSTRUM DPX400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2275
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3649.1
DW 20.850 use
DE 6.00 use
TE 299.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

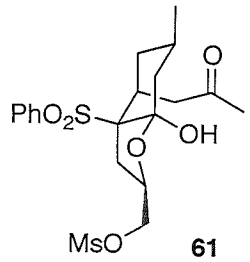
======== CHANNEL f1 =======
NUC1 13C
P1 8.30 use
PL1 -3.00 dB
SFO1 100.6517495 MH:

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.2466010 MH:

F2 - Processing parameters
SI 32768
SF 100.6416850 MH:
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Mesylate

8.019 8.017 8.003 7.998 7.995 7.755 7.739 7.736 7.659 7.638 7.620 7.291 5.604 4.530 4.503 4.475 4.467 4.442 4.431 4.414 3.349 3.303 3.092 3.046 3.029 3.010 2.993 2.351 2.328 2.305 2.282 2.188 2.156 2.132 2.120 2.100 2.096 1.923 1.907 1.887 1.639 1.621 1.004 0.998 0.722 0.705



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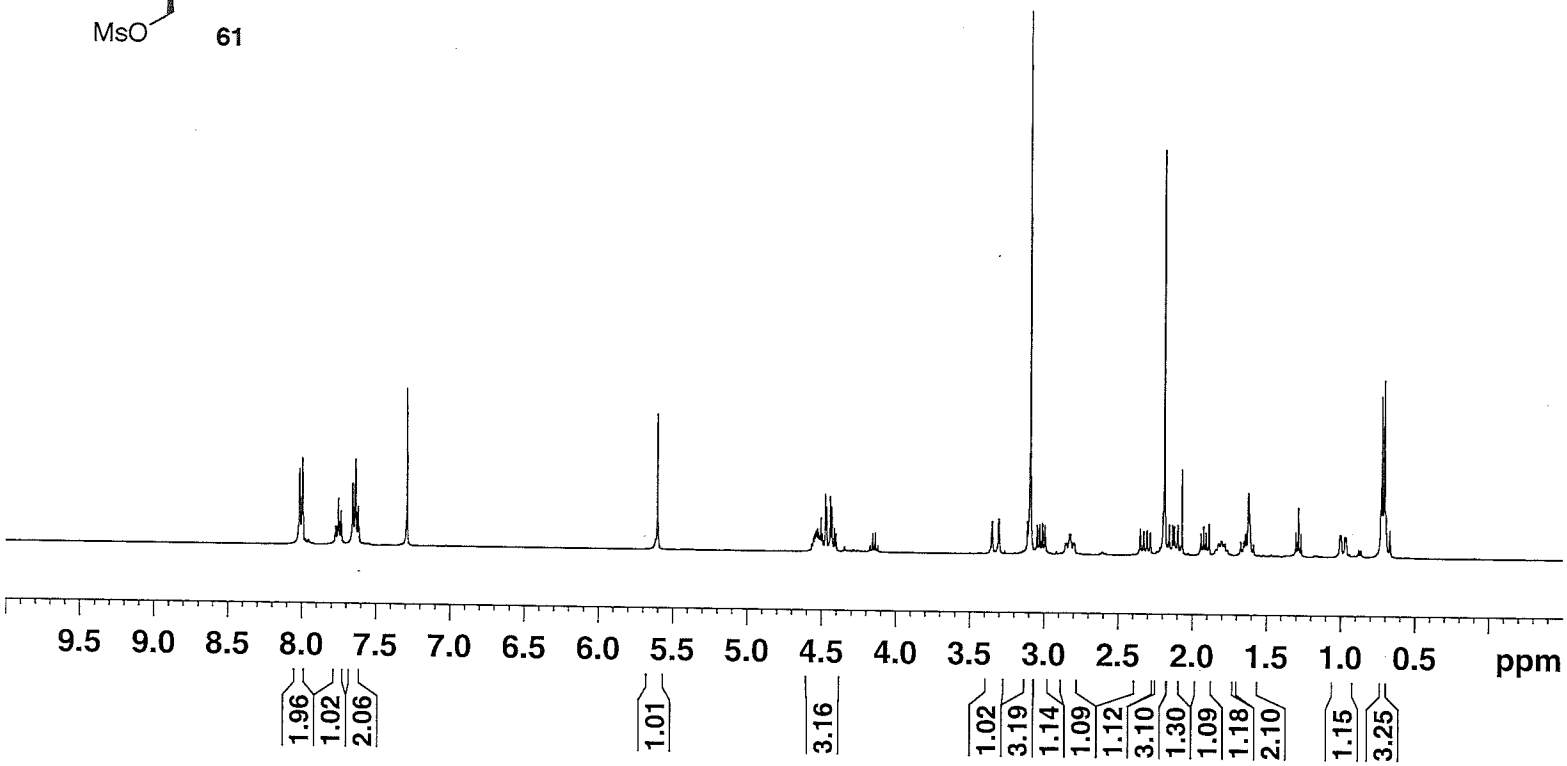
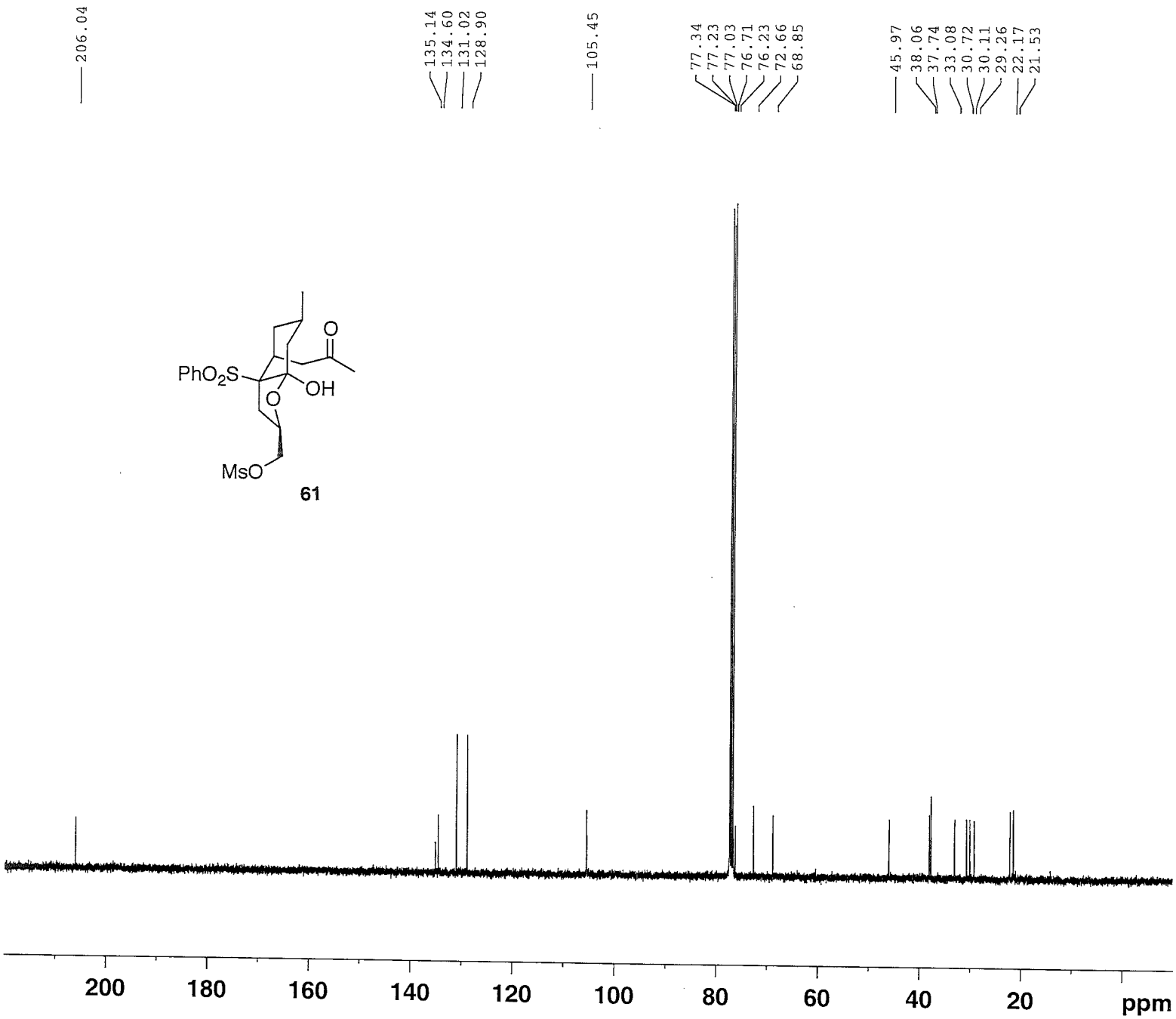


NAME	MSII62
EXPNO	2
PROCNO	1
Date_	20100418
Time	14.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 se
RG	128
DW	78.000 us
DE	6.50 us
TE	299.0 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.00 us
PL1	0.00 dB
PL1W	10.27361584 W
SFO1	400.1378009 MH
SI	32768
SF	400.1350000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

S 54

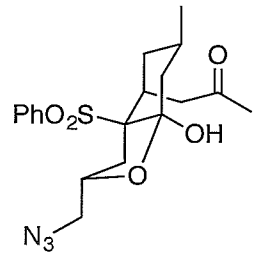
Mesylate 13C



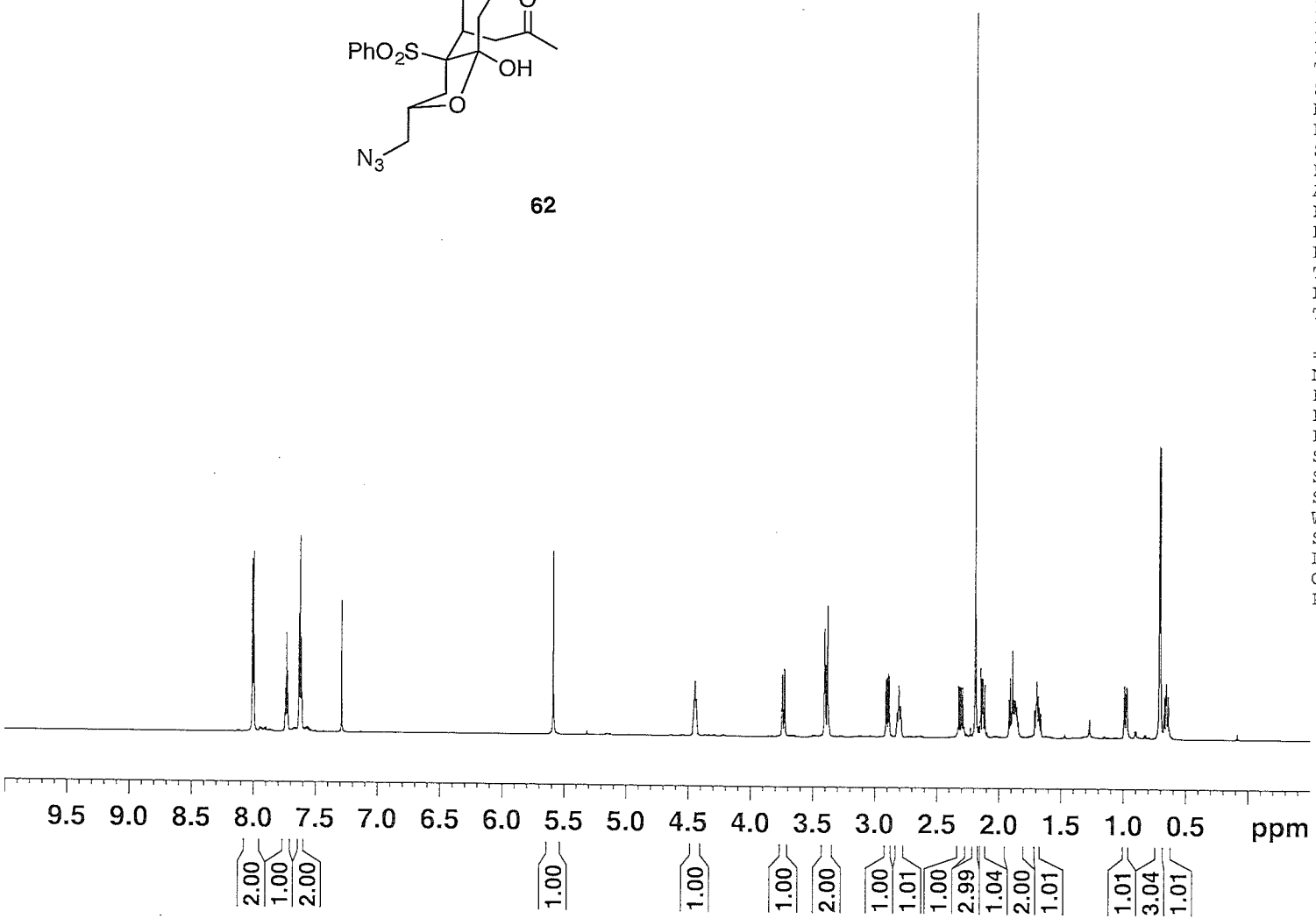
NAME	MSII62	
EXPNO	3	
PROCNO	1	
Date_	20100418	
Time	14.27	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	932	
DS	4	
SWH	25125.629	Hz
FIDRES	0.383387	Hz
AQ	1.3042164	sec
RG	14596.5	
DW	19.900	use
DE	6.50	use
TE	299.7	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 =======		
NUC1	13C	
P1	9.00	use
PL1	-2.00	dB
PL1W	46.89702606	W
SFO1	100.6255966	MHz
======== CHANNEL f2 =======		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	use
PL2	0.00	dB
PL12	16.16	dB
PL13	17.00	dB
PL2W	10.27361584	W
PL12W	0.24872722	W
PL13W	0.20498557	W
SFO2	400.1366005	MHz
SI	32768	
SF	100.6140260	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

XL-vi-23-Azide 10(S)

8.009 7.998 7.745 7.734 7.723 7.634 7.622 7.611 5.587 4.447 4.442 3.747 3.742 3.728 3.723 3.403 3.397 3.378 2.911 2.902 2.890 2.881 2.805 2.326 2.313 2.300 2.287 2.188 2.147 2.133 2.126 2.112 1.908 1.888 1.692 0.992 0.988 0.972 0.968 0.712 0.703 0.673 0.658 0.653

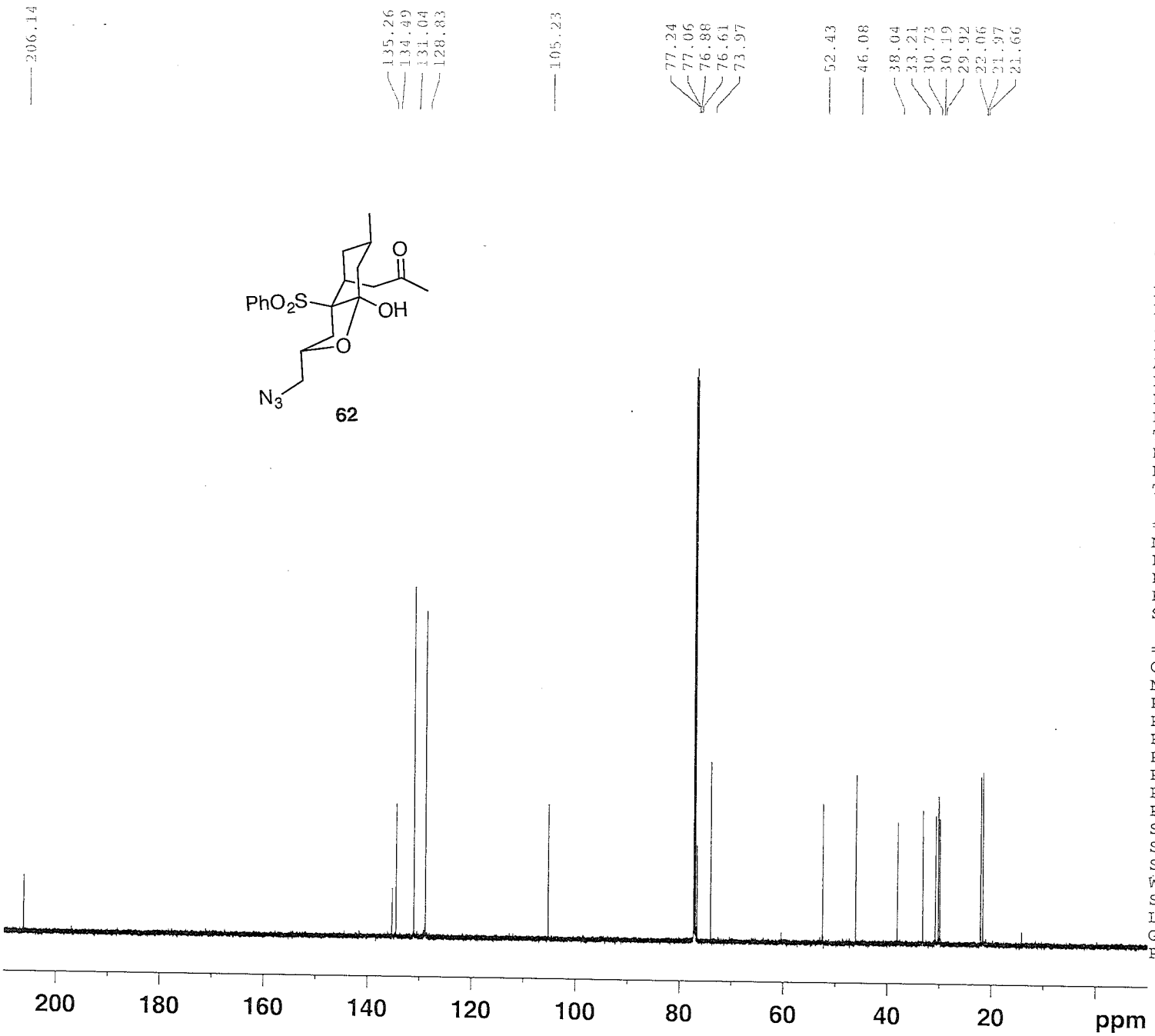


62



NAME	XL-vi-23-Azide
EXPNO	7
PROCNO	1
Date_	20150705
Time	10.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	14423.077 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	80.6
DW	34.667 usec
DE	6.50 usec
TE	298.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.75 usec
PL1	-3.00 dB
PL1W	32.08600616 W
SFO1	700.1514637 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

XL-vi-23-Azide 10(S)



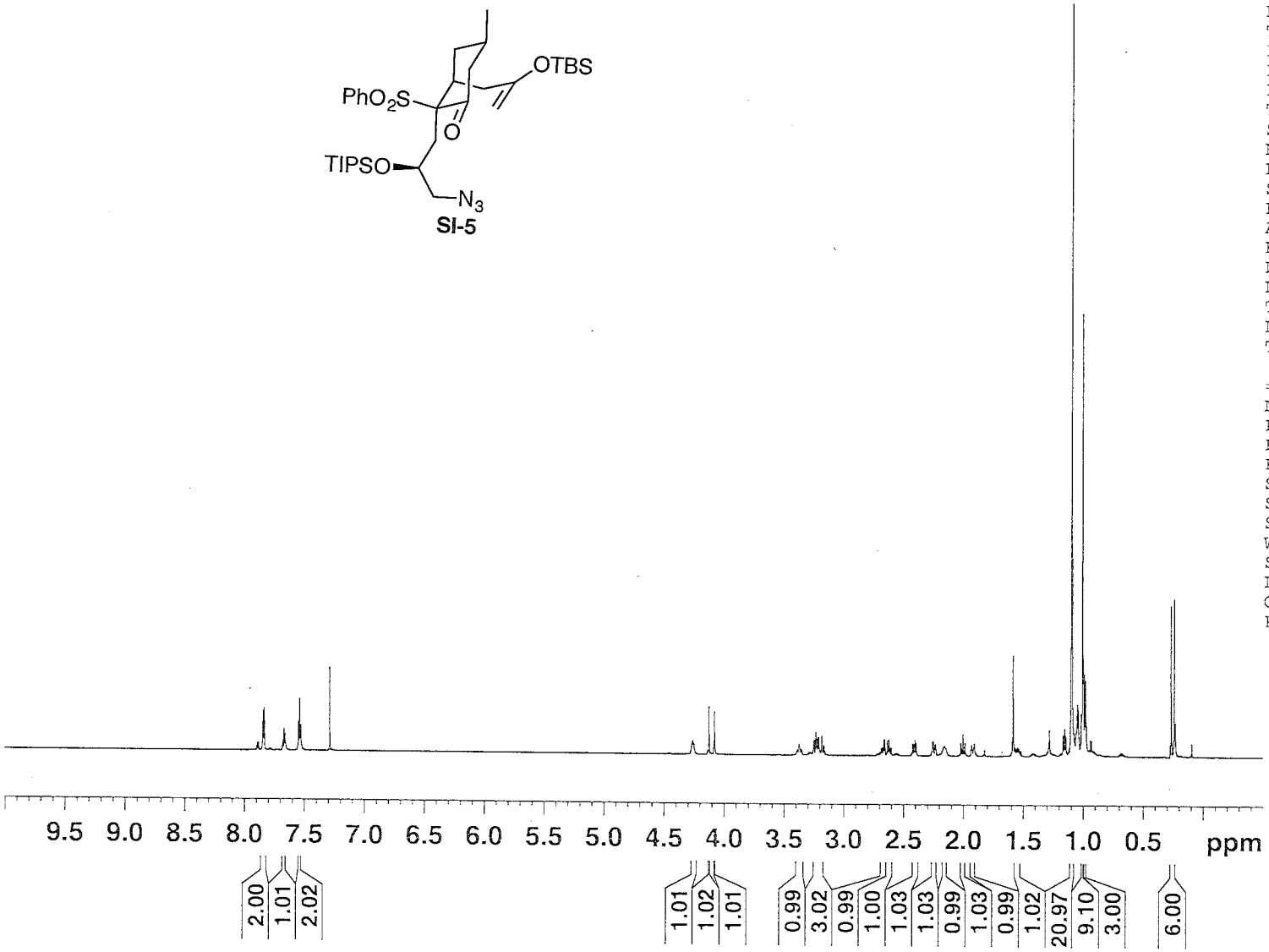
BRUKER

NAME	XL-vi-23-Azide
EXPNO	4
PROCNO	1
Date_	20150703
Time	19.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	207
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	21.27 usec
TE	299.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	2.00 dB
PL1W	67.83342743 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-3.00 dB
PL12	12.30 dB
PL13	12.30 dB
PL2W	32.08600616 W
PL12W	0.94692516 W
PL13W	0.94692516 W
SFO2	700.1499406 MHz
SI	131072
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

XL-vi-49-TIPS-ether

7.847 7.836 7.671 7.661 7.552 7.541 7.530 4.263 4.125 4.079 3.252 3.234 3.217 3.209 3.186 3.181 2.666 2.656 2.633 2.621 2.403 2.393 2.260 2.253 2.022 2.004 1.986 1.171 1.157 1.146 1.096 1.073 1.056 1.048 1.039 1.016 1.007 1.000 0.988 0.979 0.941 0.931 0.269 0.242

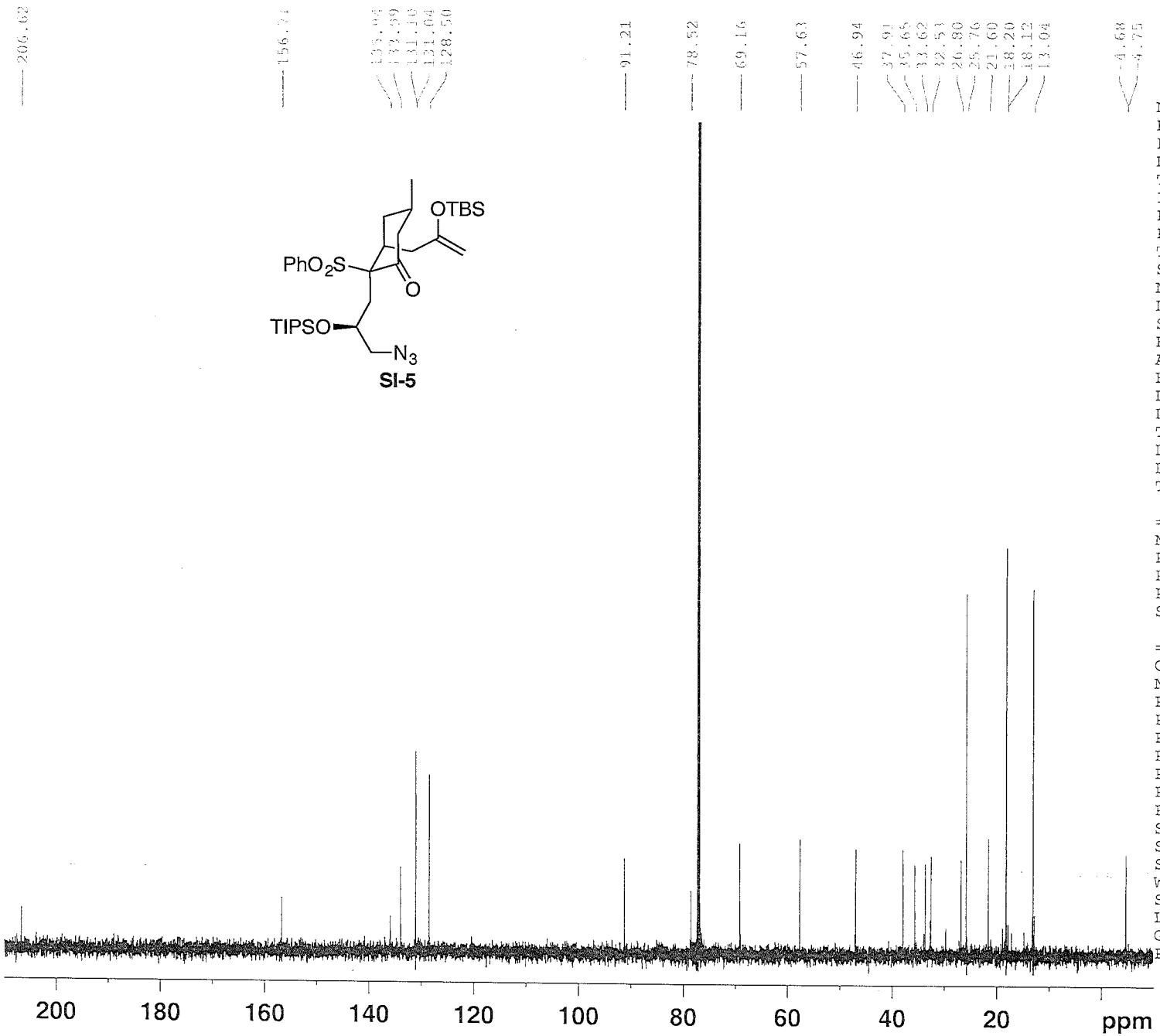
SI-5



BRUKER

NAME	XL-vi-49-TIPS-ether
EXPNO	2
PROCNO	1
Date_	20150801
Time	22.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	14423.077 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	144
DW	34.667 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.75 usec
PL1	-3.00 dB
PL1W	32.08600616 W
SFO1	700.1514637 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

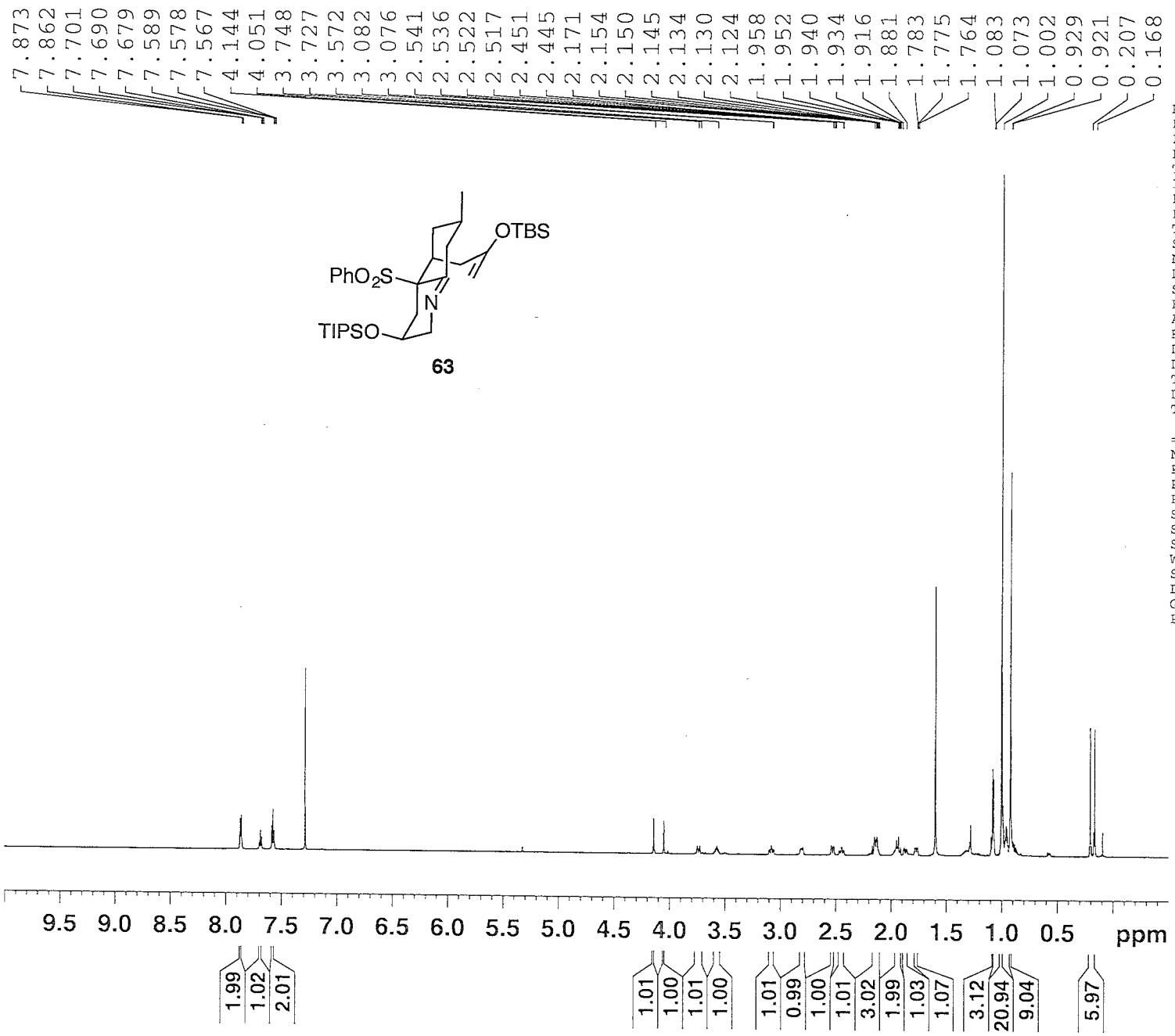
XL-vi-49-TIPS-ether



BRUKER

NAME	XL-vi-49-TIPS-ether
EXPNO	3
PROCNO	1
Date_	20150801
Time	23.06
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	16384
SOLVENT	CDCl3
NS	601
DS	0
SWH	41666.668 Hz
FIDRES	2.543132 Hz
AQ	0.1966580 sec
RG	203
DW	12.000 usec
DE	21.27 usec
TE	298.8 K
D1	0.80000001 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	2.00 dB
PL1W	67.83342743 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-3.20 dB
PL12	11.20 dB
PL13	12.30 dB
PL2W	33.59817505 W
PL12W	1.21987605 W
PL13W	0.94692516 W
SFO2	700.1499406 MHz
SI	131072
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

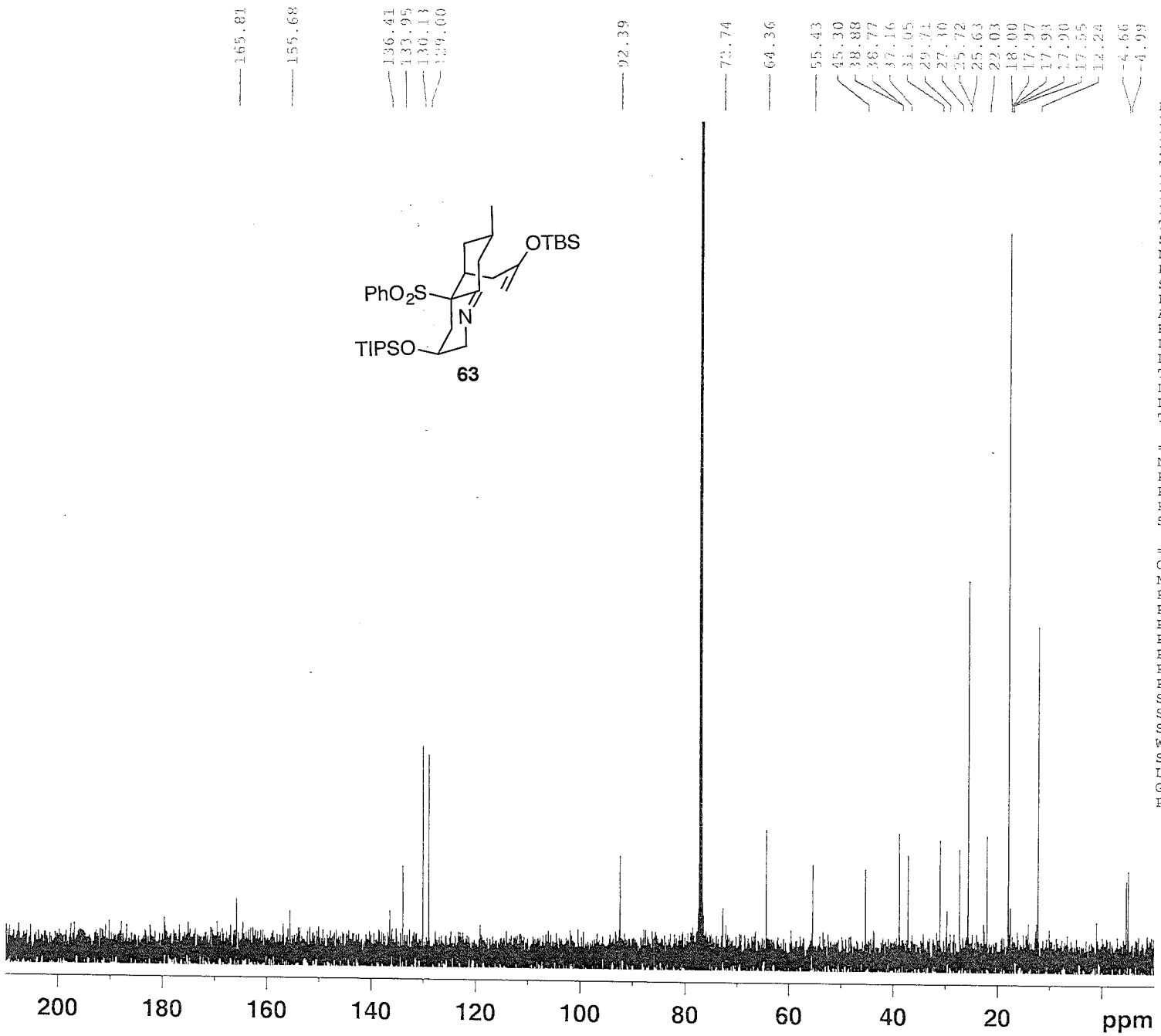
XL-vi-53-10(S)-TIPS-imine



BRUKER

NAME	XL-vi-47-10(S)-TIPS-imine
EXPNO	1
PROCNO	1
Date_	20150803
Time	9.47
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	14423.077 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	203
DW	34.667 usec
DE	6.50 usec
TE	298.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.75 usec
PL1	-3.00 dB
PL1W	32.08600616 W
SFO1	700.1514637 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

