

Electronic Supplementary Information

Studies Towards the Synthesis of Bielschowskysin. Construction of the Highly Functionalized Bicyclo[3.2.0]heptane Segment

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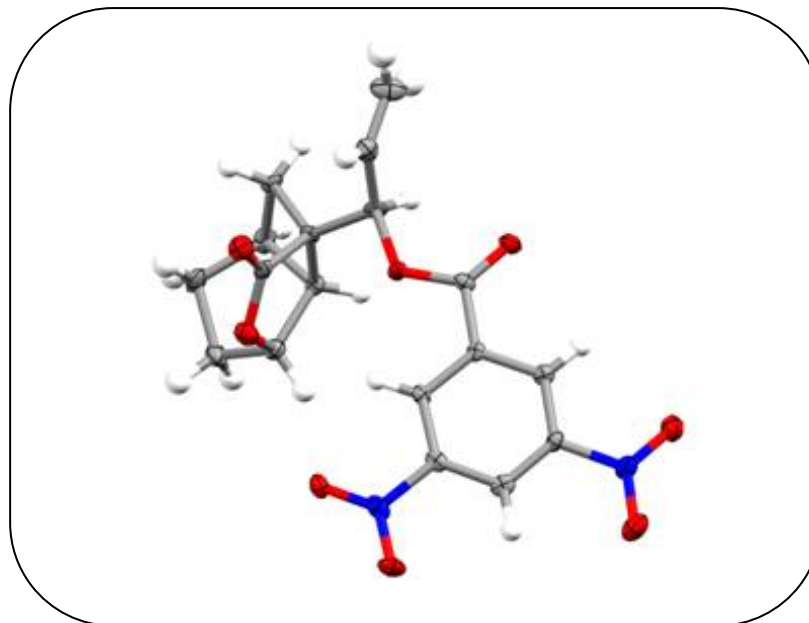
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Copies of ^1H and ^{13}C NMR spectra

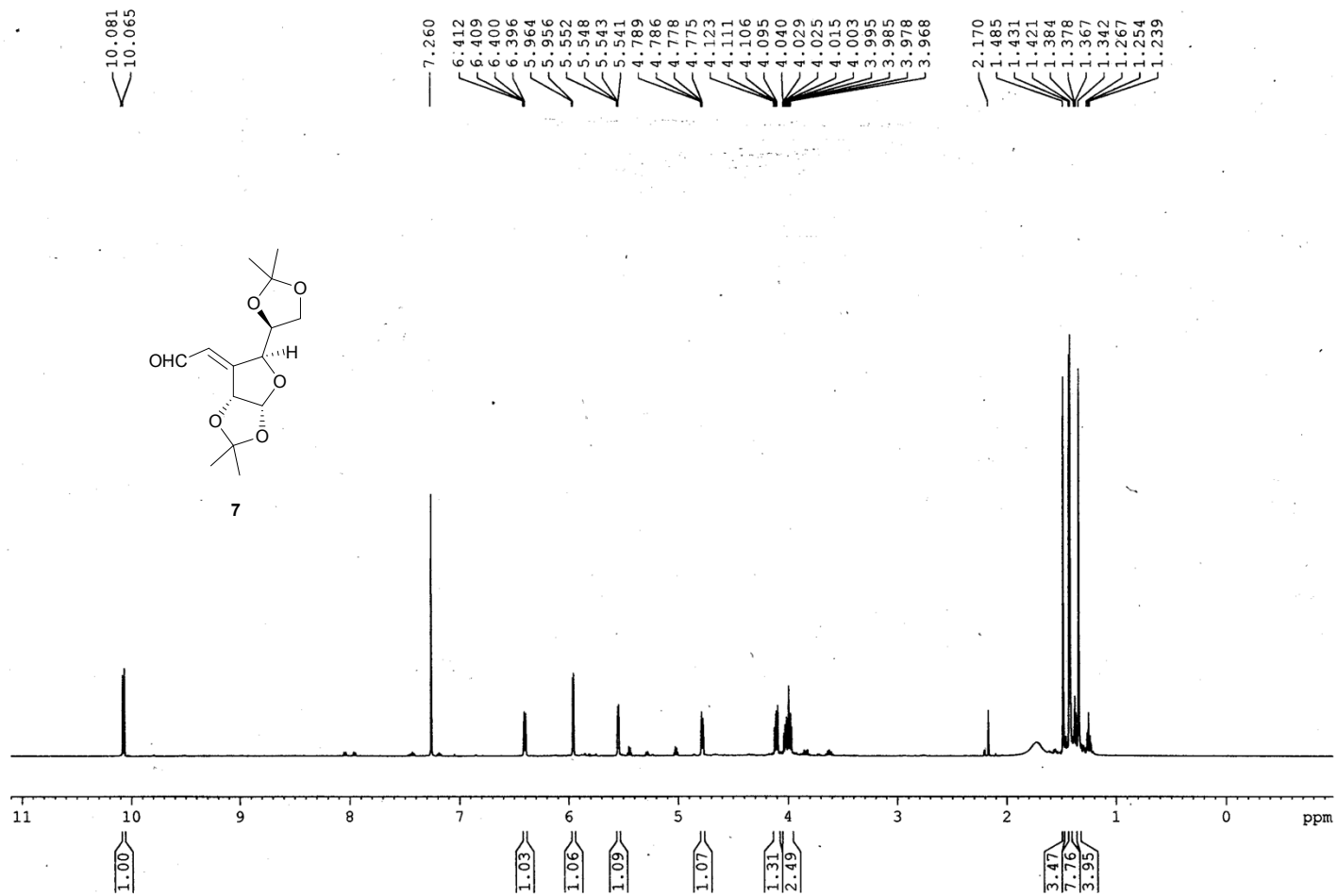
ORTEP of compound 16b



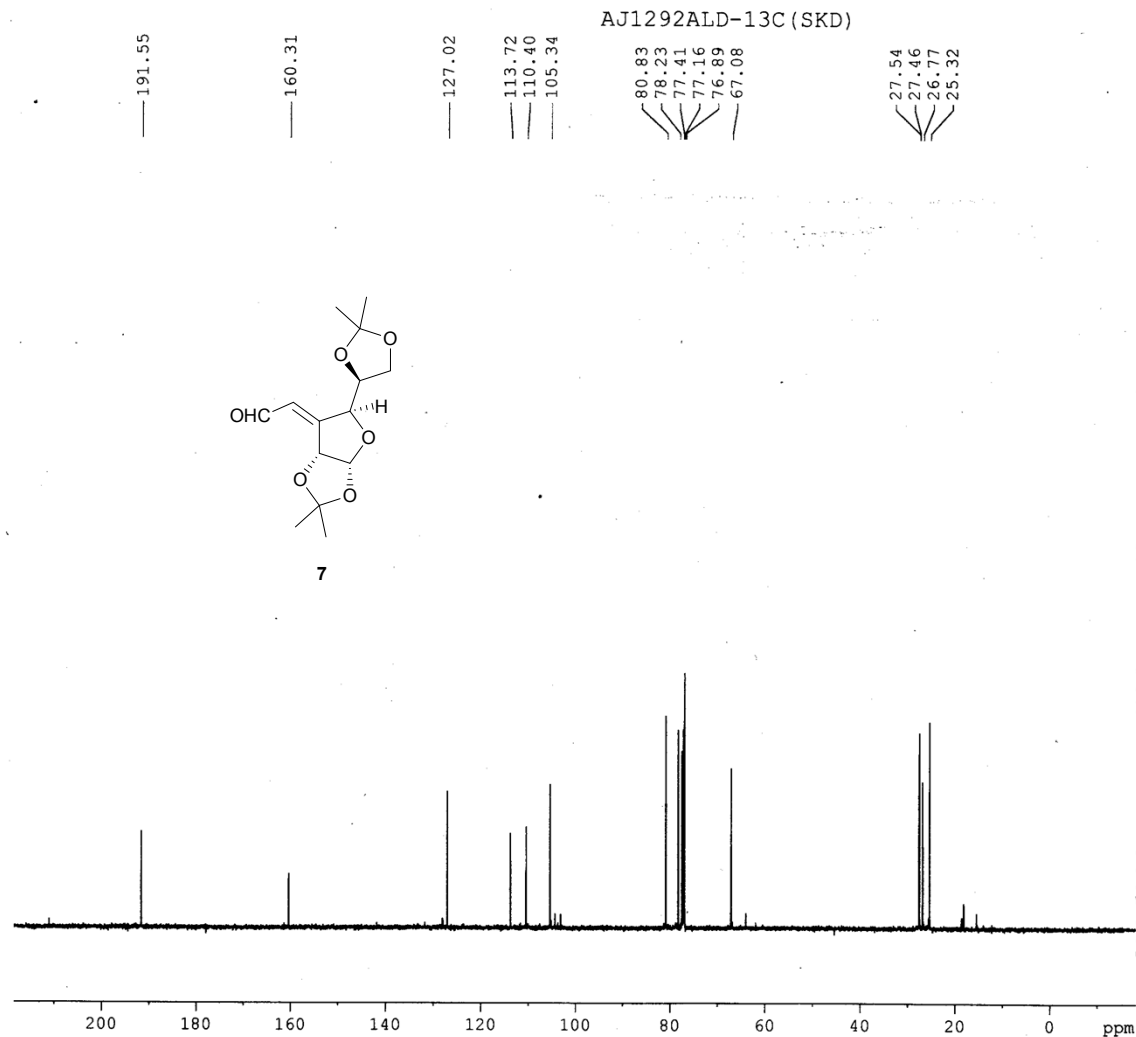
Crystal data: Compound **16b**. Colorless, needle shaped crystal. Empirical formula $C_{18}H_{16}N_2O_8$, $M_r = 388.33$, Orthorhombic space group $P2_12_12_1$, $a = 5.830(3)$, $b = 9.556(4)$, $c = 31.119(14)$ Å, $v = 1733.7(14)$ Å³, $z = 4$, $T = 150$ K, $\rho_{\text{calcd}} = 1.488$ g cm⁻³, $F(000) = 808.0$, λ (Mo-K α) = 0.71073 Å, μ mm⁻¹ = 0.119, $2\theta_{\text{max}} = 50.0^\circ$, 4171 total reflections, 2858 unique reflections, 1802 observed ($I > 2\sigma(I)$), 253 refined parameter, $R_1 = 0.0596$, $wR_2 = 0.1866$, with GOF = 0.999. X-ray single crystal data were collected using MoK α ($\lambda = 0.7107$ Å) radiation on a SMART APEX II diffractometer equipped with CCD area detector. Data collection, data reduction, structure solution/refinement were carried out using the software package of SMART APEX. The structure was solved by direct method and refined in a routine manner. Non hydrogen atoms were treated anisotropically. The hydrogen atoms were geometrically fixed. CCDC (CCDC Nos. 902058 for **16b**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

¹H NMR of Compound 7 (CDCl₃, 500 MHz)

AJ1292ALD2-1H (SR)



¹³C NMR of Compound 7 (CDCl₃, 125 MHz)



```

NAME      AJ1292ALD-13C
EXPNO     1
PROCNO    1
Date_     20120629
Time      9.59
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         299
DS         4
SWH        29761.904 Hz
FIDRES     0.908261 Hz
AQ         0.5505524 sec
RG         32
DW         16.800 usec
DE         6.50 usec
TE         298.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1
    
```

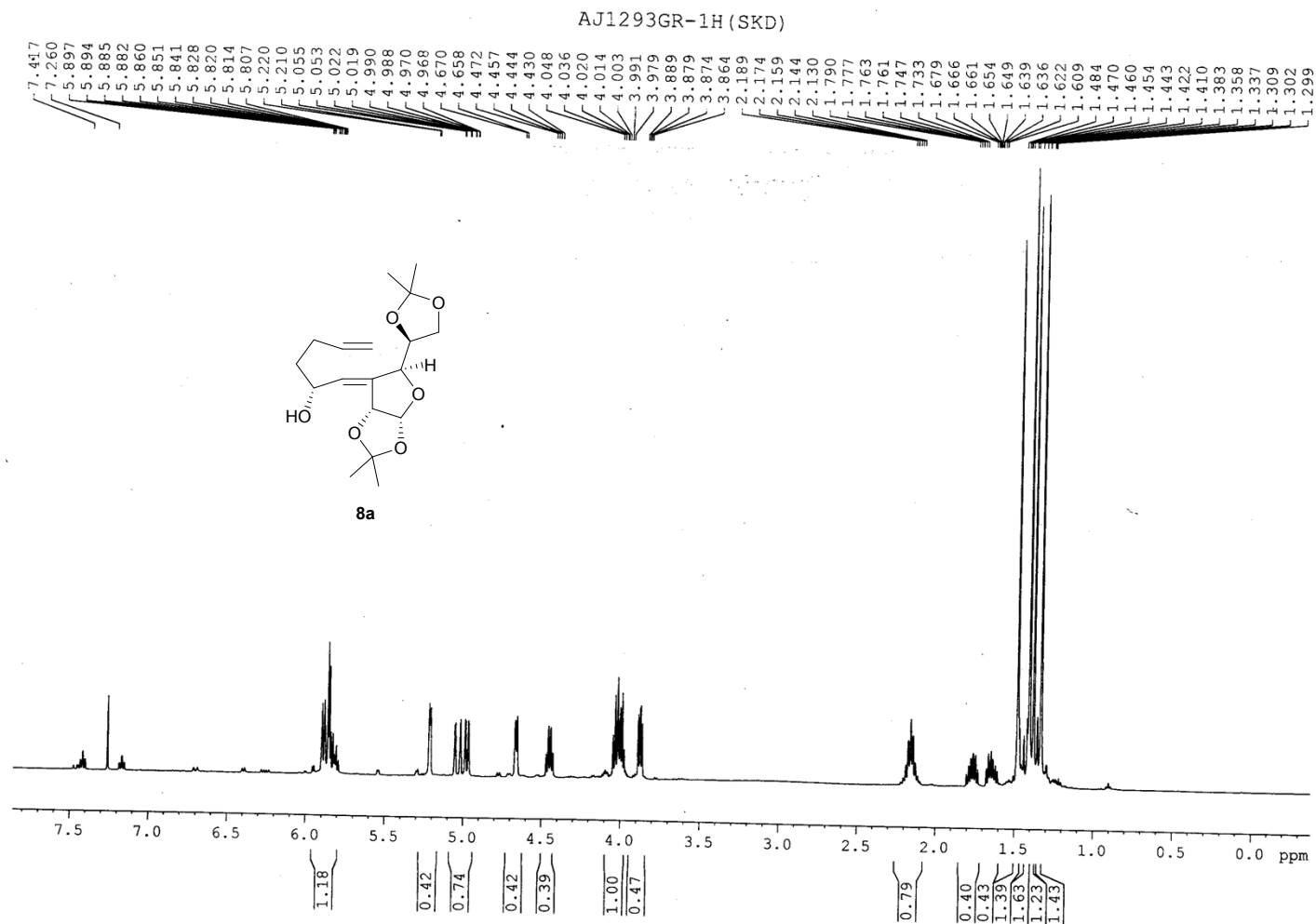
```

===== CHANNEL f1 =====
NUC1      13C
P1        9.88 usec
PL1       0.50 dB
PL1W      82.16106415 W
SFO1      125.7703643 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12      16.50 dB
PL13      20.00 dB
PL2W      15.50318813 W
PL12W     0.43693921 W
PL13W     0.19517358 W
SFO2      500.1320005 MHz
SI        16384
SF        125.7577753 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

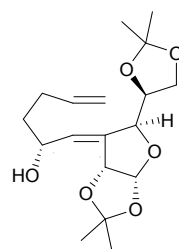
¹H NMR of Compound 8a (CDCl₃, 500 MHz)



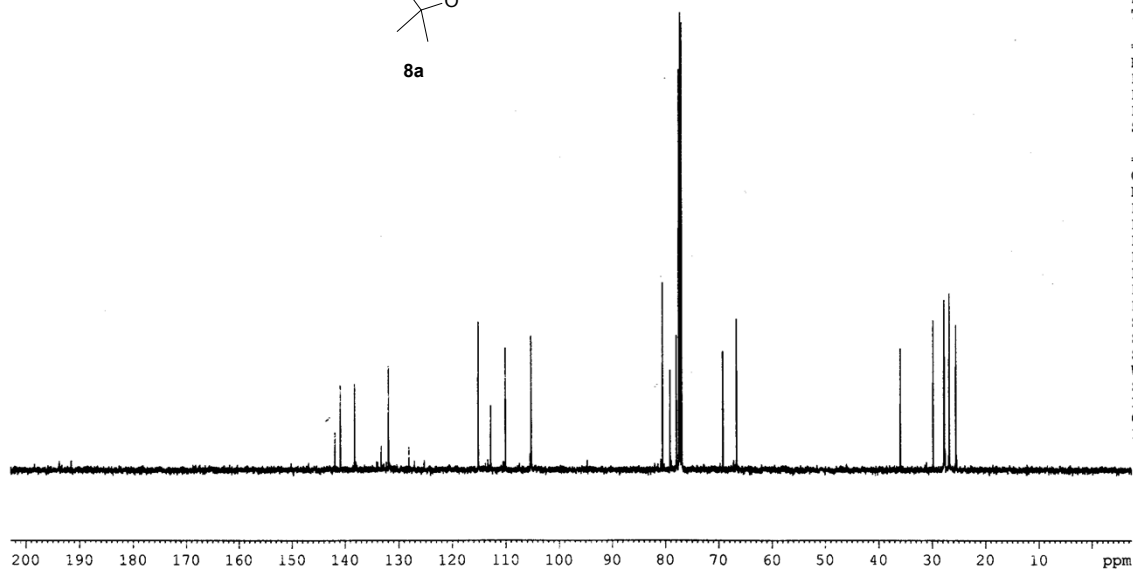
¹³C NMR of Compound 8a (CDCl₃, 125 MHz)

AJ1293GR-13C (SKD)

141.87
140.86
138.59
133.25
131.88
131.80
128.08
115.11
112.81
110.02
105.37
105.21
105.11
80.50
79.09
77.90
77.41
77.16
76.90
69.17
66.60
35.89
29.77
27.73
27.65
27.56
27.53
26.73
25.52
25.37



8a



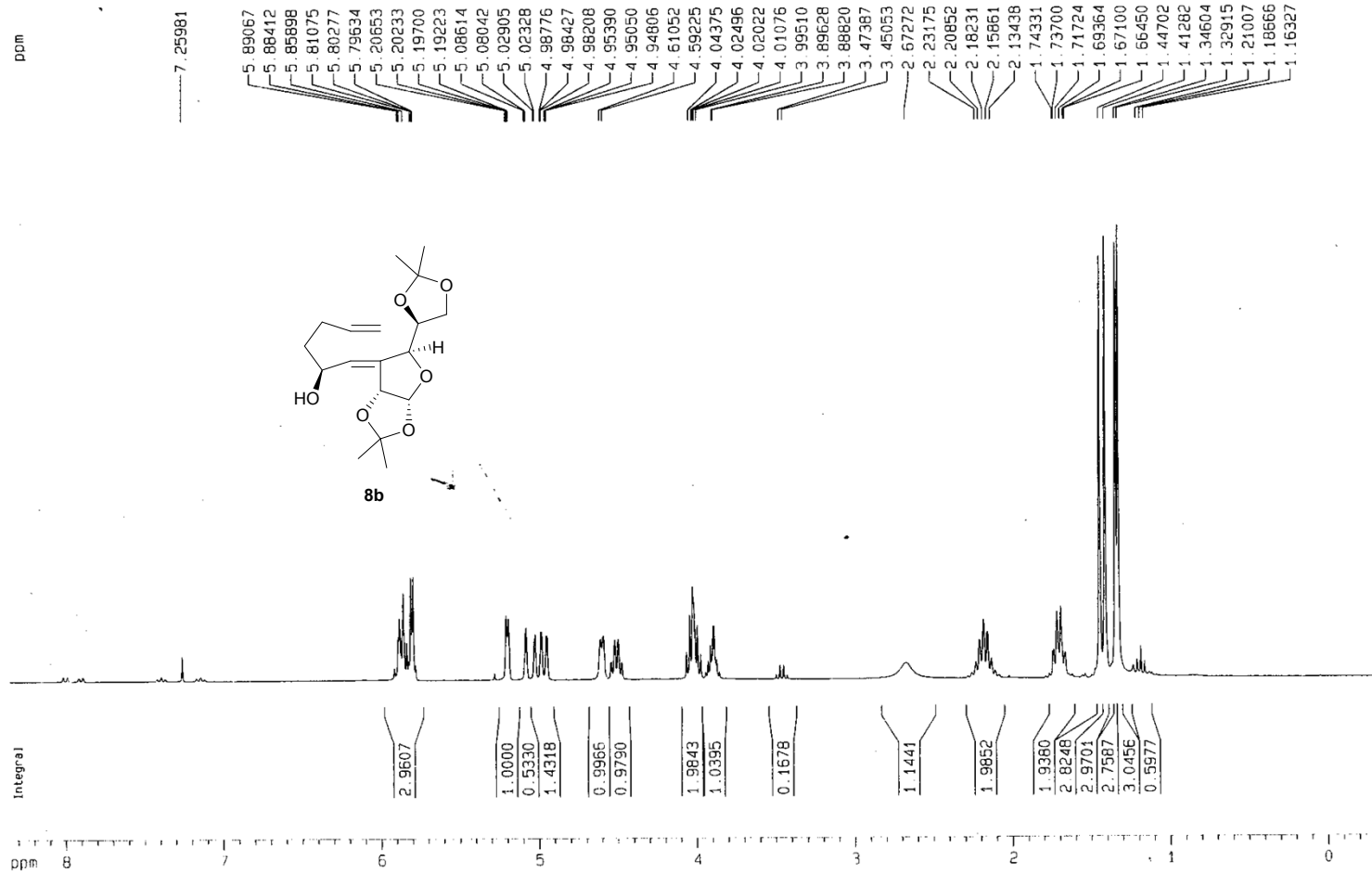
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NAME      AJ1293GR-13C
EXPNO     1
PROCNO    1
Date_     20120702
Time      13.43
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        32768
SOLVENT   CDC13
NS        551
DS        4
SWH       29761.904 Hz
FIDRES    0.908261 Hz
AQ        0.5505524 sec
RG        32
DW        16.800 usec
DE        6.50 usec
TE        299.6 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
```

```
----- CHANNEL f1 -----
NUC1      13C
P1        9.88 usec
PL1       0.50 dB
PL1W      82.16106415 W
SFO1      125.7703643 MHz
```

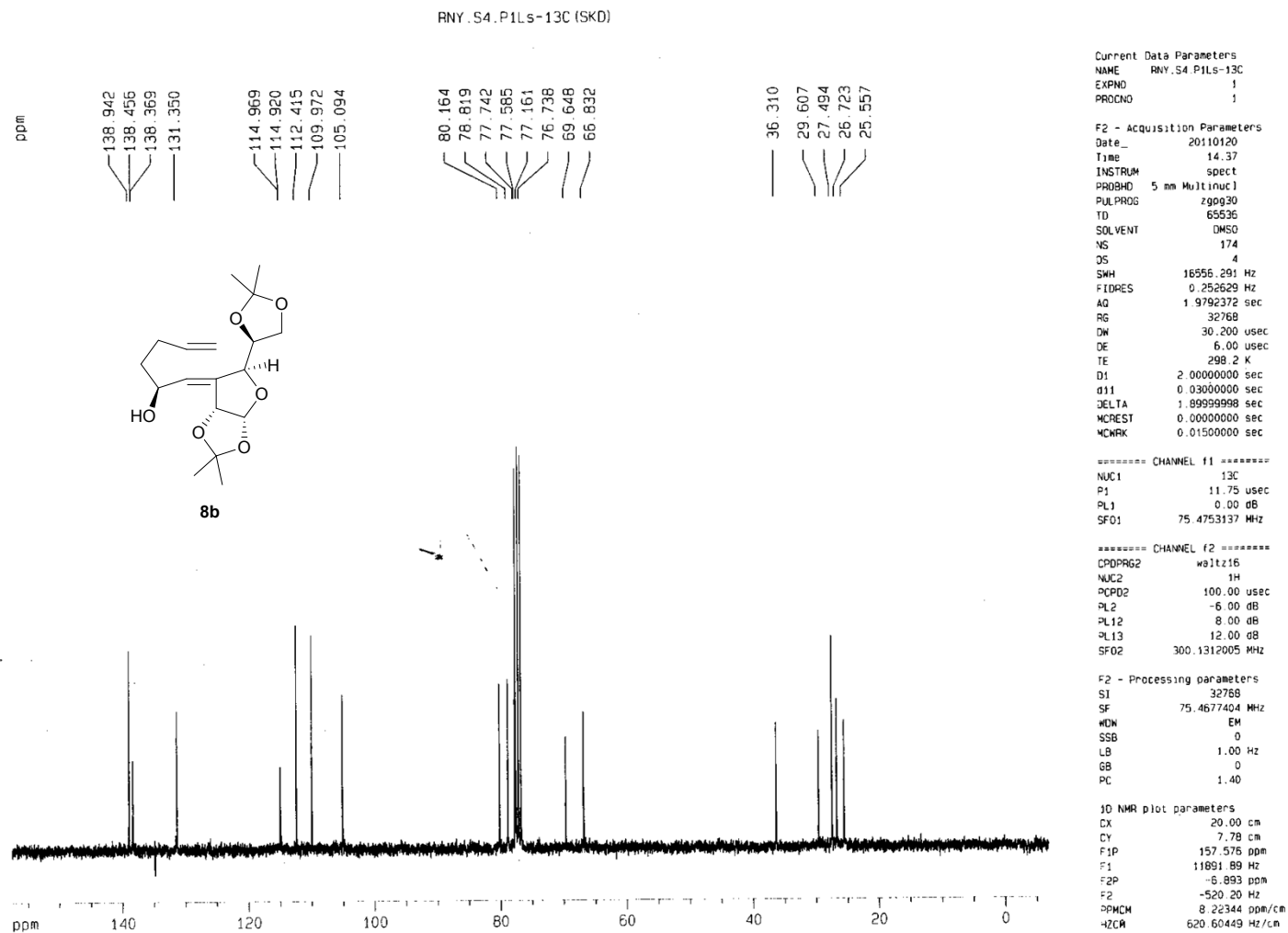
```
----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12      16.50 dB
PL13      20.00 dB
PL2W      15.50318813 W
PL12W     0.43693921 W
PL13W     0.19517358 W
SFO2      500.1320005 MHz
SI        16384
SF        125.7577717 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```

¹H NMR of Compound 8b (CDCl₃, 300 MHz)

RNY S4.P1L5-1H (SKD)

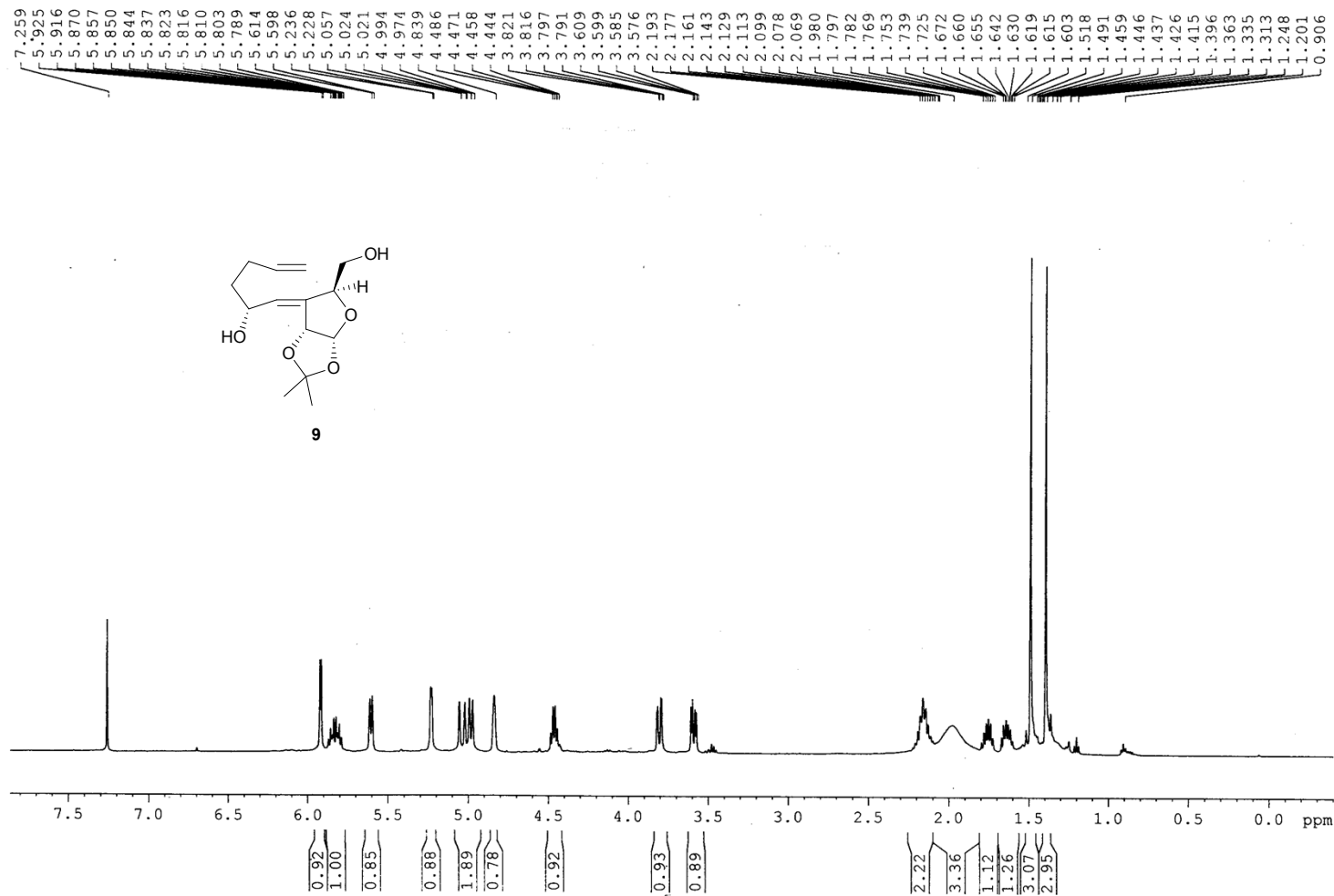


¹H NMR of Compound 8b (CDCl₃, 75 MHz)



¹H NMR of Compound 9 (CDCl₃, 500 MHz)

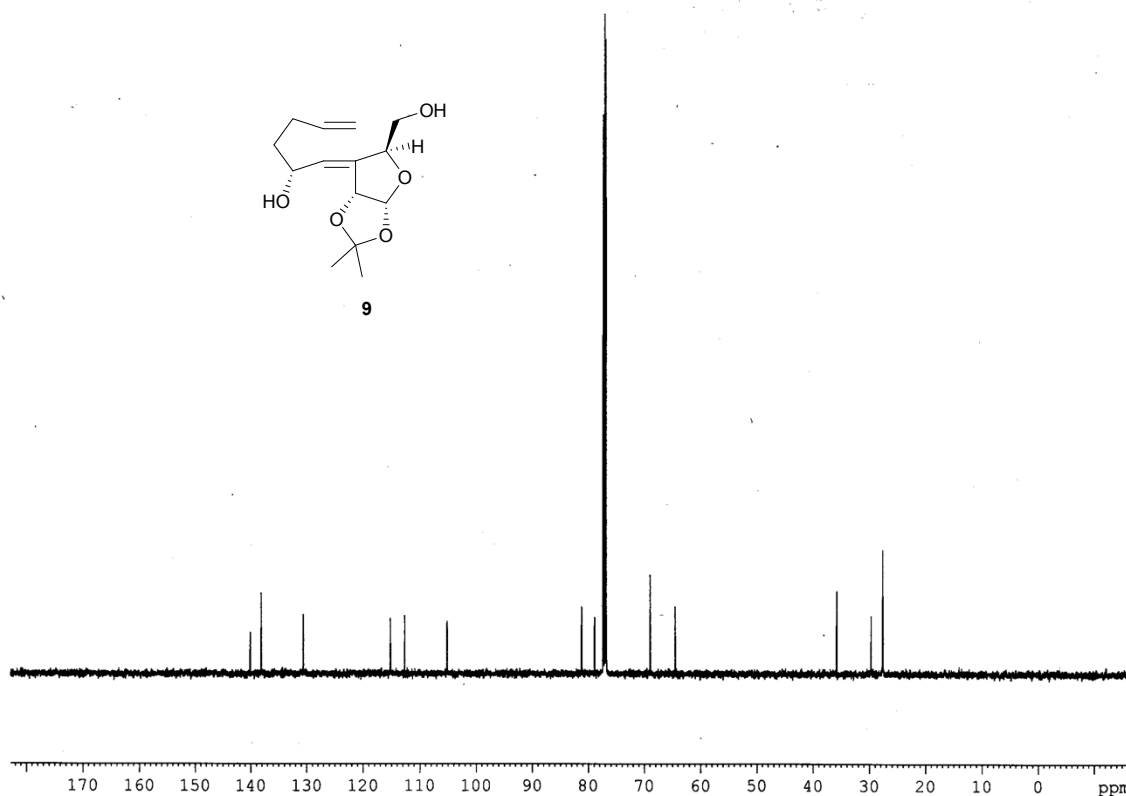
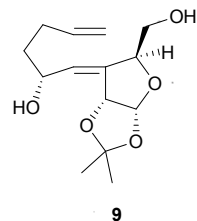
AJ1294P-1H (SR)



¹³C NMR of Compound 9 (CDCl₃, 125 MHz)

AJ1294P-13C (SR)

140.07
138.10
130.59
115.15
112.62
105.12
81.17
78.90
77.36
77.10
76.85
68.98
64.50
35.83
29.70
27.65
27.58



