

Synthesis and RCM Reactions Using a Recyclable Grubbs-Hoveyda Metathesis Catalyst Activated by a Light-fluorous Tag

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Experimental

Except as otherwise indicated, all reactions were carried out under a positive pressure of nitrogen. All the laboratory chemicals were purchased and used without purification. Solvents were removed at a heating bath temperature of 40 °C and reduced pressure by rotary evaporation. Non-volatile compounds were dried *in vacuo* at 0.01 mbar. All reactions were magnetically stirred and monitored by thin layer chromatography (TLC) using silica gel plates. Purification by chromatography was performed on silica gel (45-75 Å). NMR-spectra were recorded at 270 MHz (¹H) and 68 MHz (¹³C) and 466 MHz (¹⁹F) respectively.

Chemical shifts δ are referred in terms of ppm and *J*-coupling constants are given in Hz.

Abbreviations for multiplicity are as follows: *s* (singlet), *d* (doublet), *t* (triplet), *q* (quadruplet), *m* (multiplet), *br* (broad signal). The signals of IR-spectra are given by wave numbers (cm⁻¹).

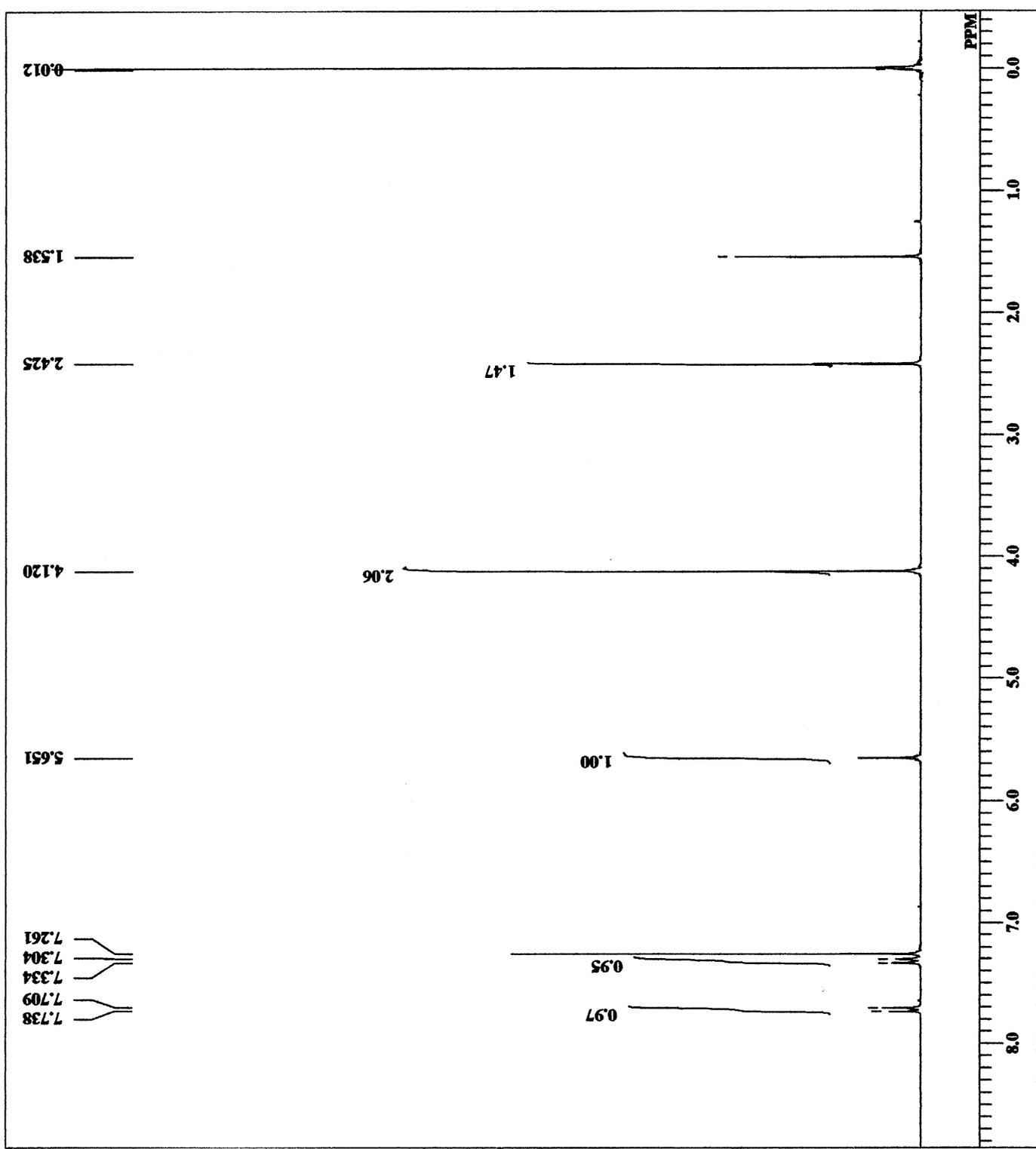
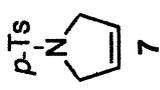
High resolution FAB and EI mass spectra were calibrated with Ultramark 1621® and PFK respectively prior to data acquisition.

f\w\afsf\fe\N\j\afj~f\@.ÅSE*

Fri Jun 04 16:36:29 2010

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 DATIM
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 EXMOD
 OBFRQ
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

1H
 NON
 270.05 MHz
 112.00 KHz
 5800.00 Hz
 16384
 5401.76 Hz
 16
 3.0331 sec
 3.9670 sec
 5.40 usec
 1H
 25.1 c
 CDCL3
 0.00 ppm
 0.12 Hz
 25

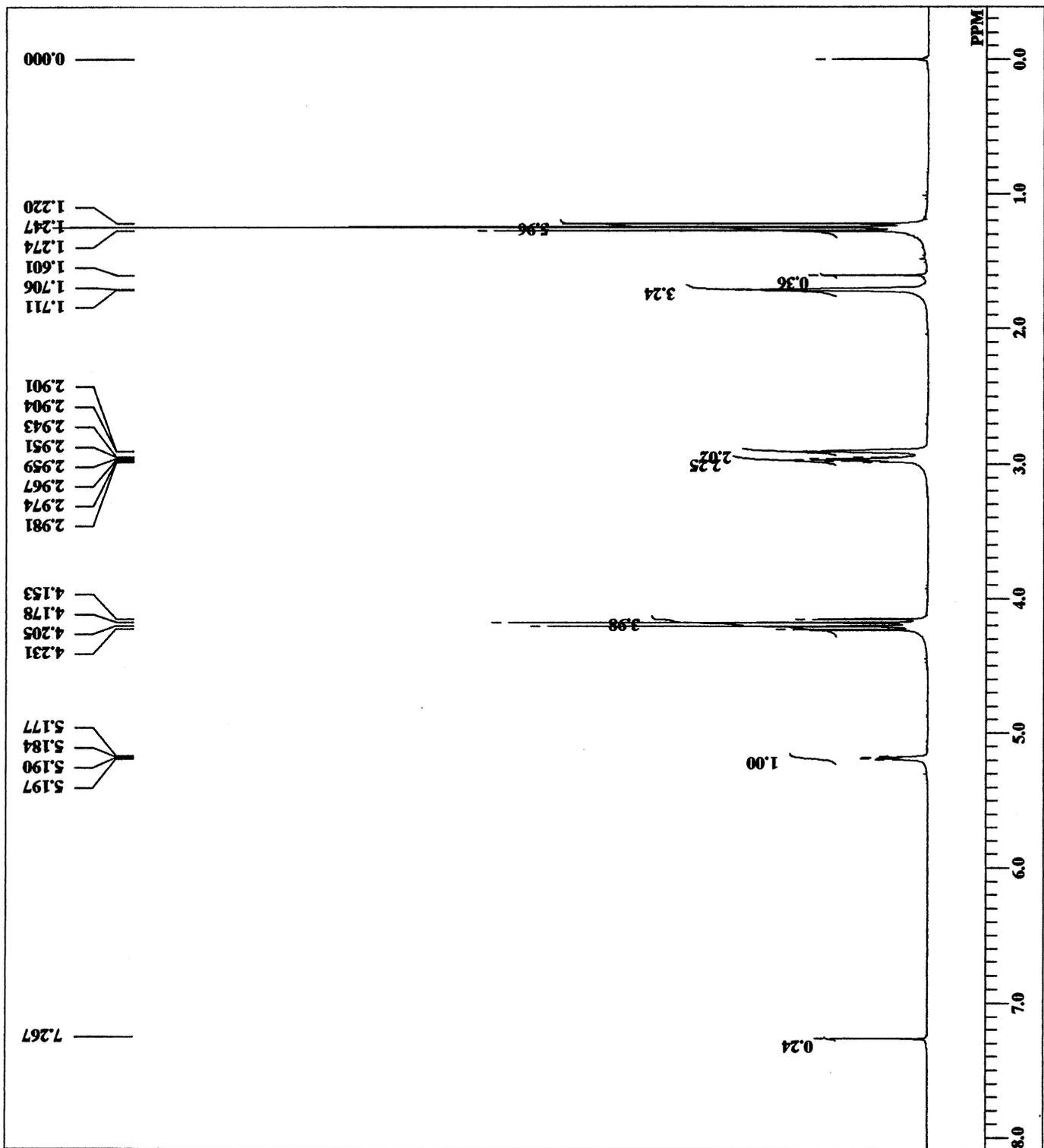
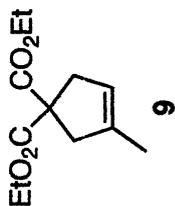


20100112 RCM 9.als

Tue Jan 12 18:12:40 2010

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SLVNT
EXREF
BF
RGAIN

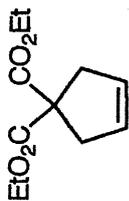
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112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
24.1 c
CDCl3
0.00 ppm
0.12 Hz
18



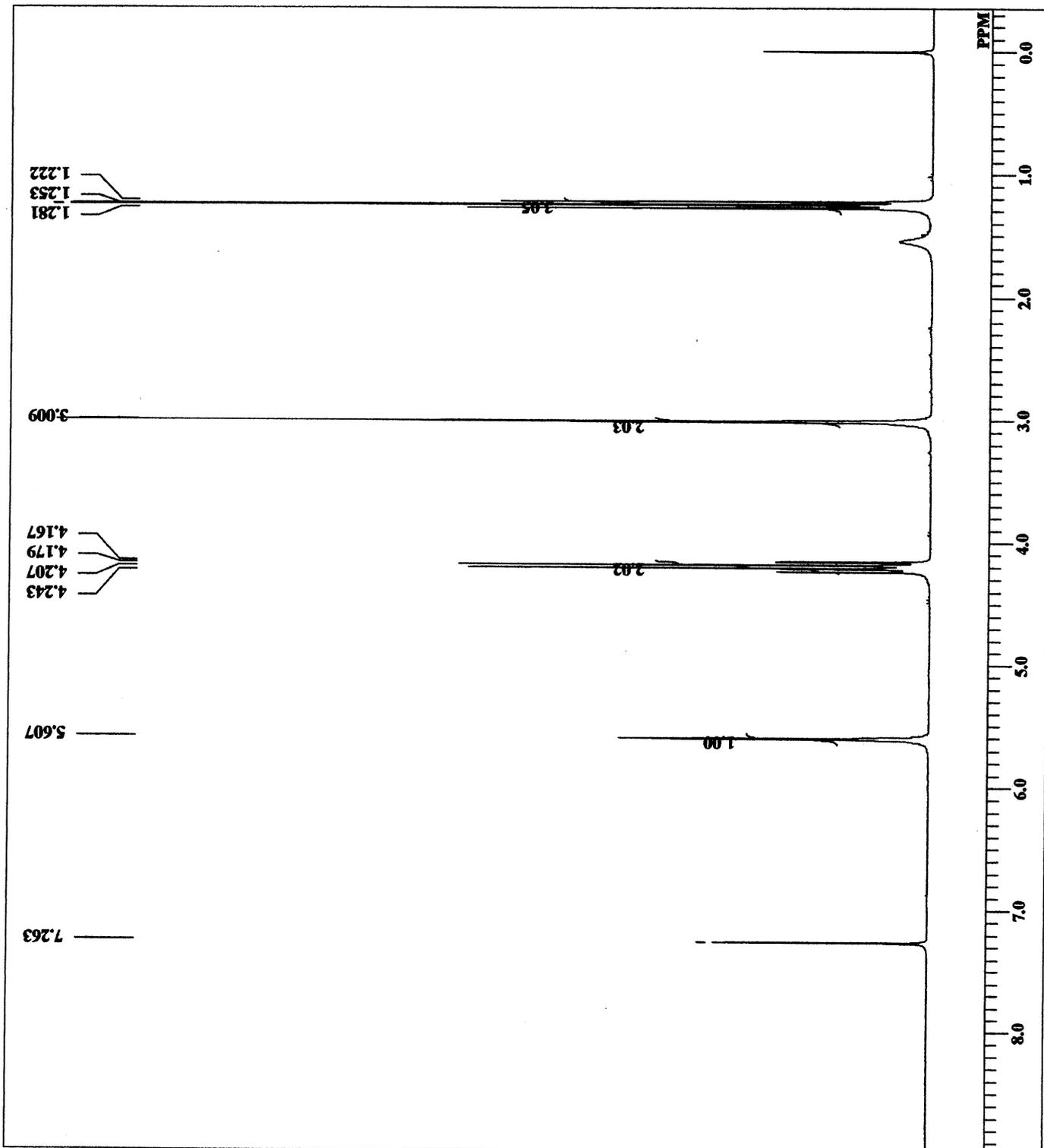
20100513 FSPE 1 diethyl.als

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FREQU 5943.54 Hz
SCANS 64
ACQTM 2.7566 sec
PD 3.9670 sec
PW1 5.40 usec
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SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22



11



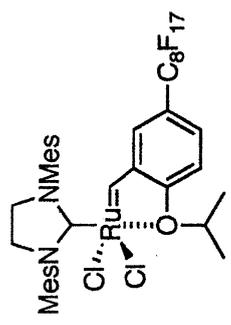

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= 19-SEP-2009 18:41:21

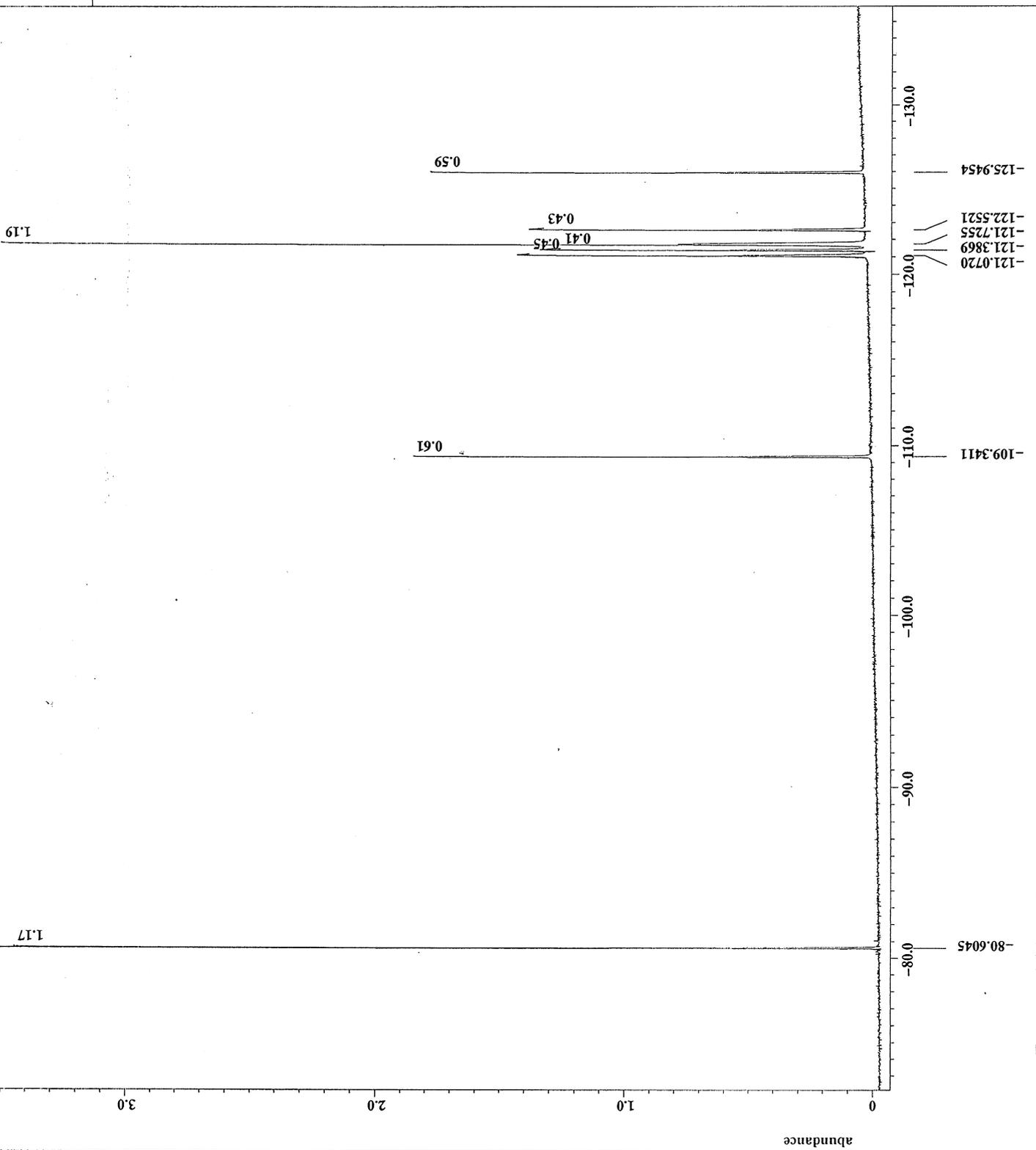
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= 26214
= 19F
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X_resolution = 7.33595628[Hz]
X_sweep = 240.38461538[kHz]
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Irr_freq = 465.88941346[MHz]
Irr_offset = 5[ppm]
Tri_domain = 19F
Tri_freq = 465.88941346[MHz]
Tri_offset = 5[ppm]
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Mod_return = 1
Scans = 8
Total_scans = 8

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X_acq_time = 0.13631488[s]
X_angle = 45[deg]
X_atn = 7.3[db]
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Dante_presat = FALSE
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2a



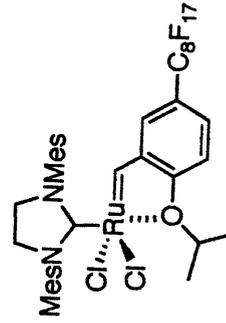
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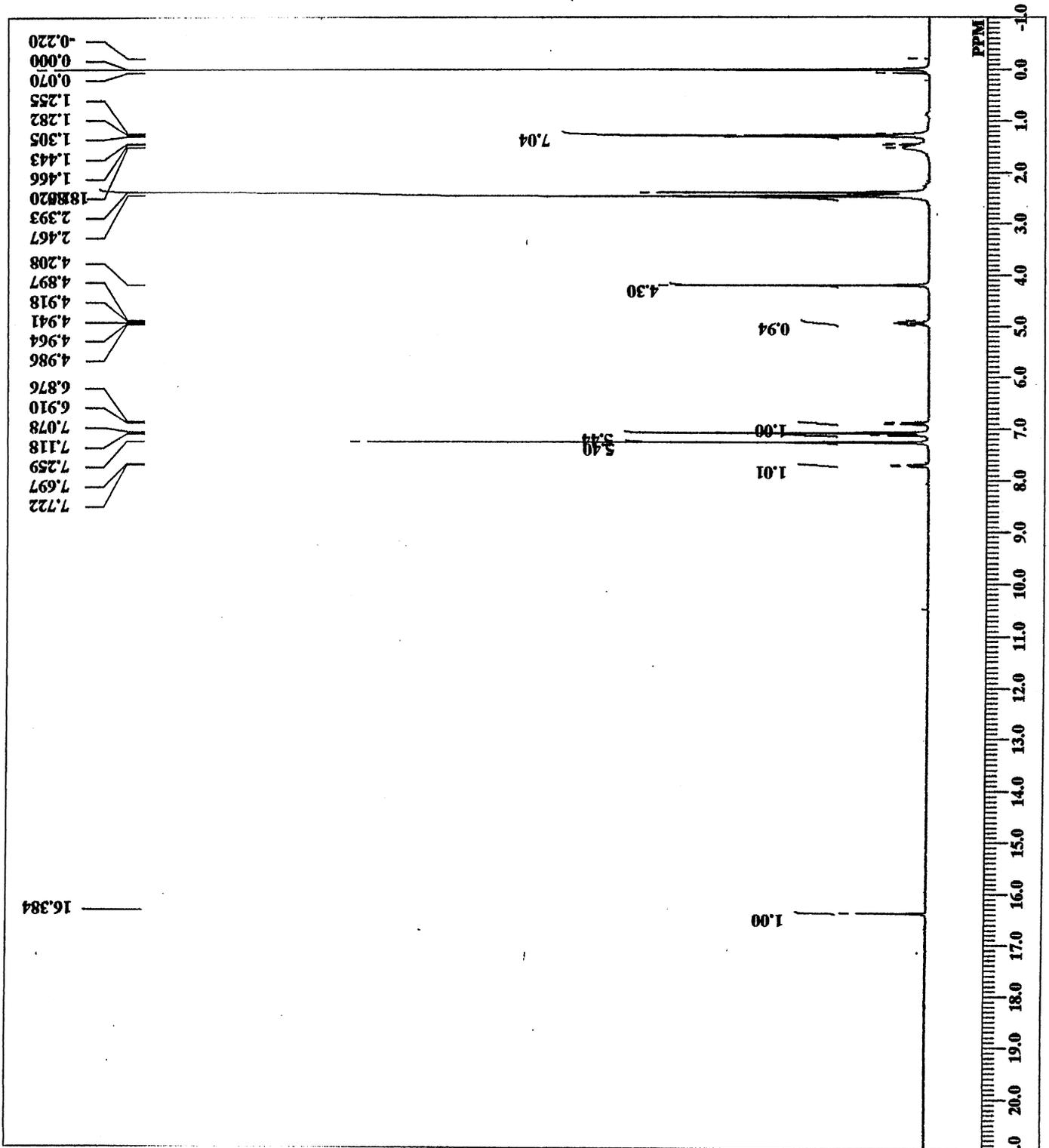
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SCANS 64
ACQTIM 2.7566 sec
PD 3.9670 sec
PW1 5.40 usec
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CTEMP 25.3 c
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EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25

1 day



2a

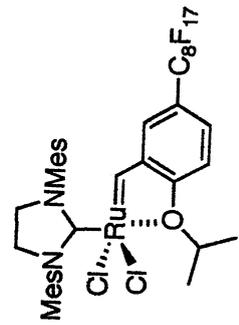


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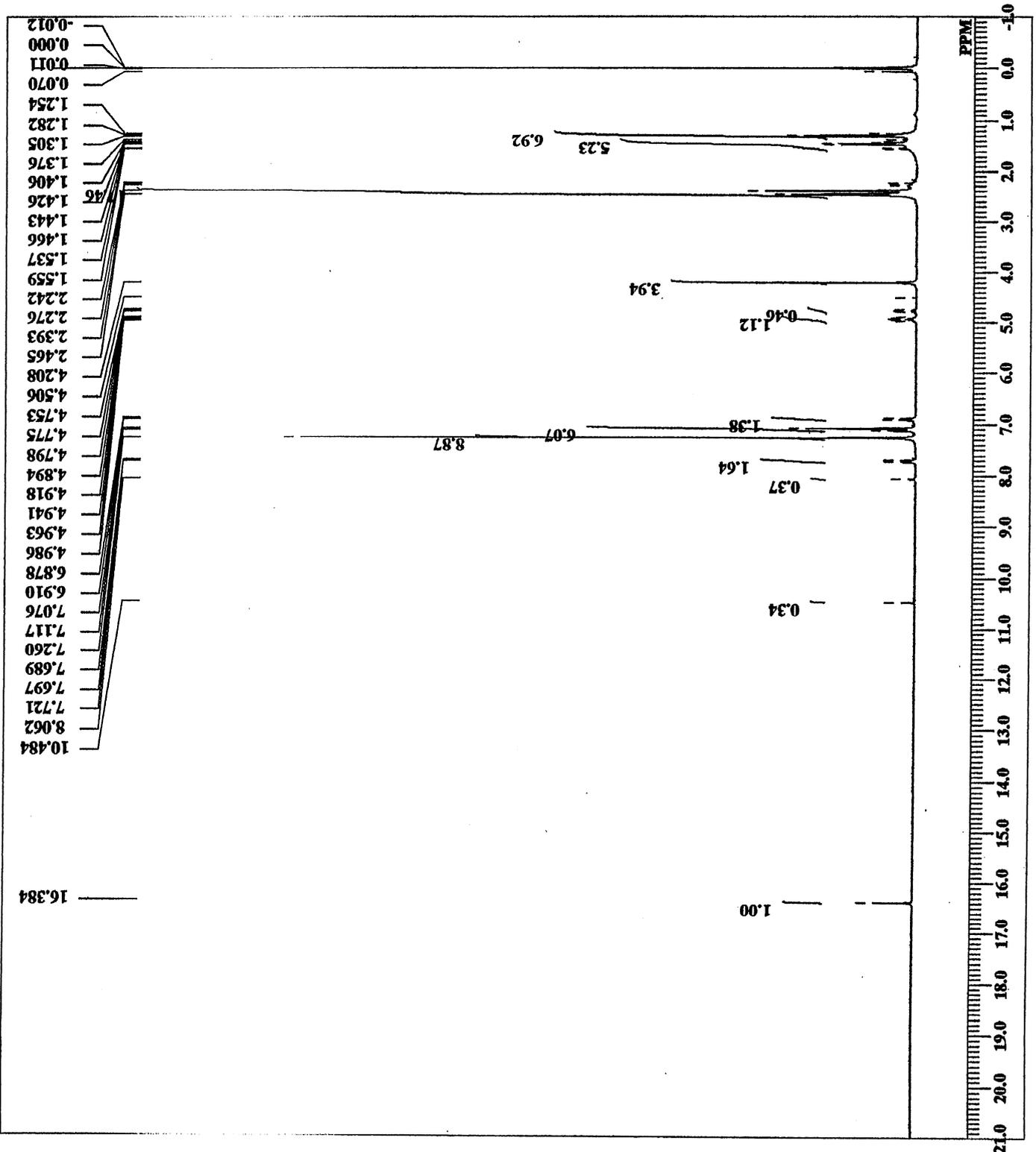
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ACQTIM 2.7566 sec
PD 3.9670 sec
PW1 5.40 usec
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CTEMP 25.4 c
SLVNT CDCL3
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BF 0.12 Hz
RGAIN 25

1 week



2a

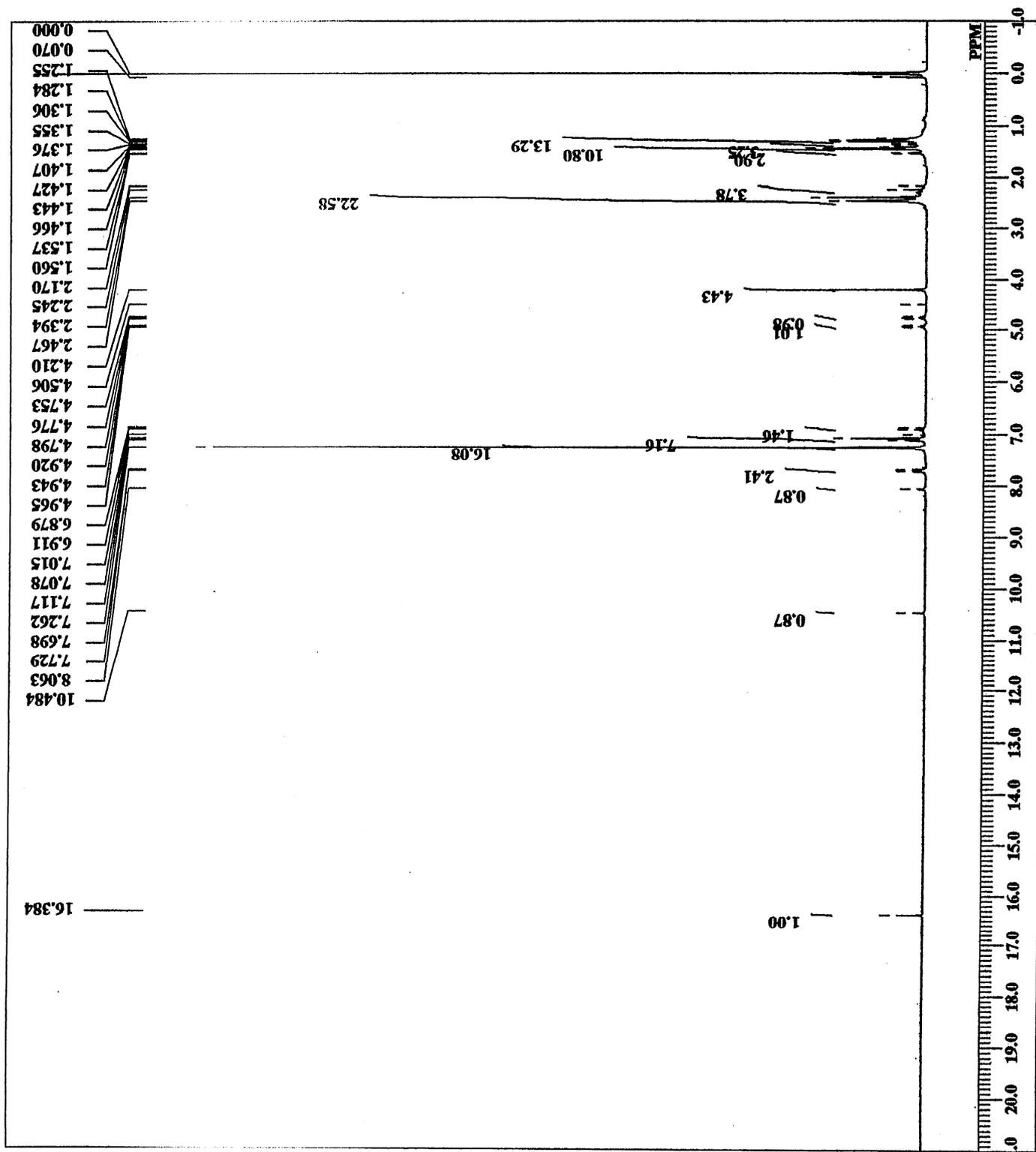


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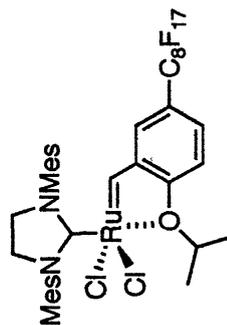
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POINT
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SCANS
ACQIM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

1H
NON
270.05 MHz
119.00 KHz
3.00 Hz
16384
5943.54 Hz
64
2.7566 sec
3.9670 sec
5.40 usec
1H
24.3 c
CDCL3
0.00 ppm
0.12 Hz
25



1 month



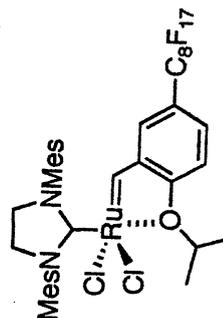
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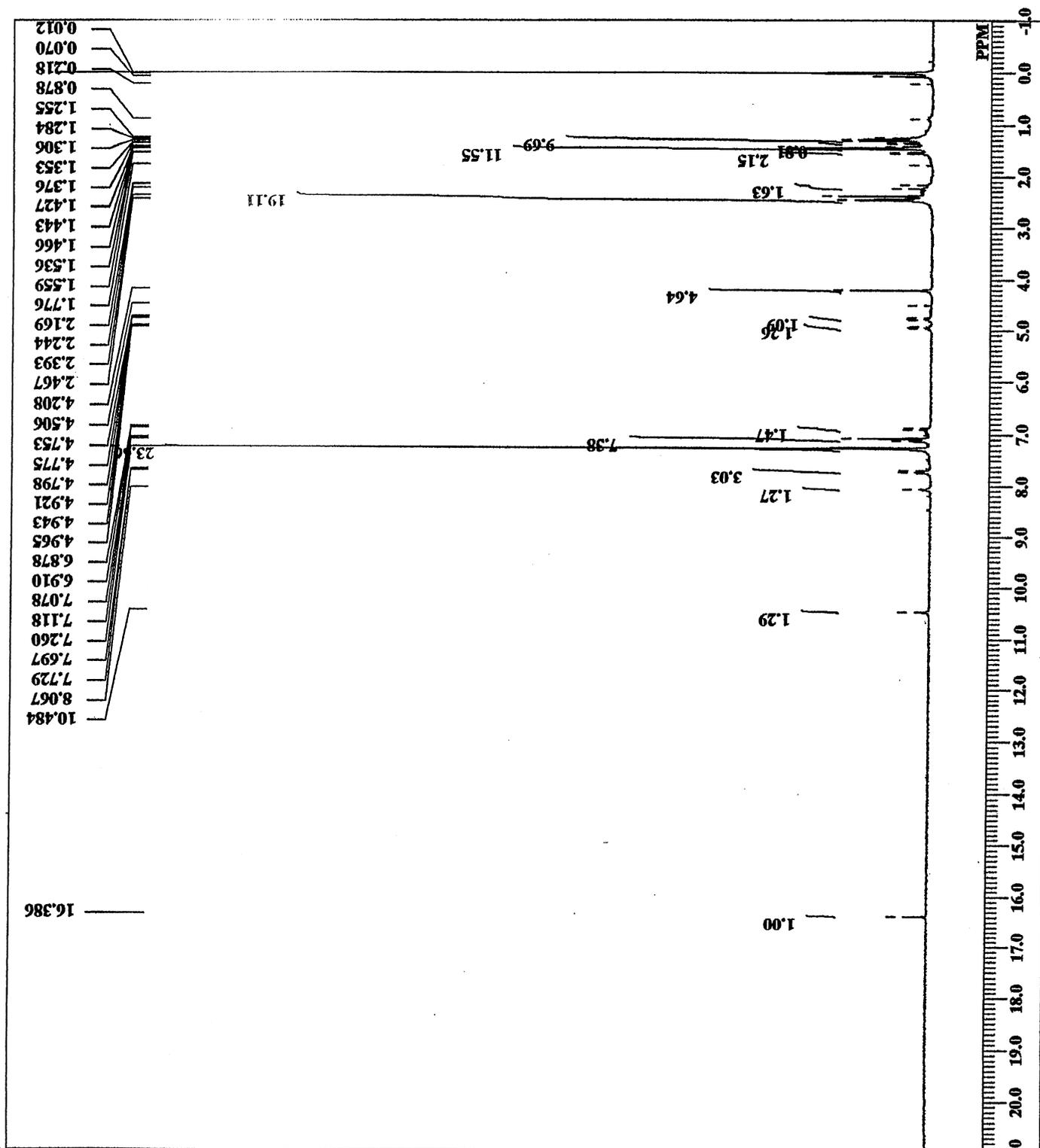
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OBFIN
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FREQU
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PD
PW1
IRNUC
CTEMP
SLYNT
EXREF
BF
RGAIN

270.05 MHz
119.00 KHz
3.00 Hz
16384
5943.54 Hz
64
2.7566 sec
3.9670 sec
5.40 usec
1H
25.8 c
CDCL3
0.00 ppm
0.12 Hz
25

2 month



2a

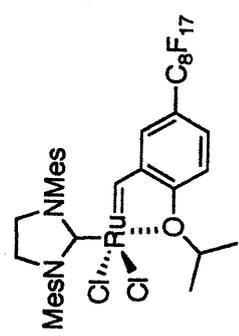


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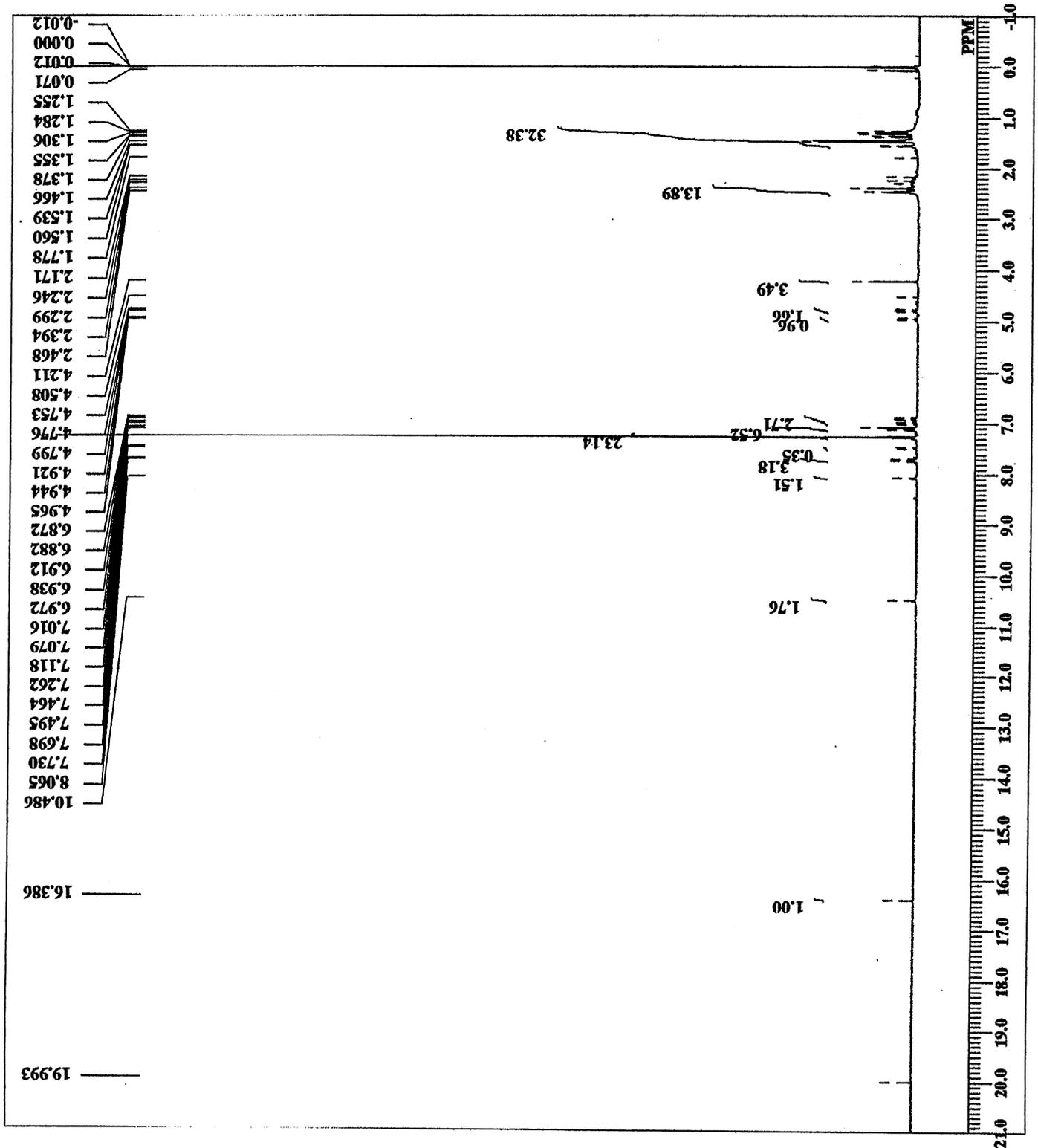
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 EXMOD
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 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLYNT
 EXREF
 BF
 RGAIN

1H
 NON
 270.05 MHz
 119.00 KHz
 3.00 Hz
 16384
 5943.54 Hz
 64
 2.7566 sec
 3.9670 sec
 5.40 usec
 1H
 24.4 c
 CDCL3
 0.00 ppm
 0.12 Hz
 26

3 month



2a



20100528 f-HG 2nd C8direct 7.als

Fri May 28 13:45:35 2010

1H
NON

270.05 MHz

119.00 KHz

3.00 Hz

16384

5943.54 Hz

64

2.7566 sec

3.9670 sec

5.40 usec

1H

24.5 c

CDCL3

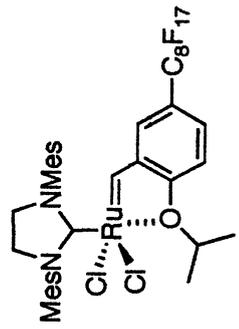
0.00 ppm

0.12 Hz

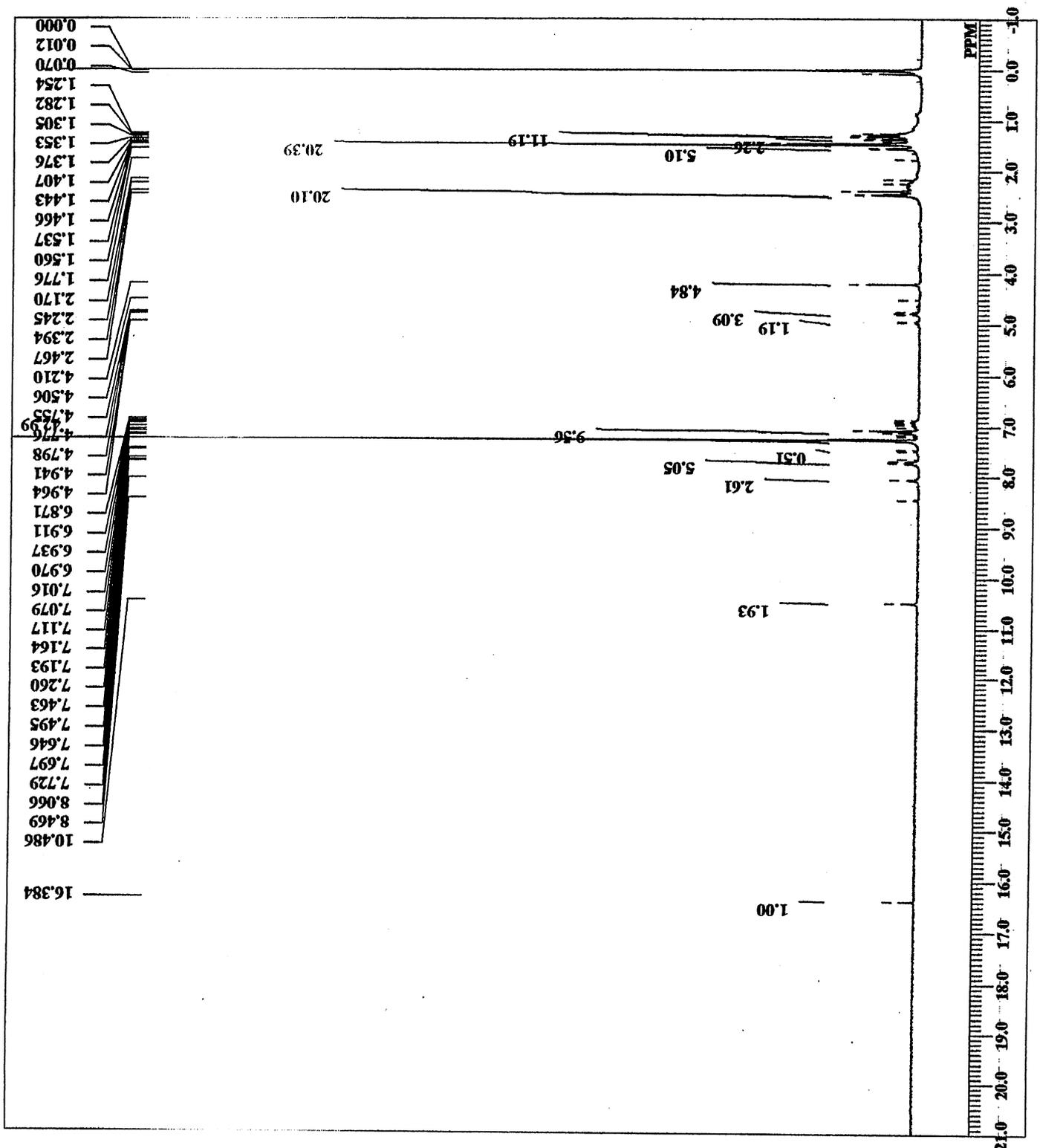
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DFILE
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 EXMOD
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 OBSET
 OBFIN
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 FREQU
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 ACQIM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

4 month



2a

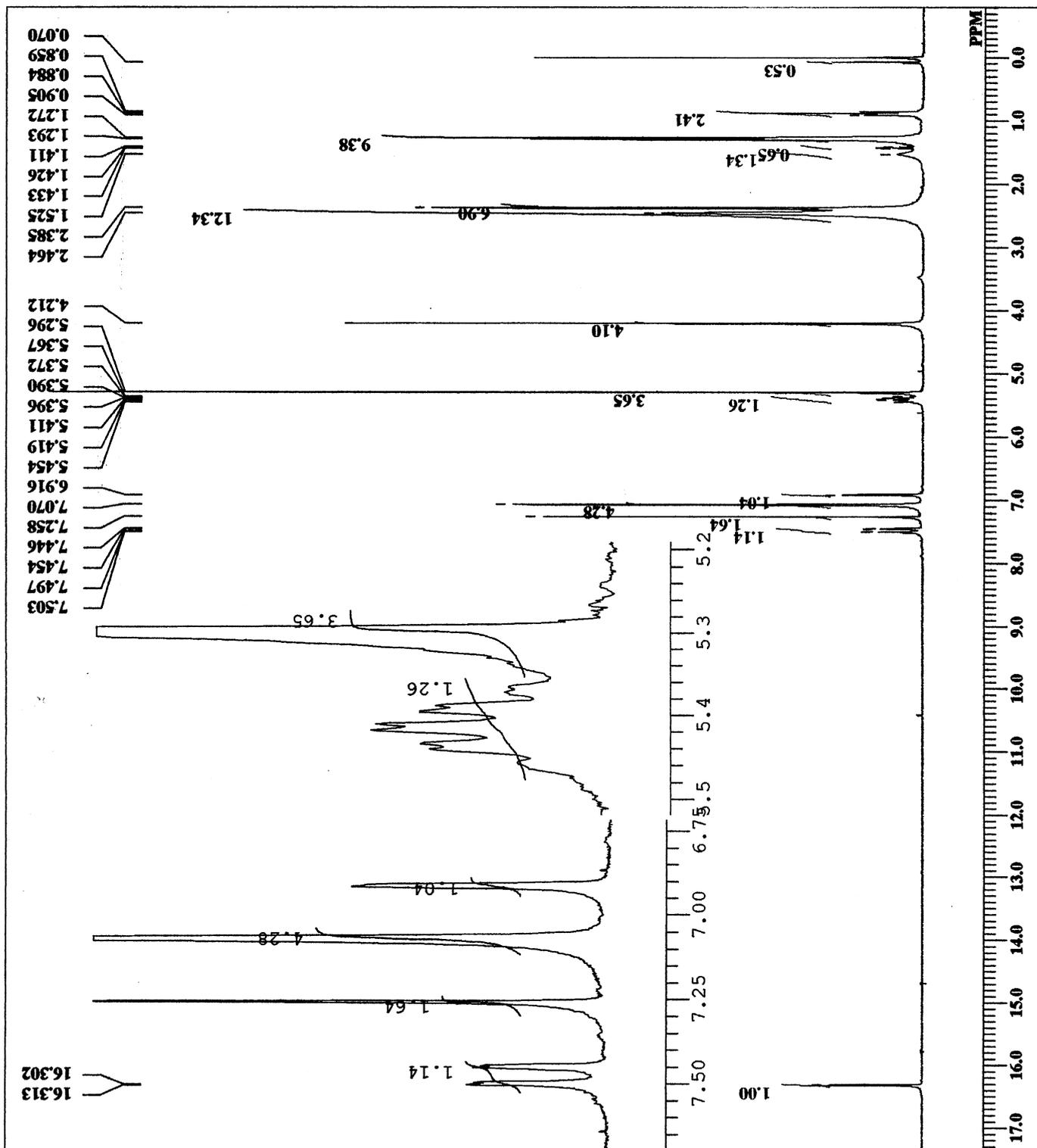
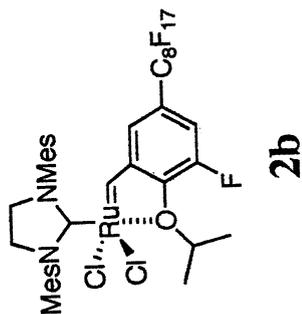


2009.12.16 no.22 fšfKf“fh00šš””

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OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

1H
NON
270.05 MHz
119.00 KHz
3.00 Hz
16384
5943.54 Hz
32
2.7566 sec
3.9670 sec
5.40 usec
1H
24.4 c
CDCL3
0.00 ppm
0.12 Hz
22



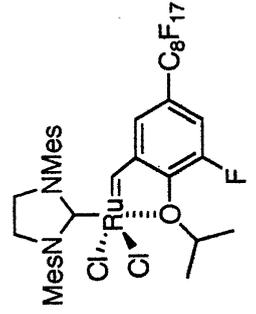
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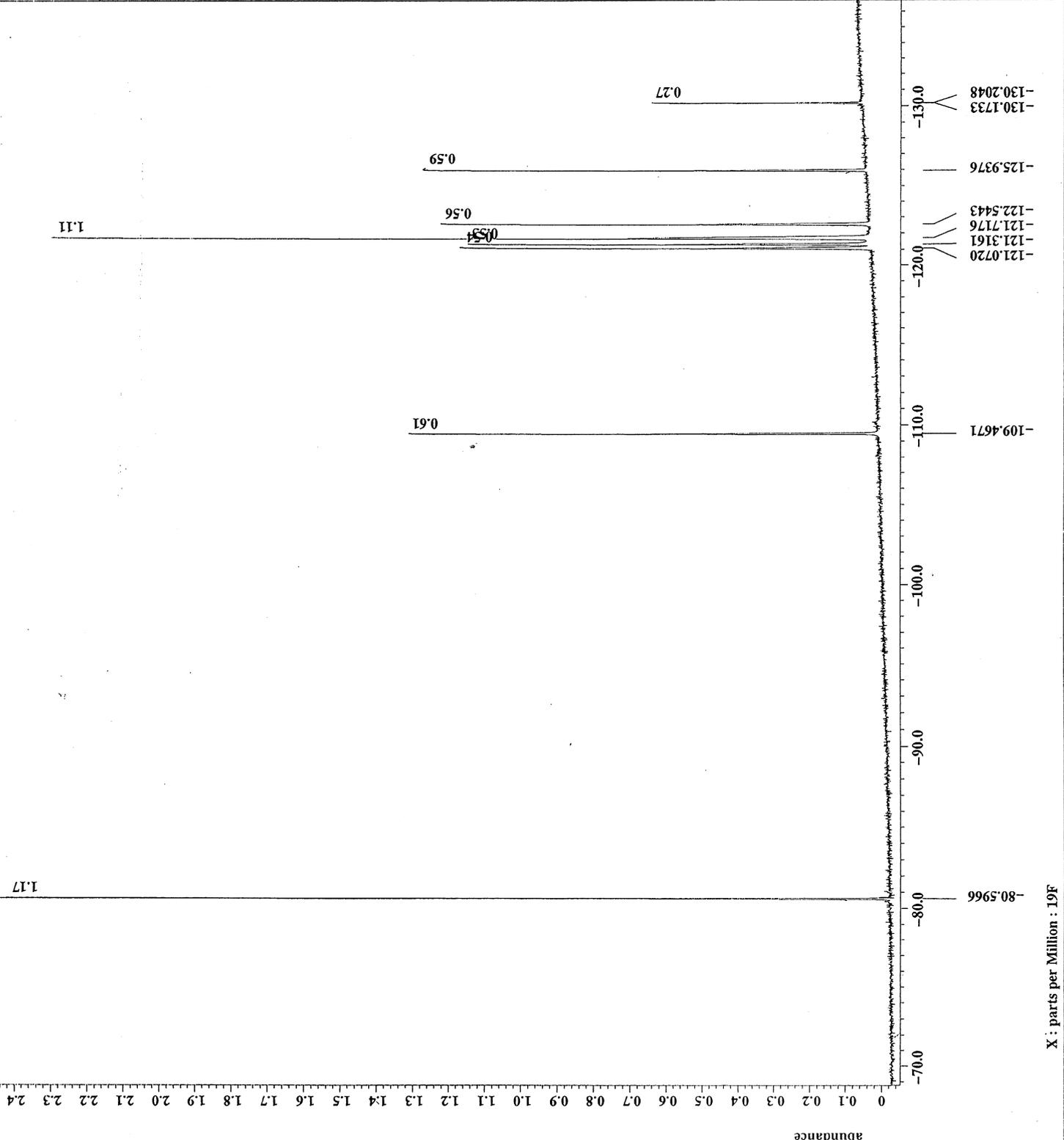
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Spectrometer = DELTA2_NMR

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X_domain = 19F
X_freq = 465.88941346[MHz]
X_offset = 0[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 7.33595628[Hz]
X_sweep = 240.38461538[kHz]
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Irr_freq = 465.88941346[MHz]
Irr_offset = 5[ppm]
Tri_domain = 19F
Tri_freq = 465.88941346[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 17[us]
X_acq_time = 0.13631488[s]
X_angle = 45[deg]
X_atn = 7.3[dB]
X_pulse = 8.5[us]
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tri_mode = Off
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Initial_wait = 1[s]
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Relaxation_delay = 5[s]
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temp_get = 23.5[degC]
  
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2b



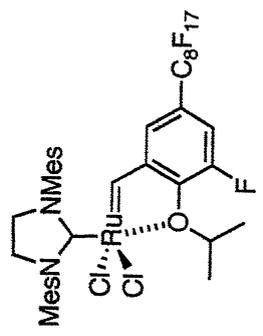
X: parts per Million : 19F

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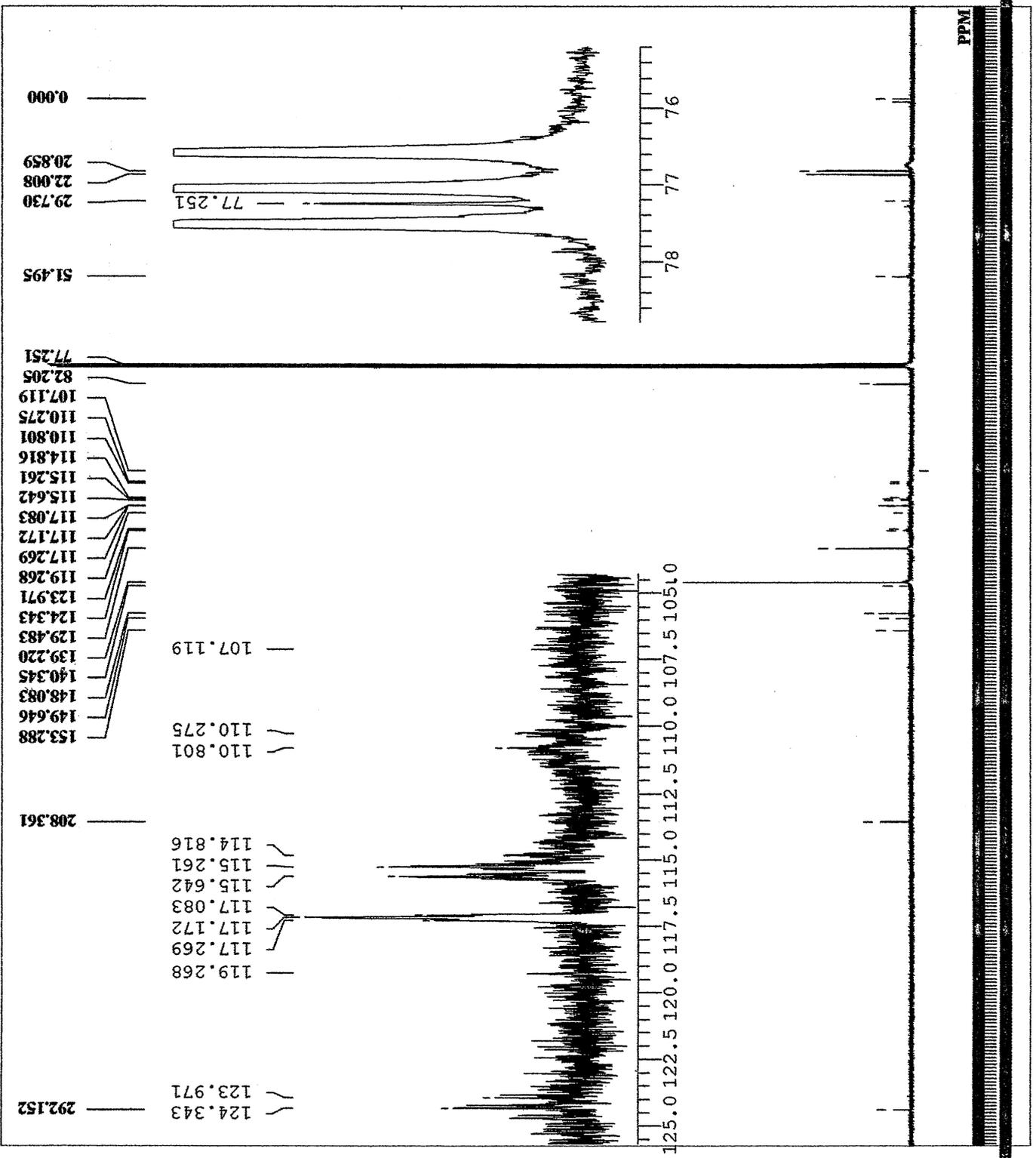
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 135.00 KHz
 5200.00 Hz
 65536
 36036.04 Hz
 39136
 1.8186 sec
 1.2100 sec
 4.50 usec
 1H
 24.9 c
 CDCL3
 0.00 ppm
 0.12 Hz
 26
 RGAIN

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFRO
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQIM
 PD
 PW1
 IRNUC
 CTEMP
 SLYNT
 EXREF
 BF
 RGAIN



2b



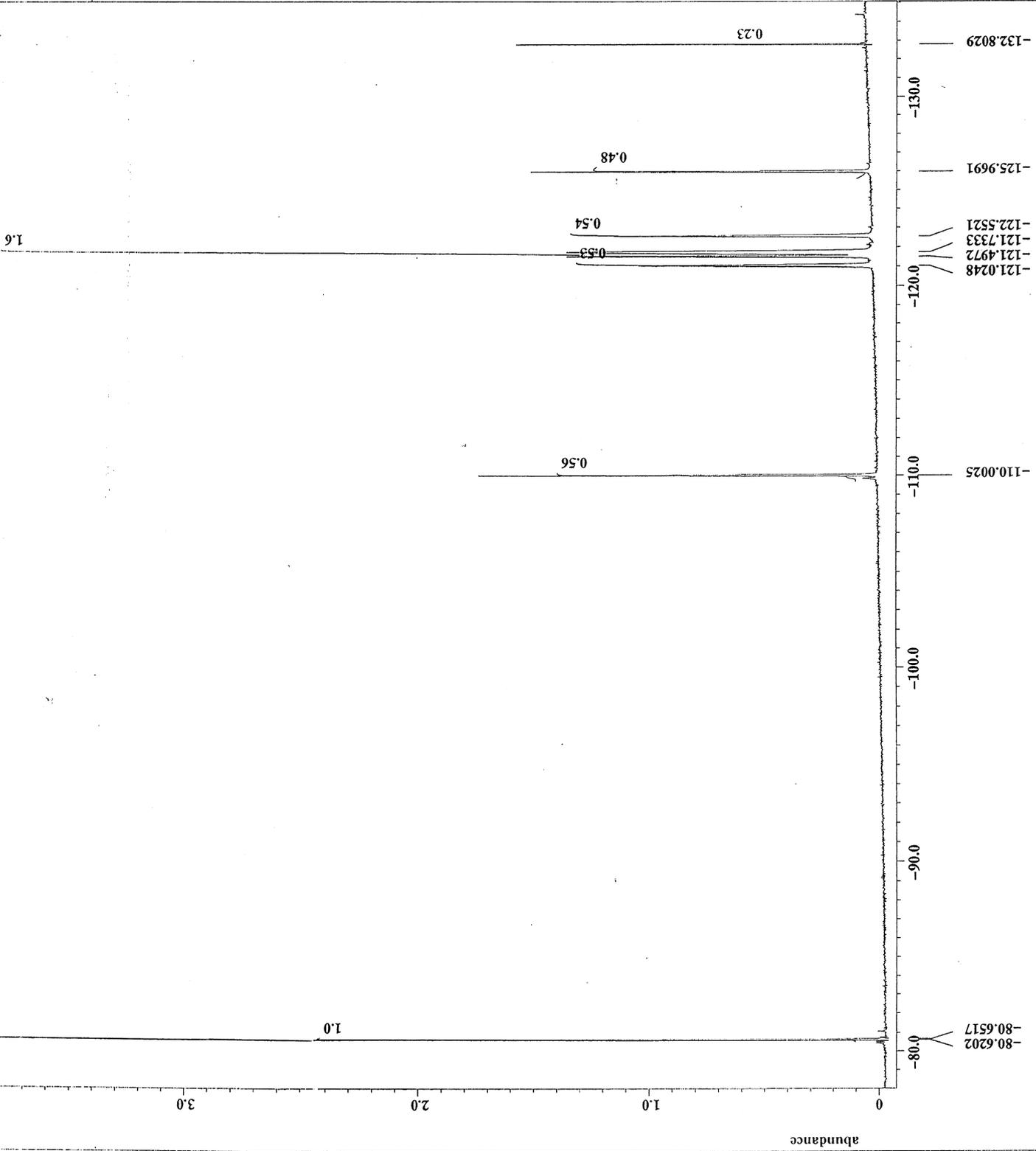
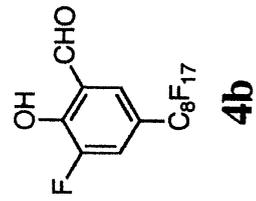

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Spectrometer = DELTA2_NMR

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X_offset = 0[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 7.33595628[Hz]
X_sweep = 240.38461538[kHz]
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Irr_freq = 465.88941346[MHz]
Irr_offset = 5[ppm]
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Tri_offset = 5[ppm]
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Mod_return = 1
Scans = 8
Total_scans = 8

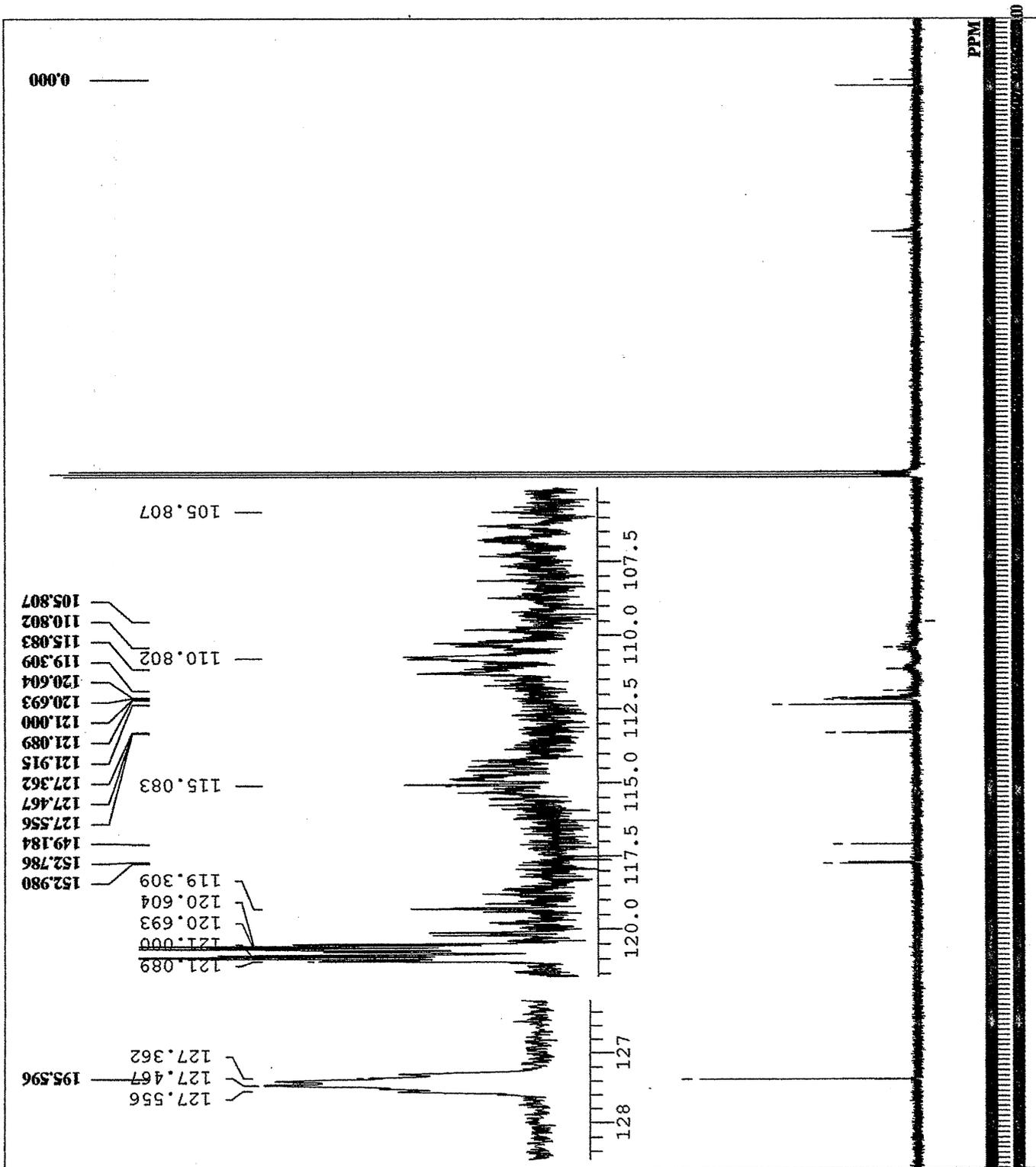
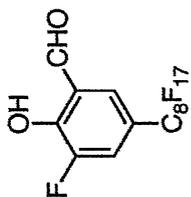
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X_acq_time = 0.13631488[s]
X_angle = 45[deg]
X_atn = 7.3[dB]
X_pulse = 8.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 5.13631488[s]
Temp_get = 23.8[degC]
  
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X : parts per Million : 19F

20100914 3-F perflin 13C 17000.als

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EXMOD BCM
OBFREQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 65536
FREQU 36036.04 Hz
SCANS 17049
ACQTM 1.8186 sec
PD 1.2100 sec
PWI 4.50 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26



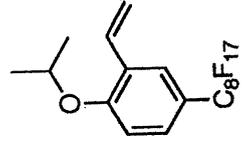

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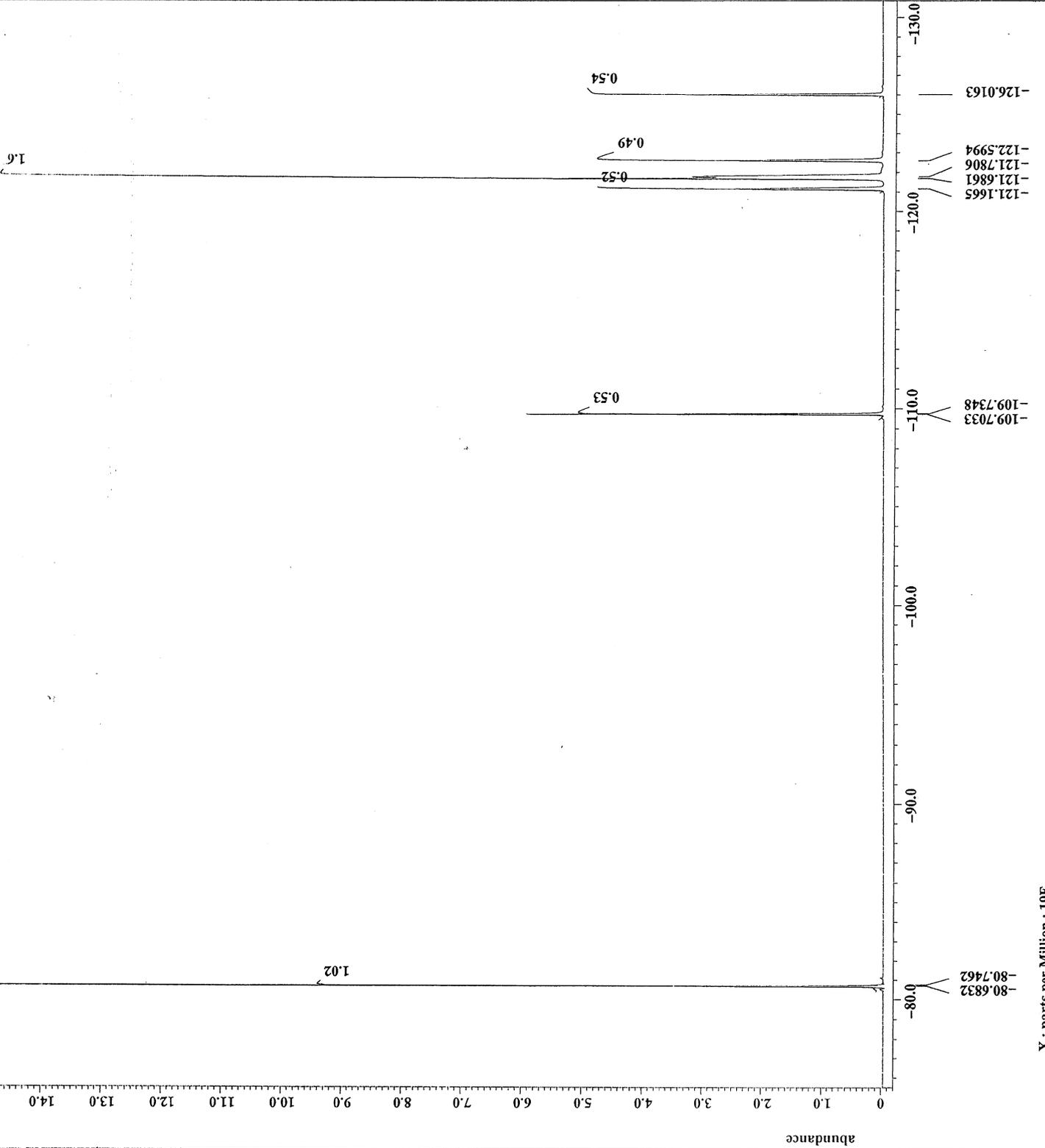
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Dim_title = 19F
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.13631488[s]
X_domain = 19F
X_freq = 465.88941346[MHz]
X_offset = 0[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 7.33595628[Hz]
X_sweep = 240.38461538[kHz]
Irr_domain = 19F
Irr_freq = 465.88941346[MHz]
Irr_offset = 5[ppm]
Tri_domain = 19F
Tri_freq = 465.88941346[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 17[us]
X_acq_time = 0.13631488[s]
X_angle = 45[deg]
X_atn = 7.3[dB]
X_pulse = 8.5[us]
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Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
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Relaxation_delay = 5[s]
Repetition_time = 5.13631488[s]
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```



5a



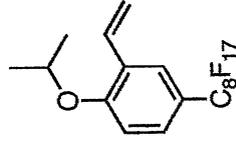
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20100913 F.E., fS/Kj fh 13C 260

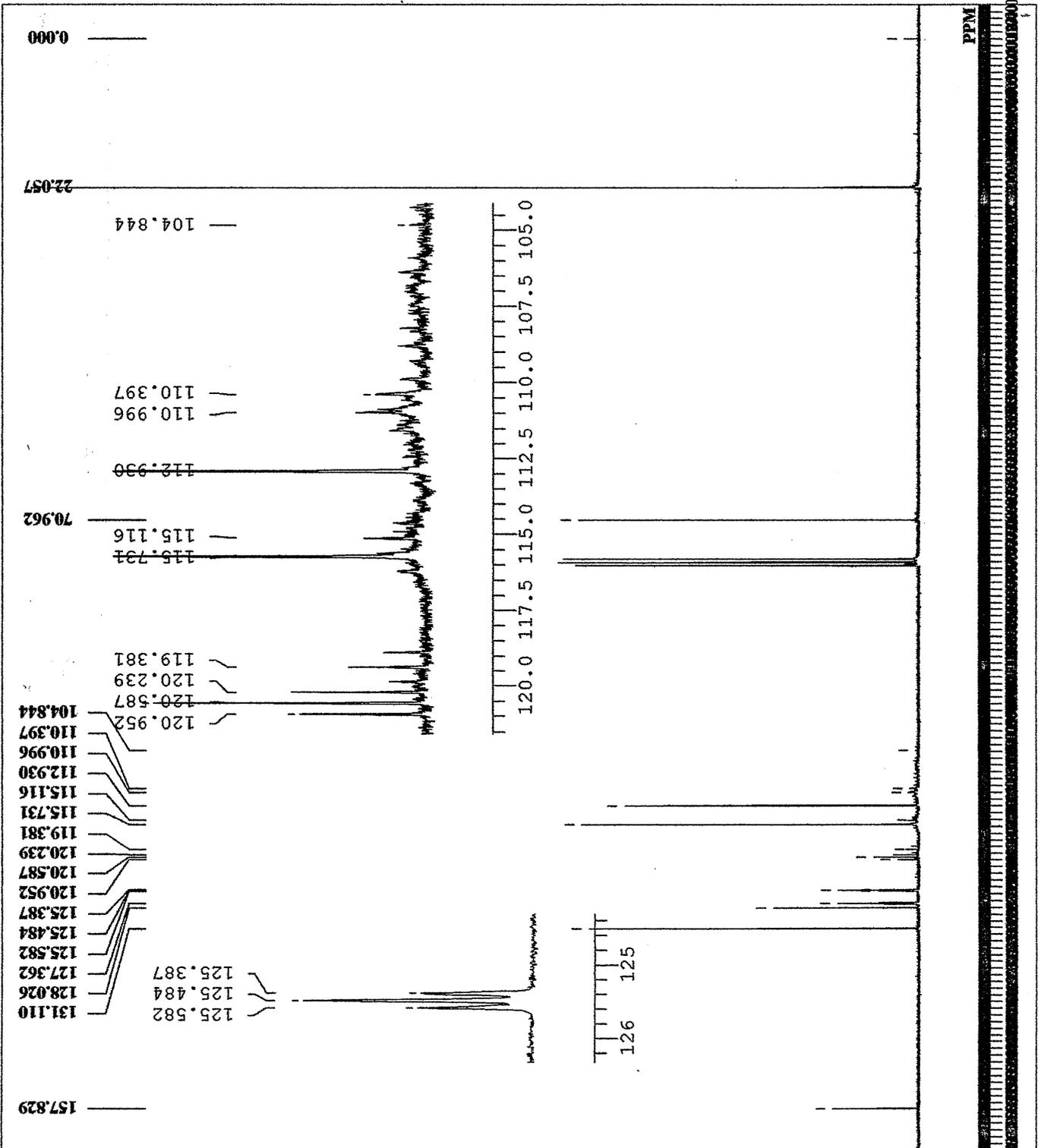
Mon Sep 13 18:02:39 2010

DFILE
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 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

13C
 BCM
 67.80 MHz
 135.00 KHz
 5200.00 Hz
 65536
 36036.04 Hz
 25066
 1.8186 sec
 1.2100 sec
 4.50 usec
 1H
 24.2 c
 CDCl3
 0.00 ppm
 0.12 Hz
 24



5a

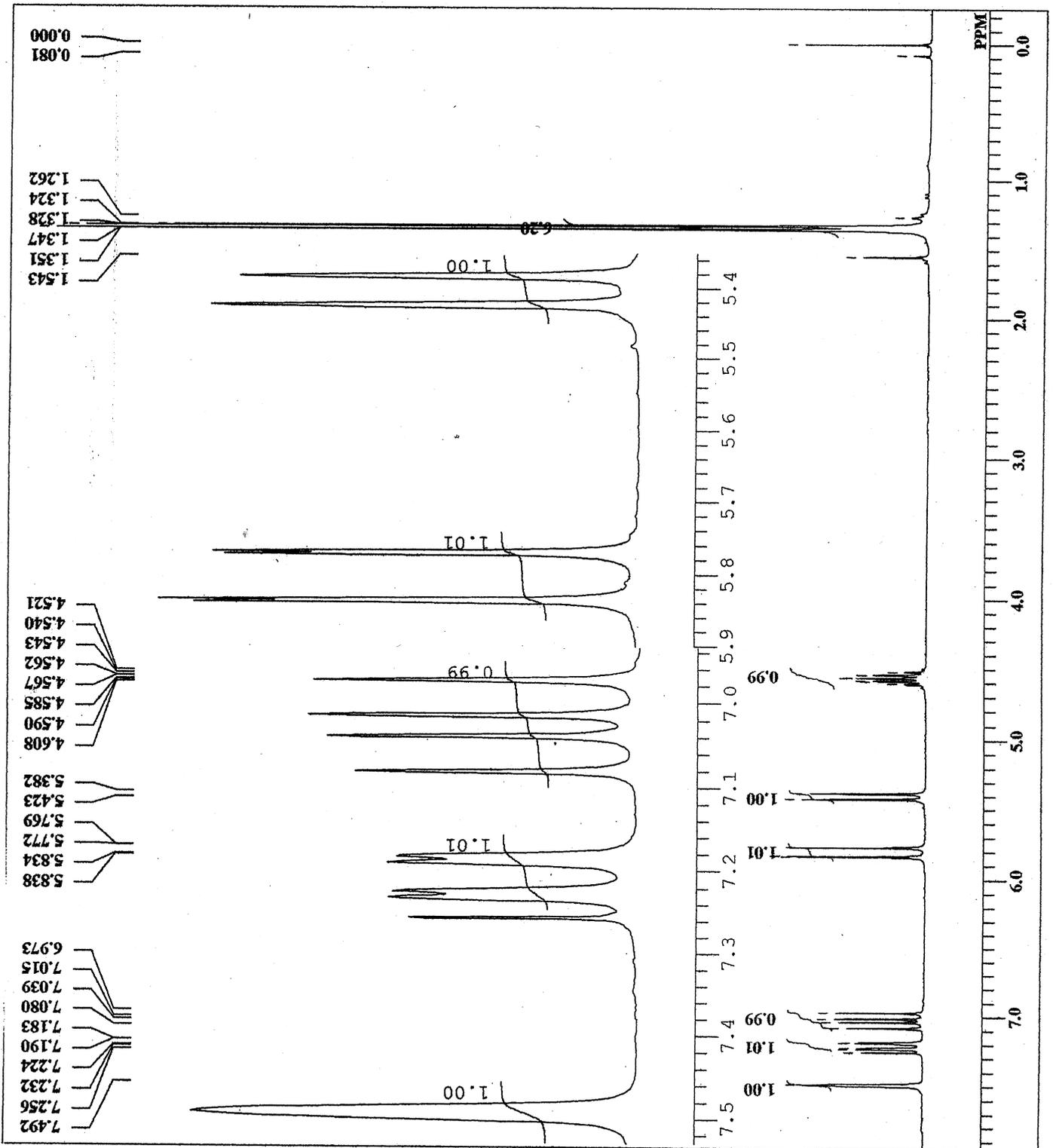
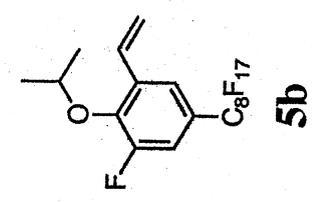


217N8CEZ26"ull@wittig"%"%zll@F.

Wed Aug 26 19:37:13 2009

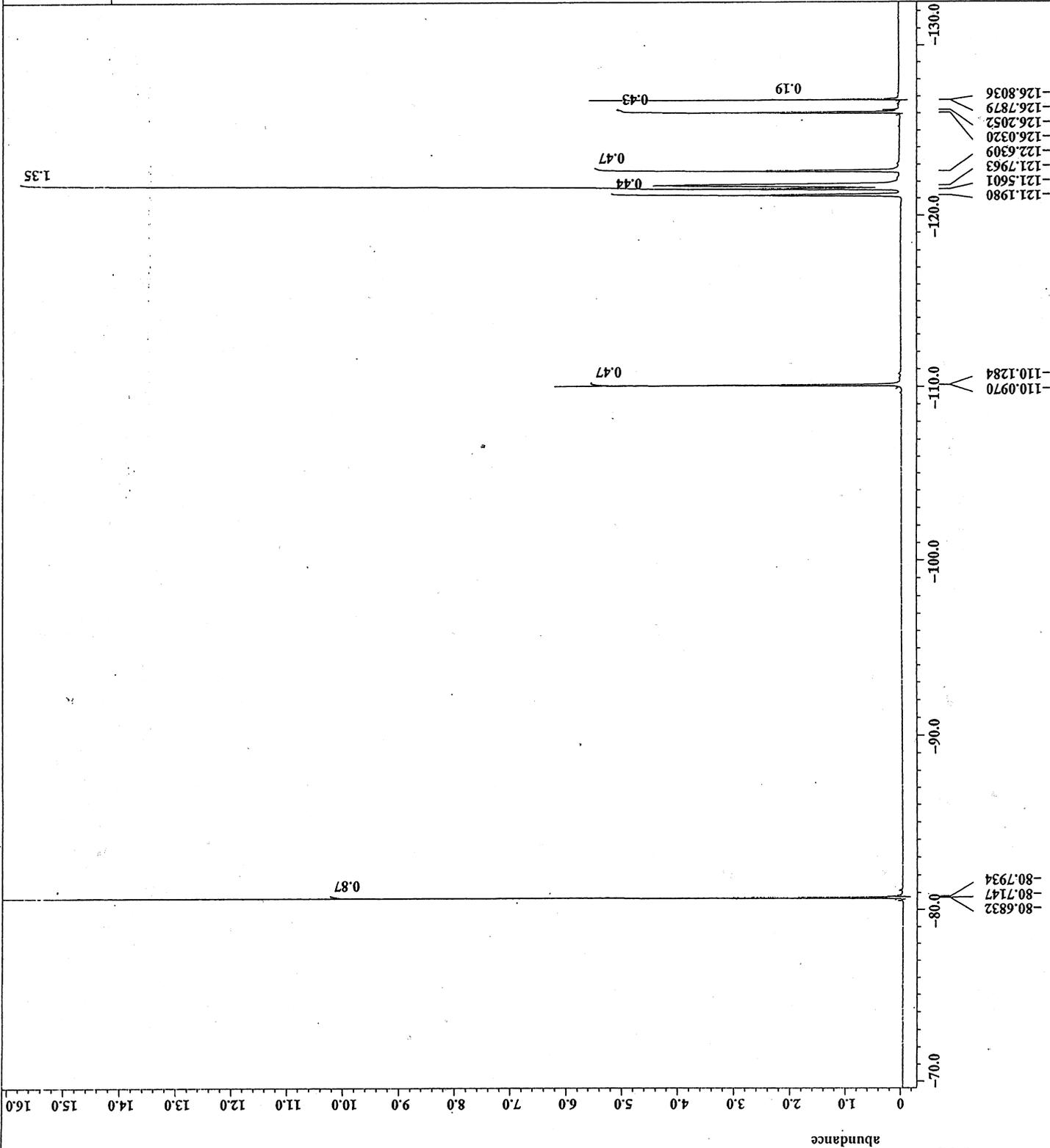
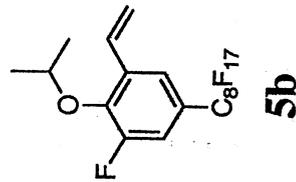
DFILE
COMINT
DATIM
OBNUC
EXMOD
OBFREQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
32
3.0331 sec
3.9670 sec
5.40 usec
1H
26.5 c
CDCL3
0.00 ppm
0.12 Hz
16





Filename = kobayasiC8Friga-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 19F-single_pulse
Solvent = CHLOROFORM-D
Creation_time = 19-SEP-2009 18:08:21
Revision_time = 19-SEP-2009 18:13:03
Current_time = 19-SEP-2009 18:13:44
Data_format = ID COMPLEX
Dim_size = 26214
Dim_title = 19F
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M]
X_acq_duration = 0.13631488[s]
X_domain = 19F
X_freq = 465.88941346[MHz]
X_offset = 0[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 7.33595628[Hz]
X_sweep = 240.38461538[kHz]
Irr_domain = 19F
Irr_freq = 465.88941346[MHz]
Irr_offset = 5[ppm]
Irr_domain = 19F
Tri_freq = 465.88941346[MHz]
Tri_offset = FALSE
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 17[us]
X_acq_time = 0.13631488[s]
X_angle = 45[deg]
X_atn = 7.3[dB]
X_pulse = 8.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 5.13631488[s]
Temp_get = 23[dc]



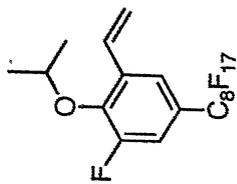
X : parts per Million : 19F

217N8E226'u fJllffl@wittig%

H18,11,16 tomioka
Thu Aug 27 02:07:44 2009

DFILE
COMINT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FRFQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
4000
1.7900 sec
1.2100 sec
4.50 usec
1H
24.3 c
CDCL3
0.00 ppm
0.12 Hz
27



5b

