Supporting Information for

Intercepting Wacker Intermediates With Arenes: C–H Functionalization and Dearomatization

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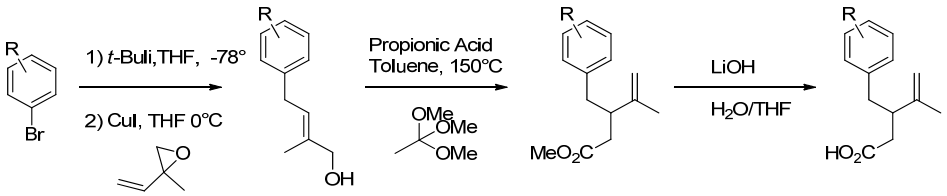
General Information

Chemicals were either used as received or purified according to *Purification of Common Laboratory Chemicals*.[1] Glassware was dried in an oven at 150°C or flame dried and cooled under a dry atmosphere prior to use. All reactions were performed using common dry, inert atmosphere techniques. Reactions were monitored by TLC and visualized by a dual short wave/long wave UV lamp and stained with an ethanolic solution of potassium permanganate or p-anisaldehyde. Column flash chromatography was performed using 230-400 mesh silica gel. NMR spectra were recorded on Varian Mercury 300, Varian Unity Plus 400, and Varian Mercury 400 spectrometers. Chemical shifts for ^{1}H NMR were reported as δ, parts per million, relative to the signal of $CHCl_3$ at 7.26 ppm. Chemical shifts for ^{13}C NMR were reported as δ, parts per million, relative to the center line signal of the $CDCl_3$ triplet at 77.0 ppm. Proton and carbon assignments were established using spectral data of similar compounds. The abbreviations s, br. s, d, dd, br. d, ddd, t, q, br. q, m, and br. m stand for the resonance multiplicity singlet, broad singlet, doublet, doublet of doublets, broad doublet, doublet of doublet of doublets, triplet, quartet, broad quartet, multiplet and broad multiplet, respectively. IR spectra were recorded on an Avatar 360 FT-IR spectrometer. Mass spectra were recorded at the Mass Spectrometry Facility at the Department of Chemistry of the Boston University in Boston, MA on a Waters Q-Tof API-US with ESI high resolution mass spectrometer. Concentration refers to removal of solvent under reduced pressure (house vacuum at ca. 20 mmHg)

[1] Armarego, W. L. F.; Perrin, D. D. *Purification of Laboratory Chemicals 4th Edition*; Butterworth-Heinemann: Oxford, 1996.

General Procedure for Preparation of Starting materials



General Procedure for Preparation of Starting Materials for Substrates 1a-6a

Formation of AllylicArene

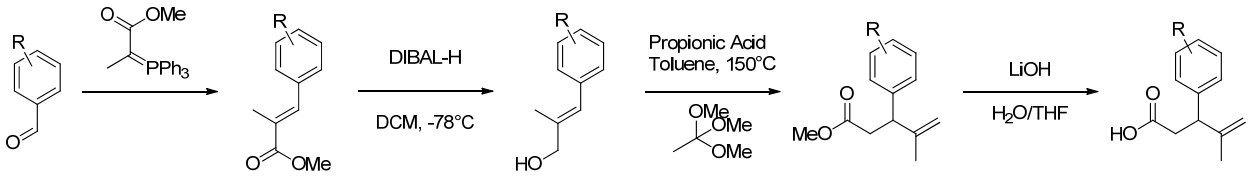
To an argon-purged, flame dried round bottom flask equipped with a rubber septum and magnetic stirbar, aryl bromide (1 equiv) was added as a solution in dry THF under argon (50mL/g of substrate). The flask was cooled to -78 °C and a solution of *t*-butyl lithium (2.2 equiv, 1.7M solution in hexanes) was added dropwise and stirred for 5 minutes. In a separate, argon-purged, flame dried flask, copper(I) iodide (15 mol %) and vinyl oxirane(1.5 equiv) were dissolved in THF and cooled to 0°C. To this, the aryl lithium solution was transferred dropwise via cannula over a span of 10 minutes. The solution stirred for 45 minutes and was quenched with a saturated solution of ammonium chloride. The THF was evaporated in vacuo and the aqueous layer was extracted with ethyl acetate (3X). The aqueous layer was discarded and the organic layer was washed with water (2X) and brine. The organic layer was concentrated in vacuo and the residue was purified by column chromatography on SiO_2.

Johnson Claisen Rearrangement

To a sealed tube, allylic arene (1 equiv) was added to a solution of toluene and of trimethyl orthoacetate (10 equiv). To this, propionic acid (0.1 equiv) was added and the reaction was refluxed at 150 °C for 12 hours. The reaction was diluted with ethyl acetate and washed with water (2 X), brine (1 X), and dried over $Mg(SO_4)_2$. The residue was concentrated in vacuo and purified by column chromatography.

Saponification

To a round bottom flask, the Claisen product (1 equiv) and LiOH (4 equiv) were dissolved in 4:1 mixture of THF and water. To this, enough methanol was added to make the mixture a homogenous solution. Remaining solution was then diluted with ethyl acetate. The aqueous layer was washed with 1M aqueous solution of NaOH, and then extracted with ethyl acetate (3 X). The aqueous layer was acidified to a ~pH 3, using an aqueous 3M HCl solution, which was then extracted with ethyl acetate (3 X). The acidified ethyl acetate was then washed with water (3X), with brine (1X), and dried over $Mg(SO_4)_2$ and concentrated in vacuo. The residue was then purified using column chromatography to afford the starting material.



General Procedure for Preparation of Starting Materials for Compounds 9a-16a

Formation of the α,β unsaturated ester

To a slurry of DBU (3.6 equiv) and LiCl (3.6 equiv) in THF (0.6M with respect to benzaldehyde), 1.2 equivalents of phosphonate was added under nitrogen. The reaction was stirred for 1 hour upon which benzaldehyde (1 equiv) was added dropwise as a solution in THF. The reaction after 12 hours, the reaction was quenched with a saturated solution of NH_4Cl. The aqueous layer was extracted with ether (3X), washed with brine (1X) and dried over $Mg(SO_4)_2$. The filtrate was concentrated in vacuo and purified by column chromatography

Reduction of the α,β unsaturated ester

A solution of α,β unsaturated ester in dichloromethane (15 mL per mmol of substrate) was cooled to -78 °C under argon. To this, DIBAL-H (2.25 equiv) was added dropwise. After 1 hour the reaction was quenched with a saturated solution of Rochelle's salt and warmed to room temperature over a period of 2 hours. The aqueous layer was extracted with dichloromethane (3X) and the organic layers were combined and washed with brine (1X) and dried over $Mg(SO_4)_2$. The organic layer was then concentrated in vacuo and purified by column chromatography.

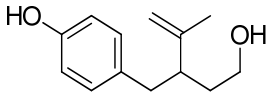
Johnson Claisen Rearrangement

To a sealed tube, allylic arene (1 equiv) was added to a solution of toluene and of trimethyl orthoacetate (10 equiv). To this, propionic acid (0.1 equiv) was added and the reaction was refluxed at 150 °C for 12 hours. The reaction was diluted with ethyl acetate and washed with water (2 X), one time with brine, and dried over $Mg(SO_4)_2$. The residue was concentrated in vacuo and purified by column chromatography.

Saponification

To a round bottom flask, the Claisen product (1 equiv) and LiOH (4 equiv) were dissolved in 4:1 mixture of THF and water. To this, enough methanol was added to make the mixture a homogenous solution. Remaining solution was then diluted with ethyl acetate. The aqueous layer was washed with 1M aqueous solution of NaOH, and then extracted with ethyl acetate (3 X). The aqueous layer was acidified to a ~pH 3, using an aqueous 3M HCl solution, which was then extracted with ethyl acetate (3 X). The acidified ethyl acetate was then washed with water (3 X), with brine (1 X), and dried over $Mg(SO_4)_2$ and concentrated in vacuo. The residue was then purified using column chromatography to afford the starting material.

Data for Starting Materials



4-(2-(2-hydroxyethyl)-3-methylbut-3-en-1-yl)phenol (1)

R_f (Hexane/EtOAc 4:1): 0.23

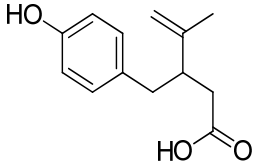
IR (Neat): 3343, 2936, 2360, 2342, 1645, 1614, 1598, 1514, 1449, 1375, 1240, 1047, 893, 828 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 6.99 (d, *J* = 8.5Hz, 2 H), 6.73 (d, *J* = 8.5, 2 H), 3.92 (br. s., 1 H), 4.74 (m, 1 H), 4.69 (m, 1 H), 3.59 (m, 2 H), 2.69 (dd, *J* = 13.7, 7.6 Hz, 1 H), 2.57 (dd, *J* = 13.4, 7.0 Hz, 1 H), 2.47 (dtd, *J* = 9.5, 7.5, 7.5, 5.2 Hz, 1 H), 1.68 (m, 3 H), 1.64 (m, 2 H);

^{13}C NMR ($CDCl_3$, 125MHz) δ: 154.0, 147.2, 132.2, 130.0, 115.1, 112.2, 61.5, 46.2, 39.7, 34.7, 18.6;

HRMS (ESI) not found.

LRMS (ESI) *m/z* for $C_{13}H_{13}O_2Na^+$([M+Na]$^+$) found 229.15



3-(4-hydroxybenzyl)-4-methylpent-4-enoic acid (starting material for compound 3)

R_f (Hexane/EtOAc 7:3): 0.36;

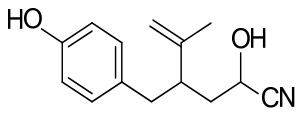
IR (Neat): 3251, 1706, 1648, 1613, 1514, 1442, 1229, 896, 829, 611cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 7.01 (d, *J* = 8.5 Hz, 2H), 6.73 (d, *J* = 8.5 Hz, 2H), 4.74 (m, 2H), 2.8 (qint, *J* = 7.5 X4 Hz, 1H), 2.7, (m, 1H), 2.56 (m, 1H), 2.13 (m, 2H), 1.71 (s, 3H);

^{13}C NMR ($CDCl_3$, 125MHz) δ: 178.1, 153.9, 145.9, 131.8, 130.2, 115.1, 112.0, 45.0, 39.1, 37.7, 20.0;

HRMS (ESI) not found.

LRMS (ESI) *m/z* for $C_{14}H_{17}O_2^+$([M+H]$^+$) found 220.10.



2-hydroxy-4-(4-hydroxybenzyl)-5-methylhex-5-enenitrile (isolated as a 1:1 mixture of diastereomers) (starting material for compound 4)

R_f (DCM/Acetone 9:1): 0.56;

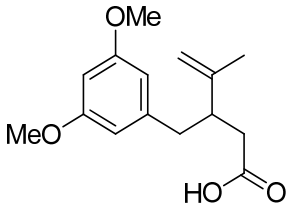
IR (Neat): 2402, 2927, 2248, 1708, 1645, 1613, 1598, 1515, 1447, 1378, 1227, 1173, 1081, 905, 830, 734 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 7.00 (d, *J* = 8.4 Hz, 2 H), 6.74 (d, *J* = 8.4Hz 2 H), 4.86 (t, *J* = 1.5 Hz, 1 H, diastereomer 1), 4.81 (t, *J* = 1.5 Hz, 1 H. diastereomer 1), 4.80 (s, 1 H, diastereomer 2), ;4.78 (s, 1 H, diastereomer 2), 4.41 (dd, *J* = 7.0, 5.8 Hz, 1 H, diastereomer 1), 4.36 (dd, *J* = 9.3, 6.3 Hz, 1 H, diastereomer 2), 2.70 (m, 2H), 2.58 (m, 2H), 1.89 (m, 2H, overlap);

^{13}C NMR ($CDCl_3$, 125MHz) δ: 154.0, 145.9, 145.2, 131.5, 130.0, 115.3, 114.22, 113.7, 60.8, 59.7, 45.5, 44.9, 39.4, 39.3, 37.6, 37.4, 30.9, 18.6, 18.6;

HRMS (ESI) not found.

LRMS (ESI) *m/z* for $C_{14}H_{18}NO_2$([M+H]$^+$)found 232.12.



4-methyl-3-(3,5-dimethoxybenzyl)pent-4-enoic acid (5)

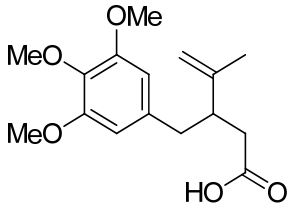
R_f (Hexane/EtOAc 1:1): 0.44;

IR (Neat): 29378, 2838, 1704, 1594, 1459, 1428, 1292, 1204, 1146, 1057, 830, 753 cm^{-1};

^{1}H NMR ($CDCl_3$, 300MHz)δ: 6.32 (m, 3 H), 4.79 (t,*J* = 2 X 2.2 Hz,1 H), 4.74 (s, 1 H), 3.77 (s, 6 H), 2.85 (ddd, *J* = 14.9, 7.9, 7.1 Hz, 1 H), 2.72 (dd, *J* = 13.5, 6.9 Hz, 1 H), 2.56 (dd, *J* = 13.5, 8.1 Hz, 1 H), 2.44-2.41 (m, 2 H), 1.73 (s, 3 H);

^{13}C NMR ($CDCl_3$, 100MHz) δ: 178.9, 160.6, 145.9, 142.0, 112.0, 107.1, 98.1, 55.2, 44.5, 40.2, 37.9, 20.0;

HRMS (ESI) *m/z* calculated for $C_{15}H_{21}O_4^+$([M+H]$^+$) 265.1440, found 265.1443.



4-methyl-3-(3,4,5-trimethoxybenzyl)pent-4-enoic acid (7)

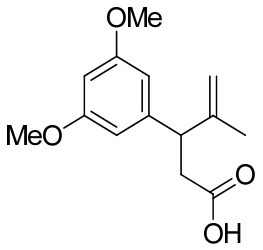
R_f (Hexane/EtOAc 4:1):0.13;

IR (Neat): 3014, 2938, 2839, 1706, 1590, 1508, 1458, 1420, 1326, 1236, 1124, 1005, 749, 666 cm^{-1};

^{1} H NMR ($CDCl_3$, 500MHz) δ: 6.37 (s, 2 H), 4.81 (s, 1 H), 4.75 (s, 1 H), 3.84 (s, 6 H), 3.82 (s, 3 H), 2.84 (dddd, *J* = 7.2 Hz, 1 H), 2.72 (dd, *J* = 13.9, 6.9 Hz, 1 H), 2.57(dd, *J* = 13.6, 8.0 Hz, 1 H), 2.48-2.35 (m, 2 H),1.74 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 160.7, 147.5, 145.6, 110.6, 106.1, 98.0, 61.2, 55.2, 49.1, 35.7, 21.1;

HRMS (ESI) *m/z* calculated for $C_{16}H_{23}O_5^+$ ($[M+H]^+$) 237.1491, found 237.1498.



3-(3,5-dimethoxyphenyl)-4-methylpent-4-enoic acid(11)

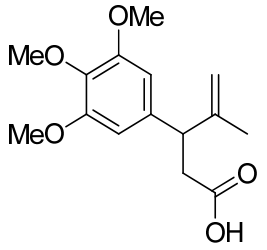
R_f (Hexane/EtOAc 3:1): 0.25;

IR (Neat):2941, 2838, 1708, 1596, 1461, 1430, 1291, 1205, 1157, 1067, 897, 835, 740, 698 cm^{-1};

^{1} H NMR ($CDCl_3$, 500MHz) δ: 6.39 (d, *J* = 2.1 Hz, 2 H), 6.34 (m, 1H), 4.92 (d, *J* = 5.2 Hz, 2 H), 3.79 (s, 6H), 3.74, (m, 1 H), 2.88 (dd, *J* = 15.6, 8.5 Hz, 1H), 2.75 (dd, *J* = 15.6, 8.5 Hz, 1 H), 1.66 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 178.2, 160.8, 146.1, 144.2, 110.7, 105.8, 98.5, 55.2, 48.3, 38.9, 21.7;

HRMS (ESI) *m/z* calculated for $C_{14}H_{19}O_4^+$($[M+H]^+$) 251.1295, found 251.1295.



4-methyl-3-(3,4,5-trimethoxyphenyl)pent-4-enoic acid (13)

R_f (Hexane/EtOAc 3:1): 0.1;

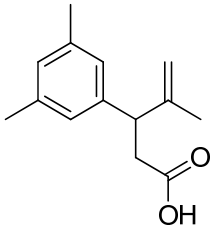
IR (Neat):2640, 2839, 1709, 1648, 1591, 1509, 1461, 1422, 1330, 1239, 1127, 1010, 903, 663 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 6.43 (s, 2 H), 4.92 (s, 2 H), 3.83 (s, 6 H), 3.83 (m, 9H), 2.87 (dd, *J* = 15.8, 8.2 Hz, 1 H), 2.72 (dd, *J* = 15.9, 7.3 Hz, 1 H), 1.65 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 178.1, 153.1, 146.3, 137.5, 136.7, 110.6, 104.6, 60.8, 56.1, 48.4, 39.3, 21.8;

HRMS (ESI) not found.

LRMS (ESI) *m/z* for $C_{15}H_{21}O_5^+$([M+H]$^+$) found 281.13.



3-(3,5-dimethylphenyl)-4-methylpent-4-enoic acid (15)

R_f (Hexane/EtOAc 9:1):0.08;

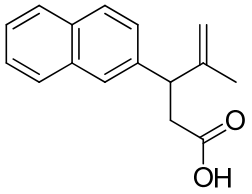
IR (Neat): 3018, 2919, 1709, 1606, 1416, 1300, 1252, 1176, 893, 849, 704 cm^{-1};

^{1}H NMR ($CDCl_3$, 400MHz) δ: 11.53(s, 1 H, OH), 6.87 (s, 1 H), 6.83(s, 2 H), 4.93 (s, 1 H), 4.89 (s, 1 H), 3.71 (t, *J* = 7.8 Hz, 1 H), 2.88 (dd, *J* = 15.7, 8.5 Hz, 1 H), 2.73 (dd, *J* = 15.7, 7.1 Hz, 1 H), 2.29 (s, 6 H), 1.63 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 178.4, 146.5, 141.7, 137.9, 128.5, 125.4, 110.4, 48.0, 39.1, 21.7, 21.3;

HRMS (ESI) not found.

LRMS (ESI) *m/z* for $C_{14}H_{19}O_2^+$([M+H]$^+$) found 241.13.



4-methyl-3-(naphthalen-1-yl)pent-4-enoic acid (17)

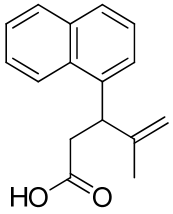
R_f (Hexane/EtOAc/AcOH 81:15:4): 0.25;

IR (Neat): 3054, 2972, 2918, 1707, 1648, 1600, 1508, 1415, 1375, 1274, 1175, 945, 896, 858, 819, 746 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz)δ: 7.80 (m, overlap, 3 H), 7.67 (s, 1 H), 7.45 (m, 2 H), 7.35 (dd, *J*= 8.6, 1.7 Hz, 2 H), 5.00 (s, 1 H), 4.95 (s, 1 H), 3.94 (t, *J* = 7.8 Hz, 1 H), 2.98 (dd, *J* = 15.9, 8.6 Hz, 1 H), 2.86 (dd, *J* = 15.9, 7.8 Hz, 1 H), 1.64 (s, 3 H);

^{13}C NMR ($CDCl_3$, 125MHz) δ: 176.7, 146.3, 139.2, 133.4, 132.5, 128.2, 127.6, 126.2, 126.0, 126.0, 125.6, 110.9, 106.2, 48.3, 38.7, 21.7;

HRMS (ESI) *m/z* calculated for $C_{16}H_{17}O_2^+$([M+H]$^+$) 241.1229, found 241.1231.



4-methyl-3-(naphthalen-1-yl)pent-4-enoic acid (19)

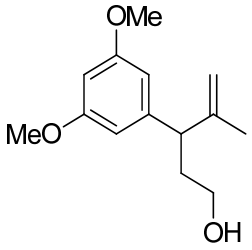
R_f (Hexane/EtOAc4:1): 0.31;

IR (Neat): 3044, 2968, 1706, 16495, 1511, 1415, 1027, 1026, 897, 798, 779 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 7.90 (d, *J* = 8.3 Hz, 1 H), 7.78 (d, *J* = 8.3 Hz, 1 H), 7.66 (d, *J* = 7.8 Hz, 1 H), 7.55 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1 H), 7.49 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 1 H), 7.33 (d, *J* = 8.3 Hz, 1 H), 4.09 (dd, *J* = 9.8, 2.7 Hz, 1 H), 3.61 (d, *J* = 17.6 Hz, 1 H), 3.33 (d, *J* = 17.4 Hz, 1 H), 3.28 (dd, *J* = 18.1, 9.8 Hz, 1 H), 2.94 (d, *J* = 18.2, 2.8 Hz, 1 H), 1.75 (s, 3 H);

^{13}C NMR ($CDCl_3$, 125MHz) δ: 146.0, 137.6, 134.0, 131.7, 128.9, 127.5, 126.2, 125.6, 125.4, 124.2, 123.1, 111.8, 43.3, 39.0, 22.0;

HRMS (ESI) *m/z* calculated for $C_{16}H_{17}O_2^+$([M+H]$^+$) 241.1229, found 241.1229.



3-(3,5-dimethoxyphenyl)-4-methylpent-4-en-1-ol (21)

R_f (Hexane/EtOAc4:1): 0.24;

IR (Neat): 3392, 2941, 2838, 2361, 1596, 1462, 1429, 1291, 1205, 1157, 1057, 894, 834, 698 cm^{-1};

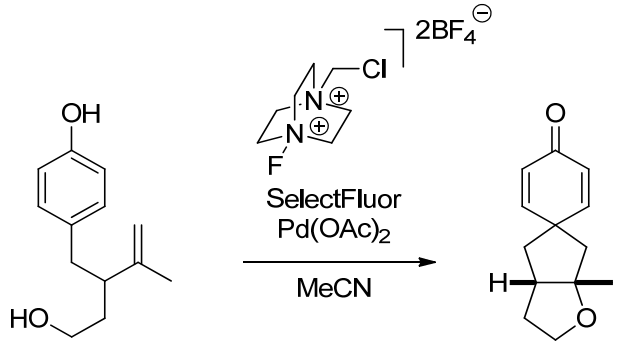
^{1}H NMR ($CDCl_3$, 400MHz) δ: 6.39 (d, *J* = 2.3 Hz, 1 H), 6.32 (t, *J* = 2.3 Hz, 1 H), 4.94(s, 1 H), 4.86 (t, *J* = 1.5 Hz, 1 H), 3.77 (s, 6 H), 3.60 (m, 2 H), 3.34 (t, *J* = 7.6 Hz, 1 H), 2.09 (dq, *J* = 13.7, 6.9 Hz, 1 H), 1.95 (ddt, *J*= 13.6, 7.9, 6.4 Hz, 1 H), 3.20 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 160.7, 147.5, 145.6, 110.6, 106.1, 98.0, 61.2, 55.2, 49.1, 35.7, 21.1;

HRMS (ESI) *m/z* calculated for $C_{14}H_{21}O_3$([M+H]$^+$) 237.1491, found 237.1498.

General Procedure for Palladium Mediated Cyclizations

A flame dried 100 mL round bottom flask is equipped with a rubber septum and magnetic stir bar and is charged with the aryl cyclization precursor (0.15 mmol, 1.0 equiv) and suspended in 100ml of acetonitrile. To this, iodobenzene diacetate (0.45 mmol, 3.0 equiv) and palladium diacetate (0.015 mmol, 0.10 equiv) were added and the reaction was stirred at room temperature for 12 hours. After the reaction is complete (as judged by TLC analysis), the mixture was concentrated *in vacuo*. The resultant residue was separated by column chromatography on silica gel, using the solvent system indicated, to afford the desired cyclized product.



6a'-methyl-2',3',3a',4',6',6a'-hexahydrospiro[cyclohexa[2,5]diene-1,5'-cyclopenta[b]furan]-4-one (2) According the general procedure, the aryl alcohol (100 mg, 0.49 mmol, 1.0 equiv), Selectfluor (258 mg, 0.73mmol, 3.0 equiv) and palladium diacetate (11 mg, 0.05 mmol, 0.10 equiv) in dry acetonitrile (78 mL) afforded the cyclized product 43 mg in 43% yield as a yellow oil after purification by chromatography on SiO_2 (80:20, Hexanes/EtOAc) (12 h reaction time).

R_f (DCM/Acetone 95:5): 0.72;

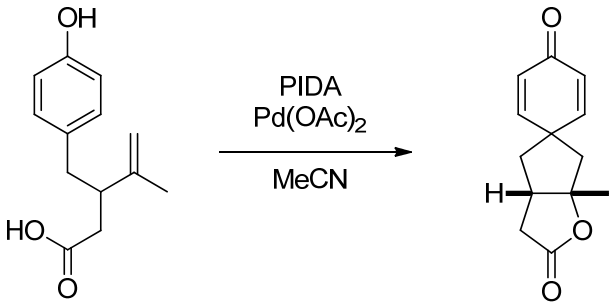
IR (Neat): 2963, 2930, 2863, 2360, 1662, 1451, 1408, 1376, 1259, 1179, 1141, 1110, 1018, 860 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 7.02 (m, 1 H), 6.93 (m, 1 H), 6.20 (m, 2 H), 3.97(m, 2 H), 2.64 (q, J = 8.31 Hz, 1 H), 2.17 (m, 2 H), 2.05 (dd, J = 13.3, 8.4 Hz, 1 H), 1.91 (d, J = 14.2 Hz, 1 H), 1.75 (m, 2 H), 1.4 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 153.1, 151.3, 128.7, 128.1, 94.9, 49.6, 48.1, 44.8, 44.4, 35.5, 26.3;

HRMS (ESI) not found.

LRMS (ESI) *m/z* for $C_{13}H_{17}O_2^+$([M+H]$^+$) found 205.13.



6a'-methyl-2',3',3a',4',6',6a'-hexahydrospiro[cyclohexa[2,5]diene-1,5'-cyclopenta[b]furan]-4-one (3) According the general procedure, the aryl carboxylic acid (85 mg, 0.38 mmol, 1.0 equiv), phenyliodine diacetate (373 mg, 1.16 mmol, 3.0 equiv) and palladium diacetate (24 mg, 0.12 mmol, 0.30 equiv) in dry acetonitrile (85 mL) afforded 17 mgs of cyclized product **5b** after purification by chromatography on SiO_2 (65:35 EtOAc/Hex) (2 hour reaction time).

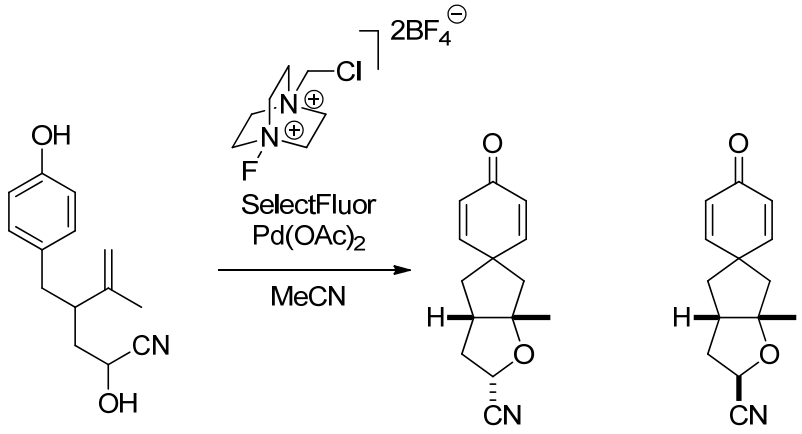
R_f (EtOAc/Hexane 65:35): 0.23;

IR (Neat): 1769, 1665, 1623, 1442, 12267, 1232, 1133, 1096, 952, 861, 740, 703 cm^{-1};

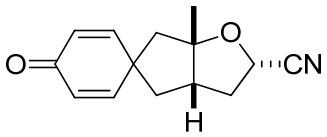
^{1}H NMR ($CDCl_3$, 500 MHz) δ: 6.87 (ddd, *J* = 16.4, 10.0, 2.7 Hz, 2 H), 6.25 (ddd, *J* = 10.0, 5.7, 1.7 Hz, 2 H), 2.94 (dd, *J* = 17.6, 9.3 Hz, 1 H), 2.86 (ddd, *J* = 8.9 Hz, 1 H), 2.50 (d, *J* = 5.6 Hz, 1 H), 2.47 (d, *J* = 2.69 Hz, 1 H), 2.22 (dd, *J* = 13.7, 8.1 Hz, 1 H), 2.08 (d, *J* = 15.2, 1 H), 1.87 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 153.1, 151.3, 128.7, 128.1, 94.9, 49.6, 48.1, 44.8, 44.4, 35.5, 26.3;

HRMS (ESI) *m/z* calculated for $C_{13}H_{15}O_3$([M+H]$^+$) 219.1021, found 219.1012.



6a'-methyl-4-oxo-2',3',3a',4',6',6a'-hexahydrospiro[cyclohexa[2,5]diene-1,5'-cyclopenta[b]furan]-2'-carbonitrile (4) According the general procedure, the aryl cyanohydrin (80 mg, 0.35mmol, 1.0 equiv), Selectfluor (184 mg, 0.52 mmol, 1.5equiv) and palladium diacetate (7.7 mg, 0.035mmol, 0.10 equiv) in dry acetonitrile (40 mL) afforded the cyclized product **13b** as a mixture of diastereoisomers (13 mg and 11 mg, 30% combined) as a yellow oil after purification by chromatography on SiO_2 (80:20, Hexanes/EtOAc) (12 h reaction time).



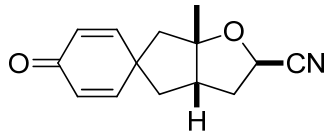
R_f (DCM/Acetone 95:5): 0.24;

IR (Neat): 2973, 2362, 2340, 1663, 1623, 1452, 1408, 1380, 1259, 1182, 1101, 1071, 1024, 860 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 6.91 (dd, *J*=10.3, 2.93 Hz, 1 H), 6.86 (dd, *J*=10.3, 2.9 Hz, 1 H), 6.22 (dd, *J*=4.9, 2.0 Hz, 1 H), 6.20 (dd, *J*=4.7, 2.0 Hz, 1 H), 4.89 (t, *J*=7.5 Hz, 1 H), 2.87 (qd, *J*=8.4, 2.0 Hz, 1 H), 2.62 (dt, *J*=13.5, 7.6 Hz, 1 H), 2.35 (ddd, *J*=13.4, 7.8, 2.1 Hz, 1 H), 2.29 (d, *J*=14.7 Hz, 1 H), 2.12 (dd, *J*=13.7, 8.3 Hz, 1 H), 1.97 (d, *J*=14.9 Hz, 1 H), 1.75 (dd, *J*=13.7, 9.1 Hz, 1 H), 1.58 (s, 3 H);

^{13}C NMR ($CDCl_3$, 100MHz) δ:185.6, 154.3, 151.6, 128.2, 127.6, 119.8, 96.4, 65.6, 50.3, 49.2, 49.1, 43.4, 36.8, 26.7;

HRMS (ESI) *m/z* calculated for $C_{14}H_{16}NO_2^+$ ([M+H$^+$]) 230.1181, found 230.1185.



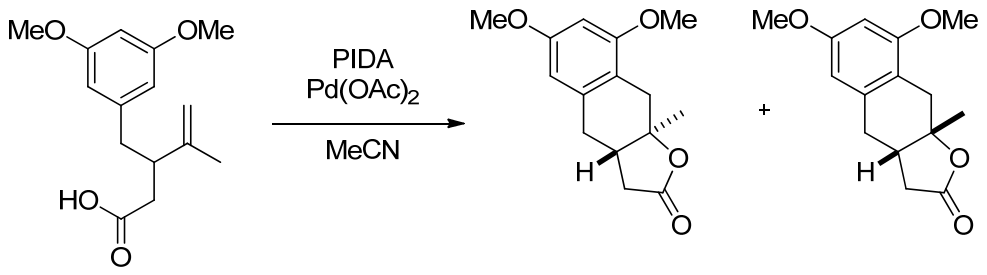
R_f (DCM/Acetone 95:5): 0.30;

IR (Neat): 2967, 2933, 2871, 1660, 1622, 1452, 1408, 1380, 1259, 1181, 1100, 1070, 1024, 859 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 7.02 (dd, *J*=9.9, 3.1 Hz, 2 H), 6.93 (dd, *J*=9.9, 3.1 Hz, 3 H), 6.26 (ddd, *J*=11.7, 10.3, 2.0 Hz, 4 H), 4.85 (dd, *J*=8.7, 2.8 Hz, 2 H), 2.81 (dtd, *J*=9.9, 8.3, 8.3, 1.3 Hz, 3 H), 2.58 (dt, *J*=13.7, 8.1 Hz, 3 H), 2.53 (d, *J*=14.7 Hz, 3 H), 2.32 (dd, *J*=14.0, 10.0 Hz, 1 H), 2.26 (ddd, *J*=13.5, 2.9, 1.2 Hz, 4 H), 2.12 (ddd, *J*=13.7, 8.3, 1.5 Hz, 3 H), 2.00 (dd, *J*=14.7, 1.5 Hz, 3 H);

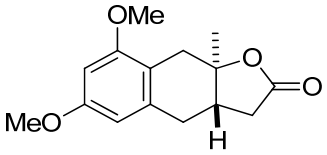
^{13}C NMR ($CDCl_3$, 125MHz) δ:185.5, 153.9, 152.4, 127.8, 127.6, 119.5, 96.6, 65.2, 49.8, 48.9, 48.8, 43.4, 37.7, 25.8;

HRMS (ESI) *m/z*calculated for $C_{14}H_{16}NO_2^+$ ($[M+H]^+$) Calculated 230.1181, found 230.1178.



5,7-dimethoxy-3,3a,8,8a-tetrahydro-2 H-indeno[2,1-b]furan-2-one (6)

According the general procedure, the aryl carboxylic acid (100 mg, 0.38 mmol, 1.0 equiv), phenyliodine diacetate (182.9 mg, 0.57 mmol, 1.5 equiv) and palladium diacetate (8.9 mg, 0.038 mmol, 0.10 equiv) in dry acetonitrile (100 m) afforded the cyclized product **2b** and **2b'** (51 mg and 39 mg, 91% combined) as a white precipitate after purification by chromatography on SiO_2 (80:20, Hexanes/EtOAc) (12 h reaction time).



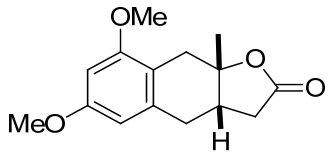
R_f (Hexane/EtOAc4:1): 0.64;

IR (Neat): 2922, 2850, 1778, 1609, 1464, 1321, 1251, 1207, 1146, 1053, 945, 832, 741 cm^{-1};

^{1}H NMR ($CDCl_3$,500MHz) δ: 6.33 (d, *J* = 2.2 Hz, 1 H), 6.26 (d, *J* = 2.2 Hz, 1 H), 3.78 (s, 3 H), 3.77 (s, 3 H), 3.11 (d, *J* = 15.7 Hz, 1 H), 2.97 (dd, *J* = 16.3, 3.8 Hz, 1 H), 2.67-2.78 (m, 2 H), 2.59-2.44 (m, 3 H), 1.20 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 176.1, 159.3, 157.8, 137.7, 115.3, 104.8, 96.9, 87.5, 55.5, 55.4, 39.9, 34.9, 34.9, 34.3, 32.4, 28.6;

HRMS (ESI) *m/z* calculated for $C_{15}H_{19}O_4^+$([M+H]$^+$) 263.1283, Found 263.1285.



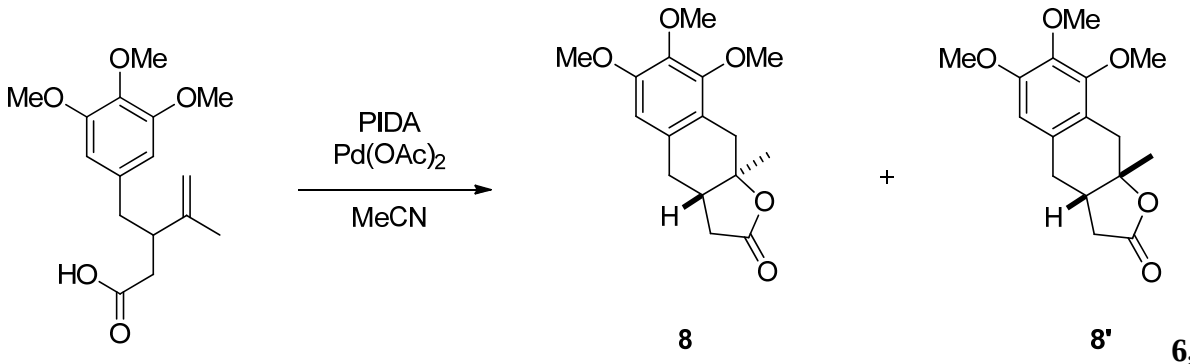
R_f (Hexane/EtOAc4:1): 0.53;

IR (Neat):2927, 2848, 1766, 1596, 1496, 1464, 1323, 1204, 1147, 1095, 1052, 955, 835, 737 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 6.34 (d, *J* = 2.2 Hz, 1 H), 6.26 (d, *J* = 2.2 Hz, 1 H), 3.79 (s, 3 H), 3.78 (s, 3 H), 3.16 (d, *J* = 15.4 Hz, 1 H), 2.81 (dd, *J* = 18.5, 8.8 Hz, 1 H), 2.63 (d, *J* = 15.4 Hz, 1 H), 2.52 (m, 2 H), 1.12 (dd), *J* = 18.5, 5.0 Hz, 1 H), 1.50 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75 MHz) δ:176.2, 158.9, 136.4, 115.8, 104.7, 96.5, 84.8, 55.3, 62.1, 36.0, 32.9, 31.0, 17.8;

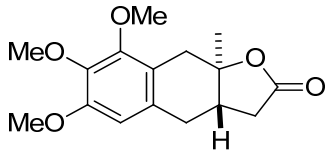
HRMS (ESI) *m/z* calculated for $C_{15}H_{19}O_4^+$($[M+H]^+$) 263.1283, Found 263.1277.



8 **8'**

6,7,8-trimethoxy-9a-methyl-3a,4,9,9a-tetrahydronaphtho[2,3-b]furan-2(3 H)-one (8)

According the general procedure, the aryl carboxylic acid (100 mg, 0.34 mmol, 1.0 equiv), phenyliodine diacetate (216 mg, 0.67 mmol, 2.0 equiv) and palladium diacetate (7.0 mg, 0.03mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized products **3b** and **3b'** in 37 mg and 28 mg respectively, for a combined yield of 65%. Crude NMR analysis revealed a 1.8:1 diastereomeric ratio in favor for product **3b**. Both diastereomers were a tan solid after purification by chromatography on SiO_2 (80:20, Hexanes) (12 h reaction time).



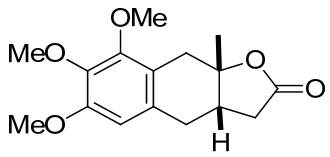
R_f (Hexane/EtOAc 4:1): 0.31;

IR (Neat): 2937, 1779, 1603, 1493, 1461, 1340, 1255, 1244, 1119, 1069, 1032, 668 cm^{-1} ;

^{1}H NMR ($CDCl_3$, 500MHz) δ: 6.52 (s, 1 H), 3.86 (s, 3 H), 3.84 (s, 3 H), 3.83 (s, 3 H), 3.11 (d, *J* = 15.6 Hz, 1 H), 3.84 (dd, *J* = 18.6, 10.4 Hz, 1 H), 2.75 (dd, *J* = 13.9, 4.7 Hz, 1 H), 2.63 (d, *J* = 15.3, 1 H), 2.56 (m, 1 H), 2.51 (dd, *J* = 14, 5.2 Hz), 2.20 (dd, *J* = 18.5, 4.7 Hz), 1.53 (s 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 175.9, 152.1, 152.0, 140.7, 130.0, 120.8, 107.9, 84.5, 60.8, 60.5, 55.9, 42.3, 36.3, 32.9, 30.7, 17.6;

HRMS (ESI) *m/z* calculated for $C_{16}H_{21}O_5^+$ ($[M+H]^+$) 293.1389, Found 293.1386.



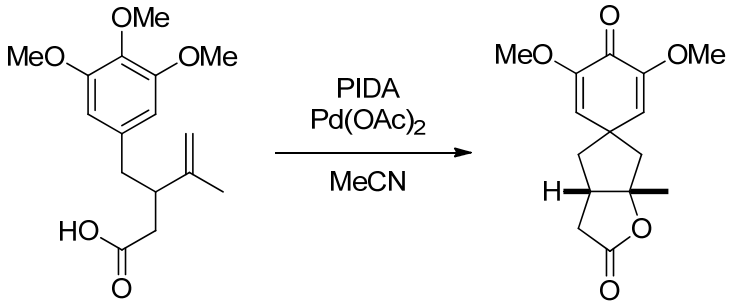
R_f (Hexane/EtOAc 4:1): 0.25;

IR (Neat): 2936, 1778, 1602, 1493, 1461, 1412, 1340, 1255, 1119, 1069, 1031, 987, 668 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 6.43 (s, 1 H), 3.85 (s, 3 H), 3.84 (s, 3 H), 3.82 (s, 3 H), 3.19 (d, *J* = 15.5 Hz, 1 H), 2.96 (dd, *J* = 16.0, 3.9 Hz, 1 H), 2.8 (d, *J* = 15.7, 1 H), 2.68 (m, 1 H), 2.57 (d, *J* = 8.4 Hz, 1 H), 2.48 (m, 1 H), 1.21 (s, 3 H)

^{13}C NMR ($CDCl_3$, 75MHz) δ: 175.9, 152.2, 151.5, 141.1, 131.3, 120.5, 107.7, 87.2, 61.3, 61.0, 56.1, 39.9, 34.9, 34.2, 33.4, 28.6;

HRMS (ESI) *m/z* calculated for $C_{16}H_{21}O_5^+$([M+H]$^+$) 292.1389, found 293.1376.



(3a'S,6a'S)-3,5-dimethoxy-6a'-methyl-3a',4',6',6a'-tetrahydrospiro[cyclohexa[2,5]diene-1,5'-cyclopenta[b]furan]-2',4(3'H)-dione (8a) According the general procedure, the aryl carboxylic acid (100 mg, 0.34 mmol, 1.0 equiv), phenyliodine diacetate (216 mg, 0.67 mmol, 2.0 equiv) and palladium diacetate (7.0 mg, 0.03mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded 23 mg of cyclized product **3c** as a brown solid in 32% yield, after purification by chromatography on SiO_2 (80:20, Hexanes/EtOAc→99:1 EtOAc/MeOH) (12 h reaction time).

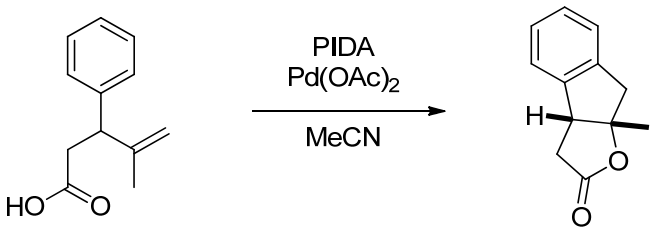
R_f (EtOAc/MeOH 99:1): 0.20;

IR (Neat): 2936, 1778, 1602, 1493, 1461, 1340, 1255, 1119, 1069, 987, 668 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 5.91-5.72 (m, 2 H), 3.66 (s, 6 H), 2.96 (dd, *J* = 18.1, 8.8 Hz, 1), 2.87 (dd, *J* = 8.5 Hz, 1 H), 2.49 (dd, *J* = 7.8, 6.6 Hz, 2 H), 2.22 (ddd, *J* = 13.5, 8.0, 1.5 Hz, 1 H), 2.11 (dd, *J* = 14.8, 1.6 Hz, 1 H), 1.91 (dd, *J* = 13.5, 9.3 Hz, 1 H), 1.6 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 176.0, 175.5, 150.3, 150.0, 120.9, 119.1, 94.9, 55.3, 55.2, 51.6, 46.4, 46.3, 44.8, 35.5, 26.5;

HRMS (ESI) *m/z* calculated for $C_{15}H_{18}O_5Na^+$([M+Na^+]) 301.1052, found 301.1061.



8a-methyl-3,3a,8,8a-tetrahydro-2 H-indeno[2,1-b]furan-2-one (10)

According the general procedure, the aryl carboxylic acid (93 mg, 0.48 mmol, 1.0 equiv), phenyliodine diacetate (460 mg, 1.4 mmol, 3.0 equiv) and palladium diacetate (12 mg, 0.048 mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized product **9b** (55 mg, 60%) as a colorless solid after purification by chromatography on SiO_2 (90:10, Hexanes/EtOAc) (12 h reaction time).

R_f(Hexane/EtOAc 7:3): 0.32;

IR (Neat): 3024, 2970, 2929, 1768, 1483, 1419, 1384, 1255, 1222, 1170, 1119, 1067, 945, 914, 775, 751, 681 cm^{-1};

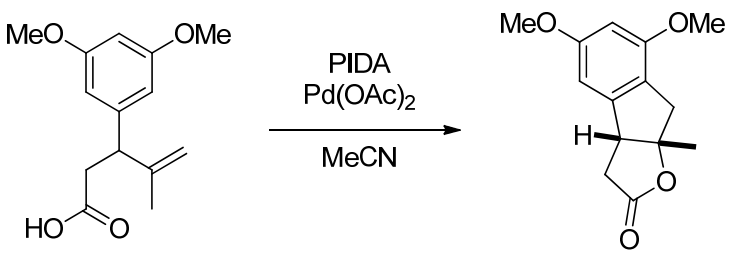
^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.29-7.10 (m, 4 H), 3.63 (d, *J* = 8.7 Hz, 1 H), 3.39 (d, *J* = 17.5 Hz, 1 H), 3.13 (d, *J* = 17.5 Hz, 1 H), 3.05 (dd, *J* = 17.9, 9.0 Hz, 1 H), 2.70 (dt, *J* = 17.8, 0.7 Hz, 1 H), 1.63 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 175.7, 142.5, 140.2, 128.2, 127.6, 125.0, 124.6, 93.7, 50.5, 44.8, 35.4, 24.4;

HRMS (ESI) not found.

LRMS (ESI) *m/z* for $C_{12}H_{13}O_2^+$([M+H]$^+$) found 189.09

Note: For characterization data for the starting material (compound **10**), see: Padwa, A.; Austin, D. J.; Price, A. T.; Semones, M. A.; Doyle, M. P.; Protopopova, M. N.; Winchester, W. R.; Tran, A. *J. Am. Chem. Soc.* **1993**, *115*, 8669.



5,7-dimethoxy-3a,8a-dimethyl-3,3a,8,8a-tetrahydro-2 H-indeno[2,1-b]furan-2-one(12)
According the general procedure, the aryl carboxylic acid (120 mg, 0.48 mmol, 1.0 equiv), phenyliodine diacetate (480 mg, 1.4 mmol, 3.0 equiv) and palladium diacetate (14 mg, 0.048 mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized product **10b** (72 mg, 60%) as a colorless solid after purification by chromatography on SiO_2 (99.5:0.5, DCM/Et_2O) (12 h reaction time).

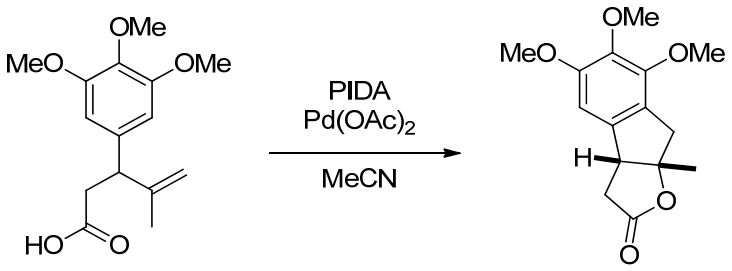
R_f (Hexane/EtOAc/AcOH 68:30:2): 0.31;

IR (Neat): 2940, 1771, 1600, 1495, 1467, 1431, 1331, 1219, 1148, 1066, 946, 836, 737 cm^{-1};

^{1}HNMR ($CDCl_3$,500 MHz) δ: 6.33 (d, *J* = 2.0 Hz, 1 H), 6.29 (d, *J* = 1.7 Hz, 1 H), 3.79 (s, 6 H), 3.61 (d, *J* = 9.0Hz, 1 H), 3.33 (d, *J* = 17.4 Hz, 1 H) , 3.04 (dd, *J* = 17.9, 9.3 Hz, 1 H), 2.96 (d, J = 17. 1 Hz, 1 H), 2.72 (dt, *J* = 17.9, 0.7 Hz, 1 H), 1.64 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 175.7, 161.4, 156.7, 144.5, 120.2, 100.1, 98.0, 94.1, 55.6, 55.2, 51.1, 41.4, 35.1, 24.7;

HRMS (ESI) *m/z* calculated for $C_{14}H_{17}O_4^+$([M+H]$^+$) 249.1127, found 249.1118.



5,6,7-trimethoxy-8a-methyl-3,3a,8,8a-tetrahydro-2 H-indeno[2,1-b]furan-2-one (14) According the general procedure, the aryl carboxylic acid (100 mg, 0.35 mmol, 1.0 equiv), phenyliodine diacetate (276.5 mg, 1.25 mmol, 3.0 equiv) and palladium diacetate (9.6 mg, 0.043 mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized product **11b** (51 mg, 52%) as a clear oil after purification by chromatography on SiO_2 (80:20 Hexanes/ Acetone) (12 h reaction time).

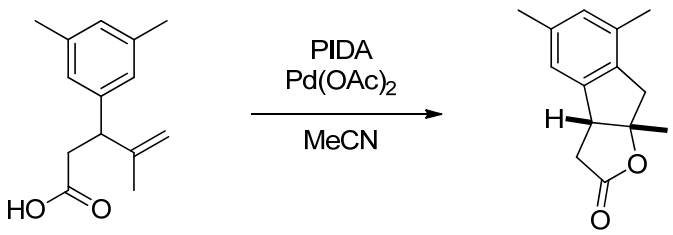
R_f(Hexane/EtOAc4:1): 0.25;

IR (Neat): 2967, 2842, 1590, 1484, 1416, 1345, 1264, 1235, 1171, 1067, 947, 807, 710 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz) δ: 6.45 (s, 1 H), 3.89 (s, 3 H), 3.84 (s, 3 H), 3.84 (s, 3 H), 3.60 (d, *J* = 8.7 Hz, 1 H), 3.41 (d, *J* = 17.1 Hz, 1 H), 2.96-3.15 (m, 2 H), 2.71 (d, *J* = 17.5 Hz, 1 H), 1.66 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 175.6, 154.2, 149.8, 141.6, 137.8, 124.7, 103.1, 94.0, 61.0, 60.4, 56.3, 50.9, 42.1, 35.3, 24.6;

HRMS (ESI) *m/z* calculated for $C_{15}H_{19}O_5^+$($[M+H]^+$) 279.1232, found 279.1233.



5,7,8a-trimethyl-3,3a,8,8a-tetrahydro-2 H-indeno[2,1-b]furan-2-one (16)

According the general procedure, the aryl carboxylic acid (110 mg, 0.48 mmol, 1.0 equiv), phenyliodine diacetate (470 mg, 1.4 mmol, 3.0 equiv) and palladium diacetate (11 mg, 0.048 mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized product **12b** (61 mg, 57%) as a colorless solid after purification by chromatography on SiO_2 (100% DCM) (12 h reaction time).

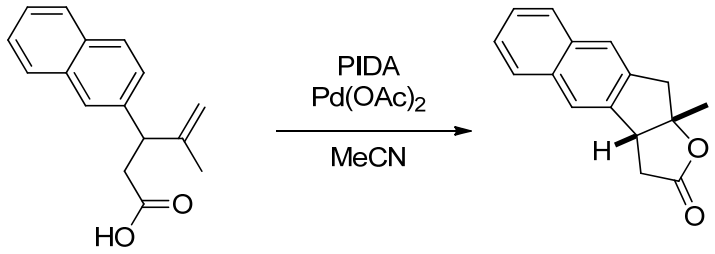
R_f(DCM): 0.28;

IR (Neat):2932, 2857, 2359, 1759, 1510, 1481, 1422, 1388, 1267, 1177, 1119, 1074, 1033, 945, 923, 857, 810, 729, 653 cm^{-1};

^{1}H NMR ($CDCl_3$, 500 MHz) δ: 6.88 (d, *J* = 0.6 Hz, 1 H), 6.81 (s, 1 H), 3.32 (d, *J* = 17.3 Hz, 1 H), 3.06 (dd, *J* = 17.8, 9.1 Hz, 1 H), 3.00 (d, *J* = 17.1 Hz, 1 H), 2.71 (dq, *J* = 17.6, 0.8 Hz, 1 H), 2.30 (s, 3 H), 2.20 (s, 3 H), 1.66 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz)δ: 175.9, 142.5, 137.7, 136.1, 134.2, 129.9, 122.4, 93.8, 50.6, 43.4, 35.5, 24.7, 21.2, 18.9;

HRMS (ESI) *m/z* calculated for $C_{14}H_{17}O_2^+$([M+H]$^+$) 217.1229, found 217.1234.



10a-methyl-3,3a,10,10a-tetrahydro-2 H-benzo[5,6]indeno[2,1-b]furan-2-one (18)

According the general procedure, the aryl carboxylic acid (100 mg, 0.48mmol, 1.0 equiv), phenyliodine diacetate (460 mg, 1.4mmol, 3.0 equiv) and palladium diacetate (10.0 mg, 0.048mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized product **13b**(55 mg, 52%) as a colorless solid after purification by chromatography on SiO_2 (100% DCM) (12 h reaction time).

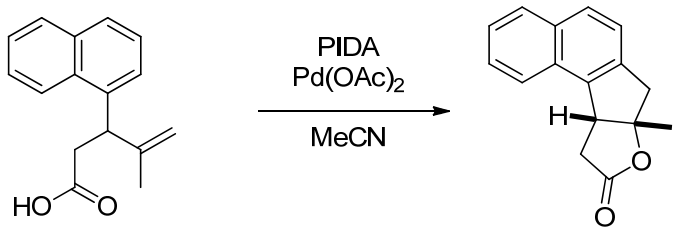
R_f(Hexane/EtOAc4:1): 0.42;

IR (Neat): 2922, 2857, 2360, 2342, 1763, 1608, 1510, 1255, 1169, 1117, 1069, 945, 921, 872, 755 cm^{-1};

^{1}H NMR ($CDCl_3$, 400MHz) δ: 7.83-7.75 (m, 2 H), 7.65 (d, *J* = 9.0 Hz, 2 H), 7.48-7.40 (m, 2 H), 3.81 (d, *J* = 8.7 Hz, 1 H), 3.59 (d, *J* = 17.5 Hz, 1 H), 3.32 (d, *J* = 17.6 Hz, 1 H), 3.19 (dd, *J* = 17.8, 8.7 Hz, 1 H), 2.86 (d, *J* = 17.8 Hz, 1 H), 1.72 (s, 3 H);

^{13}C NMR ($CDCl_3$, 75MHz) δ: 175.5, 141.9, 138.8, 133.7, 133.2, 127.7, 127.6, 125.9, 125.6, 123.6, 123.5, 94.0, 50.13, 44.3, 36.1, 24.4;

HRMS (ESI) *m/z* calculated for $C_{16}H_{15}O_2^+$ ($[M+H]^+$) 239.1072, found 239.1073.



7a-methyl-10,10a-dihydro-7H-benzo[6,7]indeno[2,1-b]furan-9(7aH)-one (20)

According the general procedure, the aryl carboxylic acid (120 mg, 0.50 mmol, 1.0 equiv), phenyliodine diacetate(320 mg, 0.99 mmol, 2.0 equiv) and palladium diacetate (11 mg, 0.05mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized product **14b** (63 mg, 53%) as a colorless solid after purification by chromatography on SiO_2(90:10, Hexanes/EtOAc) (12 h reaction time).

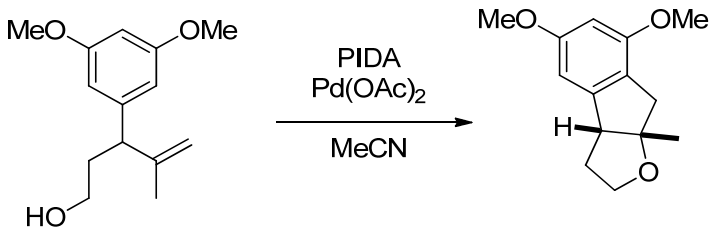
R_f(Hexane/EtOAc4:1): 0.13;

IR (Neat): 3057, 2979, 2931, 1773, 1517, 1384, 1267, 1241, 1178, 1072, 955, 779, 739 cm^{-1};

^{1}HNMR ($CDCl_3$, 500MHz) δ: 7.90 (d, *J* = 8.3 Hz, 1 H), 7.78 (d, *J* = 8.3 Hz, 1 H), 7.66 (d, *J* = 7.8 Hz, 1 H), 7.55 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1 H), 7.49 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 1 H), 7.33 (d, *J* = 8.3 Hz, 1 H), 4.09 (dd, *J* = 9.8, 2.7 Hz, 1 H), 3.61 (d, *J* = 17.6 Hz, 1 H), 3.33 (d, *J* = 17.4 Hz, 1 H), 3.28 (dd, *J* = 18.1, 9.8 Hz, 1 H), 2.94 (d, *J* = 18.2, 2.8 Hz, 1 H), 1.75 (s, 3 H);

^{13}C NMR ($CDCl_3$, 125MHz) δ: 175.9, 137.2, 137.0, 133.4, 129.5, 129.2, 129.2, 126.8, 125.4, 123.0, 123.0, 93.3, 50.1, 46.4, 34.7, 25.9;

HRMS (ESI) *m/z* calculated for $C_{16}H_{15}O_2^+$([M+H]$^+$) 239.1072, found 239.1076.



5,7-dimethoxy-8a-methyl-3,3a,8,8a-tetrahydro-2 H-indeno[2,1-b]furan(22)

According the general procedure, the aryl carboxylic acid (110 mg, 0.48 mmol, 1.0 equiv), phenyliodine diacetate (480 mg, 1.4 mmol, 3.0 equiv) and palladium diacetate (16 mg, 0.048 mmol, 0.10 equiv) in dry acetonitrile (100 mL) afforded the cyclized product **15b** (52 mg, 46%) as a colorless oil after purification by chromatography on SiO_2 (gradient of 90:10 → 80:20 Hexanes/EtOAc) (12 h reaction time).

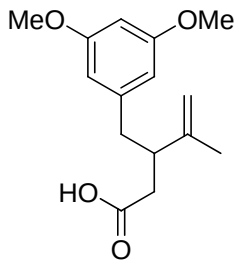
R_f (Hexane/EtOAc4:1): 0.43;

IR (Neat): 2940, 1660, 1596, 1462, 1429, 1344, 1205, 1157, 1059, 833, 744 cm^{-1};

^{1}H NMR ($CDCl_3$, 500MHz)δ: 6.32 (d, *J* = 1.5 Hz, 1 H), 6.29 (d, *J* = 1.9 Hz, 1 H), 3.91 (m, 2 H), 3.79 (s, 3 H), 3.78 (s, 3 H), 3.13 (td, *J* = 9.1, 9.1, 5.9 Hz, 1 H), 3.38, (dd, *J* = 8.6, 2.5 Hz, 2 H), 3.07 (d, *J* = 16.8 Hz, 1 H), 2.88 (d, *J* = 16.8Hz, 1 H), 2.23 (m, 1 H), 2.00 (ddt*J* = 12.1, 6.0, 2.8, 2.8 Hz, 1 H), 1.46 (s, 3 H);

^{13}C NMR ($CDCl_3$. 75MHz) δ: 160.7, 147.5, 145.6, 110.6, 106.1, 98.0, 61.3, 55.2, 49.2, 35.7, 21.1;

HRMS (ESI) not found.


MeO
OMe
HO
O

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6.32
6.32
6.31
6.31
6.30
4.80
4.79
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4.74
3.77
2.86
2.75
2.72
2.70
2.59
2.57
2.54
2.44
2.43
2.42
2.41
2.09
1.74
1.73
3.14
2.20
5.85
0.98
1.01
1.02
1.97
1.18
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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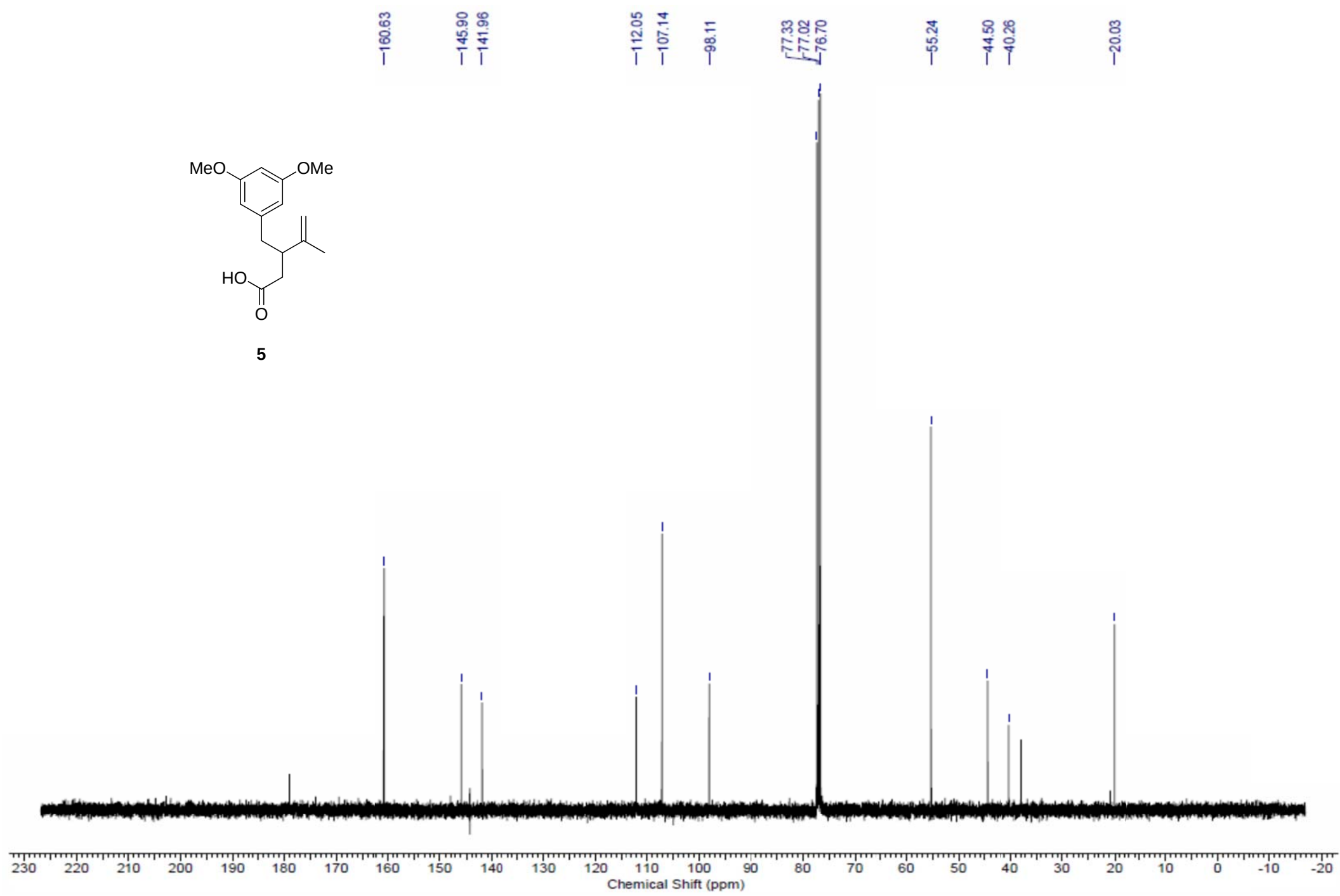
0.5
0
Chemical Shift (ppm)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112.05
107.14
98.11
77.33
77.02
76.70
55.24
44.50
40.26
20.03
MeO
OMe
HO
O
5
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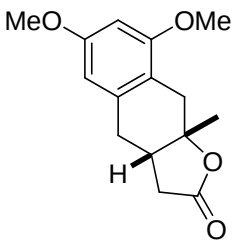
Chemical Shift (ppm)


MeO
OMe
H
O
O

6

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6.33
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2.22
1.50
2.00
5.70
1.07
2.09
1.06
2.00
1.06
2.81
10.0
9.5
9.0
8.5
8.0
7.5
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6.5
6.0
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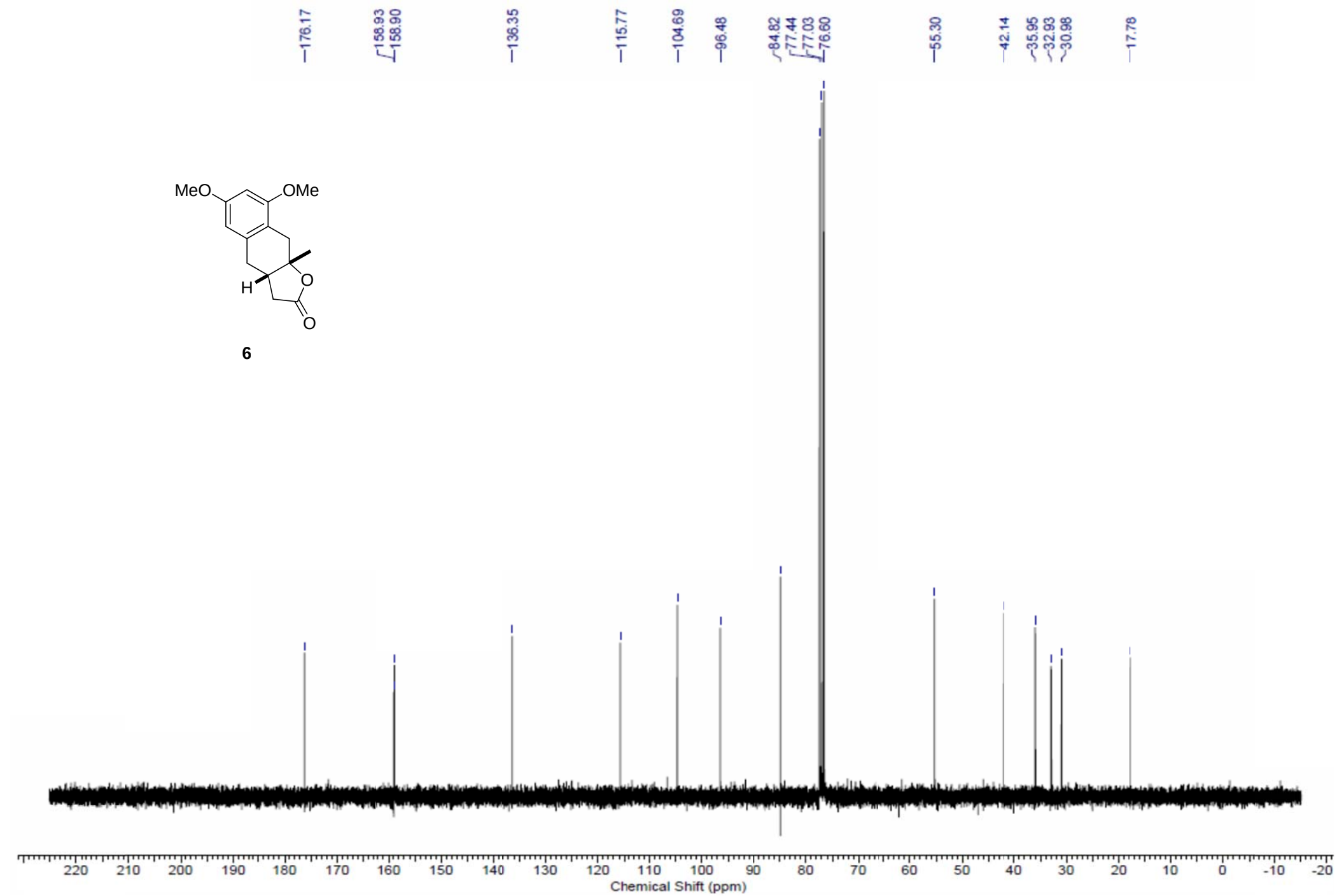
Chemical Shift (ppm)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104.69
96.48
84.82
77.44
77.03
76.60
55.30
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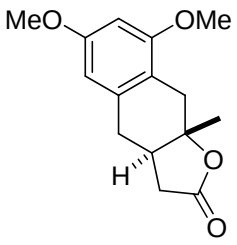
35.95
32.93
30.98
17.78
MeO
OMe
H
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
Chemical Shift (ppm)

6


MeO
OMe
H
O
O

6'

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6.33
6.33
6.26
6.26
3.79
3.78
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3.14
2.98
2.96
2.95
2.72
2.69
2.56
2.55
2.48
2.47
2.45
1.25
1.20
2.19
5.00
1.10
1.14
2.07
3.06
3.04
10.0
9.5
9.0
8.5
8.0
7.5
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6.5
6.0
5.5
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2.0
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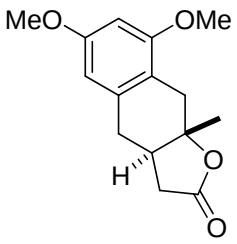
Chemical Shift (ppm)


MeO
OMe
H
O
O

6'

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115.33
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96.88
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77.28
77.02
76.77
55.54
55.35
39.92
34.92
34.30
32.42
28.56
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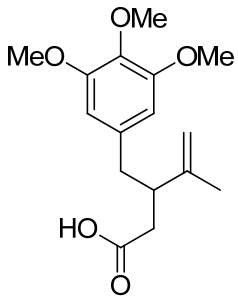
Chemical Shift (ppm)


OMe
MeO
OMe
HO
O

7

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3.82
3.81
2.86
2.84
2.74
2.73
2.71
2.59
2.57
2.56
2.44
2.43
2.42
1.74
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8.98
1.11
1.11
1.14
2.08
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7.5
7.0
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6.0
5.5
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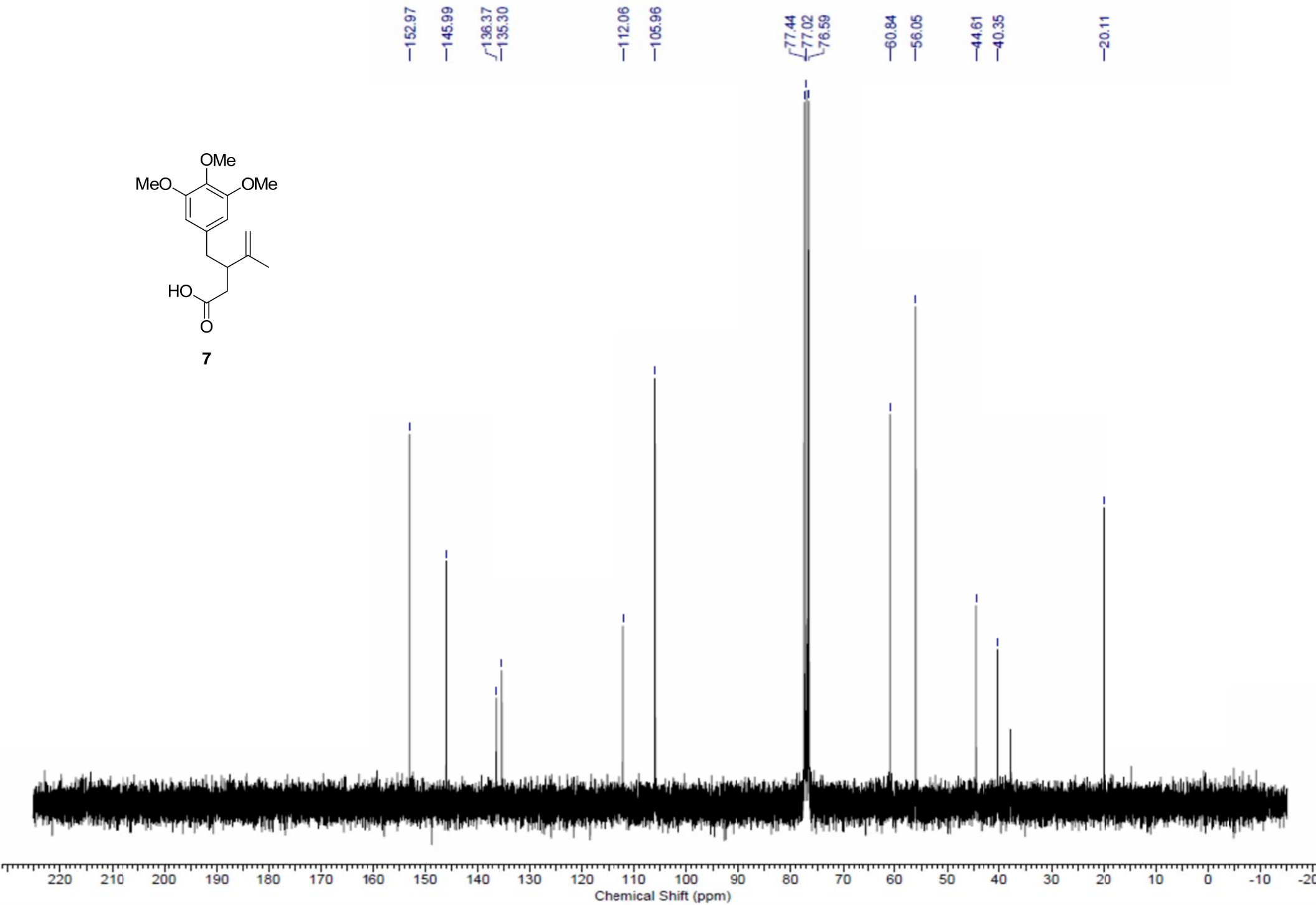
Chemical Shift (ppm)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105.96
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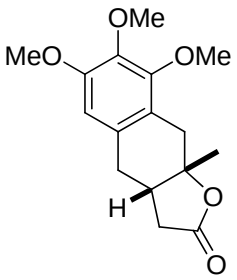
OMe
MeO
OMe
HO
O
Chemical Shift (ppm)

7


OMe
MeO
OMe
H
O
O

8

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3.84
3.84
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3.10
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2.56
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2.22
2.21
2.18
2.17
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1.01
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1.03
1.12
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6.0
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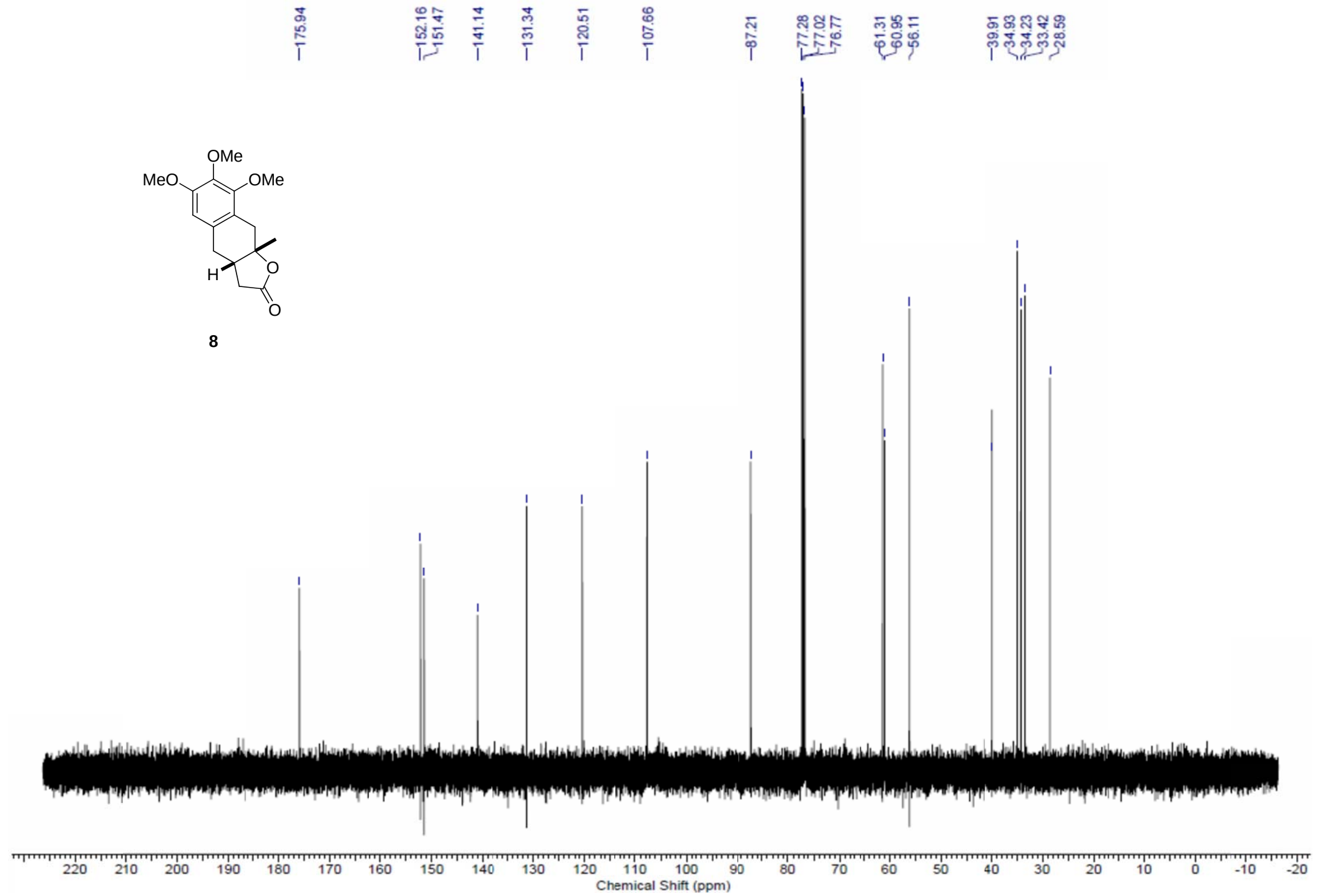
Chemical Shift (ppm)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61.31
60.95
56.11
39.91
34.93
34.23
33.42
28.59
OMe
MeO
OMe
H
O
O
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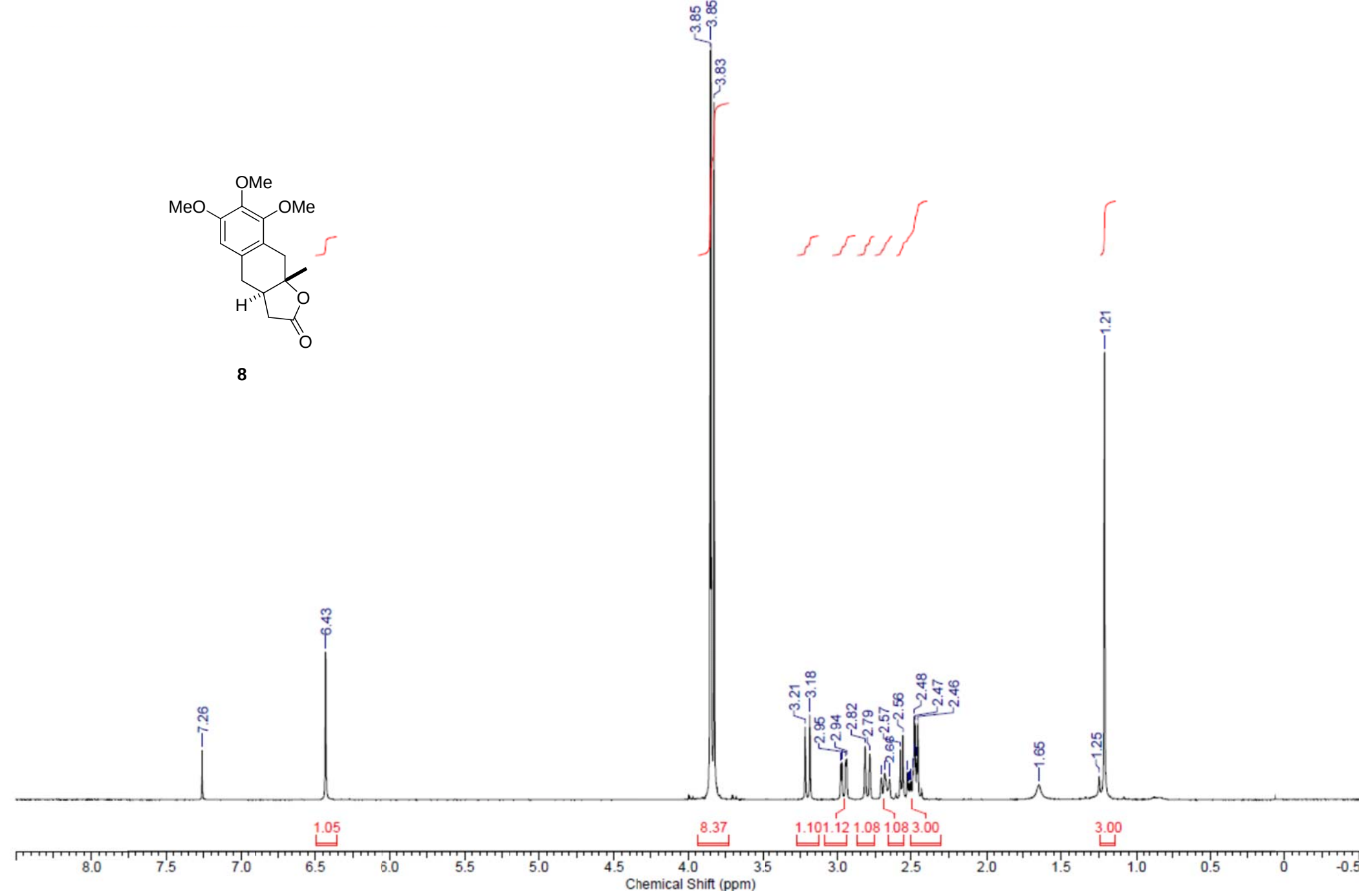
Chemical Shift (ppm)

8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3.21
3.18
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2.47
2.46
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1.08
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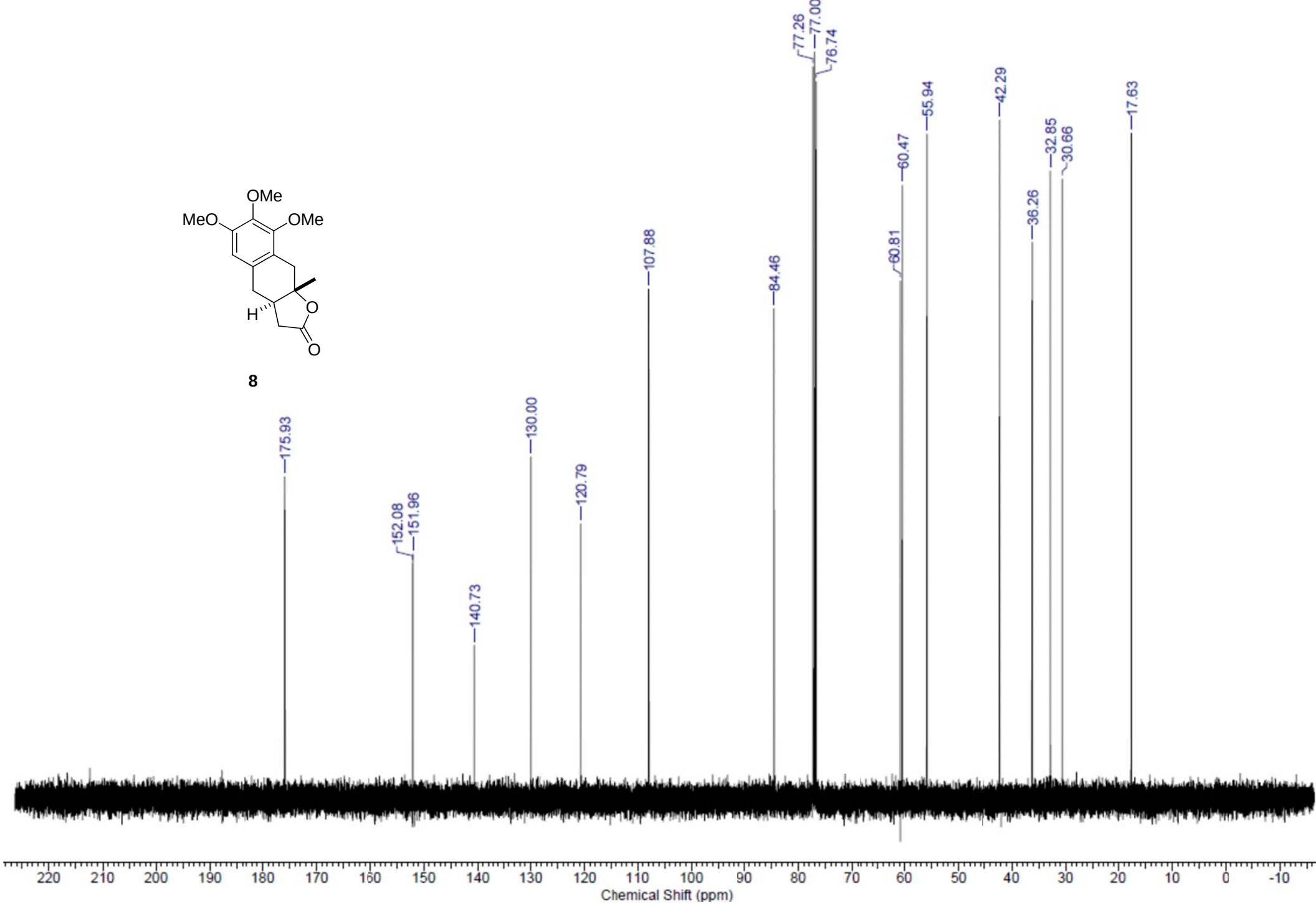
3.00
Chemical Shift (ppm)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107.88
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60.47
55.94
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30.66
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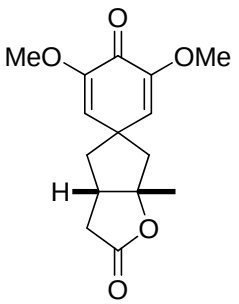
Chemical Shift (ppm)


MeO
OMe
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O
O

8a

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1.60
1.84
5.71
1.10
1.07
2.01
1.08
1.11
1.26
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6.0
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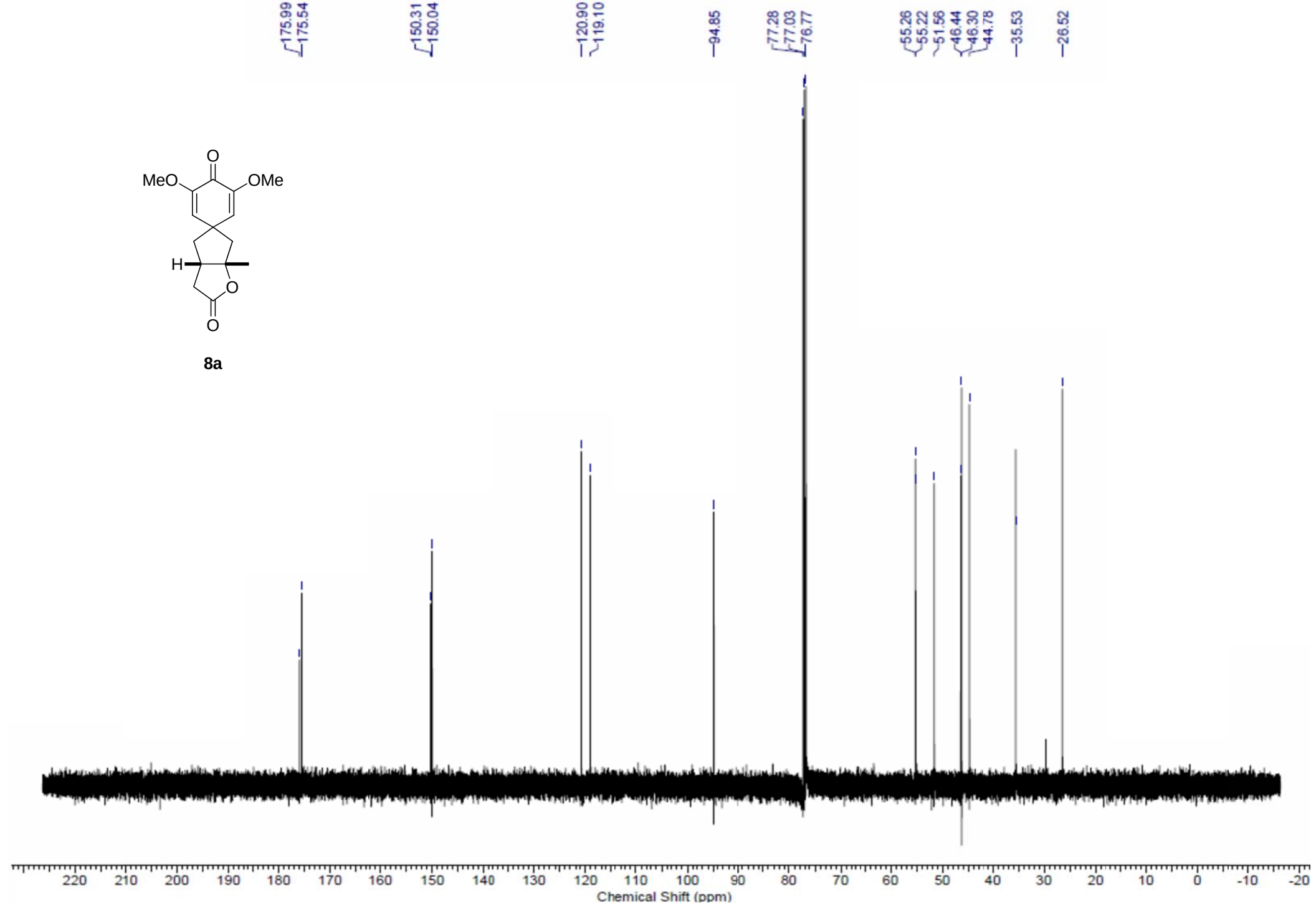
Chemical Shift (ppm)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119.10
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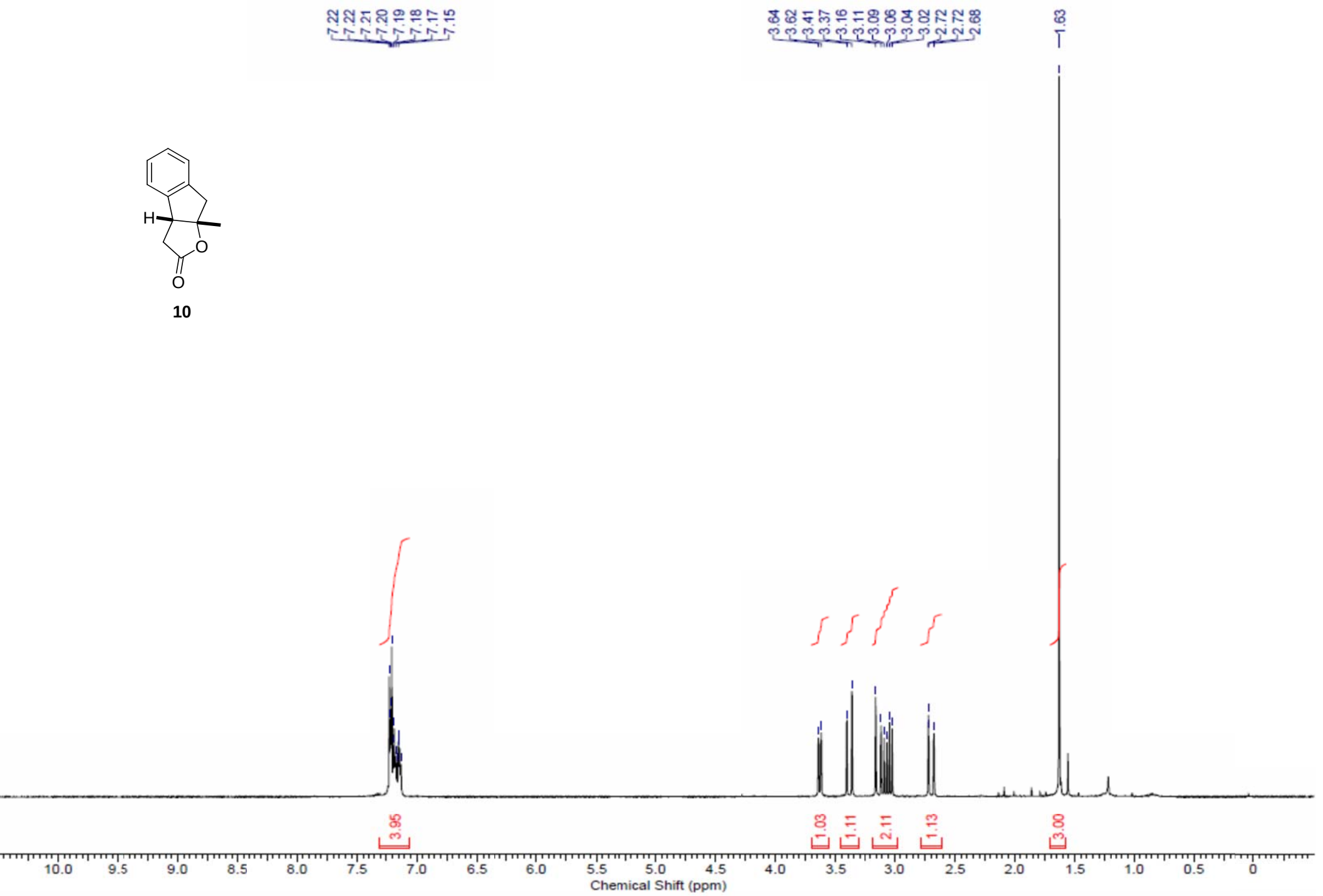
77.28
77.03
76.77
55.26
55.22
51.56
46.44
46.30
44.78
35.53
26.52
MeO
OMe
H
O
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
Chemical Shift (ppm)

8a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2.68
1.63
H
O
O
3.95
1.03
1.11
2.11
1.13
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9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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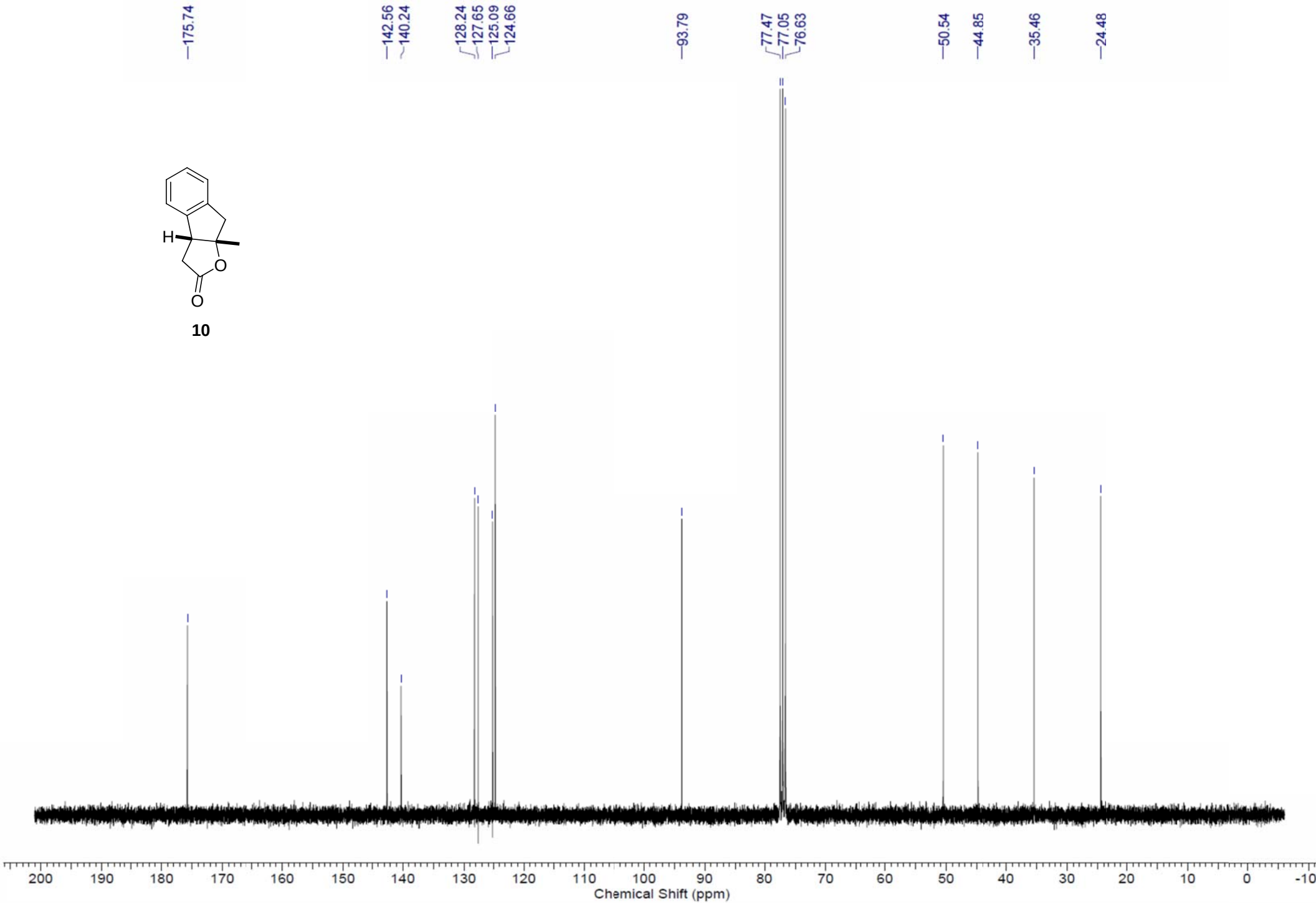
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

10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125.09
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77.05
76.63
50.54
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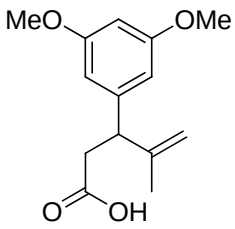
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)

10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OMe
O
OH

11

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6.34
6.33
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4.91
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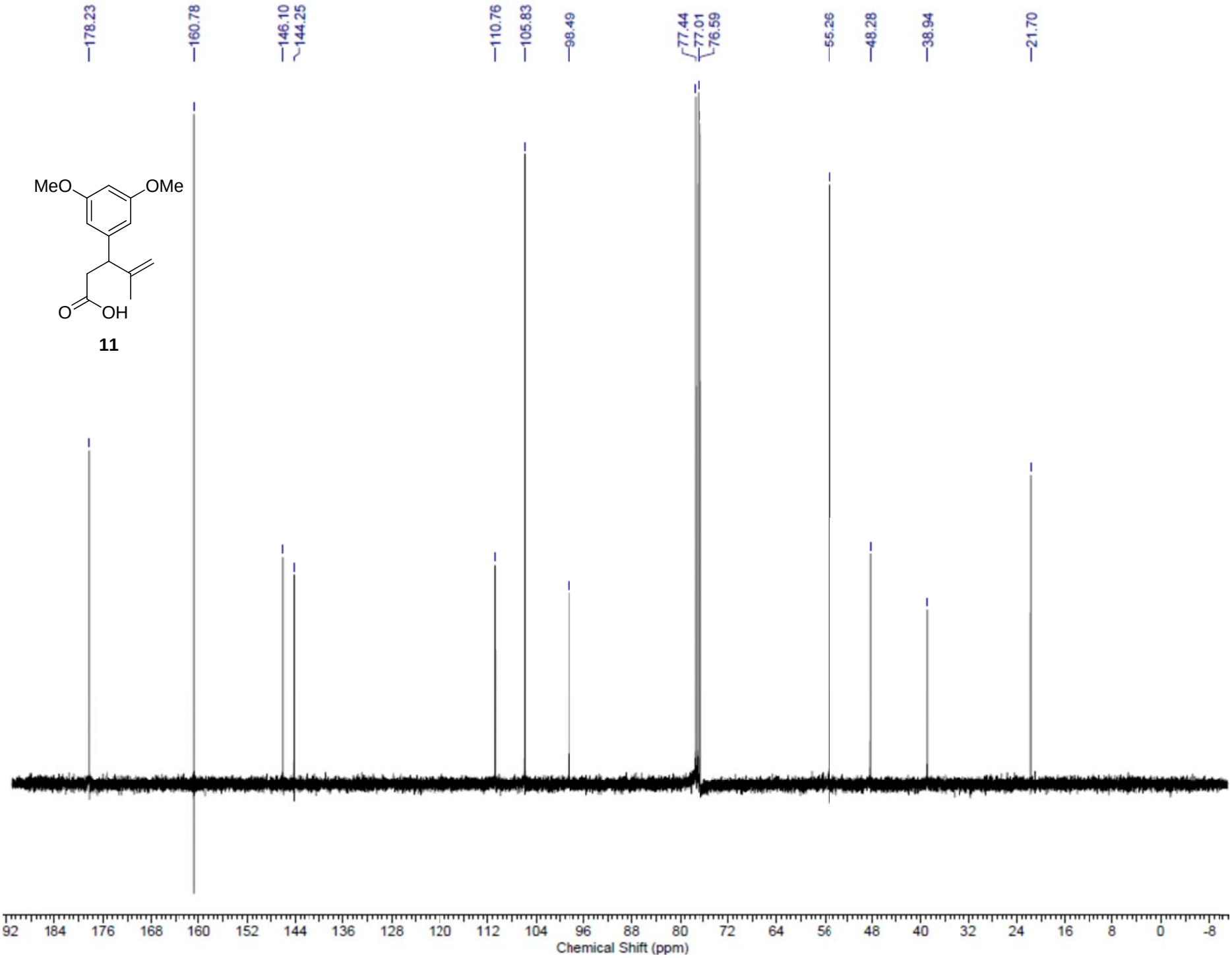
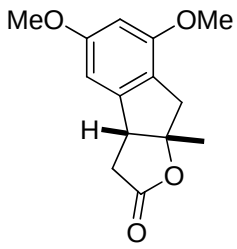
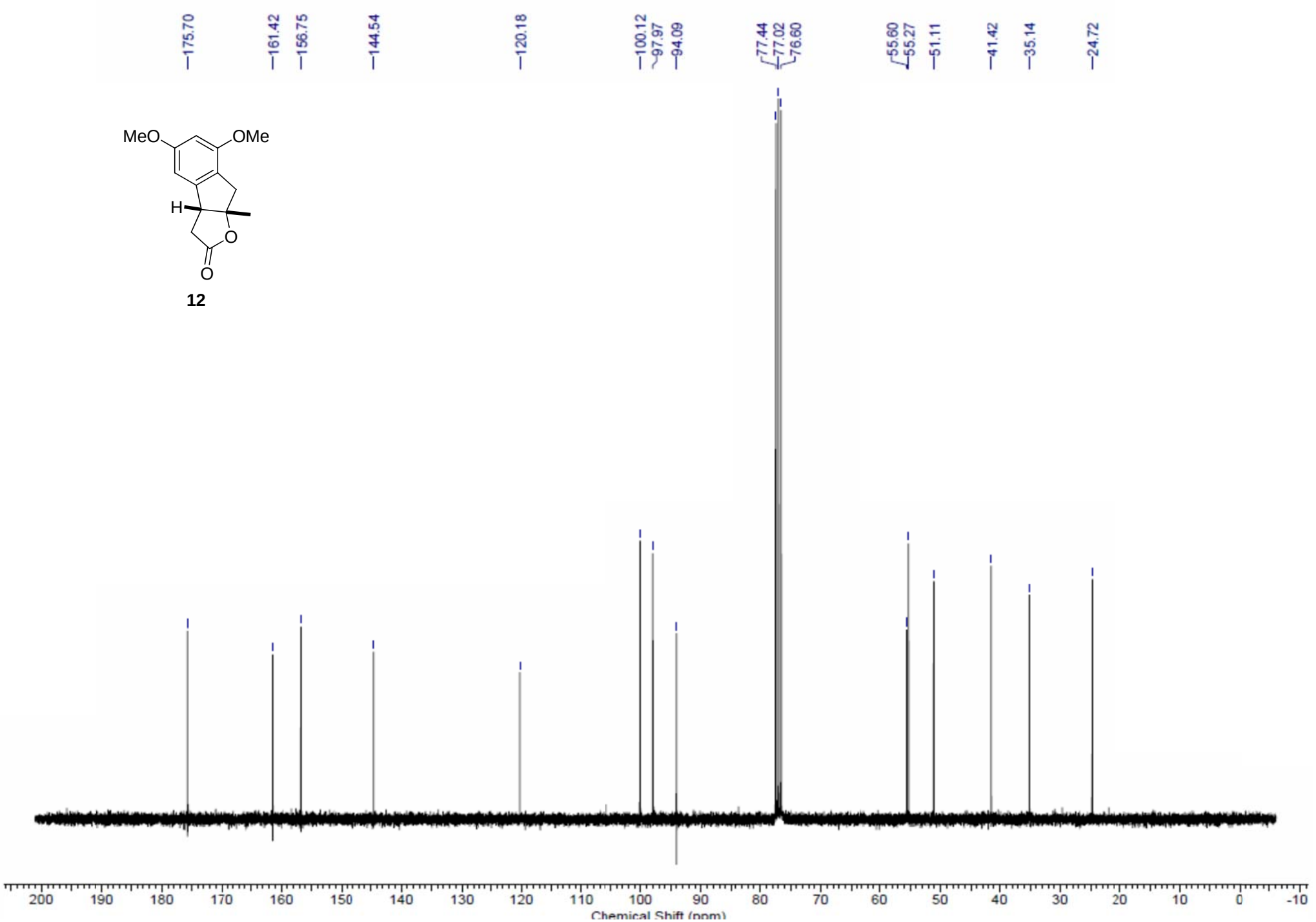
3.75
3.72
3.70
2.93
2.90
2.87
2.85
2.78
2.76
2.73
2.71
1.66
2.11
1.03
2.28
6.11
1.19
1.28
1.25
3.14
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

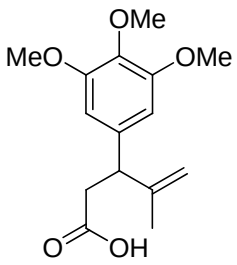

178.23
160.78
146.10
144.25
110.76
105.83
98.49
77.44
77.01
76.59
55.26
48.28
38.94
21.70
MeO
OMe
O
OH
11
92 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0 -8
Chemical Shift (ppm)


MeO
OMe
H
O
O

12

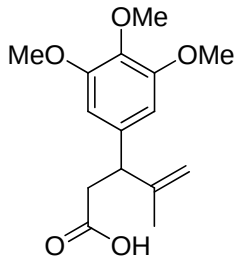
6.33
6.29
3.83
3.80
3.79
3.34
3.31
3.07
3.05
3.03
3.01
2.98
2.95
2.74
2.73
2.70
1.64
1.00
0.99
6.03
0.96
1.17
1.17
1.15
1.16
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
-0.5
Chemical Shift (ppm)


175.70
161.42
156.75
144.54
120.18
100.12
97.97
94.09
77.44
77.02
76.60
55.60
55.27
51.11
41.42
35.14
24.72
MeO
OMe
H
O
O
12
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)


OMe
MeO
OMe
O
OH

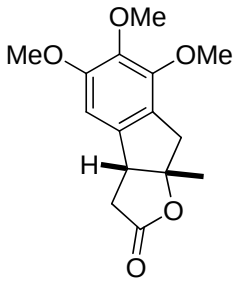
13

7.26
6.43
4.92
3.84
3.83
3.83
3.72
3.70
3.69
2.90
2.88
2.87
2.85
2.75
2.73
2.72
1.65
2.07
2.08
8.49
1.18
1.16
1.16
2.87
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
-0.5
Chemical Shift (ppm)


OMe
MeO
OMe
O
OH

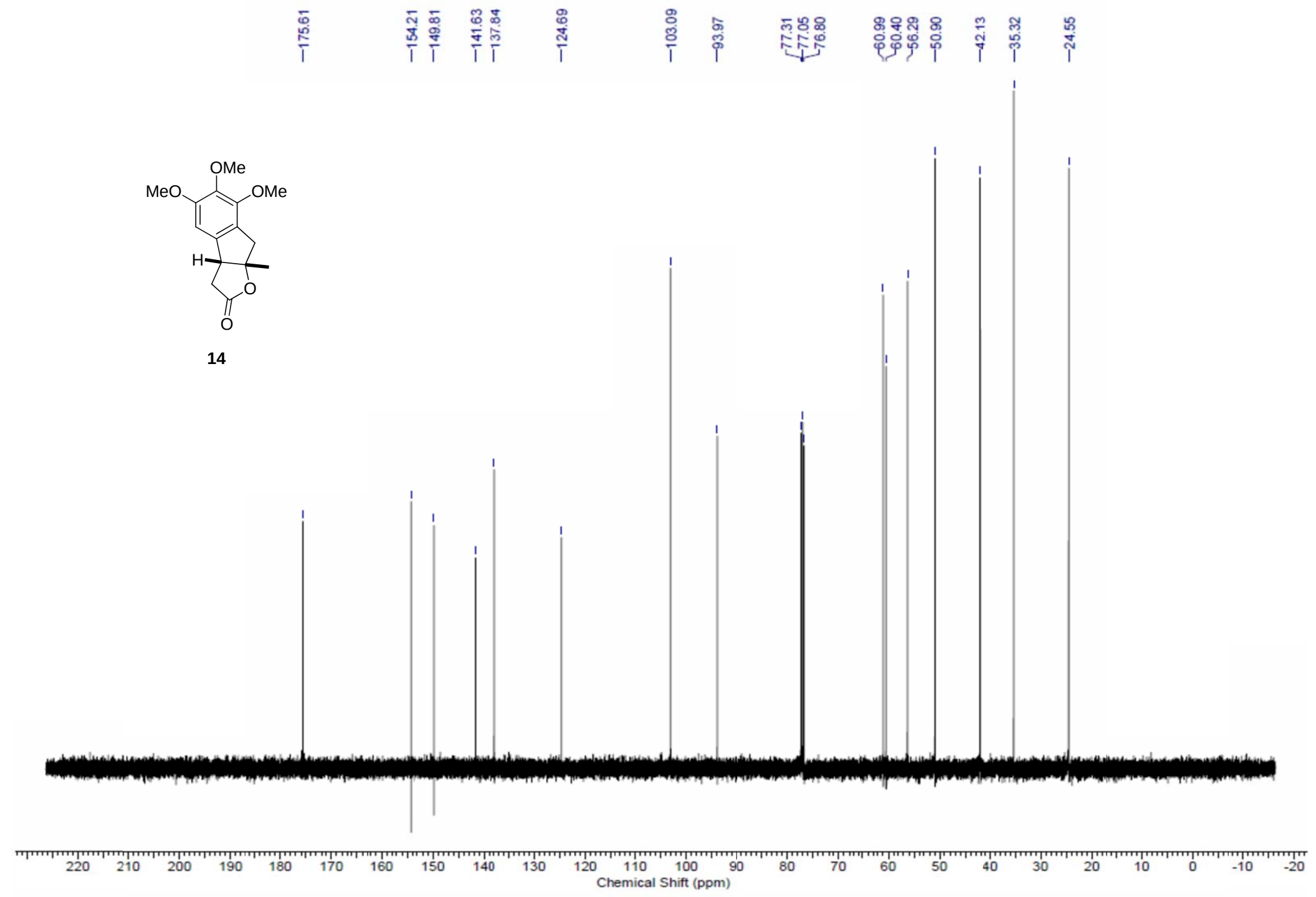
13

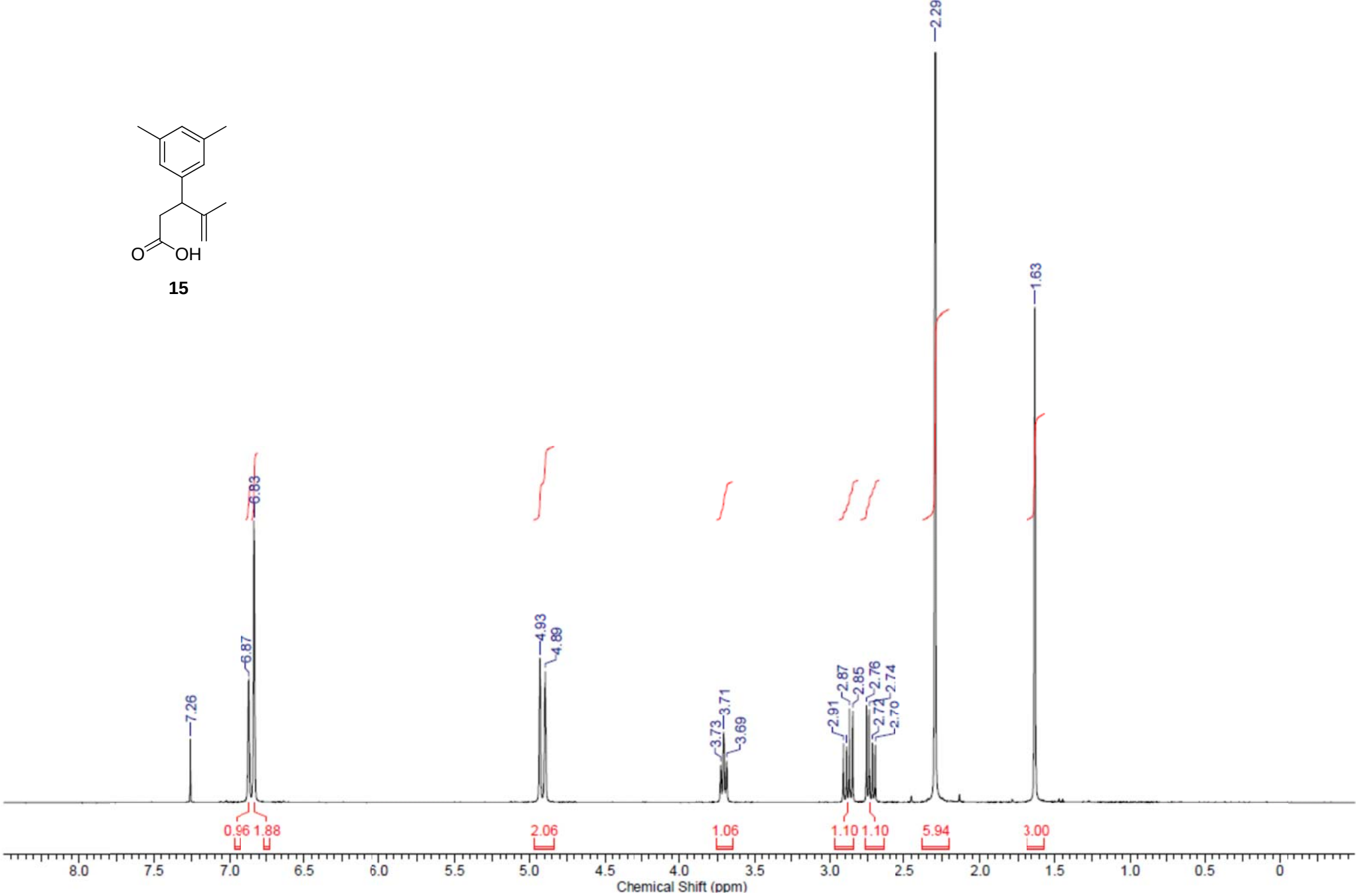
177.99
153.07
146.27
137.40
136.65
110.55
104.58
77.25
77.00
76.74
60.73
56.00
48.33
39.20
21.77
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
Chemical Shift (ppm)

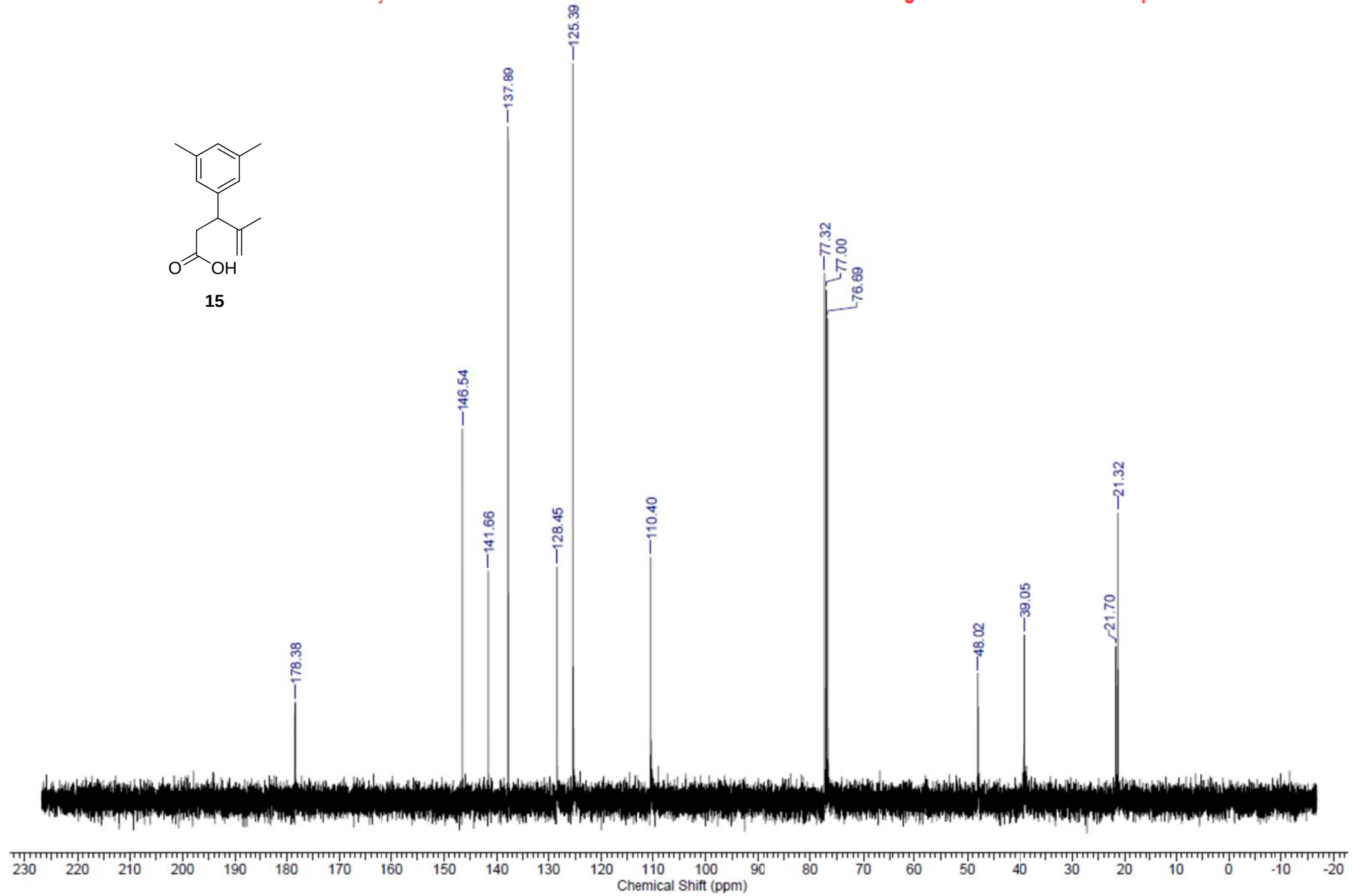

OMe
MeO
OMe
H
O
O

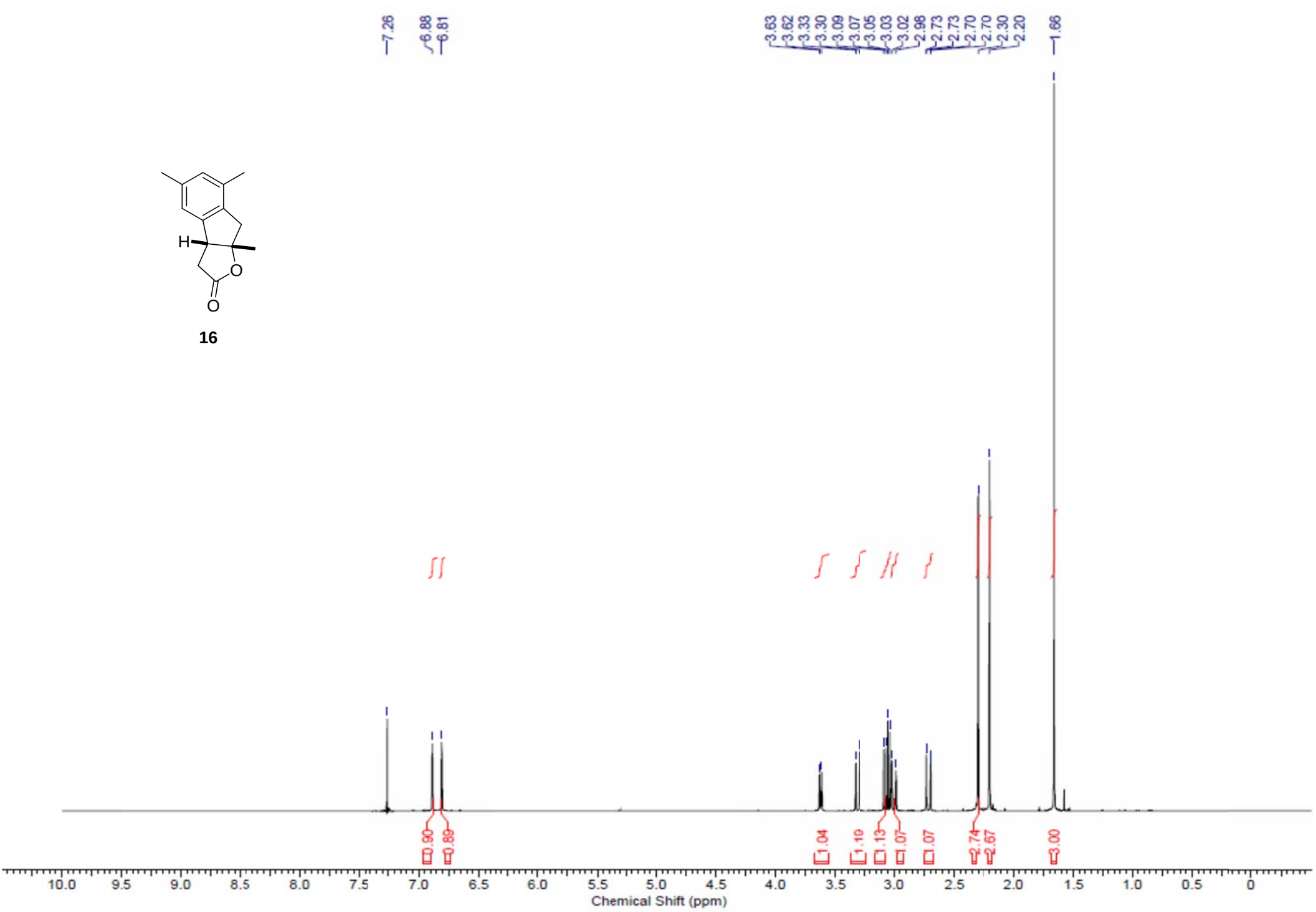
14

6.45
3.89
3.88
3.88
3.84
3.84
3.59
3.42
3.39
3.08
3.07
3.05
3.04
3.02
2.73
2.70
1.66
1.28
9.00
1.06
1.15
2.26
1.22
3.26
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
Chemical Shift (ppm)

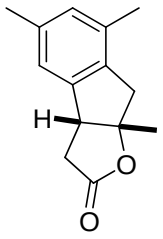

175.61
154.21
149.81
141.63
137.84
124.69
103.09
93.97
77.31
77.05
76.80
60.99
60.40
56.29
50.90
42.13
35.32
24.55
OMe
MeO
OMe
H
O
O
14
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
Chemical Shift (ppm)


O
OH
15
7.26
6.87
6.83
4.93
4.89
3.73
3.71
3.69
2.91
2.87
2.85
2.76
2.74
2.71
2.70
2.29
1.63
0.96
1.88
2.06
1.06
1.10
1.10
5.94
3.00
Chemical Shift (ppm)


O
OH
15
178.38
146.54
141.66
137.89
128.45
125.39
110.40
77.32
77.00
76.69
48.02
39.05
21.70
21.32
Chemical Shift (ppm)

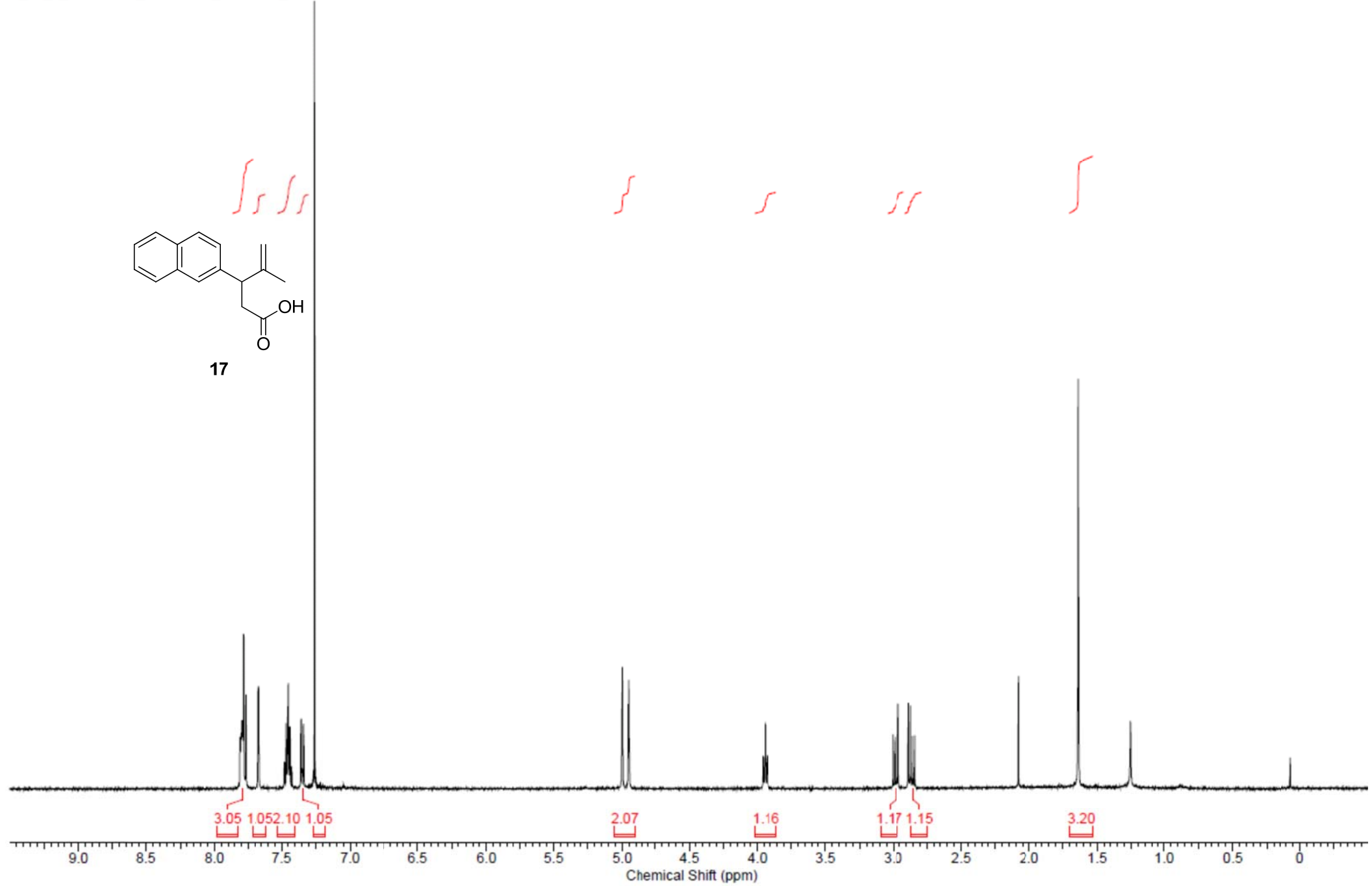

7.26
6.88
6.81
3.63
3.62
3.33
3.30
3.09
3.07
3.05
3.03
3.02
2.98
2.73
2.73
2.70
2.70
2.30
2.20
1.66
H
O
O
0.90
0.89
1.04
1.19
1.13
1.07
1.07
2.74
2.67
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

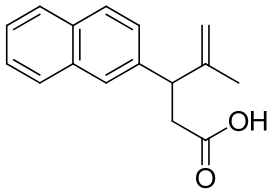
16


H
O
O

16

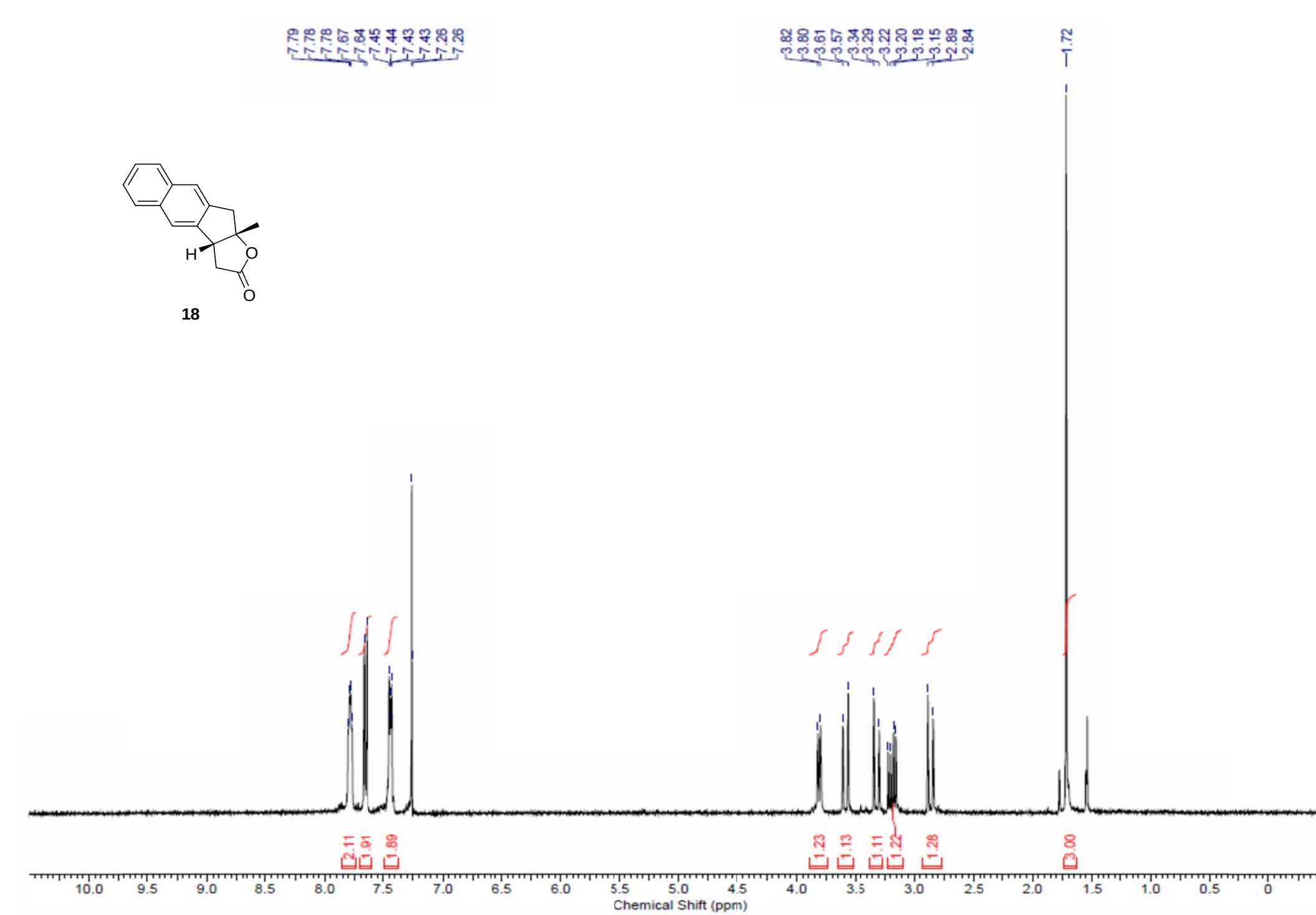
175.90
142.48
137.68
136.07
134.24
129.89
122.41
93.78
77.44
77.01
76.59
50.65
43.43
35.50
24.70
21.19
18.94
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)


OH
O
17
3.05
1.05
2.10
1.05
2.07
1.16
1.17
1.15
3.20
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
Chemical Shift (ppm)

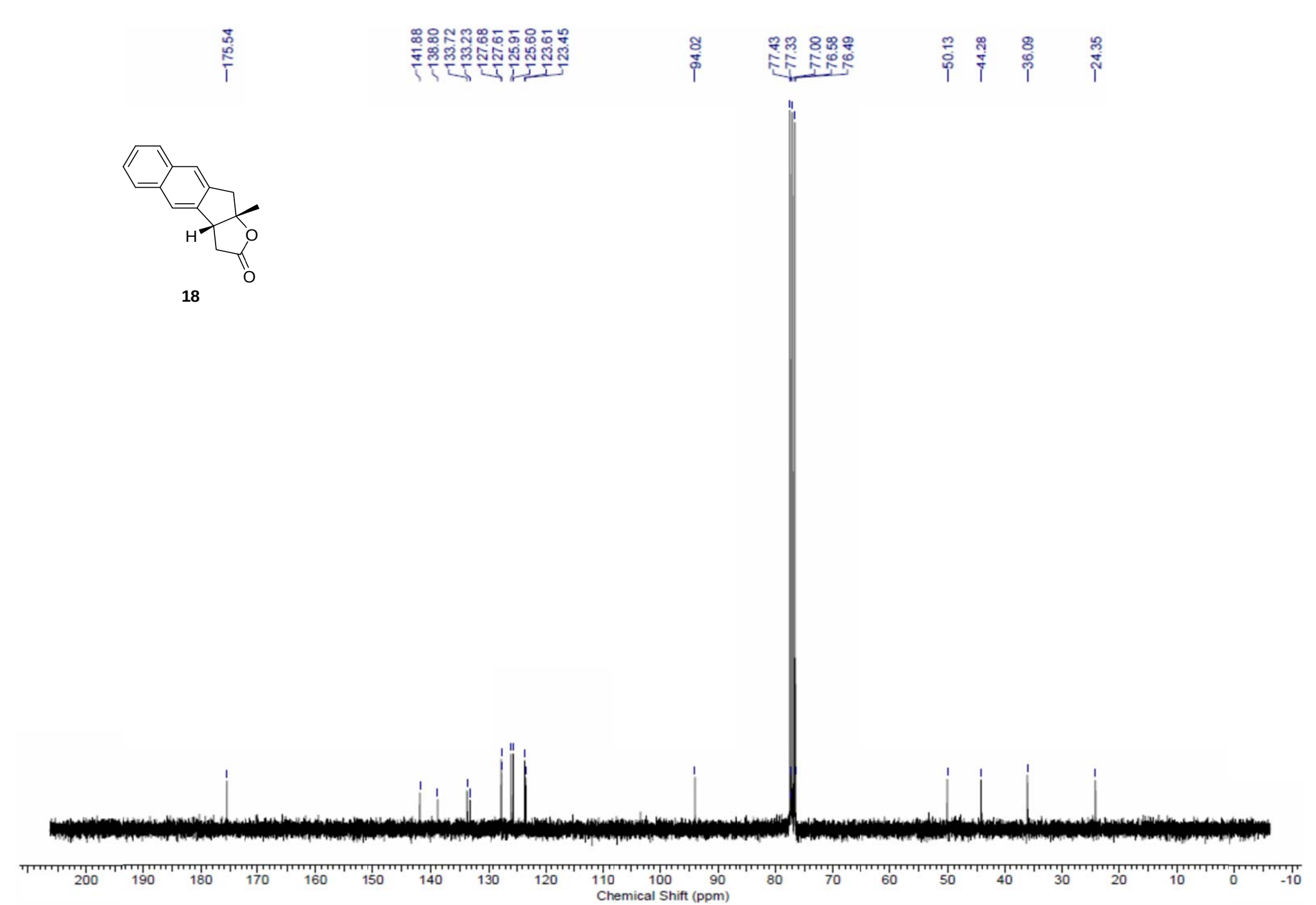

OH
O

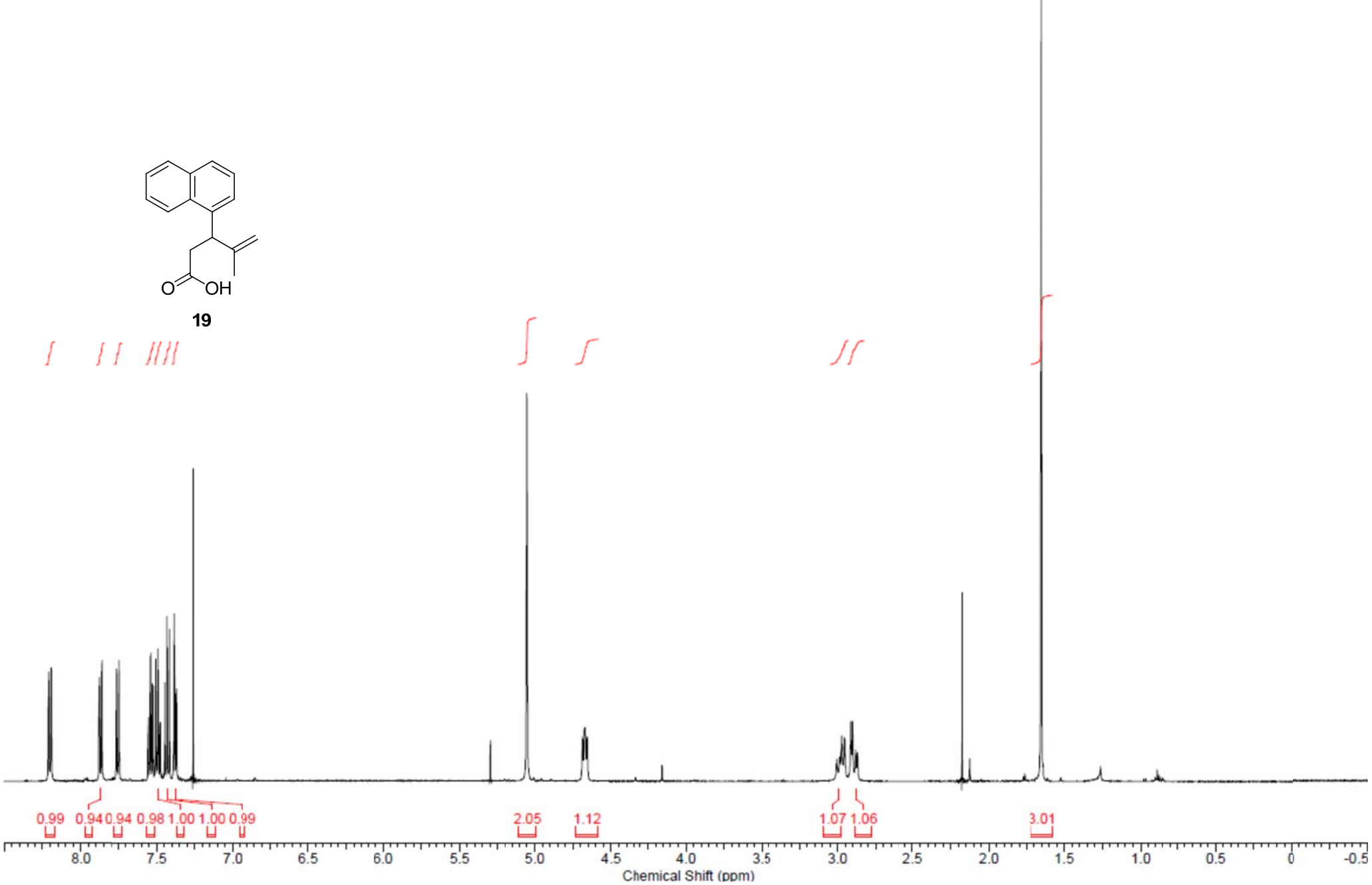
17

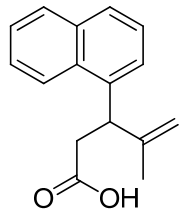
77.2
77.0
76.7
176.66
146.26
139.15
133.41
132.48
128.21
127.58
126.18
125.95
125.61
110.90
106.24
48.29
38.67
21.69
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
Chemical Shift (ppm)


7.79
7.78
7.78
7.67
7.64
7.45
7.44
7.43
7.43
7.26
7.26
3.82
3.80
3.61
3.57
3.34
3.29
3.22
3.20
3.18
3.15
2.89
2.84
1.72
H
O
O
2.11
1.91
1.89
1.23
1.13
1.11
1.22
1.28
3.00
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
Chemical Shift (ppm)

18

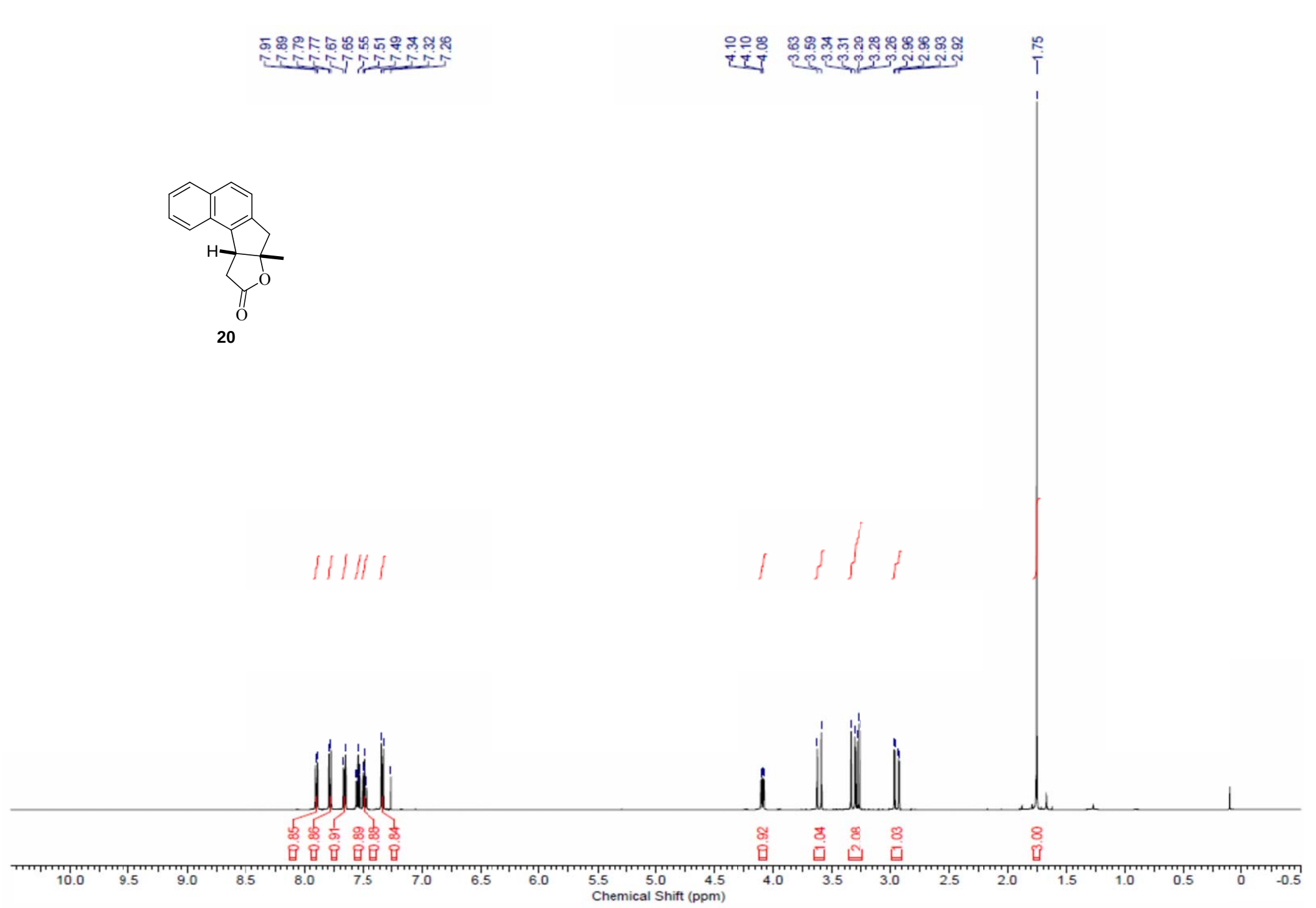

175.54
141.88
138.80
133.72
133.23
127.68
127.61
125.91
125.60
123.61
123.45
94.02
77.43
77.33
77.00
76.58
76.49
50.13
44.28
36.09
24.35
H
O
O
18
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)

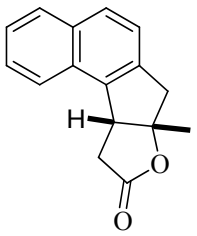

O
OH
19
0.99
0.94
0.94
0.98
1.00
1.00
0.99
2.05
1.12
1.07
1.06
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
-0.5
Chemical Shift (ppm)


O
OH

19

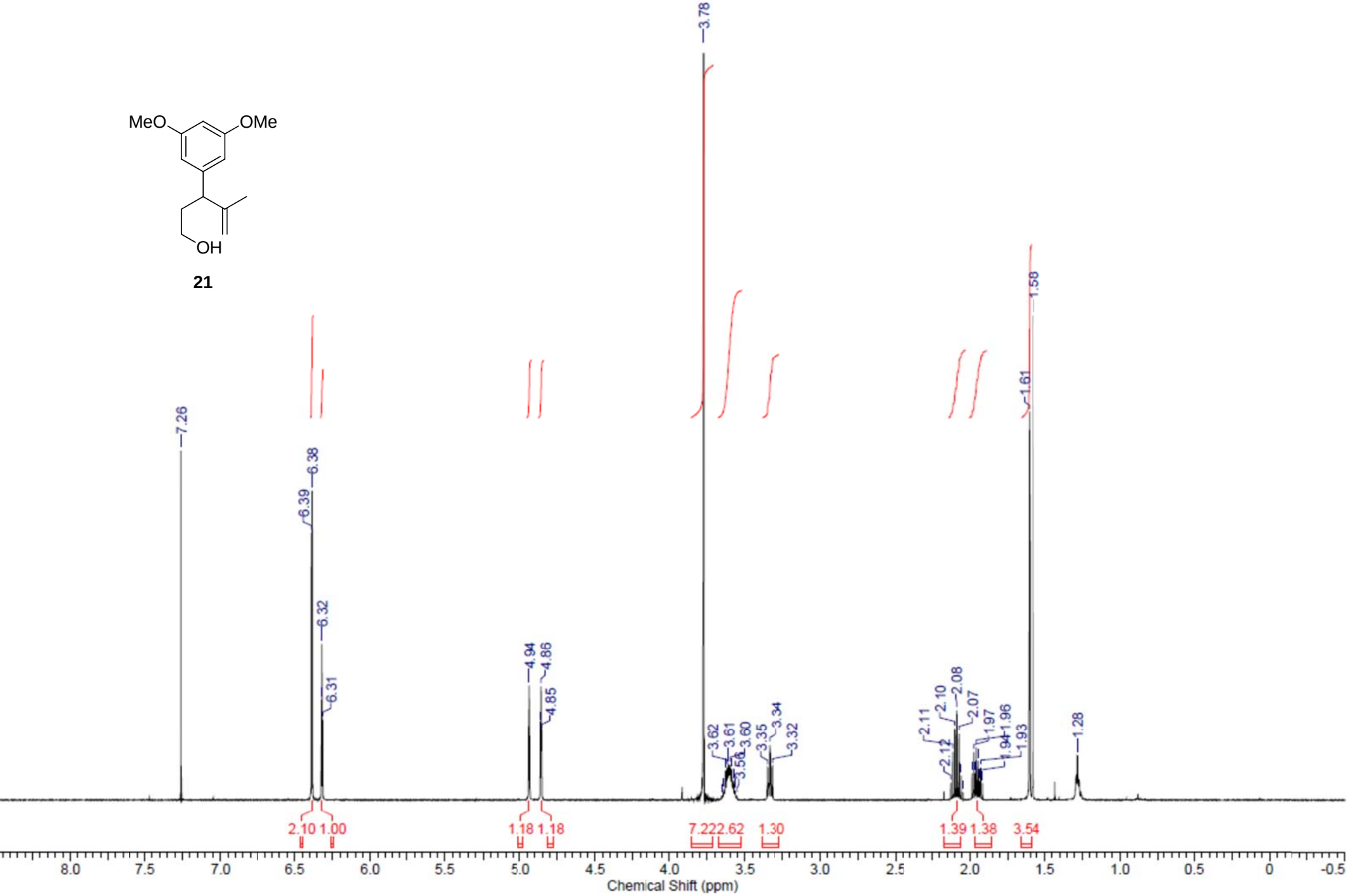
77.26
77.01
76.75
145.98
137.56
134.05
131.68
128.95
127.48
125.57
125.44
123.13
111.85
43.31
38.98
22.06
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
Chemical Shift (ppm)

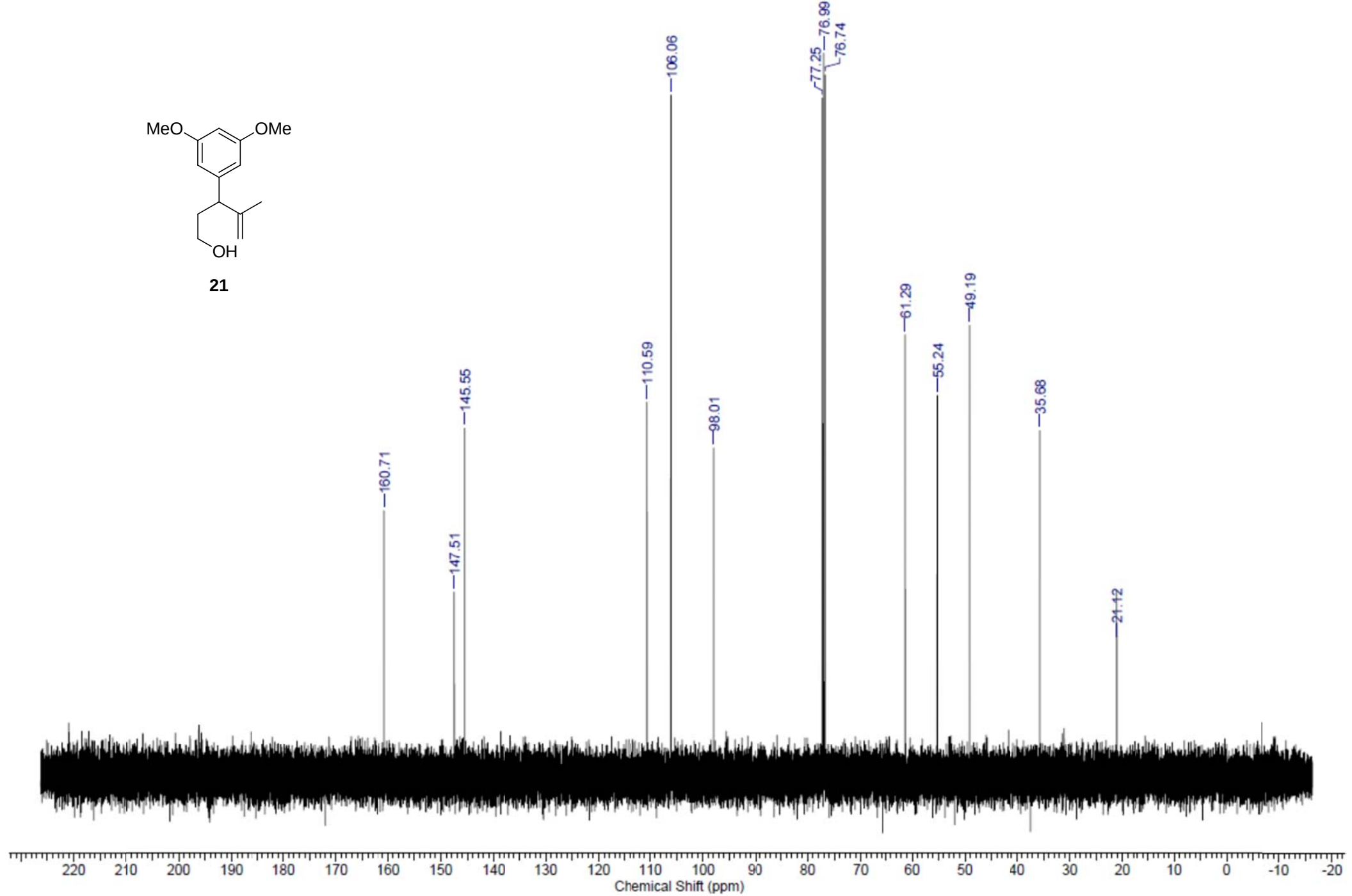

7.91
7.89
7.79
7.77
7.67
7.65
7.55
7.51
7.49
7.34
7.32
7.26
4.10
4.10
4.08
3.63
3.59
3.34
3.31
3.29
3.28
3.26
2.96
2.96
2.93
2.92
1.75
H
O
O
O
20
0.85
0.86
0.91
0.89
0.85
0.84
0.92
1.04
2.08
1.03
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
-0.5
Chemical Shift (ppm)


H
O
O

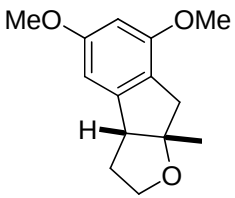
20

77.25
77.00
76.75
175.85
137.19
137.01
133.38
129.16
123.03
122.95
93.30
50.11
46.43
34.73
25.87
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)


MeO
OMe
OH
21
7.26
6.39
6.38
6.32
6.31
4.94
4.86
4.85
3.78
3.62
3.61
3.60
3.56
3.35
3.34
3.32
2.11
2.10
2.12
2.08
2.07
1.97
1.96
1.94
1.93
1.61
1.58
1.28
2.10 1.00
1.18 1.18
7.22 2.62
1.30
1.39 1.38
3.54
Chemical Shift (ppm)

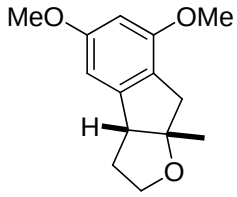

MeO
OMe
OH
160.71
147.51
145.55
110.59
106.06
98.01
77.25
76.99
76.74
61.29
55.24
49.19
35.68
21.12
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
Chemical Shift (ppm)

21


MeO
OMe
H
O

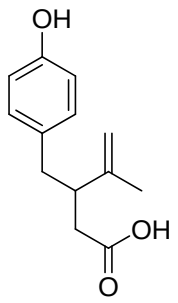
22

7.26
6.33
6.32
6.30
6.29
3.94
3.93
3.92
3.79
3.78
3.60
3.59
3.58
3.39
3.38
3.38
3.09
3.05
2.90
2.87
2.33
2.32
2.31
2.31
2.31
2.29
2.29
2.02
2.01
2.01
2.00
1.46
1.80
1.32
5.53
1.10
0.96
1.05
1.12
1.24
1.12
3.30
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
Chemical Shift (ppm)

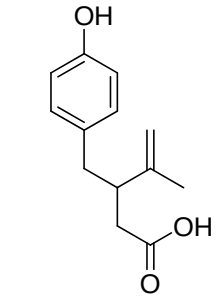

MeO
OMe
H
O

22

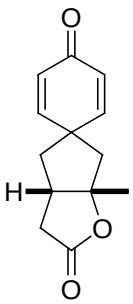
160.62
147.43
145.53
110.52
105.99
97.91
77.43
77.00
76.57
61.10
55.16
49.05
35.60
21.06
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
-8
-16
Chemical Shift (ppm)



SM for Compound 3

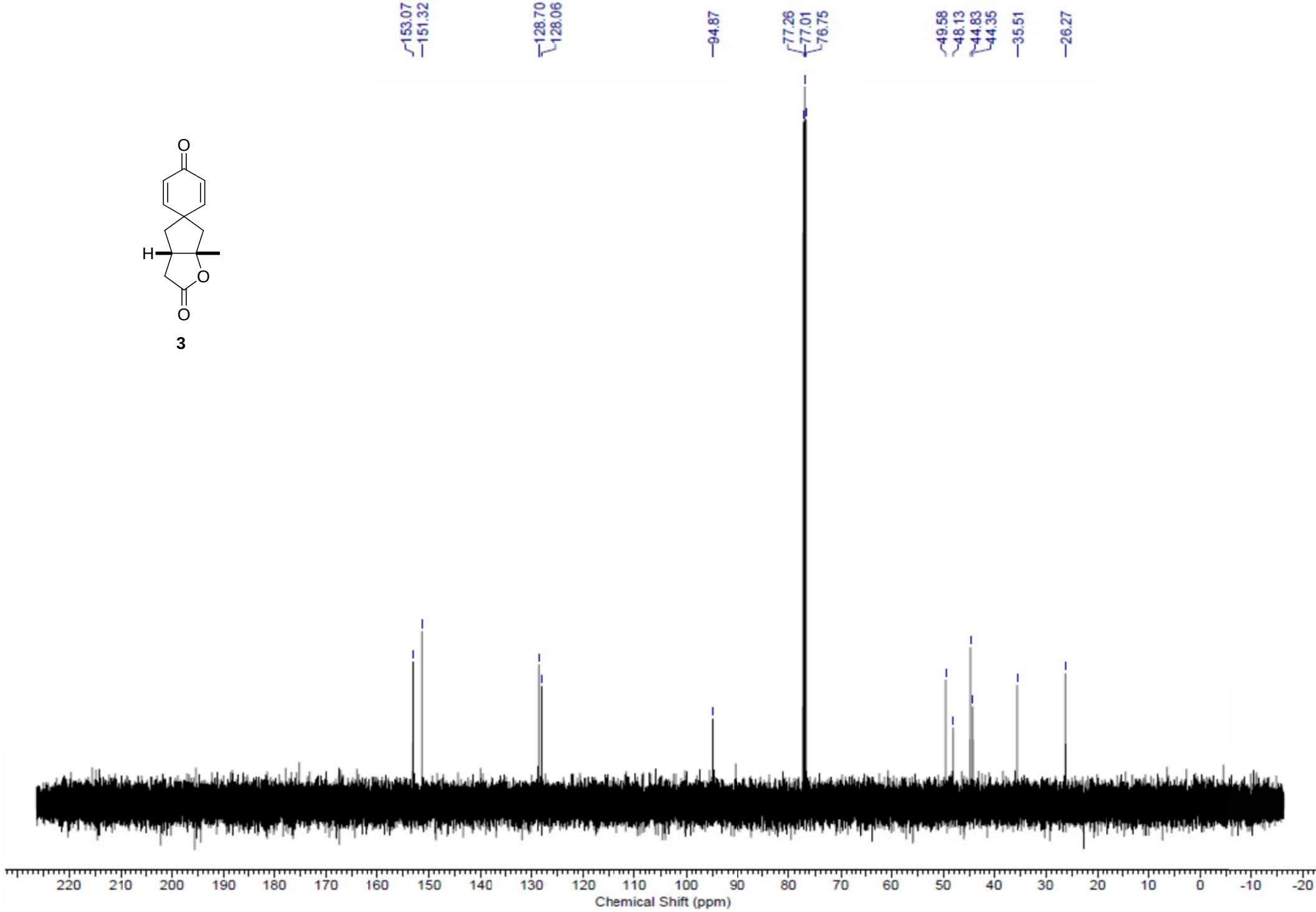


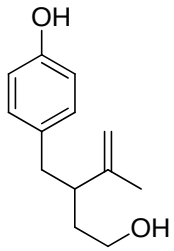
SM for Compound 3



3

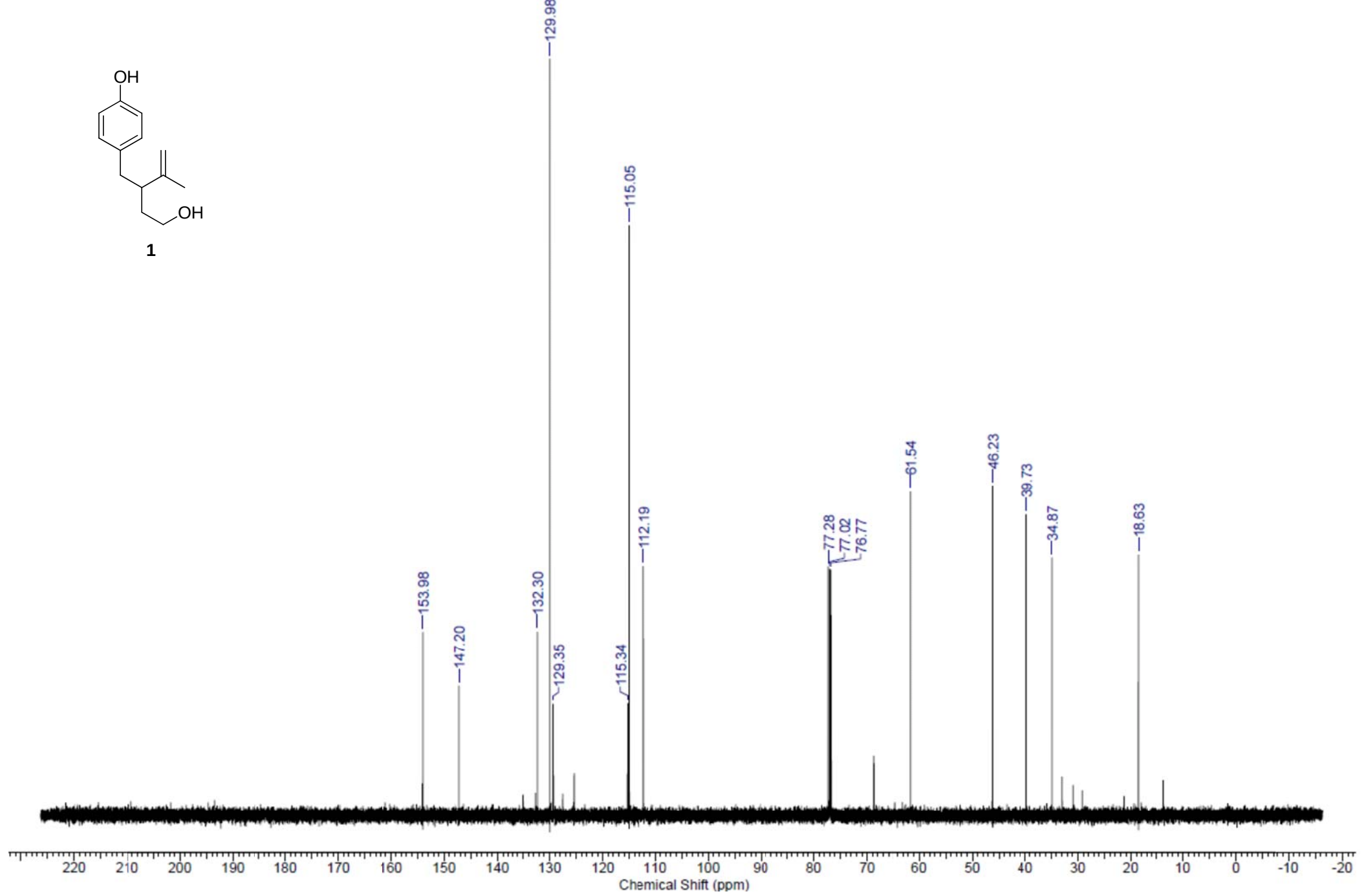
7.19
6.80
6.79
6.78
6.77
6.77
6.75
6.20
6.20
6.19
6.19
6.18
6.17
2.90
2.89
2.87
2.85
2.80
2.78
2.44
2.43
2.40
2.40
2.03
2.00
1.82
1.80
1.80
1.78
1.52
2.20
1.98
0.92
0.85
1.01
0.90
0.97
1.08
0.91
4.20
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
-0.5
Chemical Shift (ppm)

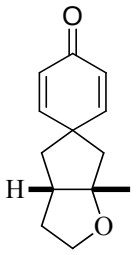

153.07
151.32
128.70
128.06
94.87
77.26
77.01
76.75
49.58
48.13
44.83
44.35
35.51
26.27
O
H
O
O
3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
Chemical Shift (ppm)


OH
OH

1

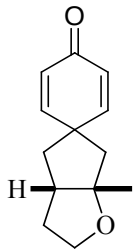
0.05
0.05
0.08
0.06
0.06
0.03
0.07
0.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)


OH
OH
1
129.98
115.05
153.98
147.20
132.30
129.35
115.34
112.19
77.28
77.02
76.77
61.54
46.23
39.73
34.87
18.63
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
Chemical Shift (ppm)


O
H
O

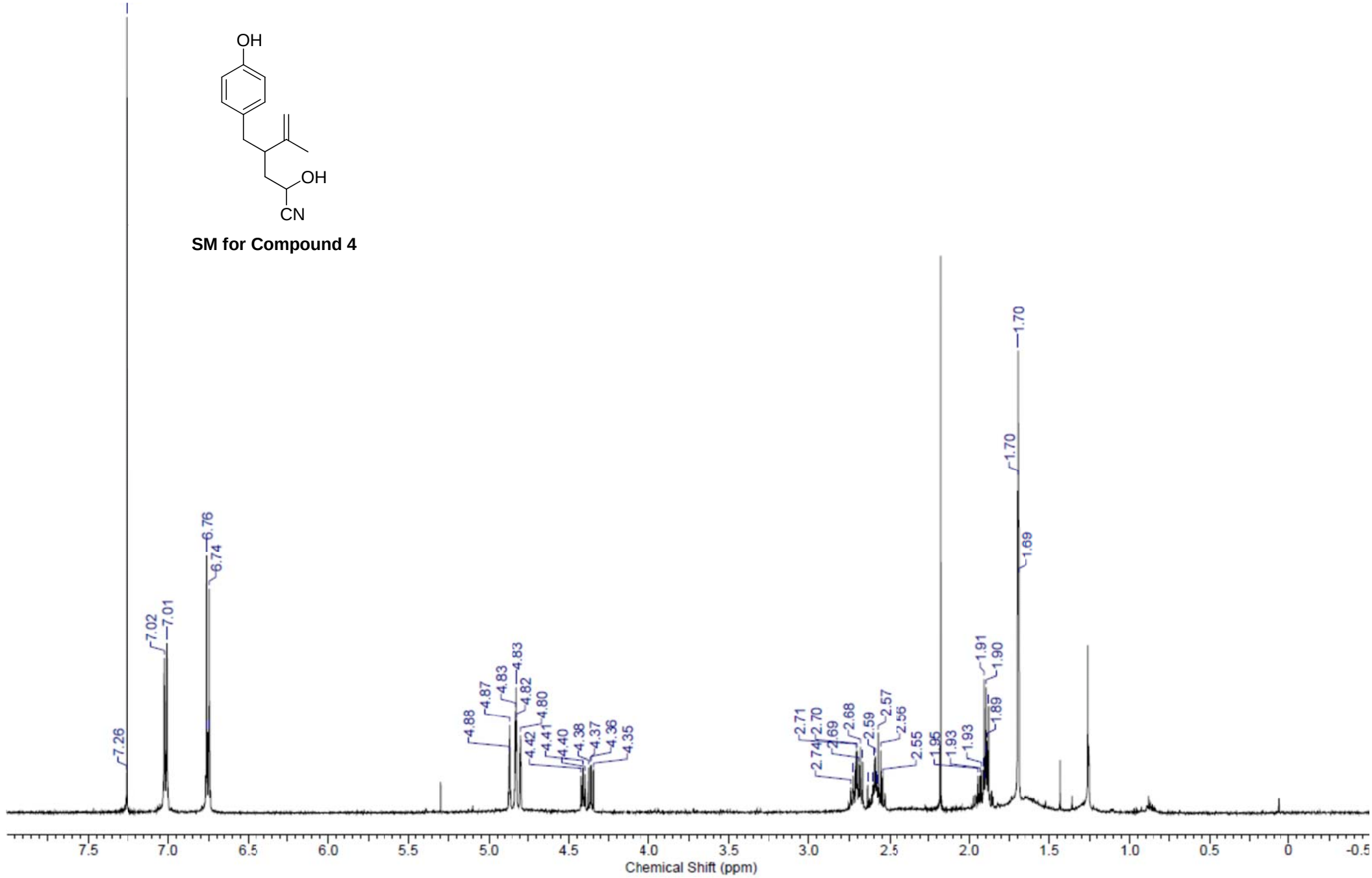
2

7.26
7.04
7.02
7.01
6.93
6.93
6.92
6.91
6.22
6.21
6.20
6.19
6.19
6.19
6.18
3.99
3.99
3.97
3.97
3.96
3.95
3.95
2.67
2.65
2.64
2.19
2.16
2.04
2.03
1.92
1.92
1.90
1.76
1.76
1.75
1.74
1.40
0.91
0.93
1.56
1.95
0.96
2.10
1.27
1.06
2.26
3.00
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
Chemical Shift (ppm)

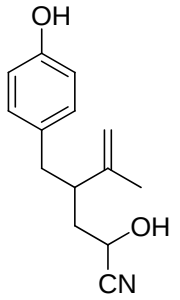


2

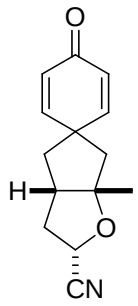
153.07
151.32
128.70
128.06
94.87
77.26
77.01
76.75
49.58
48.13
44.83
44.35
35.51
26.27
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
Chemical Shift (ppm)



SM for Compound 4

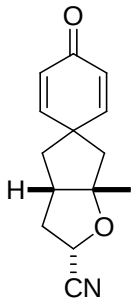


SM for Compound 4


O
H
O
CN

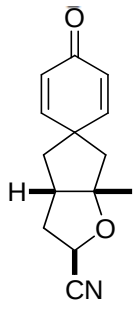
4

0.99 0.93
0.83 0.86
1.06
1.06 1.09 1.02 1.05 1.13 1.09 1.15 2.93
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 -0.5
Chemical Shift (ppm)


O
H
O
CN

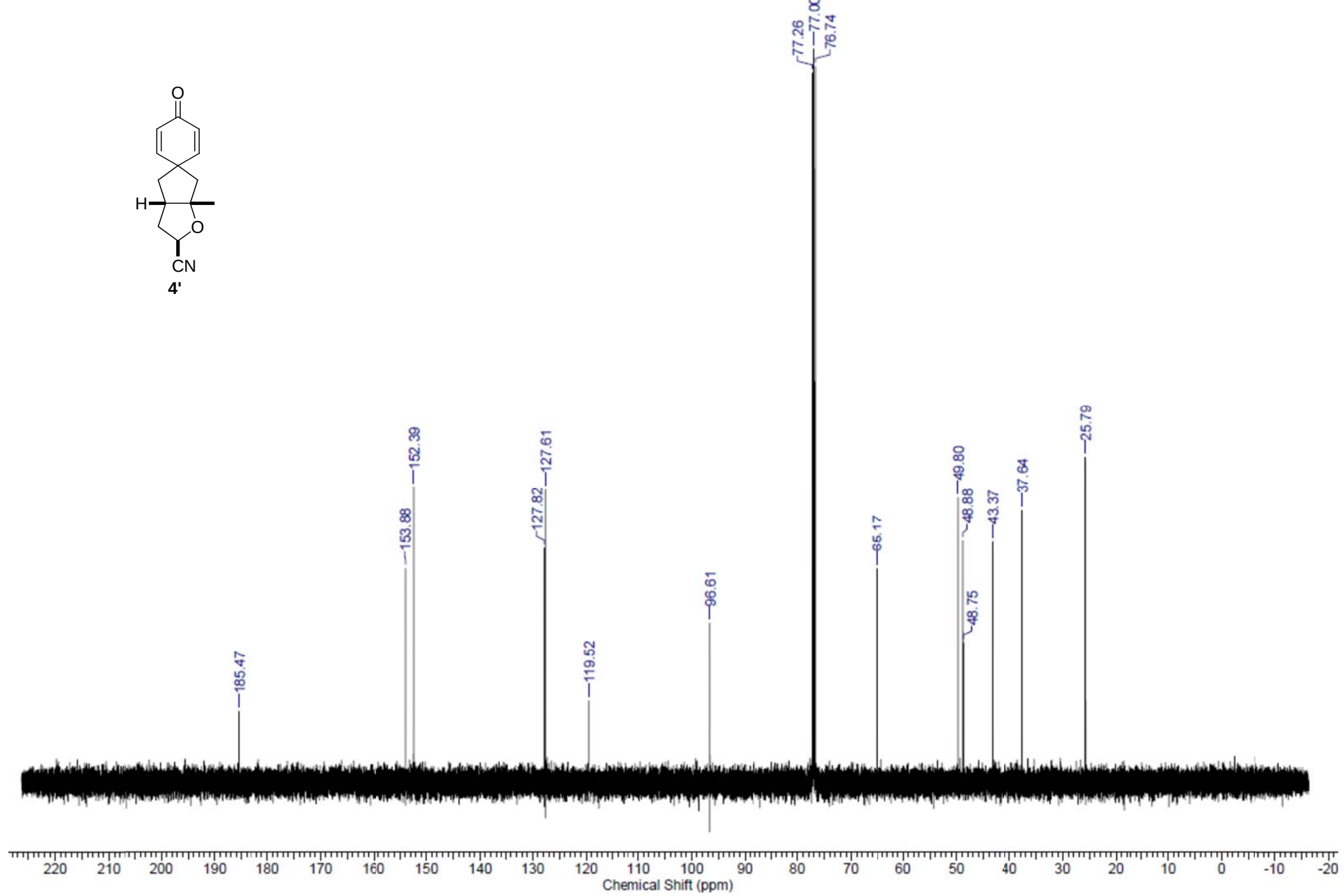
4

77.26
77.00
76.74
154.31
151.56
128.20
127.64
119.77
96.34
65.02
50.28
49.17
49.10
43.43
36.81
26.71
185.59
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
Chemical Shift (ppm)

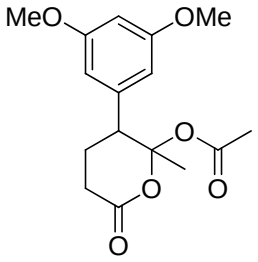

O
H
O
CN

4'

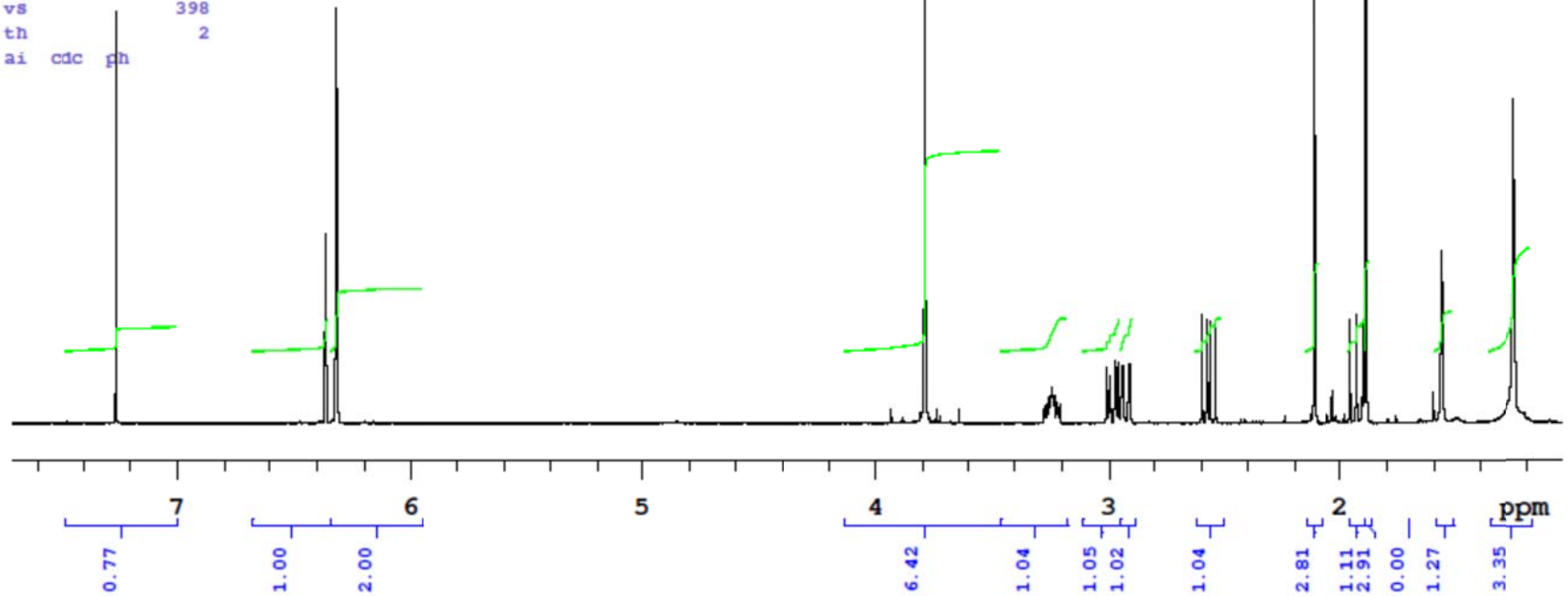
2.32
2.44
4.08
2.05
2.58
3.00
2.53
5.51
3.18
2.90
8.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

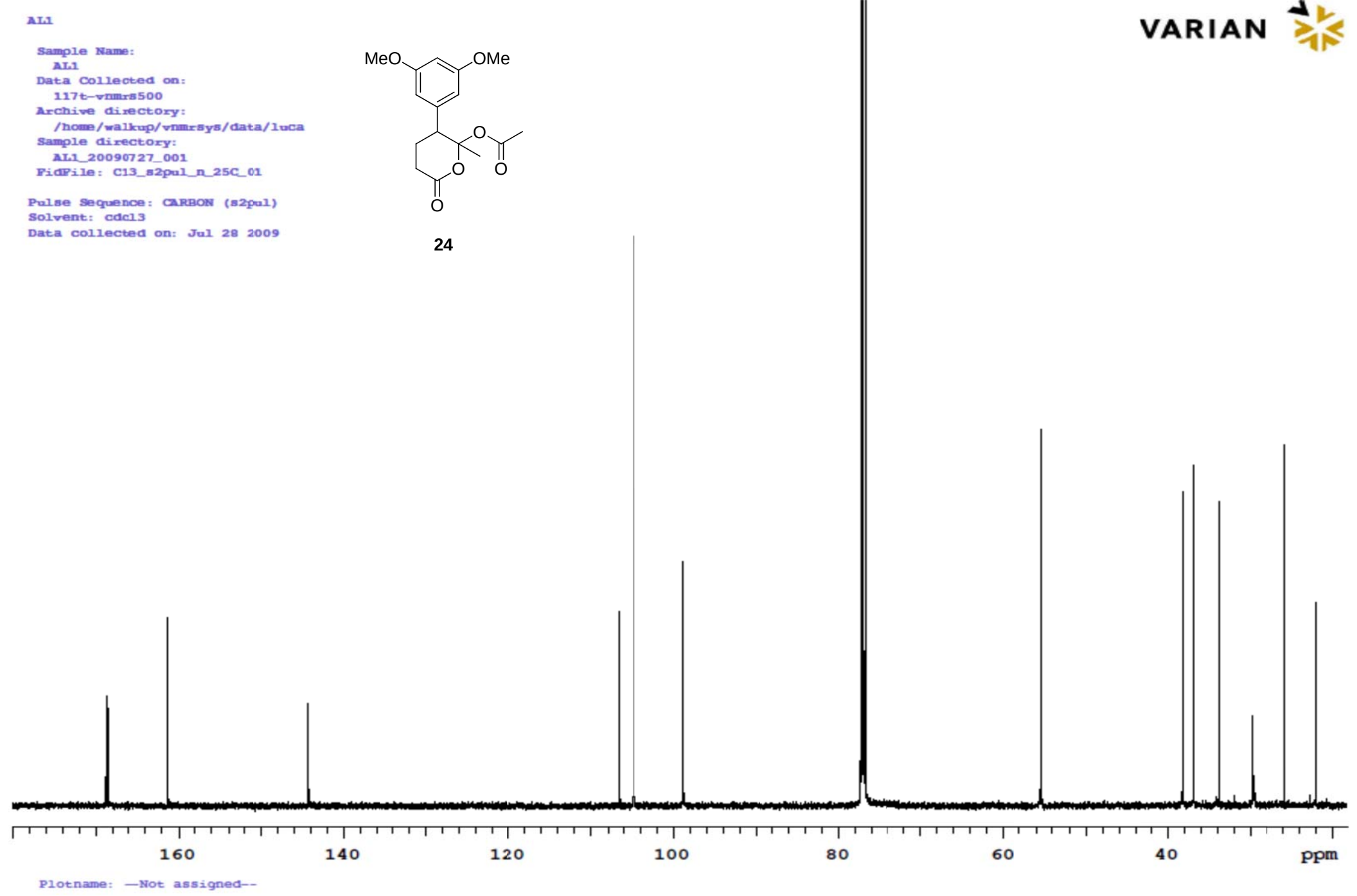

O
H
O
CN
4'
185.47
153.88
152.39
127.82
127.61
119.52
96.61
77.26
77.00
76.74
65.17
49.80
48.88
48.75
43.37
37.64
25.79
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
Chemical Shift (ppm)

SAMPLE		PRESATURATION	
date	Jul 27 2009	satmode	n
solvent	cdcl3	wet	n
file	/home/walkup/~	SPECIAL	
vnmrsys/data/luca/~		temp	25.0
AL1_20090727_001/H~		gain	not used
1_s2pul_n_25C_02.f~		spin	not used
	id	hst	0.008
ACQUISITION		pw90	10.600
sw	8012.8	alfa	10.000
at	2.045	FLAGS	
np	32768	il	n
fb	4000	in	n
bs	32	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	526.2
sfrq	499.809	wp	3328.6
tof	499.8	rfl	1005.5
tpwr	62	rfp	0
pw	5.300	rp	114.3
DECOUPLER		lp	0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
decwave	W40_HCN5mm	vs	398
dpwr	34	th	2
dmf	32258	ai	cdc ph

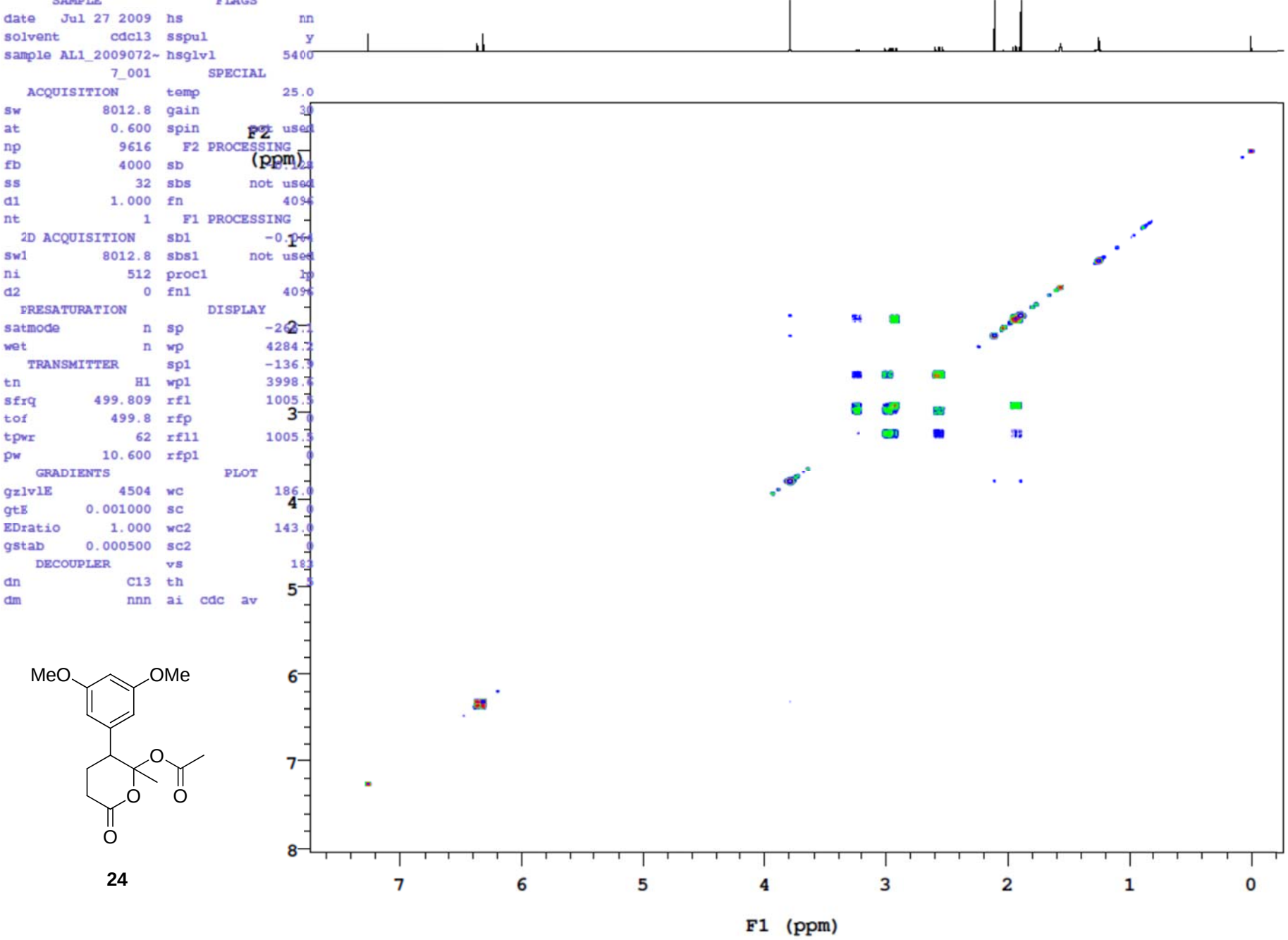


24




AL1
Sample Name:
AL1
Data Collected on:
117t-vnmrs500
Archive directory:
/home/walkup/vnmrsys/data/luca
Sample directory:
AL1_20090727_001
FidFile: C13_s2pul_n_25C_01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 28 2009
MeO
OMe
O
O
O
O
VARIAN
160
140
120
100
80
60
40
ppm
Plotname: --Not assigned--

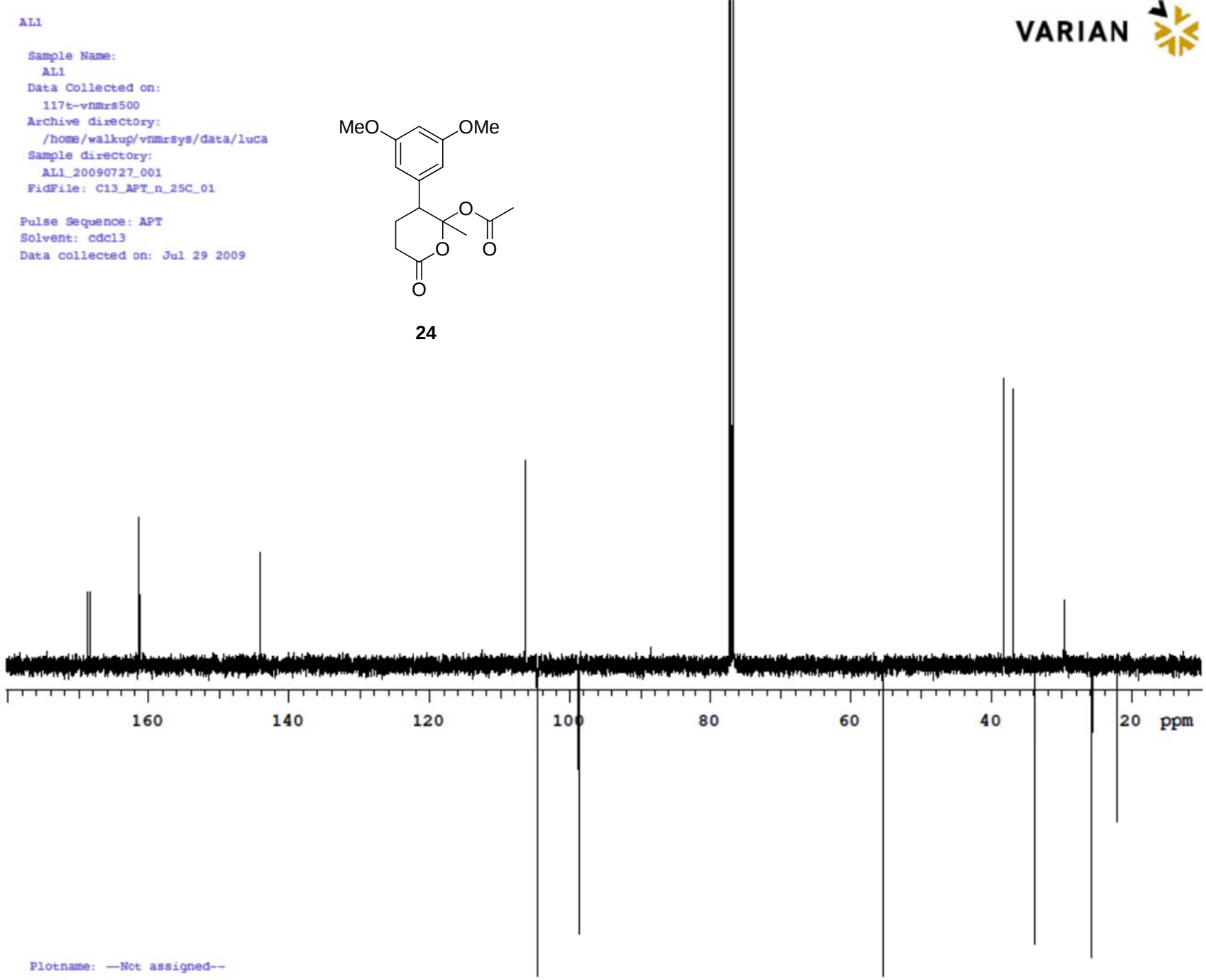
24


SAMPLE
date Jul 27 2009
solvent cdcl3
sample AL1_2009072~7_001
ACQUISITION
sw 8012.8
at 0.600
np 9616
fb 4000
ss 32
d1 1.000
nt 1
2D ACQUISITION
sw1 8012.8
ni 512
d2 0
PRESATURATION
satmode n
wet n
TRANSMITTER
tn H1
sfrq 499.809
tof 499.8
tpwr 62
pw 10.600
GRADIENTS
gzlvlE 4504
gtE 0.001000
EDratio 1.000
gstab 0.000500
DECOUPLER
dn C13
dm nnn
FLAGS
hs nn
sspul y
hsglvl 5400
SPECIAL
temp 25.0
gain 30
spin not used
F2 PROCESSING
sb
sbs not used
fn 4096
F1 PROCESSING
sb1 -0.064
sbs1 not used
proc1 lp
fn1 4096
DISPLAY
sp -262.1
wp 4284.2
sp1 -136.7
wp1 3998.5
rf1 1005.3
rfp 0
rf11 1005.3
rfp1 0
PLOT
wc 186.0
sc 0
wc2 143.0
sc2 0
vs 183
th 5
ai cdc av
F2 (ppm)
F1 (ppm)
1
2
3
4
5
6
7
8
0

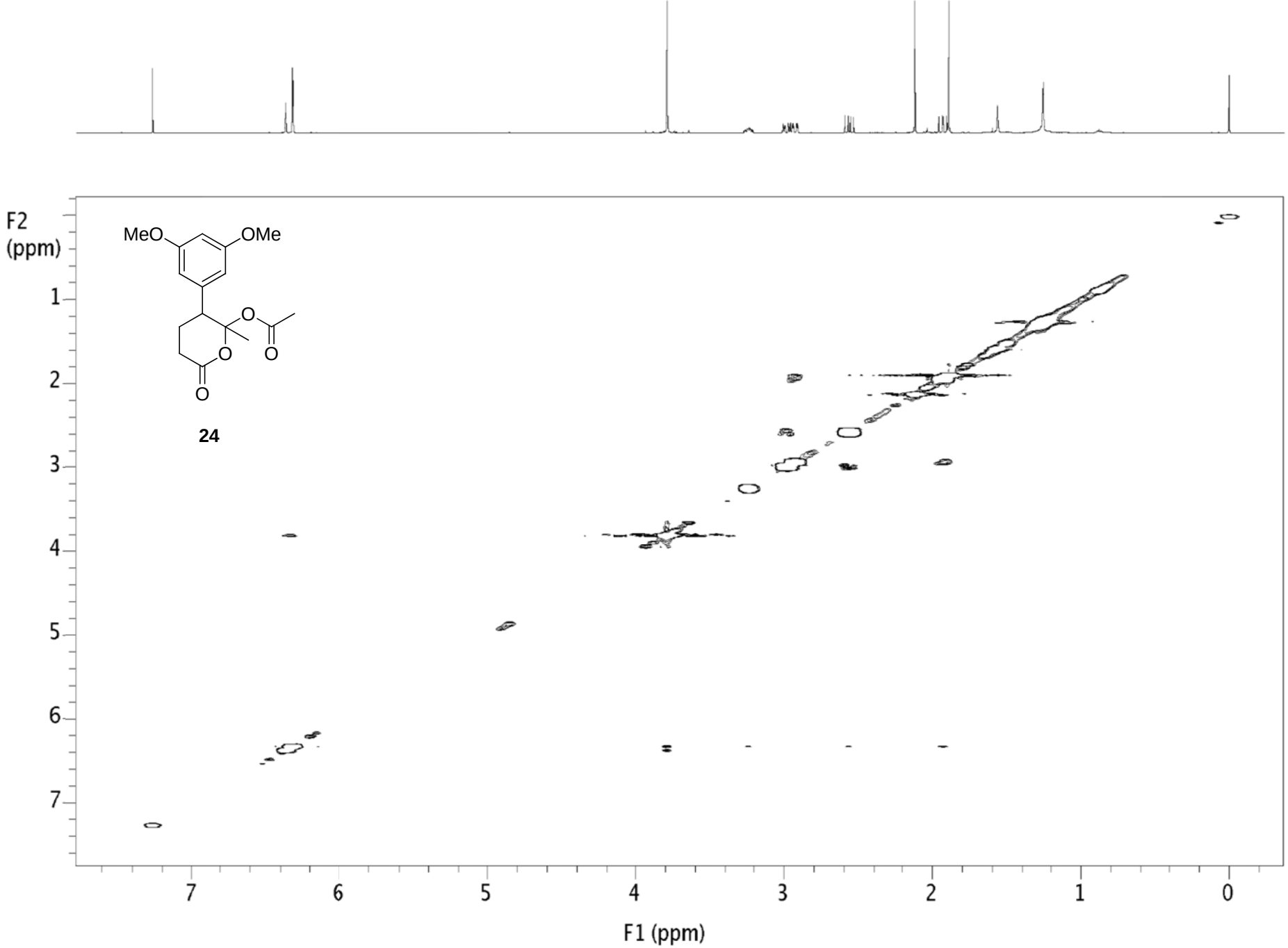
MeO
OMe
O
O
O
O

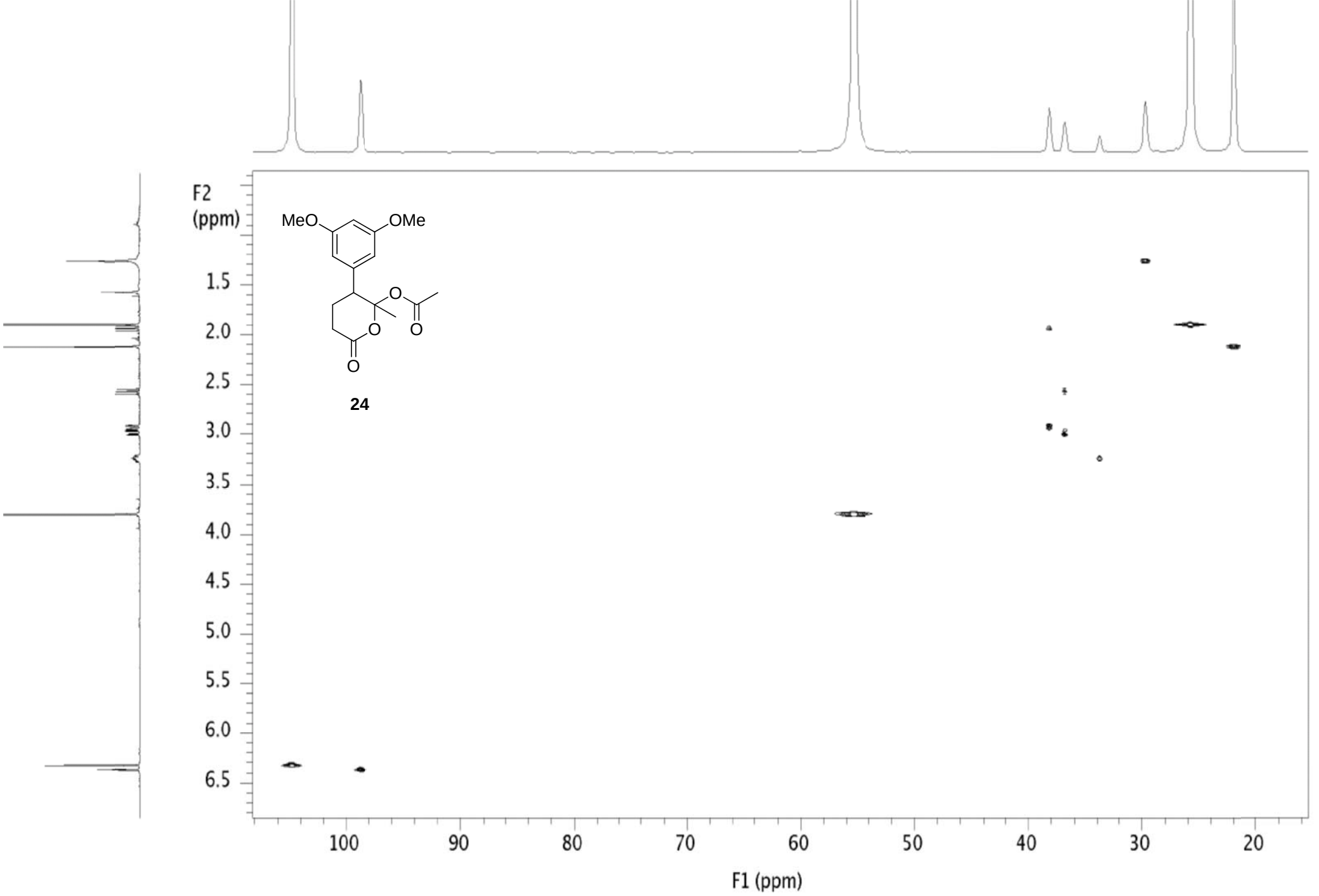
24

COSY


AL1
Sample Name:
AL1
Data Collected on:
117t-vnmrs500
Archive directory:
/home/walkup/vnmrsys/data/luca
Sample directory:
AL1_20090727_001
FidFile: C13_APT_n_25C_01
Pulse Sequence: APT
Solvent: cdcl3
Data collected on: Jul 29 2009
MeO
OMe
O
O
O
O
VARIAN
160
140
120
100
80
60
40
20
ppm
Plotname: --Not assigned--

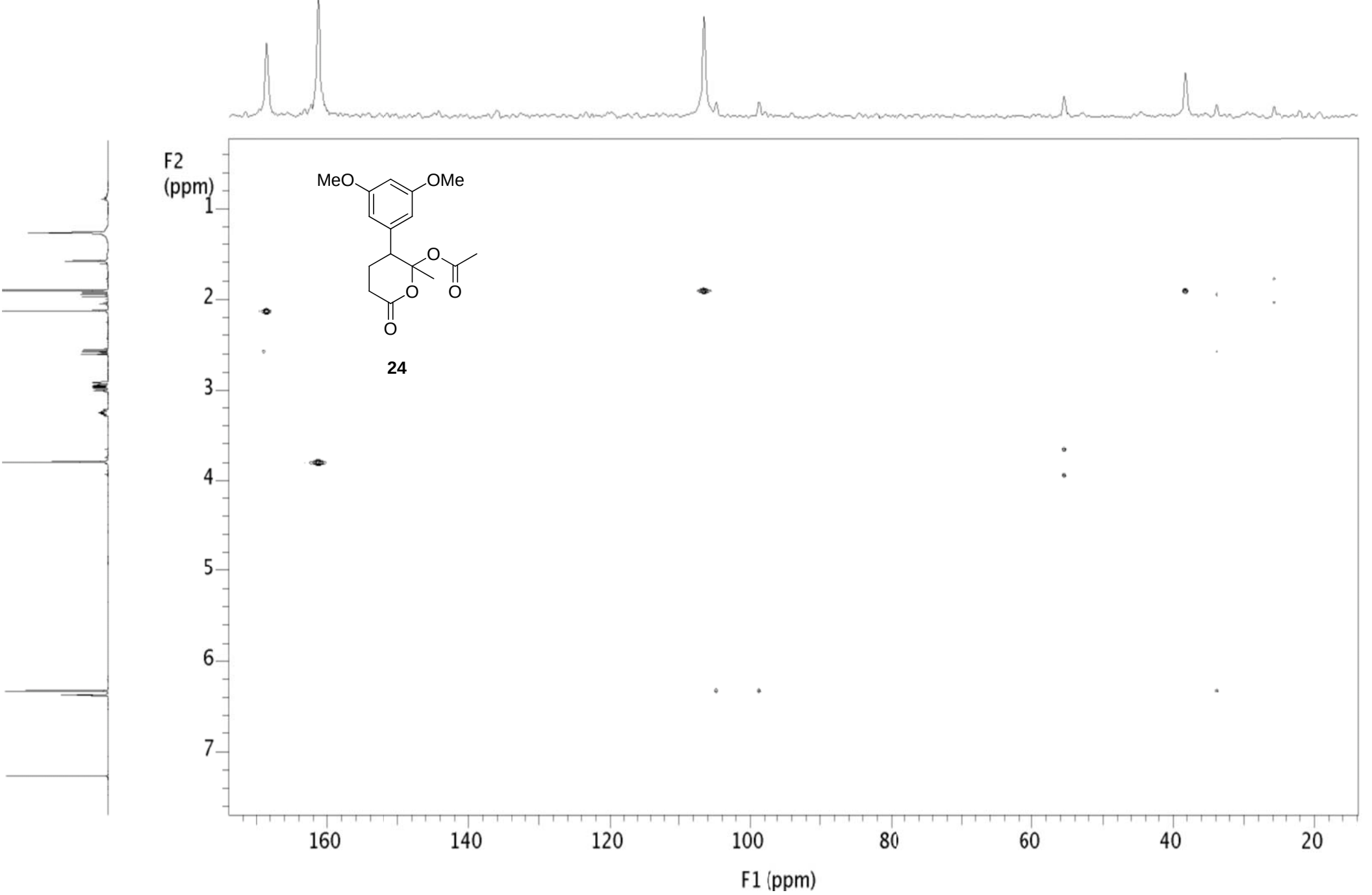
24


F2
(ppm)
MeO
OMe
O
O
O
O
24
1
2
3
4
5
6
7
7
6
5
4
3
2
1
0
F1 (ppm)


F2
(ppm)
MeO
OMe
O
O
O
O
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
100
90
80
70
60
50
40
30
20
F1 (ppm)

24

HSQC



24

HMBC