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Supporting Information

Synthesis of polycyclic frameworks through Iron-catalyzed intramolecular [5+2] cycloaddition

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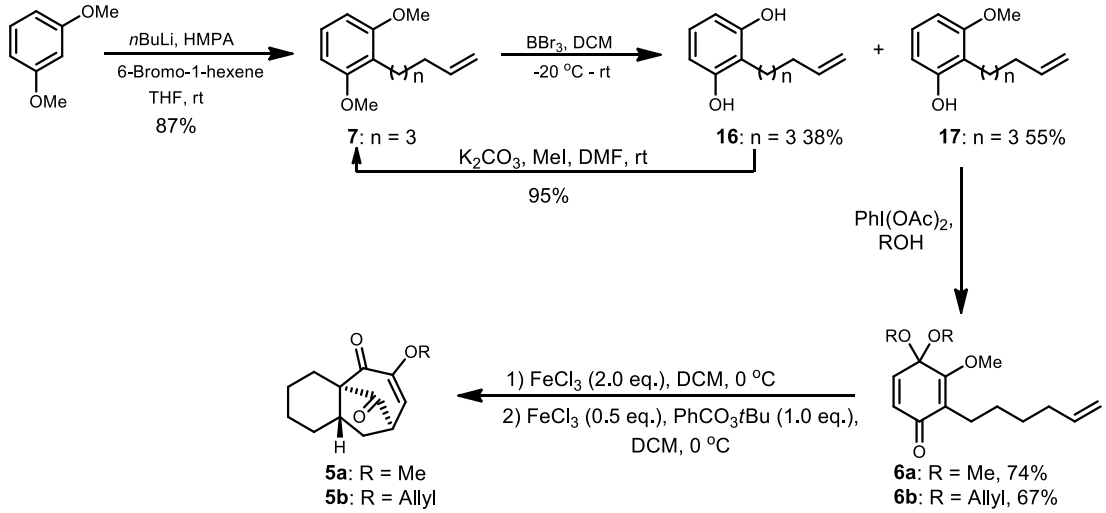
General Information

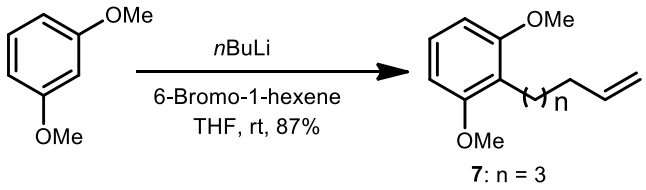
All reactions were carried out under an argon atmosphere with dry solvents under anhydrous conditions, unless otherwise noted. DCM, DMF, MeCN and Toluene were distilled from calcium hydride under argon; THF and Et_2O were distilled from sodium-benzophenone under argon. All the other chemicals were purchased commercially and used without further purification, unless otherwise stated. Flash chromatography was performed using silica gel (200-300 mesh). Reactions were monitored by thin layer chromatography (TLC). Visualization was achieved under a UV lamp (254 nm and 365 nm), I_2 and by developing the plates with *p*-anisaldehyde or phosphomolybdic acid. ^1H and ^{13}C NMR were recorded on Bruker DRX-400 MHz NMR spectrometer or Bruker AV-II 600 MHz NMR spectrometer with TMS as the internal standard and were calibrated using residual undeuterated solvent as an internal reference ($CDCl_3$: ^1H NMR = 7.26, ^{13}C NMR = 77.16; C_6D_6: ^1H NMR = 7.16, ^{13}C NMR = 128.06; DMSO-d6: ^1H NMR = 3.33, ^{13}C NMR = 39.52). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Coupling constants (*J*) are reported in Hertz (Hz). High resolution mass spectra (HRMS) were recorded by using FTMS-7 spectrometers. Infrared (IR) spectra were recorded on a NEXUS 670 FT-IR Fourier transform infrared spectrophotometer and are reported in wavenumbers (cm^{-1}).

Experimental Procedures

Procedure for the Preparation of Compound 5a and 5b



Synthesis of compound 7



To a solution of 1,3-Dimethoxybenzene (10.0 mL, 75.6 mmol) in dry THF (350 ml) at 0℃ was slowly added *n*-BuLi (2.5 M in hexane, 40.0 mL, 98.3 mmol). The solution was allowed to warm to room temperature and stirred for 1 h. The mixture was recooled to 0℃ and was treated with 6-Bromo-1-hexene (13.2 mL, 98.3 mmol). The solution was allowed to room temperature and stirred for 12 h. The reaction was quenched by saturated NH_4Cl solution (100 mL). And then extracted with EtOAc (200 mL x 3), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:100) to afford the corresponding compound **7** (14.55 g) in 87% yield as a colorless oil.

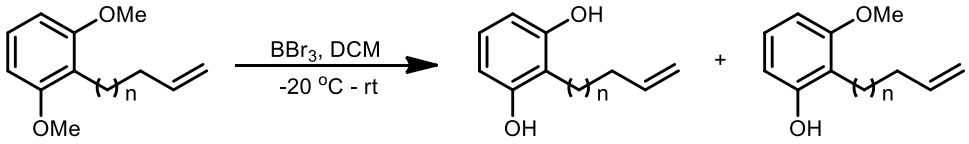
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (t, J = 8.4 Hz, 1H), 6.54 (d, J = 8.4 Hz, 2H), 5.86 (ddt, J = 6.8, 10.4, 17.2 Hz, 1H), 5.02 (ddd, J = 1.6, 3.2, 16.8 Hz, 1H), 4.94 (dt, J = 1.2, 10.4 Hz, 1H), 3.82 (s, 6H), 2.67 (t, J = 7.2 Hz, 2H), 2.11 (dd, J = 6.8, 14.0 Hz, 2H), 1.57 – 1.43 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 139.5, 126.6, 119.4, 114.1, 103.7, 55.7, 33.9, 29.1, 28.9, 22.8.

IR (neat, cm^{-1}): 3073, 2932, 2843, 1639, 1593, 1483, 1252, 910.

HRMS (ESI): *m/z* calcd for $C_{14}H_{20}NaO_2$ $[M+Na]^+$: 243.1361; Found: 243.1363.

Synthesis of compound 16 and 17



To a solution of compound **7** (11.02 g, 50.0 mmol) in dry DCM (150 mL) at -20℃ was slowly added BBr_3 (2.1 mL, 21.6 mmol) in DCM (20 mL). The mixture was stirred at the same temperature for 20 min, and then warm to 0℃ and stirred for 10 h. The reaction was quenched by saturated $NaHCO_3$ solution (50 mL). And then extracted with EtOAc (200 mL x 3), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:10) to afford the corresponding compound **17** (5.67 g) in 55% yield as a light yellow oil and compound **16** (3.67 g) in 38% yield as a yellow oil.

Compound **17:**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (t, *J* = 8.0 Hz, 1H), 6.49 (d, *J* = 8.4 Hz, 1H), 6.44 (dd, *J* = 0.8, 8.4 Hz, 1H), 5.85 (ddt, *J* = 6.8, 10.0, 16.8 Hz, 1H), 5.03 (ddd, *J* = 1.6, 3.6, 17.2 Hz, 1H), 4.96 (ddt, *J* = 1.2, 3.6, 10.4 Hz, 1H), 4.89 (s, 1H), 3.82 (s, 3H), 2.66 (t, *J* = 7.2 Hz, 2H), 2.12 (dd, *J* = 7.2, 14.4 Hz, 2H), 1.60 – 1.44 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 154.4, 139.4, 126.8, 117.2, 114.3, 108.4, 103.3, 55.8, 33.8, 29.0, 28.7, 22.9.

IR (neat, cm^{-1}): 3075, 2857, 1599, 1466, 1277, 1248, 1119, 1084.

HRMS (ESI): *m/z* calcd for $C_{13}H_{18}NaO_2$ $[M+Na]^+$: 229.1204; Found: 229.1203.

Compound **16:**

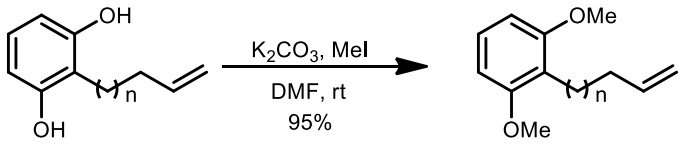
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92 (t, J = 8.4 Hz, 1H), 6.38 (d, J = 8.0 Hz, 2H), 5.82 (ddt, J = 6.4, 10.0, 16.8 Hz, 1H), 5.01 (ddd, J = 1.6, 3.6, 17.2 Hz, 1H), 4.95 (dt, J = 1.2, 10.4 Hz, 1H), 4.78 (s, 2H), 2.63 (t, J = 7.2 Hz, 2H), 2.11 (dd, J = 6.8, 14.0 Hz, 2H), 1.62 – 1.45 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 154.8, 139.2, 126.9, 115.5, 114.5, 108.1, 33.8, 28.9, 28.6, 23.0.

IR (neat, cm^{-1}): 3075, 2930, 2859, 1607, 1356, 1288, 1109, 1068.

HRMS (ESI): *m/z* calcd for $C_{12}H_{16}NaO_2$ $[M+Na]^+$: 215.1048; Found: 215.1042.

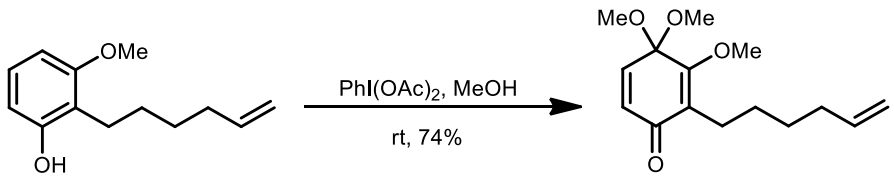
Synthesis of compound 7



16: n = 3 **7**: n = 3

To a solution of compound **16** (3.67 g, 19.0 mmol) in DMF (100 mL) at room temperature was added K_2CO_3 (7.96 g, 57.0 mmol). The mixture was stirred for 10 min at room temperature and then added MeI (6.0 mL, 95.0 mmol). The mixture was stirred at the same temperature for 11 h. The reaction was quenched by saturated NH_4Cl solution (50 mL). And then extracted with EtOAc (50 mL x 3), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:100) to afford the corresponding compound **7** (3.97 g) in 95% yield as a colorless oil.

Synthesis of compound 6a



17 **6a**

To a solution of compound **17** (4.17 g, 20.2 mmol) in MeOH (100 mL) was added $PhI(OAc)_2$ (16.63 g, 50.5 mmol) at room temperature. The mixture was stirred for 4 h at the same temperature. The reaction mixture was quenched by saturated $NaHCO_3$ solution (100 mL)

and Na_2SO_3 solution (100 mL), followed by extraction with EtOAc (200 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:10) to afford the product **6a** (3.94 g) in 74% yield as a light yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.37 (d, J = 10.4 Hz, 1H), 6.31 (d, J = 10.0 Hz, 1H), 5.79 (ddt, J = 6.4, 10.0, 16.8 Hz, 1H), 4.98 (ddd, J = 1.6, 3.6, 17.2 Hz, 1H), 4.91 (ddd, J = 1.2, 2.0, 10.0 Hz, 1H), 4.14 (s, 3H), 3.30 (s, 6H), 2.35 (t, J = 7.2 Hz, 2H), 2.05 (q, J = 6.8 Hz, 2H), 1.38 (t, J = 3.6 Hz, 4H).

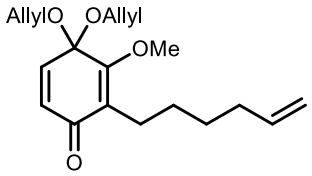
^{13}C NMR (100 MHz, $CDCl_3$) δ 186.4, 161.3, 139.6, 139.2, 132.0, 124.5, 114.3, 96.7, 59.0, 51.5, 33.7, 29.1, 28.3, 22.4.

IR (neat, cm^{-1}): 2936, 1665, 1602, 1456, 1304, 1212, 1066, 914.

HRMS (ESI): *m/z* calcd for $C_{15}H_{22}NaO_4$ $[M+Na]^+$: 289.1416; Found: 289.1413.

Synthesis of compound 6b

6b prepared according to the similar procedure for the preparation of **6a** as a light-yellow oil (561.9 mg, 67% yield).



6b

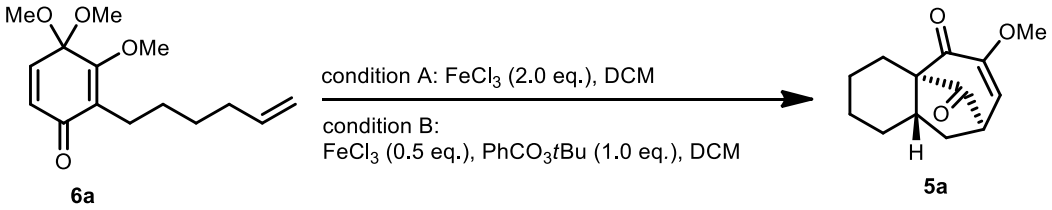
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.45 (dd, J = 0.8, 10.0 Hz, 1H), 6.27 (dd, J = 0.8, 10.0 Hz, 1H), 5.90 (ddd, J = 5.6, 10.4, 21.6 Hz, 2H), 5.79 (ddd, J = 3.6, 6.8, 16.8 Hz, 1H), 5.29 (dd, J = 1.2, 17.2 Hz, 2H), 5.18 (dd, J = 0.8, 10.4 Hz, 2H), 4.98 (d, J = 17.2 Hz, 1H), 4.91 (d, J = 10.4 Hz, 1H), 4.16 (d, J = 1.2 Hz, 3H), 4.06 (dd, J = 1.2, 5.2 Hz, 2H), 3.96 (dd, J = 1.2, 5.6 Hz, 2H), 2.35 (t, J = 6.8 Hz, 2H), 2.05 (d, J = 5.6 Hz, 2H), 1.38 (t, J = 2.8 Hz, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 186.3, 161.8, 139.7, 139.2, 133.6, 131.3, 123.8, 117.5, 114.3, 95.8, 65.0, 59.2, 33.7, 29.0, 28.2, 22.4.

IR (neat, cm^{-1}): 2929, 2854, 1742, 1666, 1603, 1466, 1304, 1050.

HRMS (ESI): *m/z* calcd for $C_{19}H_{26}NaO_4$ $[M+Na]^+$: 341.1729; Found: 341.1728.

Synthesis of compound 5a[1]



Condition A:

To a suspension of $FeCl_3$ (1.15 g, 6.95 mmol, 2.0 equiv.) in dry DCM (350 mL) was added a solution of **6a** (910.0 mg, 3.47 mmol, 1.0 equiv.) in DCM (7.5 mL) dropwise at 0℃. The mixture was stirred for 10 min at the same temperature. The reaction mixture was quenched by saturated $NaHCO_3$ solution (50 mL), the layers were diluted with DCM (100 mL), H_2O (200 mL) and separated. Then the aqueous layer was extracted with DCM (100 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:1.5) to afford the product **5a** (609.9 mg) in 80% yield as a yellow oil**.**

Condition B:

To a suspension of $FeCl_3$ (24.8 mg, 0.15 mmol, 0.5 equiv.) in DCM (30 mL) was added $PhCO_3tBu$ (0.3 M in DCM, 1 mL, 0.3 mmol, 1.0 equiv.) at 0℃. And then added a solution of **6a** (80.0 mg, 0.3 mmol, 1.0 equiv.) in DCM (5 mL) dropwise. The mixture was stirred for 10 min at the same temperature. The reaction mixture was quenched by saturated $NaHCO_3$ solution (10 mL). The layers were diluted with DCM (10 mL), H_2O (10 mL) and separated. Then the aqueous layer was extracted with DCM (15 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:1.5) to afford the product **5a** (53.6 mg) in 81% yield as a yellow oil.

^1H NMR (400 MHz, $CDCl_3$ with a drop of D_2O) δ 6.41 (d, J = 8.8 Hz, 1H), 3.62 (s, 3H), 3.20 (dd, J = 6.8, 8.8 Hz, 1H), 2.22 (ddd, J = 5.2, 12.8, 14.8 Hz, 1H), 2.01 –1.91 (m, 3H), 1.87 – 1.78 (m,

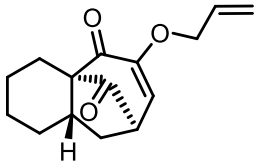
2H), 1.68 (dt, *J* = 4.0, 12.8 Hz, 1H), 1.58 (dt, *J* =4.0, 12.0 Hz, 1H), 1.31 – 1.12 (m, 2H), 1.00 – 0.90 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 205.0, 194.6, 153.3, 120.5, 66.8, 55.6, 44.2, 37.5, 33.5, 33.0, 23.3, 21.2, 20.7.

IR (neat, cm^{-1}): 2933, 1758, 1683, 1612, 1456, 1353, 1234, 1173.

HRMS (ESI): *m/z* calcd for $C_{13}H_{16}NaO_3$ $[M+Na]^+$: 243.0997; Found: 243.0990.

Synthesis of compound 5b



5b prepared according to the similar procedure for the preparation of **5a** as a yellow oil (condition A: 58.5 mg, 79%; condition B: 53.2 mg, 72%).

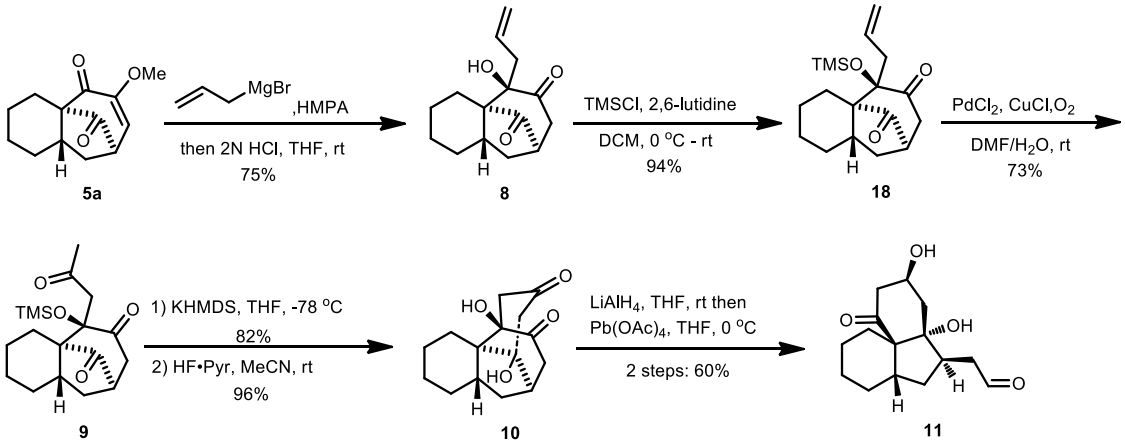
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.45 (d, *J* = 9.2 Hz, 1H), 5.95 (ddt, *J* = 5.2, 10.4, 16.8 Hz, 1H), 5.33 (dd, *J* = 1.2, 17.2 Hz, 1H), 5.27 (dd, *J* = 1.2, 10.4 Hz, 1H), 4.33 (dd, *J* = 1.2, 5.2 Hz, 2H), 3.20 (dd, *J* = 7.2, 8.8 Hz, 1H), 2.24 (ddd, *J* = 5.2, 12.4, 14.4 Hz, 1H), 2.01 – 1.93 (m, 3H), 1.88 – 1.78 (m, 2H), 1.71 – 1.67 (m, 1H), 1.62 – 1.58 (m, 1H), 1.33 – 1.13 (m, 2H), 1.01 – 0.91 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 205.0, 194.6, 152.1, 132.1, 122.2, 118.8, 69.4, 66.9, 44.3, 37.5, 33.5, 33.0, 23.4, 21.3, 20.8.

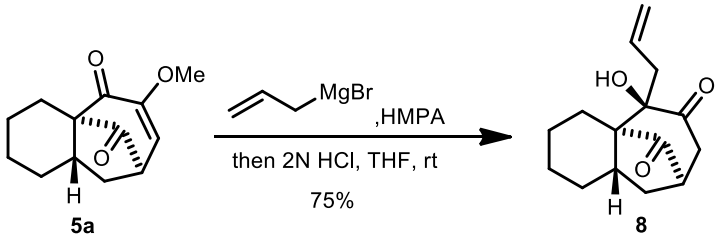
IR (neat, cm^{-1}): 2930, 2358, 2342, 1755, 1680, 1528, 1432, 1070.

HRMS (ESI): *m/z* calcd for $C_{15}H_{18}NaO_3$ $[M+Na]^+$: 269.1154; Found: 269.1153.

Procedure for the Preparation of Compound 11



Synthesis of compound 8



To a solution of **5a** (173.7 mg, 0.79 mmol) in THF (10.0 mL) at -78℃ was added HMPA (2.5 mL), and then added allylmagnesium bromide (1.0 M in Et_2O, 1.2 mL, 1.2 mmol) dropwise. The mixture was stirred at the same temperature for 45 min. The reaction mixture was quenched by saturated NH_4Cl solution (2.5 mL), The mixture was stirred at the room temperature for 5 min, then added 2N HCl (5.0 mL) dropwise, stirred at room temperature for 4 h. The reaction mixture was quenched by saturated $NaHCO_3$ solution (20 mL), followed by extraction with EtOAc (20 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:8) to afford the product **8** (146.9 mg) in 75% yield as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.46 (ddd, J = 5.6, 9.6, 12.8 Hz,1H), 4.99 (d, J = 4.8 Hz, 1H), 4.96 (d, J = 11.6 Hz, 1H), 3.93 (s, 1H), 2.76 (dt, J = 2.4, 14.0 Hz, 1H), 2.65 – 2.60 (m, 1H), 2.58 – 2.51 (m, 2H), 2.24 – 2.15 (m, 2H), 2.10 (dd, J = 8.8, 14.4 Hz, 1H), 1.88 (ddd, J = 2.4, 6.4, 13.2 Hz, 1H), 1.75 (dd, J = 9.6, 14.4 Hz, 1H), 1.64 (d, J = 13.2 Hz, 1H), 1.55 (dd, J = 5.2, 12.8 Hz, 1H),

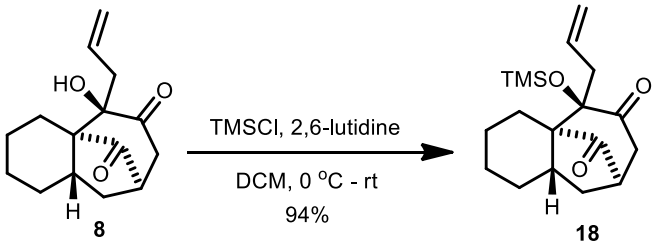
1.52 – 1.45 (m, 2H), 1.12 (ddt, *J* = 4.0, 12.8, 26.4 Hz, 1H), 1.04 (ddt, *J* = 2.4, 12.4, 25.2 Hz, 1H), 0.76 (ddd, *J* = 2.8, 12.8, 25.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 214.7, 209.9, 131.0, 119.0, 84.3, 58.5, 44.9, 42.0, 39.1, 34.9, 32.0, 31.1, 23.8, 23.0, 21.4.

IR (neat, cm^{-1}): 2565, 1746, 1720, 1640, 1441, 1188, 1093, 1002.

HRMS (ESI): *m/z* calcd for $C_{15}H_{20}NaO_3$ $[M+Na]^+$: 271.1310; Found: 271.1308.

Synthesis of compound 18



To a solution of compound **8** (146.9 mg) in DCM (10.0 mL) at 0℃ was added 2,6-lutidine (0.2 mL, 1.8 mmol), TMSOTf (0.3 mL, 2.1 mmol) in sequence. The mixture was stirred at room temperature for 3 h. The reaction mixture was quenched by saturated NH_4Cl solution (15 mL), followed by extraction with DCM (20 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:15) to afford the product **18** (180.8 mg) in 94% yield as a white solid.

mp 67 - 69℃

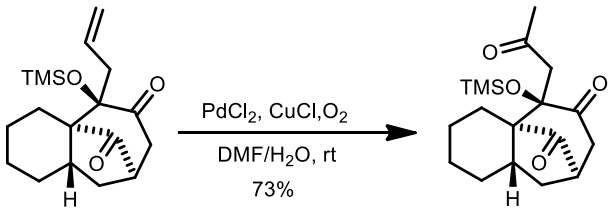
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.56 – 5.43 (m, 1H), 4.96 (d, *J* = 10.4 Hz, 1H), 4.92 (dd, *J* = 0.8, 17.2 Hz, 1H), 2.61 – 2.55 (m, 2H), 2.45 (t, *J* = 4.0 Hz, 1H), 2.41 (dt, *J* = 1.6, 4.8 Hz, 1H), 2.19 (ddd, *J* = 2.8, 5.6, 12.0 Hz, 1H), 2.14 (d, *J* = 8.4 Hz, 1H), 2.10 (d, *J* = 8.0 Hz, 1H), 1.86 (td, *J* = 2.4, 6.4 Hz, 1H), 1.74 (dd, *J* = 9.2, 14.0 Hz, 1H), 1.62 (dt, *J* = 2.8, 10.4 Hz, 1H), 1.57 – 1.46 (m, 3H), 1.12 (ddt, *J* = 4.4, 12.8, 25.6 Hz, 1H), 0.98 (ddt, *J* = 2.8, 12.8, 25.6 Hz, 1H), 0.77 (ddd, *J* = 3.2, 13.2, 25.6 Hz, 1H), 0.19 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 215.2, 207.8, 132.1, 118.5, 89.0, 59.8, 45.0, 41.6, 41.0, 34.8, 33.0, 30.9, 24.1, 23.0, 21.5, 3.2.

IR (neat, cm^{-1}): 1734, 1442, 1252, 1169, 1128, 1034, 997, 845.

HRMS (ESI): *m/z* calcd for $C_{18}H_{28}NaO_3Si$ $[M+Na]^+$: 343.1705; Found: 343.1701.

Synthesis of compound 9



18 **9**

To a solution of compound **18** (180.8 mg) in DMF/H_2O (v/v = 7:1, 8 mL) at room temperature was added CuCl (85.7 mg, 0.84 mmol), $PdCl_2$ (20.2 mg, 0.11 mmol) in sequence. An oxygen-filled balloon was placed over one neck, the mixture was stirred at room temperature for overnight. The reaction mixture was quenched by saturated NH_4Cl solution (10 mL), followed by extraction with EtOAc (20 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:8) to afford the product **9** (180.8 mg) in 73% yield as a white solid.

mp 112 - 114℃

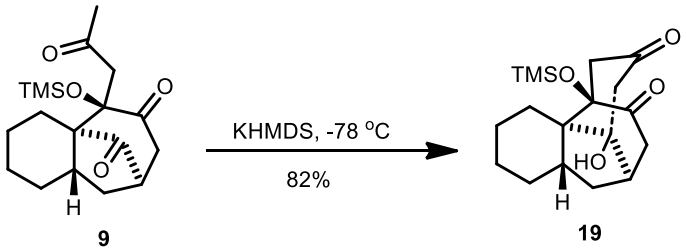
^1H NMR (400 MHz, $CDCl_3$) δ 3.00 (d, *J* = 14.8 Hz, 1H), 2.92 (dd, *J* = 1.6, 14.4 Hz, 1H), 2.57 (d, *J* = 3.2 Hz, 1H), 2.50 (d, *J* = 5.2 Hz, 1H), 2.47 (d, *J* = 6.0 Hz, 1H), 2.16 – 2.10 (m, 1H), 2.10 (s, 3H), 2.05 (dd, *J* = 4.0, 11.6 Hz, 1H), 1.87 (dd, *J* = 2.0, 6.8 Hz, 1H), 1.81 (dd, *J* = 9.6, 14.0 Hz, 1H), 1.69 – 1.41 (m, 4H), 1.15 – 1.03 (m, 1H), 0.95 (ddd, *J* = 1.6, 12.8, 25.2 Hz, 1H), 0.75 (ddd, *J* = 2.0, 12.4, 24.8 Hz, 1H), 0.14 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 215.0, 206.5, 206.0, 87.4, 60.0, 49.5, 43.8, 42.4, 34.8, 32.8, 32.5, 31.0, 23.6, 23.0, 21.6, 3.2.

IR (neat, cm^{-1}): 1732, 1414, 1362, 1250, 1186, 1159, 1030, 843.

HRMS (ESI): *m/z* calcd for $C_{18}H_{28}NaO_4Si$ $[M+Na]^+$: 359.1655; Found: 359.1656.

Synthesis of compound 19



9 19

To a solution of KHMDS (1.0 M in THF, 0.5 mL, 0.5 mmol) in dry THF (5 mL) at -78℃ was slowly added compound **9** (75.7 mg, 0.22 mmol) in THF (1.5 mL). The mixture was stirred at the same temperature for 30 min. The reaction was quenched by saturated NH_4Cl solution (2.5 mL). And then addition H_2O (10 mL), followed by extraction with EtOAc (20 mL x 3), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:3) to afford the corresponding compound **19** (61.7 mg) in 82% yield as a white solid.

mp 149 - 151℃

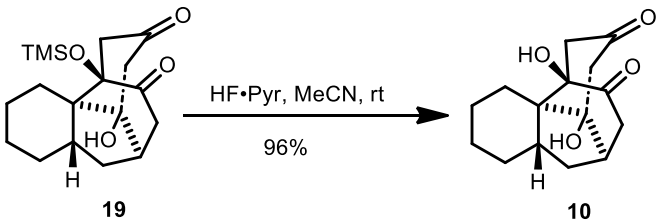
^{1}H NMR (400 MHz, C_6D_6) δ 2.47 (ddd, J = 1.6, 2.8, 17.2 Hz, 1H), 2.37 (d, J = 16.4 Hz, 1H), 2.32 (dd, J = 5.2, 17.2 Hz, 1H), 2.23 (dd, J = 2.0, 16.4 Hz, 1H), 2.06 – 1.80 (m, 5H), 1.68 – 1.66 (m, 1H), 1.59 (dt, J = 3.2, 12.0 Hz, 1H), 1.52 – 1.45 (m, 4H), 1.38 (dd, J = 9.6, 12.4 Hz, 1H), 1.29 – 1.11 (m, 2H), 0.73 (brs, 1H), 0.30 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 208.8, 206.0, 86.2, 84.1, 52.6, 48.6, 48.3, 46.6, 43.1, 37.5, 36.4, 35.4, 24.3, 23.5, 23.2, 2.8.

IR (neat, cm^{-1}): 1724, 1254, 1171, 1038, 882, 845, 756, 687.

HRMS (ESI): *m/z* calcd for $C_{18}H_{28}NaO_4Si$ $[M+Na]^+$: 359.1655; Found: 359.1655.

Synthesis of compound 10



19 10

To a solution of compound **19** (60.0 mg, 0.18 mmol) in MeCN (3.0 mL) at 0℃ was slowly added HF·Pyr (0.5 mL, 30% Pyridine/70%HF). The mixture was stirred at room temperature for overnight. The reaction was quenched by saturated $NaHCO_3$ solution (10 mL), followed by extraction with EtOAc (20 mL x 3), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:3) to afford the corresponding compound **10** (45.2 mg) in 96% yield as a colorless oil.

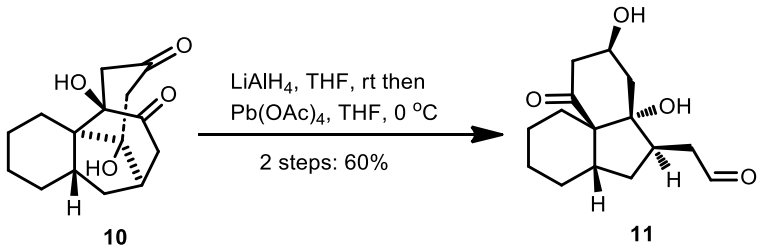
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.05 (s, 1H), 2.97 (dd, *J* = 1.6, 17.6 Hz, 1H), 2.88 (dd, *J* = 15.2, 16.8 Hz, 2H), 2.38 (d, *J* = 2.8 Hz, 2H), 2.29 (dt, *J* = 3.6, 6.8 Hz, 1H), 2.09 (dd, *J* = 1.6, 16.8 Hz, 1H), 2.05 – 1.89 (m, 4H), 1.82 – 1.77 (m, 3H), 1.67 (dd, *J* = 8.8, 13.6 Hz, 1H), 1.62 (dt, *J* = 3.6, 12.4 Hz, 1H), 1.56 – 1.42 (m, 1H), 1.28 (ddd, *J* = 3.2, 13.6, 28.0 Hz, 1H), 1.14 (ddd, *J* = 3.2, 12.8, 25.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 211.0, 206.1, 84.2, 81.8, 51.9, 48.4, 48.1, 47.1, 42.5, 37.3, 35.8, 35.2, 24.1, 23.5, 23.3.

IR (neat, cm^{-1}): 2926, 2862, 1719, 1657, 1373, 1250, 1109, 1044.

HRMS (ESI): *m/z* calcd for $C_{15}H_{20}NaO_4$ $[M+Na]^+$: 287.1254; Found: 287.1261.

Synthesis of compound 11



10 **11**

To a solution of $LiAlH_4$ (56.1 mg, 1.4 mmol) in THF (2.0 mL) at 0℃ was added compound **10** (38.3 mg, 0.14 mmol) in THF (2.0 mL). The mixture was stirred at room temperature for 2.5 h. The reaction mixture was quenched by saturated NH_4Cl solution (0.5 mL). The reaction was filtered through a pad of Celite and washed with EtOAc, The solvent was removed in vacuo to afford crude product, which was used in the next step without further purification.

To a solution of $Pb(OAc)_4$ (155.2 mg, 0.28 mmol) in THF (3.0 mL) at 0℃ was added the above product in THF (3.0 mL) and stirred for 3.5 h. The solvent was removed in vacuo. The crude product was purified by column chromatography (EtOAc) to afford the product **11** (22.9 mg) in 60% yield for 2 steps as a colorless oil.

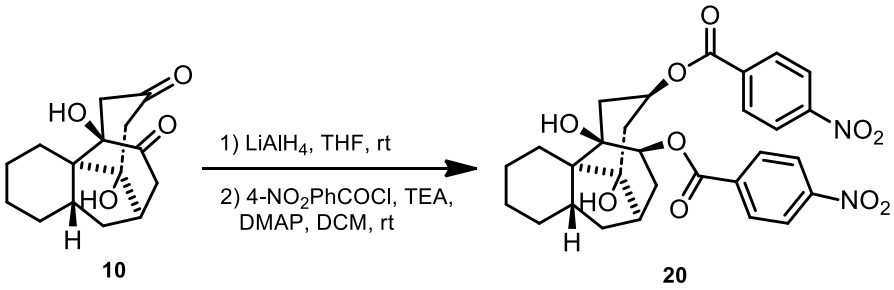
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 4.17 (tt, J = 5.2, 10.8 Hz, 1H), 3.17 (brs, 1H), 2.77 – 2.68 (m, 4H), 2.65 – 2.56 (m, 2H), 2.43 (brs, 1H), 2.12 – 2.02 (m, 1H), 1.99 (d, J = 13.6 Hz, 1H), 1.86 (ddd, J = 1.6, 4.8, 13.2 Hz, 1H), 1.82 (d, J = 10.4 Hz, 1H), 1.65 (s, 2H), 1.52 – 1.42 (m, 3H), 1.35 – 1.21 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.2, 203.2, 85.6, 67.1, 59.6, 47.3, 45.5, 41.9, 39.5, 38.7, 31.6, 26.2, 24.4, 22.7, 20.0.

IR (neat, cm^{-1}): 2934, 2862, 2737, 2251, 1699, 1447, 1238, 1059.

HRMS (ESI): *m/z* calcd for $C_{15}H_{22}NaO_4$ $[M+Na]^+$: 289.1410; Found: 289.1415.

Synthesis of compound 20



10 20

To a solution of $LiAlH_4$ (195.4 mg, 5.0 mmol) in THF (10.0 mL) at 0℃ was added compound **10** (133.4 mg, 0.5 mmol) in THF (5.0 mL). The mixture was stirred at room temperature for 5 h. The reaction mixture was quenched by saturated NH_4Cl solution (0.5 mL). The reaction was filtered through a pad of Celite and washed with EtOAc, The solvent was removed in vacuo to afford crude product, which was used in the next step without further purification.

To a solution of the above product in DCM (10.0 mL) at 0℃ was added DMAP (118.8 mg, 1.0 mmol), TEA (0.5 mL, 3.6 mmol), 4-Nitrobenzoyl chloride (235.3 mg, 1.2 mmol) in sequence. The mixture was stirred at room temperature for 30 min. The reaction mixture was quenched by saturated NH_4Cl solution (10 mL), followed by extraction with DCM (10 mL x 3).

The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:2) to afford the product **20** (109.2 mg) in 52% yield for 2 steps as a white solid.

mp 200 - 202℃

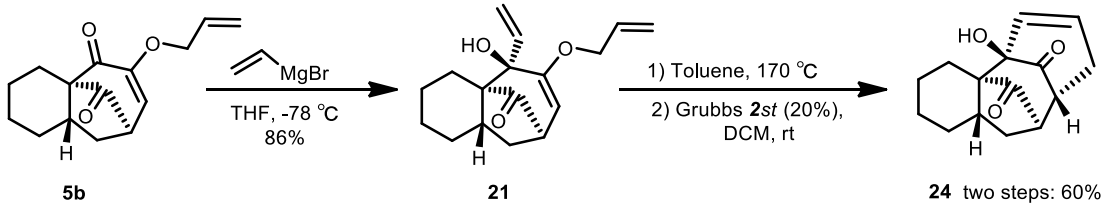
^{1}H NMR (400 MHz, DMSO-d6) δ 8.41 (d, *J* = 3.6 Hz, 2H), 8.39 (d, *J* = 3.6 Hz, 2H), 8.19 (d, *J* = 1.2 Hz, 2H), 8.16 (d, *J* = 1.2 Hz, 2H), 5.44 (t, *J* = 8.0 Hz, 1H), 5.08 (d, *J* = 5.6 Hz, 1H), 4.67 (s, 1H), 4.33 (s, 1H), 2.87 (d, *J* = 15.6 Hz, 1H), 2.68 (d, *J* = 3.6 Hz, 1H), 2.38 (dd, *J* = 8.8, 17.2 Hz, 1H), 2.34 (dd, *J* = 7.2, 16.0 Hz, 1H), 2.10 (d, *J* = 16.8 Hz, 1H), 2.02 – 1.90 (m, 3H), 1.80 – 1.37 (m, 9H), 1.08 – 1.06 (m, 1H).

^{13}C NMR (100 MHz, DMSO-d6) δ 163.8, 163.6, 150.3, 150.1, 136.1, 135.5, 130.6, 130.4, 124.1, 123.9, 83.2, 76.6, 75.7, 69.5, 49.1, 43.8, 38.8, 38.0, 36.3, 35.5, 34.0, 31.9, 24.4, 23.8, 23.4.

IR (neat, cm^{-1}): 2936, 1864, 1719, 1607, 1530, 1350, 1281, 1109.

HRMS (ESI): *m/z* calcd for $C_{29}H_{30}N_2NaO_{10}$ [M+Na]$^+$: 589.1793; Found: 589.1788.

Procedure for the Preparation of Compound 24



To a solution of **5b** (33.5 mg, 0.14 mmol) in THF (5.0 mL) at -78℃ was added Vinylmagnesium bromide (1.0 M in THF, 0.5 mL, 0.5 mmol) dropwise. The mixture was stirred at the same temperature for 3.5 h. The reaction mixture was quenched by saturated NH_4Cl solution (10 mL), followed by extraction with EtOAc (15 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:10) to afford the product **21** (33.0 mg) in 86% yield as a light yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$ with a drop of D_2O) δ 5.92 – 5.83 (m, 1H), 5.68 (dd, J = 10.8, 17.2 Hz, 1H), 5.29 – 5.16 (m, 4H), 4.97 (d, J = 7.6 Hz, 1H), 4.27 – 4.14 (m, 2H), 2.85 – 2.81 (m, 1H), 2.66 – 2.63 (m, 1H), 2.09 (dd, J = 10.0, 12.4 Hz, 1H), 1.86 – 1.80 (m, 2H), 1.62 – 1.07 (m, 7H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.0, 153.7, 138.6, 132.6, 117.8, 114.8, 100.5, 84.5, 68.6, 55.1, 43.5, 36.7, 31.7, 30.9, 20.5, 20.4, 20.0.

IR (neat, cm^{-1}): 2926, 2855, 1741, 1455, 1373, 1242, 1046, 998.

HRMS (ESI): *m/z* calcd for $C_{17}H_{22}NaO_3$ $[M+Na]^+$: 297.1467; Found: 297.1461.

To a solution of **21** (22.3 mg, 0.08 mmol) in toluene (0.5 mL) at 170℃ and stirred for overnight. The solvent was removed in vacuo to afford crude product, which was used in the next step without further purification.

To a solution of Grubbs *2st* (17.5 mg, 0.02 mmol) in DCM (8.0 mL) at room temperature was added the above product in DCM (2.5 mL) and stirred for 10 h. The solvent was removed in vacuo. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:5) to afford the product **24** (12.1 mg) in 60% yield for 2 steps as a white solid.

mp 119 - 121℃

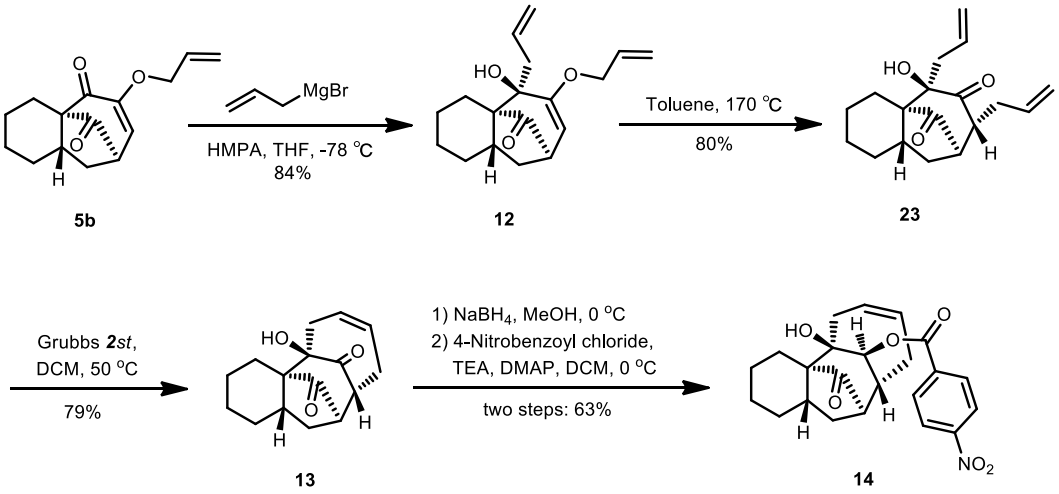
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.65 – 5.59 (m, 2H), 3.87 (s, 1H), 2.88 (t, J = 5.2 Hz, 1H), 2.73 – 2.60 (m, 2H), 2.46 (dd, J = 5.2, 7.2 Hz, 1H), 2.04 – 1.98 (m, 2H), 1.90 – 1.81 (m, 2H), 1.70 (dd, J = 5.2, 13.2 Hz, 1H), 1.66 – 1.59 (m, 1H), 1.52 – 1.47 (m, 1H), 1.42 (ddd, J = 2.8, 7.6, 14.0 Hz, 1H), 1.15 (dddd, J = 2.8, 4.0, 12.4, 25.2 Hz, 1H), 1.03 (ddt, J = 2.8, 12.8, 25.6 Hz, 1H), 0.72 (ddd, J = 3.2, 13.2, 25.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 211.8, 211.0, 132.7, 127.9, 78.9, 60.5, 50.6, 48.5, 35.2, 34.8, 31.6, 30.6, 24.0, 22.7, 20.6.

IR (neat, cm^{-1}): 2925, 2850, 1735, 1448, 1369, 1239, 1141, 1030.

HRMS (ESI): *m/z* calcd for $C_{15}H_{18}NaO_3$ $[M+Na]^+$: 269.1154; Found: 269.1151.

Procedure for the Preparation of Compound 14



To a solution of **5b** (47.7 mg, 0.19 mmol) in THF (4.0 mL) at -78℃ was added HMPA (1.0 mL), and then added allylmagnesium bromide (1.0 M in Et_2O, 0.5 mL, 0.5 mmol) dropwise. The mixture was stirred at the same temperature for 1 h. The reaction mixture was quenched by saturated NH_4Cl solution (1 mL), followed by extraction with EtOAc (20 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:15) to afford the product **12** (45.9 mg) in 84% yield as a light yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.92 (ddd, J = 5.2, 10.4, 16.0 Hz, 1H), 5.76 – 5.66 (m, 1H), 5.30 (dd, J = 1.2, 17.6 Hz, 1H), 5.22 (dd, J = 1.2, 10.4 Hz, 1H), 4.99 (d, J = 7.2 Hz, 1H), 4.96 (s, 1H), 4.92 (d, J = 7.2 Hz, 1H), 4.23 (AB, J = 5.2, 12.8 Hz, AB of A, 1H), 4.13 (AB, J = 5.2, 12.8 Hz, AB of B, 1H), 2.83 – 2.75 (m, 1H), 2.61 – 2.57 (m, 2H), 2.48 (A'B', J = 8.0, 14.0 Hz, A'B' of A', 1H), 2.40 (A'B', J = 6.8, 14.0 Hz, A'B' of B', 1H), 2.08 – 2.02 (m, 1H), 1.98 (dd, J = 9.6, 12.4 Hz, 1H), 1.84 – 1.76 (m, 1H), 1.65 – 1.56 (m, 2H), 1.51 – 1.45 (m, 1H), 1.40 – 1.23 (m, 3H), 1.14 – 1.06 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 211.9, 154.6, 133.7, 132.8, 117.8, 117.7, 99.6, 82.9, 68.4, 54.7, 43.5, 41.9, 36.1, 32.1, 30.1, 20.1, 19.9, 19.6.

IR (neat, cm^{-1}): 2936, 2860, 1743, 1642, 1456, 1210, 1043, 919.

HRMS (ESI): m/z calcd for $C_{18}H_{24}NaO_3$ $[M+Na]^+$: 311.1623; Found: 311.1618.

To a solution of **12** (33.5 mg, 0.12 mmol) in toluene (0.5 mL) at 170℃ and stirred for overnight. The solvent was removed in vacuo. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:20) to afford the product **23** (27.0 mg) in 80% yield as a colorless oil.

^{1}H NMR (400 MHz, C_6D_6) δ 5.52 – 5.38 (m, 2H), 5.07 (ddd, *J* = 1.6, 3.2, 17.2 Hz, 1H), 4.97 (dt, *J* = 0.8, 10.0 Hz, 1H), 4.89 (d, *J* = 10.0 Hz, 1H), 4.80 (ddd, *J* = 1.6, 2.8, 17.2 Hz, 1H), 3.94 (s, 1H), 2.51 – 2.44 (m, 2H), 2.27 – 2.12 (m, 6H), 1.59 – 1.50 (m, 3H), 1.35 – 1.14 (m, 3H), 1.10 (ddd, *J* = 2.8, 8.4, 14.4 Hz, 1H), 0.92 (ddt, *J* = 2.8, 12.8, 25.6 Hz, 1H), 0.52 (ddd, *J* = 3.2, 13.2, 25.6 Hz, 1H).

^{13}C NMR (100 MHz, C_6D_6) δ 213.0, 211.9, 134.5, 132.2, 119.1, 118.3, 84.5, 59.0, 58.5, 44.9, 39.8, 34.8, 33.6, 31.8, 31.7, 24.0, 23.3, 22.2.

IR (neat, cm^{-1}): 2931, 2858, 1743, 1710, 1641, 1445, 1364, 1036.

HRMS (ESI): *m/z* calcd for $C_{18}H_{24}NaO_3$ $[M+Na]^+$: 311.1623; Found: 311.1618.

To a solution of Grubbs *2st* (24.2 mg, 0.03 mmol) in DCM (14.0 mL) was added **23** (40.5 mg, 0.14 mmol) in DCM (2.0 mL) at 50℃ and stirred for 90 min. The solvent was removed in vacuo. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:7) to afford the product **13** (29.0 mg) in 79% yield as a white solid.

mp 72 - 74℃

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.82 – 5.64 (m, 2H), 4.06 (s, 1H), 2.96 (q, *J* = 4.0 Hz, 1H), 2.65 (dd, *J* = 8.4, 16.0 Hz, 1H), 2.49 (ddd, *J* = 4.0, 7.6, 16.4 Hz 1H), 2.39 (dd, *J* = 3.6, 7.6 Hz, 1H), 2.31 – 2.23 (m, 1H), 2.22 – 2.14 (m, 1H), 2.07 – 2.03 (m, 1H), 1.80 – 1.58 (m, 5H), 1.50 – 1.39 (m, 2H), 1.32 – 1.21 (m, 1H), 1.01 (ddt *J* = 2.8, 13.2, 26.0 Hz, 1H), 0.73 (ddd, *J* = 2.8, 12.4, 25.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 214.0, 211.4, 131.3, 126.7, 82.9, 55.7, 55.1, 46.4, 35.9, 33.7, 30.5, 30.3, 28.5, 23.7, 22.7, 21.5.

IR (neat, cm^{-1}): 2931, 1742, 1709, 1630, 1440, 1057, 923, 797.

HRMS (ESI): *m/z* calcd for $C_{16}H_{20}NaO_3$ $[M+Na]^+$: 283.1310; Found: 283.1306.

To a solution of **13** (10.1 mg, 0.04 mmol) in MeOH (1.0 mL) at 0℃ was added $NaBH_4$ (47.9 mg, 1.24 mmol). The mixture was stirred at the same temperature for 20 min. The reaction mixture was quenched by water (2 mL), followed by extraction with EtOAc (10 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:5 to 1:3) to afford the product as a colorless oil.

To a solution of the above product in DCM (2.0 mL) at 0℃ was added DMAP (23.7 mg, 0.2 mmol), TEA (0.3 mL, 2.1 mmol), 4-Nitrobenzoyl chloride (72.8 mg, 0.38 mmol) in sequence. The mixture was stirred at room temperature for 90 min. The reaction mixture was quenched by saturated NH_4Cl solution (2 mL), followed by extraction with DCM (10 mL x 3). The combined organic layers dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc/petroleum ether = 1:5) to afford the product **14** (10.3 mg) in 63% yield for 2 steps as a white solid.

mp 185 - 187℃

^1H NMR (400 MHz, $CDCl_3$ with a drop of D_2O) δ 8.35 (dd, J = 2.4, 8.8 Hz, 2H), 8.21 (dd, J = 2.0, 8.4 Hz, 2H), 5.63 – 5.59 (m, 1H), 5.49 – 5.43 (m, 1H), 5.34 (s, 1H), 2.75 (ddd, J = 2.4, 8.4, 16.8 Hz, 1H), 2.54 – 2.37 (m, 5H), 2.28 – 2.27 (m, 1H), 2.12 (d, J = 16.4 Hz, 1H), 2.01 (brs, 2H), 1.73 – 1.62 (m, 2H), 1.50 (d, J = 9.6 Hz, 1H), 1.40 – 1.35 (m, 1H), 1.26 – 1.17 (m, 1H), 0.99 (ddd J = 2.0, 12.8, 25.6 Hz, 1H), 0.86 – 0.77 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.8, 164.8, 150.9, 135.4, 130.8, 129.5, 124.2, 124.1, 81.0, 79.0, 54.2, 47.1, 43.4, 38.8, 35.2, 32.5, 30.8, 30.4, 23.8, 23.3, 22.1.

IR (neat, cm^{-1}): 2929, 2857, 1728, 1529, 1277, 1102, 1043, 719.

HRMS (ESI): *m/z* calcd for $C_{23}H_{25}NNaO_6$ $[M+Na]^+$: 434.1580; Found: 434.1586.

References:

(1) Liu, Y.; Wang, X.; Chen, S.; Fu, S.; Liu, B. *Org. Lett.* **2018**, *20*, 2934.

X-ray crystallographic analysis of compound 19 (CCDC 1842252)

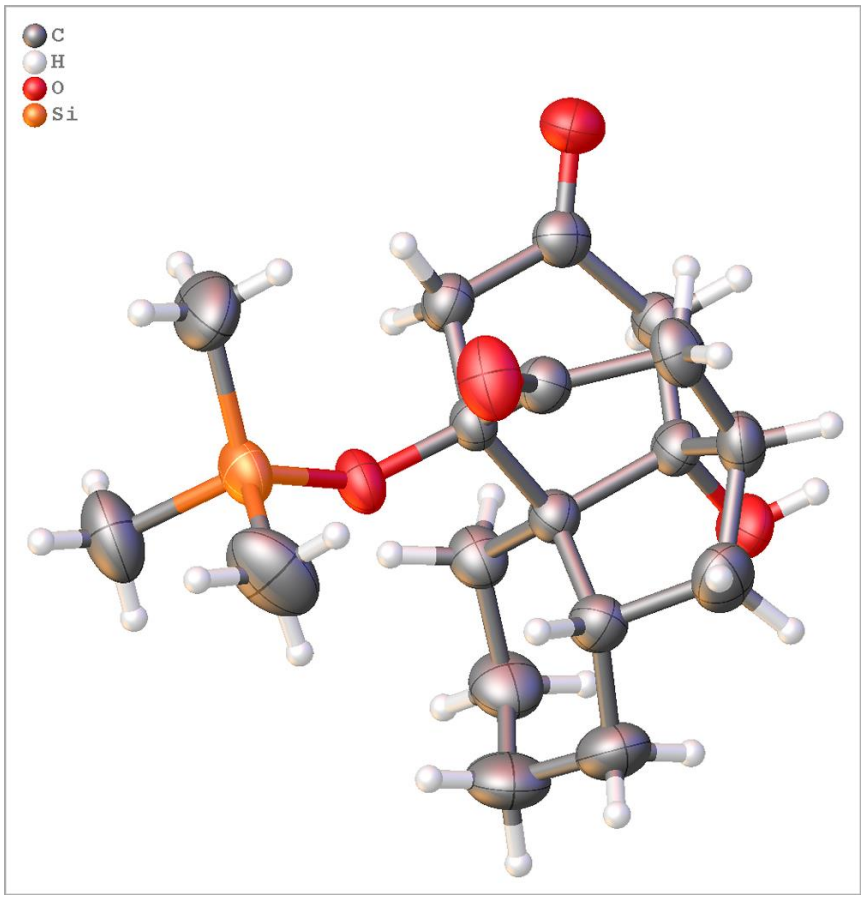


Table Crystal data and structure refinement for **19**

Identification code	19
Empirical formula	$C_{18}H_{28}O_4Si$
Formula weight	336.49
Temperature/K	293.15
Crystal system	orthorhombic
Space group	Pbca
a/Å	9.2091(5)
b/Å	12.7627(7)
c/Å	32.2949(14)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	3795.7(4)
Z	8
ρ_{calc}g/cm^3	1.178
μ/mm^{-1}	0.140
F(000)	1456.0

Crystal size/mm^3	0.35 × 0.3 × 0.25
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.01 to 52.742
Index ranges	-11 ≤ h ≤ 8, -12 ≤ k ≤ 15, -26 ≤ l ≤ 40
Reflections collected	10295
Independent reflections	3864 [R_{int} = 0.0413, R_{sigma} = 0.0603]
Data/restraints/parameters	3864/0/215
Goodness-of-fit on F^2	0.957
Final R indexes [I>=2σ (I)]	R_1 = 0.0508, wR_2 = 0.1026
Final R indexes [all data]	R_1 = 0.1073, wR_2 = 0.1205
Largest diff. peak/hole / e Å^{-3}	0.23/-0.22

X-ray crystallographic analysis of compound 20 (CCDC1856089)

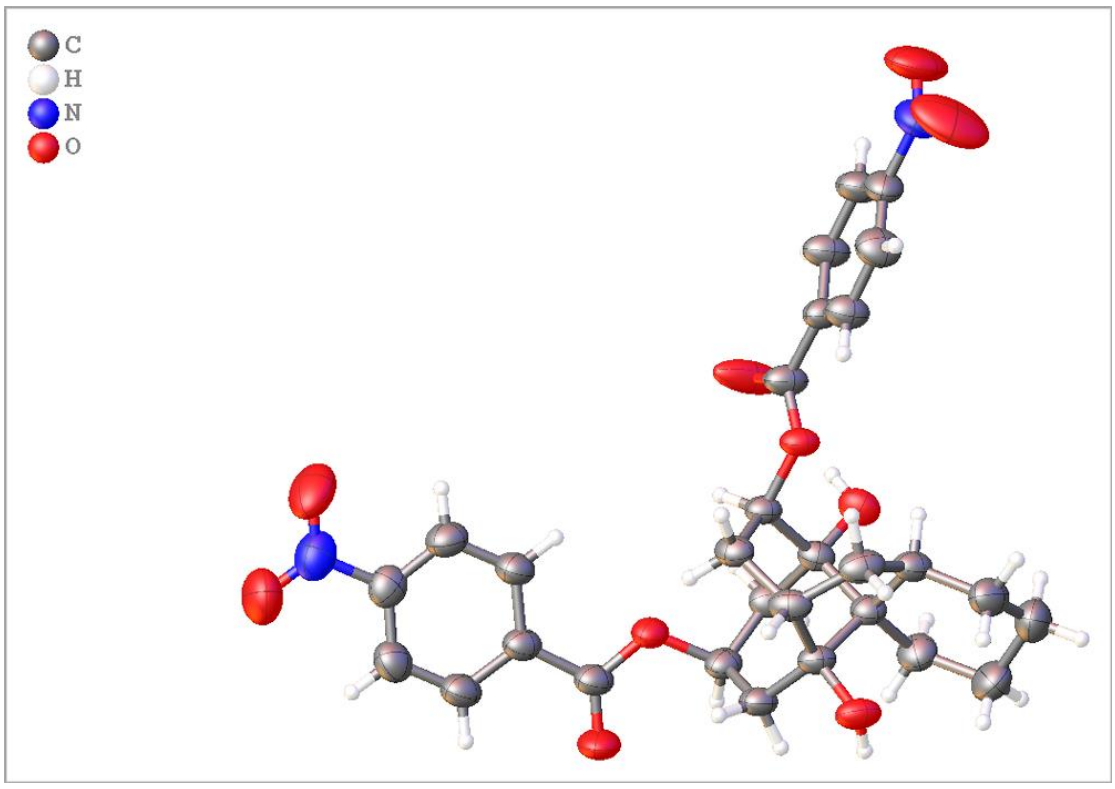


Table Crystal data and structure refinement for **20**

Identification code	20
Empirical formula	$C_{29}H_{30}N_2O_{10}$
Formula weight	566.55
Temperature/K	296.0(8)
Crystal system	triclinic
Space group	P-1
a/Å	11.4254(5)

b/Å	12.6087(6)
c/Å	12.6497(7)
α/°	65.320(5)
β/°	68.680(5)
γ/°	64.069(4)
Volume/Å^3	1452.16(15)
Z	2
ρ_{calc}g/cm^3	1.385
μ/mm^{-1}	0.877
F(000)	636.0
Crystal size/mm^3	0.5 × 0.4 × 0.3
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.888 to 145.974
Index ranges	-14 ≤ h ≤ 12, -15 ≤ k ≤ 15, -15 ≤ l ≤ 15
Reflections collected	16365
Independent reflections	5703 [R_{int} = 0.0316, R_{sigma} = 0.0266]
Data/restraints/parameters	5703/0/372
Goodness-of-fit on F^2	1.050
Final R indexes [I>=2σ (I)]	R_1 = 0.0583, wR_2 = 0.1669
Final R indexes [all data]	R_1 = 0.0665, wR_2 = 0.1786
Largest diff. peak/hole / e Å^{-3}	0.36/-0.31

X-ray crystallographic analysis of compound 24 (CCDC 1524268)

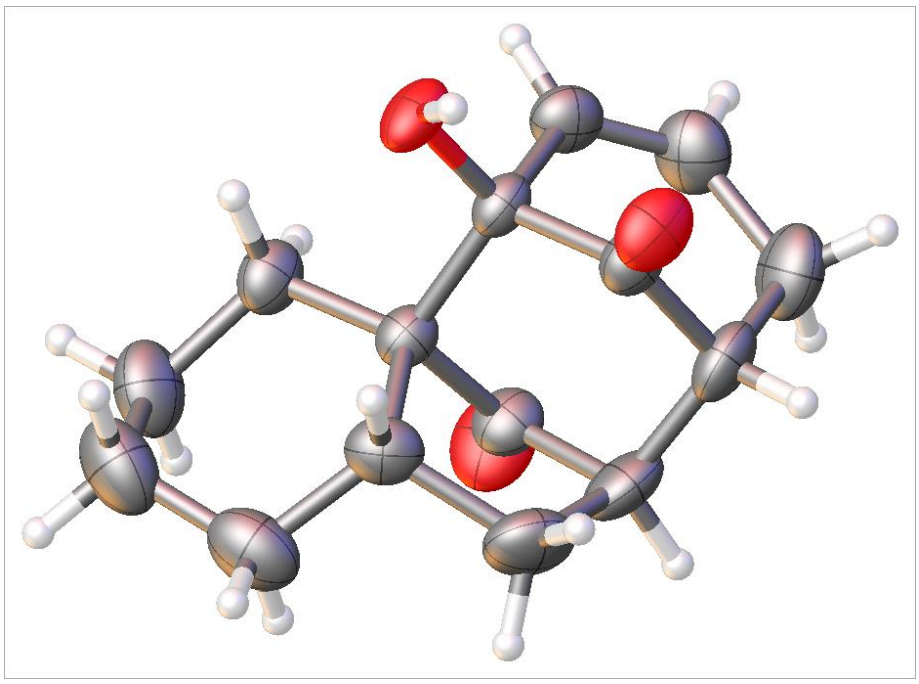


Table Crystal data and structure refinement for **24**

Identification code	24
Empirical formula	$C_{15}H_{18}O_3$
Formula weight	246.29
Temperature/K	293.15
Crystal system	triclinic
Space group	P-1
a/Å	7.1658(6)
b/Å	8.0997(5)
c/Å	11.1352(7)
α/°	83.180(5)
β/°	88.881(6)
γ/°	80.694(6)
Volume/Å^3	633.28(8)
Z	2
ρ_{calc}g/cm^3	1.292
μ/mm^{-1}	0.089
F(000)	264.0
Crystal size/mm^3	0.4 × 0.3 × 0.3
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.958 to 52.74
Index ranges	-8 ≤ h ≤ 8, -10 ≤ k ≤ 9, -13 ≤ l ≤ 8
Reflections collected	5188
Independent reflections	2585 [R_{int} = 0.0184, R_{sigma} = 0.0363]
Data/restraints/parameters	2585/0/164
Goodness-of-fit on F^2	1.040
Final R indexes [I>=2σ (I)]	R_1 = 0.0466, wR_2 = 0.1031
Final R indexes [all data]	R_1 = 0.0692, wR_2 = 0.1171
Largest diff. peak/hole / e Å^{-3}	0.15/-0.19

X-ray crystallographic analysis of compound 14 (CCDC 1524267)

