Enantioselective Synthesis of the Anti-inflammatory Agent (–)-Acanthoic Acid

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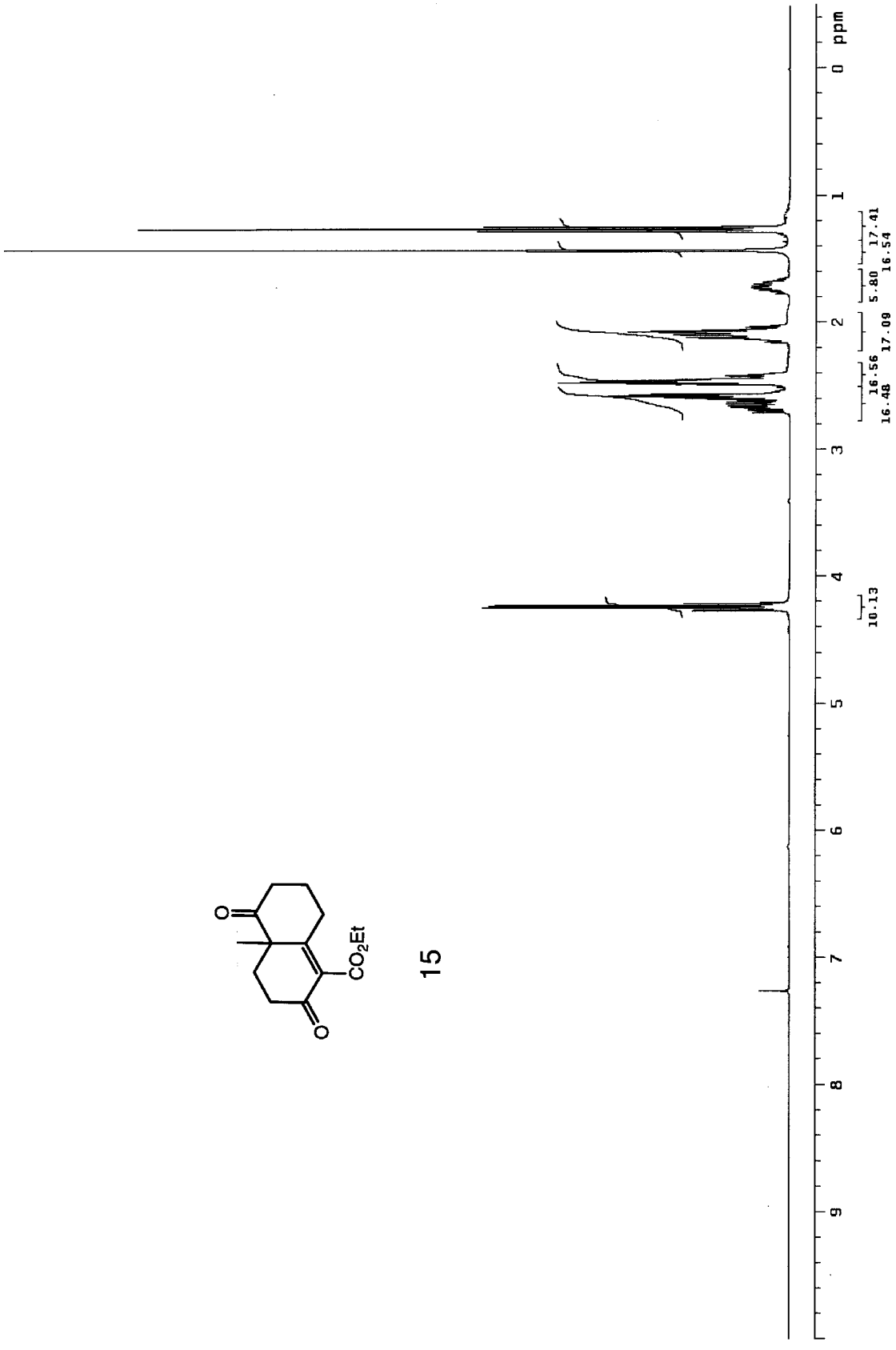
Supporting Information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O
CO2Et
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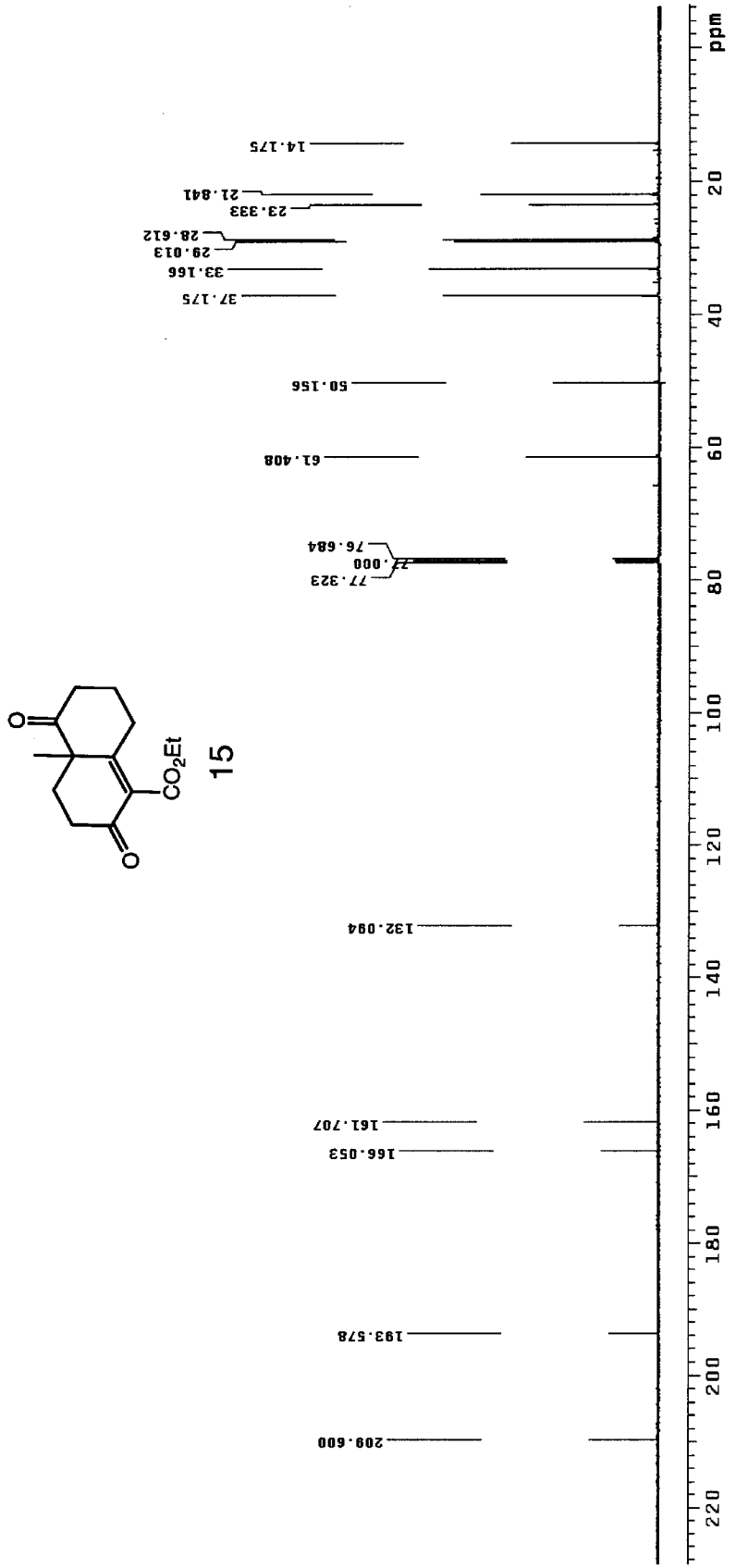
16.56
17.09
5.80
16.54
17.41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O
CO2Et
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77.000
76.684
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33.166
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160
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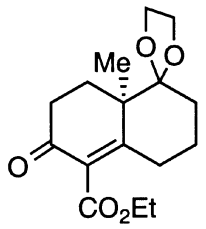
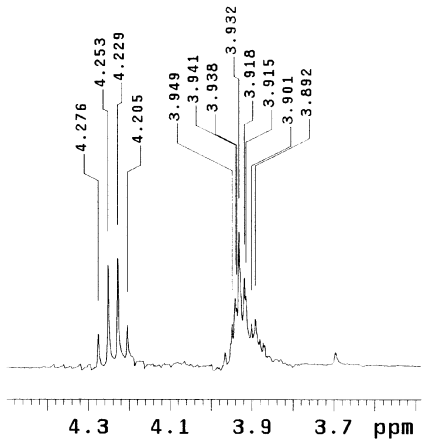
80
60
40
20


Me
O
O
O
CO2Et

16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4.253
4.229
4.205
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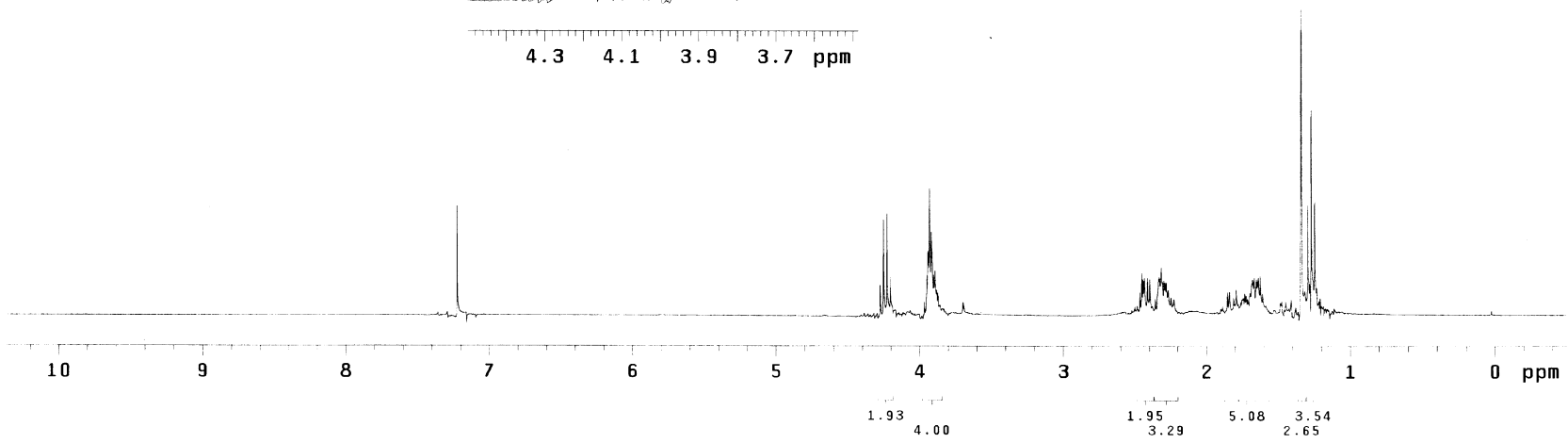
3.932
3.918
3.915
3.901
3.892
4.3
4.1
3.9
3.7 ppm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9
8
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3
2
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0 ppm
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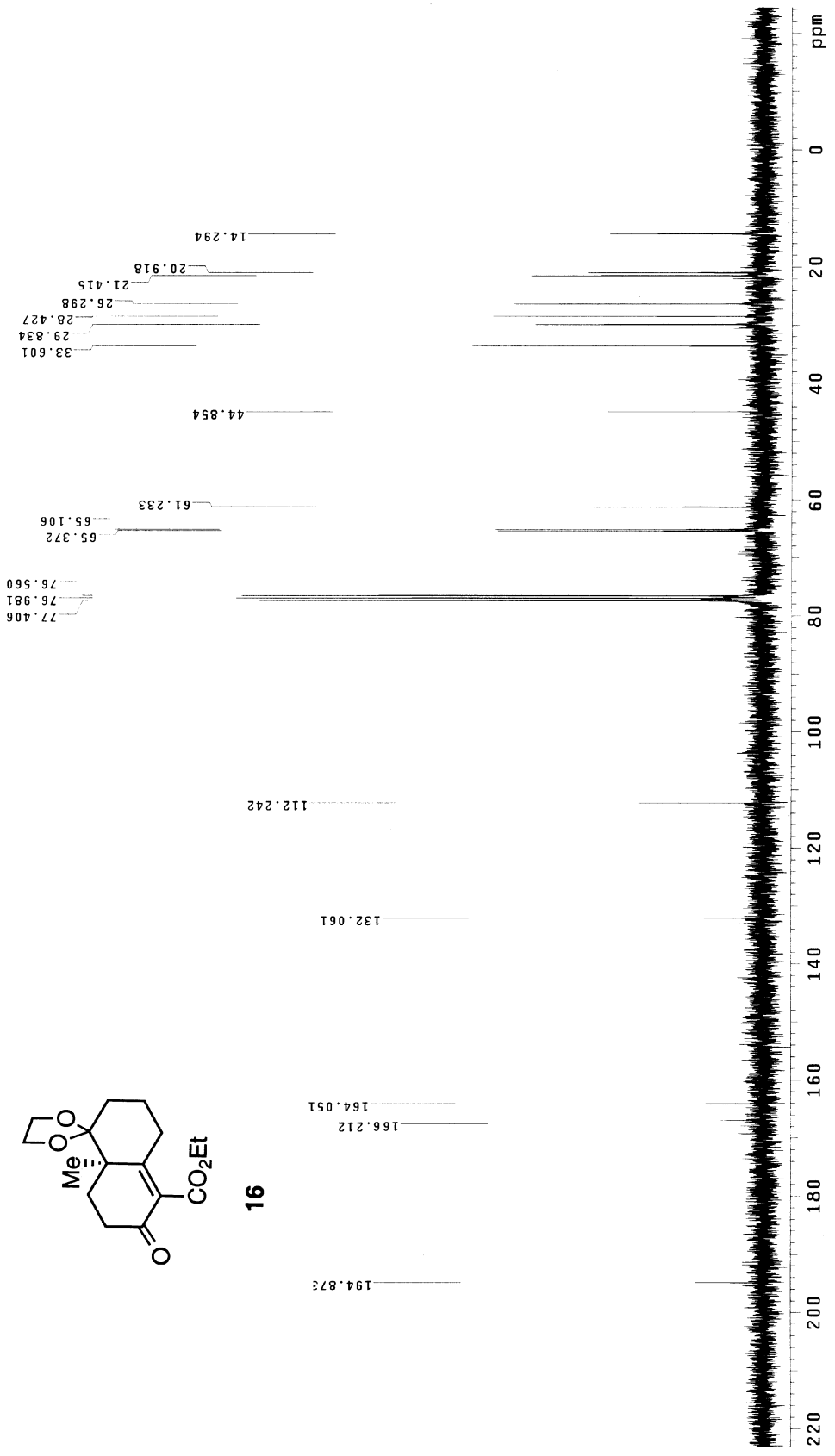
4.00
1.95
3.29
5.08
3.54
2.65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20
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200
220
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21.415
26.298
28.427
29.834
33.601
44.854
61.233
65.106
65.372
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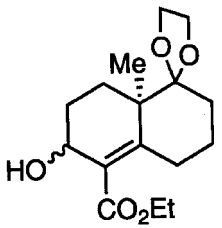
166.212
194.873
Me
CO2Et
O
O
O
16


Me
O
O
HO
CO2Et

17

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4.218
3.963
3.936
3.932
3.929
4.4
4.0
3.6
3.2
ppm
10
9
8
7
6
5
4
3
2
1
ppm
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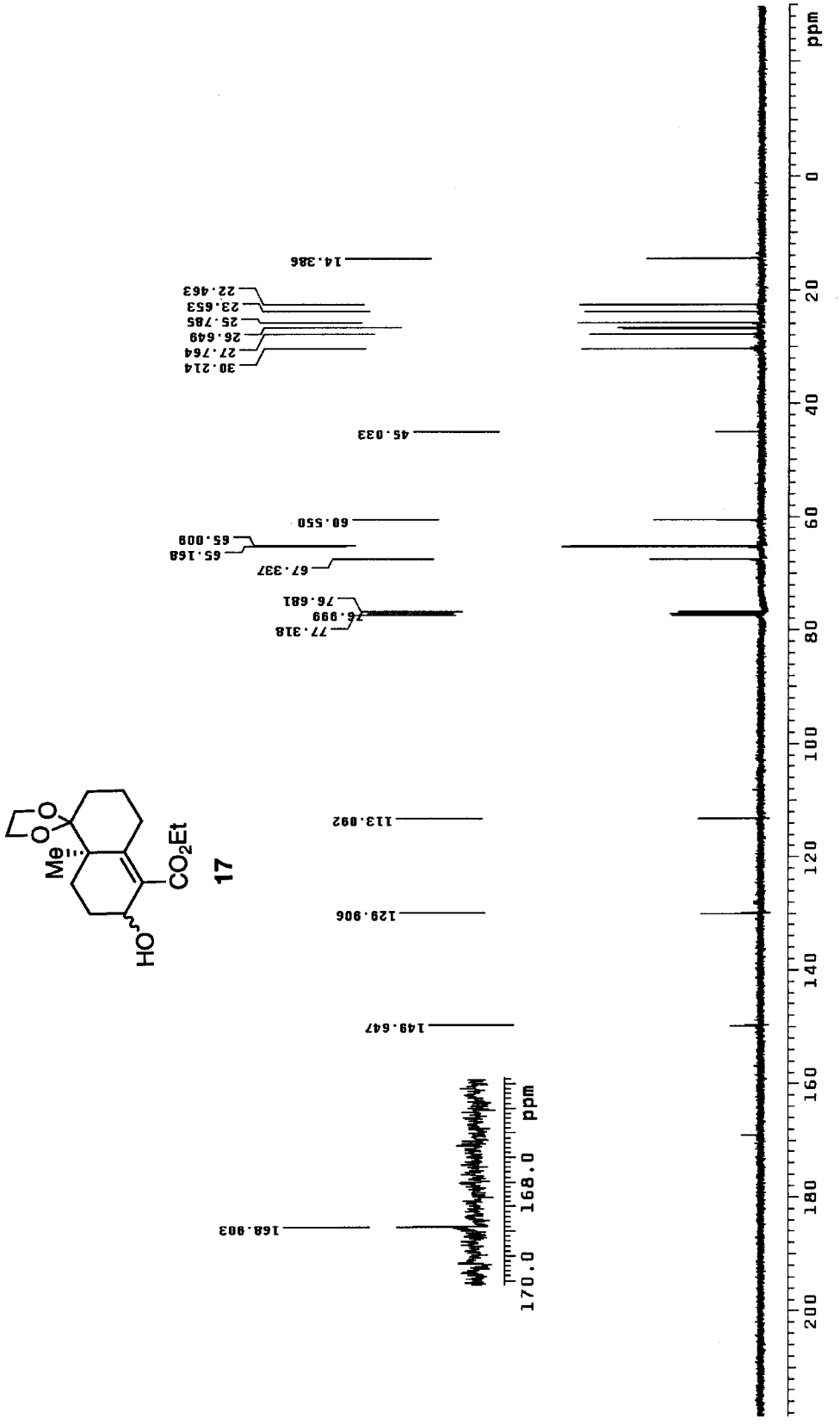
1.01
2.71
5.04
2.45
3.06
3.97


17
Me
CO2Et
HO
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129.906
113.092
77.318
76.999
76.681
67.337
65.168
65.009
60.550
45.033
30.214
27.764
26.649
25.785
23.653
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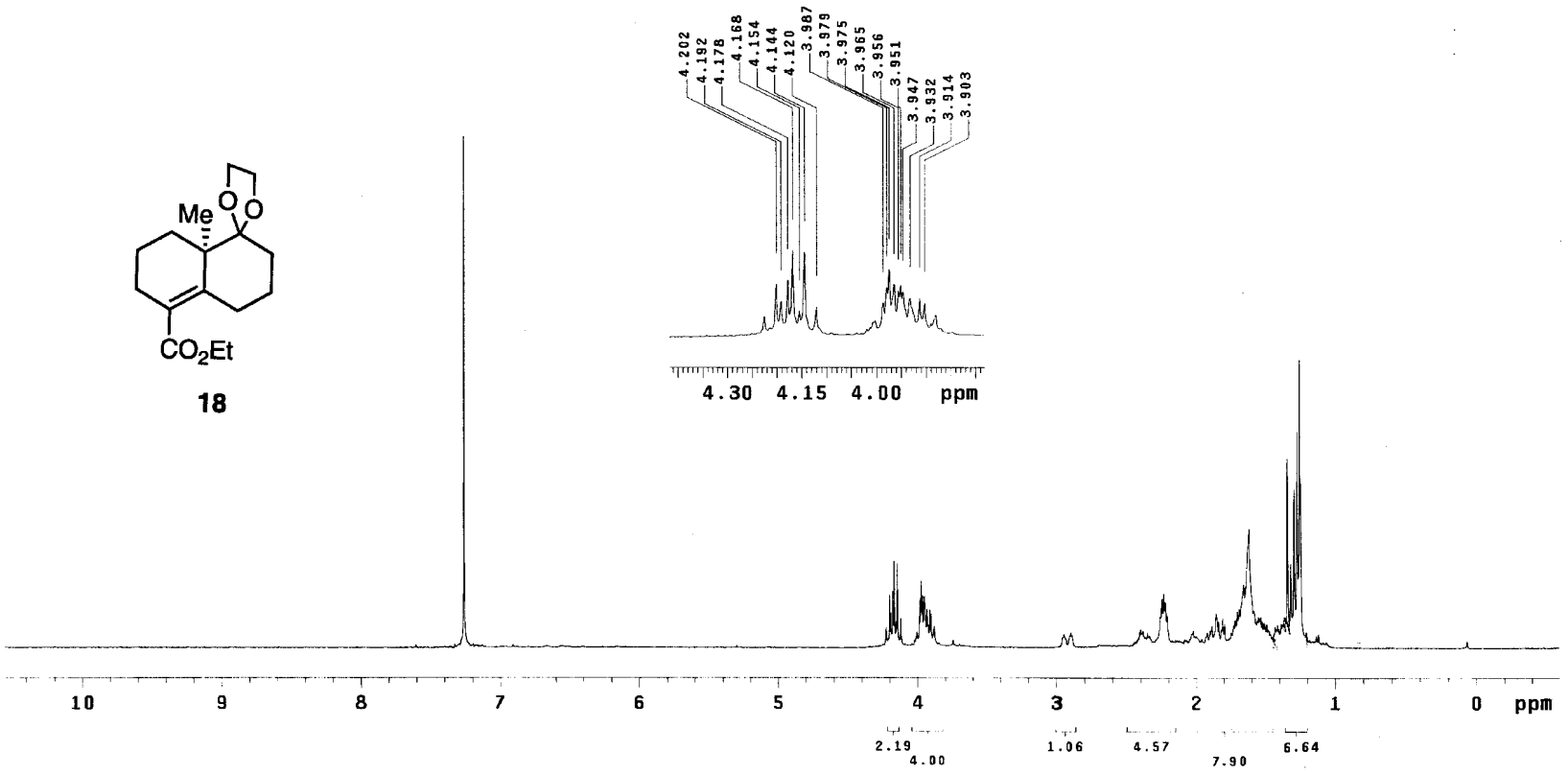
14.386
170.0
168.0
ppm
ppm


Me
O
O
CO2Et
18
4.202
4.192
4.178
4.168
4.154
4.144
4.120
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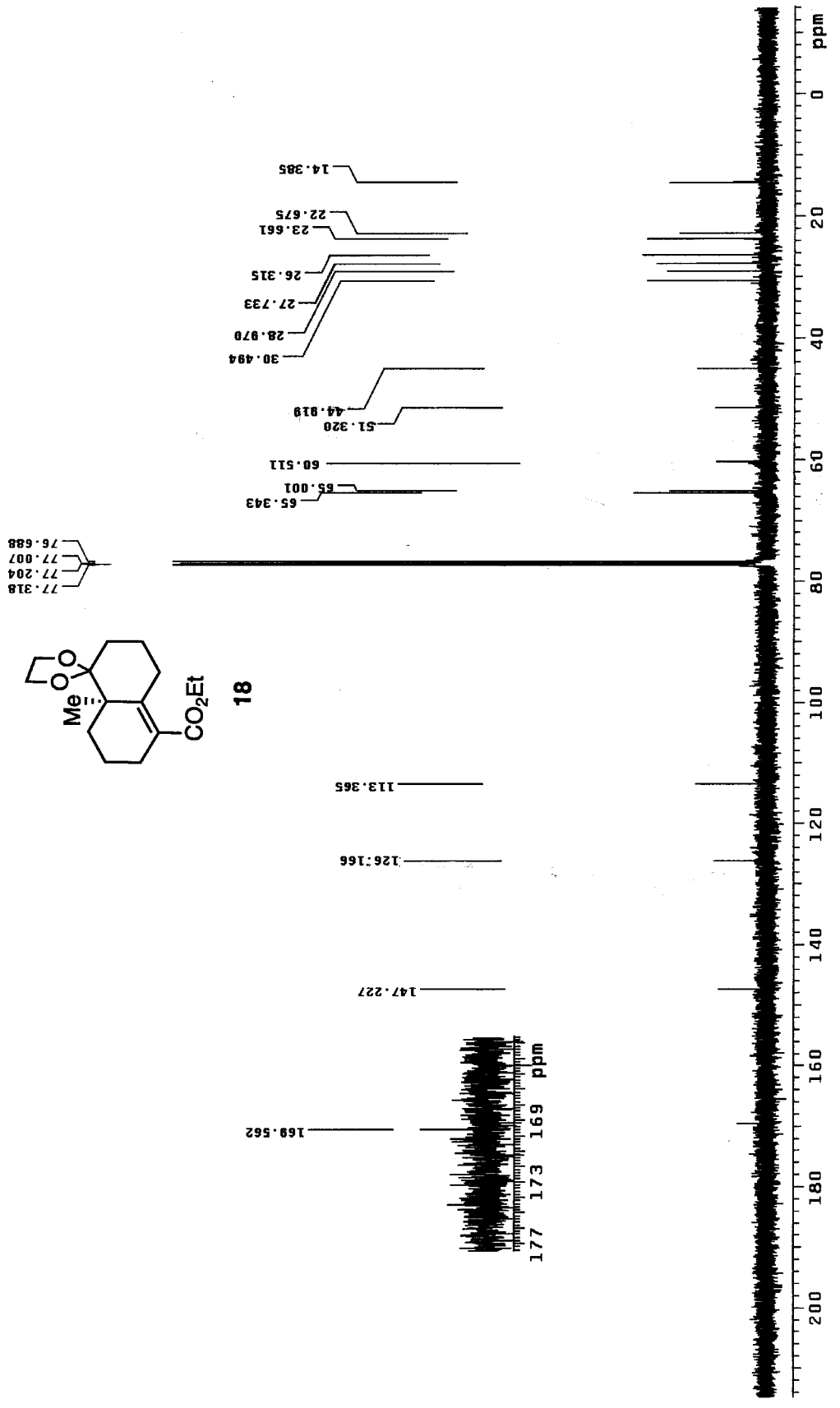
3.979
3.975
3.965
3.956
3.951
3.947
3.932
3.914
3.903
4.30
4.15
4.00
ppm
10
9
8
7
6
5
4
3
2
1
0
ppm
2.19
4.00
1.06
4.57
7.90
6.64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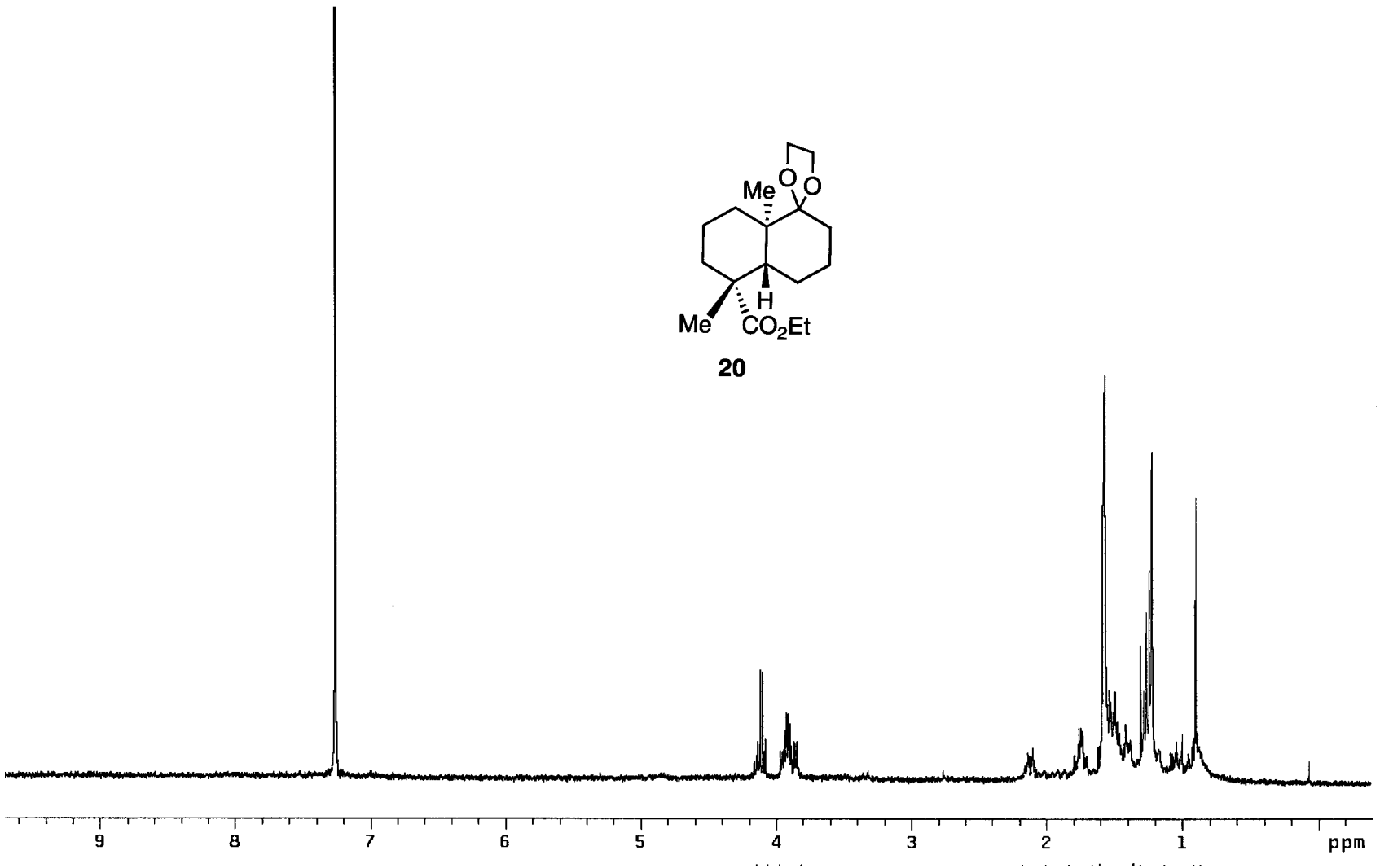
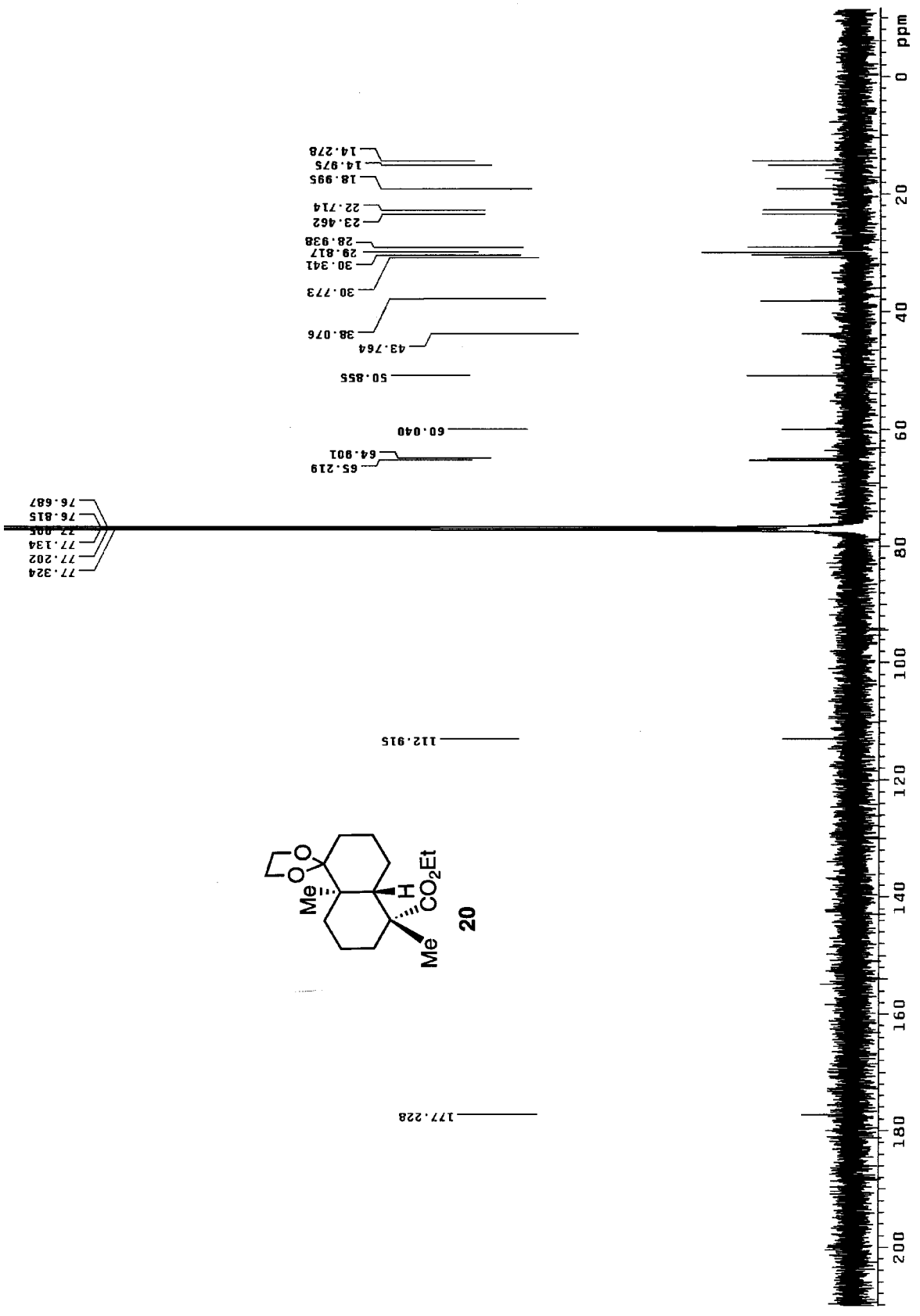
44.919
51.320
60.511
65.001
65.343
77.318
77.204
77.007
76.688
113.365
126.166
147.227
169.562
Me
CO2Et
18
ppm
0
20
40
60
80
100
120
140
160
180
200
169
173
177



20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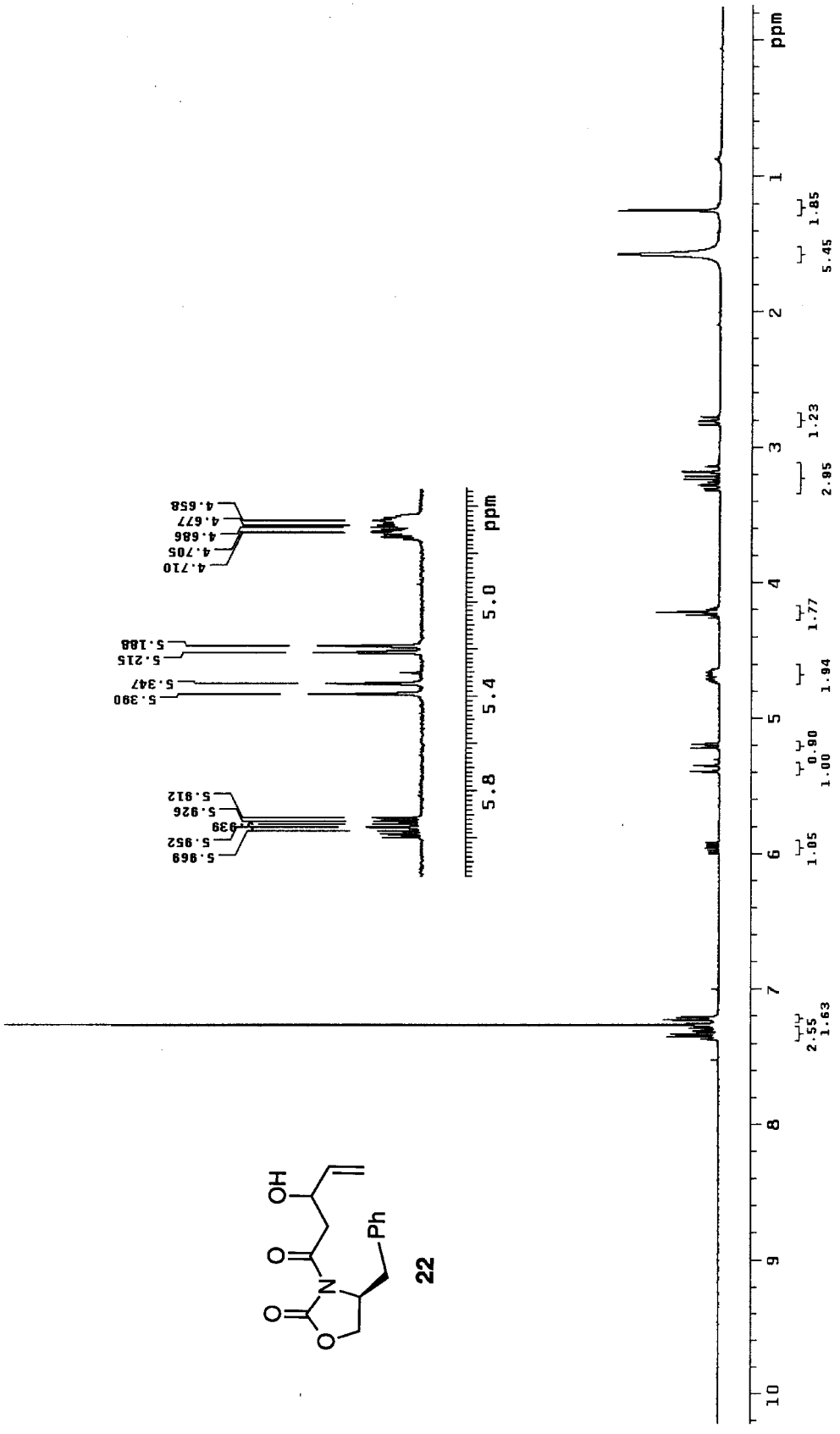
14.975
18.995
22.714
23.462
28.938
29.817
30.341
30.773
38.076
43.764
50.855
60.040
64.801
65.219
76.687
76.815
77.005
77.134
77.202
77.324
112.915
177.228
Me
H
CO2Et
Me
20
0
20
40
60
80
100
120
140
160
180
200
ppm