```

NAME      AJ1294P-13C
EXPNO     1
PROCNO    1
Date_     20120704
Time      16.49
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        32768
SOLVENT   CDCl3
NS        768
DS        4
SWH       36231.883 Hz
FIDRES    1.105709 Hz
AQ        0.4522484 sec
RG        32
DW        13.800 usec
DE        6.50 usec
TE        300.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

```

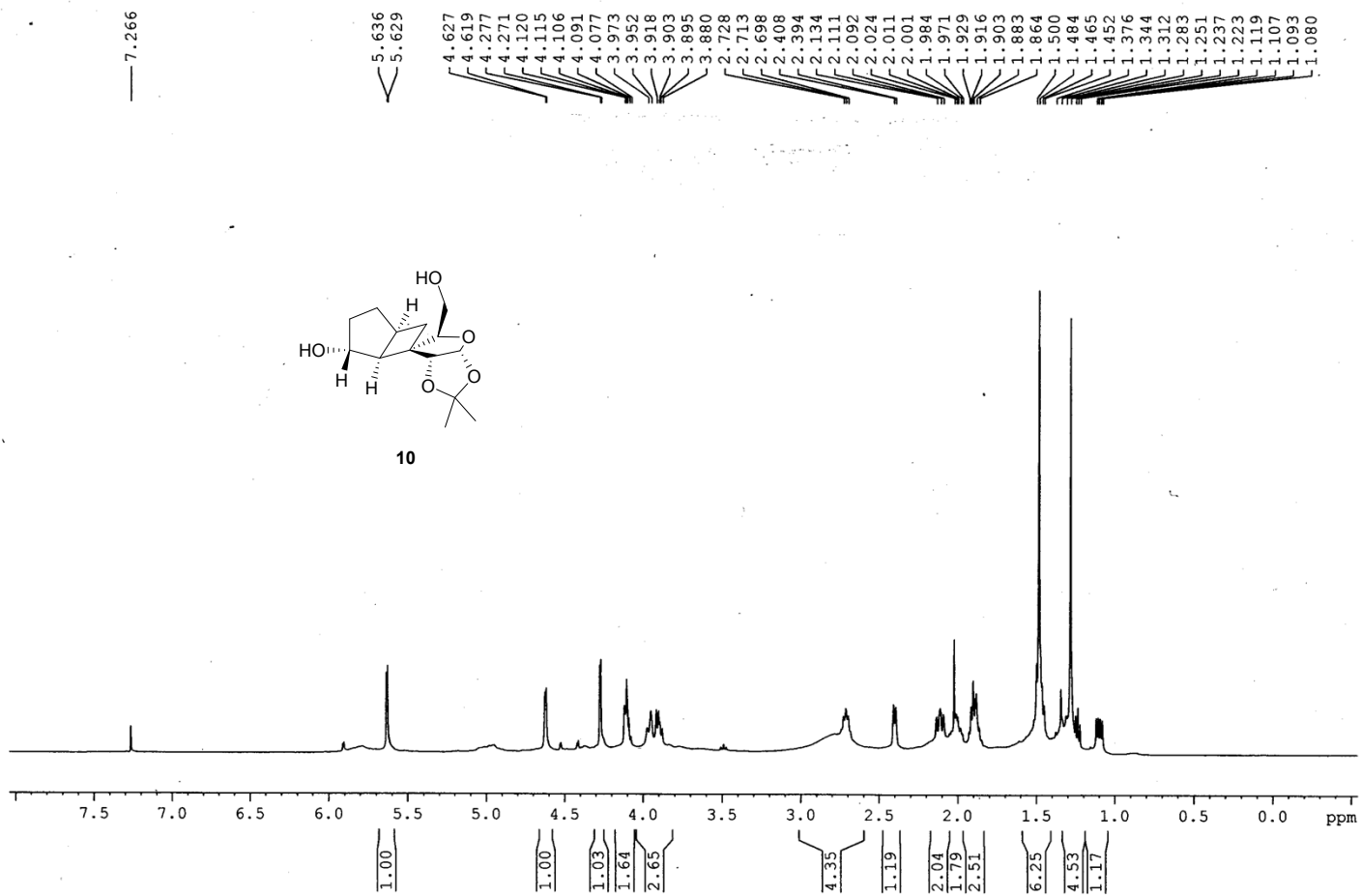
===== CHANNEL f1 =====
NUC1      13C
P1        9.88 usec
PL1       0.50 dB
PL1W      82.16106415 W
SFO1      125.7703643 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12      16.50 dB
PL13      20.00 dB
PL2W      15.50318813 W
PL12W     0.43693921 W
PL13W     0.19517358 W
SFO2      500.1320005 MHz
SI        16384
SF        125.7577768 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

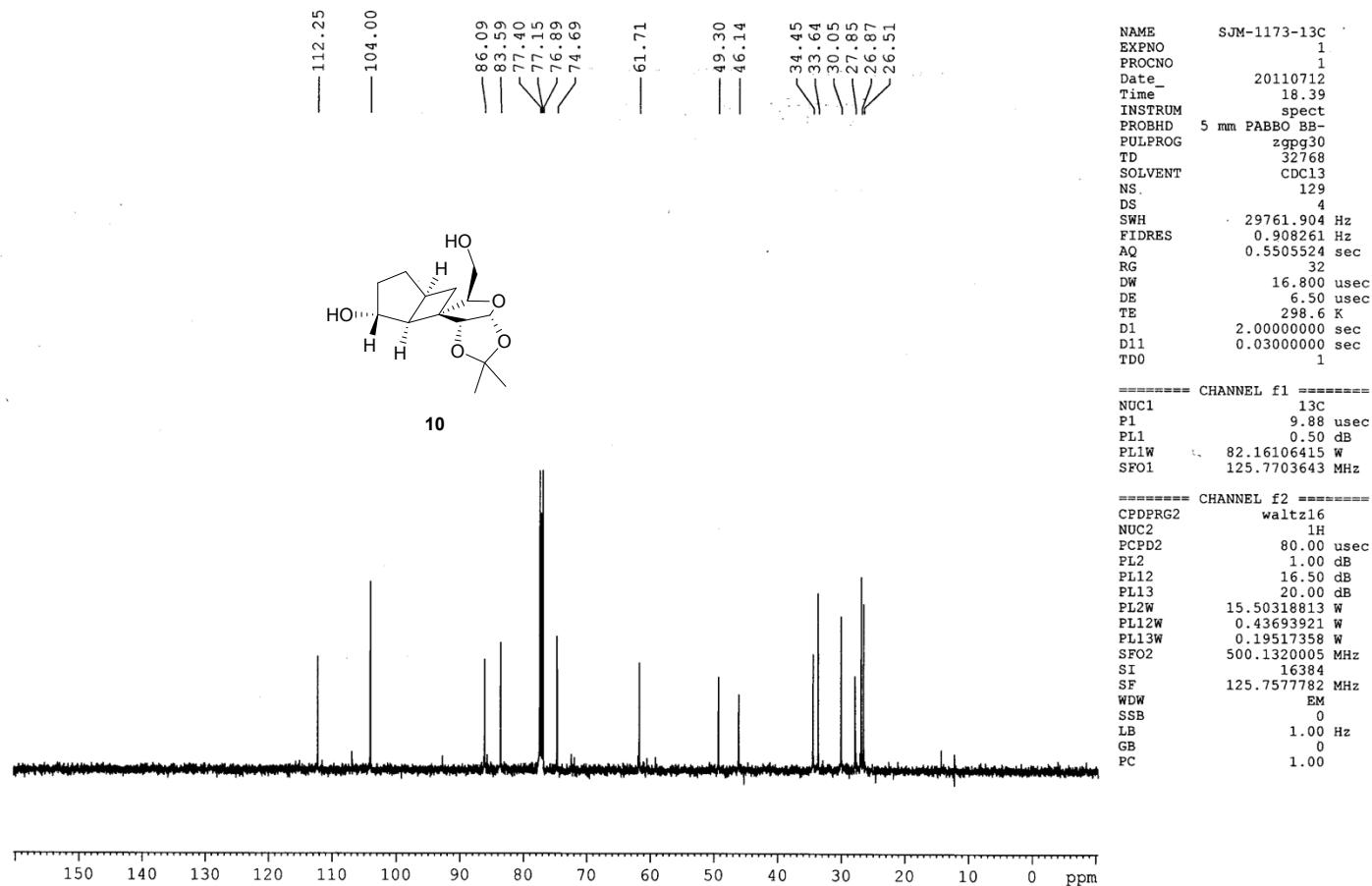
¹H NMR of Compound 10 (CDCl₃, 500 MHz)

SJM-1173-1H (SR)

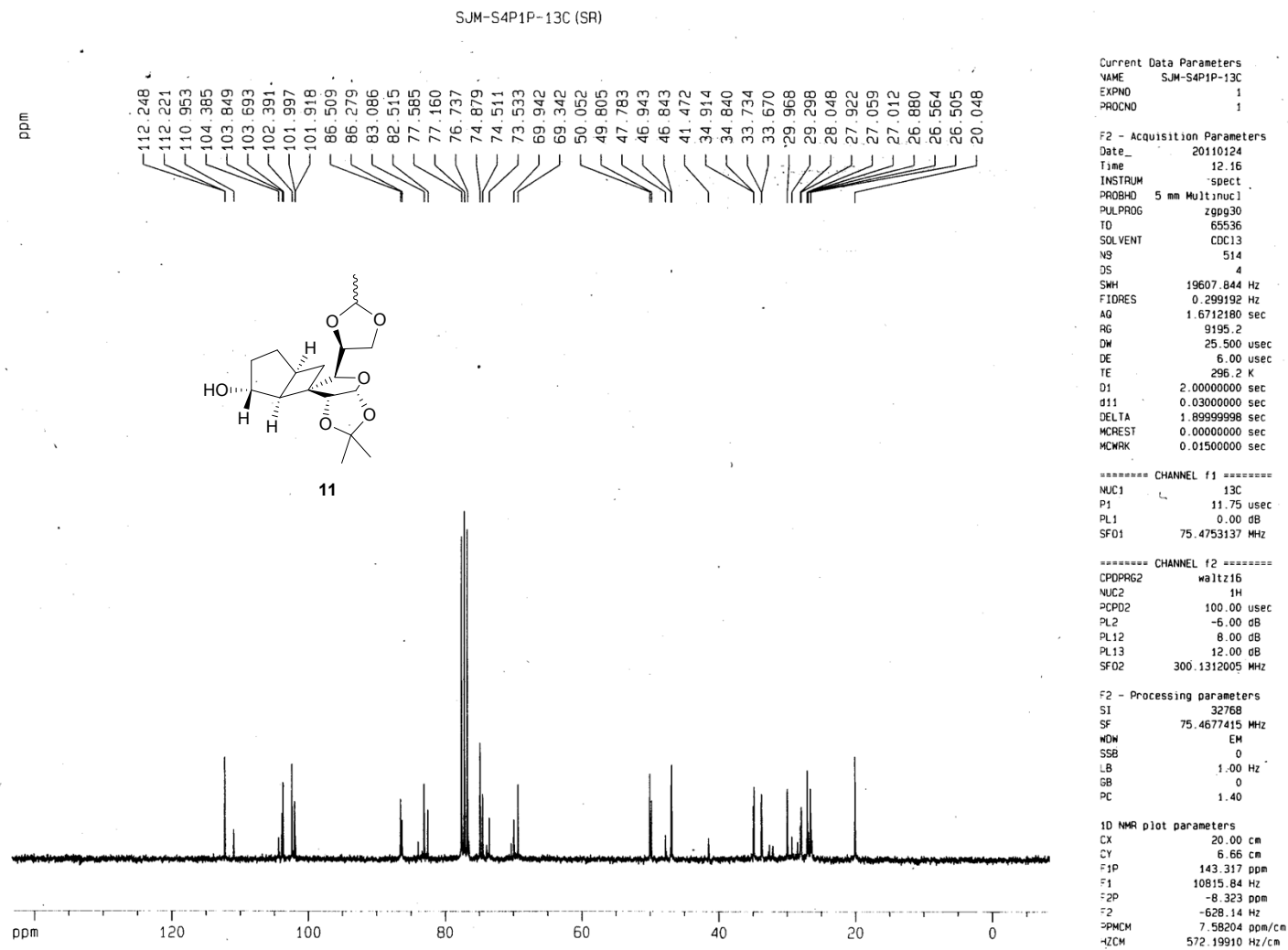


¹³C NMR of Compound 10 (CDCl₃, 125 MHz)

SJM-1173-13C (SR)

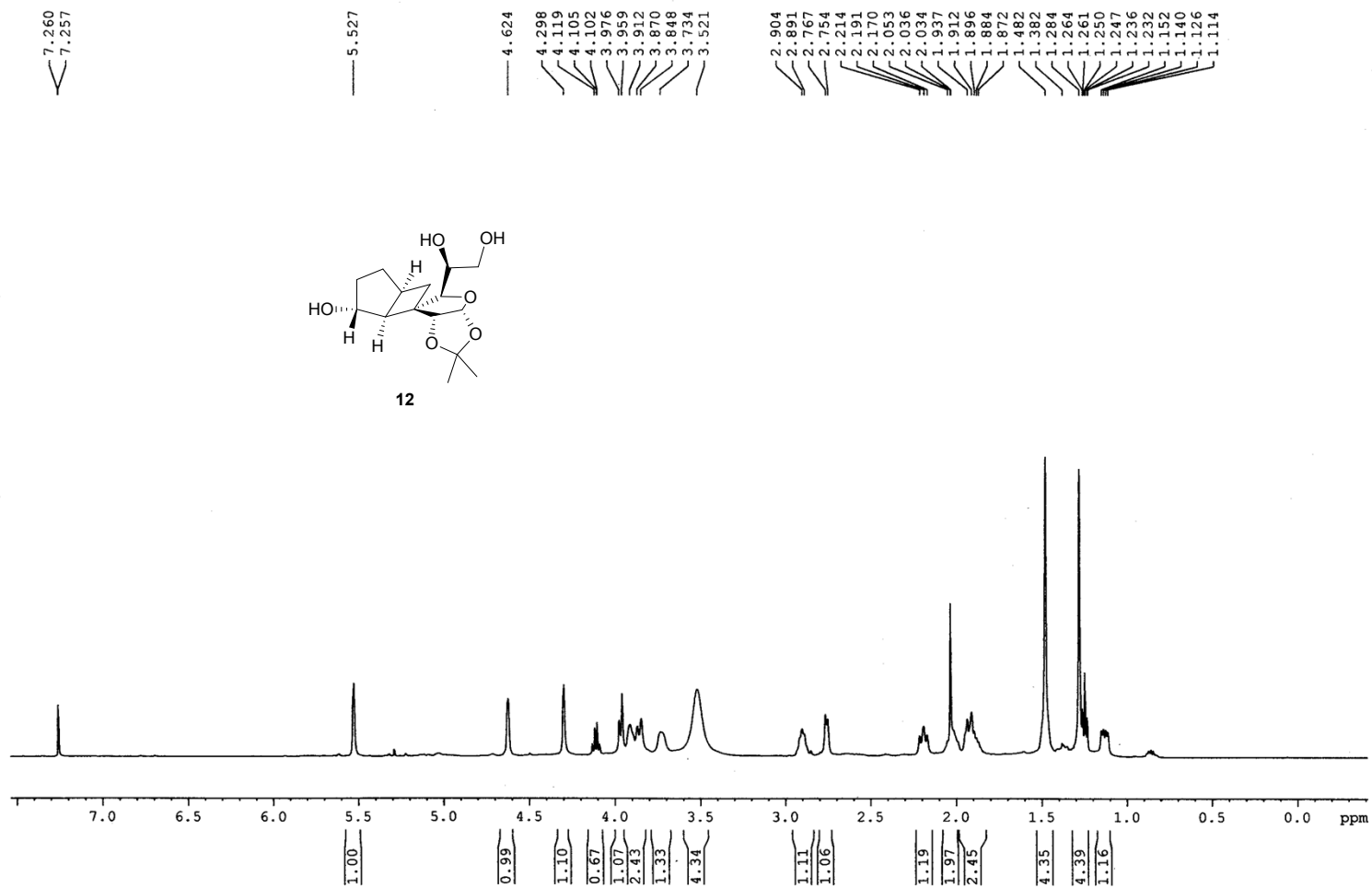


¹³C NMR of Compound 11 (CDCl₃, 75 MHz)



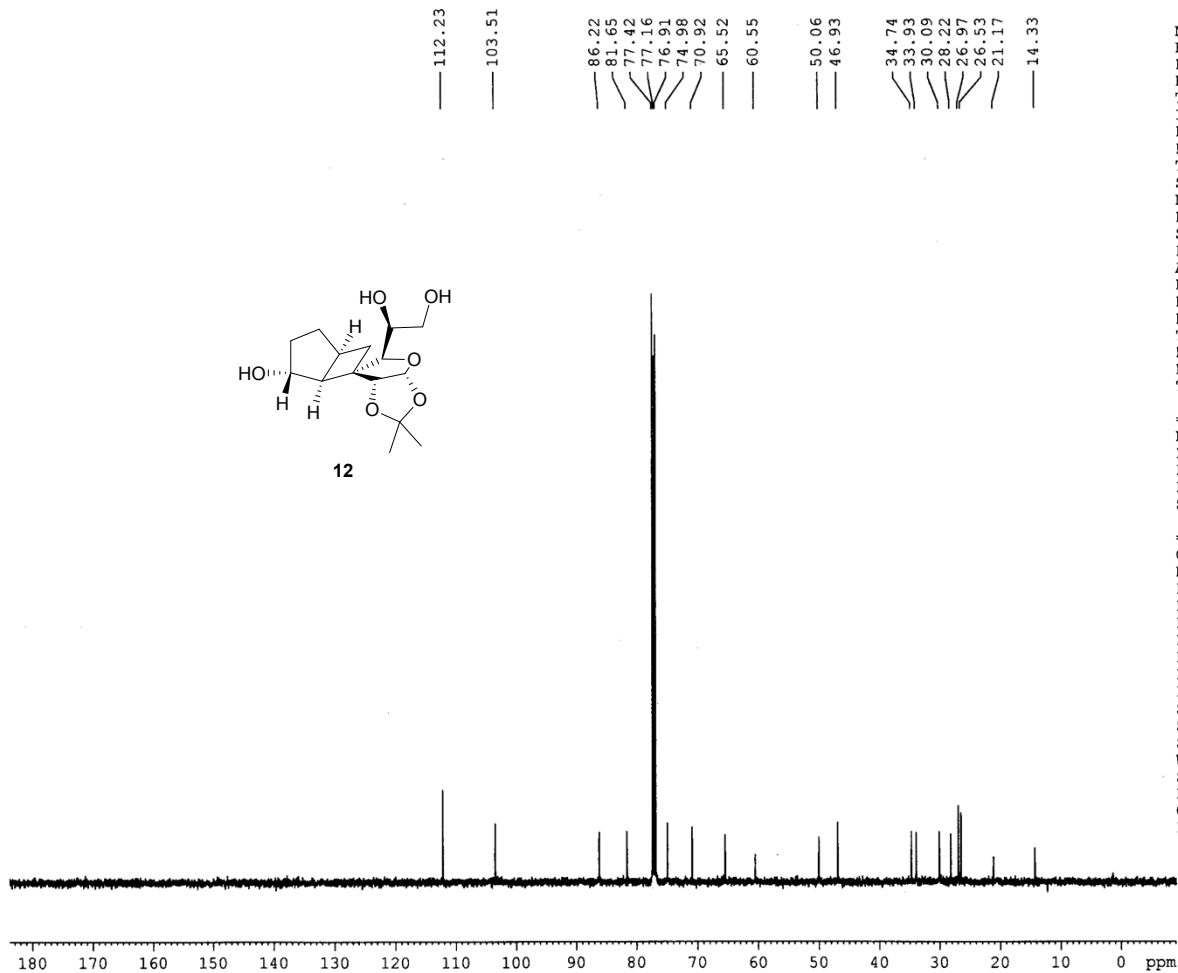
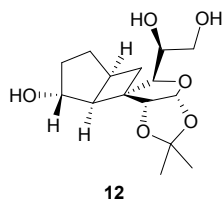
¹H NMR of Compound 12 (CDCl₃, 500 MHz)

AJ1276TP2-1H(SKD)



¹³C NMR of Compound 12 (CDCl₃, 125 MHz)

AJ1276TP2-13C (SKD)



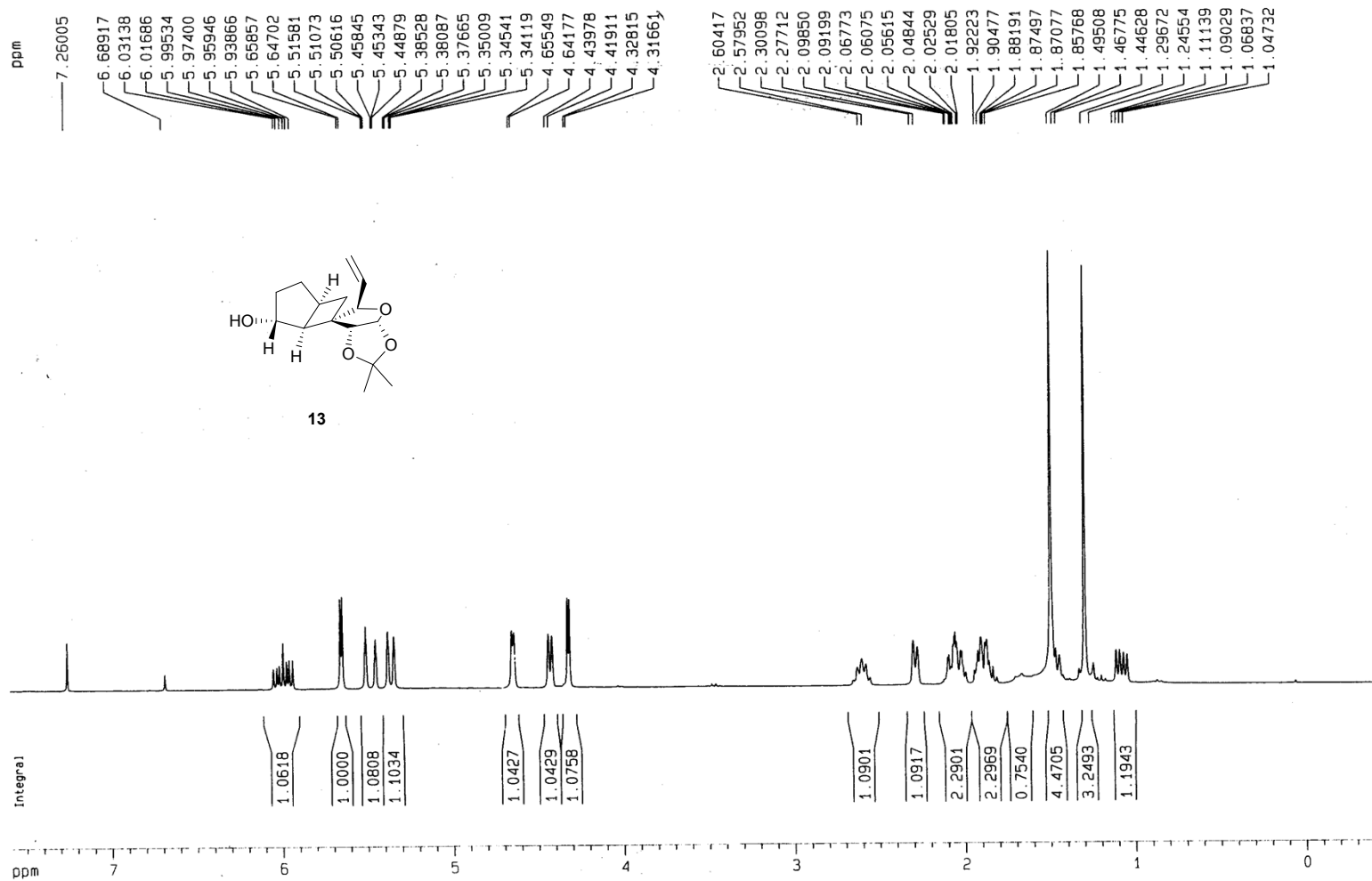
```
NAME      AJ1276TP2-13C
EXPNO     1
PROCNO    1
Date_     20120530
Time      14.40
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         542
DS         4
SWH        29761.904 Hz
FIDRES     0.908261 Hz
AQ         0.5505524 sec
RG         32
DW         16.800 usec
DE         6.50 usec
TE         298.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1
```

```
===== CHANNEL f1 =====
NUC1      13C
P1         9.88 usec
PL1        0.50 dB
PL1W       82.16106415 W
SF01      125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         1.00 dB
PL12       16.50 dB
PL13       20.00 dB
PL2W       15.50318813 W
PL12W      0.43693921 W
PL13W      0.19517358 W
SF02       500.1320005 MHz
SI         16384
SF         125.7577698 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
```

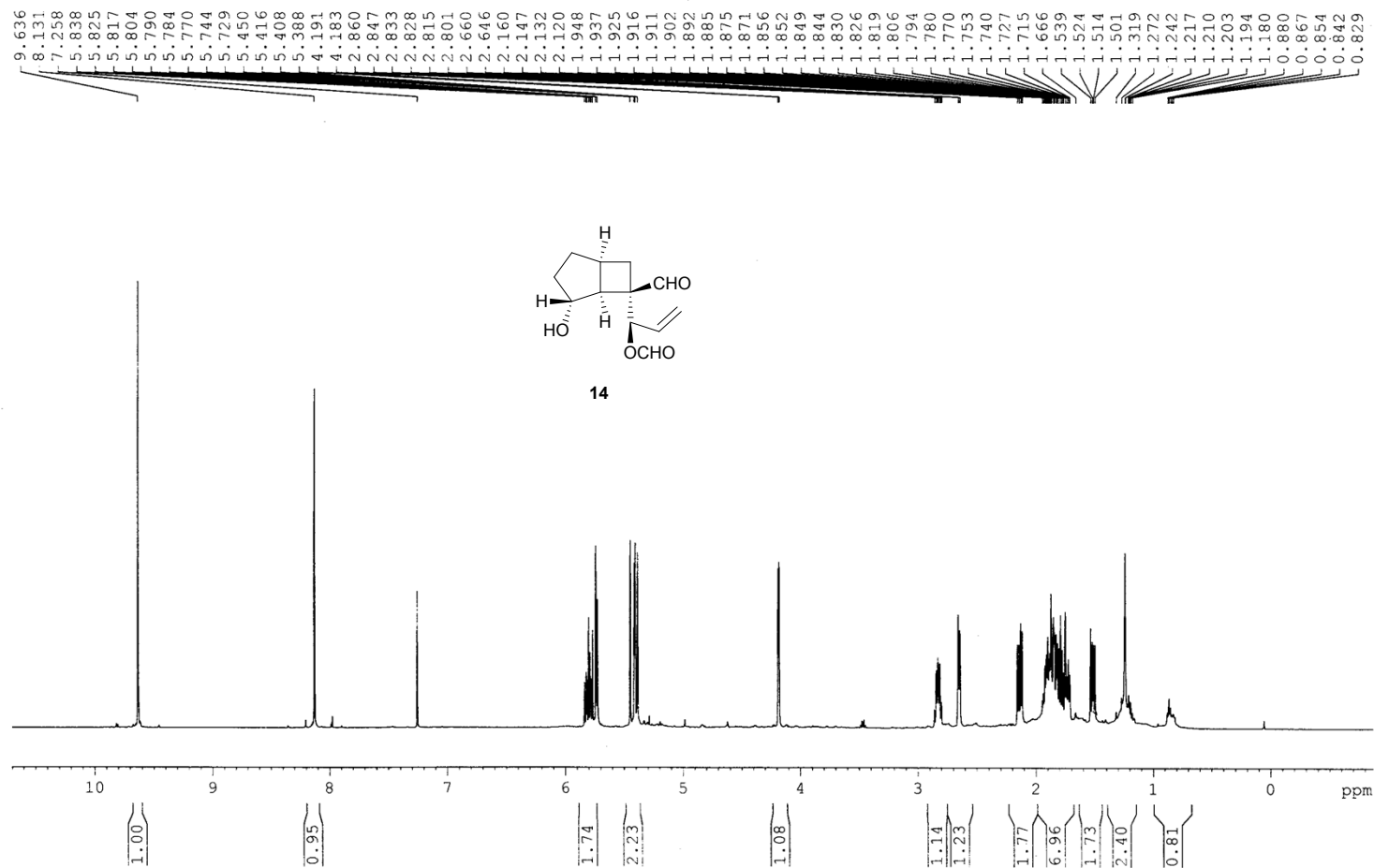

¹H NMR of Compound 13 (CDCl₃, 300 MHz)

SJM. S4. P4A-1H (SKD)



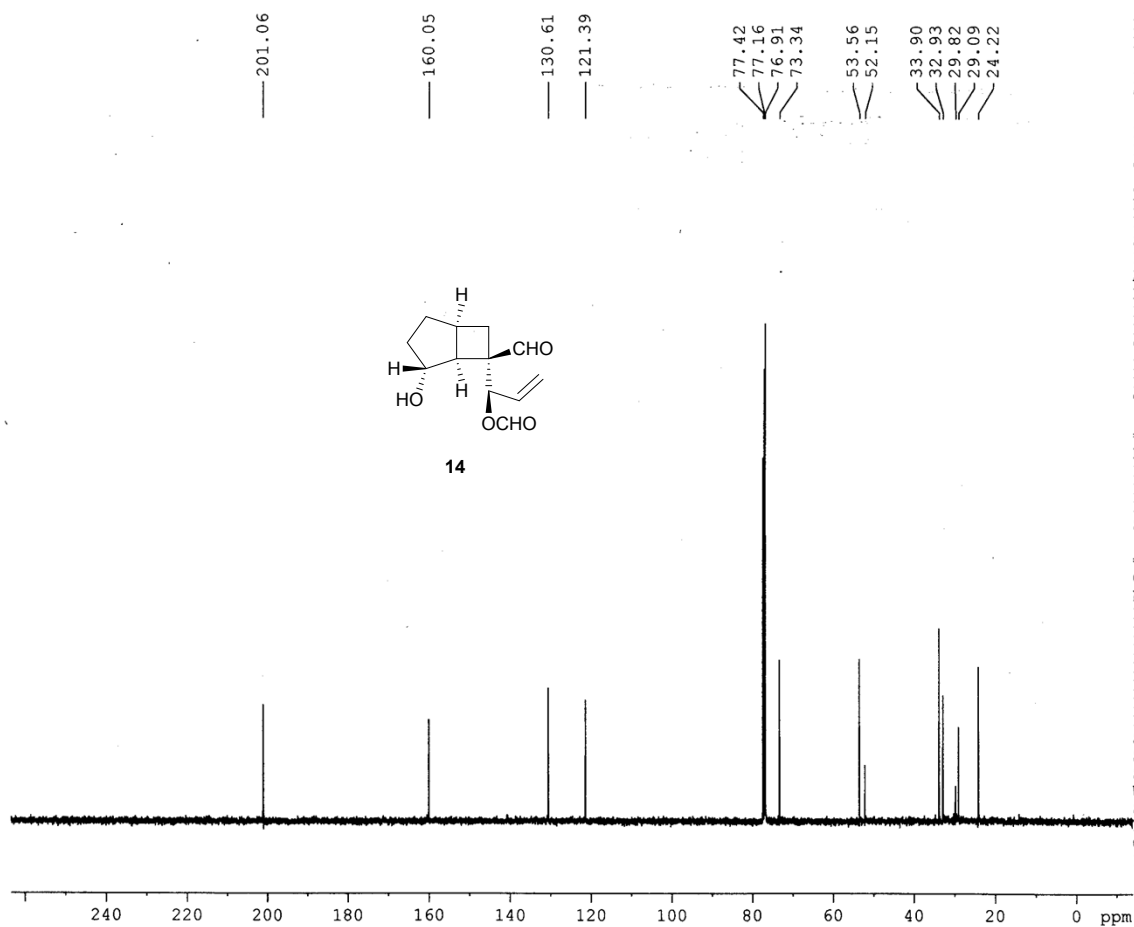
¹H NMR of Compound 14 (CDCl₃, 500 MHz)

AJ1291FRM-1H(SR)



¹³C NMR of Compound 14 (CDCl₃, 125 MHz)

AJ1291FRM-13C (SR)



```

NAME      AJ1291FRM-13C
EXPNO     1
PROCNO    1
Date_     20120626
Time      18.50
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         523
DS         4
SWH       41666.668 Hz
FIDRES    1.271566 Hz
AQ         0.3932660 sec
RG         32
DW         12.000 usec
DE         6.50 usec
TE         299.5 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
    
```

```

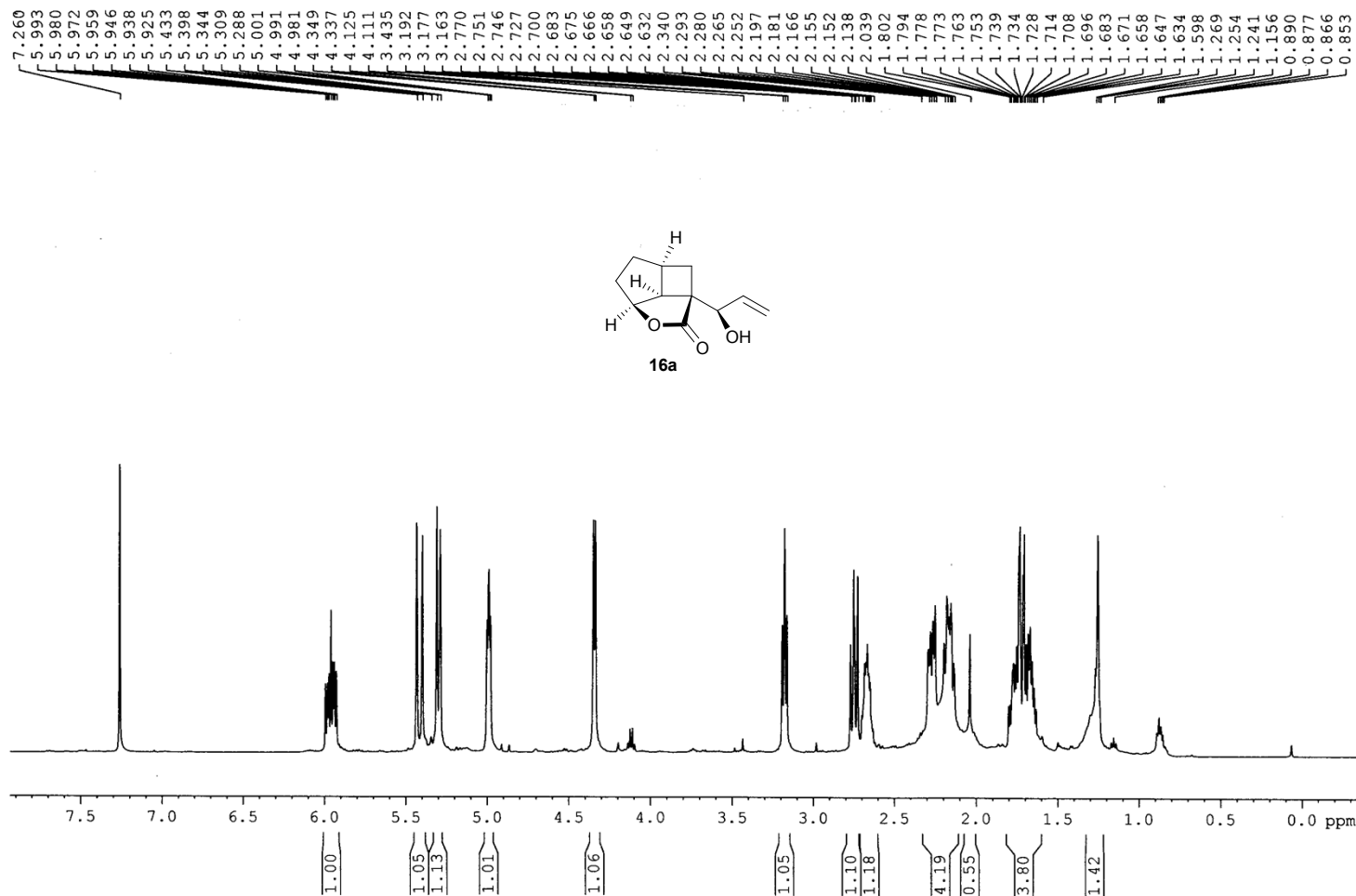
===== CHANNEL f1 =====
NUC1      13C
P1         9.88 usec
PL1        0.50 dB
PL1W       82.16106415 W
SFO1      125.7703643 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        1.00 dB
PL12       16.50 dB
PL13       20.00 dB
PL2W      15.50318813 W
PL12W     0.43693921 W
PL13W     0.19517358 W
SFO2      500.1320005 MHz
SI         16384
SF         125.7577706 MHz
WDW       EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

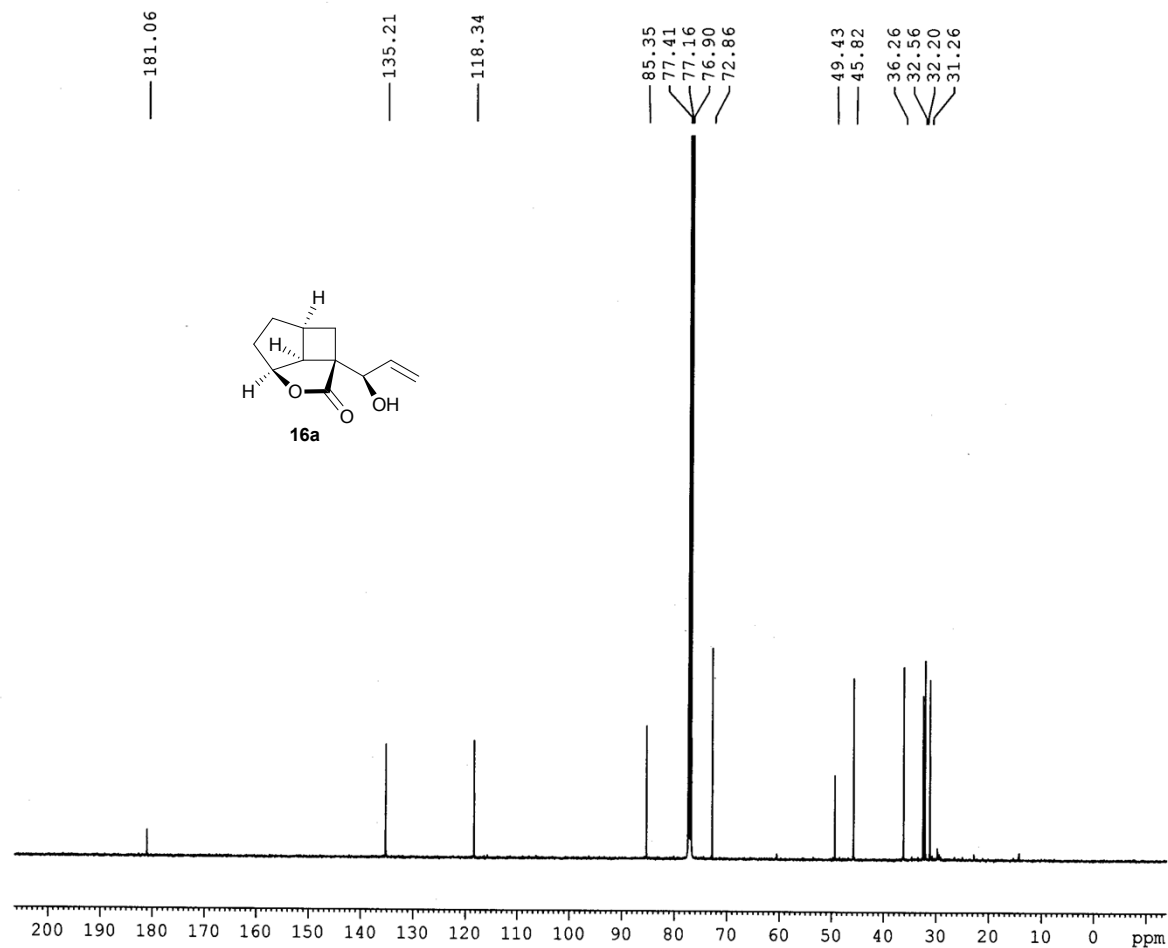
¹H NMR of Compound 16a (CDCl₃, 500 MHz)

SJM-S4AP6-1H (SR)



¹³C NMR of Compound 16a (CDCl₃, 125 MHz)

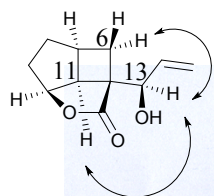
SJM-S4AP6-13C (SR)



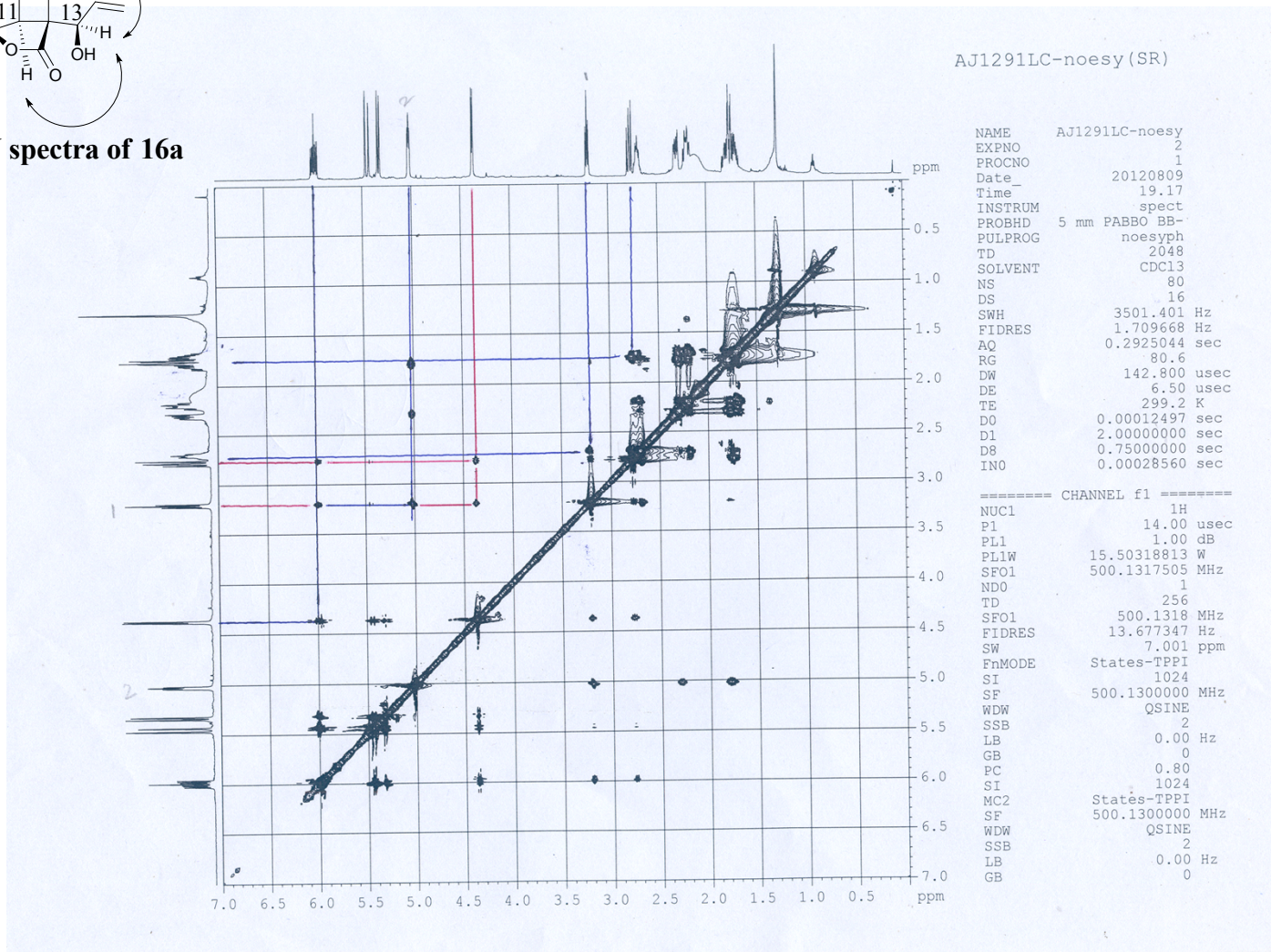
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NAME      SJM-S4AP6-13C
EXPNO     1
PROCNO    1
Date_     20110407
Time      11.18
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         22571
DS         4
SWH        29761.904 Hz
FIDRES     0.908261 Hz
AQ         0.5505524 sec
RG         32
DW          16.800 usec
DE          6.50 usec
TE         299.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
```

```
===== CHANNEL f1 =====
NUC1       13C
P1         9.88 usec
PL1        0.50 dB
PL1W       82.16106415 W
SFO1       125.7703643 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        1.00 dB
PL12       16.50 dB
PL13       20.00 dB
PL2W       15.50318813 W
PL12W      0.43693921 W
PL13W      0.19517358 W
SFO2       500.1320005 MHz
SI         16384
SF         125.7577693 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```



