Supporting information

Antitumor Agents 274. A New Synthetic Strategy for E-ring SAR Study of Antofine and Cryptopleurine Analogs

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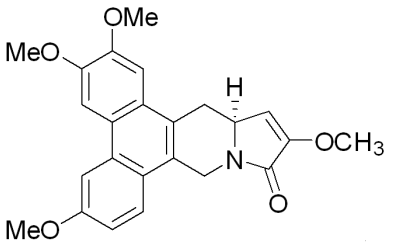
Contents

yxm-JOC-15

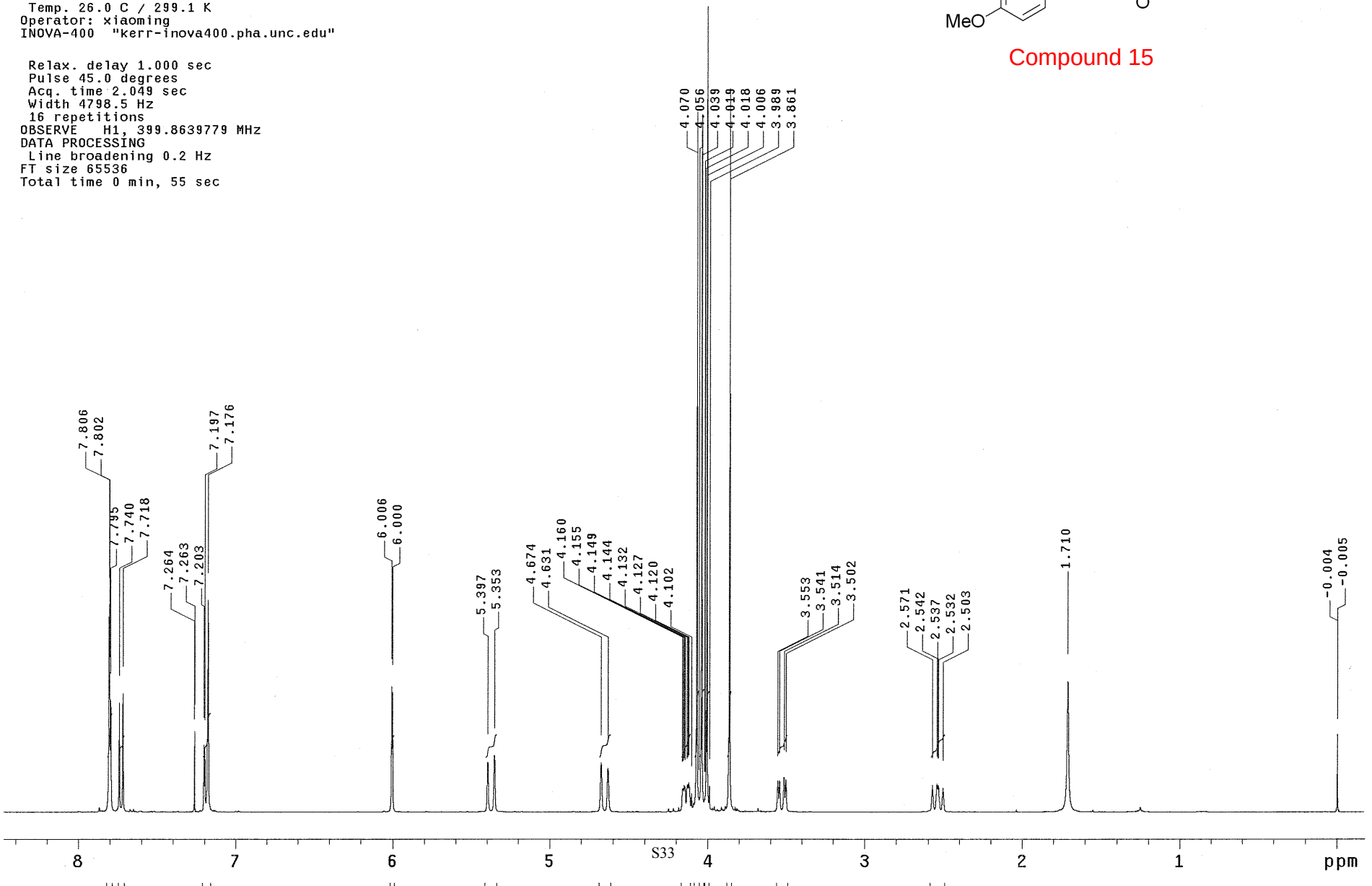
File: Proton

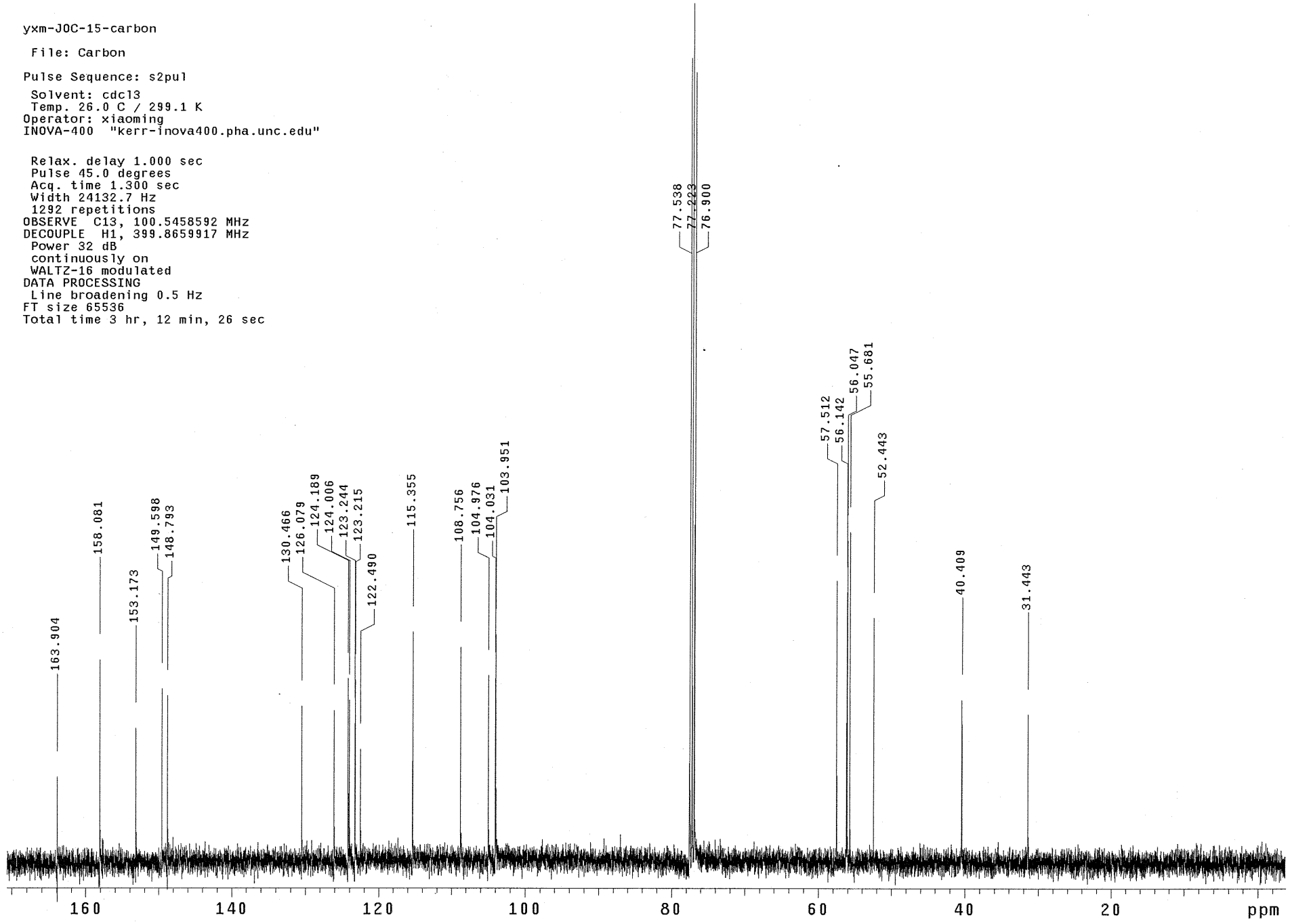
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: xiaoming
INOVA-400 "kerr-inova400.pha.unc.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 4798.5 Hz
16 repetitions
OBSERVE H1, 399.8639779 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec



Compound 15




yxm-JOC-15-carbon
File: Carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: xiaoming
INOVA-400 "kerr-inova400.pha.unc.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24132.7 Hz
1292 repetitions
OBSERVE C13, 100.5458592 MHz
DECOUPLE H1, 399.8659917 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 12 min, 26 sec
163.904
158.081
153.173
149.598
148.793
130.466
126.079
124.189
124.006
123.244
123.215
122.490
115.355
108.756
104.976
104.031
103.951
77.538
77.223
76.900
57.512
56.142
56.047
55.681
52.443
40.409
31.443
160
140
120
100
80
60
40
20
ppm

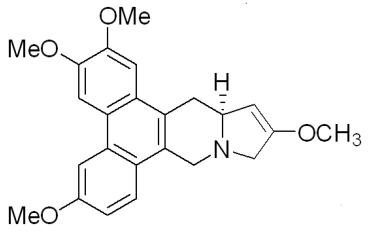
yxm-JOC-16

File: Proton

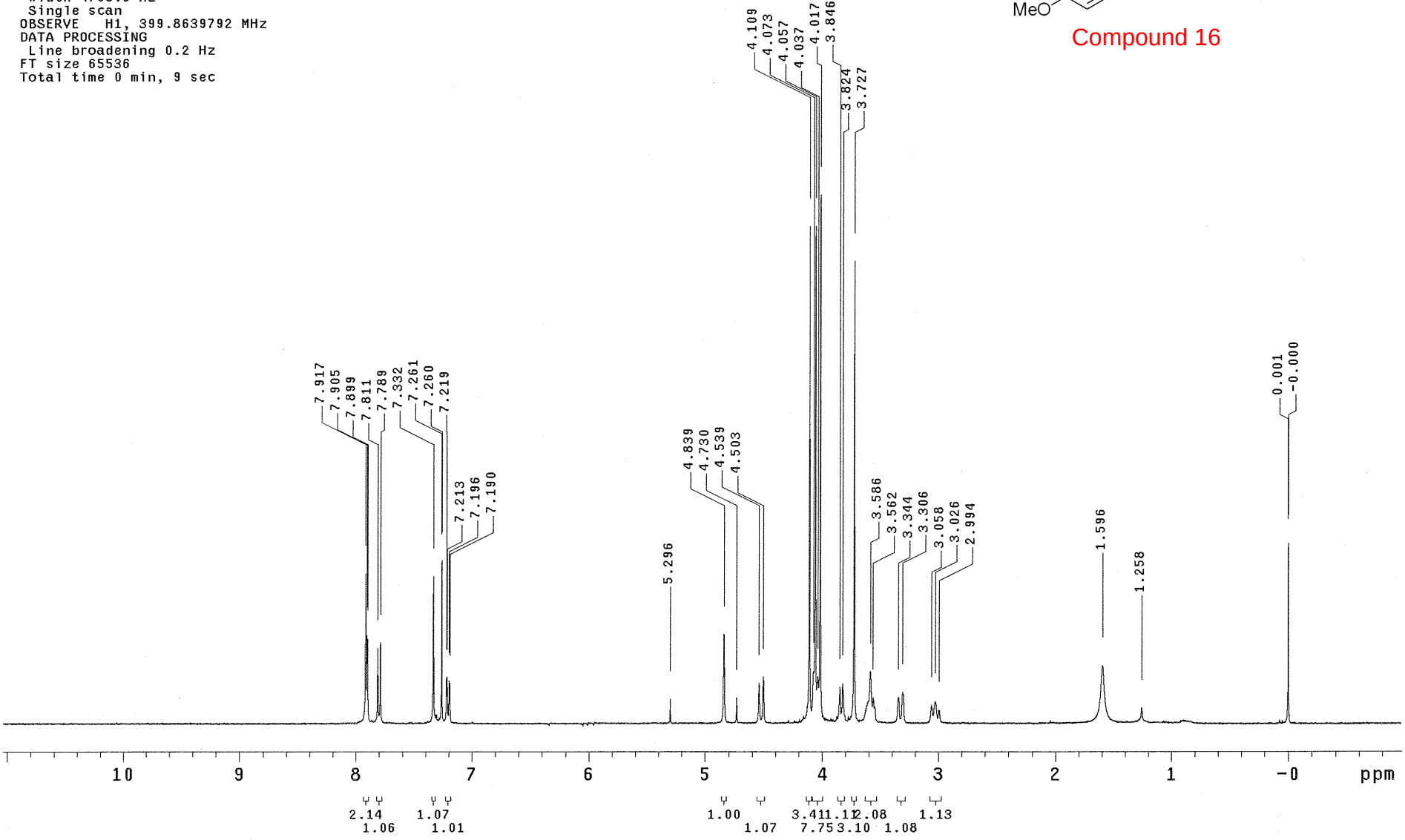
Pulse Sequence: s2pul

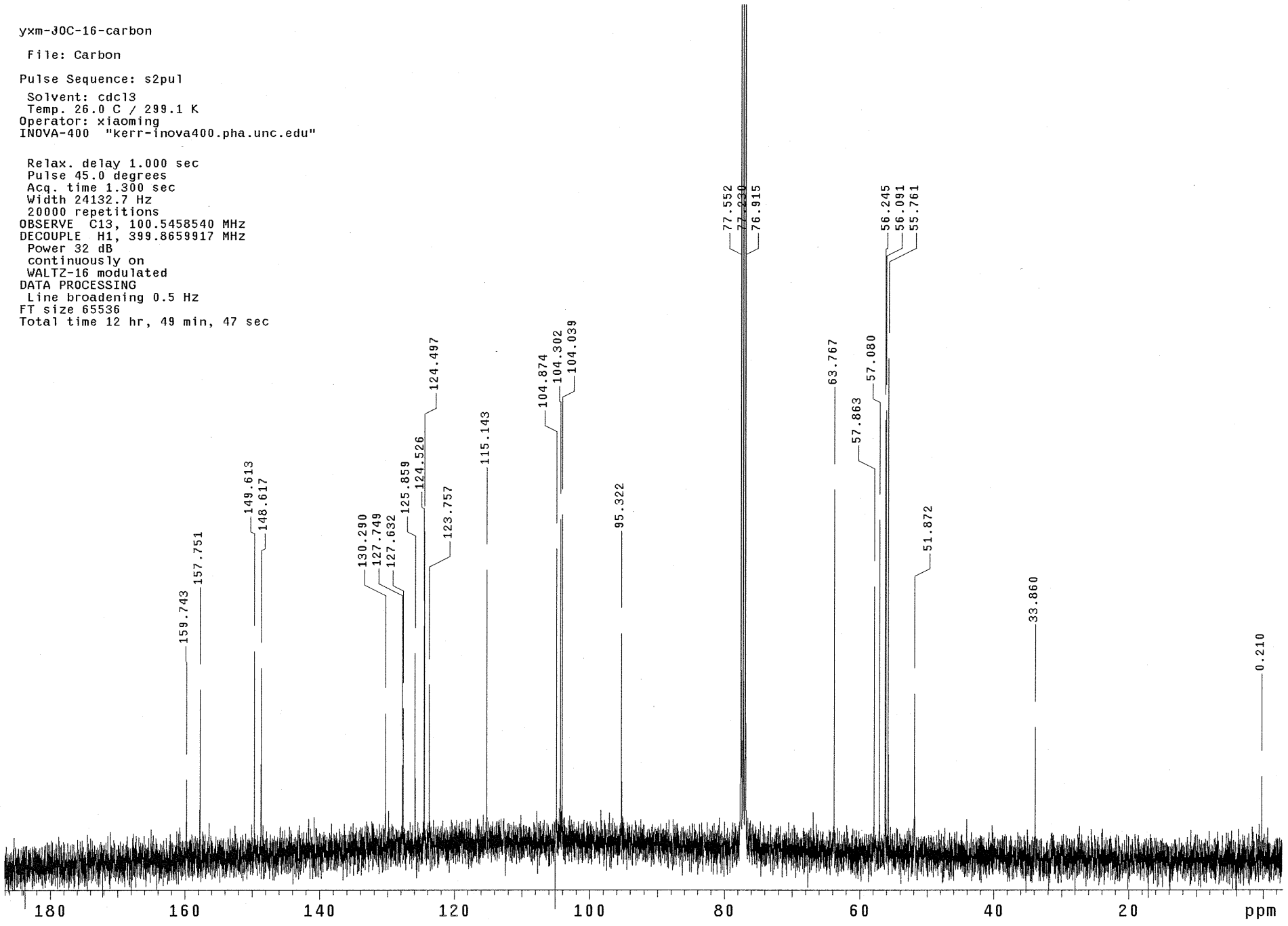
Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: xiaoming
INOVA-400 "kerr-inova400.pha.unc.edu"

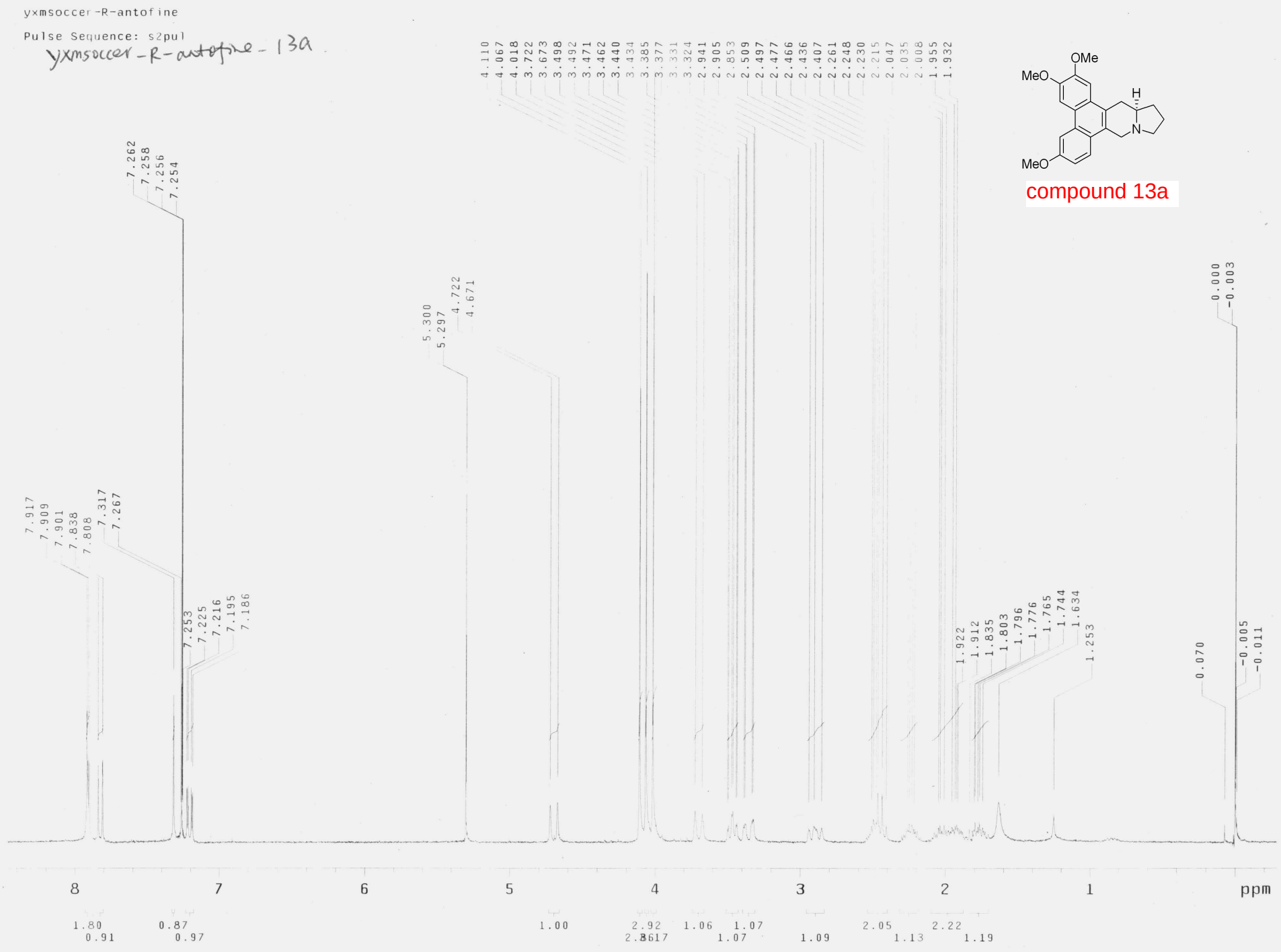
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 4798.5 Hz
Single scan
OBSERVE H1, 399.8639792 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 9 sec

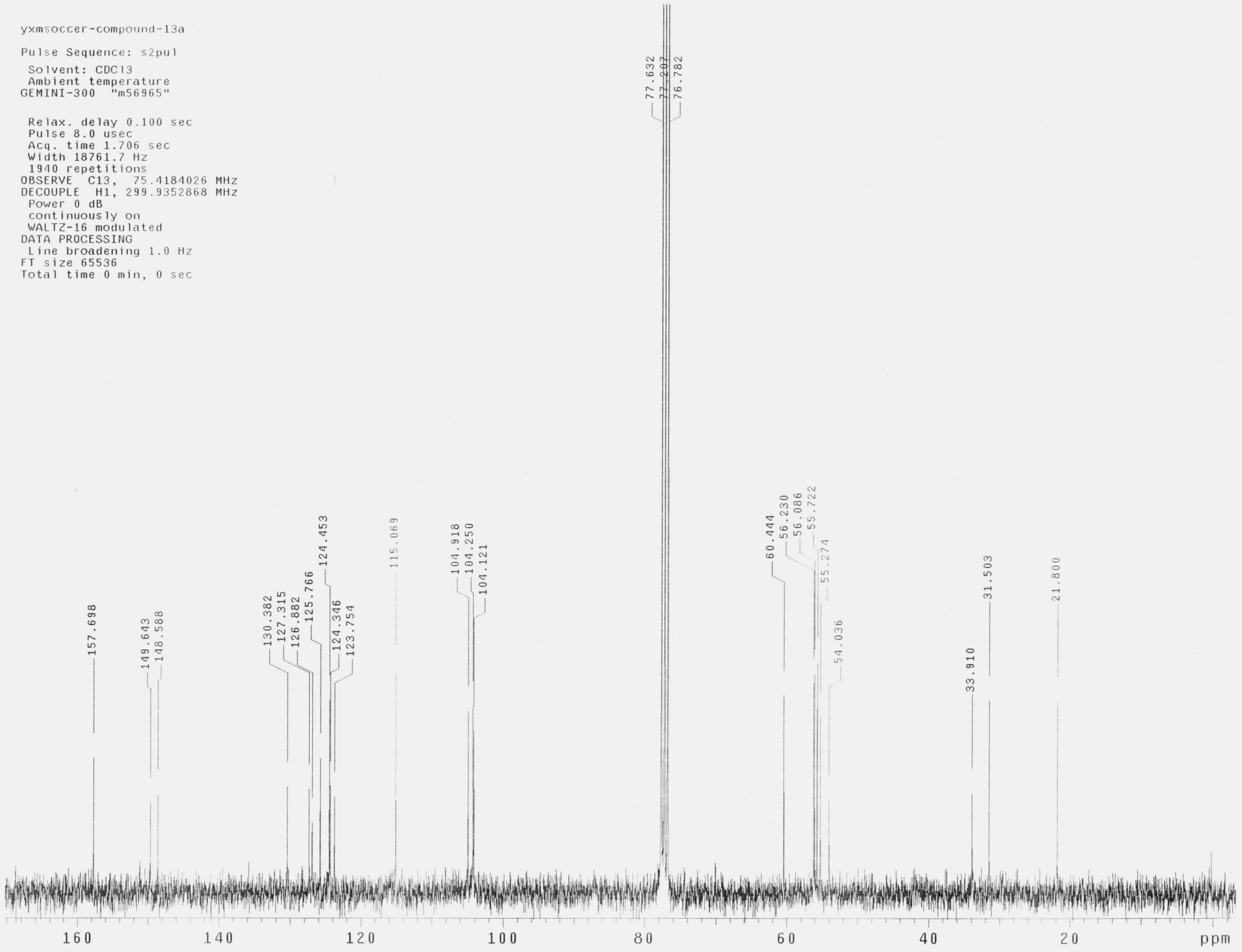


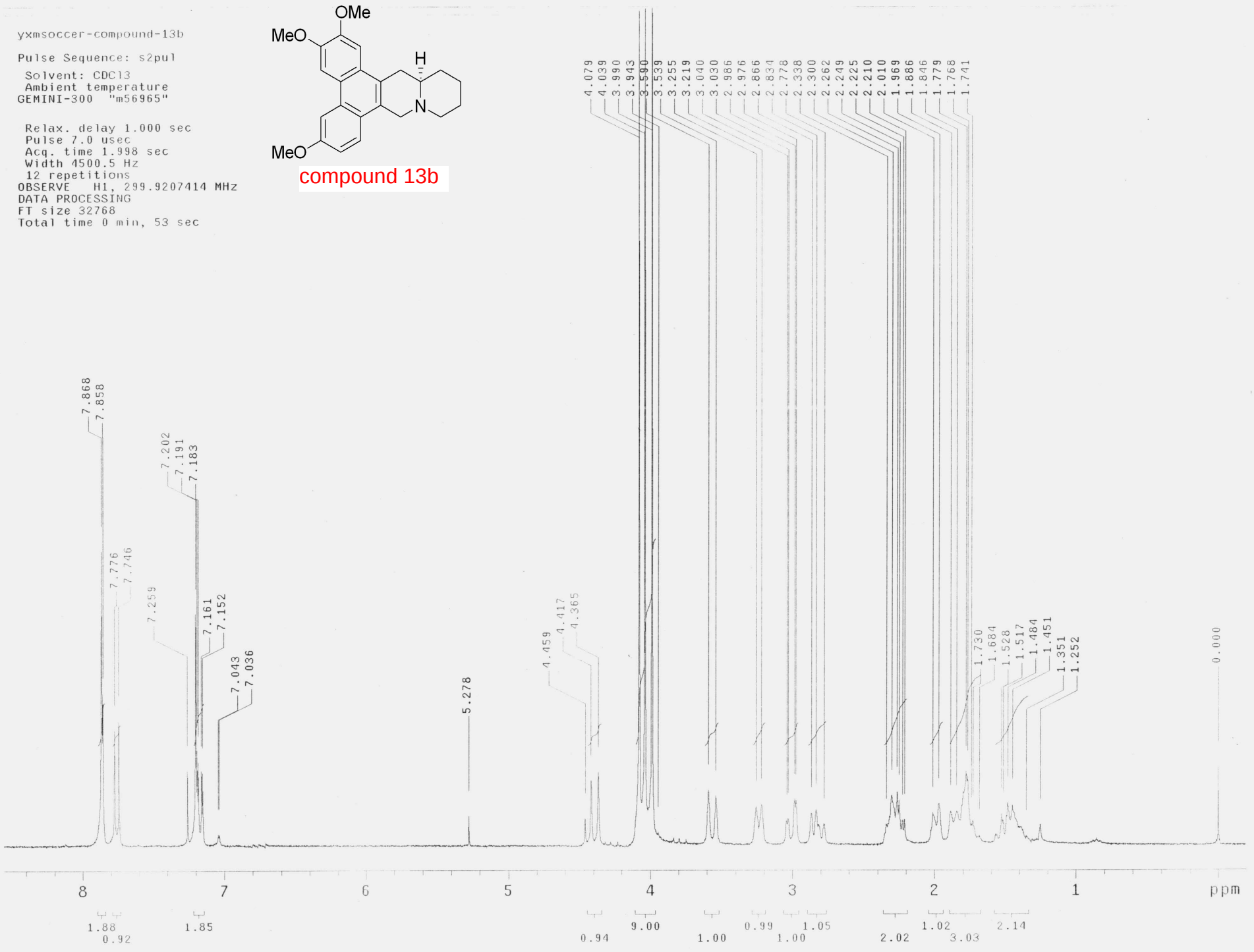
Compound 16

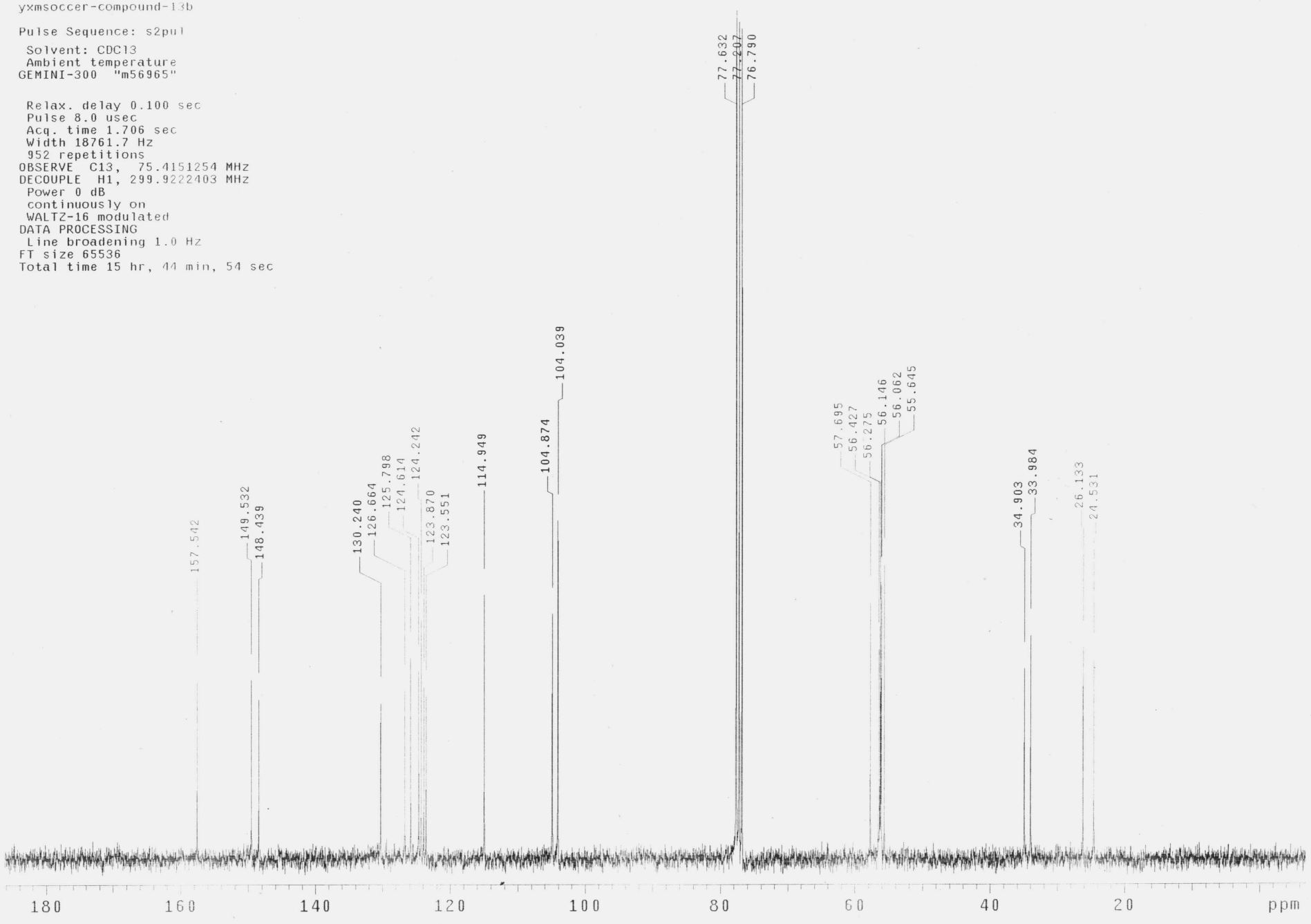



yxm-JOC-16-carbon
File: Carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: xiaoming
INOVA-400 "kerr-inova400.pha.unc.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24132.7 Hz
20000 repetitions
OBSERVE C13, 100.5458540 MHz
DECOUPLE H1, 399.8659917 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 12 hr, 49 min, 47 sec
159.743
157.751
149.613
148.617
130.290
127.749
127.632
125.859
124.526
124.497
123.757
115.143
104.874
104.302
104.039
95.322
77.552
77.230
76.915
63.767
57.863
57.080
56.245
56.091
55.761
51.872
33.860
0.210
180
160
140
120
100
80
60
40
20
ppm


yxmsoccer-R-antofine
Pulse Sequence: s2pul
yxmsoccer-R-antofine-13a
OMe
MeO
MeO
H
N
compound 13a
ppm


yxmsoccer-compound-13a
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "m56965"
Relax. delay 0.100 sec
Pulse 8.0 usec
Acq. time 1.706 sec
Width 18761.7 Hz
1940 repetitions
OBSERVE C13, 75.4184026 MHz
DECOUPLE H1, 299.9352868 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 0 min, 0 sec
77.632
77.207
76.782
157.698
149.643
148.588
130.382
127.315
126.882
125.766
124.453
124.346
123.754
115.069
104.918
104.250
104.121
60.444
56.230
56.086
55.722
55.274
54.036
33.910
31.503
21.800
160
140
120
100
80
60
40
20
ppm

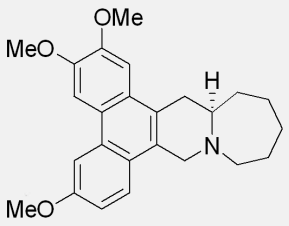

yxmsoccer-compound-13b
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "m56965"
Relax. delay 1.000 sec
Pulse 7.0 usec
Acq. time 1.998 sec
Width 4500.5 Hz
12 repetitions
OBSERVE H1, 299.9207414 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 53 sec
OMe
MeO
MeO
H
N
compound 13b
7.868
7.858
7.776
7.746
7.259
7.202
7.191
7.183
7.161
7.152
7.043
7.036
5.278
4.459
4.417
4.365
4.079
4.039
3.990
3.943
3.590
3.539
3.255
3.219
3.040
3.030
2.986
2.976
2.866
2.834
2.778
2.338
2.300
2.262
2.249
2.225
2.210
2.010
1.969
1.886
1.846
1.779
1.768
1.741
1.730
1.684
1.528
1.517
1.484
1.451
1.351
1.252
0.000
8
7
6
5
4
3
2
1
ppm
1.88
0.92
1.85
0.94
9.00
1.00
0.99
1.00
1.05
2.02
1.02
3.03
2.14


yxmsoccer-compound-13b
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "m56965"
Relax. delay 0.100 sec
Pulse 8.0 usec
Acq. time 1.706 sec
Width 18761.7 Hz
952 repetitions
OBSERVE C13, 75.4151254 MHz
DECOUPLE H1, 299.9222403 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 15 hr, 44 min, 54 sec
157.542
149.532
148.439
130.240
126.664
125.798
124.614
124.242
123.870
123.551
114.949
104.874
104.039
77.632
77.207
76.790
57.695
56.427
56.275
56.146
56.062
55.645
34.903
33.984
26.133
24.531
180
160
140
120
100
80
60
40
20
ppm

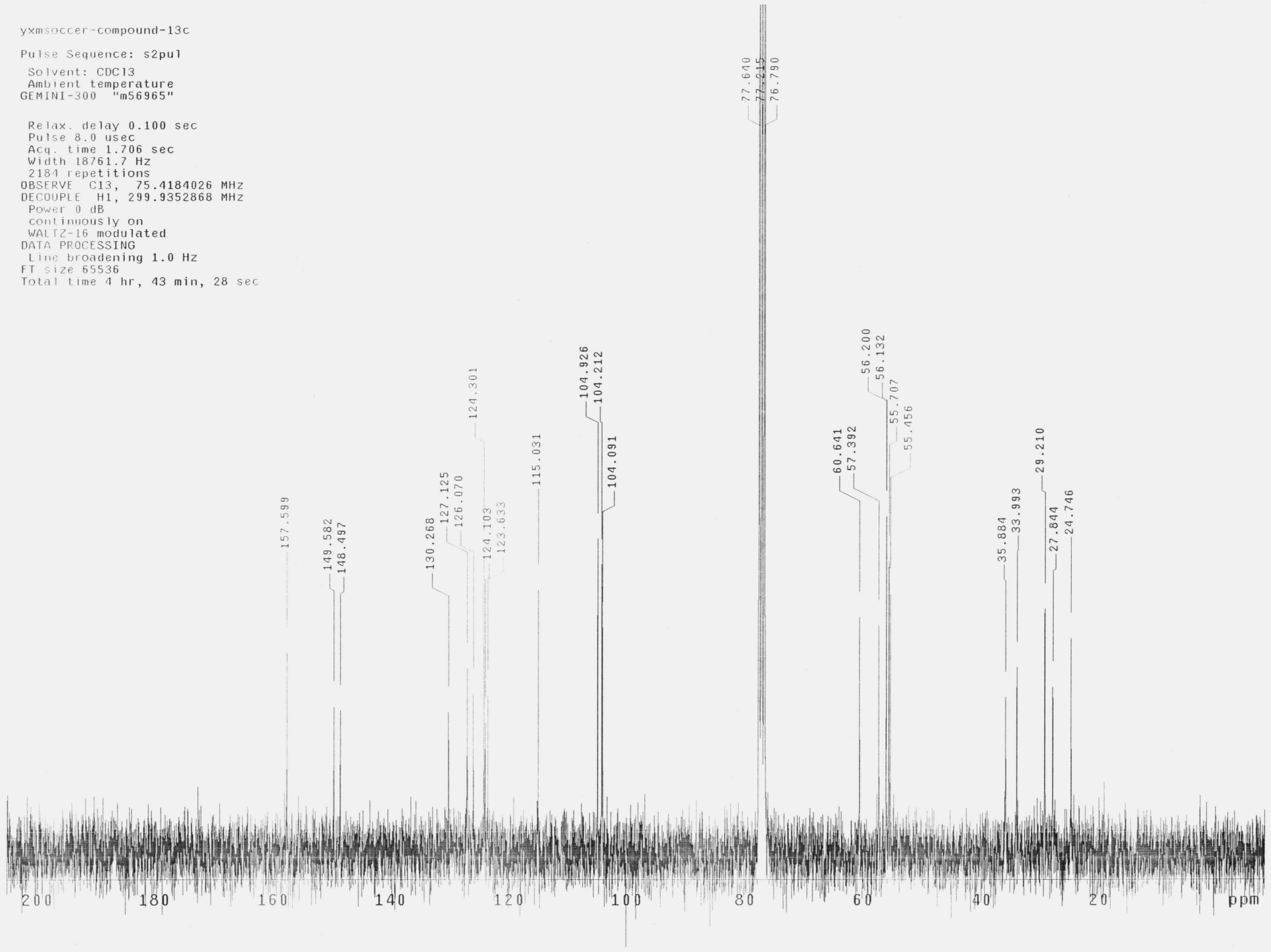
yxmsoccer-compound-13c

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "m56965"

Relax. delay 1.000 sec
Pulse 7.0 usec
Acq. time 1.998 sec
Width 4500.5 Hz
60 repetitions
OBSERVE H1, 299.9337832 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 35 sec



compound 13c (E7)


yxmsoccer-compound-13c
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "m56965"
Relax. delay 0.100 sec
Pulse 8.0 usec
Acq. time 1.706 sec
Width 18761.7 Hz
2184 repetitions
OBSERVE C13, 75.4184026 MHz
DECOUPLE H1, 299.9352868 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 4 hr, 43 min, 28 sec
157.599
149.582
148.497
130.268
127.125
126.070
124.301
124.103
123.633
115.031
104.926
104.212
104.091
77.640
77.215
76.790
60.641
57.392
56.200
56.132
55.707
55.456
35.884
33.993
29.210
27.844
24.746
200
180
160
140
120
100
80
60
40
20
ppm

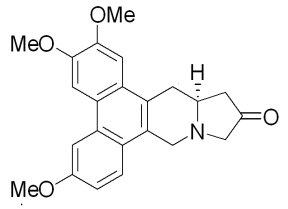
yxm-JOC-17

File: Proton

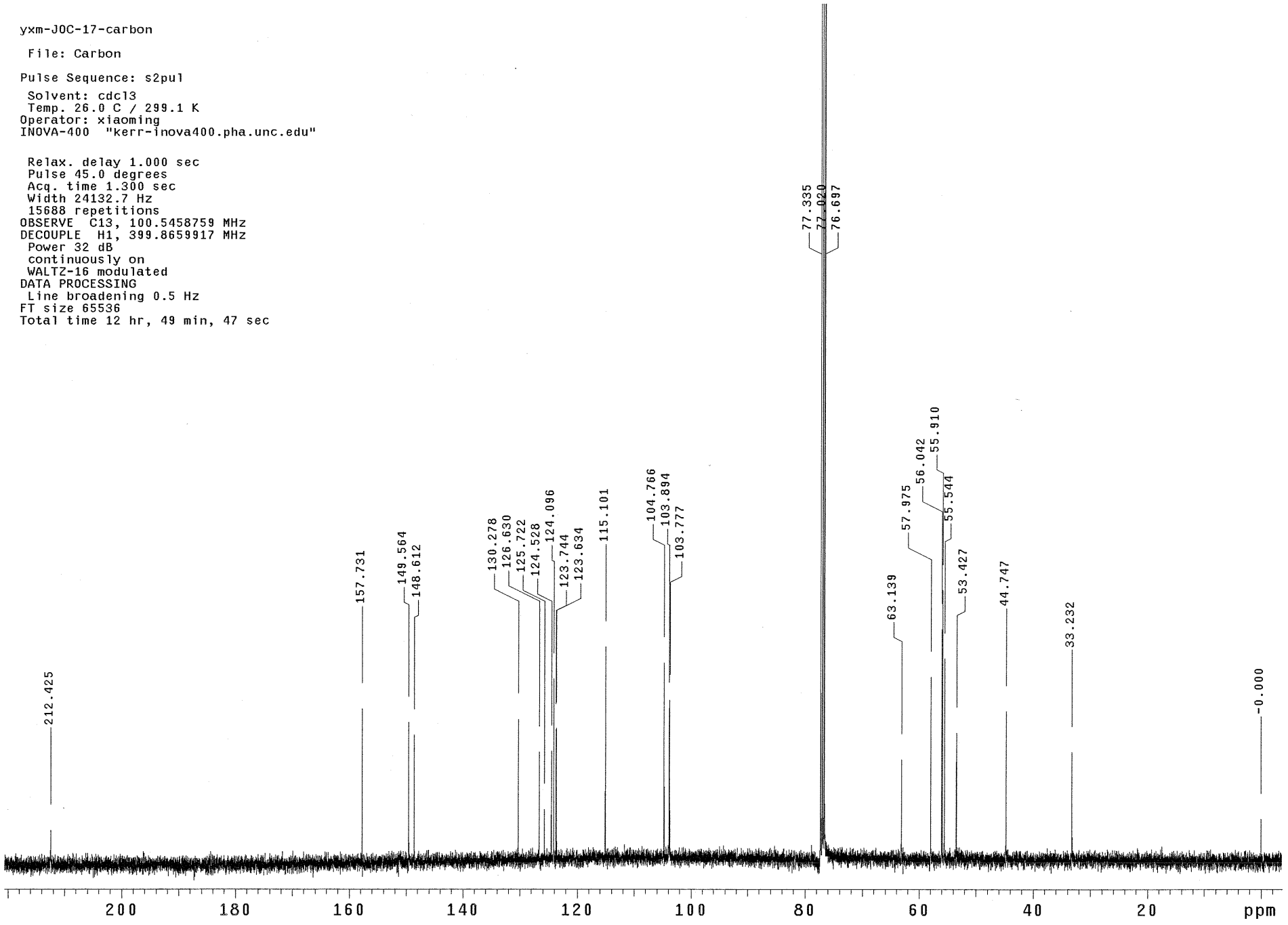
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: xiaoming
INOVA-400 "kerr-inova400.pha.unc.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 4798.5 Hz
16 repetitions
OBSERVE H1, 399.8639779 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec



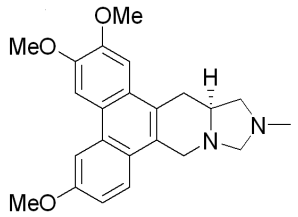
compound 17


yxm-JOC-17-carbon
File: Carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: xiaoming
INOVA-400 "kerr-inova400.pha.unc.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24132.7 Hz
15688 repetitions
OBSERVE C13, 100.5458759 MHz
DECOUPLE H1, 399.8659917 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 12 hr, 49 min, 47 sec
212.425
157.731
149.564
148.612
130.278
126.630
125.722
124.528
124.096
123.744
123.634
115.101
104.766
103.894
103.777
77.335
77.020
76.697
63.139
57.975
56.042
55.910
55.544
53.427
44.747
33.232
-0.000
200
180
160
140
120
100
80
60
40
20
ppm

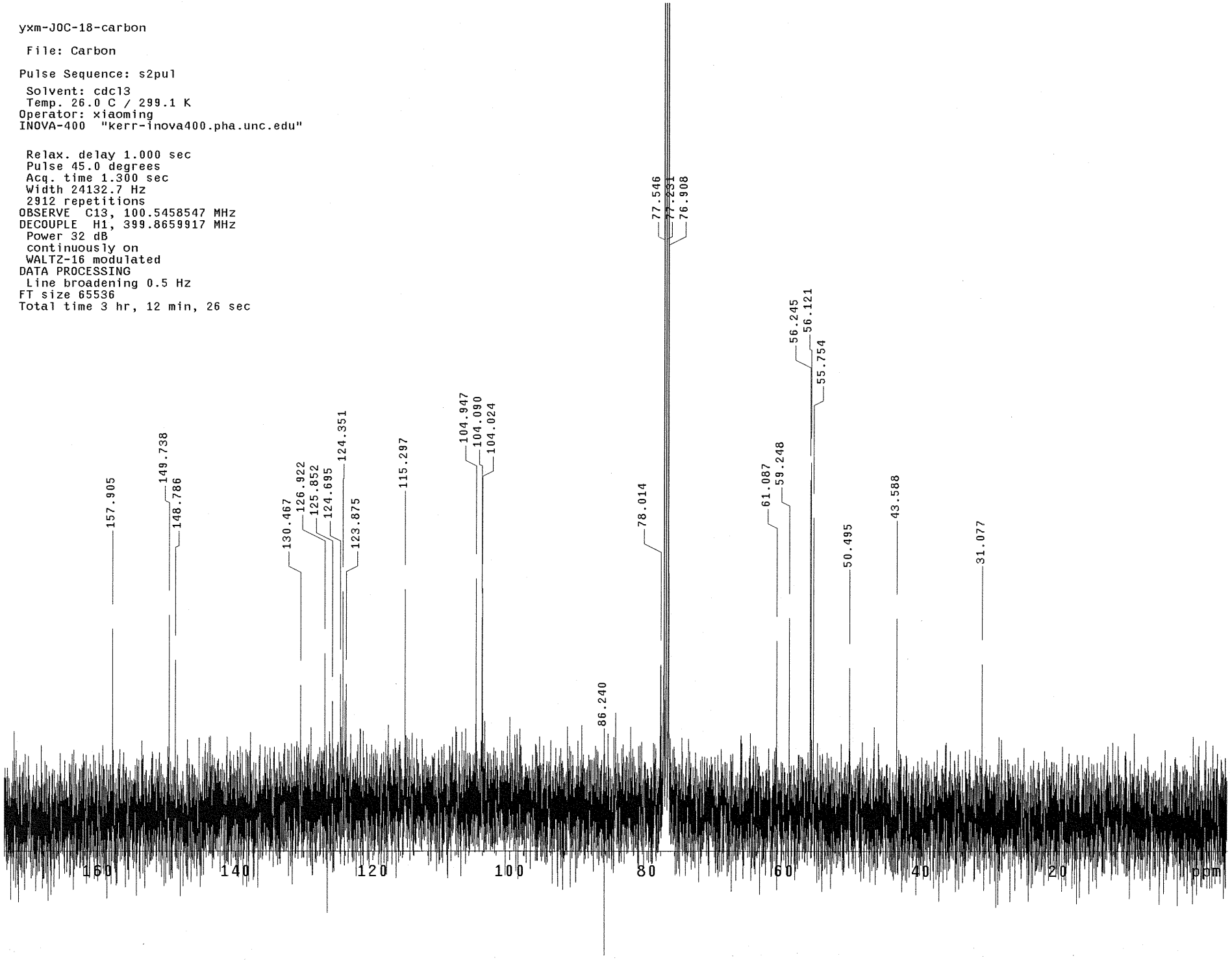
yxm~~131~~ JOC-18

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "m56965"

Relax. delay 1.000 sec
Pulse 7.0 usec
Acq. time 1.998 sec
Width 4500.5 Hz
64 repetitions
OBSERVE H1, 299.9207370 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 35 sec



compound 18


yxm-JOC-18-carbon
File: Carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 26.0 C / 299.1 K
Operator: xiaoming
INOVA-400 "kerr-inova400.pha.unc.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24132.7 Hz
2912 repetitions
OBSERVE C13, 100.5458547 MHz
DECOUPLE H1, 399.8659917 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 12 min, 26 sec
157.905
149.738
148.786
130.467
126.922
125.852
124.695
124.351
123.875
115.297
104.947
104.090
104.024
86.240
78.014
77.546
77.231
76.908
61.087
59.248
56.245
56.121
55.754
50.495
43.588
31.077
160
140
120
100
80
60
40
20
ppm