Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Hot Water–Promoted Cyclopropylcarbinyl Rearrangement Facilitates Construction of Homoallylic Alcohols

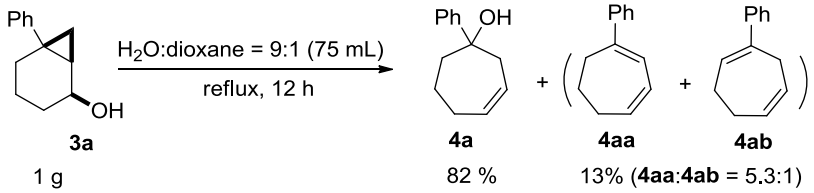
Pei-Fang Li, Cheng-Bo Yi, Jin Qu*

The State Key Laboratory of Elemento-Organic Chemistry, Collaborative Innovation Center of Chemical Science and Engineering (Tianjin), Nankai University, Tianjin 300071, China

Fax: +86-(022)-23503627 and E-mail: qujin@nankai.edu.cn

Table of Contents

1. Large scale rearrangement of substrate **3a** in the mixed solvent of water and 1,4-dioxane (v:v = 9:1).



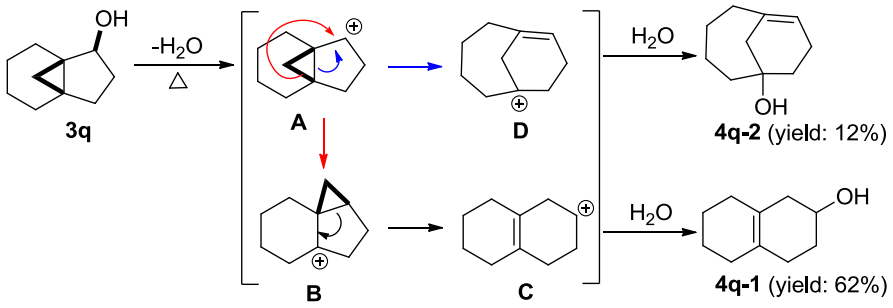
3a 1 g

4a 82 %

4aa **4ab** 13% (**4aa**:**4ab** = 5.3:1)

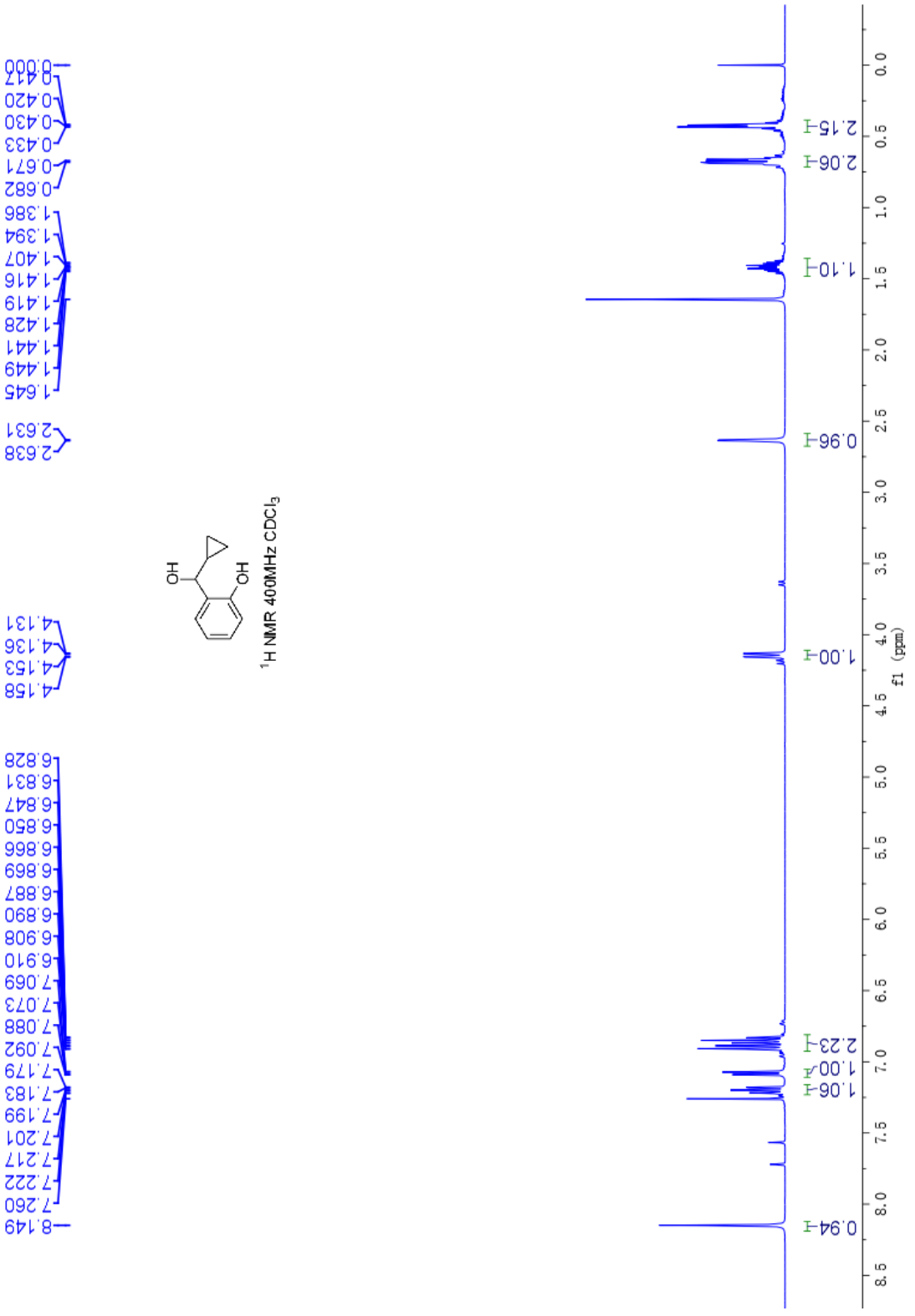
To a 200 mL round-bottom flask containing the substrate **3a** (1.00 g, 5.3 mmol) was added 1,4-dioxane (7.5 mL), and then to the solution was added H_2O (67.5 mL). The flask was fitted with a condenser, stirred vigorously under refluxing condition. After completion judged by TLC, the resulting solution was cooled to room temperature. The mixture was extracted with EtOAc (3 × 75 mL), washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel column chromatography (MTBE/hexane = 1:5) to afford the desired product **4a** (817 mg, 82%) as a colorless oil. The minor products of the reaction was the mixture of cyclic diene **4aa** and **4ab** (120 mg, 13%, **4aa**:**4ab** = 5.3:1).

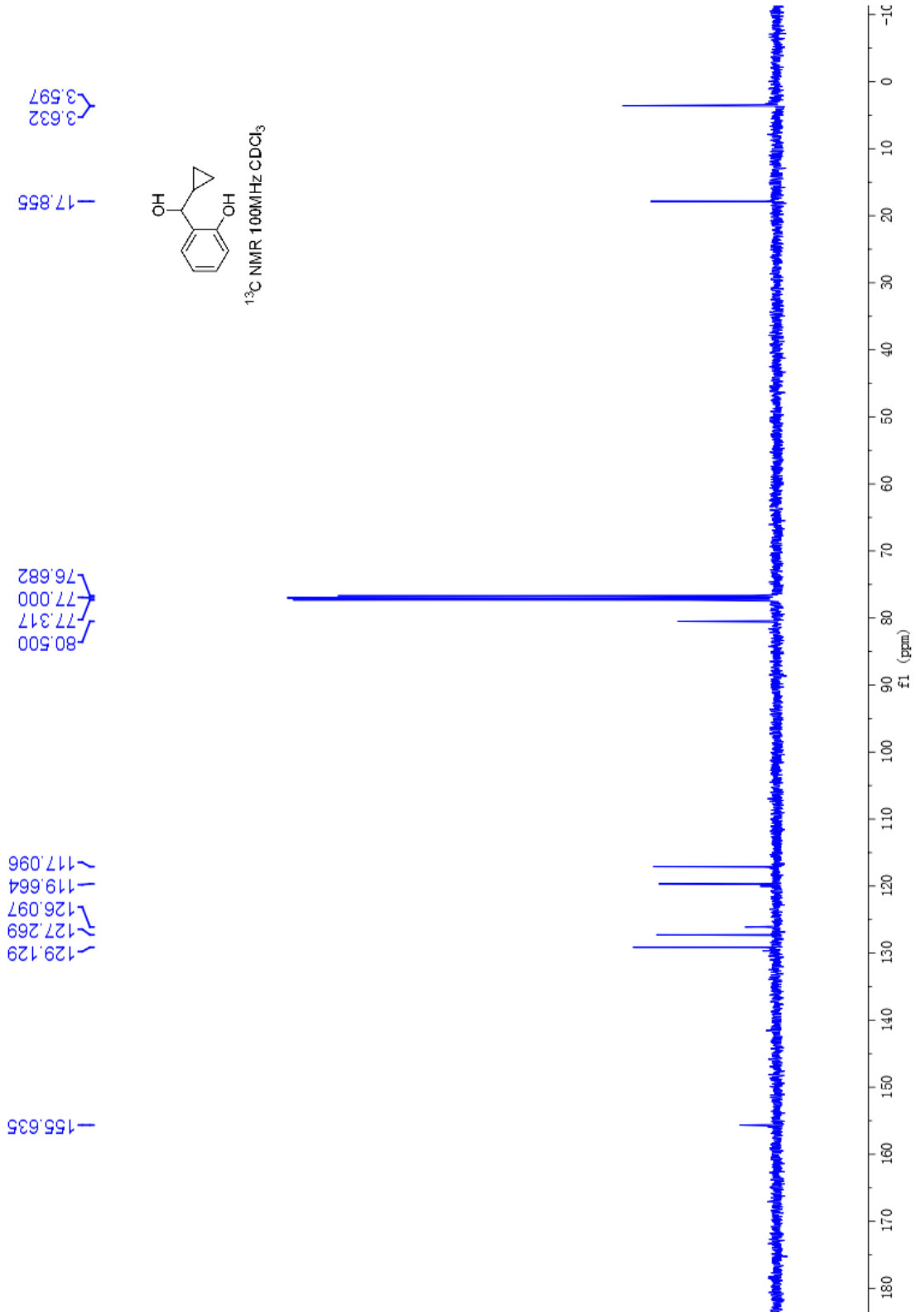
2. Possible mechanism for the formation of products **4q-1** and **4q-2**.



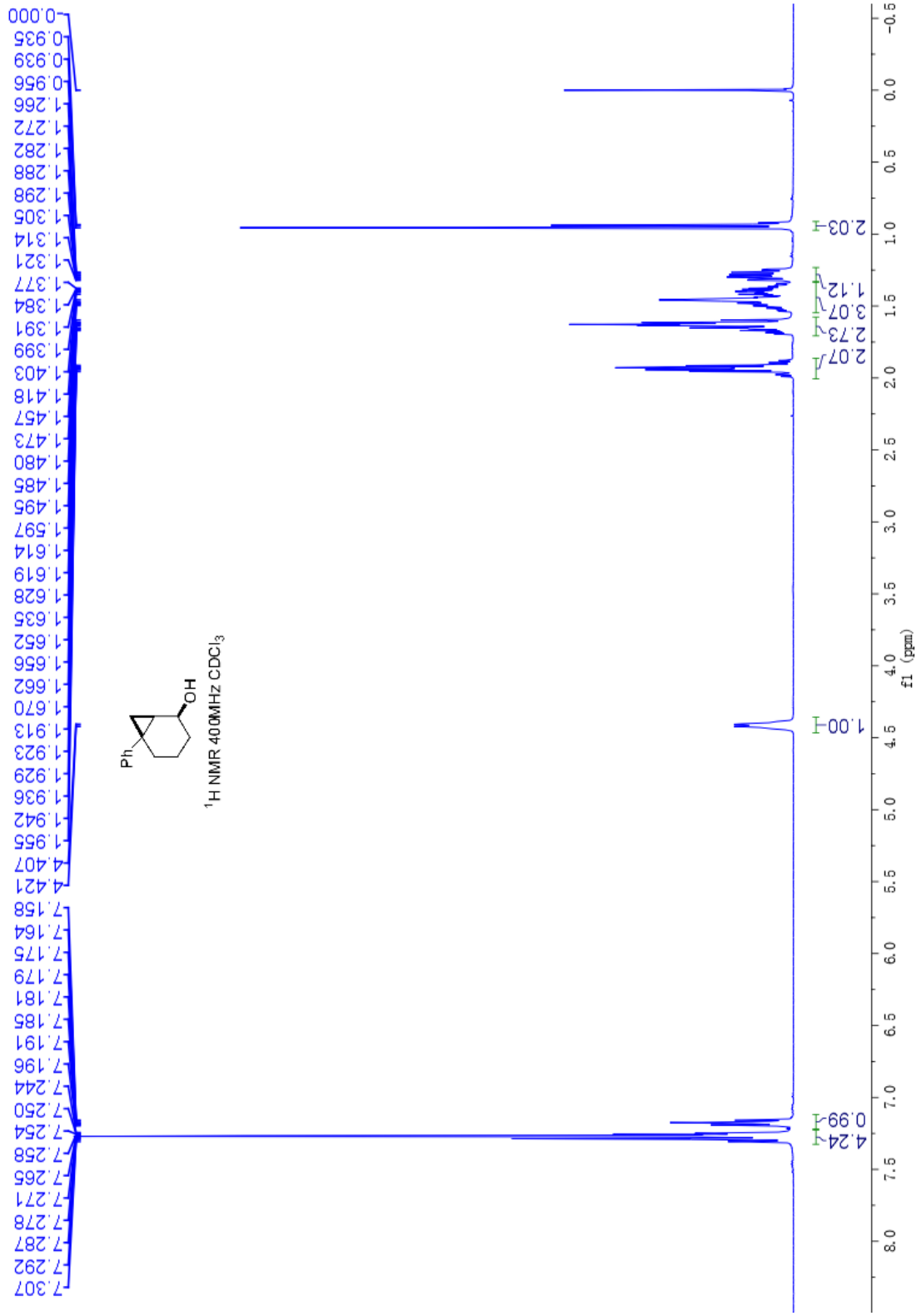
3. NMR spectra

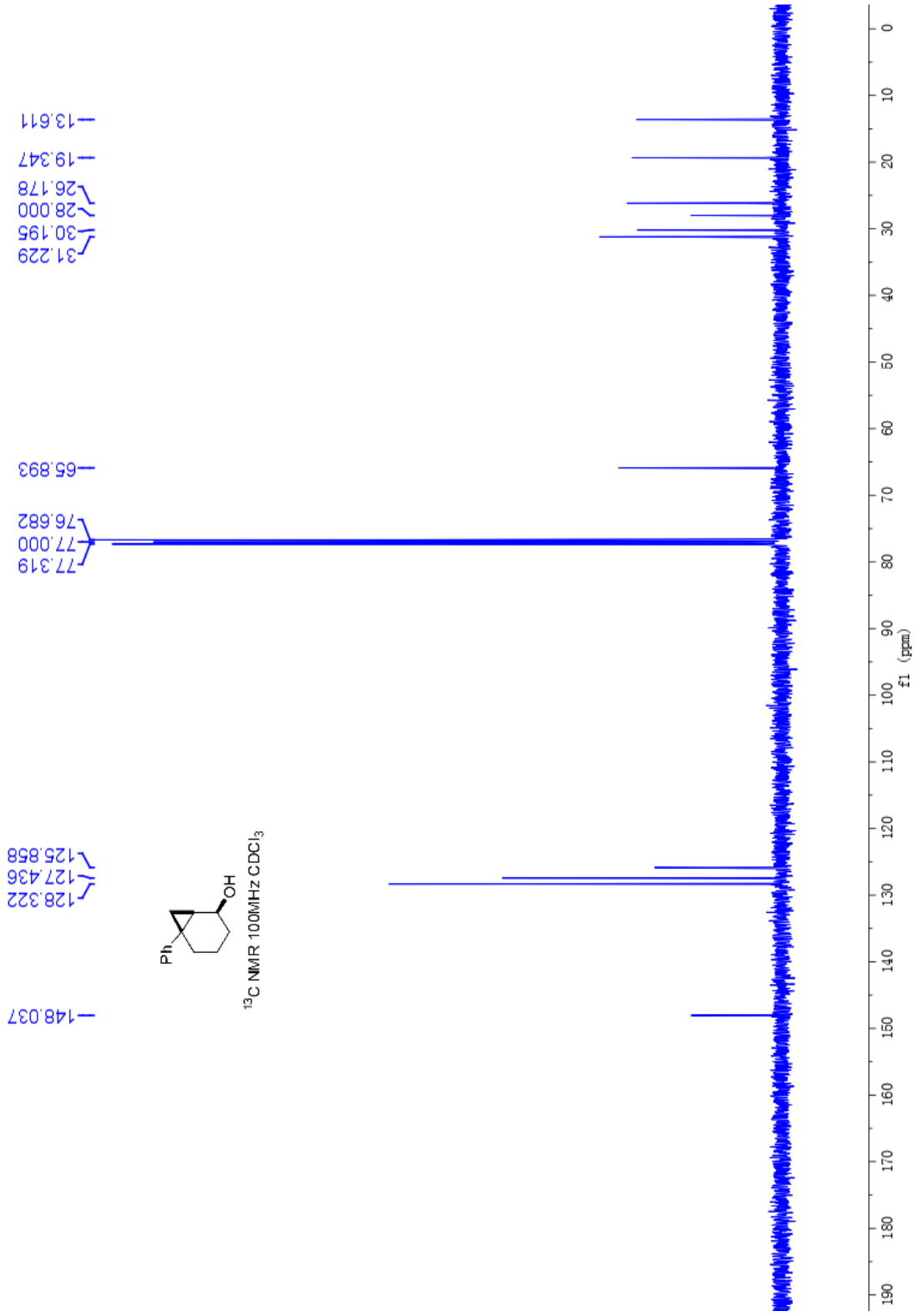
2-(cyclopropyl(hydroxy)methyl)phenol(1c).

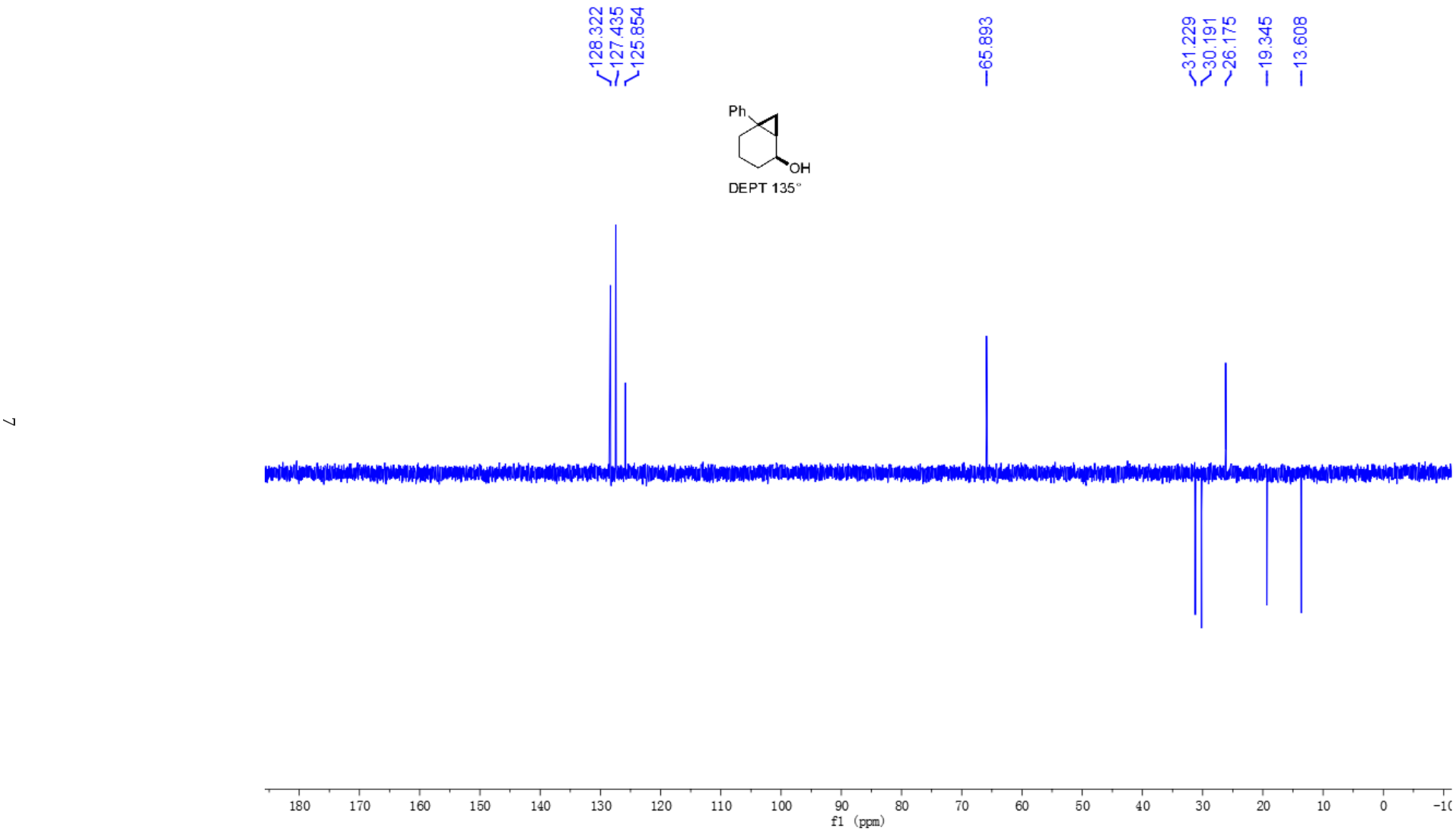



^{13}C NMR 100MHz $CDCl_3$
OH
OH
3.632
3.597
17.855
76.682
77.000
77.317
80.500
117.096
119.664
126.097
127.269
129.129
155.635
f1 (ppm)

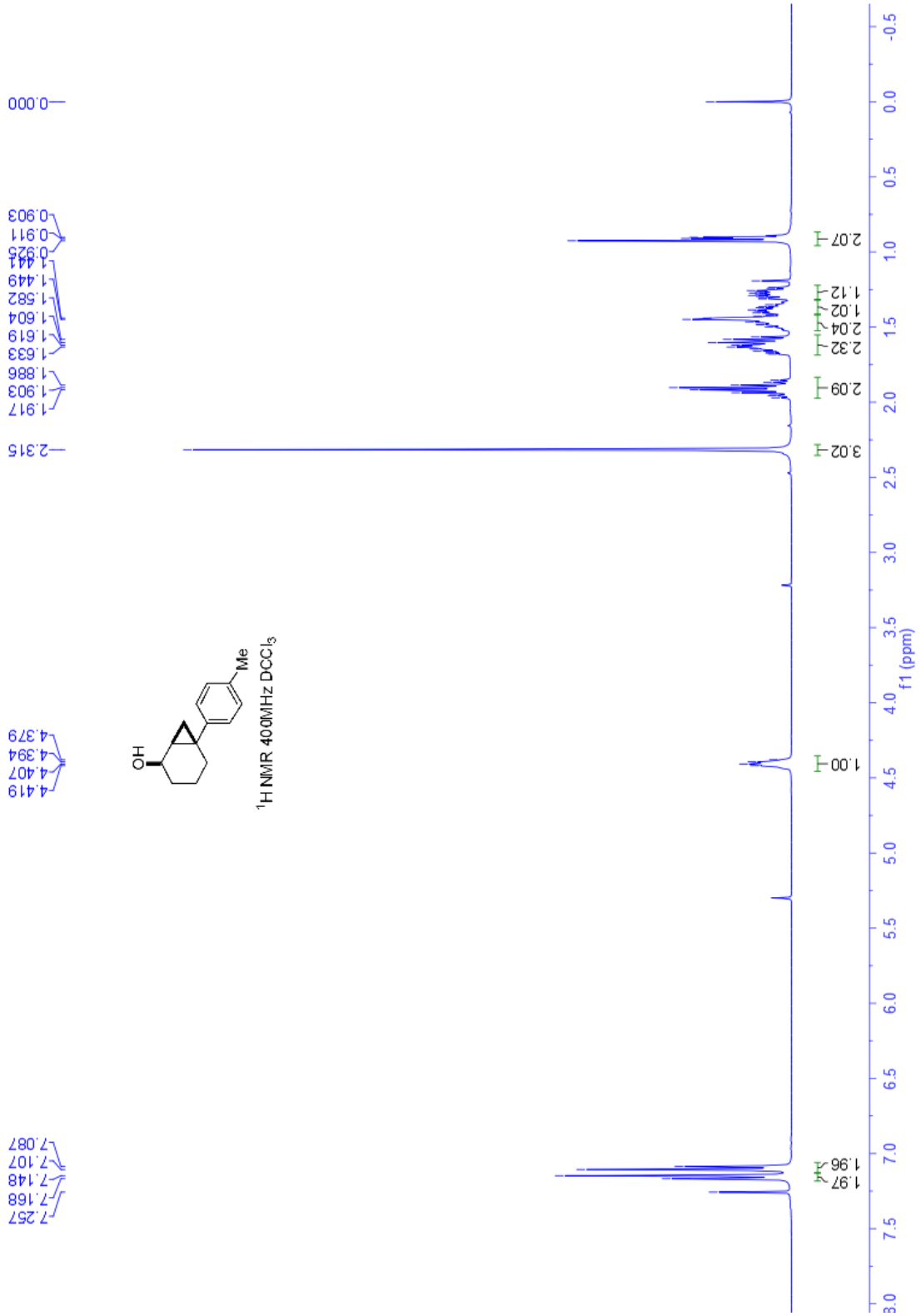
6-Phenylbicyclo[4.1.0]heptan-2-ol (3a).

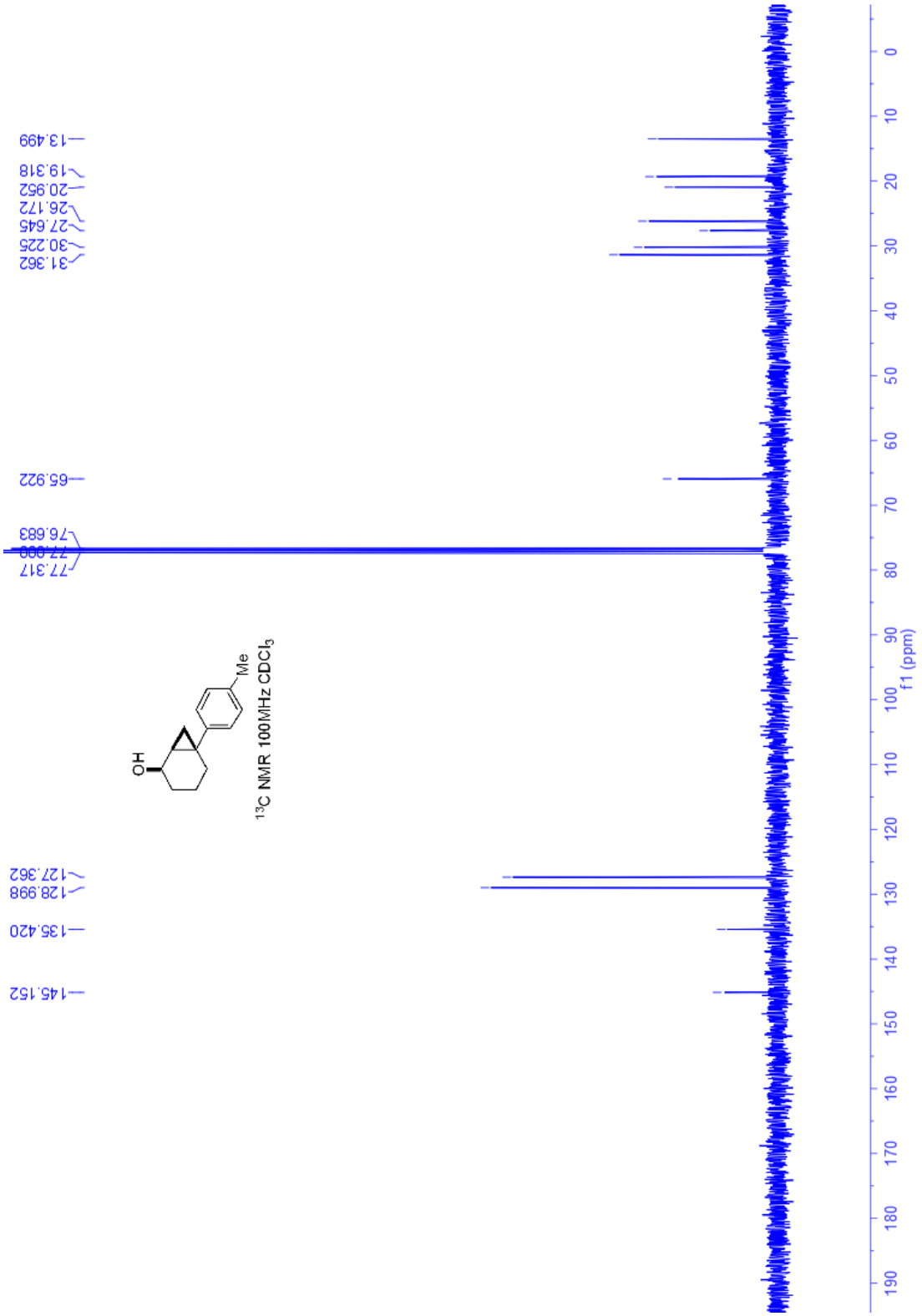


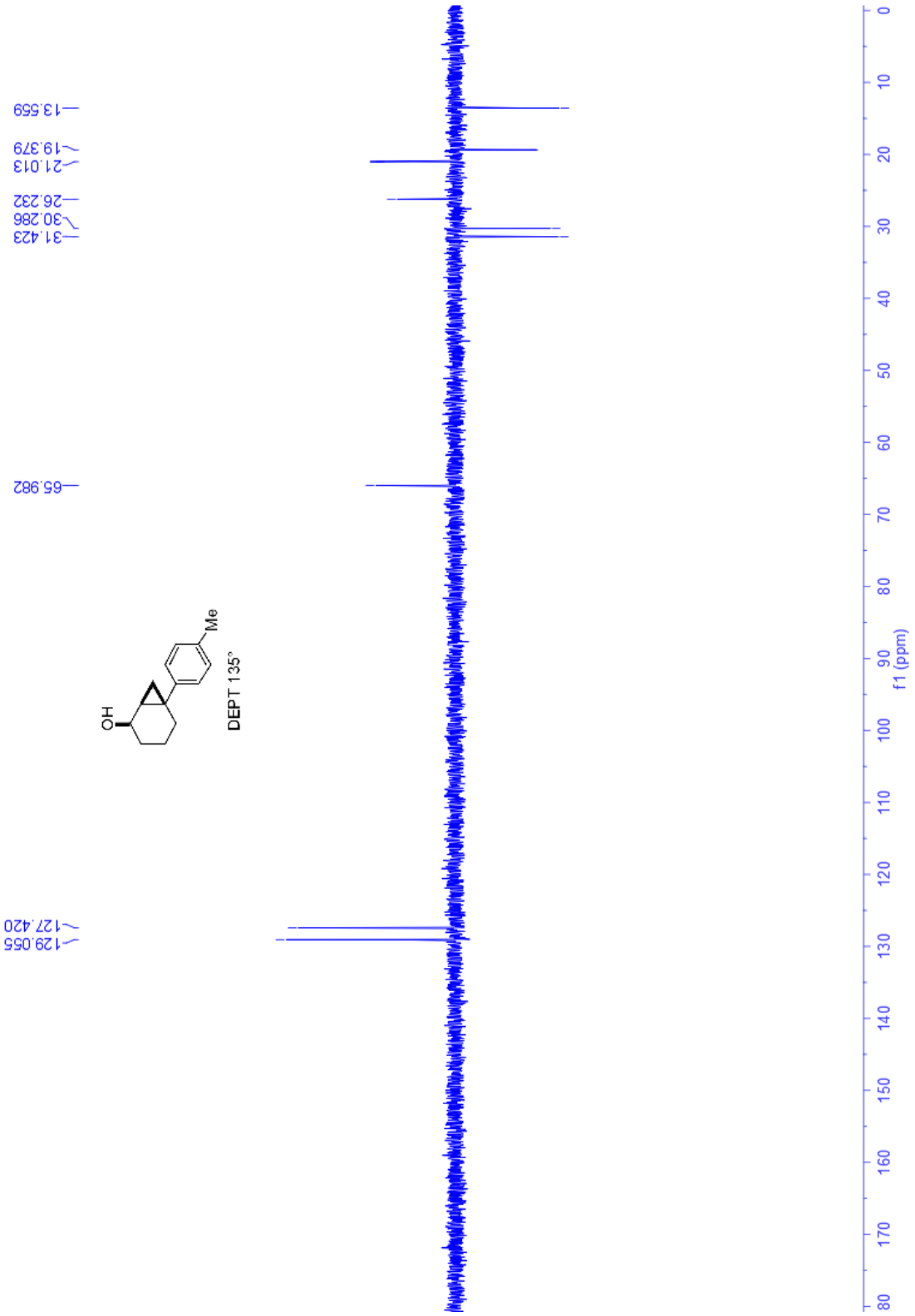

13.611
19.347
26.178
28.000
30.195
31.229
65.893
76.682
77.000
77.319
125.858
127.436
128.322
148.037
OH
Ph
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


128.322
127.435
125.854
65.893
31.229
30.191
26.175
19.345
13.608
Ph
OH
DEPT 135°
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

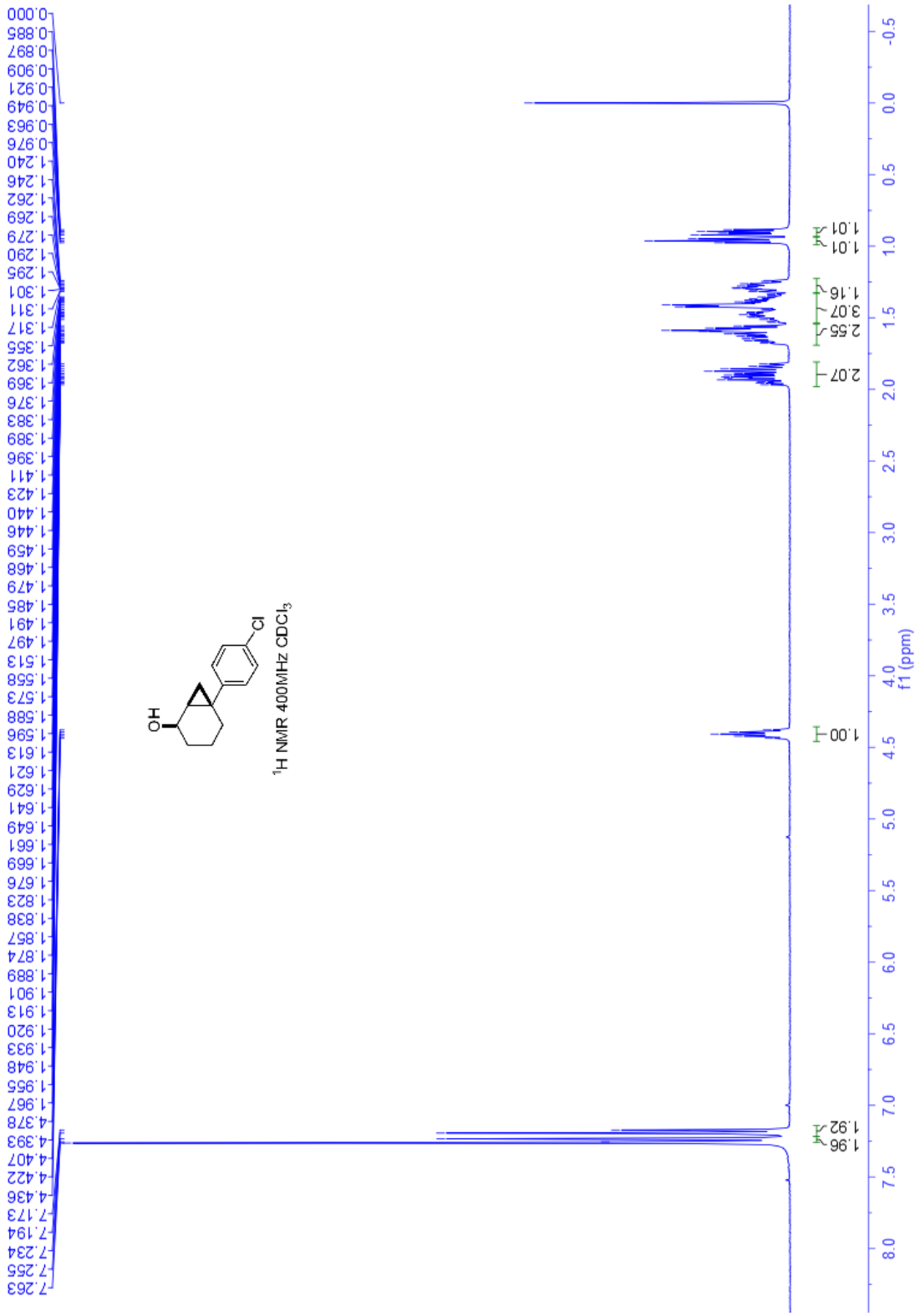
6-(p-Tolyl)bicyclo[4.1.0]heptan-2-ol (3b).

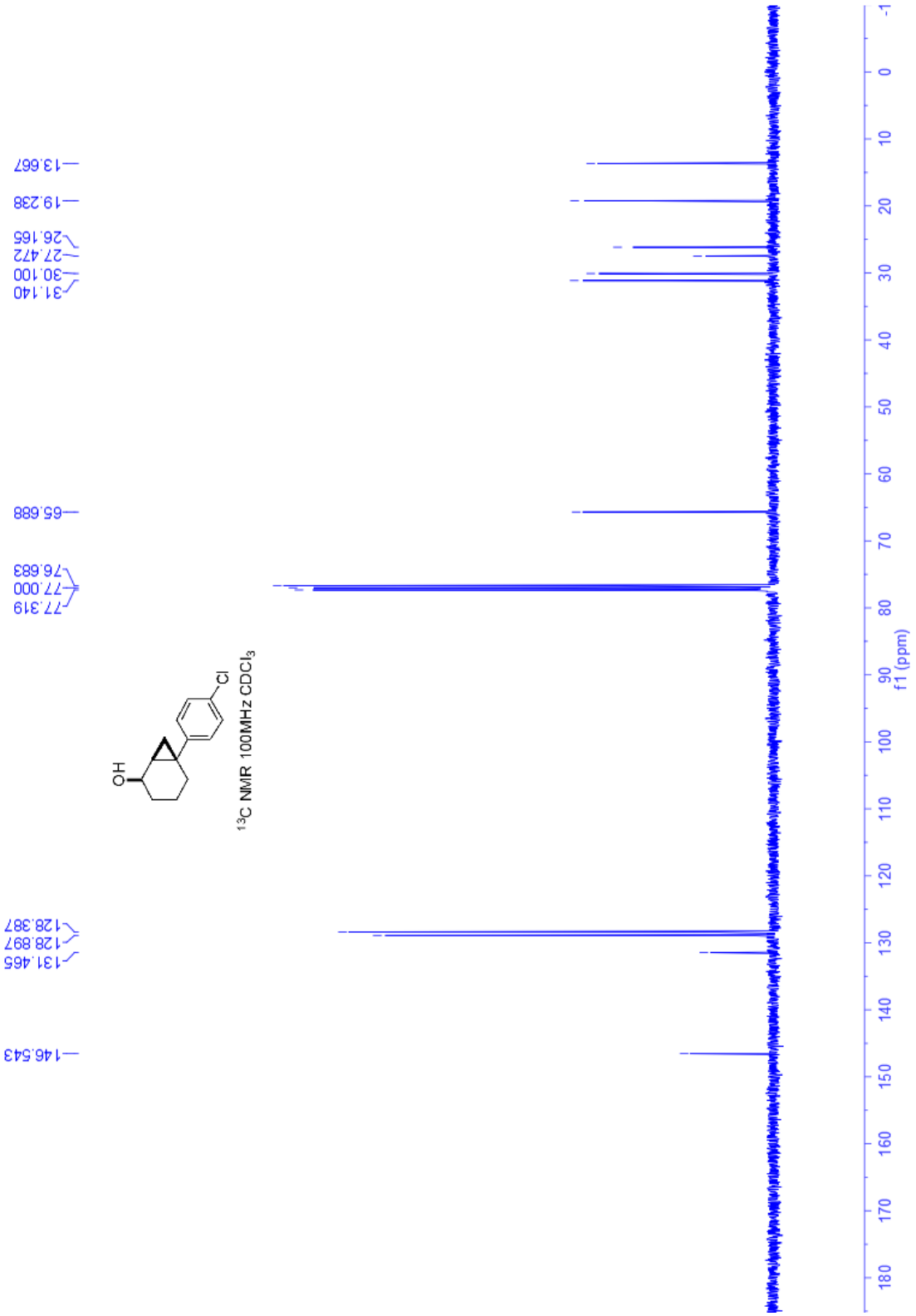


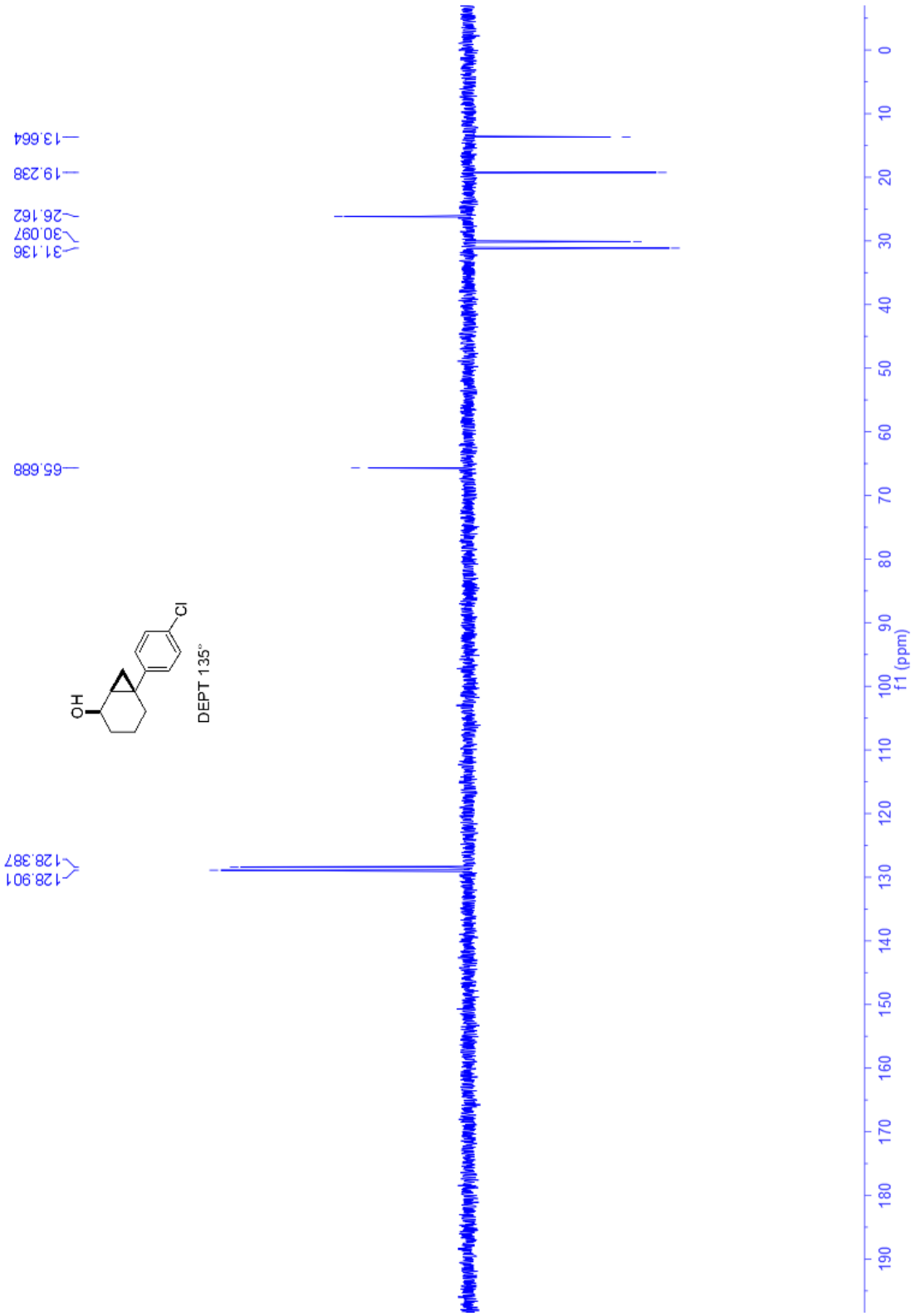

13.499
19.318
20.952
26.172
27.645
30.225
31.362
65.922
76.683
77.000
77.317
127.362
128.998
135.420
145.152
OH
Me
^{13}C NMR 100MHz CDCl$_3$
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
f1 (ppm)


13.559
19.379
21.013
26.232
30.286
31.423
65.982
127.420
129.055
OH
Me
DEPT 135°
f1 (ppm)

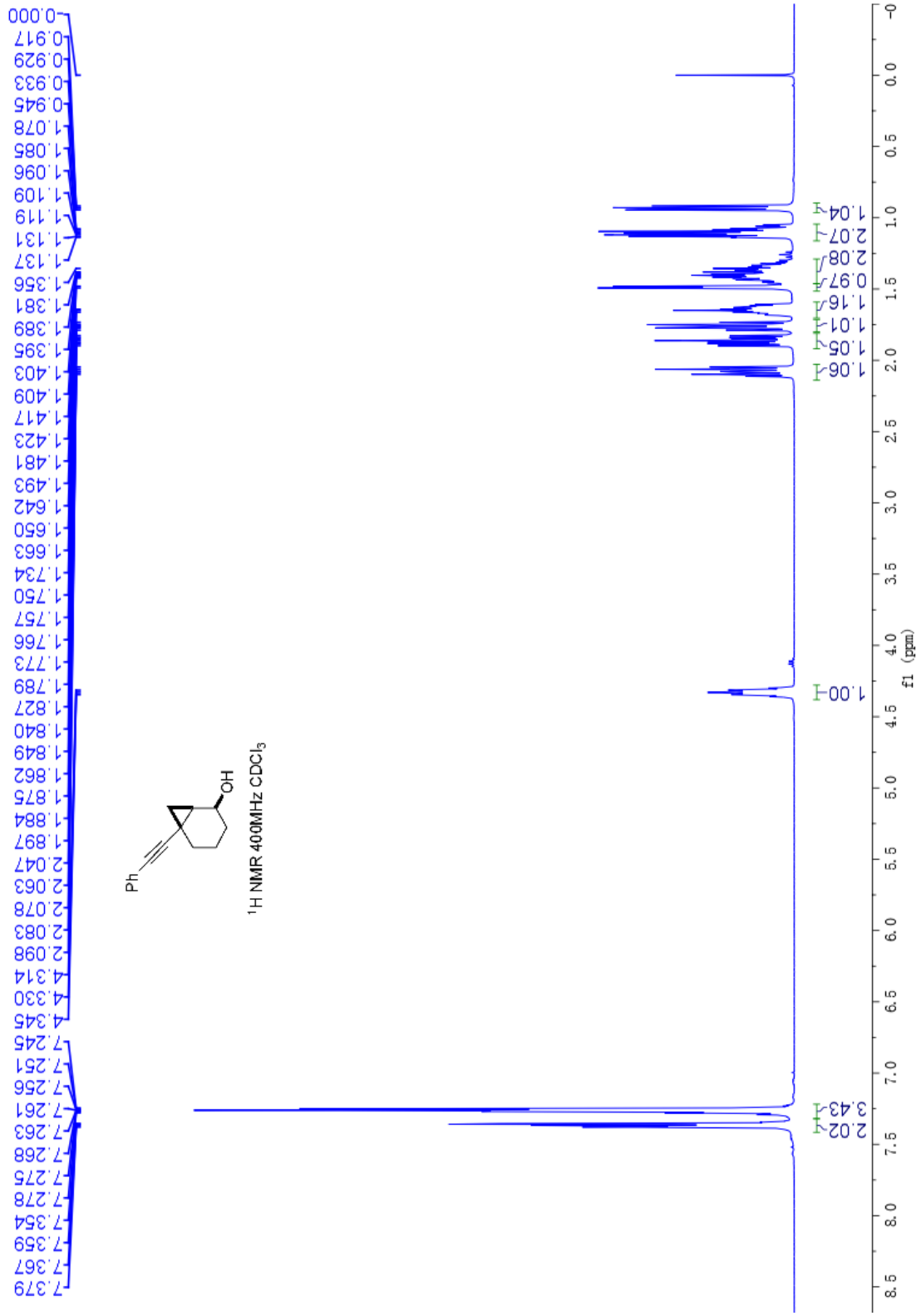
6-(4-Chlorophenyl)bicyclo[4.1.0]heptan-2-ol (3c).

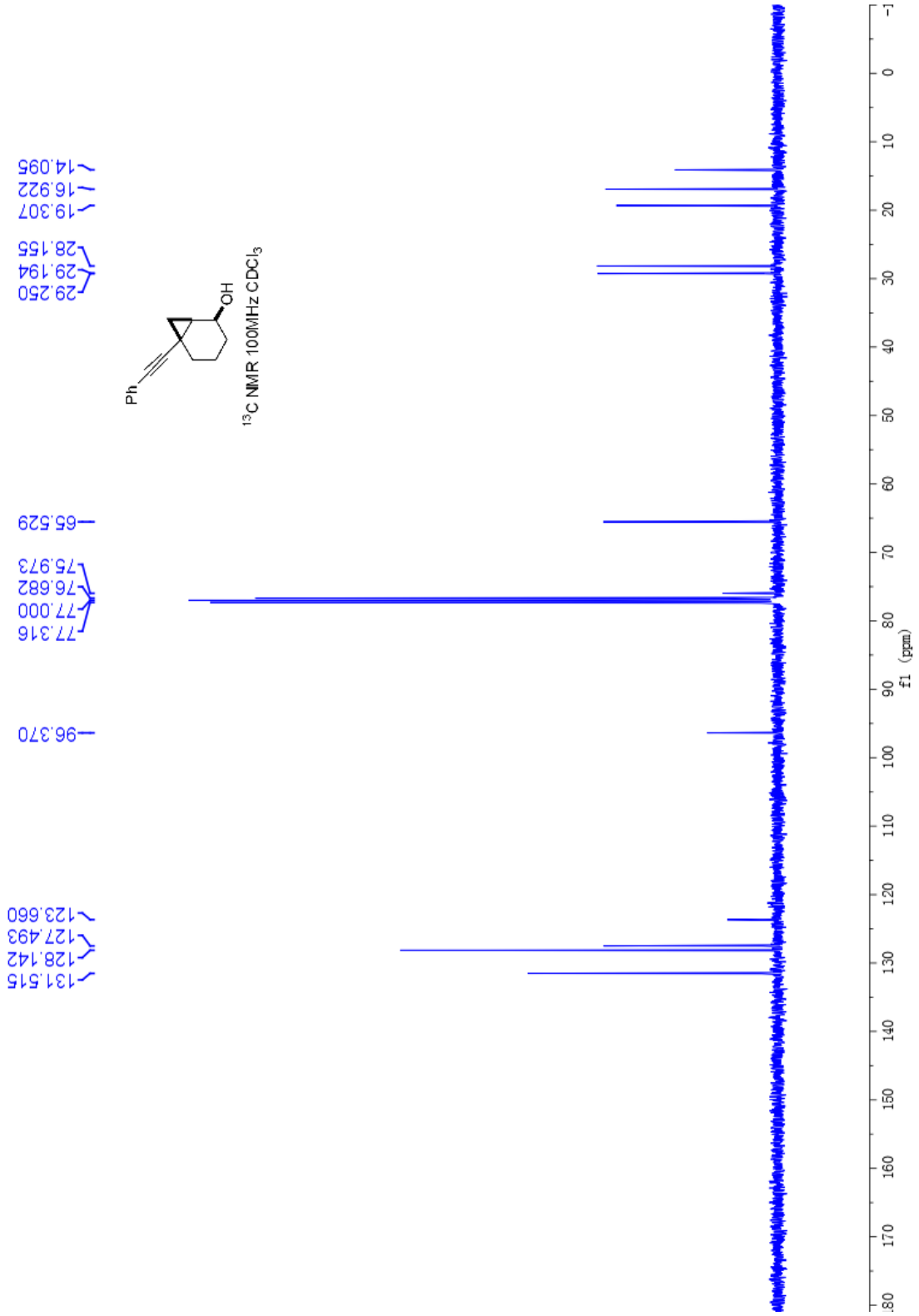


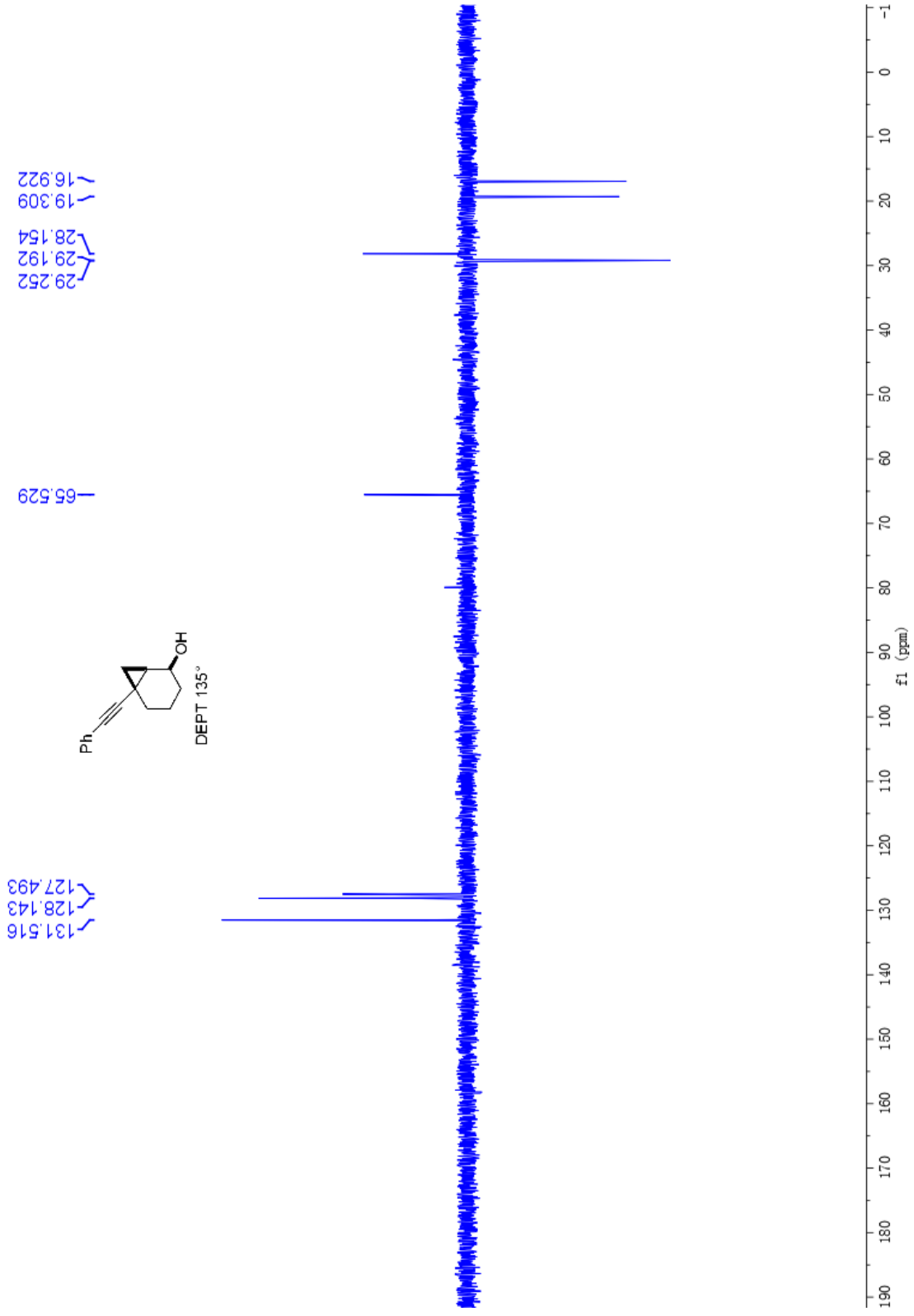

13.667
19.238
26.165
27.472
30.100
31.140
65.688
76.683
77.000
77.319
128.387
128.897
131.465
146.543
OH
Cl
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


13.664
19.238
26.162
30.097
31.136
65.688
128.901
128.387
OH
Cl
DEPT 135°
f1 (ppm)

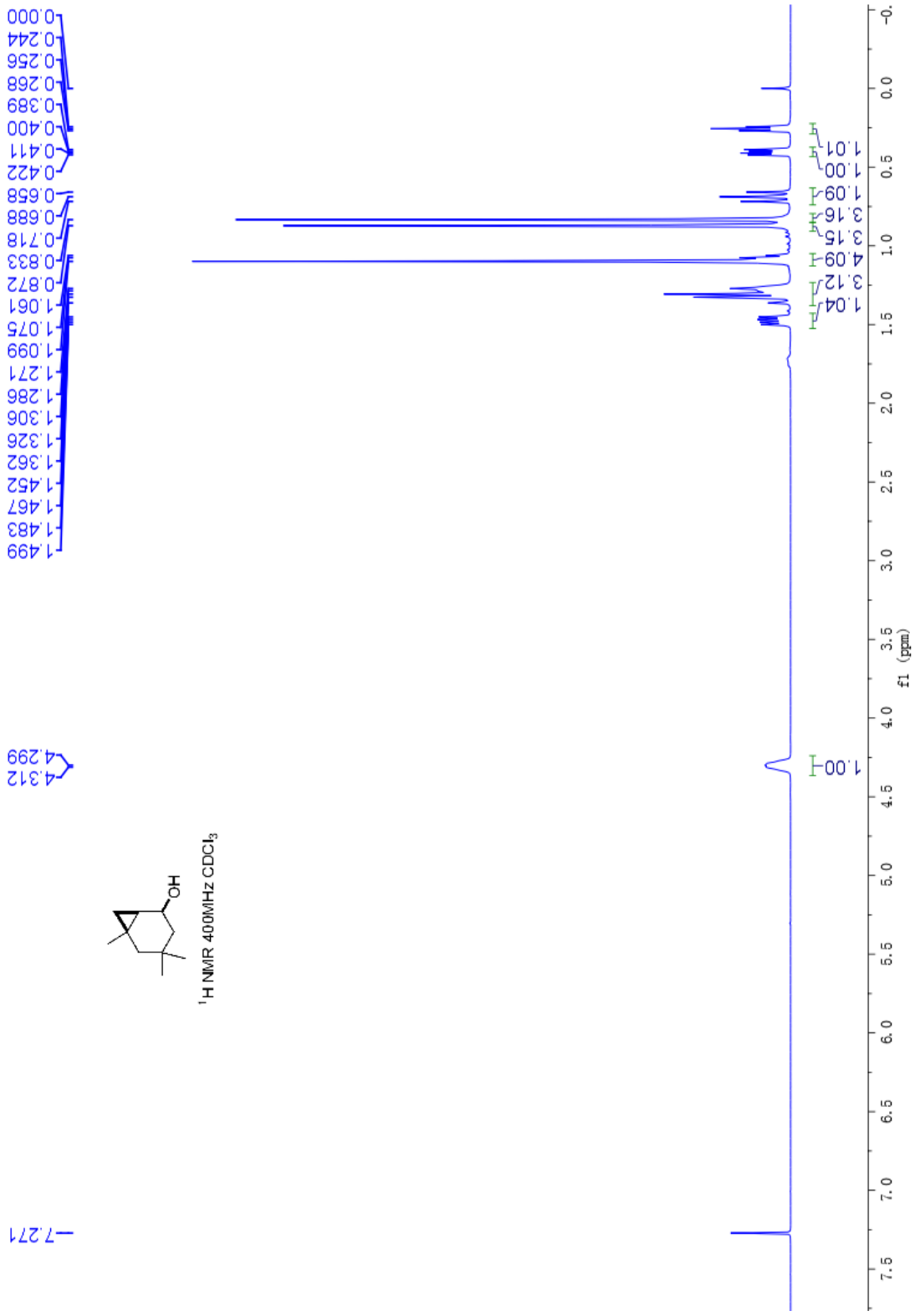
6-(Phenylethynyl)bicyclo[4.1.0]heptan-2-ol (3g).

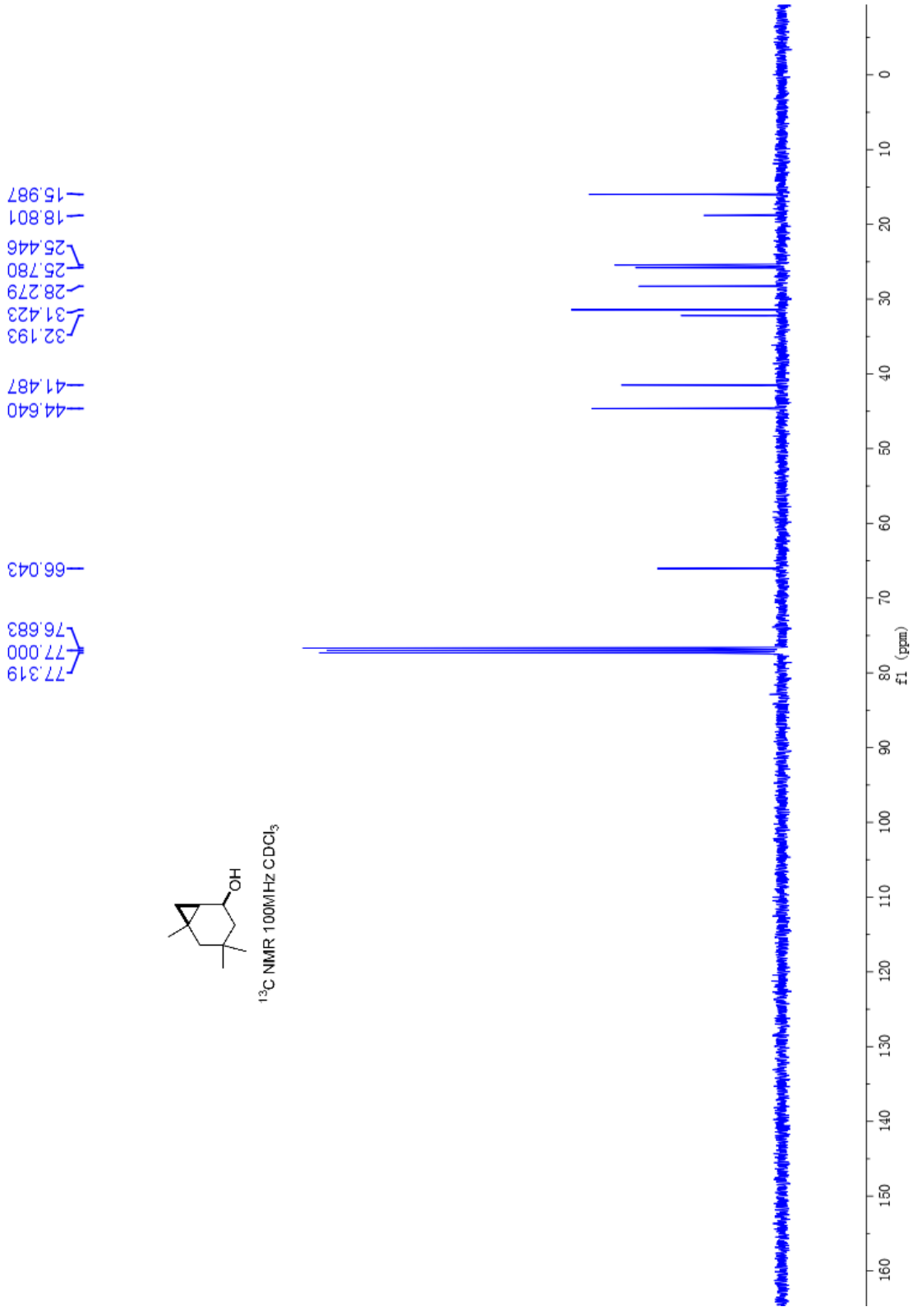


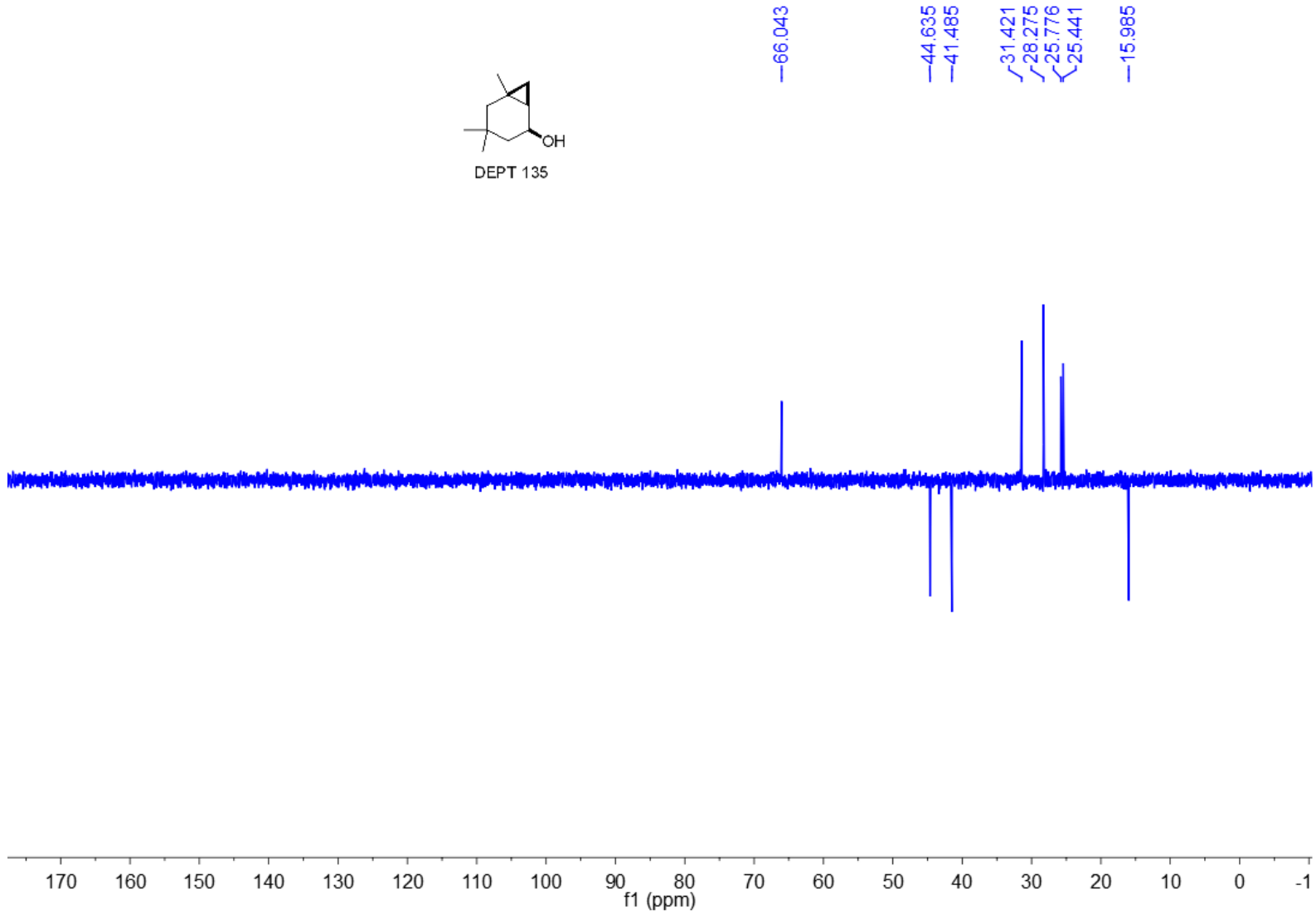

14.095
16.922
19.307
28.155
29.194
29.250
65.529
75.973
76.682
77.000
77.316
96.370
123.660
127.493
128.142
131.515
OH
Ph
^{13}C NMR 100MHz CDCl$_3$
f1 (ppm)


16.922
19.309
28.154
29.192
29.252
65.529
127.493
128.143
131.516
OH
Ph
DEPT 135°
f1 (ppm)

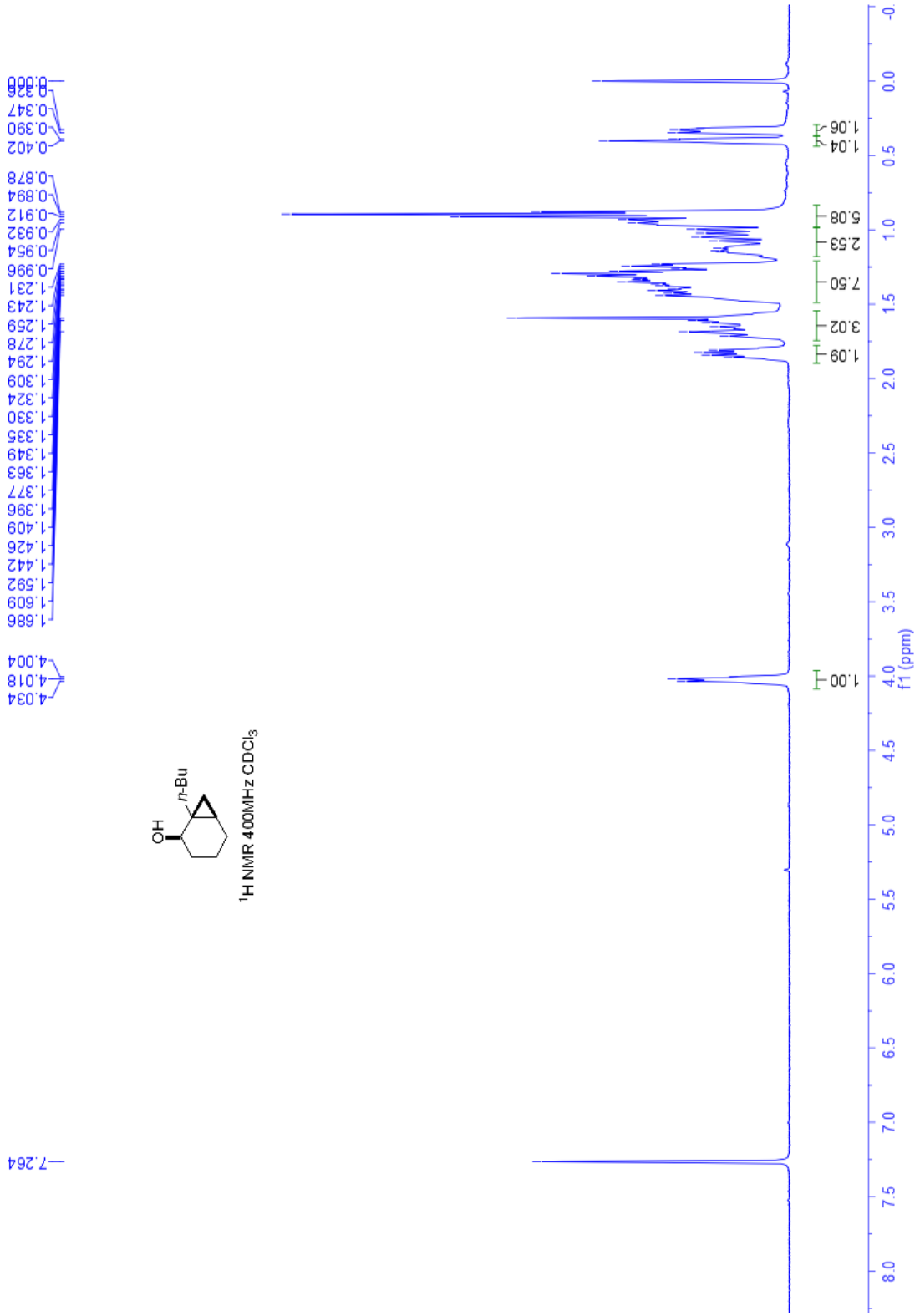
4,4,6-trimethylbicyclo[4.1.0]heptan-2-ol (3h).

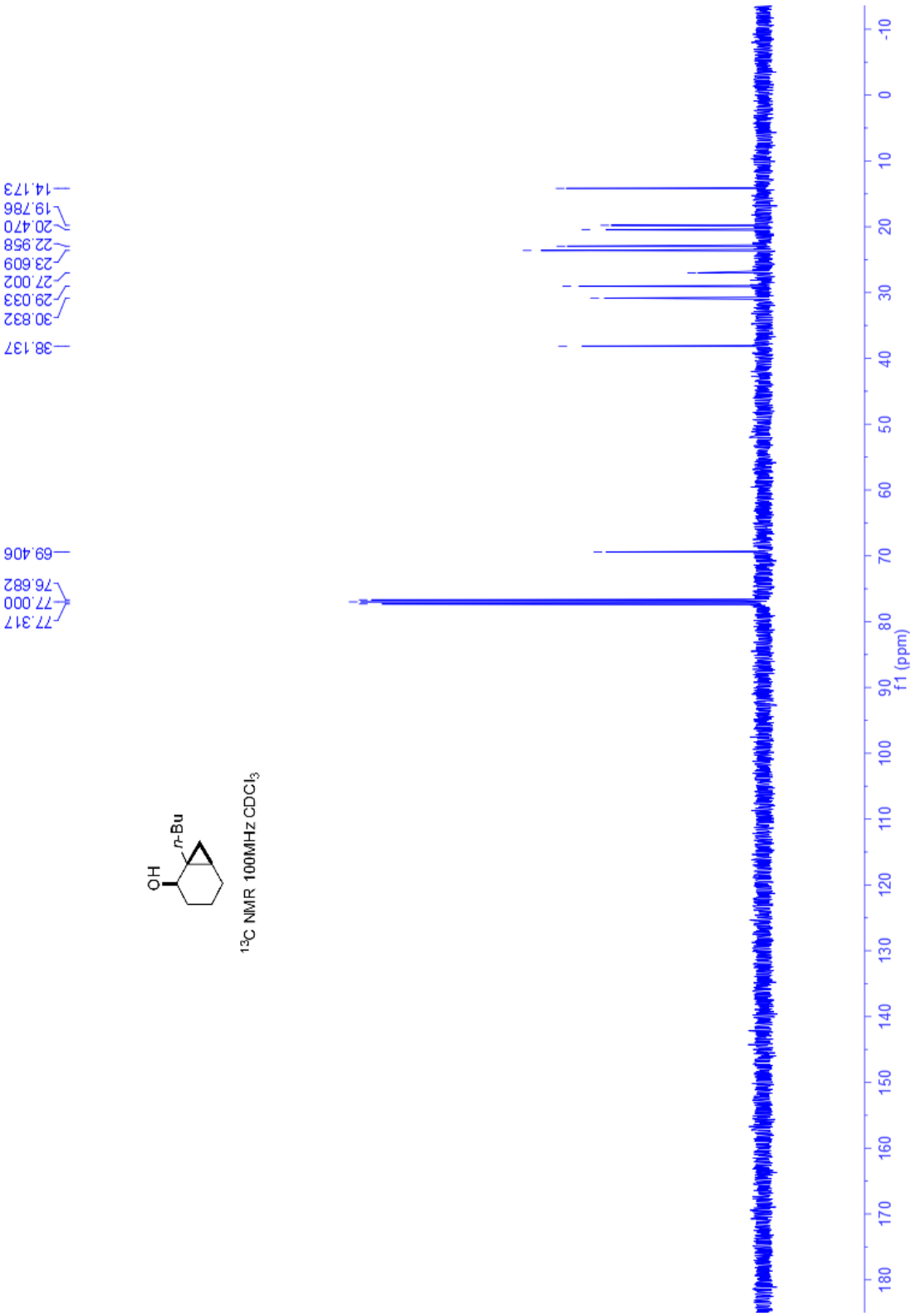


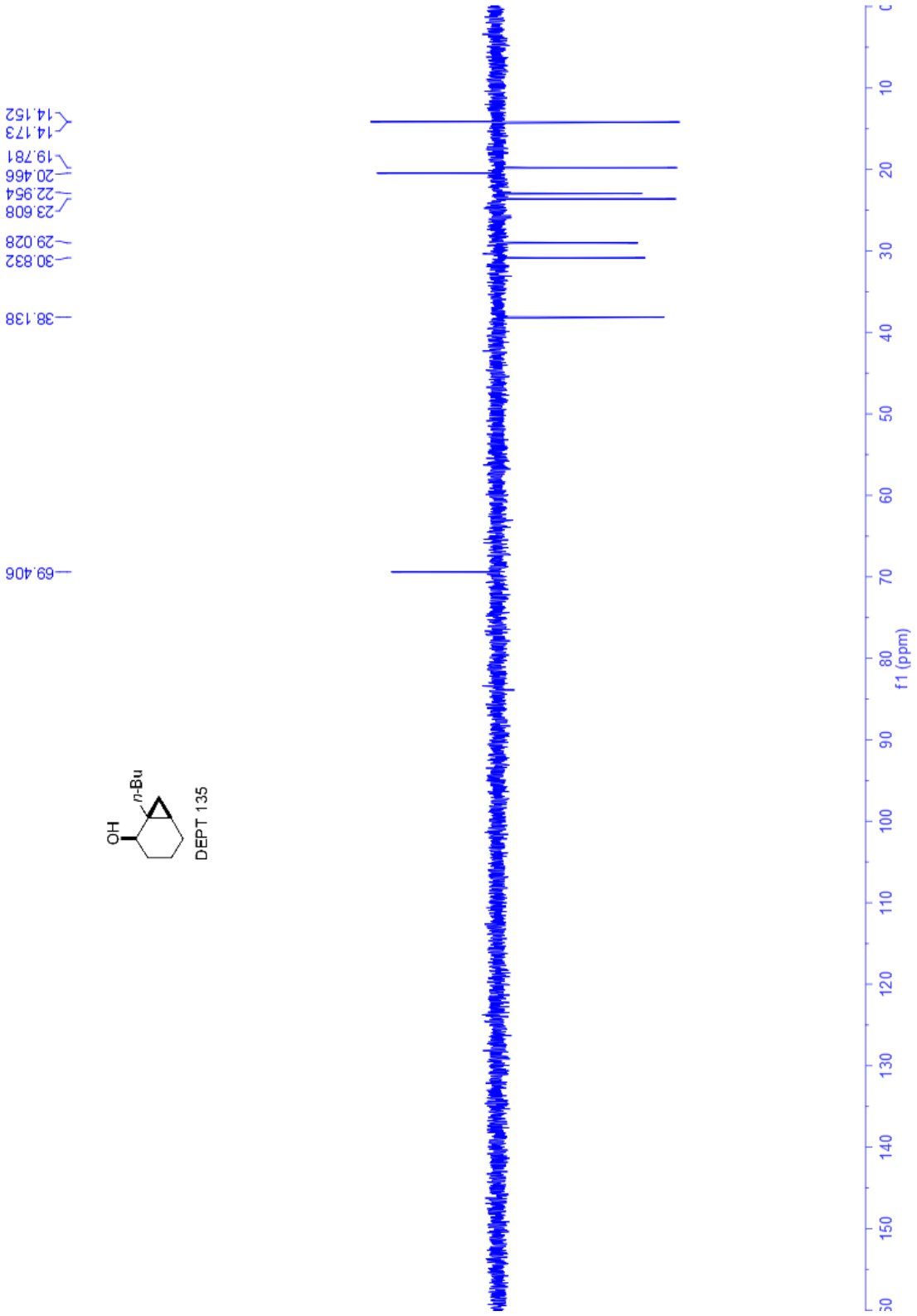

15.987
18.801
25.446
25.780
28.279
31.423
32.193
41.487
44.640
66.043
76.683
77.000
77.319
OH
^{13}C NMR 100MHz CDCl$_3$
f1 (ppm)


66.043
44.635
41.485
31.421
28.275
25.776
25.441
15.985
OH
DEPT 135
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

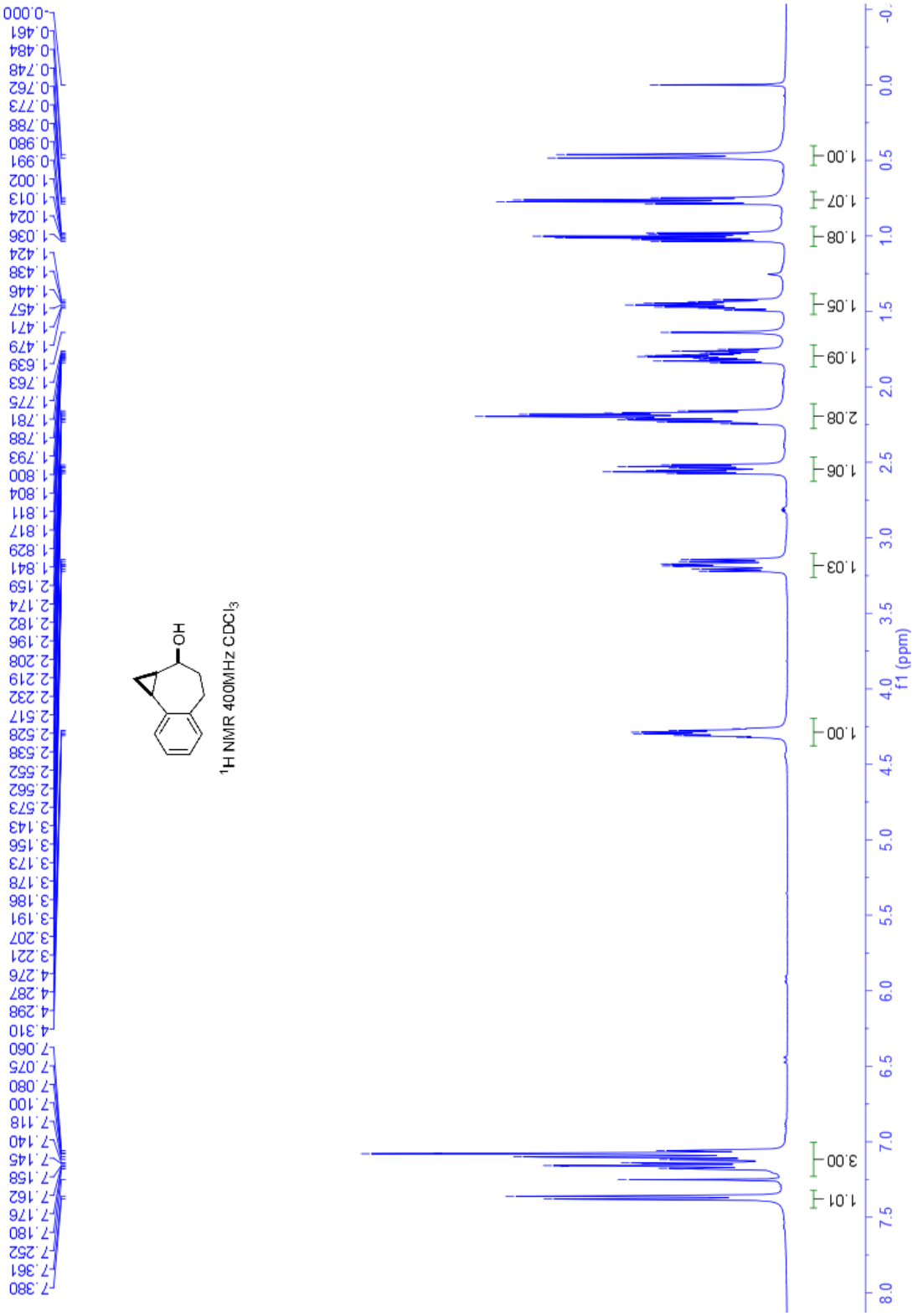
1-butylbicyclo[4.1.0]heptan-2-ol (3j).

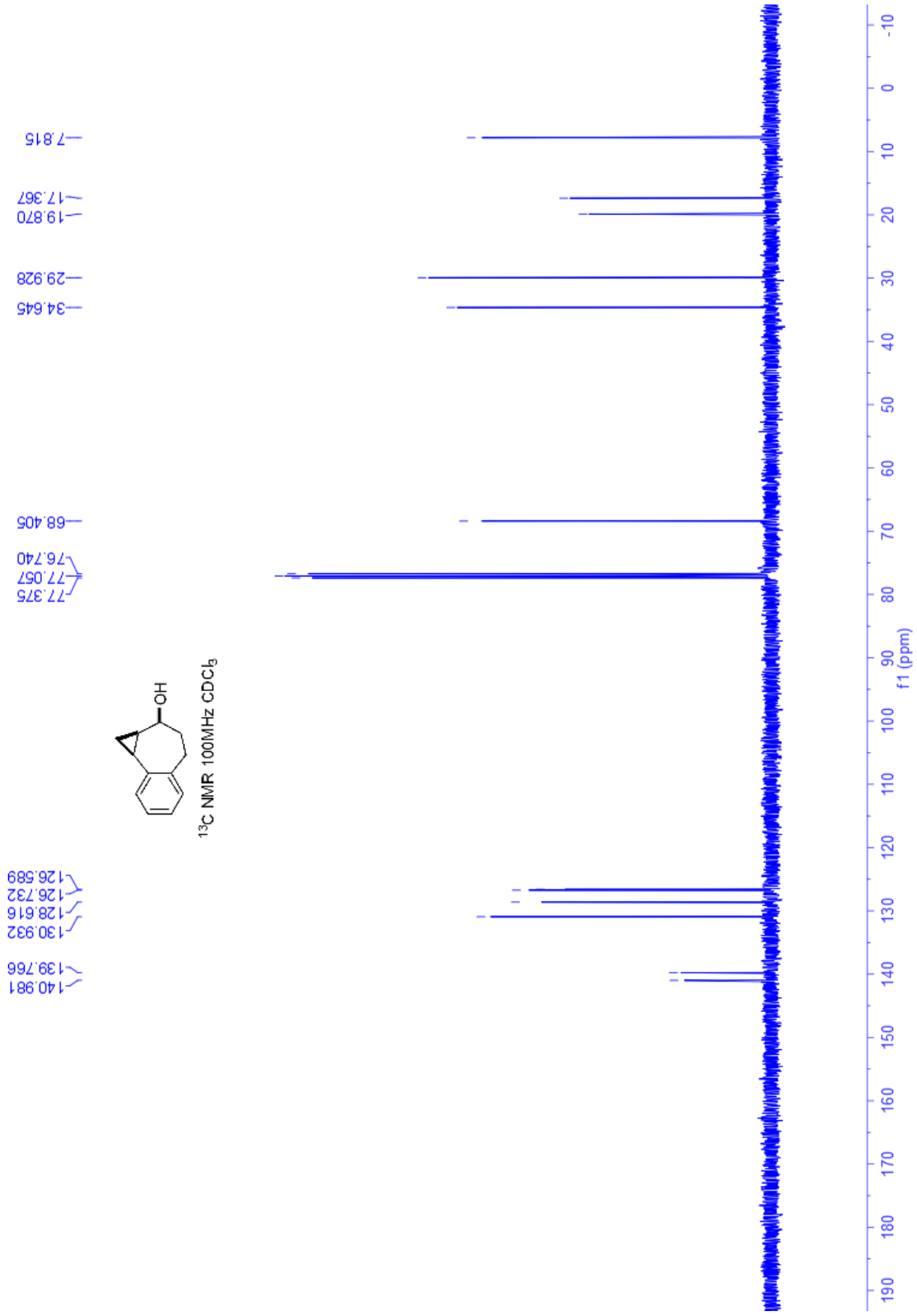


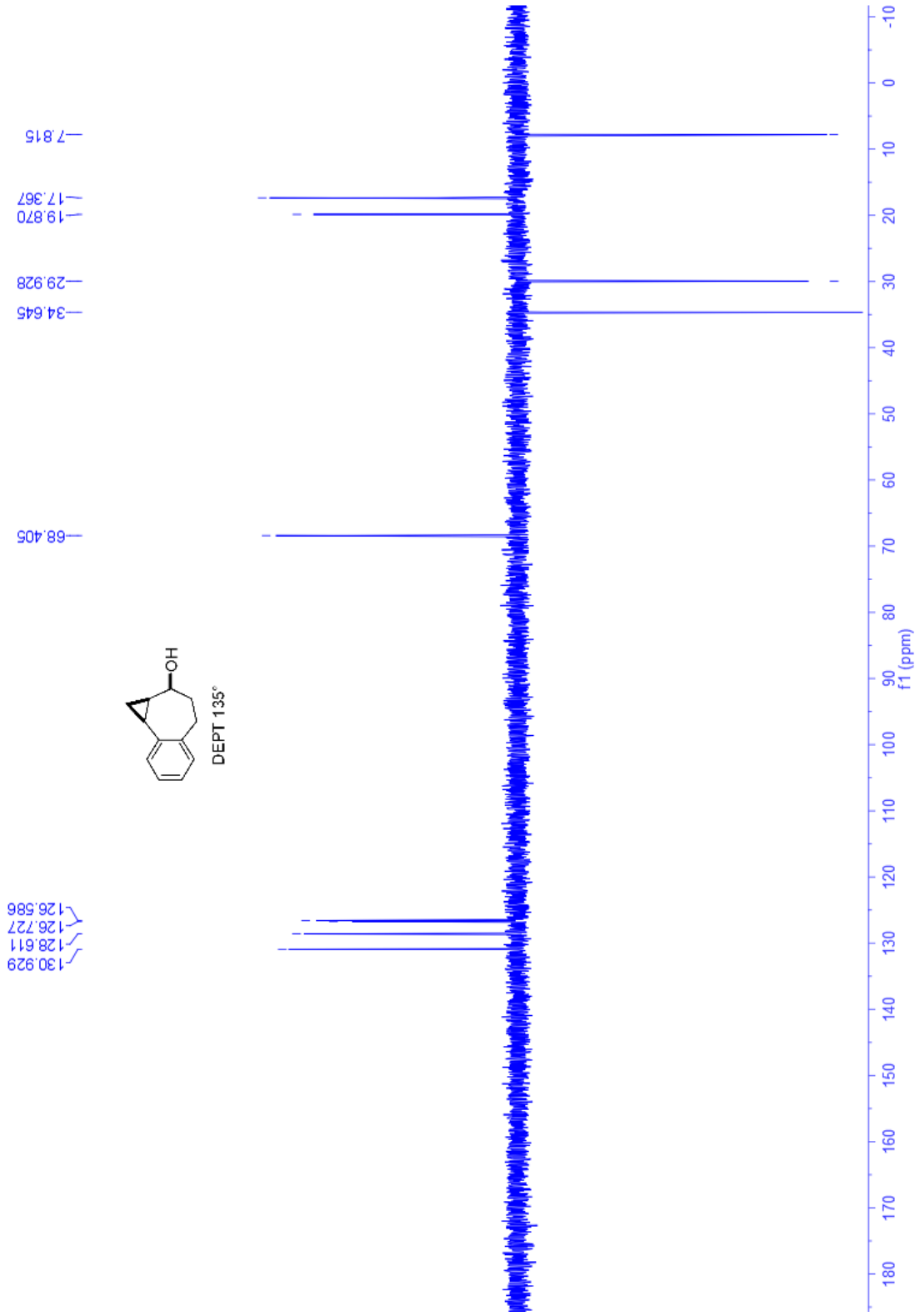

14.173
19.786
20.470
22.958
23.609
27.002
29.033
30.832
38.137
69.406
76.682
77.000
77.317
OH
n-Bu
^{13}C NMR 100MHz $CDCl_3$
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
f1 (ppm)


14.152
14.173
19.781
20.466
22.954
23.608
29.028
30.832
38.138
69.406
OH
n-Bu
DEPT 135
f1 (ppm)

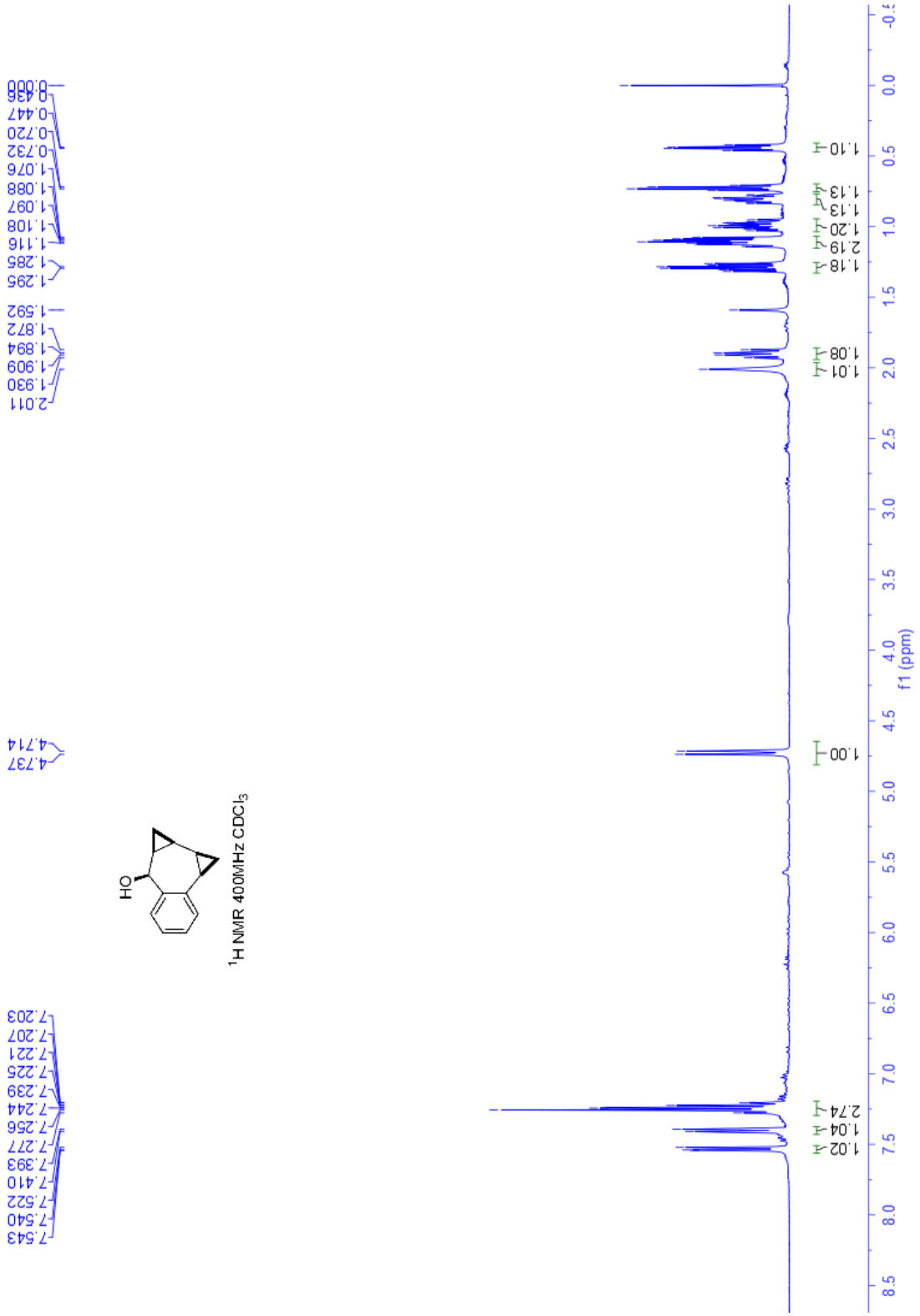
Compound 3n.

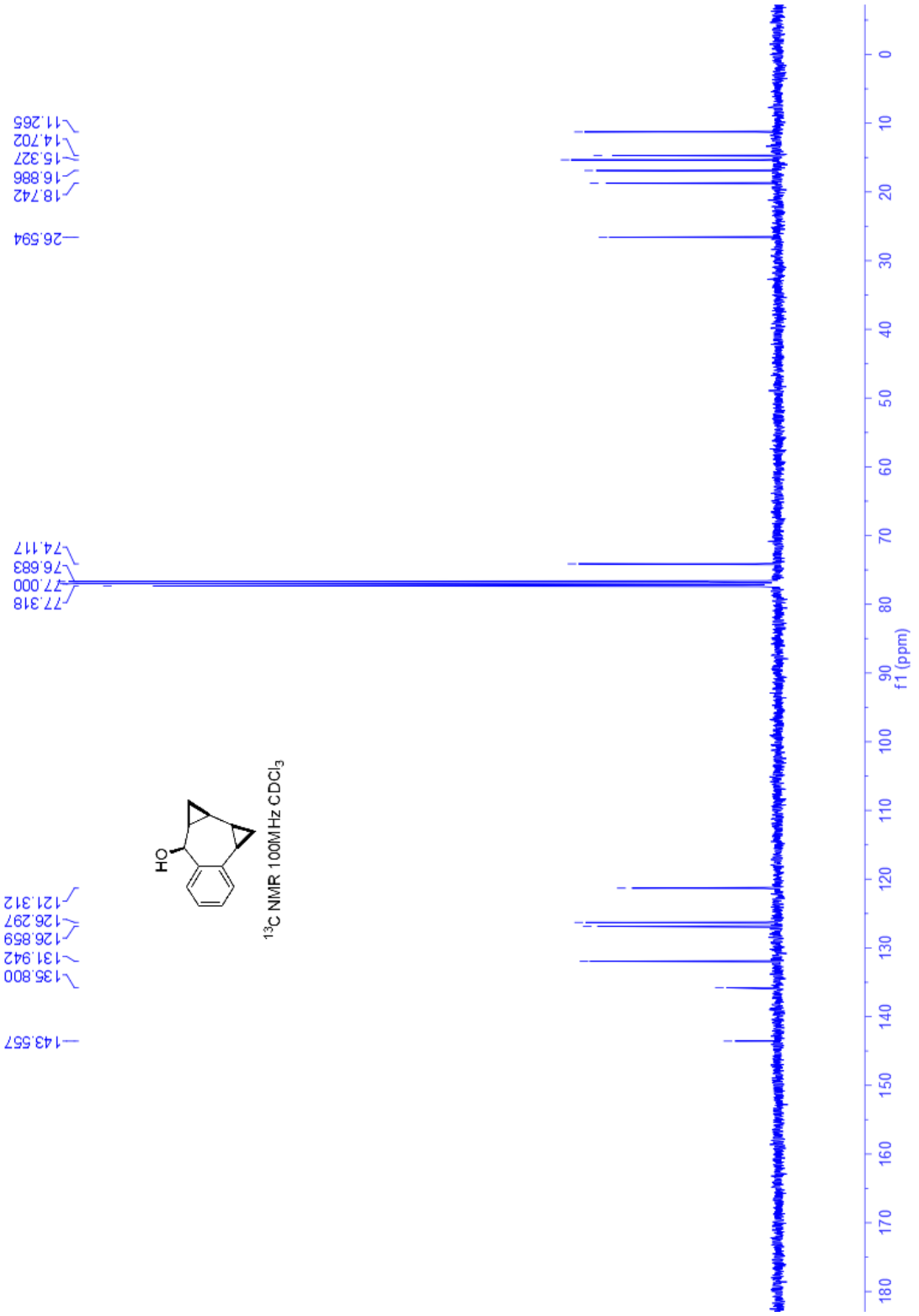


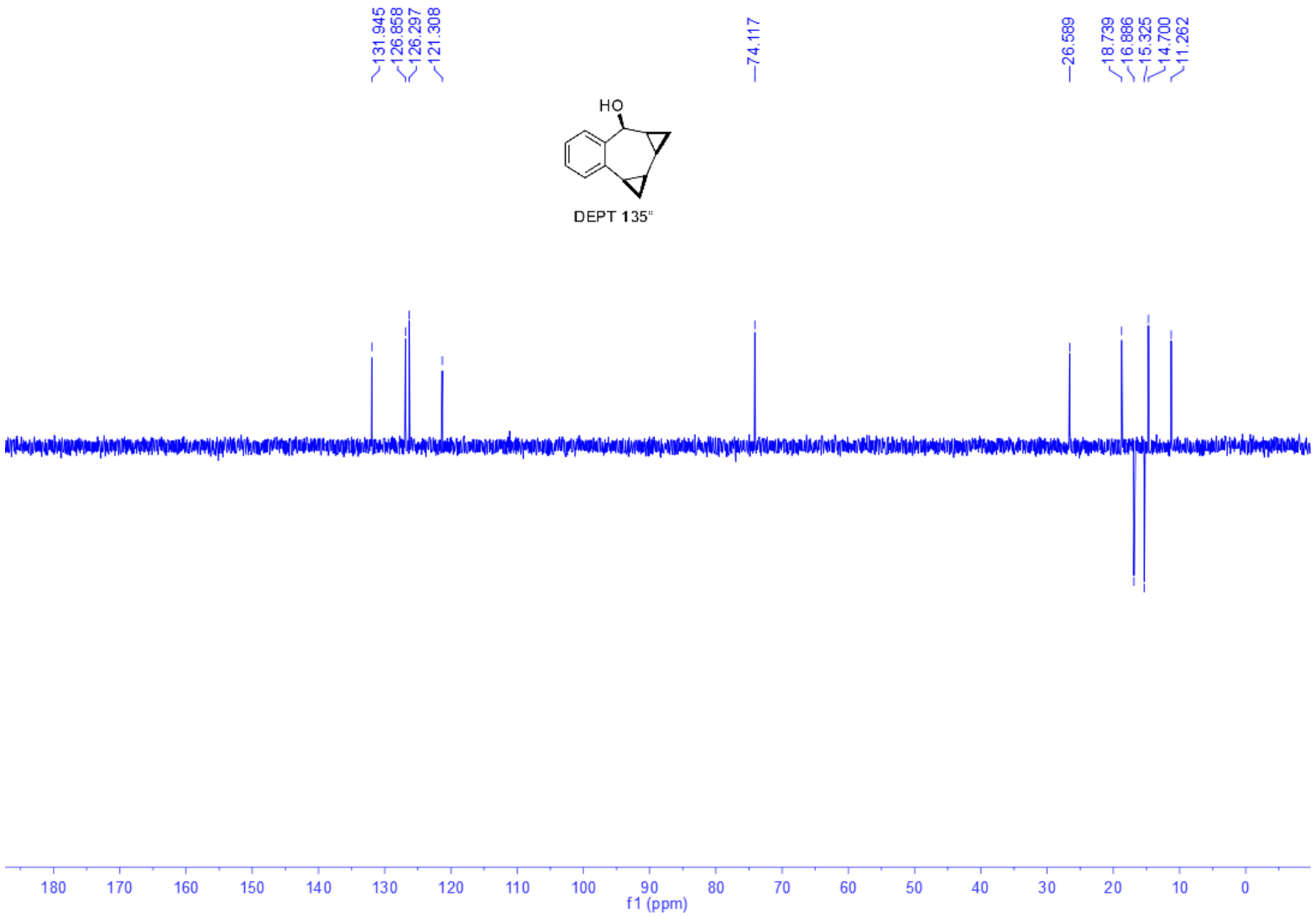

OH
^{13}C NMR 100MHz $CDCl_3$
7.815
17.367
19.870
29.928
34.645
68.405
76.740
77.057
77.375
126.589
126.732
128.616
130.932
139.766
140.981
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
f1 (ppm)


7.815
17.367
19.870
29.928
34.645
68.405
126.586
126.727
128.611
130.929
OH
DEPT 135°
f1 (ppm)

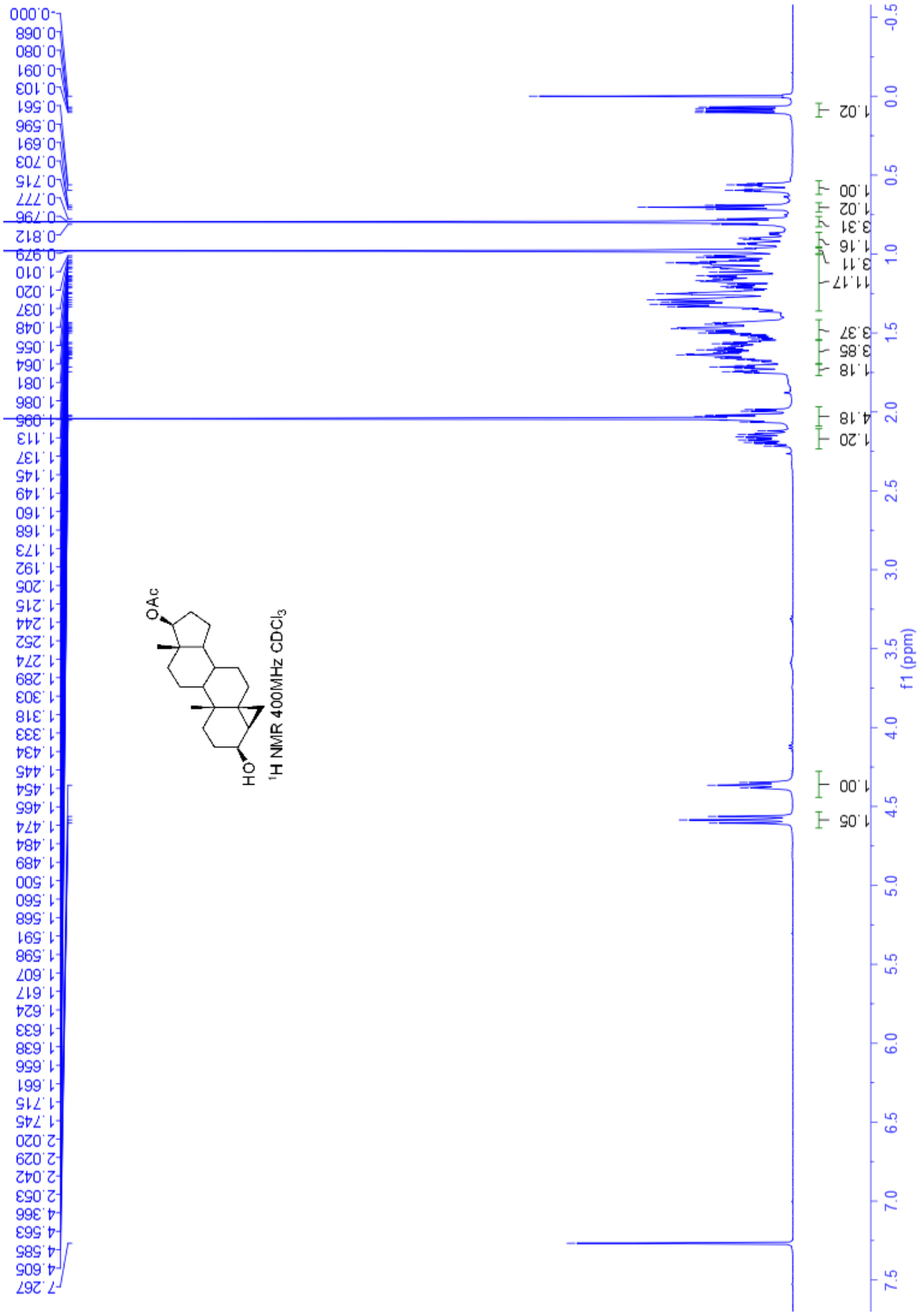
Compound 3o.

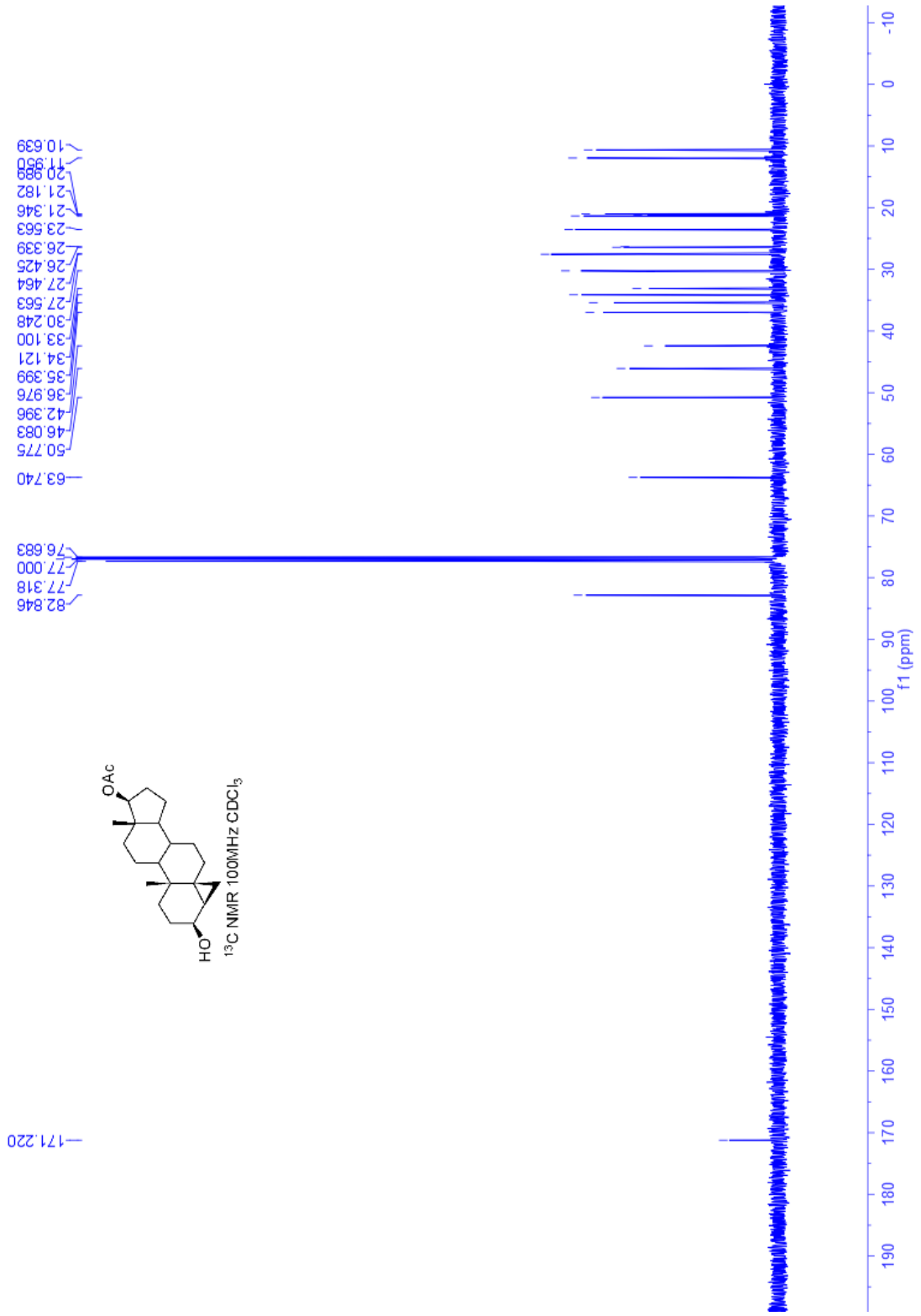

0.000
0.436
0.447
0.720
0.732
1.076
1.088
1.097
1.108
1.116
1.285
1.295
1.592
1.872
1.894
1.909
1.930
2.011
4.714
4.737
7.203
7.207
7.221
7.225
7.239
7.244
7.256
7.277
7.393
7.410
7.522
7.540
7.543
HO
^{1}H NMR 400MHz $CDCl_3$
1.10
1.13
1.13
1.20
2.19
1.18
1.01
1.08
1.00
2.74
1.04
1.02
f1 (ppm)

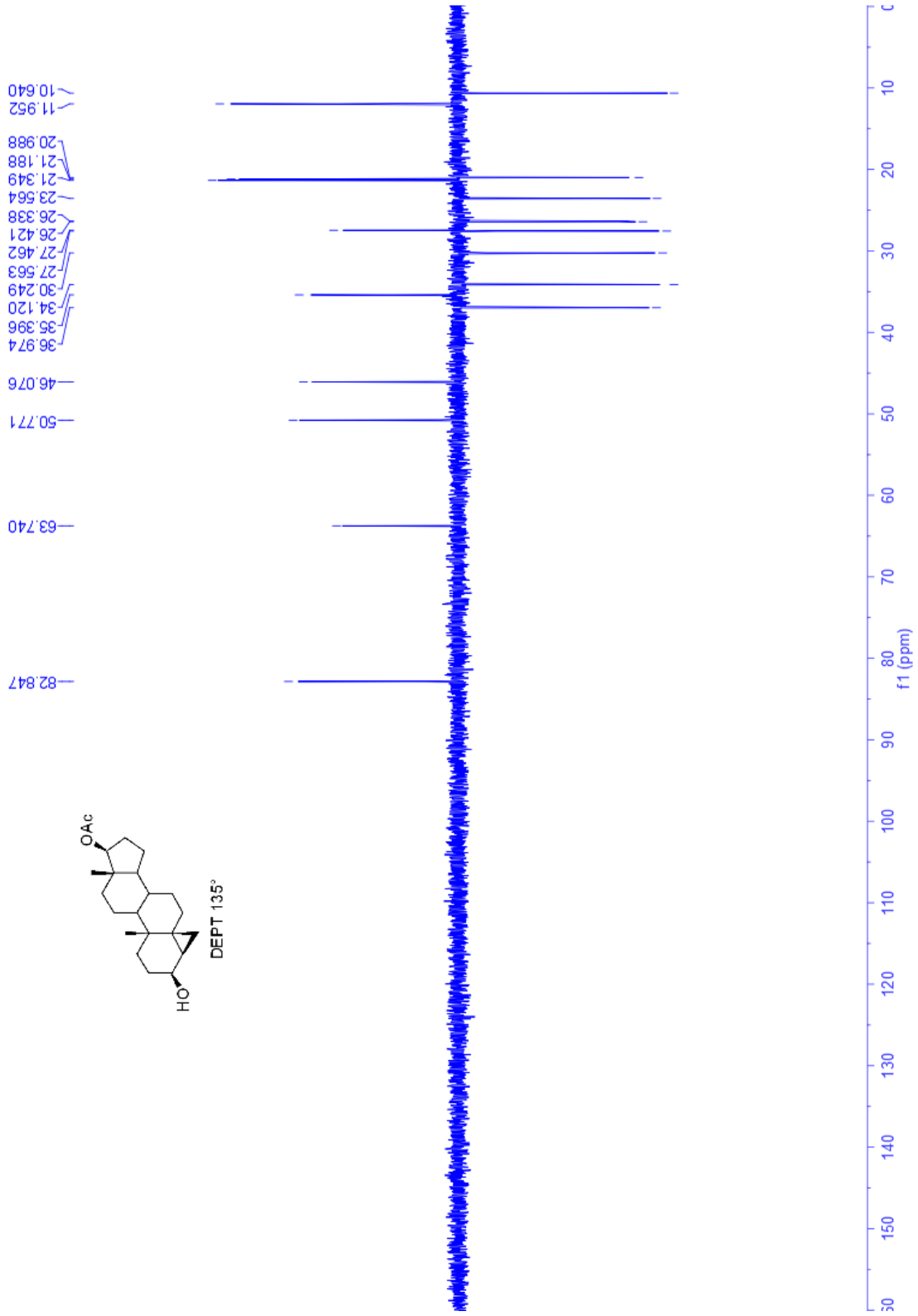

11.265
14.702
15.327
16.886
18.742
26.594
74.117
76.683
77.000
77.318
121.312
126.297
126.859
131.942
135.800
143.557
HO
^{13}C NMR 100MHz $CDCl_3$
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
f1 (ppm)


131.945
126.858
126.297
121.308
74.117
26.589
18.739
16.886
15.325
14.700
11.262
HO
DEPT 135°
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

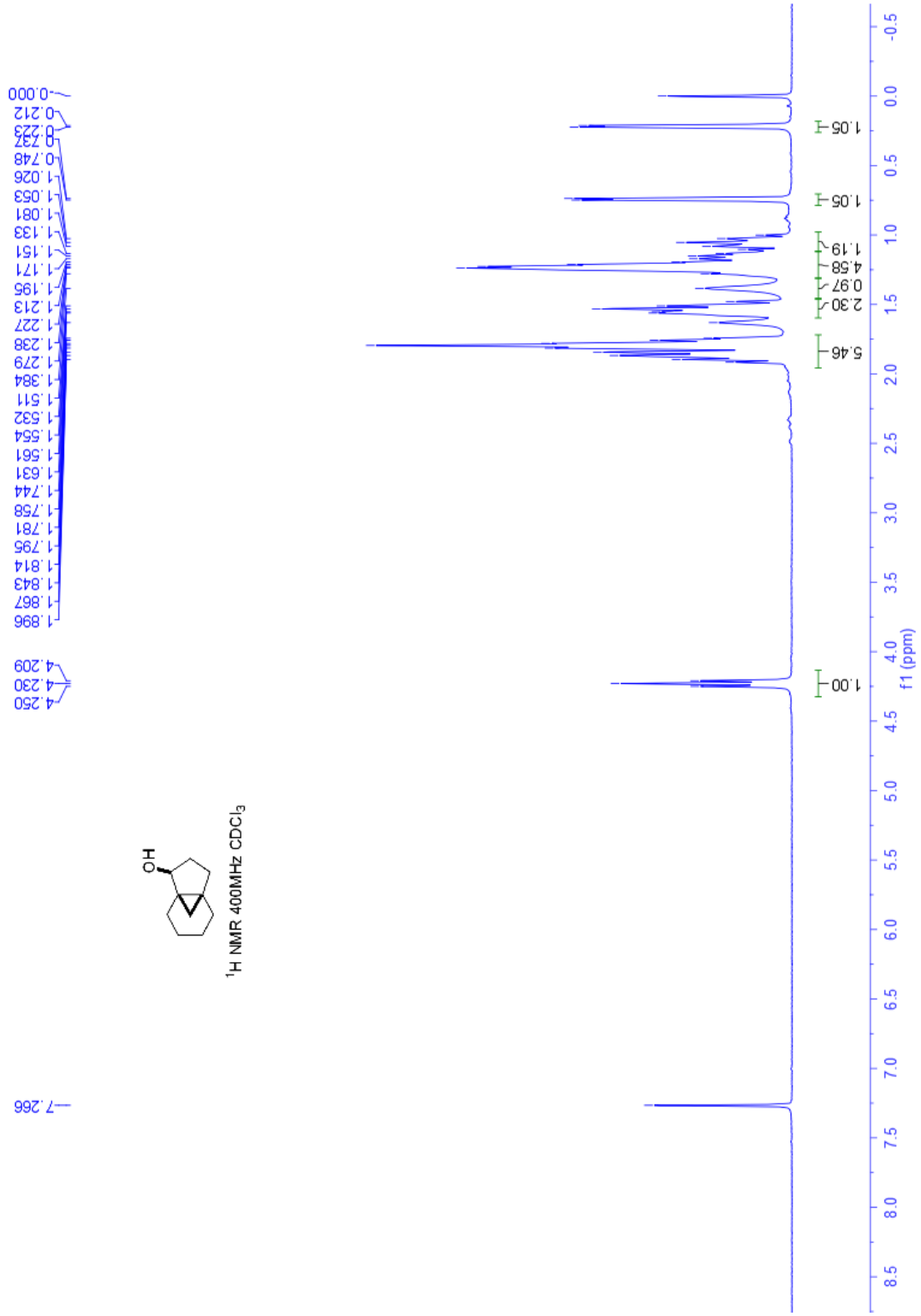
Compound 3p.

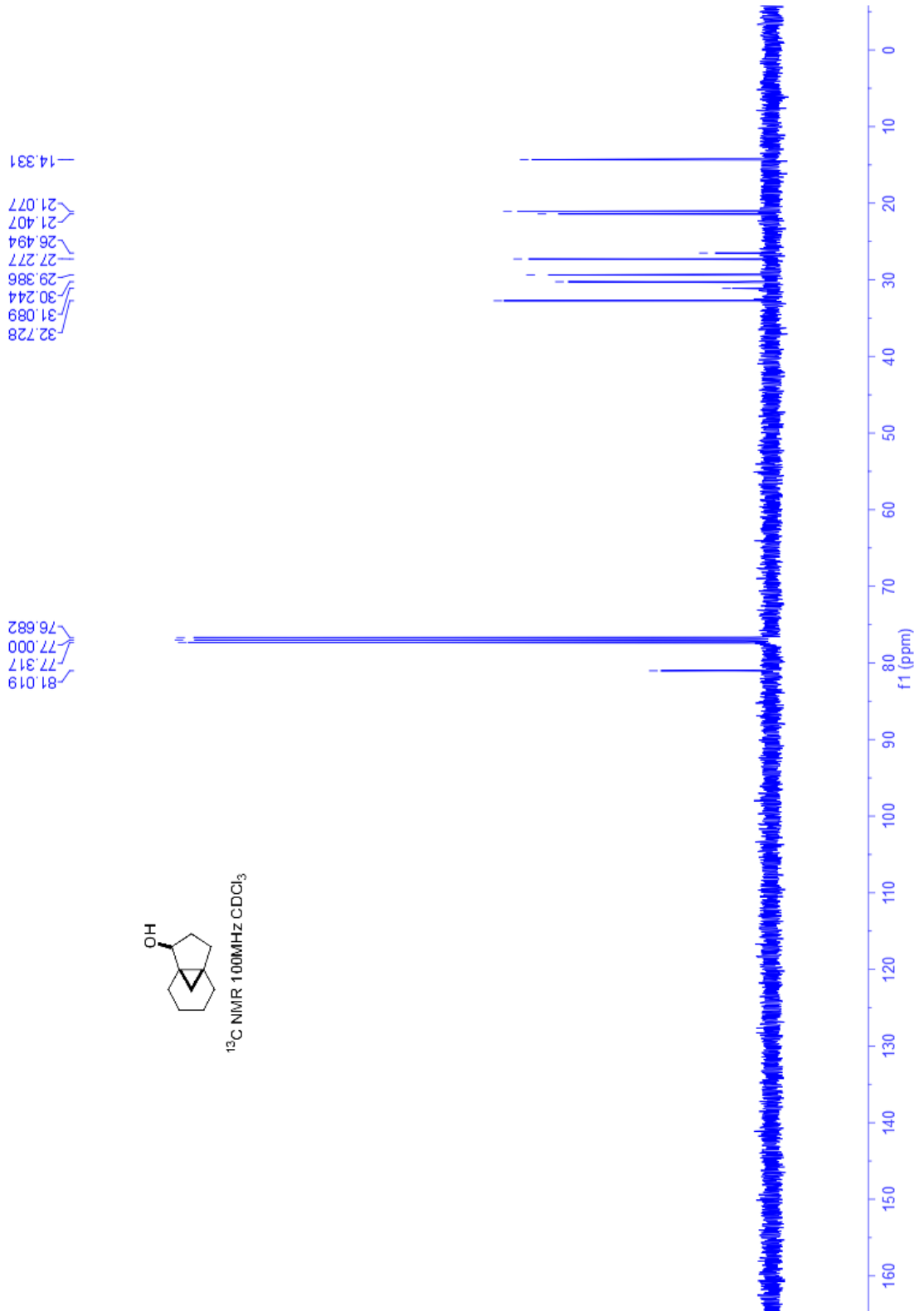


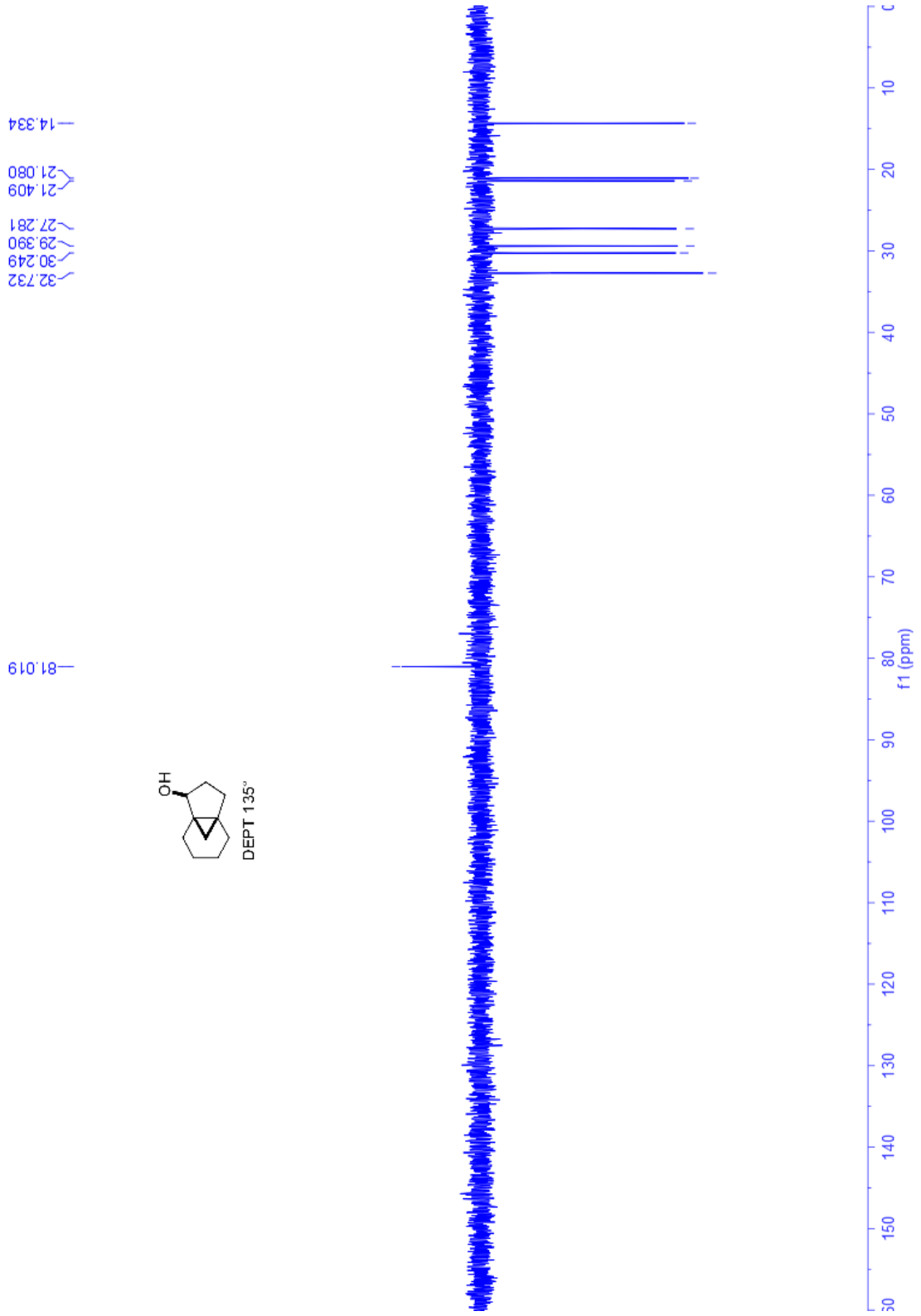

171.220
82.846
77.318
77.000
76.683
63.740
50.775
46.083
42.396
36.976
35.399
34.121
33.100
30.248
27.563
27.464
26.425
26.339
23.563
21.346
21.182
20.989
11.950
10.639
OAc
HO
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


10.640
11.952
20.988
21.188
21.349
23.564
26.338
26.421
27.462
27.563
30.249
34.120
35.396
36.974
46.076
50.771
63.740
82.847
OAc
HO
DEPT 135°
f1 (ppm)

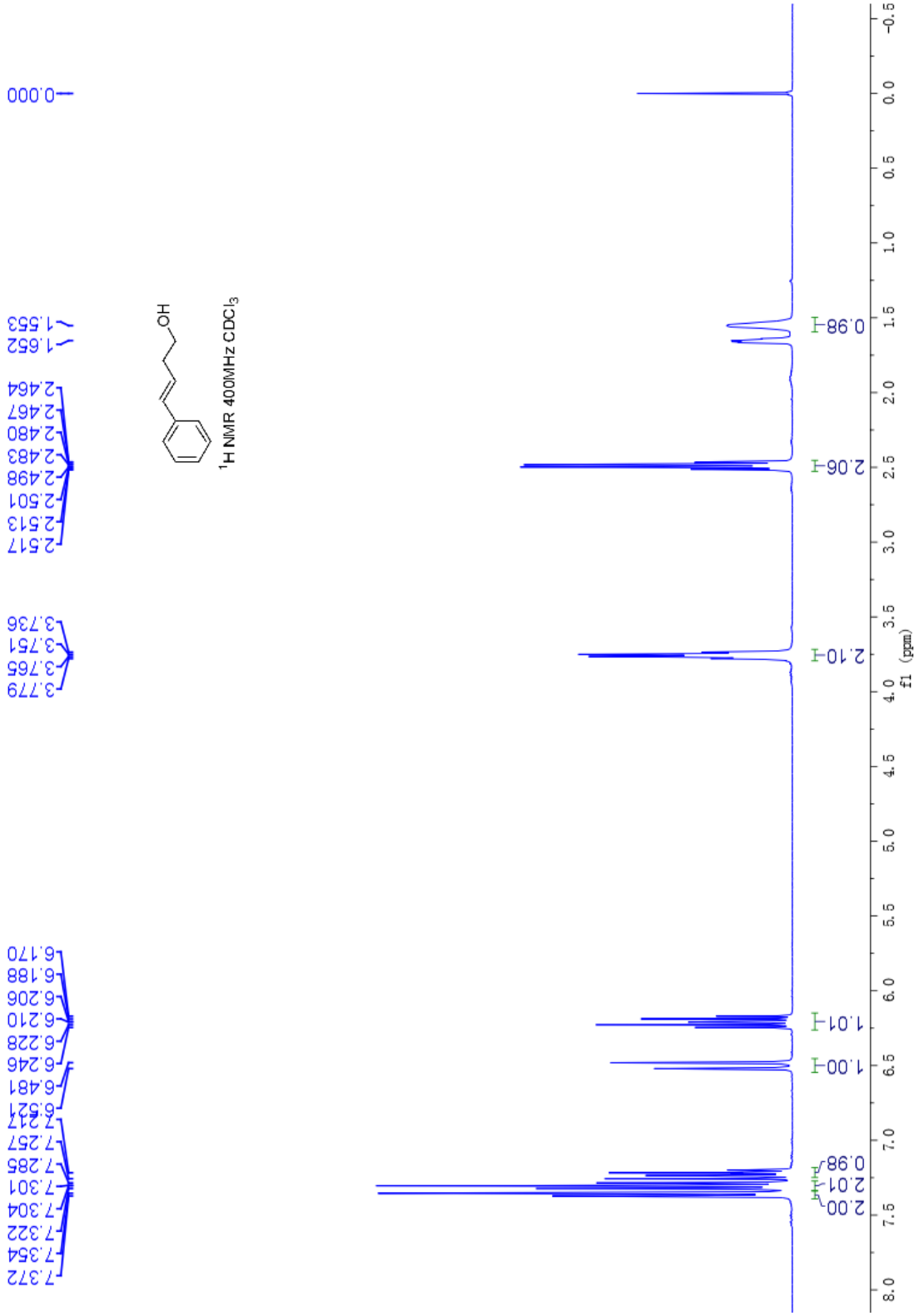
Compound 3q.

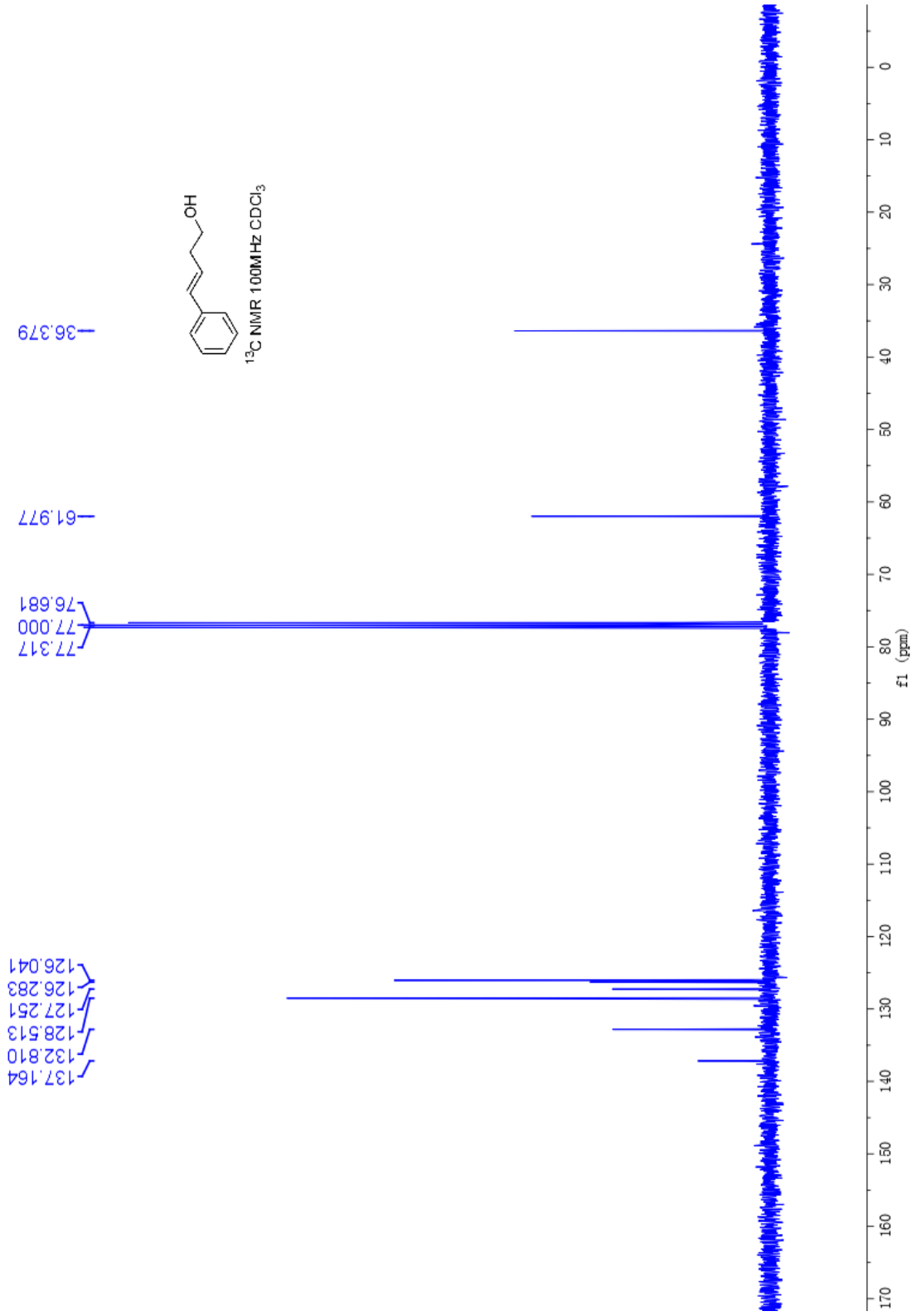



14.331
21.077
21.407
26.494
27.277
29.386
30.244
31.089
32.728
76.682
77.000
77.317
81.019
OH
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)

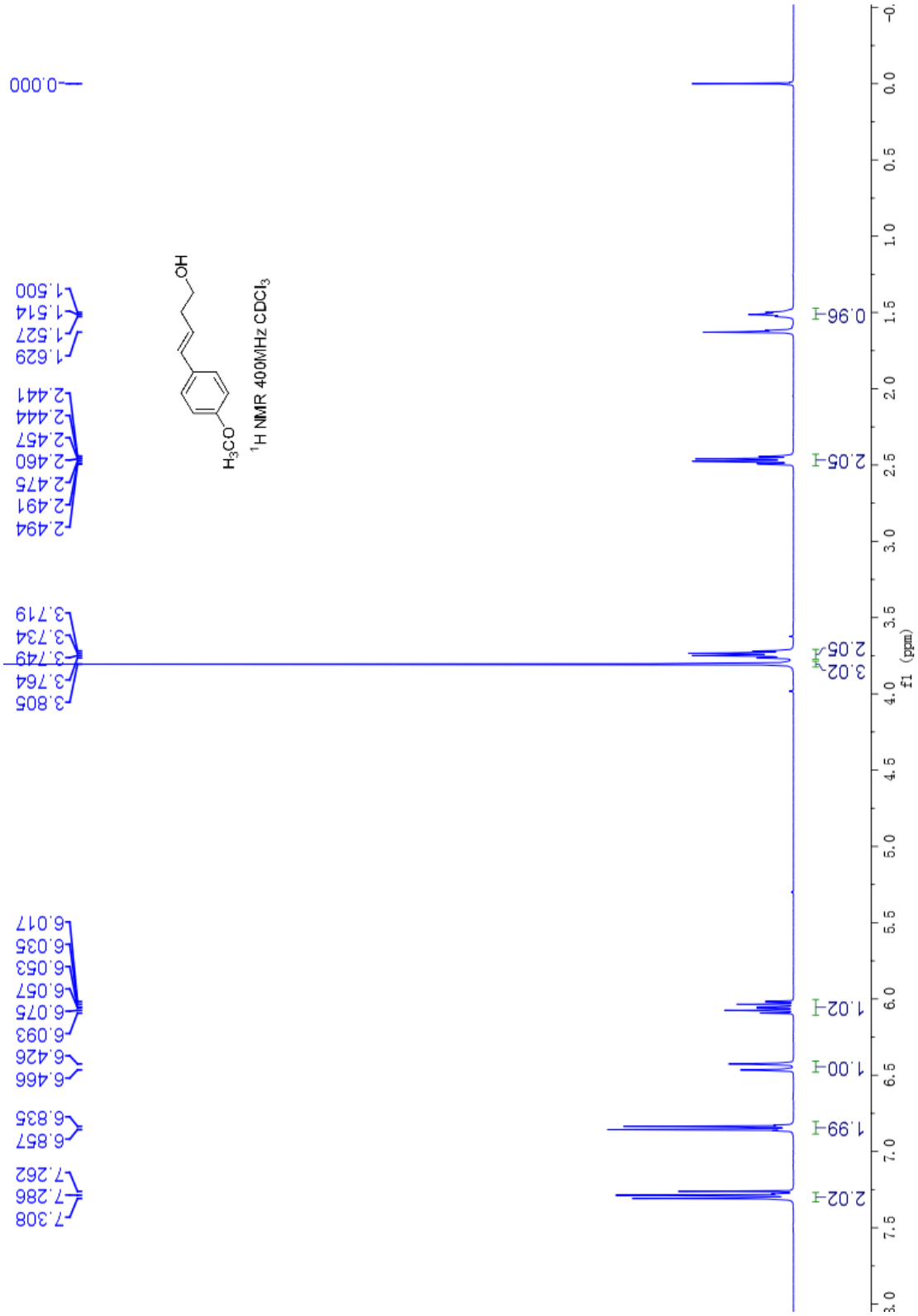

14.334
21.080
21.409
27.281
29.390
30.249
32.732
81.019
OH
DEPT 135°
f1 (ppm)

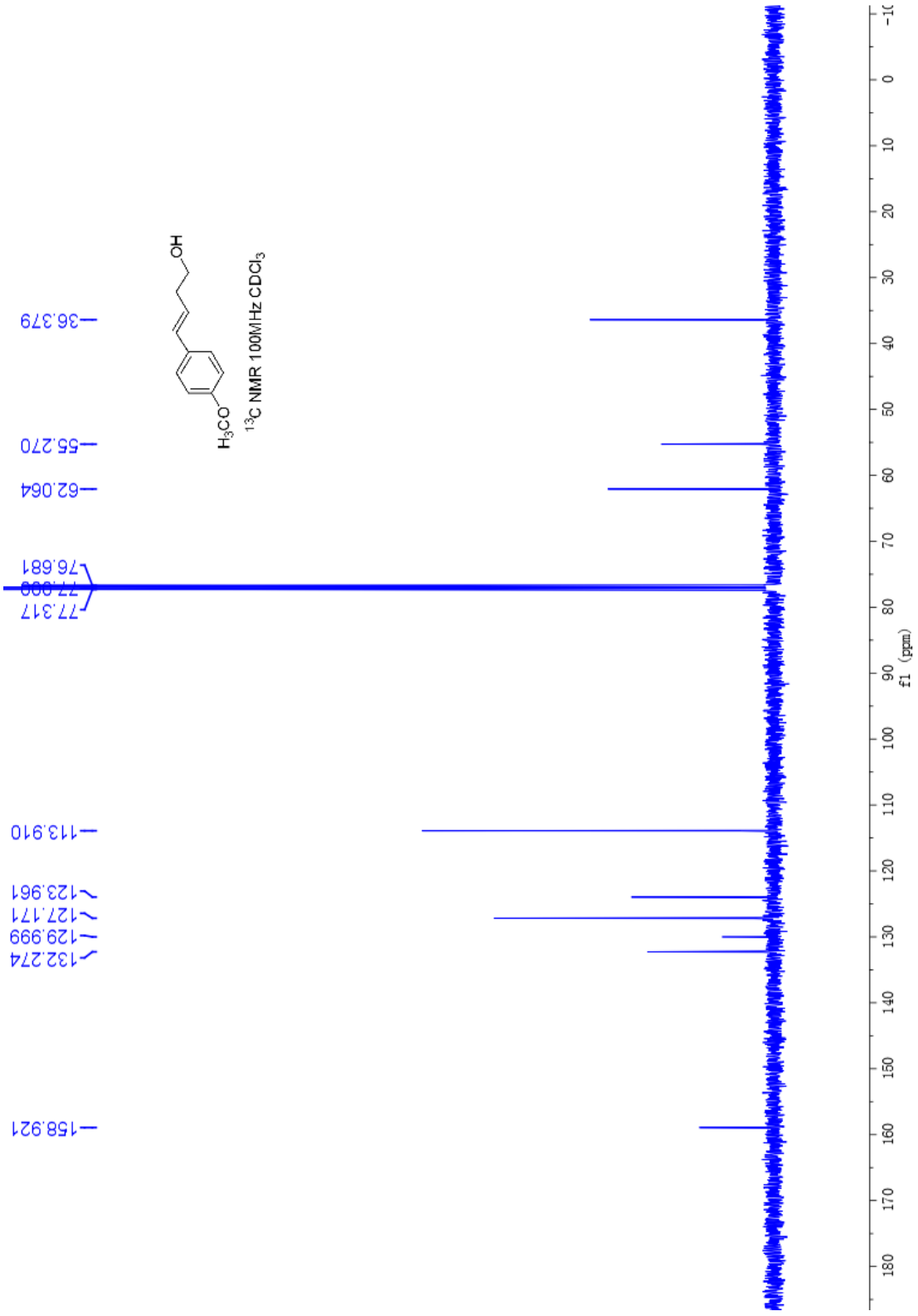
cinnamyl alcohol (2a).



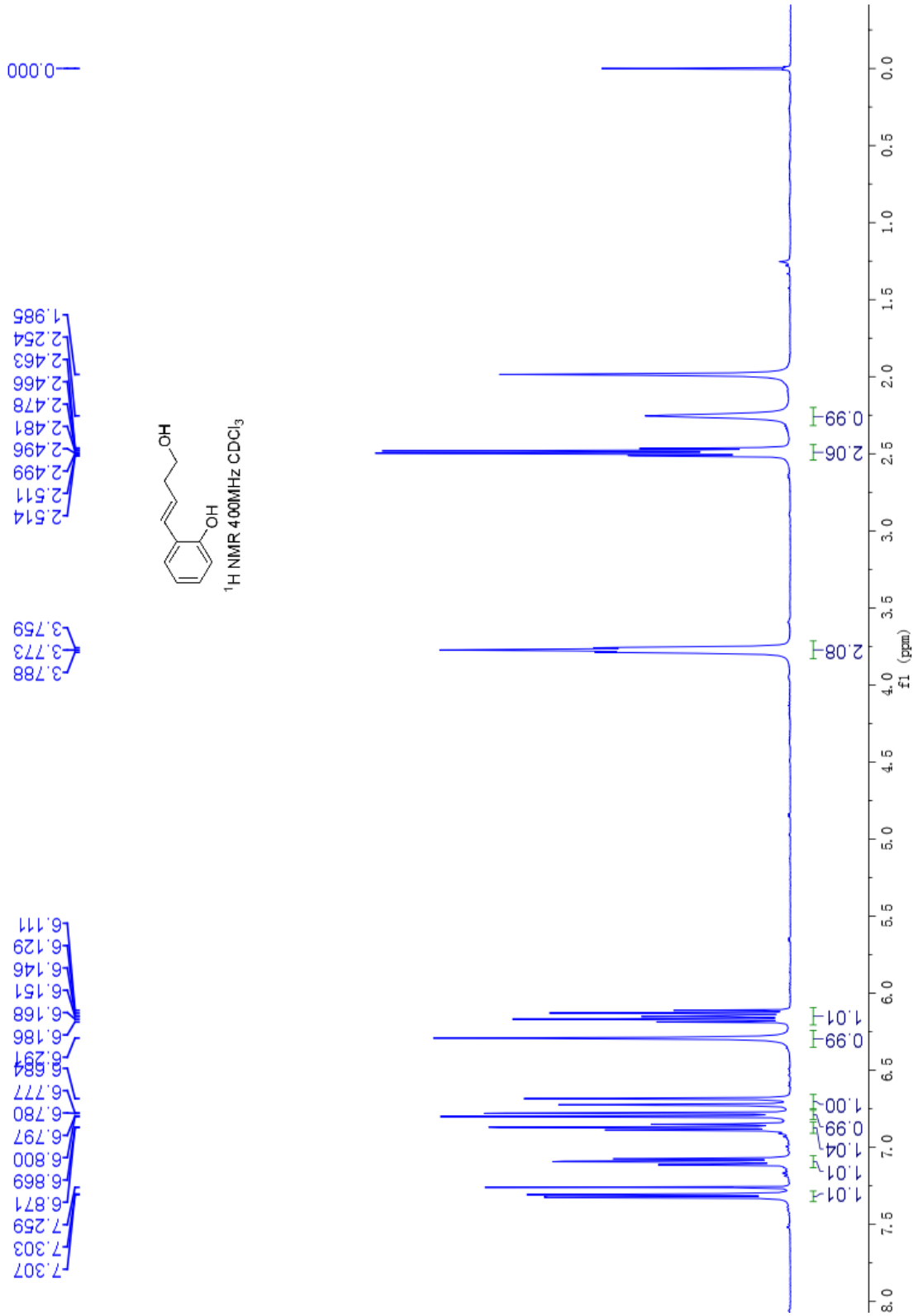

OH
^{13}C NMR 100MHz $CDCl_3$
36.379
61.977
76.681
77.000
77.317
126.041
126.283
127.251
128.513
132.810
137.164
f1 (ppm)

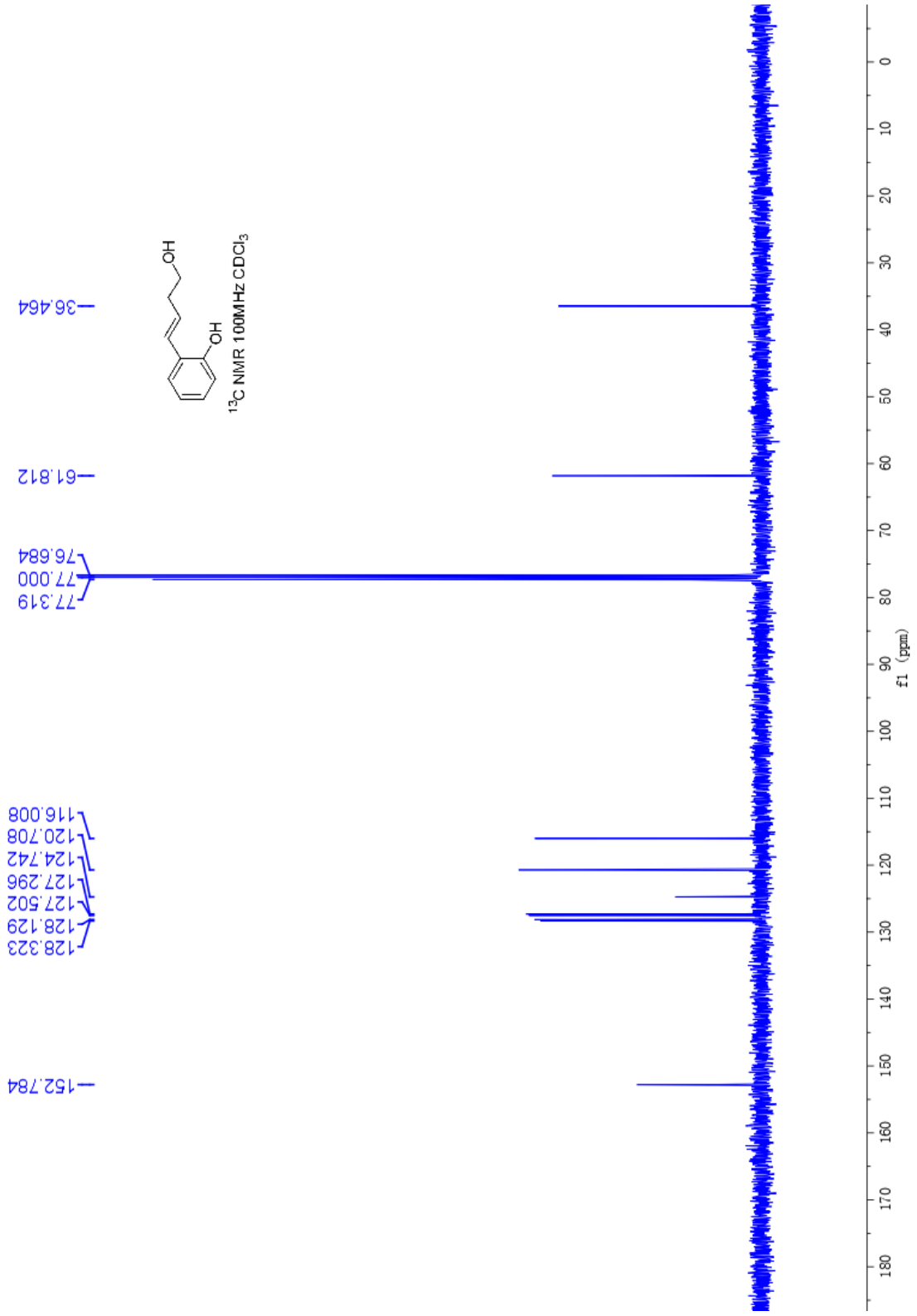
(*E*)-4-(4-methoxyphenyl)but-3-en-1-ol (2b).



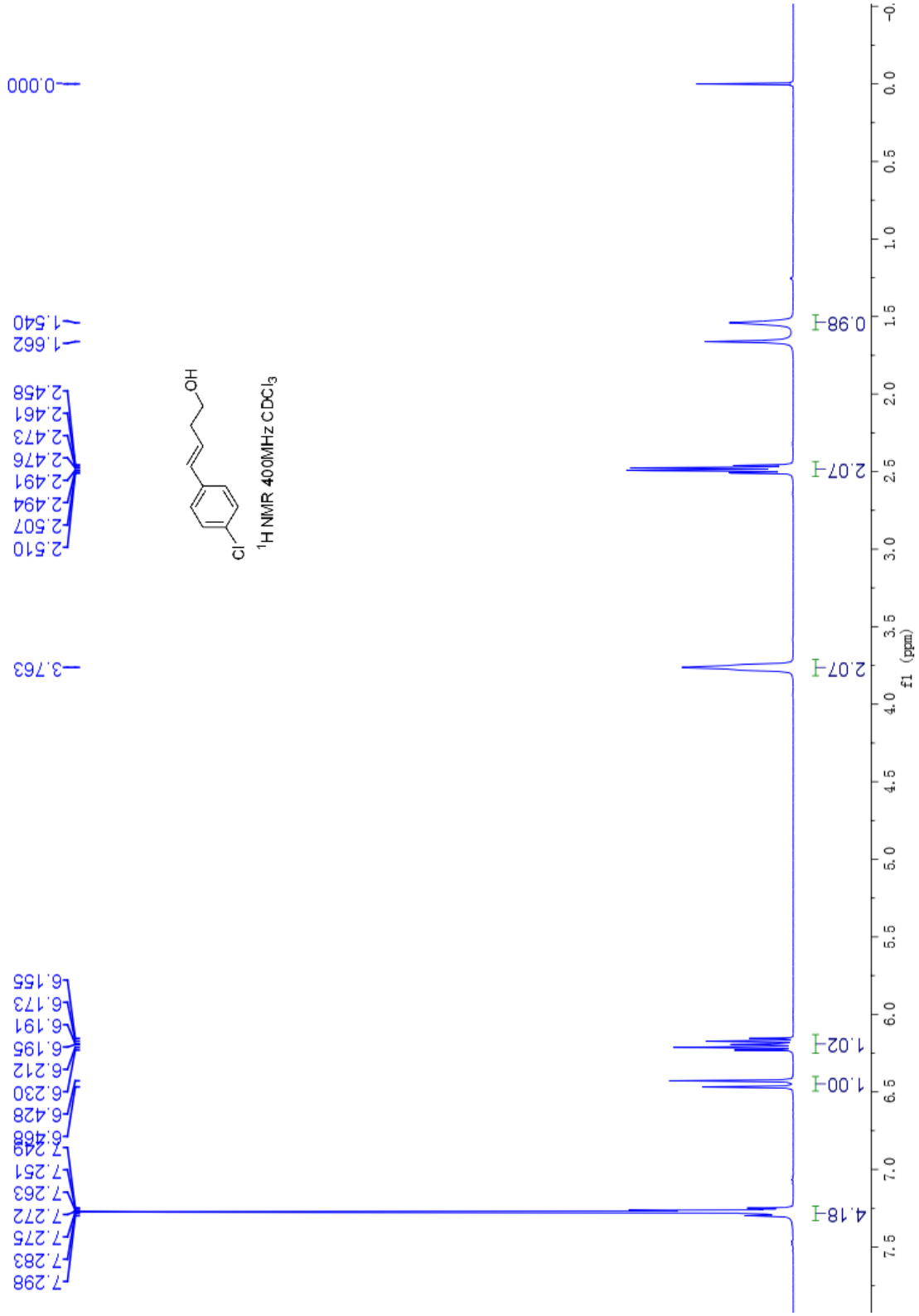

OH
H_3CO
^{13}C NMR 100MHz $CDCl_3$
36.379
55.270
62.064
76.681
77.000
77.317
113.910
123.961
127.171
129.999
132.274
158.921
f1 (ppm)

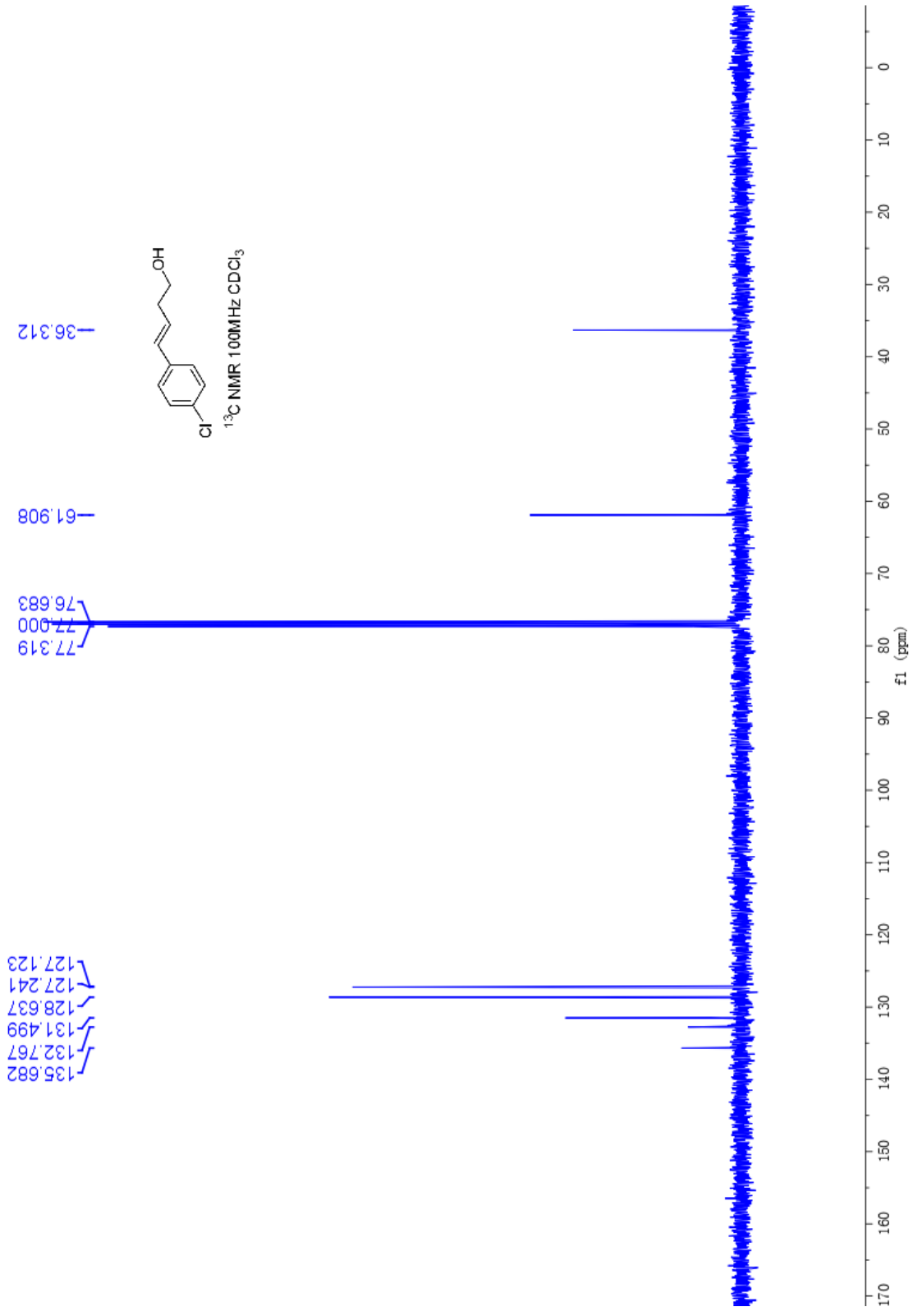
(*E*)-2-(4-hydroxybut-1-en-1-yl)phenol (2c).



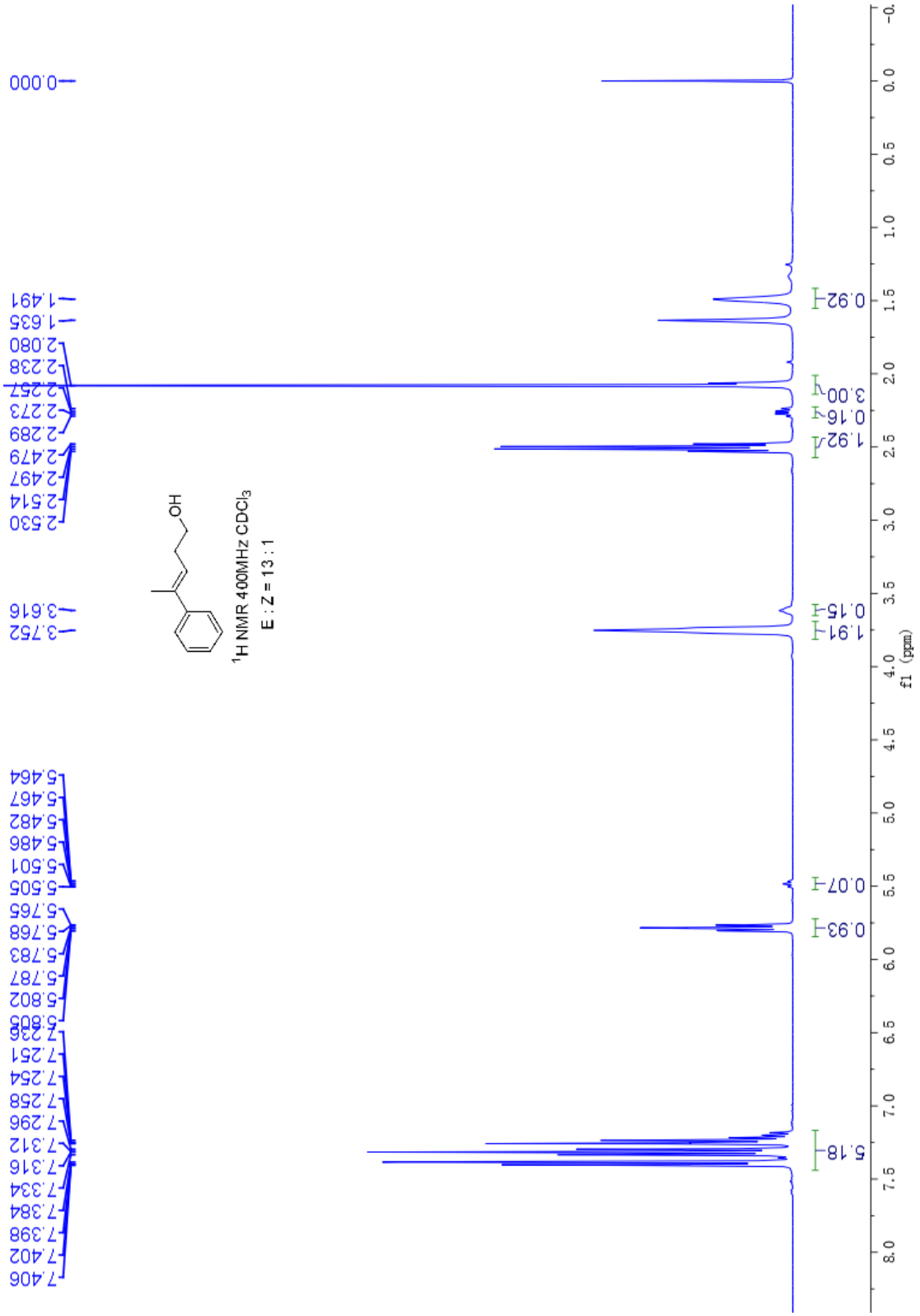

OH
OH
^{13}C NMR 100MHz $CDCl_3$
36.464
61.812
76.684
77.000
77.319
116.008
120.708
124.742
127.296
127.502
128.129
128.323
152.784
f1 (ppm)

(*E*)-4-(4-chlorophenyl)but-3-en-1-ol (2d).

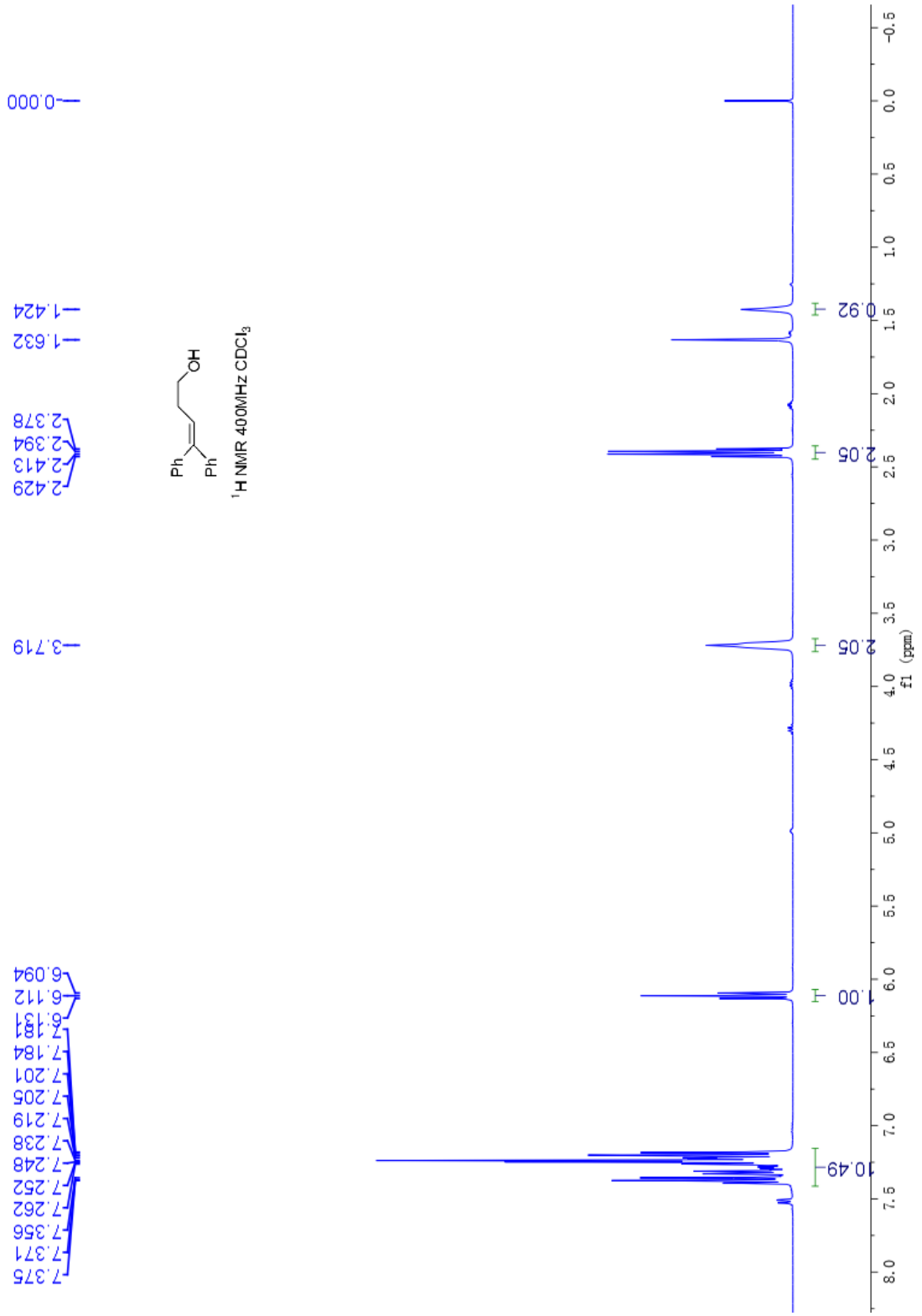


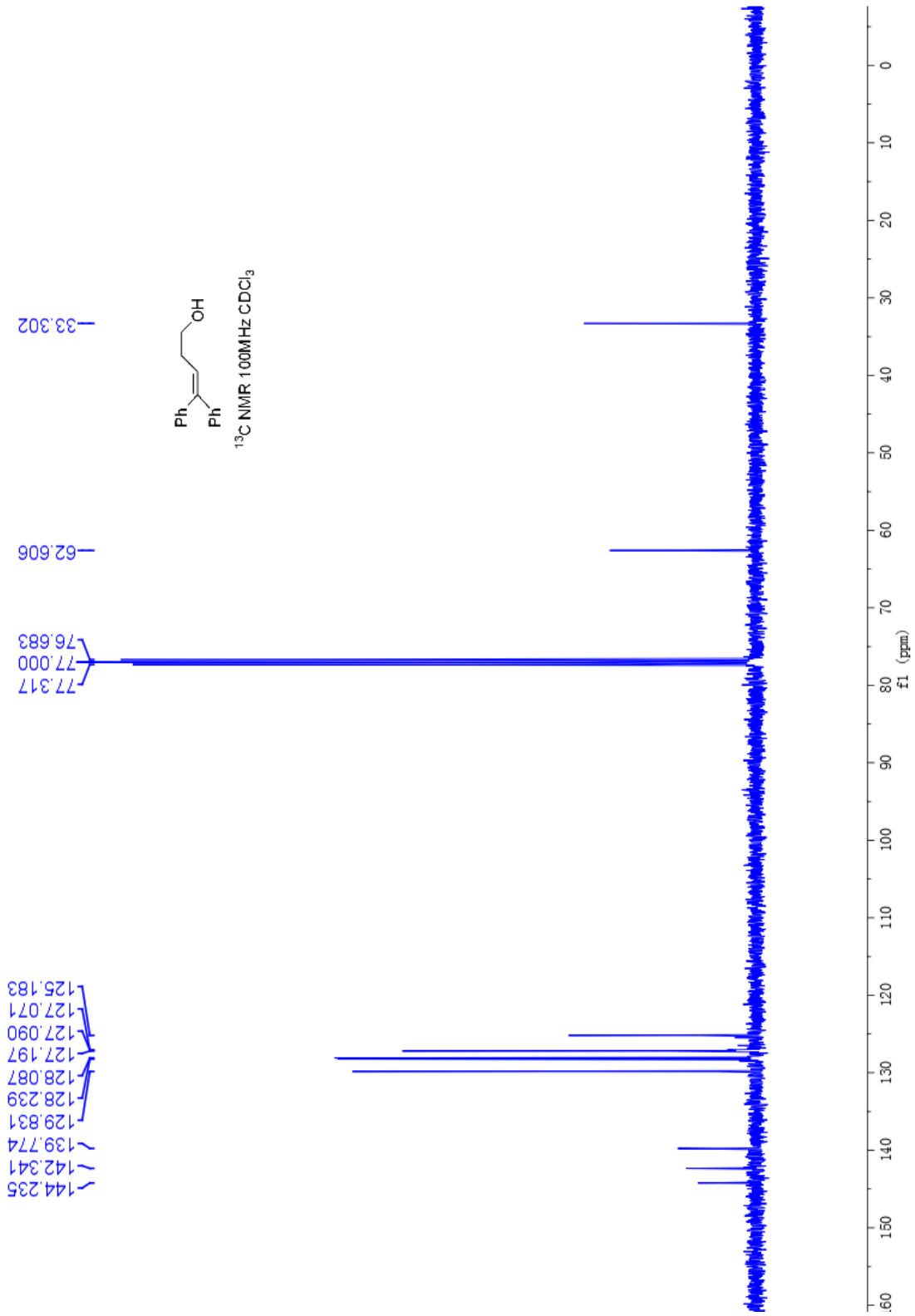

OH
Cl
^{13}C NMR 100MHz $CDCl_3$
36.312
61.908
76.683
77.000
77.319
127.123
127.241
128.637
131.499
132.767
135.682
f1 (ppm)

4-phenylpent-3-en-1-ol (2e).

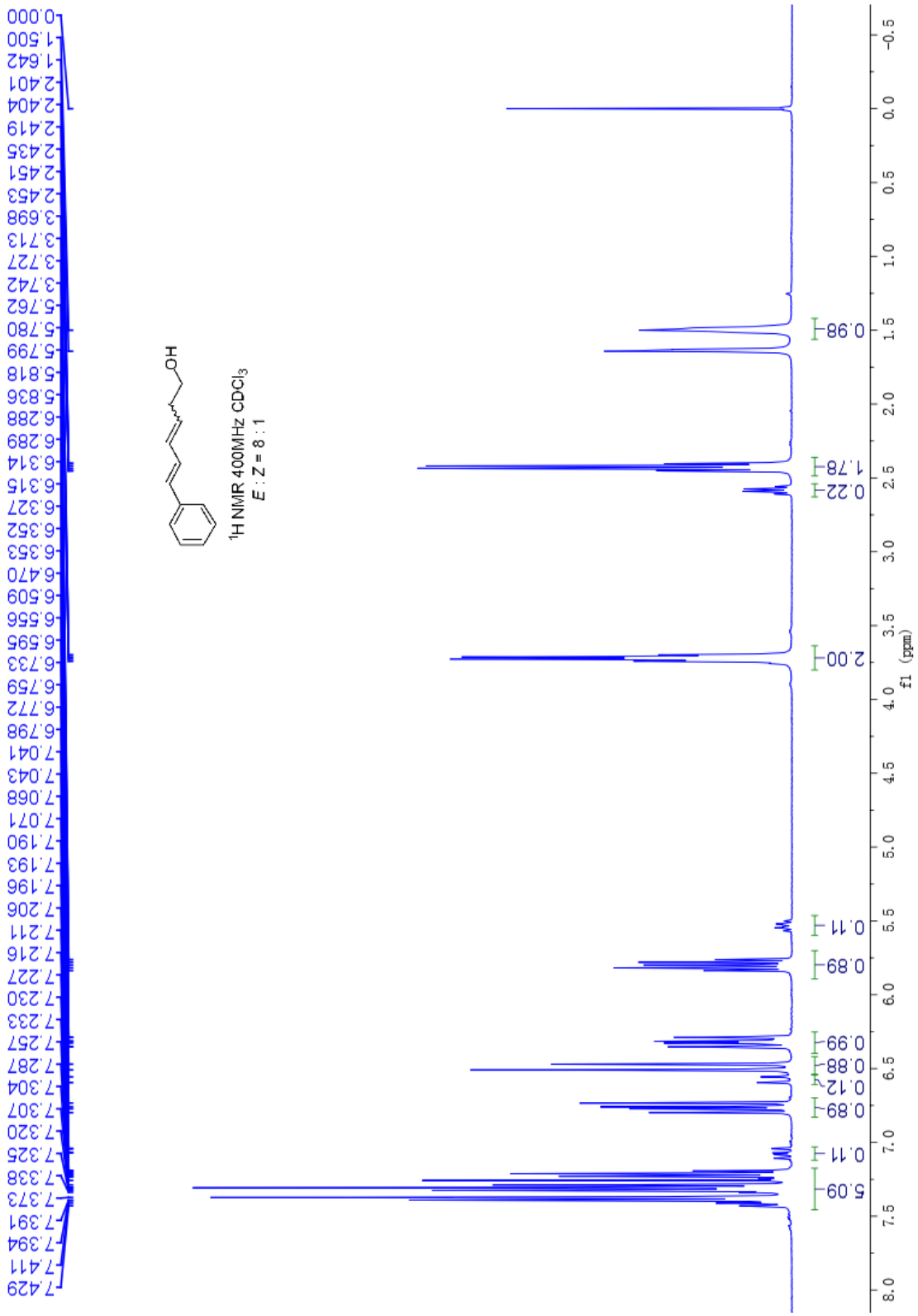


4,4-diphenylbut-3-en-1-ol (2f).

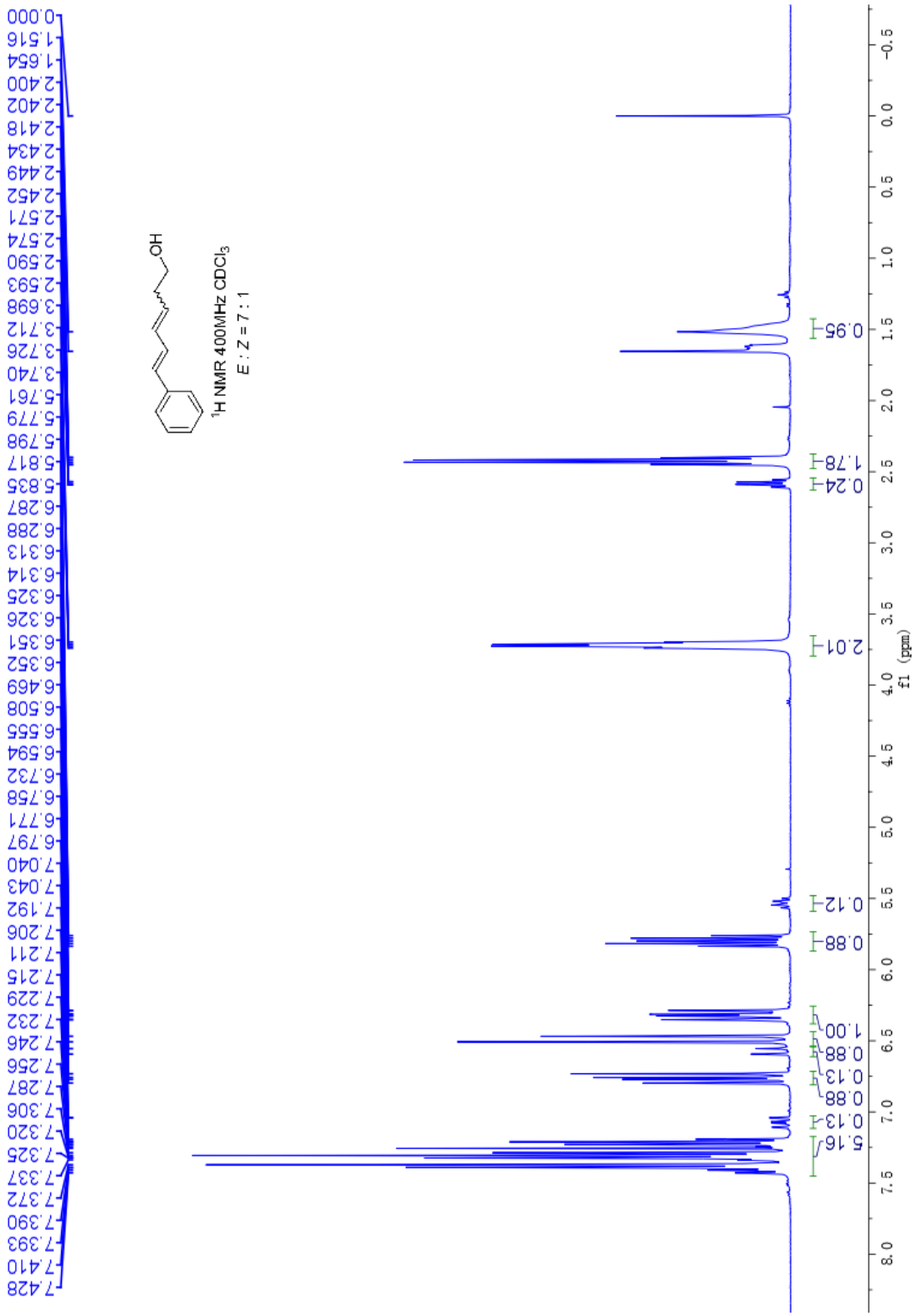



33.302
62.606
76.683
77.000
77.317
125.183
127.071
127.090
127.197
128.087
128.239
129.831
139.774
142.341
144.235
OH
Ph
Ph
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)

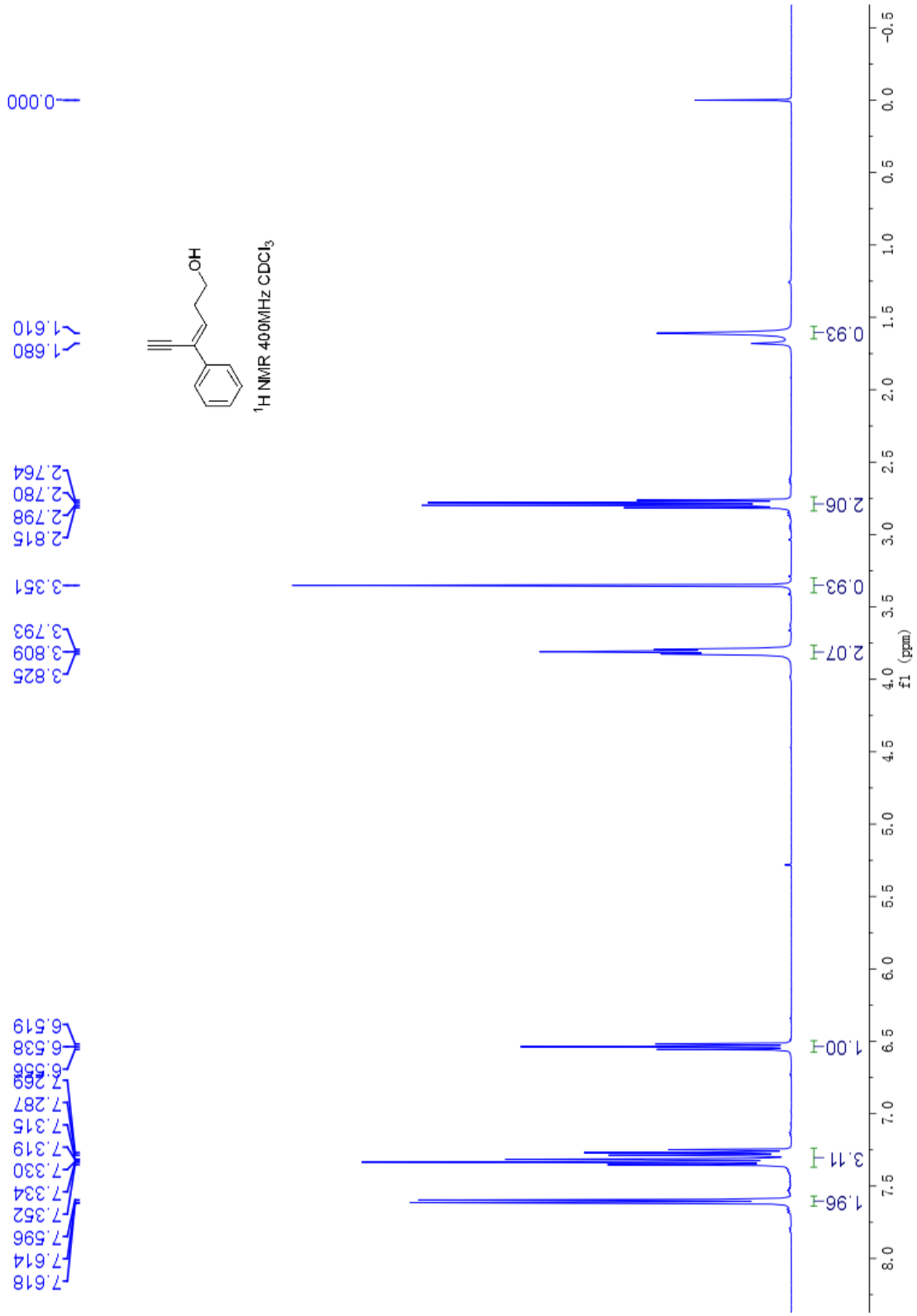
6-phenylhexa-3(*E* or *Z*), 5(*E*)-dien-1-ol (2g). (*E* : *Z* = 8 : 1)

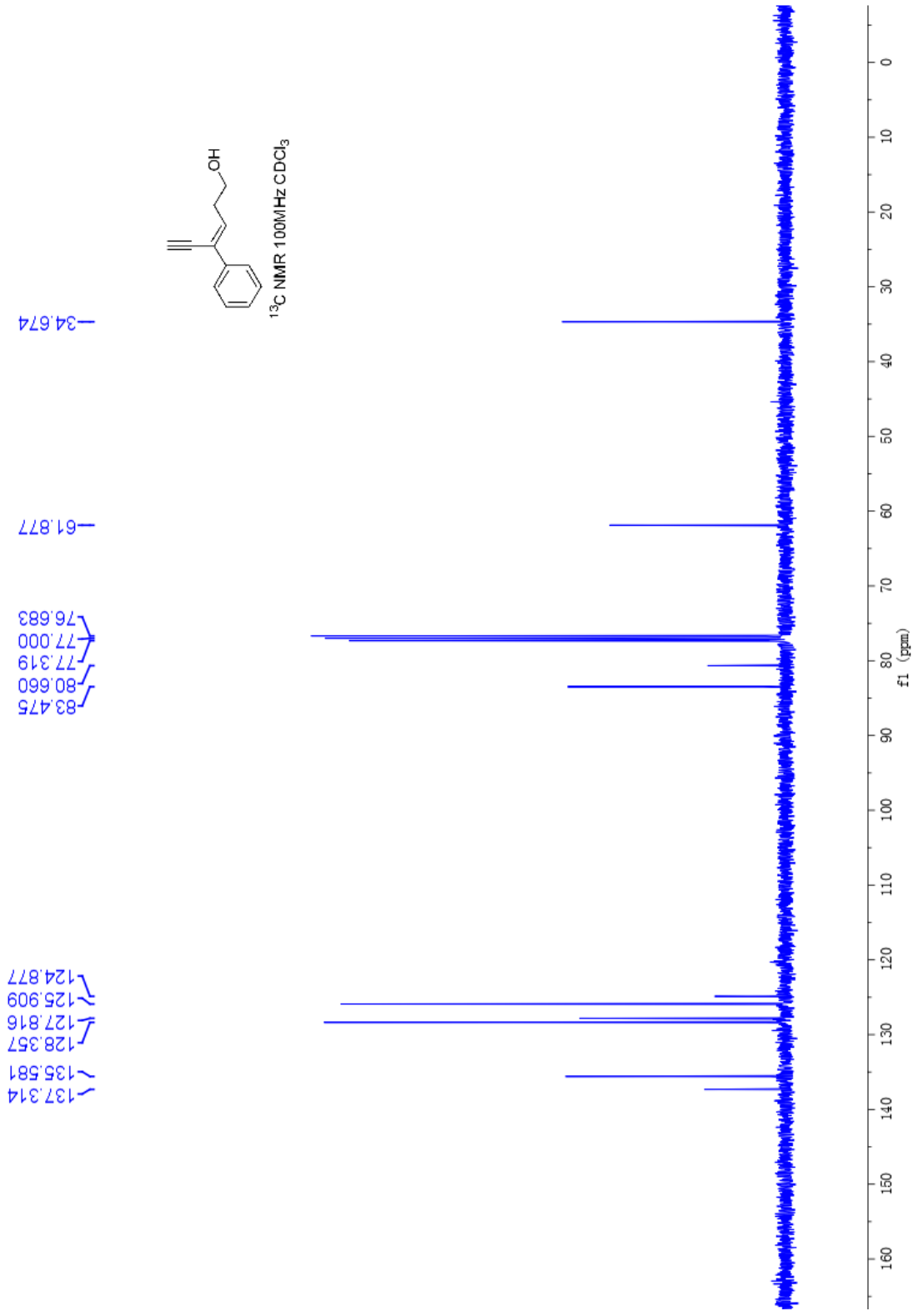


6-phenylhexa-3(*E* or *Z*), 5(*E*)-dien-1-ol (2h). (*E* : *Z* = 7 : 1)

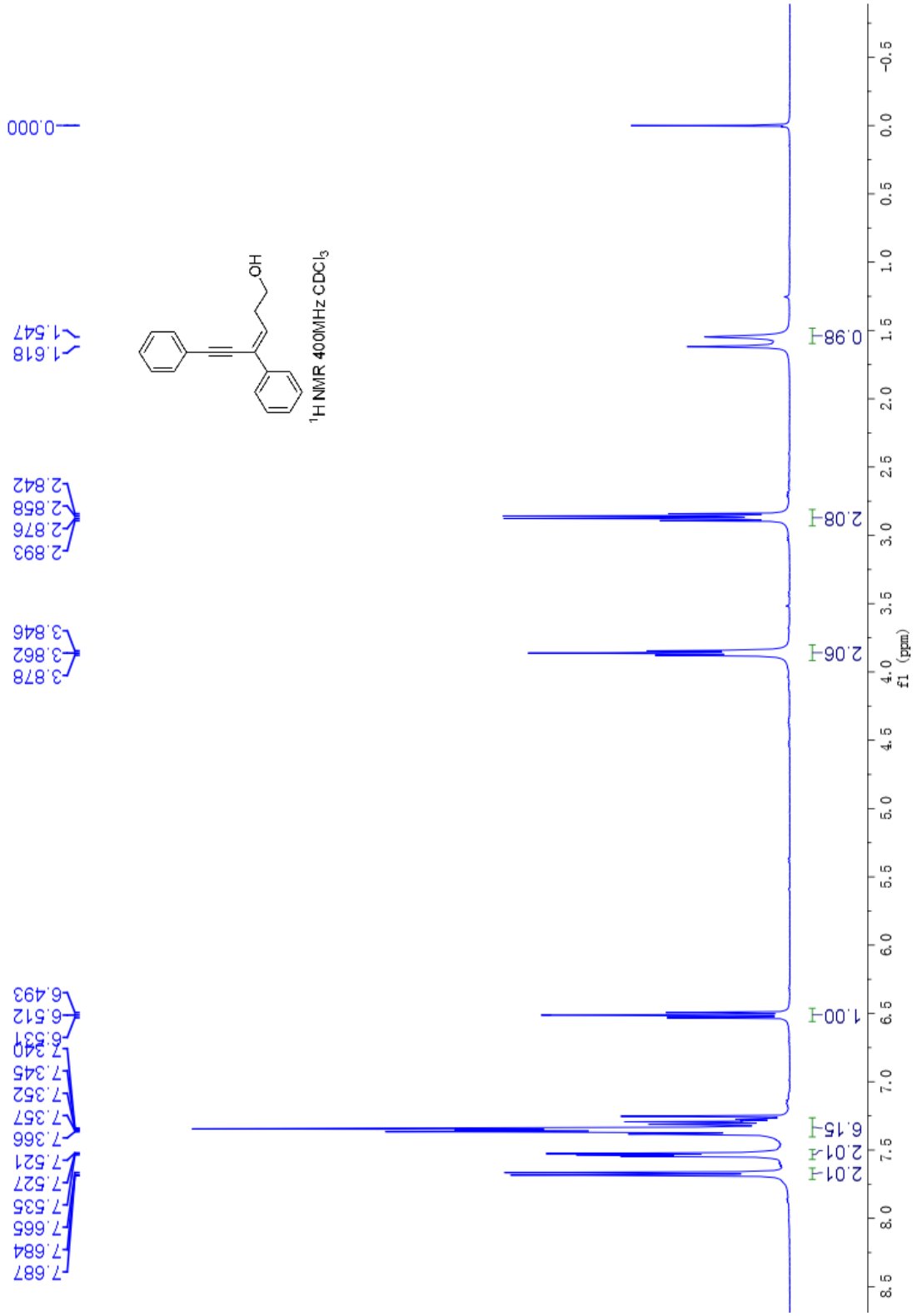


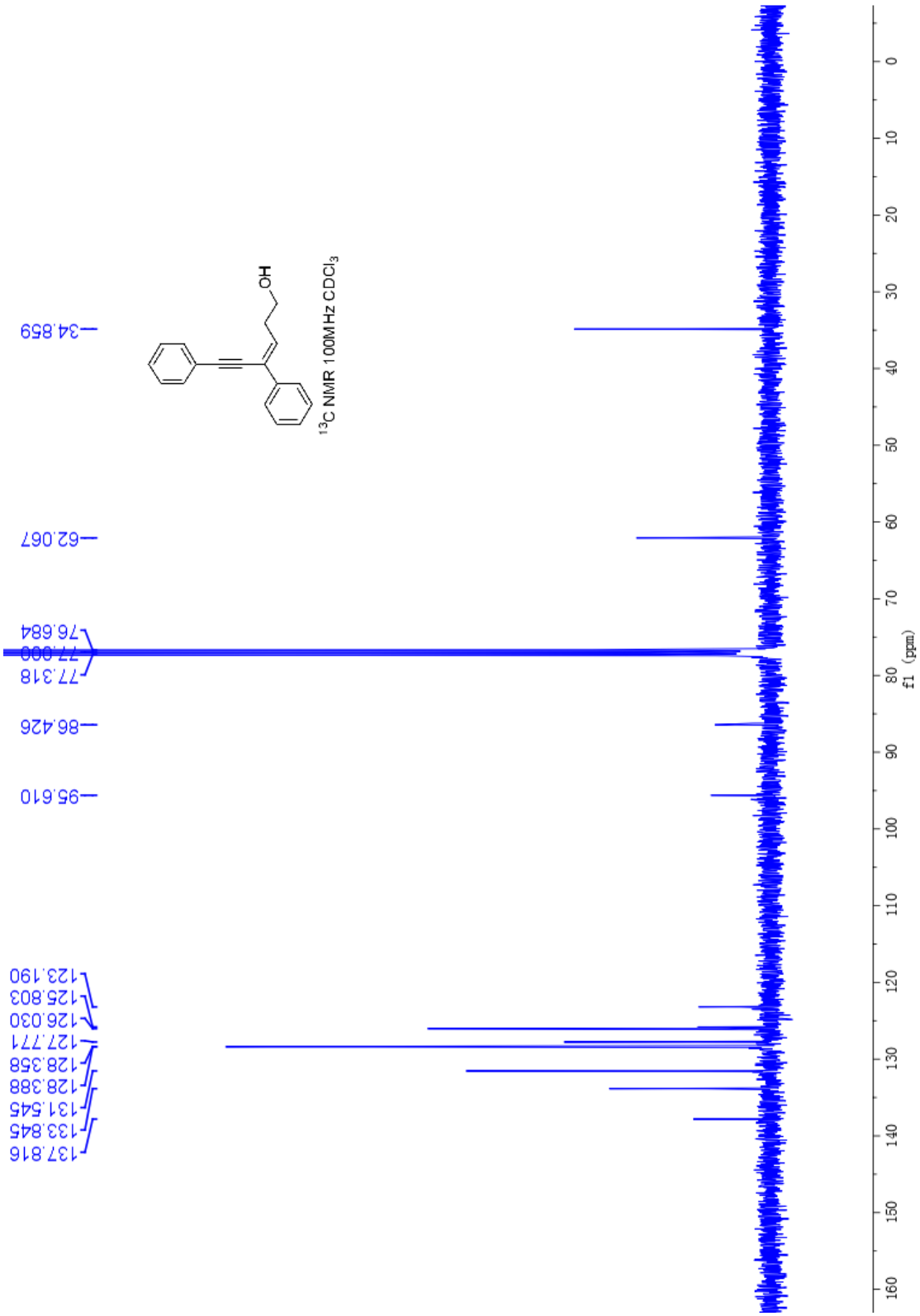
(*E*)-4-phenylhex-3-en-5-yn-1-ol (2i).



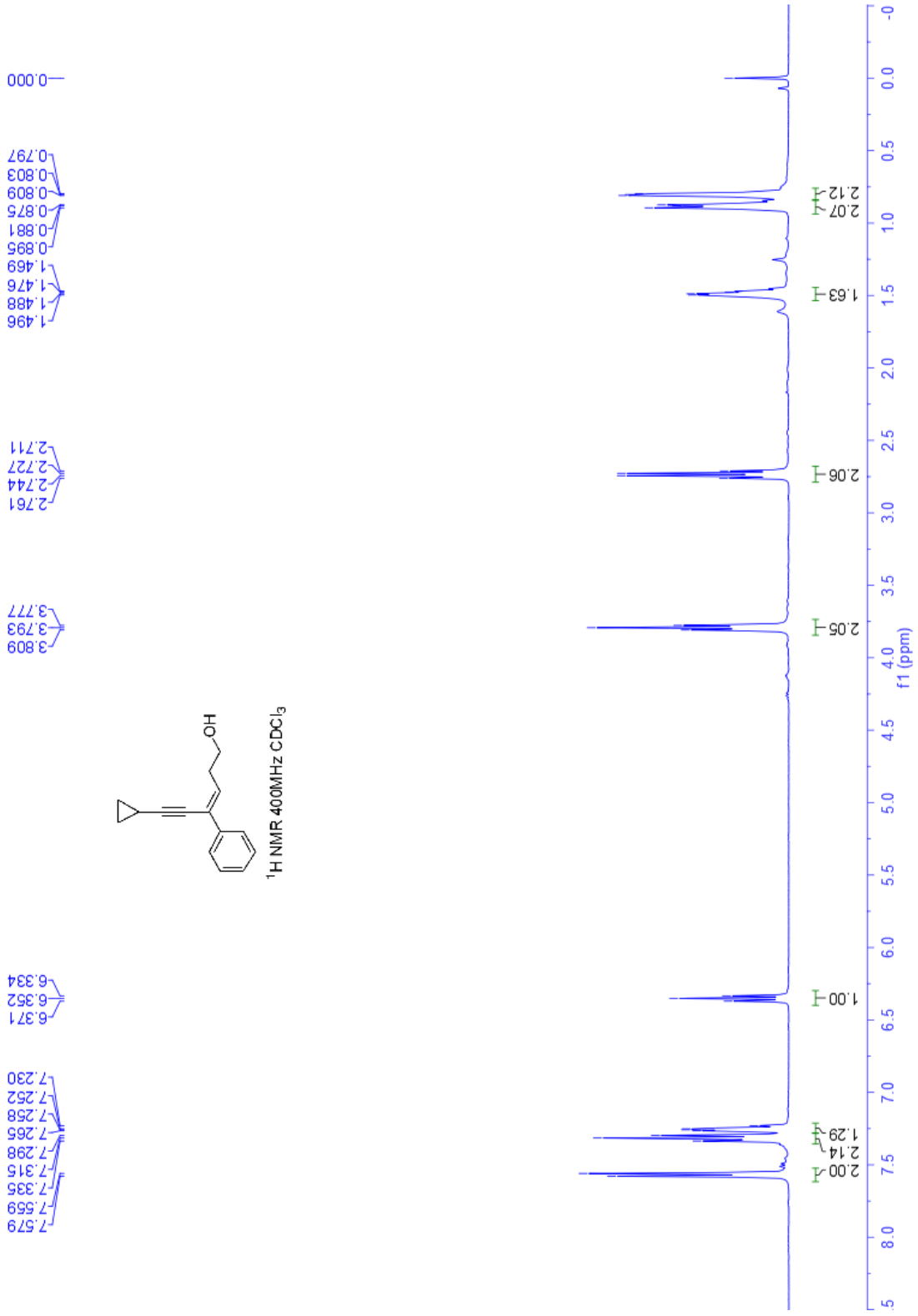

OH
^{13}C NMR 100MHz $CDCl_3$
34.674
61.877
76.683
77.000
77.319
80.660
83.475
124.877
125.909
127.816
128.357
135.581
137.314
f1 (ppm)

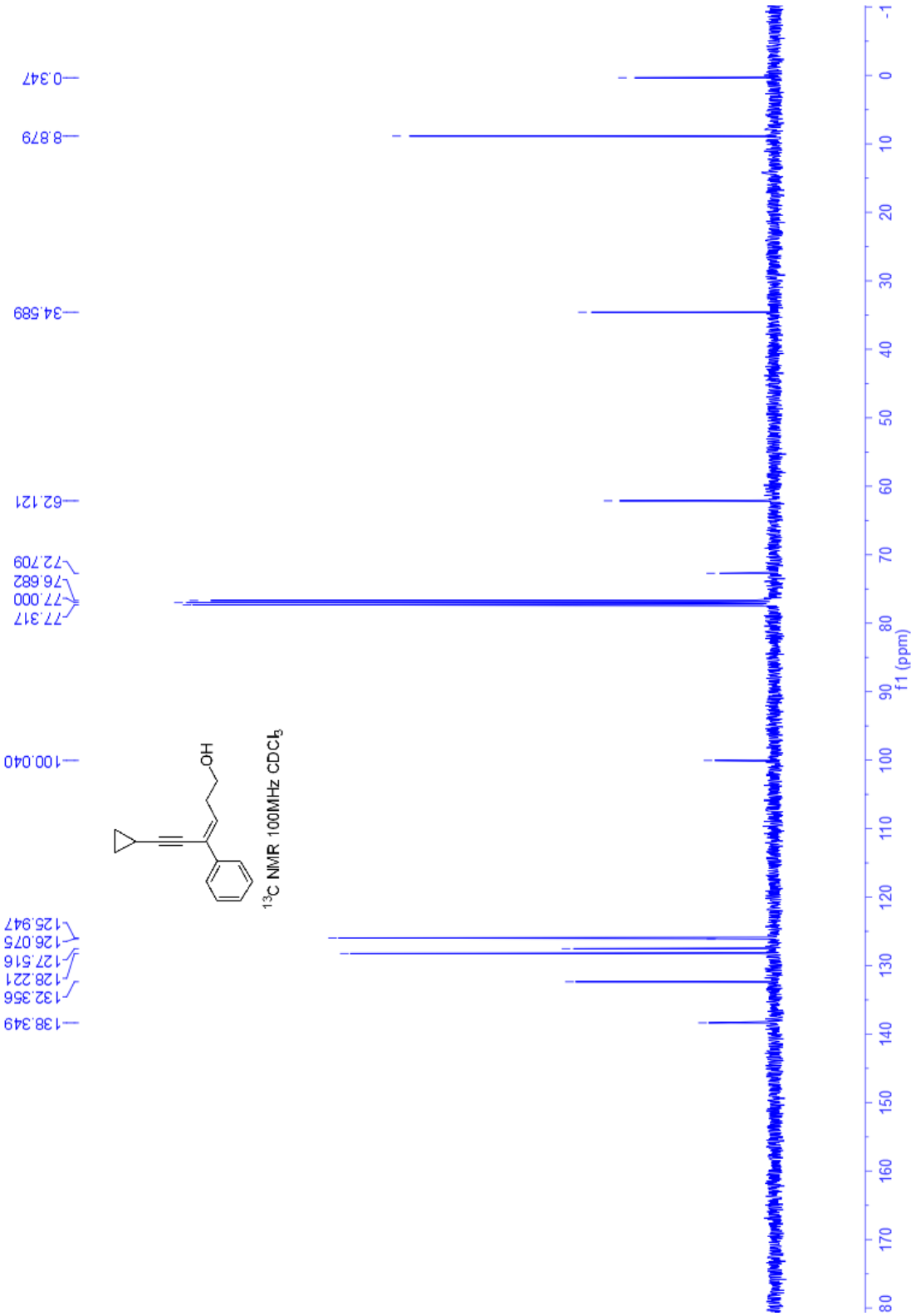
(*Z*)-4,6-diphenylhex-3-en-5-yn-1-ol (2j).



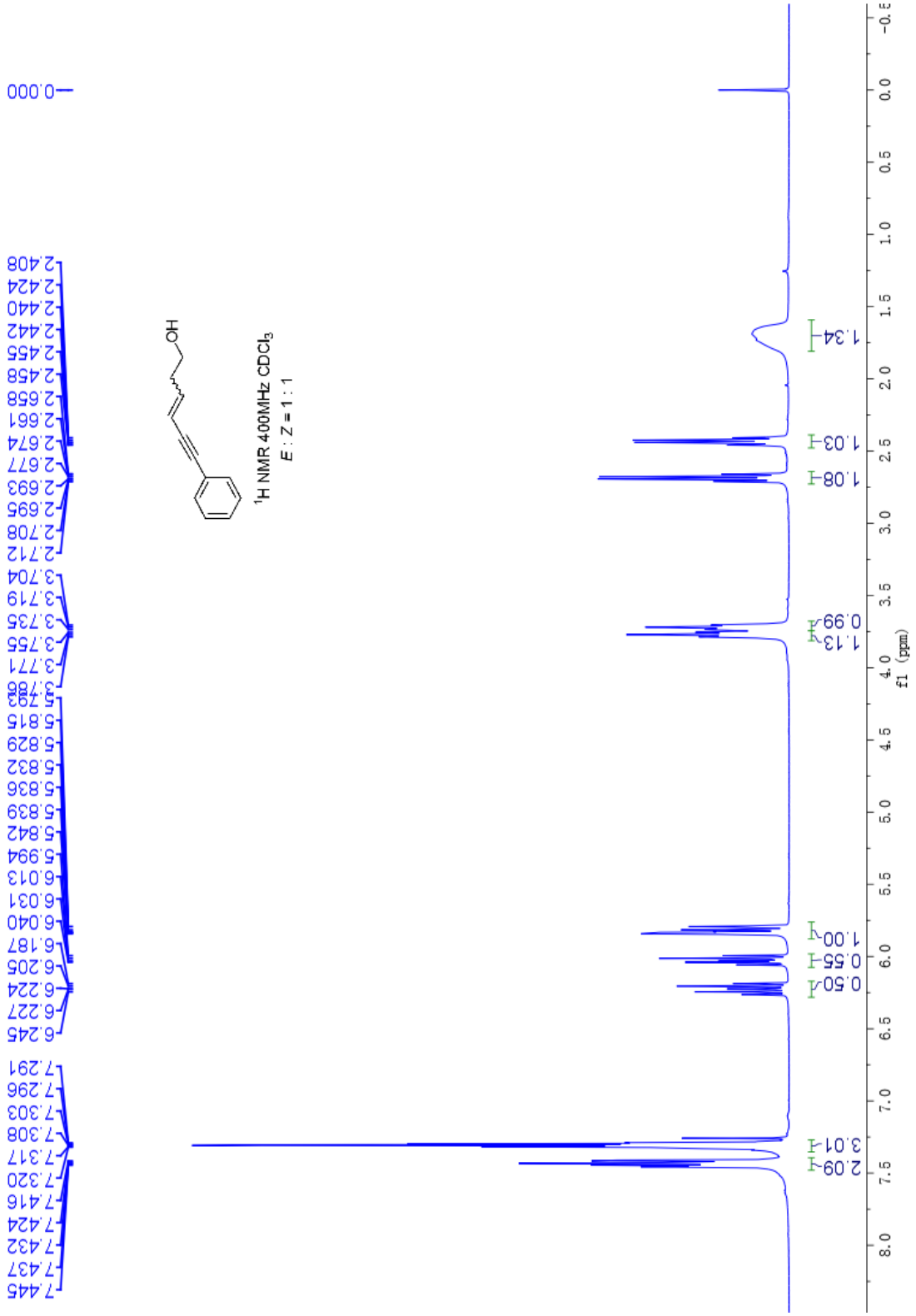

OH
^{13}C NMR 100MHz $CDCl_3$
34.859
62.067
76.684
77.009
77.318
86.426
95.610
123.190
125.803
126.030
127.771
128.358
128.388
131.545
133.845
137.816
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
f1 (ppm)

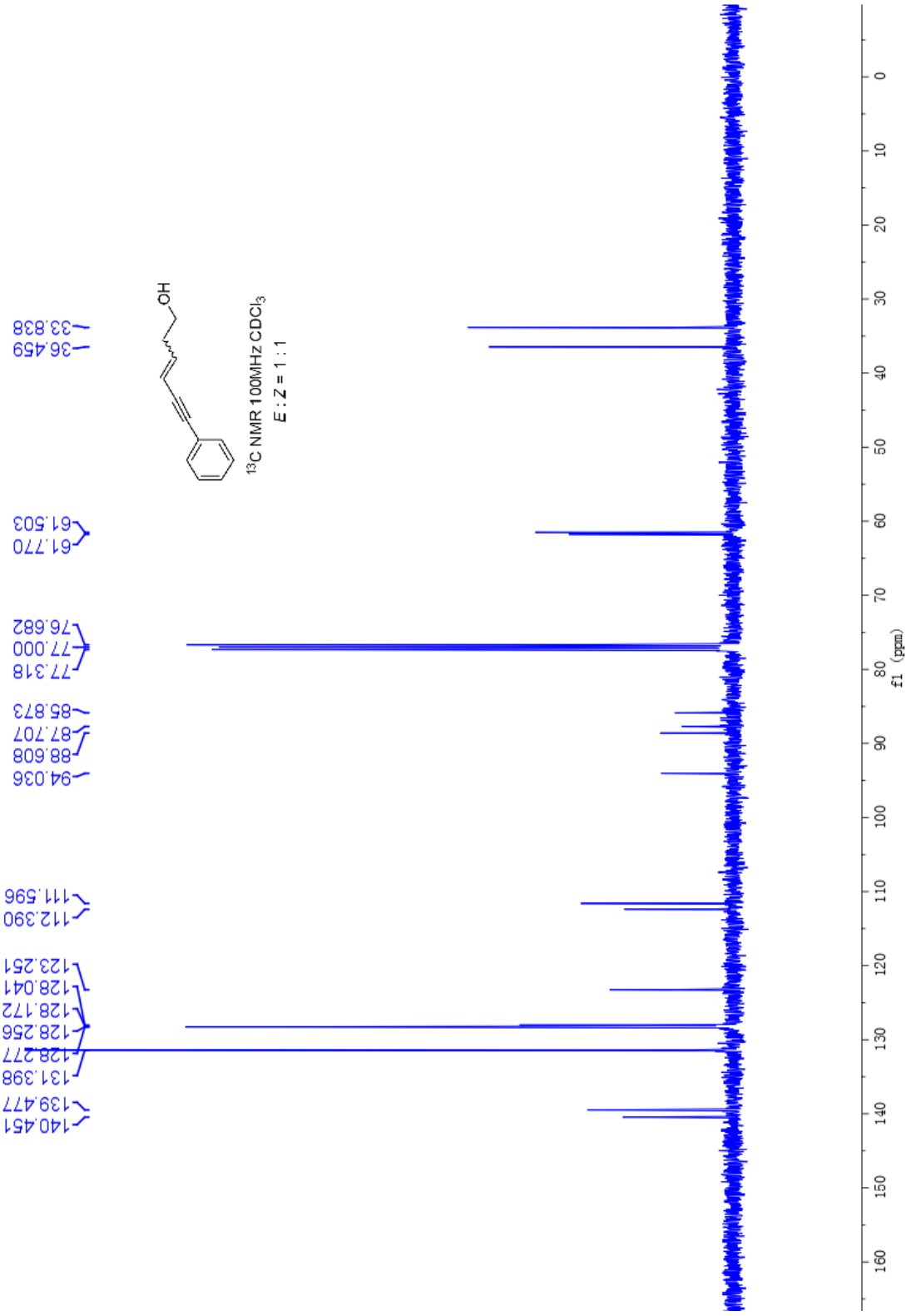
(*Z*)-6-cyclopropyl-4-phenylhex-3-en-5-yn-1-ol (2k).



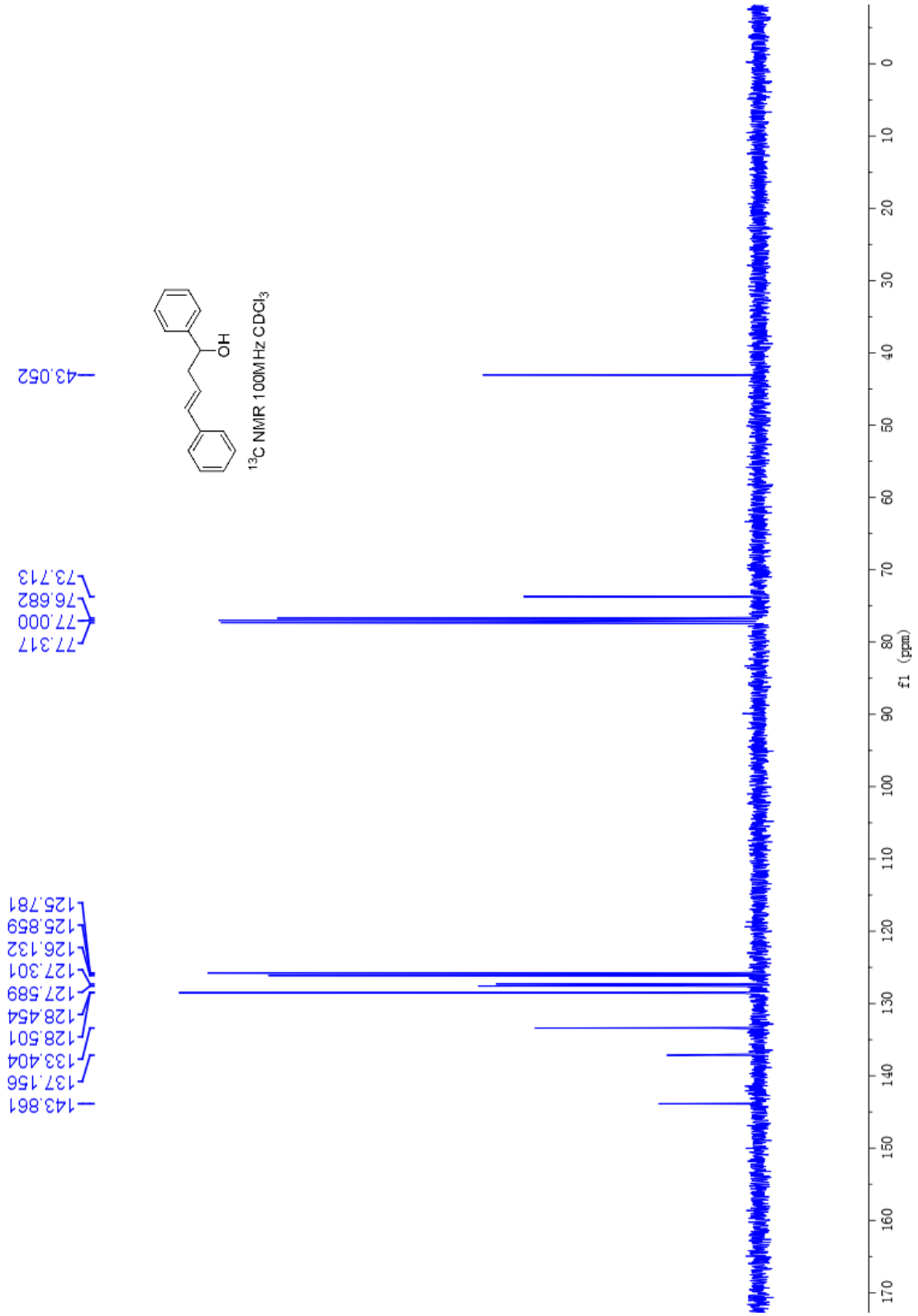

0.347
8.879
34.589
62.121
72.709
76.682
77.000
77.317
100.040
125.947
126.075
127.516
128.221
132.356
138.349
OH
^{13}C NMR 100MHz CDCl$_3$
f1 (ppm)

6-phenylhex-3-en-5-yn-1-ol (2l).

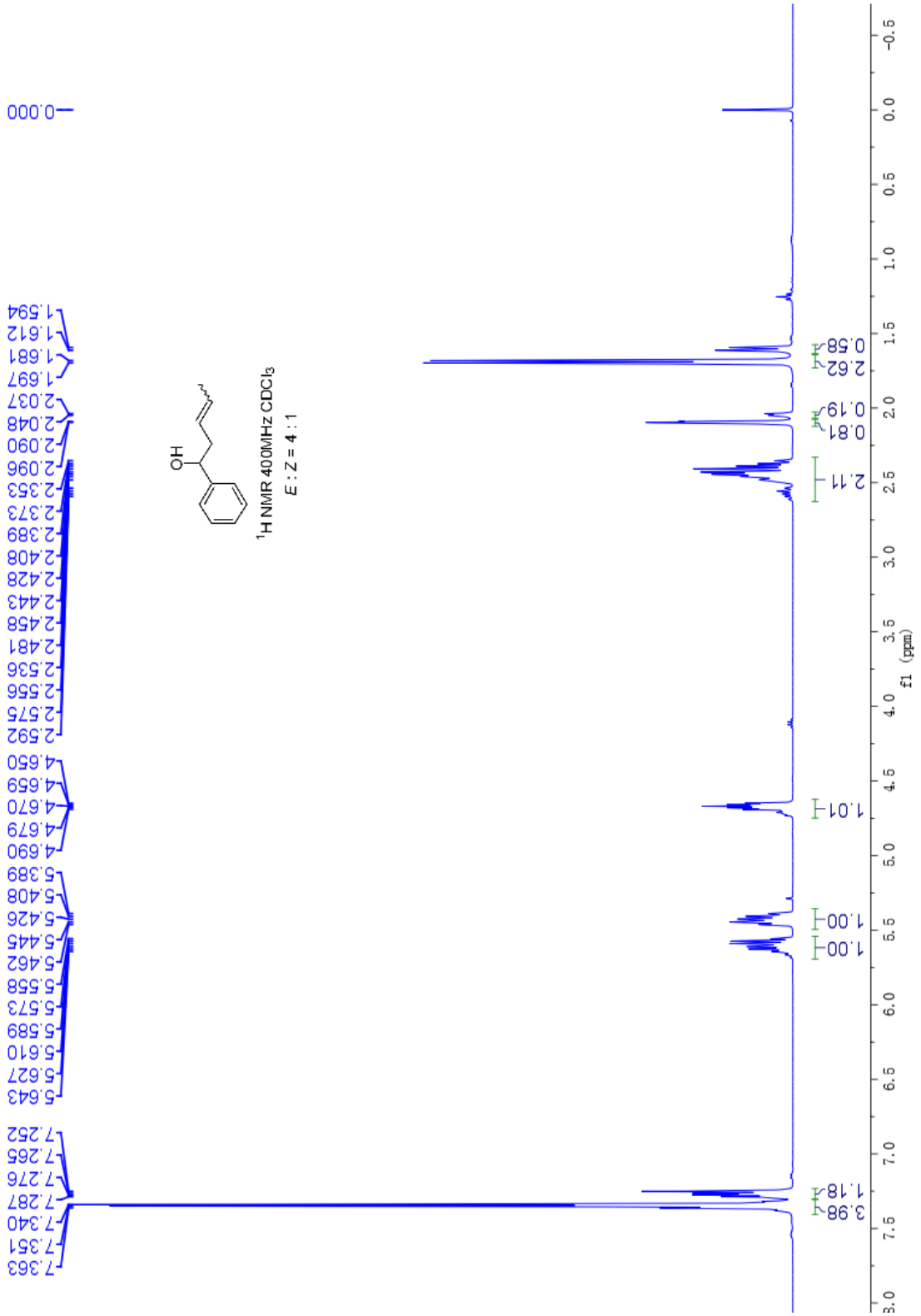



OH
^{13}C NMR 100MHz $CDCl_3$
E : Z = 1 : 1
33.838
36.459
61.503
61.770
76.682
77.000
77.318
85.873
87.707
88.608
94.036
111.596
112.390
123.251
128.041
128.172
128.256
128.277
131.398
139.477
140.451
f1 (ppm)

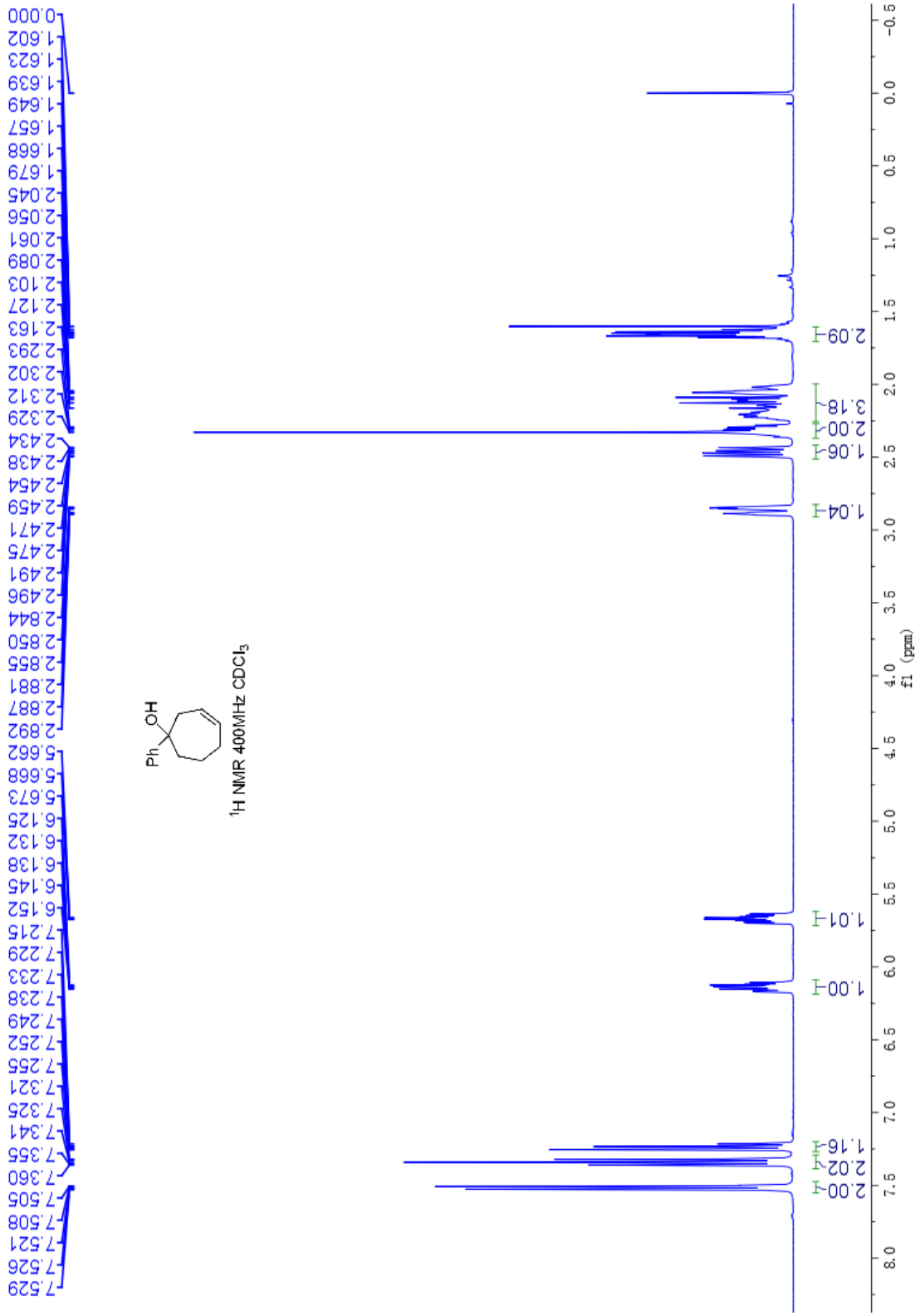
(*E*)-1,4-diphenylbut-3-en-1-ol (2m).

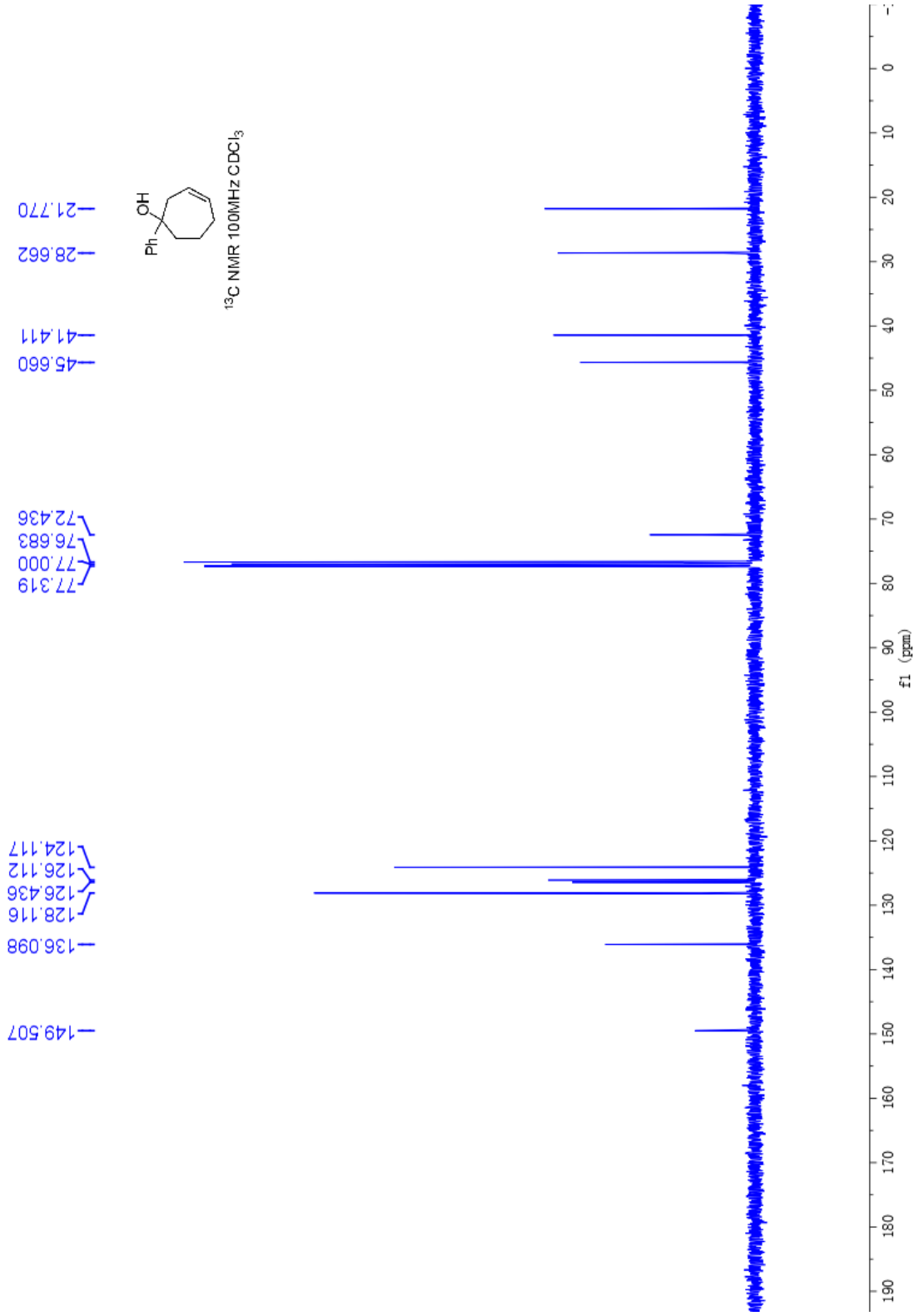


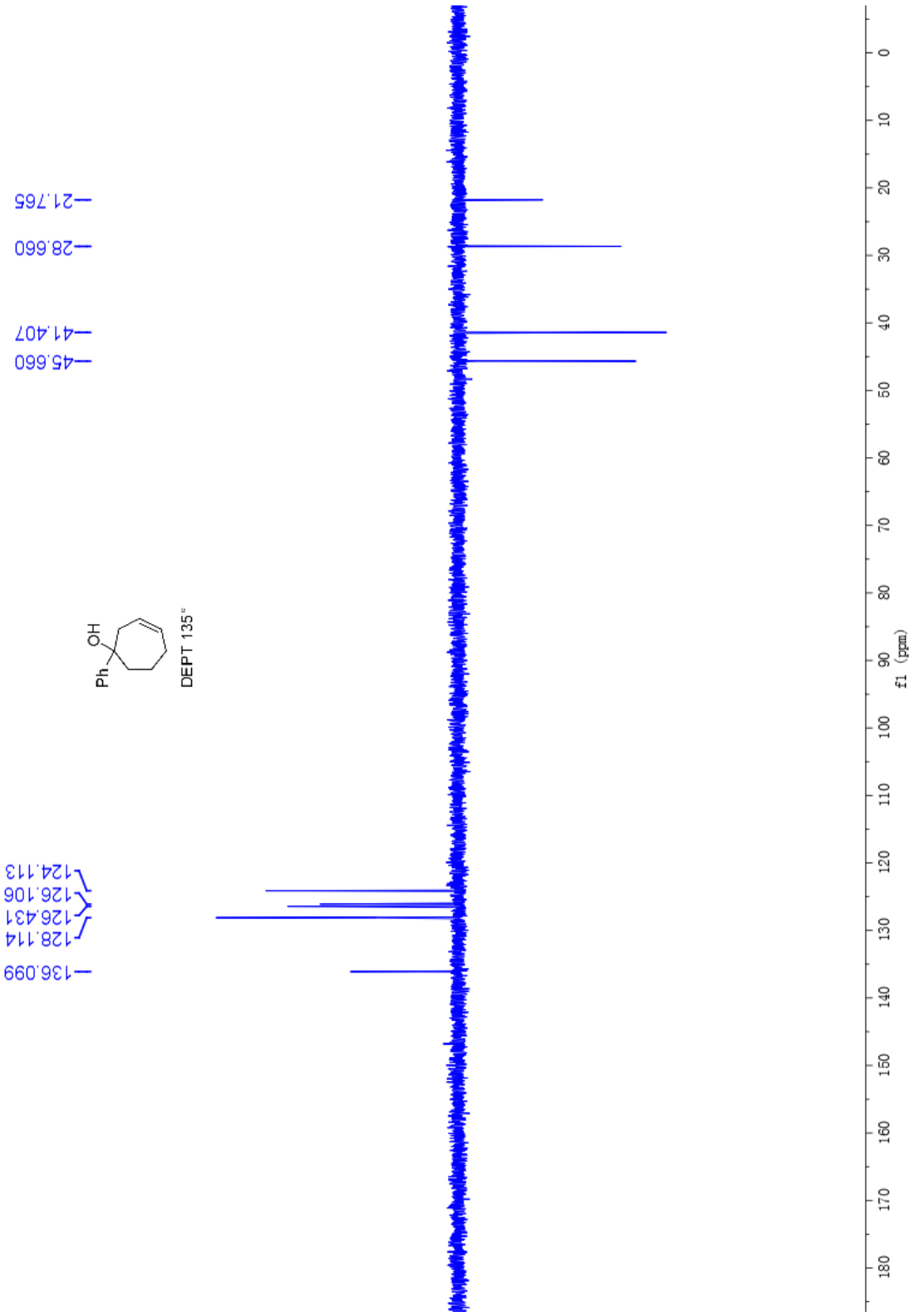
1-phenylpent-3(*E* or *Z*)-en-1-ol (2n).



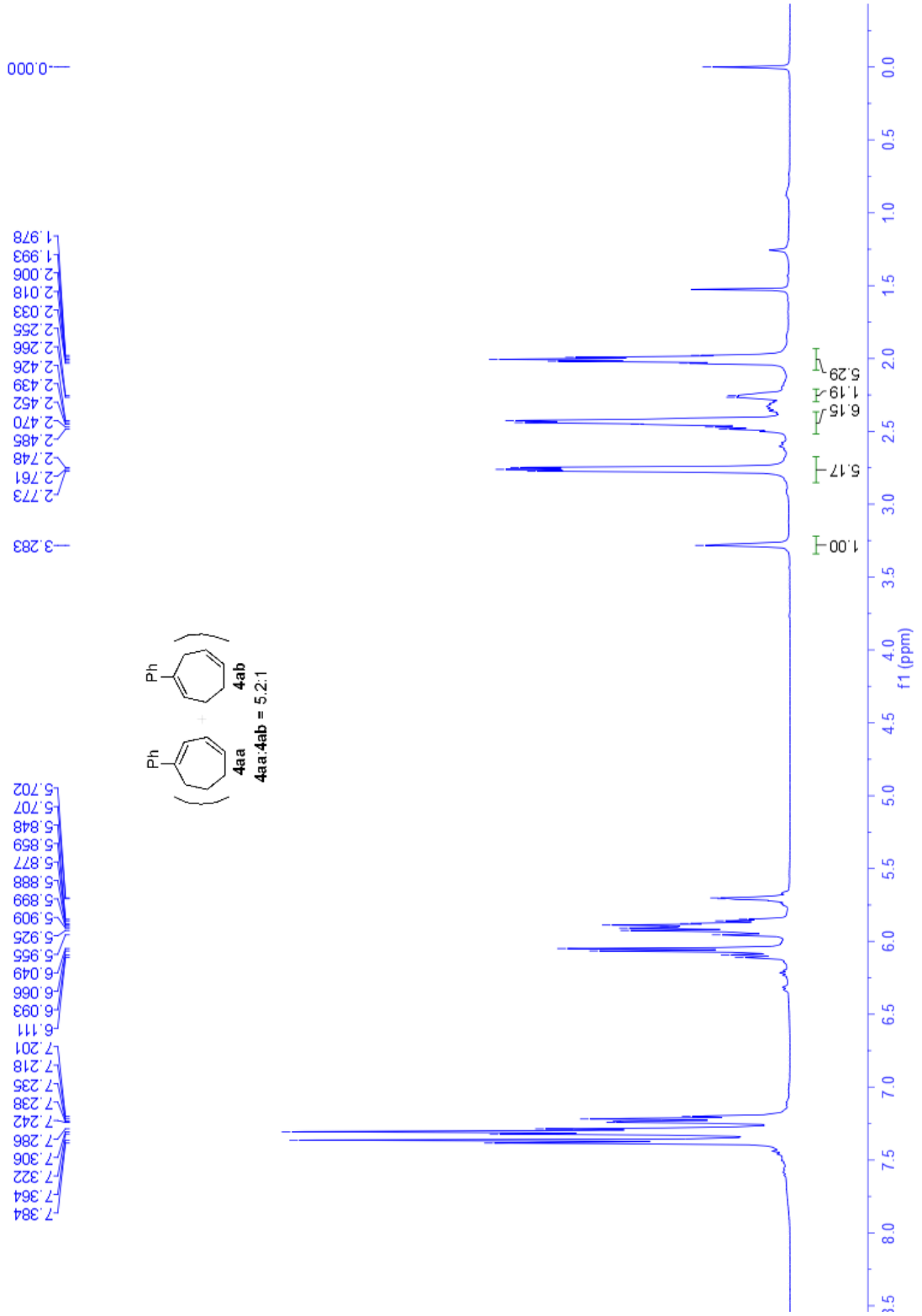
1-phenylcyclohept-3-enol (4a).

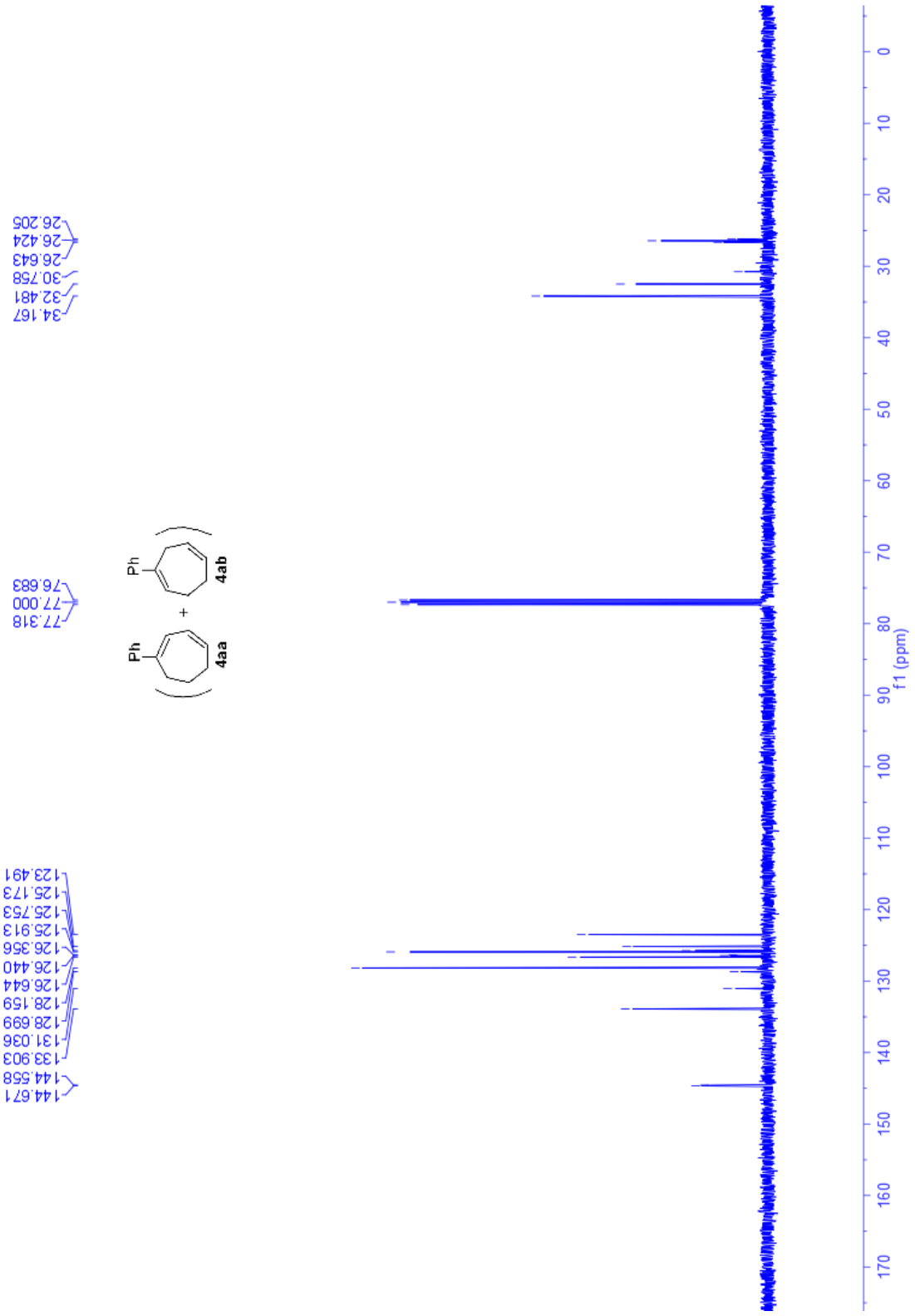



^{13}C NMR 100MHz $CDCl_3$
OH
Ph
21.770
28.662
41.411
45.660
72.436
76.683
77.000
77.319
124.117
126.112
126.436
128.116
136.098
149.507
f1 (ppm)

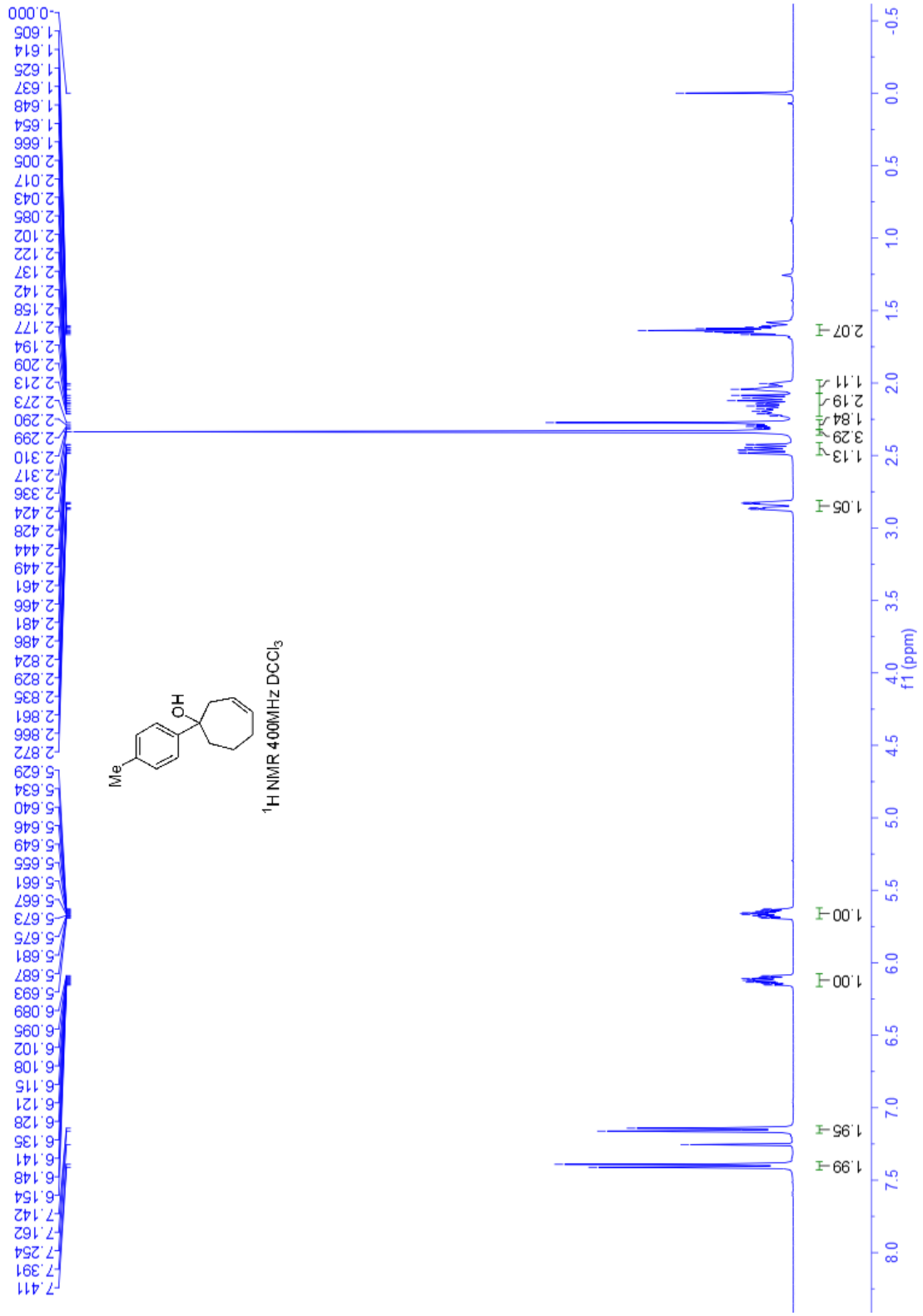

21.765
28.660
41.407
45.660
124.113
126.106
126.431
128.114
136.099
OH
Ph
DEPT 135°
f1 (ppm)

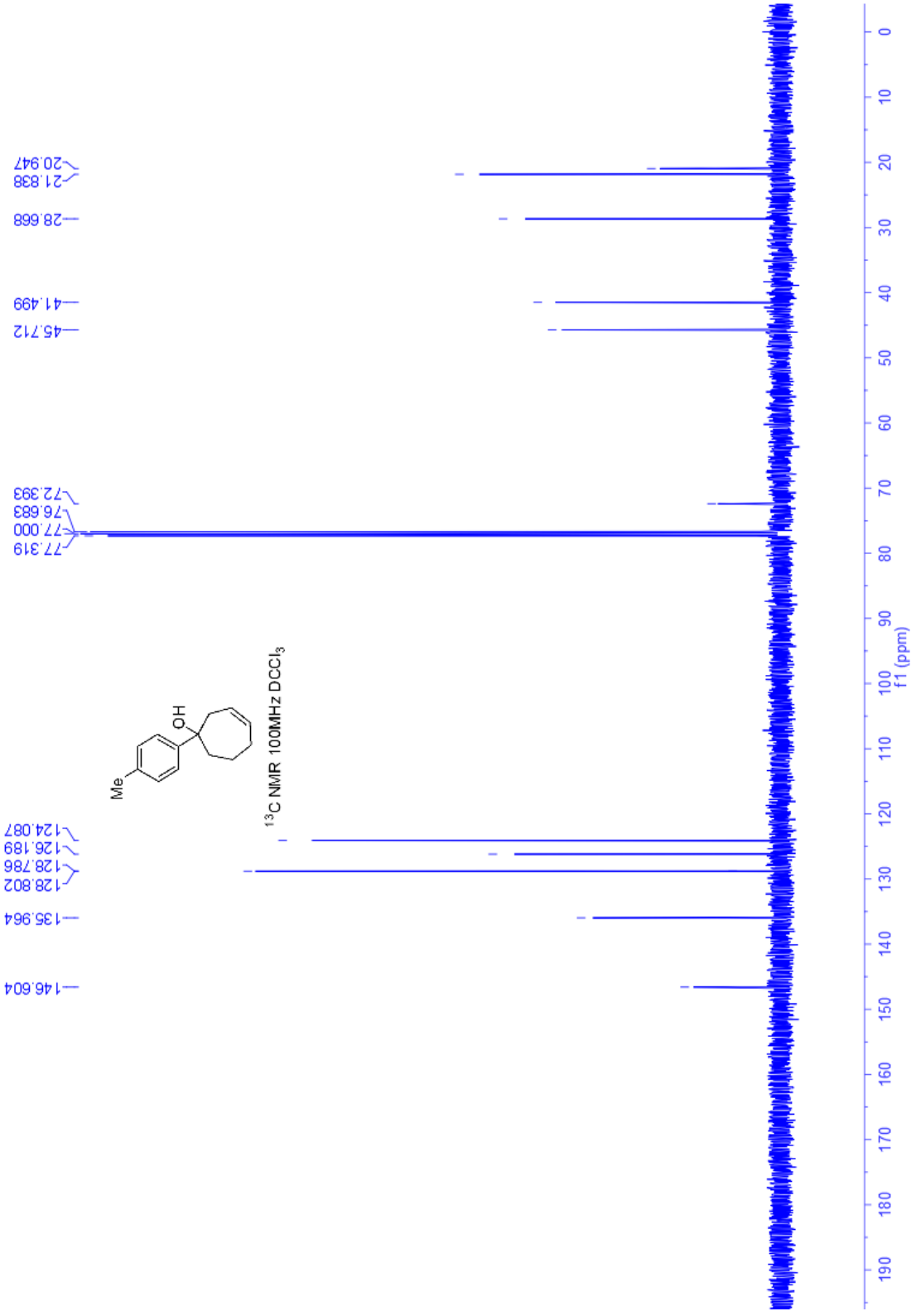
4aa and **4ab.**

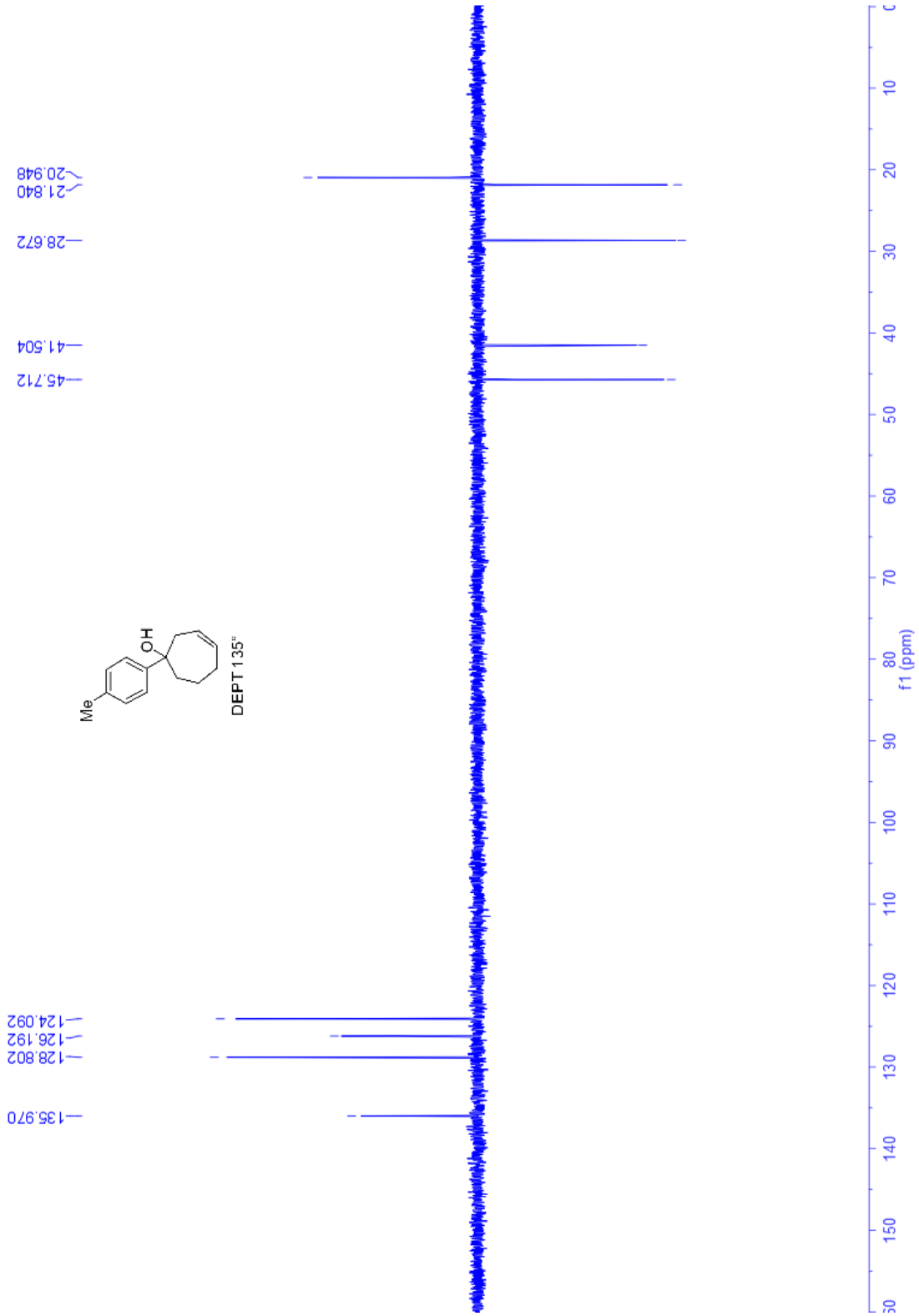



144.671
144.558
133.903
131.036
128.699
128.159
126.644
126.440
126.356
125.913
125.753
125.173
123.491
77.318
77.000
76.683
Ph
4ab
+
Ph
4aa
34.167
32.481
30.758
26.643
26.424
26.205
f1 (ppm)

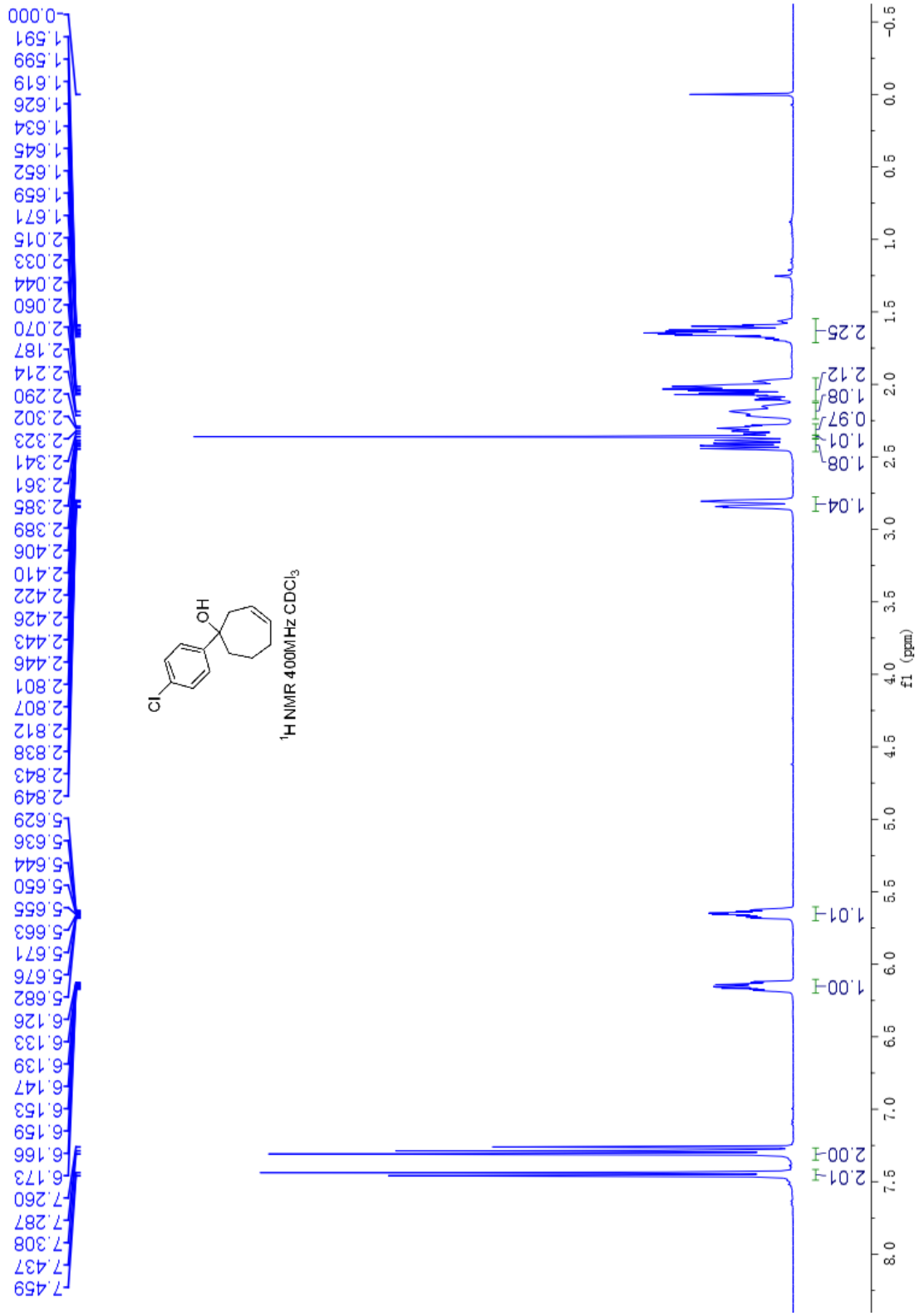
1-(p-tolyl)cyclohept-3-enol (4b).

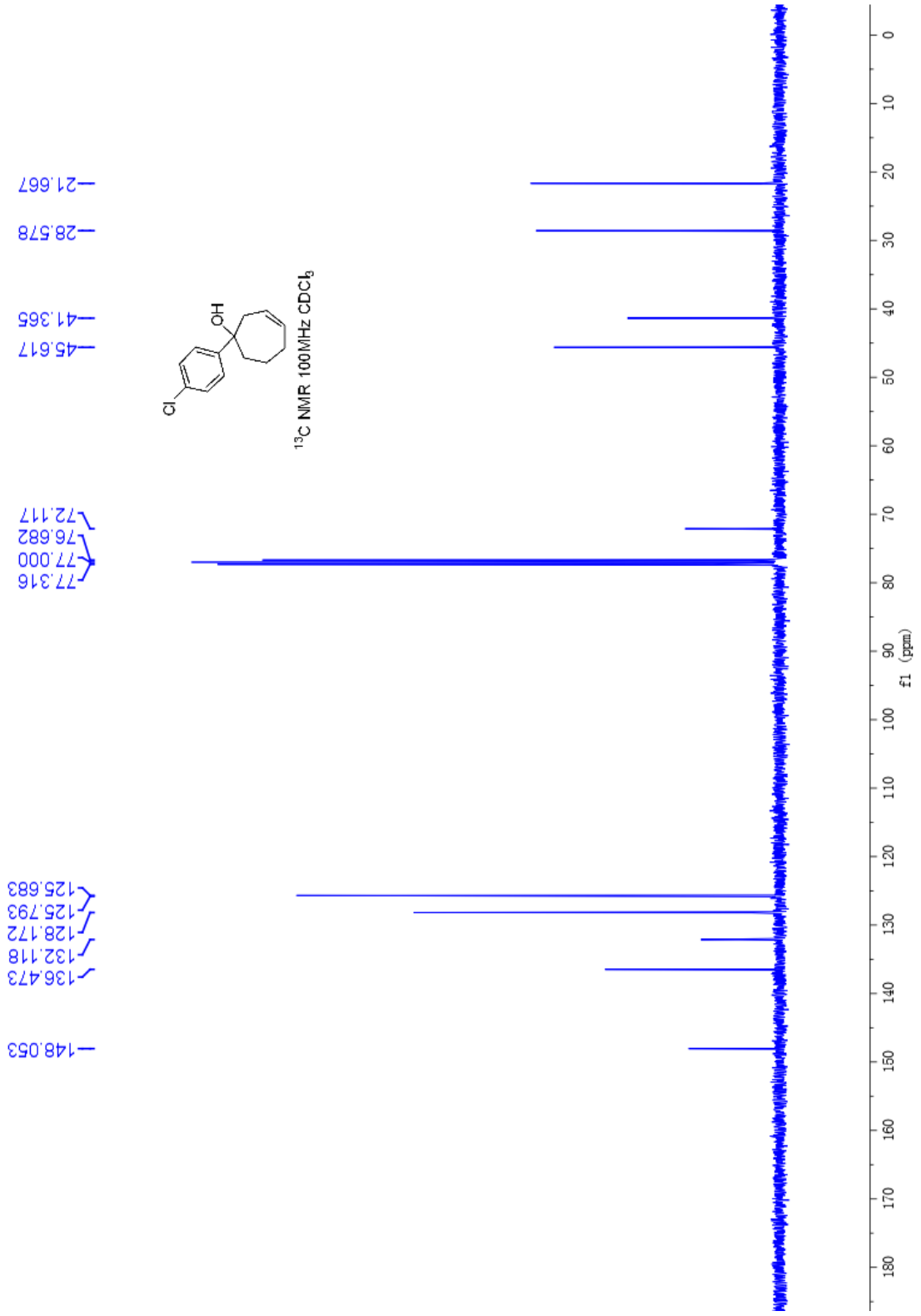


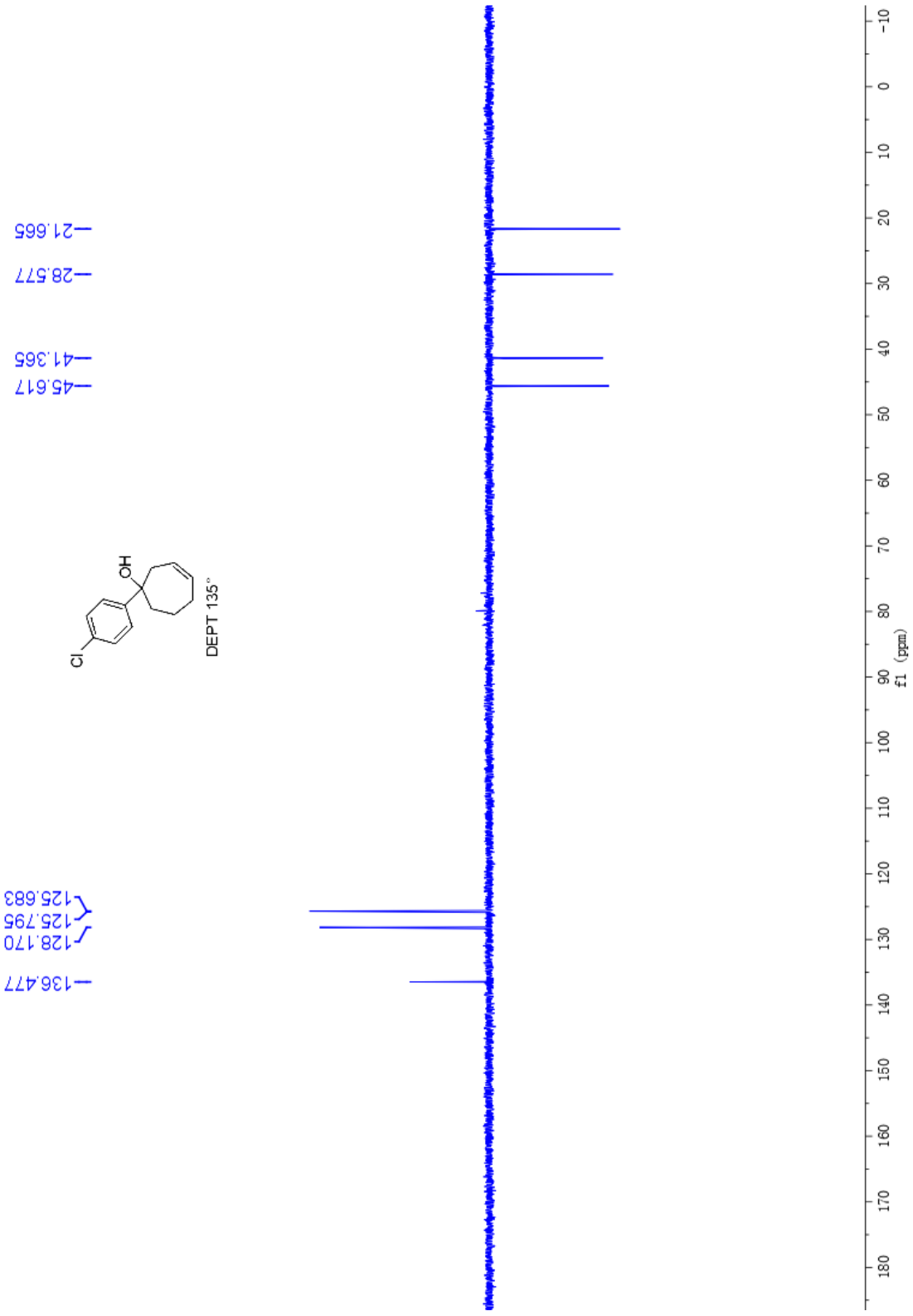

20.947
21.838
28.668
41.499
45.712
72.393
76.683
77.000
77.319
124.087
126.189
128.786
128.802
135.964
146.604
OH
Me
^{13}C NMR 100MHz DCCl$_3$
f1 (ppm)


20.948
21.840
28.672
41.504
45.712
124.092
126.192
128.802
135.970
OH
Me
DEPT135
f1 (ppm)

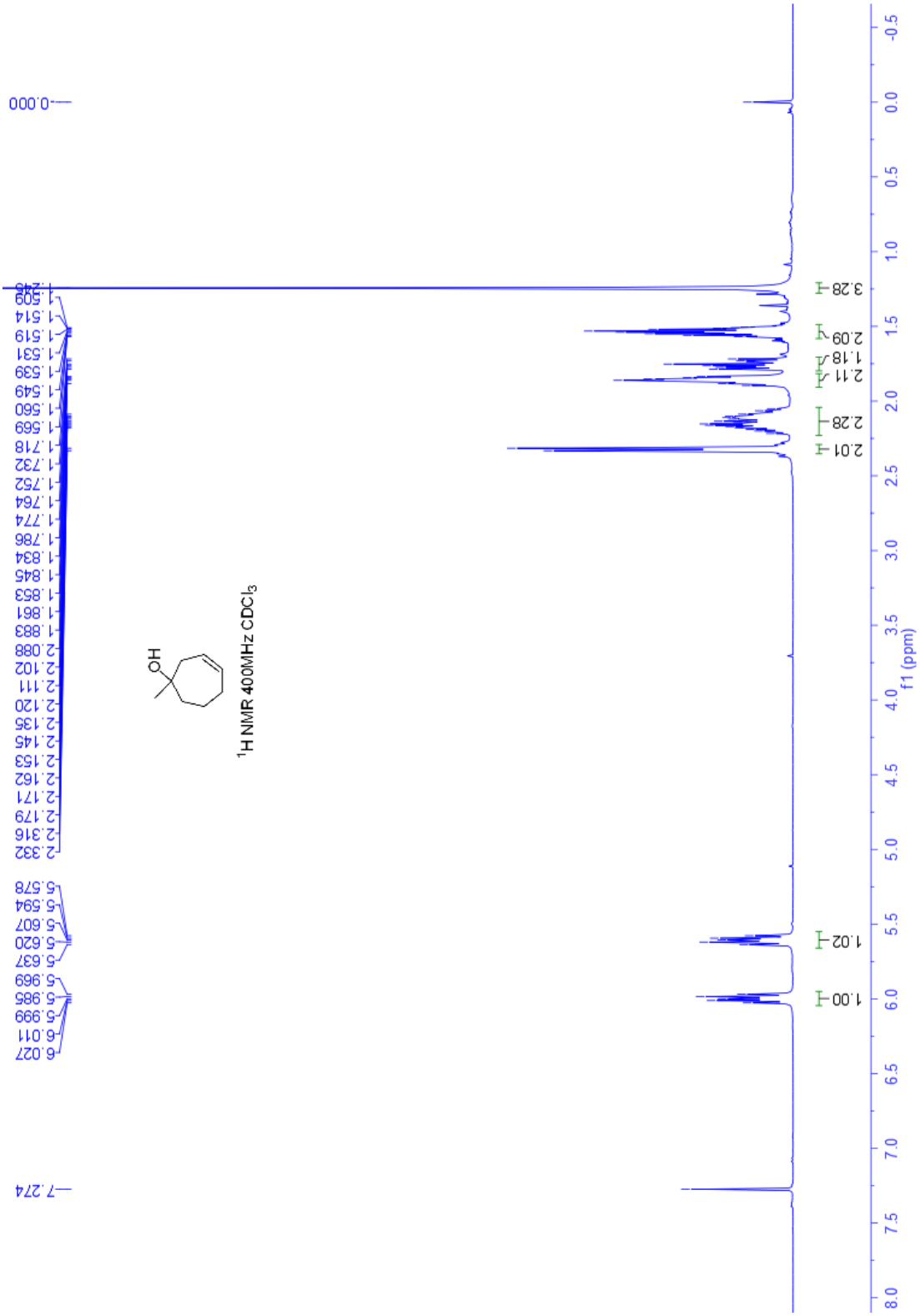
1-(4-chlorophenyl)cyclohept-3-enol (4c).

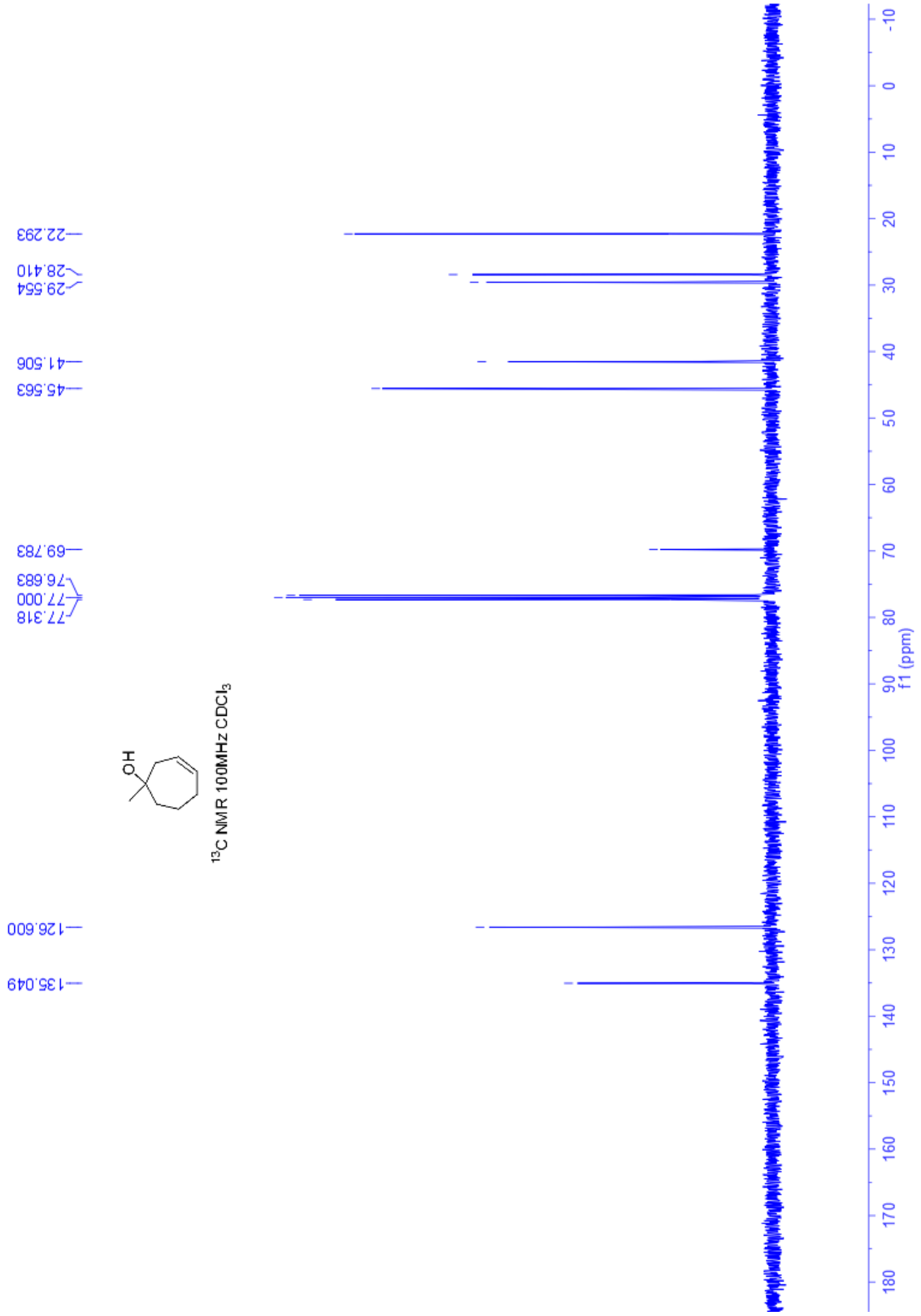


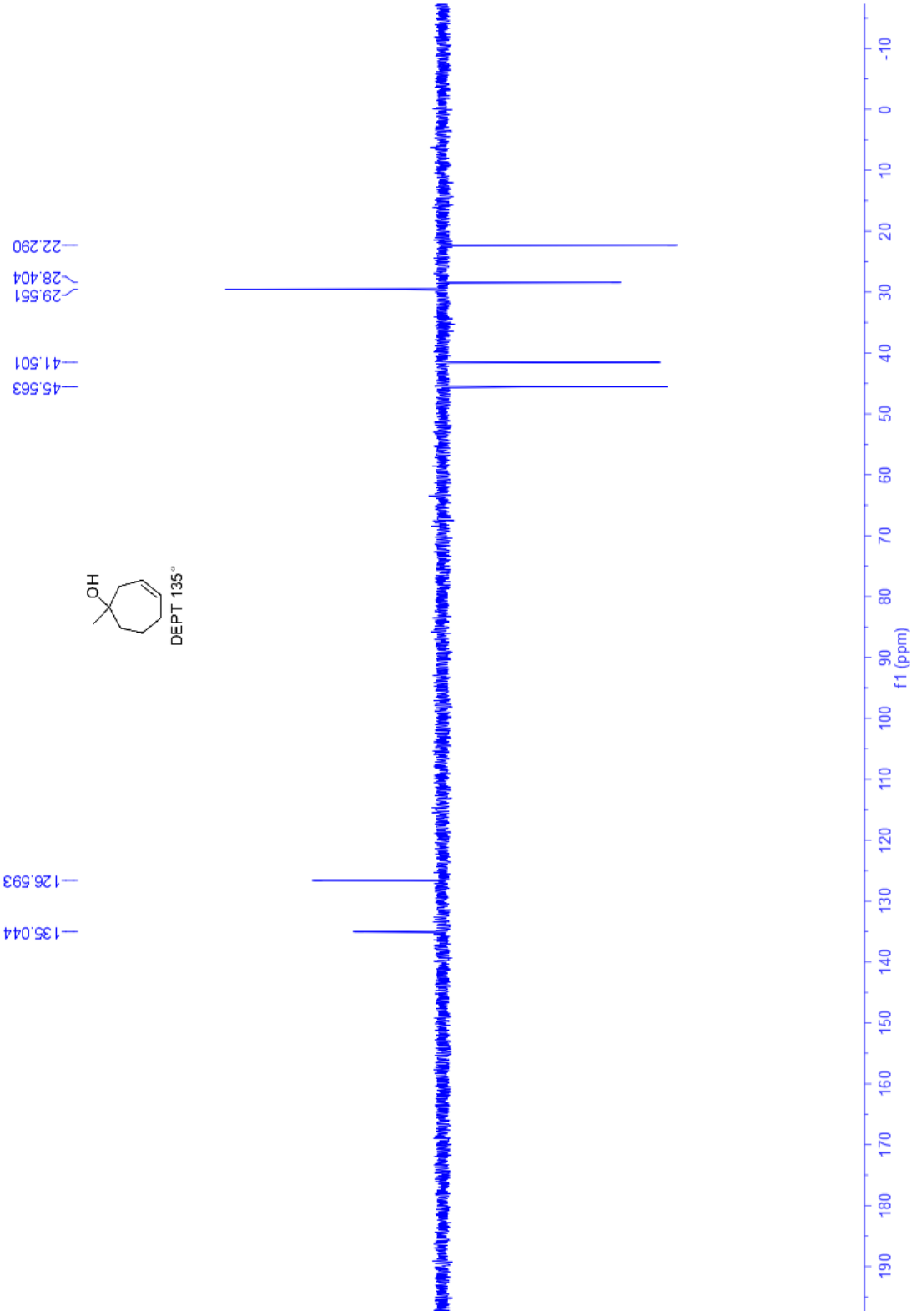

21.667
28.578
41.365
45.617
72.117
76.682
77.000
77.316
125.683
125.793
128.172
132.118
136.473
148.053
OH
Cl
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


21.665
28.577
41.365
45.617
OH
Cl
DEPT 135°
125.683
125.795
128.170
136.477
f1 (ppm)

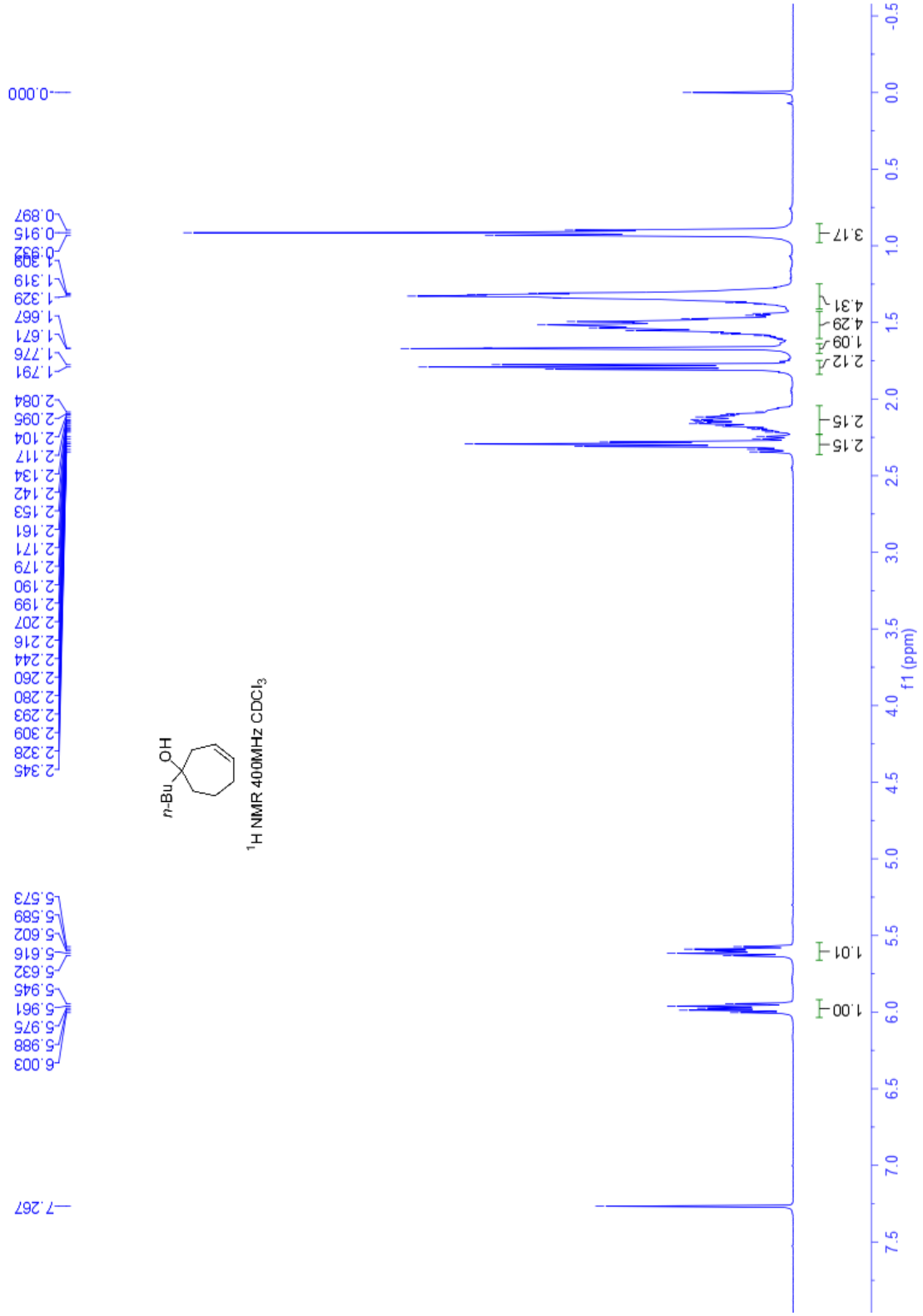
1-methylcyclohept-3-enol (4d).

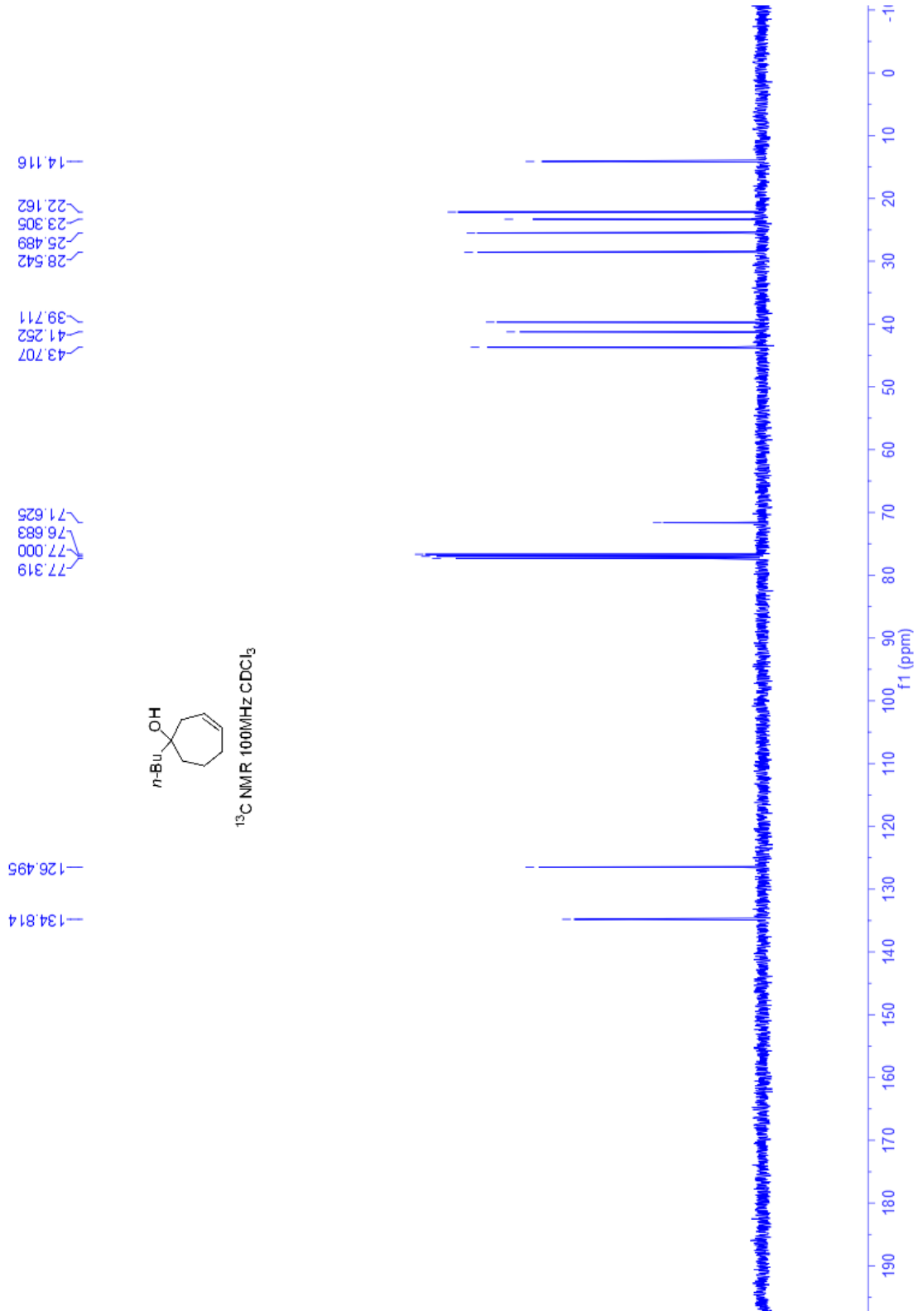


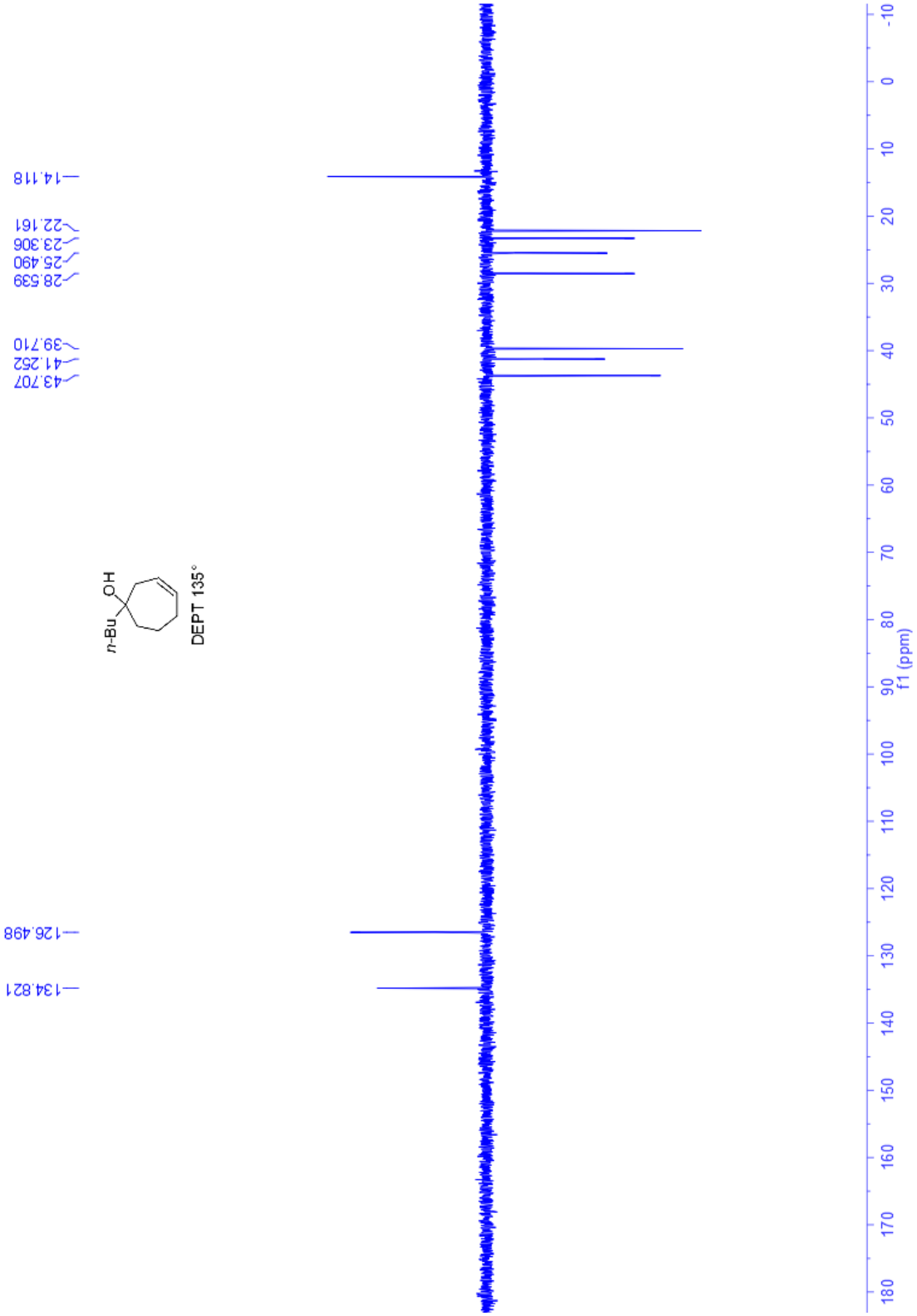

22.293
28.410
29.554
41.506
45.563
69.783
76.683
77.000
77.318
126.600
135.049
OH
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


22.290
28.404
29.551
41.501
45.563
126.593
135.044
OH
DEPT 135°
f1 (ppm)

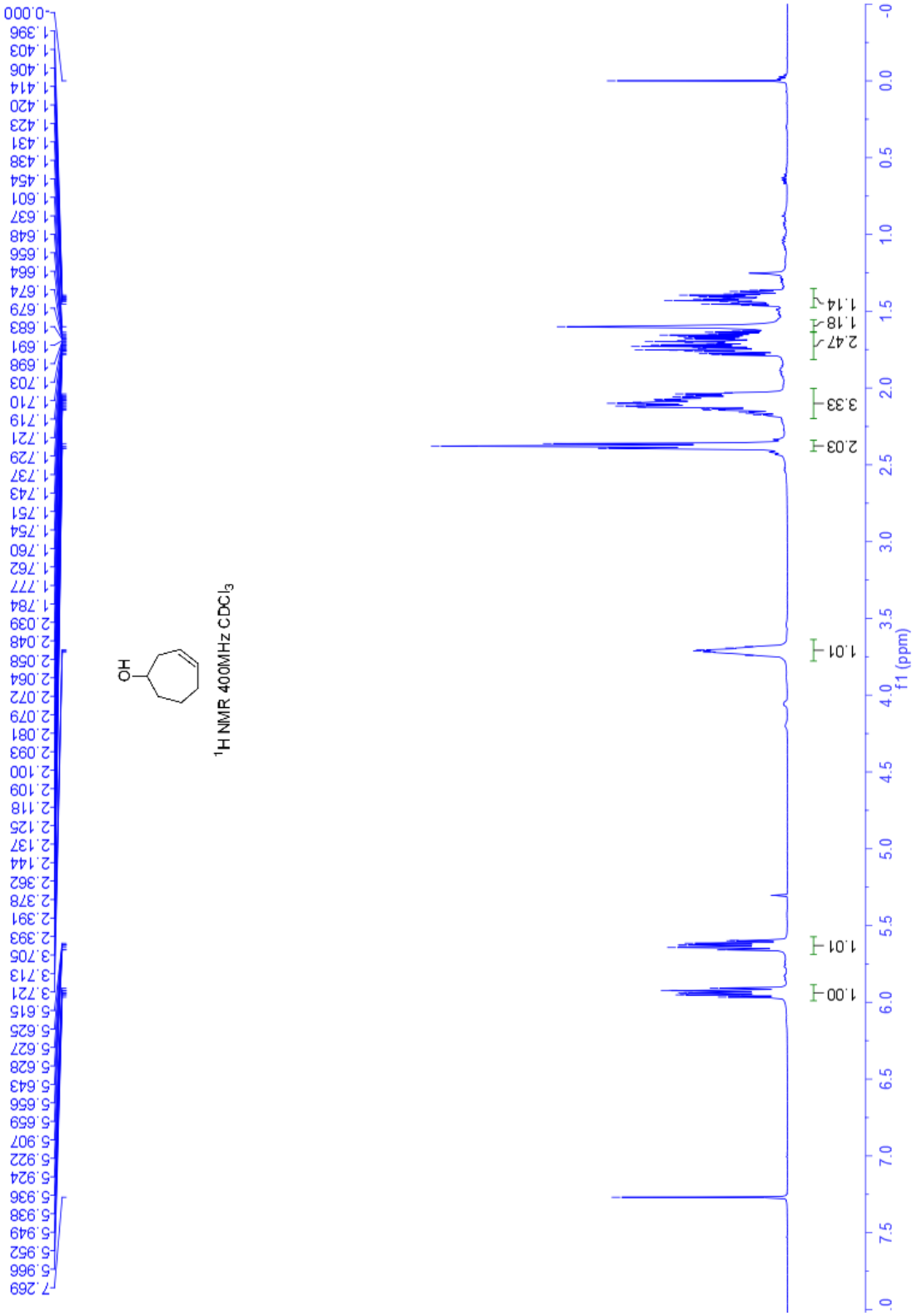
1-butylcyclohept-3-enol (4e).

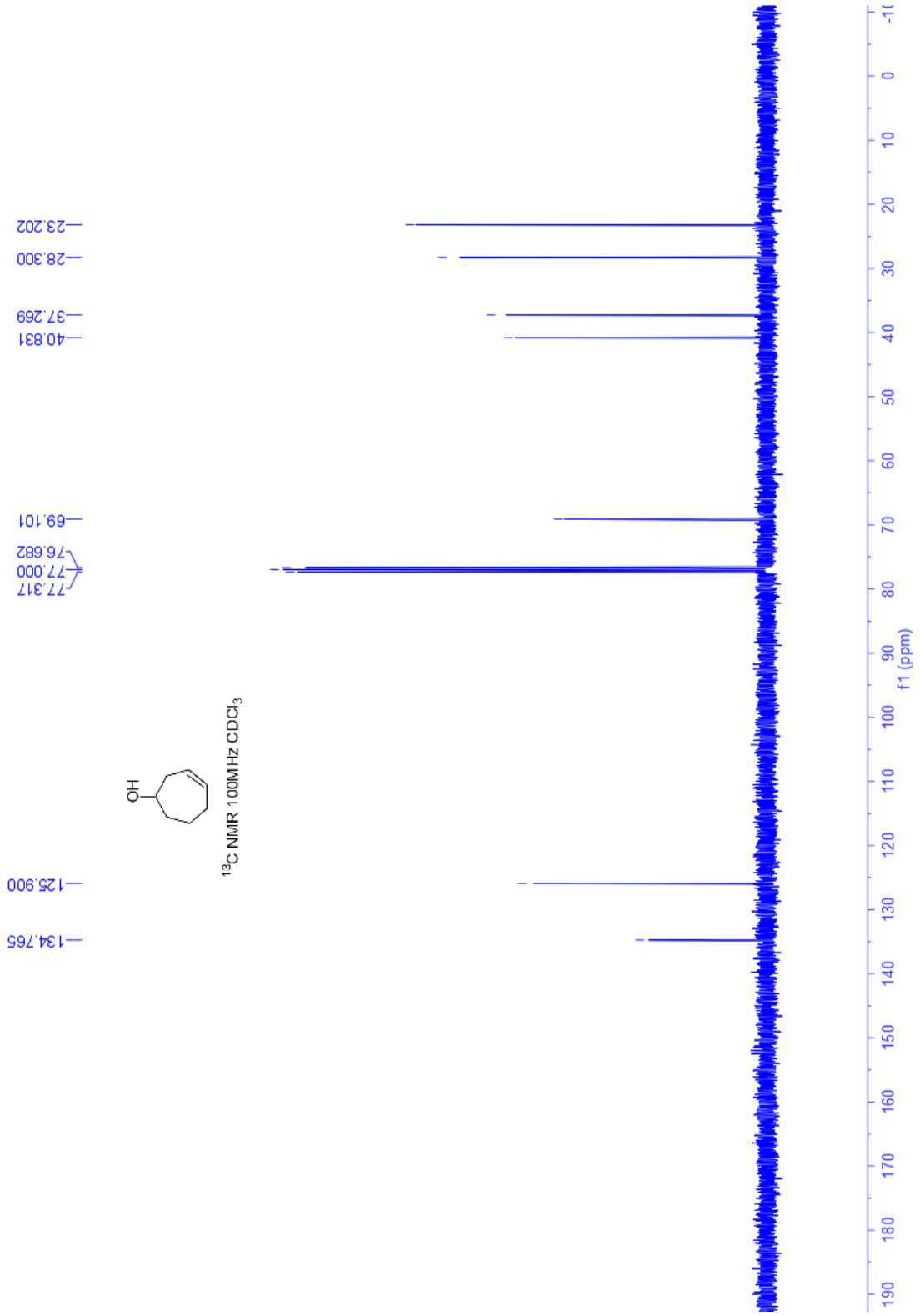


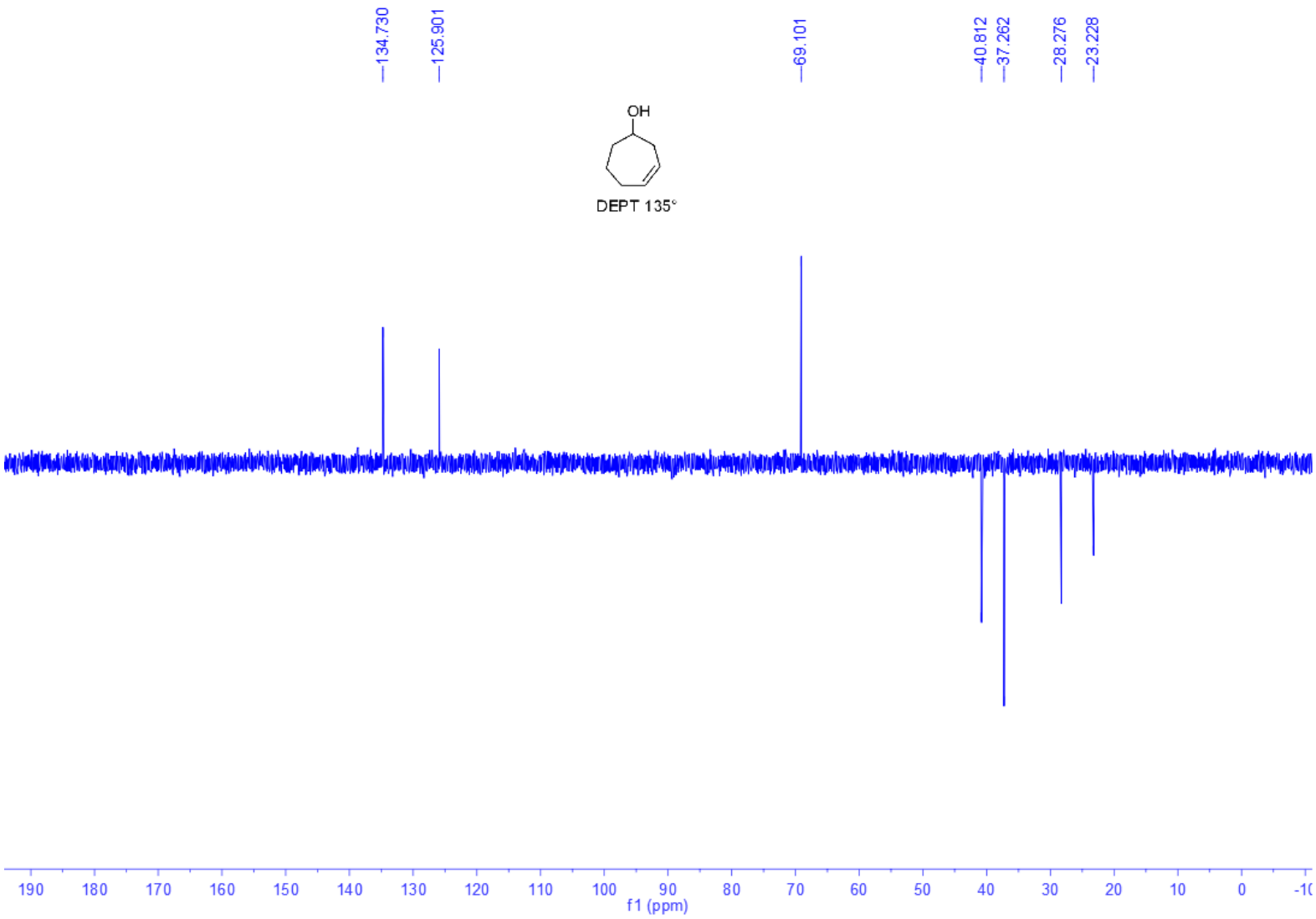

14.116
22.162
23.305
25.489
28.542
39.711
41.252
43.707
71.625
76.683
77.000
77.319
126.495
134.814
OH
n-Bu
^{13}C NMR 100MHz CDCl$_3$
f1 (ppm)


14.118
22.161
23.306
25.490
28.539
39.710
41.252
43.707
126.498
134.821
n-Bu
OH
DEPT 135°
f1 (ppm)

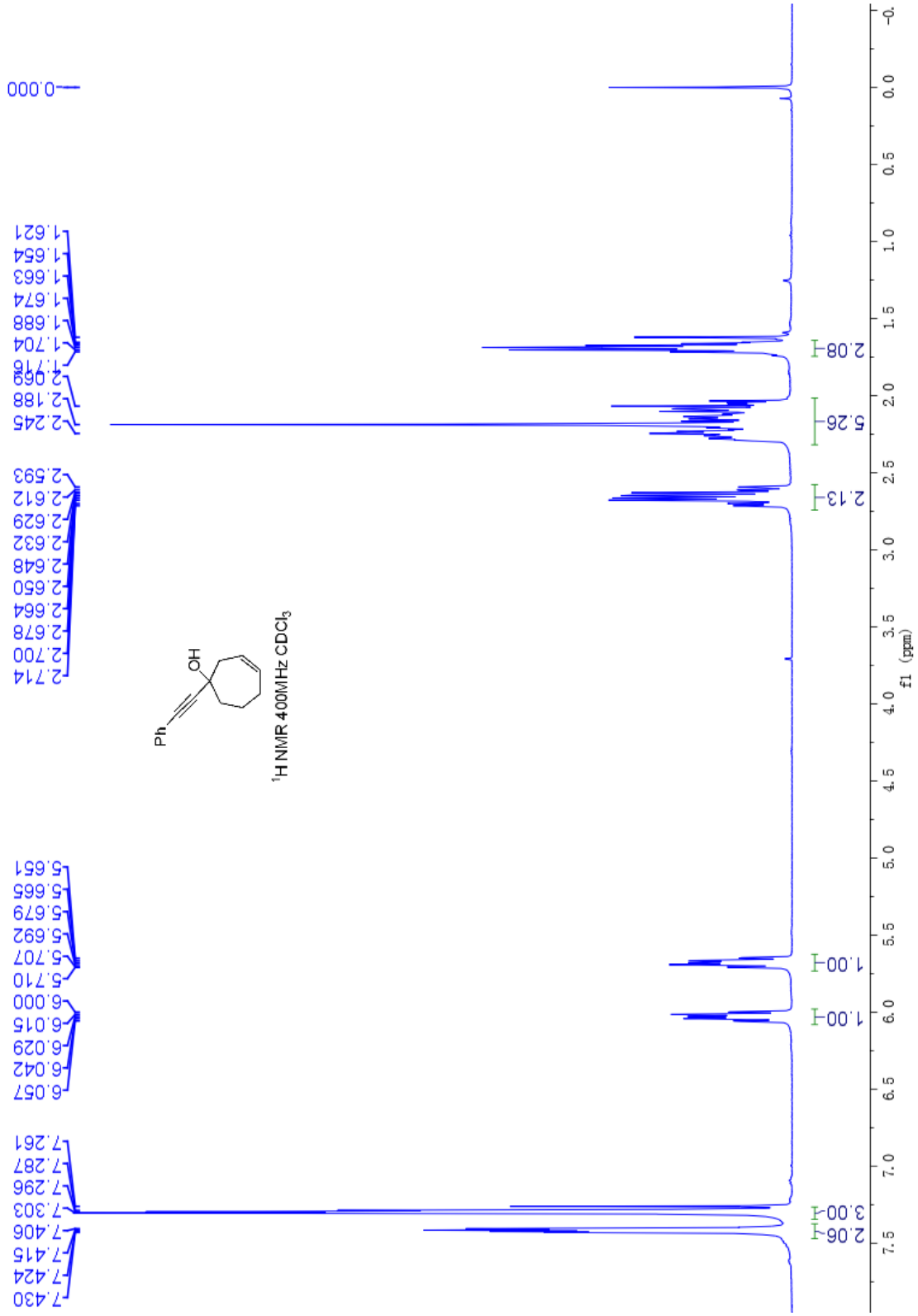
cyclohept-3-enol (4f).

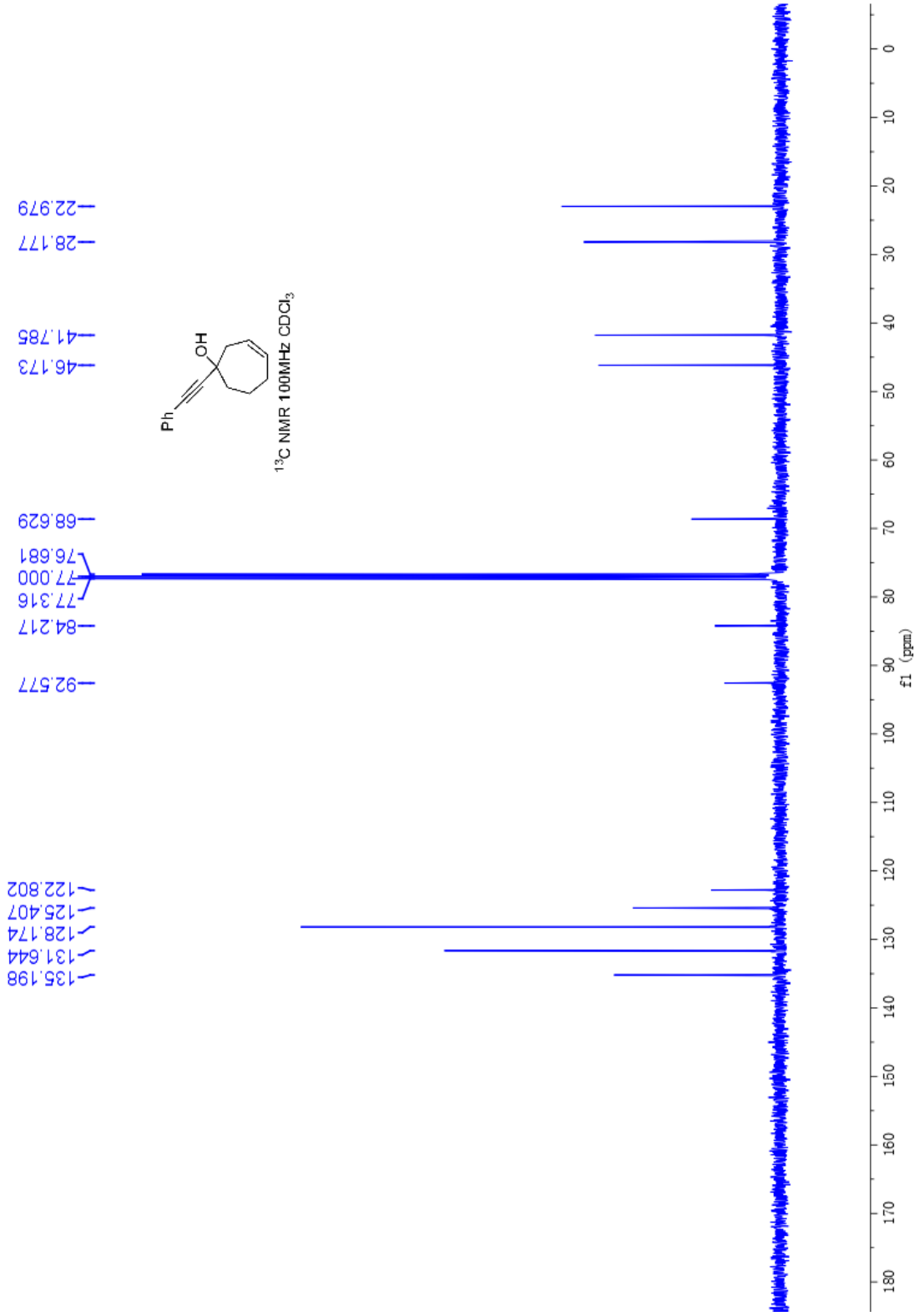


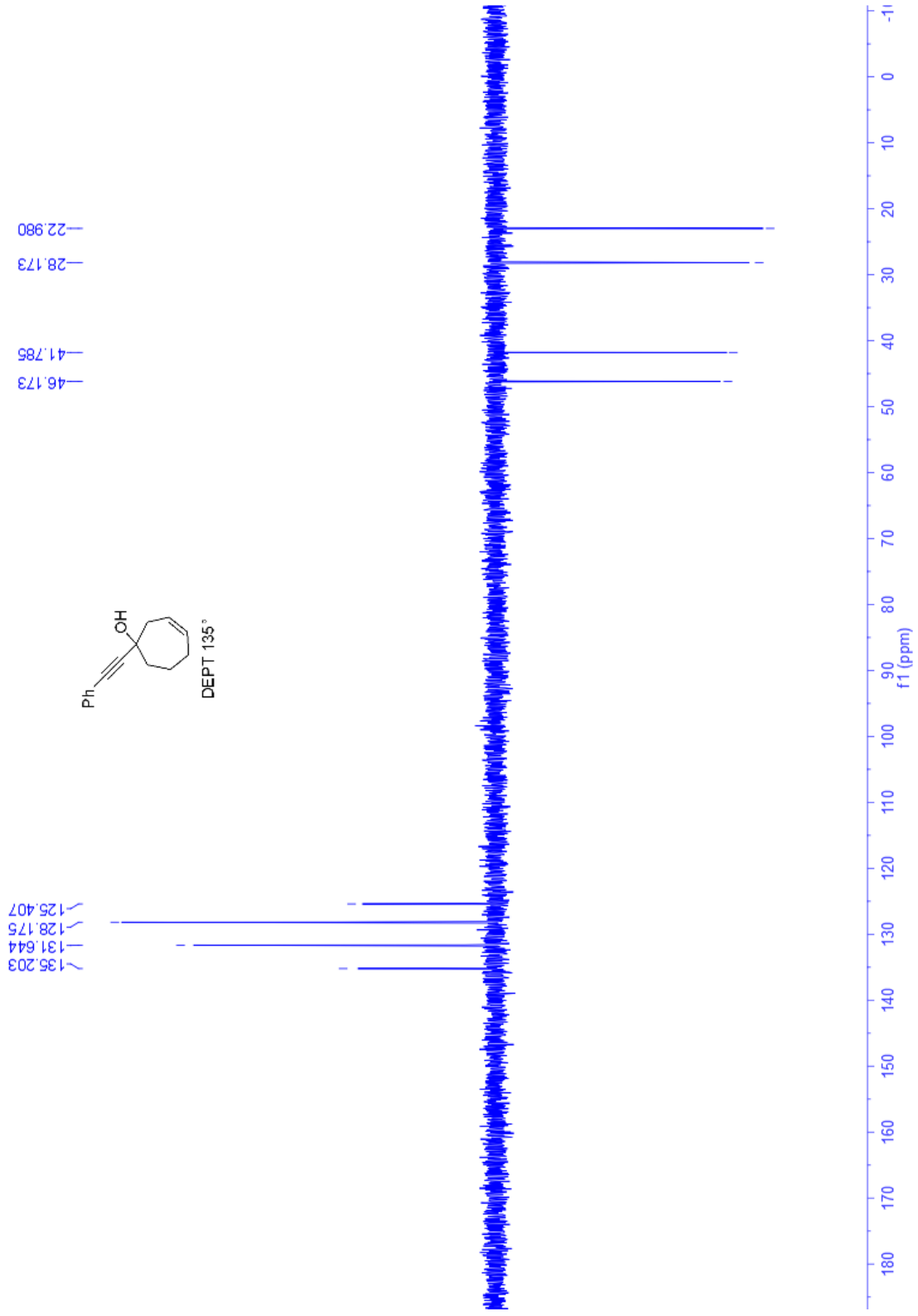

23.202
28.300
37.269
40.831
69.101
76.682
77.000
77.317
125.900
134.765
OH
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


134.730
125.901
69.101
40.812
37.262
28.276
23.228
OH
DEPT 135°
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

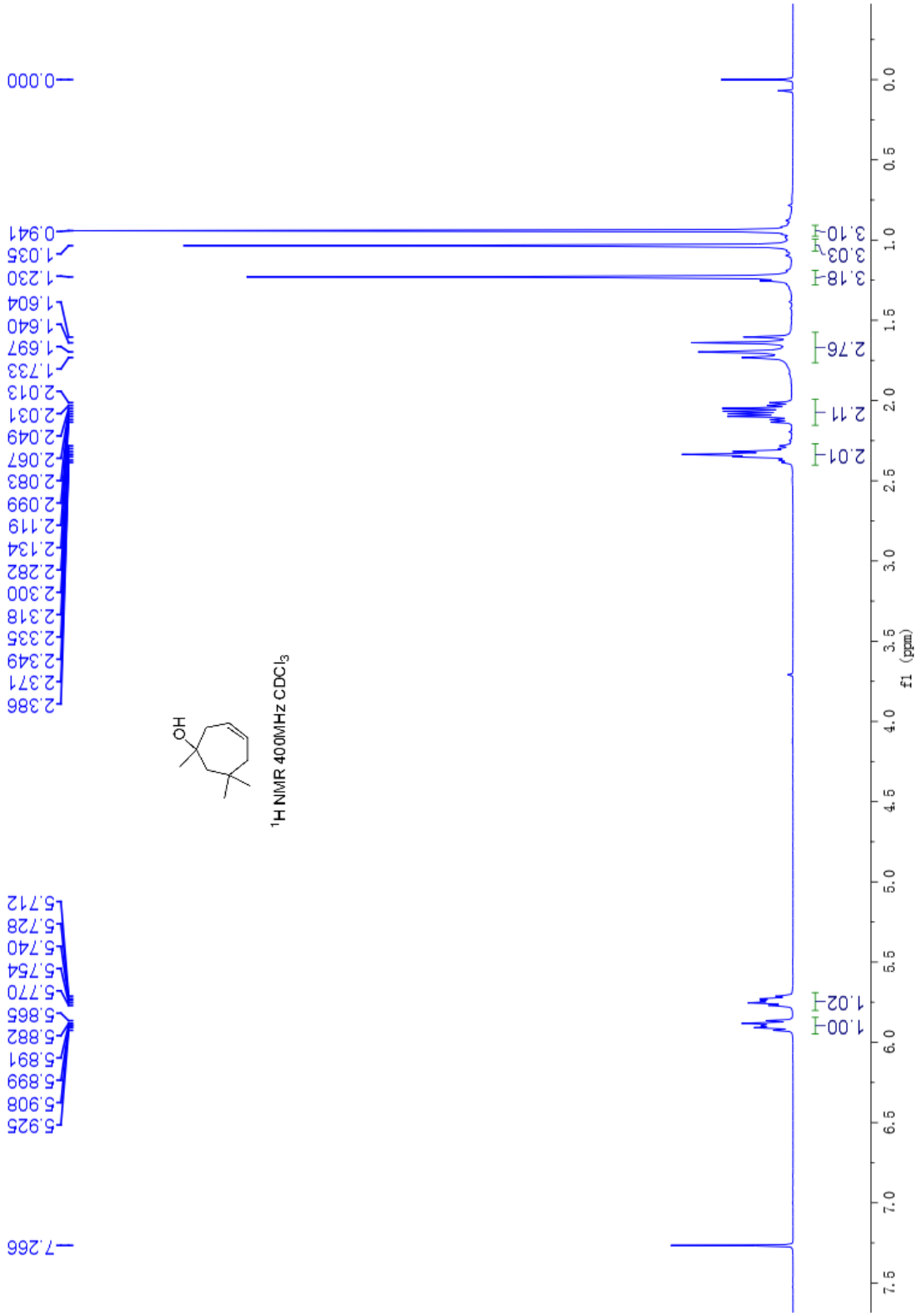
1-(phenylethynyl)cyclohept-3-enol (4g).

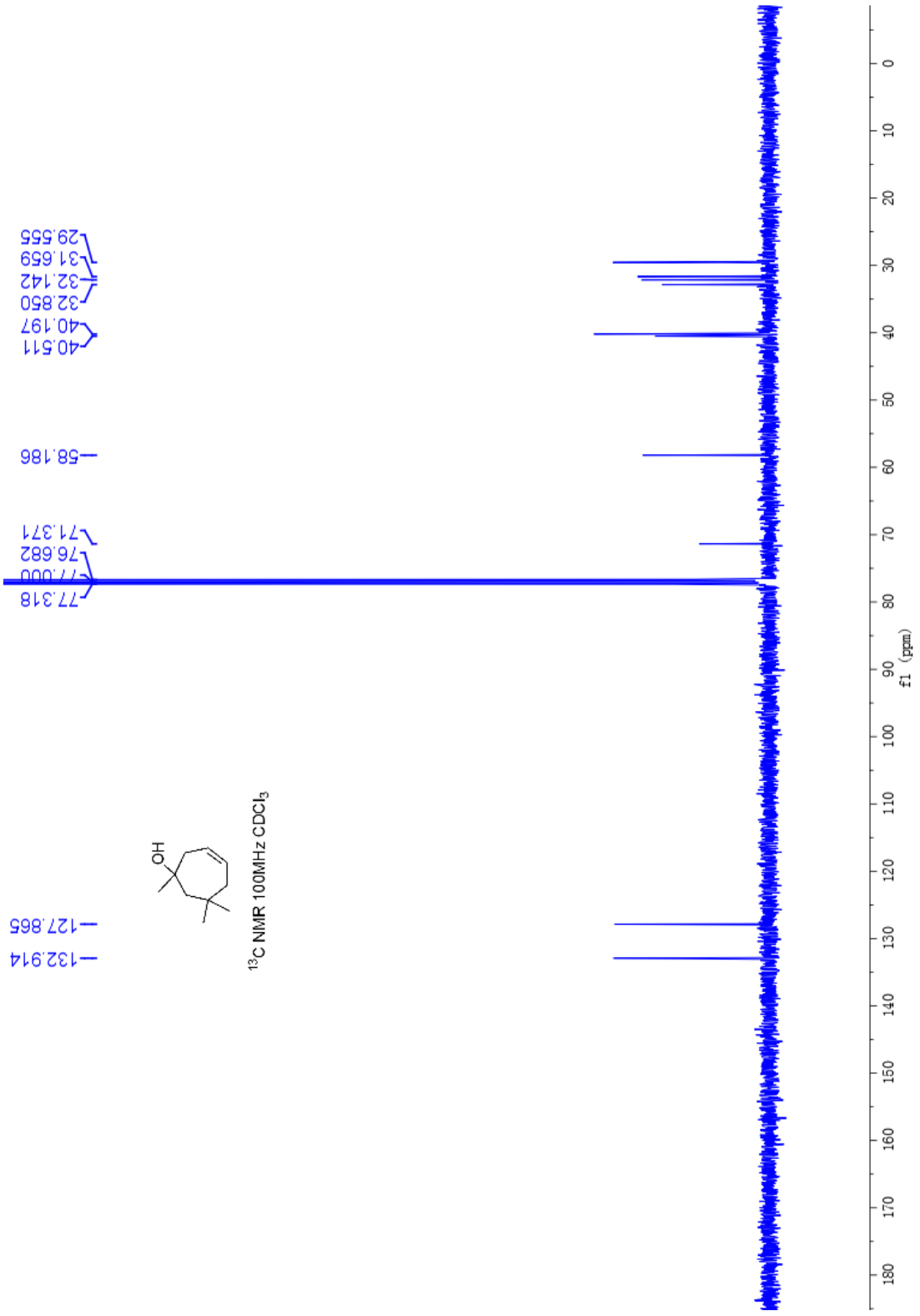


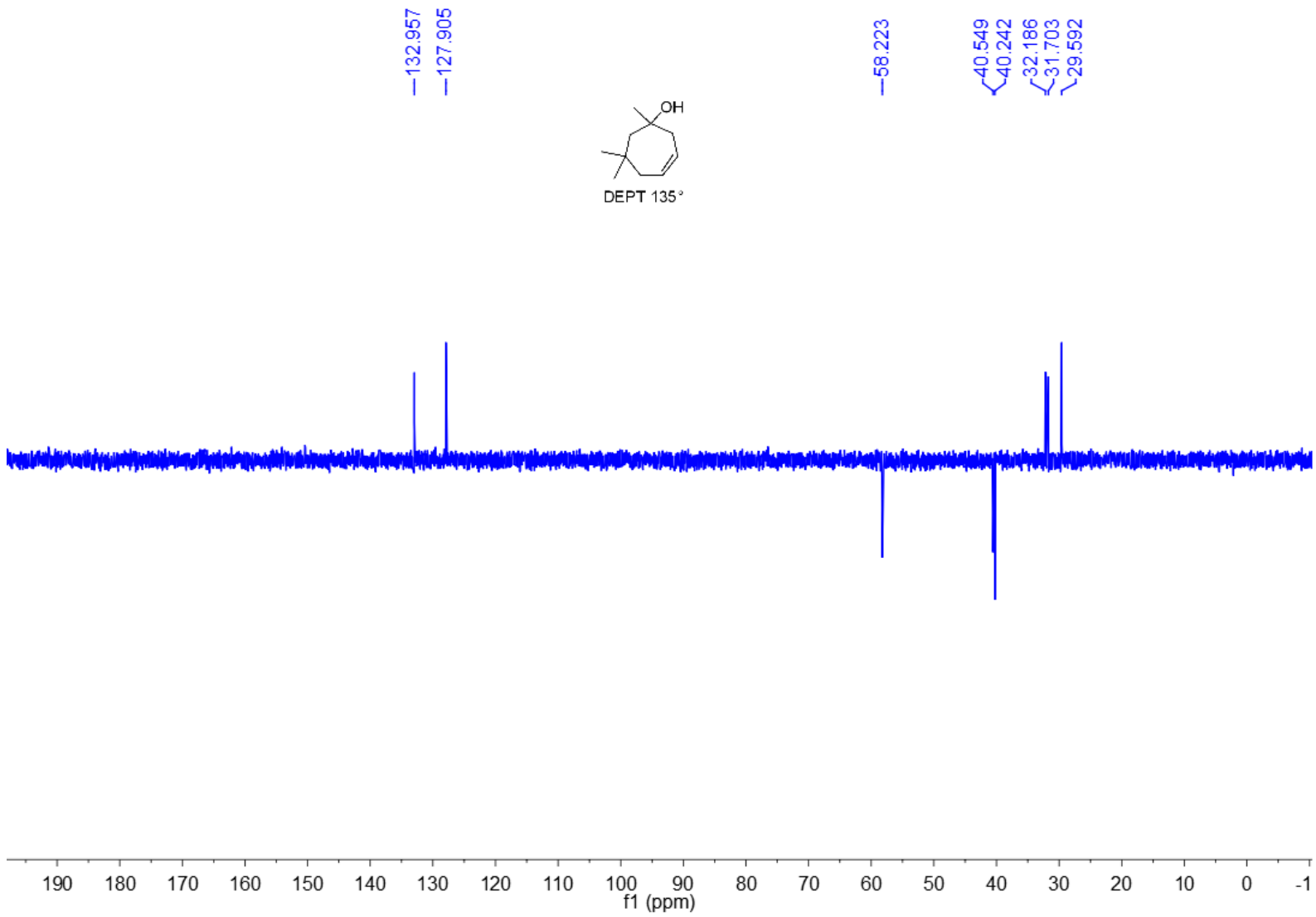

22.979
28.177
41.785
46.173
68.629
76.681
77.000
77.316
84.217
92.577
122.802
125.407
128.174
131.644
135.198
OH
Ph
^{13}C NMR 100MHz $CDCl_3$
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
f1 (ppm)


22.980
28.173
41.785
46.173
125.407
128.175
131.644
135.203
OH
Ph
DEPT 135°
f1 (ppm)

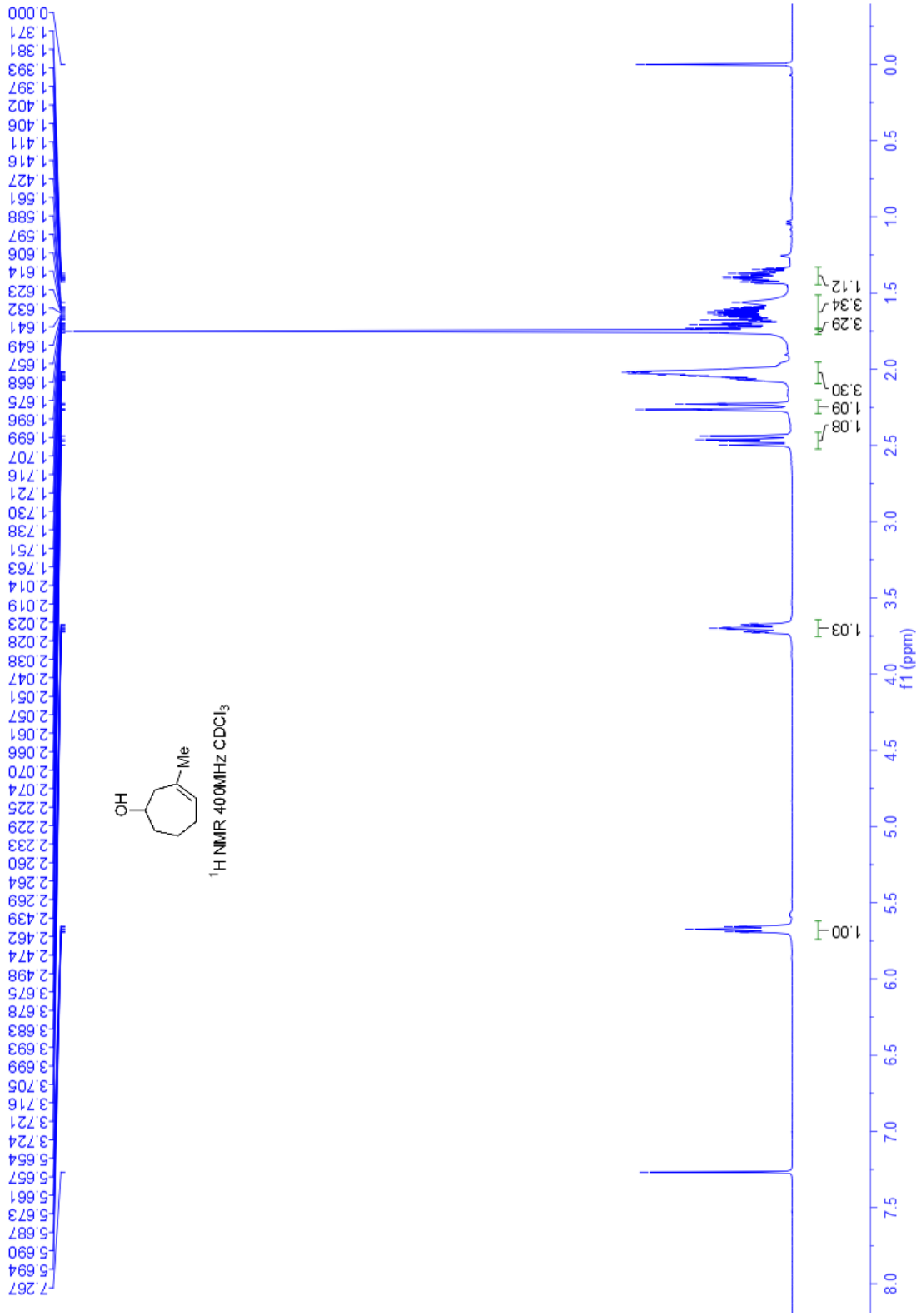
1,6,6-trimethylcyclohept-3-enol (4h).

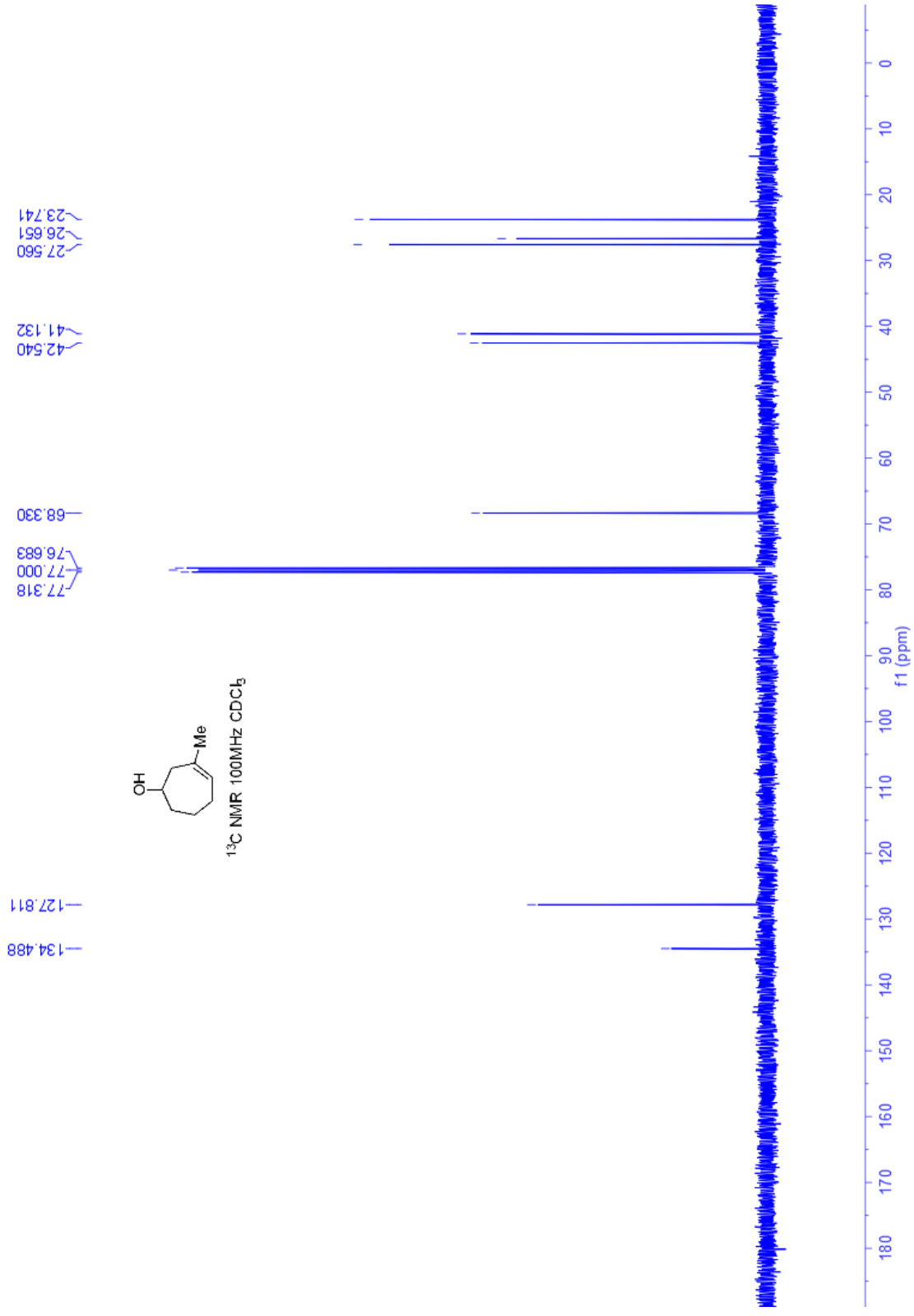


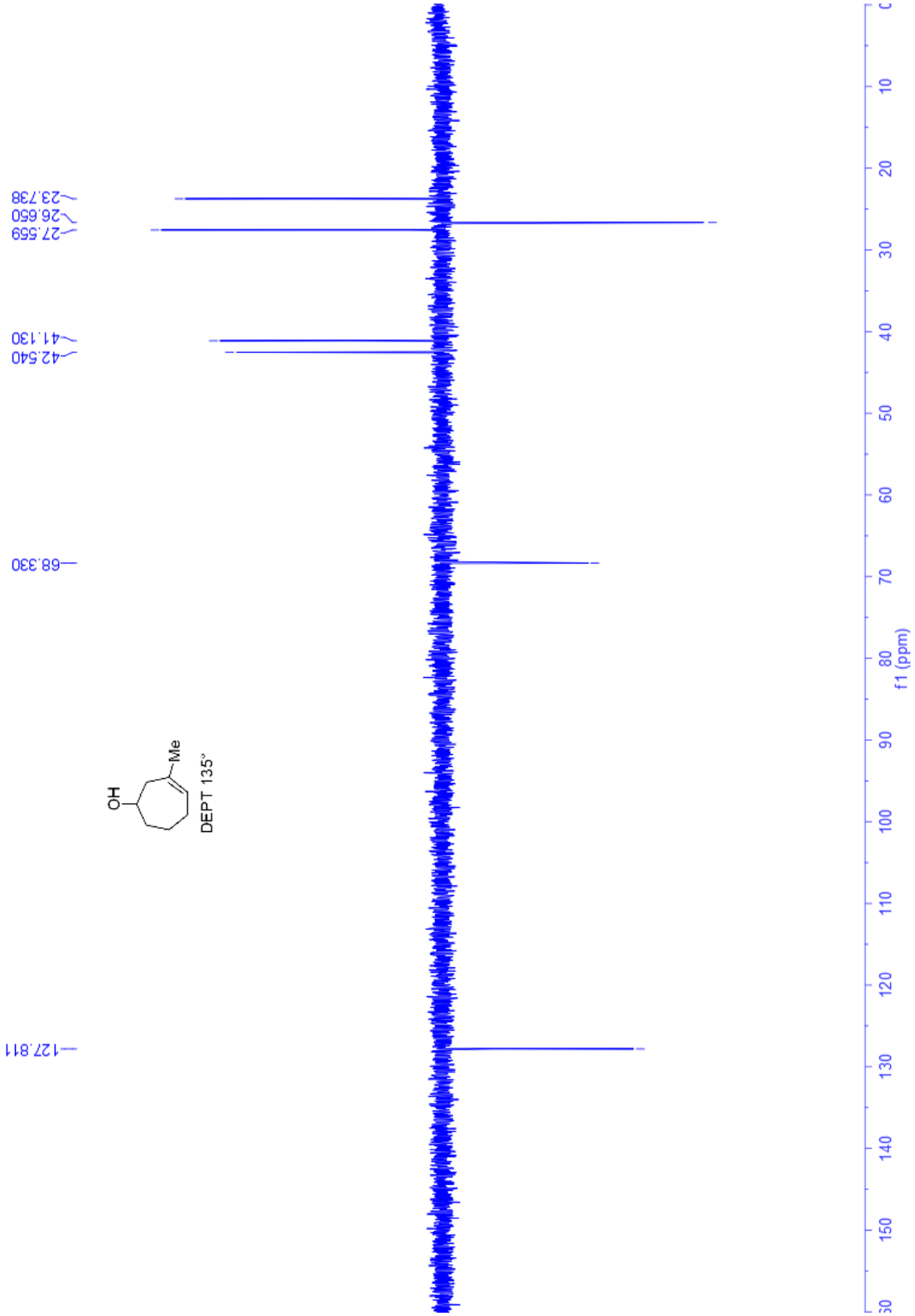

29.555
31.659
32.142
32.850
40.197
40.511
58.186
71.371
76.682
77.000
77.318
127.865
132.914
OH
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


132.957
127.905
58.223
40.549
40.242
32.186
31.703
29.592
OH
DEPT 135°
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

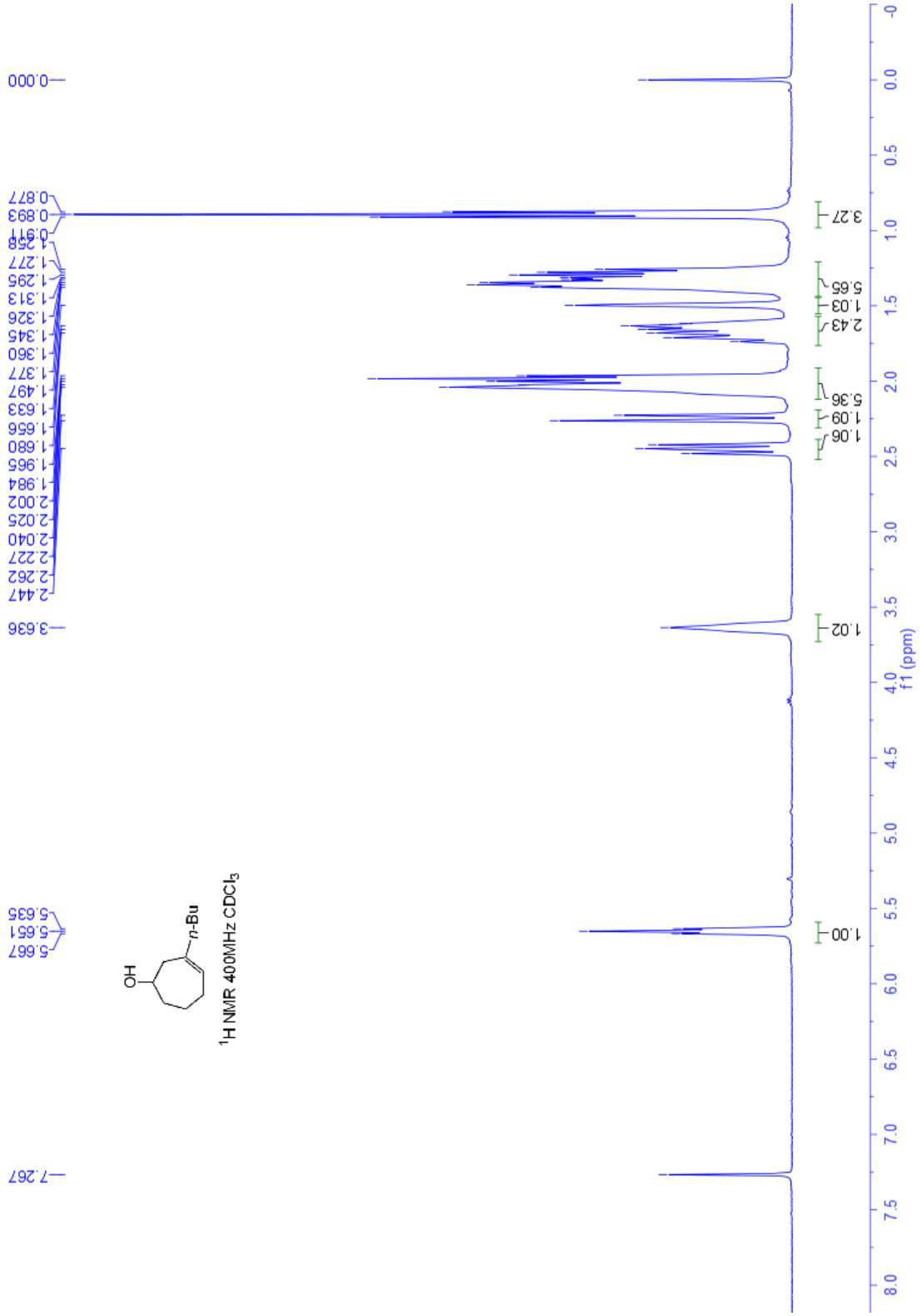
3-methylcyclohept-3-enol (4i).

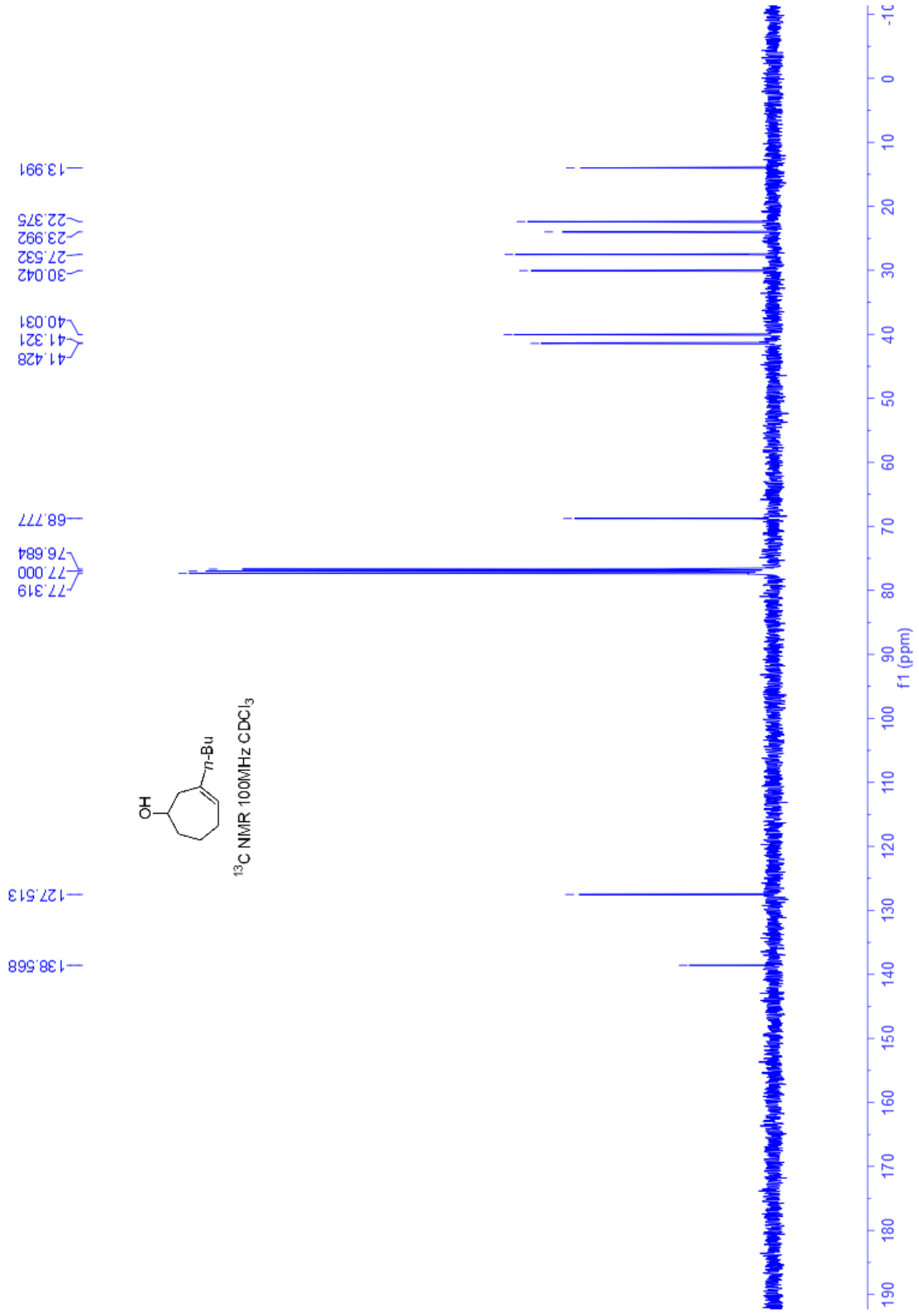


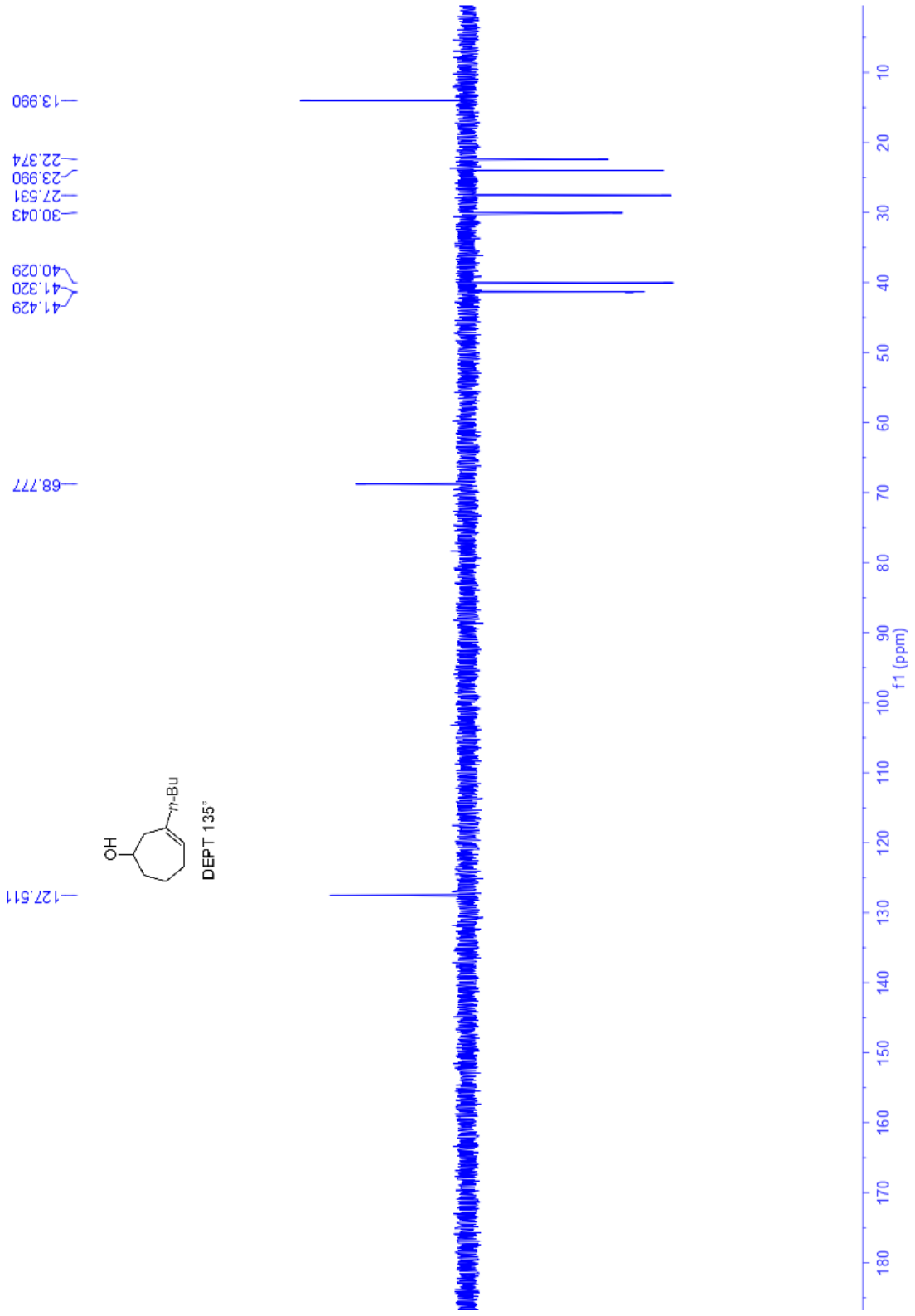

23.741
26.651
27.560
41.132
42.540
68.330
76.683
77.000
77.318
127.811
134.488
OH
Me
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


23.738
26.650
27.559
41.130
42.540
68.330
127.811
OH
Me
DEPT 135°
f1 (ppm)

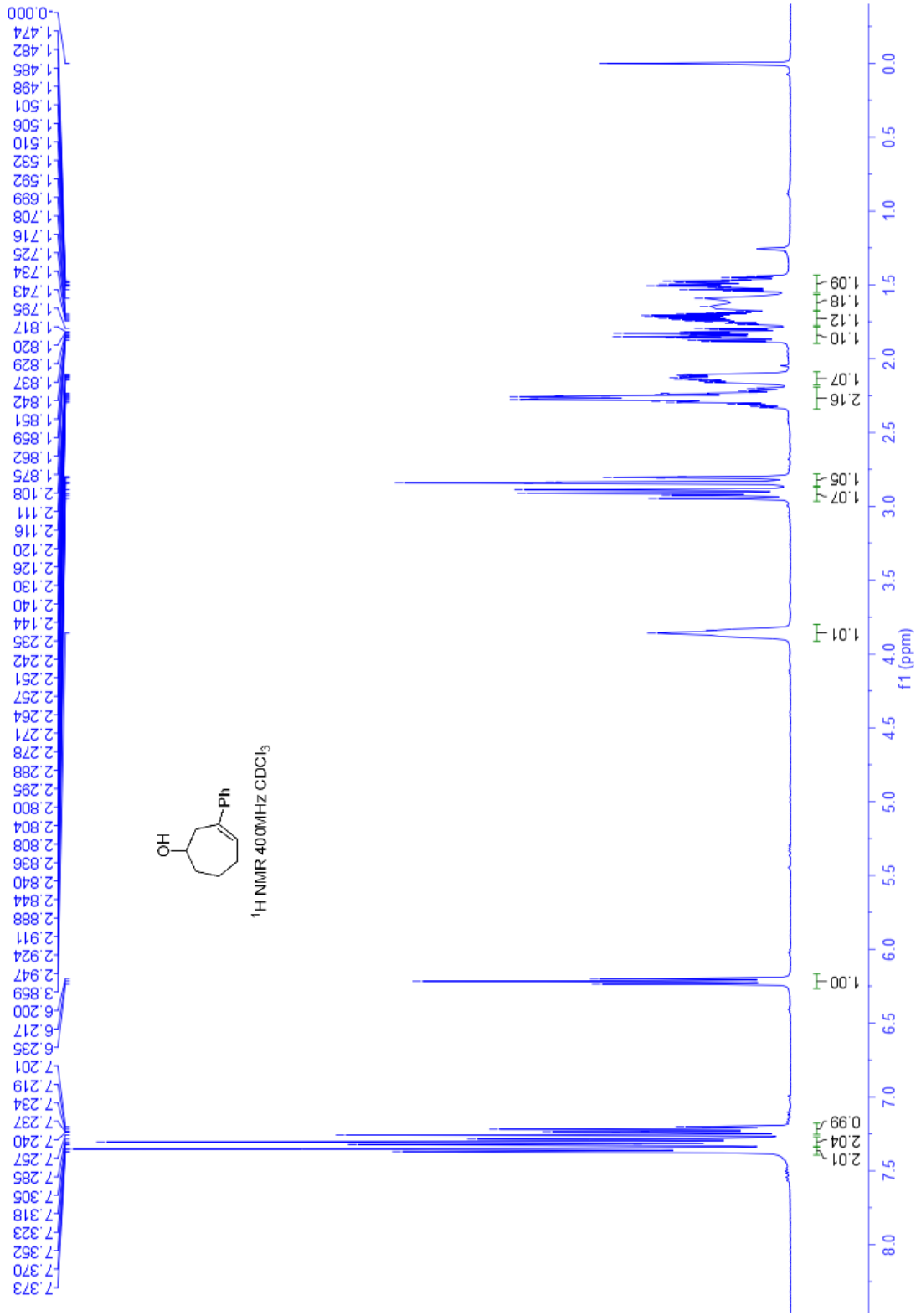
3-butylcyclohept-3-enol (4j).

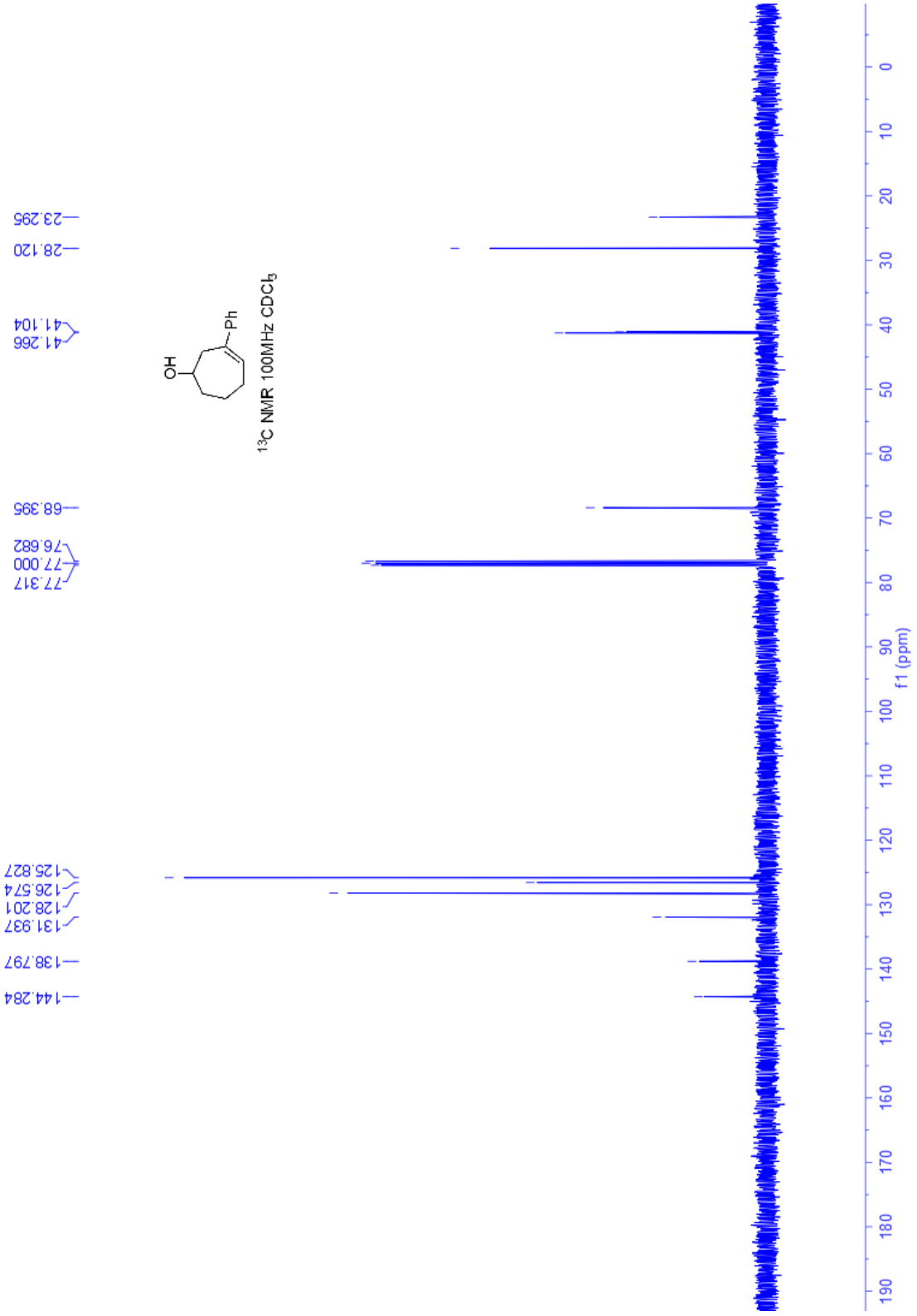


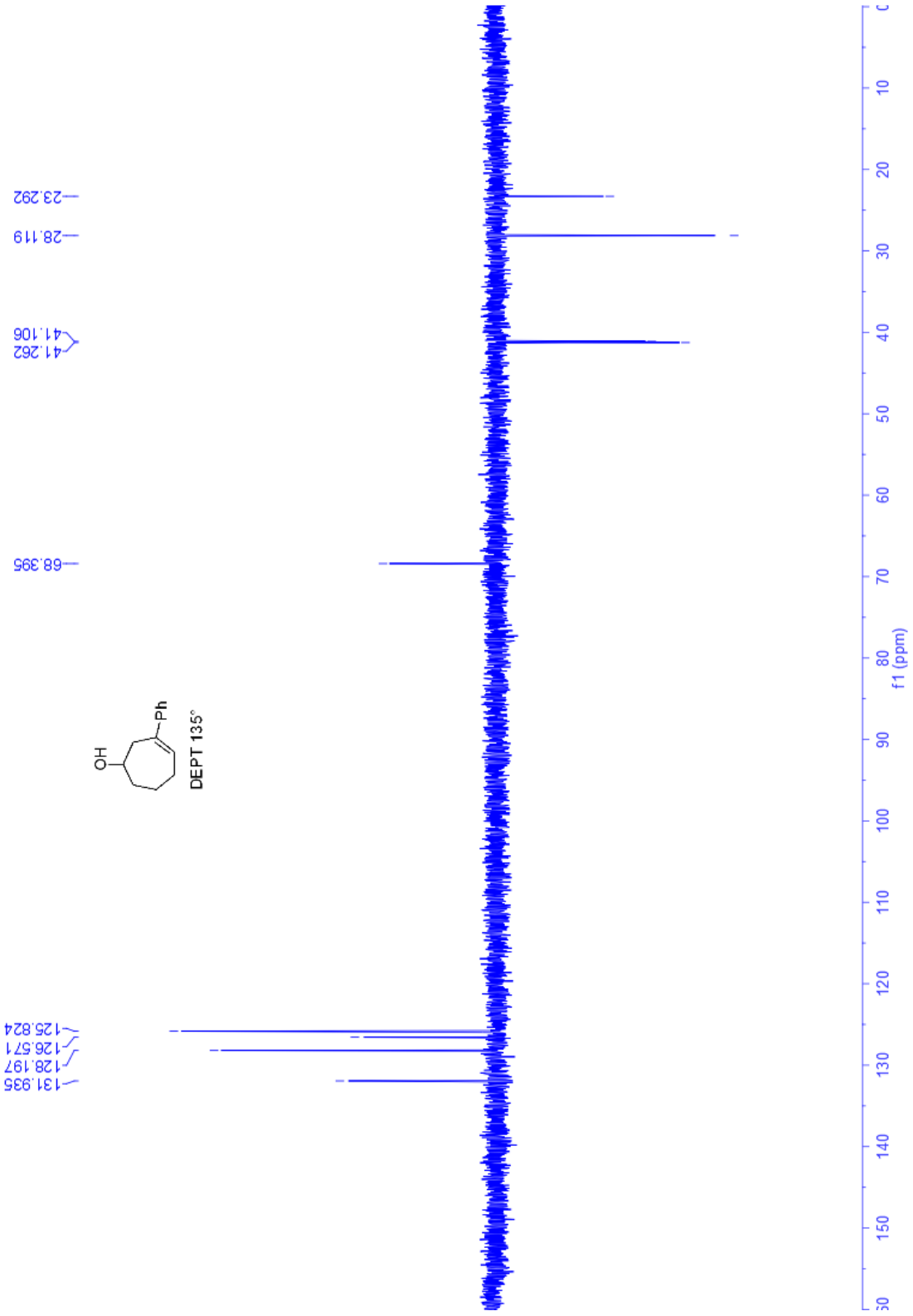

13.991
22.375
23.992
27.532
30.042
40.031
41.321
41.428
68.777
76.684
77.000
77.319
127.513
138.568
OH
n-Bu
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


13.990
22.374
23.990
27.531
30.043
40.029
41.320
41.429
68.777
127.511
OH
n-Bu
DEPT 135°
f1 (ppm)

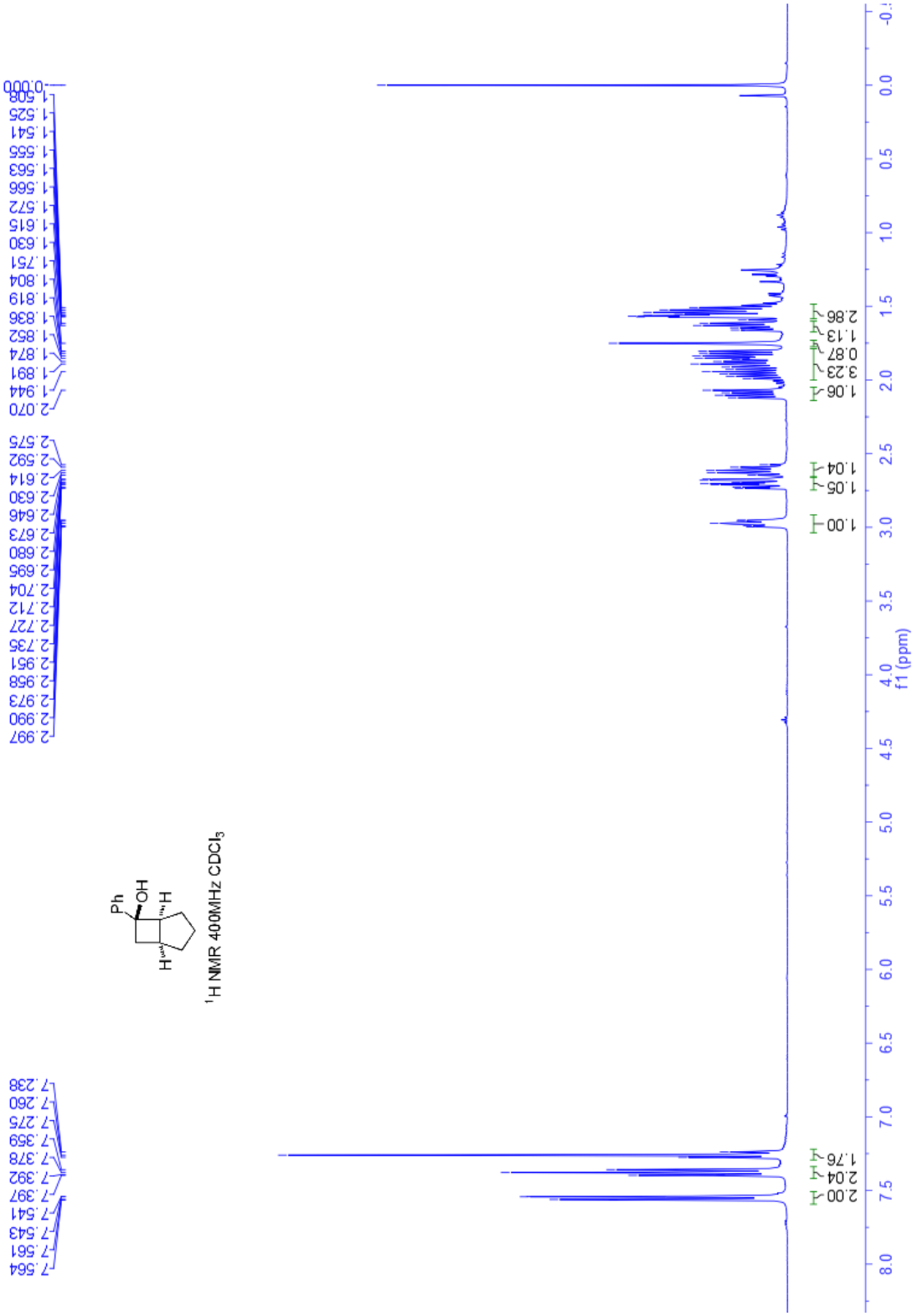
3-phenylcyclohept-3-enol (4k-1).

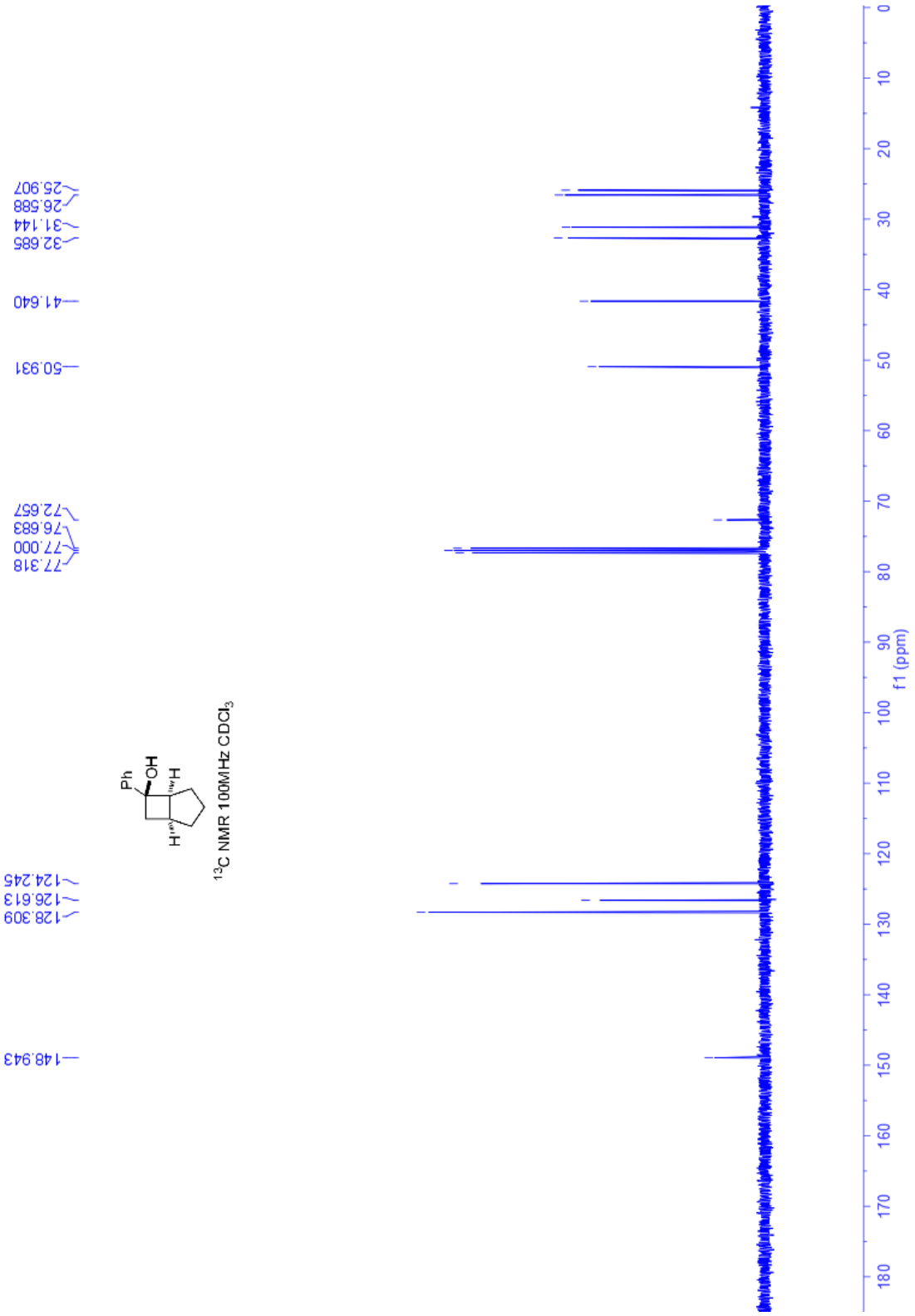


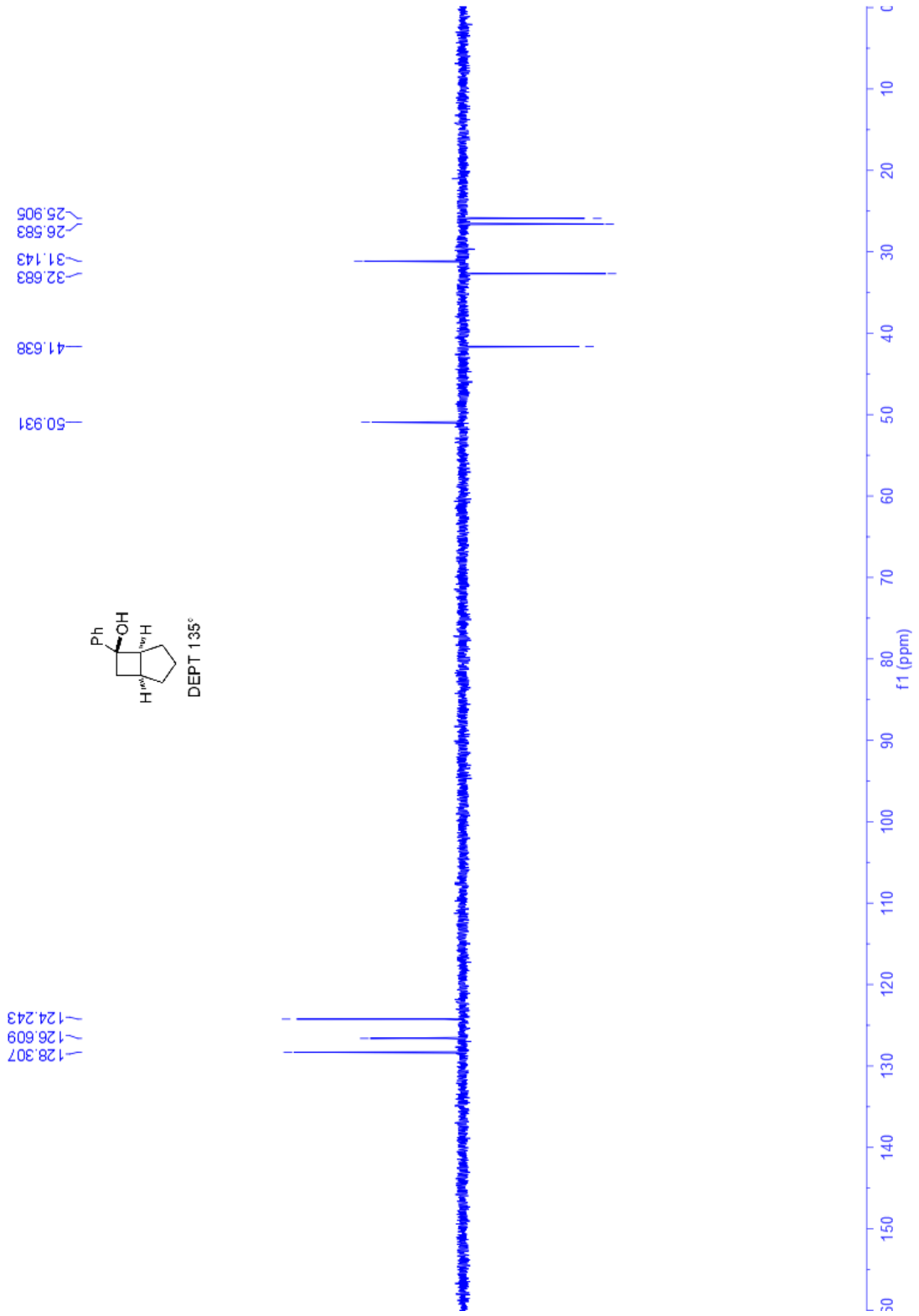

23.295
28.120
41.104
41.266
68.395
76.682
77.000
77.317
125.827
126.574
128.201
131.937
138.797
144.284
OH
Ph
^{13}C NMR 100MHz CDCl$_3$
f1 (ppm)


23.292
28.119
41.106
41.262
68.395
125.824
126.571
128.197
131.935
OH
Ph
DEPT 135°
f1 (ppm)

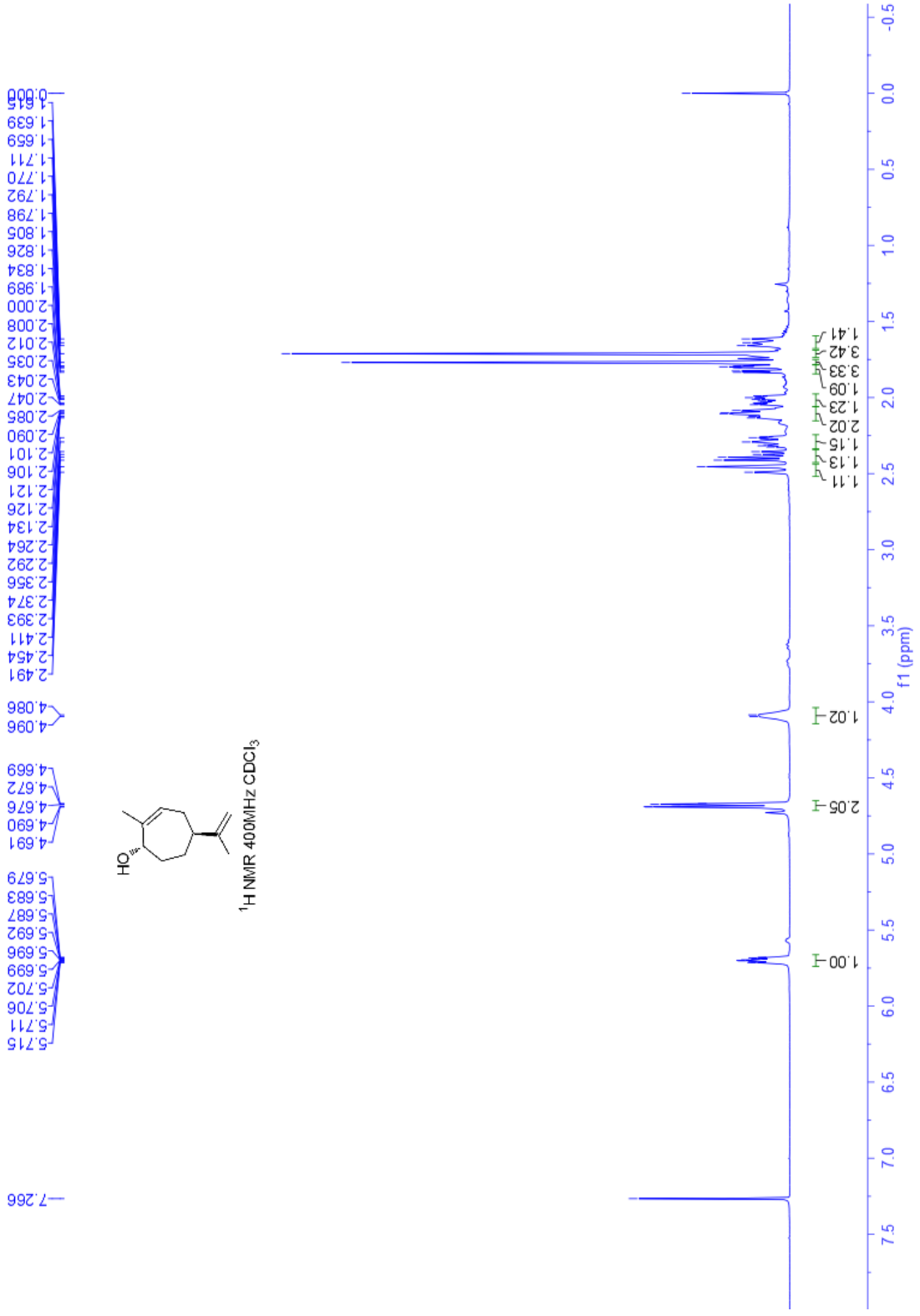
6-phenylbicyclo[3.2.0]heptan-6-ol (4k-2).

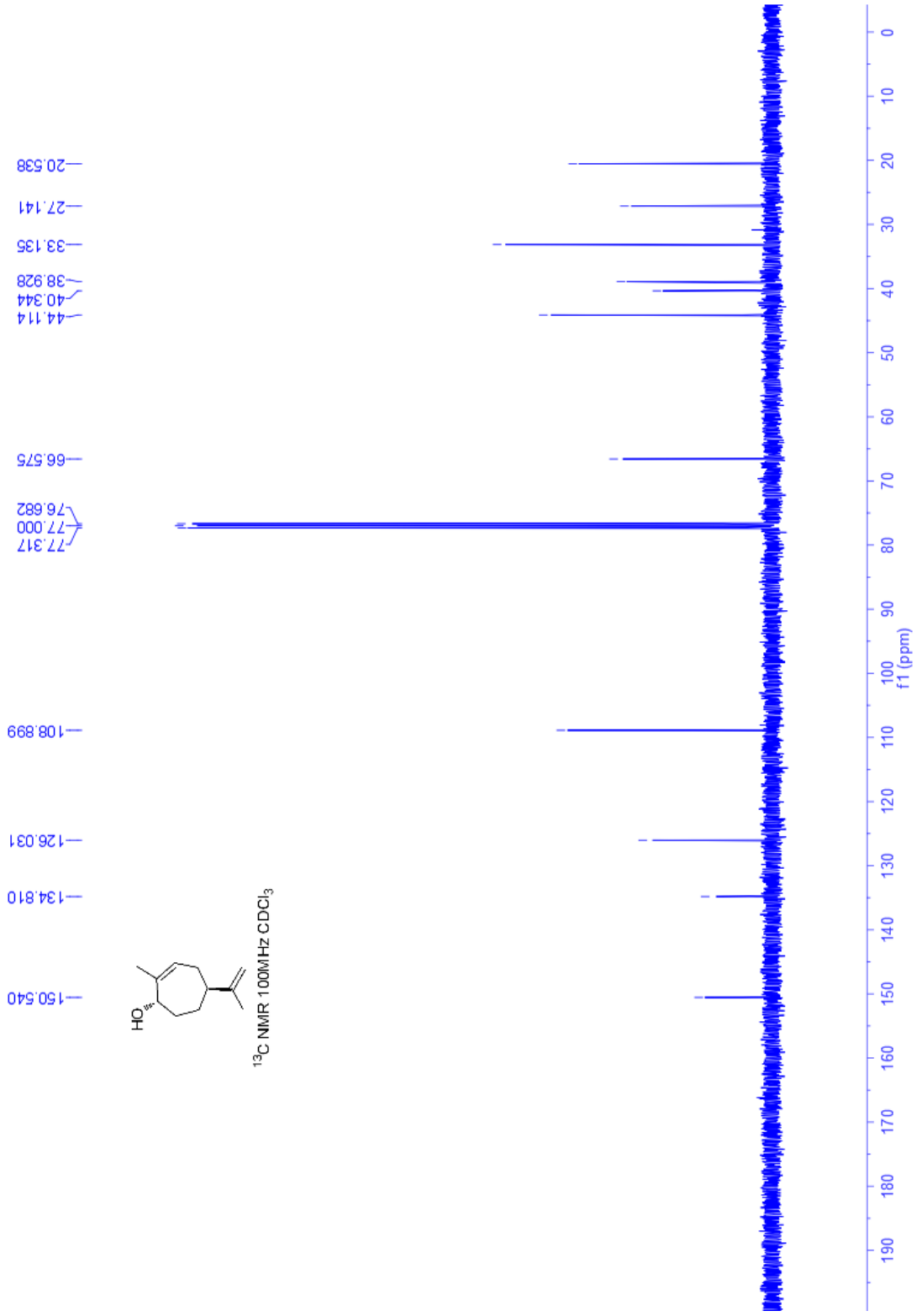



25.907
26.588
31.144
32.685
41.640
50.931
72.657
76.683
77.000
77.318
124.245
126.613
128.309
148.943
Ph
OH
H
H
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


DEPT 135°
Ph
OH
H
H
25.905
26.583
31.143
32.683
41.638
50.931
124.243
126.609
128.307
f1 (ppm)

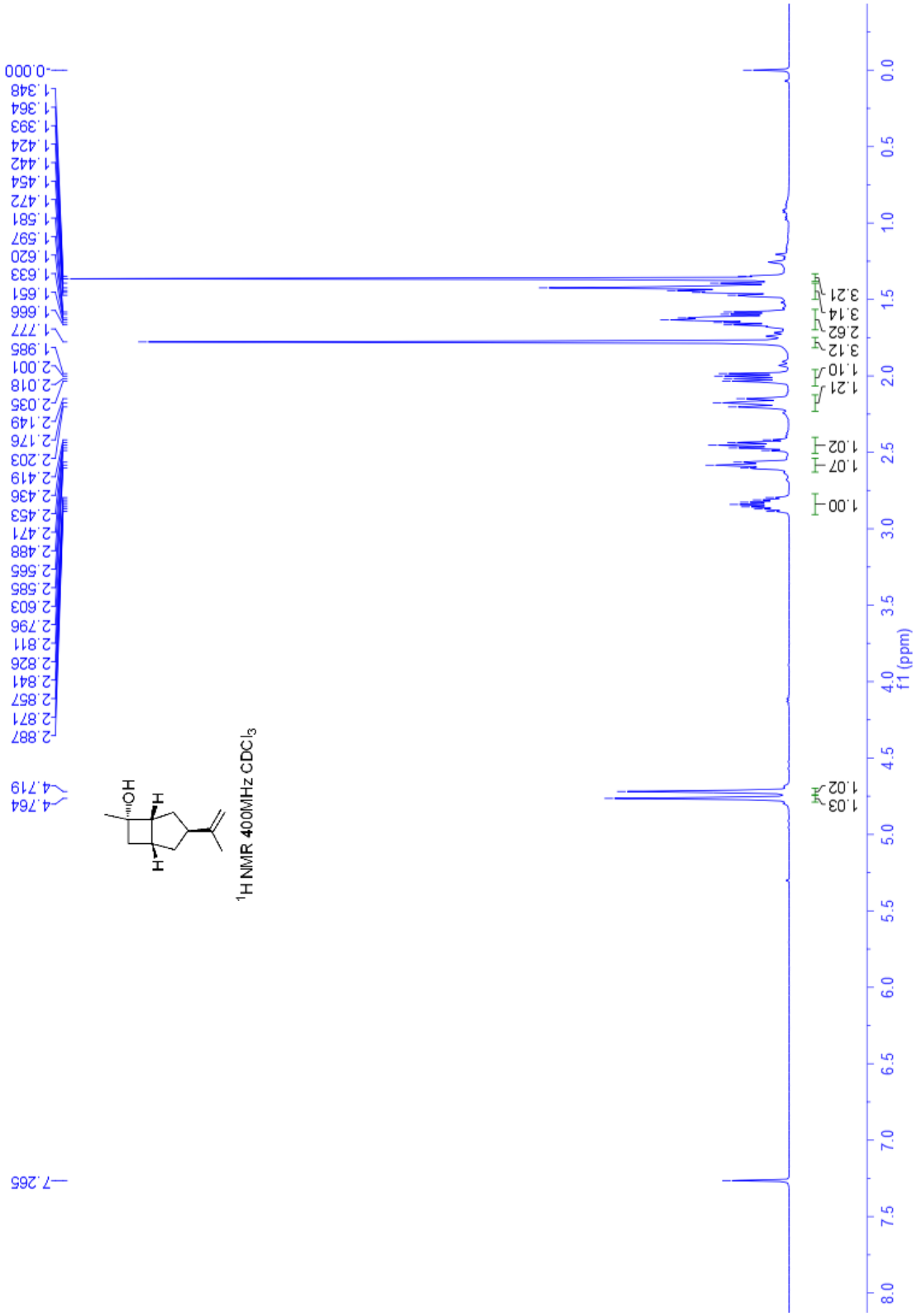
2-methyl-5-(prop-1-en-2-yl)cyclohept-2-enol (4l-1).

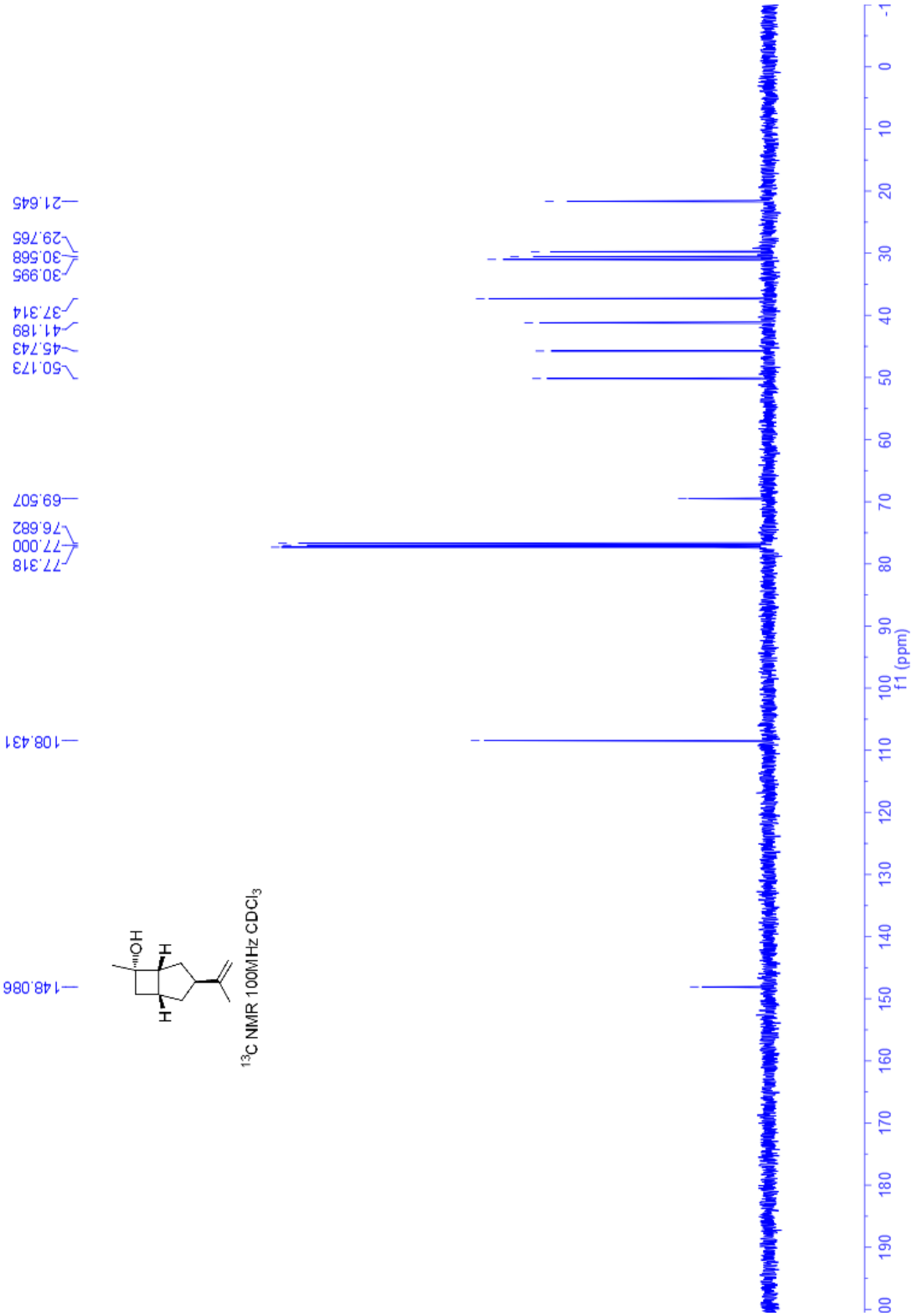


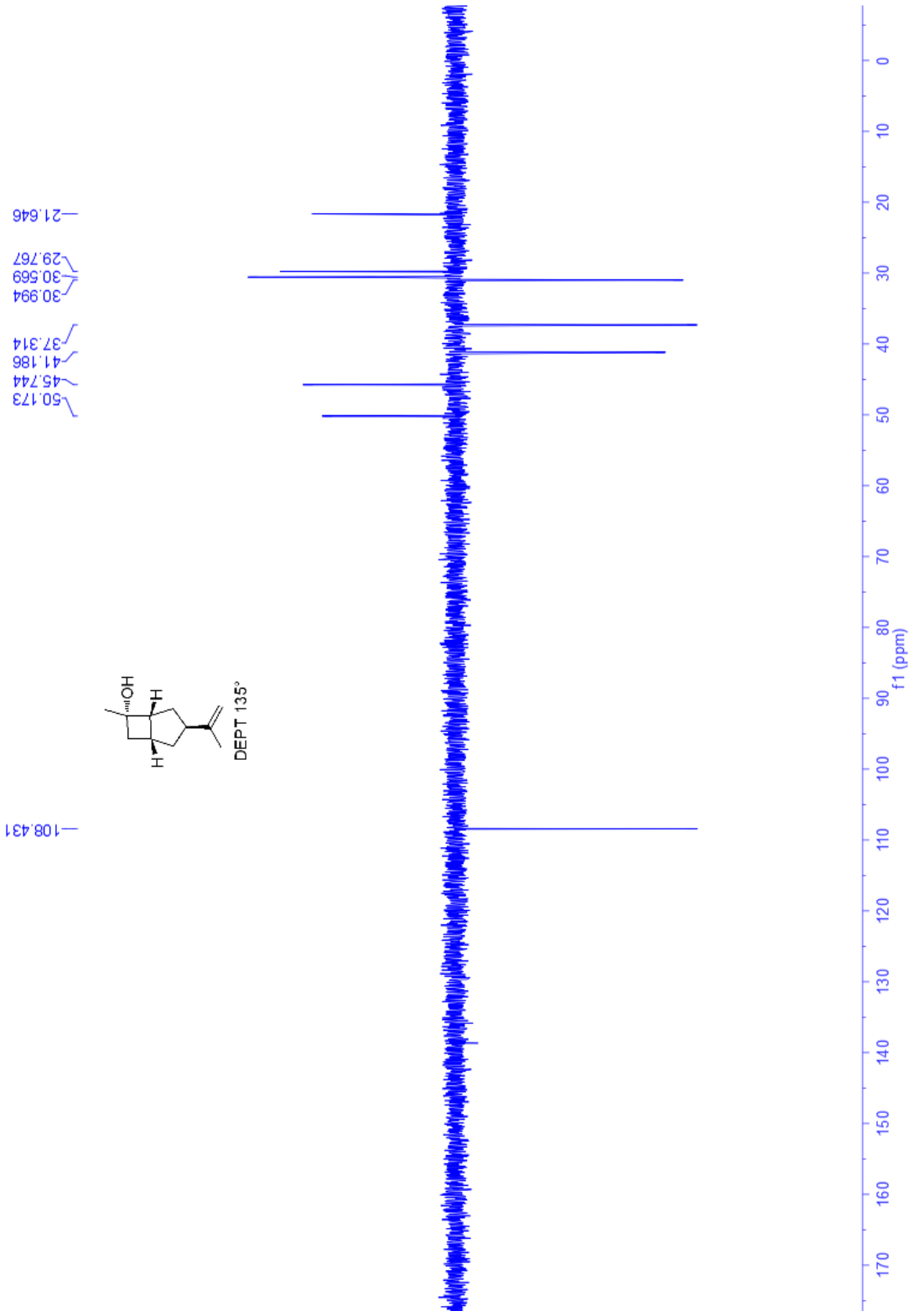

20.538
27.141
33.135
38.928
40.344
44.114
66.575
76.682
77.000
77.317
108.899
126.031
134.810
150.540
HO
^{13}C NMR 100MHz $CDCl_3$
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
f1 (ppm)

20.528
27.122
33.107
38.970
40.385
44.071
66.575
125.955
108.894
DEPT 135°
HO
f1 (ppm)

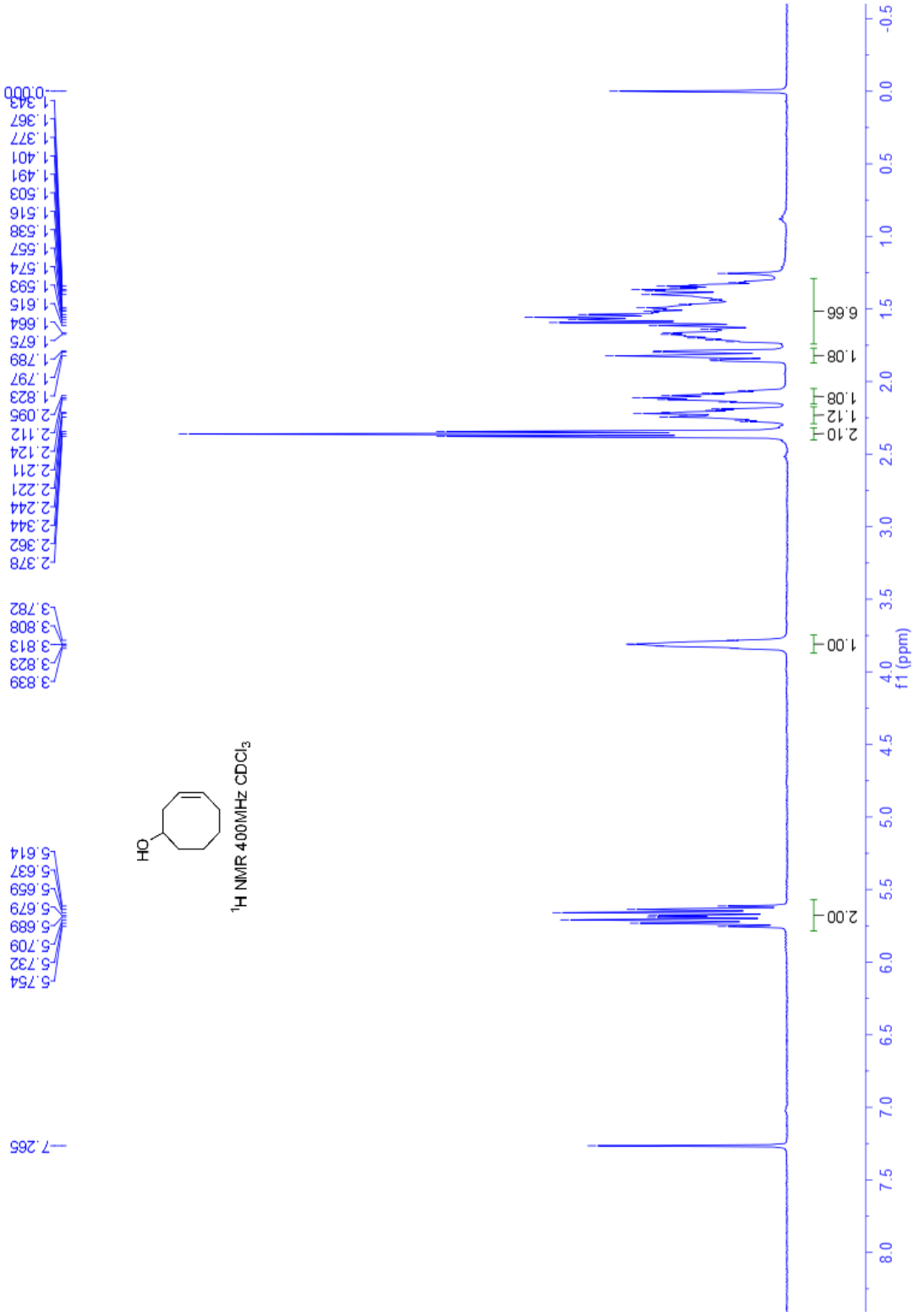
6-methyl-3-(prop-1-en-2-yl)bicyclo[3.2.0]heptan-6-ol (4l-2).

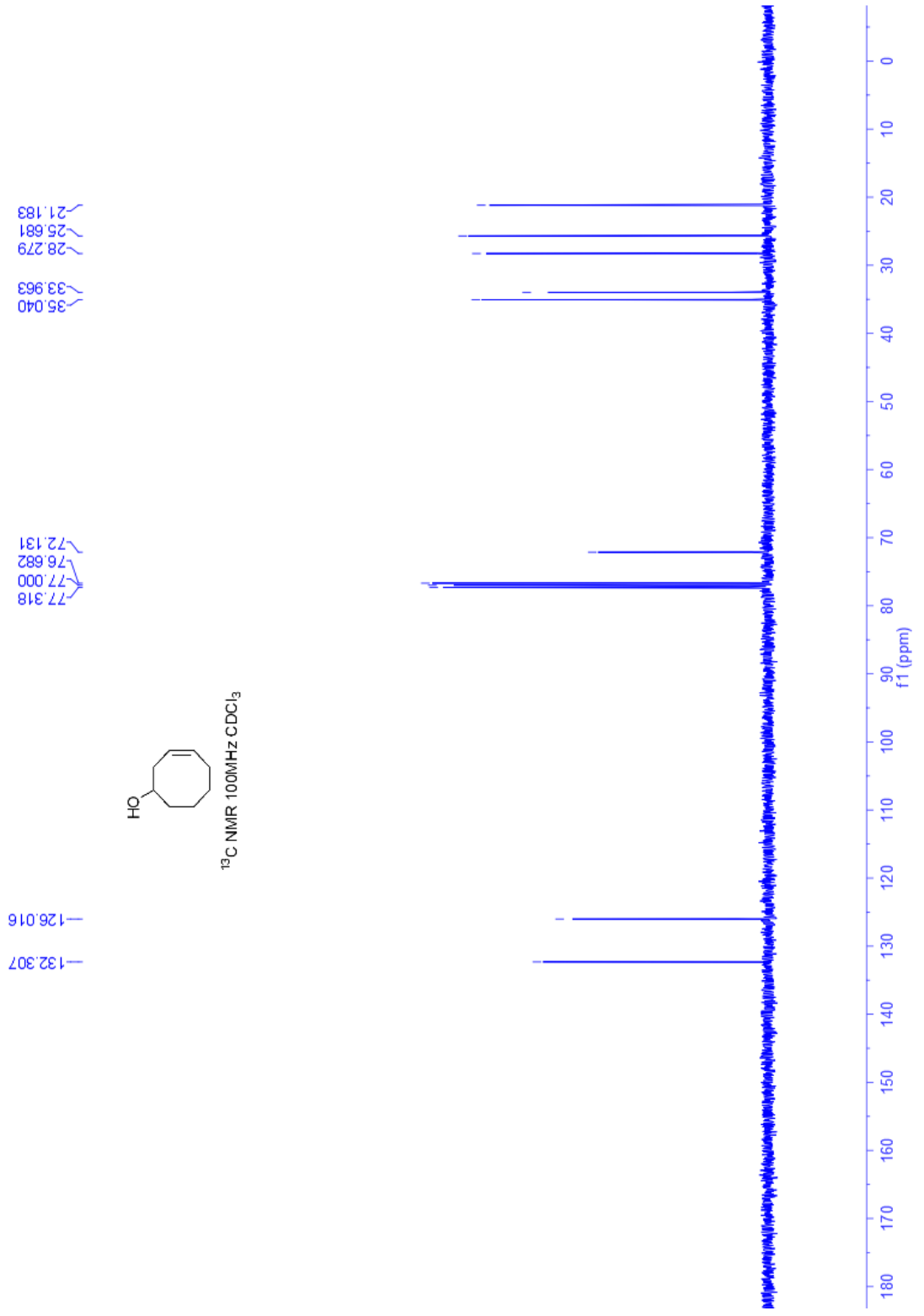


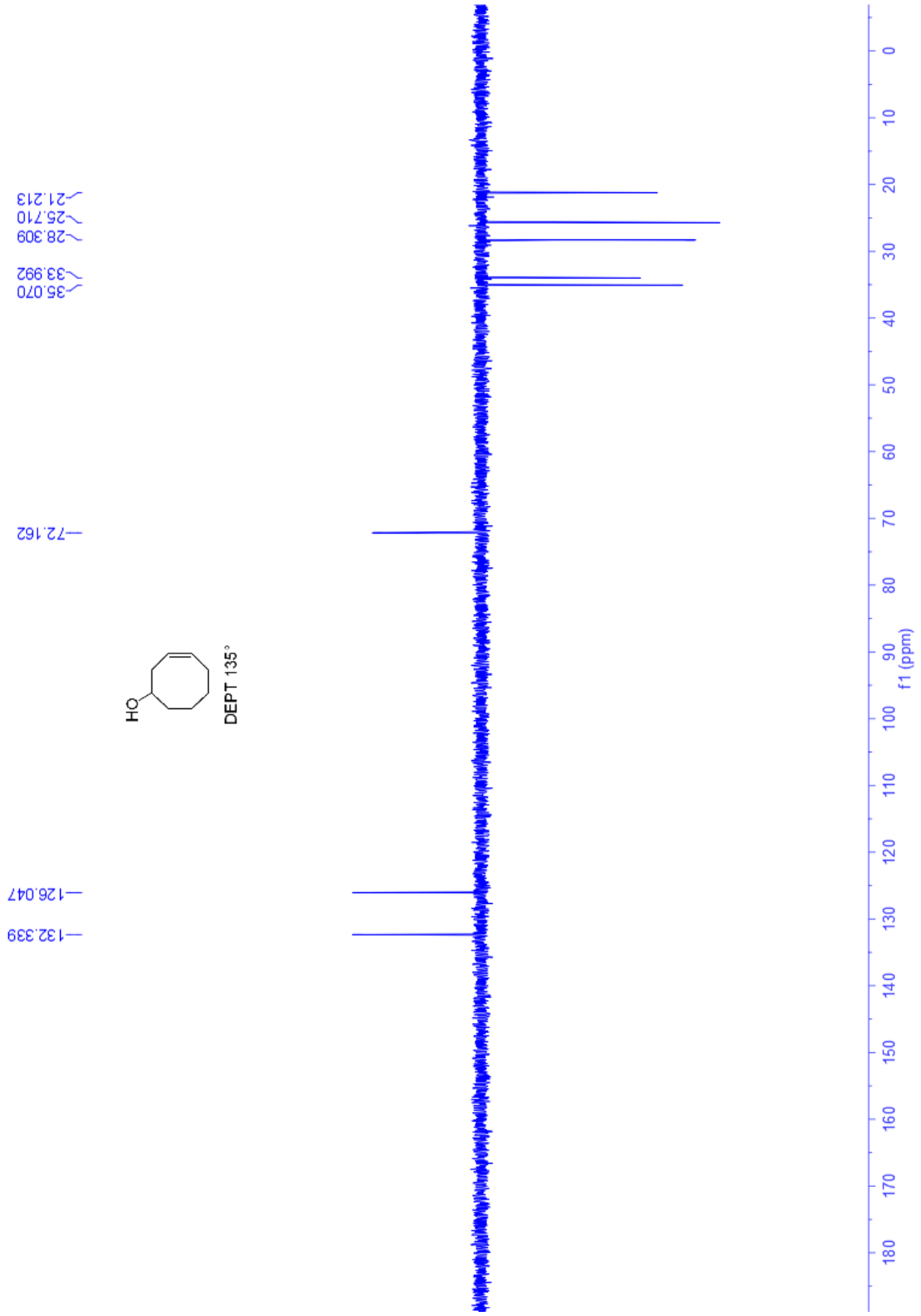

148.086
108.431
69.507
77.318
77.000
76.682
50.173
45.743
41.189
37.314
30.995
30.568
29.765
21.645
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


21.646
29.767
30.569
30.994
37.314
41.186
45.744
50.173
108.431
DEPT 135°
OH
H
H
f1 (ppm)

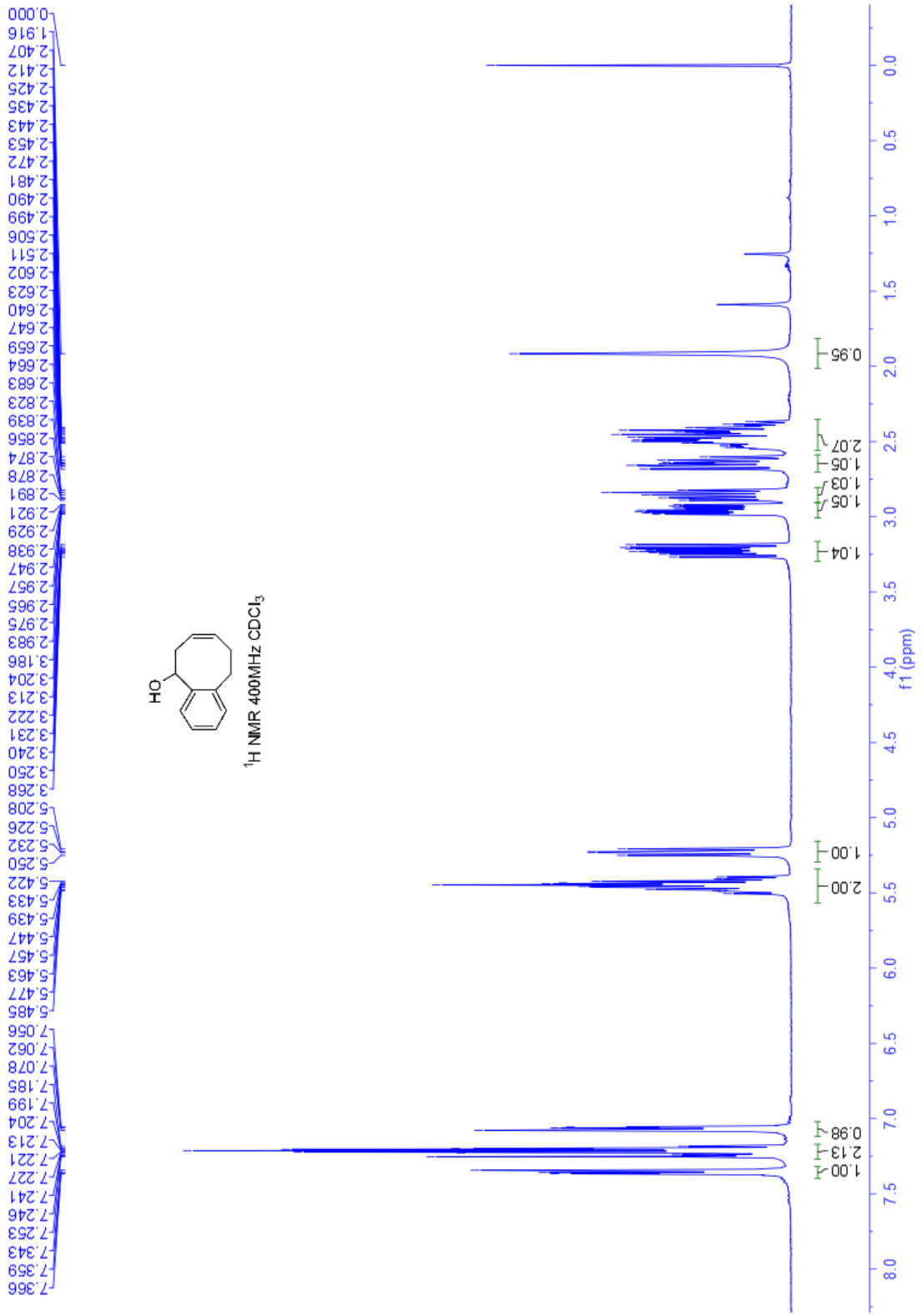
cyclooct-3-enol (4m).

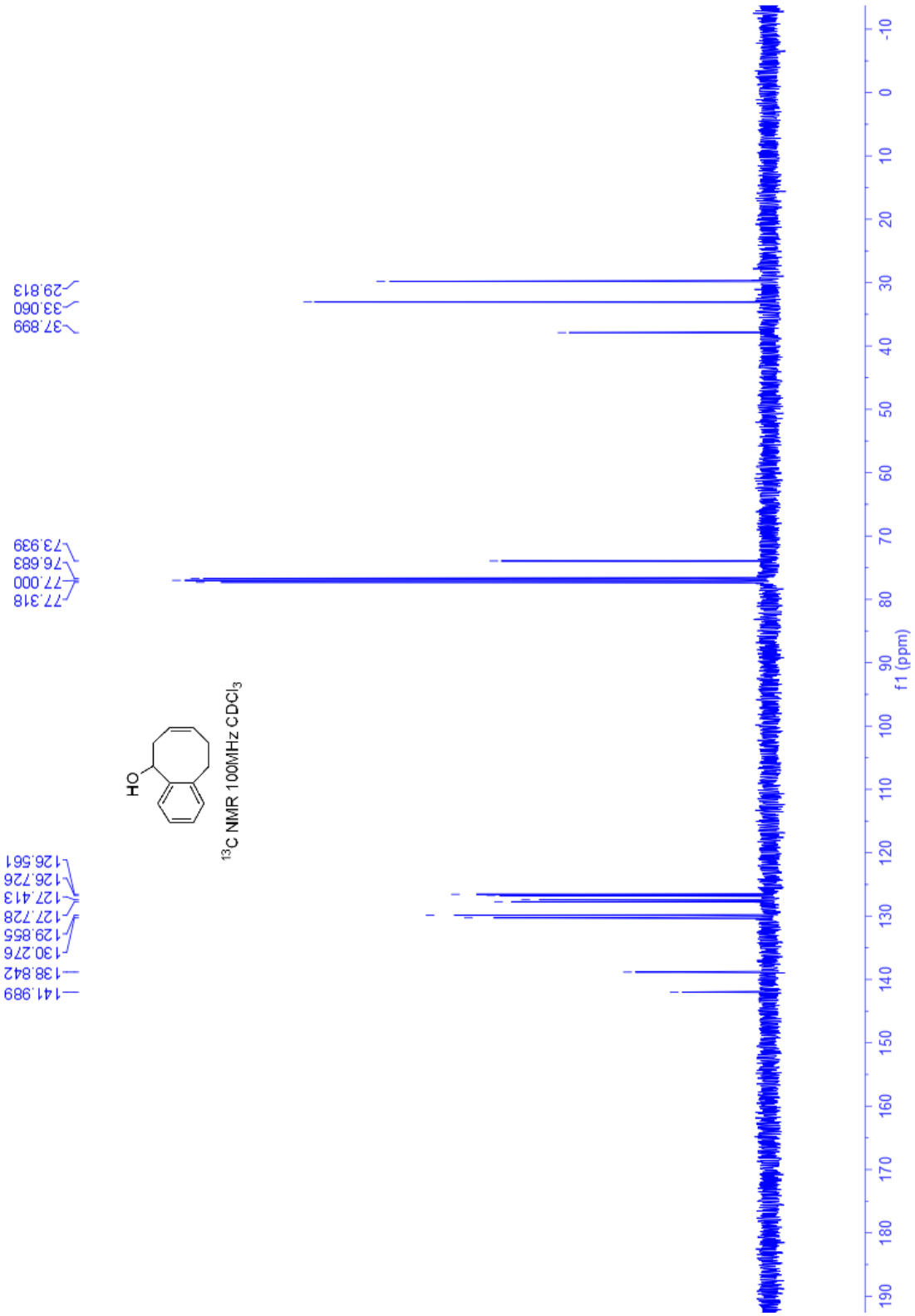


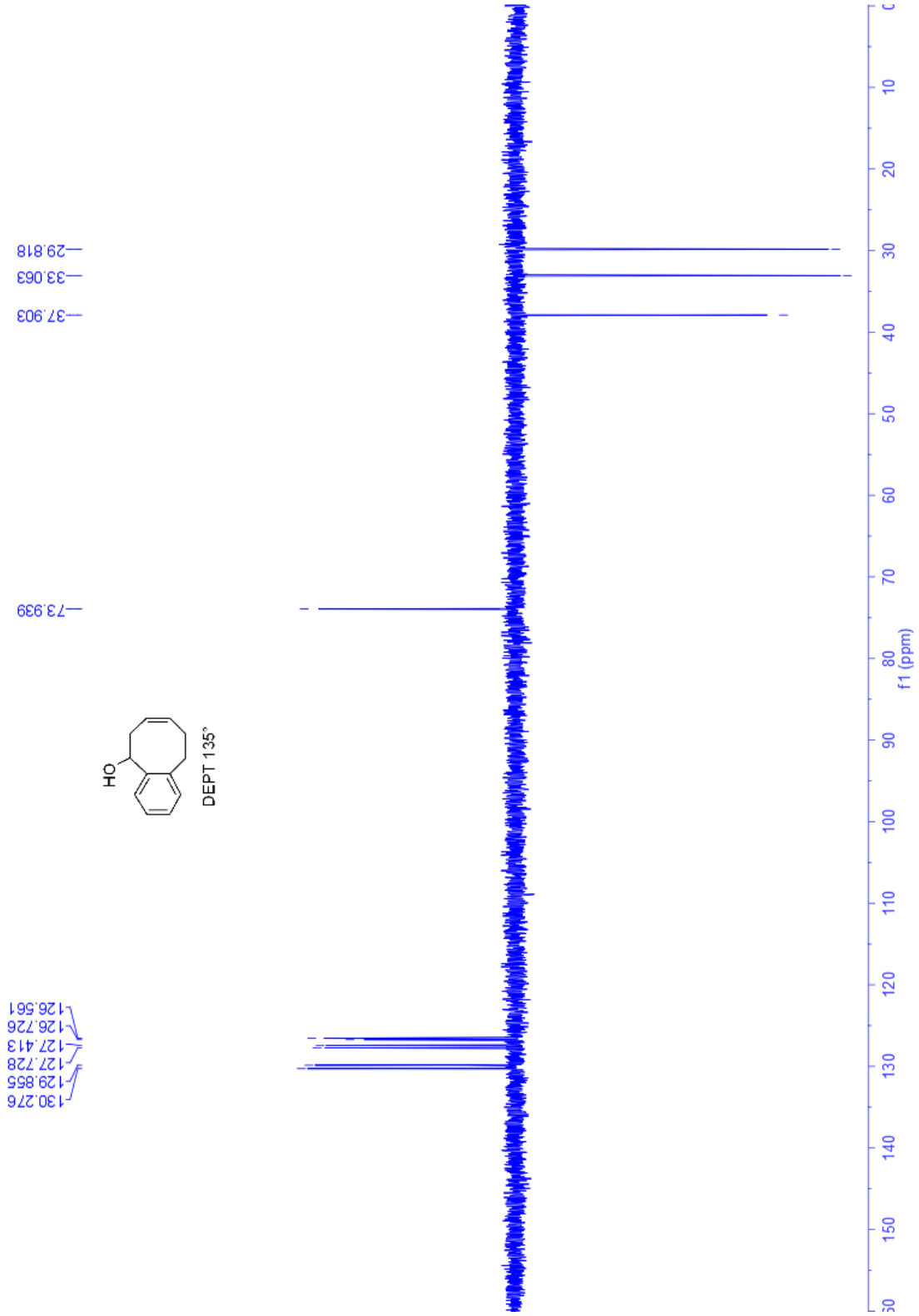

^{13}C NMR 100MHz $CDCl_3$
HO
21.183
25.681
28.279
33.963
35.040
72.131
76.682
77.000
77.318
126.016
132.307
f1 (ppm)


21.213
25.710
28.309
33.992
35.070
72.162
126.047
132.339
DEPT 135°
OH
f1 (ppm)

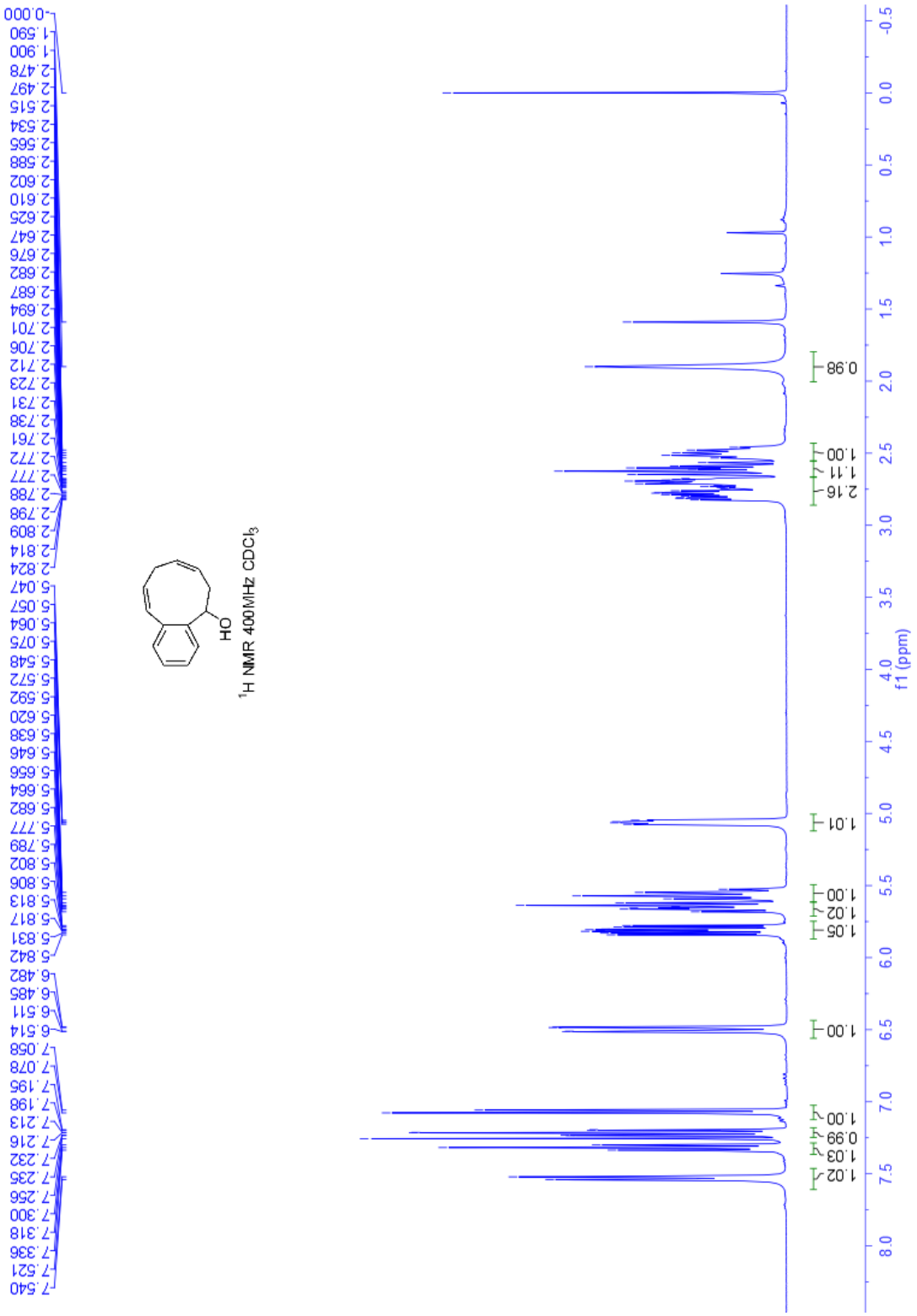
Compound **4n.**

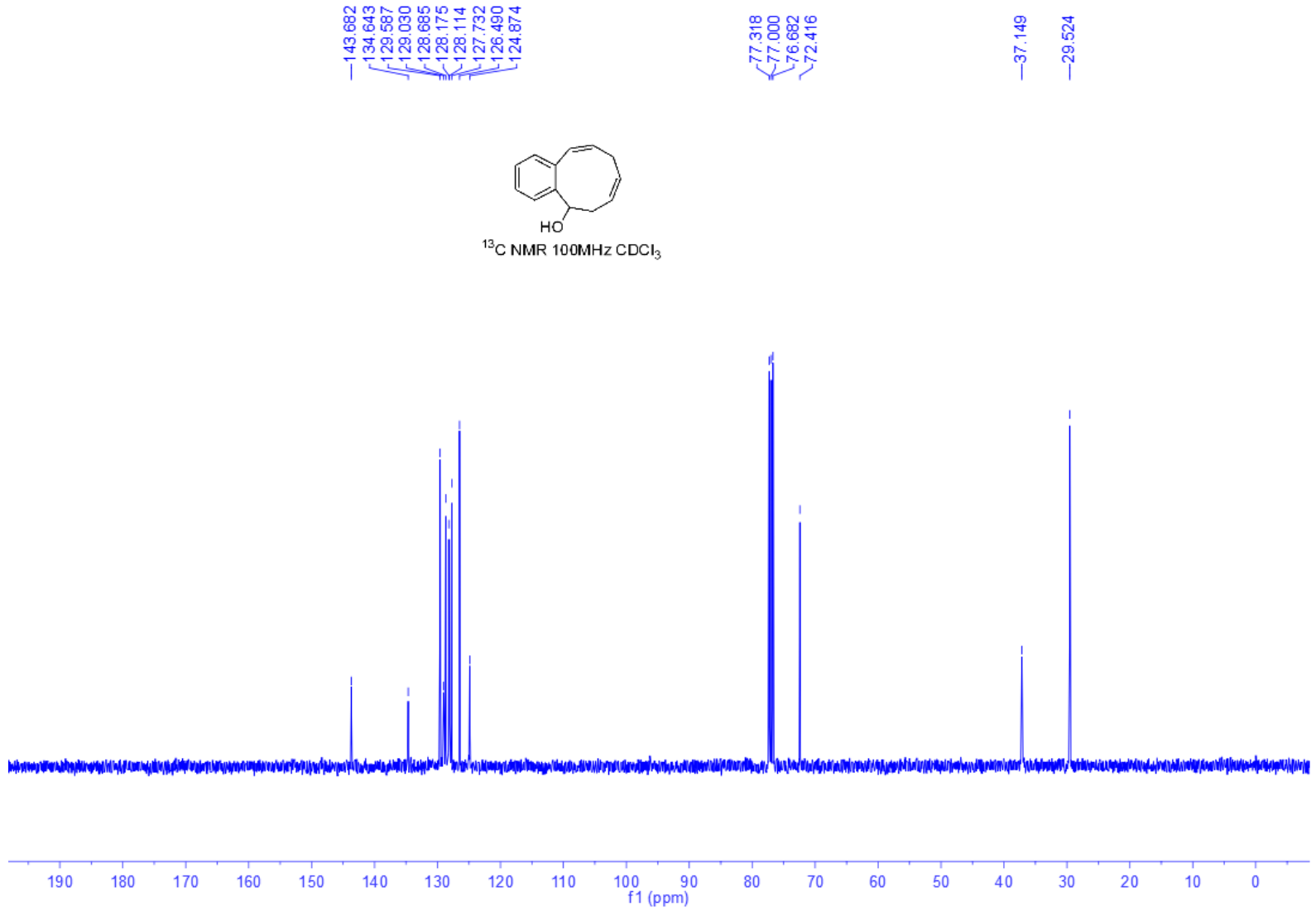


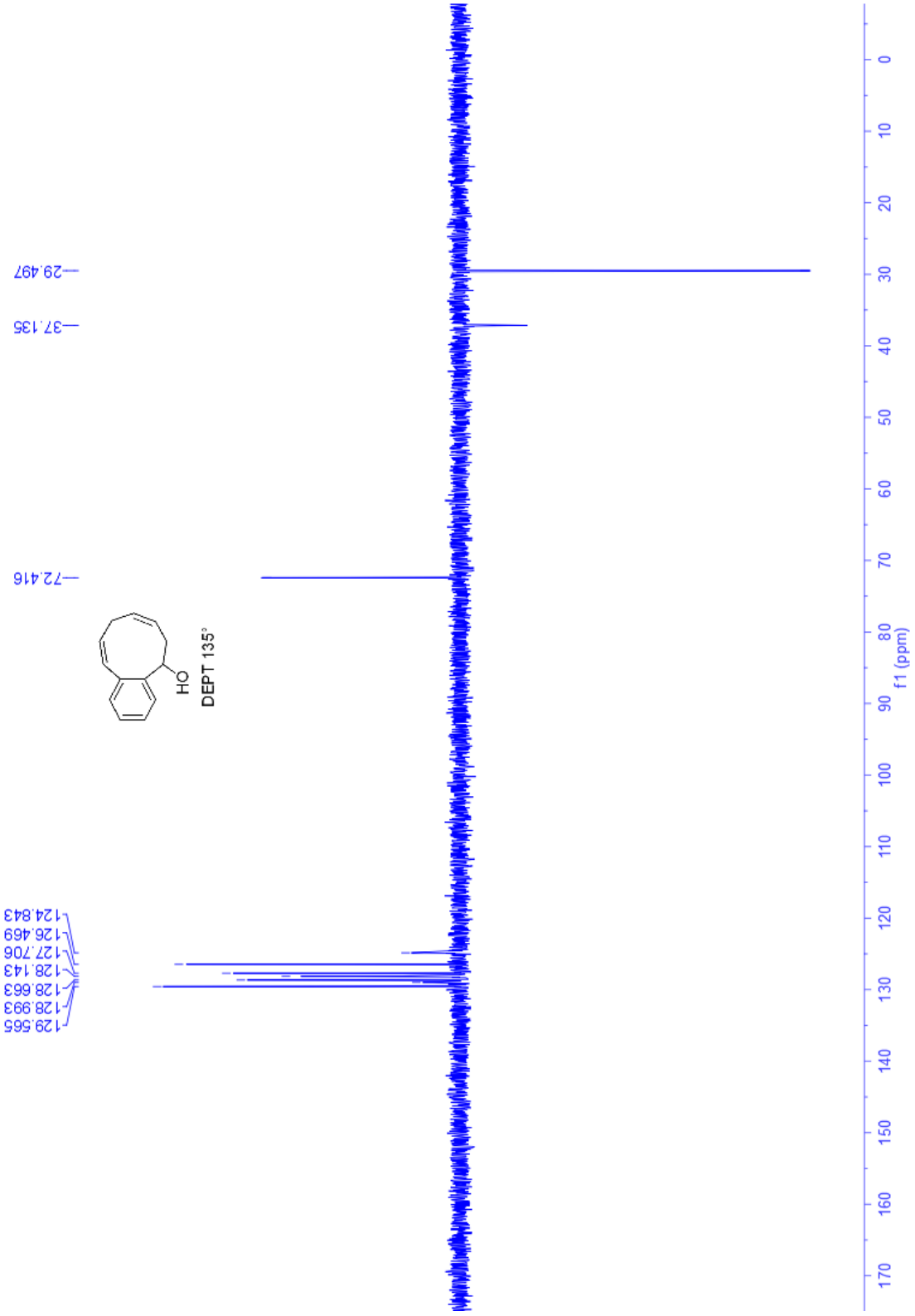

29.813
33.060
37.899
73.939
76.683
77.000
77.318
126.561
126.726
127.413
127.728
129.855
130.276
138.842
141.989
HO
^{13}C NMR 100MHz CDCl$_3$
f1 (ppm)


29.818
33.063
37.903
73.939
126.561
126.726
127.413
127.728
129.855
130.276
HO
DEPT 135°
f1 (ppm)

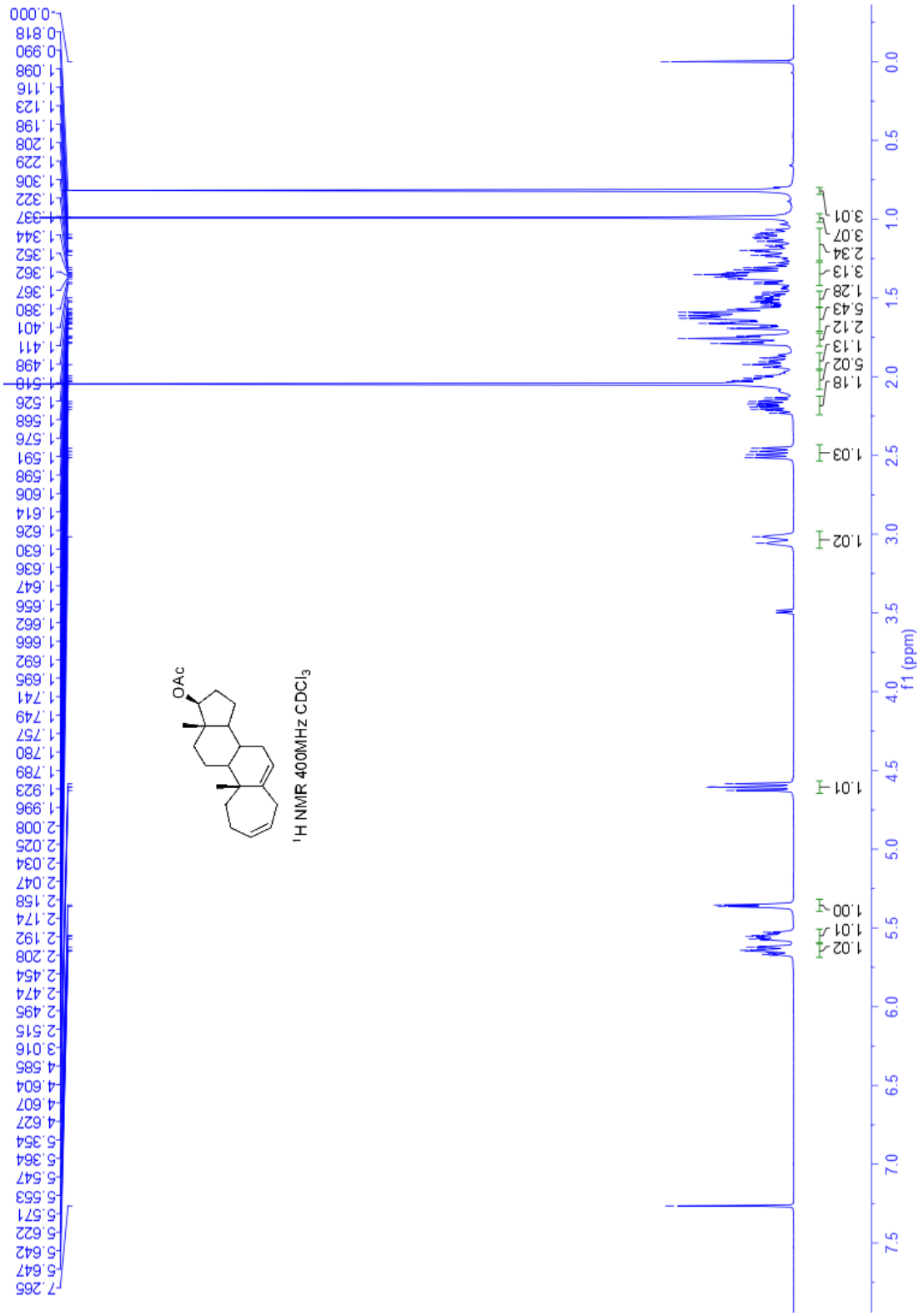
Compound **4o.**

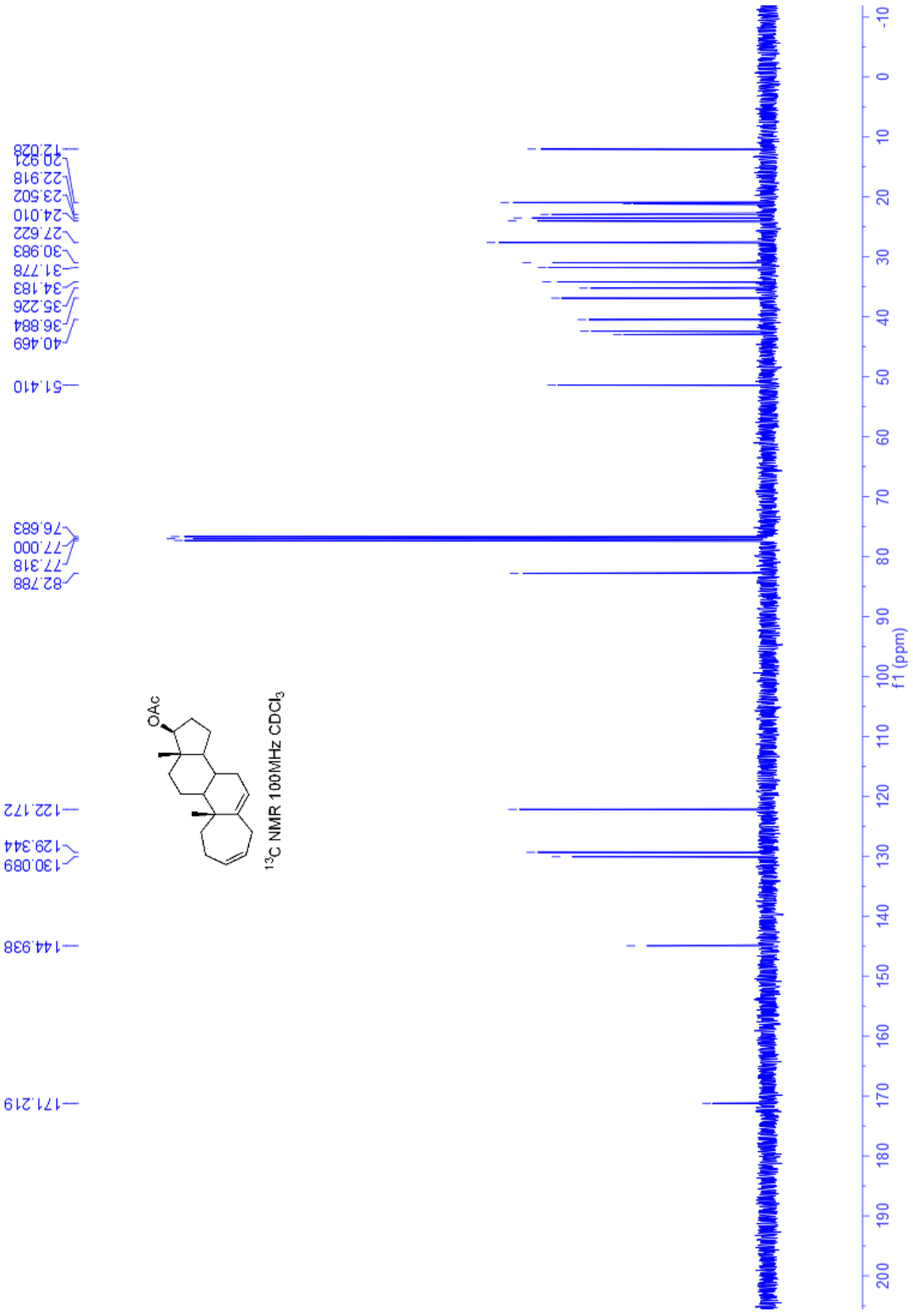


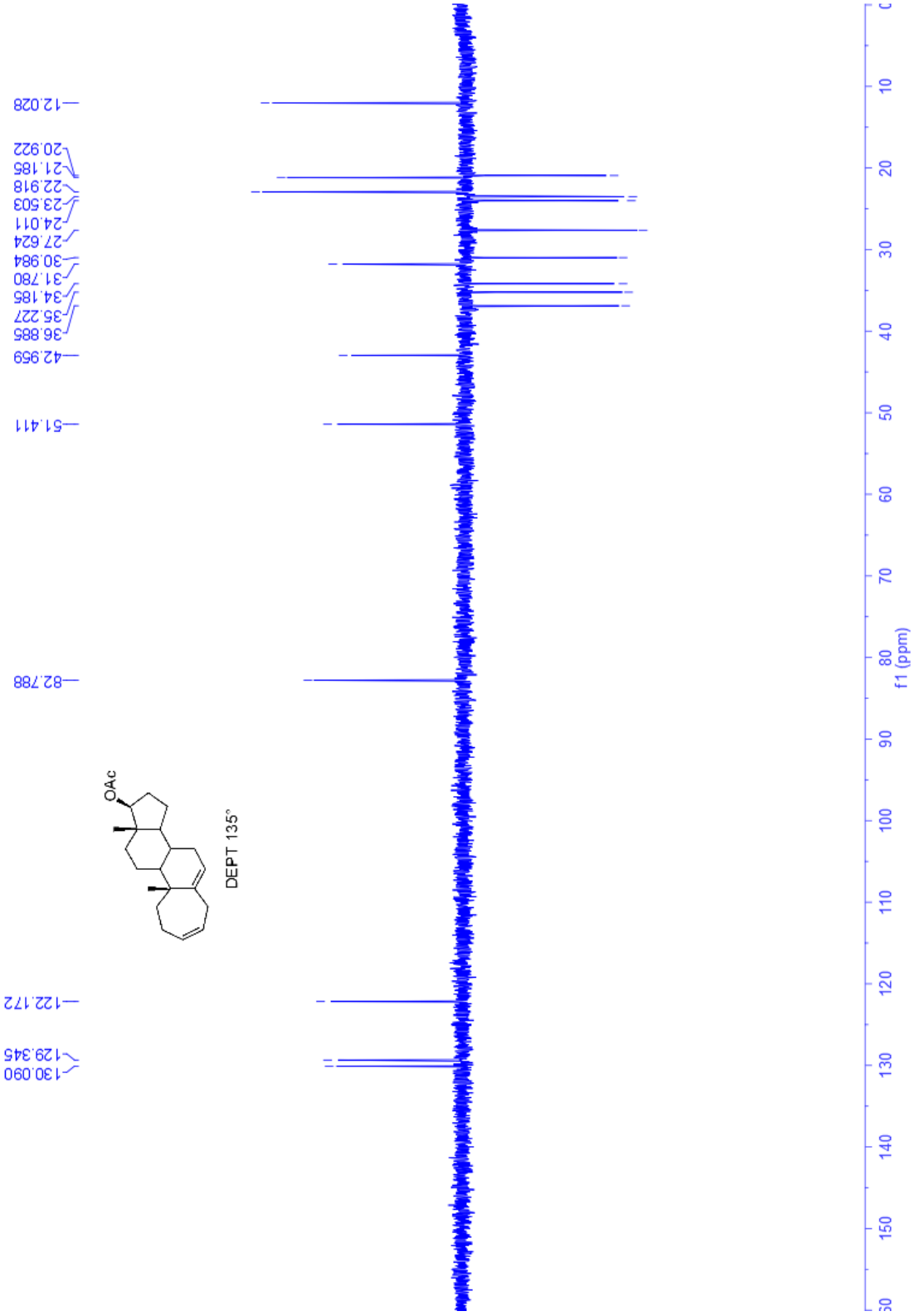

143.682
134.643
129.587
129.030
128.685
128.175
128.114
127.732
126.490
124.874
77.318
77.000
76.682
72.416
37.149
29.524
HO
13C NMR 100MHz CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


29.497
37.135
72.416
HO
DEPT 135°
124.843
126.469
127.706
128.143
128.663
128.993
129.565
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
f1 (ppm)

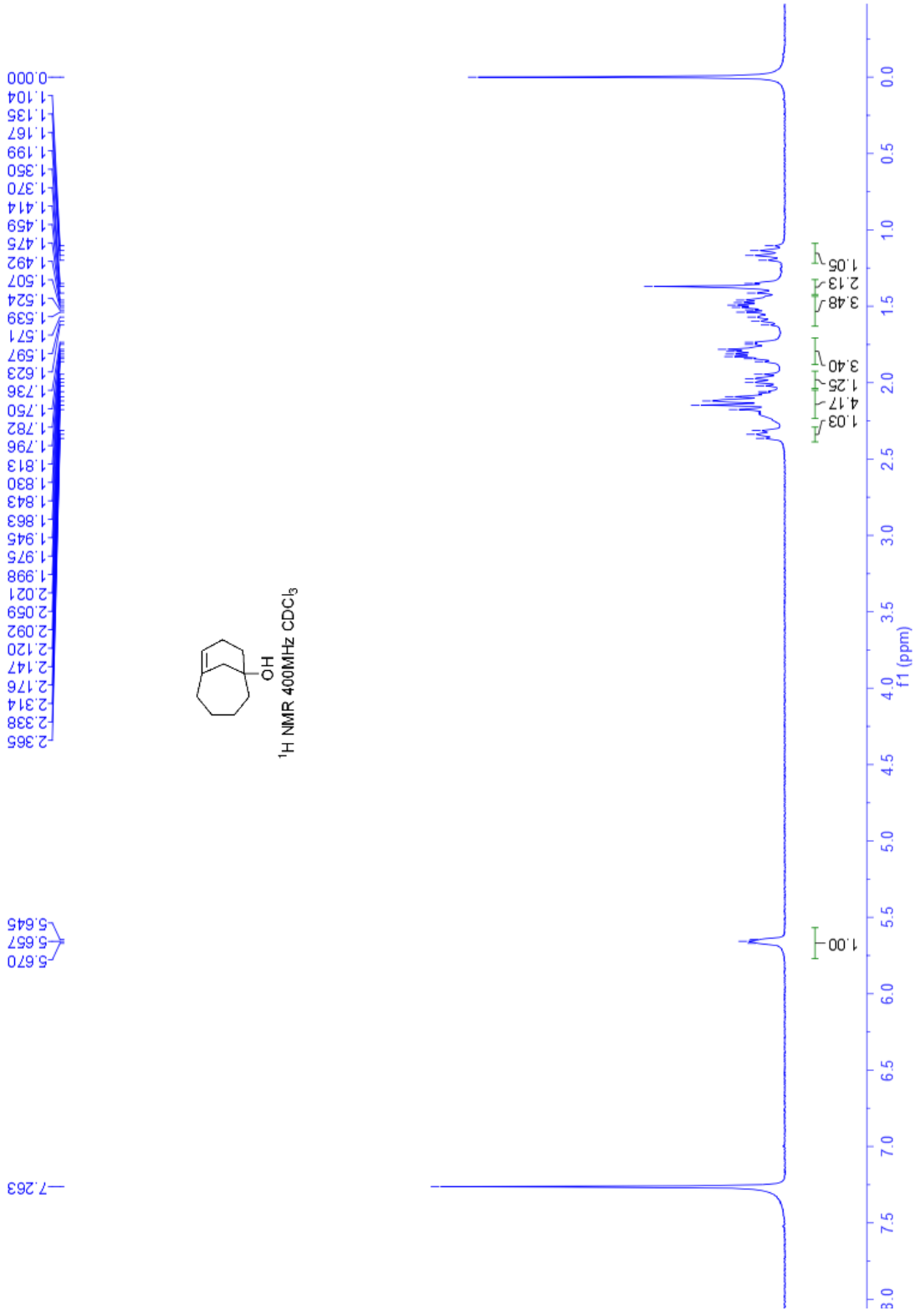
Compound **4p.**

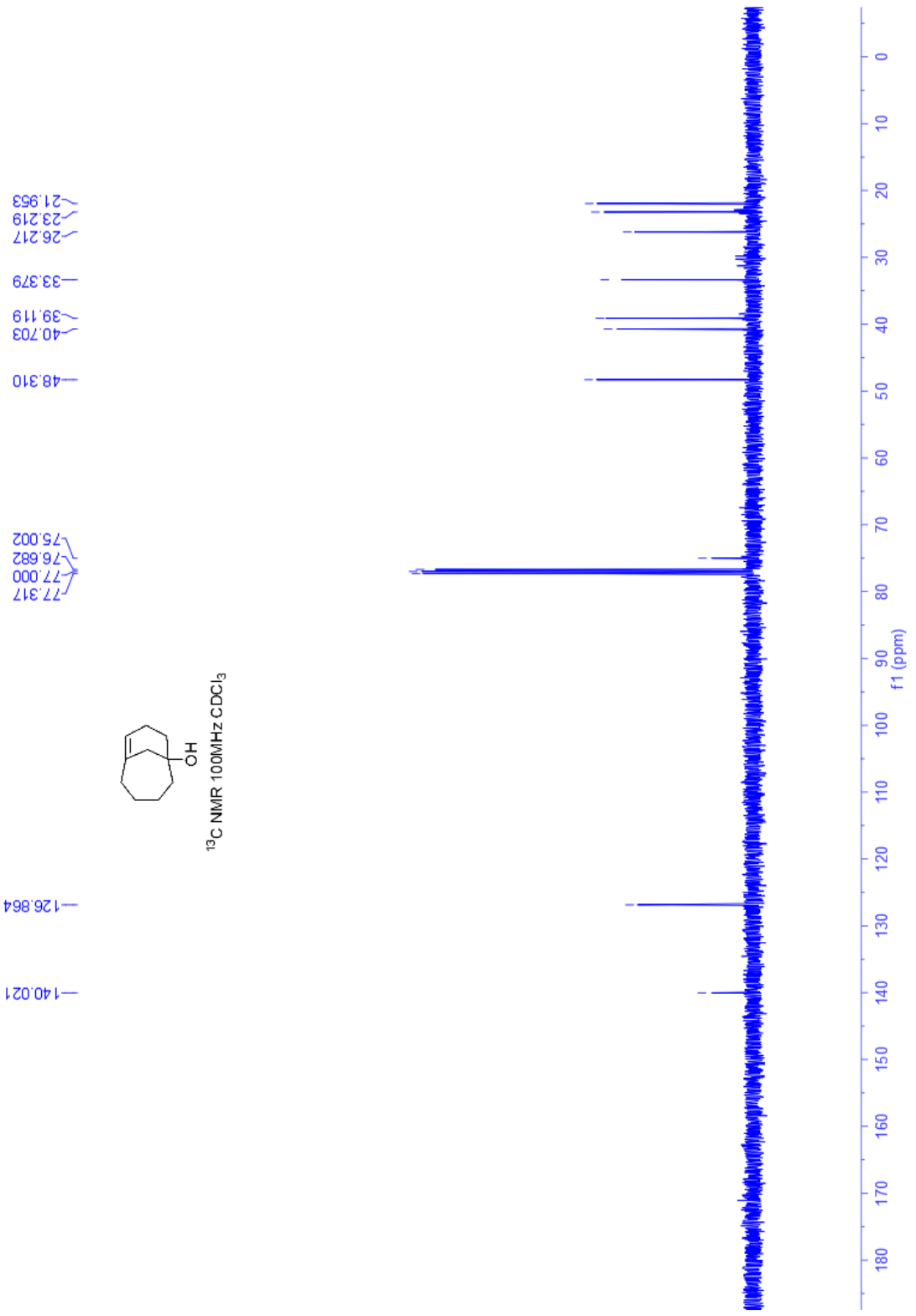


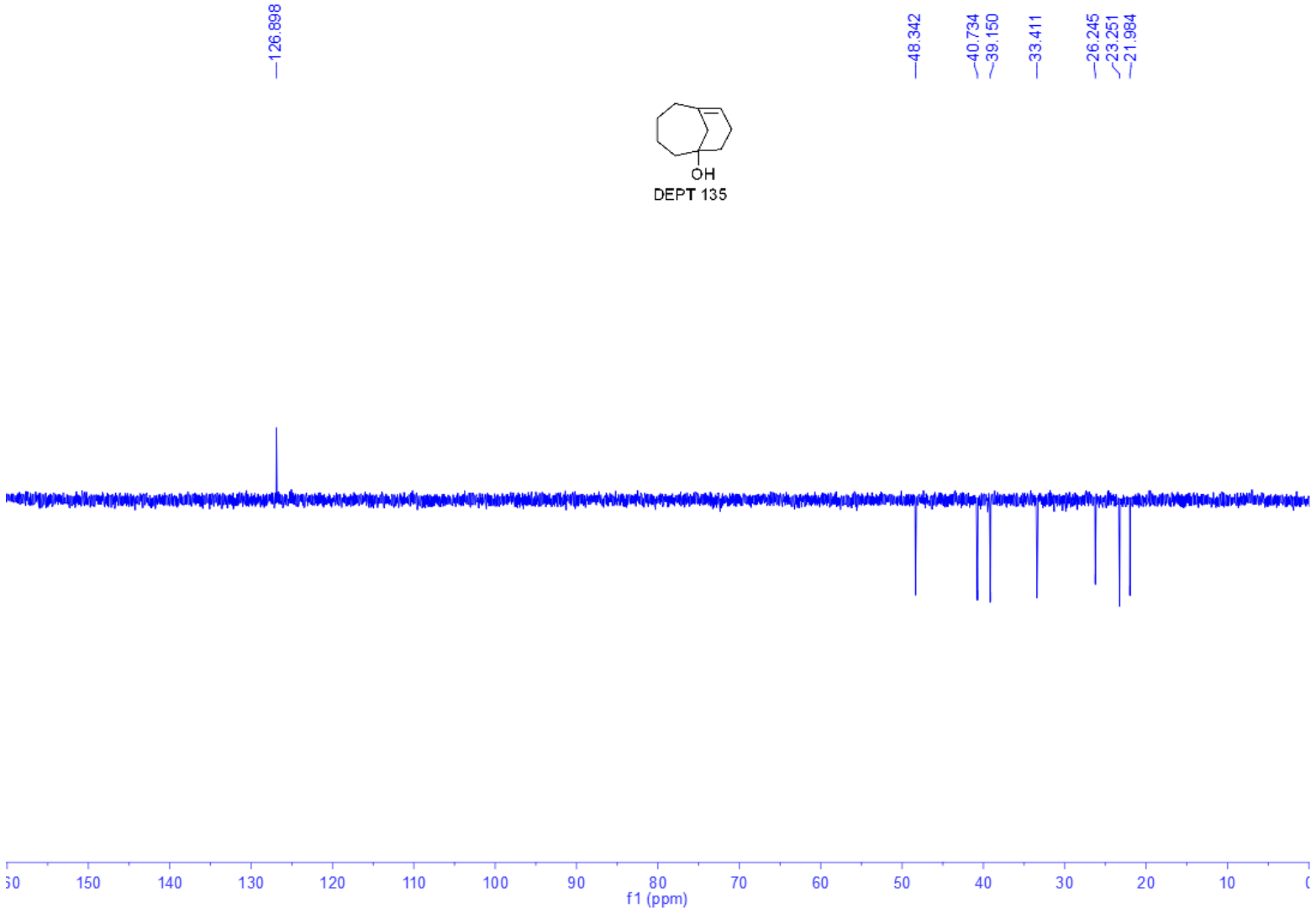

OAc
^{13}C NMR 100MHz $CDCl_3$
12.028
20.921
22.918
23.502
24.010
27.622
30.983
31.778
34.183
35.226
36.884
40.469
51.410
76.683
77.000
77.318
82.788
122.172
129.344
130.089
144.938
171.219
f1 (ppm)


12.028
20.922
21.185
22.918
23.503
24.011
27.624
30.984
31.780
34.185
35.227
36.885
42.959
51.411
82.788
122.172
129.345
130.090
OAc
DEPT 135°
f1 (ppm)

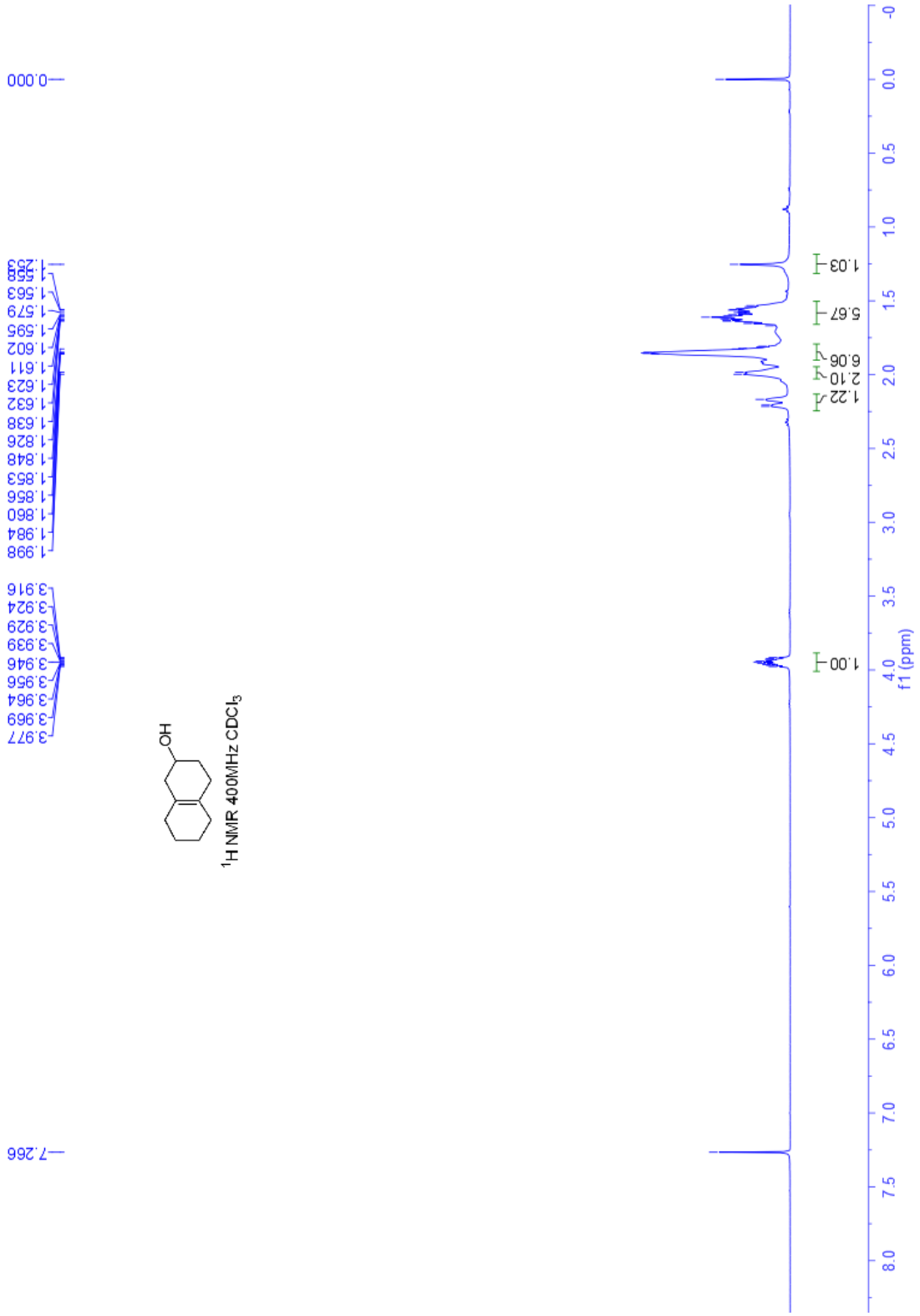
Compound 4q-2.

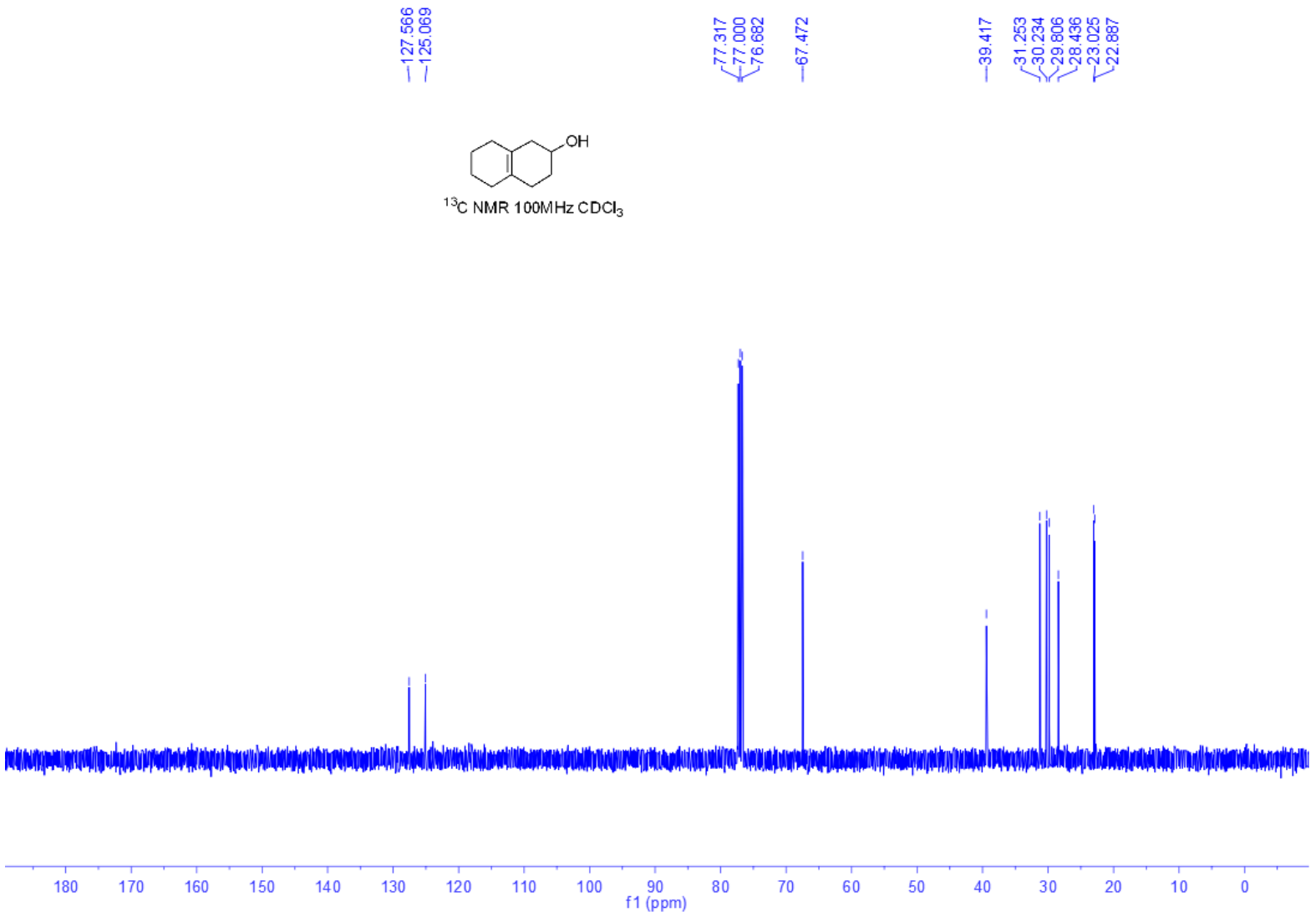


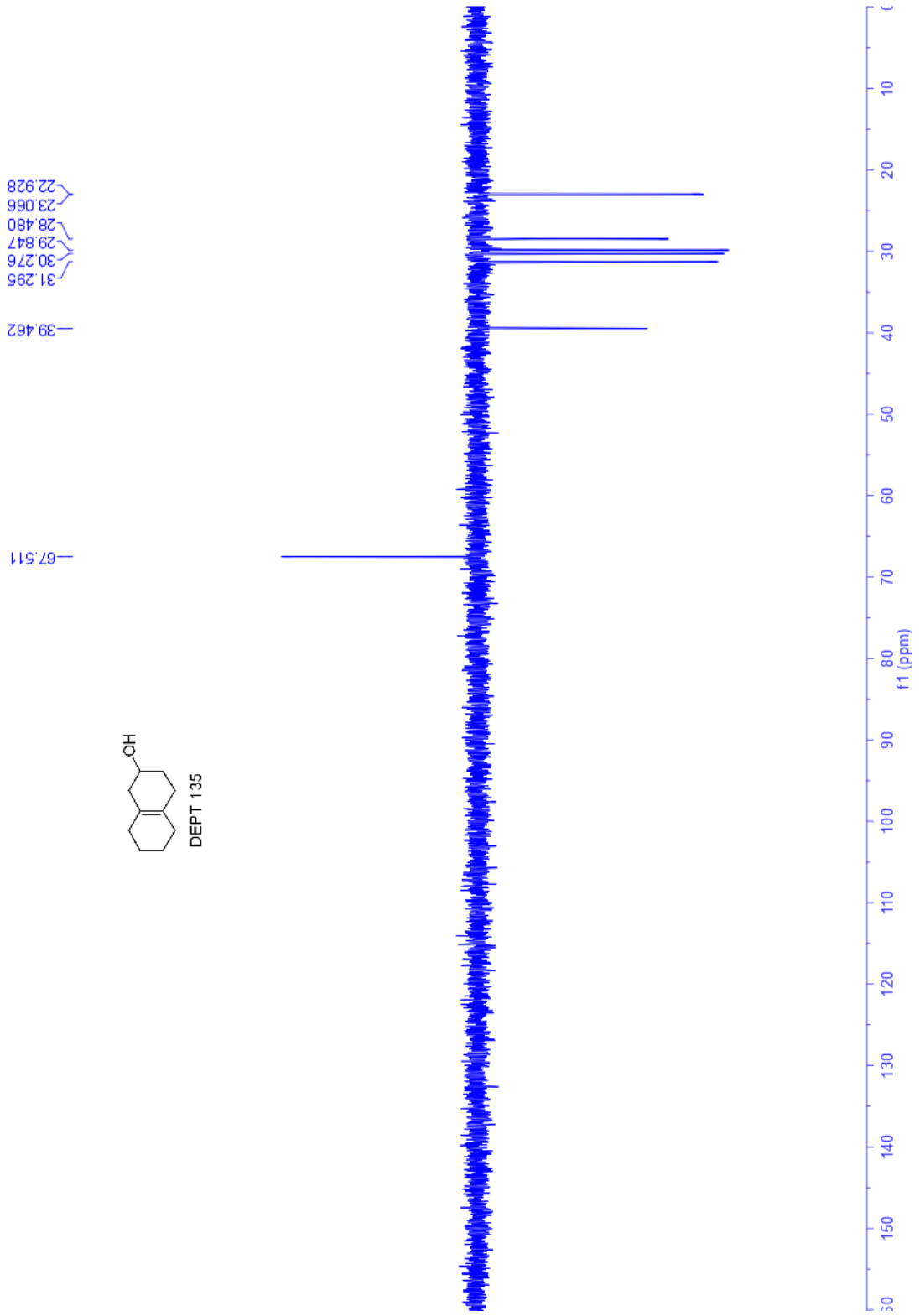

21.953
23.219
26.217
33.379
39.119
40.703
48.310
75.002
76.682
77.000
77.317
126.864
140.021
OH
^{13}C NMR 100MHz $CDCl_3$
f1 (ppm)


126.898
48.342
40.734
39.150
33.411
26.245
23.251
21.984
OH
DEPT 135
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

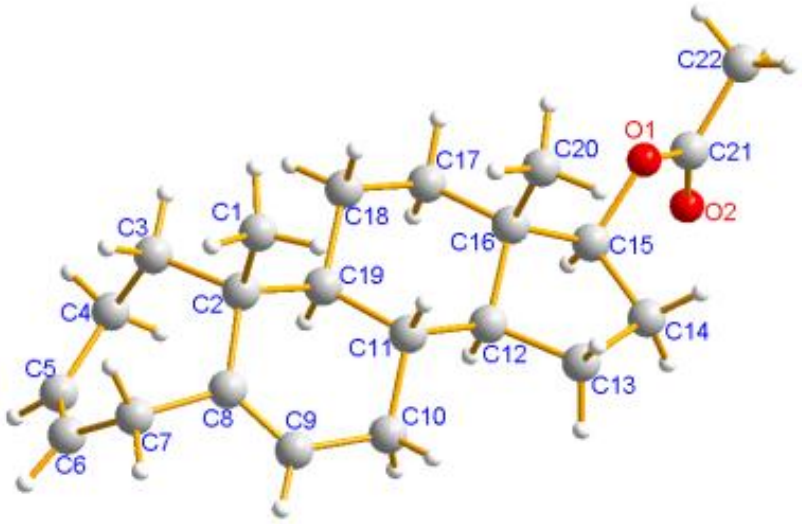
Compound 4q-1.




127.566
125.069
77.317
77.000
76.682
67.472
39.417
31.253
30.234
29.806
28.436
23.025
22.887
OH
^{13}C NMR 100MHz CDCl$_3$
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


22.928
23.066
28.480
29.847
30.276
31.295
39.462
67.511
OH
DEPT 135
f1 (ppm)

4. Crystalline state of compound **4p.** (CCDC No: 1008768)



Empirical formula	C22 H32 O2
Formula weight	328.48
Temperature	113(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 7.9008(16) A alpha = 90 deg.
	b = 9.2433(18) A beta = 92.35(3) deg.
	c = 12.558(3) A gamma = 90 deg.
Volume	916.3(3) A^3
Z, Calculated density	2, 1.191 Mg/m^3
Absorption coefficient	0.074 mm^-1
F(000)	360
Crystal size	0.20 x 0.18 x 0.12 mm
Theta range for data collection	1.62 to 27.93 deg.
Limiting indices	-10<=h<=10, -12<=k<=12, -16<=l<=16
Reflections collected / unique	11463 / 4258 [R(int) = 0.0422]
Completeness to theta = 27.93	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9912 and 0.9854

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4258 / 1 / 221
Goodness-of-fit on F^2	0.941
Final R indices [I>2sigma(I)]	R1 = 0.0352, wR2 = 0.0798
R indices (all data)	R1 = 0.0546, wR2 = 0.0885
Absolute structure parameter	0.6(10)
Extinction coefficient	0.105(6)
Largest diff. peak and hole	0.208 and -0.191 e.A^-3