

# **A Concise Synthesis of the Neo-Clerodane Skeleton of Teucrolivin A Using An Oxy-Cope/Claisen/ene Cascade: Supporting Information**

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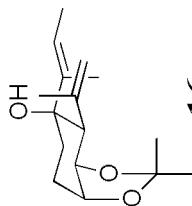
## **General Experimental**

All reactions were performed under argon in flame-dried glassware equipped with a magnetic stirbar and a rubber septum unless otherwise indicated. Solvents used were freshly distilled prior to use: ether, THF and 1,2-dimethoxyethane (DME) over sodium and benzophenone; dichloromethane, toluene and DMF over calcium hydride. All other commercial reagents were used without purification.

Microwave reactions were performed using a CEM Model ESP-1500 Plus microwave oven equipped with a pressure monitoring device and an EST-300 Plus fiber optic temperature probe. The reaction vessel was a quartz tube, and in each case was added a carboflon<sup>TM</sup> to aid in the absorption of microwave radiation.

Reactions were monitored by TLC analysis using glass plates precoated (250  $\mu\text{m}$  thickness) with silica gel 60 F<sub>254</sub> (E. Merck). TLC plates were viewed using UV light, *p*-anisaldehyde staining solution, phosphomolybdic acid staining solution or potassium permanganate staining solution. Flash chromatography was carried out on 230-400 mesh silica gel 60.

<sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on Bruker Avance 300 MHz, Bruker Avance 500 MHz or Varian INOVA 500 MHz spectrometers in the specified deuterated solvent. IR spectra were recorded on a Bomen Michaelson 100 FTIR spectrometer. HRMS spectra were obtained using a Kratos Analytical Concept spectrometer, and melting points were recorded using a Gallenkamp P1106G Melting Point Apparatus.



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S3

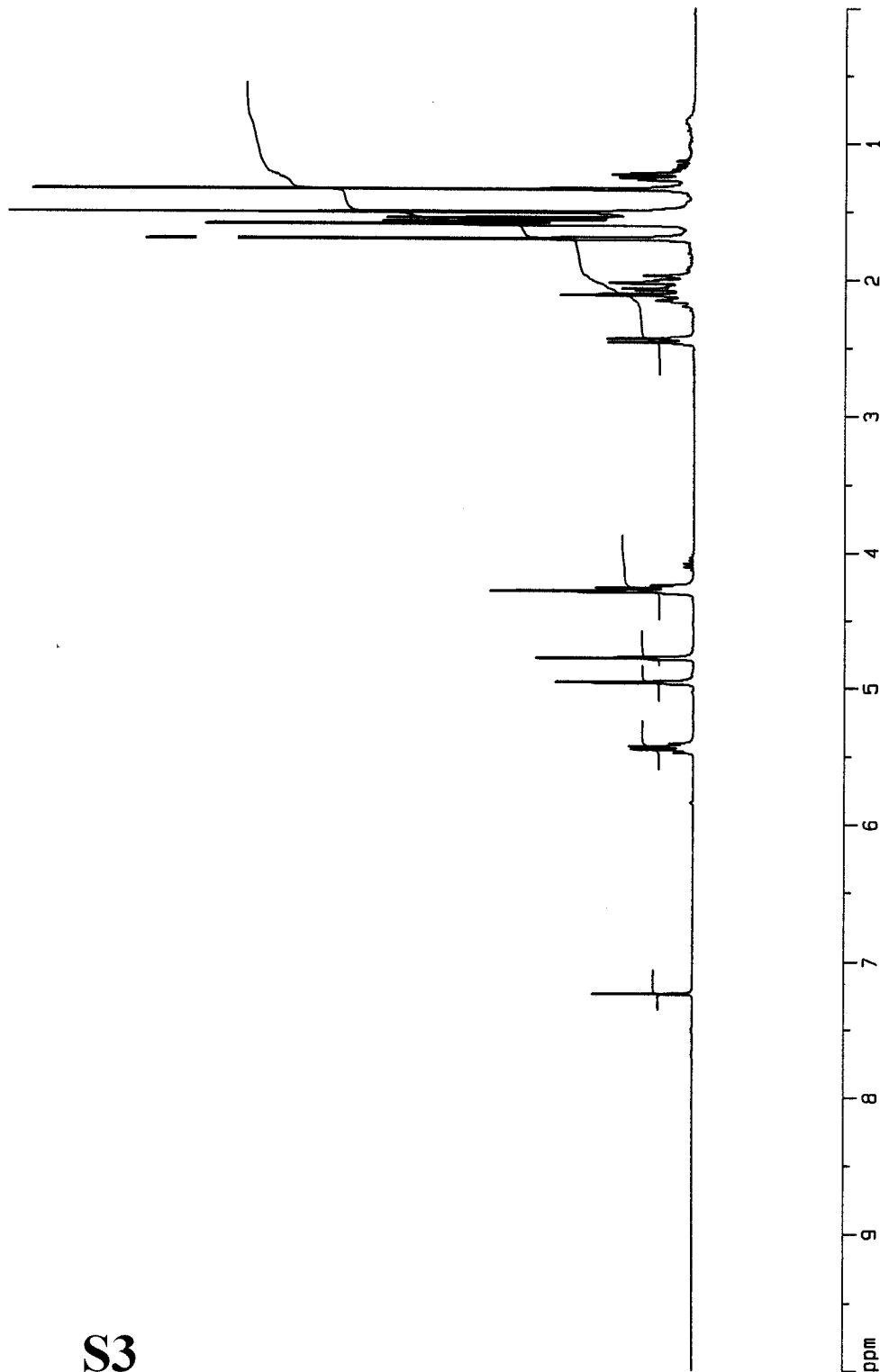
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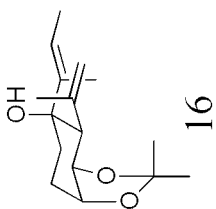
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1D NMR plot parameters  
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 CY 10.00 cm  
 F1P 10.000 ppm  
 F1 3001.30 Hz  
 F2P 0.000 ppm  
 F2 0.00 Hz  
 PPMCM 0.50000 ppm/cm  
 HZCM 150.06500 Hz/cm



<sup>13</sup>C with proton decoupling



```

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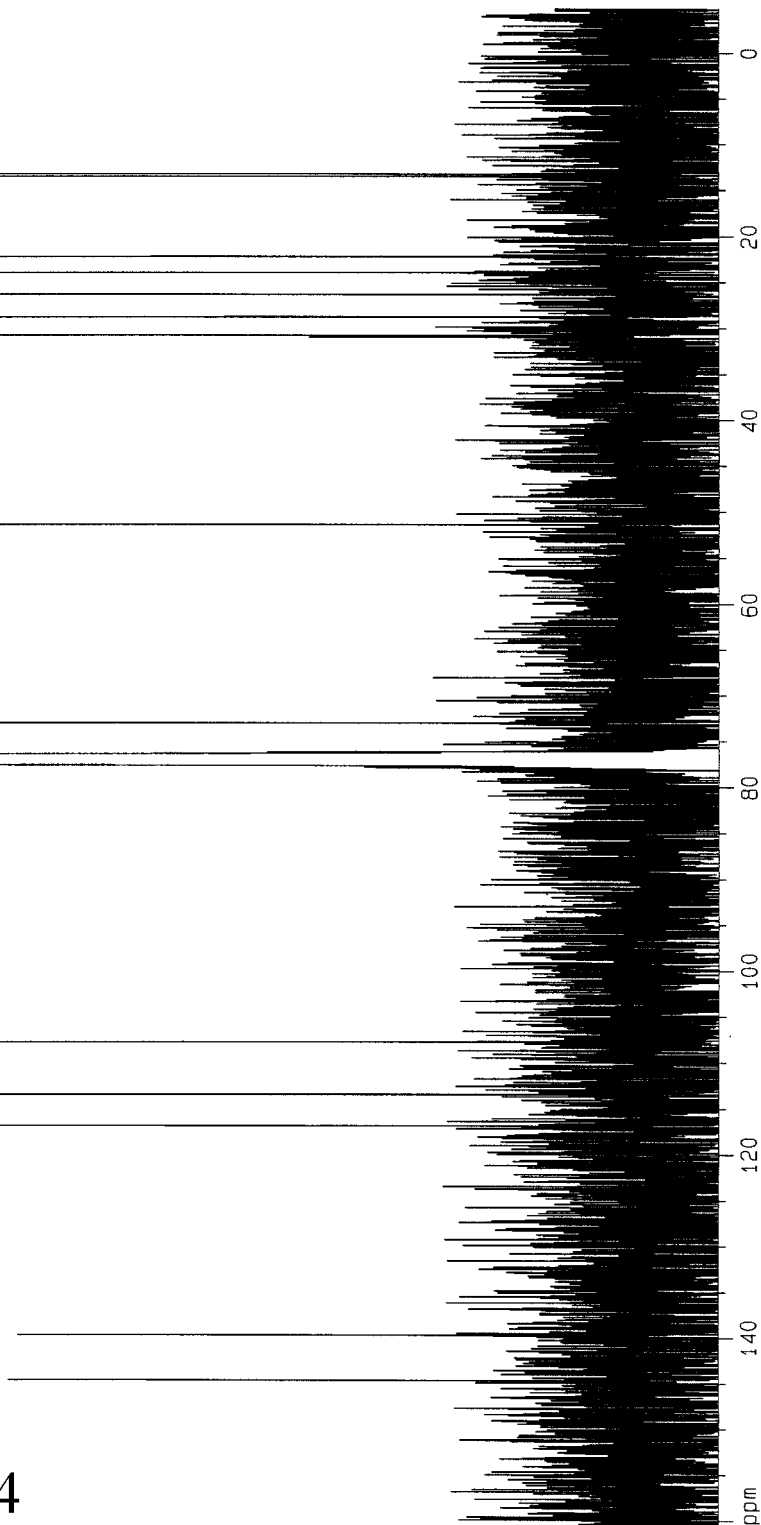
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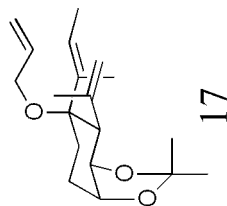
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CY         12.50 cm
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F1         12108.21 Hz
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```





S5

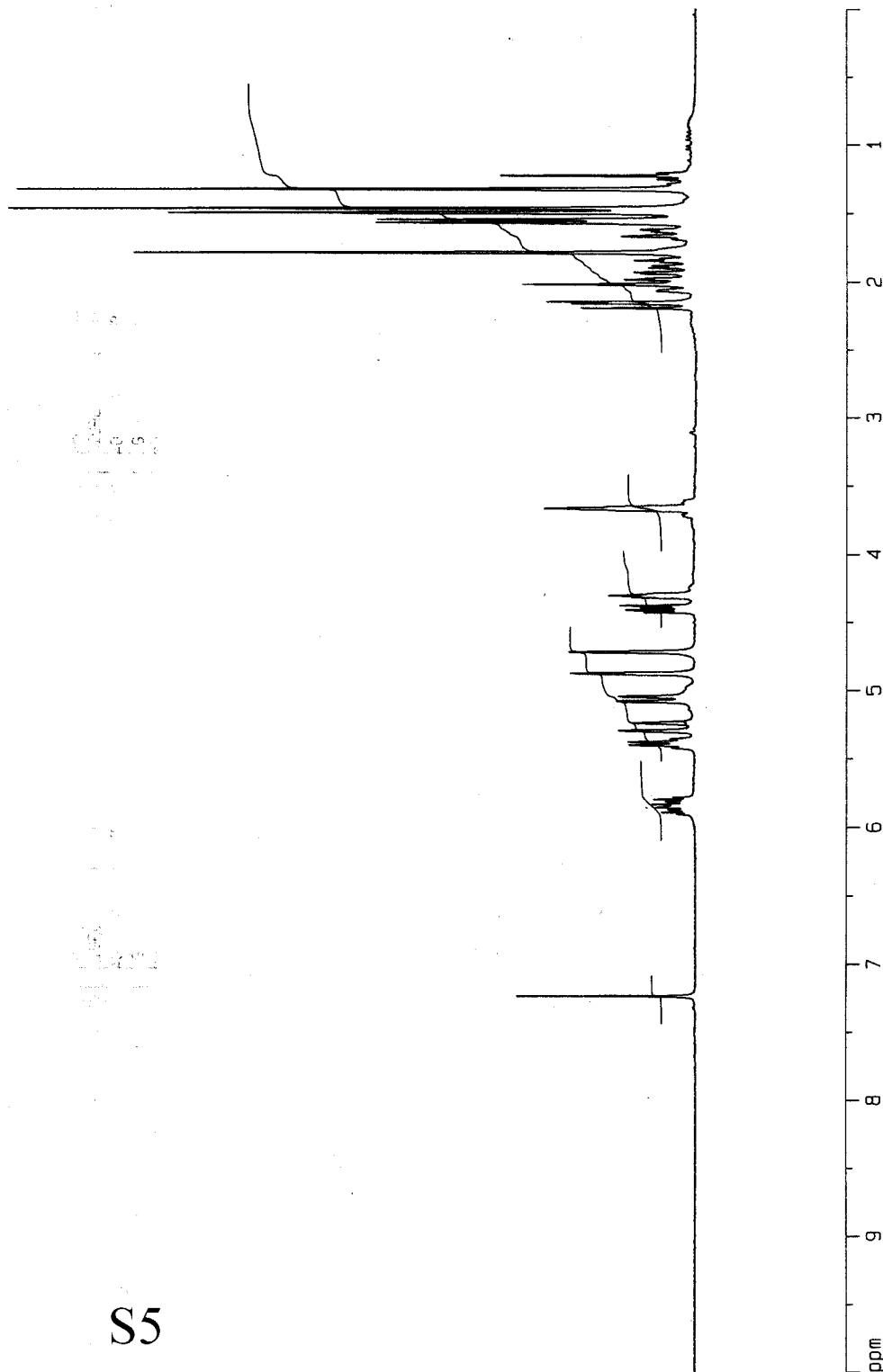
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TD 30720  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 5081.301 Hz  
FIDRES 0.165407 Hz  
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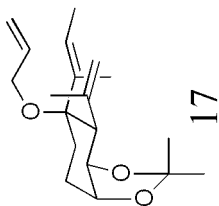
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1D NMR plot parameters  
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<sup>13</sup>C with proton decoupling



```

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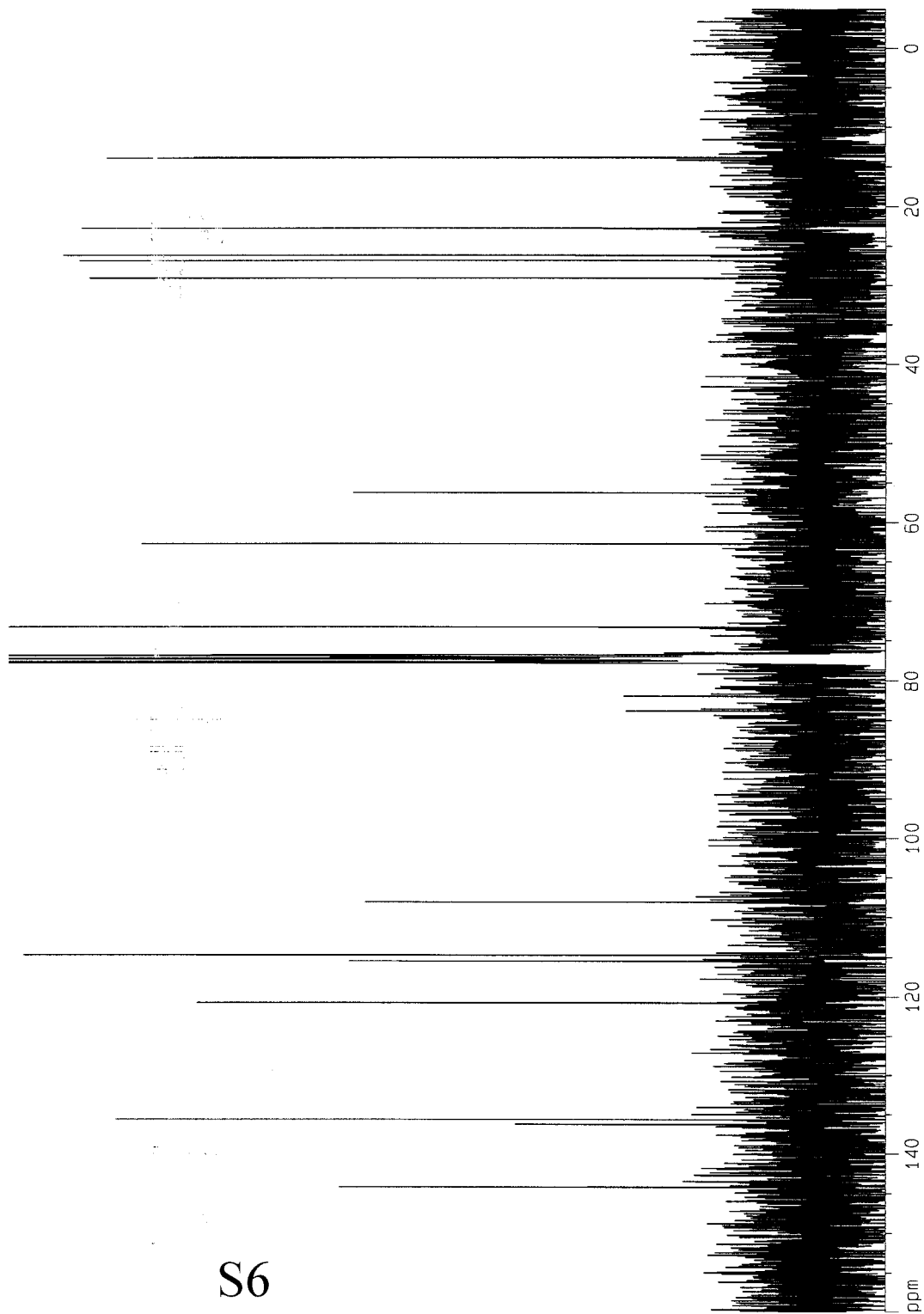
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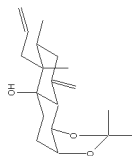
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PCPD2      70.00 usec
PL2        -3.00 dB
PL12       13.48 dB
PL13       15.63 dB
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F2 - Processing parameters
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SF         75.4677190 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         20.00 cm
CY         12.50 cm
F1P        160.000 ppm
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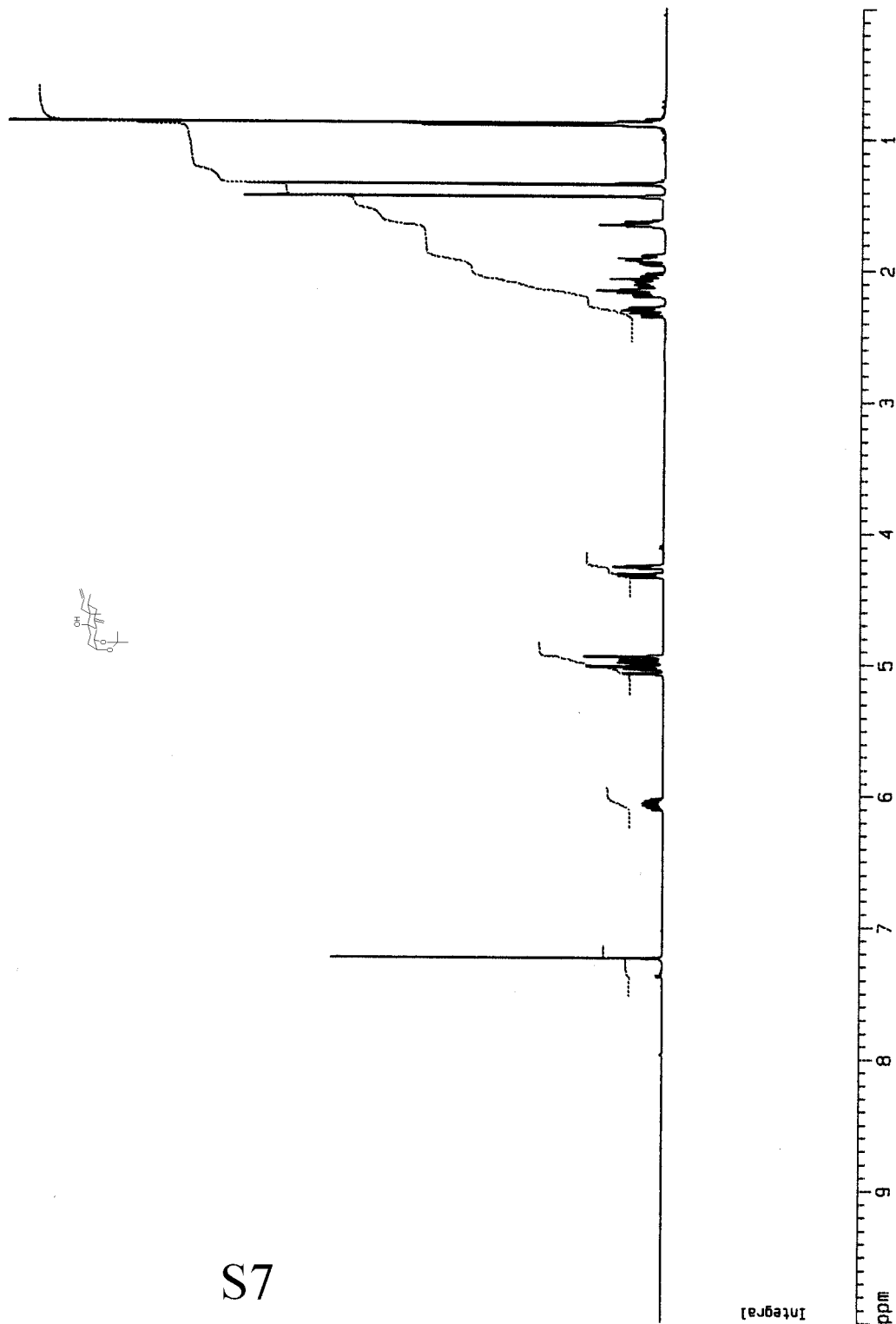




