Electronic Supplementary Material (ESI) for ChemComm.

Bioinspired Total Synthesis and Structural Revision of Yuremamine, an Alkaloid from the Entheogenic Plant *Mimosa tenuiflora*

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SUPPLEMENTARY INFORMATION

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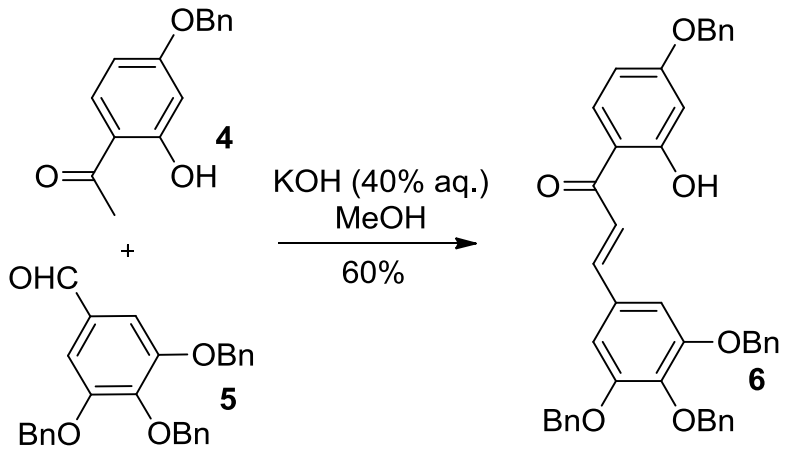
General

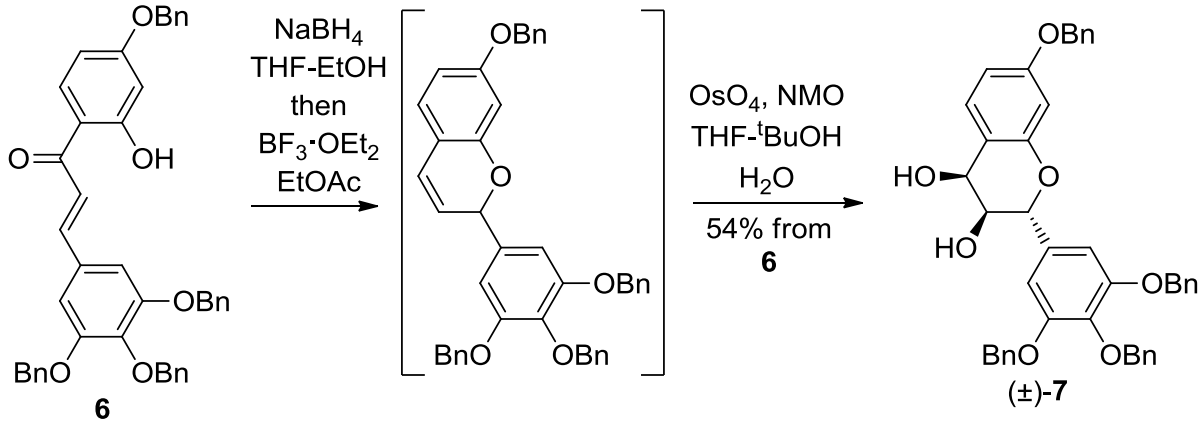
Commercially available reagents were used throughout without purification unless otherwise stated. Anhydrous solvents were used as supplied. Tetrahydrofuran was distilled from sodium benzophenone ketyl under nitrogen atmosphere. Dichloromethane was distilled from calcium hydride under a nitrogen atmosphere. Ether refers to diethyl ether. All reactions were routinely carried out in oven-dried glassware under a nitrogen or argon atmosphere unless otherwise stated. Analytical thin layer chromatography was performed using silica plates and compounds were visualized at 254 and/or 360 nm ultraviolet irradiation followed by staining with either alkaline permanganate or ethanolic vanillin solution. Infrared spectra were obtained using a Perkin Elmer spectrum One Fourier Transform Infrared spectrometer as thin films between sodium chloride plates. Absorption maxima are expressed in wavenumbers (cm^{-1}). Melting points were recorded on an Electrothermal melting point apparatus and are uncorrected. NMR spectra were recorded as indicated on either a spectrometer operating at 400 MHz for ^{1}H nuclei and 100 MHz for ^{13}C nuclei or on a spectrometer operating at 500 MHz and 125 MHz for ^{1}H and ^{13}C nuclei, respectively. Chemical shifts are reported in parts per million (ppm) relative to the tetramethylsilane peak recorded as δ 0.00 ppm in $CDCl_3$/ TMS solvent, or the residual chloroform (δ 7.26 ppm), DMSO (δ 2.50 ppm) or methanol (δ 3.31 ppm) peaks. The ^{13}C NMR values were referenced to the residual chloroform (δ 77.1 ppm), DMSO (δ 39.5 ppm) or methanol (δ 49.0 ppm) peaks. ^{13}C NMR values are reported as chemical shift δ, multiplicity and assignment. ^{1}H NMR shift values are reported as chemical shift δ, relative integral, multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet), coupling constant (*J* in Hz) and assignment. Assignments are made with the aid of DEPT 135, COSY, NOESY, HMBC and HSQC experiments. High resolution mass spectra were obtained by electrospray ionization in positive ion mode at a nominal accelerating voltage of 70 eV on a microTOF mass spectrometer.

(*E*)-1-(4-(Benzyloxy)-2-hydroxyphenyl)-3-(3,4,5-tris(benzyloxy)phenyl)prop-2-en-1-one (6)



Acetophenone **4**[1] (1.45 g, 6.0 mmol) and aldehyde **5**[2] (2.54 g, 6.0 mmol) were suspended in MeOH (33 mL) and warmed to 50 °C with stirring. Potassium hydroxide (40% w/w aqueous solution, 18 mL) was added and the reaction mixture was stirred at 80 °C for 24 h. Upon cooling to room temperature, the reaction mixture was diluted with water (50 mL) and extracted exhaustively with ethyl acetate. The combined organics were dried (sodium sulfate), filtered and concentrated *in vacuo* to yield a yellow-brown powder, which was recrystallized from dichloromethane/ethyl acetate to yield the *title compound* (2.35 g, 3.6 mmol, 60%) as a yellow solid; m.p. 145-155 °C; ν_{max}/cm^{-1} 3066, 3031, 2864, 1634, 1578, 1502, 1376, 1334, 1271, 1250, 1227, 1212, 1193, 1127, 1023, 974, 833, 798, 730, 693; HRMS Found: $[M + Na]^+$, 671.2408. $[C_{43}H_{36}O_6 + Na]^+$ requires 671.2404; δ_H (400 MHz, $CDCl_3$) 13.41 (1 H, s, OH), 7.77 (1 H, d, *J* = 8.5, ArH), 7.72 (1 H, d, *J* = 15.3, CH), 7.45-7.27 (21 H, m, 20 x ArH + CH), 6.91 (2 H, s, 2 x ArH), 6.58-6.55 (2 H, m, 2 x ArH), 5.16 (4 H, s, 2 x CH_2), 5.13 (2 H, s, CH_2), 5.12 (2 H, s, CH_2); δ_C (100 MHz, $CDCl_3$) 191.7 (C=O), 166.8 (C), 165.4 (C), 153.2 (2 x C), 144.6 (CH), 141.1 (C), 137.6 (C), 136.9 (2 x C), 136.0 (C), 131.4 (CH), 130.4 (C), 128.9 (2 x CH), 128.8 (4 x CH), 128.7 (2 x CH), 128.5 (CH), 128.4 (2 x CH), 128.2 (2 x CH), 128.1 (CH), 127.7 (2 x CH), 127.6 (4 x CH), 119.7 (CH), 114.4 (C), 108.7 (2 x CH), 108.4 (CH), 102.2 (CH), 75.5 (CH_2), 71.6 (2 x CH_2), 70.4 (CH_2).

(±)-(2*R*,3*S*,4*S*)-7-(Benzyloxy)-2-(3,4,5-tris(benzyloxy)phenyl)chroman-3,4-diol [(±)-7]

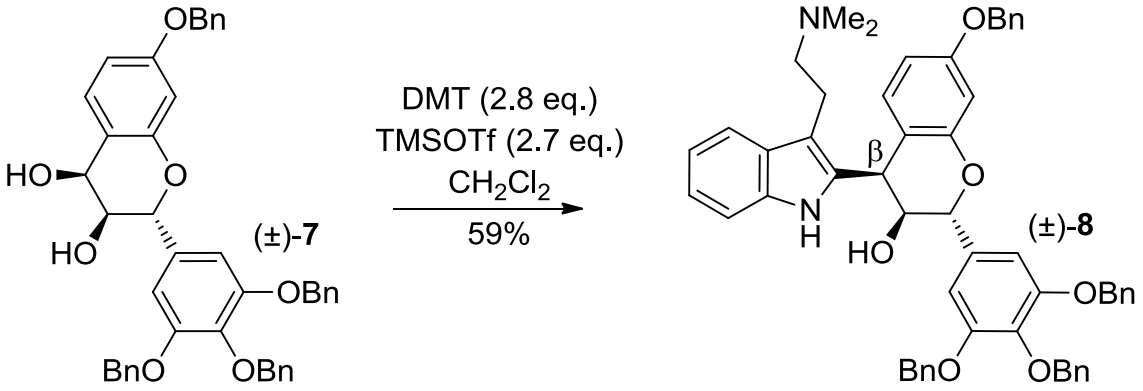


Chalcone **6** (1.95 g, 3.0 mmol) was taken up in THF-EtOH (2:1, 69 mL) and treated with sodium borohydride (114 mg, 3.0 mmol) at room temperature. The reaction mixture was stirred at this temperature for 1 hour, whereupon the bright yellow solution had turned colourless. The reaction mixture was diluted with ethyl acetate (200 mL) and washed with brine until the washings were neutral. The combined organic extracts were dried (sodium sulfate), filtered and the filtrate (~250 mL) immediately treated with $BF_3{\cdot}OEt_2$ (0.4 mmol, 1.0 mL of a 0.4M solution in CH_2Cl_2) dropwise over 10 minutes at 0 °C. The reaction mixture was stirred for 1 hour at room temperature, washed with water, saturated aqueous sodium hydrogen carbonate solution, brine, dried (sodium sulfate), filtered and concentrated *in vacuo* to give the crude flavene (~1.3 g) as a brown oil that was used immediately in the next step.

Osmium tetroxide (0.06 mmol, 0.3 mL of a 0.2 M solution in ^{t}BuOH) was added to a solution of *N*-methylmorpholine *N*-oxide (387 mg, 3.3 mmol) in THF-water (20:1, 26.25 mL) followed by dropwise addition of crude flavene (~1.3 g) in THF (10 mL). The reaction mixture was stirred at room temperature for 13 hours, diluted with dichloromethane (100 mL), washed with sodium thiosulfate (1 M, 100 mL) and brine, dried (sodium sulfate), filtered and concentrated *in vacuo* to yield a brown solid, which was recrystallized from dichloromethane/diethyl ether to yield the *title compound* (1.08 g, 1.6 mmol, 54% from **6**) as a colourless solid; m.p. 185-187 °C; ν_{max}/cm^{-1} 3383, 3031, 2927, 1584, 1503, 1435, 1239, 1166, 1113, 1078, 1023, 820, 730, 693; HRMS Found: $[M + Na]^+$, 689.2513. $[C_{43}H_{38}O_7 + Na]^+$ requires 689.2510; δ_H (400 MHz, DMSO-d_6) 7.48-7.26 (20 H, m, 20 x ArH), 7.19 (1 H, d, *J* = 8.5, ArH), 6.90 (2 H, s, 2 x ArH), 6.61 (1 H, dd, *J* = 8.5, 2.5, ArH), 6.46 (1 H, d, *J* = 2.5, ArH), 5.25 (1 H, d, *J* = 4.9, OH), 5.12 (4 H, s, 2 x CH_2), 5.09 (2 H, s, CH_2), 4.95-4.92 (3 H, m, CH_2 + CH), 4.78 (1 H, d, *J* = 7.2, OH), 4.45 (1 H, dd, *J* = 4.9, 3.5, CH), 3.93 (1 H, ddd,

J = 9.3, 7.2, 3.5, CH); δ_C (100 MHz, DMSO-d_6) 159.1 (C), 154.5 (C), 151.9 (2 x C), 137.8 (C), 137.12 (C), 137.10 (2 x C), 136.9 (C), 134.8 (C), 131.3 (CH), 128.40 (2 x CH), 128.36 (4 x CH), 128.1 (2 x CH), 128.0 (2 x CH), 127.8 (2 x CH), 127.72 (2 x CH), 127.66 (4 x CH), 127.5 (2 x CH), 117.0 (C), 108.1 (CH), 107.0 (2 x CH), 101.4 (CH), 76.8 (CH), 74.3 (CH_2), 70.3 (2 x CH_2), 69.8 (CH), 69.1 (CH_2), 65.3 (CH).

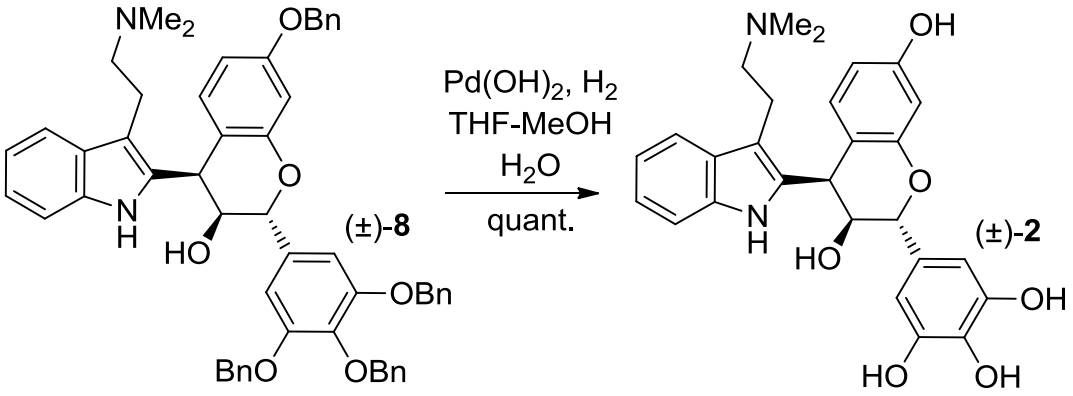
(±)-(2*R*,3*S*,4*R*)-7-(Benzyloxy)-4-(3-(2-(dimethylamino)ethyl)indol-2-yl)-2-(3,4,5-tris(benzyloxy)phenyl)chroman-3-ol [(±)-8]