NOESY spectra of 16a



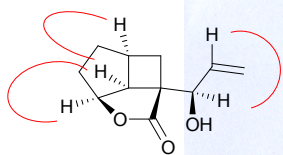
AJ1291LC-noesy (SR)

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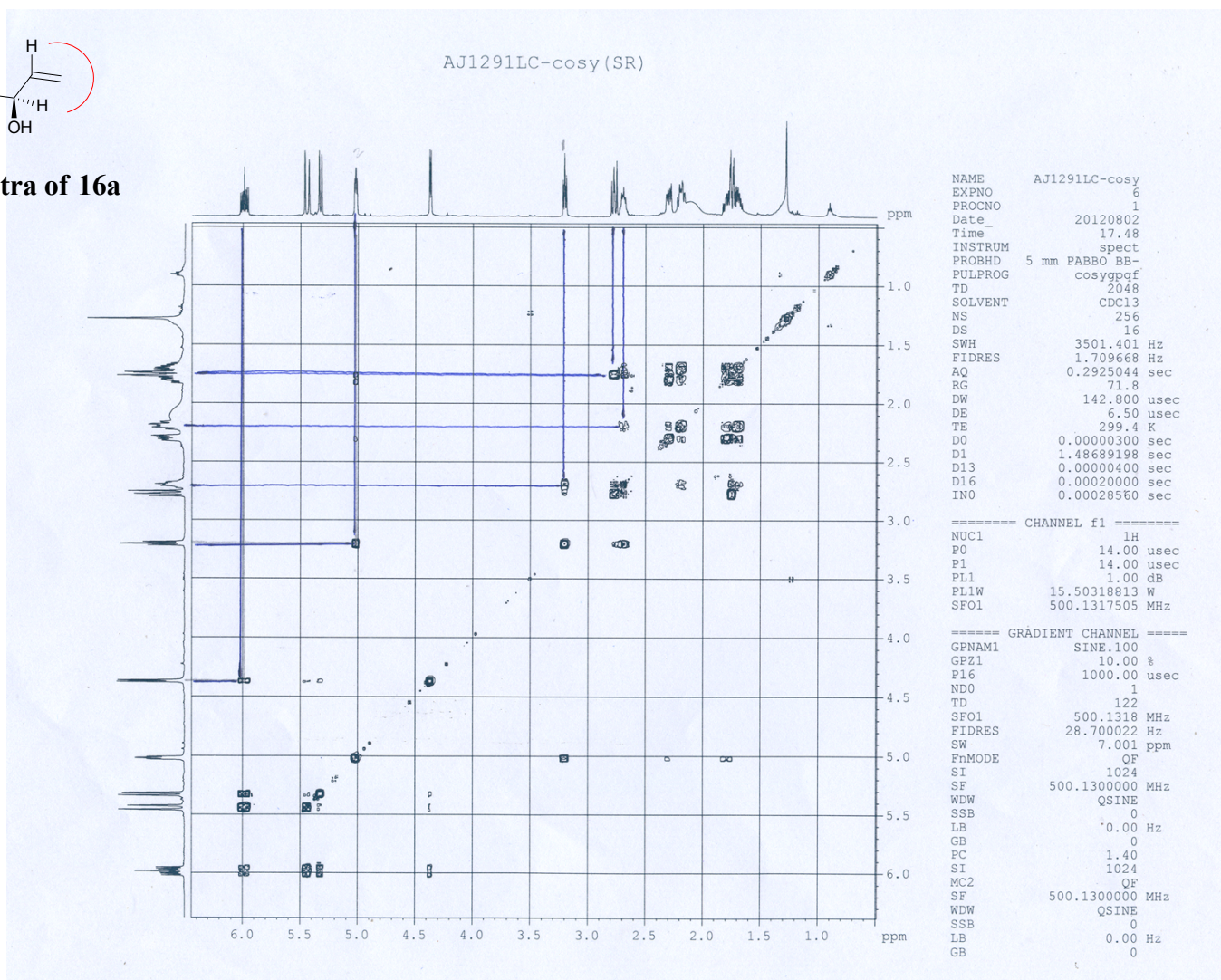
NAME      AJ1291LC-noesy
EXPNO     2
PROCNO    1
Date_     20120809
Time      19.17
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   noesyph
TD         2048
SOLVENT   CDC13
NS         80
DS         16
SWH        3501.401 Hz
FIDRES     1.709668 Hz
AQ         0.2925044 sec
RG         80.6
DW         142.800 usec
DE         6.50 usec
TE         299.2 K
D0         0.00012497 sec
D1         2.00000000 sec
D8         0.75000000 sec
IN0        0.00028560 sec
  
```

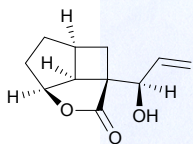
```

===== CHANNEL f1 =====
NUC1      1H
P1         14.00 usec
PL1        1.00 dB
PL1W      15.50318813 W
SFO1      500.1317505 MHz
ND0        1
TD         256
SFO1      500.1318 MHz
FIDRES     13.677347 Hz
SW         7.001 ppm
FnMODE     States-TPPI
SI         1024
SF         500.1300000 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         0.80
SI         1024
MC2        States-TPPI
SF         500.1300000 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
  
```

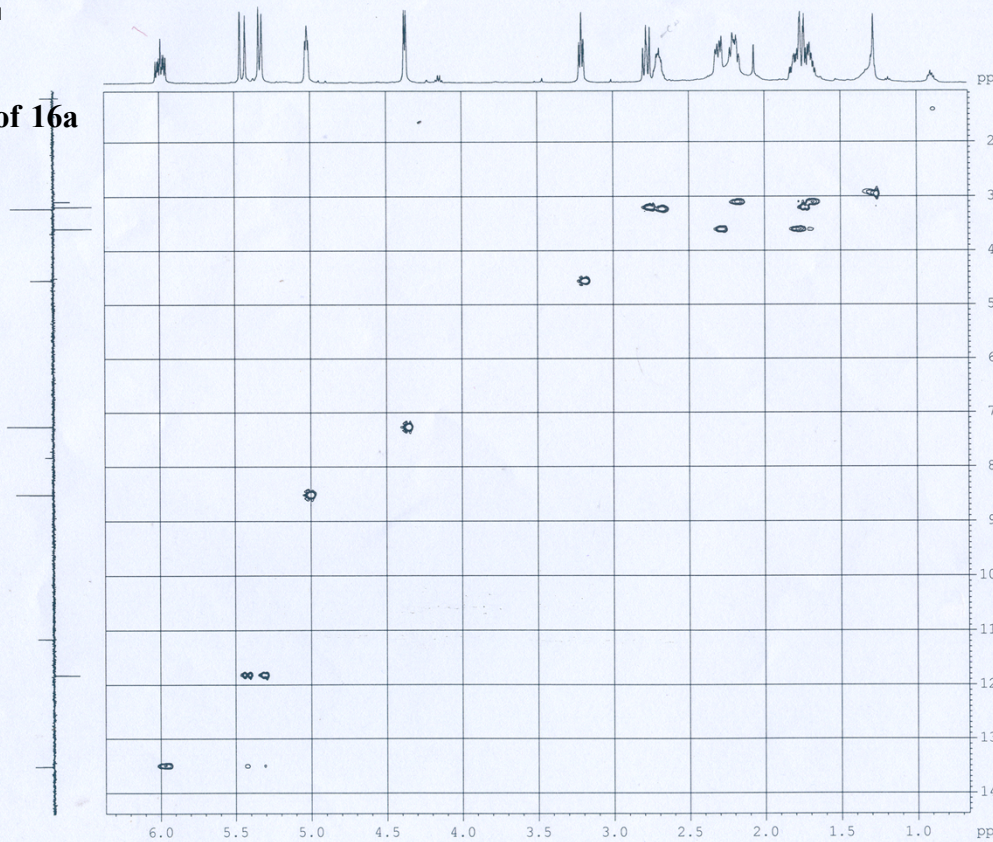


COSY spectra of 16a





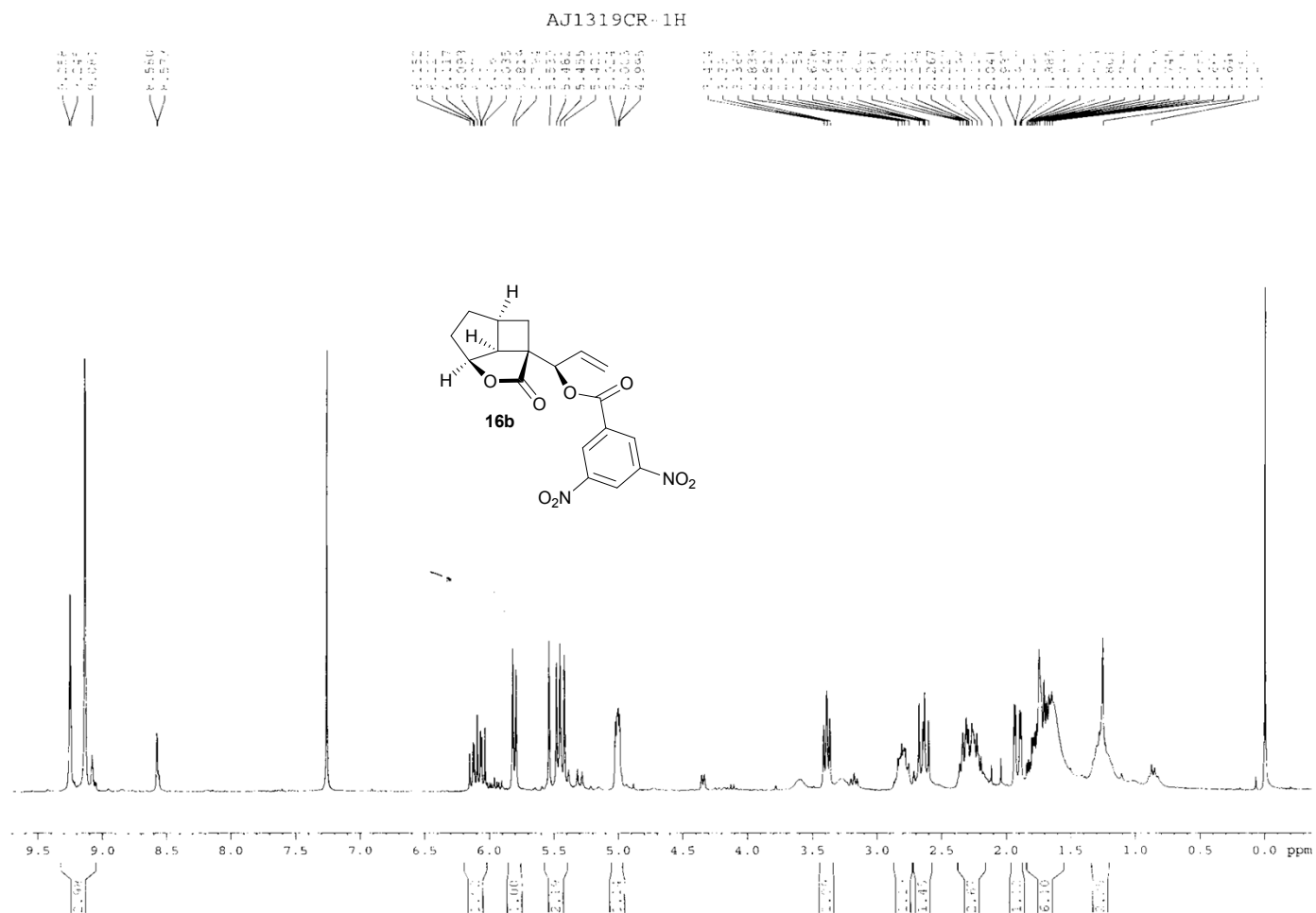
HSQC spectra of 16a



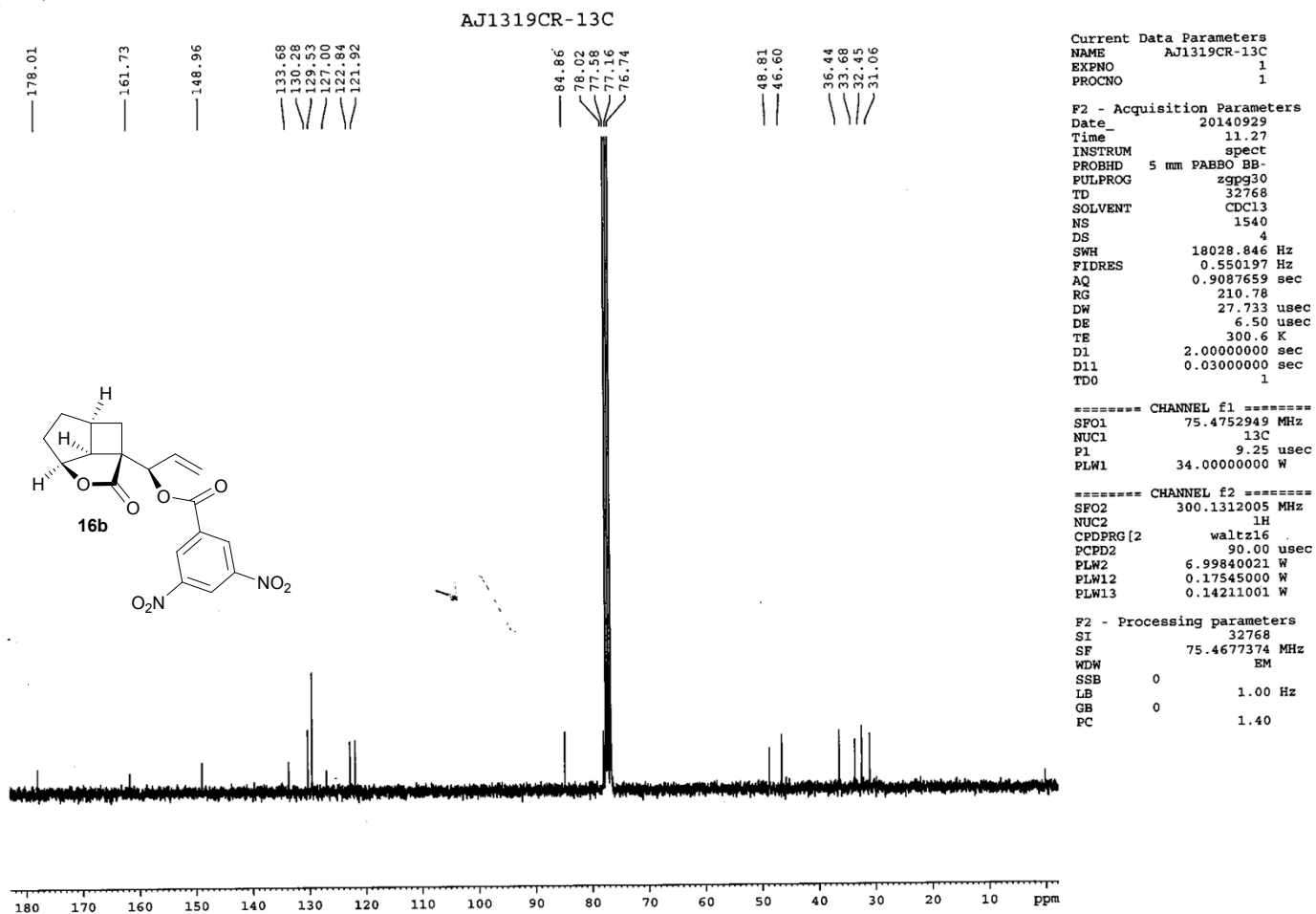
SJM-S4AP6-HSQC (SR)

NAME	SJM-S4AP6-HSQC
EXPNO	9
PROCNO	1
Date_	20120813
Time	19.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hsqcetgpsi2
TD	1024
SOLVENT	CDC13
NS	128
DS	16
SWH	3501.401 Hz
FIDRES	3.419337 Hz
AQ	0.1462772 sec
RG	57
DW	142.800 usec
DE	6.50 usec
TE	299.7 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.50000000 sec
D4	0.00172414 sec
D11	0.03000000 sec
D13	0.00000400 sec
D16	0.00020000 sec
D24	0.00086207 sec
INO	0.00002650 sec
ZGPTNS	
===== CHANNEL f1 =====	
NUC1	1H
P1	14.00 usec
F2	28.00 usec
F28	0.00 usec
PL1	1.00 dB
PL1W	15.50318813 W
SFO1	500.1317505 MHz
===== CHANNEL f2 =====	
CPDPRG2	garp4
NUC2	13C
P3	9.88 usec
P4	19.76 usec
ECPD2	70.00 usec
PL2	0.50 dB
PL12	17.50 dB
PL2W	82.16106415 W
PL12W	1.63932884 W
SFO2	125.7672208 MHz
===== GRADIENT CHANNEL =====	
GPNAME1	SINE.100
GPNAME2	SINE.100
GPNAME3	SINE.100
GPNAME4	SINE.100
GPZ1	80.10 %
GPZ2	20.10 %

¹H NMR of Compound 16b (CDCl₃, 300 MHz)

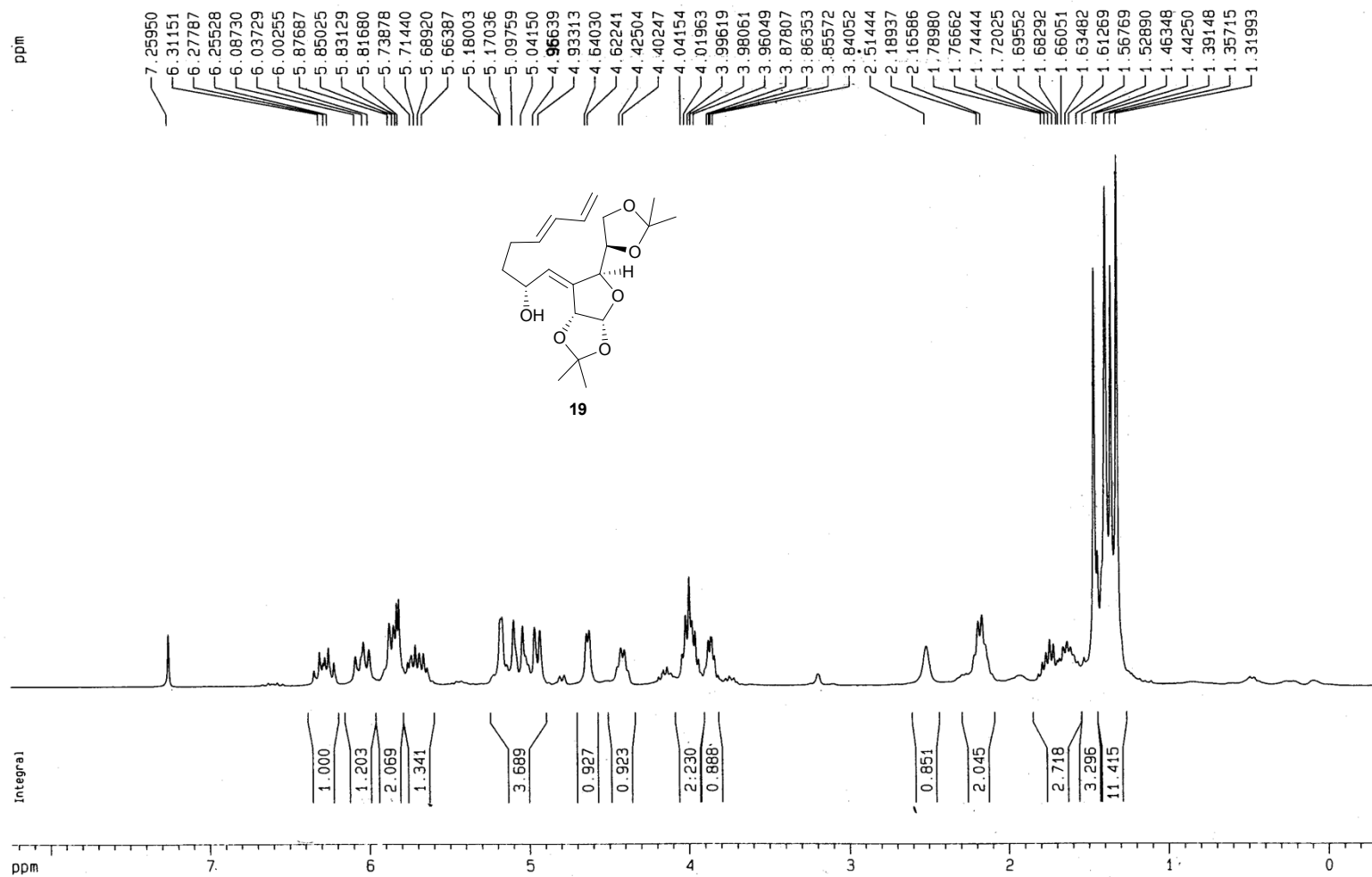


¹³C NMR of Compound 16b (CDCl₃, 75 MHz)



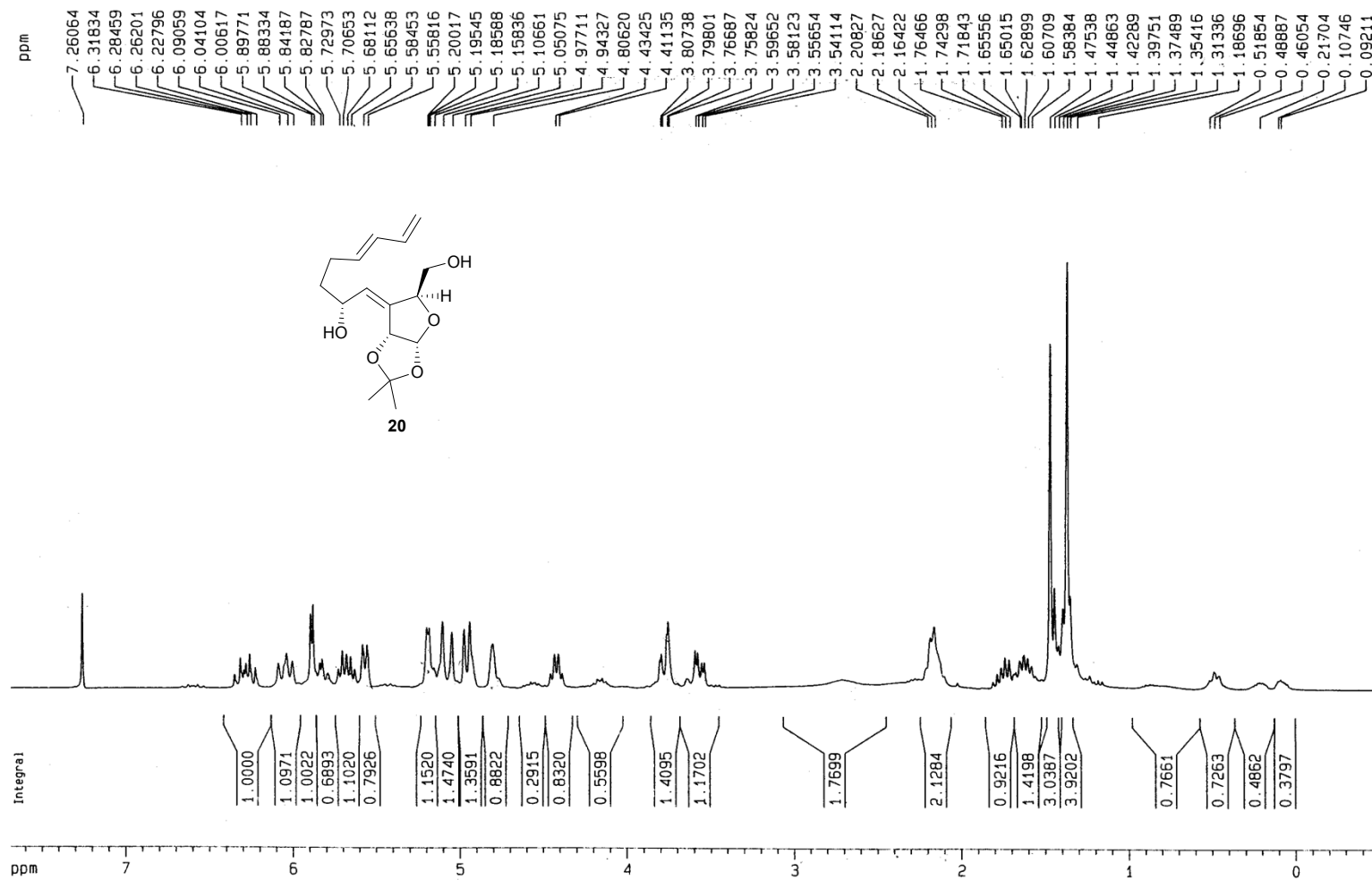
¹H NMR of Compound 19 (CDCl₃, 300 MHz)

AJ1211-1H (ND)

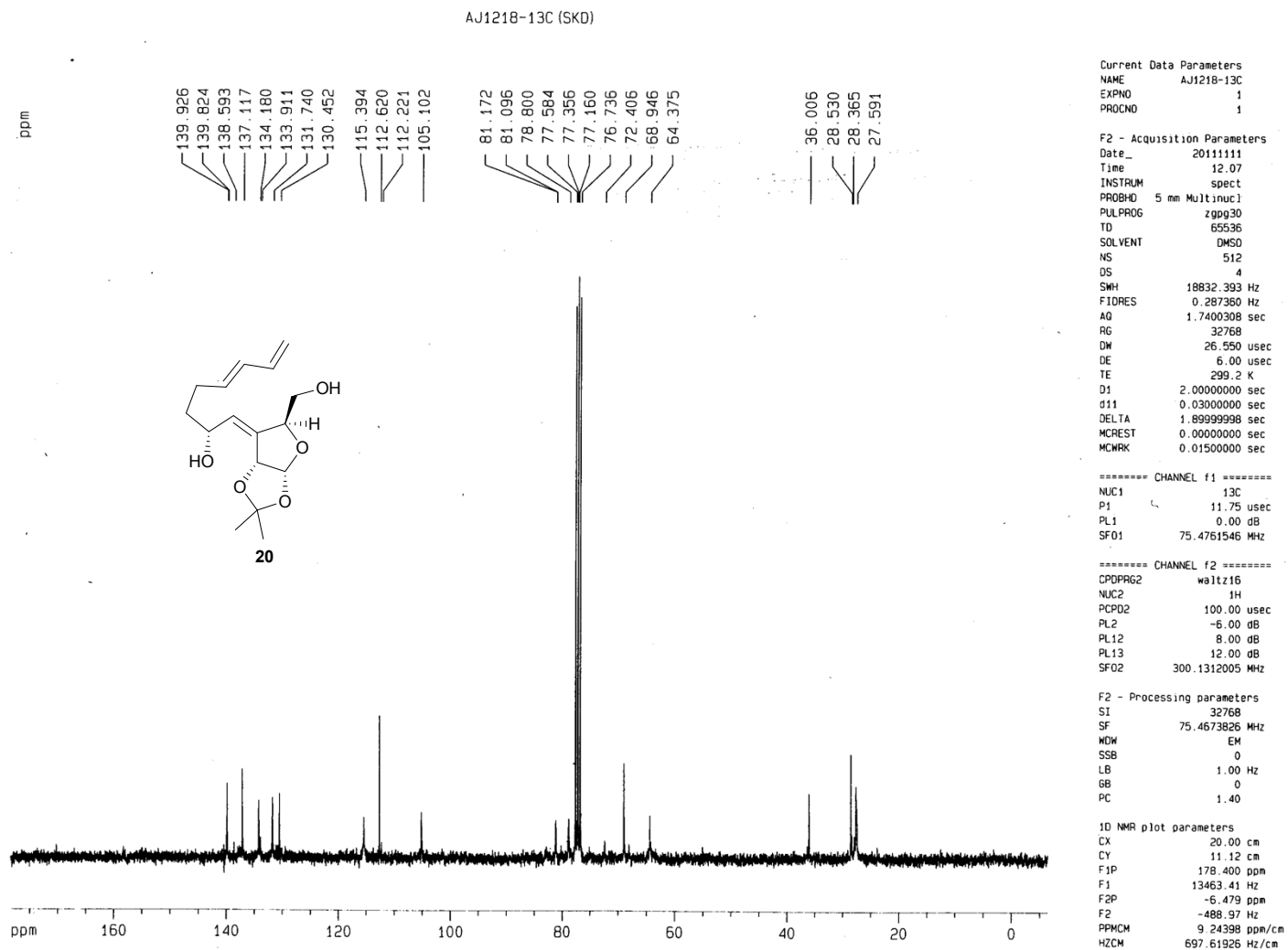


¹H NMR of Compound 20 (CDCl₃, 300 MHz)

AJ1218-1H (SKD)

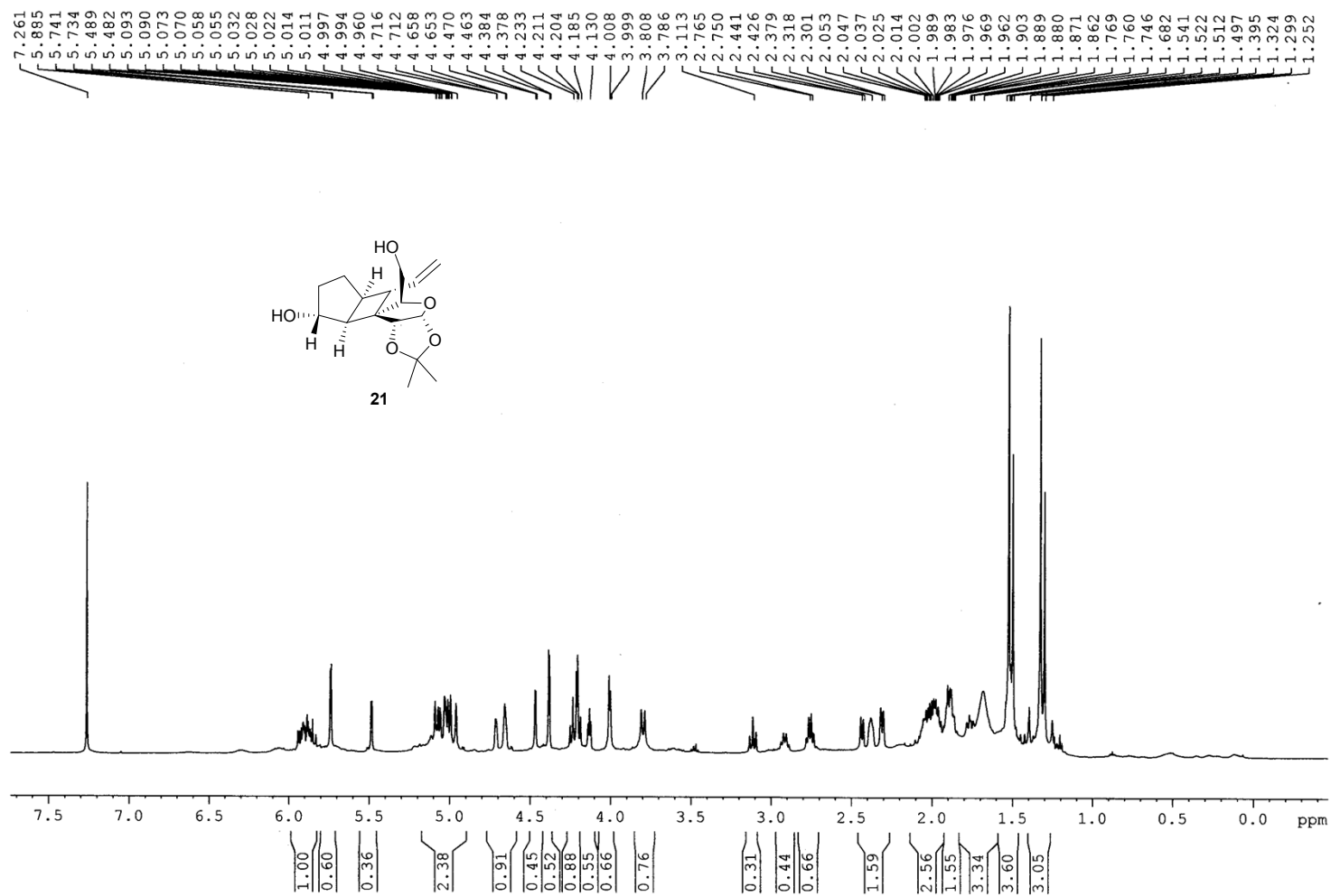


¹³C NMR of Compound 20 (CDCl₃, 75 MHz)



¹H NMR of Compound 21 (CDCl₃, 500 MHz)

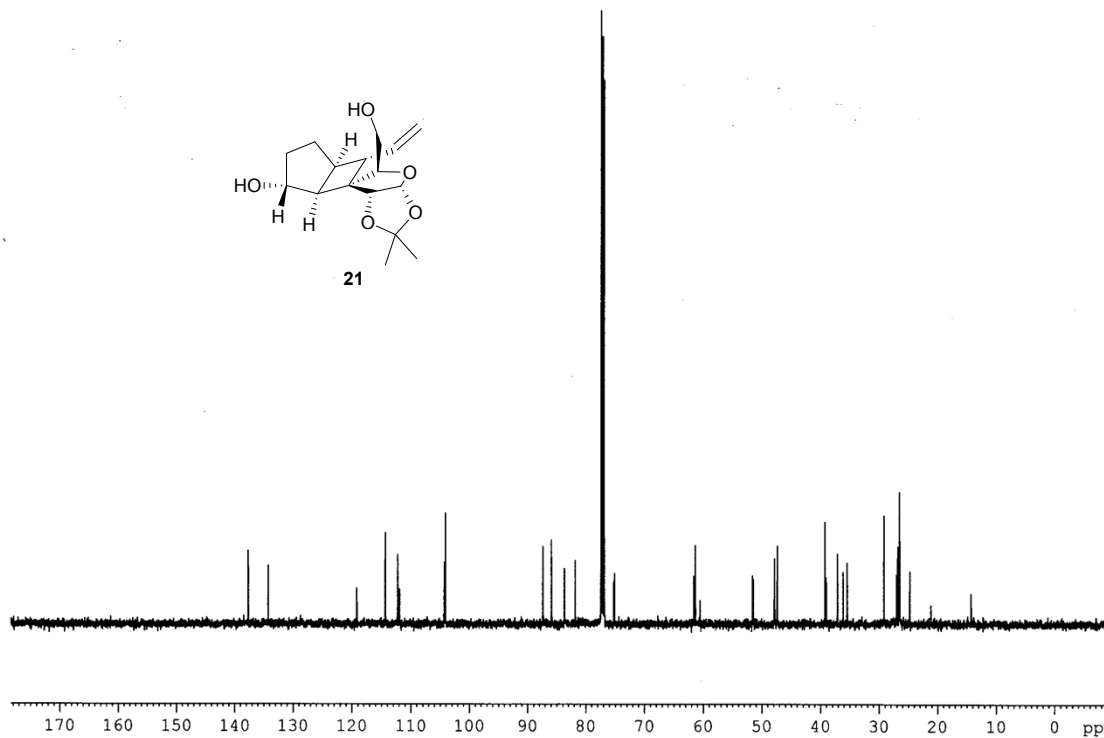
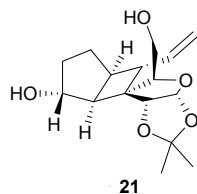
AJ-1172A-1H (SR)



¹³C NMR of Compound 21 (CDCl₃, 125 MHz)

AJ-1172-13C (SR)

—137.66
 —134.26
 —119.17
 —114.34
 —112.23
 —111.92
 —104.28
 —104.10
 87.38
 85.91
 83.70
 81.82
 77.42
 77.16
 76.91
 75.23
 75.14
 61.61
 61.34
 51.62
 51.50
 47.84
 47.39
 39.24
 39.03
 37.12
 36.16
 35.48
 29.17
 27.04
 26.84
 26.57
 26.50
 24.78
 14.31



```

NAME      AJ-1172-13C
EXPNO     1
PROCNO    1
Date_     20110712
Time      18.03
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        32768
SOLVENT   CDCl3
NS        568
DS        4
SWH       29761.904 Hz
FIDRES    0.908261 Hz
AQ        0.5505524 sec
RG        32
DW        16.800 usec
DE        6.50 usec
TE        298.7 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

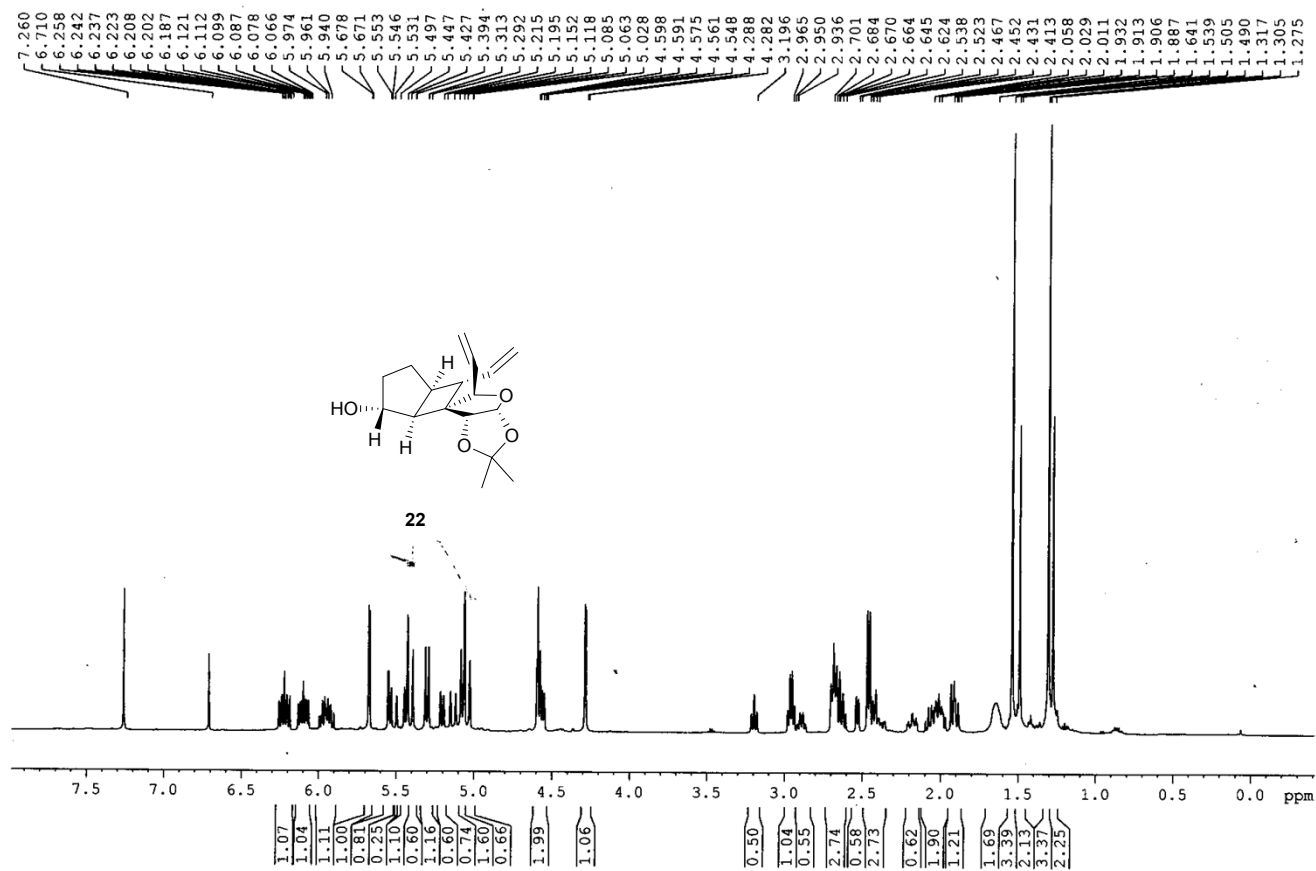
===== CHANNEL f1 =====
NUC1      13C
P1        9.88 usec
PL1       0.50 dB
PL1W     82.16106415 W
SF01     125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12     16.50 dB
PL13     20.00 dB
PL2W     15.50318813 W
PL12W    0.43693921 W
PL13W    0.19517358 W
SFO2     500.1320005 MHz
SI        16384
SF        125.7577720 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.00
  
```

¹H NMR of Compound 22 (CDCl₃, 500 MHz)

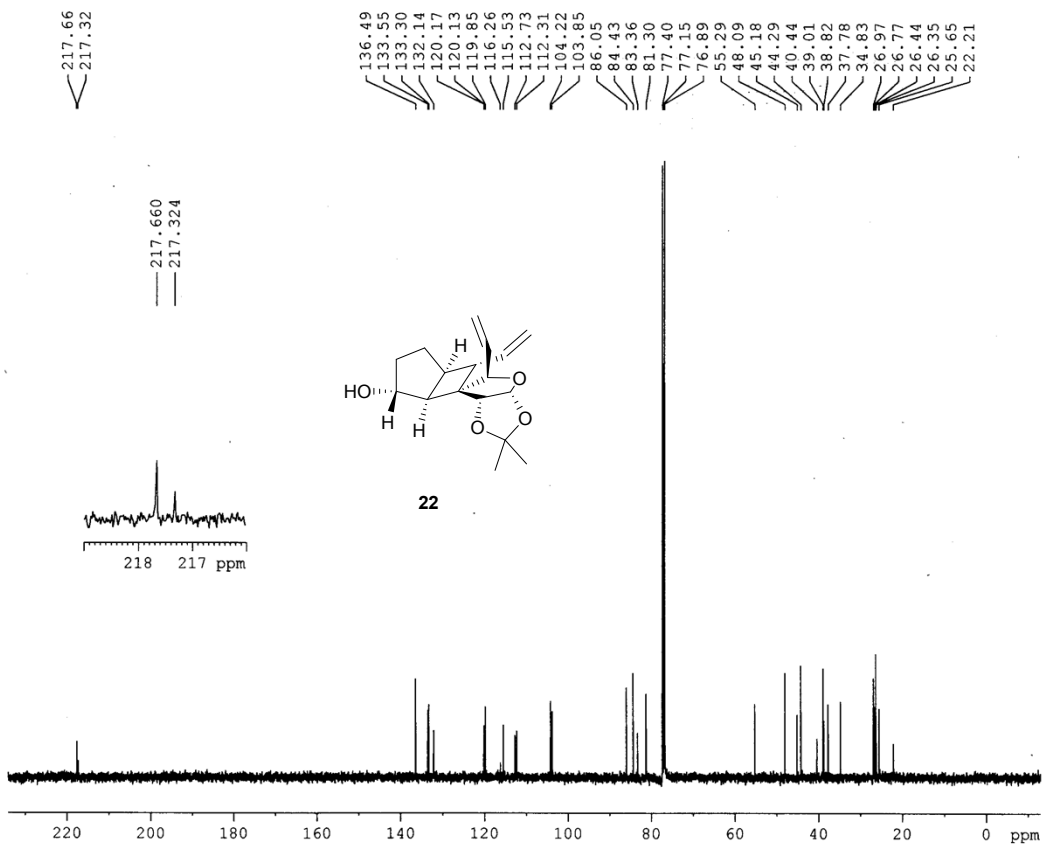
AJ1261-1H (SR)



0.92 2.06

¹³C NMR of Compound 22 (CDCl₃, 125 MHz)

AJ1260-13C (SR)