1H NMR

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S7



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PULPROG zg30  
TD 65536  
SOLVENT Acetone  
NS 16  
DS 0  
SMH 7440.476 Hz  
FIDRES 0.113533 Hz  
AQ 4.4040694 sec  
RG 203.2  
DN 67.200 usec  
DE 6.00 usec  
TE 300.0 K  
D1 0.01000000 sec

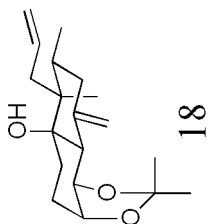
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GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
CY 10.00 cm  
FIP 10.000 ppm  
F1 5001.30 Hz  
F2 0.000 ppm  
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Integral

<sup>13</sup>C with proton decoupling



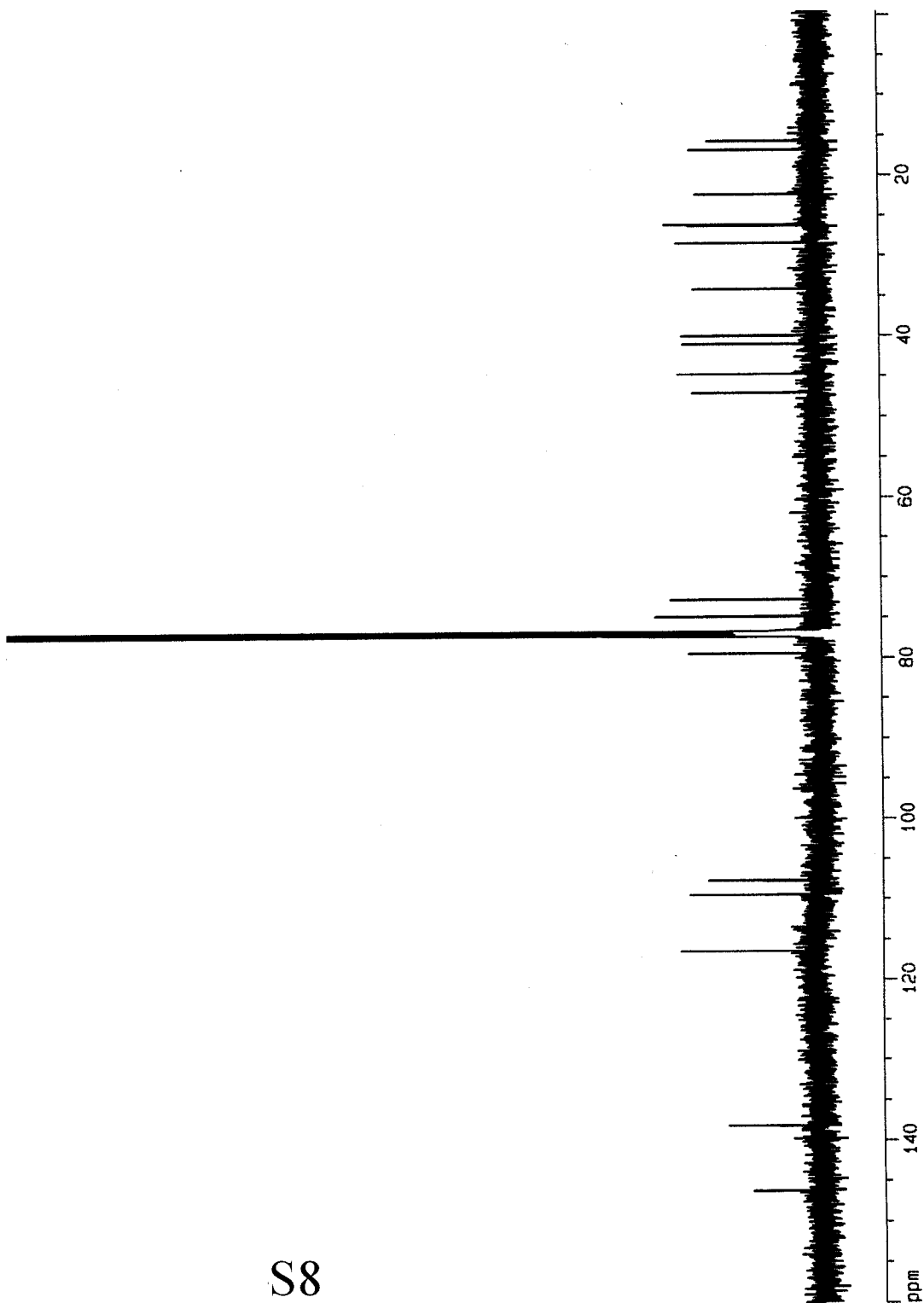
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SOLVENT CDCl<sub>3</sub>  
NS 1024  
DS 0  
SWH 30030.029 Hz  
FIDRES 0.458222 Hz  
AQ 1.0912244 sec  
RG 3251  
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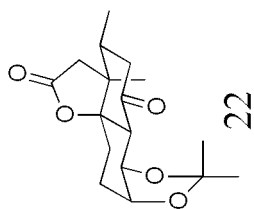
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PL12 20.64 dB  
PL13 23.56 dB  
SF02 500.1320005 MHz

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EXPNO 1  
PROCNO 1

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PULPROG zg30  
TD 30720  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 5081.301 Hz  
FIDRES 0.165407 Hz  
AQ 3.0228980 sec  
RG 724.1  
DM 98.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

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

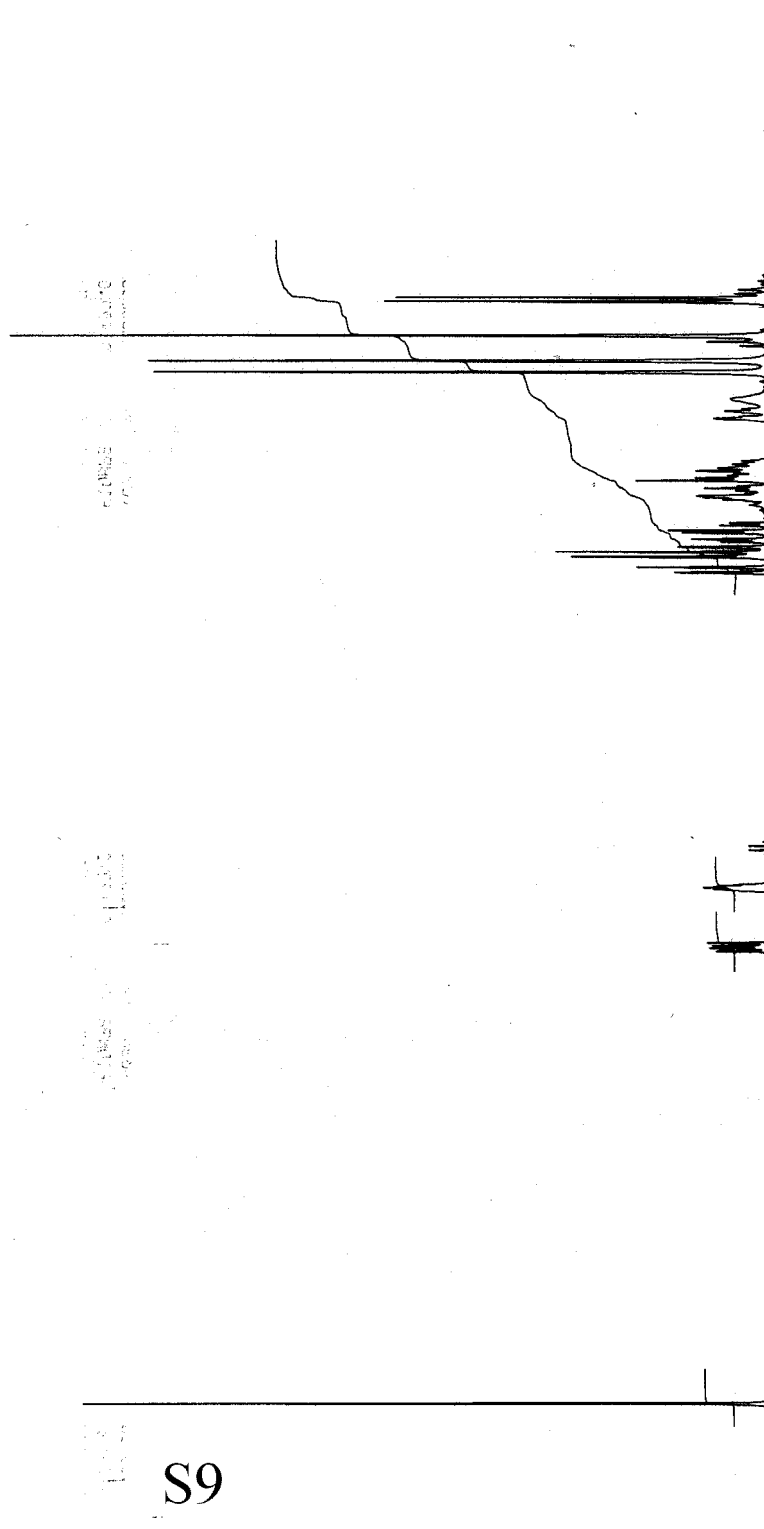
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SF01 300.1319477 MHz

F2 - Processing parameters

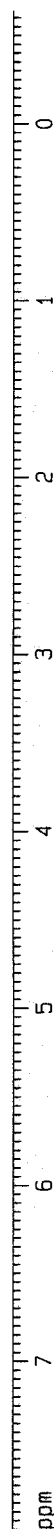
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1D NMR plot parameters

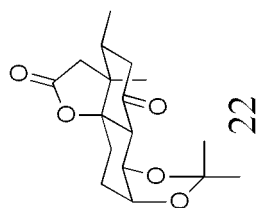
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F1 2386.28 Hz  
F2P -0.640 ppm  
F2 -192.18 Hz  
PPMCM 0.42956 ppm/cm  
HZCM 128.92288 Hz/cm



S9



13C with proton decoupling



S10

Current Data Parameters  
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EXPNO 4  
PROCNO 1

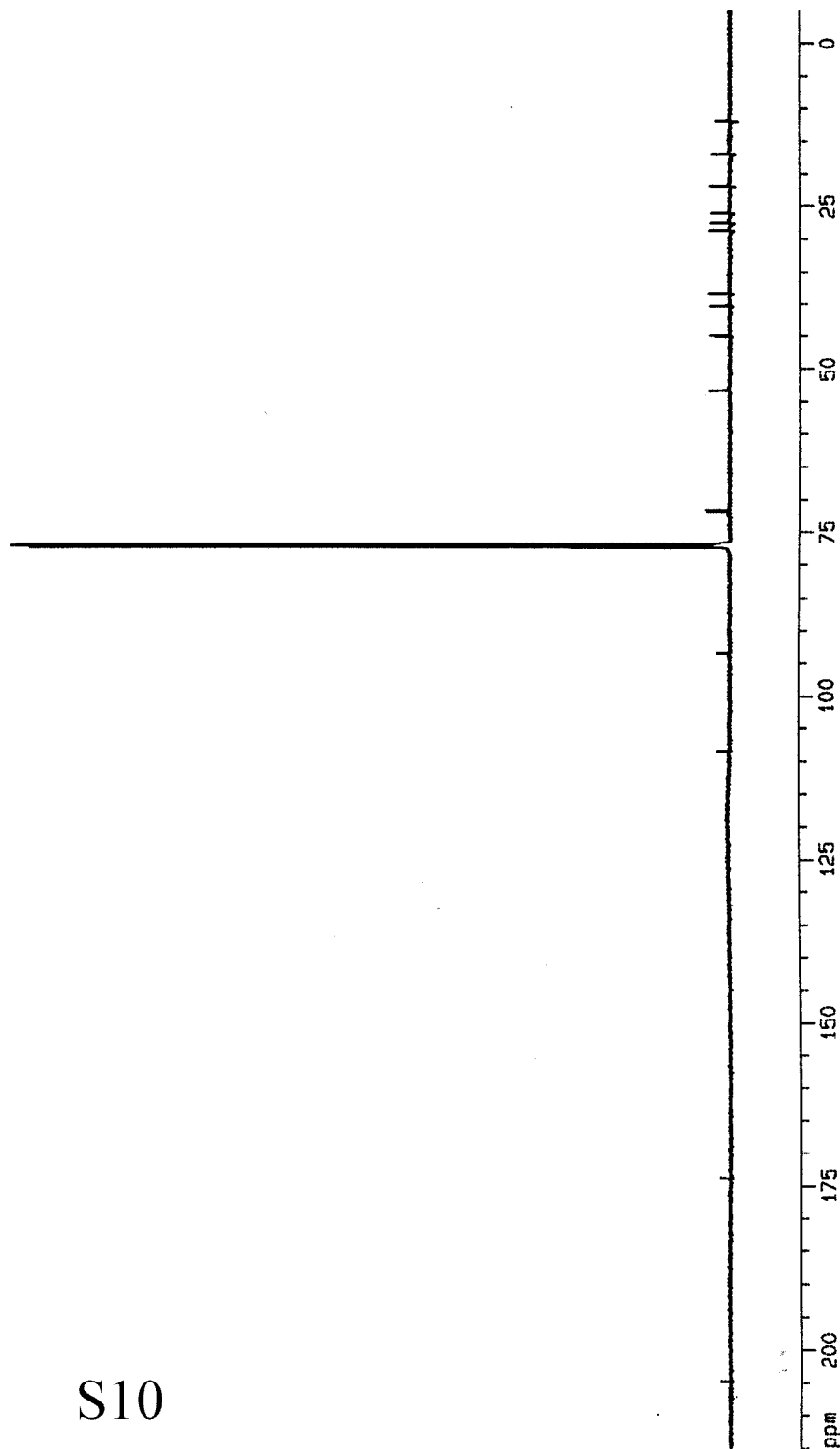
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TD 65536  
SOLVENT CDC13  
NS 10240  
DS 0  
SMH 30030.029 Hz  
FIDRES 0.458222 Hz  
AQ 1.0912244 sec  
RG 3251  
DM 16.650 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

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PL1 -3.00 dB  
SF01 125.7703643 MHz

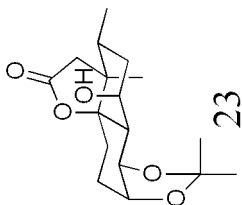
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PL12 20.64 dB  
PL13 23.56 dB  
SF02 500.1320005 MHz

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GB 0  
PC 1.00

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1H NMR



S11

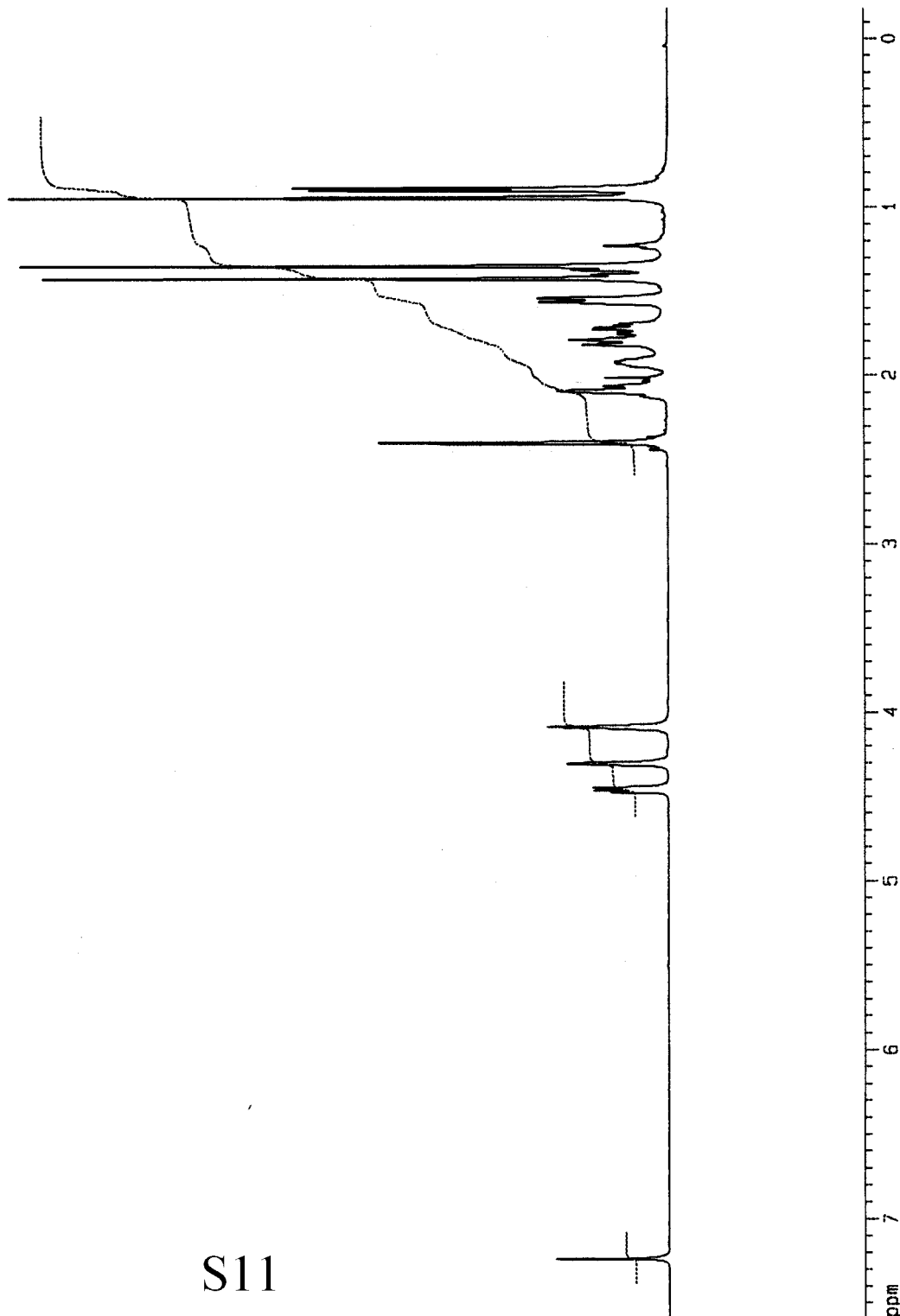
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SOLVENT Acetone  
NS 16  
DS 0  
SWH 7440.476 Hz  
FIDRES 0.113533 Hz  
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RG 101.6  
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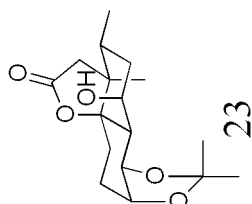
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WDW EM  
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GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
CY 10.00 cm  
FIP 7.587 ppm  
F1 3794.58 Hz  
F2p -0.179 ppm  
F2 -89.68 Hz  
PPMCM 0.38832 ppm/cm  
HZCM 194.21283 Hz/cm



<sup>13</sup>C with proton decoupling



S12

```

Current Data Parameters
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EXPNO     4
PROCNO    1

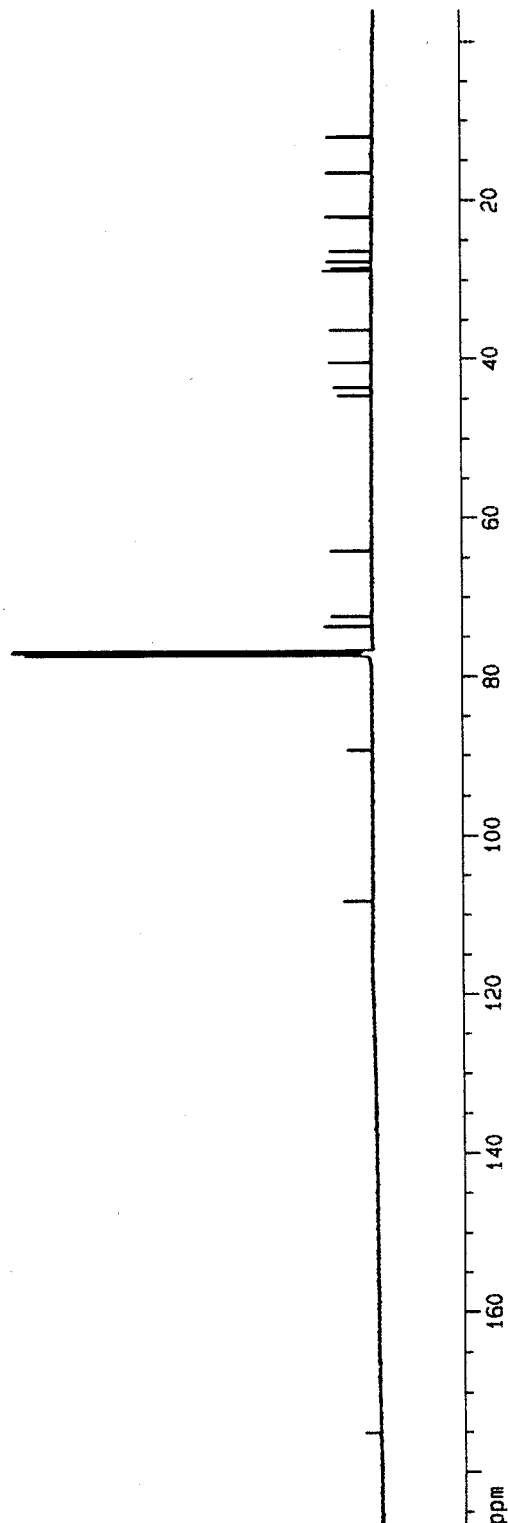
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SOLVENT    CDCl3
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SMH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912244 sec
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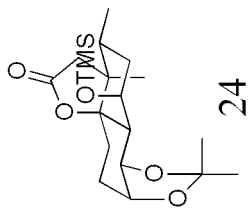
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NUC2        1H
PCPD2       80.00 usec
PL2         3.00 dB
PL12        20.64 dB
PL13        23.56 dB
SF02       500.1320005 MHz

F2 - Processing parameters
SI          32768
SF          125.7577850 MHz
MOM         EN
SSB         0
LB          1.00 Hz
GB          0
PC          1.00

ID NMR plot parameters
CX          20.00 cm
CY          5.00 cm
FIP         186.644 ppm
F1          23471.96 Hz
F2          -4.055 ppm
PPMCH       -509.93 Hz
HZCM        9.53496 ppm/cm
            1199.09485 Hz/cm
    
```