XL-vi-53-10(S)-TIPS-imine



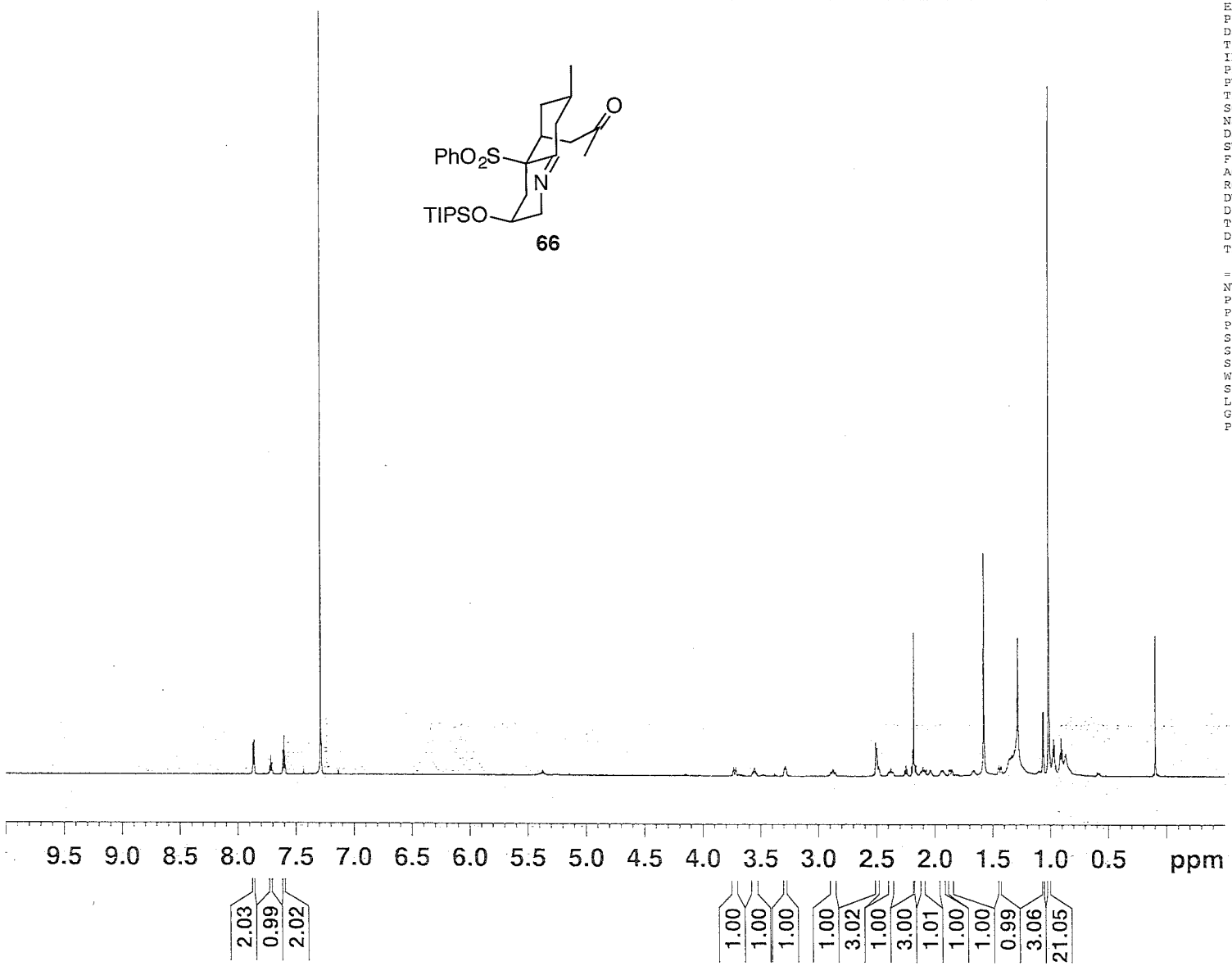
BRUKER

NAME	XL-vi-47-10(S)-TIPS-imine
EXPNO	2
PROCNO	1
Date_	20150803
Time	10.09
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	16384
SOLVENT	CDCl3
NS	1024
DS	0
SWH	41666.668 Hz
FIDRES	2.543132 Hz
AQ	0.1966580 sec
RG	203
DW	12.000 usec
DE	21.27 usec
TE	298.3 K
D1	0.80000001 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	2.00 dB
PL1W	67.83342743 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-3.20 dB
PL12	11.20 dB
PL13	12.30 dB
PL2W	33.59817505 W
PL12W	1.21987605 W
PL13W	0.94692516 W
SFO2	700.1499406 MHz
SI	131072
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

XL-vi-54-10(s)-TIPS-methyl-ketone

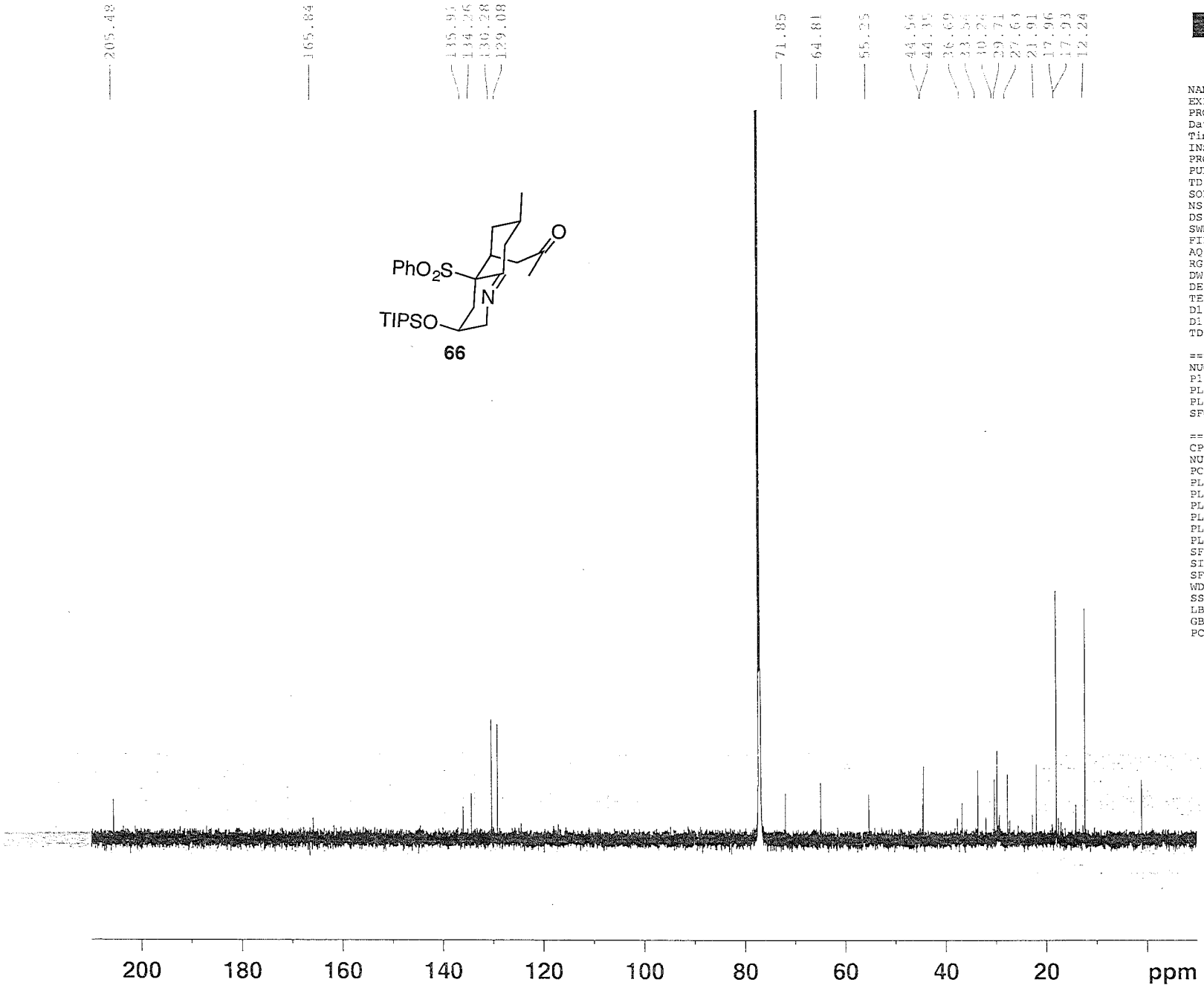
7.871 7.860 7.728 7.717 7.706 7.614 7.603 7.592 3.736 3.715 3.562 3.554 3.546 3.297 3.291 3.284 3.278 2.876 2.870 2.506 2.501 2.494 2.482 2.475 2.375 2.247 2.236 2.199 2.188 2.179 2.162 2.101 2.087 2.072 2.041 1.938 1.929 1.870 1.851 1.848 1.666 1.068 1.059 1.016

66



NAME	XL-vi-54-10(s)-TIPS-methyl-ketone
EXPNO	5
PROCNO	1
Date_	20150805
Time	21.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	14423.077 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	203
DW	34.667 usec
DE	6.50 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.25 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1514637 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

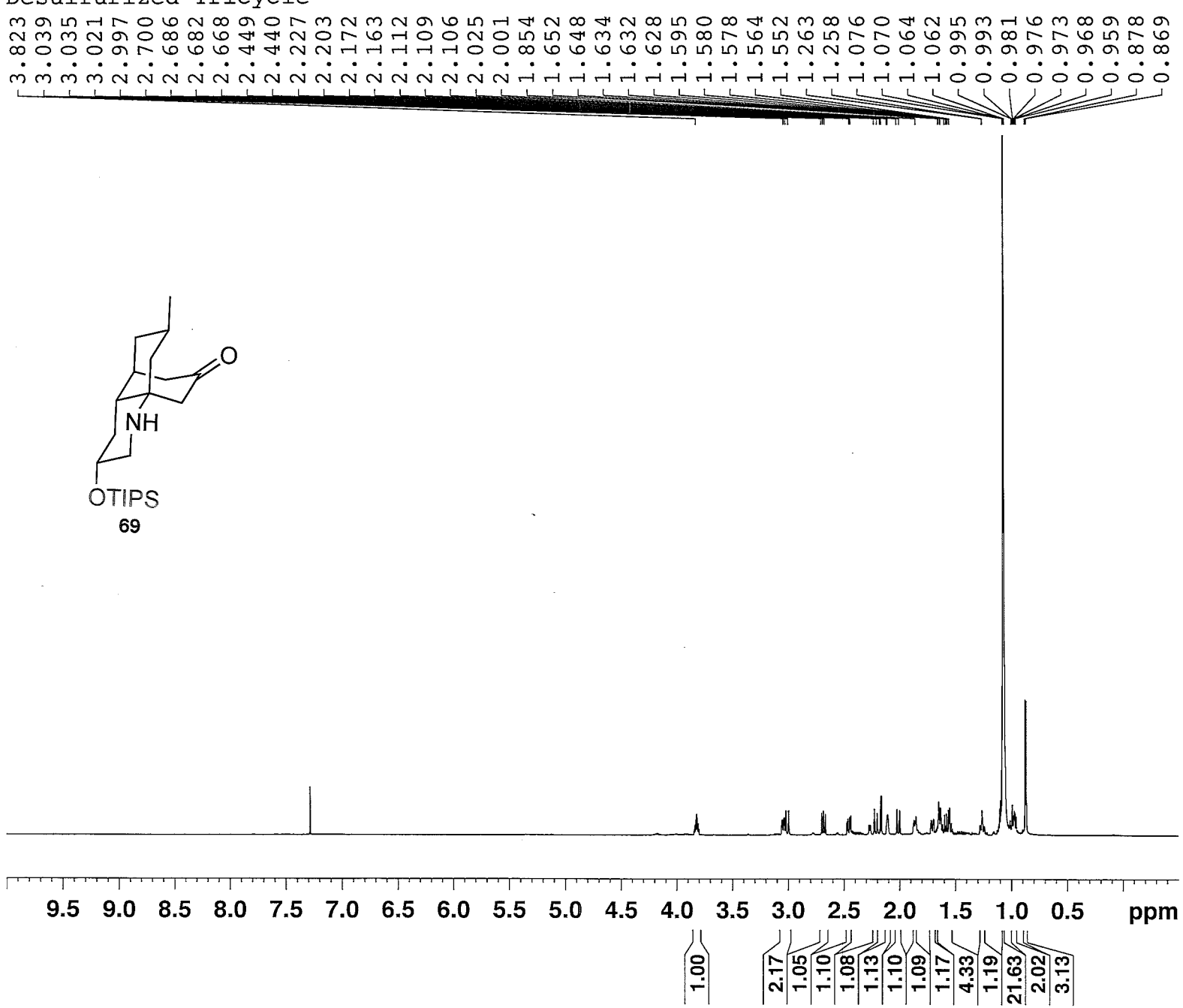
XL-vi-54-10(s)-TIPS-methyl-ketone



BRUKER

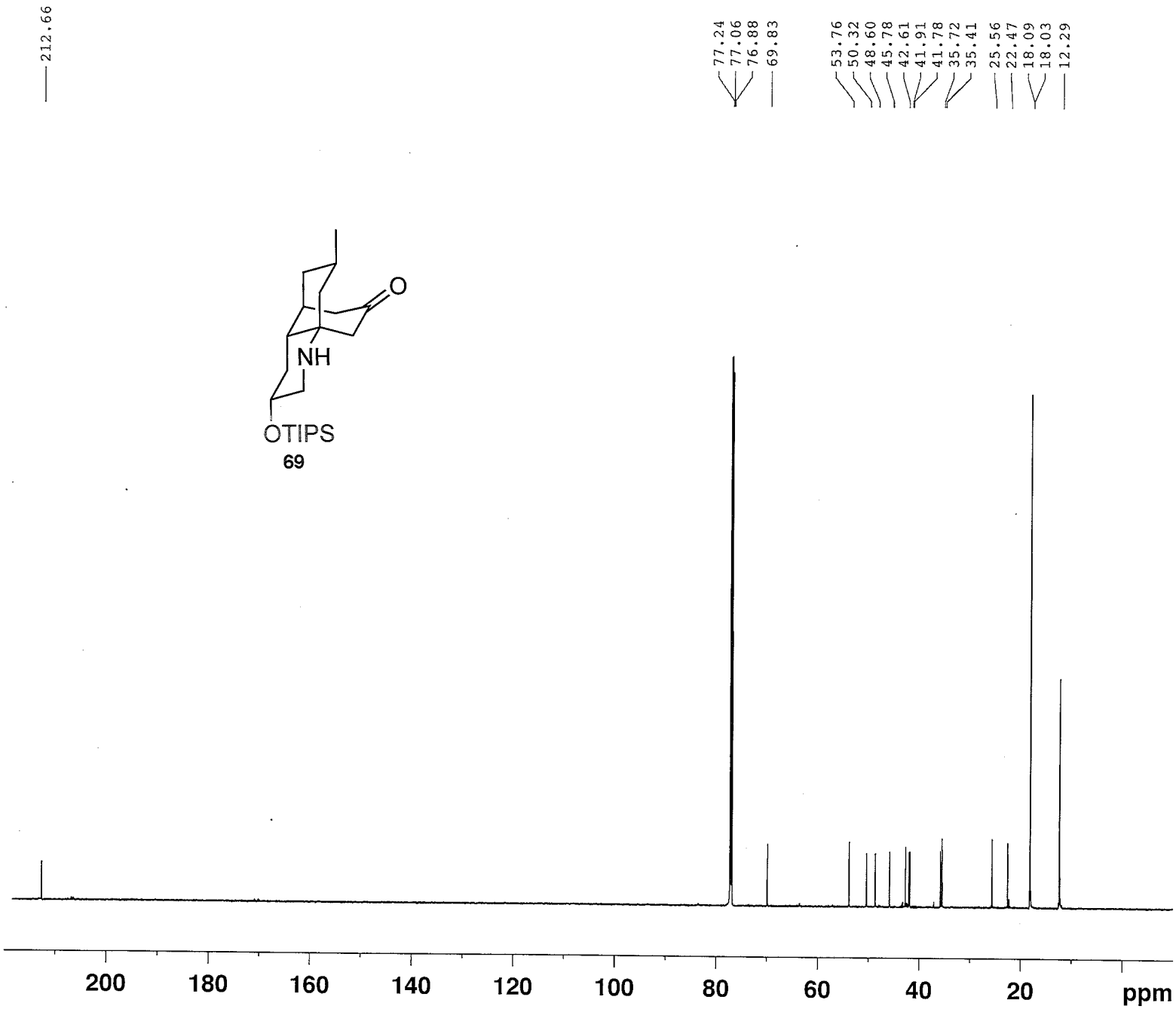
```
NAME        XL-vi-54-10(s)-TIPS-methyl-ketone
EXPNO                                       6
PROCNO                                      1
Date_                            20150805
Time                                    21.32
INSTRUM                                 spect
PROBHD                   5 mm PABBO BB-
PULPROG                                zgpg30
TD                                      65536
SOLVENT                                 CDCl3
NS                                       8192
DS                                          4
SWH                             41666.668 Hz
FIDRES                           0.635783 Hz
AQ                              0.7864820 sec
RG                                        203
DW                                   12.000 usec
DE                                    21.27 usec
TE                                    299.8 K
D1                             2.00000000 sec
D11                            0.03000000 sec
TD0                                         1

======== CHANNEL f1 ========
NUC1                                      13C
P1                                       9.30 usec
PL1                                      2.00 dB
PL1W                          67.83342743 W
SFO1                         176.0697436 MHz

======== CHANNEL f2 ========
CPDPRG2                            waltz16
NUC2                                       1H
PCPD2                                   80.00 usec
PL2                                     -3.20 dB
PL12                                    11.20 dB
PL13                                    12.30 dB
PL2W                          33.59817505 W
PL12W                          1.21987605 W
PL13W                          0.94692516 W
SFO2                         700.1499406 MHz
SI                                     131072
SF                           176.0521380 MHz
WDW                                        EM
SSB                                         0
LB                                       1.00 Hz
GB                                          0
PC                                       1.40
```

