

Supporting Information

Synthesis of Naphthols by Rh(III)-Catalyzed Domino C-H Activation, Annulation and Lactonization using Sulfoxonium Ylide as a Traceless Directing Group

Vinayak Hanchate, Anil Kumar and Kandikere Ramaiah Prabhu *

Department of Organic Chemistry,

Indian Institute of Science,

Bangalore 560 012,

Karnataka, India

*To whom correspondence should be addressed. E-mail: prabhu@iisc.ac.in

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GENERAL INFORMATION

All reactions were carried out using distilled solvents. Reactions were monitored by using precoated silica TLC plates. Mass spectra were recorded on EI, and ESI (TOF) modes. NMR spectra were recorded in at 400 MHz spectrometers in CDCl_3 , $\text{DMSO-}d_6$, tetramethylsilane (TMS; $\delta = 0.00$ ppm) served as an internal standard for ^1H NMR. The corresponding residual non-deuterated solvent signal (CDCl_3 ; $\delta = 77.16$ ppm and $\text{DMSO-}d_6$; $\delta = 39.52$ ppm) was used as internal standard for ^{13}C NMR. Column chromatography was carried out on silica gel 230-400 mesh or 100-200 mesh (Merck) and thin-layer chromatography was carried out using SILICA GEL GF-254. Chemicals obtained from commercial suppliers were used without further purification. All sulfoxonium ylides derivatives¹ and 4-hydroxy alkynoate derivatives² were synthesized according to a reported literature procedure.

Experimental Section

(A) General Experimental Procedure

In a 8-mL screw cap reaction vial, sulfoxonium ylide derivative (0.2 mmol), 4-hydroxyalkynoate derivative (0.3 mmol), chloroacetic acid (19 mg, 0.2 mmol), $[\text{Cp}^*\text{RhCl}_2]_2$ catalyst (6.2 mg, 5 mol %), AgNTf_2 (16 mg, 20 mol %) were added followed by the addition of ethyl acetate (3 mL). This vial was sealed with a screw cap after flushing with argon and placed in a pre-heated metal block at 80 °C and the reaction mixture was stirred at the same temperature for 16 h. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature filtered through a short silica gel bed (100-200 mesh) and concentrated under

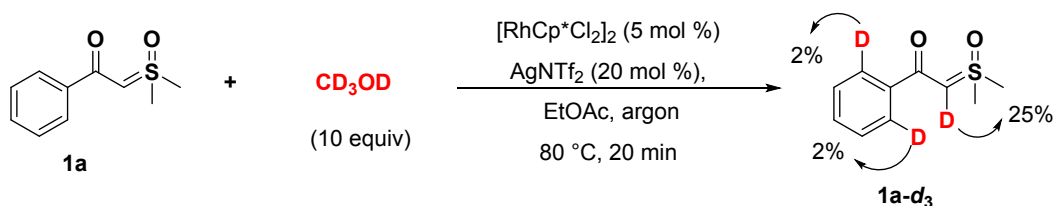
vacuum. The crude product was purified on a flash column using ethyl acetate and petroleum ether.

(B) Experimental procedure for scale-up reaction

In a 50-mL pressure tube sulfoxonium ylide **1a** (5.1 mmol, 1 g), ethyl 4-hydroxy-4-methylpent-2-ynoate (7.6 mmol, 1.19 g), rhodium catalyst (158 mg, 5 mol %), AgNTf₂ (408 mg, 20 mol %), chloroacetic acid (482 mg, 5.1 mmol) were added followed by the addition of EtOAc (40 mL). This tube was sealed with a screw cap after flushing with argon and placed in a pre-heated oil bath at 80 °C and the reaction mixture was stirred at the same temperature for 16 h. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature and filtered through a short silica gel bed (100-200 mesh) and concentrated under vacuum. The crude product was purified on flash column chromatography using ethyl acetate and petroleum ether.

