Electronic Supplementary Material (ESI) for ChemComm.

SUPPORTING INFORMATION

Mo(CO)6 Catalysed Chemoselective Hydrosilylation of α,β-Unsaturated Amides for the Formation of Allylamines

Alexey Volkov,[a] Fredrik Tinnis,*[a] Tove Slagbrand,[a] Ida Pershagen[a] and Hans Adolfsson*[a]

[a] Department of Organic Chemistry,

Stockholm University, the Arrhenius Laboratory, SE-106 91 Stockholm, Sweden

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Instrumentation

Characterizations were made by ^{1}H and ^{13}C NMR spectroscopy. NMR spectra were recorded at Bruker 400, 500 MHz (^{1}H) and 100, 125 MHz (^{13}C), and were referenced internally with $CDCl_3$ (δH 7.26, δC 77.16 ppm) $(CD_3)_2SO$ (δH 2.50, δC 39.52 ppm). High temperature experiments were performed at Bruker 500 MHz (^{1}H) and 125 MHz (^{13}C). HRMS was performed on Bruker micrOTOF/ESI.

Material

Unless otherwise noted, materials were purchased from commercial suppliers and were used without purification. $Mo(CO)_6$, sublimed 99,9+% was purchased from Sigma-Aldrich and used as received. THF was purchased from Fischer Scientific, and dispersed from a solvent drying system.

General

The 1 mmol scale catalytic reduction of amides was performed in microwave tubes 2-5 mL from Biotage, with a Teflon-coated magnetic stirring bar. The tubes were fitted with a cap containing a septum and the reactions were run under nitrogen atmosphere.

Substrate scope investigation

General procedure for catalytic reduction of amides.

Amide (1.0 mmol) and $Mo(CO)_6$ (0.0132 g, 0.05 mmol) were added to an oven dried 10 mL microwave tube equipped with a magnetic stirring bar. To the sealed tube, dry THF (2 mL) and TMDS (0.265 mL, 1.5 mmol) were added and the reaction mixture was stirred at 65 °C for 24 h. The reaction was quenched with NaOH (Aq. 2M, 10 mL) and the stirring was continued at r.t for 8 h. The mixture was extracted with DCM (3 x 20 mL), dried with Na_2SO_4 and evaporated under reduced pressure. The crude products were purified by column chromatography.

Evaluation of β,γ-unsaturated amide 7

Amide **7** (1.0 mmol) and $Mo(CO)_6$ (0.0132 g, 0.05 mmol) were added to an oven dried 10 mL microwave tube equipped with a magnetic stirring bar. To the sealed tube, dry THF (2 mL) and TMDS (0.265 mL, 1.5 mmol) were added and the reaction mixture was stirred at 65 °C for 24 h. The solvent was evaporated and 1,3,5-trimethoxybenzene (0.056 g, 0.33 mmol) was added as internal standard. The mixture was dissolved in $CDCl_3$ (3 mL) where after the ^1H NMR spectrum was immediately recorded.

Synthesis of Naftifine (14)

Synthesis of N-(naphthalen-1-ylmethyl)cinnamamide (12)

Dry THF (17 mL) was added to the carboxylic acid **10** (0.741 g, 5.0 mmol), activated molecular sieves 4Å (2.5 g) and zirconium(IV)chloride (0.118 g, 10 mol%) under nitrogen atmosphere and the mixture was heated under stirring to 100ºC in a capped microwave vial. The amine **11** (0.943 g, 6.0 mmol) was added dropwise and the reaction was stirred at the same temperature for 24 h and then cooled to r.t. The mixture was filtered through a plug of silica (4 x 3.5 cm) with 150 mL of an EtOAc:Et_3N (200:1) eluent. The solvent was removed under reduced pressure affording analytically pure compound **12** (1.306 g, 91 %).

Synthesis of N-methyl-N-(naphthalen-1-ylmethyl)cinnamamide (13)

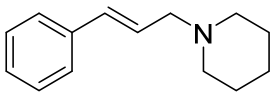
In 25 mL round-bottom flask to a suspension of NaH (0.12 g, 5.0 mmol) in 10 mL of dry DMF 1 g (3.5 mmol) of amide (**12**) was added drop wise as a solution in 2 mL of DMF at 0 ˚C and the reaction mixture was stirred for 2 h. 0.36 mL (5,9 mmol) of methyl iodide was thereafter added drop wise and the temperature was raised to r.t and the mixture was left stirring for 10 h. The reaction was quenched with 2 mL of 95% ethanol followed by water (40 mL). The reaction was then extracted with EtOAc (3x40 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Crude product was purified by column chromatography using pentane/ethyl acetate (4:1) as an eluent yielding 0.976 g (93 %) of the target compound (**13**).

Synthesis of (E)-N-methyl-N-(naphthalen-1-ylmethyl)-3-phenylprop-2-en-1-amine (Naftifine) (14)

Amide **13** was reduced following general procedure for catalytic reduction of amides.

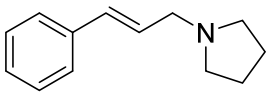
Compound characterization.

1-cinnamylpiperidine 2a



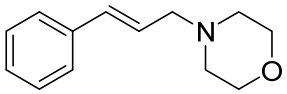
0.176 g, 87 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.39 – 7.36 (m, 2H), 7.32 – 7.28 (m, 2H), 7.24 – 7.19 (m, 1H), 6.50 (d, *J* = 15.8 Hz, 1H), 6. 30 (dt, J_1= 6.7 Hz, J_2 = 15.8 Hz, 1H), 3.12 (dd, J_1 = 1.2 Hz, J_2 = 6.7 Hz, 2H), 2.44 (bs, 4H), 1.65 – 1.57 (m, 4H), 1.49 – 1.40 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 137.2, 132.8, 128.7, 127.5, 127.3, 126.4, 62.0, 54.7, 26.1, 24.5; **HRMS** (ESI, m/z) calcd. for $C_{14}H_{20}N$ $[M + H]^+$ 202.1590, found 202.1585.

1-cinnamylpyrrolidine 2b



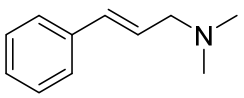
0.167 g, 89 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.39 – 7.36 (m, 2H), 7.33 – 7.28 (m, 2H), 7.24 – 7.19 (m, 1H), 6.53 (d, *J* = 15.9, 1H), 6. 34 (dt, J_1 = 6.7 Hz, J_2 = 15.9 Hz, 1H), 3.26 (dd, J_1 = 1.3 Hz, J_2 = 6.7 Hz, 2H), 2.59 – 2.53 (m, 4H), 1.83 – 1.77 (m, 4H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 137.3, 131.9, 128.7, 127.9, 127.5, 126.4, 58.6, 54.2, 23.6; **HRMS** (ESI, m/z) calcd. for $C_{13}H_{18}N$ $[M + H]^+$ 188.1434, found 188.1433.

4-cinnamylmorpholine 2c



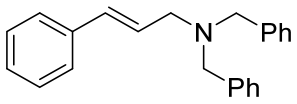
0.180 g, 89 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.39 – 7.36 (m, 2H), 7.34 – 7.28 (m, 2H), 7.26 – 7.21 (m, 1H), 6.54 (d, *J* = 15.9 Hz, 1H), 6. 26 (dt, J_1 = 6.7 Hz, J_2 = 15.9 Hz, 1H), 3.77 – 3.72 (m, 4H), 3.16 (dd, 2H, J_1 = 1.32 Hz, J_2 = 6.8 Hz), 2.53 – 2.48 (m, 4H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 136.9, 133.5, 128.7, 127.7, 126.5, 126.2, 67.1, 61.6, 53.8; **HRMS** (ESI, m/z) calcd. for $C_{13}H_{18}NO$ $[M + H]^+$ 204.1383, found 204.1379.

***(E)*-*N*,*N*-dimethyl-3-phenylprop-2-en-1-amine 2d**



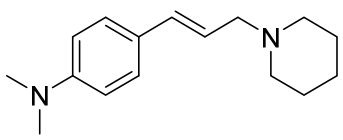
0.117 g, 73 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.40 – 7.36 (m, 2H), 7.33 – 7.28 (m, 2H), 7.25 – 7.20 (m, 1H), 6.52 (d, *J* = 15.7 Hz, 1H), 6.27 (dt, J_1 = 6.6 Hz, J_2 = 15.7 Hz, 1H), 3.08 (dd, J_1 = 1.3 Hz, J_2 = 6.7 Hz, 2H), 2.28 (s, 6H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 137.2, 132.6, 128.7, 127.6, 127.5, 126.4, 62.2, 45.4; **HRMS** (ESI, m/z) calcd. for $C_{11}H_{16}N$ $[M + H]^+$ 162.1277, found 162.1276.

***(E)*-*N*,*N*-dibenzyl-3-phenylprop-2-en-1-amine 2e**



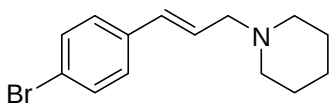
0.16 g, 51 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.42 – 7.19 (m, 15H), 6.54 (d, *J* = 15.8 Hz, 1H), 6.31 (dt, J_1 = 6.5 Hz, J_2 = 15.8 Hz, 1H), 3.64 (s, 4H), 3.23 (dd, J_1 = 1.2 Hz, J_2 = 6.5 Hz, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 139.8, 137.4, 132.6, 128.9, 128.7, 128.4, 127.9, 127.4, 127.0, 126.4, 58.1, 55.9; **HRMS** (ESI, m/z) calcd. for $C_{23}H_{24}N$ $[M + H]^+$ 314.1903, found 314.1911.

***(E)*-*N*,*N*-dimethyl-4-(3-(piperidin-1-yl)prop-1-en-1-yl)aniline 4a**



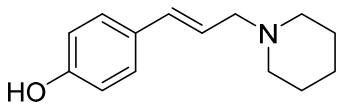
0.210 g, 82 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.29 – 7.25 (m, 2H), 6.69 – 6.66 (m, 2H), 6.40 (d, *J* = 15.7 Hz, 1H), 6.09 (dt, J_1 = 7.0 Hz, J_2 = 15.7 Hz, 1H), 3.09 (dd, J_1 = 1.1 Hz, J_2 = 7.0 Hz, 2H), 2.94 (s, 6H), 2.42 (bs, 4H), 1.64 – 1.56 (m, 4H), 1.48 – 1.42 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 150.0, 132.7, 127.2, 125.8, 122.7, 112.5, 62.1, 54.5, 40.5, 26.0, 24.4; **HRMS** (ESI, m/z) calcd. for $C_{16}H_{24}N_2Na_2$ $[M + 2Na]^{2+}$ 145.0862, found 145.0863.

(E)-1-(3-(4-bromophenyl)allyl)piperidine 4b



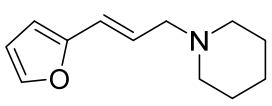
0.256 g, 91 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.41 – 7.37 (m, 2H), 7.22 – 7.19 (m, 2H), 6.40 (d, *J* = 15.9 Hz, 1H), 6.27 (dt, J_1 = 6.6 Hz, J_2 = 15.8 Hz, 1H), 3.07 (dd, J_1 = 1.0 Hz, J_2 = 6.6 Hz, 2H), 2.40 (bs, 4H), 1.63 – 1.55 (m, 4H), 1.45 – 1.41 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 136.1, 131..7, 131.4, 128.3, 127.9, 121.1, 61.8, 54.7, 26.0, 24.4; **HRMS** (ESI, m/z) calcd. for $C_{14}H_{19}BrN$ [M + H+]: 280.0695; found: 280.0685.

(E)-4-(3-(piperidin-1-yl)prop-1-en-1-yl)phenol 4c



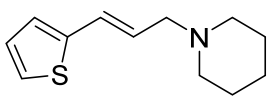
0.196 g, 90 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 8.87 (bs, 1H), 7.11 – 7.04 (m, 2H), 6.75 – 6.64 (m, 2H), 6.39 (d, *J* = 16.0 Hz, 1H), 5.98 (dt, J_1 = 7.1 Hz, J_2 = 15.9 Hz, 1H), 3.12 (d, J_2 = 7.0 Hz, 2H), 2.57 (bs, 4H), 1.73 – 1.62 (m, 4H), 1.53 – 1.42 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 157.0, 134.3, 128.3, 127.9, 122.0, 116.4, 61.7, 54.4, 25.2, 24.1; **HRMS** (ESI, m/z) calcd. for $C_{14}H_{20}NO$ [M + H+]: 218.1539; found: 218.1544.

***(E)*-1-(3-(furan-2-yl)allyl)piperidine 4d**



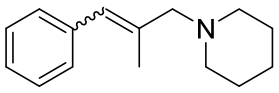
0.170 g, 89 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.34 – 7.30 (m, 1H), 6.37 – 6.28 (m, 2H), 6.26-6.16 (m, 2H), 3.09 – 3.06 (m, 2H), 2.41 (bs, 4H), 1.65 – 1.53 (m, 4H), 1.49 – 1.39 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 152.9, 141.9, 126.2, 121.2, 111.3, 107.2, 61.7, 54.7, 26.1, 24.5; **HRMS** (ESI, m/z) calcd. for $C_{12}H_{18}NO$ $[M + H]^+$ 192.1383, found 192.1375.

***(E)*-1-(3-(thiophen-2-yl)allyl)piperidine 4e**



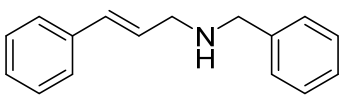
0.190 g, 92 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.14 – 7.10 (m, 1H), 6.97 – 6.88 (m, 2H), 6.62 (d, 1H, J = 15.7 Hz), 6.13 (dt, 1H, J_1 = 6.9 Hz, J_2 = 15.7 Hz) 3.07 (dd, 2H, J_1 = 1.3 Hz, J_2 = 6.9 Hz), 2.42 (bs, 4H), 1.67-1.53 (m, 4H), 1.51 – 1.38 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 142.5, 127.4, 127.3, 125.8, 125.2, 124.0, 61.7, 54.7, 26.1, 24.5; **HRMS** (ESI, m/z) calcd. for $C_{12}H_{18}NS$ $[M + H]^+$ 208.1154, found 208.1158.

1-(2-methyl-3-phenylallyl)piperidine 6



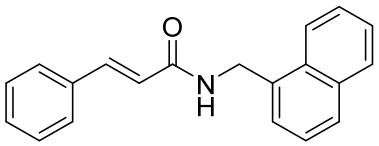
0.191 g, 88 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.40 – 7.19 (m, 5H, minor and major), 6.52 (s, 1H, minor), 6.48 (s, 1H, major), 3.12 – 3.10 (m, 2H, minor), 3.04 – 3.01 (m, 2,H, major), 2.42 (bs, 4H, major), 2.32 (bs, 4H, minor), 2.02 – 2.00 (m, 3H, minor), 1.98 – 1.96 (m, 3H, major), 1.69 – 1.56 (m, 4H, major and minor), 1.55 – 1.40 (m, 2H, major and minor); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 138.2, 136.5, 128.9, 128.1, 127.1, 126.2, 68.8, 54.7, 26.1, 24.6, 16.9 (major); 138.2, 137.3, 129.2, 128.6, 127.9, 126.1, 59.8, 54.5, 26.1, 24.5, 23.2 (minor); **HRMS** (ESI, m/z) calcd. for $C_{15}H_{22}N$ [M + H+]: 216.1747; found: 216.1737.

(E)-N-benzyl-3-phenylprop-2-en-1-amine 9



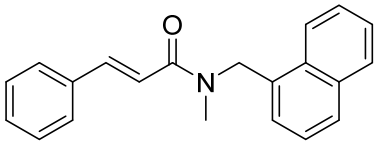
0.159 g, 71 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.46 – 7.24 (m, 10H), 6.59 (d, J = 15.9 Hz, 1H), 6.38 (dt, J = 6.3 Hz, 15.9 Hz, 1H), 3.90 (s, 2H), 3.49 (d, J = 5.5 Hz, 2H), 2.66 (s, 1H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 139.6, 137.0, 131.8, 128.5, 128.3, 127.8, 127.4, 127.1, 126.3, 53.0, 50.1; **HRMS** (ESI, m/z) calcd. for $C_{16}H_{18}N$ [M + H+]: 224.1434; found: 224.1444.

N-(naphthalen-1-ylmethyl)cinnamamide 12



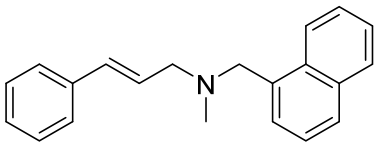
1.306 g, 91 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 8.08 – 8.04 (m, 1H), 7.90 – 7.80 (m, 2H), 7.69 (d, *J* = 15.6 Hz, 1H), 7.59 – 7.42 (m, 6H), 7.36 – 7.31 (m, 3H), 6.36 (d, *J* = 15.6 Hz, 1H), 5.91 (bs, 1H), 5.03 – 5.00 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 165.6, 141.6, 134.9, 134.1, 133.6, 131.6, 129.9, 128.9, 128.9, 127.9, 127.1, 126.9, 126.2, 125.6, 123.7, 120.4, 42.2; **HRMS** (ESI, m/z) calcd. for $C_{21}H_{19}NNaO$ [M + Na+]: 324.1359; found: 324.1345.

N-methyl-N-(naphthalen-1-ylmethyl)cinnamamide 13



0.976 g, 93 % yield; **^{1}H-NMR** (500 MHz, $(CD_3)_2SO$): δ = 7.66 – 7.62 (m, 1H), 7.45 – 7.42 (m, 1H), 7.14 – 6.94 (m, 6H), 6.89 – 6.81 (m, 4H), 6.68 – 6.62 (m, 1H), 4.68 (s, 2H), 2.55 (s, 3H); **^{13}C-NMR** (125 MHz, $(CD_3)_2SO$): δ = 165.6, 140.9, 134.8, 133.1, 132.6, 130.7, 128.8, 128.1, 128.0, 127.3, 127.2, 125.7, 125.2, 124.8, 122.8, 118.5, 48.5, 33.8; **HRMS** (ESI, m/z) calcd. for $C_{20}H_{17}NNaO$ [M + Na+]: 310.1202; found: 310.1208.

(E)-N-methyl-N-(naphthalen-1-ylmethyl)-3-phenylprop-2-en-1-amine 14



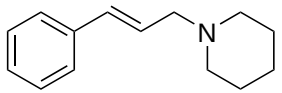
0.263 g, 92 % yield; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 8.40 – 8.36 (m, 1H), 7.93 – 7.88 (m, 1H), 7.87 – 7.81 (m, 1H), 7.63 – 7.51 (m, 3H), 7.50 – 7.44 (m, 3H), 7.41 – 7.35 (m, 2H), 7.32 – 7.26 (m, 1H), 6.65 (d, *J* = 16.0 Hz, 1H), 6.45 (dt, *J* = 6.6 Hz, 16.0 Hz, 1H), 4.02 (s, 2H), 3.35 (dd, *J* = 1.2 Hz, 6.6 Hz, 2H), 2.35 (s, 3H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 137.3, 135.0, 134.0, 132.8, 132.6, 128.7, 128.6, 128.0, 127.7, 127.6, 127.5, 126.4, 126.0, 125.7, 125.2, 124.8, 60.5, 60.2, 42.6; **HRMS** (ESI, m/z) calcd. for $C_{21}H_{22}N$ [M + H+]: 288.1747; found: 288.1747.

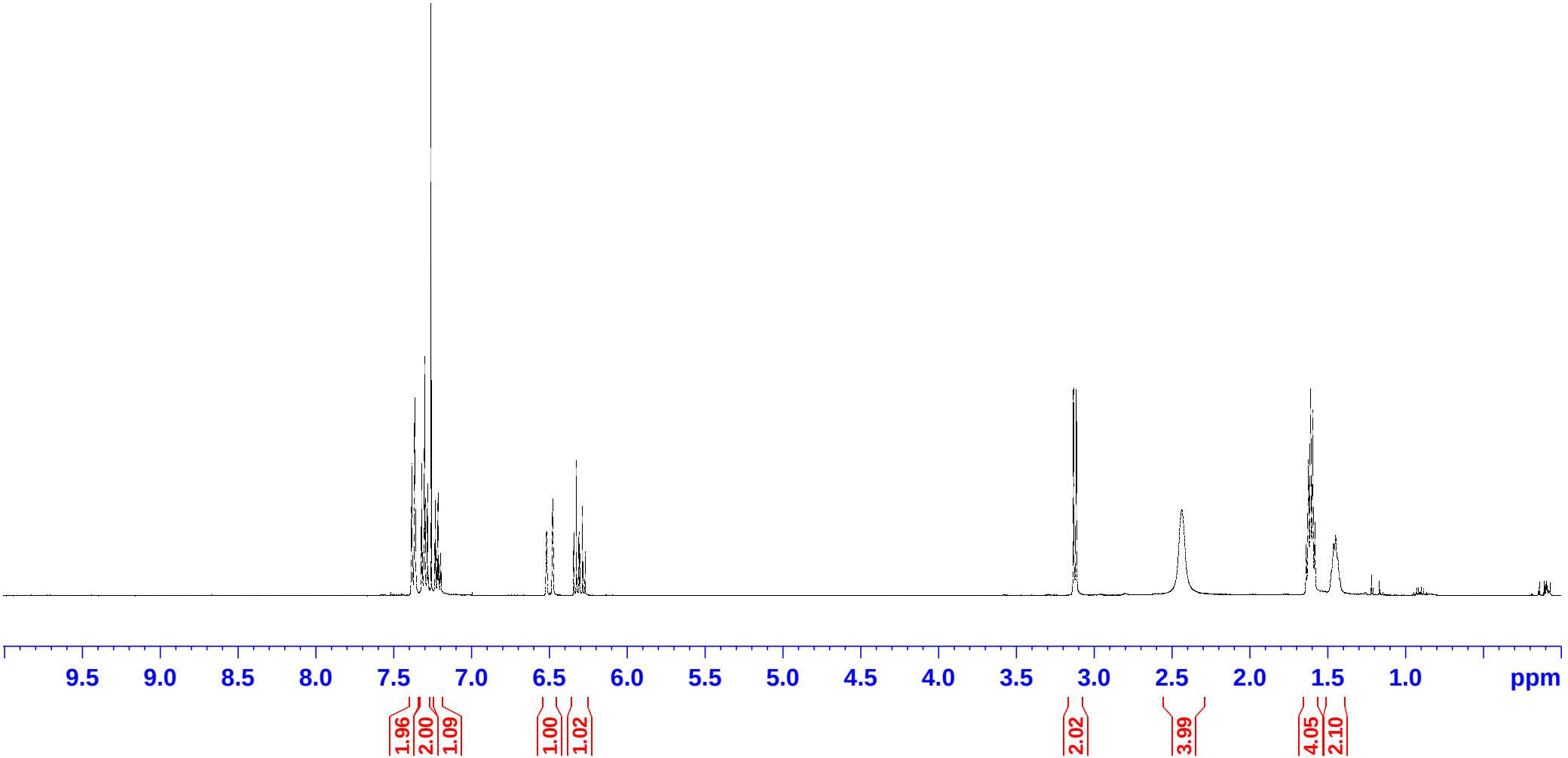
Spectroscopic data.

CDCl3
400 MHz

7.382 7.364 7.319 7.301 7.281 7.233 7.215 7.197 6.518 6.478 6.343 6.326 6.309 6.303 6.286 6.269

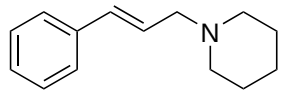
3.133 3.130 3.116 3.113 2.437 1.637 1.623 1.609 1.595 1.581 1.477 1.448





CDCl3
100 MHz

137.22
132.78
128.66
127.48
127.34
126.41
62.00
54.73
26.10
24.47

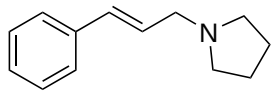


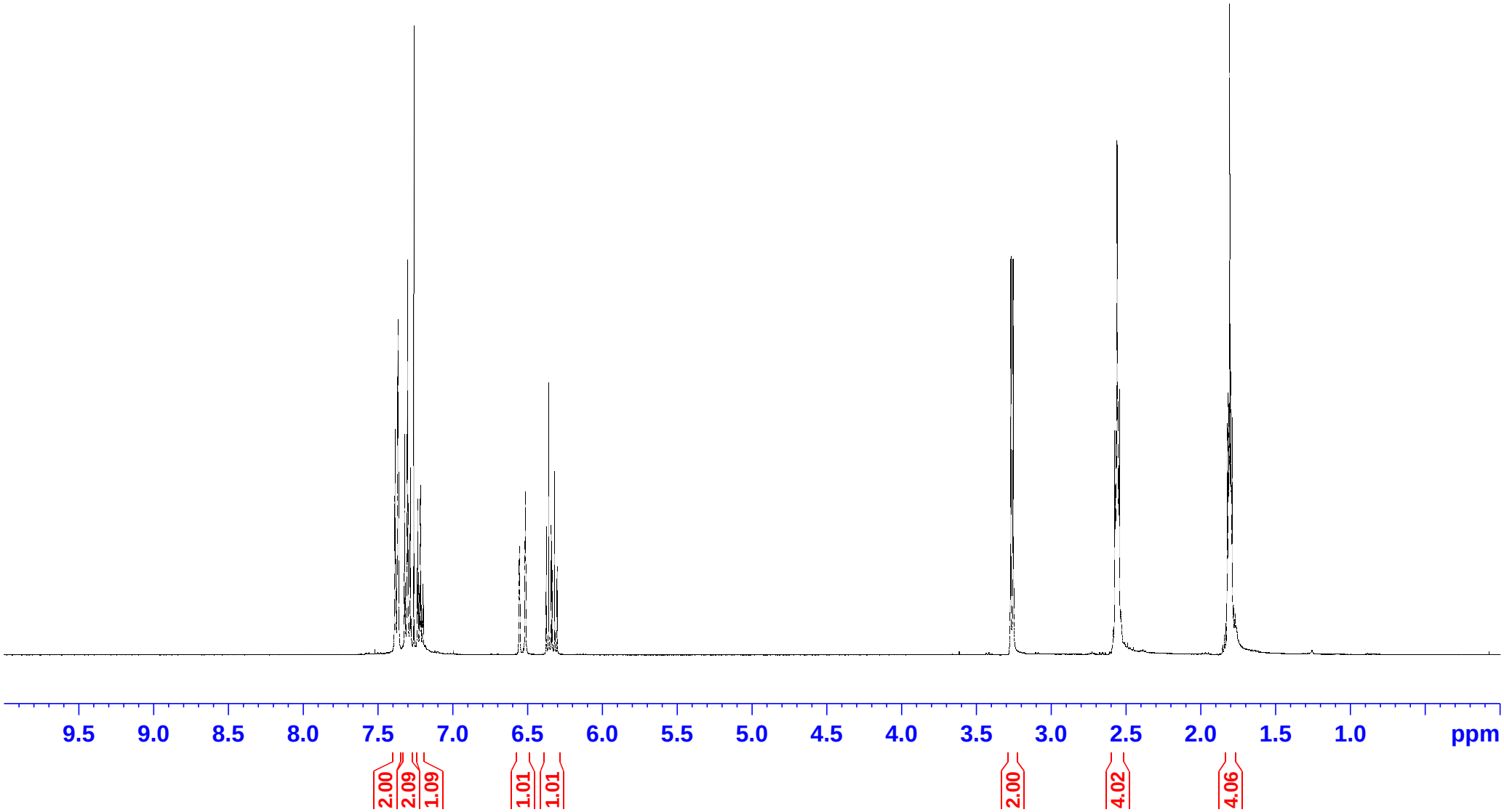
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

CDCl3
400 MHz

7.383 7.366 7.321 7.302 7.283 7.234 7.215 7.197 6.554 6.514 6.374 6.358 6.341 6.335 6.318 6.301

3.271 3.268 3.254 3.251 2.575 2.558 2.541 1.822 1.815 1.805 1.796 1.789

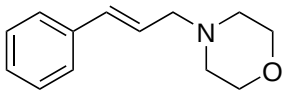


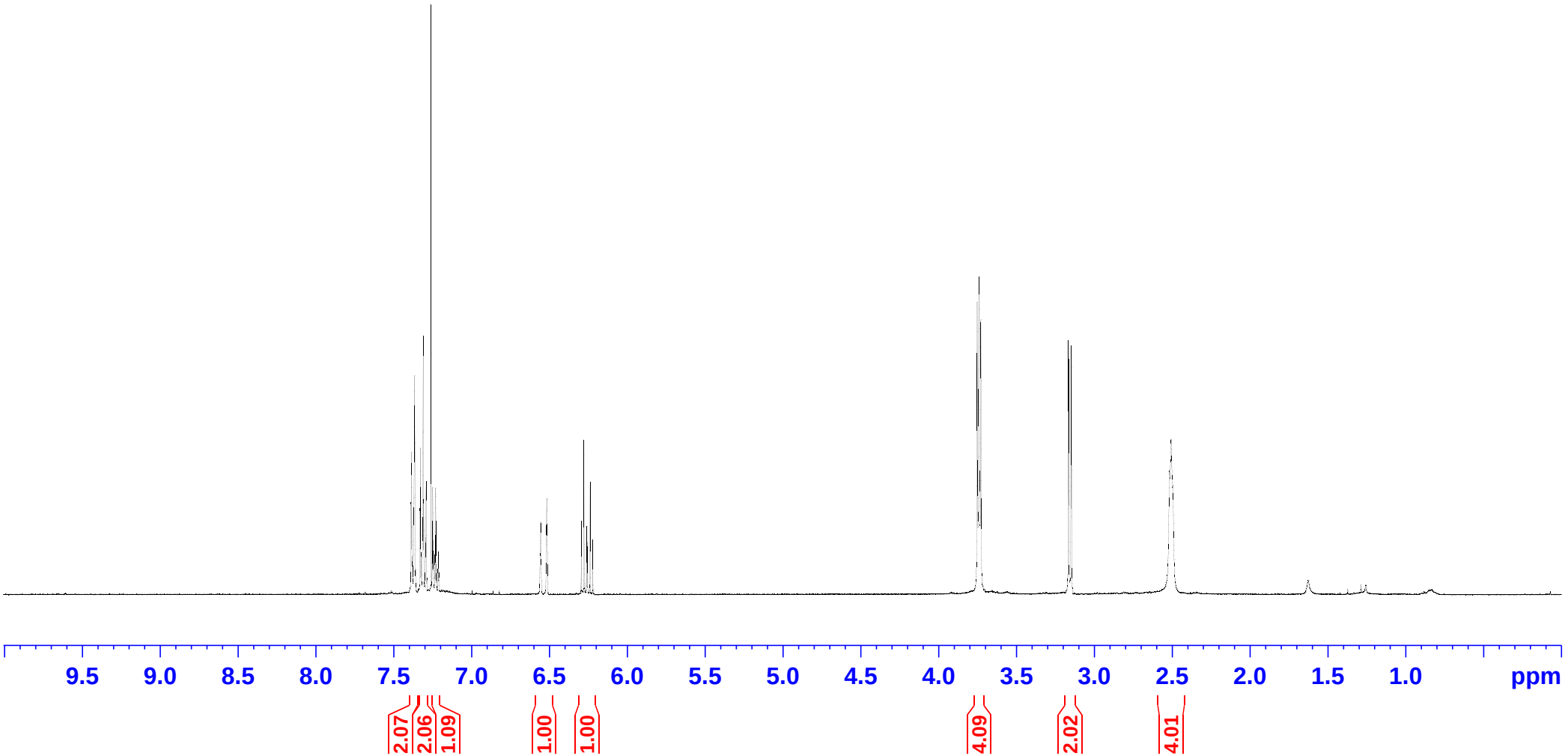


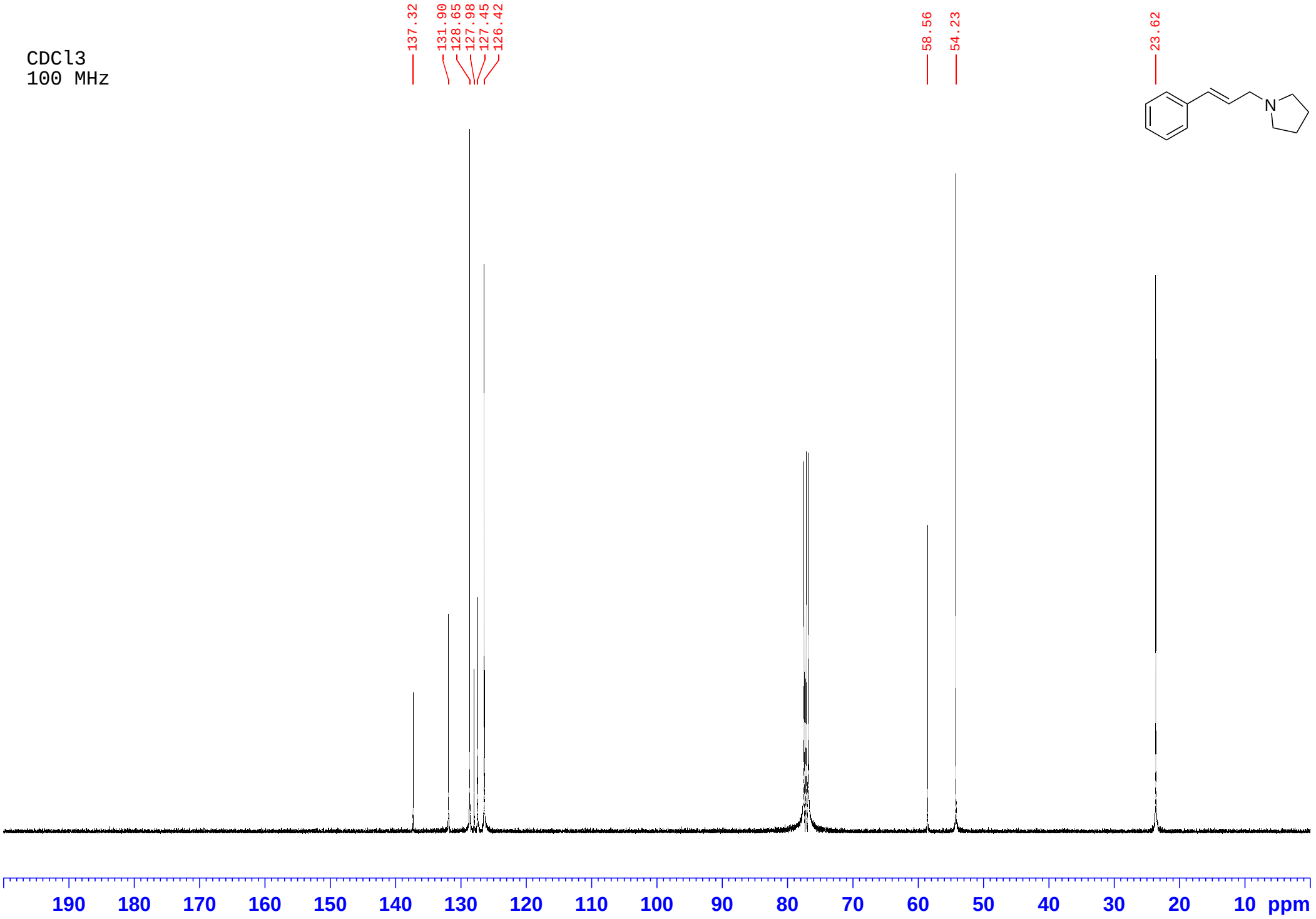
CDCl3
400 MHz

7.385
7.367
7.329
7.311
7.291
7.249
7.231
7.213
6.556
6.517
6.295
6.278
6.261
6.255
6.238
6.221

3.752
3.740
3.728
3.167
3.163
3.150
3.146
2.517
2.506
2.495

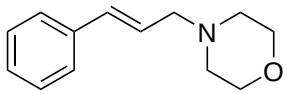


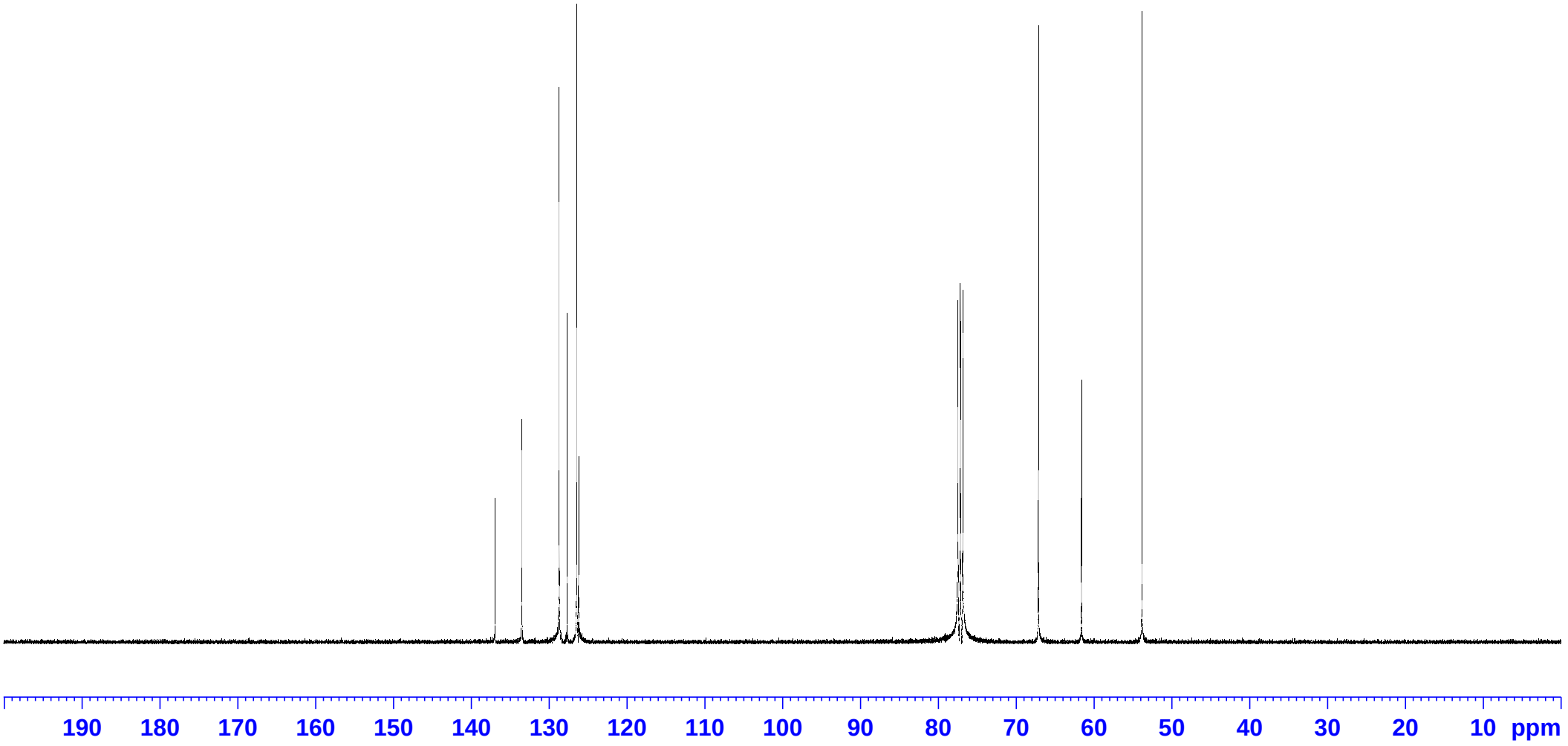


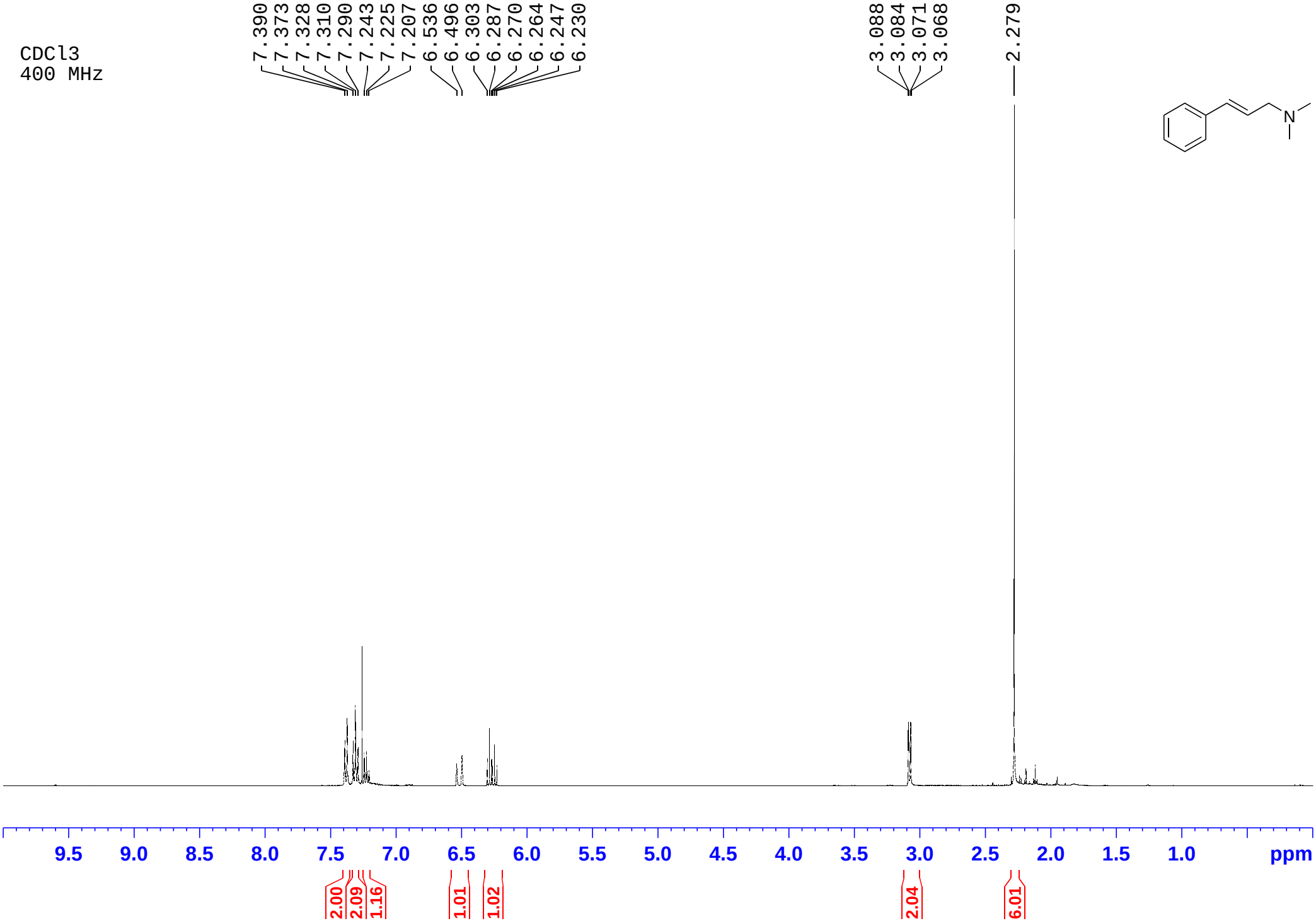

CDCl3
100 MHz
137.32
131.90
128.65
127.98
127.45
126.42
58.56
54.23
23.62
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

CDCl3
100 MHz

136.94
133.50
128.70
127.70
126.45
126.20
67.13
61.60
53.83

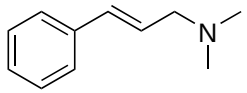


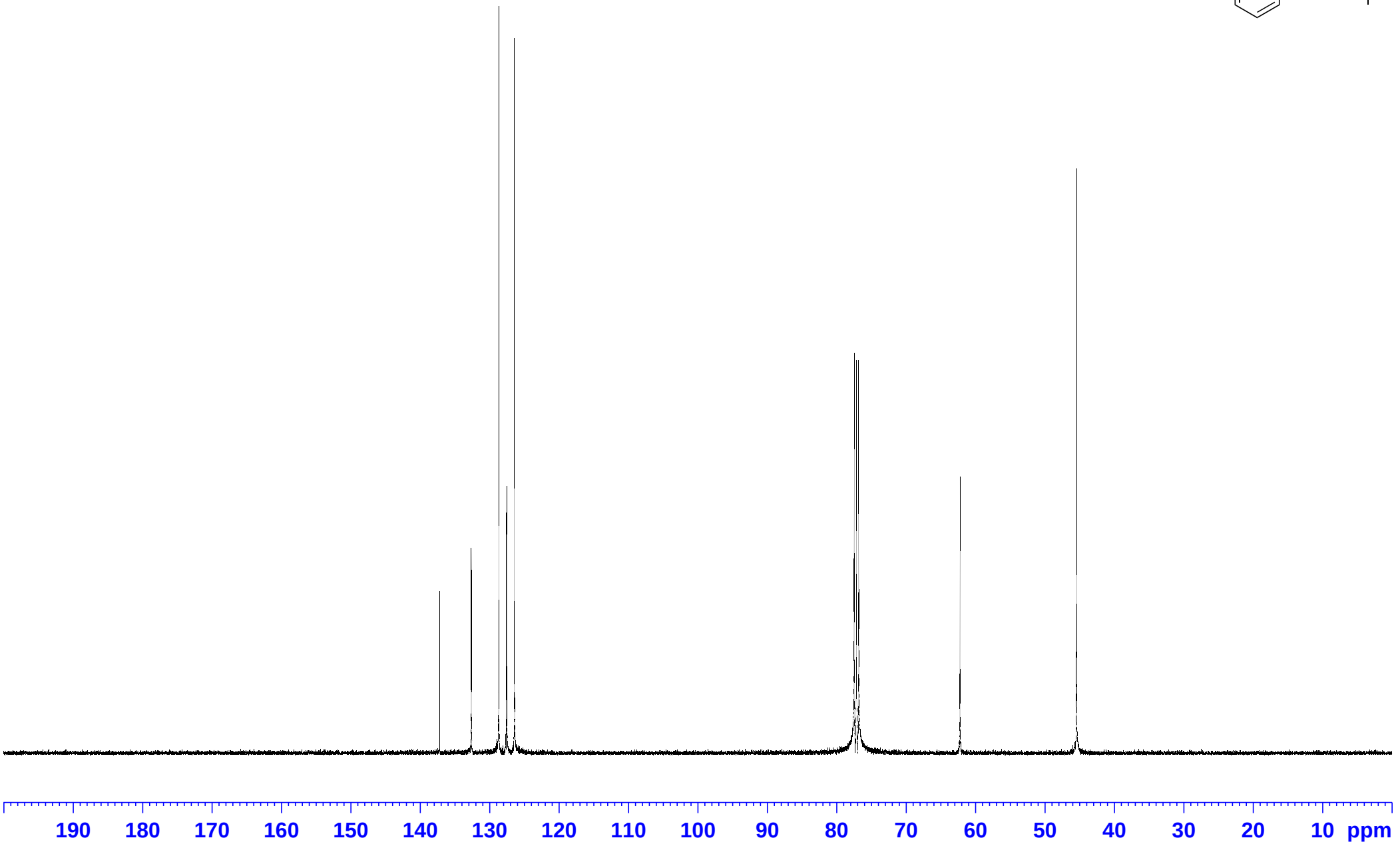



CDCl3
400 MHz
7.390
7.373
7.328
7.310
7.290
7.243
7.225
7.207
6.536
6.496
6.303
6.287
6.270
6.264
6.247
6.230
3.088
3.084
3.071
3.068
2.279
N
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.00
2.09
1.16
1.01
1.02
2.04
6.01

CDCl3
100 MHz

137.20
132.64
128.68
127.61
127.54
126.43
62.23
45.44



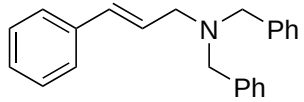


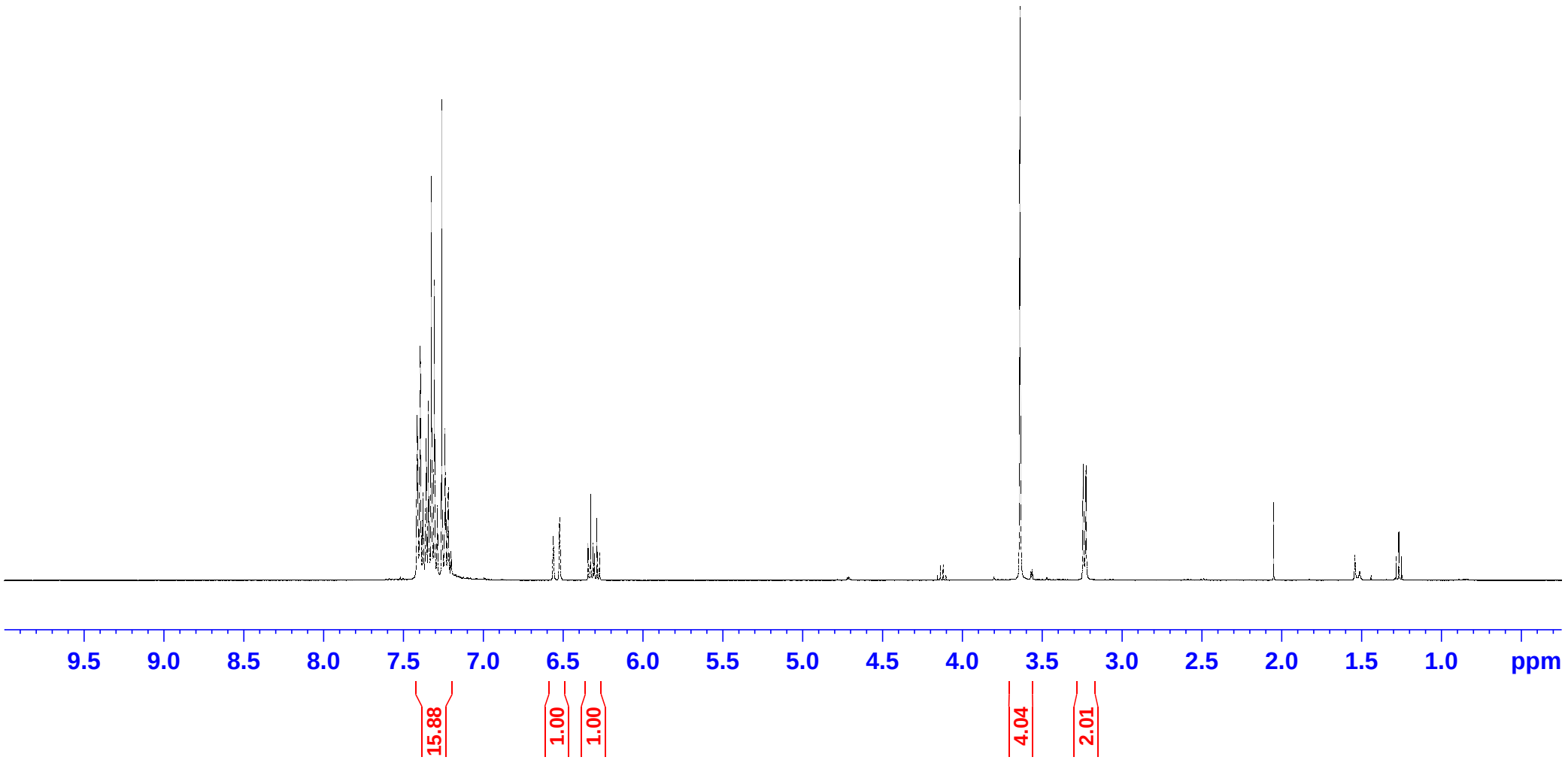
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

CDCl3
400 MHz

7.413
7.396
7.377
7.343
7.325
7.306
7.287
7.240
7.219
7.201
6.561
6.522
6.344
6.328
6.312
6.304
6.288
6.272

3.639
3.244
3.241
3.228
3.225

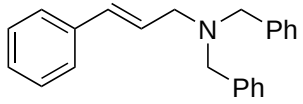


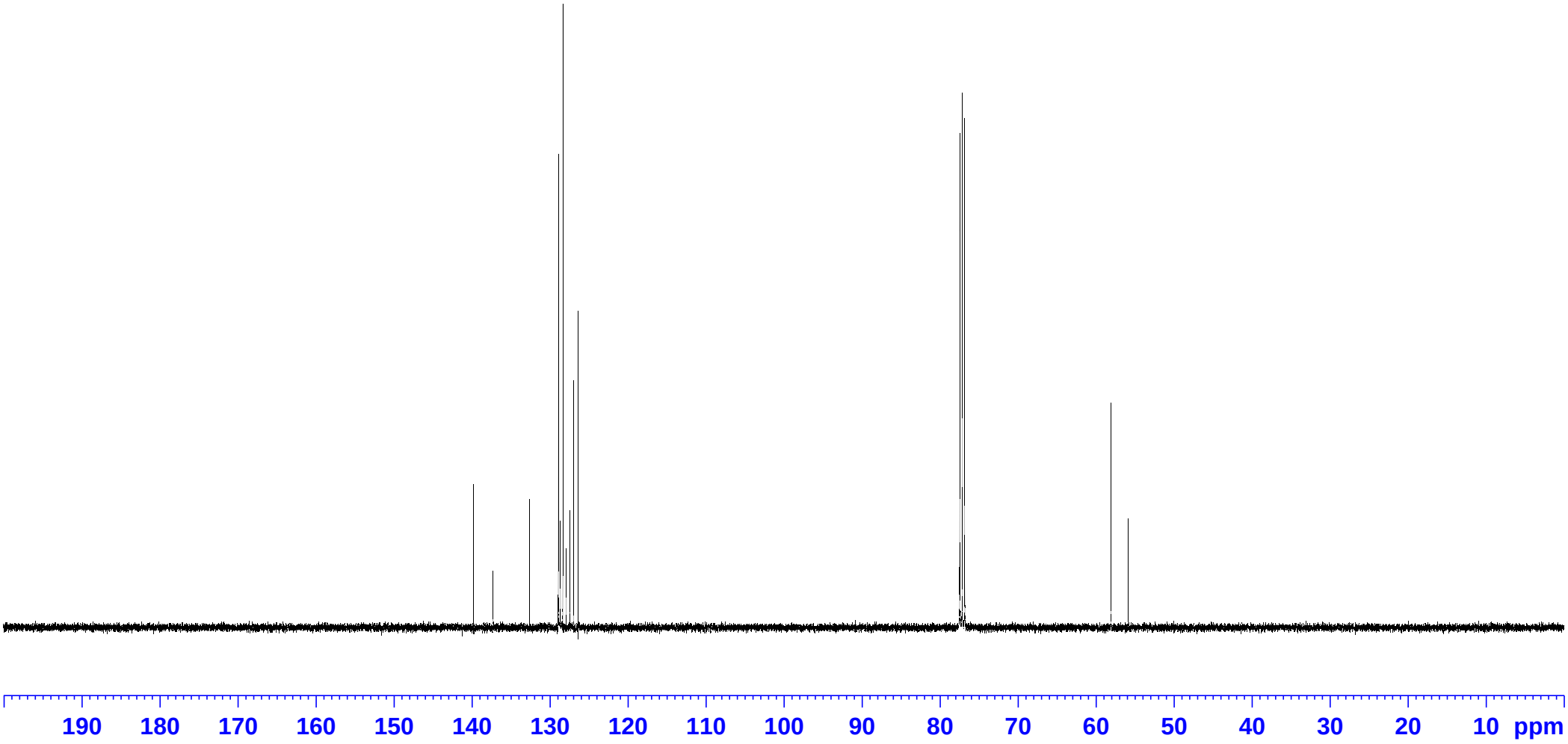


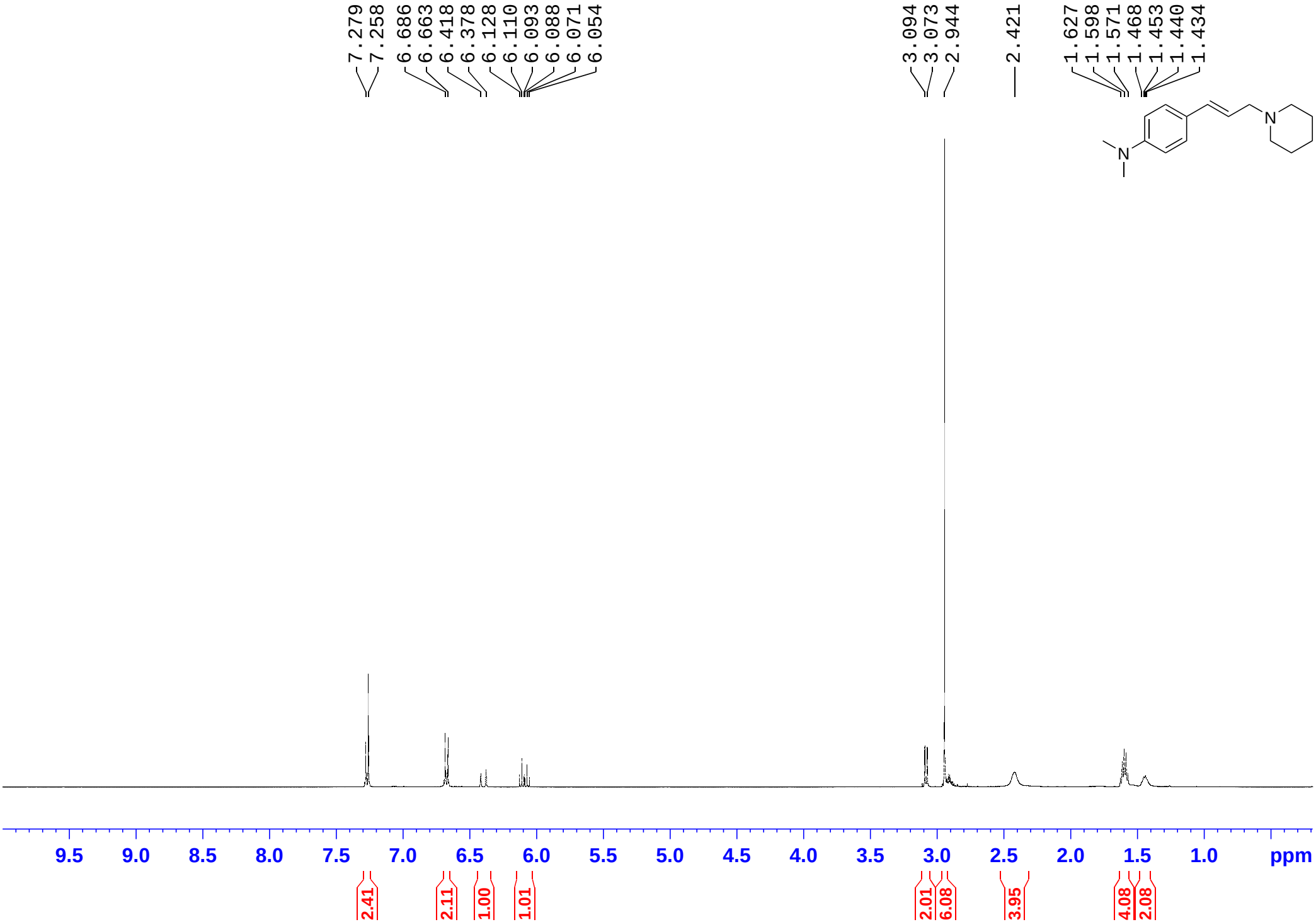
CDCl3
100 MHz

139.77
137.35
132.61
128.93
128.65
128.36
127.91
127.44
126.99
126.39

58.09
55.91

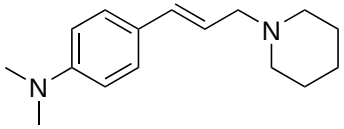


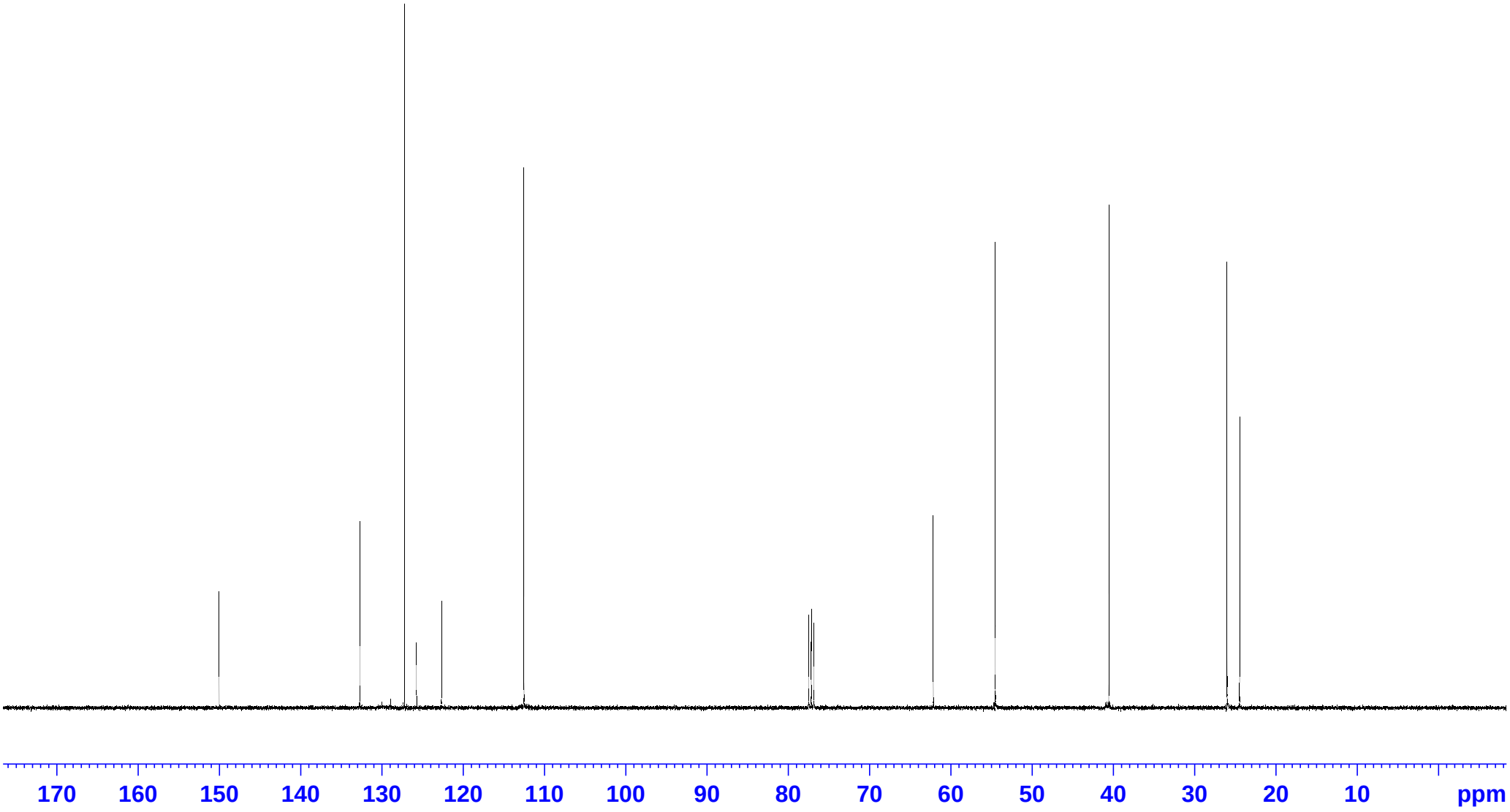



7.279
7.258
6.686
6.663
6.418
6.378
6.128
6.110
6.093
6.088
6.071
6.054
3.094
3.073
2.944
2.421
1.627
1.598
1.571
1.468
1.453
1.440
1.434
N
N
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.41
2.11
1.00
1.01
2.01
6.08
3.95
4.08
2.08

CDCl3
100 MHz

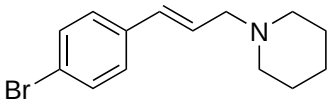
150.02
132.71
127.22
125.75
122.65
112.52
62.14
54.52
40.54
26.00
24.44

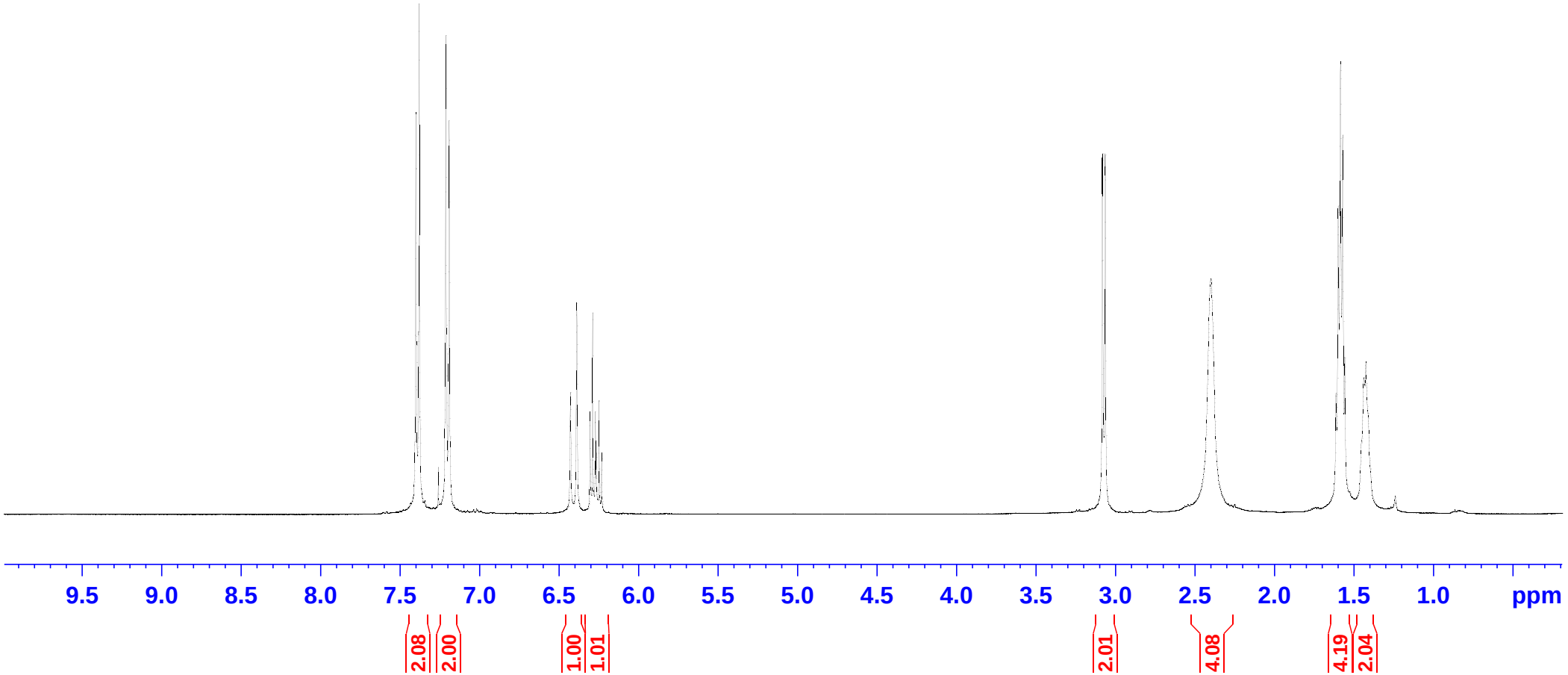




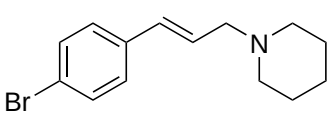
CDCl3
400 MHz

7.401
7.380
7.213
7.191
6.429
6.389
6.305
6.289
6.272
6.266
6.249
6.232
3.083
3.064
2.400
1.613
1.599
1.585
1.571
1.557
1.437
1.425





CDCl3
100 MHz



136.08
131.66
131.42
128.26
127.86
121.11
61.81
54.71
26.03
24.37

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

CDCl3
400 MHz

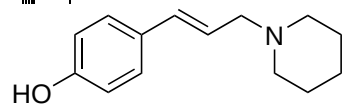
8.869

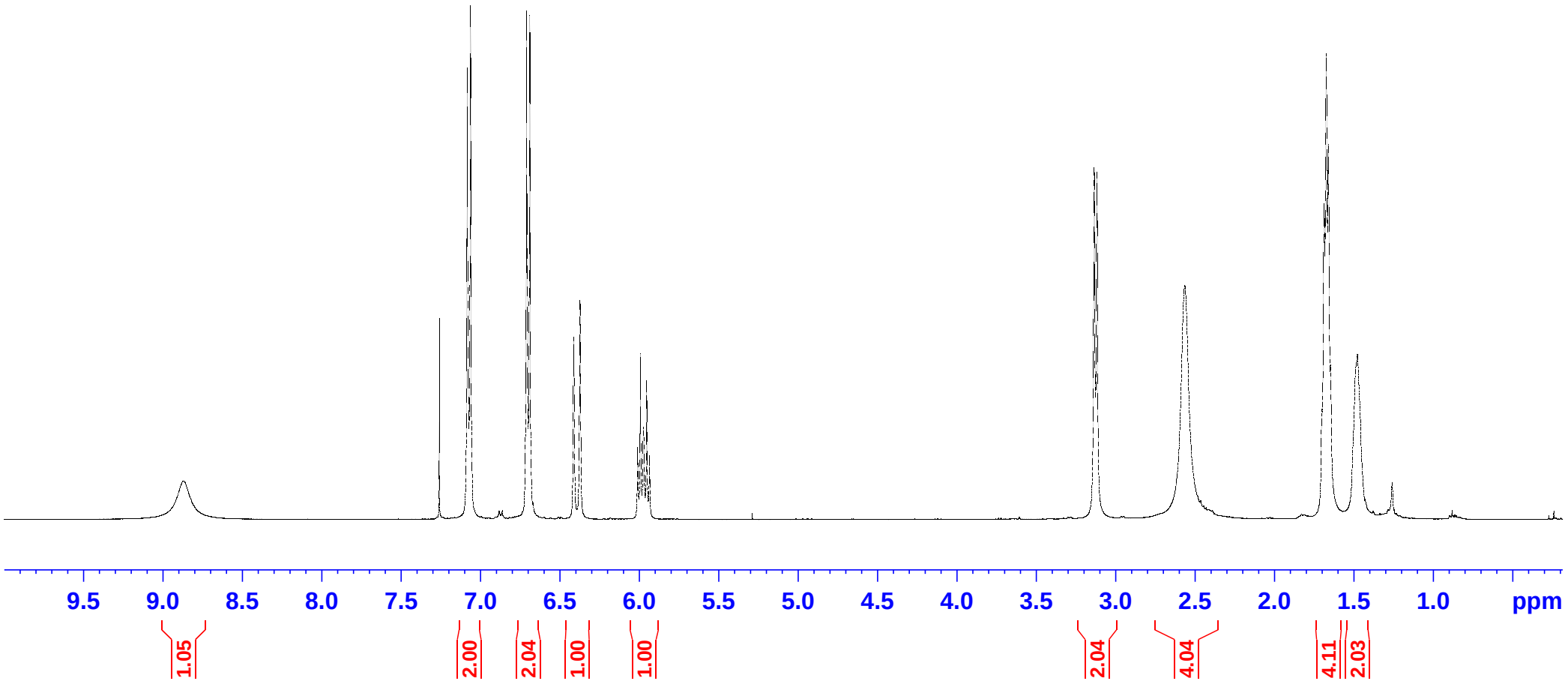
7.084
7.063
6.711
6.690
6.413
6.374
6.011
5.993
5.975
5.954
5.936

3.135
3.118

2.565

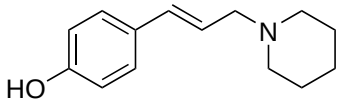
1.701
1.688
1.674
1.660
1.647
1.478

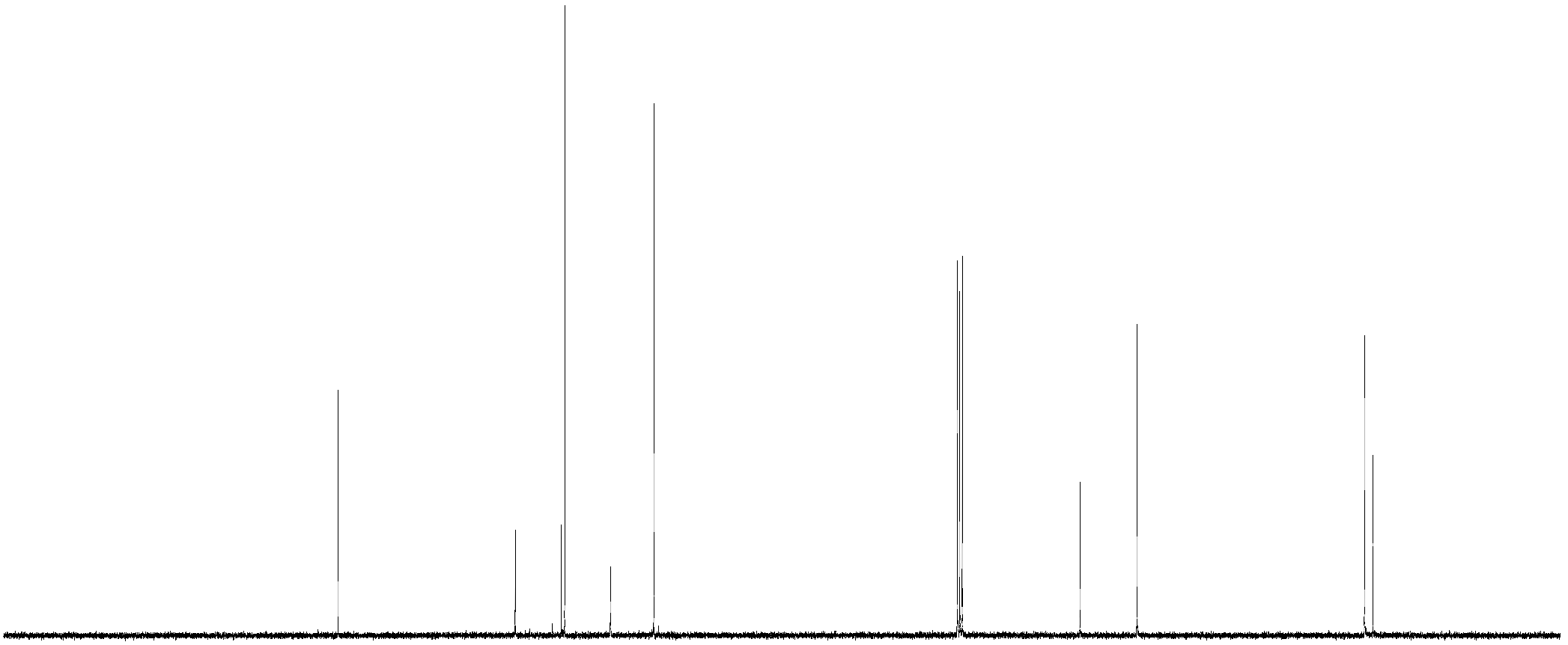




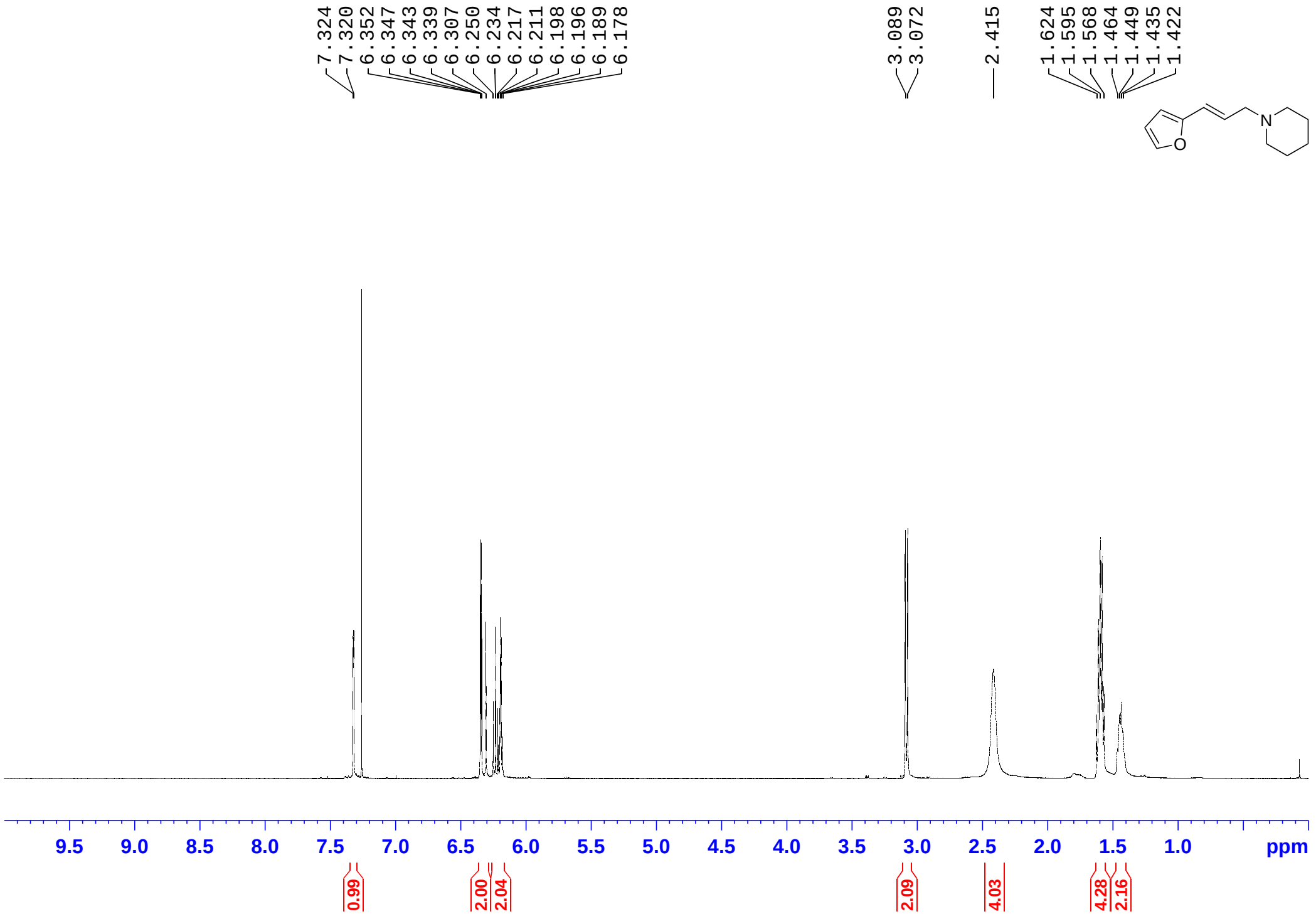
CDCl3
100 MHz

157.02
134.26
128.32
127.90
122.00
116.44
61.72
54.39
25.16
24.10



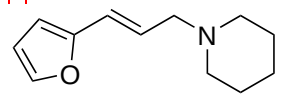


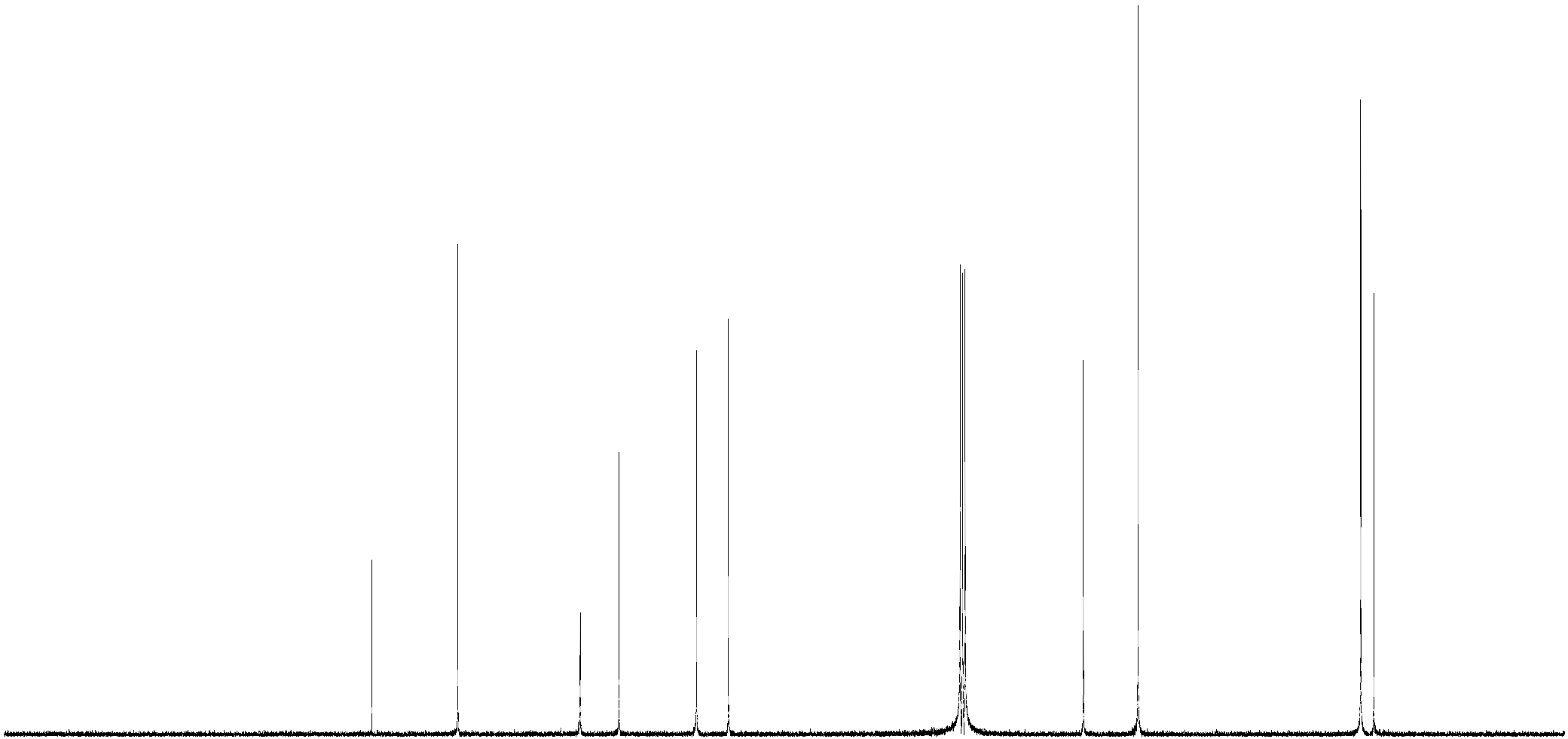
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.324
7.320
6.352
6.347
6.343
6.339
6.307
6.250
6.234
6.217
6.211
6.198
6.196
6.189
6.178
3.089
3.072
2.415
1.624
1.595
1.568
1.464
1.449
1.435
1.422
N
O
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
ppm
0.99
2.00
2.04
2.09
4.03
4.28
2.16

CDCl3
100 MHz

152.86
141.85
126.17
121.20
111.25
107.17
61.67
54.66
26.12
24.45





190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

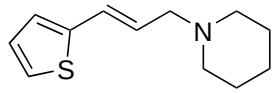
CDCl3
400 MHz

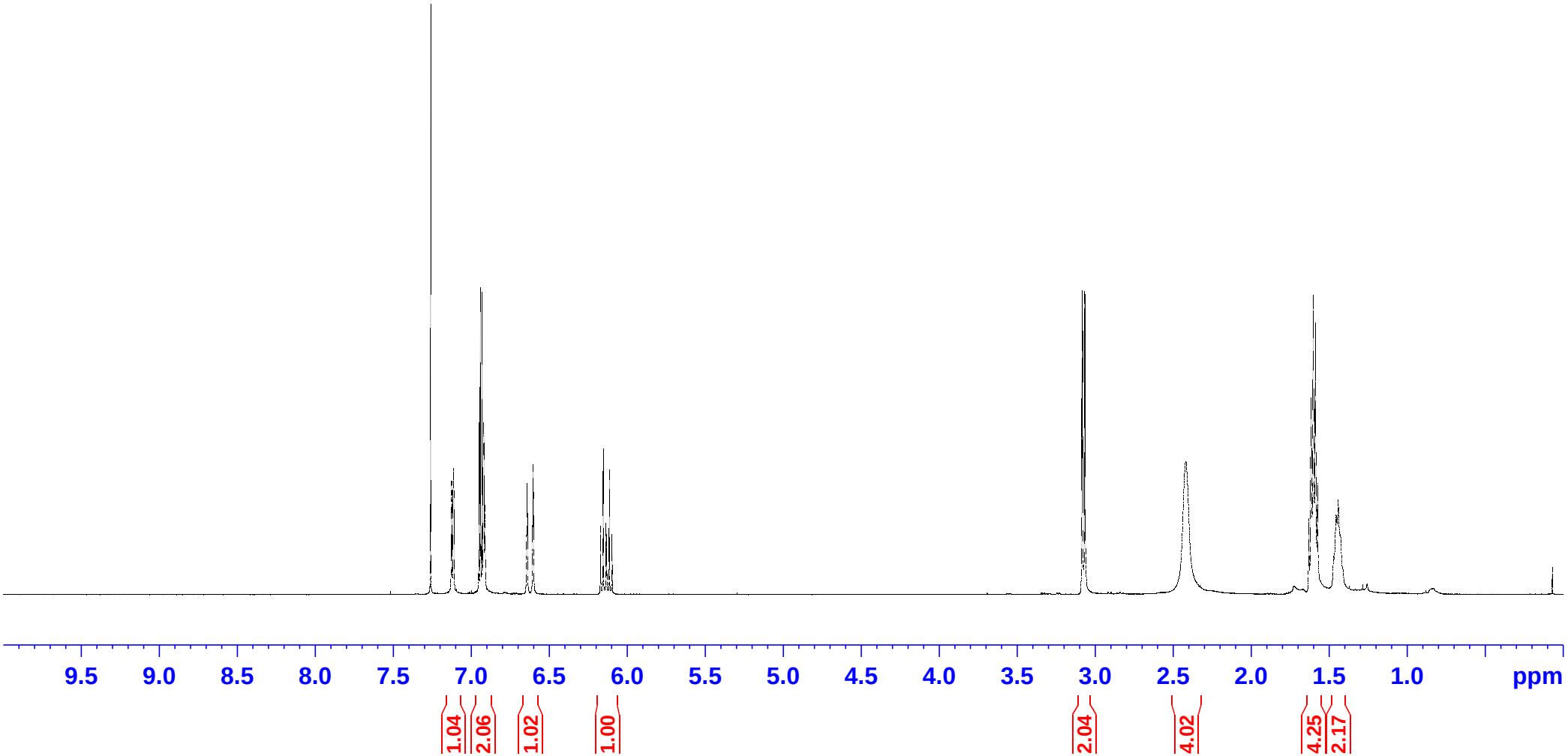
7.126
7.113
6.951
6.942
6.938
6.930
6.919
6.913
6.642
6.603
6.170
6.153
6.136
6.131
6.114
6.097

3.084
3.064

2.419

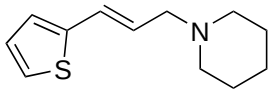
1.630
1.601
1.574
1.471
1.456
1.442
1.428

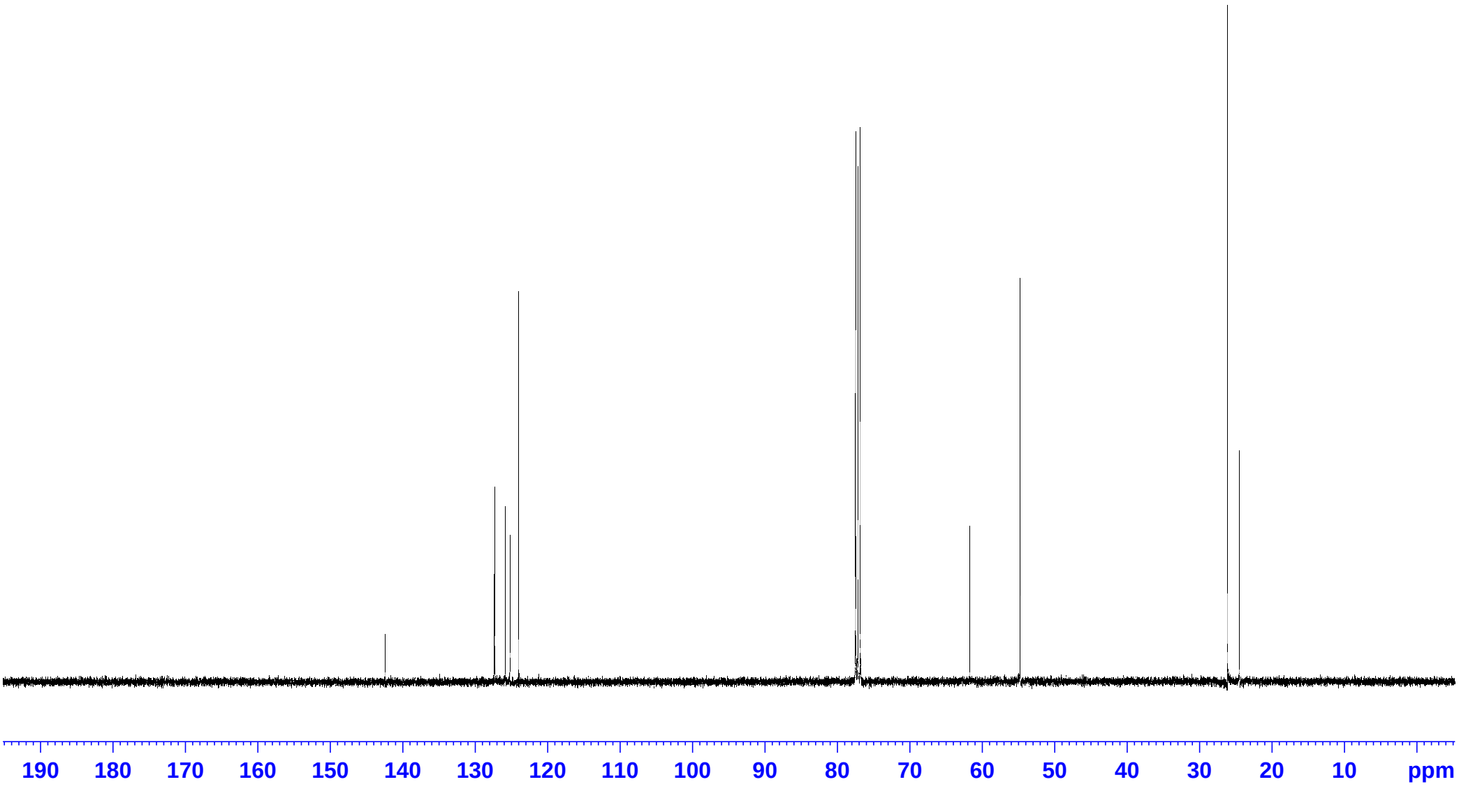


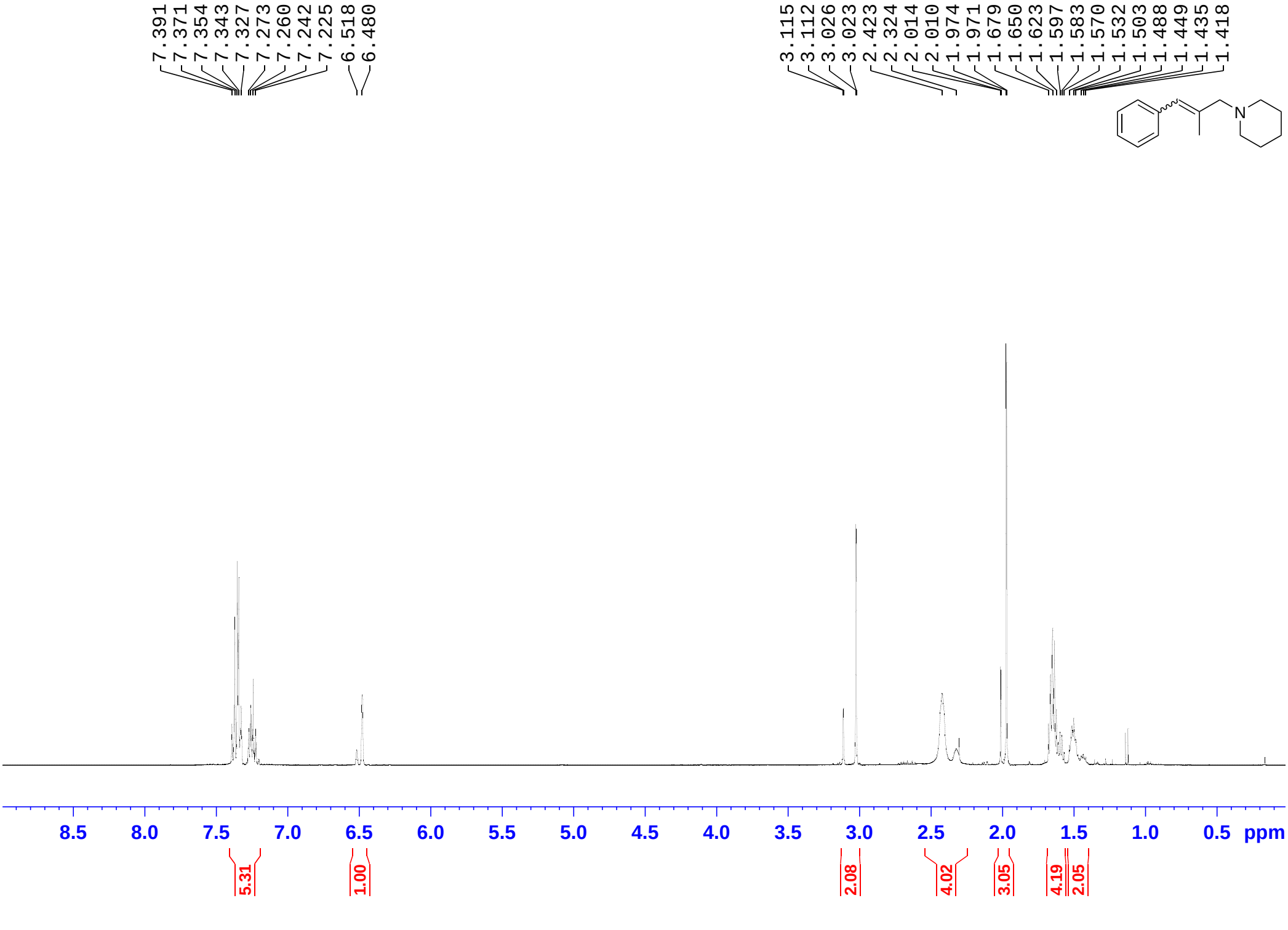


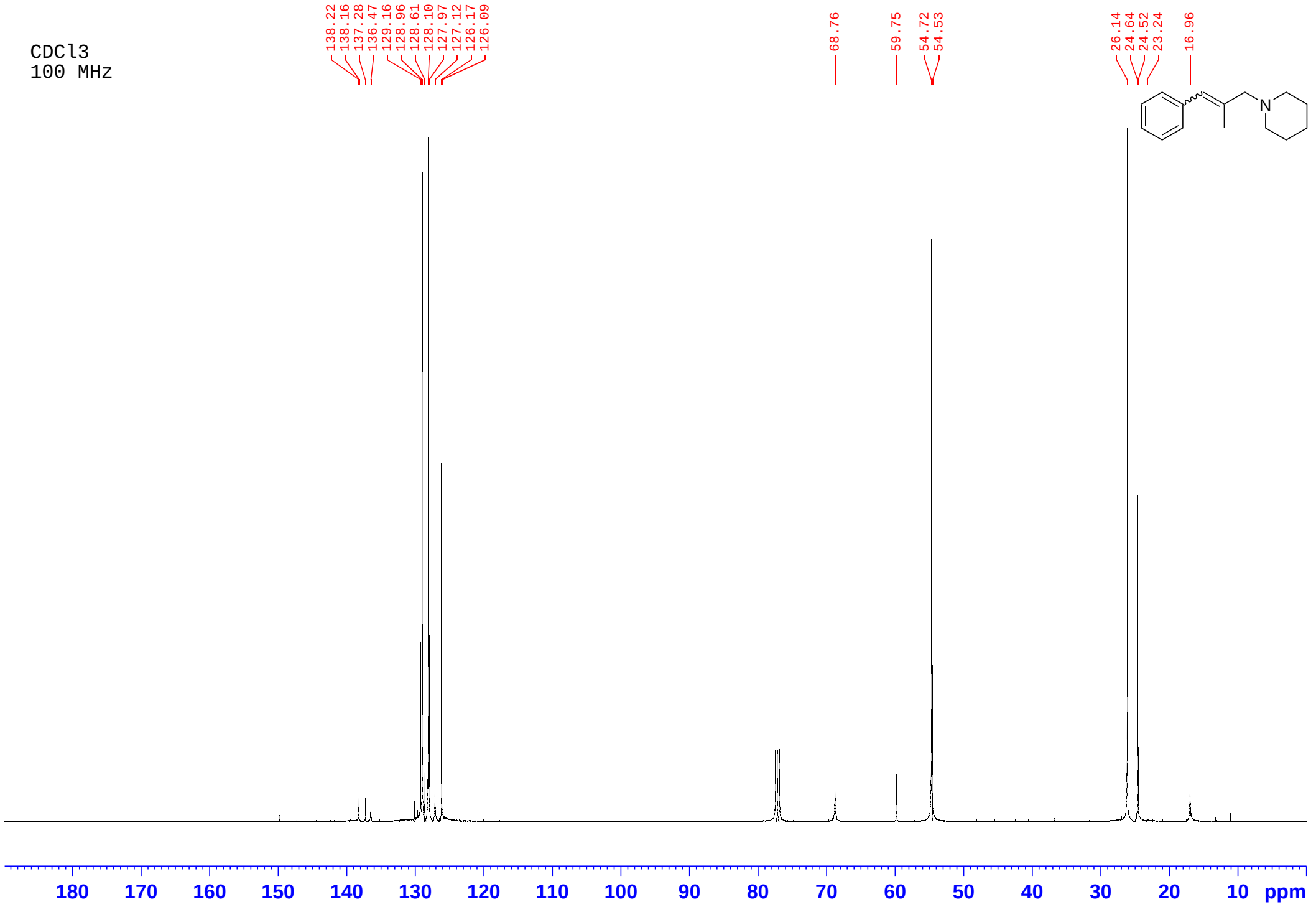
CDCl3
100 MHz

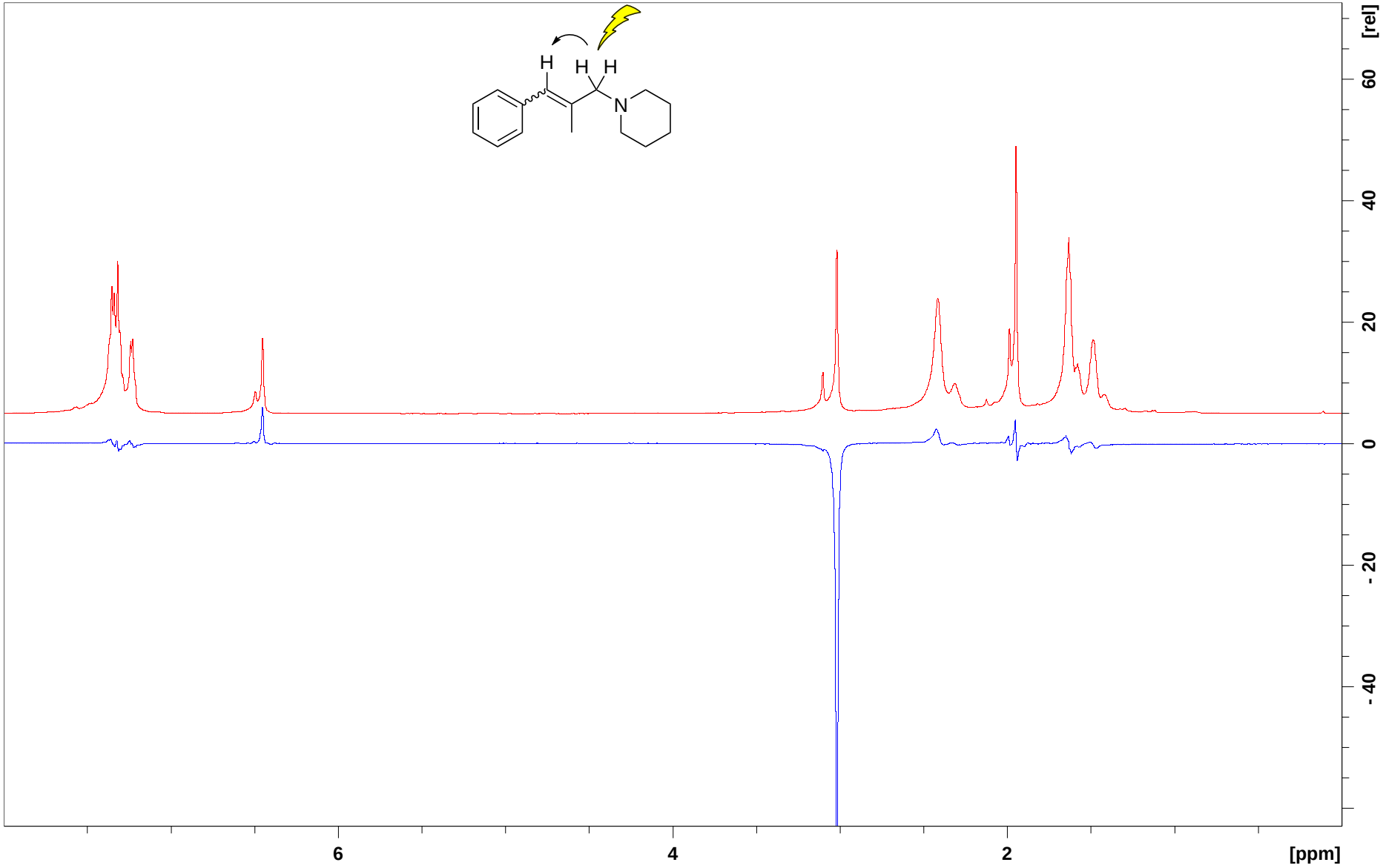
142.45
127.38
127.26
125.83
125.19
124.03
61.74
54.74
26.14
24.47

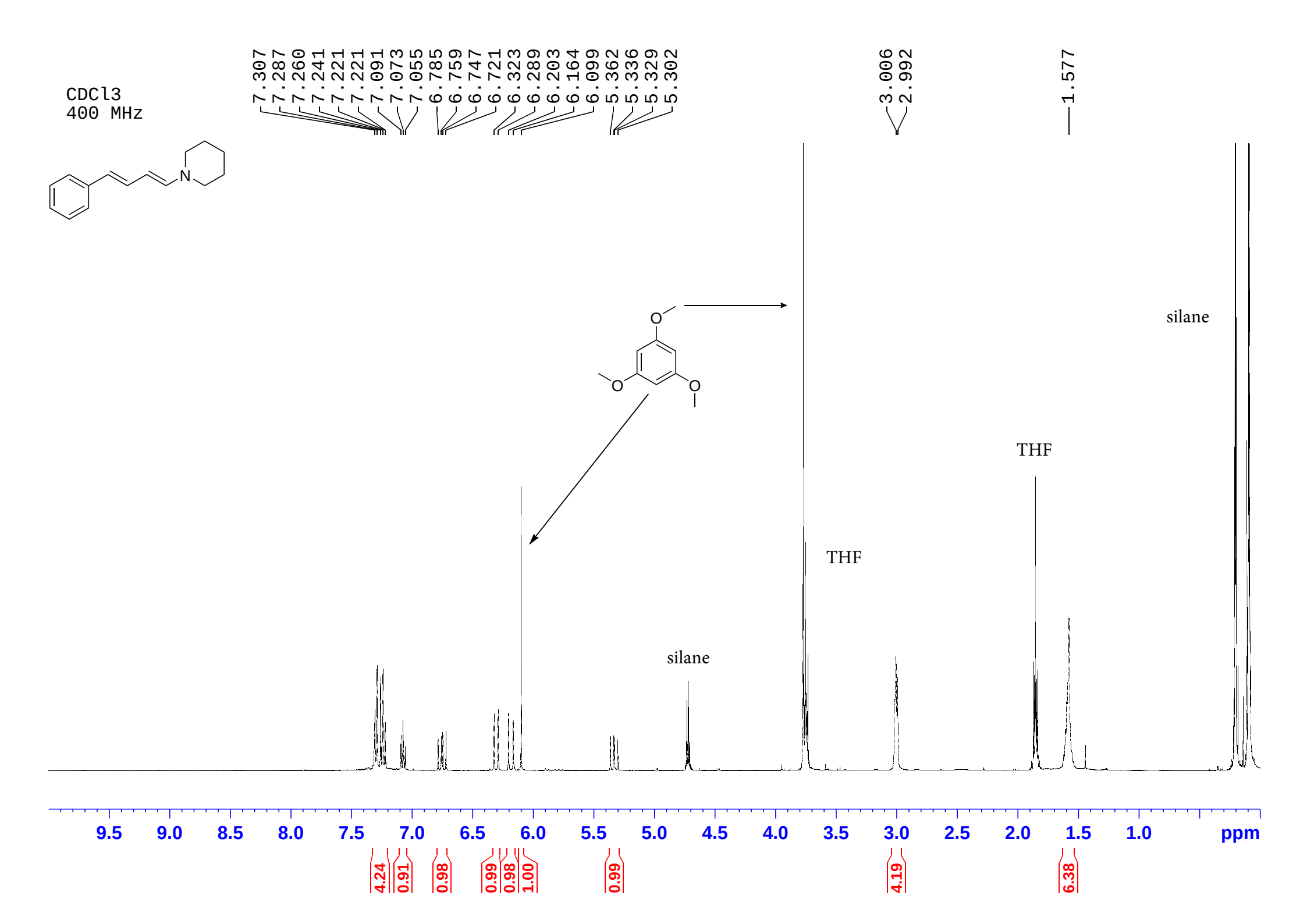





7.391
7.371
7.354
7.343
7.327
7.273
7.260
7.242
7.225
6.518
6.480
3.115
3.112
3.026
3.023
2.423
2.324
2.014
2.010
1.974
1.971
1.679
1.650
1.623
1.597
1.583
1.570
1.532
1.503
1.488
1.449
1.435
1.418
N
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.31
1.00
2.08
4.02
3.05
4.19
2.05


CDCl3
100 MHz
138.22
138.16
137.28
136.47
129.16
128.96
128.61
128.10
127.97
127.12
126.17
126.09
68.76
59.75
54.72
54.53
26.14
24.64
24.52
23.24
16.96
N
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


H
H
H
N
60
40
20
0
- 20
- 40
[rel]
6
4
2
[ppm]

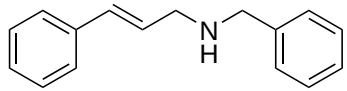

CDCl3
400 MHz
7.307
7.287
7.260
7.241
7.221
7.221
7.091
7.073
7.055
6.785
6.759
6.747
6.721
6.323
6.289
6.203
6.164
6.099
5.362
5.336
5.329
5.302
3.006
2.992
1.577
N
O
O
O
silane
THF
THF
silane
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
4.24
0.91
0.98
0.99
0.98
1.00
0.99
4.19
6.38

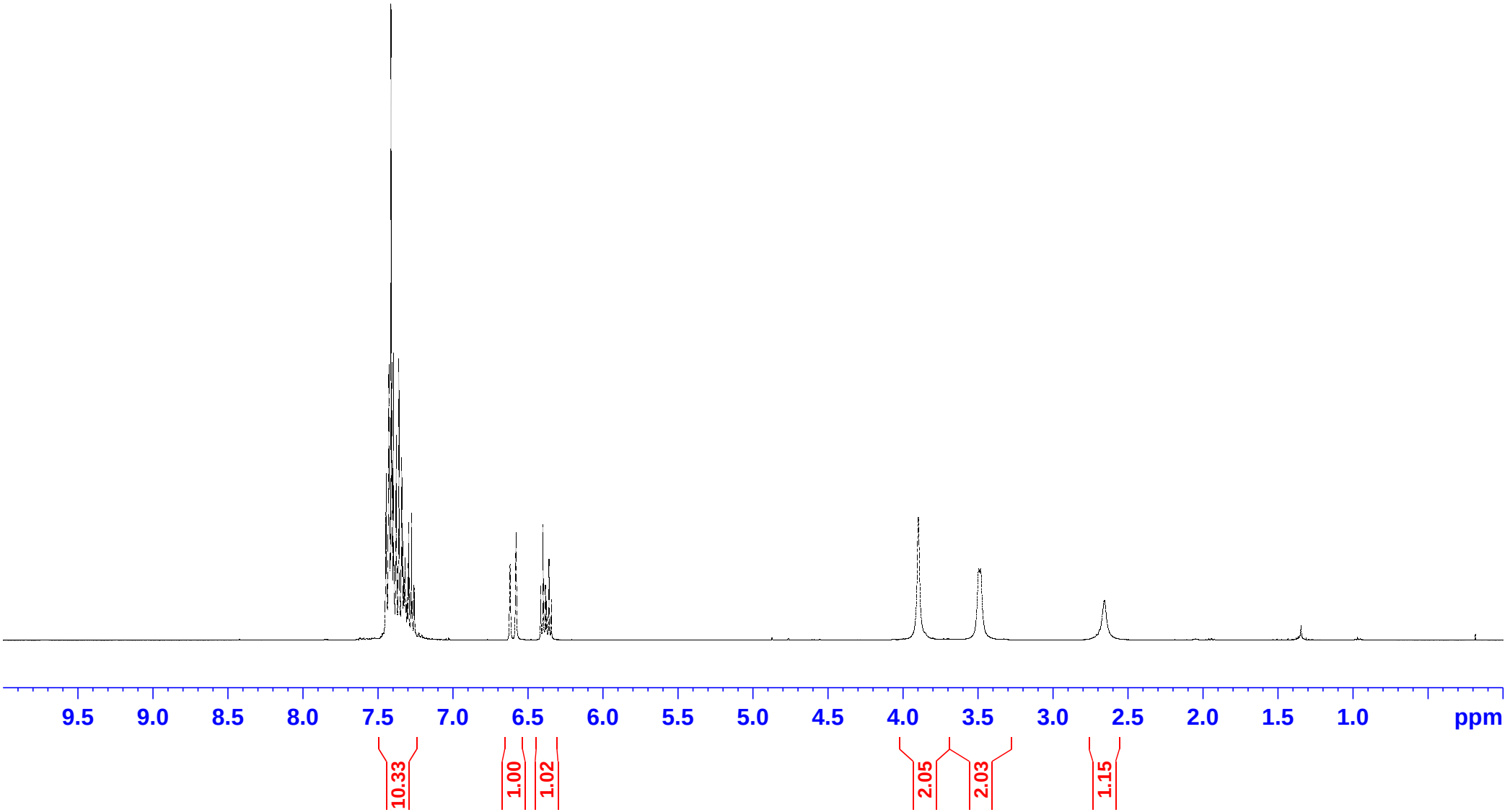
CDCl3
400 MHz

7.444
7.426
7.412
7.395
7.360
7.341
7.294
7.276
7.258
6.619
6.579
6.414
6.399
6.383
6.375
6.359
6.343

3.897
3.494
3.482

2.657

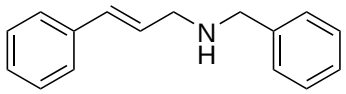


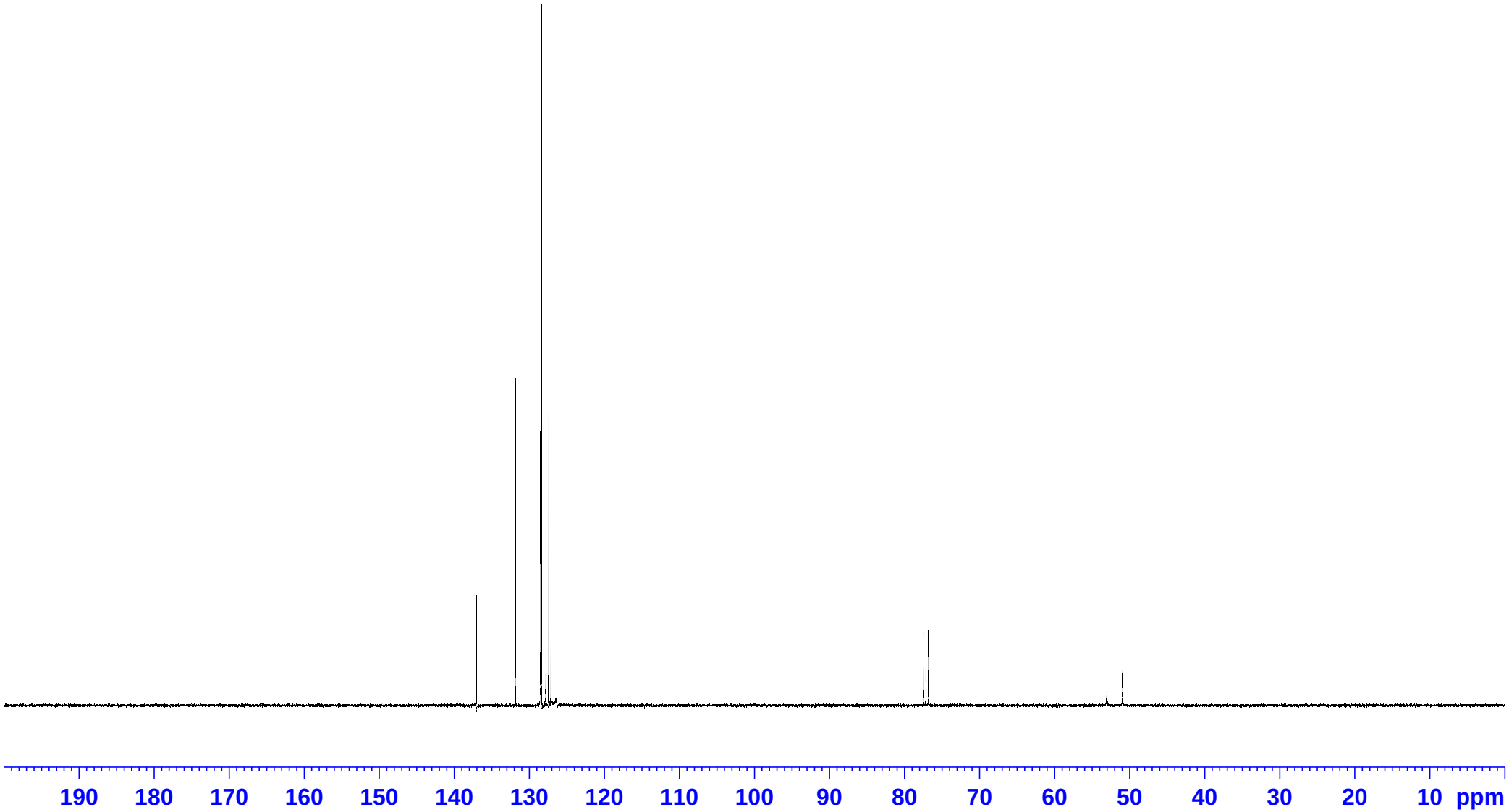


CDCl3
100 MHz

139.62
137.03
131.82
128.54
128.47
128.32
127.80
127.43
127.12
126.31

53.03
50.93

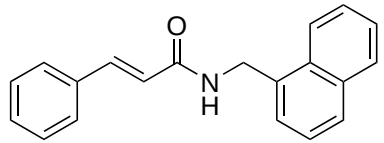


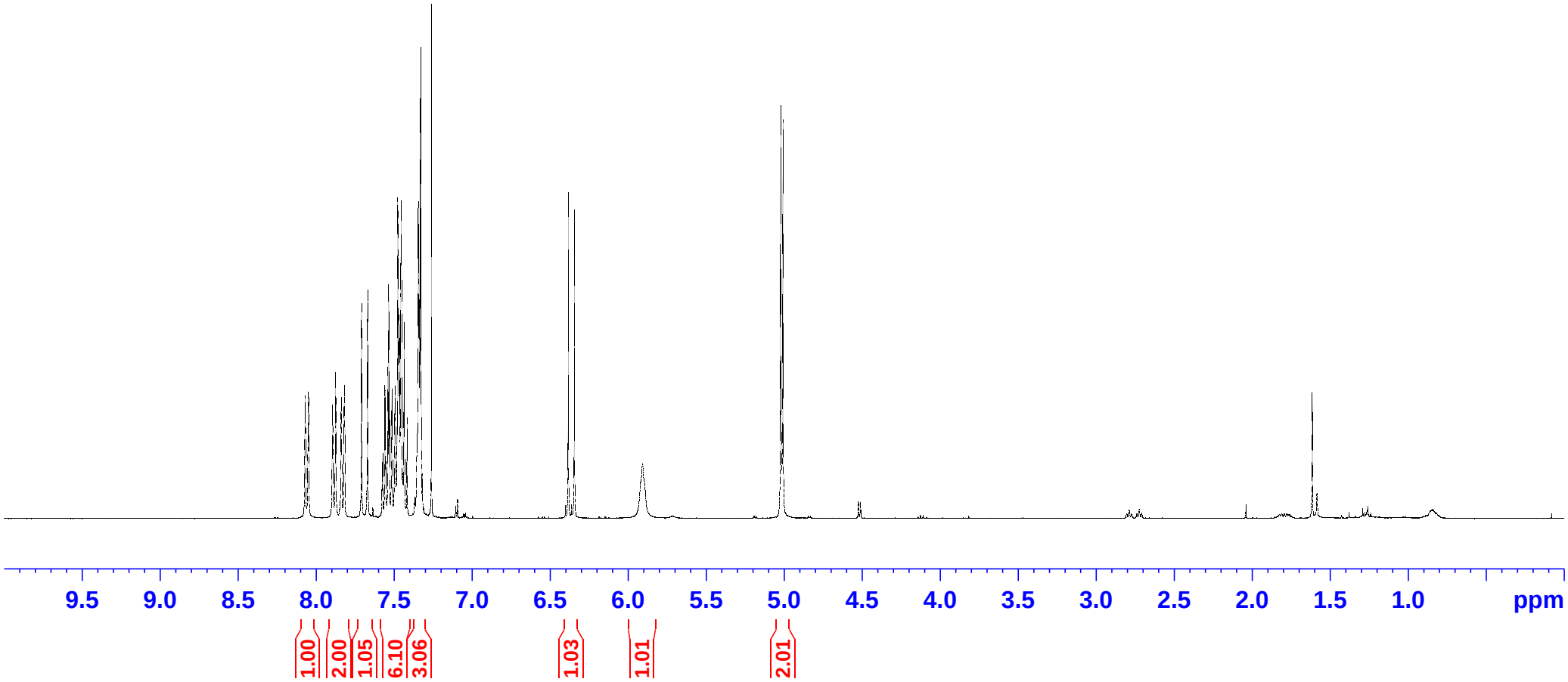


CDCl3
400 MHz

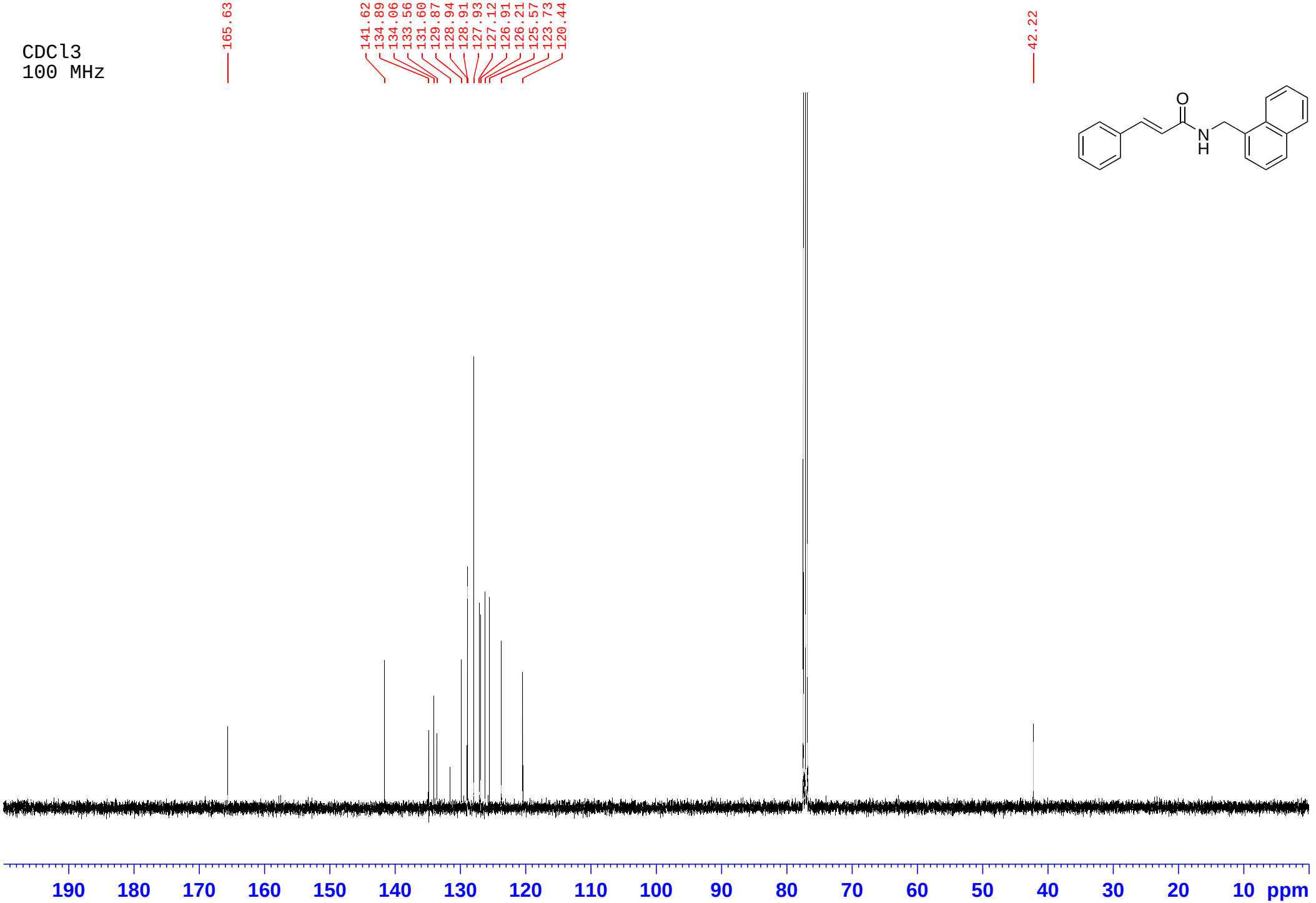
8.069
8.049
7.896
7.876
7.838
7.818
7.708
7.669
7.572
7.559
7.534
7.514
7.494
7.476
7.454
7.434
7.344
7.334
7.327
6.383
6.344
5.908

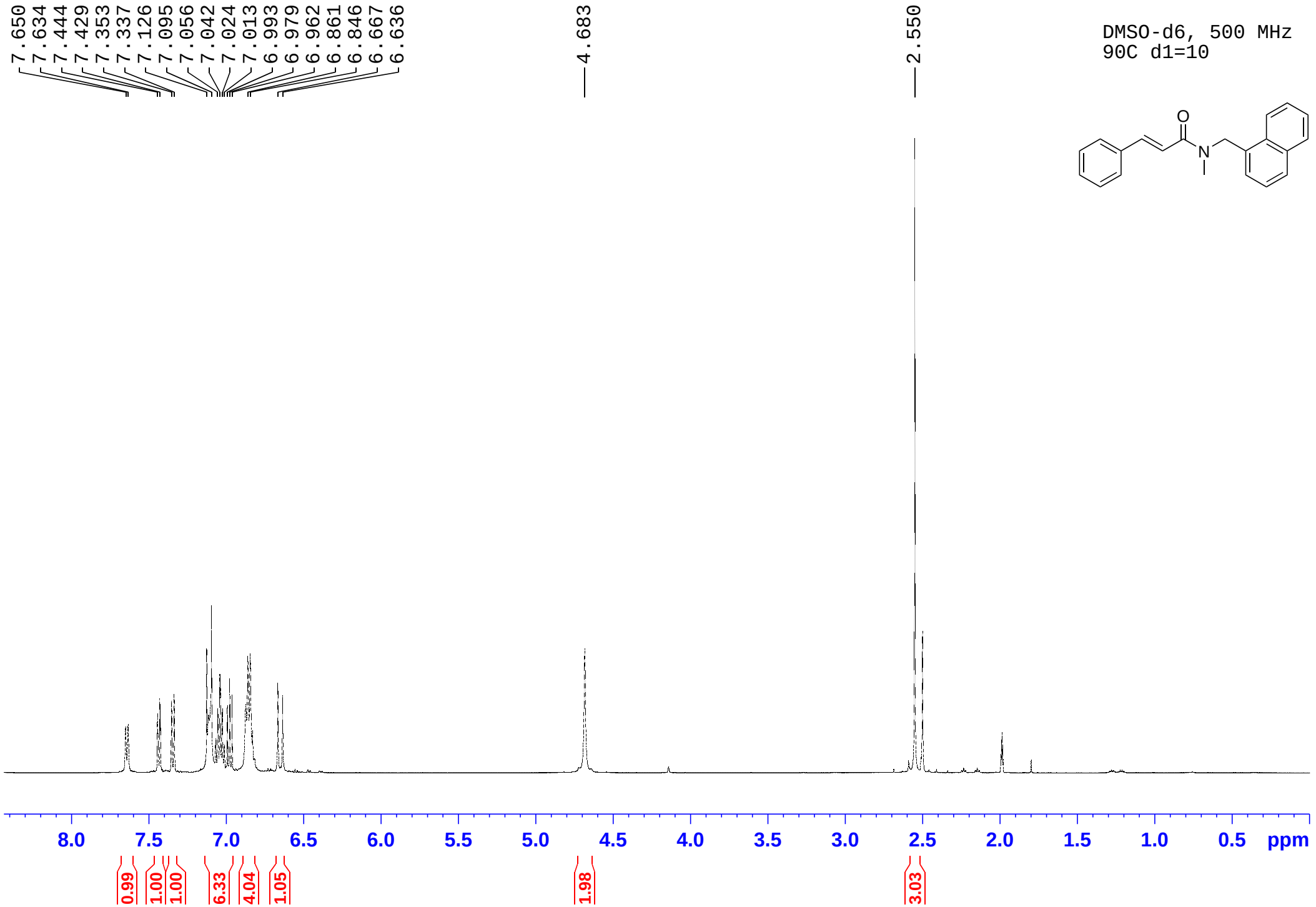
5.021
5.007





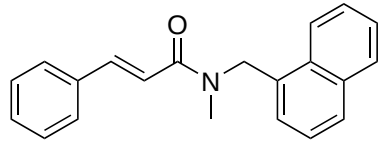
CDCl3
100 MHz



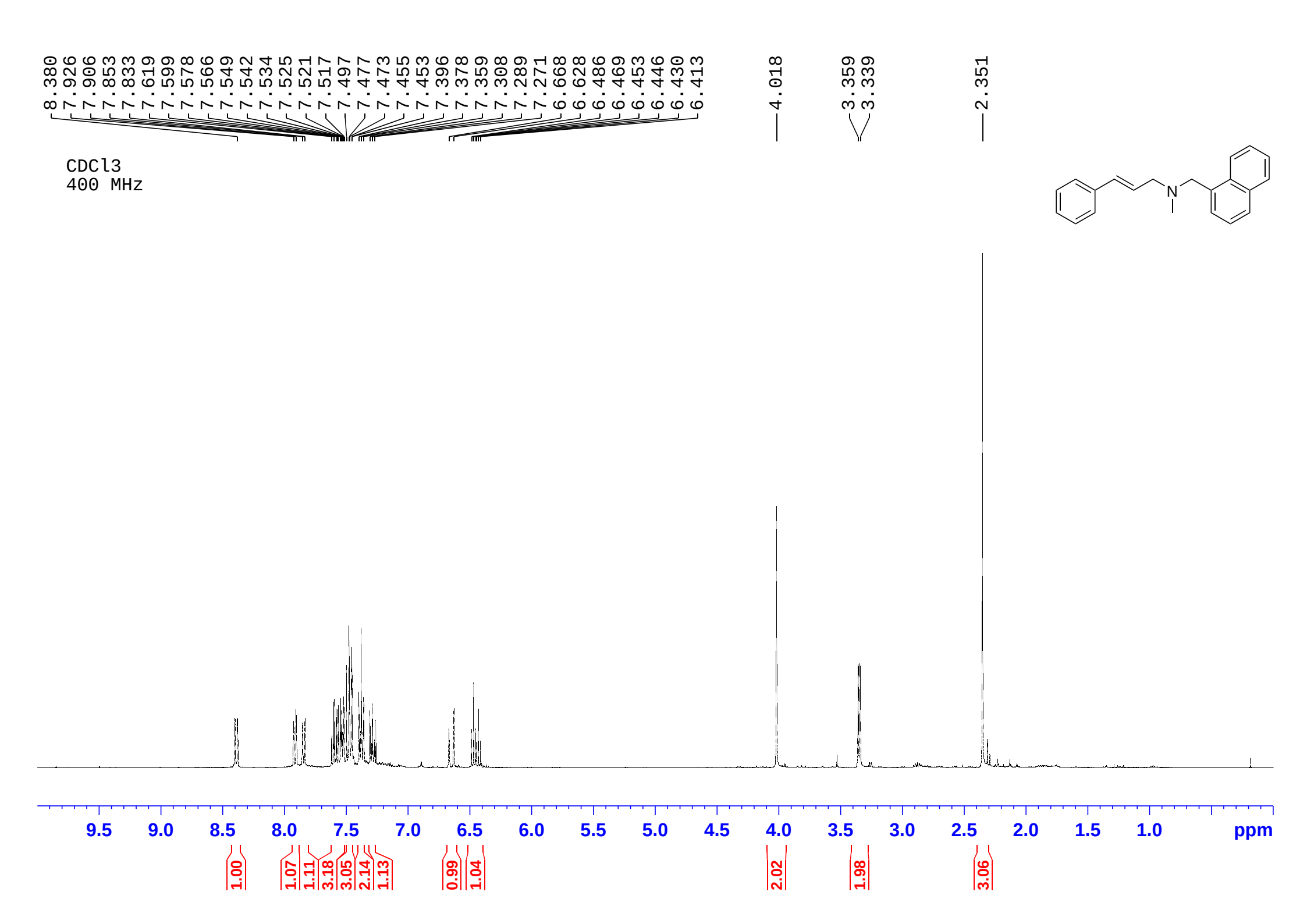

7.650
7.634
7.444
7.429
7.353
7.337
7.126
7.095
7.056
7.042
7.024
7.013
6.993
6.979
6.962
6.861
6.846
6.667
6.636
4.683
2.550
DMSO-d6, 500 MHz
90C d1=10
O
N
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.99
1.00
1.00
6.33
4.04
1.05
1.98
3.03

DMSO-d6, 125 MHz
95C d1=10

165.60
140.90
134.83
133.08
132.55
130.67
128.78
128.12
128.01
127.25
127.19
125.68
125.24
124.83
122.75
118.50
48.46
33.83



190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.380
7.926
7.906
7.853
7.833
7.619
7.599
7.578
7.566
7.549
7.542
7.534
7.525
7.521
7.517
7.497
7.477
7.473
7.455
7.453
7.396
7.378
7.359
7.308
7.289
7.271
6.668
6.628
6.486
6.469
6.453
6.446
6.430
6.413
4.018
3.359
3.339
2.351
CDCl3
400 MHz
N
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
ppm
1.00
1.07
1.11
3.18
3.05
2.14
1.13
0.99
1.04
2.02
1.98
3.06

CDCl3
100 MHz

137.26
135.01
134.01
132.76
132.62
128.65
128.55
128.04
127.74
127.54
127.48
126.43
125.99
125.69
125.23
124.76

60.50
60.23

42.58