Desulfurized Tricycle



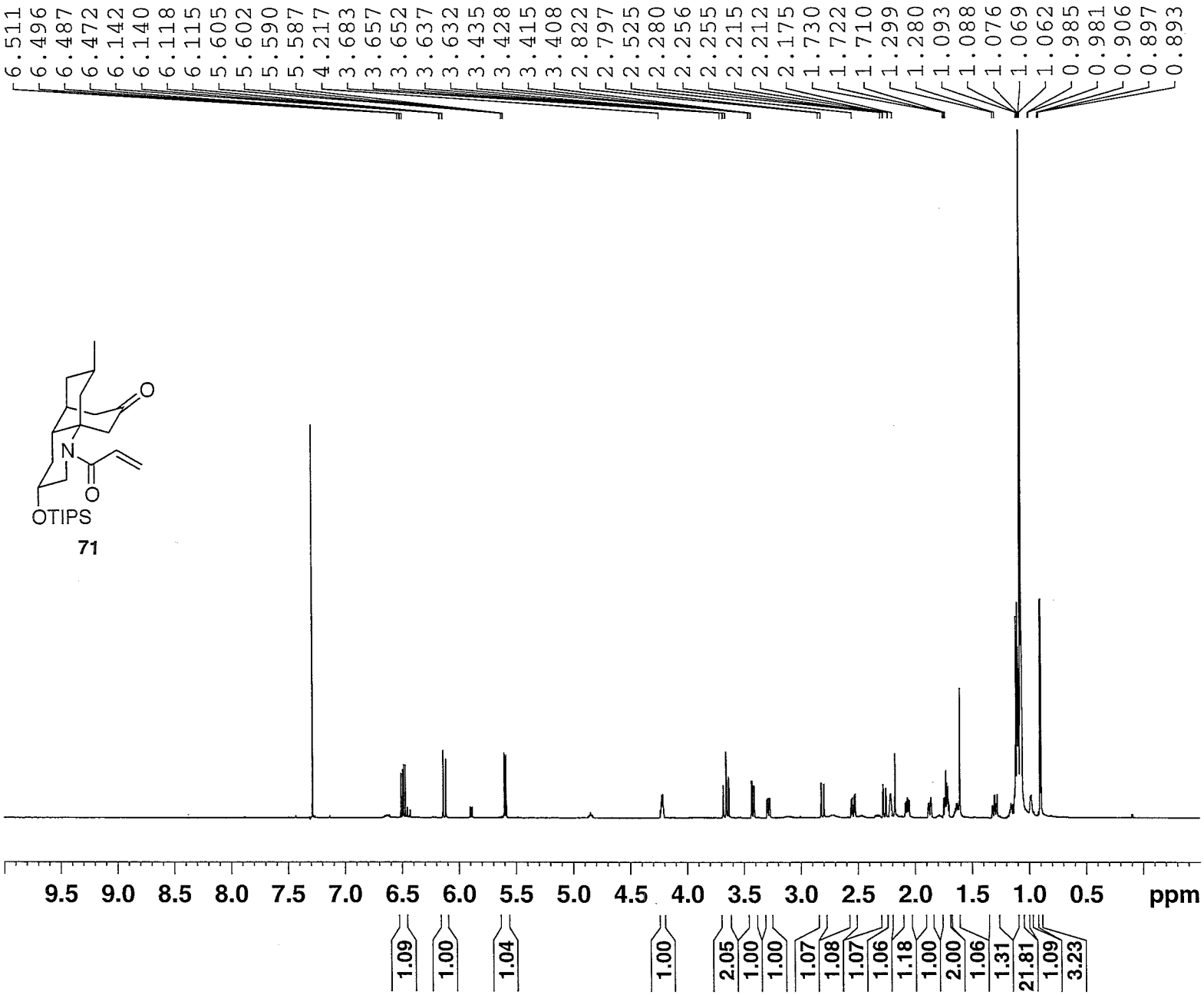
NAME	MS IX-19
EXPNO	1
PROCNO	1
Date_	20120904
Time	10.43
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	7
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 se
RG	18
DW	42.000 us
DE	6.50 us
TE	296.3 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	9.40 us
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MH
SI	131072
SF	700.1471400 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Desulfurized Tricycle carbon



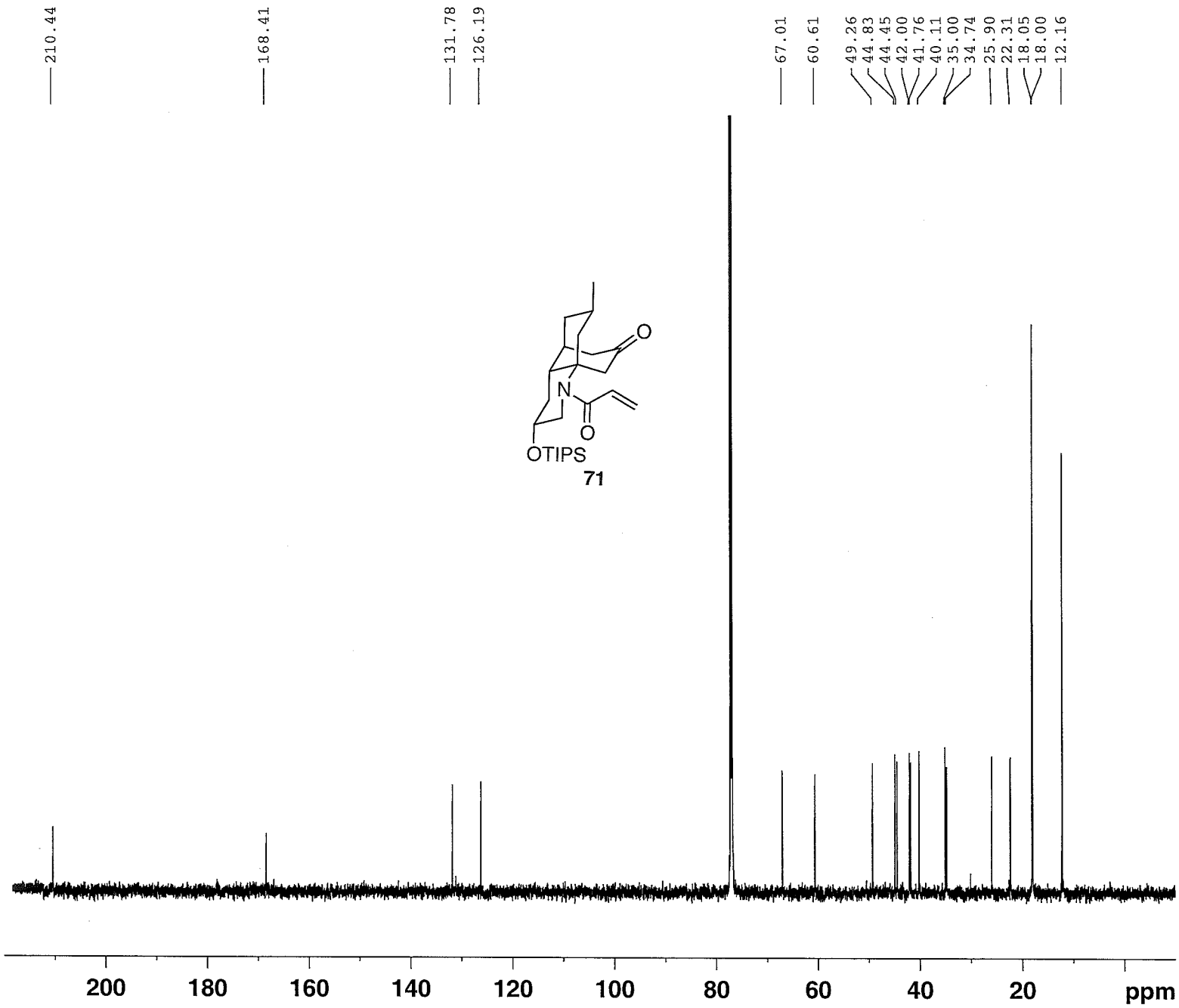
NAME	MS IX-19
EXPNO	1
PROCNO	1
Date_	20120904
Time	10.50
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	53
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 se
RG	203
DW	12.000 us
DE	16.50 us
TE	296.2 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.00 us
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MH
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 us
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MH
SI	32768
SF	176.0521380 MH
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Acryloyl chloride addition




```
NAME        MS IX-78 Acylatio
EXPNO                    1
PROCNO                   1
Date_             20130108
Time                 12.26
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   95236
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              11904.762 Hz
FIDRES            0.125003 Hz
AQ               3.9999621 se
RG                     203
DW                  42.000 us
DE                    6.50 us
TE                   298.5 K
D1              2.00000000 se
TD0                      1

======== CHANNEL f1 ======
NUC1                    1H
P1                   13.75 us
PL1                  -3.00 dB
PL1W           32.08600616 W
SFO1           700.1516910 MH
SI                  131072
SF             700.1471400 MH
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

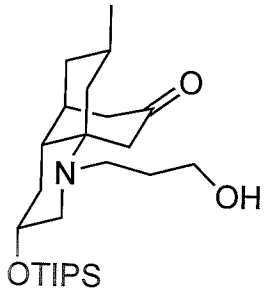
Acryloyl chloride addition carbon



NAME	MS IX-78 Acylation	
EXPNO	3	
PROCNO	1	
Date_	20130108	
Time	12.47	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	41666.668	Hz
FIDRES	0.635783	Hz
AQ	0.7864820	sec
RG	203	
DW	12.000	use
DE	16.50	use
TE	299.5	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 =======		
NUC1	13C	
P1	9.30	use
PL1	2.00	dB
PL1W	67.83342743	W
SFO1	176.0697436	MHz
======== CHANNEL f2 =======		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	use
PL2	-3.00	dB
PL12	12.30	dB
PL13	12.30	dB
PL2W	32.08600616	W
PL12W	0.94692516	W
PL13W	0.94692516	W
SFO2	700.1499406	MHz
SI	32768	
SF	176.0521380	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.40	

Alcohol check

3.853 3.846 3.840 3.800 3.797 3.790 3.786 3.075 3.069 2.709 2.685 2.534 2.518 2.509 2.252 2.228 2.216 2.197 2.192 2.186 2.169 2.155 2.140 2.130 1.845 1.721 1.702 1.607 1.605 1.594 1.585 1.496 1.299 1.293 1.275 1.120 1.115 1.110 1.101 1.097 1.089 1.081 1.077 1.072 0.931 0.922



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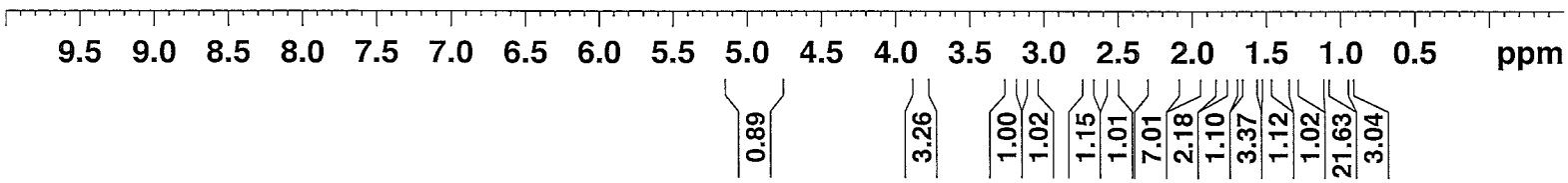
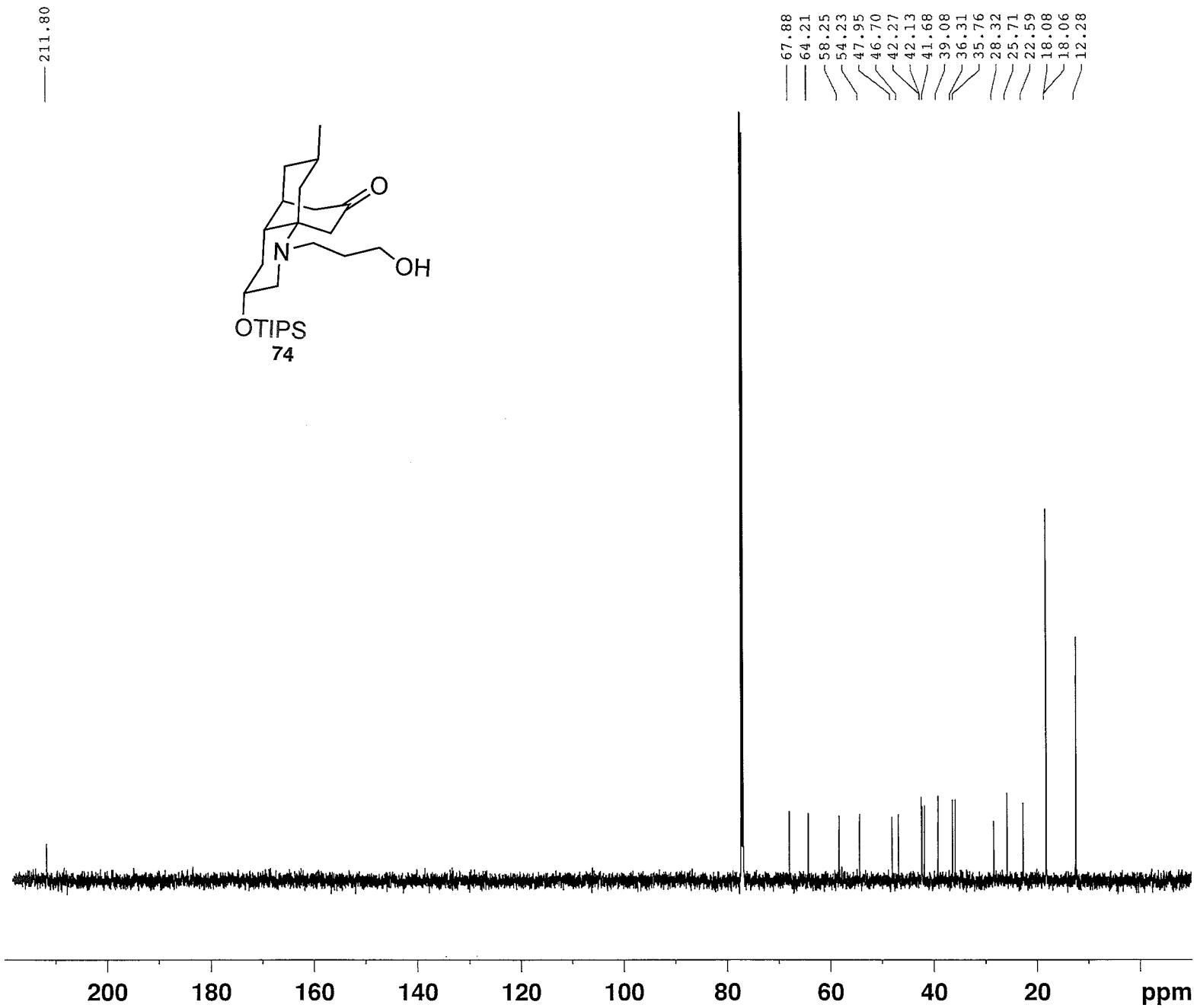


NAME	MS IX-24 Alkylati
EXPNO	1
PROCNO	1
Date_	20120912
Time	2.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	6
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 se
RG	114
DW	42.000 us
DE	6.50 us
TE	298.4 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	13.75 us
PL1	-3.00 dB
PL1W	32.08600616 W
SFO1	700.1516910 MH
SI	131072
SF	700.1471400 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Alkylation carbon



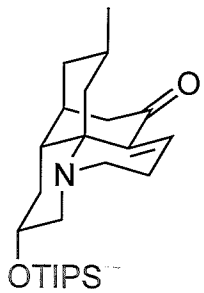

```
NAME        MS IX-24 Alkylati
EXPNO                       2
PROCNO                      1
Date_            20120912
Time                     2.32
INSTRUM                 spect
PROBHD     5 mm PABBO BB-
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        40
DS                         4
SWH               41666.668 Hz
FIDRES             0.635783 Hz
AQ                0.7864820 se
RG                       203
DW                    12.000 us
DE                     16.50 us
TE                     299.0 K
D1               2.00000000 se
D11              0.03000000 se
TD0                        1

======== CHANNEL f1 ======
NUC1                     13C
P1                      9.30 us
PL1                     2.00 dB
PL1W            67.83342743 W
SFO1           176.0697436 MH

======== CHANNEL f2 ======
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 us
PL2                    -3.00 dB
PL12                   12.30 dB
PL13                   12.30 dB
PL2W            32.08600616 W
PL12W            0.94692516 W
PL13W            0.94692516 W
SFO2           700.1499406 MH
SI                     32768
SF             176.0521380 MH
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.40
```

Tetracycle

7.288 7.022 7.016 7.011 3.939 2.717 2.696 2.674 2.664 2.647 2.637 2.452 2.437 2.422 2.354 2.327 2.162 2.157 2.143 2.139 1.759 1.740 1.640 1.621 1.394 1.381 1.379 1.376 1.375 1.369 1.361 1.357 1.118 1.116 1.108 1.106 1.103 1.100 1.095 1.089 1.082 1.076 1.071 1.066 0.925 0.916



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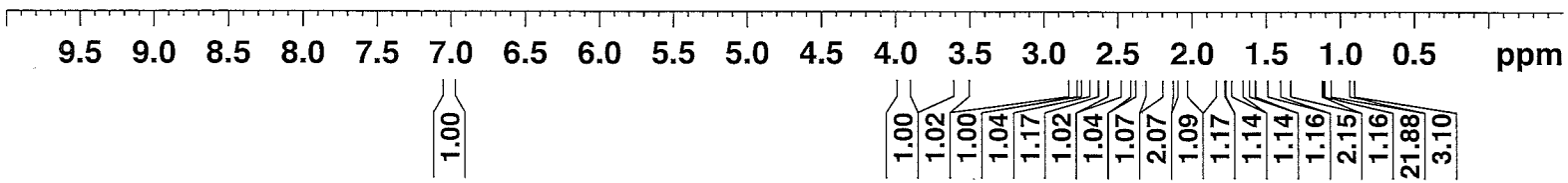
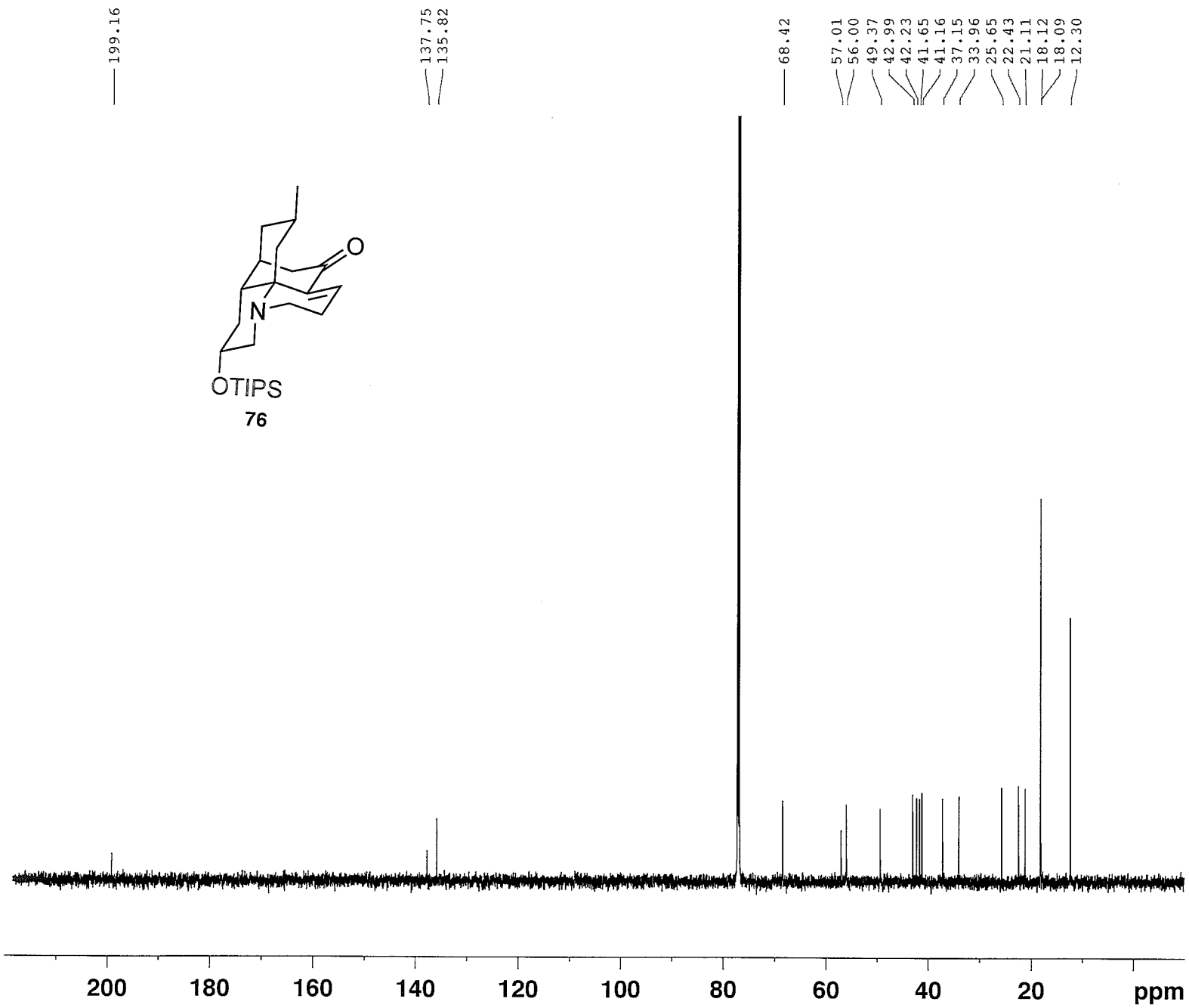


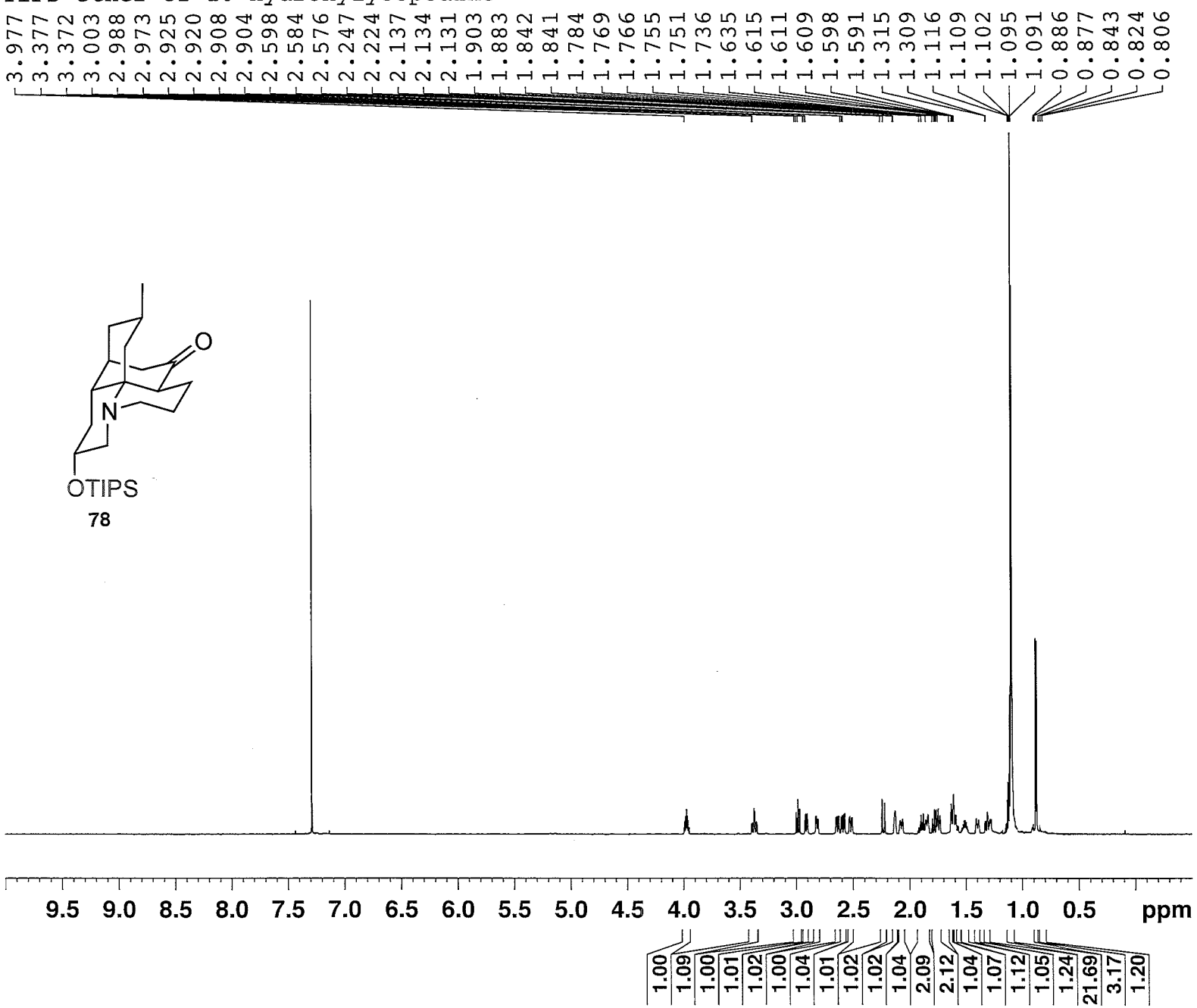
NAME	MS IX-28 Tetracyc
EXPNO	2
PROCNO	1
Date_	20120913
Time	16.48
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	1
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 se
RG	203
DW	42.000 us
DE	6.50 us
TE	298.2 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	13.75 us
PL1	-3.00 dB
PL1W	32.08600616 W
SFO1	700.1516910 MH
SI	131072
SF	700.1471400 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Tetracycle carbon



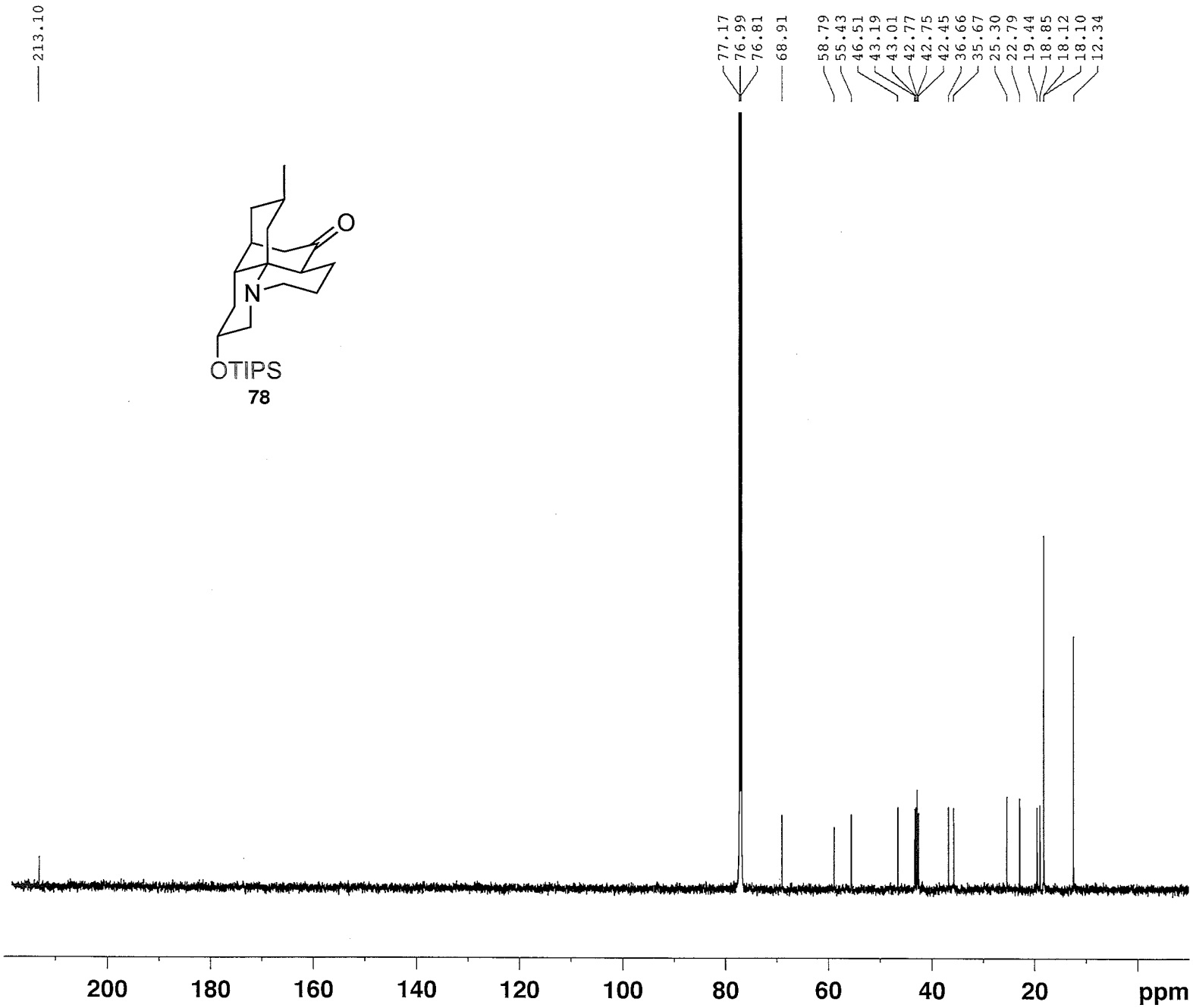
NAME	MS IX-28 Tetracyc
EXPNO	3
PROCNO	1
Date_	20120913
Time	16.55
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 se
RG	203
DW	12.000 us
DE	16.50 us
TE	299.4 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.30 us
PL1	2.00 dB
PL1W	67.83342743 W
SFO1	176.0697436 MH
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 us
PL2	-3.00 dB
PL12	12.30 dB
PL13	12.30 dB
PL2W	32.08600616 W
PL12W	0.94692516 W
PL13W	0.94692516 W
SFO2	700.1499406 MH
SI	32768
SF	176.0521380 MH
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

TIPS ether of 10-hydroxylycopodine



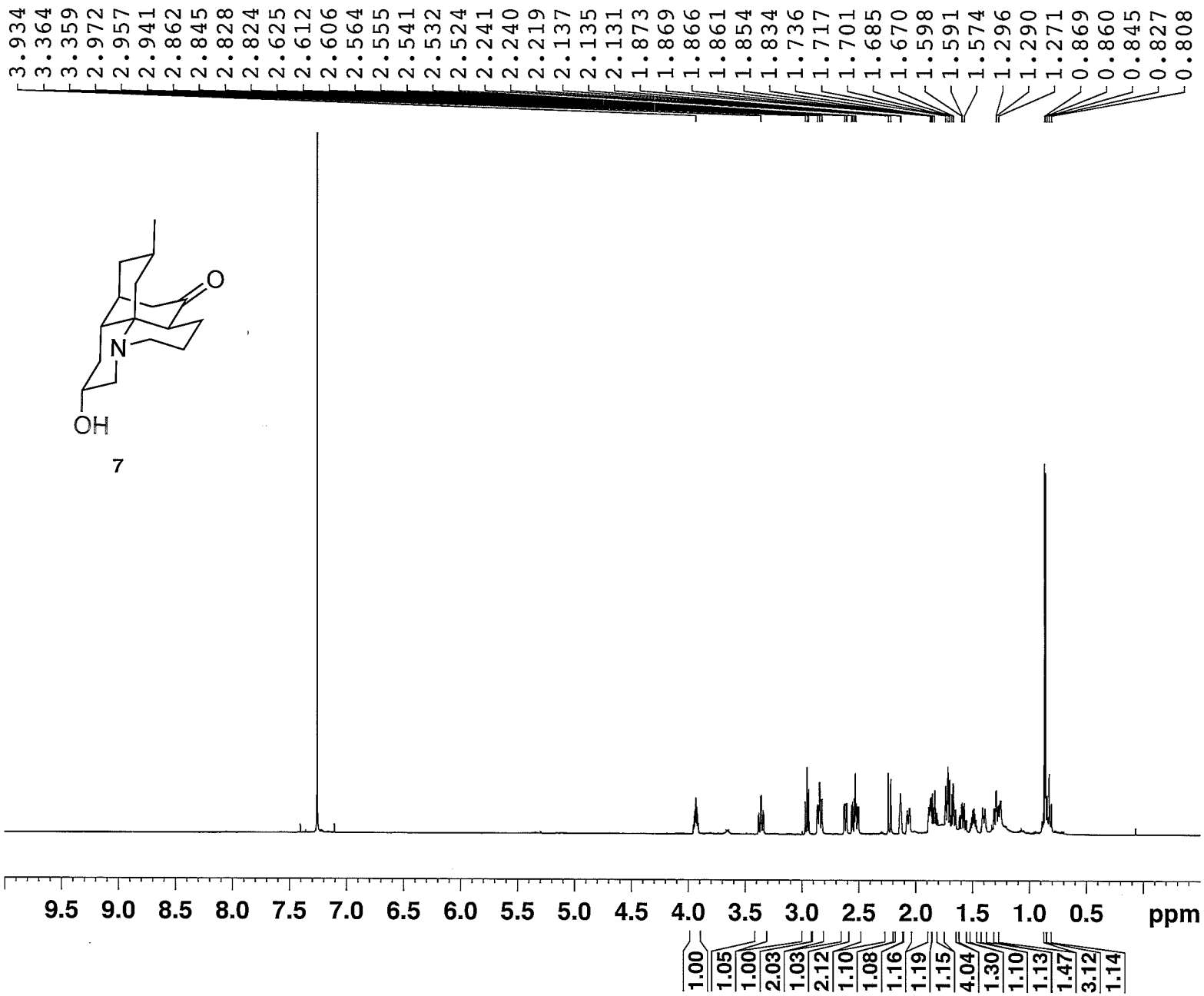
NAME	MS VIII-55 hydrogenati
EXPNO	1
PROCNO	1
Date_	20120629
Time	10.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	10
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	203
DW	42.000 usec
DE	6.50 usec
TE	289.9 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.75 usec
PL1	-3.00 dB
PL1W	32.08600616 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

TIPS ether of 10-hydroxylycopodine carbon



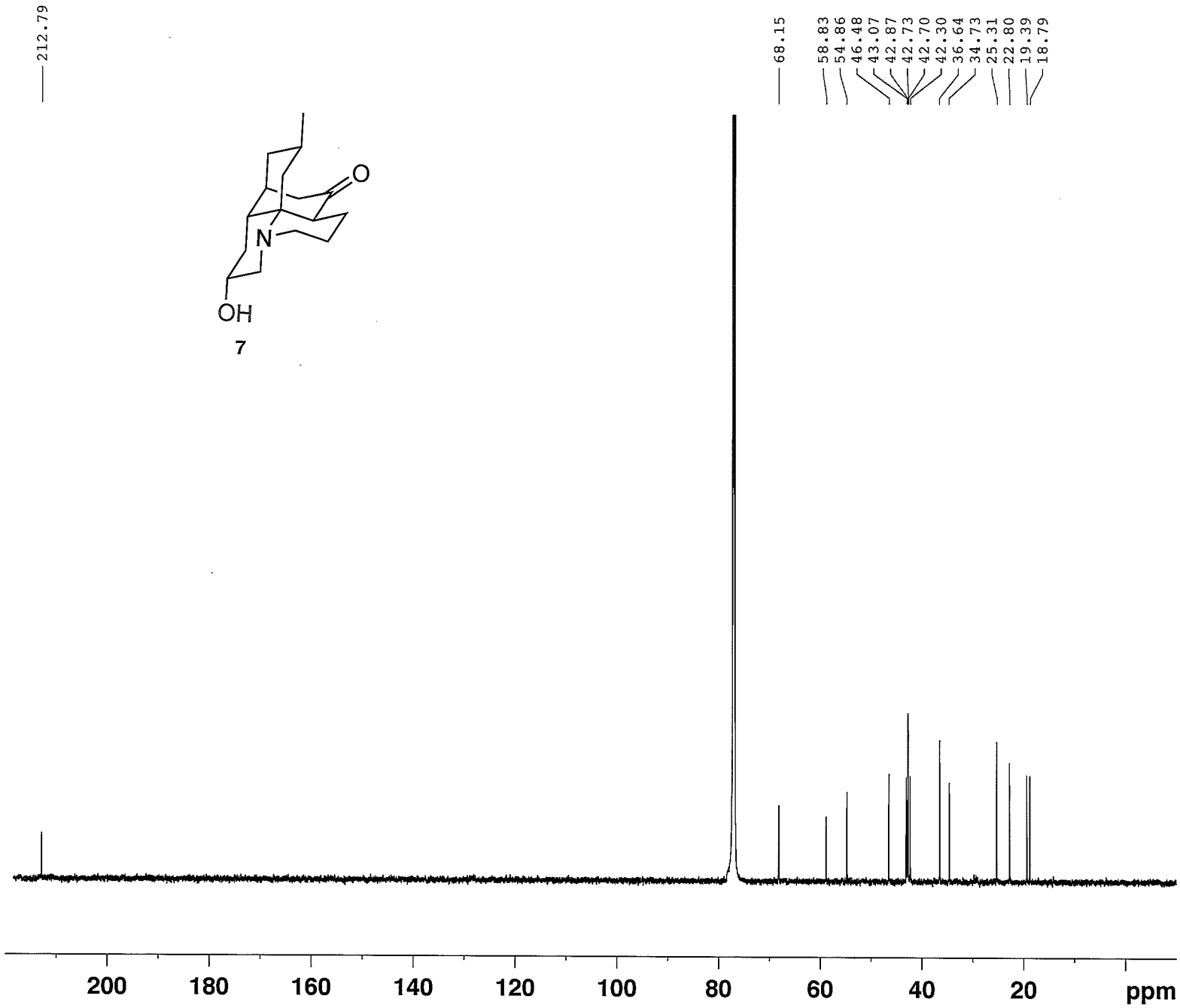
NAME	MS VIII-55 hydrogenati
EXPNO	3
PROCNO	1
Date_	20120629
Time	10.53
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2000
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	292.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	2.00 dB
PL1W	67.83342743 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-3.00 dB
PL12	12.30 dB
PL13	12.30 dB
PL2W	32.08600616 W
PL12W	0.94692516 W
PL13W	0.94692516 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

10-hydroxylycopodine



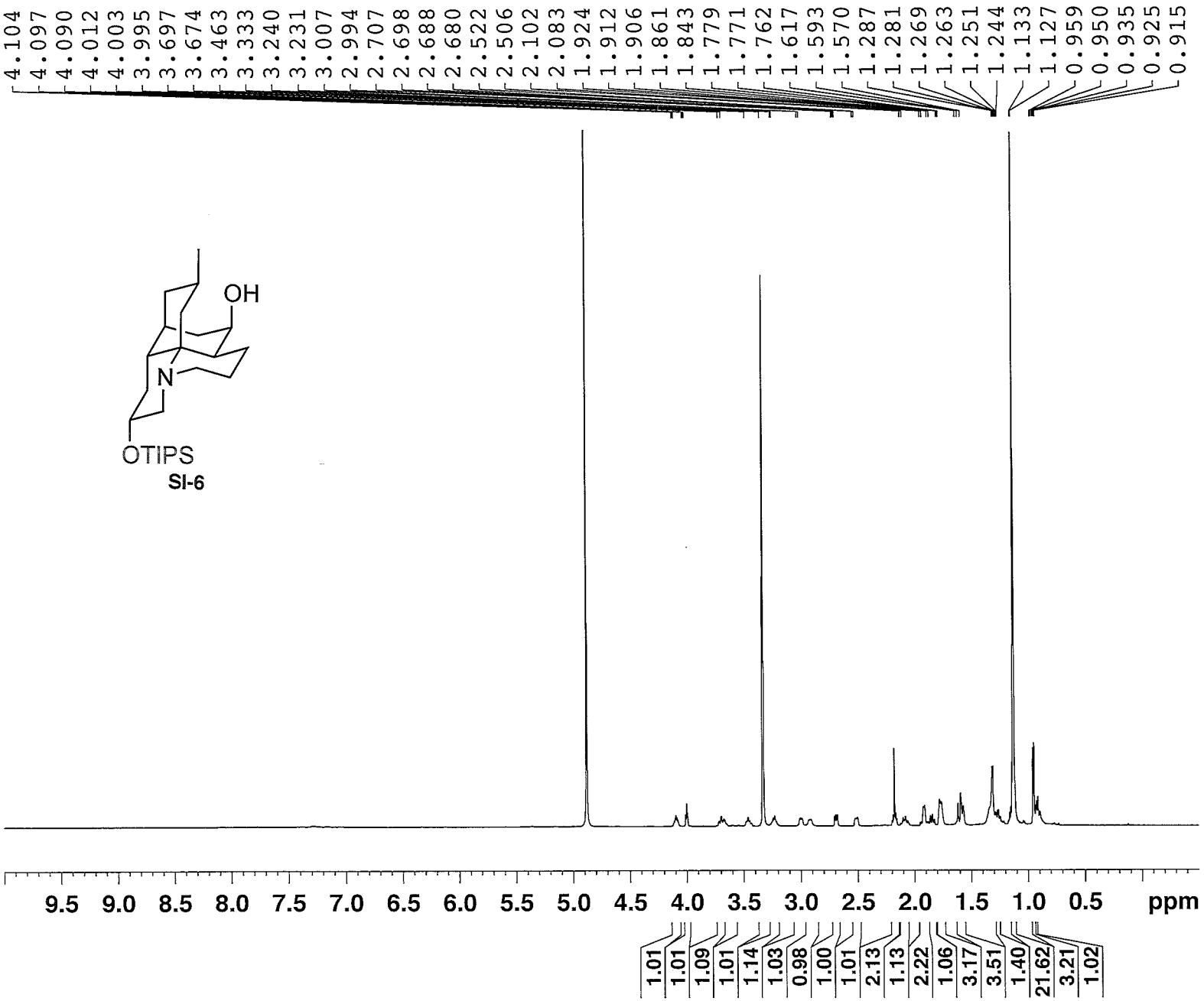
NAME	MS VIII-23 10-hydroxylycopodi	
EXPNO	1	
PROCNO	1	
Date_	20120520	
Time	9.41	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zg30	
TD	95236	
SOLVENT	CDCl3	
NS	32	
DS	2	
SWH	11904.762	Hz
FIDRES	0.125003	Hz
AQ	3.9999621	sec
RG	36	
DW	42.000	usec
DE	6.50	usec
TE	298.2	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	9.40	usec
PL1	-3.20	dB
PL1W	33.59817505	W
SFO1	700.1516910	MHz
SI	131072	
SF	700.1471597	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

10-hydroxylycopodine carbon