```

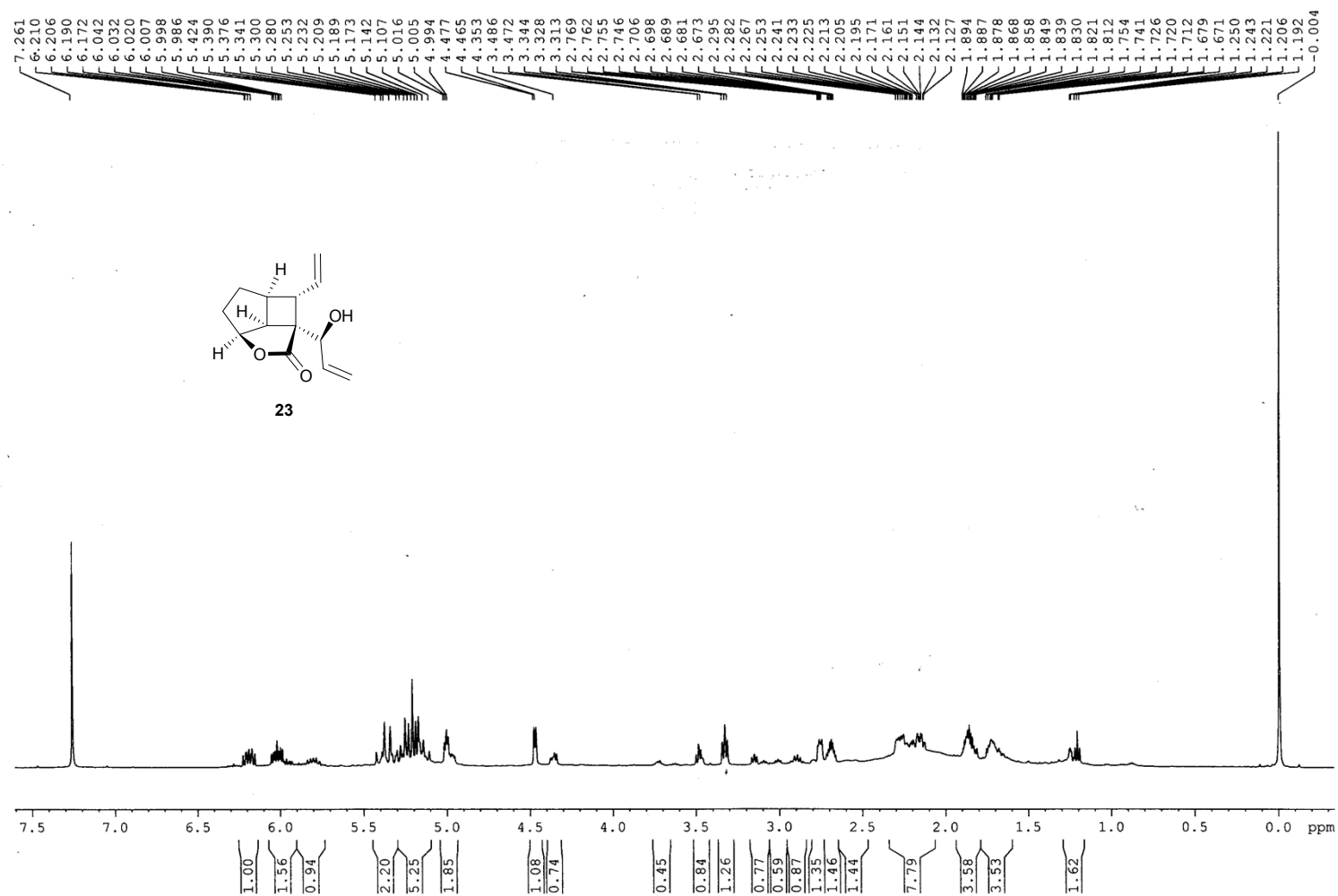
NAME          AJ1260-13C
EXPNO         2
PROCNO        1
Date_         20120411
Time          17.58
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            512
DS            4
SWH           36231.883 Hz
FIDRES        1.105709 Hz
AQ            0.4522484 sec
RG            32
DW            13.800 usec
DE            6.50 usec
TE            299.4 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           13C
P1             9.88 usec
PL1            0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2            1.00 dB
PL12           16.50 dB
PL13           20.00 dB
PL2W          15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2           500.1320005 MHz
SI             16384
SF            125.7577724 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.00
    
```

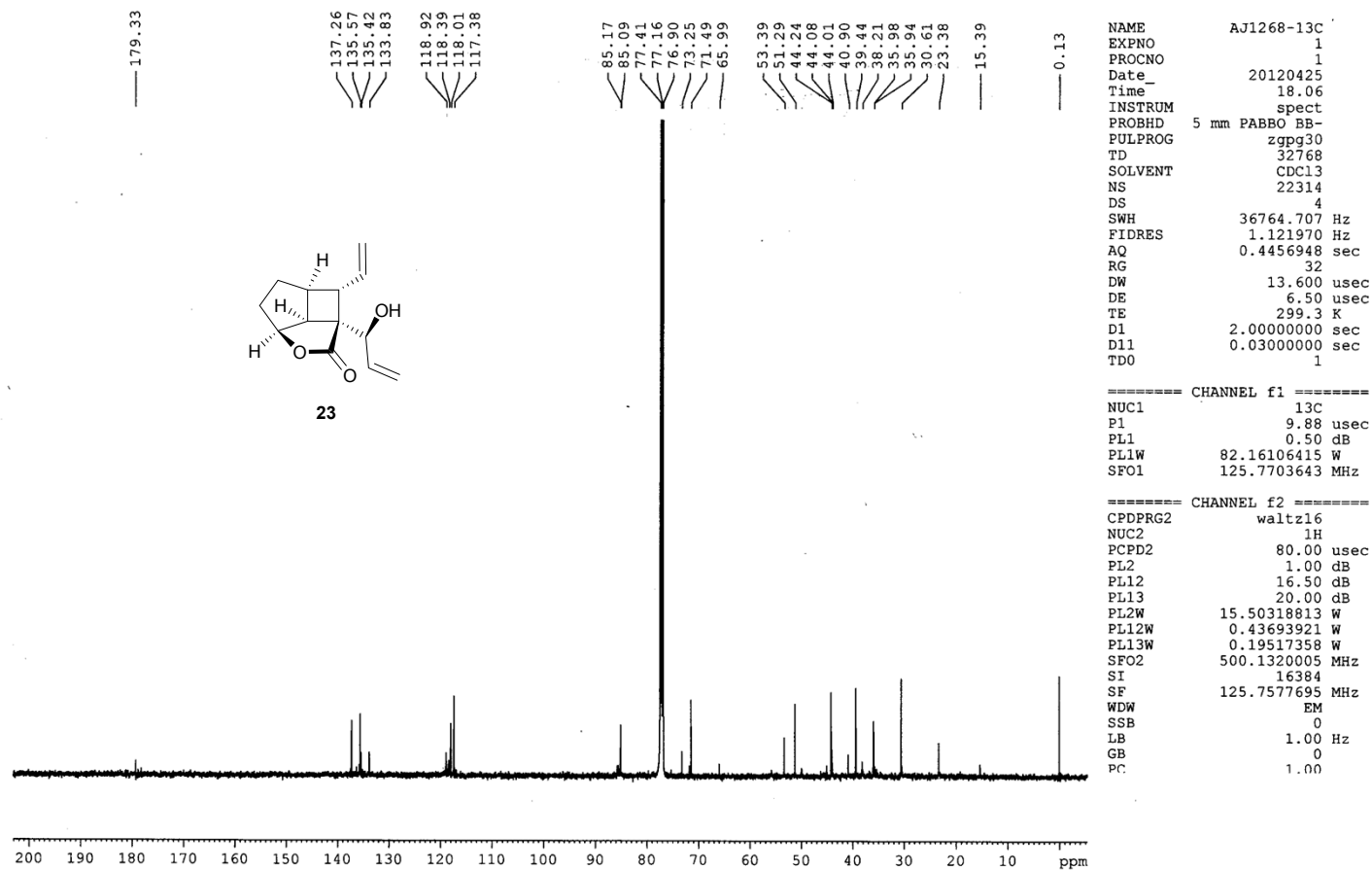
¹H NMR of Compound 23 (CDCl₃, 300 MHz)

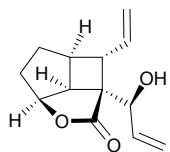
AJ1268-1H (SKD)



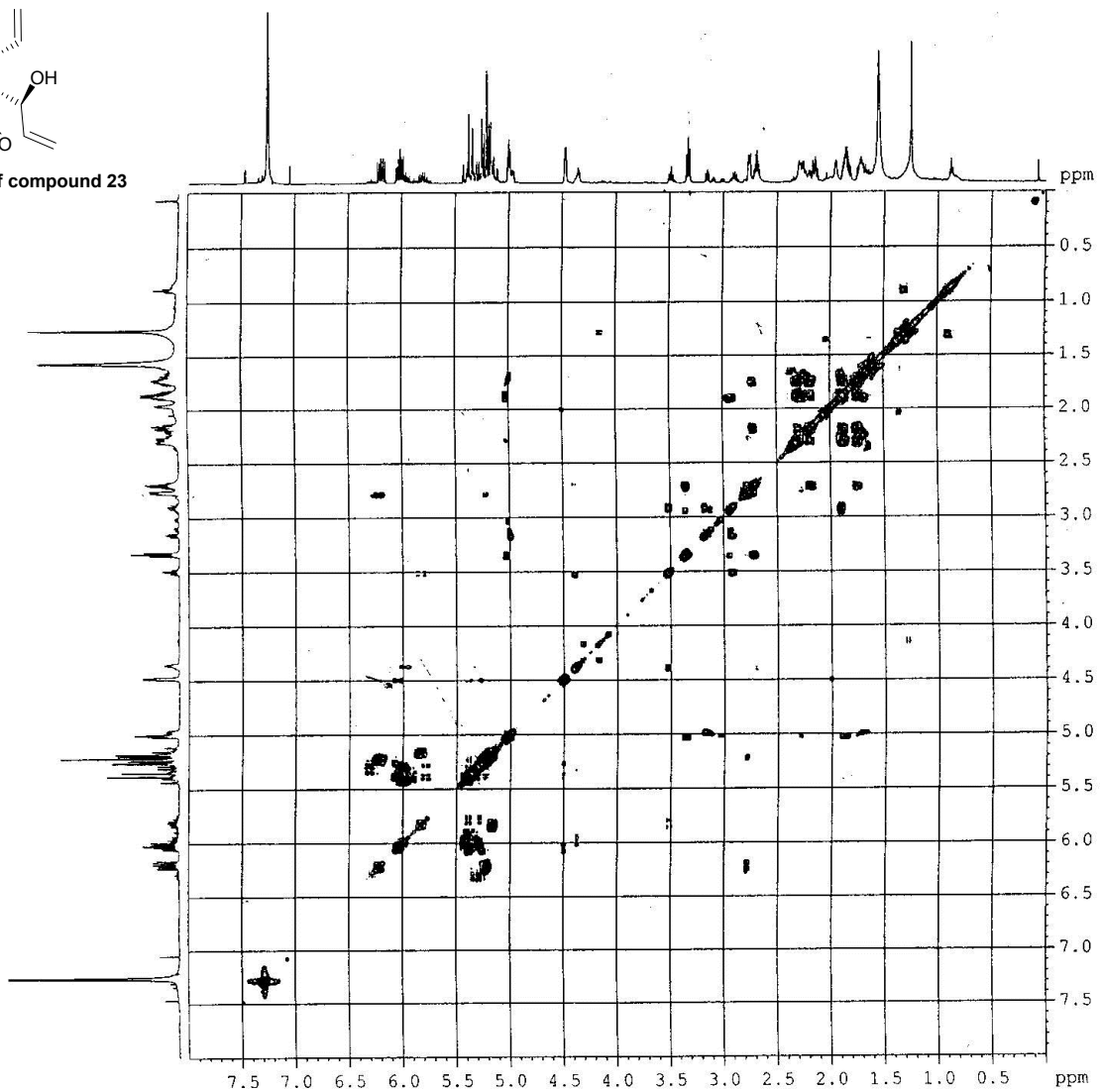
¹³C NMR of Compound 23 (CDCl₃, 125 MHz)

AJ1268-13C (SR)





COSY spectra of compound 23



AJ1268D-cosy (SR)

```

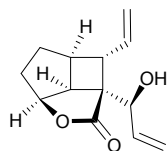
NAME      AJ1268D-cosy
EXPNO     2
PROCNO    1
Date_     20120524
Time      18.40
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   cosygpgf
TD         2048
SOLVENT   CDCl3
NS         256
DS         16
SWH       4000.000 Hz
FIDRES    1.953125 Hz
AQ         0.2560500 sec
RG         71.8
DW         125.000 usec
DE         6.50 usec
TE         300.0 K
DO         0.00000300 sec
D1         1.48689198 sec
D13        0.00000400 sec
D16        0.00020000 sec
IN0        0.00025000 sec
  
```

```

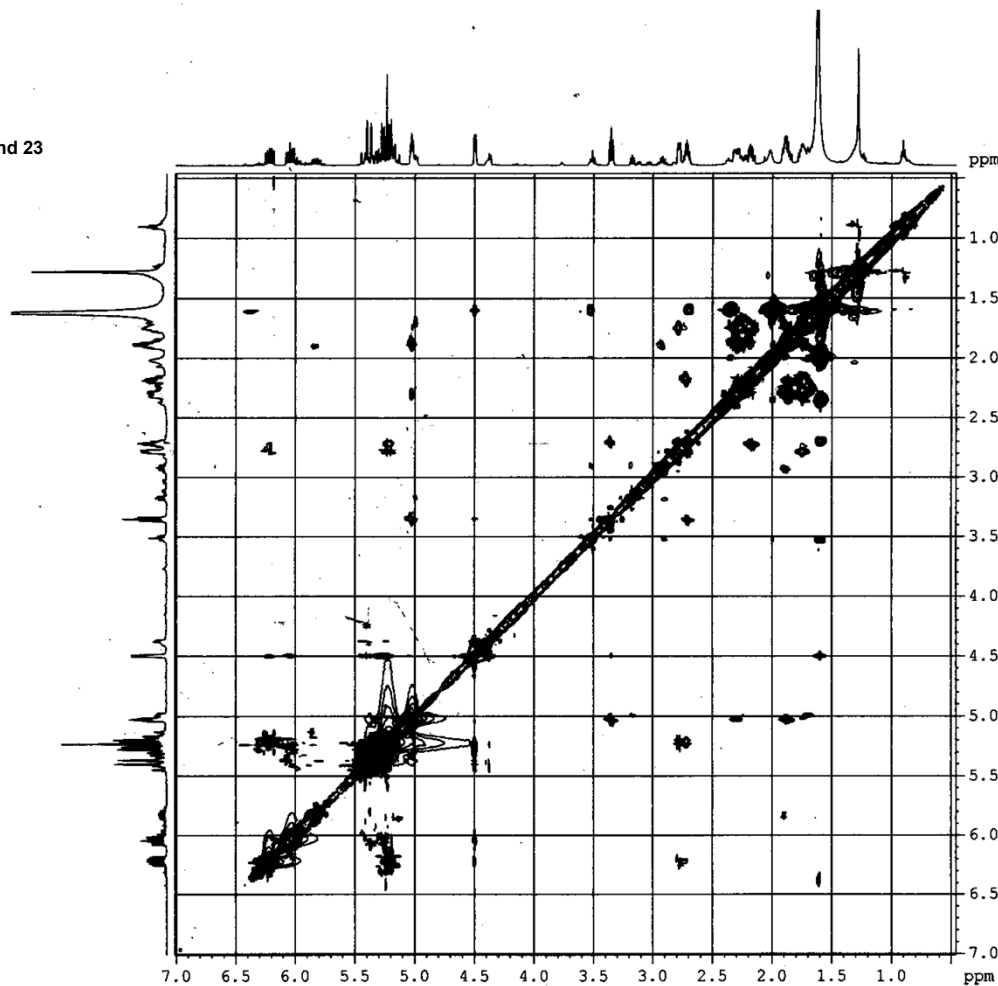
===== CHANNEL f1 =====
NUC1      1H
P0        14.00 usec
P1        14.00 usec
PL1       1.00 dB
PL1W      15.50318813 W
SFO1      500.1320005 MHz
  
```

```

===== GRADIENT CHANNEL =====
GPNAM1    SINE.100
GP21      10.00 %
P16       1000.00 usec
NDO        1
TD         122
SFO1      500.132 MHz
FIDRES    32.786919 Hz
SW         7.998 ppm
FrMODE    QF
SI         1024
SF         500.1300000 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40
SI         1024
MC2        QF
SF         500.1300000 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
  
```



NOESY spectra of compound 23



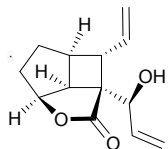
AJ1268D-noesy (SR)

```

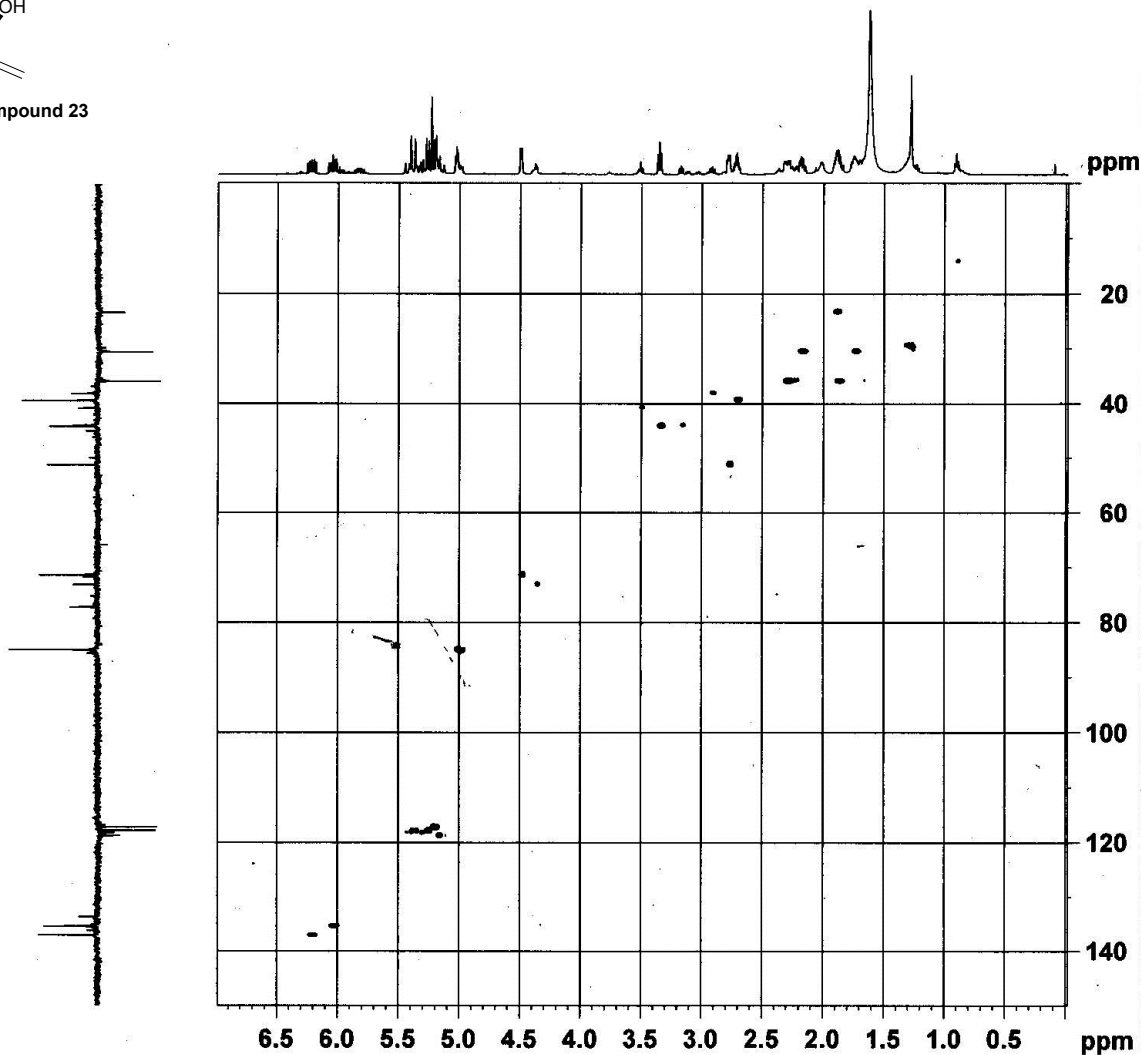
NAME      AJ1268D-noesy
EXPNO     2
PROCNO    1
Date_     20120521
Time_     19.06
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   noesyph
TD         2048
SOLVENT   CDC13
NS         80
DS         16
SWH        4000.000 Hz
FIDRES     1.953125 Hz
AQ         0.2560500 sec
RG         203
DW         125.000 usec
DE         6.50 usec
TE         299.6 K
D0         0.00010717 sec
D1         2.00000000 sec
D8         0.75000000 sec
IN0        0.00025000 sec
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         14.00 usec
PL1        1.00 dB
PL1W       15.50318813 W
SFO1       500.1320005 MHz
ND0         1
TD         213
SFO1       500.132 MHz
FIDRES     18.779352 Hz
SW         7.998 ppm
FnMODE     States-TPPI
SI         1024
SF         500.1300000 MHz
WDW        QSINE
SSB         2
LB         0.00 Hz
GB         0
PC         0.80
SI         1024
MC2        States-TPPI
SF         500.1300000 MHz
WDW        QSINE
SSB         2
LB         0.00 Hz
GB         0
  
```



HSQC spectra of compound 23



AJ1268D-HSQC (SR)

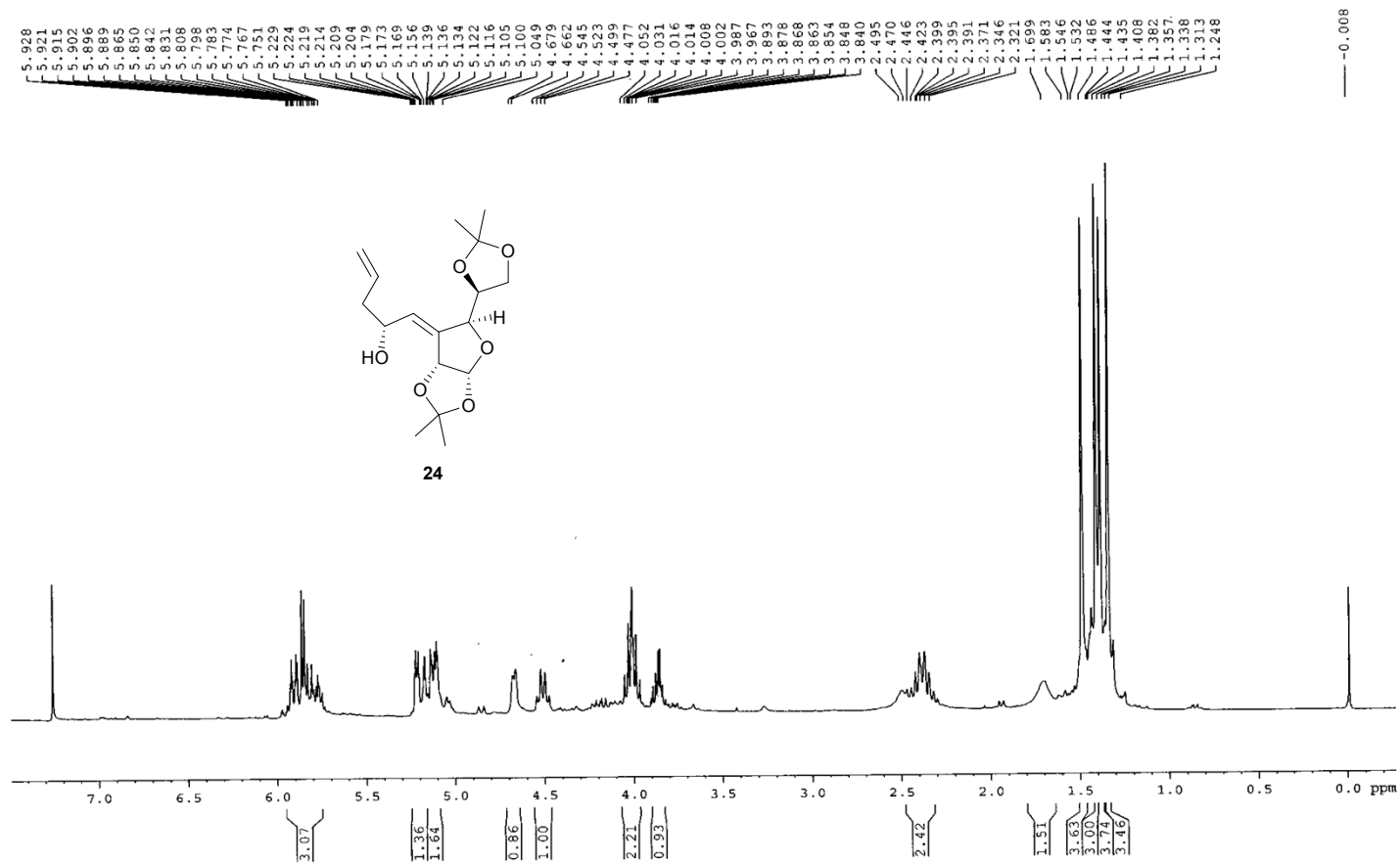
```

NAME      AJ1268D-HSQC
EXPNO     1
PROCNO    1
Date_     20120613
Time      19.16
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   hsqcetgps12
TD         1024
SOLVENT   CDCl3
NS         128
DS         16
SWH        4000.000 Hz
FIDRES     3.906250 Hz
AQ         0.1280500 sec
RG         80.6
DW         125.000 usec
DE         6.50 usec
TE         299.4 K
CNST2     145.0000000
D0         0.0000000 sec
D1         1.5000000 sec
D4         0.00172414 sec
D11        0.03000000 sec
D13        0.00000400 sec
D16        0.00020000 sec
D24        0.00086207 sec
IN0        0.00002650 sec
===== CHANNEL f1 =====
NUC1       1H
P1         14.00 usec
P2         28.00 usec
P28        0.00 usec
PL1        1.00 dB
PL1W       15.5031813 W
SFO1       500.1320005 MHz
===== CHANNEL f2 =====
CPDPRG2    garp4
NUC2       13C
P3         9.88 usec
P4         19.76 usec
PCPD2     70.00 usec
PL2        0.50 dB
PL12       17.50 dB
PL2W       82.16106415 W
PL12W      1.63932884 W
SFO2       125.7672208 MHz
===== GRADIENT CHANNEL =====
GPNAM1     SINE.100
GPNAM2     SINE.100
GPNAM3     SINE.100
GPNAM4     SINE.100
GPZ1       80.00 %
GPZ2       20.10 %
GPZ3       11.00 %
GPZ4       -5.00 %
P16        1000.00 usec
P19        600.00 usec
NDO        2
TD         256
SFO1       125.7672 MHz
FIDRES     73.691734 Hz
SW         150.000 ppm
FrMODE     Echo-Antiecho
SI         1024
SF         500.1300093 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.40
SI         1024
MC2        echo-antiecho
SF         125.7577890 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0

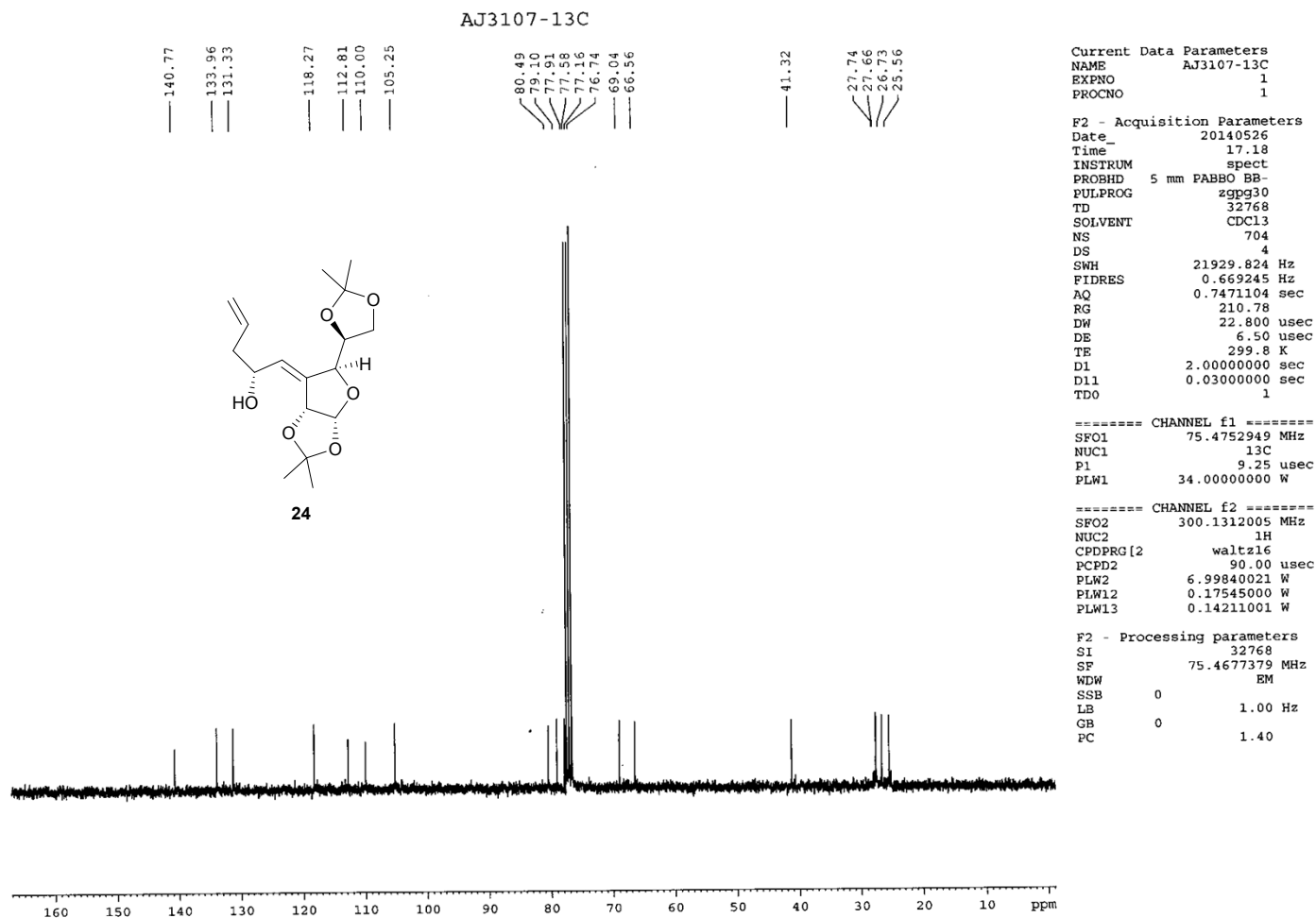
```


¹H NMR of Compound 24 (CDCl₃, 300 MHz)

AJ3107-1H

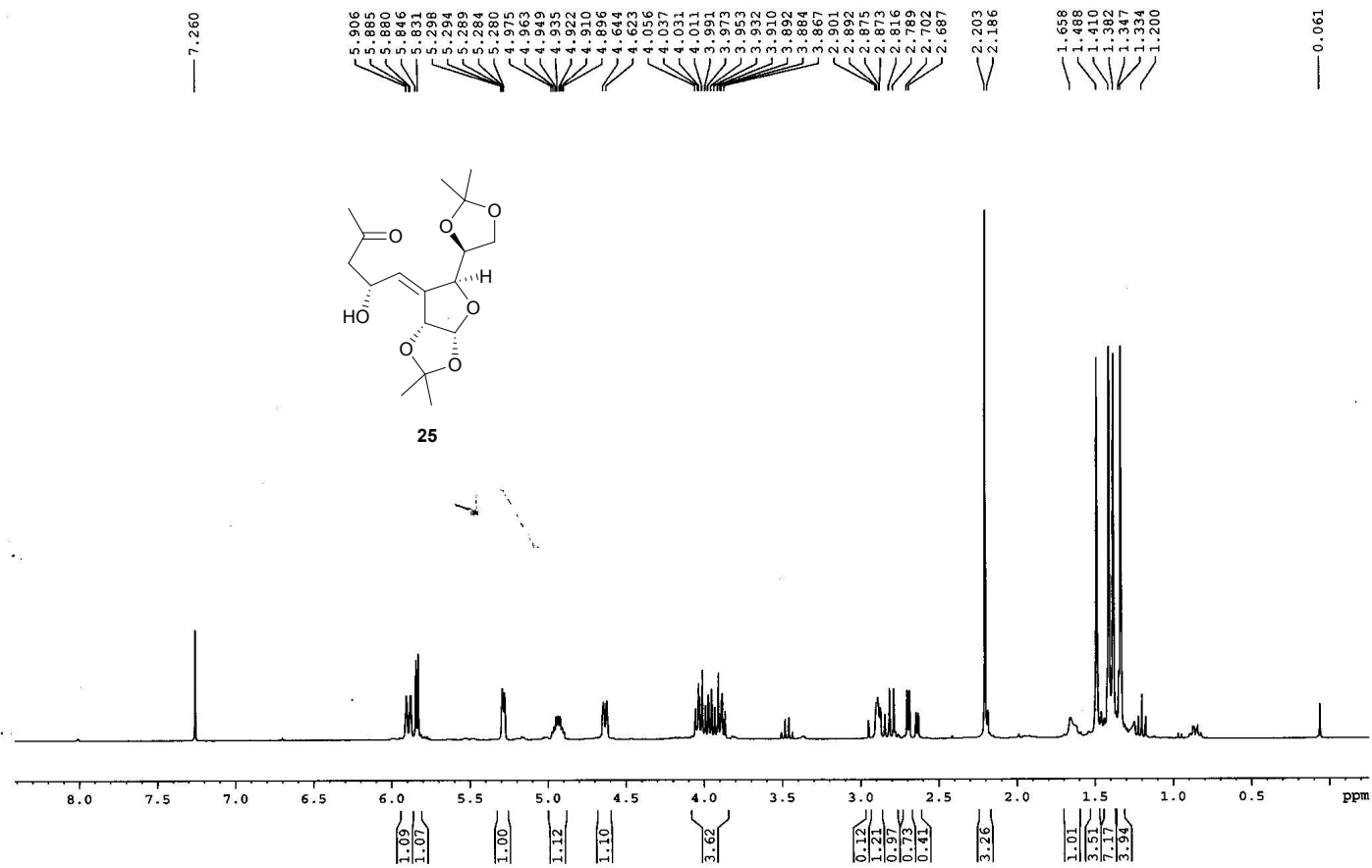


¹³C NMR of Compound 24 (CDCl₃, 75 MHz)



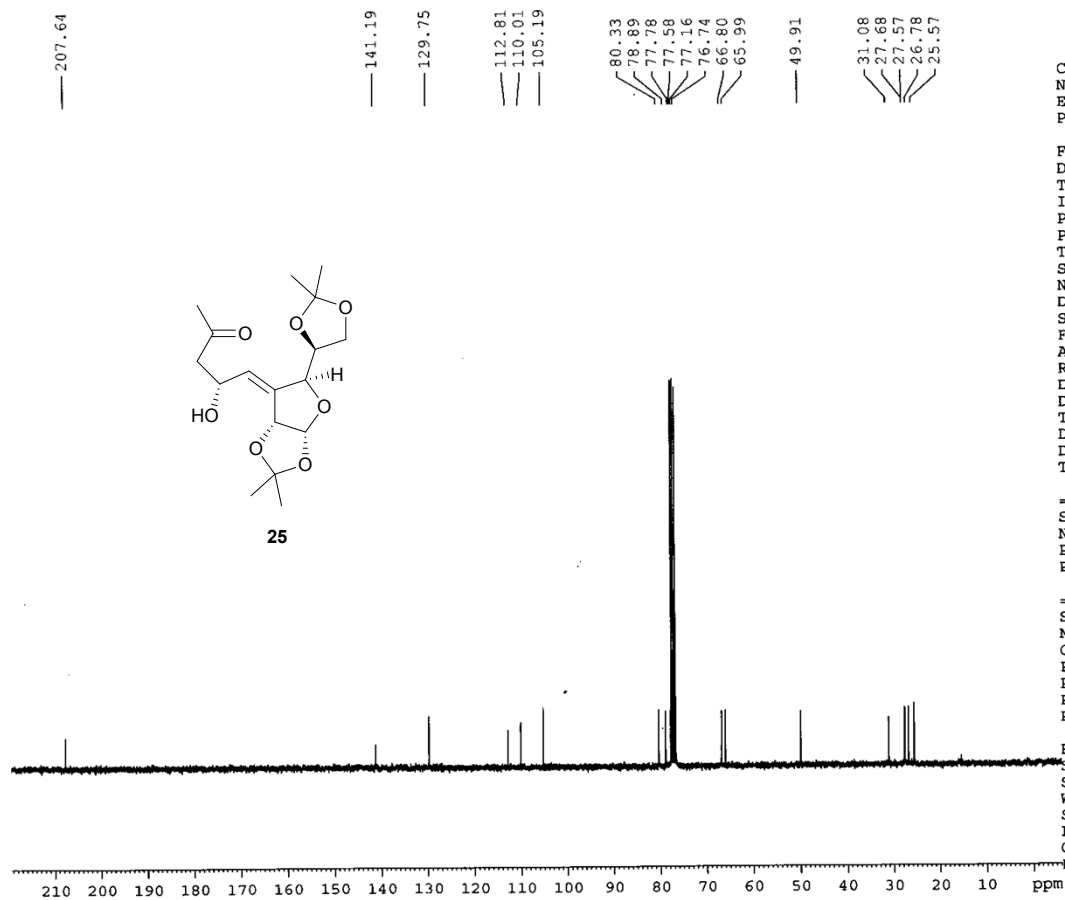
¹H NMR of Compound 25 (CDCl₃, 300 MHz)

AJ1344-1H (ND)



¹³C NMR of Compound 25 (CDCl₃, 75 MHz)

AJ1344-13C (ND)



Current Data Parameters
 NAME AJ1344-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121206
 Time_ 14.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 928
 DS 4
 SWH 18028.846 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 210.78
 DW 27.733 usec
 DE 6.50 usec
 TE 298.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

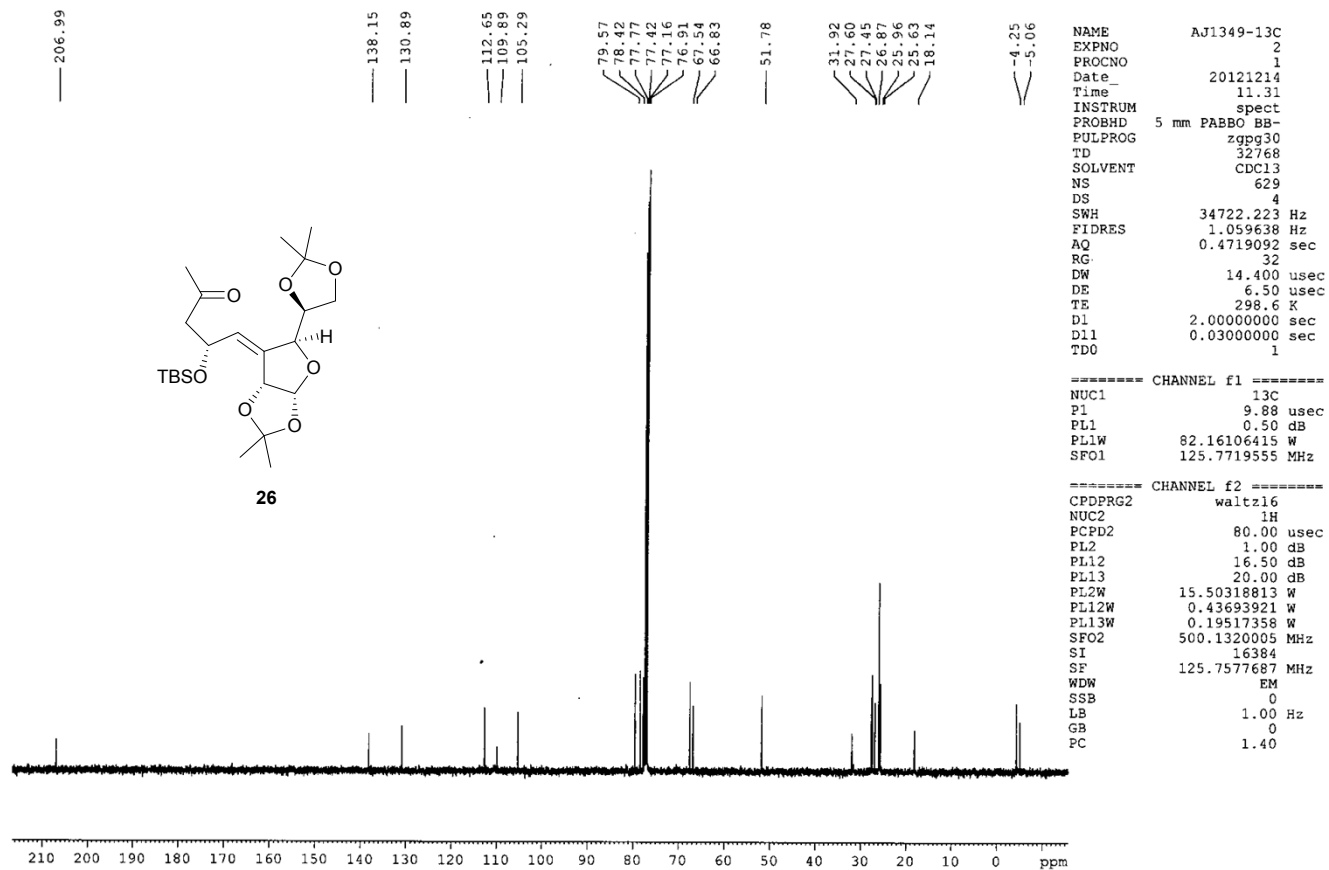
===== CHANNEL f1 =====
 SFO1 75.4752949 MHz
 NUC1 13C
 P1 9.25 usec
 PLW1 34.00000000 W

===== CHANNEL f2 =====
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 6.99840021 W
 PLW12 0.17545000 W
 PLW13 0.14211001 W

F2 - Processing parameters
 SI 32768
 SF 75.4677378 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

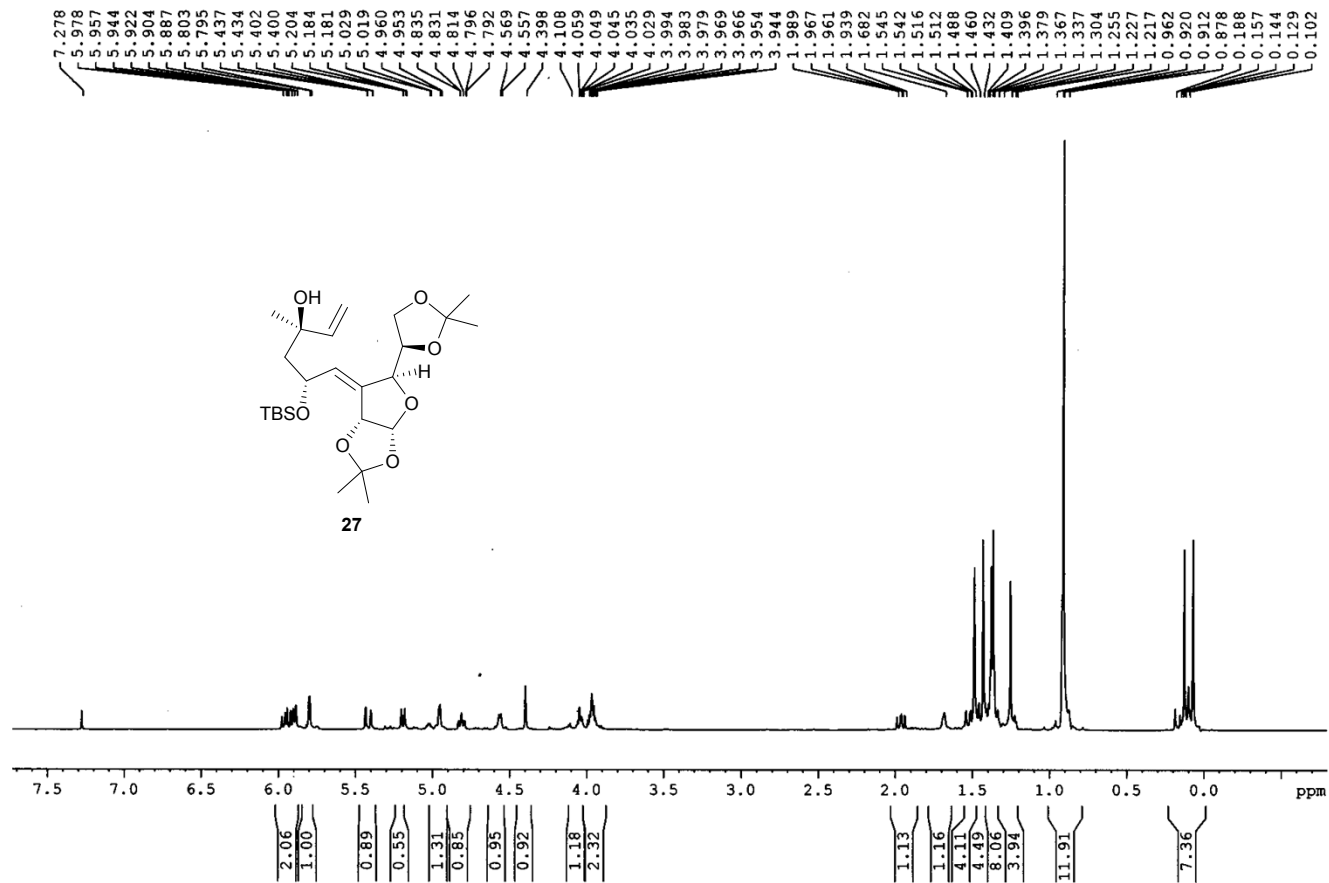
¹³C NMR of Compound 26 (CDCl₃, 125 MHz)

AJ1349-13C (SKD)

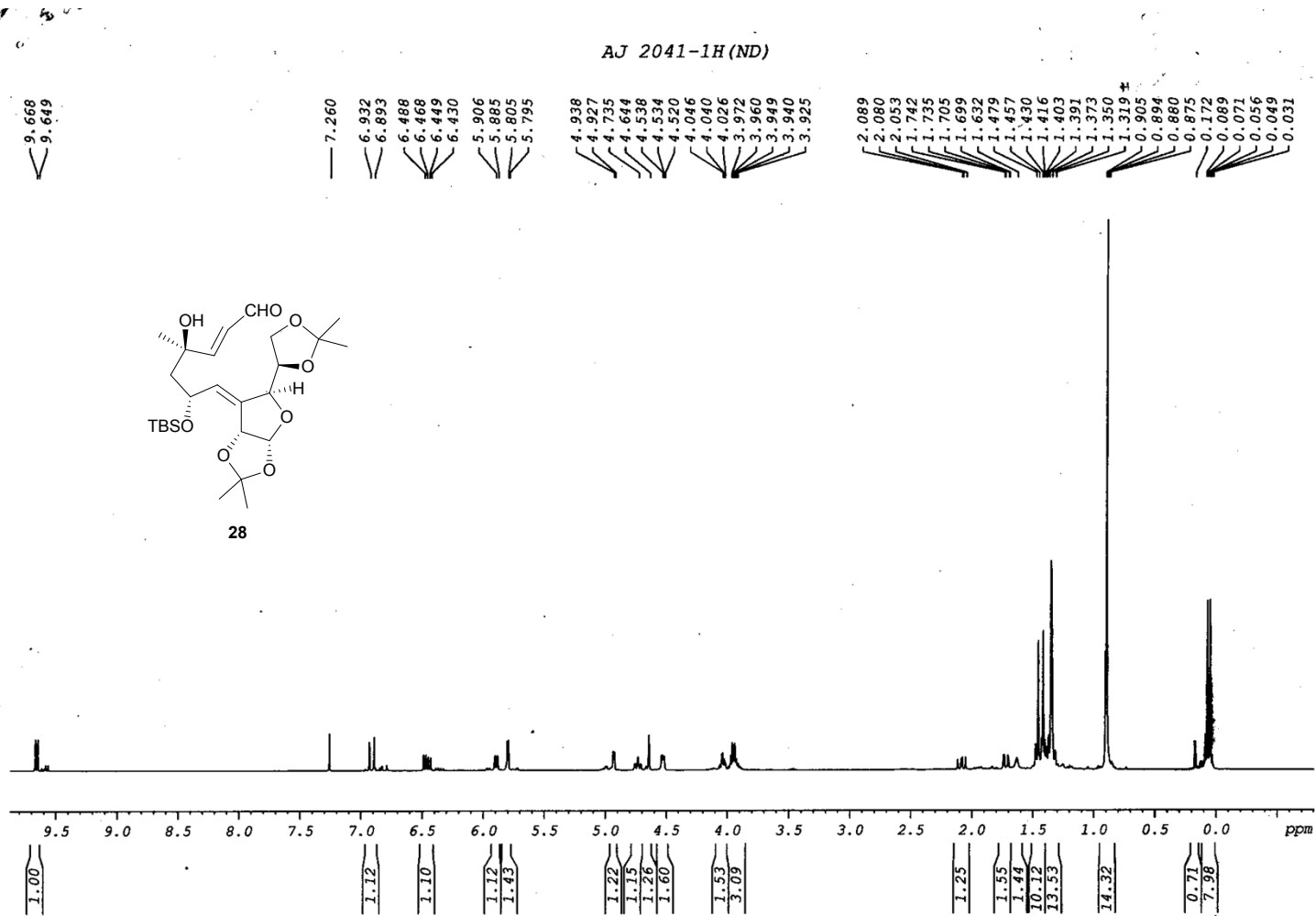


¹H NMR of Compound 27 (CDCl₃, 300 MHz)

AJ1351-1H(SR)



¹H NMR of Compound 28 (CDCl₃, 400 MHz)



¹³C NMR of Compound 28 (CDCl₃, 75 MHz)

AJ-3087 300MHZ-13C

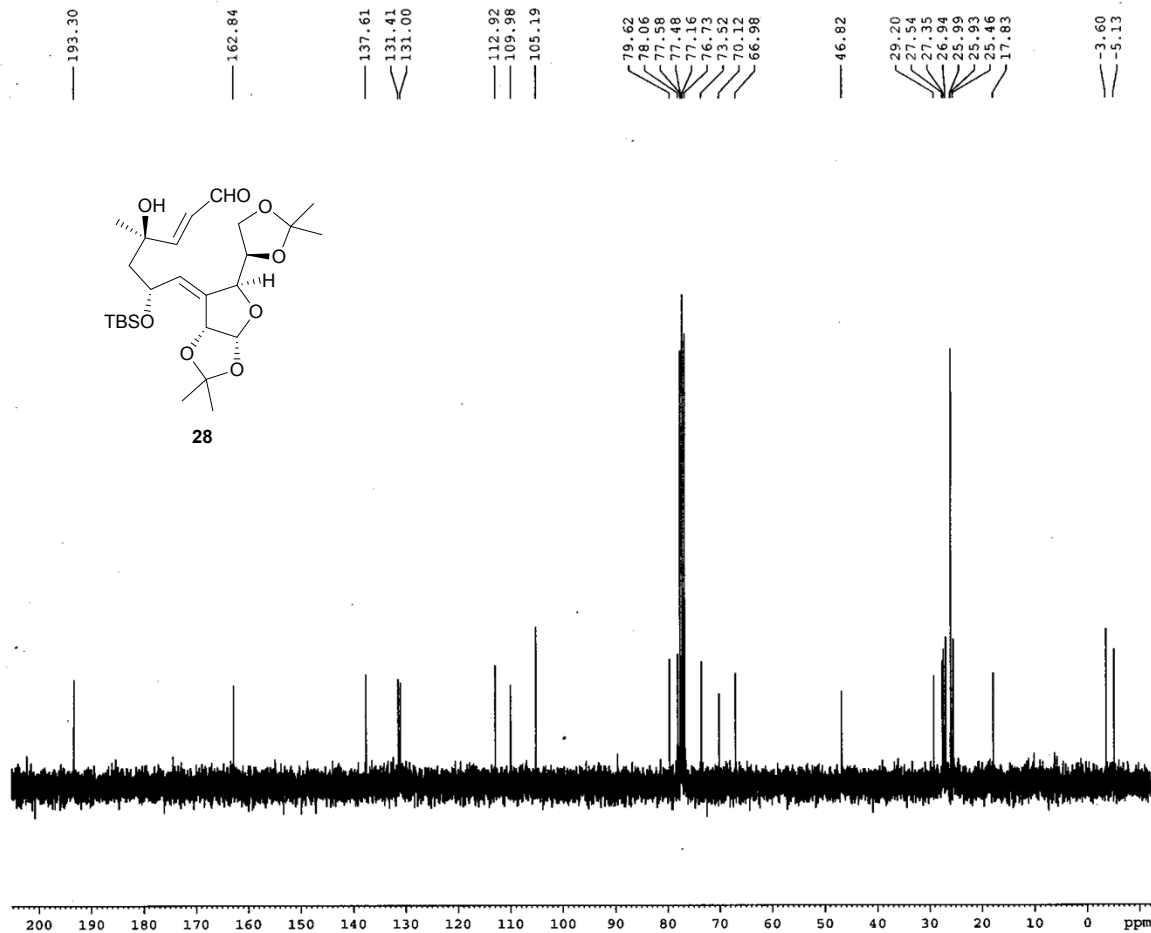
Current Data Parameters
 NAME AJ-3087 300MHZ-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20140120
 Time 12.58
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 90
 DS 4
 SWH 21929.824 Hz
 FIDRES 0.669245 Hz
 AQ 0.7471104 sec
 RG 16.41
 DW 22.800 usec
 DE 6.50 usec
 TE 296.6 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 75.4752949 MHz
 NUC1 13C
 P1 9.25 usec
 PLW1 34.0000000 W

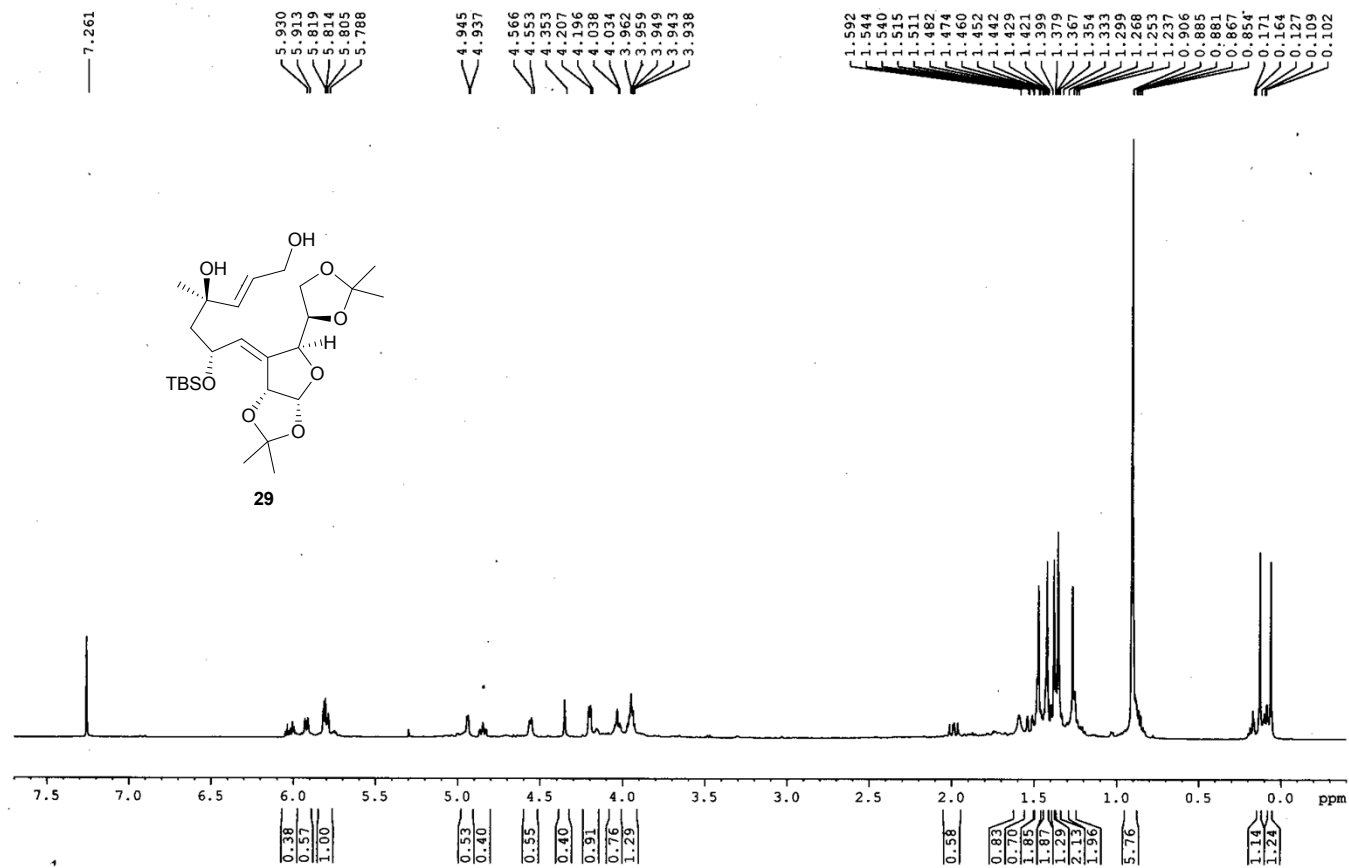
===== CHANNEL f2 =====
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 6.99840021 W
 PLW12 0.17545000 W
 PLW13 0.14211001 W

F2 - Processing parameters
 SI 32768
 SF 75.4677402 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.40



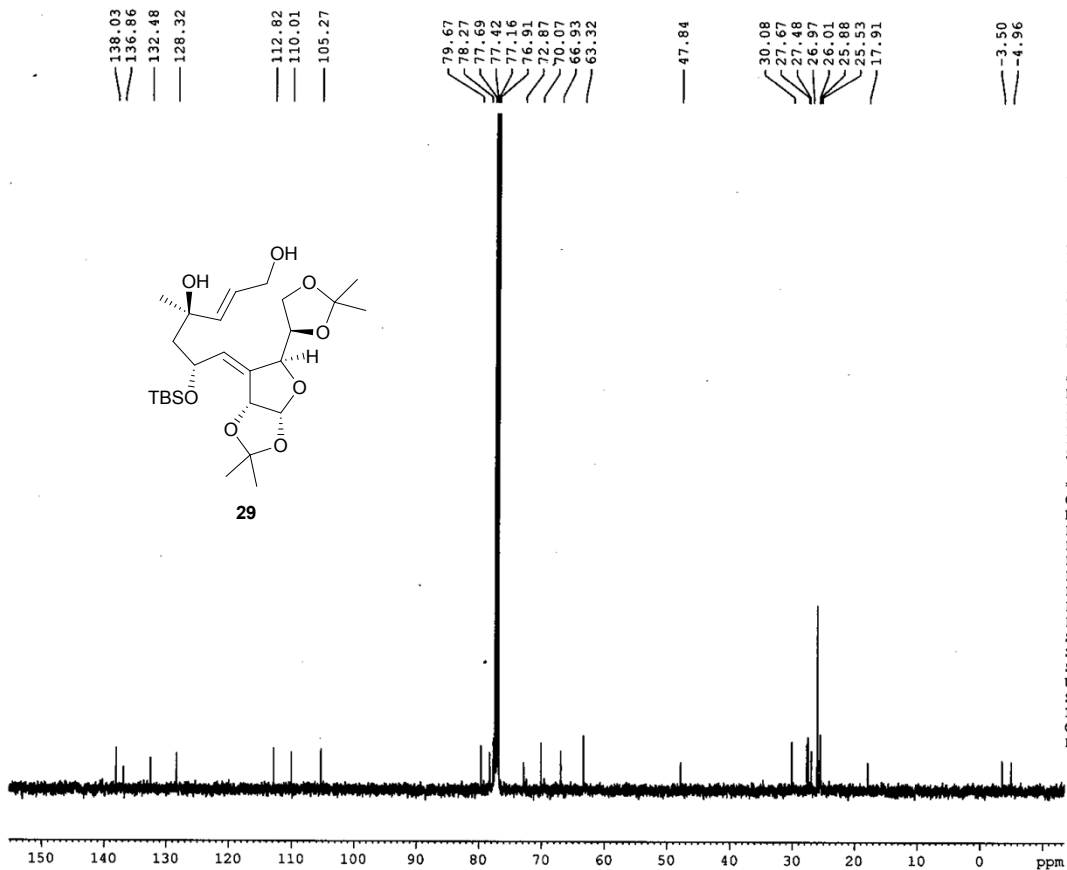
¹H NMR of Compound 29 (CDCl₃, 500 MHz)

AJ2041-1H (SKD)



¹³C NMR of Compound 29 (CDCl₃, 125 MHz)

AJ2041-13C (SKD)



```

NAME      AJ2041-13C
EXPNO     1
PROCNO    1
Date_     20130214
Time      13.06
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        32768
SOLVENT   CDCl3
NS        1633
DS        4
SWH       29761.904 Hz
FIDRES    0.908261 Hz
AQ        0.5505524 sec
RG        32
DW        16.800 usec
DE        6.50 usec
TE        298.8 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

```

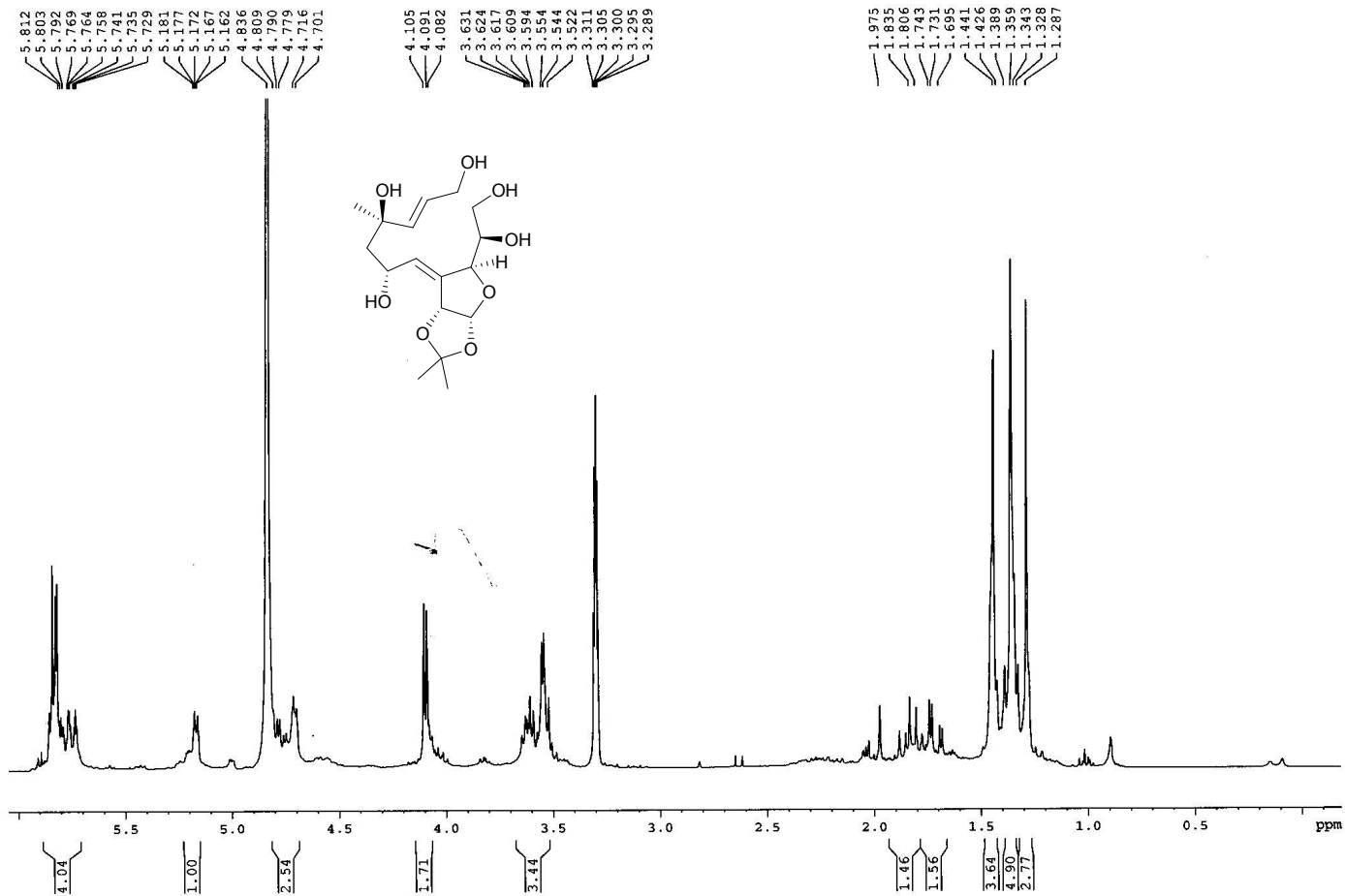
===== CHANNEL f1 =====
NUC1      13C
P1        9.88 usec
PL1       0.50 dB
PL1W     82.16106415 W
SFO1     125.7703643 MHz
    
```

```

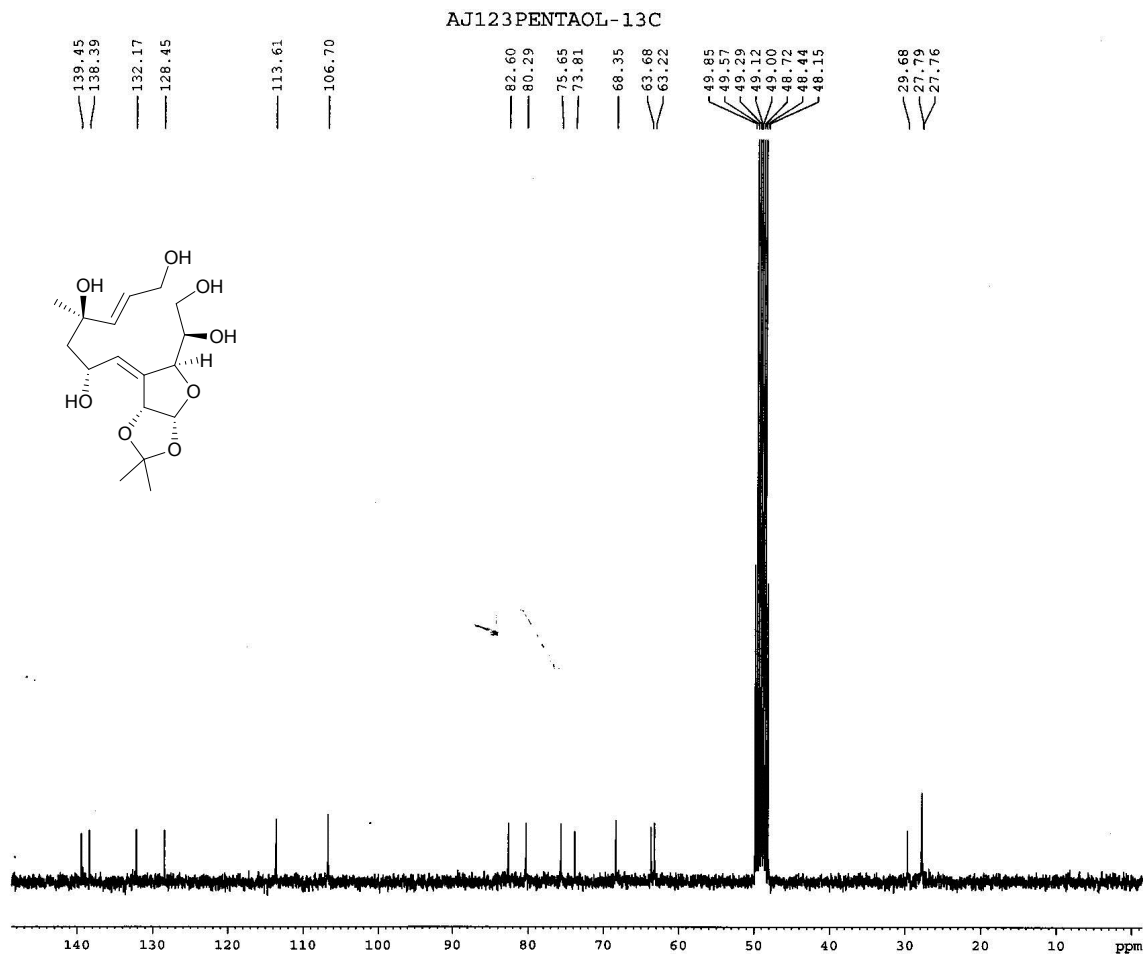
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12     16.50 dB
PL13     20.00 dB
PL2W     15.50318813 W
PL12W    0.43693921 W
PL13W    0.19517358 W
SFO2     500.1320005 MHz
SI        16384
SF       125.7577680 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

^1H NMR (CD₃OD, 300 MHz)

AJ123PENTAOL-1H



¹³C NMR (CD₃OD, 300 MHz)



Current Data Parameters
 NAME AJ123PENTAOL-13C
 EXPNO 1
 PROCNO 1

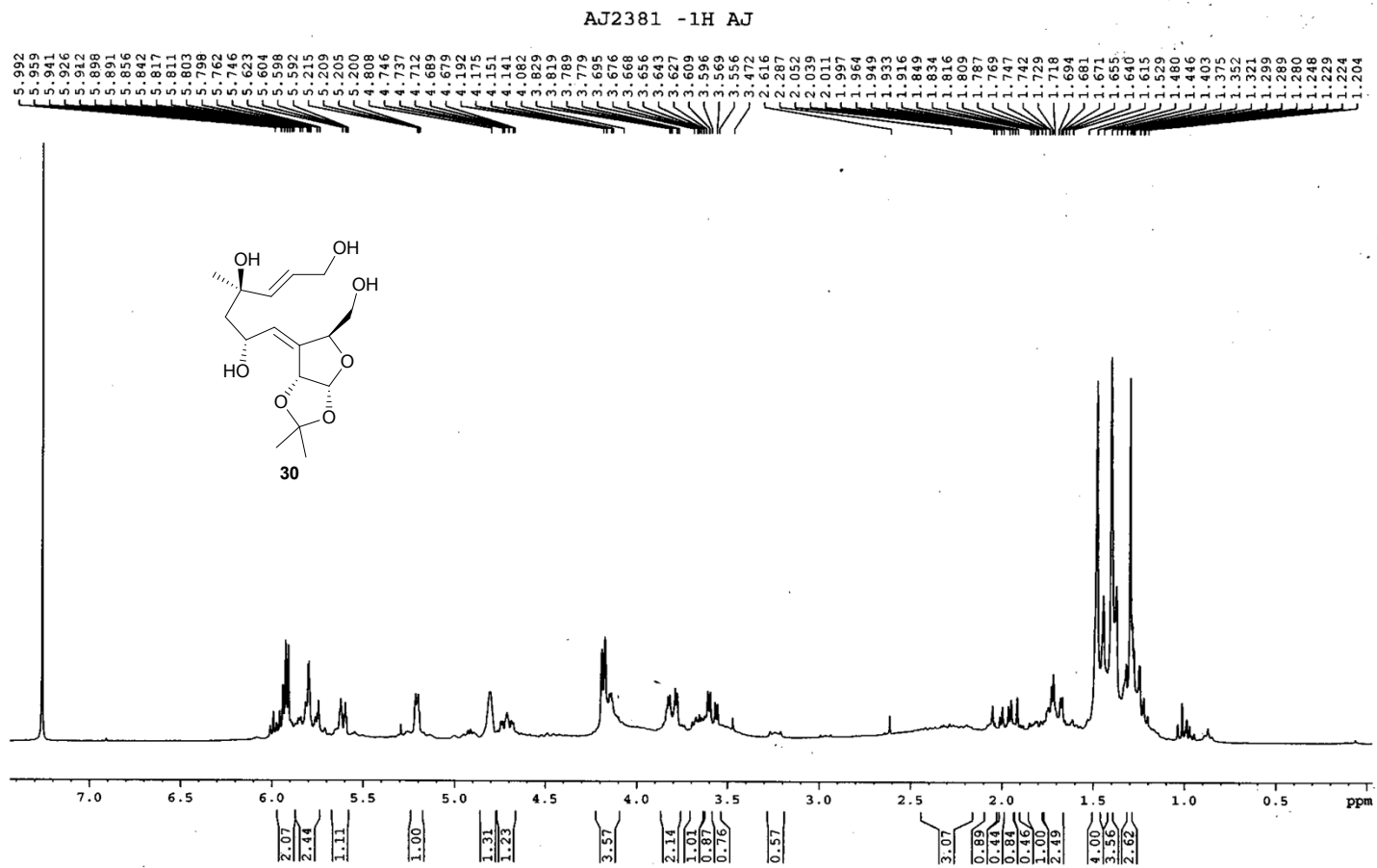
F2 - Acquisition Parameters
 Date_ 20140908
 Time 18.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT MeOD
 NS 819
 DS 4
 SWH 22727.273 Hz
 FIDRES 0.693581 Hz
 AQ 0.7208960 sec
 RG 210.78
 DW 22.000 usec
 DE 6.50 usec
 TE 299.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 75.4752949 MHz
 NUC1 13C
 P1 9.25 usec
 PLW1 34.00000000 W

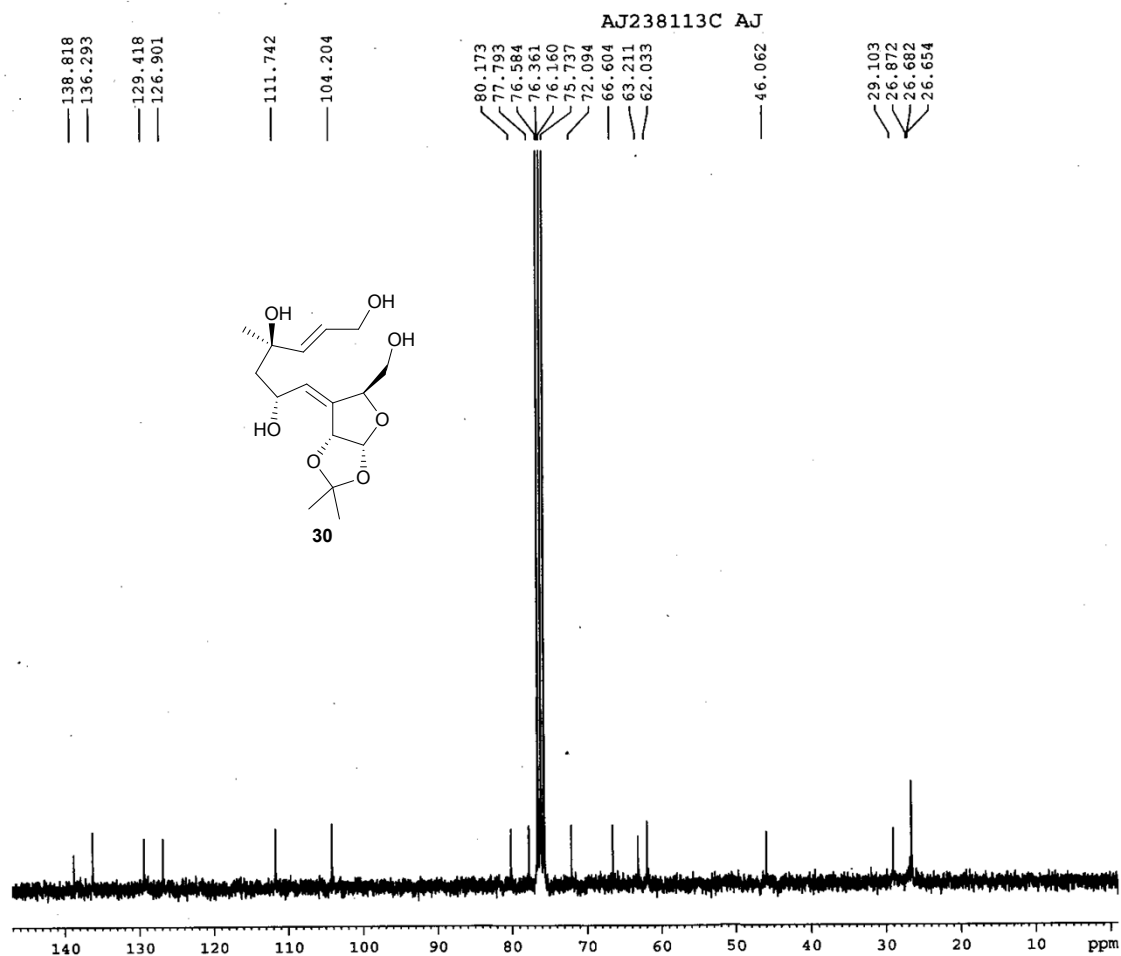
==== CHANNEL f2 =====
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 6.99840021 W
 PLW12 0.17545000 W
 PLW13 0.14211001 W

F2 - Processing parameters
 SI 32768
 SF 75.4676426 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹H NMR of Compound 30 (CDCl₃, 300 MHz)



¹³C NMR of Compound 30 (CDCl₃, 75 MHz)



Current Data Parameters
 NAME AJ238113C AJ
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130816
 Time 12.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1753
 DS 4
 SWH 18028.846 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 210.78
 DW 27.733 usec
 DE 6.50 usec
 TE 298.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

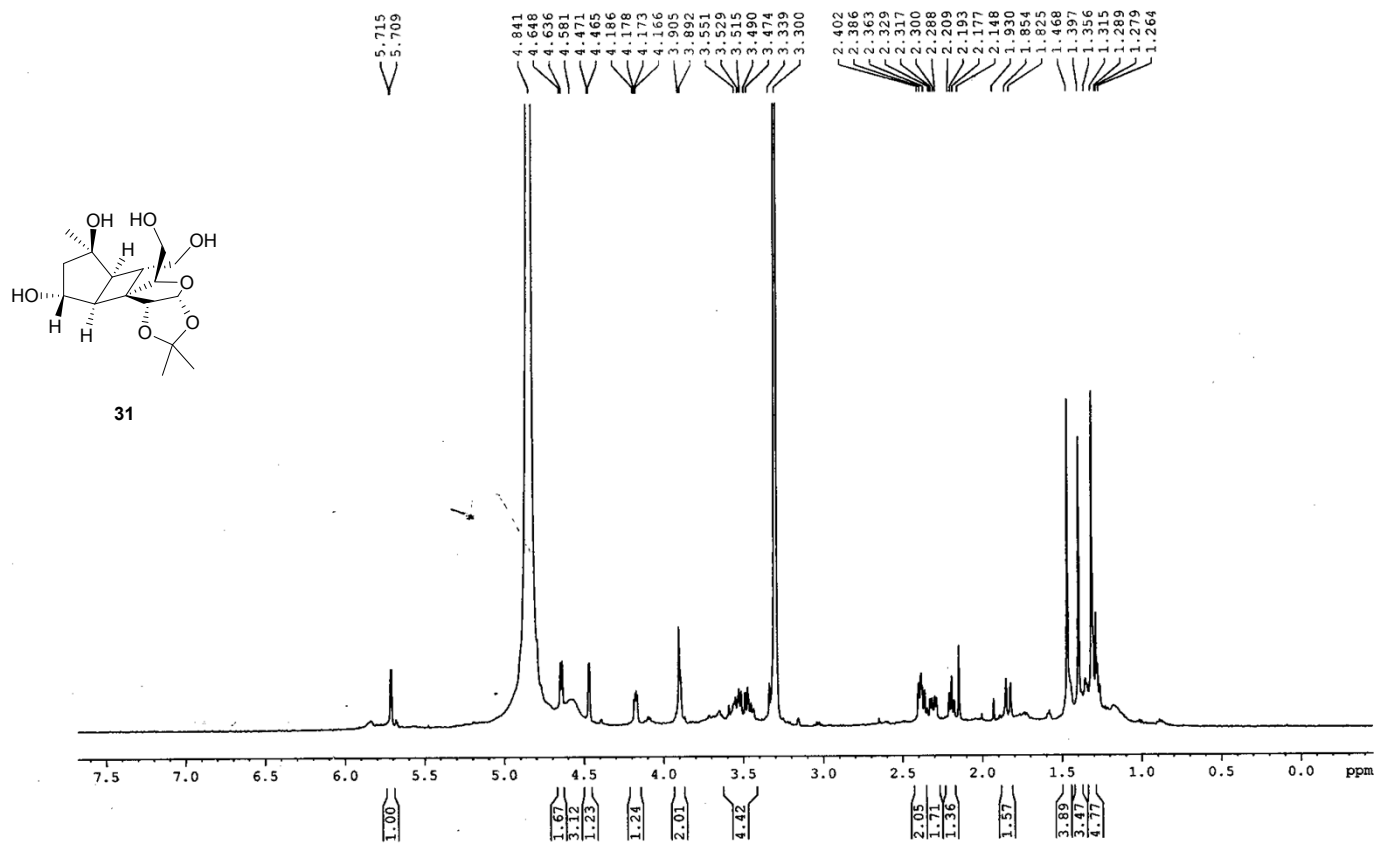
===== CHANNEL f1 =====
 SFO1 75.4752949 MHz
 NUC1 13C
 P1 9.25 usec
 PLW1 34.00000000 W

===== CHANNEL f2 =====
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 6.99840021 W
 PLW12 0.17545000 W
 PLW13 0.14211001 W

F2 - Processing parameters
 SI 32768
 SF 75.4678132 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

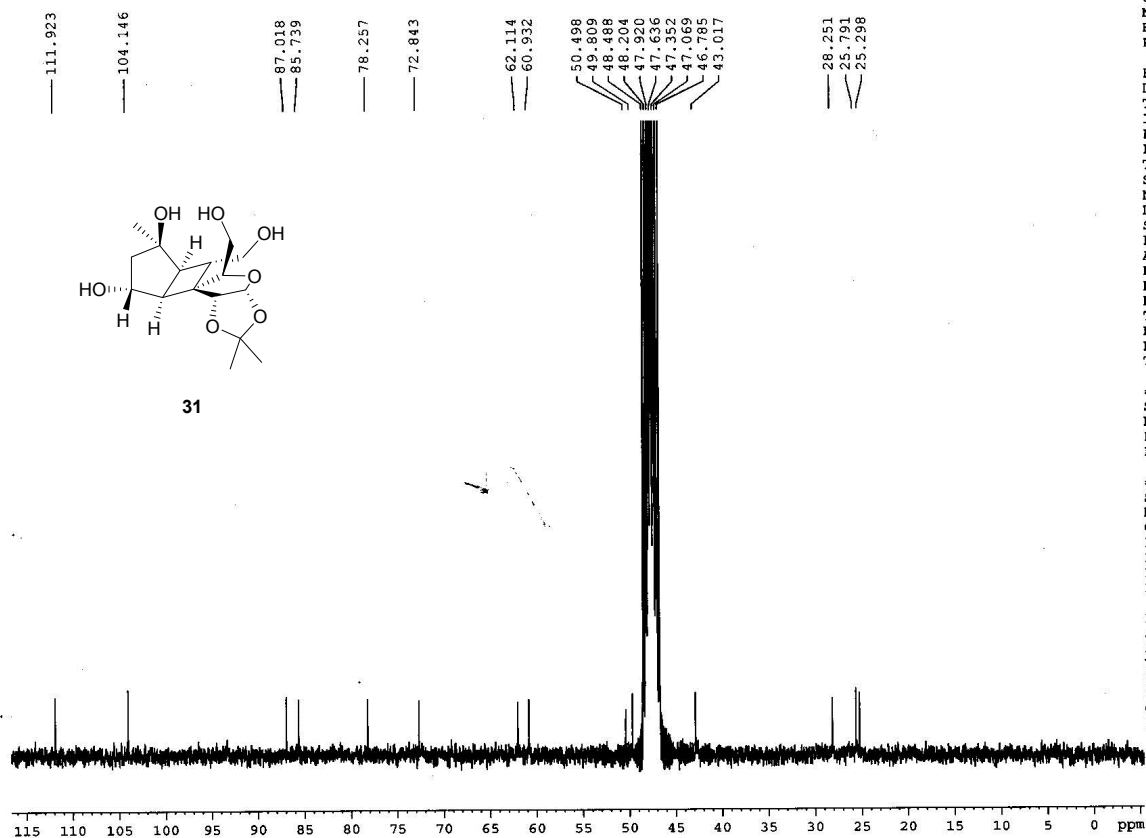
¹H NMR of Compound 31 (CD₃OD, 500 MHz)

AJ2328B-1H (SR) 500MHz.



¹³C NMR of Compound 31 (CD₃OD, 75 MHz)

AJ2382C2-13C(SR)



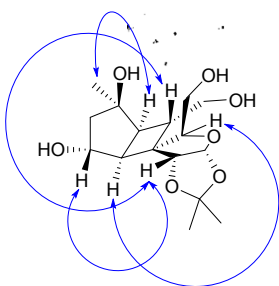
Current Data Parameters
 NAME AJ2382C2-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130821
 Time_ 9.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT MeOD
 NS 17135
 DS 4
 SWH 21929.824 Hz
 FIDRES 0.669245 Hz
 AQ 0.7471104 sec
 RG 210.78
 DW 22.800 usec
 DE 6.50 usec
 TE 299.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

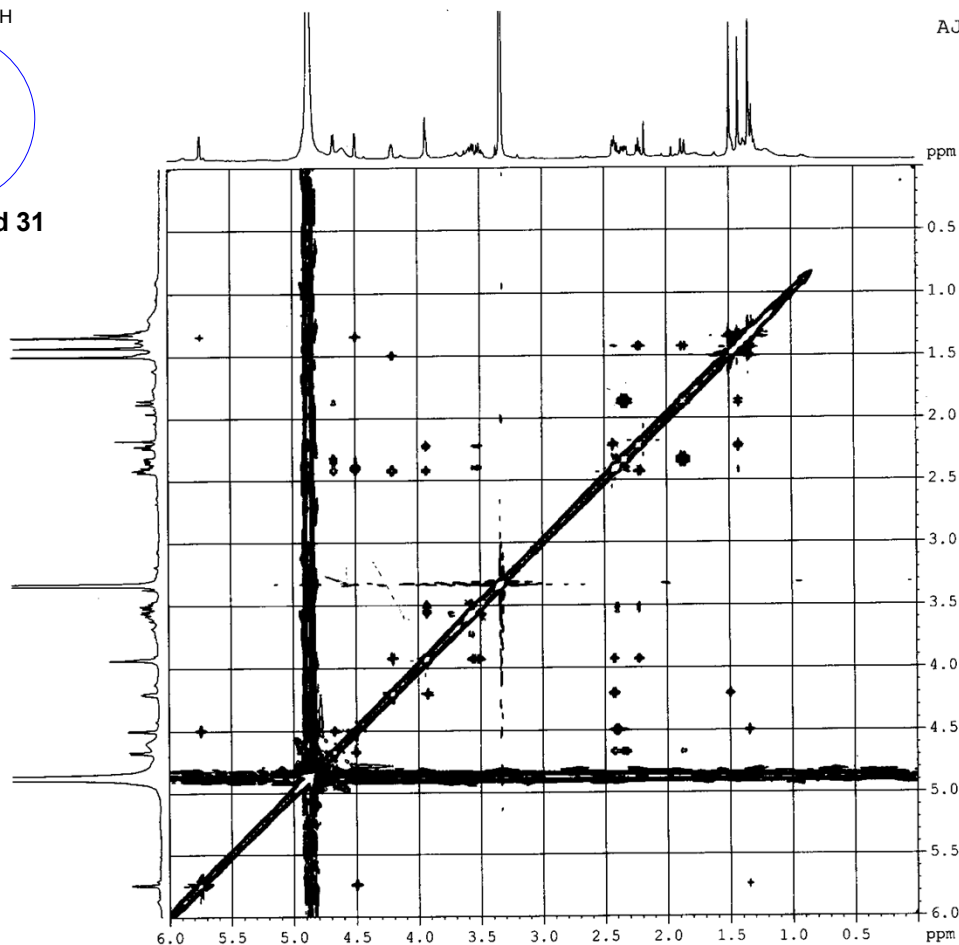
==== CHANNEL f1 =====
 SFO1 75.4752949 MHz
 NUC1 13C
 P1 9.25 usec
 PLW1 34.00000000 W

==== CHANNEL f2 =====
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 6.99840021 W
 PLW12 0.17545000 W
 PLW13 0.14211001 W

F2 - Processing parameters
 SI 32768
 SF 75.4677451 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



NOESY of compound 31



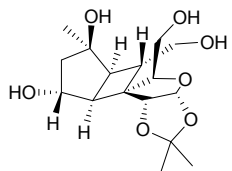
AJ2382B-noesy (SR)