<sup>1</sup>H NMR

S13

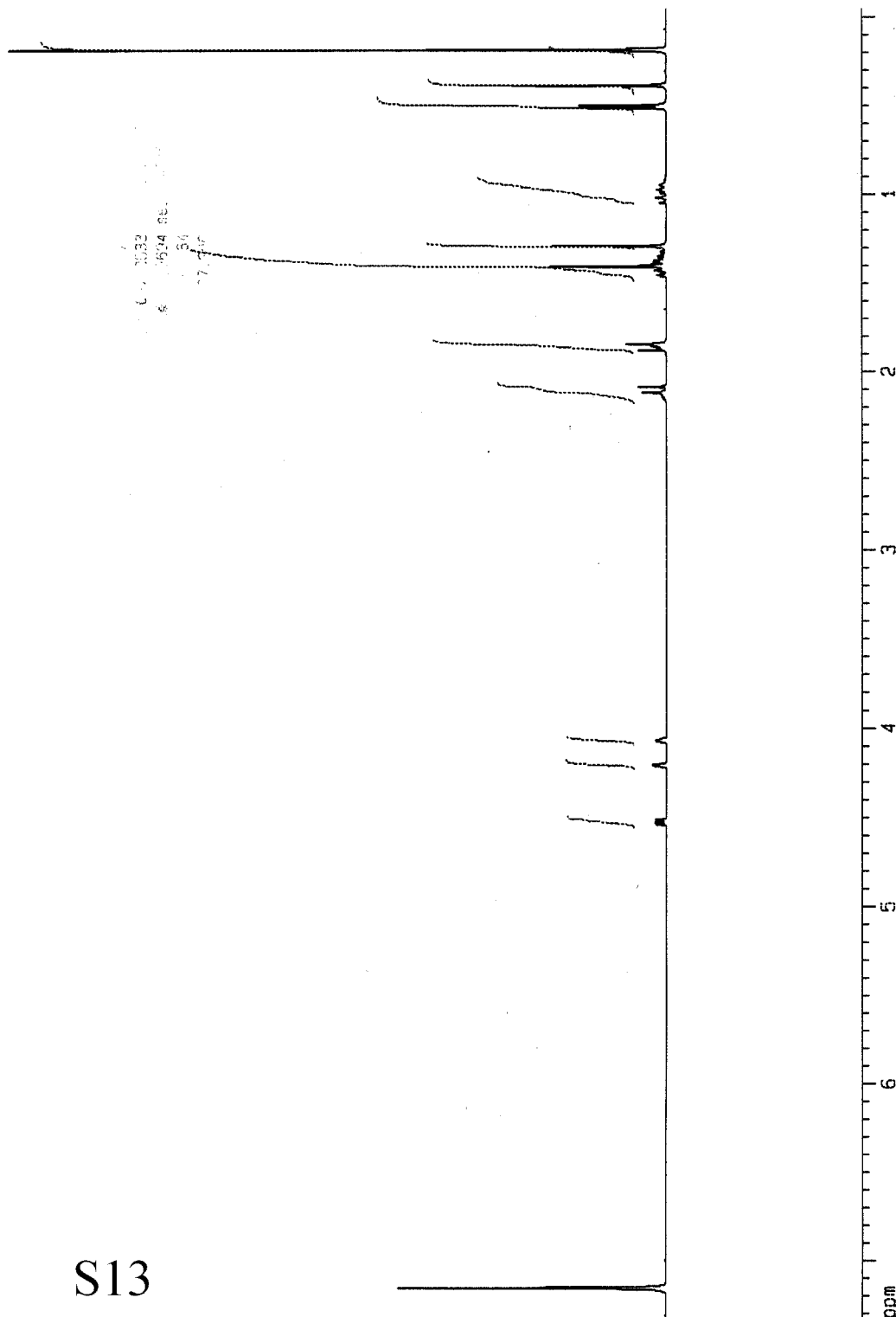
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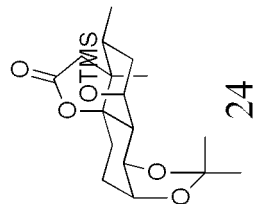
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 SOLVENT Acetone  
 NS 16  
 DS 0  
 SWH 7440.476 Hz  
 FIDRES 0.113533 Hz  
 AQ 4.4040694 sec  
 RG 64  
 DW 67.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 0.01000000 sec

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 PL1 3.00 dB  
 SF01 500.1327766 MHz

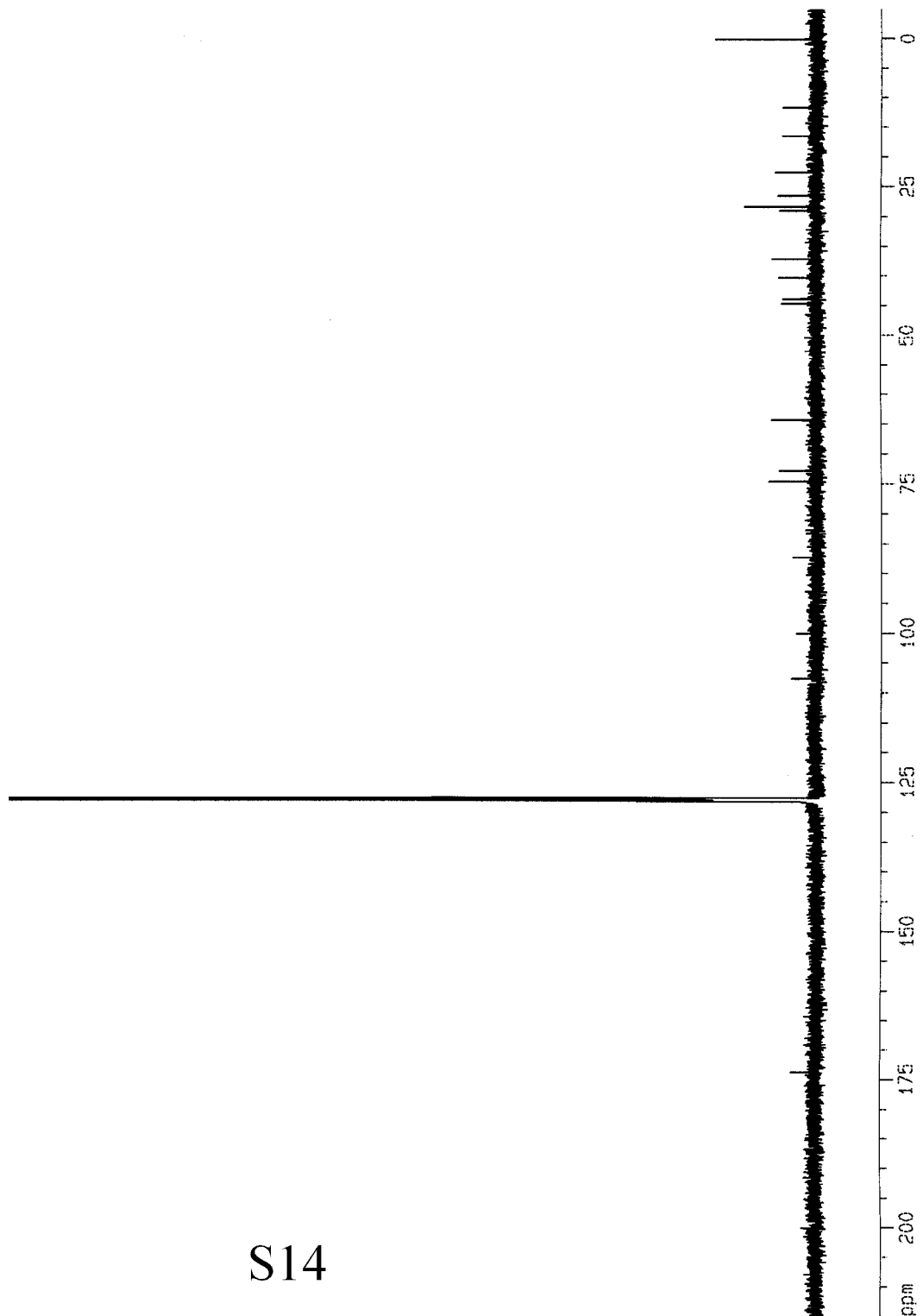
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1D NMR plot parameters  
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 CY 10.00 cm  
 F1P 7.330 ppm  
 F1 3666.05 Hz  
 F2P -0.049 ppm  
 F2 -24.37 Hz  
 PPMCM 0.36895 ppm/cm  
 HZCM 184.52081 Hz/cm





<sup>13</sup>C with proton decoupling



```

Current Data Parameters
NAME      sa-240
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20040421
Time      17.01
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PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         293
DS         0
SMH       30030.029 Hz
FIDRES    0.456222 Hz
AQ         1.0912244 sec
RG         456.1
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DE         6.00 usec
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d12        0.0002000 sec

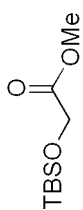
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PL1        -3.00 dB
SF01       125.7703643 MHz

===== CHANNEL f2 =====
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NUC2       1H
PCPD2      80.00 usec
PL2         3.00 dB
PL12       20.64 dB
PL13       23.56 dB
SF02       500.1320005 MHz

F2 - Processing parameters
SI         32768
SF         125.7577654 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00

1D NMR plot parameters
CX         20.00 cm
CY         50.00 cm
F1P        215.000 ppm
F1         27037.92 Hz
F2P        -5.000 ppm
F2         -628.79 Hz
PNUC1      11.00000 ppm/cm
HZCN       1383.33545 Hz/cm
  
```

1H NMR



27

S15

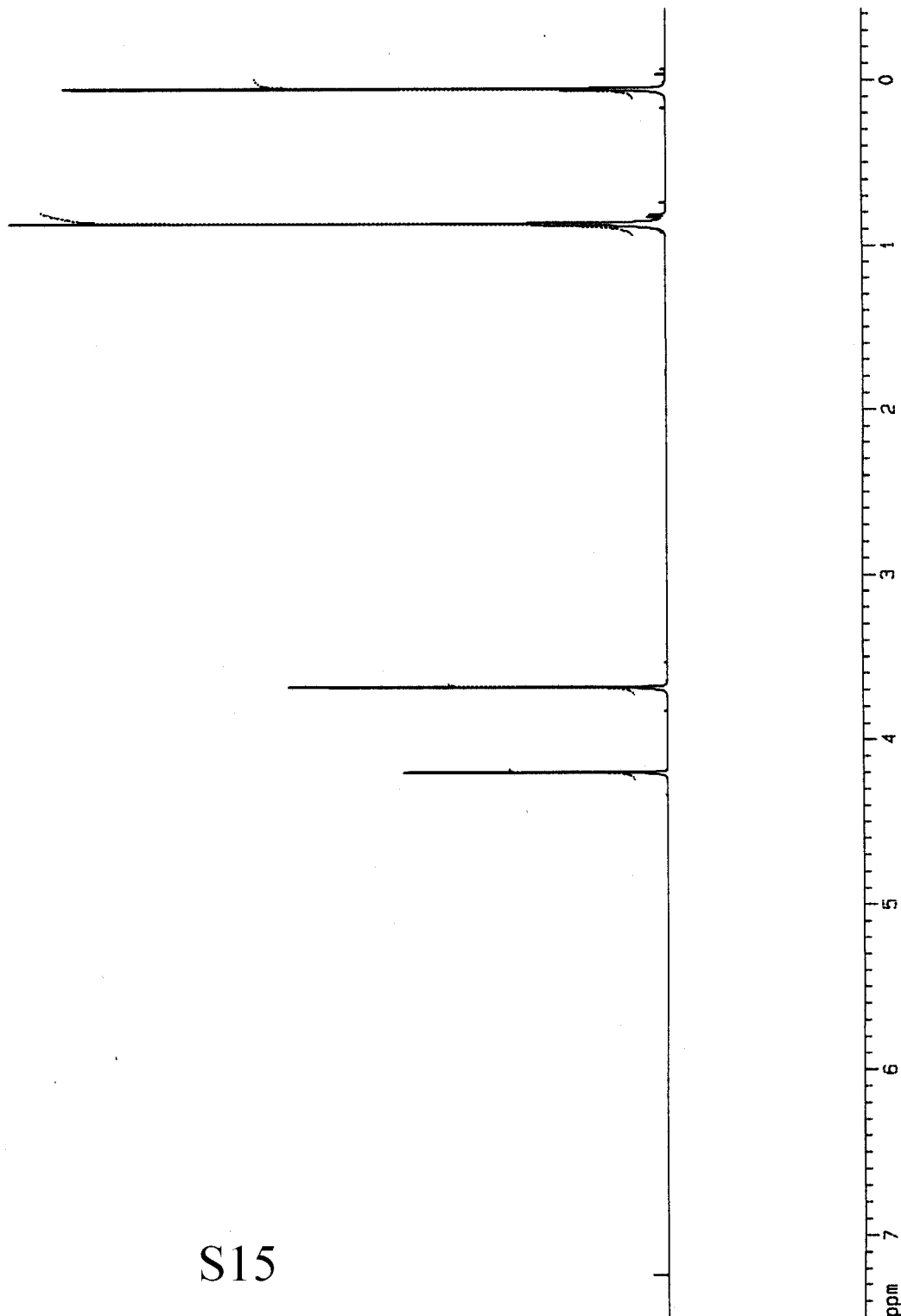
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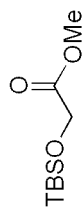
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 SOLVENT Acetone  
 NS 16  
 DS 0  
 SWH 7440.476 Hz  
 FIDRES 0.113533 Hz  
 AQ 4.4040694 sec  
 RG 16  
 DN 67.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 0.01000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 3.00 dB  
 SF01 500.1327766 MHz

F2 - Processing parameters  
 SI 65536  
 SF 500.1300038 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 10.00 cm  
 FIP 7.497 ppm  
 F1 3749.51 Hz  
 F2p -0.433 ppm  
 F2 -216.76 Hz  
 PPMCH 0.39652 ppm/cm  
 HZCM 198.31329 Hz/cm





27

<sup>13</sup>C with proton decoupling

```

Current Data Parameters
NAME      sa-187
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20040308
Time      12.36
INSTRUM   AV500WB
PROBHD    5 mm BBI 1H-BB
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         90
DS         0
SNH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912244 sec
RG         3251
DM         16.650 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
d11        0.03000000 sec
d12        0.0002000 sec

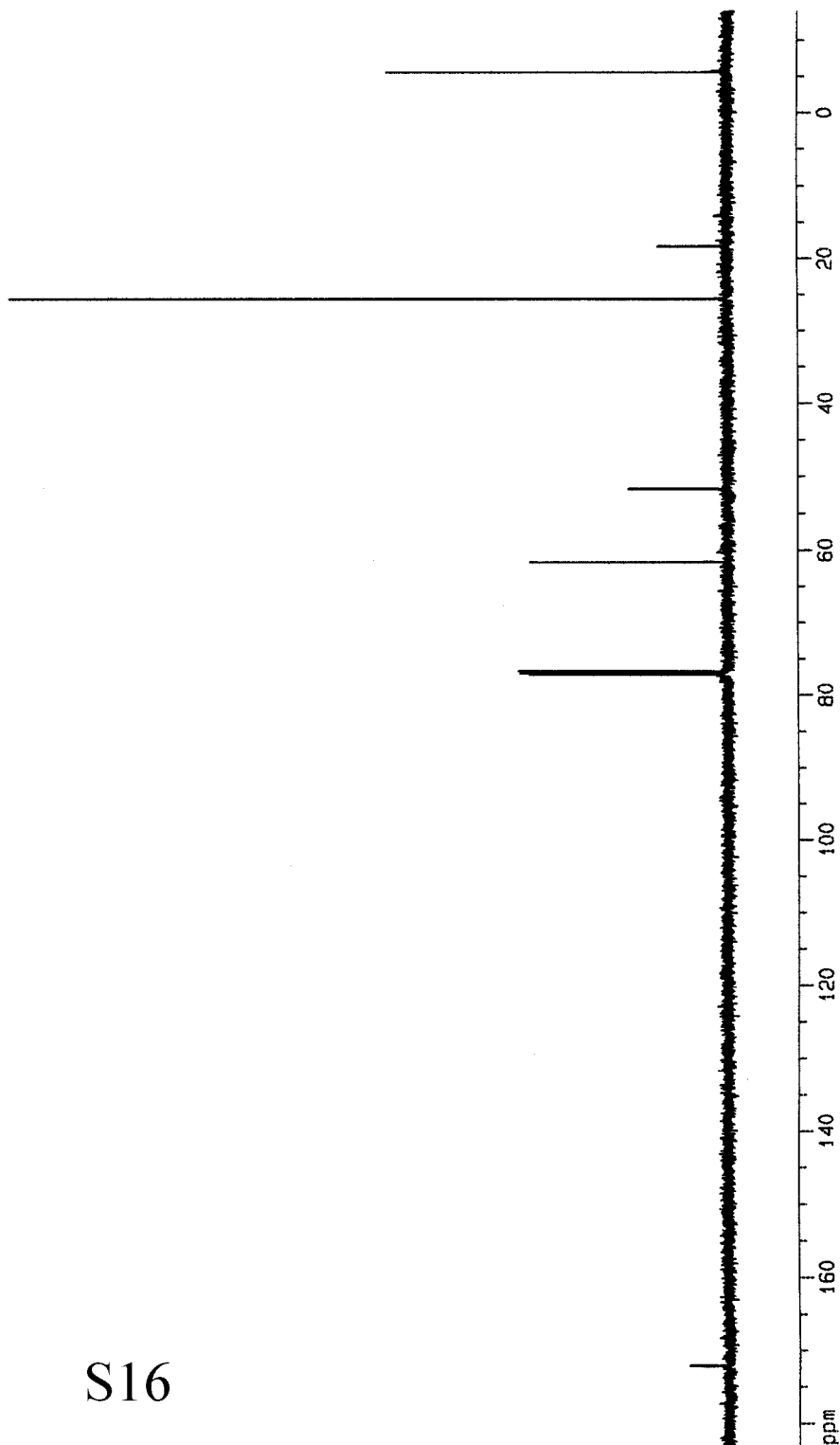
===== CHANNEL f1 =====
NUC1       13C
P1         14.50 usec
PL1        -3.00 dB
SF01       125.7703643 MHz

===== CHANNEL f2 =====
CQDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         3.00 dB
PL12        20.64 dB
PL13        23.56 dB
SF02        500.1320005 MHz

F2 - Processing parameters
SI          32768
SF          125.7577896 MHz
MDM         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.00

1D NMR plot parameters
CX          20.00 cm
CY          10.00 cm
FIP         183.019 ppm
F1          23016.03 Hz
F2P         -14.141 ppm
F2          -1778.30 Hz
PRMCM       9.85797 ppm/cm
HZCM        1239.71655 Hz/cm

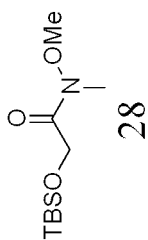
```



S16



1H NMR



S17

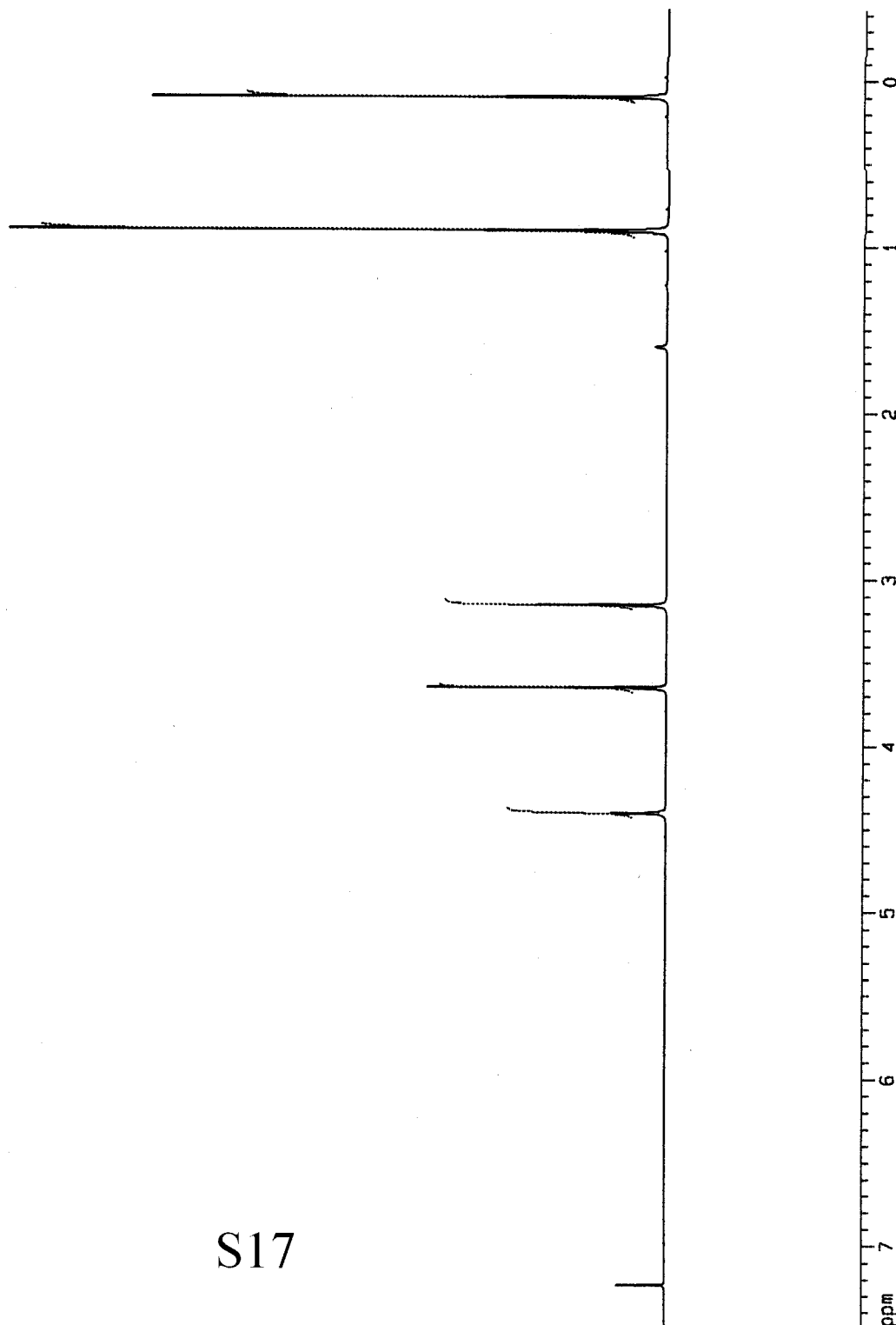
Current Data Parameters  
 NAME sa-188  
 EXPNO 1  
 PROCNO 1

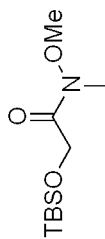
F2 - Acquisition Parameters  
 Date\_ 20040308  
 Time 18.43  
 INSTRUM AV500MB  
 PROBHD 5 mm BBI 1H-88  
 PULPROG zg30  
 TD 65536  
 SOLVENT Acetone  
 NS 16  
 DS 0  
 SMH 7440.476 Hz  
 FIDRES 0.113533 Hz  
 AQ 4.4040694 sec  
 RG 203.2  
 DW 67.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 0.01000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 3.00 dB  
 SF01 500.1327766 MHz

F2 - Processing parameters  
 SI 65536  
 SF 500.1300040 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 10.00 cm  
 F1P 7.482 ppm  
 F1 3741.83 Hz  
 F2P -0.434 ppm  
 F2 -216.99 Hz  
 PPMCM 0.39578 ppm/cm  
 HZCM 197.94052 Hz/cm

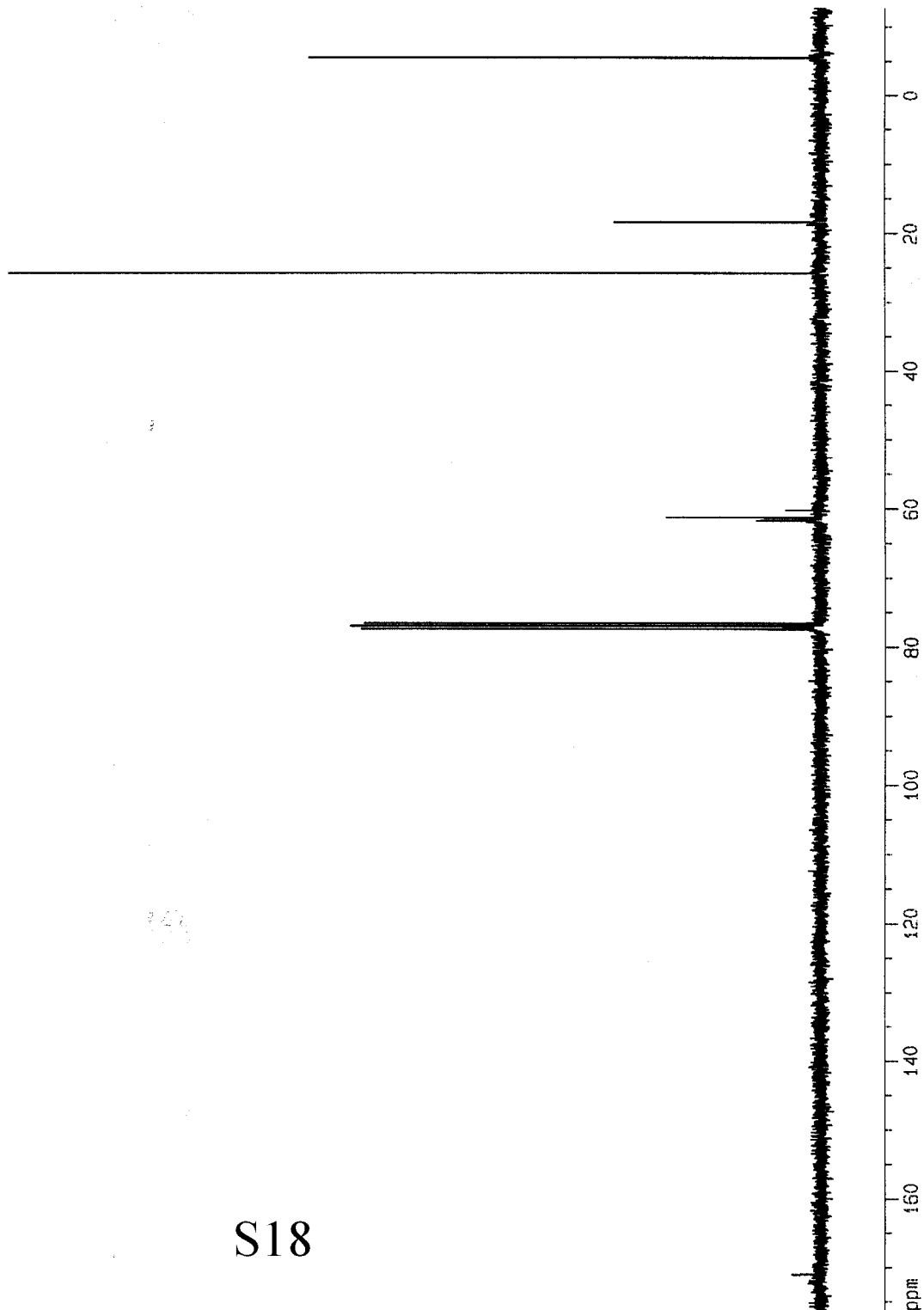




<sup>13</sup>C with proton decoupling

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S18



```

Current Data Parameters
NAME      sa-213-2nd
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20040402
Time      13.16
INSTRUM   av300
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         31
DS         0
SMH        17985.611 Hz
FIDRES     0.548877 Hz
AQ          0.9110004 sec
RG          3649.1
DM          27.800 usec
DE          6.00 usec
TE         300.0 K
D1         20.00000000 sec
d11         0.03000000 sec
d12         0.0002000 sec

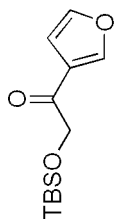
===== CHANNEL f1 =====
NUC1       13C
P1          5.00 usec
PL1         -6.00 dB
SF01       75.4752653 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       70.00 usec
PL2         -3.00 dB
PL12        13.48 dB
PL13        15.63 dB
SF02       300.1314860 MHz

F2 - Processing parameters
SI          65536
SF          75.4677516 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

1D NMR plot parameters
CX          20.00 cm
CY          12.50 cm
F1P         176.274 ppm
F1          13303.03 Hz
F2P         -12.723 ppm
F2          -960.18 Hz
PPMCM       9.44988 ppm/cm
HZCM        713.16089 Hz/cm

```



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# Current Data Parameters

NAME sa-203  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20040322  
Time 10.25  
INSTRUM av300  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 30720  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 5081.301 Hz  
FIDRES 0.165407 Hz  
AQ 3.0228980 sec  
RG 912.3  
DW 98.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

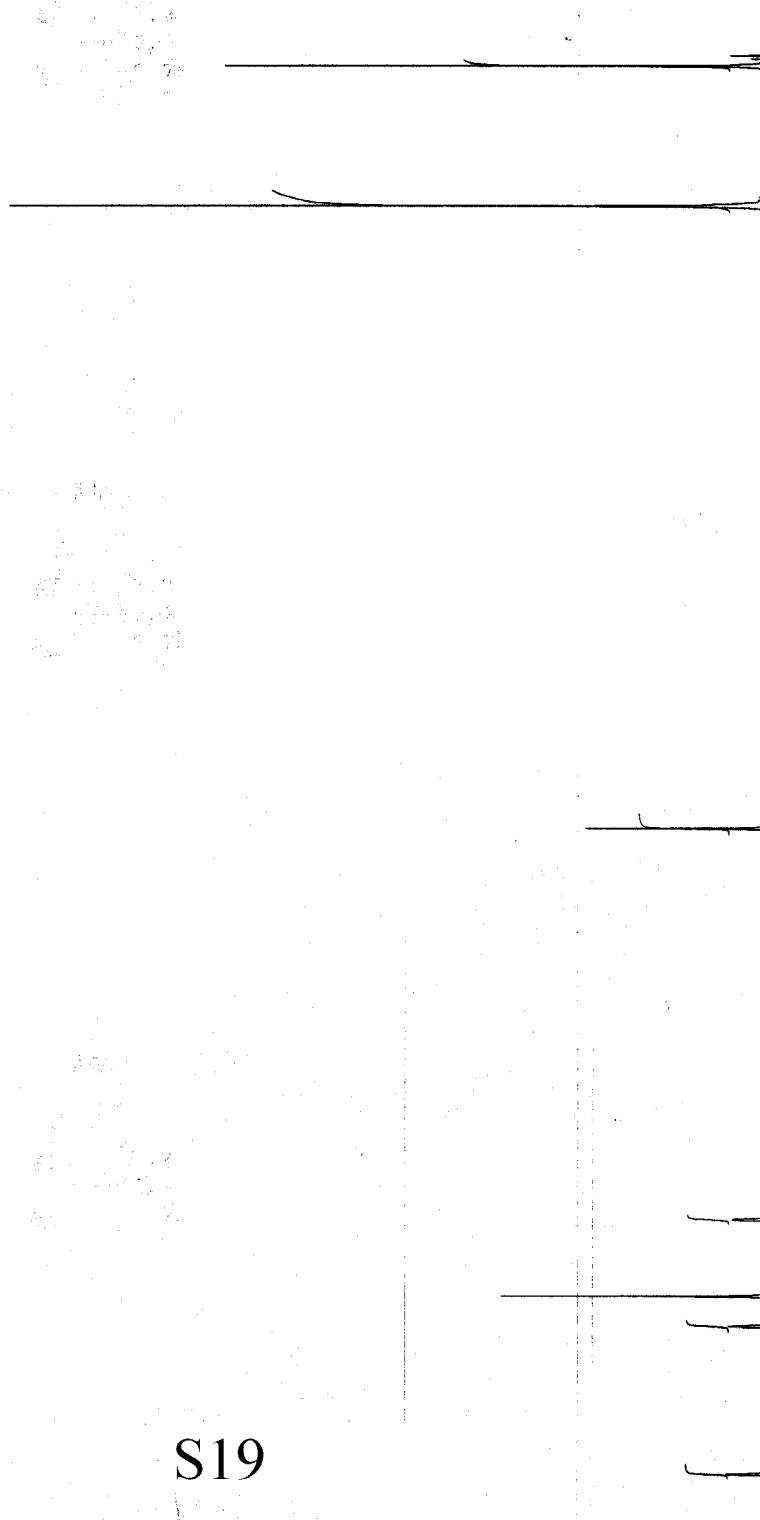
NUC1 1H  
P1 10.50 usec  
PL1 -3.00 dB  
SF01 300.1319477 MHz

## F2 - Processing parameters

SI 65536  
SF 300.1300000 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00

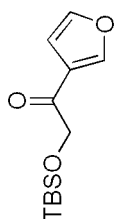
## 1D NMR plot parameters

CX 20.00 cm  
CY 10.00 cm  
F1P 8.547 ppm  
F1 2565.30 Hz  
F2P -0.251 ppm  
F2 -75.20 Hz  
PPMCM 0.43989 ppm/cm  
HZCM 132.02505 Hz/cm



S19

<sup>13</sup>C with proton decoupling



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S20

```

Current Data Parameters
NAME          sa-20B
EXPNO         2
PROCNO        1

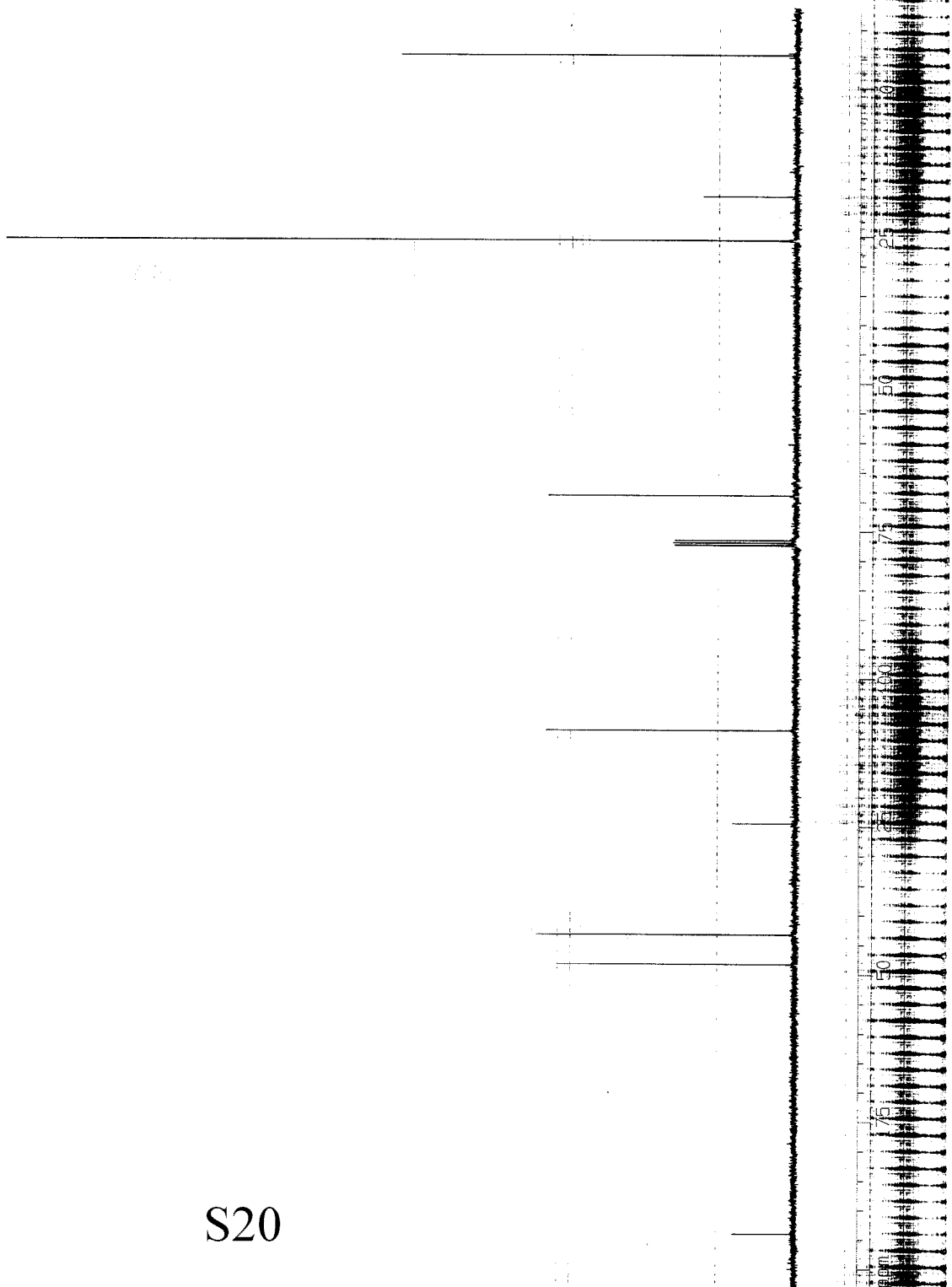
F2 - Acquisition Parameters
Date_         20040324
Time          9.50
INSTRUM       av300
PROBHD        5 mm QNP 1H/1
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            78
DS            0
SWH           17985.611 Hz
FIDRES        0.548877 Hz
AQ            0.9110004 sec
RG            2298.8
DM            27.800 usec
DE            6.00 usec
TE            300.0 K
D1            1.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

===== CHANNEL f1 =====
NUC1          13C
P1            5.00 usec
PL1           -6.00 dB
SF01          75.4752653 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL2           -3.00 dB
PL12          13.48 dB
PL13          15.63 dB
SF02          300.1314860 MHz

F2 - Processing parameters
SI            65536
SF            75.4677506 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
GC            1.40

1D NMR plot parameters
CX            20.00 cm
CY            12.50 cm
F1P           203.126 ppm
F1            15329.44 Hz
F2P           -13.688 ppm
F2            -1033.02 Hz
  
```



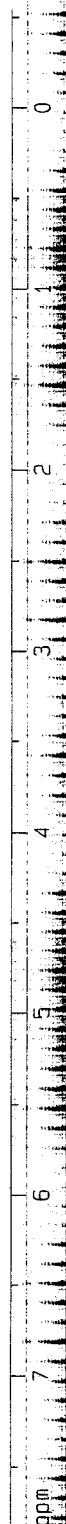


PROCNO 1

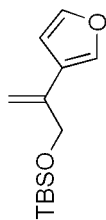
## D1 1.000000000

SF01 300.1319477

PC 1.00

125.73816  
HZCM

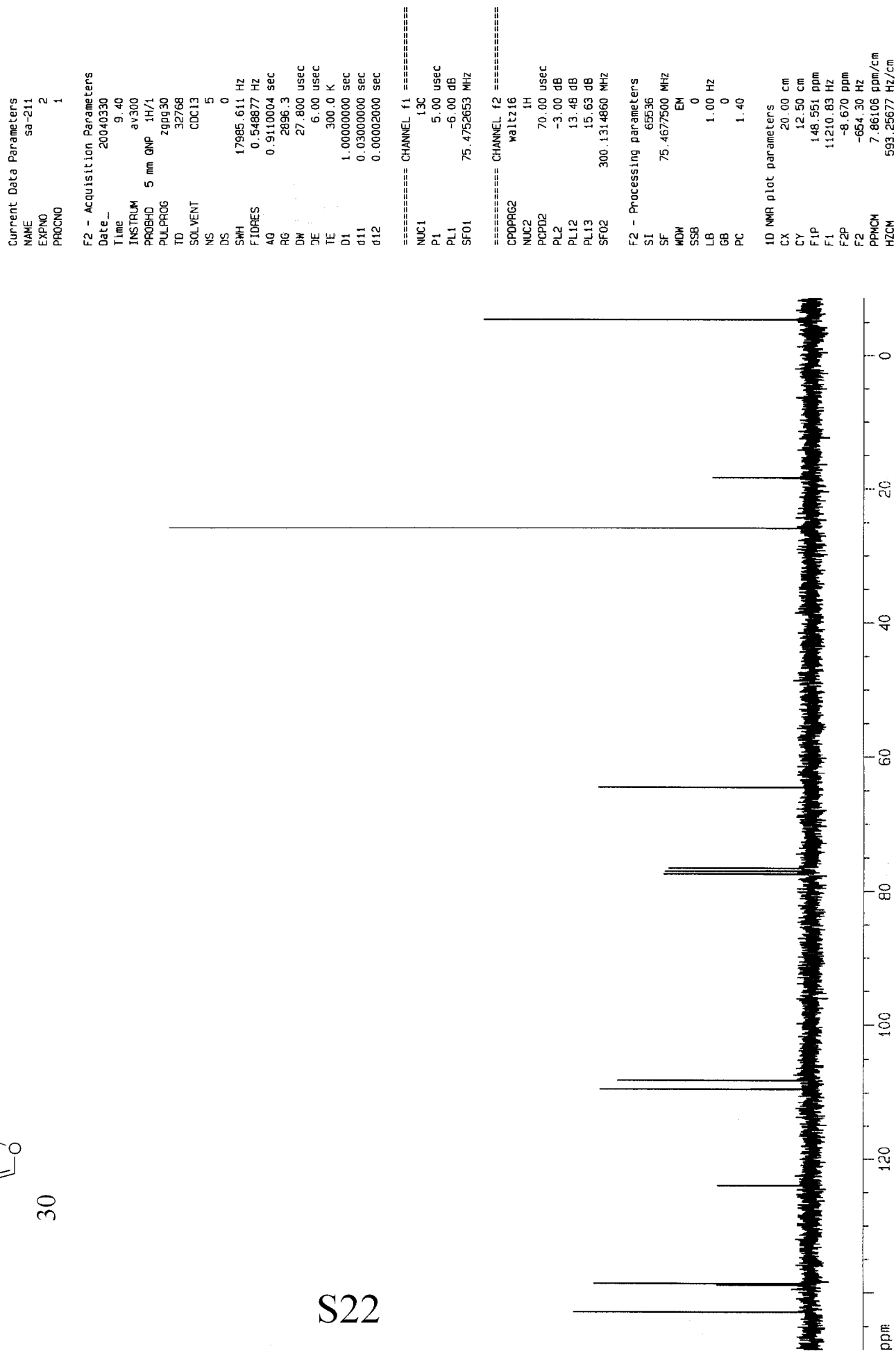
S21

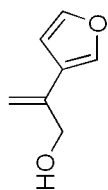


<sup>13</sup>C with proton decoupling

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S22





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S23

```

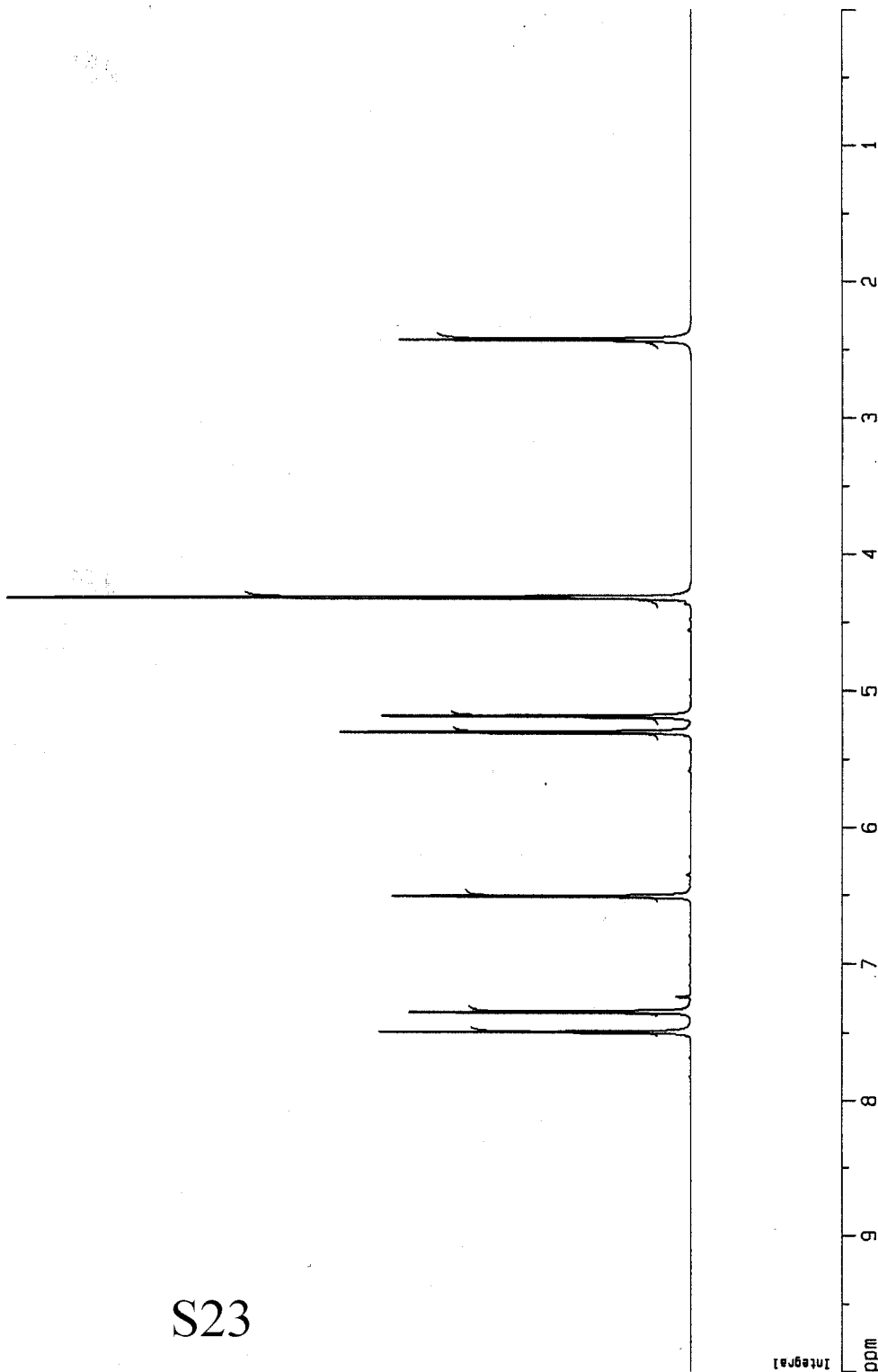
Current Data Parameters
NAME      sa-222
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20040406
Time      10.50
INSTRUM   av300
PROBHD    5 mm QNP 1H/1
PULPROG   zg30
TD         30720
SOLVENT    CDCl3
NS         16
DS         0
SWH        5081.301 Hz
FIDRES     0.165407 Hz
AQ         3.0228980 sec
RG         64
DM         98.400 usec
DE         6.00 usec
TE         300.0 K
D1         1.0000000 sec

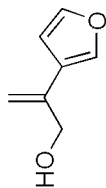
===== CHANNEL f1 =====
NUC1       1H
P1         10.50 usec
PL1        -3.00 dB
SF01       300.1319477 MHz

F2 - Processing parameters
SI         65536
SF         300.1299988 MHz
WDW        EM
SSB        0
LB         0.10 Hz
GB         0
PC         1.00

1D NMR plot parameters
CX         20.00 cm
CY         10.00 cm
F1P        10.000 ppm
F1         3001.30 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPHMC      0.50000 ppm/cm
HZCM       150.06500 Hz/cm
  