```
NAME        MS VIII-23 10-hydroxylycopodi
EXPNO                               2
PROCNO                              1
Date_                        20120520
Time                             9.48
INSTRUM                         spect
PROBHD     5 mm CPDCH 13C
PULPROG                        zgpg30
TD                              65536
SOLVENT                         CDCl3
NS                               1352
DS                                  4
SWH                        41666.668 Hz
FIDRES                      0.635783 Hz
AQ                         0.7864820 sec
RG                                203
DW                           12.000 usec
DE                            16.50 usec
TE                            298.2 K
D1                       2.00000000 sec
D11                      0.03000000 sec
TD0                                 1

======== CHANNEL f1 ========
NUC1                              13C
P1                               9.00 usec
PL1                              4.50 dB
PL1W                     38.14553833 W
SFO1                    176.0697436 MHz

======== CHANNEL f2 ========
CPDPRG2                      waltz16
NUC2                              1H
PCPD2                           65.00 usec
PL2                             -3.20 dB
PL12                            13.60 dB
PL13                           120.00 dB
PL2W                     33.59817505 W
PL12W                     0.70196527 W
PL13W                     0.00000000 W
SFO2                    700.1499406 MHz
SI                               32768
SF                      176.0521368 MHz
WDW                                EM
SSB                                 0
LB                               3.00 Hz
GB                                  0
PC                               1.40
```

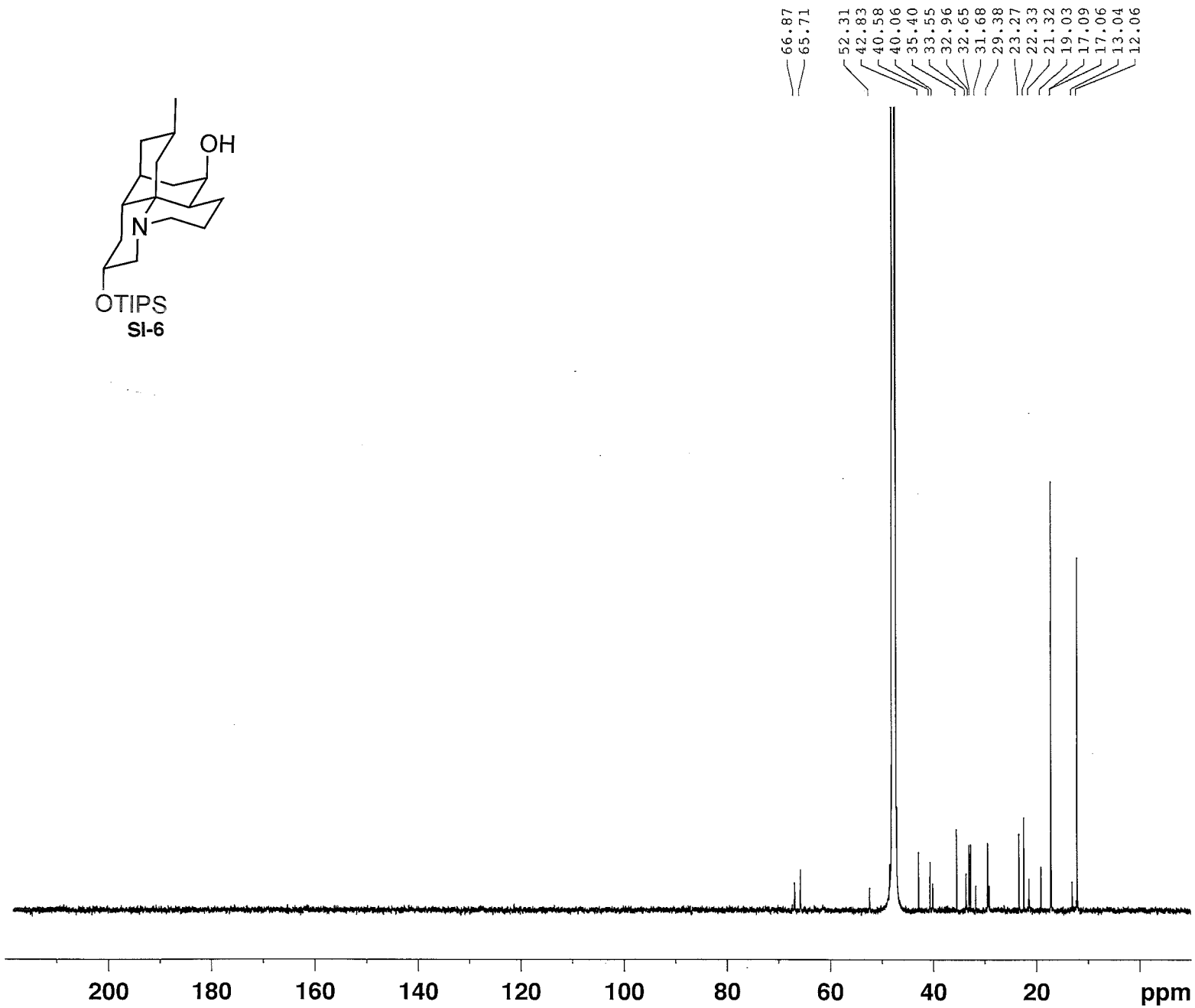
Deacetylpaniculine-C10-TIPS ether



NAME	MS VIII-13 DIBAL reducti	
EXPNO	7	
PROCNO	1	
Date_	20120510	
Time	11.30	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zg30	
TD	95236	
SOLVENT	MeOD	
NS	32	
DS	2	
SWH	11904.762	Hz
FIDRES	0.125003	Hz
AQ	3.9999621	sec
RG	80.6	
DW	42.000	usec
DE	6.50	usec
TE	298.2	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	9.40	usec
PL1	-3.20	dB
PL1W	33.59817505	W
SFO1	700.1516910	MHz
SI	131072	
SF	700.1471400	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

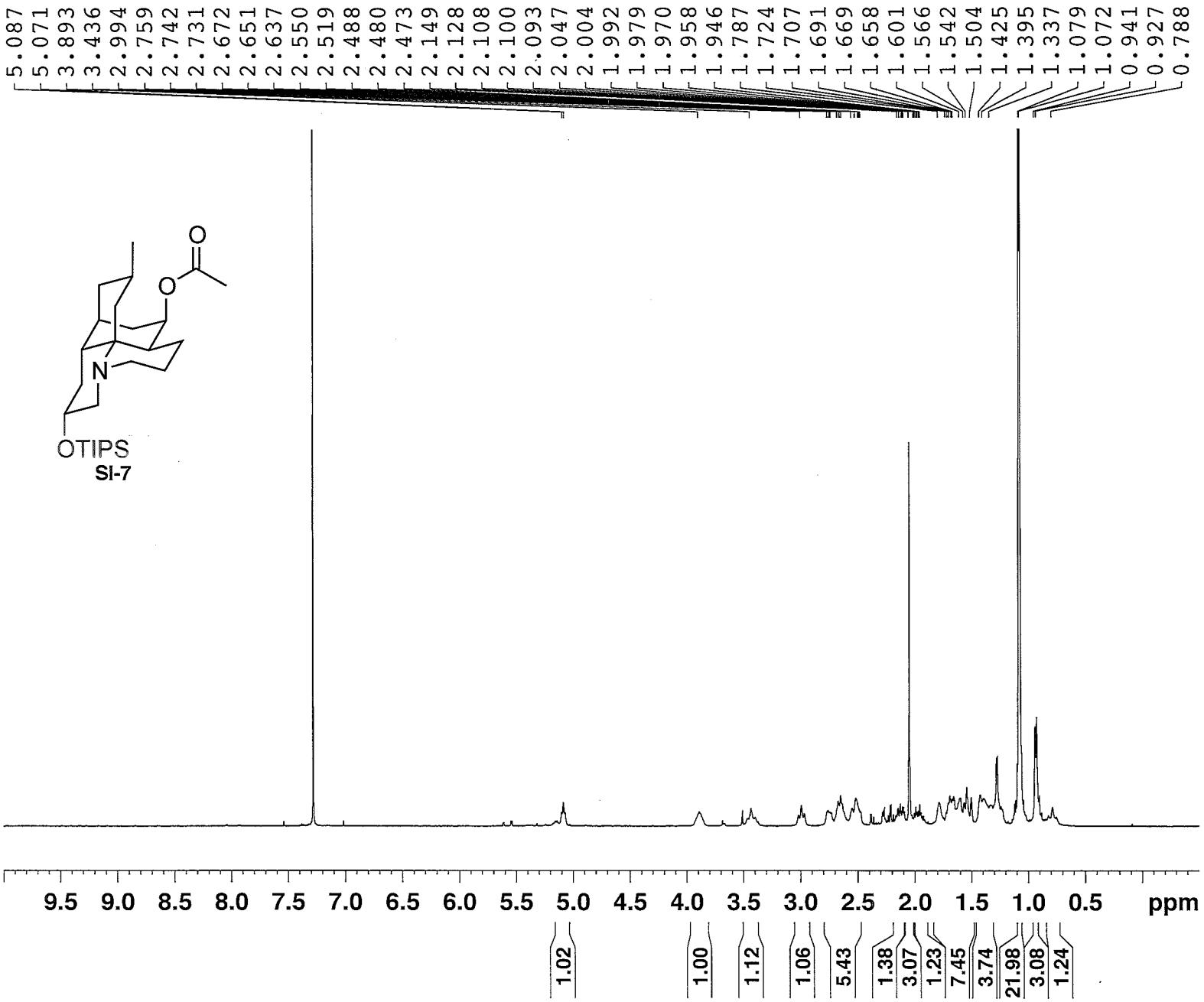
Deacetylpaniculine-C10-TIPS ether carbon

SI-6



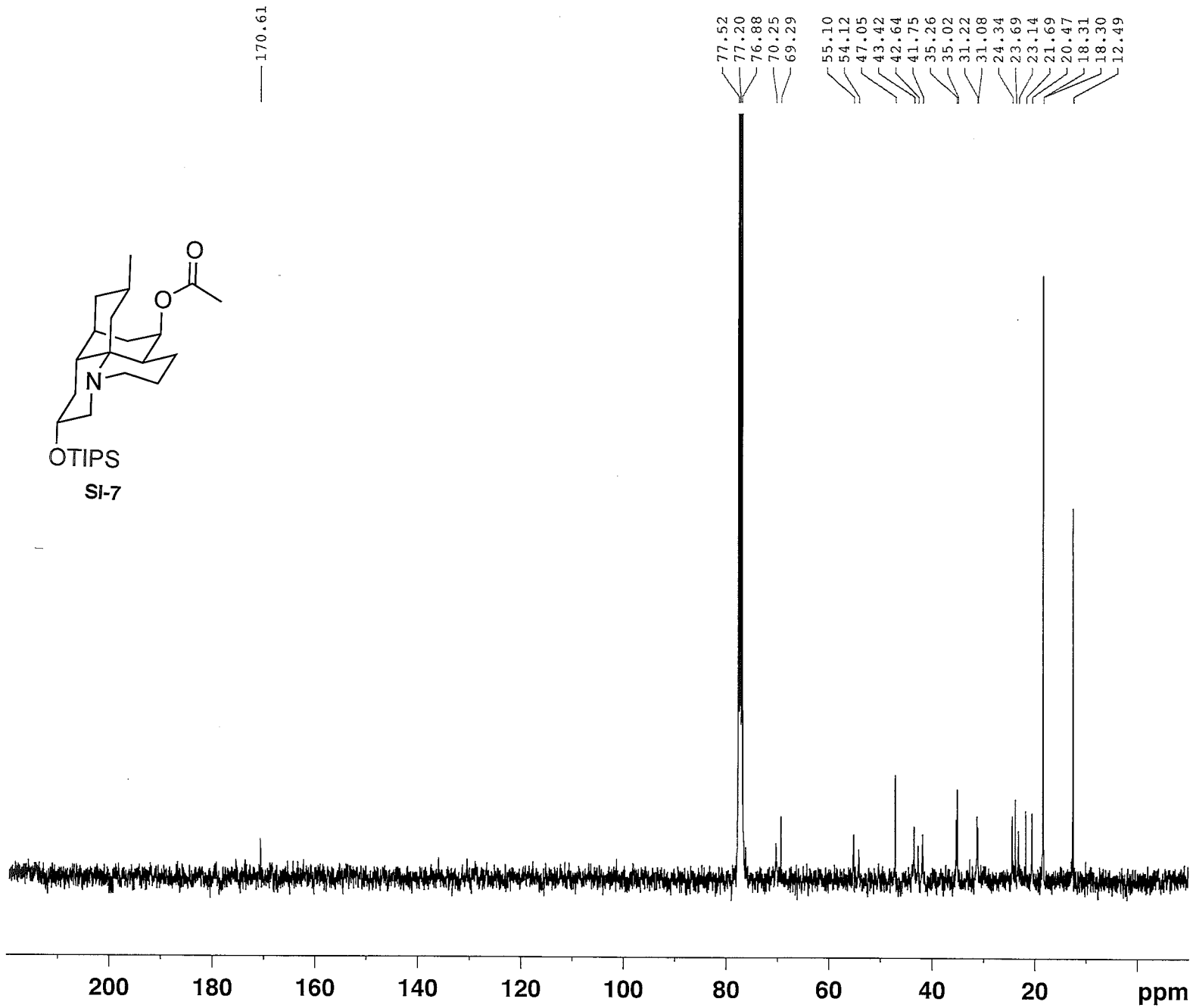

```
NAME        MS VIII-13 DIBAL reducti
EXPNO                              9
PROCNO                             1
Date_                       20120510
Time                           11.50
INSTRUM                        spect
PROBHD      5 mm CPDCH 13C
PULPROG                       zgpg30
TD                             65536
SOLVENT                         MeOD
NS                              2057
DS                                 4
SWH                        41666.668 Hz
FIDRES                      0.635783 Hz
AQ                         0.7864820 sec
RG                               203
DW                            12.000 usec
DE                             16.50 usec
TE                             298.2 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                                1

======== CHANNEL f1 ========
NUC1                             13C
P1                              9.00 usec
PL1                             4.50 dB
PL1W                    38.14553833 W
SFO1                   176.0697436 MHz

======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                             1H
PCPD2                          65.00 usec
PL2                            -3.20 dB
PL12                           13.60 dB
PL13                          120.00 dB
PL2W                    33.59817505 W
PL12W                    0.70196527 W
PL13W                    0.00000000 W
SFO2                   700.1499406 MHz
SI                               32768
SF                     176.0521380 MHz
WDW                                EM
SSB                                 0
LB                              3.00 Hz
GB                                  0
PC                               1.40
```

Acetate



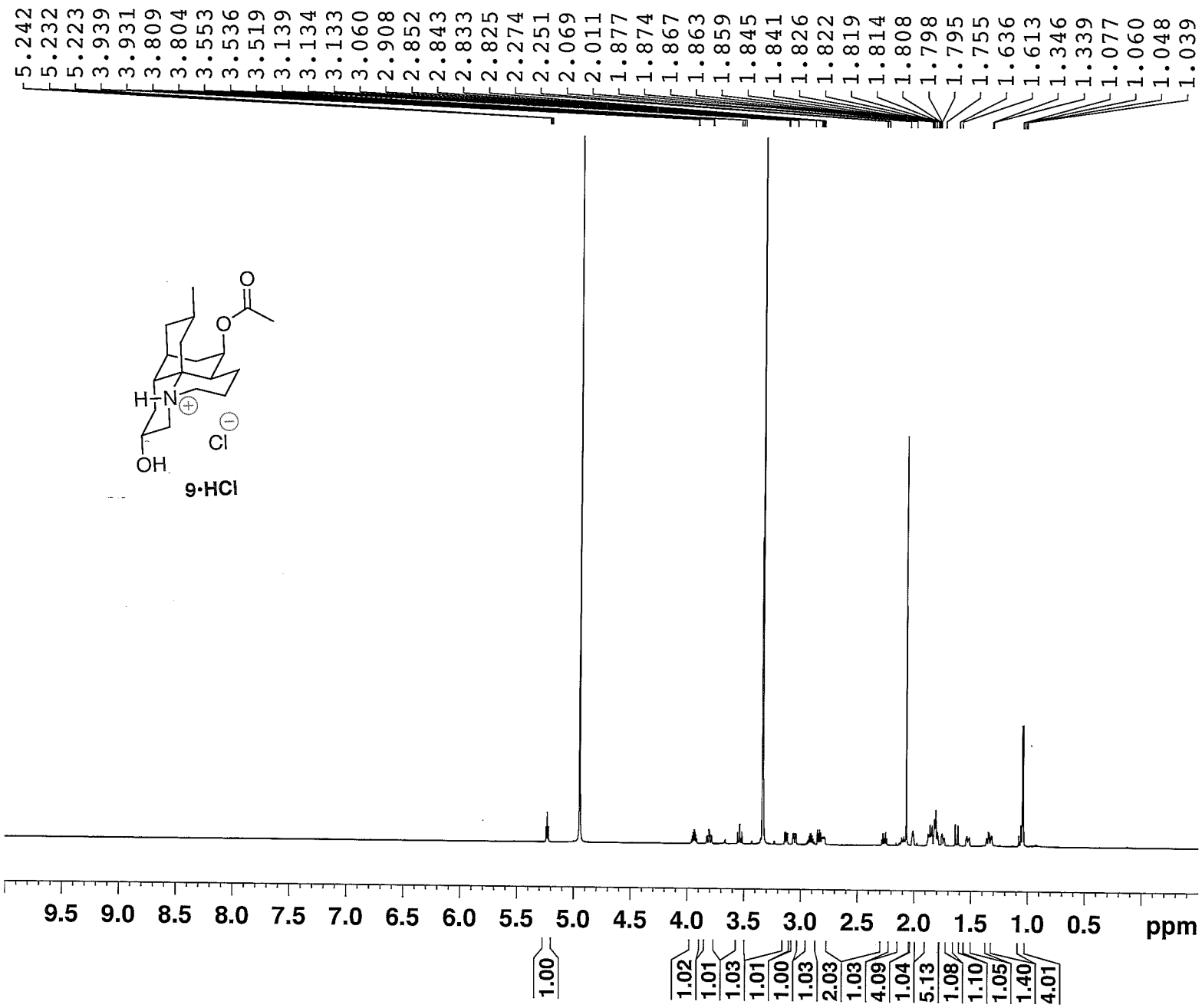
NAME	MS IX-34 Acetate
EXPNO	1
PROCNO	1
Date_	20120920
Time	3.37
INSTRUM	robinson
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	7183.908 Hz
FIDRES	0.219235 Hz
AQ	2.2807028 se
RG	181
DW	69.600 us
DE	6.50 us
TE	298.2 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	11.20 us
PL1	0.00 dB
SFO1	400.1428010 MH
SI	32768
SF	400.1400000 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Acetate carbon



NAME	MS IX-34 Acetate
EXPNO	3
PROCNO	1
Date_	20120920
Time	3.55
INSTRUM	robinson
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	8497
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 se
RG	32768
DW	20.850 us
DE	6.50 us
TE	298.9 K
D1	1.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.00 us
PL1	-2.00 dB
SFO1	100.6253446 MH
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 us
PL2	0.00 dB
PL12	16.16 dB
PL13	17.00 dB
SFO2	400.1416006 MH
SI	32768
SF	100.6152633 MH
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

paniculine HCl salt

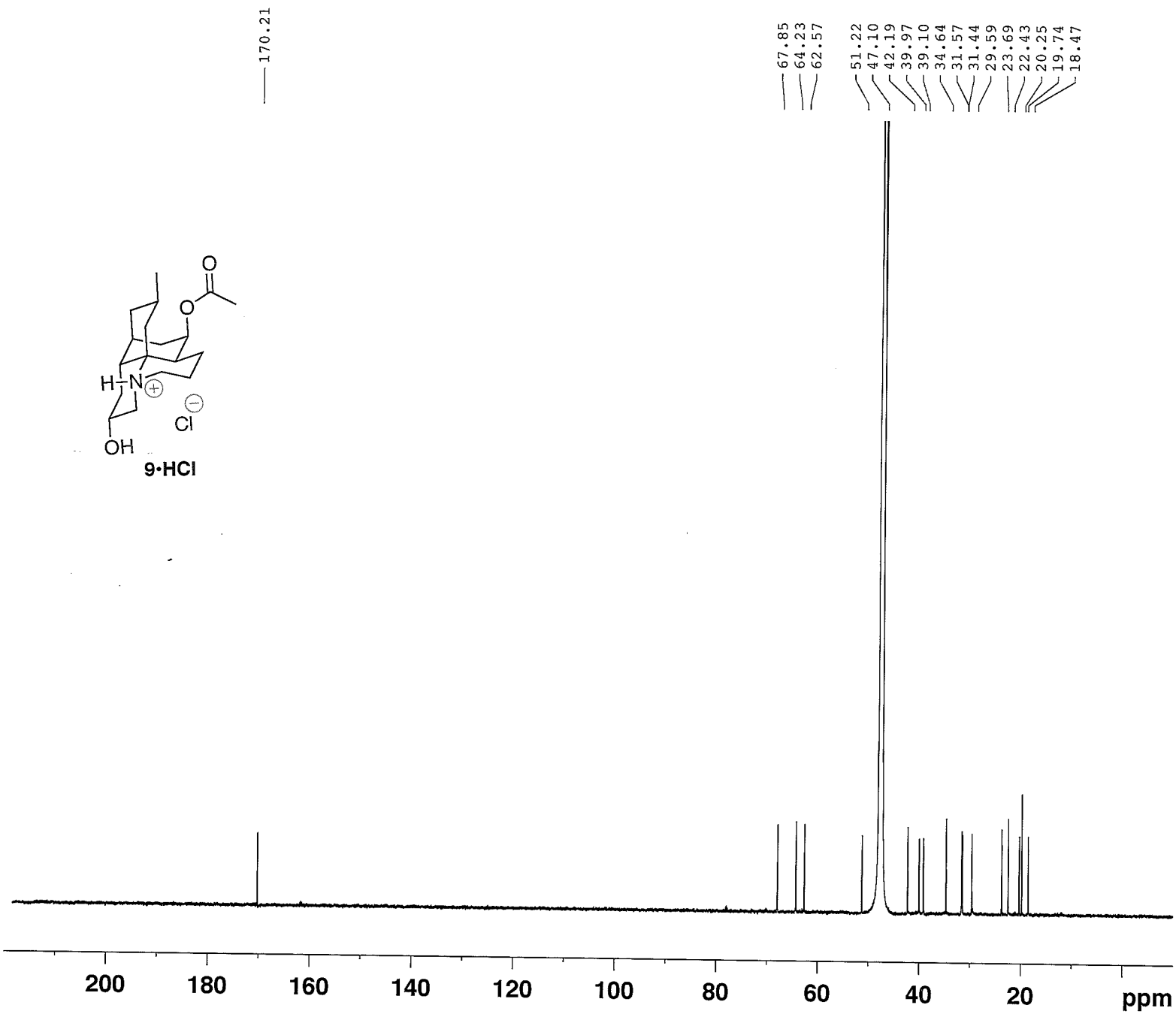


9·HCl

```
NAME      MS IX-45 paniculine HCl sa
EXPNO                  2
PROCNO                 1
Date_           20120930
Time                0.33
INSTRUM            spect
PROBHD   5 mm CPDCH 13C
PULPROG             zg30
TD                 95236
SOLVENT             MeOD
NS                    32
DS                     2
SWH            11904.762 Hz
FIDRES          0.125003 Hz
AQ             3.9999621 sec
RG                    32
DW                42.000 usec
DE                  6.50 usec
TE                 298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.40 usec
PL1                -3.20 dB
PL1W        33.59817505 W
SFO1        700.1516910 MHz
SI               131072
SF          700.1471400 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