```

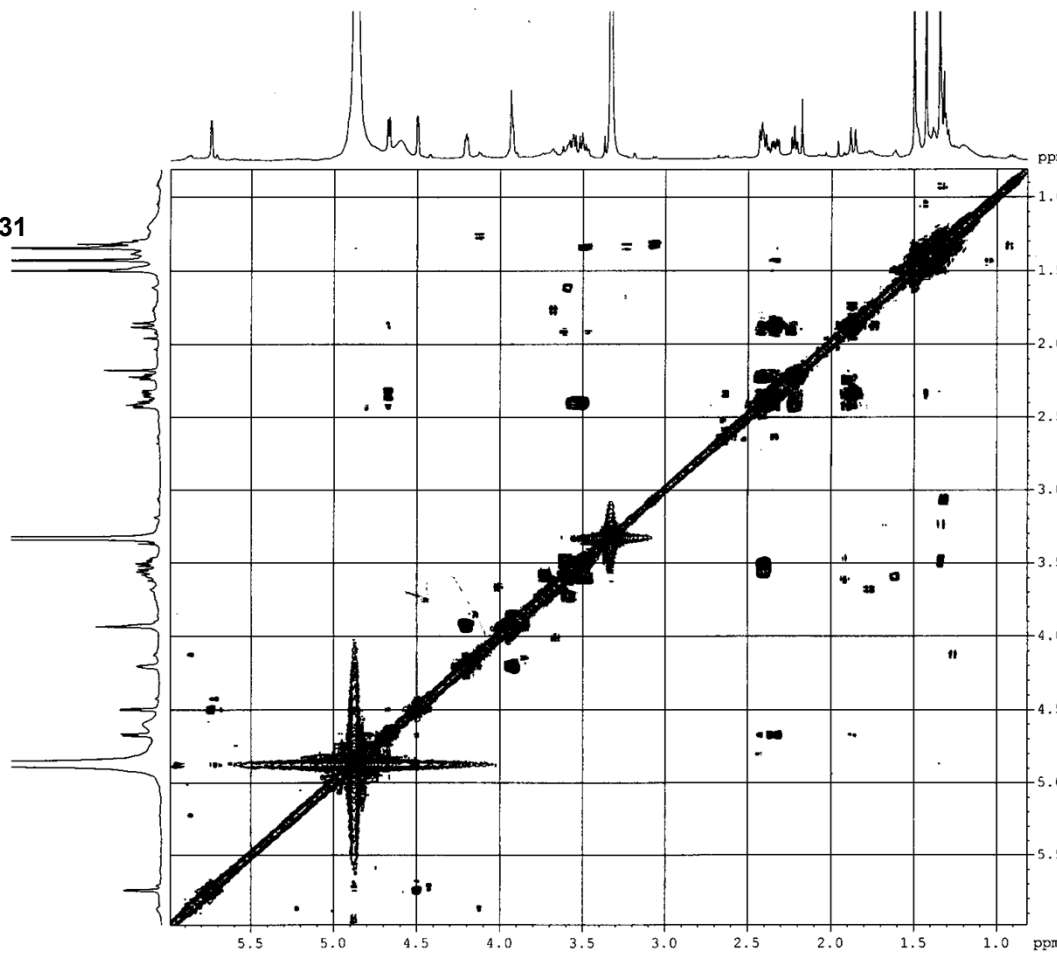
NAME      AJ2382B-noesy
EXPNO     2
PROCNO    1
Date_     20130918
Time      19.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   noesyph
TD         2048
SOLVENT   MeOD
NS         80
DS         16
SWH        3001.200 Hz
FIDRES     1.465430 Hz
AQ         0.3412468 sec
RG         57
DW         166.600 usec
DE         6.50 usec
TE         294.5 K
DO         0.00014706 sec
D1         2.00000000 sec
D8         0.80000001 sec
IN0        0.00033320 sec
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         15.35 usec
PL1        1.00 dB
PL1W      15.50318813 W
SF01      500.1315004 MHz
ND0        1
TD         209
SF01      500.1315 MHz
FIDRES     14.359808 Hz
SW         6.001 ppm
FrMODE    States-TPPI
SI         1024
SF         500.1300000 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         0.80
SI         1024
MC2       States-TPPI
SF         500.1300000 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
  
```

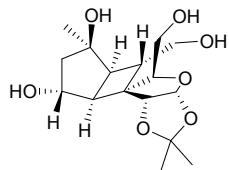


COSY of compound 31

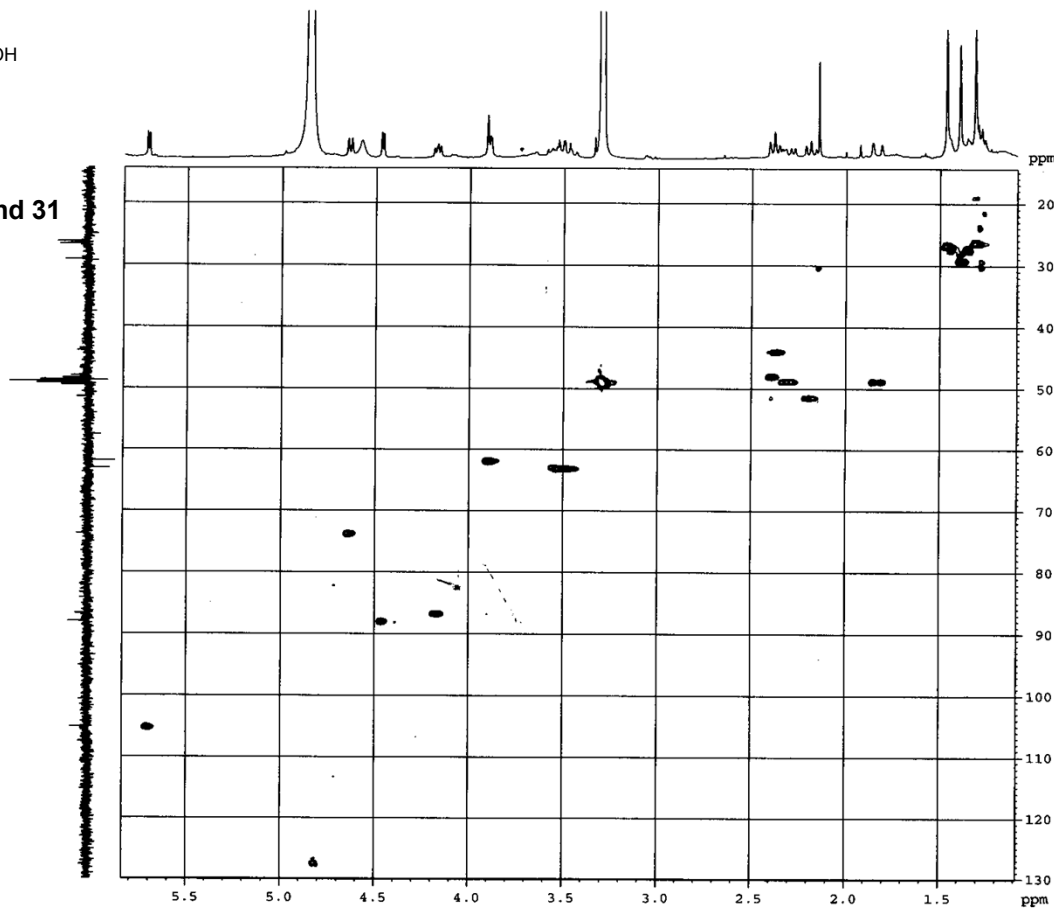


AJ2382B-cosy (SR)

NAME	AJ2382B-cosy
EXPNO	7
PROCNO	1
Date_	20130916
Time	19.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	cosyppqf
TD	2048
SOLVENT	MeOD
NS	256
DS	16
SWH	3001.200 Hz
FIDRES	1.465430 Hz
AQ	0.3412468 sec
RG	80.6
DW	166.600 usec
DE	6.50 usec
TE	295.0 K
DO	0.00000300 sec
D1	1.4869198 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00033320 sec
===== CHANNEL f1 =====	
NUC1	1H
PO	15.35 usec
P1	15.35 usec
PL1	1.00 dB
PL1W	15.50318813 w
SFO1	500.1315004 MHz
===== GRADIENT CHANNEL =====	
GFNAM1	SINE.100
GPZ1	10.00 %
P16	1000.00 usec
NDO	1
TD	110
SFO1	500.1315 MHz
FIDRES	27.283609 Hz
SW	6.001 ppm
FhMODE	QF
SI	1024
SF	500.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	500.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0



HSQC of compound 31



AJ2382C2-HSQC (SR)

Current Data Parameters
 NAME AJ2382C2-HSQC
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130826
 Time 20.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG hsqcetgpsi2
 TD 1024
 SOLVENT MeOD
 NS 128
 DS 16
 SWH 1802.451 Hz
 FIDRES 1.760206 Hz
 AQ 0.2840576 sec
 RG 57.75
 DW 277.400 usec
 DE 6.50 usec
 TE 299.3 K
 CNST2 145.0000000
 D0 0.00000300 sec.
 D1 1.50000000 sec
 D4 0.00172414 sec
 D11 0.03000000 sec
 D16 0.00020000 sec
 D24 0.00086207 sec
 IN0 0.00005100 sec
 ZGPTNS

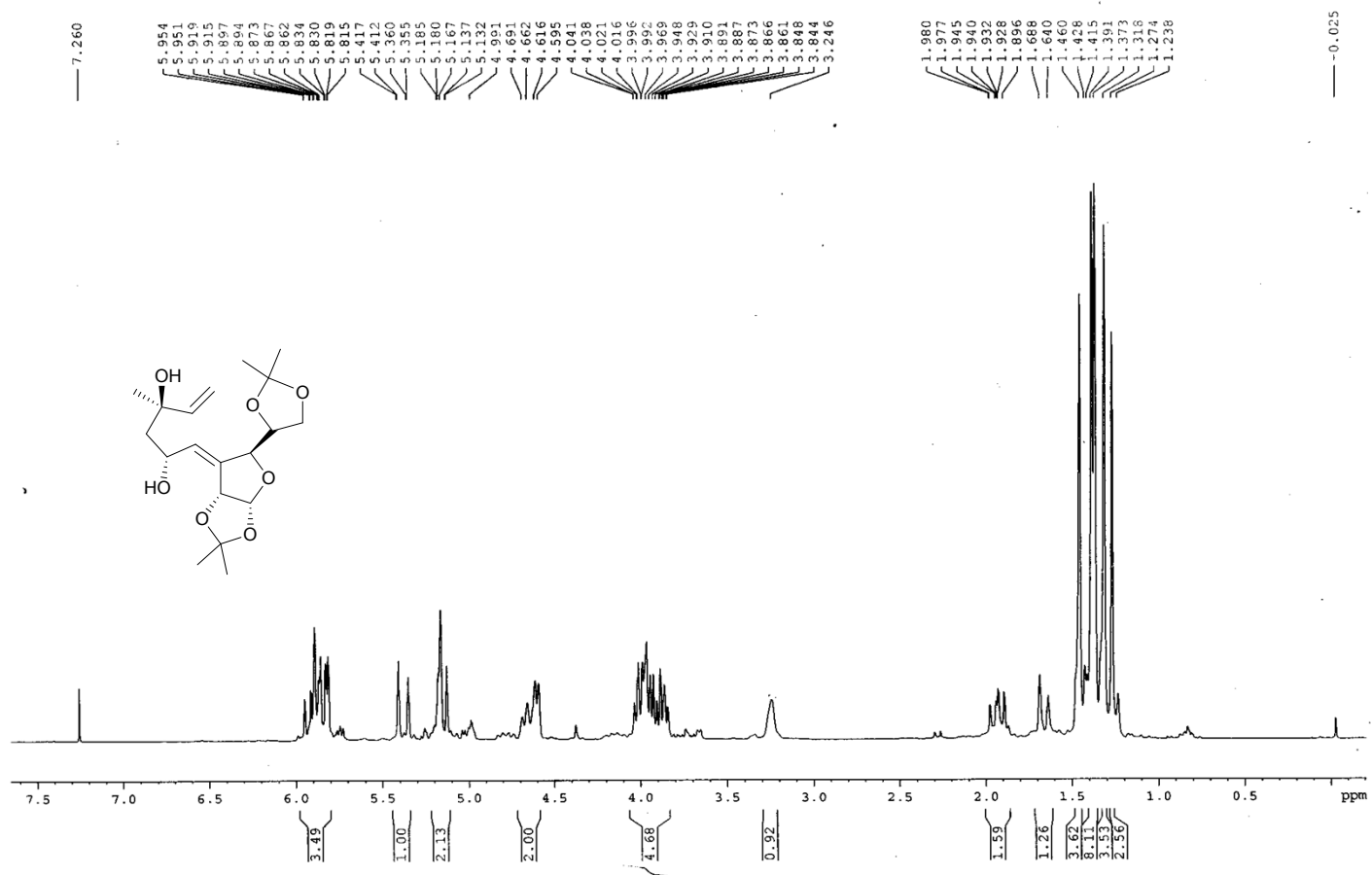
==== CHANNEL f1 =====
 SFO1 300.1309004 MHz
 NUC1 1H
 P1 14.25 usec
 P2 28.50 usec
 P28 0 usec
 PLW1 7.00000000 W

==== CHANNEL f2 =====
 SFO2 75.4726544 MHz
 NUC2 13C
 CPDPRG2 garp
 P3 9.25 usec
 P4 18.50 usec
 PCPD2 80.00 usec
 PLW2 34.00000000 W
 PLW12 0.45455000 W

==== GRADIENT CHANNEL =====
 GPNAM[1] SMSQ10.100
 GPNAM[2] SMSQ10.100
 GPNAM[3] SMSQ10.100
 GPNAM[4] SMSQ10.100
 GPZ1 80.00 %
 GPZ2 20.10 %

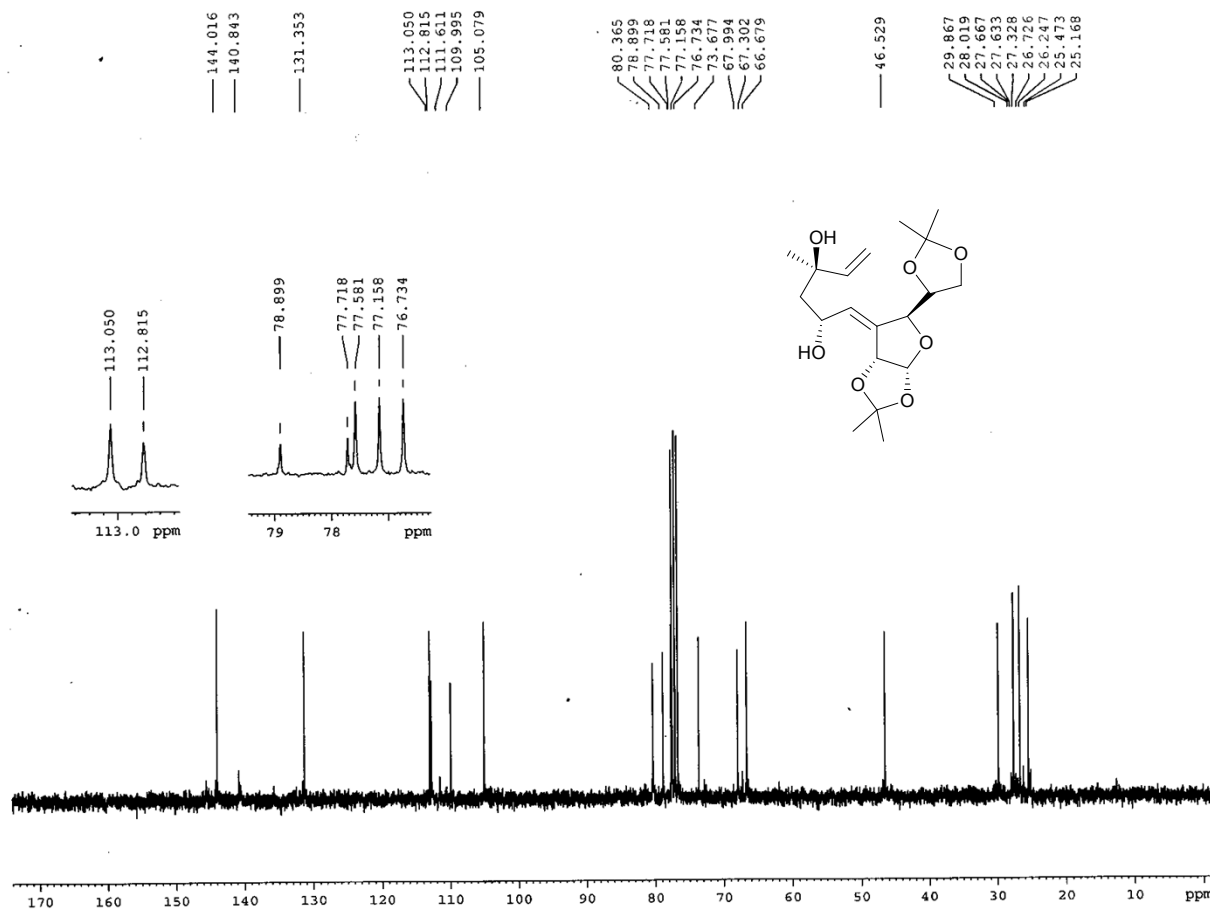
¹H NMR (CDCl₃, 300 MHz)

AJ3112-1H



¹³C NMR (CDCl₃, 75 MHz)

AJ3112-13C



Current Data Parameters
NAME AJ3112-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140320
Time 13.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 108
DS 4
SWH 21929.824 Hz
FIDRES 0.669245 Hz
AQ 0.7471104 sec
RG 210.78
DW 22.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

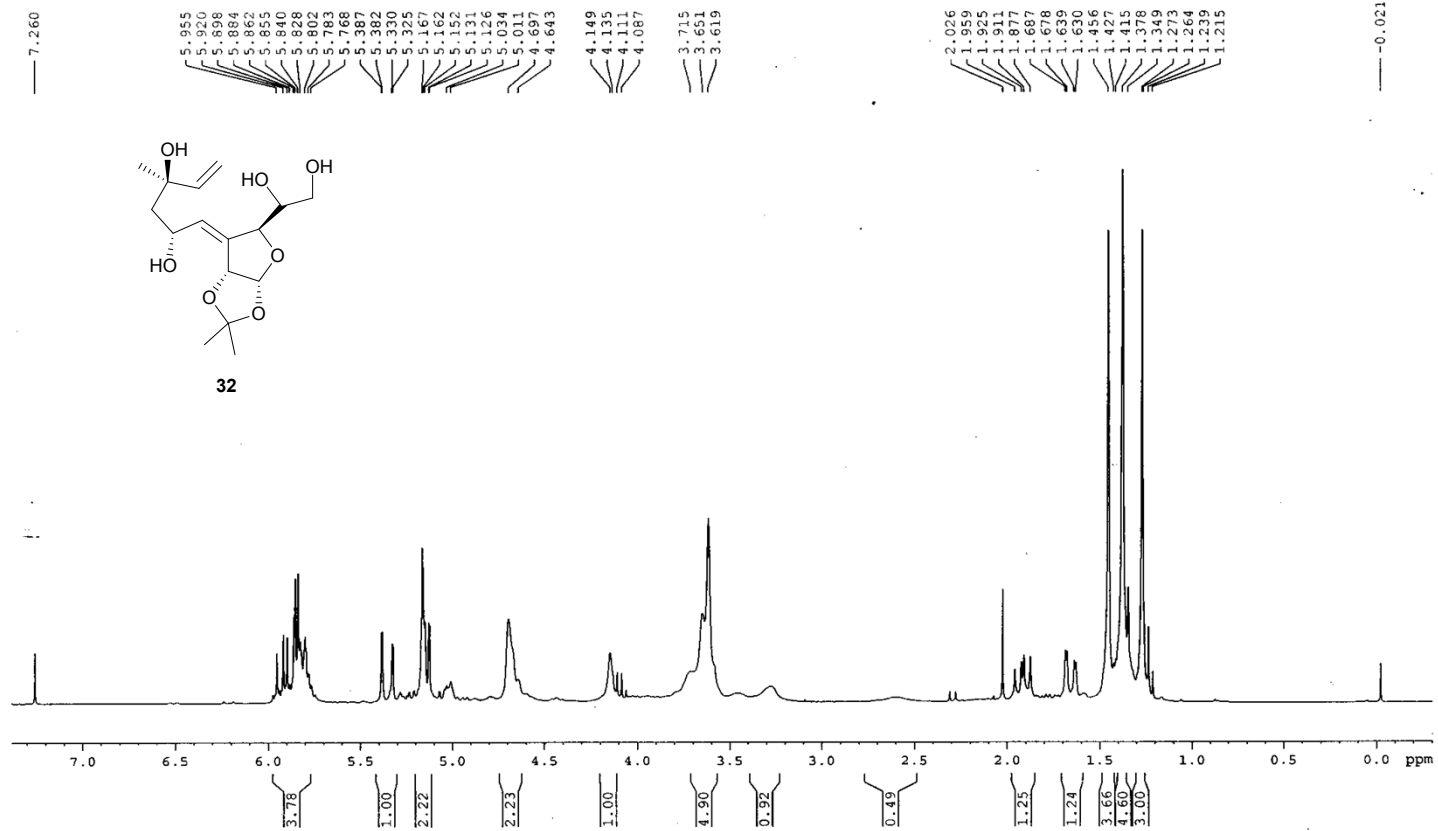
===== CHANNEL f1 =====
SFO1 75.4752949 MHz
NUC1 13C
P1 9.25 usec
PLW1 34.00000000 W

===== CHANNEL f2 =====
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 6.99840021 W
PLW12 0.17545000 W
PLW13 0.14211001 W

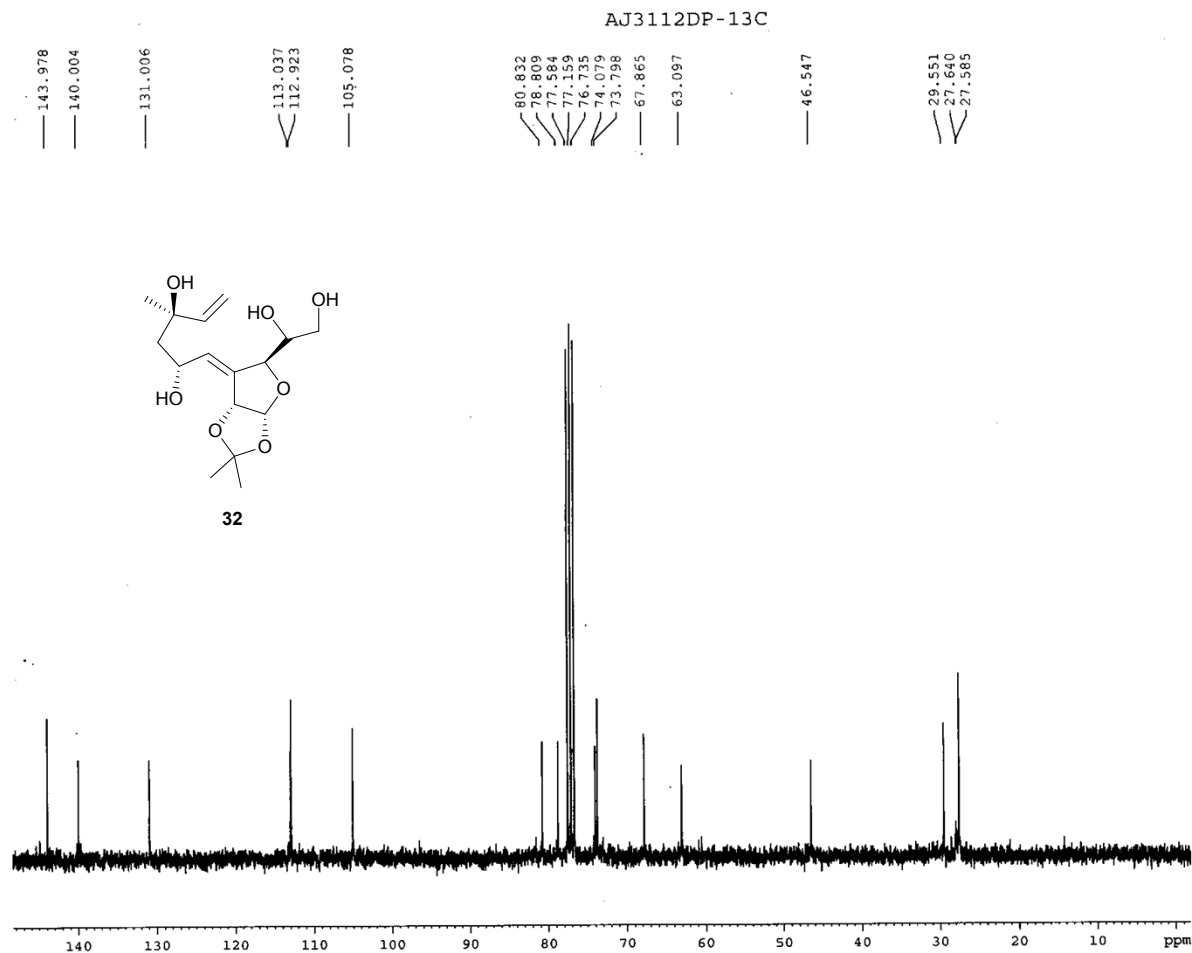
F2 - Processing parameters
SI 32768
SF 75.4677408 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR of Compound 32 (CDCl₃, 300 MHz)

AJ3112DP-1H



¹³C NMR of Compound 32 (CDCl₃, 75 MHz)



```

Current Data Parameters
NAME      AJ3112DP-13C
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20140324
Time      13.15
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         178
DS         4
SWH        21929.824 Hz
FIDRES     0.669245 Hz
AQ         0.7471104 sec
RG         210.78
DW         22.800 usec
DE         6.50 usec
TE         297.4 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

