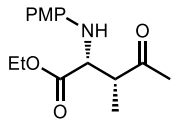
Supporting Information

Total Synthesis and Absolute Structure of N55, a Positive Modulator of GLP-1 Signaling

*Nai-Pin Lin and Rong-Jie Chein**
Institute of Chemistry, Academia Sinica, Taipei 11529, Taiwan

Table of Contents	**Page**

HPLC analysis of *ent*-8

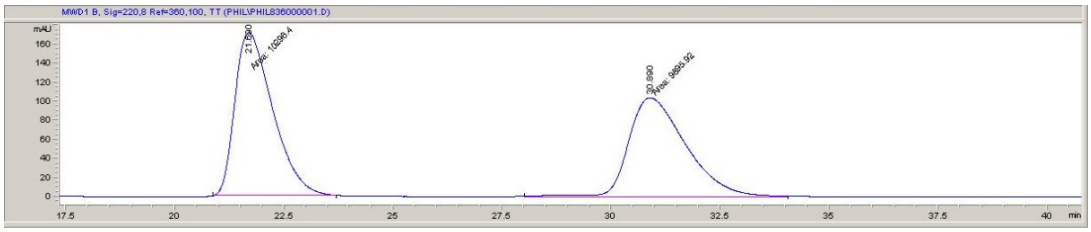


ent-**8**

HPLC condition: Chiralpak-AS, 1.0 mL/min, 220 nm, hexane/*i*-PrOH 97/3, 21.7 (minor) and 30.9 min (major).

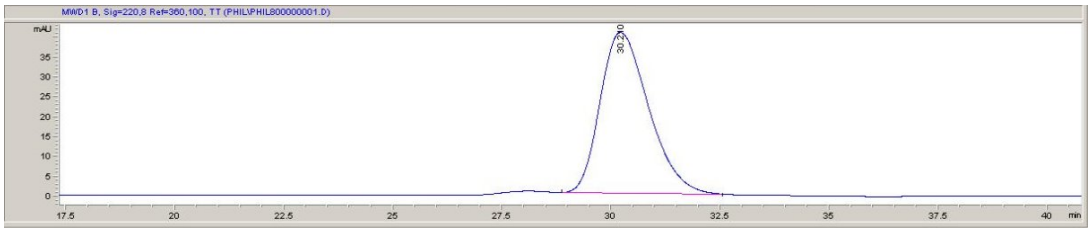
Racemic **8**:

#	Time	Area	Height	Width	Area%	Symmetry
1	21.69	10296.4	171.2	1.0023	50.992	0.565
2	30.89	9895.9	104.8	1.5736	49.008	0.608



ent-**8** with >99% ee:

#	Time	Area	Height	Width	Area%	Symmetry
1	30.21	3119.4	40.3	1.1442	100.000	0.688



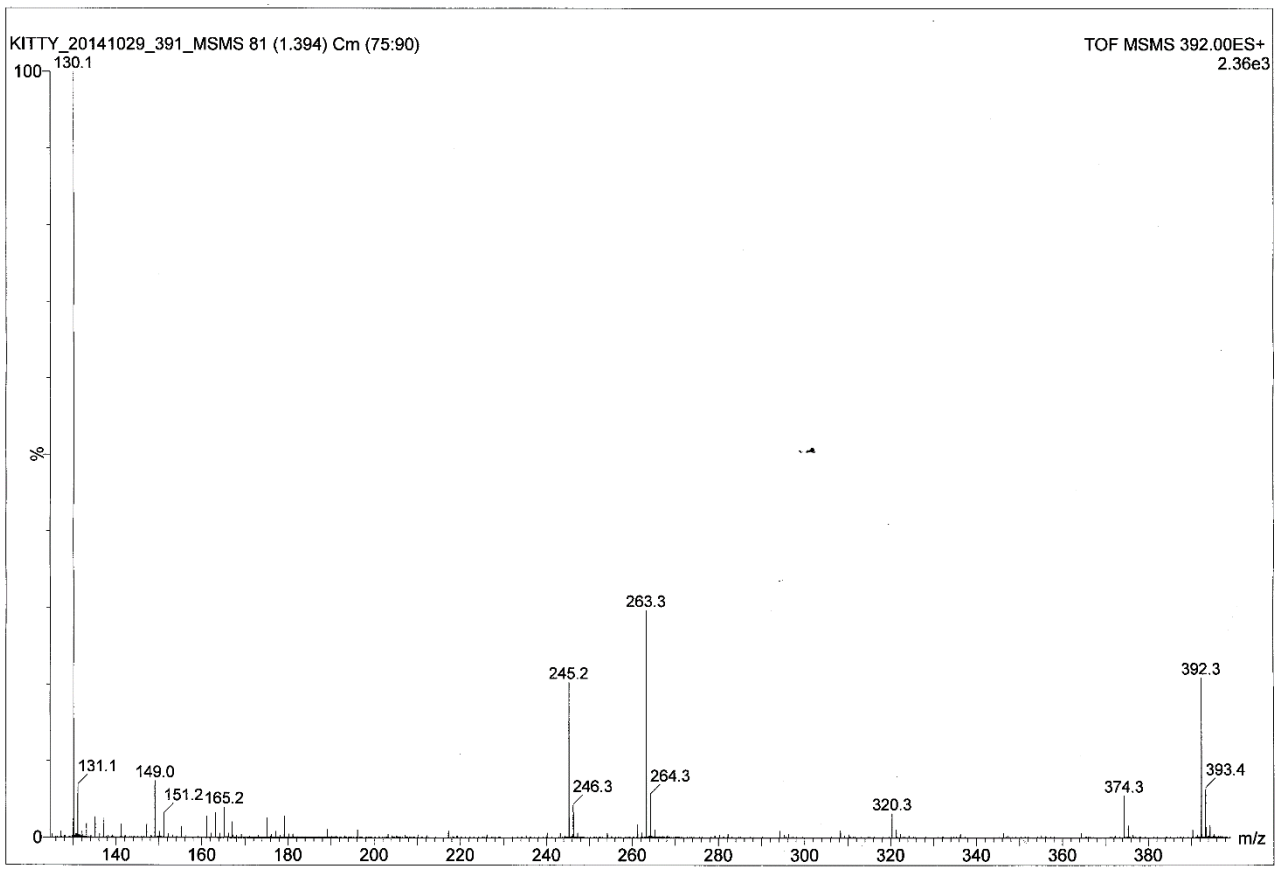
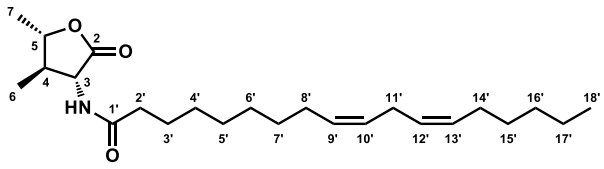


Figure S1. The MS/MS spectra of **N55**

Table S1. Comparative ^{1}H and ^{13}C NMR spectra data between isolated **N55** and synthetic **N55**



Source	Isolated **N55** [a]	Synthetic **N55** [a]	Isolated **N55** [a]	Synthetic **N55** [a]
Position	δ_H (ppm)	δ_H (ppm)	δ_C (ppm)	δ_C (ppm)
2	-	-	**176.2** (s)	**176.2** (s)
3	**4.38** (d, *J* = 11.8 Hz)	**4,38** (d, *J* = 11.8 Hz)	**57.5** (d)	**57.5** (d)
4	**2.17-2.04** (m)	**2.17-2.04** (m)	**45.6** (d)	**45.6** (d)
5	**4.19** (dt, *J* = 9.8, 6.1 Hz)	**4.19** (dt, *J* = 9.8, 6.1 Hz)	**81.4** (d)	**81.4** (d)
6	**1.13** (d, *J* = 6.6 Hz)	**1.13** (d, *J* = 6.6 Hz)	**14.0** (q)	**14.0** (q)
7	**1.41** (d, *J* = 6.1 Hz)	**1.41** (d, *J* = 6.1 Hz)	**18.8** (q)	**18.8** (q)
1'	-	-	**176.5** (s)	**176.5** (s)
2'	**2.26** (t, *J* = 7.4 Hz)	**2.26** (t, *J* = 7.5 Hz)	**36.9** (t)	**36.9** (t)
3'	**1.66-1.60** (m)	**1.66-1.60** (m)	**26.8** (t)	**26.8** (t)
4'-7' & 15'-17'	**1.40-1.28** (m)	**1.40-1.28** (m)	**33-23** (t)	**33-23** (t)
8' & 14'	**2.06** (m)	**2.06** (m)	**28.2** (t)	**28.2** (t)
9', 10', 12' & 13'	**5.39-5.29** (m)	**5.39-5.29** (m)	**130.9 & 129.1** (d)	**130.9 & 129.1** (d)
11'	**2.78** (t, *J* = 6.5 Hz)	**2.78** (t, *J* = 6.5 Hz)	**26.5** (t)	**26.5** (t)
18'	**0.91** (t, *J* = 6.8 Hz)	**0.91** (t, *J* = 6.9 Hz)	**14.4** (q)	**14.4** (q)

[a] 500 MHz NMR in d_4-methanol (δ_H = 3.31 ppm; δ_C = 49.0 ppm)

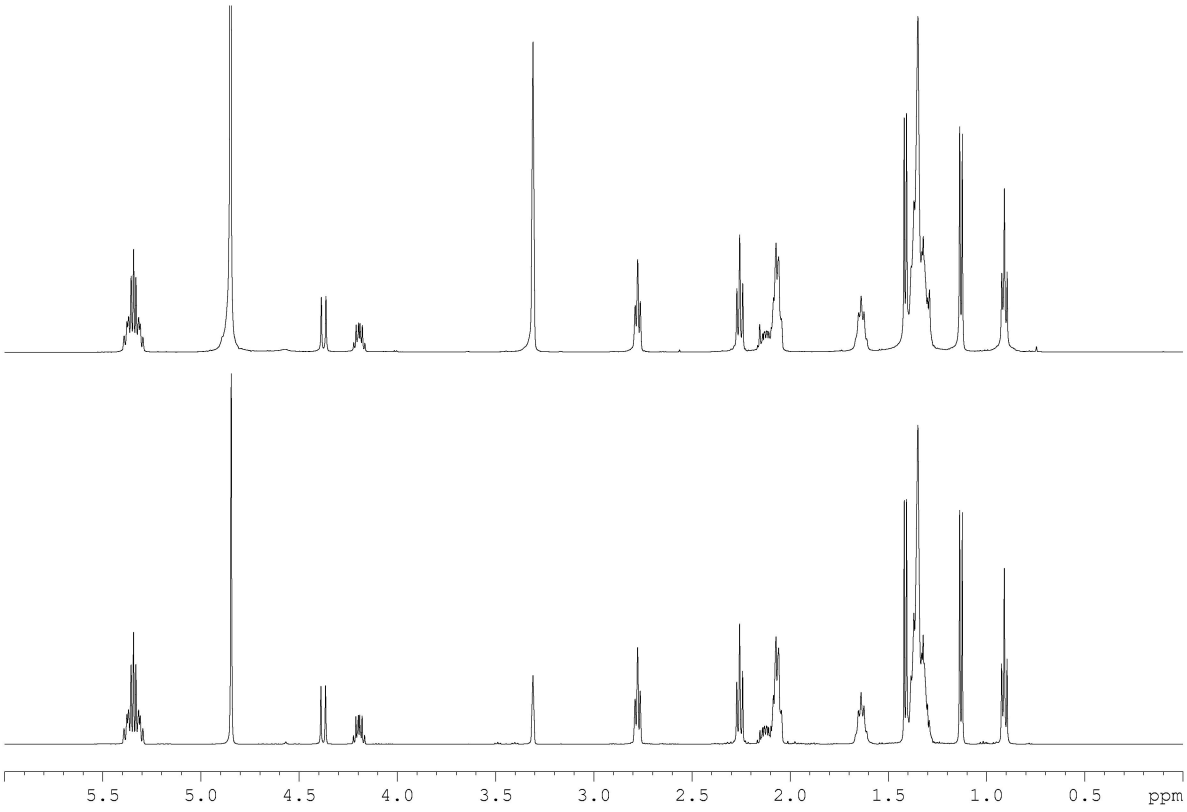


Figure S2. Comparative 500 MHz ^{1}H NMR spectra between isolated **N55** (upper) and synthetic **N55** (lower) in d_4-methanol (δ_H = 3.31 ppm)

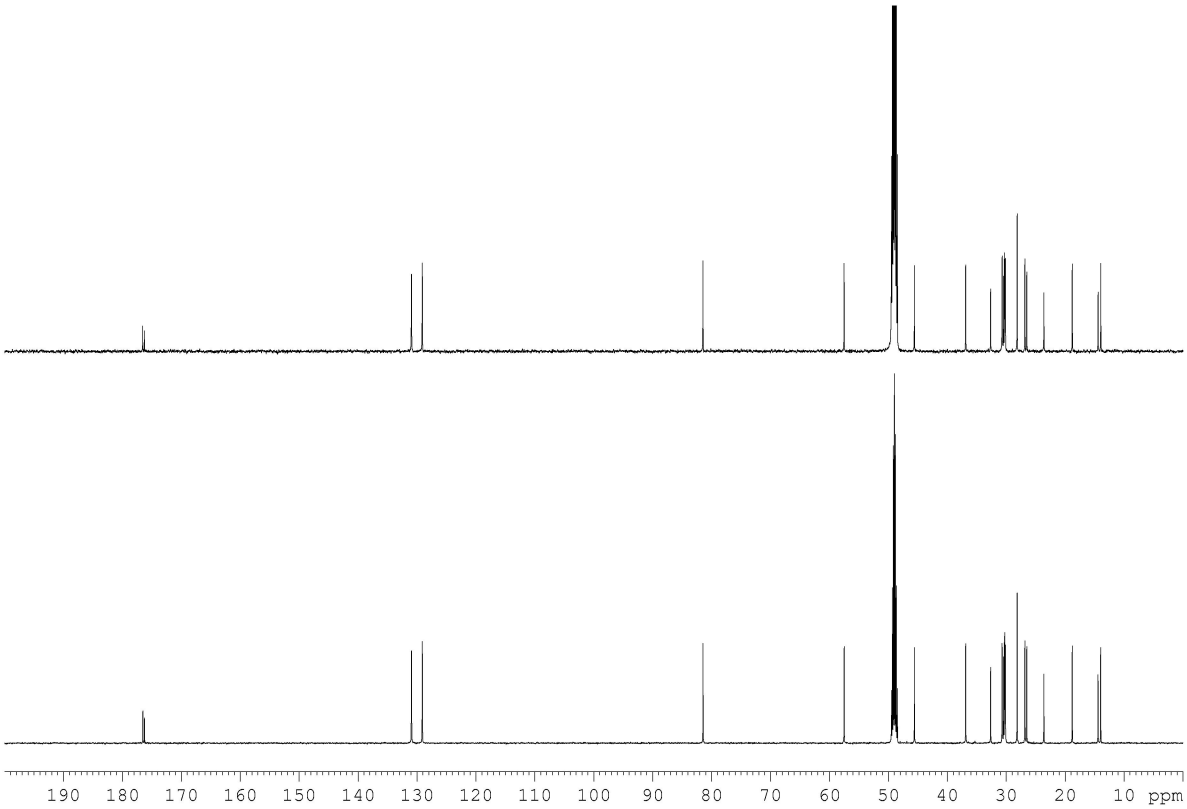
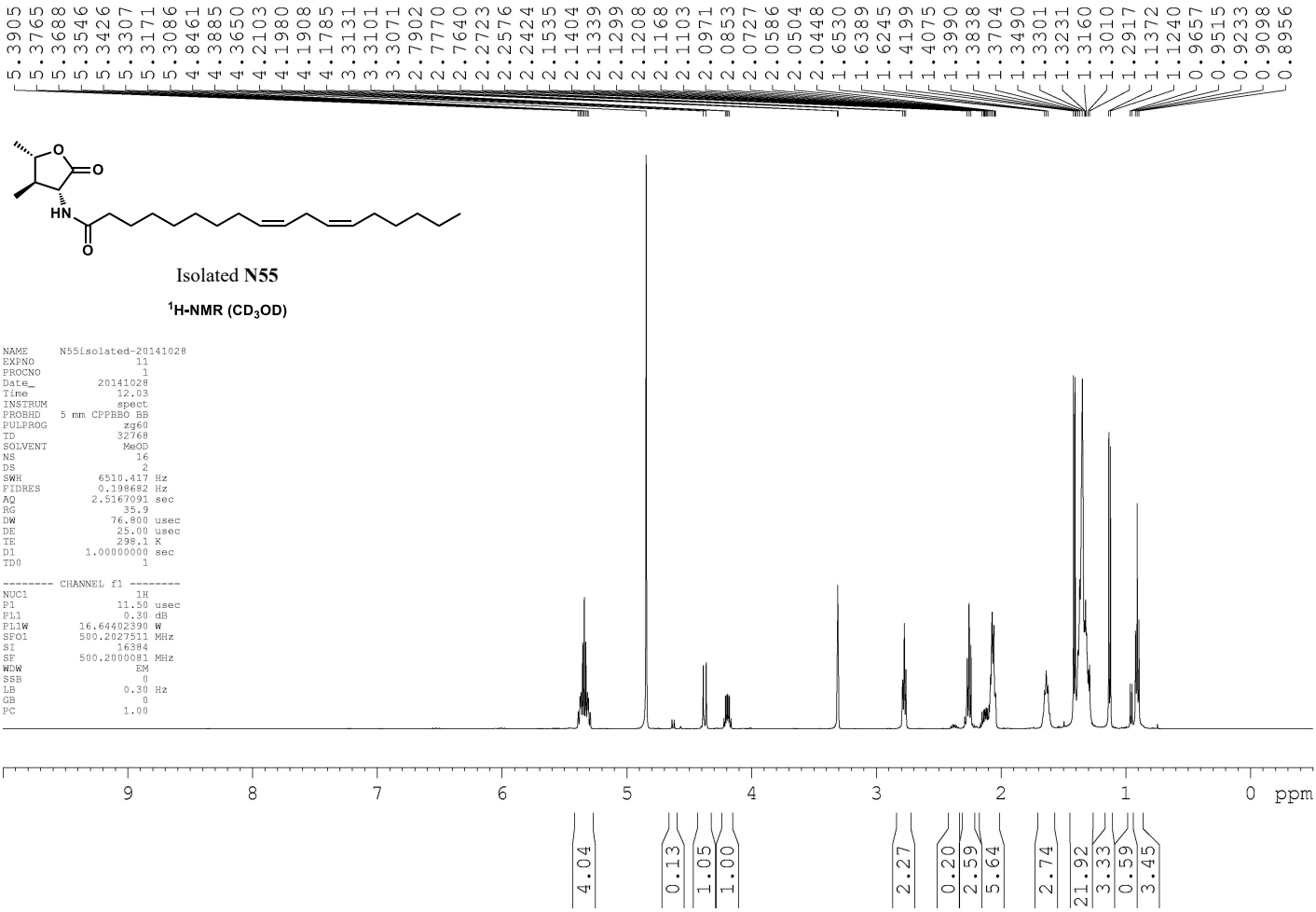
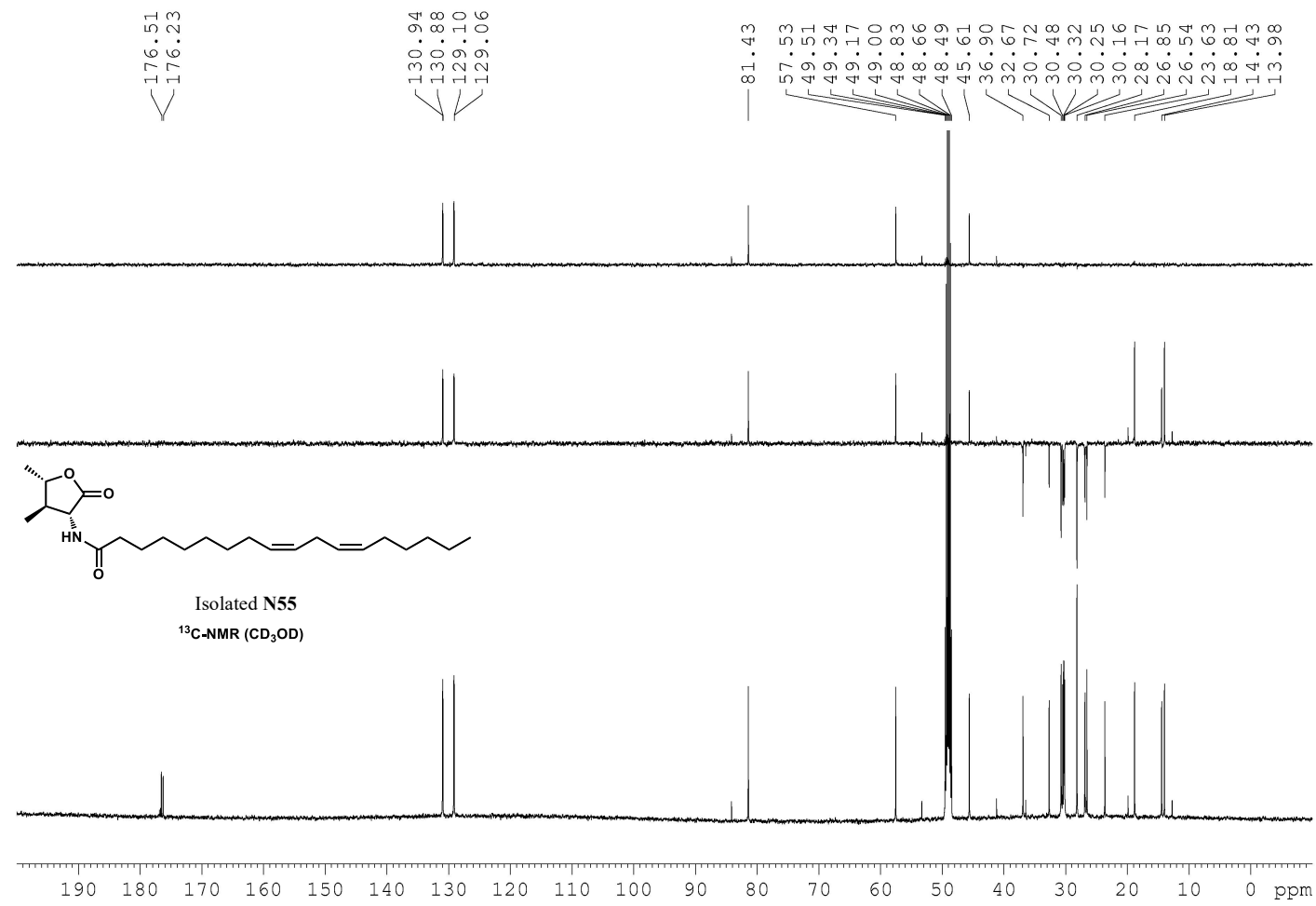
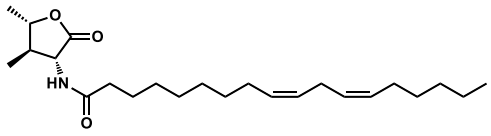


Figure S3. Comparative 125 MHz ^{13}C NMR spectra between isolated **N55** (upper) and synthetic **N55** (lower) in d_4-methanol (δ_C = 49.0 ppm)


5.3905
5.3765
5.3688
5.3546
5.3426
5.3307
5.3171
5.3086
4.8461
4.3885
4.3650
4.2103
4.1980
4.1908
4.1785
3.3131
3.3101
3.3071
3.7902
2.7770
2.7640
2.2723
2.2576
2.2424
2.1535
2.1404
2.1339
2.1299
2.1208
2.1168
2.1103
2.0971
2.0853
2.0727
2.0586
2.0504
2.0448
1.6530
1.6389
1.6245
1.4199
1.4075
1.3990
1.3838
1.3704
1.3490
1.3301
1.3231
1.3160
1.3010
1.2917
1.1372
1.1240
0.9657
0.9515
0.9233
0.9098
0.8956
HN
O
O
Isolated N55
1H-NMR (CD3OD)
NAME N55isolated-20141028
EXPNO 11
PROCNO 1
Date_ 20141028
Time 12.03
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg60
TD 32768
SOLVENT MeOD
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5167091 sec
RG 35.9
DW 76.800 usec
DE 25.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 11.50 usec
PL1 0.30 dB
PL1W 16.64402390 W
SFO1 500.2027511 MHz
SI 16384
SF 500.2000081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
4.04
0.13
1.05
1.00
2.27
0.20
2.59
5.64
2.74
21.92
3.33
0.59
3.45

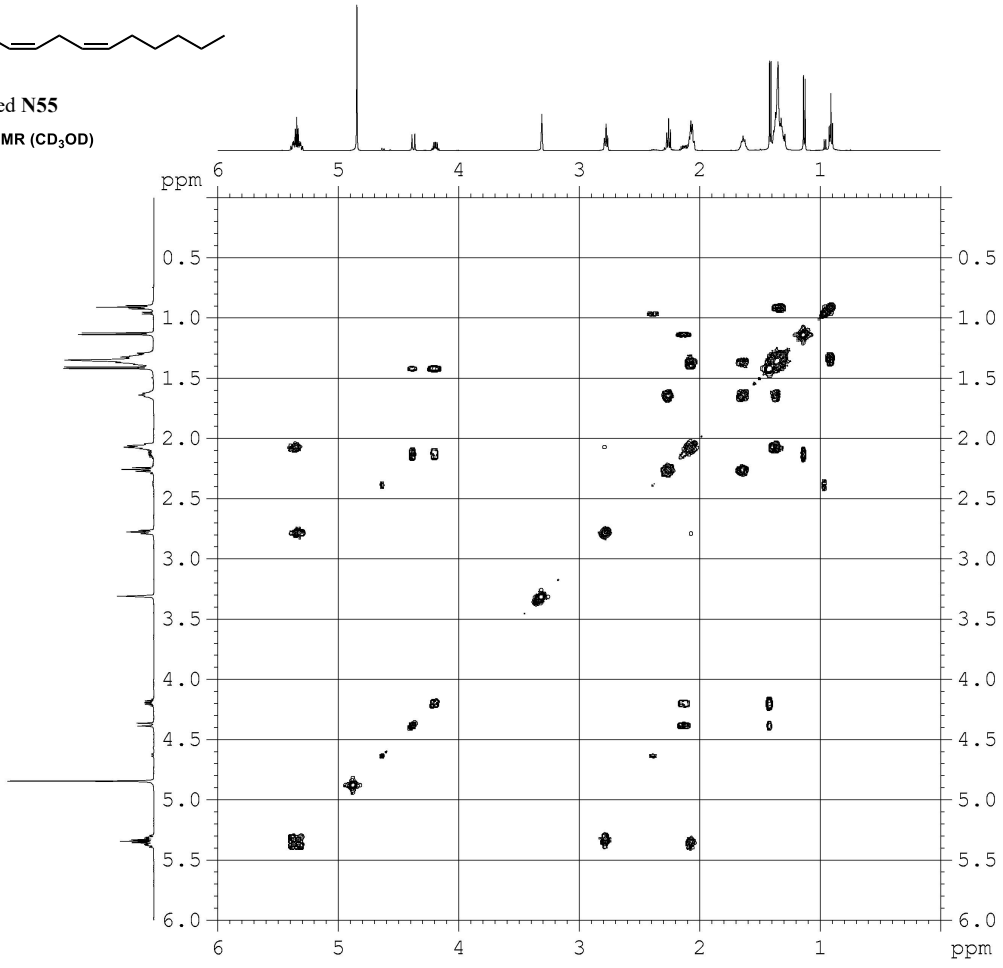

176.51
176.23
130.94
130.88
129.10
129.06
81.43
57.53
49.51
49.34
49.17
49.00
48.83
48.66
48.49
45.61
36.90
32.67
30.72
30.48
30.32
30.25
30.16
28.17
26.85
26.54
23.63
18.81
14.43
13.98
HN
O
O
Isolated N55
13C-NMR (CD3OD)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

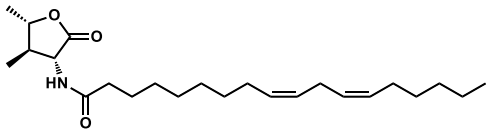


Isolated **N55**

HH-COSY-NMR (CD_3OD)

```
NAME            C1773NS55
EXPNO                  40
PROCNO                  1
Date_            20130430
Time                18.22
INSTRUM             spect
PROBHD    5 mm SEI 1H-13
PULPROG          cosygpqf
TD                   2048
SOLVENT              MeOD
NS                      4
DS                      8
SWH              2866.972 Hz
FIDRES           1.399889 Hz
AQ               0.3573956 sec
RG                     57
DW                174.400 usec
DE                  6.00 usec
TE                300.0 K
d0             0.00000300 sec
D1             1.28290999 sec
d13            0.00000400 sec
D16            0.00010000 sec
IN0            0.00034880 sec
MCREST         0.00000000 sec
MCWRK          1.28290999 sec

======== CHANNEL f1 ========
NUC1                   1H
P0                  10.50 usec
P1                  10.50 usec
PL1                  5.00 dB
SFO1         500.1216345 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               10.00 %
GPZ2               10.00 %
P16              1000.00 usec
ND0                    1
TD                   128
SFO1            500.1216 MHz
FIDRES         22.398222 Hz
SW                 5.733 ppm
FnMODE                 QF
SI                   1024
SF           500.1200081 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                    QF
SF           500.1200081 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
```

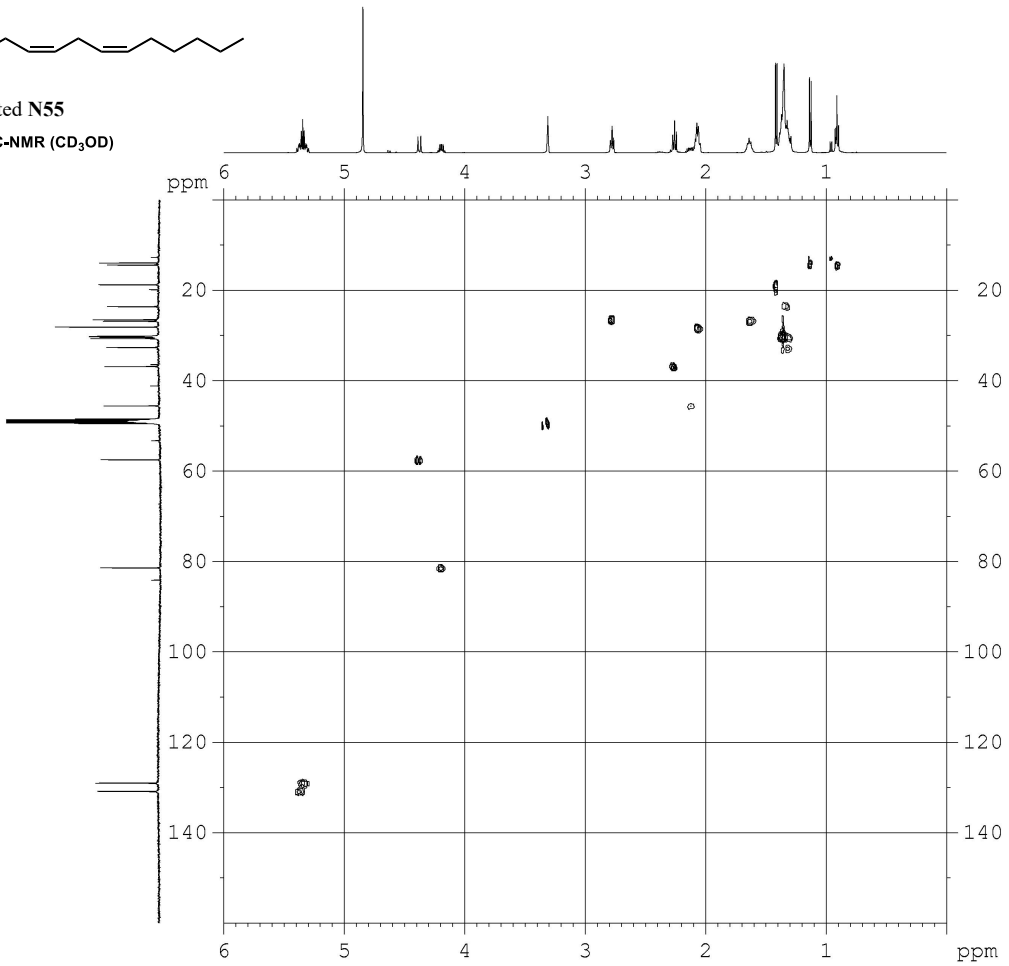


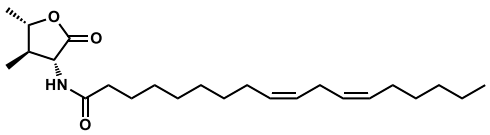


Isolated **N55**

HC-HSQC-NMR (CD_3OD)

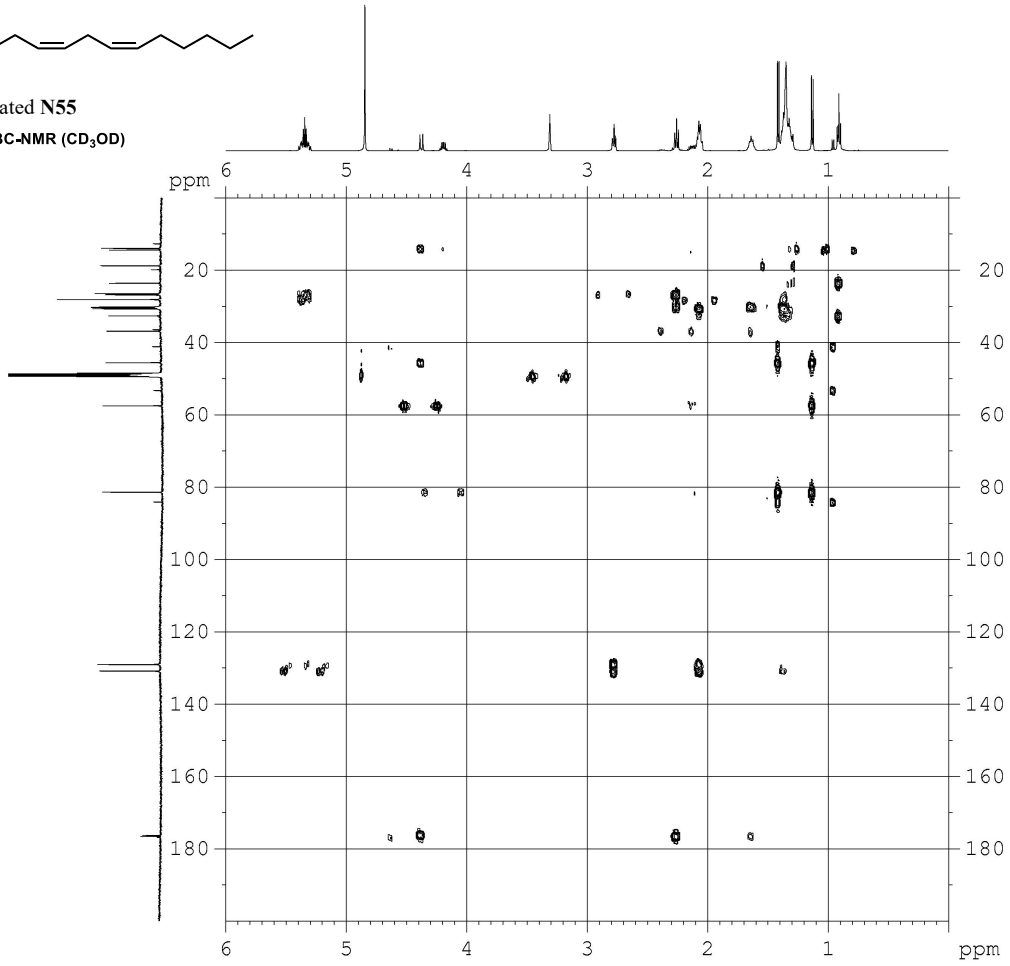
```
NAME            C1773NS55
EXPNO                  20
PROCNO                  1
Date_            20130430
Time                17.05
INSTRUM             spect
PROBHD    5 mm SEI 1H-13
PULPROG        invietgpsi
TD                   2048
SOLVENT              MeOD
NS                      8
DS                      8
SWH              2866.972 Hz
FIDRES           1.399889 Hz
AQ               0.3573956 sec
RG                 18390.4
DW                174.400 usec
DE                  6.00 usec
TE                300.0 K
CNST2          145.0000000
d0             0.00000300 sec
D1             1.86810696 sec
d4             0.00172414 sec
d11            0.03000000 sec
d13            0.00000400 sec
D16            0.00010000 sec
D24            0.00089286 sec
DELTA          0.00117700 sec
DELTA1         0.00110800 sec
IN0            0.00002485 sec
MCREST         0.00000000 sec
MCWRK          0.31135178 sec
ST1CNT                 0

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.50 usec
p2                  21.00 usec
P28               2000.00 usec
PL1                  5.00 dB
SFO1         500.1216345 MHz

======== CHANNEL f2 ========
CPDPRG2             garp
NUC2                 13C
P3                  11.20 usec
p4                  22.40 usec
PCPD2               70.00 usec
PL2                  2.00 dB
PL12                16.00 dB
SFO2         125.7652844 MHz

------ GRADIENT CHANNEL -----
GPNAM1           SINE.100
GPNAM2           SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec
ND0                    2
TD                   128
SFO1            125.7653 MHz
FIDRES         157.193161 Hz
SW                159.986 ppm
FnMODE     Echo-Antiecho
SI                   2048
SF           500.1200081 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2        echo-antiecho
```



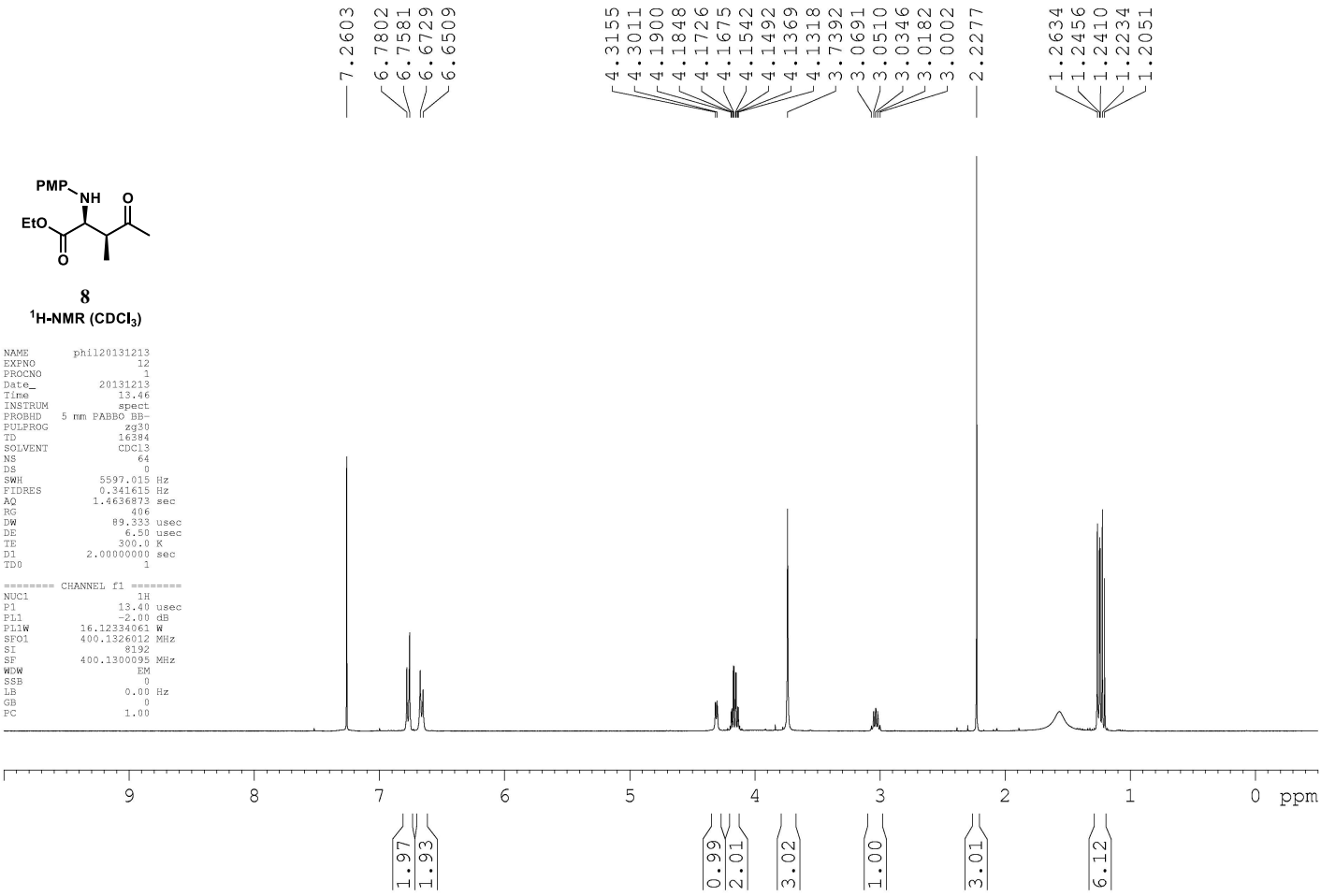


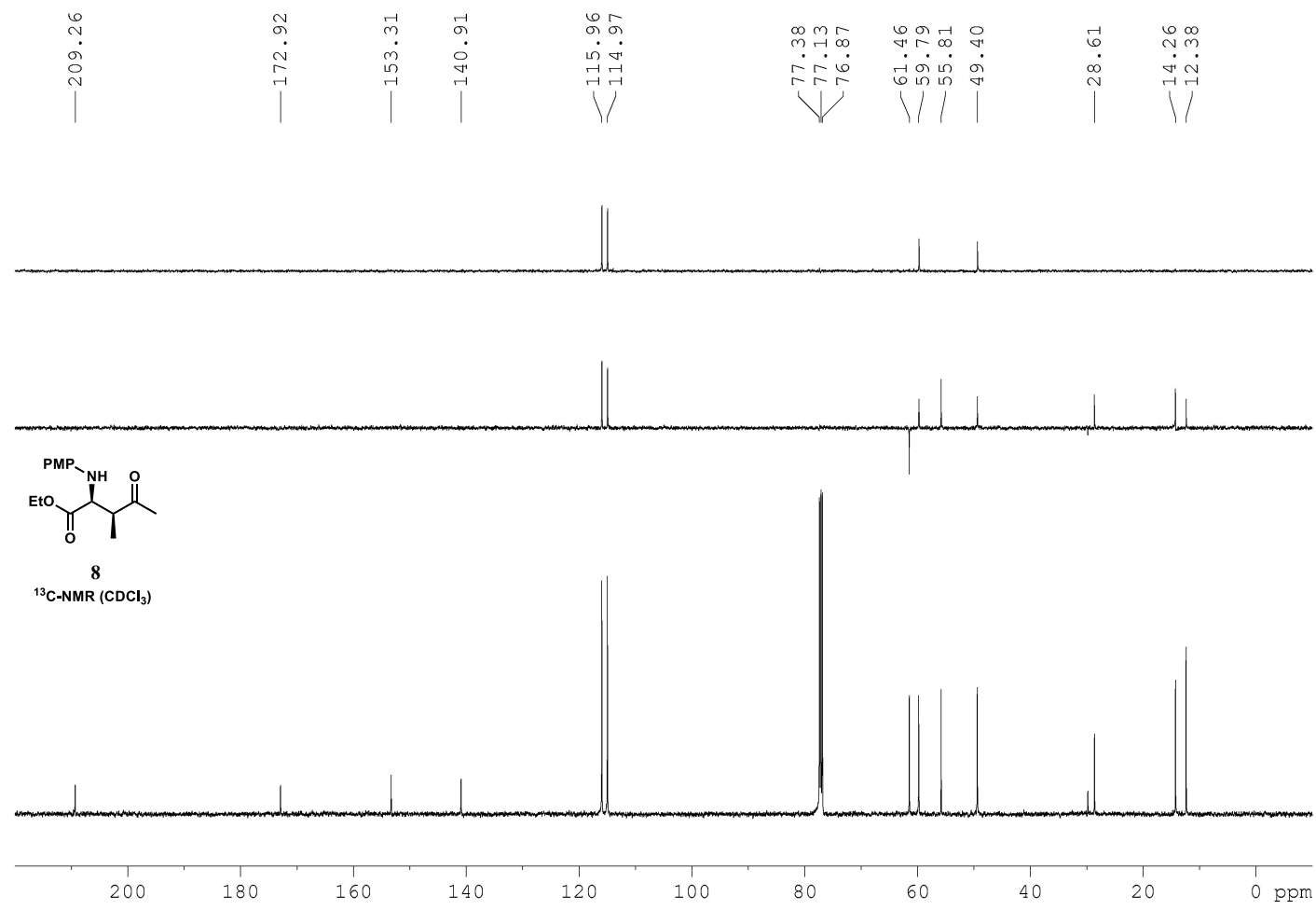
Isolated **N55**

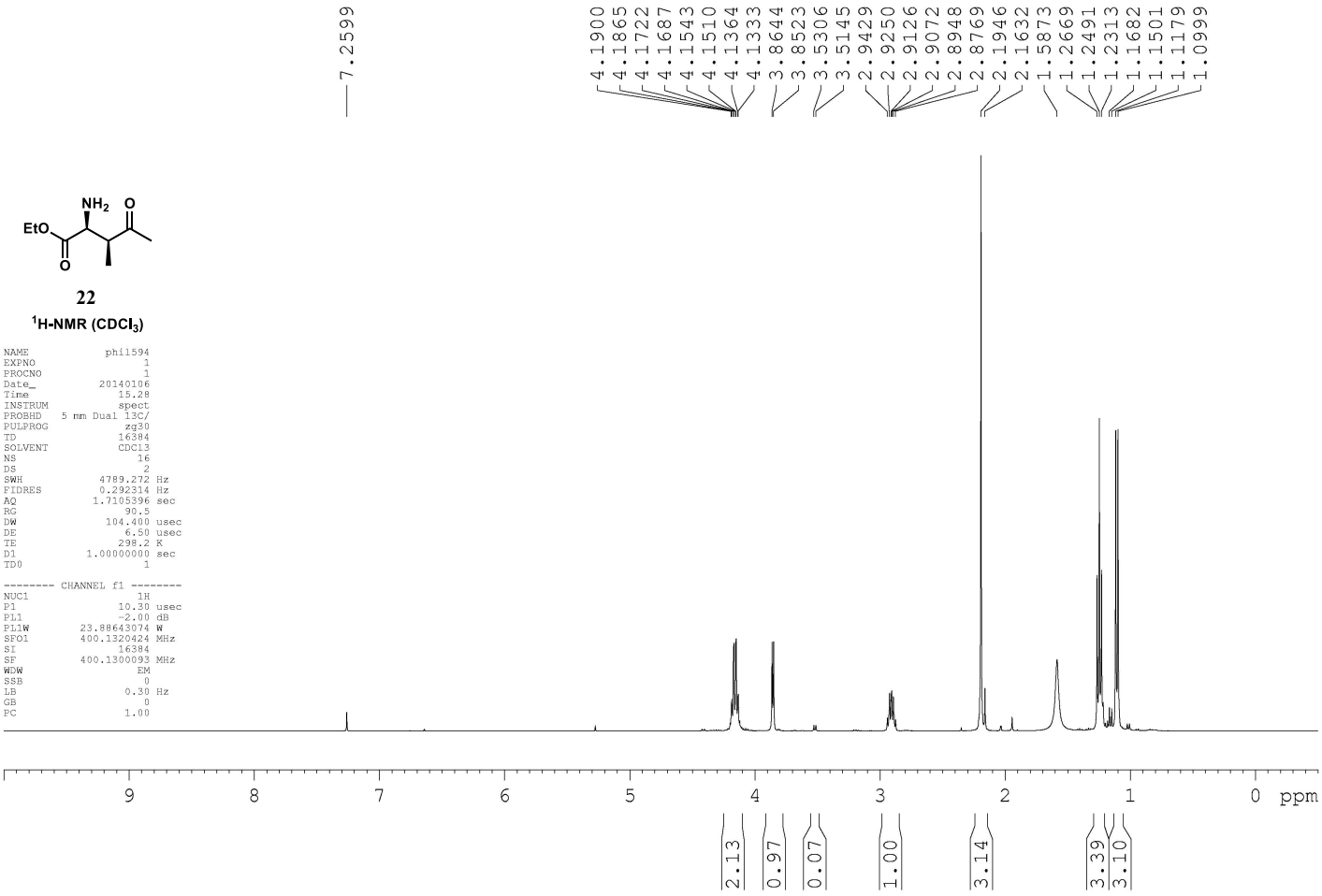
HC-HMBC-NMR (CD_3OD)

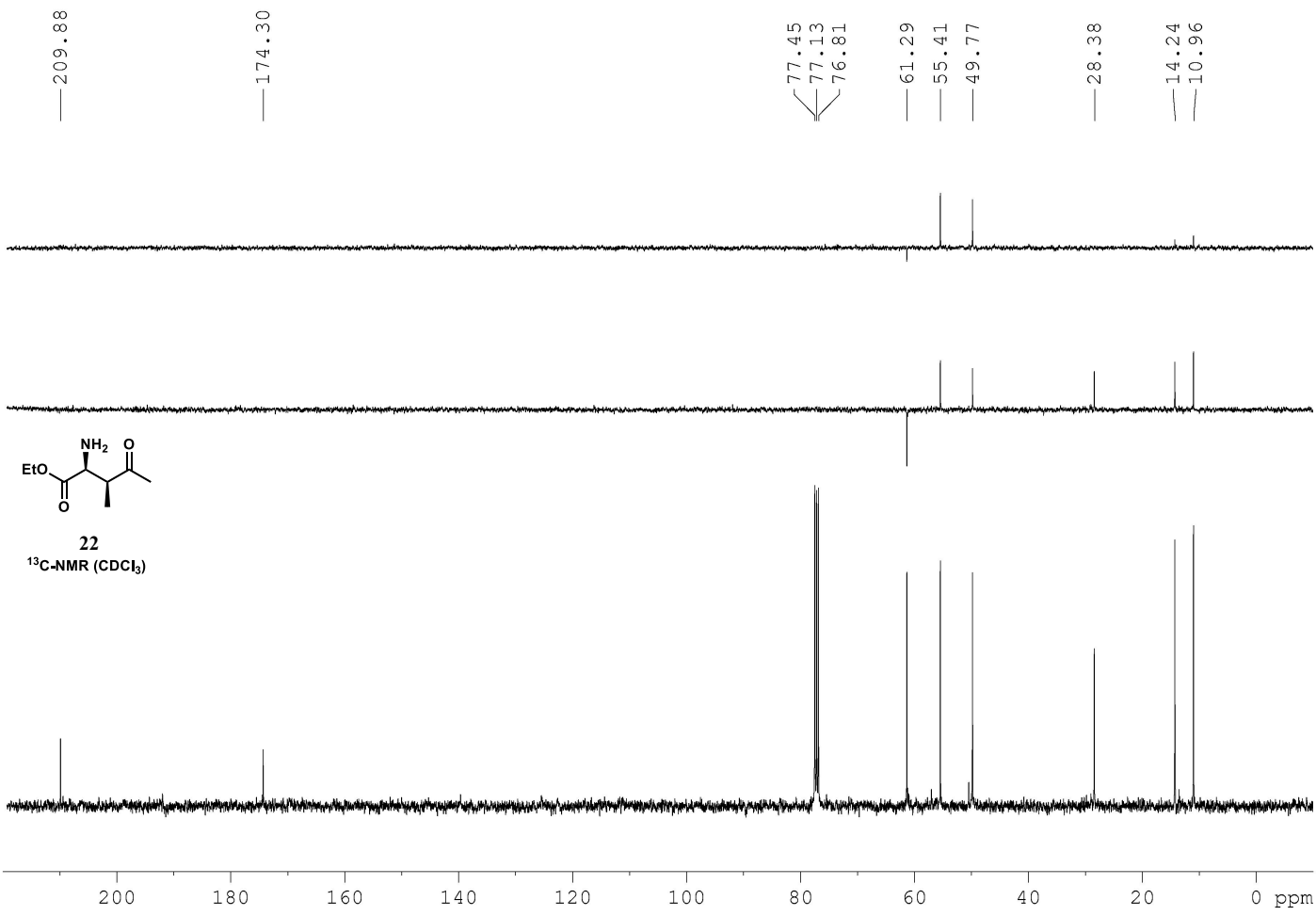


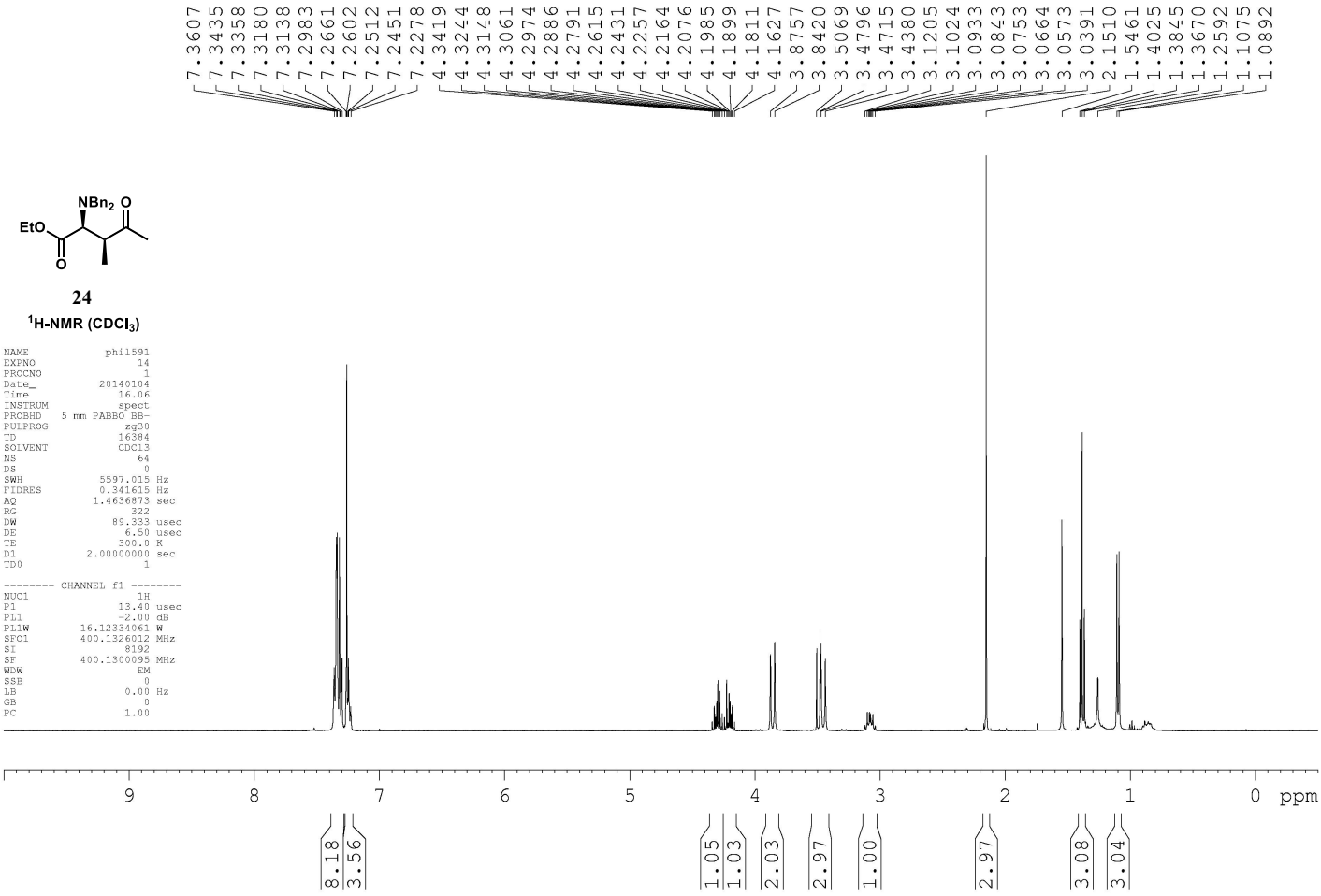

```
NAME            C1773NS55
EXPNO                  30
PROCNO                  1
Date_            20130430
Time                17.44
INSTRUM              spect
PROBHD    5 mm SEI 1H-13
PULPROG       inv4gslrnd
TD                   2048
SOLVENT              MeOD
NS                      8
DS                      8
SWH              2866.972 Hz
FIDRES           1.399889 Hz
AQ               0.3573956 sec
RG                 20642.5
DW                 174.400 usec
DE                    6.00 usec
TE                   300.0 K
D0             0.00000300 sec
D1             1.79396904 sec
D6             0.07000000 sec
D13            0.00000300 sec
D16            0.00010000 sec
IN0            0.00001790 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.50 usec
P2                  21.00 usec
PL1                  5.00 dB
SFO1         500.1216345 MHz

======== CHANNEL f2 ========
NUC2                  13C
P3                  11.20 usec
PL2                  2.00 dB
SFO2         125.7677312 MHz

====== GRADIENT CHANNEL =====
P16                 1000.00 usec
ND0                       2
TD                      128
SFO1              125.7677 MHz
FIDRES         218.226257 Hz
SW                 222.100 ppm
FnMODE                 QF
SI                    2048
SF            500.1200081 MHz
WDW                 SINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                    QF
SF           125.7550840 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
```

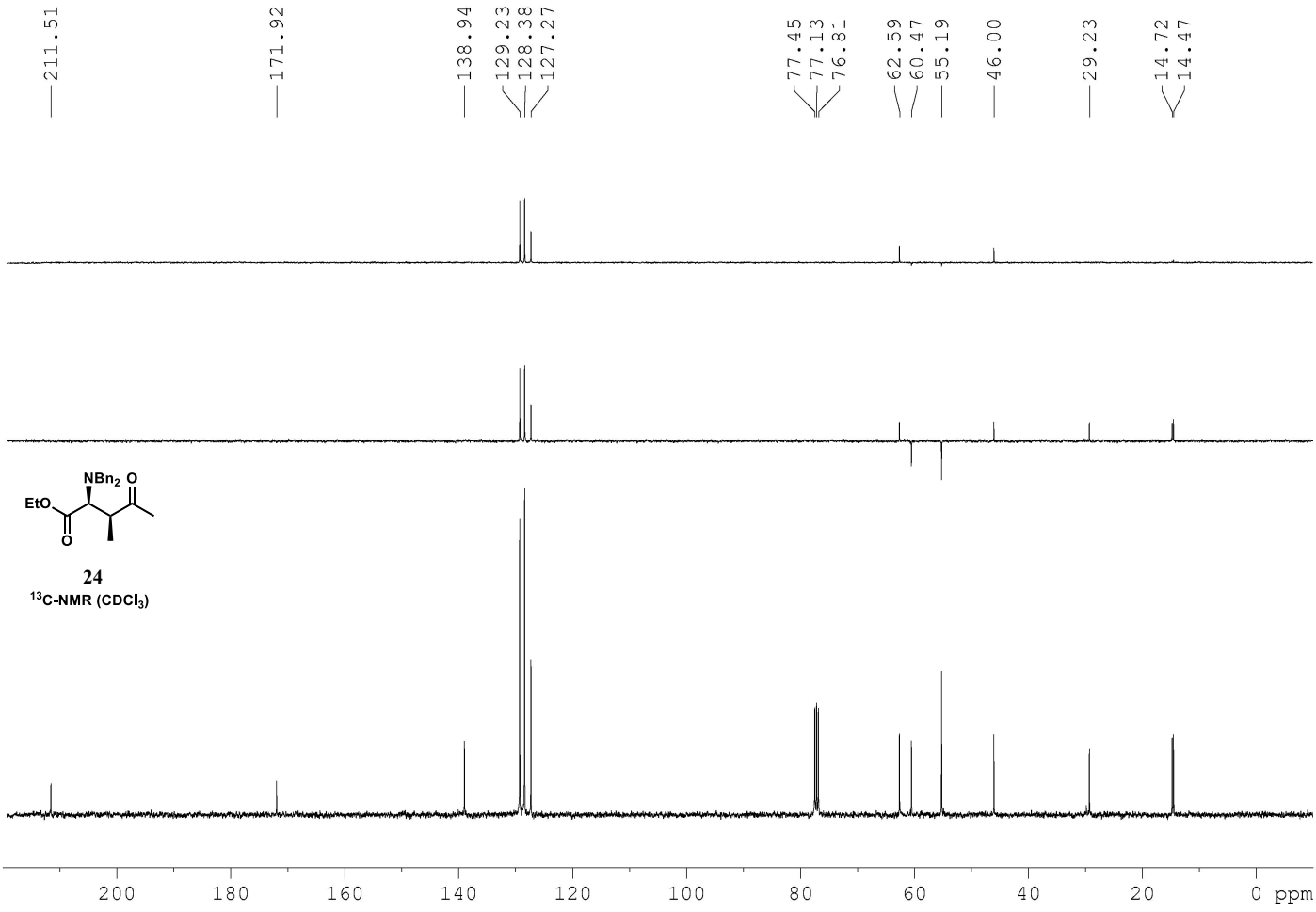

7.2603
6.7802
6.7581
6.6729
6.6509
4.3155
4.3011
4.1900
4.1848
4.1726
4.1675
4.1542
4.1492
4.1369
4.1318
3.7392
3.0691
3.0510
3.0346
3.0182
3.0002
2.2277
1.2634
1.2456
1.2410
1.2234
1.2051
PMP
NH
O
EtO
O
8
1H-NMR (CDCl3)
NAME phil20131213
EXPNO 12
PROCNO 1
Date_ 20131213
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 406
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
SI 8192
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
1.97
1.93
0.99
2.01
3.02
1.00
3.01
6.12

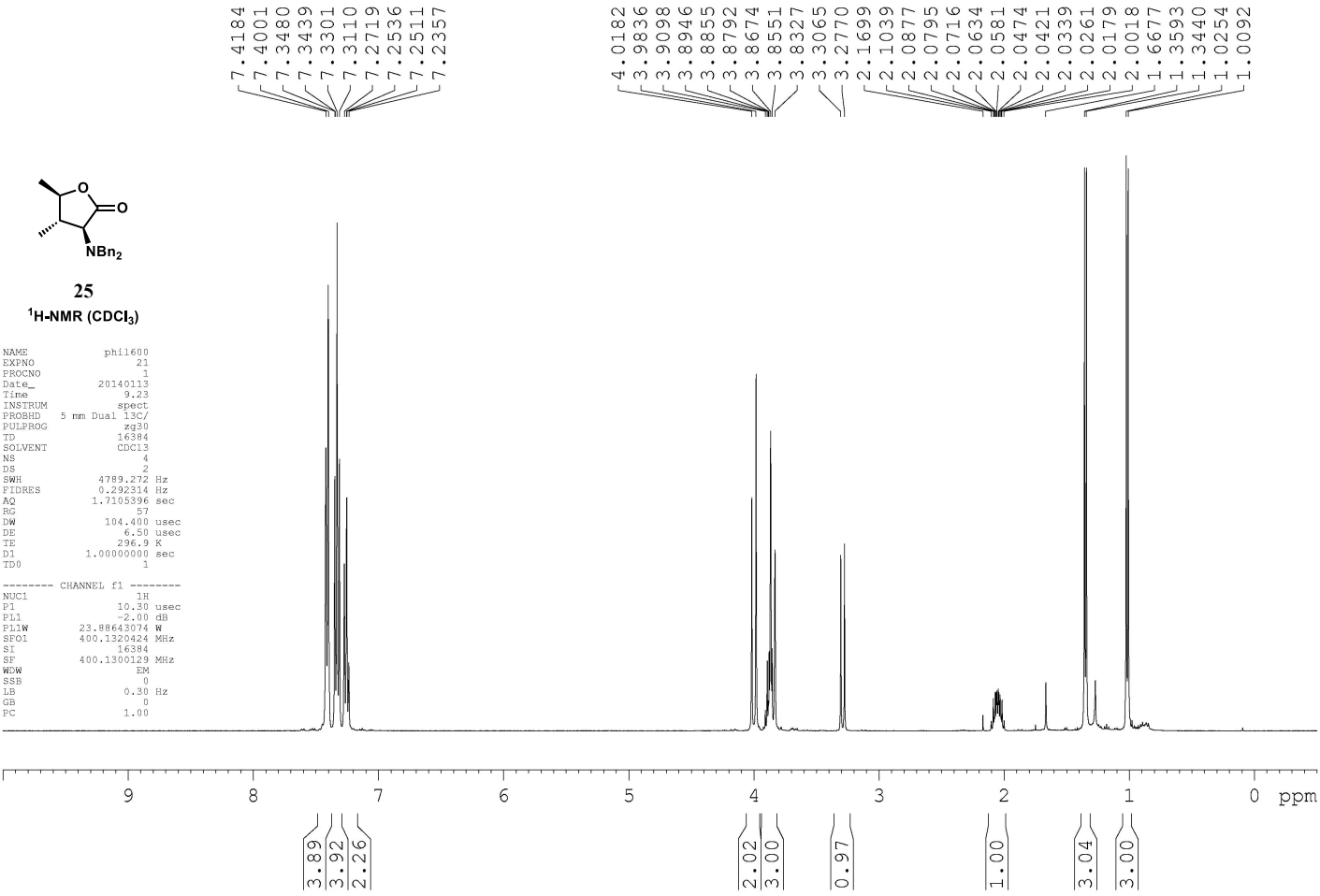

209.26
172.92
153.31
140.91
115.96
114.97
77.38
77.13
76.87
61.46
59.79
55.81
49.40
28.61
14.26
12.38
PMP
NH
O
EtO
O
8
13C-NMR (CDCl3)
200
180
160
140
120
100
80
60
40
20
0 ppm

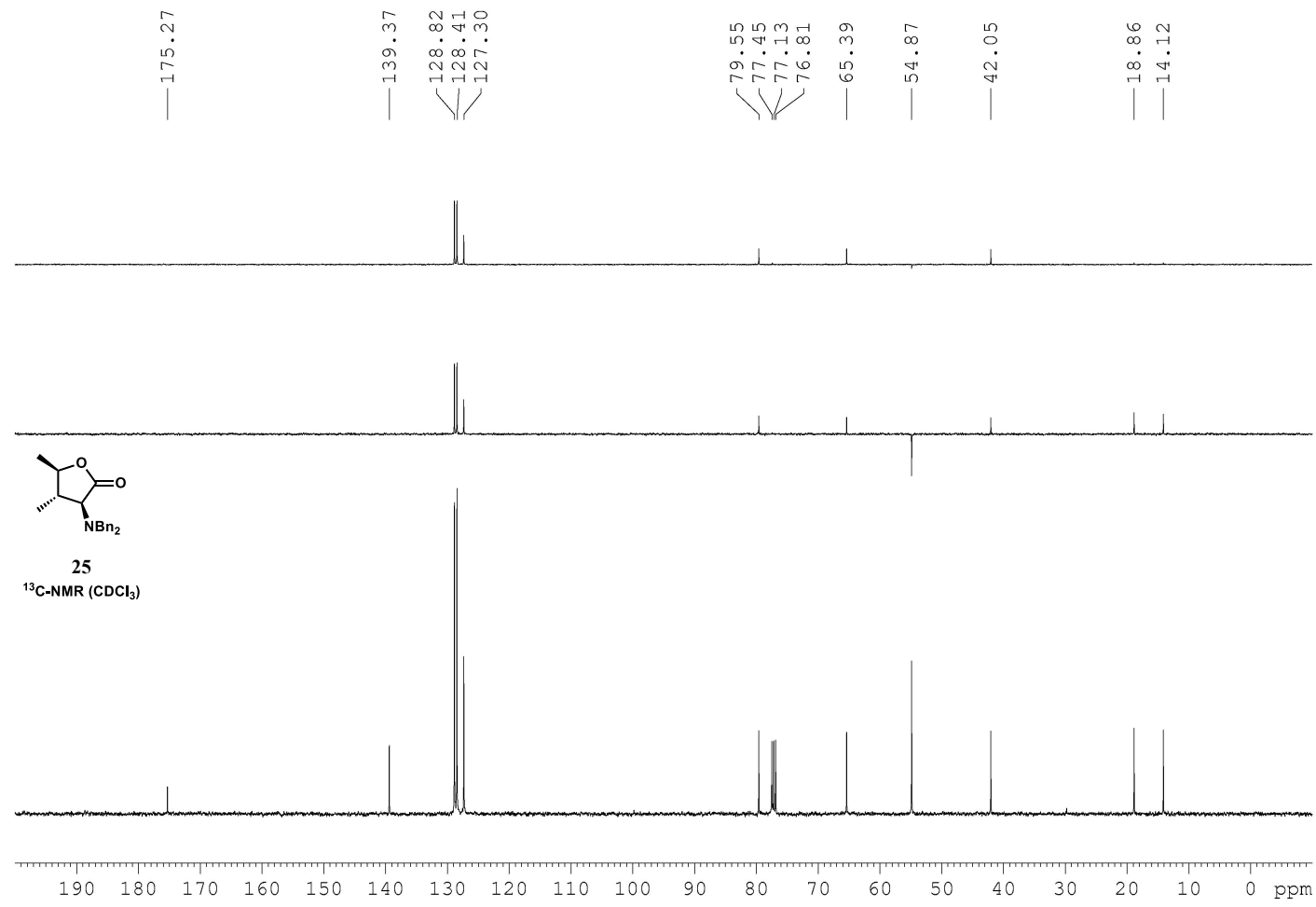

7.2599
4.1900
4.1865
4.1722
4.1687
4.1543
4.1510
4.1364
4.1333
3.8644
3.8523
3.5306
3.5145
2.9429
2.9250
2.9126
2.9072
2.8948
2.8769
2.1946
2.1632
1.5873
1.2669
1.2491
1.2313
1.1682
1.1501
1.1179
1.0999
NH2
O
EtO
O
22
1H-NMR (CDCl3)
NAME phi1594
EXPNO 1
PROCNO 1
Date_ 20140106
Time 15.28
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 90.5
DW 104.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
SI 16384
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
2.13
0.97
0.07
1.00
3.14
3.39
3.10

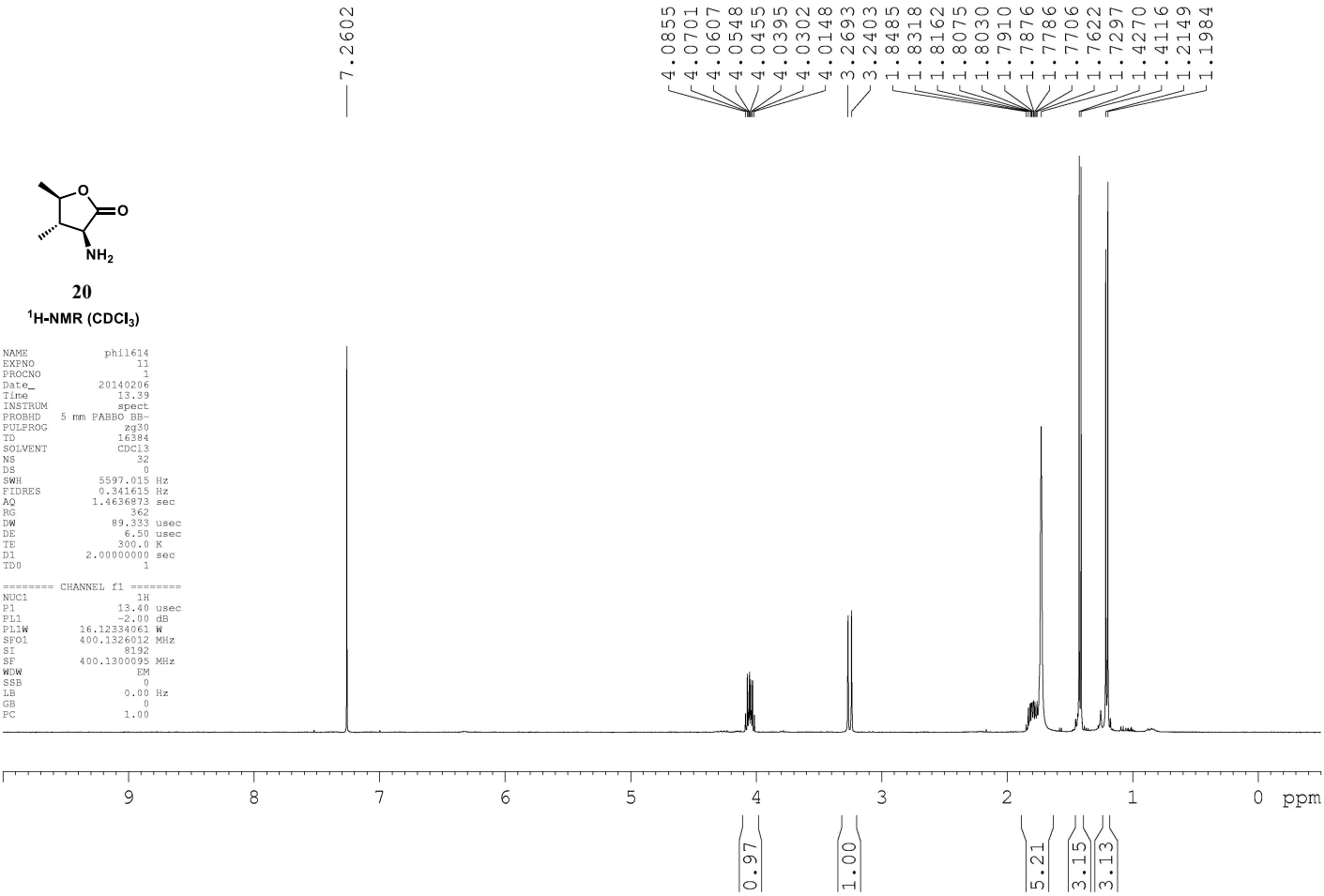

209.88
174.30
77.45
77.13
76.81
61.29
55.41
49.77
28.38
14.24
10.96
NH2
O
EtO
O
22
13C-NMR (CDCl3)
200
180
160
140
120
100
80
60
40
20
0 ppm

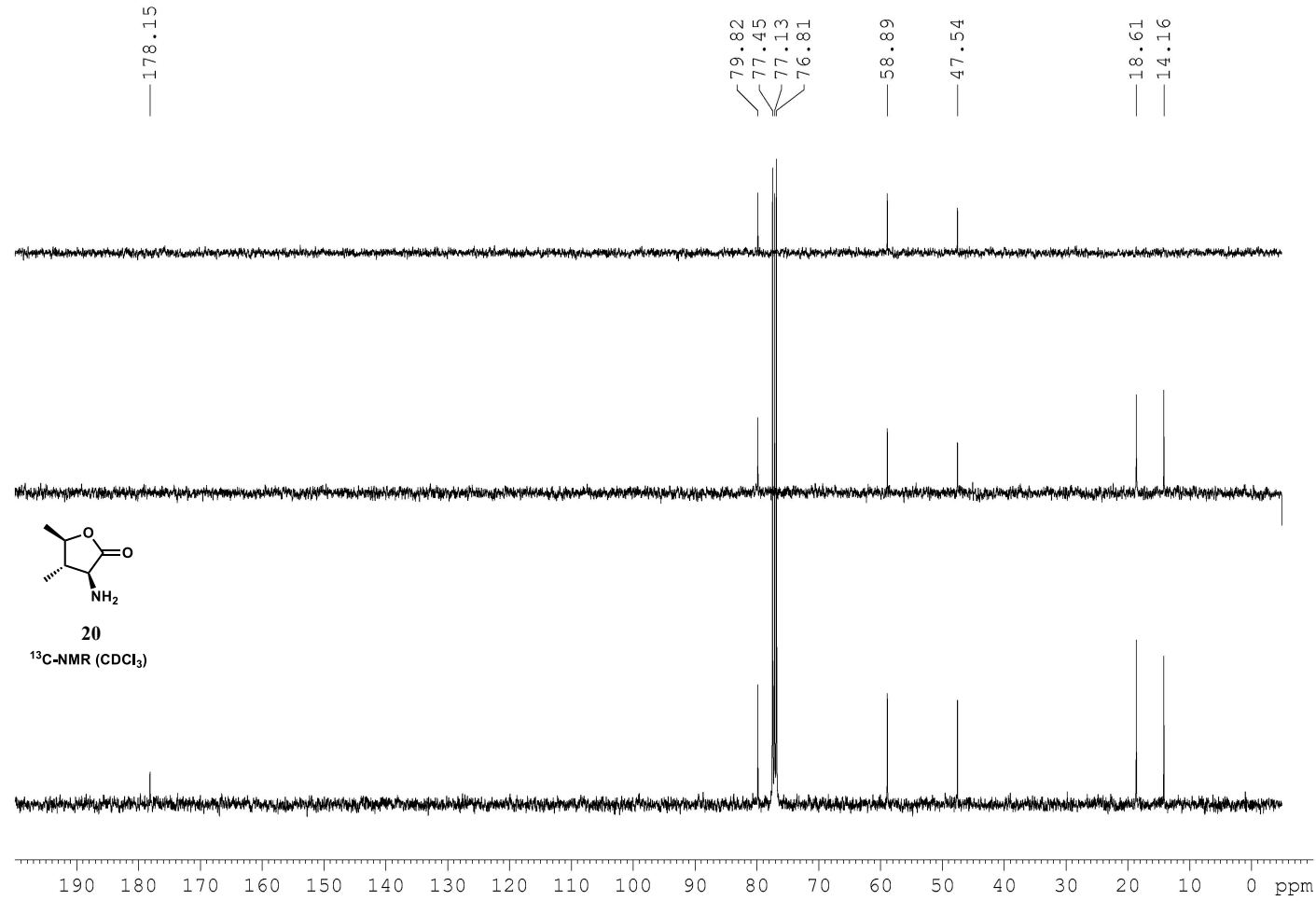

7.3607
7.3435
7.3358
7.3180
7.3138
7.2983
7.2661
7.2602
7.2512
7.2451
7.2278
4.3419
4.3244
4.3148
4.3061
4.2974
4.2886
4.2791
4.2615
4.2431
4.2257
4.2164
4.2076
4.1985
4.1899
4.1811
4.1627
3.8757
3.8420
3.5069
3.4796
3.4715
3.4380
3.1205
3.1024
3.0933
3.0843
3.0753
3.0664
3.0573
3.0391
2.1510
1.5461
1.4025
1.3845
1.3670
1.2592
1.1075
1.0892
NBn2 O
EtO
O
24
1H-NMR (CDCl3)
NAME phi1591
EXPNO 14
PROCNO 1
Date_ 20140104
Time 16.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 322
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
SI 8192
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
8.18
3.56
1.05
1.03
2.03
2.97
1.00
2.97
3.08
3.04

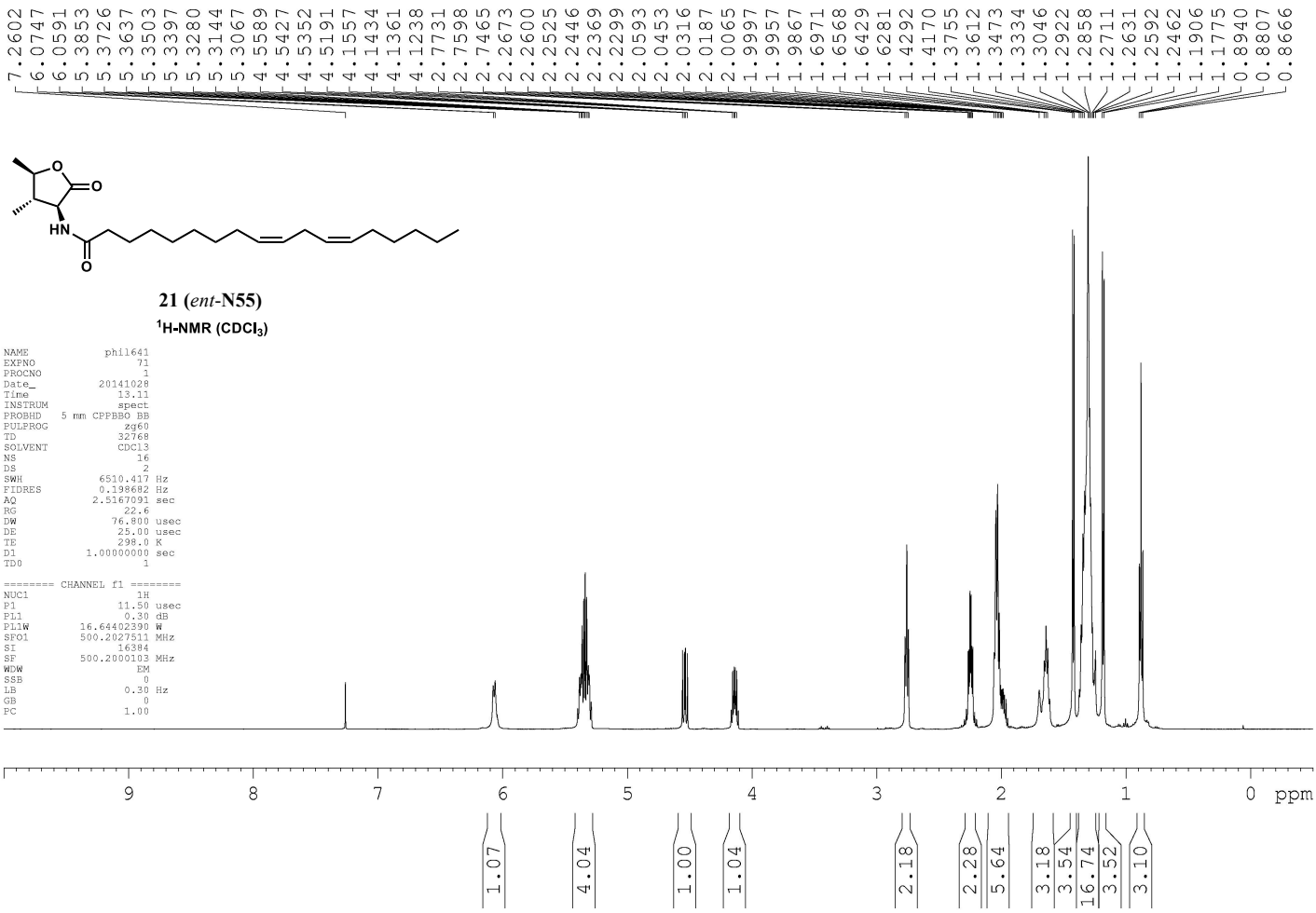

211.51
171.92
138.94
129.23
128.38
127.27
77.45
77.13
76.81
62.59
60.47
55.19
46.00
29.23
14.72
14.47
NBn2 O
EtO
O
24
13C-NMR (CDCl3)
200
180
160
140
120
100
80
60
40
20
0 ppm

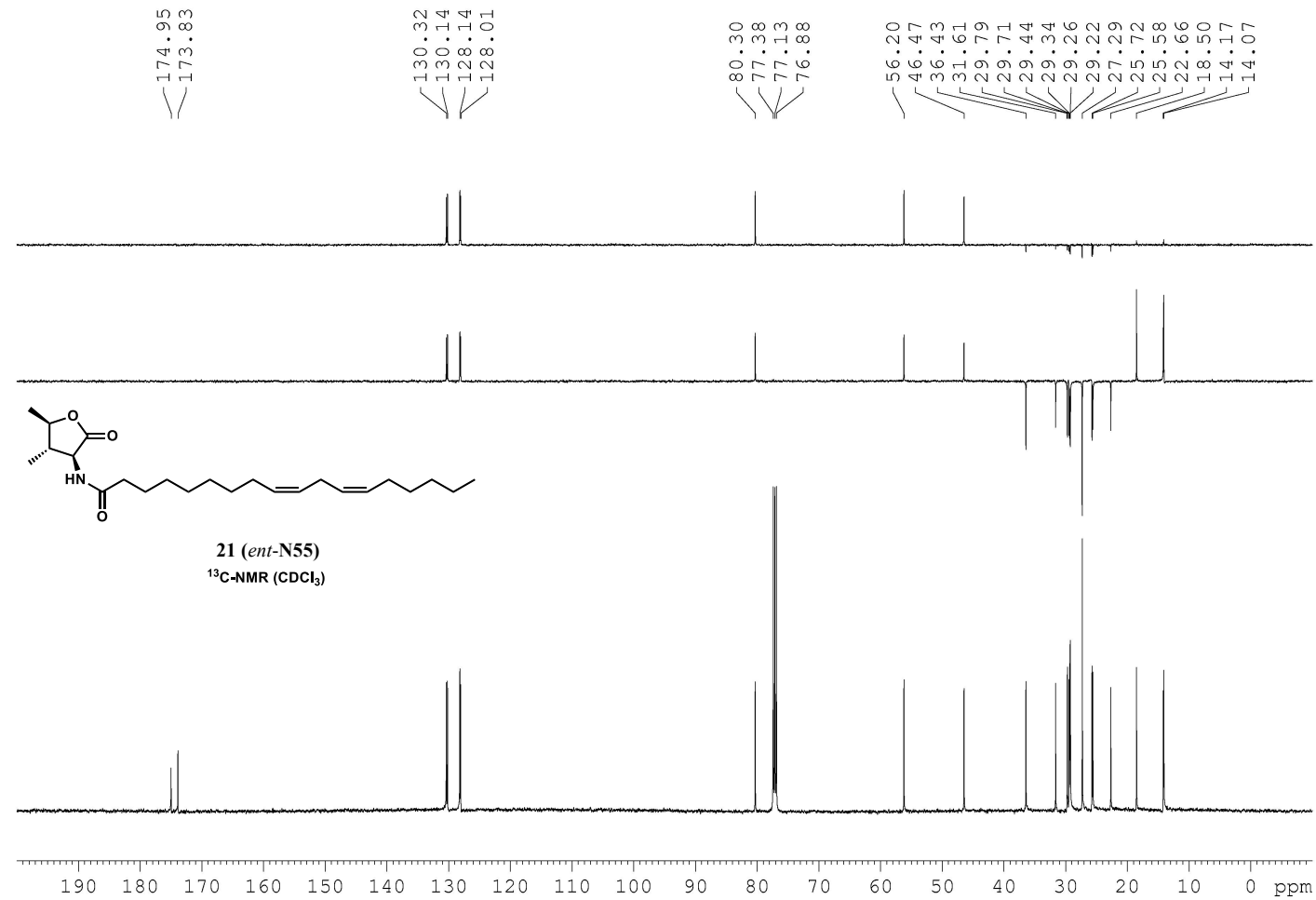

7.4184
7.4001
7.3480
7.3439
7.3301
7.3110
7.2719
7.2536
7.2511
7.2357
4.0182
3.9836
3.9098
3.8946
3.8855
3.8792
3.8674
3.8551
3.8327
3.3065
3.2770
2.1699
2.1039
2.0877
2.0795
2.0716
2.0634
2.0581
2.0474
2.0421
2.0339
2.0261
2.0179
2.0018
1.6677
1.3593
1.3440
1.0254
1.0092
O
O
NBn2
25
1H-NMR (CDCl3)
NAME phi1600
EXPNO 21
PROCNO 1
Date_ 20140113
Time 9.23
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 57
DW 104.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
3.89
3.92
2.26
2.02
3.00
0.97
1.00
3.04
3.00

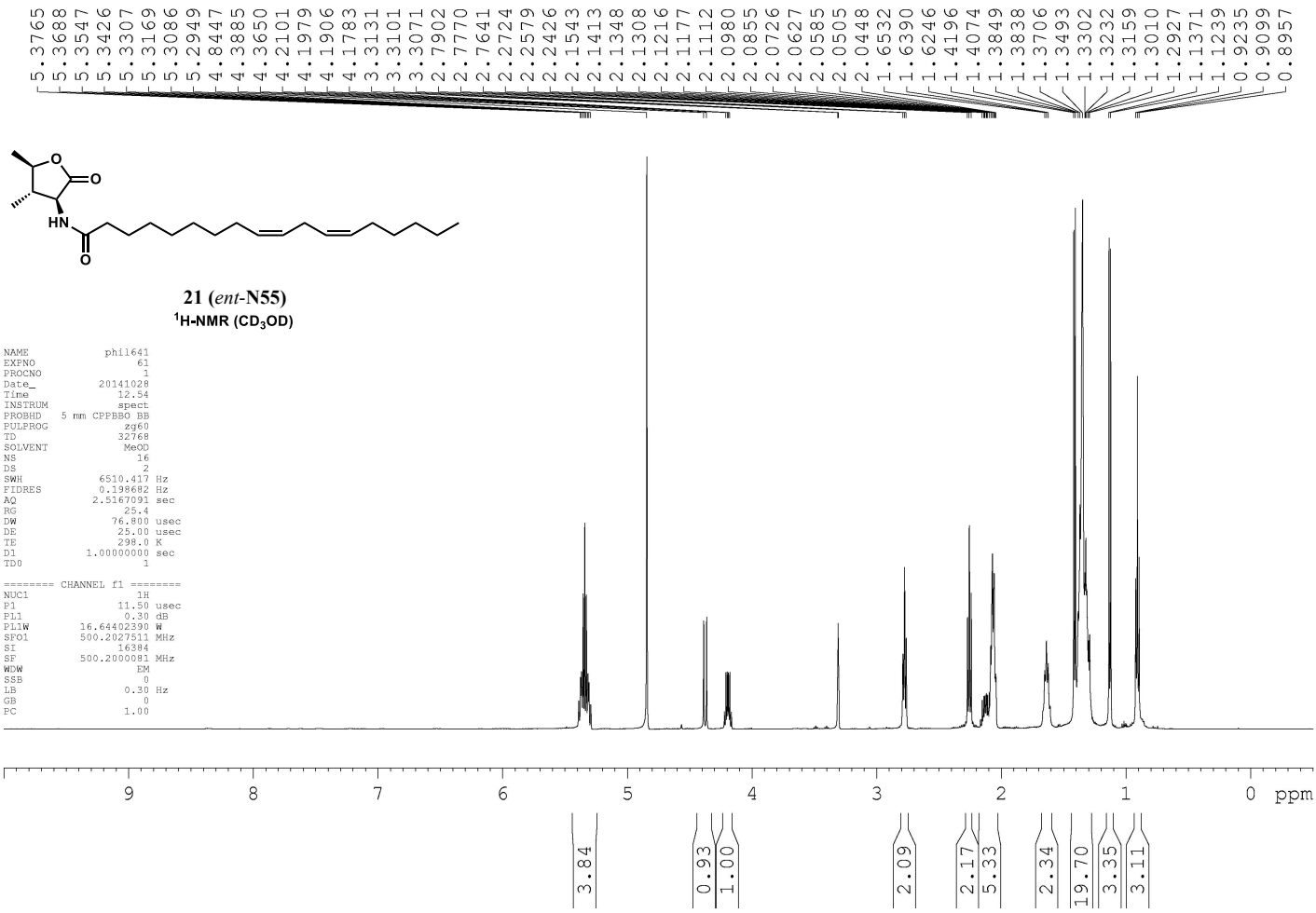

175.27
139.37
128.82
128.41
127.30
79.55
77.45
77.13
76.81
65.39
54.87
42.05
18.86
14.12
O
O
NBn2
25
13C-NMR (CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

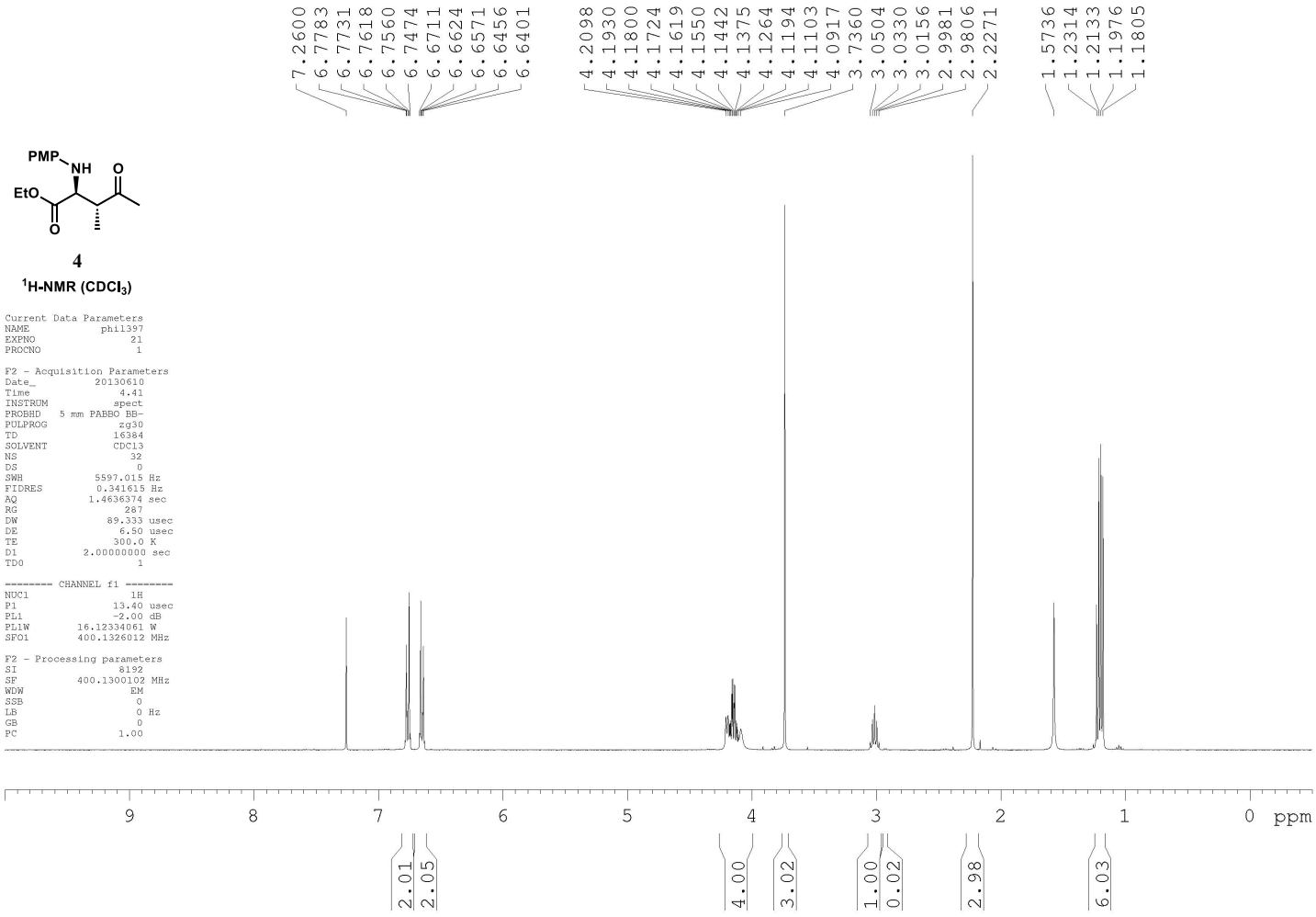

7.2602
4.0855
4.0701
4.0607
4.0548
4.0455
4.0395
4.0302
4.0148
3.2693
3.2403
1.8485
1.8318
1.8162
1.8075
1.8030
1.7910
1.7876
1.7786
1.7706
1.7622
1.7297
1.4270
1.4116
1.2149
1.1984
O
O
NH2
20
1H-NMR (CDCl3)
NAME phi1614
EXPNO 11
PROCNO 1
Date_ 20140206
Time 13.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 362
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
SI 8192
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
0.97
1.00
5.21
3.15
3.13

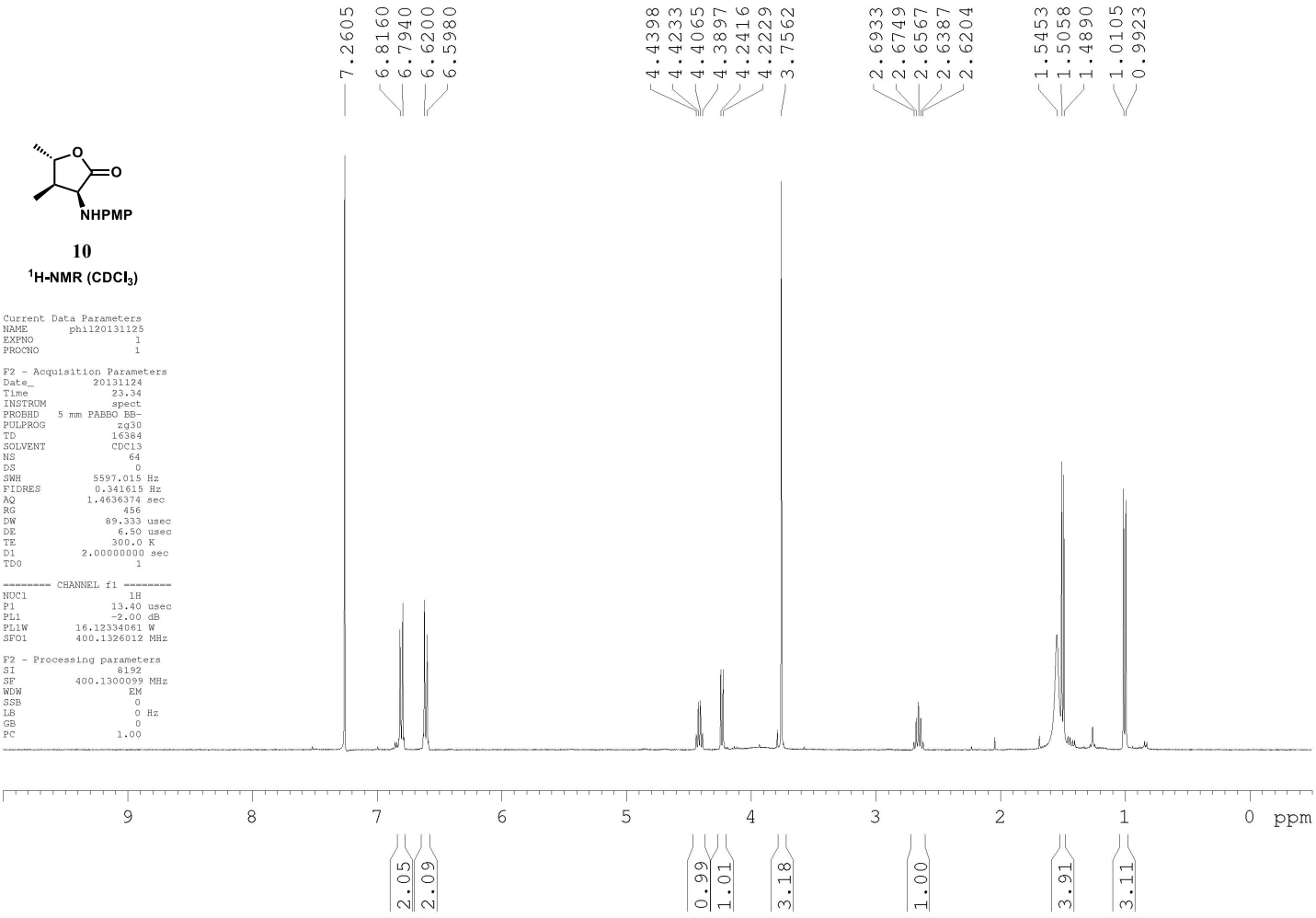

178.15
79.82
77.45
77.13
76.81
58.89
47.54
18.61
14.16
O
O
NH2
20
13C-NMR (CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

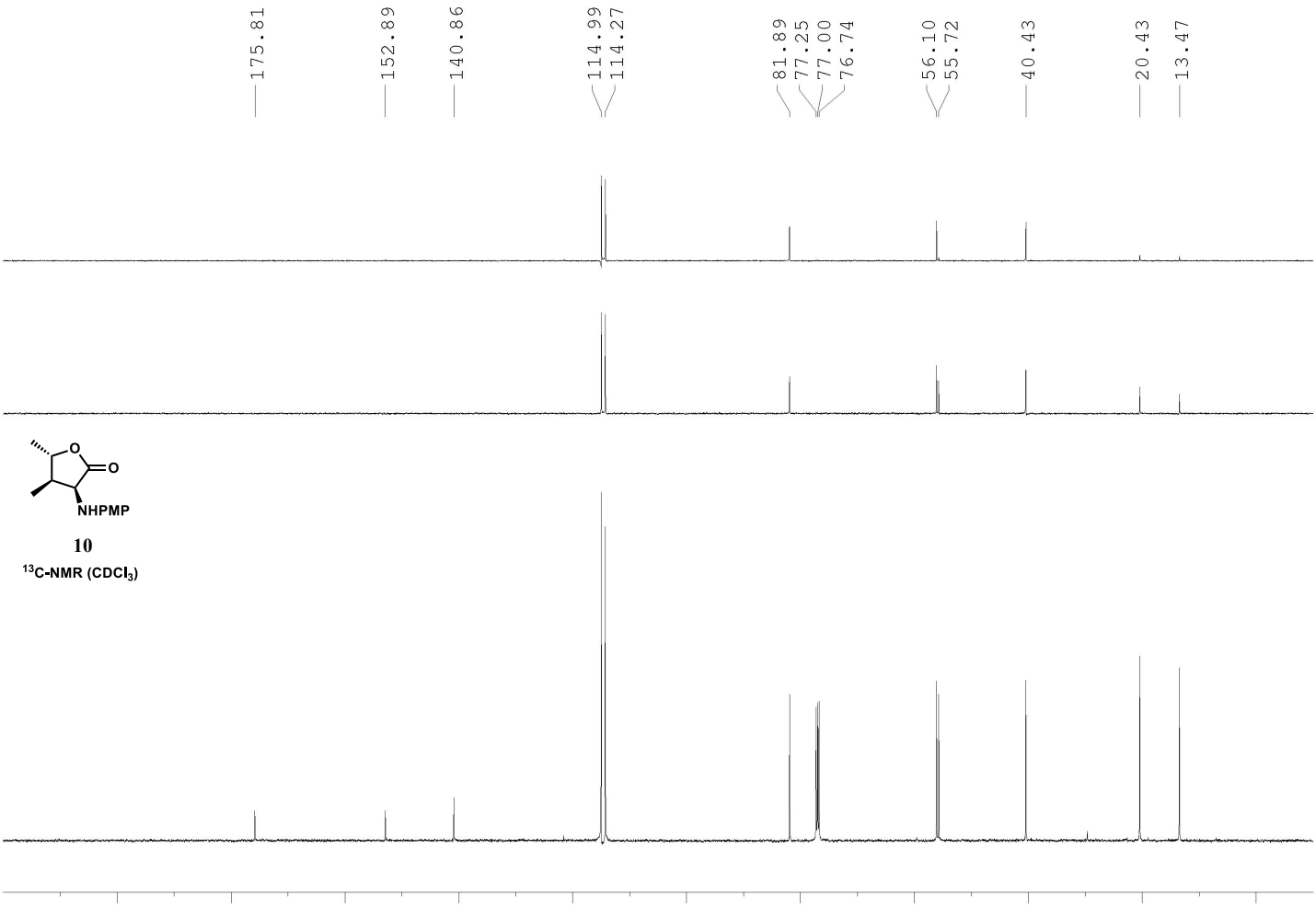

7.2602
6.0747
6.0591
5.3853
5.3726
5.3637
5.3503
5.3397
5.3280
5.3144
5.3067
4.5589
4.5427
4.5352
4.5191
4.1557
4.1434
4.1361
4.1238
2.7731
2.7598
2.7465
2.2673
2.2600
2.2525
2.2446
2.2369
2.2299
2.0593
2.0453
2.0316
2.0187
2.0065
1.9997
1.9957
1.9867
1.6971
1.6568
1.6429
1.6281
1.4292
1.4170
1.3755
1.3612
1.3473
1.3334
1.3046
1.2922
1.2858
1.2711
1.2631
1.2592
1.2462
1.1906
1.1775
0.8940
0.8807
0.8666
21 (ent-N55)
1H-NMR (CDCl3)
NAME phil641
EXPNO 71
PROCNO 1
Date_ 20141028
Time 13.11
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg60
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5167091 sec
RG 22.6
DW 76.800 usec
DE 25.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 0.30 dB
PL1W 16.64402390 W
SFO1 500.2027511 MHz
SI 16384
SF 500.2000103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.07 4.04 1.00 1.04 2.18 2.28 5.64 3.18 3.54 16.74 3.52 3.10

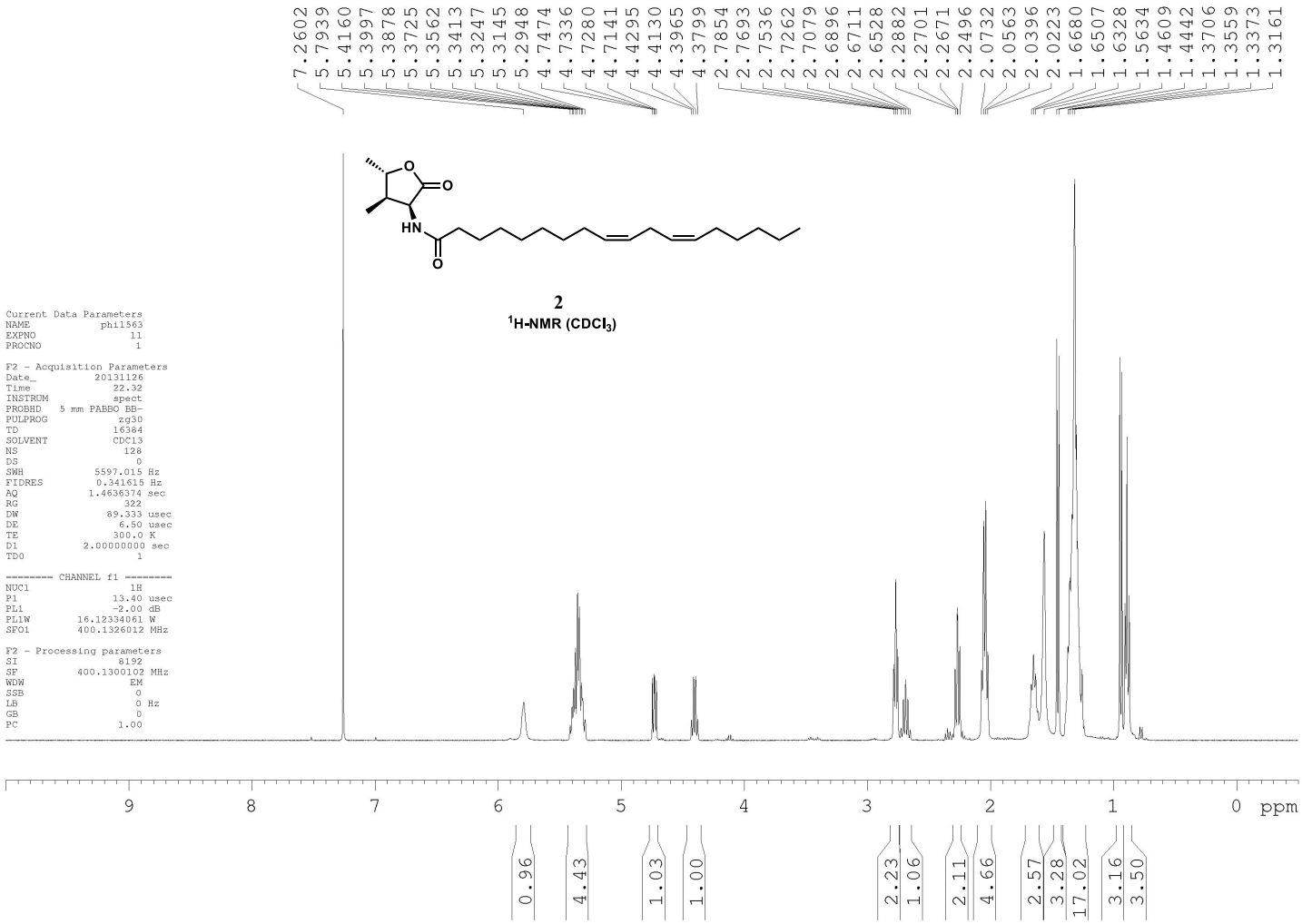

174.95
173.83
130.32
130.14
128.14
128.01
80.30
77.38
77.13
76.88
56.20
46.47
36.43
31.61
29.79
29.71
29.44
29.34
29.26
29.22
27.29
25.72
25.58
22.66
18.50
14.17
14.07
21 (ent-N55)
13C-NMR (CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

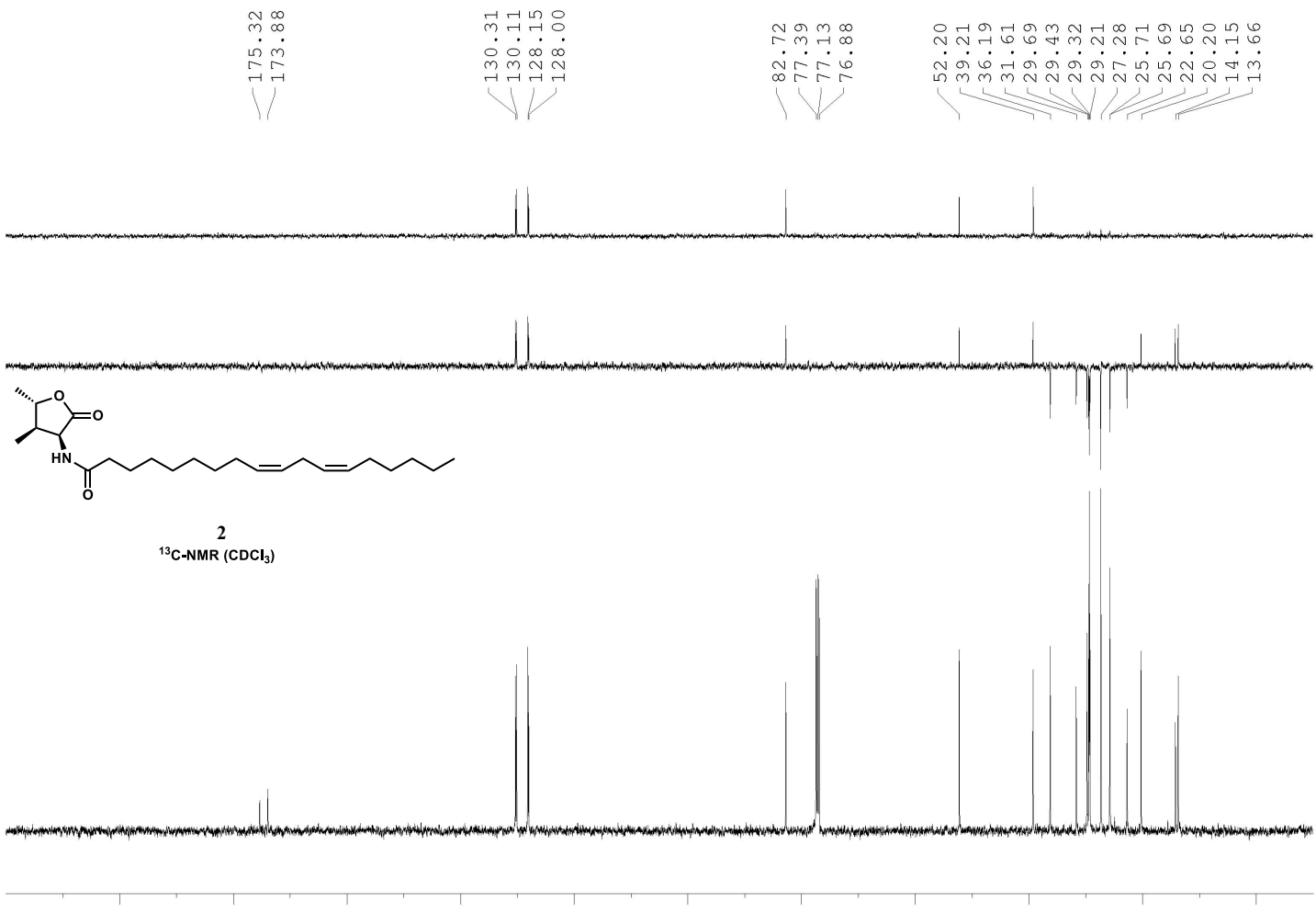

5.3765
5.3688
5.3547
5.3426
5.3307
5.3169
5.3086
5.2949
4.8447
4.3885
4.3650
4.2101
4.1979
4.1906
4.1783
3.3131
3.3101
3.3071
2.7902
2.7770
2.7641
2.2724
2.2579
2.2426
2.1543
2.1413
2.1348
2.1308
2.1216
2.1177
2.1112
2.0980
2.0855
2.0726
2.0627
2.0585
2.0505
2.0448
1.6532
1.6390
1.6246
1.4196
1.4074
1.3849
1.3838
1.3706
1.3493
1.3302
1.3232
1.3159
1.3010
1.2927
1.1371
1.1239
0.9235
0.9099
0.8957
O
O
HN
O
21 (ent-N55)
1H-NMR (CD3OD)
NAME phil641
EXPNO 61
PROCNO 1
Date_ 20141028
Time 12.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg60
TD 32768
SOLVENT MeOD
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5167091 sec
RG 25.4
DW 76.800 usec
DE 25.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 0.30 dB
PL1W 16.64402390 W
SFO1 500.2027511 MHz
SI 16384
SF 500.2000081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
3.84
0.93
1.00
2.09
2.17
5.33
2.34
19.70
3.35
3.11
176.49
176.22
130.94
130.88
129.10
129.05
81.42
57.53
49.51
49.34
49.17
49.00
48.83
48.66
48.49
45.60
36.90
32.66
30.72
30.48
30.32
30.25
30.16
28.17
26.84
26.54
23.63
18.82
14.44
13.98
O
O
HN
O
21 (ent-N55)
13C-NMR (CD3OD)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

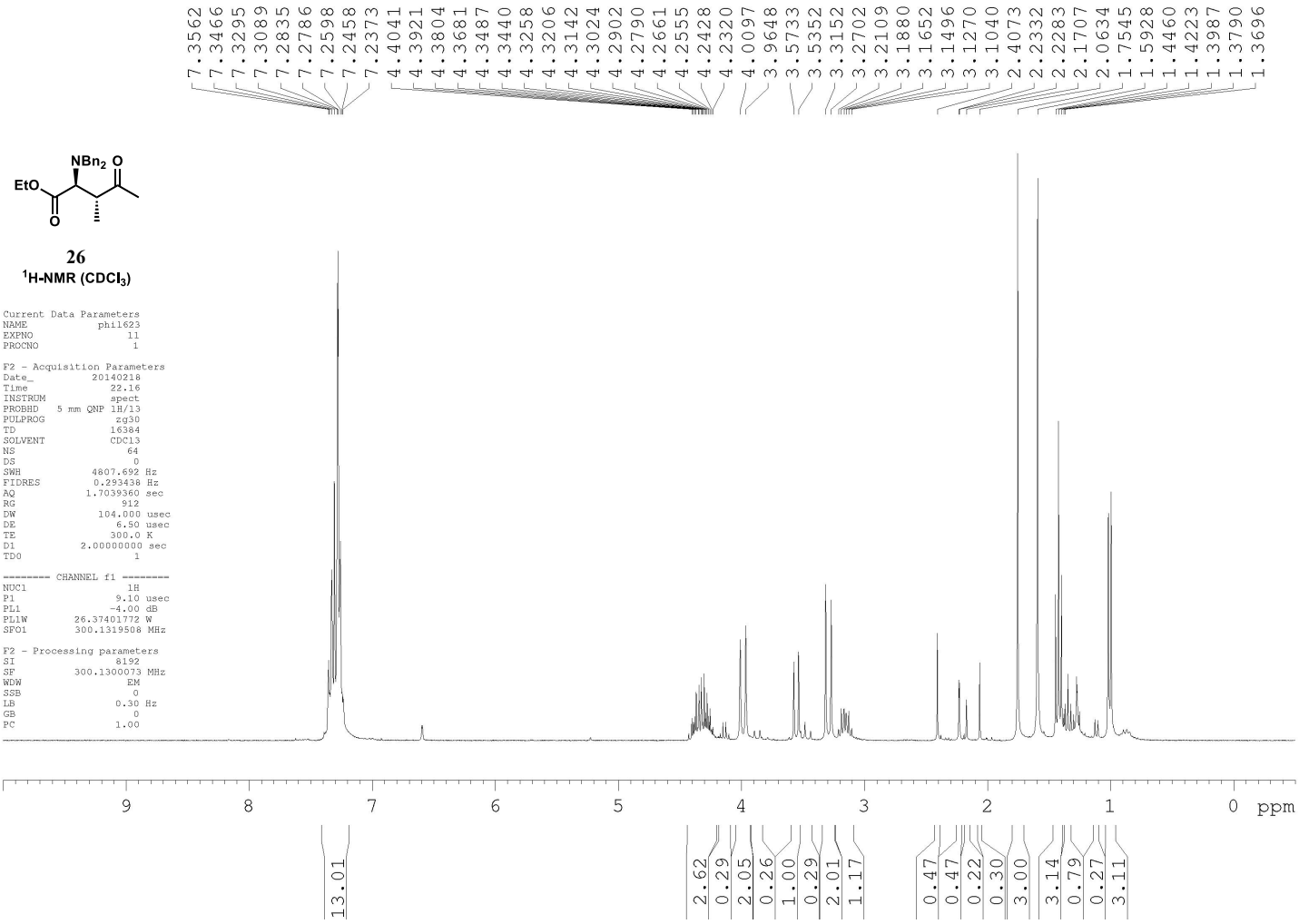

7.2600
6.7783
6.7731
6.7618
6.7560
6.7474
6.6711
6.6624
6.6571
6.6456
6.6401
4.2098
4.1930
4.1800
4.1724
4.1619
4.1550
4.1442
4.1375
4.1264
4.1194
4.1103
4.0917
3.7360
3.0504
3.0330
3.0156
2.9981
2.9806
2.2271
1.5736
1.2314
1.2133
1.1976
1.1805
PMP
NH
O
EtO
O
4
1H-NMR (CDCl3)
Current Data Parameters
NAME phil397
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130610
Time 4.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636374 sec
RG 287
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
2.01
2.05
4.00
3.02
1.00
0.02
2.98
6.03
209.62
172.65
153.23
140.81
115.91
114.96
77.45
77.13
76.81
61.40
60.64
55.79
49.49
28.73
14.26
13.13
PMP
NH
O
EtO
O
4
13C-NMR (CDCl3)
200
180
160
140
120
100
80
60
40
20
0 ppm

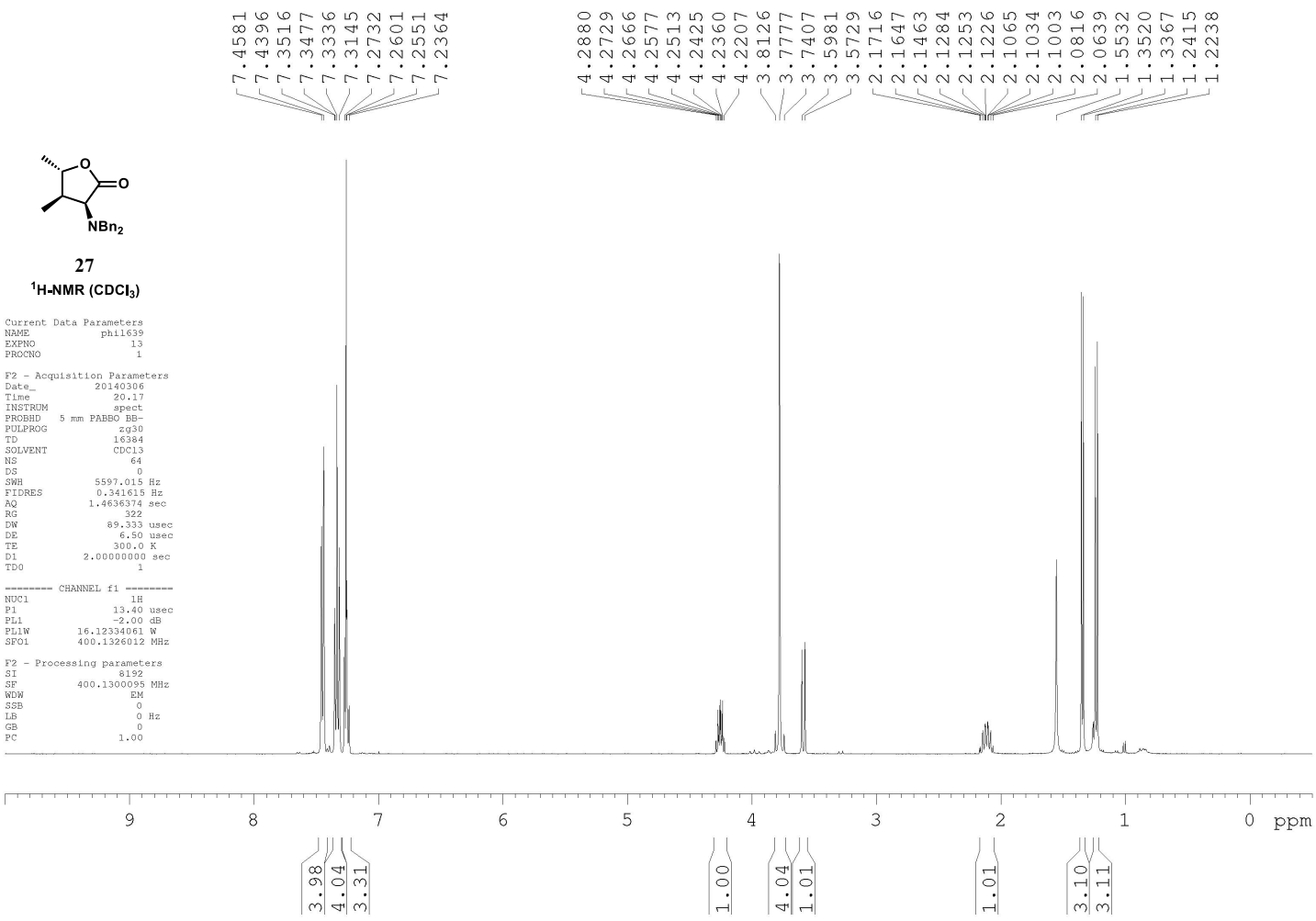

7.2605
6.8160
6.7940
6.6200
6.5980
4.4398
4.4233
4.4065
4.3897
4.2416
4.2229
3.7562
2.6933
2.6749
2.6567
2.6387
2.6204
1.5453
1.5058
1.4890
1.0105
0.9923
O
O
NHPMP
10
1H-NMR (CDCl3)
Current Data Parameters
NAME phi120131125
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131124
Time 23.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636374 sec
RG 456
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
2.05
2.09
0.99
1.01
3.18
1.00
3.91
3.11


175.81
152.89
140.86
114.99
114.27
81.89
77.25
77.00
76.74
56.10
55.72
40.43
20.43
13.47
O
O
NHPMP
10
13C-NMR (CDCl3)
200
180
160
140
120
100
80
60
40
20
0
ppm


7.2602
5.7939
5.4160
5.3997
5.3878
5.3725
5.3562
5.3413
5.3247
5.3145
5.2948
4.7474
4.7336
4.7280
4.7141
4.4295
4.4130
4.3965
4.3799
2.7854
2.7693
2.7536
2.7262
2.7079
2.6896
2.6711
2.6528
2.2882
2.2701
2.2671
2.2496
2.0732
2.0563
2.0396
2.0223
1.6680
1.6507
1.6328
1.5634
1.4609
1.4442
1.3706
1.3559
1.3373
1.3161
O
O
HN
O
2
1H-NMR (CDCl3)
Current Data Parameters
NAME phil563
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131126
Time 22.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 128
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636374 sec
RG 322
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
0.96
4.43
1.03
1.00
2.23
1.06
2.11
4.66
2.57
3.28
17.02
3.16
3.50

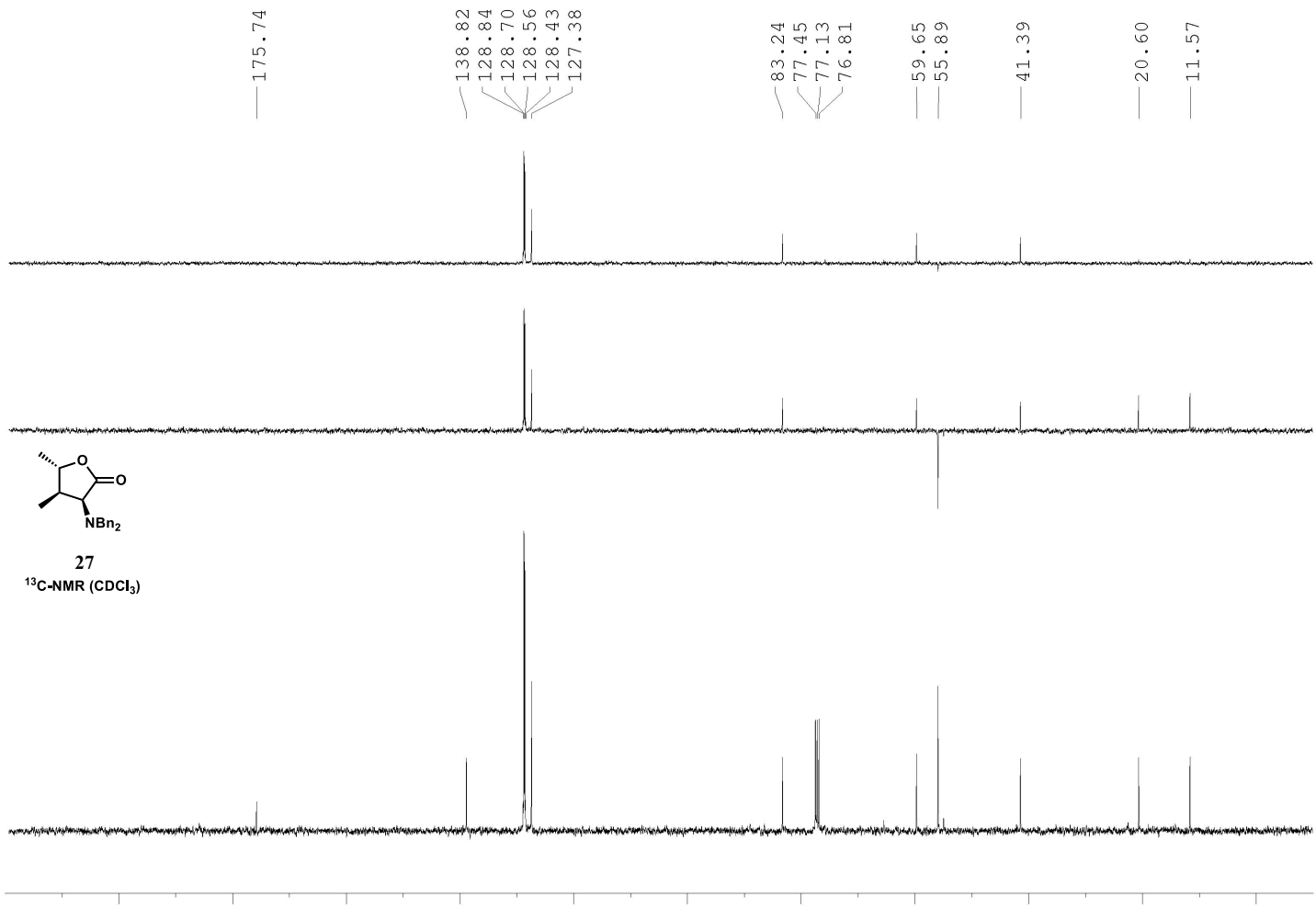

175.32
173.88
130.31
130.11
128.15
128.00
82.72
77.39
77.13
76.88
52.20
39.21
36.19
31.61
29.69
29.43
29.32
29.21
27.28
25.71
25.69
22.65
20.20
14.15
13.66
O
O
HN
O
2
13C-NMR (CDCl3)
200
180
160
140
120
100
80
60
40
20
0
ppm


7.3562
7.3466
7.3295
7.3089
7.2835
7.2786
7.2598
7.2458
7.2373
4.4041
4.3921
4.3804
4.3681
4.3487
4.3440
4.3258
4.3206
4.3142
4.3024
4.2902
4.2790
4.2661
4.2555
4.2428
4.2320
4.0097
3.9648
3.5733
3.5352
3.3152
3.2702
3.2109
3.1880
3.1652
3.1496
3.1270
3.1040
2.4073
2.2332
2.2283
2.1707
2.0634
1.7545
1.5928
1.4460
1.4223
1.3987
1.3790
1.3696
NBn2 O
EtO
O
26
1H-NMR (CDCl3)
Current Data Parameters
NAME phi1623
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140218
Time 22.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 4807.692 Hz
FIDRES 0.293438 Hz
AQ 1.7039360 sec
RG 912
DW 104.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.10 usec
PL1 -4.00 dB
PL1W 26.37401772 W
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 8192
SF 300.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
13.01
2.62
0.29
2.05
0.26
1.00
0.29
2.01
1.17
0.47
0.47
0.22
0.30
3.00
3.14
0.79
0.27
3.11
209.65
169.84
138.65
129.53
128.30
127.32
77.45
77.13
76.81
64.14
60.53
54.72
47.56
25.84
14.79
14.27
NBn2 O
EtO
O
26
13C-NMR (CDCl3)
200
180
160
140
120
100
80
60
40
20
0 ppm



27

^{1}H-NMR ($CDCl_3$)



27

^{13}C-NMR ($CDCl_3$)