S 80

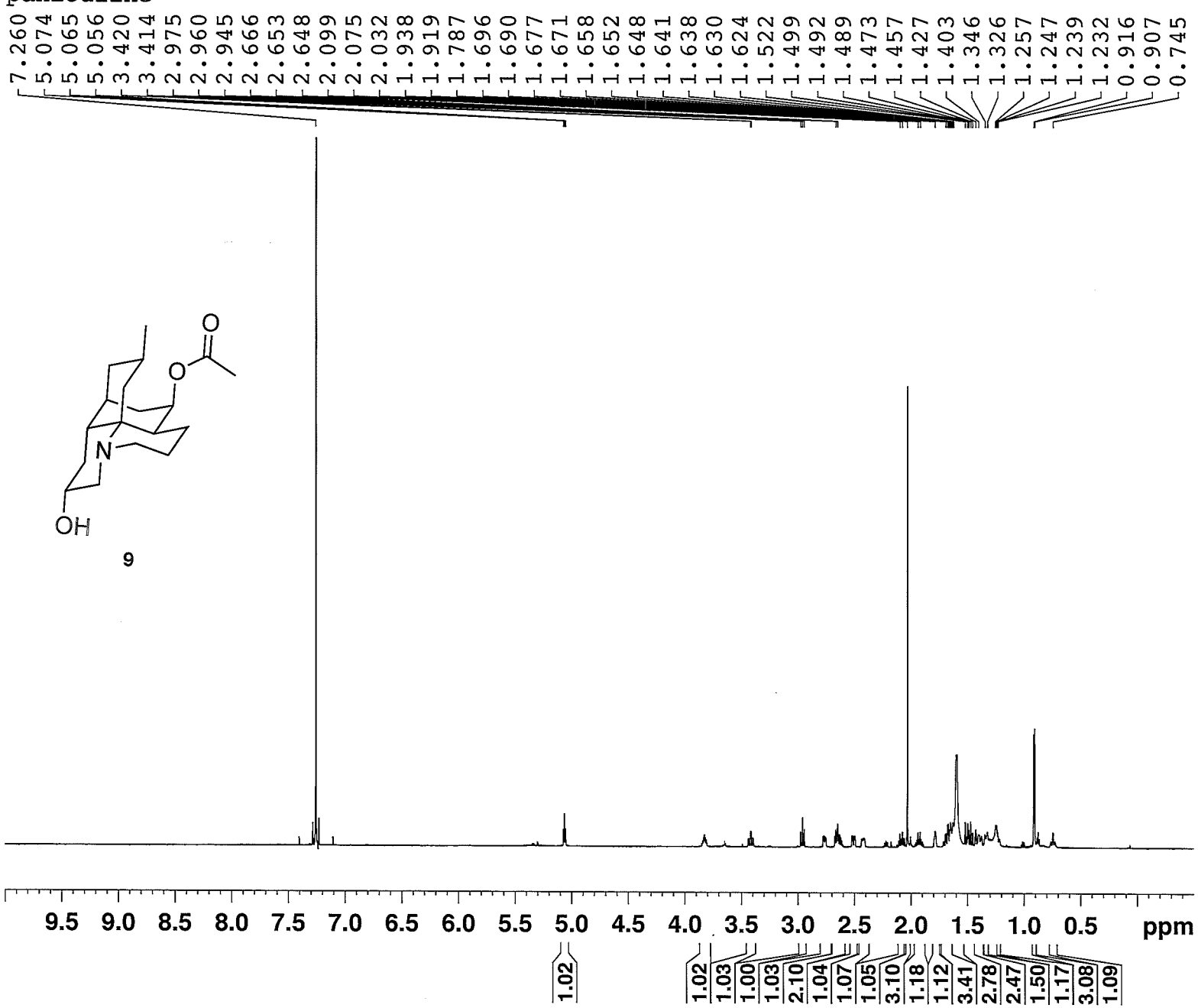
paniculine HCl salt carbon



9·HCl

```
NAME        MS IX-45 paniculine HCl sa
EXPNO                    4
PROCNO                   1
Date_             20120930
Time                  0.52
INSTRUM              spect
PROBHD    5 mm CPDCH 13C
PULPROG            zgpg30
TD                  65536
SOLVENT              MeOD
NS                  12267
DS                       4
SWH            41666.668 Hz
FIDRES          0.635783 Hz
AQ             0.7864820 sec
RG                     203
DW                12.000 usec
DE                 16.50 usec
TE                 298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                  4.50 dB
PL1W        38.14553833 W
SFO1       176.0697436 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2               65.00 usec
PL2                 -3.20 dB
PL12                13.60 dB
PL13               120.00 dB
PL2W        33.59817505 W
PL12W        0.70196527 W
PL13W        0.00000000 W
SFO2       700.1499406 MHz
SI                 32768
SF         176.0521380 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40
```

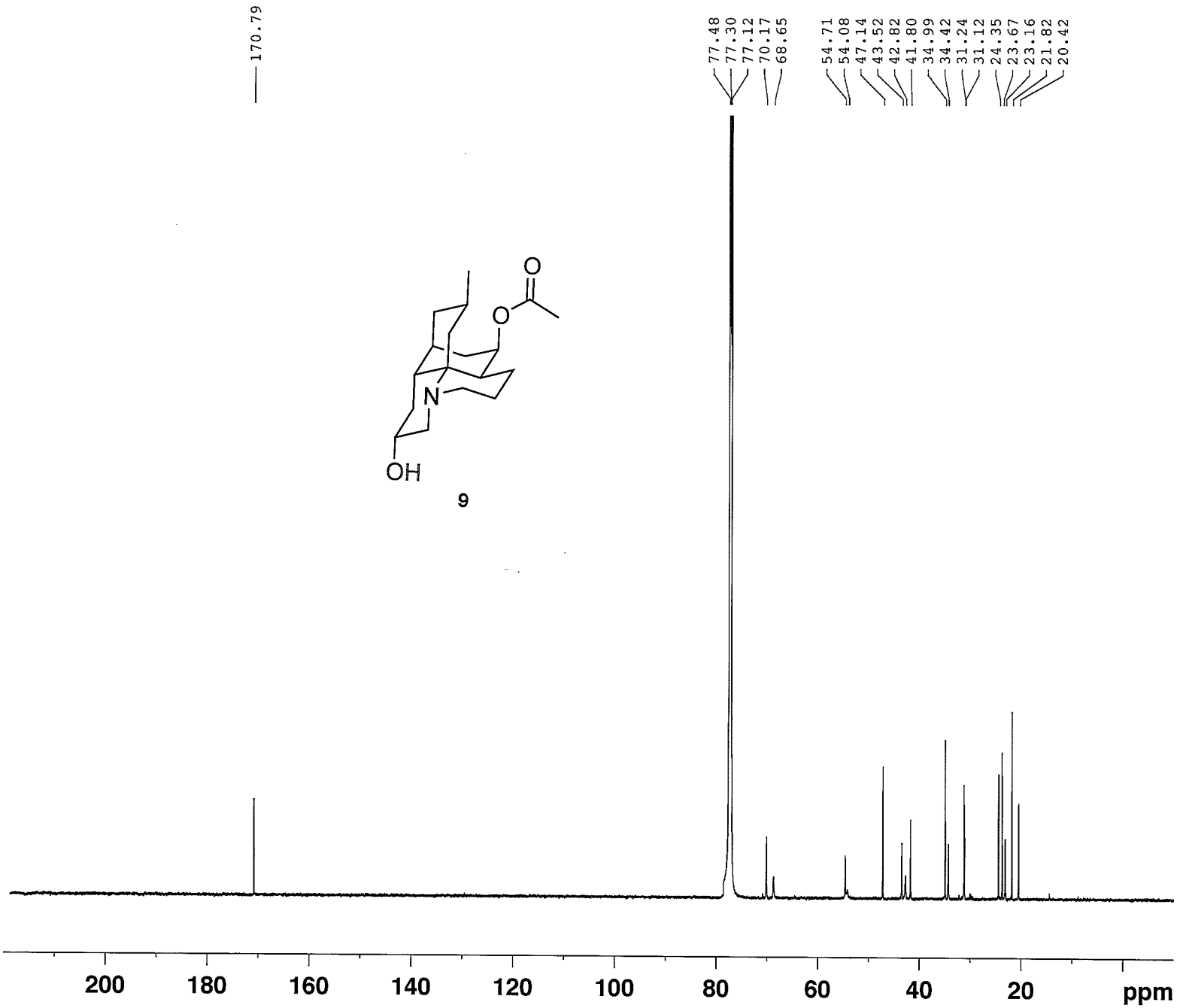
paniculine



S 82

NAME	MS IX-47 paniculi
EXPNO	3
PROCNO	1
Date_	20121002
Time	19.36
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	15
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 se
RG	22.6
DW	42.000 us
DE	6.50 us
TE	298.3 K
D1	2.00000000 se
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	9.40 us
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MH
SI	131072
SF	700.1471600 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

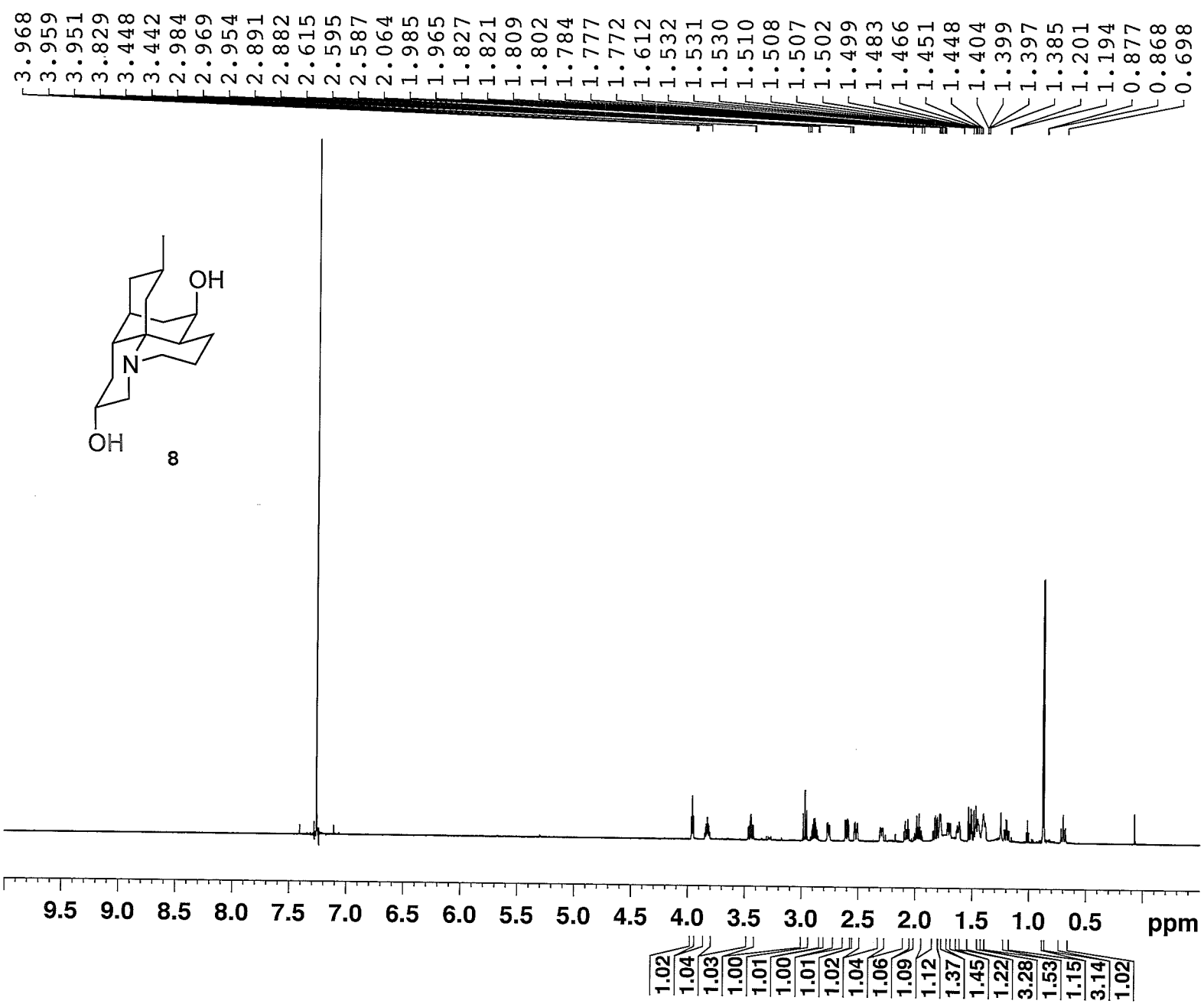
paniculine carbon



S 83

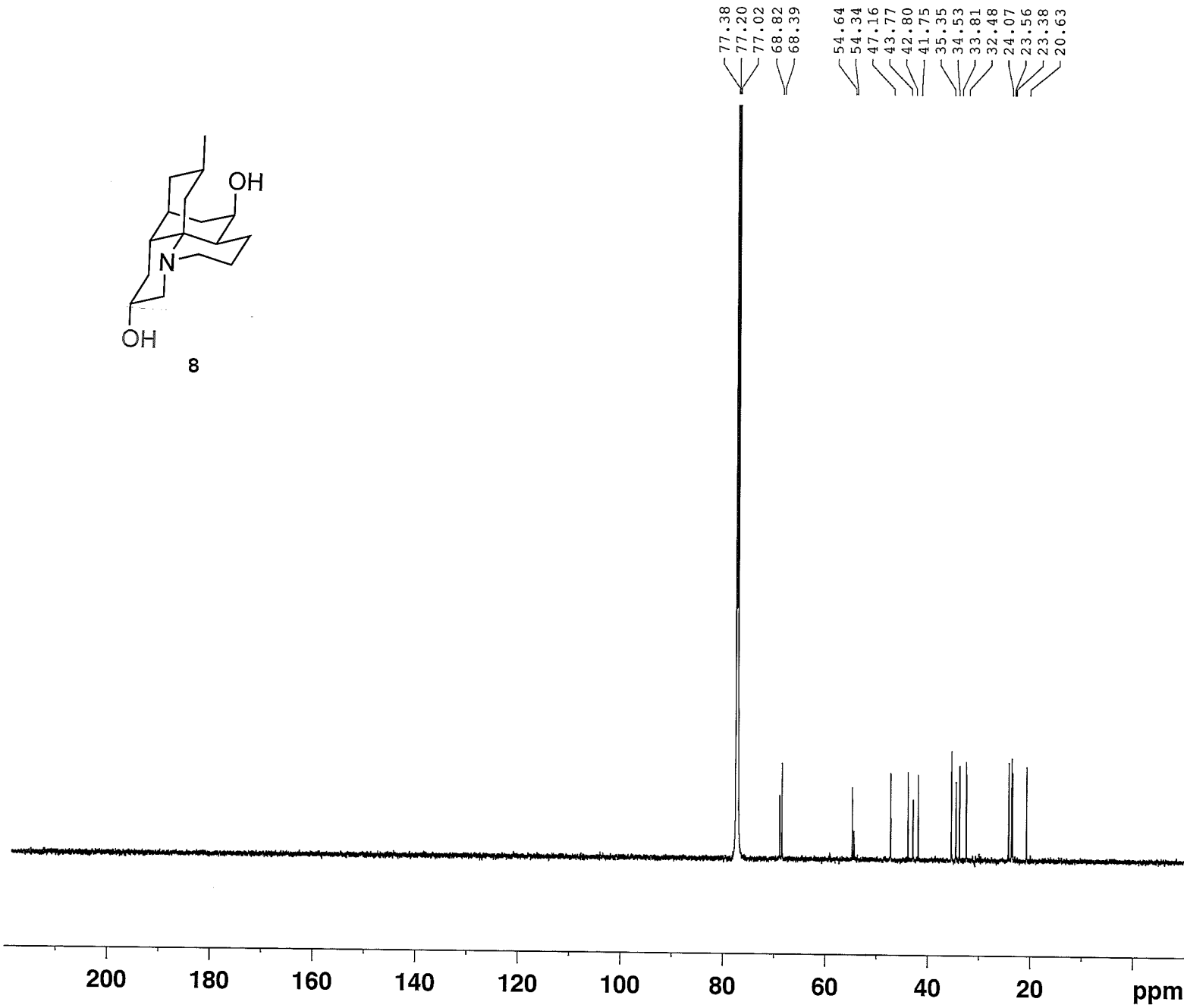
```
NAME        MS IX-44 paniculi
EXPNO                    3
PROCNO                   1
Date_             20120928
Time                 20.34
INSTRUM              spect
PROBHD     5 mm CPDCH 13C
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                   18204
DS                       4
SWH             41666.668 Hz
FIDRES           0.635783 Hz
AQ              0.7864820 se
RG                     203
DW                  12.000 us
DE                   16.50 us
TE                   298.2 K
D1             2.00000000 se
D11            0.03000000 se
TD0                      1

======== CHANNEL f1 ======
NUC1                   13C
P1                    9.00 us
PL1                   4.50 dB
PL1W          38.14553833 W
SFO1          176.0697436 MH

======== CHANNEL f2 ======
CPDPRG2            waltz16
NUC2                    1H
PCPD2                65.00 us
PL2                  -3.20 dB
PL12                 13.60 dB
PL13                120.00 dB
PL2W          33.59817505 W
PL12W          0.70196527 W
PL13W          0.00000000 W
SFO2          700.1499406 MH
SI                   32768
SF            176.0520930 MH
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40
```

deacetylpaniculine



NAME	MS IX-50 Deacetoxypaniculi
EXPNO	2
PROCNO	1
Date_	20121009
Time	8.47
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	32
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	25.4
DW	42.000 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471600 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

deacetylpaniculine carbon



NAME	MS IX-50 Deacetoxypaniculi
EXPNO	3
PROCNO	1
Date_	20121009
Time	1.48
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	801
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521108 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40