

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines Via Lithiation of *N*-BOC Methanopyrrolidines. Constrained C^γ-exo and C^γ-endo Flp and Hyp Conformer Mimics.

Grant R. Krow,^{*,†} Matthew D. Shoulders,[‡] Ramakrishna Edupuganti,[†] Deepa Gandla,[†] Fang Yu,[†] Philip E.

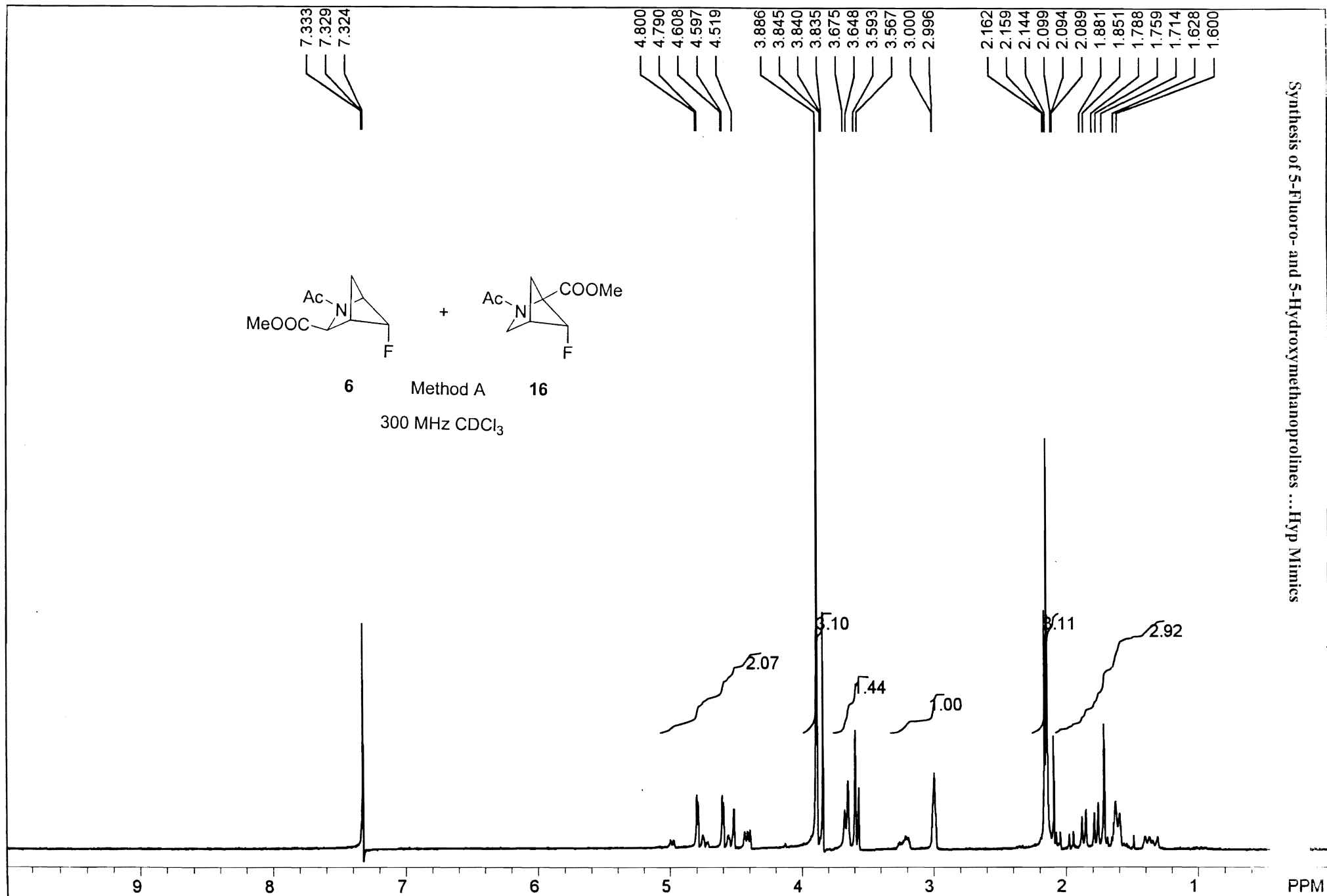
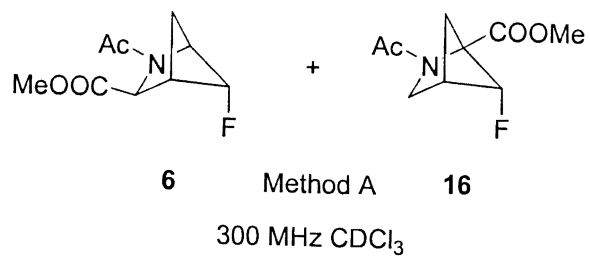
Sonnet,[†] Matthew Sender,[†] Amit Choudhary,[§] Charles DeBrosse,[†] Charles W. Ross, III,[‡] Patrick Carroll,[‡]

and Ronald T. Raines^{‡§}

Supplemental Section

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S-2 to 7	Spectra for 6 + 16
S-8 to 24	Spectra for 7 + 11
S-25 to 27	Spectra for 8
S-28 to 30	Spectra for 9
S-31	Spectrum for 10 (¹ H NMR in D ₂ O) Known compound
S-32 to 34	Spectra for 12 Known compound
S-35 to 36	Spectra for 14 + 15
S-37 to 38	Spectra for 18 + 19
S-39 to 40	Spectra for 20
S-41 to 42	Spectra for 21 + 22
S-43 to 44	Spectra for 23
S-45 to 46	Spectra for 24
S-47 to 48	Spectra for 25
S-49 to 50	Spectra for 27
S-51 to 52	Spectra for 28
S-53 to 54	Spectra for 29
S-55 to 57	Spectra for 30
S-58 to 60	Spectra for 31
S-61 to 62	Spectra for 33 + 34
S-63 to 65	Spectra for 35
S-66 to 68	Spectra for 36



Ac-N

USER: -- DATE: May 2 2005

F1: 299.986

F2: 299.986

SW1: 3600

OF1: 1500.0

PTS1d: 16384

EX: s2pul

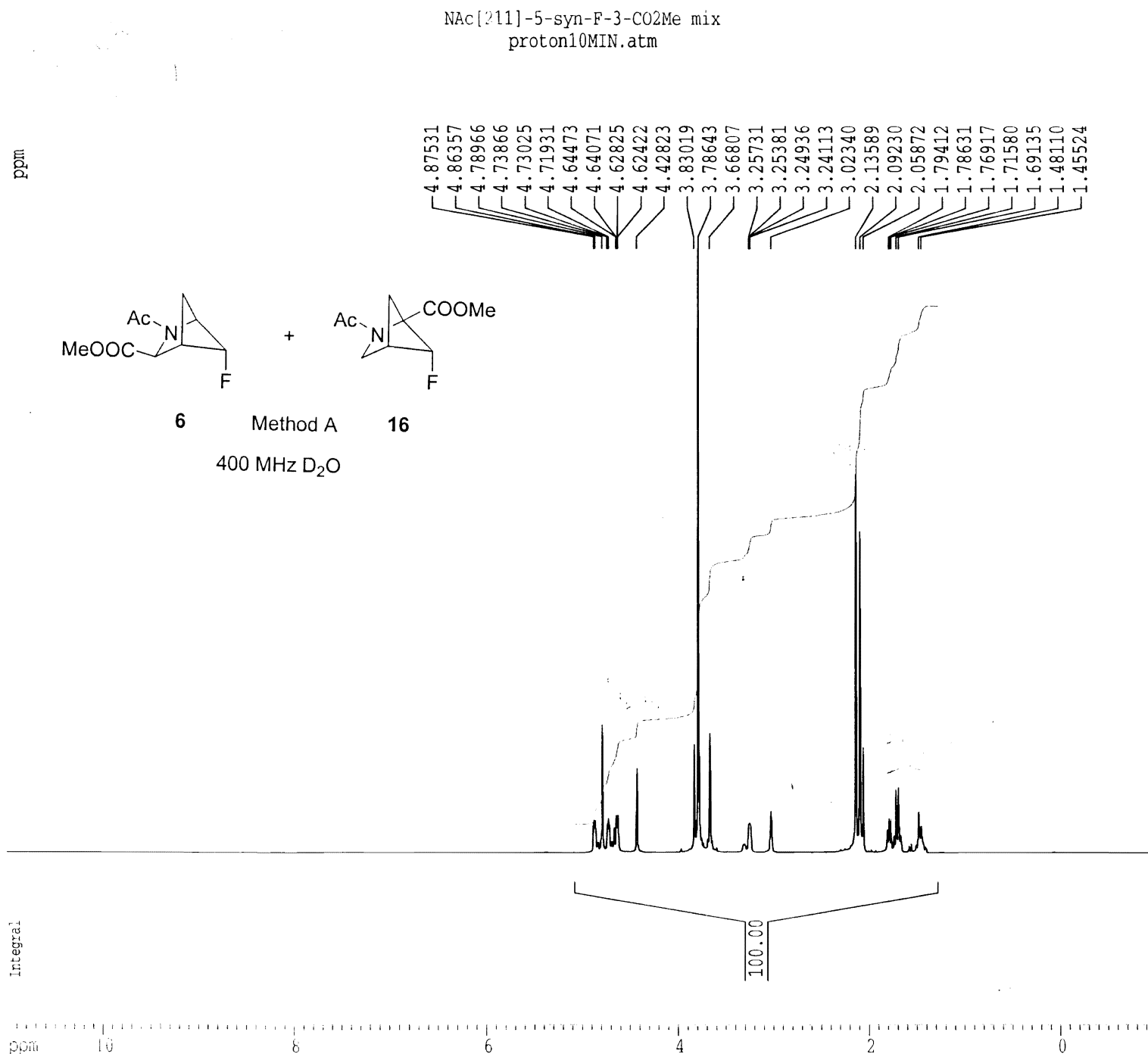
PW: 5.8 usec

PD: 1.0 sec

NA: 8

LB: 0.0

WinNuts - \$PROTON.fid



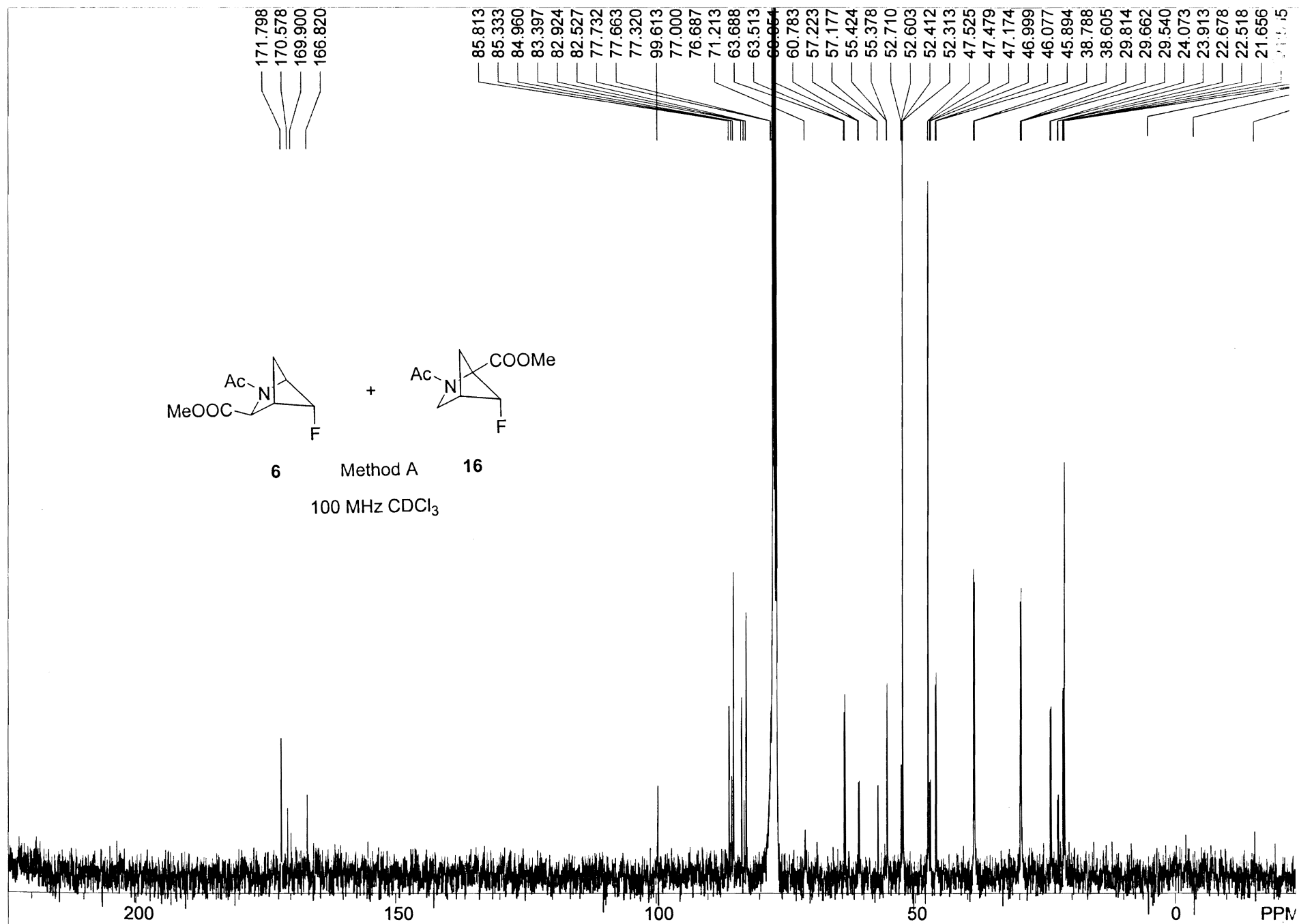
Current Data Parameters
NAME deepa03
EXPNO 5000
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070308
Time 11.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT D2O
NS 128
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 0.00 dB
SFO1 400.1931165 MHz

F2 - Processing parameters
SI 32768
SF 400.1910820 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.000 ppm
F1 4402.10 Hz
F2P -1.000 ppm
F2 -400.19 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.11464 Hz/cm

Ac-N₁ carbonOVNTE.atm,

USER: temple -- DATE: May 3 07:18:17 2005 EDT (UT-4h) templ

F1: 100.585

F2: 1.000

SW1: 25126

OF1: 10019.0

PTS1d: 32768

EX: zgpg60

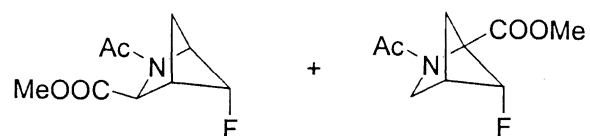
PW: 5.7 us

PD: 2.0 sec

NA: 12000

LB: 2.0

Nuts - \$Desktop_50252.1



6

Method A

16

100 MHz D₂O

175.412
175.127
173.663
172.594
169.662

86.376
85.925
85.806
83.995
83.553
83.406
71.747
71.569
65.135
64.967
62.198
62.029
58.324
58.277
56.391
56.350
53.866
53.610
53.365
48.225
48.187
47.397
47.213
46.449
46.273
39.192
39.008
29.725
29.577
23.680
23.514
22.482
22.316
21.297
21.112
20.918

200

150

100

50

0 PPM

3-CO₂Me NAc[2.1.1] F, carbonOVNTE atm.

USER: temple -- DATE: Apr 24 08:31:53 2007 EDT (UT-4h) templ

F1: 100.628 F2: 1.000

SWH: 23148

OF1: 10621.8

PTS1d: 32768

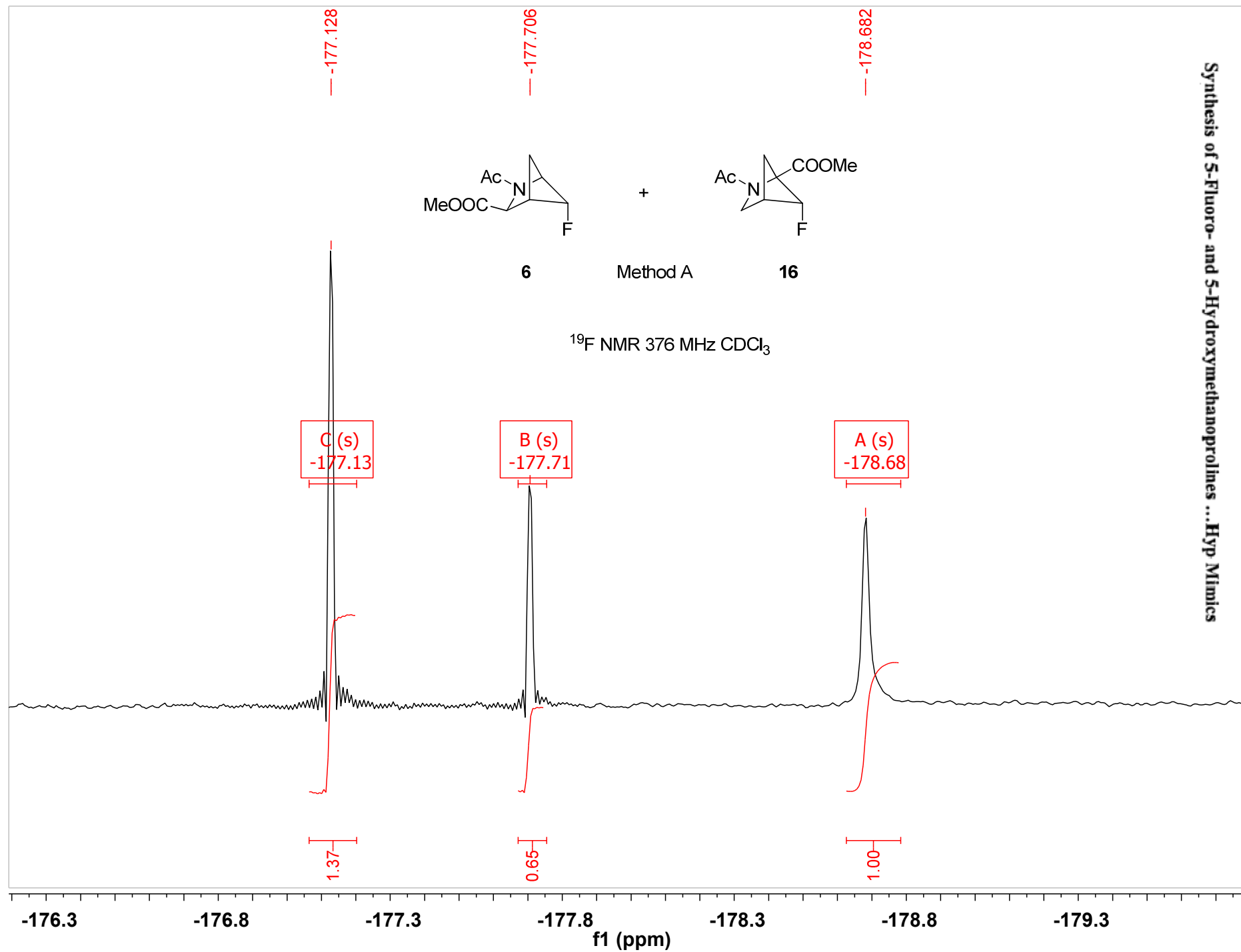
EX: s2pul

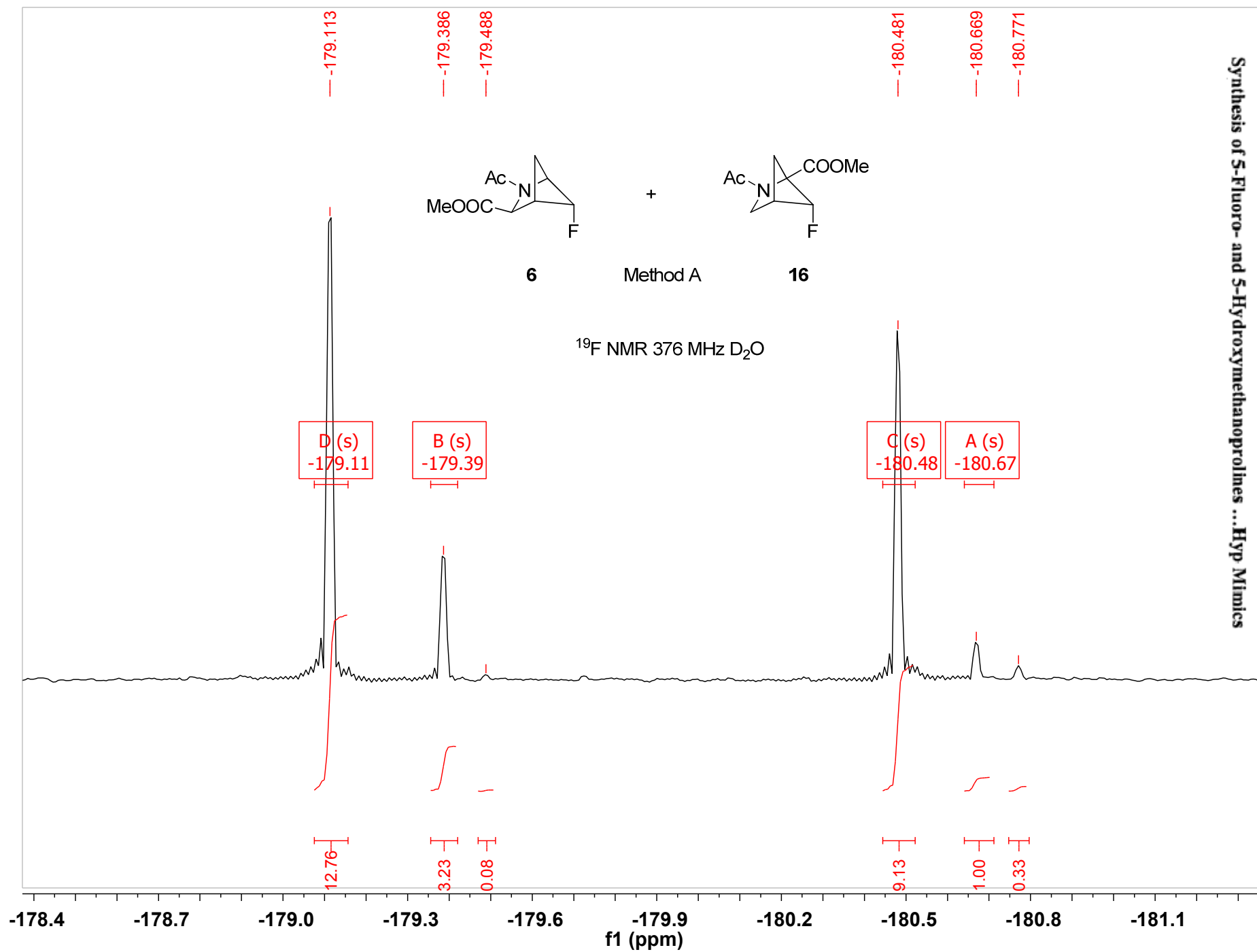
PW: 5.7 usec

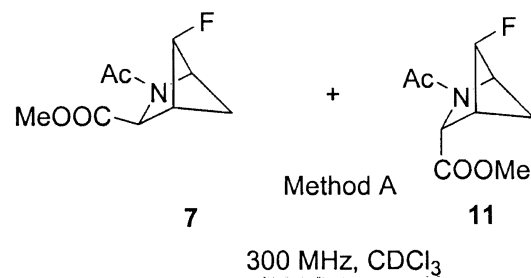
PD: 2.0 sec

NA: 10000

LB: 3.0 WinNuts - NAc[211]syn-F ester fluorine in D₂O carbon t1st





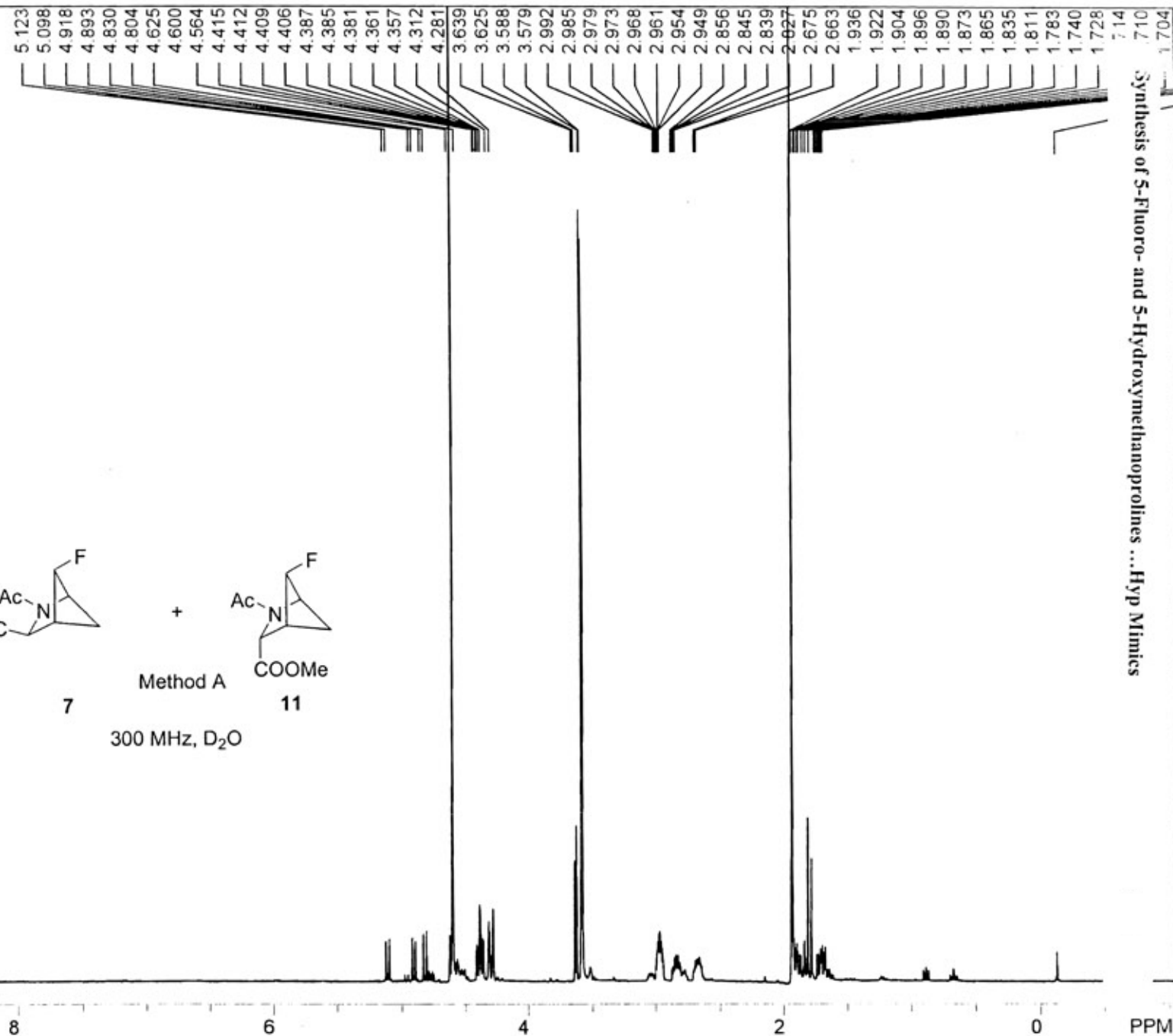
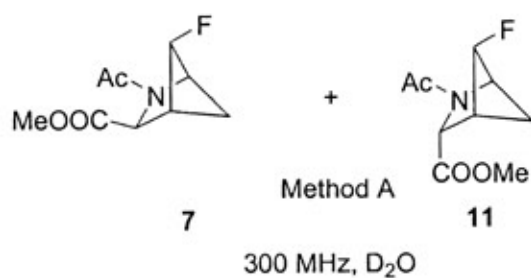


10 8 6 4 2 0 PPM

deepa/f42_21Nov2006

USER: -- DATE: Nov 21 2006

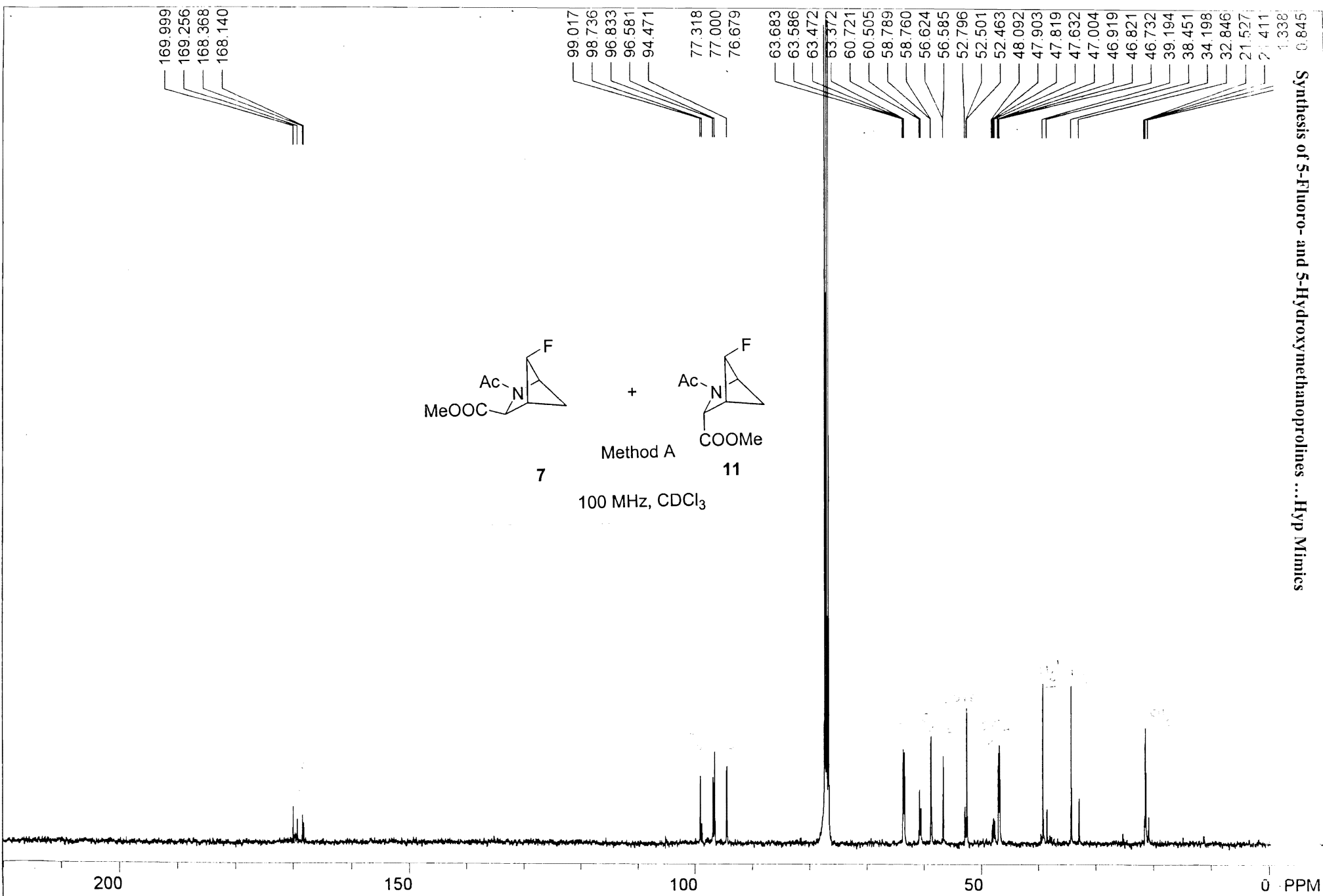
F1: 300.018	F2: 300.018	SW1: 3600	OF1: 1487.5	PTS1d: 16384
EX: s2pul	PW: 4.0 usec	PD: 1.0 sec	NA: 8	LB: 0.2
WinNuts - NAc[211]-5-F-3-Co2Me mix				



deepa/1000_26Sep2006

USER: -- DATE: Sep 26 2006

F1: 300.019	F2: 300.019	SW1: 3600	OF1: 1480.2	PTS1d: 16384
EX: s2pul	PW: 4.0 usec	PD: 1.0 sec	NA: 8	LB: 0.2 WinNuts - NAc[211]-5-anti-F-3-Co2Me mix in D2O proton.txt



NBAc[211]5-F-3-Co2Me mix, carbonOVNTE, atm.

