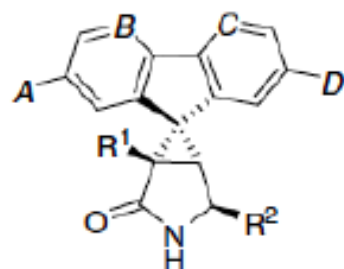


**Uncatalysed diaryldiazo cyclopropanations on bicyclic lactams:
access to annulated prolines**

Lawrence Harris, Martin Gilpin, Amber L. Thompson, Andrew R.
Cowley and Mark G. Moloney*

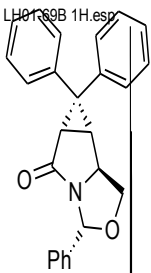


Entry	A	B	C	D	R ¹	R ²	S.aureus /(cm)	E.coli /(cm)	Concentration /(mg/mL)
1	H	(C)H	(C)H	H	H	CH ₂ OH	2.2	1.3	2.5
2	H	(C)H	(C)H	H	CO ₂ Et	CH ₂ OH	2.3	1.4	3.9
3	H	(C)H	(C)H	H	CH ₂ OH	CH ₂ OH	1.4	-	4.0
4	H	(C)H	(C)H	H	CO ₂ H	CH ₂ OH	-	1.5	4.1
5	H	(C)H	(C)H	H	CO ₂ H	CO ₂ H	2.3	1.3	4.0
6	H	(C)H	(C)H	H	H	CO ₂ H	1.4	2.7	4.0
7	H	(C)H	(C)H	Br	CO ₂ Et	CH ₂ OH	-	-	4.0
8	Br	(C)H	(C)H	H	CO ₂ Et	CH ₂ OH	1.6(H)	2.4	4.0
9	H	(C)CO ₂ Me	(C)H	H	CO ₂ Et	CH ₂ OH	1.4(H)	1.7	4.2
10	H	(C)H	CO ₂ Me	H	CO ₂ Et	CH ₂ OH	1.7(H)	1.9	4.0
11	H	N	(C)H	H	CO ₂ Et	CH ₂ OH	-	1.4(H)	4.0
12	H	(C)H	N	H	CO ₂ Et	CH ₂ OH	1.4(H)	1.6(H)	4.0

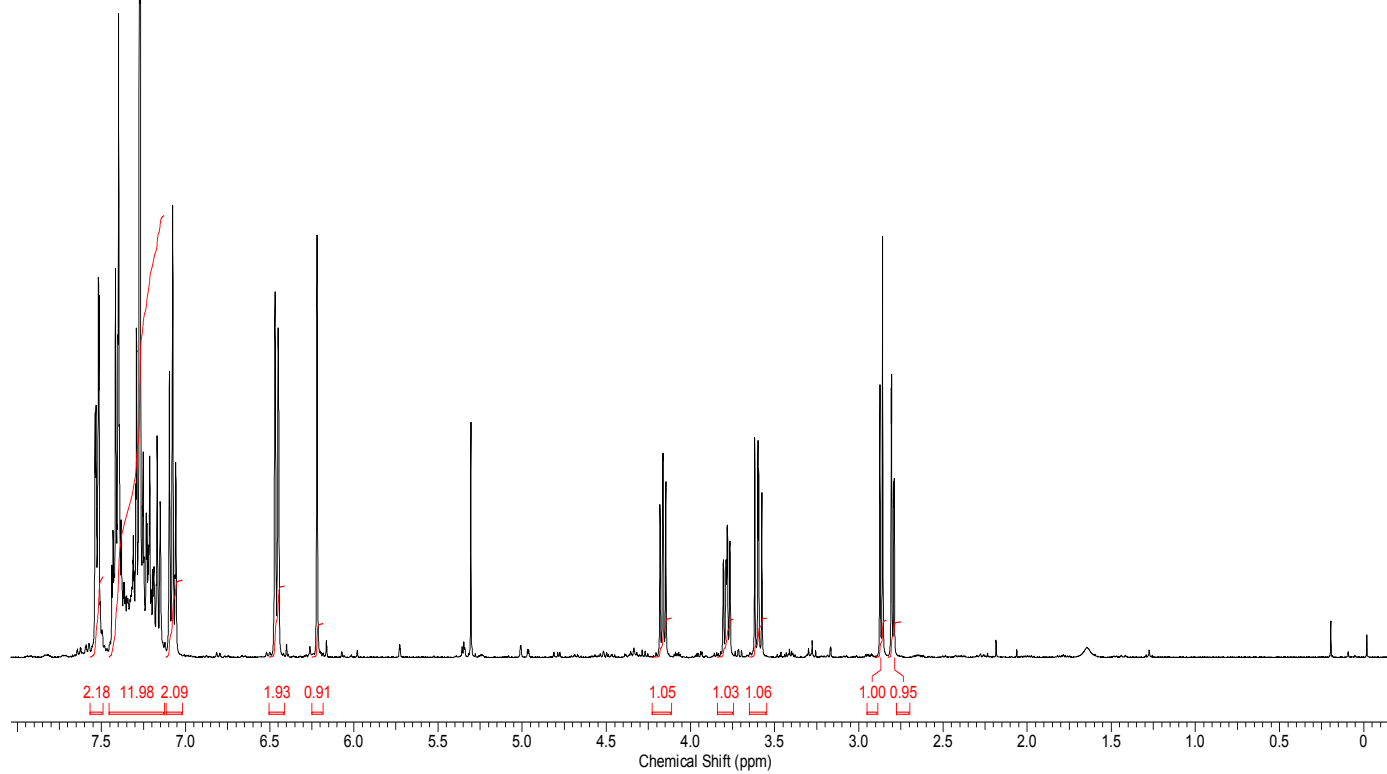
		R ³	X			
13		H	H	-	-	4.0
14		CO ₂ Et	H	-	-	4.0
15		CO ₂ Et	OMe	-	-	4.0
16		CO ₂ H	OMe	-	-	4.0

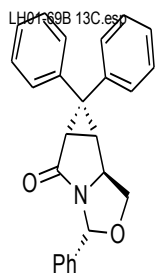
Table 4: Antibacterial activity data of selected compounds ((H) denotes hazy-zone)

LH01-39B 1H.esy

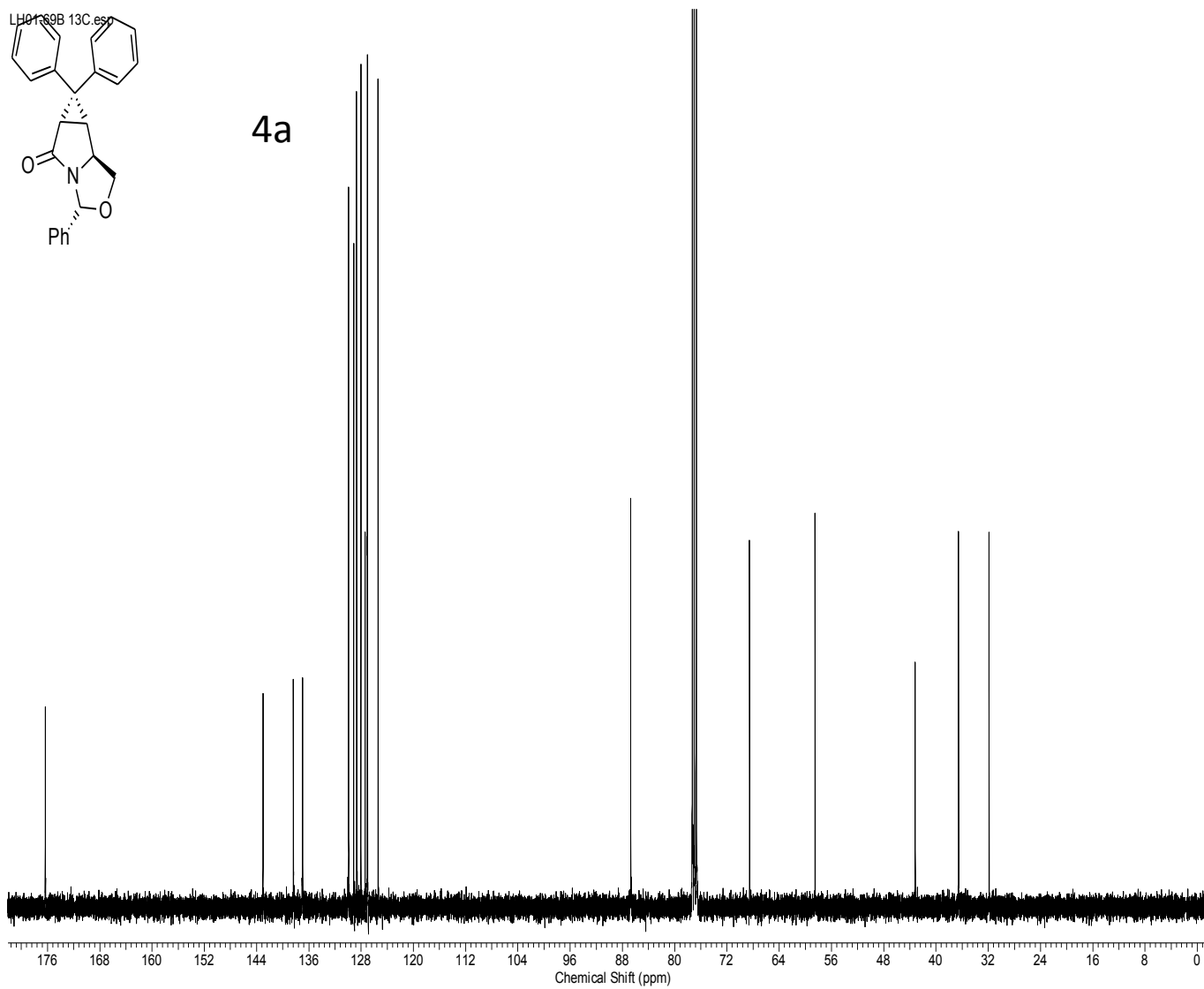


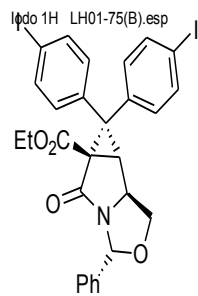
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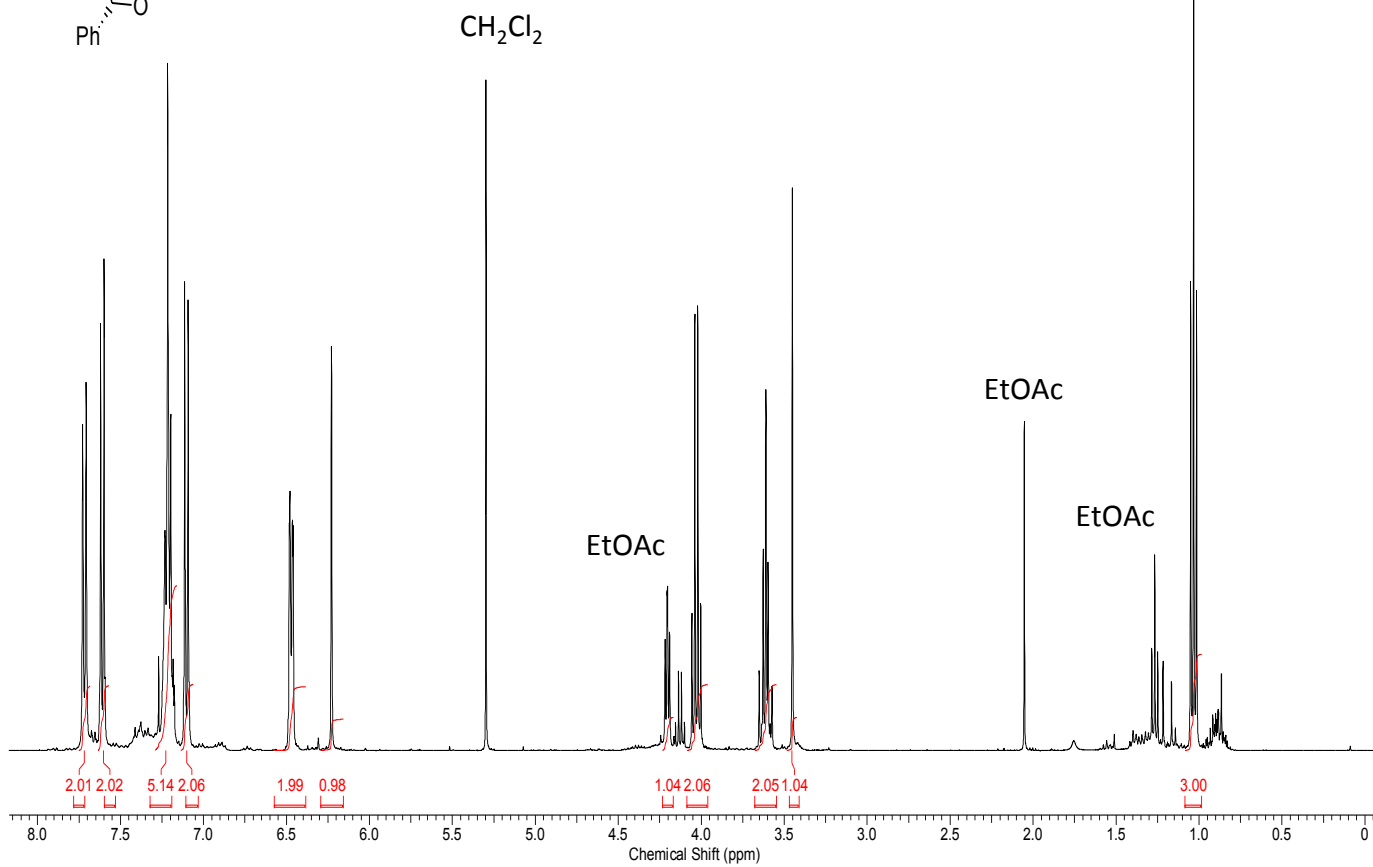


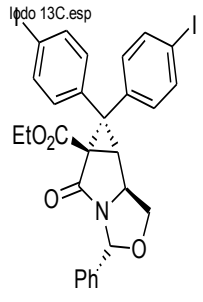
4a



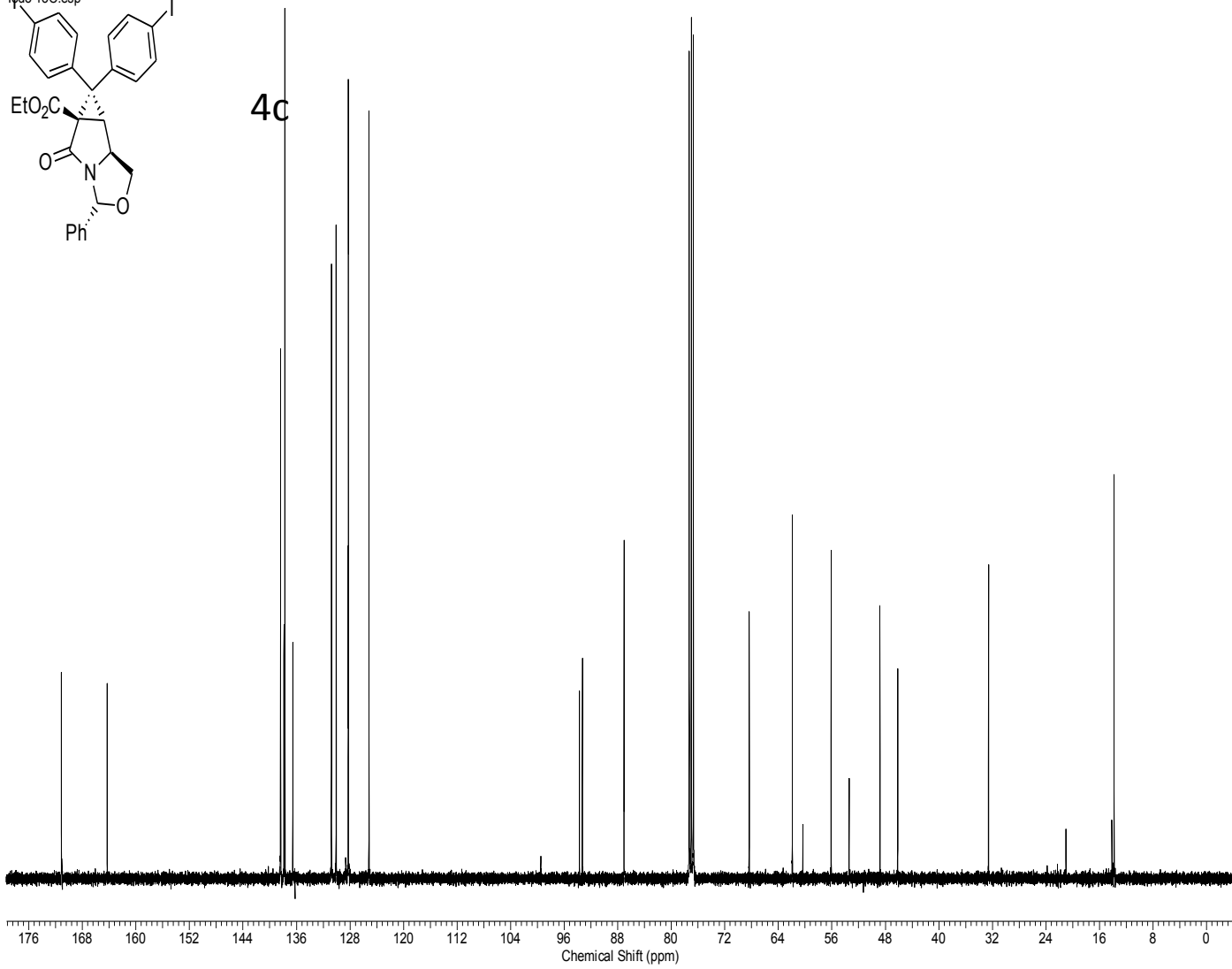


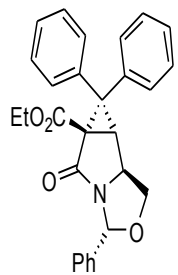
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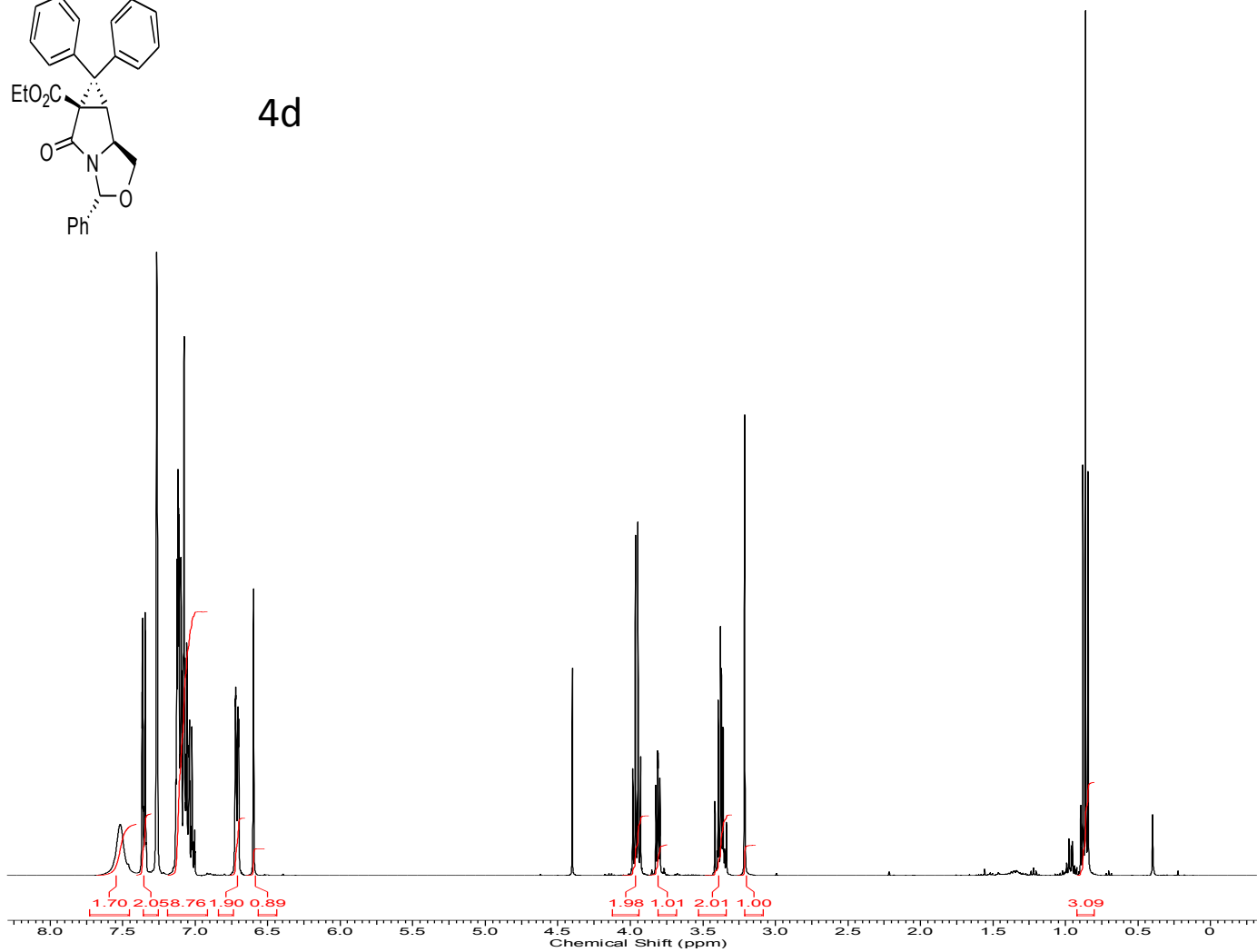


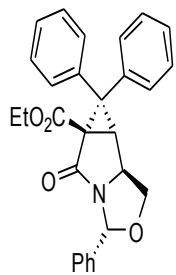
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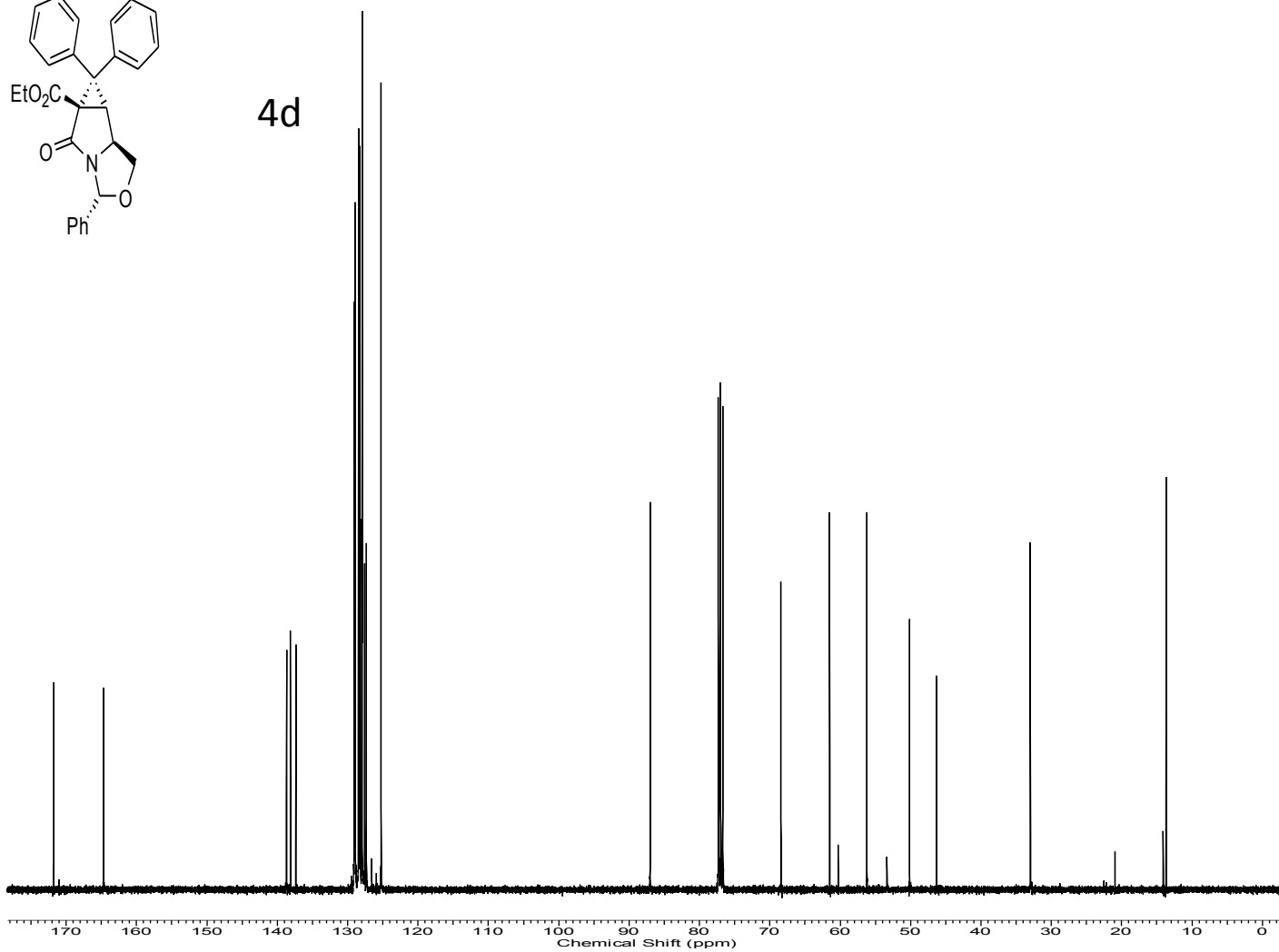


4d

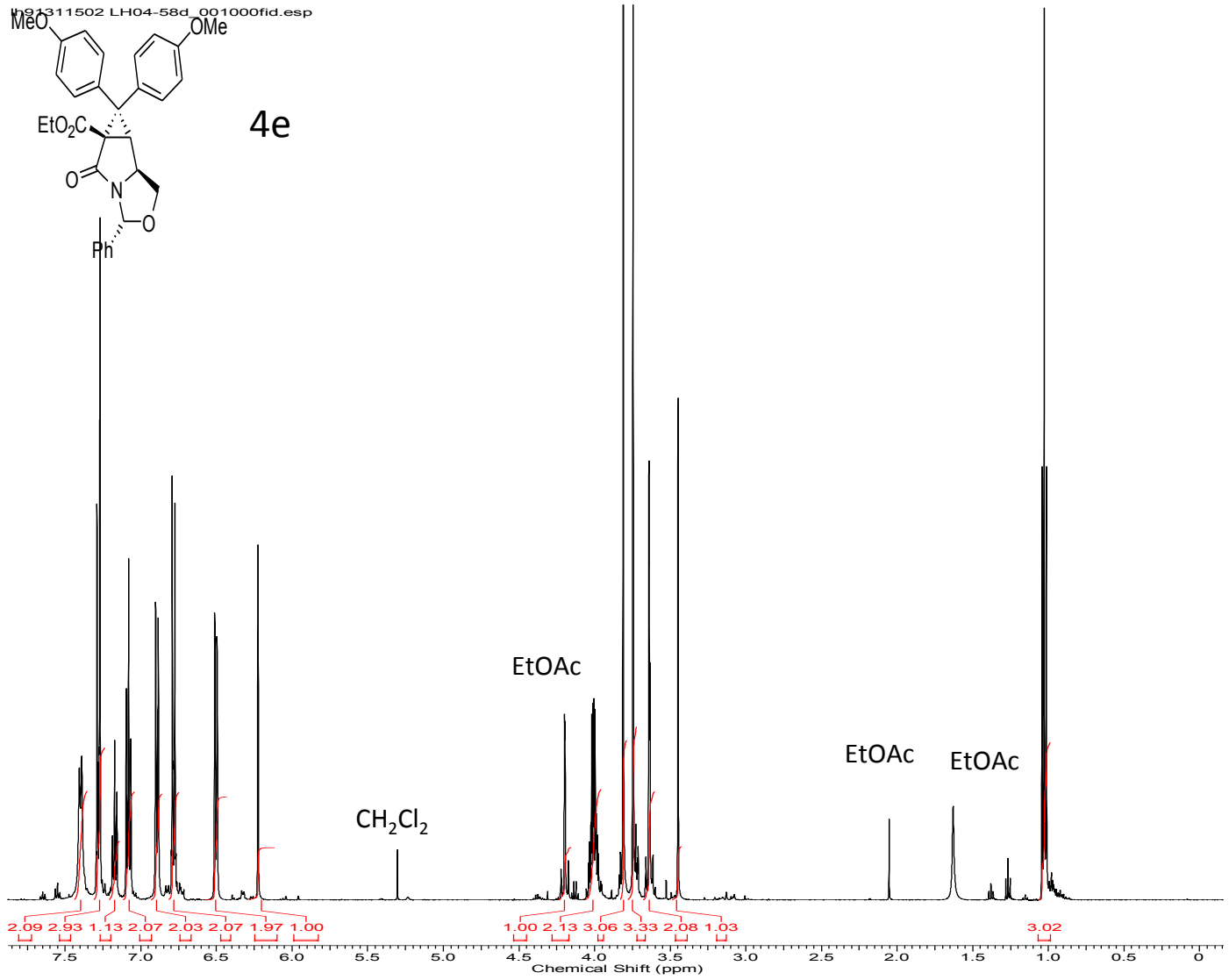


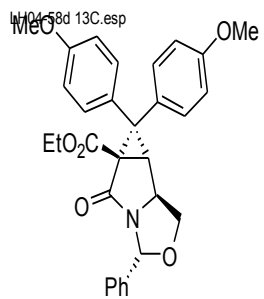


4d

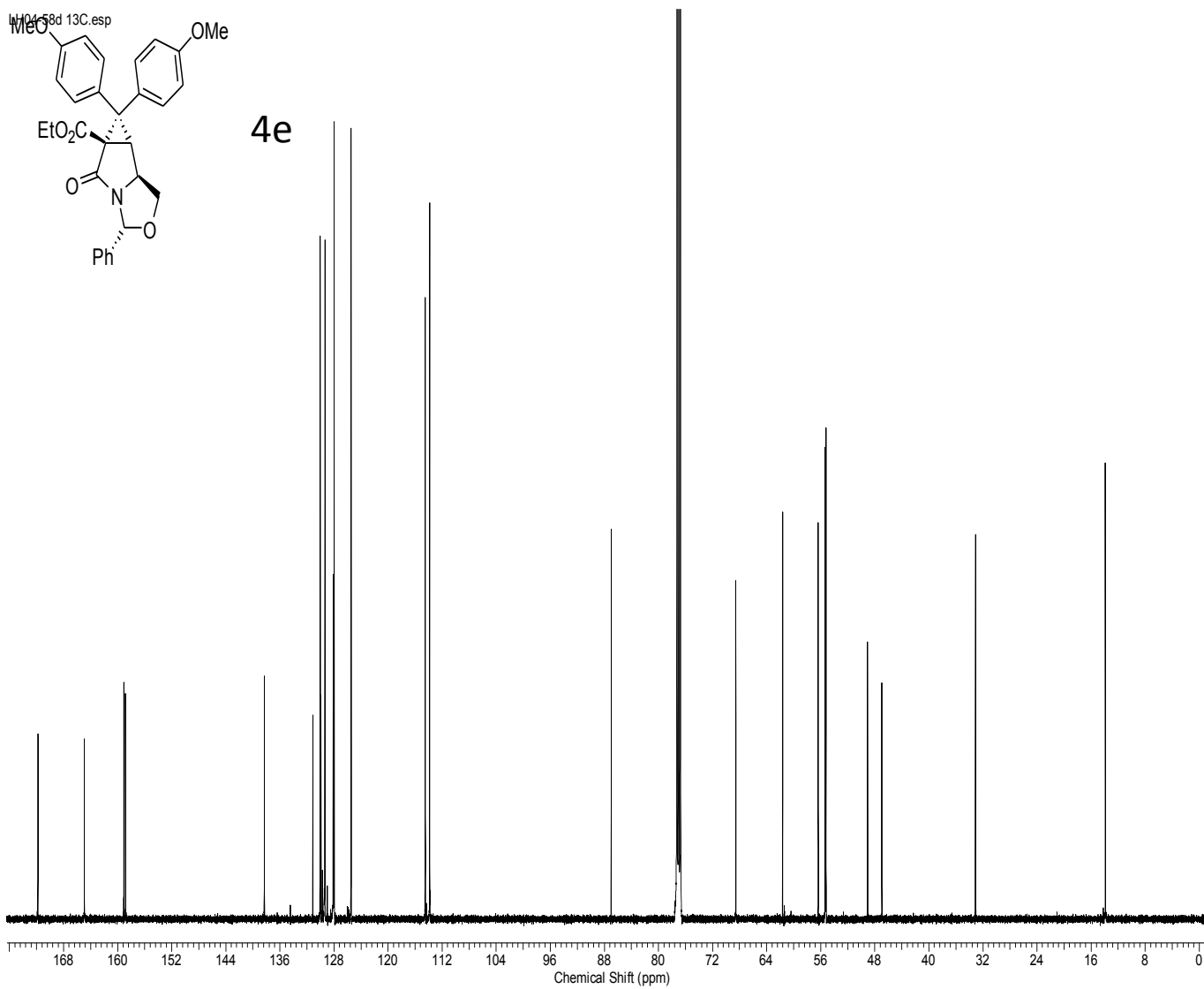


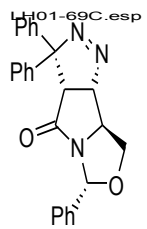
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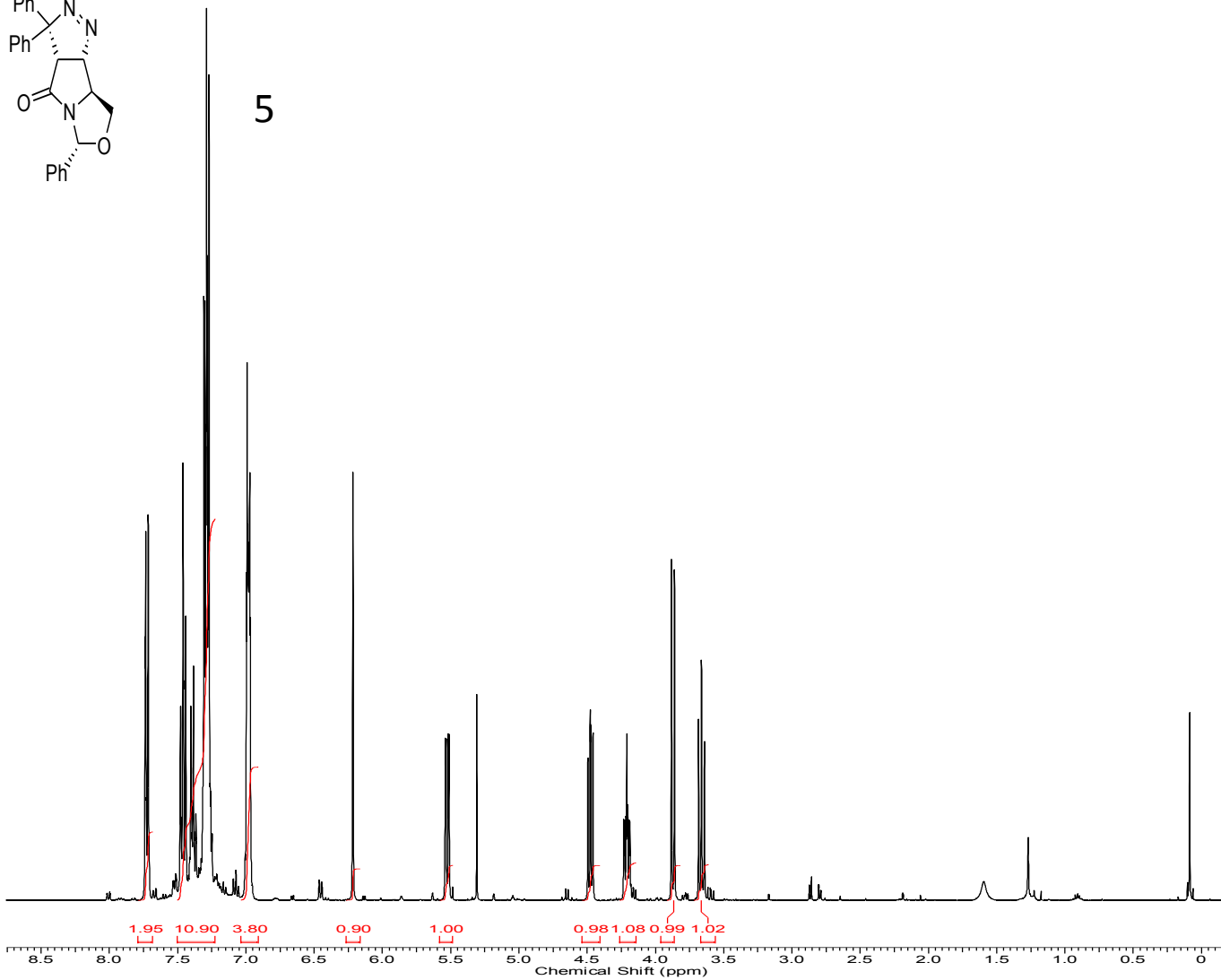


4e

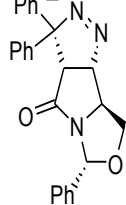




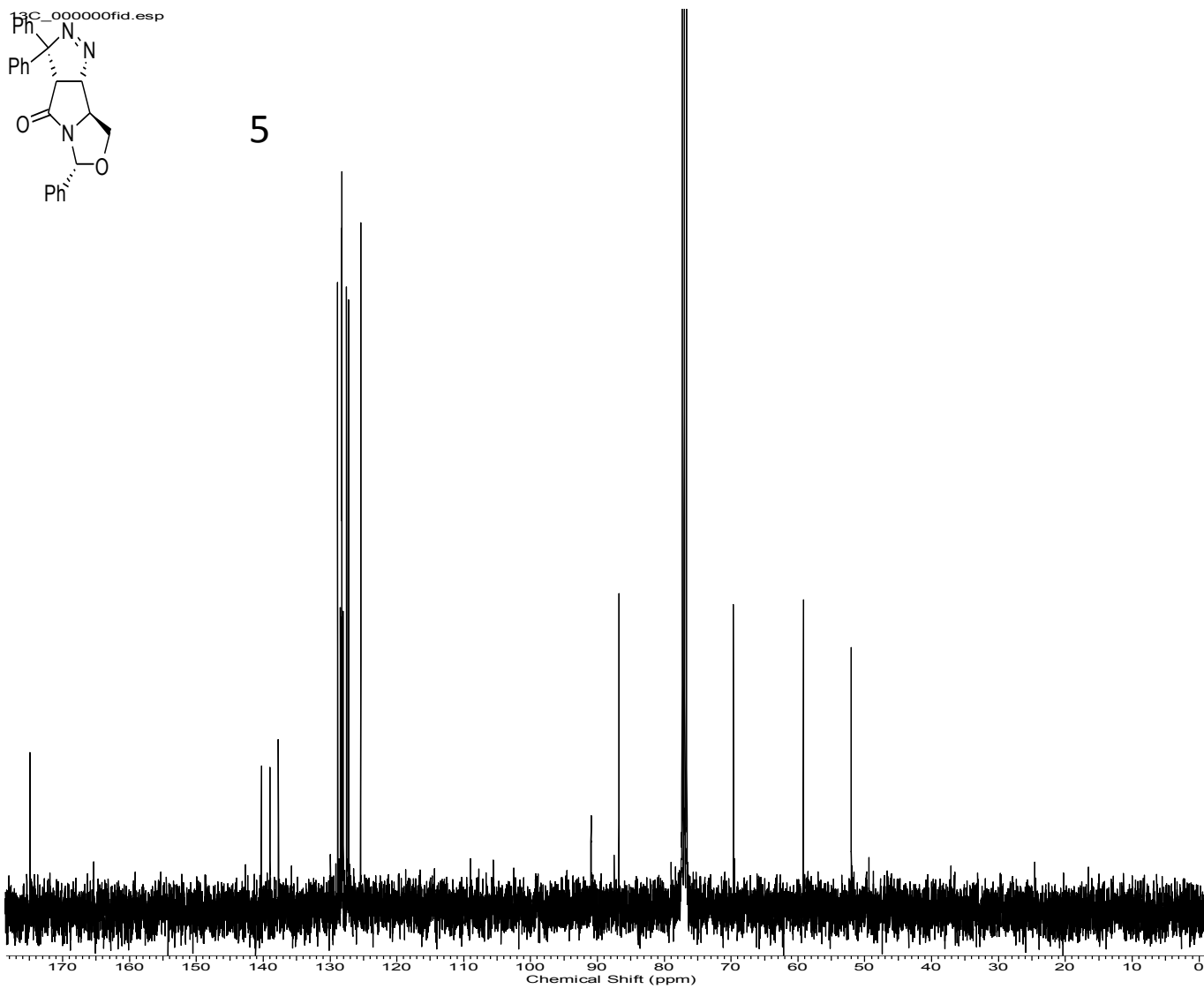
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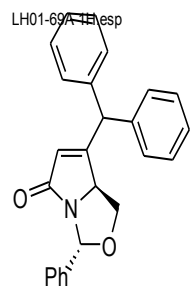


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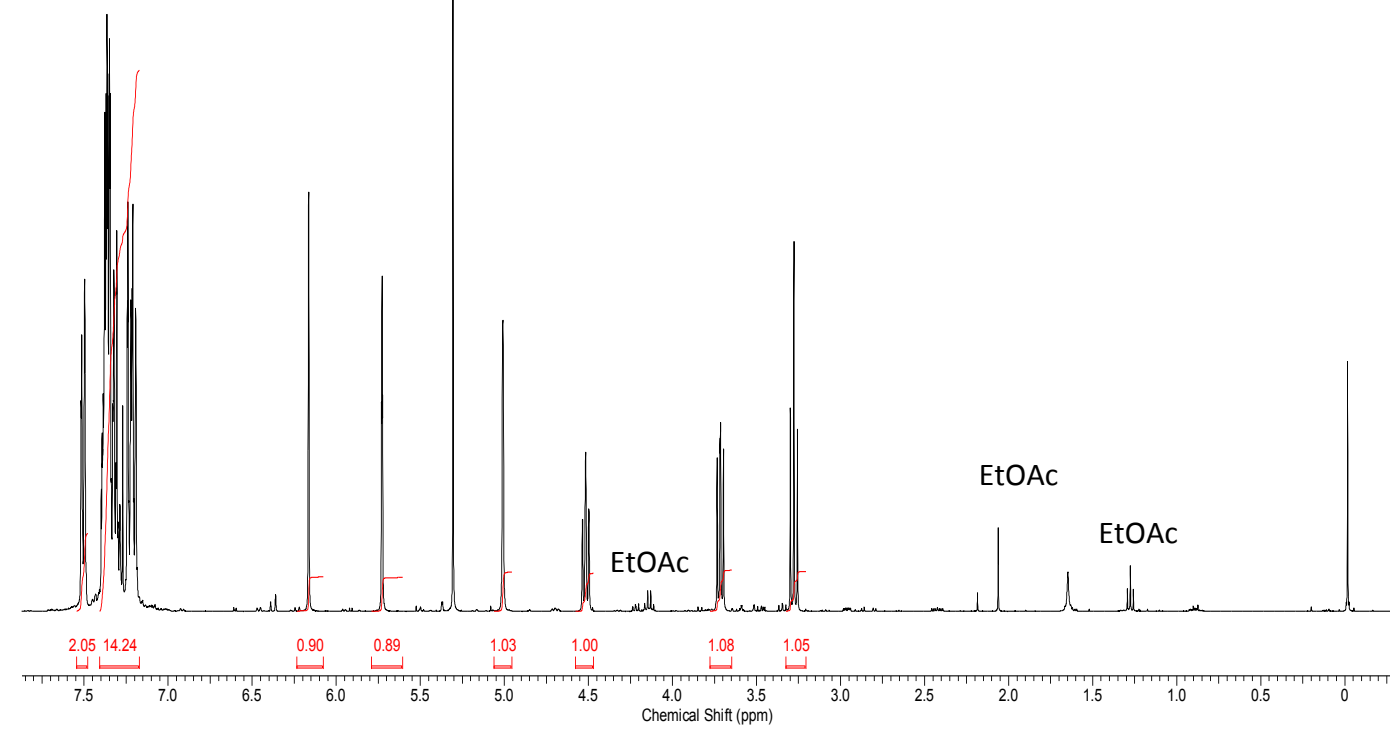
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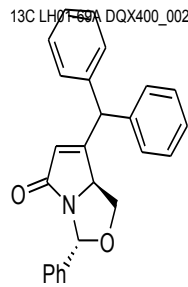


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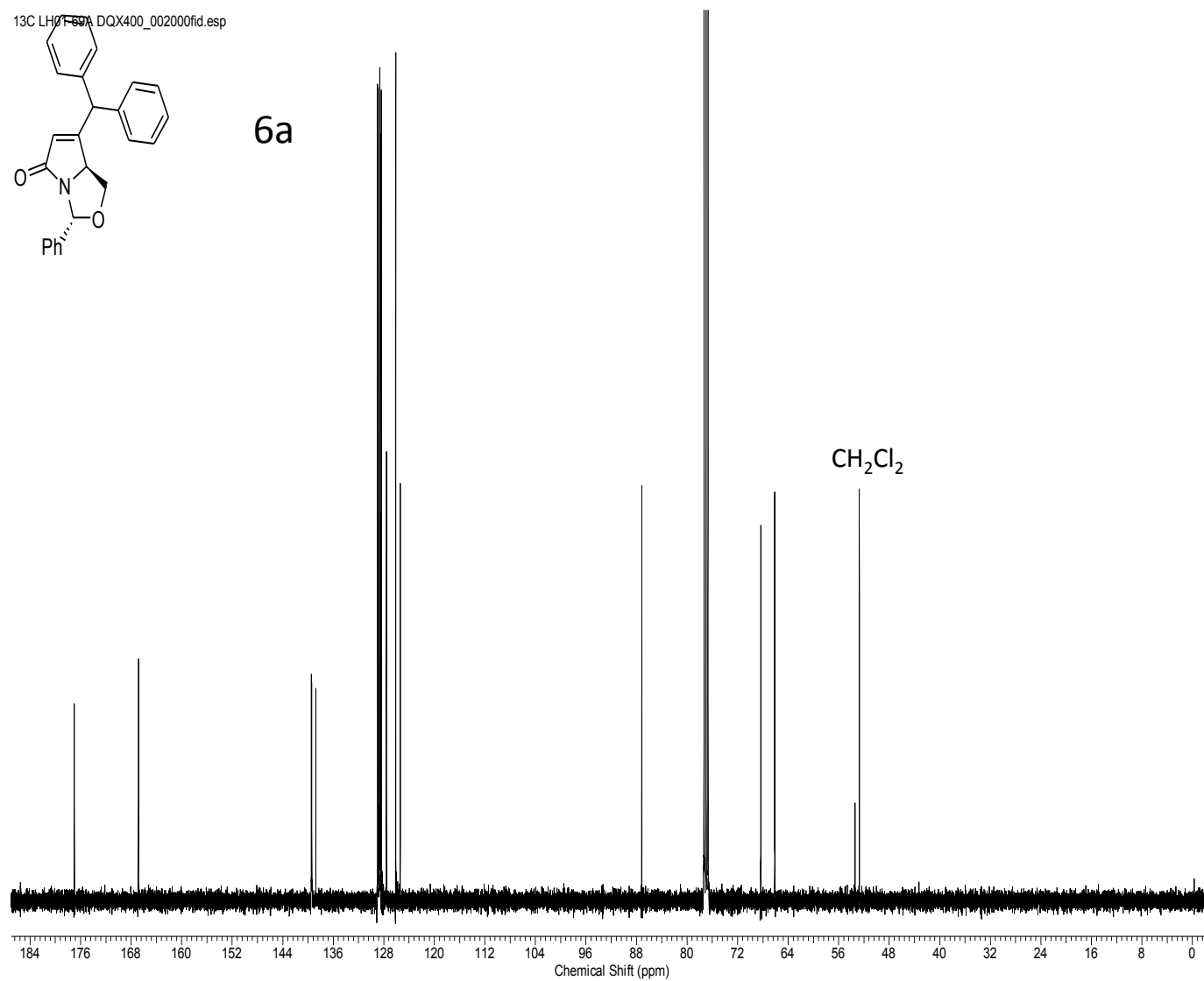
CH₂Cl₂

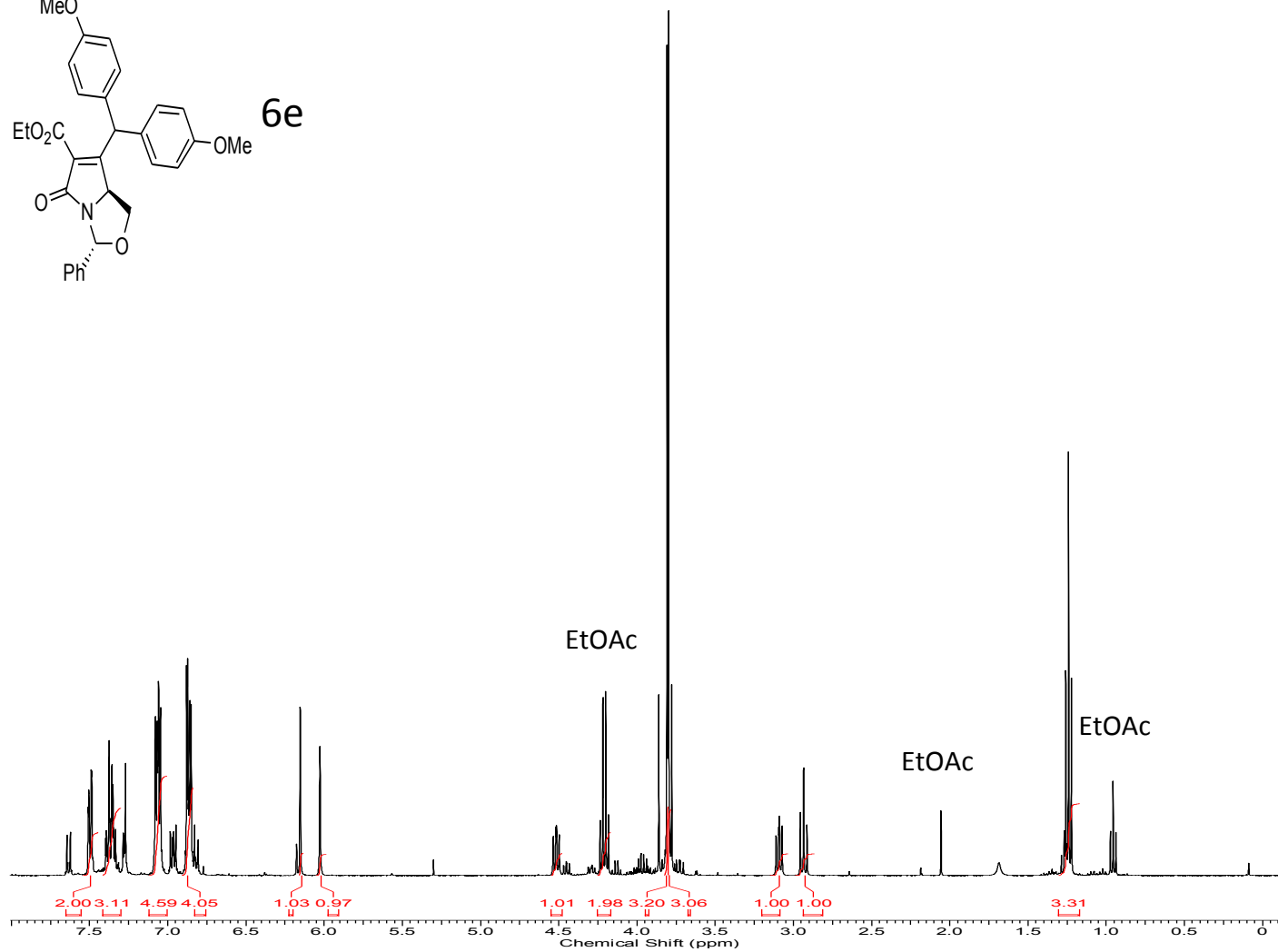
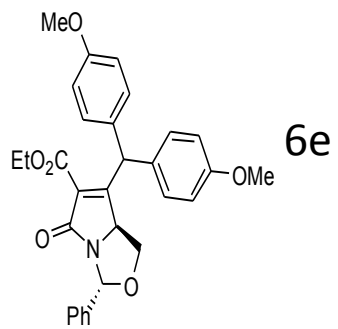


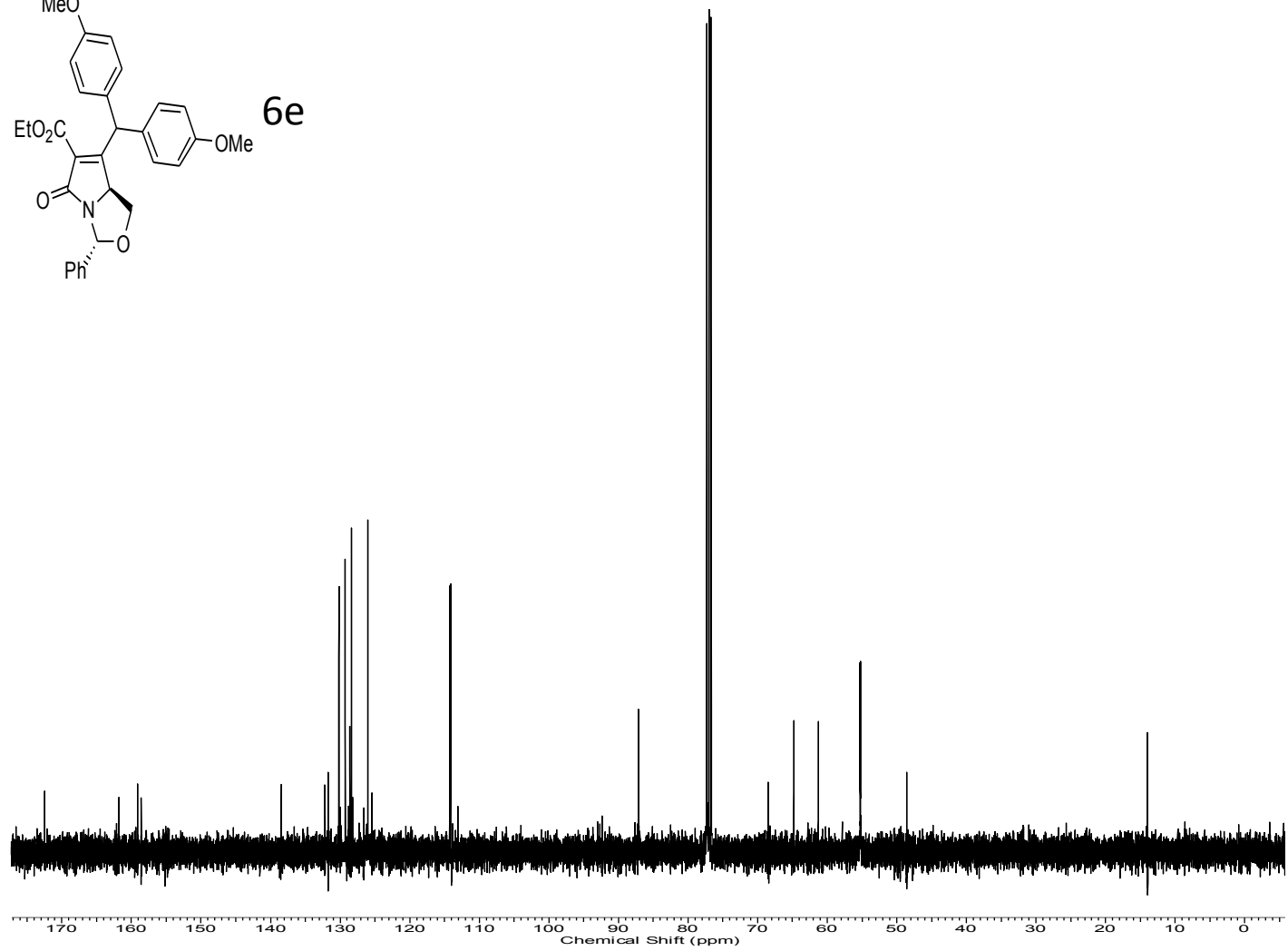
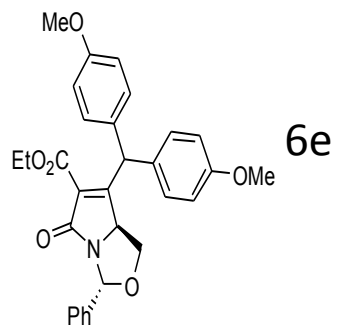
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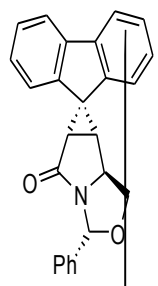


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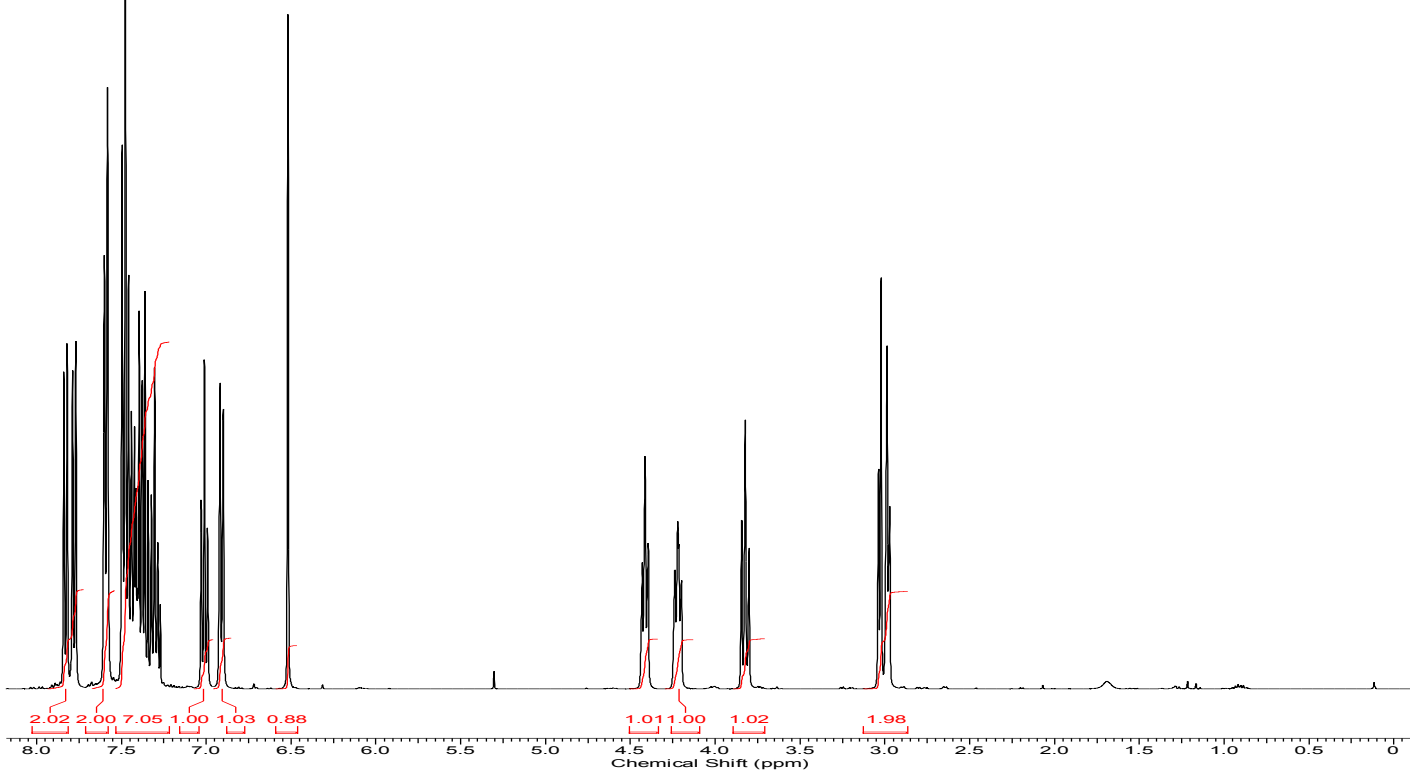


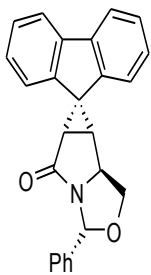




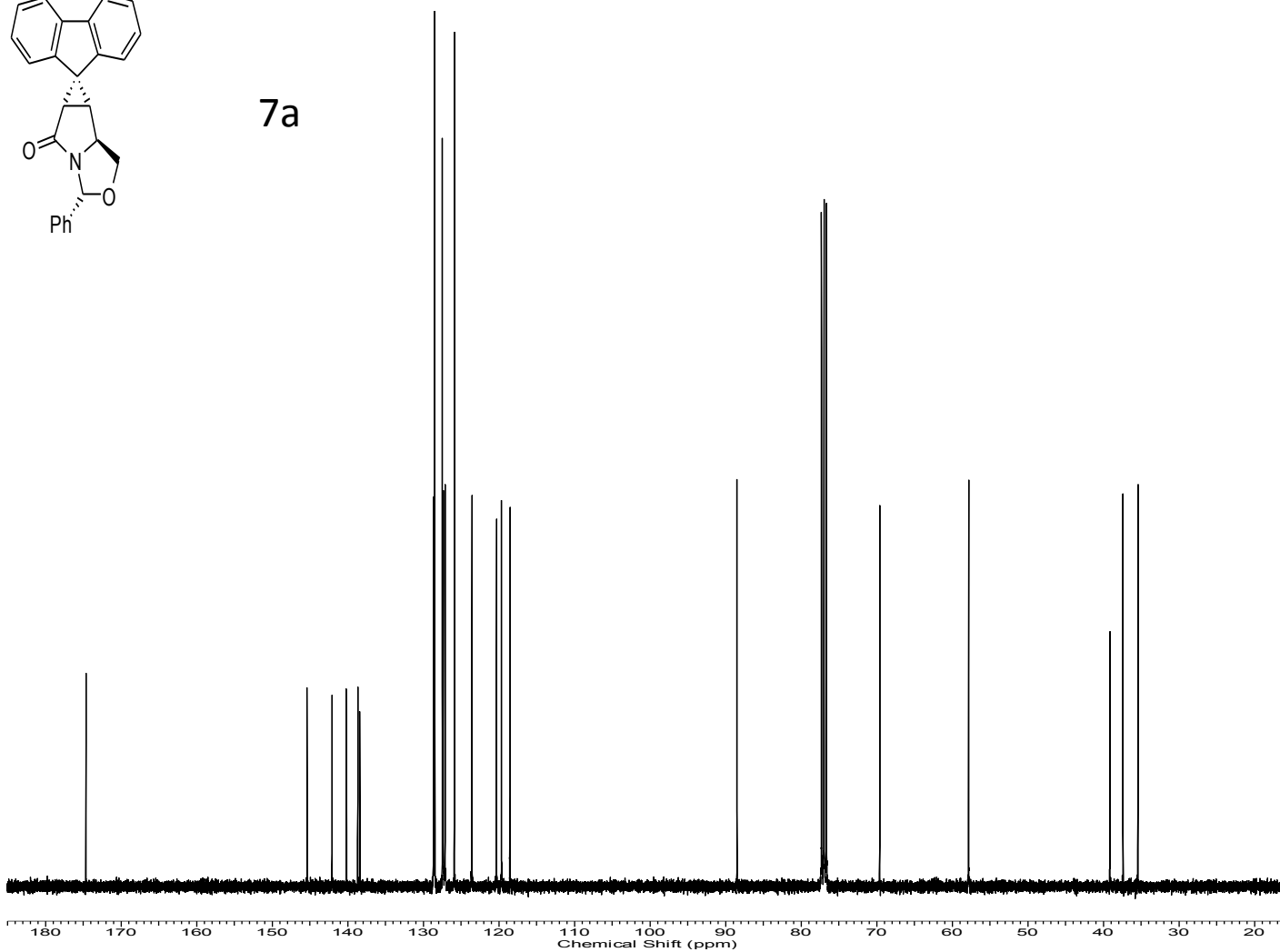


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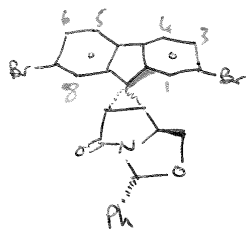


7a



7b

Instrument AVC500
9694 Lawrence Harris 2/4/08



3.044 (2H, s, C(6)H, C(7)H) → 3.10

3.84 (1H, dd, J 8.0, 9.0, C(4)H'H)

4.24 (1H, dd, J 6.4, 9.0, C(5)H)

4.45 (1H, dd, J 6.4, 8.0, C(6)H'H)

6.51 (1H, s, C(2)H)

7.06 (1H, d, J 1.6, Ar(1')H)

7.37-7.61 (1H, m, ArH)

7.44-7.53 (4H, m, ArH)

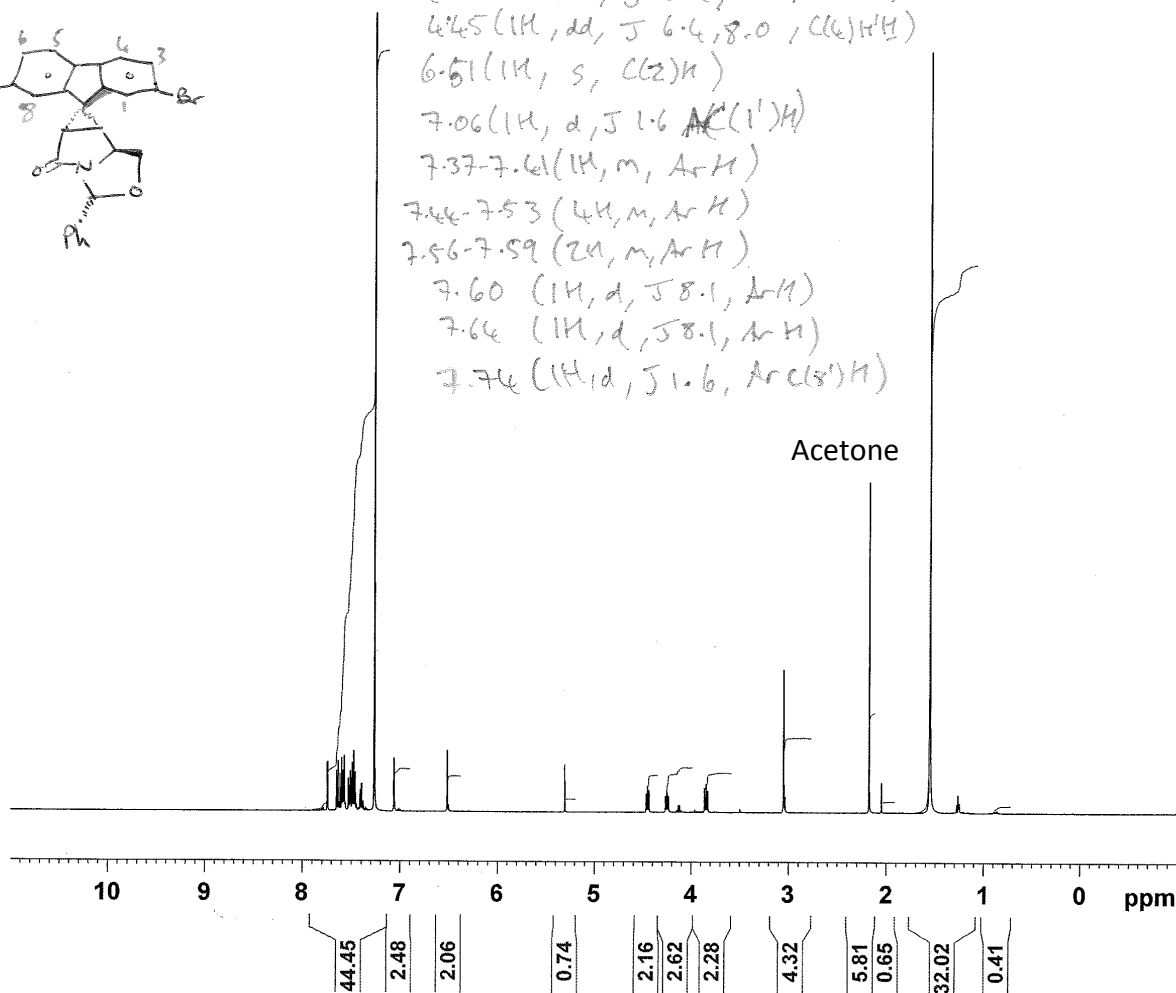
7.56-7.59 (2H, m, ArH)

7.60 (1H, d, J 8.1, ArH)

7.64 (1H, d, J 8.1, ArH)

7.74 (1H, d, J 1.6, Ar(5')H)

Acetone



NMROCHEM.OX

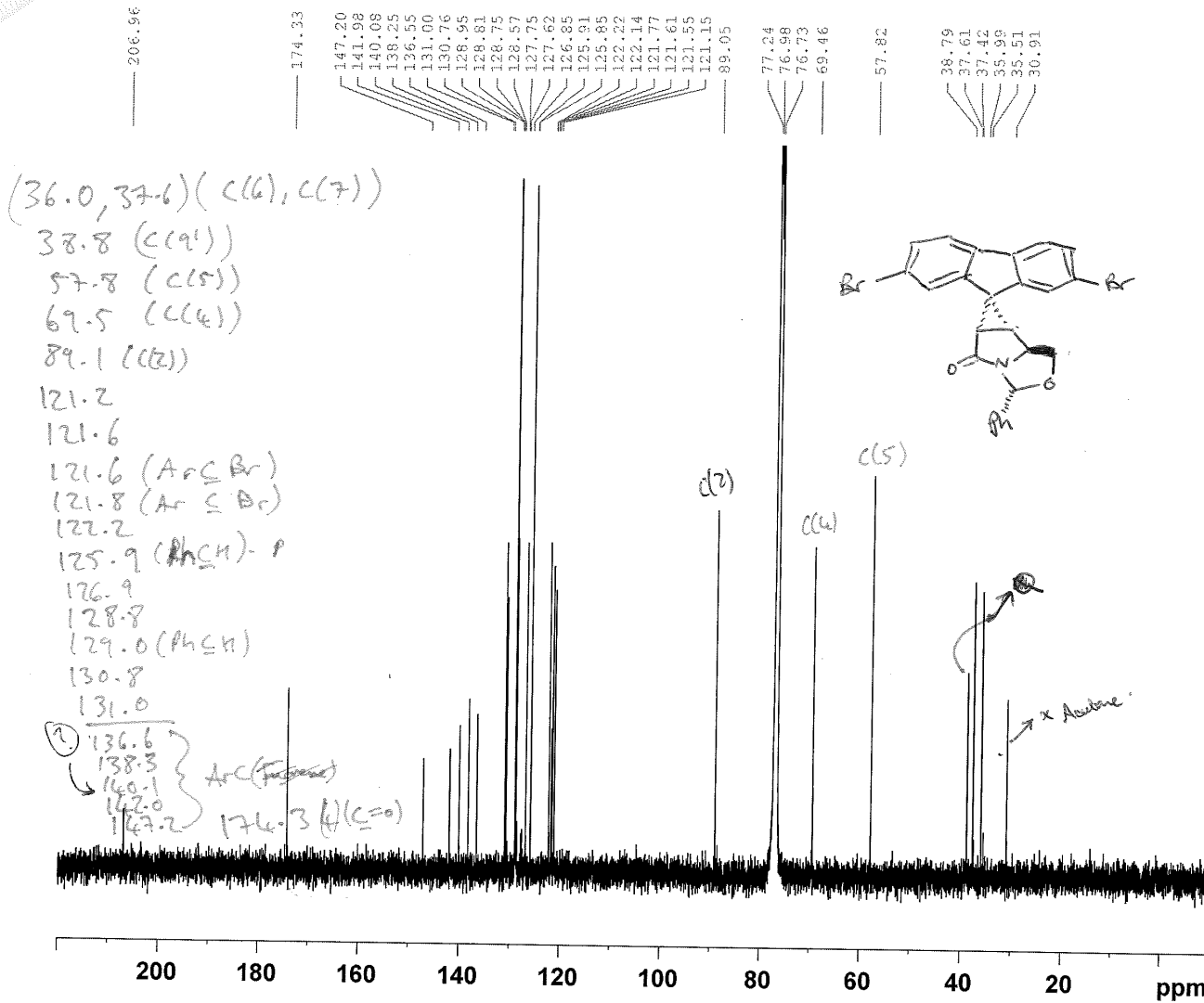
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PROCNO 1

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PROBHD 5 mm CPDUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 4
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 500.3030896 MHz

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SF 500.3000240 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7b



NMR@CHEM.OX

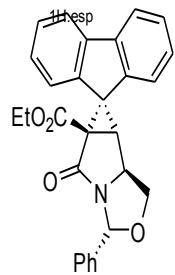
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 3072
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
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 DE 20.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

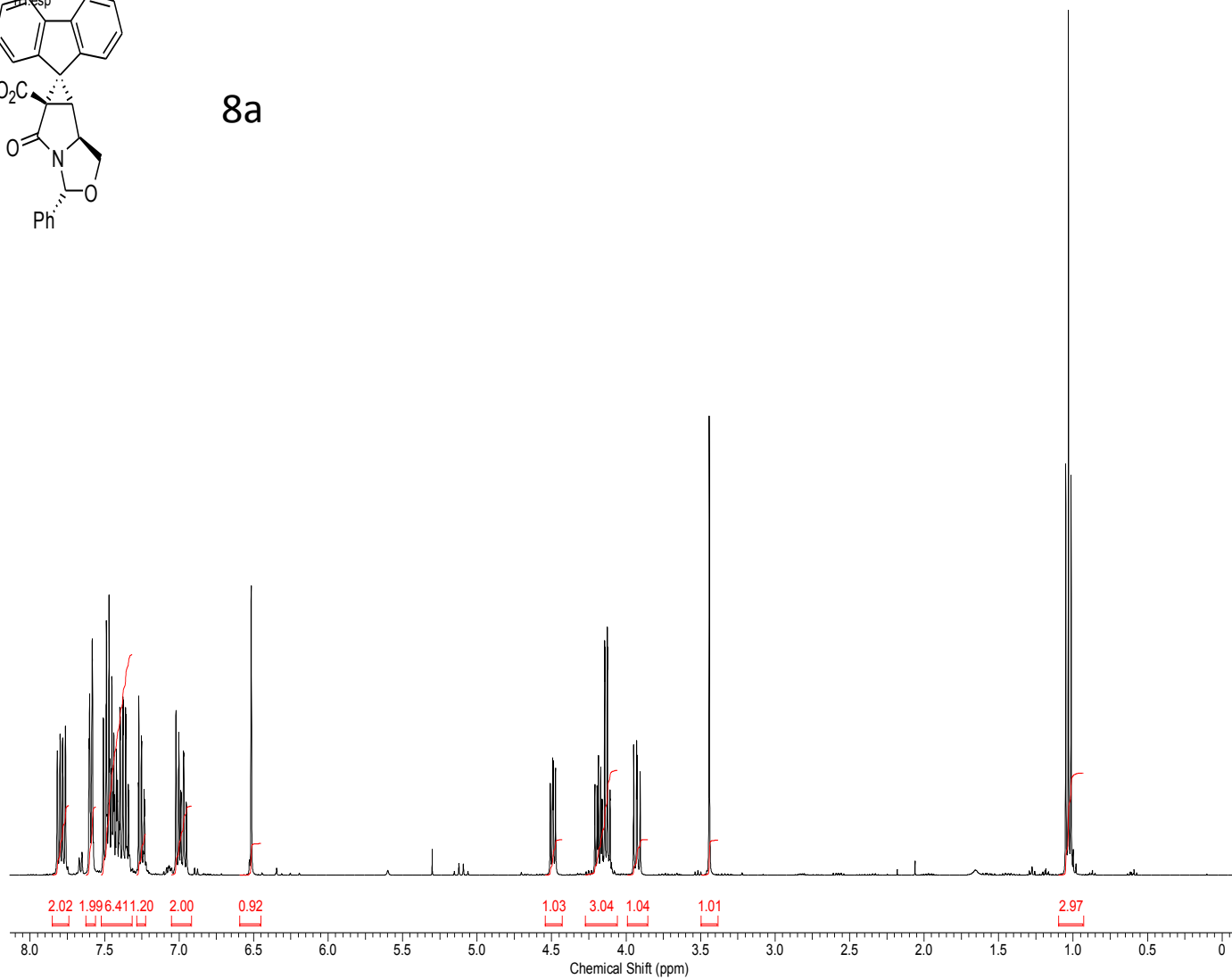
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 PL1 -4.40 dB
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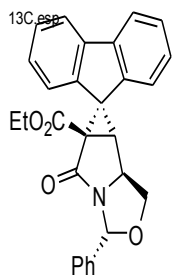
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 PL12 12.40 dB
 PL13 17.00 dB
 PL2 -6.00 dB
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F2 - Processing parameters
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 SF 125.8005438 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

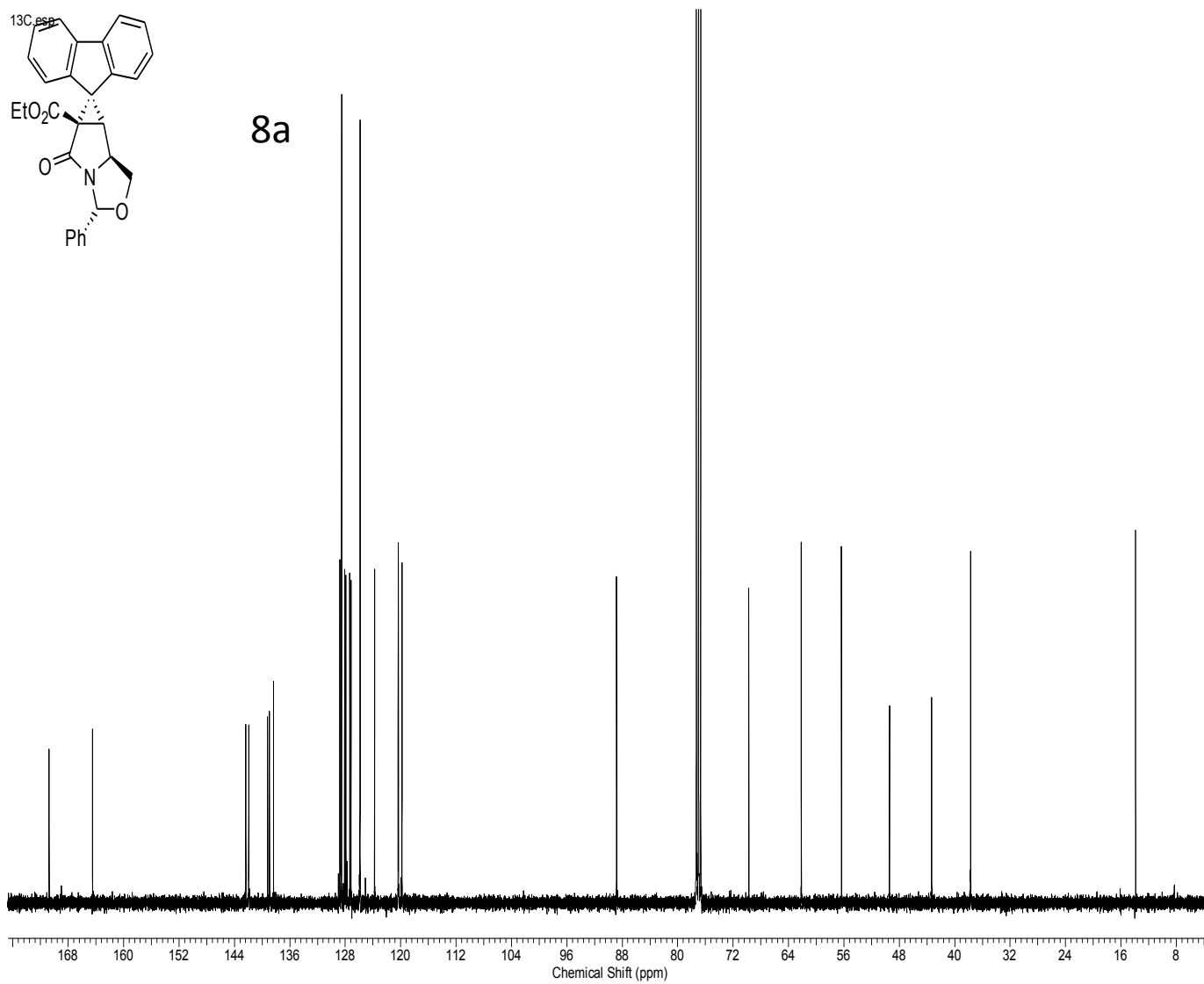


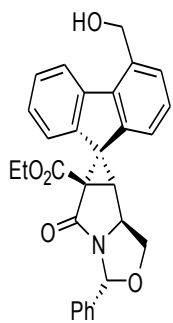
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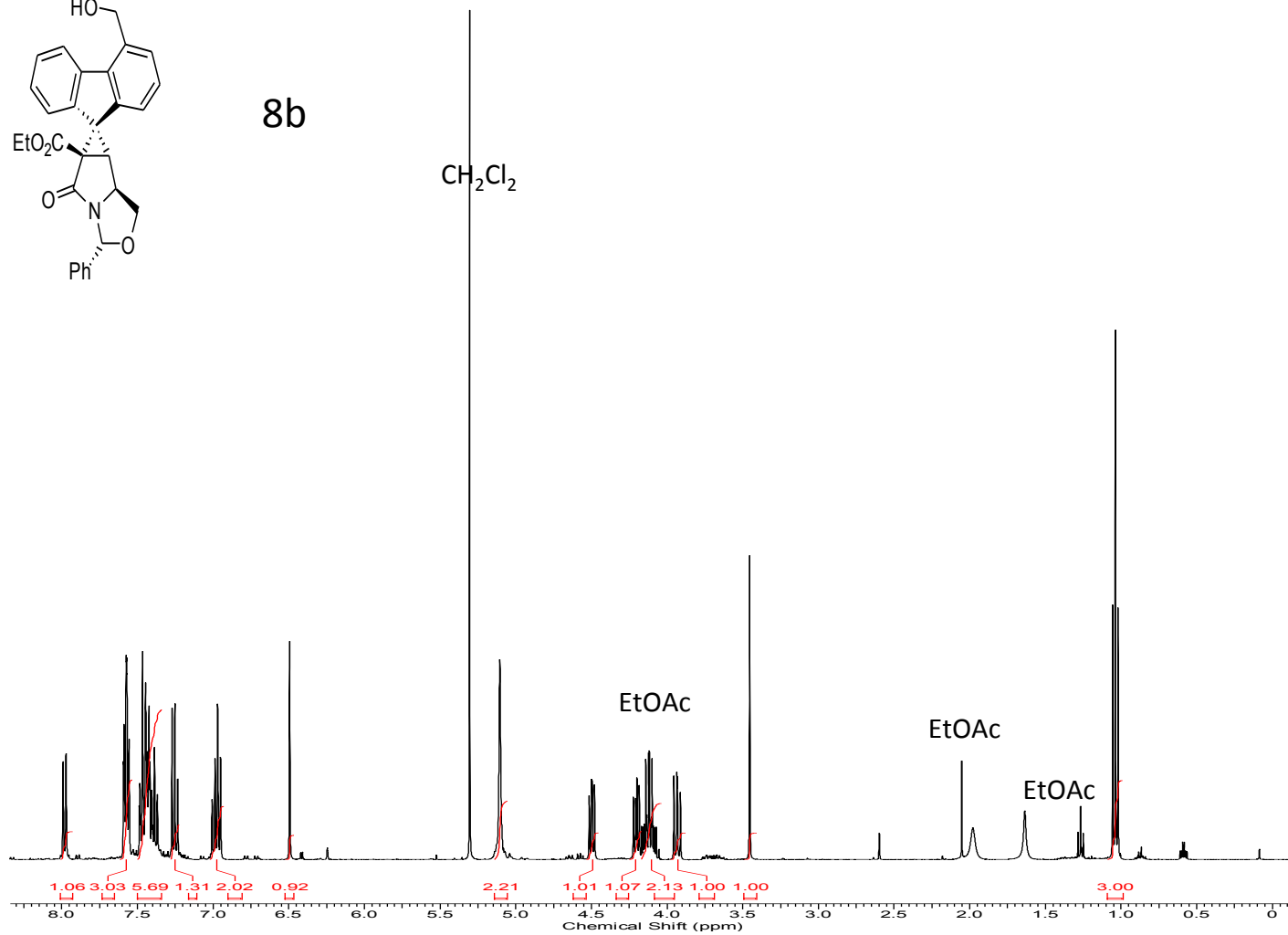


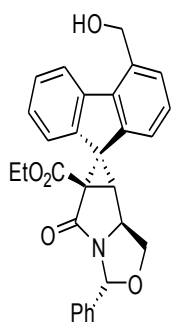
8a



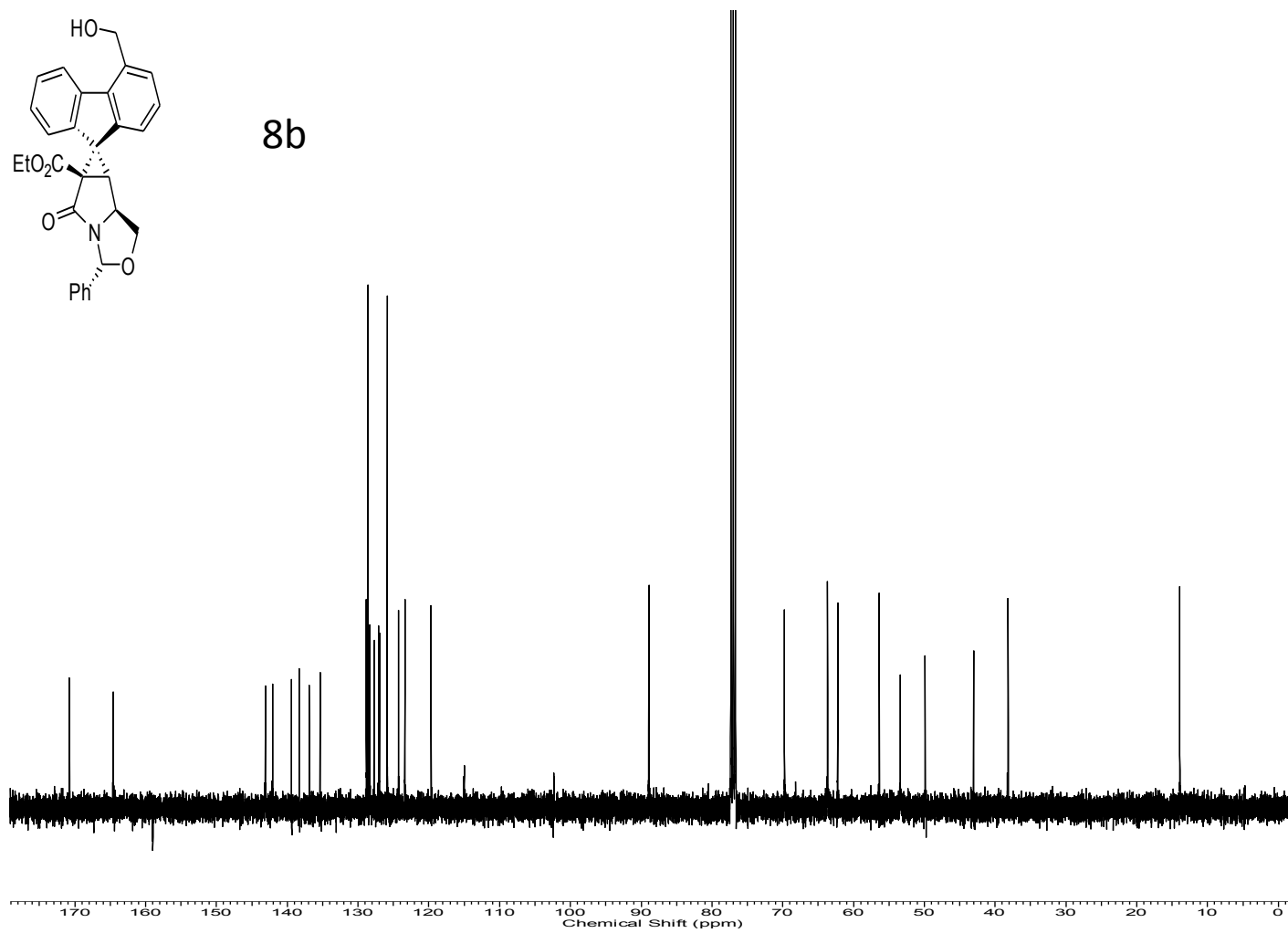


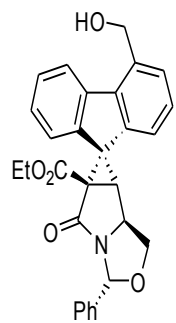
8b



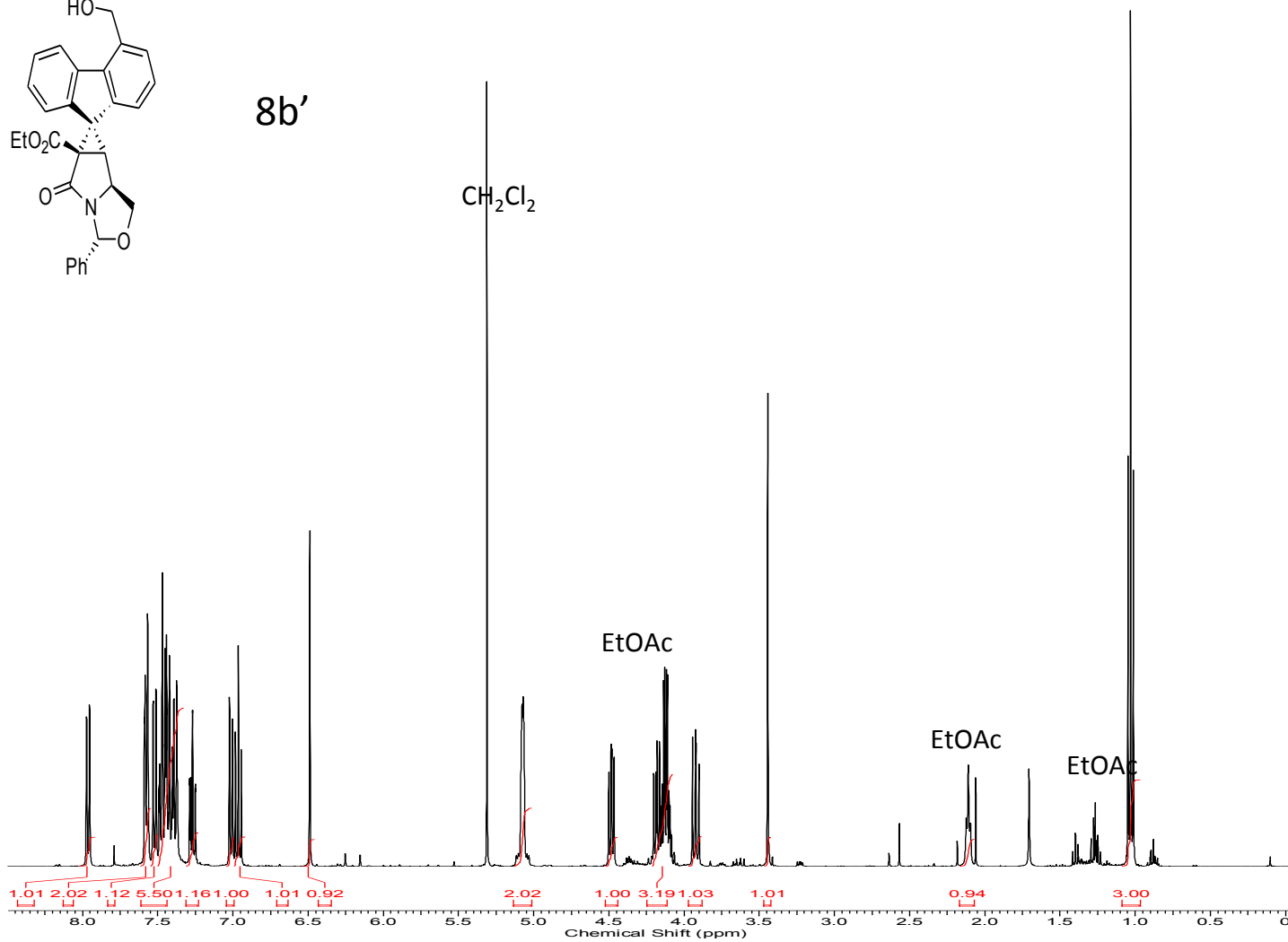


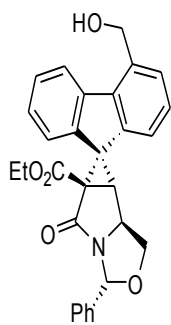
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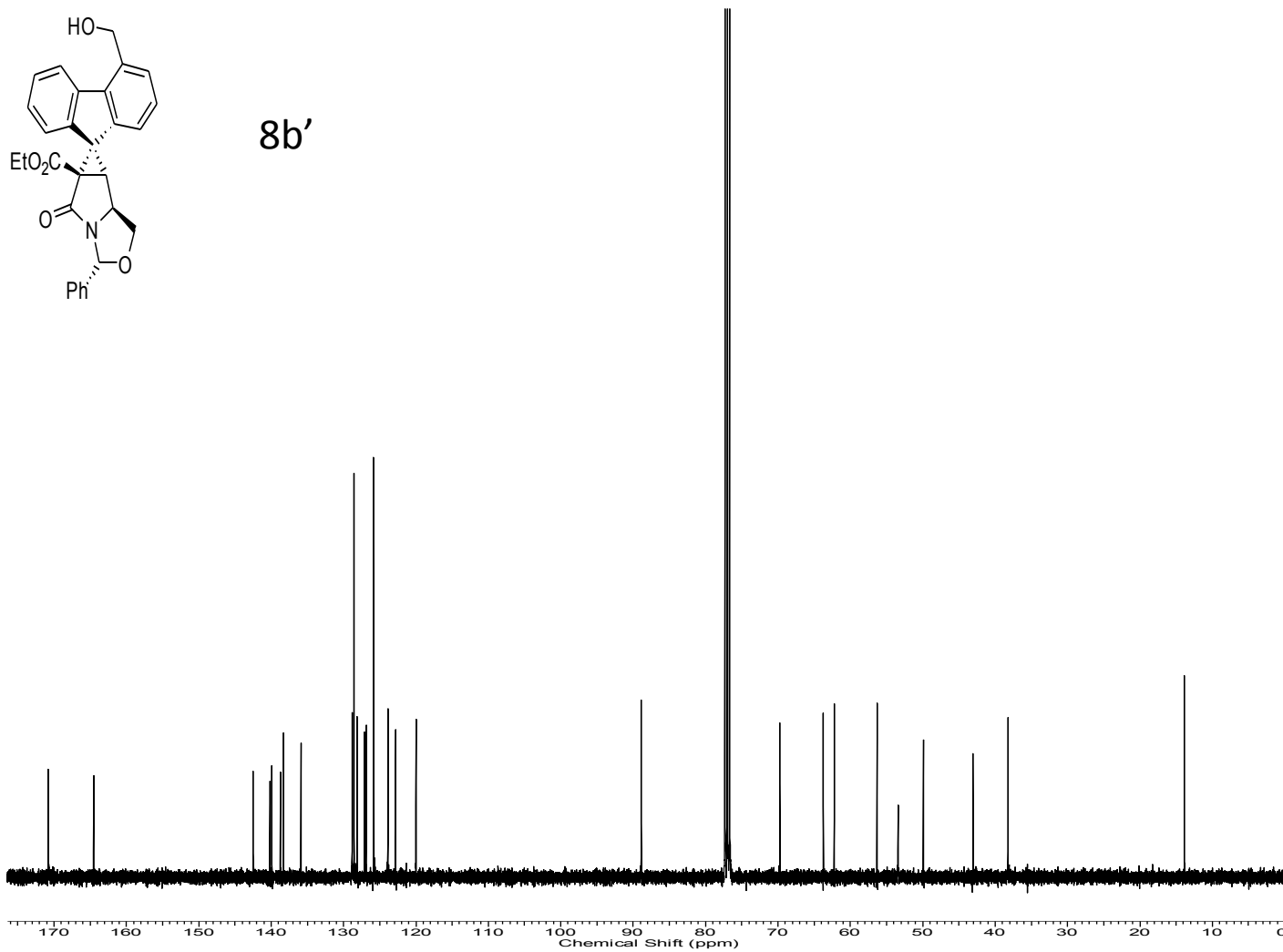


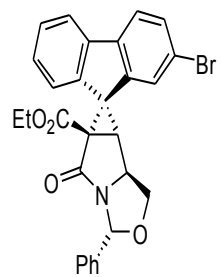
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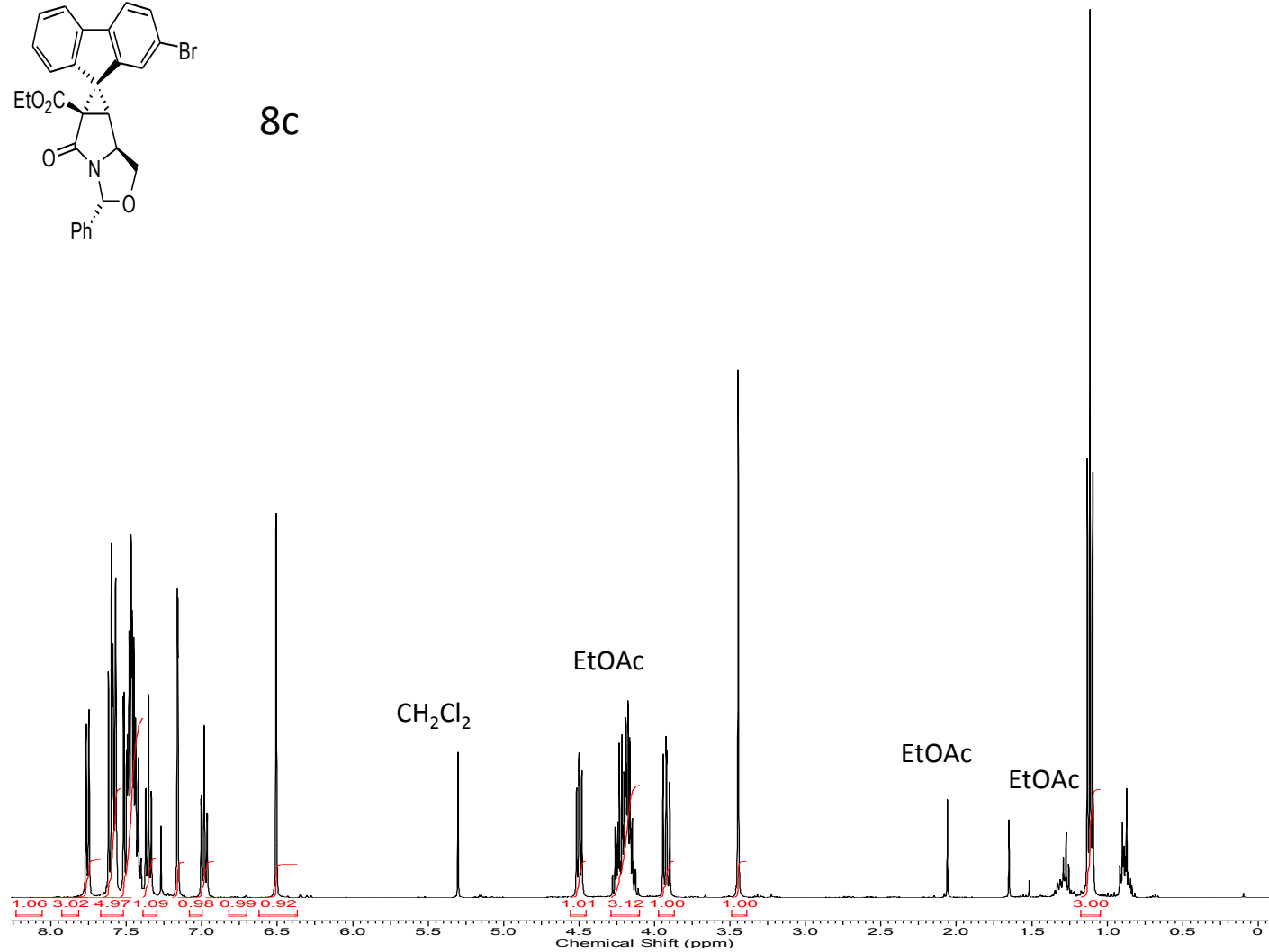


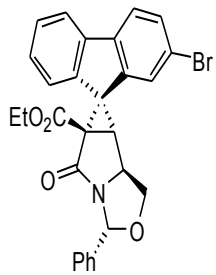
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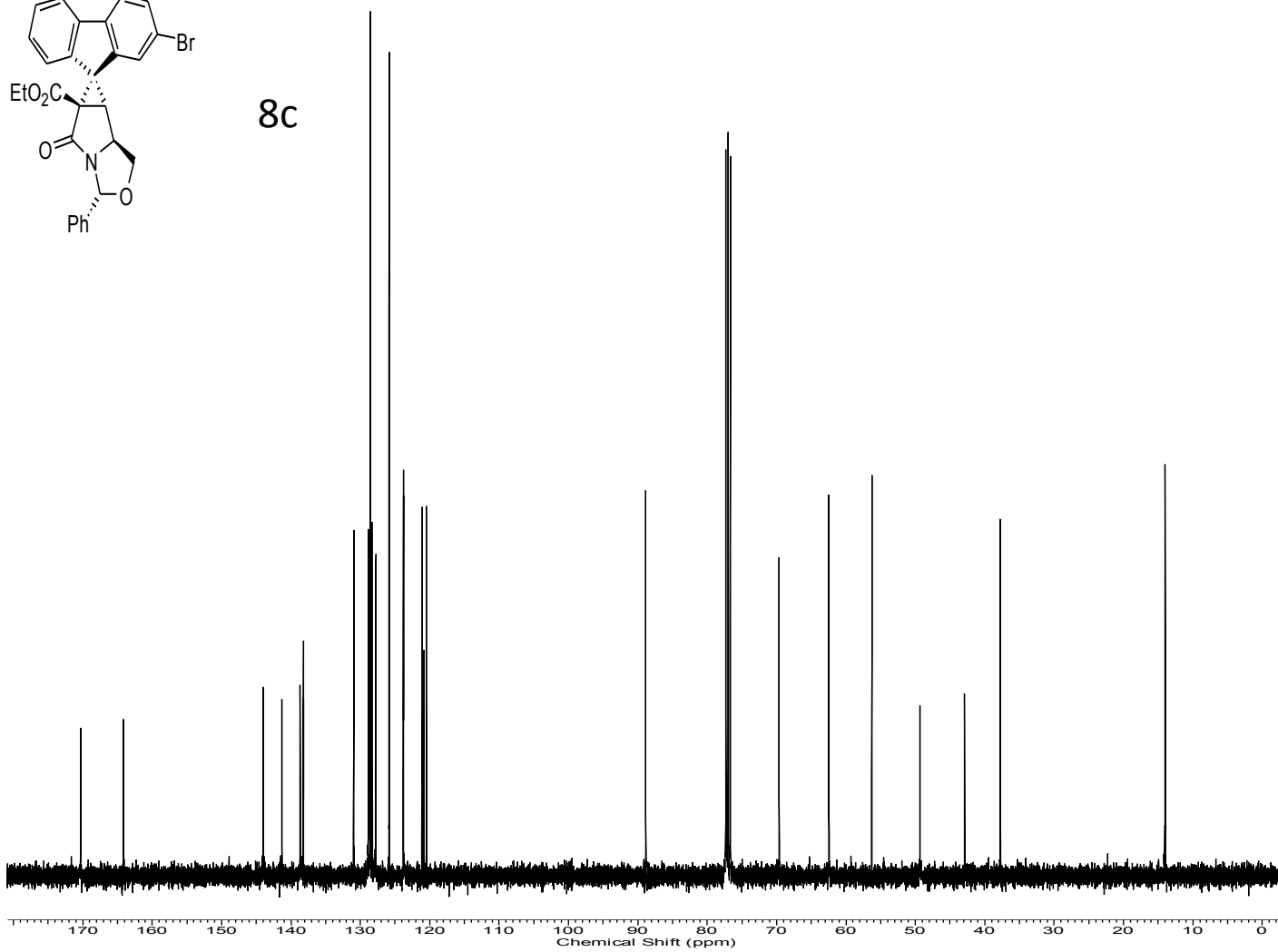


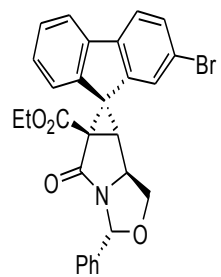
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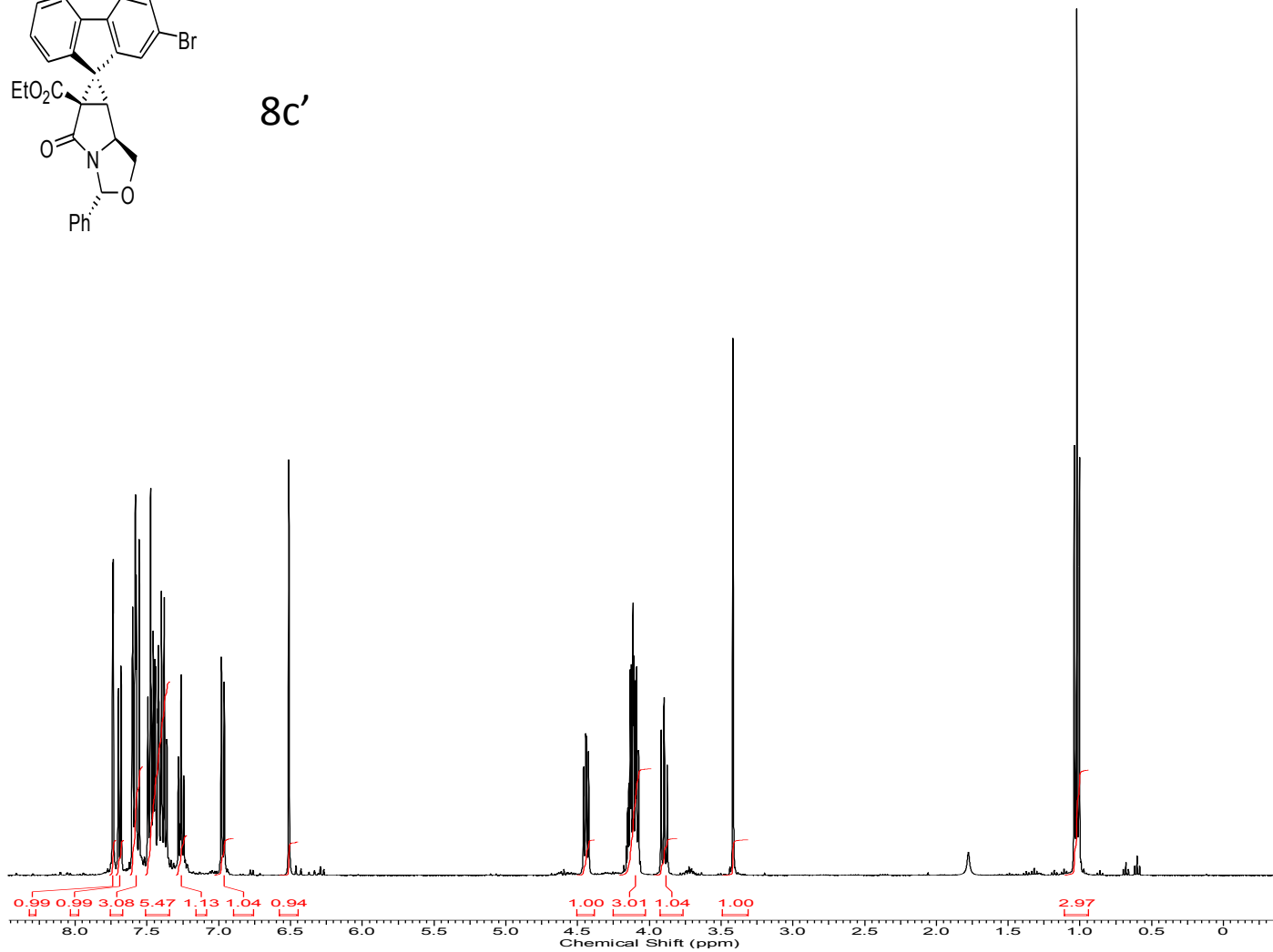


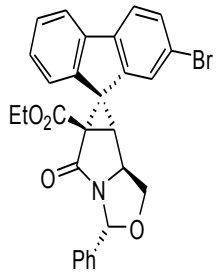
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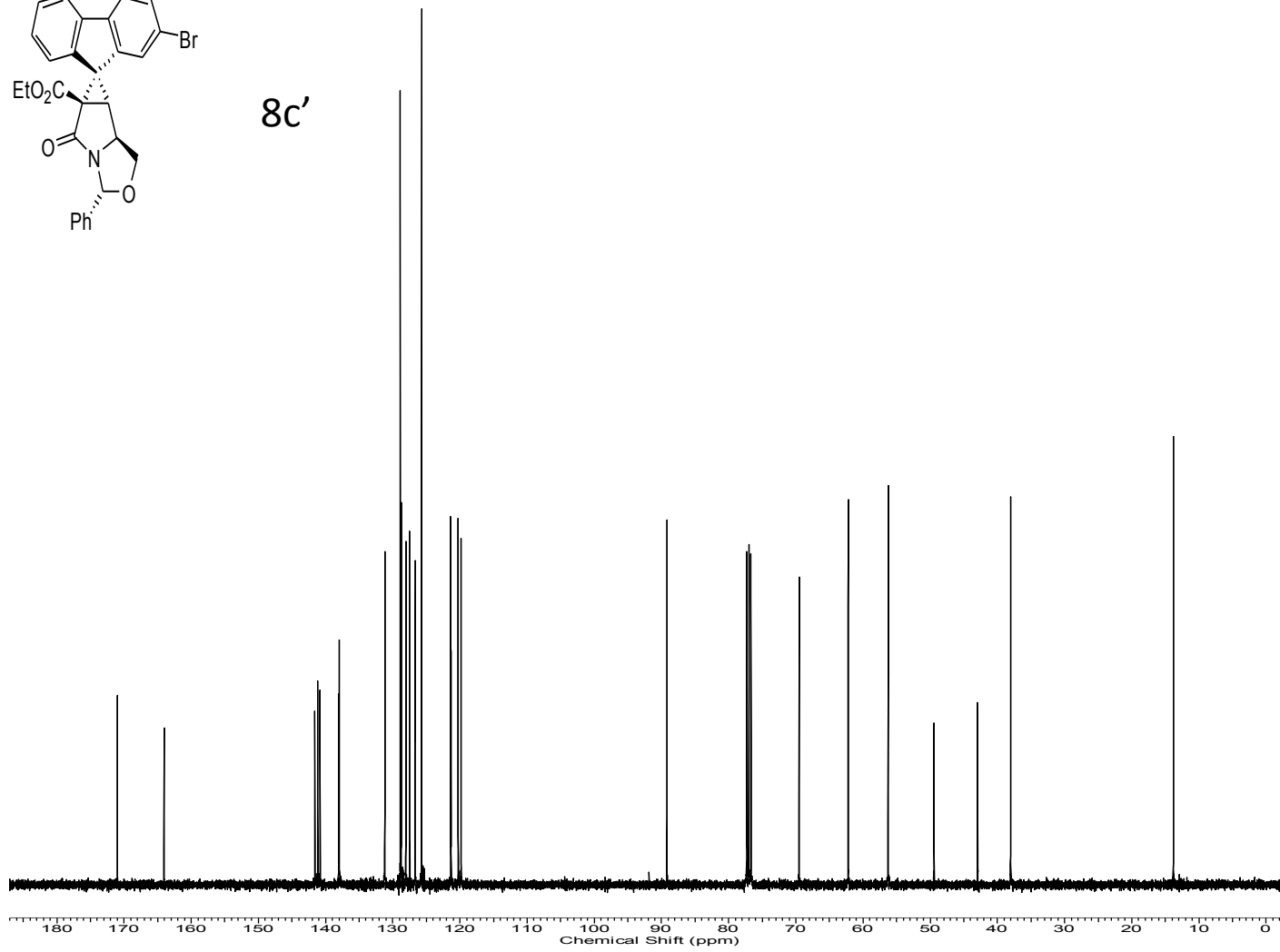


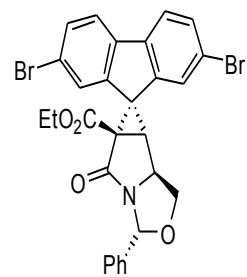
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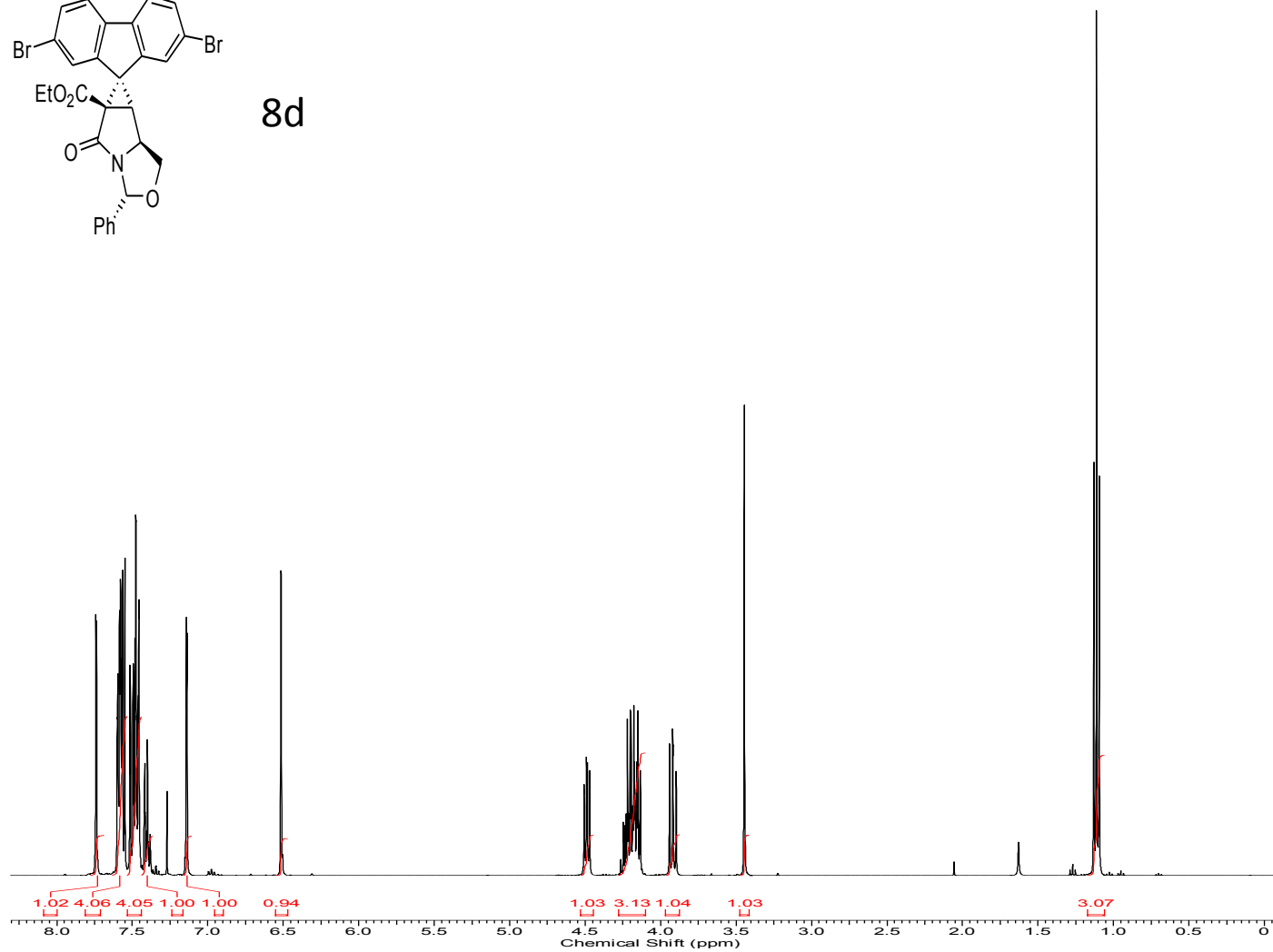


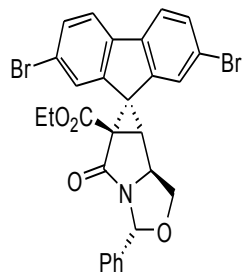
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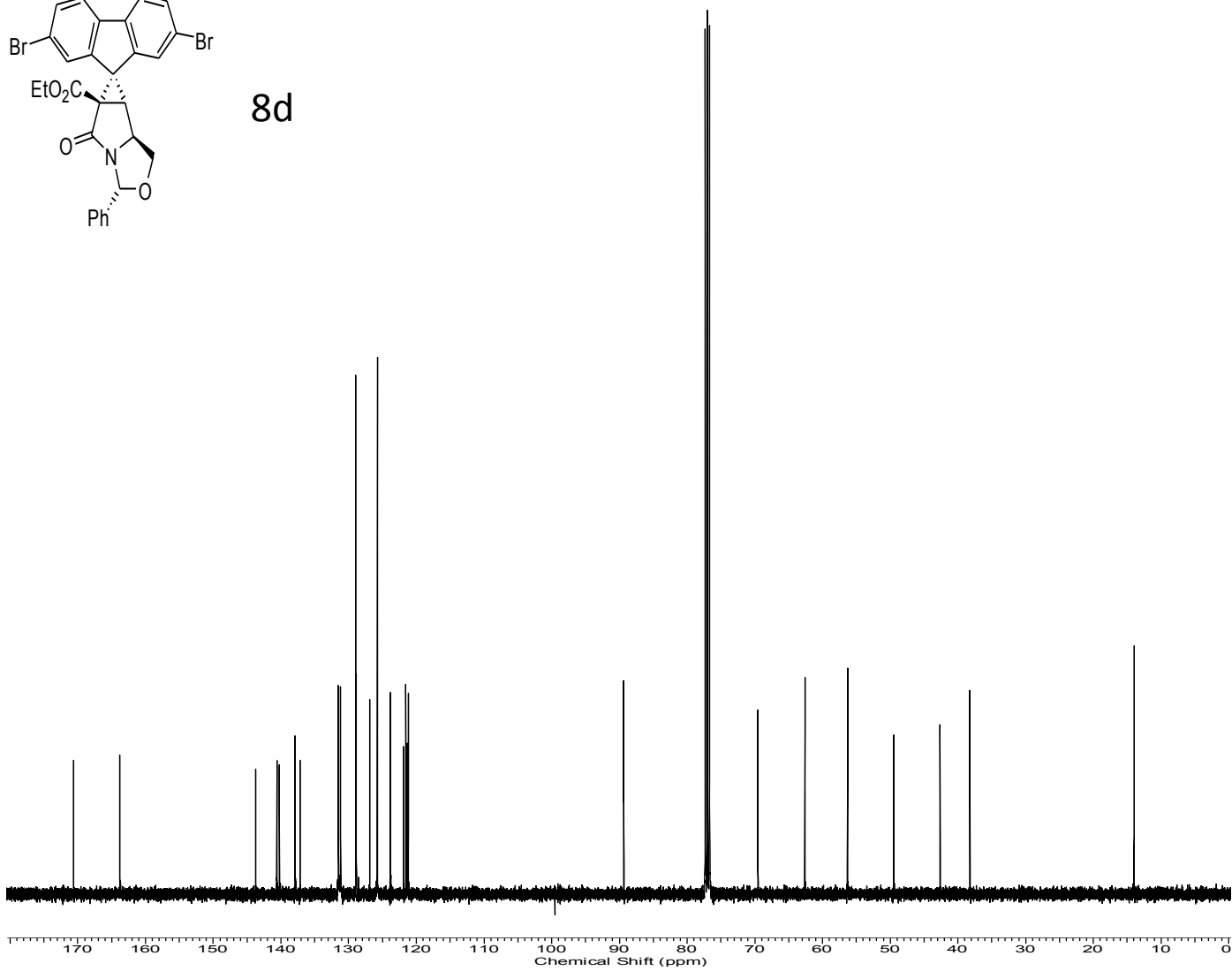


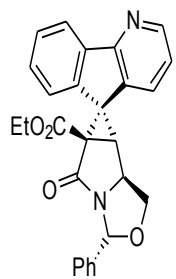
8d





8d

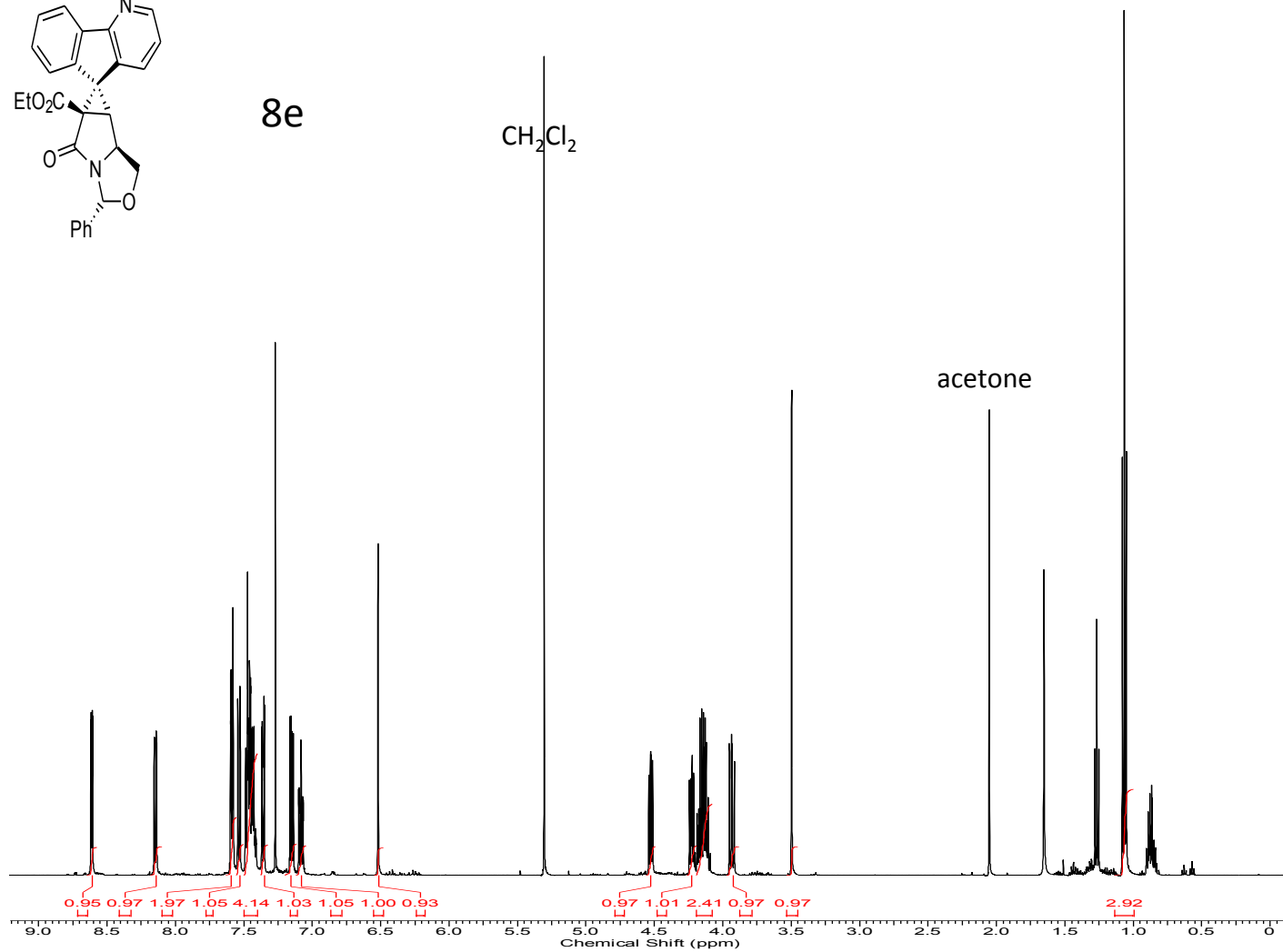


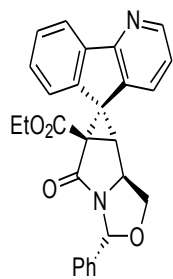


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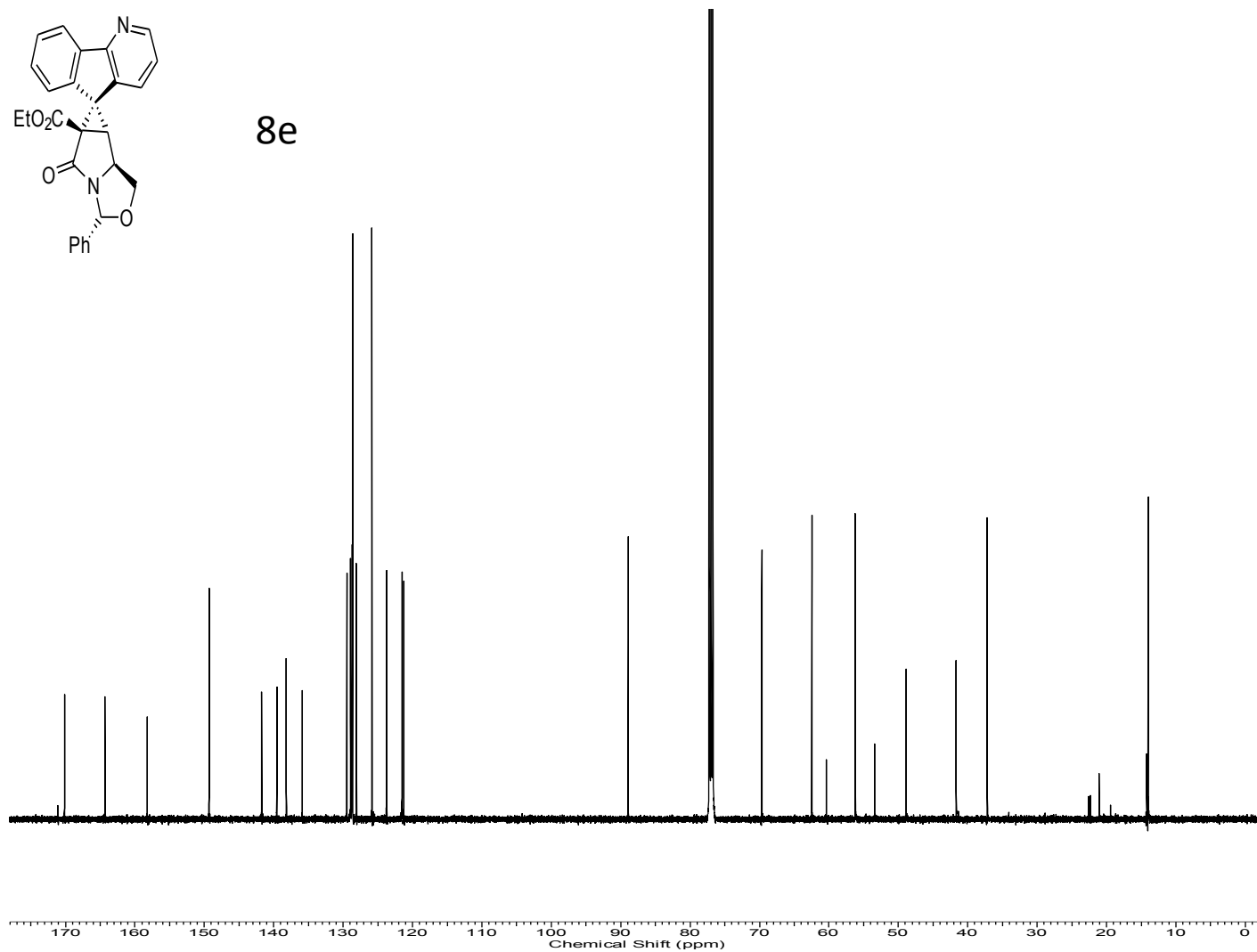
CH₂Cl₂

acetone

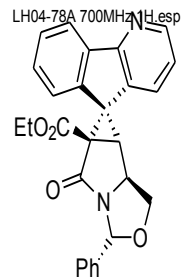




8e

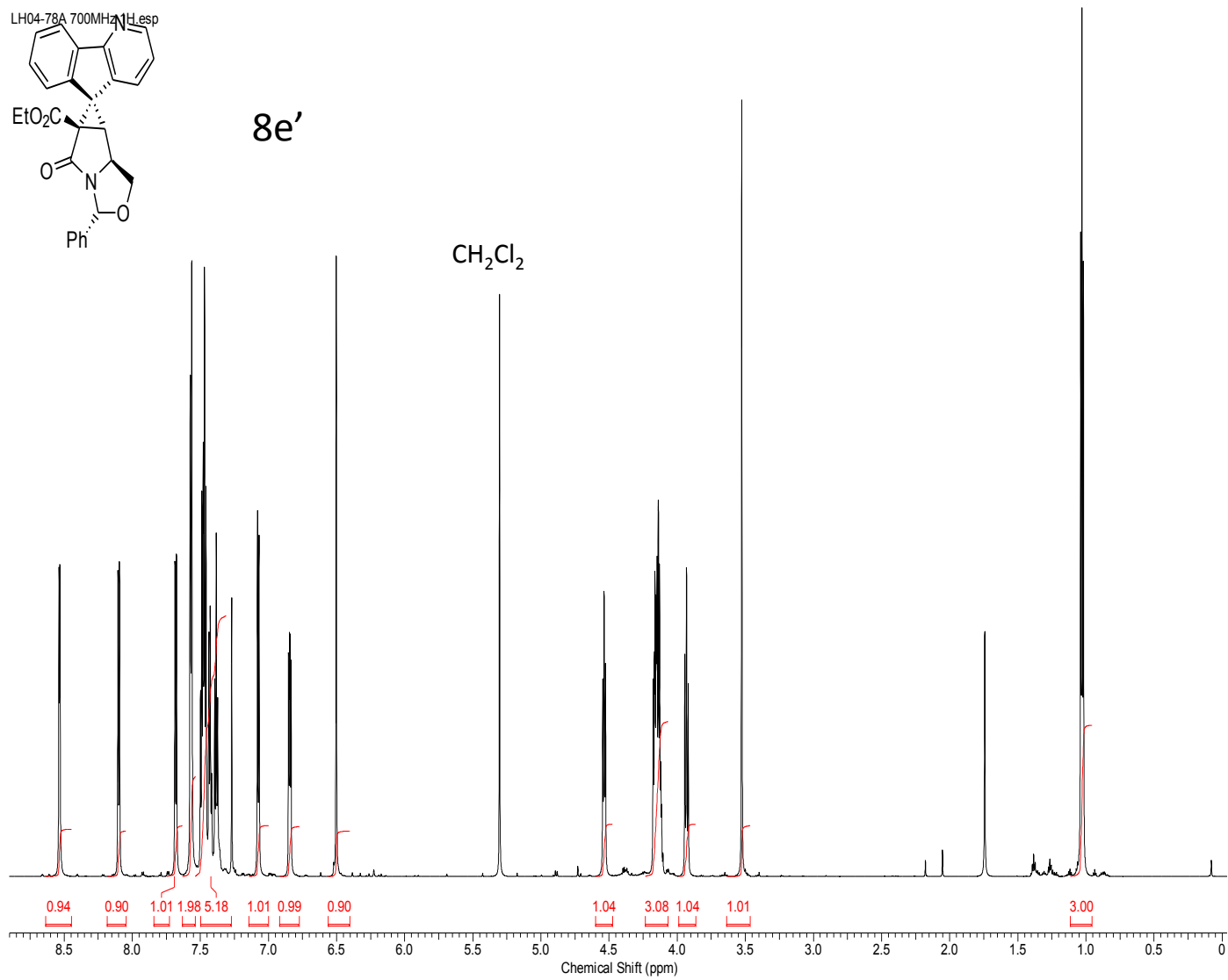


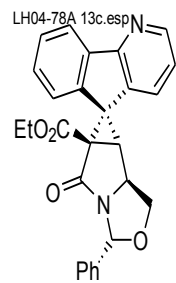
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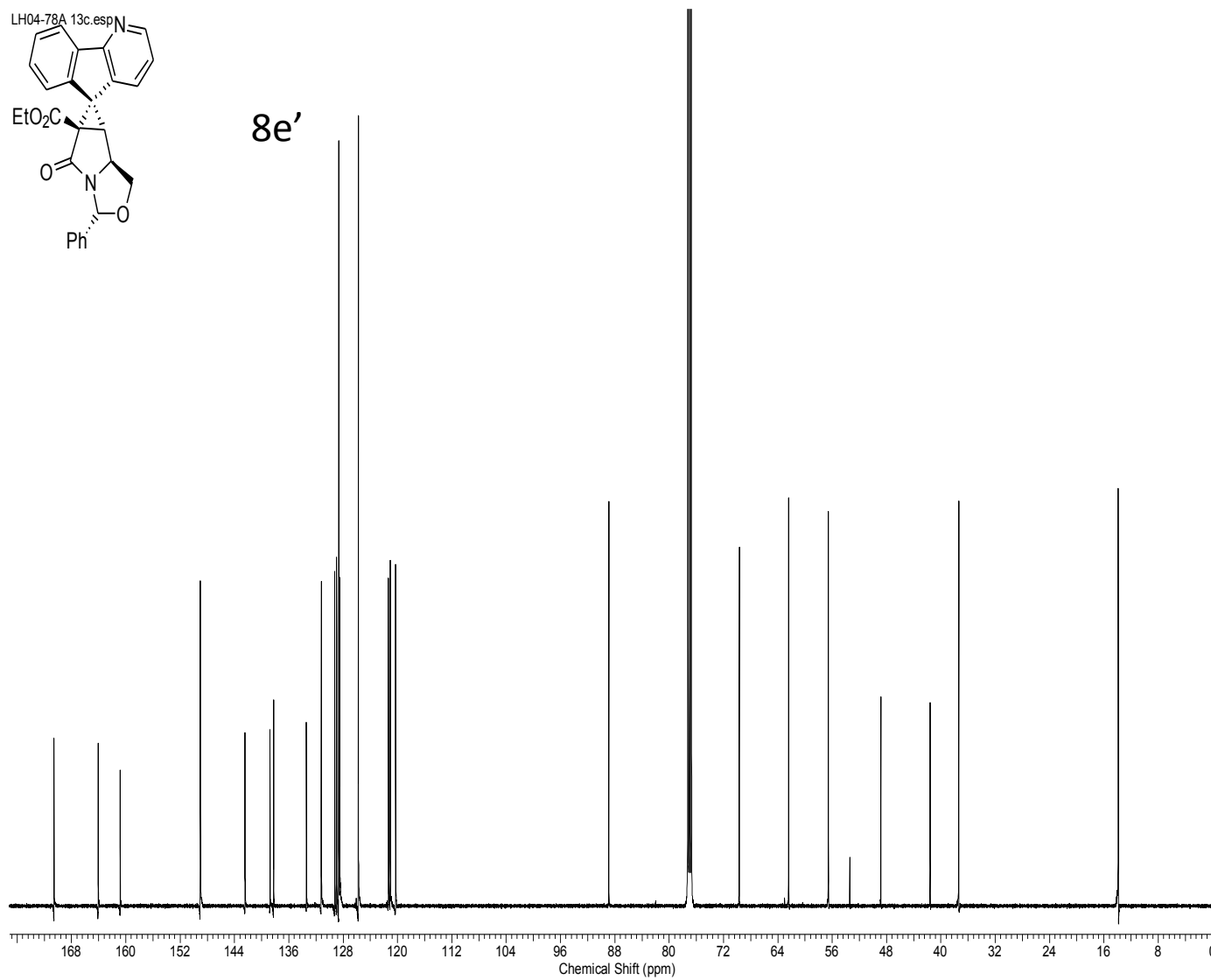
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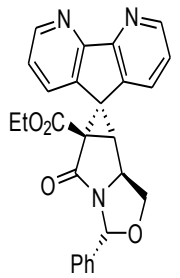
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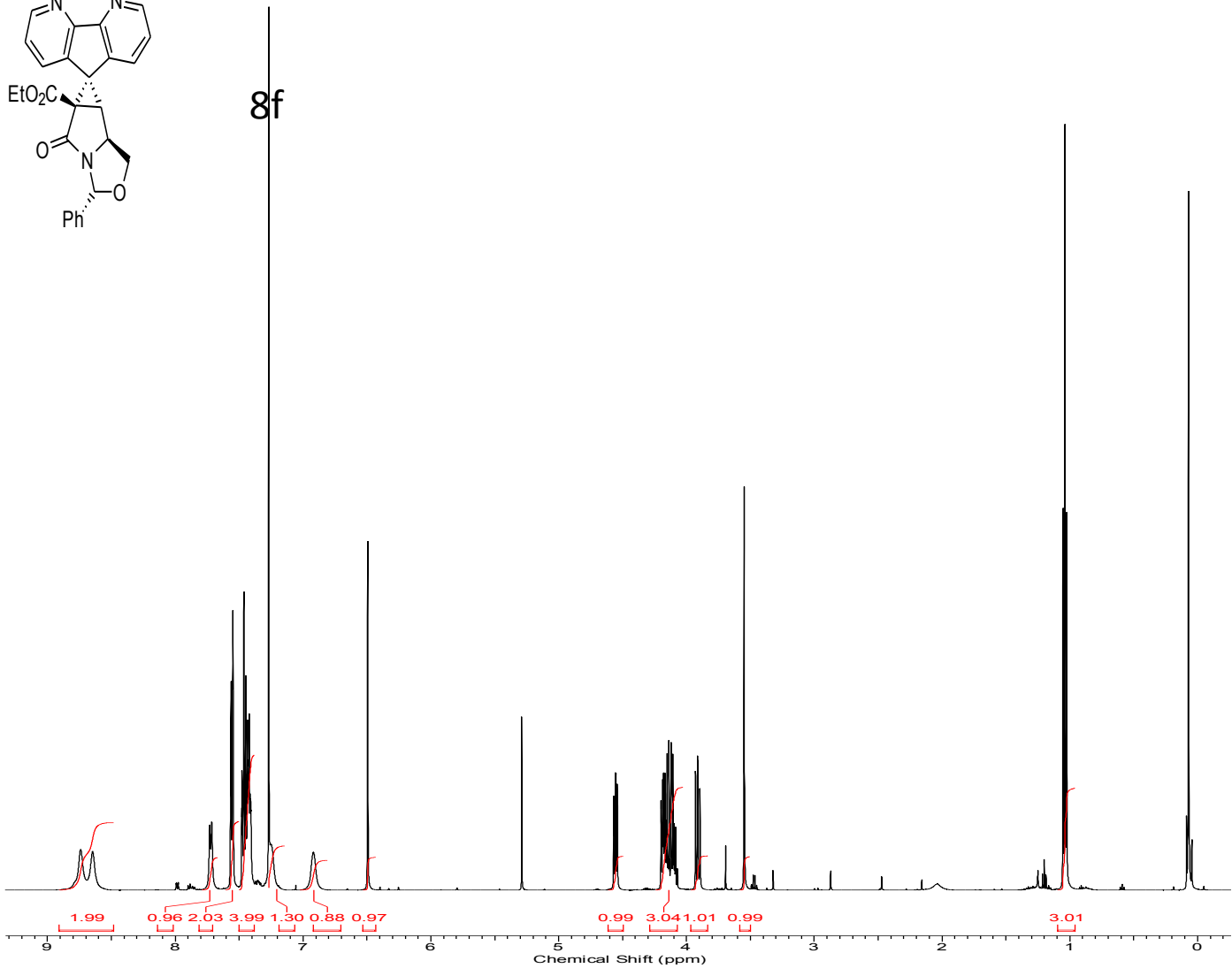


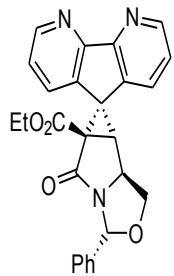
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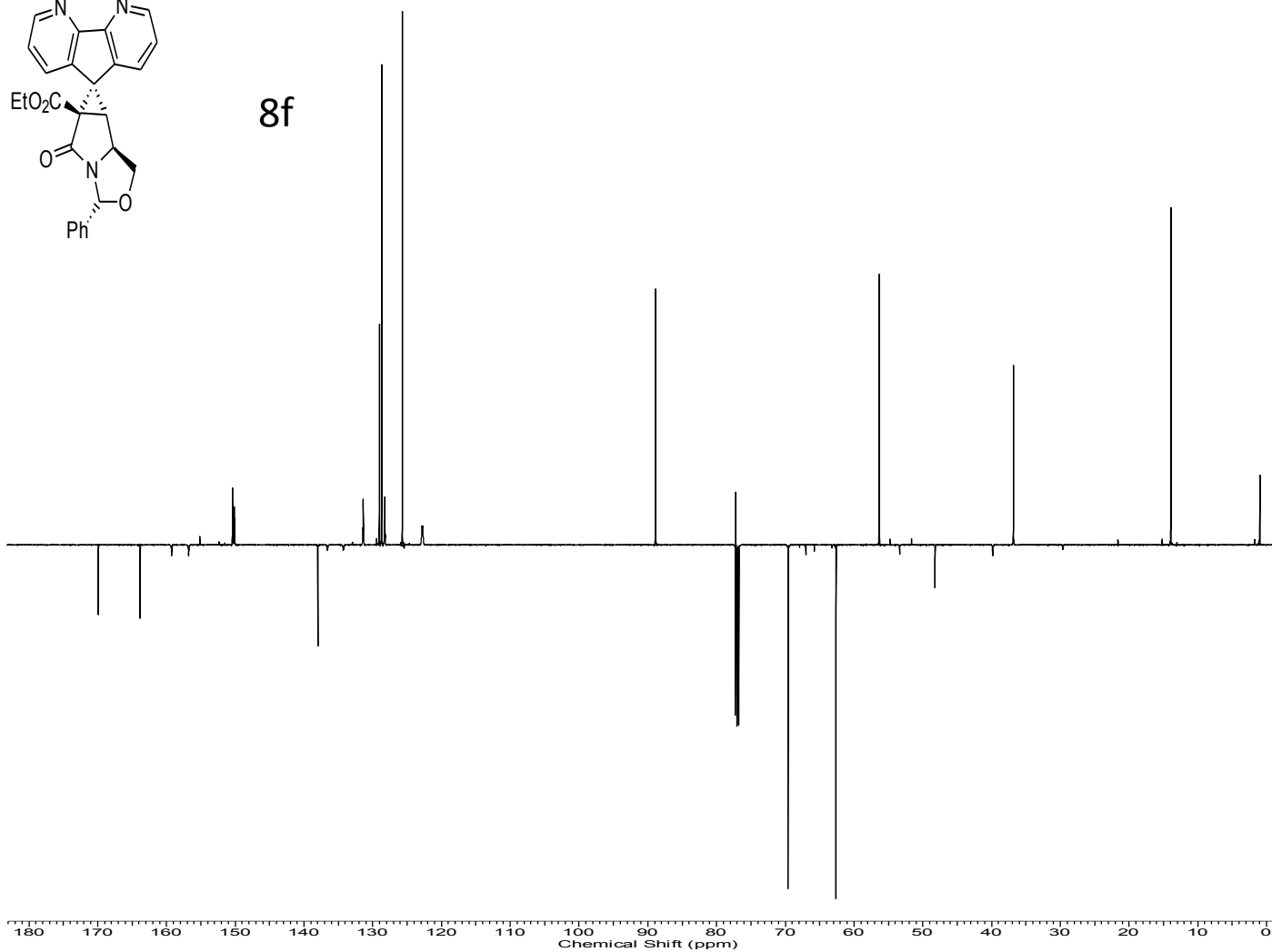


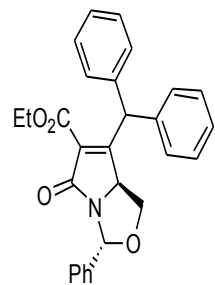
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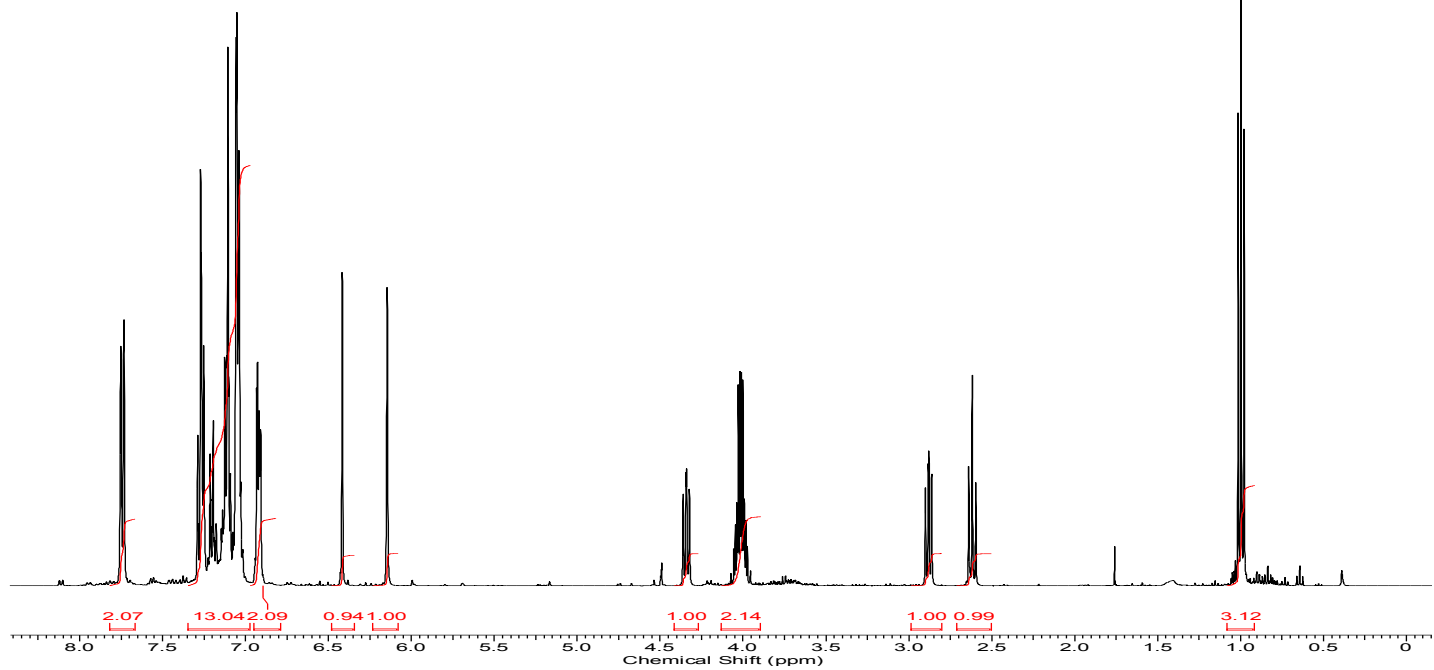


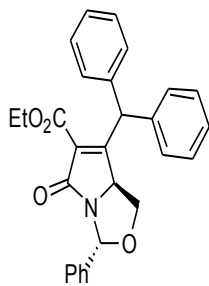
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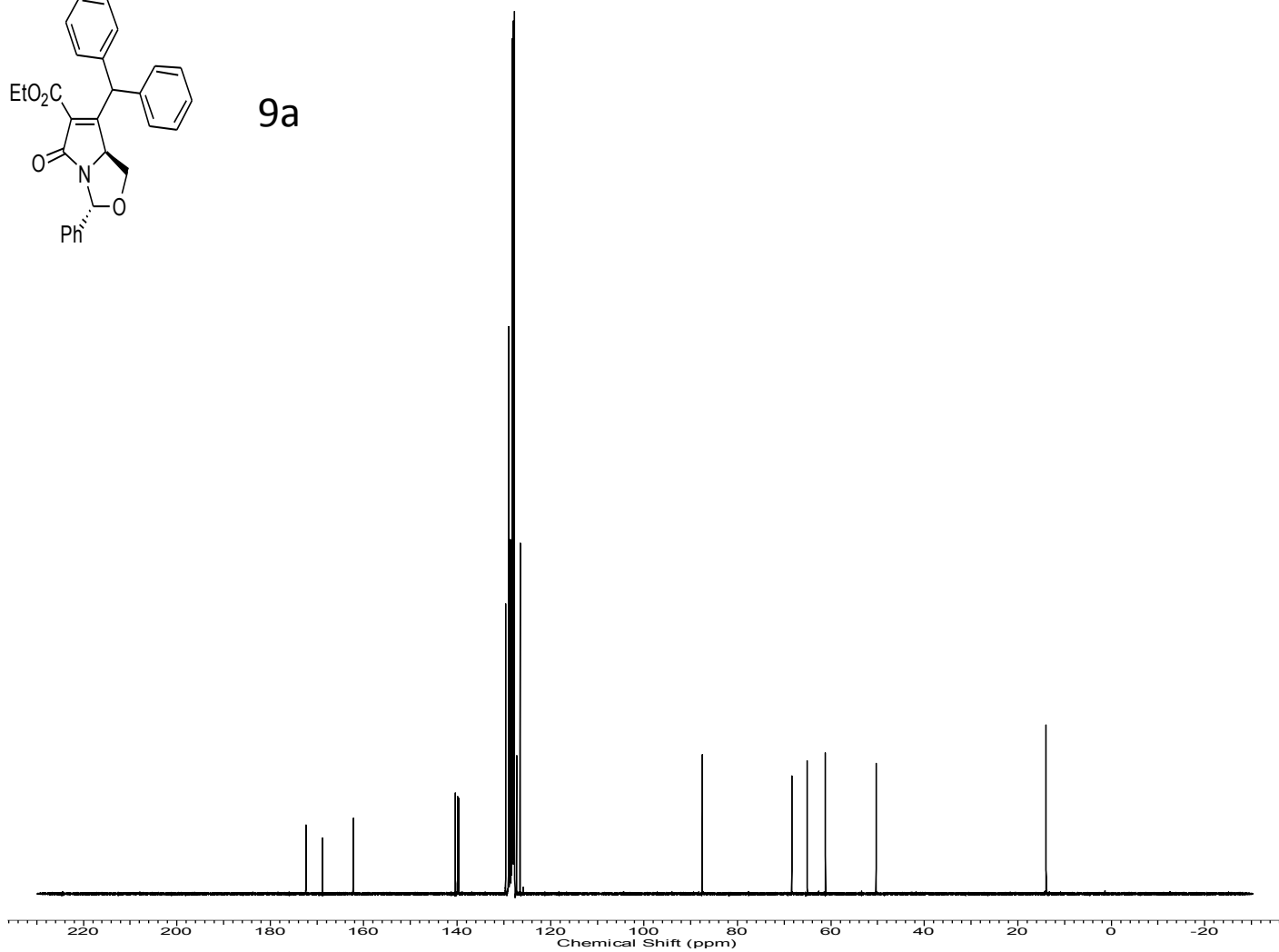


9a

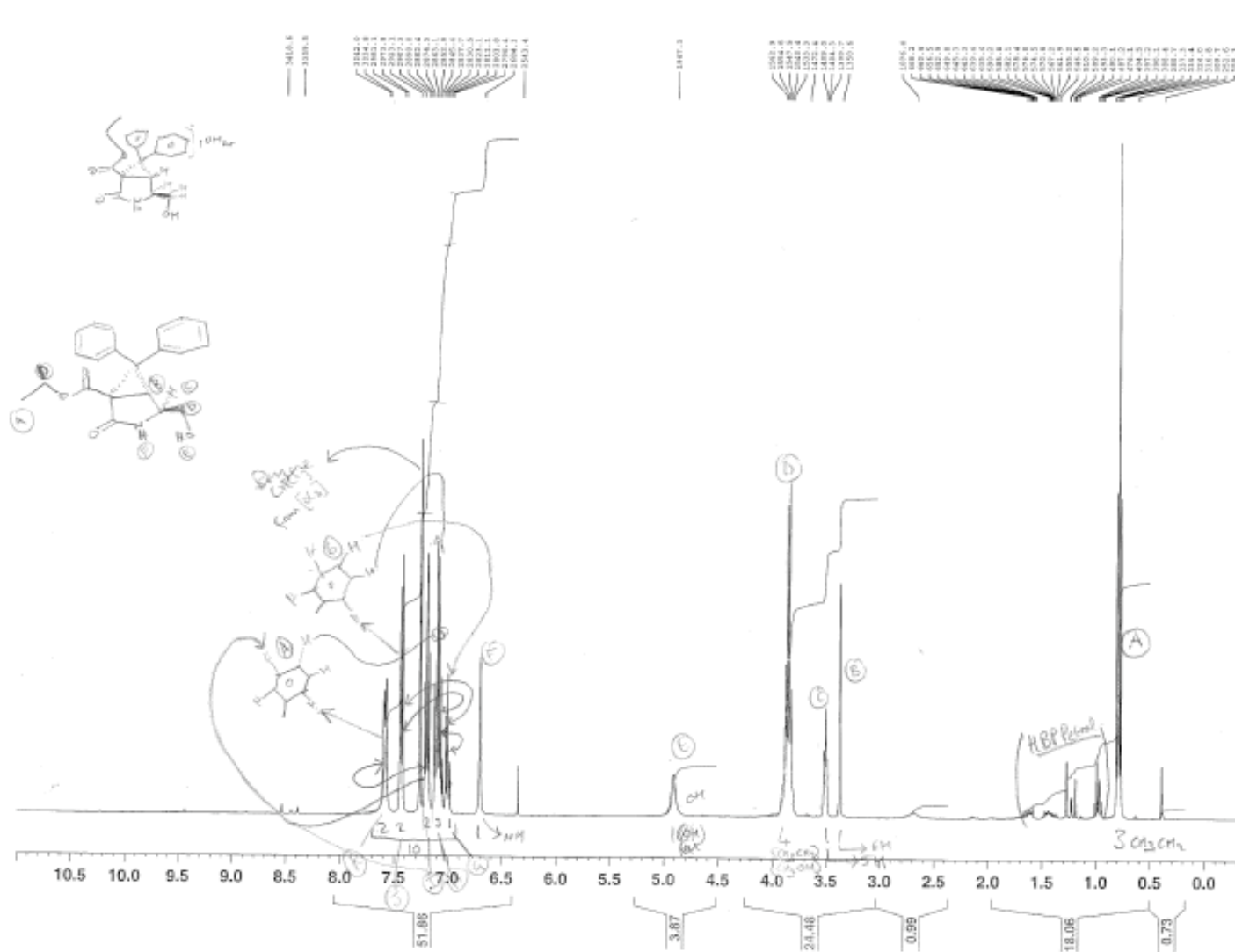




9a



13a



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PROCNO   1

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TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SMB      8278.146 Hz
FIDRES   0.126314 Hz
AQ       3.9584243 sec
RG       50.8
DW       60.400 usec
DE       7.50 usec
TE       295.4 K
D1       1.00000000 sec

----- CHANNEL f1 -----
NUC1     1H
P1       9.00 usec
PL1      0.00 dB
SFO1     400.2024714 MHz

F2 - Processing parameters
SI       32768
SF       400.2000000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

Assignment

A: 3H, t, 0.792 ppm, $J = 7.07$ Hz

B: 1H, s, 3.38 ppm

C: 1H, t, 3.51 ppm, $J = 6.61$

D: 4H, m = q (3.858, $J = 7.07$)
 ↳ 3.81-3.93

E: 1H, br s, 4.92 ppm (OH)

F: 1H, s, 6.71 ppm

G: 1H, m, 7.006 ppm (6.18-7.03)

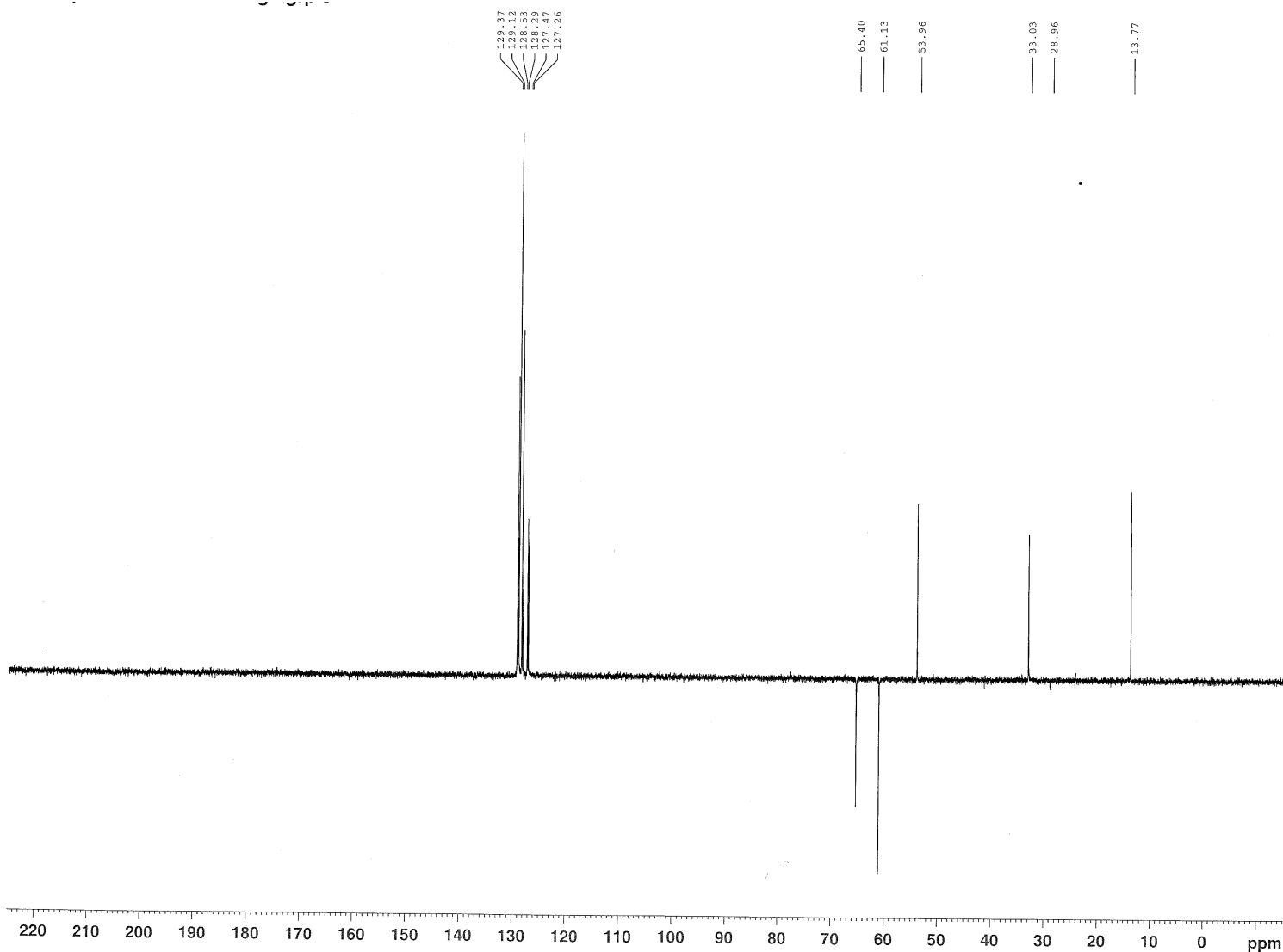
H: 3H, m, 7.05-7.14

I: 2H, t, 7.20; $J = 7.77$

J: 2H, d, 7.44; $J = 7.33$

K: 2H, d, 7.59; $J = 7.2$

13a



Current Data Parameters
NAME Apr18-2006-8
EXPNO 3
PROCNO 1

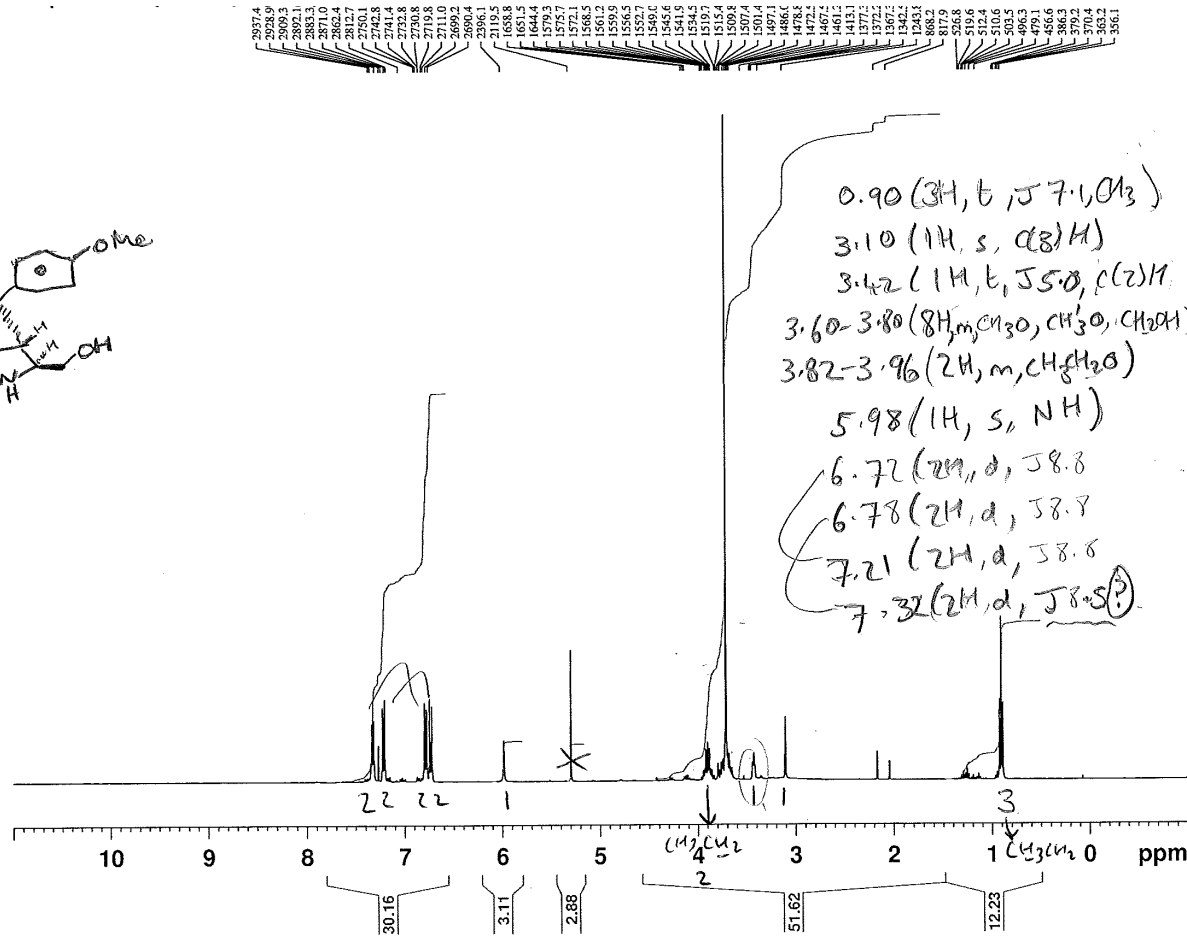
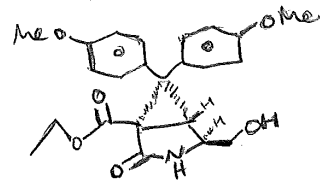
F2 - Acquisition Parameters
Date_ 20060418
Time 12.59
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG dept135
TD 65536
SOLVENT C6D6
NS 256
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 32768
DW 19.100 usec
DE 7.50 usec
TE 295.5 K
CNST2 145.0000000
d1 2.0000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001210 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

=====
CHANNEL f1
NUC1 13C
P1 9.50 usec
p2 19.00 usec
PL1 0.00 dB
SFO1 100.6403931 MHz

=====
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 9.00 usec
P4 18.00 usec
PCPD2 80.00 usec
PL2 0.00 dB
PL12 19.00 dB
SFO2 400.2016008 MHz

F2 - Processing parameters
SI 32768
SF 100.6303515 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

13b



0.90 (3H, t, J 7.1, CH₃)
 3.10 (1H, s, C(8)H)
 3.42 (1H, t, J 5.0, C(2)H)
 3.60-3.80 (8H, m, CH₃O, CH₂O, CH₂OH)
 3.82-3.96 (2H, m, CH₂OH)
 5.98 (1H, s, NH)
 6.72 (2H, d, J 8.8)
 6.78 (2H, d, J 8.8)
 7.21 (2H, d, J 8.8)
 7.32 (2H, d, J 8.5)

```

Current Data Parameters
NAME      Mar26-2008-28
EXPNO    1
PROCNO    1

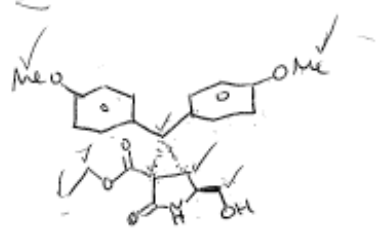
F2 - Acquisition Parameters
Date_    20080327
Time     2.30
INSTRUM  av400
PROBHD   5 mm QNP 1H/13
PULPROG  zg60
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ       3.9584243 sec
RG       90.5
DW       60.400 usec
DE       7.50 usec
TE       300.0 K
D1       1.00000000 sec

===== CHANNEL f1 =====
NUC1     1H
P1       9.00 usec
PL1      0.00 dB
SFO1    400.2024714 MHz

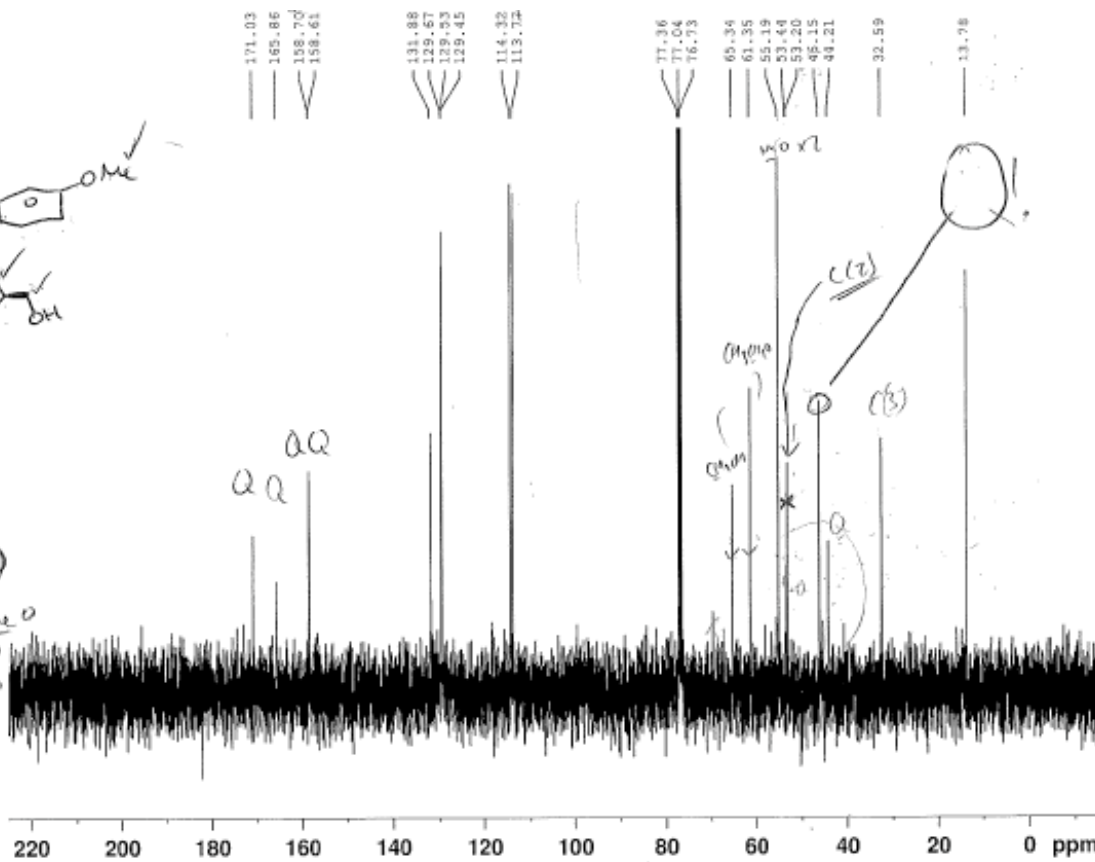
F2 - Processing parameters
SI       32768
SF       400.2000028 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

13b

171.03



13.7 CH₃
 32.6 (C3)
 46.2 (O)
 46.1 - O
 53.2 (C2)
 [53.2]??
 55.2 2x MeO
 61.3 CH₂CH₂O
 65.3 CH₂OH



Current Data Parameters
 NAME Mar26-2008-28
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080327
 Time 2.42
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl₃
 NS 256
 DS 4
 SMH 26178.010 Hz
 FIDRES 0.798889 Hz
 AQ 0.6259188 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 d11 0.0300000 sec
 DELTA 0.89999998 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.00 dB
 PL13 25.00 dB
 PL2 0.00 dB
 SFO2 400.2016008 MHz

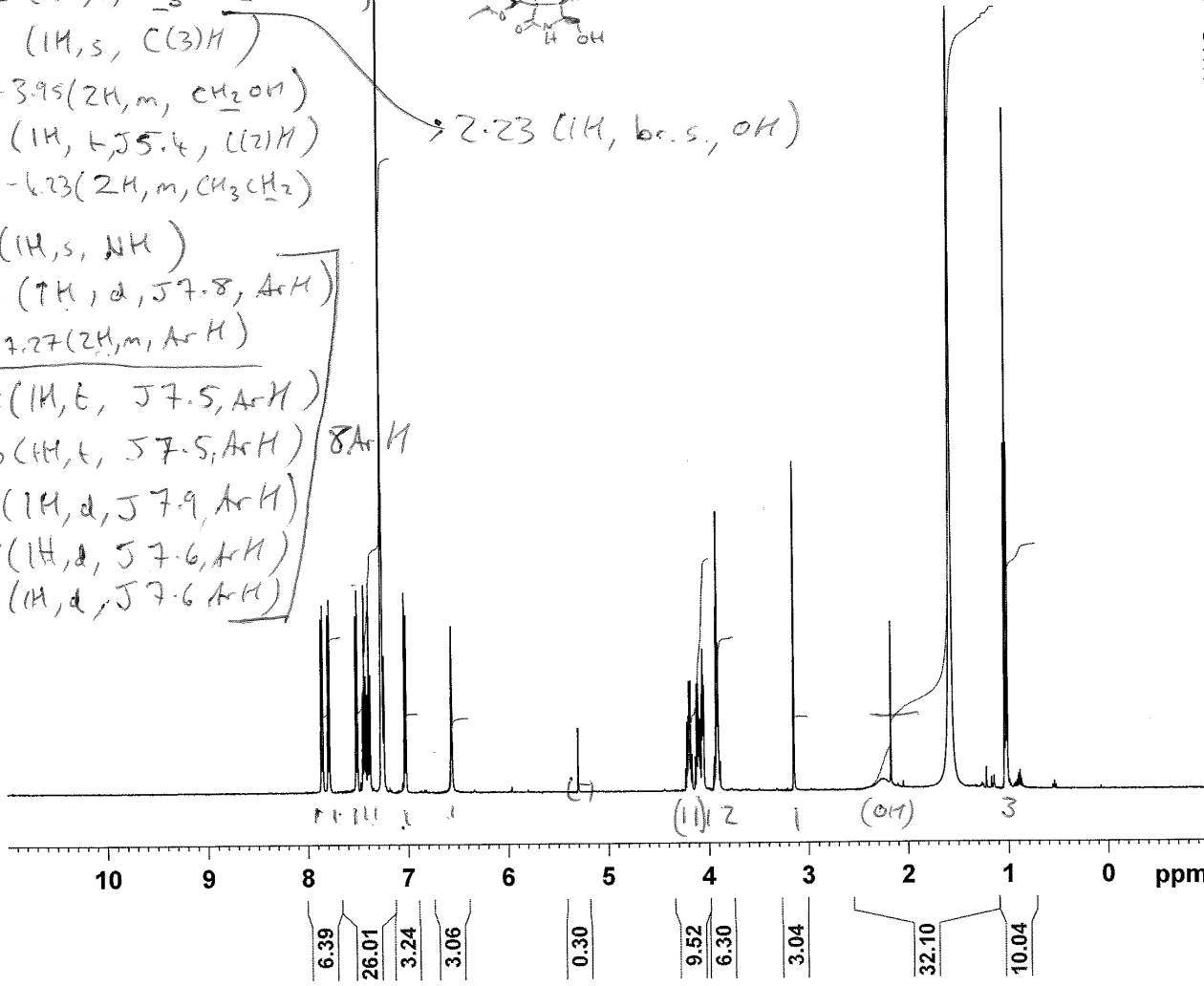
F2 - Processing parameters
 SI 32768
 SF 100.6303718 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

13c



- 1.02 (3H, t, CH₂CH₂[J=7.1])
- 3.14 (1H, s, C(3)H)
- 3.88-3.95 (2H, m, CH₂OH)
- 4.06 (1H, t, J 5.4, C(2)H)
- 4.07-4.23 (2H, m, CH₂CH₂)
- 6.56 (1H, s, NH)
- 7.02 (7H, d, J 7.8, ArH)
- 7.23-7.27 (2H, m, ArH)
- 7.38 (1H, t, J 7.5, ArH)
- 7.43 (1H, t, J 7.5, ArH)
- 7.51 (1H, d, J 7.9, ArH)
- 7.78 (1H, d, J 7.6, ArH)
- 7.85 (1H, d, J 7.6, ArH)

→ 2.23 (1H, br. s., OH)



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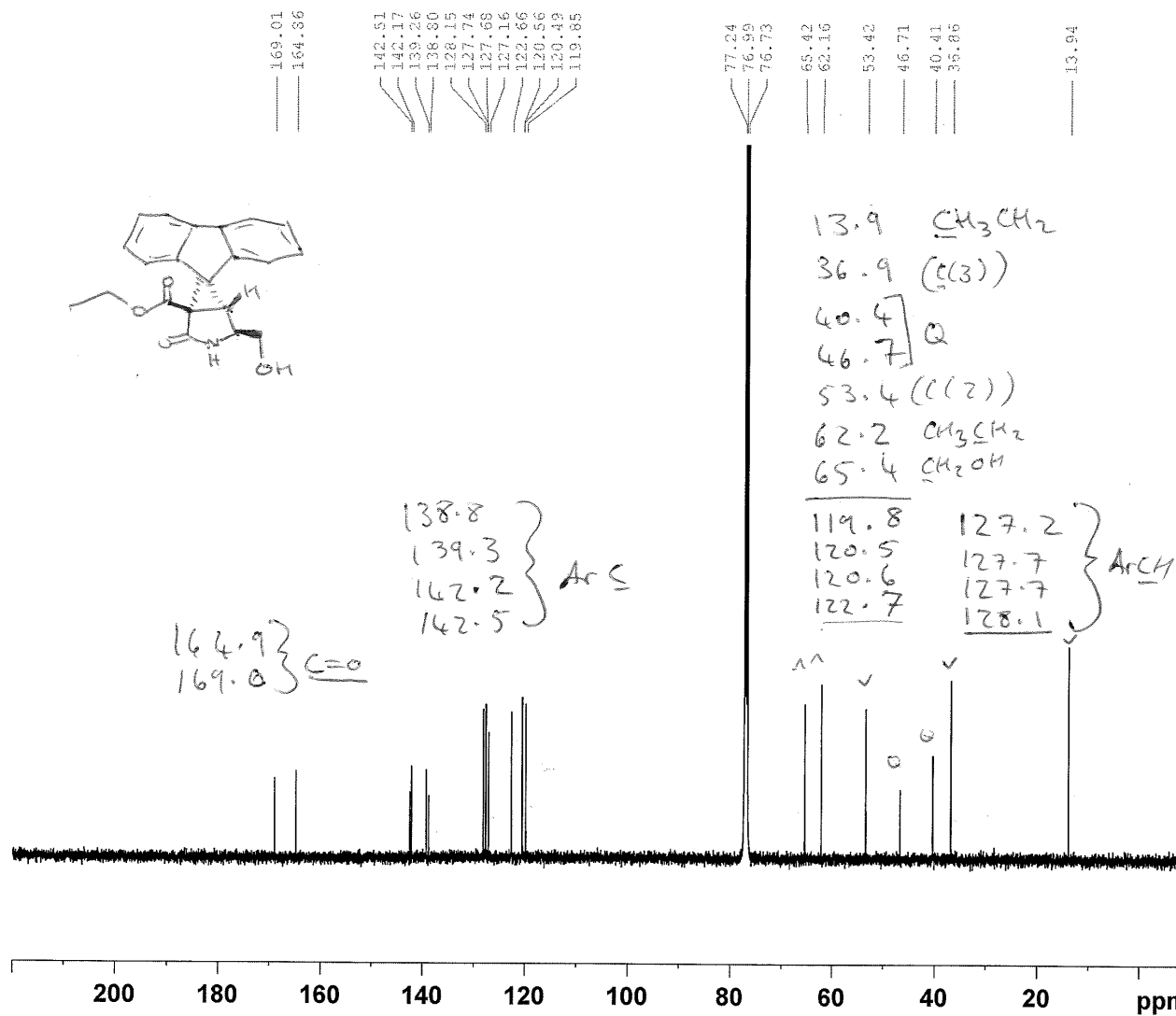
Current Data Parameters
 NAME 1h51100409
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060905
 Time 6.37
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.171923 sec
 RG 10
 DW 48.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.60 usec
 PL1 -6.00 dB
 SFO1 500.3030896 MHz

F2 - Processing parameters
 SI 32768
 SF 500.3000240 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

13c



NMR CHEM 10X

Current Data Parameters
 NAME lh51100409
 EXPNO 1
 PROCNO 1

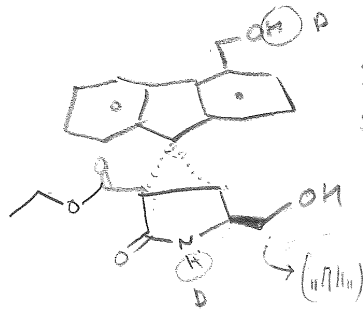
F2 - Acquisition Parameters
 Date_ 20060905
 Time_ 5.00
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 1620
 DW 16.000 usec
 DE 20.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -4.40 dB
 SFO1 125.8131151 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 12.40 dB
 PL13 17.00 dB
 SFO2 500.3020012 MHz

F2 - Processing parameters
 SI 32768
 SF 125.8005438 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

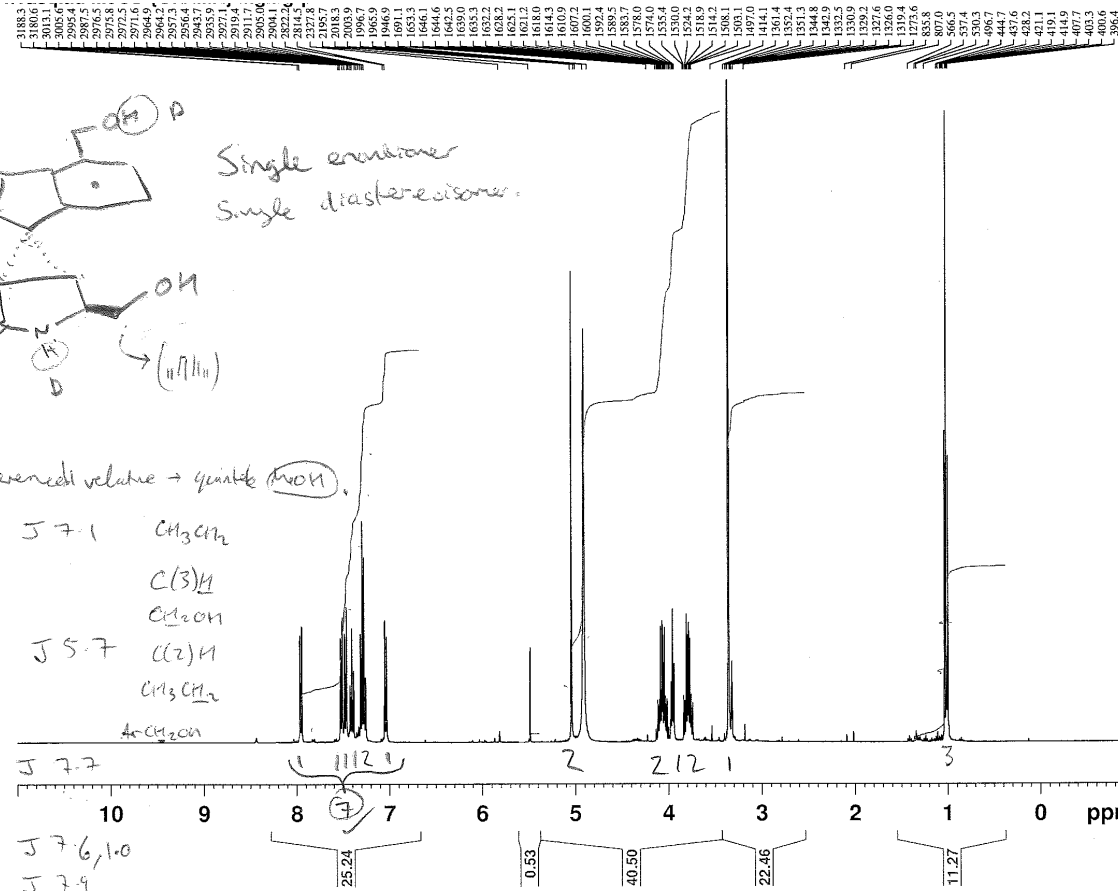
13d



Analysis:

¹H-NMR. Referenced relative to quartet (H₂O).

3H	1.007	t	J 7.1	CH ₃ CH ₂
1H	3.344	s		C(3)H
2H	3.72-3.83	m		CH ₂ OH
1H	3.95	t	J 5.7	C(2)H
2H	3.98-4.12	m		CH ₂ CH ₂
2H	5.032	s		Ar-CH ₂ OH
1H	7.031	d	J 7.7	
2H	7.26-7.31	m		
1H	7.40	td	J 7.6, 1.0	
1H	7.46	d	J 7.9	
1H	7.51	d	J 7.5	
1H	7.95	d	J 7.8	



Current Data Parameters
NAME Nov13-2007-4
EXPNO 1
PROCNO 1

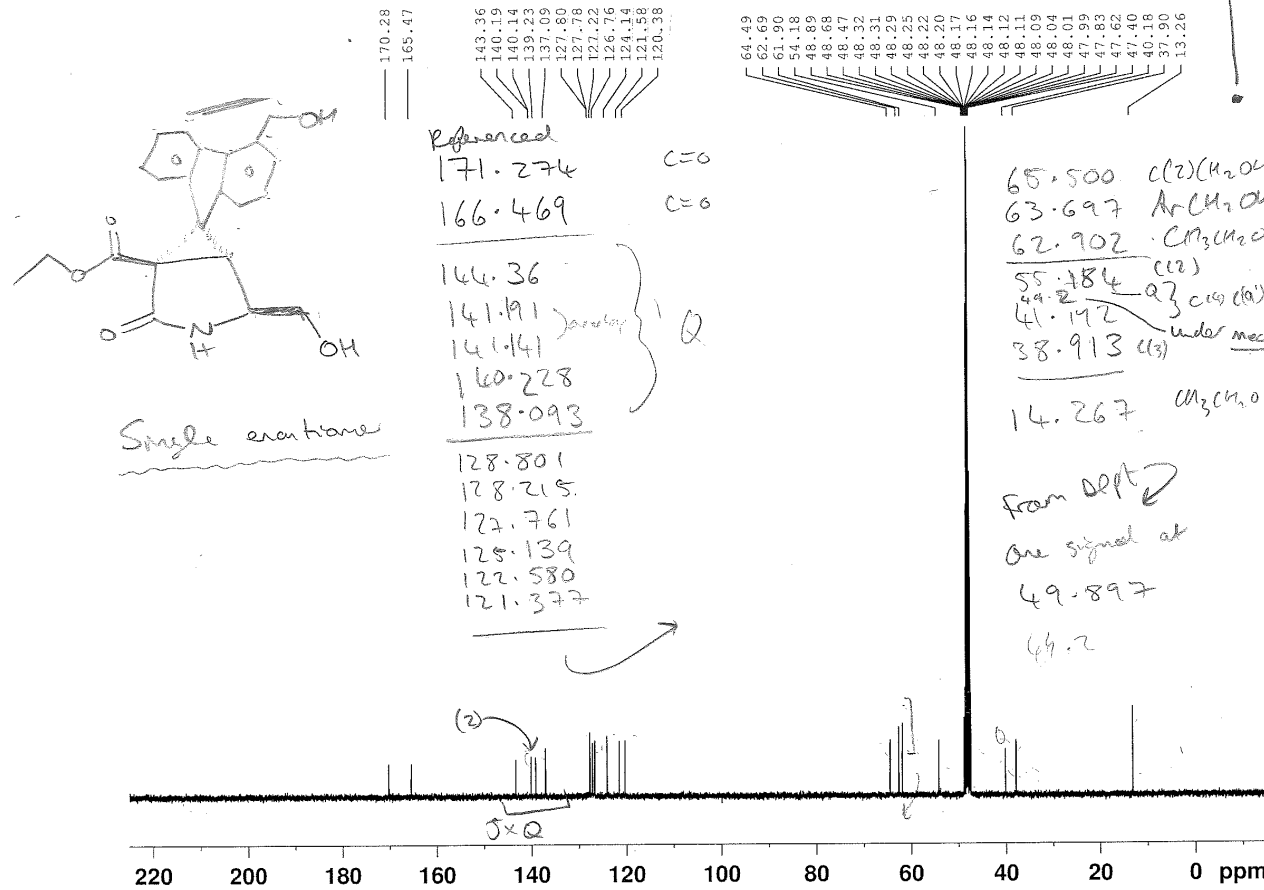
F2 - Acquisition Parameters
Date_ 20071114
Time 2.37
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg60
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 7.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 400.2024714 MHz

F2 - Processing parameters
SI 32768
SF 400.2000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

MeO2 Hz
P12 Ka
B1 F1 2
HOMEP1

13d



Current Data Parameters
NAME Nov13-2007-4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071114
Time 2.45
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT MeOD
NS 256
DS 4
SWH 26178.010 Hz
FIDRES 0.798889 Hz
AQ 0.6259188 sec
RG 32768
DW 19.100 usec
DE 7.50 usec
TE 300.0 K
D1 1.0000000 sec
d11 0.0300000 sec
DELTA 0.89999998 sec
TD0 1

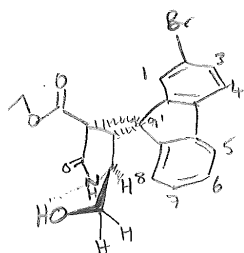
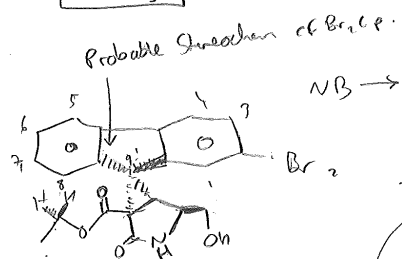
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 0.00 dB
SFO1 100.6403931 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 25.00 dB
PL2 0.00 dB
SFO2 400.2016008 MHz

F2 - Processing parameters
SI 32768
SF 100.6303300 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

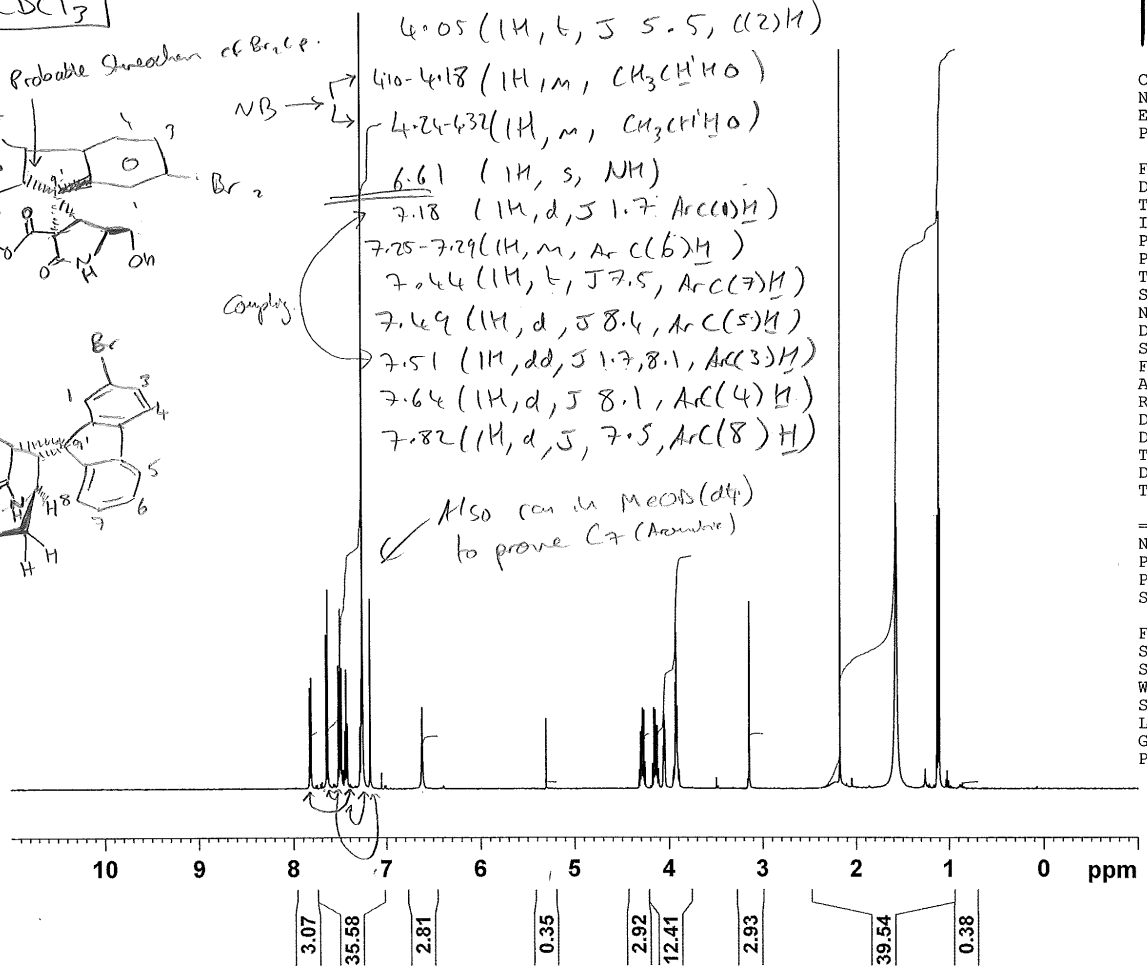
Instrument AVC500
9664 Lawrence Harris 1/4/08

CDC13



- 1.11 (3H, t, J 7.1, CH₃CH₂O)
- 3.14 (1H, s, C(3)H)
- 3.88-3.96 (2H, m, CH₂OH)
- 4.05 (1H, t, J 5.5, C(2)H)
- 4.10-4.18 (1H, m, CH₃CH₂H₂O)
- 4.24-4.32 (1H, m, CH₃CH₂H₂O)
- 6.61 (1H, s, NH)
- 7.18 (1H, d, J 1.7, ArC(1)H)
- 7.25-7.29 (1H, m, ArC(6)H)
- 7.44 (1H, t, J 7.5, ArC(7)H)
- 7.69 (1H, d, J 8.4, ArC(5)H)
- 7.51 (1H, dd, J 1.7, 8.1, ArC(3)H)
- 7.64 (1H, d, J 8.1, ArC(4)H)
- 7.82 (1H, d, J 7.5, ArC(8)H)

Also run in MeOD (d₄)
to prove C7 (Aromatic)



NMR@CHEM.OX

Current Data Parameters
NAME lh96640104
EXPNO 1
PROCNO 1

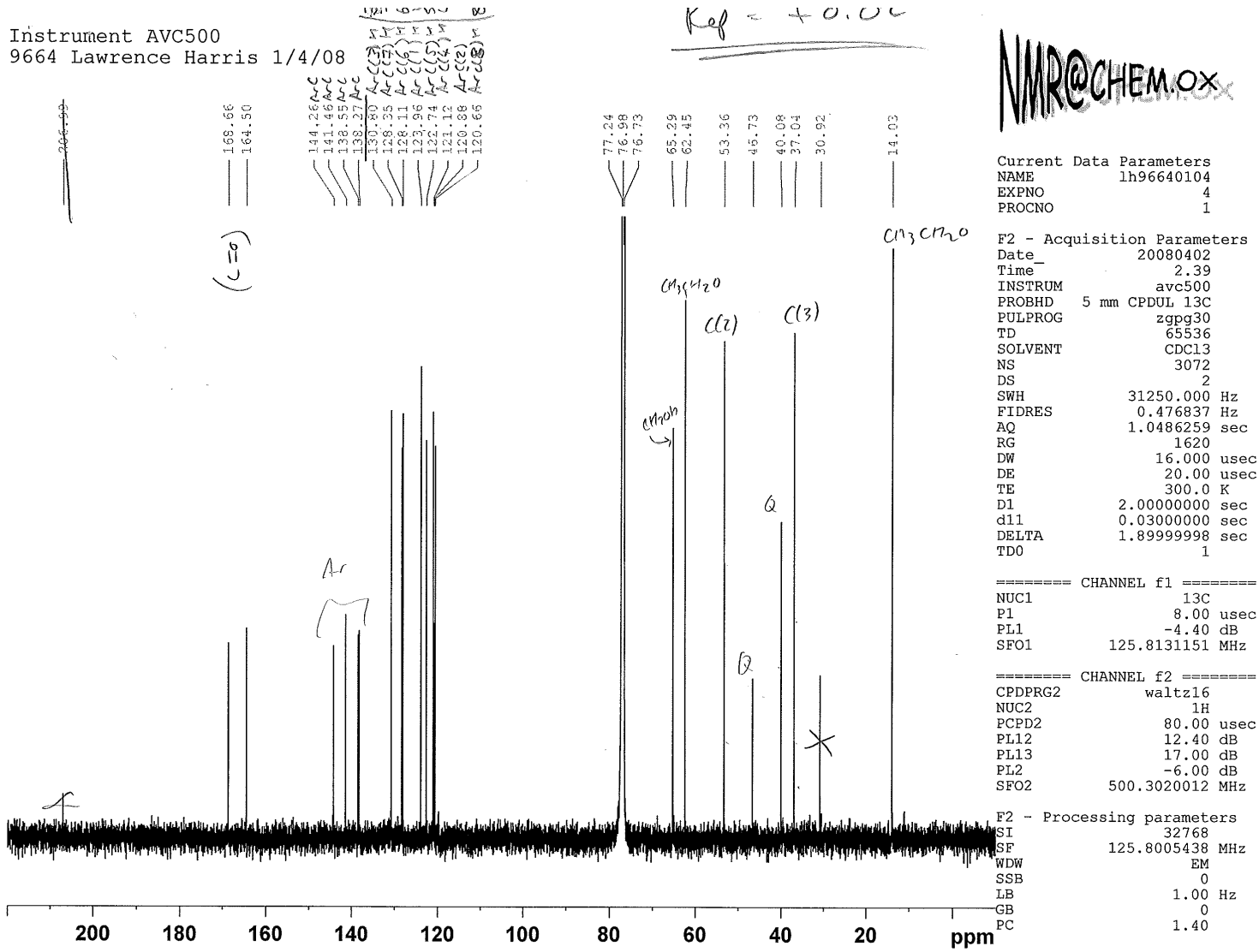
F2 - Acquisition Parameters
Date 20080401
Time 23.15
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 4
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 500.3030896 MHz

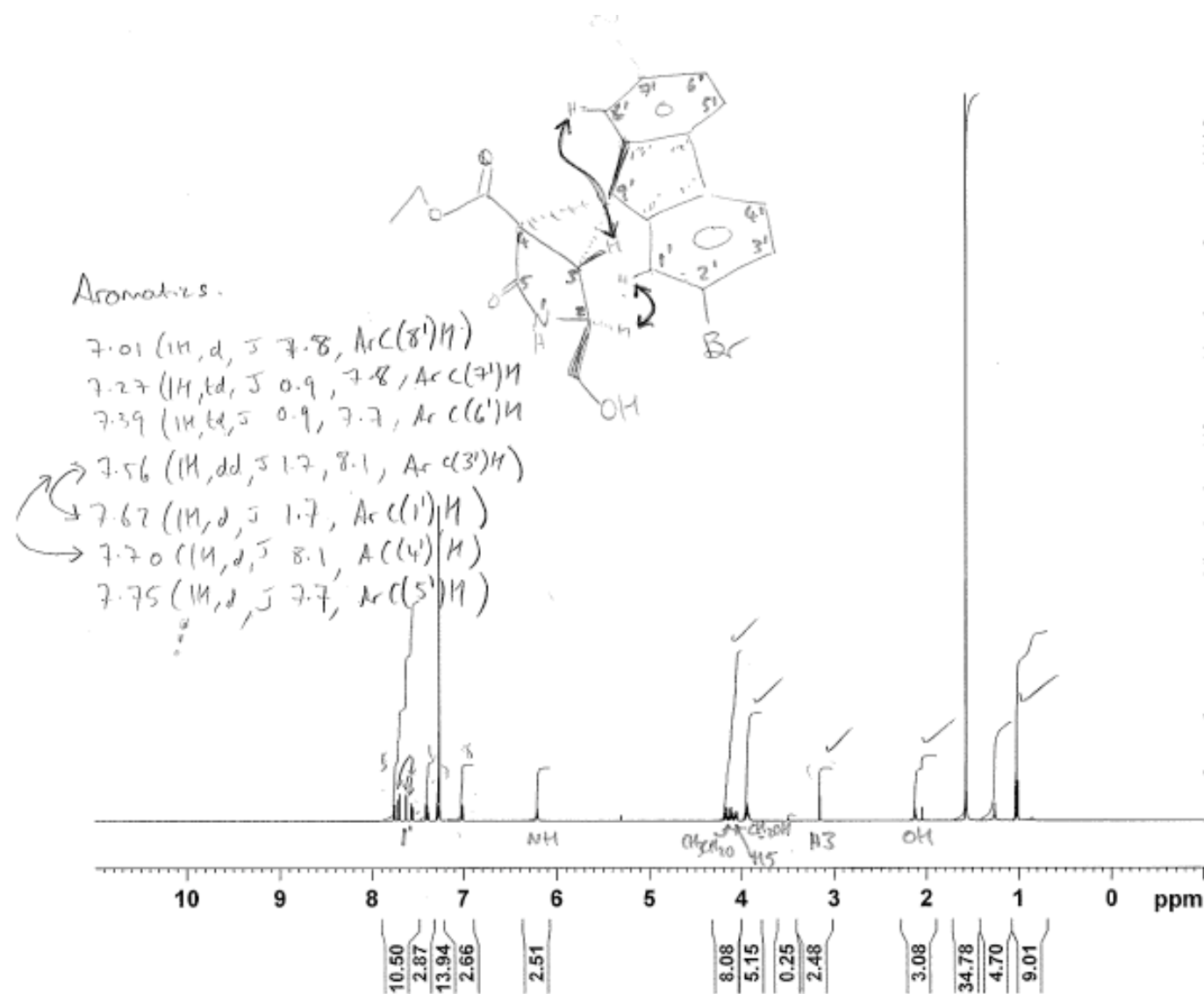
F2 - Processing parameters
SI 32768
SF 500.3000240 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

from di-

13e



13f



NMR@CHEM.OX

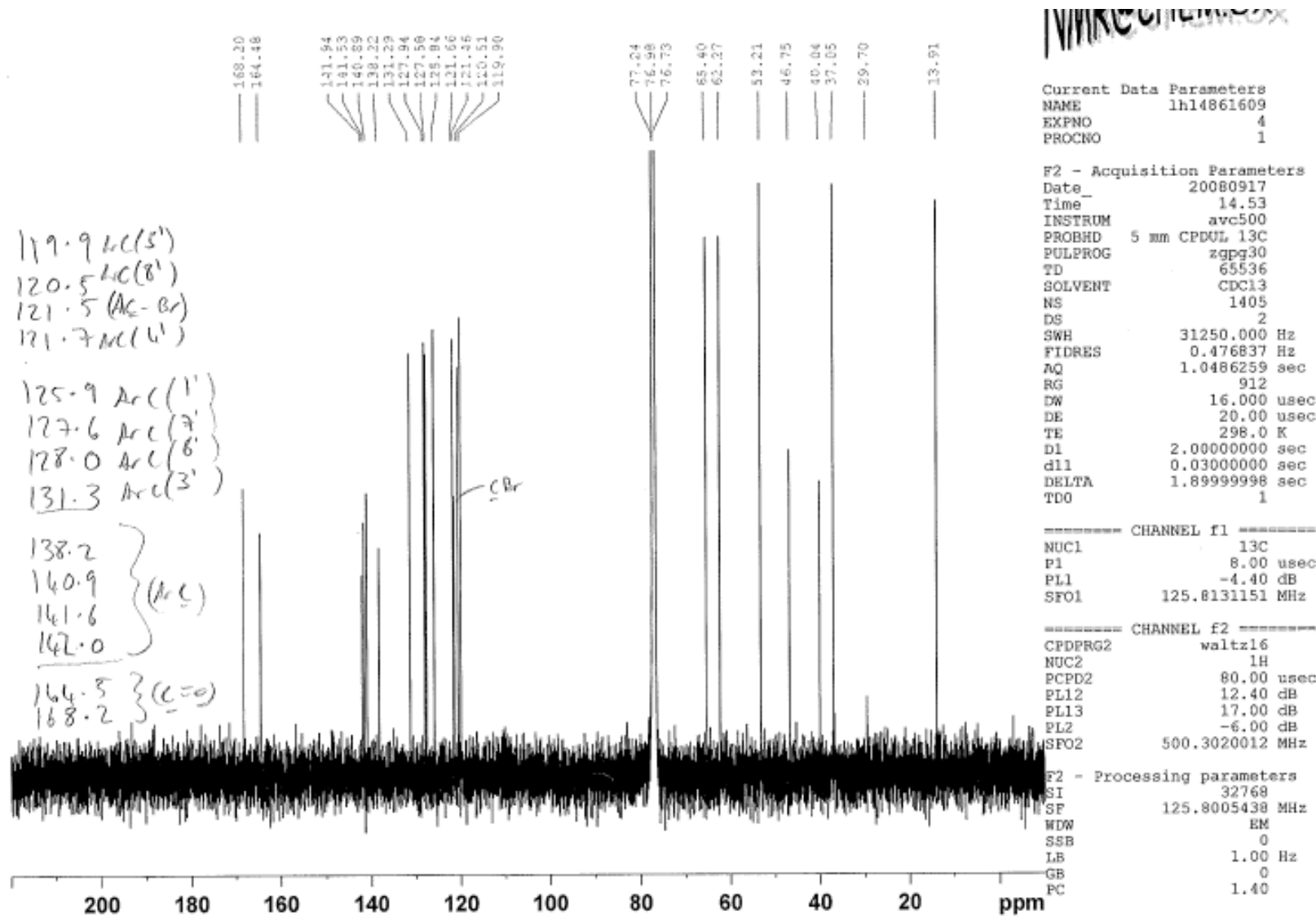
Current Data Parameters
 NAME lh14861609
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080917
 Time 13.24
 INSTRUM avo500
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 4
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

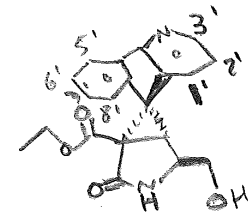
CHANNEL f1
 NUC1 1H
 P1 9.60 usec
 PL1 -6.00 dB
 SFO1 500.3030896 MHz

F2 - Processing parameters
 SI 32768
 SF 500.3000240 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

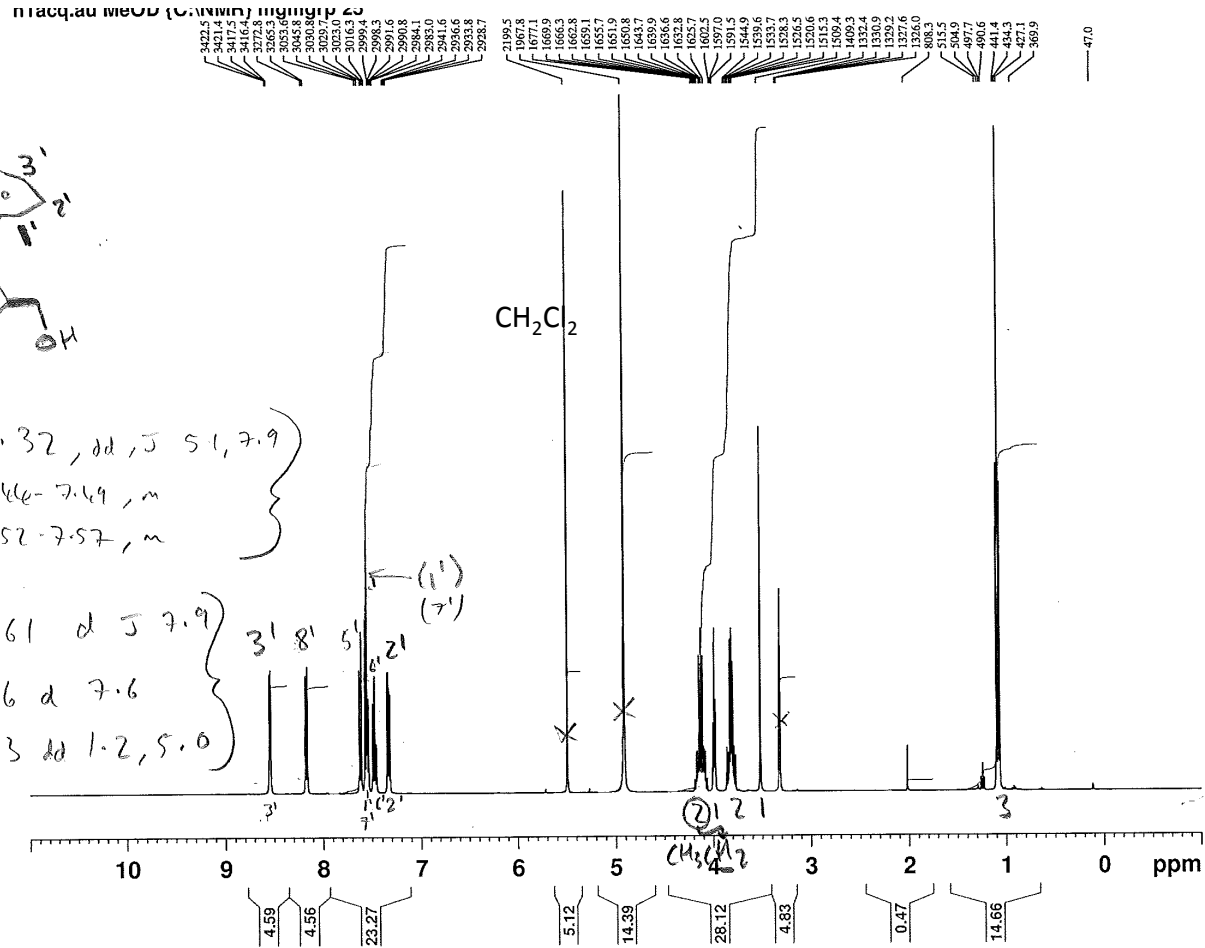
13f



13h



2' - 7.32, dd, J 5.1, 7.9
 6' - 7.46-7.49, m
 1' - 7.52-7.57, m
 7' -
 5' - 7.61 d J 7.9
 8' - 8.16 d 7.6
 3' - 8.53 dd 1.2, 5.0



Current Data Parameters
 NAME Apr15-2008-25
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080415
 Time 13.15
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg60
 TD 65536
 SOLVENT MeOD
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 90.5
 DW 60.400 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.00000000 sec

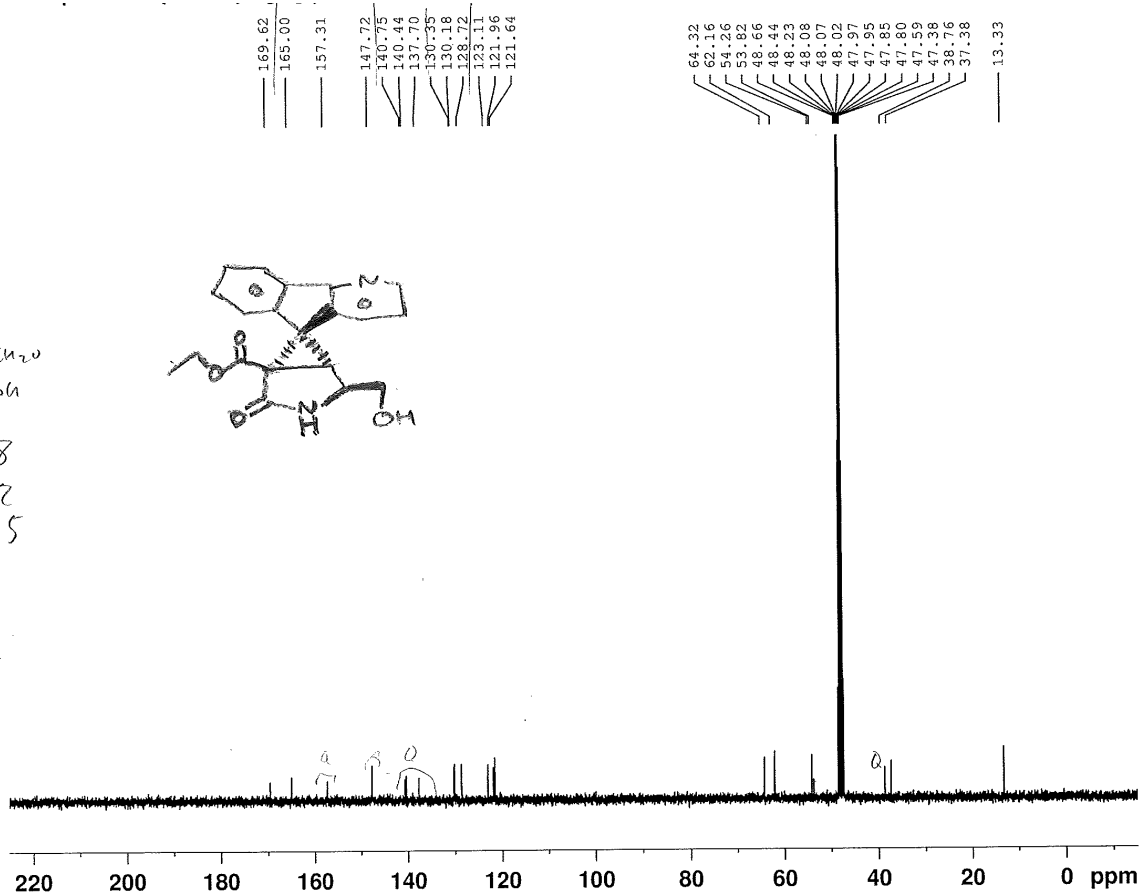
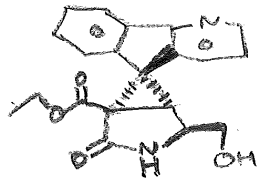
===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.2024714 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

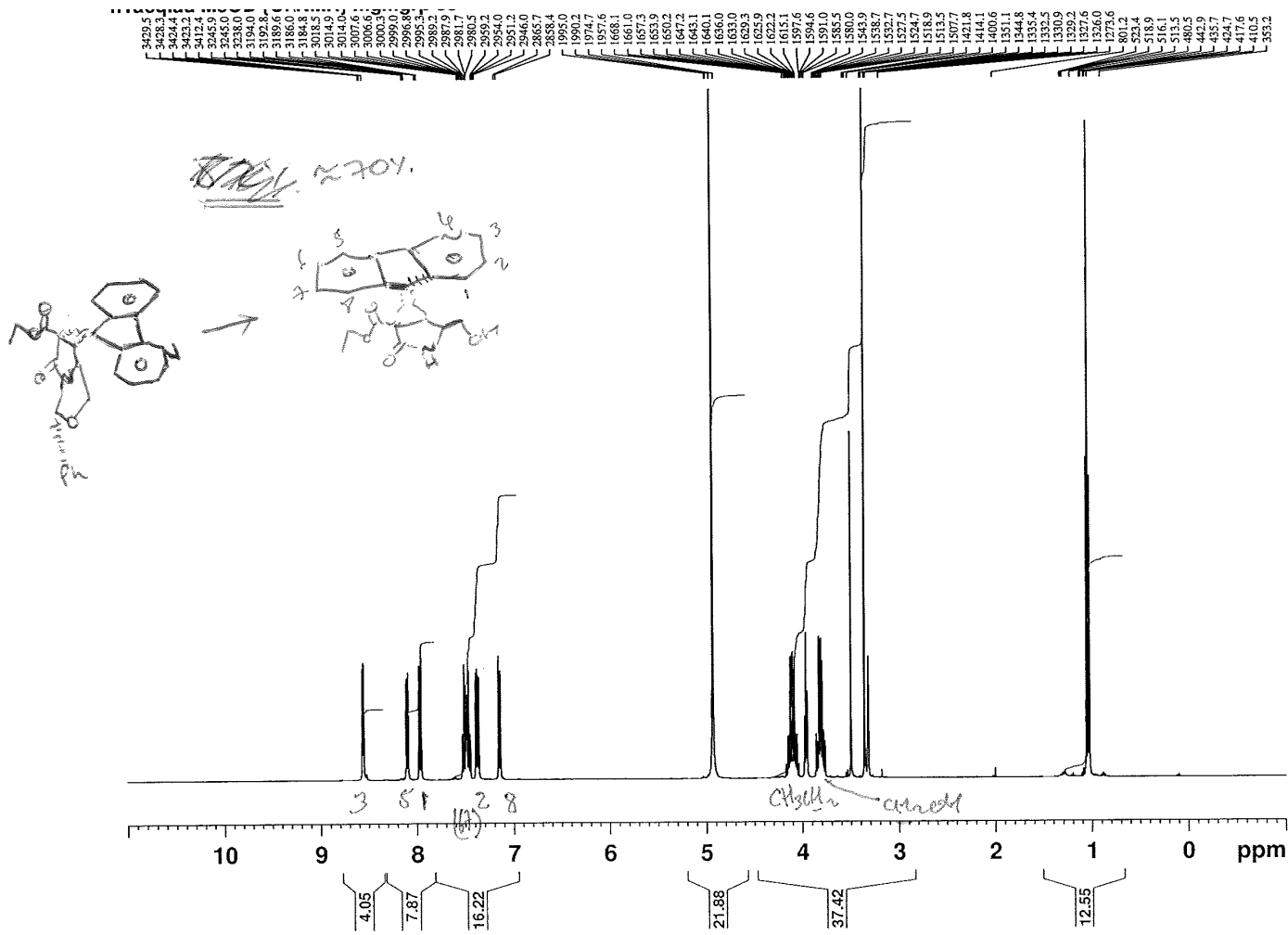
47.0

13h

16.4 CH₃
 38.4 (13)
 39.8 } 0
 54.9 } 0
 55.3 (12)
 63.2 CH₂CH₂O
 65.4 CH₂OH
 122.7 - 8
 123.0 - 2
 124.1 - 5
 129.8 - 6
 131.2 } 1
 131.4 } 7
 138.7 } 0
 141.5 } 0
 141.8 } 0
 148.7 - 3
 158.3 - O-N
 166.0 } CO
 170.6 } CO



Current Data Parameters
 NAME Apr15-2008-25
 EXPNO 3
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20080415
 Time 13.27
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT MeOD
 NS 256
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.798889 Hz
 AQ 0.6259188 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 d11 0.0300000 sec
 DELTA 0.89999998 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.00 dB
 PL13 25.00 dB
 PL2 0.00 dB
 SFO2 400.2016008 MHz
 F2 - Processing parameters
 SI 32768
 SF 100.6303300 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

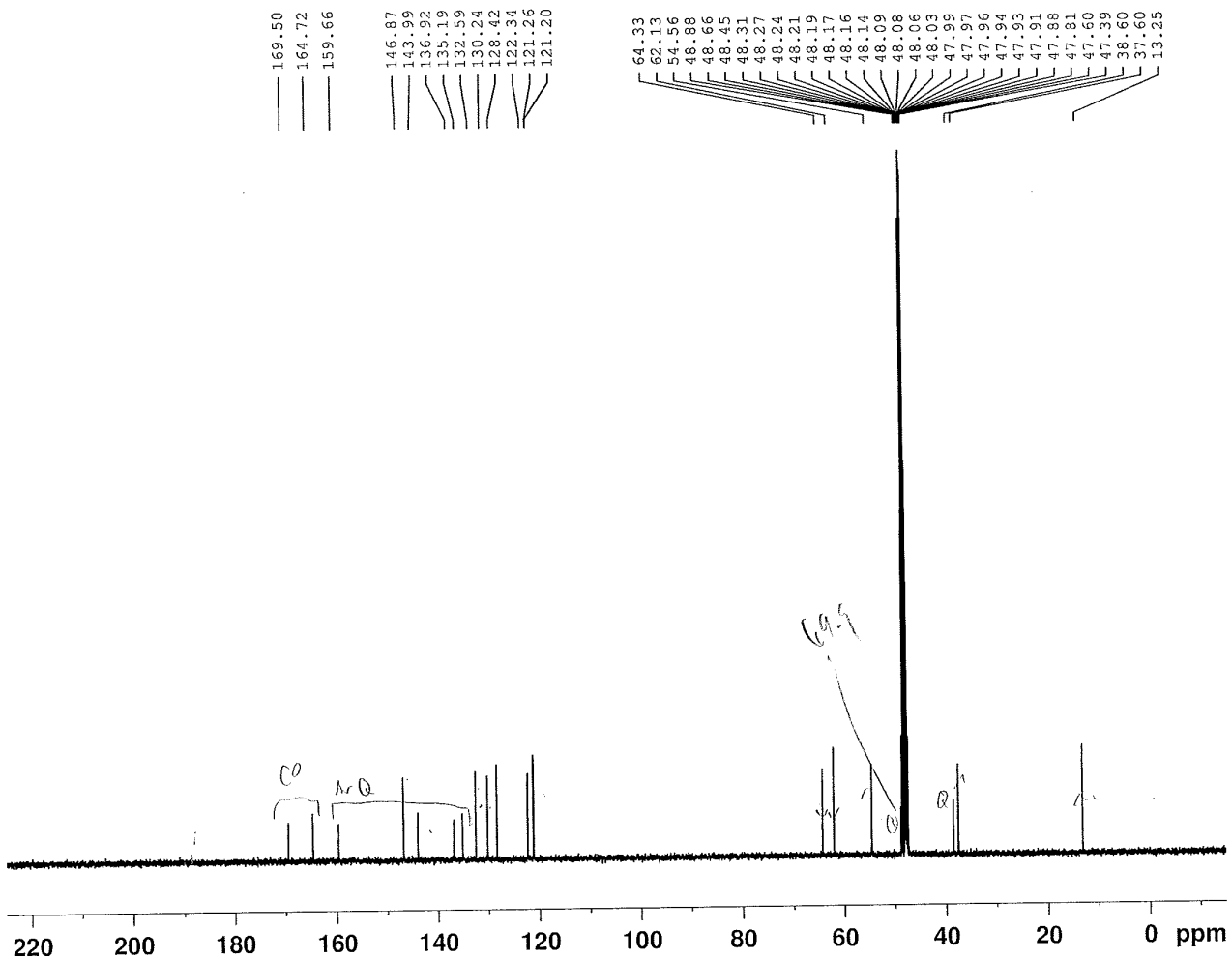


Current Data Parameters
 NAME May16-2008-55
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080516
 Time 19.24
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg60
 TD 65536
 SOLVENT MeOD
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 57
 DW 60.400 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.2024714 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME May16-2008-55
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080516
 Time 19.32
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT MeOD
 NS 256
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.798889 Hz
 AQ 0.6259188 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 d11 0.0300000 sec
 DELTA 0.89999998 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz

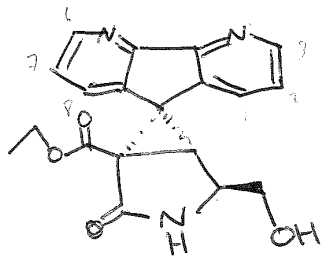
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.00 dB
 PL13 25.00 dB
 PL2 0.00 dB
 SFO2 400.2016008 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6303300 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

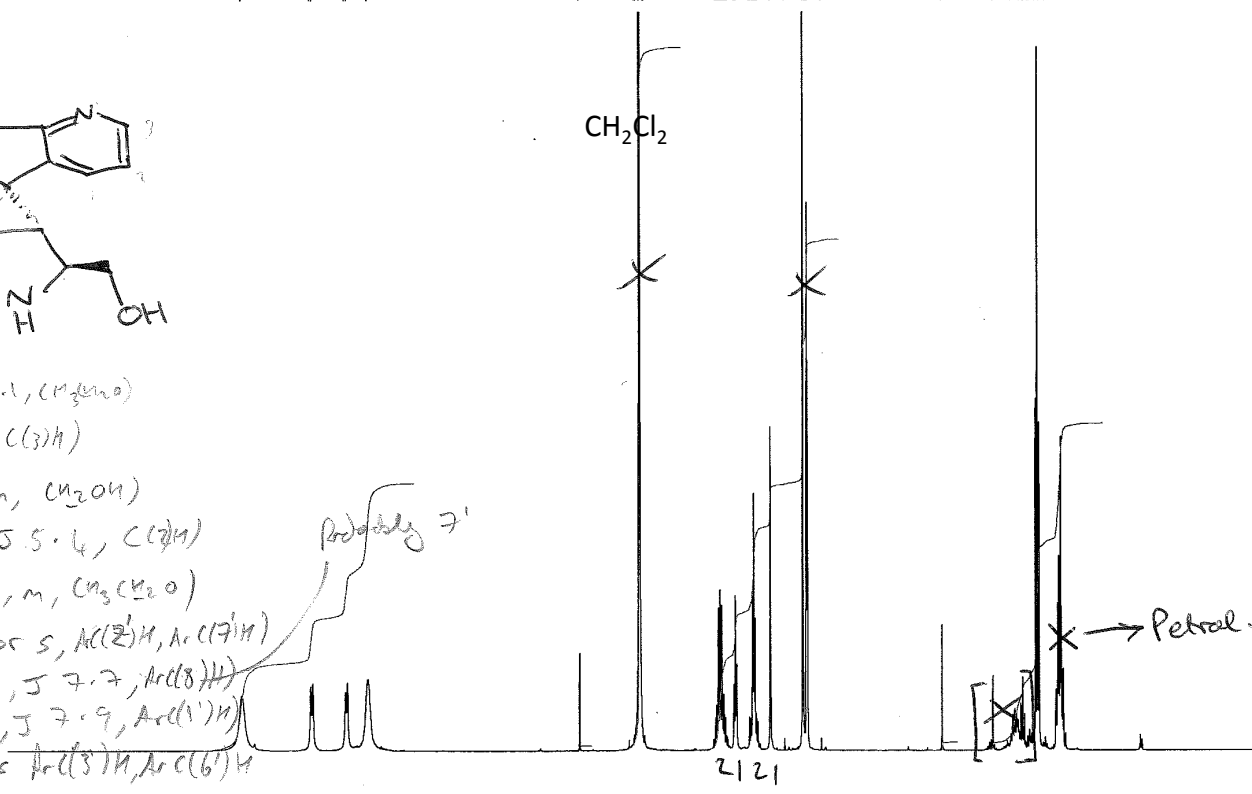
13j

Instrument DQX400
 Chemist LDH
 Group mgm
 LH04-77

h1accq.au MeOD (C:NMR) mgmgrp 53



- 1.10 (3H, t, J 7.1, (CH₃)₂CHO)
- 3.66 (1H, s, C(3)H)
- 3.77-3.87 (2H, m, CH₂OH)
- 4.00 (1H, t, J 5.4, C(2)H)
- 4.07-4.21 (2H, m, CH₂CH₂O)
- 7.53 (2H, br s, Ar(2')H, Ar(7')H)
- 7.76 (1H, d, J 7.7, Ar(8)H)
- 8.07 (1H, d, J 7.9, Ar(1')H)
- 8.74 (2H, br s, Ar(3')H, Ar(6')H)



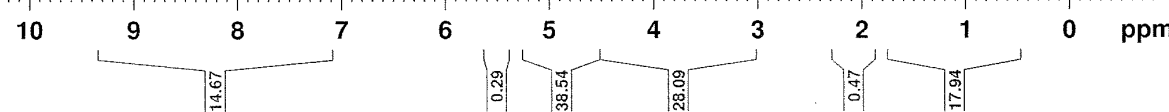
NMRO@CHEM.OX

Current Data Parameters
 NAME May20-2008-53
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080521
 Time 0.29
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg60
 TD 65536
 SOLVENT MeOD
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 90.5
 DW 60.400 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.00000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.2024714 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



13j

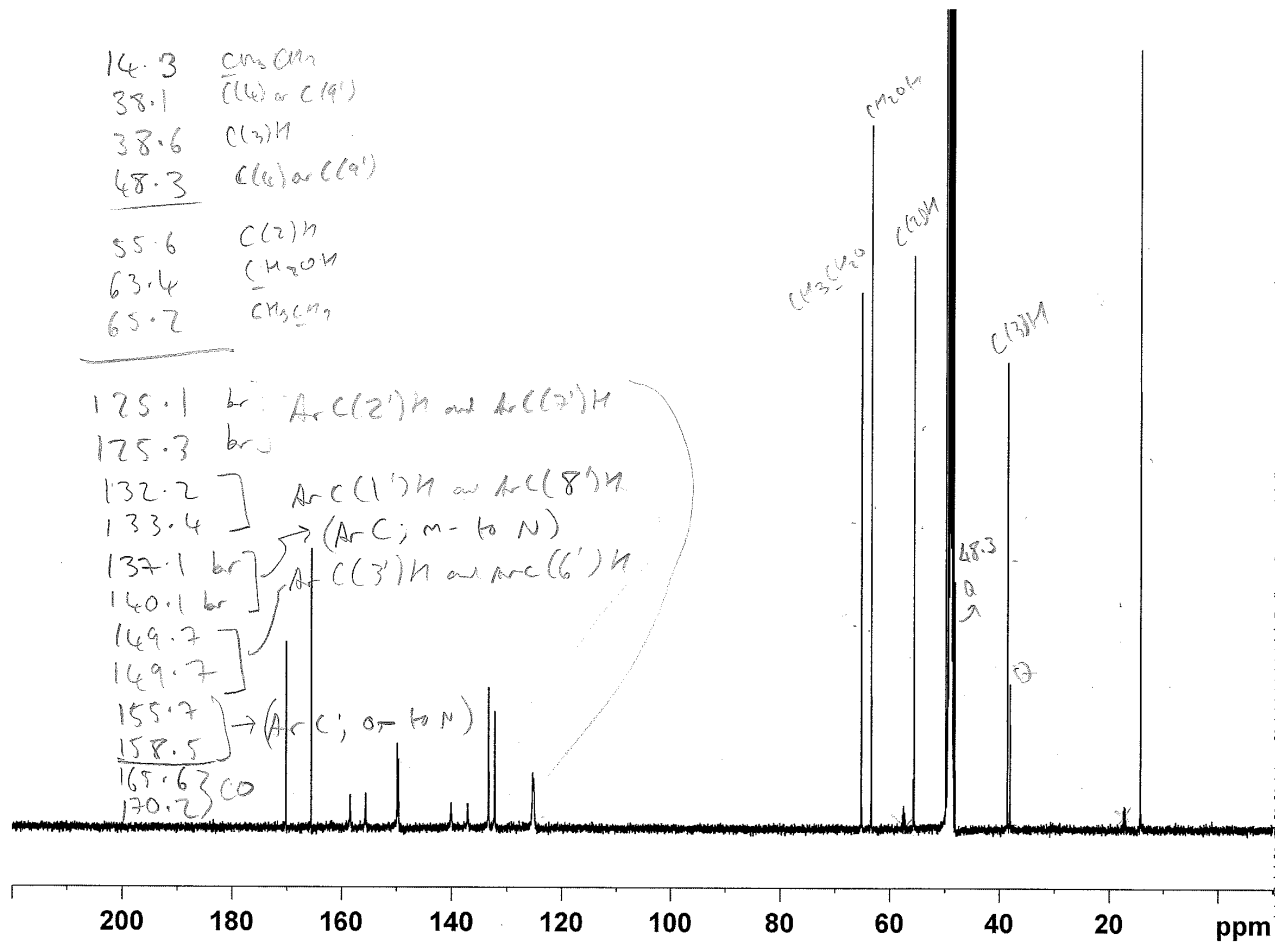
NMR@CHEM.OX

170.28
170.19
165.62
158.48
155.65
149.90
149.68
140.11
137.13
133.38
132.24
125.20
125.13

65.23
63.44
57.68
57.51
57.34
57.17
55.76
55.54
49.88
49.66
49.55
49.49
49.38
49.32
49.21
49.03
48.87
48.69
48.52
48.24
38.65
38.60
38.12
17.46
17.30
17.15
17.00
14.47
14.33

14.3 CH_2OH
38.1 ((6) or C(4))
38.6 C(3)H
48.3 C(6) or C(4')
55.6 C(2)H
63.4 CH_2OH
65.2 CH_2OH

125.1 br: Ar C(2)H and Ar C(7)H
125.3 br
132.2 } Ar C(1)H and Ar C(8)H
133.4 }
137.1 br } Ar C(3)H and Ar C(6)H
140.1 br }
149.7 }
149.7 }
155.7 } Ar C(5) or Ar C(N)
158.5 }
165.6 } CO
170.2 } CO



Current Data Parameters
NAME lh04592105
EXPNO 4
PROCNO 1

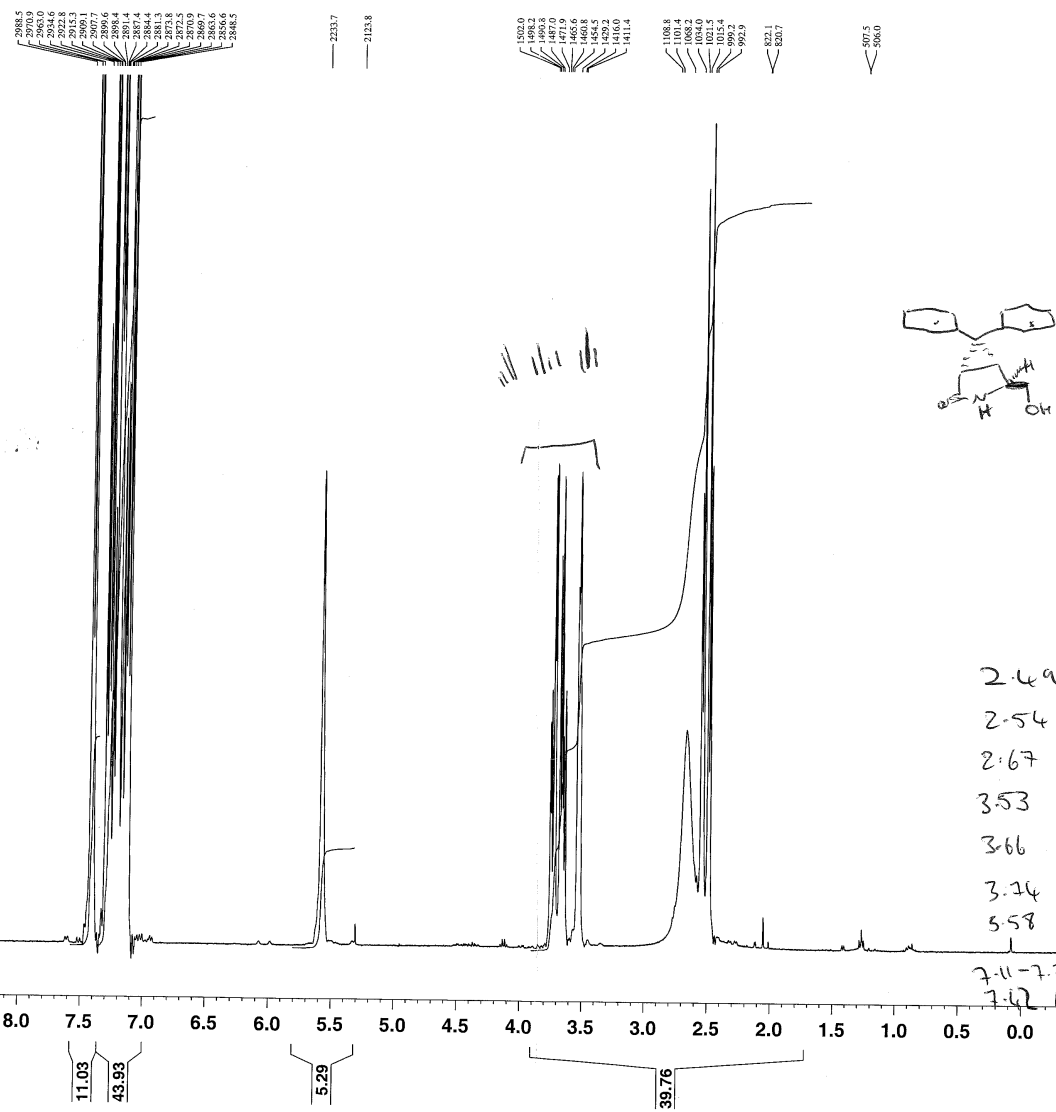
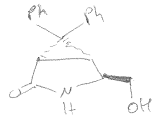
F2 - Acquisition Parameters
Date_ 20080522
Time_ 7.19
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 1820
DW 16.000 usec
DE 20.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -4.40 dB
SFO1 125.8131151 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.40 dB
PL13 17.00 dB
PL2 -6.00 dB
SFO2 500.3020012 MHz

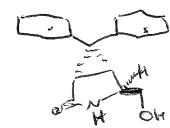
F2 - Processing parameters
SI 32768
SF 125.8003564 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

13k



Current Data Parameters
NAME Jul18-2006-5
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060718
Time 19.22
INSTRUM dpx400
PROBHD 5 mm Dual 1H/1
PULPROG zg60
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 128
DW 89.400 use
DE 17.00 use
TE 0.0 K
D1 1.00000000 sec



==== CHANNEL f1 =====
NUC1 1H
P1 7.30 use
PL1 0.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1300182 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.60

2.49 (1H, d, J^{6.2} 6.28, (4H))
2.54 (1H, d J^{6.07} 6.07, (3H))
2.67 (1H, br.s, (CH₂OH))
3.53 (1H, t, J = 6.5, (2H))
3.66 (1H, dd, J = 11.1, 6.3 CH(OH))
3.74 (1H, dd, J = 11.2, 3.8 CH(OH))
5.58 (1H, s, NH)

7.11-7.32 (8H, m, ArH)
7.42 (2H, d, J 7.42, ArH)

7.584.5
7.570.9
7.566.0
7.561.8
7.557.6
7.553.3
7.549.1
7.544.9
7.540.6
7.536.4
7.532.1
7.527.9
7.523.6
7.519.4
7.515.2
7.510.9
7.506.7
7.502.4
7.498.2
7.493.9

2.233.7
2.123.8

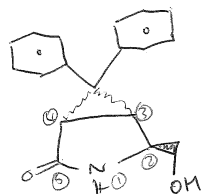
1.982.0
1.977.7
1.973.5
1.969.2
1.965.0
1.960.8
1.956.6
1.952.4
1.948.2
1.944.0
1.939.7
1.935.5
1.931.3
1.927.1
1.922.9
1.918.7
1.914.5

1.108.8
1.104.6
1.100.4
1.096.2
1.092.0
1.087.8
1.083.6
1.079.4
1.075.2
1.071.0
1.066.8
1.062.6
1.058.4
1.054.2
1.050.0
1.045.8
1.041.6
1.037.4
1.033.2
1.029.0
1.024.8
1.020.6
1.016.4
1.012.2
1.008.0
1.003.8
1.000.0

507.5
506.0

11.03
43.93
5.29
39.76

13k



175.48
 143.42
 136.45
 129.79
 128.89
 127.46
 127.03
 126.74
 77.34
 76.92
 76.70
 65.58
 55.43
 39.46
 35.09
 30.86

30.86 ①
 35.09 ②
 39.46 (Q) C(Ph)₂
 55.43 ②
 65.58 CH₂OH
 P- ← [126.74
 127.03
 127.46
 128.53
 128.89
 129.79] 6 Ar C
 136.45 (Q)H
 143.42 (Q)H
 175.48 = (C=O)

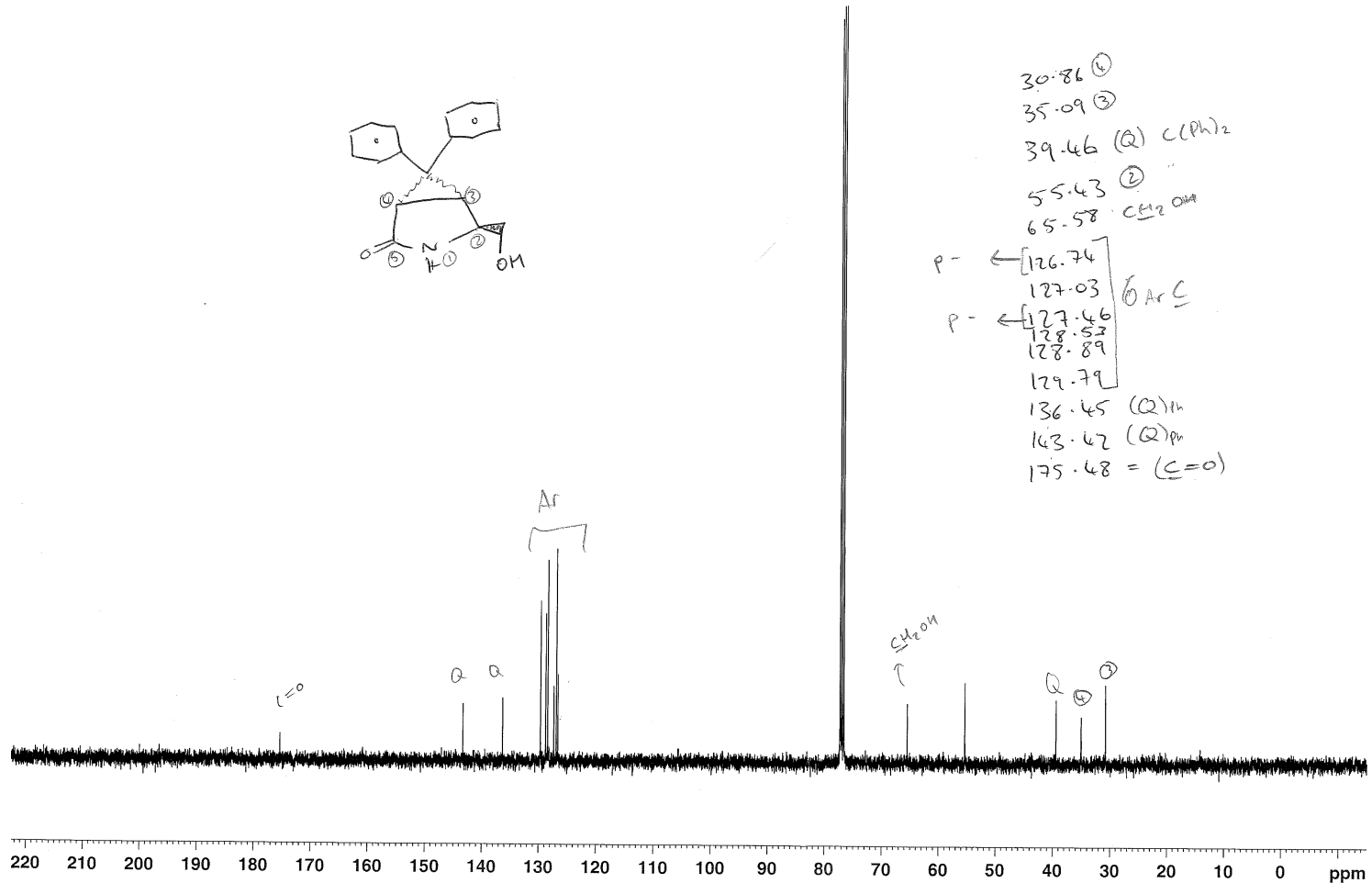
Current Data Parameters
 NAME Mar20-2006-25
 EXPNO 2
 PROCNO 1

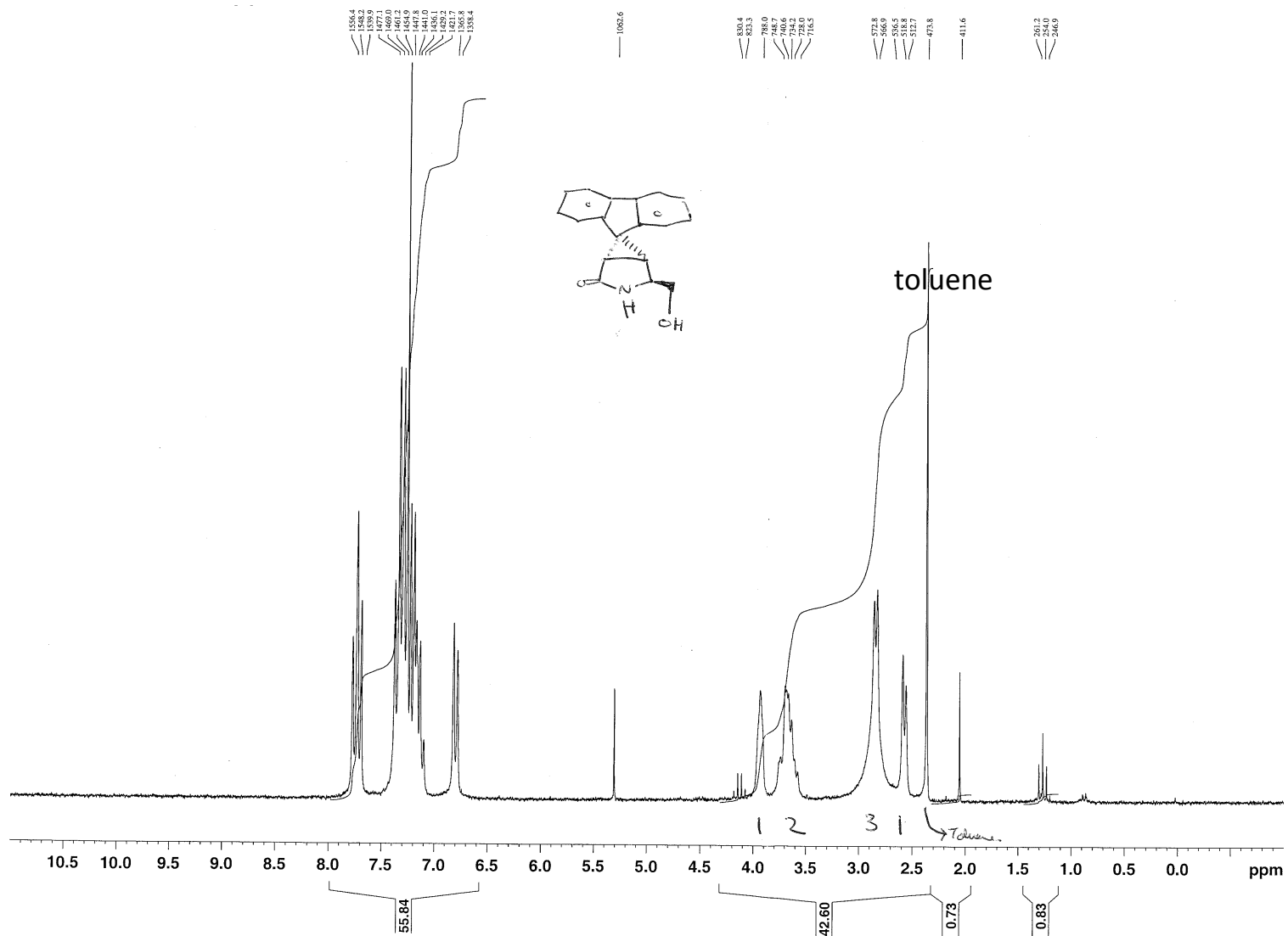
F2 - Acquisition Parameters
 Date_ 20060321
 Time 10.12
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 256
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 296.3 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 19.00 dB
 PL13 25.00 dB
 SFO2 400.2016008 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6303718 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



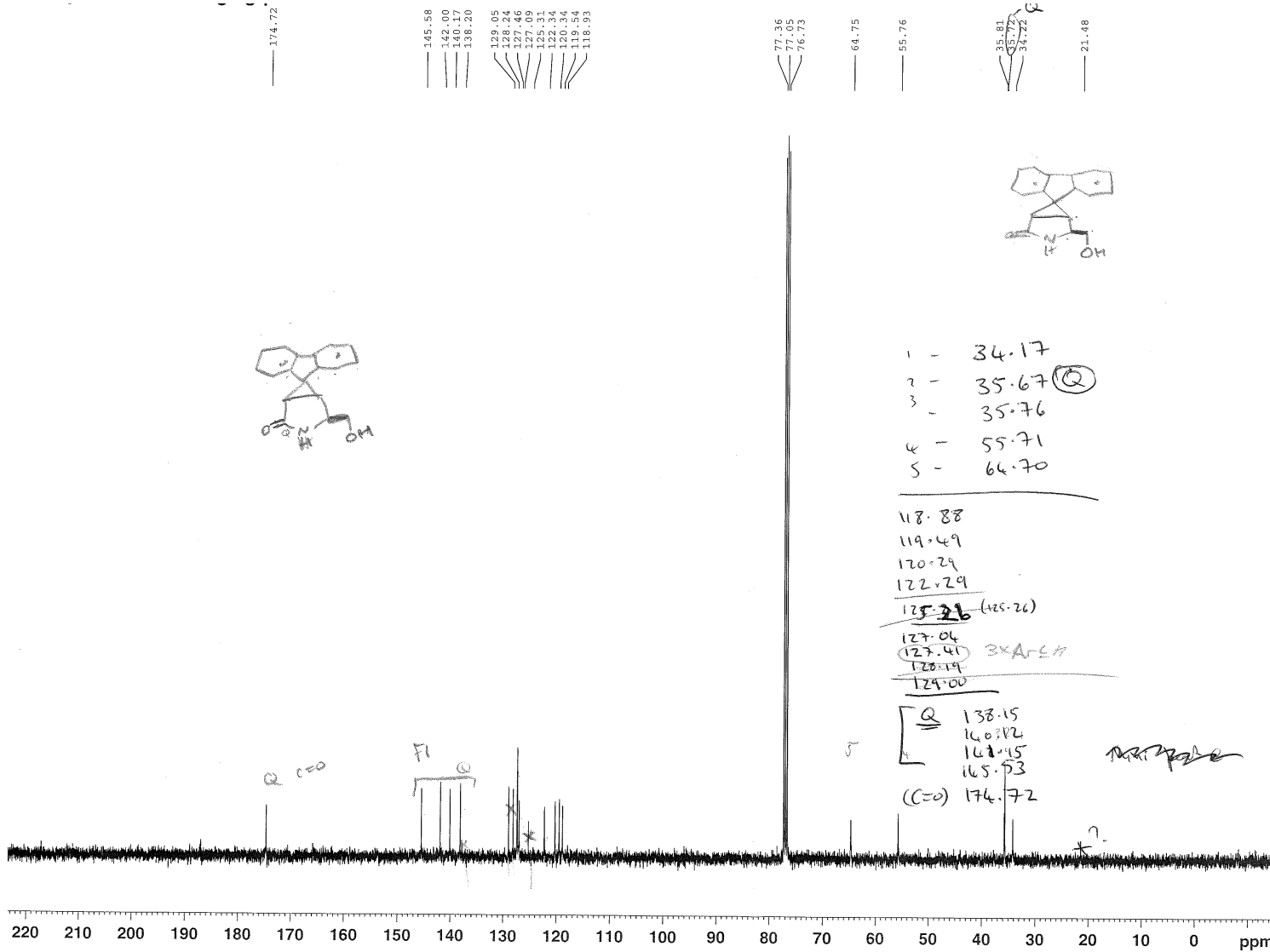


Current Data Parameters
 NAME Mar27-2006-44
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060327
 Time 22.47
 INSTRUM dpx200
 PROBHD 5 mm Dual 13C/
 PULPROG zg60
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 3205.128 Hz
 FIDRES 0.195625 Hz
 AQ 2.5559540 sec
 RG 1625.5
 DW 156.000 use
 DE 6.00 use
 TE 300.0 K
 D1 1.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 7.80 use
 PL1 -3.00 dB
 SFO1 200.1310007 MHz

F2 - Processing parameters
 SI 16384
 SF 200.1300125 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME Apr03-2006-21
 EXPNO 2
 PROCNO 1

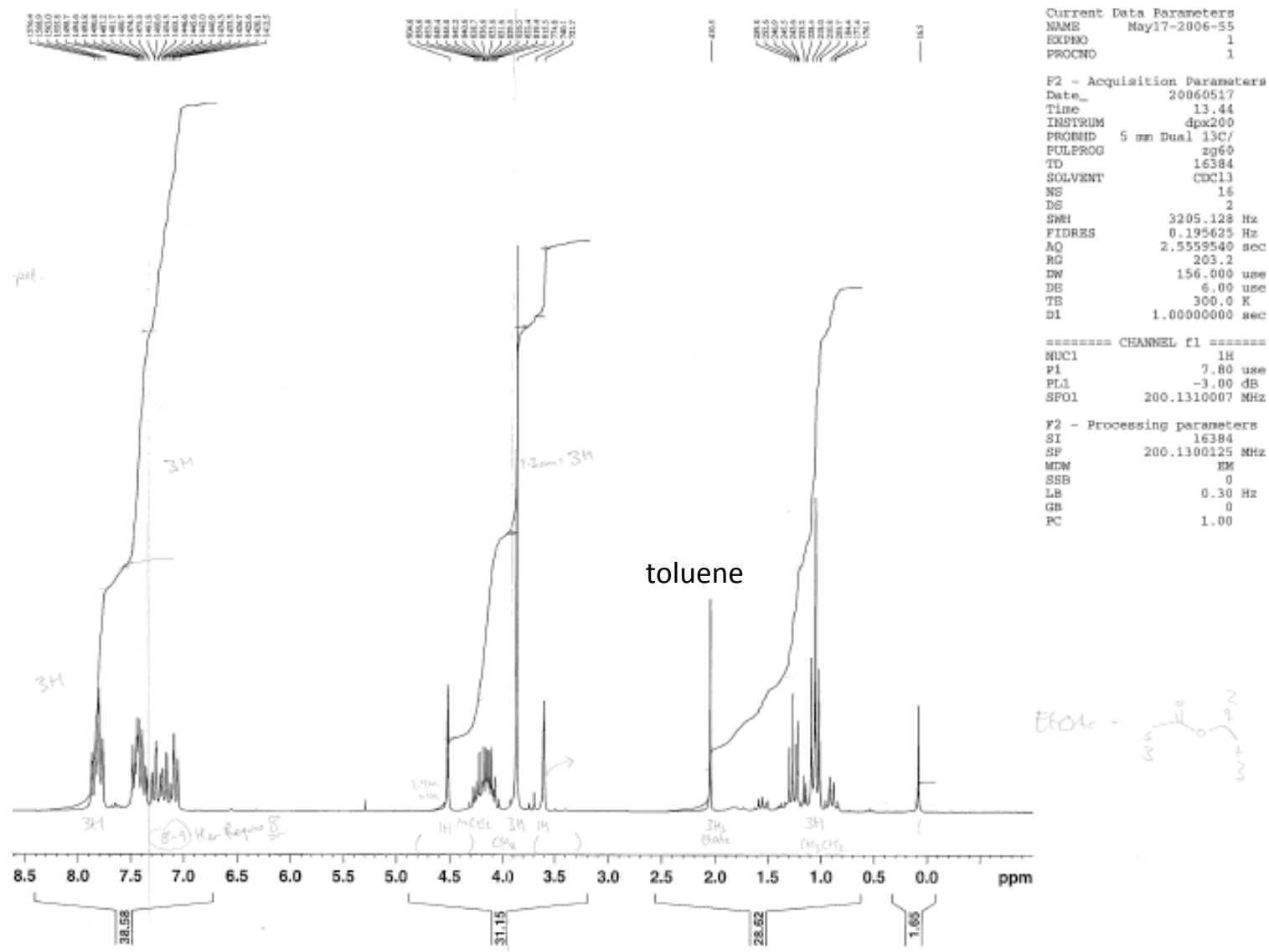
F2 - Acquisition Parameters
 Date_ 20060403
 Time 22.42
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 295.6 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz

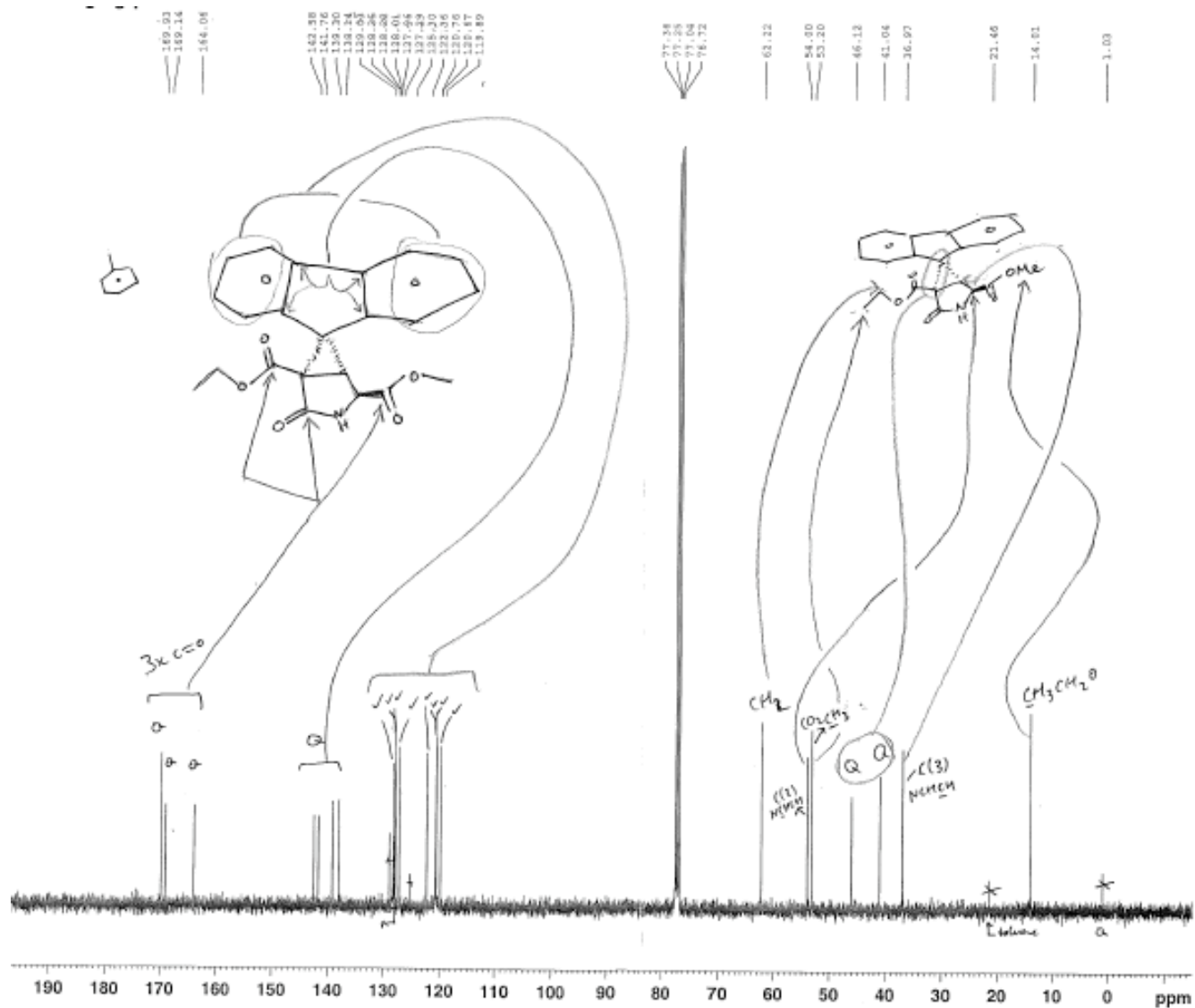
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 19.00 dB
 PL13 25.00 dB
 SFO2 400.2016008 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6303718 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

14a



14a



Current Data Parameters
 NAME May17-2006-25
 EXPNO 3
 PROCNO 1

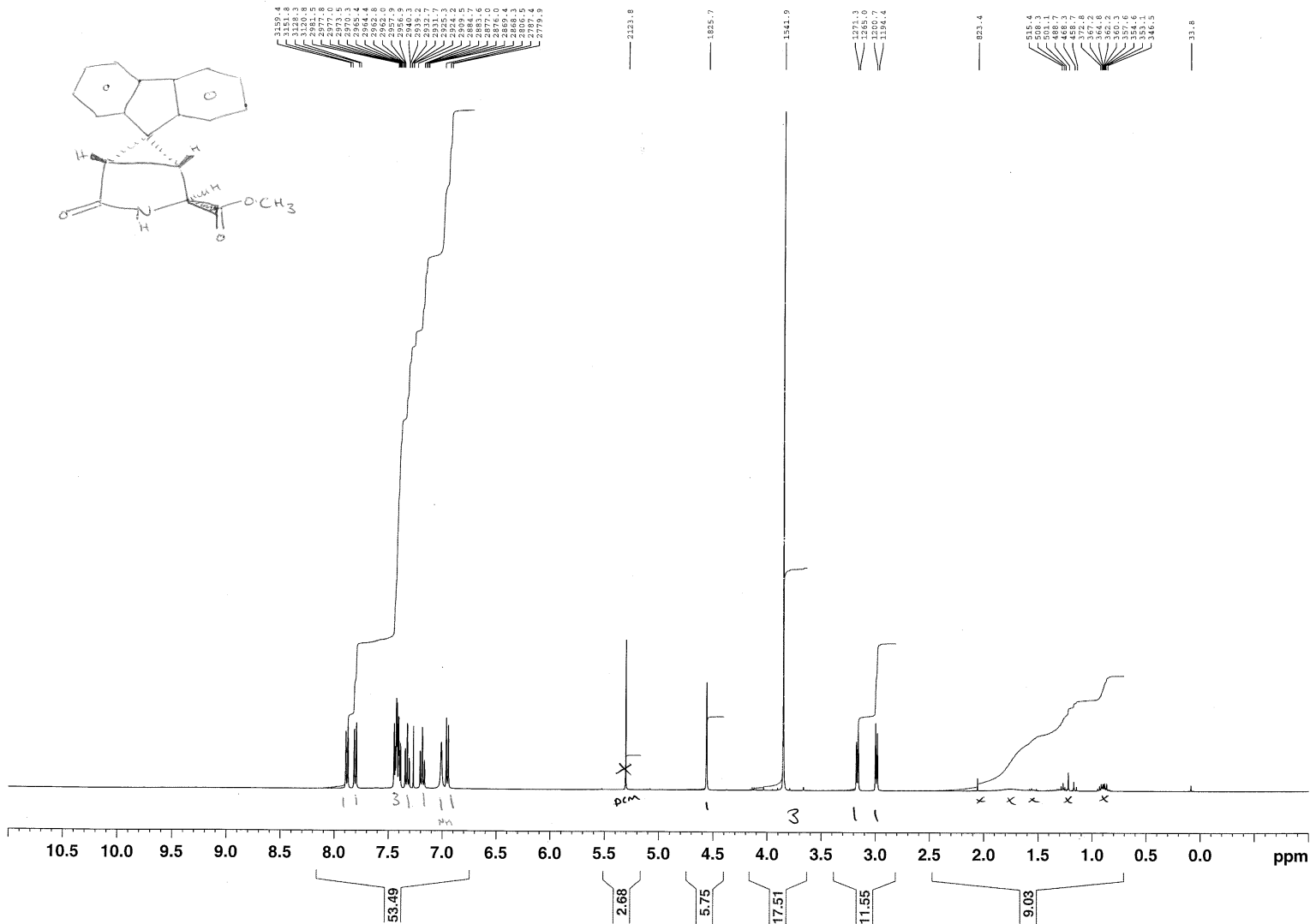
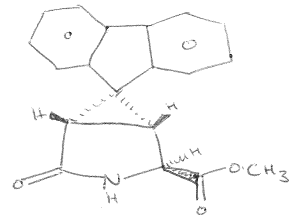
F2 - Acquisition Parameters +
 Date_ 20060519
 Time 2.14
 INSTRUM av400
 PROBRD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 256
 DS 4
 SMI 26178.010 Hz
 FIDRES 0.399405 Hz
 AQ 1.2517875 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 296.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 MCREST 0.0000000 sec
 MCNEX 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 19.00 dB
 PL13 25.00 dB
 SFO2 400.2016008 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6303718 MHz
 WDM 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

14b



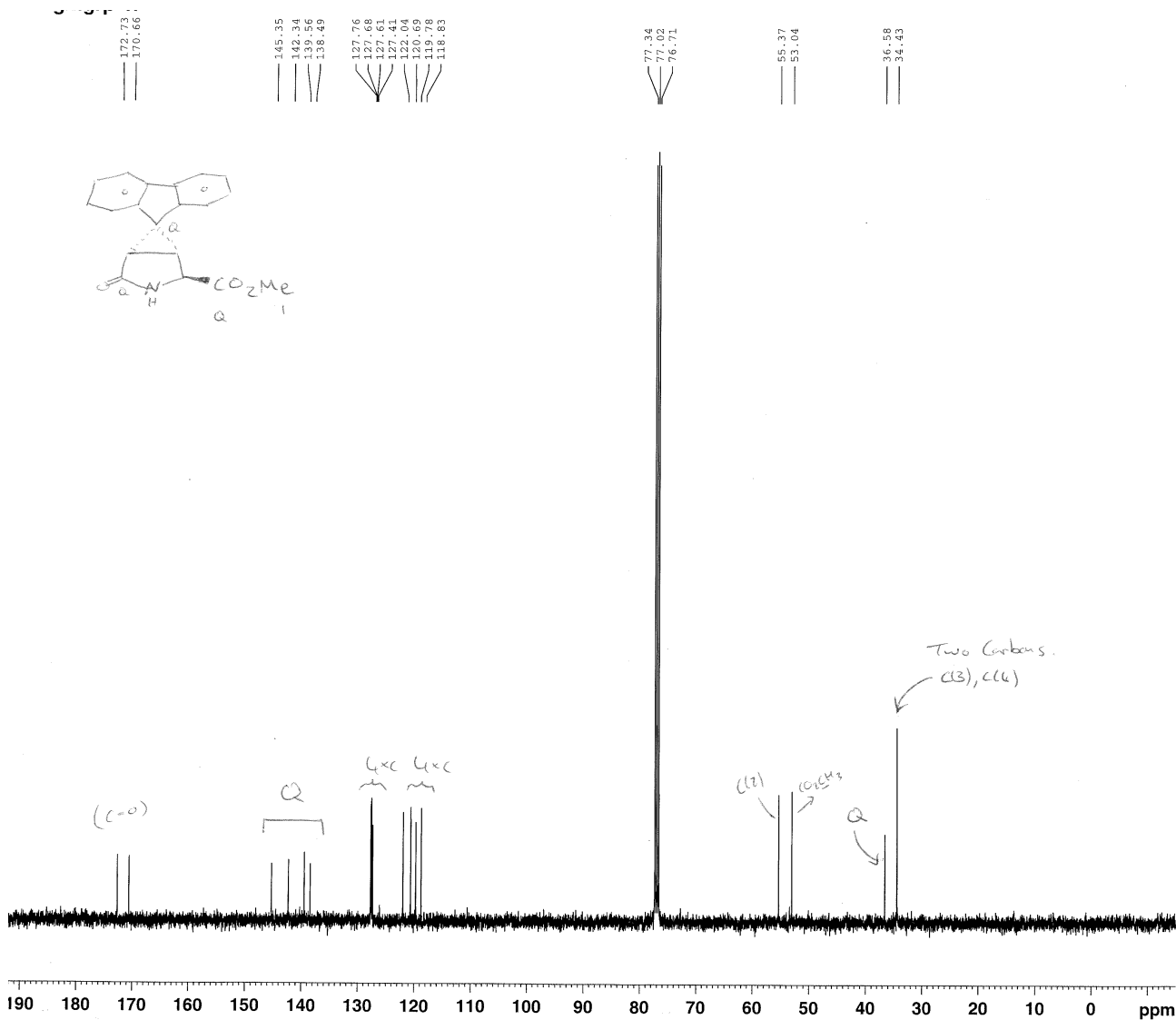
Current Data Parameters
NAME Jul104-2006-47
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060704
Time 15.30
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 7.50 usec
TE 296.1 K
D1 1.0000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 400.2024714 MHz

F2 - Processing parameters
SI 32768
SF 400.200028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

14b



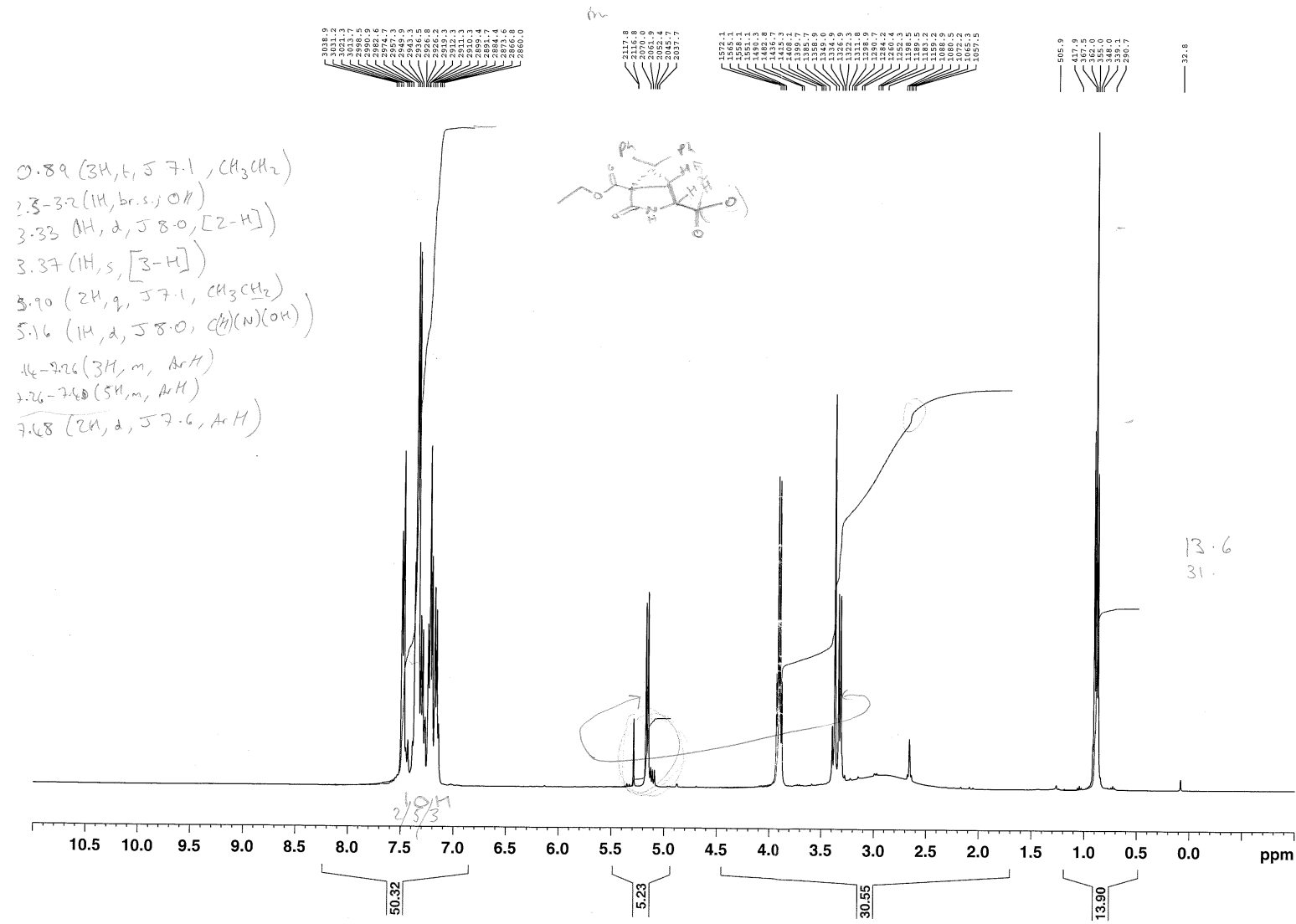
Current Data Parameters
 NAME Jul04-2006-47
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060704
 Time 15.48
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 296.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 19.00 dB
 PL13 25.00 dB
 SFO2 400.2016008 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6303718 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

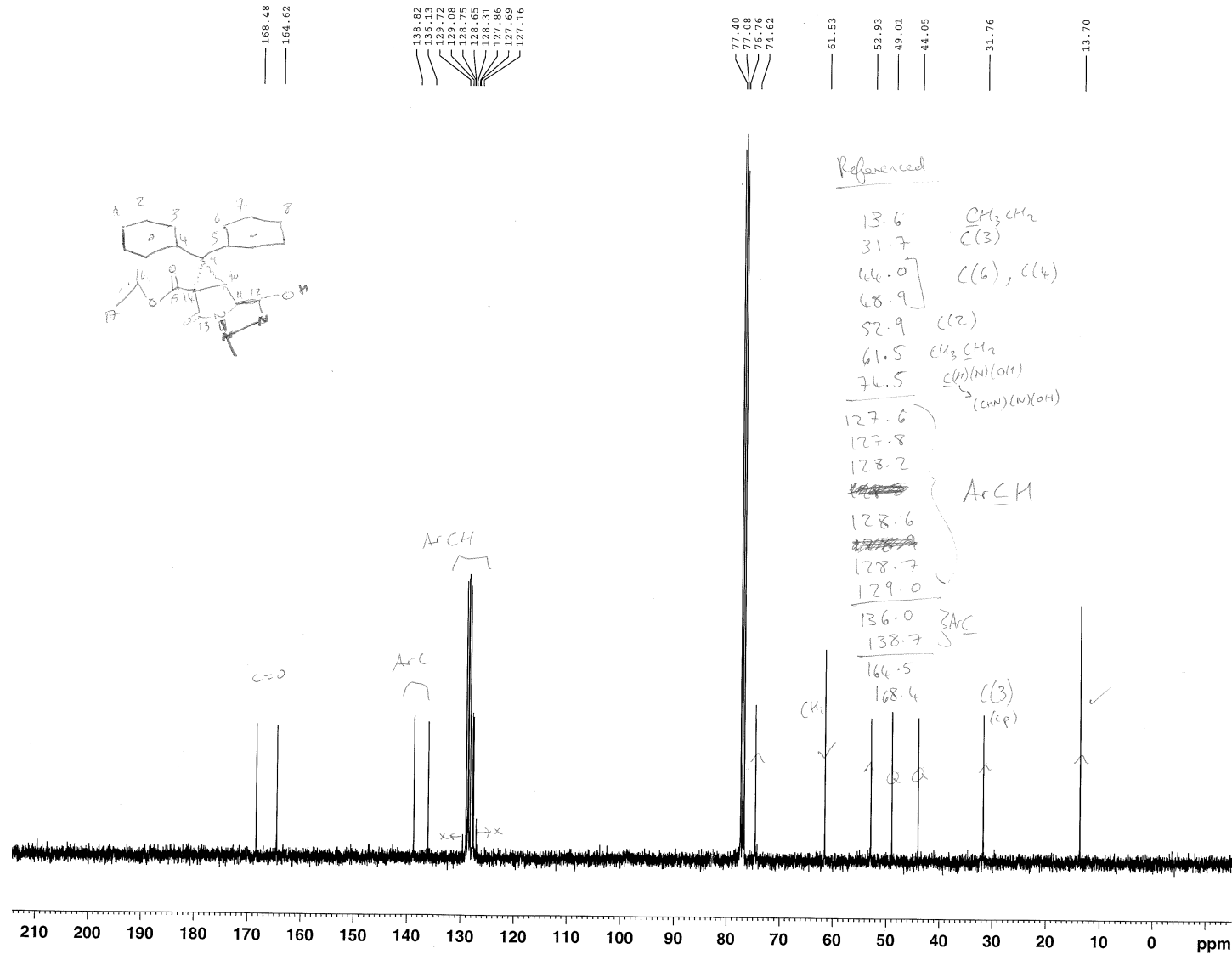


NAME Jul27-2006-25
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060727
Time 19.47
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zg60
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 7.50 usec
TE 296.2 K
D1 1.00000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 400.2024714 MHz

F2 - Processing parameters
SI 32768
SF 400.2000028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



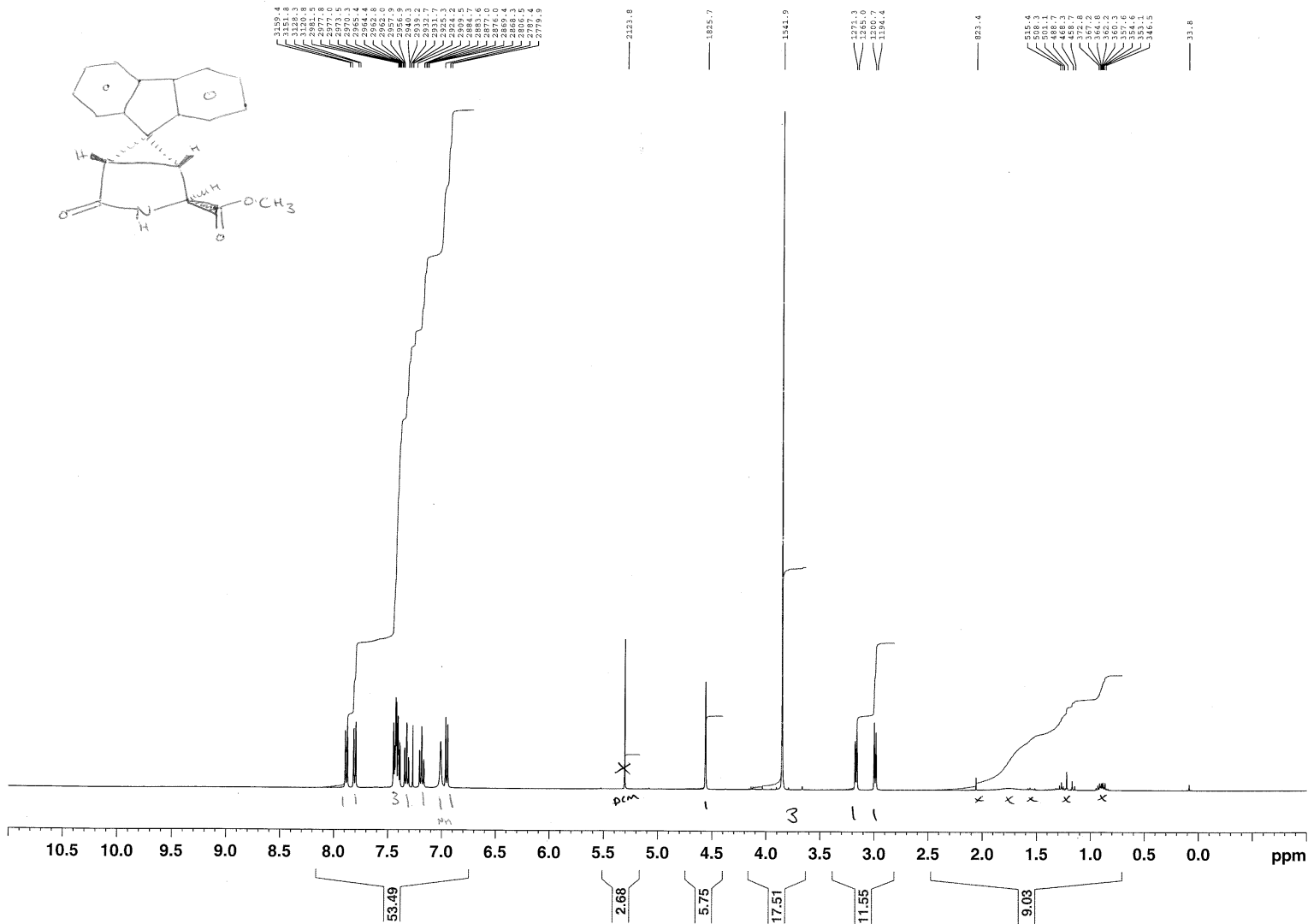
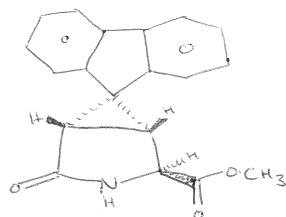
Current Data Parameters
 NAME Jul27-2006-25
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060727
 Time 20.02
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 32768
 DW 19.100 usec
 DE 7.50 usec
 TE 296.6 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 100.6403931 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 19.00 dB
 PL13 25.00 dB
 SFO2 400.2016008 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6303718 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

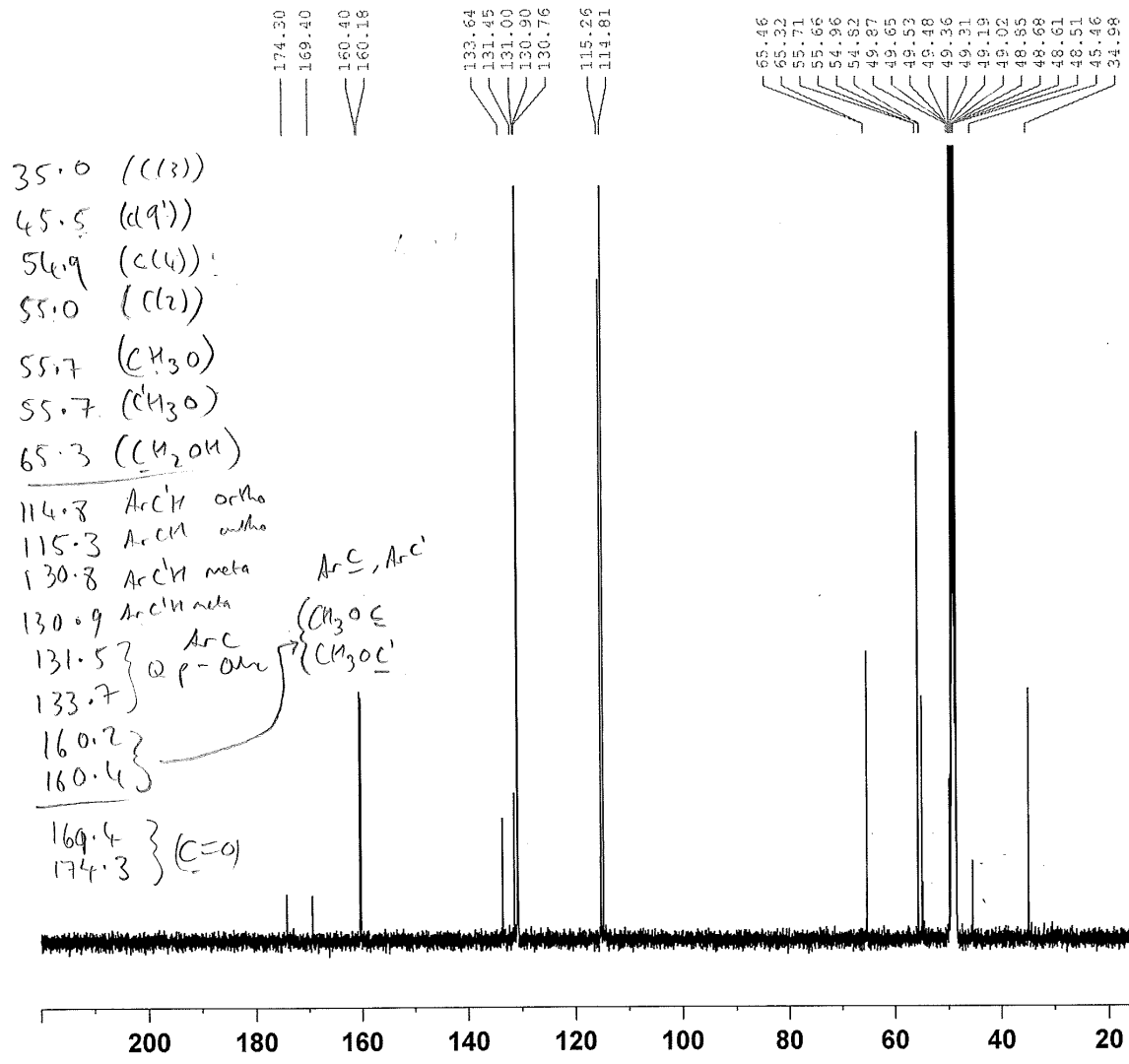


Current Data Parameters
 NAME Jul104-2006-47
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060704
 Time 15.30
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg60
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 228.1
 DW 60.400 usec
 DE 7.50 usec
 TE 296.1 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.2024714 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2000028 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME 1h98300904
 EXPNO 4
 PROCNO 1

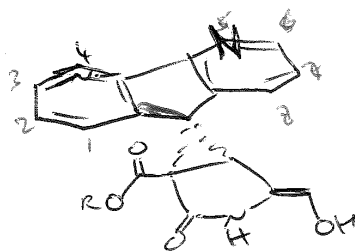
F2 - Acquisition Parameters
 Date_ 20080410
 Time_ 17.29
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD
 NS 256
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 812
 DW 16.000 usec
 DE 20.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -4.40 dB
 SFO1 125.8131151 MHz

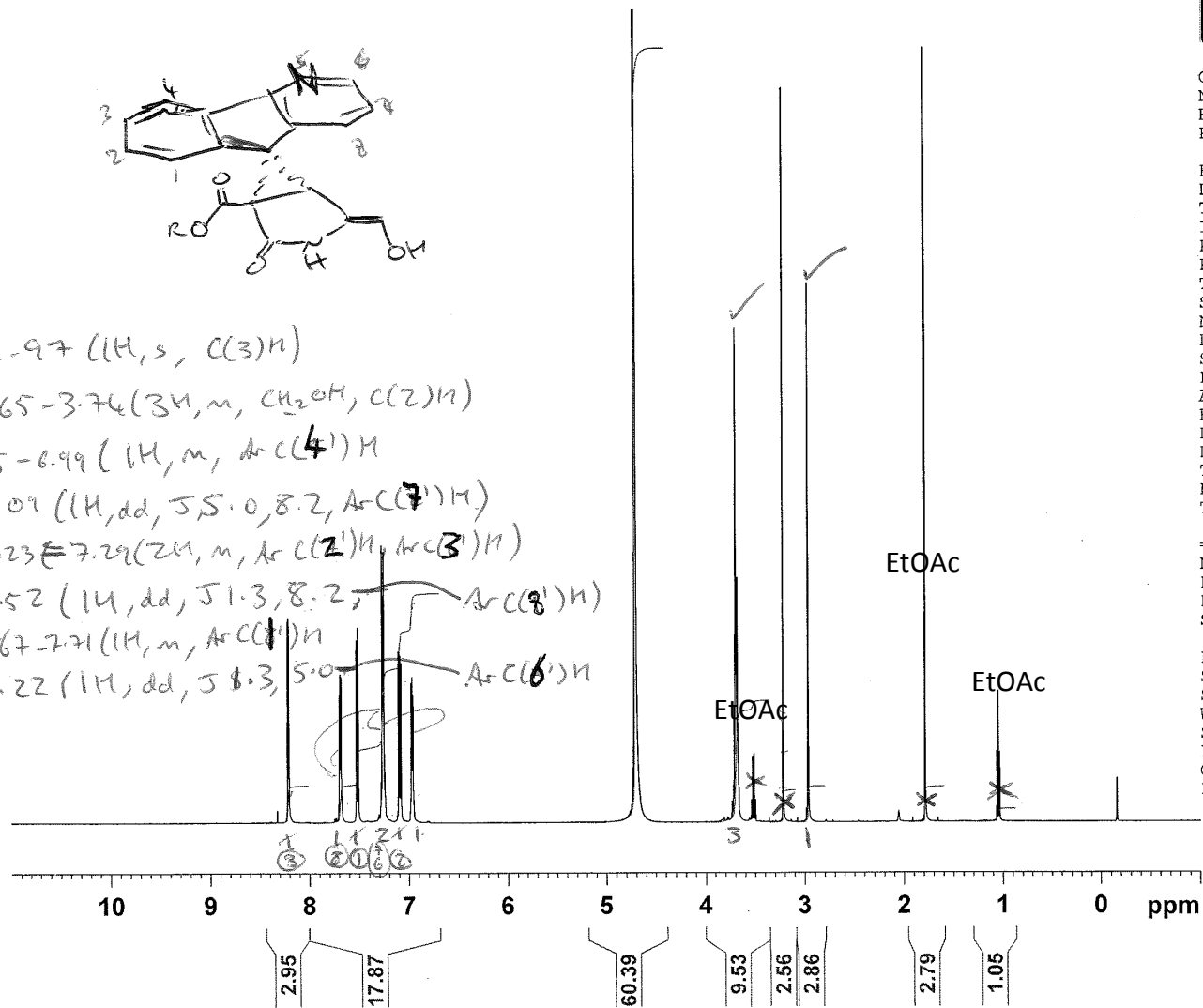
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.40 dB
 PL13 17.00 dB
 PL2 -6.00 dB
 SFO2 500.3020012 MHz

F2 - Processing parameters
 SI 32768
 SF 125.8003564 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

17b



2.97 (1H, s, C(3)H)
 3.65-3.74 (3H, m, CH₂OH, C(2)H)
 4.95-6.99 (1H, m, Ar C(4)H)
 7.09 (1H, dd, J 5.0, 8.2, Ar C(7)H)
 7.23 = 7.29 (2H, m, Ar C(2)H, Ar C(3)H)
 7.52 (1H, dd, J 1.3, 8.2, Ar C(8)H)
 7.67-7.71 (1H, m, Ar C(1)H)
 8.22 (1H, dd, J 8.3, 5.0, Ar C(6)H)



NMR CHEMICAL

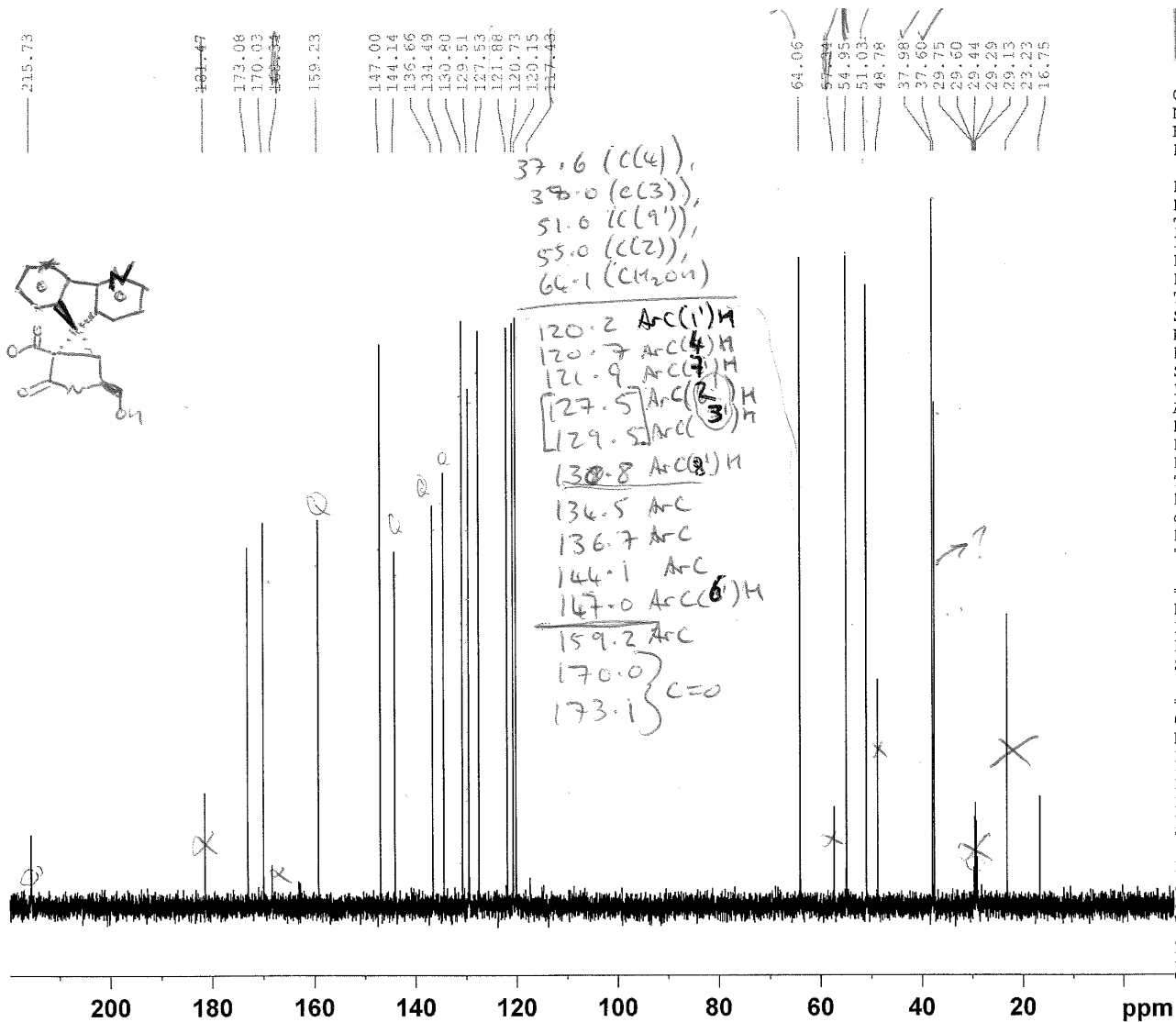
Current Data Parameters
 NAME lh12271208
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20080813
 Time 12.06
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT D₂O ✓
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 4
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.60 usec
 PL1 -6.00 dB
 SF01 500.3030896 MHz

F2 - Processing parameters
 SI 32768
 SF 500.3000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

17b



Current Data Parameters
NAME lh12271208
EXPNO 4
PROCNO 1

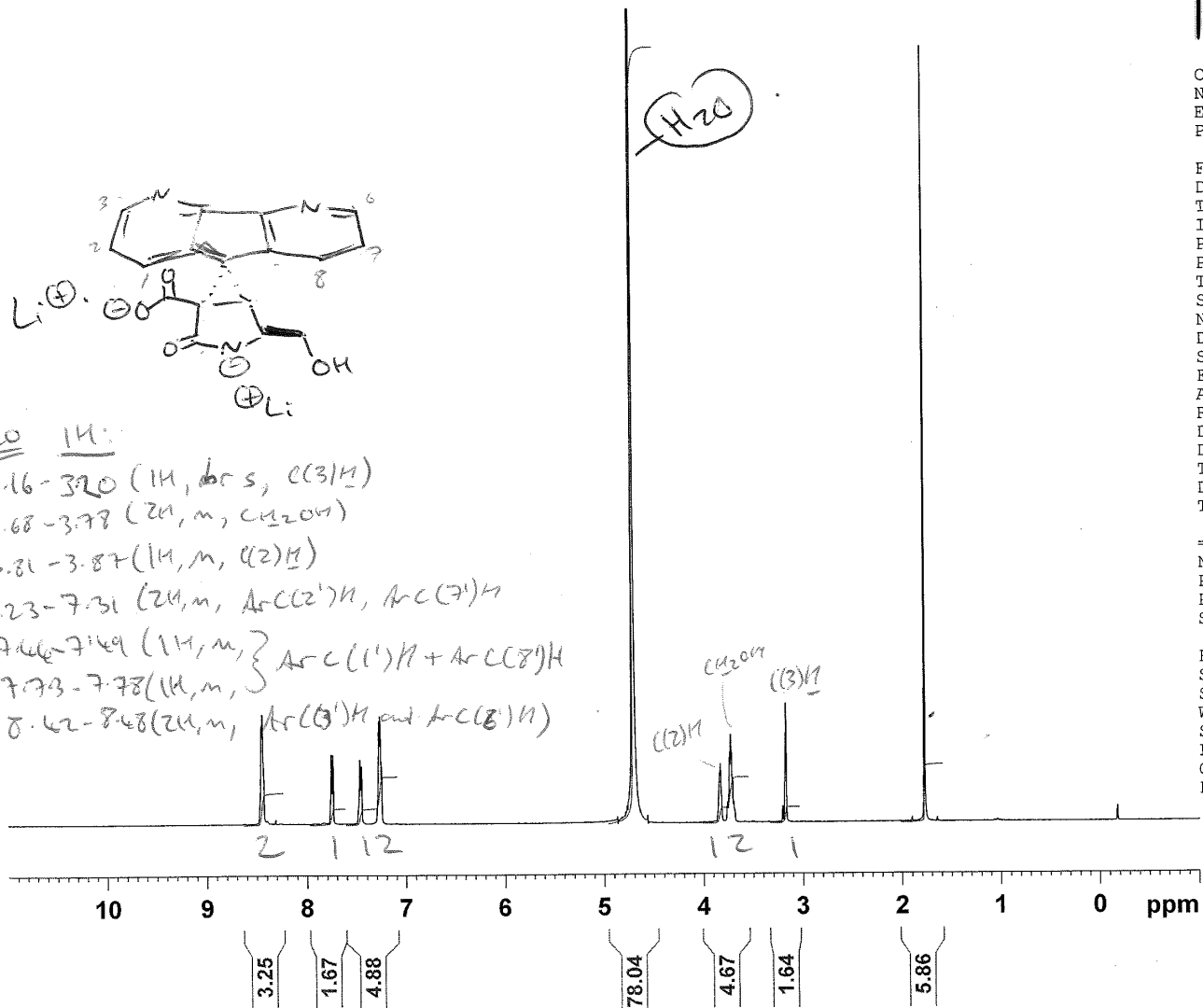
F2 - Acquisition Parameters
Date_ 20080813
Time_ 13.44
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 1820
DW 16.000 usec
DE 20.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -4.40 dB
SFO1 125.8131151 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.40 dB
PL13 17.00 dB
PL2 -6.00 dB
SFO2 500.3020012 MHz

F2 - Processing parameters
SI 32768
SF 125.8005350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

17c



INSTRUMENT

Current Data Parameters
 NAME lh12251208
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080813
 Time_ 6.45
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT D2O
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 7.12
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.60 usec
 PL1 -6.00 dB
 SFO1 500.3030896 MHz

F2 - Processing parameters
 SI 32768
 SF 500.3000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

17c



WIRECHEMIX

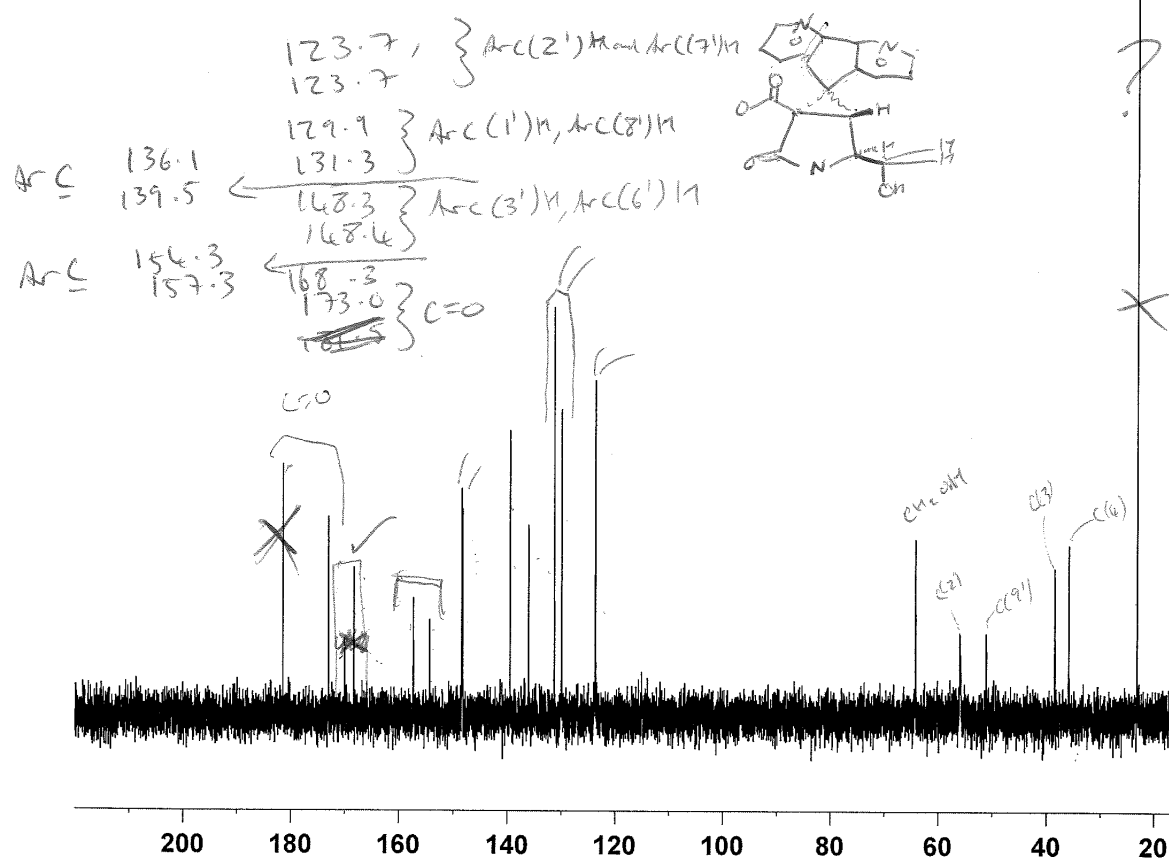
Current Data Parameters
 NAME lh12251208
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080813
 Time_ 8.22
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 1024
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 1820
 DW 16.000 usec
 DE 20.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -4.40 dB
 SFO1 125.8131151 MHz

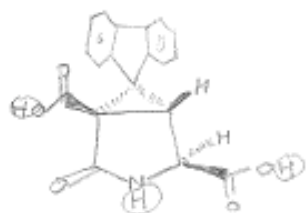
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.40 dB
 PL13 17.00 dB
 PL2 -6.00 dB
 SFO2 500.3020012 MHz

F2 - Processing parameters
 SI 32768
 SF 125.8005350 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



18a

WIRECHEM

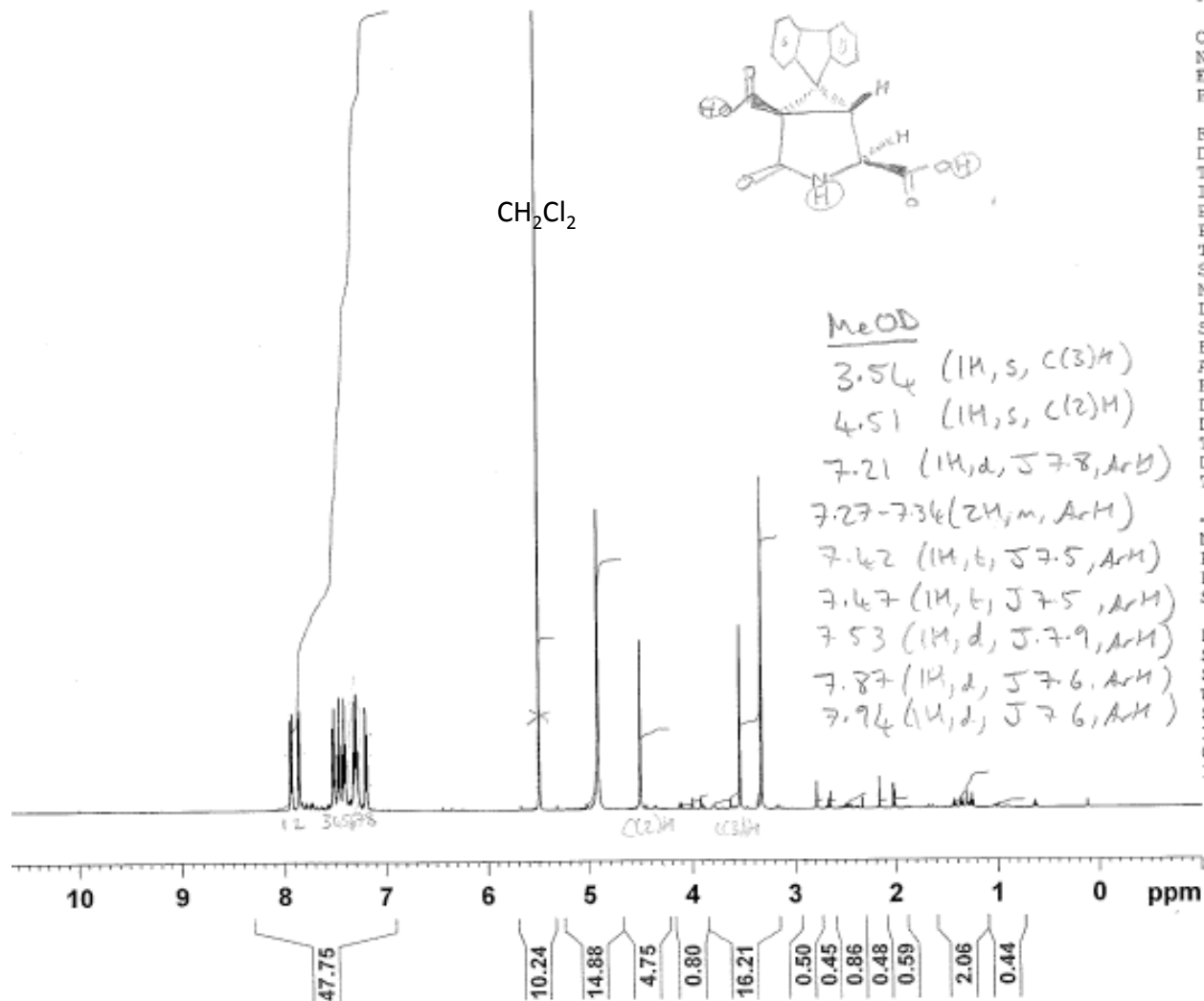


Current Data Parameters
 NAME 1h64341603
 EXPNO 1
 PROCNO 1

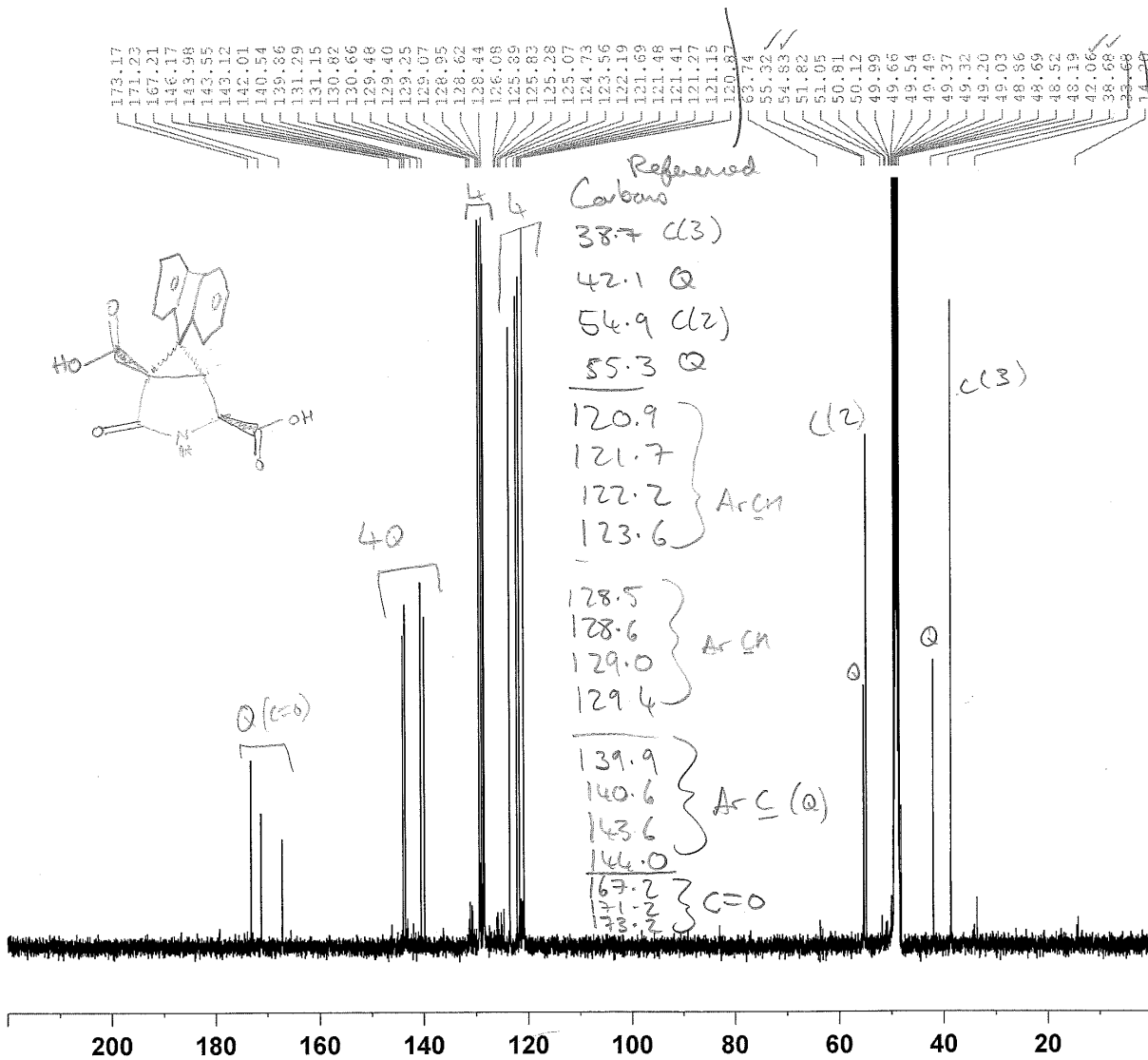
F2 - Acquisition Parameters
 Date_ 20070316
 Time_ 12.41
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT MeOD
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 4
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 1H
 P1 9.60 usec
 PL1 -6.00 dB
 SFO1 500.3030896 MHz

F2 - Processing parameters
 SI 32768
 SF 500.3000000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



18a



WIRECHEM.OX

Current Data Parameters
 NAME 1h64341603
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date 20070316
 Time 13.10
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD MeOD
 NS 256
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 1150
 DW 16.000 usec
 DE 20.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -4.40 dB
 SFO1 125.8131151 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.40 dB
 PL13 17.00 dB
 PL2 -6.00 dB
 SFO2 500.3020012 MHz

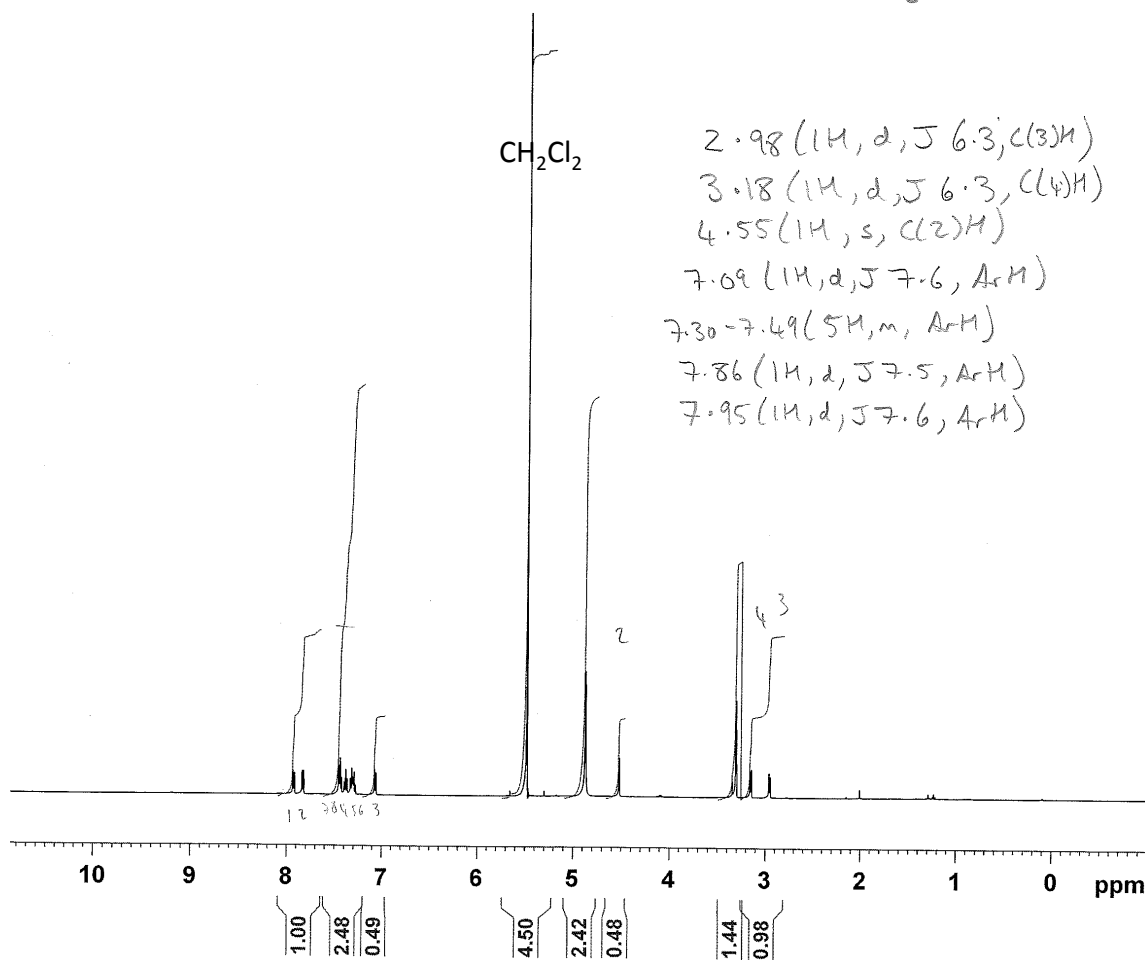
F2 - Processing parameters
 SI 32768
 SF 125.8003564 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

18b

Instrument AVC500
1421 Lawrence Harris 15/3/07



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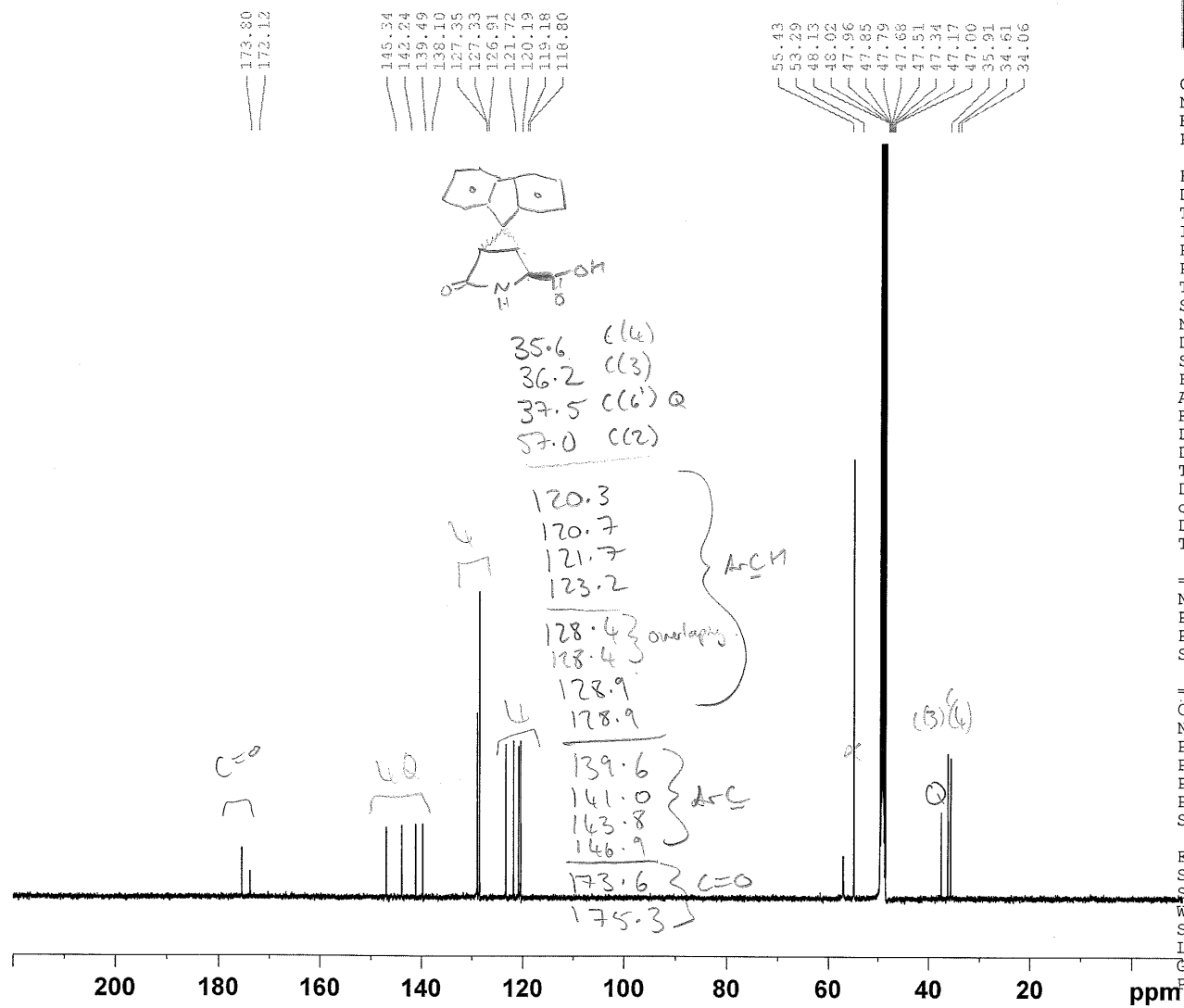
Current Data Parameters
NAME 1h64211503
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070315
Time 14.24
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 4
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TDO 1

==== CHANNEL f1 =====
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 500.3030896 MHz

F2 - Processing parameters
SI 32768
SF 500.2992393 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

18b



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Current Data Parameters
NAME lh64211503
EXPNO 4
PROCNO 1

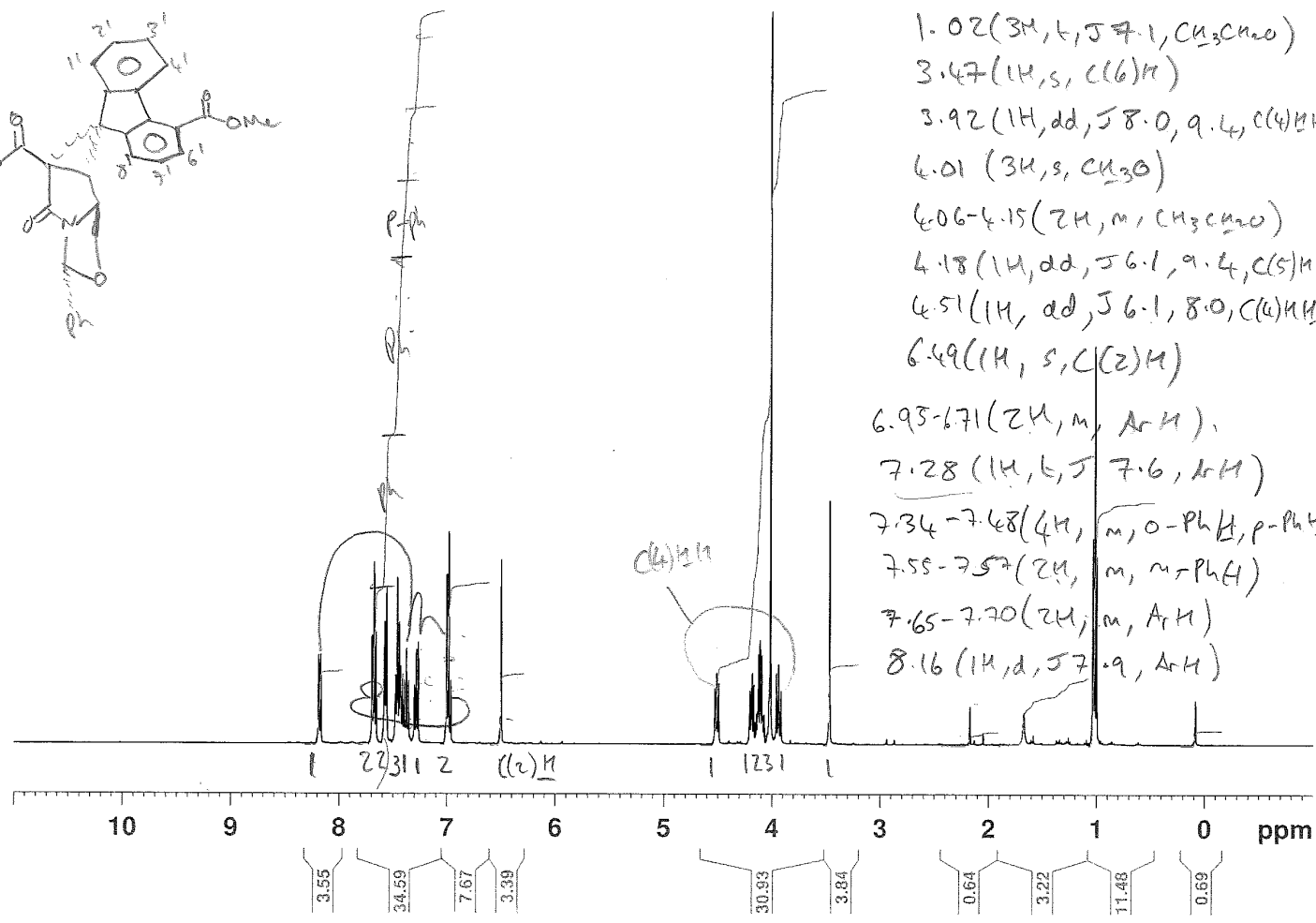
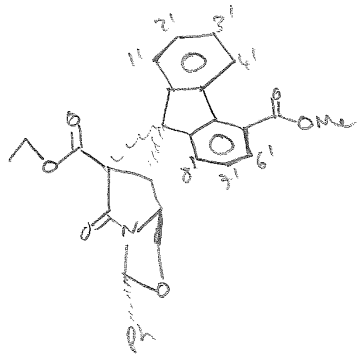
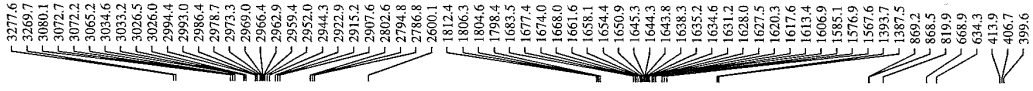
F2 - Acquisition Parameters
Date_ 20070315
Time 14.54
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 1290
DW 16.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -4.40 dB
SFO1 125.8131151 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.40 dB
PL13 17.00 dB
PL2 -6.00 dB
SFO2 500.3020012 MHz

F2 - Processing parameters
SI 32768
SF 125.8001512 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

19a



Current Data Parameters

NAME May02-2009-2

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20090502

Time 11.14

INSTRUM dpx400

PROBHD 5 mm Dual 1H/1

PULPROG zg60

TD 32768

SOLVENT CDC13

NS 16

DS 2

SWH 5592.841 Hz

FIDRES 0.170680 Hz

AQ 2.9295092 sec

RG 71.8

DW 89.400 usec

DE 17.00 usec

TE 300.0 K

D1 1.00000000 sec

==== CHANNEL f1 =====

NUC1 1H

P1 7.30 usec

PL1 0.00 dB

SFO1 400.1320007 MHz

F2 - Processing parameters

SF 32768

SF 400.1300182 MHz

WDW EM

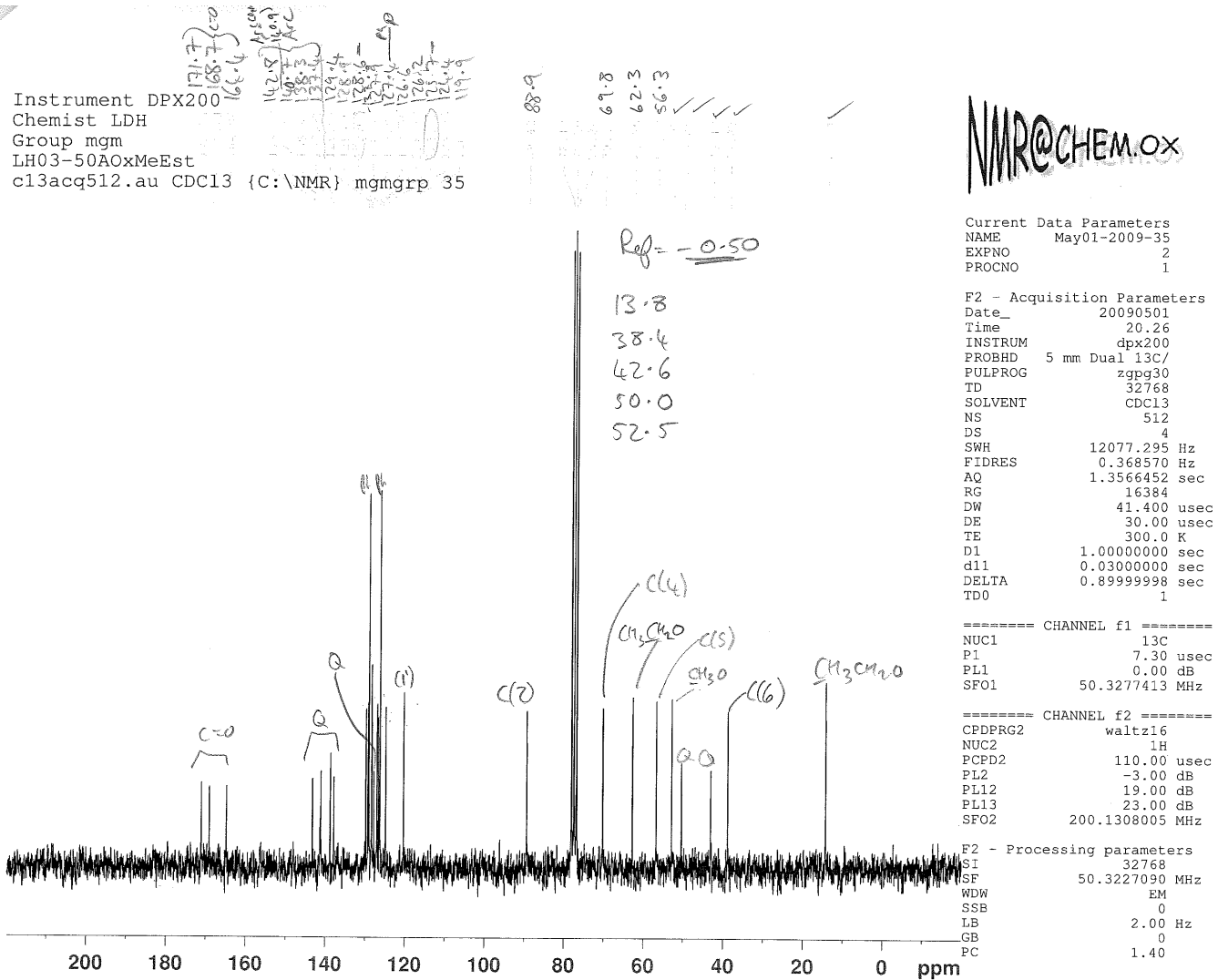
SSB 0

LB 0.30 Hz

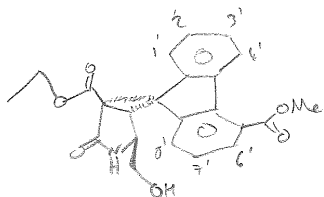
GB 0

PC 0.60

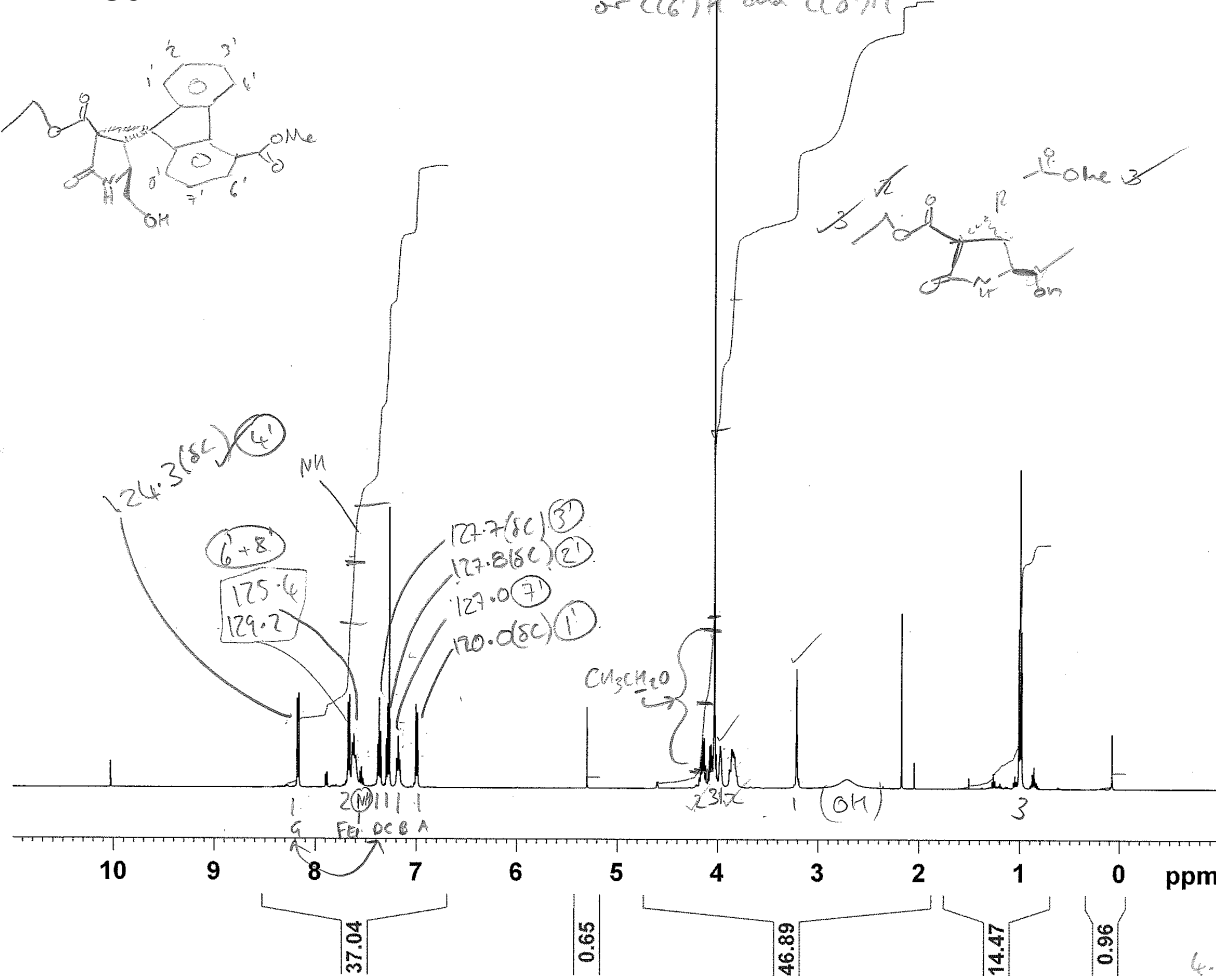
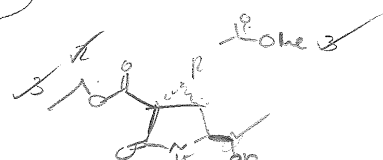
19a



Lh05-80
20a



C(6') and C(8') are
or C(6')H and C(8')H



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NAME lh41040505
EXPNO 1
PROCNO 1
Date 20090505
Time 20.25
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 4
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
PL1W 15.19999981 W
SFO1 500.3030896 MHz
SI 32768
SF 500.3000240 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1.00 (3H, t, J 7.1, CH₃CH₂O)

2.71 (1H, br s, OH)

3.21 (1H, s, C(3)H)

3.74-3.90 (2H, m, C(2)CH₂OH)

3.96 (1H, s, C(2)H)

4.03 (3H, s, CO₂CH₃)

4.01-4.18 (2H, m, CH₃CH₂O)

Aromatic: 7.00 (1H, d, J 7.6, Ar-C(1')H), 7.18 (1H, t, J 7.7, Ar-C(7')H), 7.27-7.31 (1H, m, Ar-C(2')H), 7.37 (1H, t, J 7.3, Ar-C(3')H)
7.60 (1H, br s, NH), 7.63 (1H, d, J 7.9, Ar-C(6' or 8')H), 7.67 (1H, d, J 7.9, Ar-C(6' or 8')H), 8.18 (1H, d, J 7.6, Ar-C(4')H);

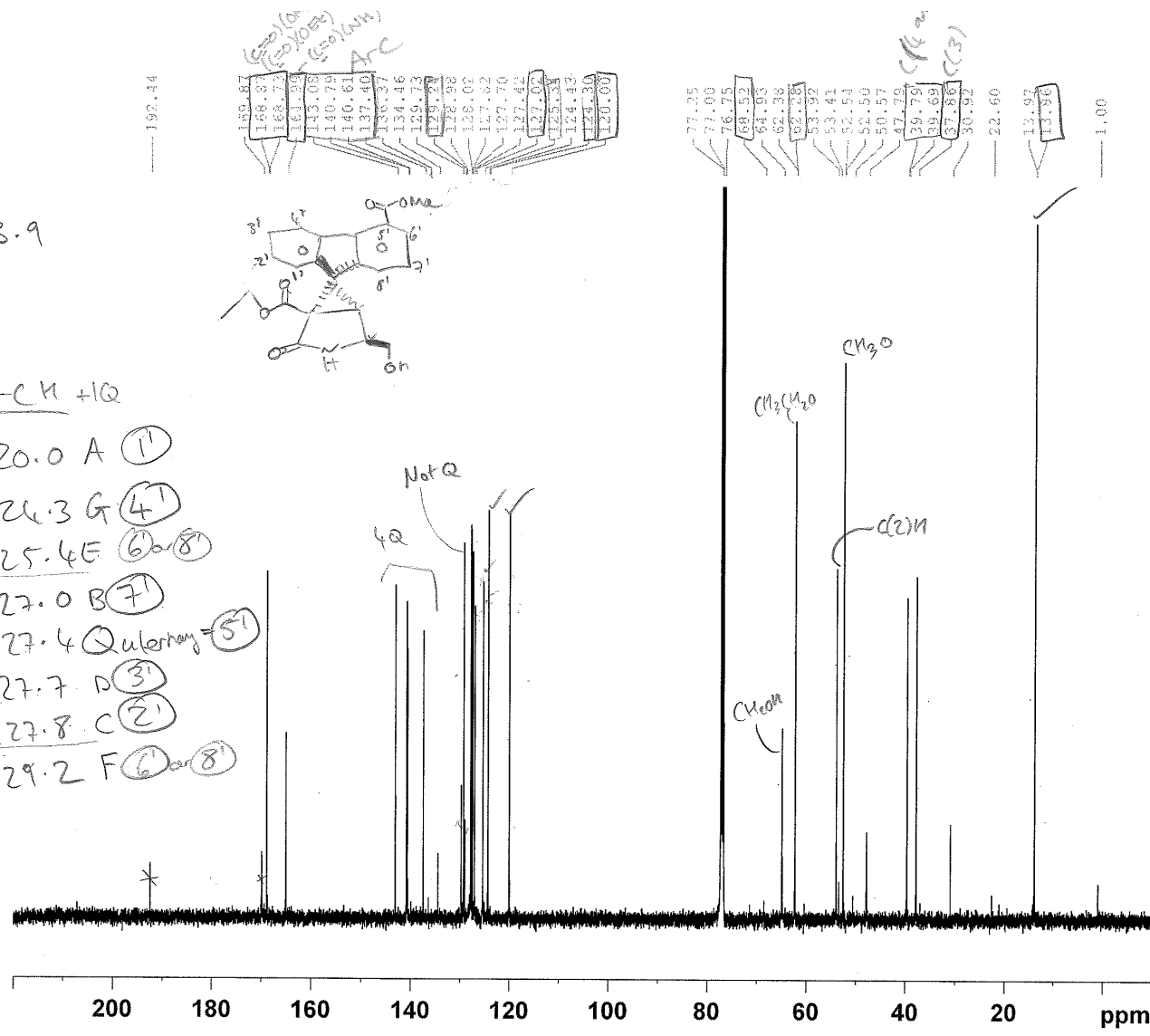
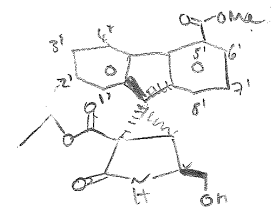
20a

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13.9

Arc H + 1Q

- 120.0 A (1)
- 126.3 G (4)
- 125.4 E (6, 8)
- 127.0 B (7)
- 127.4 Q (Quaternary) (5)
- 127.7 D (3)
- 127.8 C (2)
- 129.2 F (6 or 8)



```

NAME      1h41040505
EXPNO     3
PROCNO    1
Date_     20090505
Time      20.49
INSTRUM   avc500
PROBHD    5 mm CPDUL 13C
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         256
DS         2
SWH        31250.000 Hz
FIDRES     0.476837 Hz
AQ         1.0486259 sec
RG         1820
DW         16.000 usec
DE         20.00 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

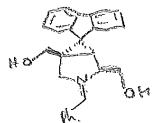
===== CHANNEL f1 =====
NUC1      13C
P1         8.00 usec
PL1        -4.40 dB
PL1W      28.15752029 W
SFO1      125.8131151 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        -6.00 dB
PL12       12.40 dB
PL13       17.00 dB
PL2W      15.19999981 W
PL12W     0.21970686 W
PL13W     0.07618046 W
SFO2      500.3020012 MHz
SI         32768
SF         125.8005438 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

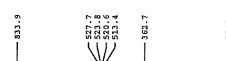
21a

Instrument DQX400
 Chemist LDH
 Group MGM
 LH03-Purandiolyoufool
 h1acq.au CDC13 e:\ mgmgrp 13



After column 2 after 1st decomposition

≈ 70mg



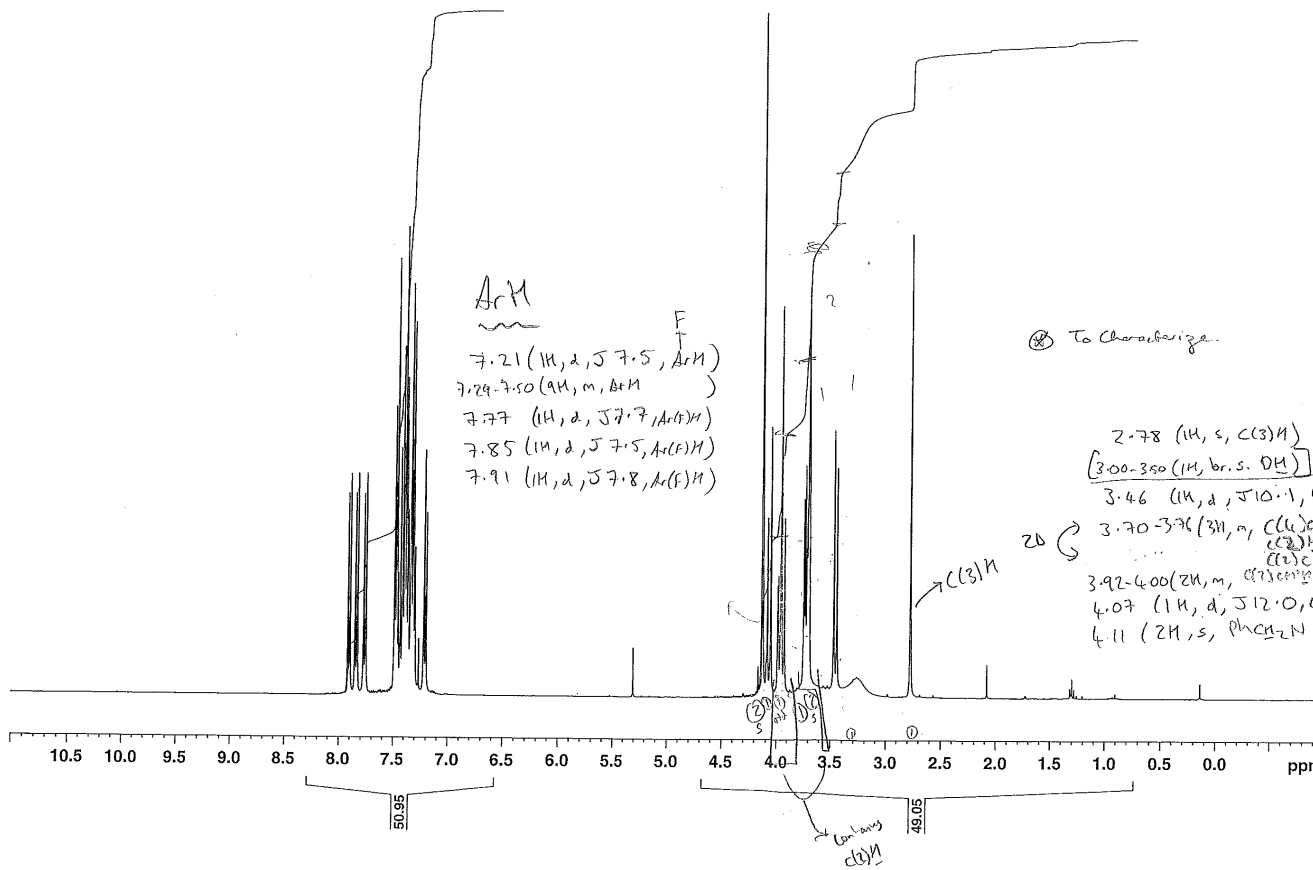
NMR@CHEM.OX

Current Data Parameters
 NAME Dec19-2006-13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061219
 Time 23.38
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 40.3
 DW 60.400 usec
 DE 7.50 usec
 TE 295.7 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUCL1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 400.2024714 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2000028 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



ArH
 7.21 (1H, d, J 7.5, ArH)
 7.29-7.50 (9H, m, ArH)
 7.77 (1H, d, J 7.7, ArH)
 7.85 (1H, d, J 7.5, ArH)
 7.91 (1H, d, J 7.8, ArH)

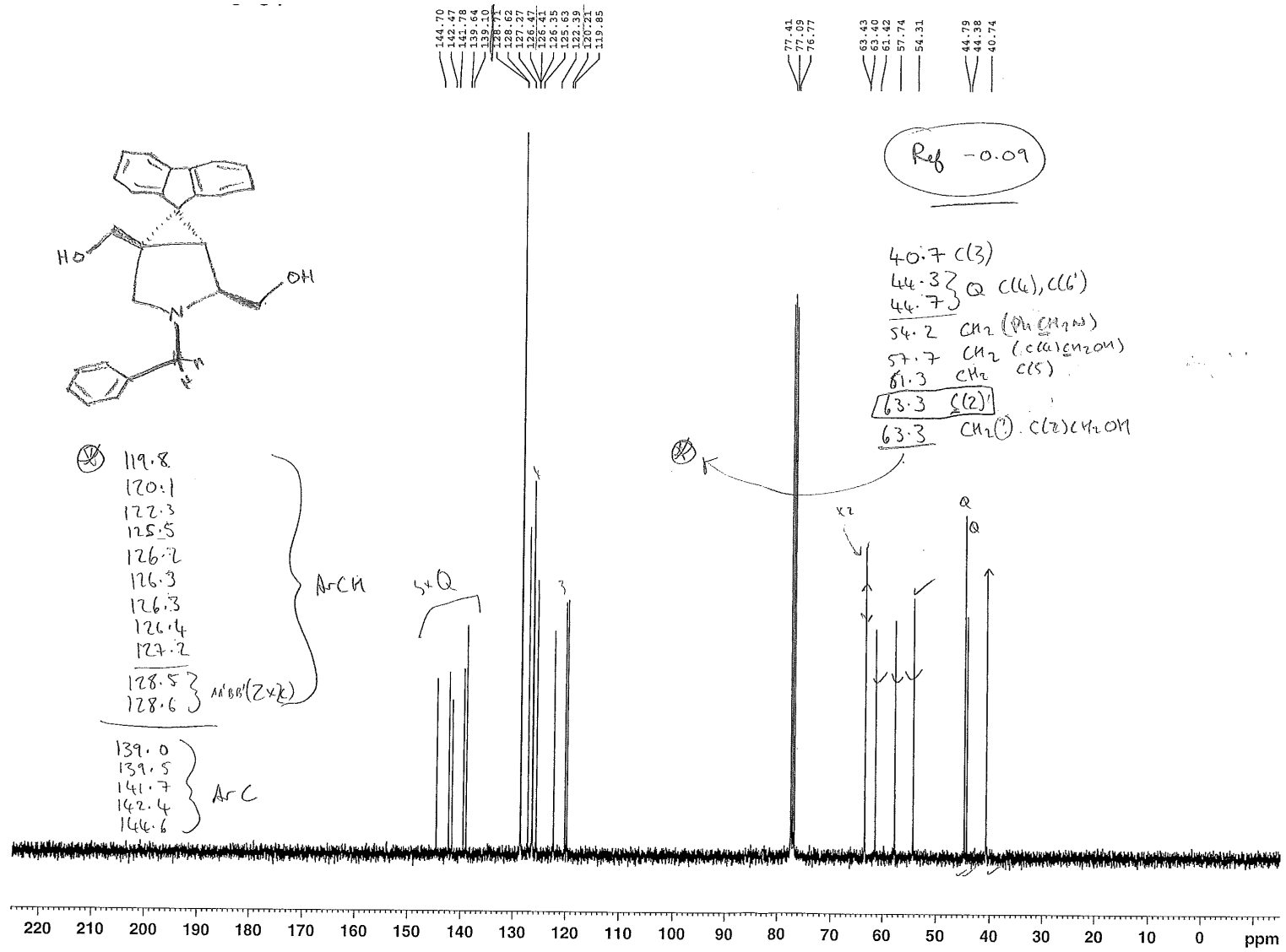
⊗ To Characterize.

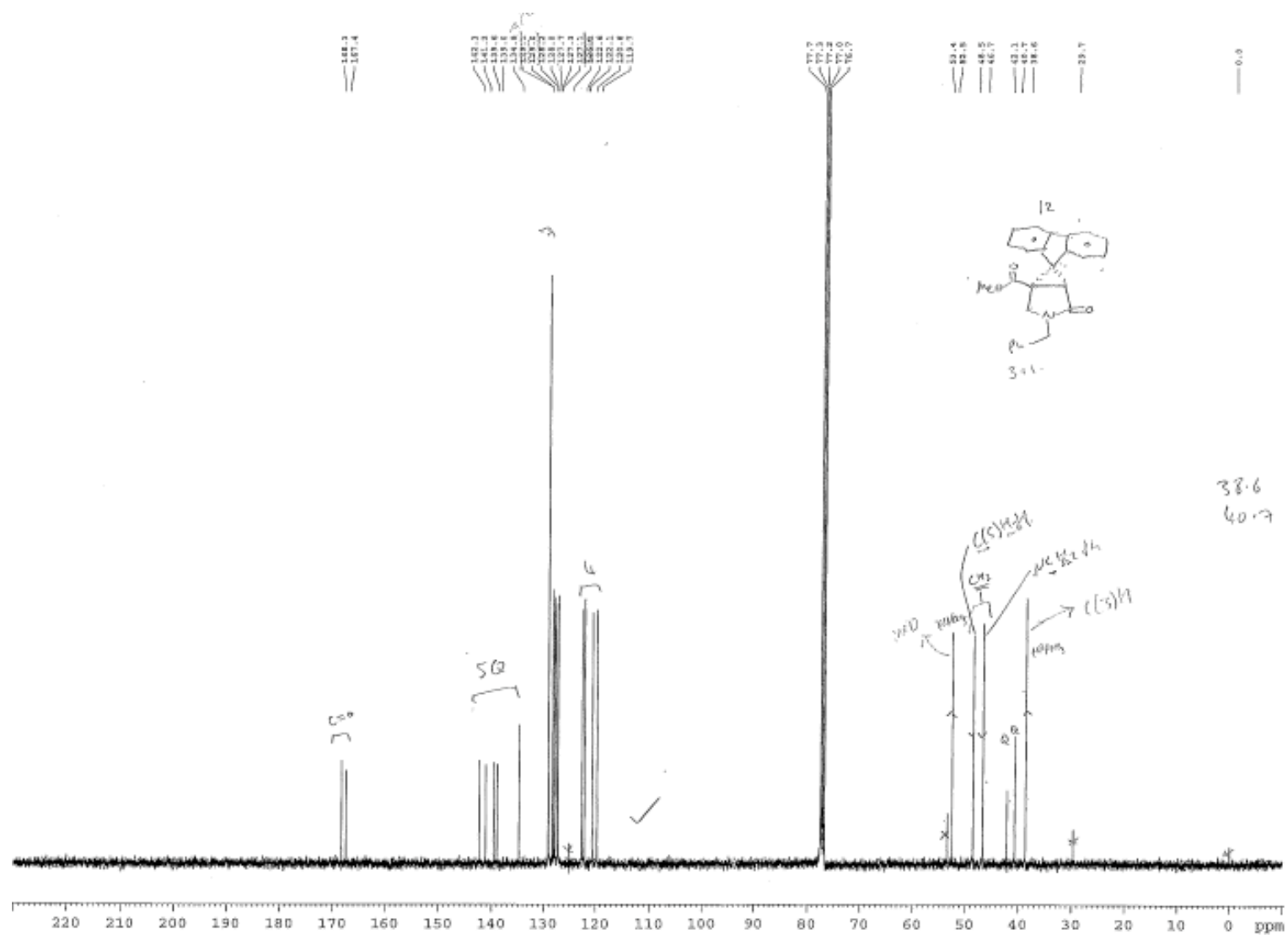
2.78 (1H, s, C(3)H)
 3.00-3.50 (1H, br. s. OH) → Other spectrum for more details
 3.46 (1H, d, J 10.1, (H)CH₂OH) ⊗
 3.70-3.76 (3H, m, C(6)CH₂OH) ⊗
 (2)CH₂OH
 3.92-4.00 (2H, m, C(7)CH₂OH, C(8)H) ⊗ → scalar 3.76 (m, d, J)
 4.07 (1H, d, J 12.0, (H)H) ⊗
 4.11 (2H, s, PhCH₂N) ⊗

→ C(3)H

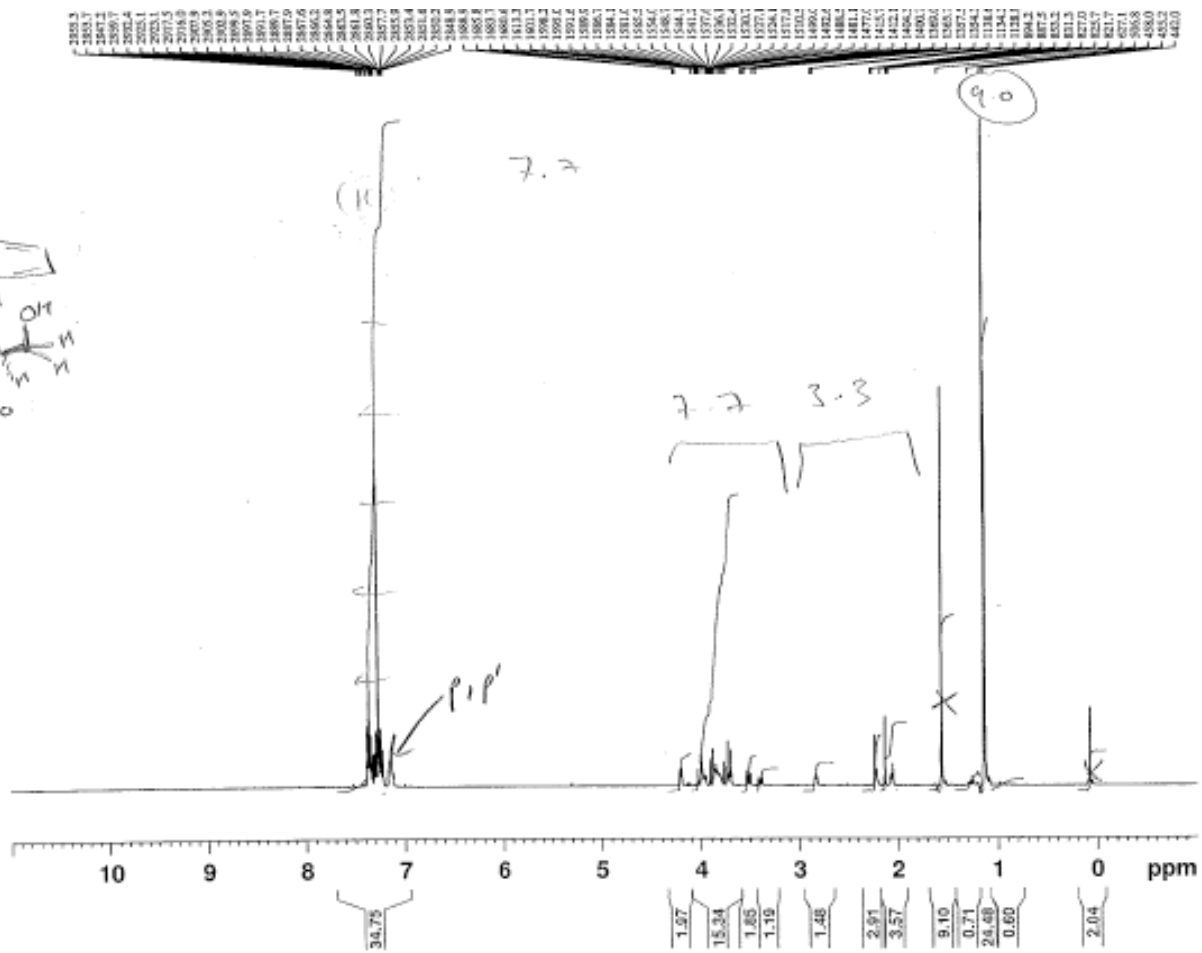
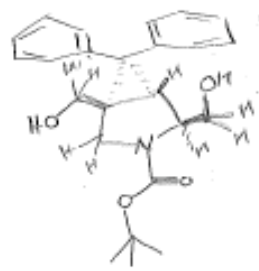
contains CDCl₃

21a





24a



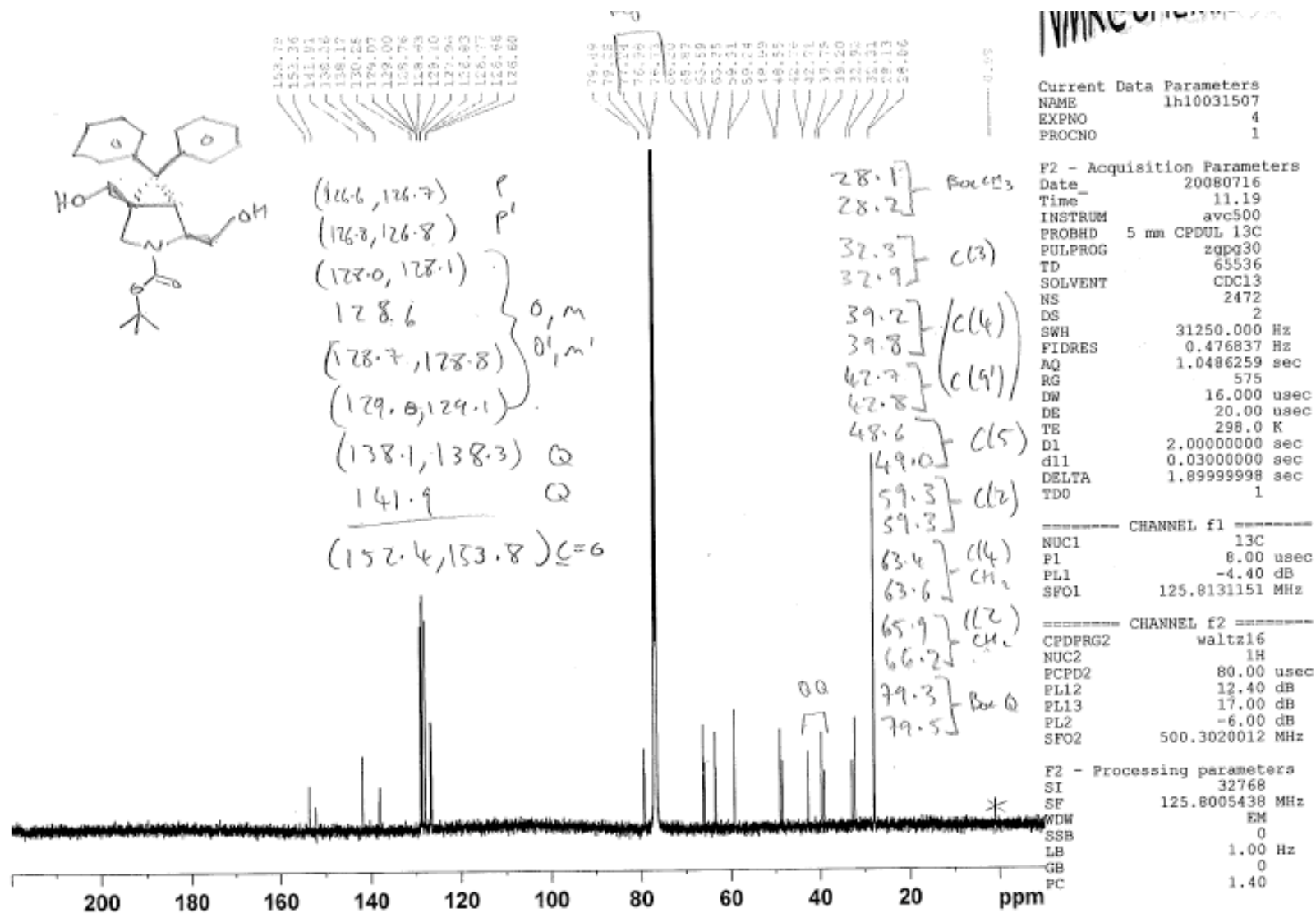
Current Data Parameters
 NAME Jul15-2008-36
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080715
 Time 12.44
 INSTRUM dpx400
 PROBHD 5 mm Dual 1H/1
 PULPROG zg60
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 322.5
 DW 89.400 usec
 DE 17.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.30 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

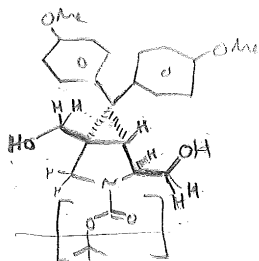
F2 - Processing parameters
 SI 32768
 SF 100.1300182 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 0.60

24a



24b

Instrument AVC500
LAURENCE HARRIS 9894 14/04/08



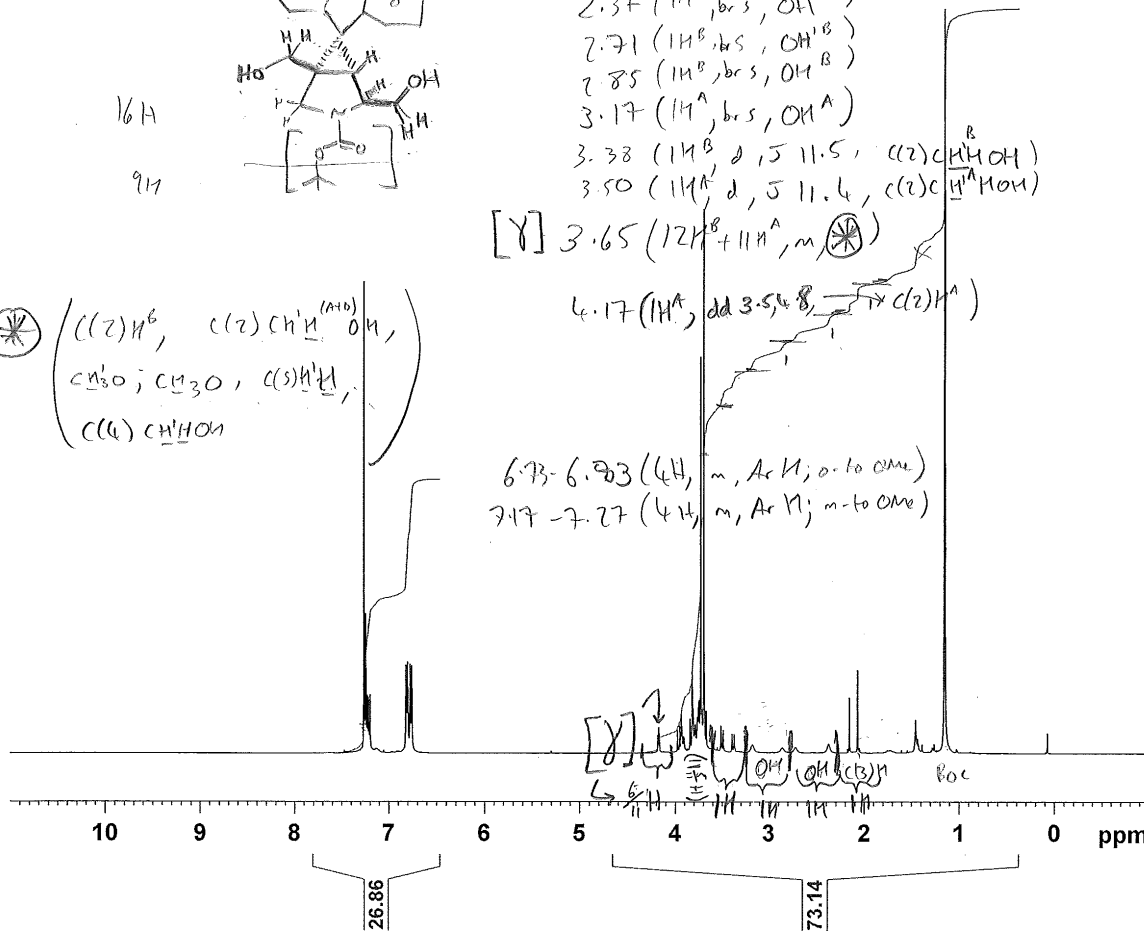
(*) (C(2)H^B, C(2)CH^(A+B)OH,
CH₃O, CH₃O, C(5)H^AH,
C(4)CH^AH₂O)

1.14 (9H^D, s, (CH₃)₃CO)
1.15 (9H^A, s, (CH₃)₃CO)
2.06 (1H^A, s, C(3)H)
2.15 (1H^B, s, C(3)H)
2.37 (1H^A, br s, OH^A)
2.71 (1H^B, br s, OH^B)
2.85 (1H^B, br s, OH^B)
3.17 (1H^A, br s, OH^A)
3.38 (1H^B, d, J 11.5, C(2)CH^BOH)
3.50 (1H^A, d, J 11.4, C(2)CH^AH₂O)

[γ] 3.65 (12H^B + 11H^A, m, *)

4.17 (1H^A, dd 3-5, 8, C(2)H^A)

6.73-6.93 (4H, m, Ar H; o-to OMe)
7.17-7.27 (4H, m, Ar H; m-to OMe)



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Current Data Parameters
NAME lh98941404
EXPNO 1
PROCNO 1

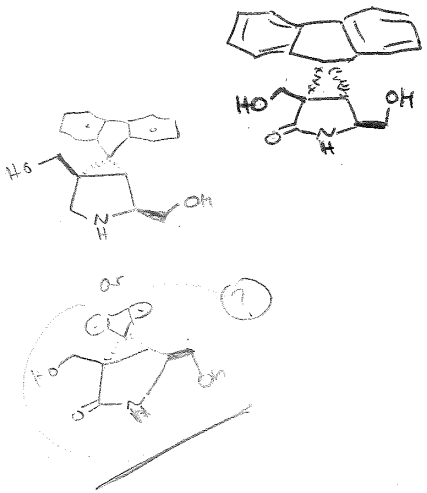
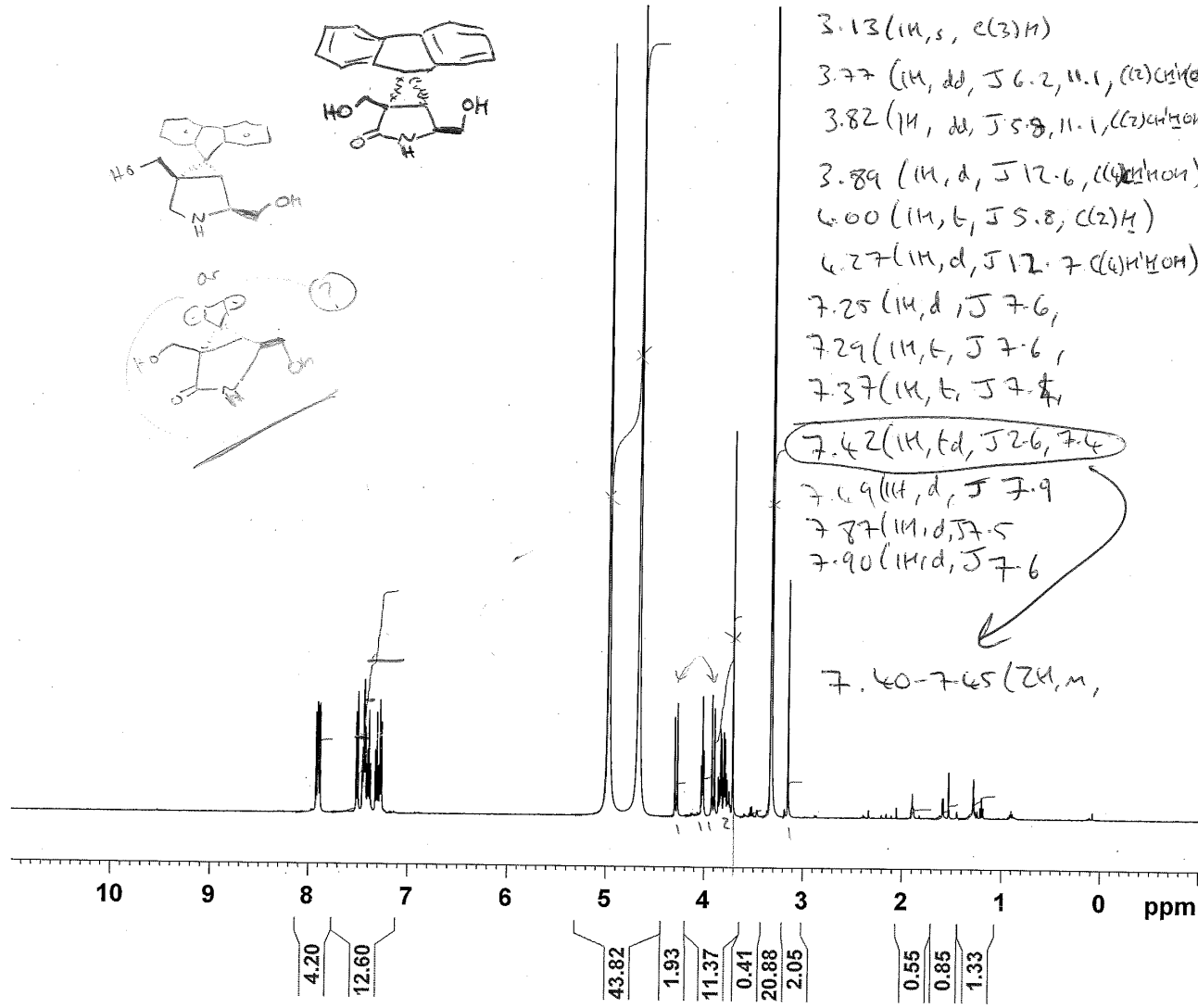
F2 - Acquisition Parameters
Date_ 20080415
Time_ 17.54
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 4
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 500.3030896 MHz

F2 - Processing parameters
SI 32768
SF 500.3000240 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

2.1

24c



3.13 (m, s, c(3)H)
 3.77 (m, dd, J 6.2, 11.1, (2)CH₂)
 3.82 (m, dd, J 5.8, 11.1, (2)CH₂)
 3.89 (m, d, J 12.6, (4)H₂O)
 6.00 (m, t, J 5.8, (2)H)
 6.27 (m, d, J 12.7, (4)H₂O)
 7.25 (m, d, J 7.6,
 7.29 (m, t, J 7.6,
 7.37 (m, t, J 7.6,
 7.42 (m, td, J 2.6, 7.4
 7.49 (m, d, J 7.9
 7.87 (m, d, J 7.5
 7.90 (m, d, J 7.6
 7.40-7.45 (2H, m,

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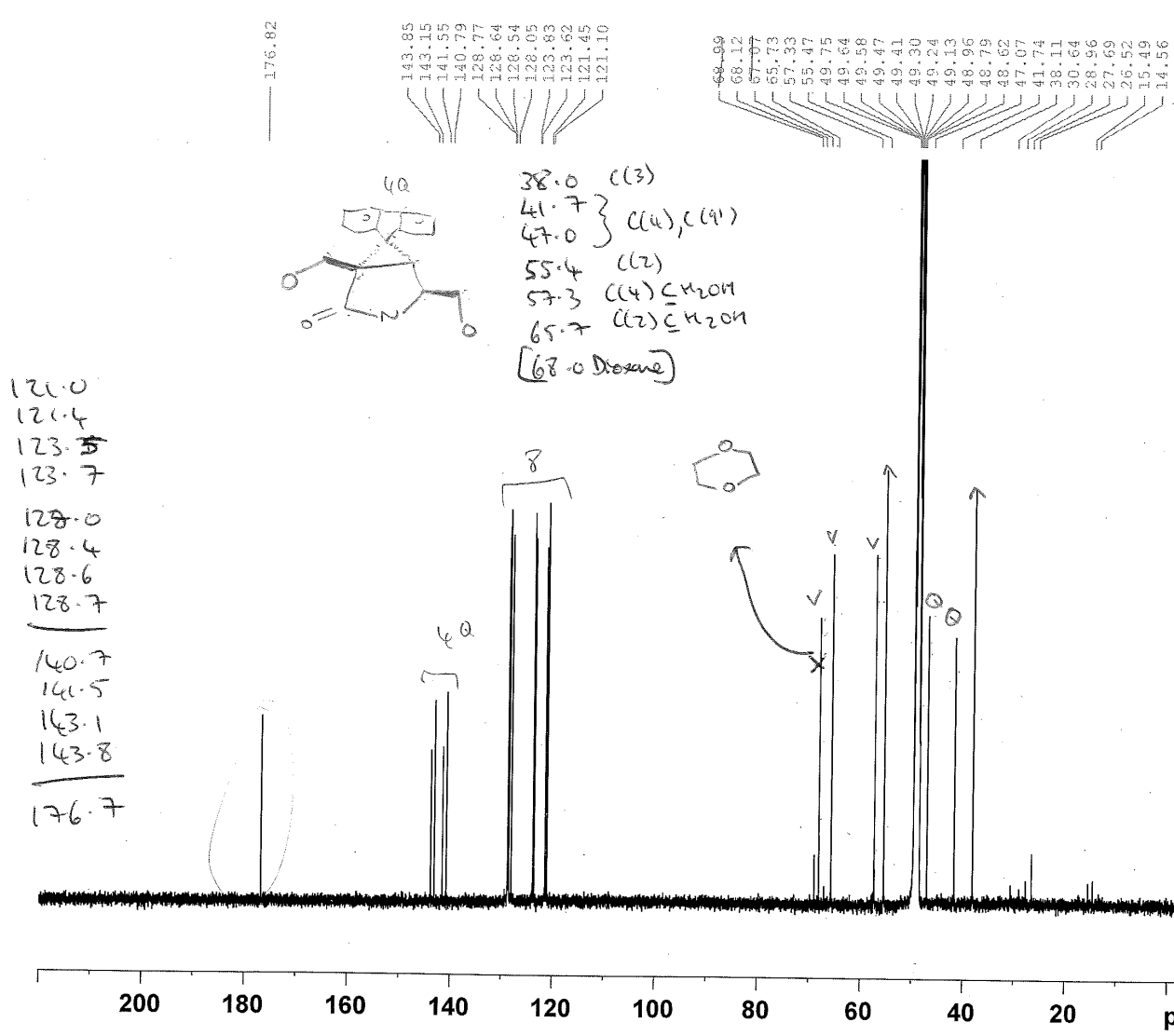
Current Data Parameters
 NAME 1h79651909
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070919
 Time_ 16.37
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT MeOD
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 4
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.60 usec
 PL1 -6.00 dB
 SFO1 500.3030896 MHz

F2 - Processing parameters
 SI 32768
 SF 500.300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

24c



WIRECENTER

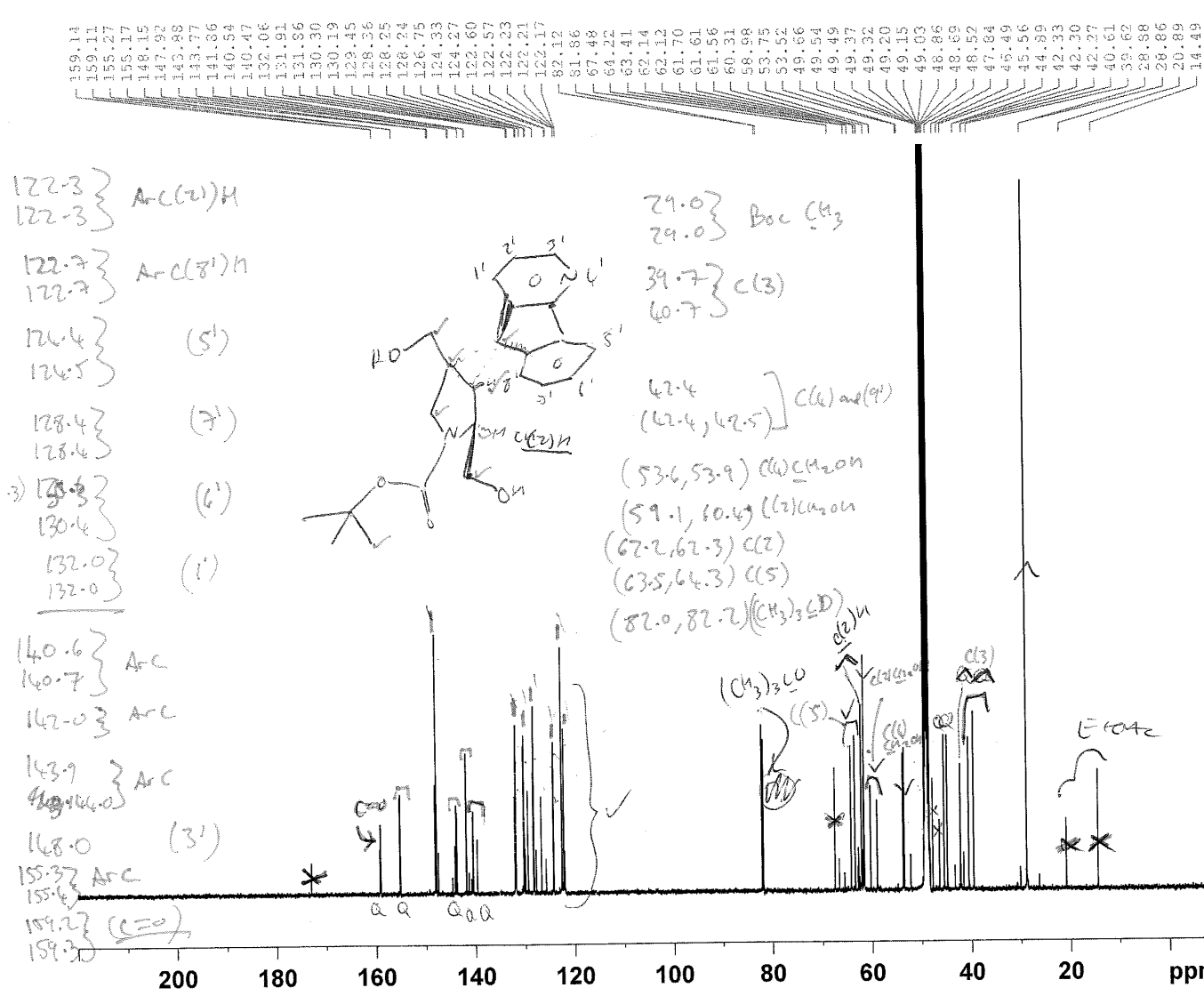
Current Data Parameters
 NAME lh79651909
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070919
 Time 20.53
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD
 NS 4096
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 1820
 DW 16.000 usec
 DE 20.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -4.40 dB
 SFO1 125.8131151 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.40 dB
 PL13 17.00 dB
 PL2 -6.00 dB
 SFO2 500.3020012 MHz

F2 - Processing parameters
 SI 32768
 SF 125.8003564 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME 1h15181909
 EXPNO 2
 PROCNO 1

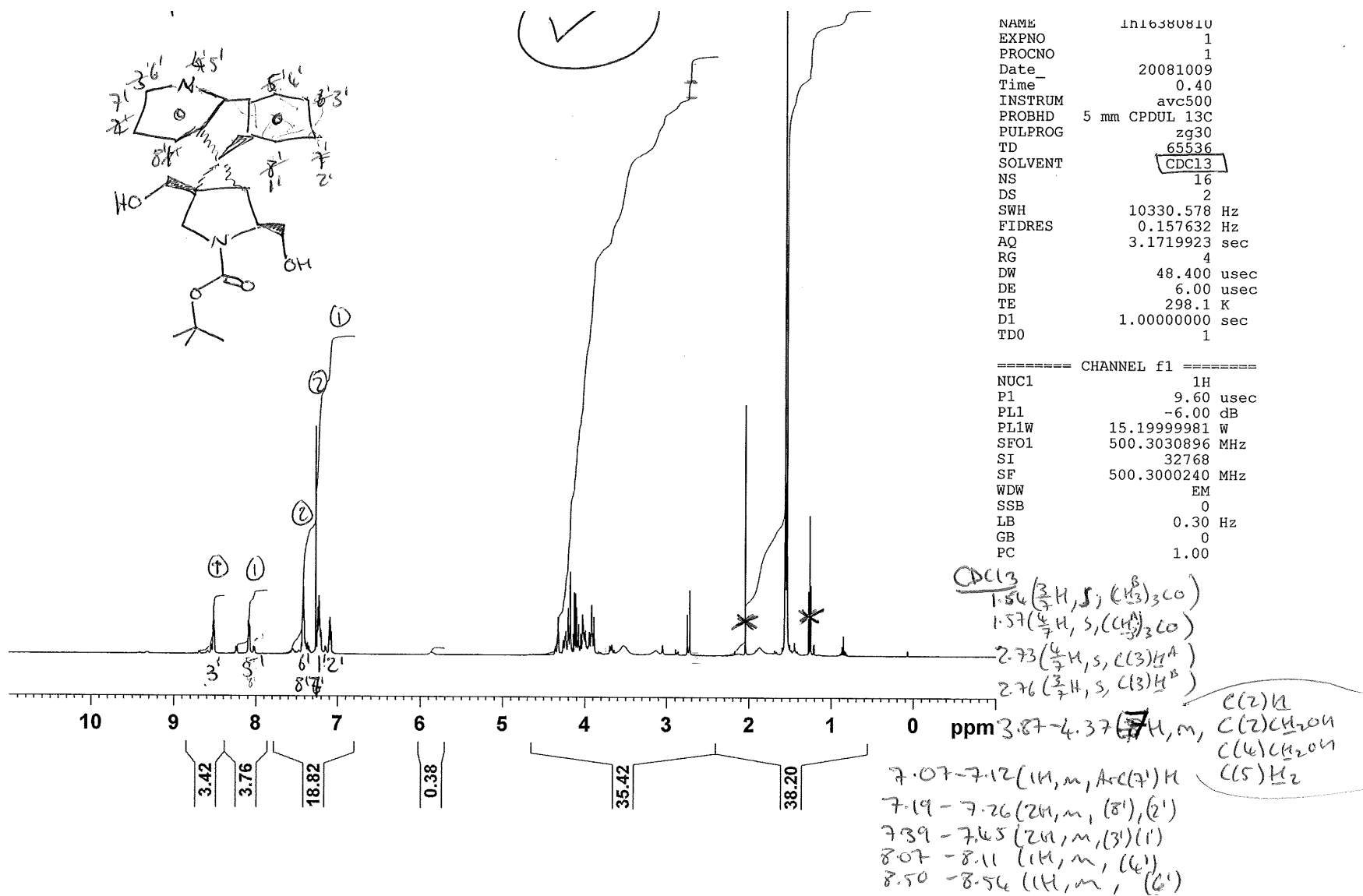
F2 - Acquisition Parameters
 Date 20080920
 Time 3.20
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD
 NS 1024
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 1820
 DW 16.000 usec
 DE 20.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

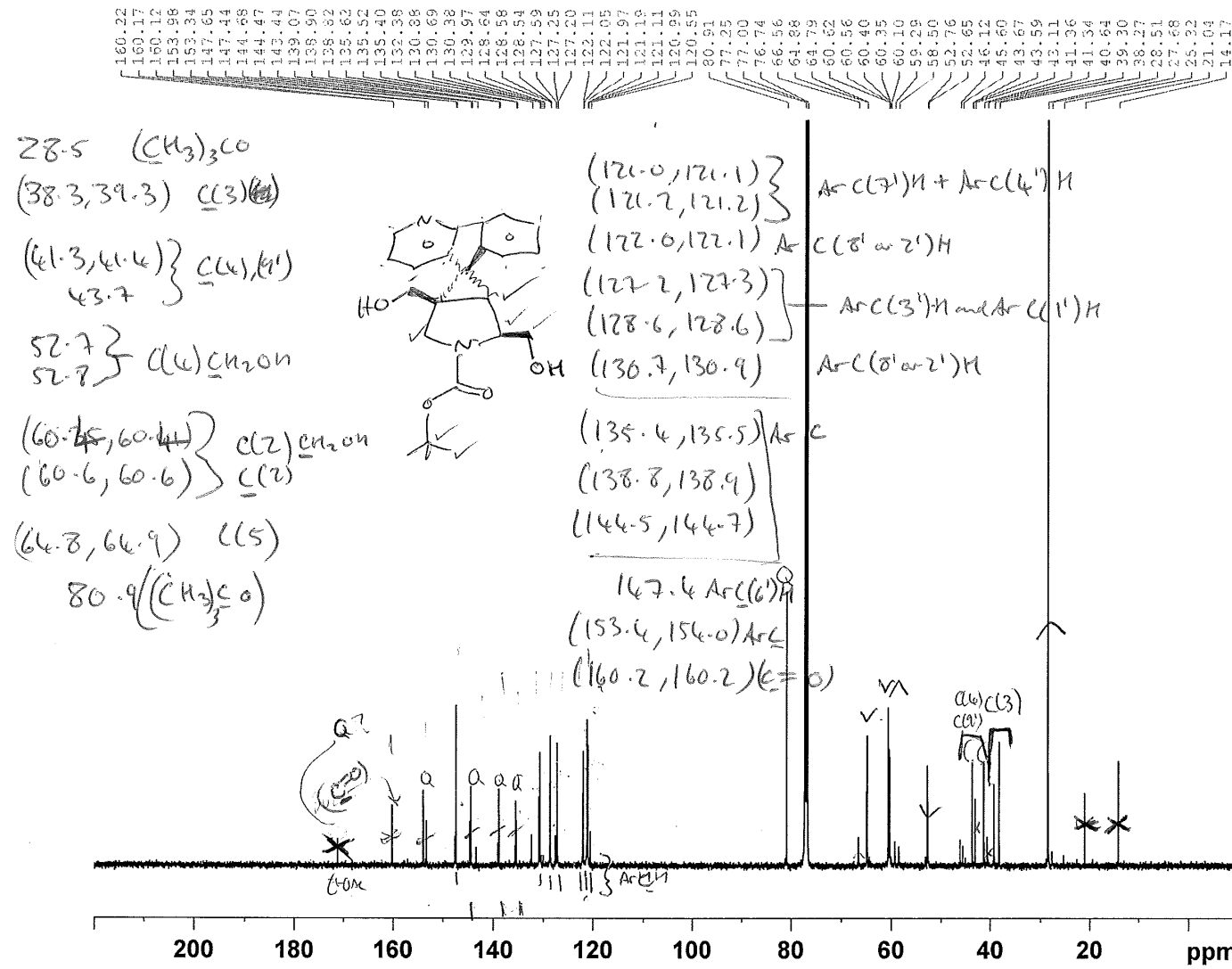
==== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -4.40 dB
 SFO1 125.8131151 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.40 dB
 PL13 17.00 dB
 PL2 -6.00 dB
 SFO2 500.3020012 MHz

F2 - Processing parameters
 SI 32768
 SF 125.8003564 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

24e





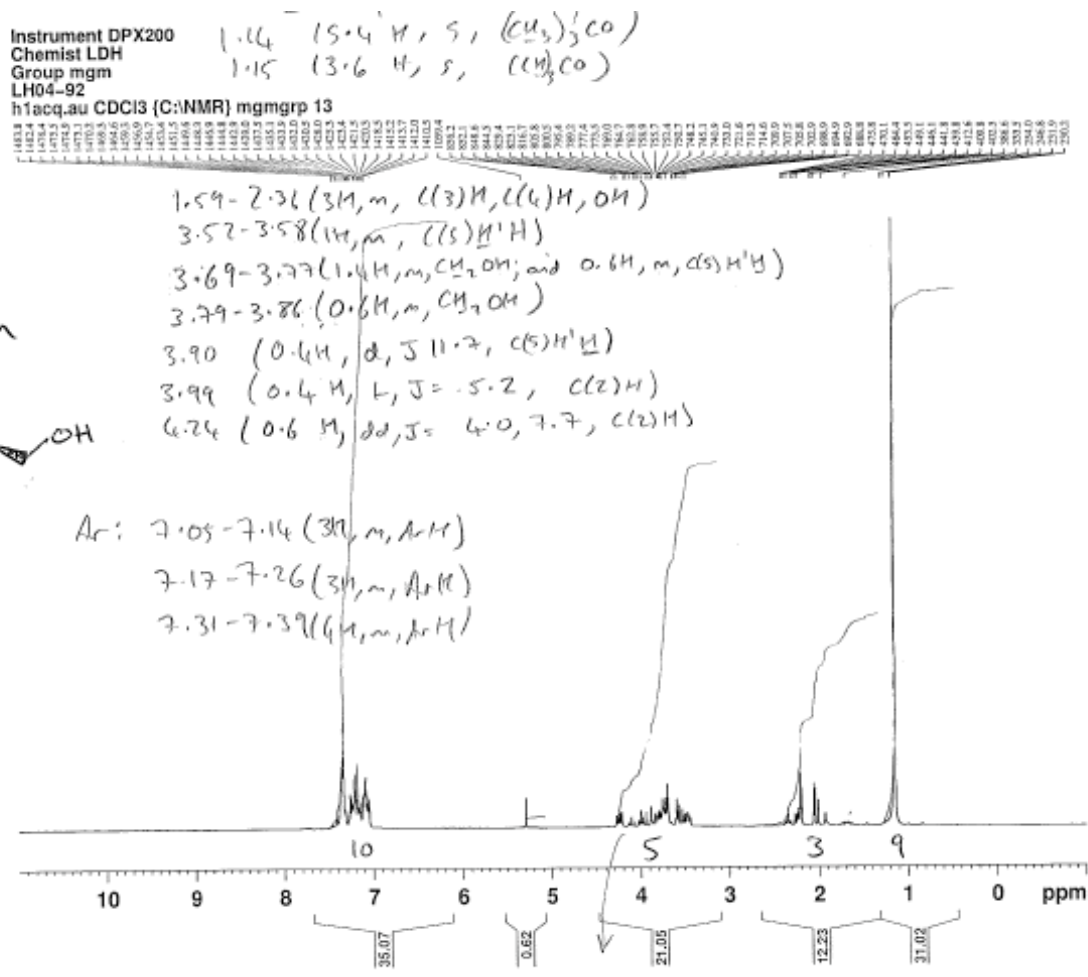
```

NAME          lh16380810
EXPNO         4
PROCNO        1
Date_         20081009
Time          1.49
INSTRUM       avc500
PROBHD        5 mm CPDUL 13C
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            1024
DS            2
SWH           31250.000 Hz
FIDRES        0.476837 Hz
AQ            1.0486259 sec
RG            1820
DW            16.000 usec
DE            20.00 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            8.00 usec
PL1           -4.40 dB
PL1W          28.15752029 W
SFO1          125.8131151 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -6.00 dB
PL12          12.40 dB
PL13          17.00 dB
PL2W          15.19999981 W
PL12W         0.21970686 W
PL13W         0.07618046 W
SFO2          500.3020012 MHz
SI            32768
SF            125.8005438 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

24g



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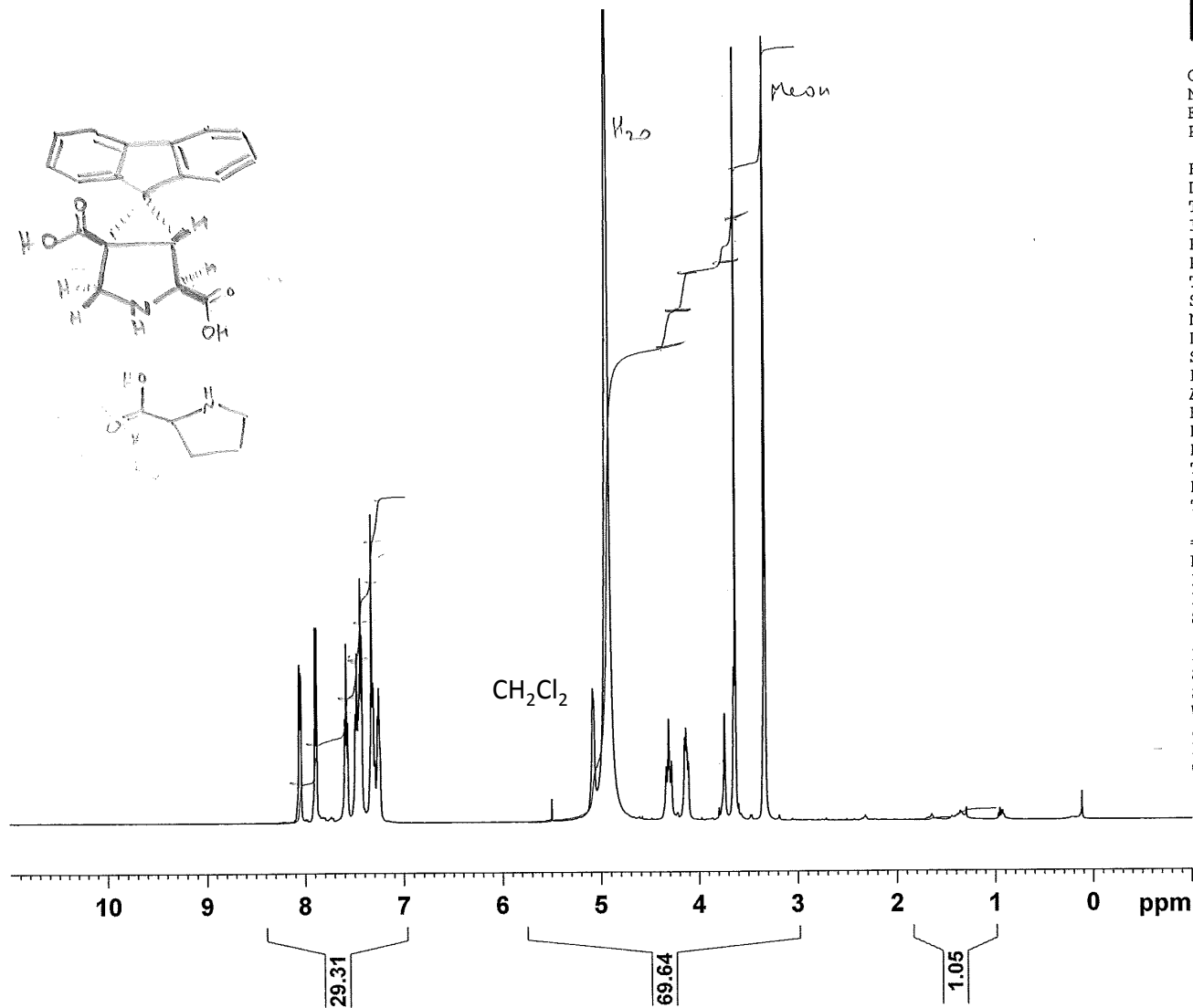
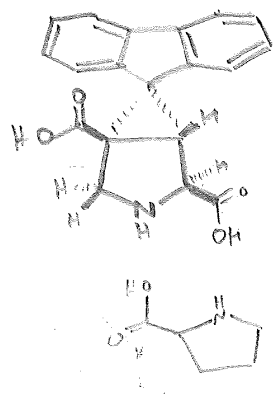
Current Data Parameters
 NAME Apr22-2008-13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080422
 Time 16.13
 INSTRUM dpx200
 PROBHD 5 mm Dual 13C/
 PULPROG zgpg30
 TD 16384
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 2796.421 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 161.3
 DM 178.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.80 usec
 PL1 -3.00 dB
 SFO1 200.1310007 MHz

F2 - Processing parameters
 SI 32768
 SF 200.1300125 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

26a



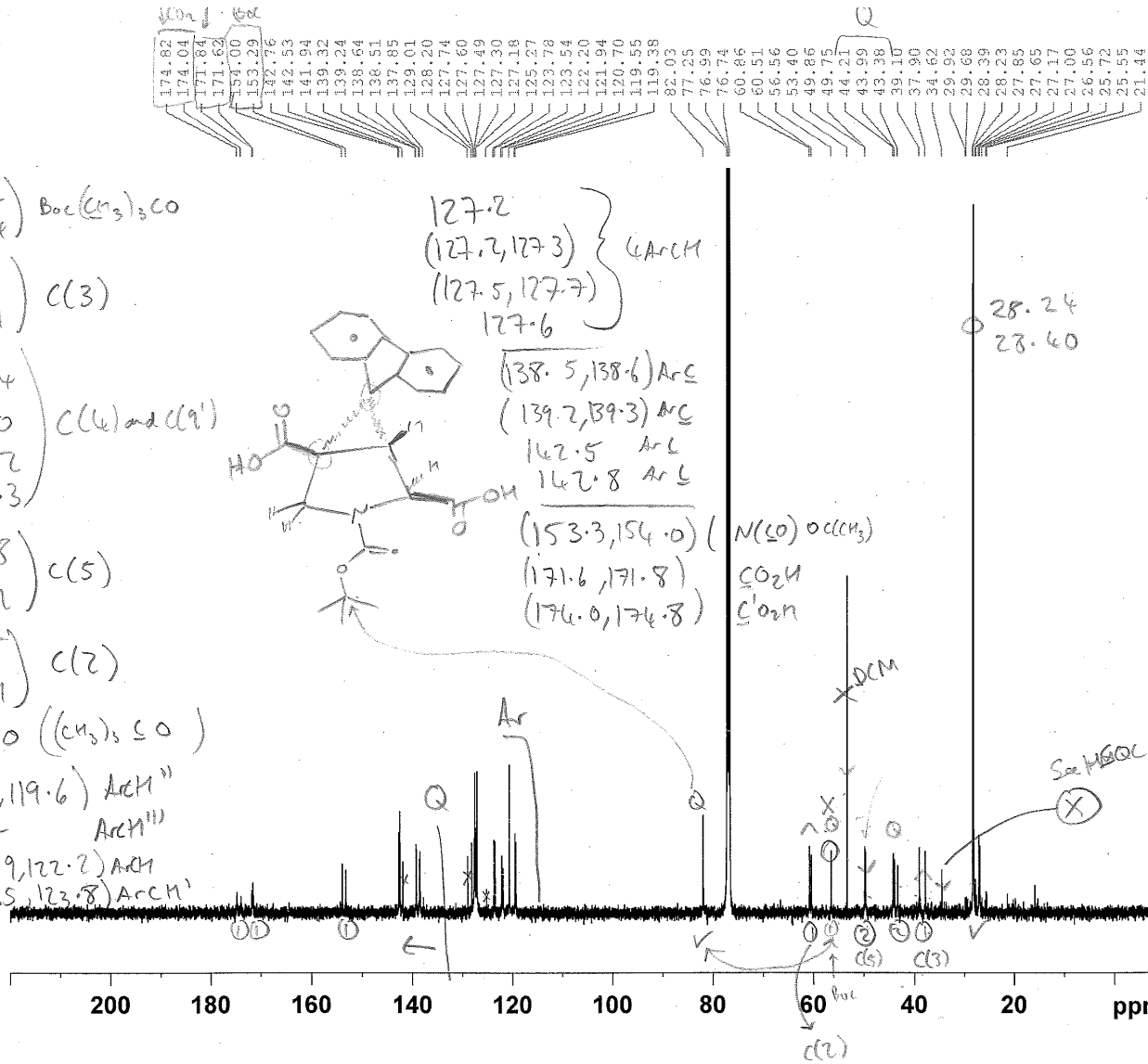
Current Data Parameters
NAME lh07341206
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20080612
Time 14.44
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 4
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 500.3030896 MHz

F2 - Processing parameters
SI 32768
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

26a



Current Data Parameters

NAME lh81081110
EXPNO 4
PROCNO 1

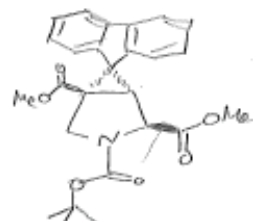
F2 - Acquisition Parameters
Date_ 20071011
Time 22.18
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCL3
NS 1024
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 1820
DW 16.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -4.40 dB
SFO1 125.8131151 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.40 dB
PL13 17.00 dB
PL2 -6.00 dB
SFO2 500.3020012 MHz

F2 - Processing parameters
SI 32768
SF 125.8005438 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Instrument DQX400
 Chemist Lawrence
 Group mgn
 LH05-6
 h1acq.au CDC13 (C:NMR) mgngrp 39

Rotamers (11:10)
 A:6



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Current Data Parameters
 NAME Jun03-2008-39
 EXPNO 1
 PROCNO 1

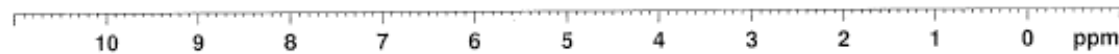
F2 - Acquisition Parameters
 Date_ 20080603
 Time 18.58
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 90.5
 DW 60.400 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.00000000 sec

----- CHANNEL f1 -----
 NUC1 13
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.2024714 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2000028 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

- 1.50 (9H_s, s, Boc)
- 1.57 (9H_s, s, Boc)
- 3.15 (1H_A, s, C(3)H_A)
- 3.18 (1H_B, s, C(3)H_B)
- 3.58 (3H_s, s, C(6)CO₂CH₃^B)
- 3.58 (3H_A, s, C(6)CO₂CH₃^A)
- 3.86 (3H_A, s, C(2)CO₂CH₃^A)
- 3.87 (3H_B, s, C(2)CO₂CH₃^B)
- 4.06 (1H_B, d, J 12.1, C(5)H_B)
- 4.11 (1H_A, d, J 12.2, C(5)H_A)
- 6.25 (1H_B, d, J 12.1, C(5)H_B)
- 6.30 (1H_A, d, J 12.2, C(5)H_A)
- 4.58 (1H_A, s, C(2)H_A)
- 4.70 (1H_B, s, C(2)H_B)

Aromatic
 7.11 (1H_B, d, J 7.9, ArH_B)
 7.14 (1H_A, d, J 7.9, ArH_A)
 7.23-7.68 (5H_m, m, ArH_m)
 7.79 (1H_A, d, J 7.5, ArH_A)
 7.90 (1H_B, d, J 7.3, ArH_B)



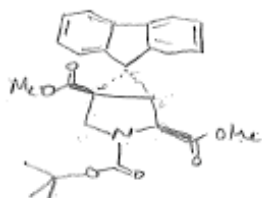
26b

ArCH

(119.6, 119.6), 120.7, (121.7, 121.8),
 (123.7, 123.7), (127.1, 127.1),
 (127.3), 127.4, (127.5), 127.7

ArC

Instrument DQX400
 Chemist Lawrence
 Group mgm
 LH05-6
 c13acq.au CDC13 (C:NMR) mgmgrp 39



Ref - 0.04

(138.3, 138.4), 139.4,
 (142.2, 142.3), (142.6, 142.6)

(152.6, 153.1), (167.5, 167.5), (171.0, 171.3) (C(2)CO₂Me)

(28.2, 28.4) Boc-CH₃

(37.9, 38.8) (C(3))

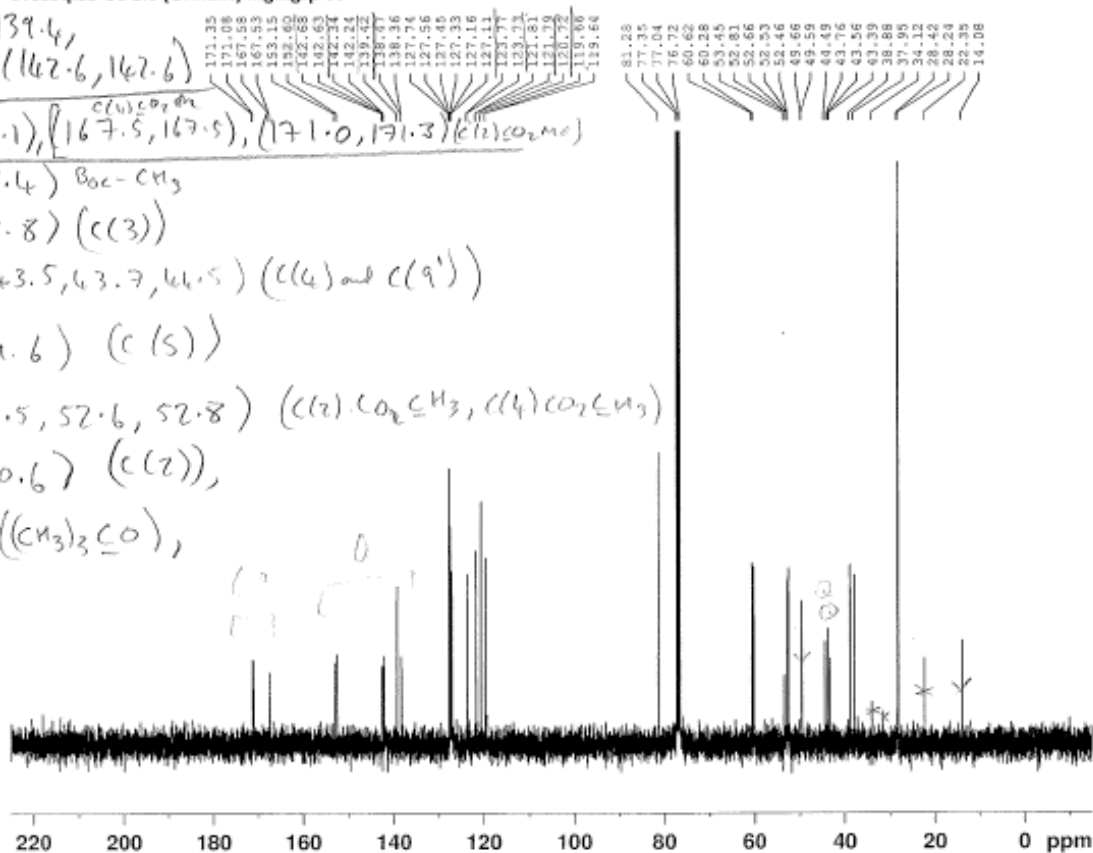
(43.4, 43.5, 43.7, 44.5) (C(4) and C(9'))

(49.5, 49.6) (C(5))

(52.4, 52.5, 52.6, 52.8) (C(2)CO₂CH₃, C(4)CO₂CH₃)

(60.2, 60.6) (C(2)),

81.2 ((CH₃)₃CO),



NMROCHEM.OX

Current Data Parameters
 NAME Jun03-2008-39
 EXPNO 2
 PROCNO 1

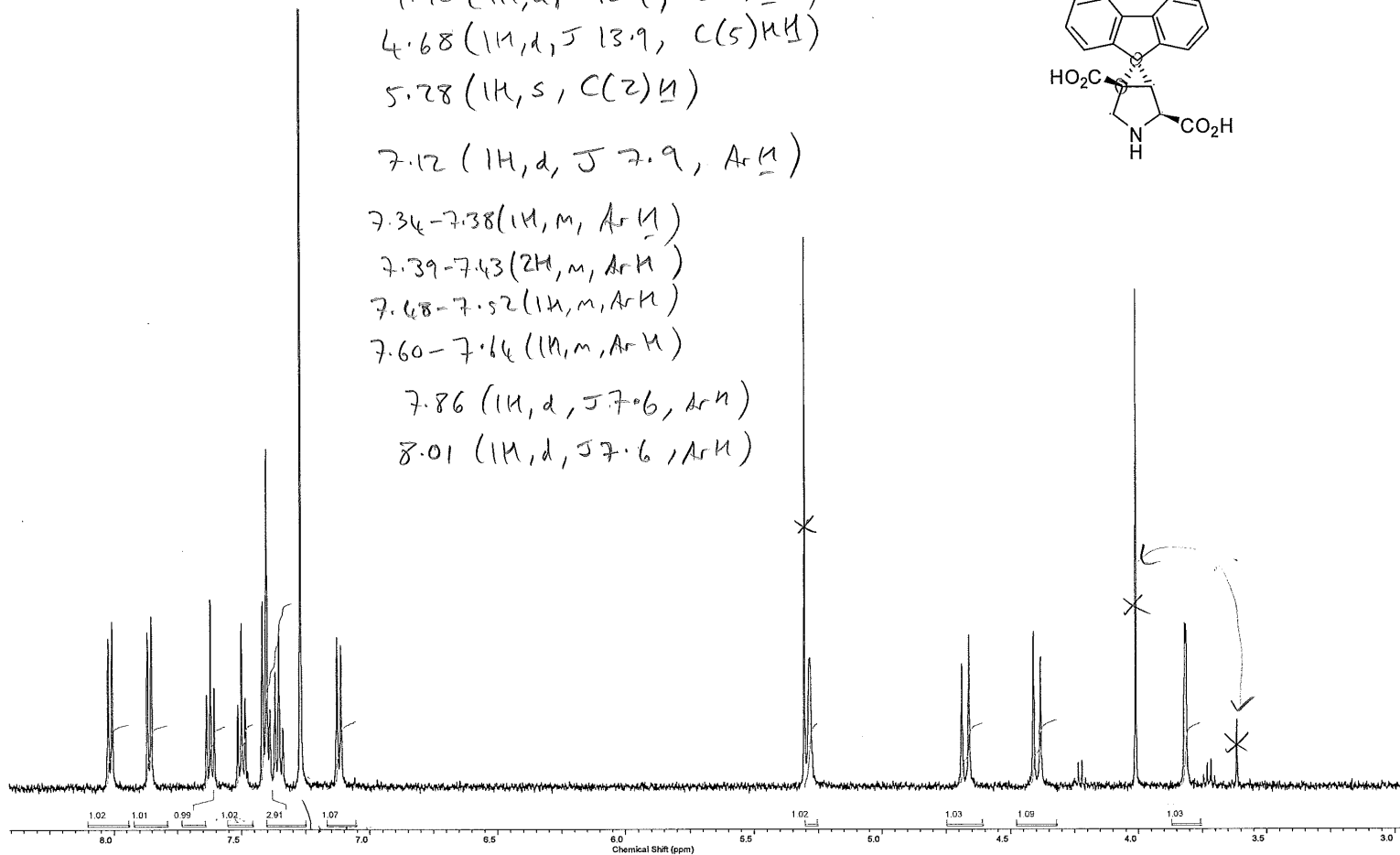
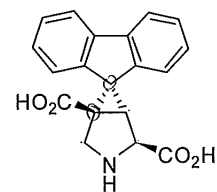
F2 - Acquisition Parameters
 Date_ 20080603
 Time 19.06
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.798889 Hz
 AQ 0.6259188 sec
 RG 32768
 DM 19.100 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TDO 1

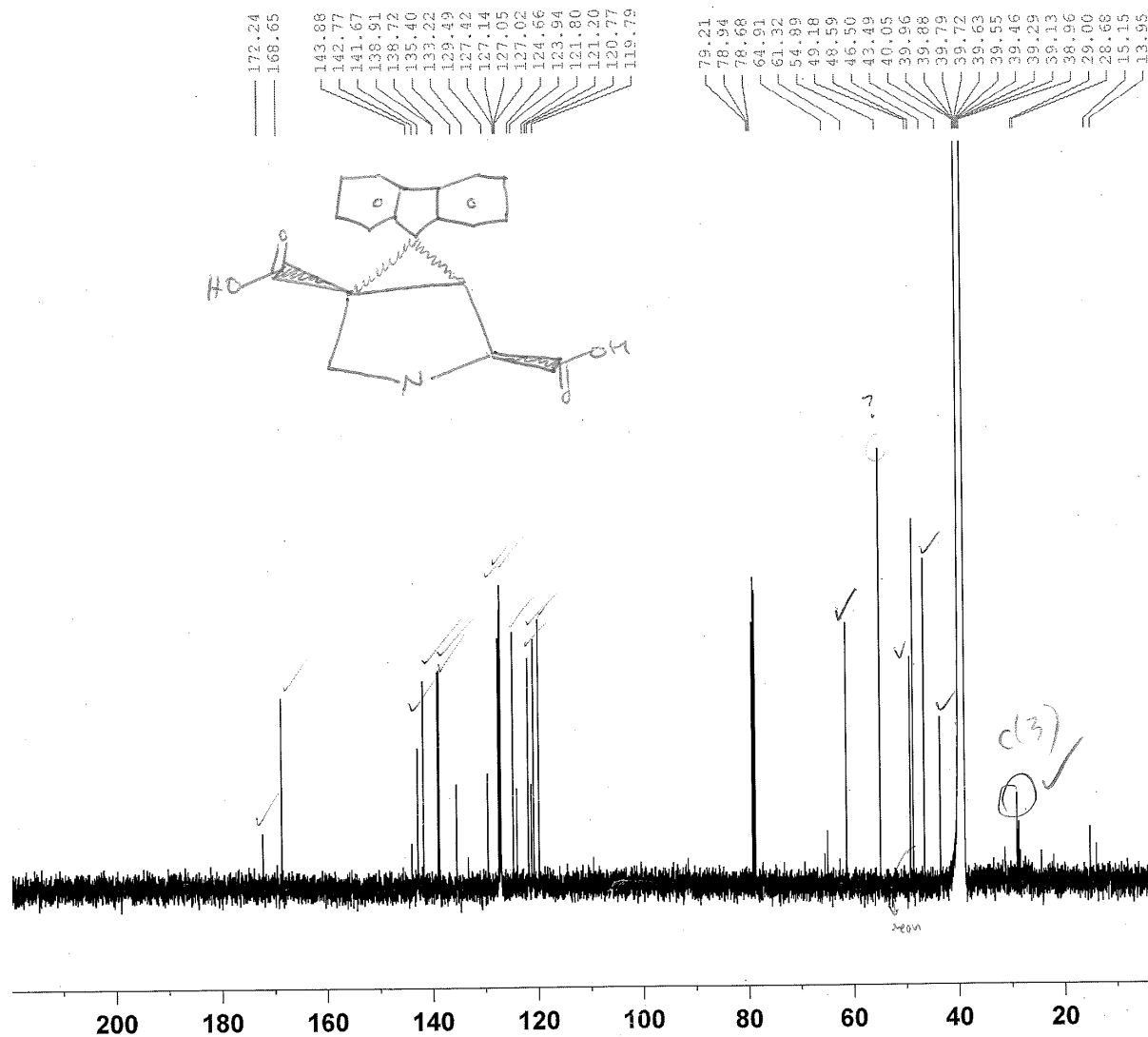
==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 0.00 dB
 SF01 100.6403931 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.00 dB
 PL13 25.00 dB
 PL2 0.00 dB
 SF02 400.2016008 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6303718 MHz
 NDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

3.82 (1H, d, J 2.5, C(3)H)
 4.40 (1H, d, J 13.9, C(5)H)
 4.68 (1H, d, J 13.9, C(5)H)
 5.28 (1H, s, C(2)H)
 7.12 (1H, d, J 7.9, ArH)
 7.34-7.38 (1H, m, ArH)
 7.39-7.43 (2H, m, ArH)
 7.48-7.52 (1H, m, ArH)
 7.60-7.64 (1H, m, ArH)
 7.86 (1H, d, J 7.6, ArH)
 8.01 (1H, d, J 7.6, ArH)





NAME lh81151210

Current Data Parameters
 NAME lh81151210
 EXPNO 5
 PROCNO 1

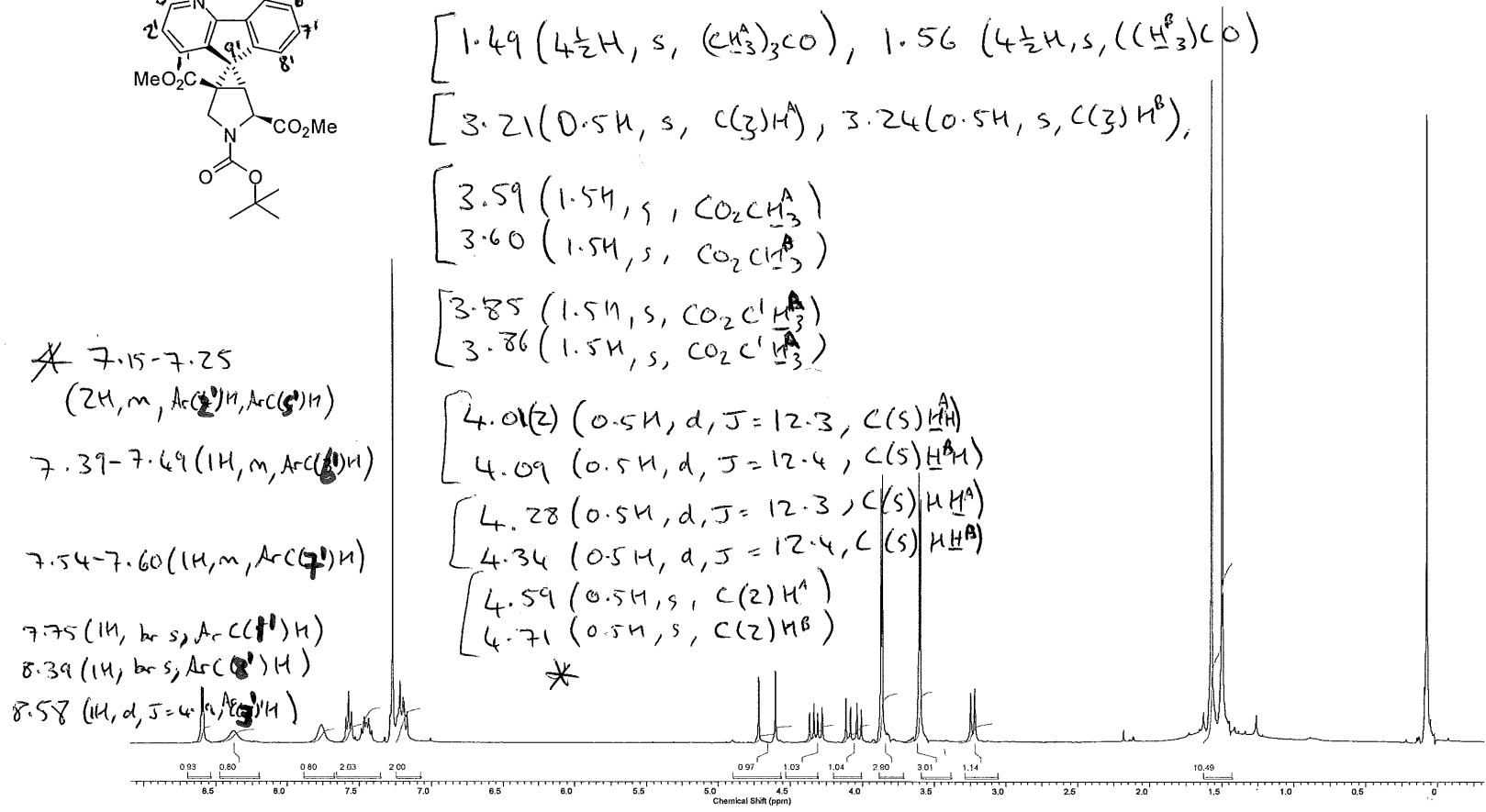
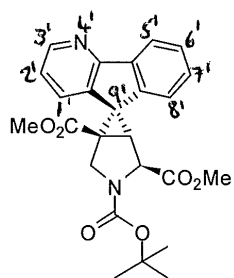
F2 - Acquisition Parameters
 Date_ 20071012
 Time_ 22.30
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3072
 DS 2
 SWH 31250.000 Hz
 FIDRES 0.476837 Hz
 AQ 1.0486259 sec
 RG 1820
 DW 16.000 usec
 DE 20.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -4.40 dB
 SFO1 125.8131151 MHz

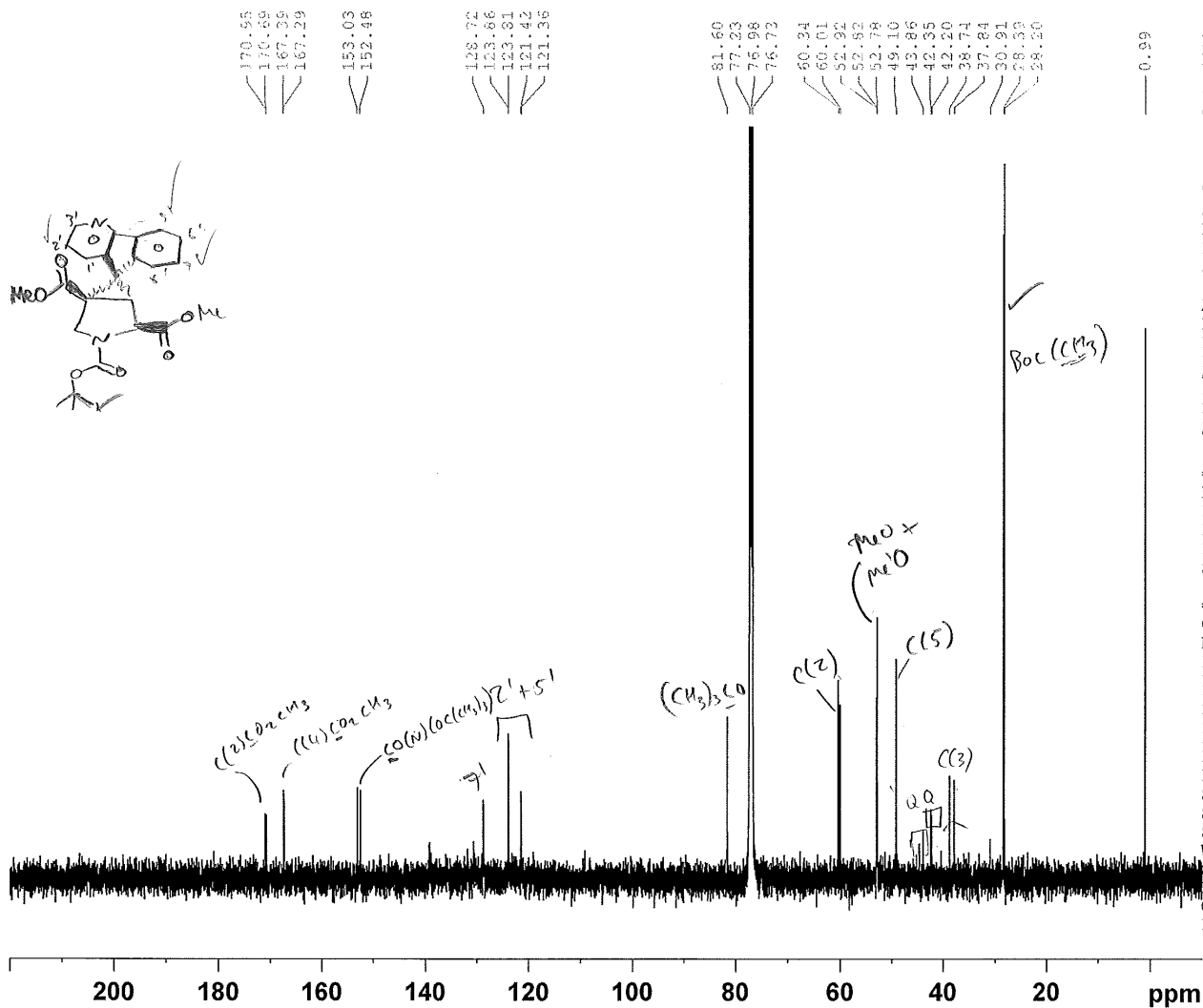
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.40 dB
 PL13 17.00 dB
 PL2 -6.00 dB
 SFO2 500.3020012 MHz

F2 - Processing parameters
 SI 32768
 SF 125.8005954 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

28a



28a



NMR CHEM.OX

```

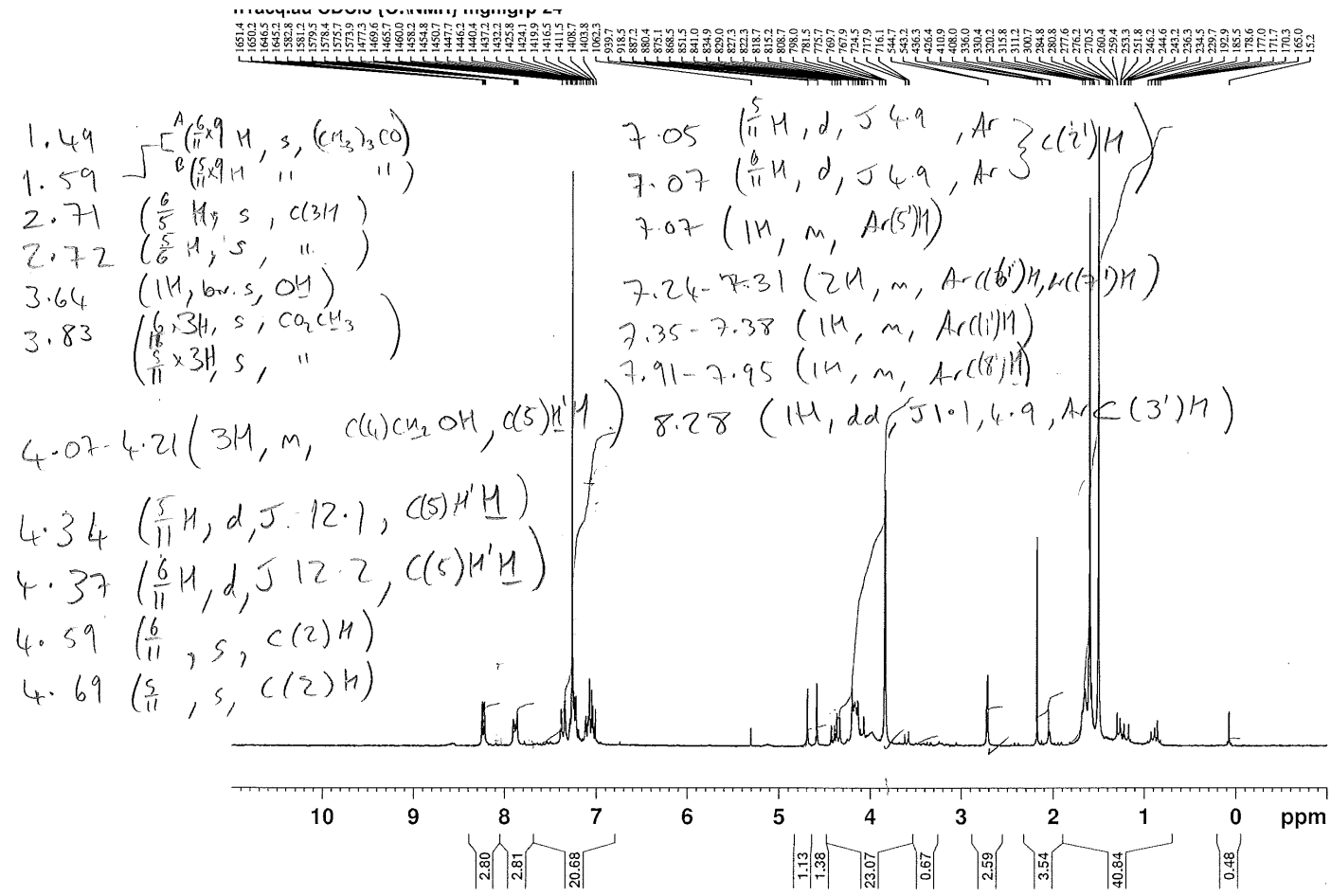
NAME          lh41390605
EXPNO         4
PROCNO       1
Date_        20090506
Time         14.22
INSTRUM      avc500
PROBHD       5 mm CPDUL 13C
PULPROG      zgpg30
TD           65536
SOLVENT      CDC13
NS           1024
DS           2
SWH          31250.000 Hz
FIDRES       0.476837 Hz
AQ           1.0486259 sec
RG           1820
DW           16.000 usec
DE           20.000 usec
TE           298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1
  
```

```

===== CHANNEL f1 =====
NUC1         13C
P1           8.00 usec
PL1          -4.40 dB
PL1W        28.15752029 W
SFO1        125.8131151 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2     waltz16
NUC2         1H
PCPD2       80.00 usec
PL2         -6.00 dB
PL12        12.40 dB
PL13        17.00 dB
PL2W        15.19999981 W
PL12W       0.21970686 W
PL13W       0.07618046 W
SFO2        500.3020012 MHz
SI          32768
SF          125.8005438 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```



1651.4
1646.5
1645.5
1645.2
1582.8
1581.2
1581.2
1578.4
1575.7
1573.9
1477.3
1469.9
1468.9
1460.0
1458.2
1454.8
1450.7
1446.2
1440.4
1437.2
1432.2
1424.1
1419.9
1416.5
1411.5
1408.8
1403.3
939.7
918.5
887.2
887.2
875.1
868.5
851.5
841.0
839.0
837.3
822.3
818.7
818.7
818.7
798.0
781.5
775.7
769.7
754.5
754.5
717.9
716.1
714.7
714.7
436.4
436.3
410.9
408.0
408.0
330.4
330.4
315.8
311.2
311.2
284.7
280.8
277.6
276.2
270.5
270.5
259.4
253.3
251.8
246.2
243.9
243.9
236.5
234.5
229.7
185.5
185.5
178.6
177.0
176.0
187.0
182.0
152.0

Current Data Parameters
 NAME Oct29-2008-24
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20081029
 Time 11.37
 INSTRUM dpx200
 PROBHD 5 mm Dual 13C/
 PULPROG zg60
 TD 16384
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 2796.421 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 1290.2
 DW 178.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

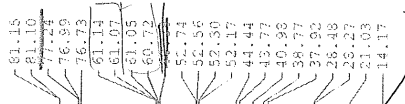
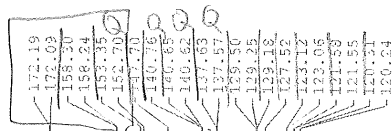
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.80 usec
 PL1 -3.00 dB
 SF01 200.1310007 MHz

F2 - Processing parameters
 SI 32768
 SF 200.1300125 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

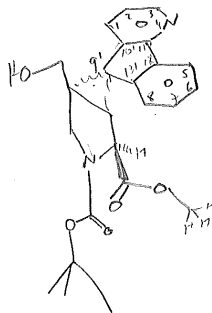
Instrument AVC500
1862 Lawrence Harris 29/10/08

Aromatics: (120.2, 120.3) (ArC(2')H), (121.6, 121.6) (ArC(8')H)
(123.1, 123.1) (ArC(5')H), (127.5) (ArC(7')H)
(129.2, 129.3) (ArC(1')H), (129.3, 129.5) ArC(6')H

NMROCHEM.OX



(28.3, 28.5) Boc
(37.9, 38.3) C(3)
41.0
(43.3, 44.5) } C(6)+C(9)
(60.7, 61.1) ↑
(61.1, 61.2) ↓
77.2 (CD₃OC(=O))
(81.1, 81.2) Boc @ (12)(10)OC(=O)

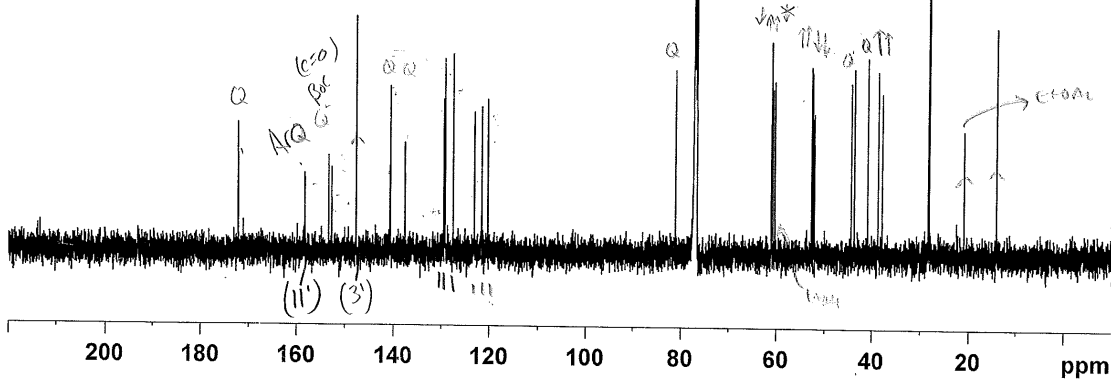


(137.6, 137.6)
(140.6, 140.7) } (ArC)
(140.7, 140.8)
147.7 (ArC(3')H)
(152.7, 153.4) (C=O) Boc
(158.3, 158.3) (ArC(11'))
(172.1, 172.2) CO₂CH₃

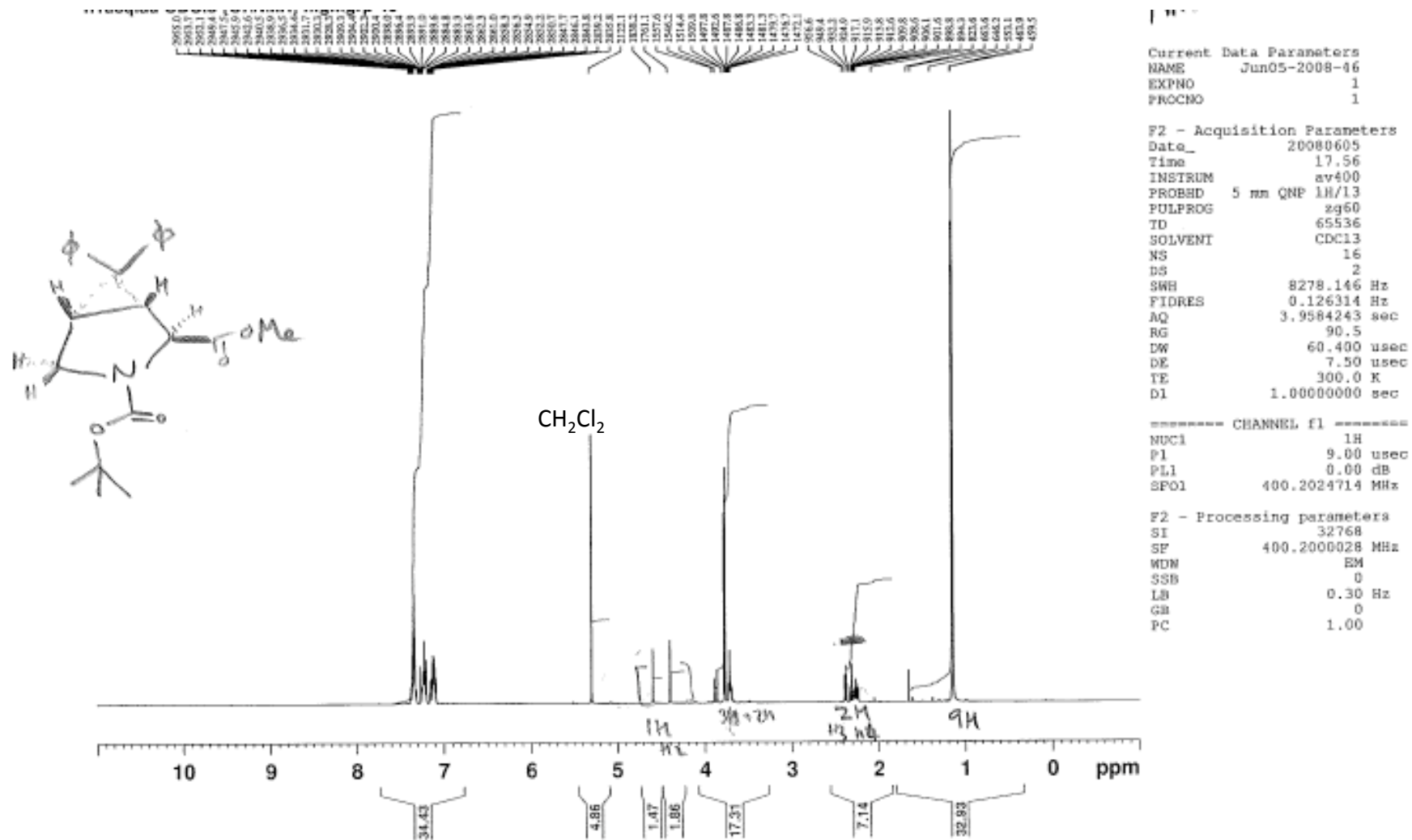
NAME 1h18622910
EXPNO 3
PROCNO 1
Date_ 20081030
Time_ 0.06
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 1820
DW 16.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -4.40 dB
PL1W 28.15752029 W
SFO1 125.8131151 MHz

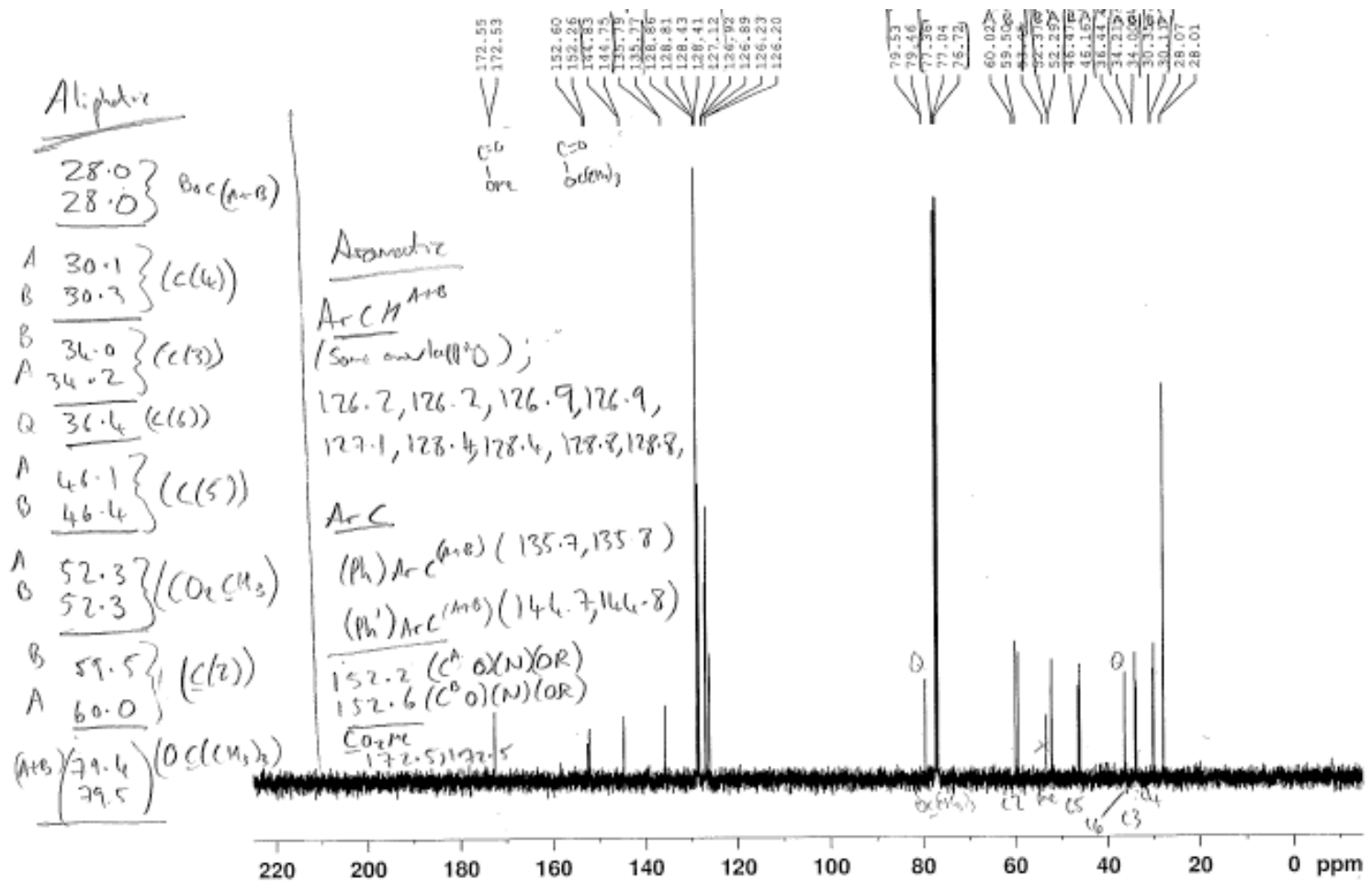
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 12.40 dB
PL13 17.00 dB
PL2W 15.19999981 W
PL12W 0.21970686 W
PL13W 0.07618046 W
SFO2 500.3020012 MHz
SI 32768
SF 125.8005438 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



29a



29a



Current Data Parameters

NAME Jun05-2008-46

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20080605

Time 18.03

INSTRUM av400

PROBHD 5 mm QNP 1H/13

PULPROG zgpg30

ID 32768

SOLVENT CDC13

NS 256

DS 4

SWH 26178.010 Hz

FIDRES 0.798889 Hz

AQ 0.6259188 sec

RG 32768

DW 19.100 usec

DE 7.50 usec

TE 300.0 K

D1 1.00000000 sec

d11 0.03000000 sec

DELTA 0.89999998 sec

TD0 1

----- CHANNEL f1 -----

NUC1 13C

P1 9.50 usec

PL1 0.00 dB

SFO1 100.6403931 MHz

----- CHANNEL f2 -----

CPDPRG2 valtz16

NUC2 1H

PCPD2 80.00 usec

PL12 19.00 dB

PL13 25.00 dB

PL2 0.00 dB

SFO2 400.2016008 MHz

F2 - Processing parameters

SI 32768

SF 100.6303718 MHz

WDW EM

SSB 0

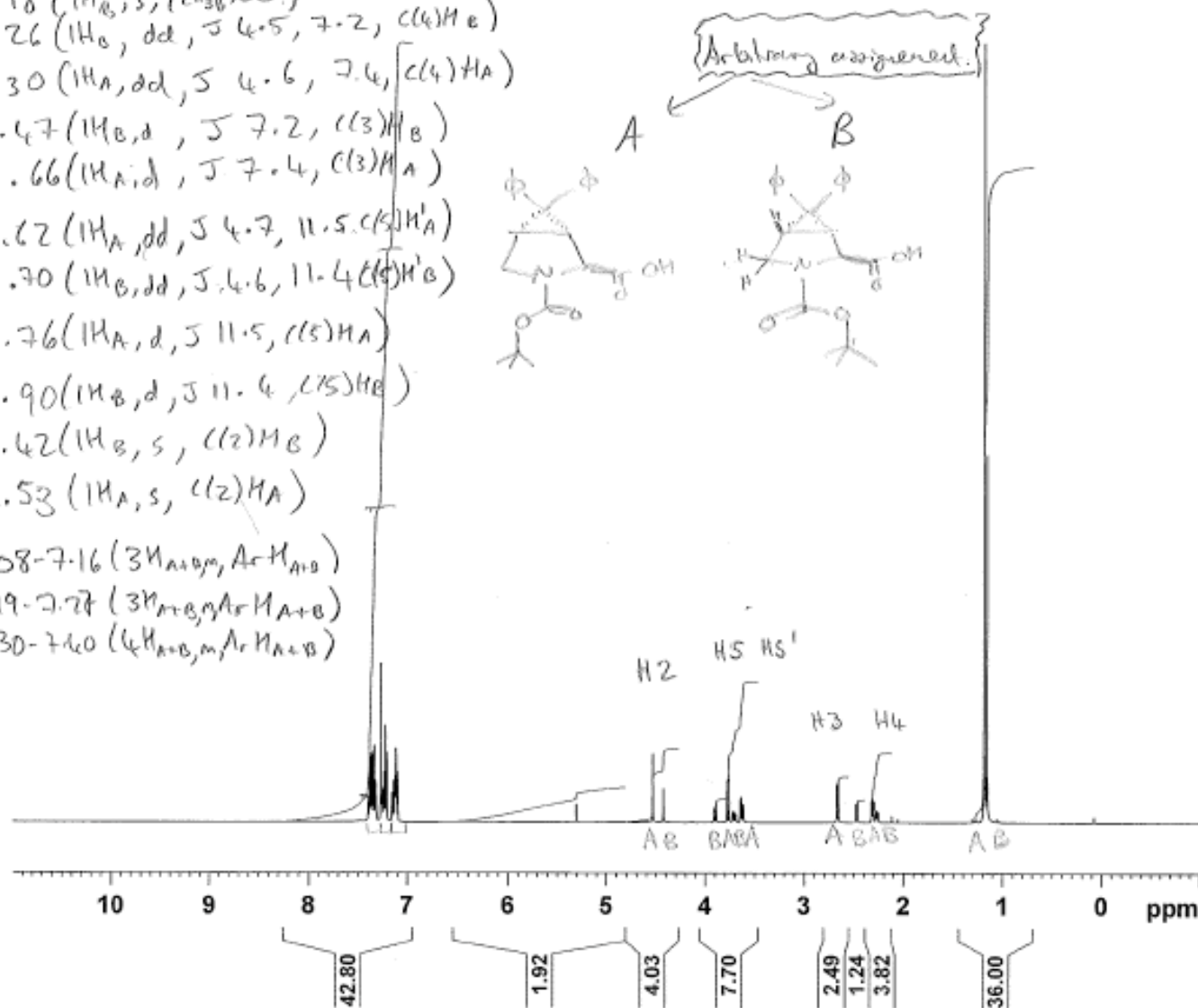
LB 1.00 Hz

GB 0

PC 1.40

29b

- 1.15 (1H_B, s, (CH₃)₂CO)
- 1.18 (1H_B, s, (CH₃)₂CO)
- 2.26 (1H_A, dd, J 4.5, 7.2, C(4)H_e)
- 2.30 (1H_A, dd, J 4.6, 7.4, C(4)H_A)
- 2.47 (1H_B, d, J 7.2, C(3)H_B)
- 2.66 (1H_A, d, J 7.4, C(3)H_A)
- 3.62 (1H_A, dd, J 4.7, 11.5, C(5)H_A)
- 3.70 (1H_B, dd, J 4.6, 11.4, C(5)H_B)
- 3.76 (1H_A, d, J 11.5, C(5)H_A)
- 3.90 (1H_B, d, J 11.4, C(5)H_B)
- 4.42 (1H_B, s, C(2)H_B)
- 4.53 (1H_A, s, C(2)H_A)
- 7.08-7.16 (3M_{A+B}, ArH_{A+B})
- 7.19-7.27 (3M_{A+B}, ArH_{A+B})
- 7.30-7.40 (4H_{A+B}, m, ArH_{A+B})



NMR@CHEM.OX

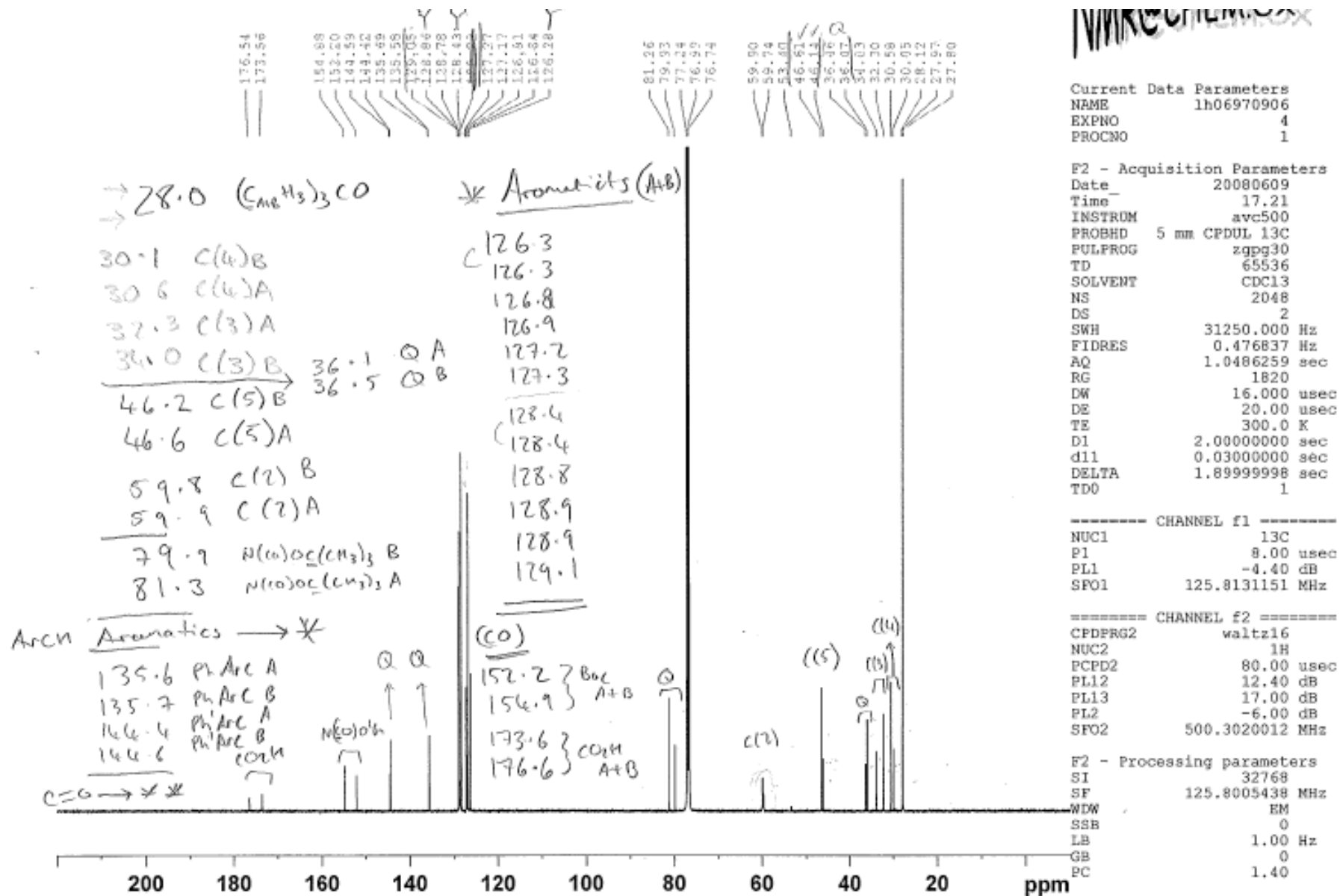
Current Data Parameters
 NAME lh06970906
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080609
 Time 14.50
 INSTRUM avc500
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 4
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

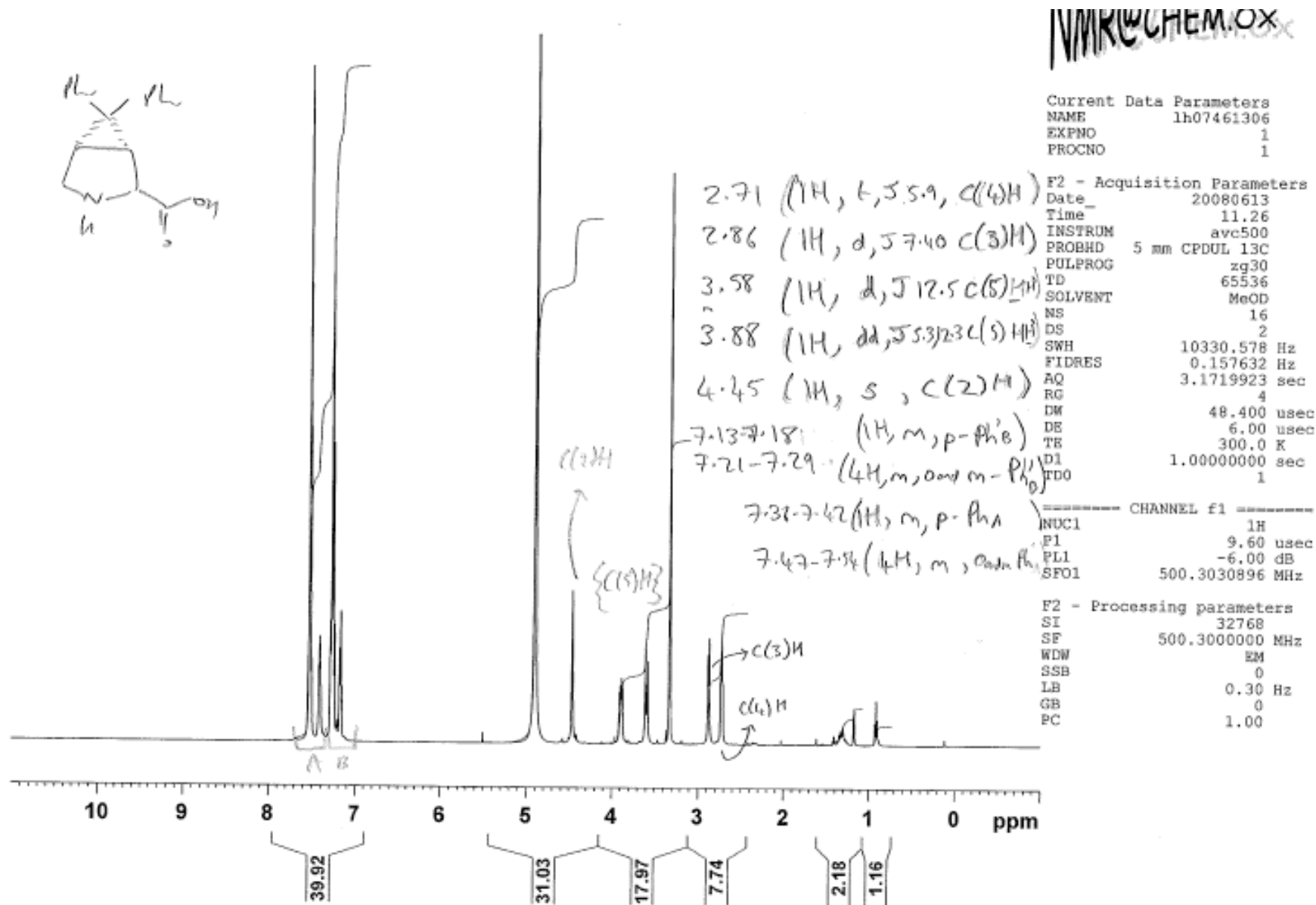
----- CHANNEL f1 -----
 NUC1 1H
 P1 9.60 usec
 PL1 -6.00 dB
 SFO1 500.3030896 MHz

F2 - Processing parameters
 SI 32768
 SF 500.3000240 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

29b

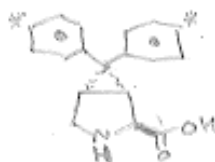


29c



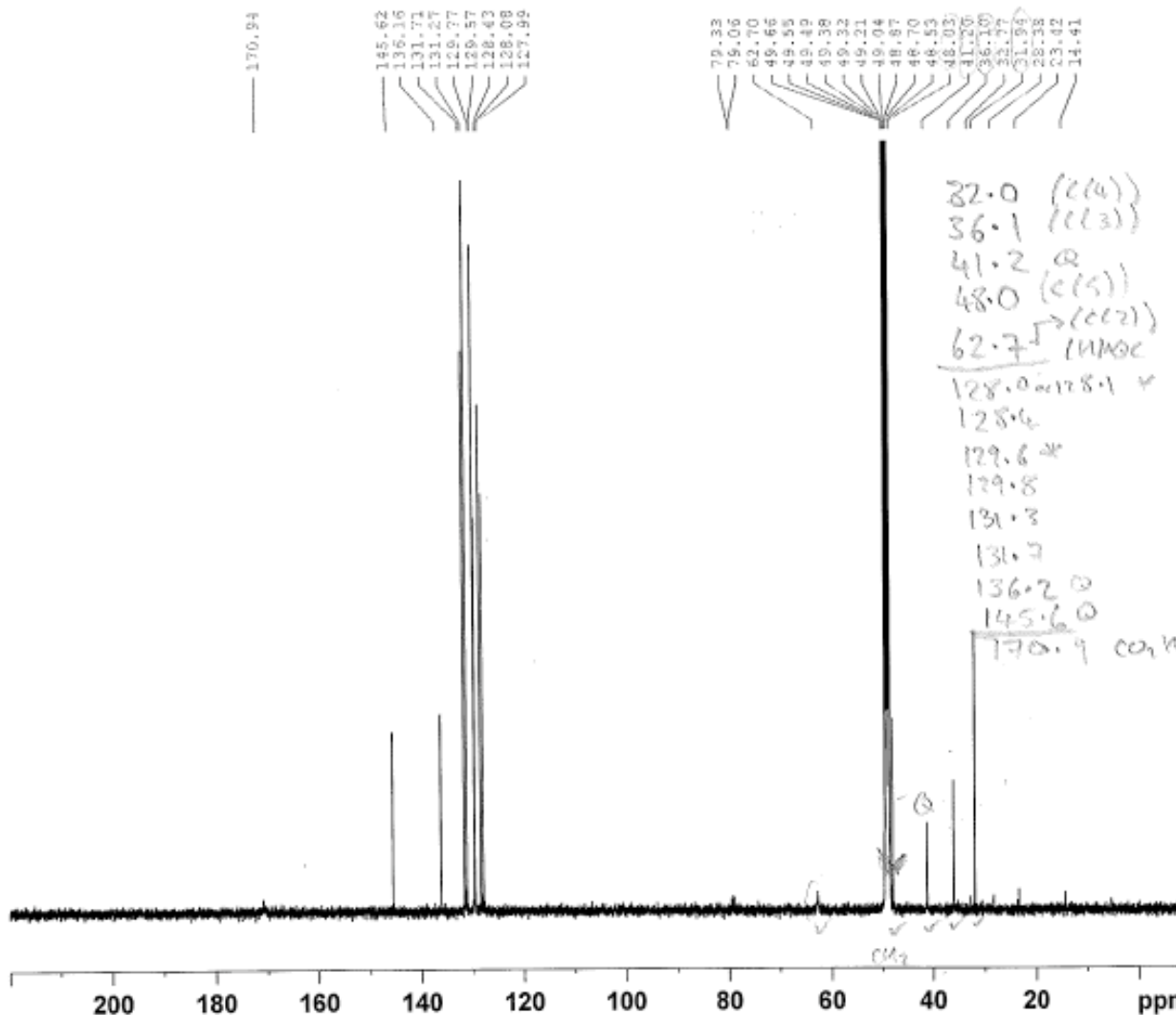
29c

Instrument AVC500
0746 Lawrence Harris 13/6/08



0.015

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Current Data Parameters
NAME lh07461306
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date 20080613
Time 11.55
INSTRUM avc500
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 1820
DW 16.000 usec
DE 20.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

----- CHANNEL f1 -----
NUC1 13C
P1 8.00 usec
PL1 -4.40 dB
SFO1 125.8131151 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.40 dB
PL13 17.00 dB
PL2 -6.00 dB
SFO2 500.3020012 MHz

F2 - Processing parameters
SI 32768
SF 125.8003564 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40