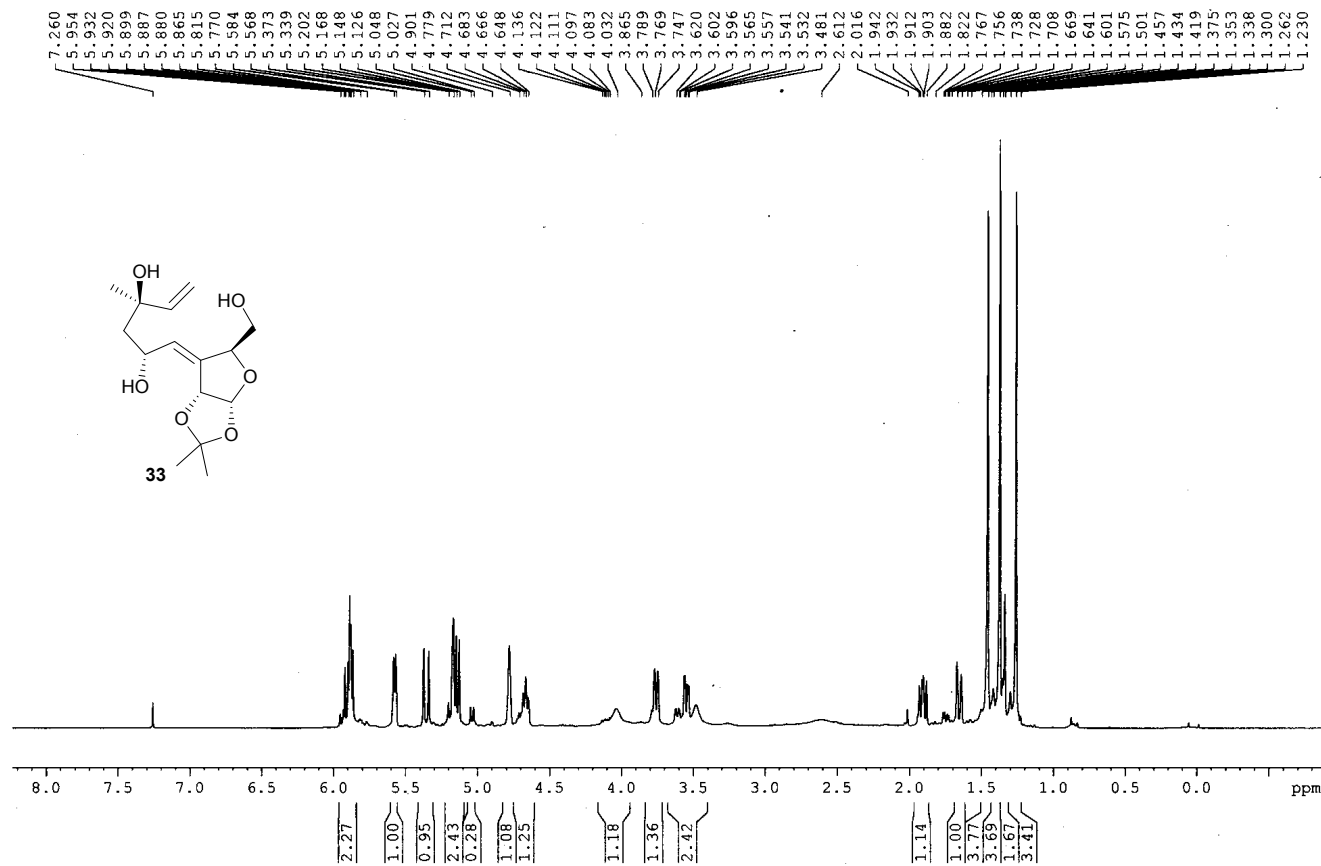
===== CHANNEL f1 =====
SFO1      75.4752949 MHz
NUC1       13C
P1         9.25 usec
PLW1       34.0000000 W

===== CHANNEL f2 =====
SFO2      300.1312005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2     90.00 usec
PLW2      6.99840021 W
PLW12     0.17545000 W
PLW13     0.14211001 W

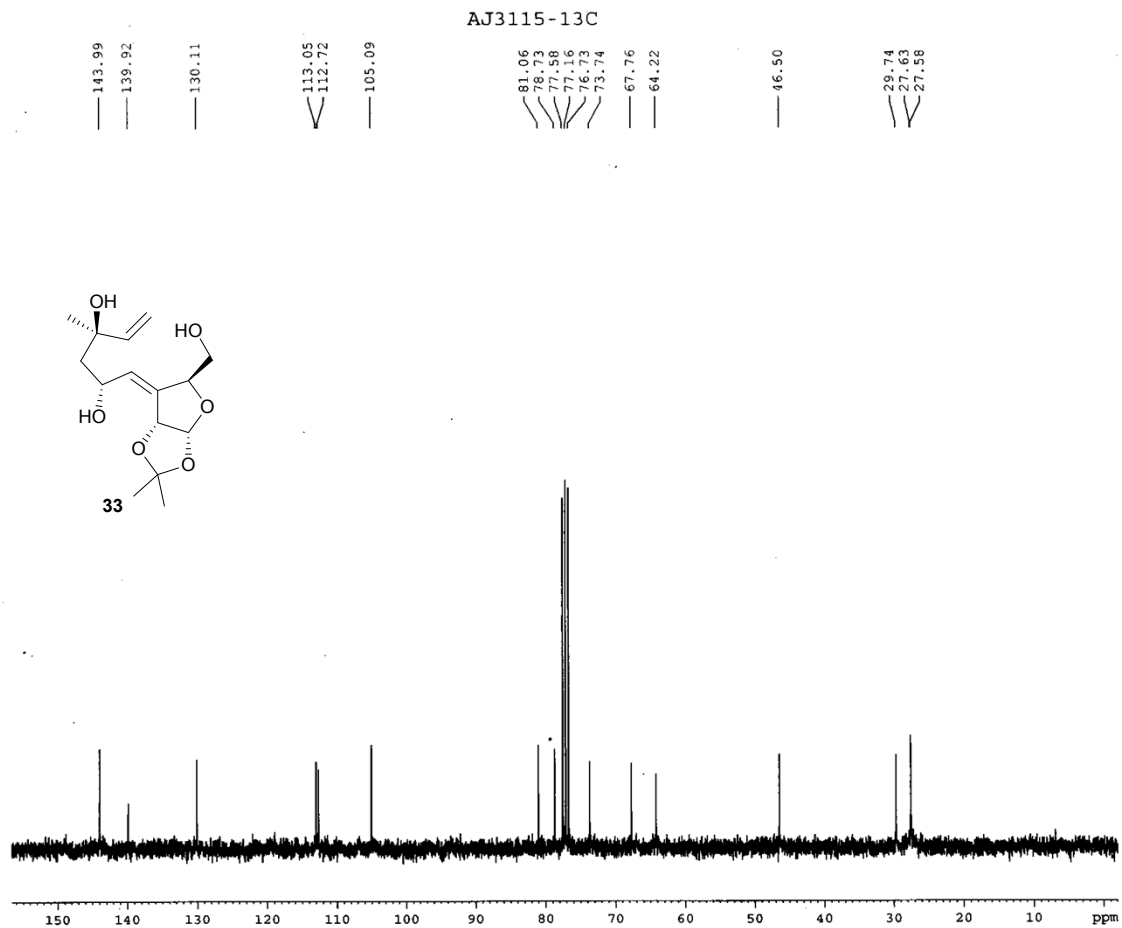
F2 - Processing parameters
SI         32768
SF         75.4677402 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

¹H NMR of Compound 33 (CDCl₃, 500 MHz)

AJ2241-1H(SR) 500MHz.



¹³C NMR of Compound 33 (CDCl₃, 75 MHz)



```

Current Data Parameters
NAME      AJ3115-13C
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20140326
Time      13.12
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         107
DS         4
SWH        21929.824 Hz
FIDRES     0.669245 Hz
AQ         0.7471104 sec
RG         16.41
DW         22.800 usec
DE         6.50 usec
TE         298.5 K
D1         2.0000000 sec
DL1        0.0300000 sec
TD0        1

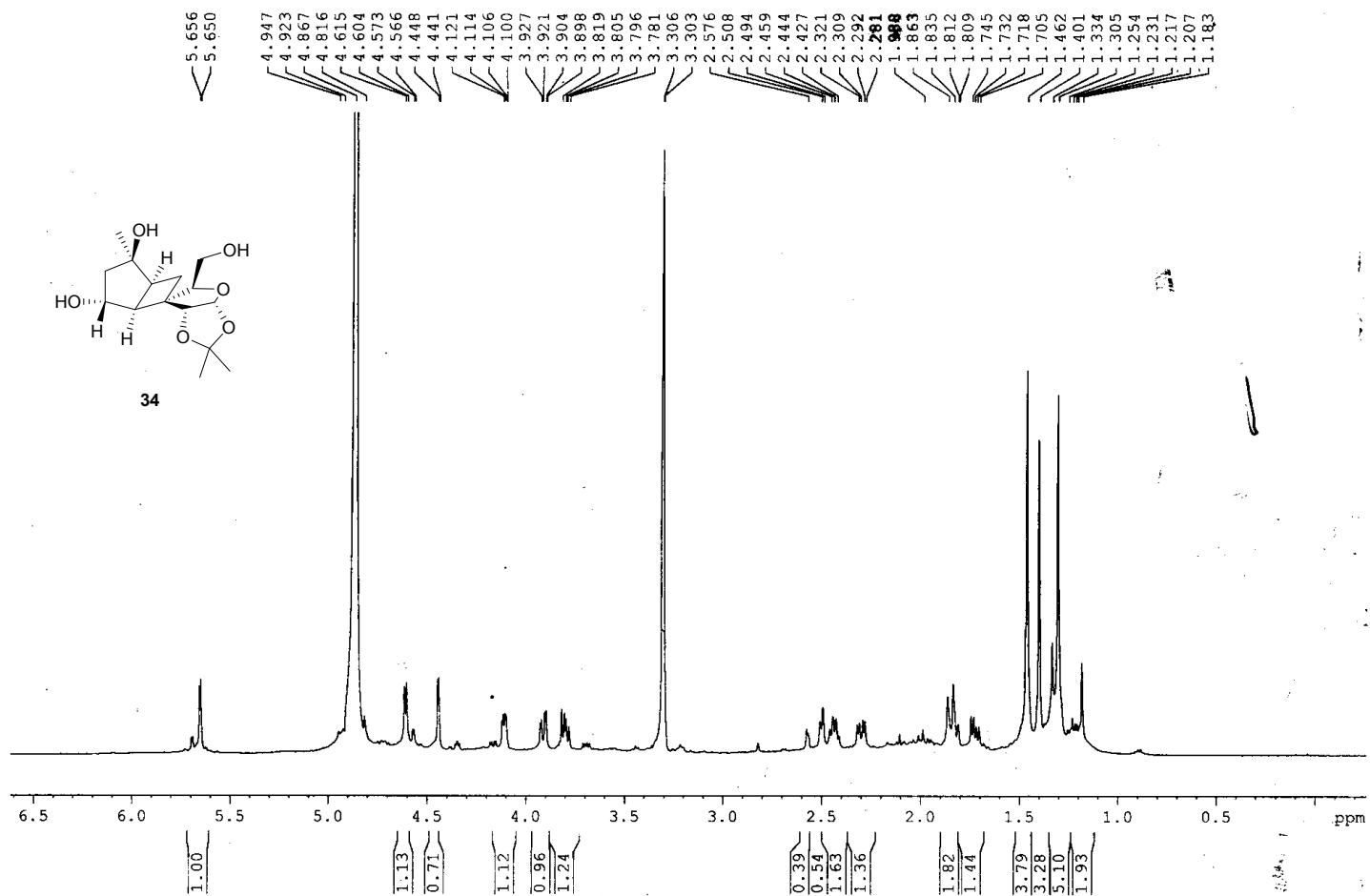
===== CHANNEL f1 =====
SFO1       75.4752949 MHz
NUC1        13C
P1          9.25 usec
PLW1       34.0000000 W

===== CHANNEL f2 =====
SFO2       300.1312005 MHz
NUC2         1H
CPDPRG[2]  waltz16
PCPD2      90.00 usec
PLW2       6.99840021 W
PLW12      0.17545000 W
PLW13      0.14211001 W

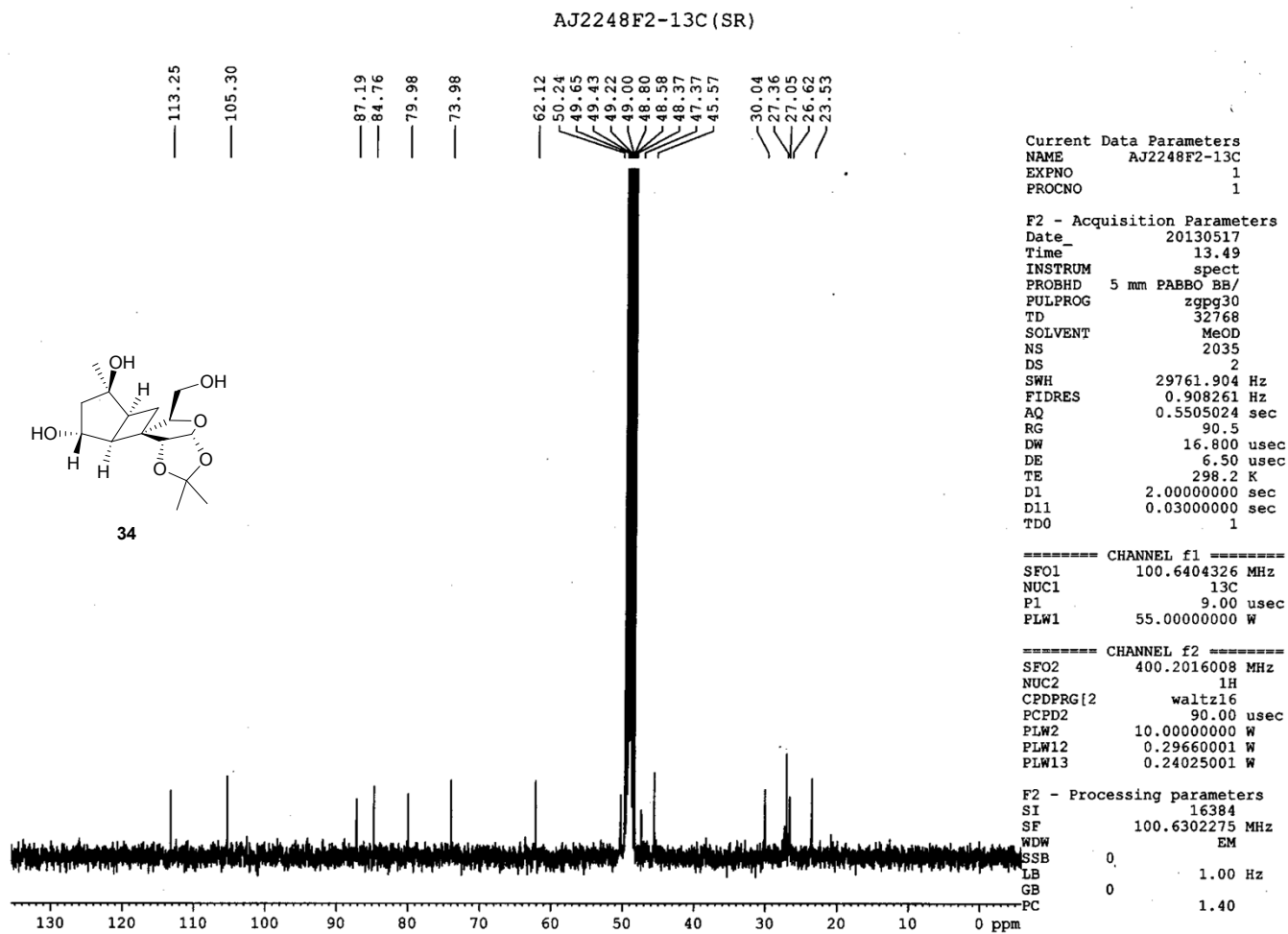
F2 - Processing parameters
SI          32768
SF          75.4677402 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
    
```

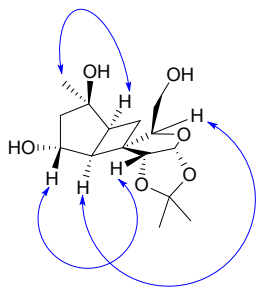
¹H NMR of Compound 34 (CD₃OD, 500 MHz)

AJ2248F2-1H(SR) 500MHz.

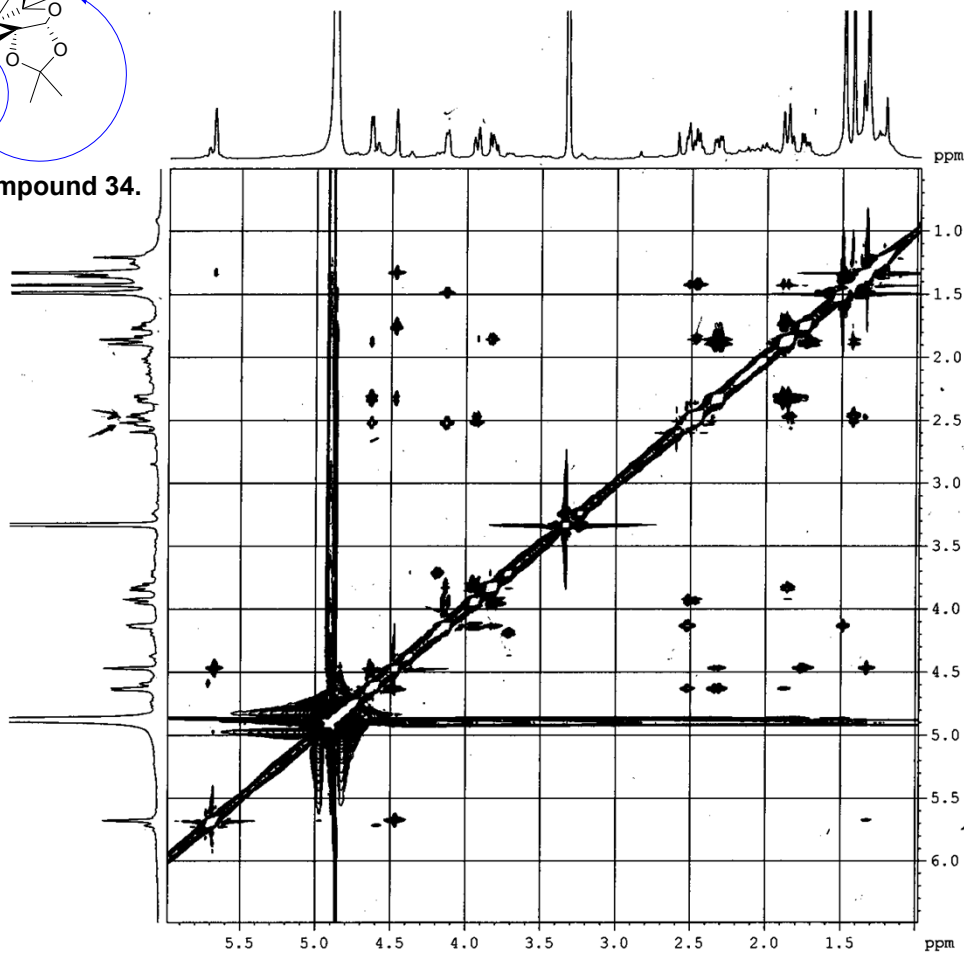


¹³C NMR of Compound 34 (CD₃OD, 125 MHz)





NOESY of compound 34.



AJ2248F2-noesy (SR)

```

Current Data Parameters
NAME      AJ2248F2-noesy
EXPNO    3
PROCNO    1

F2 - Acquisition Parameters
Date_     20130520
Time      19.59
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   noesypphpp
TD        2048
SOLVENT   MeOD
NS        64
DS        16
SWH       3898.129 Hz
FIDRES    1.903383 Hz
AQ        0.2626901 sec
RG        64
DW        128.267 usec
DE        6.50 usec
TE        299.2 K
D0        0.00010856 sec
D1        2.00000000 sec
D8        0.80000001 sec
D11       0.03000000 sec
D12       0.00002000 sec
D16       0.00020000 sec
IN0       0.00025660 sec

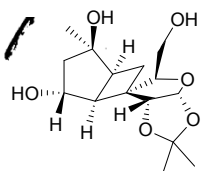
----- CHANNEL f1 -----
SF01      400.2019510 MHz
NUC1      1H
P1        15.50 usec
P2        31.00 usec
P17       2500.00 usec
PLW1      10.00000000 W
PLW10     3.55399990 W

===== GRADIENT CHANNEL =====
GPNAM[1]  SMSQ10.100
GP21      40.00 %
P16       1000.00 usec

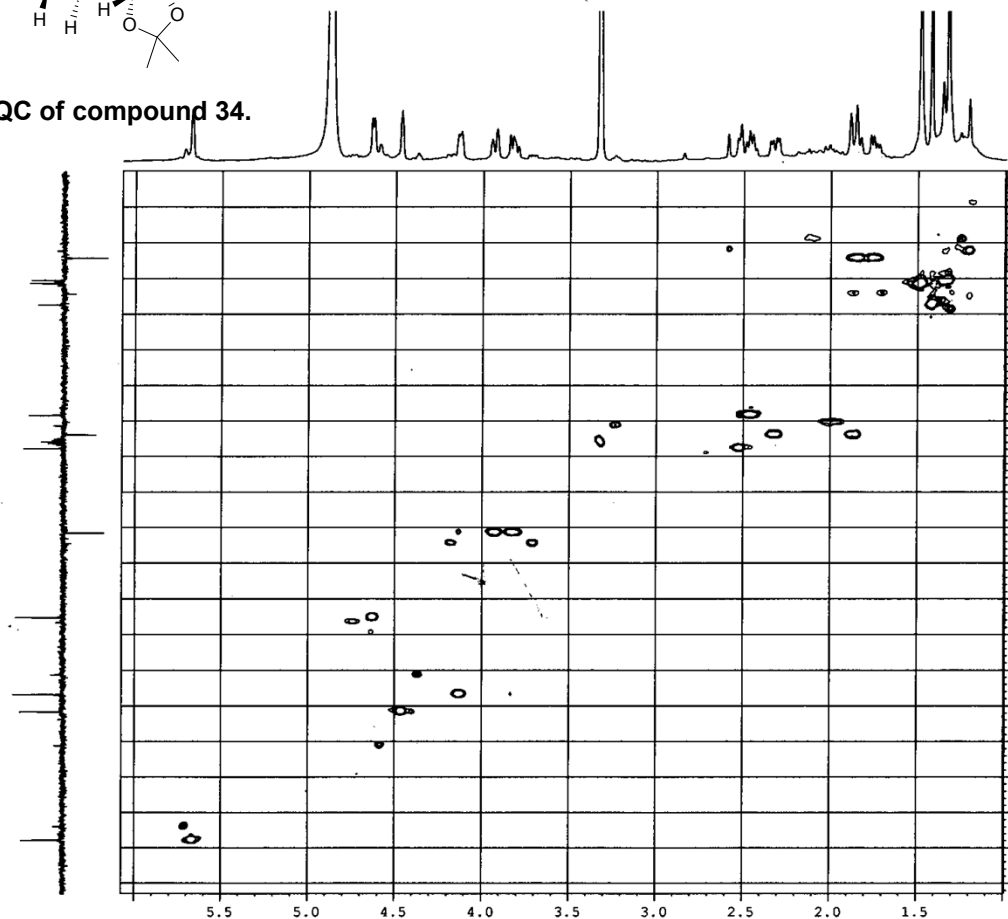
F1 - Acquisition parameters
TD        249
SF01      400.202 MHz
FIDRES    15.651069 Hz
SW        9.738 ppm
FnMODE    States-TPPI

F2 - Processing parameters
SI        1024
SF        400.2000000 MHz
WDW       QSINE
SSB       2
LB        0 Hz
GB        0
PC        1.00

F1 - Processing parameters
SI        1024
MC2       States-TPPI
SF        400.2000000 MHz
WDW       QSINE
SSB       2
LB        0 Hz
GB        0
  
```



HSQC of compound 34.



AJ2248F2-HSQC (SR)

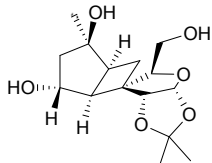
Current Data Parameters
 NAME AJ2248F2-HSQC
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130522
 15 Time 19.49
 INSTRUM spect
 20 PROBHD 5 mm PABBO BB/
 PULPROG hsqcetgps12
 TD 1024
 25 SOLVENT MeOD
 NS 128
 30 DS 16
 SWH 4000.000 Hz
 FIDRES 3.906250 Hz
 35 AQ 0.1280000 sec
 RG 32
 40 DW 125.000 usec
 DE 6.50 usec
 45 TE 298.6 K
 CNST2 145.0000000
 D0 0.00000300 sec
 50 D1 1.50000000 sec
 D4 0.00172414 sec
 55 D11 0.03000000 sec
 D16 0.00020000 sec
 60 D24 0.00086207 sec
 INO 0.00003820 sec

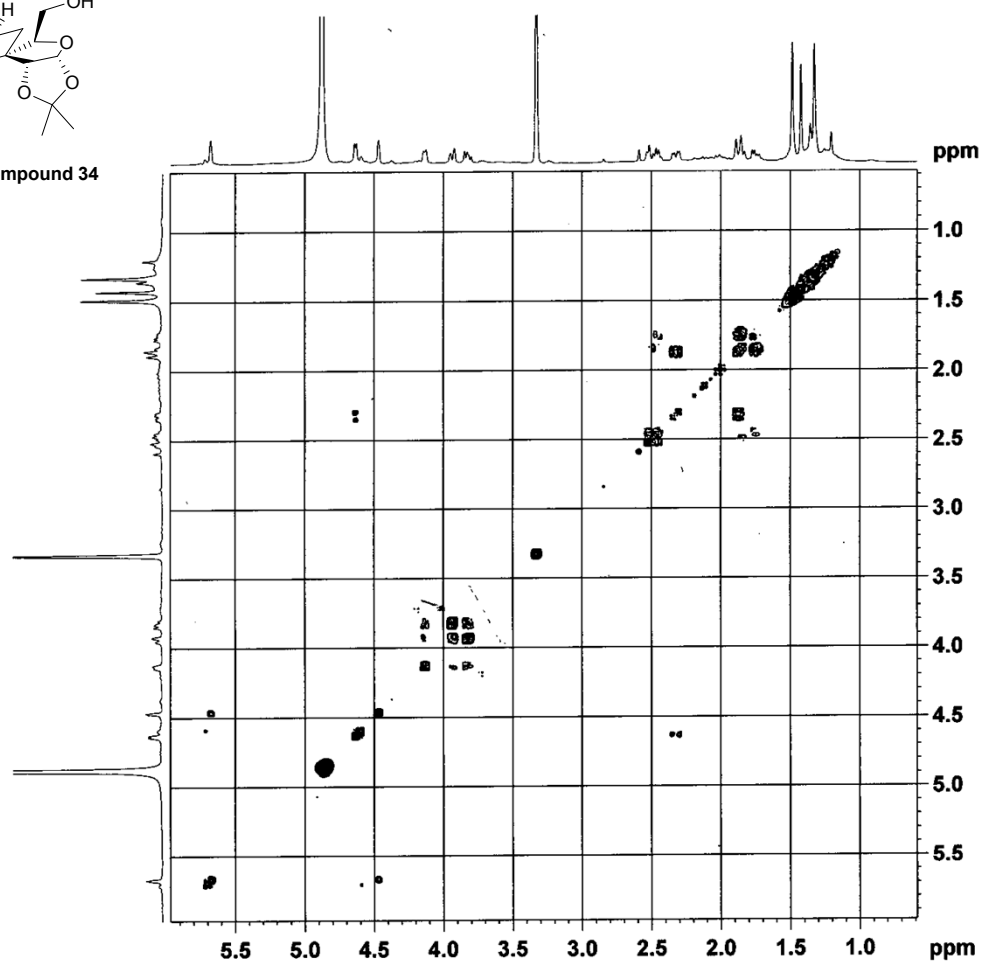
65 ===== CHANNEL f1 =====
 70 SFO1 400.2020010 MHz
 NUC1 1H
 75 P1 15.50 usec
 P2 31.00 usec
 P28 0 usec
 80 PLW1 10.00000000 W

85 ===== CHANNEL f2 =====
 SFO2 100.6369110 MHz
 90 NUC2 13C
 CPDPRG[2] garp
 P3 9.00 usec
 95 P4 18.00 usec
 PCPD2 80.00 usec
 100 PLW2 55.00000000 W
 PLW12 0.69608998 W

105 ===== GRADIENT CHANNEL =====
 110 GPNAM[1] SMSQ10.100
 GPNAM[2] SMSQ10.100
 GPNAM[3] SMSQ10.100
 GPNAM[4] SMSQ10.100
 GPZ1 80.00 %
 GPZ2 20.10 %



COSY of compound 34



AJ2248F2-cosy (SR)

```

Current Data Parameters
NAME      AJ2248F2-cosy
EXPNO    2
PROCNO    1

F2 - Acquisition Parameters
Date_    20130517
Time     18.36
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  cosygpppgf
TD       2048
SOLVENT  MeOD
NS       256
DS       16
SWH      3898.129 Hz
FIDRES   1.903383 Hz
AQ       0.2626901 sec
RG       64
DW       128.267 usec
DE       6.50 usec
TE       298.1 K
D0       0.00000300 sec
D1       2.00000000 sec
D11      0.03000000 sec
D12      0.00002000 sec
D13      0.00000400 sec
D16      0.00020000 sec
IN0      0.00025660 sec

----- CHANNEL f1 -----
SFO1     400.2019510 MHz
NUC1     1H
P0       15.50 usec
P1       15.50 usec
P17      2500.00 usec
PLW1     10.00000000 W
PLW10    3.55399990 W

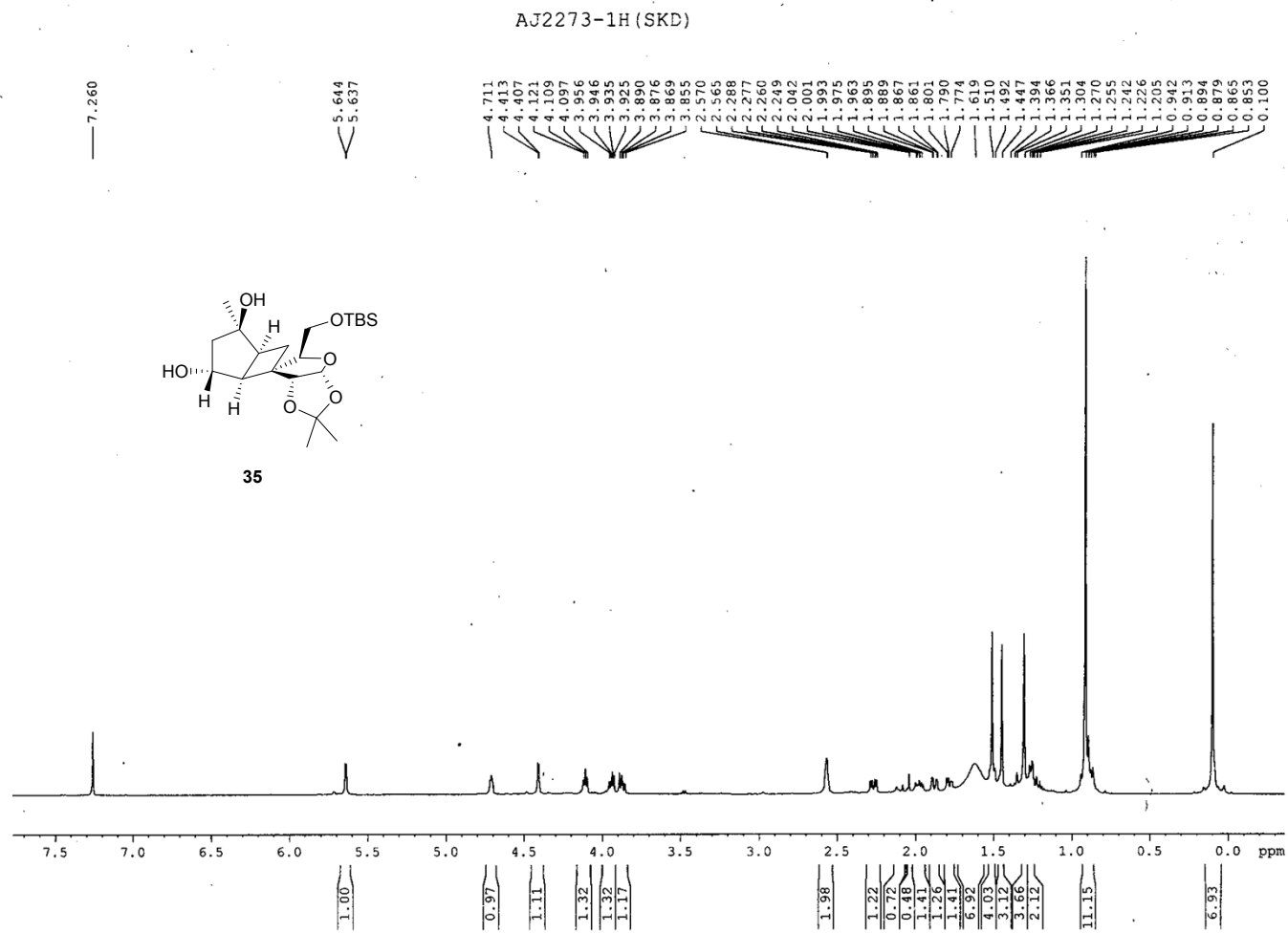
----- GRADIENT CHANNEL -----
GPNAM(1) SMSQ10.100
GP21     10.00 %
P16      1000.00 usec

F1 - Acquisition parameters
TD       128
SFO1     400.202 MHz
FIDRES   30.446220 Hz
SW       9.738 ppm
FnMODE   QF

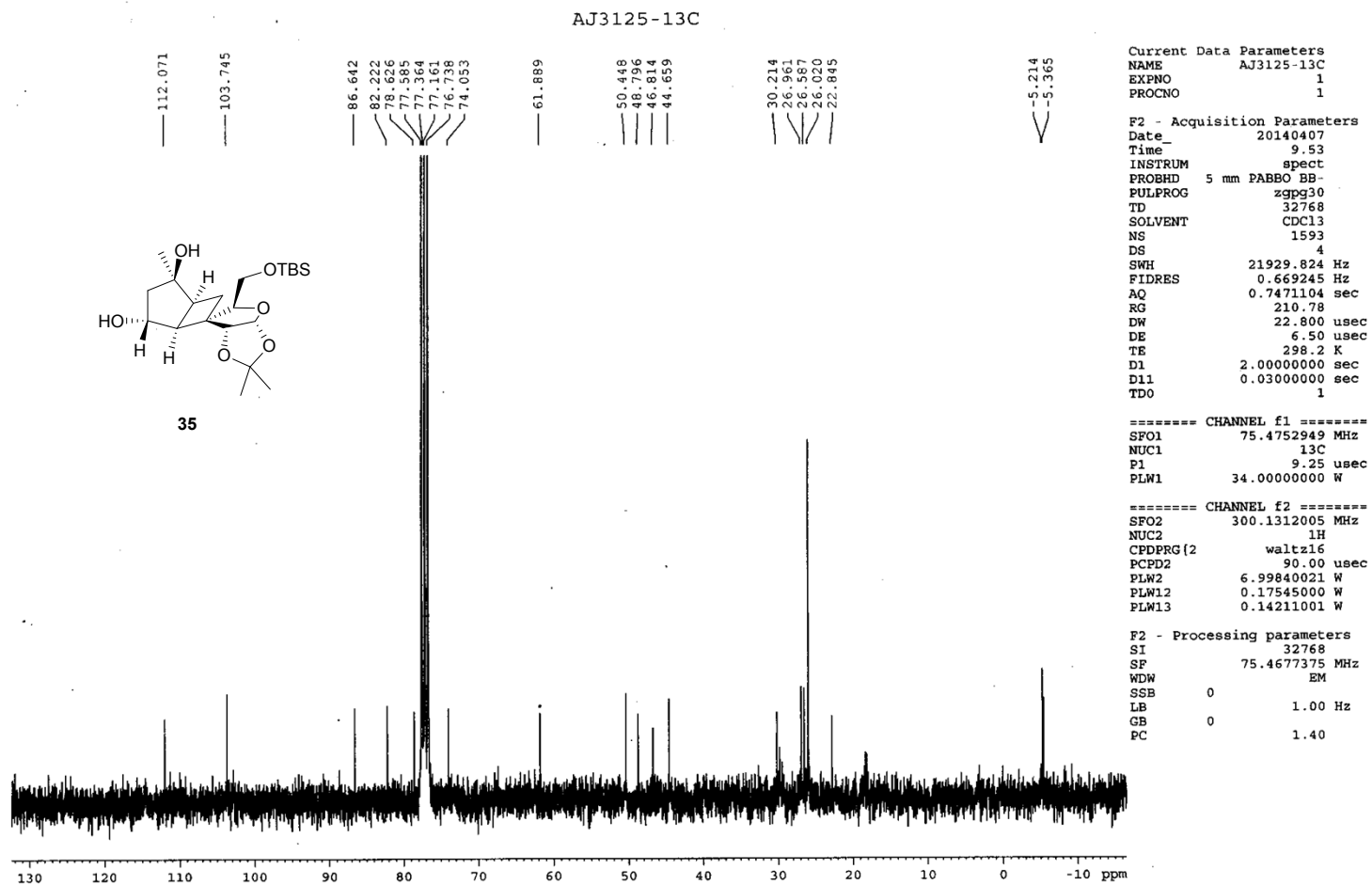
F2 - Processing parameters
SI       1024
SF       400.2000000 MHz
WDW      QSINE
SSB      0
LB       0 Hz
GB       0
PC       4.00

F1 - Processing parameters
SI       1024
MC2      QF
SF       400.2000000 MHz
WDW      QSINE
SSB      0
LB       0 Hz
GB       0
  
