SUPPORTING INFORMATION

Total Synthesis of (*S*)-(+)-Citreofuran by Ring Closing Alkyne Metathesis

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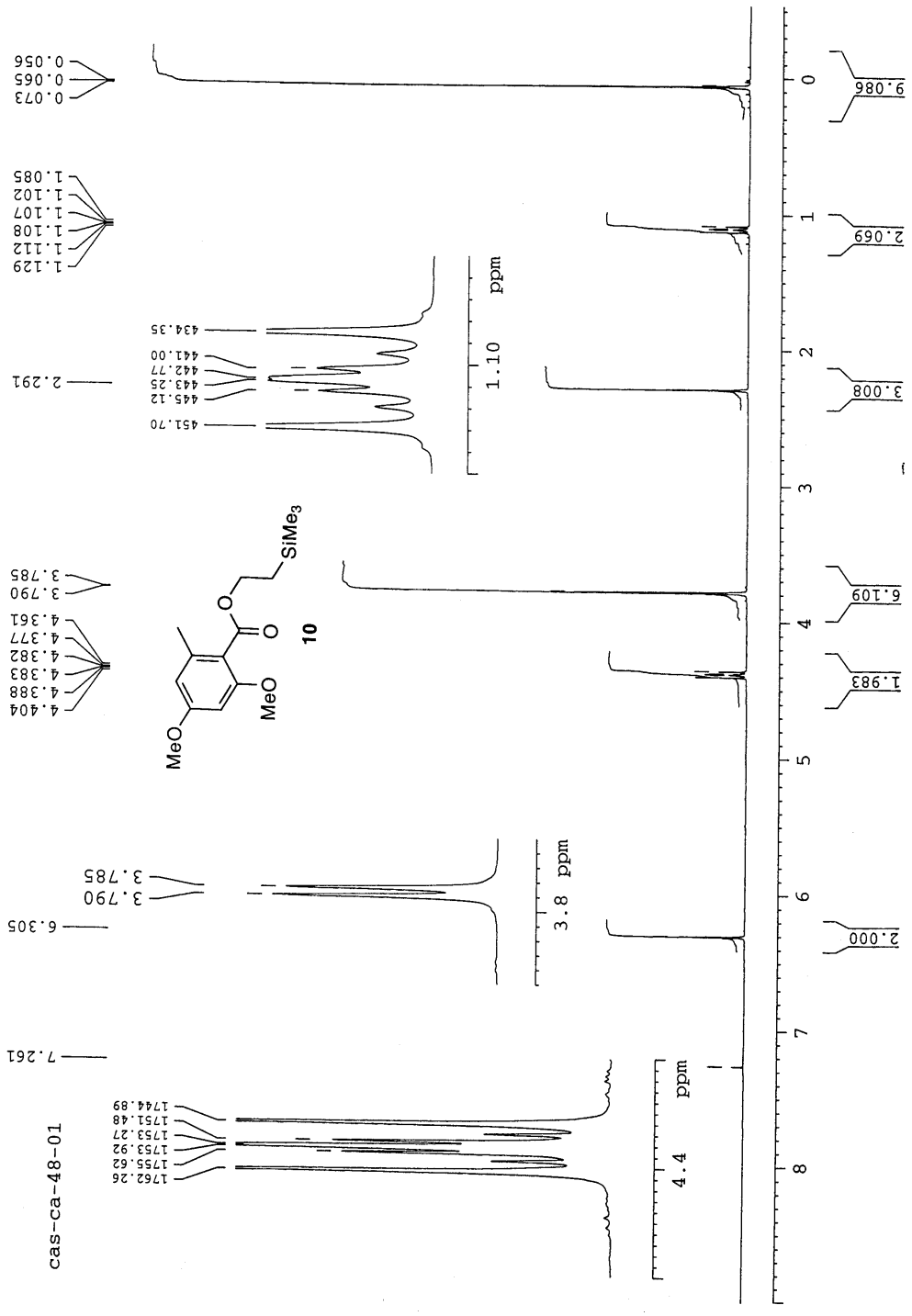
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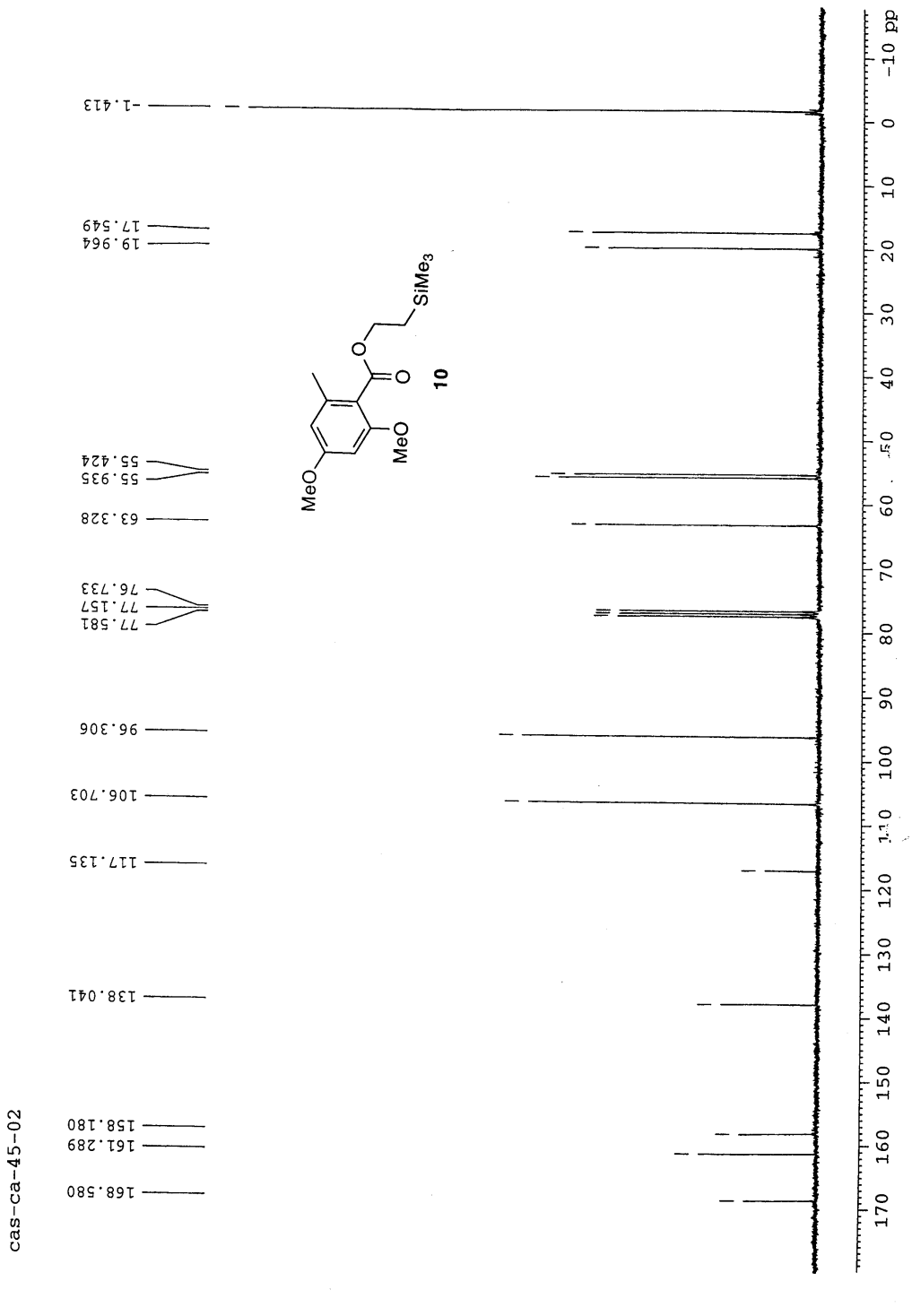
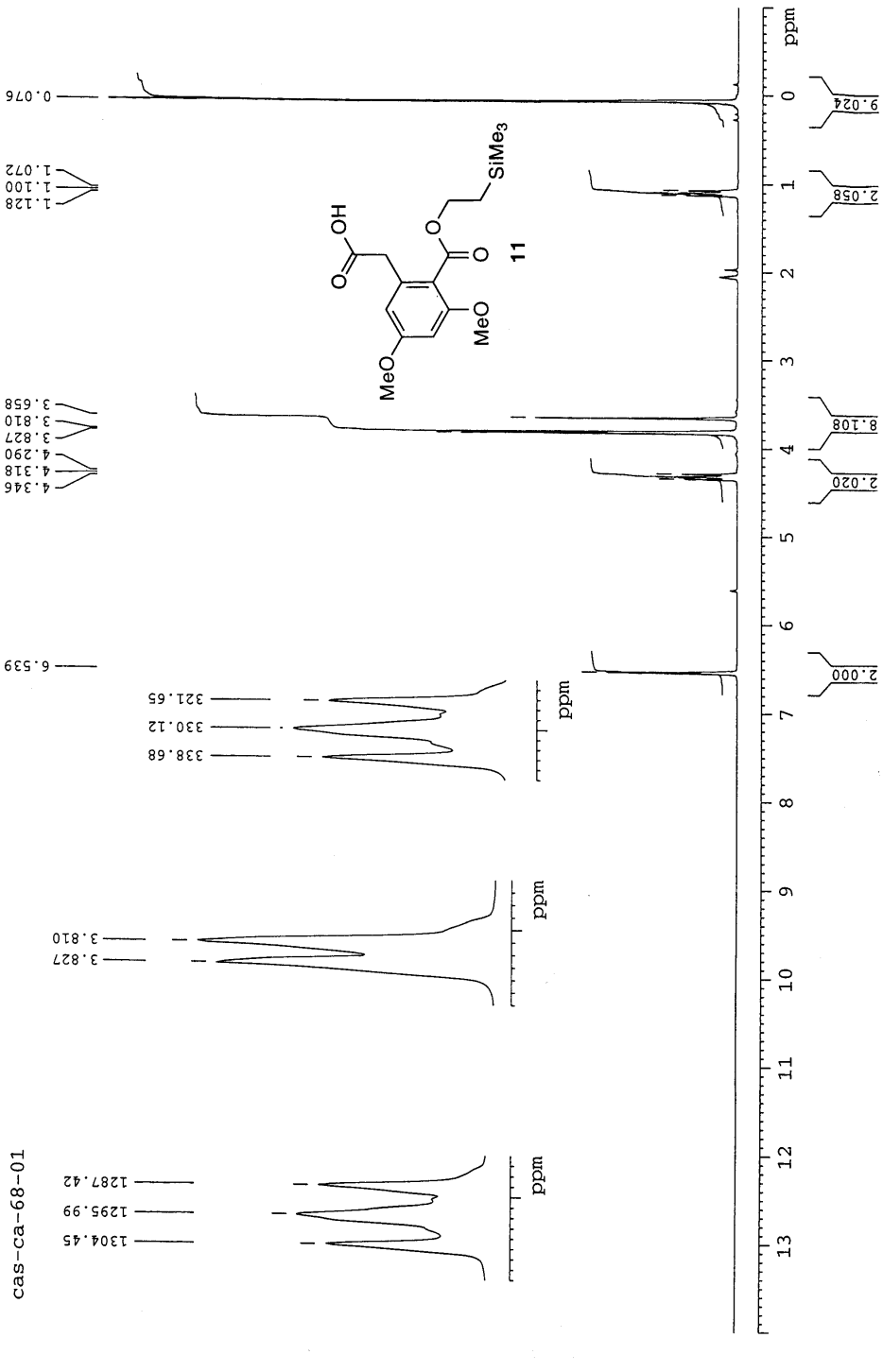
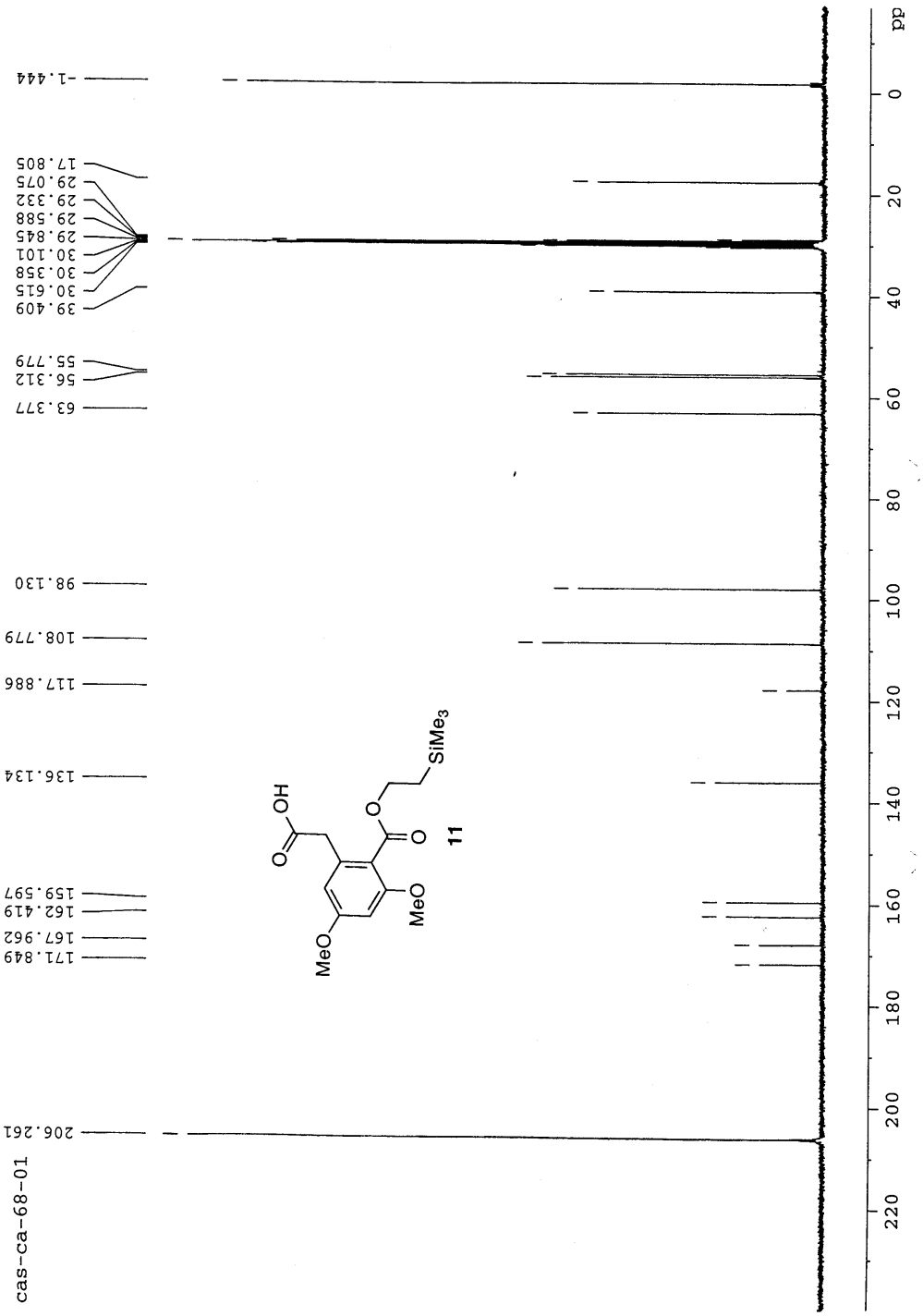