A solution of flavanol (±)-**7** (860 mg, 1.3 mmol) and *N*,*N*-dimethyltryptamine (700 mg, 3.7 mmol) in dichloromethane (300 mL) was cooled to 0 °C. Trimethylsilyl trifluoromethanesulfonate (630 µL, 3.5 mmol) was added over 20 minutes, at which stage complete consumption of starting material was observed by TLC. The reaction mixture was diluted with water (300 mL), the layers were partitioned and the aqueous layer was further extracted with dichloromethane (2 x 200 mL). The combined organic layers were dried (sodium sulfate), filtered and concentrated *in vacuo*. The crude mixture was purified by flash chromatography on silica gel (97:3 CH_2Cl_2-MeOH + 0.1% NH_4OH) to yield the *title compound* (640 mg, 0.76 mmol, 59%, 4β:4α > 30:1) as a colourless solid; m.p. 71-73°C; ν_{max}/cm^{-1} 2859, 1618, 1586, 1499, 1453, 1427, 1328, 1248, 1161, 1099, 1016, 833, 730, 694; HRMS Found: $[M + Na]^+$, 859.3704. $[C_{55}H_{52}N_2O_6 + Na]^+$ requires 859.3718; δ_H (500 MHz, $CDCl_3$) 7.78 (1 H, br s, NH), 7.53 (1 H, d, *J* = 7.3, ArH), 7.43-7.23 (21 H, m, 21 x ArH), 7.15 (1 H, app td, *J* = 7.3, 1.2, ArH), 7.10 (1 H, app td, *J* = 7.3, 1.1, ArH), 6.82 (1 H, d, *J* = 8.5, ArH), 6.76 (2 H, s, 2 x ArH), 6.67 (1 H, d, *J* = 2.5, ArH), 6.55 (1 H, dd, *J* = 8.5, 2.5, ArH), 5.08 (2 H, s, CH_2), 5.06 (2 H, s, CH_2), 5.05 (2 H, s, CH_2), 5.03 (2 H, s, CH_2), 4.98 (1 H, d, *J* = 8.1, CH), 4.42 (1 H, d, *J* = 5.7, CH), 4.23 (1 H, dd, *J* = 8.1, 5.7, CH), 3.01-2.84 (2 H, m, CH_2), 2.71-2.54 (2 H, m, CH_2), 2.19 (6 H, s, 2 x Me), 1 x OH not observed; δ_C (125 MHz, $CDCl_3$) 159.4 (C), 154.8 (C), 152.9 (2 x C), 138.5 (C), 138.2 (C), 137.3 (2 x C), 136.9 (C),

136.3 (C), 134.9 (C), 134.3 (C), 131.5 (CH), 128.8 (2 x CH), 128.60 (2 x CH), 128.56 (4 x CH), 128.31 (C), 128.27 (2 x CH), 128.2 (CH), 127.9 (2 x CH), 127.8 (CH), 127.64 (4 x CH), 127.60 (2 x CH) 122.0 (CH), 119.5 (CH), 118.2 (CH), 113.8 (C), 111.2 (CH), 109.7 (CH), 106.8 (2 x CH), 102.4 (CH), 79.7 (CH), 75.3 (CH_2), 71.3 (2 x CH_2), 70.2 (CH_2), 70.0 (CH), 60.1 (CH_2), 45.1 (2 x Me), 38.5 (CH), 22.3 (CH_2) 1 x C not observed.

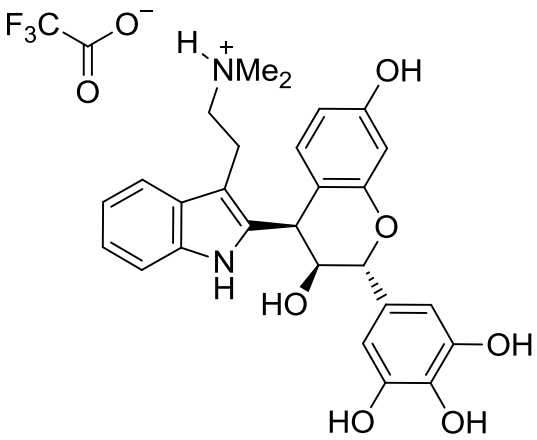
(±)-Yuremamine (2)



To a solution of indole-flavanoid (±)-**8** (45 mg, 54 µmol) in THF-MeOH-H_2O (4.5 mL, 4:4:1) was added palladium hydroxide (20% on carbon, 20 mg). The reaction mixture was stirred under an atmosphere of hydrogen for 18 hours, diluted with methanol (10 mL), filtered through Celite® and concentrated *in vacuo*. The resulting solid was taken up in acetone (10 mL), filtered through Celite® and concentrated *in vacuo* to yield the *title compound* (free base, 25 mg, 53 µmol, quant.) as a colourless solid; m.p. 210-220 °C (decomp.); ν_{max}/cm^{-1} 2928, 1590, 1501, 1455, 1326, 1220, 1158, 1102, 1012, 838, 734, 695; HRMS Found: $[M + H]^+$, 477.2025. $[C_{27}H_{28}N_2O_6 + H]^+$ requires 477.2020; δ_H (500 MHz, CD_3OD) 7.50 (1 H, d, *J* = 7.7, ArH), 7.28 (1 H, d, *J* = 7.9, ArH), 7.04 (1 H, app td, *J* = 7.6, 0.9, ArH), 6.99 (1 H, app td, *J* = 7.3, 0.7, ArH), 6.49 (1 H, d, *J* = 2.4, ArH), 6.45 (1 H, d, *J* = 8.4, ArH), 6.38 (2 H, s, 2 x ArH), 6.27 (1 H, dd, *J* = 8.4, 2.4, ArH), 5.20 (1 H, d, *J* = 3.9, CH), 4.23 (1 H, d, *J* = 3.1, CH), 4.20 (1 H, app t, *J* = 3.7, CH), 2.94-2.73 (3 H, m, CH_2 + CH of CH_2), 2.67-2.60 (1 H, m, CH of CH_2), 2.42 (6 H, s, 2 x Me); δ_C (125 MHz, CD_3OD) 158.7 (C), 155.9 (C), 147.2 (2 x C), 137.6 (C), 135.9 (C), 133.9 (C), 132.2 (C), 131.2 (CH), 128.6 (C), 122.3 (CH), 119.8 (CH), 118.5 (CH), 113.9 (C), 112.1 (CH), 110.2 (C), 109.4 (CH), 105.5 (2 x CH), 103.6 (CH), 81.6 (CH), 71.9 (CH), 60.7 (CH_2), 44.4 (2 x Me), 35.5 (CH), 21.9 (CH_2).

(±)-Yuremamine (TFA Salt)

The free base of yuremamine was subjected to reverse phase column chromatography (Alltech C18 SPE cartridge, 0.1% aqueous trifluoroacetic acid in a gradient of 0-20% acetonitrile) to yield the *title compound* as a purple solid.



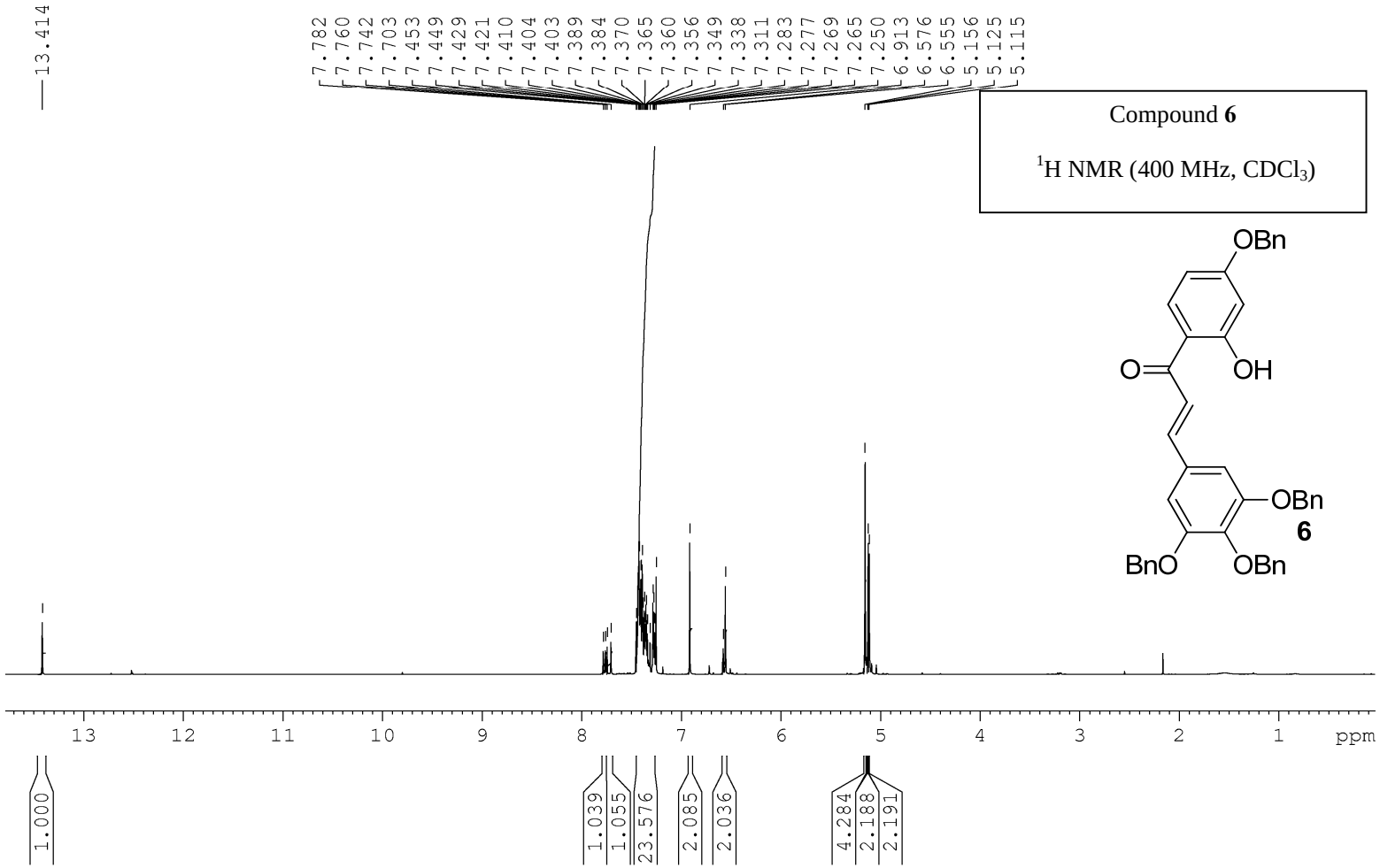
m.p. 168-173 °C; ν_{max}/cm^{-1} 3232, 2698, 1672, 1625, 1537, 1509, 1459, 1346, 1193, 1134, 1033, 840, 797, 717; HRMS Found: $[M + H]^+$, 477.2022. $[C_{27}H_{28}N_2O_6 + H]^+$ requires 477.2020;

δ_H (500 MHz, CD_3OD): see manuscript Table 1; δ_C (125 MHz, CD_3OD): see manuscript Table 1;

δ_H (500 MHz, DMSO-d_6) 10.39 (1 H, s, NH), 9.89 (1 H, br s, NH), 9.34 (1 H, br s, OH), 8.90 (2 H, br s, 2 x OH), 8.14 (1 H, br s, OH), 7.55 (1 H, d, *J* = 7.7, ArH), 7.33 (1 H, d, *J* = 7.7, ArH), 7.02 (1 H, td, *J* = 7.7, 1.2, ArH), 6.97 (1 H, t, *J* = 7.6, ArH), 6.43 (1 H, d, *J* = 8.3, ArH), 6.37 (1 H, d, *J* = 2.4, ArH), 6.28 (2 H, s, 2 x ArH), 6.24 (1 H, dd, *J* = 8.3, 2.4, ArH), 5.44 (1 H, bs, OH), 5.03 (1 H, d, *J* = 5.4, CH), 4.20 (1 H, d, *J* = 4.1, CH), 4.07 (1 H, app t, *J* = 5.1, CH), 3.16-3.10 (1 H, m, CH of CH_2), 3.03-2.92 (3 H, m, CH_2 + CH of CH_2), 2.75 (3 H, d, *J* = 4.9, Me), 2.71 (3 H, d, *J* = 4.9, Me); δ_C (125 MHz, DMSO-d_6) 157.3 (C), 154.4 (C), 146.0 (2 x C), 135.9 (C), 135.6 (C), 132.4 (C), 130.0 (CH), 129.9 (C), 126.9 (C), 120.6 (CH), 118.2 (CH), 117.5 (CH), 112.4 (C), 111.5 (CH), 108.2 (CH), 106.9 (C), 104.8 (2 x CH), 102.3 (CH), 79.1 (CH), 69.2 (CH), 57.1 (CH_2), 42.2 (Me), 41.6 (Me), 35.1 (CH), 19.2 (CH_2); δ_F (470 MHz, DMSO-d_6) -73.8

