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The evolution of a stereoselective synthesis of the C1-C16 fragment of bryostatins

Matthew Ball, Anne Baron, Ben Bradshaw, Raphaël Dumeunier, Matthew O'Brien, Hiroki Omori and Eric J. Thomas

Supplementary data

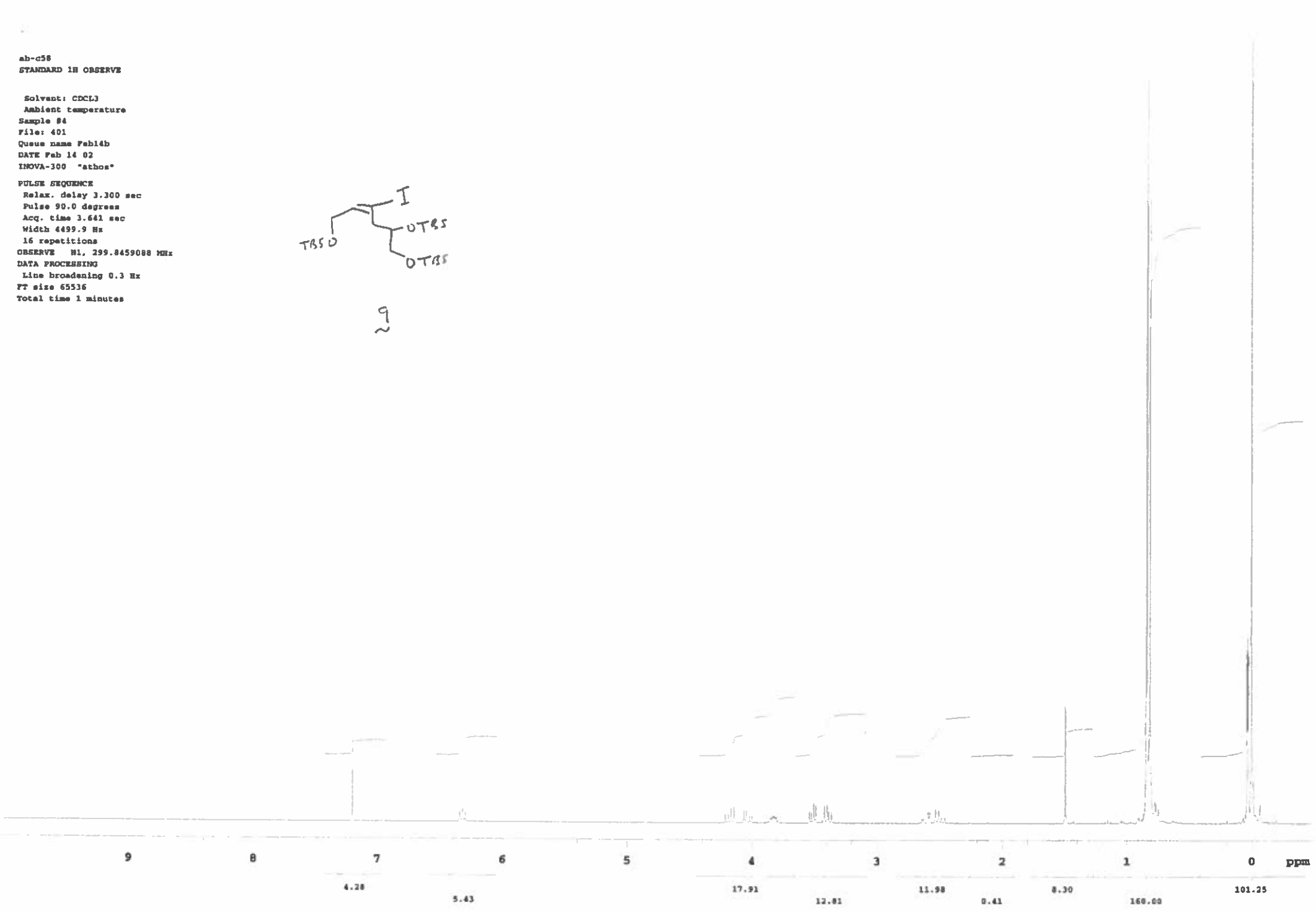
Copies of ^{1}H and ^{13}C NMR spectra of some key compounds – in numerical order

ab-c58
STANDARD 1H OBSERVE

Solvent: CDCL3
Ambient temperature
Sample 84
File: 401
Queue name Feb14b
DATE Feb 14 02
INOVA-300 "athos"

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4499.9 Hz
16 repetitions
OBSERVE H1, 299.8459088 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 minutes

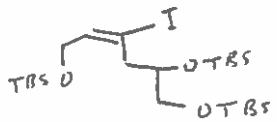
9



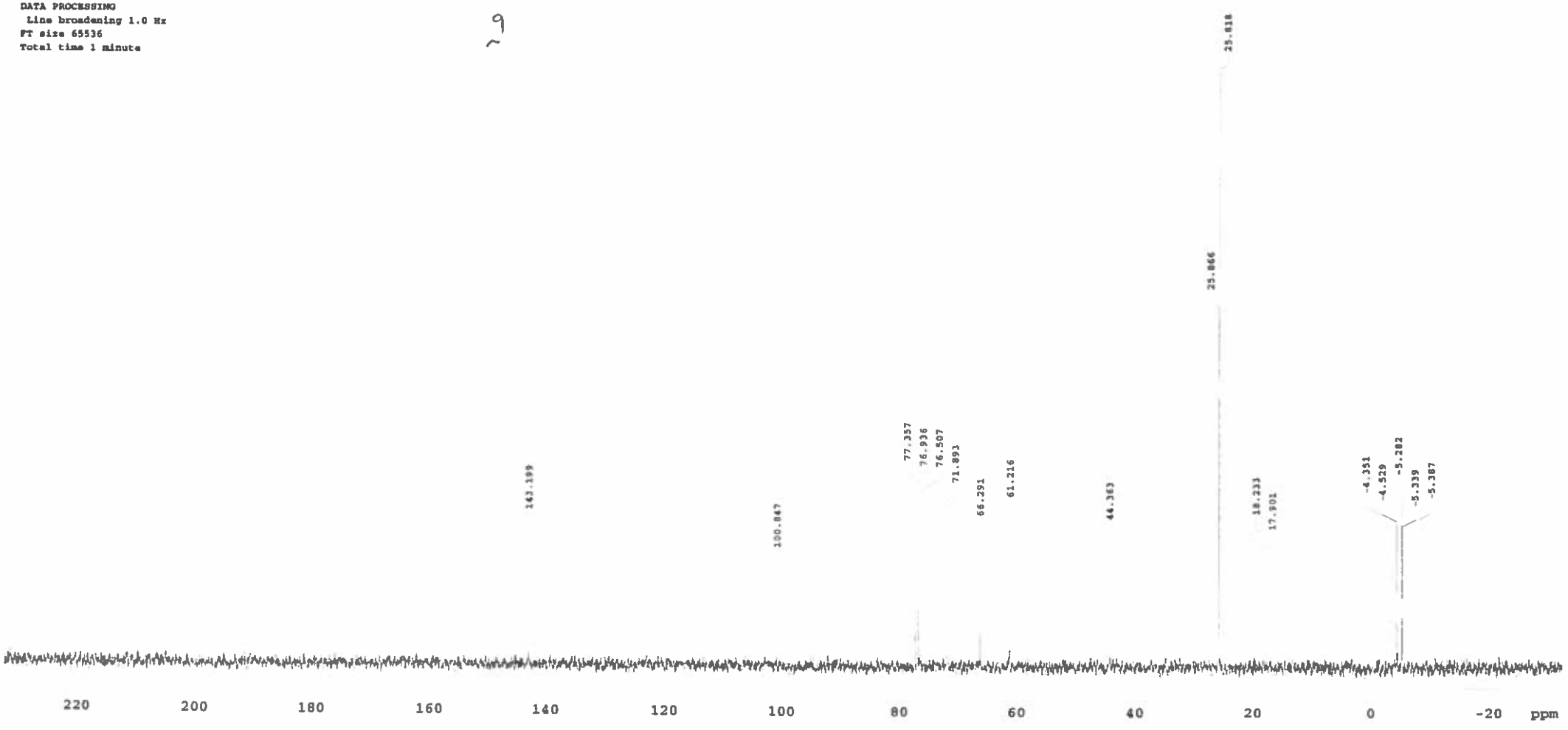
AB-C14
STANDARD 1H OBSERVE

Solvent: CDCL3
Ambient temperature
Sample #33
File: 3302
Queue name Nov27b
DATE Nov 27 01
INOVA-300 "sthoe"

PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 1.638 sec
Width 20000.0 Hz
16 repetitions
OBSERVE C13, 75.3963141 MHz
DECOUPLE H1, 299.8473785 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 minute



9

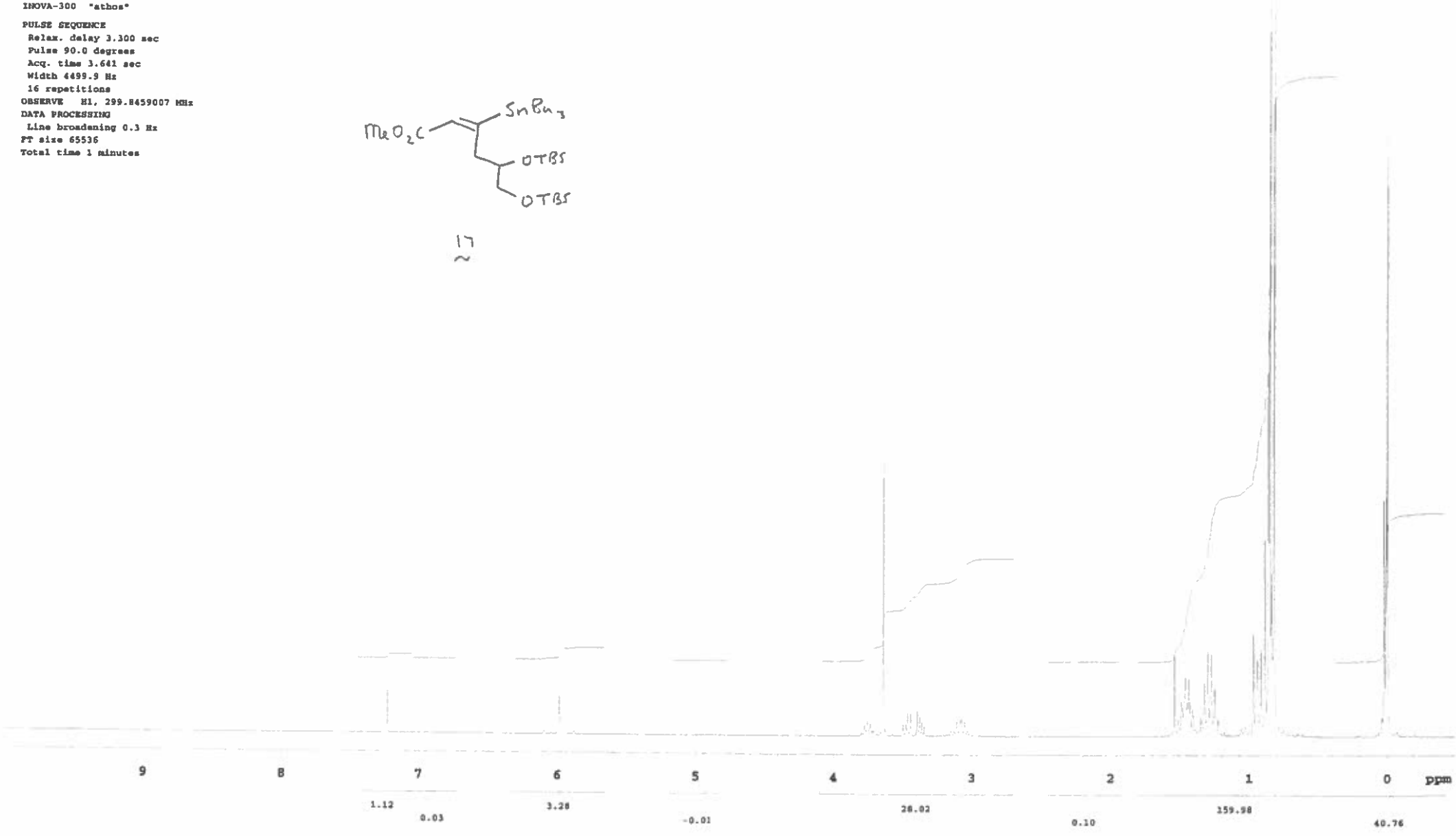


AB-B99
STANDARD 1H OBSERVE

Solvent: CDCL3
Ambient temperature
Sample #5
File: 501
Queue name Nov07b
DATE Nov 7 01
INOVA-300 "athos"

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4499.9 Hz
16 repetitions
OBSERVE H1, 299.8459007 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 minutes

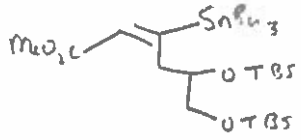
17



AB-B99
13C OBSERVE

Solvent: CDCL3
Ambient temperature
Sample 832
File: 3201
Queue name Nov07b
DATE Nov 8 01
INOVA-300 "athos"

PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 1.638 sec
Width 20000.0 Hz
1024 repetitions
OBSERVE C13, 75.3963141 MHz
DECOUPLE H1, 299.8473785 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 27 minutes



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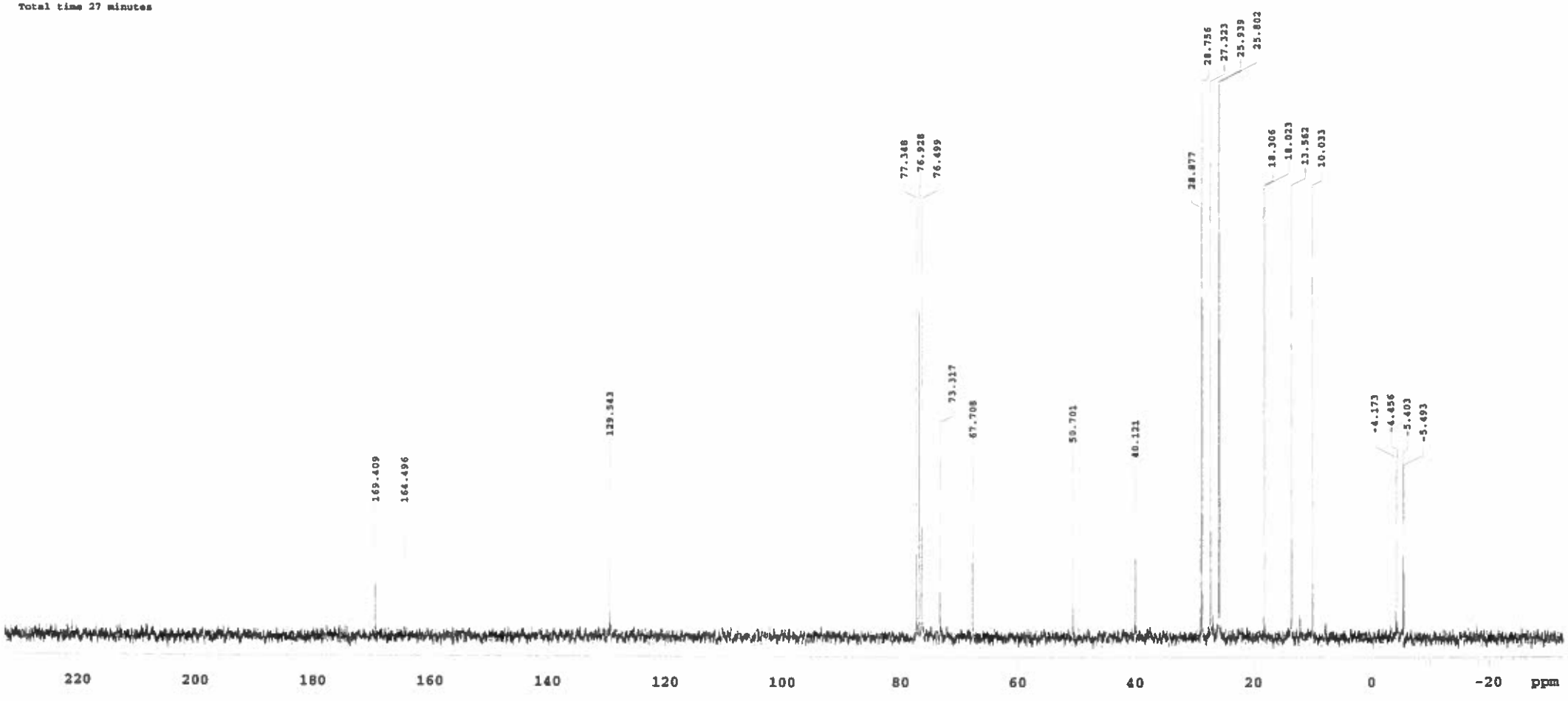
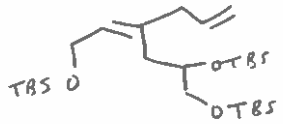


A Baron
AB-C66
cdcl3
Inova 400(RS)
Aramis
Proton 400MHz
04.03.02
434

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
File: 434-04-03-AB-RS
INOVA-400 "aramis"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.009 sec
Width 3597.8 Hz
16 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 24 sec



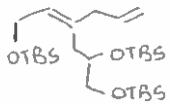
24

Integral Scale = 45000.0
Vertical Scale = 200.0

8 7 6 5 4 3 2 1 0 ppm

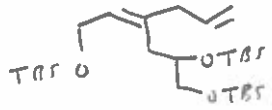
1.16 1.15 2.36 2.59 1.19 2.65 2.35 1.23 1.13 2.14 36.35 20.00

ab-c18
13C OBSERVE

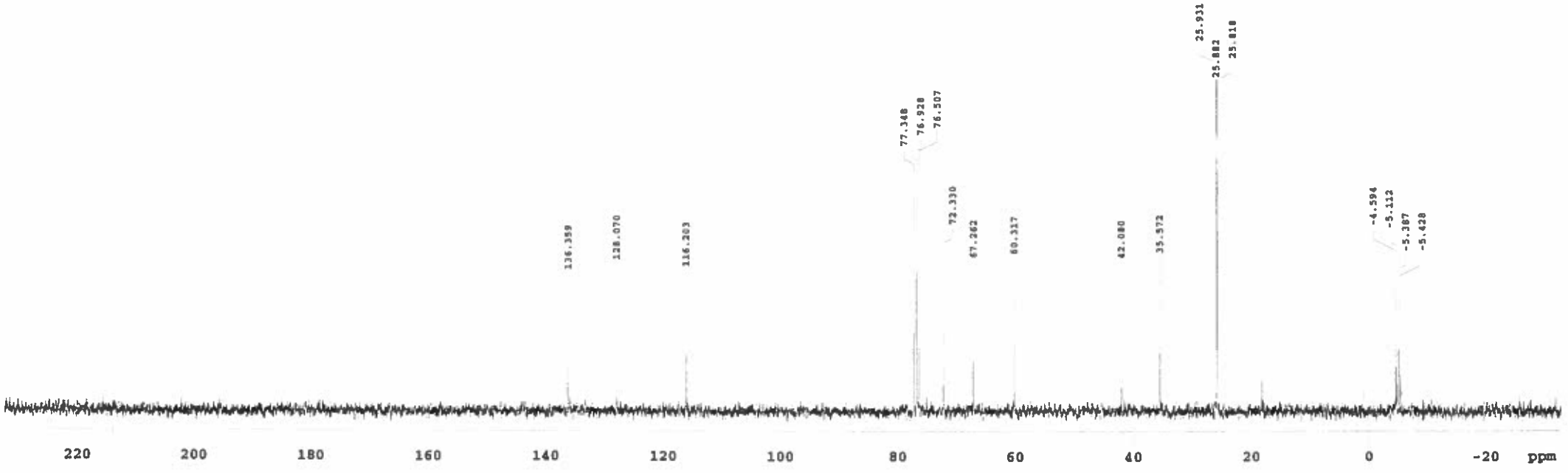


Solvent: CDCL3
Ambient temperature
Sample 05
File: 501
Queue name Dec05a
DATE Dec 5 01
INOVA-300 "athos"

PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 1.638 sec
Width 20000.0 Hz
256 repetitions
OBSERVE C13, 75.3963141 MHz
DECOUPLE H1, 299.8473785 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 minutes



24



ab-e251
STANDARD 1H OBSERVE

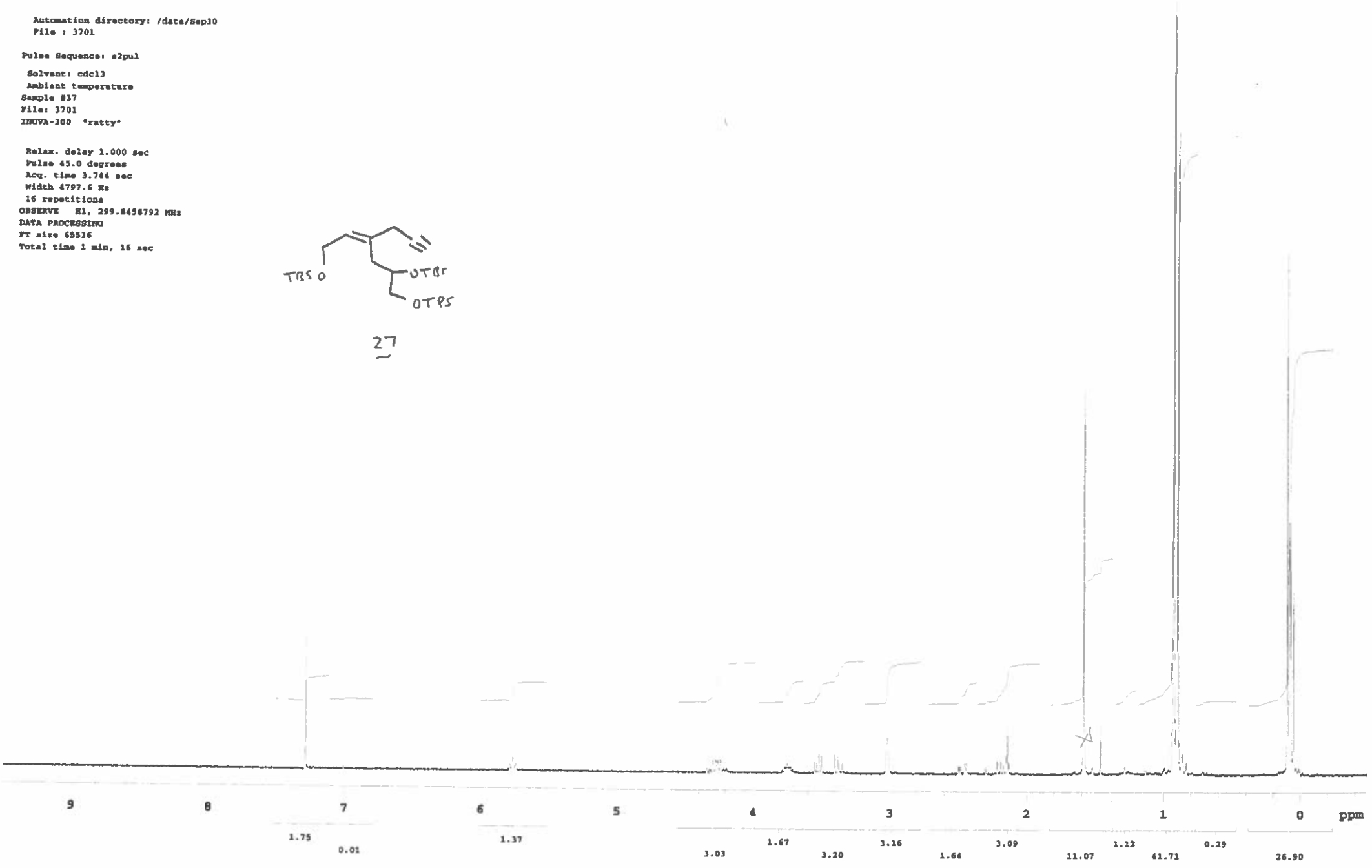
Automation directory: /data/Sep30
File : 3701

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample #37
File: 3701
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec

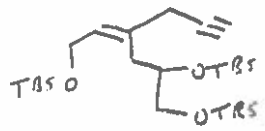
27



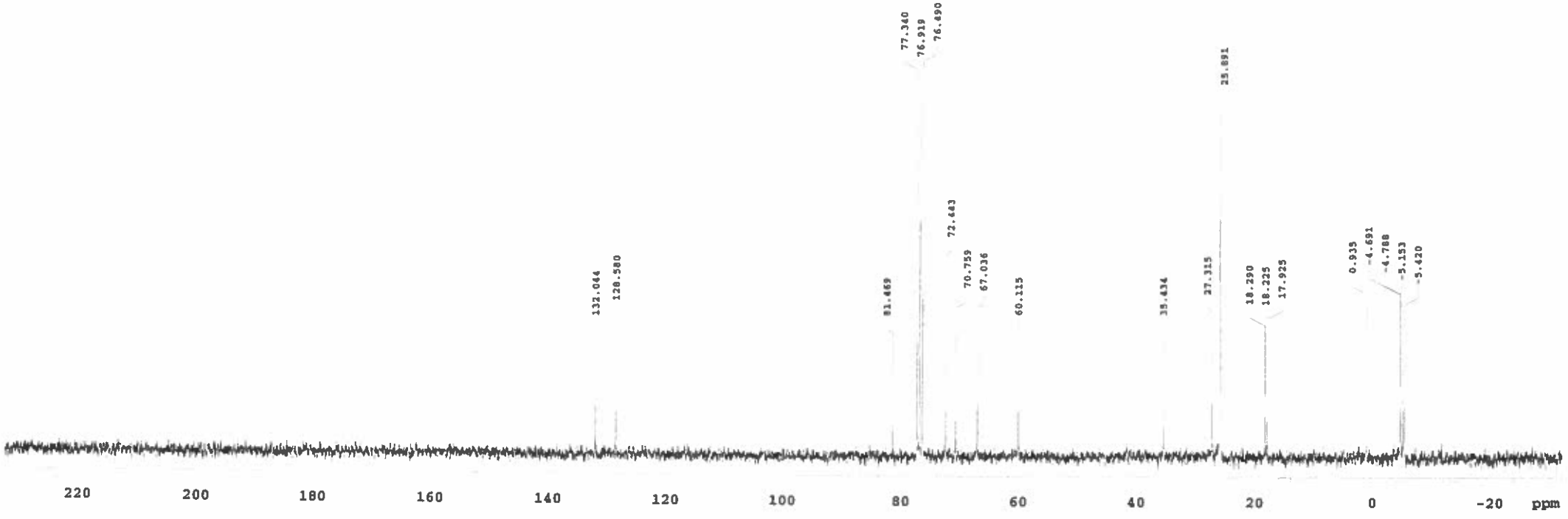
ab-c86
13C OBSERVE

Solvent: CDCL3
Ambient temperature
Sample #25
File: 2501
Queue name Mar22b
DATE Mar 22 02
INOVA-300 "athos"

PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 1.638 sec
Width 20000.0 Hz
1024 repetitions
OBSERVE C13, 75.3963141 MHz
DECOUPLE H1, 299.8473785 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 27 minutes



27

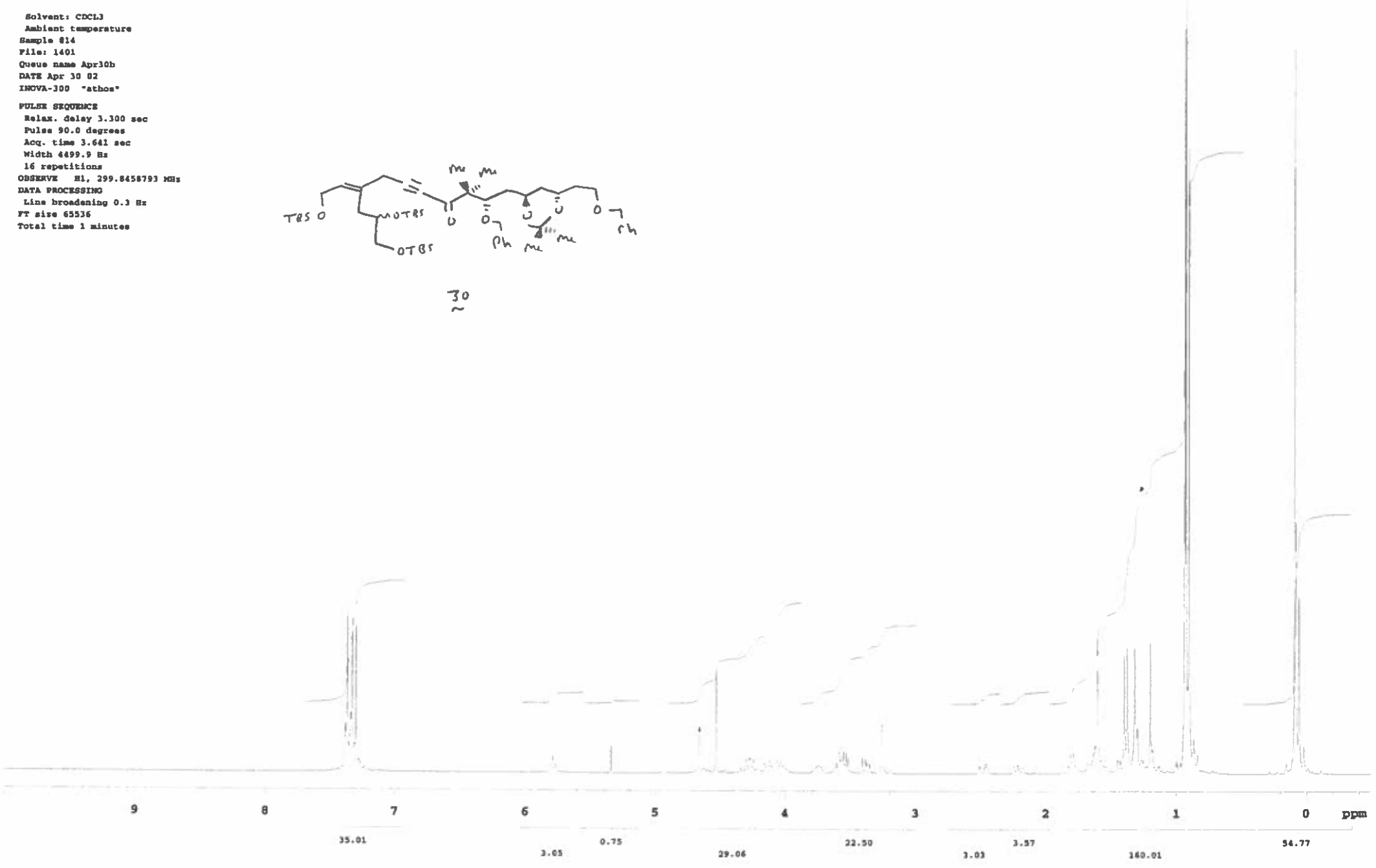


ab-d8
STANDARD 1H OBSERVE

Solvent: CDCL3
Ambient temperature
Sample 814
File: 1401
Queue name Apr30b
DATE Apr 30 02
INOVA-300 "athos"

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4499.9 Hz
16 repetitions
OBSERVE H1, 299.8458793 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 minutes

30



Anne Baron
AB-Dg
cdcl3
Inova 400(GS)
Armis
13C 100MHz
03.05.02
678c

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
File: 678c-03-05-AB-GS
INOVA-400 "armis"

Pulse 36.0 degree
Acq. time 1.311 sec
Width 25000.0 Hz
16896 repetitions
OBSERVE C13, 100.5696308 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 366542 hr, 47 min, 28 sec

30

192.959
138.625
138.405
131.024
129.453
128.308
128.217
127.640
127.496
127.329
127.246
100.231
93.099
81.758
79.831
77.350
77.236
77.031
76.720
74.649
73.124
72.760
66.949
66.653
63.634
63.474
60.038
53.036
38.887
38.614
36.027
35.587
29.798
28.251
26.051
25.960
25.451
25.246
21.302
19.936
18.461
18.404
18.100
-4.454
-4.507
-4.963
-4.947
-5.175
-5.205

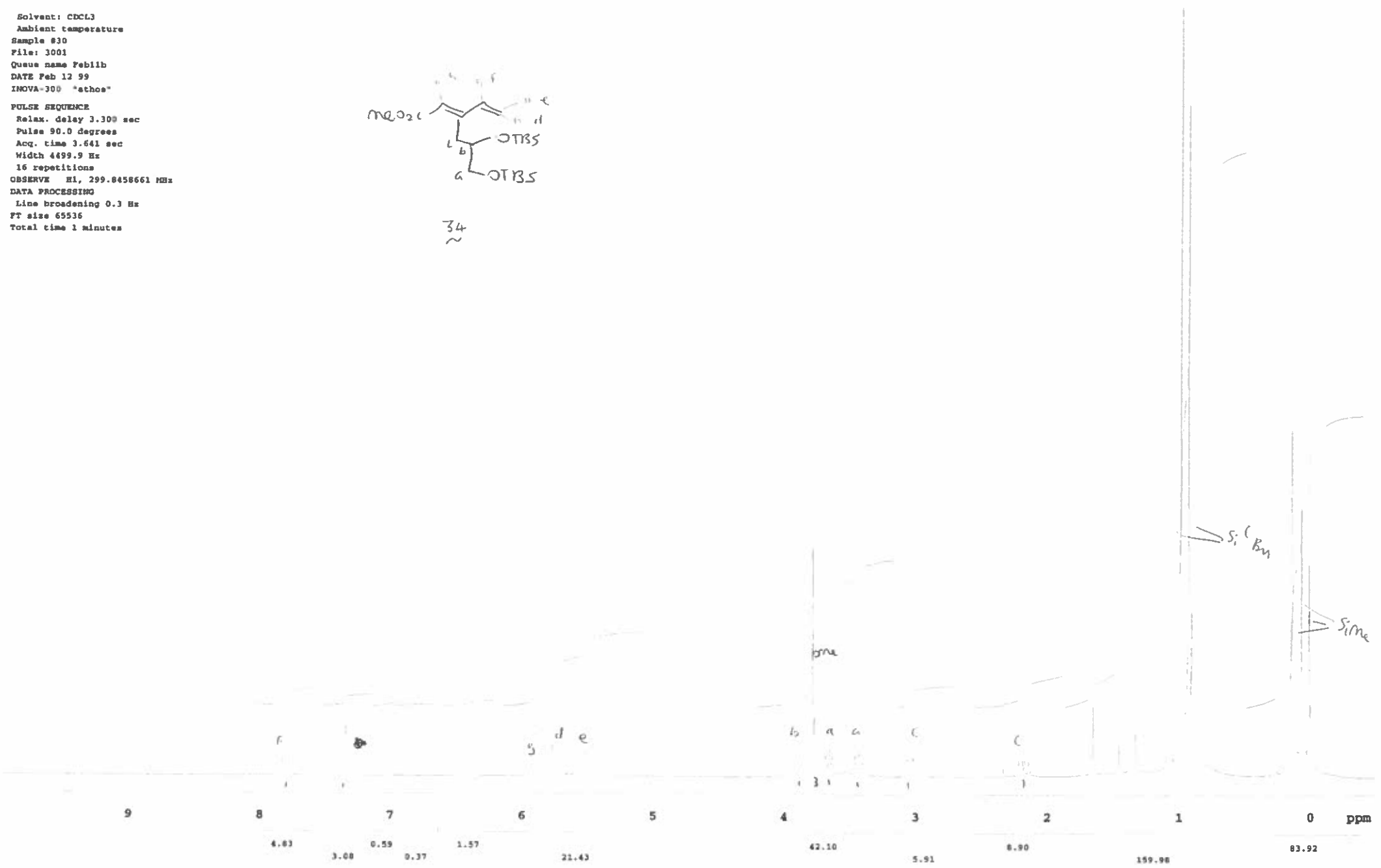
200 180 160 140 120 100 80 60 40 20 0 ppm

matthew o'brien mob1/051
STANDARD 1H OBSERVE

Solvent: CDCL3
Ambient temperature
Sample 830
File: 3001
Queue name Feb11b
DATE Feb 12 99
INOVA-300 "athos"

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4499.9 Hz
16 repetitions
OBSERVE H1, 299.8458661 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 minutes

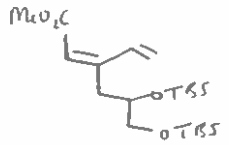
34



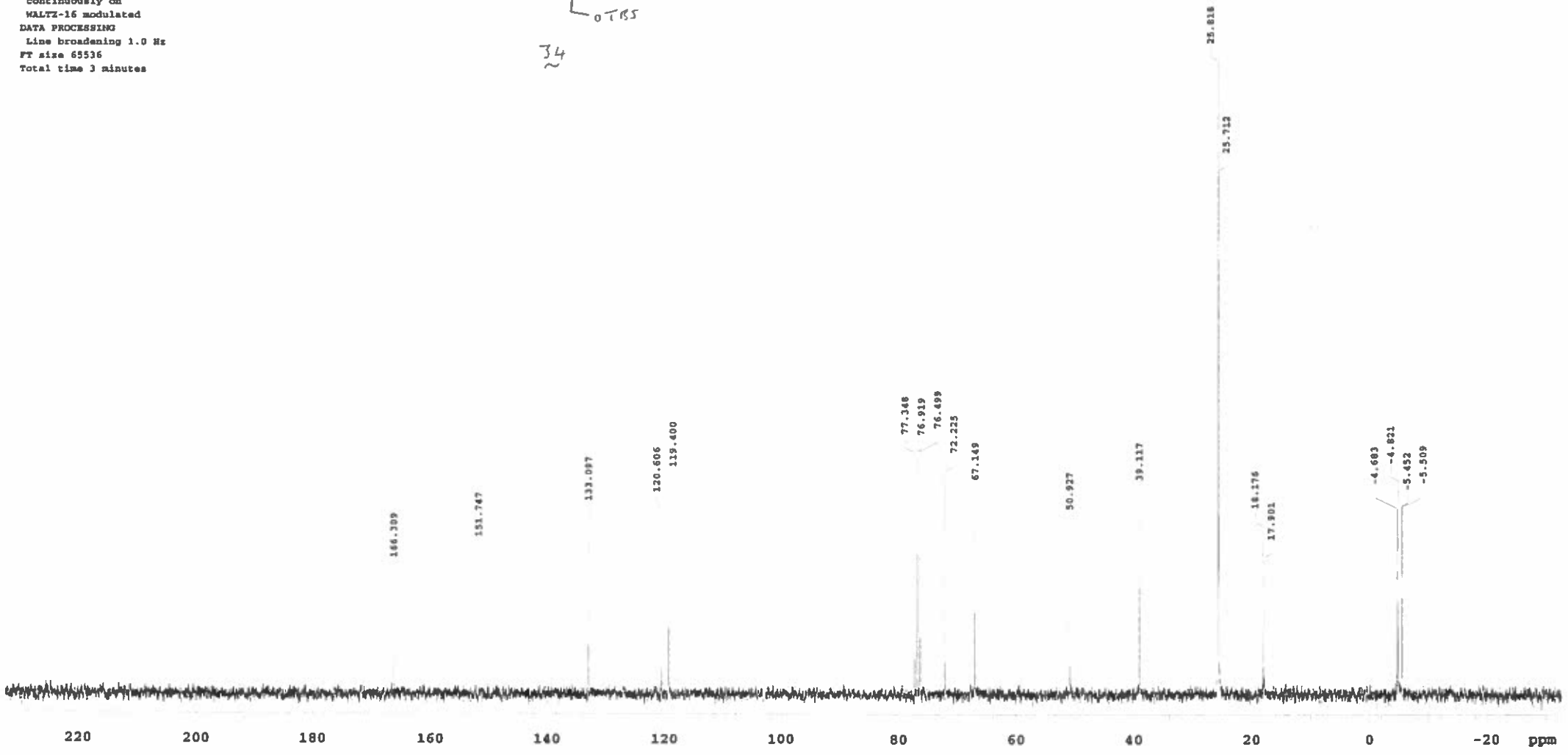
matthew o'brien mob/VI
13C OBSERVE

Solvent: CDCL3
Ambient temperature
Sample #15
File: 1501
Queue name Aug27a
DATE Aug 27 99
INOVA-300 "athos"

PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 1.638 sec
Width 20000.0 Hz
128 repetitions
OBSERVE C13, 75.3963141 MHz
DECOUPLE H1, 399.8473785 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 minutes



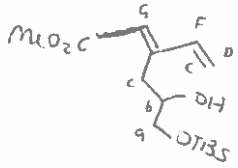
34



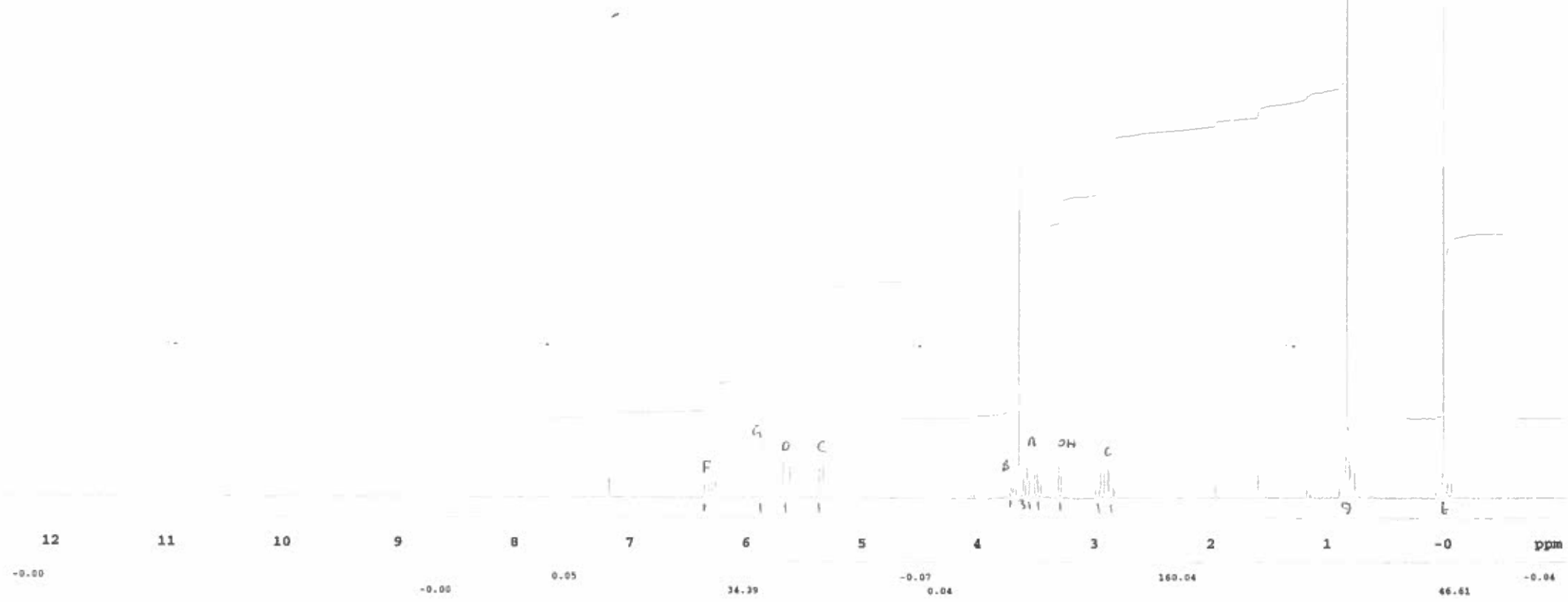
matthew o'brien mob1/091/a
STANDARD 1H OBSERVE

Solvent: CDCL3
Ambient temperature
Sample 849
File: 4901
Queue name Apr27b
DATE Apr 27 99
INOVA-300 "athos"

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4499.9 Hz
16 repetitions
OBSERVE H1, 299.8459134 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 minutes



76

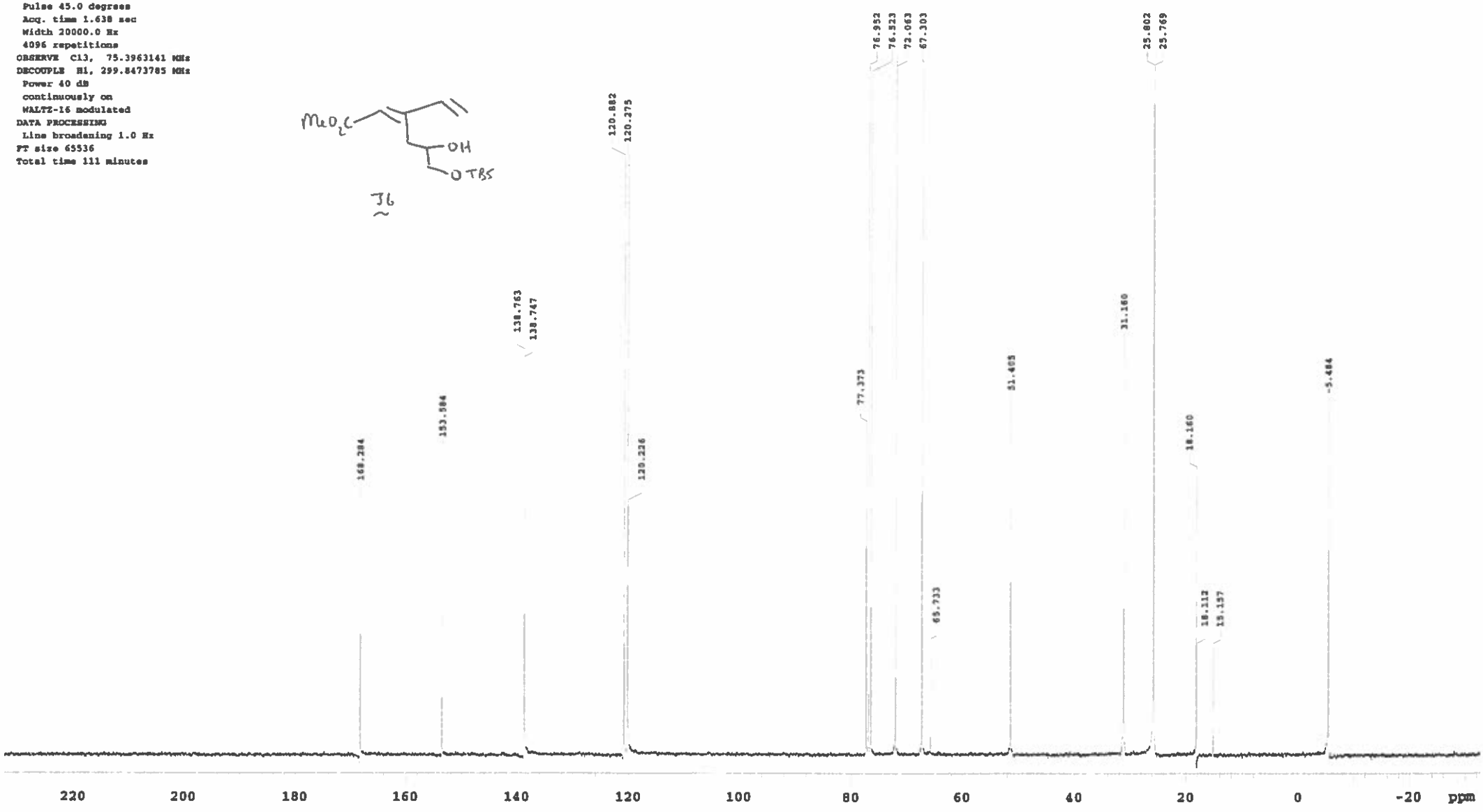


MATTHEW O'BRIEN MOB10/67/EP
13C OBSERVE

Solvent: CDCL3
Ambient temperature
Sample #8
File: 801
Queue name Jan19a
DATE Jan 19 02
INOVA-300 "athos"

PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 1.638 sec
Width 20000.0 Hz
4096 repetitions
OBSERVE C13, 75.3963141 MHz
DECOUPLE H1, 299.8473785 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 111 minutes

J6

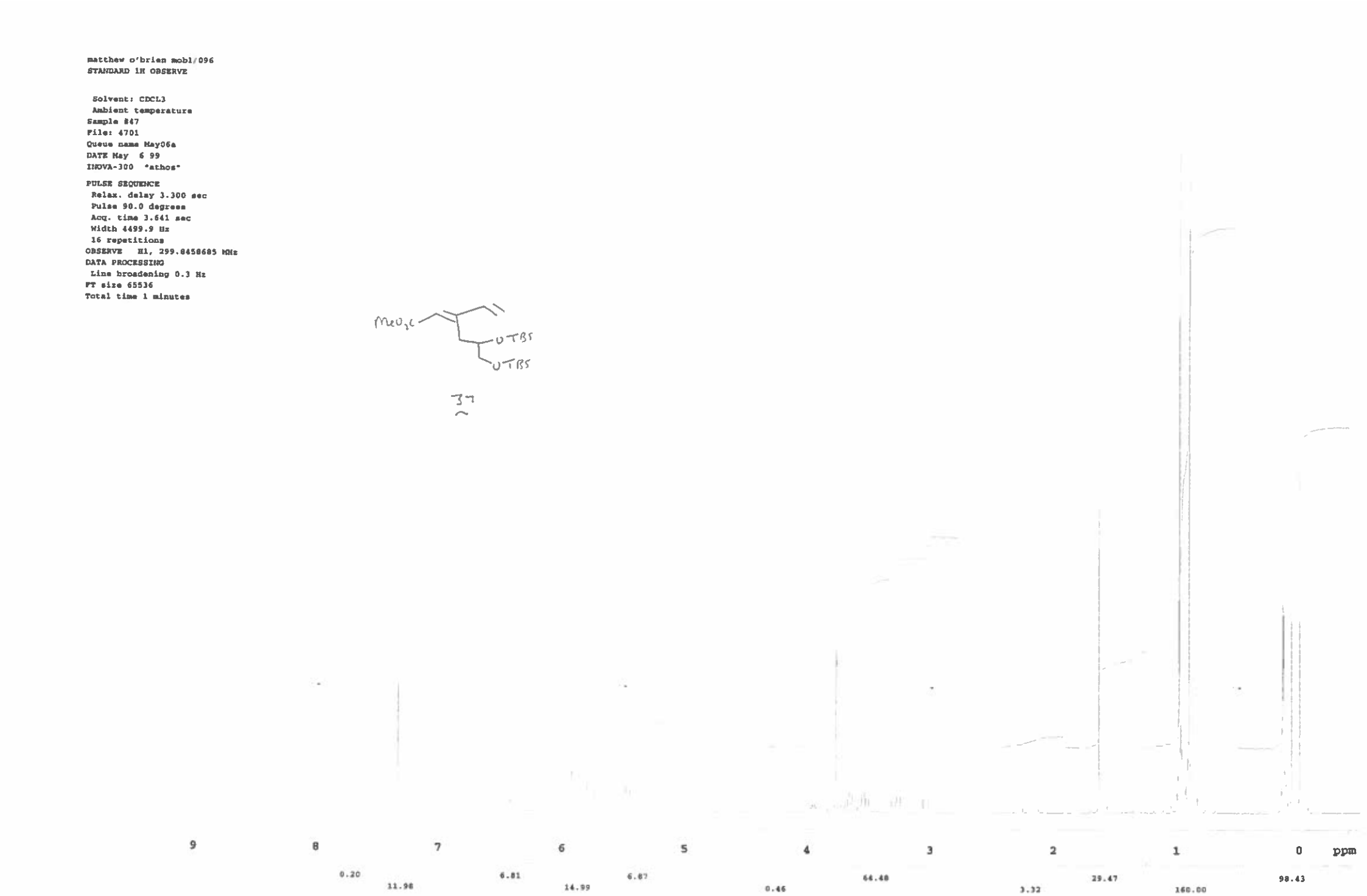


matthew o'brien mobl/096
STANDARD 1H OBSERVE

Solvent: CDCL3
Ambient temperature
Sample #47
File: 4701
Queue name May06a
DATE May 6 99
INOVA-300 "athos"

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4499.9 Hz
16 repetitions
OBSERVE H1, 299.8458685 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 minutes

37

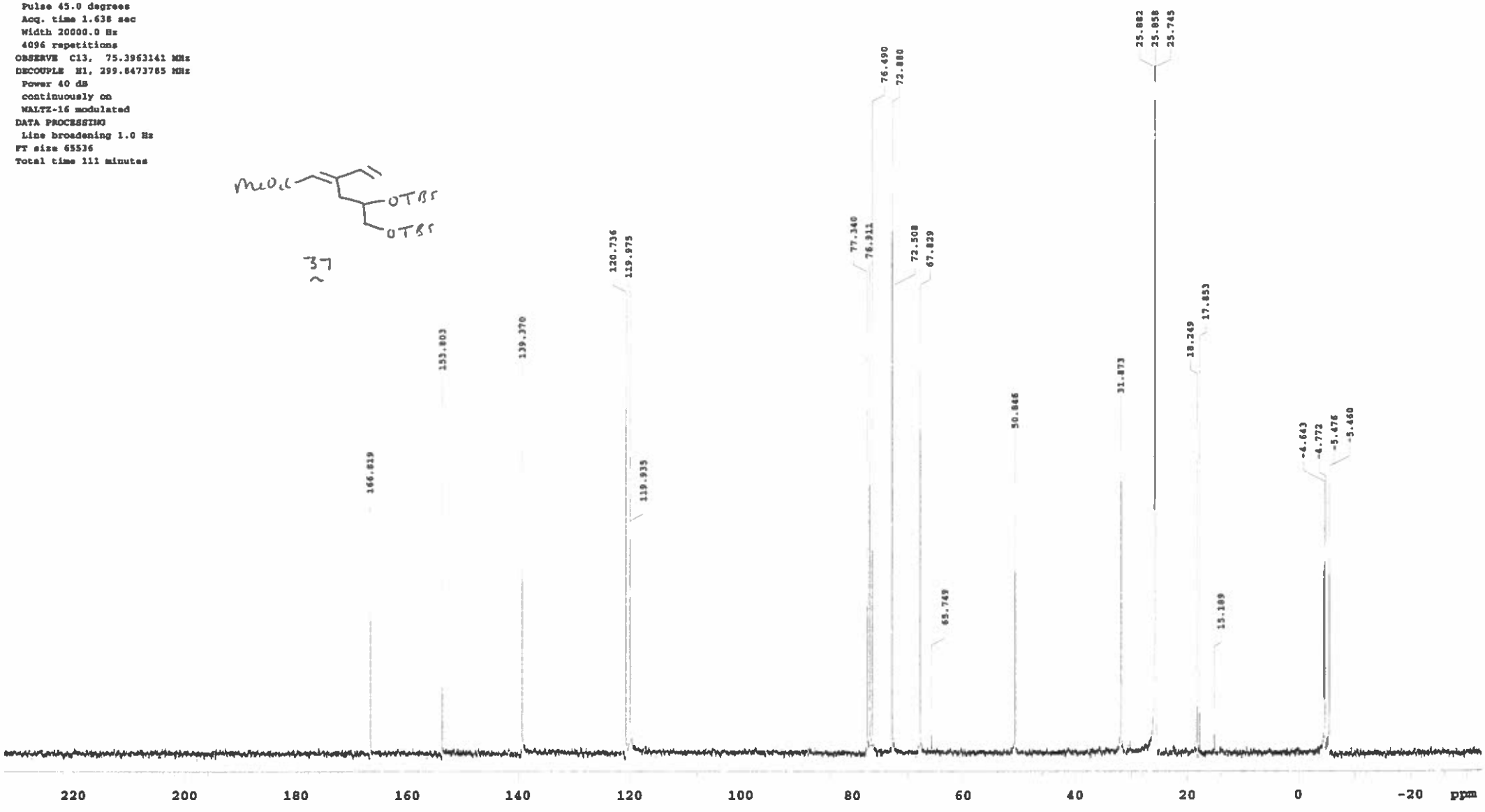


MATTHEW O'BRIEN MOB10/70/COLL
13C OBSERVE

Solvent: CDCL3
Ambient temperature
Sample 87
File: 701
Queue name Jan19a
DATE Jan 19 02
INOVA-300 "athos"

PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 1.638 sec
Width 20000.0 Hz
4096 repetitions
OBSERVE C13, 75.3963141 MHz
DECOUPLE H1, 299.8473785 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 111 minutes

87

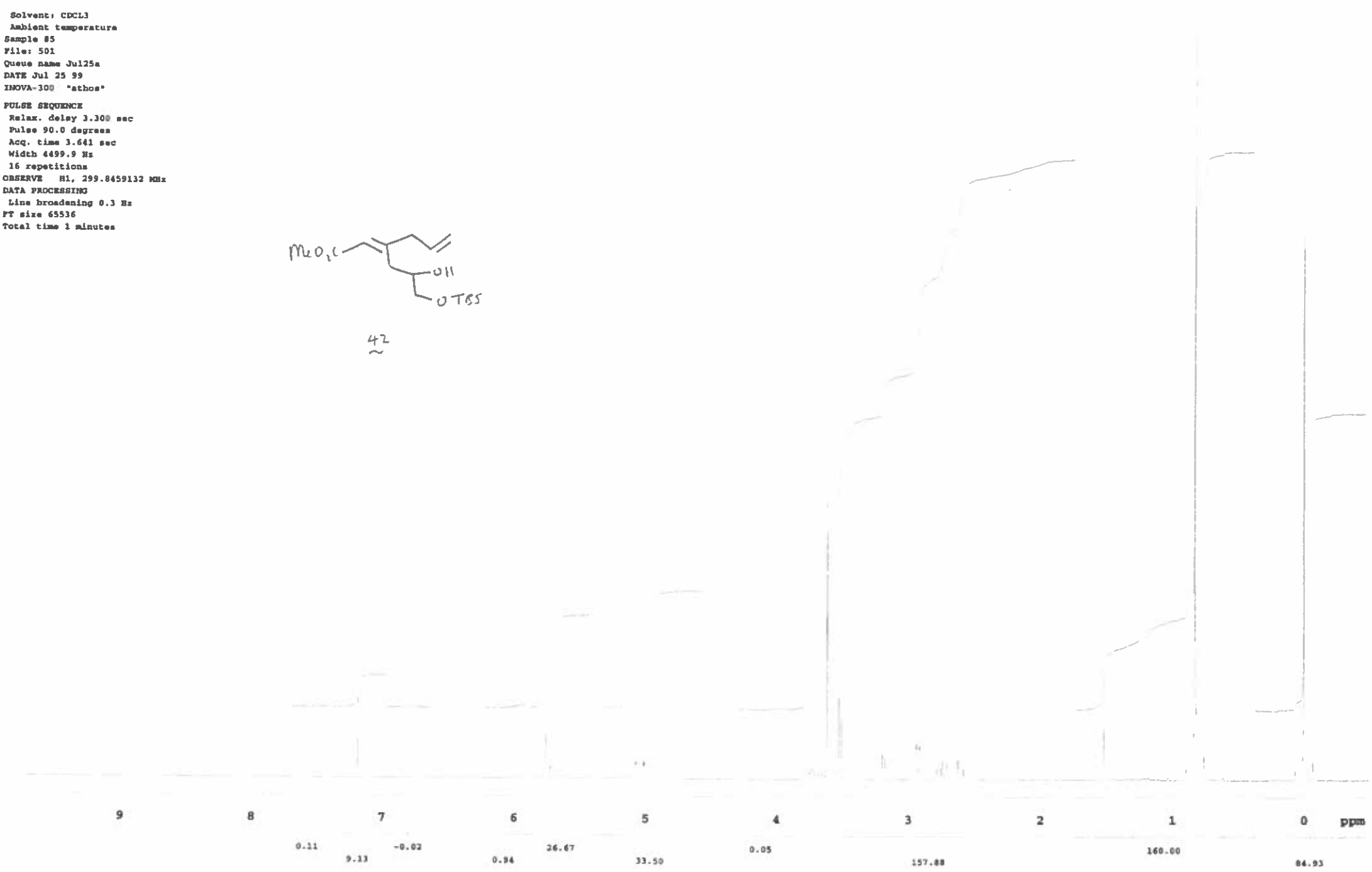


MATTHEW O'BRIEN MOB2/039/A
STANDARD 1H OBSERVE

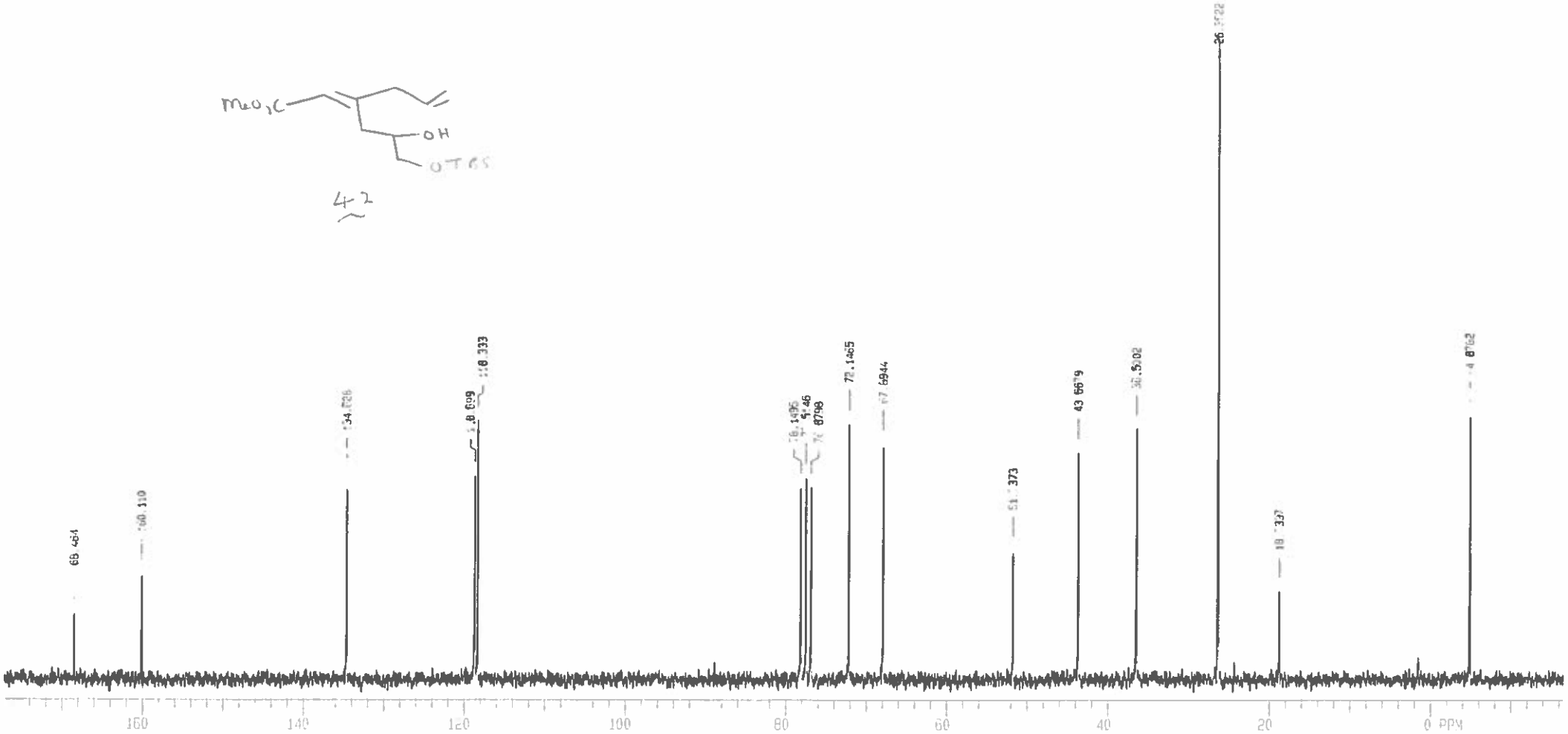
Solvent: CDCL3
Ambient temperature
Sample 85
File: 501
Queue name Jul25a
DATE Jul 25 99
INOVA-300 "athos"