USER: temple -- DATE: Aug 8 05:50:09 2006 EDT (UT-4h) templ

F1: 100.628

F2: 1.000

SW1: 23148

OF1: 10585.1

PTS1d: 32768

EX:

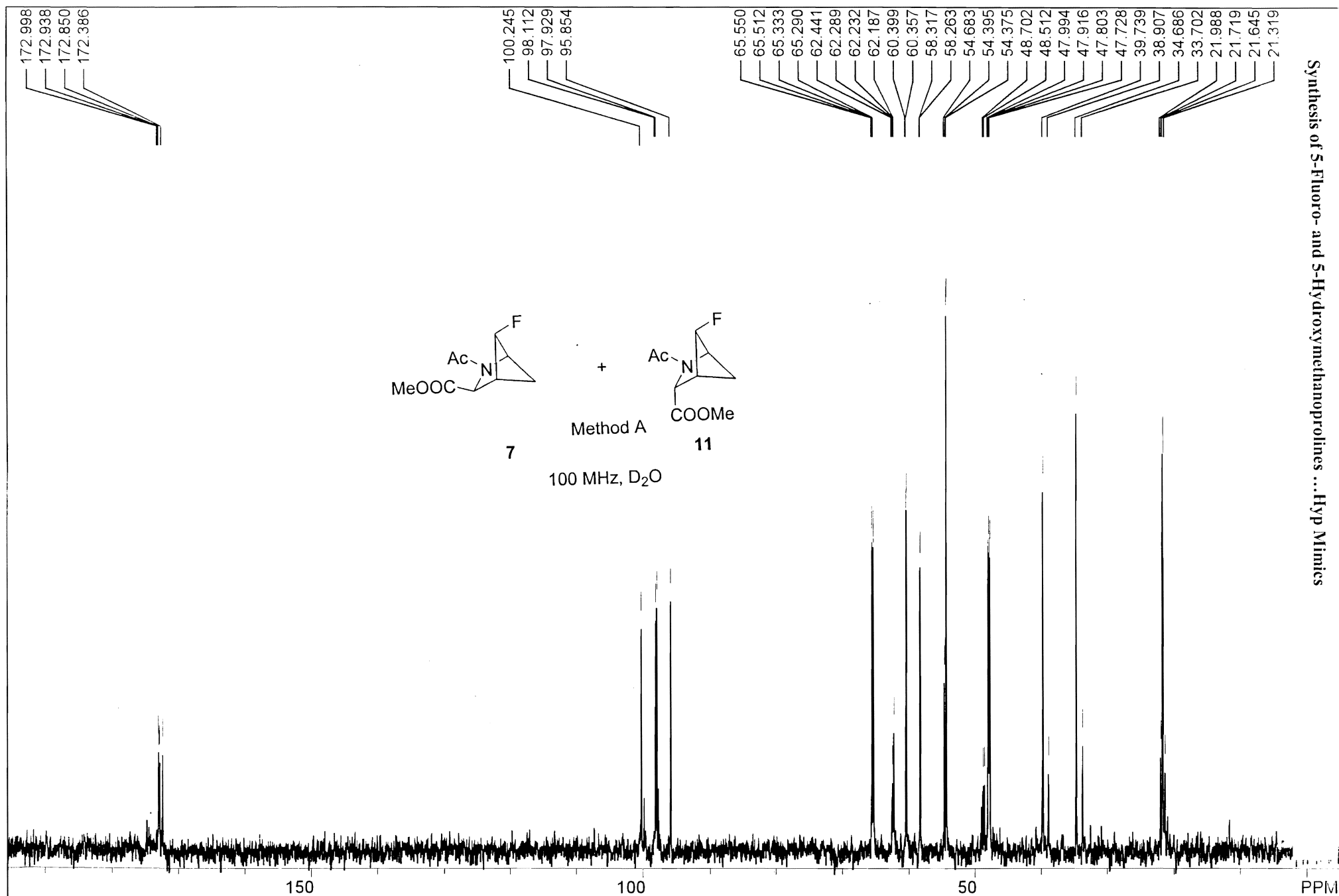
PW: 5.7 usec

PD: 2.0 sec

NA: 10000

LB: 3.0

WinNuts - NBAc[211]-5-anti-F-3-Co2Me mix carbon



NAC[211]-1-CO2Me-5F. carbonOVNTE.atm.

USER: temple -- DATE: Apr 21 06:28:43 2007 EDT (UT-4h) templ

F1: 100.628

F2: 1.000

SW1: 23148

OF1: 10700.9

PTS1d: 32768

EX:

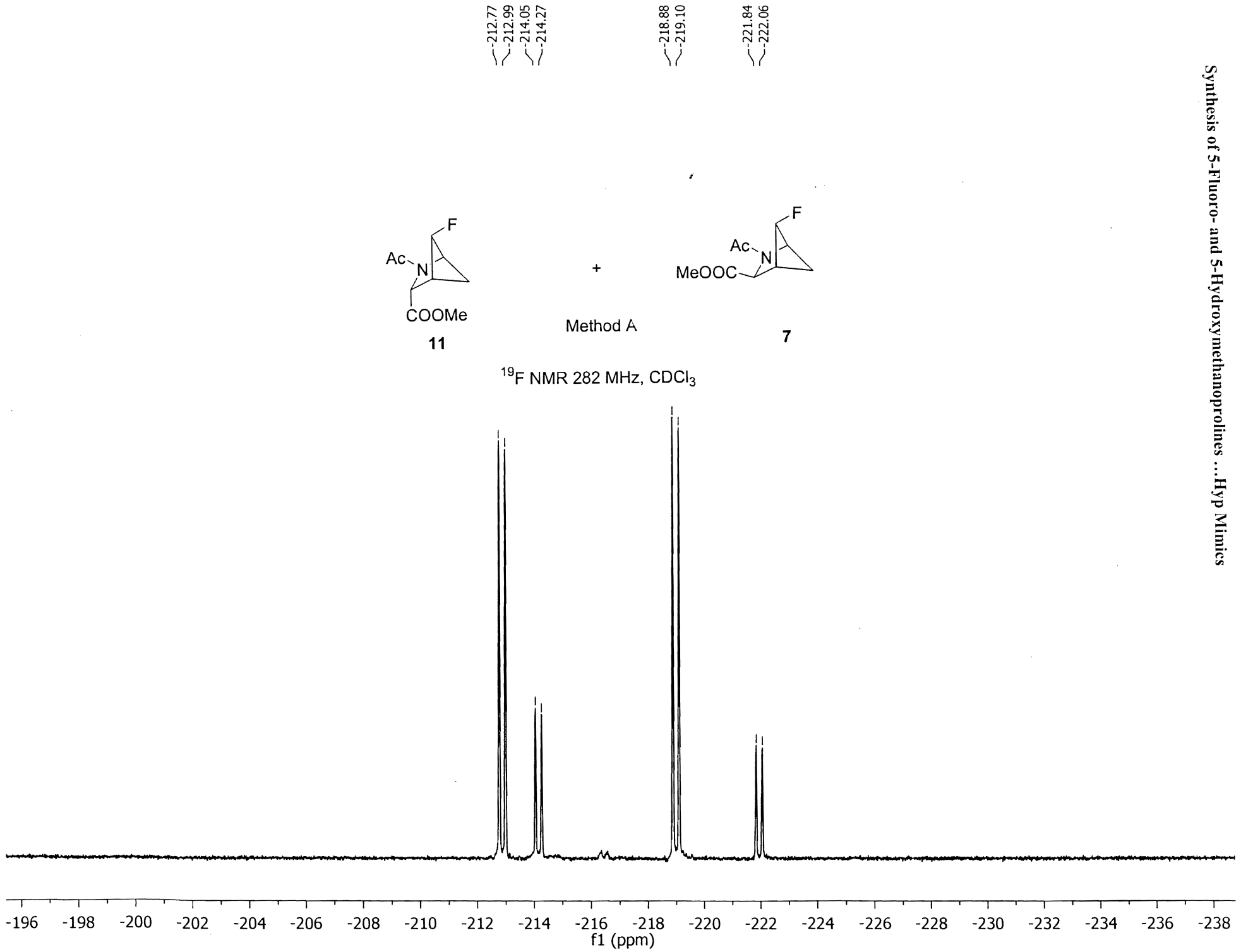
PW: 5.7 usec

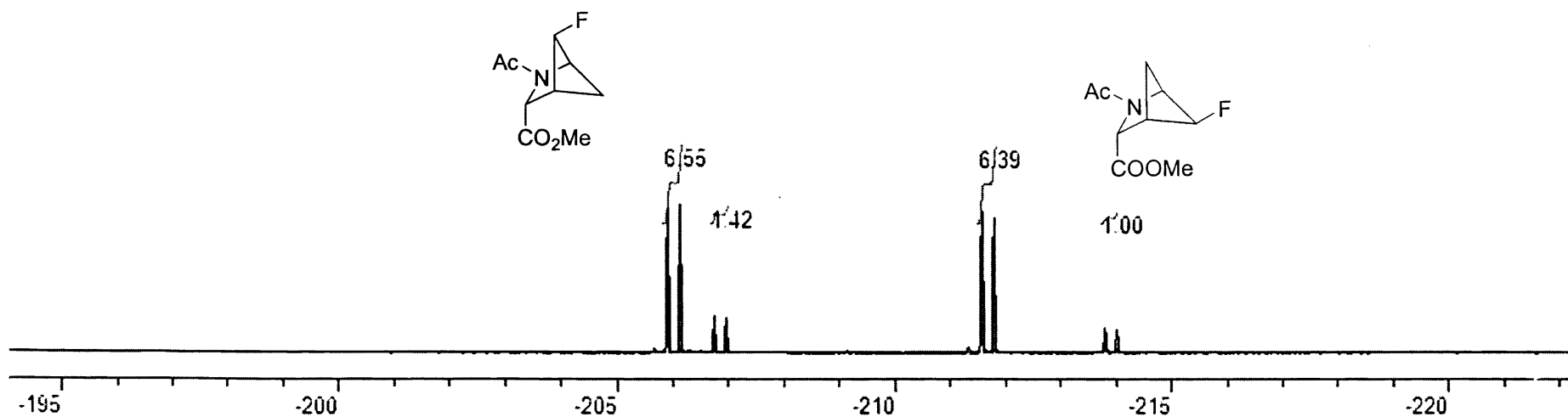
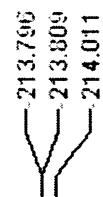
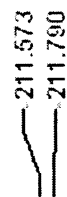
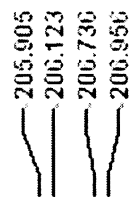
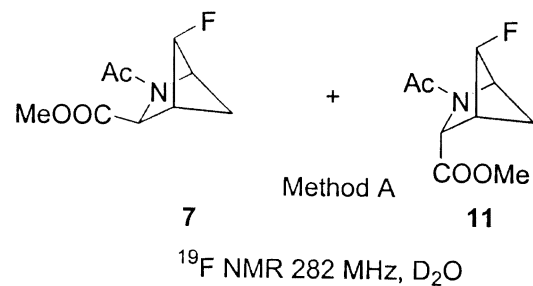
PD: 2.0 sec

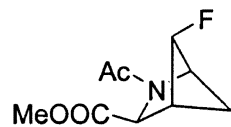
NA: 10000

LB: 3.0

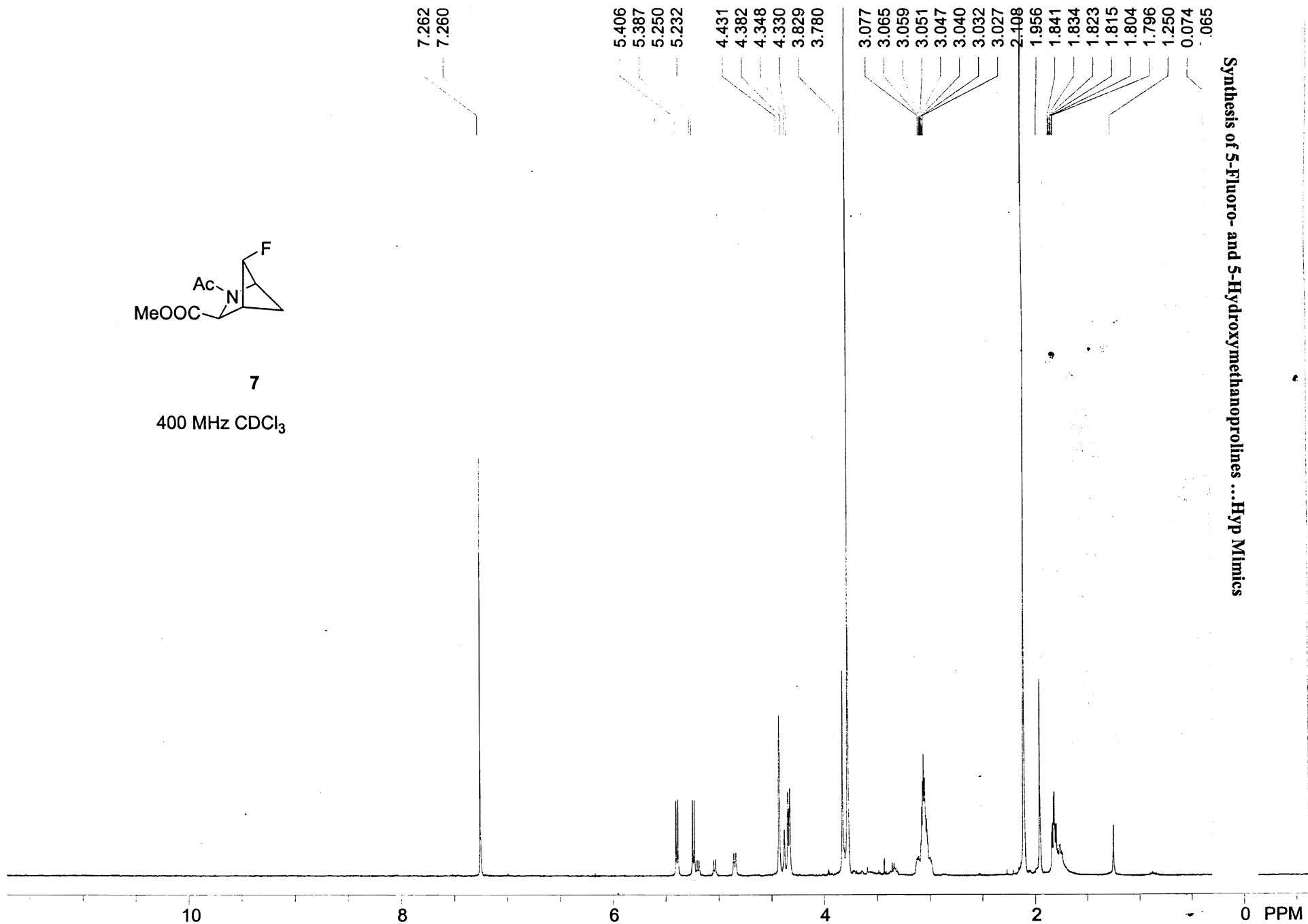
WinNuts - anti f nmr carbon in d2o.txt



^{19}F NMR in D_2O 



7

400 MHz CDCl₃

3-endo-CO2Me-NAc[2.1.1] anti-F, proton 10MIN.atm.

USER: temple -- DATE: Oct 28 09:54:34 2008 EST (UT-5h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2004.2

PTS1d: 32768

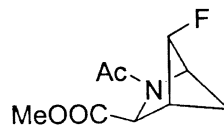
EX:

PW: 9.8 us

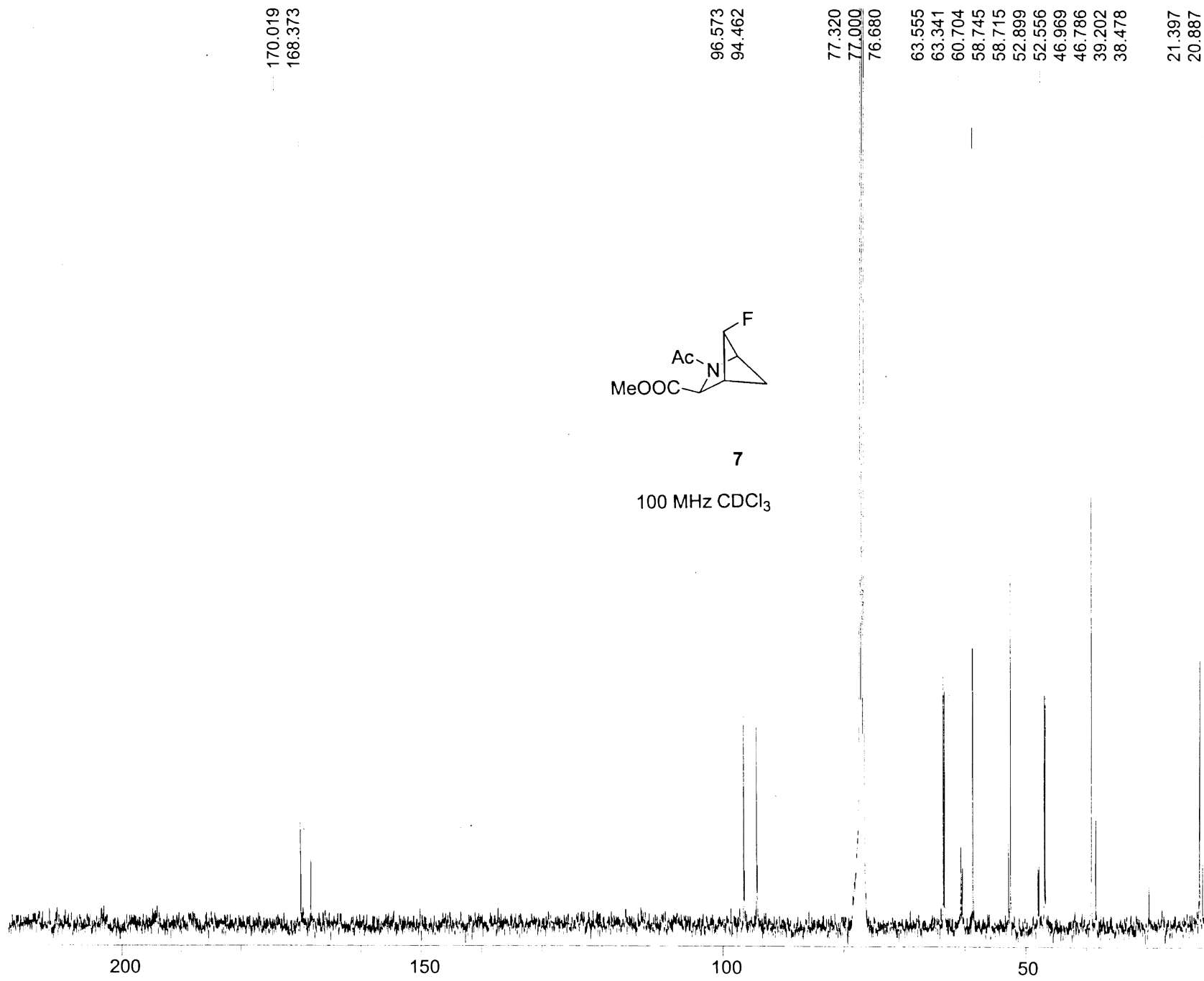
PD: 1.0 sec

NA: 328

NBts: 3-endo-CO2Me NAc[2.1.1] anti-F--H-CDCl3--eram-1625



7

100 MHz CDCl₃3-endo-CO₂Me NAc[2.1.1] anti-F, carbonOVNTE.all,

USER: temple -- DATE: Oct 30 06:53:26 2008 EST (UT-5h) templ

F1: 100.613 F2: 1.000

SW1: 25126

OF1: 10021.9

PTS1d: 32768

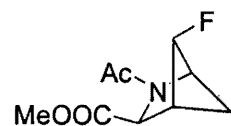
EX:

PW: 5.7 us

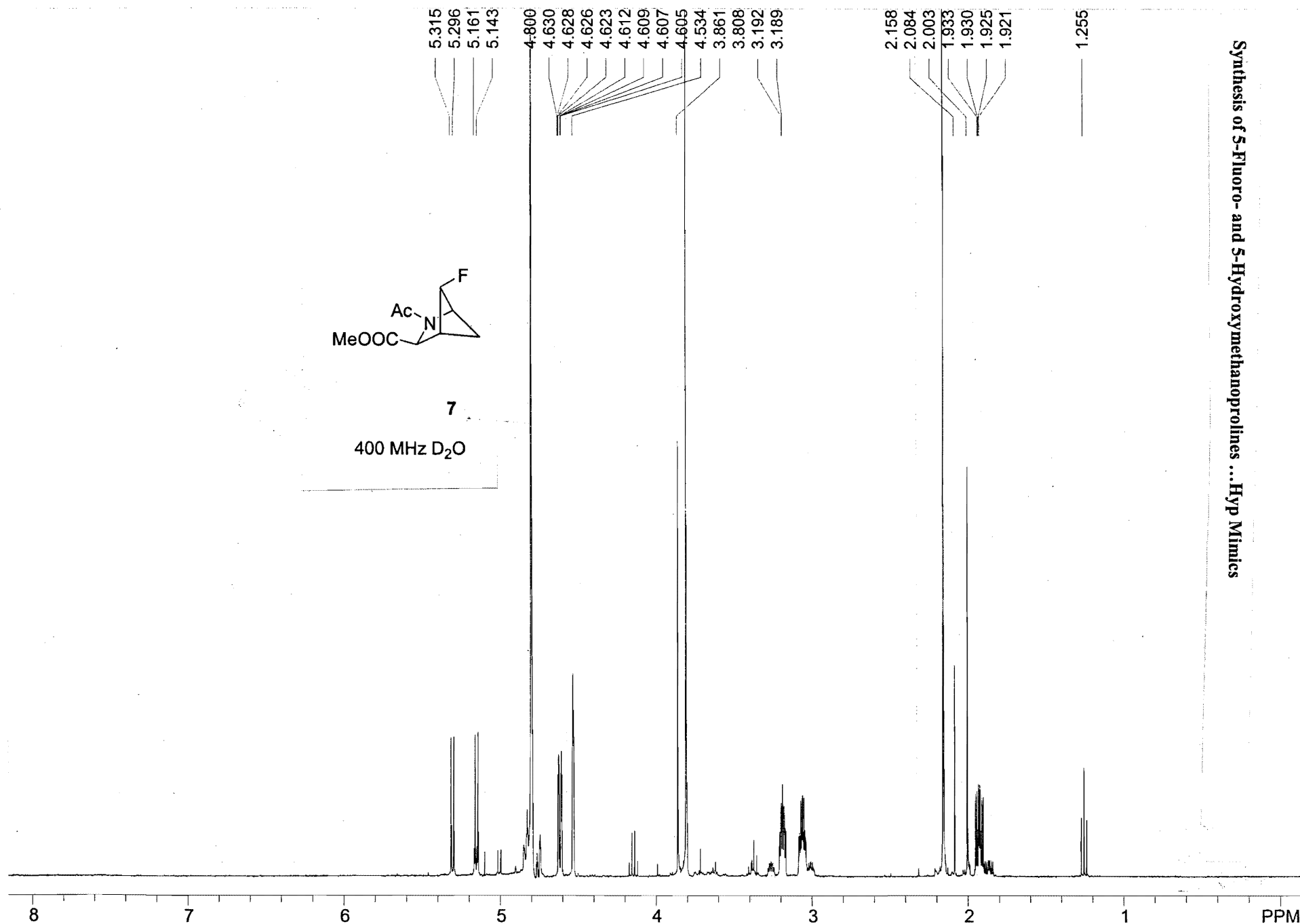
PD: 2.0 sec

NA: 12500

NuBs 3-endo-3-CO₂Me NAc[2.1.1] anti-F--CDCl₃--C--eram1629



7

400 MHz D₂O3-endo-CO₂Me NAc[2.1.1] anti-F, proton10MIN.atm,

USER: temple -- DATE: Nov 16 15:38:38 2008 EST (UT-5h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2041.0

PTS1d: 32768

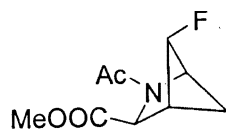
EX:

PW: 9.8 us

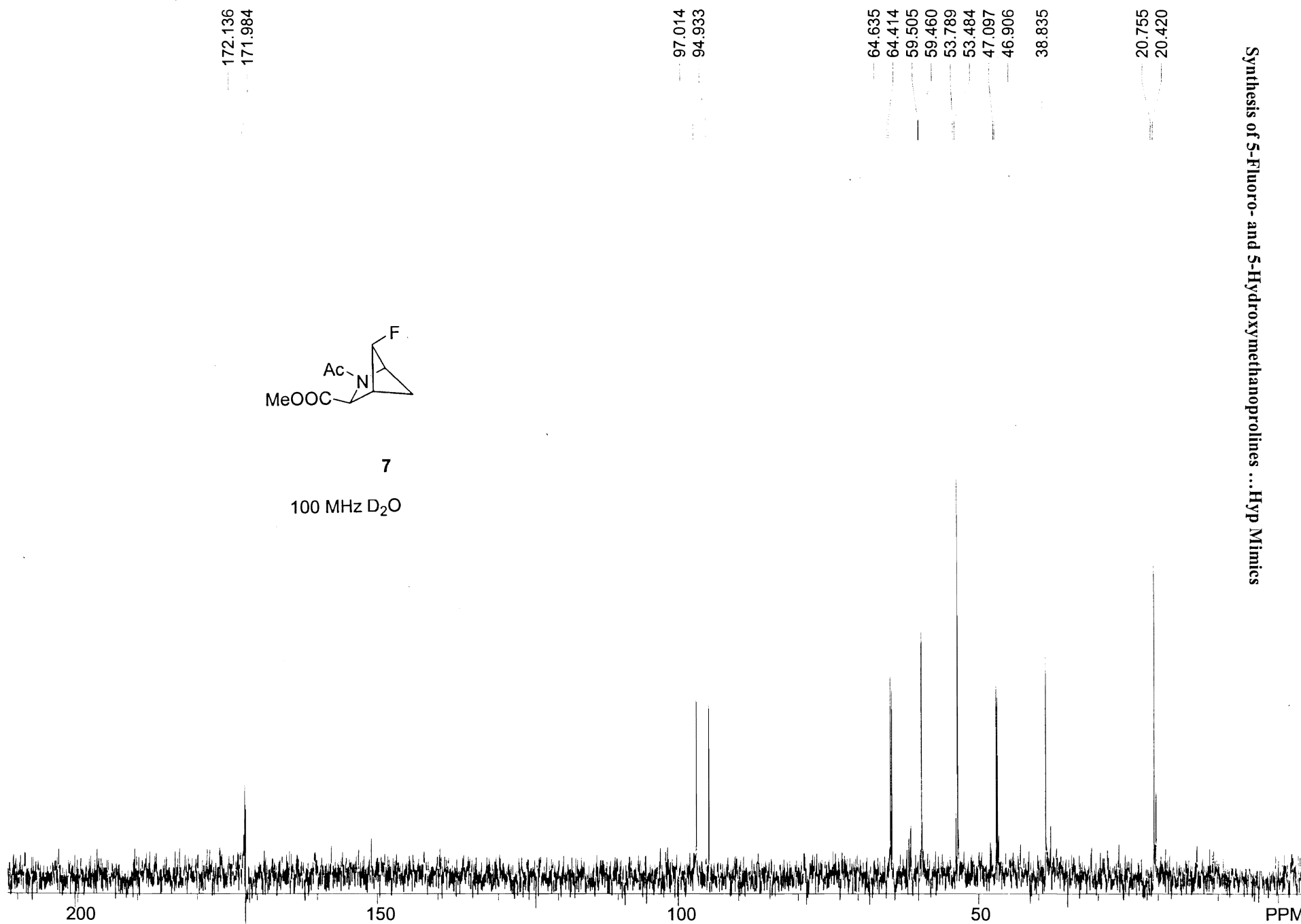
PD: 1.0 sec

NA: 500

LB: 3-endo-3-CO₂Me NAc[2.1.1] anti-F--H-D₂O--eram-1666



7

100 MHz D₂Oendo-3-CO₂Me NAc[2.1.1] anti-F, carbonOVNTE.all,

USER: temple -- DATE: Mar 27 08:29:26 2009 EST (UT-5h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10060.0

PTS1d: 32768

EX: zgpg60

PW: 5.7 us

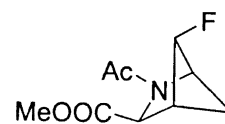
PD: 3.0 sec

NA: 11000

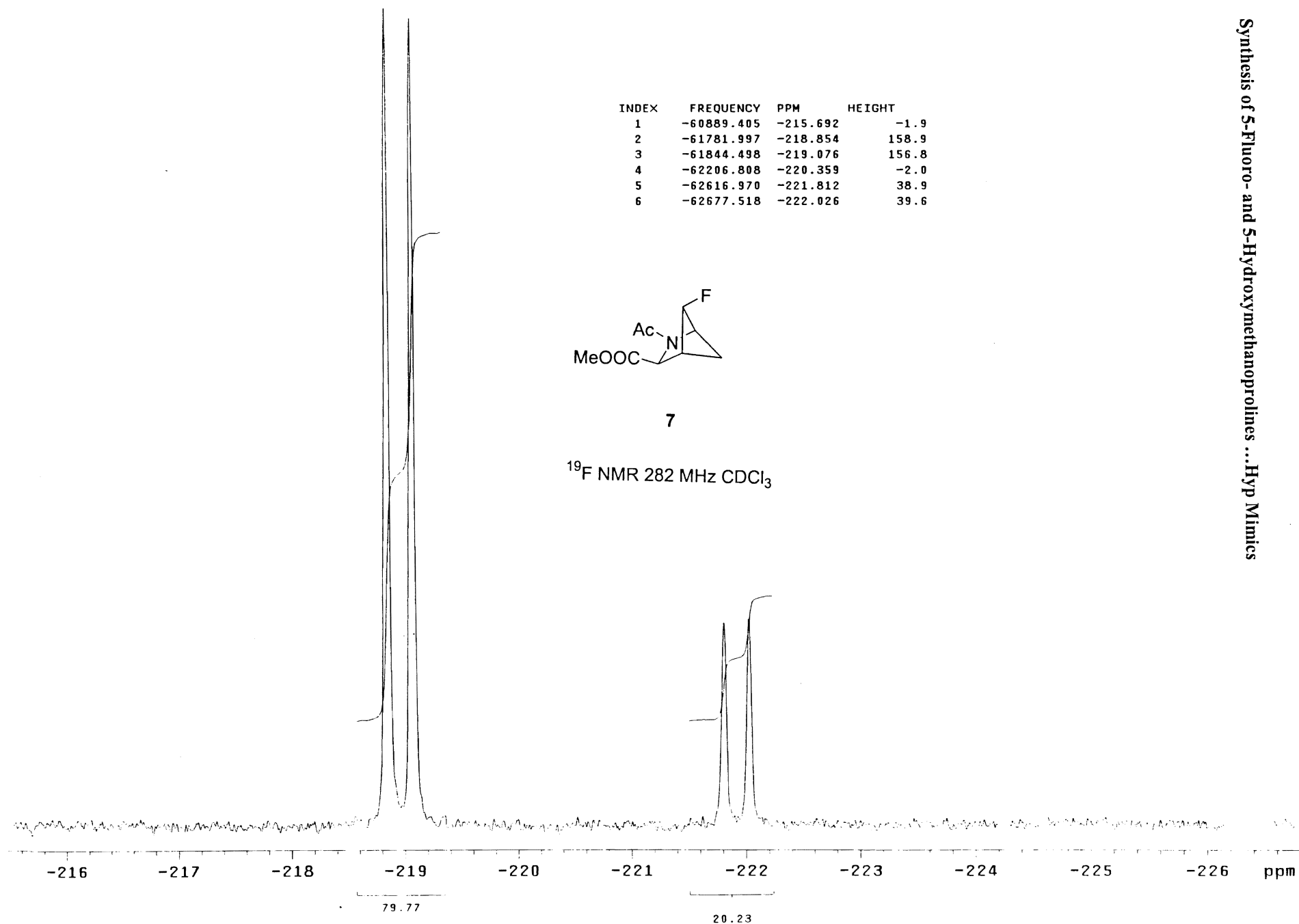
LB: 3.0

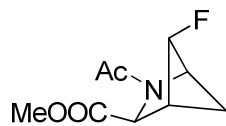
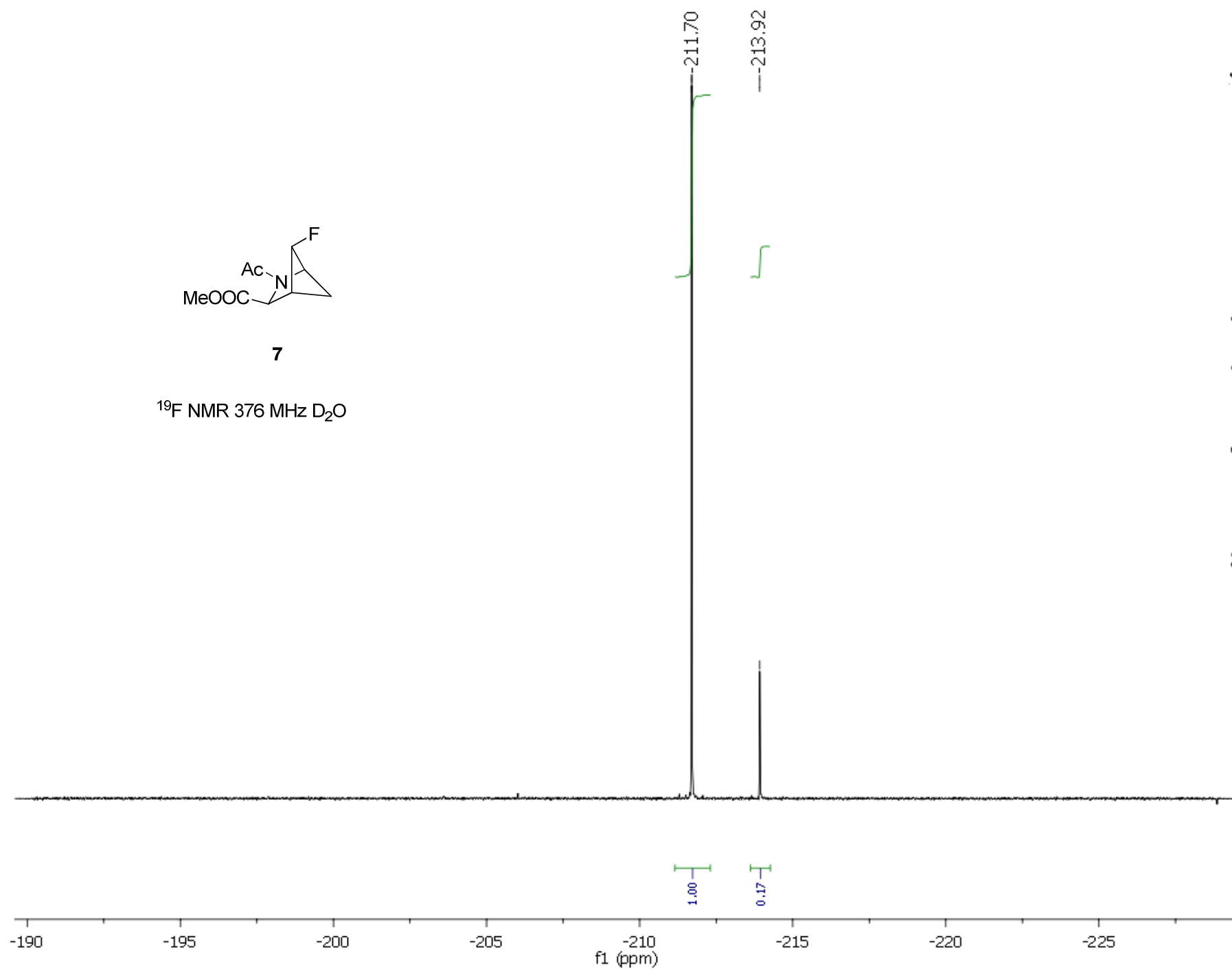
Nuts - \$WS_FTP_2101.1