References

1. R. S. Khupse, J. G. Sarver, J. A. Trendel, N. R. Bearss, M. D. Reese, T. E. Wiese, S. M. Boue, M. E. Burow, T. E. Cleveland, D. Bhatnagar, *J. Med. Chem.* **2011**, *54*, 3506-3523.
2. T.-J. Ding, X.-L. Wang, X.-P. Cao, *Chin. J. Chem.* **2006**, *24*, 1618-1624.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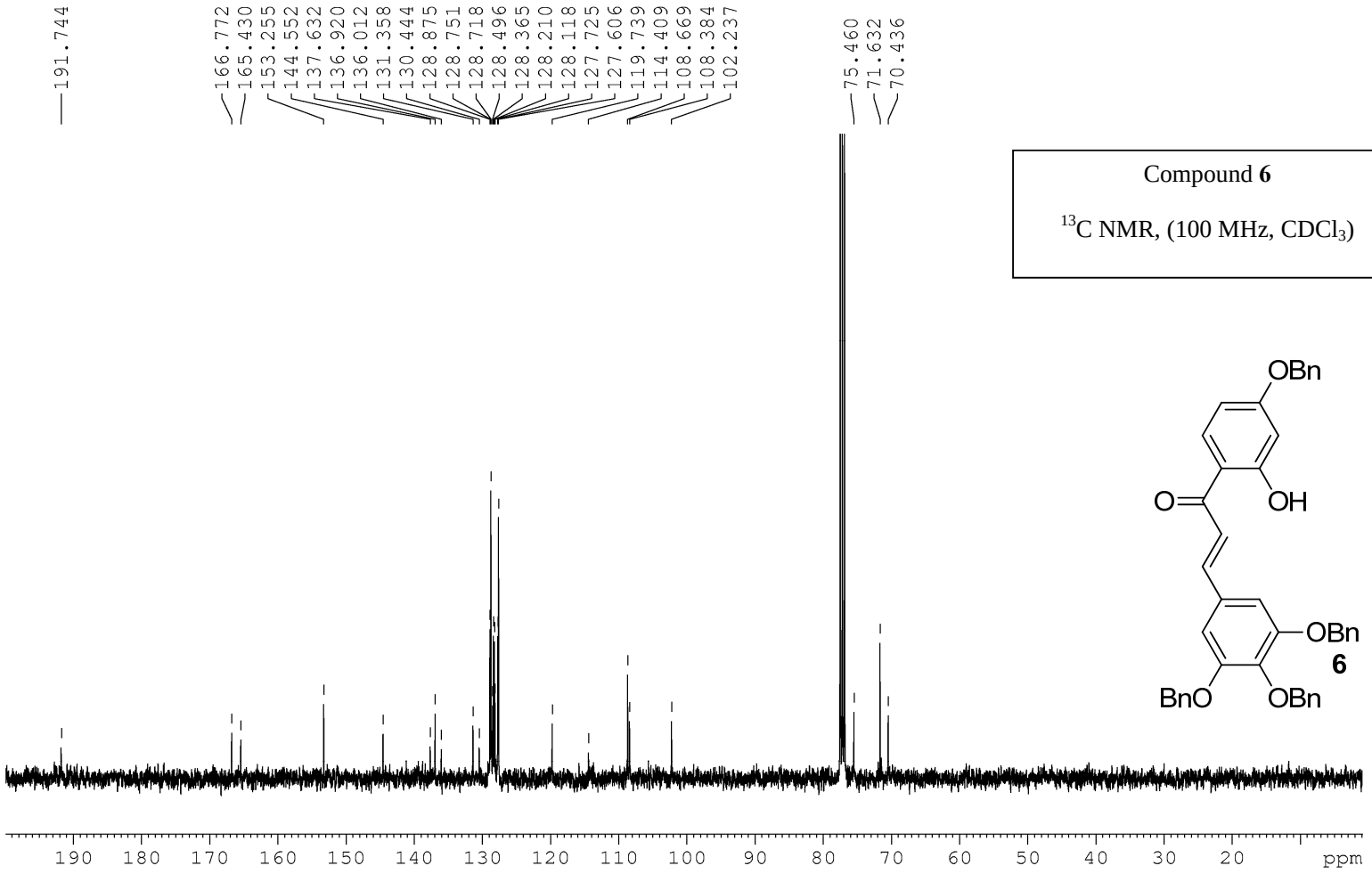
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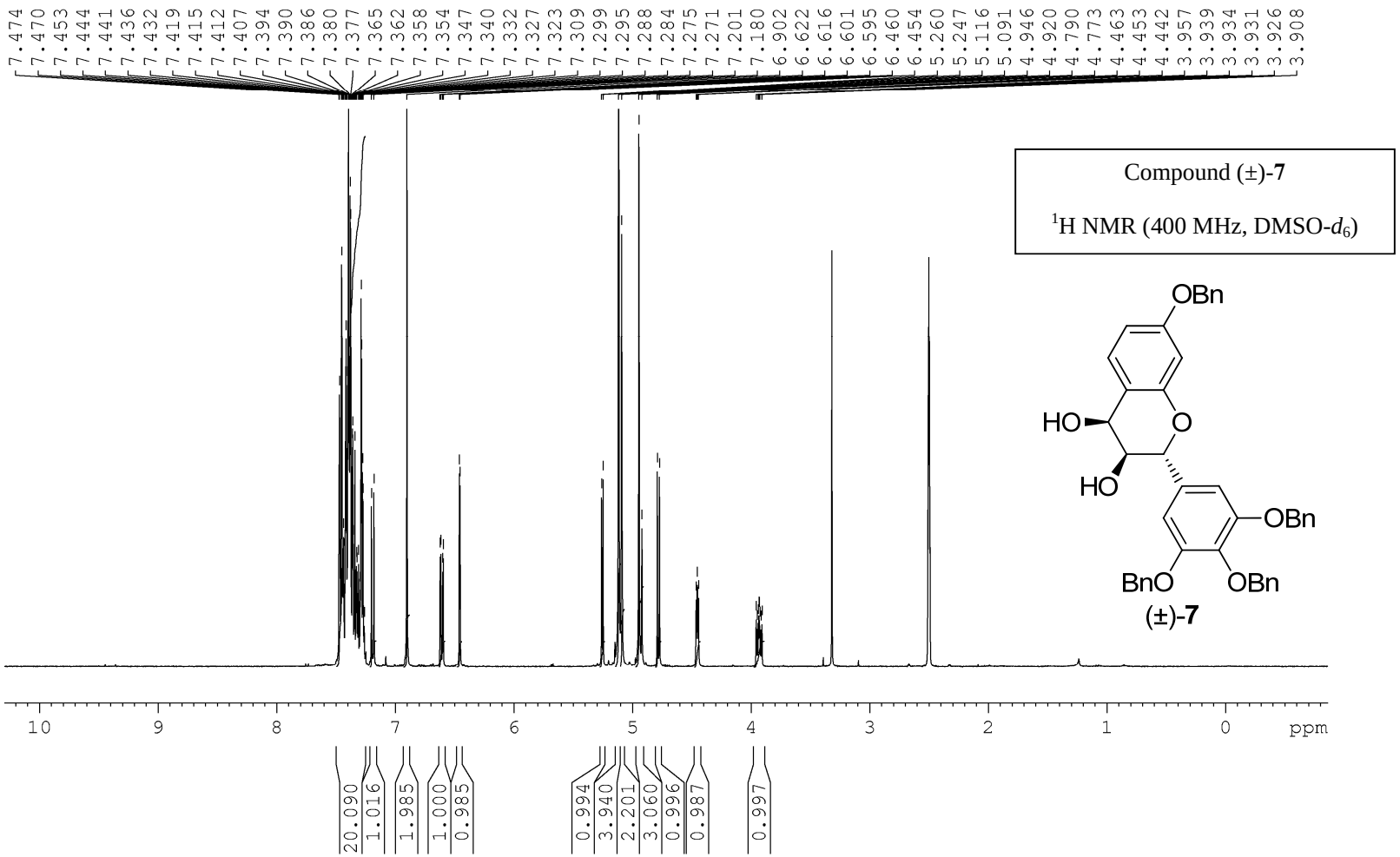
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3.908
Compound (±)-7
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O
HO
OBn
BnO
OBn
(±)-7
10
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8
7
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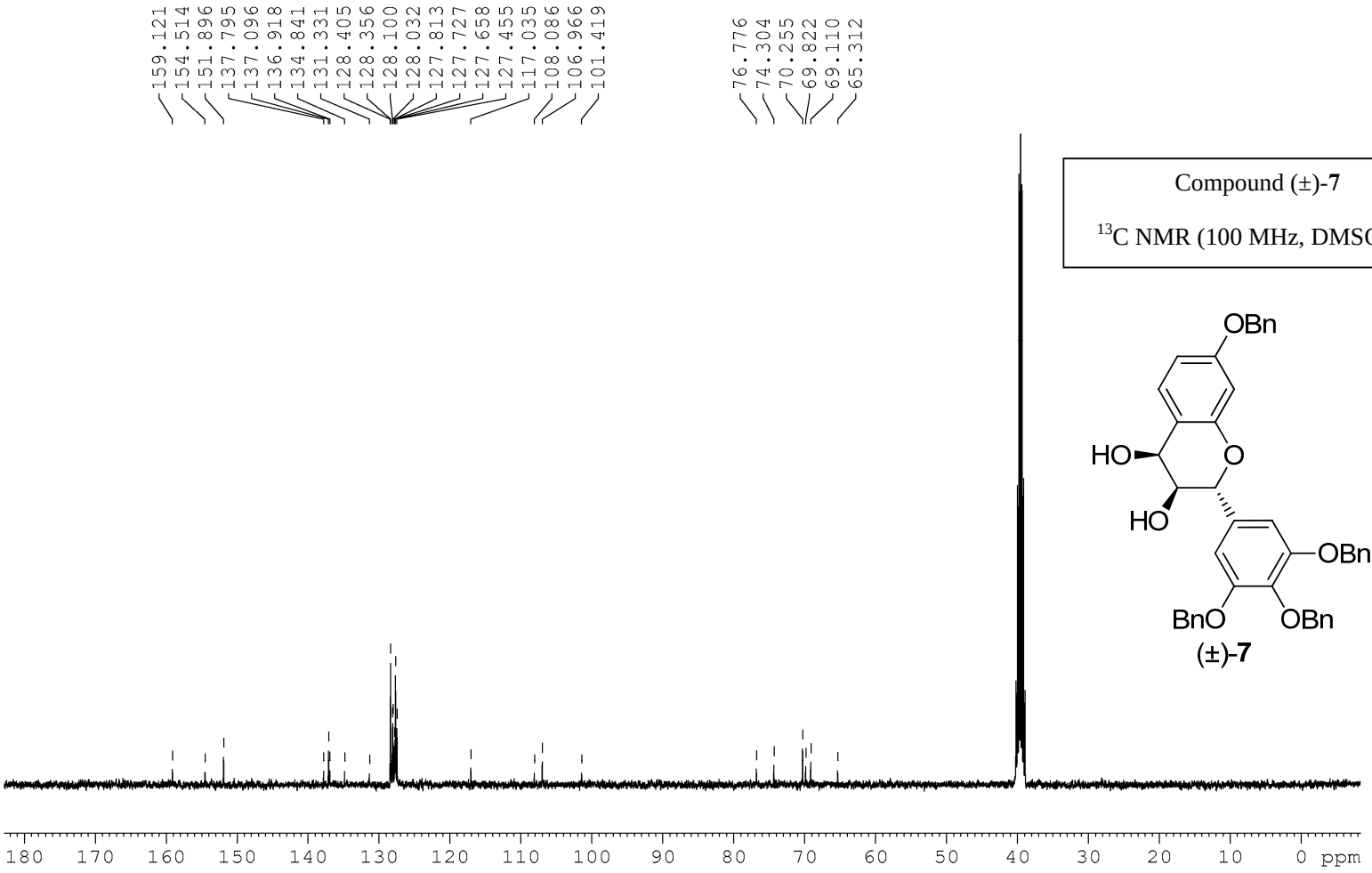
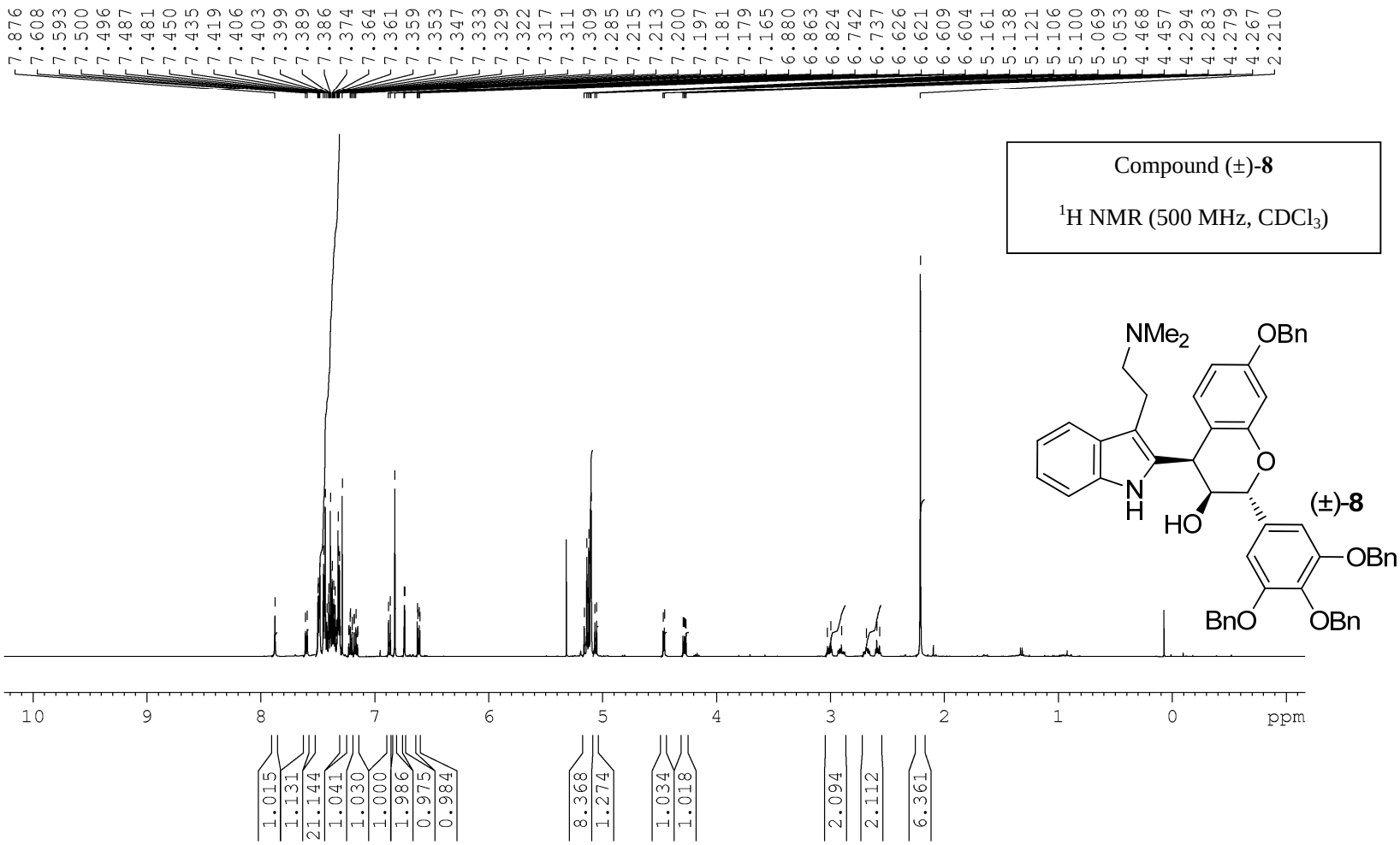


Compound (±)-**7**

^{13}C NMR (100 MHz, DMSO-d_6)

(±)-**7**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Compound (±)-8
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O
N
H
HO
(±)-8
OBn
BnO
OBn
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9
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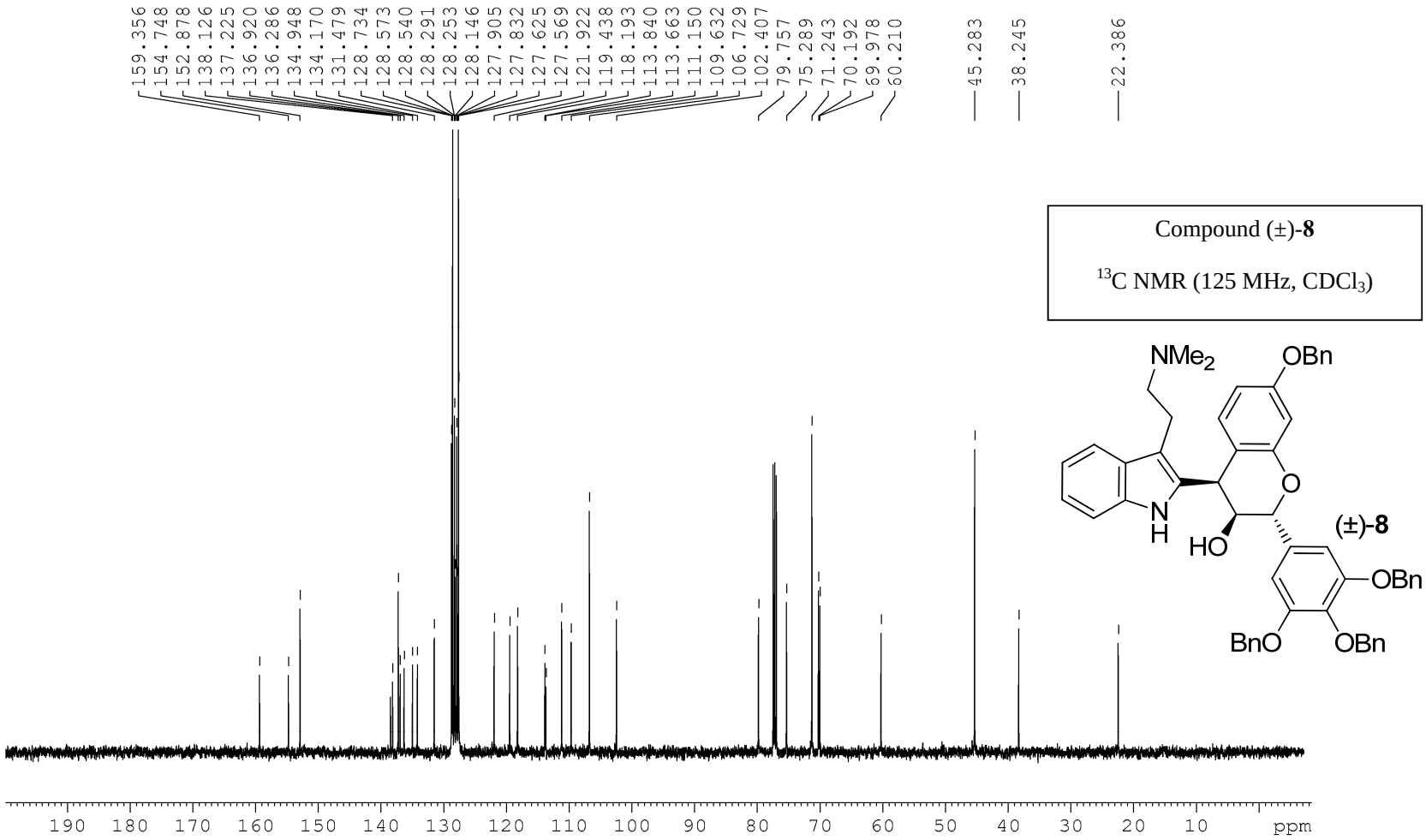
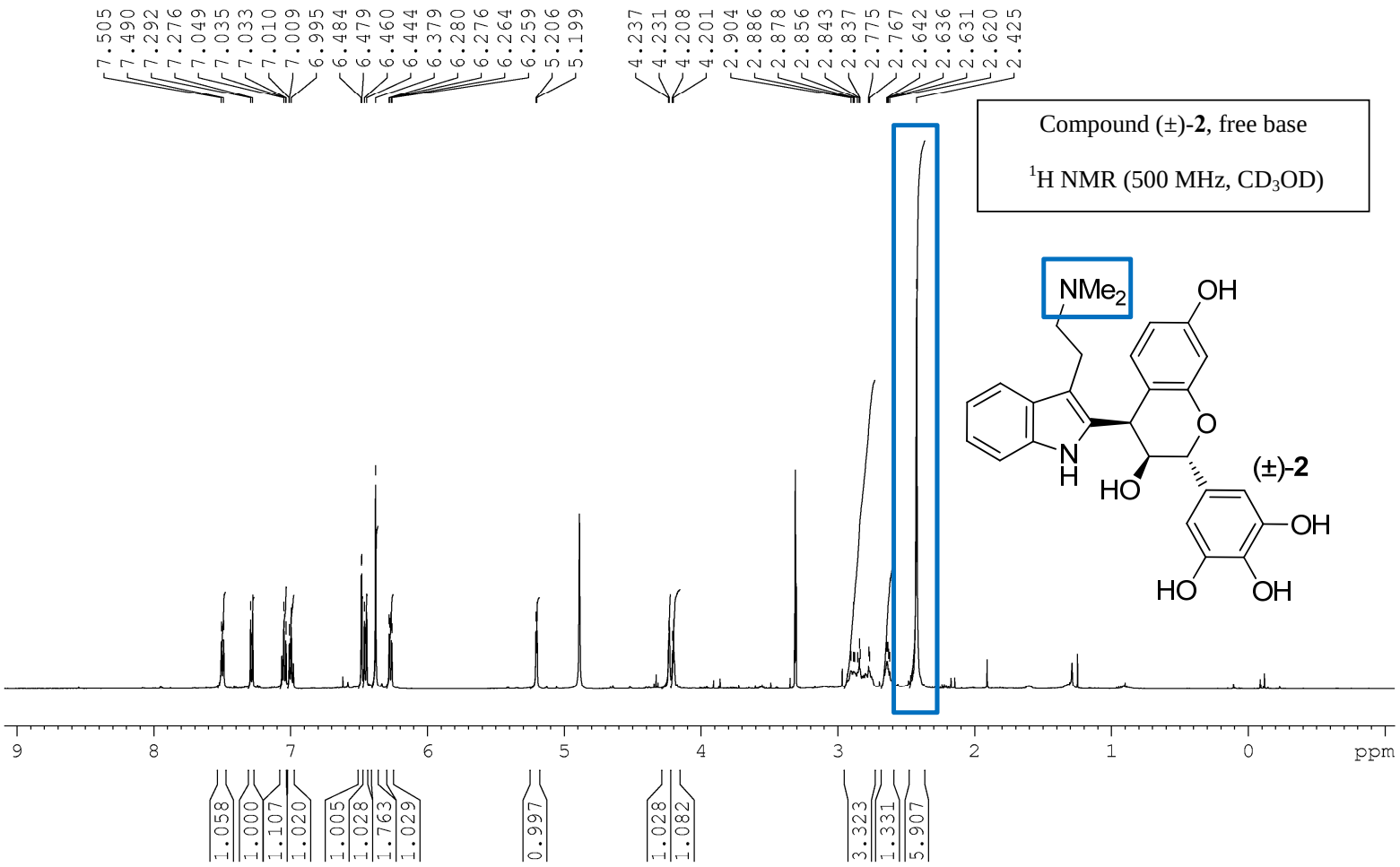


Compound (±)-**8**

^{13}C NMR (125 MHz, $CDCl_3$)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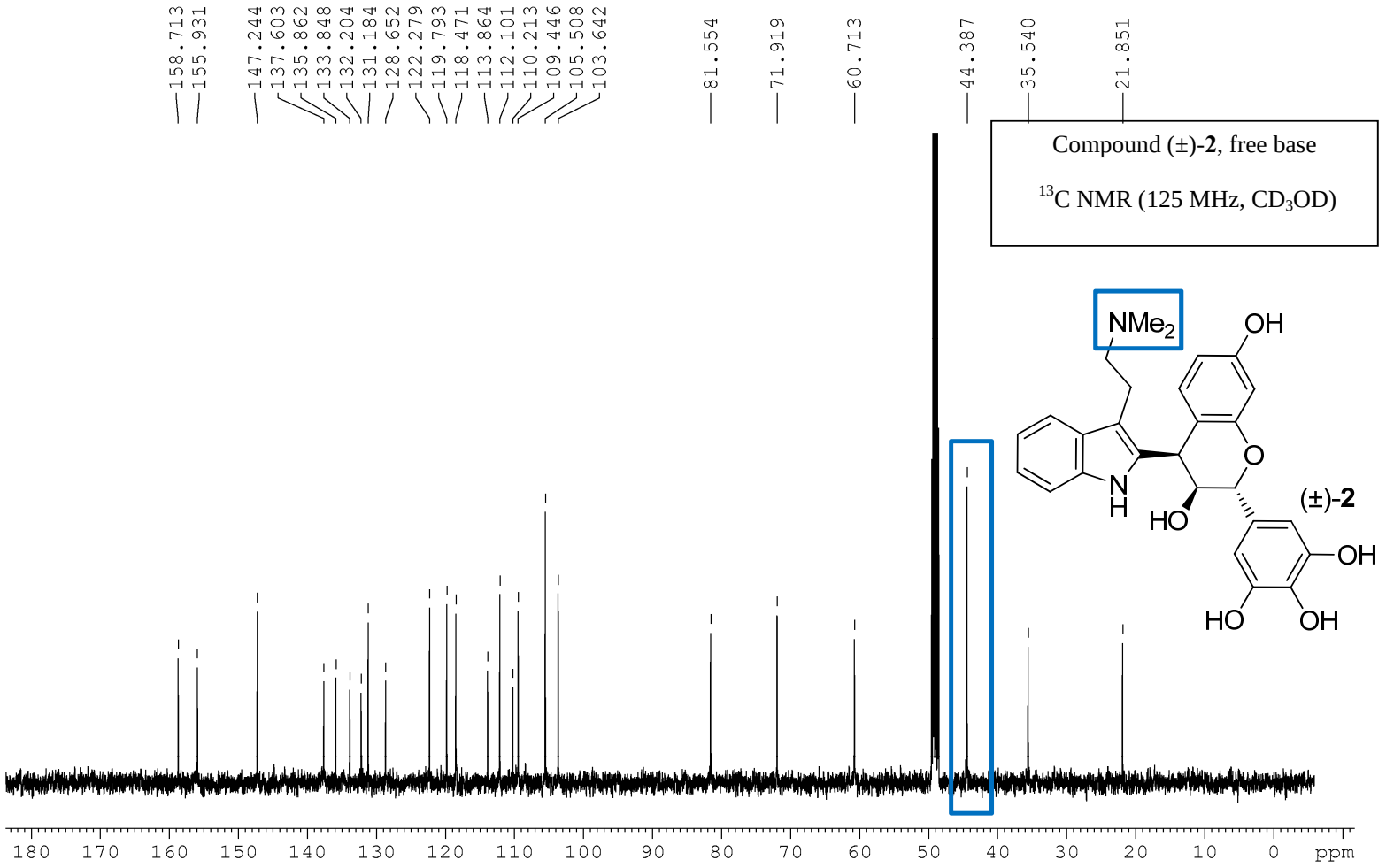
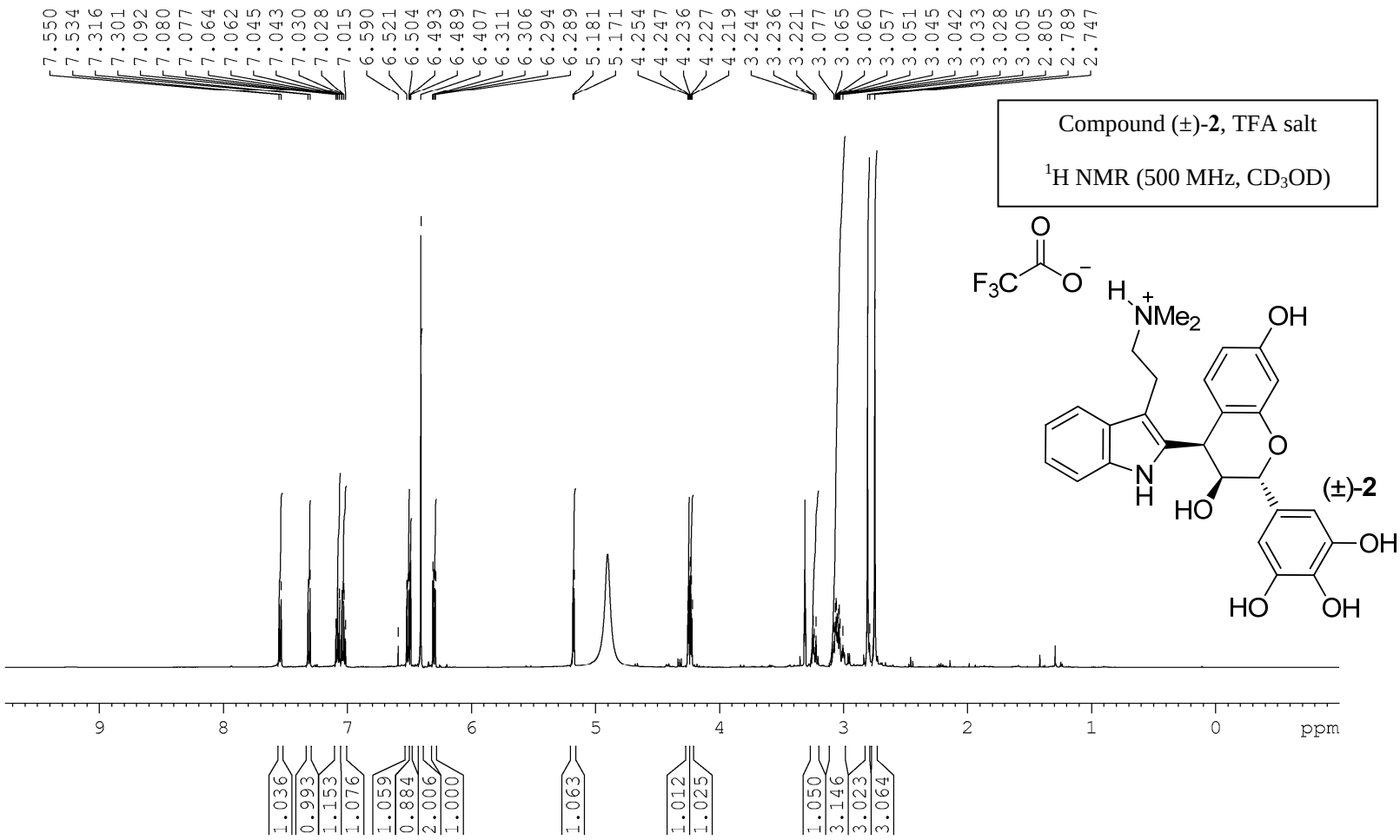
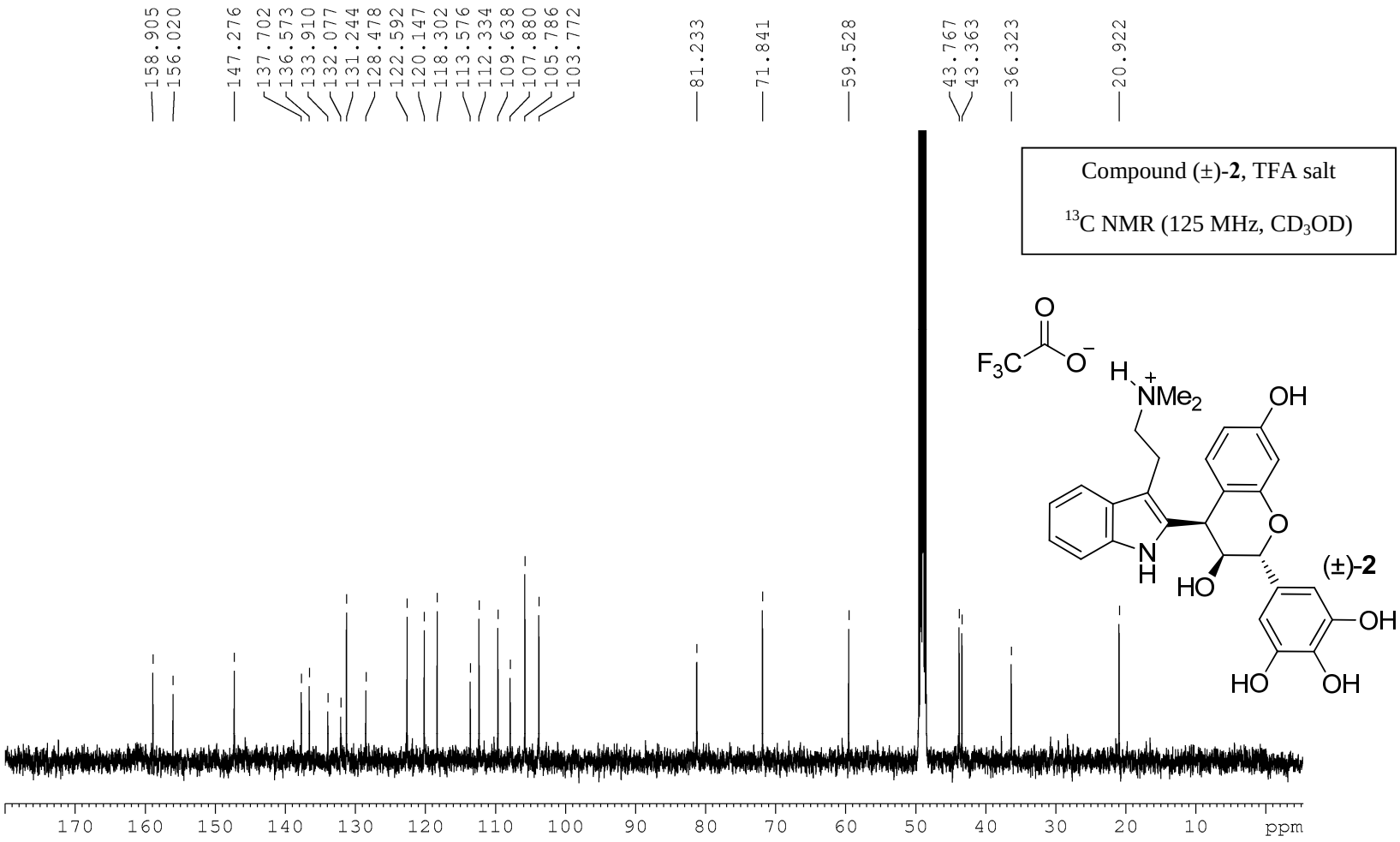
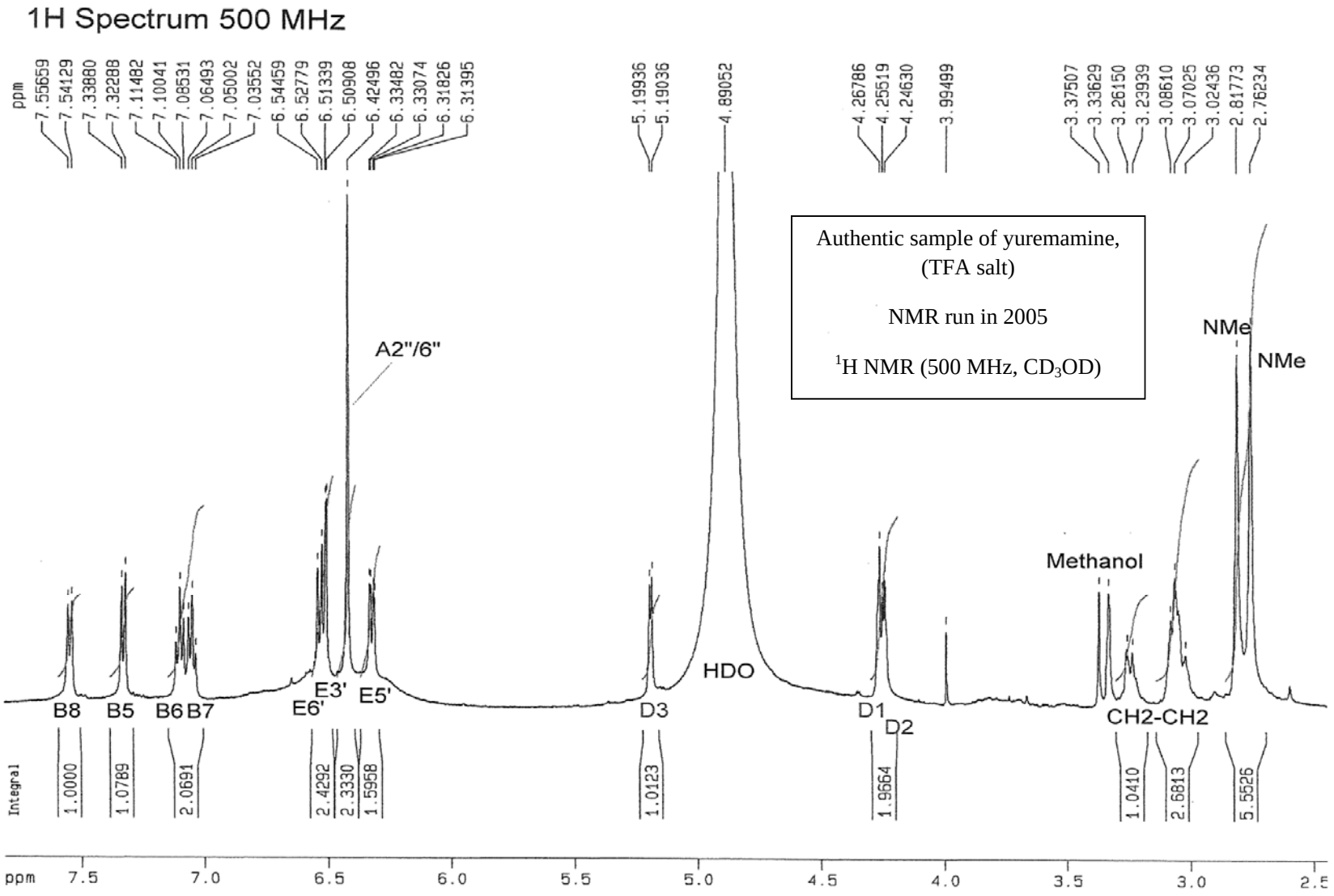
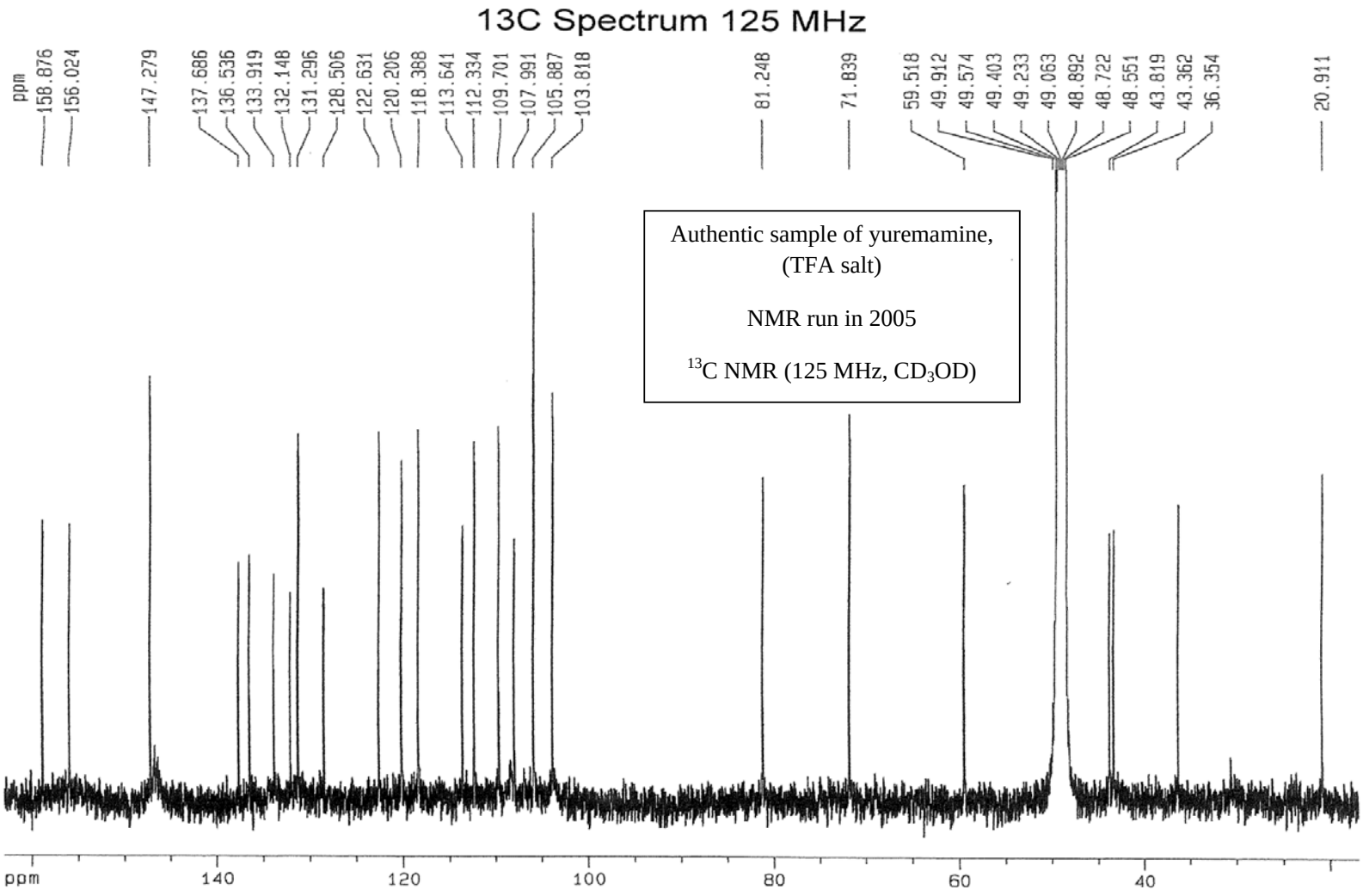
6.460
6.444
6.379
6.280
6.276
6.264
6.259
5.206
5.199
4.237
4.231
4.208
4.201
2.904
2.886
2.878
2.856
2.843
2.837
2.775
2.767
2.642
2.636
2.631
2.620
2.425
Compound (±)-2, free base
1H NMR (500 MHz, CD3OD)
NMe2
OH
O
N
H
HO
(±)-2
OH
HO
OH
9
8
7
6
5
4
3
2
1
0
ppm
1.058
1.000
1.107
1.020
1.005
1.028
1.763
1.029
0.997
1.028
1.082
3.323
1.331
5.907