PULSE SEQUENCE
Relax. delay 3.300 sec
Pulse 90.0 degrees
Acq. time 3.641 sec
Width 4499.9 Hz
16 repetitions
OBSERVE H1, 299.8459132 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 minutes

42



ALLYL-ENONE-ALCOHOL



bbb cuprate addition
STANDARD IN OBSERVE

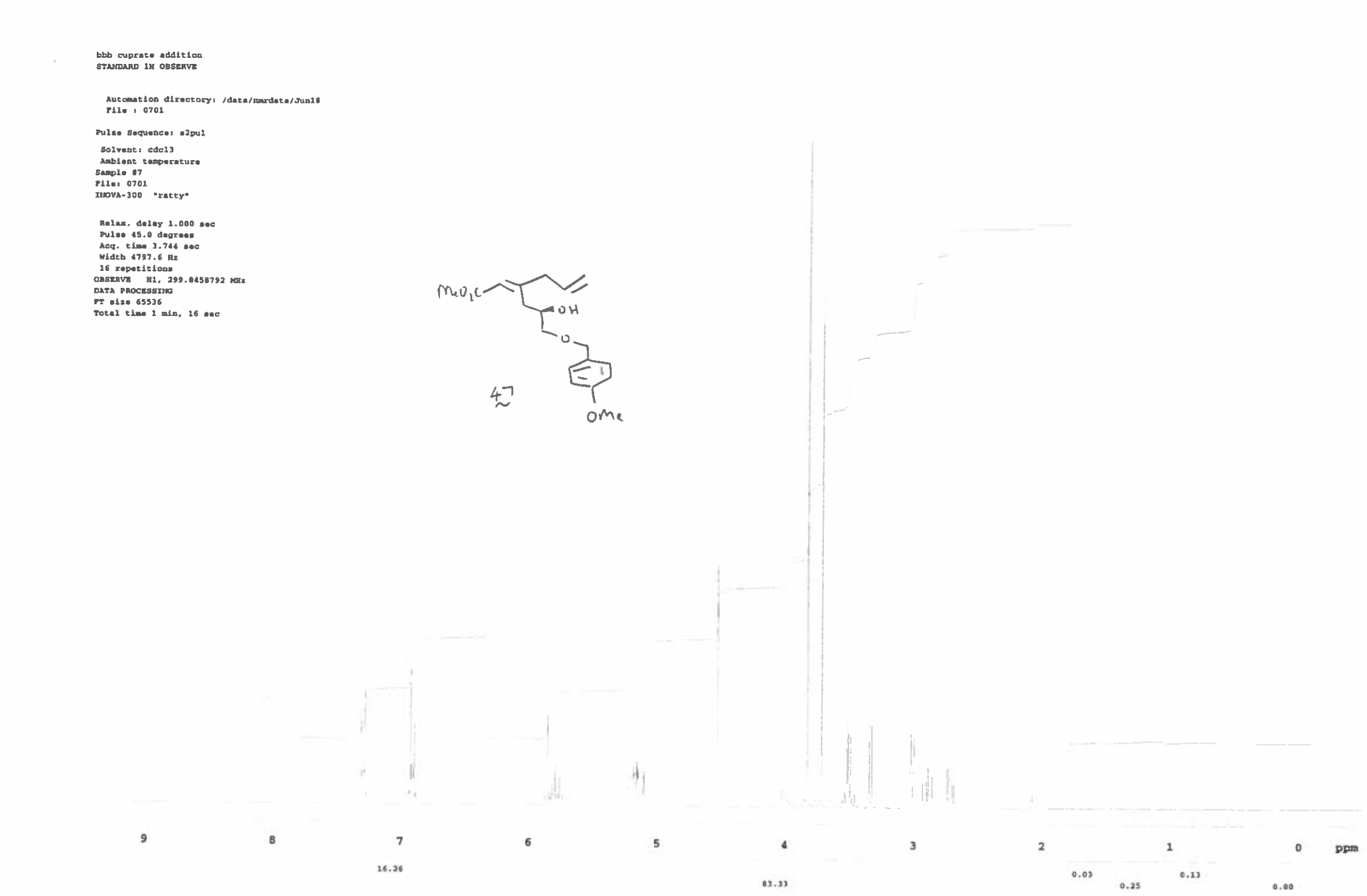
Automation directory: /data/nmrdata/Jun18
File : 0701

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample #7
File: 0701
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec

47



bbb cuprate addition
STANDARD 1H OBSERVE

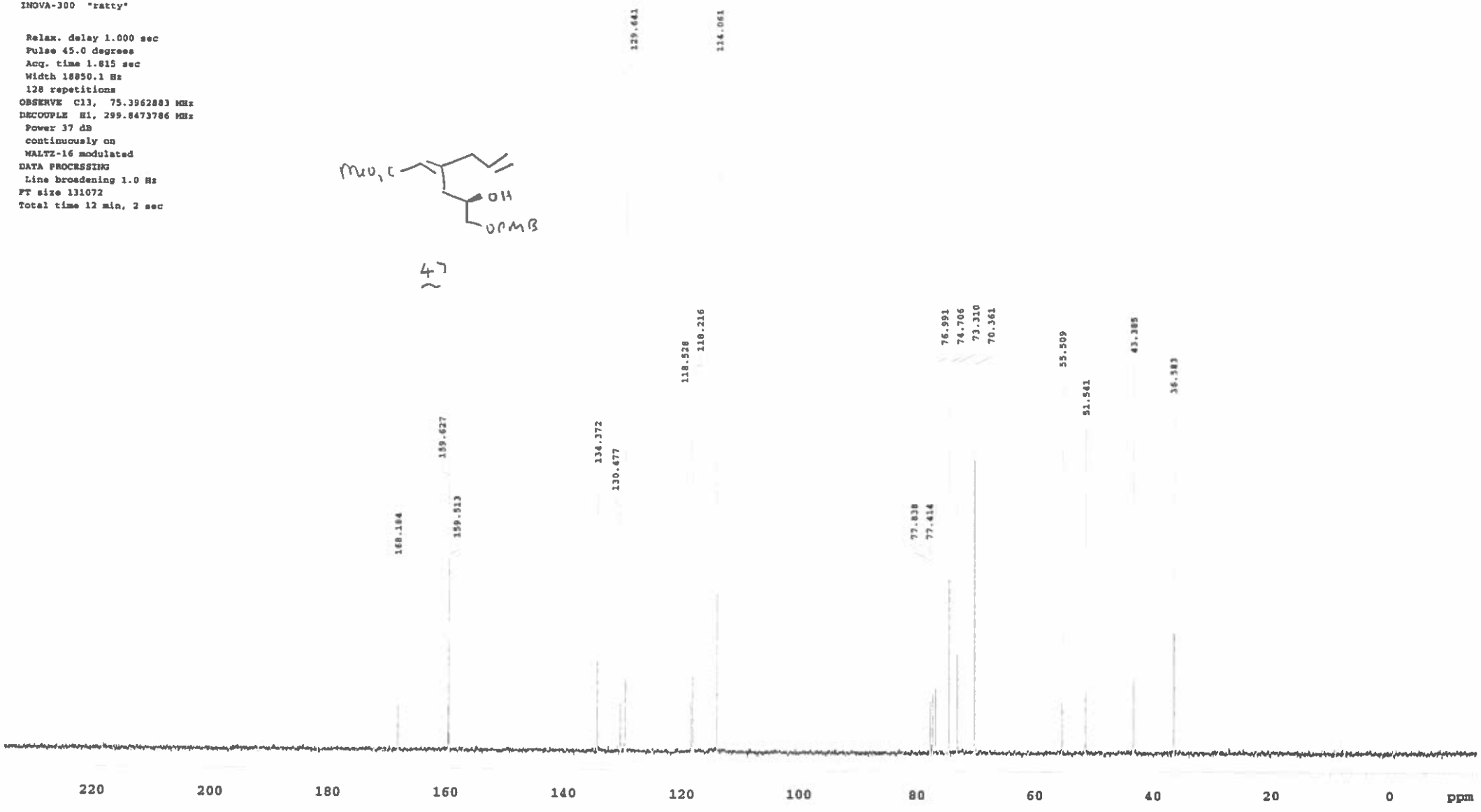
Automation directory: /data/nmrdata/Jun18
File : 0702

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample #7
File: 0702
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
128 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec

47



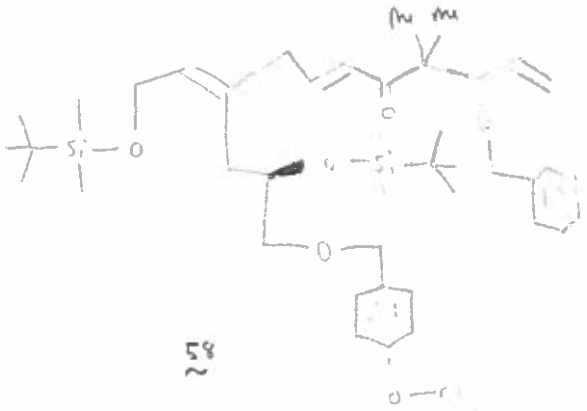
bbb phosphonate coupling product phase 1 series
STANDARD 1H OBSERVE

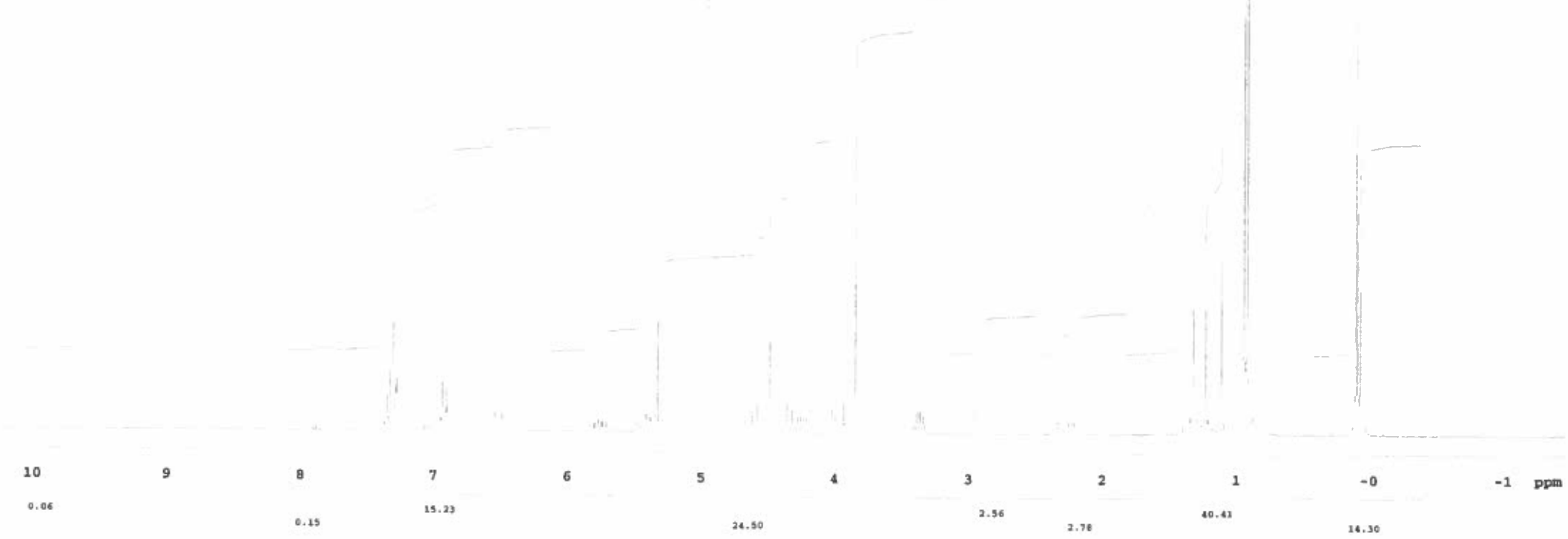
Automation directory: /data/nmrdata/Jun17
File : 4502

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample 865
File: 4502
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 3827.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 16 sec





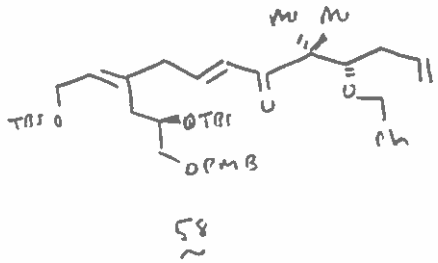
bbb phosphonate coupling product phase 1 series
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jun17
File : 4503

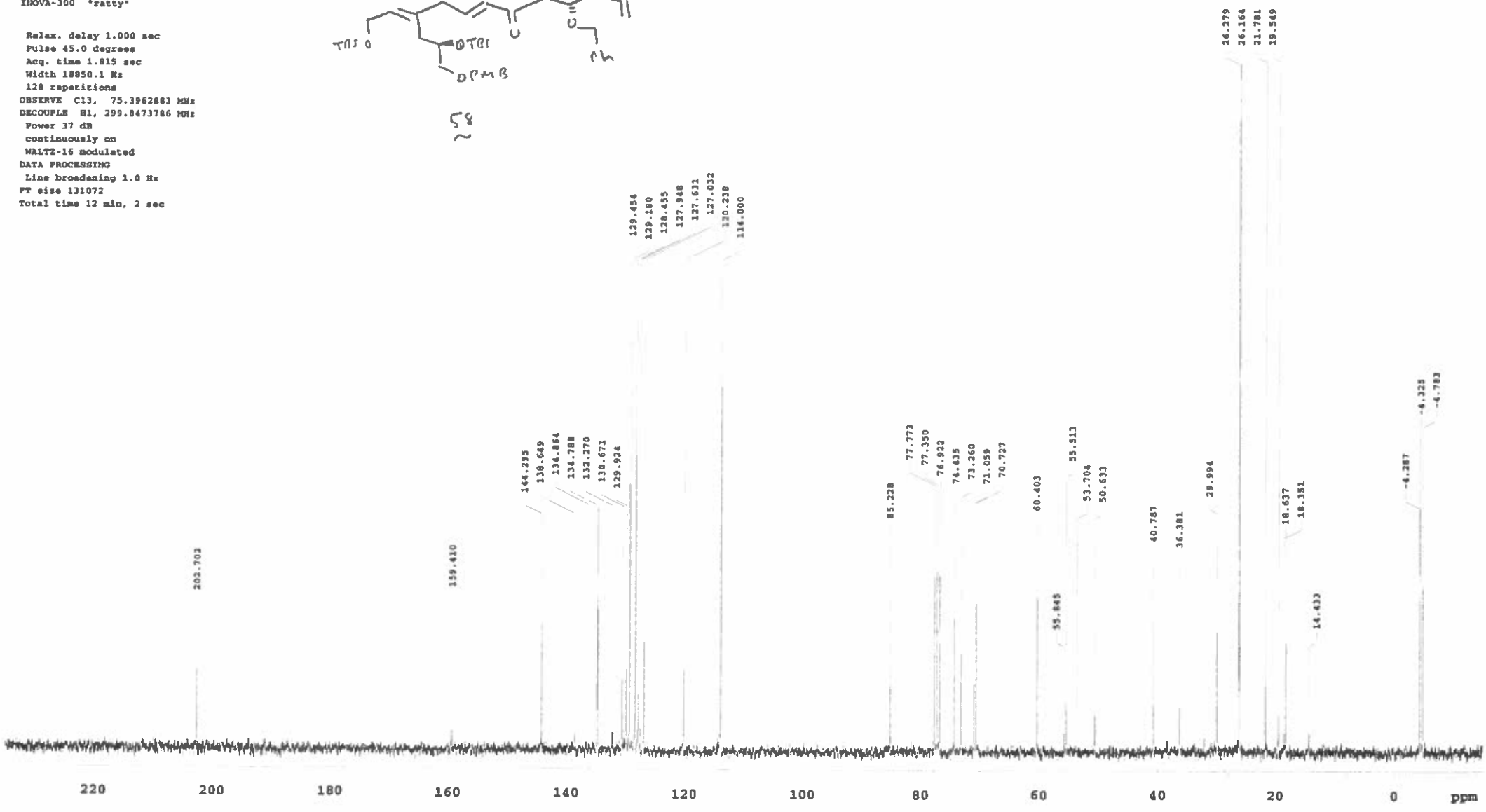
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample 845
File: 4503
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
128 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



58



abl 190/1 [a]
STANDARD 1H OBSERVE

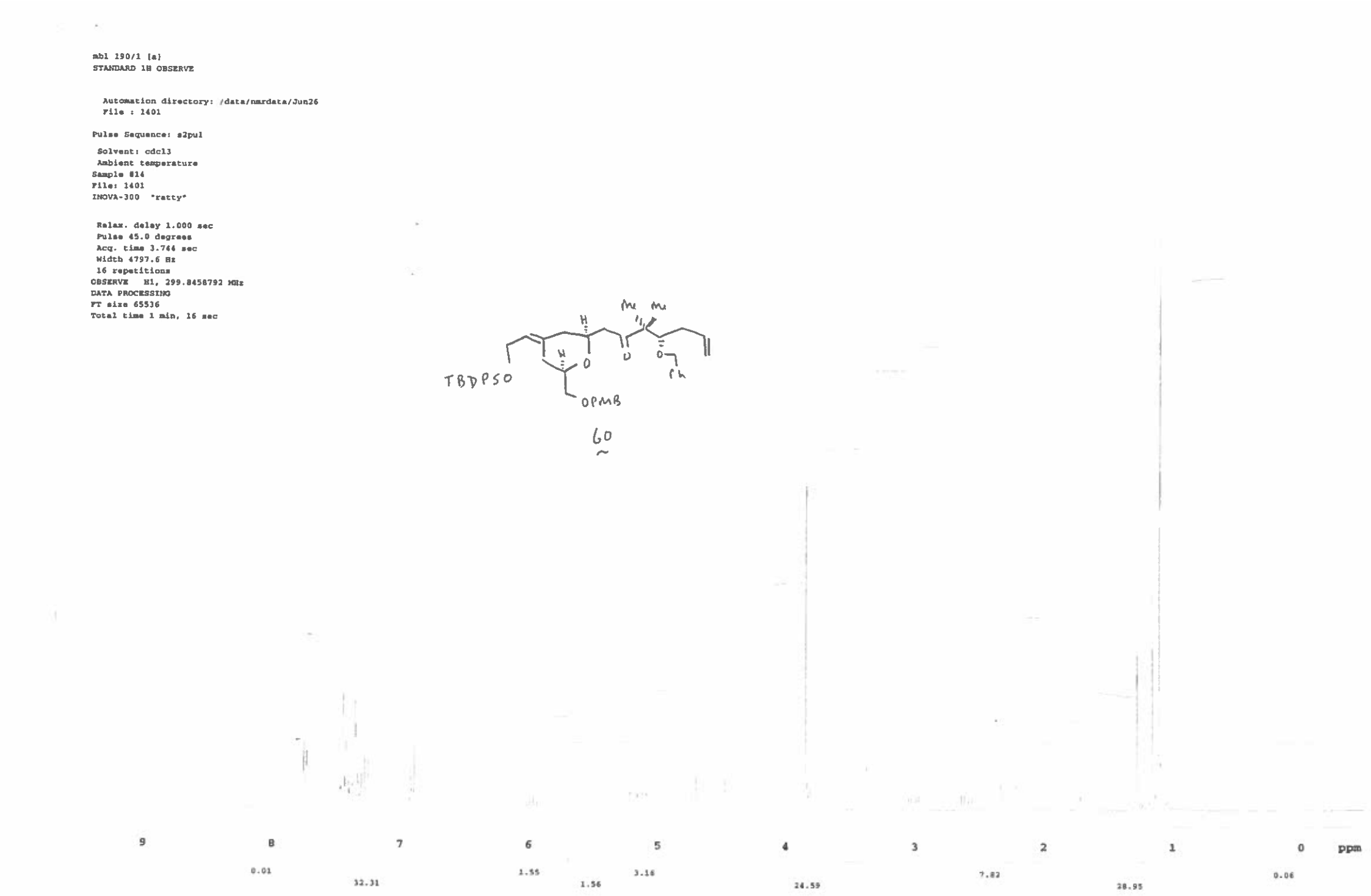
Automation directory: /data/nmrdata/Jun26
File : 1401

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample #14
File: 1401
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec

60



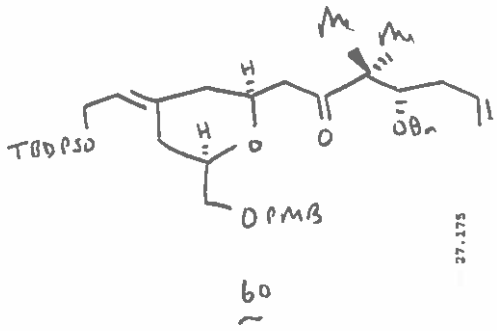
abl 190/1 [a]
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jun26
File : 1503

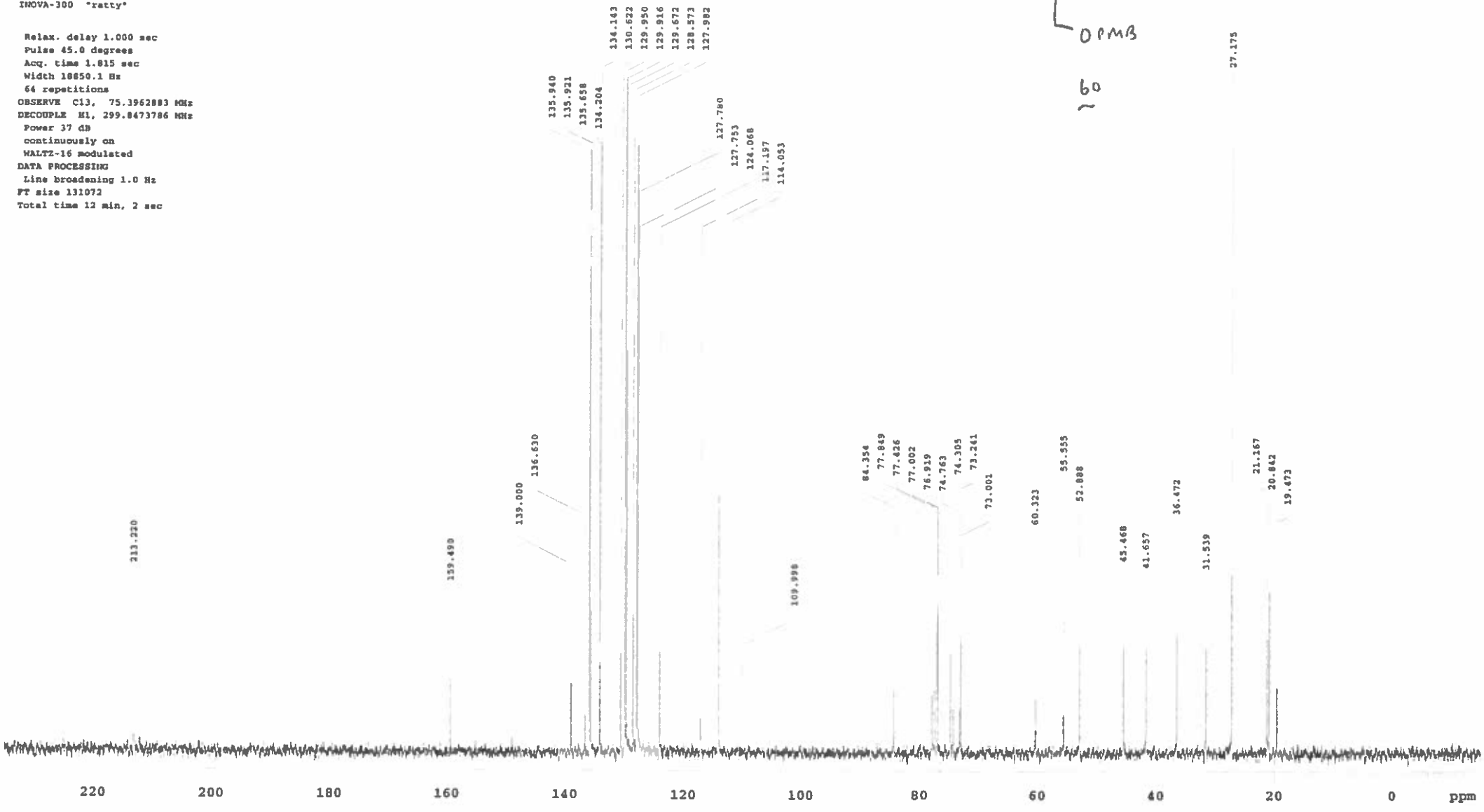
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample 815
File: 1503
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18650.1 Hz
64 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



60



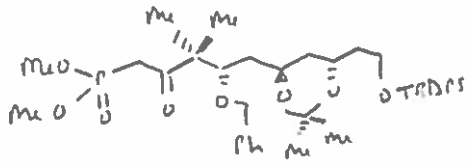
mbl 202/1 pure
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Aug08
File : 1502

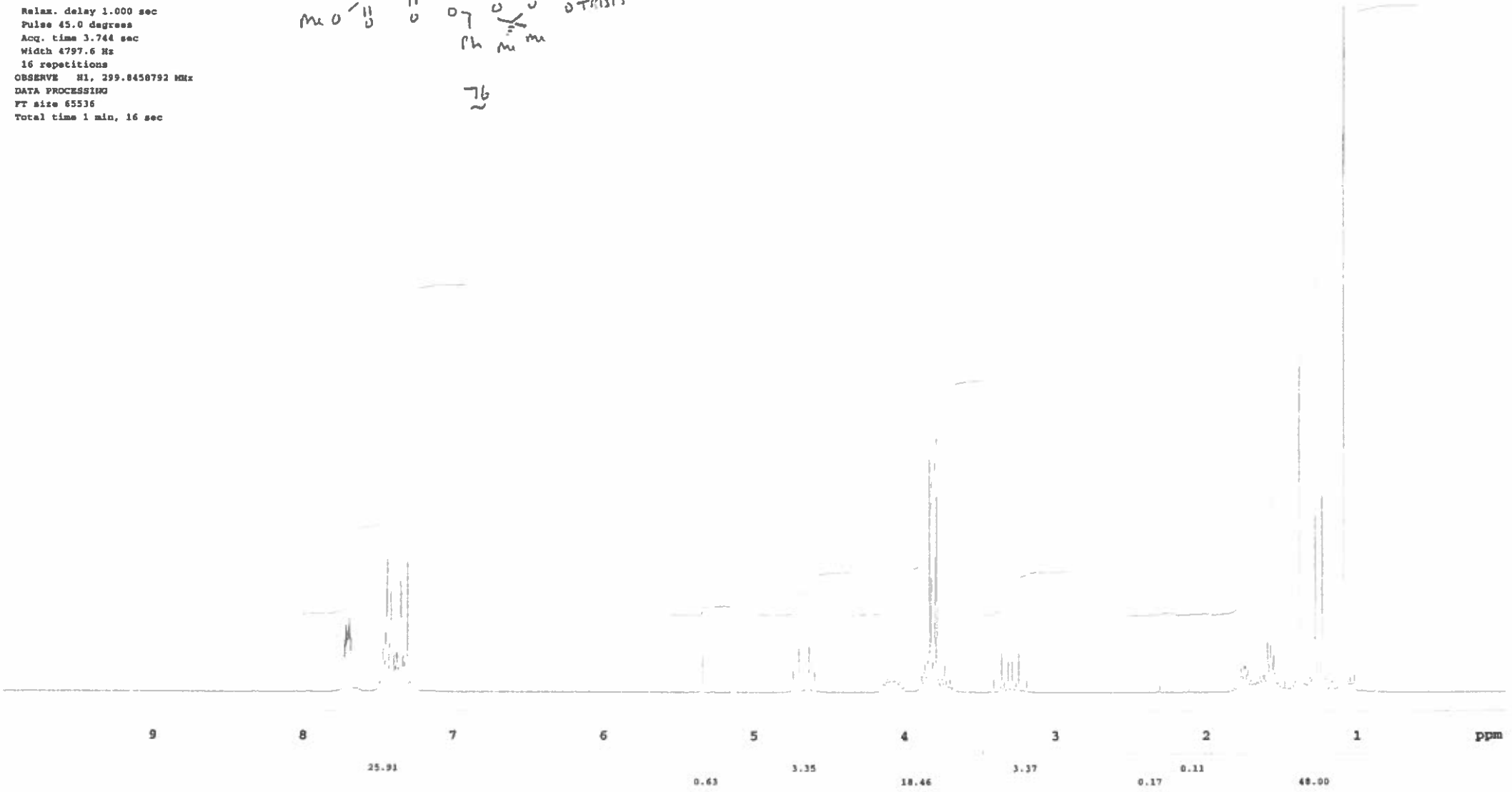
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample #15
File: 1502
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec



76



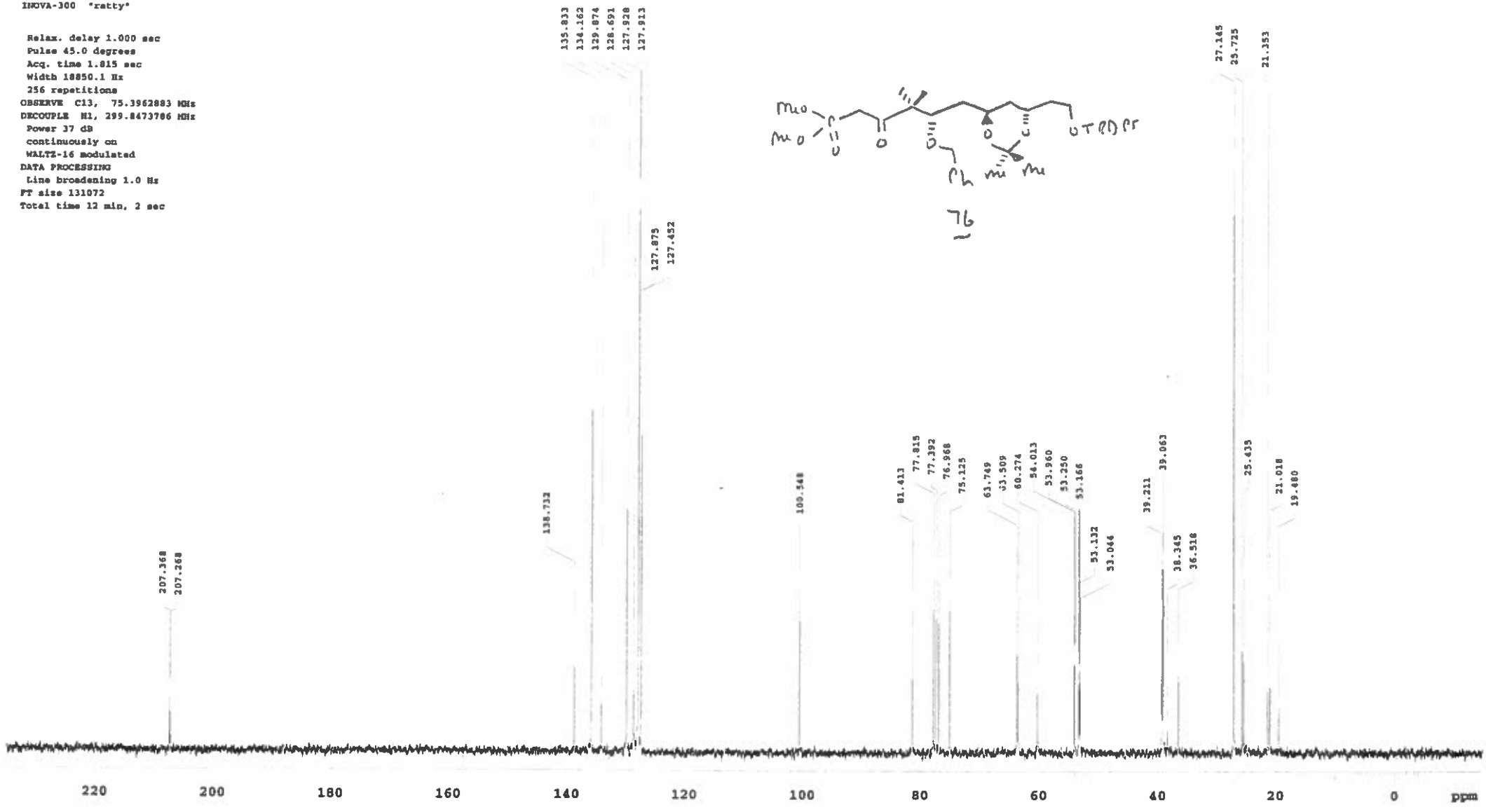
mbl 202/1 pure
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Aug11
File : 2803

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample #28
File: 2803
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
256 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



mbl 213/1 pure
STANDARD 1H OBSERVE

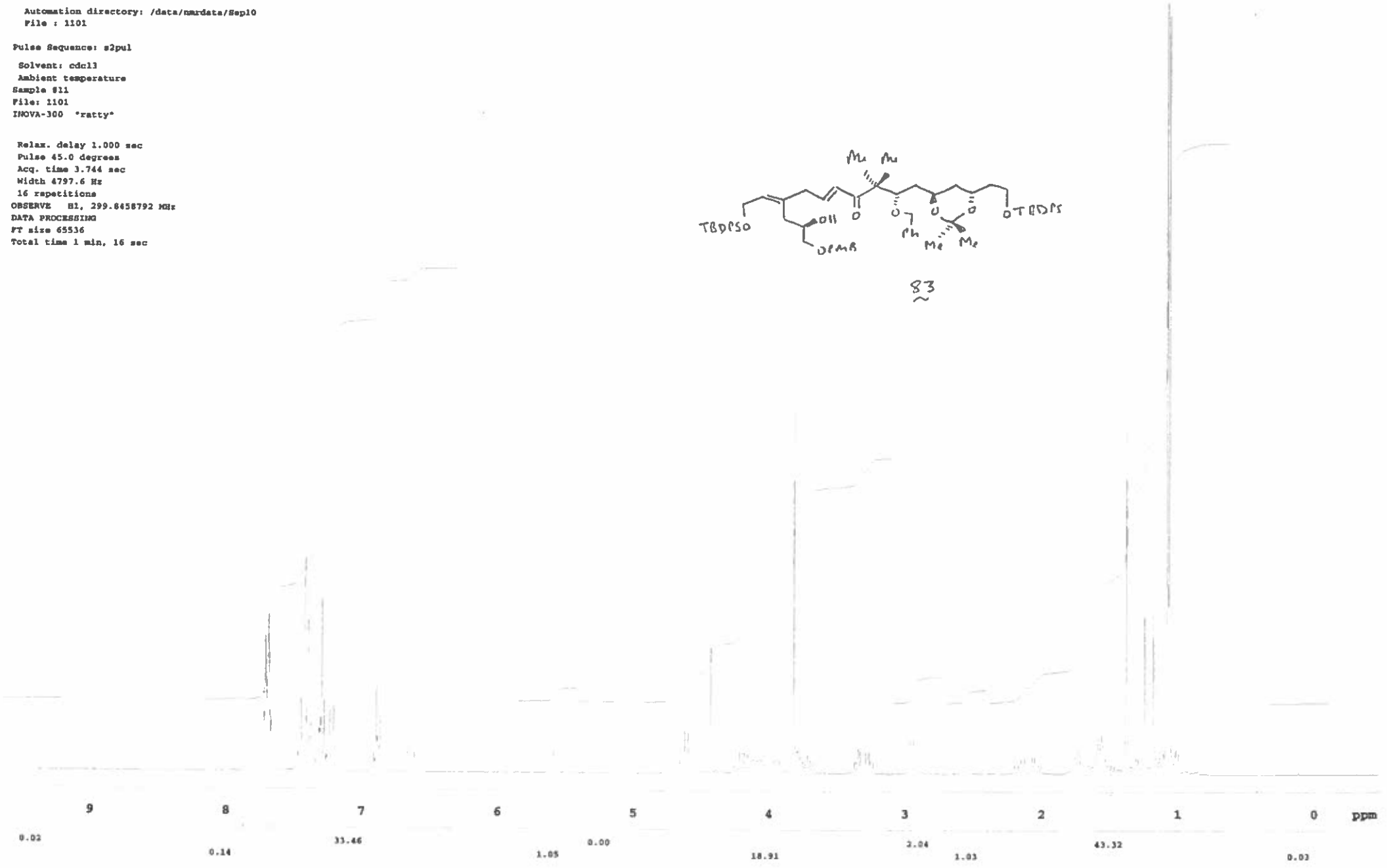
Automation directory: /data/nmrdata/Sep10
File : 1101

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample 811
File: 1101
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec

83



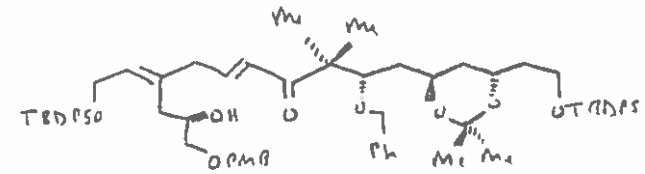
mbl 213
STANDARD 1H OBSERVE

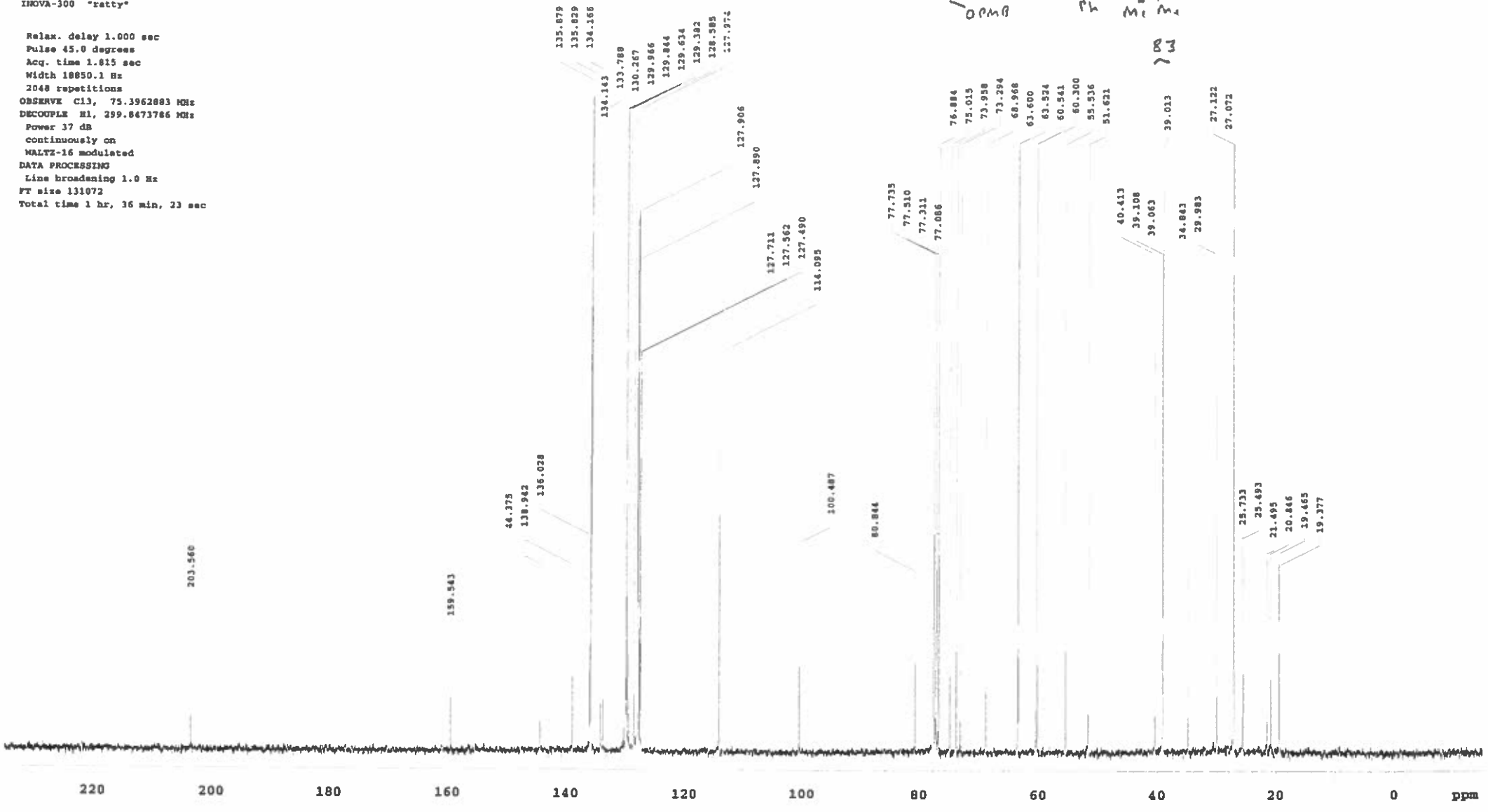
Automation directory: /data/nmrdata/Sep11
File : 0704

Pulse Sequence: s2pul

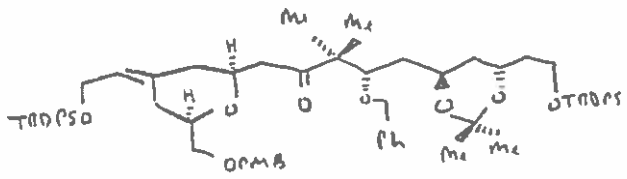
Solvent: cdcl3
Ambient temperature
Sample 87
File: 0704
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
2048 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 36 min, 23 sec

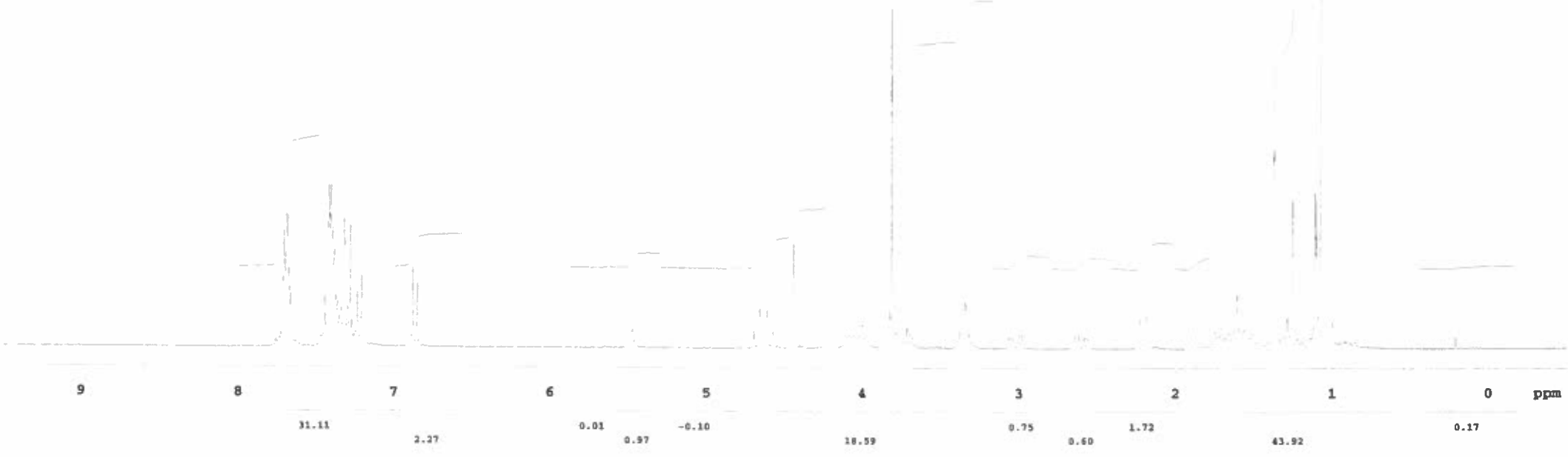




```
abl 215
STANDARD 1H OBSERVE


  Automation directory: /data/nmrdata/Sep15
  File : 2301

Pulse Sequence: s2pul

 Solvent: cdcl3
 Ambient temperature
Sample 823
File: 2301
INOVA-300  "ratty"


 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.744 sec
 Width 4797.6 Hz
 32 repetitions
OBSERVE   H1, 299.8658792 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 32 sec
```



84



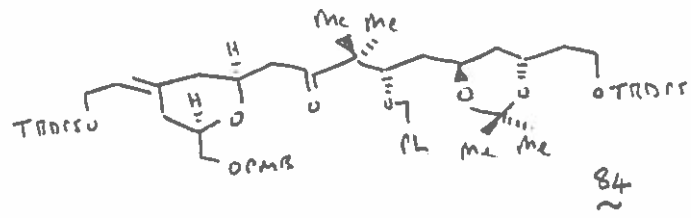
mbl 215/8 crd
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Dec03
File : 4402

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample 844
File: 4402
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
128 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



84

135.165
134.196
130.607
129.920
129.676
128.684
127.983
135.683
135.703
135.948
135.921
127.787
127.460
124.056
114.057
213.606
159.486
139.095
100.587
80.840
77.857
77.430
77.006
76.903
75.152
74.698
73.226
72.974
66.274
66.187
63.806
63.597
60.342
55.555
53.136
45.342
41.676
39.333
39.173
31.555
27.390
26.931
25.935
25.714
25.516
21.090
20.774
19.526
19.473
15.639

220 200 180 160 140 120 100 80 60 40 20 0 ppm

mbl 221/1 col2 (sample in benzene)
STANDARD 1H OBSERVE

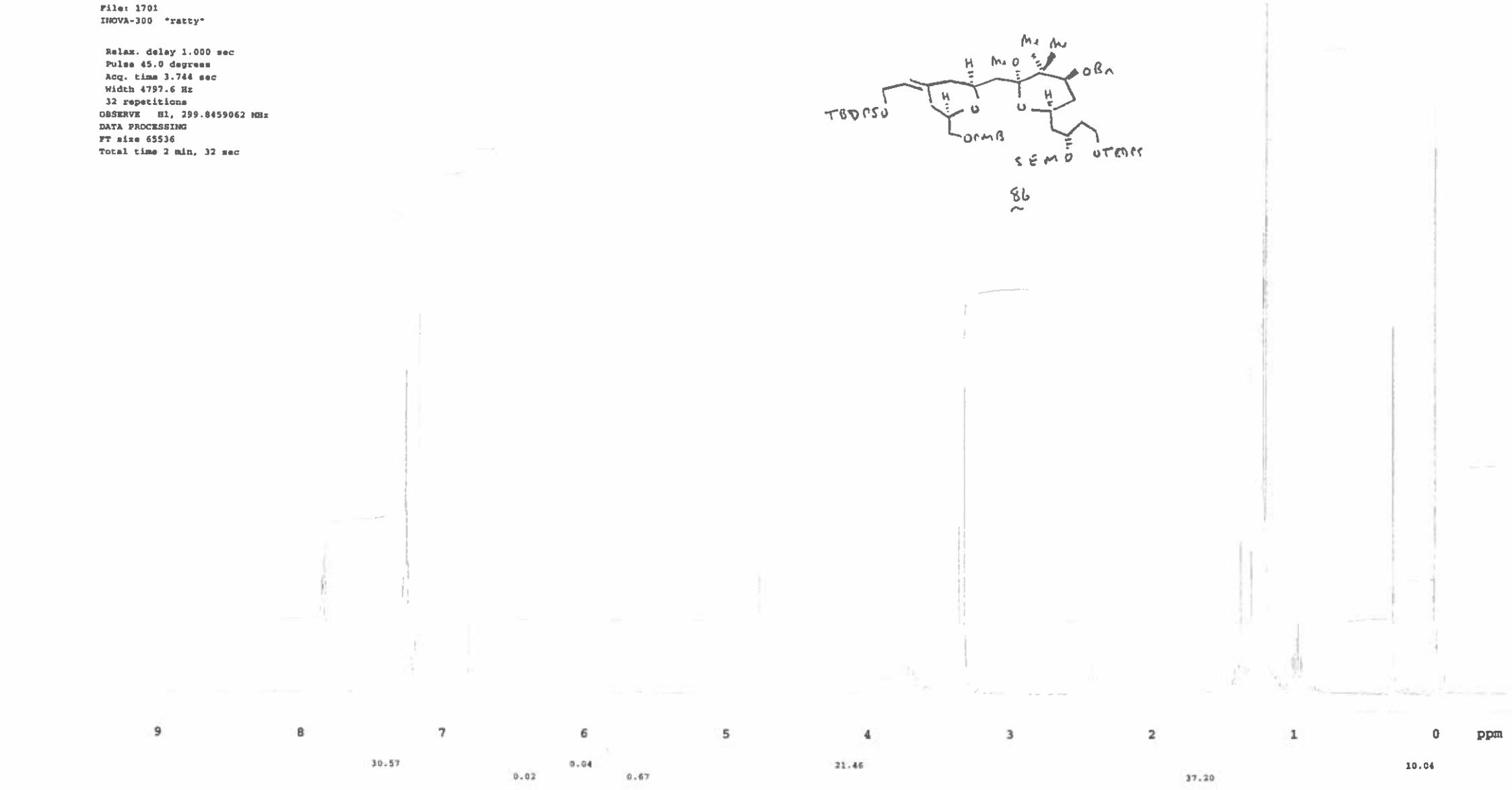
Automation directory: /data/nmrdata/Dec10
File : 1701

Pulse Sequence: s2pul

Solvent: C6D6
Ambient temperature
Sample #17
File: 1701
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
32 repetitions
OBSERVE H1, 299.8459062 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 32 sec

86



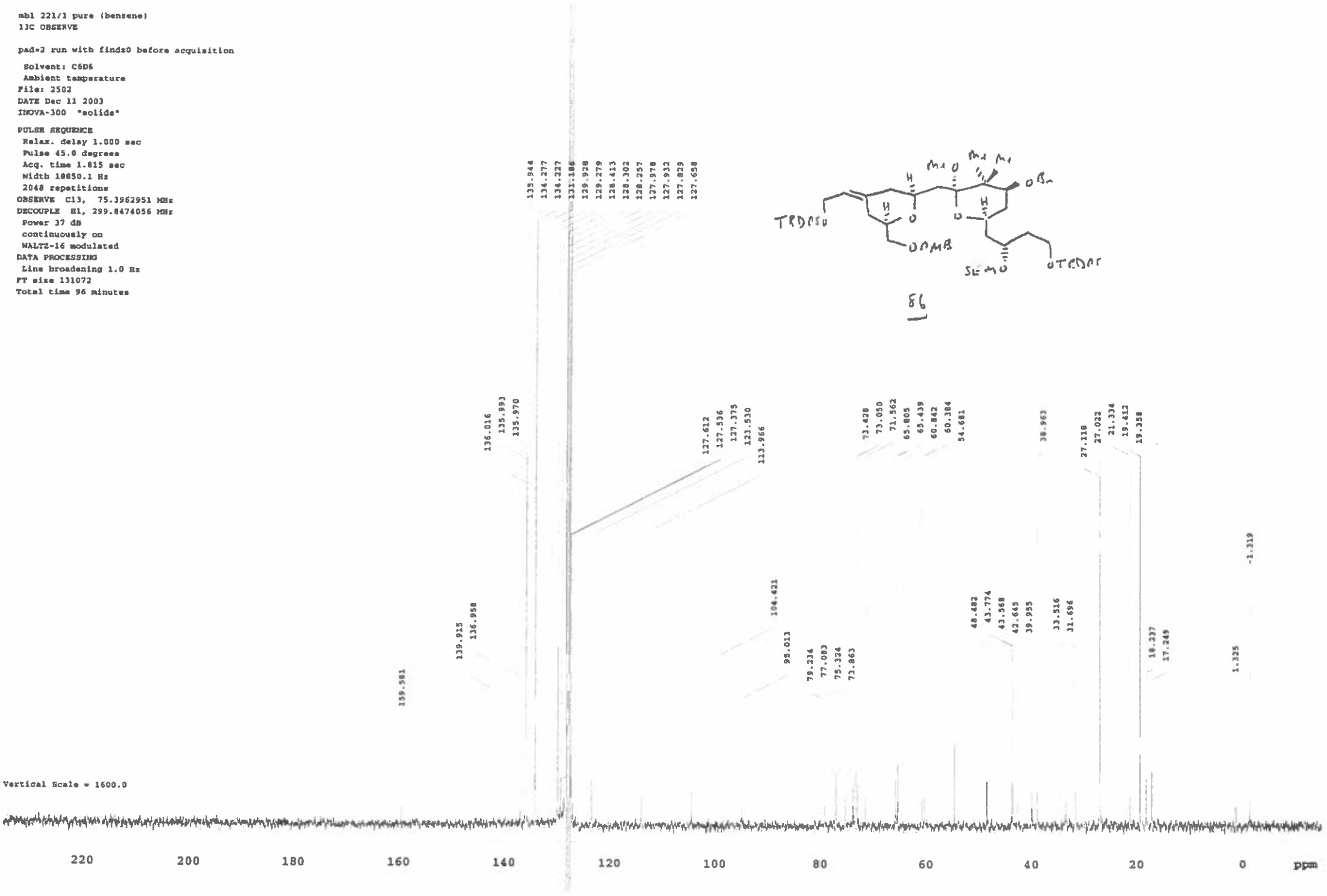
mbl 221/1 pure (benzene)
13C OBSERVE

pad=2 run with finds0 before acquisition

Solvent: C6D6
Ambient temperature
File: 2502
DATE Dec 11 2003
INOVA-300 "solids"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18650.1 Hz
2048 repetitions
OBSERVE C13, 75.3962951 MHz
DECOUPLE H1, 299.8474056 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 96 minutes

86



mbl 222/2 crd
STANDARD 1H OBSERVE

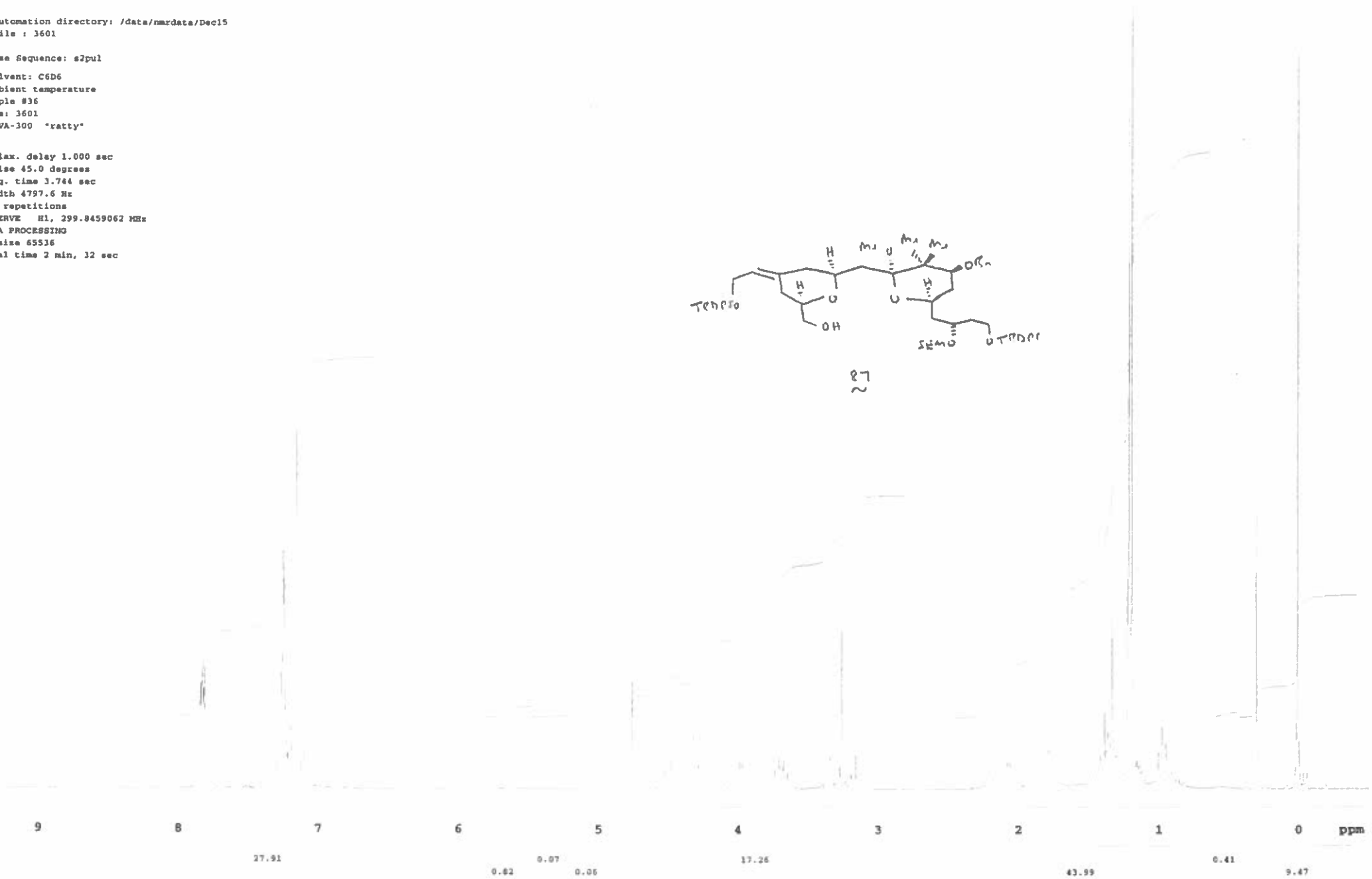
Automation directory: /data/nmrdata/Dec15
File : 3601

Pulse Sequence: s2pul

Solvent: C6D6
Ambient temperature
Sample #36
File: 3601
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
32 repetitions
OBSERVE H1, 299.8459062 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 32 sec

87



mbl 222/4 pure
13C OBSERVE

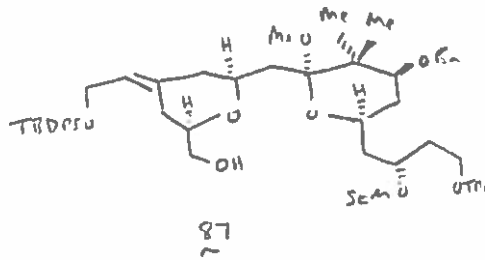
pad=2 run with finds=0 before acquisition

Automation directory: /data/nmrdata/Dec16
File : 1303

Pulse Sequence: s2pul

Solvent: C6D6
Ambient temperature
Sample 813
File: 1303
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
2048 repetitions
OBSERVE C13, 75.3962951 MHz
DECOUPLE H1, 299.8474056 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 36 min, 23 sec



135.959
135.925
134.239
134.212
134.185
134.159
130.019
129.973
129.916
128.287
128.043
127.967
127.643
135.994
127.532
127.410
127.112
124.018
104.272
94.914
139.062
79.246
77.827
75.046
73.729
71.555
65.996
65.703
65.466
60.865
60.369
48.322
43.328
43.232
42.607
39.479
38.910
33.447
30.441
30.120
27.110
26.985
26.855
21.331
19.408
19.332
18.233
17.272
1.317
-1.330

220 200 180 160 140 120 100 80 60 40 20 0 ppm

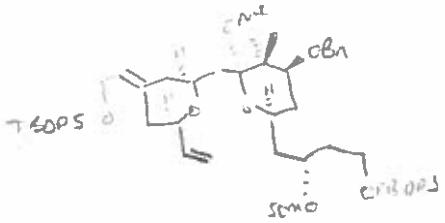
224/1

mbl 23974 pure
STANDARD 1H OBSERVE

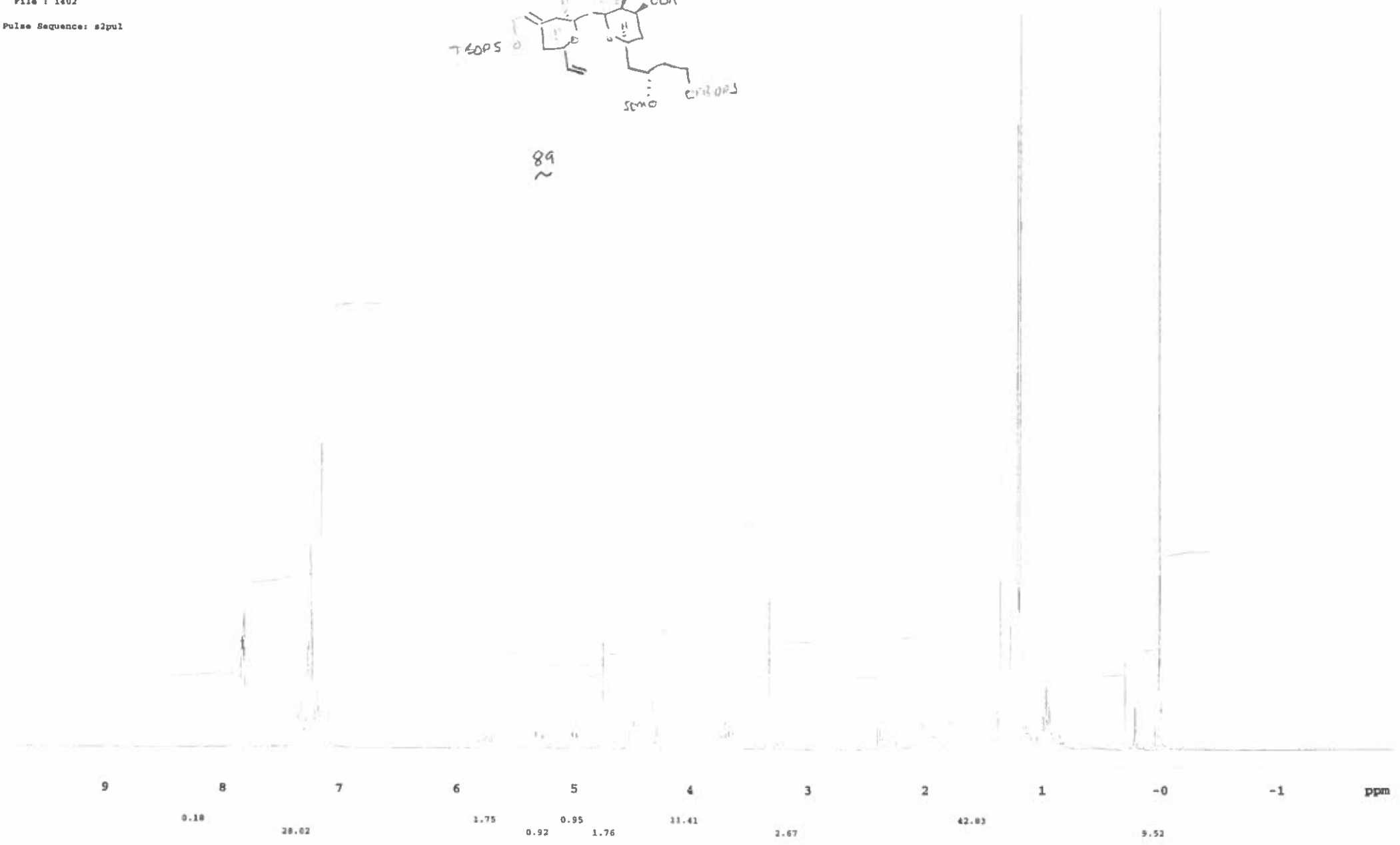
FULL spectrum

Automation directory: /data/nmrdata/Dec16
File : 1402

Pulse Sequence: s2pul



89



226/1

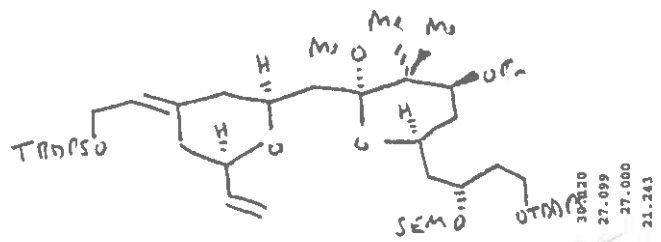
mbl 22X/4 pure
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Dec16
File : 1404

Pulse Sequence: s2pul

Solvent: C6D6
Ambient temperature
Sample #14
File: 1404
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
2048 repetitions
OBSERVE C13, 75.3962951 MHz
DECOUPLE H1, 299.8474056 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 36 min, 23 sec



~89

135.978
135.959
135.932
134.208
129.920
128.283
128.032
127.963
127.639
136.009
127.534
127.372
123.484
114.111
104.421
94.994
139.900
139.263
136.856
79.219
77.674
75.133
73.806
71.562
65.768
65.436
60.842
60.350
48.440
46.643
43.534
42.695
39.071
38.971
34.931
33.504
30.820
27.099
27.000
21.243
19.400
19.343
18.229
17.203
12.259
1.314
-1.339

220 200 180 160 140 120 100 80 60 40 20 0 ppm

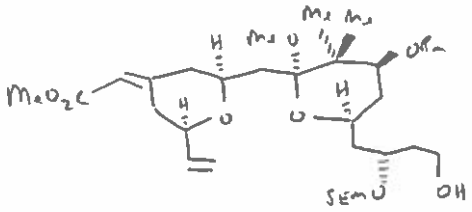
H115
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jun15
File : 0202

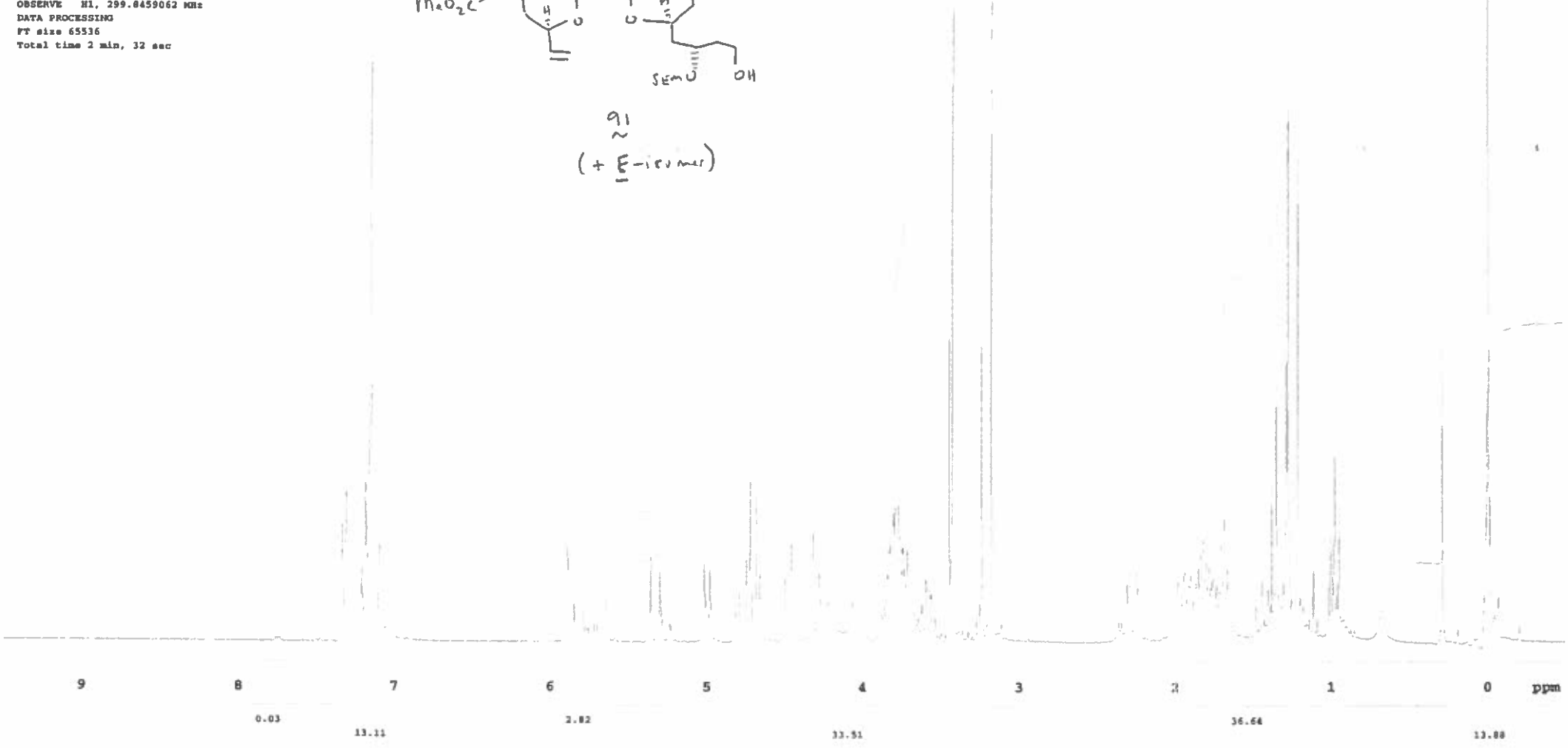
Pulse Sequence: s2pul

Solvent: C6D6
DATE Jun 15 2004
Sample #2
File: 0202
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
32 repetitions
OBSERVE H1, 299.8459062 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 32 sec



91
~
(+ E-isomer)



H115
13C OBSERVE

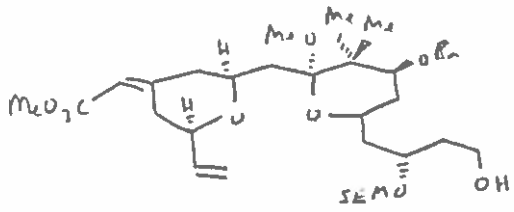
pad=2 run with findz0 before acquisition

Automation directory: /data/rmrdata/Jun16
File : 1801

Pulse Sequence: s2pul

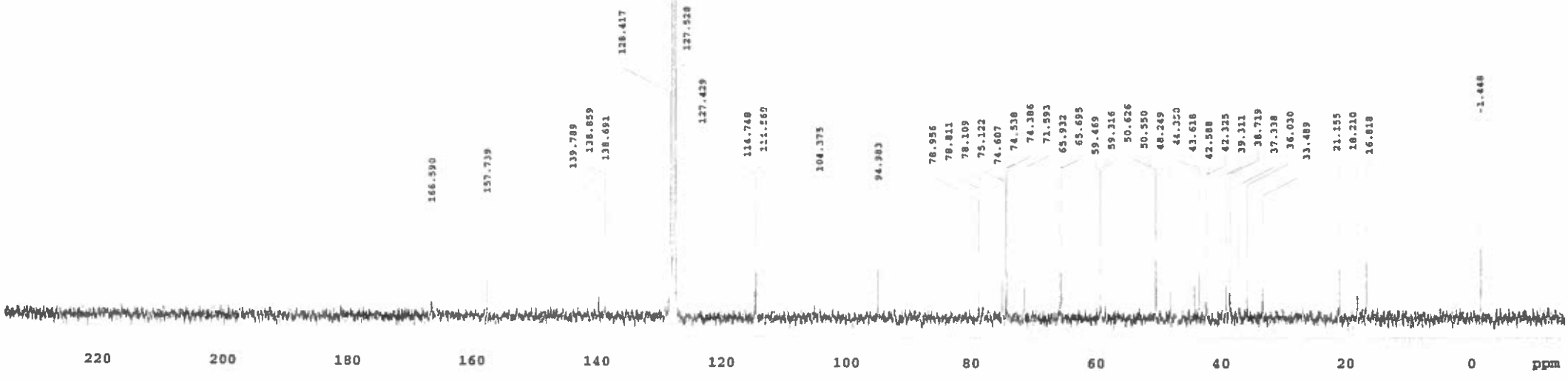
Solvent: C6D6
DATE Jun 16 2004
Sample 818
File: 1801
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
256 repetitions
OBSERVE C13, 75.3962951 MHz
DECOUPLE H1, 299.8474056 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



91

(+ ca 20% E-isomer)



rd57
STANDARD 1H OBSERVE

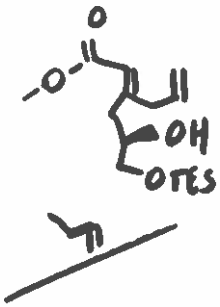
Automation directory: /data/nmrdata/Oct27
File : 1902

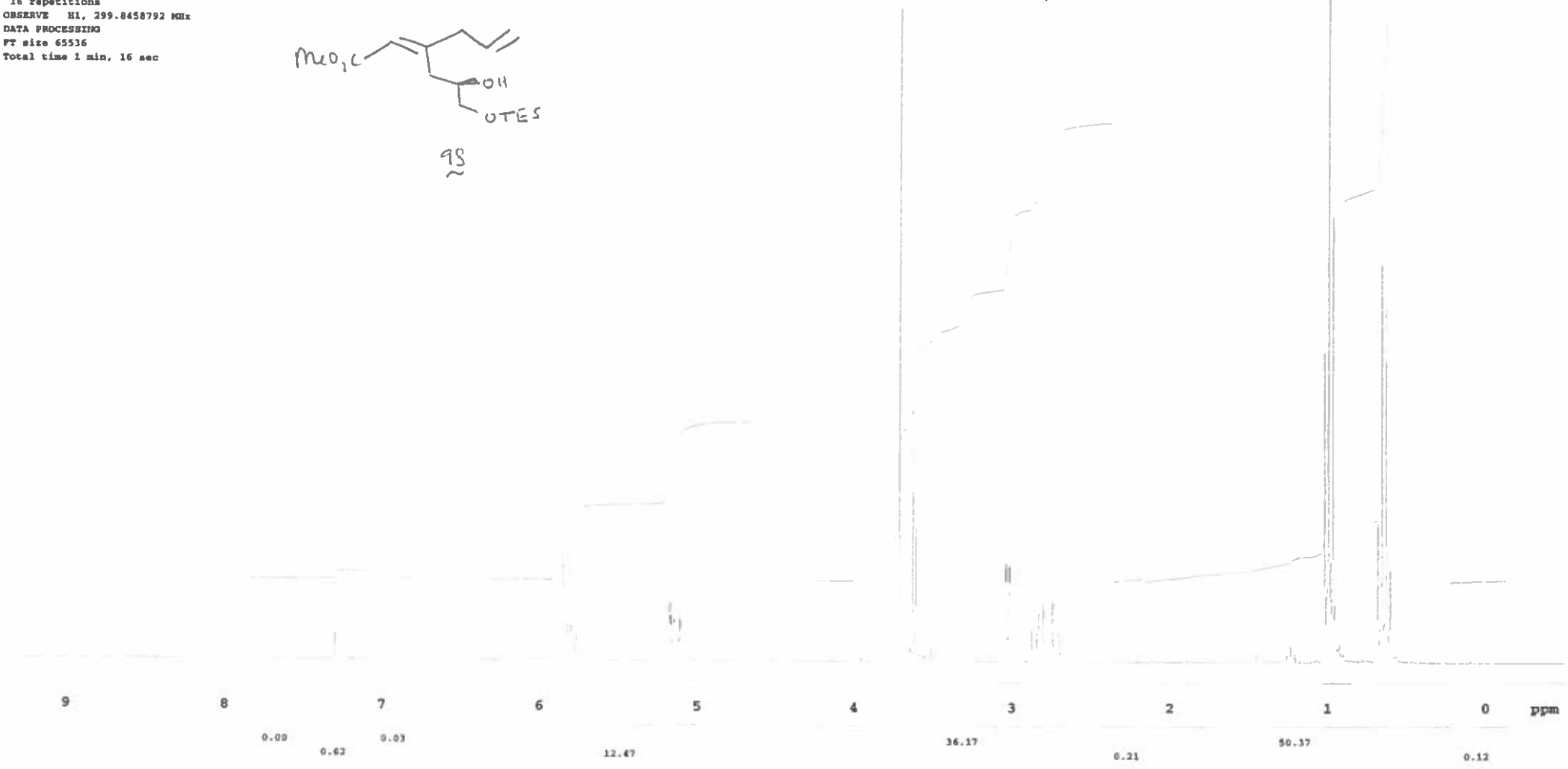
Pulse Sequence: s2pul

Solvent: cdcl3
DATE Oct 27 2004
Sample 819
File: 1902
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec

98





rd57
STANDARD 1H OBSERVE

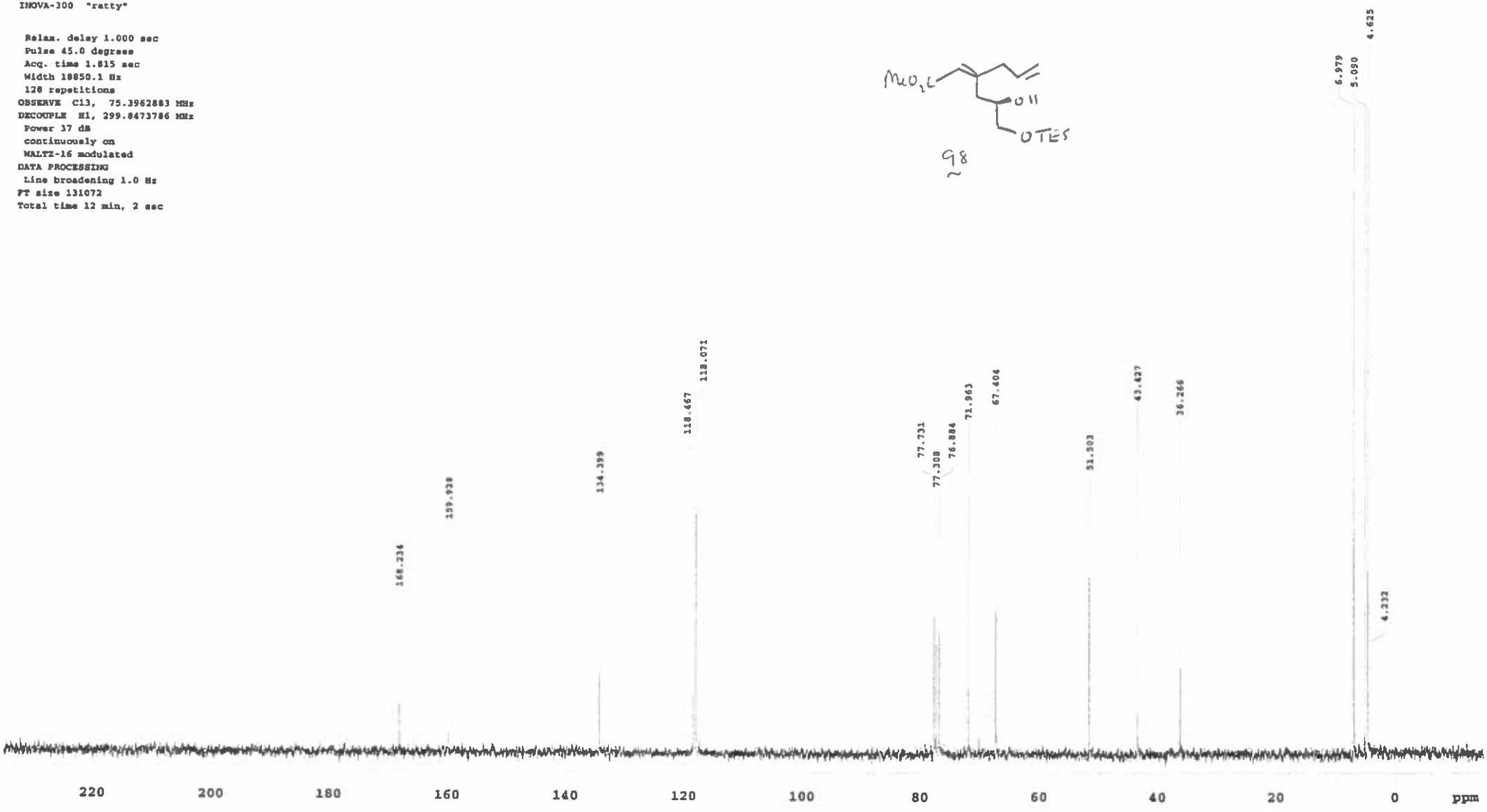
Automation directory: /data/nmrdata/Oct27
File : 1903

Pulse Sequence: s2pul

Solvent: cdcl3
DATE Oct 27 2004
Sample #19
File: 1903
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
128 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec

98



diol
STANDARD 1H OBSERVE RD42

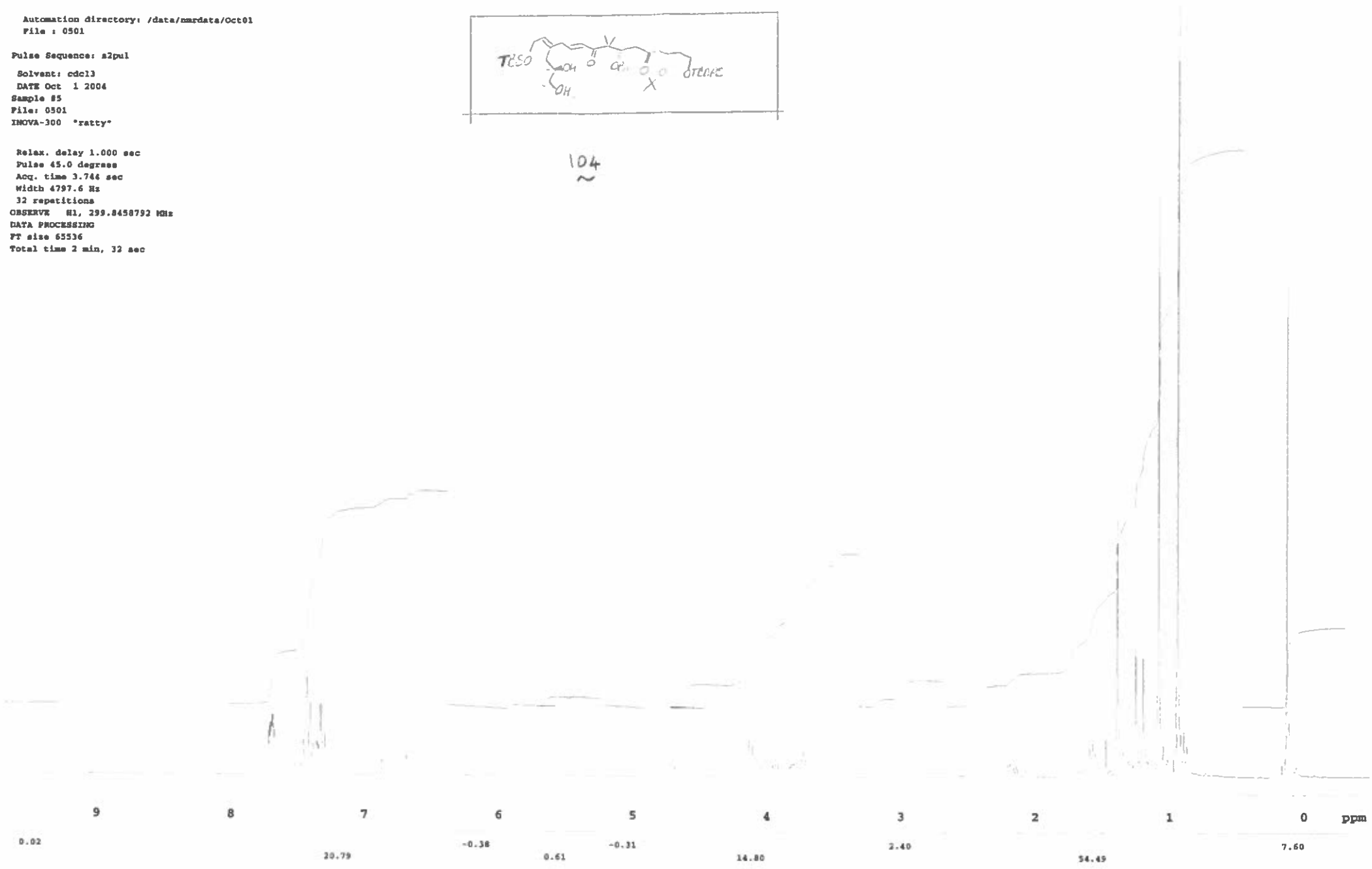
Automation directory: /data/nmrdata/Oct01
File : 0501

Pulse Sequence: s2pul

Solvent: cdcl3
DATE Oct 1 2004
Sample #5
File: 0501
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
32 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 32 sec

104



diol
STANDARD 1H OBSERVE

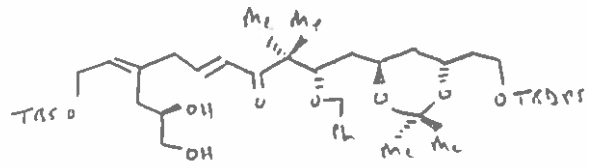
RD42

Automation directory: /data/rmrdata/Oct01
File : 0502

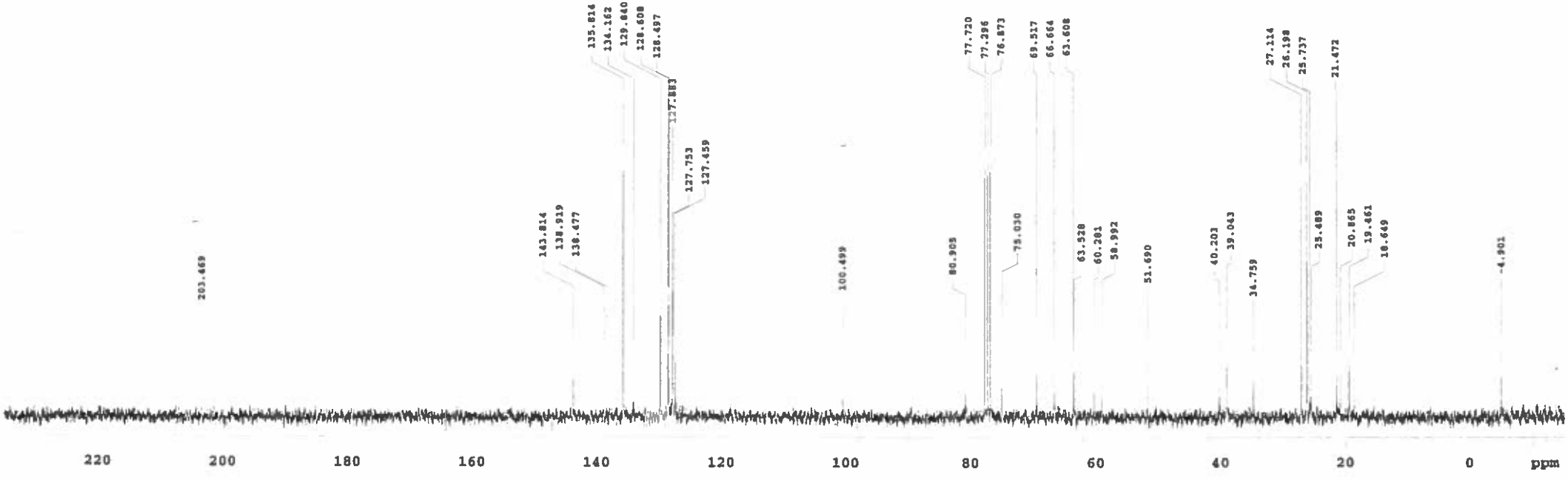
Pulse Sequence: s2pul

Solvent: cdcl3
DATE Oct 1 2004
Sample #5
File: 0502
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
256 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