OH
O
N
O
O
Ph
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4.677
4.686
4.705
4.710
5.188
5.215
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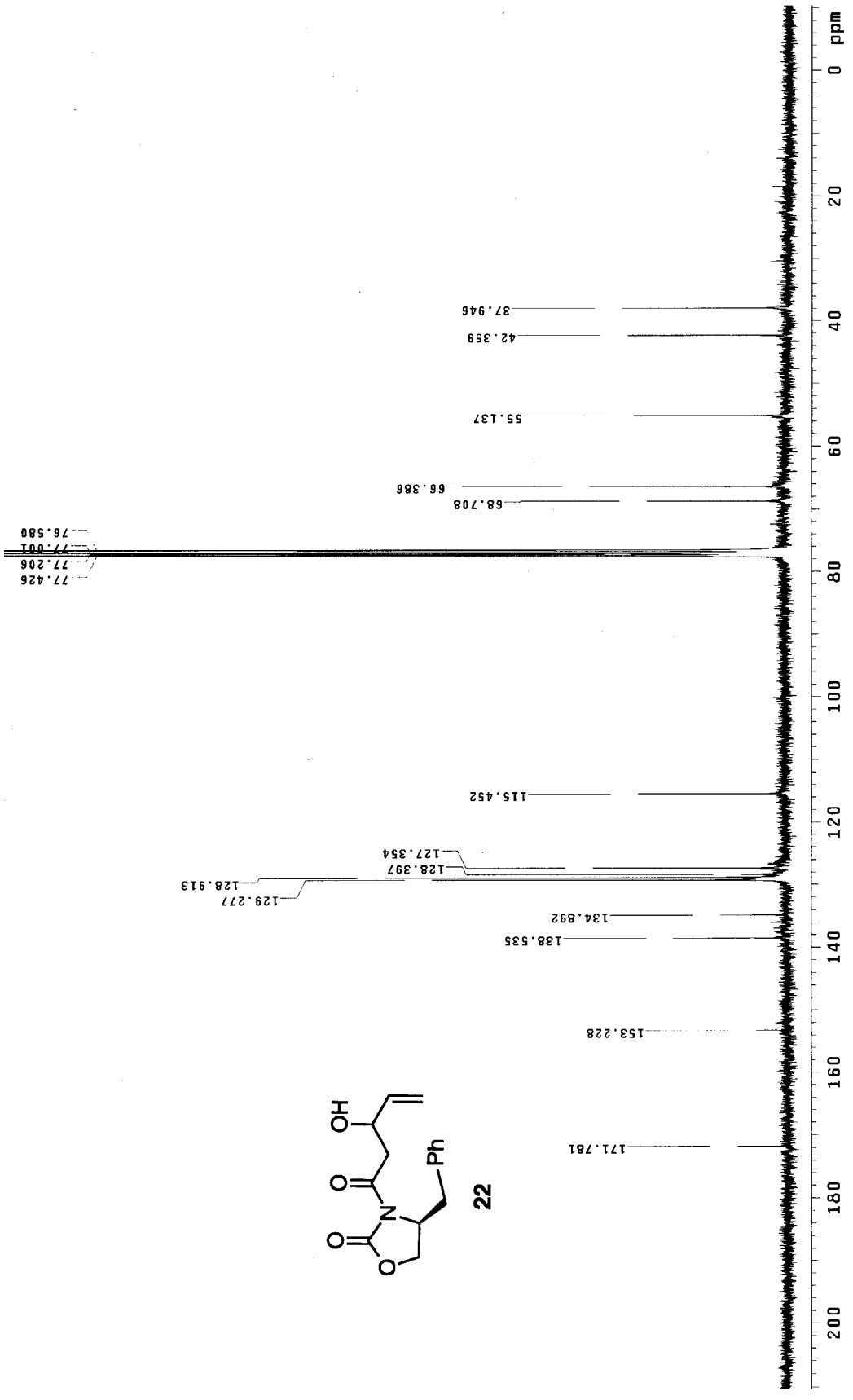
5.380
5.912
5.926
5.939
5.952
5.969
5.8
5.4
5.0
ppm
ppm
1.63
2.55
1.05
1.00
0.90
1.94
1.77
2.95
1.23
5.45
1.85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138.535
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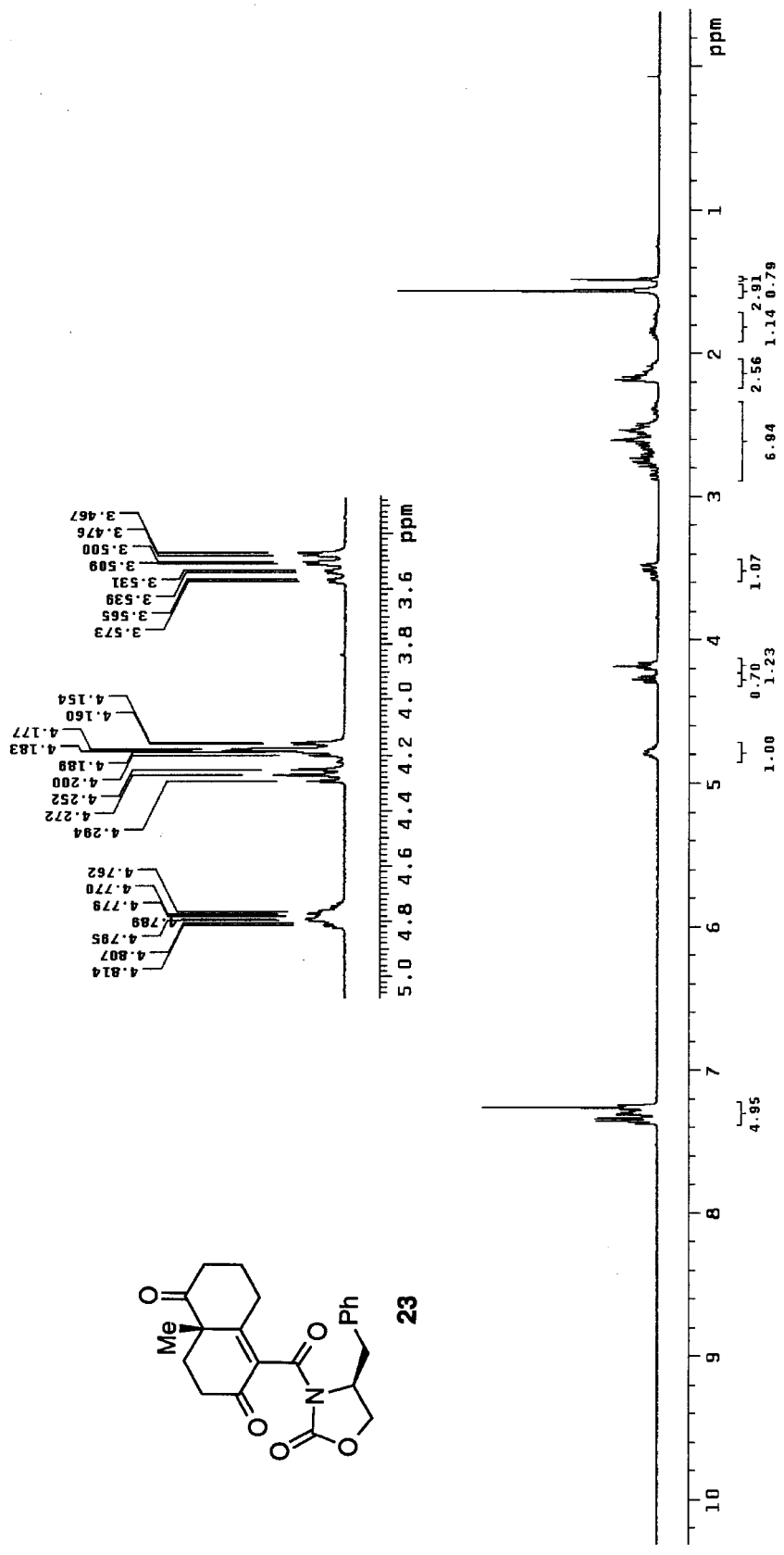
129.277
128.913
128.397
127.354
115.452
77.426
77.206
77.001
76.580
68.708
66.386
55.137
42.359
37.946
0 ppm
20
40
60
80
100
120
140
160
180
200
OH
O
N
O
O
Ph
22


Me
O
O
N
O
O
O
Ph
23
4.814
4.807
4.785
4.769
4.779
4.770
4.762
4.294
4.272
4.252
4.200
4.189
4.183
4.177
4.160
4.154
3.573
3.565
3.539
3.531
3.509
3.500
3.476
3.467
5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 ppm
ppm
1 2 3 4 5 6 7 8 9 10
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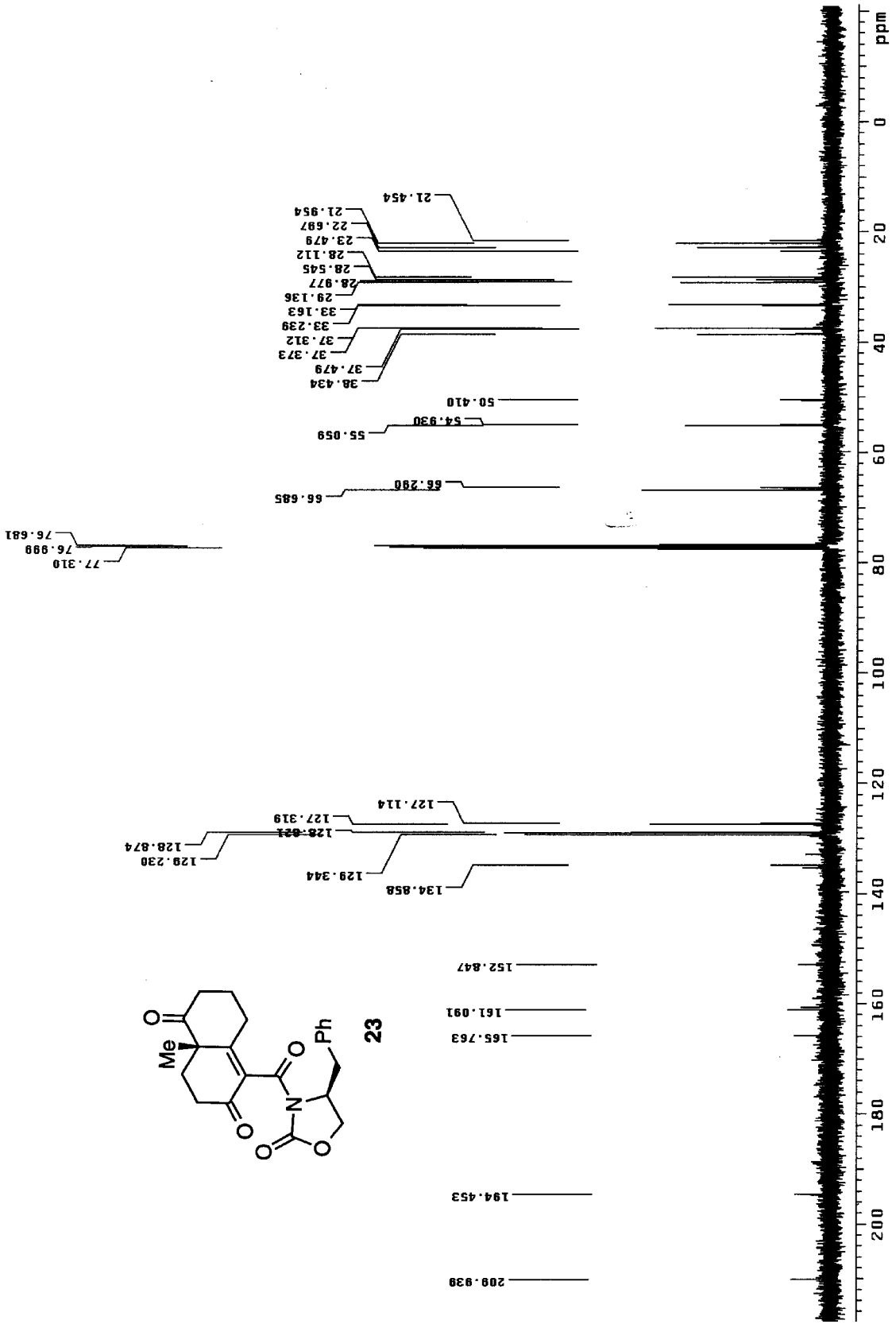
1.07
6.84
2.56
2.91
1.14 0.79


ppm
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20
40
60
80
100
120
140
160
180
200
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28.545
28.977
29.136
33.163
33.239
37.312
37.373
37.479
38.434
50.410
54.930
55.059
66.280
66.685
76.681
76.999
77.310
127.114
127.319
128.021
128.874
129.230
129.344
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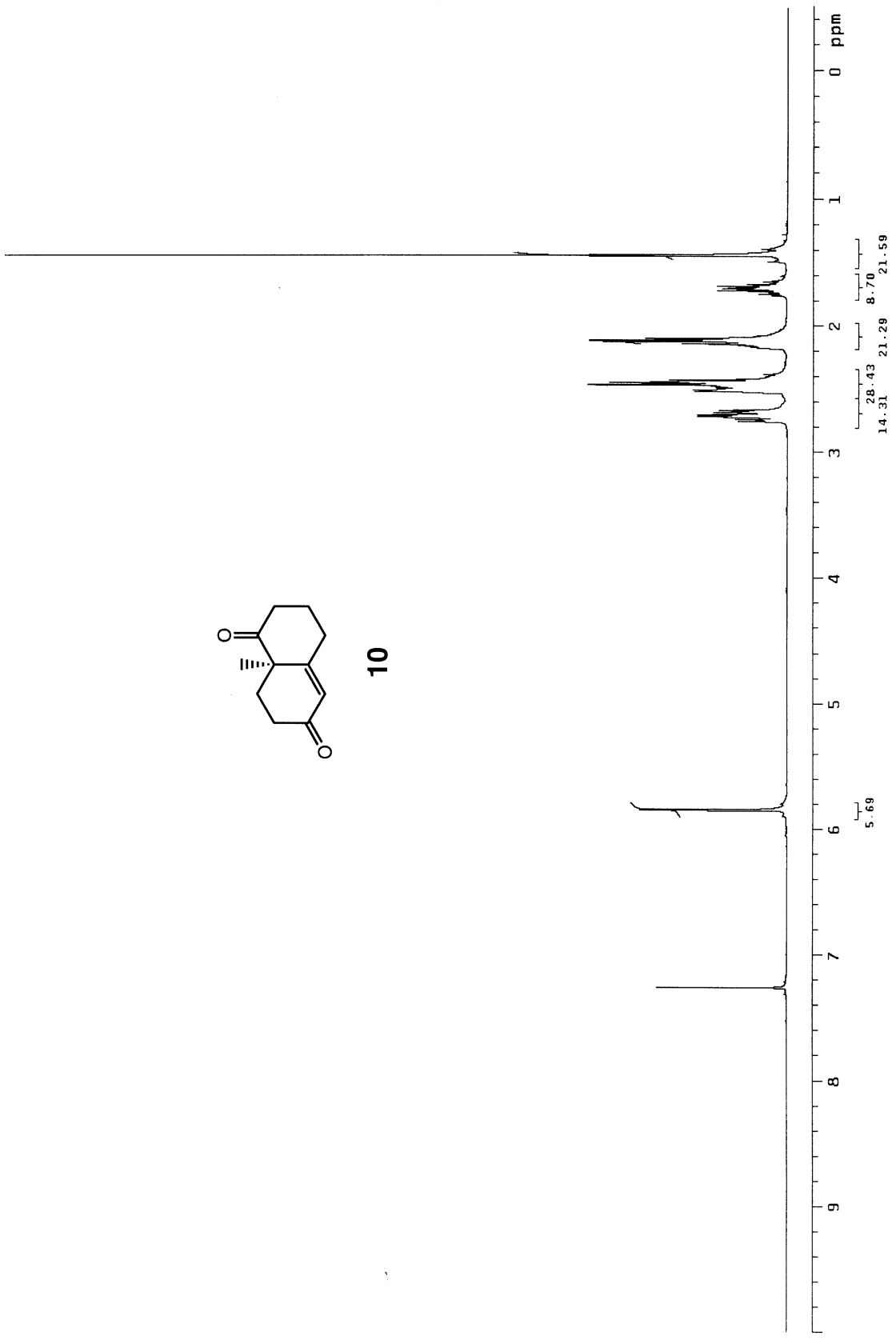
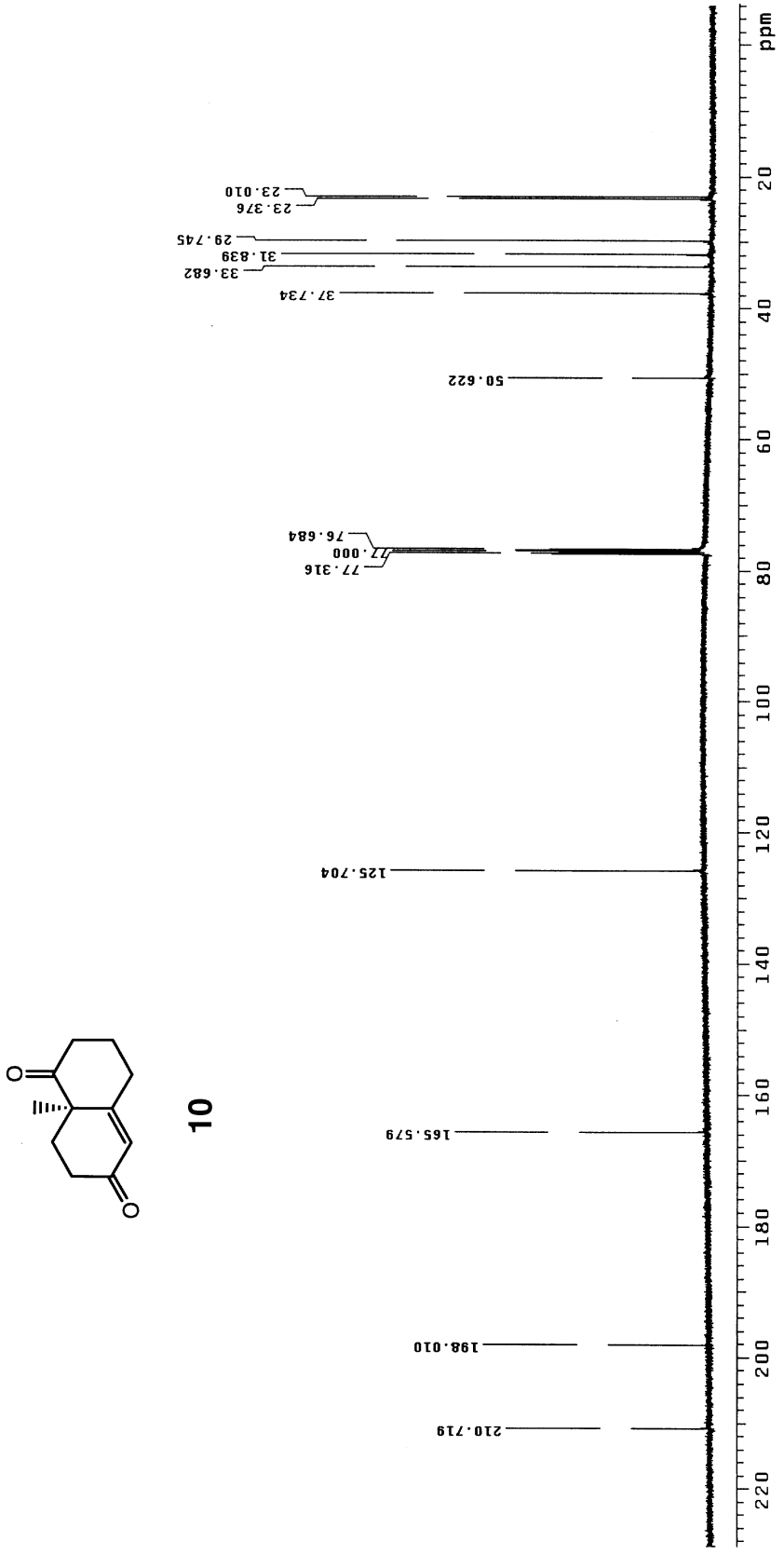
152.847
161.091
165.763
194.453
208.939
Me
Ph
O
N
O
O
O
O
23


10
0 ppm
1
2
3
4
5
6
7
8
9
5.69
14.31
28.43
21.29
8.70
21.59


ppm
20
40
60
80
100
120
140
160
180
200
220
210.719
198.010
165.579
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77.316
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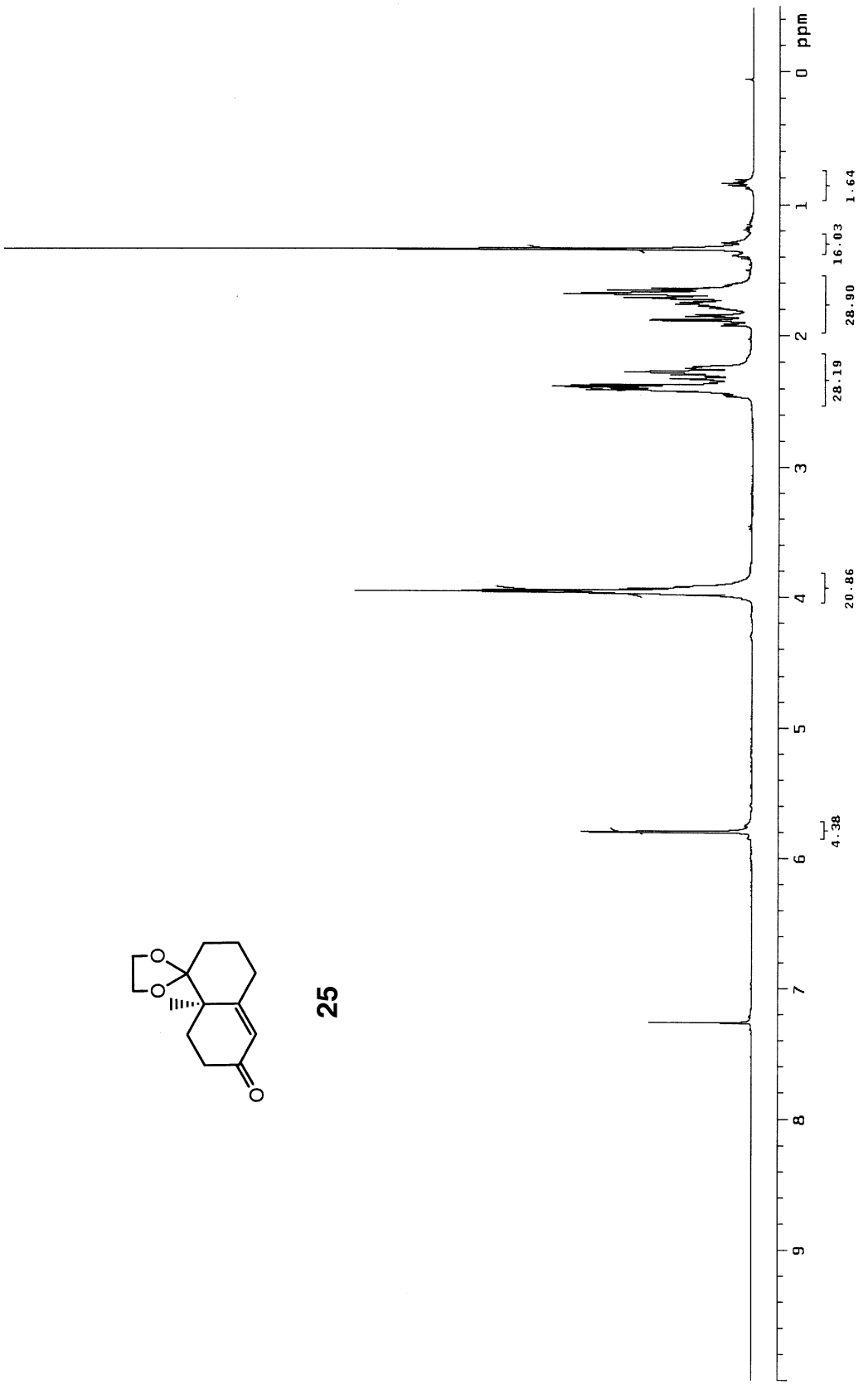
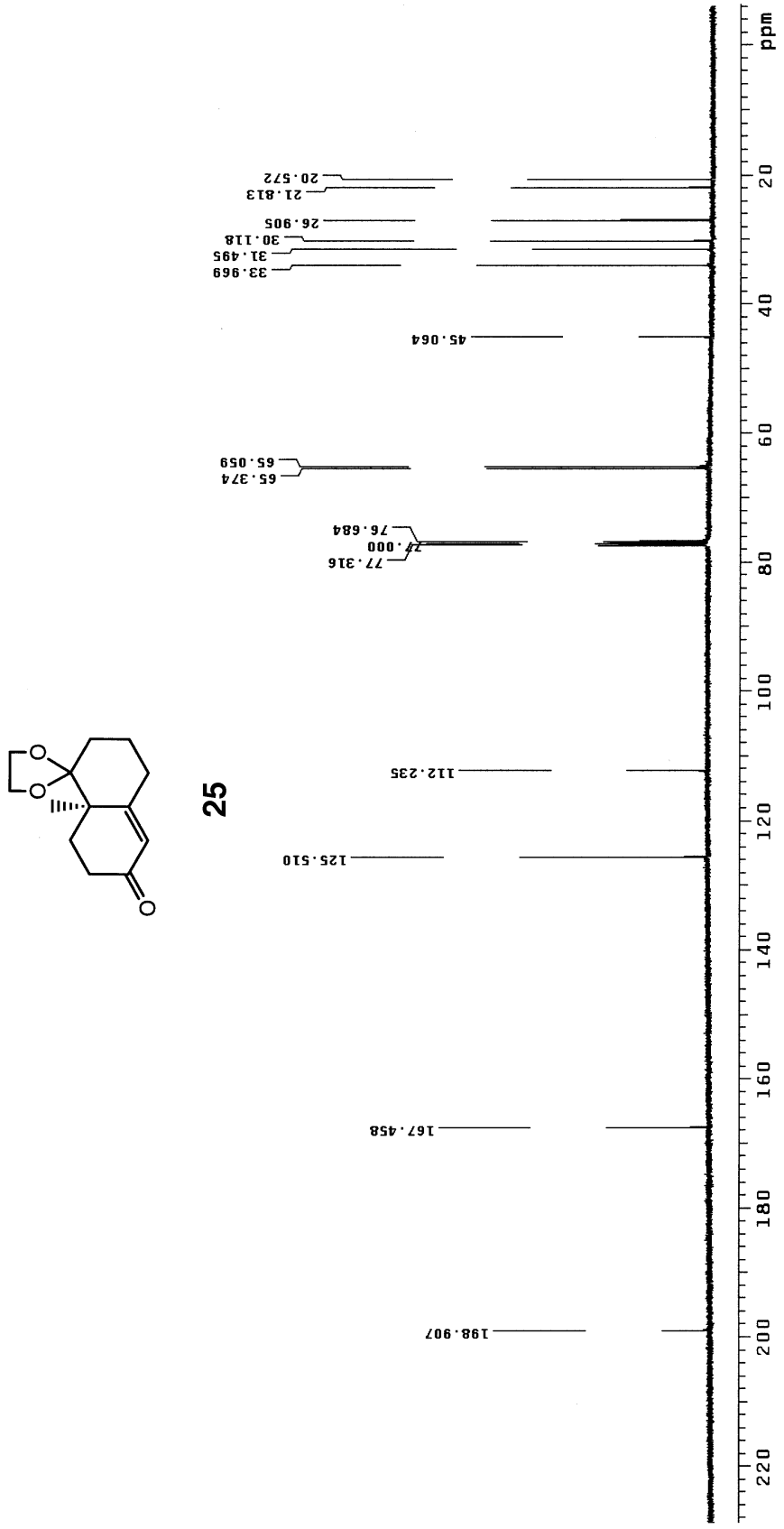
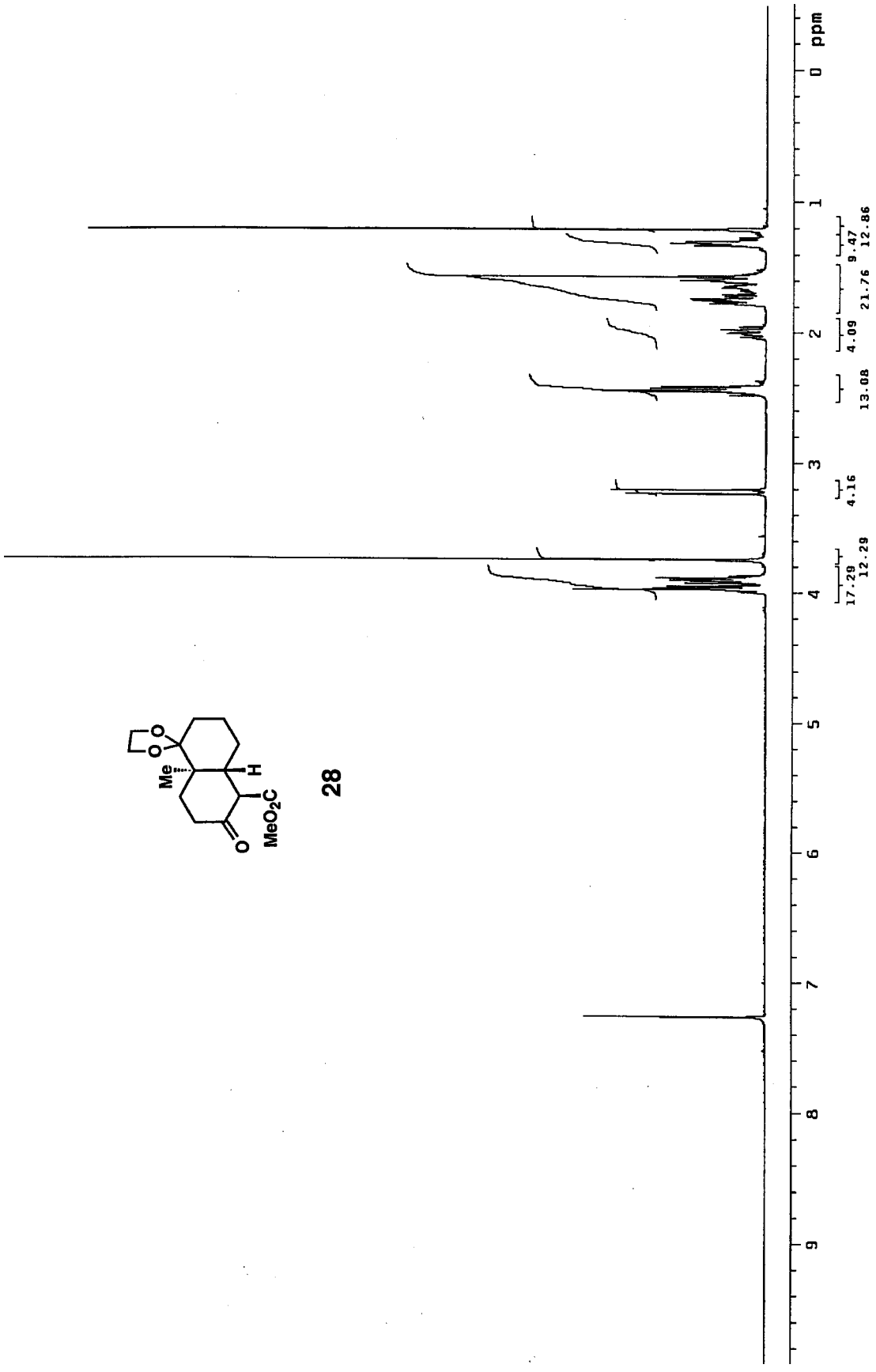
76.684
50.622
37.734
33.682
31.839
29.745
23.376
23.010

10


25
O
O
O
9
8
7
6
5
4
3
2
1
0 ppm
4.38
20.86
28.19
28.90
16.03
1.64


25
198.907
167.458
125.510
112.235
77.316
77.000
76.684
65.374
65.059
45.064
33.969
31.495
30.118
26.905
21.813
20.572
ppm


Me
H
MeO2C
O
O
O
28
ppm
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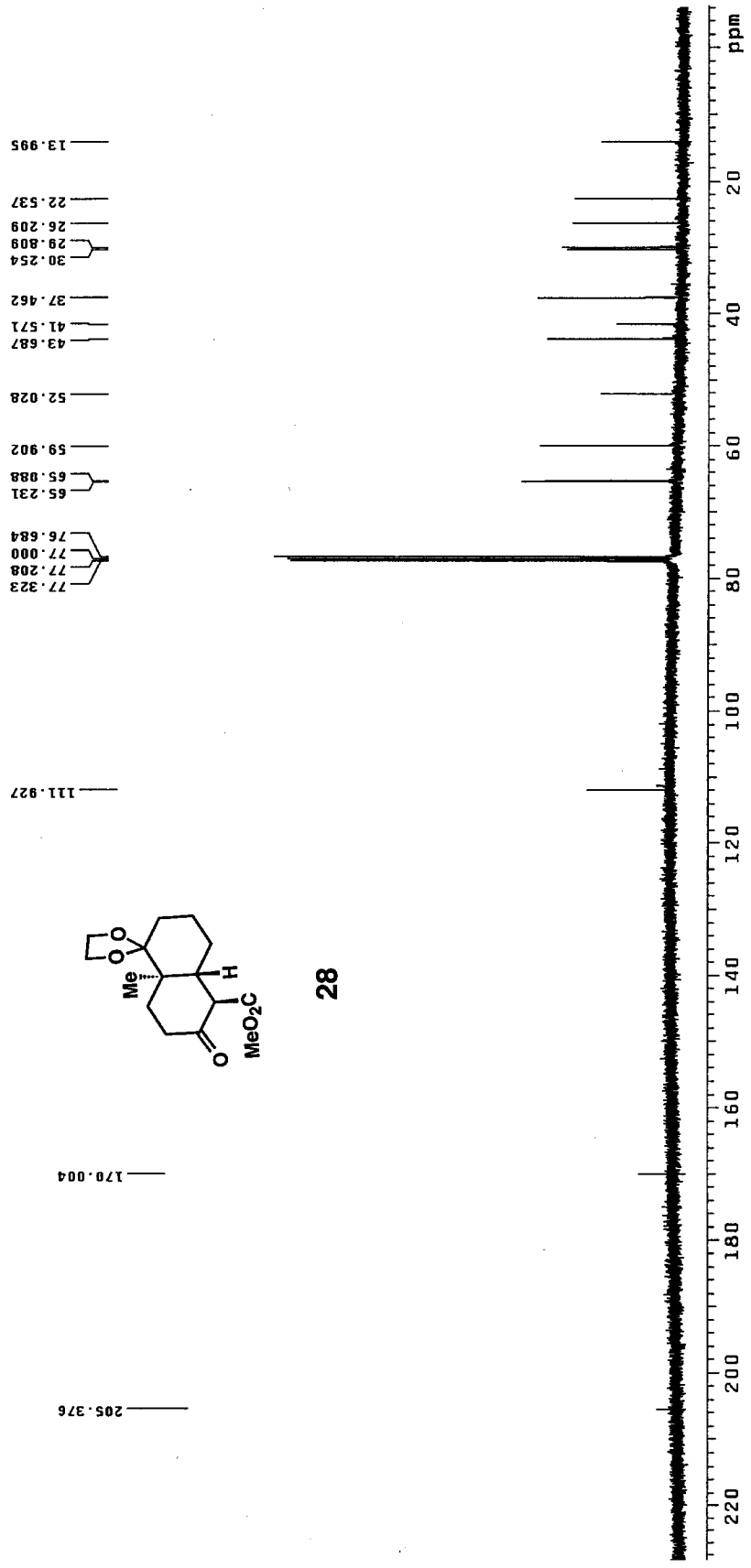
1
2
3
4
5
6
7
8
9
17.20
12.29
4.16
13.08
4.09
21.76
9.47
12.86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77.000
76.684
65.231
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52.028
43.687
41.571
37.462
30.254
29.809
26.209
22.537
13.995
ppm
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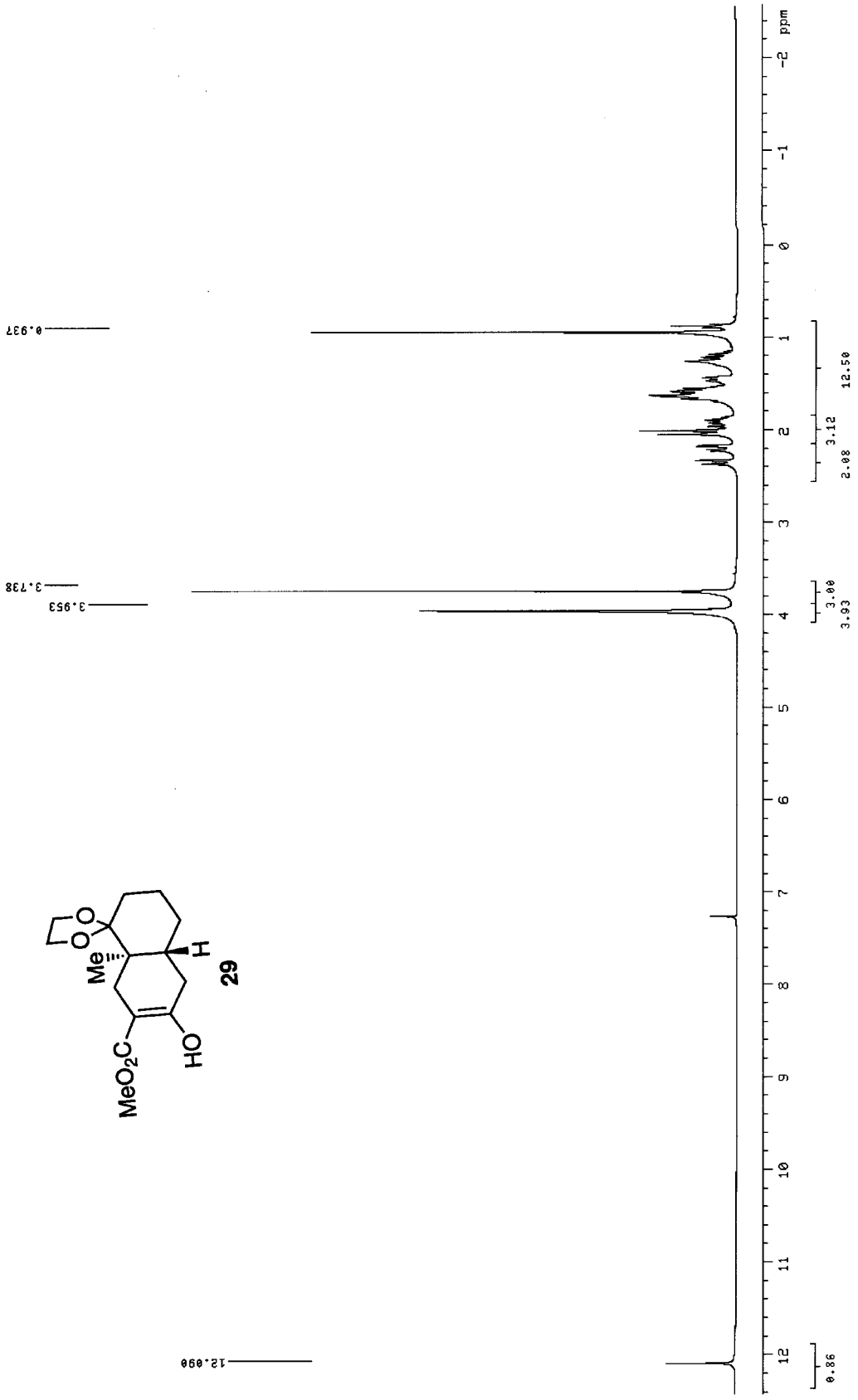
40
60
80
100
120
140
160
180
200
220
28
Me
H
MeO2C
O
O
O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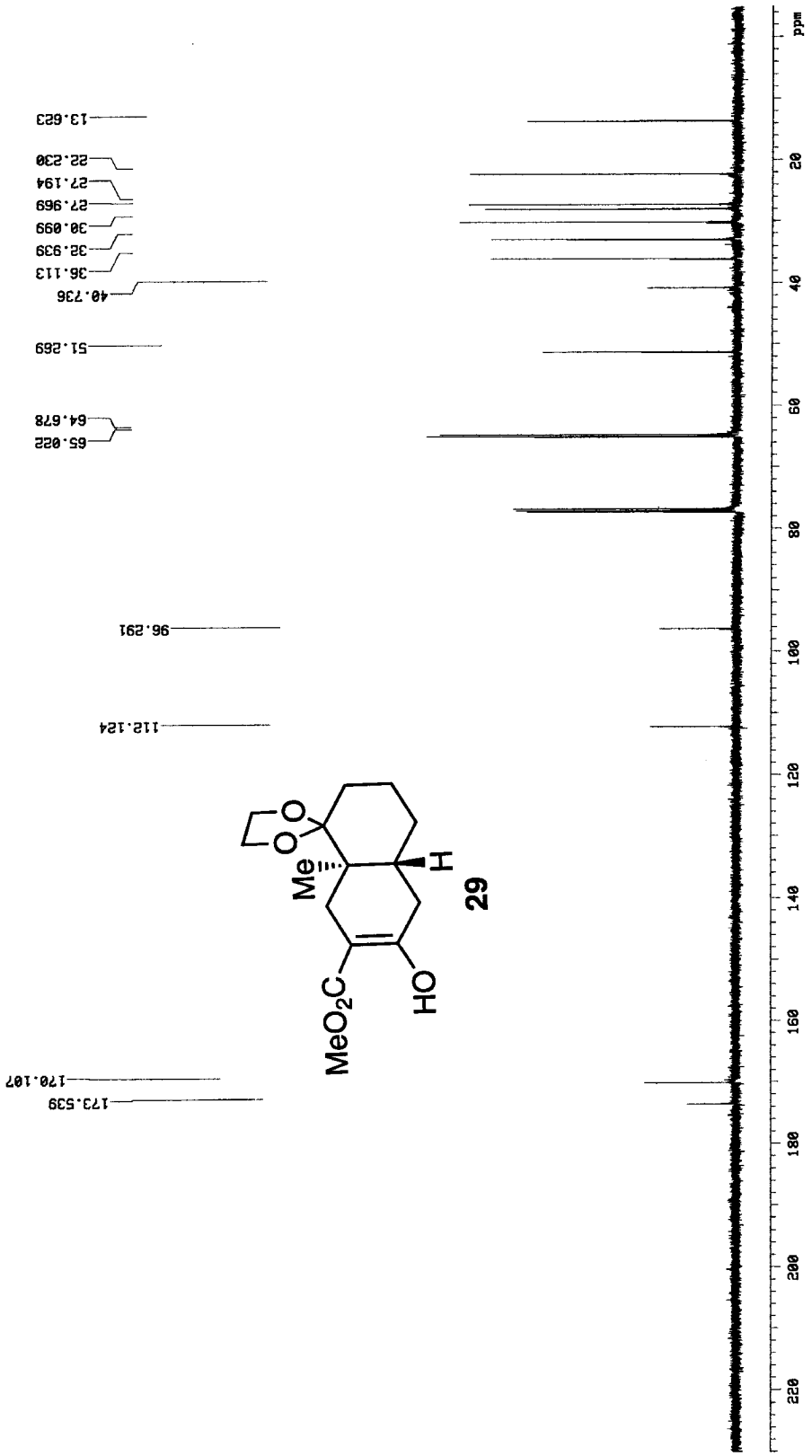
0.86
Me
H
O
O
HO
MeO2C
29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27.194
27.969
30.099
32.939
36.113
40.736
51.269
64.678
65.022
96.291
112.124
170.107
173.539
ppm
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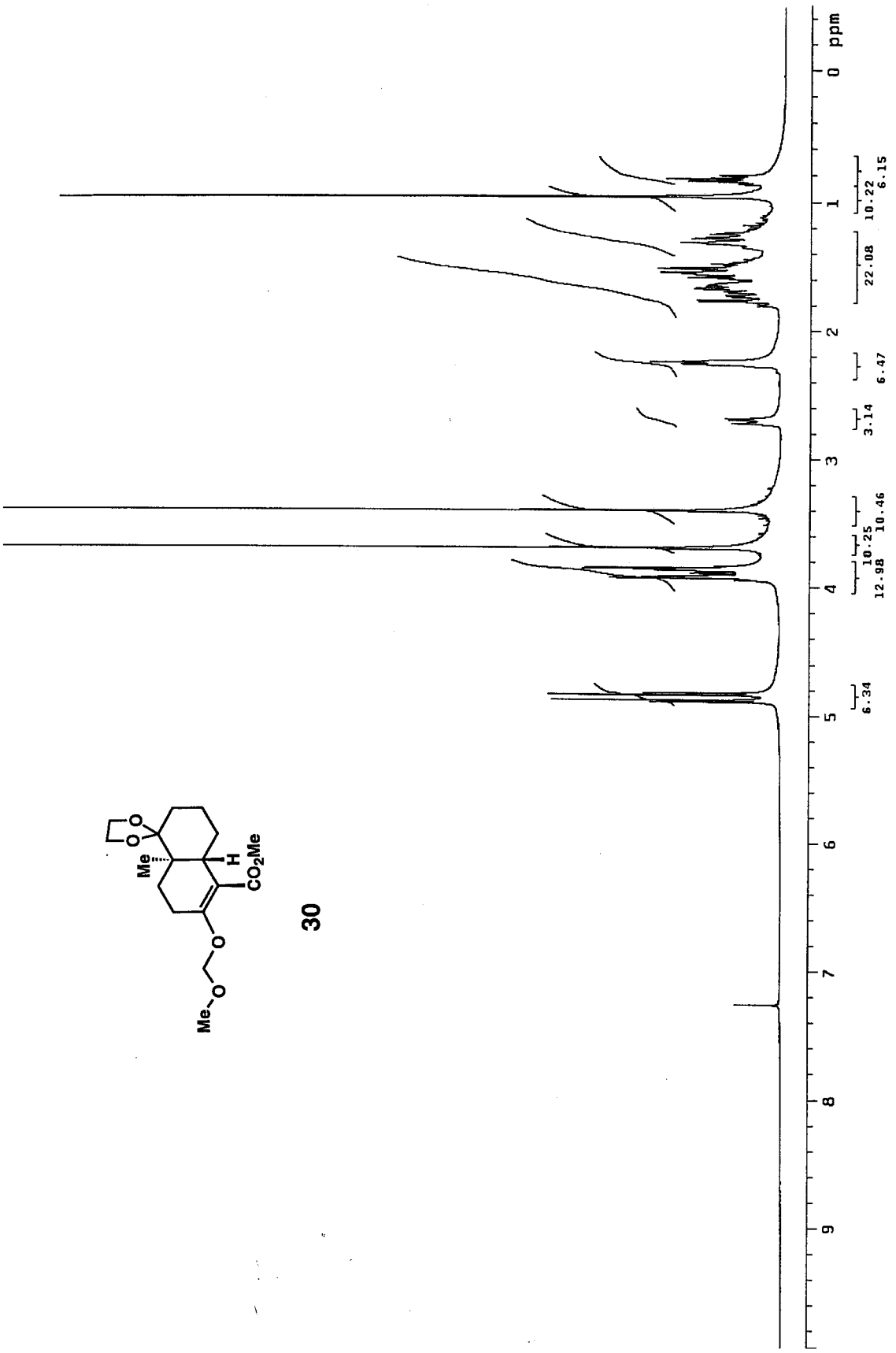
80
100
120
140
160
180
200
220
Me
H
O
O
MeO2C
HO
29


Me
H
CO2Me
Me
O
O
O
O
30
ppm
0
1
2
3
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9
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10.46
3.14
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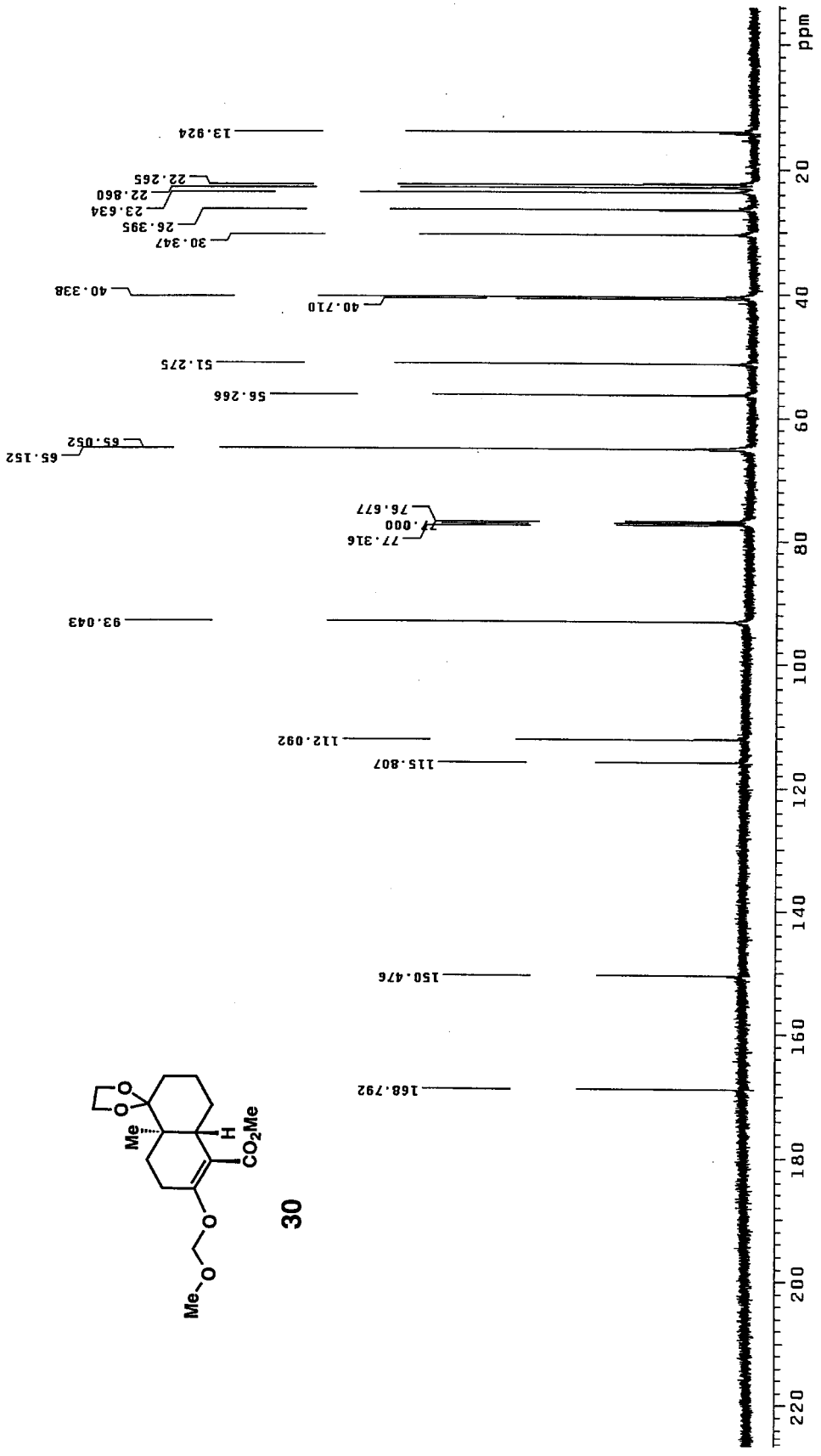
22.08
10.22
6.15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40.710
51.275
56.266
65.052
65.152
76.677
77.000
77.316
93.043
112.092
115.807
150.476
168.792
ppm
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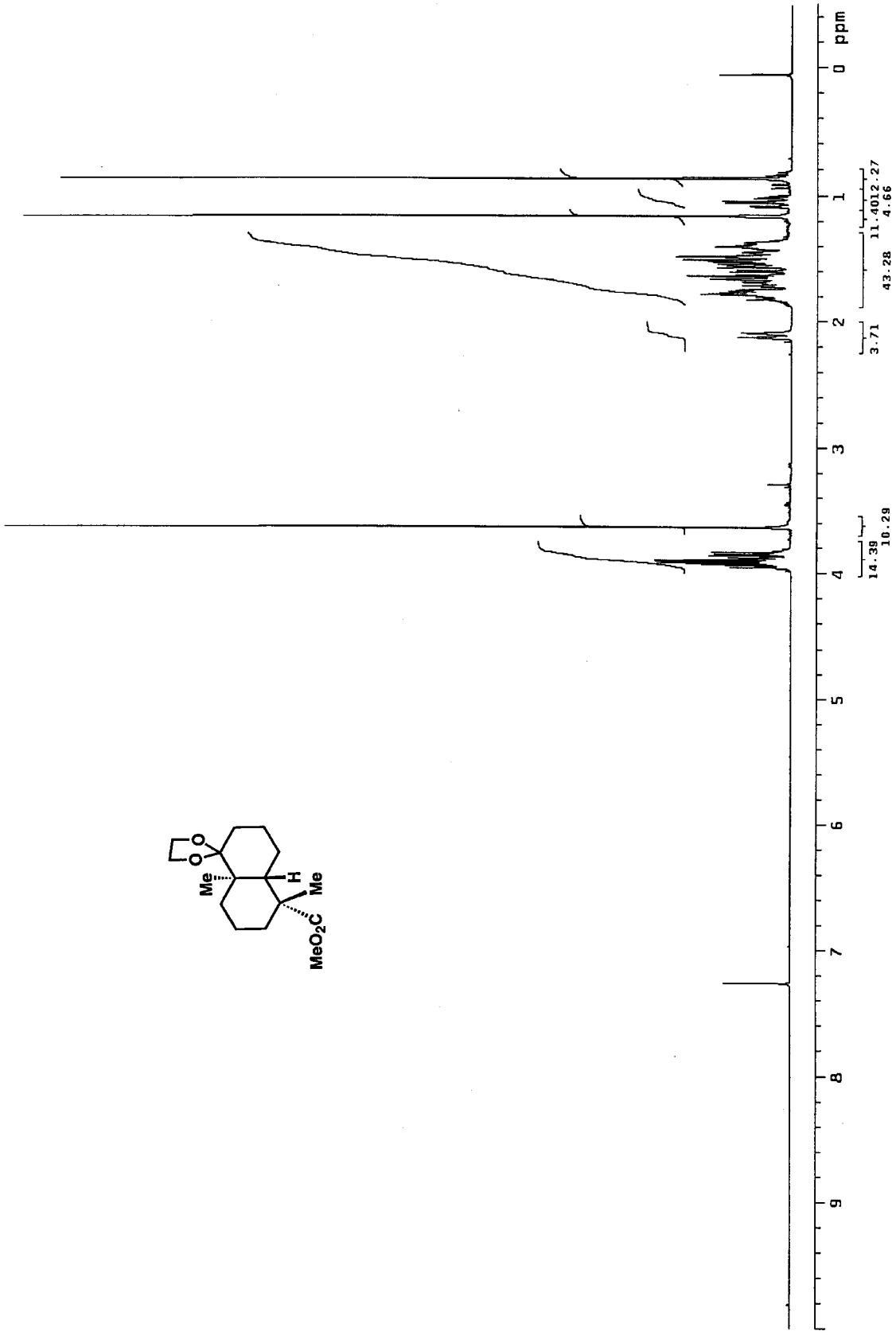
40
60
80
100
120
140
160
180
200
220
Me
H
CO2Me
O
O
O
O
Me
30


Me
H
Me
MeO2C
0 ppm
1
2
3
4
5
6
7
8
9
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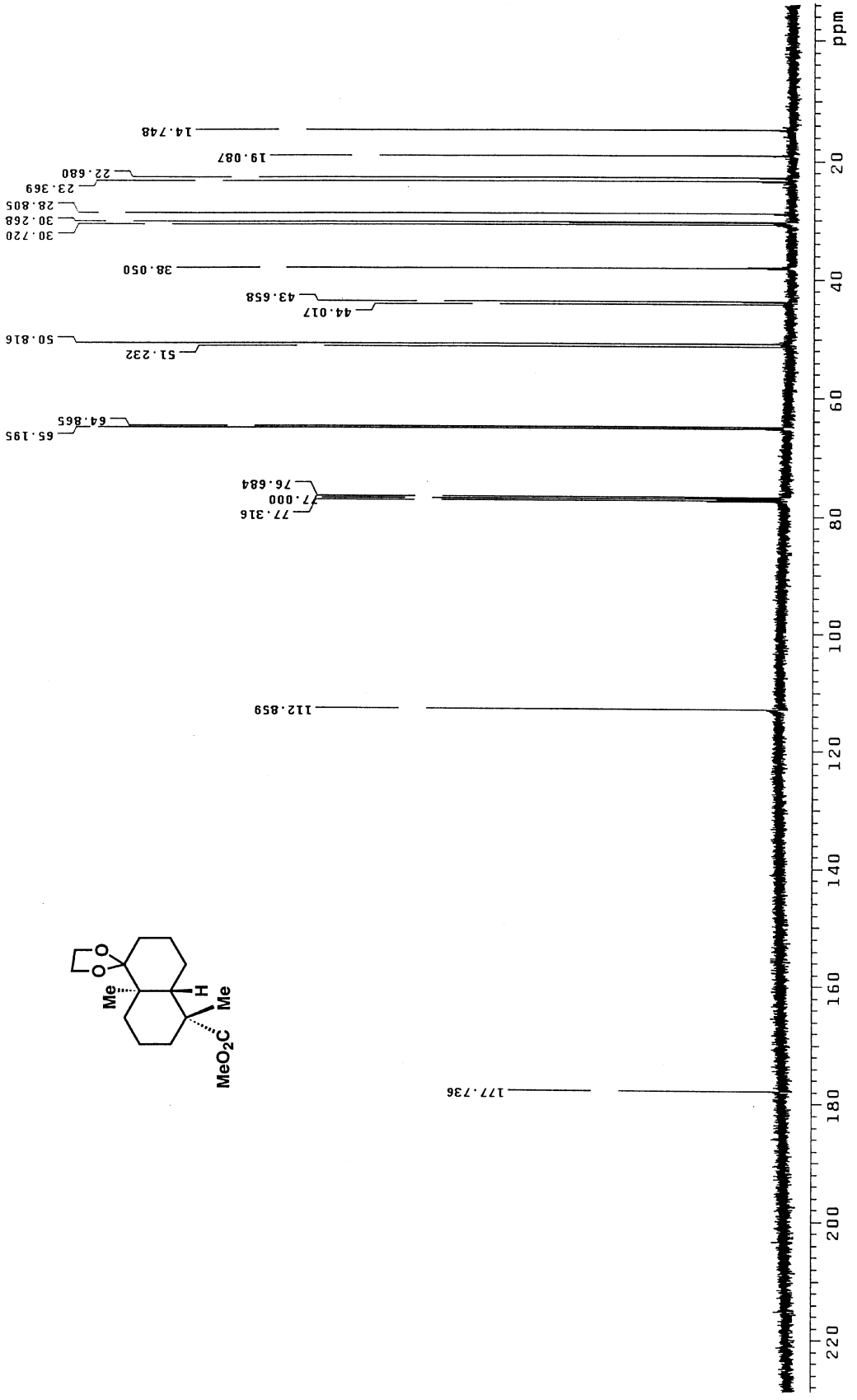
4.66
43.28
3.71
14.39
10.29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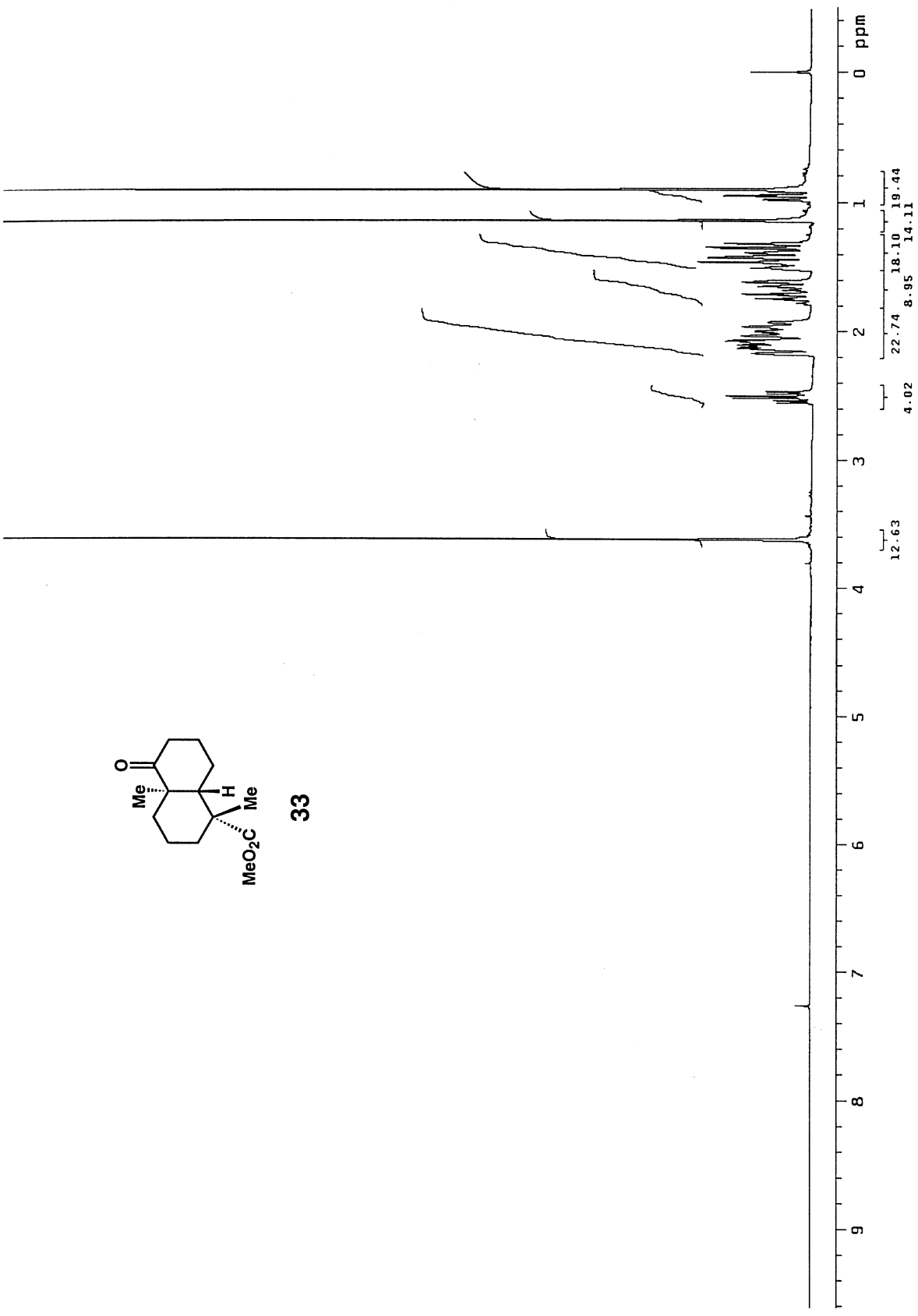
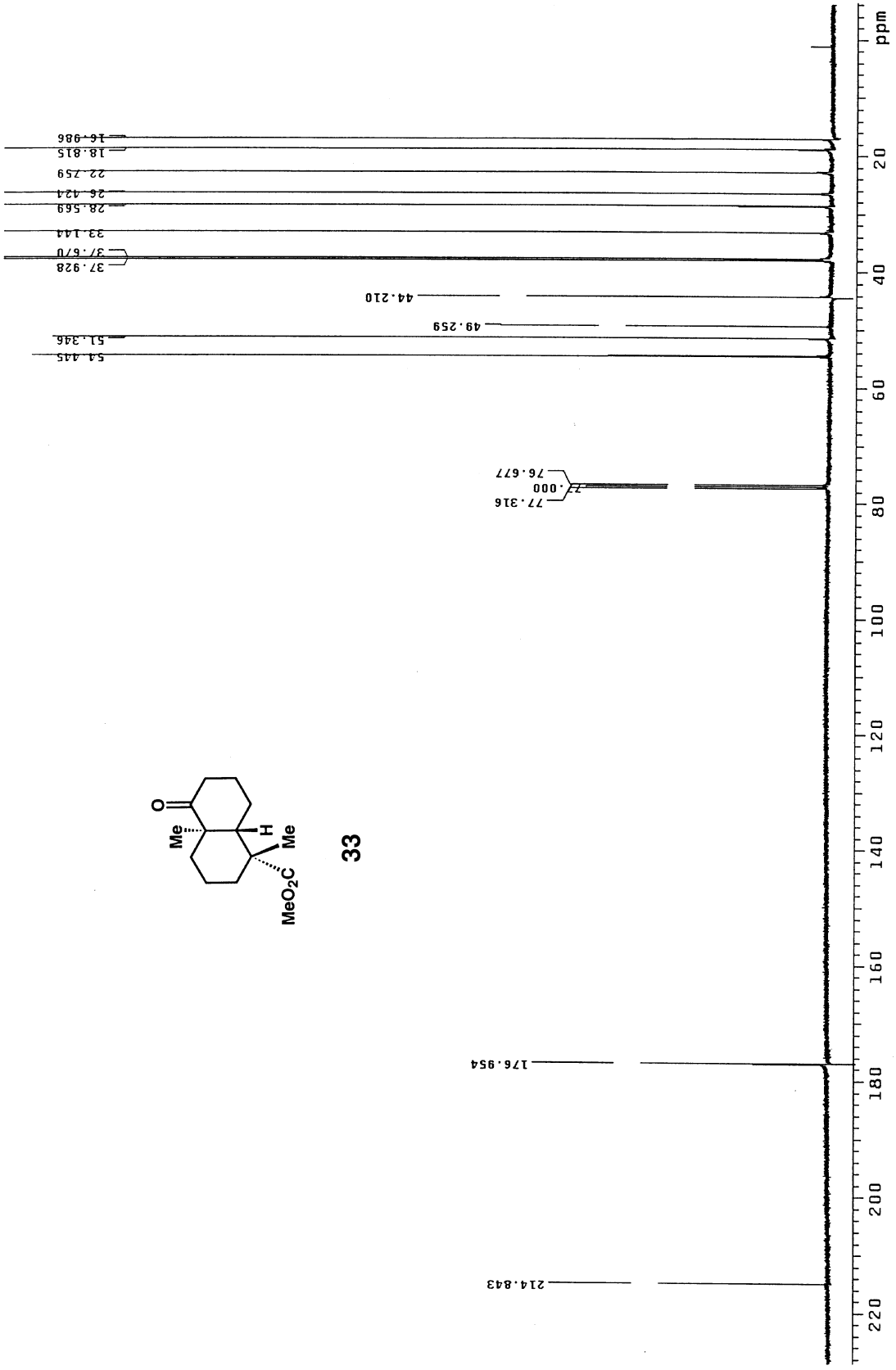
22.680
23.369
28.805
30.268
30.720
38.050
43.658
44.017
50.816
51.232
64.865
65.195
76.684
77.000
77.316
112.859
177.736
ppm
20
40
60
80
100
120
140
160
180
200
220
O
O
Me
H
Me
MeO2C


O
Me
H
Me
MeO2C
33
0 ppm
1
2
3
4
5
6
7
8
9
12.63
4.02
22.74
8.95
18.10
14.11
19.44


214.843
176.954
77.316
77.000
76.677
54.415
51.346
49.259
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37.928
37.670
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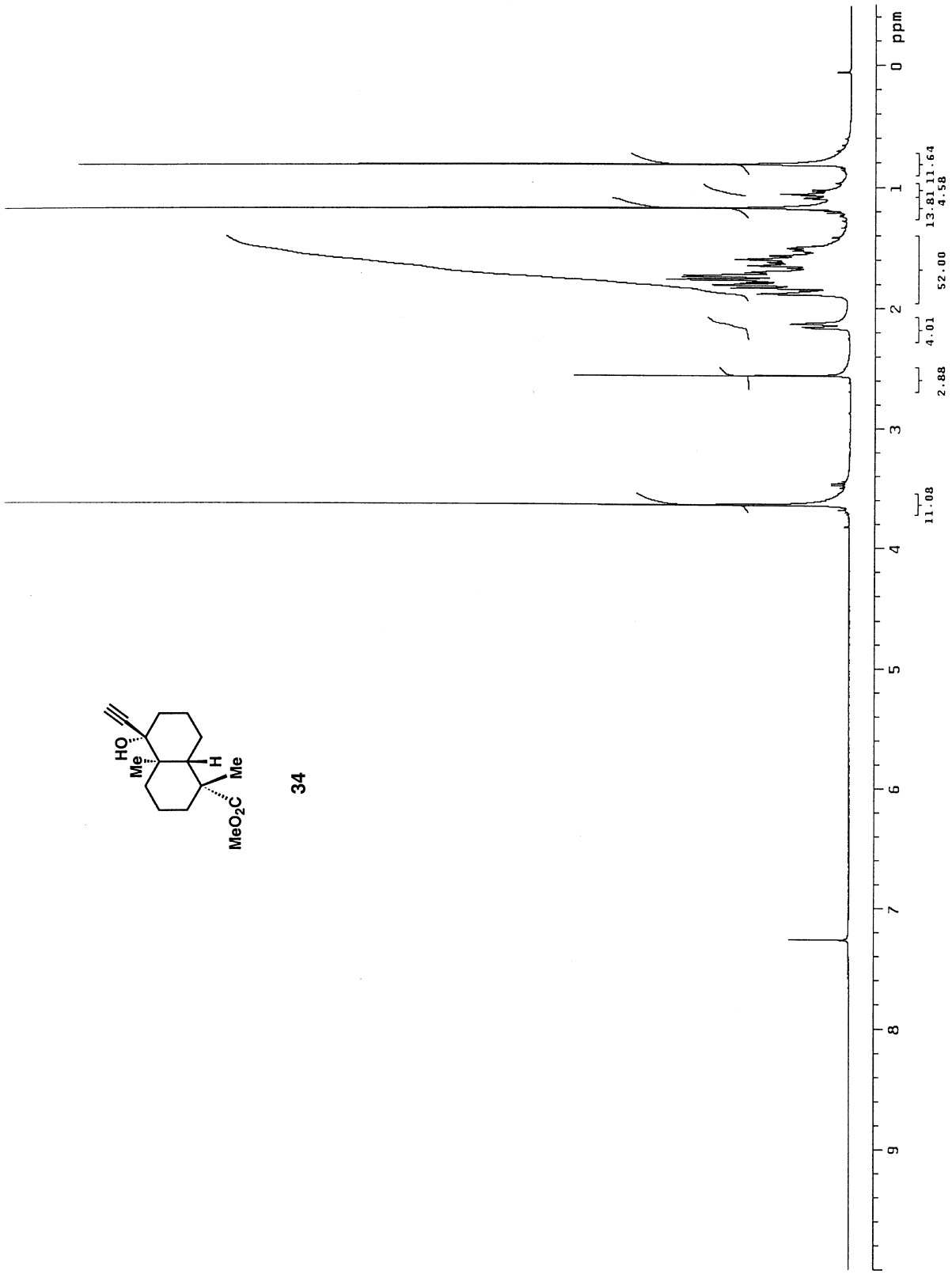
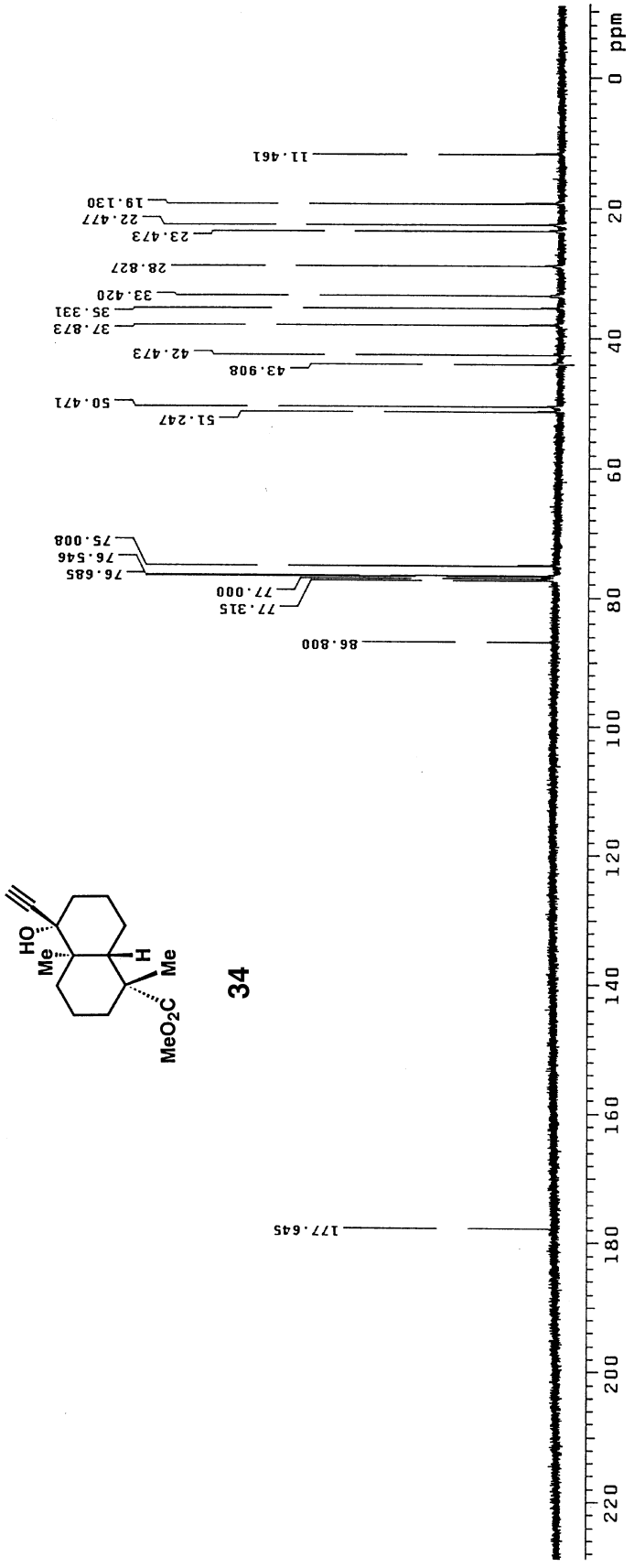
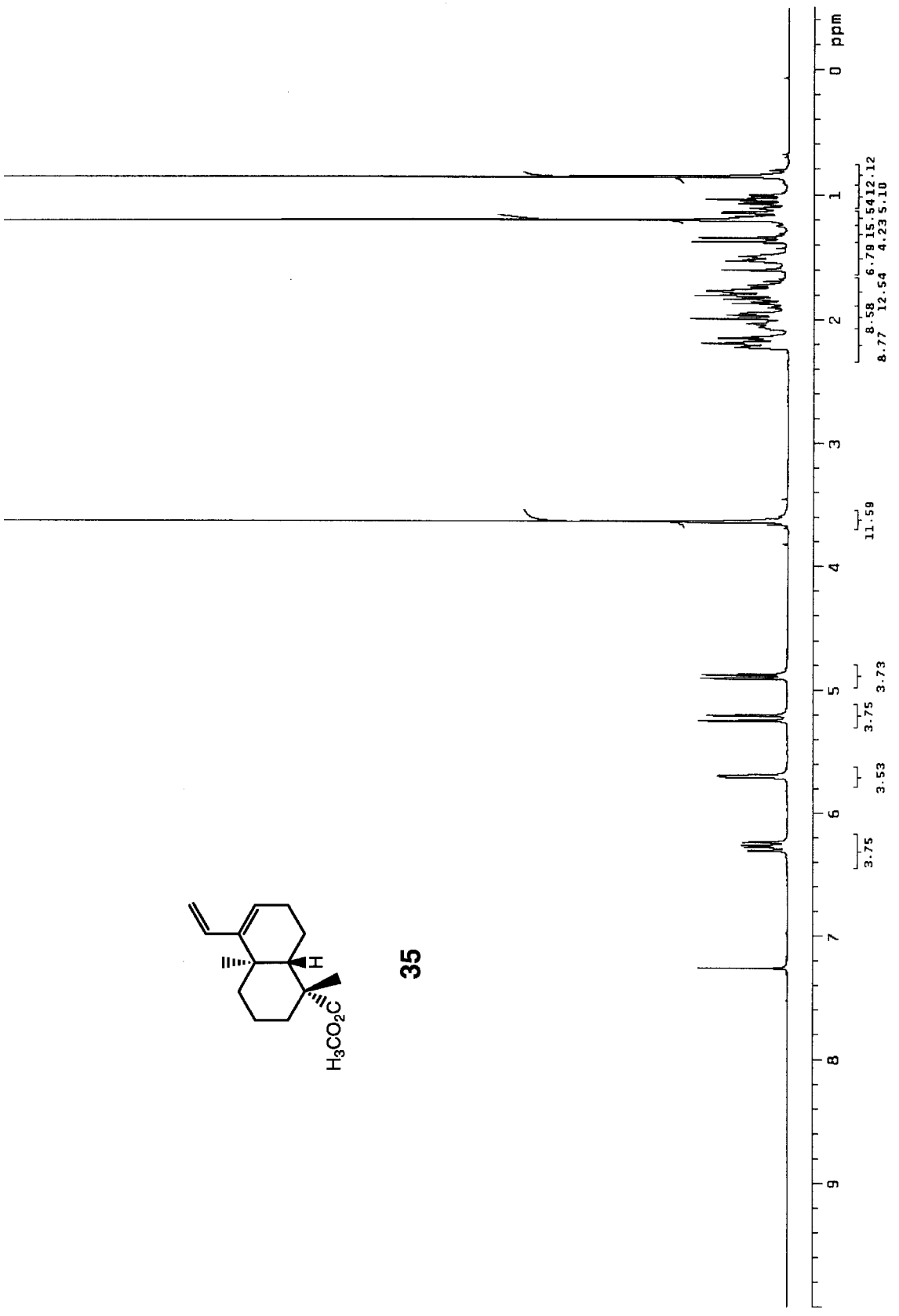
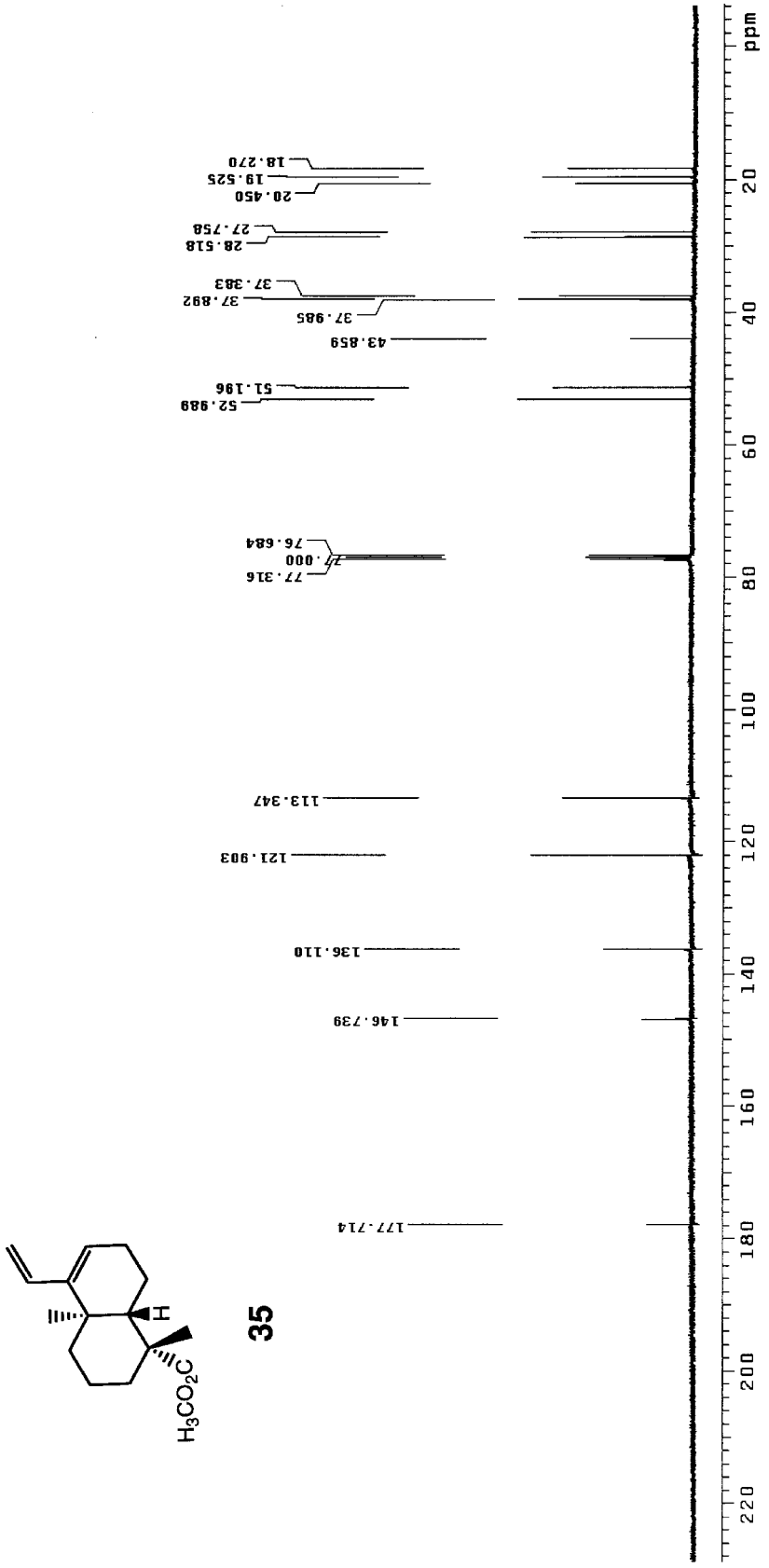
28.569
26.424
22.259
18.815
16.986
ppm
20
40
60
80
100
120
140
160
180
200
220
O
Me
H
Me
MeO2C

33

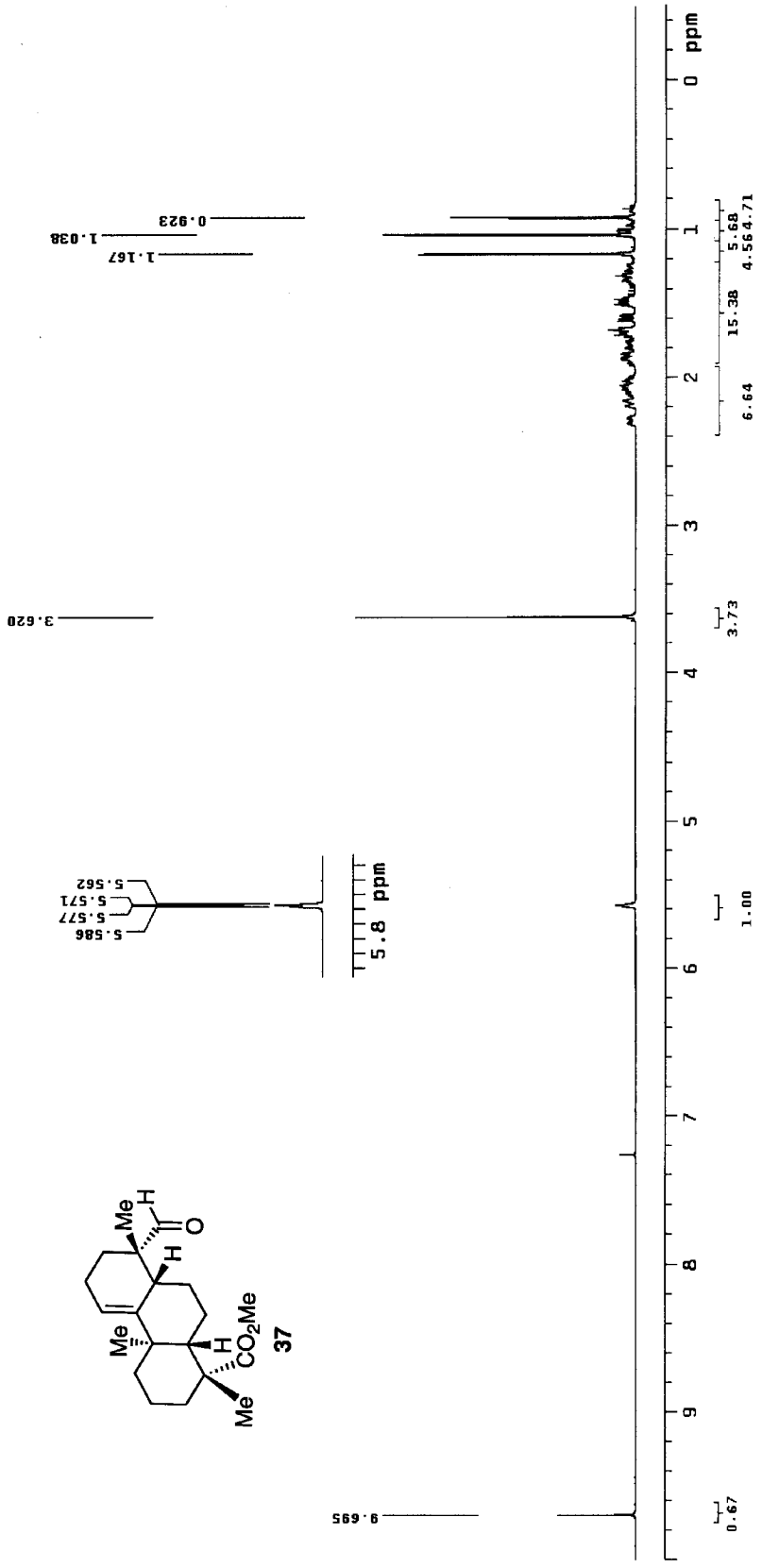

HO
Me
H
Me
MeO2C
34
0 ppm
1
2
3
4
5
6
7
8
9
11.08
2.88
4.01
52.00
13.81
11.64
4.58

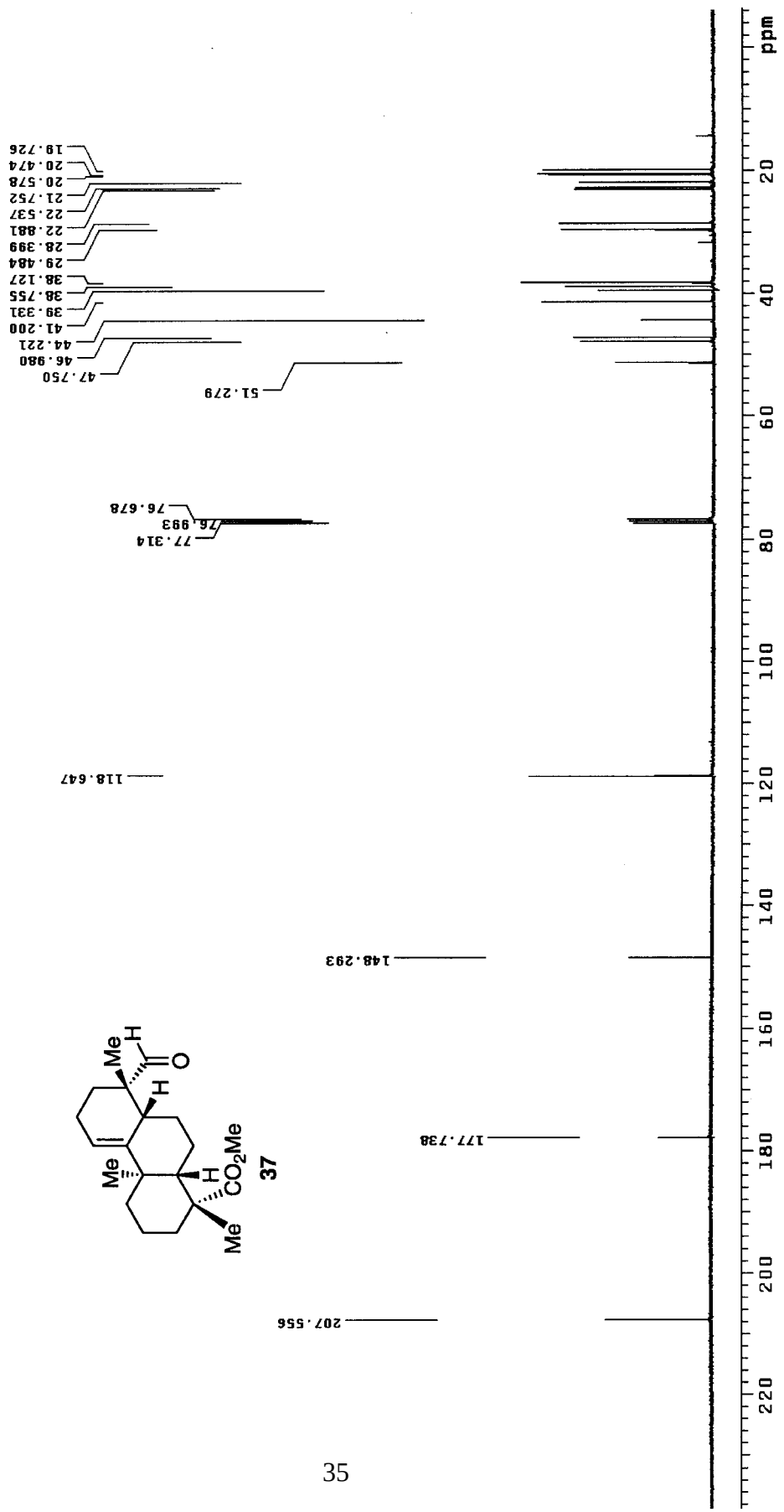

11.461
19.130
22.477
23.473
28.827
33.420
35.331
37.873
42.473
43.908
50.471
51.247
75.008
76.546
76.685
77.000
77.315
86.800
177.645
0 ppm
20
40
60
80
100
120
140
160
180
200
220
HO
Me
H
Me
MeO2C
34

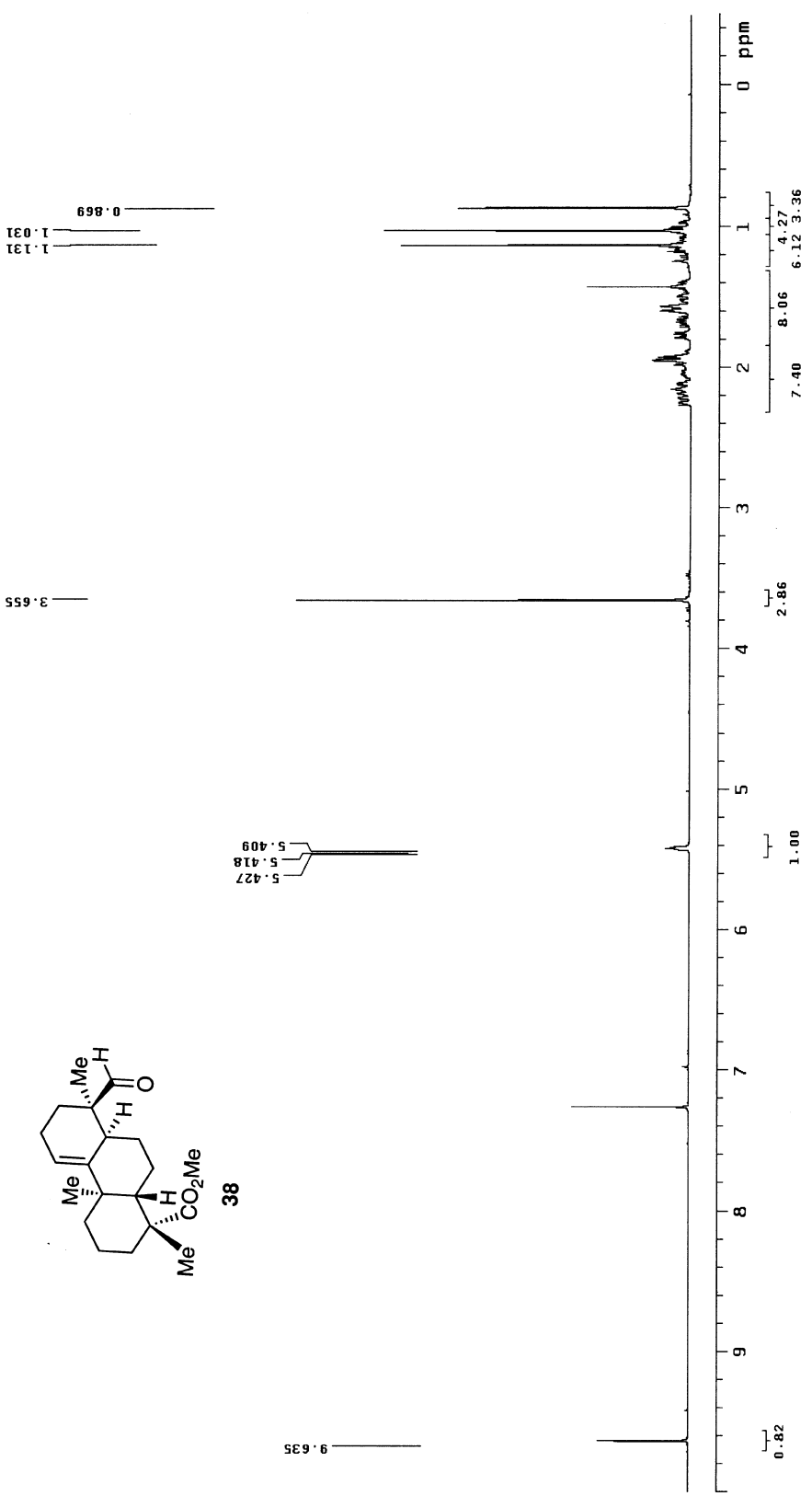

H
H3CO2C
35
ppm
0
1
2
3
4
5
6
7
8
9
3.75
3.53
3.75
3.73
11.59
8.77
8.58
12.54
6.79
15.54
4.23
12.12
5.10

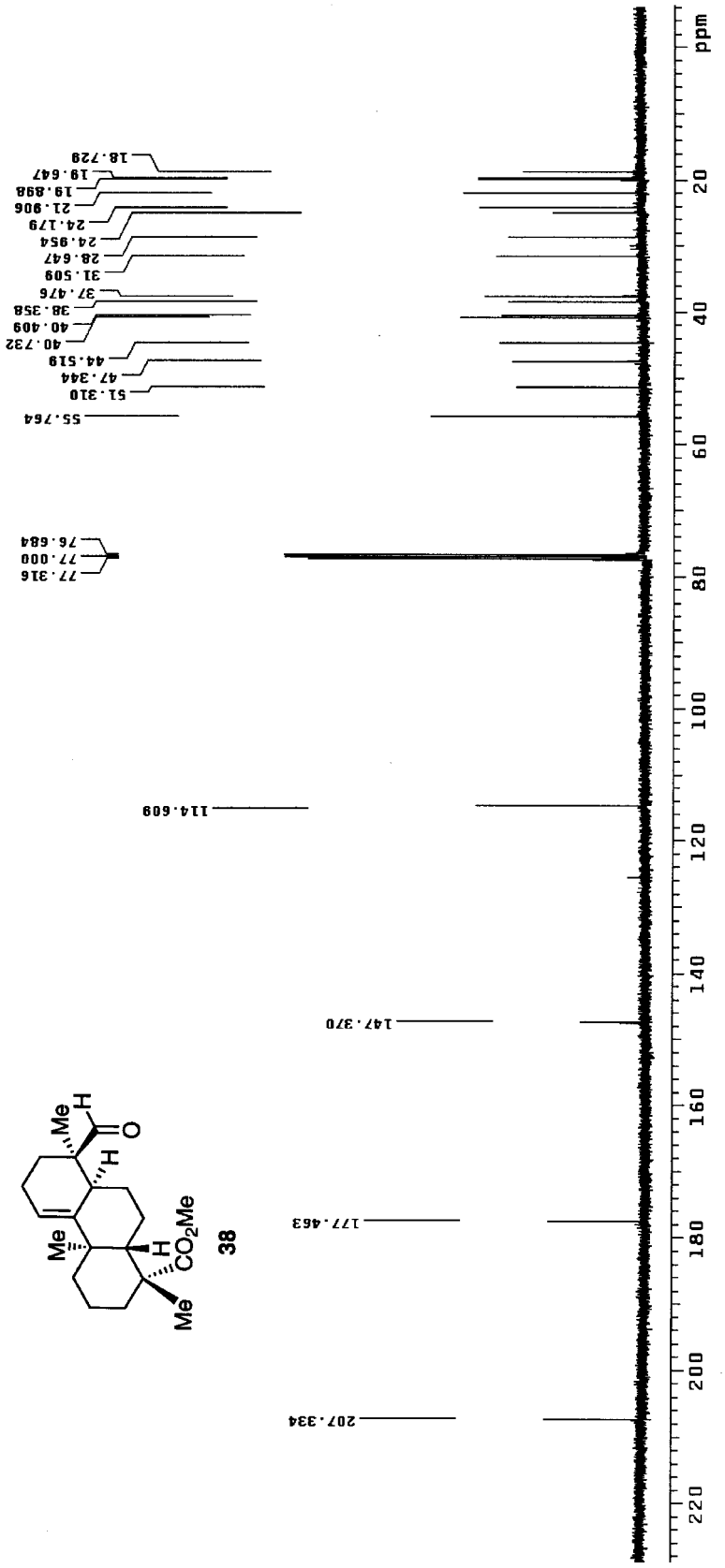

177.714
146.739
136.110
121.903
113.347
77.316
77.000
76.684
52.989
51.196
43.859
37.985
37.892
37.383
28.518
27.758
20.450
19.525
18.270
ppm
20
40
60
80
100
120
140
160
180
200
220
H
H_3CO_2C

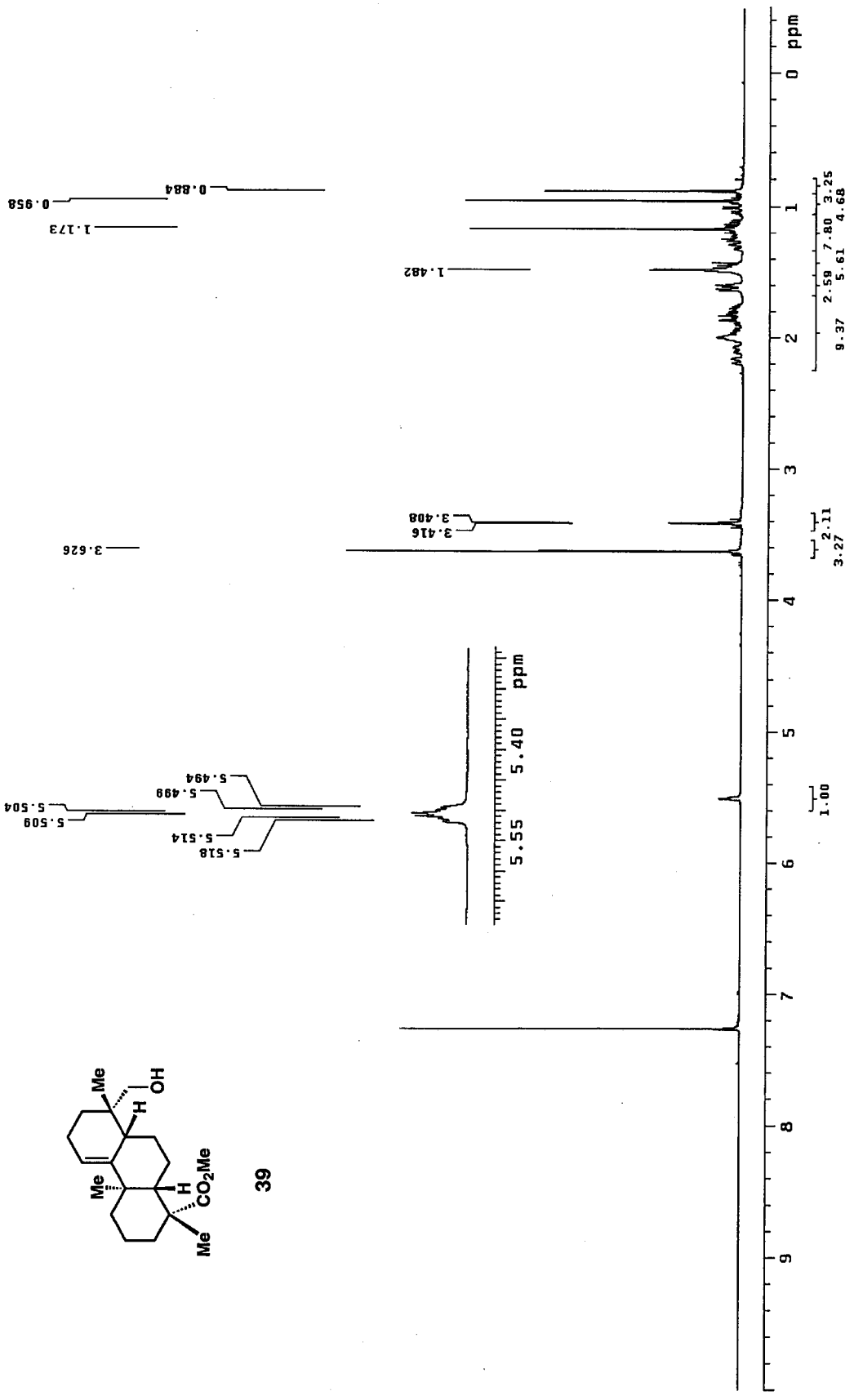
35


9.695
5.586
5.577
5.571
5.562
3.620
1.167
1.038
0.923
5.8 ppm
0 ppm
0.67
1.00
3.73
6.64
15.38
5.68
4.56
4.71
Me
H
O
H
Me
H
CO2Me
Me
37

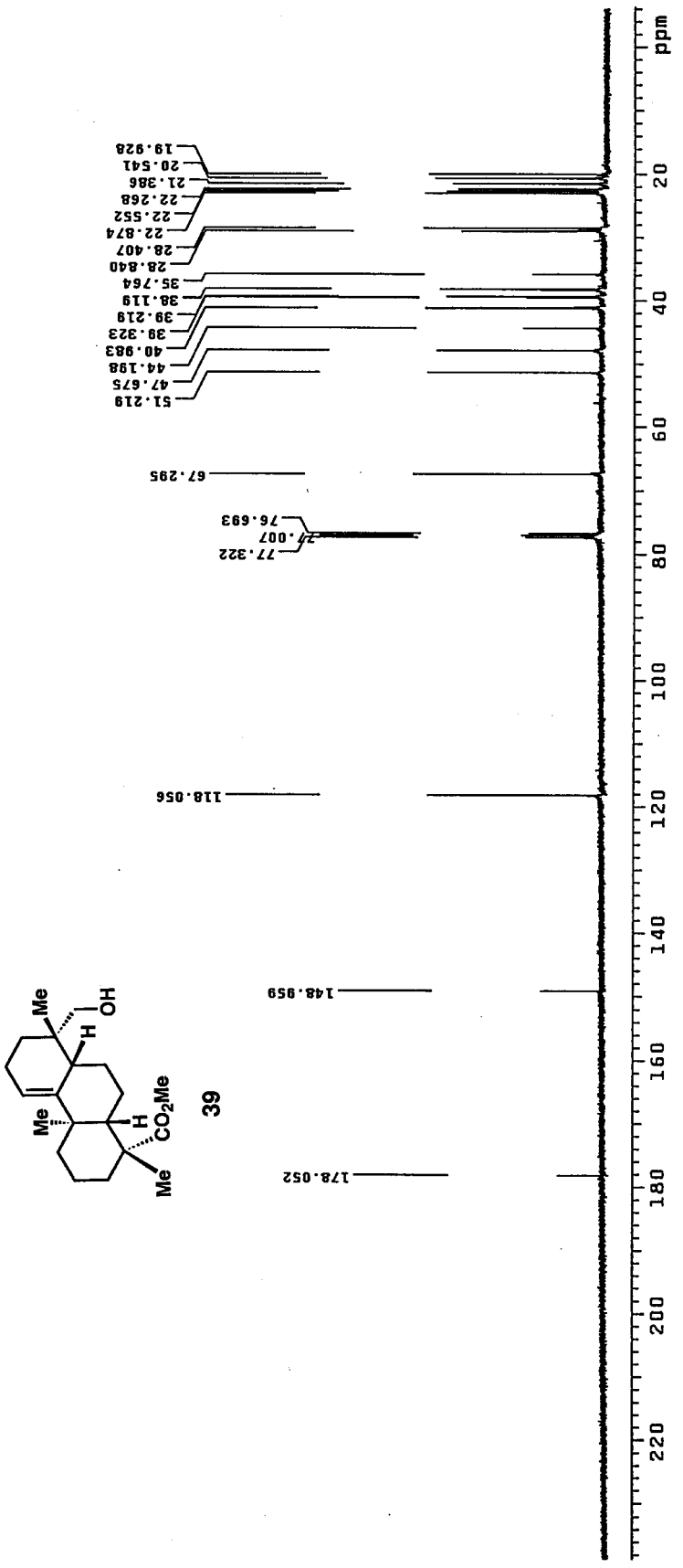

ppm
207.556
177.738
148.293
118.647
76.678
76.993
77.314
51.279
47.750
46.980
44.221
41.200
39.331
38.755
38.127
29.484
28.399
22.881
22.537
21.752
20.578
20.474
19.726
Me
H
O
H
Me
H
CO2Me
Me
37

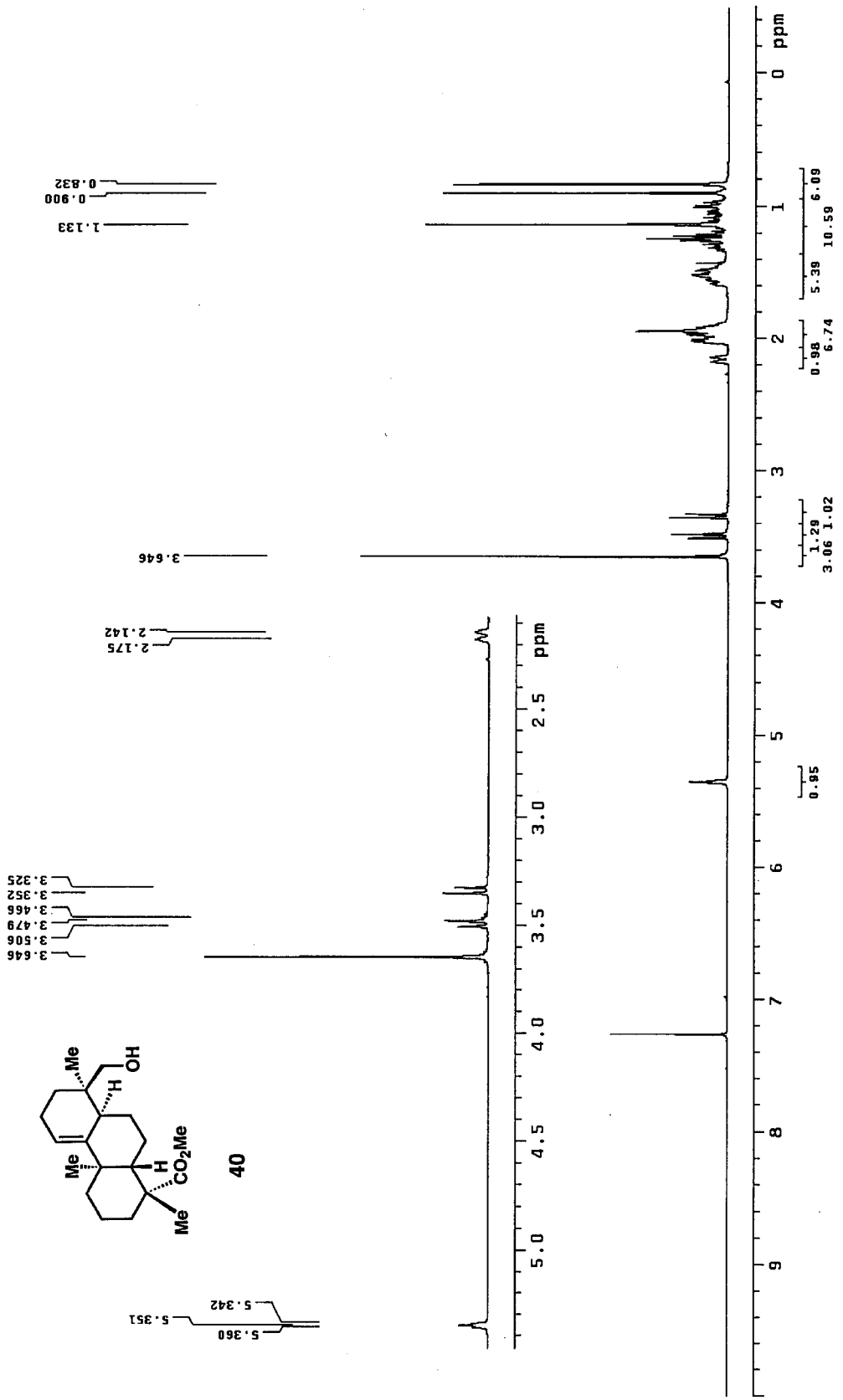

0.869
1.031
1.131
3.655
5.409
5.418
5.427
9.635
0.82
1.00
2.86
7.40
8.06
6.12
3.36
4.27
0 ppm
Me
H
O
H
Me
H
CO2Me
Me
38

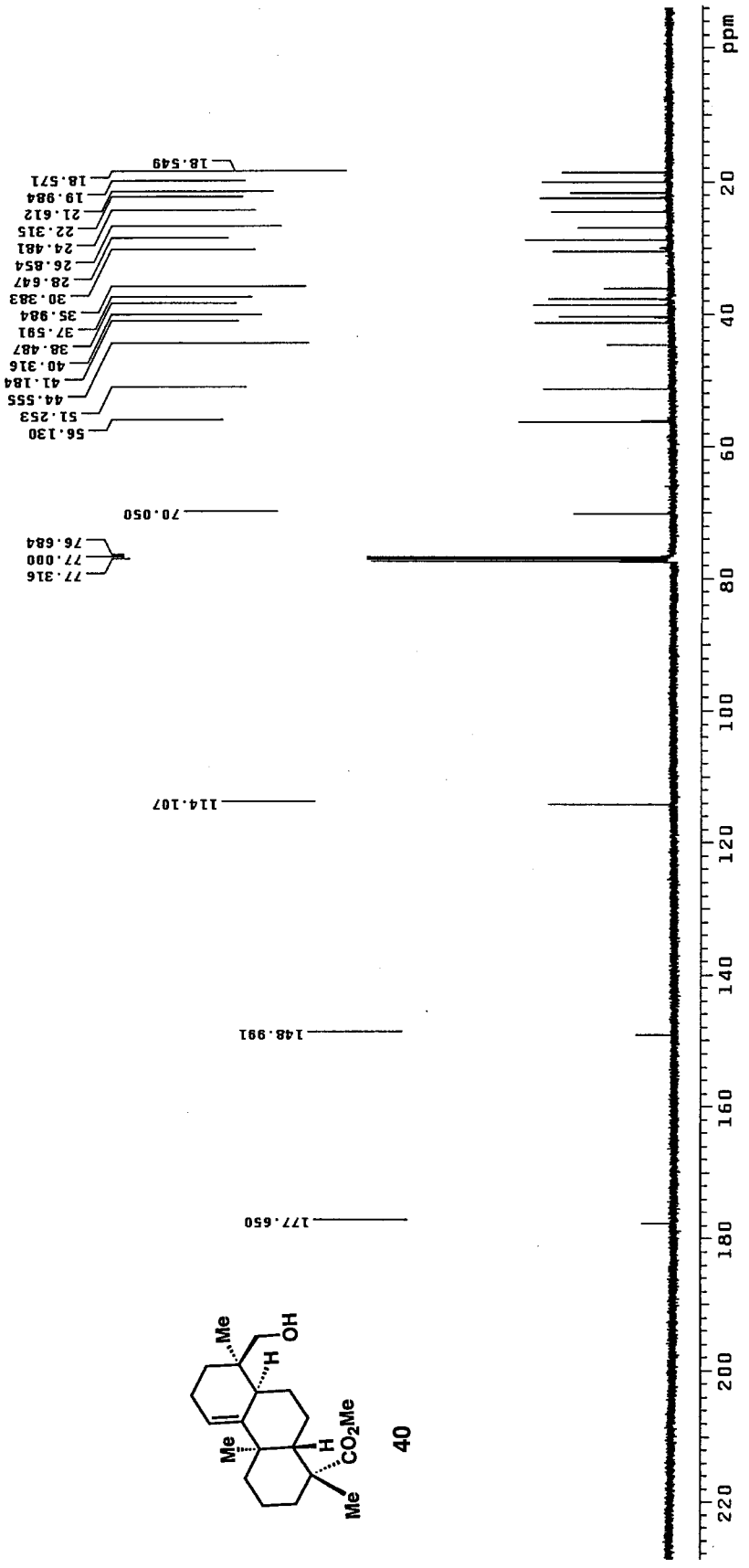

ppm
20
40
60
80
100
120
140
160
180
200
220
207.334
177.463
147.370
114.609
77.316
77.000
76.684
55.764
51.310
47.344
44.519
40.732
40.409
38.358
37.476
31.509
28.647
24.954
24.179
21.906
19.898
19.647
18.729
Me
H
O
H
Me
H
CO2Me
Me
38

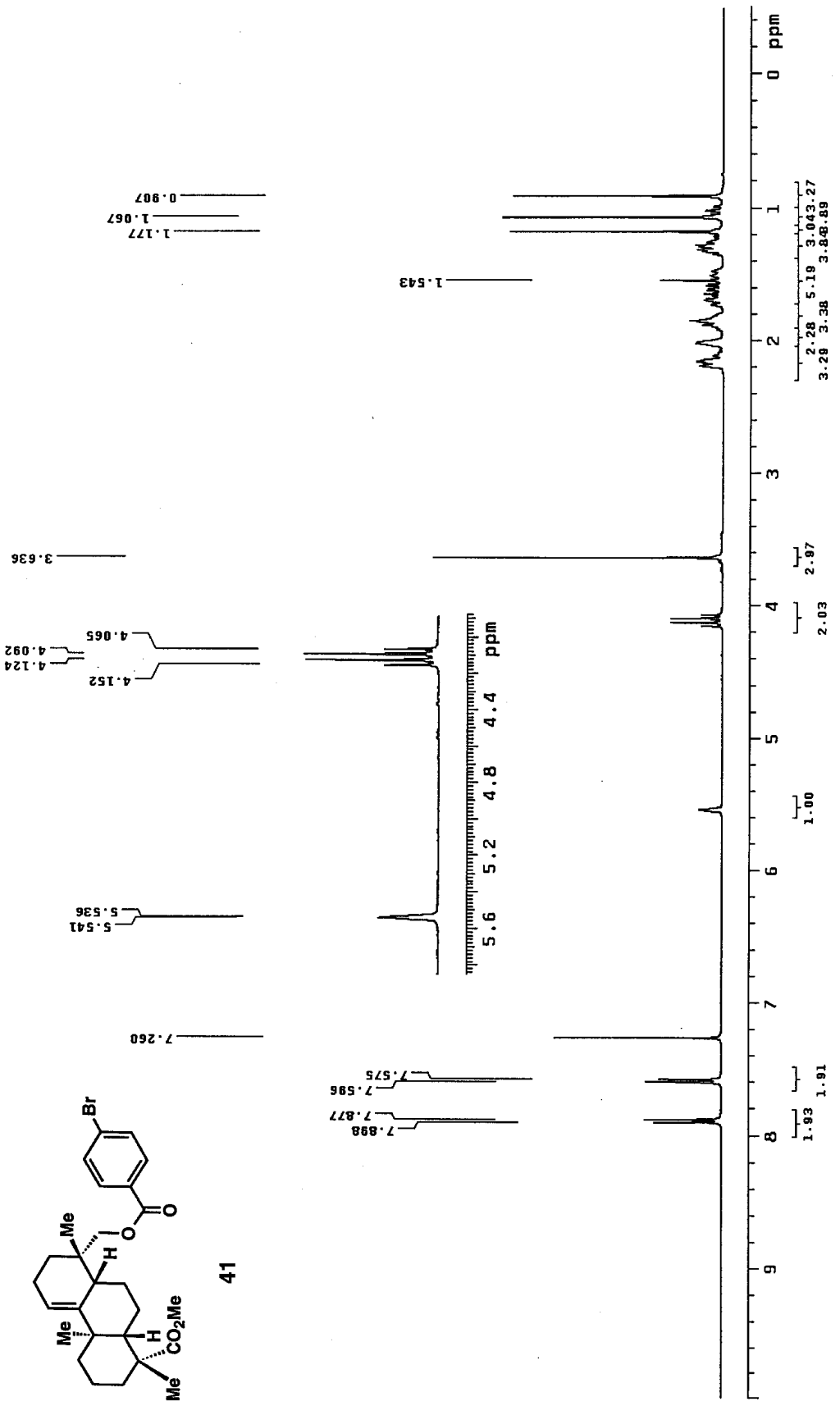

0.884
0.958
1.173
1.482
3.408
3.416
3.626
5.494
5.499
5.504
5.509
5.514
5.518
5.55
5.40
ppm
1.00
2.11
3.27
9.37
2.59
5.61
7.80
4.68
3.25
0
1
2
3
4
5
6
7
8
9
ppm
Me
OH
H
Me
H
CO2Me
Me

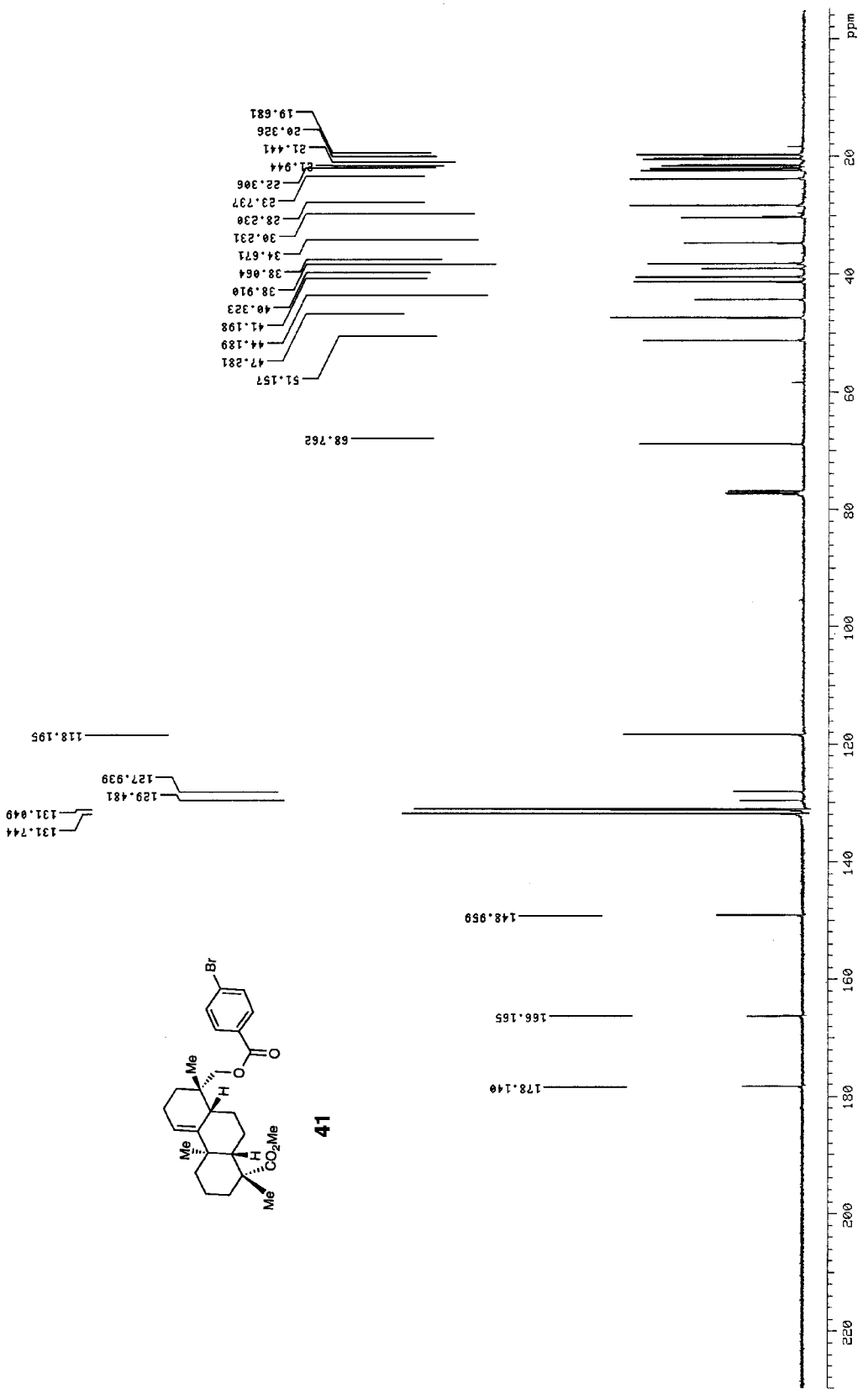
39

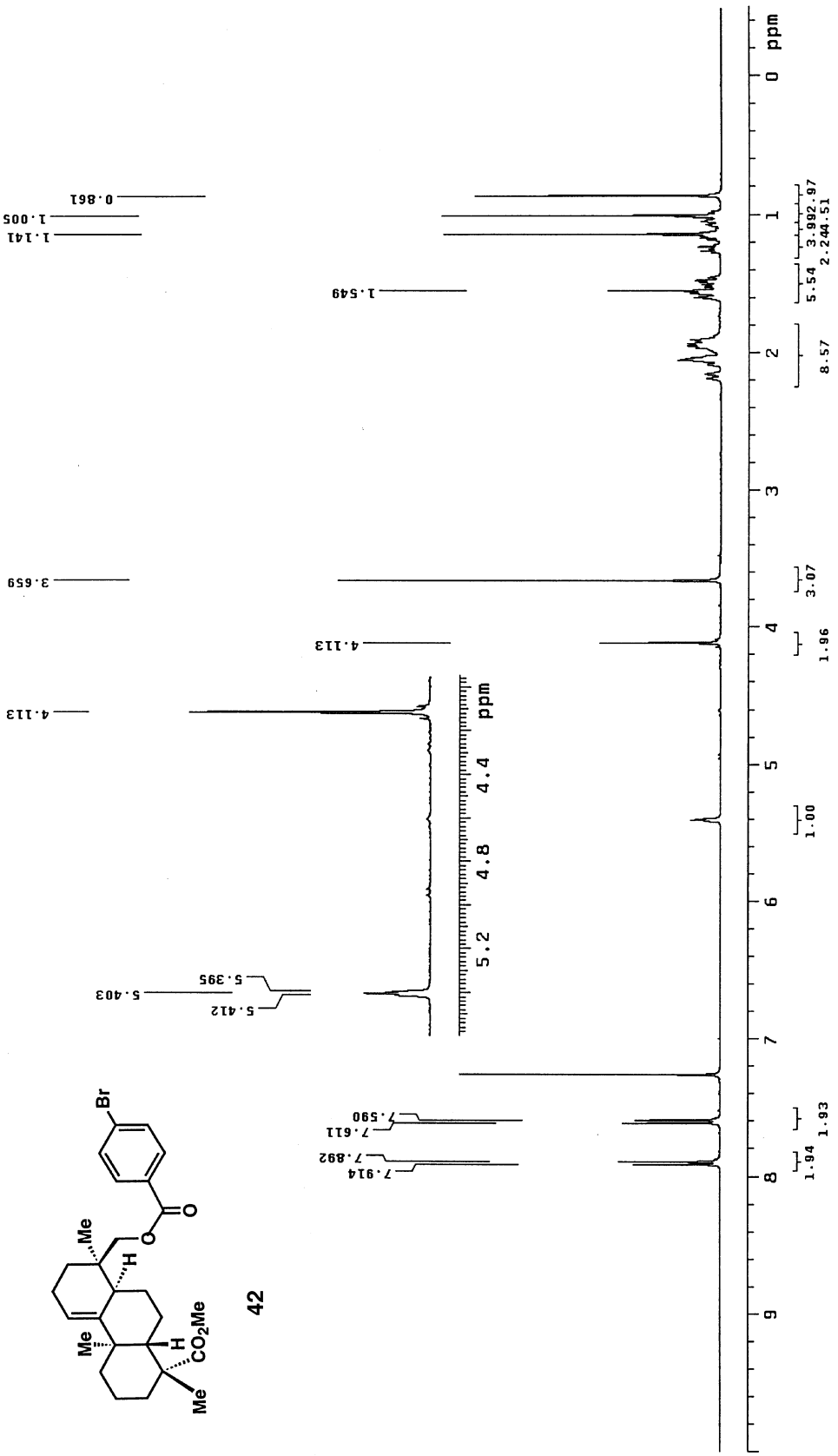

ppm
20
40
60
80
100
120
140
160
180
200
220
19.928
20.541
21.386
22.268
22.552
22.874
28.407
28.840
35.764
38.119
39.219
39.323
40.983
44.198
47.675
51.219
67.295
76.693
77.007
77.322
118.056
148.959
178.052
Me
OH
H
Me
H
CO2Me
Me
39

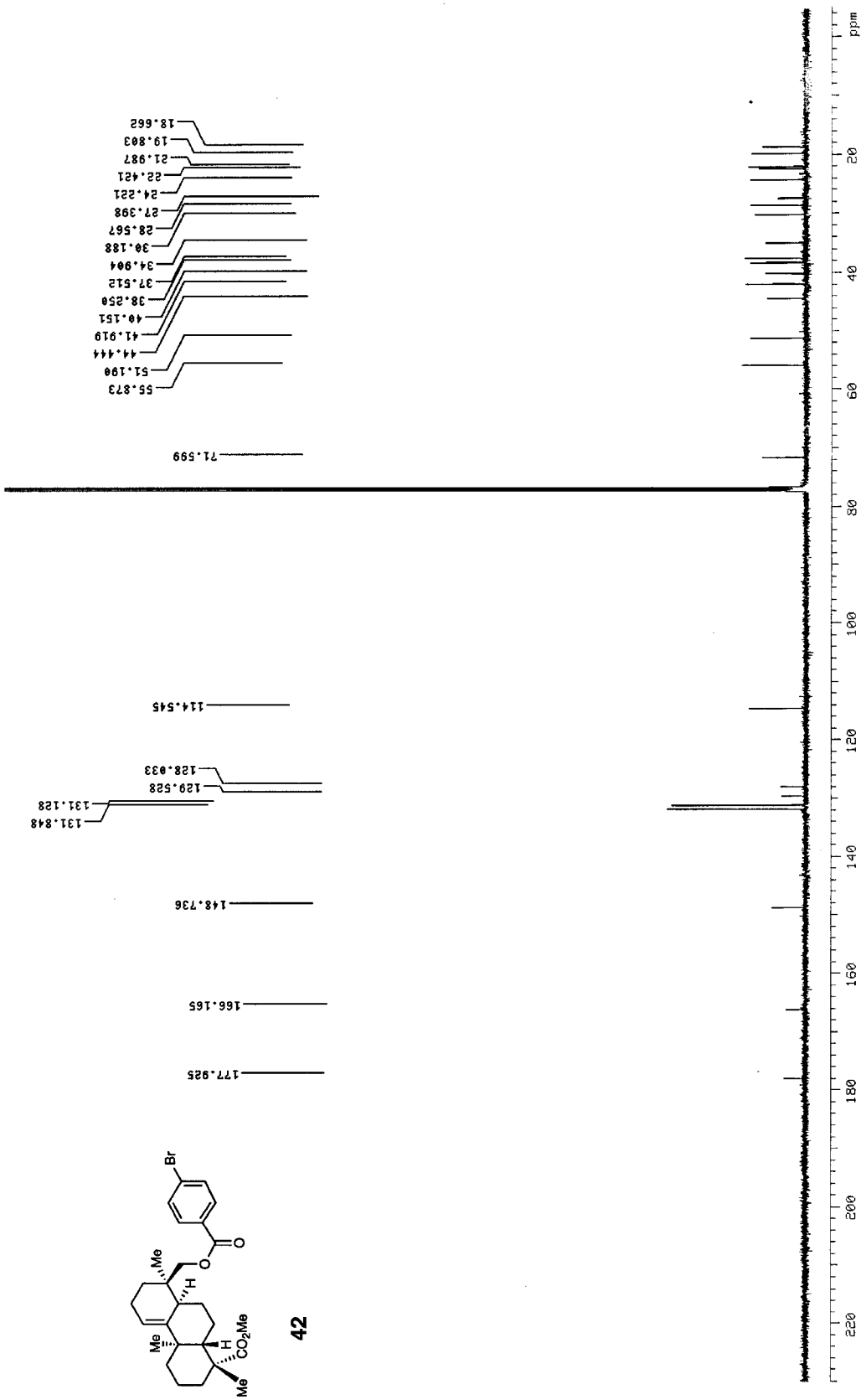

Me
OH
H
Me
H
CO2Me
Me
40
3.646
3.506
3.479
3.466
3.352
3.325
5.360
5.351
5.342
2.175
2.142
3.646
1.133
0.900
0.832
ppm
0.95
1.29
3.06
1.02
0.98
6.74
5.39
10.59
6.09

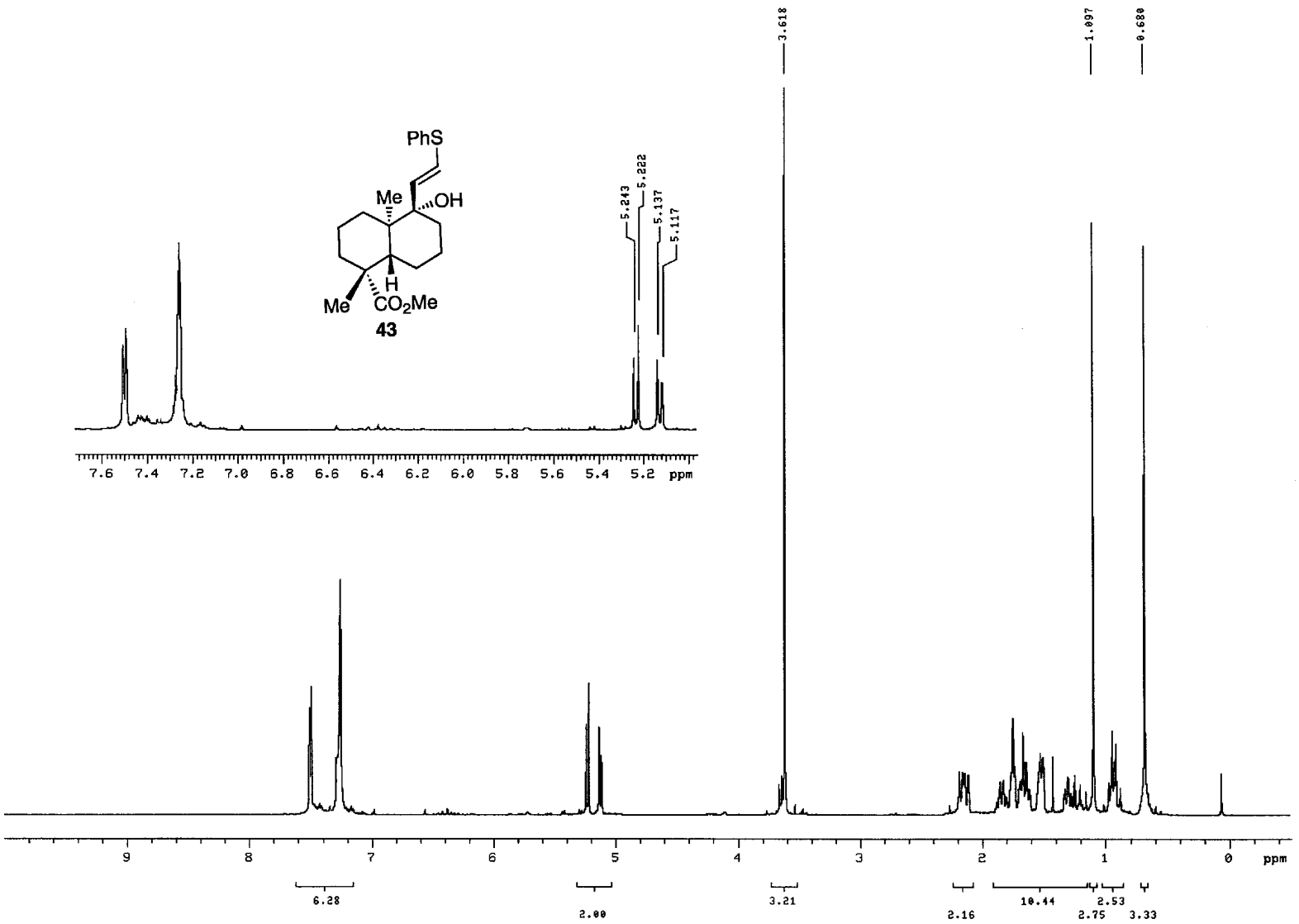

18.549
18.571
19.984
21.612
22.315
24.481
26.854
28.647
30.383
35.984
37.591
38.487
40.316
41.184
44.555
51.253
56.130
70.050
76.684
77.000
77.316
114.107
148.991
177.650
Me
OH
H
Me
H
CO2Me
Me
40
ppm
20
40
60
80
100
120
140
160
180
200
220

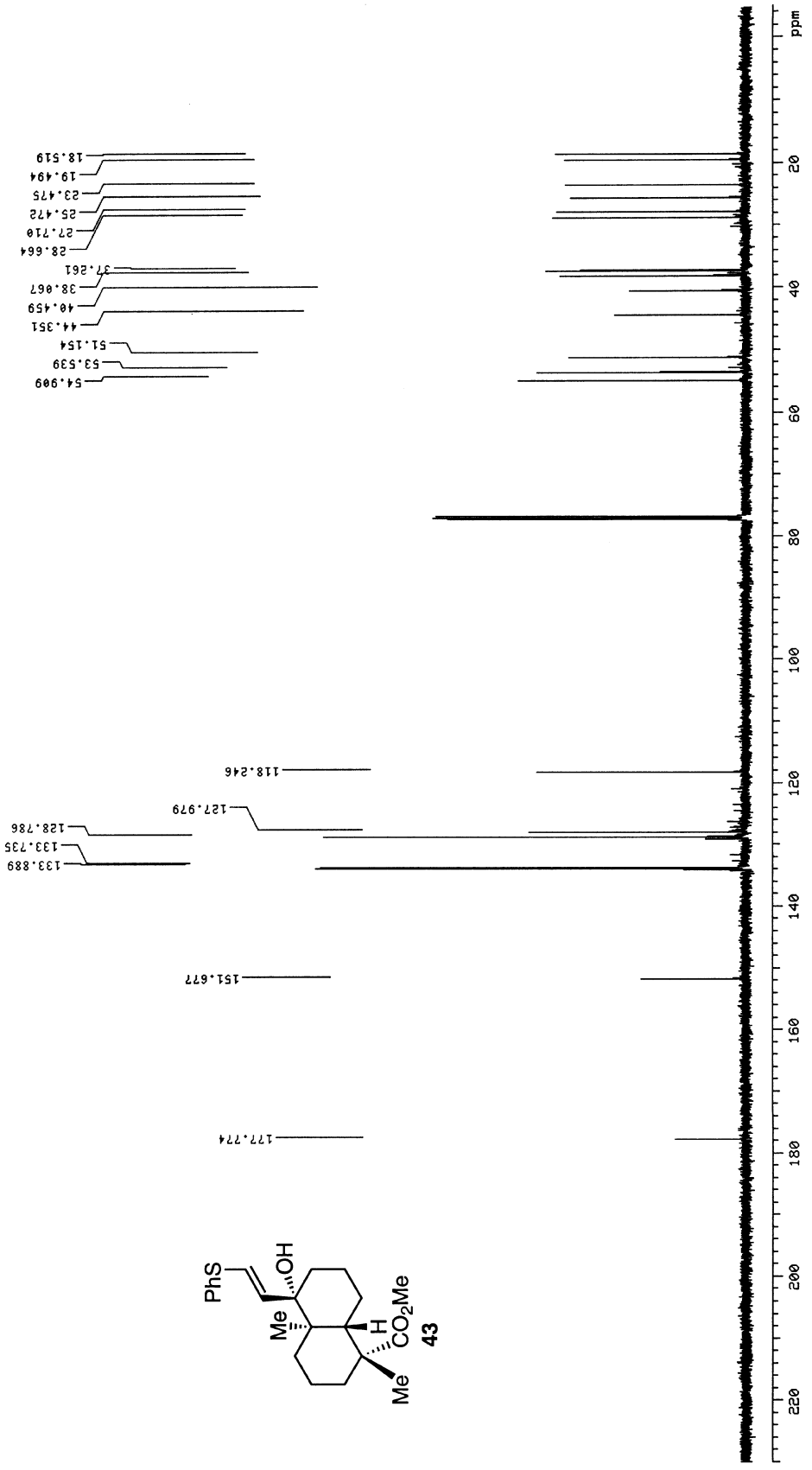

41
Br
O
O
Me
H
Me
H
CO2Me
Me
0.807
1.067
1.177
1.543
3.636
4.065
4.092
4.124
4.152
5.536
5.541
7.260
7.575
7.586
7.877
7.898
ppm
5.6
5.2
4.8
4.4
ppm
0
1
2
3
4
5
6
7
8
9
2.97
2.03
1.00
1.91
1.93
2.28
3.29
3.38
5.19
3.043.27
3.48.89

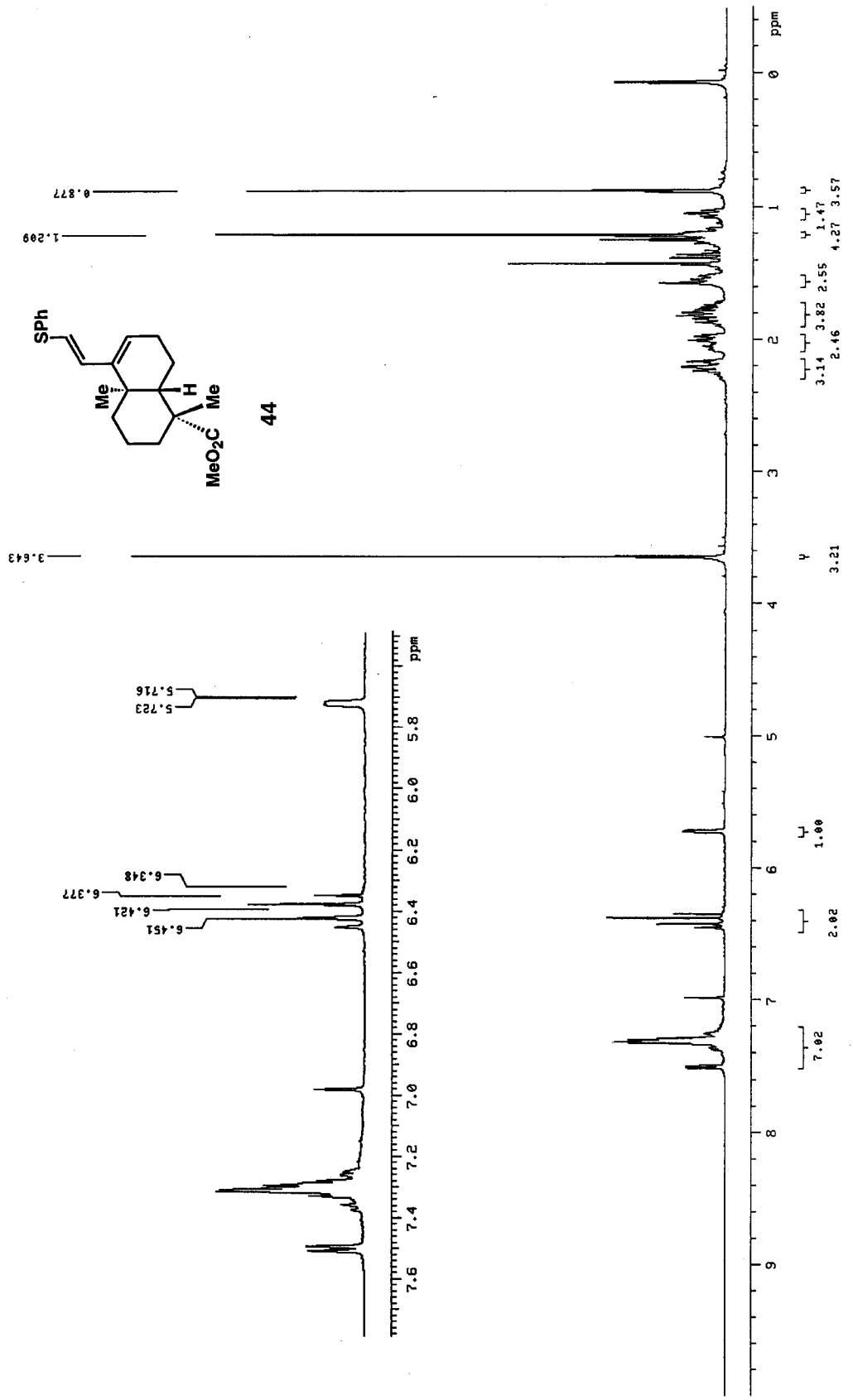

19.681
20.326
21.441
21.944
22.306
23.737
28.230
30.231
34.671
38.064
38.910
40.323
41.198
44.189
47.281
51.157
68.762
118.195
127.939
129.481
131.049
131.744
148.959
166.165
178.140
ppm
20
40
60
80
100
120
140
160
180
200
220
Br
O
O
Me
H
Me
H
CO2Me
Me
41

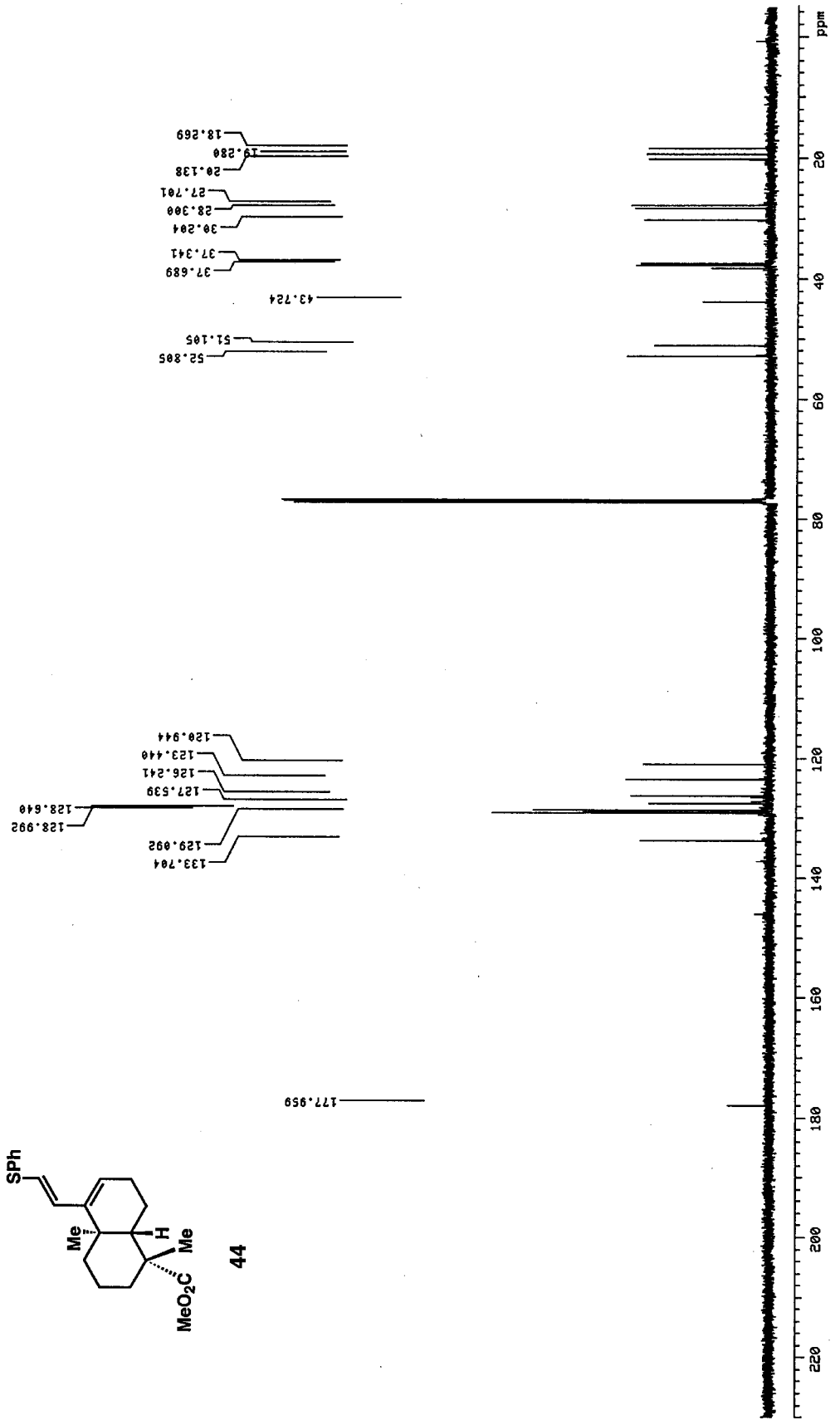

Br
O
O
Me
H
Me
H
CO2Me
Me
42
7.914
7.892
7.611
7.590
5.412
5.403
5.395
4.113
4.113
3.659
1.549
0.861
1.005
1.141
5.2
4.8
4.4
ppm
0
1
2
3
4
5
6
7
8
9
ppm
1.94
1.93
1.00
1.96
3.07
8.57
5.54
3.992.97
2.244.51

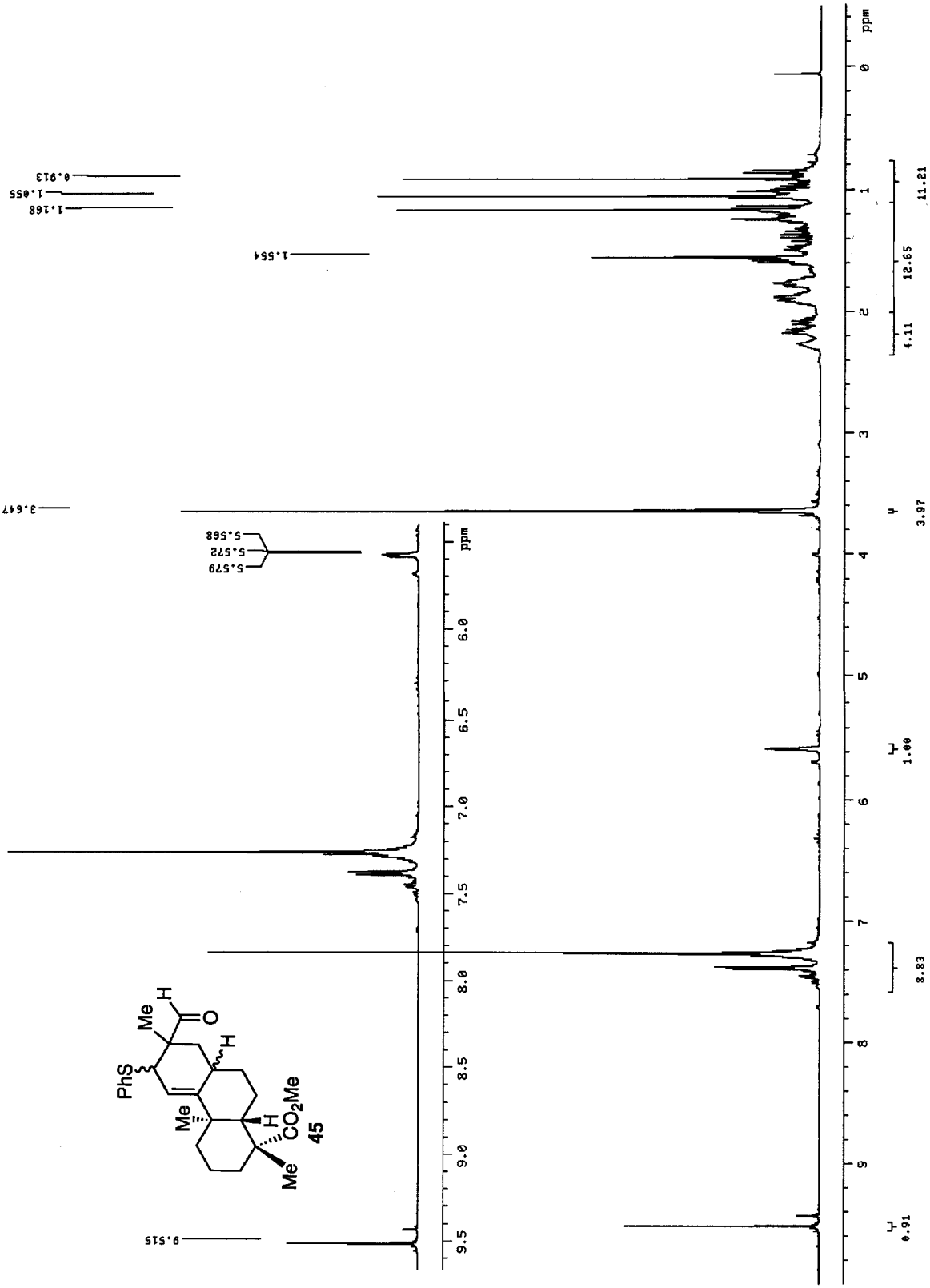

18.662
19.803
21.987
22.421
24.221
27.398
28.567
30.188
34.904
37.512
38.250
40.151
41.919
44.444
51.190
55.873
71.599
114.545
128.033
129.528
131.128
131.848
148.736
166.165
177.925
ppm
20
40
60
80
100
120
140
160
180
200
220
Br
O
O
Me
H
Me
H
CO2Me
Me
42

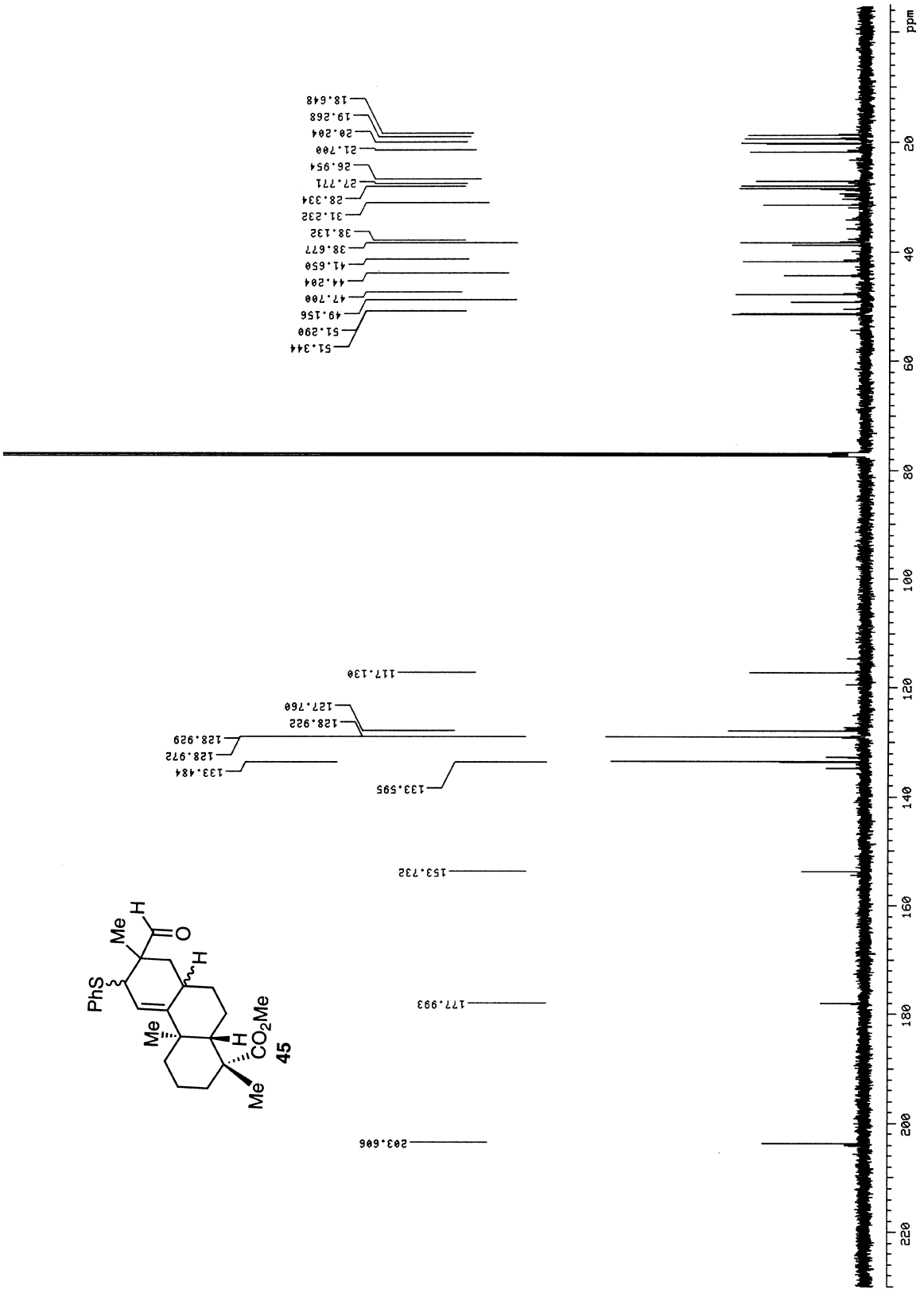

PhS
Me
OH
H
Me
CO2Me
43
5.243
5.222
5.137
5.117
3.618
1.097
0.680
7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 ppm
9 8 7 6 5 4 3 2 1 0 ppm
6.28
2.00
3.21
2.16
10.44
2.53
2.75
3.33

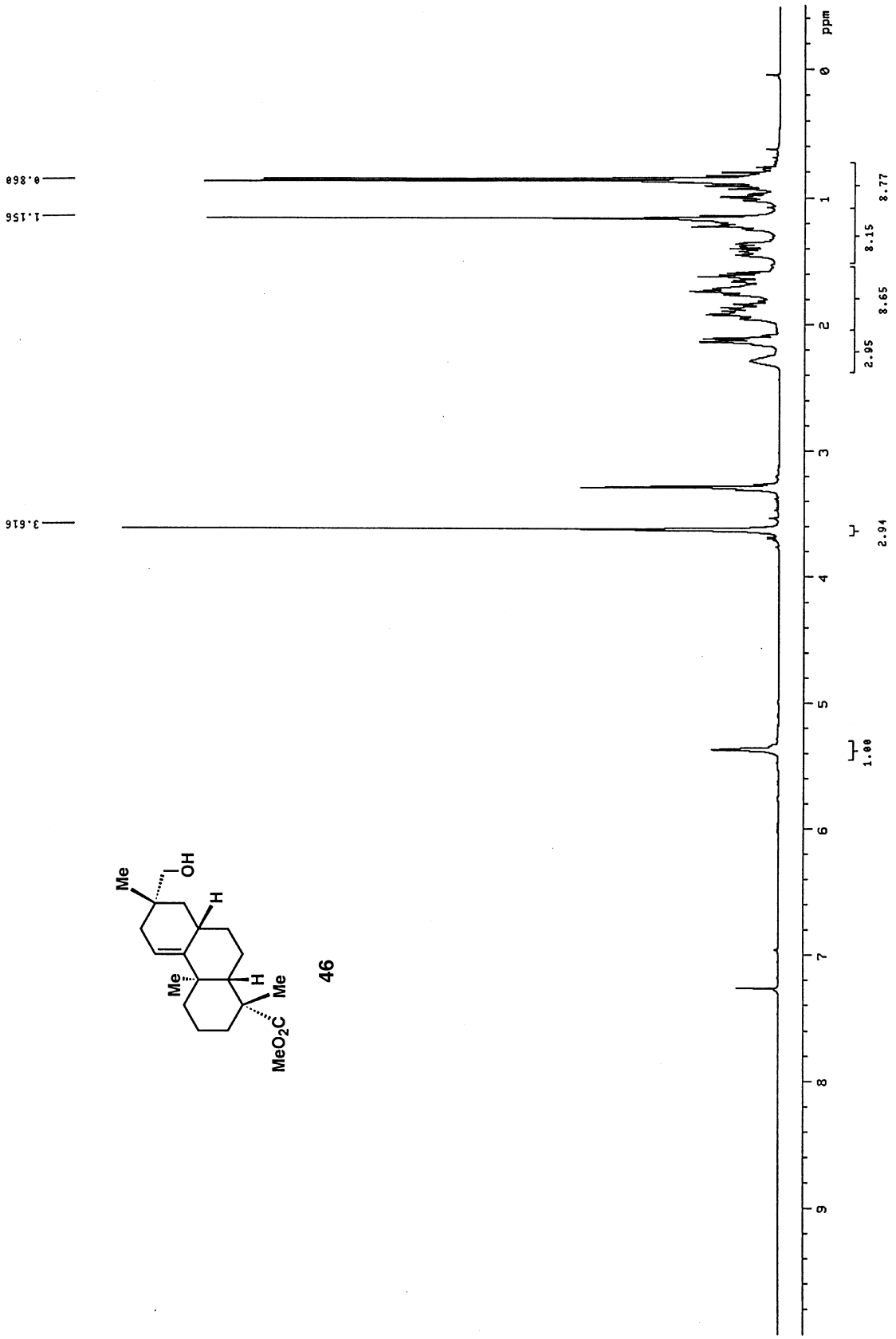

177.774
151.677
133.889
133.735
128.786
127.979
118.246
54.909
53.539
51.154
44.351
40.459
38.067
37.261
28.664
27.710
25.472
23.475
19.494
18.519
ppm
20
40
60
80
100
120
140
160
180
200
220
PhS
OH
Me
H
CO2Me
Me
43


SPh
Me
H
Me
MeO2C
44
0.877
1.209
3.643
5.723
5.716
6.348
6.377
6.421
6.451
ppm

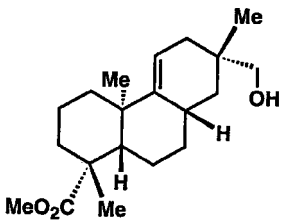

ppm
20
40
60
80
100
120
140
160
180
200
220
18.269
19.280
20.138
27.701
28.300
30.204
37.341
37.689
43.724
51.105
52.805
120.944
123.440
126.241
127.539
128.640
128.992
129.092
133.704
177.959
SPh
Me
H
Me
MeO2C
44


PhS
Me
H
O
H
Me
H
CO2Me
Me
45
9.515
5.579
5.572
5.568
3.647
1.554
1.168
1.055
0.913
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
0
1
2
3
4
5
6
7
8
9
0.91
1.00
8.83
4.11
12.65
11.21
3.97


203.606
177.993
153.732
133.595
133.484
128.972
128.929
128.922
127.760
117.130
51.344
51.290
49.156
47.700
44.284
41.650
38.677
38.132
31.232
28.334
27.771
26.954
21.700
20.204
19.268
18.648
ppm
20
40
60
80
100
120
140
160
180
200
220
PhS
Me
H
O
H
Me
H
CO2Me
Me
45

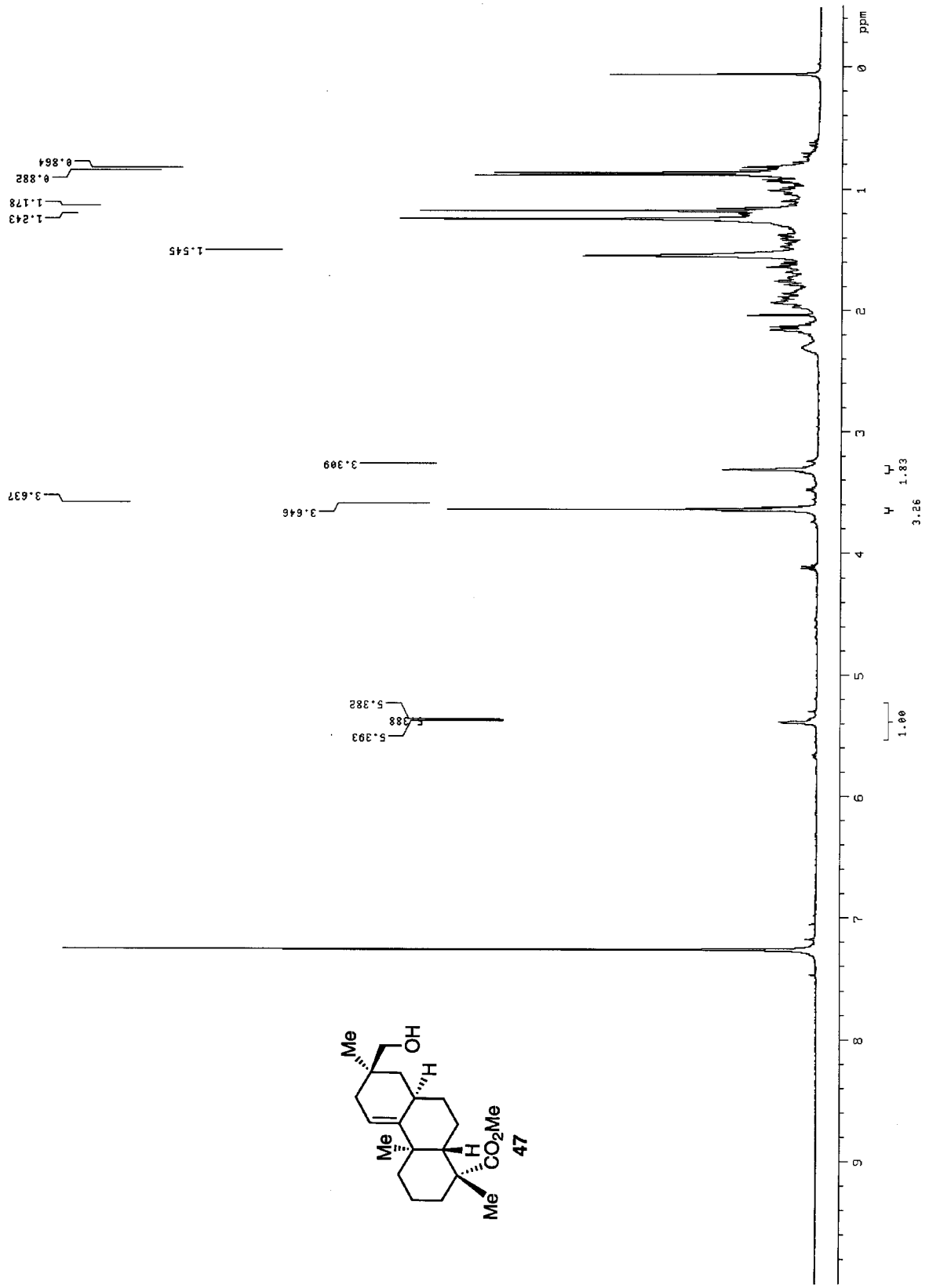

0.860
1.156
3.616
ppm
0
1
2
3
4
5
6
7
8
9
1.00
2.94
2.95
8.65
8.15
8.77
Me
OH
H
Me
H
Me
MeO2C

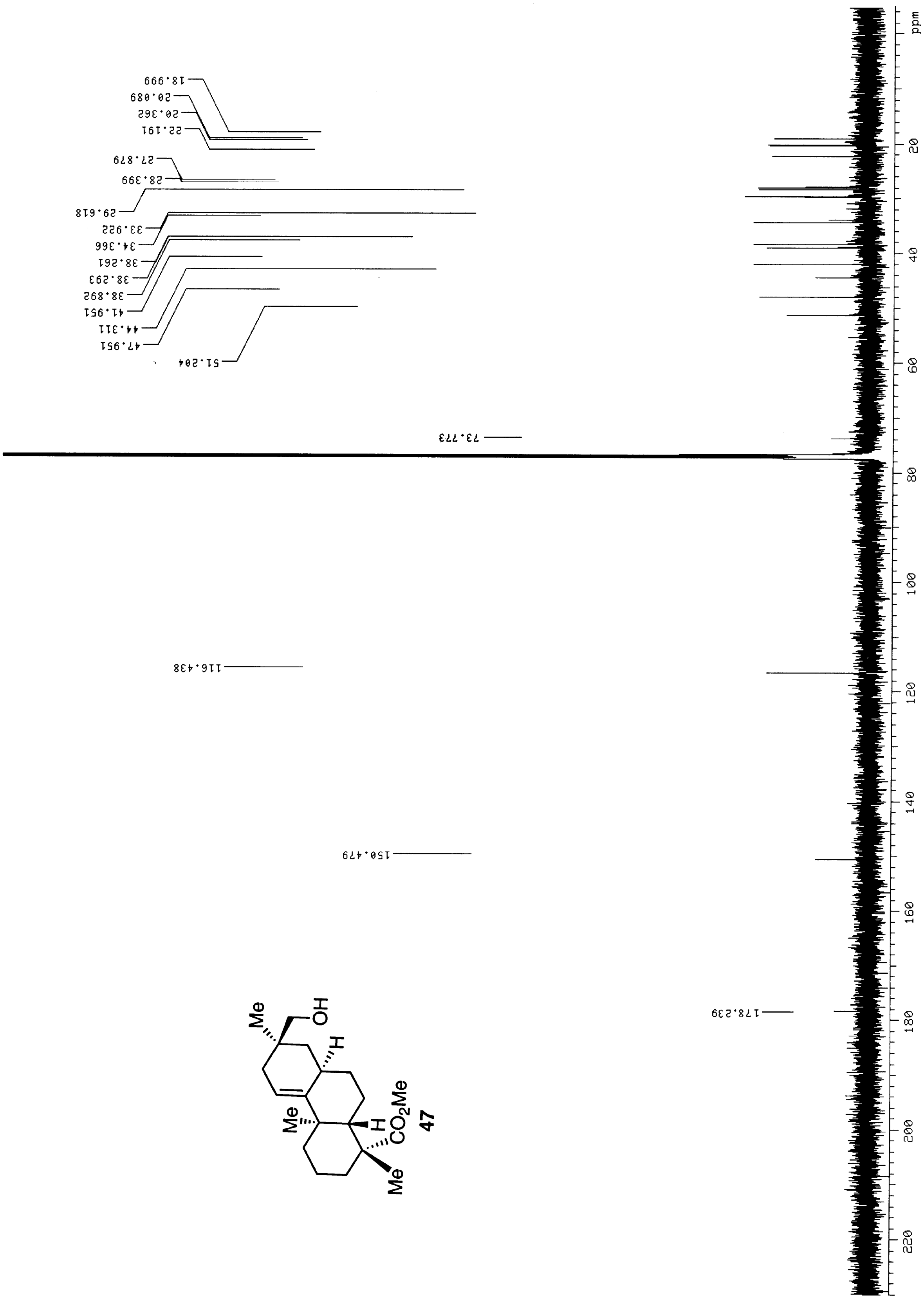
46

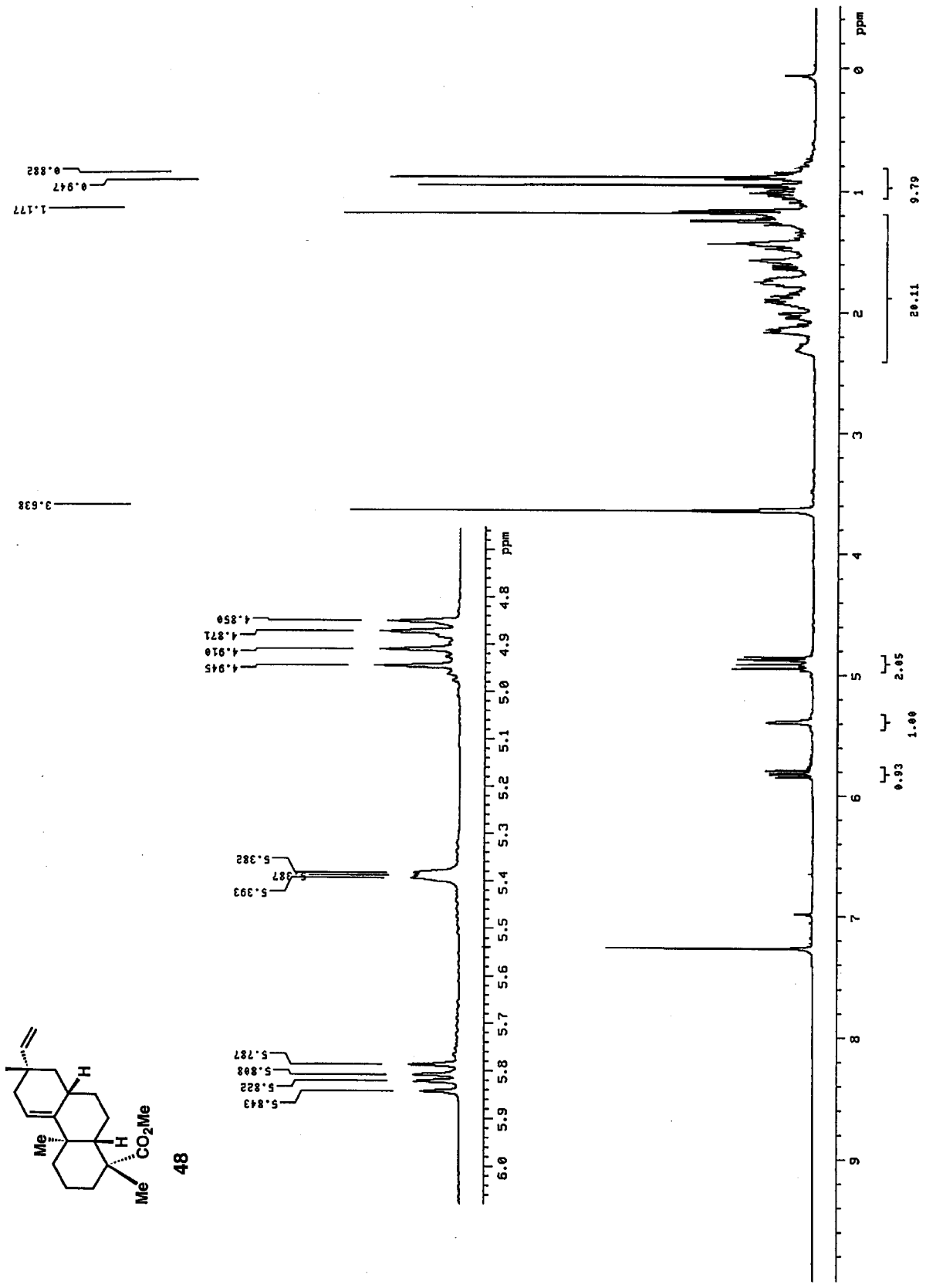

Me
Me
OH
H
H
MeO2C
Me

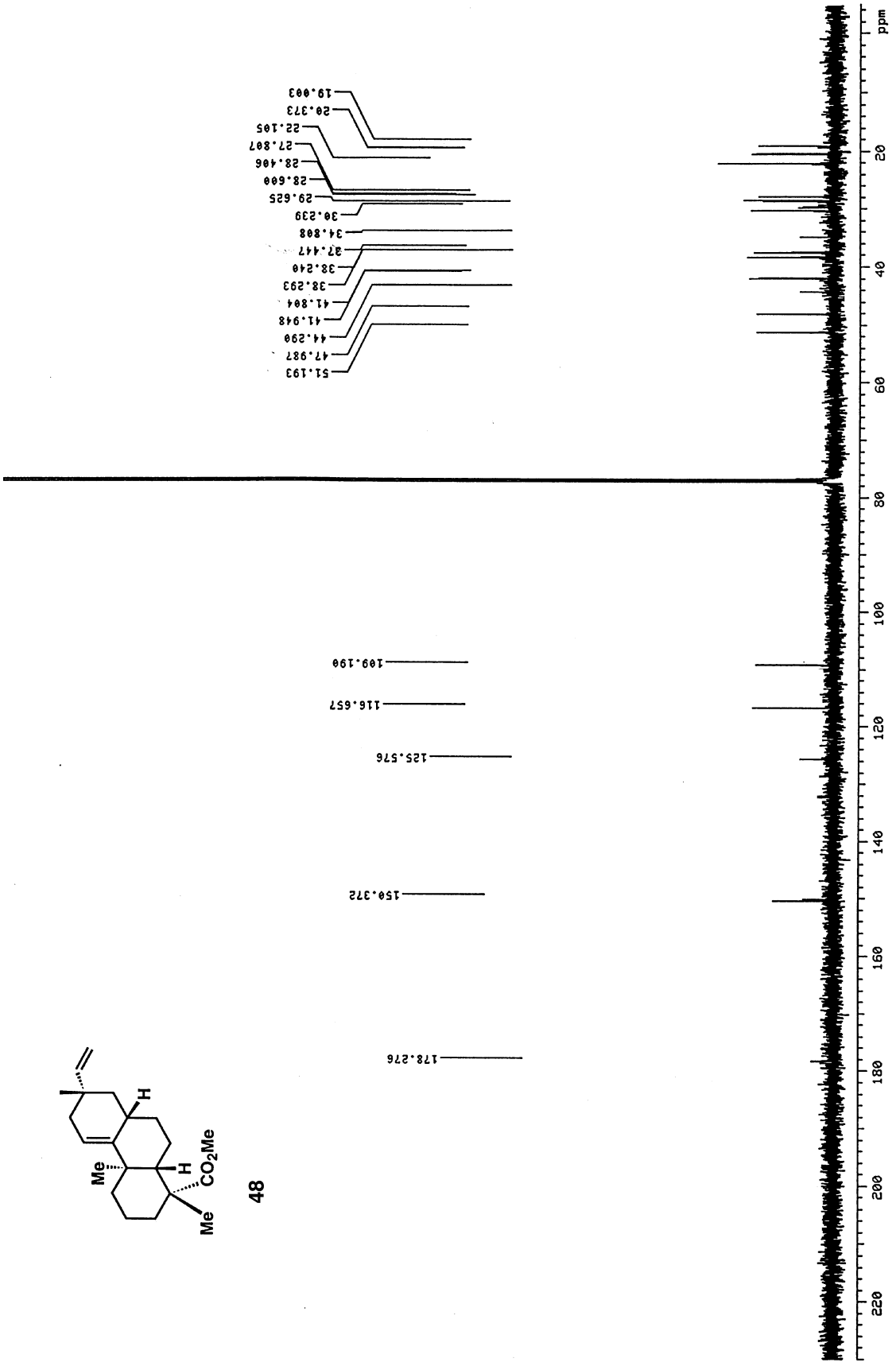
46

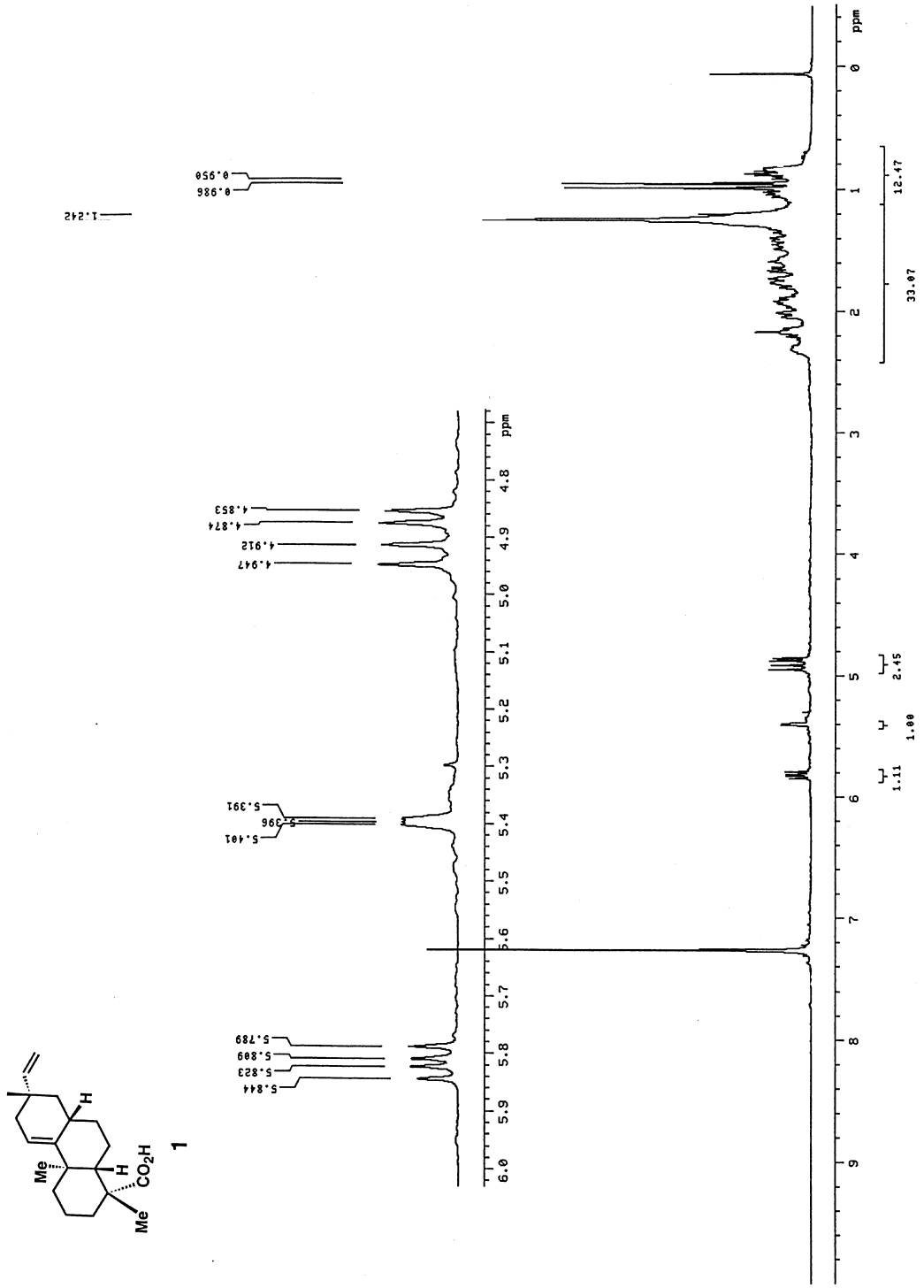
178.244
159.382
116.427
73.604
51.161
47.897
44.257
41.894
38.856
38.232
38.200
34.327
33.875
28.341
28.223
27.832
22.137
20.308
20.061
18.949
220
200
180
160
140
120
100
80
60
40
20
ppm

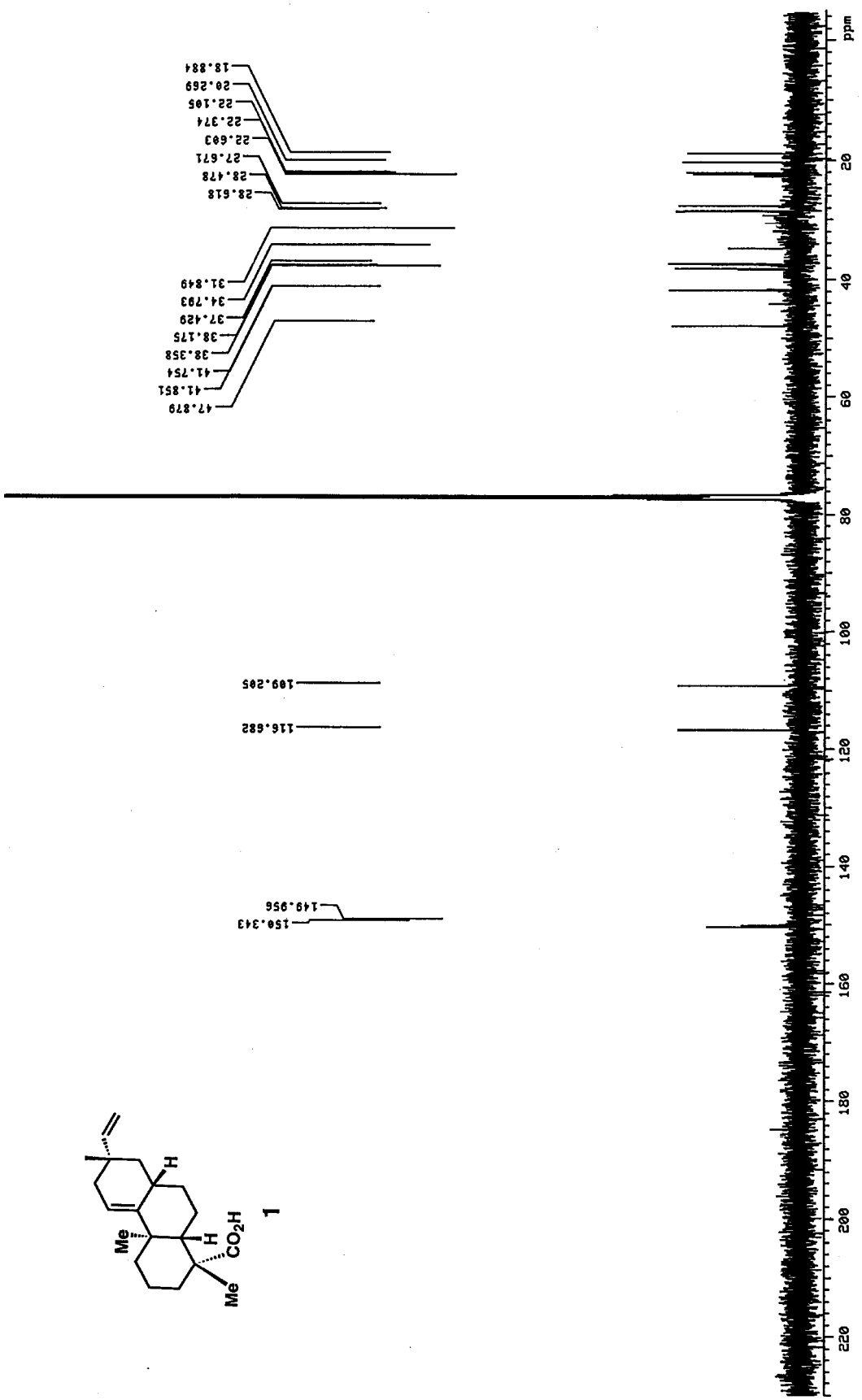

0.864
0.882
1.178
1.243
1.545
3.309
3.637
3.646
5.382
5.388
5.393
ppm
1.83
3.26
1.00
Me
OH
H
Me
H
CO2Me
Me
47


18.999
20.089
20.362
22.191
27.879
28.399
29.618
33.922
34.366
38.261
38.293
38.892
41.951
44.311
47.951
51.204
73.773
116.438
150.479
178.239
ppm
20
40
60
80
100
120
140
160
180
200
220
Me
OH
H
Me
H
CO2Me
Me
47


48
Me
H
H
CO2Me
Me
0.882
0.947
1.177
3.638
4.845
4.871
4.918
4.858
5.382
5.387
5.393
5.787
5.808
5.822
5.843
ppm
9.79
20.11
2.05
1.00
0.93


19.003
20.373
22.105
27.807
28.406
28.600
29.625
30.239
34.808
37.447
38.240
38.293
41.804
41.948
44.290
47.987
51.193
109.190
116.657
125.576
150.372
178.276
ppm
20
40
60
80
100
120
140
160
180
200
220
Me
H
H
CO2Me
Me
48


Me
H
H
CO2H
Me
1
5.844
5.823
5.809
5.789
5.401
5.396
5.391
4.947
4.912
4.874
4.853
0.986
0.958
1.242
ppm
1.11
1.00
2.45
33.07
12.47


18.884
20.269
22.105
22.374
22.603
27.671
28.478
28.618
31.849
34.793
37.429
38.175
38.358
41.754
41.851
47.879
109.205
116.682
149.956
150.343
ppm
20
40
60
80
100
120
140
160
180
200
220
Me
H
H
CO2H
Me
1

X-Ray data of compound **23**

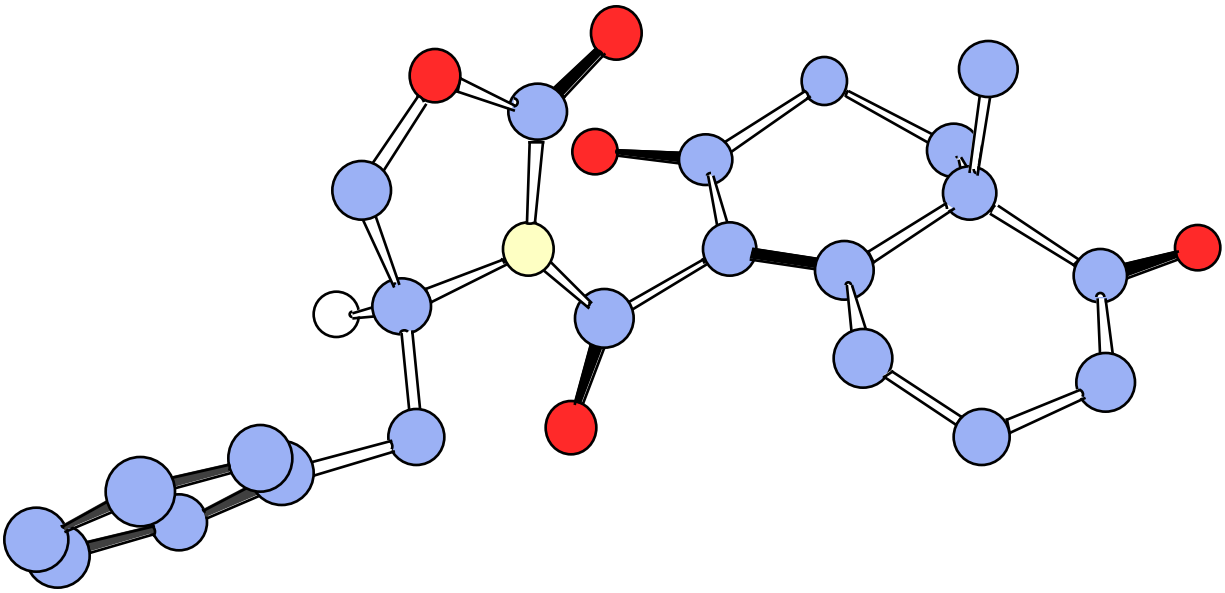


Table 1. Crystal data and structure refinement for **23**

Empirical formula	C22 H23 N O5
Formula weight	381.41
Temperature	23 C
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)
Unit cell dimensions	a = 8.841(7)A alpha = 90
	b = 8.758(7)A beta = 95.26(7)
	c = 12.444(11)A gamma = 90
Crystal size	0.80 x 0.45 x 0.20 mm
Density (calculated)	1.320 Mg/m^3
Absorption coefficient	0.094 mm^-1
Volume, Z, F(000)	959.6(13) A^3, 2, 404
Theta range for data collection	1.64 to 25.00 degrees
Limiting indices	0 < h < 10, -10 < k <0, -14 < l < 14
Reflections collected	1940, 1554 observed [I>2sigma(I)]
Independent reflections	1817 (Rint = 0.0204)
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1817 / 1 / 255
Goodness-of-fit on F^2	1.032
Final R indices [I>2sigma(I)]	R1 = 0.0453, wR2 = 0.1129
R indices (all data)	R1 = 0.0552, wR2 = 0.1219
Absolute structure parameter	0.64(207)
Largest diff. peak and hole	0.179 and -0.212 e/A^3

Scan speed, range, type 5 degrees/minute, 0.6 degrees, Wyckoff
Background range, % time 0.6 degrees, 25% each side

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [A^2 x 10^3] for **23** U(eq) is defined as one third of the trace of the orthogonalized U(ij) tensor.

	x	y	z	U(eq)
O(1)	5418(3)	364(4)	1191(3)	80(1)
O(2)	802(3)	5419(4)	-351(2)	71(1)
O(3)	7359(3)	3449(4)	2059(2)	70(1)
O(4)	3905(3)	1168(6)	3400(2)	100(1)
O(5)	5568(3)	1081(4)	4835(2)	75(1)
N(1)	6219(3)	2450(4)	3436(2)	48(1)
C(1)	4465(4)	1370(5)	1121(3)	50(1)
C(2)	2950(4)	1180(5)	489(3)	55(1)
C(3)	2313(4)	2685(4)	62(3)	47(1)
C(4)	2246(3)	3904(4)	943(2)	40(1)
C(5)	1723(4)	5426(5)	429(3)	51(1)
C(6)	2255(7)	6891(6)	932(5)	95(2)
C(7)	3647(9)	6843(6)	1638(5)	119(3)
C(8)	4091(5)	5478(6)	2214(3)	69(1)
C(9)	3785(4)	4026(5)	1608(2)	42(1)
C(10)	4736(3)	2848(4)	1682(2)	41(1)
C(11)	6216(3)	2933(5)	2369(3)	48(1)
C(12)	1043(4)	3473(6)	1729(3)	64(1)
C(13)	5101(4)	1536(6)	3827(3)	64(1)
C(14)	6984(4)	1777(6)	5183(3)	64(1)
C(15)	7612(4)	2464(5)	4186(3)	48(1)
C(16)	8313(4)	4026(5)	4416(3)	56(1)
C(17)	9627(4)	3870(4)	5281(3)	49(1)
C(18)	10989(4)	3232(5)	5046(3)	60(1)
C(19)	12171(5)	3061(6)	5838(4)	71(1)
C(20)	12019(5)	3541(6)	6868(4)	73(1)
C(21)	10673(5)	4171(6)	7118(3)	71(1)
C(22)	9496(5)	4336(5)	6333(3)	60(1)

Table 3. Bond lengths [A] and angles [degrees] for **23**

O(1)-C(1)	1.216(5)
O(2)-C(5)	1.208(4)
O(3)-C(11)	1.202(4)
O(4)-C(13)	1.184(5)
O(5)-C(13)	1.344(5)
O(5)-C(14)	1.424(5)
N(1)-C(13)	1.394(5)
N(1)-C(11)	1.394(4)
N(1)-C(15)	1.476(4)
C(1)-C(10)	1.480(5)
C(1)-C(2)	1.499(5)
C(2)-C(3)	1.511(5)
C(3)-C(4)	1.536(5)
C(4)-C(9)	1.530(4)
C(4)-C(5)	1.531(6)
C(4)-C(12)	1.556(5)
C(5)-C(6)	1.486(7)
C(6)-C(7)	1.445(7)
C(7)-C(8)	1.430(8)
C(8)-C(9)	1.490(6)
C(9)-C(10)	1.329(5)

C(10)-C(11)	1.497(4)
C(14)-C(15)	1.528(5)
C(15)-C(16)	1.518(6)
C(16)-C(17)	1.515(5)
C(17)-C(18)	1.383(5)
C(17)-C(22)	1.387(5)
C(18)-C(19)	1.377(5)
C(19)-C(20)	1.369(7)
C(20)-C(21)	1.372(7)
C(21)-C(22)	1.368(6)
C(13)-O(5)-C(14)	110.1(3)
C(13)-N(1)-C(11)	124.6(3)
C(13)-N(1)-C(15)	111.2(3)
C(11)-N(1)-C(15)	121.9(3)
O(1)-C(1)-C(10)	121.2(3)
O(1)-C(1)-C(2)	122.5(4)
C(10)-C(1)-C(2)	116.3(3)
C(1)-C(2)-C(3)	112.0(3)
C(2)-C(3)-C(4)	113.1(3)
C(9)-C(4)-C(5)	112.7(3)
C(9)-C(4)-C(3)	110.1(3)
C(5)-C(4)-C(3)	109.7(3)
C(9)-C(4)-C(12)	107.6(3)
C(5)-C(4)-C(12)	106.0(3)
C(3)-C(4)-C(12)	110.7(3)
O(2)-C(5)-C(6)	120.4(4)
O(2)-C(5)-C(4)	119.1(4)
C(6)-C(5)-C(4)	120.3(3)
C(7)-C(6)-C(5)	116.7(4)
C(8)-C(7)-C(6)	120.5(5)
C(7)-C(8)-C(9)	115.6(3)
C(10)-C(9)-C(8)	122.9(3)
C(10)-C(9)-C(4)	120.6(3)
C(8)-C(9)-C(4)	116.3(3)
C(9)-C(10)-C(1)	124.9(3)
C(9)-C(10)-C(11)	121.0(3)
C(1)-C(10)-C(11)	114.1(3)
O(3)-C(11)-N(1)	119.5(3)
O(3)-C(11)-C(10)	123.5(3)
N(1)-C(11)-C(10)	117.0(3)
O(4)-C(13)-O(5)	121.6(4)
O(4)-C(13)-N(1)	129.2(4)
O(5)-C(13)-N(1)	109.2(3)
O(5)-C(14)-C(15)	107.2(3)
N(1)-C(15)-C(16)	115.4(3)
N(1)-C(15)-C(14)	99.5(3)
C(16)-C(15)-C(14)	112.0(3)
C(17)-C(16)-C(15)	109.0(3)
C(18)-C(17)-C(22)	117.9(3)
C(18)-C(17)-C(16)	120.8(3)
C(22)-C(17)-C(16)	121.3(3)
C(19)-C(18)-C(17)	120.7(4)
C(20)-C(19)-C(18)	120.3(4)
C(19)-C(20)-C(21)	119.8(4)
C(22)-C(21)-C(20)	119.9(4)
C(21)-C(22)-C(17)	121.3(4)

Table 4. Anisotropic displacement parameters [A^2 x 10^3] for **23**. The anisotropic displacement factor exponent takes the form: -2(pi)^2 [(ha*)^2U11 + ... + 2hka*b*U12]

	U11	U22	U33	U23	U13	U12
O(1)	72(2)	63(2)	100(2)	-3(2)	-13(2)	26(2)
O(2)	72(2)	75(2)	61(2)	-2(2)	-18(1)	23(2)
O(3)	38(1)	123(3)	50(1)	18(2)	2(1)	-14(2)
O(4)	52(2)	167(4)	77(2)	47(2)	-10(1)	-39(2)
O(5)	58(2)	111(3)	54(2)	31(2)	0(1)	-12(2)
N(1)	33(1)	72(2)	39(1)	10(2)	-2(1)	-2(1)
C(1)	48(2)	49(2)	53(2)	8(2)	-1(2)	4(2)
C(2)	54(2)	47(2)	60(2)	-5(2)	-7(2)	-5(2)
C(3)	42(2)	50(2)	46(2)	-4(2)	-9(1)	-2(2)
C(4)	31(2)	50(2)	39(2)	-1(2)	-1(1)	-1(2)
C(5)	45(2)	54(2)	55(2)	-6(2)	3(2)	7(2)
C(6)	95(4)	56(3)	127(5)	-18(3)	-30(3)	16(3)
C(7)	173(7)	56(3)	115(5)	-7(3)	-62(4)	-25(4)
C(8)	57(2)	79(3)	69(3)	-36(3)	-10(2)	6(2)
C(9)	34(2)	57(2)	34(2)	-3(2)	3(1)	-2(2)
C(10)	33(2)	56(2)	33(2)	1(2)	0(1)	-3(2)
C(11)	34(2)	69(3)	40(2)	7(2)	2(1)	-1(2)
C(12)	46(2)	89(3)	59(2)	-2(2)	14(2)	-11(2)
C(13)	41(2)	97(3)	55(2)	19(2)	4(2)	-3(2)
C(14)	54(2)	88(3)	49(2)	17(2)	-4(2)	-2(2)
C(15)	35(2)	68(3)	40(2)	5(2)	-3(1)	5(2)
C(16)	52(2)	61(2)	52(2)	7(2)	-7(2)	0(2)
C(17)	49(2)	50(2)	47(2)	1(2)	-7(1)	3(2)
C(18)	55(2)	70(3)	55(2)	-13(2)	-2(2)	1(2)
C(19)	48(2)	74(3)	86(3)	-10(3)	-16(2)	8(2)
C(20)	73(3)	72(3)	67(3)	-1(2)	-32(2)	0(2)
C(21)	85(3)	79(3)	47(2)	-6(2)	-10(2)	-6(3)
C(22)	59(2)	66(3)	53(2)	-5(2)	1(2)	5(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **23**

	x	y	z	U(eq)
H(2A)	2245(4)	721(5)	947(3)	65
H(2B)	3055(4)	493(5)	-111(3)	65
H(3A)	1297(4)	2519(4)	-282(3)	56
H(3B)	2937(4)	3061(4)	-483(3)	56
H(6A)	2390(7)	7615(6)	359(5)	114
H(6B)	1458(7)	7285(6)	1341(5)	114
H(7A)	4468(9)	7112(6)	1207(5)	143
H(7B)	3587(9)	7647(6)	2168(5)	143
H(8A)	3566(5)	5442(6)	2865(3)	83
H(8B)	5171(5)	5536(6)	2434(3)	83
H(12A)	70(9)	3349(38)	1329(5)	96
H(12B)	985(27)	4270(18)	2253(17)	96
H(12C)	1333(20)	2534(20)	2089(20)	96

H(14A)	7684(4)	1023(6)	5513(3)	77
H(14B)	6841(4)	2568(6)	5710(3)	77
H(15A)	8369(4)	1774(5)	3923(3)	58
H(16A)	8672(4)	4442(5)	3764(3)	67
H(16B)	7557(4)	4717(5)	4659(3)	67
H(18A)	11108(4)	2915(5)	4345(3)	72
H(19A)	13076(5)	2616(6)	5671(4)	85
H(20A)	12825(5)	3442(6)	7399(4)	88
H(21A)	10562(5)	4486(6)	7820(3)	86
H(22A)	8591(5)	4769(5)	6509(3)	72

```
-Experimental
R1=(&GS&GB&GBF&Vo&0&GB-&GBF&Vc&0&GB&GB/&GS&GBF&Vo&0&GB),
& wR2=&GSw(F&Vo&0&^2&0-F&Vc&0&^2&0)&^2&0/&GSw[(F&Vo&0&^2&0)&^2&0]&^&Gh&0,
& S=[&GSw(F&Vo&0&^2&0-F&Vc&0&^2&0)&^2&0/(n-p)]&^&Gh&0
```

References:

H.D.Flack. Acta Crystallographica, A39, 876-881(1983).

SHELXTL-PC.& G.M.Sheldrick, Siemens Analytical X-ray Instruments
Inc. Madison, WI. (1990).

The worst:

h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Res(A)
-2	-2	5	40.49	58.29	3.61	0.072	2.00
4	-2	0	67.13	91.28	3.42	0.091	1.97
-6	-2	3	34.36	49.00	3.24	0.066	1.35
-6	0	3	113.55	87.27	3.21	0.089	1.42
-6	0	12	16.43	8.53	3.14	0.028	0.88
7	0	6	28.92	42.14	3.13	0.062	1.03
-4	0	11	15.41	8.42	3.01	0.028	1.04
-3	0	6	316.69	258.04	2.93	0.152	1.77
-4	0	2	50.09	37.71	2.90	0.058	2.14
-9	-1	7	8.82	16.32	2.90	0.038	0.89
5	0	4	276.71	225.43	2.88	0.142	1.47
0	0	7	234.98	192.15	2.82	0.132	1.77
-4	-1	2	118.12	94.43	2.82	0.092	2.08
-7	-1	10	7.51	2.75	2.82	0.016	0.92
-7	-3	9	11.21	5.43	2.77	0.022	0.92
-3	0	4	805.46	671.66	2.75	0.246	2.24
-2	-3	6	83.90	66.24	2.72	0.077	1.62
-3	0	8	15.19	9.53	2.70	0.029	1.42
4	0	3	447.08	371.93	2.70	0.183	1.87
-6	-3	5	35.71	26.02	2.66	0.048	1.20
2	-5	5	27.94	19.77	2.66	0.042	1.34
3	-3	5	84.00	105.73	2.65	0.098	1.54
-6	0	5	14.12	8.75	2.64	0.028	1.32
7	-7	3	-0.81	3.07	2.61	0.017	0.86
1	0	6	78.83	62.92	2.60	0.075	1.97
-8	-5	5	11.35	18.53	2.59	0.041	0.90
-1	0	1	110.76	136.21	2.59	0.111	7.51
-7	-7	1	1.15	5.57	2.58	0.022	0.89
2	0	6	187.63	155.03	2.57	0.118	1.81
-7	-3	11	7.56	2.93	2.55	0.016	0.84
6	-7	3	1.33	5.49	2.54	0.022	0.92
-6	-6	8	5.31	1.36	2.52	0.011	0.89

-5	-4	9	9.26	4.47	2.50	0.020	1.01
-5	-1	8	128.68	105.14	2.50	0.097	1.21
9	-3	2	-0.74	2.48	2.44	0.015	0.91
-1	0	2	9427.82	11108.30	2.44	1.000	5.30
1	-4	7	33.68	25.34	2.39	0.048	1.35
3	-5	3	29.12	21.61	2.38	0.044	1.39
-2	-1	5	119.18	98.80	2.37	0.094	2.18
7	0	10	-2.32	0.58	2.36	0.007	0.84
-5	-2	12	5.11	1.51	2.35	0.012	0.91
1	-4	4	31.29	23.76	2.33	0.046	1.73
2	-4	3	338.76	288.14	2.32	0.161	1.74
2	-3	2	508.92	435.07	2.32	0.198	2.23
7	-6	1	10.17	5.58	2.32	0.022	0.95
2	-4	2	77.97	63.69	2.32	0.076	1.85
0	-10	0	-2.25	0.51	2.30	0.007	0.88
-2	0	6	729.05	625.98	2.29	0.237	1.94
-6	-3	9	9.77	15.34	2.29	0.037	0.99
0	-1	5	364.20	311.60	2.27	0.167	2.38

X-Ray data of compound **28**

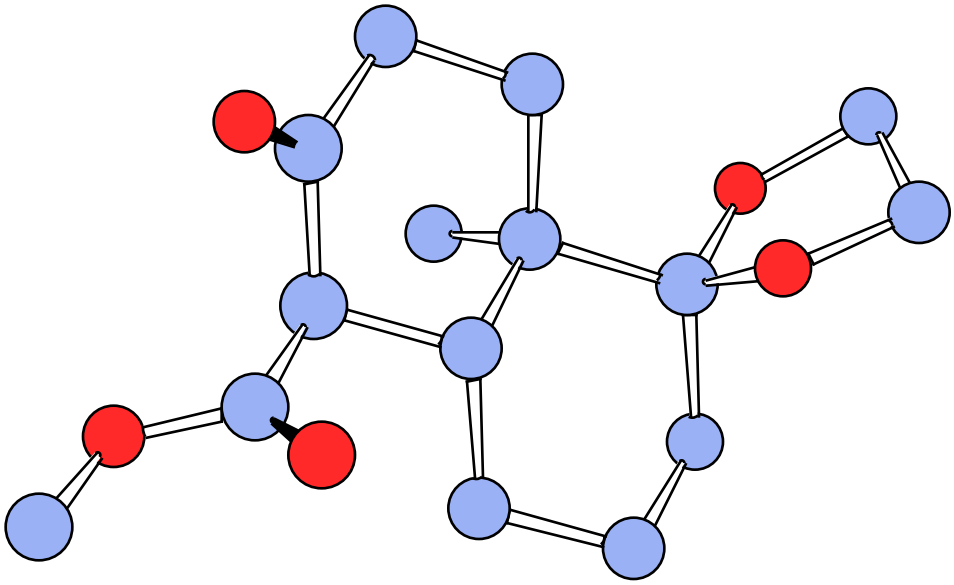


Table 1. Crystal data and structure refinement for **28**

Empirical formula	C15 H22 O5
Formula weight	282.33
Temperature	23 C
Wavelength	0.71073 A
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 9.089(8)A alpha = 87.97(6)
	b = 10.019(9)A beta = 79.22(6)
	c = 16.778(12)A gamma = 74.72(7)
Crystal size	1.40 x 1.00 x 0.70 mm
Density (calculated)	1.295 Mg/m^3
Absorption coefficient	0.096 mm^-1
Volume, Z, F(000)	1448(2) A^3, 4, 608
Theta range for data collection	2.11 to 22.54 degrees
Limiting indices	-9 < h < 0, -10 < k <10, -18 < l < 17
Reflections collected	4059, 3220 observed [I>2sigma(I)]
Independent reflections	3769 (Rint = 0.0171)
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3767 / 0 / 362
Goodness-of-fit on F^2	1.030
Final R indices [I>2sigma(I)]	R1 = 0.0412, wR2 = 0.1078
R indices (all data)	R1 = 0.0491, wR2 = 0.1158
Extinction coefficient&9&1	0.0124(17)
Largest diff. peak and hole	0.208 and -0.199 e/A^3

Scan speed, range, type 10 degrees/minute, 0.6 degrees, Wyckoff
Background range, % time 0.6 degrees, 25% each side

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [A^2 x 10^3] for **28**. U(eq) is defined as one third of the trace of the orthogonalized U(ij) tensor.

	x	y	z	U(eq)
O(1)	6124(2)	6469(2)	5549(1)	54(1)
O(2)	4664(2)	7958(1)	6578(1)	46(1)
O(3)	334(2)	4967(3)	7891(1)	93(1)
O(4)	2544(2)	3425(2)	9117(1)	62(1)
O(5)	2037(2)	5725(2)	9220(1)	74(1)
C(1)	5545(3)	7782(2)	5207(1)	59(1)
C(2)	5155(3)	8762(2)	5901(1)	54(1)
C(3)	5589(2)	6565(2)	6415(1)	43(1)
C(4)	6975(3)	6302(2)	6839(1)	54(1)
C(5)	6470(3)	6350(3)	7757(1)	58(1)
C(6)	5483(3)	5346(2)	8041(1)	49(1)
C(7)	4079(2)	5610(2)	7616(1)	37(1)
C(8)	4557(2)	5553(2)	6678(1)	38(1)
C(9)	5501(3)	4093(2)	6362(1)	51(1)
C(10)	3066(3)	5986(2)	6311(1)	48(1)
C(11)	1913(3)	5114(3)	6599(1)	56(1)
C(12)	1623(3)	4920(2)	7501(1)	53(1)
C(13)	3037(2)	4623(2)	7907(1)	41(1)
C(14)	2489(3)	4682(2)	8815(1)	49(1)
C(15)	1925(4)	3351(3)	9975(2)	90(1)
O(1')	7631(2)	333(2)	9605(1)	55(1)
O(2')	7066(2)	2197(1)	8788(1)	51(1)
O(3')	12679(2)	1756(2)	6787(1)	59(1)
O(4')	9829(2)	2256(2)	5949(1)	64(1)
O(5')	11503(2)	326(2)	5371(1)	73(1)
C(1')	6862(4)	1494(3)	10108(2)	92(1)
C(2')	6517(4)	2664(3)	9590(2)	87(1)
C(3')	7603(2)	729(2)	8779(1)	42(1)
C(4')	6451(3)	129(2)	8455(1)	54(1)
C(5')	6408(3)	502(3)	7569(1)	59(1)
C(6')	8031(3)	136(3)	7035(1)	54(1)
C(7')	9150(2)	756(2)	7385(1)	39(1)
C(8')	9275(2)	284(2)	8266(1)	39(1)
C(9')	9908(3)	-1289(2)	8327(2)	57(1)
C(10')	10345(2)	1015(2)	8586(1)	48(1)
C(11')	11976(3)	735(3)	8058(1)	55(1)
C(12')	11893(2)	1068(2)	7189(1)	44(1)
C(13')	10780(2)	470(2)	6825(1)	42(1)
C(14')	10752(3)	979(3)	5966(1)	50(1)
C(15')	9757(4)	2851(4)	5154(2)	89(1)

Table 3. Bond lengths [A] and angles [degrees] for **28**

O(1)-C(1)	1.426(3)
O(1)-C(3)	1.442(3)
O(2)-C(3)	1.433(3)
O(2)-C(2)	1.440(3)
O(3)-C(12)	1.220(3)
O(4)-C(14)	1.333(3)
O(4)-C(15)	1.451(3)

O(5)-C(14)	1.201(3)
C(1)-C(2)	1.479(3)
C(3)-C(4)	1.520(3)
C(3)-C(8)	1.555(3)
C(4)-C(5)	1.522(3)
C(5)-C(6)	1.524(3)
C(6)-C(7)	1.535(3)
C(7)-C(8)	1.551(3)
C(7)-C(13)	1.549(3)
C(8)-C(10)	1.546(3)
C(8)-C(9)	1.543(3)
C(10)-C(11)	1.535(3)
C(11)-C(12)	1.501(3)
C(12)-C(13)	1.522(3)
C(13)-C(14)	1.509(3)
O(1')-C(1')	1.405(3)
O(1')-C(3')	1.430(3)
O(2')-C(2')	1.396(3)
O(2')-C(3')	1.423(3)
O(3')-C(12')	1.219(3)
O(4')-C(14')	1.335(3)
O(4')-C(15')	1.446(3)
O(5')-C(14')	1.203(3)
C(1')-C(2')	1.436(4)
C(3')-C(4')	1.523(3)
C(3')-C(8')	1.559(3)
C(4')-C(5')	1.524(3)
C(5')-C(6')	1.535(3)
C(6')-C(7')	1.533(3)
C(7')-C(8')	1.550(3)
C(7')-C(13')	1.560(3)
C(8')-C(9')	1.536(3)
C(8')-C(10')	1.540(3)
C(10')-C(11')	1.539(3)
C(11')-C(12')	1.495(3)
C(12')-C(13')	1.527(3)
C(13')-C(14')	1.515(3)
C(1)-O(1)-C(3)	108.7(2)
C(3)-O(2)-C(2)	106.3(2)
C(14)-O(4)-C(15)	116.9(2)
O(1)-C(1)-C(2)	104.0(2)
O(2)-C(2)-C(1)	103.1(2)
O(2)-C(3)-O(1)	105.7(2)
O(2)-C(3)-C(4)	109.5(2)
O(1)-C(3)-C(4)	109.4(2)
O(2)-C(3)-C(8)	109.2(2)
O(1)-C(3)-C(8)	109.8(2)
C(4)-C(3)-C(8)	112.9(2)
C(5)-C(4)-C(3)	111.4(2)
C(4)-C(5)-C(6)	111.6(2)
C(5)-C(6)-C(7)	111.3(2)
C(6)-C(7)-C(8)	112.4(2)
C(6)-C(7)-C(13)	112.4(2)
C(8)-C(7)-C(13)	110.7(2)
C(10)-C(8)-C(9)	109.4(2)
C(10)-C(8)-C(7)	108.3(2)
C(9)-C(8)-C(7)	112.0(2)
C(10)-C(8)-C(3)	110.4(2)

C(9)-C(8)-C(3)	107.9(2)
C(7)-C(8)-C(3)	108.9(2)
C(11)-C(10)-C(8)	113.9(2)
C(12)-C(11)-C(10)	113.6(2)
O(3)-C(12)-C(11)	122.4(2)
O(3)-C(12)-C(13)	121.3(2)
C(11)-C(12)-C(13)	116.3(2)
C(14)-C(13)-C(12)	108.4(2)
C(14)-C(13)-C(7)	112.7(2)
C(12)-C(13)-C(7)	112.0(2)
O(5)-C(14)-O(4)	123.6(2)
O(5)-C(14)-C(13)	124.9(2)
O(4)-C(14)-C(13)	111.6(2)
C(1')-O(1')-C(3')	108.6(2)
C(2')-O(2')-C(3')	108.9(2)
C(14')-O(4')-C(15')	116.1(2)
O(1')-C(1')-C(2')	107.3(2)
O(2')-C(2')-C(1')	107.8(2)
O(2')-C(3')-O(1')	106.2(2)
O(2')-C(3')-C(4')	108.6(2)
O(1')-C(3')-C(4')	109.7(2)
O(2')-C(3')-C(8')	109.1(2)
O(1')-C(3')-C(8')	109.9(2)
C(4')-C(3')-C(8')	113.1(2)
C(5')-C(4')-C(3')	111.5(2)
C(4')-C(5')-C(6')	112.3(2)
C(5')-C(6')-C(7')	111.2(2)
C(6')-C(7')-C(8')	112.2(2)
C(6')-C(7')-C(13')	112.1(2)
C(8')-C(7')-C(13')	111.5(2)
C(9')-C(8')-C(10')	109.4(2)
C(9')-C(8')-C(7')	112.8(2)
C(10')-C(8')-C(7')	108.8(2)
C(9')-C(8')-C(3')	108.8(2)
C(10')-C(8')-C(3')	110.3(2)
C(7')-C(8')-C(3')	106.8(2)
C(11')-C(10')-C(8')	113.2(2)
C(12')-C(11')-C(10')	111.3(2)
O(3')-C(12')-C(11')	122.5(2)
O(3')-C(12')-C(13')	121.6(2)
C(11')-C(12')-C(13')	115.9(2)
C(14')-C(13')-C(12')	109.3(2)
C(14')-C(13')-C(7')	114.4(2)
C(12')-C(13')-C(7')	111.6(2)
O(5')-C(14')-O(4')	124.0(2)
O(5')-C(14')-C(13')	124.1(2)
O(4')-C(14')-C(13')	111.9(2)

Table 4. Anisotropic displacement parameters [A^2 x 10^3]
for TAOT4&NThe anisotropic displacement factor exponent takes the form:
-2(pi)^2 [(ha*)^2U11 + ... + 2hka*b*U12]

	U11	U22	U33	U23	U13	U12
O(1)	72(1)	44(1)	38(1)	1(1)	12(1)	-15(1)
O(2)	60(1)	36(1)	38(1)	3(1)	3(1)	-13(1)
O(3)	51(1)	144(2)	83(1)	22(1)	-3(1)	-34(1)
O(4)	75(1)	54(1)	48(1)	18(1)	7(1)	-16(1)
O(5)	104(1)	60(1)	47(1)	-7(1)	16(1)	-25(1)
C(1)	84(2)	52(1)	40(1)	7(1)	2(1)	-26(1)
C(2)	72(2)	45(1)	43(1)	8(1)	-1(1)	-20(1)
C(3)	49(1)	40(1)	35(1)	0(1)	4(1)	-11(1)
C(4)	46(1)	54(1)	63(2)	6(1)	-5(1)	-19(1)
C(5)	57(1)	70(2)	58(2)	7(1)	-21(1)	-26(1)
C(6)	52(1)	51(1)	44(1)	6(1)	-11(1)	-13(1)
C(7)	41(1)	32(1)	35(1)	2(1)	-2(1)	-7(1)
C(8)	45(1)	34(1)	34(1)	1(1)	-2(1)	-10(1)
C(9)	62(1)	41(1)	44(1)	-3(1)	4(1)	-13(1)
C(10)	58(1)	50(1)	41(1)	9(1)	-14(1)	-19(1)
C(11)	57(1)	66(2)	55(1)	12(1)	-20(1)	-27(1)
C(12)	51(1)	53(1)	58(2)	7(1)	-7(1)	-21(1)
C(13)	46(1)	35(1)	39(1)	3(1)	-1(1)	-10(1)
C(14)	52(1)	48(1)	43(1)	7(1)	1(1)	-15(1)
C(15)	102(2)	95(2)	56(2)	32(2)	14(2)	-22(2)
O(1')	62(1)	55(1)	40(1)	11(1)	-3(1)	-10(1)
O(2')	63(1)	40(1)	42(1)	1(1)	-4(1)	-3(1)
O(3')	63(1)	64(1)	55(1)	6(1)	-2(1)	-33(1)
O(4')	67(1)	74(1)	43(1)	19(1)	-4(1)	-9(1)
O(5')	74(1)	97(1)	41(1)	-12(1)	1(1)	-15(1)
C(1')	131(3)	78(2)	47(2)	0(2)	1(2)	-3(2)
C(2')	119(2)	74(2)	46(2)	-11(1)	-15(2)	14(2)
C(3')	48(1)	39(1)	36(1)	9(1)	-4(1)	-11(1)
C(4')	48(1)	60(2)	56(1)	3(1)	-1(1)	-22(1)
C(5')	48(1)	79(2)	58(2)	-2(1)	-11(1)	-27(1)
C(6')	55(1)	69(2)	45(1)	-3(1)	-9(1)	-27(1)
C(7')	40(1)	38(1)	39(1)	2(1)	-5(1)	-12(1)
C(8')	41(1)	36(1)	39(1)	6(1)	-6(1)	-12(1)
C(9')	58(1)	42(1)	63(2)	11(1)	-2(1)	-8(1)
C(10')	52(1)	56(1)	39(1)	6(1)	-11(1)	-19(1)
C(11')	50(1)	71(2)	50(1)	10(1)	-15(1)	-26(1)
C(12')	40(1)	44(1)	46(1)	1(1)	-3(1)	-10(1)
C(13')	44(1)	41(1)	39(1)	1(1)	-4(1)	-12(1)
C(14')	45(1)	66(2)	42(1)	3(1)	-5(1)	-20(1)
C(15')	84(2)	118(3)	53(2)	37(2)	-10(2)	-12(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for TAOT4

	x	y	z	U(eq)
H(1A)	4633(3)	7800(2)	4981(1)	71
H(1B)	6328(3)	8002(2)	4784(1)	71
H(2A)	6053(3)	9062(2)	5974(1)	65
H(2B)	4326(3)	9567(2)	5827(1)	65
H(4A)	7669(3)	5402(2)	6673(1)	65
H(4B)	7539(3)	6995(2)	6677(1)	65
H(5A)	5878(3)	7282(3)	7929(1)	70
H(5B)	7381(3)	6118(3)	8009(1)	70
H(6A)	5125(3)	5448(2)	8623(1)	59
H(6B)	6112(3)	4405(2)	7927(1)	59
H(7A)	3449(2)	6552(2)	7765(1)	45
H(9A)	6428(3)	3822(2)	6590(1)	76
H(9B)	5775(3)	4101(2)	5781(1)	76
H(9C)	4888(3)	3447(2)	6518(1)	76
H(10A)	2554(3)	6949(2)	6451(1)	58
H(10B)	3353(3)	5913(2)	5724(1)	58
H(11A)	935(3)	5558(3)	6433(1)	68
H(11B)	2309(3)	4213(3)	6334(1)	68
H(13A)	3653(2)	3676(2)	7757(1)	50
H(15A)	2027(4)	2401(3)	10123(2)	134
H(15B)	848(4)	3850(3)	10084(2)	134
H(15C)	2489(4)	3755(3)	10286(2)	134
H(1'A)	7518(4)	1644(3)	10472(2)	110
H(1'B)	5912(4)	1356(3)	10431(2)	110
H(2'A)	5406(4)	3077(3)	9675(2)	104
H(2'B)	7017(4)	3357(3)	9710(2)	104
H(4'A)	6741(3)	-869(2)	8503(1)	65
H(4'B)	5425(3)	482(2)	8778(1)	65
H(5'A)	5941(3)	1486(3)	7535(1)	71
H(5'B)	5762(3)	13(3)	7365(1)	71
H(6'A)	7960(3)	484(3)	6493(1)	65
H(6'B)	8432(3)	-863(3)	6995(1)	65
H(7'A)	8713(2)	1762(2)	7409(1)	47
H(9'A)	9243(3)	-1747(2)	8127(2)	85
H(9'B)	9943(3)	-1529(2)	8883(2)	85
H(9'C)	10935(3)	-1575(2)	8008(2)	85
H(10C)	9871(2)	2004(2)	8612(1)	57
H(10D)	10441(2)	708(2)	9133(1)	57
H(11C)	12560(3)	1292(3)	8257(1)	66
H(11D)	12519(3)	-231(3)	8098(1)	66
H(13B)	11210(2)	-536(2)	6791(1)	50
H(15D)	9065(4)	3763(4)	5210(2)	133
H(15E)	9385(4)	2281(4)	4834(2)	133
H(15F)	10774(4)	2902(4)	4893(2)	133

-Experimental
R1=(&GS&GB&GBF&Vo&0&GB-&GBF&Vc&0&GB&GB/&GS&GBF&Vo&0&GB),
& wR2=&GSw(F&Vo&0&^2&0-F&Vc&0&^2&0)&^2&0/&GSw[(F&Vo&0&^2&0)&^2&0]&^&Gh&0,
& S=[&GSw(F&Vo&0&^2&0-F&Vc&0&^2&0)&^2&0/(n-p)]&^&Gh&0

References:
H.D.Flack. Acta Crystallographica, A39, 876-881(1983).
SHELXTL-PC.& G.M.Sheldrick, Siemens Analytical X-ray Instruments
Inc. Madison, WI. (1990).

The worst:

	h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Res(A)
	7	5	8	30.80	9.08	5.77	0.020	1.11
	4	3	8	7.07	0.00	4.56	0.000	1.59
	5	5	0	9.53	1.15	4.41	0.007	1.50
	4	3	6	230.62	165.43	4.20	0.087	1.80
	3	2	3	45.20	74.96	4.13	0.058	2.70
	3	3	7	25.90	47.24	3.96	0.046	1.85
	-2	-1	1	15.95	6.47	3.88	0.017	4.06
	0	0	10	14.00	5.12	3.87	0.015	1.65
	0	3	0	3.27	0.00	3.83	0.000	3.22
	-7	0	5	55.21	33.10	3.77	0.039	1.09
*	0	0	8	-13.27	3.01	3.77	0.012	2.06
	-1	0	11	18.09	7.37	3.71	0.018	1.43
*	-4	6	1	-5.55	0.12	3.45	0.002	1.14
	2	5	4	4.88	0.28	3.41	0.004	1.82
	-2	0	10	108.17	77.36	3.39	0.059	1.45
	4	2	3	2.02	9.04	3.34	0.020	2.18
	7	1	13	-4.39	0.71	3.31	0.006	0.99
	-4	-3	2	115.15	84.10	3.30	0.062	1.92
	2	4	2	35.48	21.57	3.26	0.031	2.35
	0	-2	8	235.25	180.03	3.24	0.091	1.91
	-2	2	6	11.52	4.49	3.17	0.014	1.86
	5	3	1	3.27	10.78	3.17	0.022	1.74
	0	-4	6	68.39	46.83	3.14	0.046	1.83
	8	6	8	14.84	5.82	3.11	0.016	0.99
	-2	-3	1	86.63	117.88	3.09	0.073	2.88
	-2	-3	6	199.97	155.54	3.09	0.084	1.91
	1	-4	2	57.77	39.63	3.07	0.043	2.14
	2	-9	1	-2.29	3.72	3.06	0.013	0.98
	-4	1	2	46.28	30.60	3.05	0.037	1.87
	0	-5	4	116.05	87.83	2.98	0.063	1.76
	-1	-3	7	85.93	62.83	2.98	0.054	1.87
	-1	4	5	168.26	130.82	2.95	0.077	1.77
	0	0	16	14.44	6.75	2.92	0.018	1.03
	8	0	10	29.68	17.37	2.86	0.028	0.99
	-2	-2	6	87.35	64.98	2.84	0.054	2.07
	5	3	3	-0.26	4.10	2.84	0.014	1.73
	-2	-2	7	519.27	427.99	2.82	0.140	1.87
	0	1	3	20.17	32.18	2.81	0.038	4.75
	-3	0	6	4.17	0.51	2.81	0.005	1.82
	3	2	6	24.14	37.49	2.79	0.041	2.16
	5	-1	7	10.79	4.41	2.76	0.014	1.46
	-3	-2	5	135.98	106.11	2.75	0.070	1.98
	2	1	2	207.04	258.06	2.74	0.109	4.20
	0	3	4	105.53	136.39	2.73	0.079	2.52
	6	1	7	43.83	29.77	2.73	0.037	1.37
	2	-6	1	-2.73	0.25	2.72	0.003	1.40
	5	-4	4	19.62	10.50	2.70	0.022	1.25
	0	-1	15	98.88	75.48	2.70	0.059	1.09
	7	2	1	13.08	5.75	2.70	0.016	1.29
	2	1	3	874.81	1048.87	2.69	0.219	3.78

X-Ray data of compound **29**

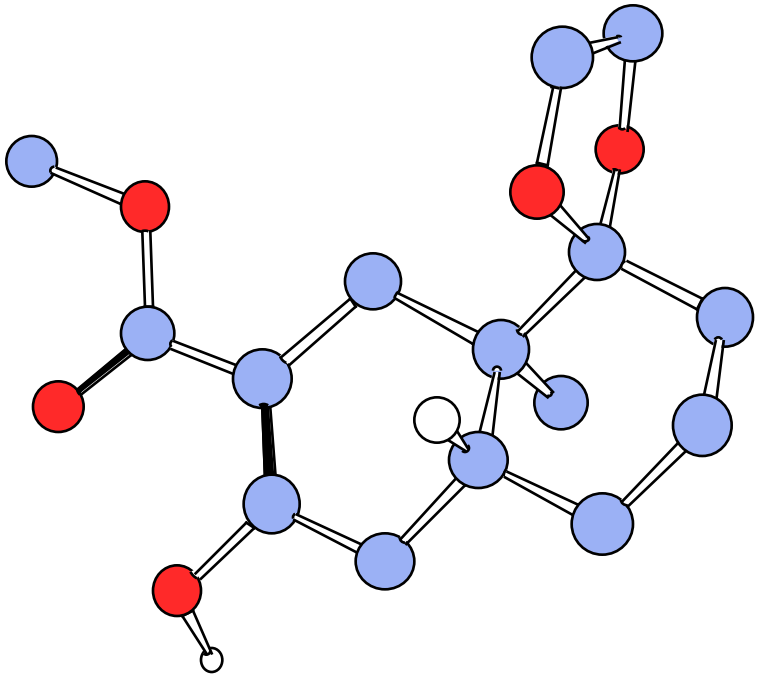


Table 1. Crystal data and structure refinement for **29**

Empirical formula	C15 H22 O5
Formula weight	282.33
Temperature	-86 C
Wavelength	0.71073 A
Crystal system	Orthorhombic
Space group	Pbca
Unit cell dimensions	a = 13.33(2)A alpha = 90
	b = 10.92(2)A beta = 90
	c = 19.57(5)A gamma = 90
Crystal size	2.50 x 1.00 x 0.40 mm
Density (calculated)	1.317 Mg/m^3
Absorption coefficient	0.098 mm^-1
Volume, Z, F(000)	2849(10) A^3, 8, 1216
Theta range for data collection	2.58 to 22.49 degrees
Limiting indices	-1 < h < 14, 0 < k <11, -21 < l < 0
Reflections collected	1903, 1104 observed [I>2sigma(I)]
Independent reflections	1748 (Rint = 0.0943)
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1738 / 0 / 181
Goodness-of-fit on F^2	1.155
Final R indices [I>2sigma(I)]	R1 = 0.1054, wR2 = 0.2333
R indices (all data)	R1 = 0.1661, wR2 = 0.2881
Largest diff. peak and hole	0.274 and -0.235 e/A^3

Scan speed, range, type 10 degrees/minute, 1.2 degrees, Wyckoff
Background range, % time 1.5 degrees, 25% each side

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [A^2 x 10^3] for **29**. U(eq) is defined as one third of the trace of the orthogonalized U(ij) tensor.

	x	y	z	U(eq)
O(1)	7360(4)	6752(5)	3647(2)	50(2)
O(2)	9139(4)	6315(5)	1272(2)	49(2)
O(3)	8390(5)	6145(6)	4485(2)	68(2)
O(4)	7793(4)	5014(5)	1240(2)	57(2)
O(5)	9994(5)	4966(6)	4174(2)	68(2)
C(1)	7596(9)	5975(10)	760(4)	76(3)
C(2)	8403(8)	6882(8)	865(4)	68(3)
C(3)	8844(5)	5055(7)	1385(3)	41(2)
C(4)	9431(6)	4211(8)	914(4)	53(2)
C(5)	10543(7)	4240(9)	1061(4)	60(2)
C(6)	10757(7)	3981(8)	1812(4)	59(2)
C(7)	10145(6)	4781(7)	2292(3)	46(2)
C(8)	8992(5)	4730(6)	2153(3)	36(2)
C(9)	8572(6)	3432(7)	2285(3)	49(2)
C(10)	8460(6)	5670(7)	2601(3)	43(2)
C(11)	8805(6)	5607(7)	3346(3)	41(2)
C(12)	9677(6)	5076(8)	3524(4)	47(2)
C(13)	10365(6)	4477(8)	3037(4)	55(2)
C(14)	8177(6)	6161(7)	3868(4)	46(2)
C(15)	6772(7)	7387(8)	4145(4)	63(2)

Table 3. Bond lengths [A] and angles [degrees] for **29**.

O(1)-C(14)	1.338(9)
O(1)-C(15)	1.430(9)
O(2)-C(2)	1.408(10)
O(2)-C(3)	1.449(9)
O(3)-C(14)	1.241(9)
O(4)-C(3)	1.430(9)
O(4)-C(1)	1.433(10)
O(5)-C(12)	1.346(9)
C(1)-C(2)	1.477(13)
C(3)-C(4)	1.519(11)
C(3)-C(8)	1.558(10)
C(4)-C(5)	1.511(12)
C(5)-C(6)	1.523(11)
C(6)-C(7)	1.521(11)
C(7)-C(13)	1.523(11)
C(7)-C(8)	1.561(11)
C(8)-C(10)	1.524(10)
C(8)-C(9)	1.546(10)
C(10)-C(11)	1.532(10)
C(11)-C(12)	1.345(11)
C(11)-C(14)	1.453(11)
C(12)-C(13)	1.475(11)
C(14)-O(1)-C(15)	117.3(6)
C(2)-O(2)-C(3)	108.3(6)
C(3)-O(4)-C(1)	106.7(6)
O(4)-C(1)-C(2)	105.5(7)
O(2)-C(2)-C(1)	107.0(7)
O(4)-C(3)-O(2)	105.4(6)

O(4)-C(3)-C(4)	111.5(6)
O(2)-C(3)-C(4)	110.1(6)
O(4)-C(3)-C(8)	108.0(5)
O(2)-C(3)-C(8)	109.2(5)
C(4)-C(3)-C(8)	112.5(6)
C(5)-C(4)-C(3)	112.2(6)
C(4)-C(5)-C(6)	111.2(7)
C(7)-C(6)-C(5)	113.0(7)
C(6)-C(7)-C(13)	111.3(6)
C(6)-C(7)-C(8)	113.6(6)
C(13)-C(7)-C(8)	110.4(6)
C(10)-C(8)-C(9)	110.7(6)
C(10)-C(8)-C(3)	110.0(6)
C(9)-C(8)-C(3)	108.9(5)
C(10)-C(8)-C(7)	109.5(6)
C(9)-C(8)-C(7)	111.1(6)
C(3)-C(8)-C(7)	106.5(5)
C(8)-C(10)-C(11)	112.1(6)
C(12)-C(11)-C(14)	119.7(6)
C(12)-C(11)-C(10)	121.7(6)
C(14)-C(11)-C(10)	118.5(7)
C(11)-C(12)-O(5)	123.6(7)
C(11)-C(12)-C(13)	124.1(6)
O(5)-C(12)-C(13)	112.2(7)
C(12)-C(13)-C(7)	113.7(7)
O(3)-C(14)-O(1)	120.5(7)
O(3)-C(14)-C(11)	123.1(7)
O(1)-C(14)-C(11)	116.3(6)

Table 4. Anisotropic displacement parameters [A^2 x 10^3] for **29**. The anisotropic displacement factor exponent takes the form: -2(pi)^2 [(ha*)^2U11 + ... + 2hka*b*U12]

	U11	U22	U33	U23	U13	U12
O(1)	60(3)	62(3)	29(2)	-4(2)	5(2)	16(3)
O(2)	58(3)	47(3)	41(3)	15(2)	-9(2)	-8(3)
O(3)	90(5)	91(4)	24(3)	-3(3)	-9(3)	15(4)
O(4)	54(4)	82(4)	33(3)	8(3)	-7(2)	-2(3)
O(5)	78(4)	93(4)	31(3)	-1(3)	-20(3)	18(4)
C(1)	83(7)	93(7)	52(5)	17(5)	-21(5)	7(6)
C(2)	91(7)	64(6)	48(5)	10(4)	-3(5)	15(6)
C(3)	43(4)	53(5)	25(3)	1(3)	-4(3)	-5(4)
C(4)	64(6)	63(5)	31(4)	2(4)	1(4)	-4(4)
C(5)	62(6)	76(6)	41(4)	-6(4)	5(4)	8(5)
C(6)	59(5)	72(6)	47(4)	-1(4)	2(4)	20(5)
C(7)	50(5)	56(5)	32(4)	-1(3)	-7(4)	3(4)
C(8)	46(4)	36(4)	25(3)	0(3)	-4(3)	1(3)
C(9)	67(5)	48(4)	32(4)	3(3)	0(4)	-10(4)
C(10)	56(5)	50(4)	24(4)	-1(3)	-9(3)	11(4)
C(11)	51(5)	45(4)	27(4)	0(3)	-7(3)	4(4)
C(12)	57(5)	55(5)	30(4)	0(4)	-10(4)	-4(4)
C(13)	57(5)	64(5)	43(4)	1(4)	-16(4)	10(4)
C(14)	56(5)	47(5)	33(4)	0(3)	-5(4)	1(4)
C(15)	75(6)	69(6)	44(4)	-6(4)	9(4)	18(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **29**.

	x	y	z	U(eq)
H(5A)	9577(5)	5269(6)	4432(2)	101
H(1A)	6944(9)	6340(10)	846(4)	91
H(1B)	7608(9)	5663(10)	296(4)	91
H(2A)	8686(8)	7128(8)	429(4)	81
H(2B)	8142(8)	7604(8)	1093(4)	81
H(4A)	9186(6)	3380(8)	968(4)	64
H(4B)	9317(6)	4455(8)	444(4)	64
H(5A)	10810(7)	5038(9)	941(4)	72
H(5B)	10879(7)	3632(9)	781(4)	72
H(6A)	10610(7)	3128(8)	1907(4)	71
H(6B)	11464(7)	4114(8)	1900(4)	71
H(7A)	10358(6)	5630(7)	2217(3)	55
H(9A)	8915(6)	2853(7)	1999(3)	73
H(9B)	7868(6)	3418(7)	2182(3)	73
H(9C)	8671(6)	3217(7)	2756(3)	73
H(10A)	7742(6)	5530(7)	2580(3)	52
H(10B)	8590(6)	6485(7)	2423(3)	52
H(13A)	10322(6)	3597(8)	3098(4)	66
H(13B)	11047(6)	4724(8)	3141(4)	66
H(15A)	6211(7)	7771(8)	3925(4)	94
H(15B)	7177(7)	8000(8)	4363(4)	94
H(15C)	6534(7)	6816(8)	4481(4)	94

-Experimental
R1=(&GS&GB&GBF&Vo&0&GB-&GBF&Vc&0&GB&GB/&GS&GBF&Vo&0&GB),
& wR2=&GSw(F&Vo&0&^2&0-F&Vc&0&^2&0)&^2&0/&GSw[(F&Vo&0&^2&0)&^2&0]&^&Gh&0,
& S=[&GSw(F&Vo&0&^2&0-F&Vc&0&^2&0)&^2&0/(n-p)]&^&Gh&0

References:

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SHELXTL-PC.& G.M.Sheldrick, Siemens Analytical X-ray Instruments
Inc. Madison, WI. (1990).

The worst:

	h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Res(A)
	7	8	3	718.95	0.08	8.39	0.004	1.09
*	6	4	2	-168.88	32.77	5.88	0.078	1.70
*	6	3	4	-113.89	2.55	5.61	0.022	1.77
	8	6	1	461.95	66.73	5.04	0.112	1.23
	5	8	5	367.78	49.06	4.93	0.096	1.16
	7	6	5	493.67	115.03	4.90	0.147	1.25
	10	3	4	431.92	65.88	4.87	0.111	1.21
	1	2	1	1790.77	1048.74	4.74	0.444	4.89
	10	5	1	510.72	115.09	4.73	0.147	1.14
	0	10	5	430.32	108.18	4.35	0.142	1.05
	10	4	1	249.72	7.65	4.27	0.038	1.20
	2	7	2	291.56	53.00	4.26	0.100	1.50
	5	5	3	-74.09	160.48	4.26	0.174	1.64
	0	8	6	299.11	31.73	4.26	0.077	1.26

	0	8	1	628.96	257.74	4.12	0.220	1.36
	8	3	2	-101.27	121.79	4.11	0.151	1.50
*	9	2	4	-113.36	5.41	3.99	0.032	1.37
	7	7	6	169.23	0.48	3.90	0.010	1.13
	5	6	3	550.17	200.70	3.90	0.194	1.46
*	10	3	0	-94.88	62.51	3.80	0.108	1.25
*	2	6	5	-74.80	2.36	3.73	0.021	1.60
	3	7	4	673.12	327.14	3.58	0.248	1.41
	3	11	5	241.19	42.11	3.55	0.089	0.94
	5	10	6	152.80	3.41	3.48	0.025	0.97
	8	3	0	-5.95	217.47	3.48	0.202	1.52
	8	4	1	-19.01	212.70	3.41	0.200	1.42
	1	3	1	6112.58	5024.23	3.41	0.971	3.46
	0	10	4	1028.28	598.96	3.36	0.335	1.07
	5	9	4	169.68	15.88	3.35	0.055	1.08
	0	8	8	79.42	310.13	3.24	0.241	1.19
	2	10	4	155.91	10.01	3.23	0.043	1.05
	4	6	1	-74.53	56.26	3.18	0.103	1.59
*	2	7	6	-62.10	7.64	3.18	0.038	1.38
	9	7	4	146.85	1.77	3.12	0.018	1.05
*	9	1	4	-64.41	19.04	3.11	0.060	1.41
*	10	2	1	-79.15	10.19	3.10	0.044	1.29
	2	6	4	572.91	967.27	3.06	0.426	1.65
	8	5	3	321.77	107.22	3.05	0.142	1.30
	2	7	5	174.65	421.75	2.96	0.281	1.42
*	11	1	2	-79.51	1.68	2.93	0.018	1.20
*	9	3	13	-76.31	0.73	2.91	0.012	1.01
	6	7	5	457.33	221.04	2.88	0.204	1.21
	4	9	7	143.74	20.33	2.82	0.062	1.06
	9	5	4	590.94	315.29	2.81	0.243	1.19
	8	6	7	141.61	20.73	2.77	0.062	1.13
	6	8	2	368.23	165.66	2.70	0.176	1.15
	10	8	1	137.43	6.25	2.68	0.034	0.95
	5	10	3	112.73	6.11	2.66	0.034	1.00
	4	5	3	-60.23	1.12	2.65	0.014	1.76
	8	2	4	-44.86	25.13	2.65	0.069	1.52

X-Ray data of compound **41**

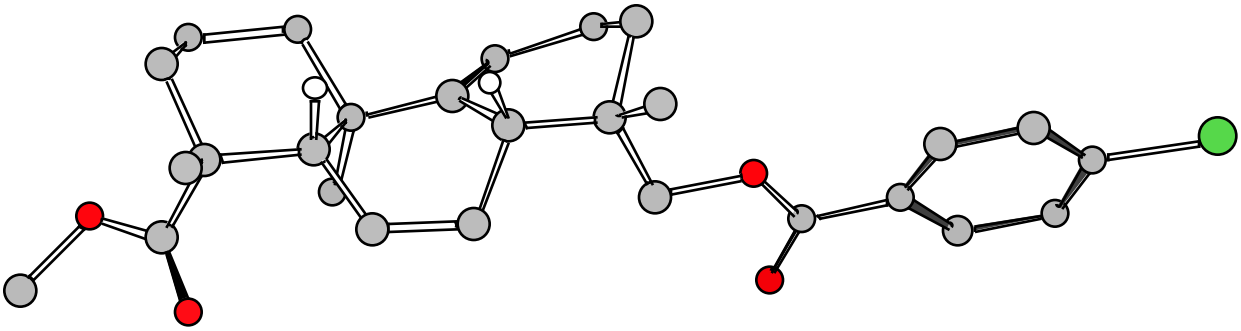

Table 1. Crystal data and structure refinement for **41**

Empirical formula	$C_{27}\ H_{34}\ BrO_4$
Formula weight	502.45
Temperature	23 C
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 11.00(2)Å alpha = 90°
	b = 19.19(2)Å beta = 109.82(9)°
	c = 12.459(13)Å gamma = 90°
Crystal size	0.50 x 0.50 x 0.15 mm
Density (calculated)	1.349 Mg/m^3
Absorption coefficient	1.691 mm^{-1}
Volume, Z, F(000)	2474(5) $Å^3$, 4, 1052
θ range for data collection	1.74 to 22.50°
Limiting indices	$0 \le h \le 11$, $0 \le k \le 20$, $-13 \le l \le 12$
Reflections collected	3502, 1796 observed [I>2σ(I)]
Independent reflections	3318 (R_{int} = 0.1414)
Absorption correction	Semi-empirical from psi-scans
Max. and min. transmission	0.9479 and 0.7019
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3318 / 1 / 257
Goodness-of-fit on F^2	1.034
Final R indices [I>2σ(I)]	R1 = 0.1129, wR2 = 0.2667
R indices (all data)	R1 = 0.1942, wR2 = 0.3406
Absolute structure parameter	-0.08(4)
Largest diff. peak and hole	0.738 and -1.052 $eÅ^{-3}$
Scan speed, range, type	10°/minute, 0.7°, Wyckoff
Background range, % time	1.0°, 25% each side

Table 2. Atomic coordinates [$x10^4$] and equivalent isotropic displacement

parameters [A^2 x 10^3] for **41**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1)	8405(13)	2734(6)	816(8)	74(9)
C(2)	8509(14)	3436(7)	1125(10)	53(6)
C(3)	8062(14)	3949(5)	289(13)	58(7)
C(4)	7512(15)	3759(6)	-855(11)	49(6)
C(5)	7408(12)	3057(6)	-1164(8)	58(7)
C(6)	7855(9)	2545(5)	-328(9)	47(6)
C(7)	7787(15)	1807(5)	-654(13)	35(5)
O(1)	7574(16)	1615(9)	-1648(14)	68(5)
O(2)	8029(15)	1378(9)	171(13)	57(4)
O(3)	8620(25)	-2925(12)	-1086(23)	122(9)
O(4)	7014(17)	-3555(10)	-1053(15)	71(5)
Br(1)	8205(3)	4885(1)	676(3)	83(1)
C(8)	8056(27)	632(13)	-75(22)	64(7)
C(9)	7978(23)	256(13)	936(19)	48(6)
C(10)	9131(27)	475(16)	2022(23)	79(8)
C(11)	6785(24)	503(15)	1267(22)	67(7)
C(12)	5619(32)	329(18)	293(28)	97(10)
C(13)	5581(25)	-427(13)	-155(21)	58(7)
C(14)	6614(23)	-821(13)	186(20)	55(6)
C(15)	7983(22)	-513(12)	858(20)	43(6)
C(16)	8907(20)	-826(11)	292(17)	39(5)
C(17)	9013(26)	-1614(15)	370(24)	72(8)
C(18)	7825(19)	-1976(11)	511(18)	37(5)
C(19)	6543(22)	-1576(13)	-165(19)	51(6)
C(20)	6270(26)	-1626(15)	-1451(21)	75(8)
C(21)	5405(25)	-1914(14)	129(24)	69(7)
C(22)	5349(23)	-2740(14)	-38(22)	69(7)
C(23)	6644(24)	-3062(16)	734(23)	72(8)
C(24)	7818(22)	-2806(13)	419(19)	51(6)
C(25)	8980(25)	-3051(16)	1302(23)	74(8)
C(26)	7873(23)	-3080(13)	-650(20)	47(6)
C(27)	7065(31)	-3908(21)	-2076(28)	102(11)
C(1')	7019(14)	3324(7)	4237(10)	75(9)
C(2')	6872(16)	2627(8)	3895(10)	80(9)
C(3')	7284(16)	2098(6)	4706(14)	62(7)
C(4')	7844(15)	2265(6)	5859(13)	51(6)
C(5')	7992(13)	2961(7)	6201(9)	72(8)
C(6')	7579(11)	3491(6)	5391(11)	49(6)
C(7')	7773(19)	4223(6)	5745(17)	73(8)
O(1')	8054(19)	4397(11)	6737(17)	85(6)
O(2')	7517(17)	4655(9)	4905(15)	69(5)
O(3')	8879(31)	8804(13)	6739(24)	165(14)
O(4')	7655(19)	9646(10)	5912(17)	79(6)
Br(2)	7085(4)	1170(1)	4275(3)	103(1)
C(8')	7776(30)	5400(15)	5187(25)	78(8)
C(9')	7786(28)	5743(16)	4082(24)	74(8)
C(10')	8943(29)	5517(17)	3808(26)	92(10)
C(11')	6569(31)	5576(20)	3156(29)	103(11)
C(12')	5407(34)	5792(19)	3318(31)	108(12)
C(13')	5492(31)	6514(17)	3927(26)	88(9)
C(14')	6641(24)	6877(14)	4277(20)	59(7)
C(15')	7783(29)	6565(17)	4337(27)	85(9)
C(16')	8908(29)	6792(16)	5391(25)	85(9)

C(17')	9066(31)	7615(18)	5372(26)	91(9)
C(18')	7929(22)	7946(12)	4603(21)	50(6)
C(19')	6632(24)	7669(14)	4632(21)	58(7)
C(20')	6519(27)	7689(15)	5817(22)	78(8)
C(21')	5492(28)	8042(15)	3764(25)	82(9)
C(22')	5500(33)	8865(19)	3898(32)	106(11)
C(23')	6827(28)	9093(18)	3837(27)	86(9)
C(24')	7959(29)	8787(17)	4673(26)	82(9)
C(25')	9205(23)	8992(14)	4436(21)	65(7)
C(26')	8155(30)	9058(18)	5868(28)	77(8)
C(27')	7931(27)	9970(20)	7016(25)	87(9)

Table 3. Bond lengths [Å] and angles [°] for **41**

C(1)-C(6)	1.40
C(1)-C(2)	1.40
C(2)-C(3)	1.40
C(3)-C(4)	1.39
C(3)-Br(1)	1.854(10)
C(4)-C(5)	1.40
C(5)-C(6)	1.40
C(6)-C(7)	1.47
C(7)-O(1)	1.24(2)
C(7)-O(2)	1.27(2)
O(2)-C(8)	1.47(3)
O(3)-C(26)	1.17(3)
O(4)-C(26)	1.29(3)
O(4)-C(27)	1.46(4)
C(8)-C(9)	1.48(3)
C(9)-C(15)	1.48(3)
C(9)-C(10)	1.57(3)
C(9)-C(11)	1.58(4)
C(11)-C(12)	1.47(4)
C(12)-C(13)	1.55(4)
C(13)-C(14)	1.31(3)
C(14)-C(19)	1.51(3)
C(14)-C(15)	1.57(3)
C(15)-C(16)	1.54(3)
C(16)-C(17)	1.52(3)
C(17)-C(18)	1.54(3)
C(18)-C(19)	1.58(3)
C(18)-C(24)	1.60(3)
C(19)-C(20)	1.53(3)
C(19)-C(21)	1.56(3)
C(21)-C(22)	1.60(4)
C(22)-C(23)	1.55(3)
C(23)-C(24)	1.55(4)
C(24)-C(25)	1.45(3)
C(24)-C(26)	1.45(3)
C(1')-C(2')	1.40
C(1')-C(6')	1.40
C(2')-C(3')	1.40
C(3')-C(4')	1.39
C(3')-Br(2)	1.853(12)

C(4')-C(5')	1.39
C(5')-C(6')	1.40
C(6')-C(7')	1.47
C(7')-O(1')	1.21(2)
C(7')-O(2')	1.29(2)
O(2')-C(8')	1.48(3)
O(3')-C(26')	1.21(3)
O(4')-C(26')	1.26(4)
O(4')-C(27')	1.44(3)
C(8')-C(9')	1.53(4)
C(9')-C(11')	1.48(4)
C(9')-C(10')	1.49(4)
C(9')-C(15')	1.61(4)
C(11')-C(12')	1.42(4)
C(12')-C(13')	1.57(5)
C(13')-C(14')	1.38(4)
C(14')-C(15')	1.37(4)
C(14')-C(19')	1.58(4)
C(15')-C(16')	1.53(4)
C(16')-C(17')	1.59(4)
C(17')-C(18')	1.44(4)
C(18')-C(19')	1.53(3)
C(18')-C(24')	1.62(4)
C(19')-C(20')	1.52(4)
C(19')-C(21')	1.53(4)
C(21')-C(22')	1.59(5)
C(22')-C(23')	1.55(4)
C(23')-C(24')	1.45(4)
C(24')-C(26')	1.52(4)
C(24')-C(25')	1.55(4)
C(6)-C(1)-C(2)	120.0
C(3)-C(2)-C(1)	120.0
C(4)-C(3)-C(2)	120.0
C(4)-C(3)-Br(1)	119.2(8)
C(2)-C(3)-Br(1)	120.8(8)
C(3)-C(4)-C(5)	120.0
C(6)-C(5)-C(4)	120.0
C(5)-C(6)-C(1)	120.0
C(5)-C(6)-C(7)	120.1
C(1)-C(6)-C(7)	119.9
O(1)-C(7)-O(2)	122.2(14)
O(1)-C(7)-C(6)	122.7(12)
O(2)-C(7)-C(6)	115.0(10)
C(7)-O(2)-C(8)	118(2)
C(26)-O(4)-C(27)	115(2)
O(2)-C(8)-C(9)	107(2)
C(8)-C(9)-C(15)	115(2)
C(8)-C(9)-C(10)	110(2)
C(15)-C(9)-C(10)	108(2)
C(8)-C(9)-C(11)	112(2)
C(15)-C(9)-C(11)	110(2)
C(10)-C(9)-C(11)	101(2)
C(12)-C(11)-C(9)	107(2)
C(11)-C(12)-C(13)	115(3)
C(14)-C(13)-C(12)	121(3)
C(13)-C(14)-C(19)	120(2)
C(13)-C(14)-C(15)	122(2)
C(19)-C(14)-C(15)	118(2)

C(9)-C(15)-C(16)	116(2)
C(9)-C(15)-C(14)	113(2)
C(16)-C(15)-C(14)	106(2)
C(17)-C(16)-C(15)	114(2)
C(16)-C(17)-C(18)	114(2)
C(17)-C(18)-C(19)	111(2)
C(17)-C(18)-C(24)	115(2)
C(19)-C(18)-C(24)	118(2)
C(14)-C(19)-C(20)	110(2)
C(14)-C(19)-C(21)	107(2)
C(20)-C(19)-C(21)	109(2)
C(14)-C(19)-C(18)	111(2)
C(20)-C(19)-C(18)	111(2)
C(21)-C(19)-C(18)	108(2)
C(19)-C(21)-C(22)	112(2)
C(23)-C(22)-C(21)	109(2)
C(24)-C(23)-C(22)	113(2)
C(25)-C(24)-C(26)	106(2)
C(25)-C(24)-C(23)	108(2)
C(26)-C(24)-C(23)	115(2)
C(25)-C(24)-C(18)	107(2)
C(26)-C(24)-C(18)	115(2)
C(23)-C(24)-C(18)	106(2)
O(3)-C(26)-O(4)	123(3)
O(3)-C(26)-C(24)	126(2)
O(4)-C(26)-C(24)	111(2)
C(2')-C(1')-C(6')	120.0
C(1')-C(2')-C(3')	120.0
C(4')-C(3')-C(2')	120.0
C(4')-C(3')-Br(2)	119.2(9)
C(2')-C(3')-Br(2)	120.8(9)
C(3')-C(4')-C(5')	120.0
C(4')-C(5')-C(6')	120.0
C(5')-C(6')-C(1')	120.0
C(5')-C(6')-C(7')	120.1
C(1')-C(6')-C(7')	119.9
O(1')-C(7')-O(2')	124(2)
O(1')-C(7')-C(6')	122.0(14)
O(2')-C(7')-C(6')	113.7(11)
C(7')-O(2')-C(8')	117(2)
C(26')-O(4')-C(27')	118(3)
O(2')-C(8')-C(9')	105(2)
C(11')-C(9')-C(10')	112(3)
C(11')-C(9')-C(8')	109(3)
C(10')-C(9')-C(8')	111(3)
C(11')-C(9')-C(15')	108(3)
C(10')-C(9')-C(15')	113(3)
C(8')-C(9')-C(15')	104(3)
C(12')-C(11')-C(9')	117(3)
C(11')-C(12')-C(13')	115(3)
C(14')-C(13')-C(12')	120(3)
C(15')-C(14')-C(13')	121(3)
C(15')-C(14')-C(19')	120(3)
C(13')-C(14')-C(19')	119(3)
C(14')-C(15')-C(16')	112(3)
C(14')-C(15')-C(9')	119(3)
C(16')-C(15')-C(9')	113(3)
C(15')-C(16')-C(17')	109(3)

C(18')-C(17')-C(16')	112(3)
C(17')-C(18')-C(19')	116(2)
C(17')-C(18')-C(24')	114(2)
C(19')-C(18')-C(24')	110(2)
C(20')-C(19')-C(21')	111(2)
C(20')-C(19')-C(18')	113(2)
C(21')-C(19')-C(18')	112(2)
C(20')-C(19')-C(14')	108(2)
C(21')-C(19')-C(14')	109(2)
C(18')-C(19')-C(14')	103(2)
C(19')-C(21')-C(22')	115(3)
C(23')-C(22')-C(21')	104(3)
C(24')-C(23')-C(22')	116(3)
C(23')-C(24')-C(26')	112(3)
C(23')-C(24')-C(25')	111(3)
C(26')-C(24')-C(25')	105(2)
C(23')-C(24')-C(18')	112(3)
C(26')-C(24')-C(18')	113(3)
C(25')-C(24')-C(18')	104(2)
O(3')-C(26')-O(4')	119(3)
O(3')-C(26')-C(24')	125(3)
O(4')-C(26')-C(24')	115(3)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for **41**&N. The factor exponent takes the form: $-2\pi^2[(ha^*)^2U_{11}+...+ 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
O(3)	151(20)	80(16)	179(23)	-42(16)	115(19)	-29(15)
O(4)	80(12)	51(13)	82(12)	-28(10)	29(10)	-17(9)
Br(1)	85(2)	32(2)	132(3)	-13(2)	37(2)	0(2)
O(3')	193(28)	78(17)	143(23)	-35(16)	-49(21)	40(18)
O(4')	99(15)	47(13)	96(15)	7(10)	39(12)	21(10)
Br(2)	132(3)	39(2)	134(3)	-19(2)	39(2)	1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **41**

	x	y	z	U(eq)
H(1)	8713(19)	2382(7)	1391(10)	88
H(2)	8887(20)	3567(9)	1912(10)	64
H(4)	7204(21)	4112(7)	-1431(14)	58
H(5)	7030(17)	2927(8)	-1952(8)	69
H(8C)	8850(27)	514(13)	-209(22)	77
H(8D)	7331(27)	510(13)	-747(22)	77
H(10D)	9074(27)	229(16)	2675(23)	119
H(10E)	9093(27)	968(16)	2142(23)	119
H(10F)	9933(27)	363(16)	1916(23)	119
H(11C)	6773(24)	265(15)	1950(22)	80
H(11D)	6829(24)	1001(15)	1409(22)	80
H(12C)	5557(32)	649(18)	-327(28)	116
H(12D)	4866(32)	403(18)	518(28)	116
H(13B)	4818(25)	-602(13)	-670(21)	69
H(15B)	8242(22)	-696(12)	1638(20)	51

H(16C)	9359(20)	-556(11)	-66(17)	46
H(17B)	9772(26)	-1736(15)	1013(24)	87
H(17C)	9138(26)	-1790(15)	-315(24)	87
H(18B)	7925(19)	-1892(11)	1312(18)	44
H(20D)	6224(26)	-2107(15)	-1672(21)	112
H(20E)	6952(26)	-1400(15)	-1637(21)	112
H(20F)	5463(26)	-1401(15)	-1850(21)	112
H(21C)	4597(25)	-1712(14)	-355(24)	83
H(21D)	5502(25)	-1807(14)	915(24)	83
H(22C)	4639(23)	-2930(14)	164(22)	83
H(22D)	5206(23)	-2852(14)	-830(22)	83
H(23C)	6779(24)	-2942(16)	1523(23)	86
H(23D)	6589(24)	-3565(16)	668(23)	86
H(25D)	8990(25)	-2884(16)	2031(23)	110
H(25E)	9727(25)	-2880(16)	1152(23)	110
H(25F)	8991(25)	-3551(16)	1306(23)	110
H(27D)	6392(31)	-4251(21)	-2313(28)	152
H(27E)	7889(31)	-4131(21)	-1911(28)	152
H(27F)	6947(31)	-3573(21)	-2675(28)	152
H(1')	6736(20)	3688(9)	3680(12)	90
H(2')	6486(23)	2512(11)	3102(11)	96
H(4')	8128(22)	1901(8)	6417(16)	61
H(5')	8377(19)	3077(10)	6995(9)	86
H(8A)	8603(30)	5461(15)	5789(25)	93
H(8B)	7105(30)	5601(15)	5434(25)	93
H(10A)	9711(29)	5635(17)	4430(26)	138
H(10B)	8952(29)	5749(17)	3128(26)	138
H(10C)	8911(29)	5022(17)	3690(26)	138
H(11A)	6600(31)	5788(20)	2459(29)	124
H(11B)	6530(31)	5076(20)	3043(29)	124
H(12A)	5160(34)	5441(19)	3765(31)	129
H(12B)	4728(34)	5816(19)	2579(31)	129
H(13A)	4769(31)	6697(17)	4053(26)	106
H(15A)	7988(29)	6777(17)	3704(27)	102
H(16A)	9699(29)	6567(16)	5393(25)	103
H(16B)	8742(29)	6654(16)	6077(25)	103
H(17A)	9804(31)	7854(18)	5806(26)	109
H(18A)	7934(22)	7835(12)	3836(21)	60
H(20A)	7243(27)	7452(15)	6348(22)	117
H(20B)	5733(27)	7463(15)	5798(22)	117
H(20C)	6508(27)	8165(15)	6052(22)	117
H(21A)	4697(28)	7862(15)	3830(25)	98
H(21B)	5489(28)	7931(15)	3003(25)	98
H(22A)	4798(33)	9075(19)	3287(32)	127
H(22B)	5421(33)	8996(19)	4623(32)	127
H(23A)	6844(28)	8980(18)	3083(27)	103
H(23B)	6891(28)	9595(18)	3919(27)	103
H(25A)	9939(23)	8784(14)	5001(21)	97
H(25B)	9298(23)	9489(14)	4466(21)	97
H(25C)	9152(23)	8829(14)	3693(21)	97
H(27A)	7491(27)	10409(20)	6927(25)	130
H(27B)	8845(27)	10043(20)	7355(25)	130
H(27C)	7640(27)	9671(20)	7499(25)	130

Experimental $R1=(\sum||F_o|-|F_c||/\sum|F_o|)$, $wR2=\sum w(F_o^2-F_c^2)^2/\sum w[(F_o^2)^2]_{1/2}$, $S=[\sum w(F_o^2-F_c^2)^2/(n-p)]^{1/2}$
Phenyl rings were treated as regular hexagons of D_{6h} symmetry with C-C = 1.395 Å and C-C-C = 120°. Unit cell dimensions and standard deviations were

obtained by least squares fit to 16 reflections (14<2 <26°).

References

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h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Res(A)
-1	0	1	187.31	21.98	4.58	0.014	9.52
1	9	0	11224.33	5492.79	4.42	0.228	2.09
-3	14	5	-93.15	273.07	4.08	0.051	1.18
-2	5	1	649.08	247.09	3.91	0.048	3.14
1	9	1	28888.57	17067.81	3.29	0.401	2.03
-1	9	1	60034.12	36897.02	3.05	0.590	2.08
-2	2	7	331.36	109.37	3.00	0.032	1.75
1	2	11	-204.61	2.23	2.98	0.005	1.02
1	5	0	18363.22	11442.89	2.93	0.328	3.60
-6	10	10	-156.03	101.36	2.93	0.031	1.00
0	12	3	1498.62	855.68	2.91	0.090	1.48
1	0	0	700.98	376.68	2.87	0.060	10.35
3	0	5	-71.53	57.43	2.86	0.023	1.69
2	1	8	90.16	356.22	2.84	0.058	1.30
11	0	0	340.68	27.89	2.83	0.016	0.94
-4	4	10	-149.05	14.52	2.83	0.012	1.20
9	3	4	278.13	16.88	2.81	0.013	0.95
-1	16	3	296.76	714.14	2.81	0.082	1.15
-1	10	2	162.69	11.54	2.77	0.010	1.83
11	3	0	-261.83	1.14	2.77	0.003	0.93
-7	5	5	-76.52	111.15	2.77	0.032	1.40
5	15	4	-148.71	87.60	2.75	0.029	0.97
1	15	3	523.32	215.94	2.75	0.045	1.19
1	1	8	9.98	216.98	2.71	0.045	1.38
0	1	5	52.18	196.97	2.69	0.043	2.33
8	8	1	239.20	21.71	2.69	0.014	1.10
5	13	5	428.84	141.58	2.69	0.037	0.99
2	5	5	-18.30	135.38	2.68	0.036	1.71
-10	3	10	-224.90	9.82	2.68	0.010	0.94
1	4	0	4661.34	2964.77	2.67	0.167	4.35
-5	12	8	-68.53	164.16	2.67	0.039	1.07
1	5	8	-3.66	194.00	2.67	0.043	1.30
-11	1	5	144.39	507.13	2.67	0.069	1.00
8	2	4	-180.65	13.12	2.66	0.011	1.05
2	2	8	-28.94	161.35	2.66	0.039	1.29
-8	8	8	-124.43	95.62	2.61	0.030	1.07
0	8	4	392.67	175.69	2.61	0.041	1.86
-4	10	10	-161.36	24.94	2.59	0.015	1.04
0	14	3	622.70	319.96	2.56	0.055	1.29
4	14	0	1026.68	566.40	2.56	0.073	1.21
5	6	5	-57.89	131.83	2.55	0.035	1.24
-9	8	4	-64.87	135.79	2.55	0.036	1.09
1	14	1	4230.83	2715.46	2.55	0.160	1.34
3	3	6	110.45	351.03	2.54	0.058	1.46
-7	2	10	9.65	236.98	2.54	0.047	1.11
0	12	0	1768.25	1080.90	2.54	0.101	1.60
9	3	2	-168.34	19.40	2.52	0.014	1.05
-2	12	5	1064.94	608.80	2.52	0.076	1.34
-1	16	5	109.46	403.03	2.52	0.062	1.08
-10	9	6	-186.64	32.39	2.51	0.017	0.96

X-Ray data of compound **42**

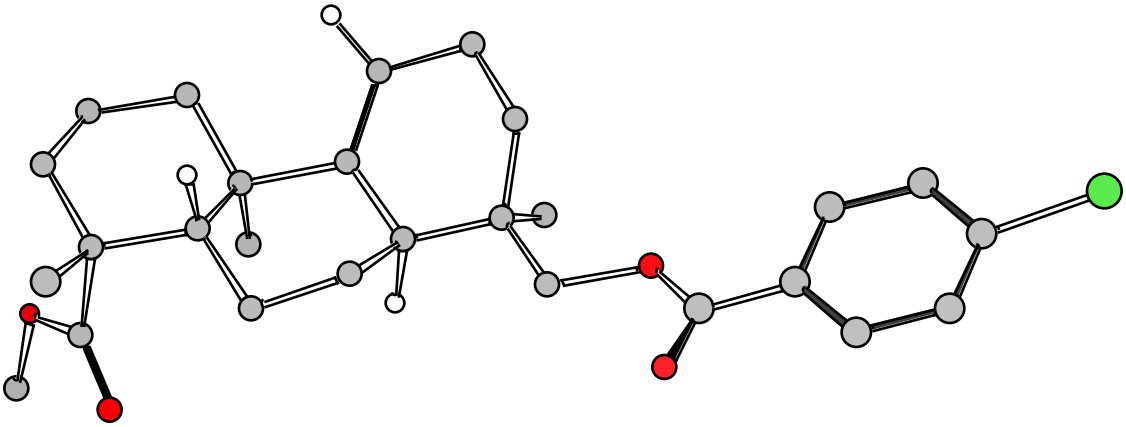

Table 1. Crystal data and structure refinement for **42**

Empirical formula	$C_{27}\ H_{34.50}BrO_4$
Formula weight	502.96
Temperature	22 C
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 10.801(6)Å alpha = 90° b = 22.481(11)Å beta = 109.45(4)° c = 10.819(5)Å gamma = 90°
Crystal size	0.80 x 0.50 x 0.06 mm
Density (calculated)	1.349 Mg/m^3
Absorption coefficient	1.689 mm^{-1}
Volume, Z, F(000)	2477(2) $Å^3$, 4, 1054
θ range for data collection	1.81 to 22.50°
Limiting indices	$-11 \le h \le 10$, $0 \le k < 21$, $-10 \le l \le 10$
Reflections collected	2728, 1179 observed [I>2σ(I)]
Independent reflections	2579 (R_{int} = 0.0747)
Absorption correction None	
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2578 / 1 / 257
Goodness-of-fit on F^2	1.035
Final R indices [I>2σ(I)]	R1 = 0.1126, wR2 = 0.2667
R indices (all data)	R1 = 0.2329, wR2 = 0.3694
Absolute structure parameter	0.00(6)
Largest diff. peak and hole	0.625 and -0.627 $eÅ^3$
Scan speed, range, type	5°/minute, 0.6°, Wyckoff
Background range, % time	0.6°, 25% each side

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for **42**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

x	y	z	U(eq)

C(1)	-4359(19)	-2240(9)	-3220(19)	75(12)
C(2)	-3091(20)	-2403(9)	-3148(21)	82(13)
C(3)	-2423(15)	-2074(11)	-3820(21)	41(10)
C(4)	-3023(19)	-1583(11)	-4566(22)	74(12)
C(5)	-4291(19)	-1419(9)	-4638(19)	60(11)
C(6)	-4959(15)	-1748(8)	-3965(16)	35(9)
C(7)	-6309(17)	-1589(13)	-4075(26)	53(11)
Br(1)	-675(5)	-2235(3)	-3662(5)	99(2)
O(1)	-6805(25)	-1163(12)	-4847(24)	64(8)
O(2)	-6846(29)	-1821(14)	-3383(28)	93(10)
O(3)	-14596(22)	-191(16)	-6693(24)	83(9)
O(4)	-15258(20)	631(10)	-7812(21)	55(7)
C(8)	-8113(36)	-947(17)	-4814(36)	56(11)
C(9)	-8475(36)	-399(17)	-5678(34)	51(11)
C(10)	-8369(35)	-484(16)	-6955(32)	54(11)
C(11)	-7453(34)	88(16)	-5041(33)	54(11)
C(12)	-7697(34)	666(16)	-5729(36)	61(11)
C(13)	-9105(35)	830(19)	-6190(35)	67(12)
C(14)	-10088(31)	429(15)	-6317(29)	36(9)
C(15)	-9801(33)	-199(20)	-5809(32)	51(10)
C(16)	-10307(25)	-260(15)	-4665(26)	32(8)
C(17)	-11794(35)	-127(18)	-5067(37)	69(11)
C(18)	-12019(31)	506(15)	-5530(31)	46(9)
C(19)	-11587(30)	663(16)	-6633(30)	36(9)
C(20)	-12310(32)	330(16)	-7958(30)	54(10)
C(21)	-11675(32)	1324(15)	-6890(35)	54(10)
C(22)	-13040(30)	1557(16)	-7173(31)	46(9)
C(23)	-13583(34)	1381(16)	-6137(36)	60(11)
C(24)	-13460(32)	742(16)	-5770(31)	45(9)
C(25)	-13813(40)	665(20)	-4496(39)	87(13)
C(26)	-14501(39)	327(21)	-6854(40)	49(11)
C(27)	-16213(41)	293(20)	-8779(39)	87(14)
C(1')	-10498(21)	2(8)	-11208(21)	60(11)
C(2')	-11807(22)	128(9)	-11368(23)	81(13)
C(3')	-12670(17)	-332(12)	-11360(21)	65(12)
C(4')	-12225(21)	-919(11)	-11192(23)	78(13)
C(5')	-10916(22)	-1045(8)	-11032(22)	69(12)
C(6')	-10053(17)	-585(9)	-11040(16)	65(13)
C(7')	-8687(19)	-719(14)	-10915(28)	62(12)
Br(2)	-14430(4)	-182(3)	-11569(5)	103(2)
O(1')	-8284(25)	-1227(12)	-10352(24)	62(8)
O(2')	-7976(26)	-324(13)	-11199(25)	84(9)
O(3')	-1068(22)	-2486(14)	-10054(23)	80(10)
O(4')	-445(23)	-3407(14)	-9330(21)	72(8)
C(8')	-6860(39)	-1338(20)	-10035(41)	69(13)
C(9')	-6619(33)	-1947(16)	-9411(31)	41(10)
C(10')	-7605(40)	-2403(19)	-10277(39)	81(14)
C(11')	-6778(37)	-1936(17)	-8042(33)	54(11)
C(12')	-6533(42)	-2497(19)	-7386(43)	88(15)
C(13')	-5361(32)	-2842(16)	-7555(32)	54(10)
C(14')	-4756(41)	-2677(19)	-8398(39)	66(13)
C(15')	-5170(35)	-2170(20)	-9266(34)	56(11)
C(16')	-4155(39)	-1651(21)	-8846(43)	89(14)
C(17')	-2814(37)	-1912(17)	-8821(38)	66(11)
C(18')	-2454(35)	-2483(17)	-8035(37)	63(11)
C(19')	-3505(34)	-2953(16)	-8371(32)	44(10)
C(20')	-3739(31)	-3239(16)	-9773(29)	50(10)
C(21')	-3031(33)	-3461(16)	-7390(33)	53(10)

C(22')	-1607(30)	-3724(16)	-7224(34)	49(10)
C(23')	-695(34)	-3245(17)	-6982(32)	55(10)
C(24')	-1012(34)	-2712(16)	-7885(34)	52(10)
C(25')	18(37)	-2203(20)	-7376(37)	80(13)
C(26')	-867(38)	-2861(19)	-9073(41)	55(11)
C(27')	-271(38)	-3534(18)	-10485(38)	78(13)

Table 3. Bond lengths [Å] and angles [°] for **42**

C(1)-C(6)	1.39
C(1)-C(2)	1.40
C(2)-C(3)	1.40
C(3)-C(4)	1.39
C(3)-Br(1)	1.87(2)
C(4)-C(5)	1.39
C(5)-C(6)	1.39
C(6)-C(7)	1.47
C(7)-O(2)	1.21(3)
C(7)-O(1)	1.27(3)
O(1)-C(8)	1.51(4)
O(3)-C(26)	1.19(5)
O(4)-C(26)	1.28(4)
O(4)-C(27)	1.42(4)
C(8)-C(9)	1.52(5)
C(9)-C(10)	1.44(5)
C(9)-C(15)	1.46(5)
C(9)-C(11)	1.55(4)
C(11)-C(12)	1.48(5)
C(12)-C(13)	1.48(4)
C(13)-C(14)	1.36(4)
C(14)-C(15)	1.51(5)
C(14)-C(19)	1.63(4)
C(15)-C(16)	1.52(4)
C(16)-C(17)	1.55(4)
C(17)-C(18)	1.50(5)
C(18)-C(19)	1.46(4)
C(18)-C(24)	1.58(4)
C(19)-C(21)	1.51(5)
C(19)-C(20)	1.58(4)
C(21)-C(22)	1.50(4)
C(22)-C(23)	1.48(4)
C(23)-C(24)	1.48(5)
C(24)-C(25)	1.56(5)
C(24)-C(26)	1.62(5)
C(1')-C(2')	1.39
C(1')-C(6')	1.40
C(2')-C(3')	1.40
C(3')-C(4')	1.39
C(3')-Br(2)	1.87(2)
C(4')-C(5')	1.39
C(5')-C(6')	1.39
C(6')-C(7')	1.47
C(7')-O(2')	1.28(4)
C(7')-O(1')	1.30(4)
O(1')-C(8')	1.48(4)
O(3')-C(26')	1.32(4)
O(4')-C(27')	1.35(4)

O(4')-C(26')	1.37(4)
C(8')-C(9')	1.51(5)
C(9')-C(11')	1.55(5)
C(9')-C(10')	1.55(5)
C(9')-C(15')	1.60(5)
C(11')-C(12')	1.43(5)
C(12')-C(13')	1.55(5)
C(13')-C(14')	1.34(5)
C(14')-C(15')	1.45(5)
C(14')-C(19')	1.48(5)
C(15')-C(16')	1.56(5)
C(16')-C(17')	1.55(5)
C(17')-C(18')	1.52(5)
C(18')-C(19')	1.50(5)
C(18')-C(24')	1.60(5)
C(19')-C(21')	1.53(5)
C(19')-C(20')	1.59(4)
C(21')-C(22')	1.60(4)
C(22')-C(23')	1.42(4)
C(23')-C(24')	1.51(5)
C(24')-C(26')	1.39(5)
C(24')-C(25')	1.56(5)
C(6)-C(1)-C(2)	120.0
C(1)-C(2)-C(3)	120.0
C(4)-C(3)-C(2)	120.0
C(4)-C(3)-Br(1)	117.8(13)
C(2)-C(3)-Br(1)	122.1(13)
C(3)-C(4)-C(5)	120.0
C(4)-C(5)-C(6)	120.0
C(5)-C(6)-C(1)	120.0
C(5)-C(6)-C(7)	120.1
C(1)-C(6)-C(7)	119.9
O(2)-C(7)-O(1)	123(2)
O(2)-C(7)-C(6)	121(2)
O(1)-C(7)-C(6)	115(2)
C(7)-O(1)-C(8)	115(2)
C(26)-O(4)-C(27)	115(3)
O(1)-C(8)-C(9)	107(3)
C(10)-C(9)-C(15)	110(3)
C(10)-C(9)-C(8)	114(3)
C(15)-C(9)-C(8)	111(3)
C(10)-C(9)-C(11)	104(3)
C(15)-C(9)-C(11)	111(3)
C(8)-C(9)-C(11)	108(3)
C(12)-C(11)-C(9)	115(3)
C(11)-C(12)-C(13)	112(3)
C(14)-C(13)-C(12)	123(4)
C(13)-C(14)-C(15)	122(3)
C(13)-C(14)-C(19)	120(3)
C(15)-C(14)-C(19)	117(3)
C(9)-C(15)-C(14)	114(3)
C(9)-C(15)-C(16)	120(3)
C(14)-C(15)-C(16)	107(3)
C(15)-C(16)-C(17)	112(3)
C(18)-C(17)-C(16)	108(3)
C(19)-C(18)-C(17)	116(3)
C(19)-C(18)-C(24)	110(3)
C(17)-C(18)-C(24)	115(3)

C(18)-C(19)-C(21)	112(3)
C(18)-C(19)-C(20)	116(3)
C(21)-C(19)-C(20)	109(3)
C(18)-C(19)-C(14)	108(3)
C(21)-C(19)-C(14)	111(3)
C(20)-C(19)-C(14)	101(2)
C(22)-C(21)-C(19)	112(3)
C(23)-C(22)-C(21)	111(3)
C(22)-C(23)-C(24)	116(3)
C(23)-C(24)-C(25)	109(3)
C(23)-C(24)-C(18)	111(3)
C(25)-C(24)-C(18)	110(3)
C(23)-C(24)-C(26)	113(3)
C(25)-C(24)-C(26)	105(3)
C(18)-C(24)-C(26)	109(3)
O(3)-C(26)-O(4)	125(4)
O(3)-C(26)-C(24)	122(4)
O(4)-C(26)-C(24)	112(3)
C(2')-C(1')-C(6')	120.0
C(1')-C(2')-C(3')	120.0
C(4')-C(3')-C(2')	120.0
C(4')-C(3')-Br(2)	118.7(14)
C(2')-C(3')-Br(2)	121.3(14)
C(3')-C(4')-C(5')	120.0
C(6')-C(5')-C(4')	120.0
C(5')-C(6')-C(1')	120.0
C(5')-C(6')-C(7')	120.1
C(1')-C(6')-C(7')	119.9
O(2')-C(7')-O(1')	126(2)
O(2')-C(7')-C(6')	120(2)
O(1')-C(7')-C(6')	113(2)
C(7')-O(1')-C(8')	115(3)
C(27')-O(4')-C(26')	122(3)
O(1')-C(8')-C(9')	105(3)
C(8')-C(9')-C(11')	111(3)
C(8')-C(9')-C(10')	110(3)
C(11')-C(9')-C(10')	109(3)
C(8')-C(9')-C(15')	110(3)
C(11')-C(9')-C(15')	109(3)
C(10')-C(9')-C(15')	108(3)
C(12')-C(11')-C(9')	114(3)
C(11')-C(12')-C(13')	114(4)
C(14')-C(13')-C(12')	123(4)
C(13')-C(14')-C(15')	123(4)
C(13')-C(14')-C(19')	121(4)
C(15')-C(14')-C(19')	115(4)
C(14')-C(15')-C(16')	111(3)
C(14')-C(15')-C(9')	113(3)
C(16')-C(15')-C(9')	111(3)
C(17')-C(16')-C(15')	106(3)
C(18')-C(17')-C(16')	113(3)
C(19')-C(18')-C(17')	115(3)
C(19')-C(18')-C(24')	116(3)
C(17')-C(18')-C(24')	113(3)
C(14')-C(19')-C(18')	109(3)
C(14')-C(19')-C(21')	115(3)
C(18')-C(19')-C(21')	108(3)
C(14')-C(19')-C(20')	107(3)

C(18')-C(19')-C(20')	113(3)
C(21')-C(19')-C(20')	106(3)
C(19')-C(21')-C(22')	116(3)
C(23')-C(22')-C(21')	109(3)
C(22')-C(23')-C(24')	119(3)
C(26')-C(24')-C(23')	110(3)
C(26')-C(24')-C(25')	103(3)
C(23')-C(24')-C(25')	112(3)
C(26')-C(24')-C(18')	113(3)
C(23')-C(24')-C(18')	109(3)
C(25')-C(24')-C(18')	111(3)
O(3')-C(26')-O(4')	113(3)
O(3')-C(26')-C(24')	124(4)
O(4')-C(26')-C(24')	124(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for **42**. The factor exponent takes the form: $-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + 2hka*b*U_{12}]

	U11	U22	U33	U23	U13	U12
Br(1)	69(3)	140(5)	90(4)	5(4)	29(3)	33(4)
O(3)	42(15)	106(27)	77(18)	33(19)	-12(13)	13(17)
O(4)	37(14)	58(17)	47(15)	14(14)	-17(12)	3(14)
Br(2)	50(3)	174(6)	83(3)	18(4)	18(2)	44(4)
O(3')	46(15)	150(29)	44(16)	-5(18)	15(13)	-13(17)
O(4')	70(17)	127(26)	35(14)	3(16)	37(13)	23(17)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **42**.

	x	y	z	U(eq)
H(1)	-4818(26)	-2466(12)	-2757(28)	90
H(2)	-2678(27)	-2742(12)	-2635(30)	98
H(4)	-2563(26)	-1356(14)	-5029(31)	88
H(5)	-4704(27)	-1080(11)	-5151(28)	73
H(8A)	-8054(36)	-848(17)	-3923(36)	67
H(8B)	-8774(36)	-1253(17)	-5138(36)	67
H(10A)	-7496(35)	-612(16)	-6866(32)	81
H(10B)	-8553(35)	-116(16)	-7431(32)	81
H(10C)	-8988(35)	-781(16)	-7422(32)	81
H(11A)	-7435(34)	149(16)	-4148(33)	65
H(11B)	-6592(34)	-54(16)	-5001(33)	65
H(12A)	-7389(34)	647(16)	-6474(36)	74
H(12B)	-7199(34)	973(16)	-5141(36)	74
H(13A)	-9330(35)	1225(19)	-6405(35)	81
H(15A)	-10384(33)	-449(20)	-6501(32)	62
H(16A)	-9832(25)	13(15)	-3975(26)	38
H(16B)	-10142(25)	-661(15)	-4318(26)	38
H(17A)	-12281(35)	-394(18)	-5764(37)	83
H(17B)	-12094(35)	-185(18)	-4325(37)	83
H(18A)	-11462(31)	743(15)	-4794(31)	55
H(20A)	-11960(32)	460(16)	-8619(30)	81

H(20B)	-13232(32)	417(16)	-8232(30)	81
H(20C)	-12179(32)	-91(16)	-7830(30)	81
H(21A)	-11088(32)	1529(15)	-6131(35)	64
H(21B)	-11386(32)	1409(15)	-7630(35)	64
H(22A)	-13030(30)	1988(16)	-7231(31)	55
H(22B)	-13600(30)	1404(16)	-8011(31)	55
H(23A)	-14507(34)	1486(16)	-6426(36)	72
H(23B)	-13147(34)	1613(16)	-5357(36)	72
H(25A)	-13734(40)	253(20)	-4245(39)	130
H(25B)	-14699(40)	795(20)	-4653(39)	130
H(25C)	-13225(40)	899(20)	-3806(39)	130
H(27A)	-16733(41)	554(20)	-9457(39)	130
H(27B)	-16771(41)	92(20)	-8384(39)	130
H(27C)	-15781(41)	5(20)	-9150(39)	130
H(1A)	-9904(27)	318(9)	-11213(33)	72
H(2A)	-12113(30)	532(10)	-11484(35)	97
H(4A)	-12819(27)	-1236(13)	-11186(35)	94
H(5A)	-10610(30)	-1449(8)	-10916(33)	83
H(8C)	-6351(39)	-1039(20)	-9432(41)	83
H(8D)	-6622(39)	-1330(20)	-10824(41)	83
H(10D)	-7442(40)	-2787(19)	-9868(39)	121
H(10E)	-7499(40)	-2424(19)	-11122(39)	121
H(10F)	-8484(40)	-2280(19)	-10377(39)	121
H(11C)	-6178(37)	-1644(17)	-7503(33)	64
H(11D)	-7664(37)	-1810(17)	-8138(33)	64
H(12C)	-7316(42)	-2740(19)	-7715(43)	105
H(12D)	-6367(42)	-2431(19)	-6458(43)	105
H(13B)	-5063(32)	-3180(16)	-7048(32)	65
H(16C)	-4414(39)	-1325(21)	-9467(43)	107
H(16D)	-4096(39)	-1504(21)	-7985(43)	107
H(17C)	-2847(37)	-1989(17)	-9714(38)	79
H(17D)	-2132(37)	-1619(17)	-8449(38)	79
H(18B)	-2385(35)	-2362(17)	-7144(37)	76
H(20D)	-2941(31)	-3423(16)	-9790(29)	75
H(20E)	-3997(31)	-2934(16)	-10431(29)	75
H(20F)	-4419(31)	-3534(16)	-9946(29)	75
H(21C)	-3663(33)	-3783(16)	-7654(33)	64
H(21D)	-3029(33)	-3322(16)	-6540(33)	64
H(22C)	-1344(30)	-4003(16)	-6498(34)	59
H(22D)	-1628(30)	-3934(16)	-8014(34)	59
H(23C)	86(34)	-3260(17)	-6278(32)	66
H(25D)	4(37)	-2066(20)	-6540(37)	120
H(25E)	-196(37)	-1879(20)	-7989(37)	120
H(25F)	877(37)	-2350(20)	-7286(37)	120
H(26A)	-1783(38)	-2993(19)	-9417(41)	66
H(27D)	37(38)	-3936(18)	-10467(38)	117
H(27E)	364(38)	-3266(18)	-10620(38)	117
H(27F)	-1091(38)	-3491(18)	-11186(38)	117

Experimental

$R1=(\sum||F_o|-|F_c||/\sum|F_o|)$, $wR2=\sum w(F_o^2-F_c^2)^2/\sum w[(F_o^2)^2]^{1/2}$, $S=[\sum w(F_o^2-F_c^2)^2/(n-p)]^{1/2}$

Phenyl rings were treated as regular hexagons of D_{6h} symmetry with C-C = 1.395 Å and C-C-C = 120°. Unit cell dimensions and standard deviations were obtained by least squares fit to 14 reflections ($14<2\theta<22°$).

References

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	h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Res(A)
*	5	12	0	-452.12	47.83	3.54	0.031	1.38
	3	5	6	160.38	879.26	3.29	0.131	1.29
	6	10	2	4304.55	2244.03	3.27	0.209	1.23
	-1	20	3	563.02	4.83	3.14	0.010	1.07
	4	1	6	486.25	1385.35	3.03	0.165	1.23
	3	6	1	3304.76	1854.08	2.99	0.190	2.31
	-10	8	5	-493.07	2.62	2.96	0.007	0.99
	2	14	1	-172.32	189.38	2.95	0.061	1.49
	-3	19	2	-32.42	521.83	2.91	0.101	1.12
	4	17	0	102.37	759.92	2.90	0.122	1.17
	-1	16	2	-394.11	12.04	2.88	0.015	1.36
	2	2	6	168.91	696.11	2.87	0.117	1.46
	-7	13	3	499.89	38.35	2.86	0.027	1.15
	3	4	6	902.54	332.32	2.85	0.081	1.31
	-2	18	5	-479.42	1.34	2.84	0.005	1.08
	2	1	6	-114.31	269.56	2.81	0.073	1.47
	3	8	6	547.67	68.00	2.80	0.036	1.22
	2	5	3	607.38	1277.47	2.79	0.158	2.17
	5	7	5	650.03	143.59	2.76	0.053	1.16
	5	4	0	494.78	147.83	2.76	0.054	1.91
	1	9	6	131.37	625.21	2.73	0.111	1.34
	-6	1	2	235.45	662.42	2.67	0.114	1.79
	5	0	3	5551.73	3335.92	2.67	0.255	1.54
	2	15	5	506.72	53.90	2.66	0.032	1.13
	5	7	1	2366.40	1372.84	2.62	0.164	1.62
	-4	12	4	-332.69	9.64	2.62	0.014	1.43
	-2	7	3	472.60	191.80	2.60	0.061	2.33
	1	5	8	545.96	111.16	2.58	0.047	1.17
	5	1	5	-218.38	170.55	2.57	0.058	1.25
	3	11	3	1409.26	2636.11	2.56	0.227	1.46
	5	15	0	-29.00	435.15	2.55	0.092	1.21
	1	11	1	632.97	1241.97	2.54	0.156	1.94
	-8	3	5	320.75	911.52	2.52	0.133	1.27
	4	4	6	-272.98	158.75	2.52	0.056	1.21
	-4	19	3	-162.08	333.71	2.51	0.081	1.07
	-4	9	2	-6.82	294.67	2.50	0.076	1.82
	-10	10	4	-300.28	129.68	2.50	0.050	0.97
	1	16	4	498.19	86.97	2.49	0.041	1.20
	-8	12	1	999.79	420.93	2.47	0.091	1.08
	8	3	3	421.41	0.14	2.44	0.002	1.07
	-2	14	1	151.85	538.96	2.44	0.103	1.54
	1	10	4	-220.09	50.49	2.43	0.031	1.61
	-4	5	4	-141.61	88.85	2.42	0.042	1.98
	1	12	6	-64.83	331.86	2.38	0.081	1.21
	-2	19	1	-184.26	234.99	2.38	0.068	1.15
	4	17	2	440.18	41.94	2.36	0.029	1.11
	2	0	7	-276.39	72.57	2.36	0.038	1.29
	3	5	1	946.90	543.30	2.34	0.103	2.46
	3	10	6	-141.61	280.87	2.34	0.074	1.16
	2	7	8	-334.40	41.94	2.34	0.029	1.08