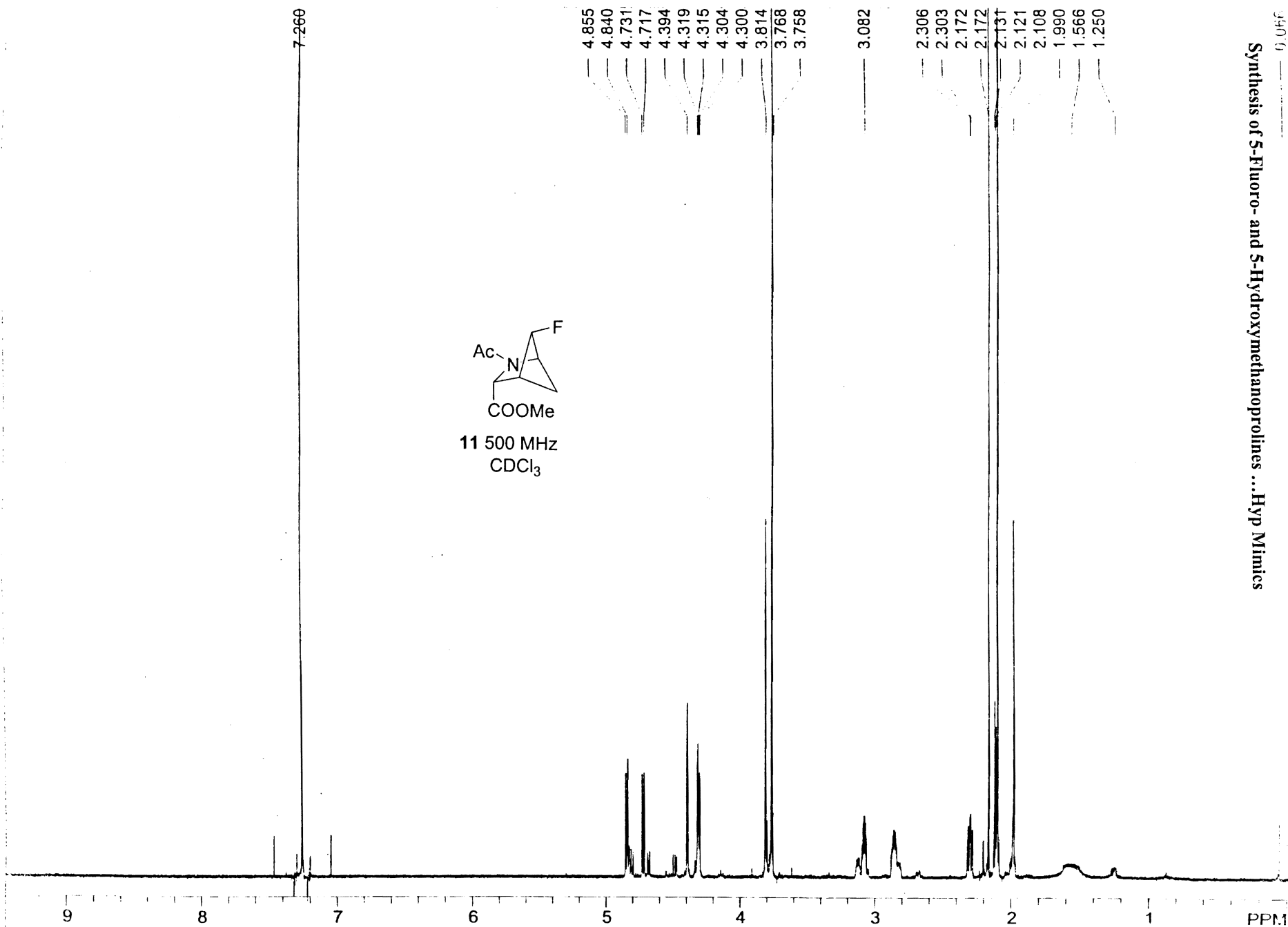
INDEX	FREQUENCY	PPM	HEIGHT
1	-60889.405	-215.692	-1.9
2	-61781.997	-218.854	158.9
3	-61844.498	-219.076	156.8
4	-62206.808	-220.359	-2.0
5	-62616.970	-221.812	38.9
6	-62677.518	-222.026	39.6



7

 ^{19}F NMR 282 MHz CDCl_3 

**7** ^{19}F NMR 376 MHz D_2O 

Ram 1619 1910 in CDCl₃, no reference spectrum.

USER: nmrsu -- DATE: 19:12:40.843 -0500 nmrsu@BH1076407

F1: 500.130

F2: 1.000

SW1: 6010

OF1: 1742.6

PTS1d: 131072

EX: zg30

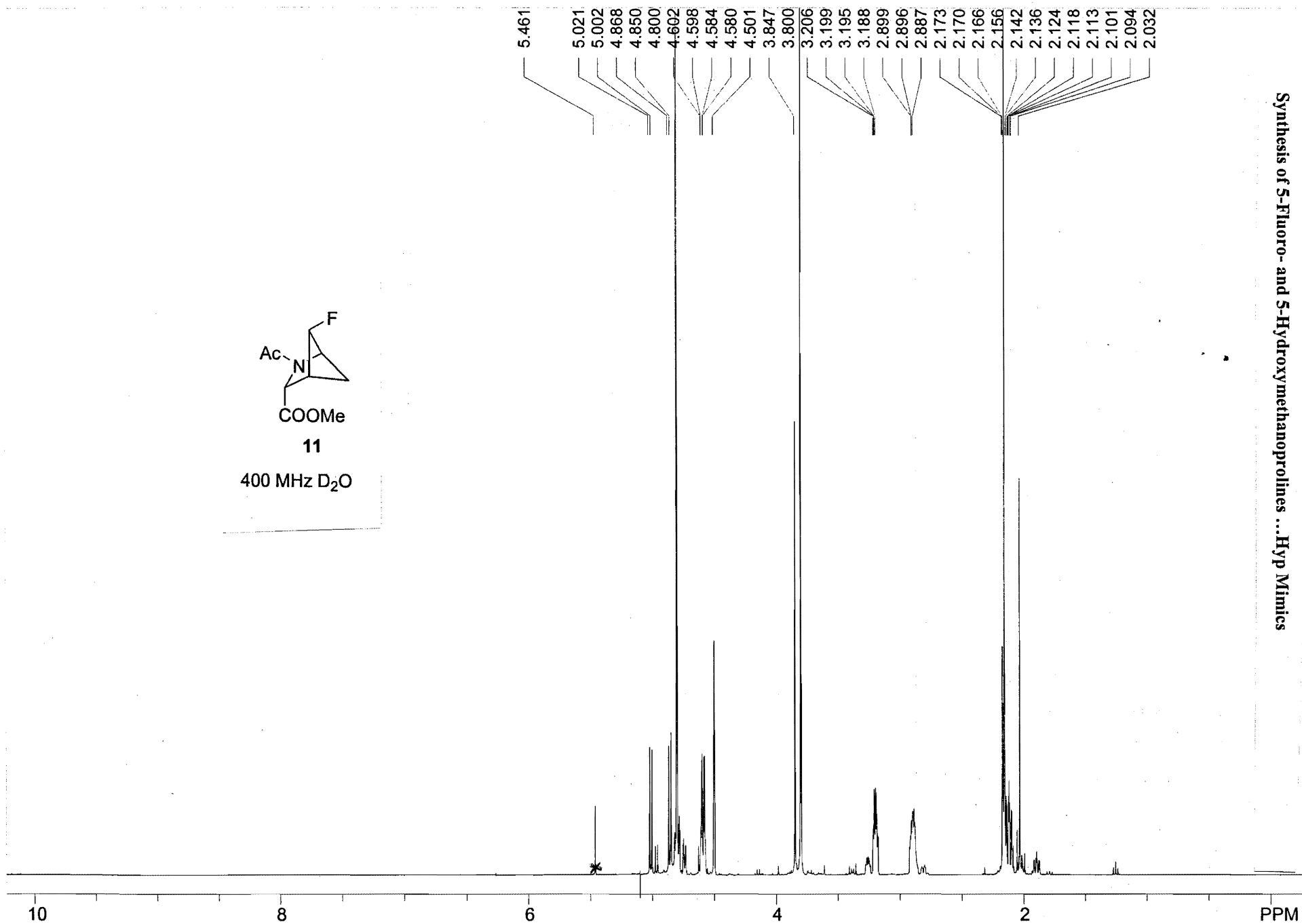
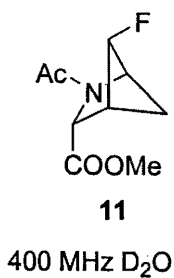
PW: 8.3 us

PD: 6.0 sec

NA: 16

LB: -0.1

Nuts - \$1 5830.1

3-exo-CO₂Me NAc[2.1.1] anti-F, proton 10 MIN. atm.

USER: temple -- DATE: Nov 16 17:28:44 2008 EST (UT-5h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2041.2

PTS1d: 32768

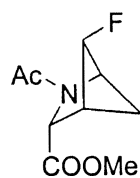
EX:

PW: 9.8 us

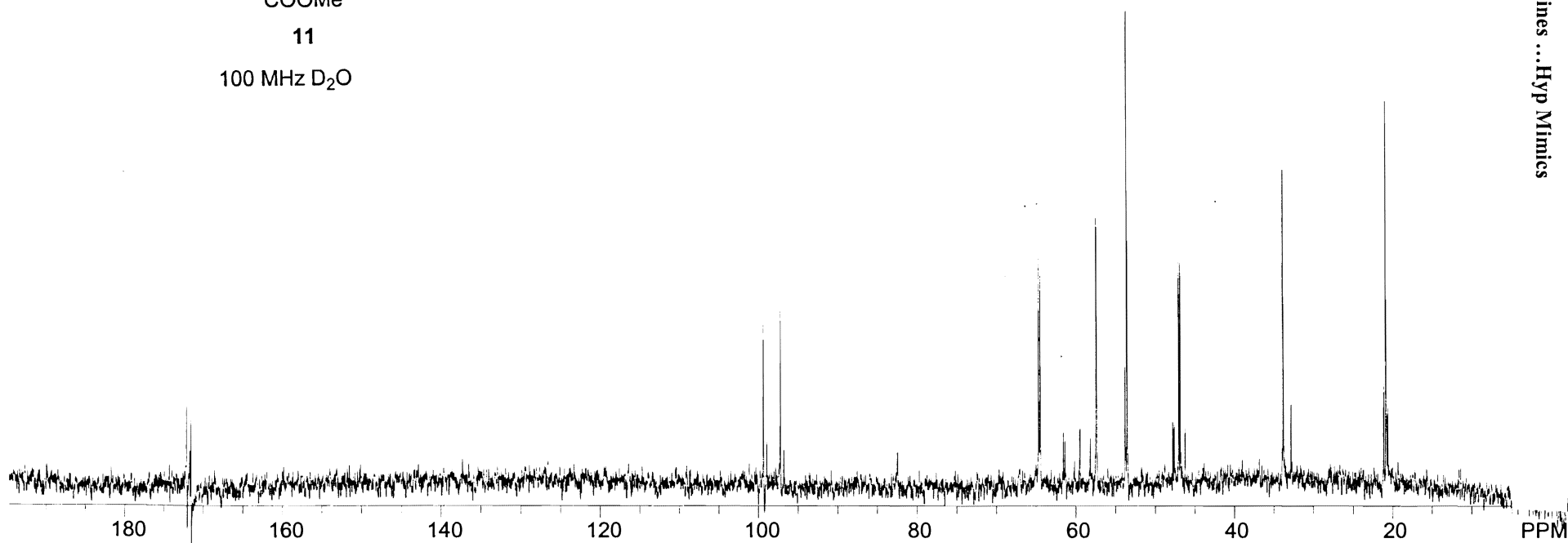
PD: 1.0 sec

NA: 528

LB: 104 - Exo-3-CO₂Me NAc[2.1.1] anti-F--D₂O--H--eram-1667



11

100 MHz D₂O

3-exo-NAc[2.1.1] anti-F, carbonOVNTE.all,

USER: temple -- DATE: Mar 29 10:14:19 2009 EST (UT-5h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10060.0

PTS1d: 32768

EX: zgpg60

PW: 5.7 us

PD: 3.0 sec

NA: 11000

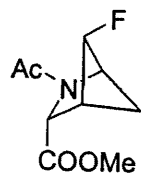
LB: 3.0

Nuts - \$WS_FTP_2011.1

charlie/F19forRam_24Jan2007

exp1 s2pu1

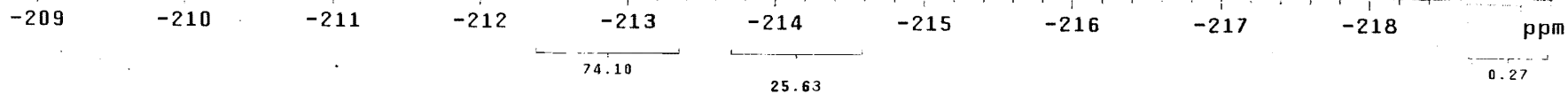
SAMPLE SPECIAL
 date Nov 11 2008 temp not used
 solvent CDCl3 gain not used
 file /home/temple/~ spin not used
 eram15-71F.fid hst 0.008
 ACQUISITION pw90 23.000
 sw 64000.0 alfa 82.400
 at 0.505
 np 64592 11
 fb 35000 in
 bs 16 dp
 dl 5.000 hs
 nt 4500
 ct 4500
 TRANSMITTER F19
 tn sfrq 282.222 1s fid
 tof 20000.0 fn 131072
 tpwr 56
 pw 7.667
 DECOUPLER H1
 dn dof 0
 dm nnn
 dmm c
 dpwr 38
 dmf 11136
 wc 250
 sc 0
 vs 981
 th 0
 al no ph



11

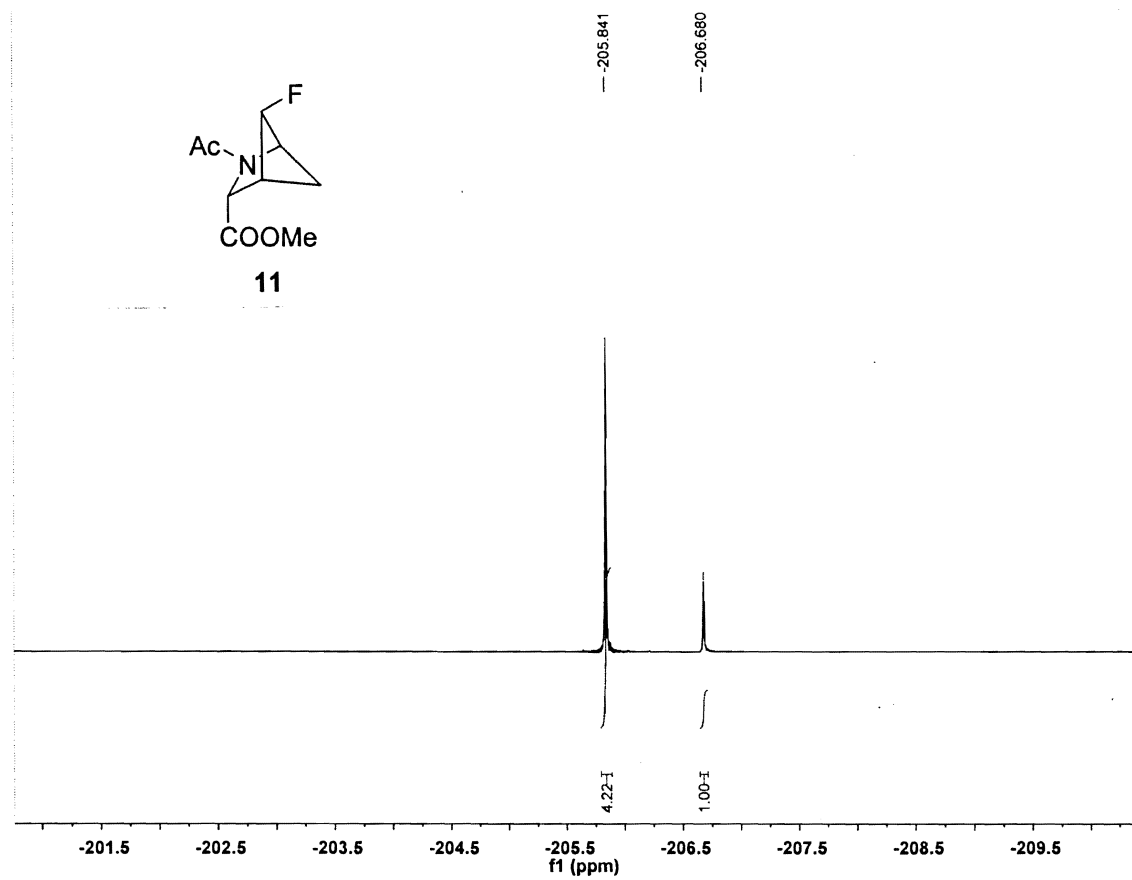
¹⁹F NMR 282 MHz CDCl₃

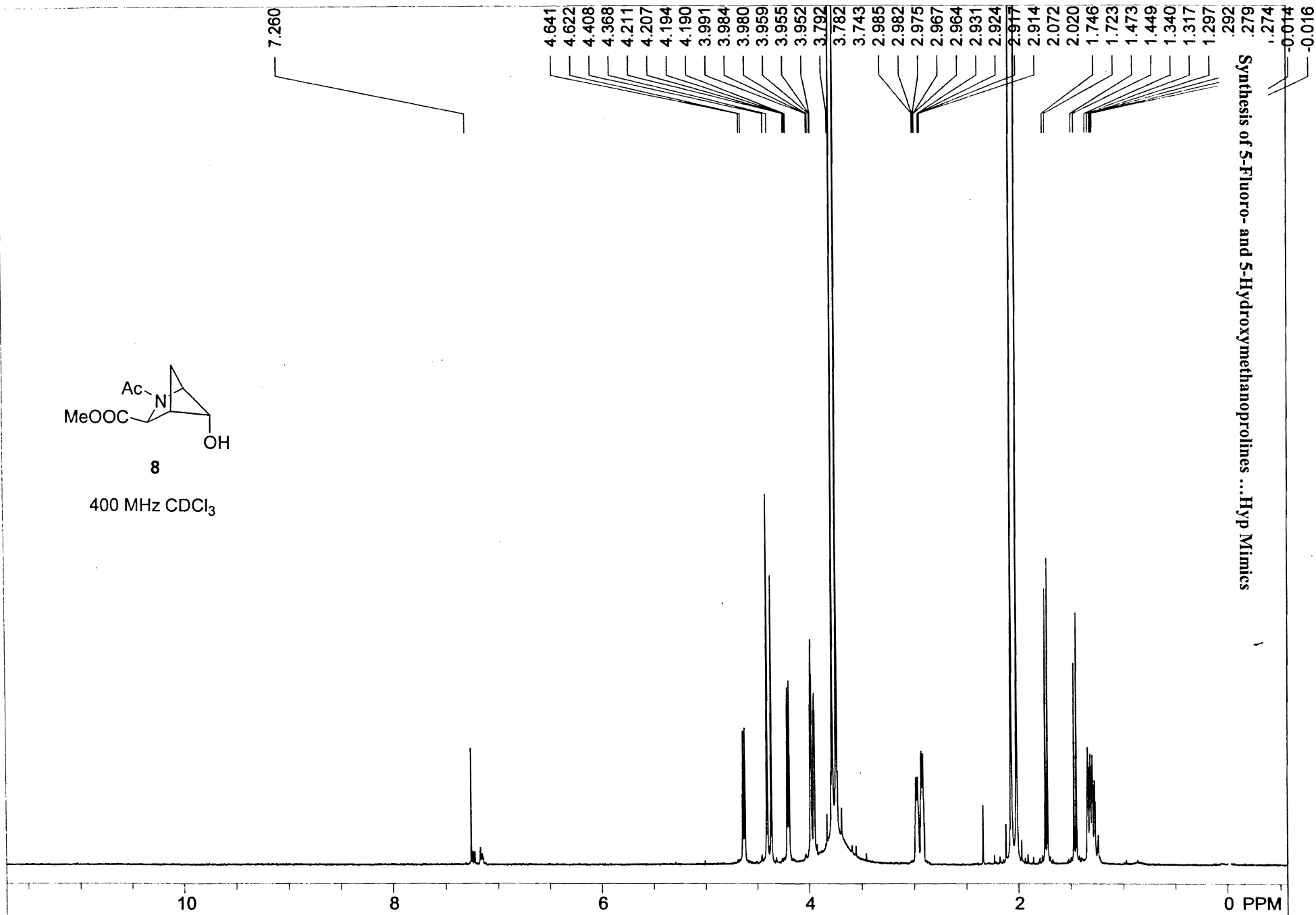
INDEX	FREQUENCY	PPM	HEIGHT
1	-59087.620	-209.310	-0.4
2	-59972.399	-212.444	1.4
3	-60056.384	-212.741	149.7
4	-60117.909	-212.959	149.0
5	-60193.105	-213.226	1.1
6	-60222.403	-213.330	-0.4
7	-60253.653	-213.440	0.9
8	-60322.990	-213.686	-0.4
9	-60416.742	-214.018	50.3
10	-60478.266	-214.236	49.9
11	-60704.832	-215.039	-0.3
12	-60826.904	-215.471	0.7
13	-61211.676	-216.834	-0.6
14	-61781.997	-218.854	1.0
15	-61829.849	-219.024	-0.3
16	-61841.568	-219.065	1.0



Synthesis of 5-Fluoro- and 5-Hydroxymethanoproline... Hyp Mimics

^{19}F NMR (376 MHz, D_2O) δ -205.84 (s), -206.68 (s).



400 MHz CDCl₃

3-CO2Me NAc[2.1.1] syn-OH, proton.atm.

USER: temple -- DATE: May 11 21:16:00 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8013

OF1: 2004.3

PTS1d: 32768

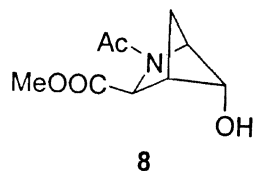
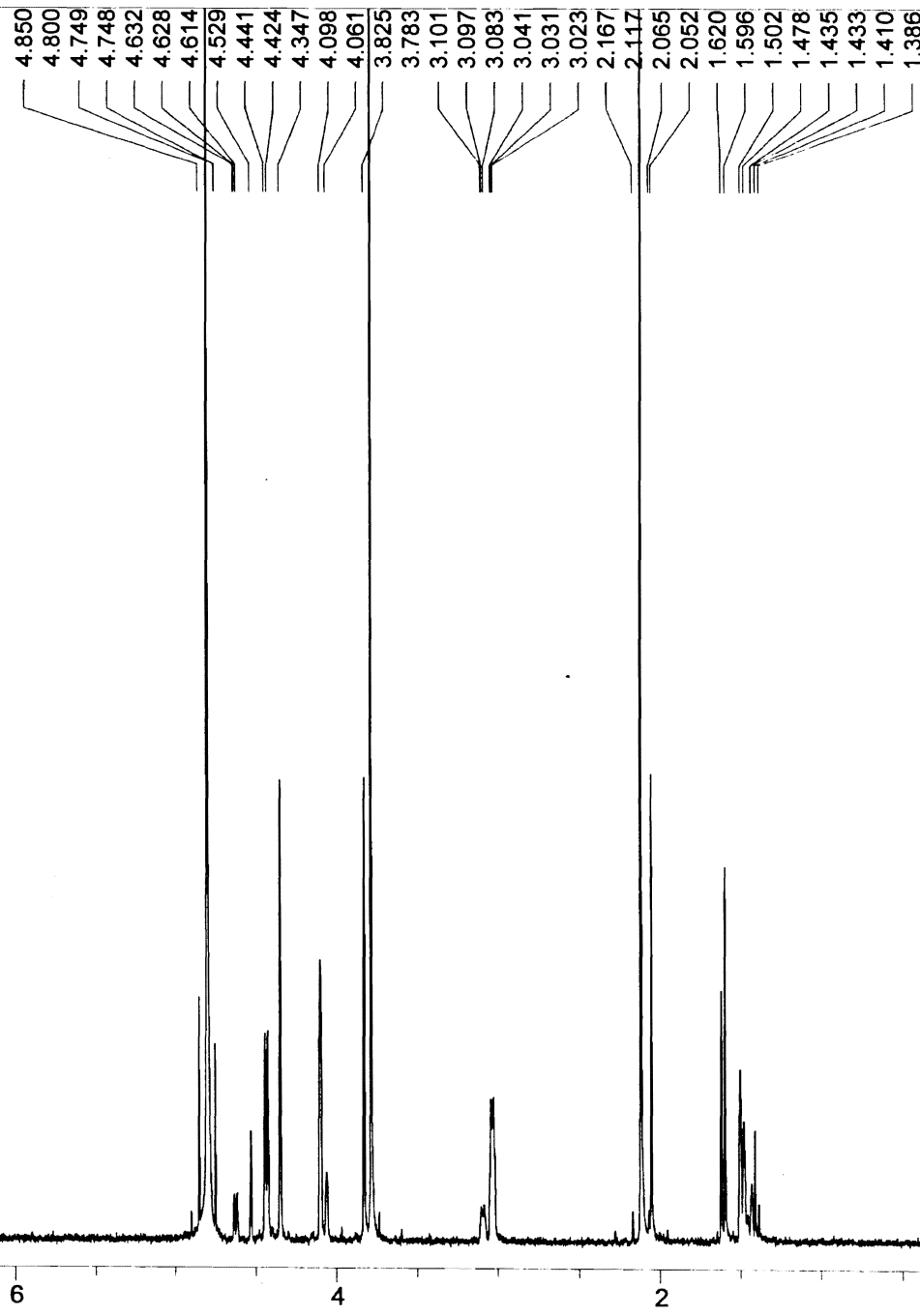
EX:

PW: 10.8 us

PD: 1.0 sec

NA: 16

100-13-CO2Me NAc[2.1.1] syn-OH---H (CDCl3)--eram-1160

400 MHz D₂O3-CO₂Me NAc[2.1.1] syn-OH, proton10MIN.atm,

USER: temple -- DATE: May 13 10:01:27 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2040.5

PTS1d: 32768

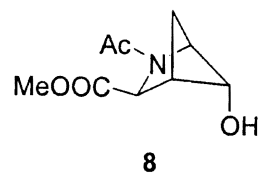
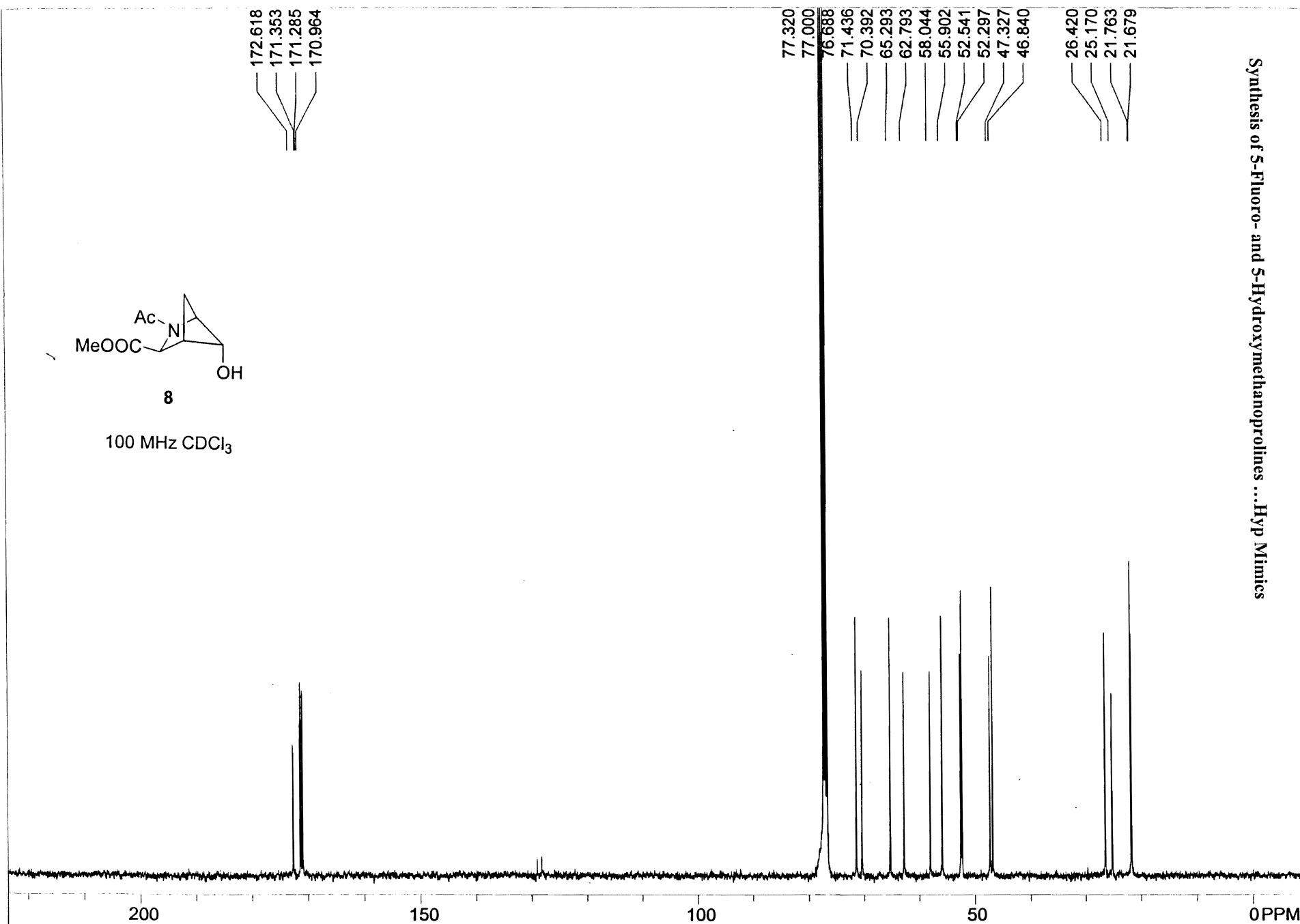
EX:

PW: 9.8 us

PD: 1.0 sec

NA: 128

LBNHts - 3-CO₂Me NAc[2.1.1] syn-OH---H (D₂O)--eram-1176

100 MHz CDCl₃

3-CO2Me NAc[2.1.1] syn-OH, carbonOVNTE.all,

USER: temple -- DATE: May 17 08:02:58 2008 EDT (UT-4h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10019.6

PTS1d: 32768

EX:

PW: 5.7 us

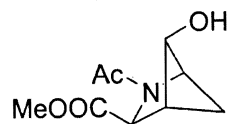
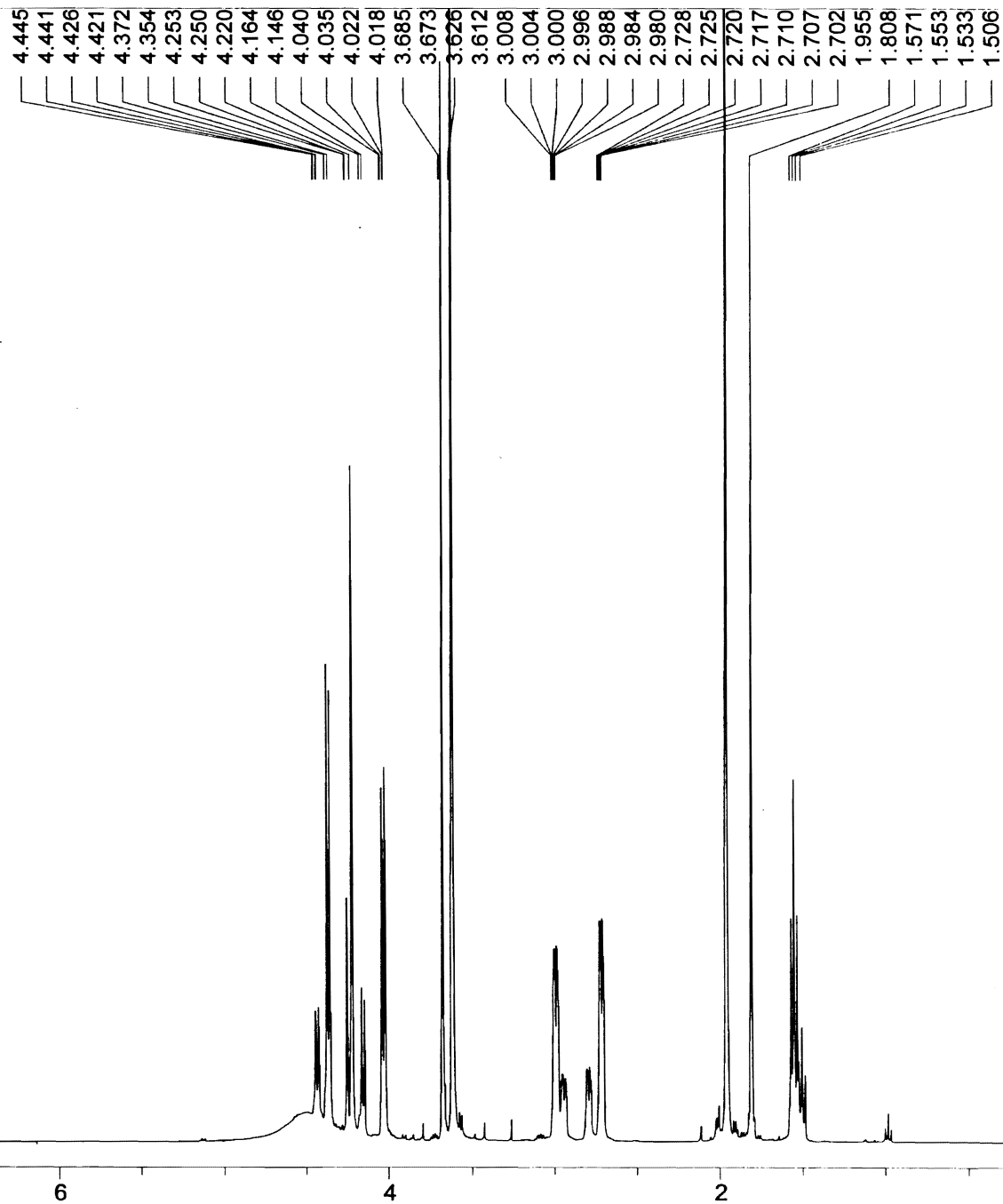
PD: 4.0 sec

NA: 7000

LB: 3.0 Nuts

3-CO2Me NAc[2.1.1] syn-OH---C--eram-1183

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ...Hyp Mimics

**9**400 MHz CDCl₃3-CO₂Me NAc[2.1.1]OH (Minor), proton.atm.

USER: temple -- DATE: Jan 25 21:15:00 2008 EST (UT-5h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2004.9

PTS1d: 32768

EX:

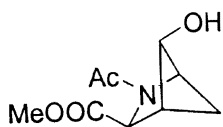
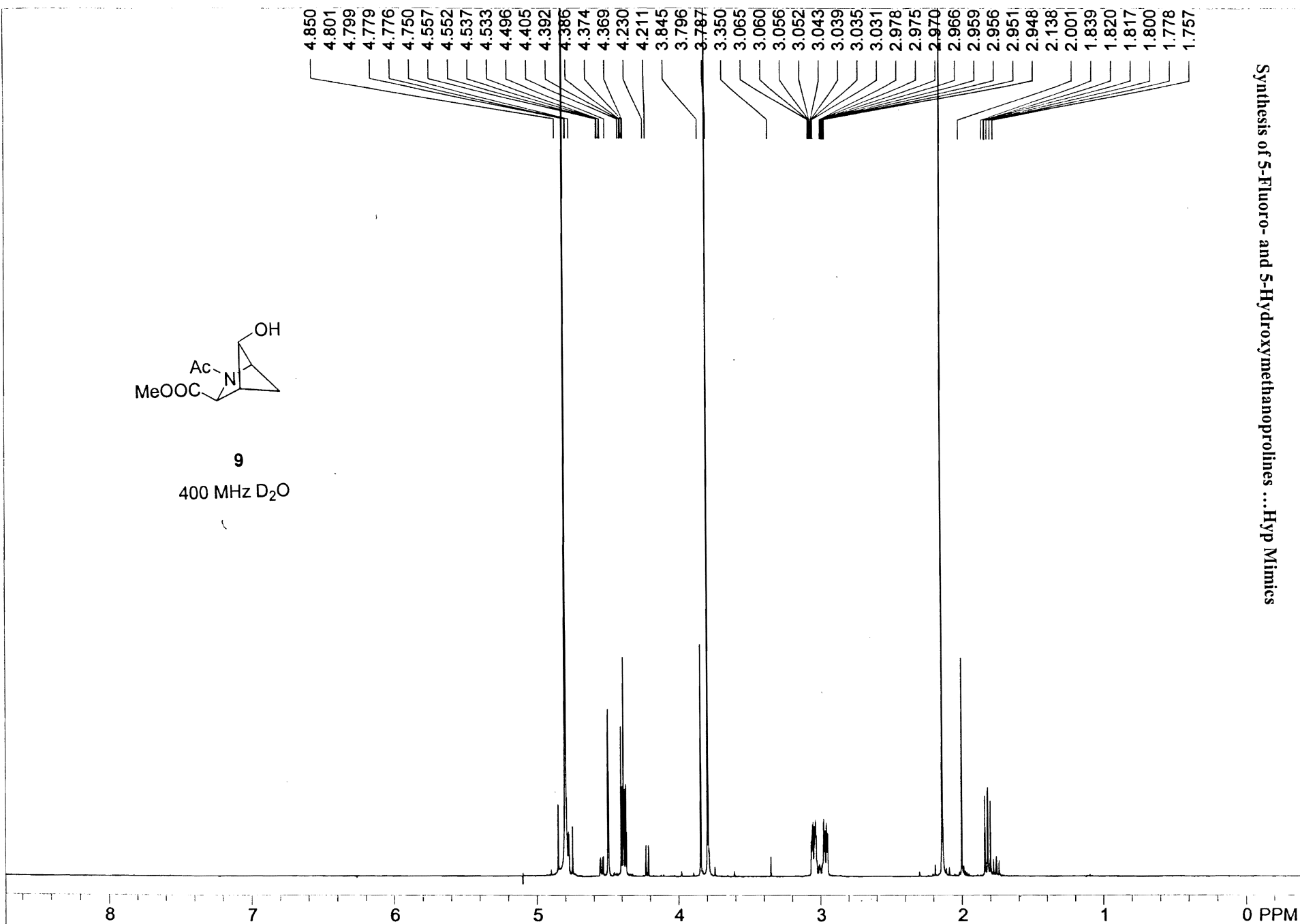
PW: 9.8 us

PD: 1.0 sec

NA: 16

NMR: Brdo -3-CO₂Me NAc[2.1.1] anti-OH--CDCl₃--H--eram822

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ...Hyp Mimics

**9**400 MHz D₂O3-CO₂Me NAc[2.1.1]OH (Minor), proton 10MIN.atm,

USER: temple -- DATE: Feb 15 21:04:28 2008 EST (UT-5h) temp

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2040.5

PTS1d: 32768

EX:

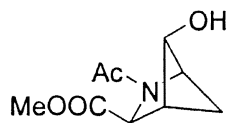
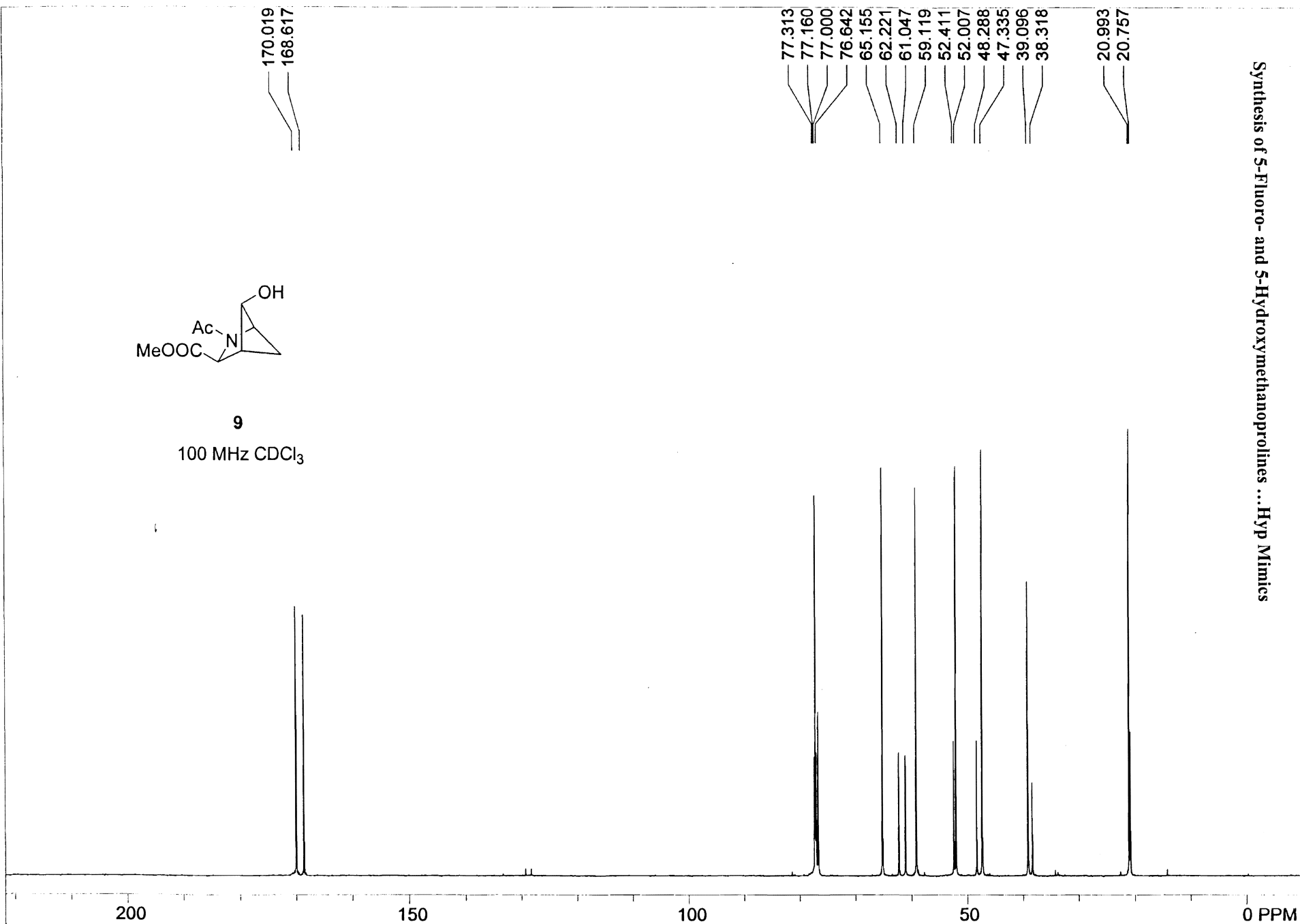
PW: 9.8 us

PD: 1.0 sec

NA: 178

NB: 0. Endo -3-CO₂Me NAc[2.1.1] anti-OH--D₂O--H--eram905

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ...Hyp Mimics

**9**100 MHz CDCl₃

3-CO2Me NAc[2.1.1]OH (Minor), carbon2HR.atm,

USER: temple -- DATE: Jan 26 01:02:41 2008 EST (UT-5h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 9999.6

PTS1d: 32768

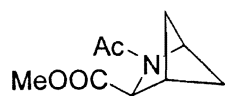
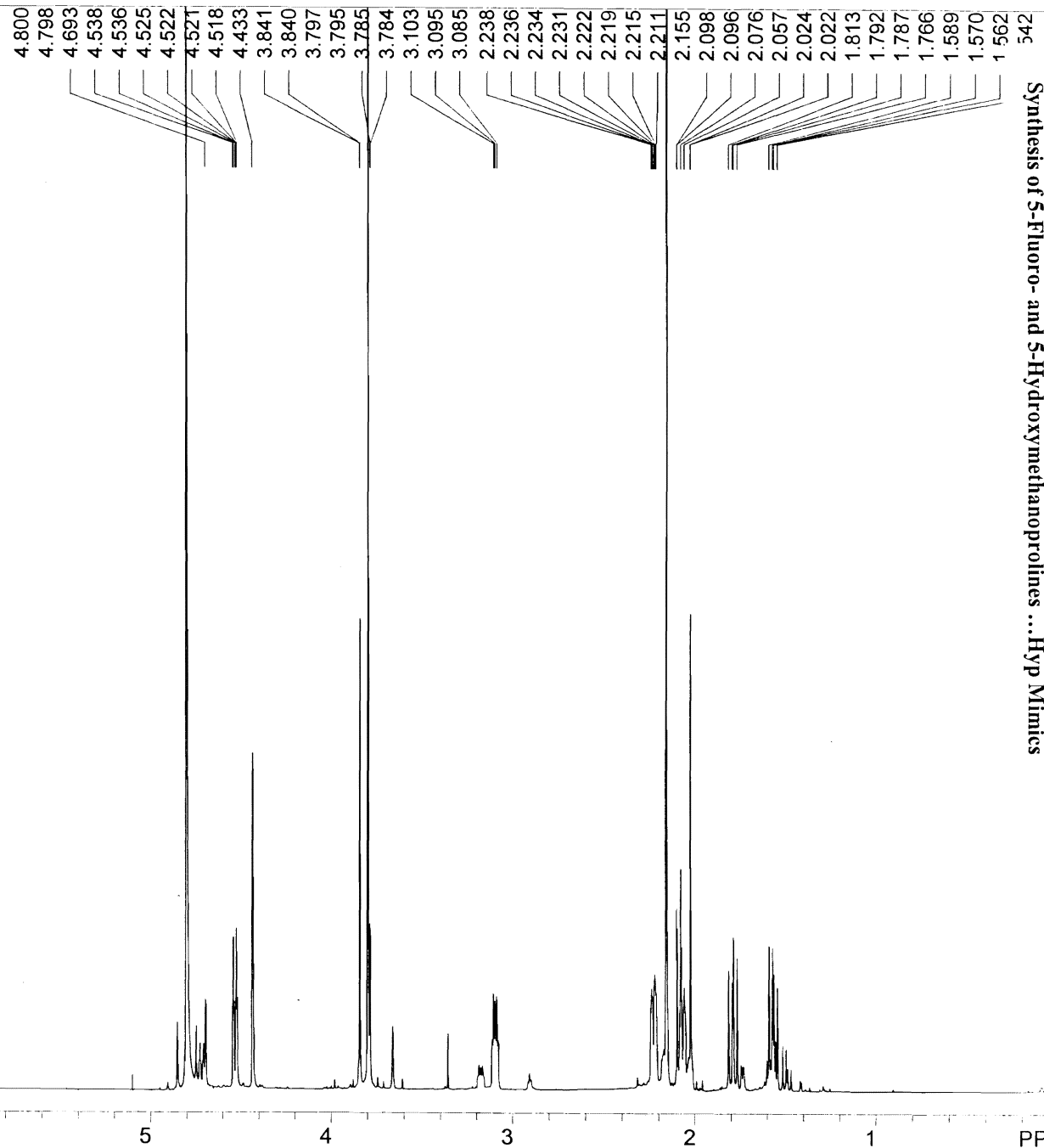
EX:

PW: 5.7 us

PD: 2.0 sec

NA: 4048

LB: 3.0 Nuts - Endo -3-CO2Me NAc[2.1.1] anti-OH--C--eram823

**10**400 MHz D₂O

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ...Hyp Mimics

3-CO₂Me NAc[2.1.1], proton.atm,

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2040.3

PTS1d: 32768

EX:

PW: 9.8 usec

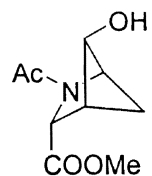
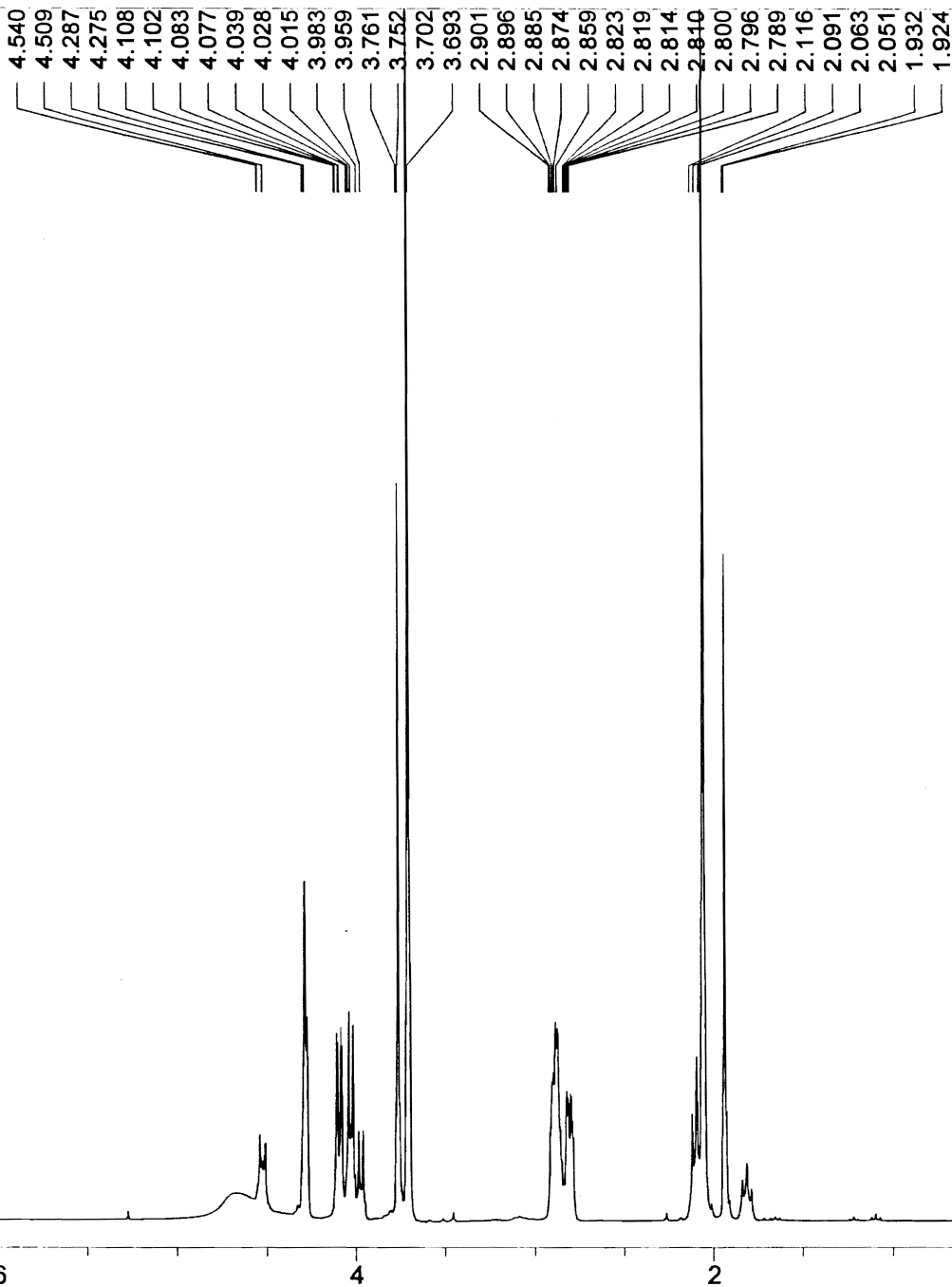
PD: 1.0 sec

NA: 16

USER: temple -- DATE: Feb 3 13:24:05 2008 EST (UT-5h) templ

LB: 0.1WinNuts - 3-CO₂Me NAc[2.1.1] proton for noe D₂O--(855)

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ... Hyp Mimics

**12**400 MHz CDCl₃

eram/13-3-20_12Dec2007

USER: -- DATE: Dec 12 2007

F1: 300.018

F2: 300.018

SW1: 4800

OF1: 1926.9

PTS1d: 16384

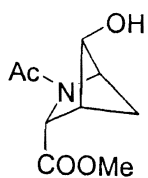
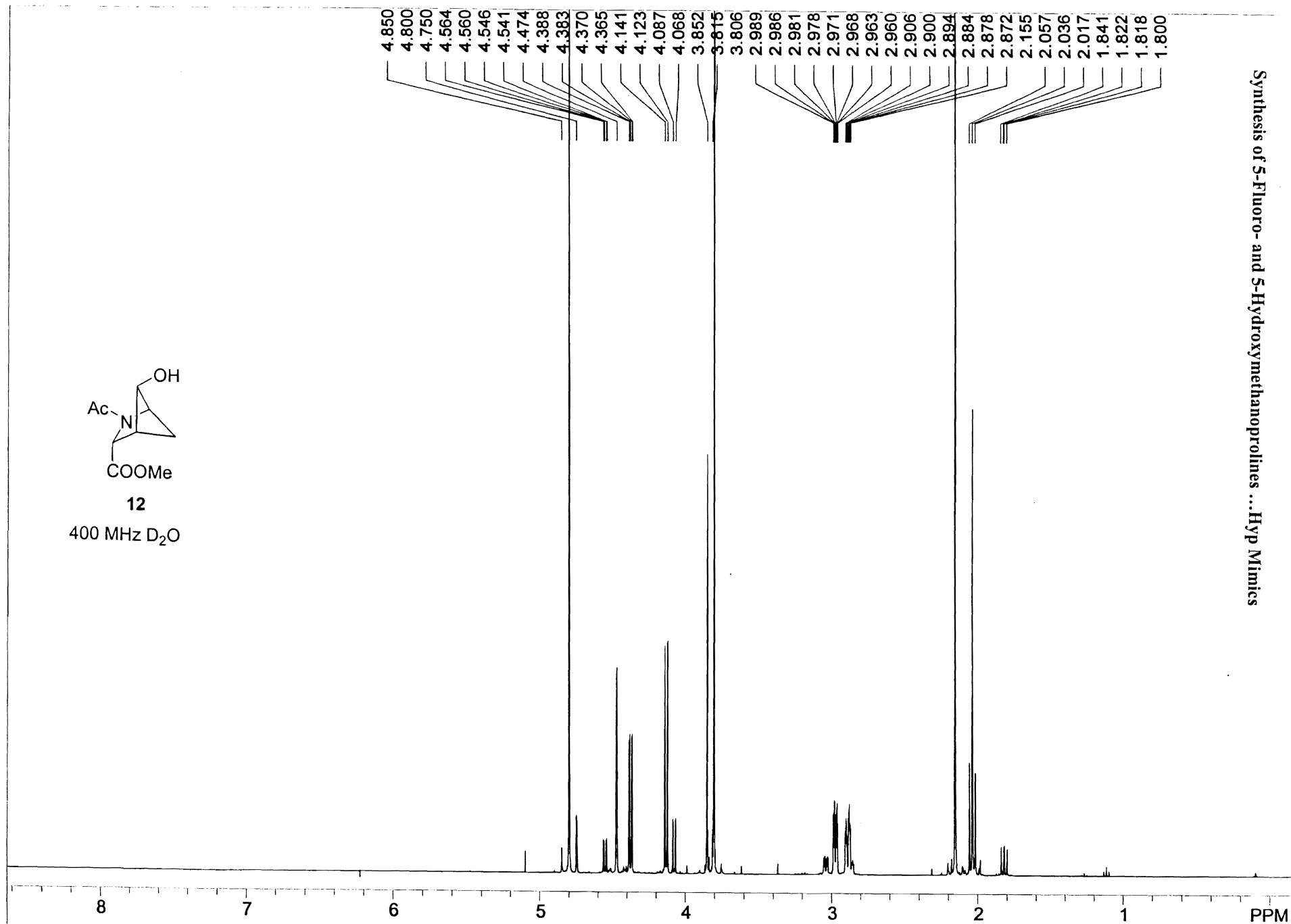
EX: s2pul

PW: 4.0 us

PD: 1.0 sec

NA: 8

Nuts-L-Exo-3-CO2Me NAc[2.1.1] anti-OH--CDCl3--H--eram13-3-20

**12**400 MHz D₂O3-CO₂Me NAc[2.1.1]OH (Major), proton.atm,

USER: temple -- DATE: Jan 25 20:55:55 2008 EST (UT-5h) temp

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2040.7

PTS1d: 32768

EX:

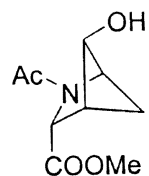
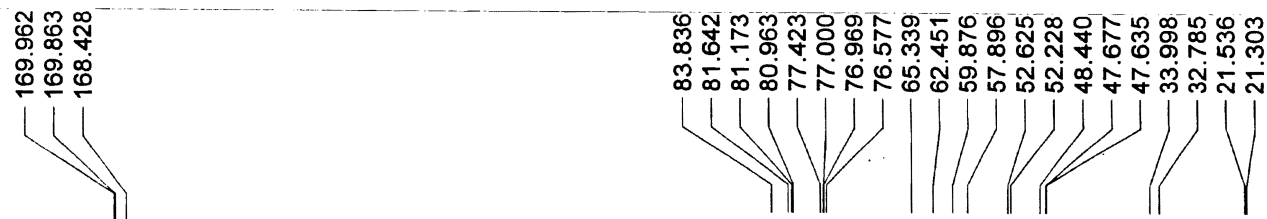
PW: 9.8 us

PD: 1.0 sec

NA: 16

Name: 1-Exo-3-CO₂Me NAc[2.1.1] anti-OH--D₂O--H--eram820

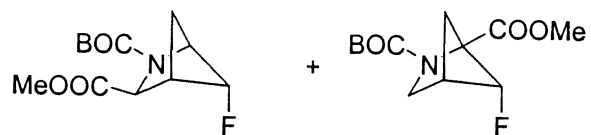
Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ...Hyp Mimics

**12**100 MHz CDCl₃

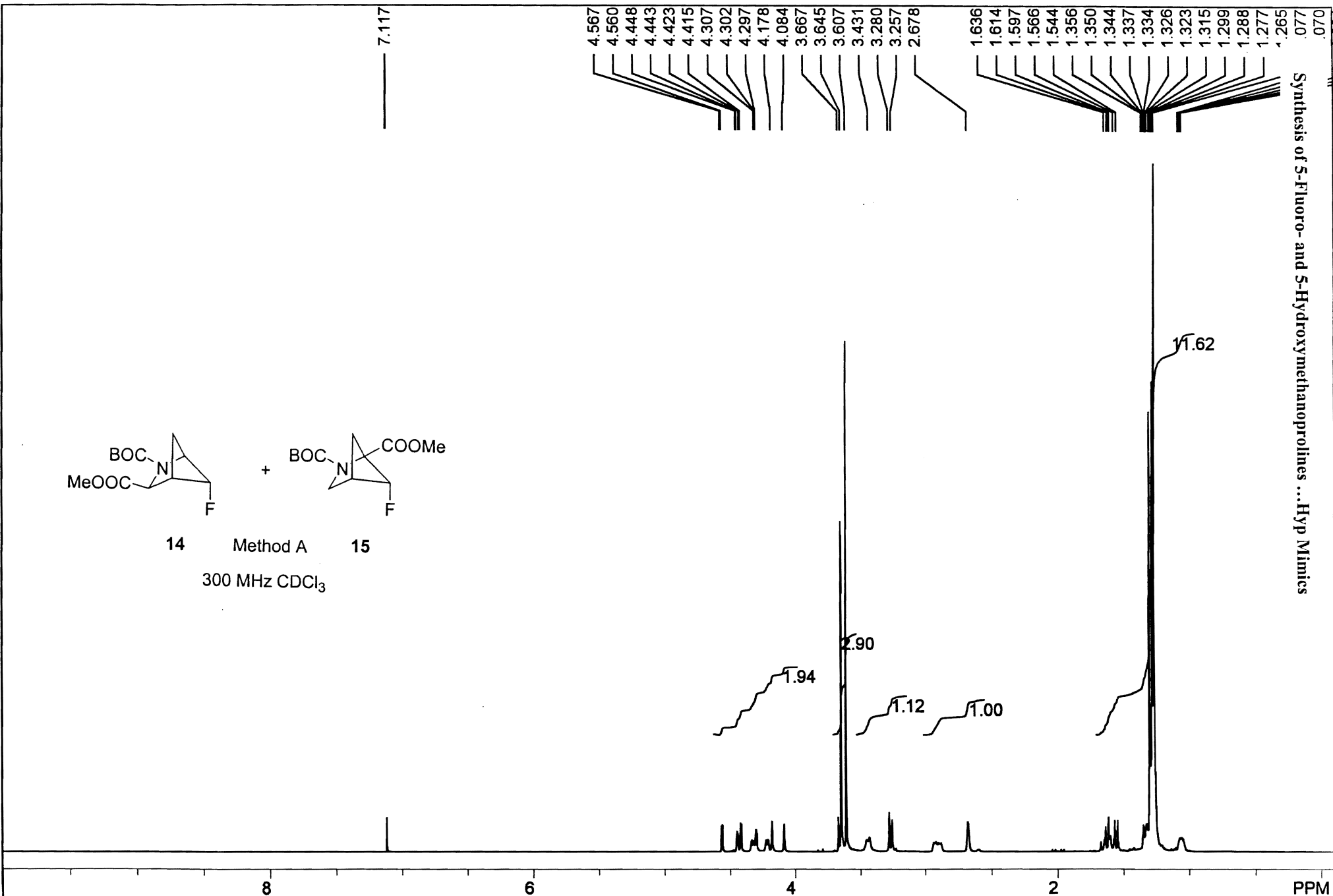
eram/13-3-100_25Dec2007

USER: -- DATE: Dec 25 2007

F1: 75.447	F2: 300.018	SW1: 18859		OF1: 8324.4		PTS1d: 65536
EX: s2pul	PW: 5.0 us	PD: 1.0 sec	NA: 256	Nuts	16.000	CO2Me
						NAc[2.1.1] anti-OH--CDCl3--C--eram13-3-100



Method A

300 MHz CDCl₃ClCO₂Me -78C Spot C. proton10MIN.atm.

USER: temple -- DATE: Apr 8 12:16:04 2005 EDT (UT-4h) templ

F1: 400.020

F2: 1.000

SW1: 8278

OF1: 1943.2

PTS1d: 32768

EX:

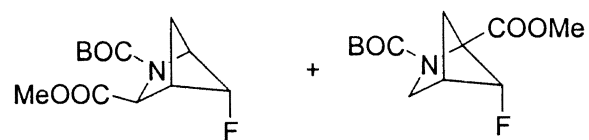
PW: 9.8 usec

PD: 1.0 sec

NA: 128

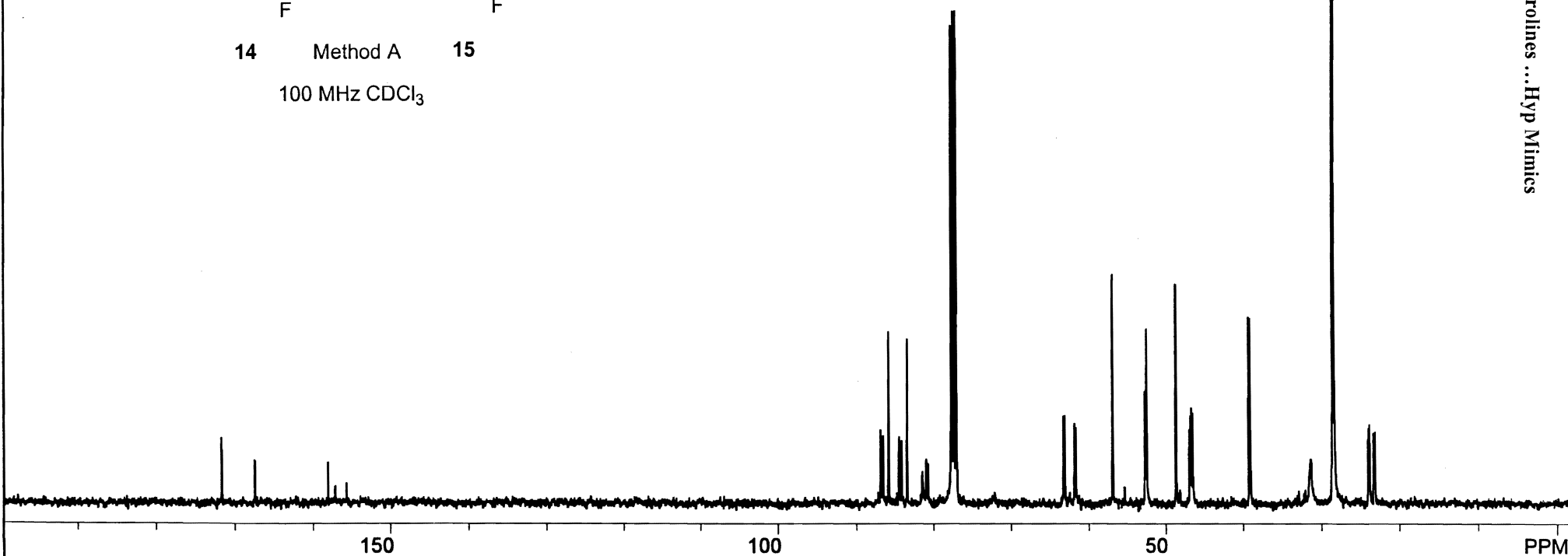
LB: 0.1

WinNuts - \$FangYU.1

100 MHz CDCl₃

158.106
155.716
171.681
167.443

86.818
86.484
85.793
84.411
84.077
83.378
81.398
80.896
80.655
77.703
77.389
77.072
63.200
63.019
61.793
61.624
56.923
56.875
52.681
52.521
52.453
48.680
48.642
46.970
46.790
46.690
46.513
39.283
39.089
31.428
28.602
28.459
24.056
23.888
23.385
23.227



ClCO2Me -78C Spot C, carbon2HR.atm.

USER: temple -- DATE: Apr 8 14:15:51 2005 EDT (UT-4h) templ

F1: 100.585

F2: 1.000

SW1: 25126

OF1: 10057.2

PTS1d: 32768

EX:

PW: 5.7 usec

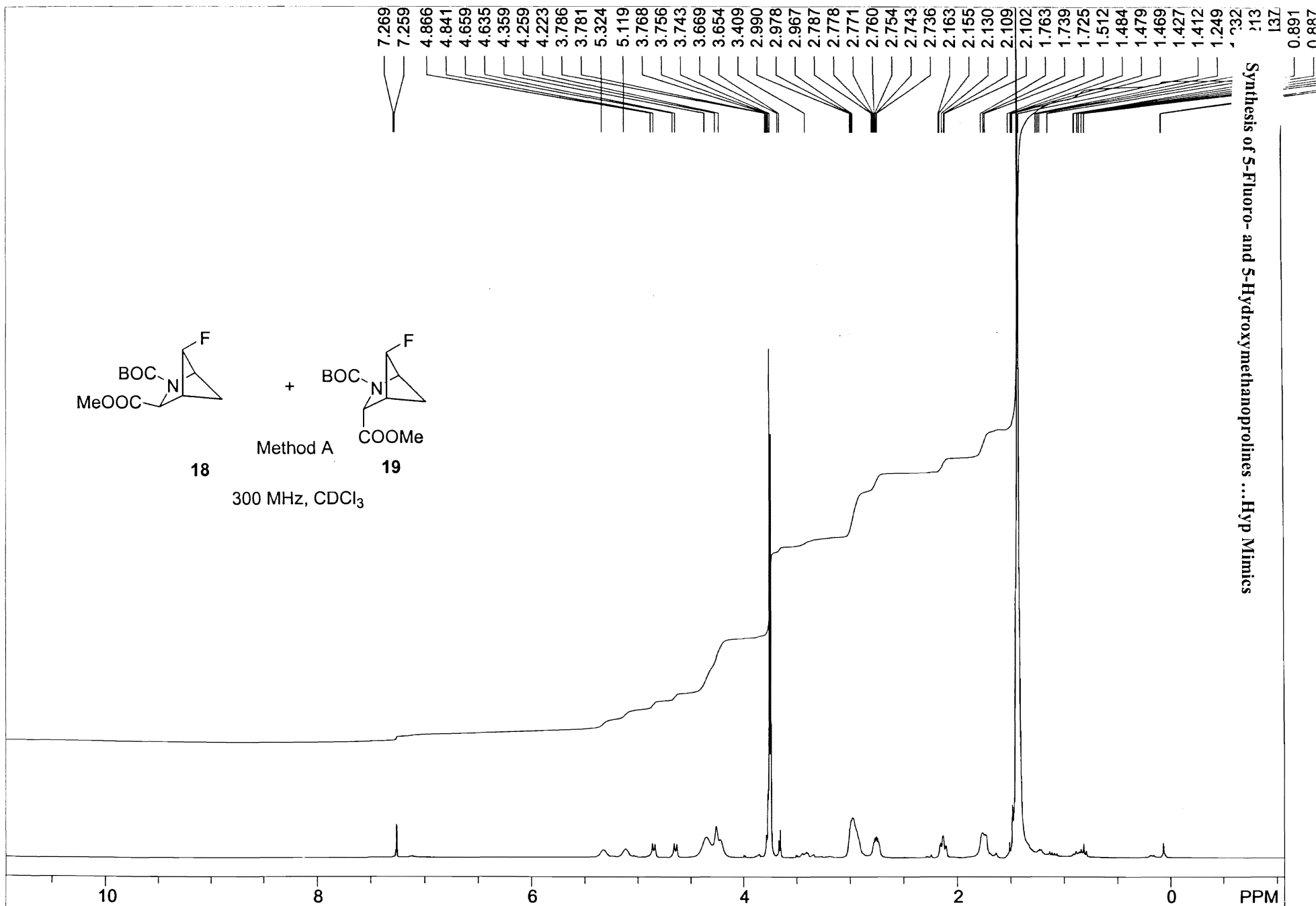
PD: 2.0 sec

NA: 2048

LB: 3.0

WinNuts - \$FangYU.1

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ...Hyp Mimics



deepa/10-160_23May2005

USER: -- DATE: May 23 2005

F1: 299.986

F2: 299.986

SW1: 3600

OF1: 1479.5

PTS1d: 16384

EX: s2pul

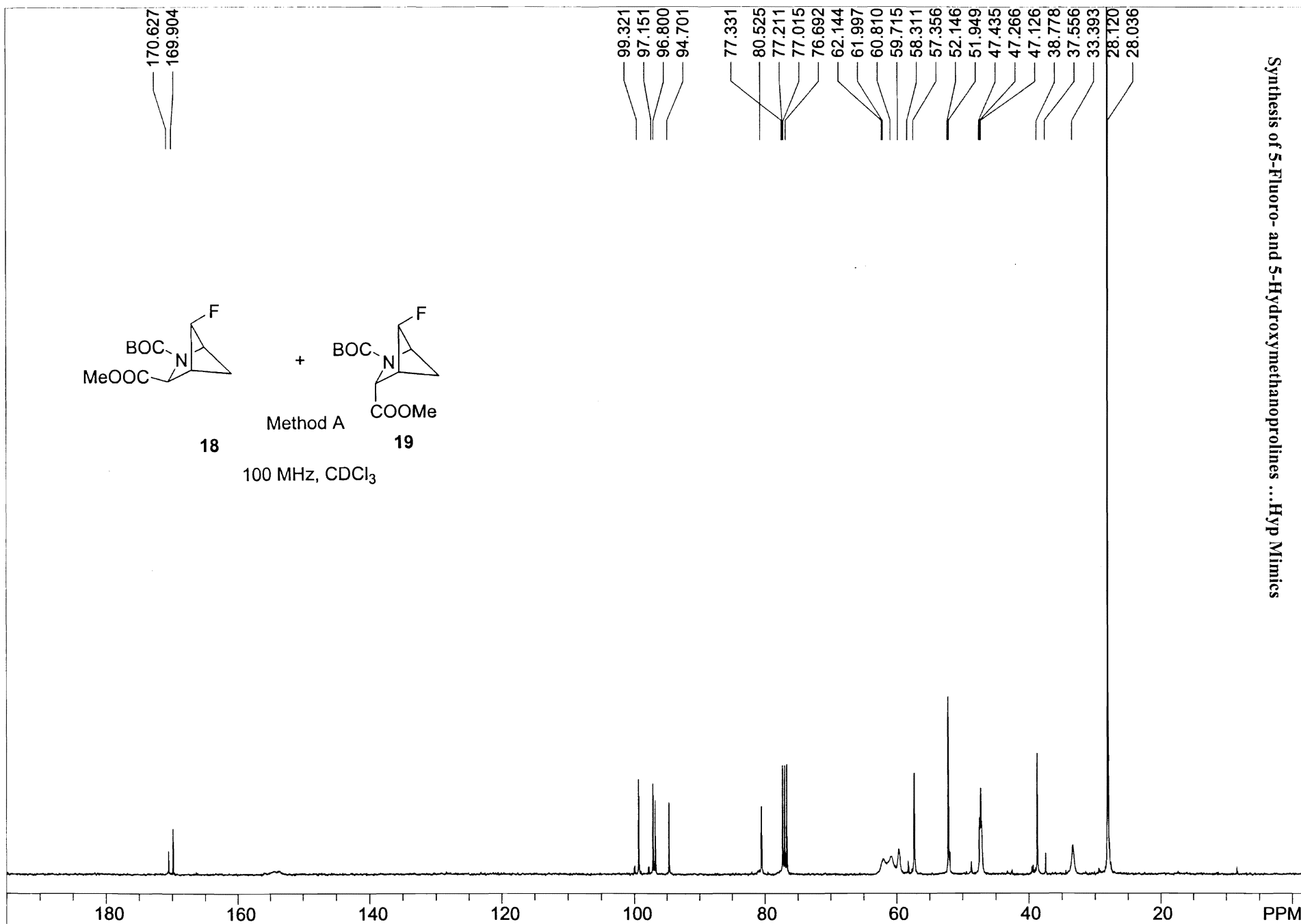
PW: 5.8 us

PD: 1.0 sec

NA: 8

LB: 0.2

Nuts - print 2



BOCFesters 1, carbon2HR.atm,

USER: temple -- DATE: Aug 8 16:24:06 2007 EDT (UT-4h) templ

F1: 100.628

F2: 1.000

SW1: 23148

OF1: 10578.0

PTS1d: 32768

EX: s2pul

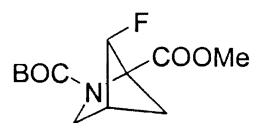
PW: 5.7 us

PD: 2.0 sec

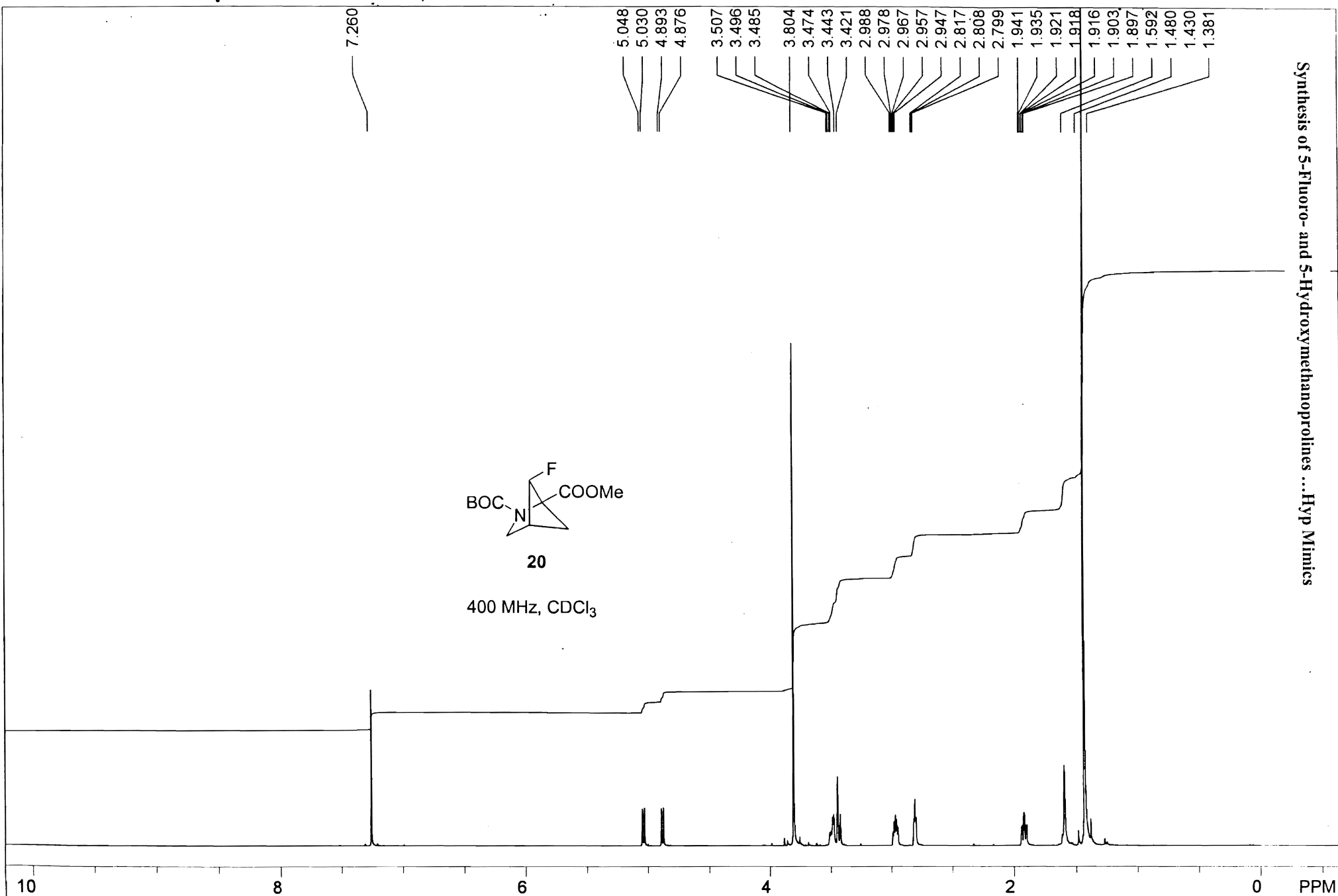
NA: 1024

LB: 3.0

Nuts - print



20

400 MHz, CDCl₃

NBOc[211]F-1-CO2Me, proton, atm,

USER: temple -- DATE: Nov 15 17:34:50 2006 EST (UT-5h) templ

F1: 400.191

F2: 1.000

SW1: 8278

OF1: 2001.1

PTS1d: 32768

EX:

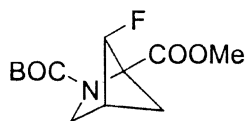
PW: 9.8 usec

PD: 1.0 sec

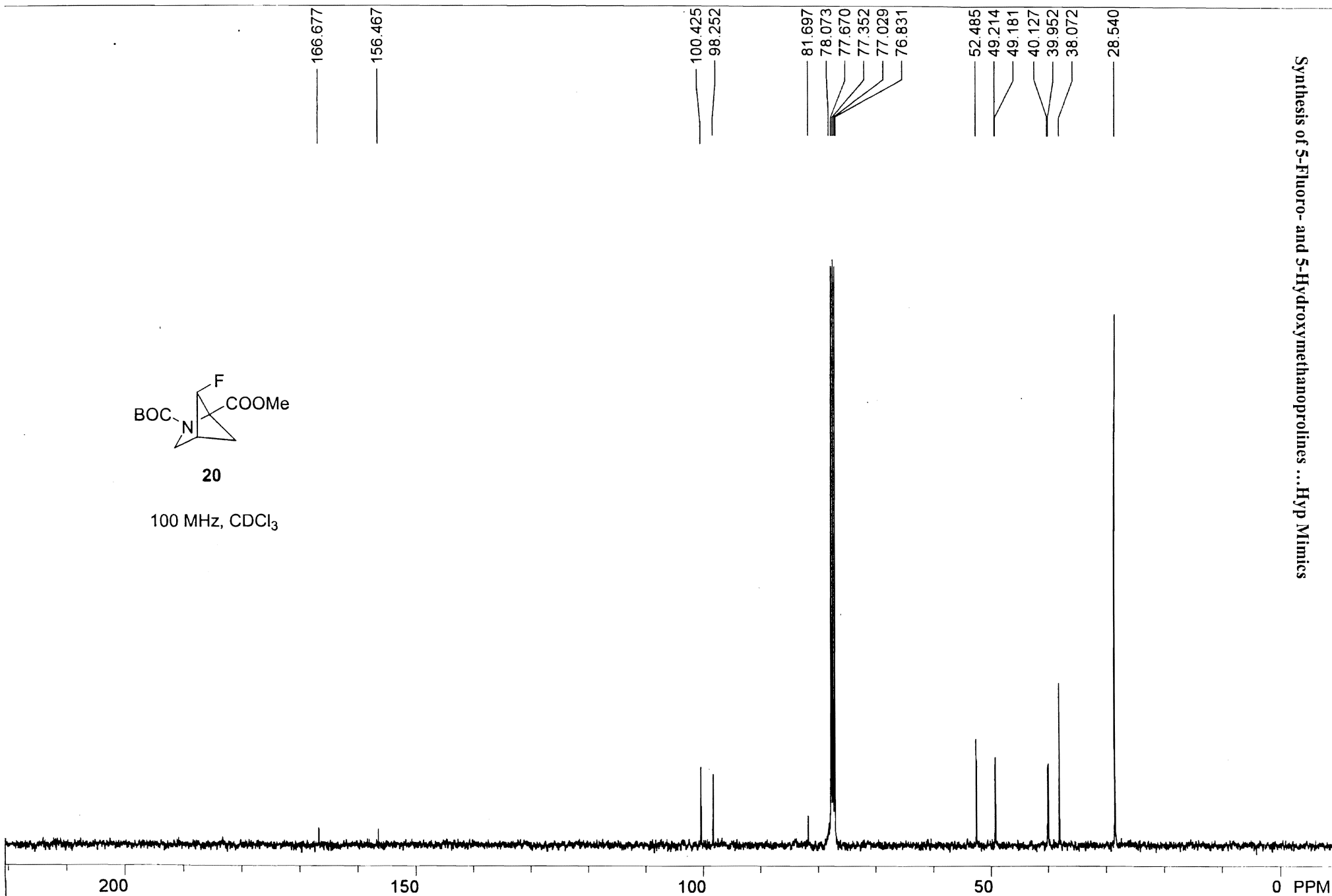
NA: 16

LB: 0.1

WinNuts - NBOc[211]-1-CO2Me-5-F



20

100 MHz, CDCl₃

carbon2HR.atm.

USER: temple -- DATE: Nov 17 18:48:28 2006 EST (UT-5h) templ

F1: 100.628

F2: 1.000

SW1: 23148

OF1: 10621.8

PTS1d: 32768

EX: s2pu]

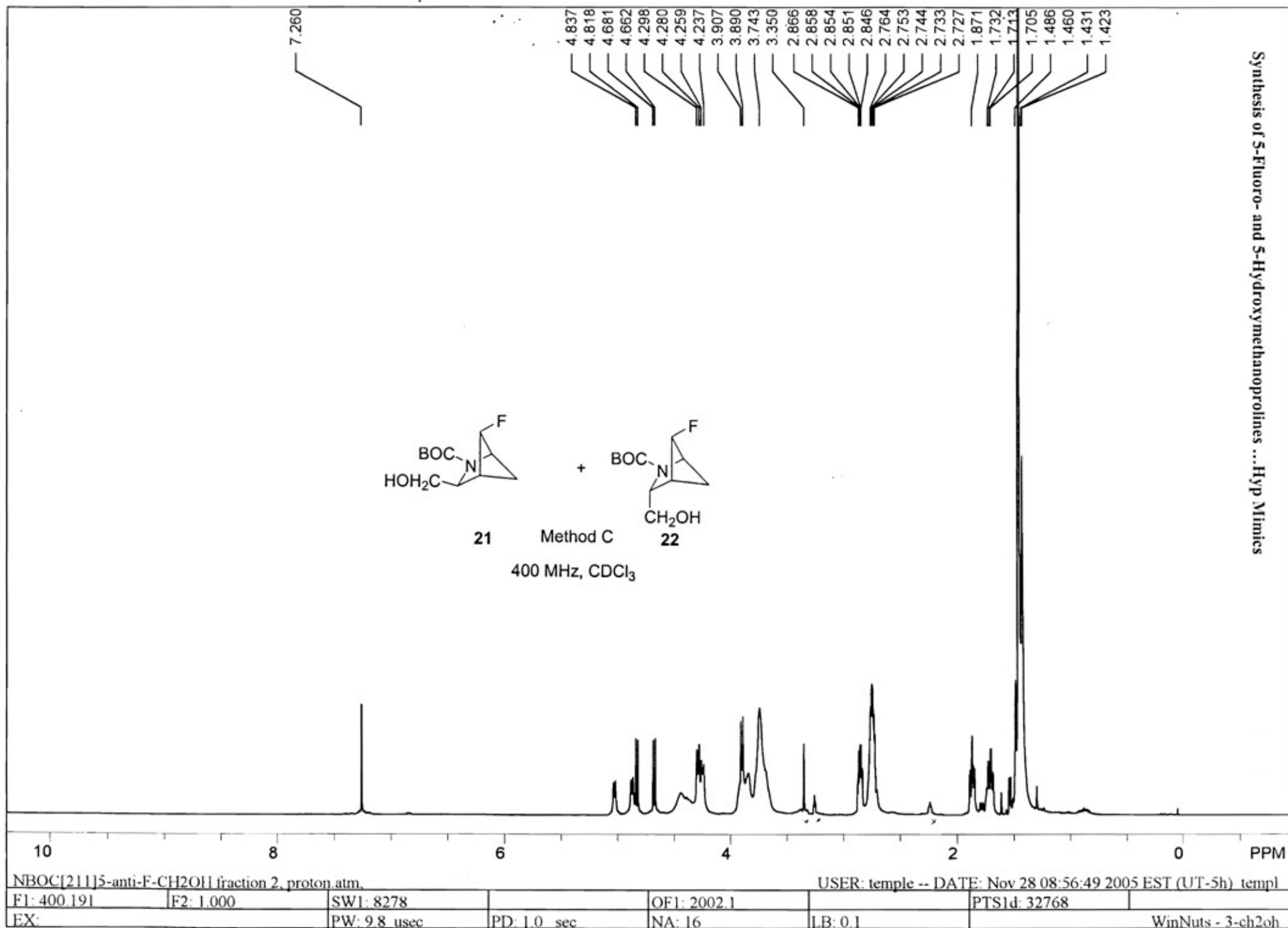
PW: 5.7 usec

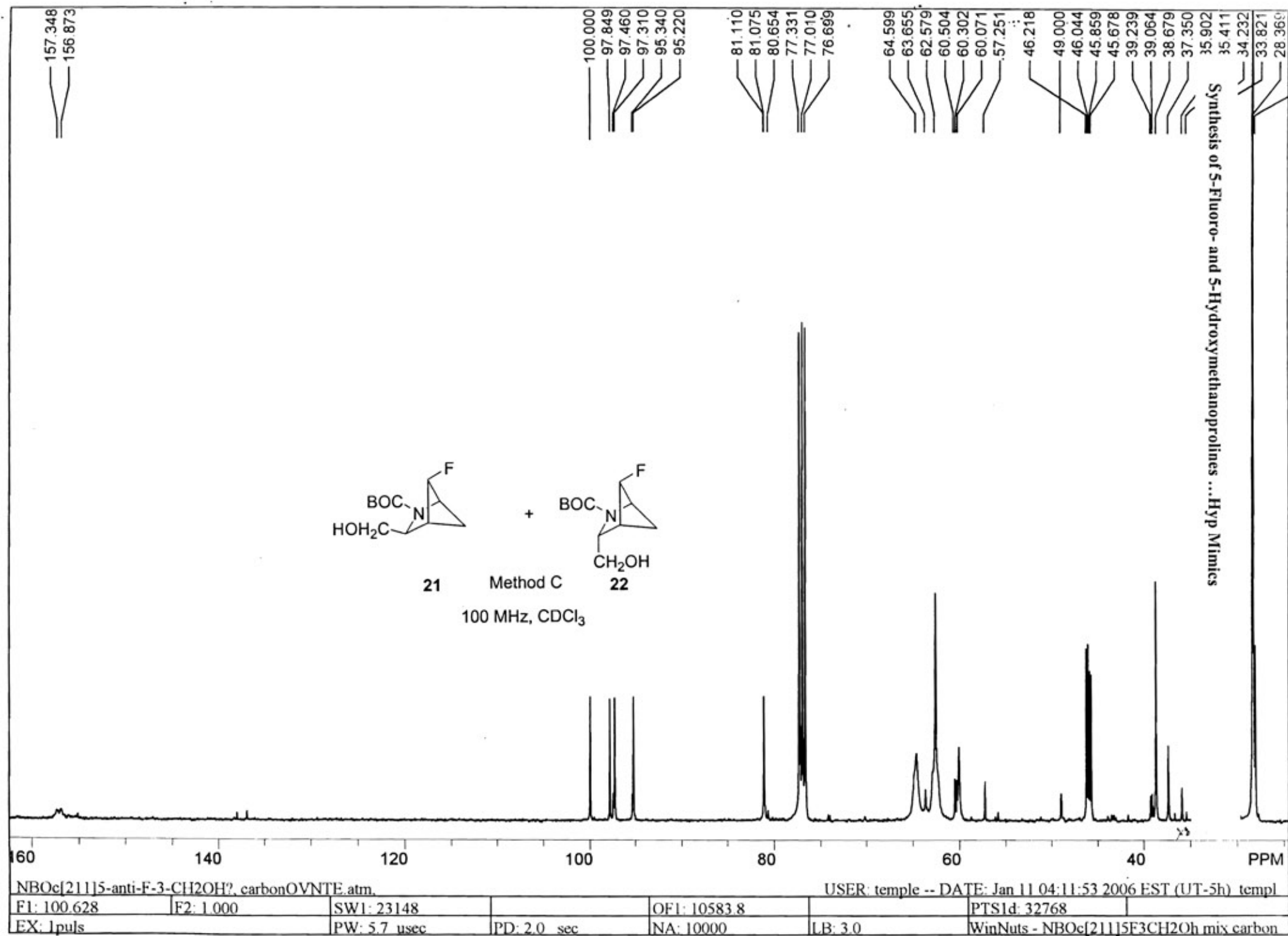
PD: 2.0 sec

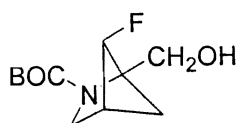
NA: 2048

LB: 3.0

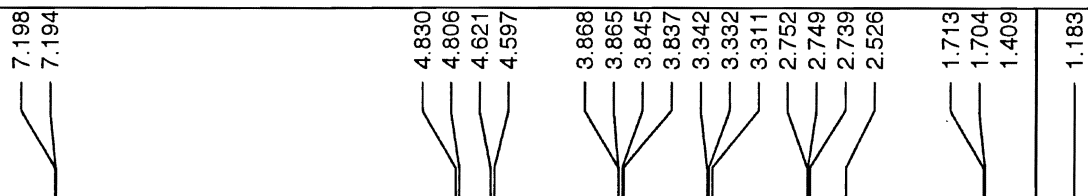
WinNuts - NBOc[211]-1-CO2Me-5-F carbon pure





**23**

Method C
300 MHz, CDCl₃



10

8

6

4

2

0

PPM

deepa/20-200 31Mar2006

USER: -- DATE: Mar 31 2006

F1: 300.018

F2: 300.018

SW1: 3600

OF1: 1467.4

PTS1d: 16384

EX: s2pul

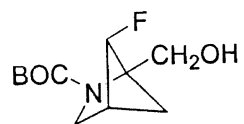
PW: 4.0 usec

PD: 1.0 sec

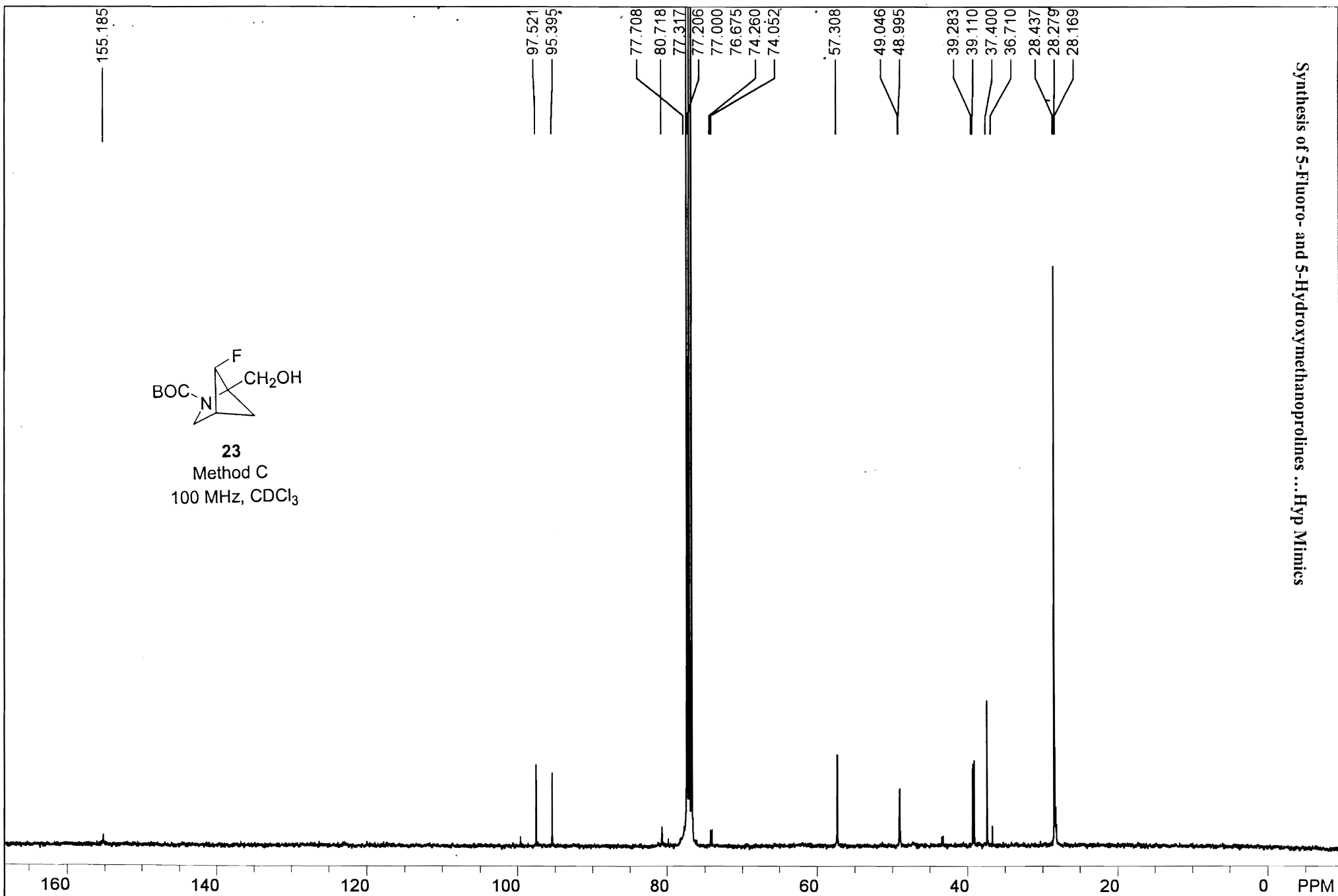
NA: 8

LB: 0.0

WinNuts - NBoc[2[1].1.1]5-anti-f-1-CH2OH.txt

**23**

Method C

100 MHz, CDCl₃N-BOC-[2.1.1]-5-antif-1-CH₂OH, carbonOVNTE.atm,

USER: temple -- DATE: Jul 13 06:04:23 2004 EDT (UT-4h) templ

F1: 100.585

F2: 1.000

SW1: 25126

OF1: 10020.7

PTS1d: 32768

EX:

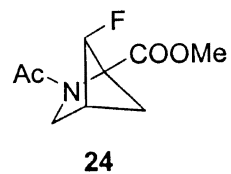
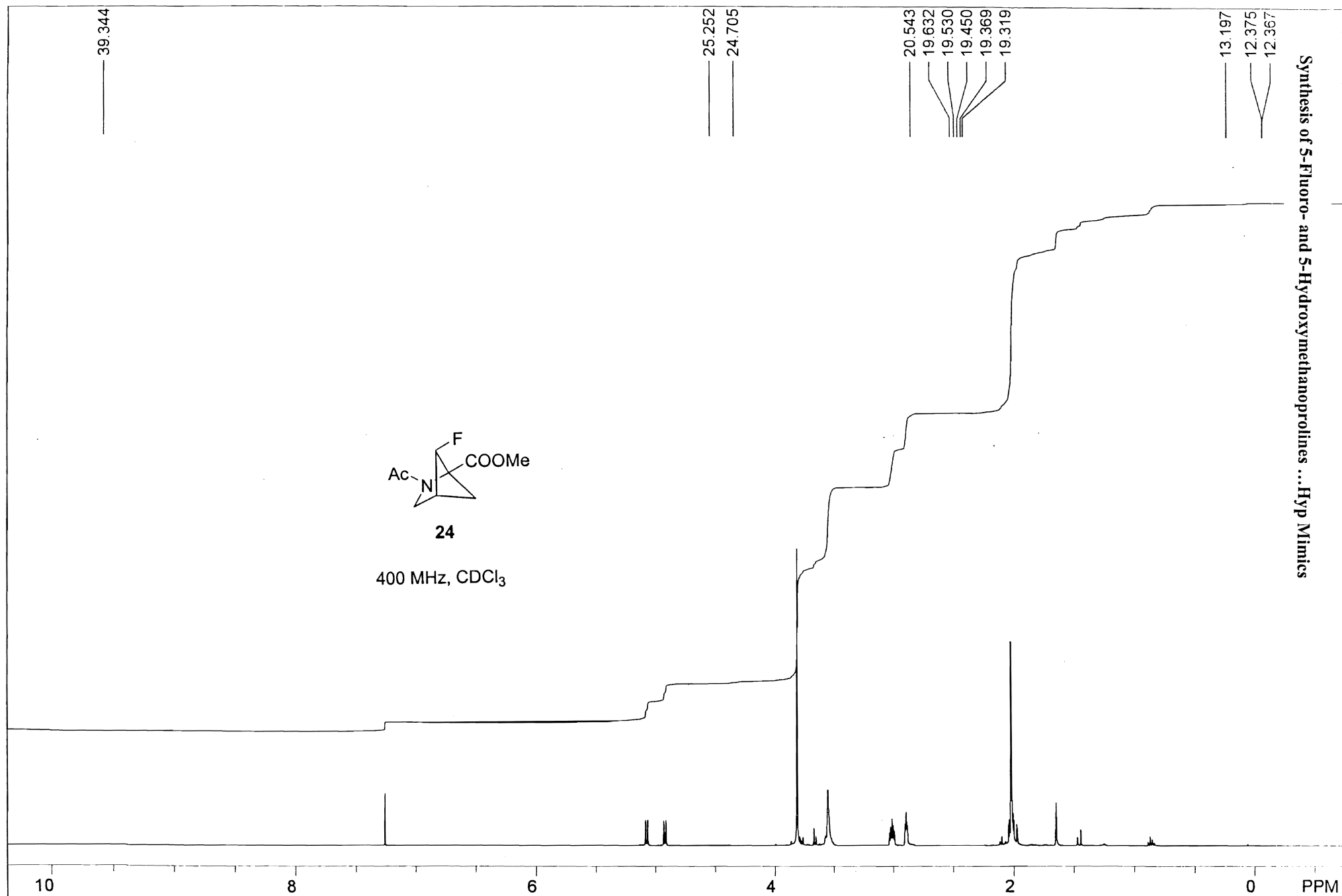
PW: 5.7 usec

PD: 2.0 sec

NA: 12000

LB: 2.0

WinNuts - \$WS_FTP.1

400 MHz, CDCl₃

NAc[211]-1-CO2Me-5-f, proton.atm.

USER: temple -- DATE: Nov 17 09:00:59 2006 EST (UT-5h) templ

F1: 400.191

F2: 1.000

SW1: 8278

OF1: 2001.3

PTSId: 32768

EX: s2pul

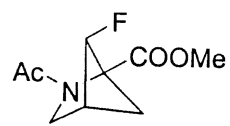
PW: 9.8 usec

PD: 1.0 sec

NA: 16

LB: 0.1

WinNuts - NAc[211]-5-anti-F-1-CO2Me b2

**24**100 MHz, CDCl₃169.955
166.16599.339
97.17577.323
77.000
76.68752.417
48.76439.623
39.451
37.995

20.951

200

150

100

50

0 PPM

NAC[211]-1-CO2Me-5-F, carbon2HR.atm.

USER: temple -- DATE: Nov 21 15:08:13 2006 EST (UT-5h) templ

F1: 100.628

F2: 1.000

SW1: 23148

OF1: 10582.9

PTS1d: 32768

EX:

PW: 5.7 usec

PD: 2.0 sec

NA: 2048

LB: 3.0

WinNuts - \$WS_FTP.1

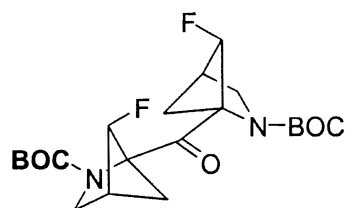
deepa/1-1032_07May2004

Archive directory: /home/temple/vnmrsys/data
Sample directory: deepa/1-1032_07May2004
File: PROTON

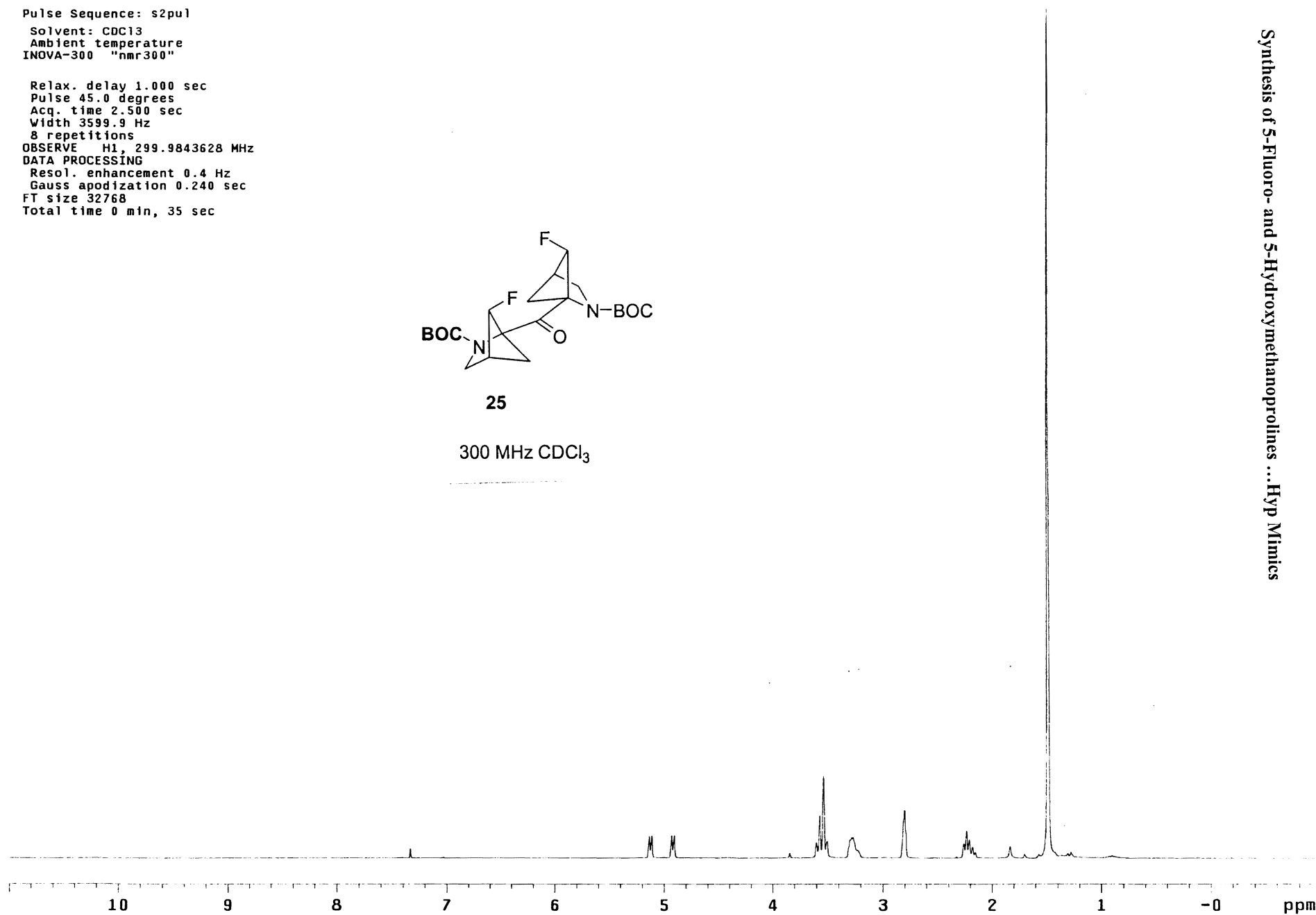
Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
INOVA-300 "nmr300"

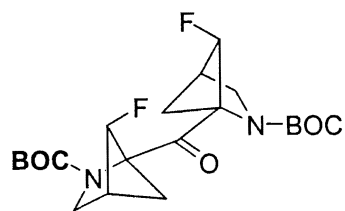
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 3599.9 Hz
8 repetitions
OBSERVE H1, 299.9843628 MHz
DATA PROCESSING
Resol. enhancement 0.4 Hz
Gauss apodization 0.240 sec
FT size 32768
Total time 0 min, 35 sec



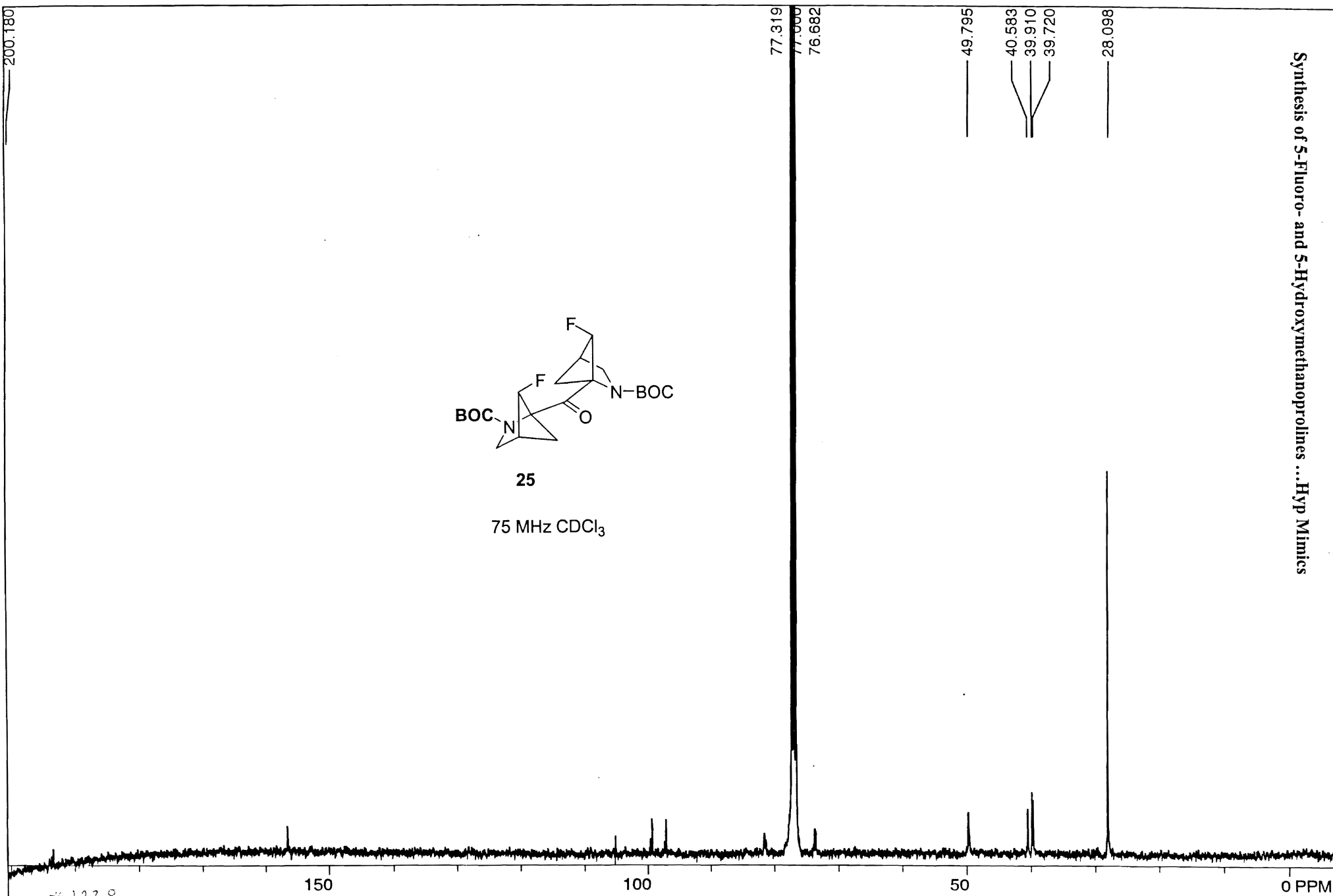
25

300 MHz CDCl₃

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ... Hyp Mimics



25

75 MHz CDCl₃

carbonOVNTE.atm.

USER: temple -- DATE: Nov 23 22:08:31 2006 EST (UT-5h) templ

F1: 100.628

F2: 1.000

SW1: 23148

OF1: 10584.8

PTS1d: 32768

EX:

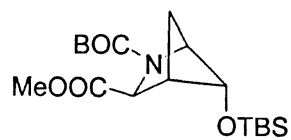
PW: 5.7 usec

PD: 5.0 sec

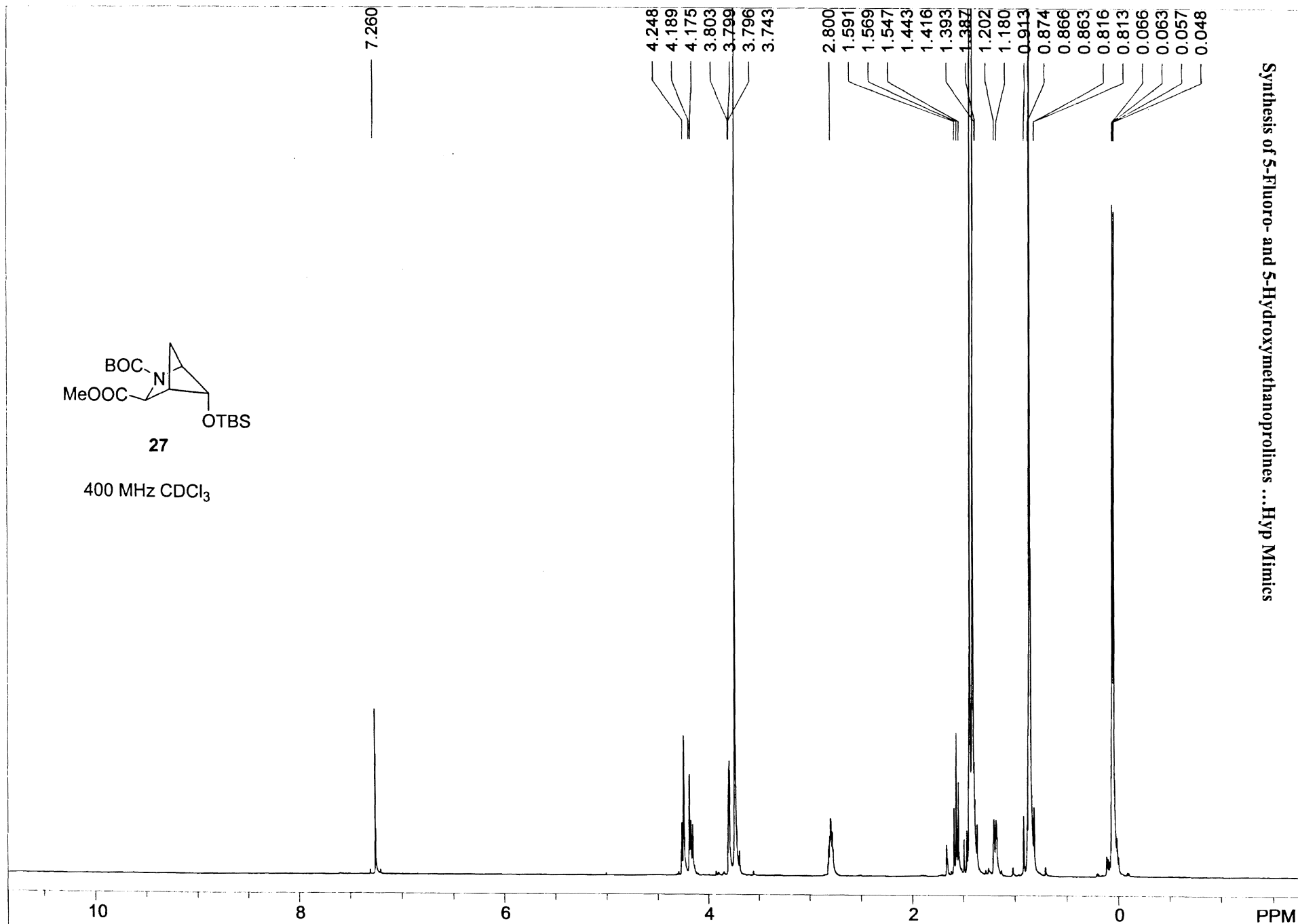
NA: 5000

LB: 3.0

WinNuts - \$WS FTP.1



27

400 MHz CDCl₃3-CO₂Me NBoc[2.1.1] syn-OTBS, proton.atm,

USER: temple -- DATE: Apr 15 10:40:12 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8013

OF1: 2004.1

PTS1d: 32768

EX:

PW: 9.8 us

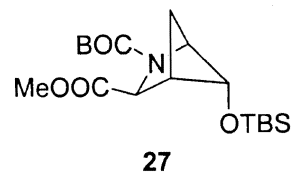
PD: 1.0 sec

NA: 16

LB: 0.Nuts - 3-CO₂Me NBoc[2.1.1] syn-OTBS---H--eram-1050

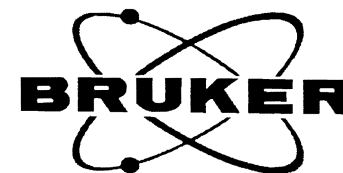
eram 14-26 13C in CDCl₃

172.45
172.37
156.54
155.40



125 MHz CDCl₃

79.65
79.43
77.20
76.69
71.63
71.47
64.23
62.90
57.04
56.73
52.03
51.86
47.84
47.74
28.37
28.27
26.04
25.66
25.58
25.37
17.92
17.83
-5.07
-5.10
-5.22



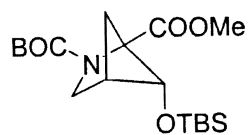
NAME krow
EXPNO 110
PROCNO 1
Date 20080416
Time 11.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 s
RG 645
DW 16.800 us
DE 6.50 us
TE 291.5 K
D1 3.00000000 s
D11 0.03000000 s
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 us
PL1 0.50 dB
PL1W 85.96688843 W
SFO1 125.7703643 MHz

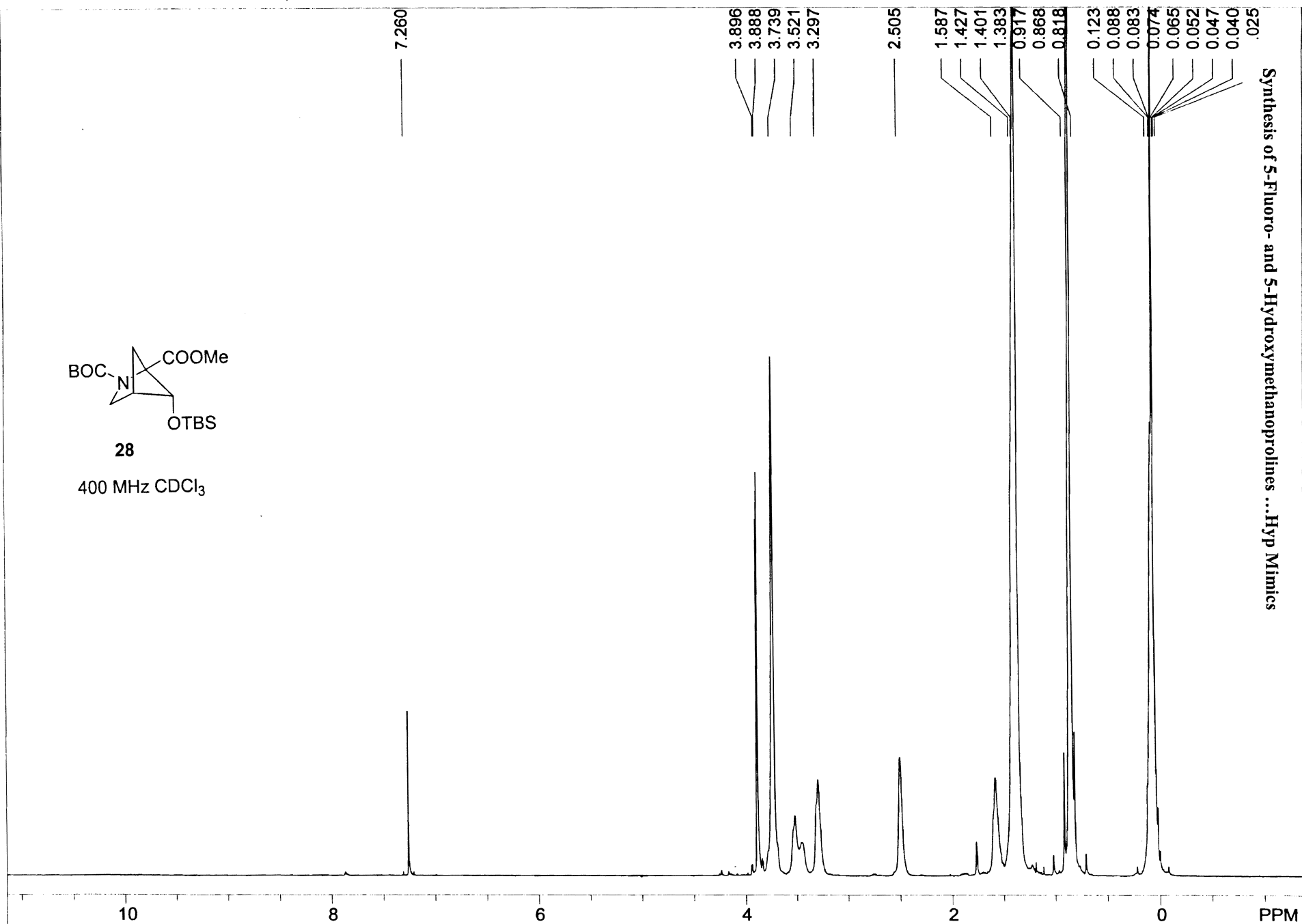
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PL2 2.00 dB
PL12 18.68 dB
PL13 18.68 dB
PL2W 18.54325104 W
PL12W 0.39827755 W
PL13W 0.39827755 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577967 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

Synthesis of 5-Fluoro- and 5-Hydroxymethanoproline ... Hyp Mimics

200 180 160 140 120 100 80 60 40 20 0 ppm



28

400 MHz CDCl₃

Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ... Hyp Mimics

1-CO₂Me NBoc[2.1.1] syn-OTBS, proton.atm,

USER: temple -- DATE: Apr 15 11:13:14 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8013

OF1: 2003.9

PTS1d: 32768

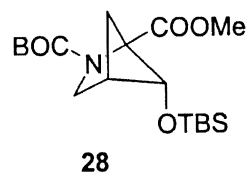
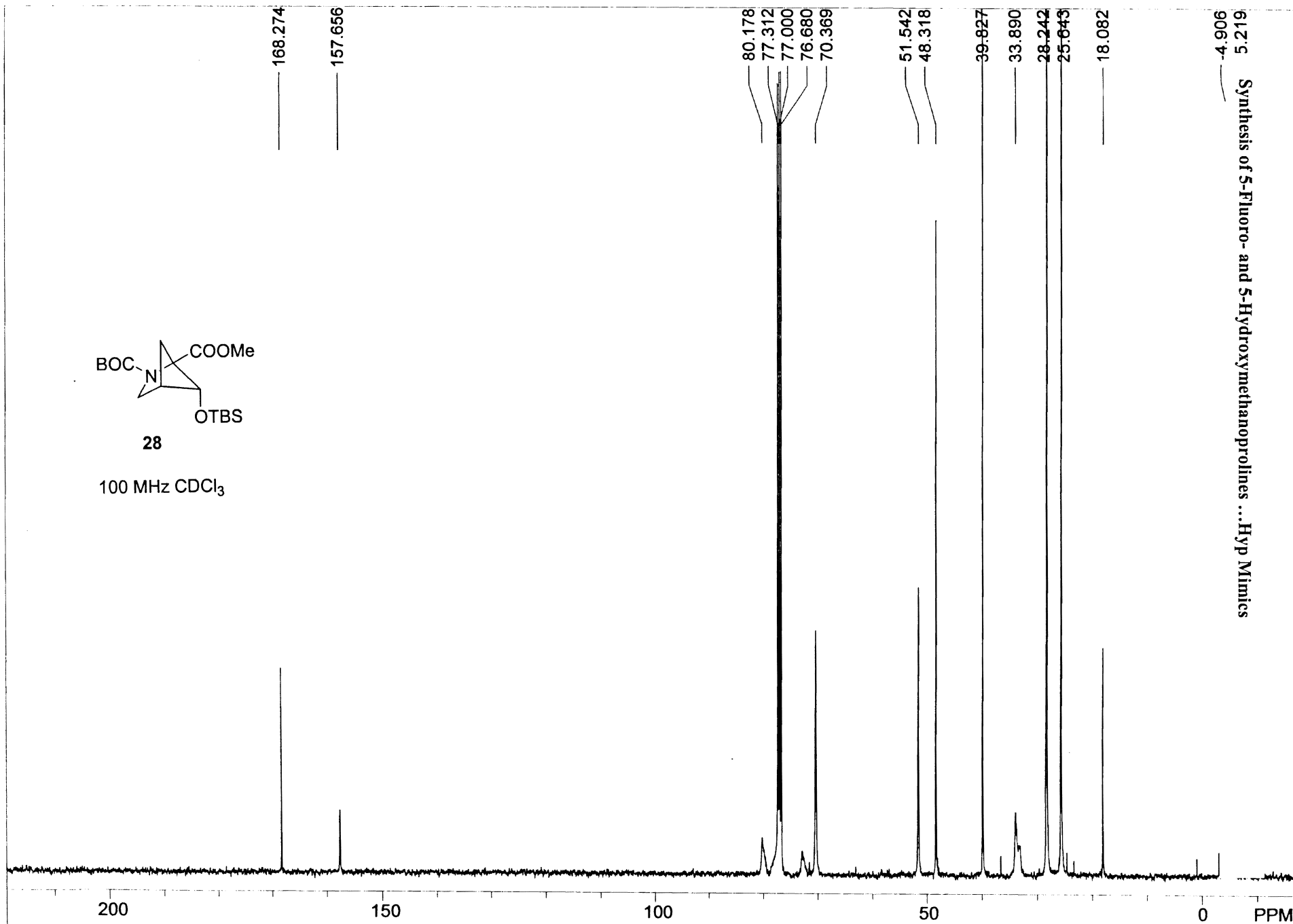
EX:

PW: 9.8 us

PD: 1.0 sec

NA: 16

LB: 0.Nuts - 1-CO₂Me NBoc[2.1.1] syn-OTBS---H--eram-1051

100 MHz CDCl₃

Synthesis of 5-Fluoro- and 5-Hydroxymethanoproline...Hyp Mimics

1-CO2Me NBoc[2.1.1] syn-OTBS, carbonOVNTE.all,

USER: temple -- DATE: Jun 27 05:43:19 2008 EDT (UT-4h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10020.3

PTS1d: 32768

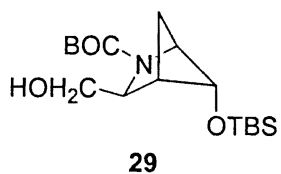
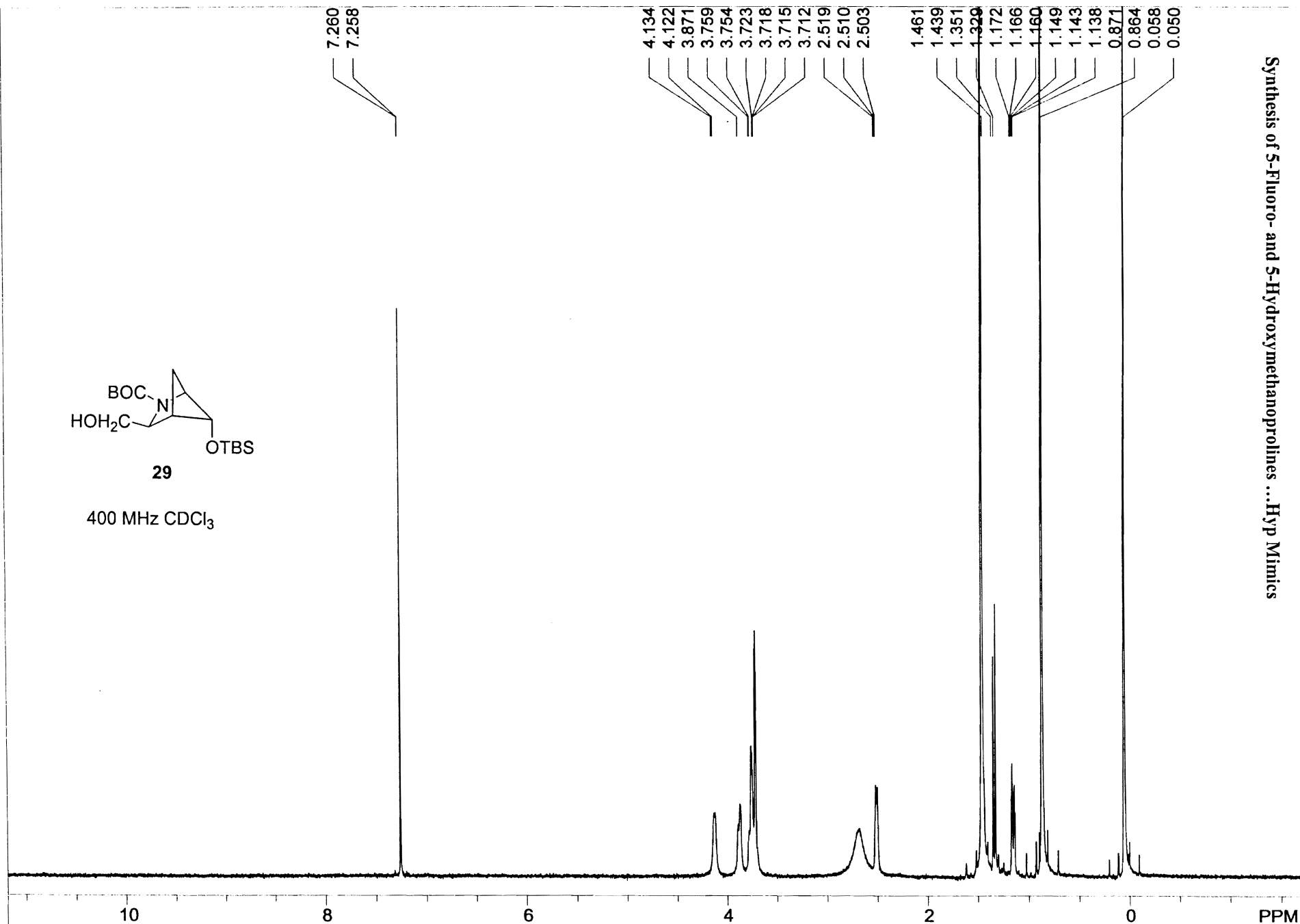
EX:

PW: 5.7 us

PD: 2.0 sec

NA: 7000

LB: 3.0 Nuts - 1-CO2Me NBoc[2.1.1] syn-OTBS---C--eram-1310

400 MHz CDCl₃3-CH₂OH NBoc[2.1.1] syn-OTBS, proton.atm,

USER: temple -- DATE: Jun 6 20:53:12 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8013

OF1: 2003.9

PTS1d: 32768

EX:

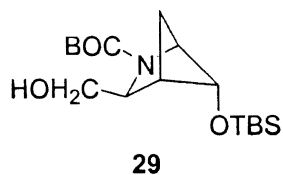
PW: 9.8 us

PD: 1.0 sec

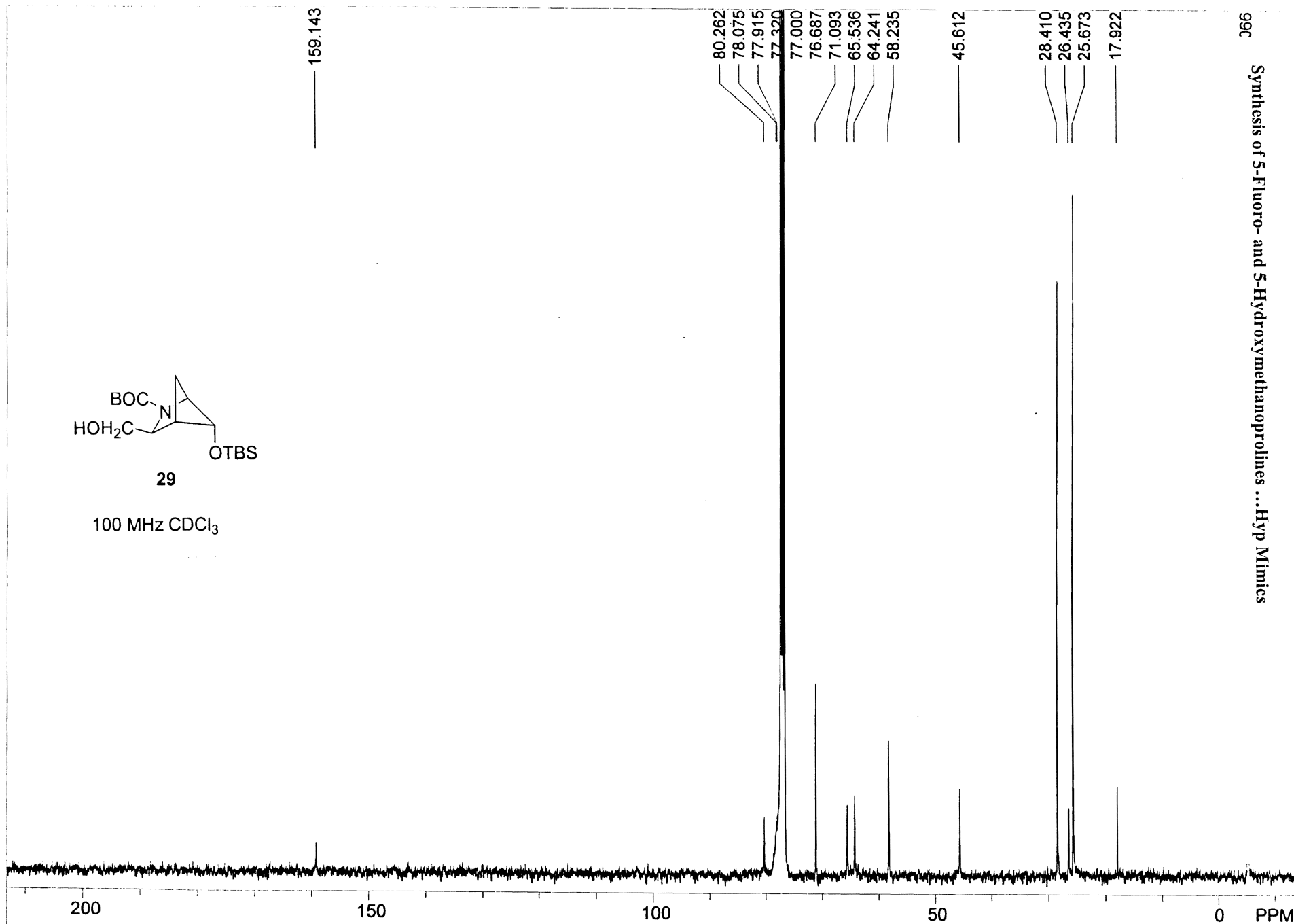
NA: 16

LB: 0.Nuts - 3-CH₂OH NBoc[2.1.1] syn-OTBS--H--eram-1260

96C Synthesis of 5-Fluoro- and 5-Hydroxymethanoprolines ... Hyp Mimics



100 MHz CDCl₃



3-CH₂OH NBoc[2.1.1] syn-OTBS, carbonOVNTE.all,

USER: temple -- DATE: Jun 8 12:37:05 2008 EDT (UT-4h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10021.9

PTS1d: 32768

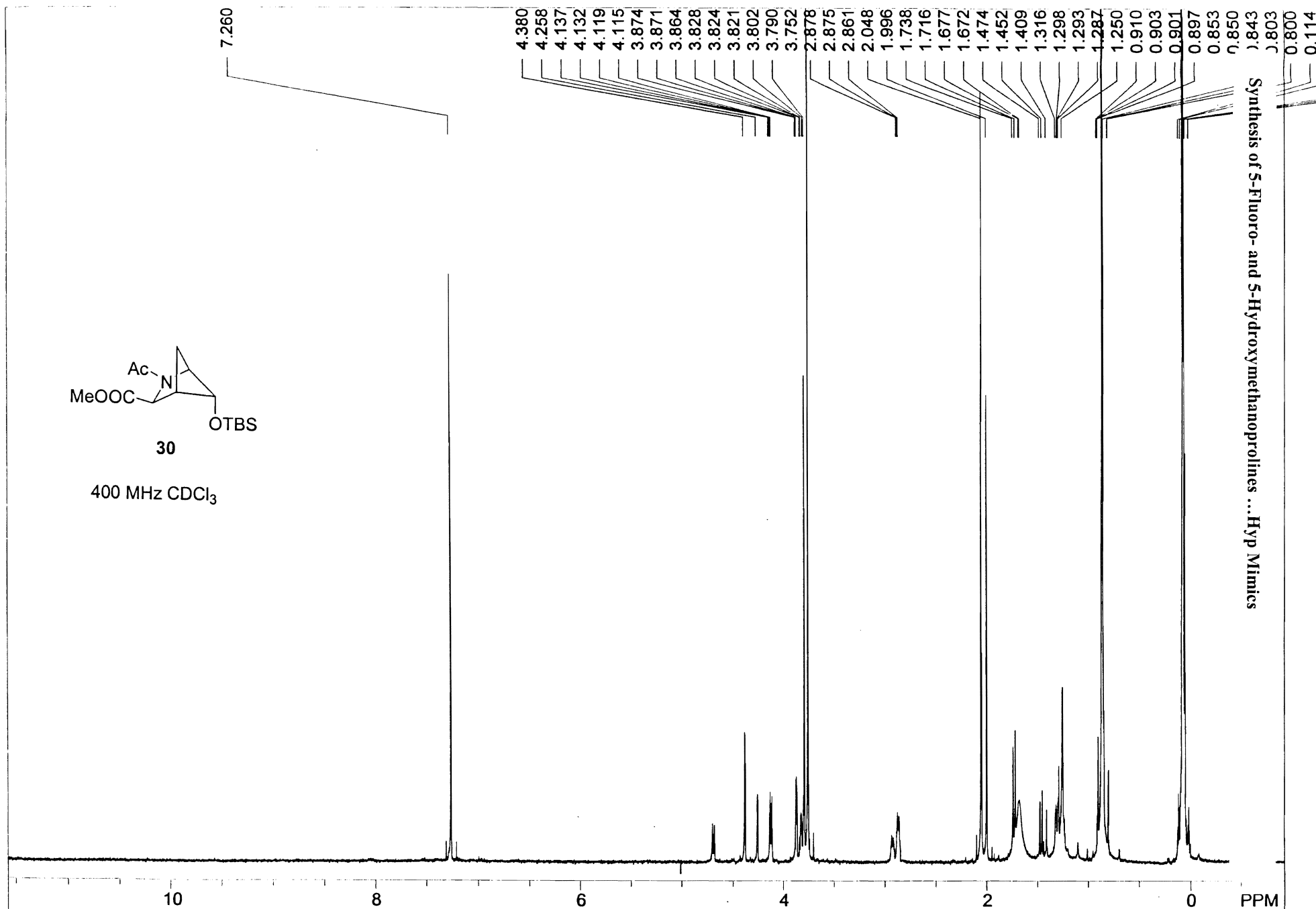
EX:

PW: 5.7 us

PD: 5.0 sec

NA: 9000

LB: 3.Nuts - 3-CH₂OH NBoc[2.1.1] syn-OTBS---C--eram-1265

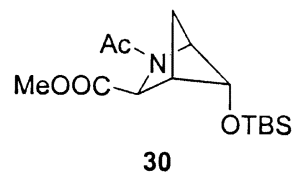
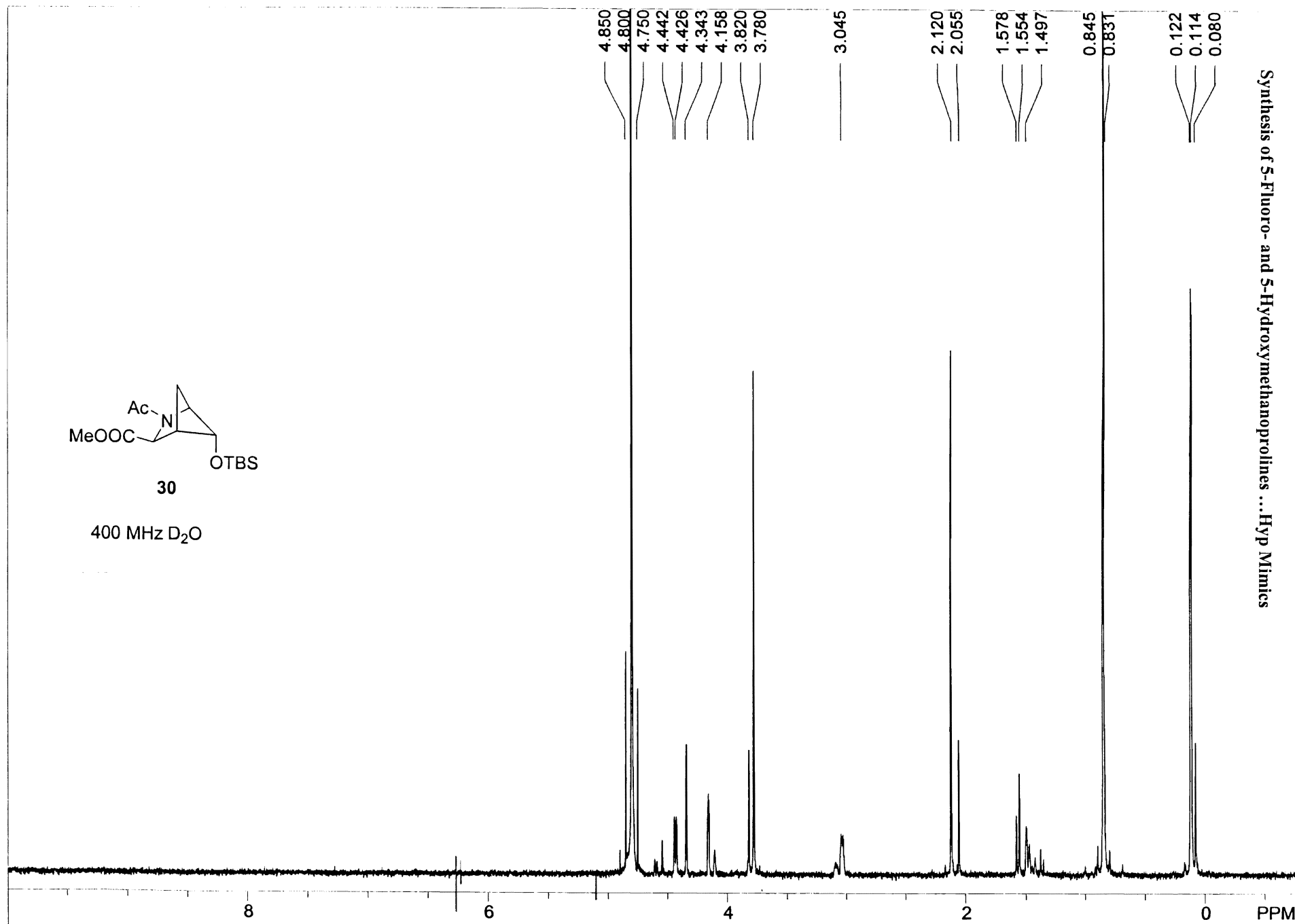


Synthesis of 5-Fluoro- and 5-Hydroxymethanoproline ... Hyp Mimics

3-CO₂Me NAc[2.1.1] syn-OTBS, proton.atm,

USER: temple -- DATE: May 10 20:18:55 2008 EDT (UT-4h) templ

F1: 400.130	F2: 1.000	SW1: 8013	OF1: 2004.7	PTS1d: 32768
EX:	PW: 10.8 us	PD: 1.0 sec	NA: Nuts - 3-CO ₂ Me NAc[2.1.1] syn-OTBS---H (CDCl ₃)--(water present) eram-1149	

400 MHz D₂O3-CO₂Me NAc[2.1.1] syn-OTBS, proton10MIN.atm,

USER: temple -- DATE: May 13 09:37:43 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2041.2

PTS1d: 32768

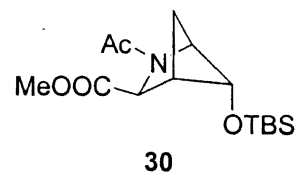
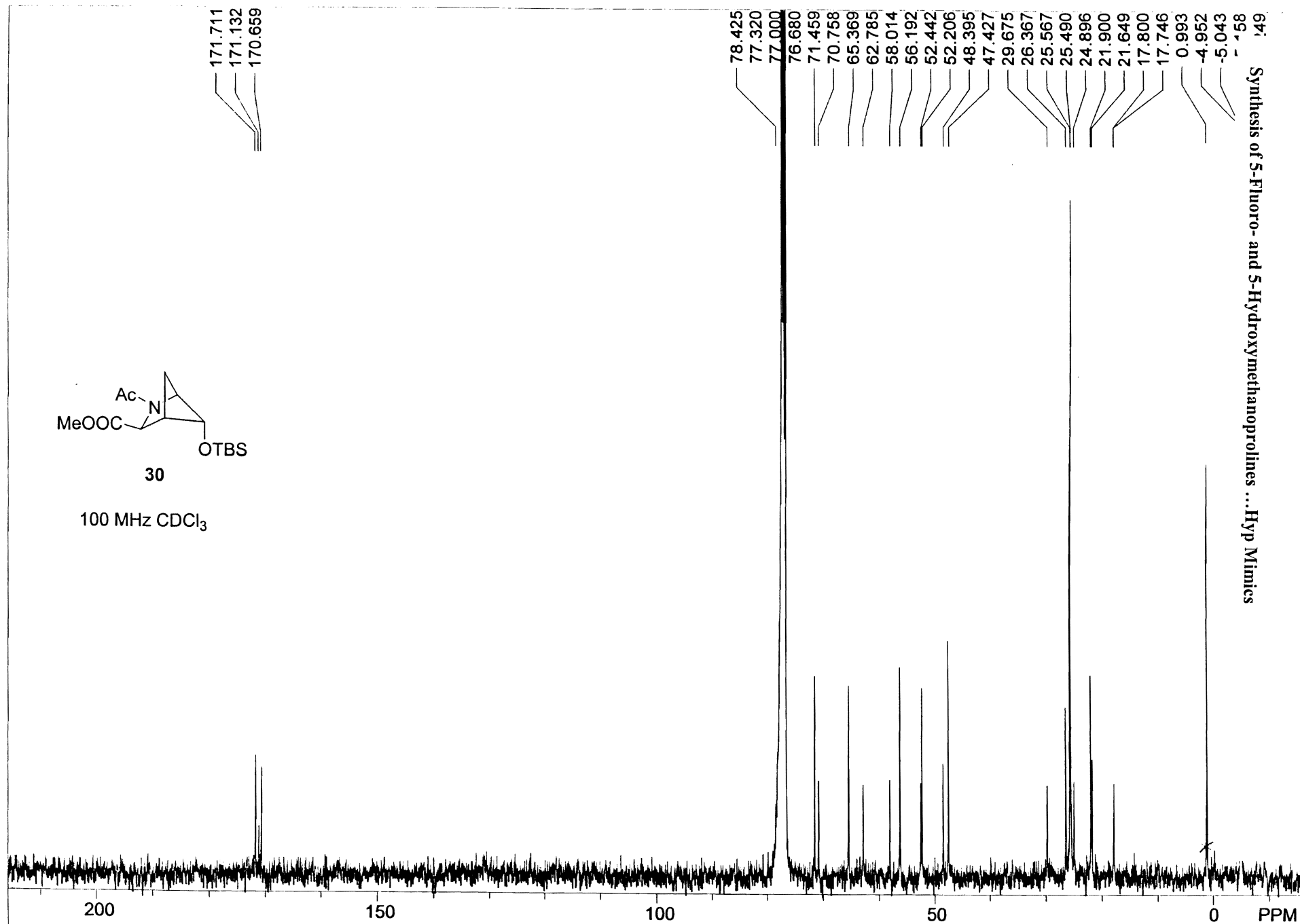
EX:

PW: 9.8 us

PD: 1.0 sec

NA: 128

Name: 3-CO₂Me NAc[2.1.1] syn-OTBS--H (D₂O)--eram-1175

100 MHz CDCl₃

3-CO2Me NAc[2.1.1] syn-OTBS, carbonOVNTE.all,

USER: temple -- DATE: May 16 09:20:14 2008 EDT (UT-4h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10021.1

PTS1d: 32768

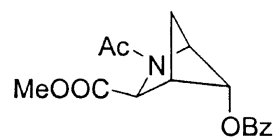
EX:

PW: 5.7 us

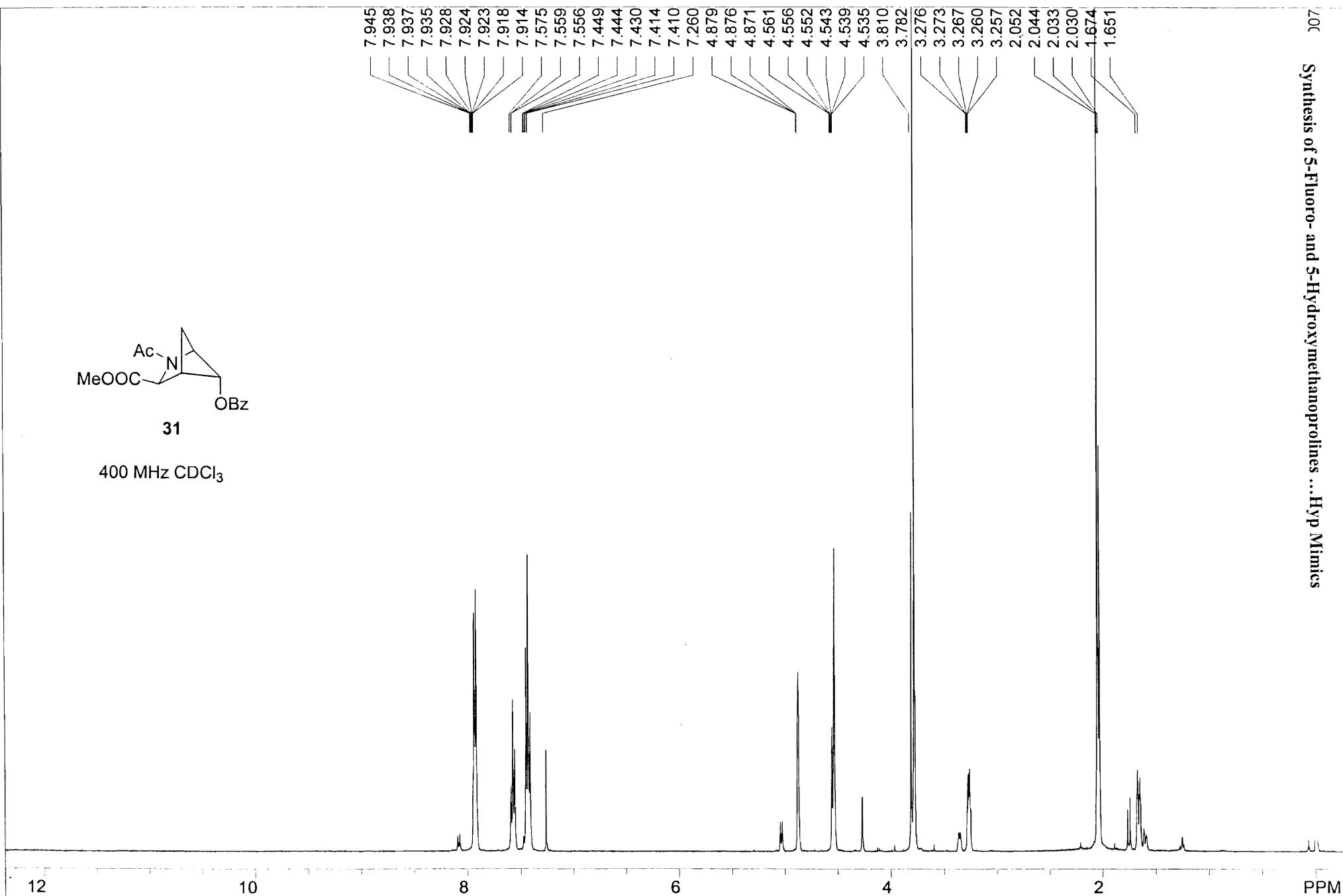
PD: 5.0 sec

NA: 7000

LB: 3.0Nuts - 3-CO2Me NAc[2.1.1] syn-OTBS---C--eram-1181



31

400 MHz CDCl₃3-CO₂Me NAc[2.1.1] syn-OBz, proton.atm,

USER: temple -- DATE: Sep 21 17:08:36 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8013

OF1: 2004.8

PTS1d: 32768

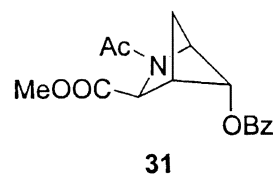
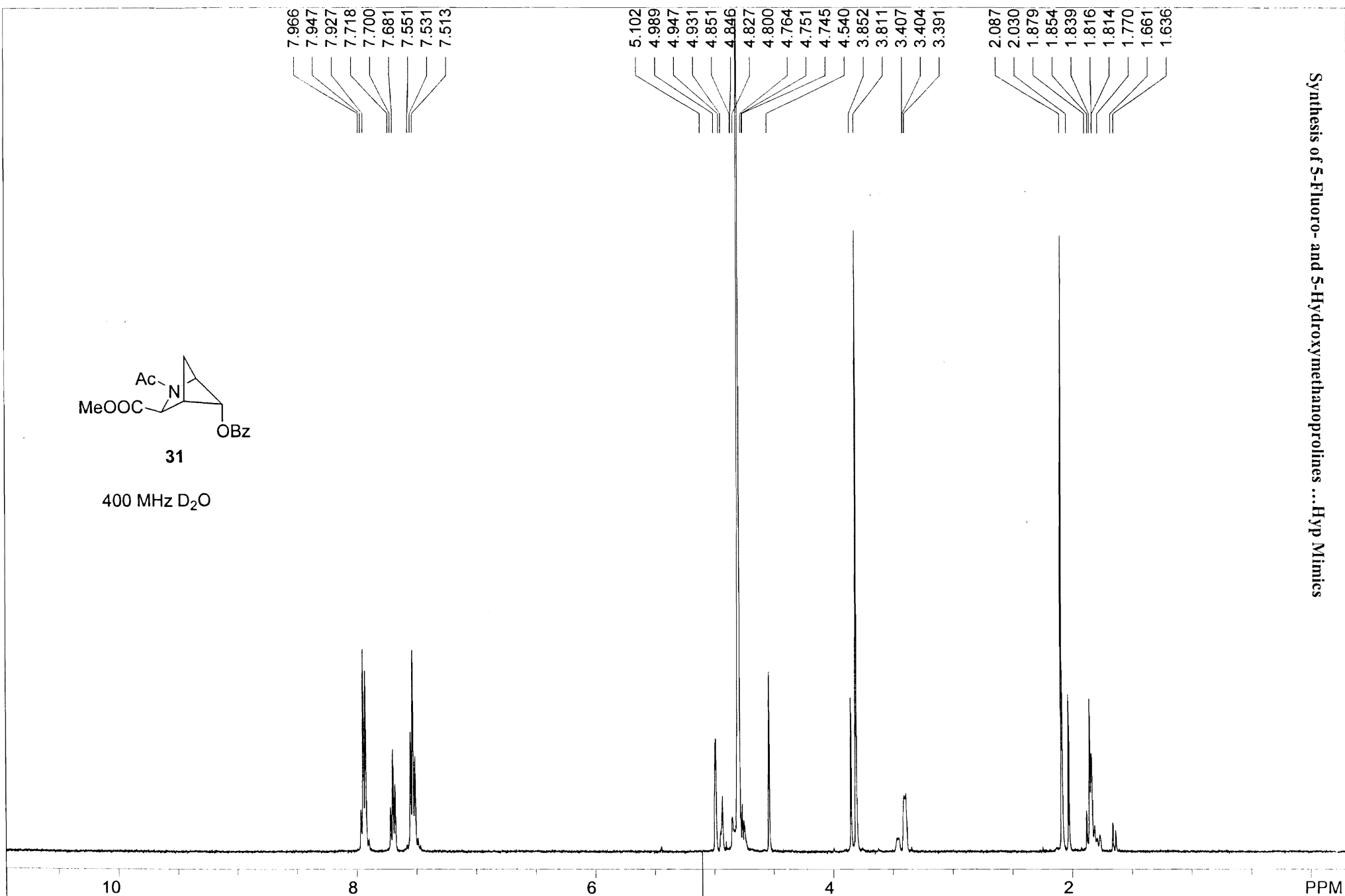
EX:

PW: 9.8 usec

PD: 1.0 sec

NA: 300

LB: 0.1 WinNuts - 3-CO₂Me NAc[2.1.1]syn-OBz---H--eram-1540

400 MHz D₂O3-CO₂Me NAc[2.1.1] syn-OBz, proton10MIN.atm,

USER: temple -- DATE: Oct 2 20:52:43 2008 EDT (UT-4h) temp

F1: 400.130 F2: 1.000

SW1: 8278

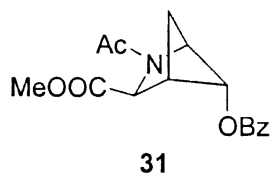
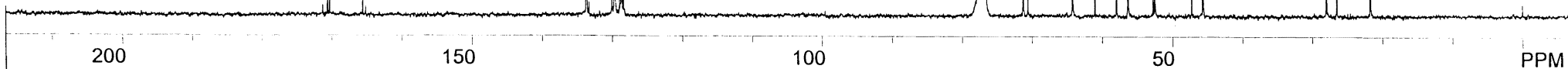
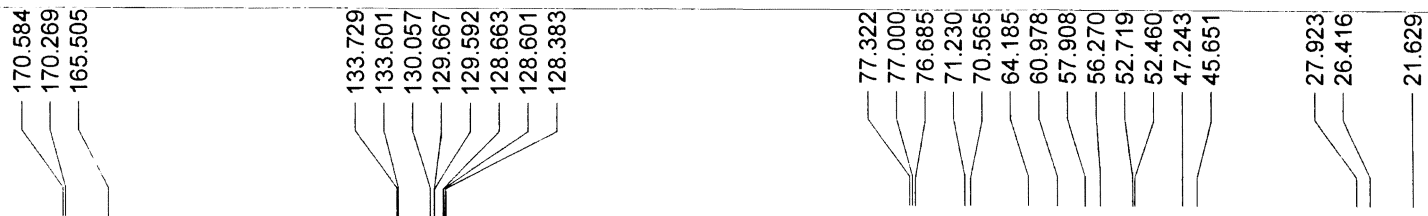
OF1: 2041.4

PTS1d: 32768

EX: PW: 9.8 usec PD: 1.0 sec

NA: 428

LBW: 1570 W: 1570 Nuts - 3-CO₂Me NAc[2.1.1]syn-OBz---H (D₂O)--eram-1570

100 MHz CDCl₃3-CO₂Me NAc[2.1.1] syn-OBz, carbonOVNTE.all,

USER: temple -- DATE: Sep 21 10:34:53 2008 EDT (UT-4h) temp

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10022.3

PTS1d: 32768

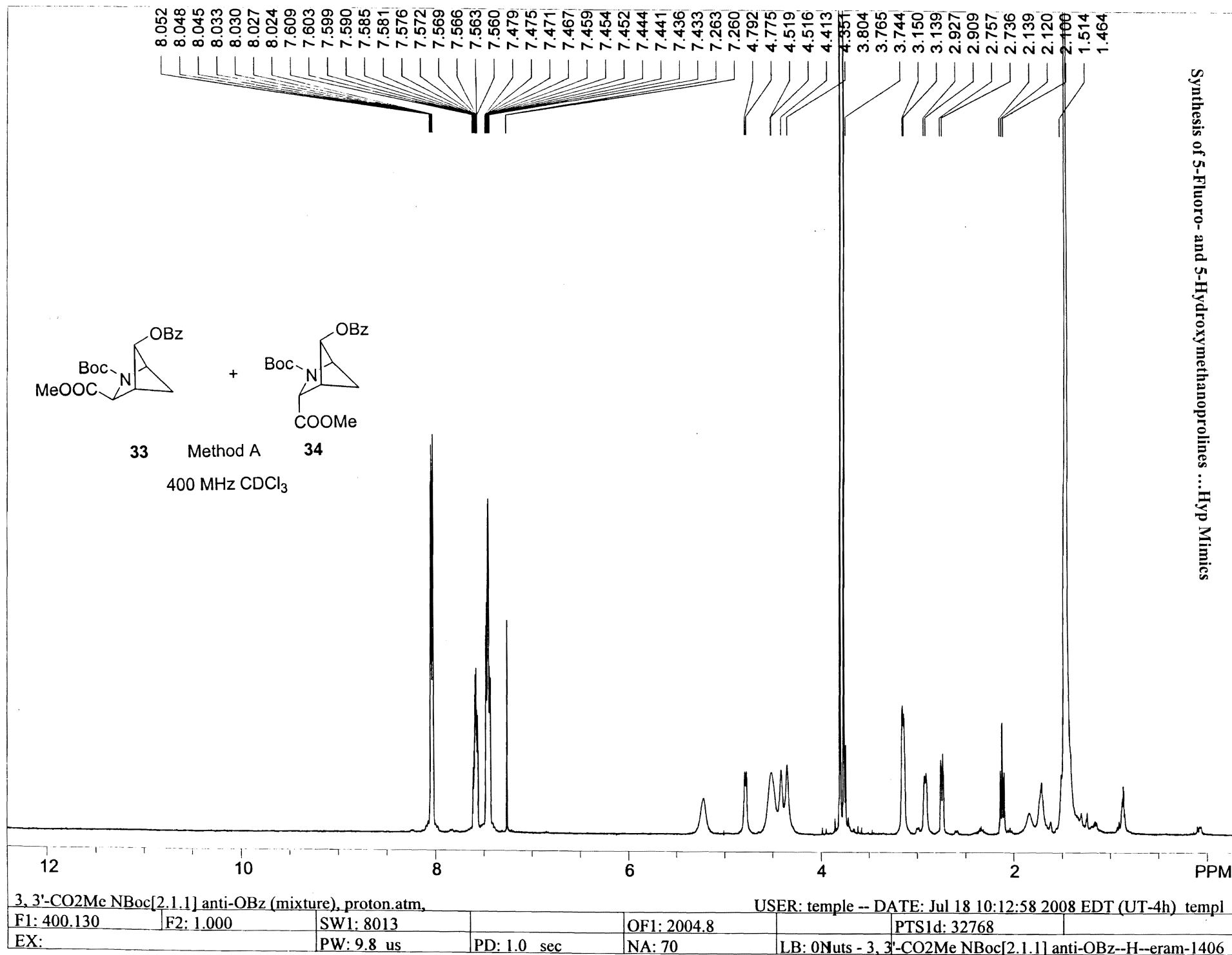
EX:

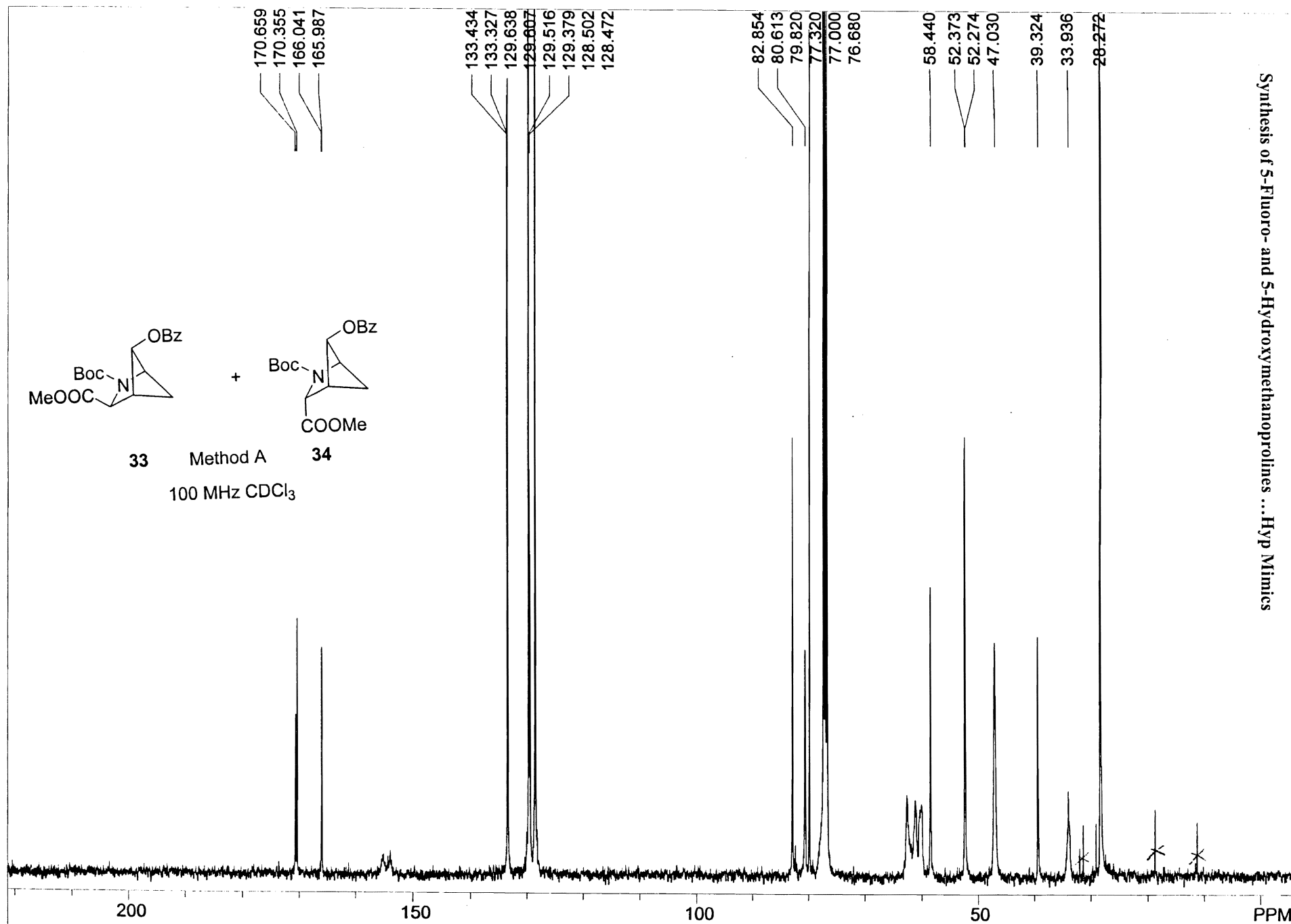
PW: 5.7 usec

PD: 2.0 sec

NA: 15000

LB: 3.0 WinNuts - 3 CO₂Me NAc[2.1.1]syn-OBz---C--eram-1536



3, 3'-CO₂Me NBoc[2.1.1] anti-OBz (mixture), carbonOVNTE.all,

USER: temple -- DATE: Jul 19 08:15:54 2008 EDT (UT-4h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10019.6

PTS1d: 32768

EX:

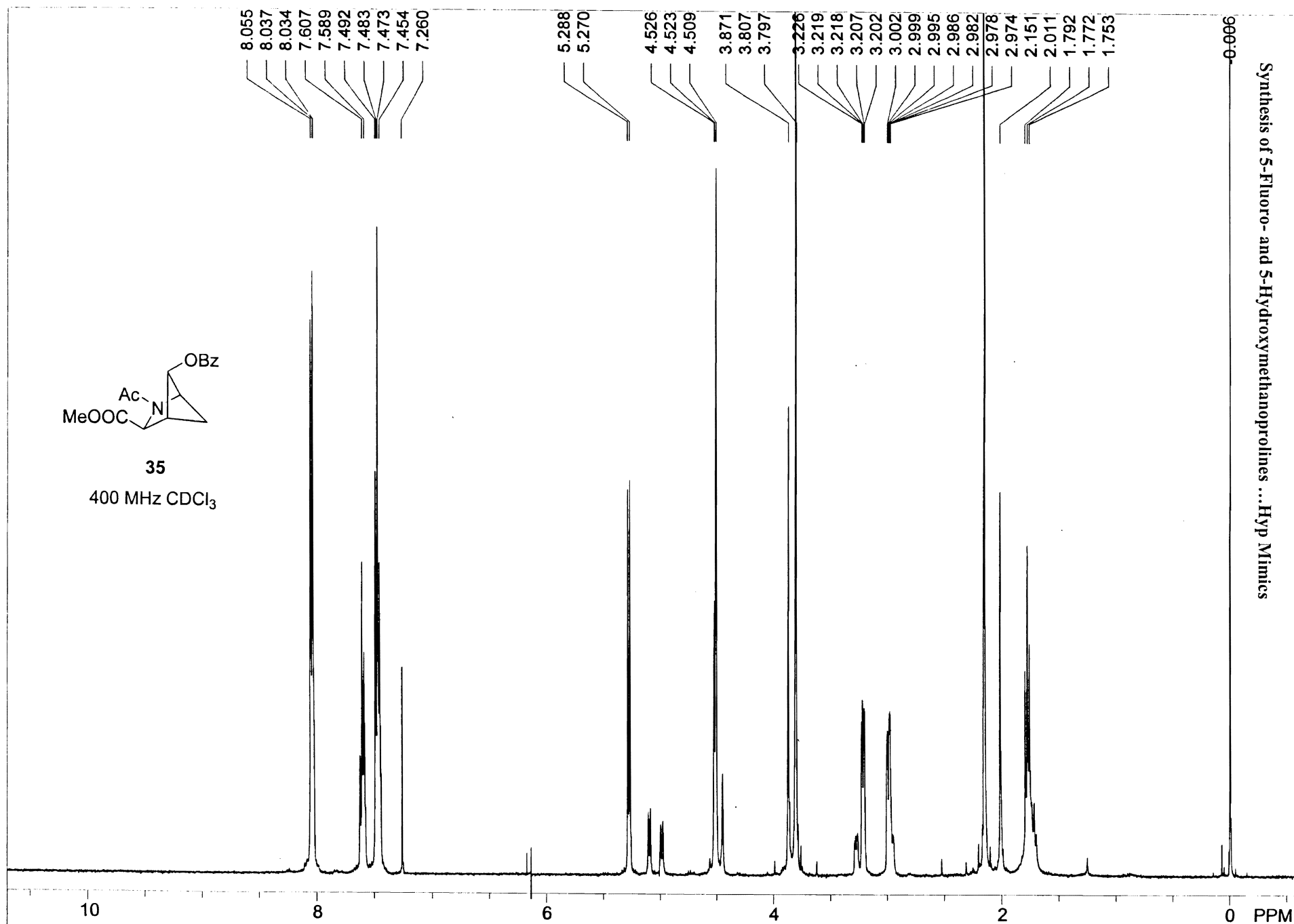
PW: 5.7 us

PD: 2.0 sec

NA: 13000

LB: 3 Hz

3, 3'-CO₂Me NBoc[2.1.1] anti-OBz--C--eram-1408



Endo-3-CO2Me NAc[2.1.1] anti-OBz, proton10MIN.atm,

USER: temple -- DATE: Jul 16 10:52:16 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2003.8

PTS1d: 32768

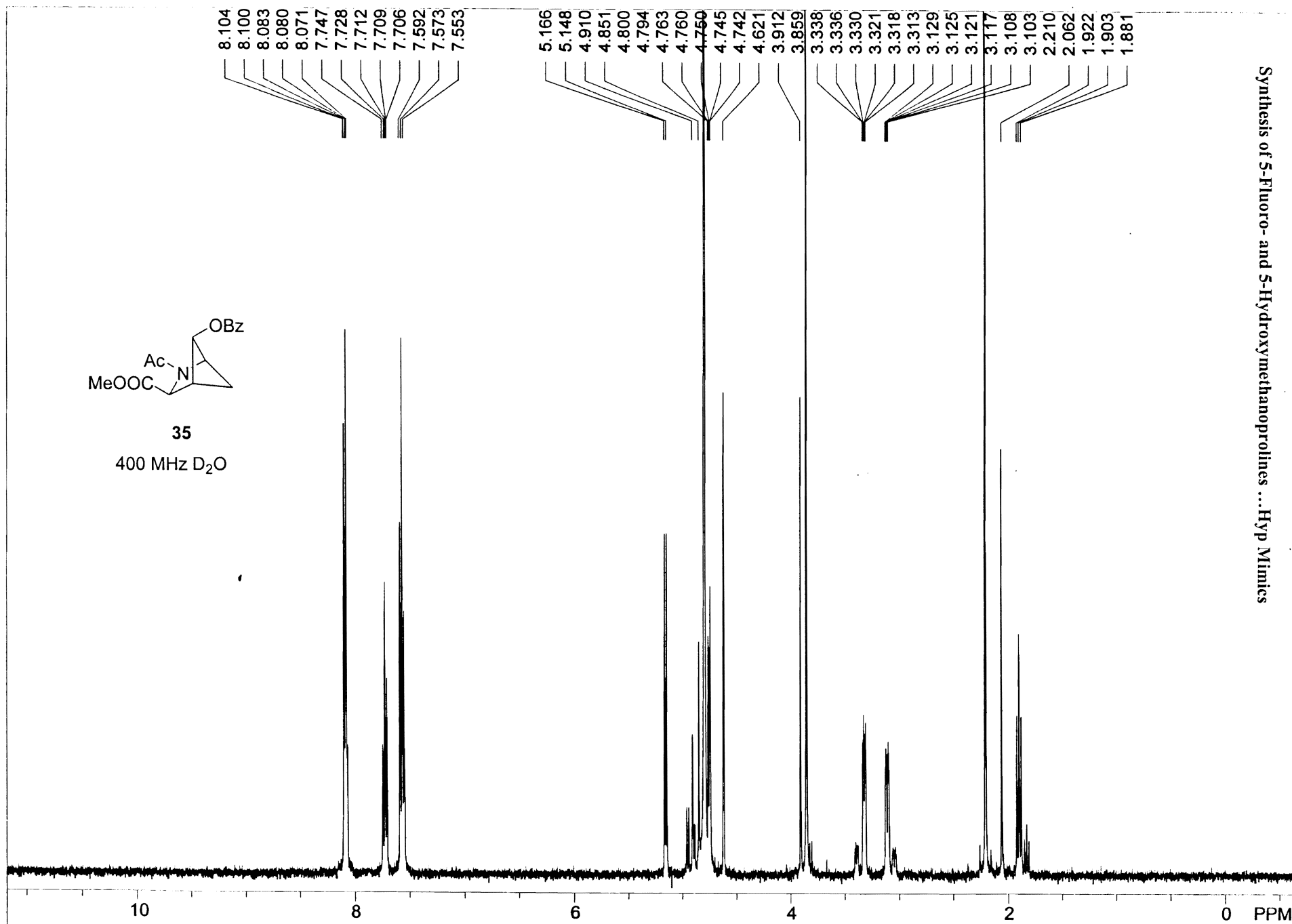
EX:

PW: 9.8 us

PD: 1.0 sec

NA: 228

Nuts-L-Endo-3-CO2Me NAc[2.1.1] anti-OBz--CDCl3--H--eram1389

endo-3-CO₂Me NAc[2.1.1] anti-OBz, proton10MIN.atm,

USER: temple -- DATE: Jul 16 16:21:53 2008 EDT (UT-4h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2040.8

PTS1d: 32768

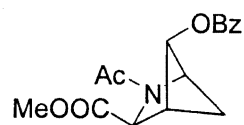
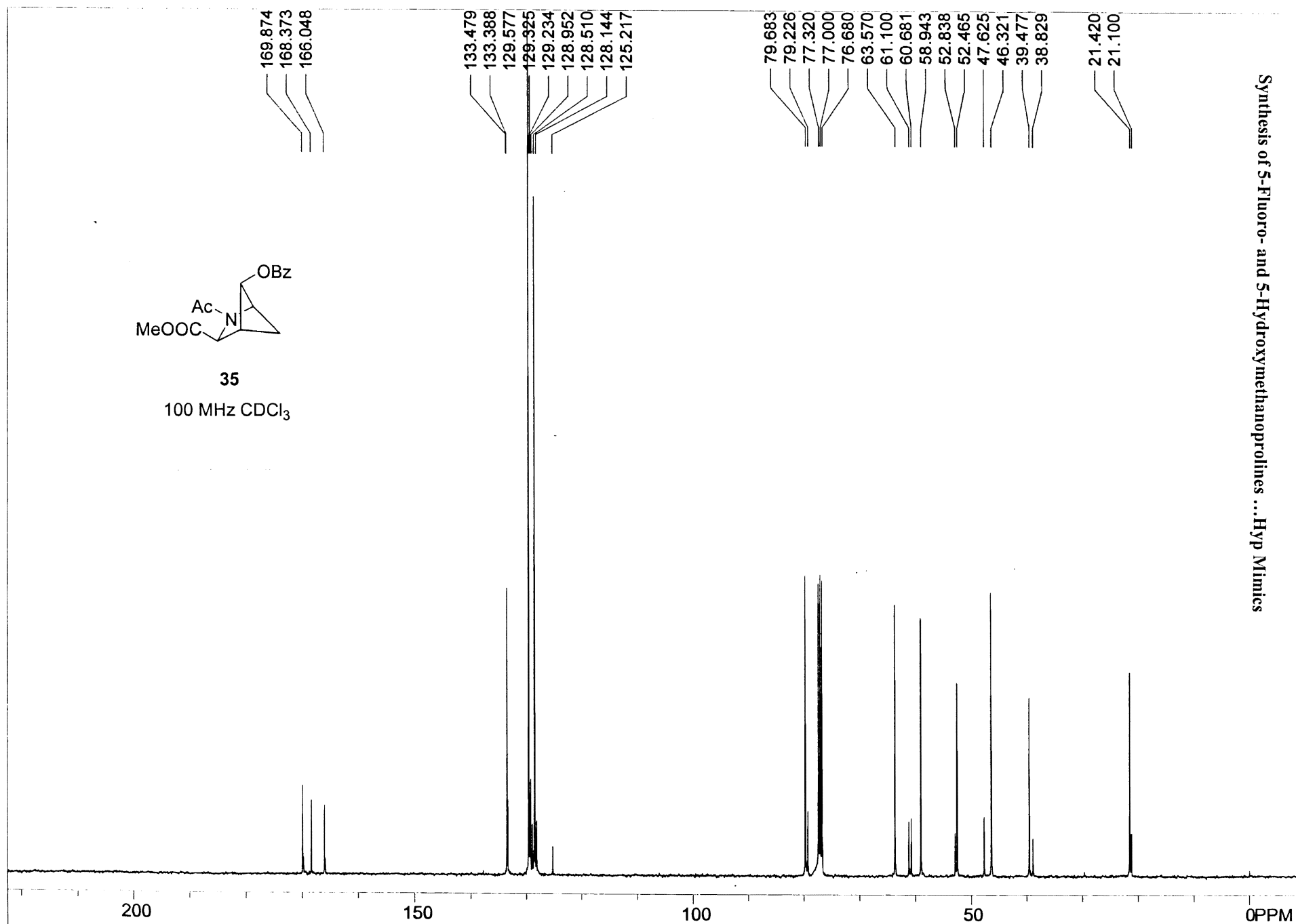
EX:

PW: 9.8 us

PD: 1.0 sec

NA: 180

N1: B: Endo -3-CO₂Me NAc[2.1.1] anti-OBz--D₂O--H--eram1401

**35**100 MHz CDCl₃3-CO₂Me NAc[2.1.1]OBz, carbon2HR.atm,

USER: temple -- DATE: Jan 28 03:14:40 2008 EST (UT-5h) templ

F1: 100.613

F2: 1.000

SW1: 25126

OF1: 10016.5

PTS1d: 32768

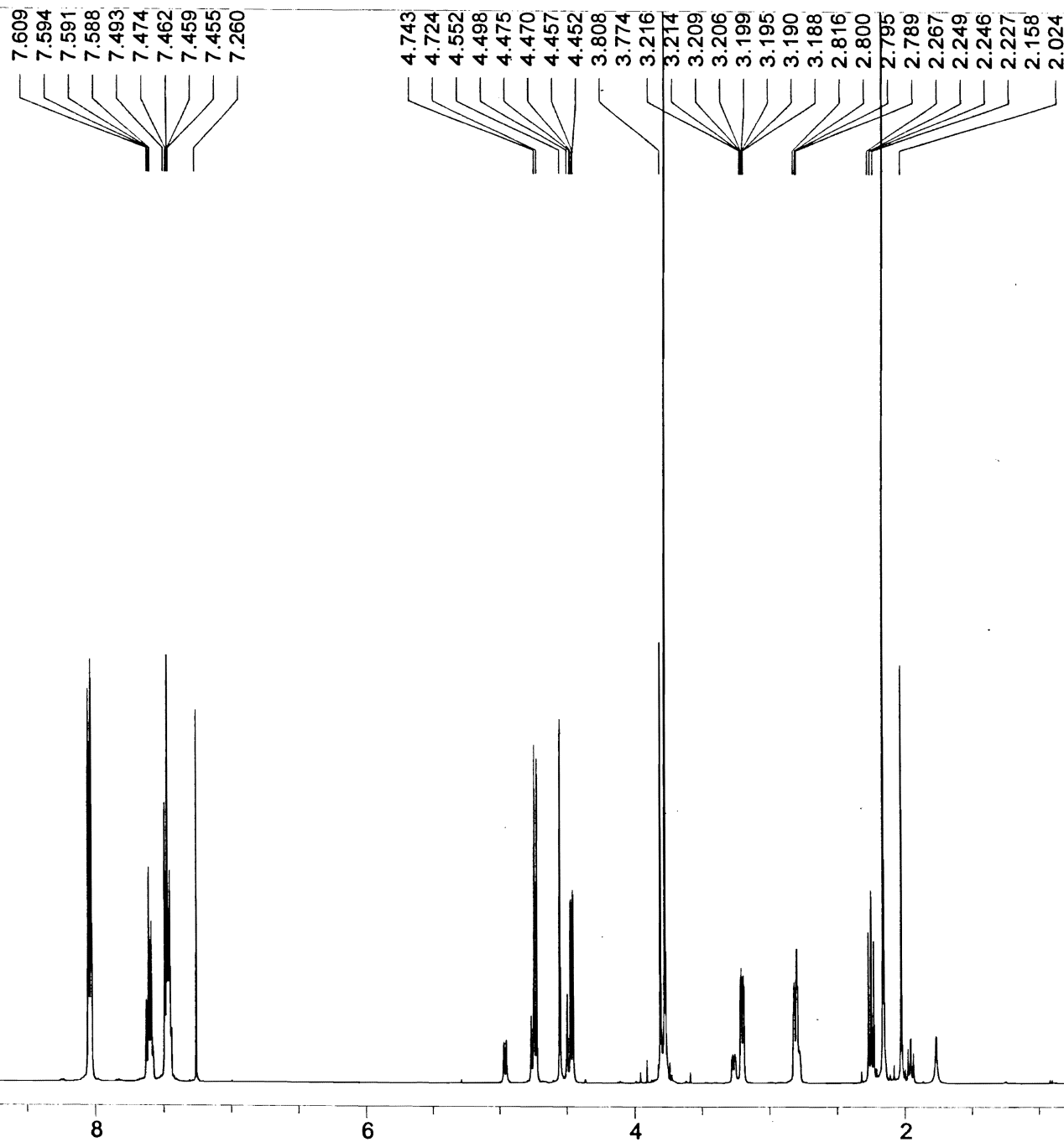
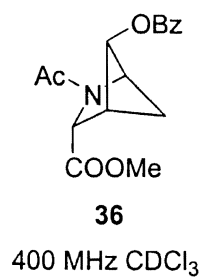
EX:

PW: 5.7 us

PD: 2.0 sec

NA: 5048

NutsBEndo-3-CO₂Me NAc[2.1.1] anti-OBz--CDCl₃--C--eram839

3-CO₂Me NAc[2.1.1] OBz (Major), proton.atm,

USER: temple -- DATE: Nov 5 00:06:55 2007 EST (UT-5h) templ

F1: 400.191

F2: 1.000

SW1: 8278

OF1: 2002.5

PTS1d: 32768

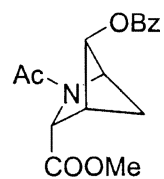
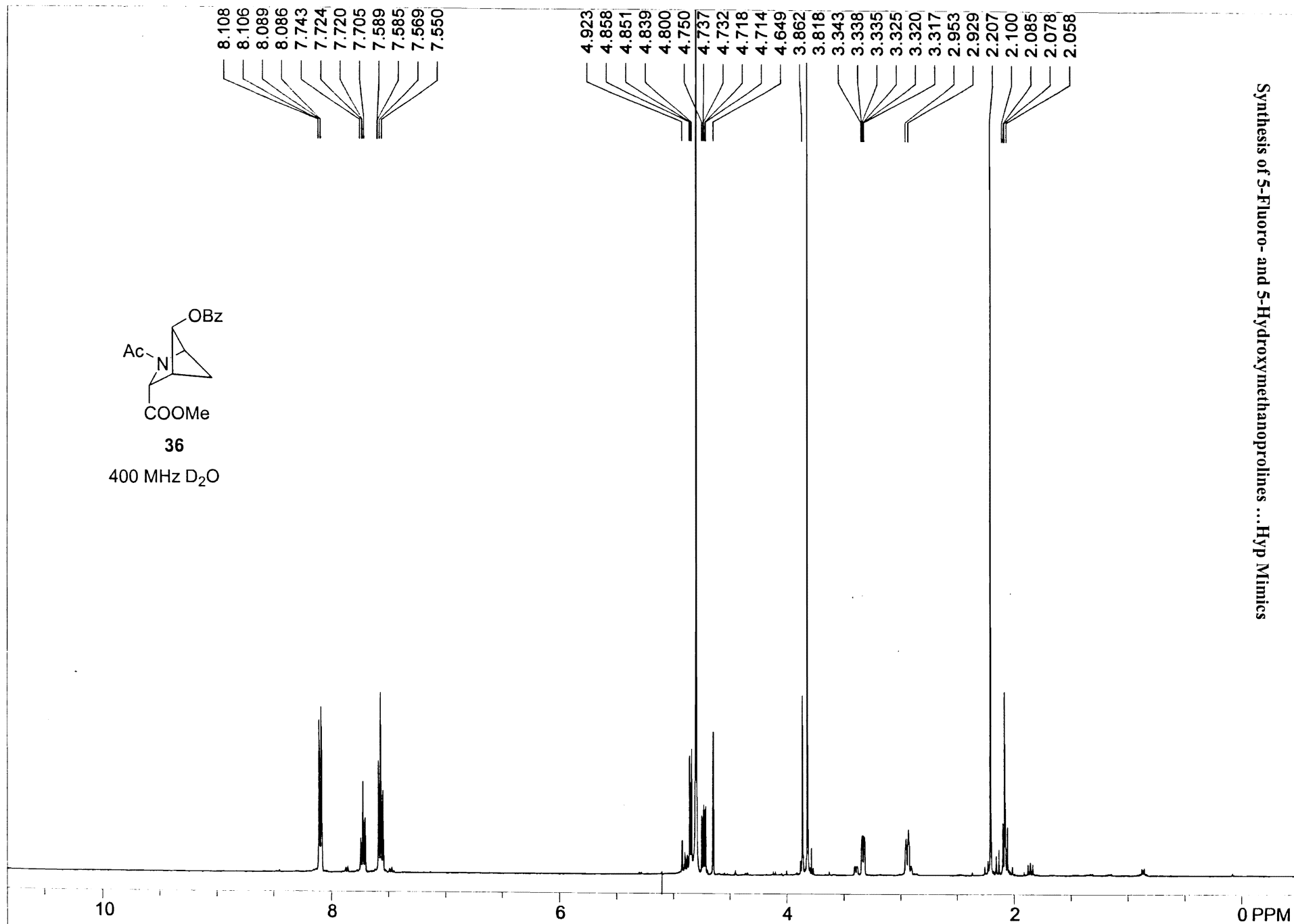
EX:

PW: 9.8 us

PD: 1.0 sec

NA: 16

NMR: Exo-3-CO₂Me NAc[2.1.1] anti-OBz--CDCl₃--H--eram-794

**36**400 MHz D₂O3-CO₂Me NAc[2.1.1]OBz, proton10MIN.atm,

USER: temple -- DATE: Feb 15 21:56:50 2008 EST (UT-5h) templ

F1: 400.130

F2: 1.000

SW1: 8278

OF1: 2040.6

PTS1d: 32768

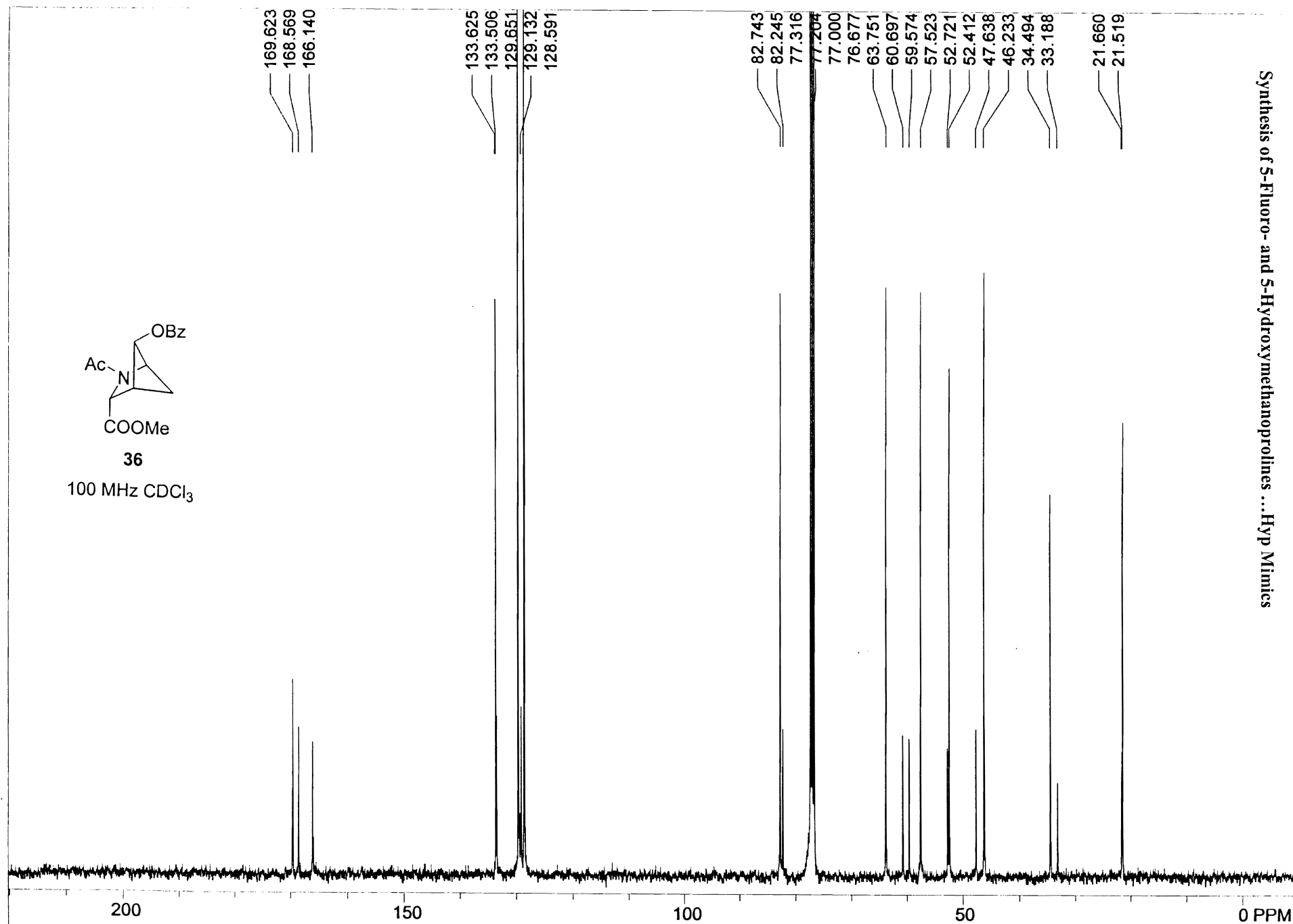
EX:

PW: 9.8 us

PD: 1.0 sec

NA: 328

D-Brt9-1Exo-3-CO₂Me NAc[2.1.1] anti-OBz--D₂O--H--eram906



3-CO2Me NAc[2.1.1] OBz, carbon2HR.atm,

USER: temple -- DATE: Nov 5 00:02:05 2007 EST (UT-5h) templ

F1: 100.628

F2: 1.000

SW1: 23148

OF1: 10581.5

PTS1d: 32768

EX:

PW: 5.7 us

PD: 2.0 sec

NA: 3248

NMR: 3-CO2Me NAc[2.1.1] anti-OBz--CDCl3--C--eram-793