104



rdfp2
STANDARD 1H OBSERVE

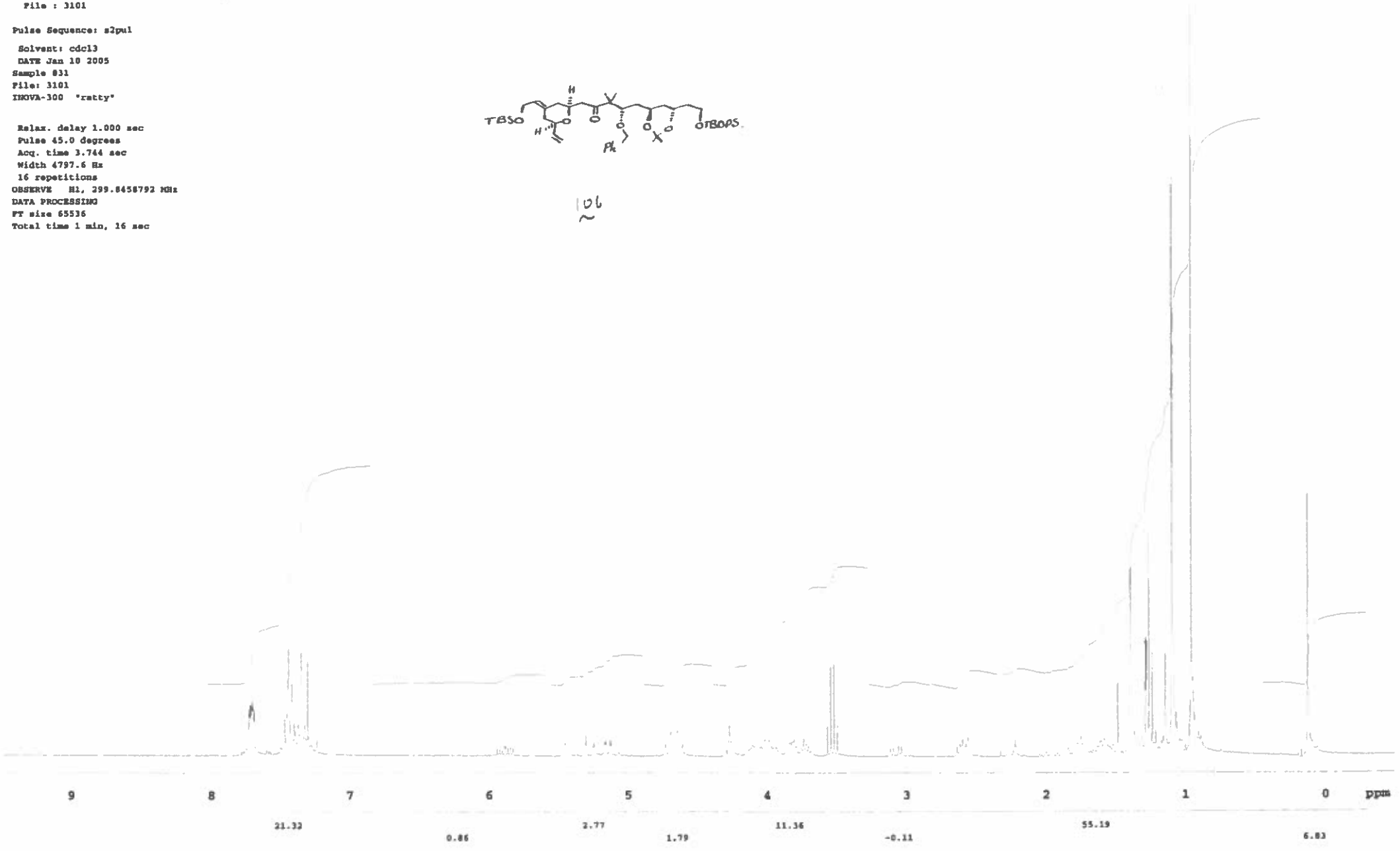
Automation directory: /data/nmrdata/Jan10
File : 3101

Pulse Sequence: s2pul

Solvent: cdcl3
DATE Jan 10 2005
Sample 831
File: 3101
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec

106



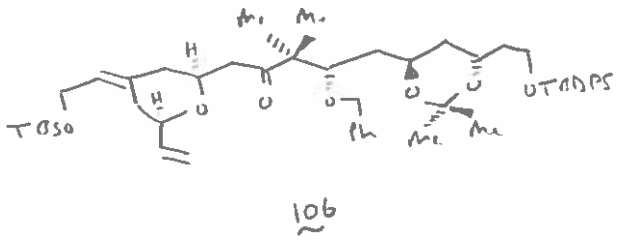
rdfp2
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jan10
File : 3102

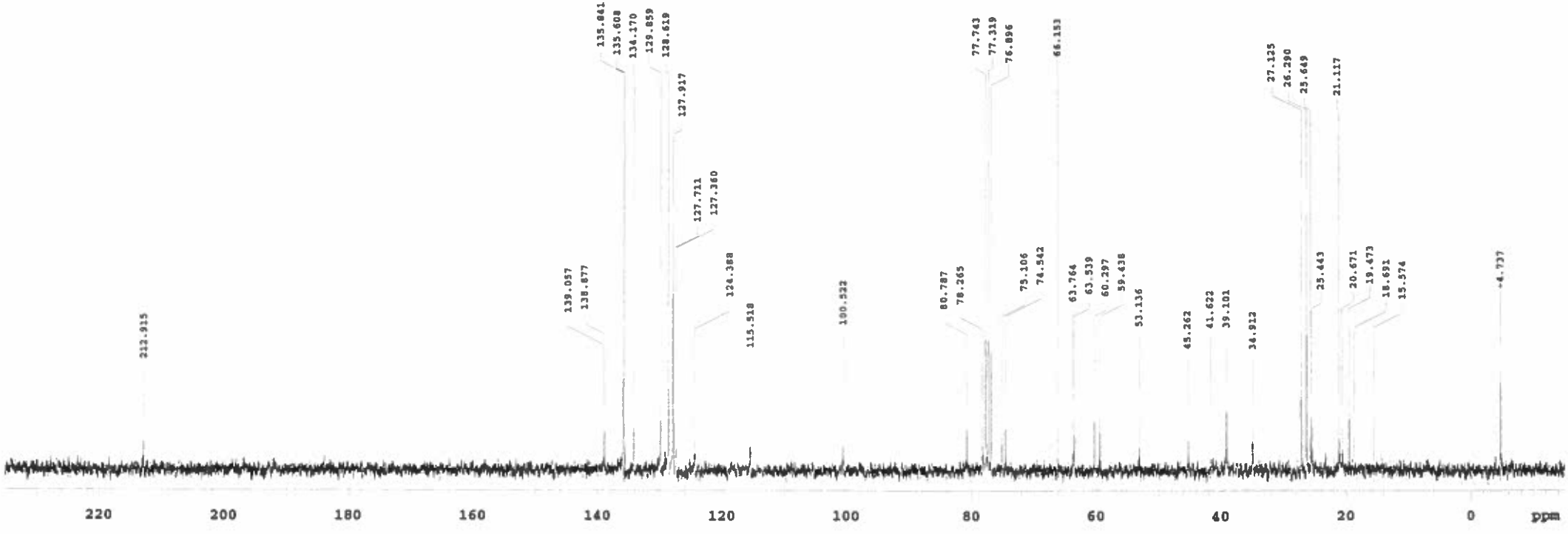
Pulse Sequence: s2pul

Solvent: cdcl3
DATE Jan 10 2005
Sample #31
File: 3102
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
256 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



106



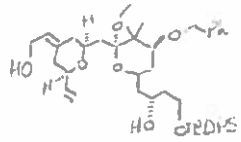
rd96
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jan13
File : 4901

Pulse Sequence: s2pul

Solvent: C6D6
DATE Jan 13 2005
Sample 849
File: 4901
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8459062 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec



107

9 8 7 6 5 4 3 2 1 0 ppm

5.33 24.42 1.24 3.88 14.72 4.86 -0.09 43.68 1.96

rd96
STANDARD 1H OBSERVE

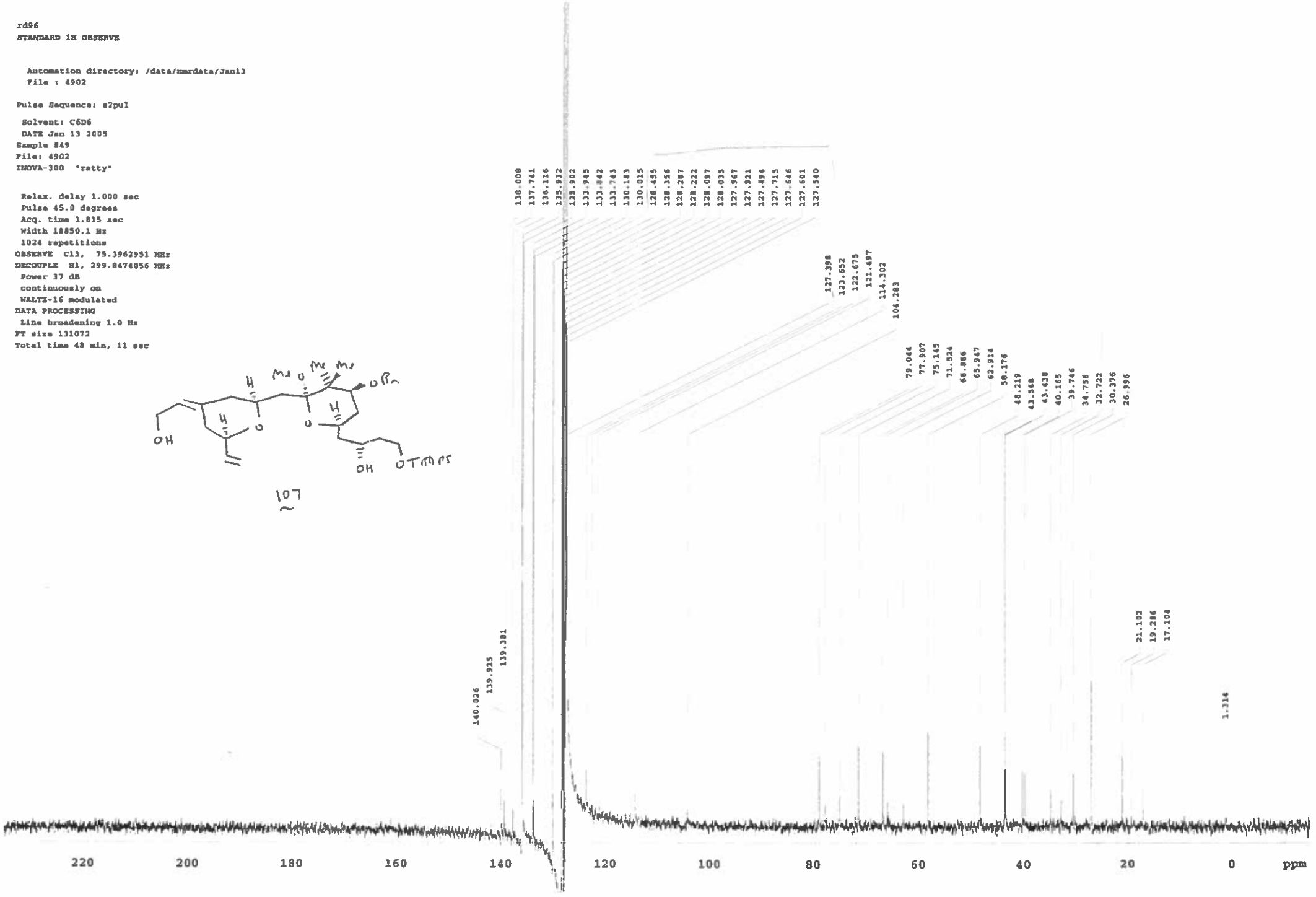
Automation directory: /data/nmrdata/Jan13
File : 4902

Pulse Sequence: s2pul

Solvent: C6D6
DATE Jan 13 2005
Sample 849
File: 4902
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
1024 repetitions
OBSERVE C13, 75.3962951 MHz
DECOUPLE H1, 299.8474056 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min, 11 sec

107



rd104p
STANDARD 1H OBSERVE

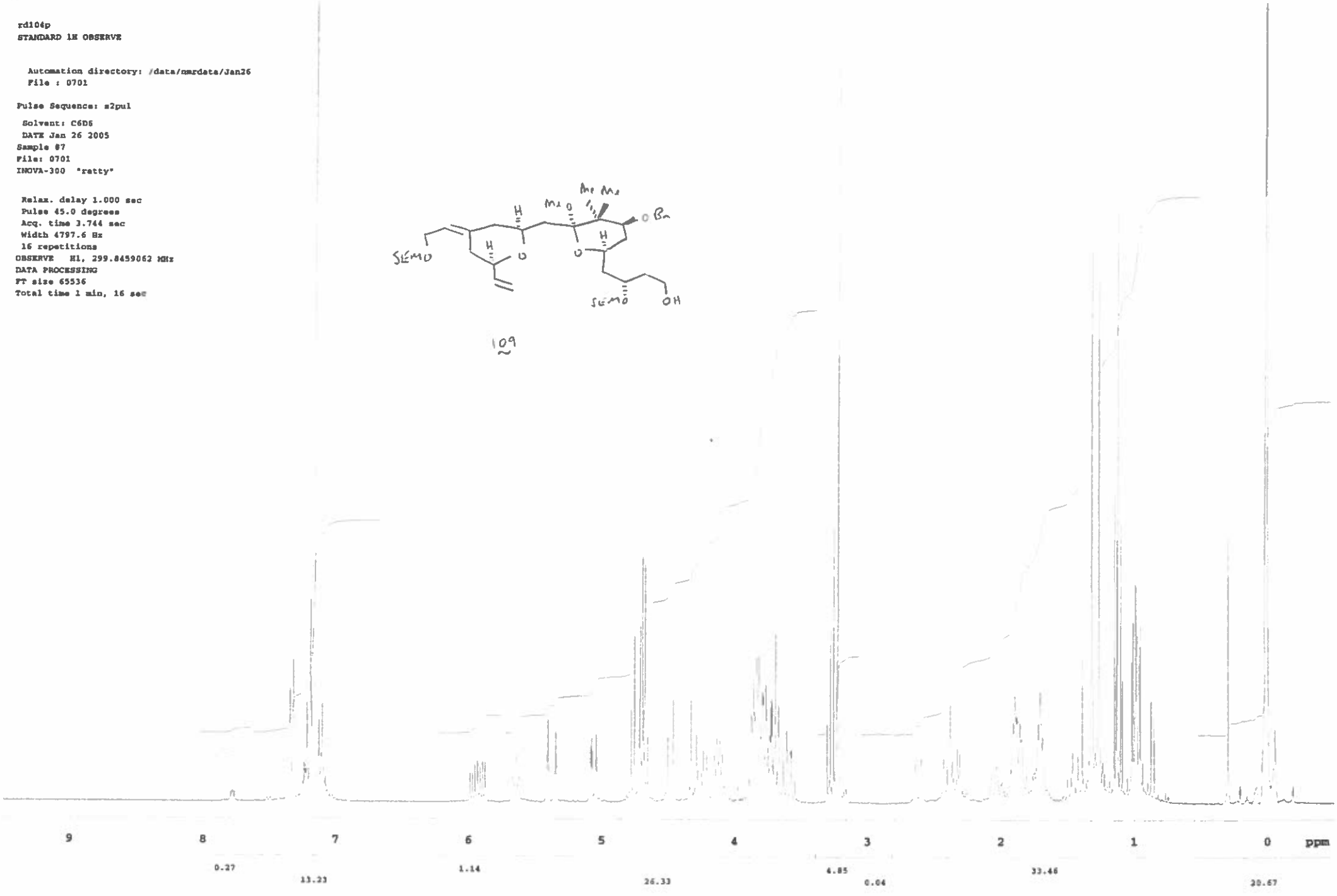
Automation directory: /data/nmrdata/Jan26
File : 0701

Pulse Sequence: s2pul

Solvent: C6D6
DATE Jan 26 2005
Sample 87
File: 0701
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8459062 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec

109

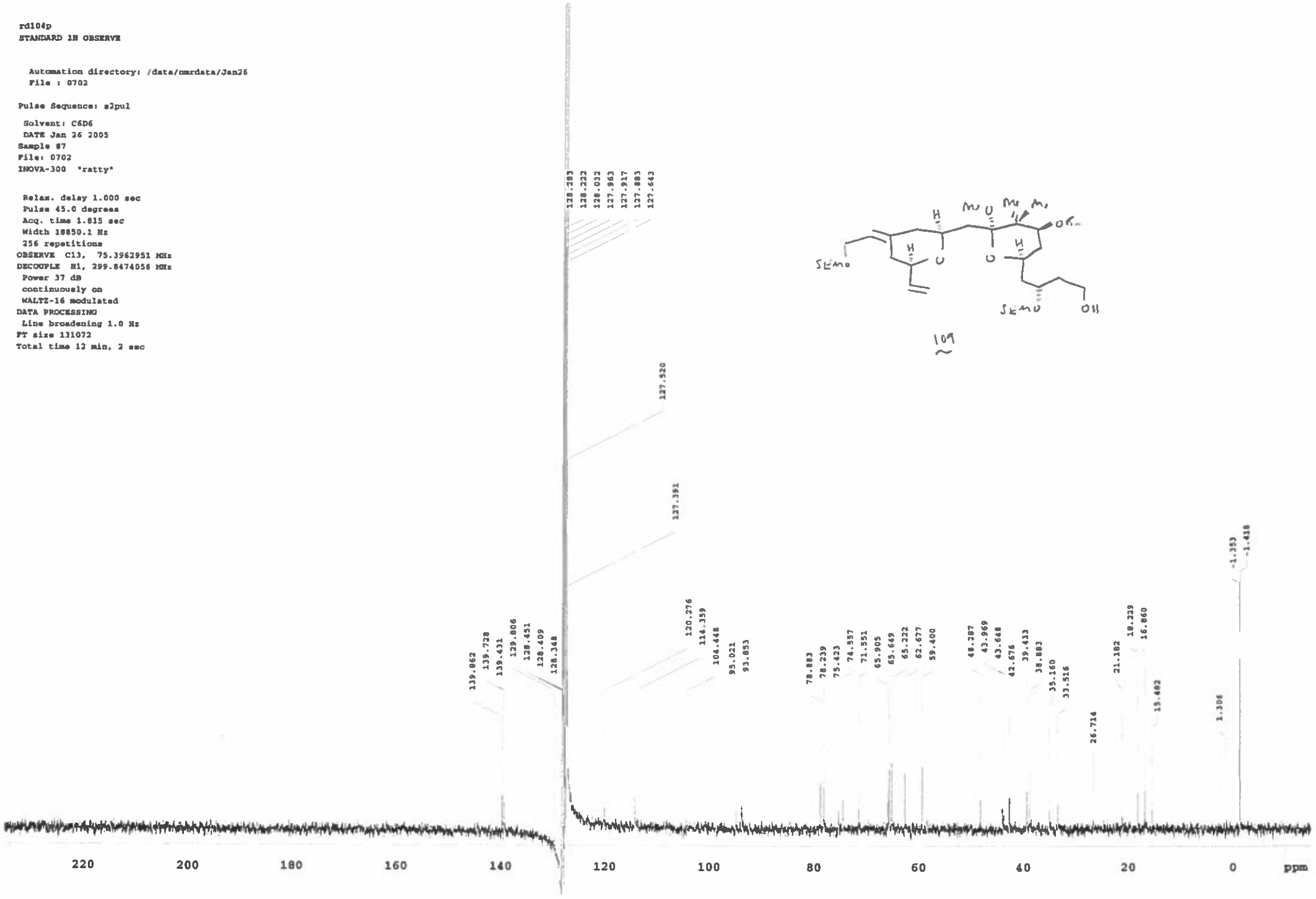


rd104p
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jan26
File : 0702

Pulse Sequence: s2pul

Solvent: C6D6
DATE Jan 26 2005
Sample #7
File: 0702
INOVA-300 "ratty"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18650.1 Hz
256 repetitions
OBSERVE C13, 75.3962951 MHz
DECOUPLE H1, 299.8474056 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec

109