```

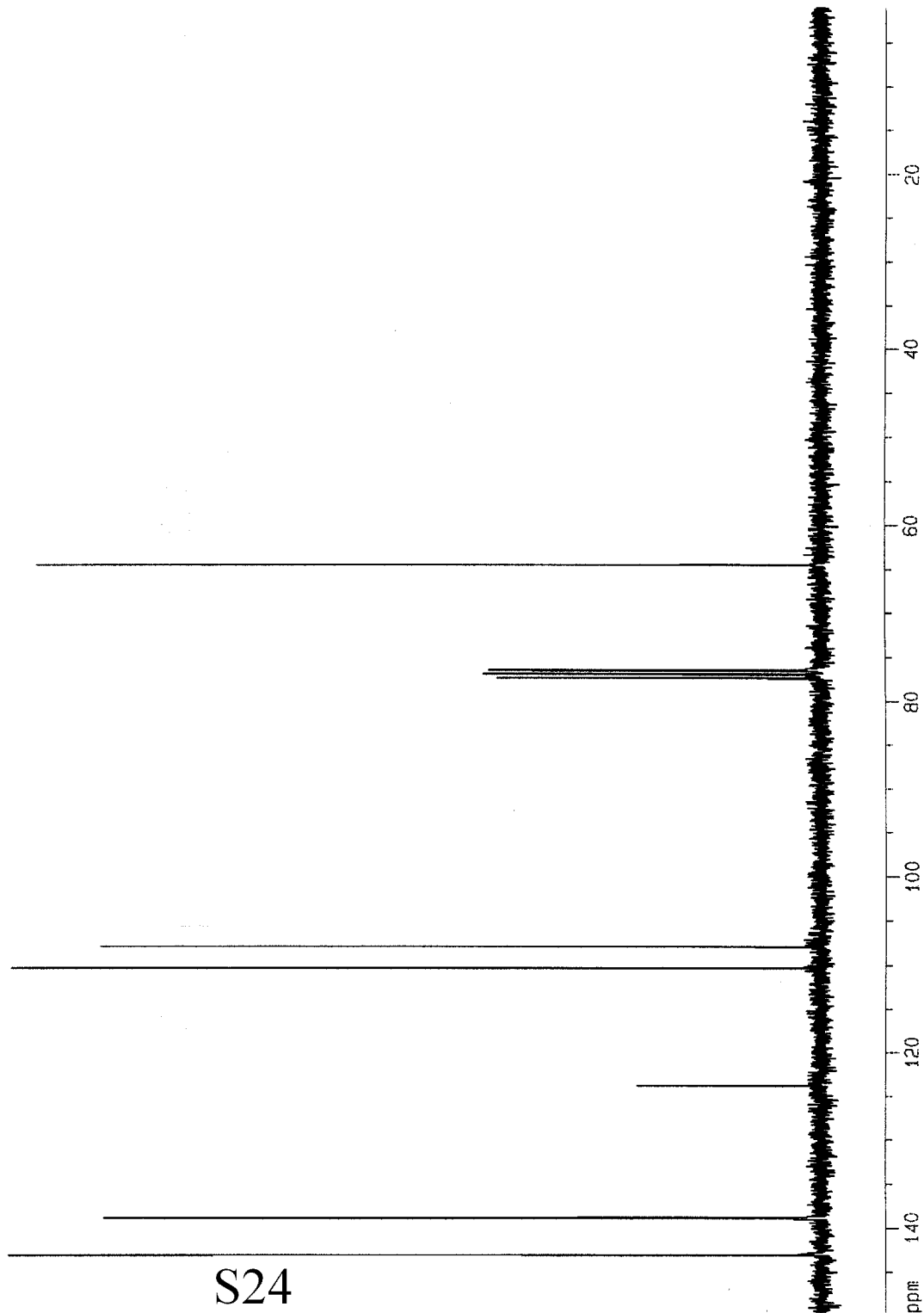


Integral



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<sup>13</sup>C with proton decoupling



```

Current Data Parameters
NAME      sa-222
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20040406
Time      10:52
INSTRUM   av300
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         62
DS         0
SMH        17985.611 Hz
FIDRES     0.548877 Hz
AQ         0.9110004 sec
RG         3649.1
DM         27.800 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
d11        0.03000000 sec
d12        0.00002000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         5.00 usec
PL1        -6.00 dB
SF01       75.4752653 MHz

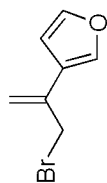
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      70.00 usec
PL2        -3.00 dB
PL12       13.48 dB
PL13       15.63 dB
SF02       300.1314860 MHz

F2 - Processing parameters
SI         65536
SF         75.4677544 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         20.00 cm
CY         12.50 cm
F1P        149.679 ppm
F1         11295.92 Hz
F2P        0.994 ppm
F2         75.05 Hz
PPMCM      7.43422 ppm/cm
HZCM       561.04376 Hz/cm
  
```

S24





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S25

```

Current Data Parameters
NAME      sa-214
EXPNO     1
PROCNO    1

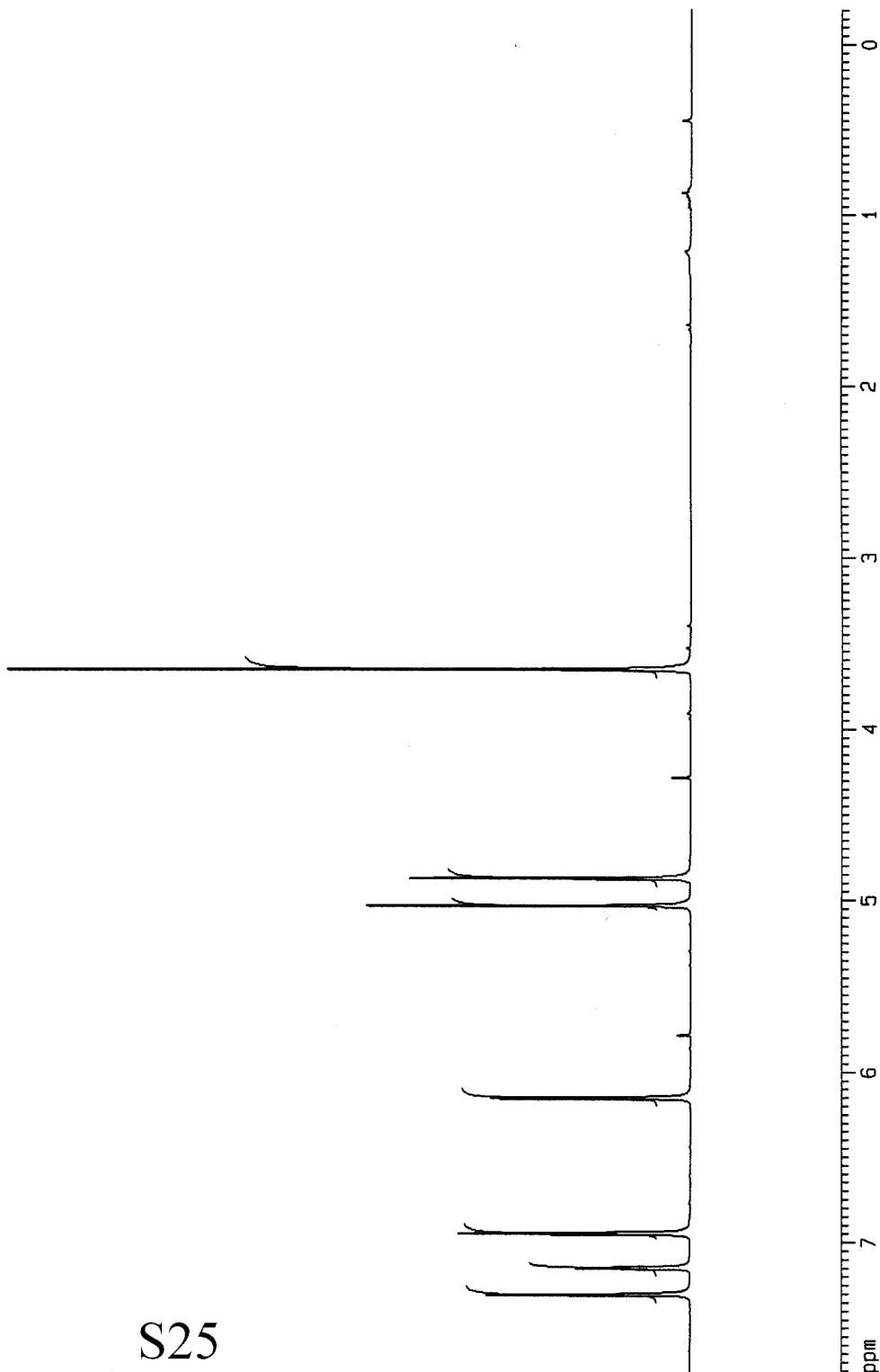
F2 - Acquisition Parameters
Date_     20040401
Time      11.20
INSTRUM   av300
PROBHD    5 mm QNP 1H/1
PULPROG   zg30
TD         30720
SOLVENT   CDCl3
NS         16
DS         0
SNH        5081.301 Hz
FIDRES     0.165407 Hz
AQ         3.0226980 sec
RG         128
DM         98.400 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec

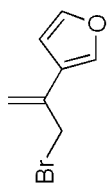
===== CHANNEL f1 =====
NUC1       1H
P1         10.50 usec
PL1        -3.00 dB
SF01       300.1319477 MHz

F2 - Processing parameters
SI         65536
SF         300.1299997 MHz
WDW        EM
SSB        0
LB         0.10 Hz
GB         0
PC         1.00

1D NMR plot parameters
CX         20.00 cm
CY         10.00 cm
F1P        7.762 ppm
F1         2329.71 Hz
F2P        -0.206 ppm
F2         -61.78 Hz
PPMCM      0.39841 ppm/cm
HZCM       119.57489 Hz/cm

```





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<sup>13</sup>C with proton decoupling

```

Current Data Parameters
NAME      sa-214
EXPNO     2
PROCNO    1

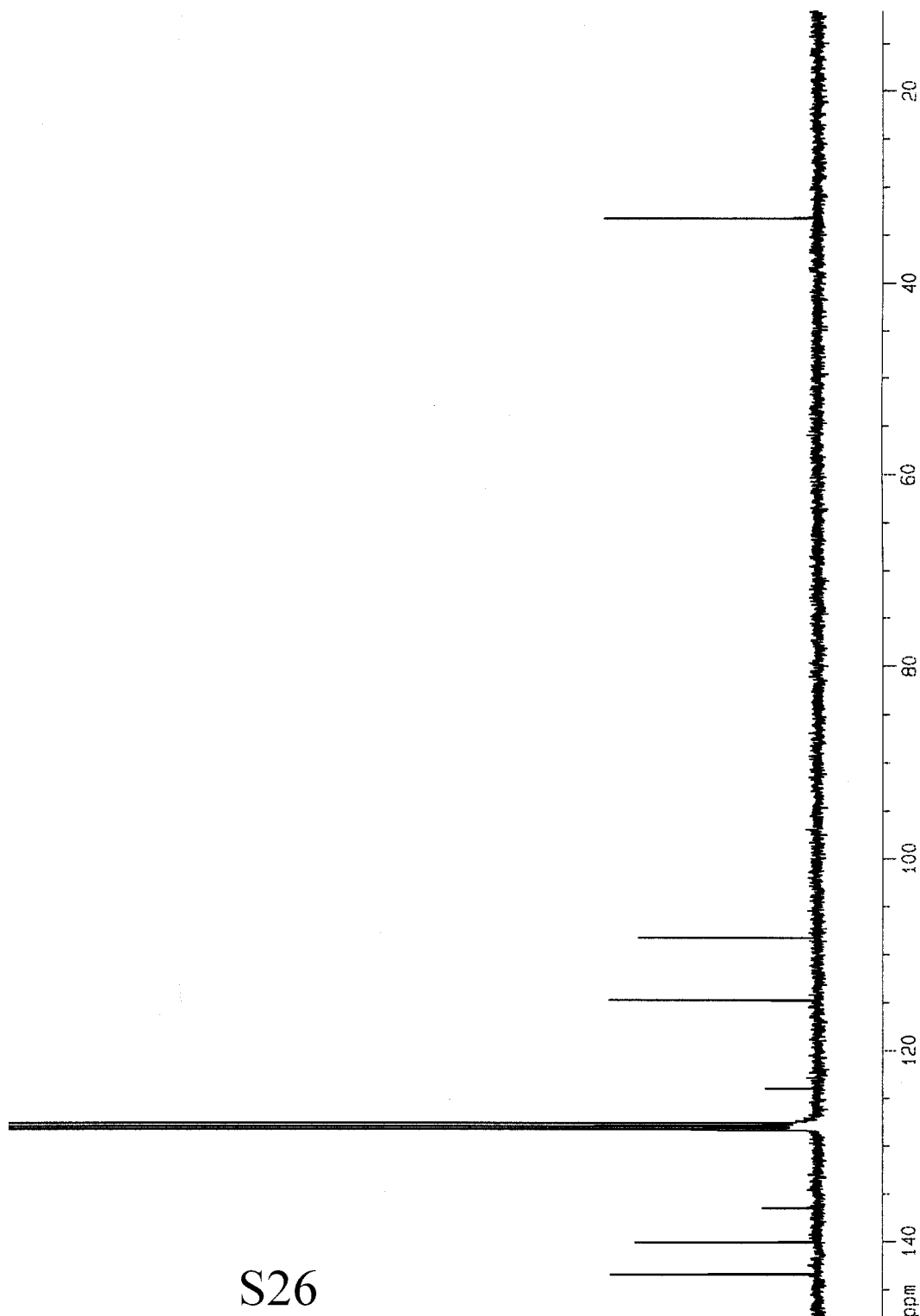
F2 - Acquisition Parameters
Date_     20040401
Time      11.21
INSTRUM   av300
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         36
DS         0
SWH        17985.611 Hz
FIDRES     0.548877 Hz
AQ          0.9110004 sec
RG          3649.1
RW          27.800 usec
QE           6.00 usec
TE          300.0 K
D1          1.00000000 sec
d11         0.03000000 sec
d12         0.00002000 sec

===== CHANNEL f1 =====
NUC1       13C
P1          5.00 usec
PL1         -6.00 dB
SFO1        75.4752653 MHz

===== CHANNEL f2 =====
CPOPRG2    waltz16
NUC2        1H
PCPD2       70.00 usec
PL2         -3.00 dB
PL12        13.48 dB
PL13        15.63 dB
SFO2        300.1314860 MHz

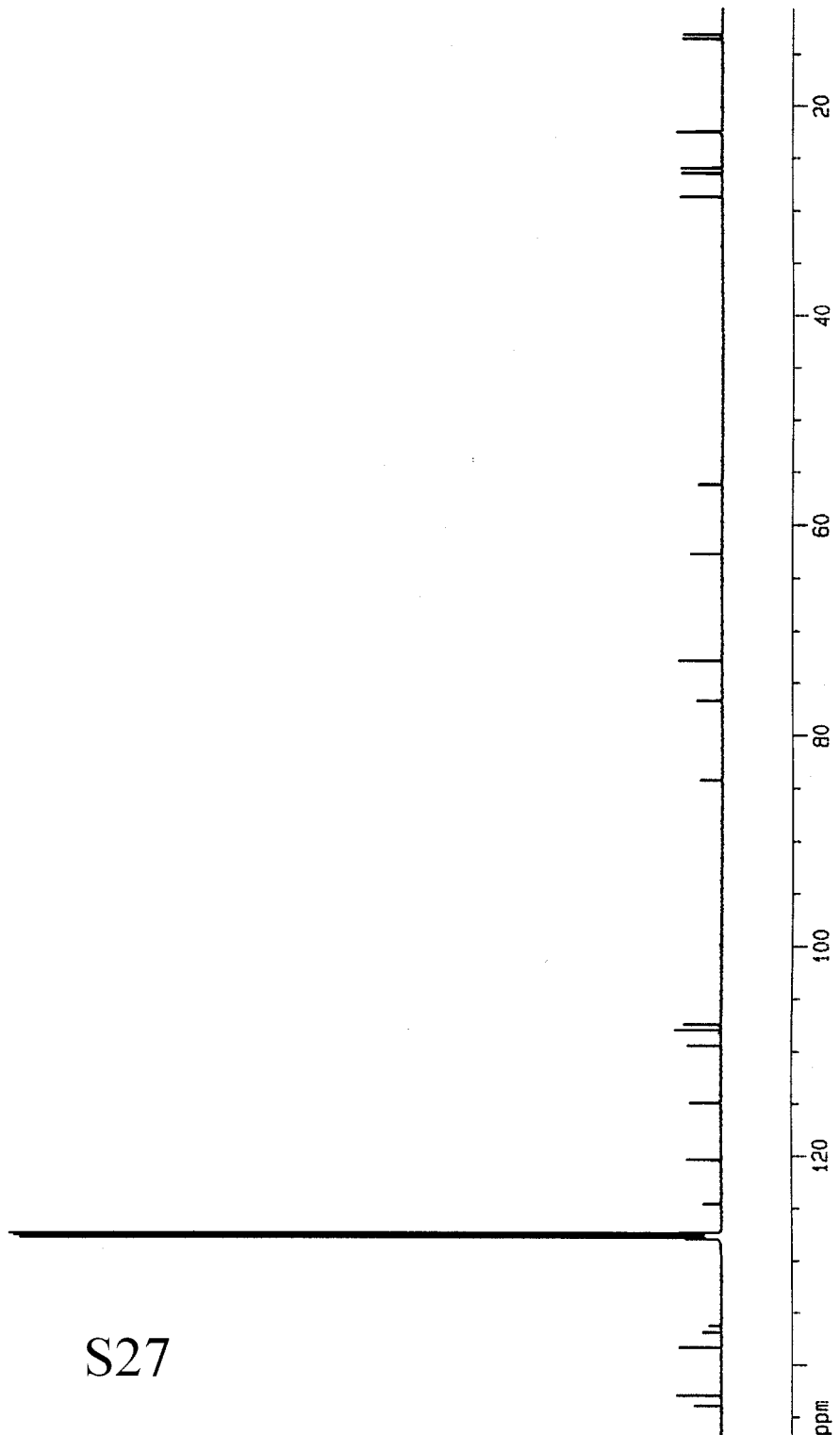
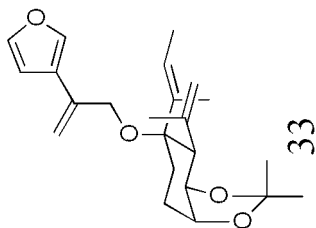
F2 - Processing parameters
SI          65536
SF          75.4677257 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

1D NMR plot parameters
CX           20.00 cm
CY           12.50 cm
F1P          147.924 ppm
F1           11163.52 Hz
F2P          11.571 ppm
F2           873.25 Hz
PPMCM        6.81767 ppm/cm
HZCM         514.51379 Hz/cm
  
```



S26

<sup>13</sup>C with proton decoupling



Current Data Parameters  
 NAME sa-274  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20040518  
 Time 16.00  
 INSTRUM AV500WB  
 PROBHD 5 mm TBO BB/1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 310  
 DS 0  
 SMH 30030.029 Hz  
 FIDRES 0.458222 Hz  
 AQ 1.0912244 sec  
 RG 203.2  
 DM 16.650 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 d11 0.03000000 sec  
 d12 0.0002000 sec

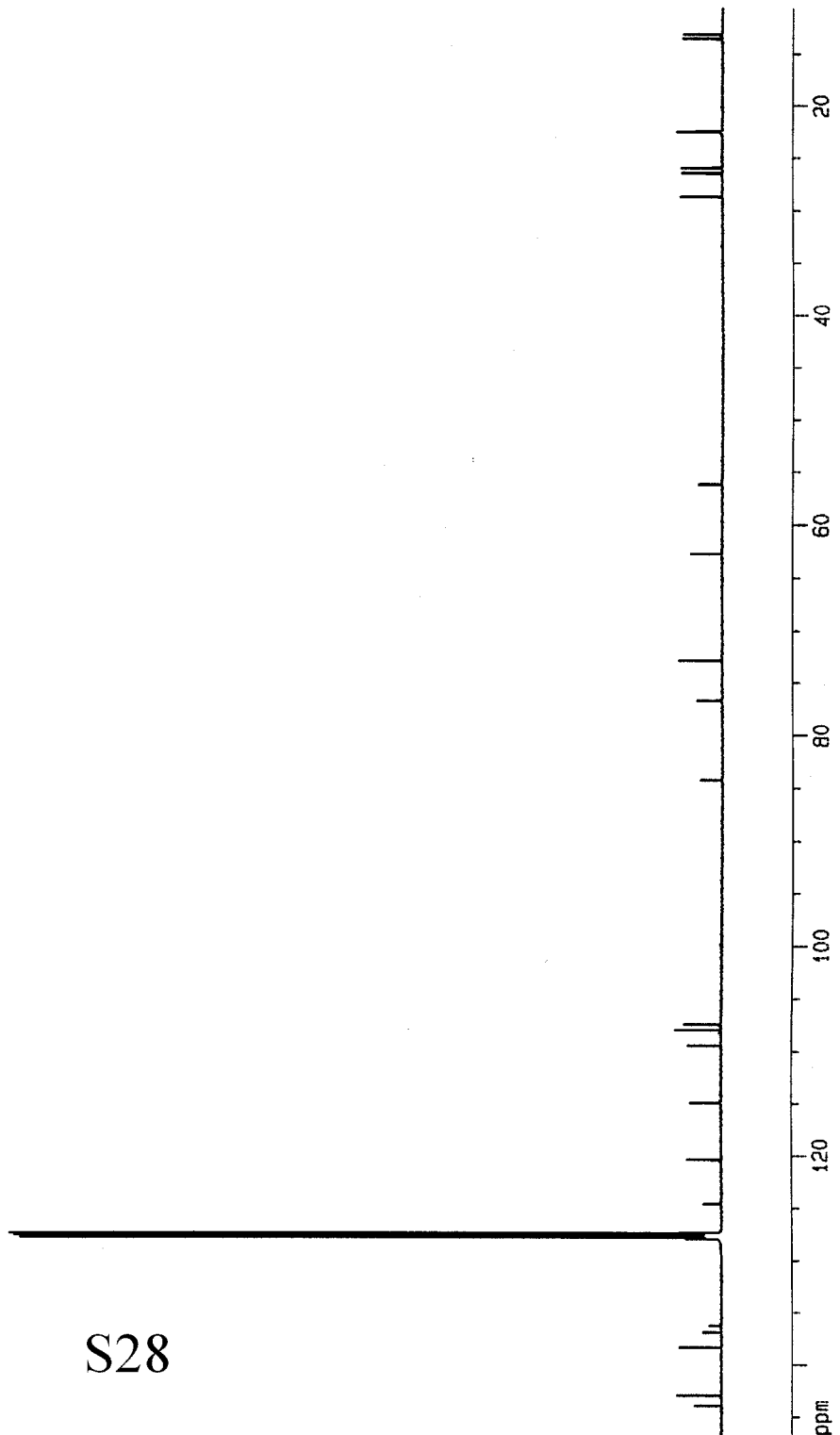
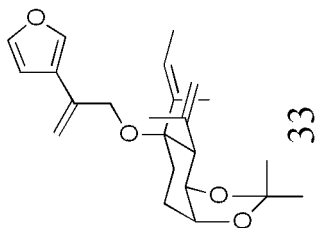
===== CHANNEL f1 =====  
 NUC1 <sup>13</sup>C  
 P1 8.00 usec  
 PL1 -0.20 dB  
 SF01 125.7703643 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 <sup>1</sup>H  
 PCPD2 80.00 usec  
 PL2 -0.20 dB  
 PL12 14.94 dB  
 PL13 14.90 dB  
 SF02 500.1320005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 125.7578051 MHz  
 NDM EN  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 10.00 cm  
 F1P 146.735 ppm  
 F1 18453.04 Hz  
 F2P 10.592 ppm  
 F2 1331.98 Hz  
 PPMCM 6.80715 ppm/cm  
 HZCM 856.05280 Hz/cm

<sup>13</sup>C with proton decoupling



Current Data Parameters  
 NAME sa-274  
 EXPNO 2  
 PROCNO 1

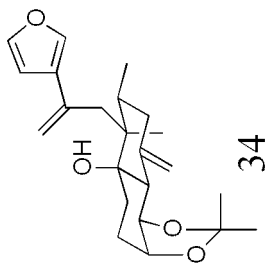
F2 - Acquisition Parameters  
 Date\_ 20040518  
 Time 16.00  
 INSTRUM AV500WB  
 PROBHD 5 mm TBO BB/1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 310  
 DS 0  
 SMH 30030.029 Hz  
 FIDRES 0.458222 Hz  
 AQ 1.0912244 sec  
 RG 203.2  
 DM 16.650 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 d11 0.03000000 sec  
 d12 0.0002000 sec

===== CHANNEL f1 =====  
 NUC1 <sup>13</sup>C  
 P1 8.00 usec  
 PL1 -0.20 dB  
 SF01 125.7703643 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 <sup>1</sup>H  
 PCPD2 80.00 usec  
 PL2 -0.20 dB  
 PL12 14.94 dB  
 PL13 14.90 dB  
 SF02 500.1320005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 125.7578051 MHz  
 NDM EN  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 10.00 cm  
 F1P 146.735 ppm  
 F1 18453.04 Hz  
 F2P 10.592 ppm  
 F2 1331.98 Hz  
 PPMCM 6.80715 ppm/cm  
 HZCM 856.05280 Hz/cm



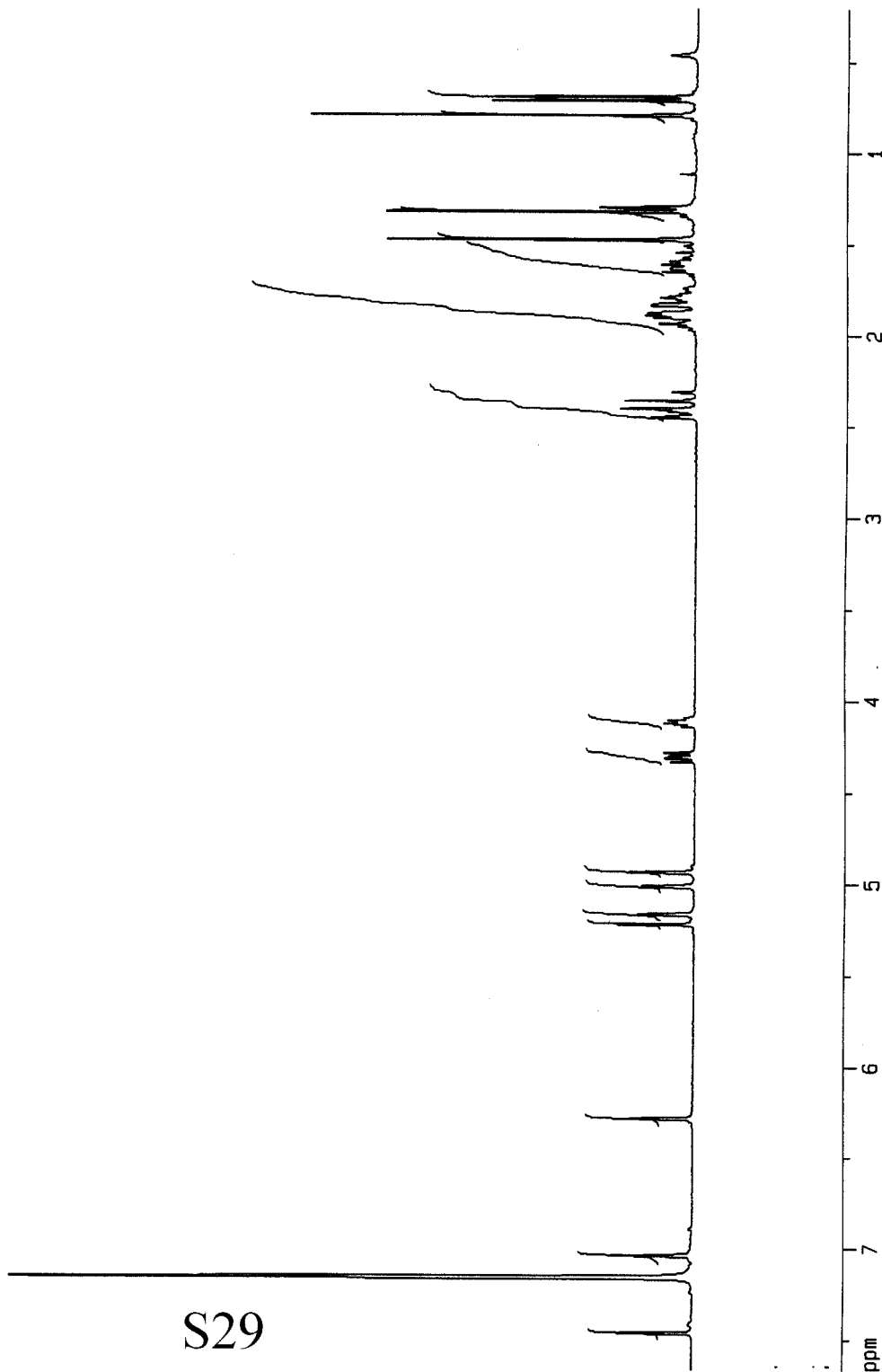
Current Data Parameters  
NAME sa-239prod  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20040422  
Time 11.20  
INSTRUM av300  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 30720  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 5081.301 Hz  
FIDRES 0.165407 Hz  
AQ 3.0228980 sec  
RG 287.4  
DN 98.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

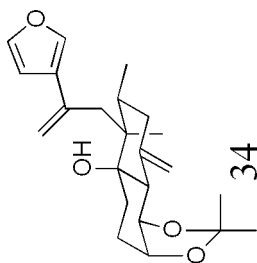
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.50 usec  
PL1 -3.00 dB  
SF01 300.1319477 MHz

F2 - Processing parameters  
SI 65536  
SF 300.1299967 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
CY 10.00 cm  
F1P 7.671 ppm  
F1 2302.30 Hz  
F2P 0.207 ppm  
F2 62.04 Hz  
PPMCM 0.37321 ppm/cm  
HZCM 112.01264 Hz/cm



<sup>13</sup>C with proton decoupling



S30

```

Current Data Parameters
NAME      sa-230
EXPNO     3
PROCNO    1

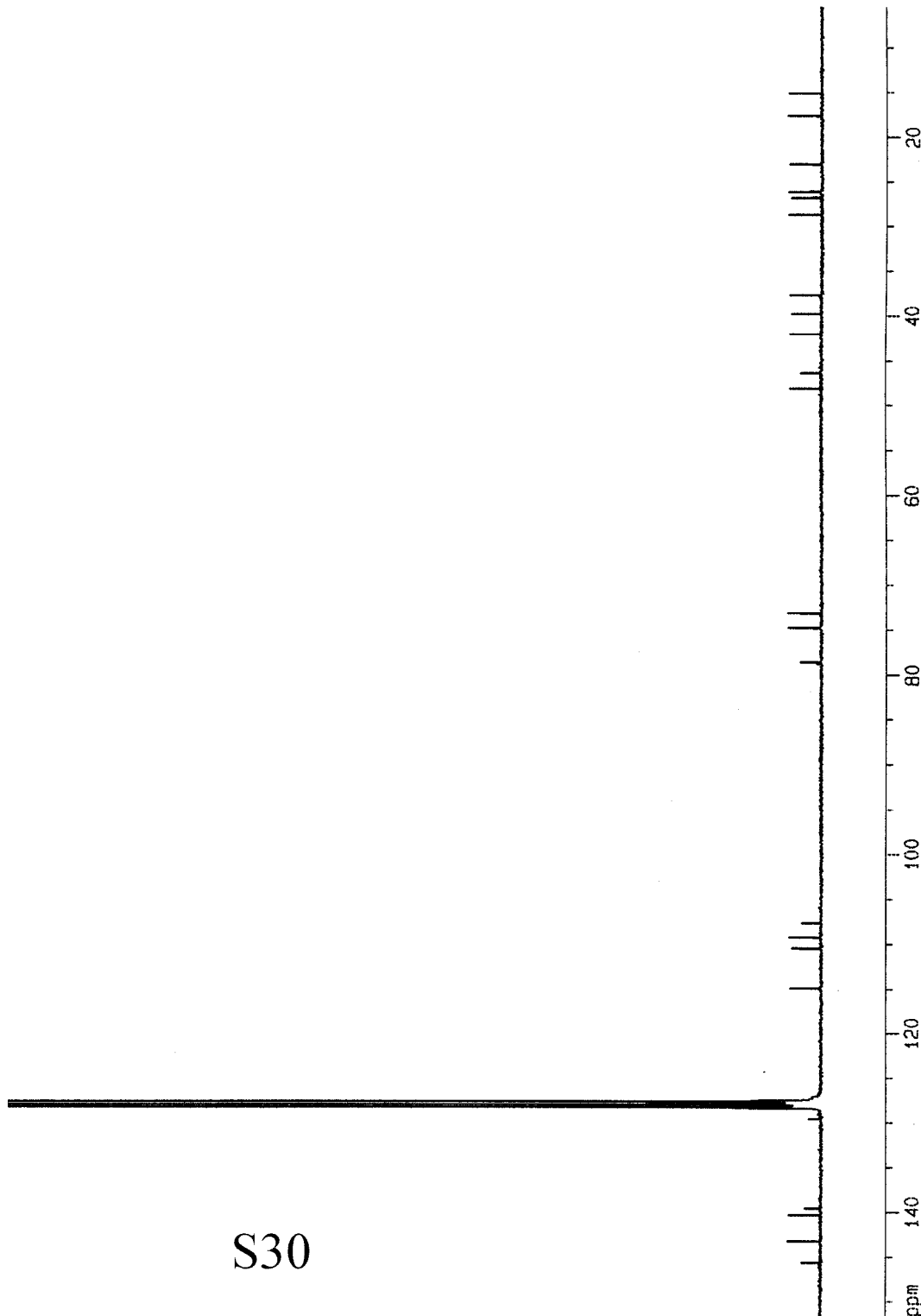
F2 - Acquisition Parameters
Date_     20040414
Time      18:53
INSTRUM   av300
PROBHD    5 mm GNP 1H/1
PULPROG   zgpg30
TD         32768
SOLVENT   CDC13
NS         1200
DS         0
SMH        17985.611 Hz
FIDRES     0.548877 Hz
AQ         0.9110004 sec
RG         3649.1
DM         27.800 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
d11        0.03000000 sec
d12        0.0002000 sec

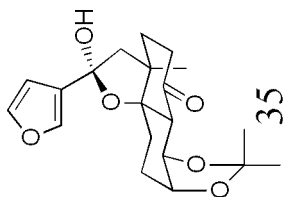
===== CHANNEL f1 =====
NUC1       13C
P1         5.00 usec
PL1        -6.00 dB
SF01       75.4752653 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      70.00 usec
PL2        -3.00 dB
PL12       13.48 dB
PL13       15.63 dB
SF02       300.1314860 MHz

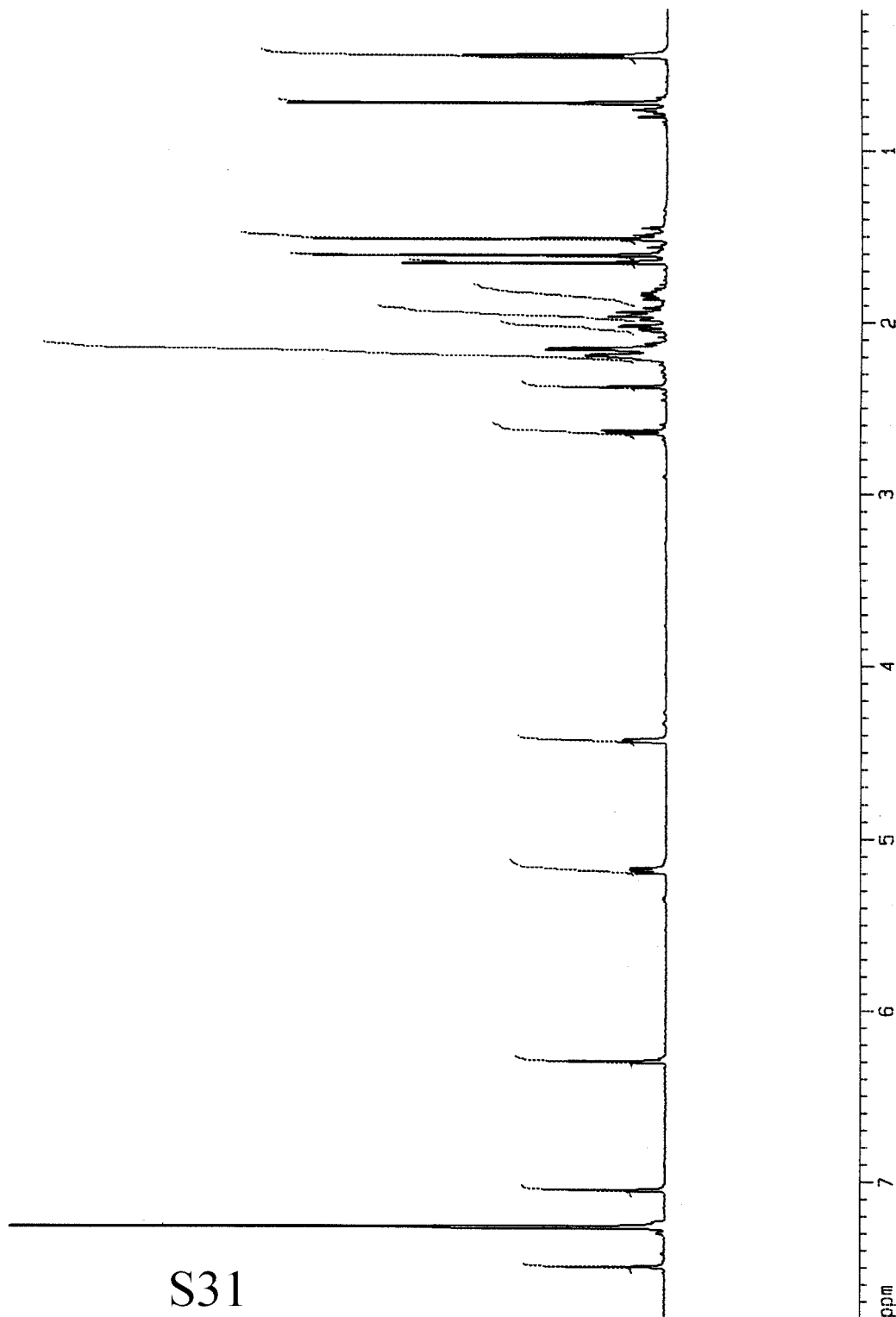
F2 - Processing parameters
SI         65536
SF         75.4677252 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         20.00 cm
CY         12.50 cm
FIP        151.726 ppm
F1         11450.41 Hz
F2         5.413 ppm
PPHMM      408.50 Hz
PNNOM      7.31565 ppm/cm
HZCM       552.09570 Hz/cm
    
```





<sup>1</sup>H NMR



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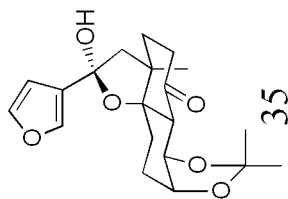
Current Data Parameters  
 NAME sa-280  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20040610  
 Time 15.04  
 INSTRUM AV500WB  
 PROBHD 5 mm TBO BB/1H  
 PULPROG zg30  
 TD 65536  
 SOLVENT Acetone  
 NS 16  
 DS 0  
 SMH 7440.476 Hz  
 FIDRES 0.113533 Hz  
 AQ 4.4040694 sec  
 RG 114  
 DM 67.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 0.01000000 sec

===== CHANNEL f1 =====  
 NUC1 <sup>1</sup>H  
 P1 14.00 usec  
 PL1 0.00 dB  
 SF01 500.1327766 MHz

F2 - Processing parameters  
 SI 65536  
 SF 500.1300051 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 10.00 cm  
 F1P 7.793 ppm  
 F1 3897.27 Hz  
 F2P 0.175 ppm  
 F2 87.57 Hz  
 PPMCM 0.38087 ppm/cm  
 HZCM 190.48512 Hz/cm



<sup>13</sup>C with proton decoupling

Current Data Parameters  
 NAME sa-280  
 EXPNO 2  
 PROCNO 1

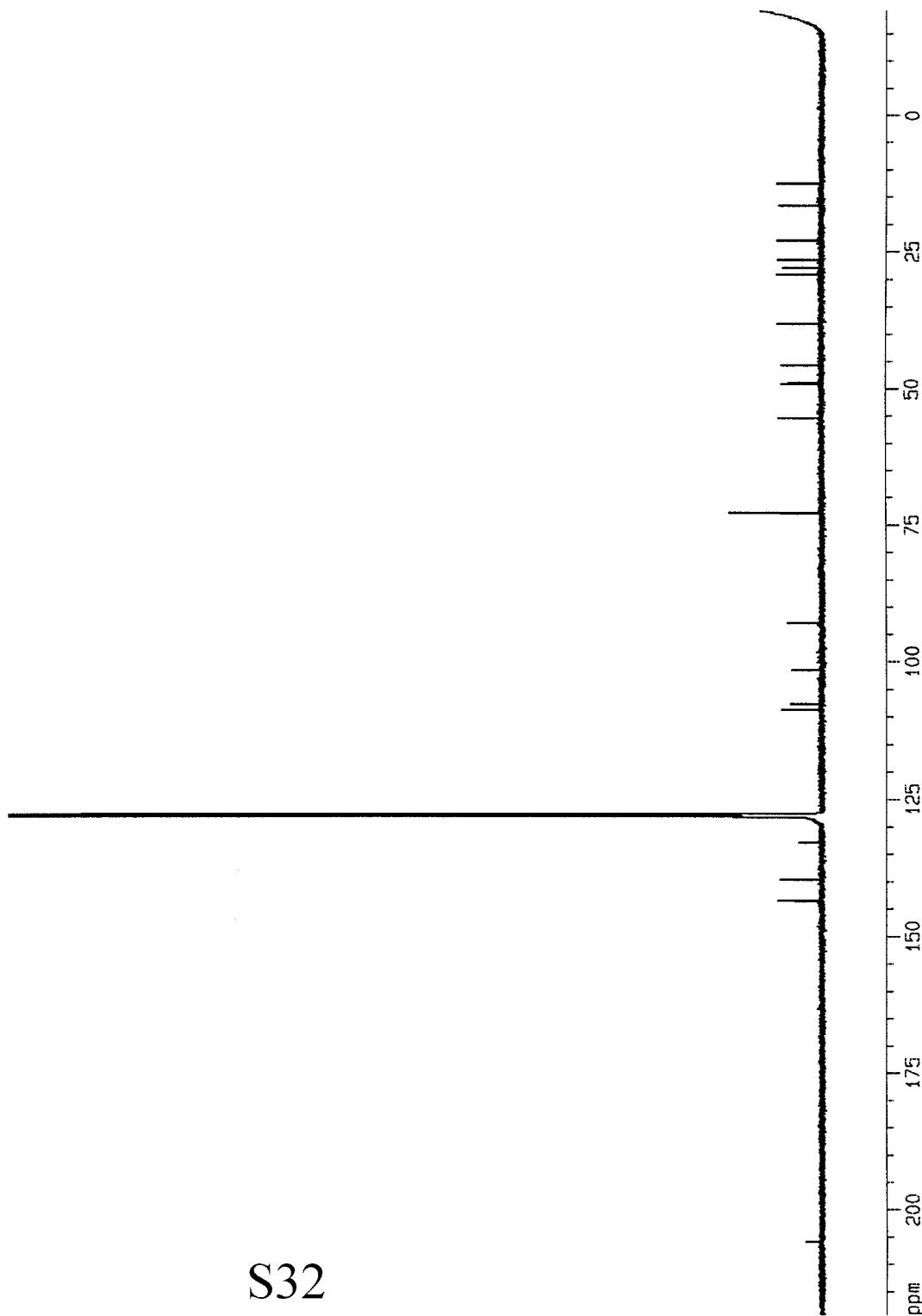
F2 - Acquisition Parameters  
 Date\_ 20040610  
 Time 15.07  
 INSTRUM AV500NB  
 PROBHD 5 mm TBO BB/1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 469  
 DS 0  
 SMH 30030.029 Hz  
 FIDRES 0.458222 Hz  
 AQ 1.0912244 sec  
 RG 161.3  
 DM 16.650 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 d11 0.03000000 sec  
 d12 0.0002000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 8.00 usec  
 PL1 -0.20 dB  
 SF01 125.7703643 MHz

===== CHANNEL f2 =====  
 CPOPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -0.20 dB  
 PL12 14.94 dB  
 PL13 14.90 dB  
 SF02 500.1320005 MHz

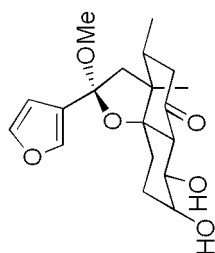
F2 - Processing parameters  
 SI 32768  
 SF 125.7577663 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 50.00 cm  
 FIP 219.264 ppm  
 F1 27574.16 Hz  
 F2 -19.528 ppm  
 F2 -2455.86 Hz  
 PPNOM 11.93963 ppm/cm  
 HZCM 1501.50098 Hz/cm

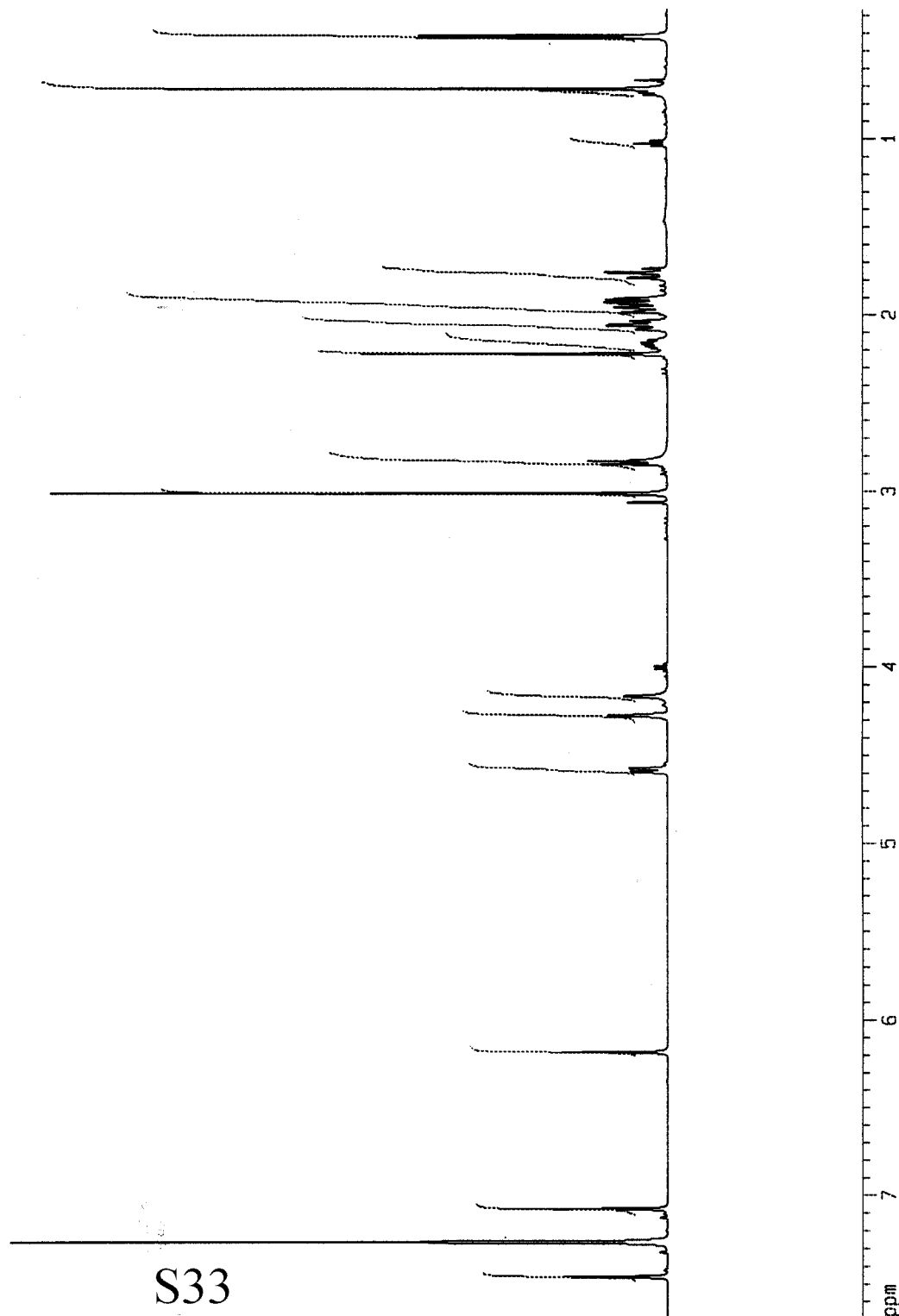




1H NMR



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S33

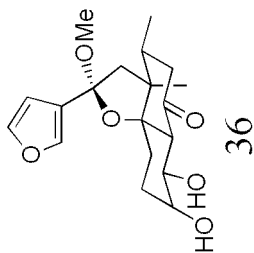
Current Data Parameters  
NAME sa-283  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20040614  
Time 12.04  
INSTRUM AV500NB  
PROBHD 5 mm TBO BB/1H  
PULPROG zg30  
TD 65536  
SOLVENT Acetone  
NS 16  
DS 0  
SMH 7440.476 Hz  
FIDRES 0.113533 Hz  
AQ 4.4040694 sec  
RG 114  
DW 67.200 usec  
DE 6.00 usec  
TE 300.0 K  
D1 0.01000000 sec

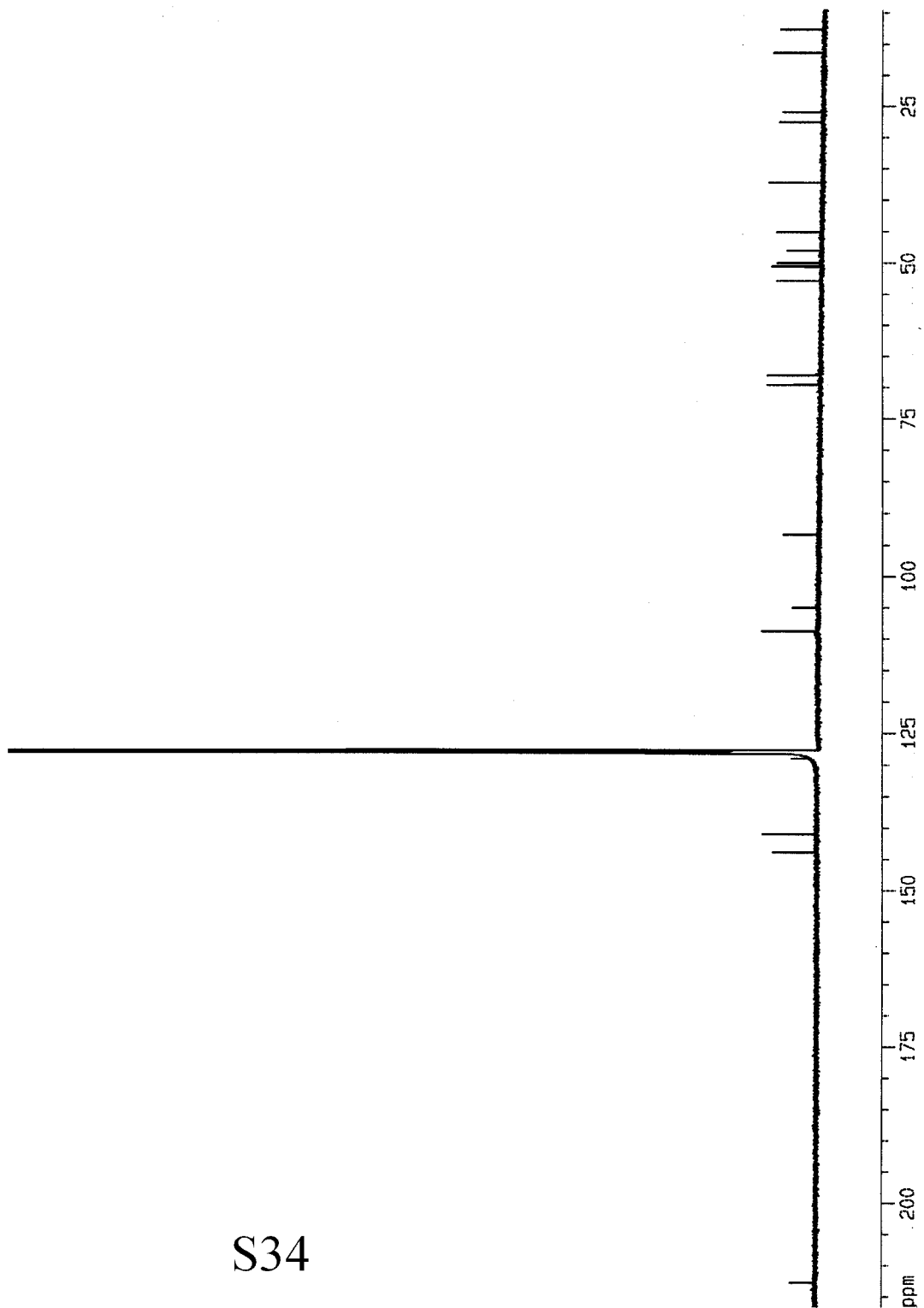
===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 0.00 dB  
SF01 500.132766 MHz

F2 - Processing parameters  
SI 65536  
SF 500.1300049 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
CY 10.00 cm  
FIP 7.689 ppm  
F1 3845.33 Hz  
F2P 0.265 ppm  
F2 132.54 Hz  
PPMCM 0.37118 ppm/cm  
HZCM 185.63913 Hz/cm



<sup>13</sup>C with proton decoupling



Current Data Parameters  
NAME sa-263  
EXPNO 2  
PROCNO 1

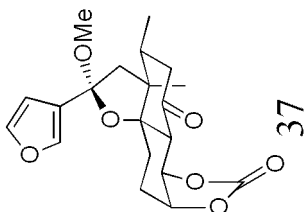
F2 - Acquisition Parameters  
Date\_ 20040614  
Time 12.31  
INSTRUM AV500WB  
PROBHD 5 mm TBO BB/1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 700  
DS 0  
SWH 30030.029 Hz  
FIDRES 0.458222 Hz  
AQ 1.0912244 sec  
RG 3251  
DM 16.650 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.0002000 sec

===== CHANNEL f1 =====  
NUC1 <sup>13</sup>C  
P1 8.00 usec  
PL1 -0.20 dB  
SF01 125.7703643 MHz

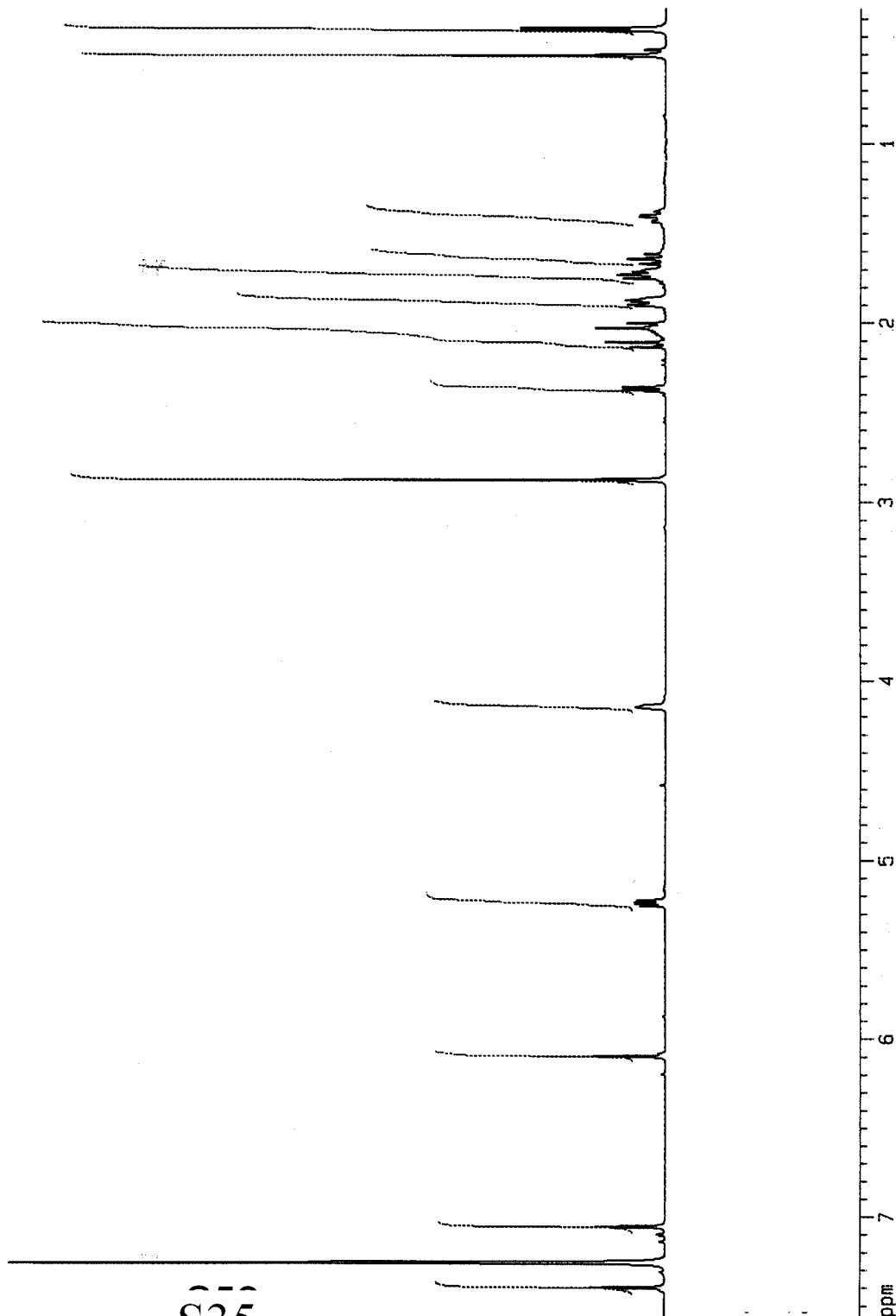
===== CHANNEL f2 =====  
CPOPRG2 waltz16  
NUC2 <sup>1</sup>H  
PCPD2 80.00 usec  
PL2 -0.20 dB  
PL12 14.34 dB  
PL13 14.30 dB  
SF02 500.1320005 MHz

F2 - Processing parameters  
SI 32768  
SF 125.7577663 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
CY 50.00 cm  
FIP 216.702 ppm  
F1 27251.95 Hz  
F2 9.493 ppm  
F2 1193.82 Hz  
PPMCM 10.36044 ppm/cm  
HZCM 1302.90613 Hz/cm



1H NMR



Current Data Parameters  
 NAME sa-288  
 EXPNO 1  
 PROCNO 1

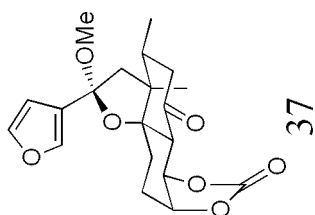
F2 - Acquisition Parameters  
 Date\_ 20040616  
 Time 12.02  
 INSTRUM AV500WB  
 PROBHD 5 mm TBO BB/1H  
 PULPROG zg30  
 TD 65536  
 SOLVENT Acetone  
 NS 16  
 DS 0  
 SMH 7440.476 Hz  
 FIDRES 0.113533 Hz  
 AQ 4.4040694 sec  
 RG 203.2  
 DW 67.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 0.01000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 14.00 usec  
 PL1 0.00 dB  
 SF01 500.132766 MHz

F2 - Processing parameters  
 SI 65536  
 SF 500.1300091 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 10.00 cm  
 FIP 7.561 ppm  
 F1 3781.46 Hz  
 F2P 0.242 ppm  
 F2 120.87 Hz  
 PPMCM 0.36596 ppm/cm  
 HZCM 183.02974 Hz/cm

<sup>13</sup>C with proton decoupling



S36

```

Current Data Parameters
NAME      sa-288
EXPNO     2
PROCNO    1

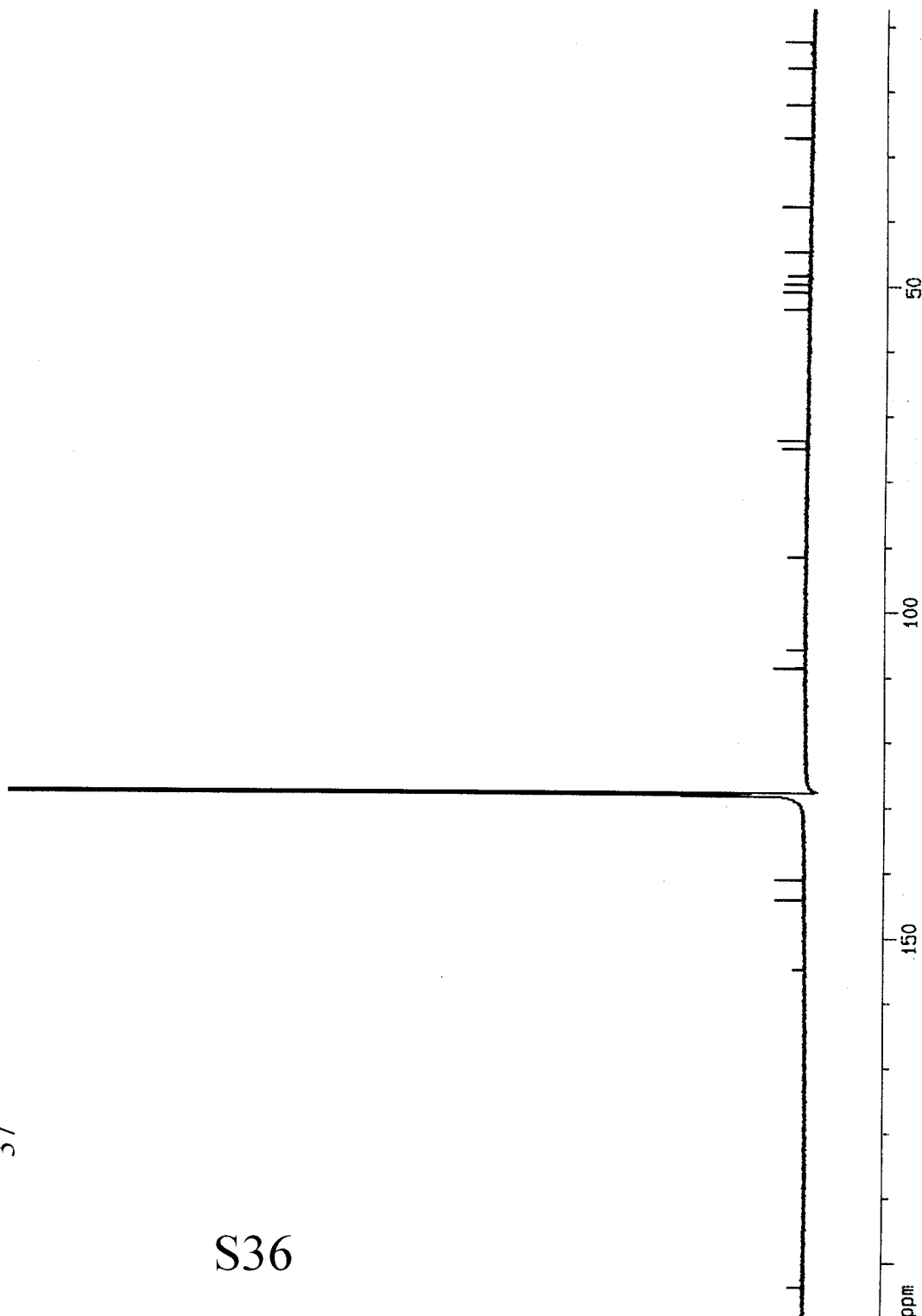
F2 - Acquisition Parameters
Date_     20040616
Time      12.42
INSTRUM   AV500WB
PROBHD    5 mm TBO BB/1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         0
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912244 sec
RG         3251
DM         16.650 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
d11        0.03000000 sec
d12        0.0002000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         8.00 usec
PL1        -0.20 dB
SF01       125.7703643 MHz

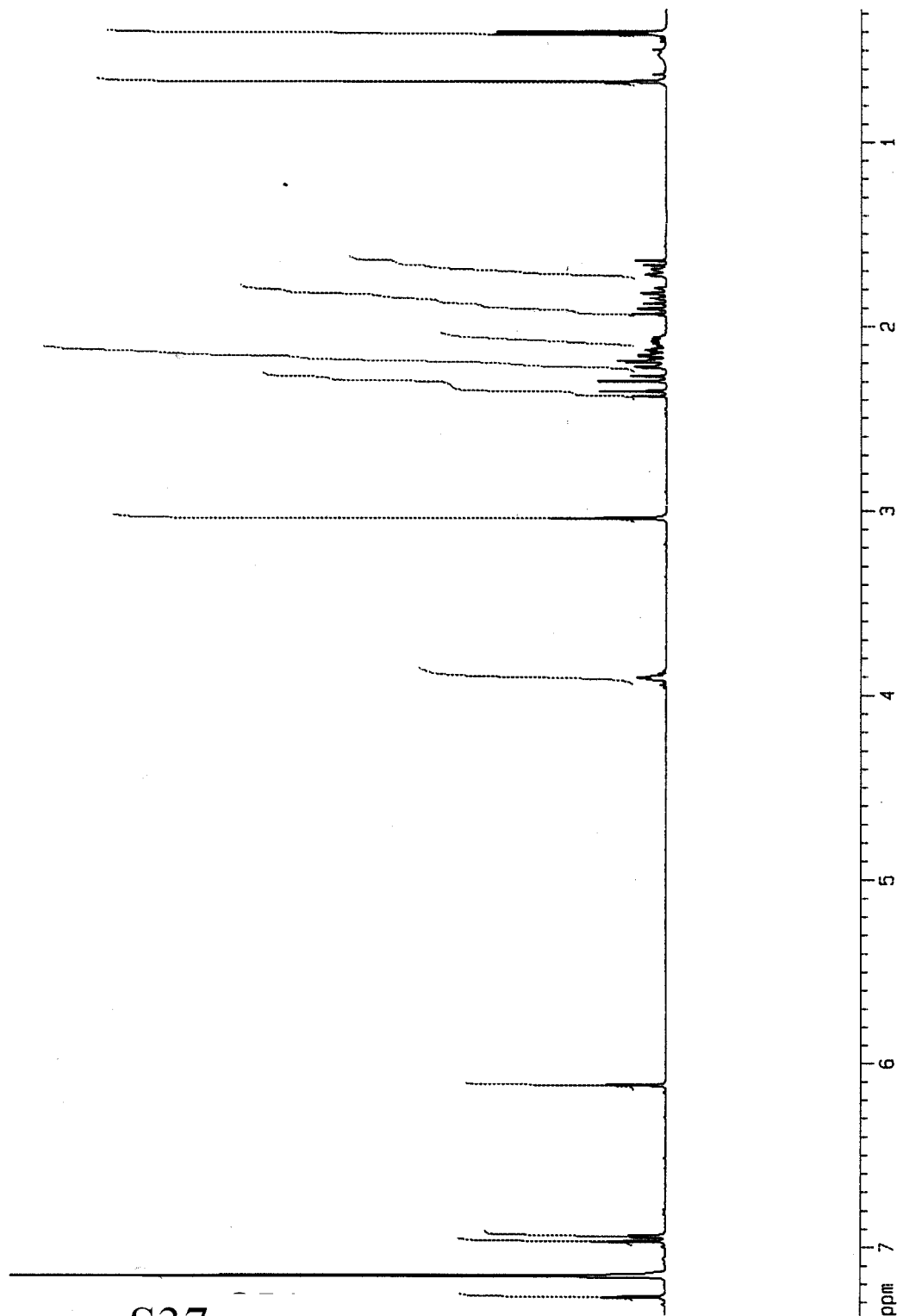
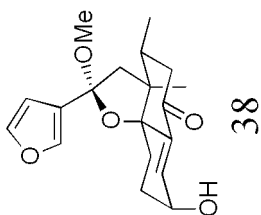
===== CHANNEL f2 =====
CPOPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2        -0.20 dB
PL12       14.94 dB
PL13       14.90 dB
SF02       500.1320005 MHz

F2 - Processing parameters
SI         32768
SF         125.7577663 MHz
WDW        EN
SSB        0
LB         1.00 Hz
GB         0
PC         1.00

1D NMR plot parameters
CX         20.00 cm
CY         40.00 cm
FIP        208.327 ppm
F1         26198.78 Hz
F2         7.340 ppm
F20        923.01 Hz
PNUC1      10.04939 ppm/cm
H2C1M      1263.78882 Hz/cm
    
```



<sup>1</sup>H NMR



Current Data Parameters  
NAME sa-284  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

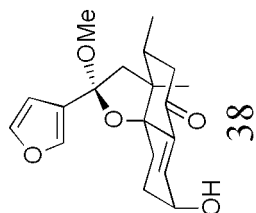
Date\_ 20040615  
Time 12.06  
INSTRUM AV500WB  
PROBHD 5 mm TBO BB/1H  
PULPROG zg30  
TD 65536  
SOLVENT Acetone  
NS 16  
DS 0  
SWH 7440.476 Hz  
FIDRES 0.113533 Hz  
AQ 4.4040694 sec  
RG 128  
DM 67.200 usec  
DE 6.00 usec  
TE 300.0 K  
D1 0.01000000 sec

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

NUC1 <sup>1</sup>H  
P1 14.00 usec  
PL1 0.00 dB  
SF01 500.1327766 MHz

F2 - Processing parameters

SI 65536  
SF 500.1300591 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00  
1D NMR plot parameters  
CX 20.00 cm  
CY 10.00 cm  
F1P 7.372 ppm  
F1 3686.71 Hz  
F2P 0.276 ppm  
F2 137.95 Hz  
PPMCM 0.35478 ppm/cm  
HZCM 177.43820 Hz/cm



<sup>13</sup>C with proton decoupling

```

Current Data Parameters
NAME      sa-284
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20040615
Time      13.03
INSTRUM   AV500MB
PROBHD    5 mm TBO BB/1H
PULPROG   zgpg30
TD         65536
SOLVENT   H2O+D2O
NS         1024
DS         0
SMH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912244 sec
RG         3251.
DE         16.650 usec
TE         300.0 K
D1         1.00000000 sec
d11        0.03000000 sec
d12        0.00020000 sec

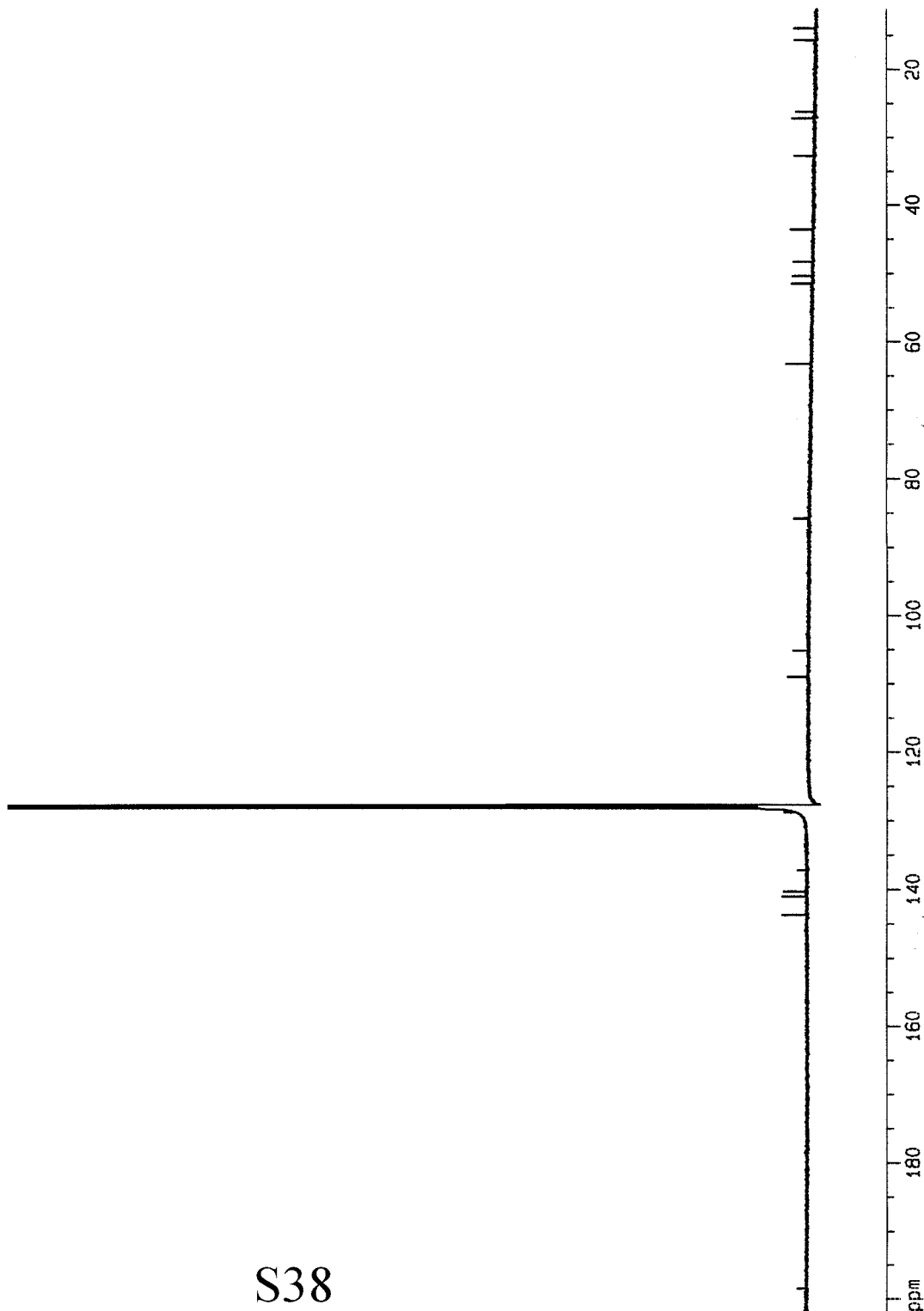
===== CHANNEL f1 =====
NUC1       13C
P1         8.00 usec
PL1        -0.20 dB
SF01       125.7703643 MHz

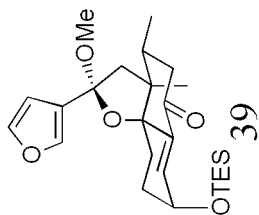
===== CHANNEL f2 =====
CPOPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -0.20 dB
PL12       14.94 dB
PL13       14.90 dB
SF02       500.1320005 MHz

F2 - Processing parameters
SI         32768
SF         125.7577654 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00

1D NMR plot parameters
CX         20.00 cm
CY         40.00 cm
FIP        201.874 ppm
F1         25387.27 Hz
F2         11.175 ppm
F2         1405.37 Hz
PPHNM      9.53496 ppm/cm
HZCM       1199.09497 Hz/cm

```





<sup>1</sup>H NMR

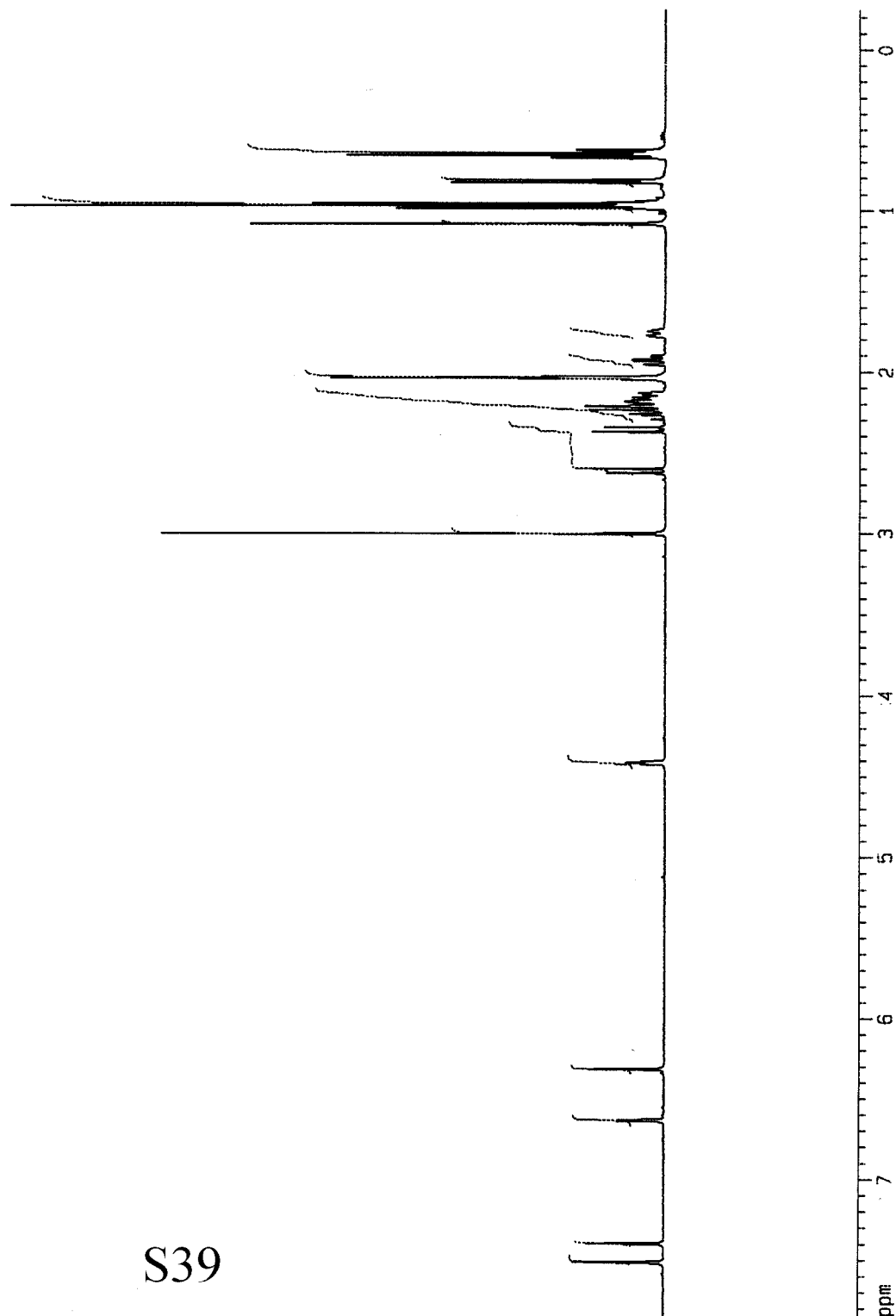
Current Data Parameters  
 NAME sa-293  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20040621  
 Time 13.15  
 INSTRUM AV500WB  
 PROBHD 5 mm TBO BB/1H  
 PULPROG zg30  
 TD 65536  
 SOLVENT Acetone  
 NS 16  
 DS 0  
 SWH 7440.476 Hz  
 FIDRES 0.113533 Hz  
 AQ 4.4040694 sec  
 RG 128  
 DW 67.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 0.01000000 sec

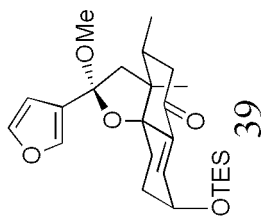
===== CHANNEL f1 =====  
 NUC1 <sup>1</sup>H  
 P1 14.00 usec  
 PL1 0.00 dB  
 SF01 500.132766 MHz

F2 - Processing parameters  
 SI 65536  
 SF 500.1300149 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

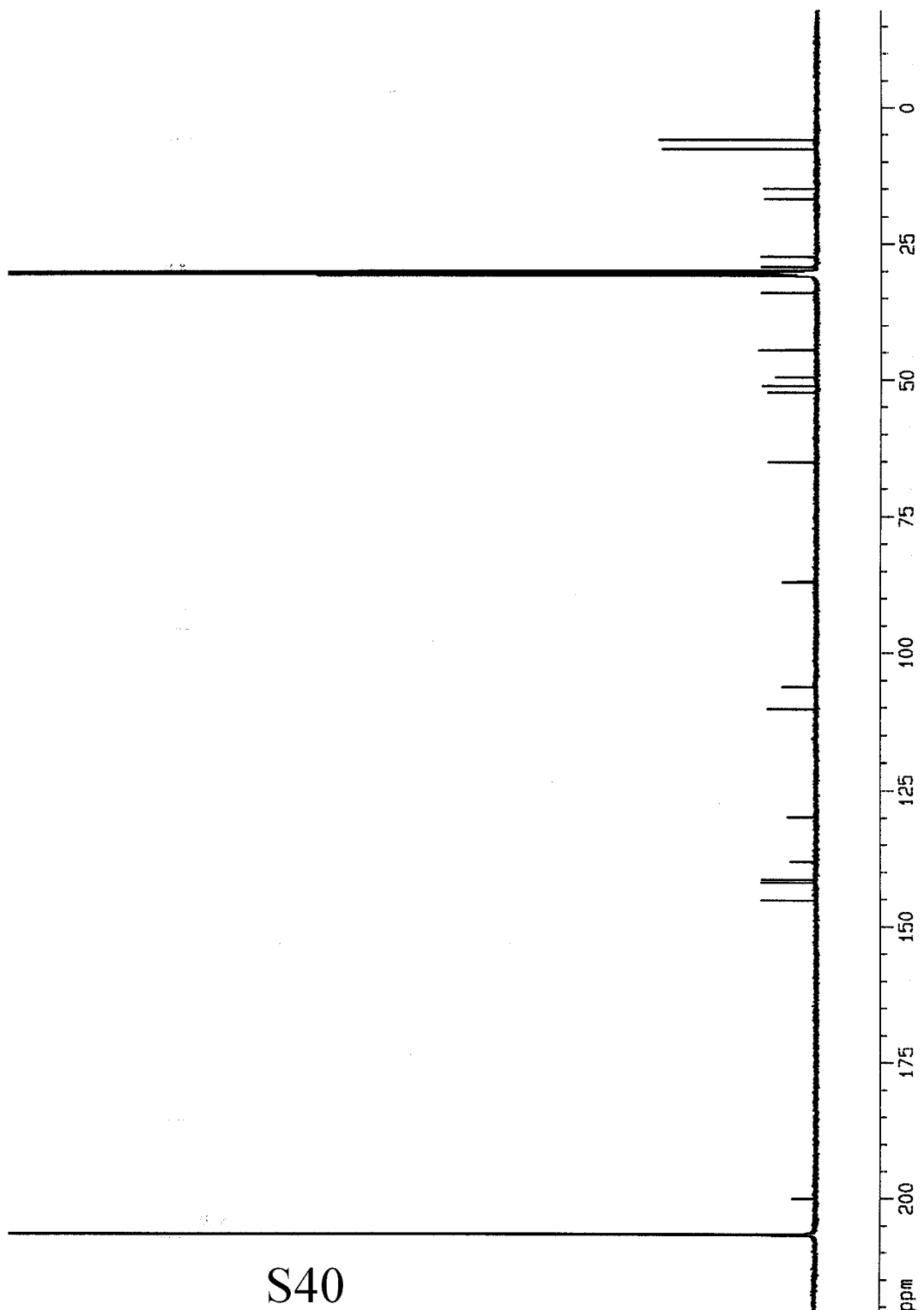
1D NMR plot parameters  
 CX 20.00 cm  
 CY 10.00 cm  
 F1P 7.848 ppm  
 F1 3924.78 Hz  
 F2P -0.247 ppm  
 F2 -123.49 Hz  
 PPMCM 0.40472 ppm/cm  
 HZCM 202.41374 Hz/cm



S39



<sup>13</sup>C with proton decoupling



Current Data Parameters  
 NAME sa-293  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20040621  
 Time 13:21  
 INSTRUM AV500HB  
 PROBHD 5 mm TBO BB/1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1800  
 DS 0  
 SMH 30030.029 Hz  
 FIDRES 0.458222 Hz  
 AQ 1.0912244 sec  
 RG 3251  
 DM 16.650 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 d11 0.03000000 sec  
 d12 0.0002000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 8.00 usec  
 PL1 -0.20 dB  
 SF01 125.7703643 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -0.20 dB  
 PL12 14.94 dB  
 PL13 14.90 dB  
 SF02 500.1320005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 125.7576071 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 0.50

1D NMR plot parameters  
 CX 20.00 cm  
 CY 50.00 cm  
 FIP 220.839 ppm  
 F1 27772.23 Hz  
 F2 -17.954 ppm  
 F2 -2257.80 Hz  
 PPMCM 11.93965 ppm/cm  
 HZCM 1501.50146 Hz/cm

S40