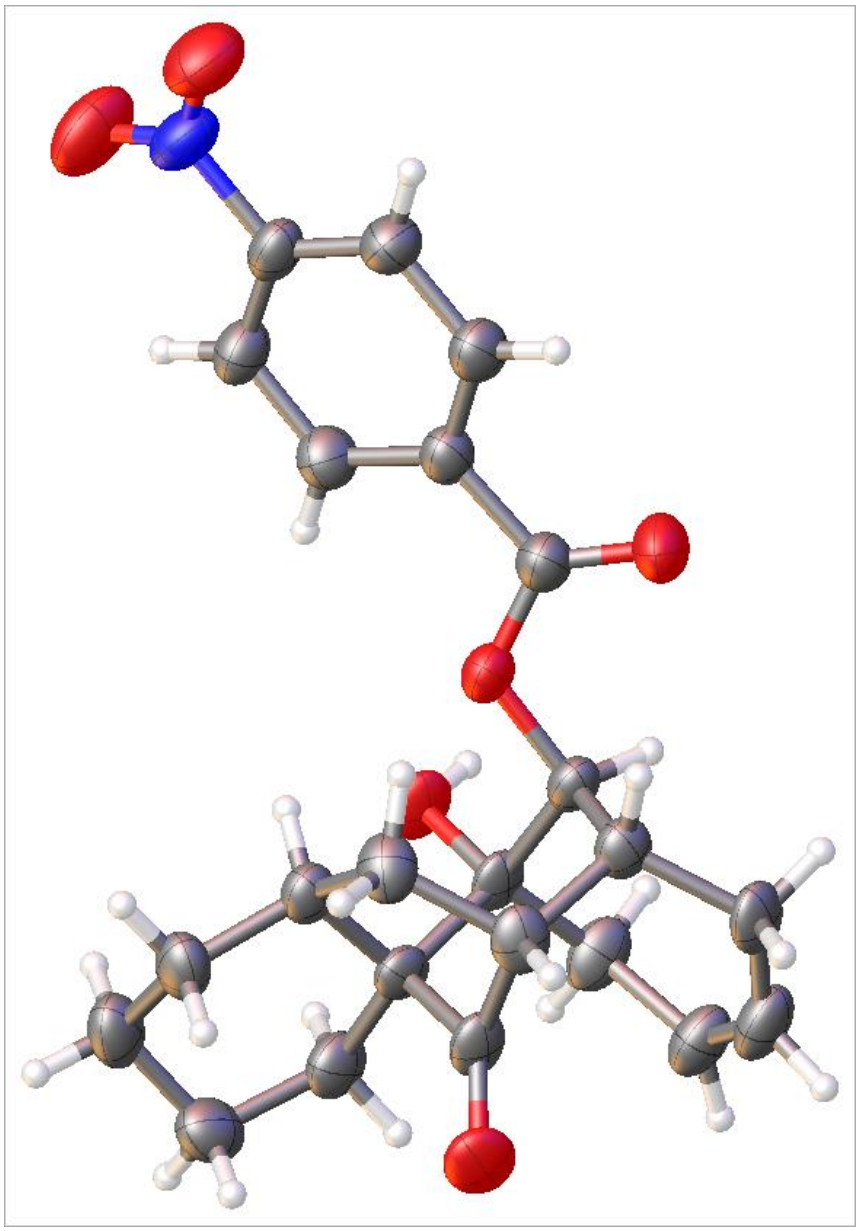
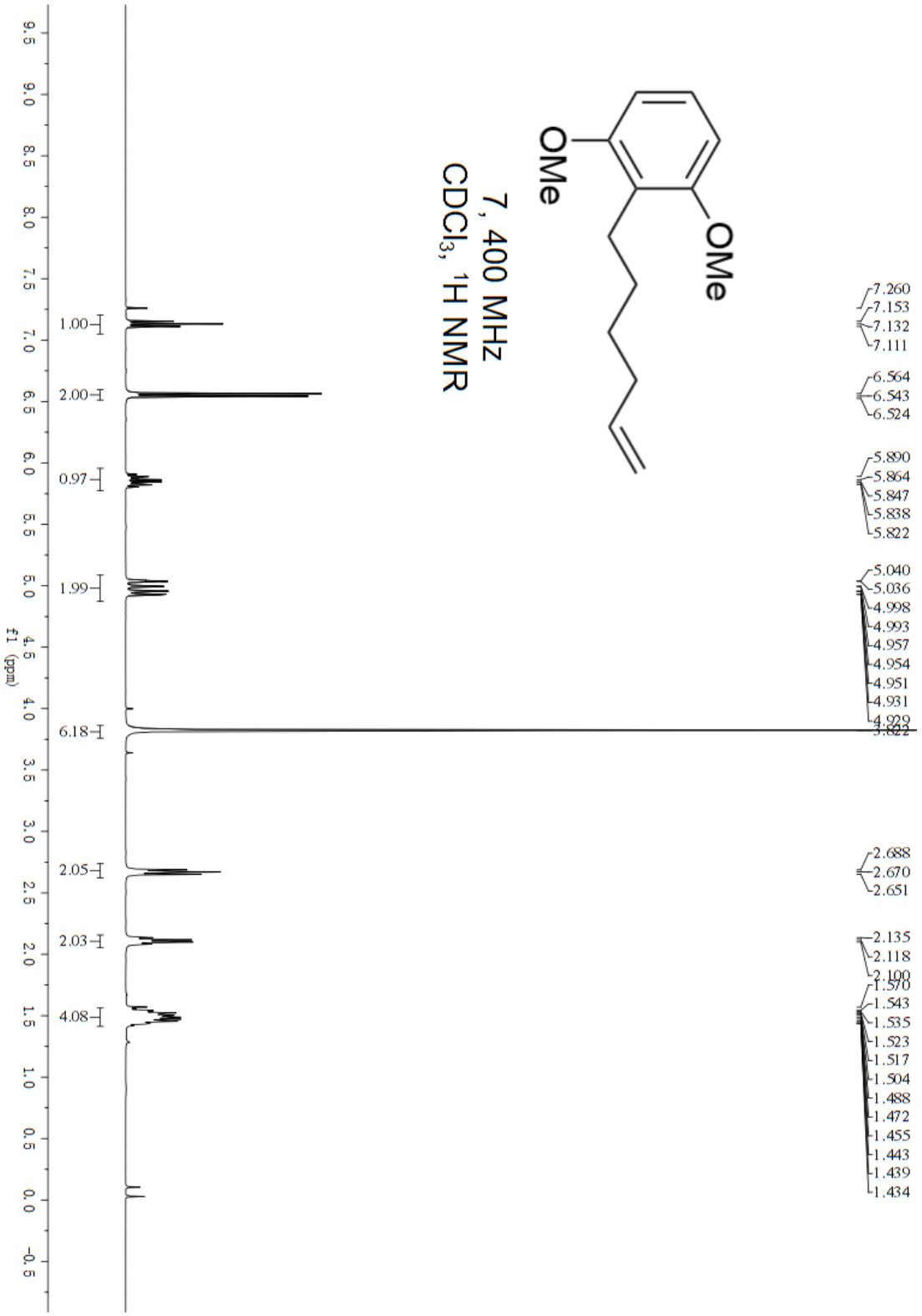


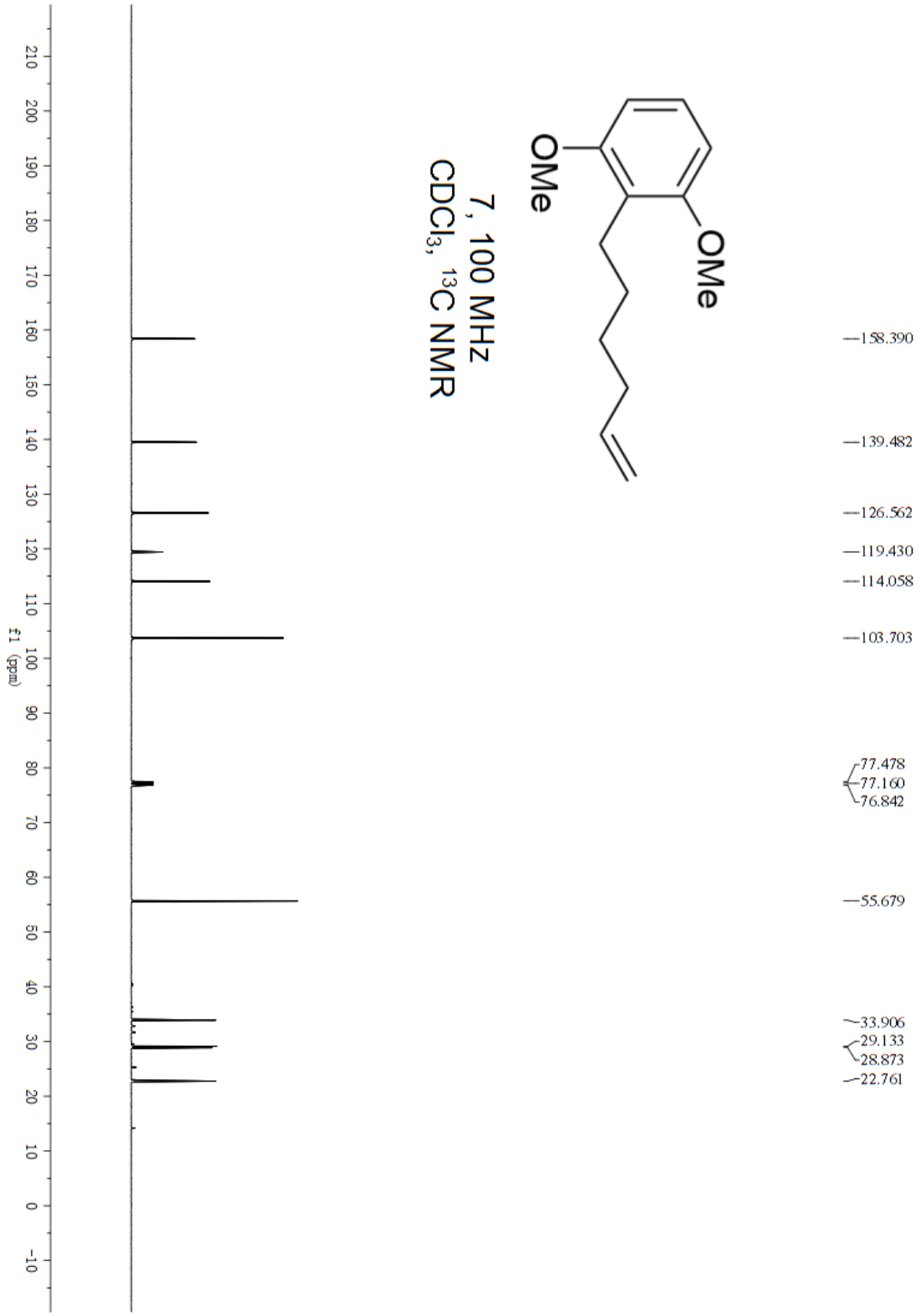
Table Crystal data and structure refinement for **14**

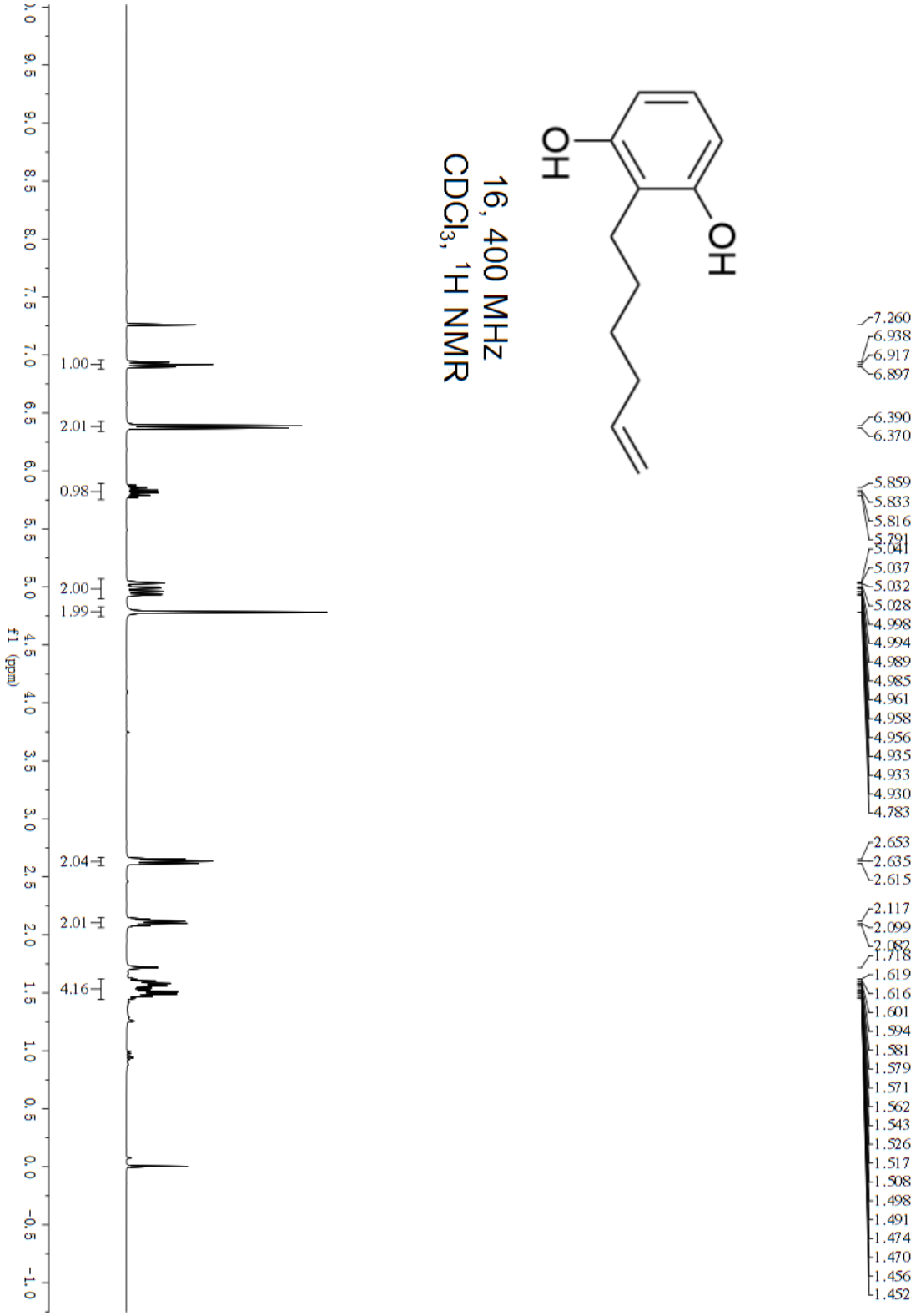
Identification code	14
Empirical formula	$C_{23}H_{25}NO_6$
Formula weight	411.44
Temperature/K	293.15
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	7.941(3)
b/Å	34.688(11)

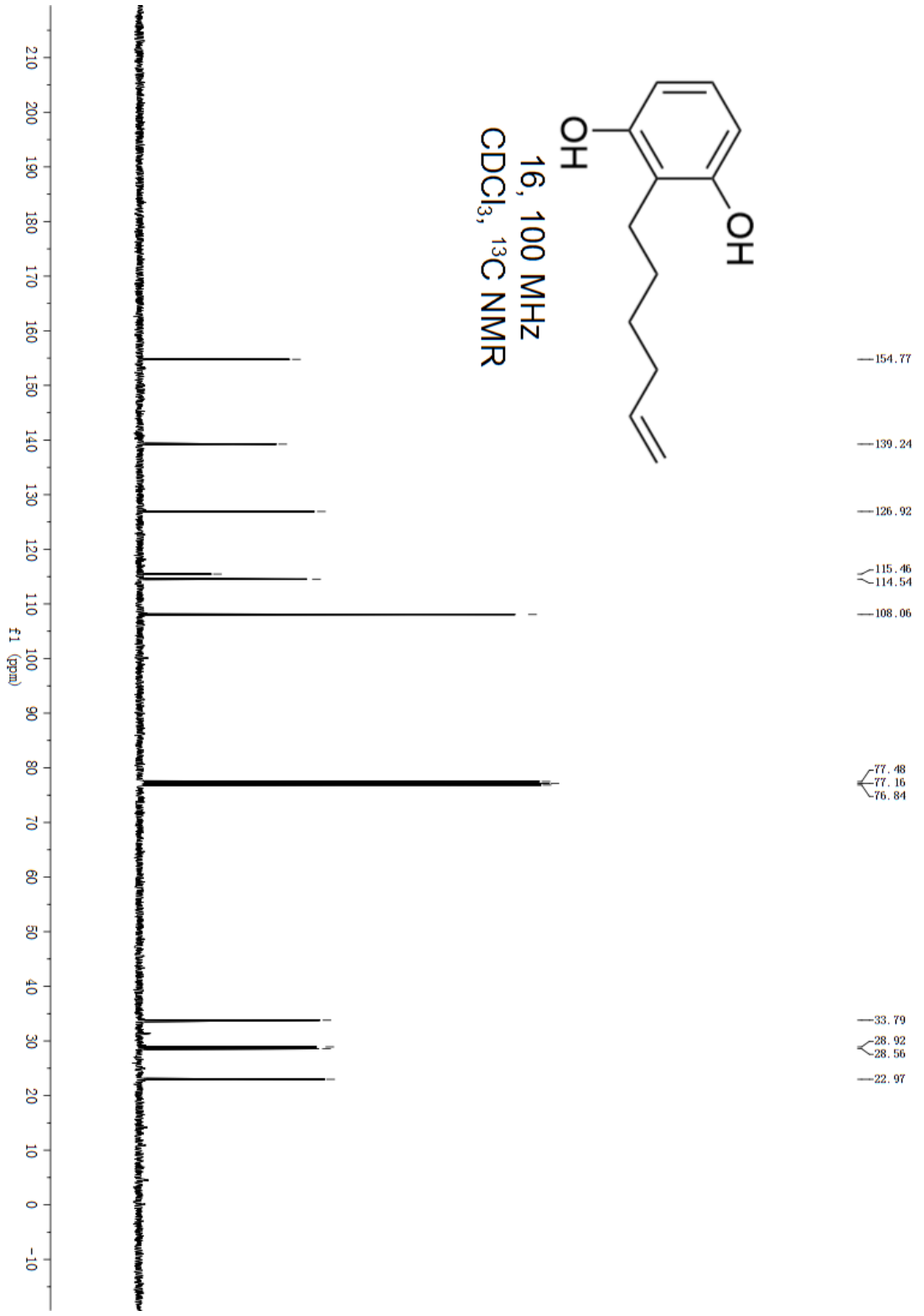
c/Å	7.185(3)
α/°	90
β/°	105.71(5)
γ/°	90
Volume/Å^3	1905.2(14)
Z	4
ρ_{calc}g/cm^3	1.434
μ/mm^{-1}	0.104
F(000)	872.0
Crystal size/mm^3	0.35 × 0.35 × 0.3
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.824 to 52.734
Index ranges	-9 ≤ h ≤ 9, -43 ≤ k ≤ 41, -8 ≤ l ≤ 7
Reflections collected	11738
Independent reflections	3853 [R_{int} = 0.1247, R_{sigma} = 0.1003]
Data/restraints/parameters	3853/0/272
Goodness-of-fit on F^2	1.215
Final R indexes [I>=2σ (I)]	R_1 = 0.1412, wR_2 = 0.3486
Final R indexes [all data]	R_1 = 0.1760, wR_2 = 0.3863
Largest diff. peak/hole / e Å^{-3}	0.56/-0.66

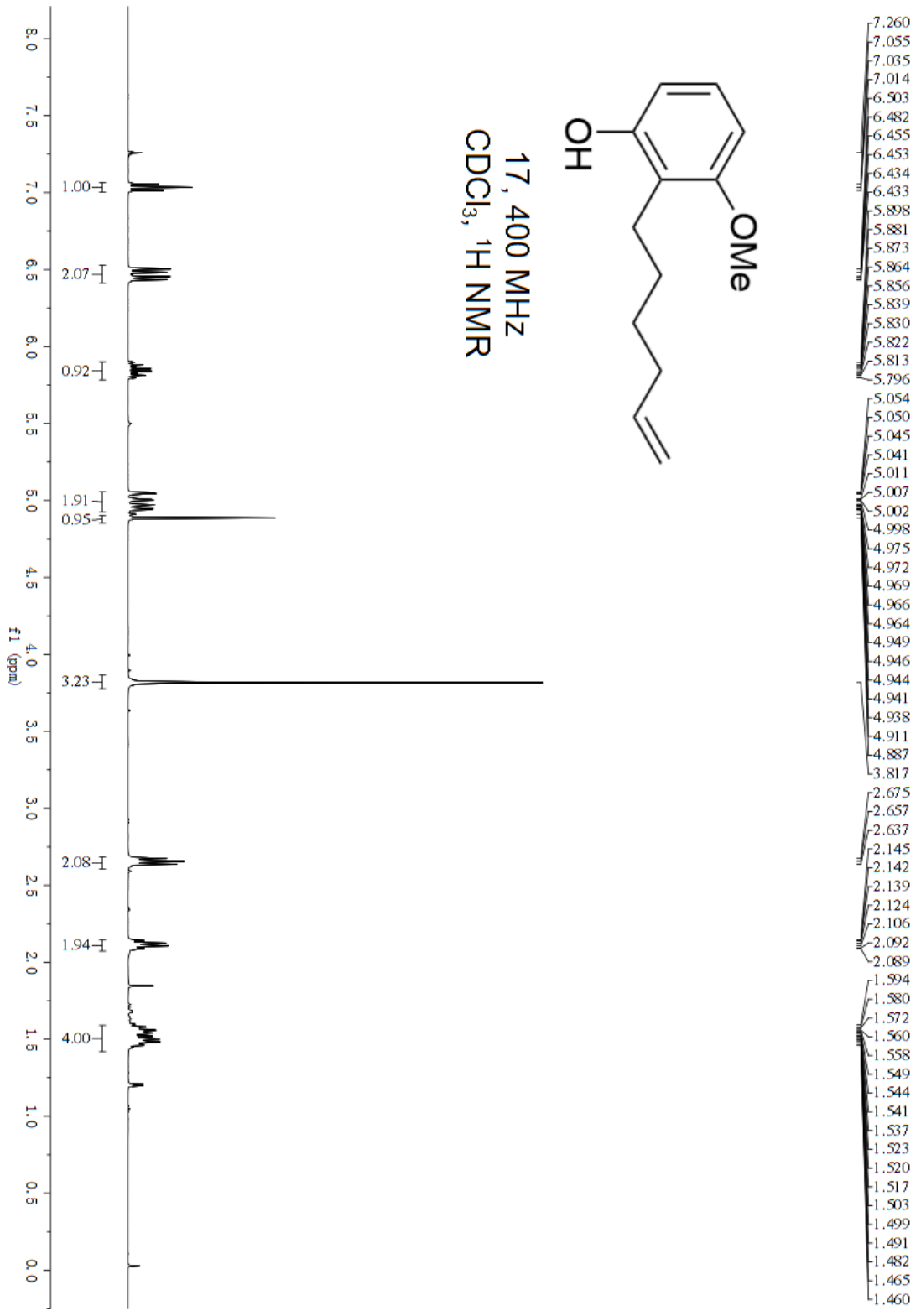
NMR Spectra

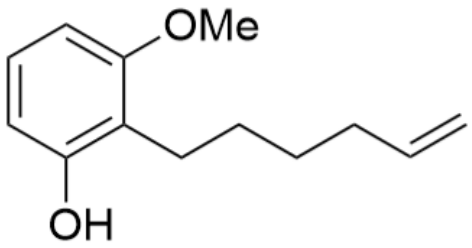

7, 400 MHz
CDCl3, 1H NMR
OMe
OMe
f1 (ppm)
1.00
2.00
0.97
1.99
6.18
2.05
2.03
4.08
7.260
7.153
7.132
7.111
6.564
6.543
6.524
5.890
5.864
5.847
5.838
5.822
5.040
5.036
4.998
4.993
4.957
4.954
4.951
4.931
2.688
2.670
2.651
2.135
2.118
2.100
1.570
1.543
1.535
1.523
1.517
1.504
1.488
1.472
1.455
1.443
1.439
1.434


7, 100 MHz
CDCl3, 13C NMR
OMe
OMe
158.390
139.482
126.562
119.430
114.058
103.703
77.478
77.160
76.842
55.679
33.906
29.133
28.873
22.761
f1 (ppm)

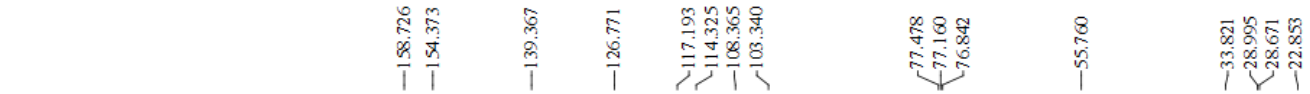

16, 400 MHz
CDCl3, 1H NMR
OH
OH
1.00
2.01
0.98
2.00
1.99
2.04
2.01
4.16
f1 (ppm)
7.260
6.938
6.917
6.897
6.390
6.370
5.859
5.833
5.816
5.791
5.041
5.037
5.032
5.028
4.998
4.994
4.989
4.985
4.961
4.958
4.956
4.935
4.933
4.930
4.783
2.653
2.635
2.615
2.117
2.099
2.082
1.718
1.619
1.616
1.601
1.594
1.581
1.579
1.571
1.562
1.543
1.526
1.517
1.508
1.498
1.491
1.474
1.470
1.456
1.452

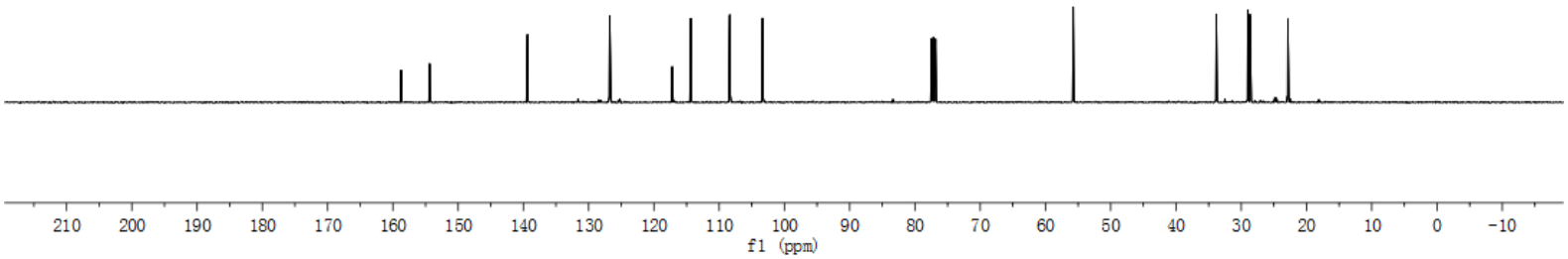

16, 100 MHz
CDCl3, 13C NMR
OH
OH
154.77
139.24
126.92
115.46
114.54
108.06
77.48
77.16
76.84
33.79
28.92
28.56
22.97
f1 (ppm)

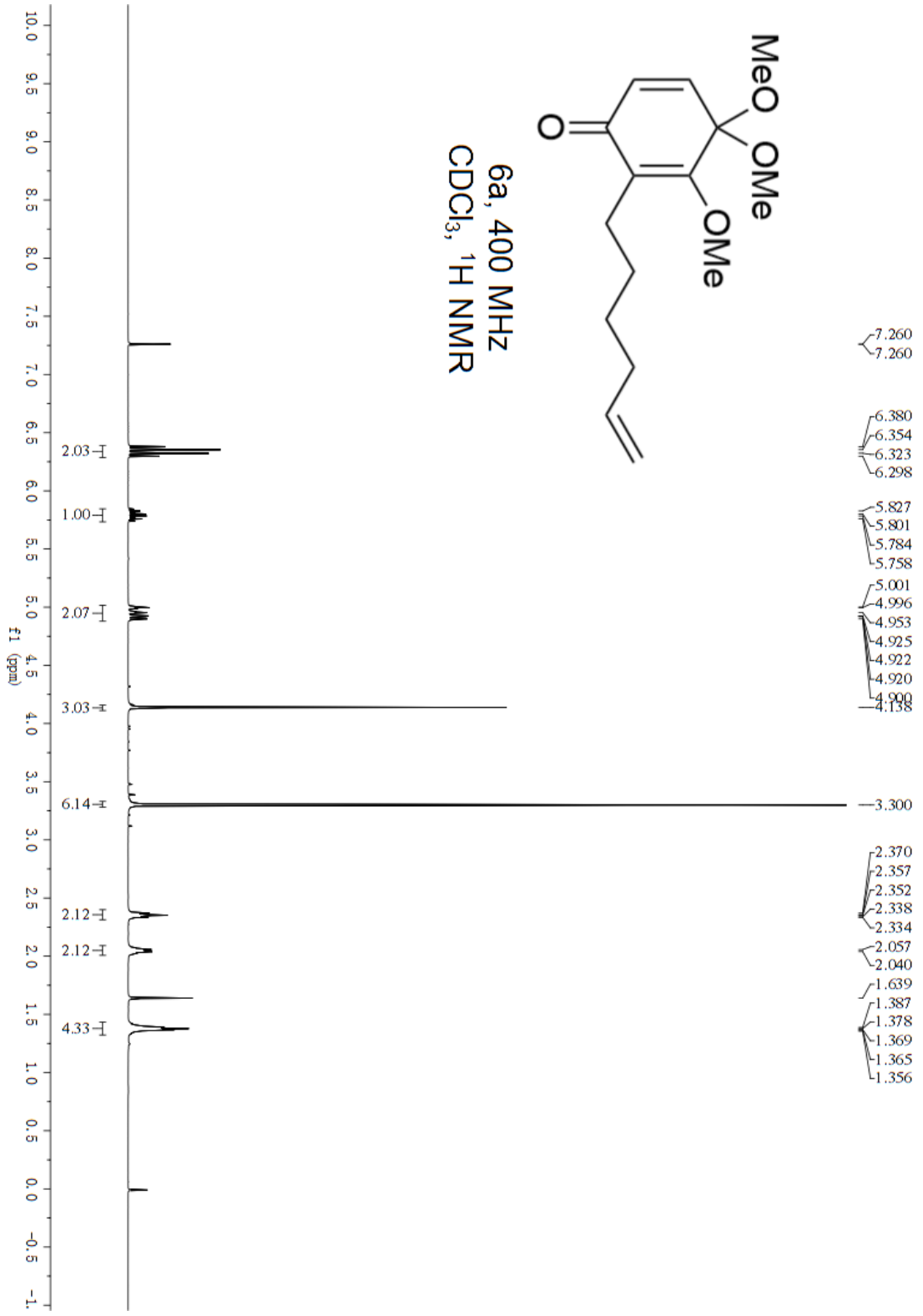

17, 400 MHz
CDCl3, 1H NMR
OH
OMe
f1 (ppm)
1.00
2.07
0.92
1.91
0.95
3.23
2.08
1.94
4.00
7.260
7.055
7.035
7.014
6.503
6.482
6.455
6.453
6.434
6.433
5.898
5.881
5.873
5.864
5.856
5.839
5.830
5.822
5.813
5.796
5.054
5.050
5.045
5.041
5.011
5.007
5.002
4.998
4.975
4.972
4.969
4.966
4.964
4.949
4.946
4.944
4.941
4.938
4.911
4.887
3.817
2.675
2.657
2.637
2.145
2.142
2.139
2.124
2.106
2.092
2.089
1.594
1.580
1.572
1.560
1.558
1.549
1.544
1.541
1.537
1.523
1.520
1.517
1.503
1.499
1.491
1.482
1.465
1.460

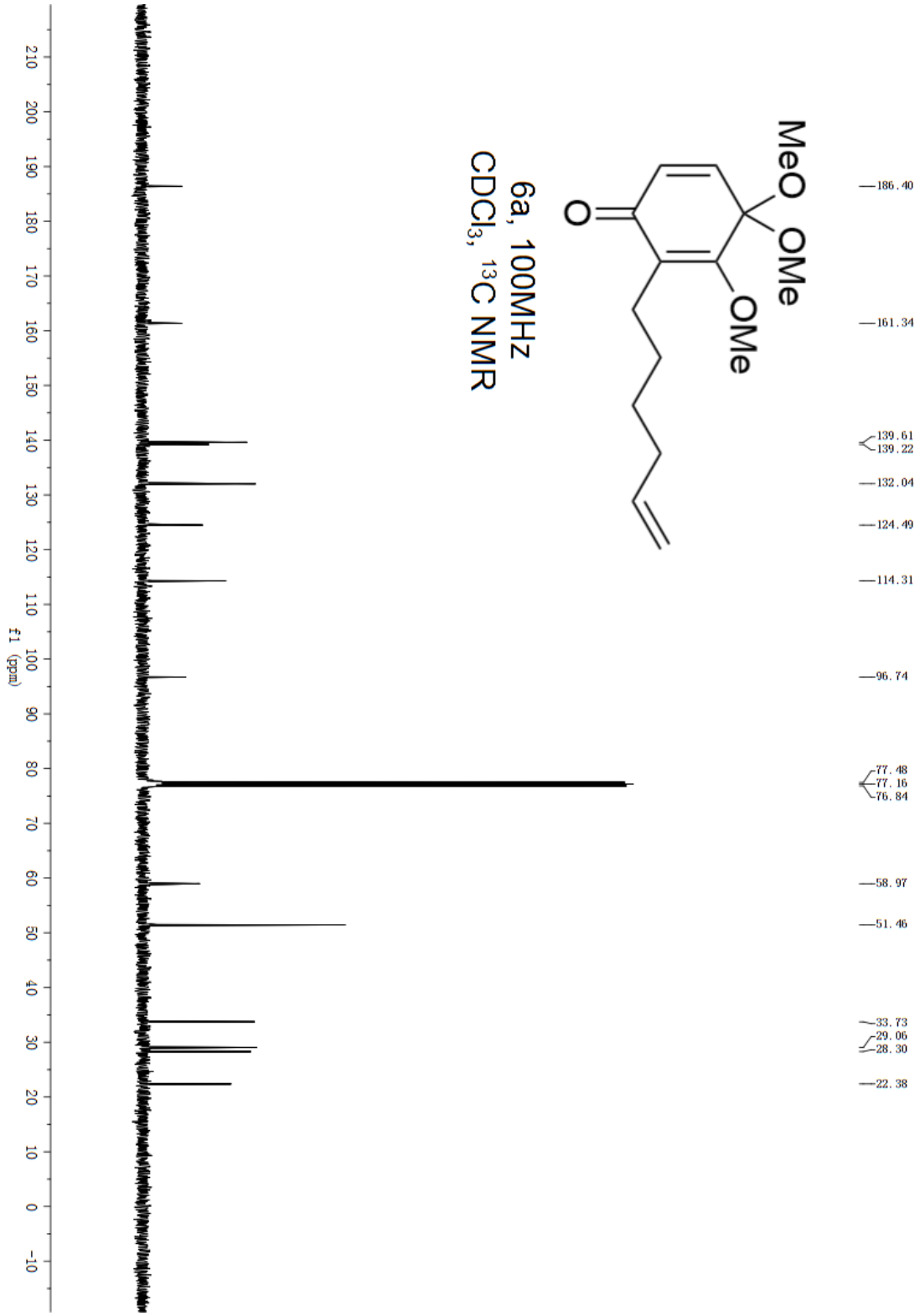


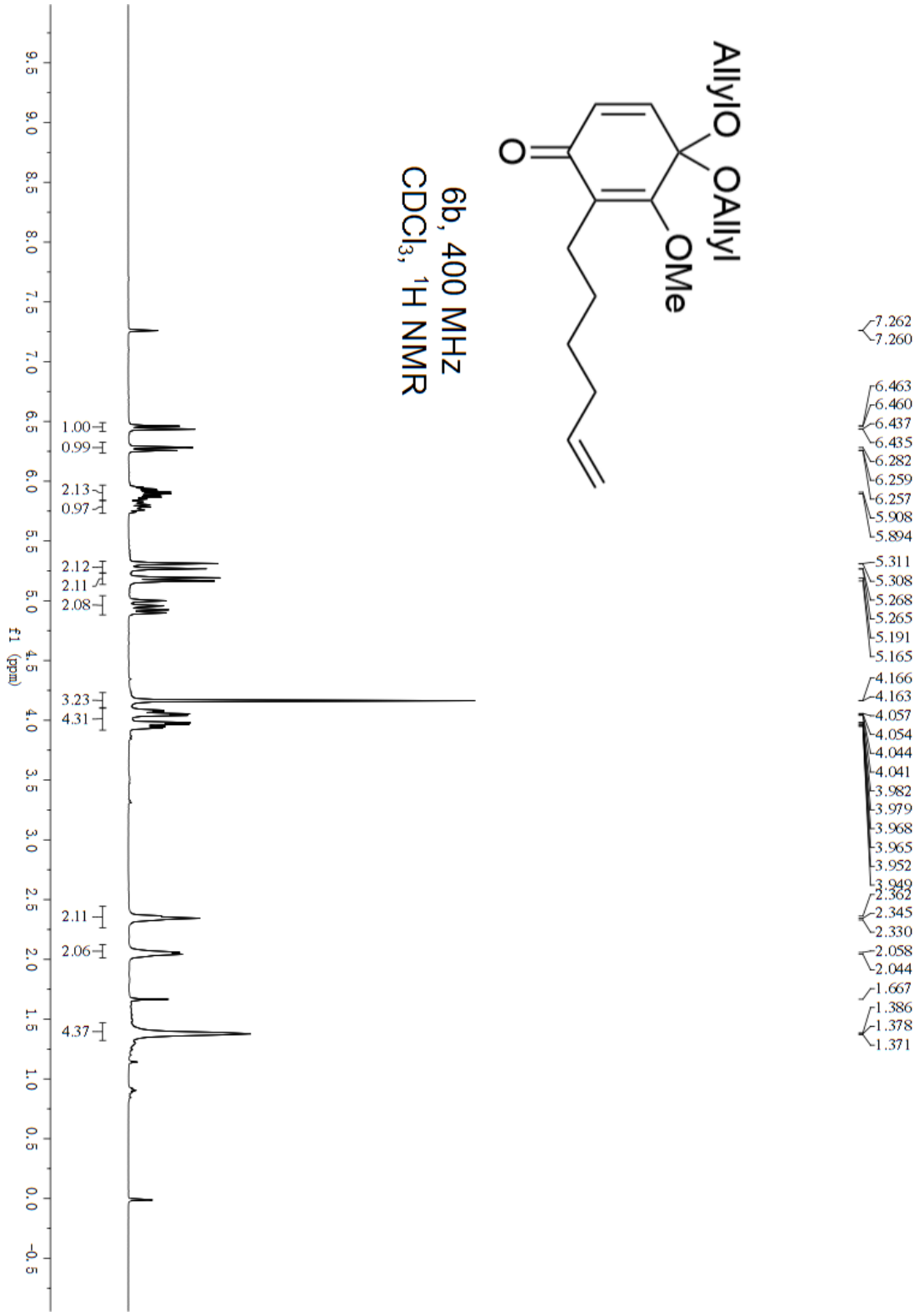
17, 100 MHz
$CDCl_3$, ^{13}C NMR

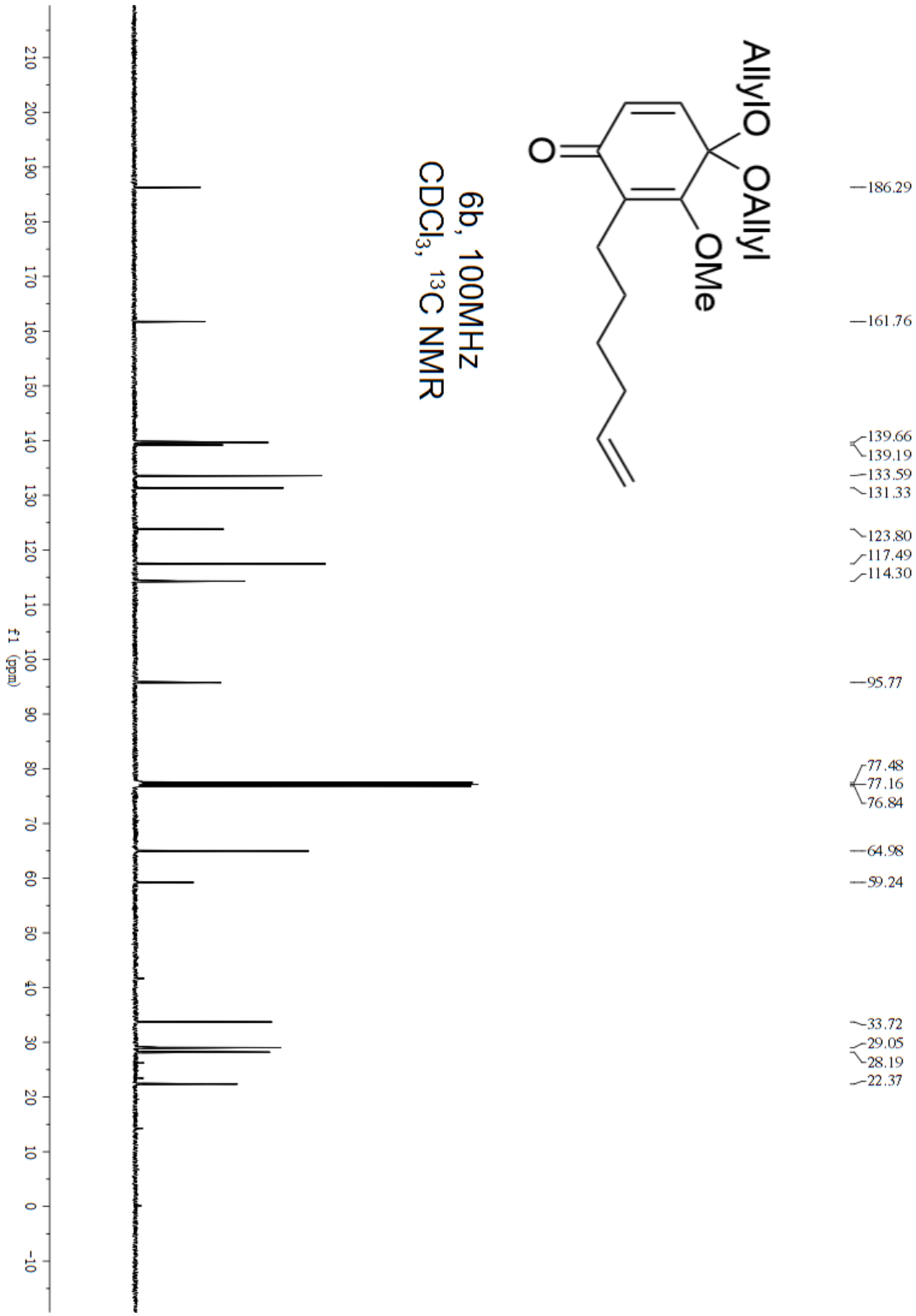


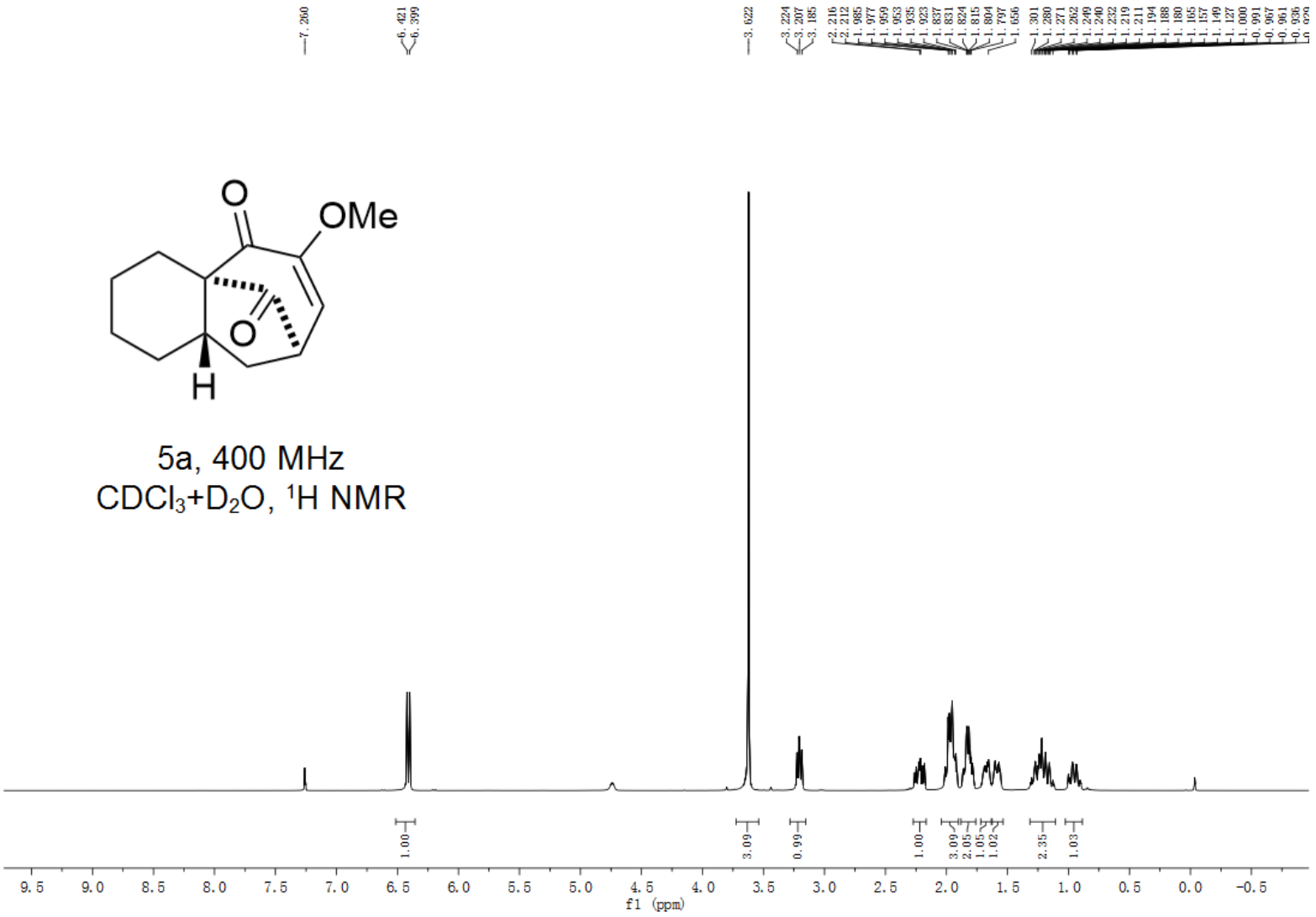


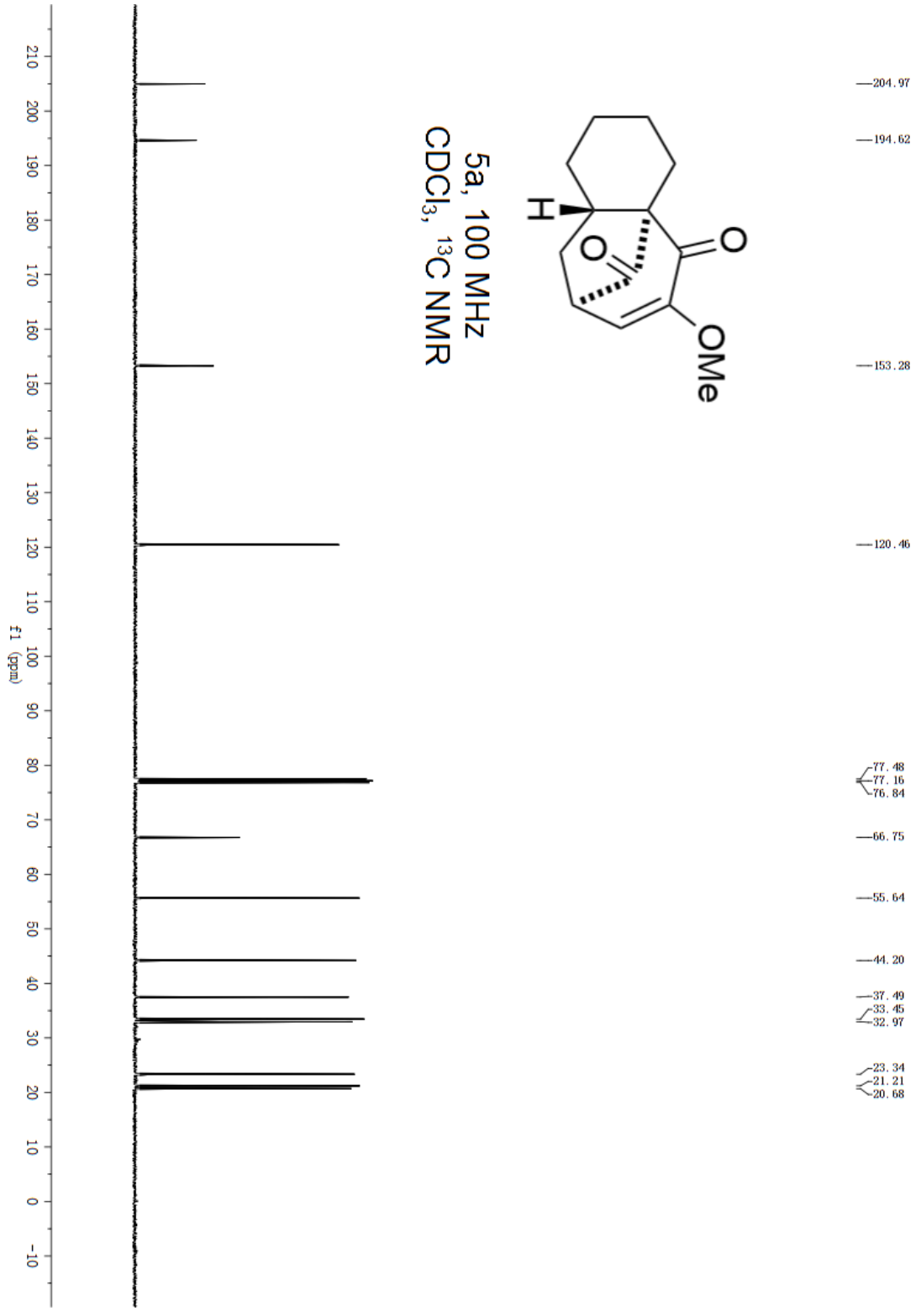

6a, 400 MHz
CDCl3, 1H NMR
MeO
OMe
OMe
O
f1 (ppm)
2.03
1.00
2.07
3.03
6.14
2.12
2.12
4.33
7.260
7.260
6.380
6.354
6.323
6.298
5.827
5.801
5.784
5.758
5.001
4.996
4.953
4.925
4.922
4.920
4.900
4.138
3.300
2.370
2.357
2.352
2.338
2.334
2.057
2.040
1.639
1.387
1.378
1.369
1.365
1.356

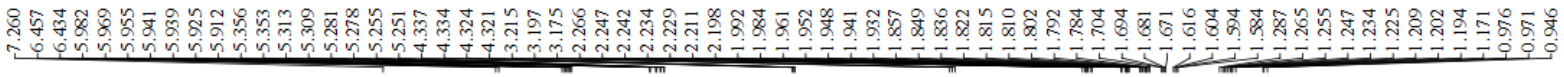

6a, 100MHz
CDCl3, 13C NMR
MeO OMe
OMe
O
186.40
161.34
139.61
139.22
132.04
124.49
114.31
96.74
77.48
77.16
76.84
58.97
51.46
33.73
29.06
28.30
22.38
f1 (ppm)

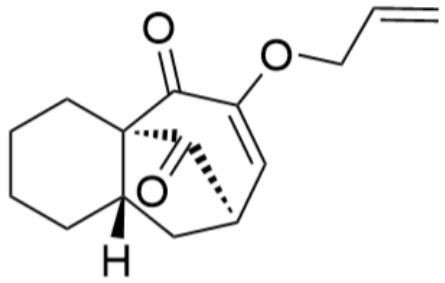

6b, 400 MHz
CDCl3, 1H NMR
AllylO
OAllyl
OMe
O
f1 (ppm)
1.00
0.99
2.13
0.97
2.12
2.11
2.08
3.23
4.31
2.11
2.06
4.37
7.262
7.260
6.463
6.460
6.437
6.435
6.282
6.259
6.257
5.908
5.894
5.311
5.308
5.268
5.265
5.191
5.165
4.166
4.163
4.057
4.054
4.044
4.041
3.982
3.979
3.968
3.965
3.952
3.949
2.362
2.345
2.330
2.058
2.044
1.667
1.386
1.378
1.371


AllylO
OAllyl
OMe
O
6b, 100MHz
CDCl3, 13C NMR
186.29
161.76
139.66
139.19
133.59
131.33
123.80
117.49
114.30
95.77
77.48
77.16
76.84
64.98
59.24
33.72
29.05
28.19
22.37
f1 (ppm)

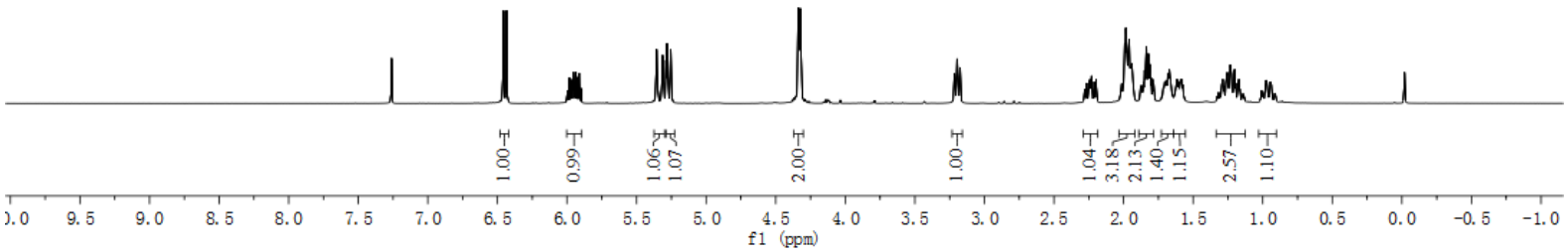

7.260
6.421
6.399
3.622
3.224
3.207
3.185
2.216
2.212
1.985
1.977
1.959
1.953
1.935
1.923
1.837
1.831
1.824
1.815
1.804
1.797
1.656
1.301
1.280
1.271
1.262
1.249
1.240
1.232
1.219
1.211
1.194
1.188
1.180
1.165
1.157
1.149
1.127
1.000
0.991
0.967
0.961
0.936
O
OMe
O
H
5a, 400 MHz
CDCl3+D2O, 1H NMR
1.00
3.09
0.99
1.00
3.09
2.05
1.05
1.02
2.35
1.03
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
-0.5
f1 (ppm)

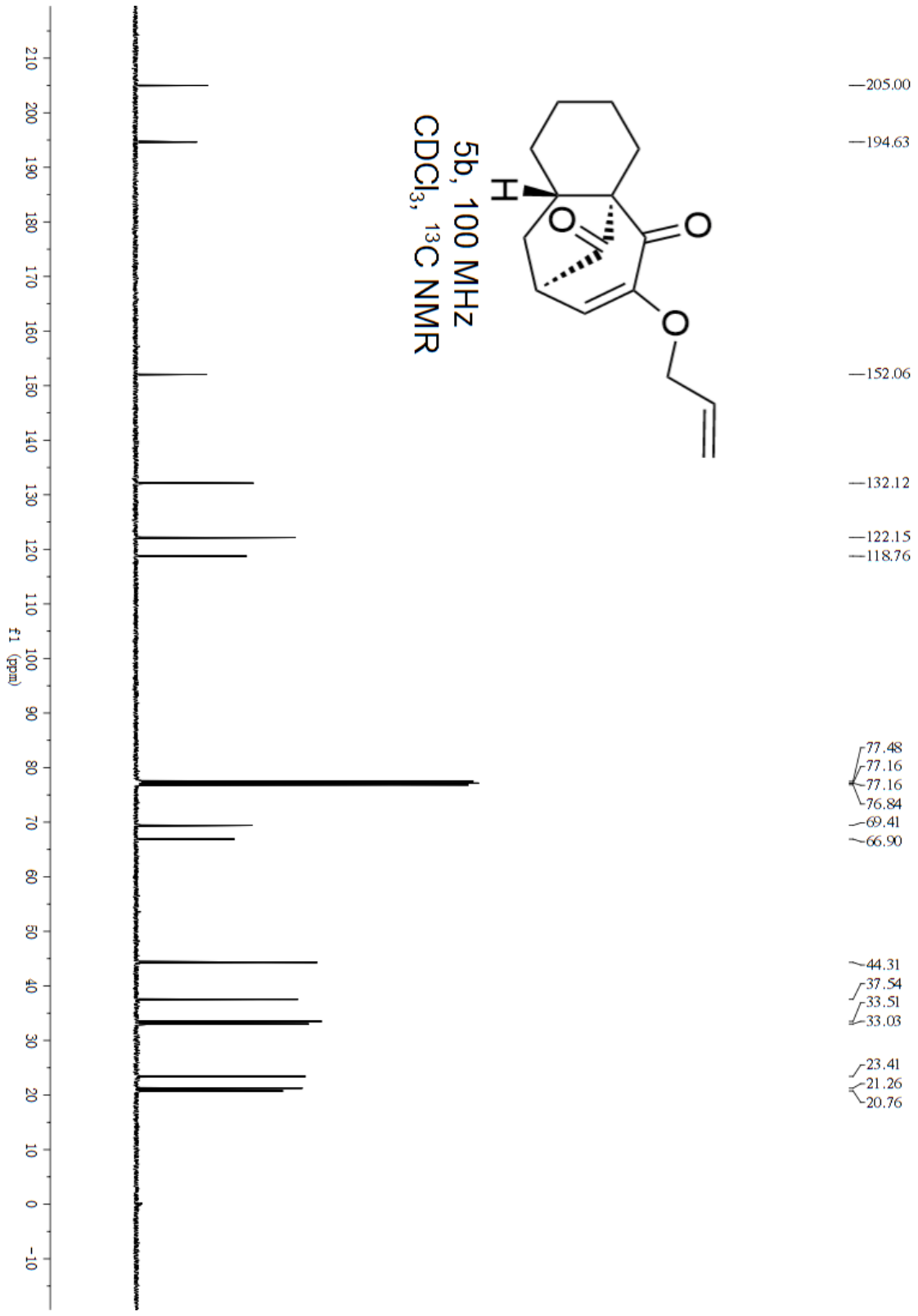

5a, 100 MHz
CDCl3, 13C NMR
H
O
O
OMe
204.97
194.62
153.28
120.46
77.48
77.16
76.84
66.75
55.64
44.20
37.49
33.45
32.97
23.34
21.21
20.68
f1 (ppm)

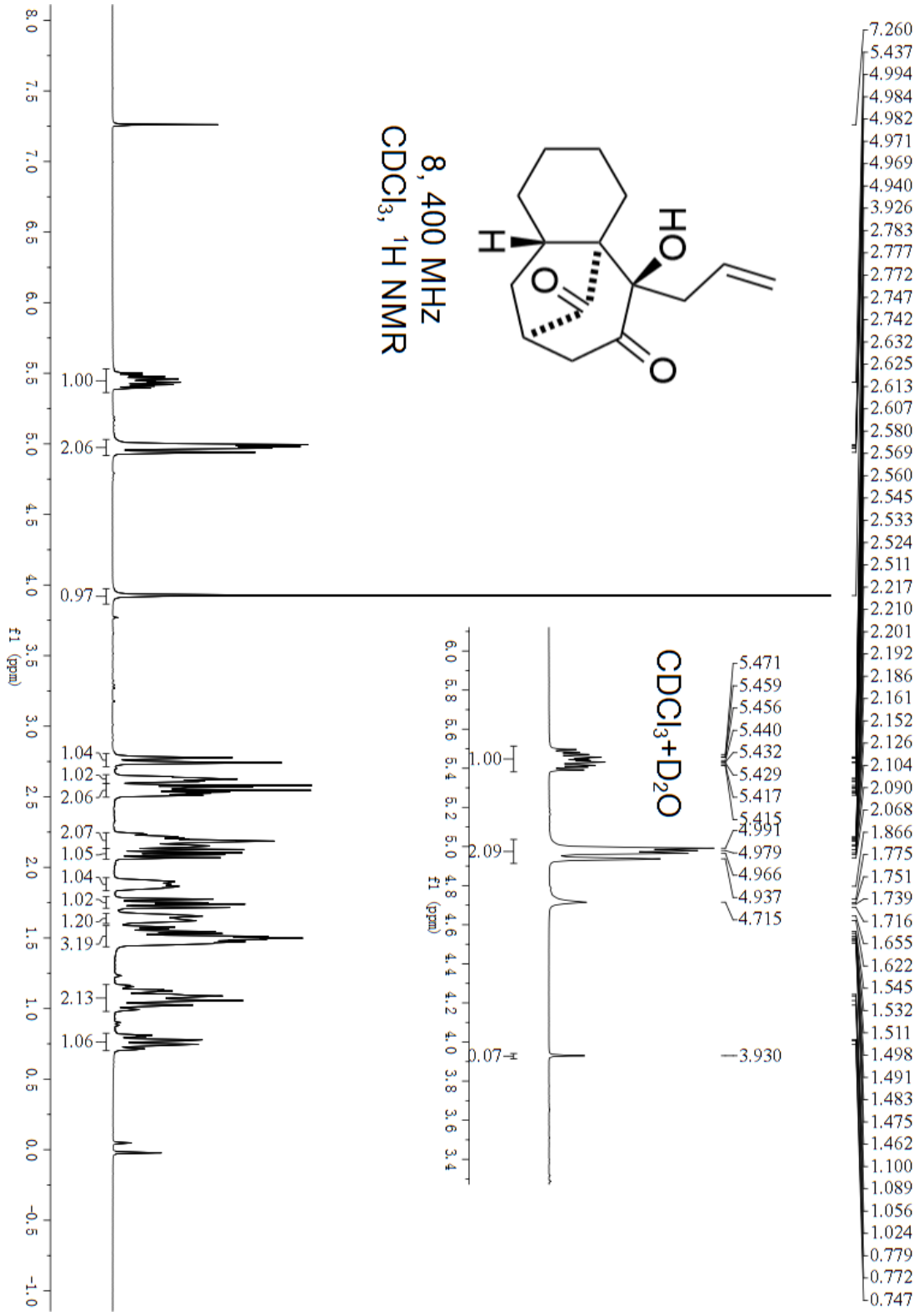


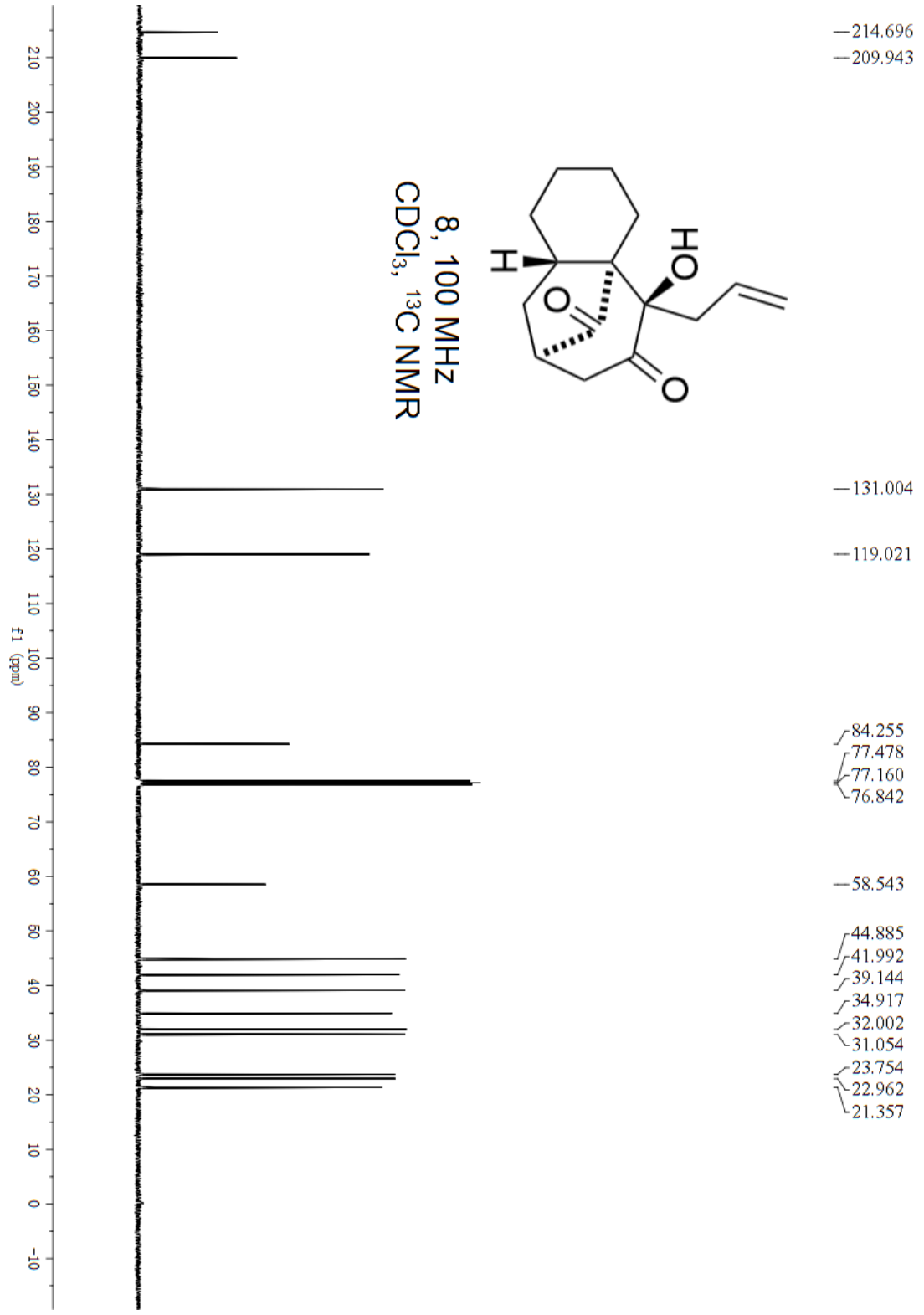


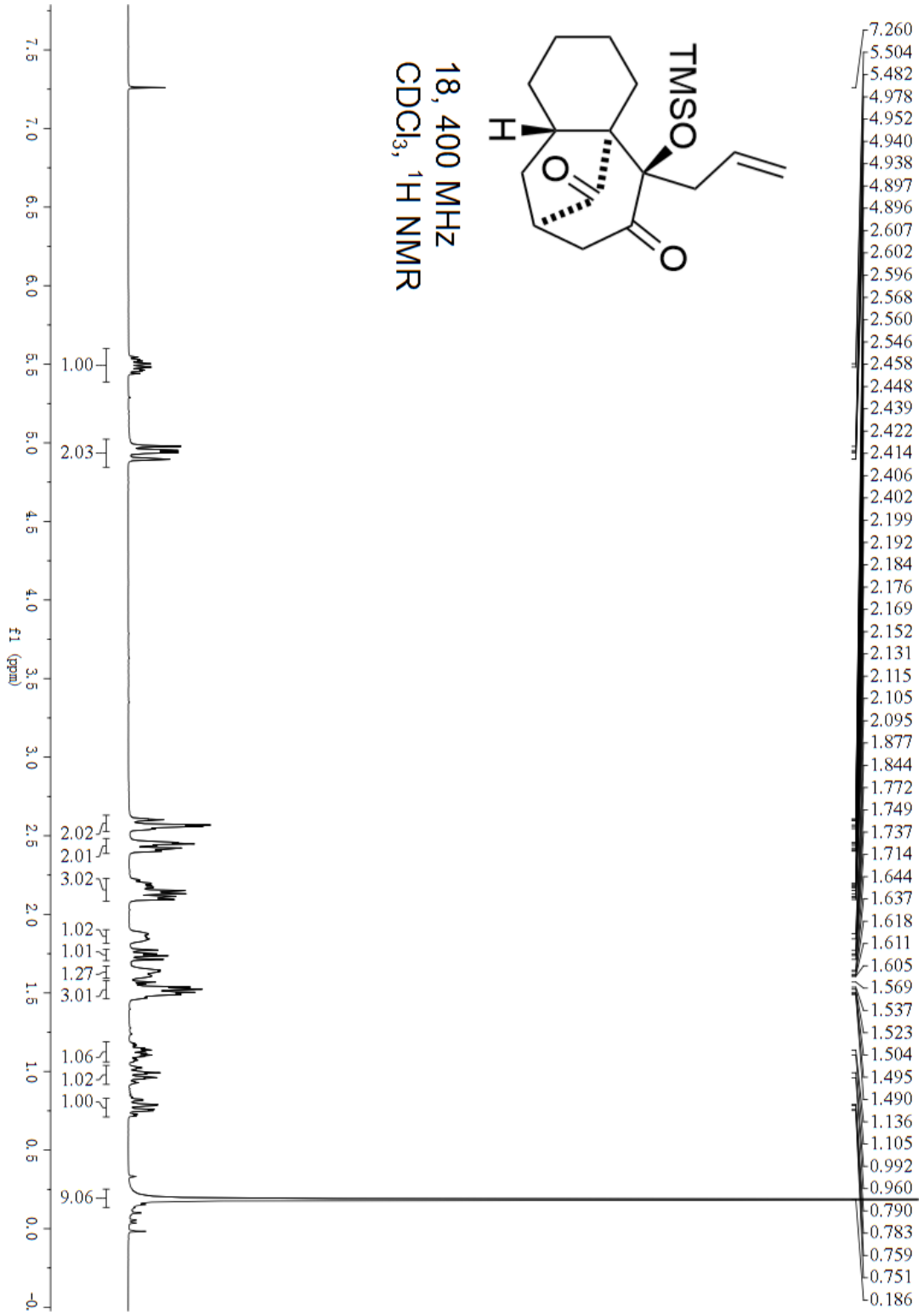
5b, 400 MHz
$CDCl_3$, ^1H NMR

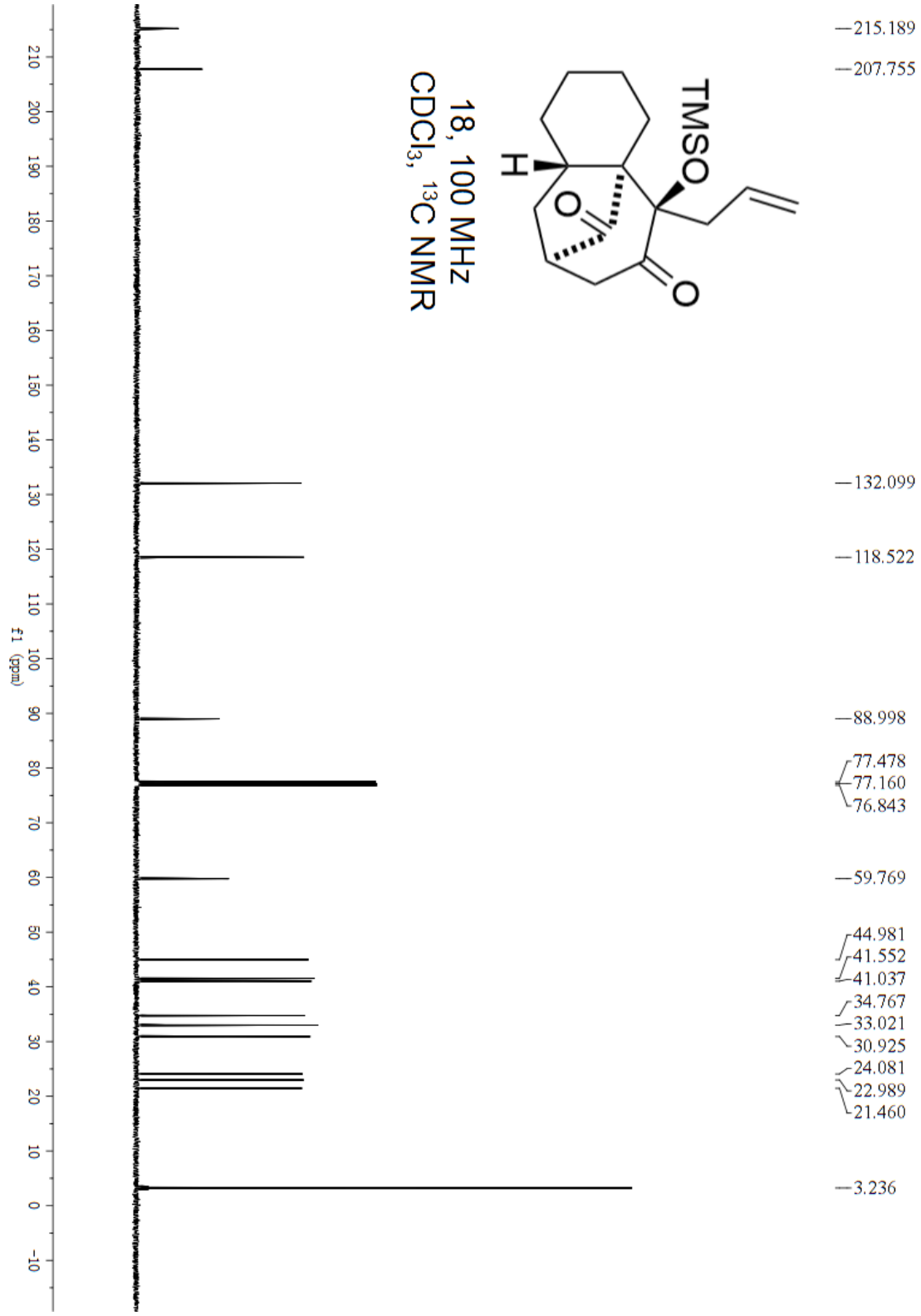


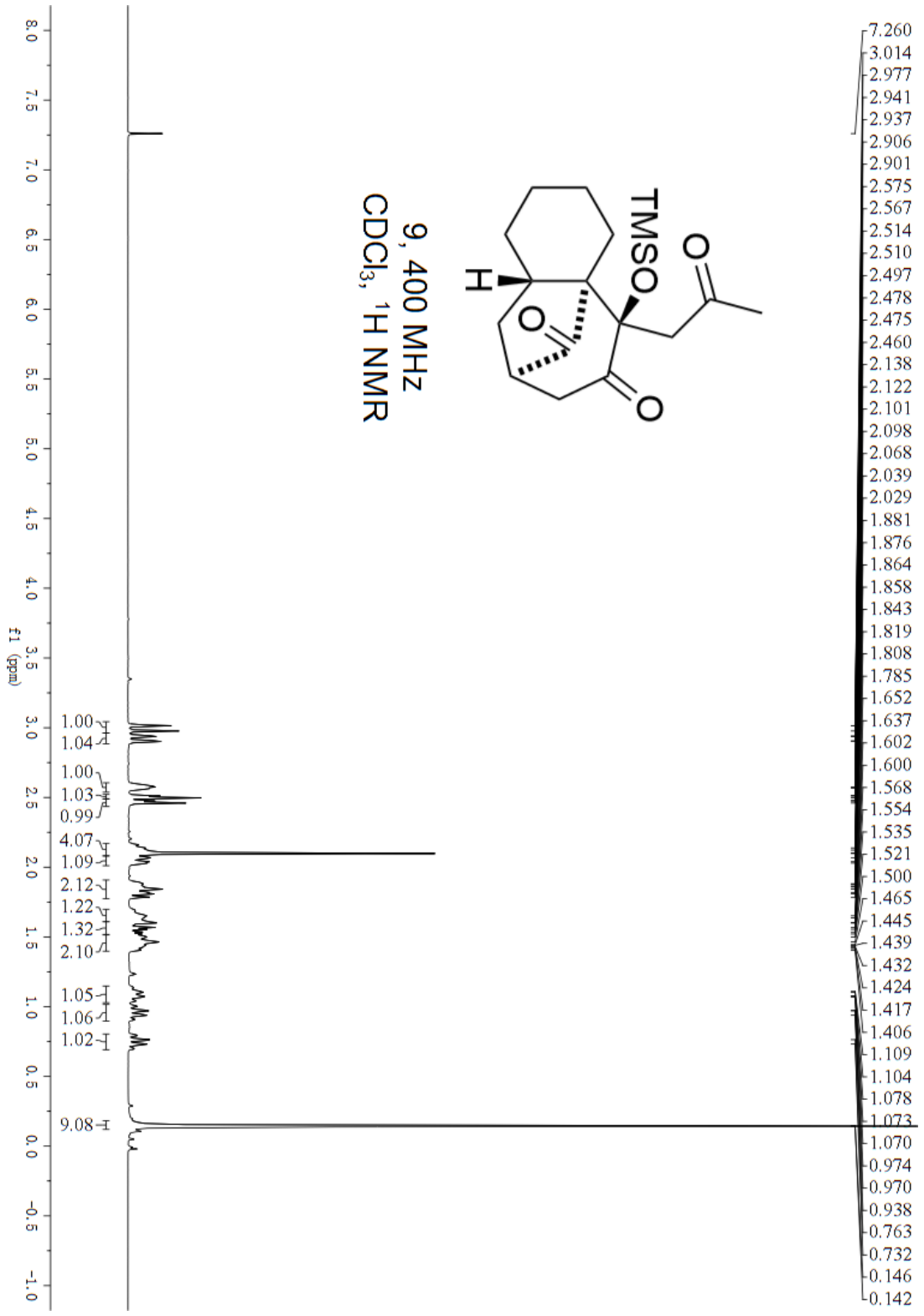

5b, 100 MHz
CDCl3, 13C NMR
205.00
194.63
152.06
132.12
122.15
118.76
77.48
77.16
77.16
76.84
69.41
66.90
44.31
37.54
33.51
33.03
23.41
21.26
20.76
f1 (ppm)

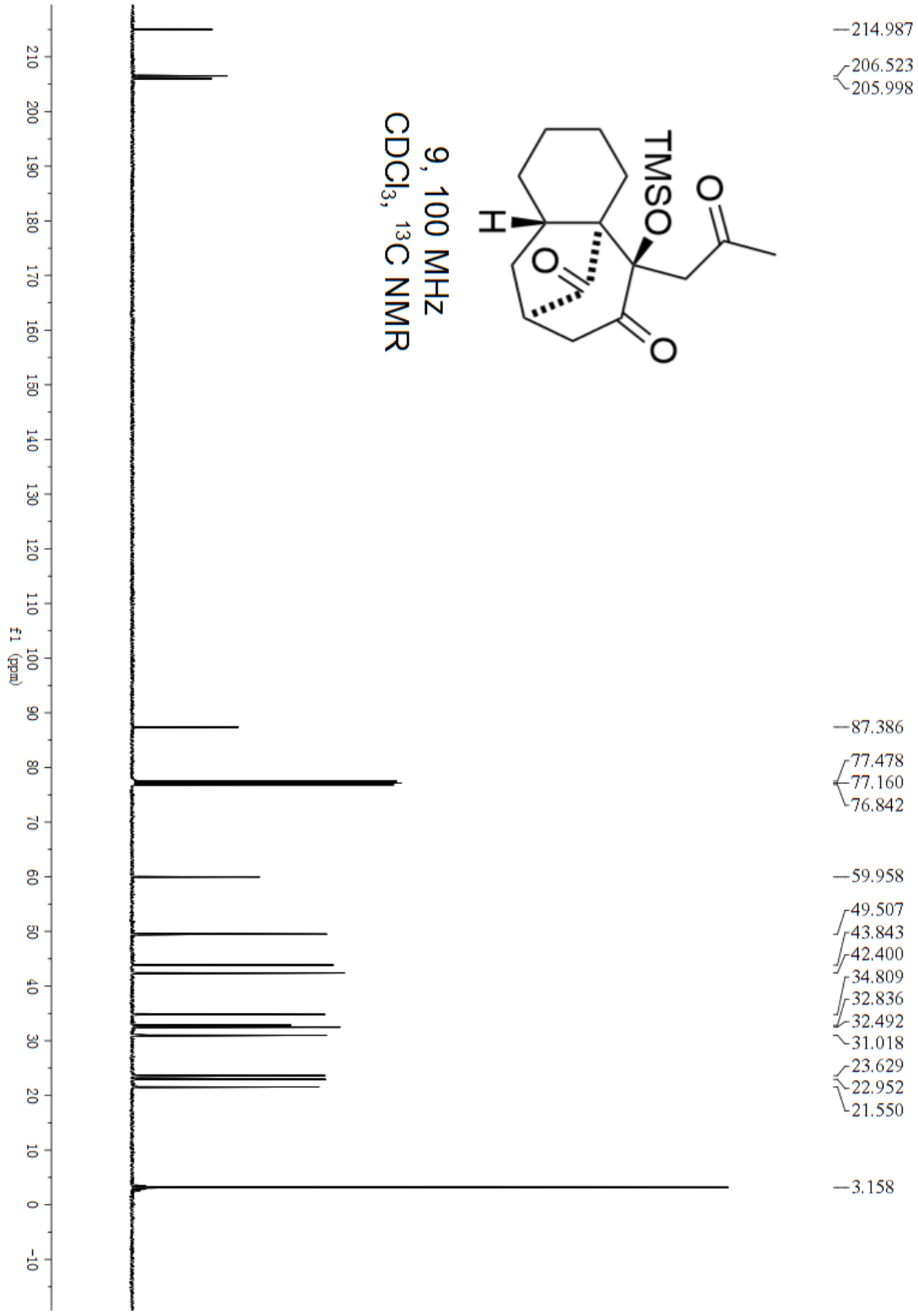

8, 400 MHz
CDCl3, 1H NMR
HO
H
O
O
CDCl3+D2O
f1 (ppm)
1.00
2.06
0.97
1.04
1.02
2.06
2.07
1.05
1.04
1.02
1.20
3.19
2.13
1.06
2.09
0.07
7.260
5.437
4.994
4.984
4.982
4.971
4.969
4.940
3.926
2.783
2.777
2.772
2.747
2.742
2.632
2.625
2.613
2.607
2.580
2.569
2.560
2.545
2.533
2.524
2.511
2.217
2.210
2.201
2.192
2.186
2.161
2.152
2.126
2.104
2.090
2.068
1.866
1.775
1.751
1.739
1.716
1.655
1.622
1.545
1.532
1.511
1.498
1.491
1.483
1.475
1.462
1.100
1.089
1.056
1.024
0.779
0.772
0.747
5.471
5.459
5.456
5.440
5.432
5.429
5.417
5.415
4.991
4.979
4.966
4.937
4.715
3.930

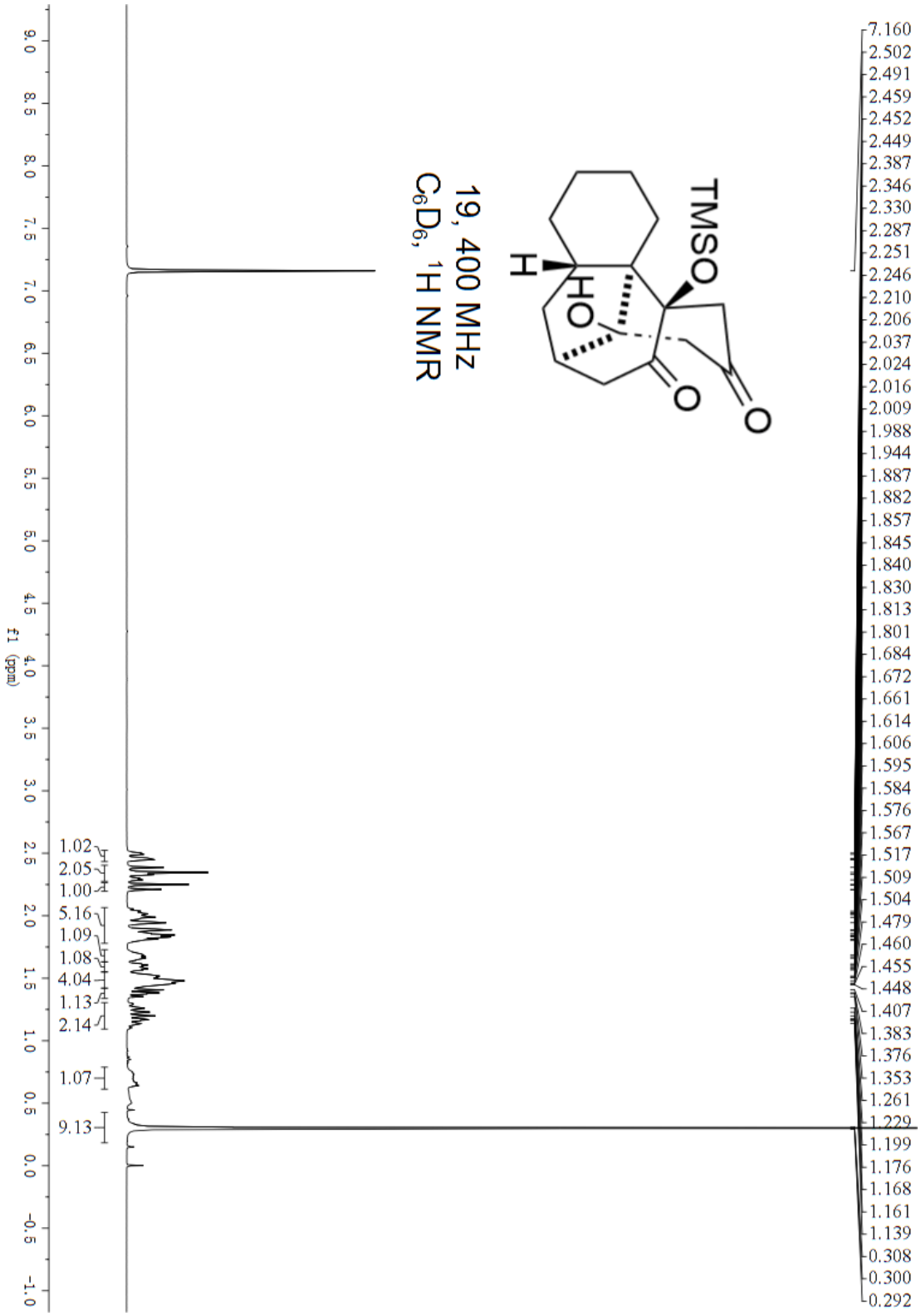

8, 100 MHz
CDCl3, 13C NMR
H
O
HO
O
214.696
209.943
131.004
119.021
84.255
77.478
77.160
76.842
58.543
44.885
41.992
39.144
34.917
32.002
31.054
23.754
22.962
21.357
f1 (ppm)

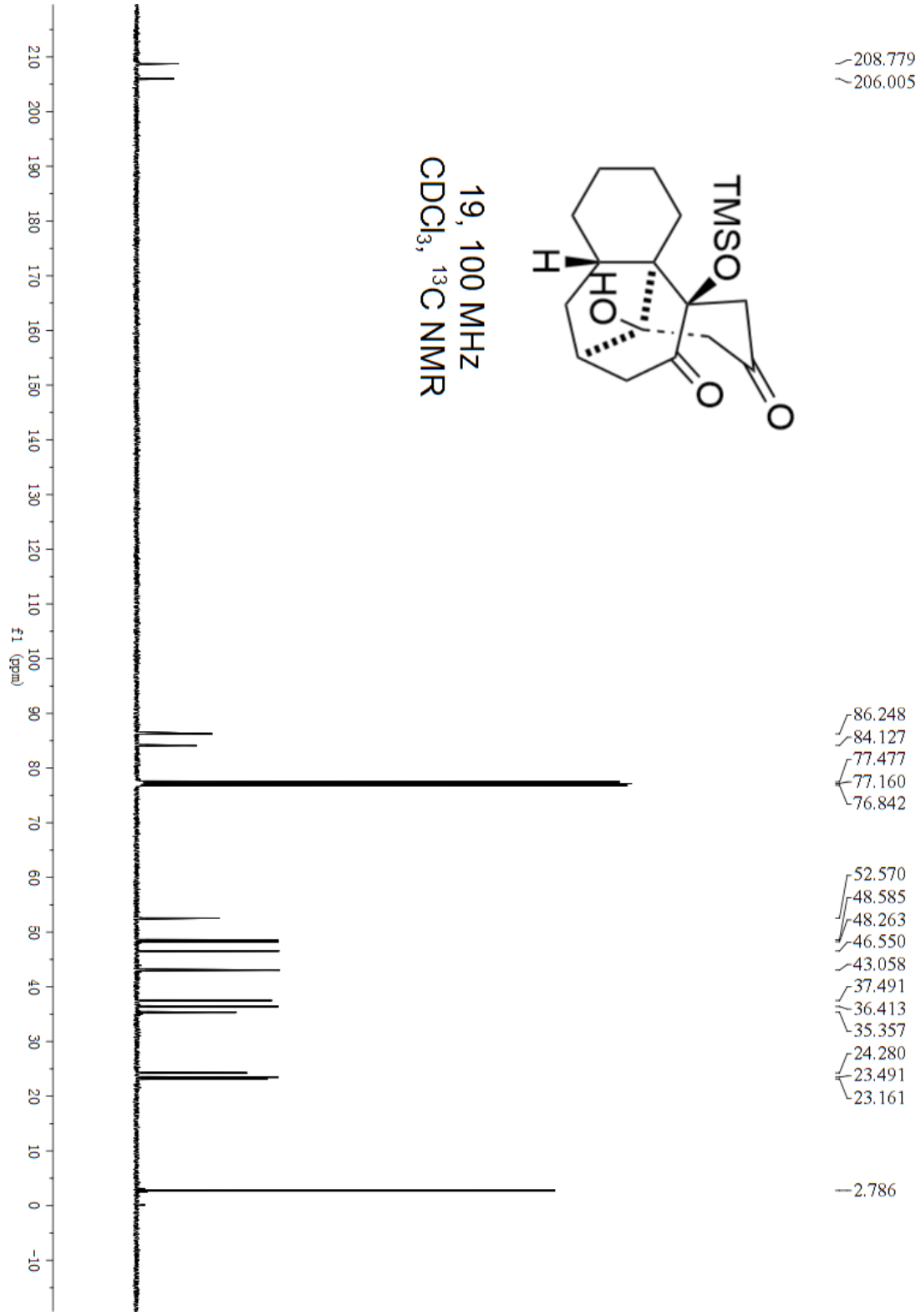

18, 400 MHz
CDCl3, 1H NMR
TMSO
H
O
O
f1 (ppm)
1.00
2.03
2.02
2.01
3.02
1.02
1.01
1.27
3.01
1.06
1.02
1.00
9.06
7.260
5.504
5.482
4.978
4.952
4.940
4.938
4.897
4.896
2.607
2.602
2.596
2.568
2.560
2.546
2.458
2.448
2.439
2.422
2.414
2.406
2.402
2.199
2.192
2.184
2.176
2.169
2.152
2.131
2.115
2.105
2.095
1.877
1.844
1.772
1.749
1.737
1.714
1.644
1.637
1.618
1.611
1.605
1.569
1.537
1.523
1.504
1.495
1.490
1.136
1.105
0.992
0.960
0.790
0.783
0.759
0.751
0.186

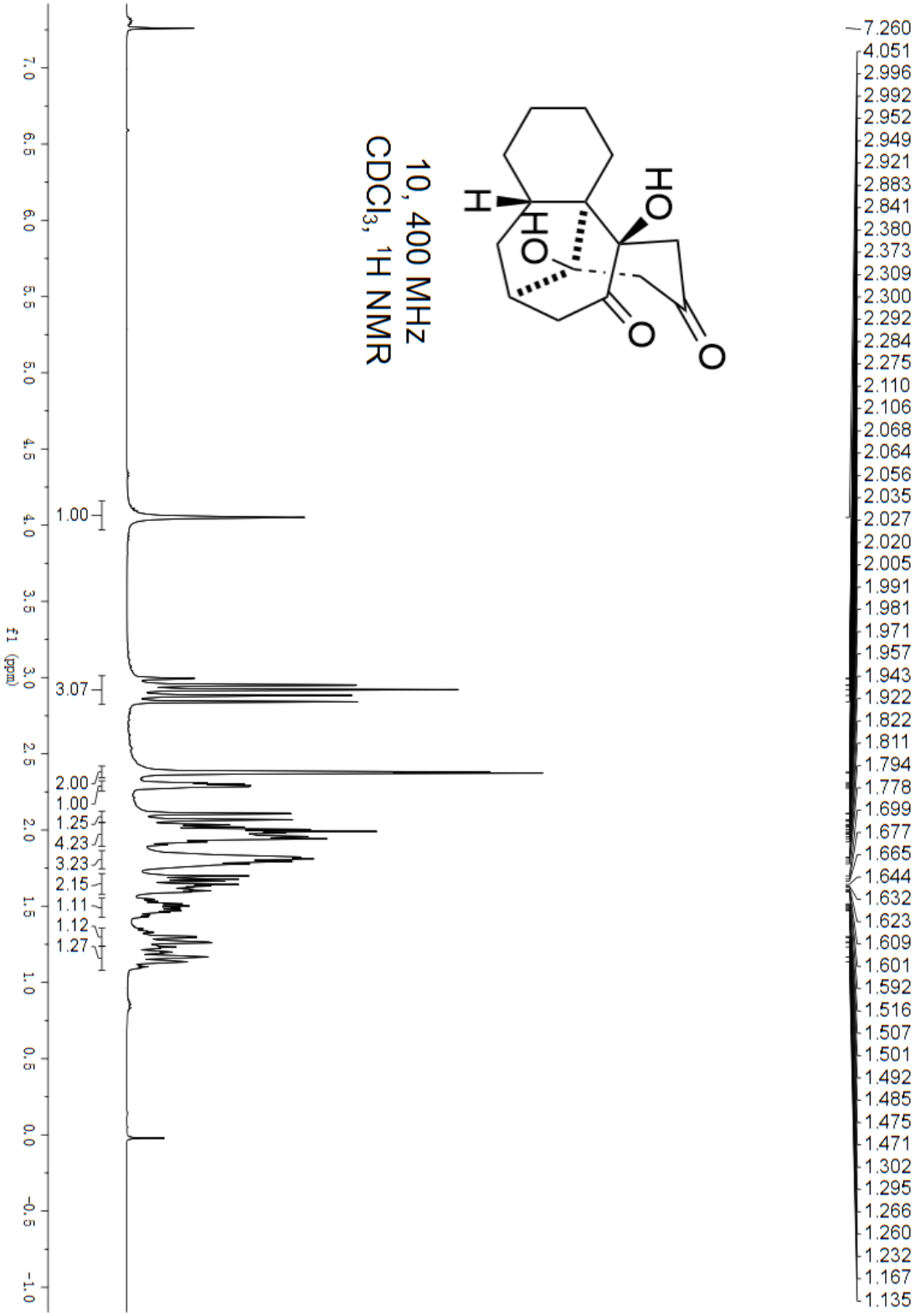

18, 100 MHz
CDCl3, 13C NMR
TMSO
H
O
O
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
215.189
207.755
132.099
118.522
88.998
77.478
77.160
76.843
59.769
44.981
41.552
41.037
34.767
33.021
30.925
24.081
22.989
21.460
3.236

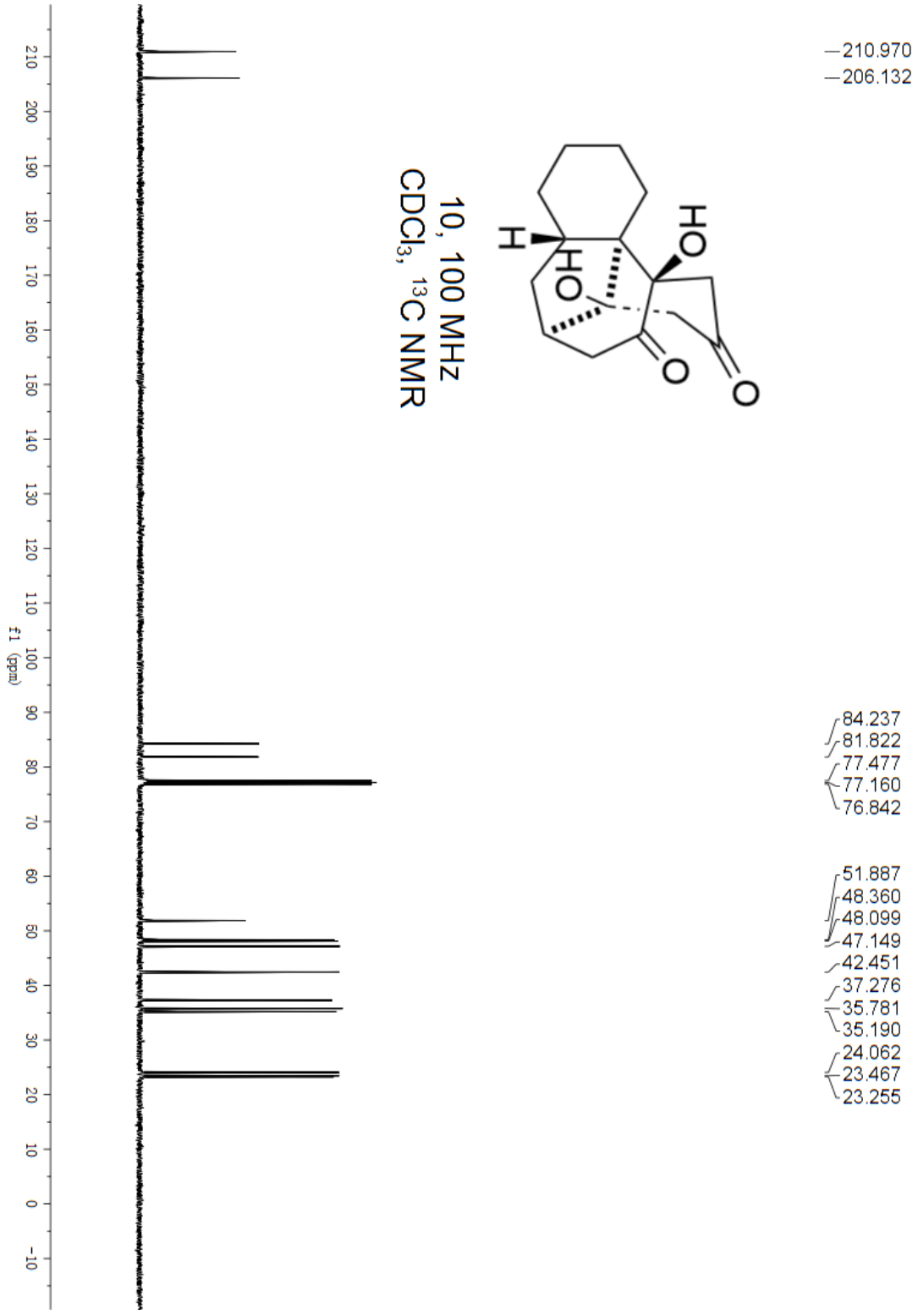

9, 400 MHz
CDCl3, 1H NMR
TMSO
H
O
O
O
f1 (ppm)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
1.00
1.04
1.00
1.03
0.99
4.07
1.09
2.12
1.22
1.32
2.10
1.05
1.06
1.02
9.08
7.260
3.014
2.977
2.941
2.937
2.906
2.901
2.575
2.567
2.514
2.510
2.497
2.478
2.475
2.460
2.138
2.122
2.101
2.098
2.068
2.039
2.029
1.881
1.876
1.864
1.858
1.843
1.819
1.808
1.785
1.652
1.637
1.602
1.600
1.568
1.554
1.535
1.521
1.500
1.465
1.445
1.439
1.432
1.424
1.417
1.406
1.109
1.104
1.078
1.073
1.070
0.974
0.970
0.938
0.763
0.732
0.146
0.142

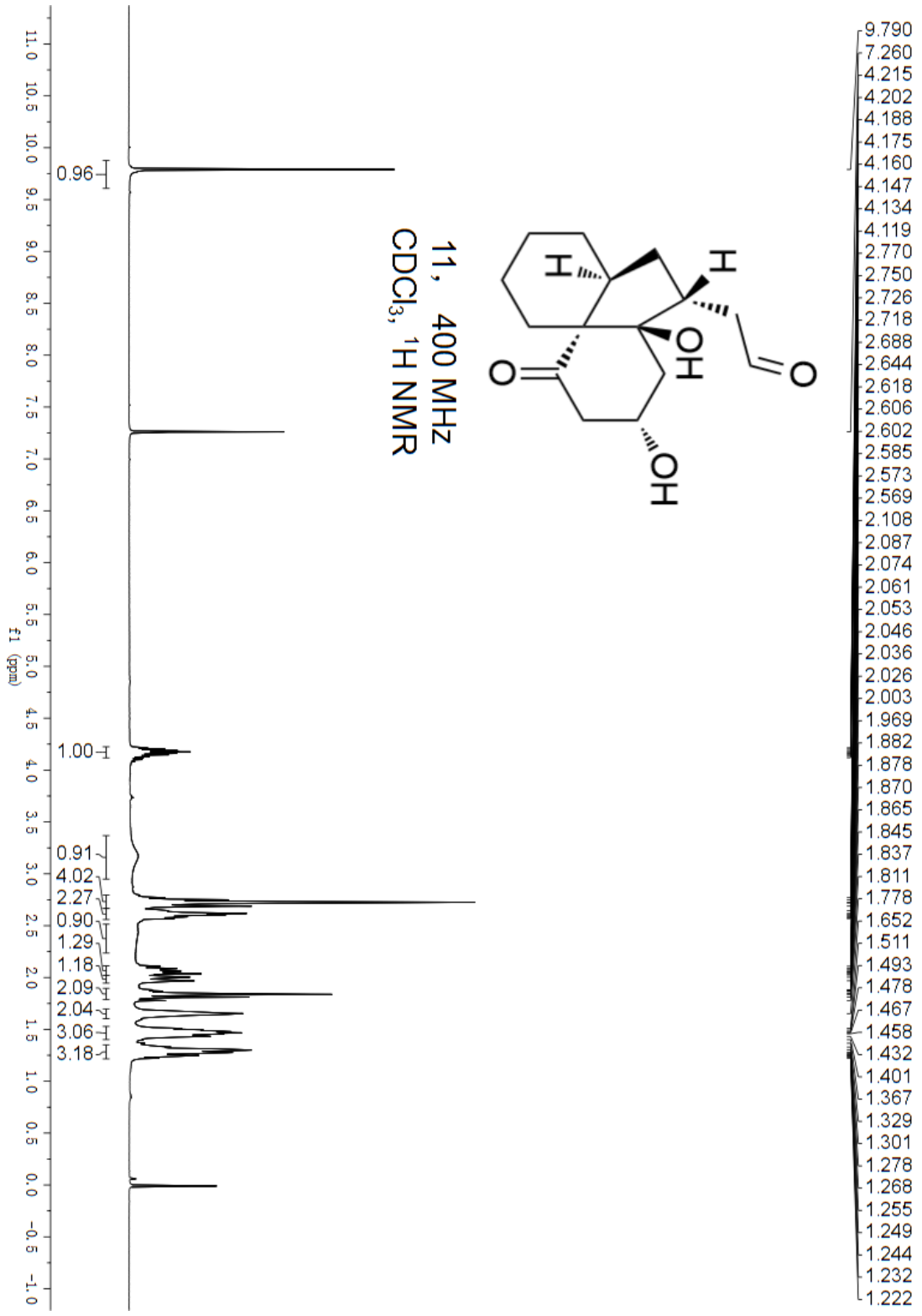

9, 100 MHz
CDCl3, 13C NMR
TMSO
H
O
O
O
214.987
206.523
205.998
87.386
77.478
77.160
76.842
59.958
49.507
43.843
42.400
34.809
32.836
32.492
31.018
23.629
22.952
21.550
3.158
f1 (ppm)

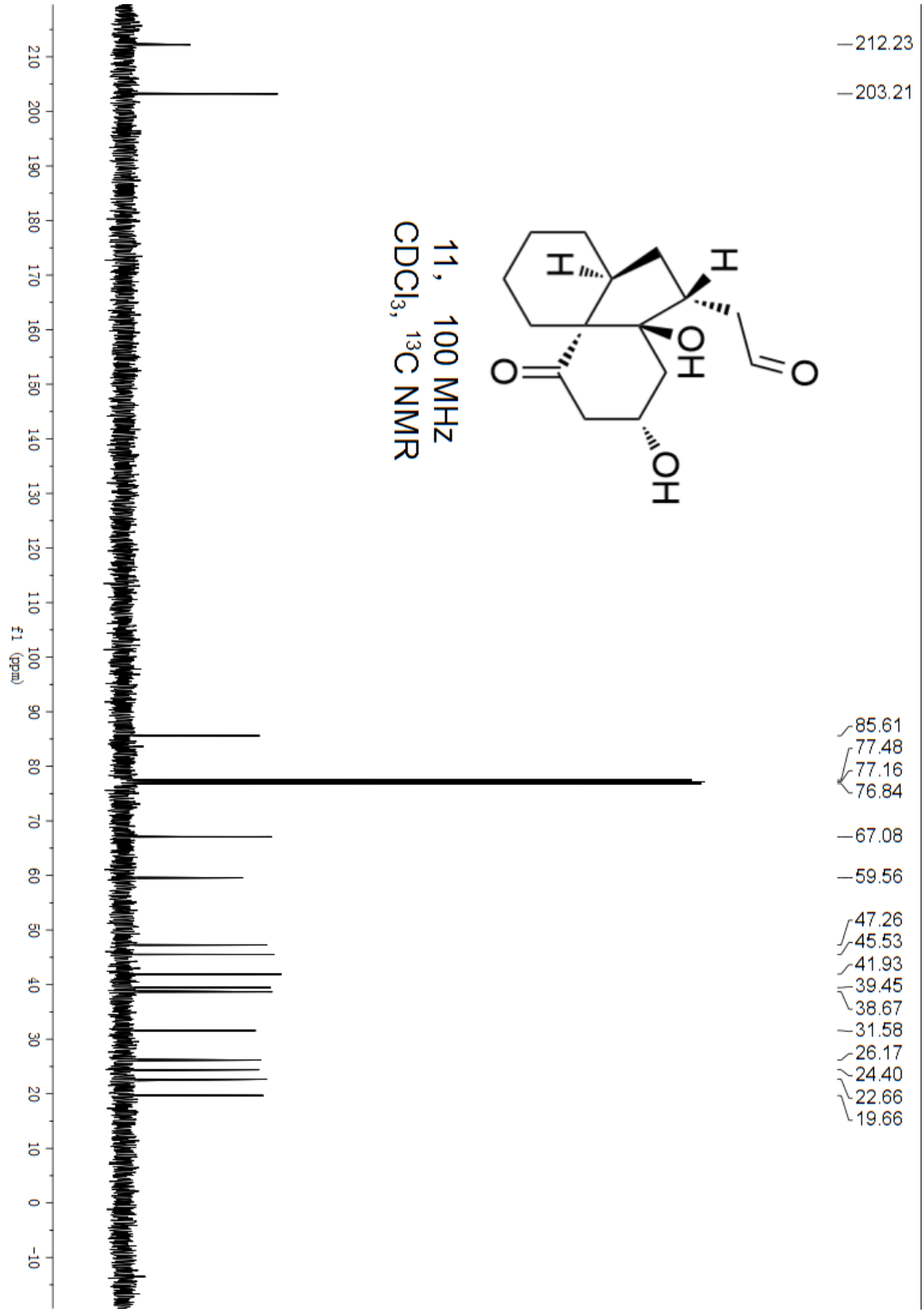

19, 400 MHz
C6D6, 1H NMR
TMSO
HO
H
O
O
f1 (ppm)
1.02
2.05
1.00
5.16
1.09
1.08
4.04
1.13
2.14
1.07
9.13
7.160
2.502
2.491
2.459
2.452
2.449
2.387
2.346
2.330
2.287
2.251
2.246
2.210
2.206
2.037
2.024
2.016
2.009
1.988
1.944
1.887
1.882
1.857
1.845
1.840
1.830
1.813
1.801
1.684
1.672
1.661
1.614
1.606
1.595
1.584
1.576
1.567
1.517
1.509
1.504
1.479
1.460
1.455
1.448
1.407
1.383
1.376
1.353
1.261
1.229
1.199
1.176
1.168
1.161
1.139
0.308
0.300
0.292

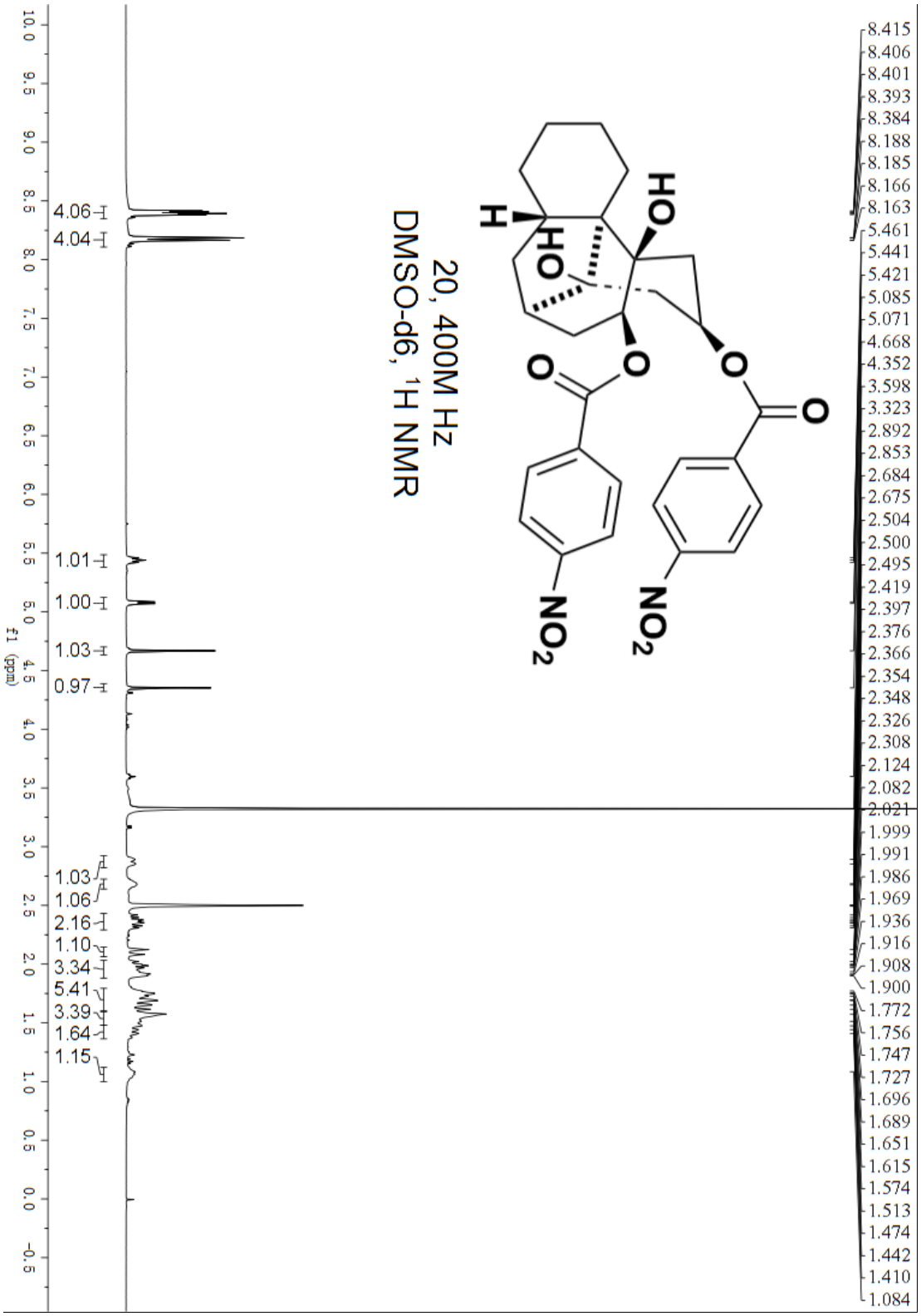

19, 100 MHz
CDCl3, 13C NMR
TMSO
HO
H
O
O
208.779
206.005
86.248
84.127
77.477
77.160
76.842
52.570
48.585
48.263
46.550
43.058
37.491
36.413
35.357
24.280
23.491
23.161
2.786
f1 (ppm)

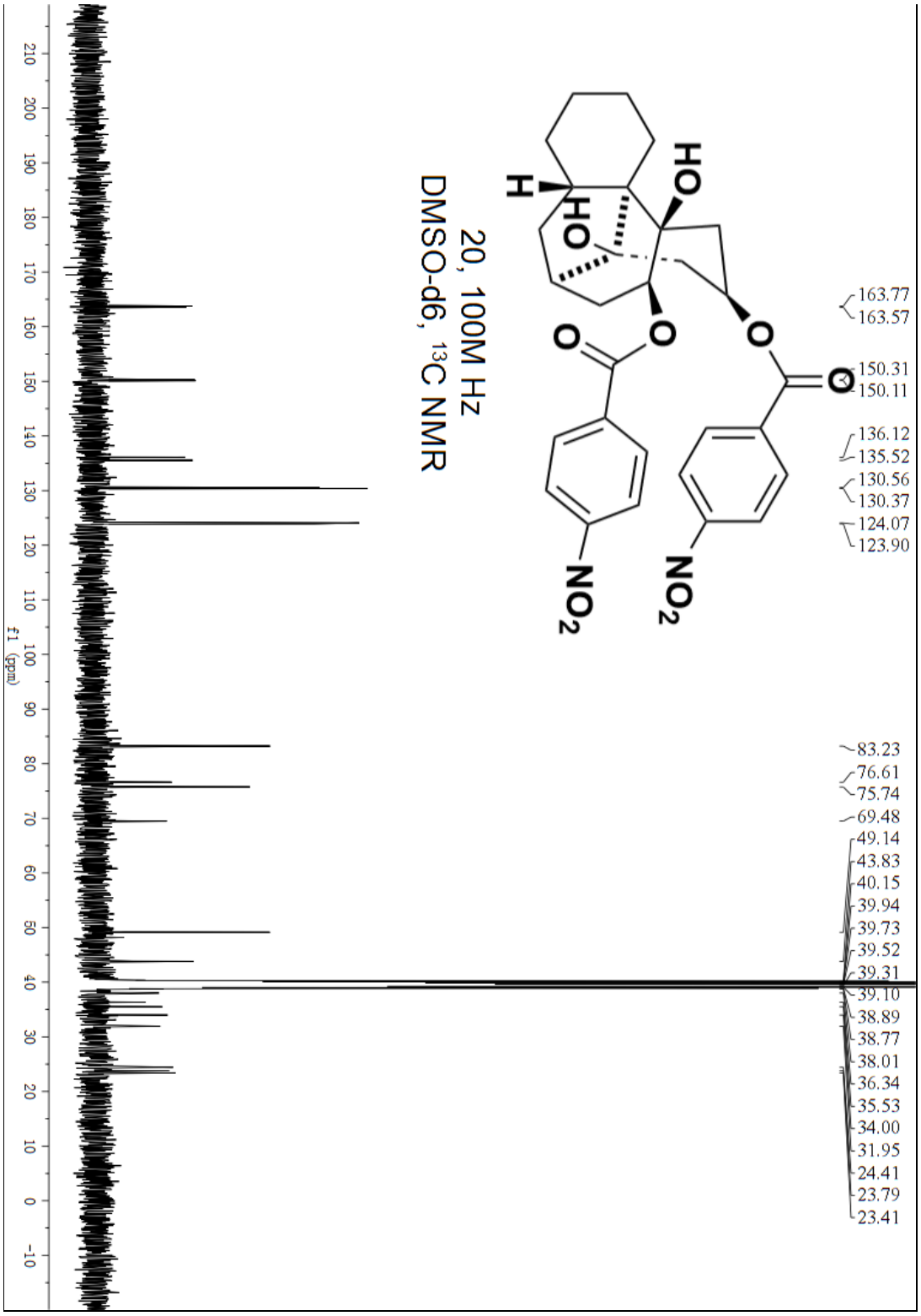

10, 400 MHz
CDCl3, 1H NMR
HO
H
HO
O
O
f1 (ppm)
1.00
3.07
2.00
1.00
1.25
4.23
3.23
2.15
1.11
1.12
1.27
7.260
4.051
2.996
2.992
2.952
2.949
2.921
2.883
2.841
2.380
2.373
2.309
2.300
2.292
2.284
2.275
2.110
2.106
2.068
2.064
2.056
2.035
2.027
2.020
2.005
1.991
1.981
1.971
1.957
1.943
1.922
1.822
1.811
1.794
1.778
1.699
1.677
1.665
1.644
1.632
1.623
1.609
1.601
1.592
1.516
1.507
1.501
1.492
1.485
1.475
1.471
1.302
1.295
1.266
1.260
1.232
1.167
1.135

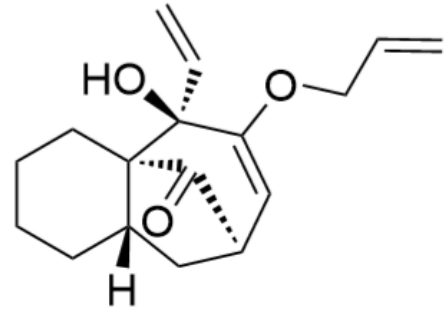

10, 100 MHz
CDCl3, 13C NMR
H
HO
HO
O
O
210.970
206.132
84.237
81.822
77.477
77.160
76.842
51.887
48.360
48.099
47.149
42.451
37.276
35.781
35.190
24.062
23.467
23.255
f1 (ppm)


11, 400 MHz
CDCl3, 1H NMR
H
H
OH
OH
O
O
0.96
1.00
0.91
4.02
2.27
0.90
1.29
1.18
2.09
2.04
3.06
3.18
f1 (ppm)
9.790
7.260
4.215
4.202
4.188
4.175
4.160
4.147
4.134
4.119
2.770
2.750
2.726
2.718
2.688
2.644
2.618
2.606
2.602
2.585
2.573
2.569
2.108
2.087
2.074
2.061
2.053
2.046
2.036
2.026
2.003
1.969
1.882
1.878
1.870
1.865
1.845
1.837
1.811
1.778
1.652
1.511
1.493
1.478
1.467
1.458
1.432
1.401
1.367
1.329
1.301
1.278
1.268
1.255
1.249
1.244
1.232
1.222

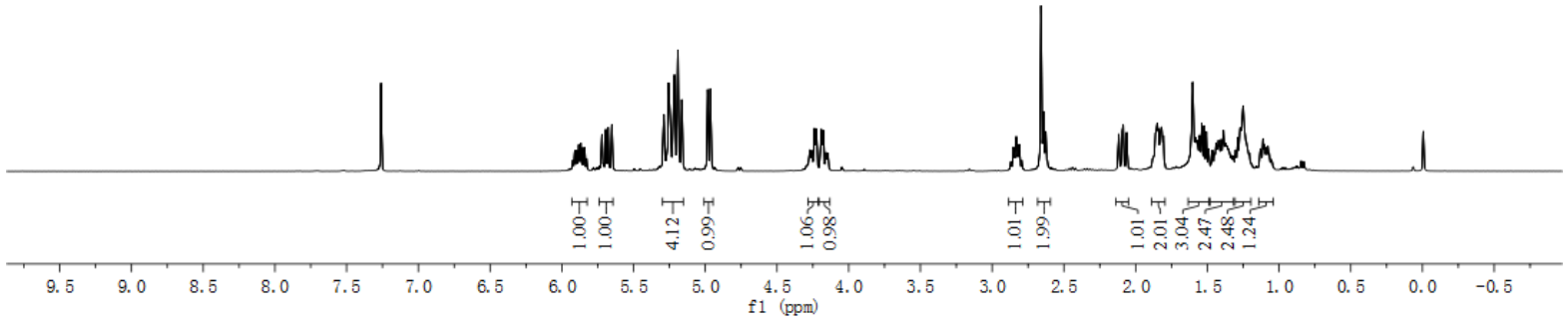

11, 100 MHz
CDCl3, 13C NMR
212.23
203.21
85.61
77.48
77.16
76.84
67.08
59.56
47.26
45.53
41.93
39.45
38.67
31.58
26.17
24.40
22.66
19.66
f1 (ppm)

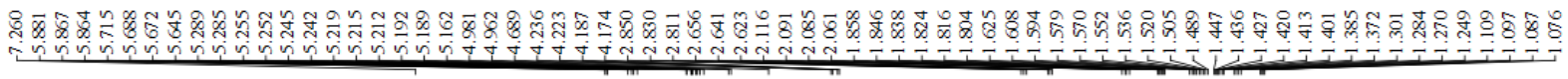

20, 400M Hz
DMSO-d6, 1H NMR
HO
HO
H
O
O
O
O
NO2
NO2
f1 (ppm)
4.06
4.04
1.01
1.00
1.03
0.97
1.03
1.06
2.16
1.10
3.34
5.41
3.39
1.64
1.15
8.415
8.406
8.401
8.393
8.384
8.188
8.185
8.166
8.163
5.461
5.441
5.421
5.085
5.071
4.668
4.352
3.598
3.323
2.892
2.853
2.684
2.675
2.504
2.500
2.495
2.419
2.397
2.376
2.366
2.354
2.348
2.326
2.308
2.124
2.082
2.021
1.999
1.991
1.986
1.969
1.936
1.916
1.908
1.900
1.772
1.756
1.747
1.727
1.696
1.689
1.651
1.615
1.574
1.513
1.474
1.442
1.410
1.084

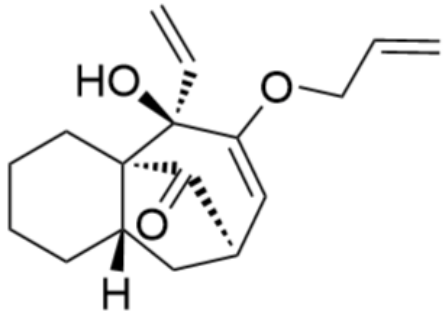

20, 100M Hz
DMSO-d6, 13C NMR
HO
H
HO
O
O
O
O
NO2
NO2
163.77
163.57
150.31
150.11
136.12
135.52
130.56
130.37
124.07
123.90
83.23
76.61
75.74
69.48
49.14
43.83
40.15
39.94
39.73
39.52
39.31
39.10
38.89
38.77
38.01
36.34
35.53
34.00
31.95
24.41
23.79
23.41
f1 (ppm)



21, 400 MHz
$CDCl_3$, ^1H NMR

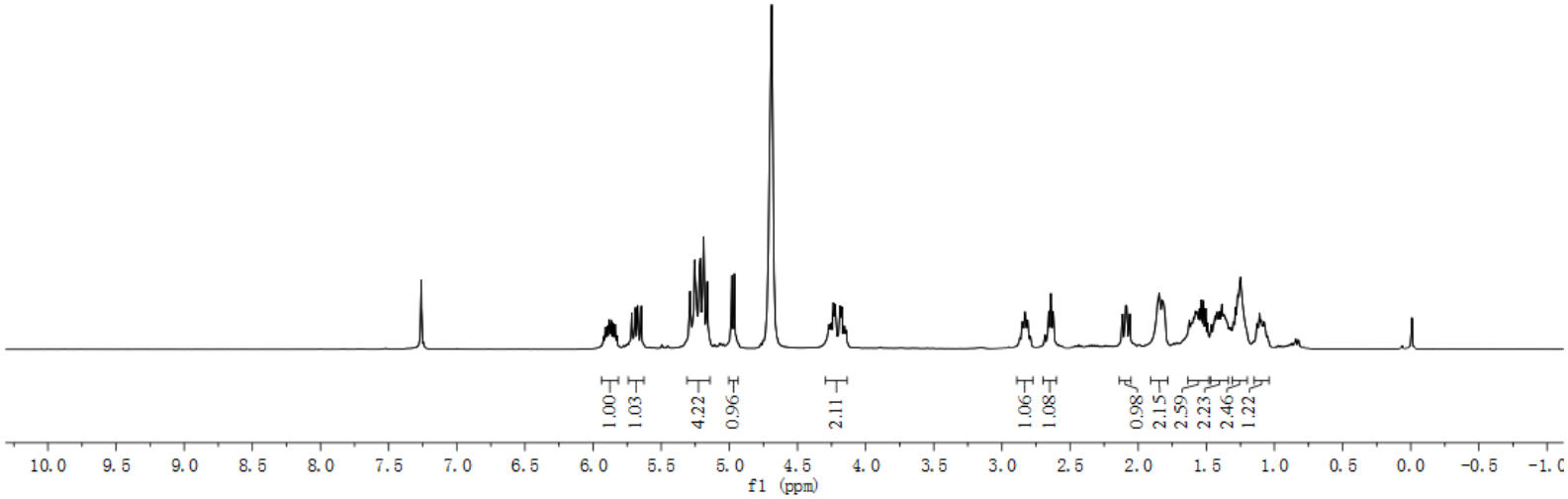


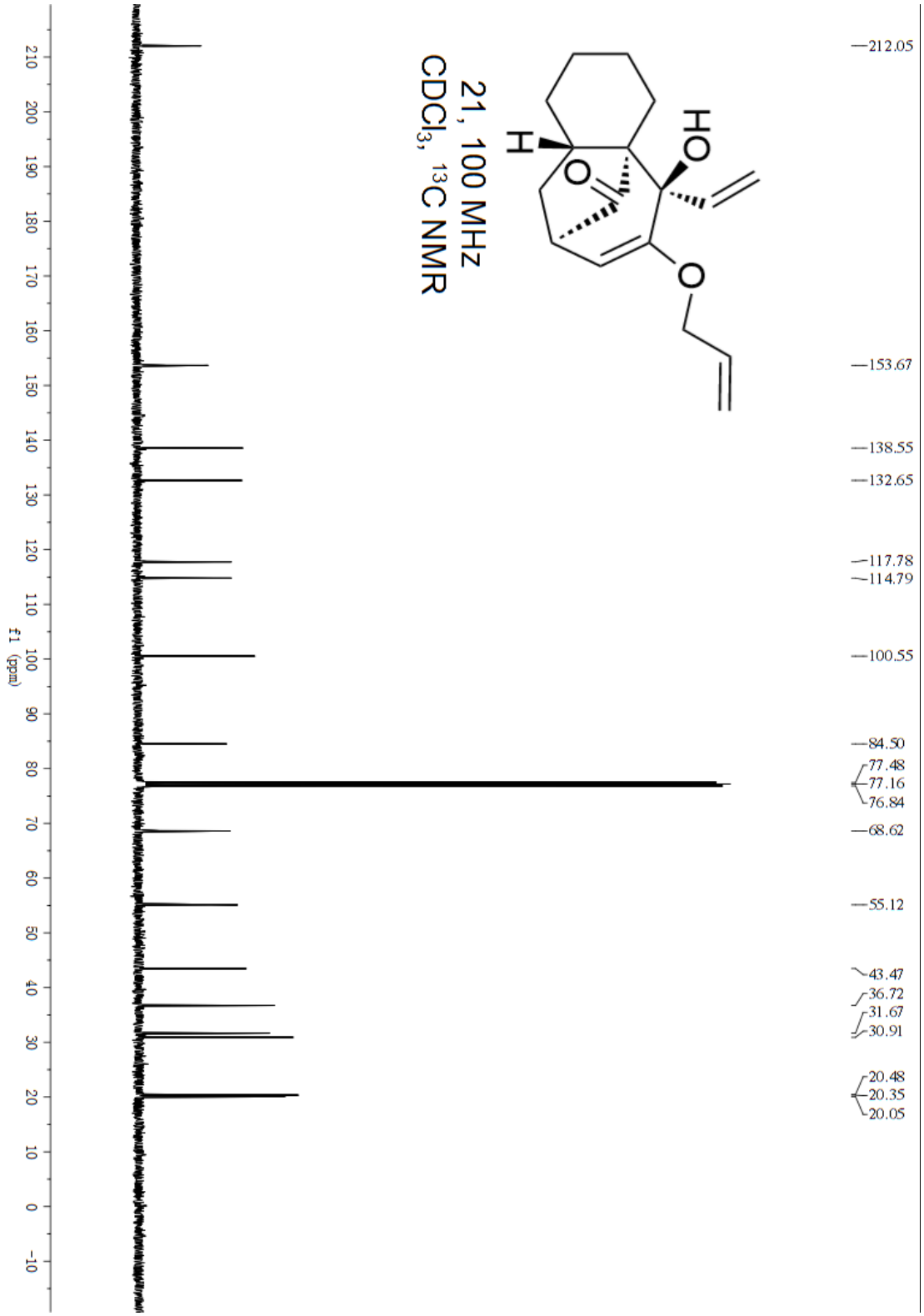


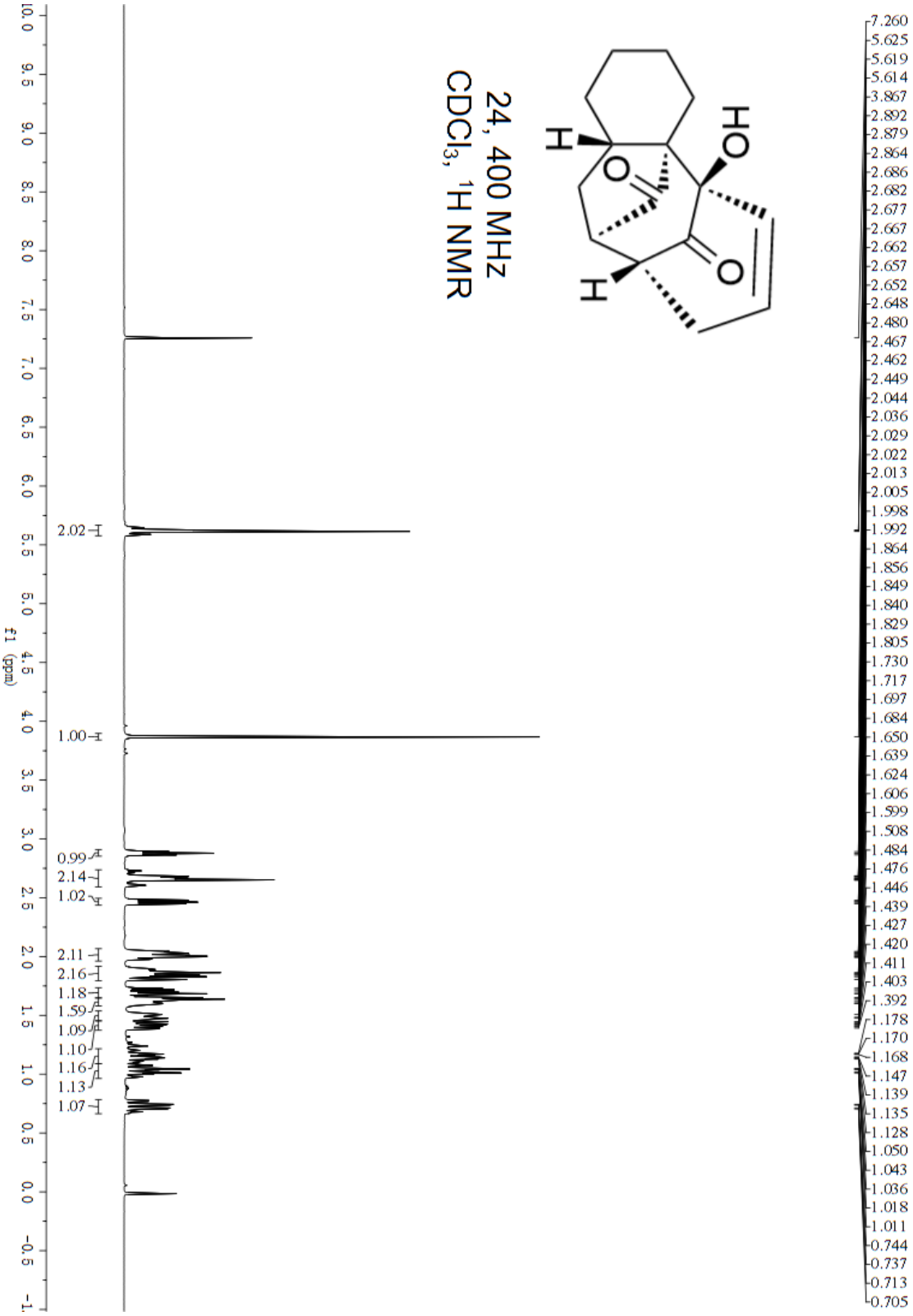


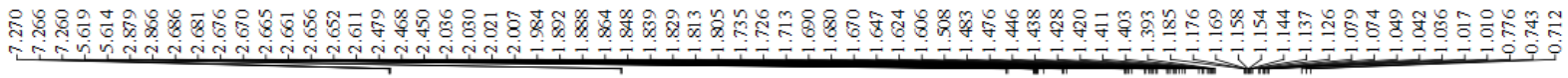
21, 400 MHz

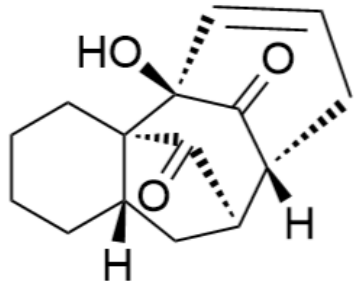
$CDCl_3+D_2O$, ^1H NMR




21, 100 MHz
CDCl3, 13C NMR
H
O
HO
O
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
212.05
153.67
138.55
132.65
117.78
114.79
100.55
84.50
77.48
77.16
76.84
68.62
55.12
43.47
36.72
31.67
30.91
20.48
20.35
20.05

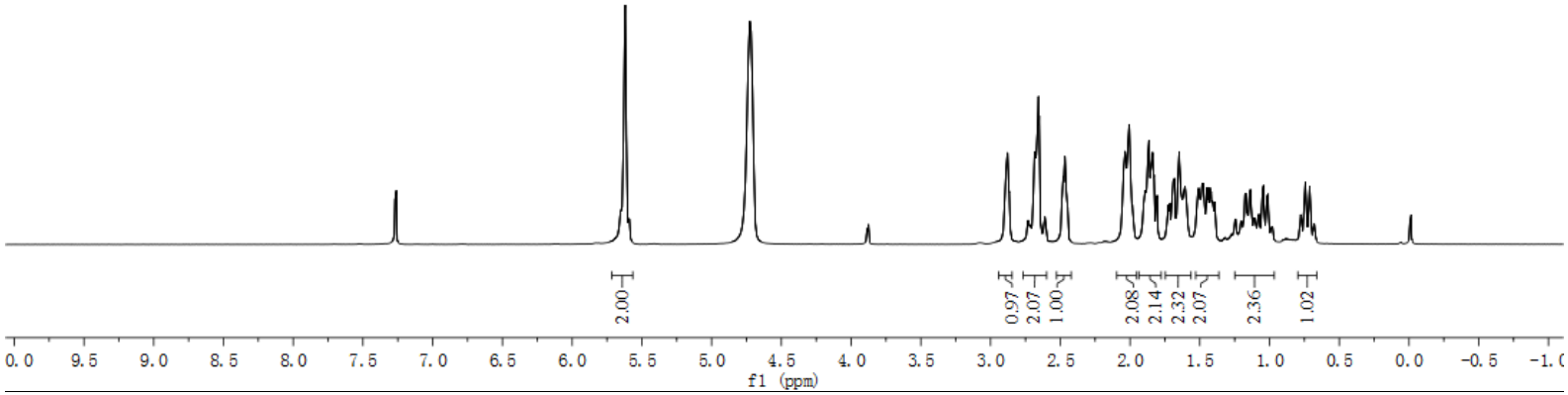

24, 400 MHz
CDCl3, 1H NMR
HO
O
O
H
H
f1 (ppm)
2.02
1.00
0.99
2.14
1.02
2.11
2.16
1.18
1.59
1.09
1.10
1.16
1.13
1.07
7.260
5.625
5.619
5.614
3.867
2.892
2.879
2.864
2.686
2.682
2.677
2.667
2.662
2.657
2.652
2.648
2.480
2.467
2.462
2.449
2.044
2.036
2.029
2.022
2.013
2.005
1.998
1.992
1.864
1.856
1.849
1.840
1.829
1.805
1.730
1.717
1.697
1.684
1.650
1.639
1.624
1.606
1.599
1.508
1.484
1.476
1.446
1.439
1.427
1.420
1.411
1.403
1.392
1.178
1.170
1.168
1.147
1.139
1.135
1.128
1.050
1.043
1.036
1.018
1.011
0.744
0.737
0.713
0.705

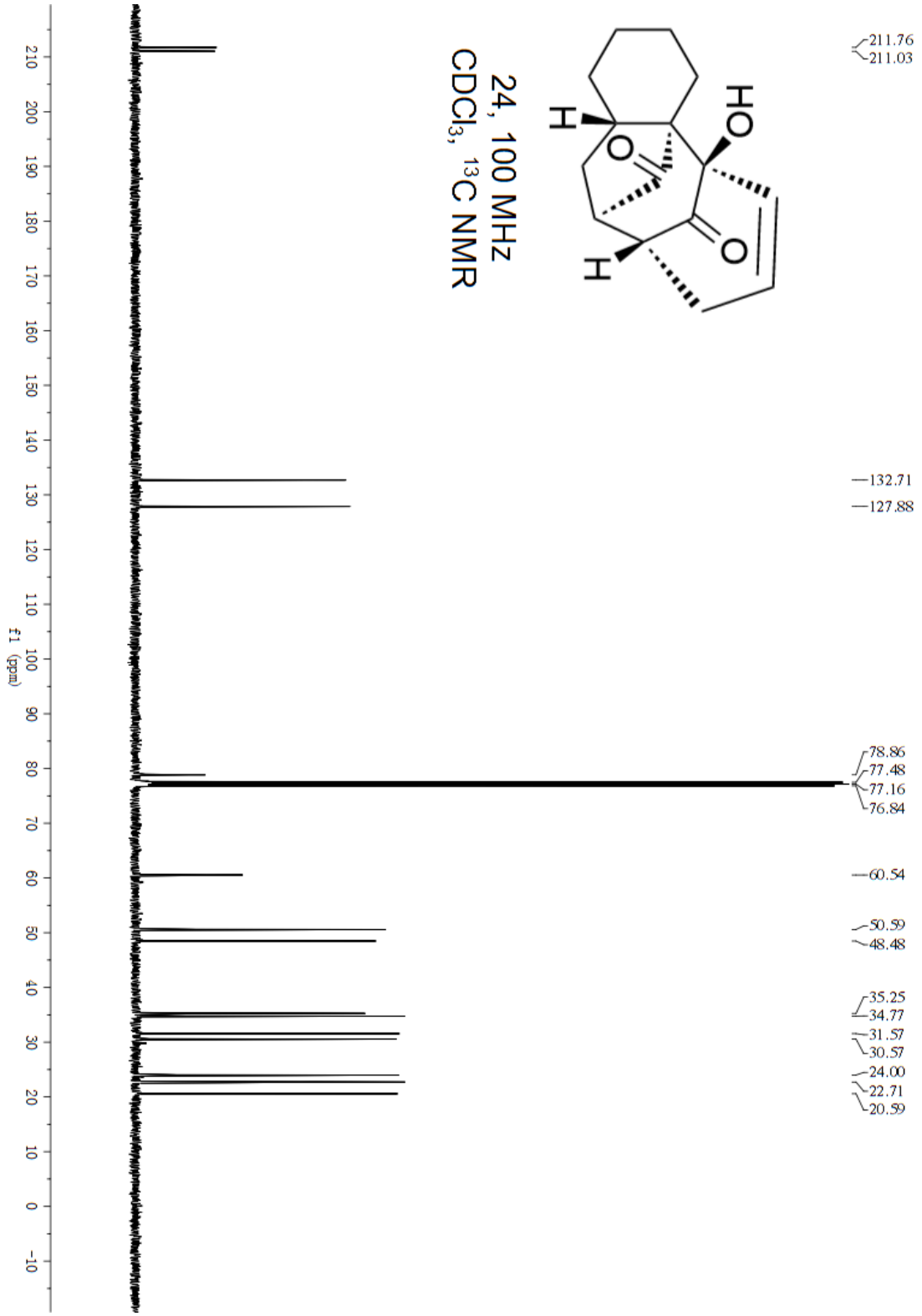


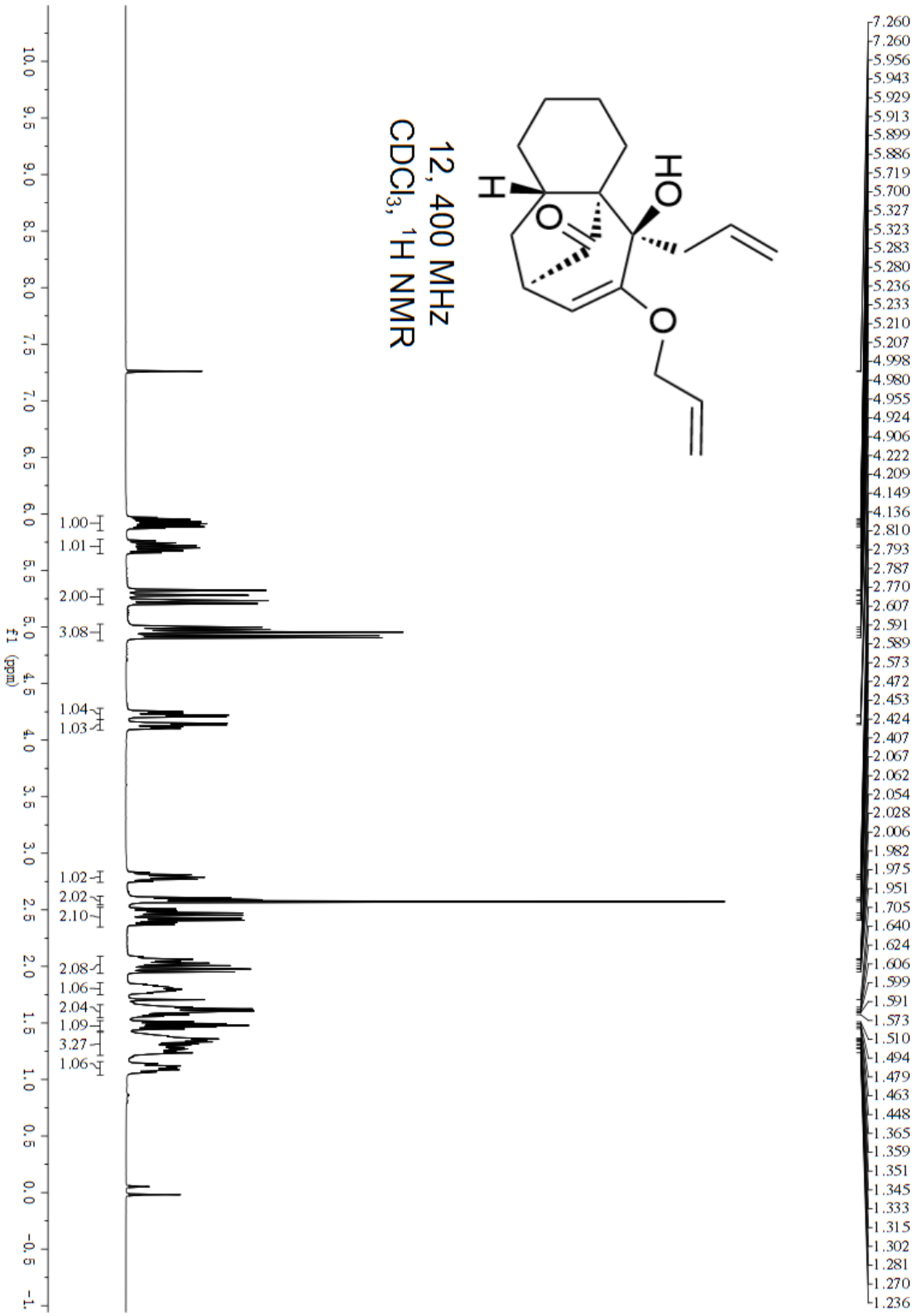


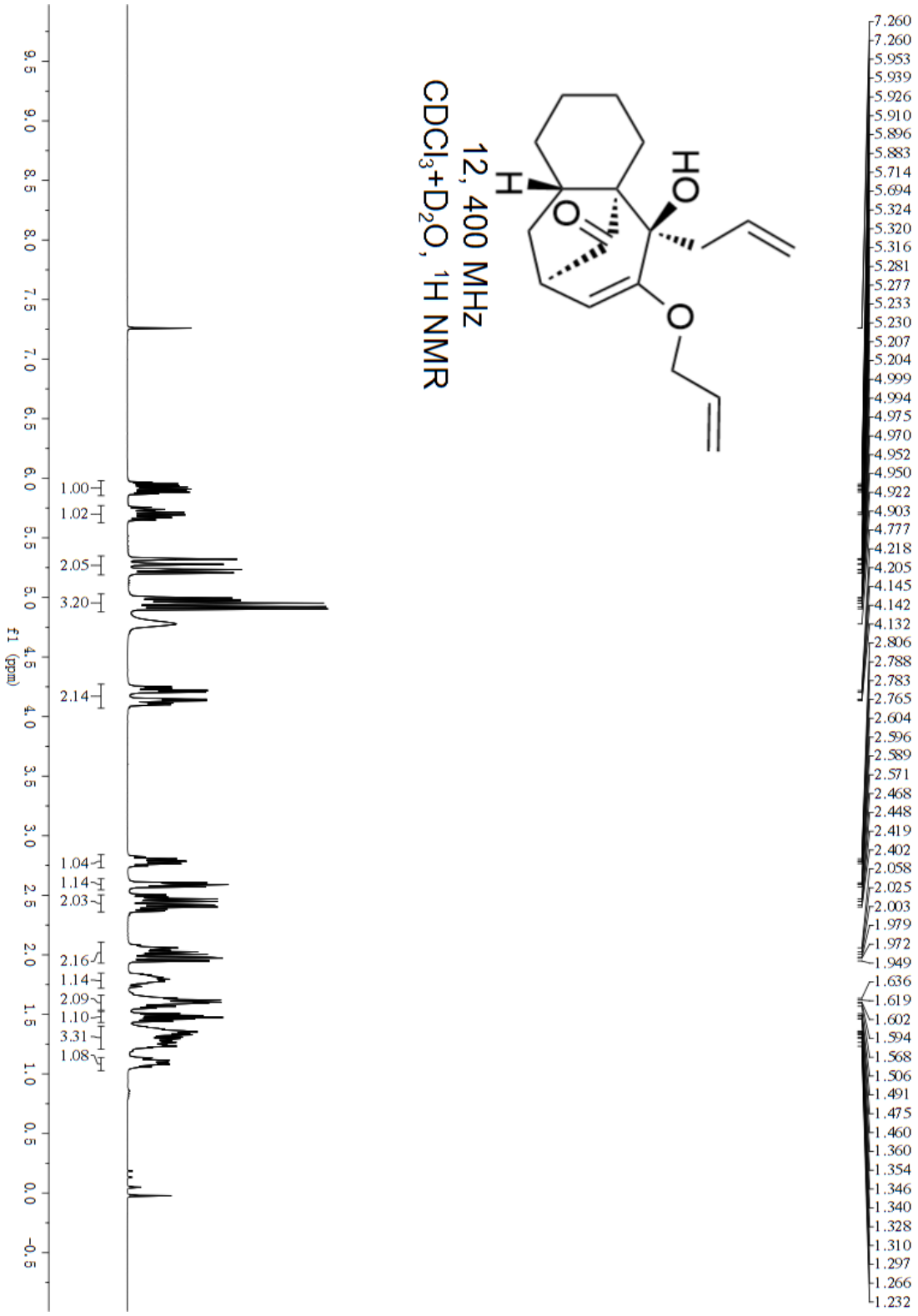
24, 400 MHz

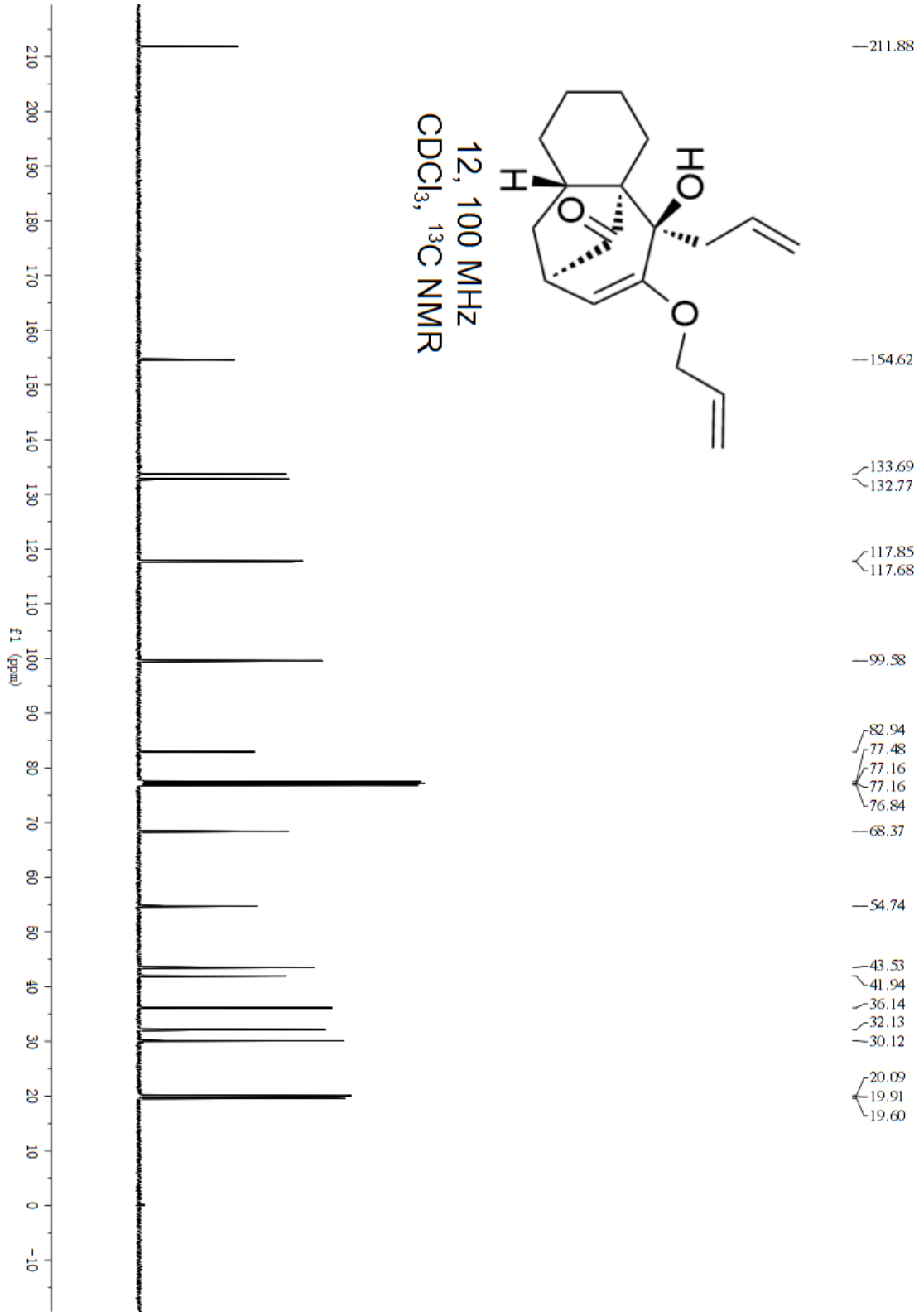
$CDCl_3+D_2O$, ^{1}HNMR

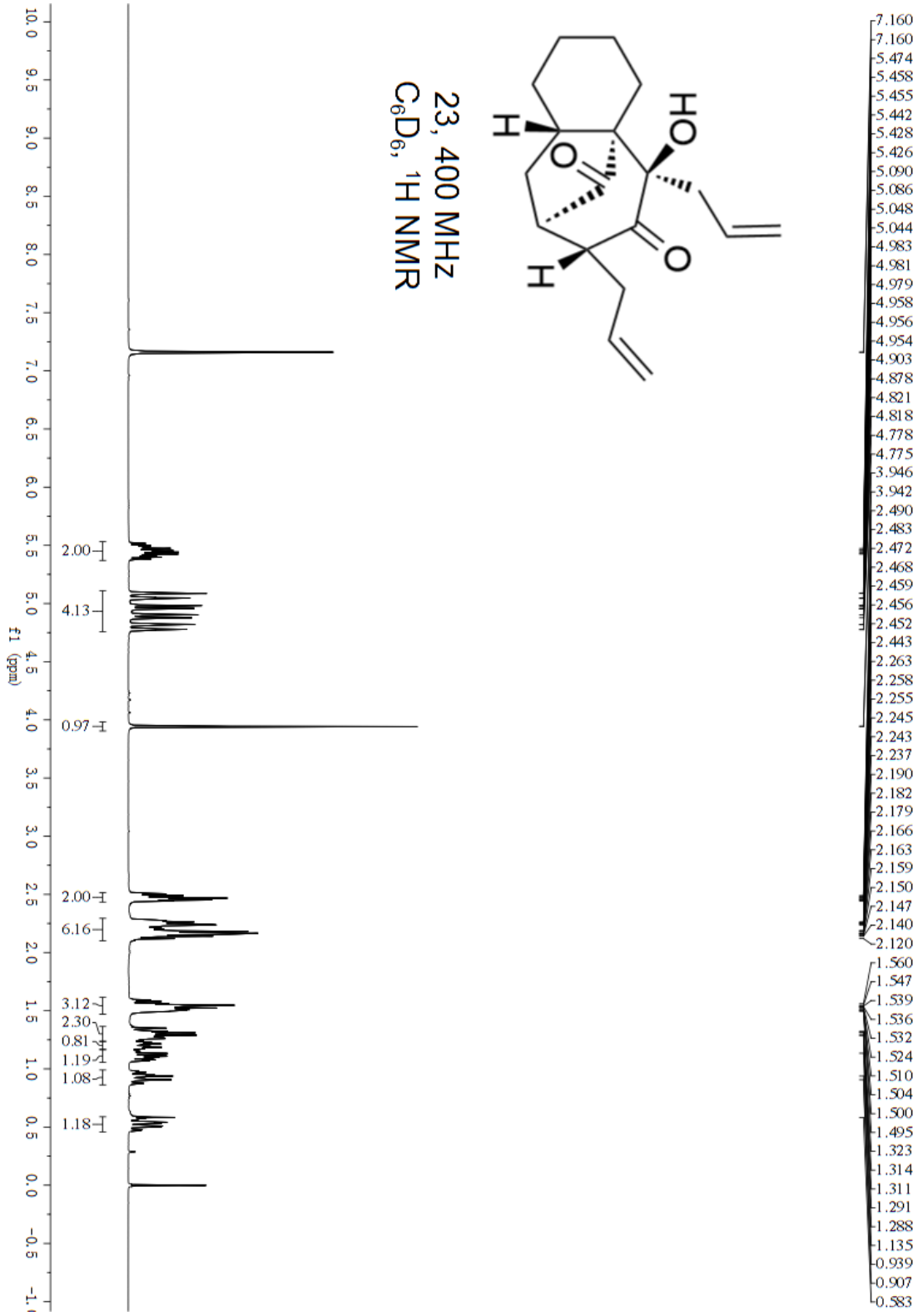


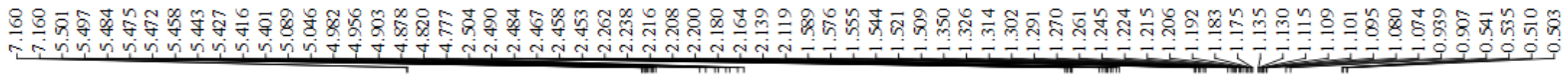

24, 100 MHz
CDCl3, 13C NMR
HO
H
O
O
H
211.76
211.03
132.71
127.88
78.86
77.48
77.16
76.84
60.54
50.59
48.48
35.25
34.77
31.57
30.57
24.00
22.71
20.59
f1 (ppm)

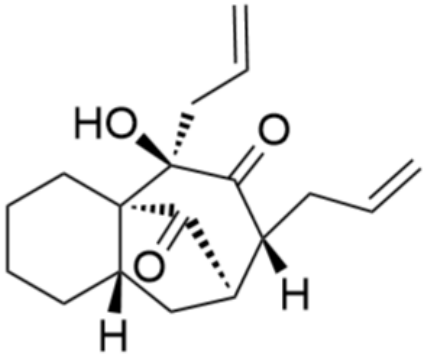

12, 400 MHz
CDCl3, 1H NMR
HO
H
O
O
f1 (ppm)
1.00
1.01
2.00
3.08
1.04
1.03
1.02
2.02
2.10
2.08
1.06
2.04
1.09
3.27
1.06
7.260
7.260
5.956
5.943
5.929
5.913
5.899
5.886
5.719
5.700
5.327
5.323
5.283
5.280
5.236
5.233
5.210
5.207
4.998
4.980
4.955
4.924
4.906
4.222
4.209
4.149
4.136
2.810
2.793
2.787
2.770
2.607
2.591
2.589
2.573
2.472
2.453
2.424
2.407
2.067
2.062
2.054
2.028
2.006
1.982
1.975
1.951
1.705
1.640
1.624
1.606
1.599
1.591
1.573
1.510
1.494
1.479
1.463
1.448
1.365
1.359
1.351
1.345
1.333
1.315
1.302
1.281
1.270
1.236


12, 400 MHz
CDCl3+D2O, 1H NMR
HO
H
O
O
f1 (ppm)
1.00
1.02
2.05
3.20
2.14
1.04
1.14
2.03
2.16
1.14
2.09
1.10
3.31
1.08
7.260
7.260
5.953
5.939
5.926
5.910
5.896
5.883
5.714
5.694
5.324
5.320
5.316
5.281
5.277
5.233
5.230
5.207
5.204
4.999
4.994
4.975
4.970
4.952
4.950
4.922
4.903
4.777
4.218
4.205
4.145
4.142
4.132
2.806
2.788
2.783
2.765
2.604
2.596
2.589
2.571
2.468
2.448
2.419
2.402
2.058
2.025
2.003
1.979
1.972
1.949
1.636
1.619
1.602
1.594
1.568
1.506
1.491
1.475
1.460
1.360
1.354
1.346
1.340
1.328
1.310
1.297
1.266
1.232


12, 100 MHz
CDCl3, 13C NMR
H
O
HO
O
211.88
154.62
133.69
132.77
117.85
117.68
99.58
82.94
77.48
77.16
77.16
76.84
68.37
54.74
43.53
41.94
36.14
32.13
30.12
20.09
19.91
19.60
f1 (ppm)

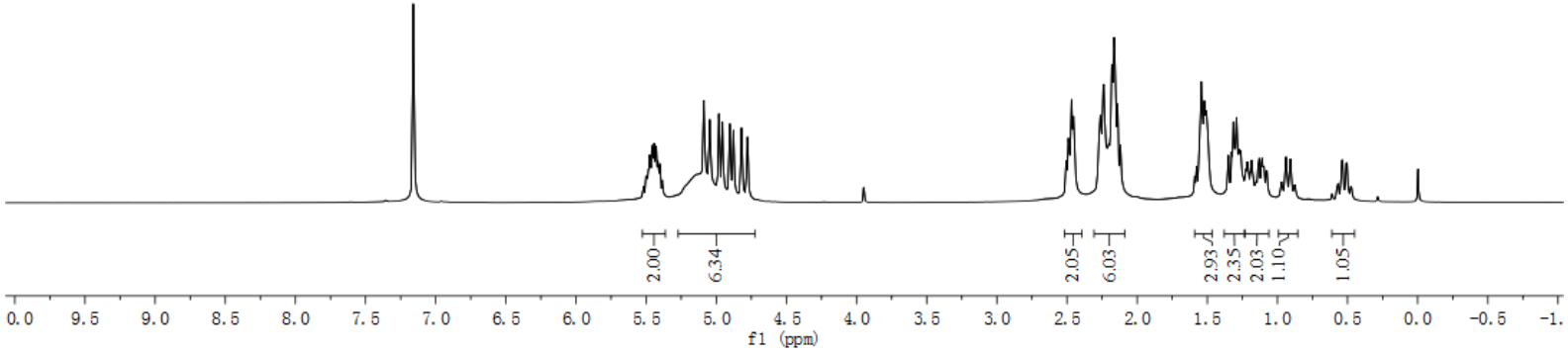

23, 400 MHz
C6D6, 1H NMR
HO
O
O
H
H
f1 (ppm)
2.00
4.13
0.97
2.00
6.16
3.12
2.30
0.81
1.19
1.08
1.18
7.160
7.160
5.474
5.458
5.455
5.442
5.428
5.426
5.090
5.086
5.048
5.044
4.983
4.981
4.979
4.958
4.956
4.954
4.903
4.878
4.821
4.818
4.778
4.775
3.946
3.942
2.490
2.483
2.472
2.468
2.459
2.456
2.452
2.443
2.263
2.258
2.255
2.245
2.243
2.237
2.190
2.182
2.179
2.166
2.163
2.159
2.150
2.147
2.140
2.120
1.560
1.547
1.539
1.536
1.532
1.524
1.510
1.504
1.500
1.495
1.323
1.314
1.311
1.291
1.288
1.135
0.939
0.907
0.583

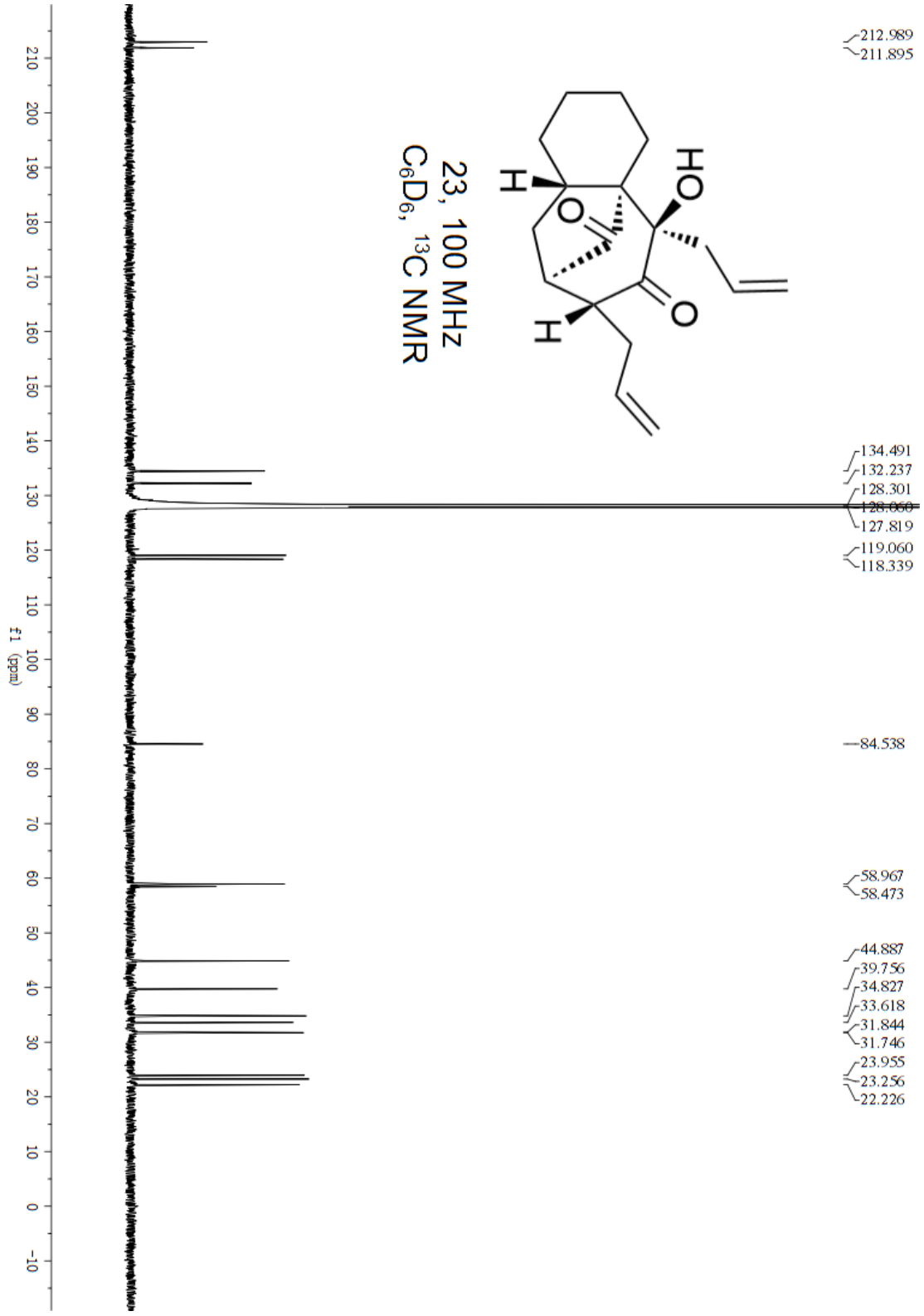


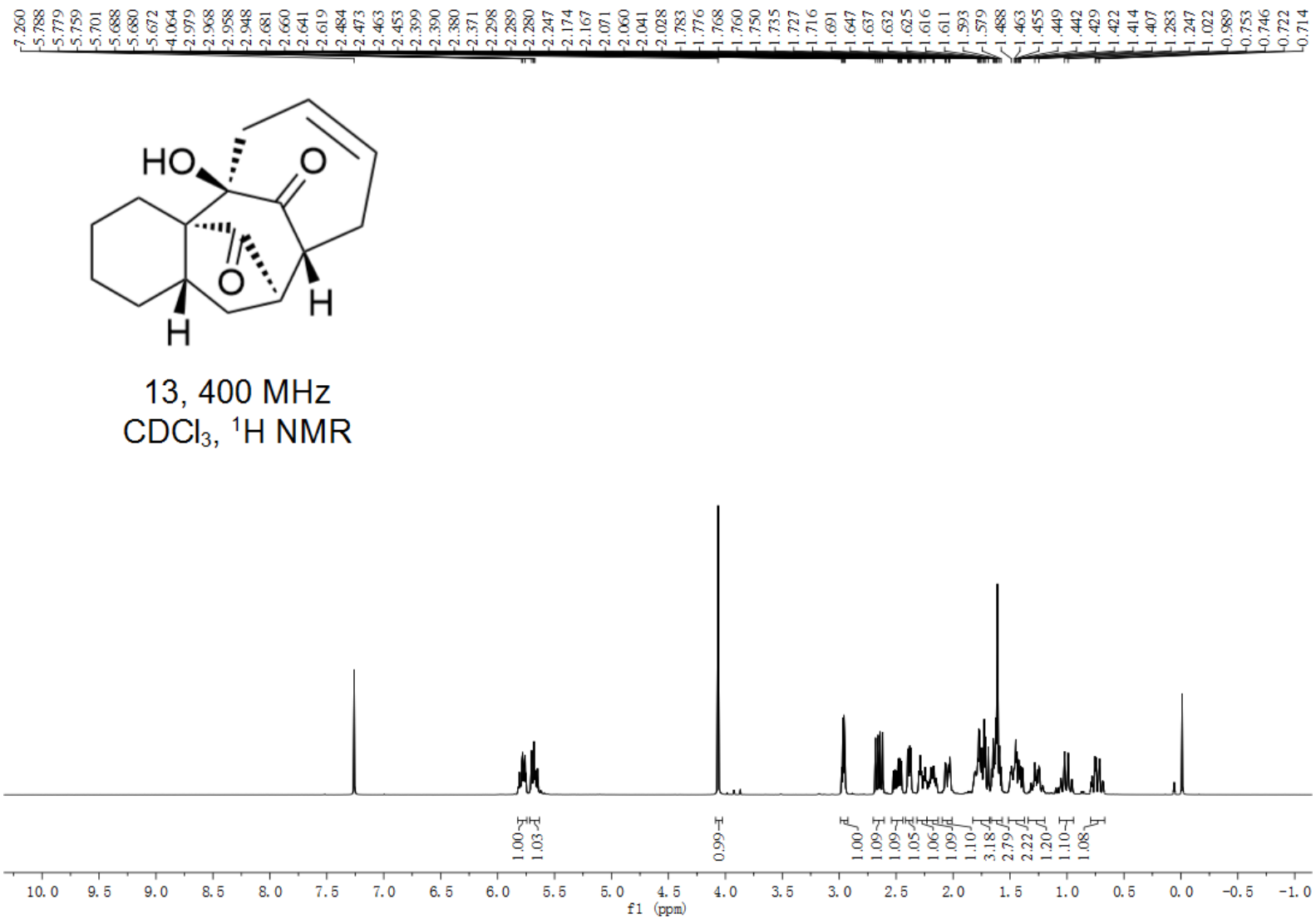


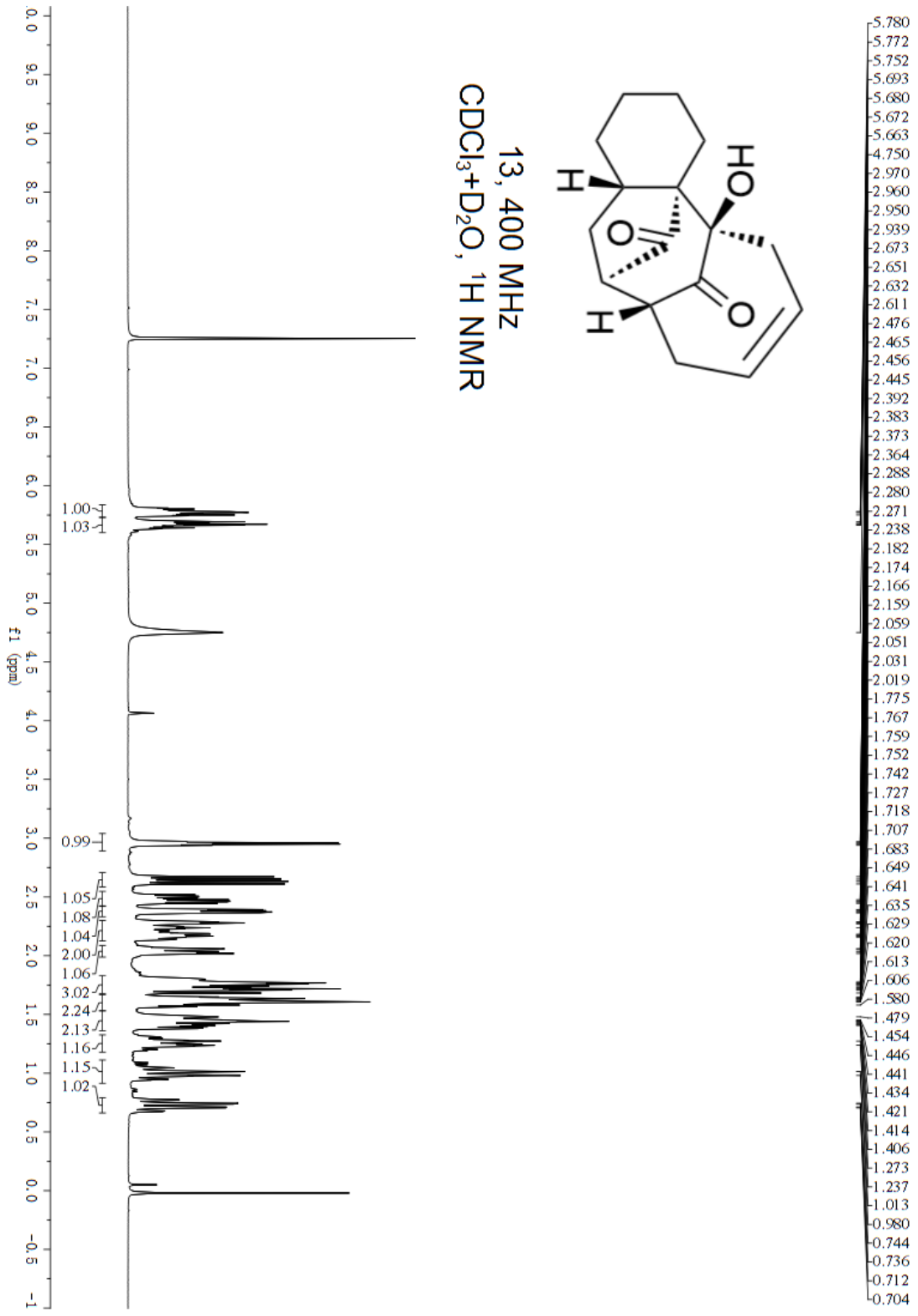
23, 400 MHz

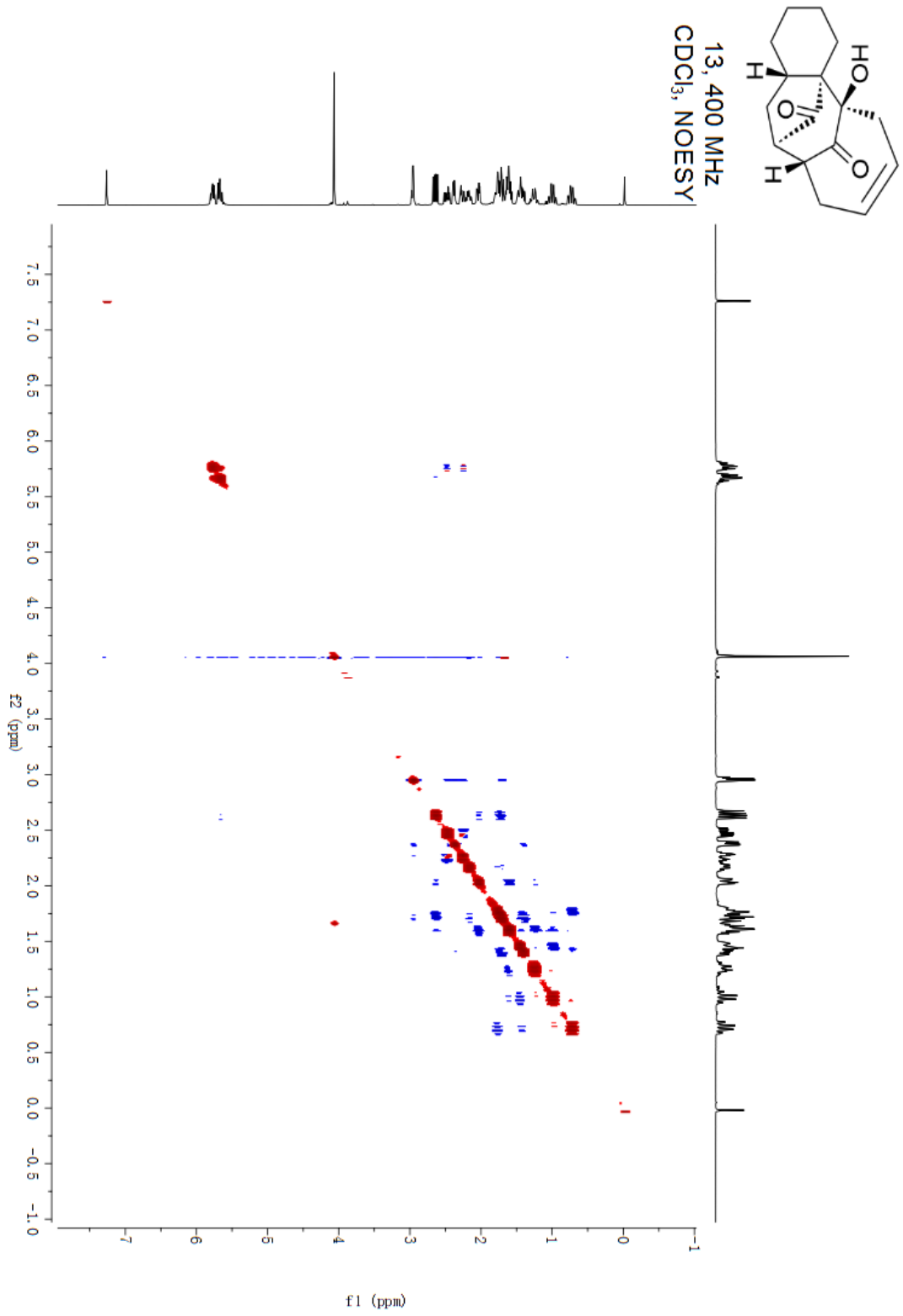
$C_6D_6+D_2O$, ^1H NMR

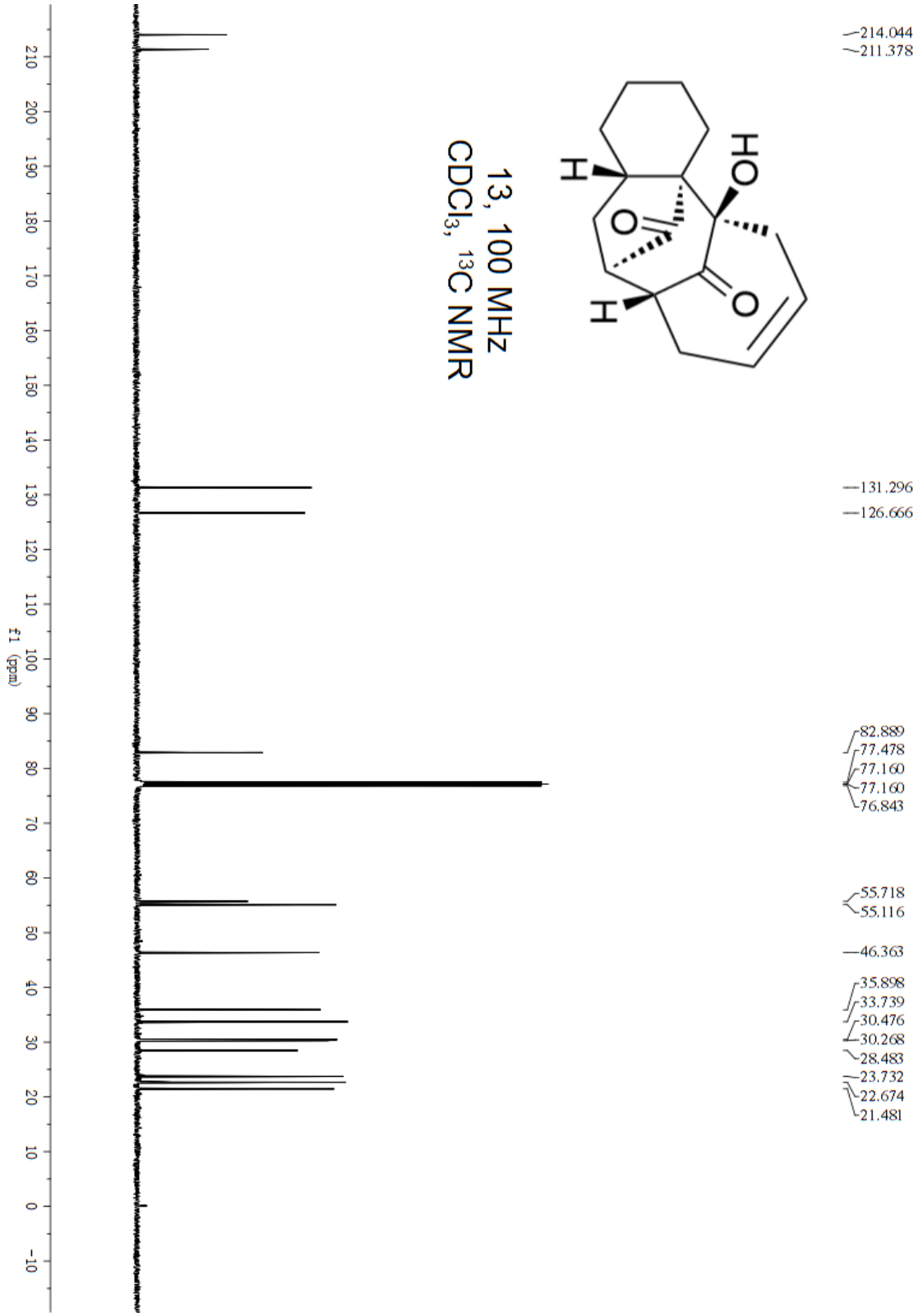


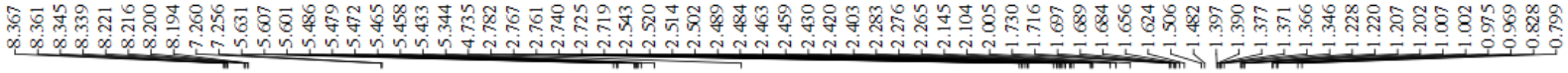

23, 100 MHz
C6D6, 13C NMR
HO
H
O
O
H
212.989
211.895
134.491
132.237
128.301
128.060
127.819
119.060
118.339
84.538
58.967
58.473
44.887
39.756
34.827
33.618
31.844
31.746
23.955
23.256
22.226
f1 (ppm)

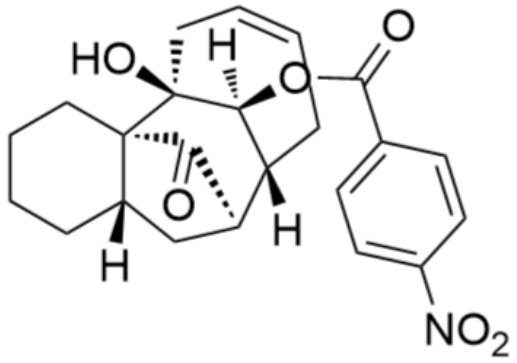

7.260
5.788
5.779
5.759
5.701
5.688
5.680
5.672
4.064
2.979
2.968
2.958
2.948
2.681
2.660
2.641
2.619
2.484
2.473
2.463
2.453
2.399
2.390
2.380
2.371
2.298
2.289
2.280
2.247
2.174
2.167
2.071
2.060
2.041
2.028
1.783
1.776
1.768
1.760
1.750
1.735
1.727
1.716
1.691
1.647
1.637
1.632
1.625
1.616
1.611
1.593
1.579
1.488
1.463
1.455
1.449
1.442
1.429
1.422
1.414
1.407
1.283
1.247
1.022
0.989
0.753
0.746
0.722
0.714
HO
O
O
H
H
13, 400 MHz
CDCl3, 1H NMR
1.00
1.03
0.99
1.00
1.09
1.09
1.05
1.06
1.09
1.10
3.18
2.79
2.22
1.20
1.10
1.08
10.0
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
-0.5
-1.0
f1 (ppm)


13, 400 MHz
CDCl3+D2O, 1H NMR
HO
H
O
O
H
f1 (ppm)
1.00
1.03
0.99
1.05
1.08
1.04
2.00
1.06
3.02
2.24
2.13
1.16
1.15
1.02
5.780
5.772
5.752
5.693
5.680
5.672
5.663
4.750
2.970
2.960
2.950
2.939
2.673
2.651
2.632
2.611
2.476
2.465
2.456
2.445
2.392
2.383
2.373
2.364
2.288
2.280
2.271
2.238
2.182
2.174
2.166
2.159
2.059
2.051
2.031
2.019
1.775
1.767
1.759
1.752
1.742
1.727
1.718
1.707
1.683
1.649
1.641
1.635
1.629
1.620
1.613
1.606
1.580
1.479
1.454
1.446
1.441
1.434
1.421
1.414
1.406
1.273
1.237
1.013
0.980
0.744
0.736
0.712
0.704


13, 400 MHz
CDCl3, NOESY
HO
H
O
O
H
f2 (ppm)
f1 (ppm)

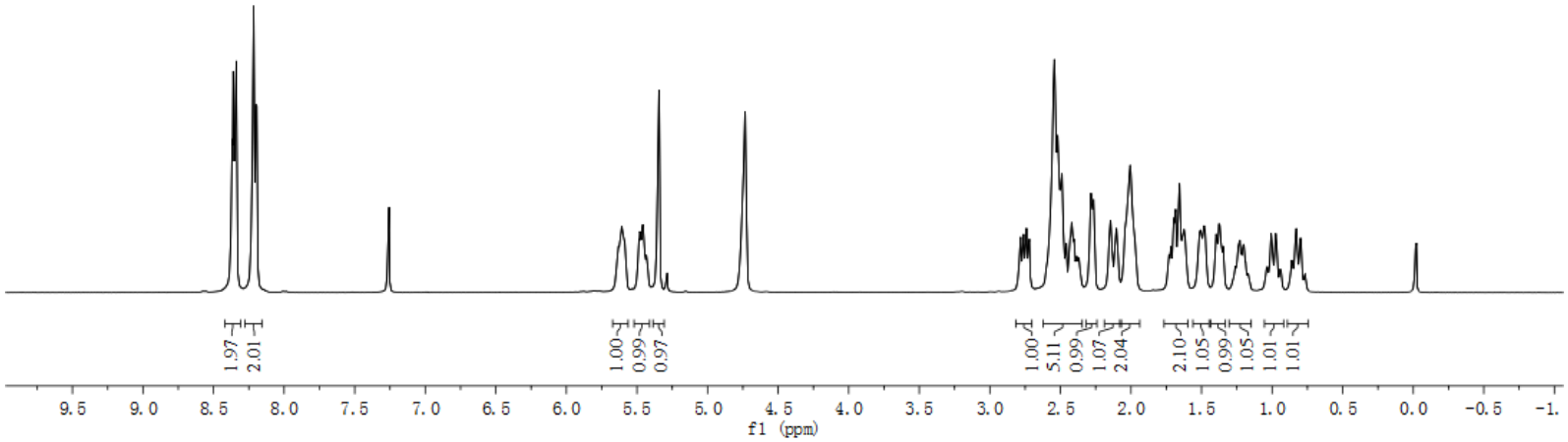

13, 100 MHz
CDCl3, 13C NMR
HO
H
O
O
H
214.044
211.378
131.296
126.666
82.889
77.478
77.160
77.160
76.843
55.718
55.116
46.363
35.898
33.739
30.476
30.268
28.483
23.732
22.674
21.481
f1 (ppm)

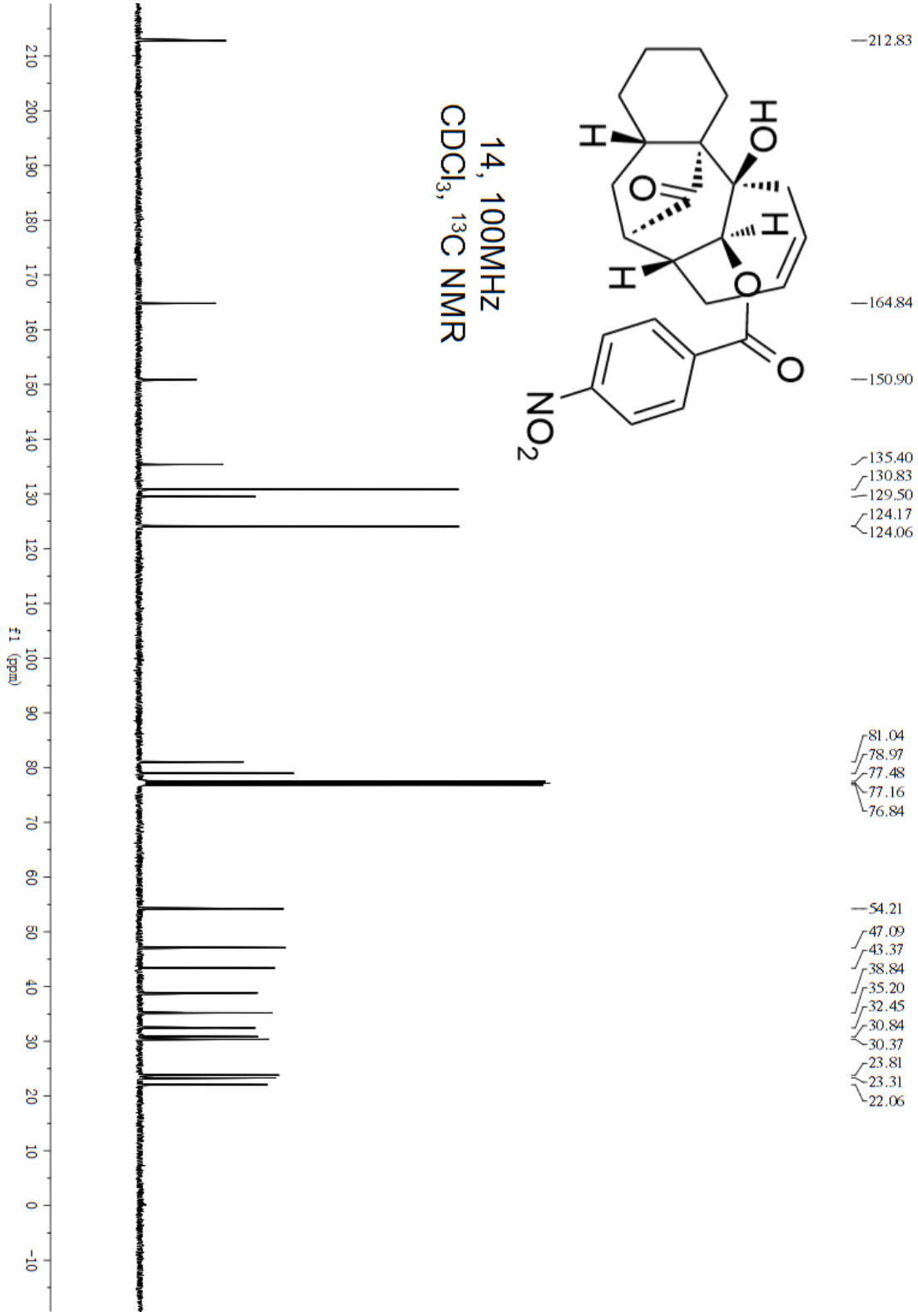




14, 400 MHz

$CDCl_3+D_2O$, ^1H NMR




14, 100MHz
CDCl3, 13C NMR
HO
H
O
H
H
O
O
NO2
212.83
164.84
150.90
135.40
130.83
129.50
124.17
124.06
81.04
78.97
77.48
77.16
76.84
54.21
47.09
43.37
38.84
35.20
32.45
30.84
30.37
23.81
23.31
22.06
f1 (ppm)