Supporting Information

Concise Total Synthesis of (±)-Aspidospermidine

Haichen Ma,[†] Xingang Xie,[†] Peng Jing,[†] Weiwei Zhang,[†] Xuegong She*,[†,‡]

[†]State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou, Gansu, 730000, P. R. China

[‡]Collaborative lnnovation Center of Chemical Science and Engineering Tianjin (P.R. China)

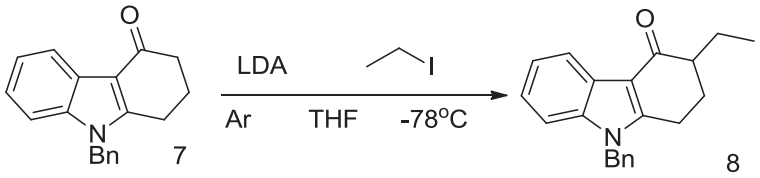
Email: shexg@lzu.edu.cn

Table of Contents

Materials and Methods

Solvents were purified and dried by standard methods prior to use. All commercially available reagents were used without further purification unless otherwise noted. Oxygen- and moisture-sensitive reactions were carried out under argon atmosphere.

Column chromatography was generally performed on silica gel (200-300 mesh) and reactions were monitored by thin layer chromatography (TLC) using silica gel GF254 plates with UV light to visualize the course of reaction. Melting points were determined with a digital Koffer apparatus and were uncorrected. ^{1}H and ^{13}C NMR data were recorded on a 400 MHz instrument using $CDCl_3$ as solvent at room temperature. The chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. High-resolution mass spectra (HRMS) were obtained on a FT-ICR spectrometer.



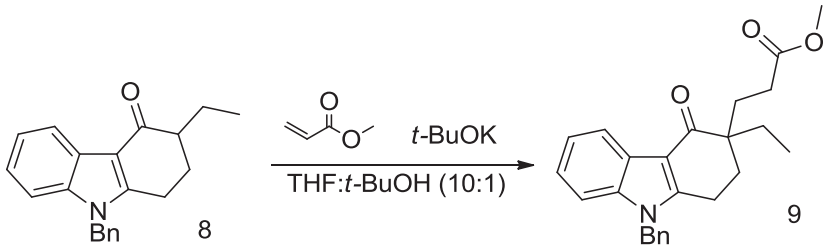
Compound 8. To a stirred solution of **7** (5.50 g, 20 mmol) in THF was added LDA (10 mL, 20 mmol) dropwise at -78 ℃. After 45 min, the ethyl iodide (3.74 g, 24 mmol) was added to the mixture and the reaction mixture was stirred for 30 min at the same temperature. Then it was warmed to room temperature and stirred overnight. The reaction was quenched by slow additional of saturated NH_4Cl aqueous solution and extracted with CH_2Cl_2 (60 mL×3). The combined organic extracts were washed with brine (50 mL), dried over anhydrous $NaSO_4$ and concentrated on vacuum. The obtained residue was purified by column chromatography (silica gel, hexane/EtOAc 5:1) to give pure product as a white solid (5.82 g 96% yield.) mp 133 °C.

IR (KBr) ν_{max} 3058, 3033, 2962, 2932, 2871, 2245, 1643, 1611, 1539, 1461, 1397, 1152, 1077, 910, 732, 700 cm^{-1}.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.00 (t, J = 7.6 Hz, 3H), 1.54 (m, 1H), 1.97 (m, 2H), 2.32 (m, 2H), 2.81 (m, 2H), 5.22 (s, 2H), 6.99 (t, J = 6.0 Hz, 2H),7.17 (t, J = 6.0 Hz, 2H), 7.24 (m, 4H), 8.31 (d, J = 8.8 Hz, 1H);

^{13}C NMR ($CDCl_3$, 100 MHz): δ 11.7, 21.0, 22.3, 27.6, 46.7, 47.6, 109.5, 112.5, 121.5, 122.4, 122.9, 125.1, 126.0, 127.7, 128.9, 136.0, 137.2, 150.9, 196.0;

HRMS (ESIMS) Calcd for $C_{21}H_{21}NO$ $[M+Na]^+$ 326.1515, found 326.1524



Compound 9. To a solution of ketone **8** (1.56 g, 5 mmol), which protected by argon, in anhydrous THF (100 mL)/t-BuOH (10 mL) at 0℃ was added t-BuOK (1.12 g, 10 mmol,) and the resulting mixture was stirred at 0℃ for 10min. methyl acrylate (3.6 mL, 40 mmol) was added to the reaction and the resulting mixture was stirred at -20℃ for 30 min and at room temperature for 12 h. The reaction mixture was quenched by addition of saturated NH_4Cl at 0℃. The resultant residue was taken

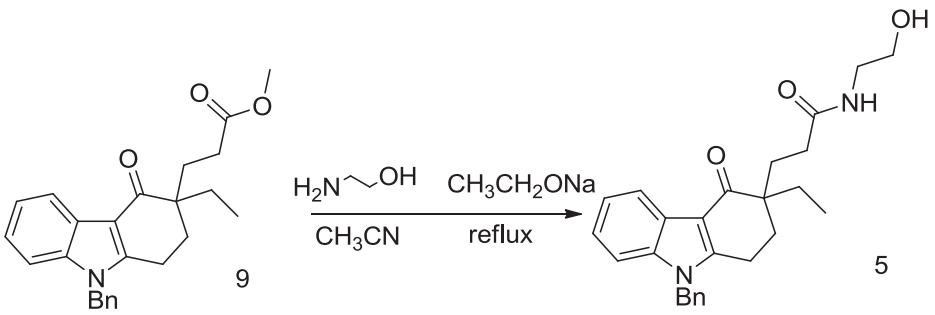
up in water (50 mL) and the aqueous mixture was extracted with CH_2Cl_2 (40 mL×3). The combined organic extracts were washed with brine (40 mL) and dried over $NaSO_4$ and the solvent was removed under vacuum. The residue was purified by column chromatography (silica gel, hexane/EtOAc 5:1) to give pure product **9 (**1.52 g, 78% yield) as a yellow solid.

IR (KBr) ν_{max} 3060, 3031, 2947, 2881, 2250, 1735, 1641, 1542, 1460, 1437, 1360, 1300, 1199, 1176, 1070, 912, 841, 732, 700, 648 cm^{-1}.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.91 (t, J = 7.6 Hz, 3H), 1.68 (m, 2H), 1.87 (m, 1H), 2.08 (m, 3H), 2.37 (m, 2H), 2.91 (m, 2H), 3.63 (s, 3H), 5.30 (s, 2H), 7.03 (d, J = 7.6Hz, 2H), 7.26 (m, 6H), 8.30 (d, J = 7.6 Hz, 1H);

^{13}C NMR ($CDCl_3$, 100 MHz): δ 8.4, 19.1, 27.2, 29.2, 31.4, 46.9, 47.2, 51.5, 109.6, 112.0, 121.7, 122.6, 123.1, 125.4, 126.0, 127.9, 129.0, 136.0, 137.4, 149.8, 174.1, 197.1;

HRMS (ESIMS) Calcd for $C_{25}H_{27}NO_3$ $[M+Na]^+$ 412.1883, found 412.1885.



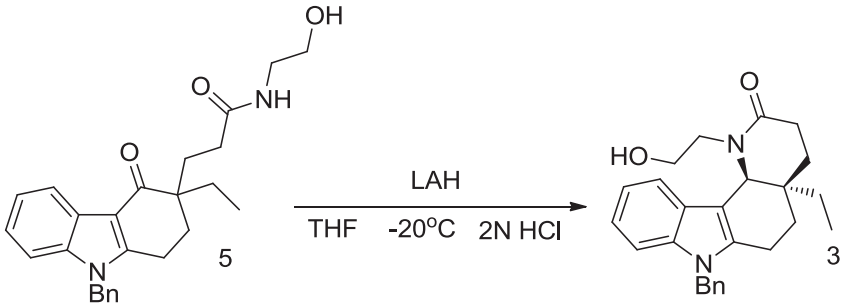
Compound 5. To a solution of **9 (**1.52 g 3.81mmol**)** was added CH_3CN. Monoethanol Amine (2.31g 38mmol**)** was added to the reaction mixture and the resulting solution was stirred and reflux at 78℃ for 10h. A trace of Sodium ethoxide was added in the system. The reaction mixture was extracted with CH_2Cl_2 (60 mL×3). The combined organic extracts were washed with brine (50 mL) and dried over $NaSO_4$ and the solvent was removed under vacuum. The mixture residue was purified by column chromatography (silica gel, hexane/EtOAc 2:1) to give the pure product **5**(1.38 g 90% yield) as yellow mucous. mp 155 ℃.

IR (KBr) ν_{max} 3317, 3060, 2932, 2878, 1725, 1637, 1540, 1459, 1439, 1359, 1196, 1137, 1069, 935, 838, 735, 700, 659, 617 cm^{-1}.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.87 (t, J = 7.6 Hz, 3H), 1.43(s, 1H), 1.62 (m, 2H), 1.87 (m, 1H), 2.07 (m, 3H), 2.22 (m, 2H), 2.87 (m, 2H), 3.37 (t, J = 5.0 Hz, 2H), 3.68 (t, J = 4.8 Hz, 2H), 5.24 (s, 2H), 6.84 (s, 1H), 7.01 (d, J = 6.4 Hz, 2H), 7.25 (m, 6H), 8.23 (d, J = 7.2 Hz, 1H);

^{13}C NMR ($CDCl_3$, 100 MHz): δ 8.4, 19.0, 27.6, 30.0, 31.0, 31.6, 42.4, 46.8, 47.5, 61.9, 109.7, 111.8, 121.4, 122.6, 123.1, 125.2, 126.0, 127.8, 129.0, 135.7, 137.4, 150.6, 174.5, 198.2;

HRMS (ESIMS) Calcd for $C_{26}H_{30}N_2O_3$ $[M+Na]^+$ 441.2149, found 4441.2153.



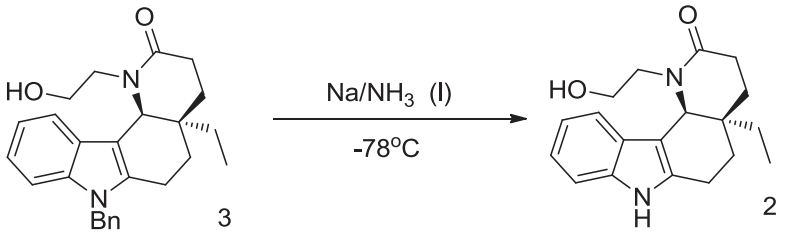
Compound 3. To a stirred solution of **5**(0.44 g 1.04 mmol**)** in THF was added LAH (0.12 g 3.13 mmol) drop wise at -20℃. Then assembling a drying tube at the flask and stirring for 30 min. The reaction was quenched by water and then adjusted by 2N HCl to attain the target of PH (2~3). The aqueous layer was separated and extracted with CH_2Cl_2 (30 mL×2). The combined organic extracts were washed with brine (30 mL×2) and dried over $NaSO_4$. The residue was purified by column chromatography (silica gel, EtOAc) to give pure product **3**. (0.40 mg 94% yield) mp 200°C;

IR (KBr) ν_{max} 3327, 3055, 2938, 2878, 1705, 1631, 1612, 1463, 1427, 1357, 1302, 1266, 1190, 1051, 1029, 736, 698 cm^{-1};

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.85 (t, J = 7.6 Hz, 3H), 1.28(m, 2H), 1.65 (m, 2H), 1.92 (m, 1H), 2.07 (m, 1H), 2.50 (m, 2H), 2.60 (m,1H), 2.73 (m, 1H), 3.31 (m, 1H), 3.55 (m, 1H), 3.63 (m, 1H), 3.74 (m, 1H), 3.96 (s, 1H), 5.28 (s, 2H), 6.92 (d, J = 7.6 Hz, 2H), 7.15 (m, 2H), 7.24 (m, 4H), 7.52 (m, 2H);

^{13}C NMR ($CDCl_3$, 100 MHz): δ 8.0, 19.3, 26.2, 28.1, 29.5, 29.6, 36.9, 46.4, 47.9, 58.9, 63.7, 106.7, 109.6, 117.6, 120.1, 121.6, 125.7, 127.5, 127.9, 128.8, 137.1, 137.3, 174.4;

HRMS (ESIMS) Calcd for $C_{26}H_{30}N_2O_2$ $[M+H]^+$ 403.2380, found 403.2384.



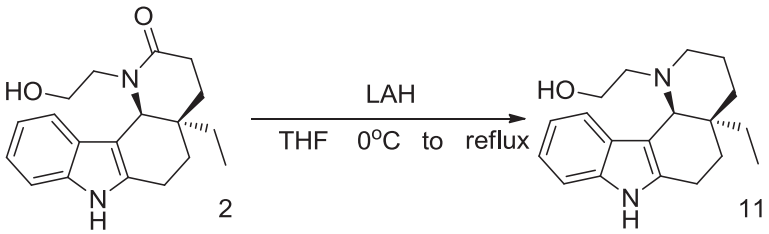
Compound 2. Freshed cut Na (229 mg, 9.96 mmol) was added to liquid ammonia (20 mL) in a 100 ml two-necked flask cooled to –78℃. After 5 min, a solution of *t*-BuOH (0.5 mL) in THF (5 mL) was added to the blue ammonia solution, followed by a solution of amide **3** (200 mg, 0.50 mmol) in THF (5 mL). The reaction mixture was stirred at –78℃ for 30 min at which the blue color had disappeared. The reaction mixture then was quenched with saturated NH_4Cl and ammonia was allowed to evaporate by replacing the cooling bath with a water bath. The mixture solution was

extracted by CH_2Cl_2 (20 mL×3). The residue was dissolved in CH_2Cl_2 (4 mL) flash column chromatography (EtOAc) of the crude residue yielded 155mg (85.2%) of amine **2** as a colorless solid. **IR (KBr)** ν_{max} 3262, 3057, 2940, 2876, 1610, 1462, 1419, 1330, 1304, 1263, 1121, 1048, 940, 739, 703, 656, 619 cm^{-1};

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.87 (t, J = 7.2 Hz, 3H), 1.29(m, 2H), 1.66 (m, 2H), 1.93 (m, 1H), 2.06 (m, 1H), 2.51 (m, 2H), 2.73 (m, 2H), 3.27 (m, 1H), 3.53 (m, 1H), 3.65 (m, 1H), 3.71 (m, 1H), 4.12 (m, 1H), 4.50 (s, 1H), 7.14 (dd, J = 7.2 Hz, J = 7.6 Hz, 2H), 7.30 (d, J = 7.6 Hz, 1H), 7.46 (d, J = 7.2 Hz, 1H), 8.69 (s, 1H),

^{13}C NMR ($CDCl_3$, 100 MHz): δ 8.0, 20.0, 26.3, 28.0, 29.5, 29.7, 37.0, 48.1, 59.1, 63.6, 106.7, 101.0, 117.4, 120.0, 121.6, 128.0, 136.1, 136.2, 174.6;

HRMS (ESIMS) Calcd for $C_{19}H_{24}N_2O_2$ $[M + H]^+$ 313.1911, found 313.1916.



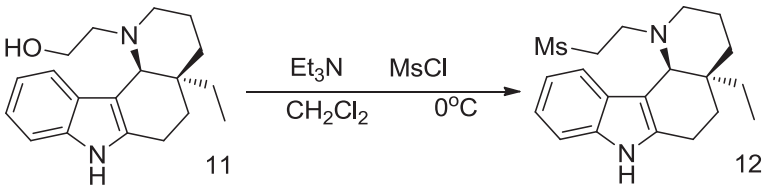
Compound 11. To a solution of **2**(0.56 g 1.19 mmol) in THF was added LAH (0.14 g 3.56 mmol) dropwise. After 10 min the mixture was warmed to 80℃, then to stir further and reflux for about 4h. The reaction was quenched by saturated NH_4Cl. The mixture was diluted with CH_2Cl_2 (20 mL×3) and washed with brine (30 mL×2). The residue was dissolved in CH_2Cl_2 (4 mL) flash column chromatography CH_2Cl_2: MeOH (30:1) of the product yielded 234 mg (66%) of amine**11** as a colorless solid. mp 74℃;

IR (KBr) ν_{max}3397, 3217, 3185, 3109, 3058, 2937, 2877, 2794, 1620, 1585, 1461, 1377, 1331, 1308, 1265, 1233, 1169, 1141, 1120, 1041, 739 cm^{-1};

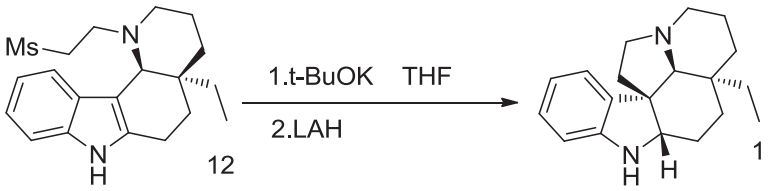
^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.71 (t, J = 7.6 Hz, 3H), 0.90 (m, 1H), 1.14 (m, 1H), 1.34 (m, 2H), 1.58 (m, 1H), 1.74 (m, 1H), 1.83 (m, 1H), 2.22 (m, 2H), 2.60 (m, 3H), 3.25 (m, 5H), 3.49 (m, 1H), 7.05 (m, 2H), 7.13 (m, 1H), 7.42 (m, 1H), 8.32 (s, 1H);

^{13}C NMR ($CDCl_3$, 100 MHz): δ 7.7, 20.1, 21.9, 24.2, 29.4, 34.6, 37.0, 52.3, 54.0, 57.8, 62.9, 110.0, 110.5, 117.5, 119.1, 120.6, 129.7, 135.5, 136.0;

HRMS (ESIMS) Calcd for $C_{19}H_{26}N_2O$ $[M + H]^+$ 299.2118, found 299.2117



Compound 12. To a solution of the alcohol **11** (100 mg, 0.34 mmol) in anhydrous CH_2Cl_2 (10 mL) at 0℃ were sequentially added triethylamine (9 μl, 0.67 mmol), Methanesulfonyl chloride (31 μl, 0.4 mmol**)**. The mixture was stirred at 0℃ for 30 min and water (3 mL) was added to quench the reaction. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (10 mL ×3). The combined organic layer was washed with water, brine and dried over Na_2SO_4. The solution was concentrated to dryness *in vacuo* to give a yellow powder. (121 mg 95% yield).