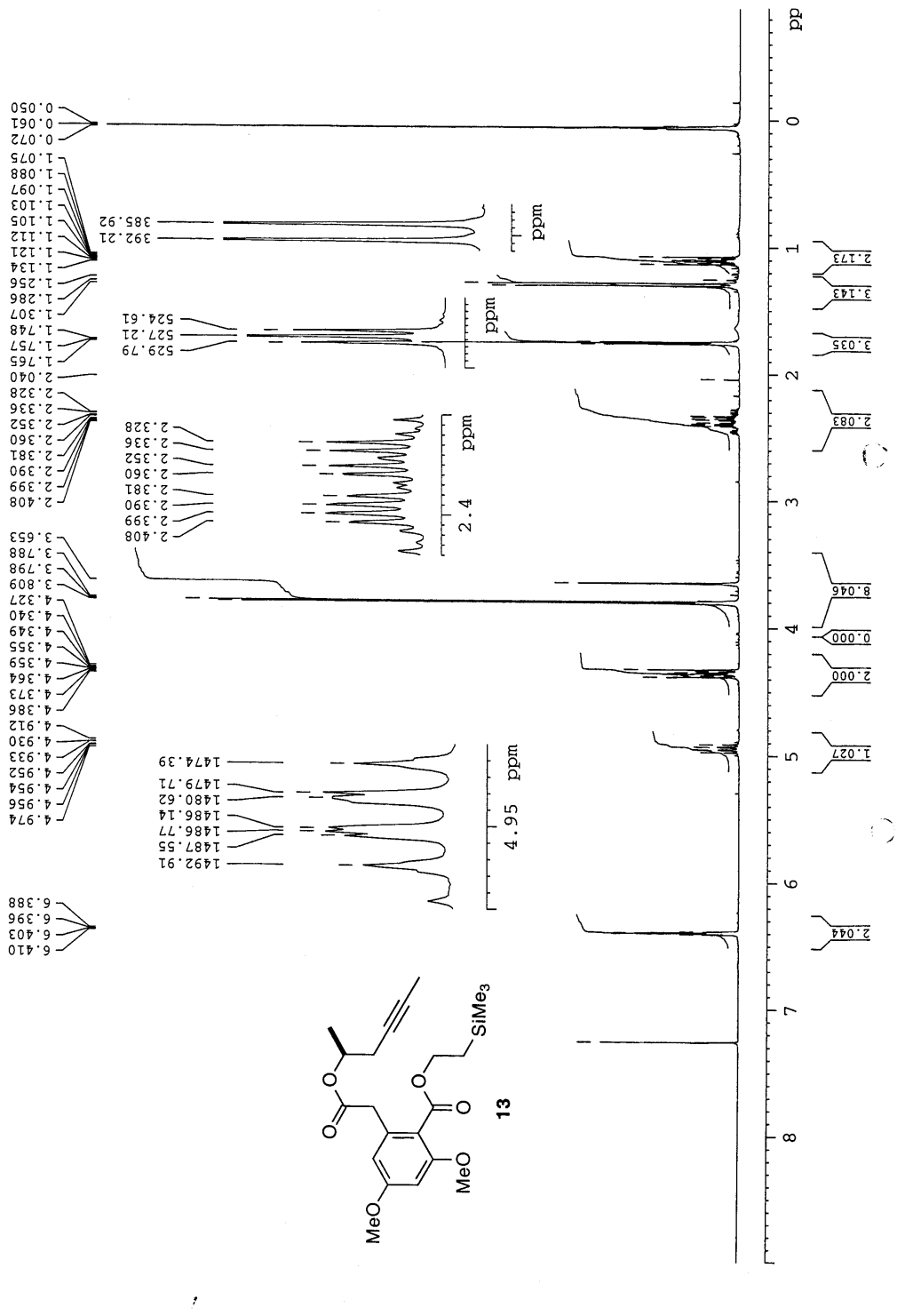
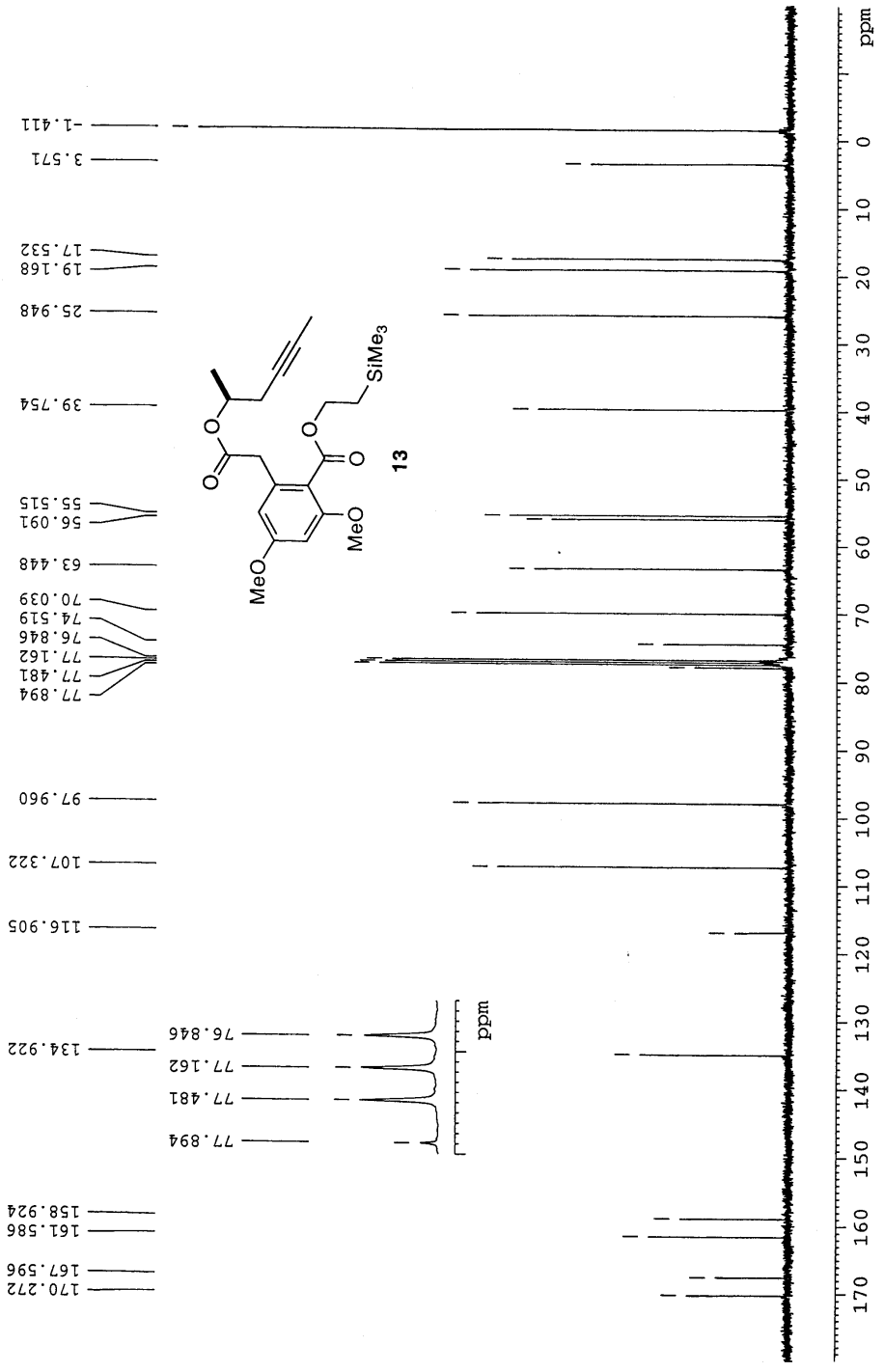
1762.26
1755.62
1753.92
1753.27
1751.48
1744.89
7.261
6.305
3.790
3.785
4.404
4.388
4.383
4.382
4.377
4.361
3.790
3.785
2.291
451.70
445.12
443.25
442.77
441.00
434.35
1.129
1.112
1.107
1.108
1.102
1.085
0.073
0.065
0.056
MeO
MeO
SiMe3
10
4.4 ppm
3.8 ppm
1.10 ppm
2.000
1.983
6.109
3.008
2.069
9.086

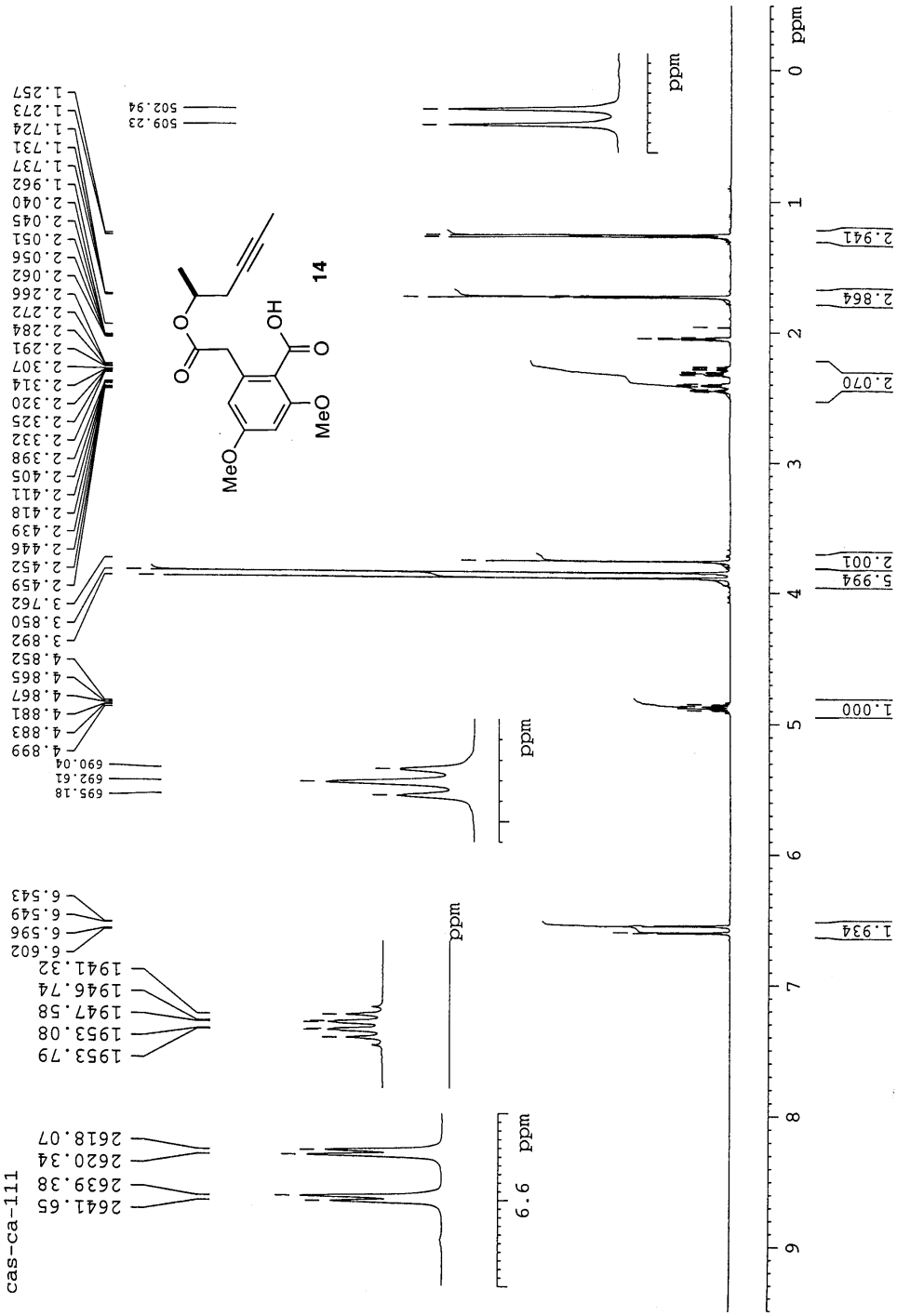

cas-ca-45-02
168.580
161.289
158.180
138.041
117.135
106.703
96.306
77.581
77.157
76.733
63.328
55.935
55.424
19.964
17.549
-1.413
MeO
MeO
SiMe3
O
O
O
10
ppm

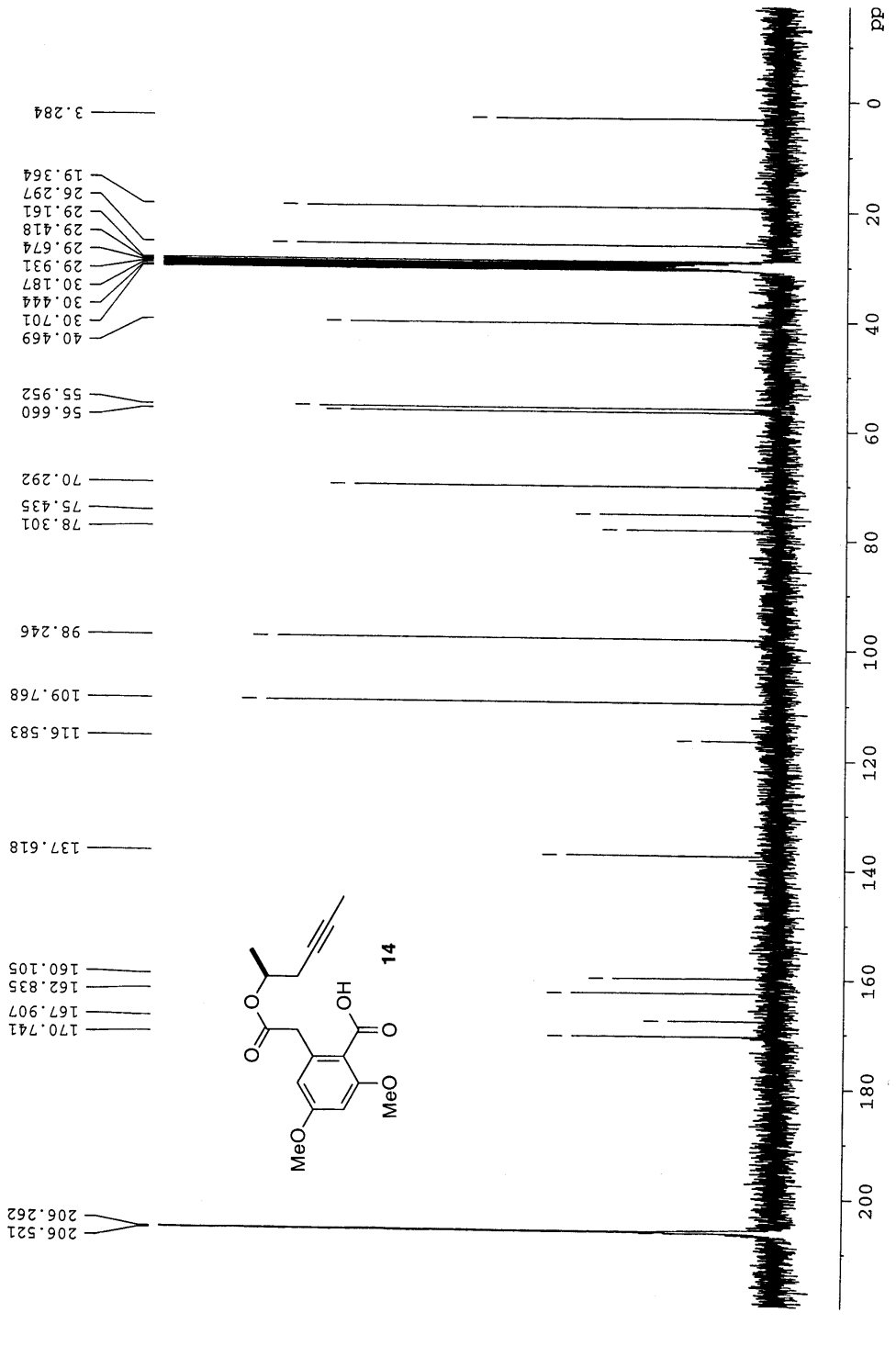

cas-ca-68-01
1304.45
1295.99
1287.42
ppm
3.827
3.810
ppm
338.68
330.12
321.65
ppm
6.539
4.346
4.318
4.290
3.827
3.810
3.658
1.128
1.100
1.072
0.076
SiMe3
O
O
O
OH
MeO
MeO
11
ppm
9.024
2.058
8.108
2.020
2.000

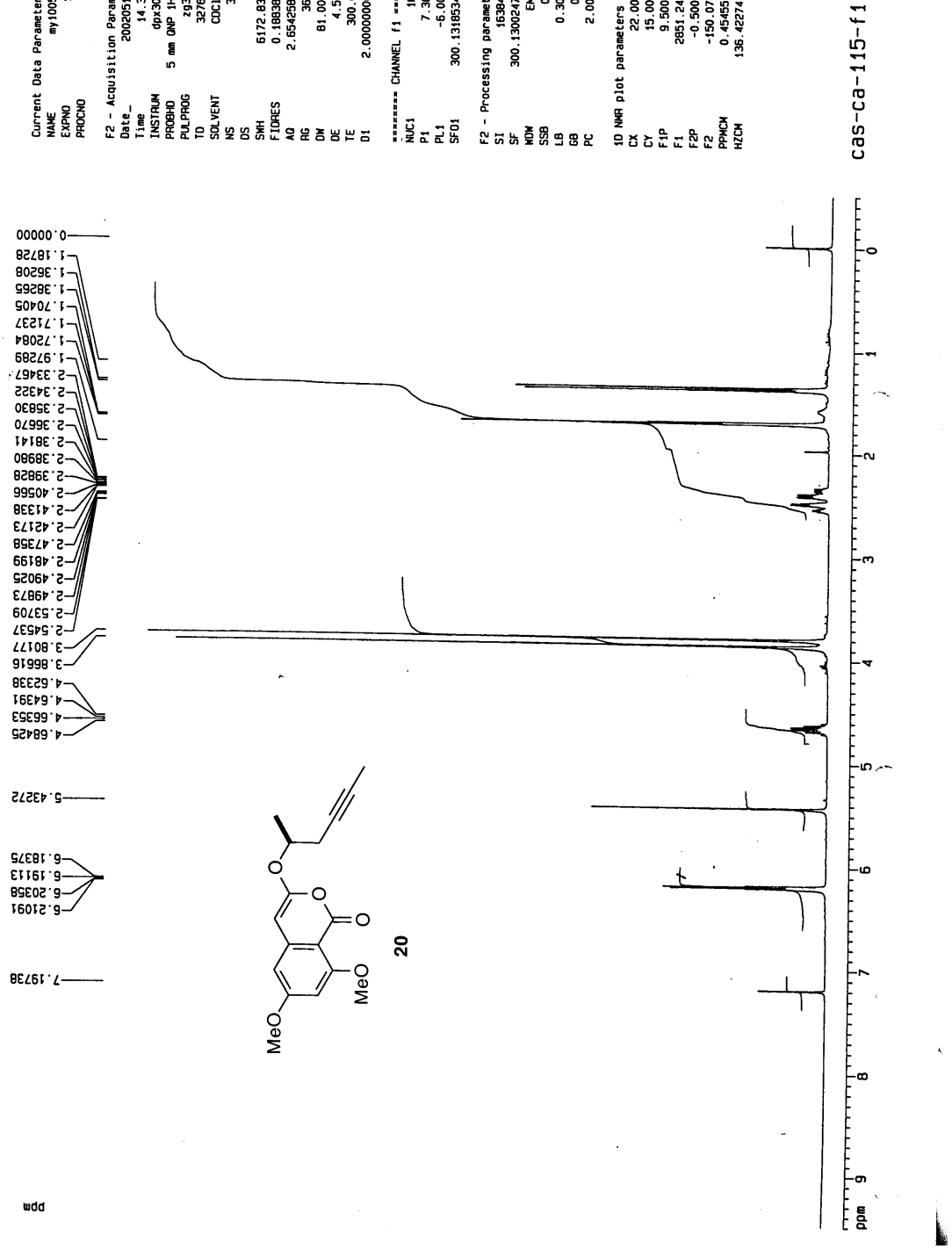

cas-ca-68-01
206.261
171.849
167.962
162.419
159.597
136.134
117.886
108.779
98.130
63.377
56.312
55.779
39.409
30.615
30.358
30.101
29.845
29.588
29.332
29.075
17.805
-1.444
ppm
0
20
40
60
80
100
120
140
160
180
200
220
OH
O
O
O
SiMe3
MeO
MeO
11


SiMe3
MeO
MeO
13
4.95 ppm
2.4 ppm
ppm
ppm
ppm
pp


-1.411
3.571
17.532
19.168
25.948
39.754
55.515
56.091
63.448
70.039
74.519
76.846
77.162
77.481
77.894
97.960
107.322
116.905
134.922
158.924
161.586
167.596
170.272
SiMe3
MeO
MeO
13
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170


cas-ca-111
14
MeO
MeO
OH
O
O
O
ppm


206.521
206.262
170.741
167.907
162.835
160.105
137.618
116.583
109.768
98.246
78.301
75.435
70.292
56.660
55.952
40.469
30.701
30.444
30.187
29.931
29.674
29.418
29.161
26.297
19.364
3.284
ppm
0
20
40
60
80
100
120
140
160
180
200
OH
O
O
O
MeO
MeO
14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EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
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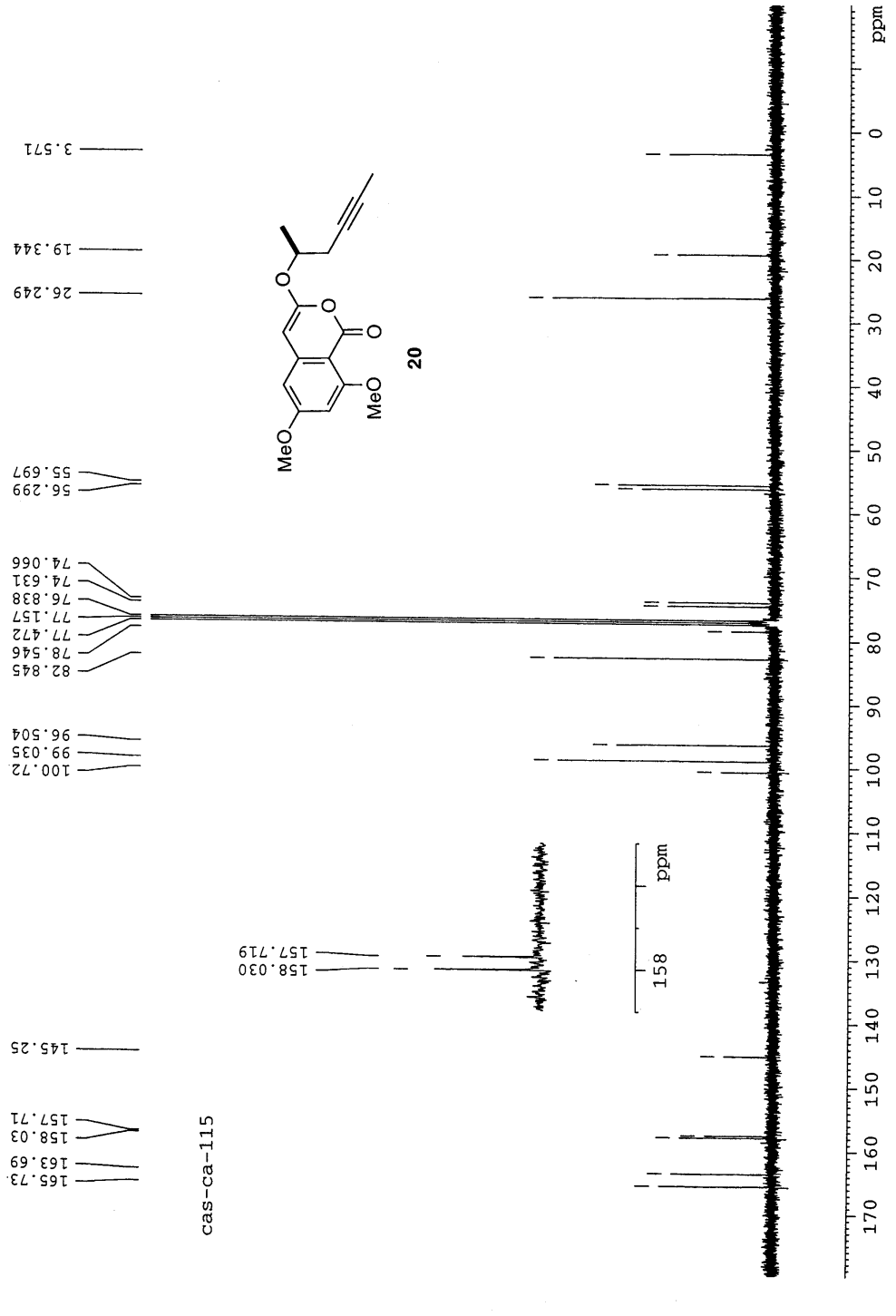
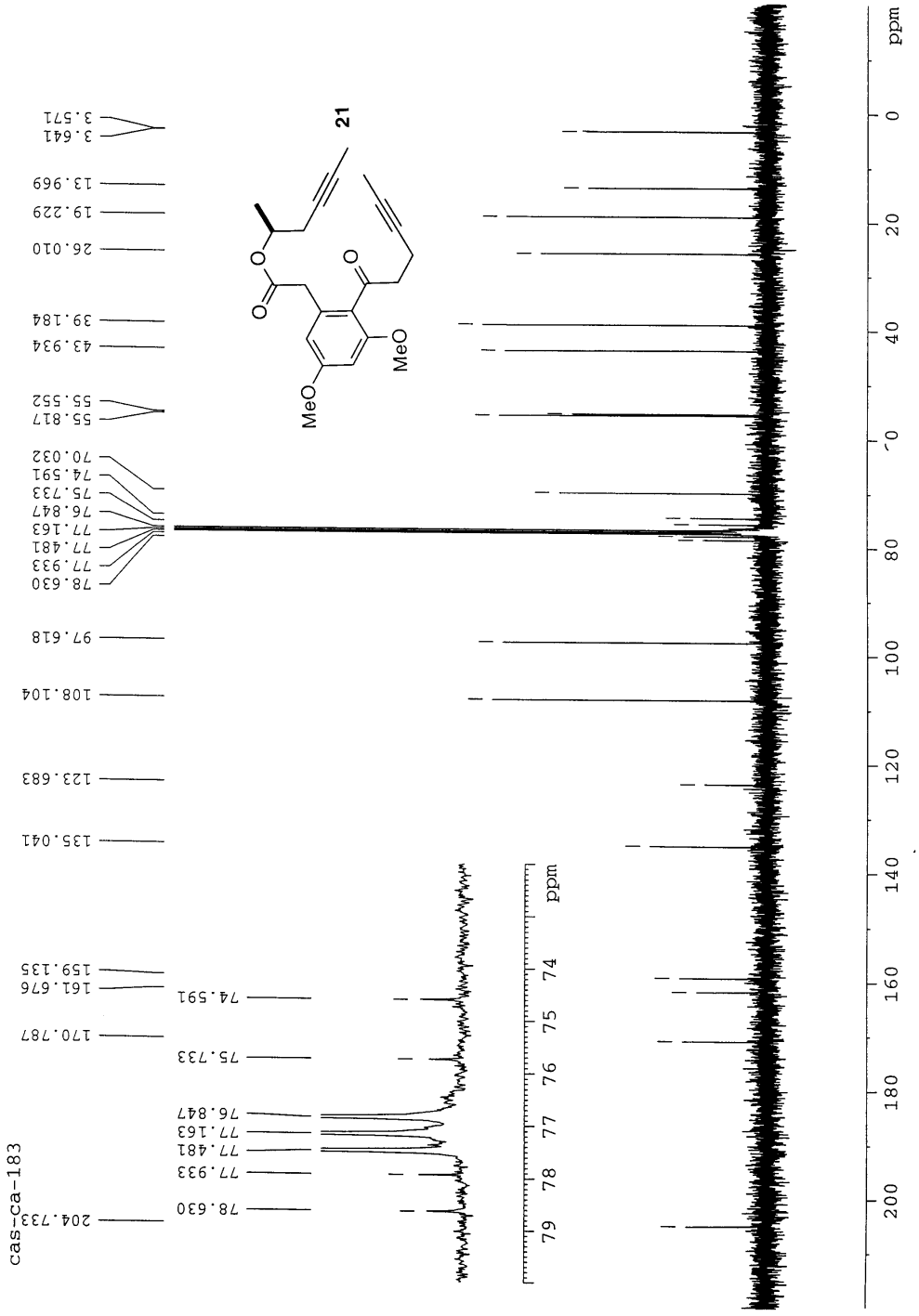
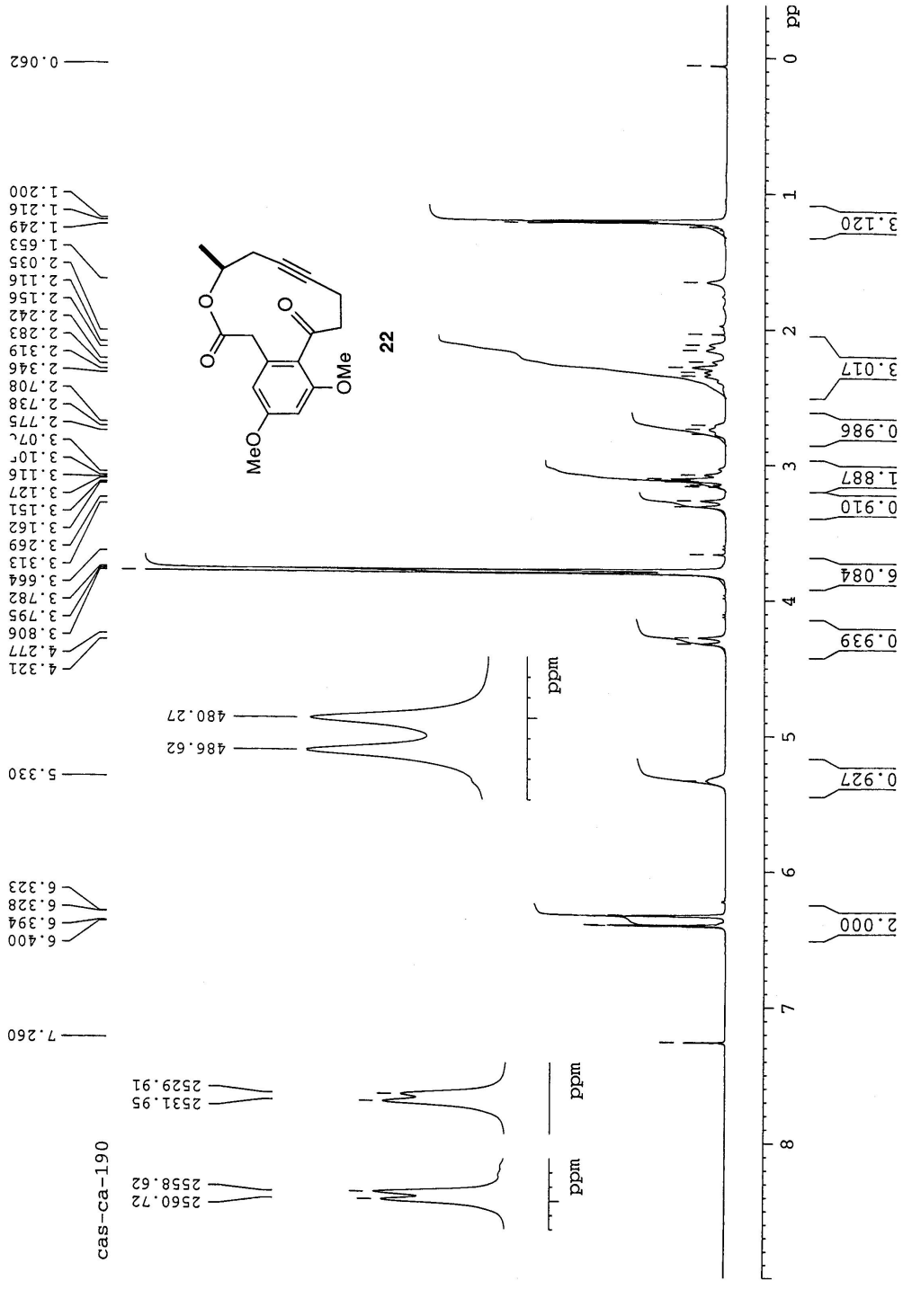
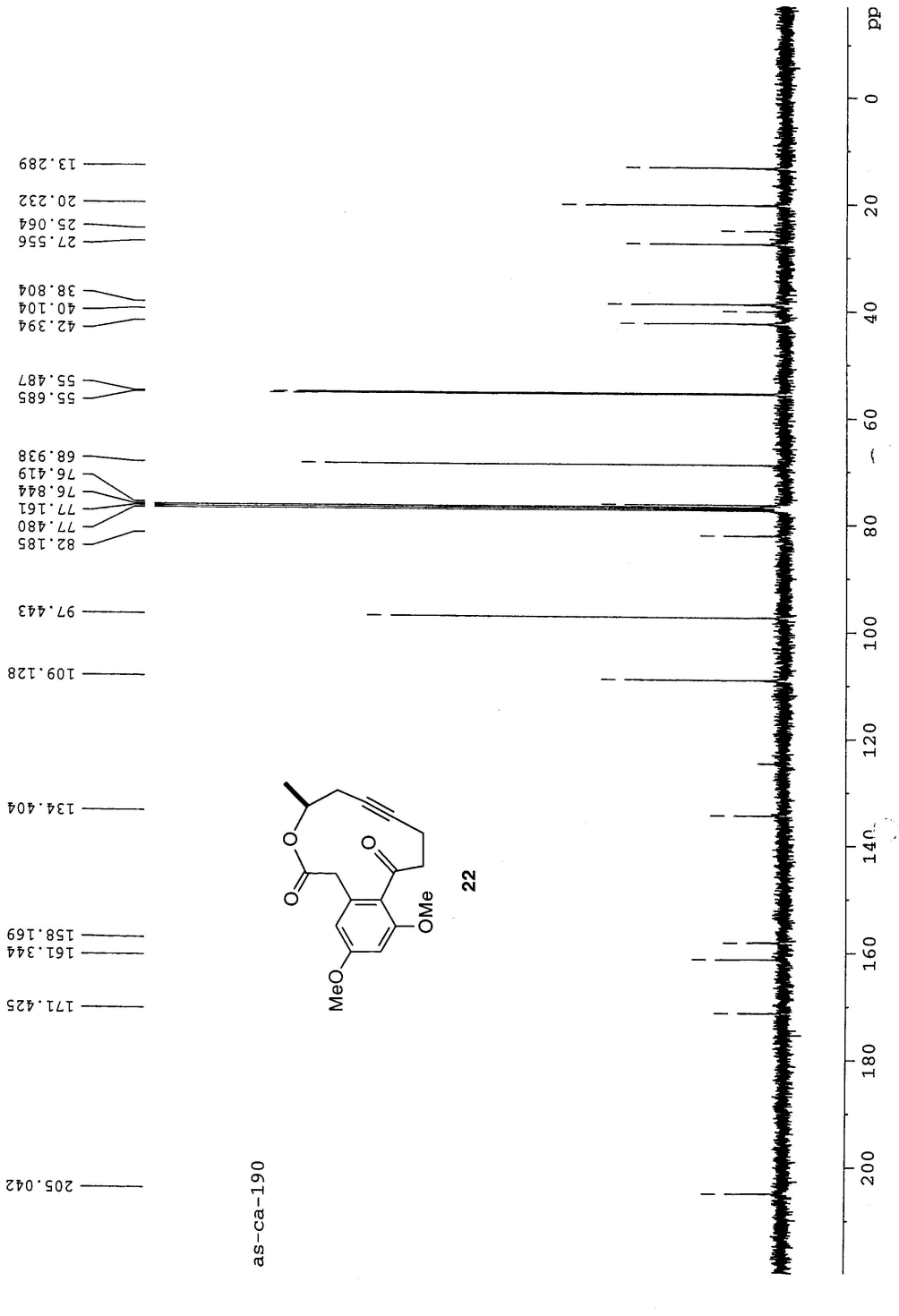
Time 14.39
INSTRUM dpx300
PROBHD 5 mm QNP 1H/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 362
DW 81.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.30 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300247 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 9.500 ppm
F1 2851.24 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.45455 ppm/cm
HZCM 136.42274 Hz/cm
cas-ca-115-f1
ppm
MeO
MeO
O
O
O
20


cas-ca-115
165.73
163.69
158.03
157.71
145.25
158.030
157.719
100.72
99.035
96.504
82.845
78.546
77.472
77.157
76.838
74.631
74.066
56.299
55.697
26.249
19.344
3.571
MeO
MeO
O
O
O
20
ppm
158
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170

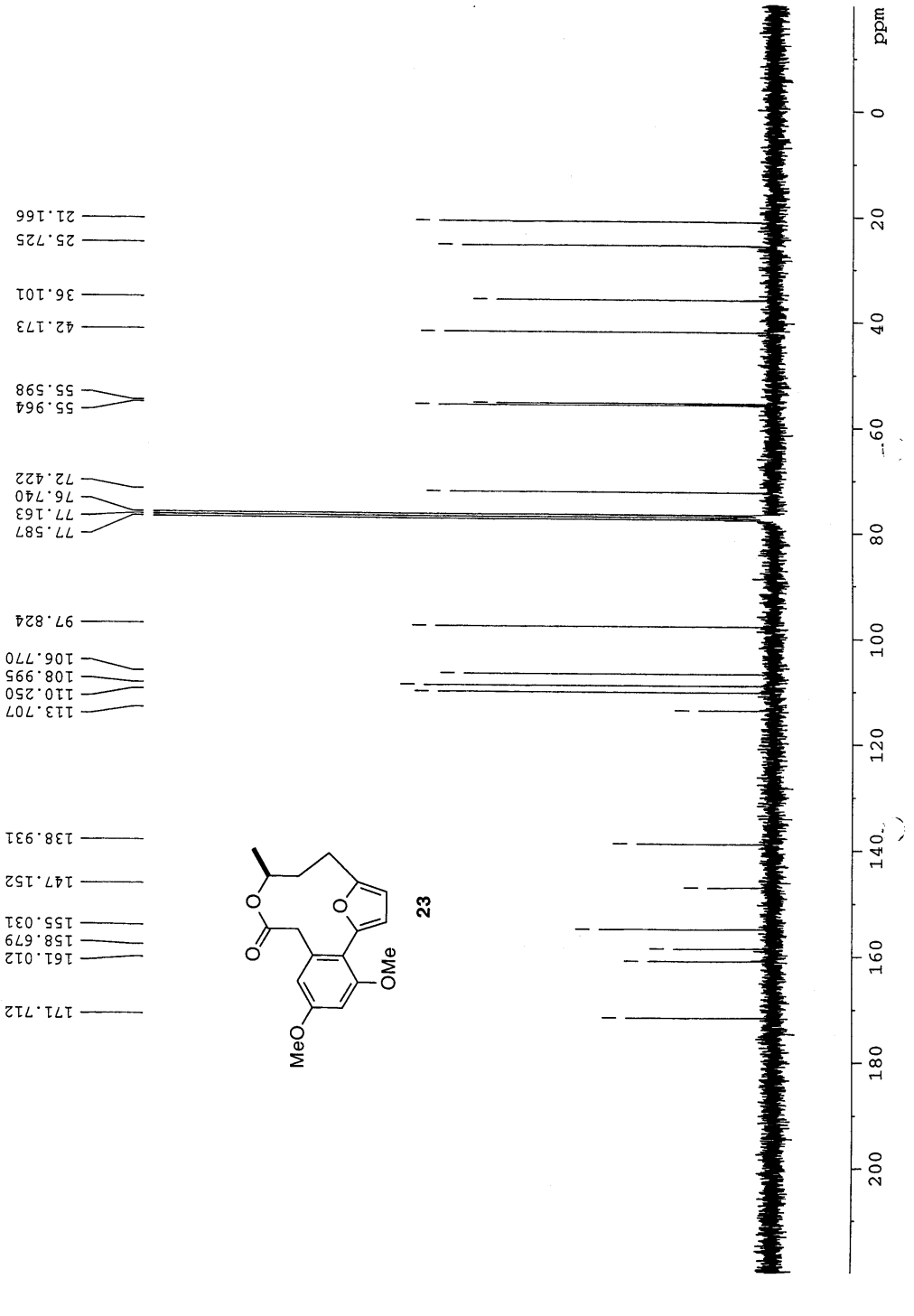
21
MeO
MeO
1.990
1.000
8.127
1.969
4.000
6.235
3.245
6.40 ppm
1.8 ppm
ppm
7.260
1489.43
1482.92
1477.27
1470.96
1918.55
1916.33
1913.68
1911.47

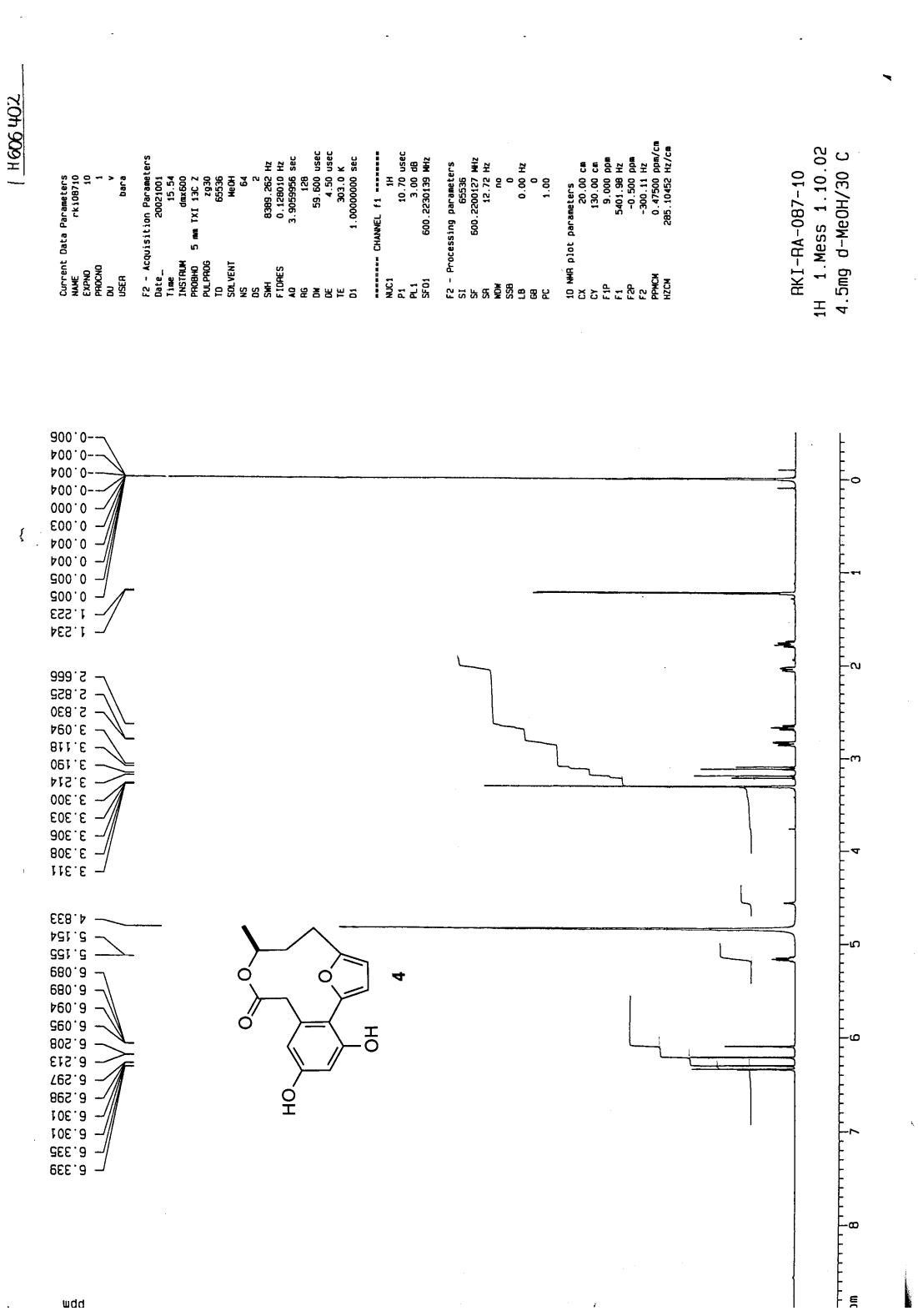

21
MeO
MeO
cas-ca-183
204.733
170.787
161.676
159.135
135.041
123.683
108.104
97.618
78.630
77.933
77.481
77.163
76.847
75.733
74.591
70.032
55.817
55.552
43.934
39.184
26.010
19.229
13.969
3.641
3.571
ppm


cas-ca-190
22
MeO
OMe
O
O
O
ppm
ppm
ppm
ppm
2560.72
2558.62
2531.95
2529.91
480.27
486.62
7.260
6.400
6.394
6.328
6.323
5.330
4.321
4.277
3.806
3.795
3.782
3.664
3.313
3.269
3.162
3.151
3.127
3.116
3.107
3.107
2.775
2.738
2.708
2.346
2.319
2.283
2.242
2.156
2.116
2.035
1.653
1.249
1.216
1.200
0.062
2.000
0.927
0.939
6.084
0.910
1.887
0.986
3.017
3.120
0
1
2
3
4
5
6
7
8
ppm


13.289
20.232
25.064
27.556
38.804
40.104
42.394
55.487
55.685
68.938
76.419
76.844
77.161
77.480
82.185
97.443
109.128
134.404
158.169
161.344
171.425
205.042
as-ca-190
22
OMe
MeO
O
O
O
O
ppm
0
20
40
60
80
100
120
140
160
180
200

cas-ca-191-01
MeO
OMe
23
ppm


21.166
25.725
36.101
42.173
55.598
55.964
72.422
76.740
77.163
77.587
97.824
106.770
108.995
110.250
113.707
138.931
147.152
155.031
158.679
161.012
171.712
MeO
OMe
O
O
O
23
ppm
0
20
40
60
80
100
120
140
160
180
200


H606402
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NAME rk108710
EXPNO 10
PROCNO 1
DU v
USER bara
F2 - Acquisition Parameters
Date_ 20021001
Time 15.54
INSTRUM dmx600
PROBHD 5 mm TXI 13C Z
PULPROG zg30
TD 65536
SOLVENT MeOH
NS 64
DS 2
SWH 8389.262 Hz
FIDRES 0.128010 Hz
AQ 3.9059956 sec
RG 128
DW 59.600 usec
DE 4.50 usec
TE 303.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.70 usec
PL1 3.00 dB
SFO1 600.2230139 MHz
F2 - Processing parameters
SI 65536
SF 600.2200127 MHz
SR 12.72 Hz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 130.00 cm
F1P 9.000 ppm
F1 5401.98 Hz
F2P -0.500 ppm
F2 -300.11 Hz
PPMCM 0.47500 ppm/cm
HZCM 285.10452 Hz/cm
RKI-RA-087-10
1H 1.Mess 1.10.02
4.5mg d-MeOH/30 C
4
HO
OH
O
O
6.339
6.335
6.301
6.301
6.298
6.297
6.213
6.208
6.095
6.094
6.089
6.089
5.155
5.154
4.833
3.311
3.308
3.306
3.303
3.300
3.214
3.190
3.118
3.094
2.830
2.825
2.666
1.234
1.223
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0.004
0.004
0.004
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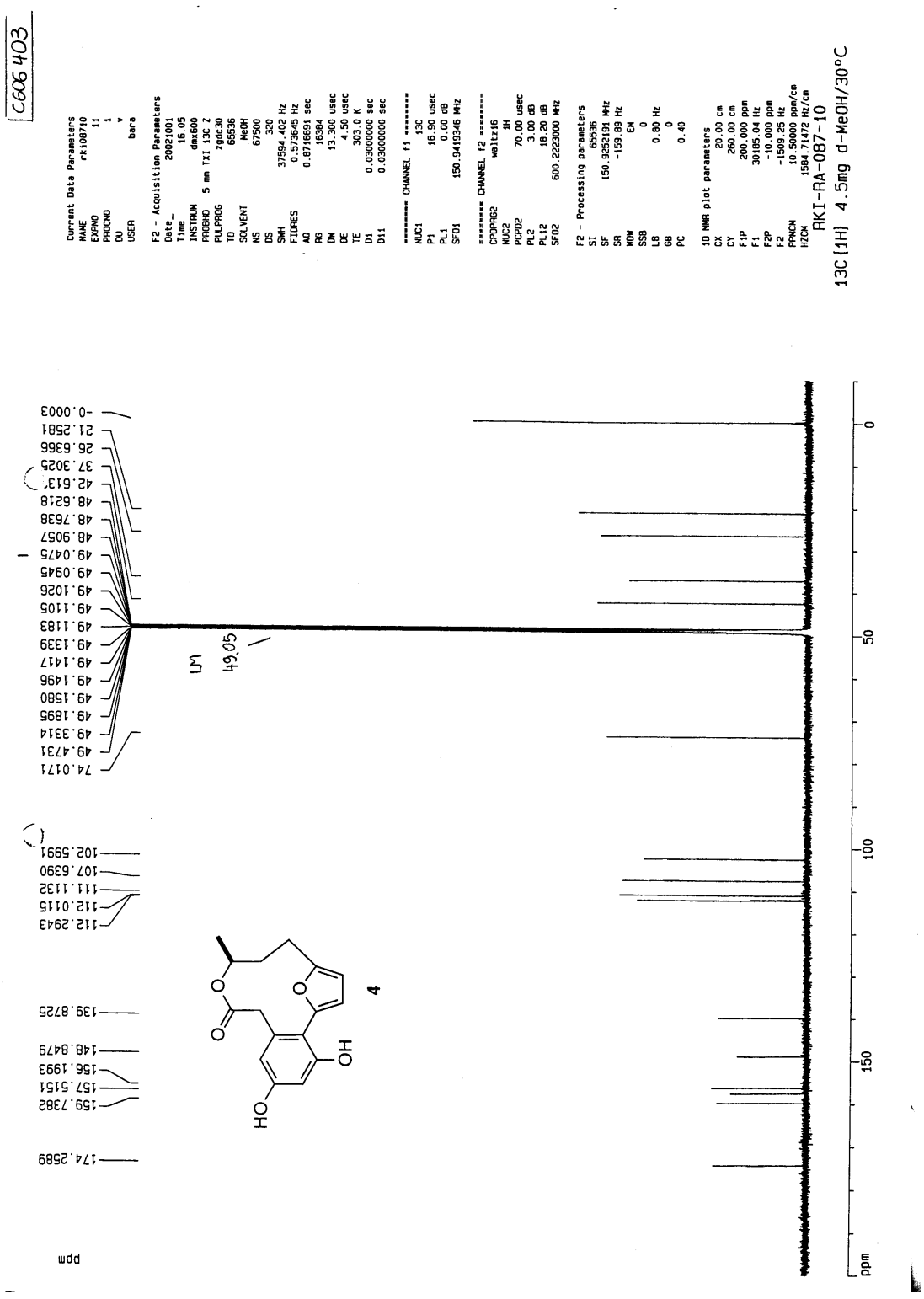
0.004
0.004
0.004
0.004
ppm


C606403
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EXPNO 11
PROCNO 1
OU v
USER bara
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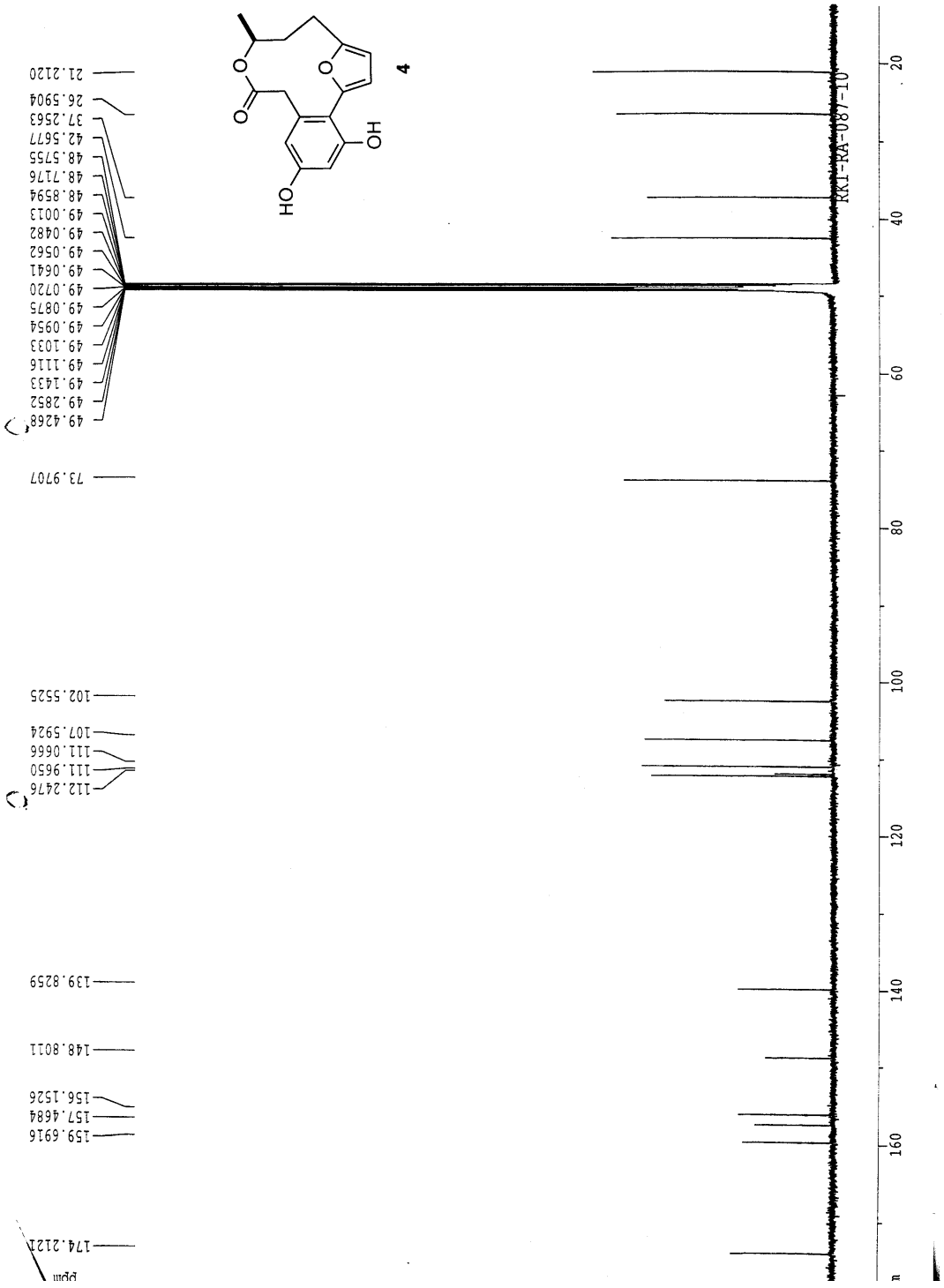
Time 16.05
INSTRUM dmx600
PROBHD 5 mm TXI 13C Z
PULPROG zgdc30
TD 65536
SOLVENT MeOH
NS 67500
DS 320
SWH 37594.402 Hz
FIDRES 0.573645 Hz
AQ 0.8716691 sec
RG 16384
DW 13.300 usec
DE 4.50 usec
TE 303.0 K
D1 0.03000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 16.90 usec
PL1 0.00 dB
SFO1 150.9419346 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 3.00 dB
PL12 18.20 dB
SFO2 600.2223000 MHz
F2 - Processing parameters
SI 65536
SF 150.9252191 MHz
SR -159.89 Hz
WDW EM
SSB 0
LB 0.80 Hz
GB 0
PC 0.40
1D NMR plot parameters
CX 20.00 cm
CY 260.00 cm
F1P 200.000 ppm
F1 30185.04 Hz
F2P -10.000 ppm
F2 -1509.25 Hz
PPMCM 10.50000 ppm/cm
HZCM 1584.71472 Hz/cm
RKI-RA-087-10
13C{1H} 4.5mg d-MeOH/30°C
ppm
-0.0003
21.2581
26.6366
37.3025
42.6137
48.6218
48.7638
48.9057
49.0475
49.0945
49.1026
49.1105
49.1183
49.1339
49.1417
49.1496
49.1580
49.1895
49.3314
49.4731
74.0171
49.05
102.5991
107.6390
111.1132
112.0115
112.2943
139.8725
148.8479
156.1993
157.5151
159.7382
174.2589
4
OH
HO
O
O
O
0
50
100
150
ppm


KK1-RA-087-10
4
HO
OH
O
O
21.2120
26.5904
37.2563
42.5677
48.5755
48.7176
48.8594
49.0013
49.0482
49.0562
49.0641
49.0720
49.0875
49.0954
49.1033
49.1116
49.1433
49.2852
49.4268
73.9707
102.5525
107.5924
111.0666
111.9650
112.2476
139.8259
148.8011
156.1526
157.4684
159.6916
174.2121
ppm