```

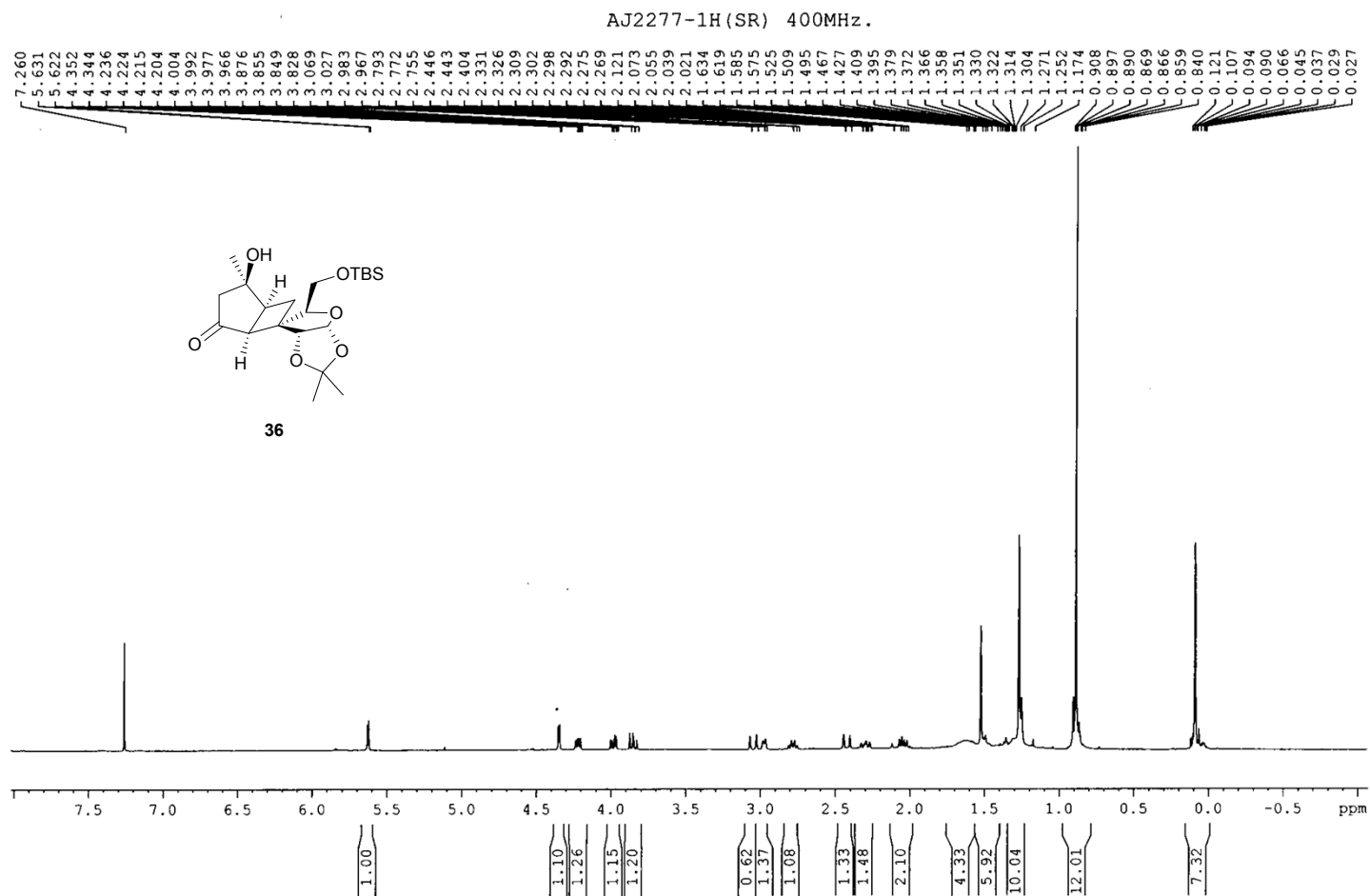

¹H NMR of Compound 35 (CDCl₃, 500 MHz)



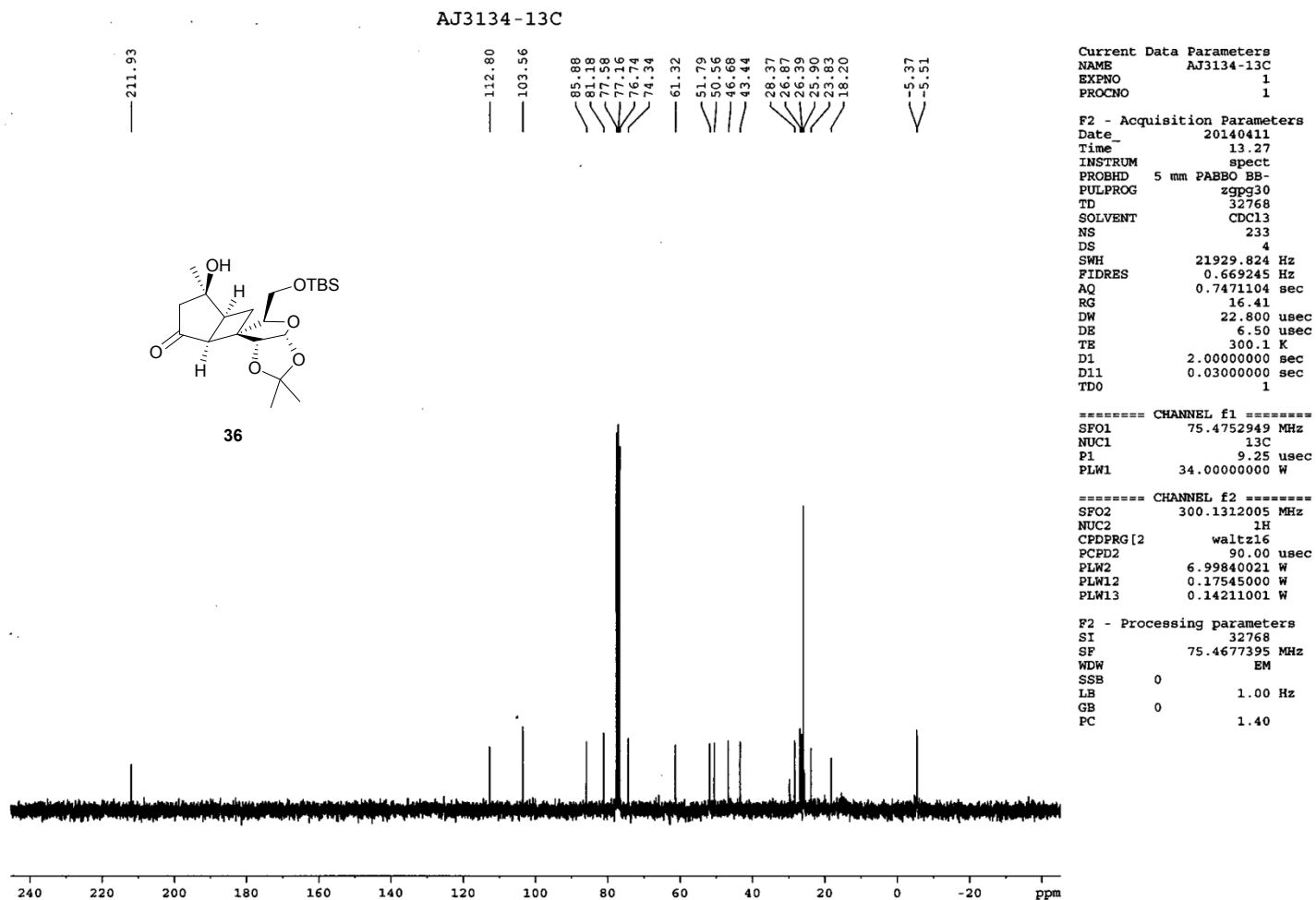
¹³C NMR of Compound 35 (CDCl₃, 75 MHz)



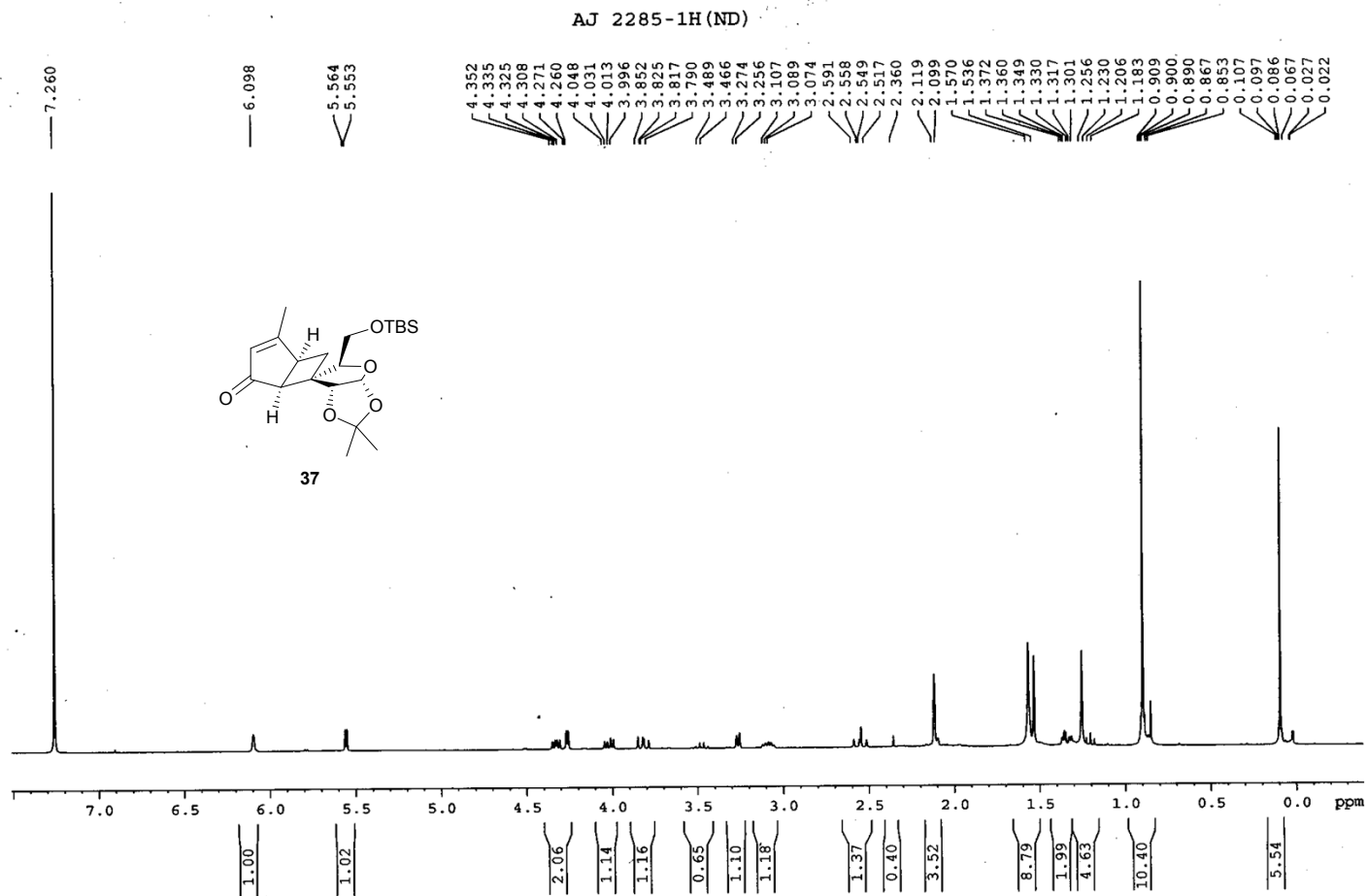
¹H NMR of Compound 36 (CDCl₃, 400 MHz)



¹³C NMR of Compound 33 (CDCl₃, 75 MHz)

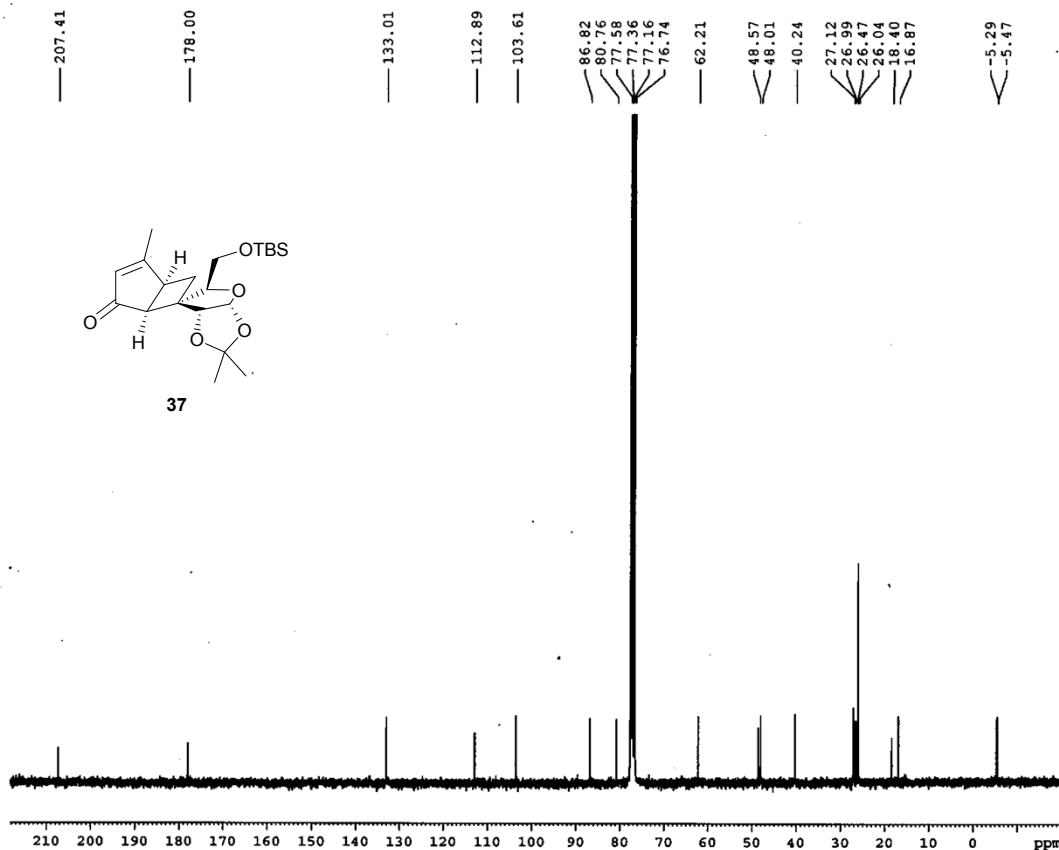


¹H NMR of Compound 37 (CDCl₃, 300 MHz)



¹³C NMR of Compound 37 (CDCl₃, 75 MHz)

AJ 2285-13C (ND)



Current Data Parameters
 NAME AJ 2285-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20130607
 Time 9.17
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 19706
 DS 4
 SWH 22727.273 Hz
 FIDRES 0.693581 Hz
 AQ 0.7208960 sec
 RG 210.78
 DW 22.000 usec
 DE 6.50 usec
 TE 300.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

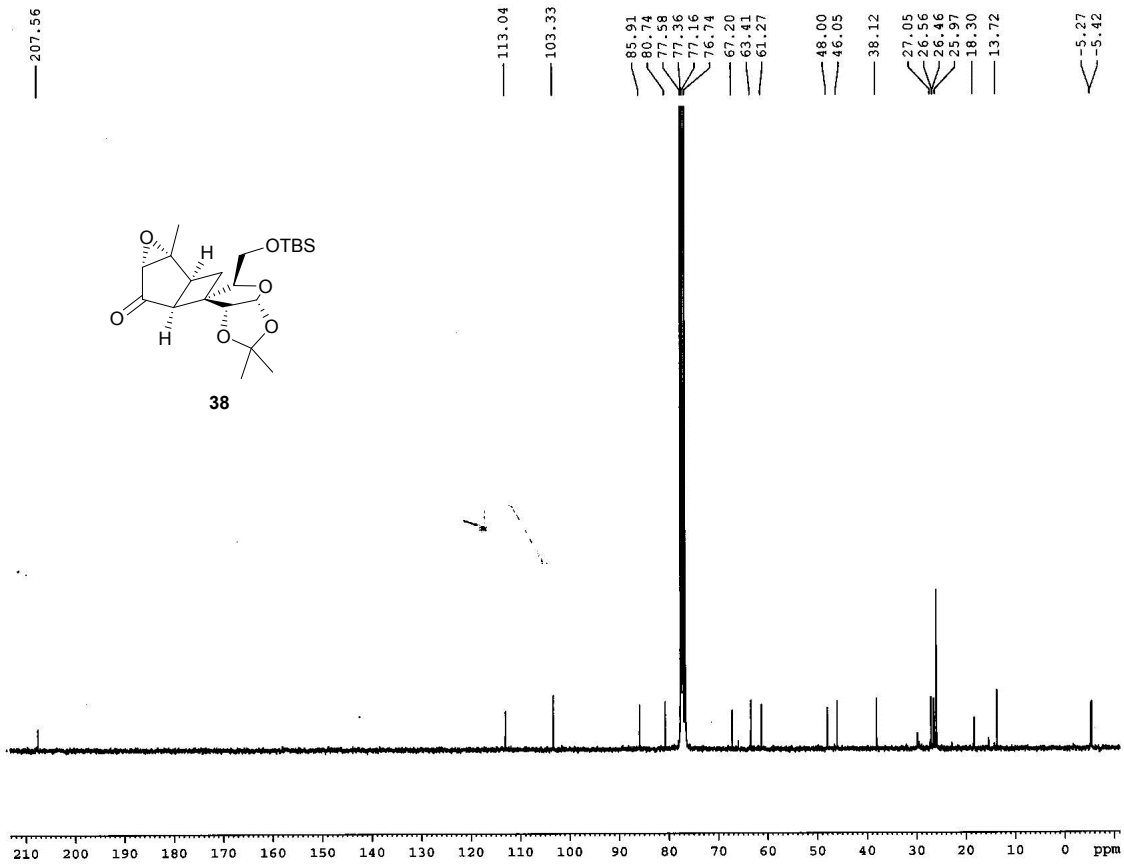
==== CHANNEL f1 =====
 SFO1 75.4752949 MHz
 NUC1 13C
 P1 9.25 usec
 PLW1 34.00000000 W

==== CHANNEL f2 =====
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 6.99840021 W
 PLW12 0.17545000 W
 PLW13 0.14211001 W

F2 - Processing parameters
 SI 32768
 SF 75.4677369 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹³C NMR of Compound 35 (CDCl₃, 75 MHz)

AJ 2290-13C (ND)



```

Current Data Parameters
NAME      AJ 2290-13C
EXPNO     1
PROCNO    1

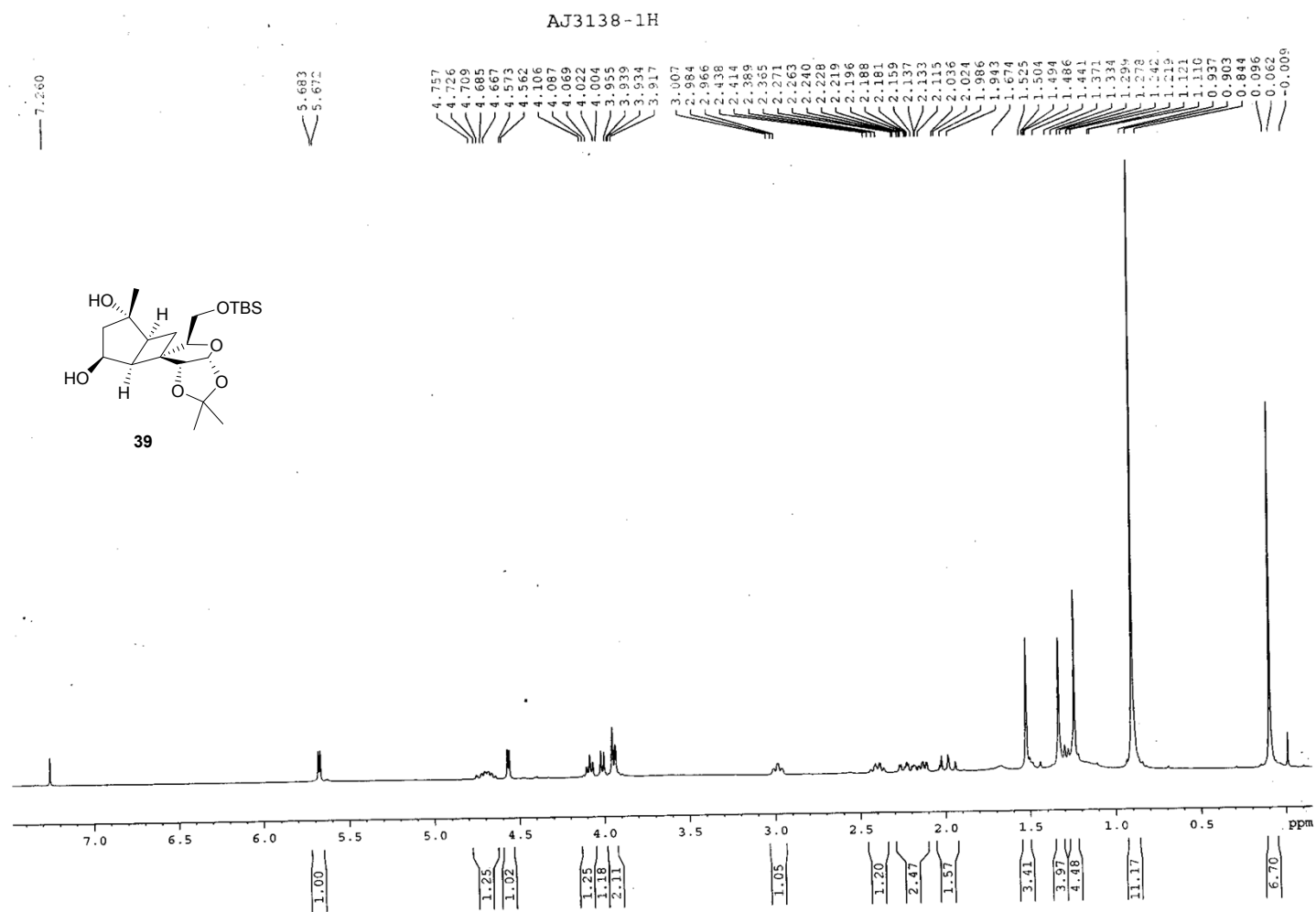
F2 - Acquisition Parameters
Date_     20130609
Time      9.37
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         57465
DS         4
SWH        22727.273 Hz
FIDRES     0.693581 Hz
AQ         0.7208960 sec
RG         210.78
DW         22.000 usec
DE         6.50 usec
TE         298.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1       75.4752949 MHz
NUC1        13C
P1          9.25 usec
PLW1       34.00000000 W

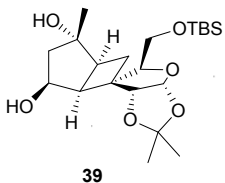
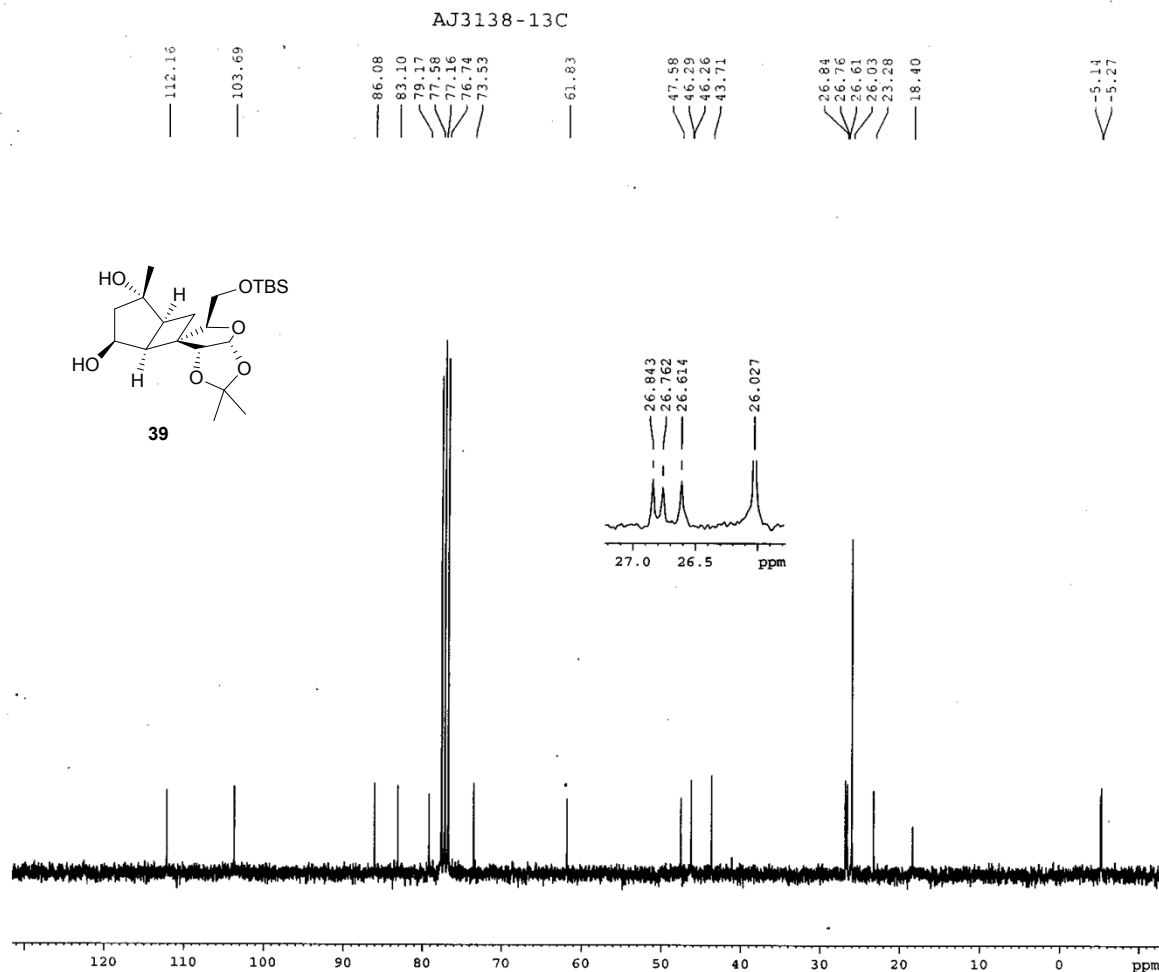
===== CHANNEL F2 =====
SFO2       300.1312005 MHz
NUC2         1H
CPDPRG[2]   waltz16
PCPD2       90.00 usec
PLW2        6.99840021 W
PLW12       0.17545000 W
PLW13       0.14211001 W

F2 - Processing parameters
SI         32768
SF         75.4677370 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```


¹H NMR of Compound 39 (CDCl₃, 300 MHz)



¹³C NMR of Compound 39 (CDCl₃, 75 MHz)



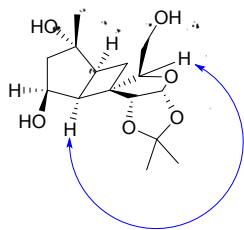
Current Data Parameters
 NAME AJ3138-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140416
 Time 12.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 334
 DS 4
 SWH 21929.824 Hz
 FIDRES 0.669245 Hz
 AQ 0.7471104 sec
 RG 16.41
 DW 22.800 usec
 DE 6.50 usec
 TE 298.7 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

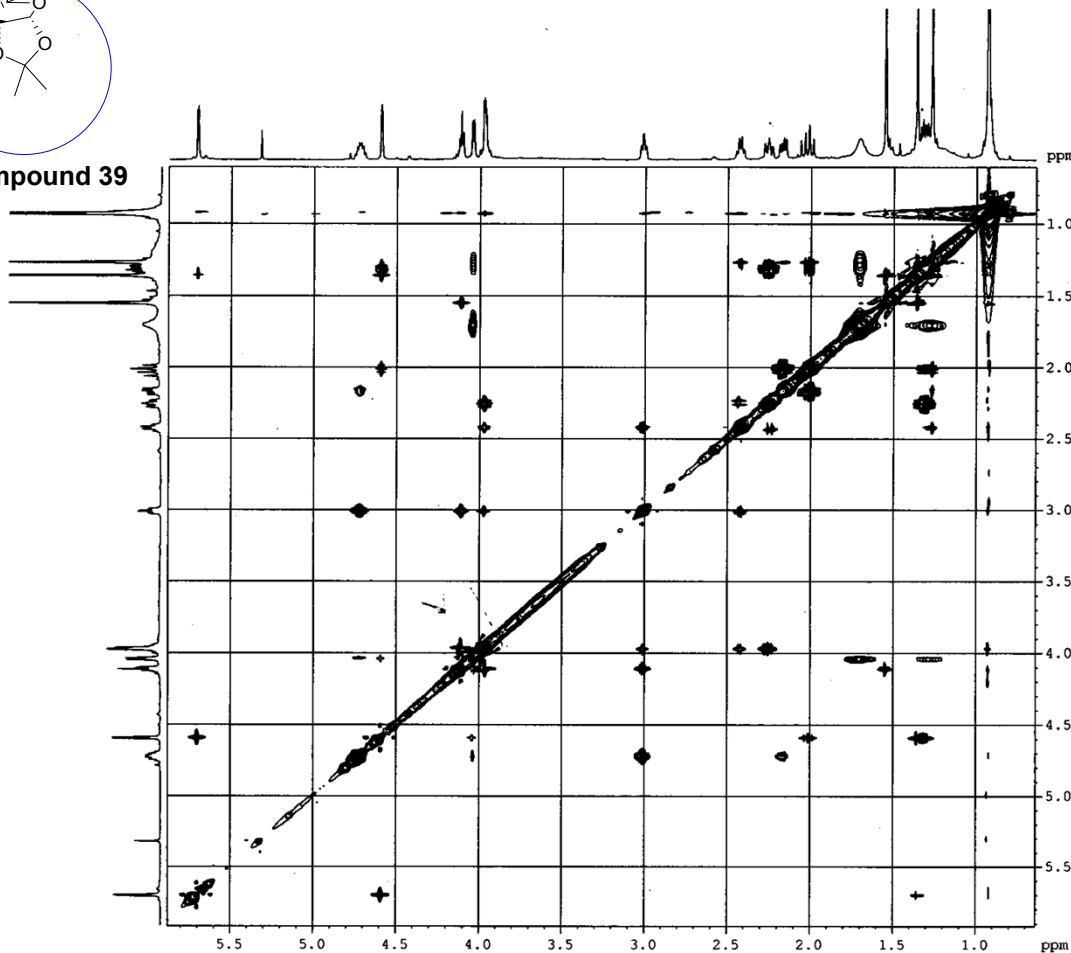
===== CHANNEL f1 =====
 SFO1 75.4752949 MHz
 NUC1 13C
 P1 9.25 usec
 PLW1 34.00000000 W

===== CHANNEL f2 =====
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 6.99840021 W
 PLW12 0.17545000 W
 PLW13 0.14211001 W

F2 - Processing parameters
 SI 32768
 SF 75.4677382 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



NOESY of compound 39



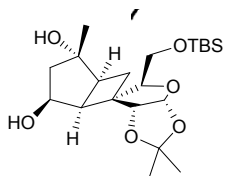
AJ3138A-noesy (SR)

```

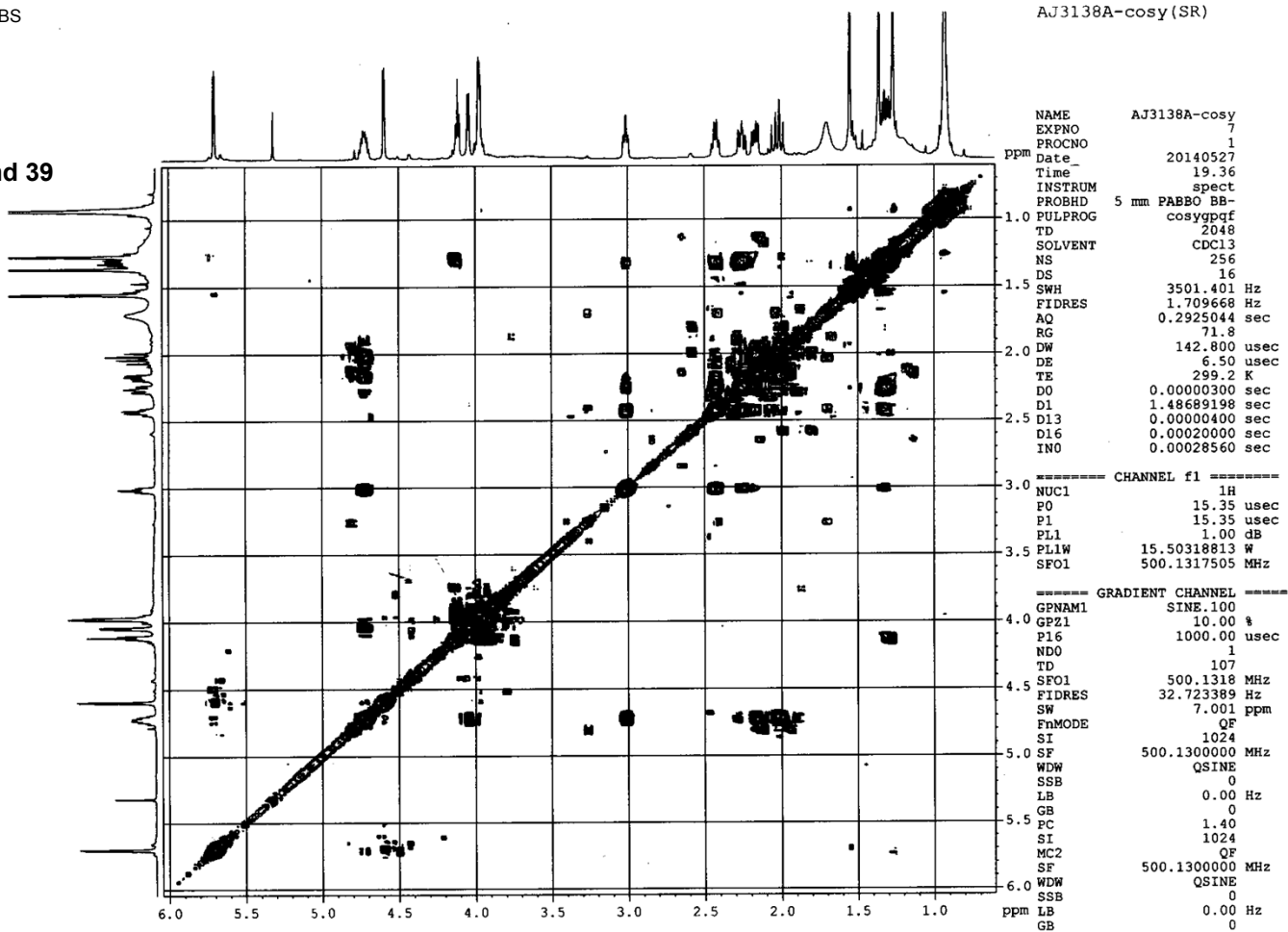
NAME      AJ3138A-noesy
EXPNO     8
PROCNO    1
Date_     20140528
Time      19.56
INSTRUM   spect
PROBHD    5 mm PABBO BB
PULPROG   noesyph
TD         2048
SOLVENT   CDCl3
NS         80
DS         16
SWH        3501.401 Hz
FIDRES     1.709668 Hz
AQ         0.2925044 sec
RG         64
DW         142.800 usec
DE         6.50 usec
TE         299.2 K
DO         0.00012325 sec
D1         2.00000000 sec
D8         0.75000000 sec
INO        0.00028560 sec
  
```

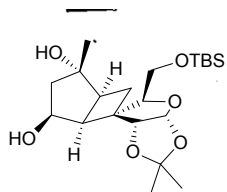
```

----- CHANNEL f1 -----
NUC1      1H
P1        15.35 usec
PL1       1.00 dB
PL1W      15.50318813 W
SFO1      500.1317505 MHz
ND0       1
TD         202
SFO1      500.1318 MHz
FIDRES    17.333775 Hz
SW         7.001 ppm
FnMODE    States-TPPI
SI         1024
SF         500.1300000 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         0.80
SI         1024
MC2       States-TPPI
SF         500.1300000 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
  
```

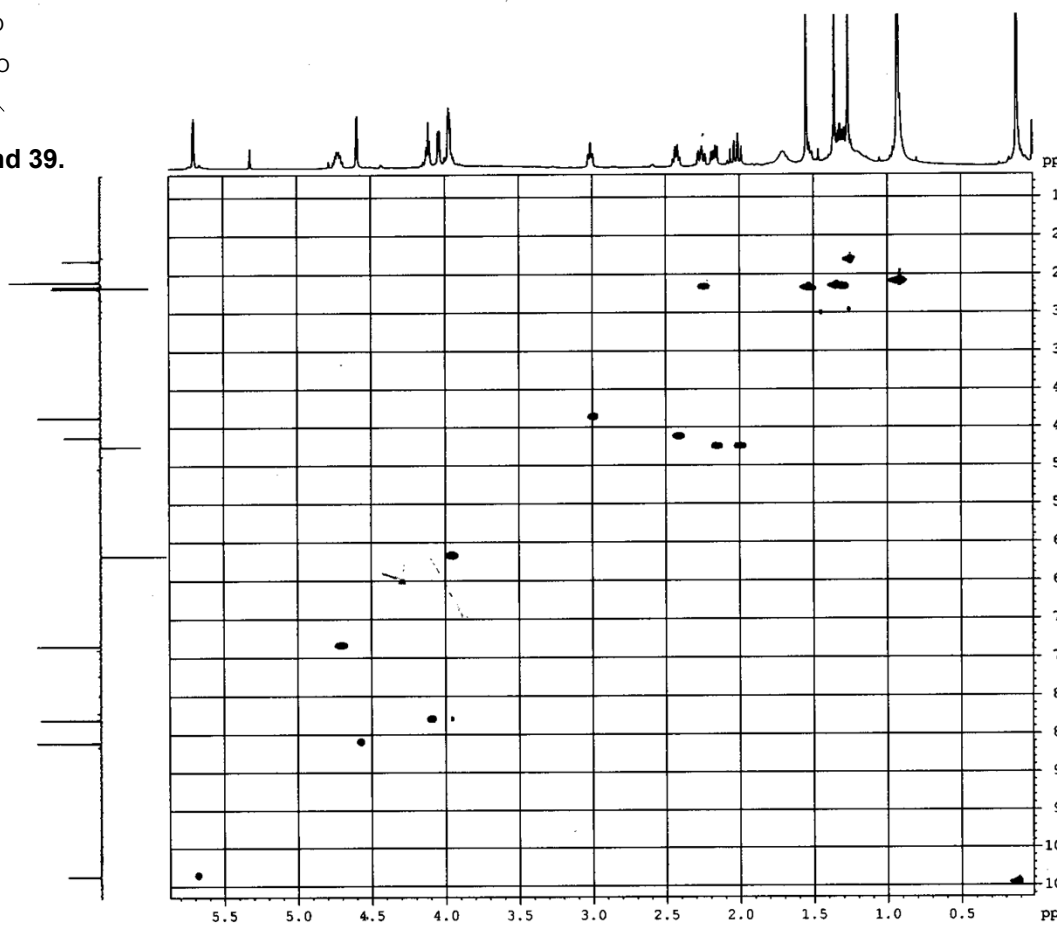


COSY of compound 39





HSQC of compound 39.



AJ3138A1-HSQC (SR)

```

NAME      AJ3138A1-HSQC
EXPNO     7
PROCNO    1
Date_     20140604
Time      20.08
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   hsqcetgps12
TD         1024
SOLVENT   CDCl3
NS         128
DS         16
SWH        3501.401 Hz
FIDRES     3.419337 Hz
AQ         0.1462772 sec
RG         57
DW         142.800 use
DE         6.50 use
TE         300.3 K
CNST2     145.0000000
DO         0.00000300 sec
D1         1.5000000 sec
D4         0.00172414 sec
D11        0.03000000 sec
D13        0.00000400 sec
D16        0.00020000 sec
D24        0.00086207 sec
IN0        0.00003615 sec
ZGPTNS

===== CHANNEL f1 =====
NUC1       1H
P1         15.35 use
P2         30.70 use
P28        0.00 use
PL1        1.00 dB
PL1W       15.50318813 W
SFO1       500.1317505 MHz

===== CHANNEL f2 =====
CPDPRG2    garp4
NUC2       13C
P3         9.88 use
P4         19.76 use
PCPD2      70.00 use
PL2        0.50 dB
PL12       17.50 dB
PL2W       82.16106415 W
PL12W      1.63932884 W
SFO2       125.7647057 MHz

===== GRADIENT CHANNEL =====
GPNAM1     SINE.100
GPNAM2     SINE.100
GPNAM3     SINE.100
GPNAM4     SINE.100
GPZ1       80.00 %
GPZ2       20.10 %
  
```