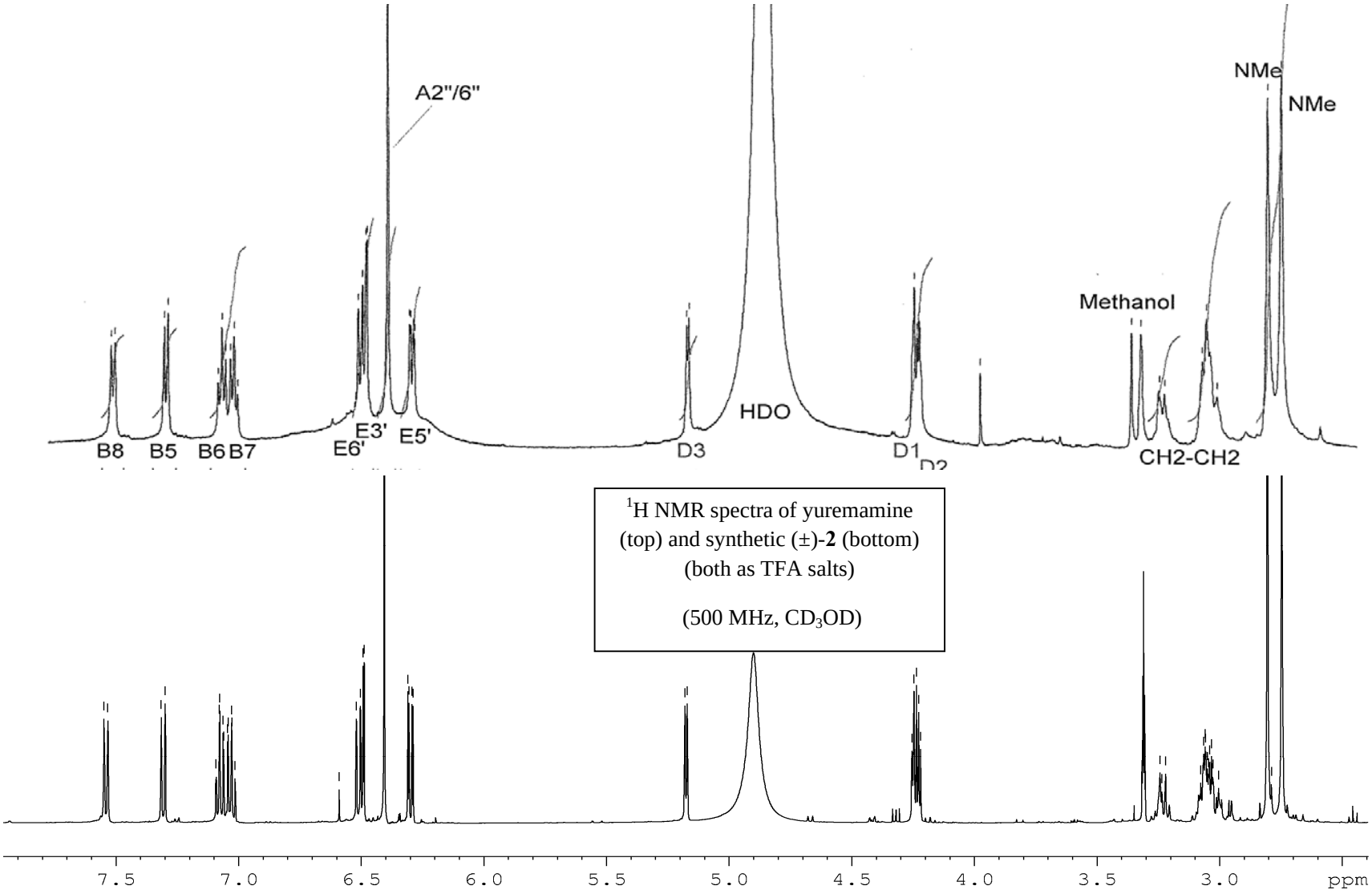

158.713
155.931
147.244
137.603
135.862
133.848
132.204
131.184
128.652
122.279
119.793
118.471
113.864
112.101
110.213
109.446
105.508
103.642
81.554
71.919
60.713
44.387
35.540
21.851
Compound (±)-2, free base
13C NMR (125 MHz, CD3OD)
NMe2
OH
O
N
H
HO
(±)-2
OH
HO
OH
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.550
7.534
7.316
7.301
7.092
7.080
7.077
7.064
7.062
7.045
7.043
7.030
7.028
7.015
6.590
6.521
6.504
6.493
6.489
6.407
6.311
6.306
6.294
6.289
5.181
5.171
4.254
4.247
4.236
4.227
4.219
3.244
3.236
3.221
3.077
3.065
3.060
3.057
3.051
3.045
3.042
3.033
3.028
3.005
2.805
2.789
2.747
Compound (±)-2, TFA salt
1H NMR (500 MHz, CD3OD)
(±)-2
ppm
1.036
0.993
1.153
1.076
1.059
0.884
2.006
1.000
1.063
1.012
1.025
1.050
3.146
3.023
3.064


158.905
156.020
147.276
137.702
136.573
133.910
132.077
131.244
128.478
122.592
120.147
118.302
113.576
112.334
109.638
107.880
105.786
103.772
81.233
71.841
59.528
43.767
43.363
36.323
20.922
Compound (±)-2, TFA salt
13C NMR (125 MHz, CD3OD)
(±)-2
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

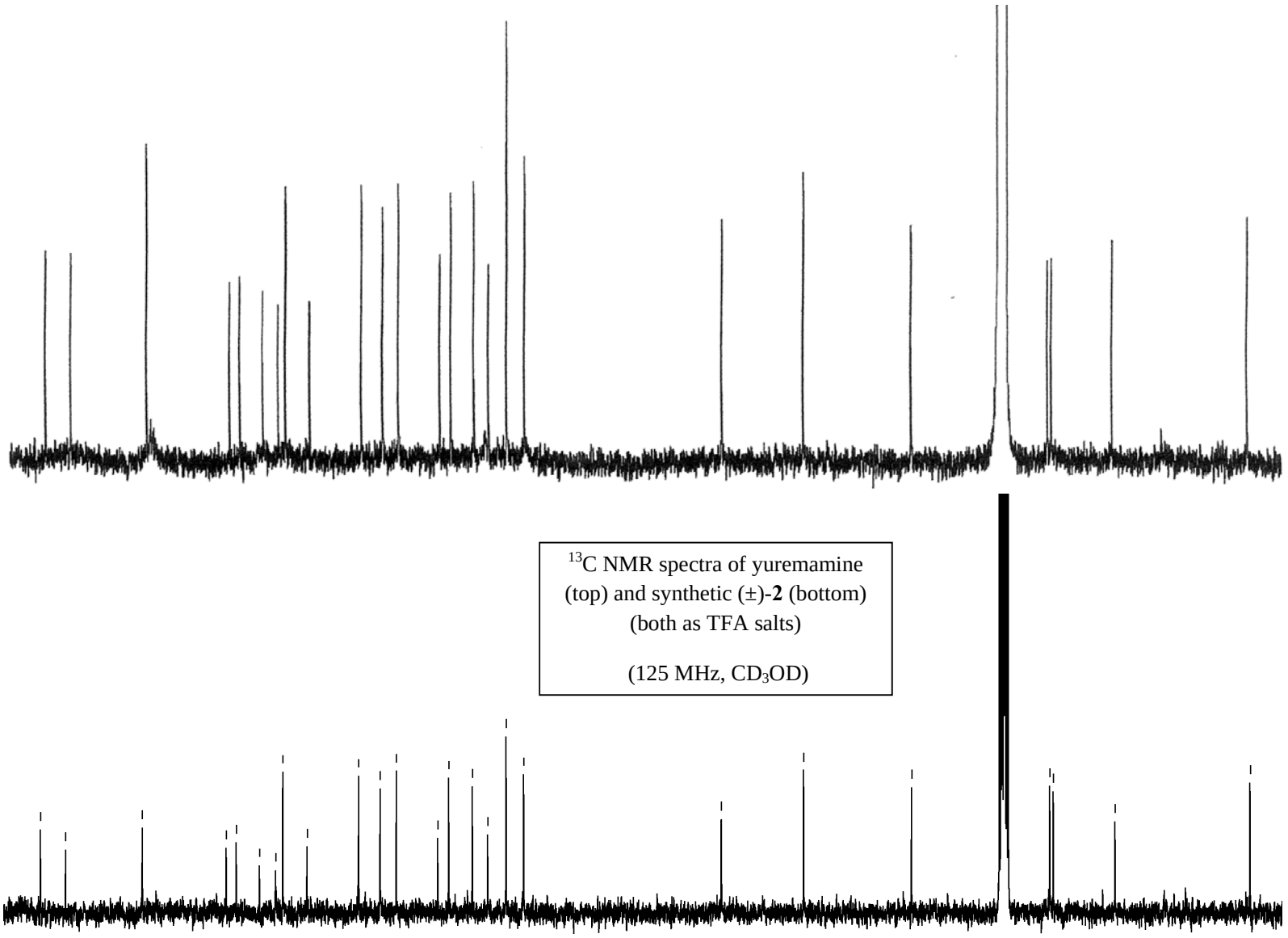

1H Spectrum 500 MHz
ppm
7.55659
7.54129
7.33880
7.32288
7.11482
7.10041
7.08531
7.06493
7.05002
7.03552
6.54459
6.52779
6.51339
6.50908
6.42496
6.33482
6.33074
6.31826
6.31395
5.19936
5.19036
4.89052
4.26786
4.25519
4.24630
3.99499
3.37507
3.33629
3.26150
3.23939
3.08610
3.07025
3.02436
2.81773
2.76234
Authentic sample of yuremamine, (TFA salt)
NMR run in 2005
1H NMR (500 MHz, CD3OD)
A2"/6"
NMe
NMe
Methanol
HDO
B8
B5
B6 B7
E6'
E3'
E5'
D3
D1
D2
CH2-CH2
Integral
1.0000
1.0789
2.0691
2.4292
2.3330
1.5958
1.0123
1.9664
1.0410
2.6813
5.5526
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0


13C Spectrum 125 MHz
ppm
158.876
156.024
147.279
137.686
136.536
133.919
132.148
131.296
128.506
122.631
120.206
118.388
113.641
112.334
109.701
107.991
105.887
103.818
81.248
71.839
59.518
49.912
49.574
49.403
49.233
49.063
48.892
48.722
48.551
43.819
43.362
36.354
20.911
Authentic sample of yuremamine, (TFA salt)
NMR run in 2005
^{13}C NMR (125 MHz, CD_3OD)
ppm
140
120
100
80
60
40



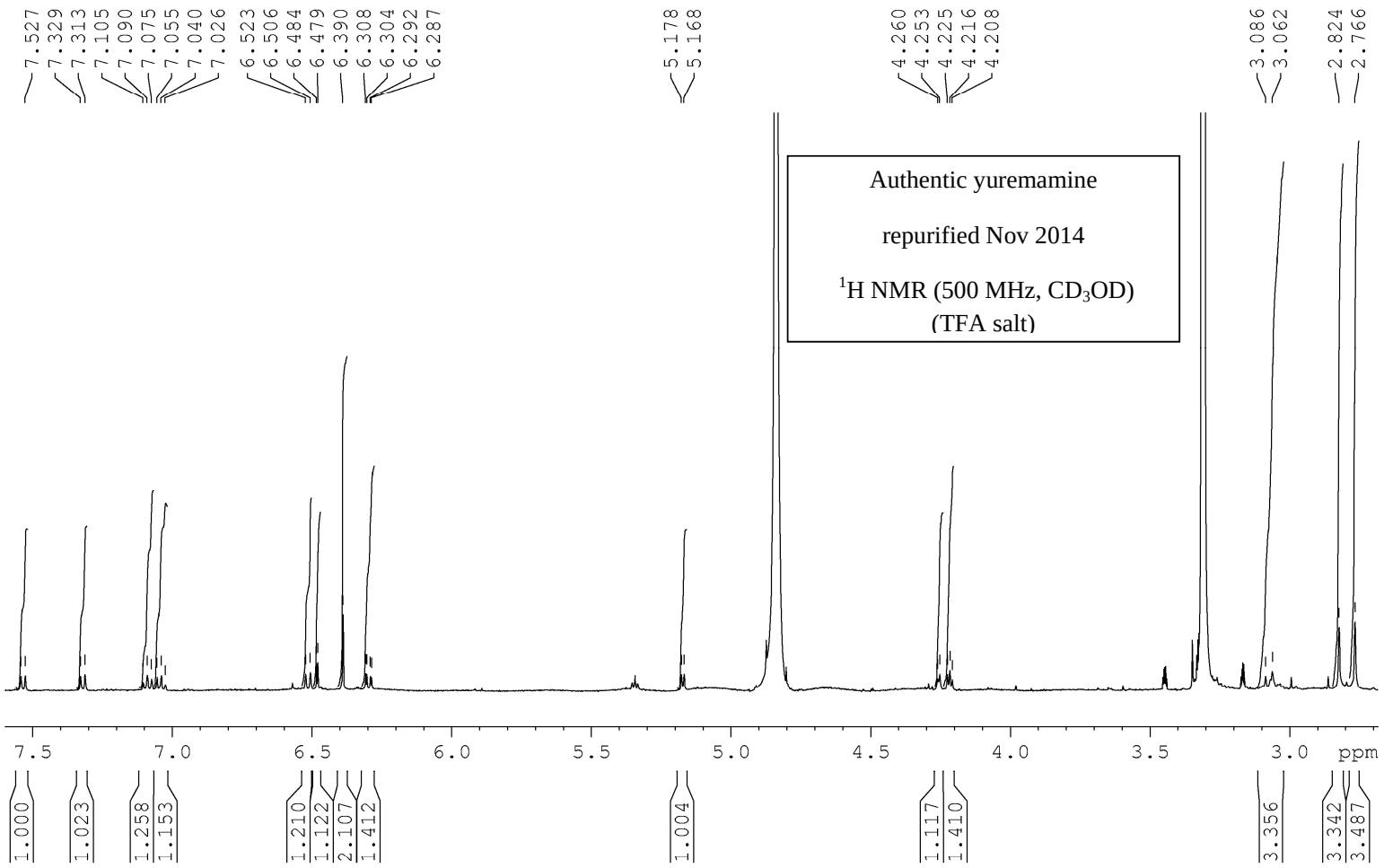
^{1}H NMR spectra of yuremamine (top) and synthetic (±)-**2** (bottom) (both as TFA salts)

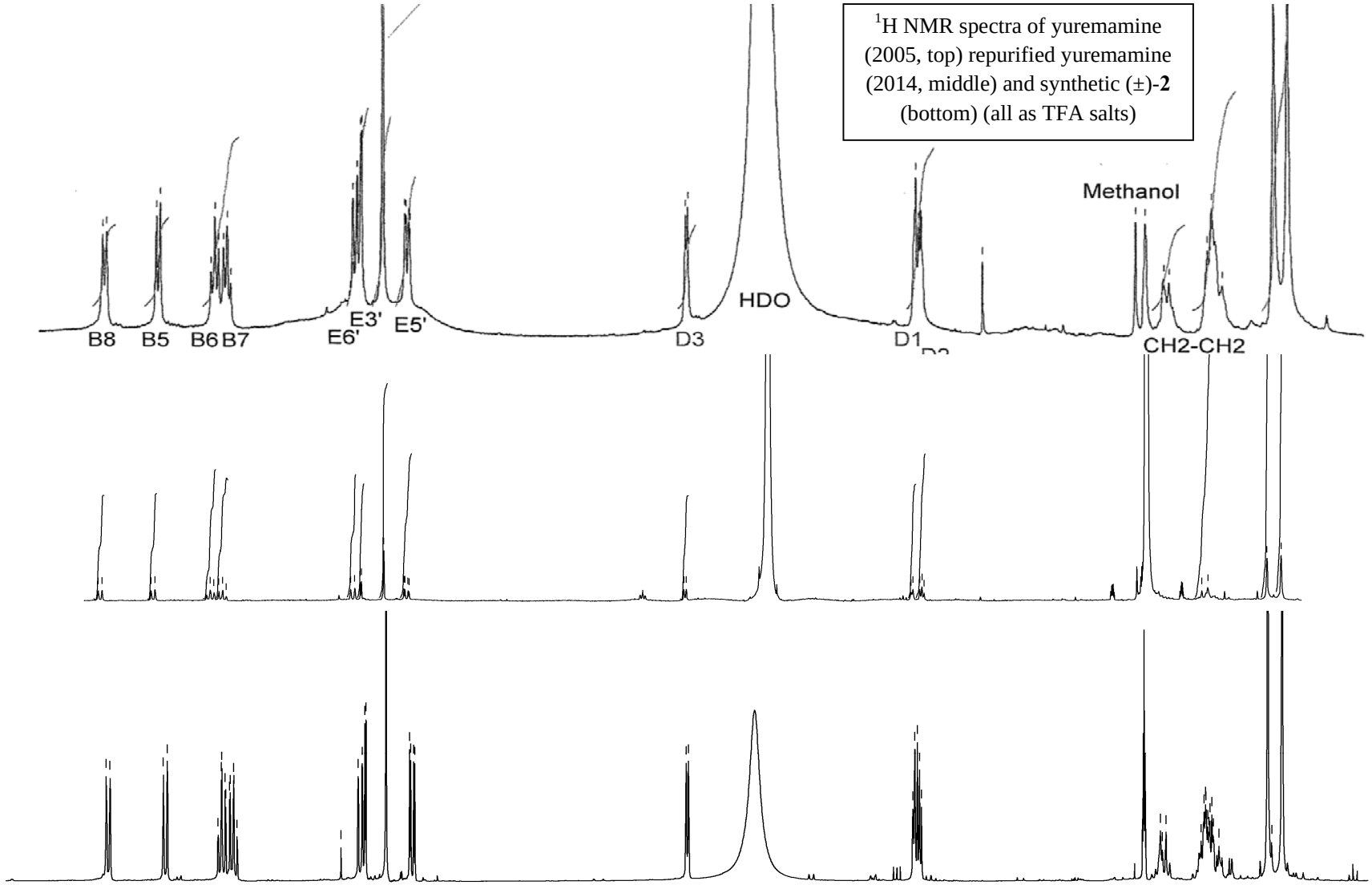
(500 MHz, CD_3OD)



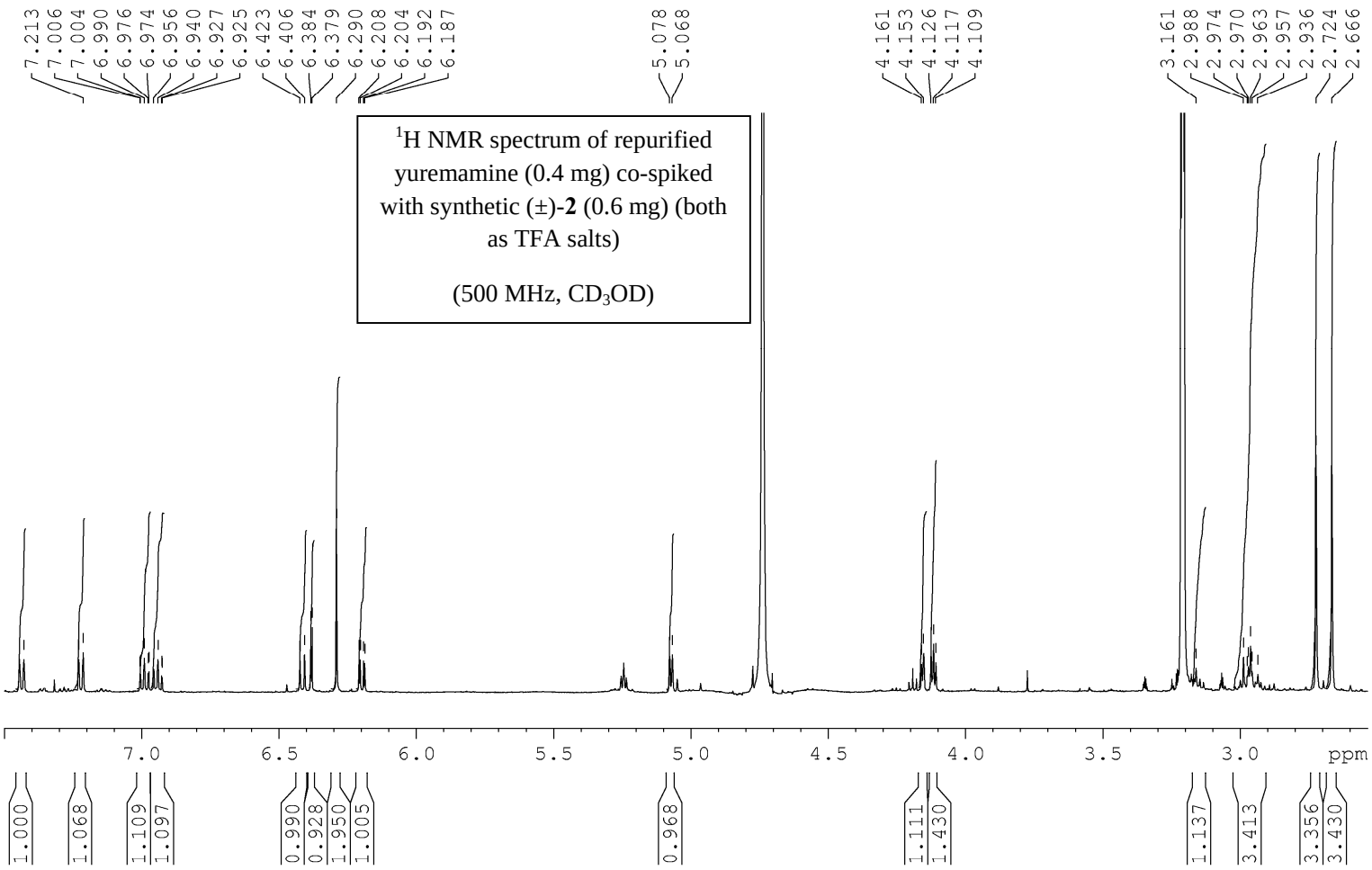
^{13}C NMR spectra of yuremamine (top) and synthetic (±)-**2** (bottom) (both as TFA salts)