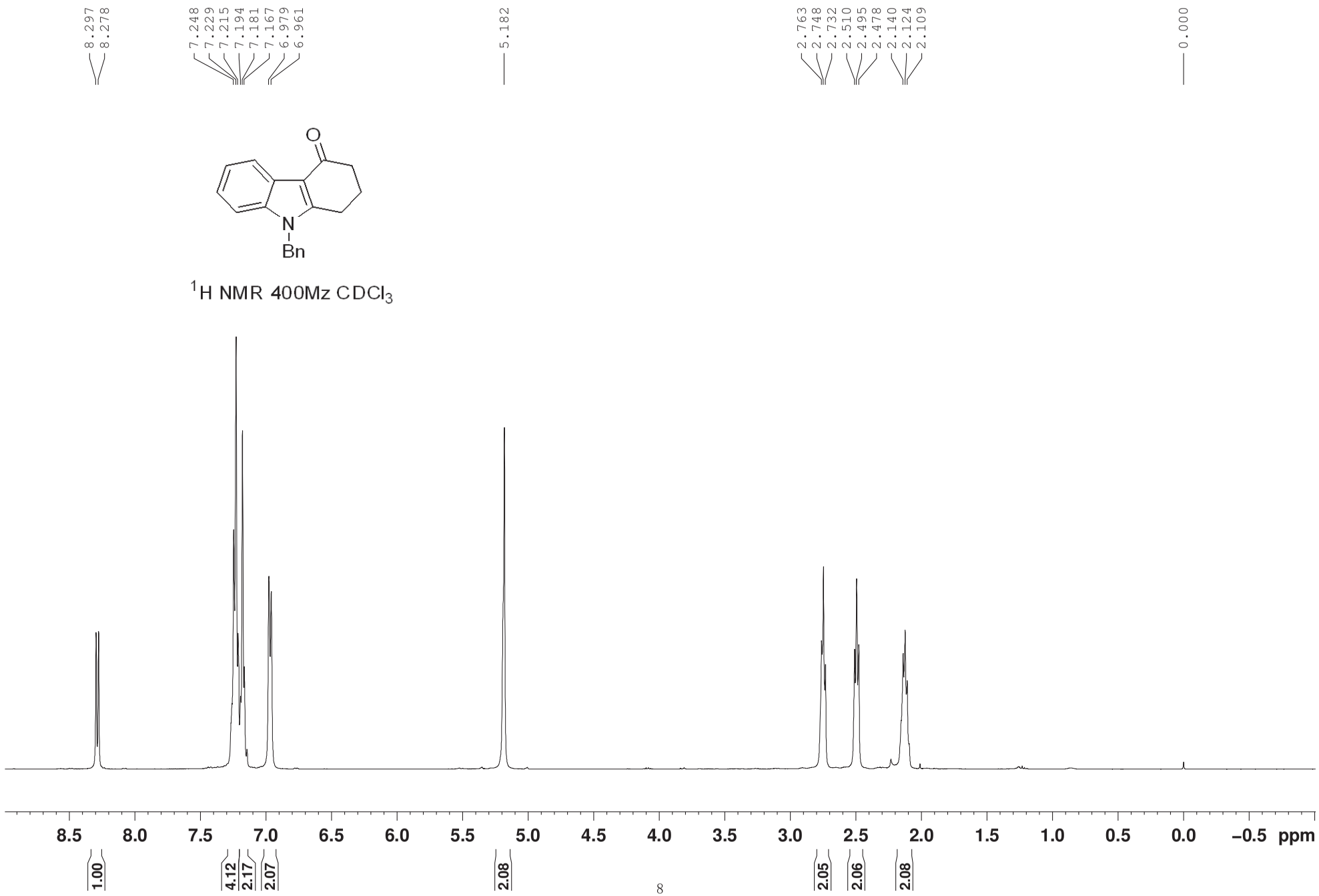


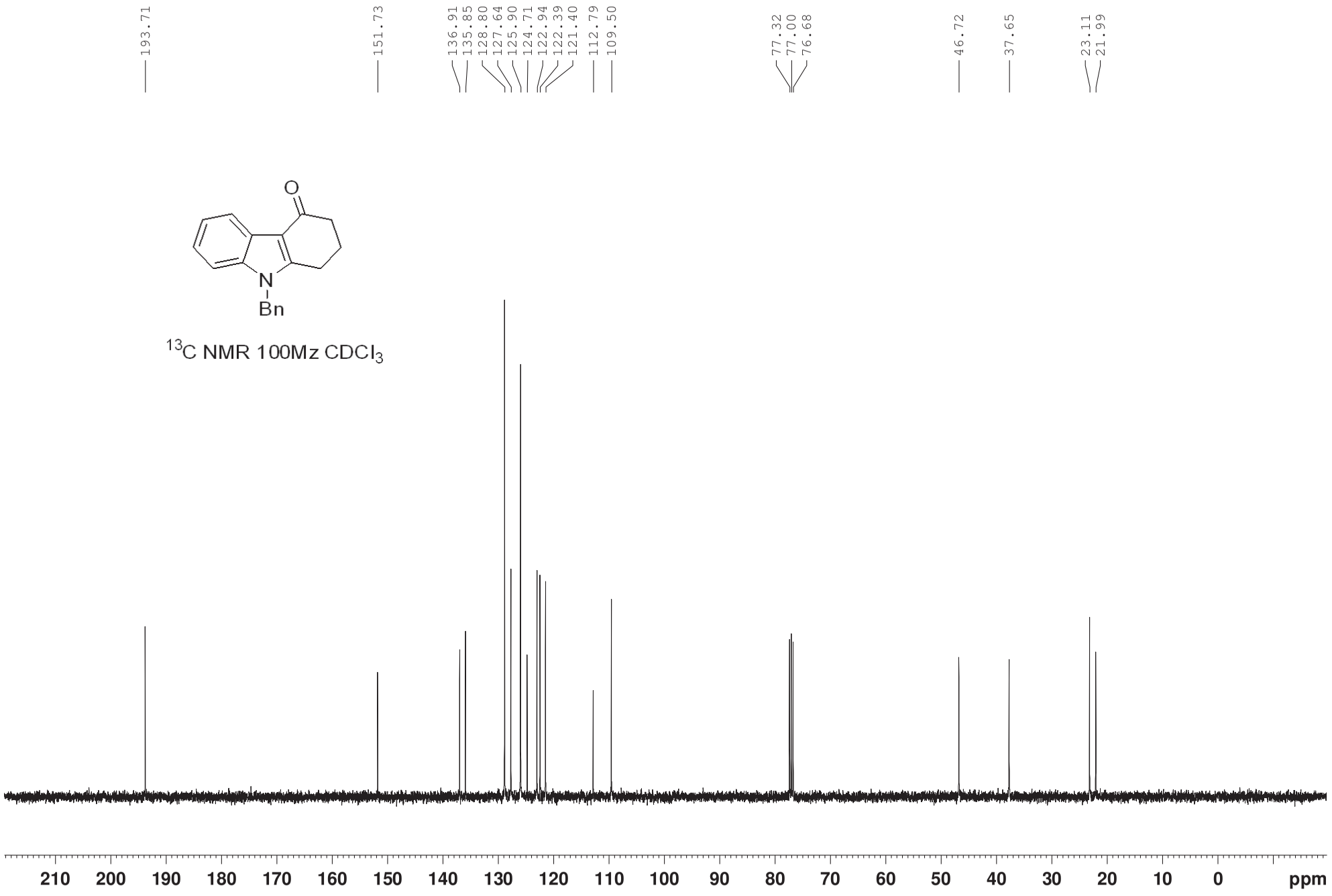
(±)-Aspidospermidine 1. To a solution of **12**(158 mg 0.45 mmol) in CH_2Cl_2 (6 mL) was added dropwise t-BuOK causing the formation of white precipitate. The reaction stirred for about 16h at room temperature. Then LAH (28.5 mg 0.75 mmol) was added in the mixture solution at 0℃ and stirred for 30 min and saturated NH_4Cl was added to quench the reaction the layers were separated and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed with water, brine and dried over Na_2SO_4. The solution was concentrated to dryness *in vacuo* to give the TM. (87 mg 62% yield).

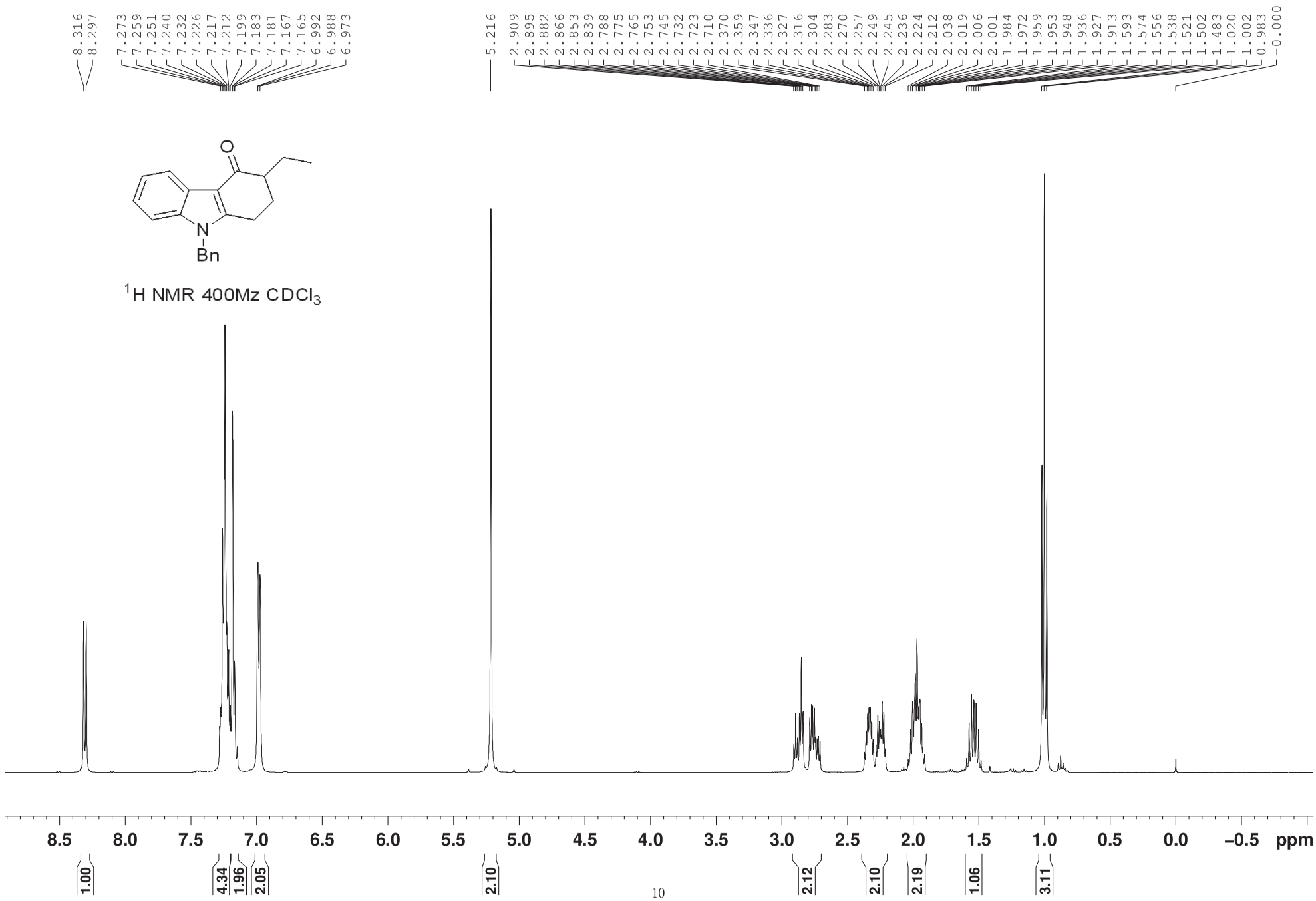
IR (KBr) ν_{max} 3635, 3362, 3048, 3027, 2934, 2882, 2861, 2780, 2722, 2678， 1606, 1481, 1463, 1376, 1331, 1318, 1259, 1179, 1134, 1024, 907, 866, 736, 642 cm^{-1};

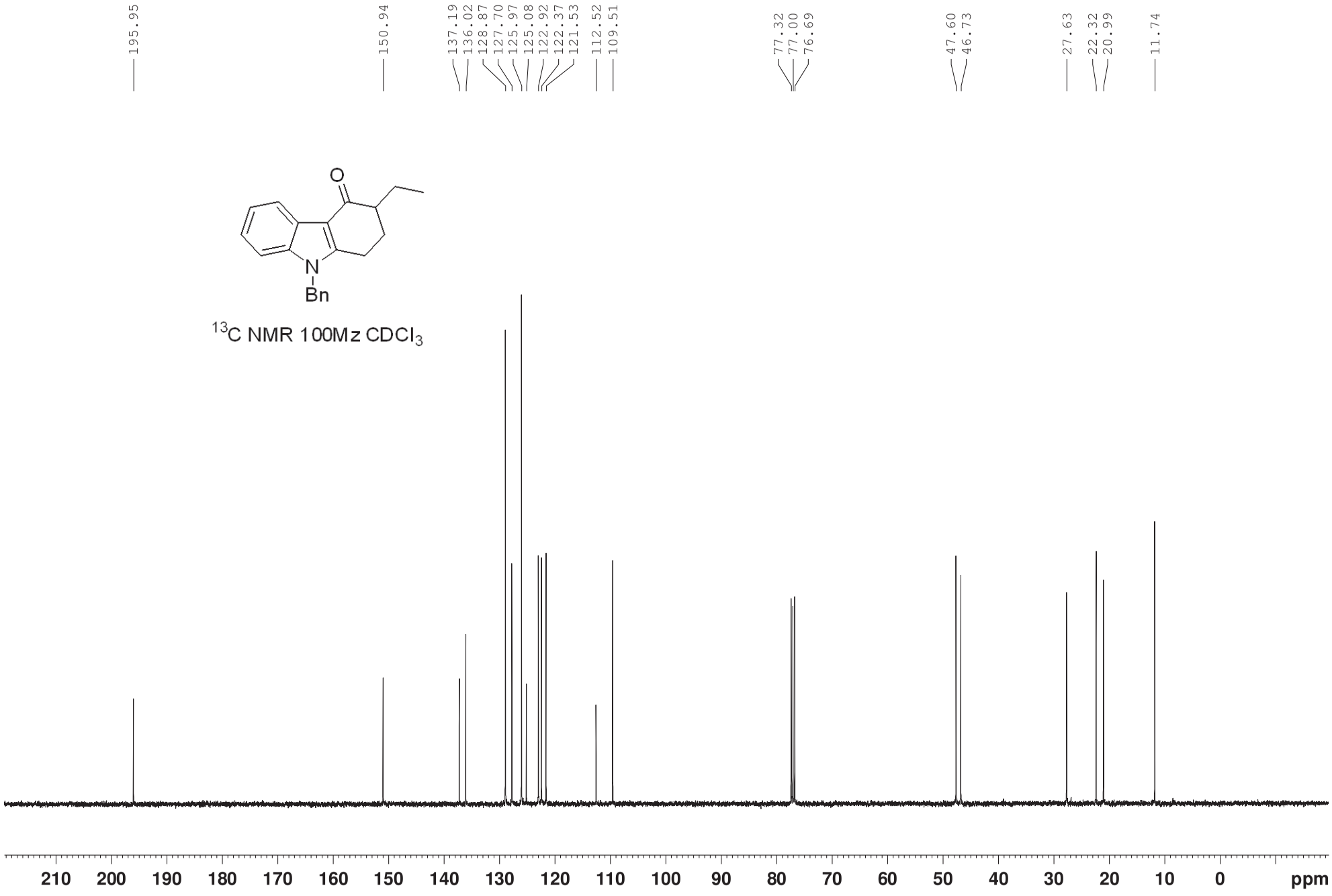
^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.63 (t, J = 7.6 Hz, 3H), 0.87(m, 1H), 1.02 (m, 2H), 1.41 (m, 4H), 1.57 (m, 2H), 1.74 (m, 1H), 1.94 (m, 2H), 2.26 (m, 3H), 3.10 (m, 2H), 3.50 (m, 1H), 3.49 (m, 1H), 6.63 (d, J = 7.6 Hz, 1H), 6.72 (t, J = 7.6 Hz, 1H), 7.01 (t, J = 7.6 Hz, 1H), 7.08 (d, J = 7.6 Hz, 1H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 6.8, 21.8, 23.0, 28.1, 29.9, 34.5, 35.6, 38.8, 53.0, 53.4, 53.9, 65.6, 71.2, 110.3, 118.9, 122.8, 127.0, 135.7, 149.4;

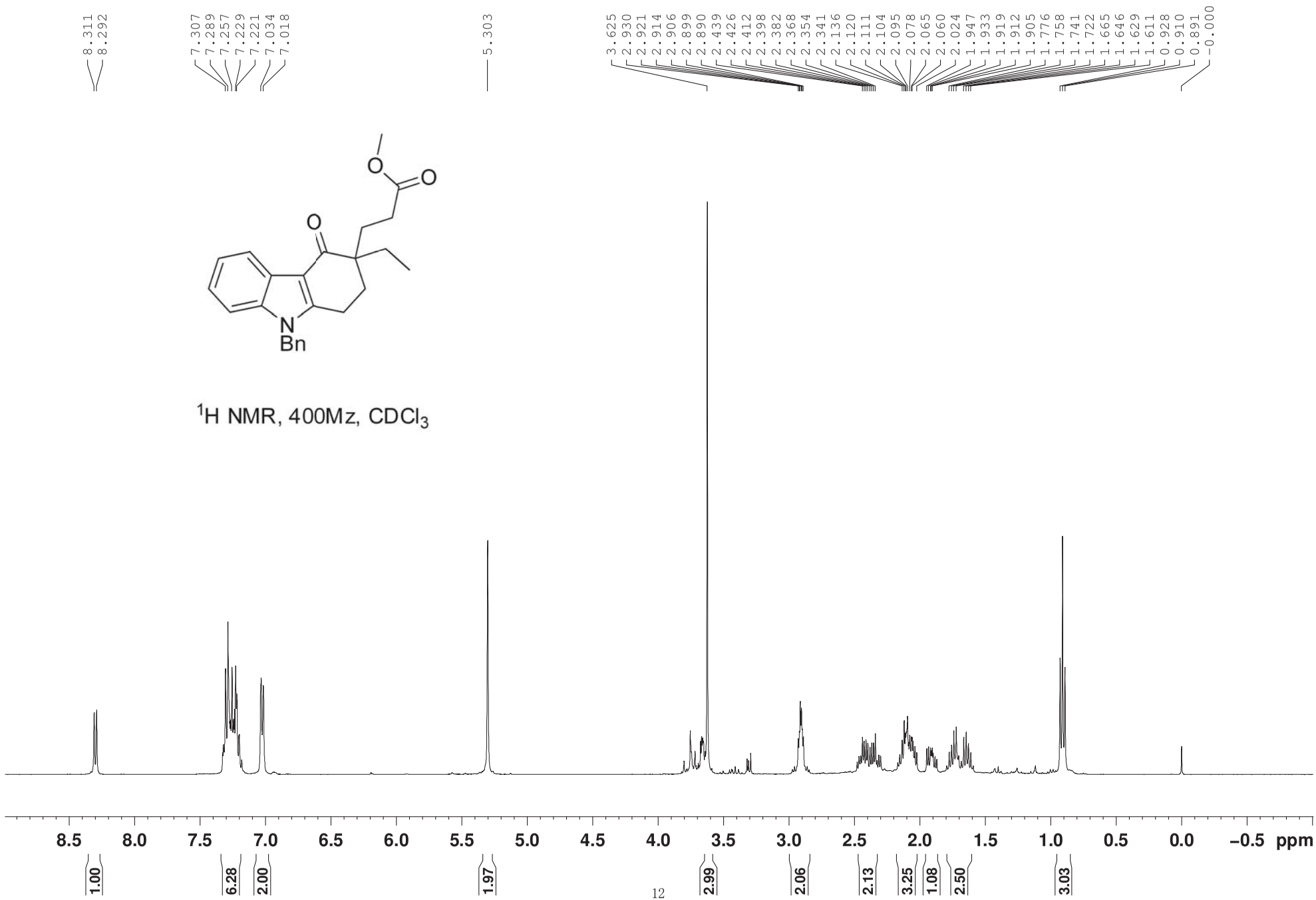
HRMS (ESIMS) Calcd for $C_{19}H_{26}N_2O$ $[M+H]^+$ 283.2169, found 283.2164.

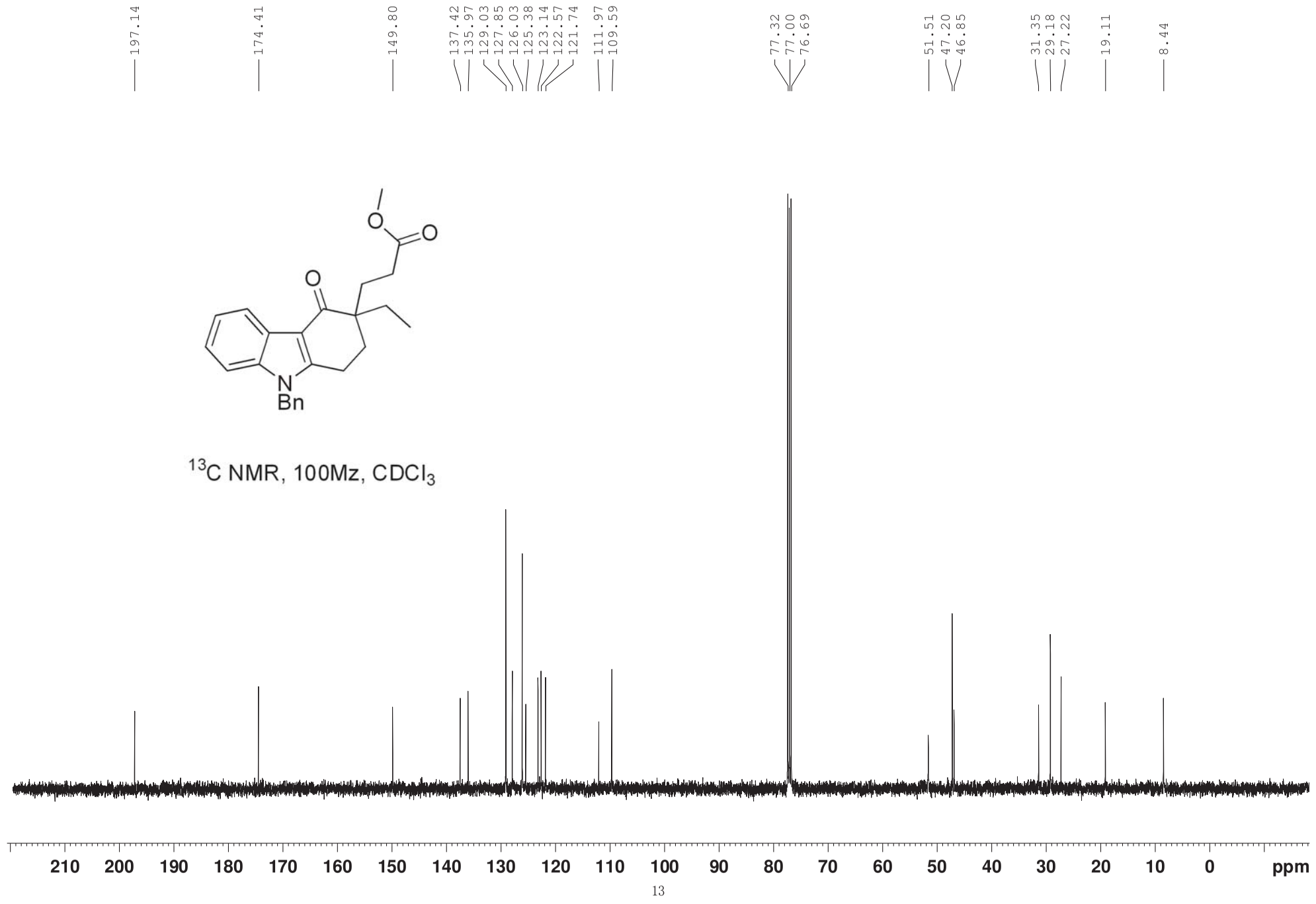

8.297
8.278
7.248
7.229
7.215
7.194
7.181
7.167
6.979
6.961
5.182
2.763
2.748
2.732
2.510
2.495
2.478
2.140
2.124
2.109
0.000
O
N
Bn
1H NMR 400Mz CDCl3
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5 ppm
1.00
4.12
2.17
2.07
2.08
2.05
2.06
2.08

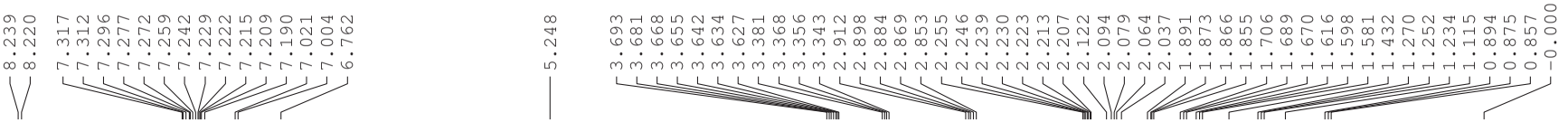

193.71
151.73
136.91
135.85
128.80
127.64
125.90
124.71
122.94
122.39
121.40
112.79
109.50
77.32
77.00
76.68
46.72
37.65
23.11
21.99
O
N
Bn
13C NMR 100Mz CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

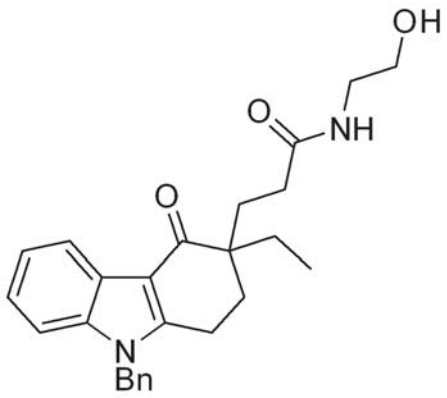

8.316
8.297
7.273
7.259
7.251
7.240
7.232
7.226
7.217
7.212
7.199
7.183
7.181
7.167
7.165
6.992
6.988
6.973
5.216
2.909
2.895
2.882
2.866
2.853
2.839
2.788
2.775
2.765
2.753
2.745
2.732
2.723
2.710
2.370
2.359
2.347
2.336
2.327
2.316
2.304
2.283
2.270
2.257
2.249
2.245
2.236
2.224
2.212
2.038
2.019
2.006
2.001
1.984
1.972
1.959
1.953
1.948
1.936
1.927
1.913
1.593
1.574
1.556
1.538
1.521
1.502
1.483
1.020
1.002
0.983
-0.000
O
N
Bn
1H NMR 400Mz CDCl3
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
–0.5
ppm
1.00
4.34
1.96
2.05
2.10
2.12
2.10
2.19
1.06
3.11


195.95
150.94
137.19
136.02
128.87
127.70
125.97
125.08
122.92
122.37
121.53
112.52
109.51
77.32
77.00
76.69
47.60
46.73
27.63
22.32
20.99
11.74
O
N
Bn
13C NMR 100Mz CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

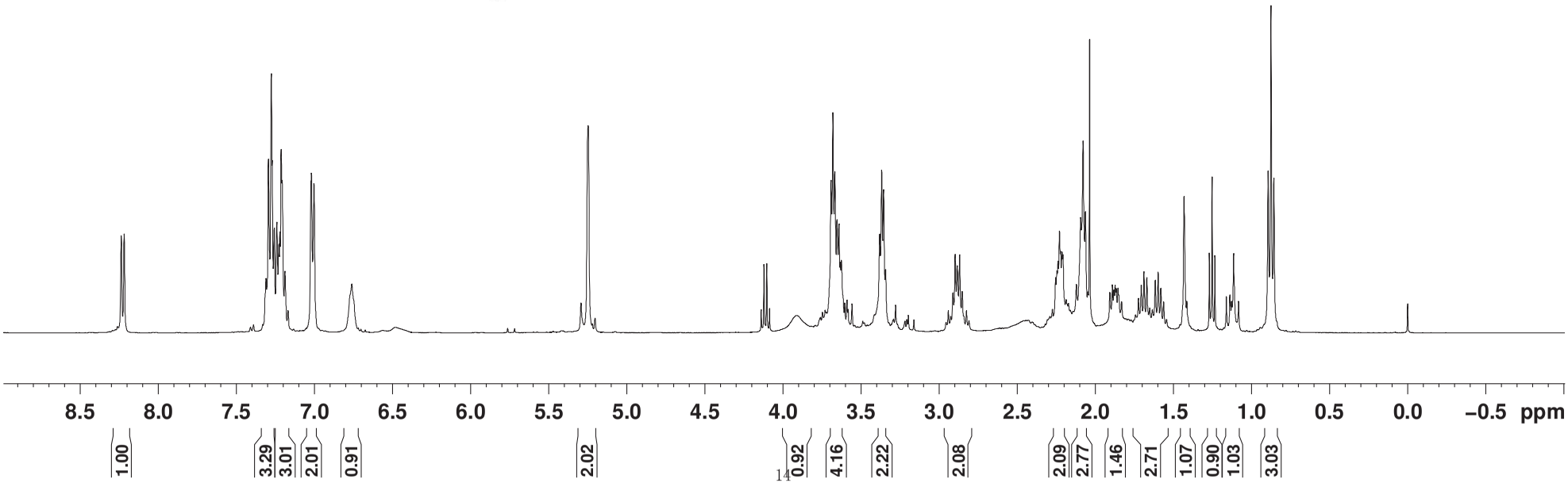

8.311
8.292
7.307
7.289
7.257
7.229
7.221
7.034
7.018
5.303
3.625
2.930
2.921
2.914
2.906
2.899
2.890
2.439
2.426
2.412
2.398
2.382
2.368
2.354
2.341
2.136
2.120
2.111
2.104
2.095
2.078
2.065
2.060
2.024
1.947
1.933
1.919
1.912
1.905
1.776
1.758
1.741
1.722
1.665
1.646
1.629
1.611
0.928
0.910
0.891
-0.000
O
O
O
N
Bn
1H NMR, 400Mz, CDCl3
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm
1.00
6.28
2.00
1.97
2.99
2.06
2.13
3.25
1.08
2.50
3.03


197.14
174.41
149.80
137.42
135.97
129.03
127.85
126.03
125.38
123.14
122.57
121.74
111.97
109.59
77.32
77.00
76.69
51.51
47.20
46.85
31.35
29.18
27.22
19.11
8.44
O
O
O
N
Bn
13C NMR, 100Mz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

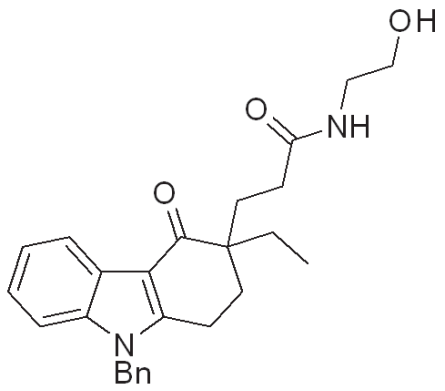




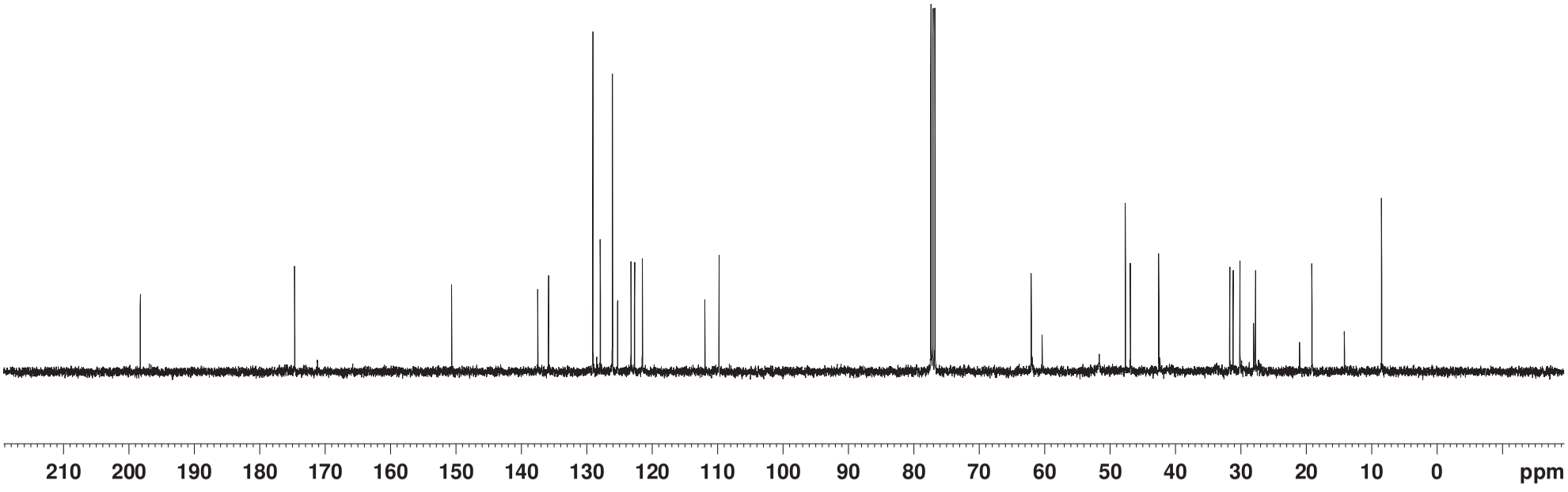
^{1}H NMR, 400Mz, $CDCl_3$

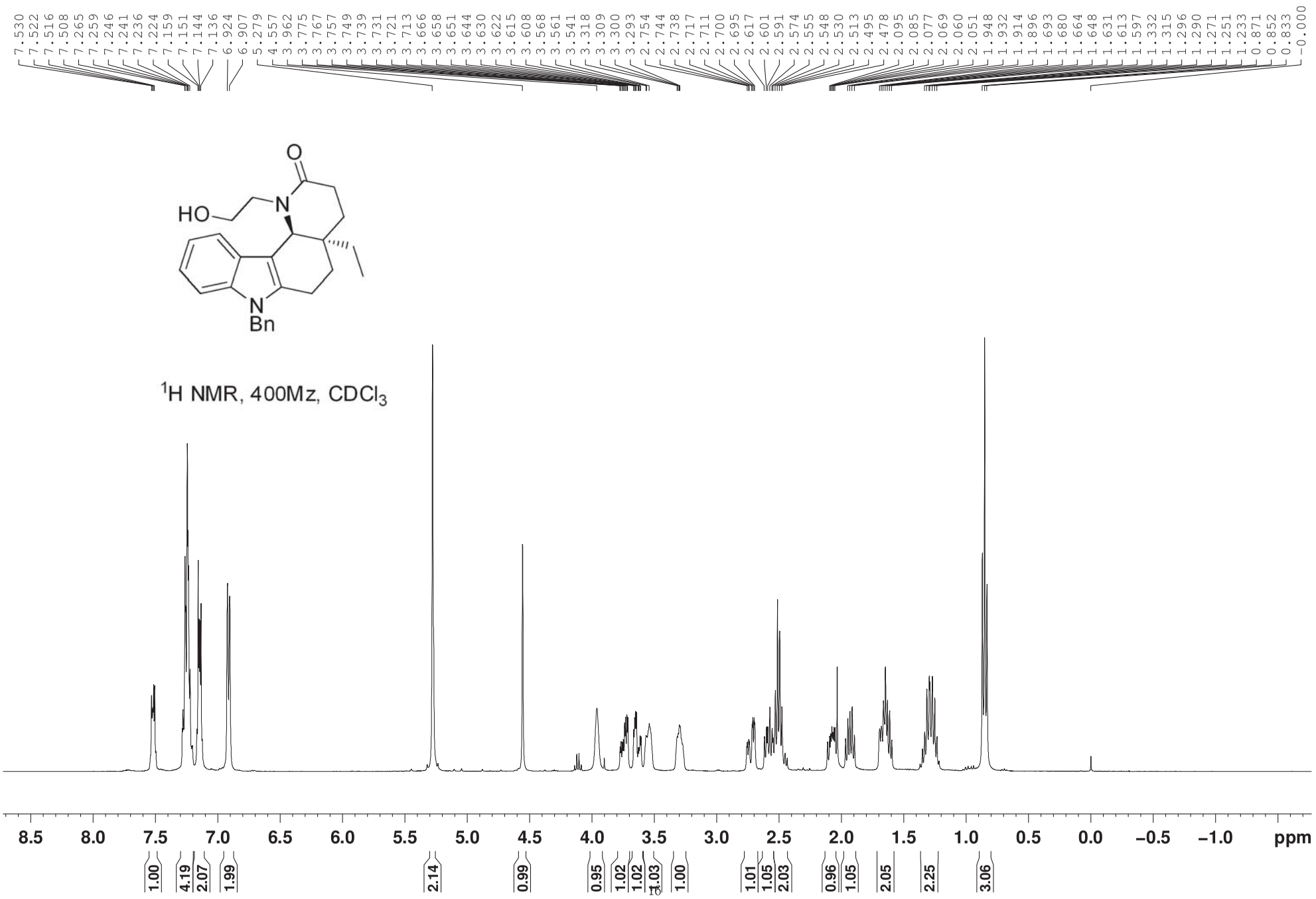


198.18
174.58
150.58
137.41
135.76
128.99
127.85
126.01
125.21
123.17
122.62
121.42
111.86
109.70
77.32
77.00
76.68
61.98
47.58
46.83
42.49
31.62
31.11
30.07
27.68
19.06
8.41

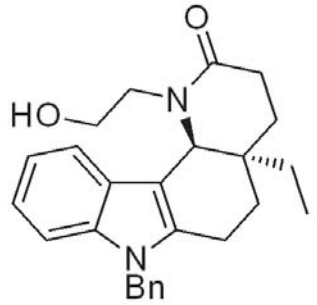


^{13}C NMR, 100Mz, $CDCl_3$

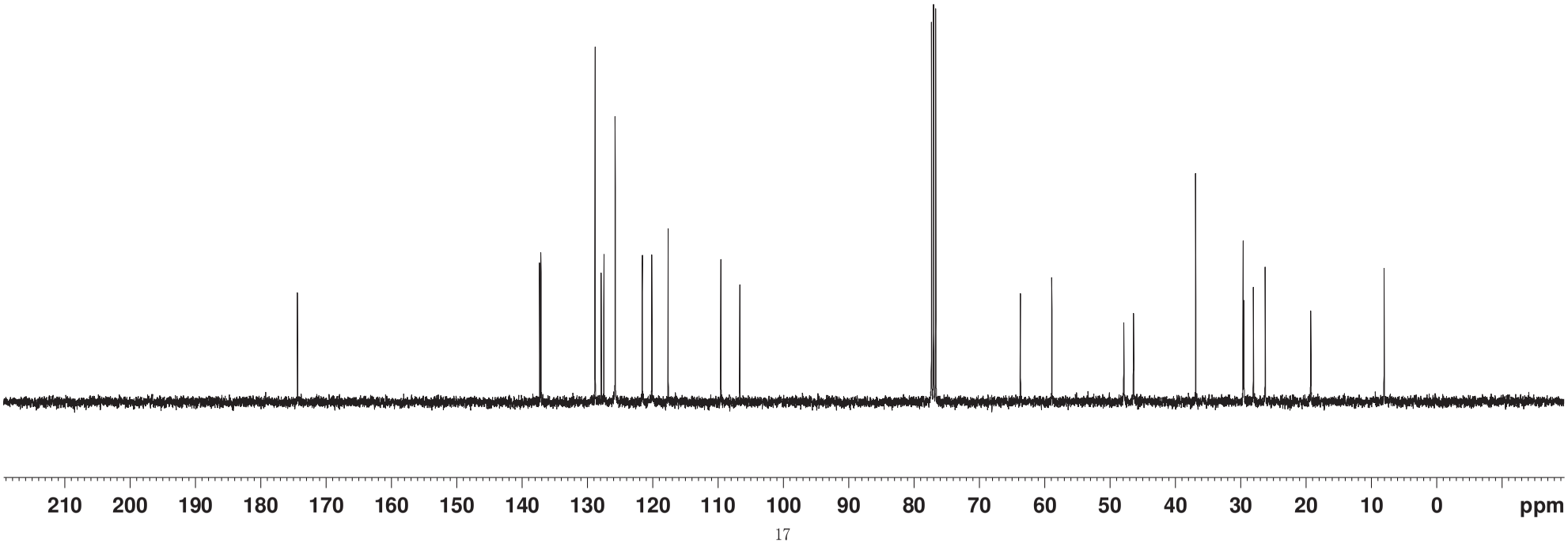


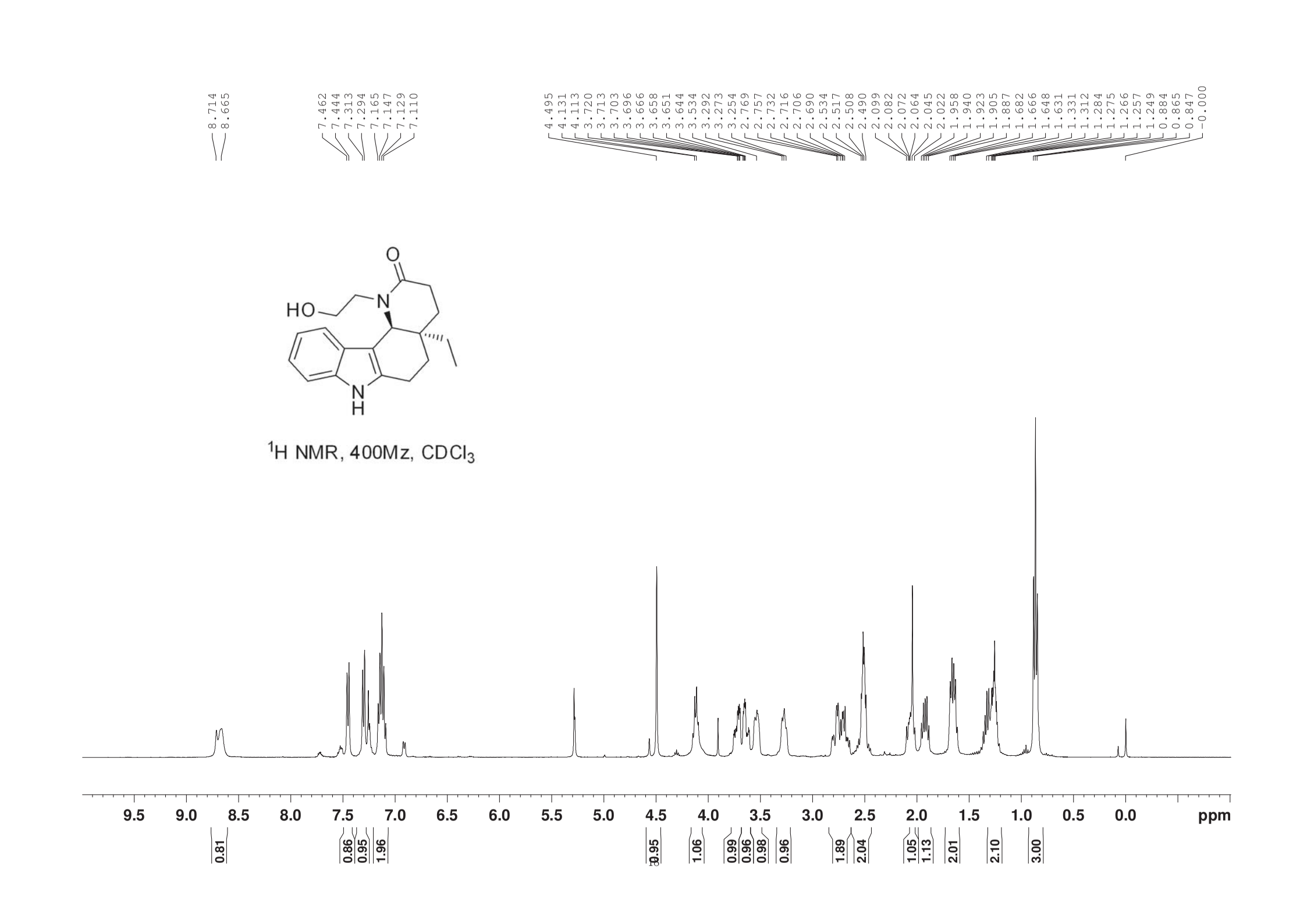

7.530
7.522
7.516
7.508
7.265
7.259
7.246
7.241
7.236
7.224
7.159
7.151
7.144
7.136
6.924
6.907
5.279
4.557
3.962
3.775
3.767
3.757
3.749
3.739
3.731
3.721
3.713
3.666
3.658
3.651
3.644
3.630
3.622
3.615
3.608
3.568
3.561
3.541
3.318
3.309
3.300
3.293
2.754
2.744
2.738
2.717
2.711
2.700
2.695
2.617
2.601
2.591
2.574
2.555
2.548
2.530
2.513
2.495
2.478
2.095
2.085
2.077
2.069
2.060
2.051
1.948
1.932
1.914
1.896
1.693
1.680
1.664
1.648
1.631
1.613
1.597
1.332
1.315
1.296
1.290
1.271
1.251
1.233
0.871
0.852
0.833
-0.000
O
HO
N
N
Bn
^{1}H NMR, 400Mz, CDCl$_3$
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
ppm
1.00
4.19
2.07
1.99
2.14
0.99
0.95
1.02
1.02
1.03
1.00
1.01
1.05
2.03
0.96
1.05
2.05
2.25
3.06

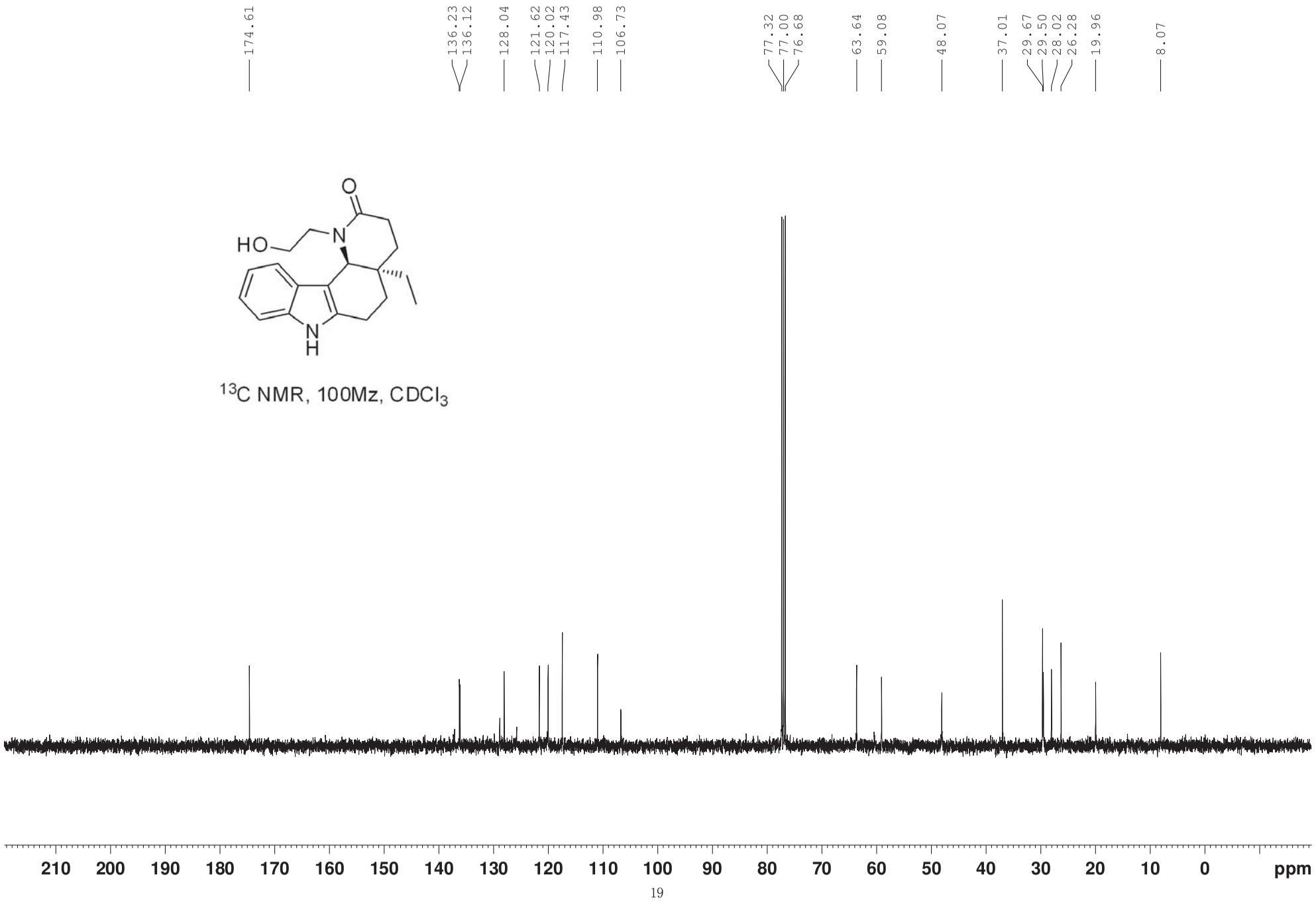
174.38
137.29
137.10
137.06
128.81
127.87
127.46
125.73
121.59
120.12
117.62
109.56
106.65
77.32
77.00
76.68
63.71
58.90
47.87
46.38
36.88
29.63
29.50
28.05
26.24
19.26
8.04



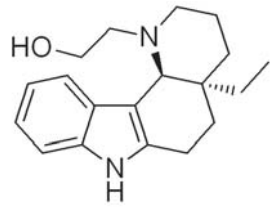
^{13}C NMR, 100Mz, $CDCl_3$



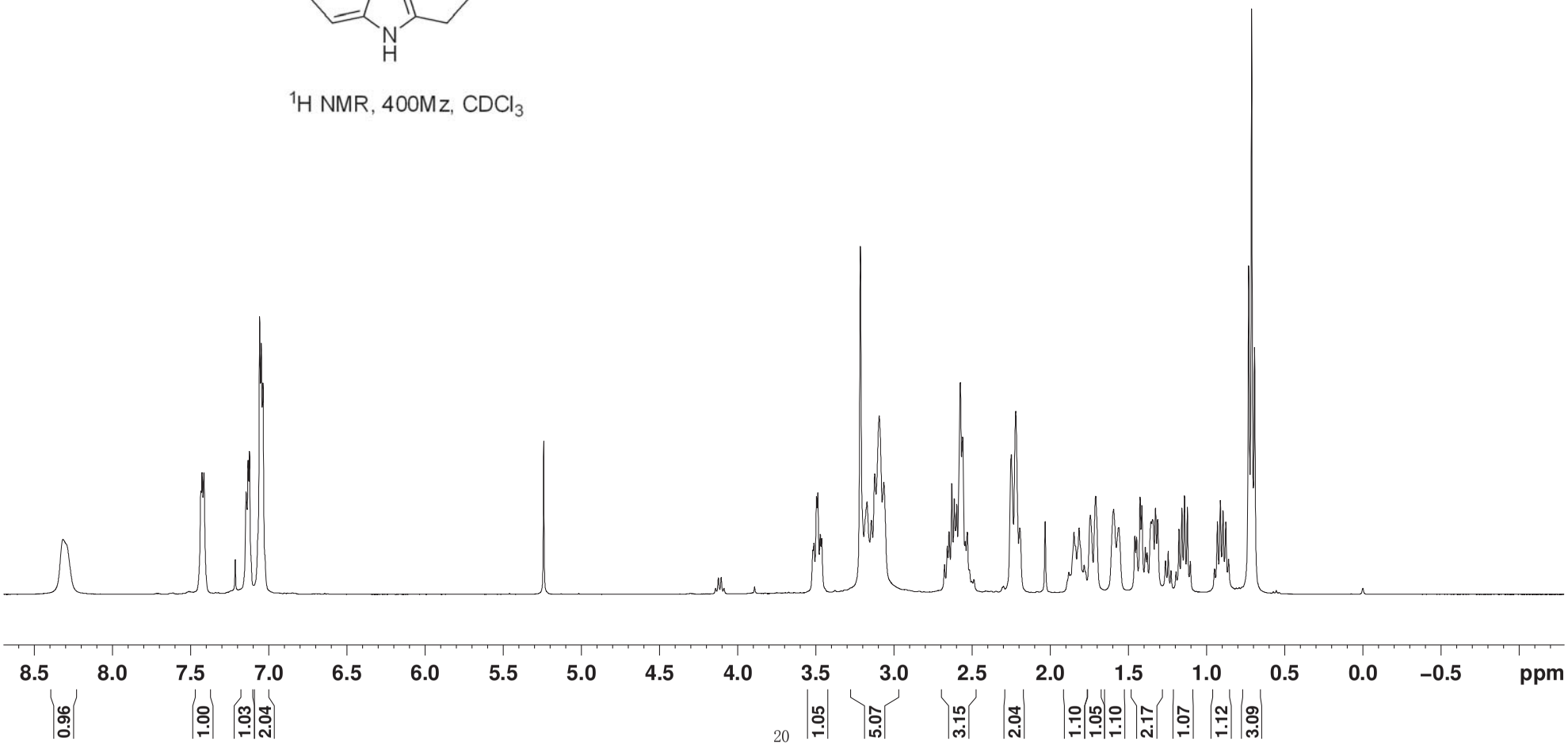

8.714
8.665
7.462
7.444
7.313
7.294
7.165
7.147
7.129
7.110
4.495
4.131
4.113
3.720
3.713
3.703
3.696
3.666
3.658
3.651
3.644
3.534
3.292
3.273
3.254
2.769
2.757
2.732
2.716
2.706
2.690
2.534
2.517
2.508
2.490
2.099
2.082
2.072
2.064
2.045
2.022
1.958
1.940
1.923
1.905
1.887
1.682
1.666
1.648
1.631
1.331
1.312
1.284
1.275
1.266
1.257
1.249
0.884
0.865
0.847
-0.000
HO
N
O
N
H
1H NMR, 400Mz, CDCl3
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.81
0.86
0.95
1.96
0.95
1.06
0.99
0.96
0.98
0.96
1.89
2.04
1.05
1.13
2.01
2.10
3.00

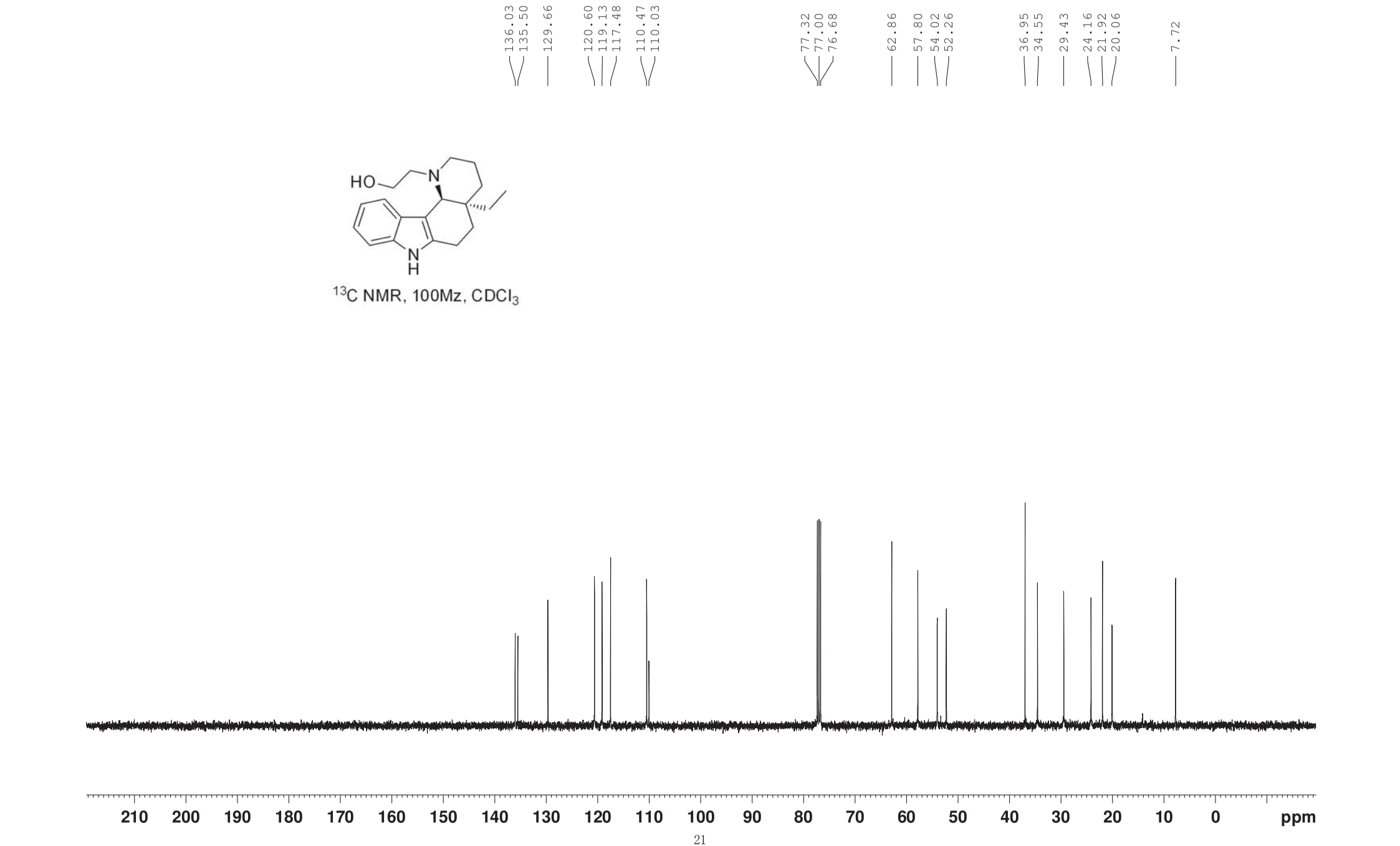

174.61
136.23
136.12
128.04
121.62
120.02
117.43
110.98
106.73
77.32
77.00
76.68
63.64
59.08
48.07
37.01
29.67
29.50
28.02
26.28
19.96
8.07
HO
N
O
N
H
13C NMR, 100Mz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

8.316
7.434
7.426
7.414
7.214
7.144
7.132
7.123
7.058
7.048
7.040
7.036
5.240
3.512
3.495
3.487
3.471
3.462
3.214
3.172
3.144
3.123
3.094
3.064
2.629
2.613
2.598
2.575
2.560
2.249
2.220
2.194
1.848
1.815
1.783
1.743
1.709
1.594
1.561
1.458
1.447
1.425
1.413
1.391
1.355
1.345
1.326
1.312
1.262
1.245
1.227
1.176
1.157
1.141
1.122
1.103
0.949
0.930
0.911
0.894
0.876
0.858
0.730
0.711
0.692
-0.000

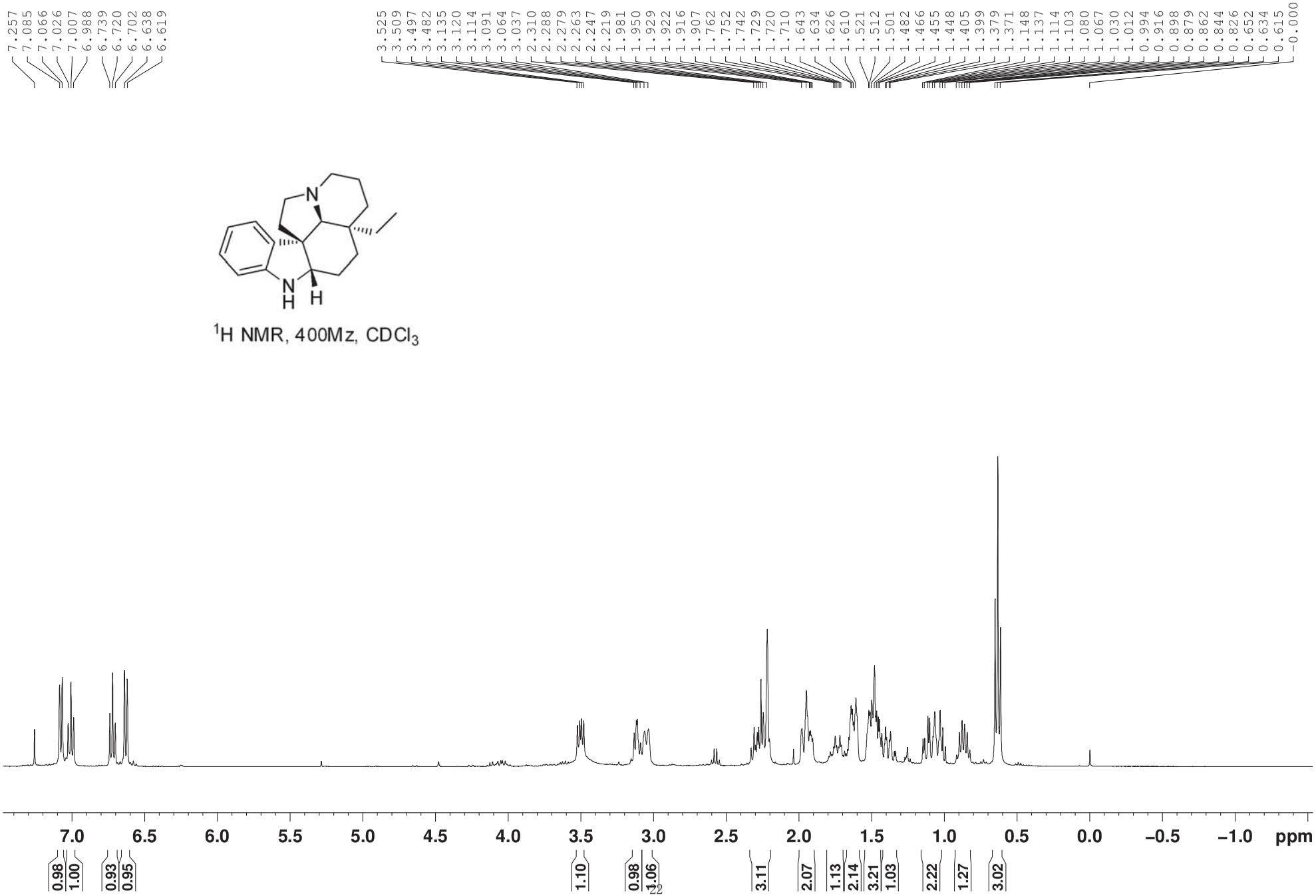


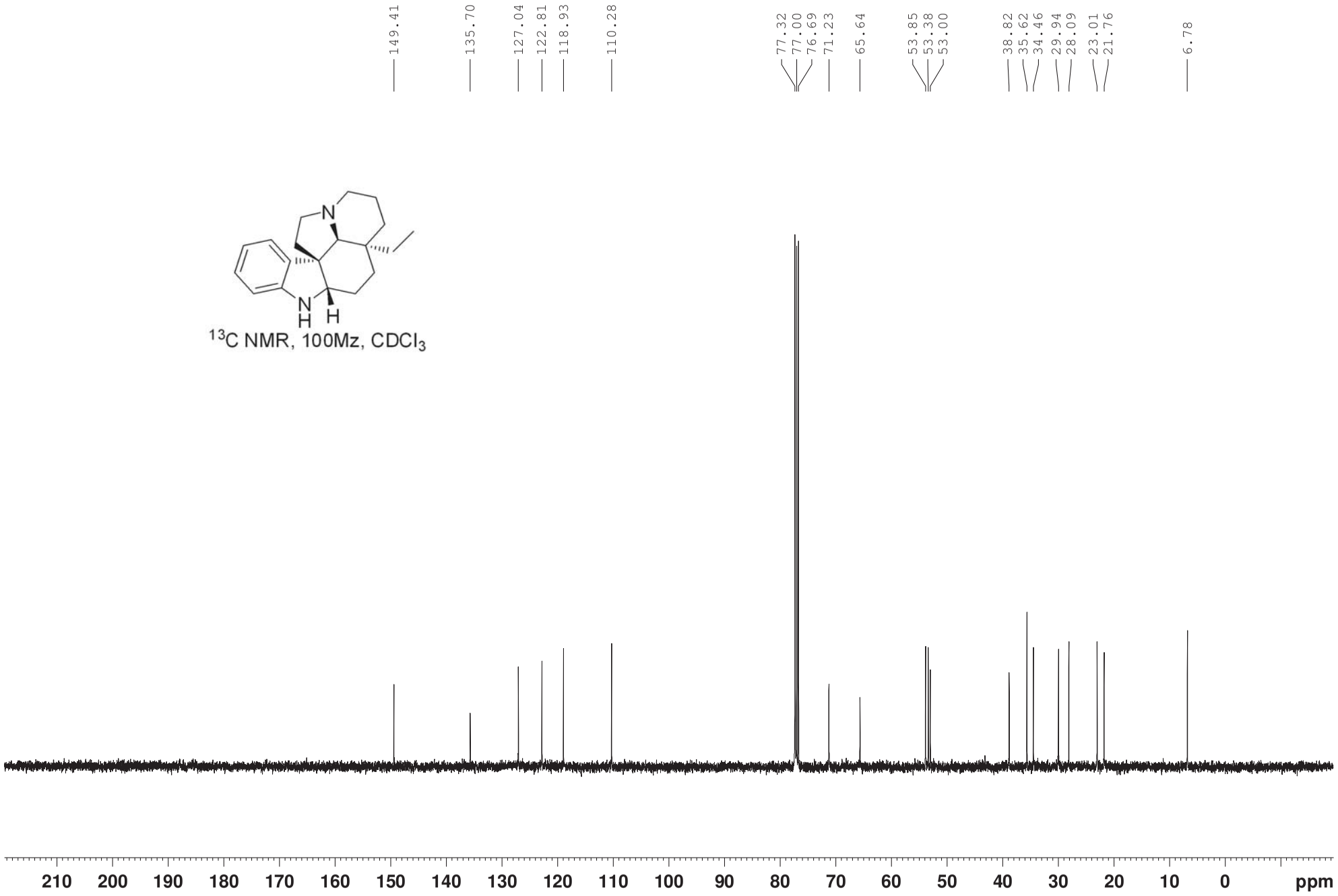
^{1}H NMR, 400Mz, $CDCl_3$





^{13}C NMR, 100Mz, $CDCl_3$


7.257
7.085
7.066
7.026
7.007
6.988
6.739
6.720
6.702
6.638
6.619
3.525
3.509
3.497
3.482
3.135
3.120
3.114
3.091
3.064
3.037
2.310
2.288
2.279
2.263
2.247
2.219
1.981
1.950
1.929
1.922
1.916
1.907
1.762
1.752
1.742
1.729
1.720
1.710
1.643
1.634
1.626
1.610
1.521
1.512
1.501
1.482
1.466
1.455
1.448
1.405
1.399
1.379
1.371
1.148
1.137
1.114
1.103
1.080
1.067
1.030
1.012
0.994
0.916
0.898
0.879
0.862
0.844
0.826
0.652
0.634
0.615
-0.000
N
N
H
H
^{1}H NMR, 400Mz, $CDCl_3$
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
–0.5
–1.0
ppm
0.98
1.00
0.93
0.95
1.10
0.98
1.06
3.11
2.07
1.13
2.14
3.21
1.03
2.22
1.27
3.02


149.41
135.70
127.04
122.81
118.93
110.28
77.32
77.00
76.69
71.23
65.64
53.85
53.38
53.00
38.82
35.62
34.46
29.94
28.09
23.01
21.76
6.78
N
N
H
H
13C NMR, 100Mz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm