An Enantiospecific, Biosynthetically-Inspired Formal Total Synthesis of Liphagal

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Supporting Information

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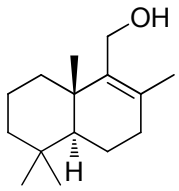
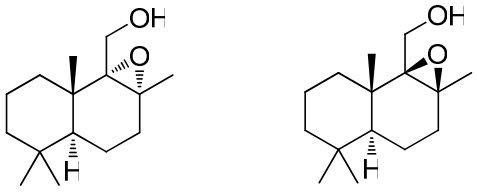
Alcohol **15**



15

A solution of α,β-unsaturated aldehyde **14** (1.00 g, 4.54 mmol) in EtOH (30 mL) was cooled to 0 °C and $NaBH_4$ (0.34 g, 9.08 mmol) was added. The resultant mixture was stirred at 0 °C for 1 h, before being quenched by addition of saturated NH_4Cl aqueous solution (15 mL) and extracted with Et_2O (3 x 30 mL). The combined organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The resultant residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 20:1 as eluent) to give alcohol **15** (0.83 g, 3.73 mmol, 82%) as a white solid. Data for **15**: R_f 0.20 (petrol/EtOAc, 5:1); $[\alpha]_D^{25}$ (c 1.05, $CHCl_3$) +114.6°; IR (KBr disc): 3323, 2941, 1434, 1374, 1301, 999 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 0.85 (s, 3H), 0.89 (s, 3H), 0.97 (s, 3H), 1.13-1.29 (m, 3H), 1.39-1.70 (m, 6H), 1.72 (s, 3H), 1.89 (br d, *J* = 12.9 Hz, 1H), 2.07 (m, 2H, 4.04 (d, *J* = 11.4 Hz, 1H), 4.20 (d, *J* = 11.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 18.85, 18.92, 19.3, 20.7, 21.6, 33.22, 33.24, 33.7, 36.8, 38.0, 41.6, 51.7, 58.3, 132.4, 140.9; HRMS ($C_{15}H_{26}ONa$, ESI): calculated 245.1876 $[M+Na]^+$, found 245.1878.

Epoxides **16** and **17**

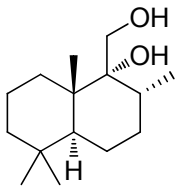


16 **17**

To a solution of alcohol **15** (1.07 g, 4.81 mmol) in CH_2Cl_2 (30 mL) was added *m*CPBA (70%, 1.78 g, 7.22 mmol). The reaction mixture was stirred at 0 °C for 1 h, then diluted with H_2O (20 mL). The organic layer was separated and the aqueous phase extracted with Et_2O (3 x 20 mL). The combined organics were washed with saturated aqueous $NaHCO_3$ solution (30 mL) and brine (30 mL), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The reside was purified by flash chromatography on silica gel (hexanes/EtOAc, 20:1→10:1) as eluent) to give alcohol **16** (0.79 g, 3.31 mmol, 69%). Data for **16**: R_f 0.25 (petrol/EtOAc, 3:1); $[\alpha]_D^{25}$ (c 0.85, $CHCl_3$) +58.2°; IR (film): 3482, 2926, 1459, 1380, 1241, 1049 cm^{-1}; ^1H NMR

(400 MHz, $CDCl_3$): 0.80 (s, 3H), 0.84 (s, 3H), 0.97 (s, 3H), 1.12-1.43 (m, 6H), 1.30 (s, 3H), 1.54 (m, 2H), 1.82 (m, 2H), 1.97 (m, 1H), 3.56 (d, *J* = 10.9 Hz, 1H), 3.88 (d, *J* = 10.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 16.1, 17.1, 18.3, 21.4, 21.5, 29.3, 32.9, 33.4, 33.9, 37.1, 41.3, 43.1, 56.9, 64.5, 71.2; HRMS ($C_{15}H_{26}O_2Na$, ESI): calculated 261.1825 $[M+Na]^+$, found 261.1825. Further elution gave alcohol **17** (0.25 g, 22%). Data for **17**: R_f 0.15 (petrol/EtOAc, 3:1); $[\alpha]_D^{25}$ (c 1.05, $CHCl_3$) +35.6°; IR (film): 3467, 2951, 1464, 1375, 1241, 1048 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 0.79 (s, 3H), 0.84 (s, 3H), 0.85 (m, 1H), 1.08 (s, 3H), 1.11-1.26 (m, 2H), 1.32-1.74 (m, 7H), 1.37 (s, 3H), 1.88 (m, 1H), 2.09 (m, 1H), 3.74 (d, *J* = 11.0 Hz, 1H), 3.84 (d, *J* = 11.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 16.68, 16.71, 19.4, 20.0, 21.8, 33.1, 33.7, 35.4, 35.8, 37.6, 41.3, 53.6, 61.1, 64.9, 71.8; HRMS ($C_{15}H_{26}O_2Na$, ESI): calculated 261.1825 $[M+Na]^+$, found 261.1825.

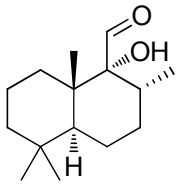
Diol **18**



18

To a solution of epoxide **16** (270 mg, 1.13 mmol) in anhydrous THF (20 mL) at room temperature was added $LiAlH_4$ (2.0 M in THF, 2.27 mmol, 4.54 mmol). The reaction mixture was heated at 60 °C for 1 h. The reaction mixture was then cooled to room temperature and quenched by careful dropwise addition of EtOAc (5 mL) followed by 1N aqueous HCl (5 mL). The mixture was extracted with EtOAc (3 x 20 mL). The combined organics were washed with water (30 mL) and brine (30 mL), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The resultant residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 10:1 as eluent) to give **18** (236 mg, 0.98 mmol, 87%) as a colourless oil. Data for **18**: R_f 0.50 (petrol/EtOAc, 2:1); $[\alpha]_D^{25}$ (c 0.65, $CHCl_3$) +5.5°; IR (KBr disc): 3374, 2936, 1459, 1380, 1262, 1145, 978 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 0.82 (s, 3H), 0.87 (s, 3H), 0.88 (s, 3H), 0.89 (d, *J* = 6.8 Hz, 3H), 1.18 (m, 1H), 1.28-1.58 (m, 10H), 1.78 (m, 1H), 2.15 (t, *J* = 5.6 Hz, 1H), 2.46 (s, 1H), 3.48 (dd, *J* = 11.1, 5.8 Hz, 1H), 3.72 (dd, *J* = 11.0, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 15.6, 16.4, 18.6, 21.6, 22.0, 31.3, 31.9, 32.3, 33.6, 35.6, 41.6, 41.9, 46.4, 63.5, 75.5; HRMS ($C_{15}H_{28}O_2Na$, ESI): calculated 263.1982 $[M+Na]^+$, found 263.1985.

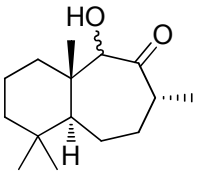
Aldehyde **19**



19

DMSO (0.10 mL, 1.45 mmol) was dissolved in anhydrous CH_2Cl_2 (3 mL) and the resultant solution was cooled to -78 °C. Oxalyl chloride (0.06 mL, 0.72 mmol) was added, and the solution was stirred at -78 °C for 10 min. A solution of diol **18** (58 mg, 0.241 mmol) in anhydrous CH_2Cl_2 (3 mL) was then added dropwise and the reaction mixture was allowed to stir at -78 °C for a further 10 min. Et_3N (0.33 mL, 2.40 mL) was then added, and the reaction mixture was allowed to warm to room temperature over 30 min before being quenched with H_2O (5 mL). The product was then extracted with CH_2Cl_2 (3 x 10 mL), and the combined organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The resultant residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 20:1 as eluent) to give **19** (51 mg, 0.214 mmol, 89%) as a colourless oil. Data for **19**: R_f 0.55 (petrol/EtOAc, 5:1); $[\alpha]_D^{25}$ (c 0.55, $CHCl_3$) –30.7°; IR (film): 3498, 2939, 1714, 1461, 1365, 1153, 1053, 804 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 0.63 (d, J = 6.6 Hz, 3H), 0.85 (s, 3H), 0.85 (m, 1H), 0.87 (s, 3H), 1.17 (m, 1H), 1.27 (s, 3H), 1.32-1.66 (m, 9H), 2.38 (m, 1H), 3.27 (s, 1H), 9.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 15.6, 17.1, 18.2, 21.3, 22.1, 30.4, 30.6, 33.1, 33.3, 33.5, 41.5, 42.4, 45.0, 83.8, 207.7; HRMS ($C_{15}H_{26}O_2Na$, ESI): calculated 261.1825 $[M+Na]^+$, found 261.1827.

α-Hydroxy ketone **22**

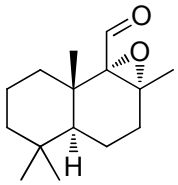


22

Aryl bromide **20** (266 mg, 0.84 mmol) was dissolved in anhydrous THF (2 mL) and the resultant solution was cooled to -78 °C. *t*-BuLi (1.7M in pentane, 0.52 mL, 0.84 mmol) was added dropwise, and the reaction mixture was stirred at -78 °C for 30 min. A solution of aldehyde **19** (50 mg, 0.21 mmol) in anhydrous THF (2 mL) was then added dropwise. The reaction mixture was stirred at -78 °C for a further 10 min, then allowed to warm to room temperature over 30 min. The reaction mixture was quenched by addition of a saturated

NH_4Cl aqueous solution (5 mL) and extracted with Et_2O (3 x 10 mL). The combined organics were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 10:1 as eluent) to give **22** (20 mg, 40%) as a colourless oil. Data for **22**: R_f 0.40 (petrol/EtOAc, 10:1); $[\alpha]_D^{25}$ (c 0.52, $CHCl_3$) +7.5°; IR (film): 3448, 2930, 1695, 1460, 1380, 1085 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): 2.12 (m, 0.6H, 2.47 (m, 1H), 2.75 (br d, *J* = 6.3 Hz, 0.6H), 2.91 (br d, *J* = 5.4 Hz, 0.4H), 4.13 (br s, 0.6H), 4.26 (br s, 0.4H); ^{13}C NMR (125 MHz, $CDCl_3$): 16.0, 18.36, 18.42, 18.6, 19.7, 19.9, 21.4, 21.69, 21.74, 21.8, 28.7, 32.26, 32.29, 32.6, 32.8, 33.7, 34.4, 34.6, 41.4, 41.6, 42.0, 42.5, 42.6, 45.6, 49.4, 52.2, 82.4, 84.7, 216.4, 217.0; HRMS ($C_{15}H_{26}O_2Na$, ESI): calculated 261.1825 $[M+Na]^+$, found 261.1829.

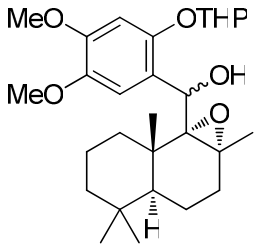
Aldehyde **23**



23

Alcohol **16** (0.79 g, 3.31 mmol) was dissolved in anhydrous DMSO (15 mL) and the resultant solution cooled to 0 °C. The reaction mixture was stirred at 0 °C for 1 h, then quenched by addition of a saturated $Na_2S_2O_3$ aqueous solution (10 mL) and extracted with Et_2O (3 x 10 mL). The combined organic extracts were washed with a saturated $NaHCO_3$ solution (10 mL) and brine (10 mL), then dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 30:1 as eluent) to give **23** (0.65 g, 2.75 mmol, 83%) as a colourless oil. Data for **23**: R_f 0.60 (petrol/EtOAc, 10:1); $[\alpha]_D^{25}$ (c 1.09, $CHCl_3$) -10.9°; IR (film): 2927, 1726, 1460, 1382, 1073, 832 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 0.84 (s, 6H), 1.18 (m, 1H), 1.25 (s, 3H), 1.32-1.66 (m, 8H), 1.37 (s, 3H), 1.86 (m, 1H), 1.99 (m, 1H), 9.70 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 16.8, 17.1, 18.1, 21.3, 21.4, 28.6, 32.8, 33.4, 35.2, 36.5, 41.3, 42.0, 64.3, 75.3, 201.7; HRMS ($C_{15}H_{24}O_2Na$, ESI): calculated 259.1669 $[M+Na]^+$, found 259.1663.

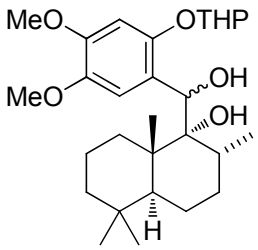
Benzylic alcohol **24**



24

Aryl bromide **20** (671 mg, 2.116 mmol) was dissolved in anhydrous THF (10 mL) and the resultant solution was cooled to -78 °C. *t*-BuLi (1.7M in pentane, 1.49 mL, 2.54 mmol) was added dropwise, and the reaction mixture was stirred at -78 °C for 30 min. A solution of aldehyde **23** (200 mg, 0.846 mmol) in anhydrous THF (5 mL) was then added dropwise. The reaction mixture was stirred at -78 °C for a further 10 min, then allowed to warm to room temperature over 30 min. The reaction mixture was quenched by addition of a saturated NH_4Cl aqueous solution (5 mL) and extracted with Et_2O (3 x 20 mL). The combined organics were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 5:1→3:1 as eluent) to give **24** (378 mg, 94%) as a white foam. ^1H NMR showed a complex mixture of four diasteroisomers so **24** was not characterised fully. Partial data for **24**: R_f 0.40 (petrol/EtOAc, 2:1); HRMS ($C_{28}H_{42}O_6Na$, ESI): calculated 497.2874 $[M+Na]^+$, found 497.2872.

Diol **10**

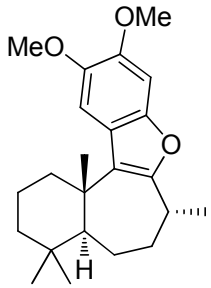


10

To a solution of **24** (200 mg, 0.421 mmol) in anhydrous THF (10 mL) was added $LiAlH_4$ (2.0 M in THF, 2.11 mL, 4.21 mmol) at room temperature. The reaction mixture was then warmed to 60 C and stirred at this temperature for 1 h. The reaction mixture was then cooled to room temperature and quenched by careful dropwise addition of EtOAc (5 mL) followed by a saturated NH_4Cl aqueous solution (10 mL). The mixture was extracted with EtOAc (3 x 20 mL). The combined organics were washed with water (30 mL) and brine (30 mL), dried over $MgSO_4$, filtered and concentrated under reduced pressure to give **10** (210 mg) as a white

foam, which was used in the next step without further purification. Partial data for **24**: R_f 0.40 (petrol/EtOAc, 2:1); HRMS ($C_{28}H_{44}O_6Na$, ESI): calculated 499.3030 $[M+Na]^+$, found 499.3029.

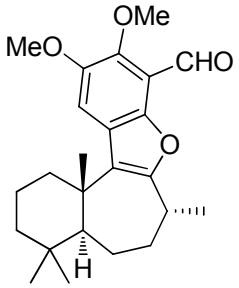
Benzofuran **7**



7

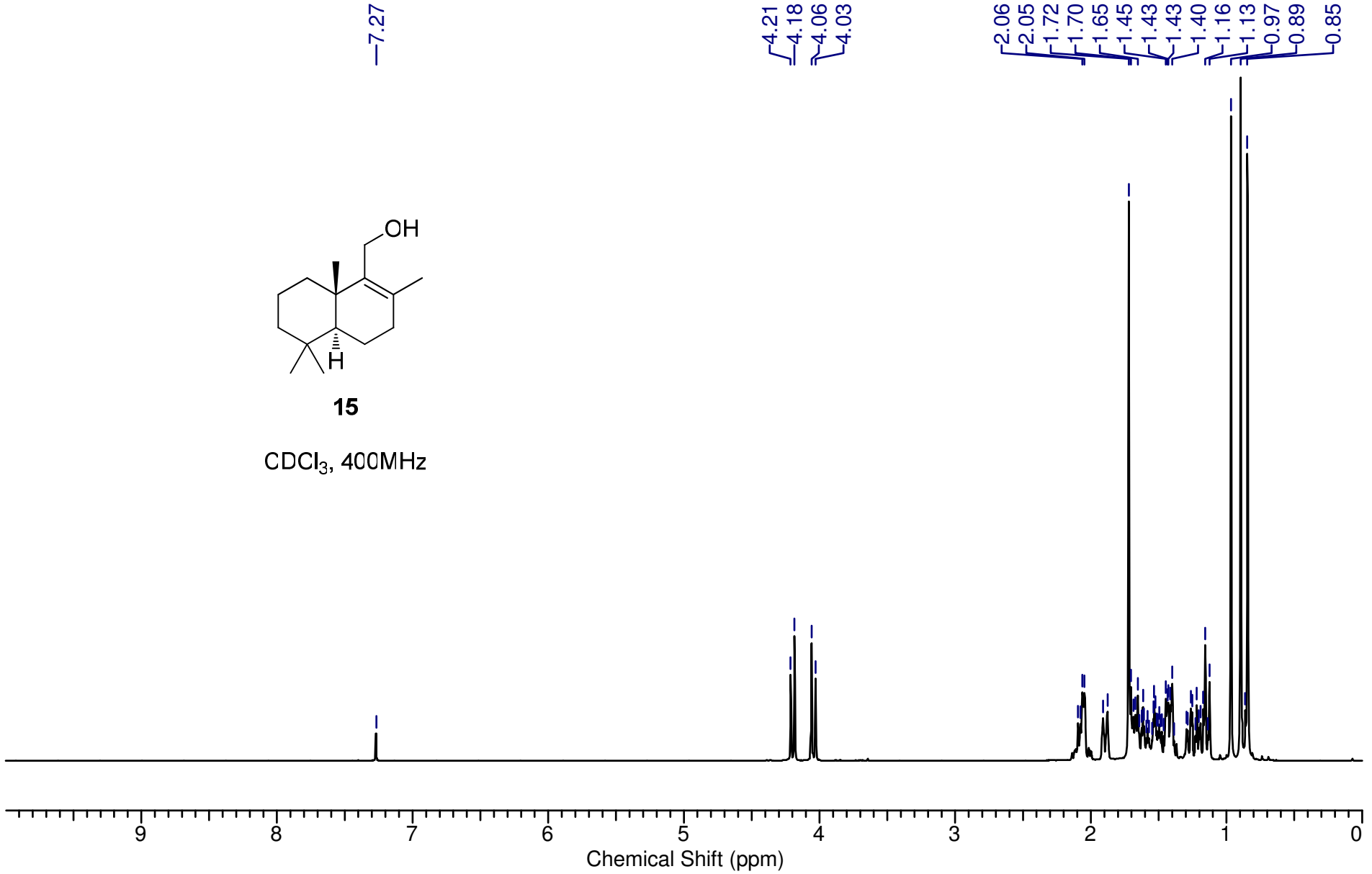
To a solution of diol **10** (210 mg) in CH_2Cl_2 (10 mL) at -78 °C was added TFA (0.16 mL, 2.11 mmol). The reaction mixture was allowed to gradually warm to room temperature over 30 min. A dark red solution was formed. The reaction mixture was quenched with a saturated $NaHCO_3$ aqueous solution (5 mL) and extracted with CH_2Cl_2 (3 x 10 mL). The combined organics were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 20:1 as eluent) to give **7** (110 mg, 0.309 mmol, 73% over 2 steps) as a white solid. Data for **7**: R_f 0.25 (petrol/EtOAc, 10:1); $[\alpha]_D^{25}$ (c 0.83, $CHCl_3$) +14.3°; IR (KBr disc): 2931, 1624, 1488, 1390, 1318, 1213, 1137 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 0.96 (s, 3H), 0.99 (s, 3H), 1.27 (m, 1H), 1.39 (s, 3H), 1.42 (d, *J* = 7.1 Hz, 3H), 1.47-1.89 (m, 8H), 2.17 (td, *J* = 6.5, 3.2 Hz, 1H), 2.61 (app br d, *J* = 11.6 Hz, 1H), 3.19 (sxt, *J* = 7.0 Hz, 1H), 3.90 (s, 3H), 3.92 (s, 3H), 6.94 (s, 1H), 7.17 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 18.9, 20.2, 21.9, 22.0, 24.3, 33.3, 33.6, 34.8, 35.2, 39.5, 40.3, 42.0, 53.6, 56.1, 57.0, 94.9, 105.5, 120.3, 125.3, 144.8, 146.8, 148.4, 155.8; HRMS ($C_{23}H_{32}O_3Na$, ESI): calculated 379.2244 $[M+Na]^+$, found 379.2242.

Dimethyl-liphagal **26**



26

Benzofuran **7** (80 mg, 0.224 mmol) was dissolved in anhydrous THF (5 mL) and the resultant solution was cooled to 0 °C. TMEDA (0.07 mL, 0.449 mmol) and *n*-BuLi (1.6 M, 0.28 mL, 0.449 mmol) were added dropwise, and the resultant pale yellow solution was stirred at 0 °C for 30 min. DMF (0.17 mL, 2.24 mmol) was then added dropwise, and the reaction mixture was allowed to warm to room temperature over 20 min. The mixture was quenched with a saturated NH_4Cl aqueous solution (5 mL) and extracted with Et_2O (3 x 10 mL). The combined organics were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexanes/EtOAc, 20:1 as eluent) to give **26** (75 mg, 0.195 mmol, 87%) as a white solid. Data for **26**: R_f 0.20 (petrol/EtOAc, 10:1); $[\alpha]_D^{25}$ (c 0.89, $CHCl_3$) +11.1°; IR (KBr disc): 2932, 2864, 1695, 1606, 1468, 1389, 1331, 1242, 1124, 1052, 980 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 0.95 (s, 3H), 0.99 (s, 3H), 1.26 (td, *J* = 13.4, 3.3 Hz), 1.37 (s, 3H), 1.47 (d, *J* = 7.1 Hz, 3H), 1.49-1.62 (m, 6H), 1.72 (m, 1H), 1.85 (m, 1H), 2.18 (m, 1H), 2.55 (app br d, *J* = 13.1 Hz, 1H), 3.31 (sxt, *J* = 6.9 Hz, 1H), 3.94 (s, 3H), 3.97 (s, 3H), 7.48 (s, 1H), 10.56 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 18.9, 20.2, 22.0, 22.1, 24.0, 33.3, 33.5, 34.8, 39.4, 40.3, 41.9, 53.4, 57.3, 62.8, 113.0, 114.8, 124.6, 125.3, 146.3, 147.9, 149.5, 158.8, 188.4; HRMS ($C_{24}H_{32}O_4Na$, ESI): calculated 407.2193 $[M+Na]^+$, found 407.2190.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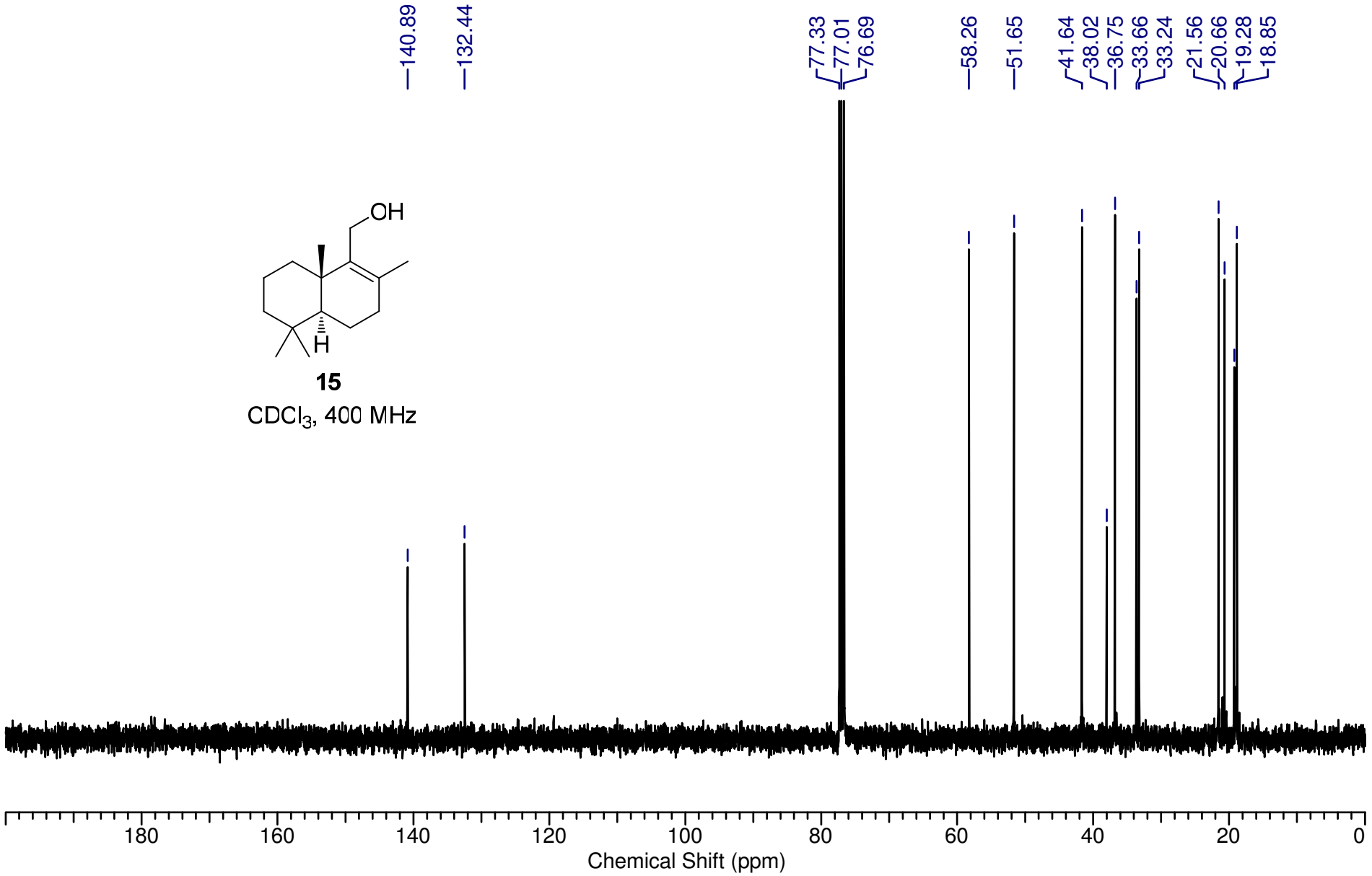
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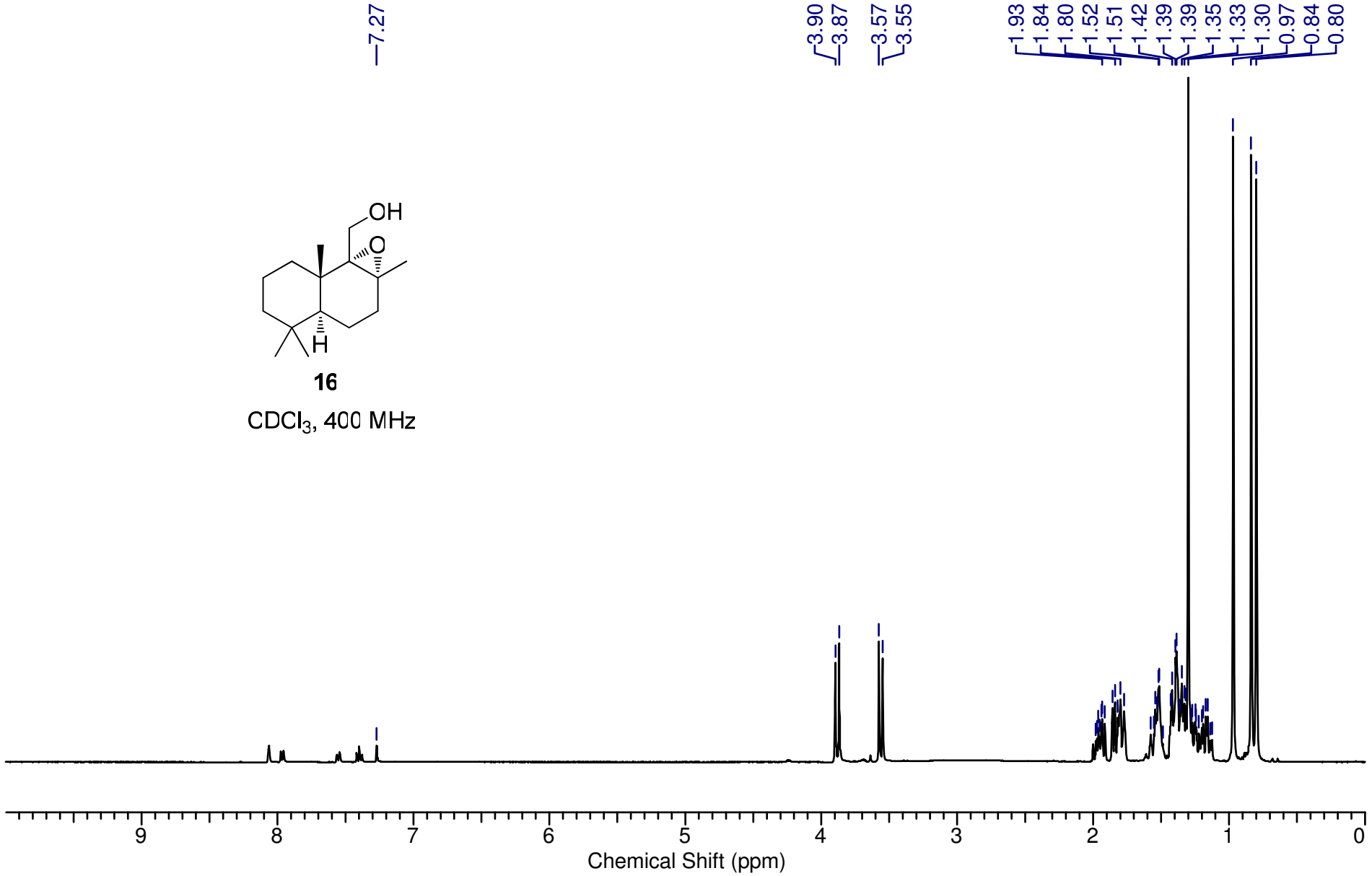
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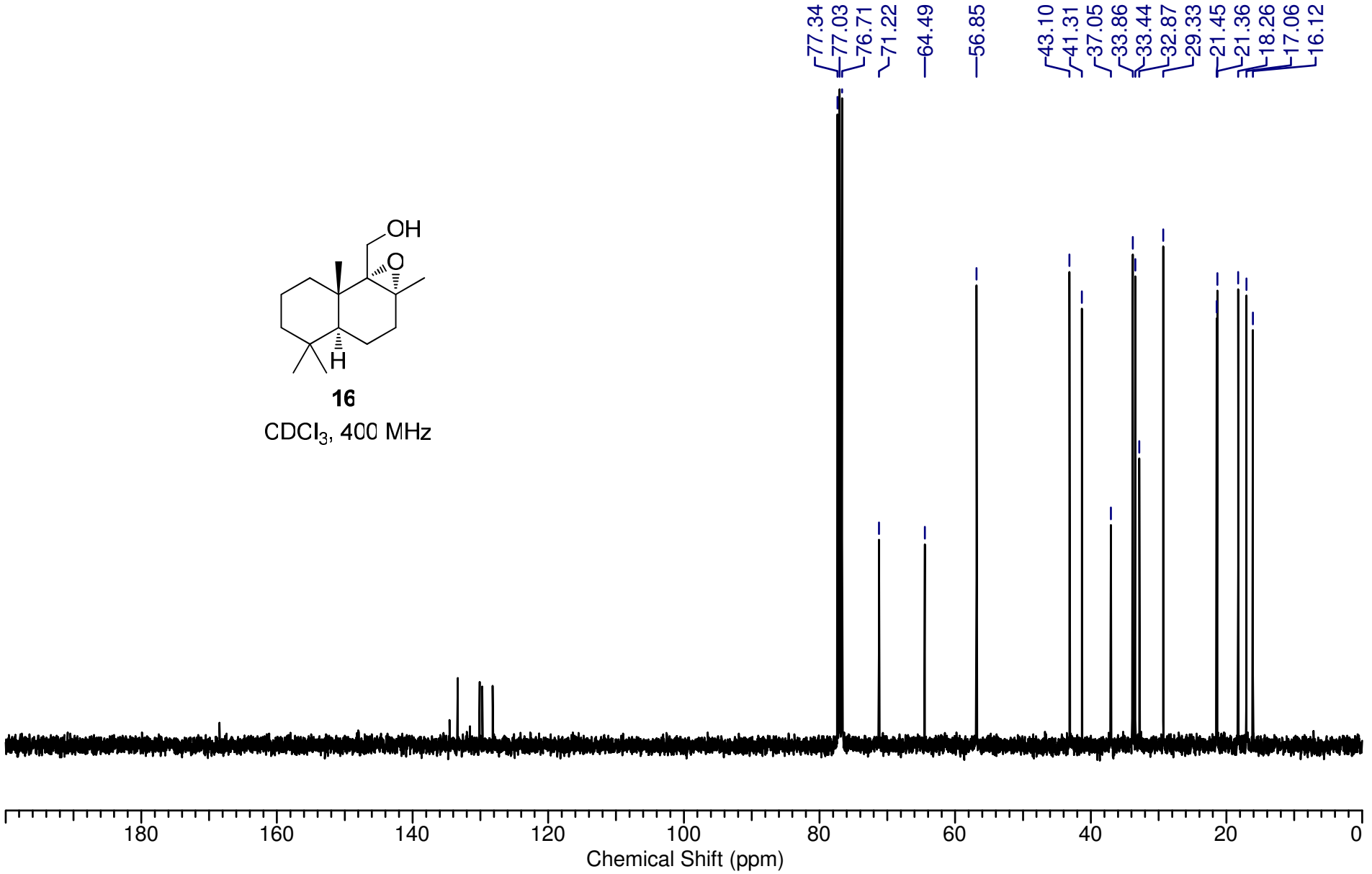
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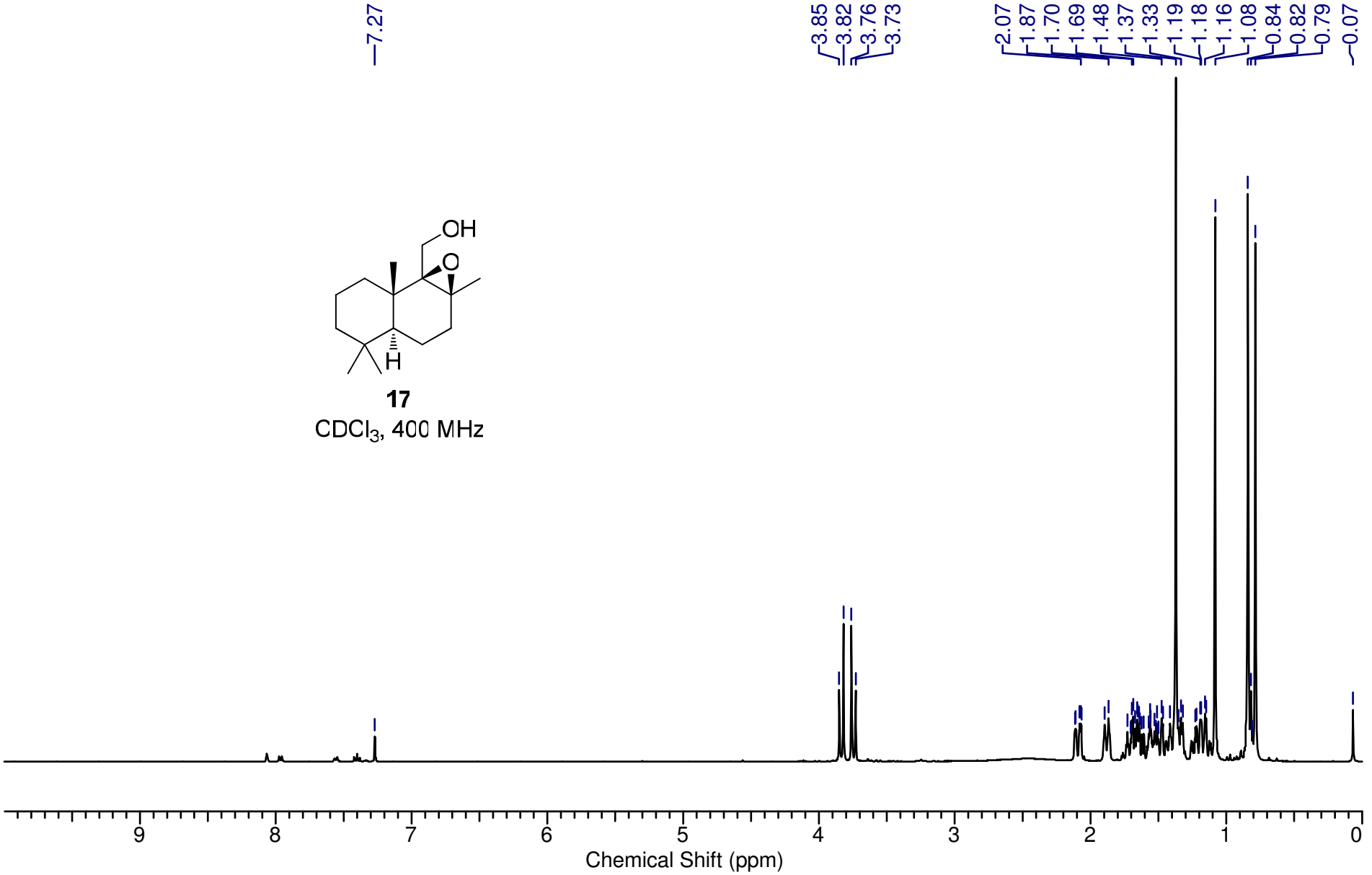
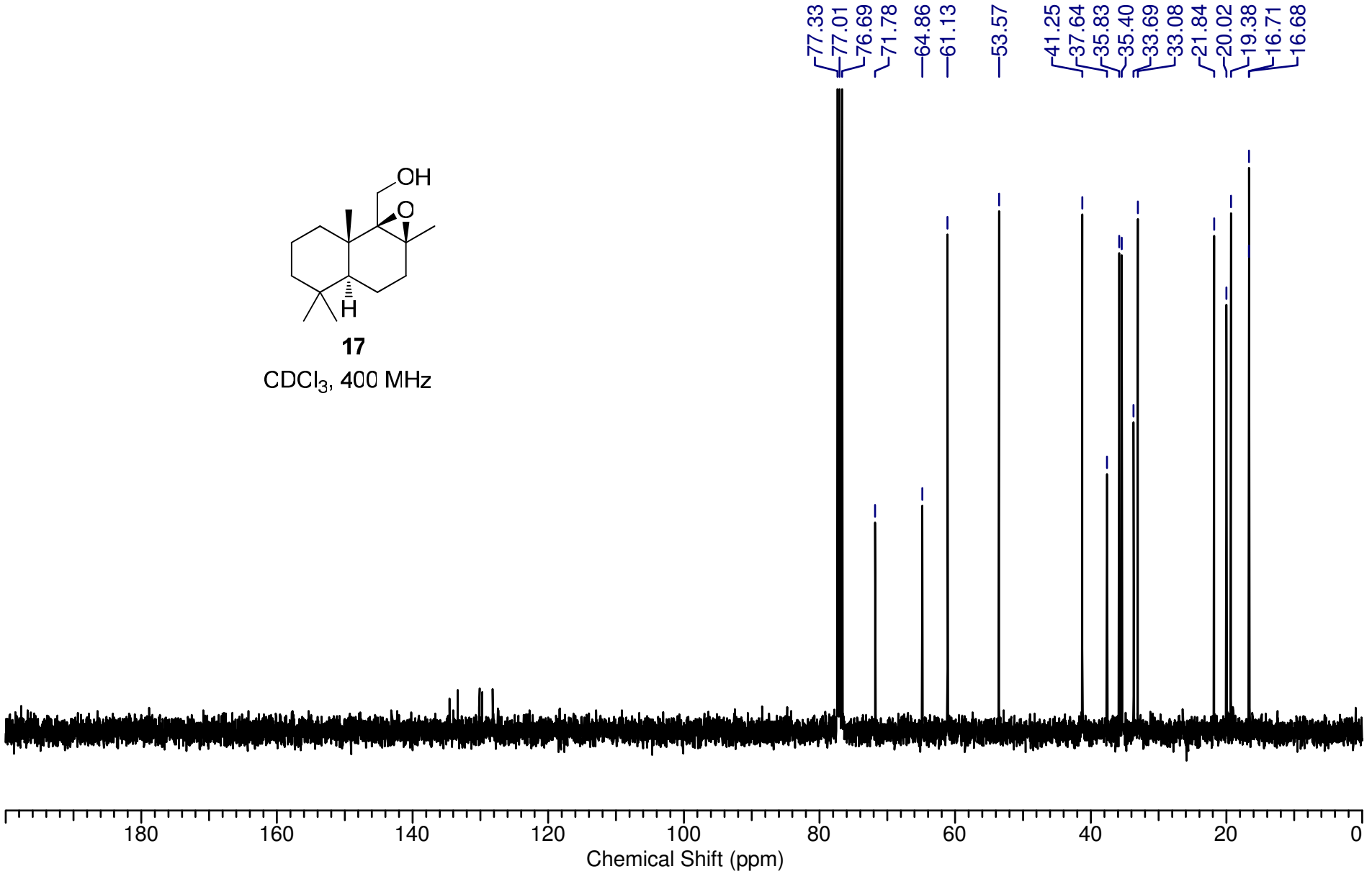
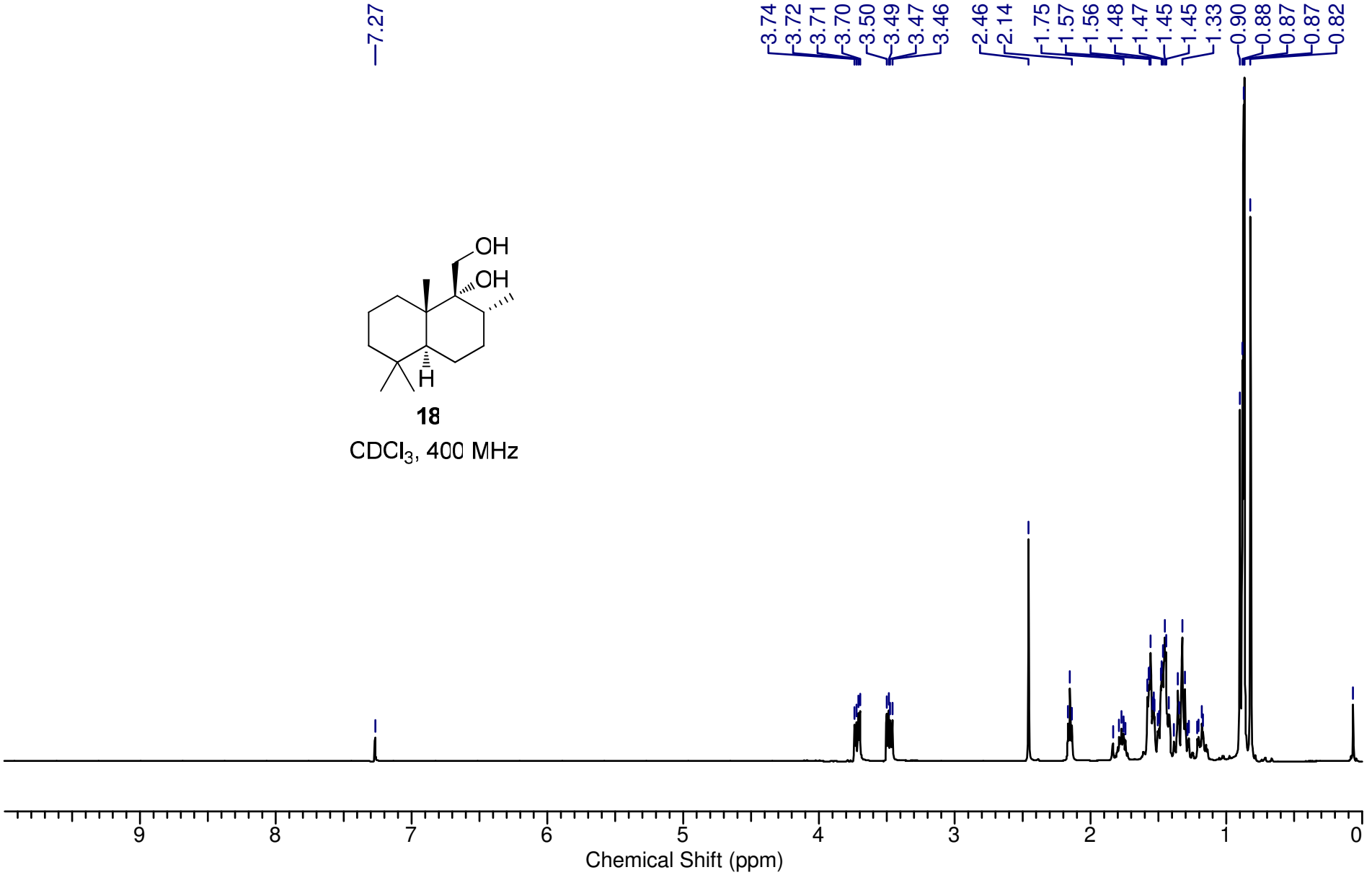
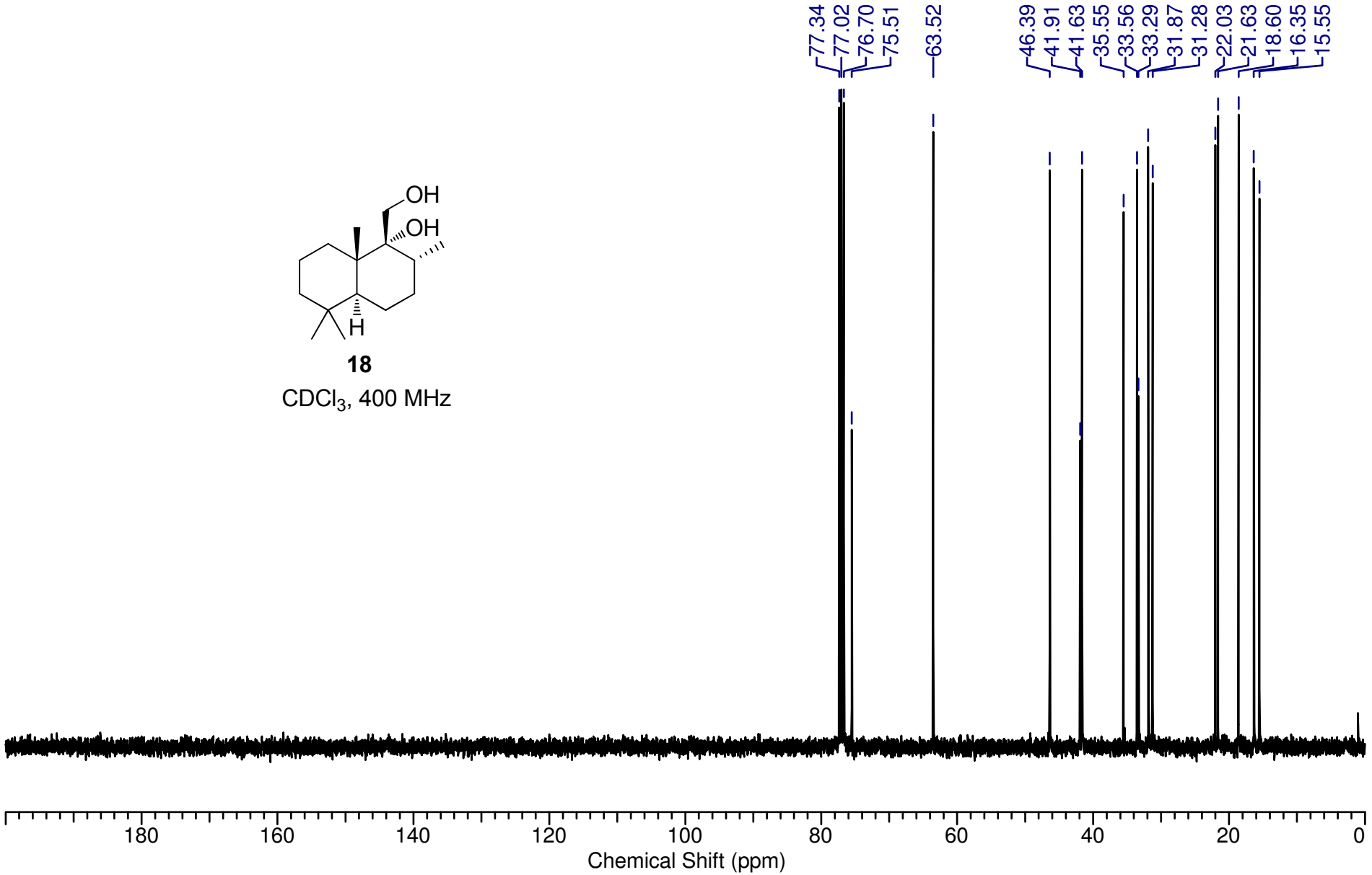


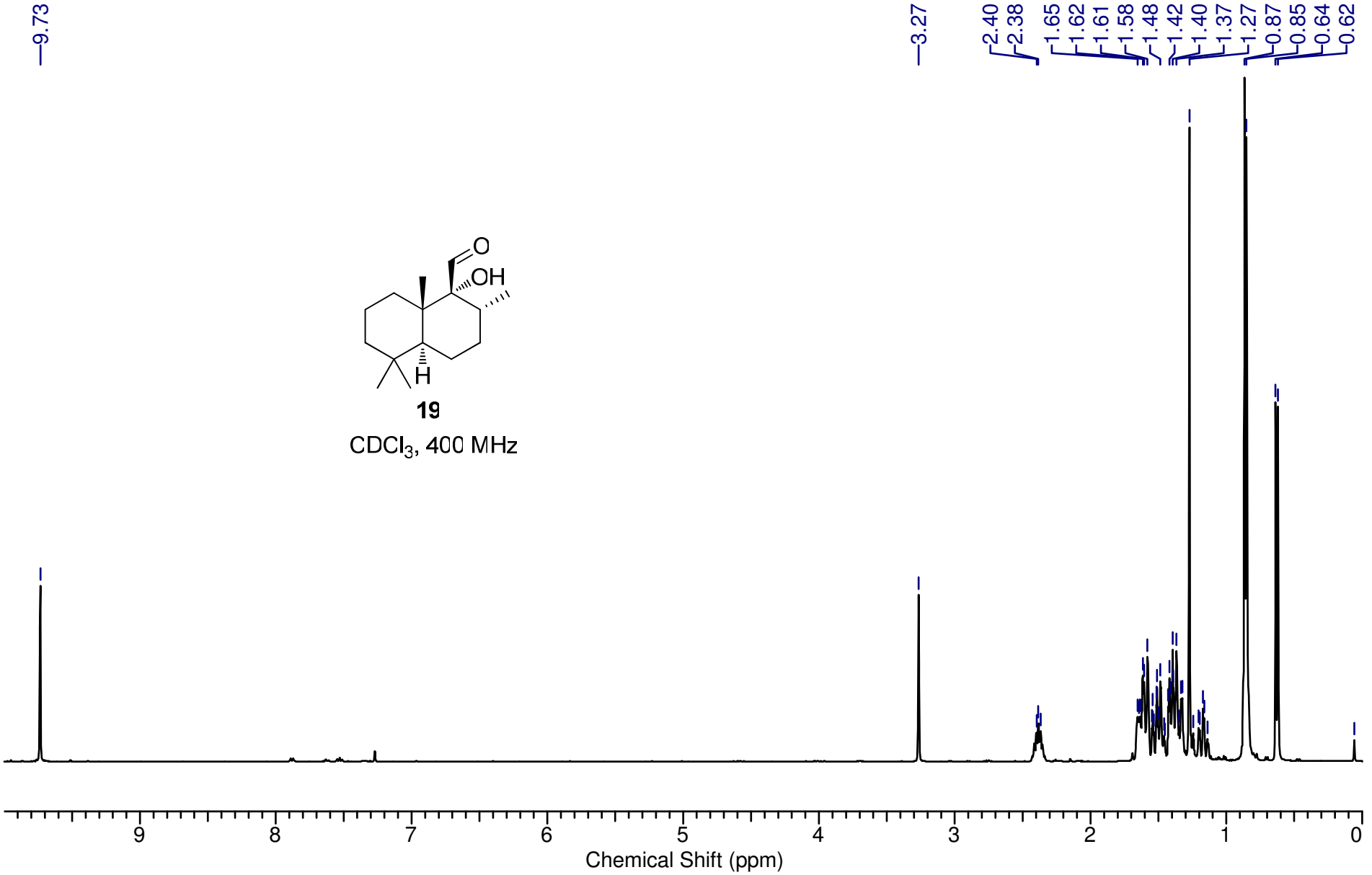
17

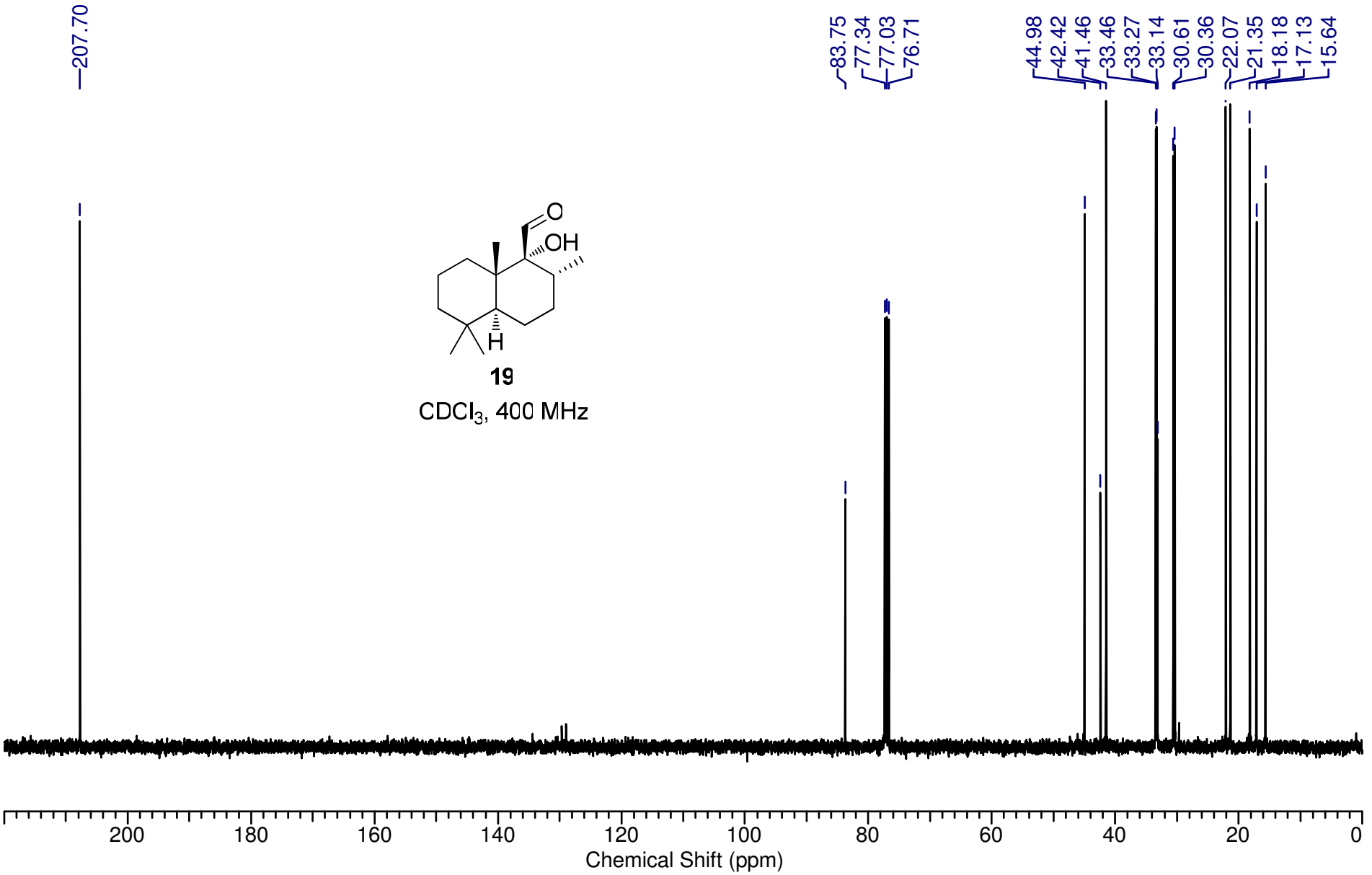
$CDCl_3$, 400 MHz

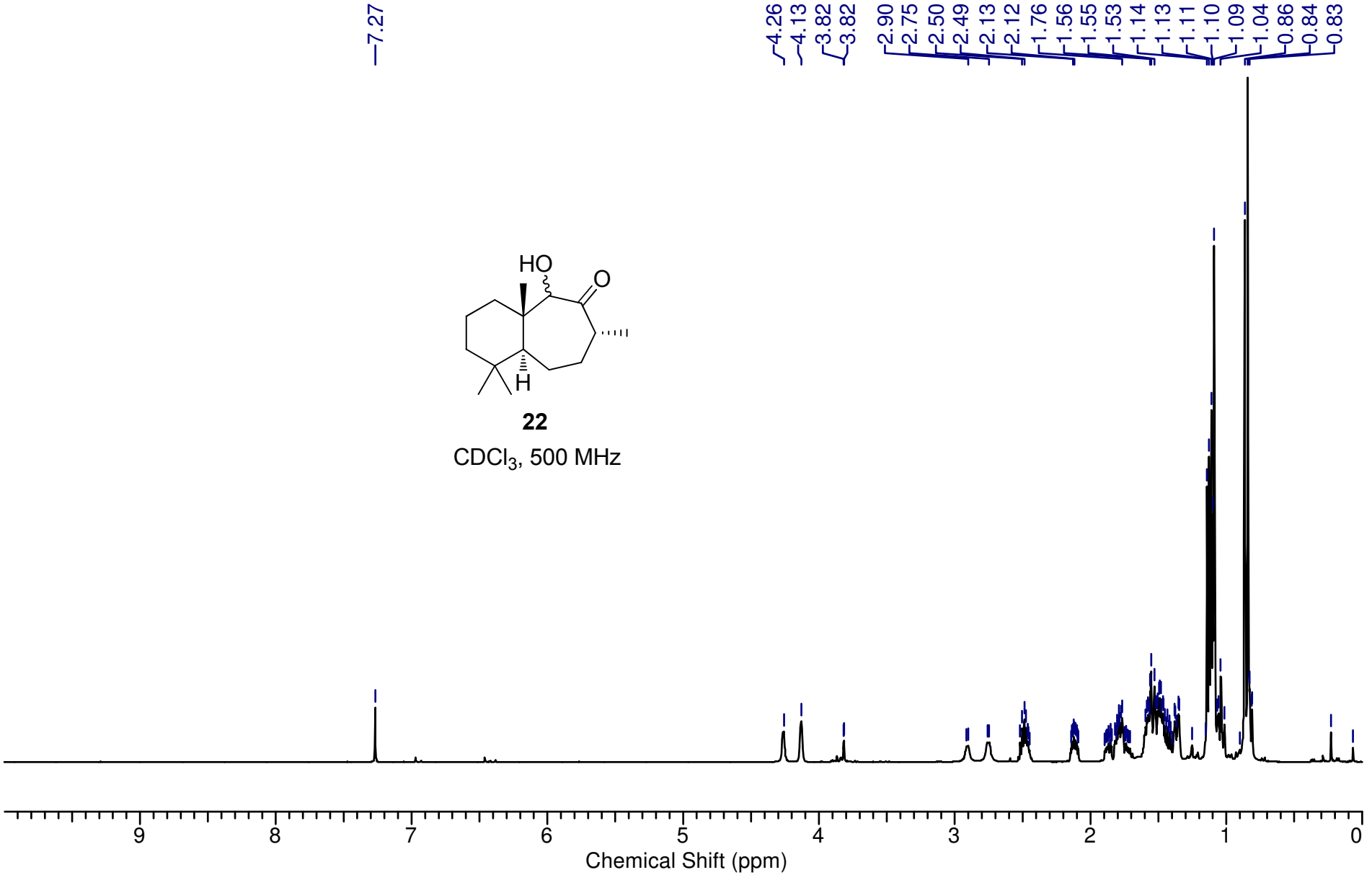

77.33
77.01
76.69
71.78
64.86
61.13
53.57
41.25
37.64
35.83
35.40
33.69
33.08
21.84
20.02
19.38
16.71
16.68
OH
O
H
17
CDCl3, 400 MHz
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

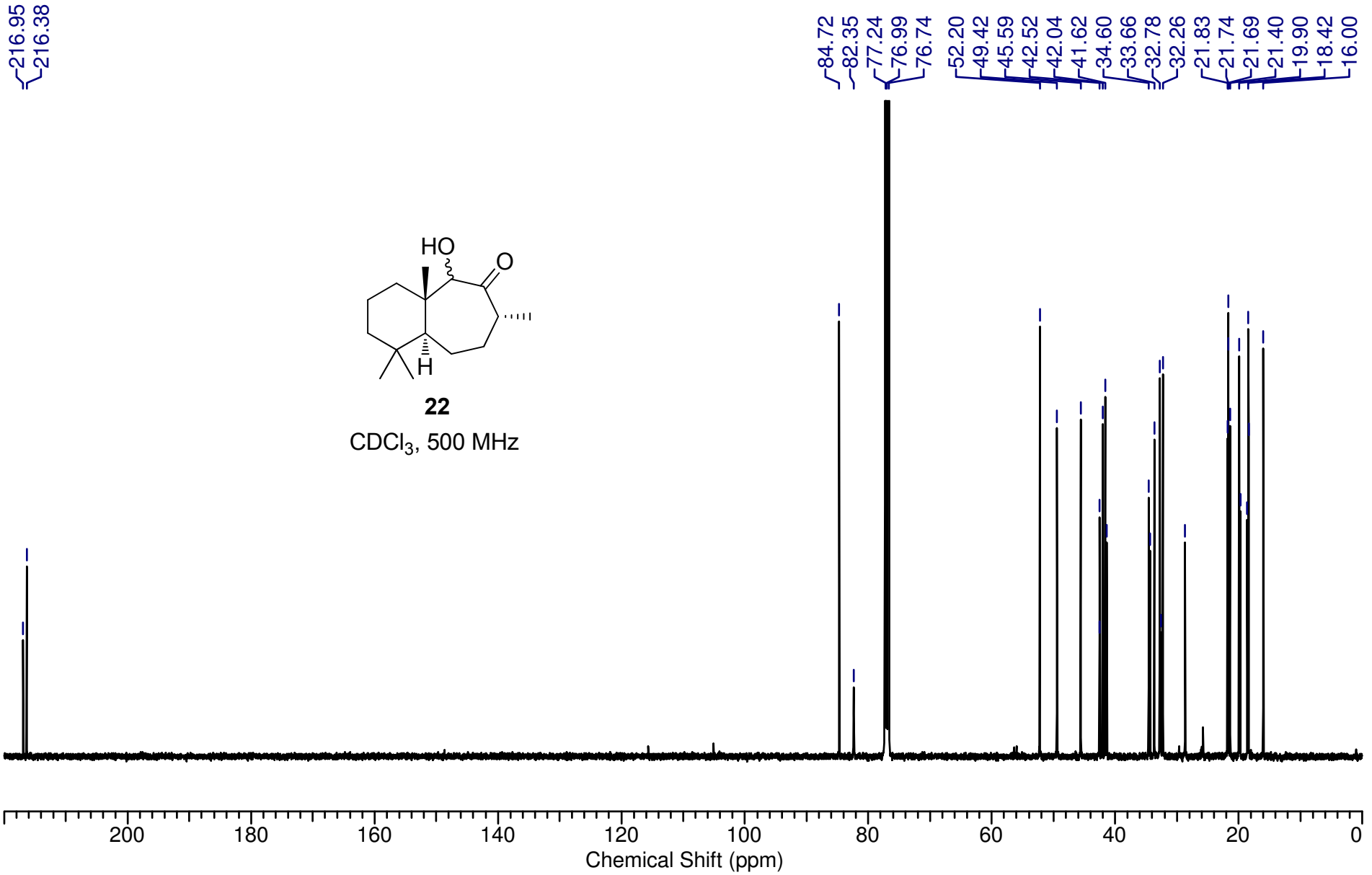

7.27
3.74
3.72
3.71
3.70
3.50
3.49
3.47
3.46
2.46
2.14
1.75
1.57
1.56
1.48
1.47
1.45
1.45
1.33
0.90
0.88
0.87
0.87
0.82
OH
OH
H
18
CDCl3, 400 MHz
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

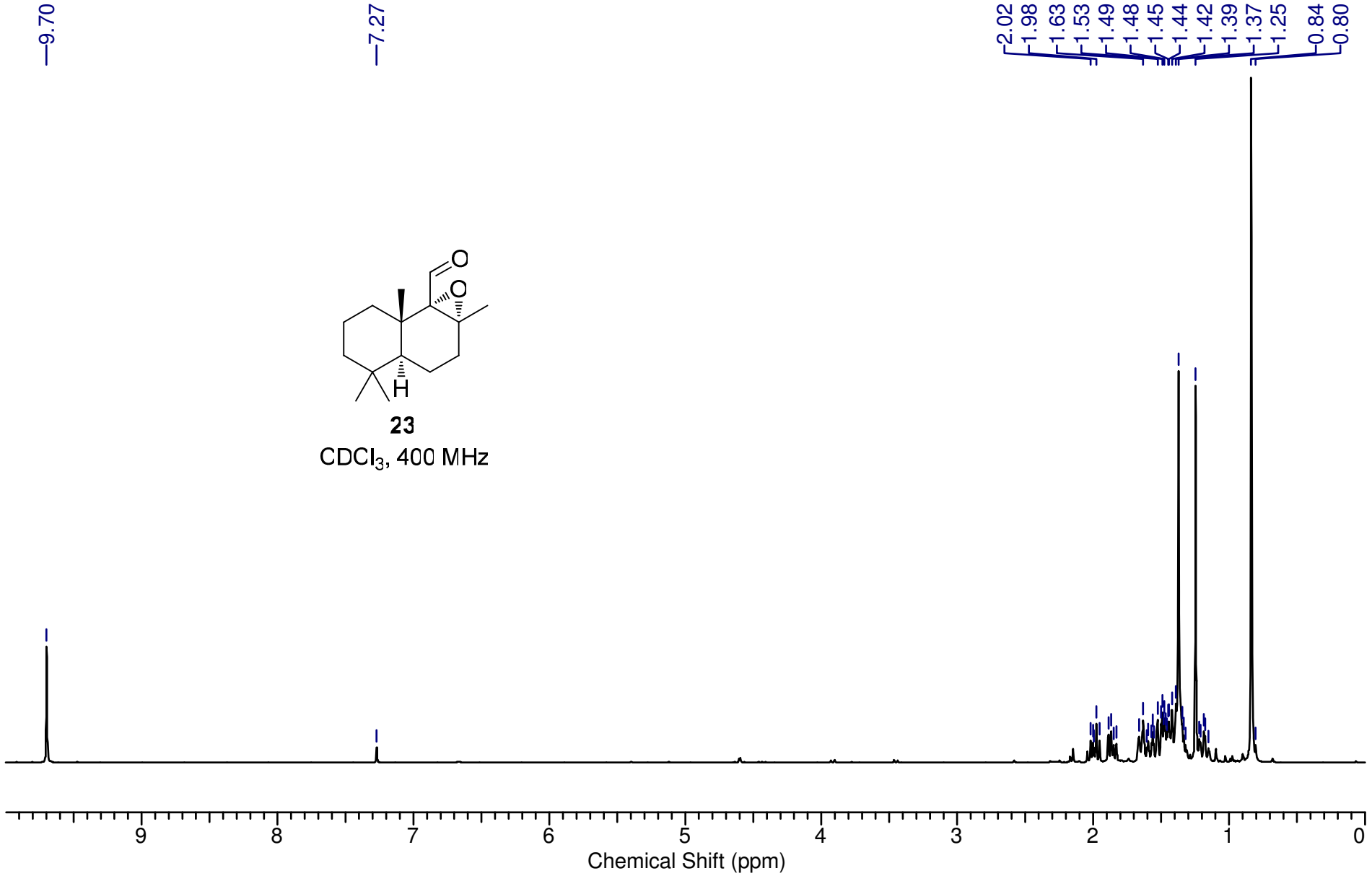

77.34
77.02
76.70
75.51
63.52
46.39
41.91
41.63
35.55
33.56
33.29
31.87
31.28
22.03
21.63
18.60
16.35
15.55
OH
OH
H
18
CDCl3, 400 MHz
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

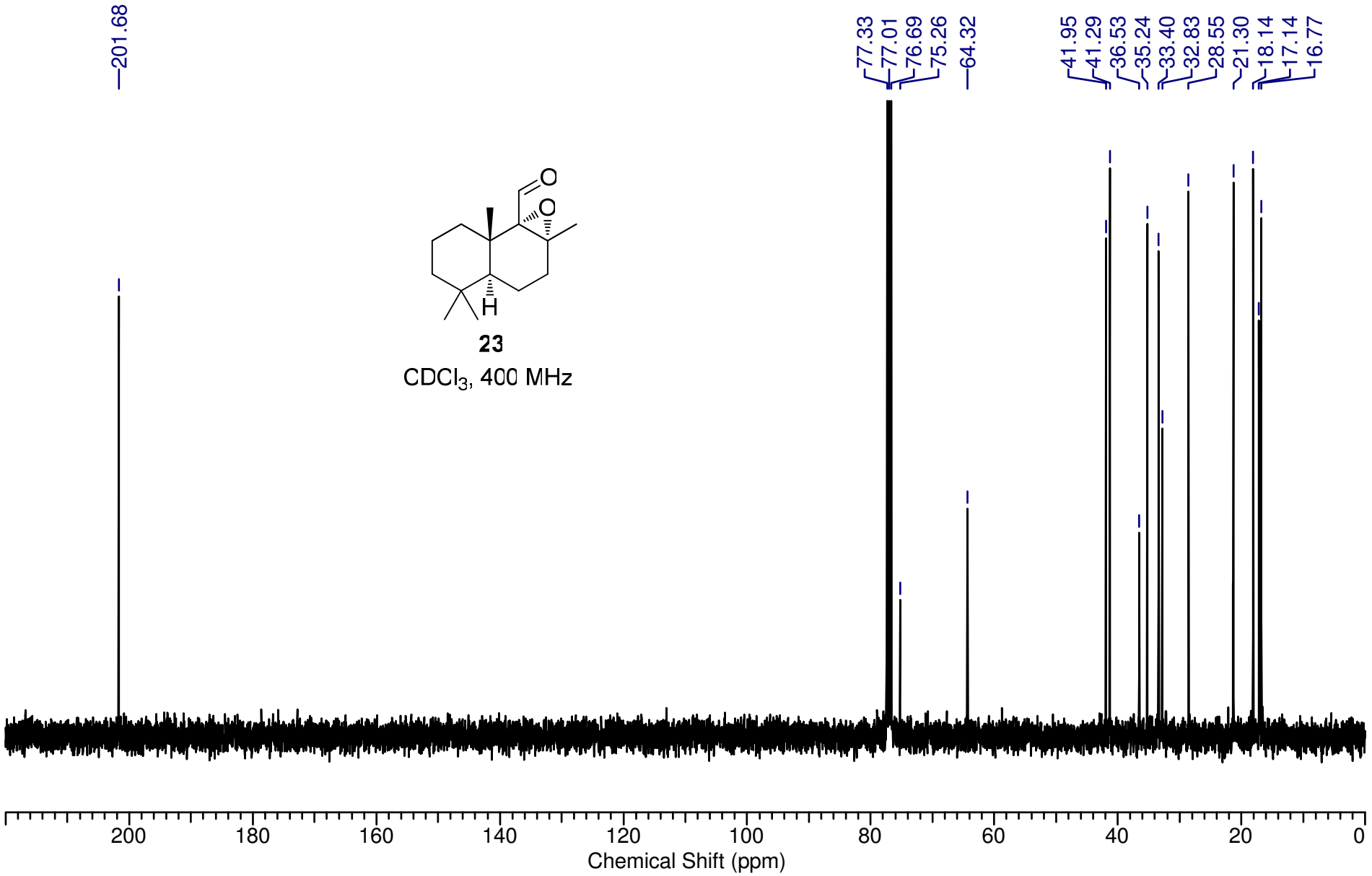

9.73
3.27
2.40
2.38
1.65
1.62
1.61
1.58
1.48
1.42
1.40
1.37
1.27
0.87
0.85
0.64
0.62
O
OH
H
19
CDCl3, 400 MHz
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

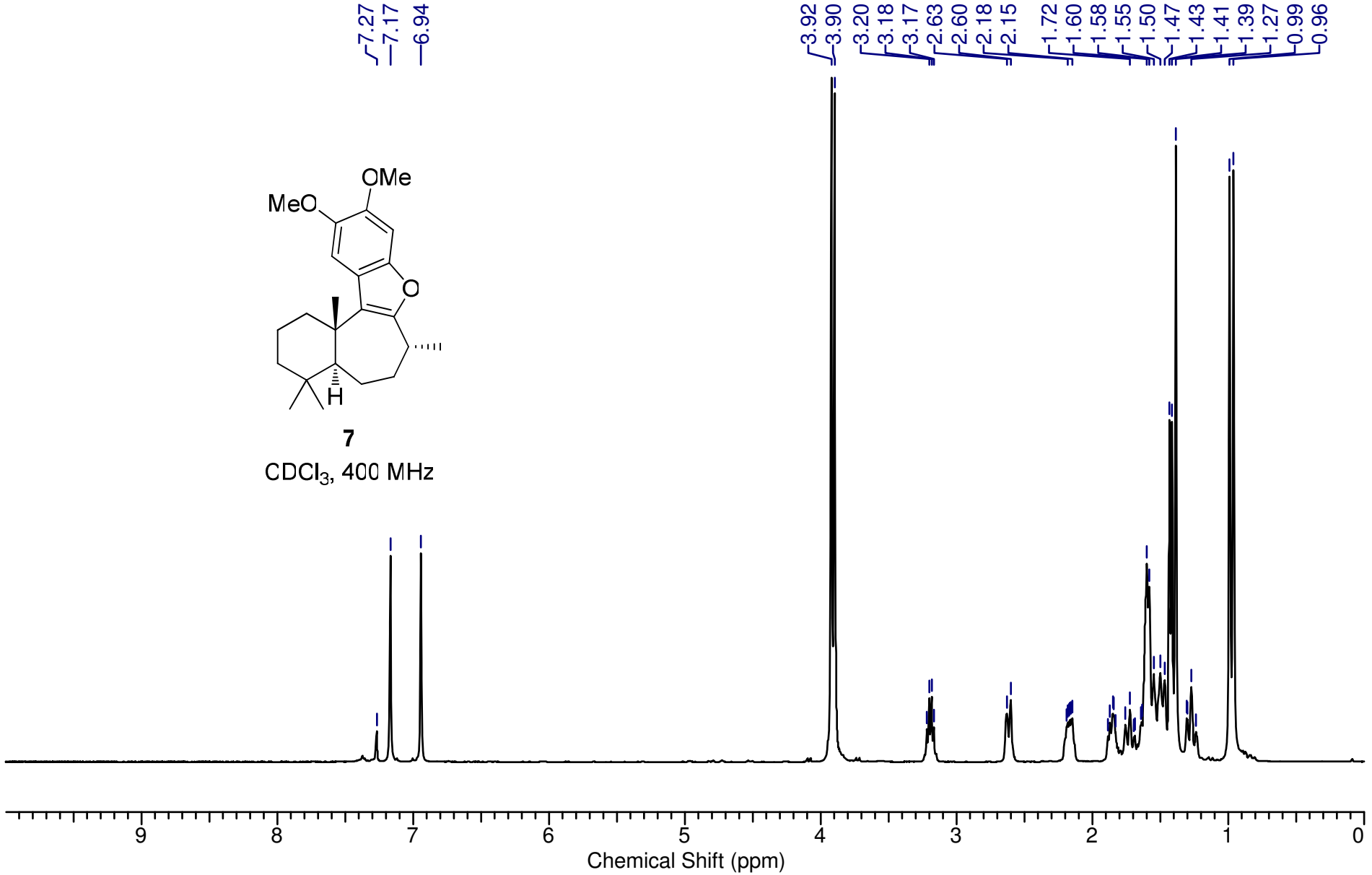

207.70
83.75
77.34
77.03
76.71
44.98
42.42
41.46
33.46
33.27
33.14
30.61
30.36
22.07
21.35
18.18
17.13
15.64
O
OH
H
19
CDCl3, 400 MHz
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

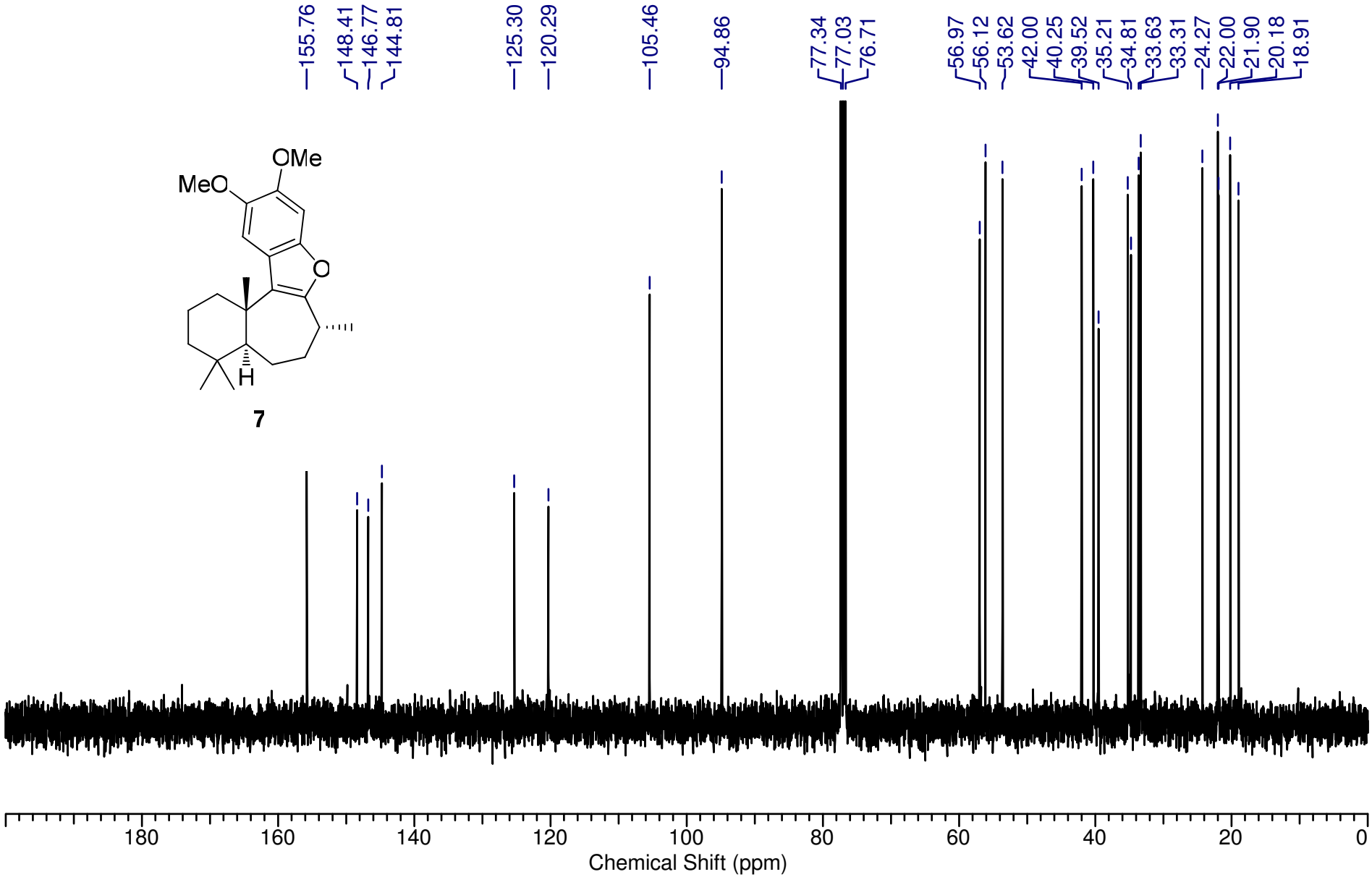

7.27
4.26
4.13
3.82
3.82
2.90
2.75
2.50
2.49
2.13
2.12
1.76
1.56
1.55
1.53
1.14
1.13
1.11
1.10
1.09
1.04
0.86
0.84
0.83
HO
O
H
22
CDCl3, 500 MHz
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

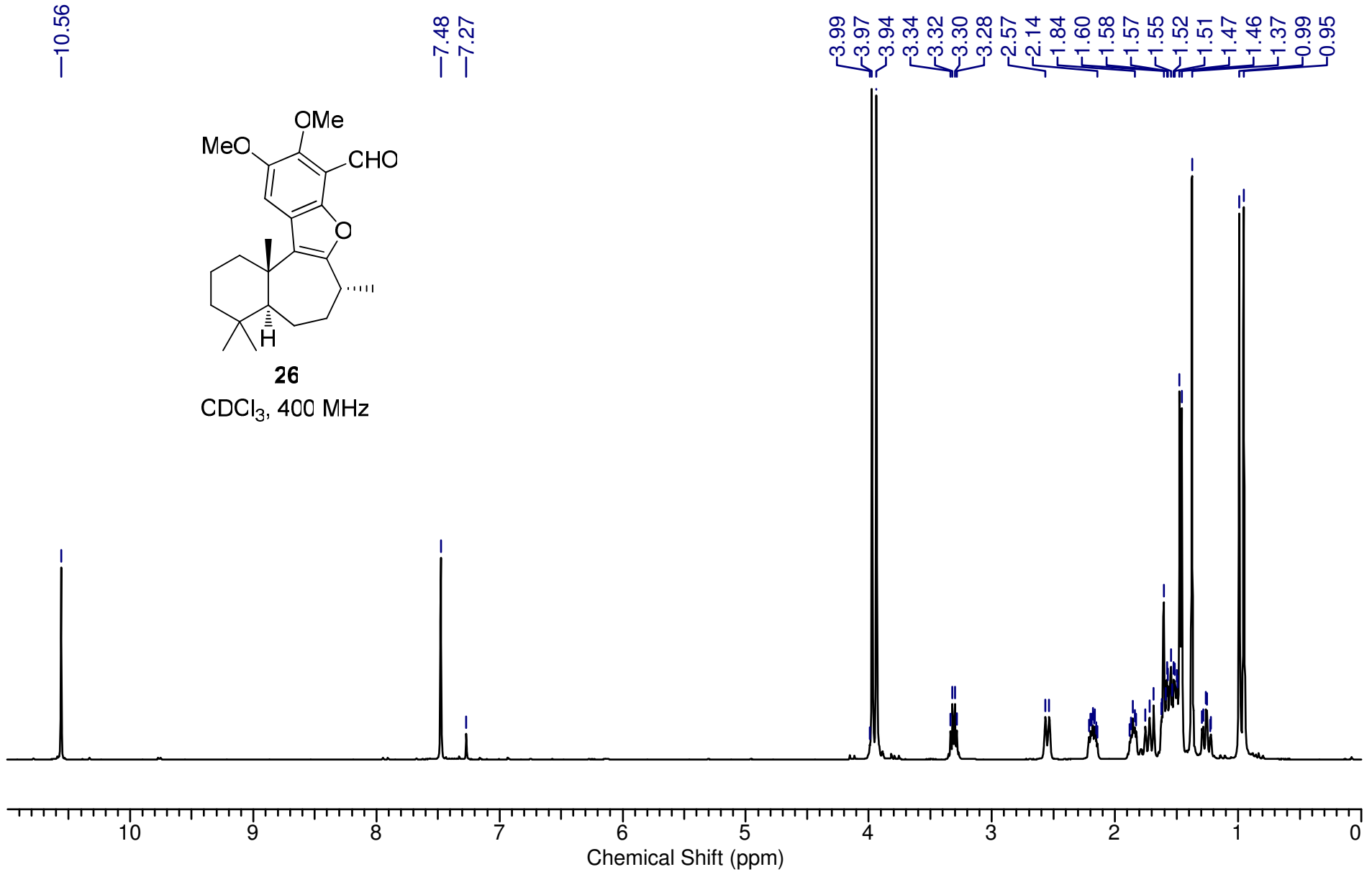

216.95
216.38
84.72
82.35
77.24
76.99
76.74
52.20
49.42
45.59
42.52
42.04
41.62
34.60
33.66
32.78
32.26
21.83
21.74
21.69
21.40
19.90
18.42
16.00
HO
O
H
22
CDCl3, 500 MHz
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

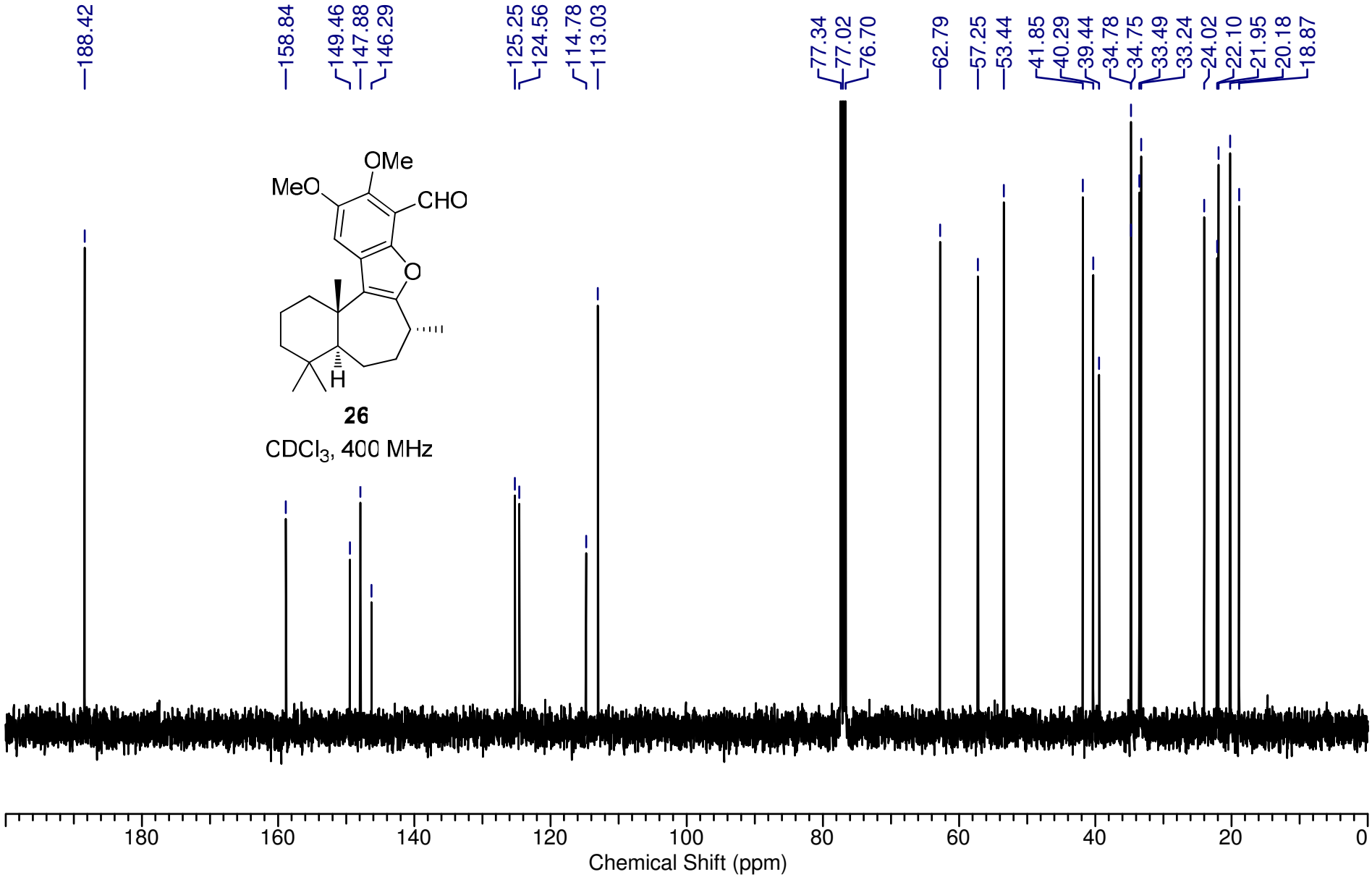

9.70
7.27
2.02
1.98
1.63
1.53
1.49
1.48
1.45
1.44
1.42
1.39
1.37
1.25
0.84
0.80
O
O
H
23
CDCl3, 400 MHz
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


201.68
77.33
77.01
76.69
75.26
64.32
41.95
41.29
36.53
35.24
33.40
32.83
28.55
21.30
18.14
17.14
16.77
O
O
H
23
CDCl3, 400 MHz
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


7.27
7.17
6.94
3.92
3.90
3.20
3.18
3.17
2.63
2.60
2.18
2.15
1.72
1.60
1.58
1.55
1.50
1.47
1.43
1.41
1.39
1.27
0.99
0.96
OMe
MeO
O
H
7
CDCl3, 400 MHz
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


155.76
148.41
146.77
144.81
125.30
120.29
105.46
94.86
77.34
77.03
76.71
56.97
56.12
53.62
42.00
40.25
39.52
35.21
34.81
33.63
33.31
24.27
22.00
21.90
20.18
18.91
OMe
MeO
O
H
7
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


10.56
7.48
7.27
3.99
3.97
3.94
3.34
3.32
3.30
3.28
2.57
2.14
1.84
1.60
1.58
1.57
1.55
1.52
1.51
1.47
1.46
1.37
0.99
0.95
OMe
MeO
CHO
O
H
26
CDCl3, 400 MHz
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


188.42
158.84
149.46
147.88
146.29
125.25
124.56
114.78
113.03
77.34
77.02
76.70
62.79
57.25
53.44
41.85
40.29
39.44
34.78
34.75
33.49
33.24
24.02
22.10
21.95
20.18
18.87
OMe
MeO
CHO
O
H
26
CDCl3, 400 MHz
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)