(125 MHz, CD_3OD)


7.527
7.329
7.313
7.105
7.090
7.075
7.055
7.040
7.026
6.523
6.506
6.484
6.479
6.390
6.308
6.304
6.292
6.287
5.178
5.168
4.260
4.253
4.225
4.216
4.208
3.086
3.062
2.824
2.766
Authentic yuremamine
repurified Nov 2014
1H NMR (500 MHz, CD3OD)
(TFA salt)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
1.000
1.023
1.258
1.153
1.210
1.122
2.107
1.412
1.004
1.117
1.410
3.356
3.342
3.487

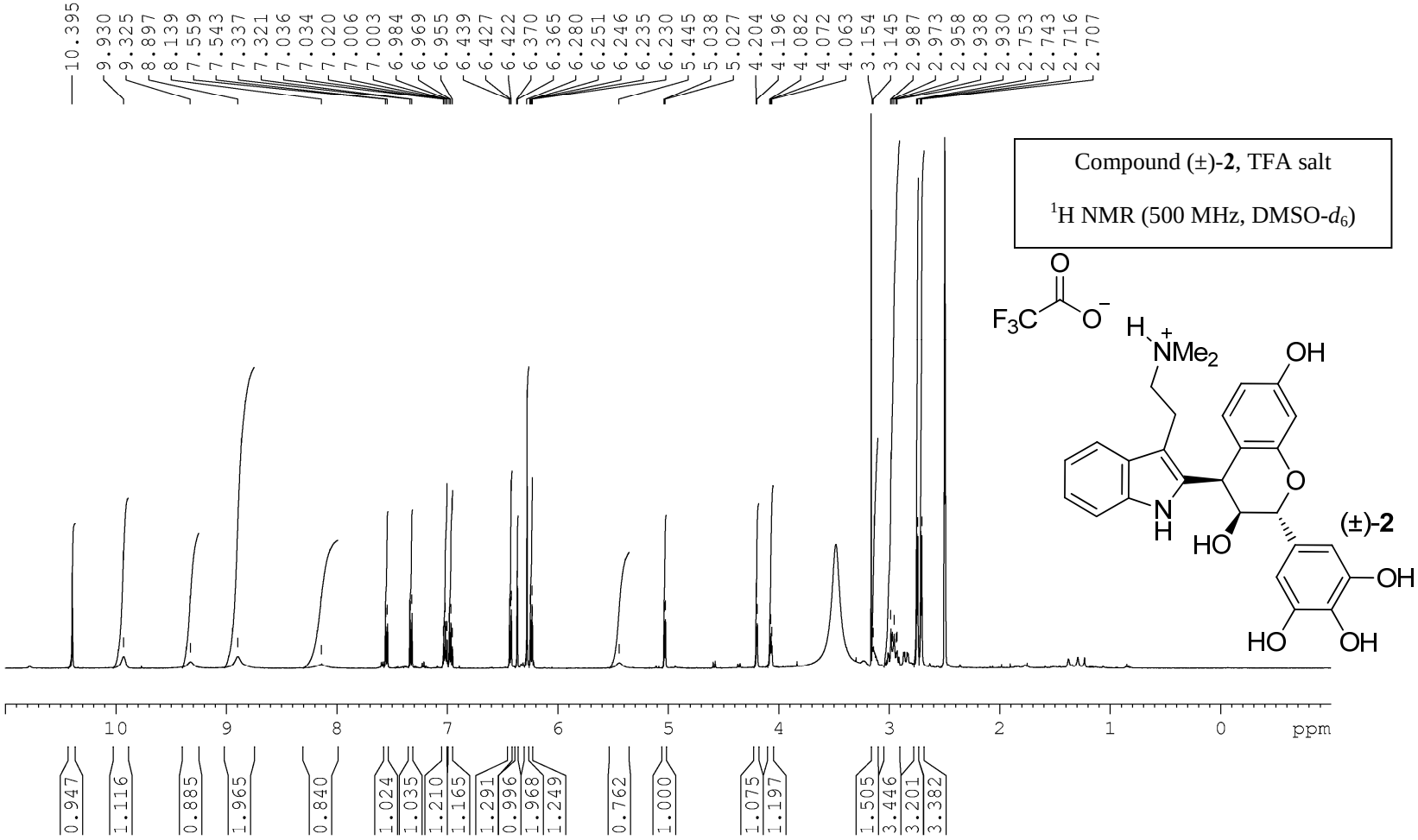


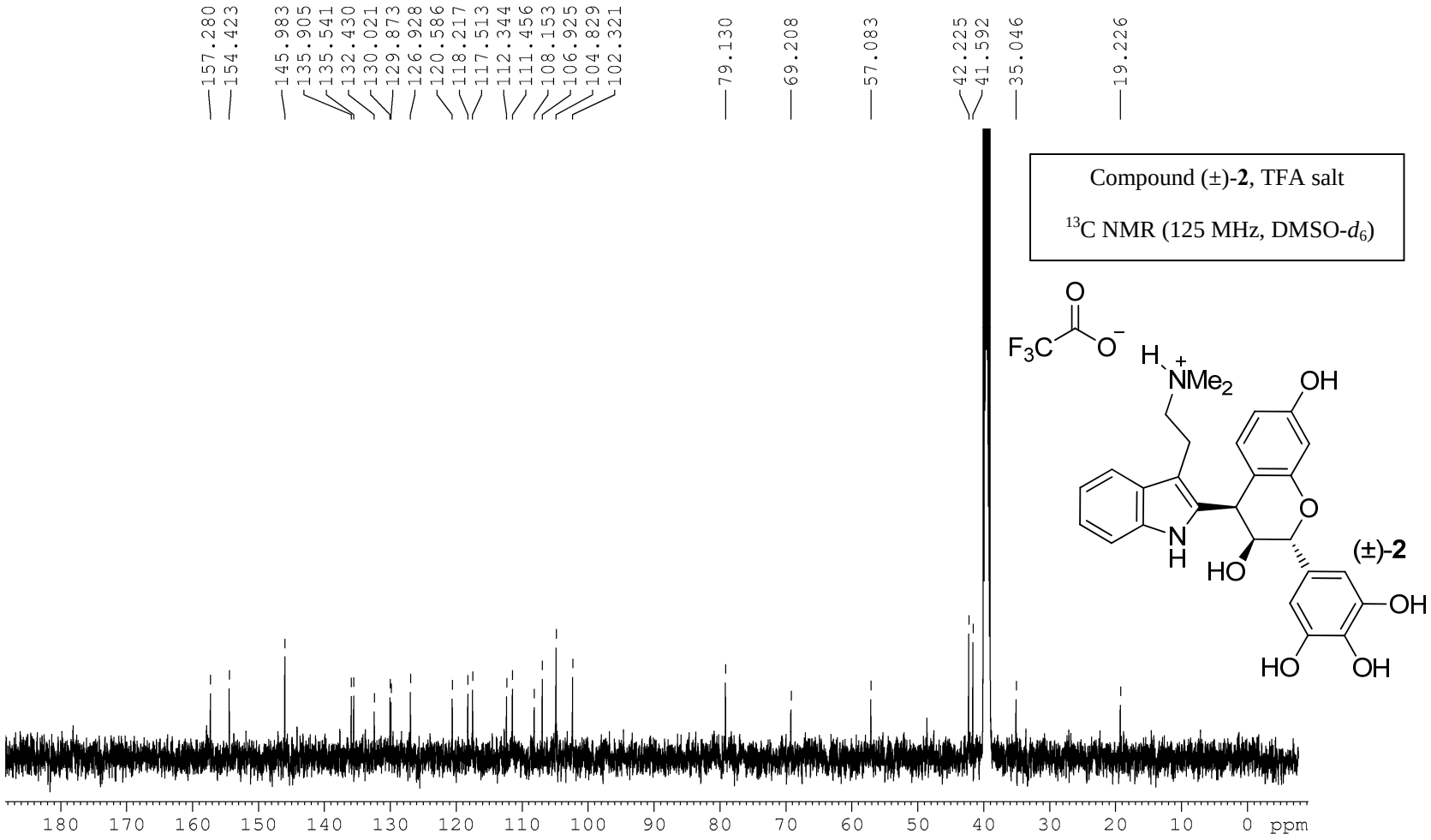
^{1}H NMR spectra of yuremamine (2005, top) repurified yuremamine (2014, middle) and synthetic (±)-**2** (bottom) (all as TFA salts)

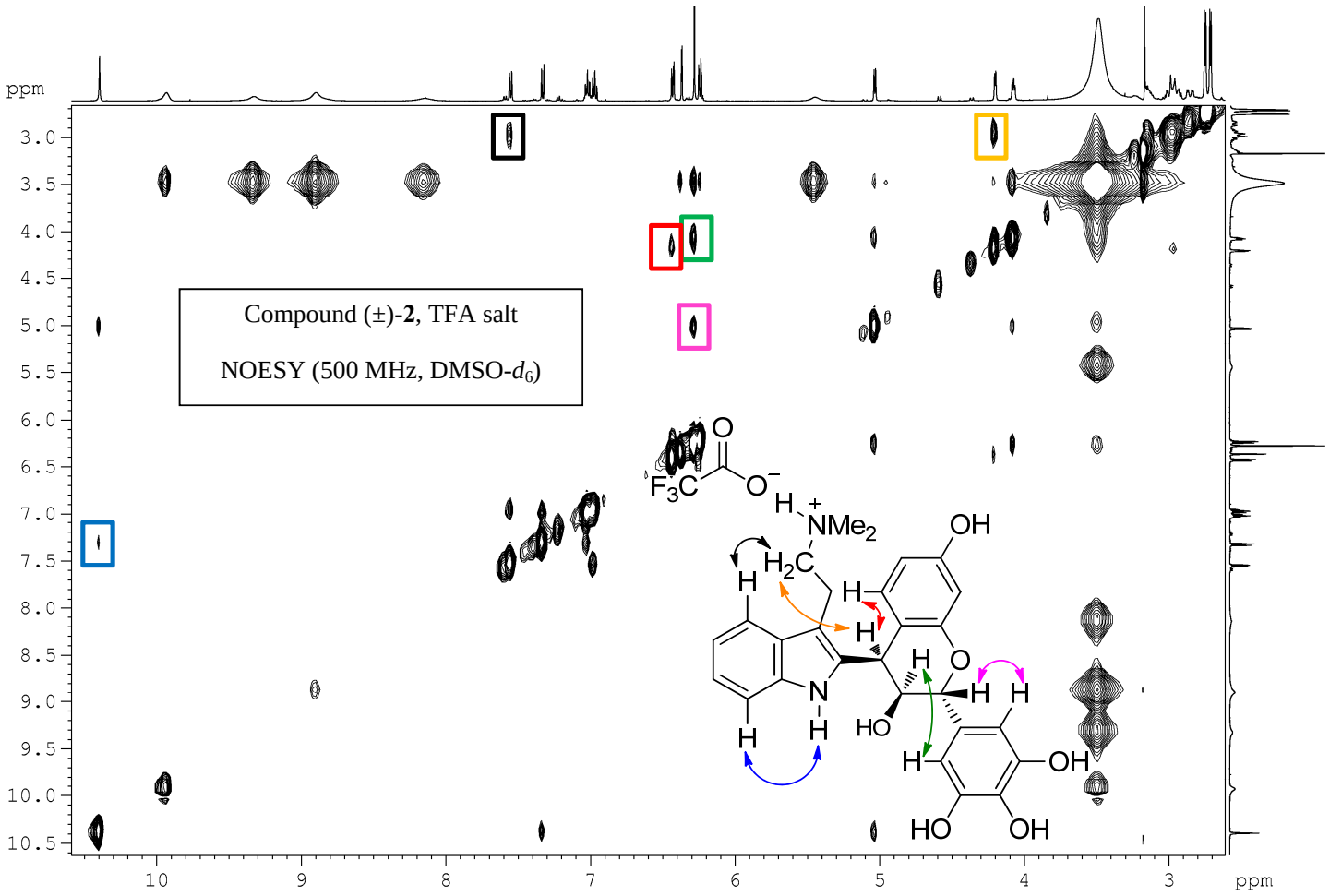


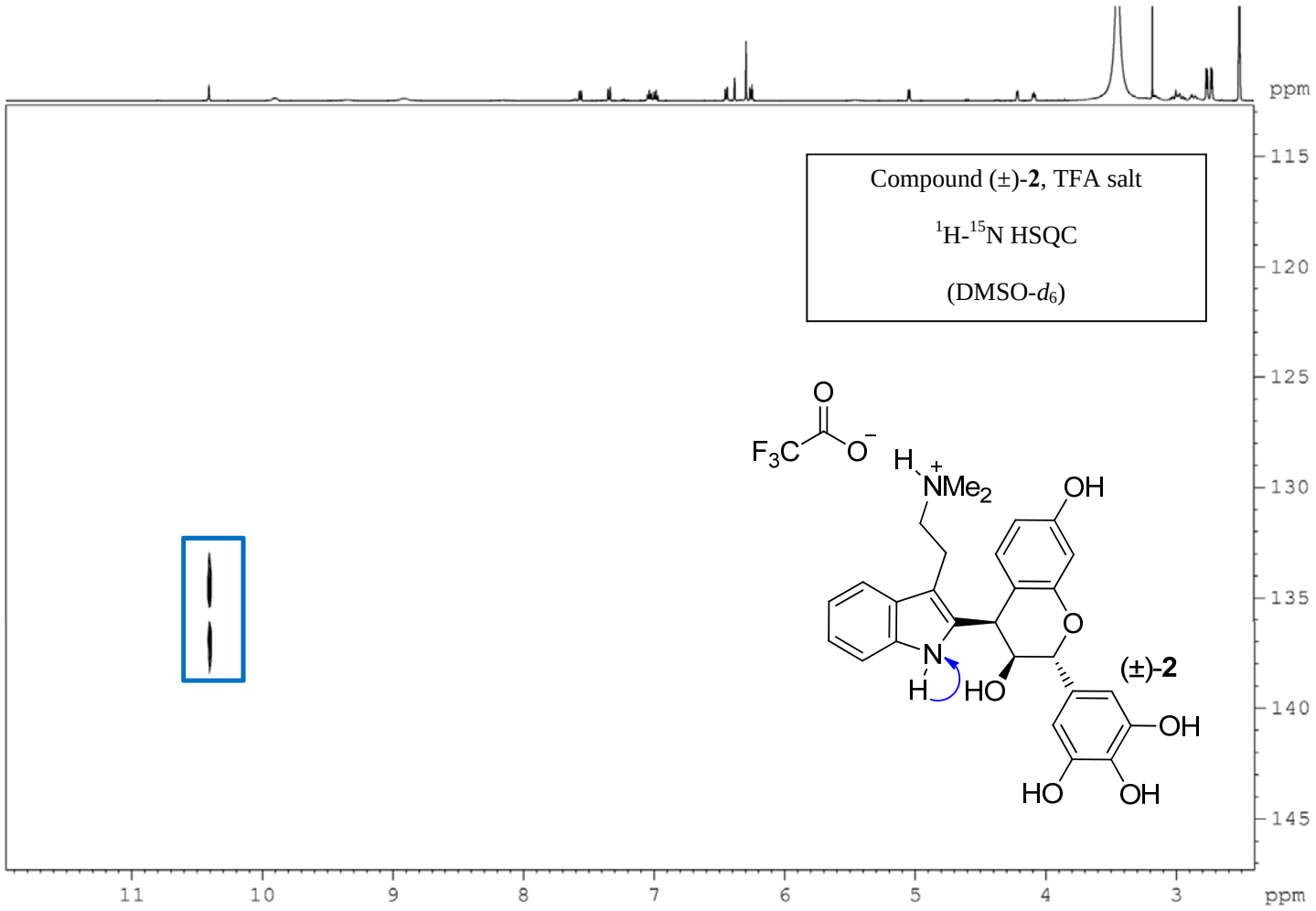
^{1}H NMR spectrum of repurified yuremamine (0.4 mg) co-spiked with synthetic (±)-**2** (0.6 mg) (both as TFA salts)

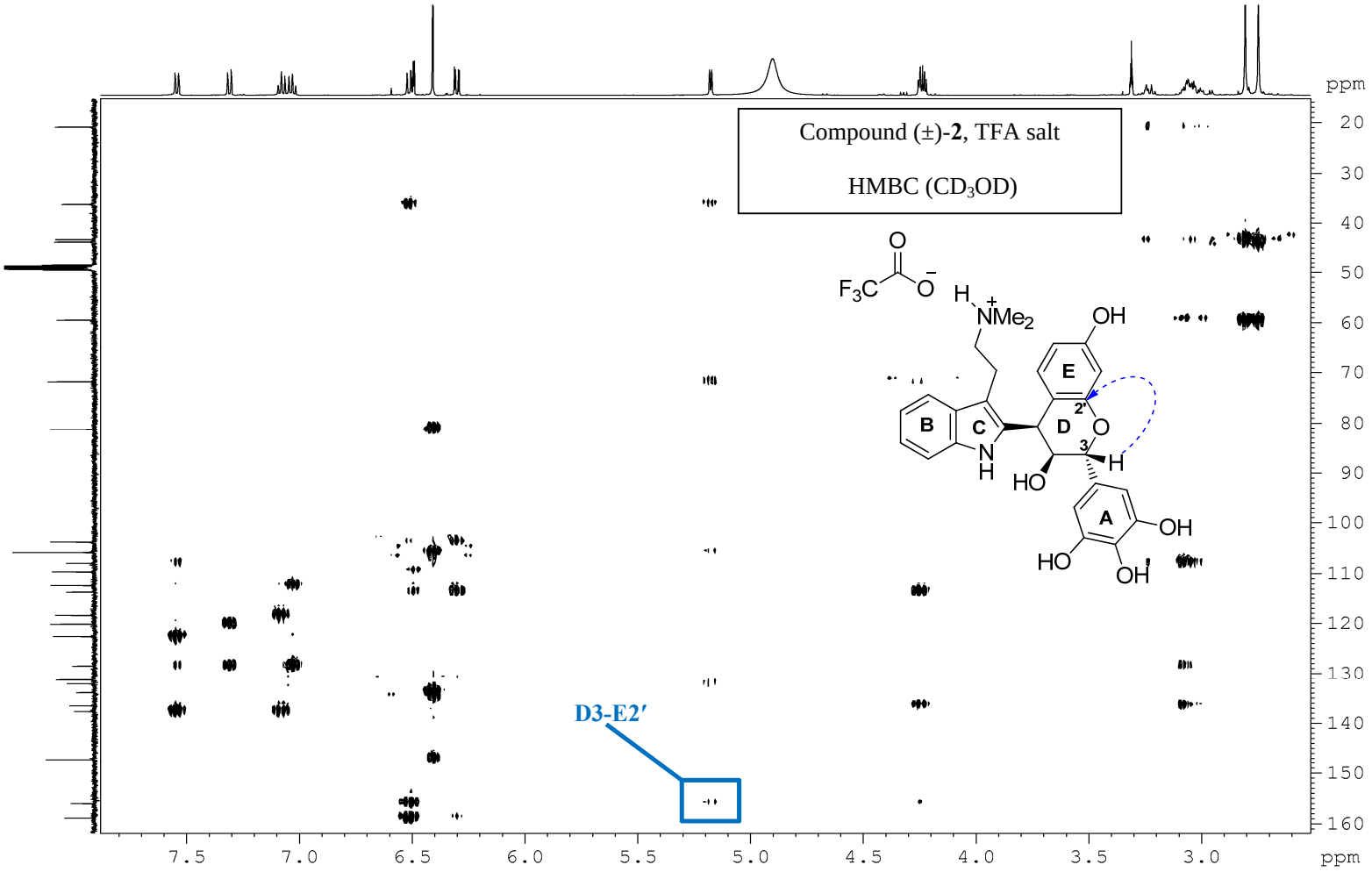
(500 MHz, CD_3OD)


10.395
9.930
9.325
8.897
8.139
7.559
7.543
7.337
7.321
7.036
7.034
7.020
7.006
7.003
6.984
6.969
6.955
6.439
6.427
6.422
6.370
6.365
6.280
6.251
6.246
6.235
6.230
5.445
5.038
5.027
4.204
4.196
4.082
4.072
4.063
3.154
3.145
2.987
2.973
2.958
2.938
2.930
2.753
2.743
2.716
2.707
Compound (±)-2, TFA salt
1H NMR (500 MHz, DMSO-d6)
(±)-2
ppm
0.947
1.116
0.885
1.965
0.840
1.024
1.035
1.210
1.165
1.291
0.996
1.968
1.249
0.762
1.000
1.075
1.197
1.505
3.446
3.201
3.382

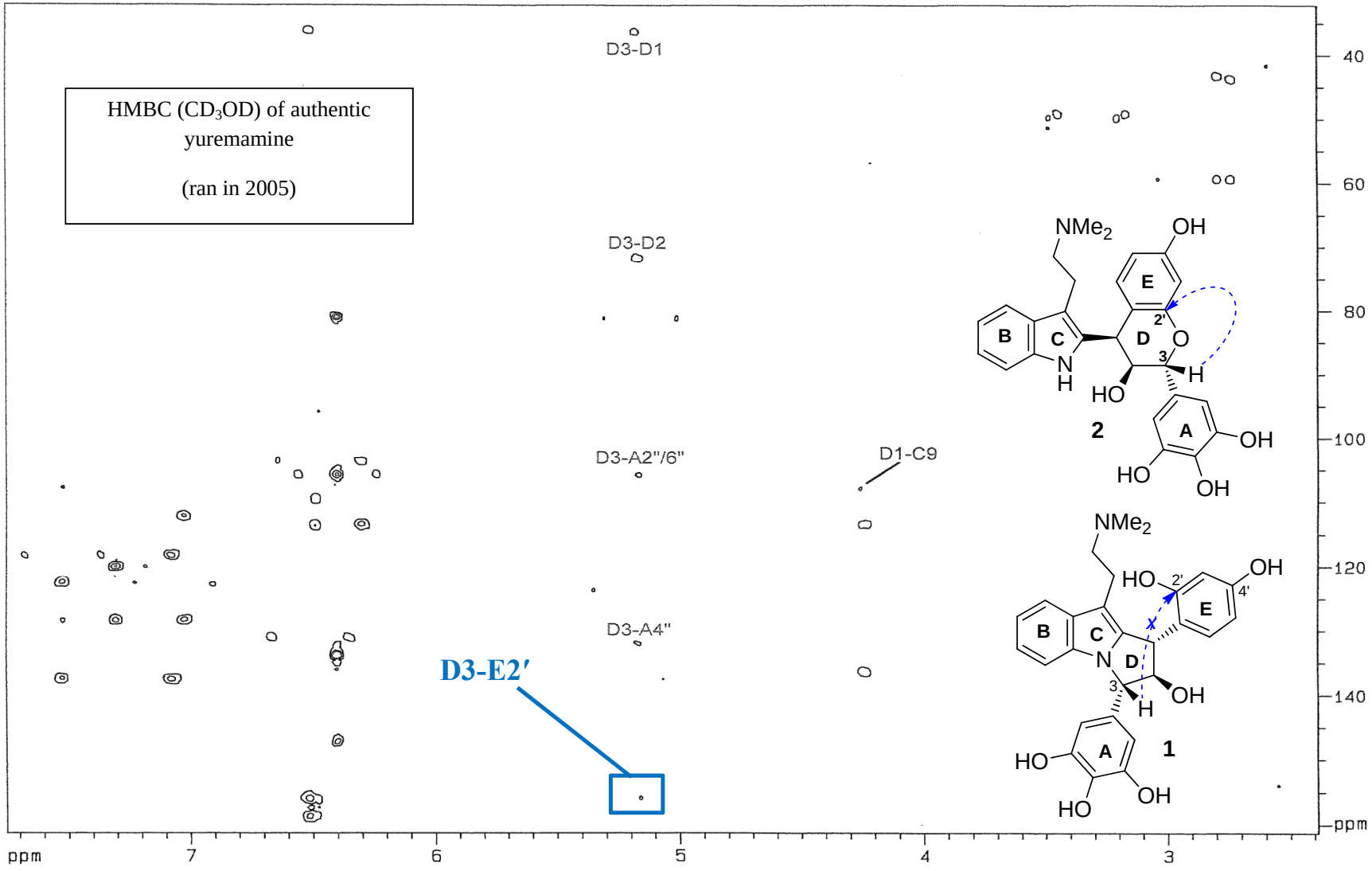

157.280
154.423
145.983
135.905
135.541
132.430
130.021
129.873
126.928
120.586
118.217
117.513
112.344
111.456
108.153
106.925
104.829
102.321
79.130
69.208
57.083
42.225
41.592
35.046
19.226
Compound (±)-2, TFA salt
13C NMR (125 MHz, DMSO-d6)
(±)-2
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

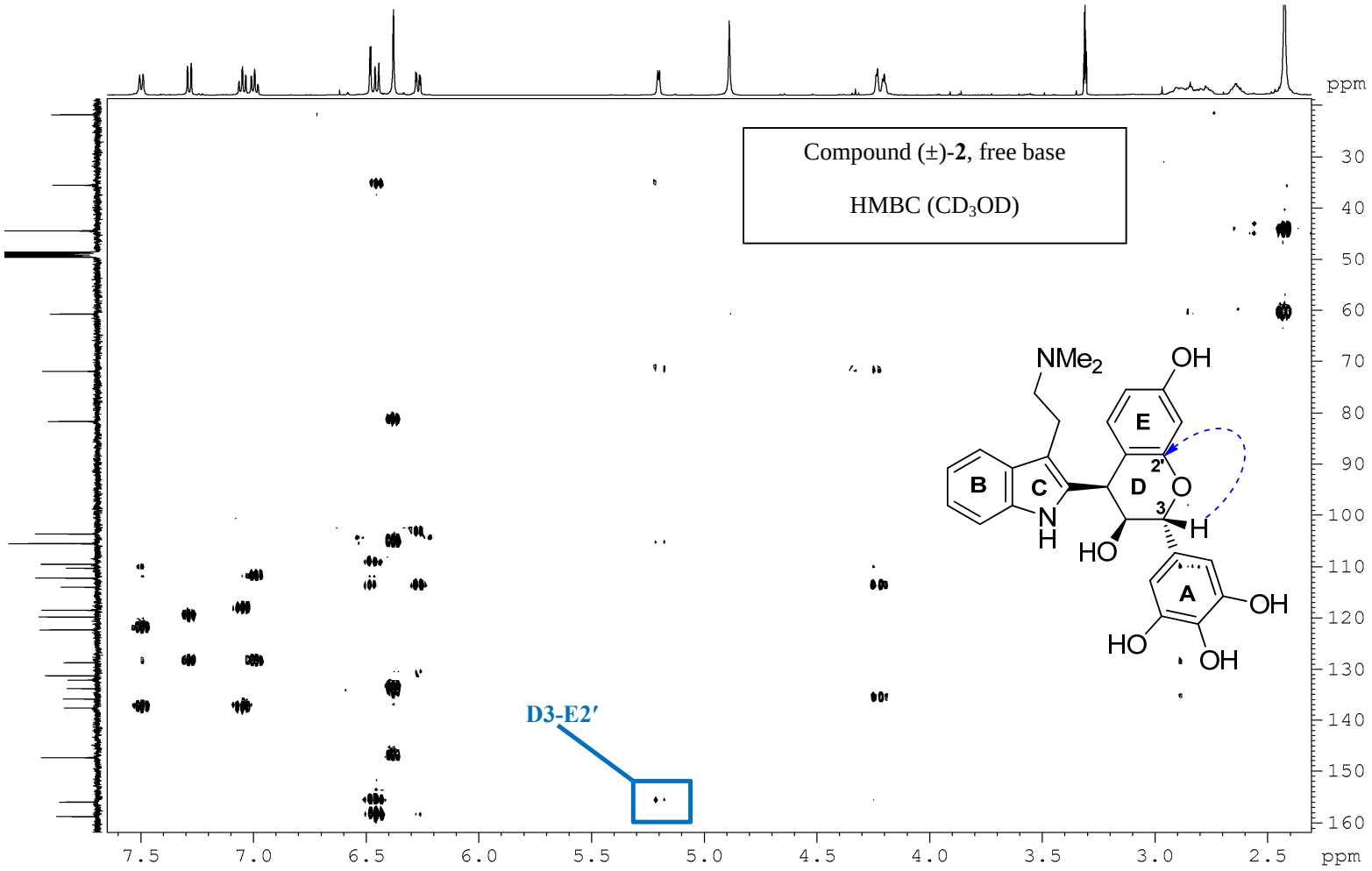

Compound (±)-2, TFA salt
NOESY (500 MHz, DMSO-d_6)
ppm
ppm


ppm
115
120
125
130
135
140
145
11
10
9
8
7
6
5
4
3
ppm
Compound (±)-2, TFA salt
^{1}H-^{15}N HSQC
(DMSO-d_6)
O
F3C
O
H
NMe2
OH
O
N
H
HO
(±)-2
OH
HO
OH


Compound (±)-2, TFA salt
HMBC (CD3OD)
D3-E2′
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
A
B
C
D
E
NMe2
OH
HO
2′
3

Long range 1H-13C correlated spectrum




Compound (±)-2, free base
HMBC (CD3OD)
D3-E2′
NMe2
OH
E
B
C
D
O
2′
3
N
H
HO
H
A
OH
HO
OH
ppm
30
40
50
60
70
80
90
100
110
120
130
140
150
160
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm