

Supplementary data for article:

Krstić, N. M.; Pavlović, V. D.; Novaković, I. T.; Matic, I. Z.; Sladić, D. Synthesis, Characterization and Biological Evaluation of Some Novel P-Heterocyclic Androst-4-Ene Derivatives. *Molecular Diversity* **2013**, *17* (3), 547–561. <https://doi.org/10.1007/s11030-013-9455-9>

H-139-S

Solvent: c6d6
Ambient temperature
GEMINI-200 "ppm"

PULSE SEQUENCE

Relax. delay arrayed
1st pulse arrayed
2nd pulse 54.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz

Arrayed repetitions

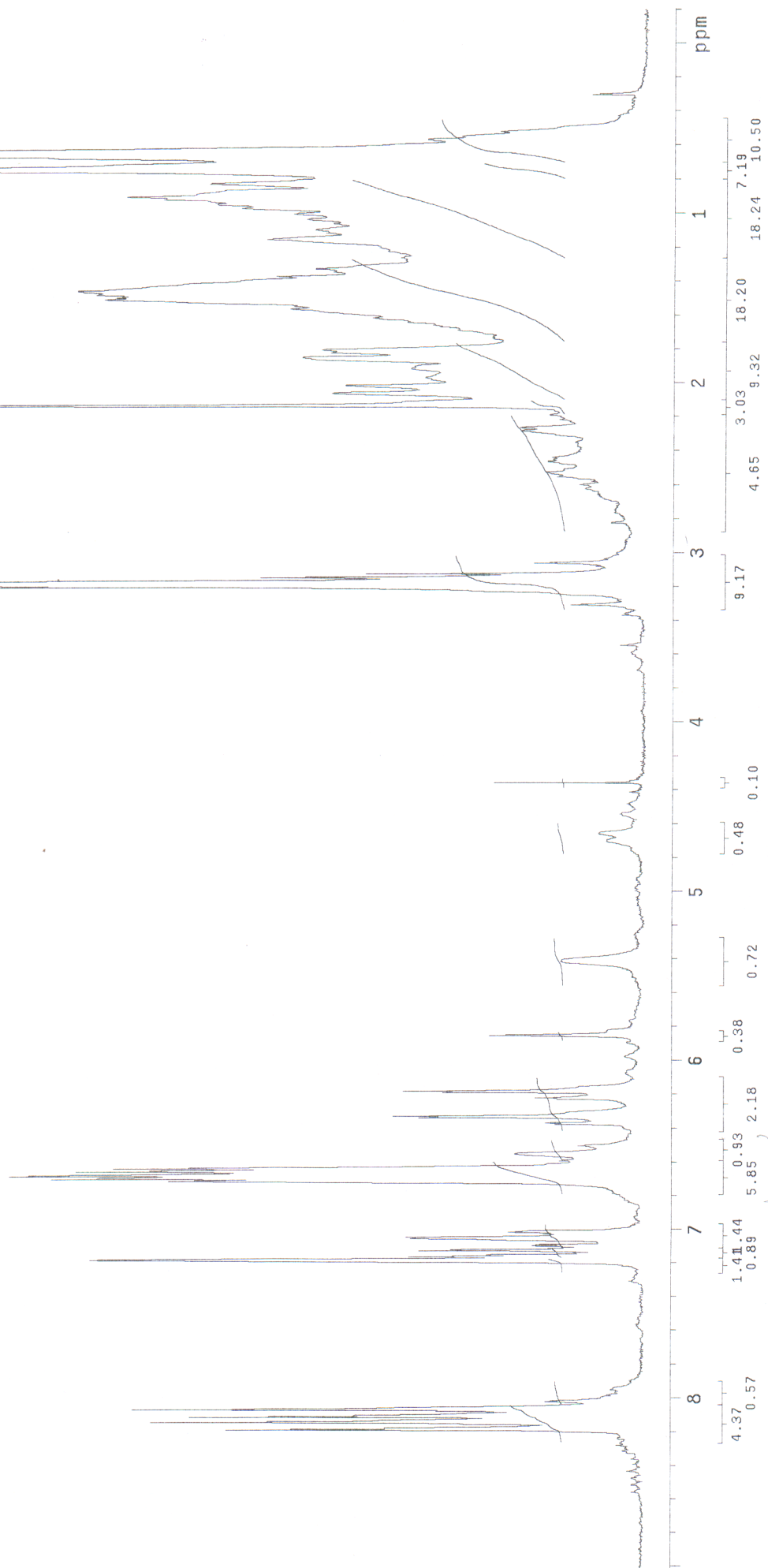
OBSERVE HI, 199.9711003 MHZ

DATA PROCESSING

Line broadening 0.2 Hz

FT size 16384

Total time 1 minute



H-139-S

Solvent: c6d6
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE

Relax. delay arrayed
1st pulse arrayed
2nd pulse 81.8 degrees
Acq. time 1.067 sec
Width 15000.0 Hz

Arrayed repetitions
OBSERVE C13, 50.2826945 MHz
DECUPLE H1, 199.9712987 MHz

Power 0 dB

continuously on

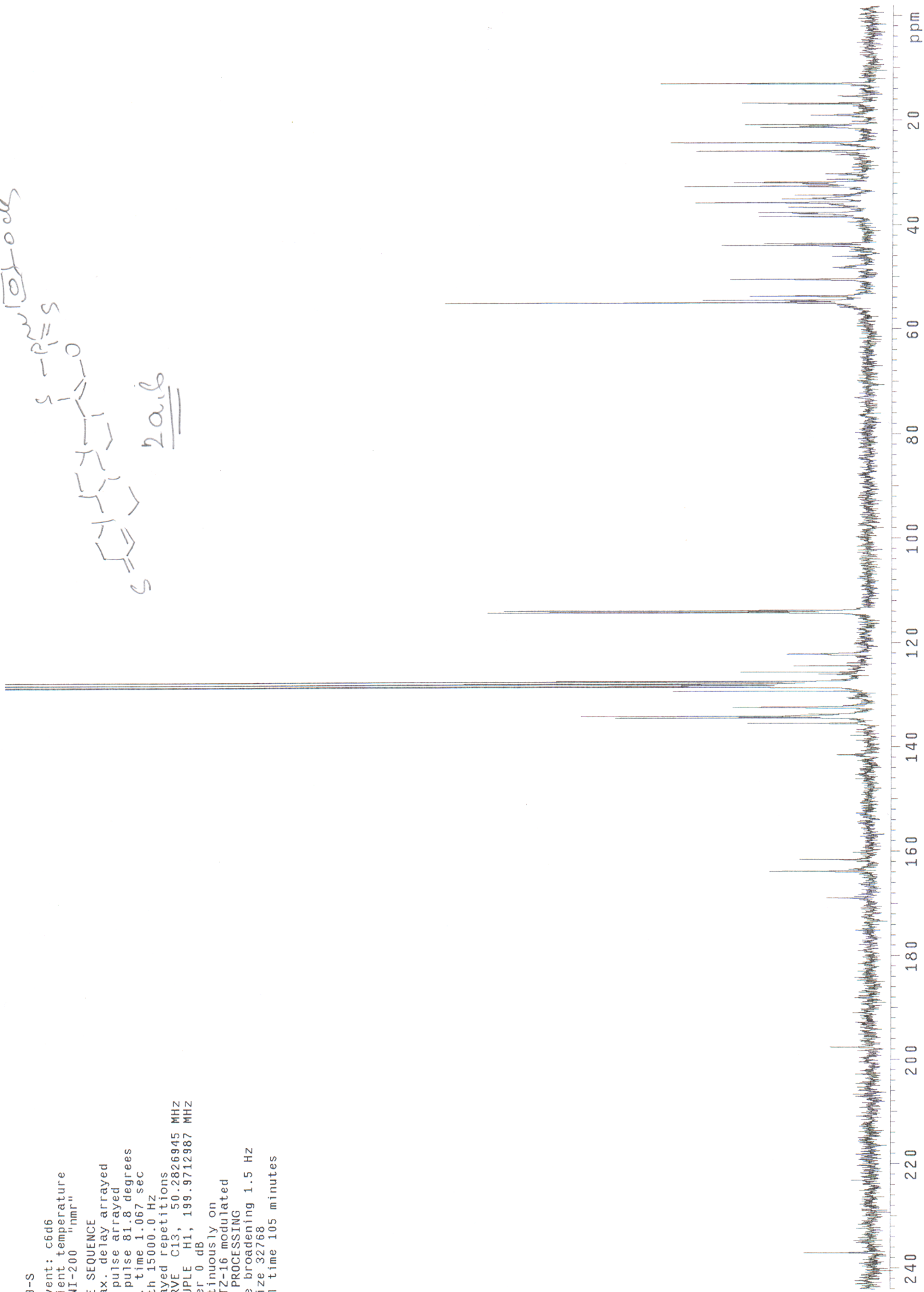
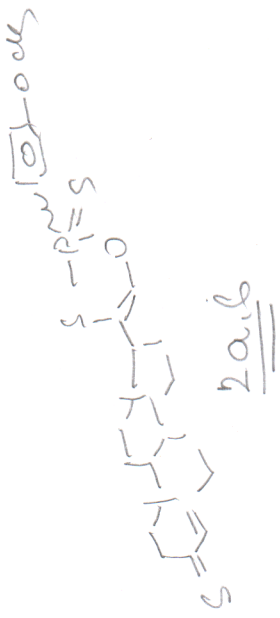
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.5 Hz

F1 size 32768

Total time 105 minutes

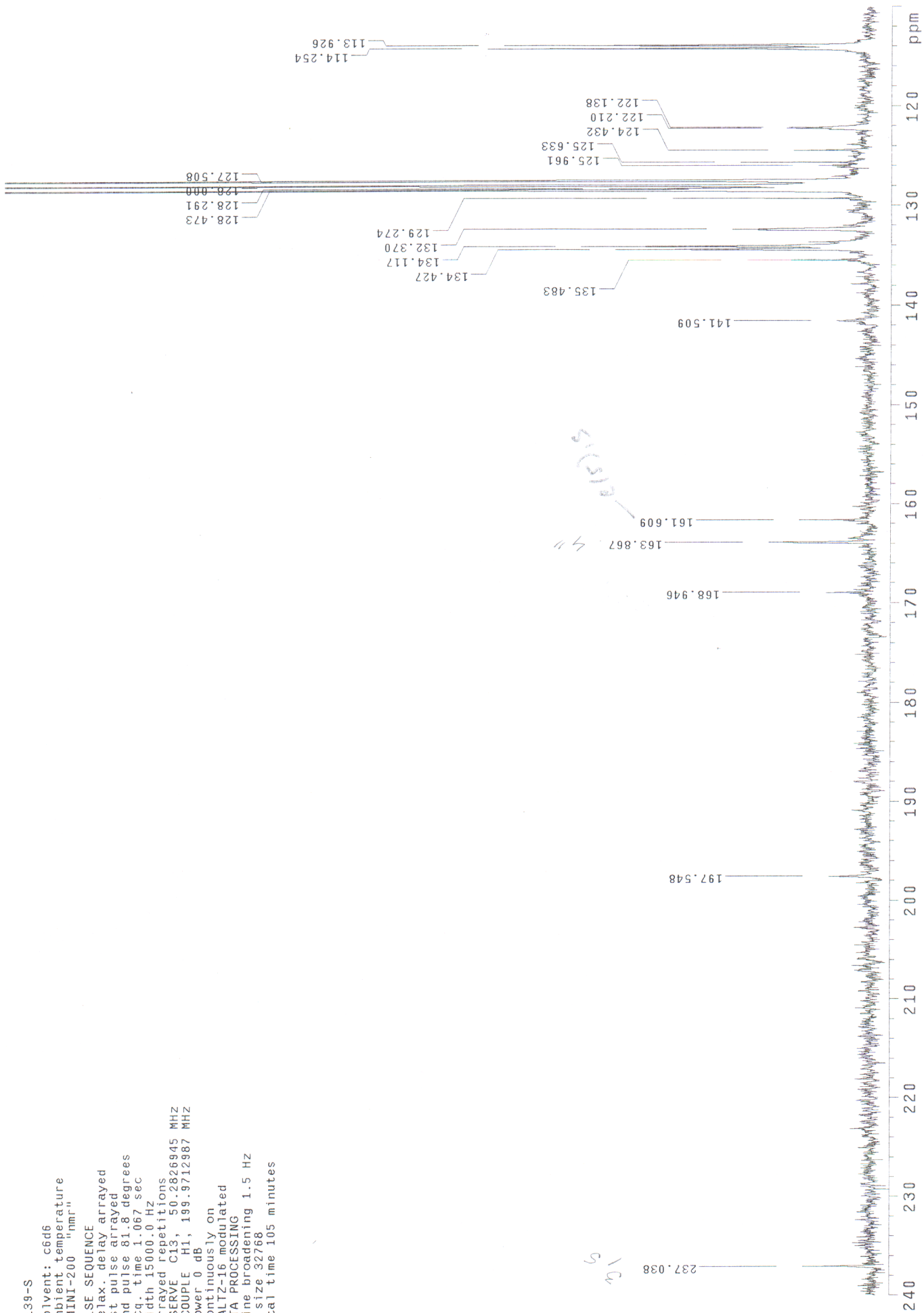


H-139-S

Solvent: c6d6
Ambient temperature
GENI-200 "nmr"

PULSE SEQUENCE

Relax. delay arrayed
1st pulse arrayed
2nd pulse 81.8 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2826945 MHZ
DECOUPLE H1, 199.9712987 MHZ
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 105 minutes



H-137-S

Solvent: c6d6
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE

Relax. delay arrayed
1st pulse arrayed
2nd pulse 54.0 degrees
Acq. time 1.391 SEC
Width 4600.0 HZ
Arrayed repetitions

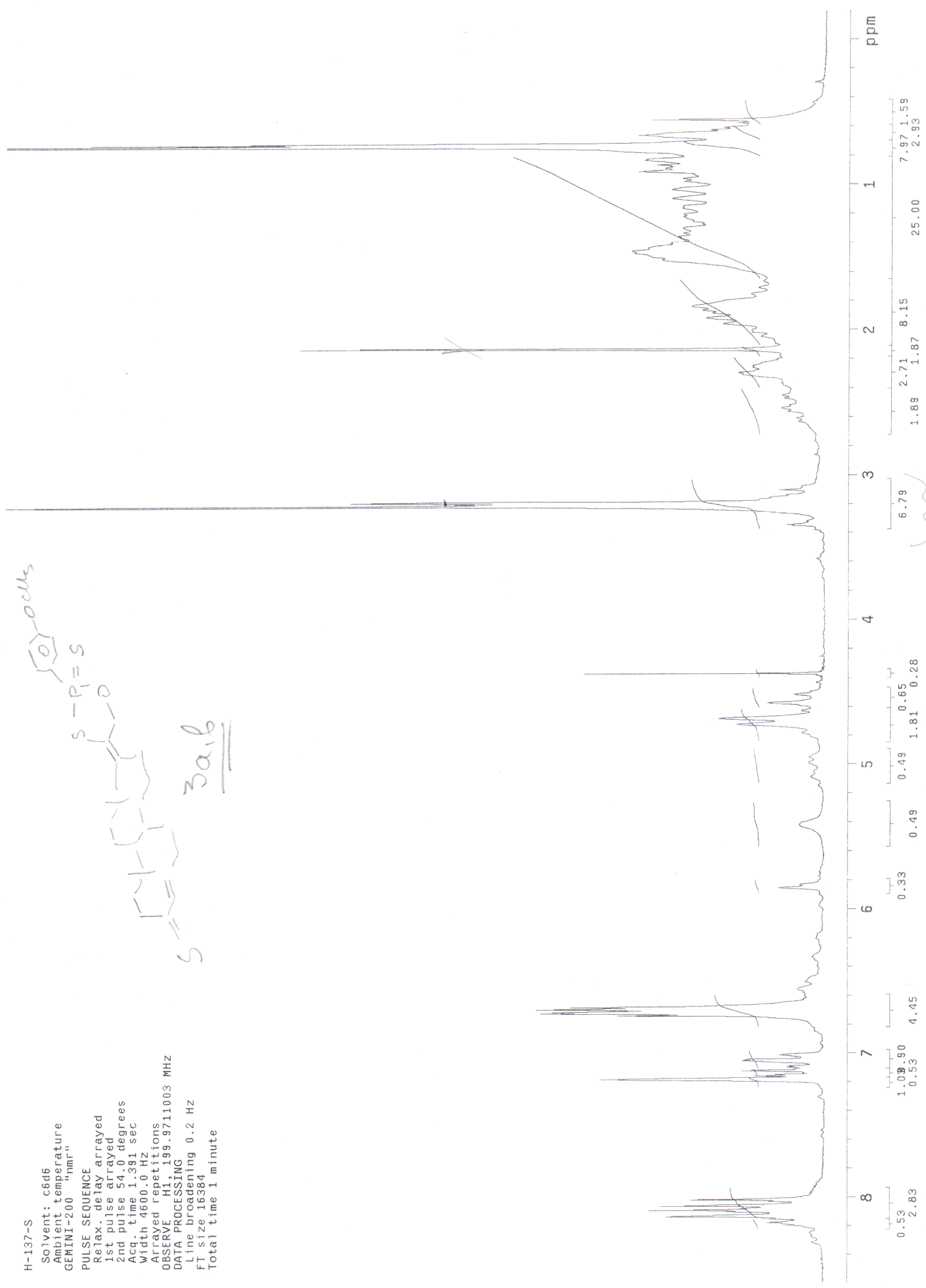
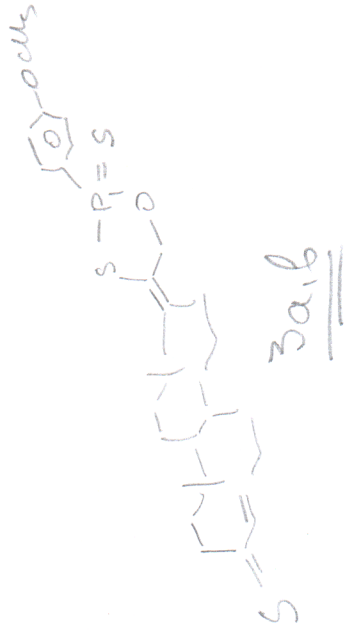
OBSERVE HI, 199.9711003 MHZ

DATA PROCESSING

Line broadening 0.2 HZ

FT size 16384

Total time 1 minute



ppm

7.97 1.59
7.93 2.93
25.00
8.15
2.71 1.87
1.89
6.79
0.65
0.49 1.81 0.28
0.49
0.33
4.45
1.03 0.90
0.53
0.53 2.83

WV

3.36

H-137-S

Solvent: c6d6
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE

Relax. delay arrayed
1st pulse arrayed
2nd pulse 81.8 degrees
Acq. time 1.067 sec
Width 15000.0 Hz

Arrayed repetitions
OBSERVE C13, 50.2826945 MHZ
DECOUPLE H1, 199.9712987 MHZ
Power 0 dB

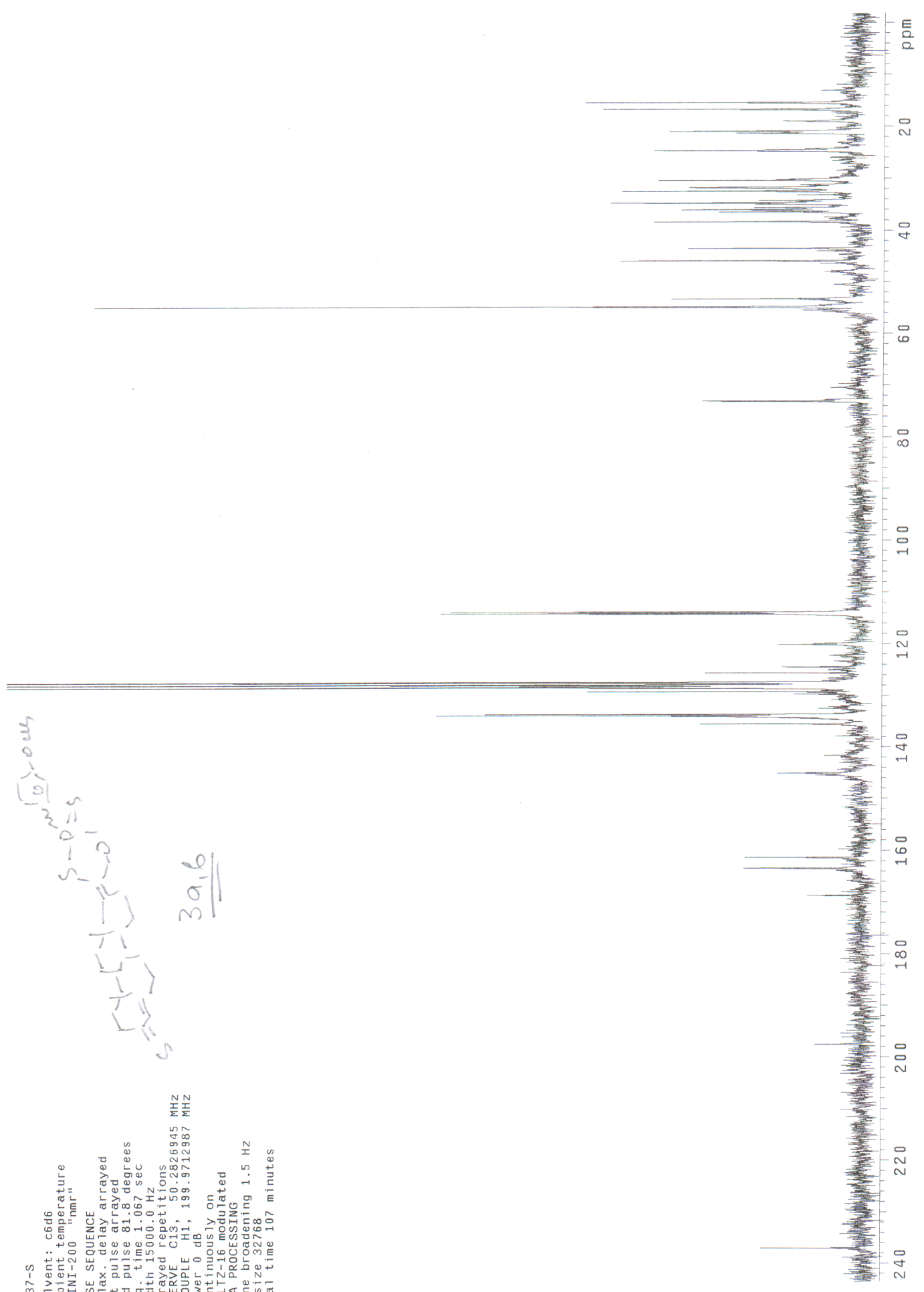
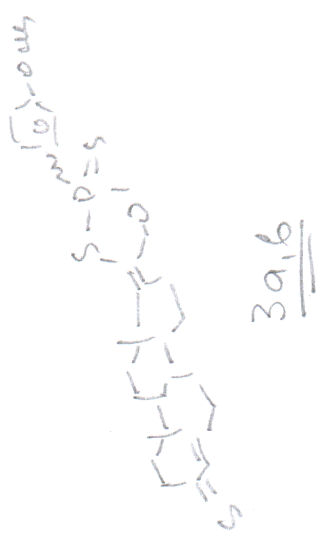
continuously on
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.5 Hz

F1 size 32768

Total time 107 minutes



H-137-S

Solvent: c6d6
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE

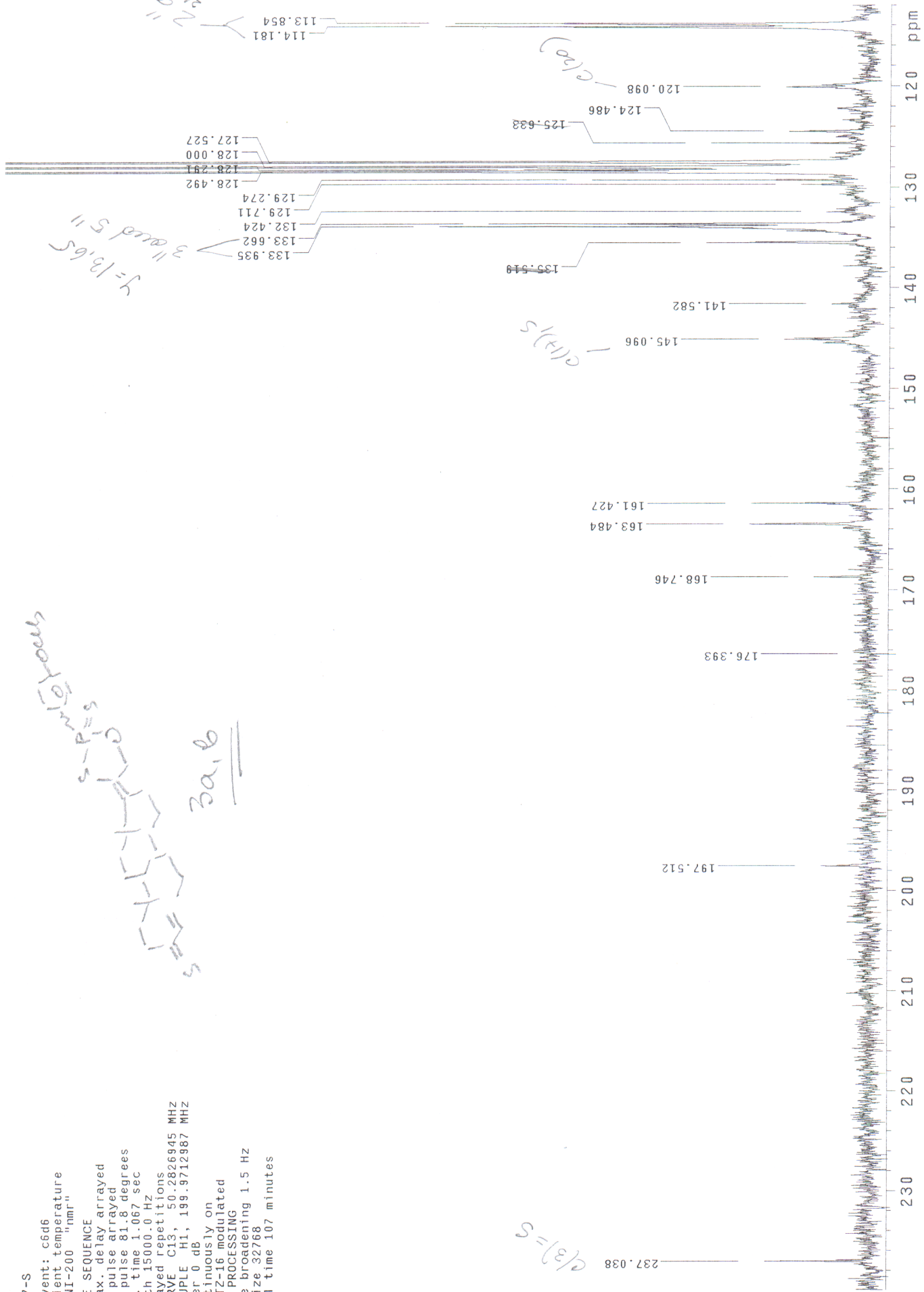
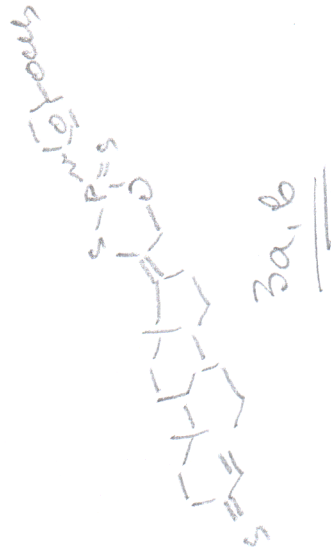
Relax. delay arrayed
1st pulse arrayed
2nd pulse 81.8 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2826945 MHZ
DECOUPLE H1, 199.9712987 MHZ
Power 0 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.5 Hz
FT size 32768
Total time 107 minutes



$\delta(13) = 5$

$\delta(13) = 5$

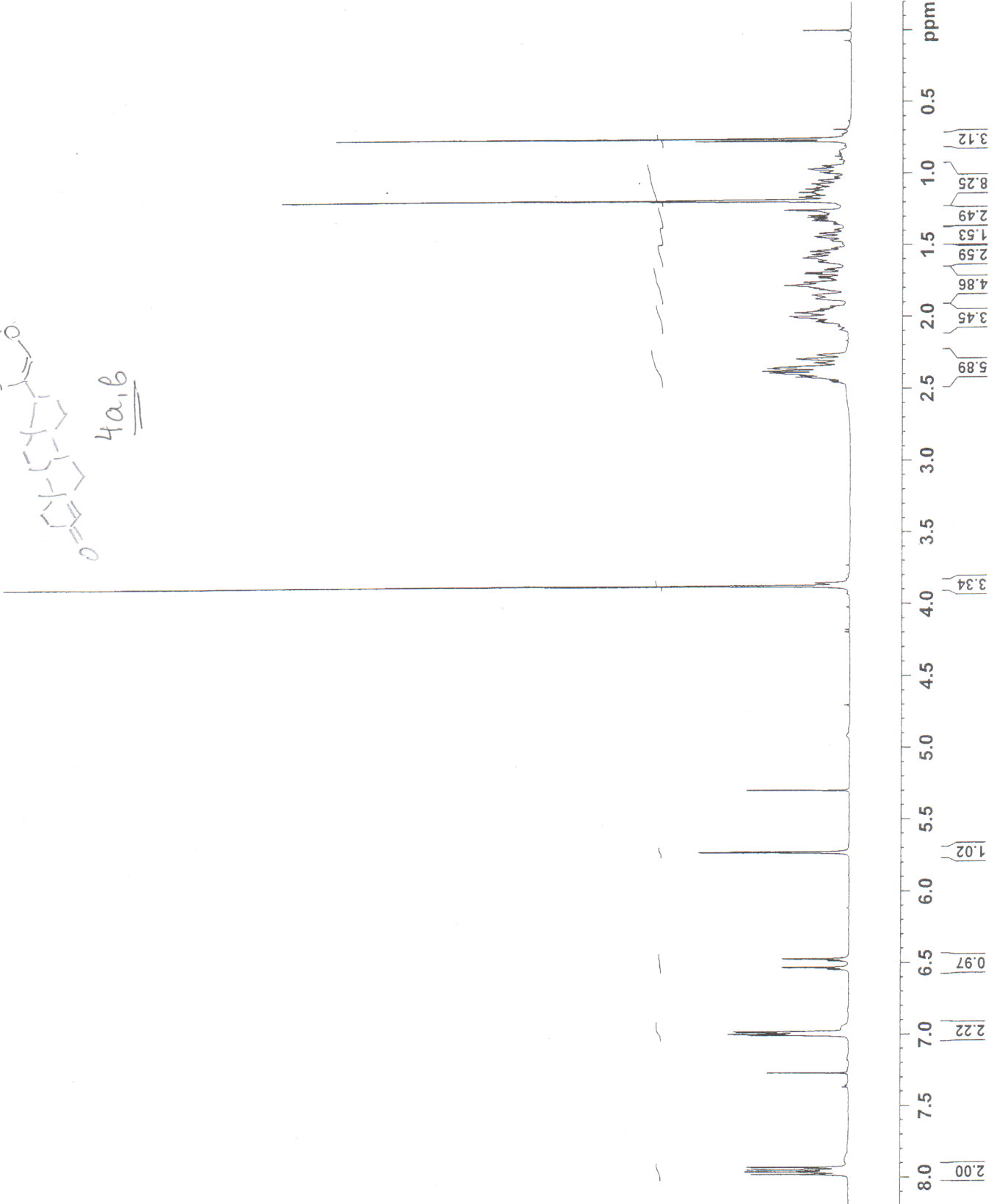
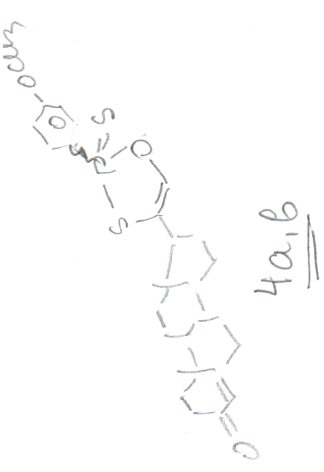
$\delta(13) = 5$

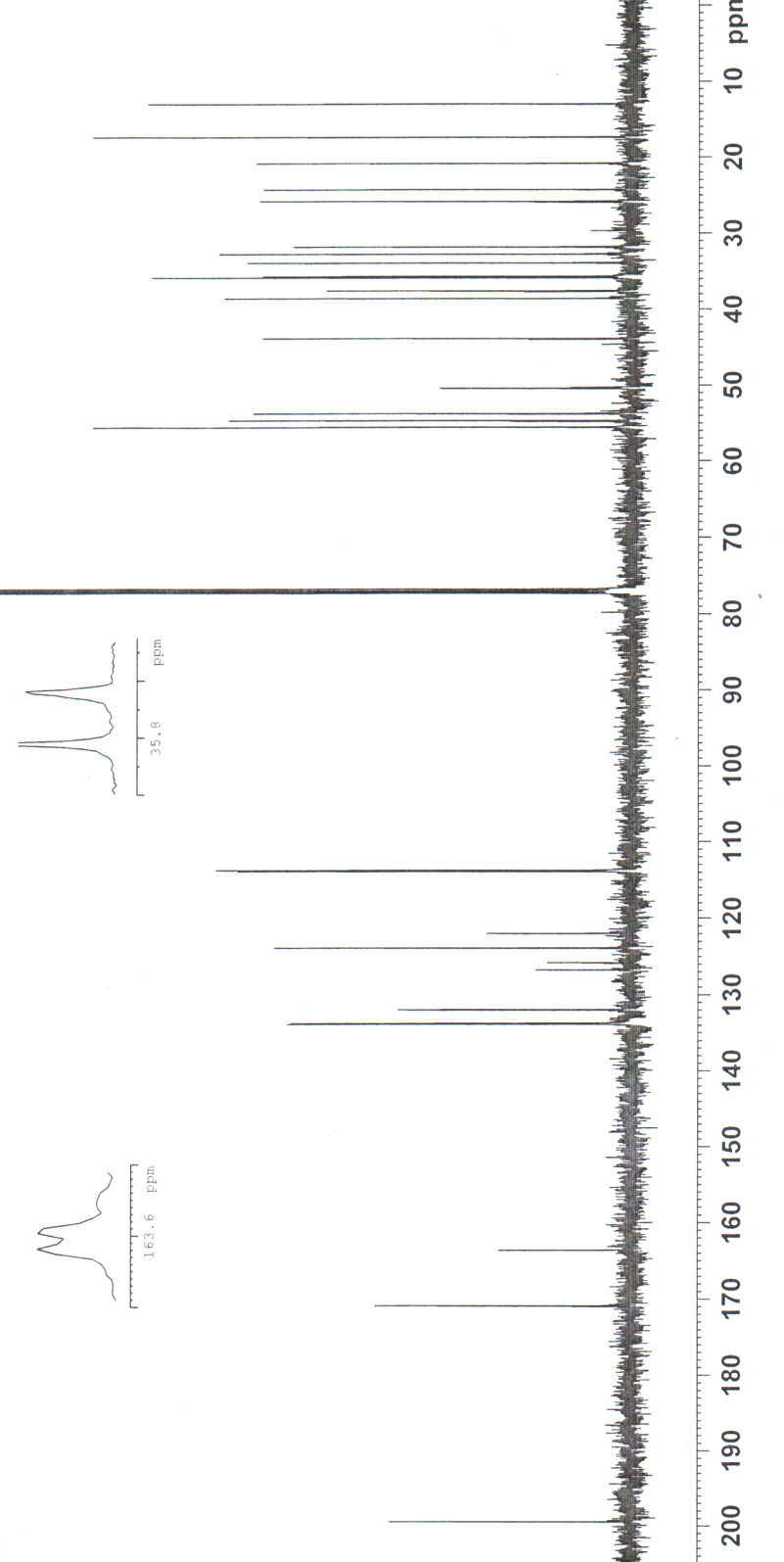
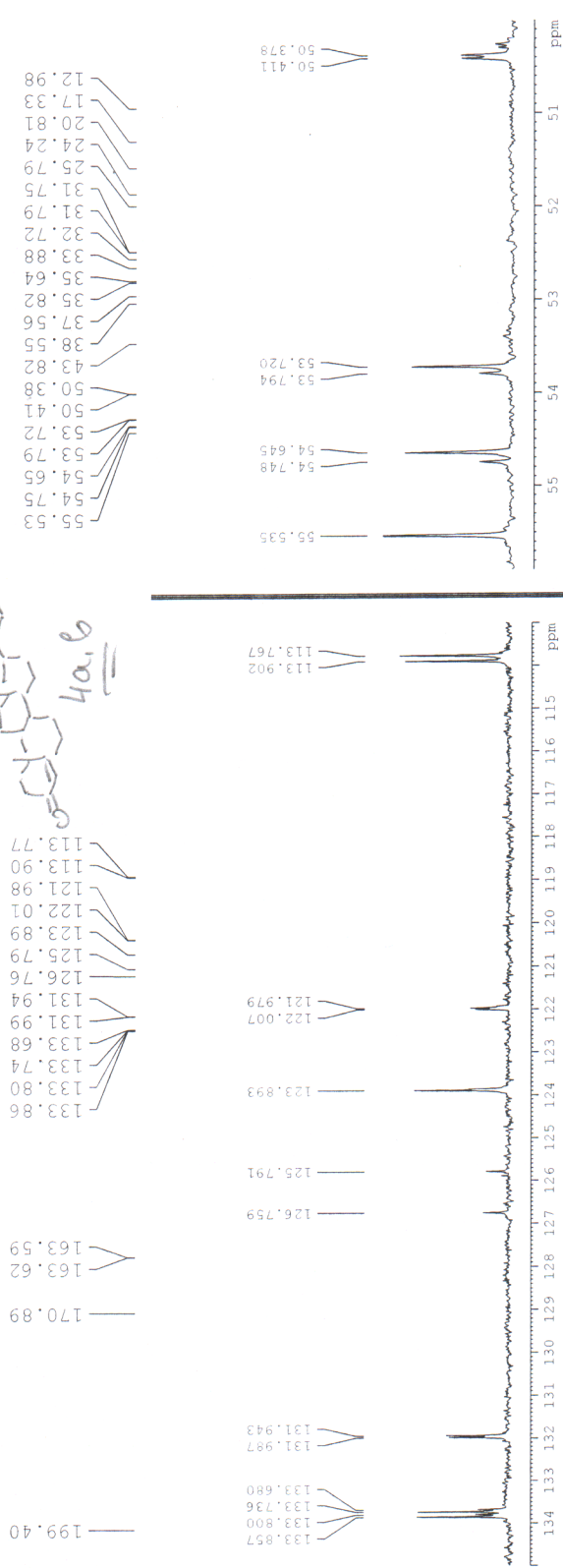
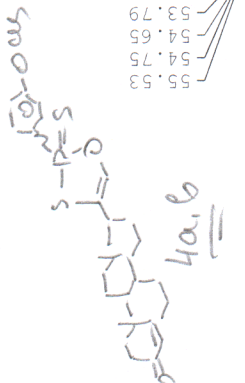
2 read 6''
x=16.35
y=13.65

3 read 5''
y=13.65

NAME H-139
 EXPNO 1
 PROCNO 1
 Date 20110418
 Time 11.11
 INSTRUM Spect
 PROBHD 5 mm BBO BB-IH
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 4882.813 Hz
 FIDRES 0.149012 Hz
 AQ 3.3554933 sec
 RG 57
 DW 102.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.35 usec
 PL1 0.00 dB
 PL1W 27.37956238 W
 SFO1 500.2620692 MHz
 SI 32768
 SF 500.2600090 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00



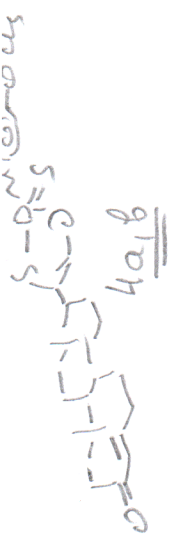


NAME H-139
 EXPNO 2
 PROCNO 1
 Date_ 20110418
 Time_ 11.24
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 265
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.908261 Hz
 AQ 0.5505524 sec
 RG 1030
 DW 16.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

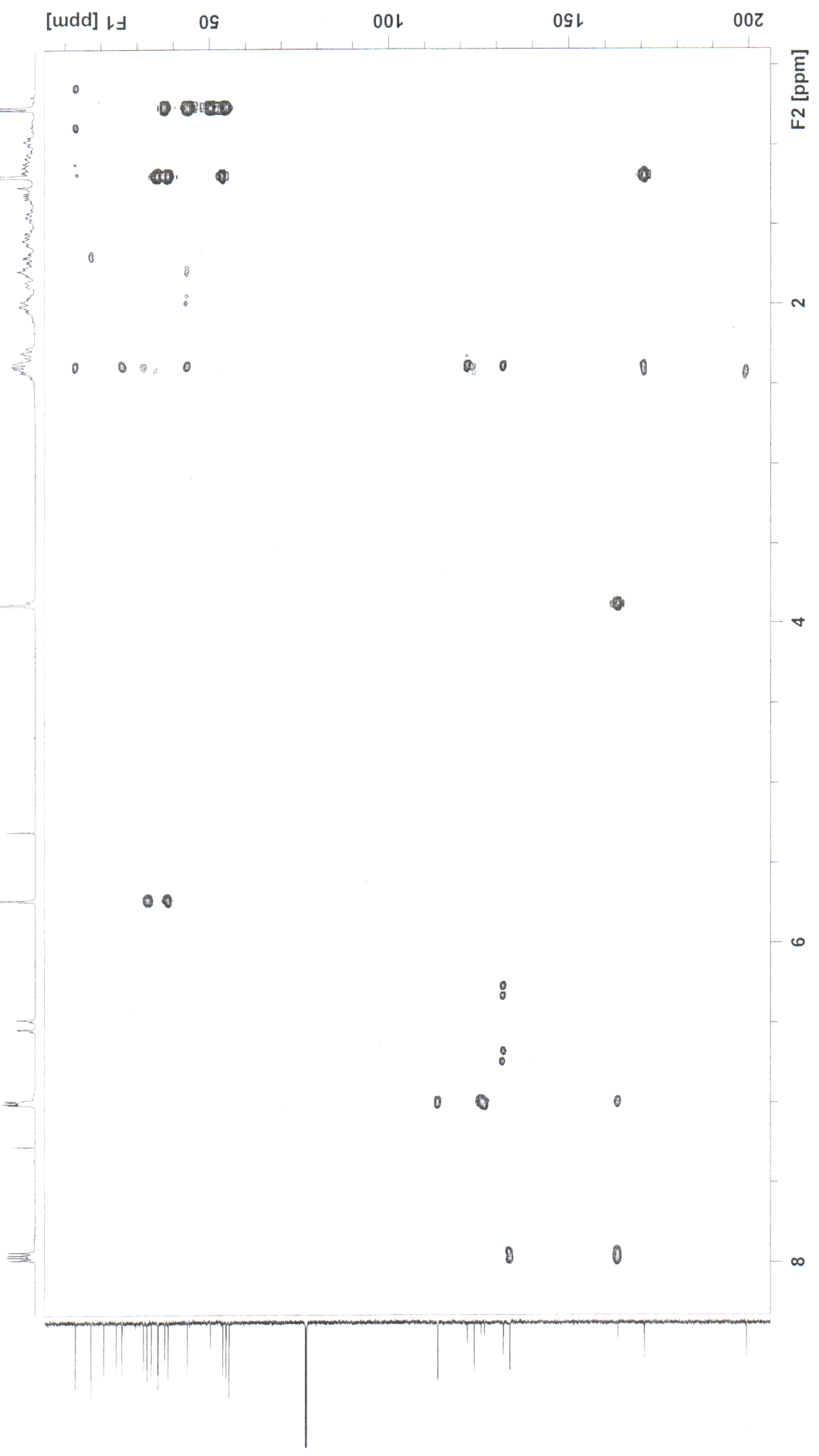
CHANNEL f1 =====
 NUC1 13C
 P1 11.50 usec
 PL1 3.00 dB
 PL1W 32.22848892 W
 SFO1 125.8043140 MHz

CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.20 dB
 PL12 18.40 dB
 PL13 18.40 dB
 PL2W 20.76952171 W
 PL12W 0.39575511 W
 PL13W 0.39575511 W
 SFO2 500.2620691 MHz
 SI 32768
 SF 125.7904858 MHz
 WDW EM
 SSB 0
 LB 1.50 Hz
 GB 0
 PC 1.40

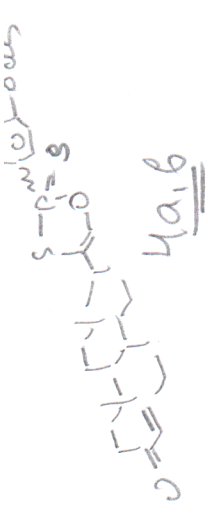
HMBC



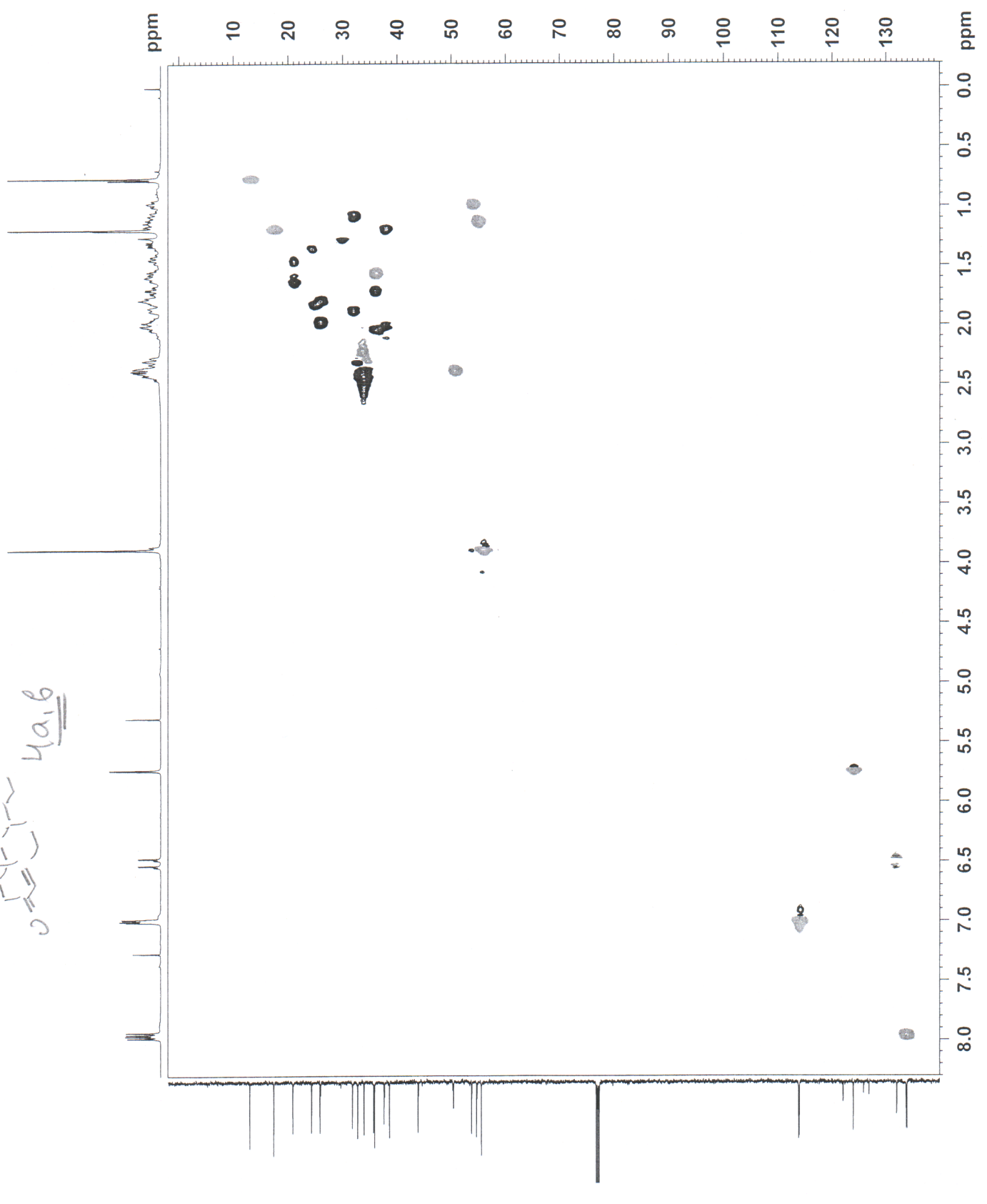
H-139 5 1 C:\Bruker\TOPSPIN\BBO_new Guest



Hsac



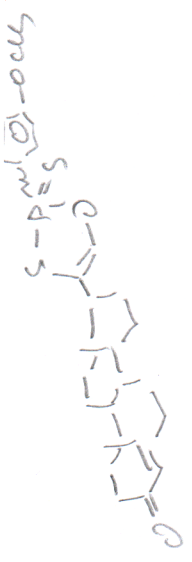
Hsac



```

NAME H-139
EXPNO 4
PROCNO 1
Date_ 20110418
Time 11.37
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG hsqcdecoupl
TD 65536
SOLVENT CDCl3
NS 16
DS 16
SWH 4882.813 Hz
FIDRES 4.768372 Hz
AQ 0.1049076 sec
RG 2050
DE 102.400 usec
TE 298.0 K
CNS1 145.0000000
D0 0.0000300 sec
D1 2.0000000 sec
D2 0.00172414 sec
D3 0.03000000 sec
D4 0.00000000 sec
D5 0.00000400 sec
D6 0.00920000 sec
D7 0.00364615 sec
D8 0.00001805 sec
D9 0.00000000 sec
D10 0.00000000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 9.35 usec
PL1 18.70 usec
PL1W 0.00 dB
SFO1 27.37956238 W
SFO1 500.2620691 MHz
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P2 11.50 usec
PL2 23.00 usec
PL2W 70.00 usec
PCPD2 3.00 dB
PL1L 18.699 dB
PL1W 1.03191831 W
SFO2 125.8030560 MHz
===== GRADIENT CHANNEL =====
GENAM1 SINE.100
GENAM2 SINE.100
GENAM3 SINE.100
GFZ1 30.00
GFZ2 80.00
GFZ3 20.10
P16 1000.00 usec
NDO 2
TD 128
SFO1 125.8031 MHz
FIDRES 216.223999 Hz
SW 220.000 ppm
F2MODE States-TpPI
SI 1024
SF 500.260000 MHz
SSB QSINE
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 States-TpPI
SF 125.7904528 MHz
SSB QSINE
LB 0.00 Hz
GB 0
  
```

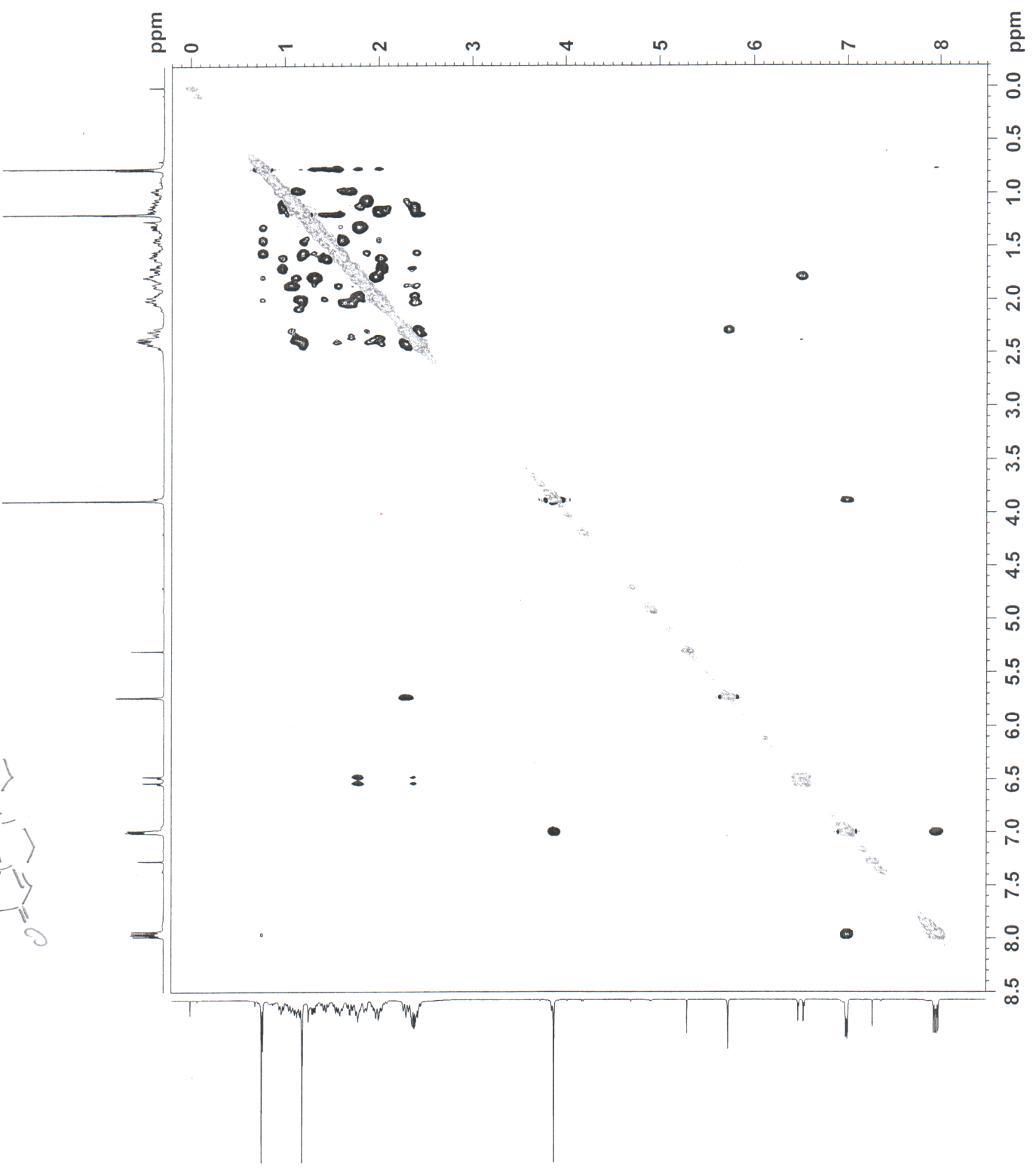
NOESY



```

NAME H-139
EXPNO 6
PROCNO 1
Date_ 20110418
Time 12.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG noesyph
TD 1024
SOLVENT CDCl3
NS 8
DS 16
SWH 4882.813 Hz
FIDRES 4.768372 Hz
AQ 0.1049076 sec
RG 40.3
DW 102.400 usec
DE 6.50 usec
TE 298.0 K
D0 0.00009050 sec
D1 2.00000000 sec
D8 1.00000000 sec
INO 0.00020480 sec
=====
CHANNEL f1
=====
NUC1 1H
P1 9.35 usec
PL1 0.00 dB
PL1W 27.37956238 W
SF01 500.2620691 MHz
ND0 1
TD 128
SF01 500.2621 MHz
FIDRES 38.146938 Hz
SW 9.761 ppm
F0MODE States-TPPI
SI 512
SF 500.2600044 MHz
SSB QSIINE
LB 2
GB 0.00 Hz
PC 1.00
SI 512
MC2 States-TPPI
SF 500.2600043 MHz
SSB QSIINE
LB 2
GB 0.00 Hz

```



NOESY

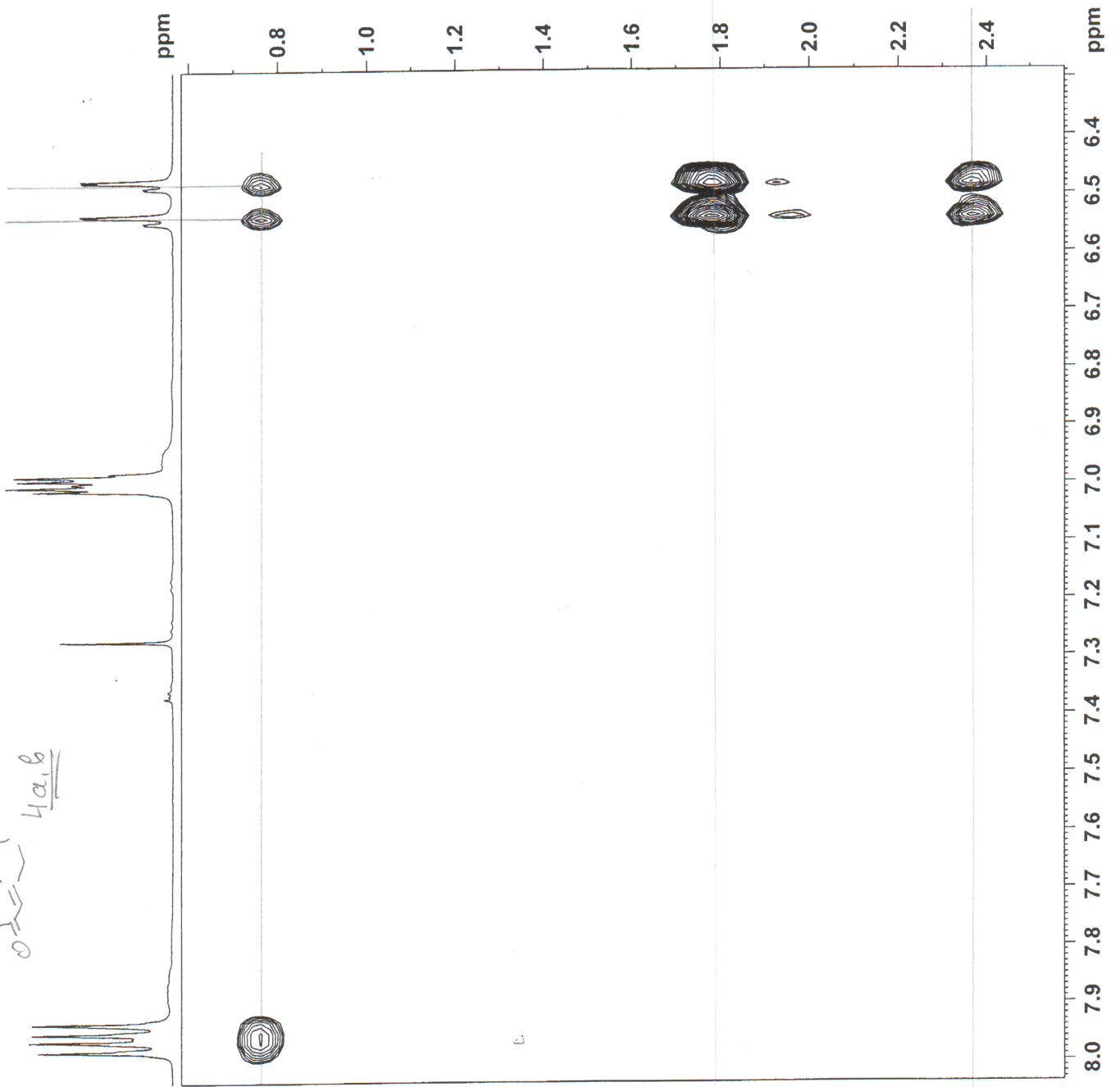


```

NAME H-139
EXPNO 6
PROCNO 1
Date_ 20110418
Time 12.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG noesyph
TD 1624
SOLVENT CDCl3
NS 8
DS 16
SWH 4882.813 Hz
FIDRES 4.768372 Hz
AQ 0.1049076 sec
RG 40.3
DE 102.400 usec
TE 298.0 K
D0 0.00009050 sec
D1 2.00000000 sec
D8 1.00000000 sec
IN0 0.00020480 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.35 usec
PL1 0.00 dB
PL1W 27.37956238 W
SF01 500.2620691 MHz
ND0 1
TD 128
SF01 500.2621 MHz
FIDRES 38.146938 Hz
SW 9.761 ppm
FMODE States-TPPI
SI 512
SF 500.2600044 MHz
WDW QSI
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 512
MC2 States-TPPI
SF 500.2600043 MHz
WDW QSI
SSB 2
LB 0.00 Hz
GB 0

```

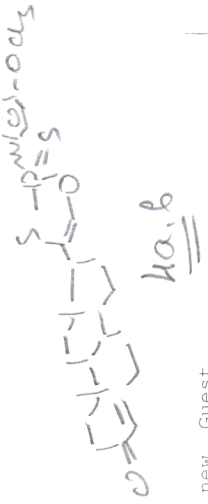


$H_{a,b}$ -18

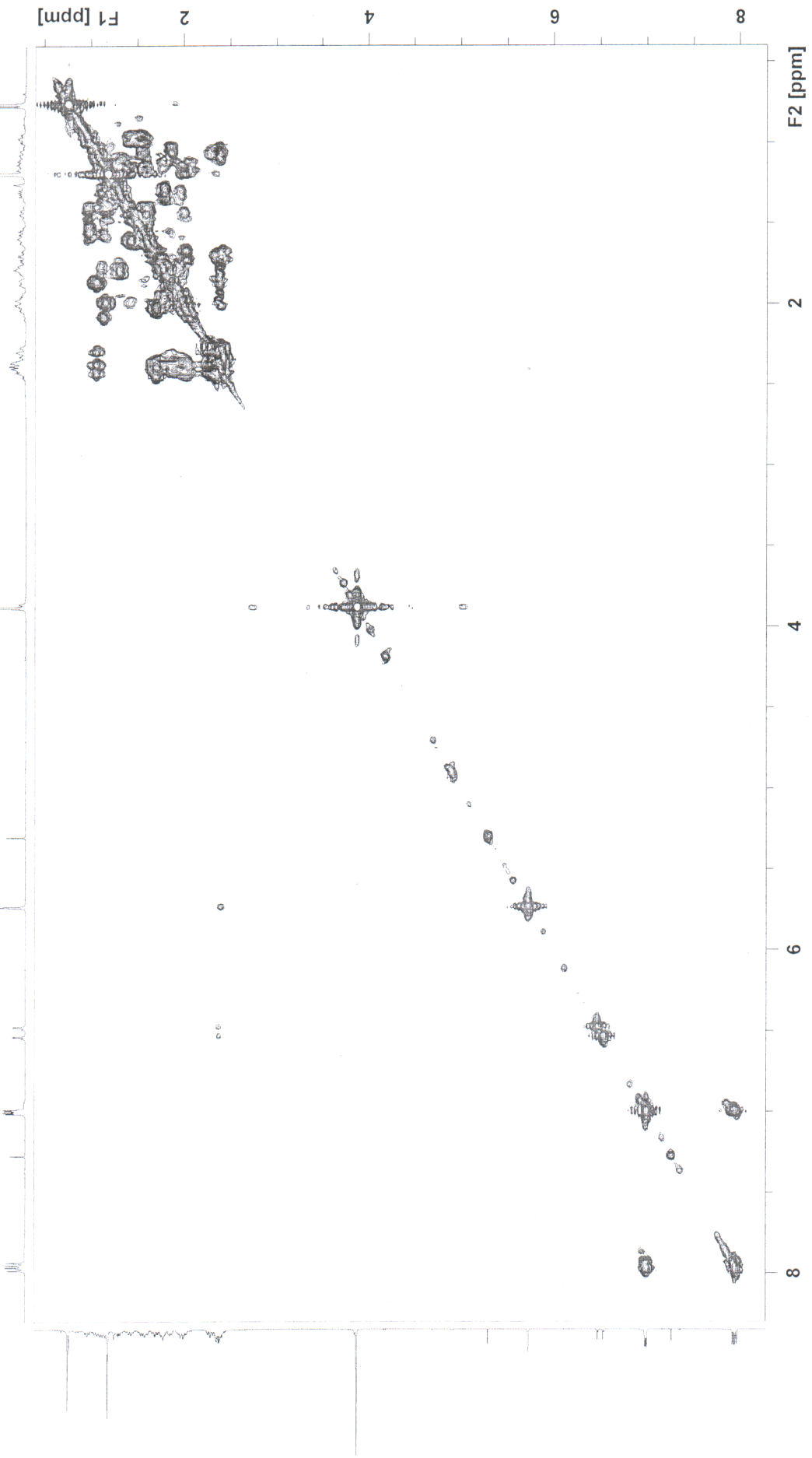
$H_{a,b}$ -16

$H_{a,b}$ -17

COSY

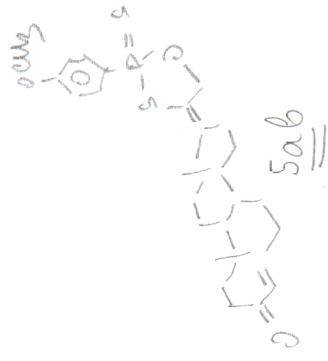


H-139 3 1 C:\Bruker\TOPSPIN\BBO_new Guest

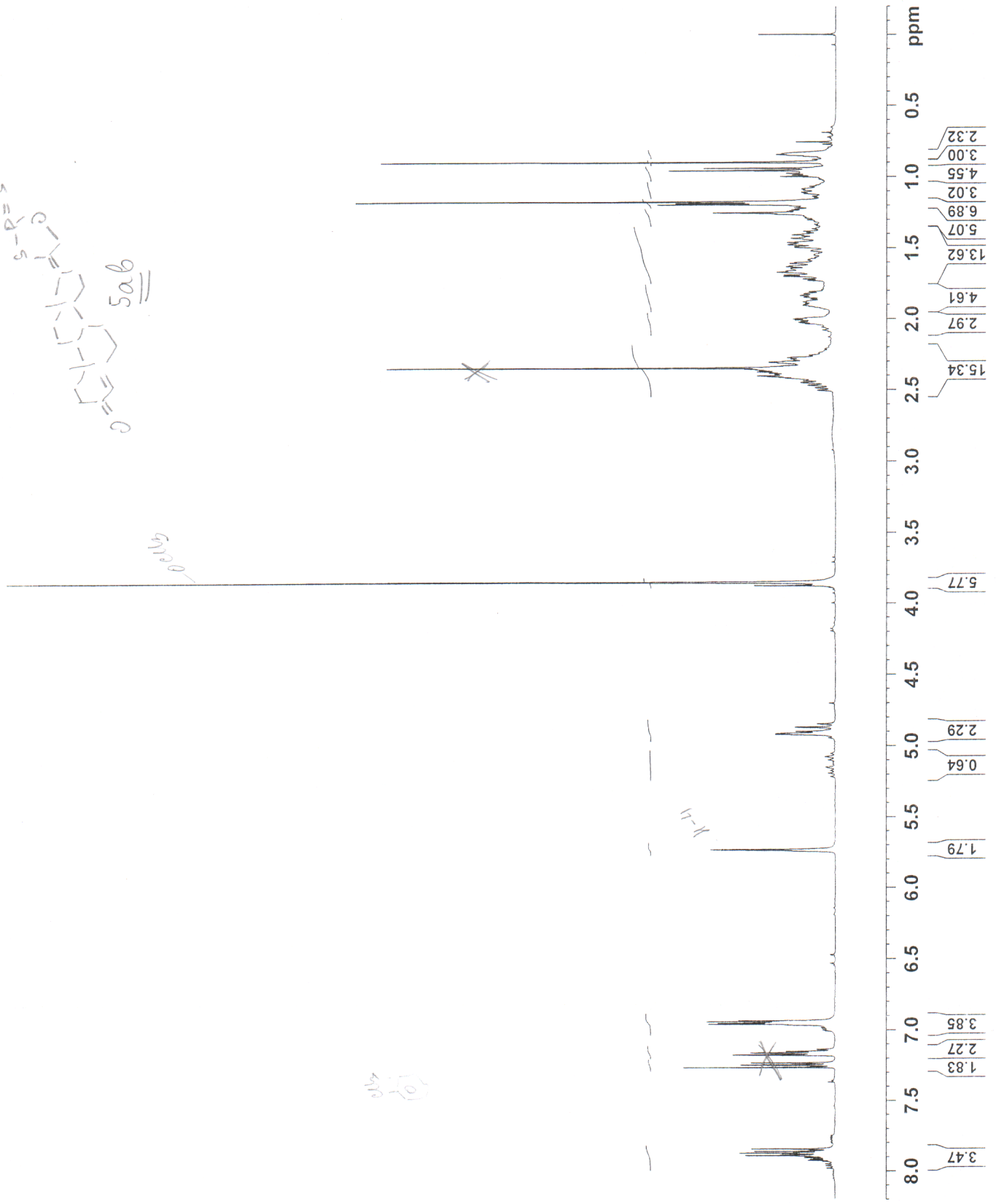
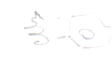


NAME H-137
 EXPNO 1
 PROCNO 1
 Date 20101110
 Time 10.05
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 0
 SWH 4743.833 Hz
 FIDRES 0.144770 Hz
 AQ 3.4537971 sec
 RG 80.6
 DW 105.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.35 usec
 PL1 0.00 dB
 PL1W 27.37956238 W
 SFO1 500.2620833 MHz
 SI 32768
 SF 500.2600112 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00



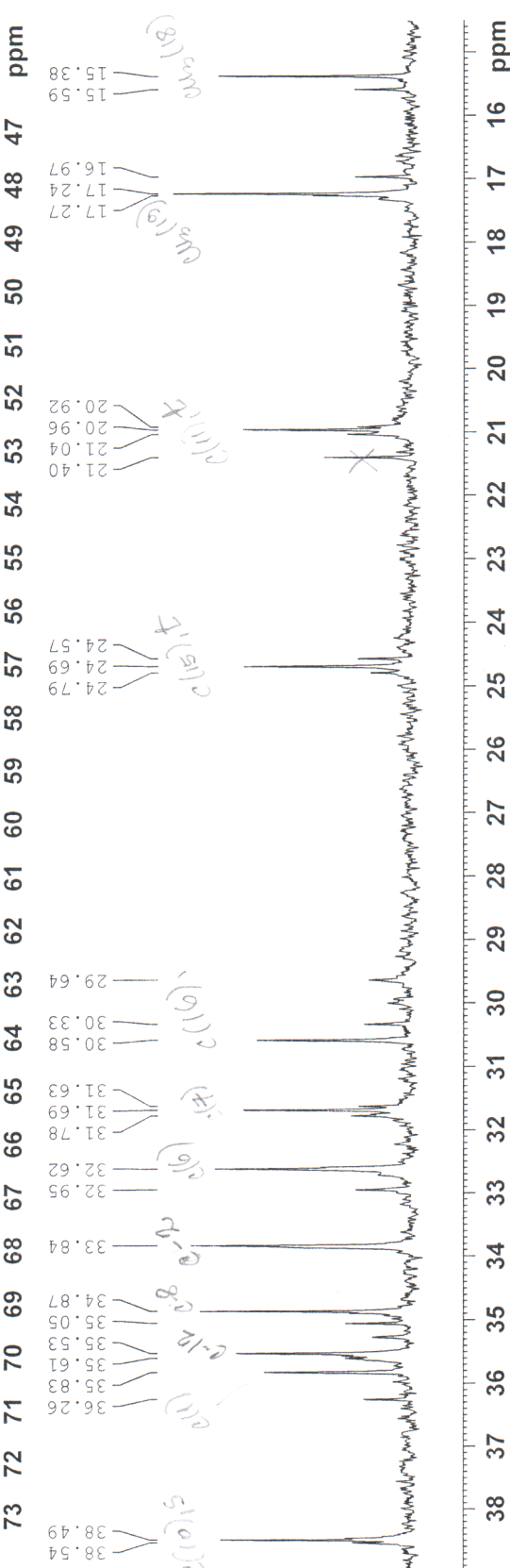
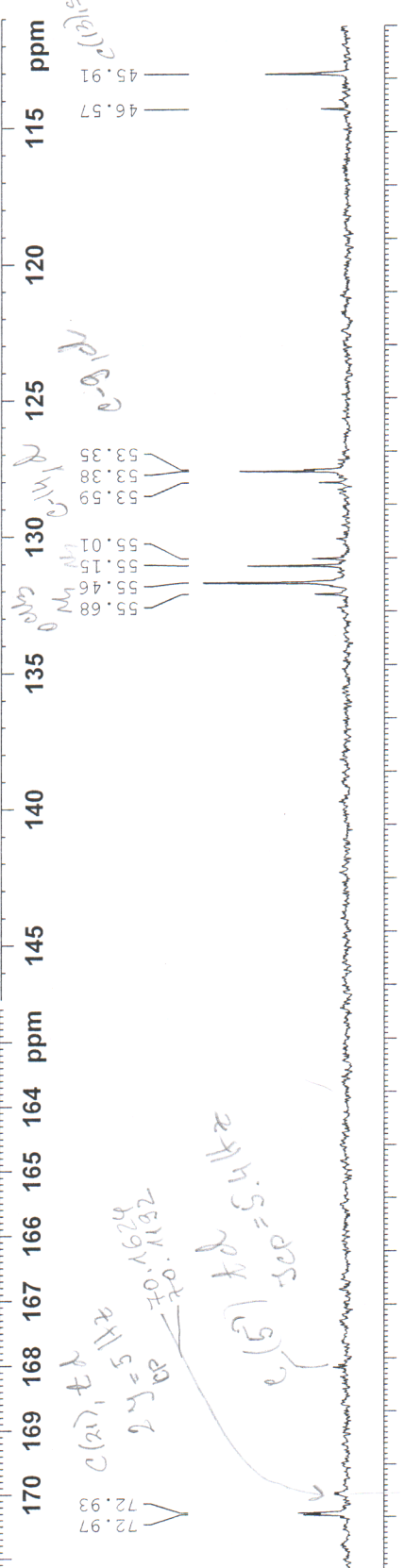
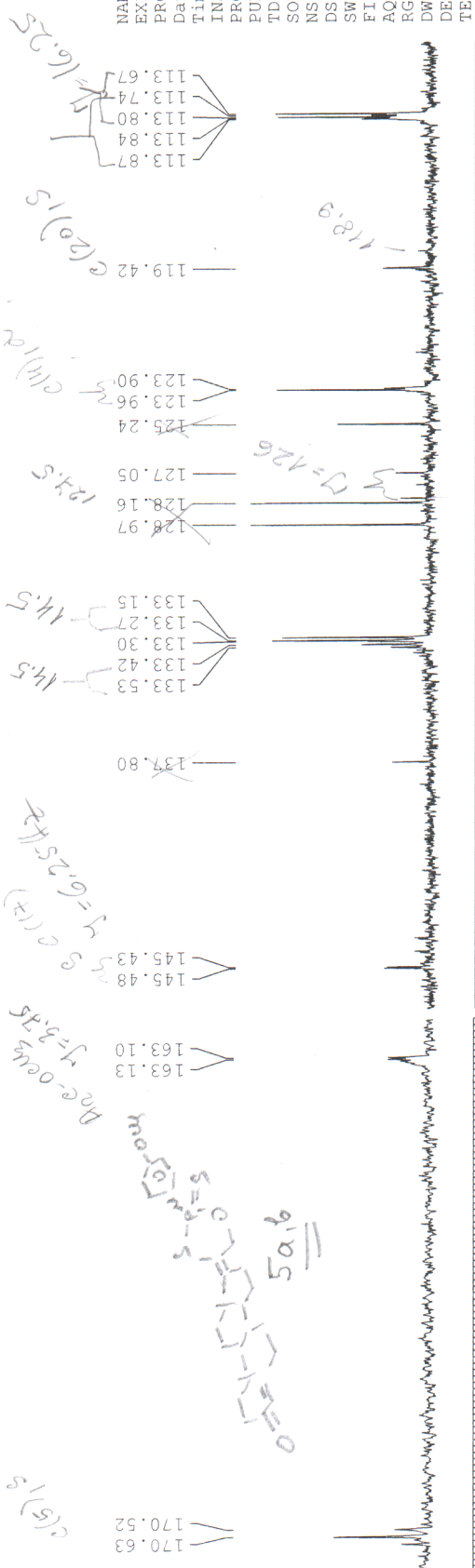
OCMe



NAME H-137
 EXPNO 2
 PROCNO 1
 Date_ 20101110
 Time 10.36
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 CDC13 402
 NS 4
 DS 29761.904 Hz
 SWH 0.908261 Hz
 FIDRES 0.5505524 sec
 AQ 1.620
 RG 1620
 DW 16.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

CHANNEL f1 =====
 NUC1 13C
 P1 11.50 usec
 PL1 3.00 dB
 PLLW 32.22848892 W
 SFO1 125.8043140 MHz

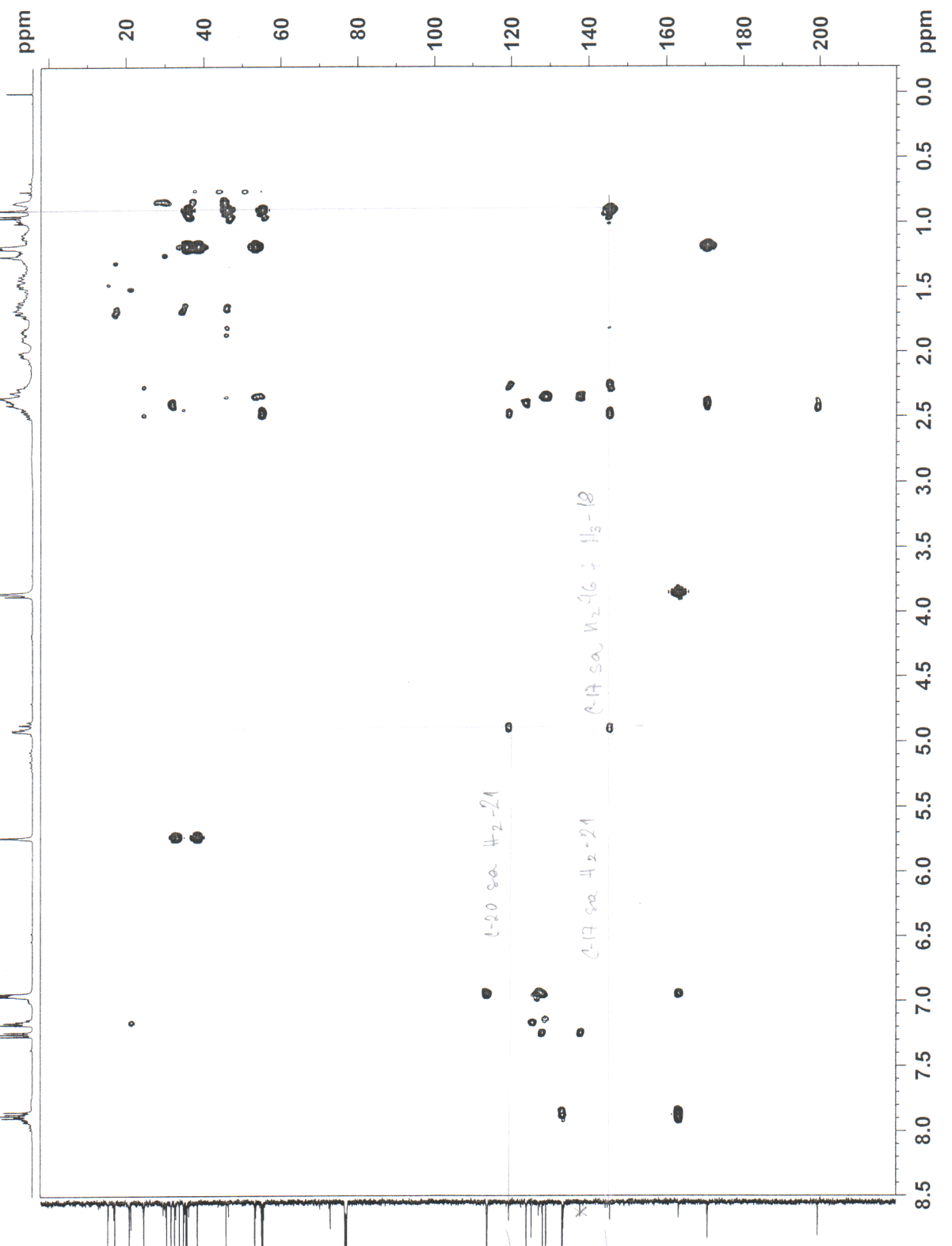
CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.20 dB
 PLL2 18.40 dB
 PLL3 18.40 dB
 PL2W 20.76952171 W
 PLL2W 0.39575511 W
 PLL3W 0.39575511 W
 SFO2 500.2620836 MHz
 SI 32768
 SF 125.7904860 MHz
 WDW EM
 SSB 0
 LB 1.50 Hz
 GB 0
 PC 1.40



HMBC



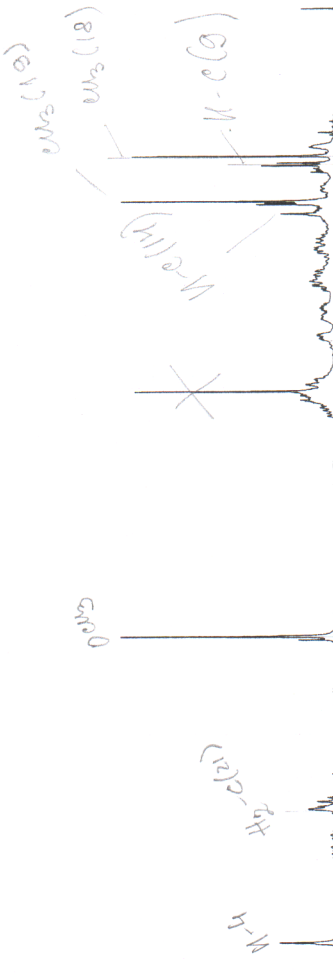
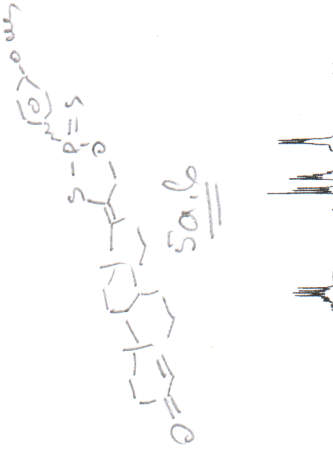
NAME H-137
 EXENO 5
 PROCNO 1
 Date 20101110
 Time 11.32
 INSTRUM Spect
 PULPROG 5 mm BBO
 F1F2 1024
 F2F3 1024
 TD 1024
 SOLVENT CDCl3
 NS 8
 DS 16
 SWH 4743.833 Hz
 FIDRES 4.632649 Hz
 AQ 0.1079796 sec
 RG 2050
 DW 105.400 usec
 DE 6.50 usec
 TE 298.0 K
 CNST2 145.0000000
 CNST13 8.0000000
 D0 0.0000300 sec
 D1 1.5000098 sec
 D2 0.0250026 sec
 D3 0.0250026 sec
 D4 0.0002000 sec
 D16 0.0002000 sec
 INO 0.00001805 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 9.35 usec
 PL1 18.70 usec
 PL1W 0.00 dB
 SFO1 27.37956238 W
 SFO1 500.2620836 MHz
 ===== CHANNEL f2 =====
 NUC2 13C
 P2 11.50 usec
 PL2 3.00 dB
 PL2W 32.22846892 W
 SFO2 125.8603060 MHz
 ===== GRADIENT CHANNEL =====
 GENM1 SINE,100
 GENM2 SINE,100
 GENM3 SINE,100
 GF21 50.00 %
 GF22 30.00 %
 GF23 40.10 %
 F16 1000.00 usec
 NDO 2
 TD 128
 SFO1 125.8031 MHz
 FIDRES 216.223999 Hz
 SW 220.000 PPM
 FMODE OF
 SI 1024
 SF 500.2600117 MHz
 SSB QSIGN
 LB 0.00 Hz
 GB 0
 PC 1.40
 SI 512
 MC2 OF
 SF 125.7904589 MHz
 SSB SINE
 LB 0
 GB 0.00 Hz



HMBC

(17)
 (20)

HsQC



HsQC

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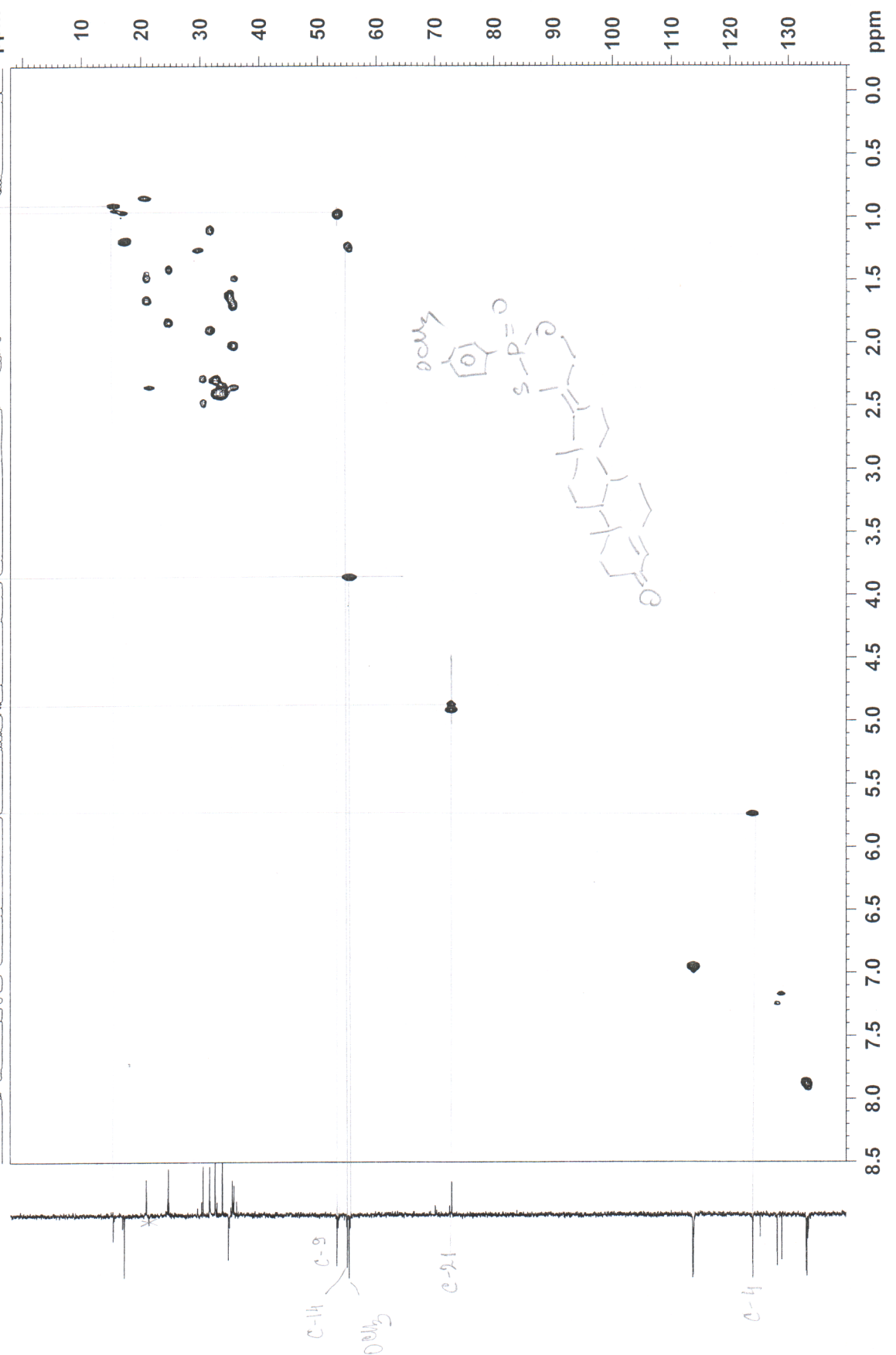
NAME H-137
EXENO 4
PROCNO 1
Date 20101110
Time 11:02
INSTRUM spect
PROBHD 5 mm BBO BB4-H
PULPROG hsqcetcp24
TD 64
SOLVENT CDCl3
NS 16
DS 16
SWH 4743.833 Hz
FIDRES 4.632649 Hz
AQ 0.11079796 sec
RG 2050
DW 105.400 usec
DE 6.50 usec
TE 298.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 2.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
TD0 0.00086297 sec
TD1 0.00002650 sec
ZGCPINS

===== CHANNEL f1 =====
NUC1 1H
P1 9.35 usec
P2 18.70 usec
P28 1000.00 usec
PL1 0.00 dB
PL1W 27.37956238 W
SFO1 500.2620836 MHz

===== CHANNEL f2 =====
CPDPRG2 gairp
NUC2 13C
P3 11.50 usec
P4 23.00 usec
P5 9.00 usec
FCPD2 0.00 dB
PL2 18.00 dB
PL2W 32.22848892 W
PL1W 1.01915431 W
SFO2 125.7992823 MHz

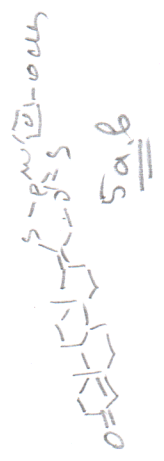
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GFZ1 80.00 Hz
GFZ2 20.10 Hz
GFZ3 11.00 Hz
GFZ4 -5.00 Hz
F16 1000.00 usec
F19 600.00 usec
ND0 2
TD 126
FIDRES 125.7992 MHz
SFO1 147.21006 Hz
SFO2 15.151006 ppm
ECHO-ANTECHO ppm
SI 1024
SF 500.2600095 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 256
MC2 echo-antecho
SF 125.7904530 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

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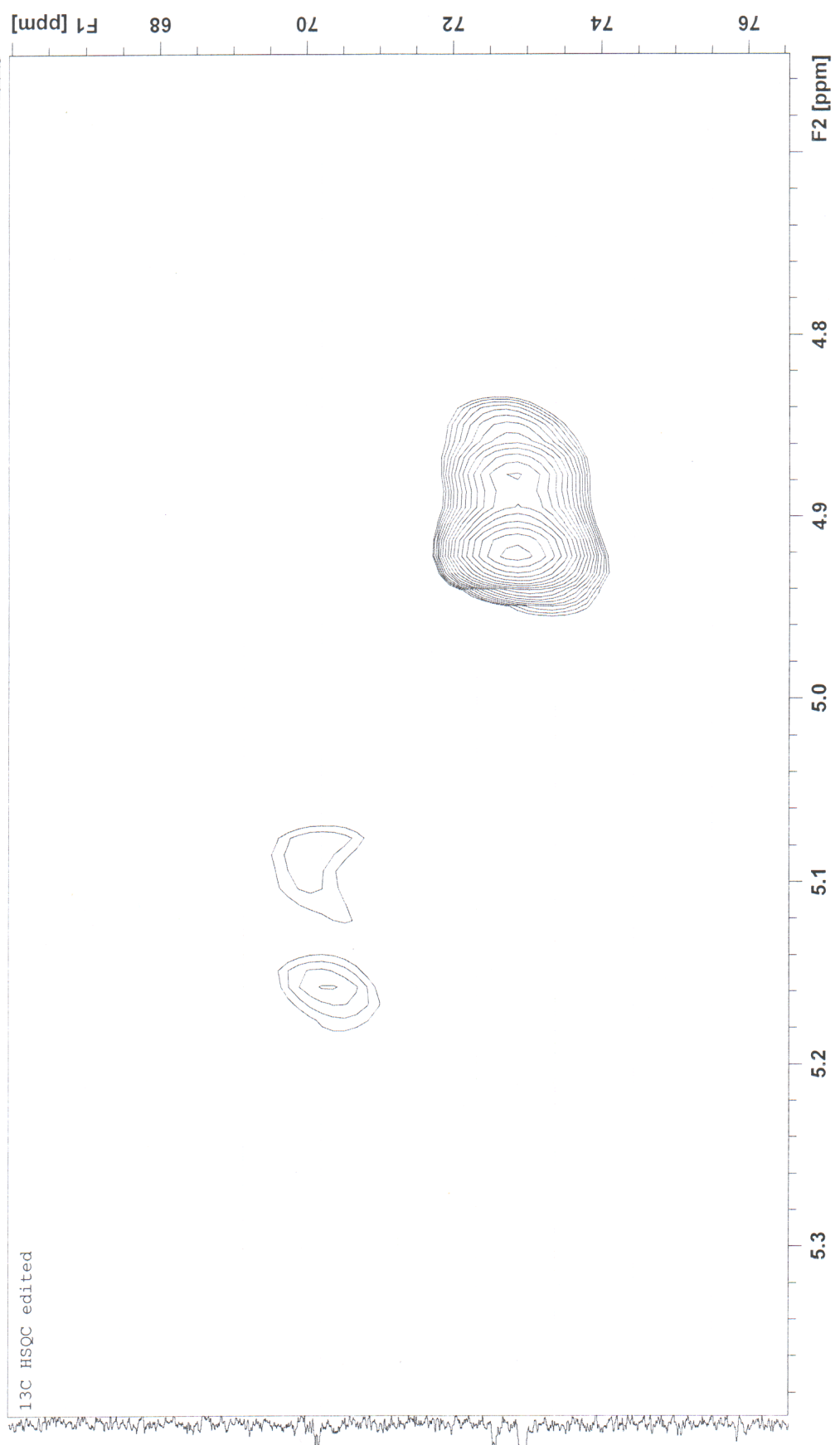


HSQC

HSQC

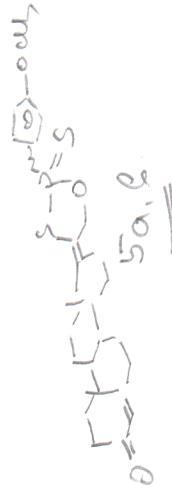


H-137



70.1
 $\gamma = 5.4 \text{ Hz}$
 70.1624
 70.1192

NOESY

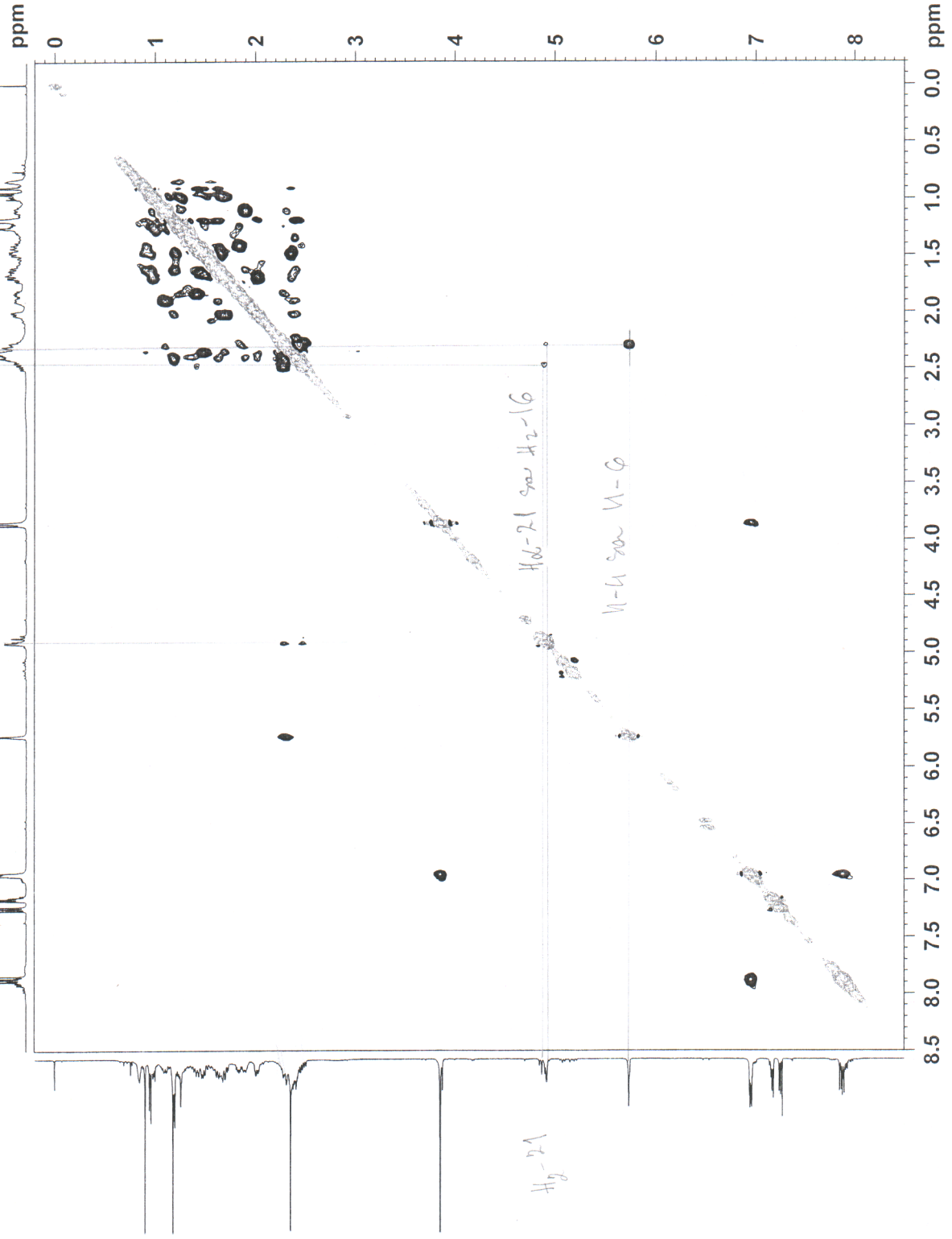


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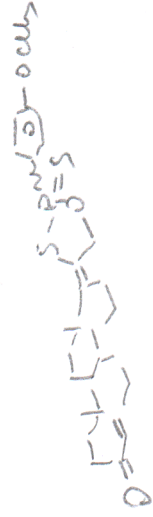
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NAME H-137
EXNO 6
PROCNO 1
Date_ 20101110
Time_ 12.14
INSTRUM spect
PROBHD 5 mm BBO BB-IH
PULPROG noesyph
TD 1024
SOLVENT CDCl3
NS 8
DS 16
SWH 4743.833 Hz
FIDRES 4.632649 Hz
AQ 0.1079796 sec
RG 90.5
DM 105.400 usec
DE 6.50 usec
TE 298.0 K
D0 0.00009350 sec
D1 2.00000000 sec
D8 1.00000000 sec
INO 0.00021080 sec
===== CHANNEL f1 =====
NUC1 1H
P1 9.35 usec
PL1 0.00 dB
PL1W 27.37956238 W
SF01 500.2620836 MHz
ND0 1
TD 128
SF01 500.2621 MHz
FIDRES 37.061214 Hz
SW 9.483 ppm
EnMODE States-TPPI
SI 512
SF 500.2600058 MHz
WDW QSI
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 512
MC2 States-TPPI
SF 500.2600065 MHz
WDW QSI
SSB 2
LB 0.00 Hz
GB 0

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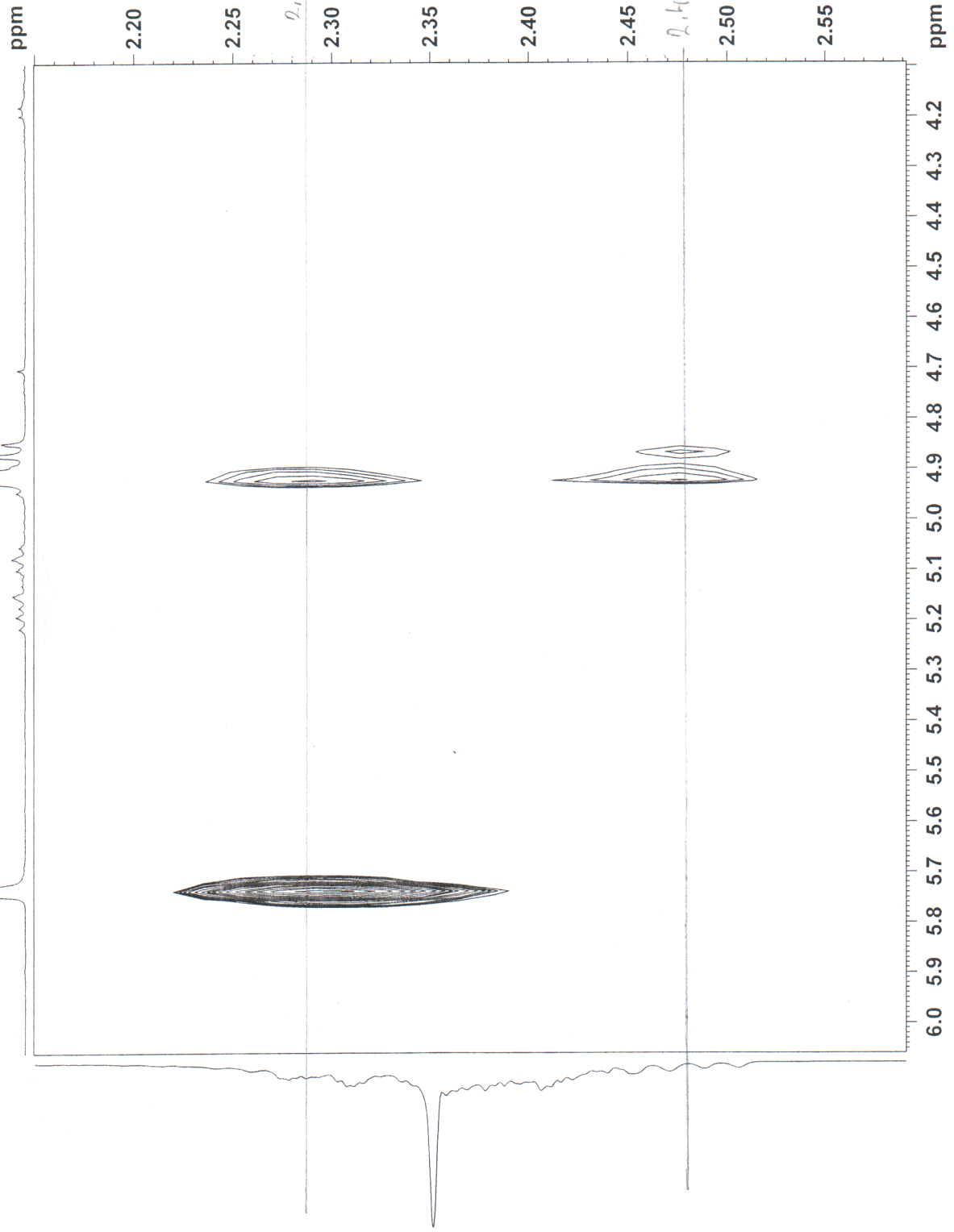
NOESY



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NAME H-137
EXPNO 6
PROCNO 1
Date_ 20101110
Time_ 12.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG noesyph
TD 1024
SOLVENT CDCl3
NS 8
DS 16
SWH 4743.833 Hz
FIDRES 4.632649 Hz
AQ 0.1079796 sec
RG 90.5
DW 105.400 usec
DE 6.50 usec
TE 298.0 K
D0 0.00009350 sec
D1 2.00000000 sec
D8 1.00000000 sec
INO 0.00021080 sec
===== CHANNEL f1 =====
NUC1 1H
P1 9.35 usec
PL1 0.00 dB
PL1W 27.37956238 W
SFO1 500.2620836 MHz
ND0 1
ID 128
SFO1 500.2621 MHz
FIDRES 37.061214 Hz
SW 9.483 ppm
FnMODE States-TpPI
SI 512
SF 500.2600058 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 512
MC2 States-TPEI
SF 500.2600065 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

```



NOESY