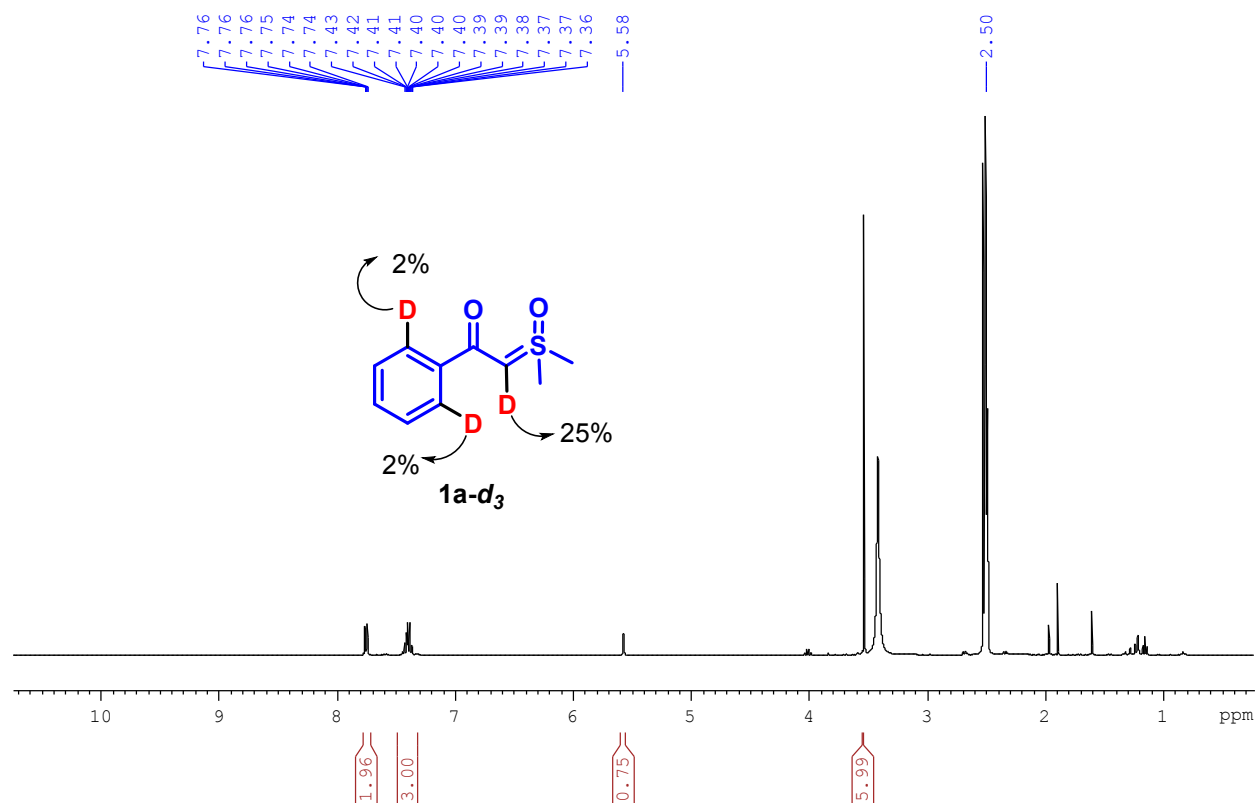
Mechanistic investigation:

Deuterium incorporation studies

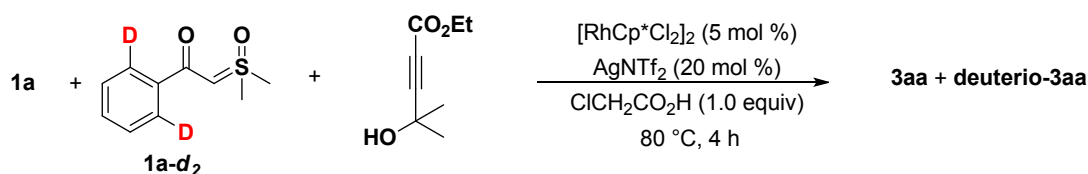


In a 8-mL screw cap reaction vial, sulfoxonium ylide **1a** (0.2 mmol), CD₃OD (10 equiv), rhodium catalyst (5 mol %), AgNTf₂ (20 mol %) were added followed by the addition of ethyl acetate (3 mL). This vial was sealed with a screw cap after flushing with argon, placed in a pre-heated metal block at 80 °C for 20 min. The reaction mixture was cooled to room temperature. The crude reaction mixture was purified on a flash column using EtOAc/ petroleum (1:1) ether to

obtain the desired product, **1a-d₃**, in 45% yield. The deuterium incorporation was calculated from ¹H NMR spectroscopy (see the following ¹H NMR spectra). 2% Deuteration incorporation was observed at *ortho*-positions, where as 25% at α -position to carbonyl group.

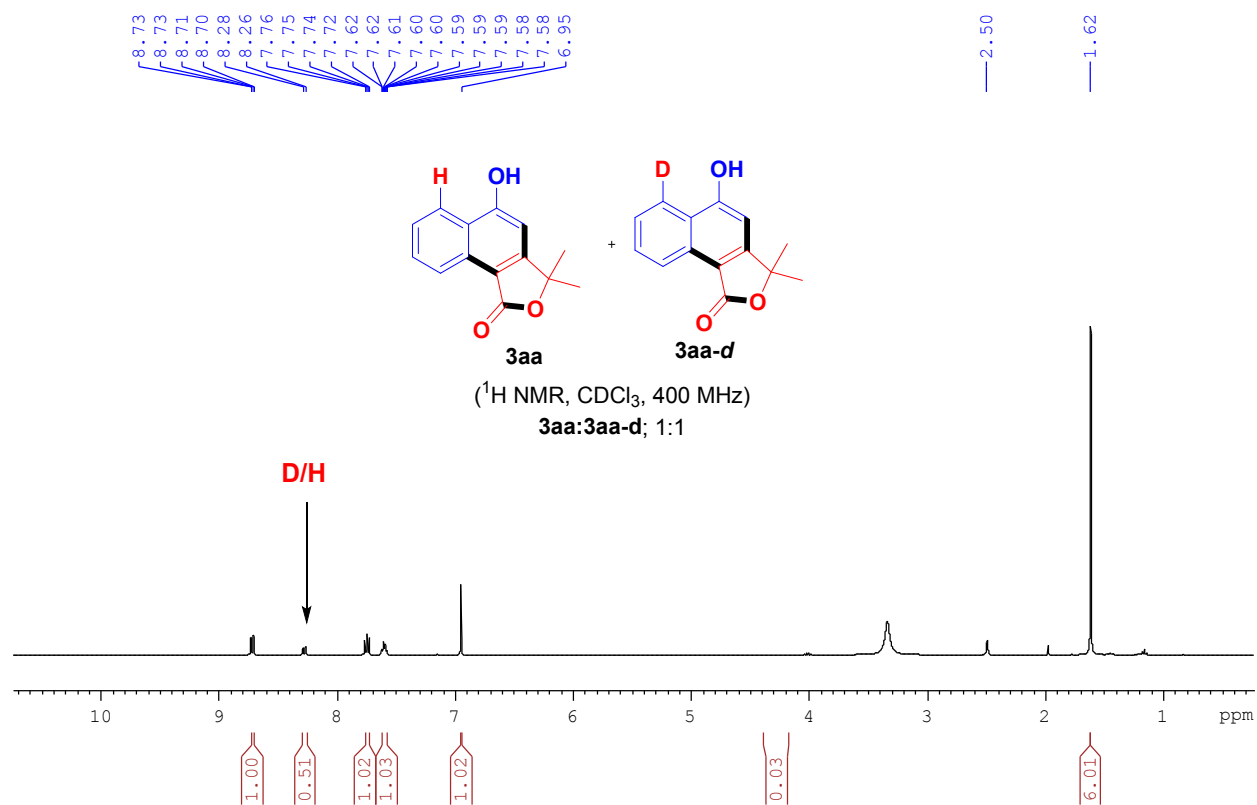


Competitive experiment:



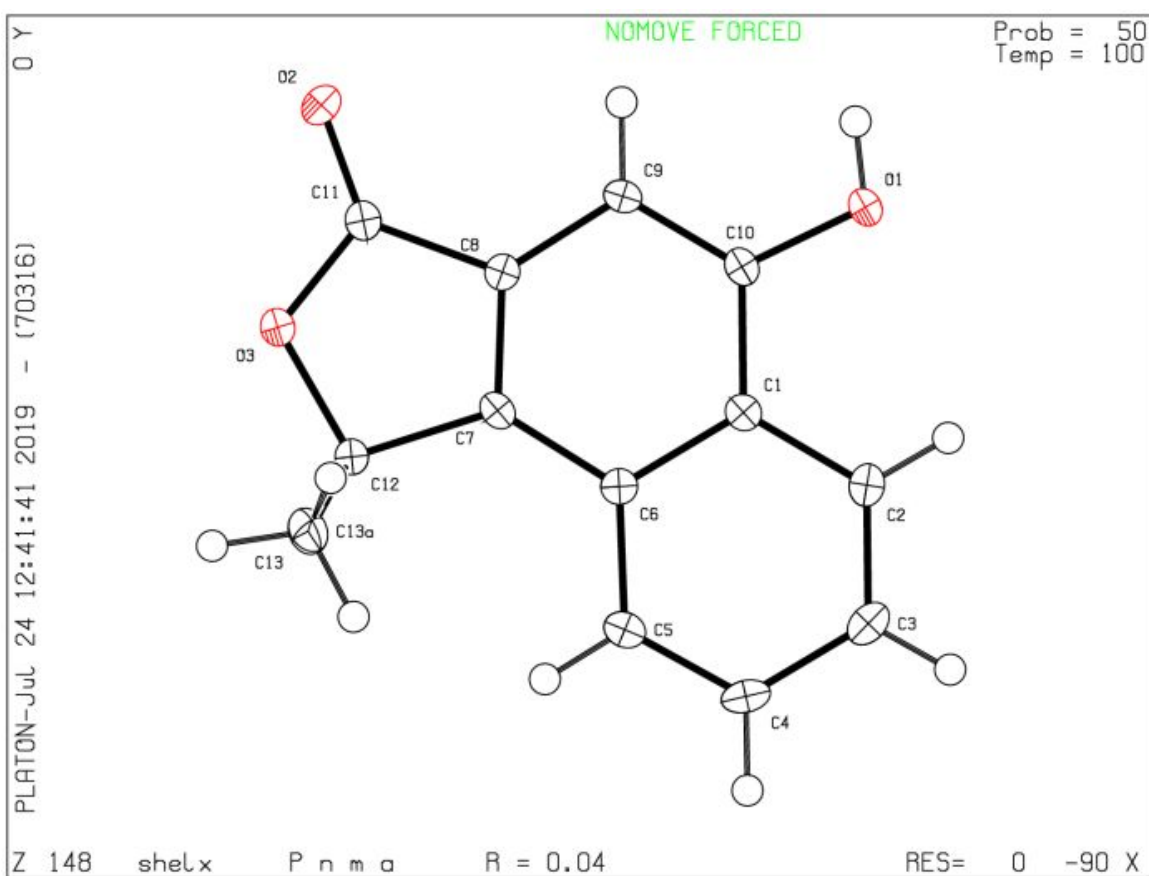
In a 8-mL screw cap reaction vial, 1:1 ratio of **1a-d₂** (100% D) [i.e. by weighing 0.15 mmol of **1a-d₂** 75% D, which contains 0.1 mmol of **1a-d₂** (100% D) and 0.05 mmol of **1a**] and **1a** (0.05 mmol) were taken (now the reaction vial contains 0.1 mmol of each of **1a** and **1a-d₂** (100% D)). Then were added **2a** (0.1 mmol), rhodium-catalyst (5 mol %), chloroacetic acid (20 mg, 0.2 mmol), Cu(OAc)₂·H₂O (1.5 equiv), AgNTf₂ (20 mol %) and ethyl acetate (3 mL) and the vial

was sealed with a screw cap after flushing argon. Then the reaction mixture was stirred at 80 °C for 4 h and concentrated under vacuum. The ^1H NMR spectrum of the product has shown the formation of **3aa** and **3aa-d** in 1:1 ratio (see the following ^1H NMR spectrum).



Crystal data of minor isomer 3aa':

The ORTEP diagram and Crystal Parameters of **3aa'** in thermal ellipsoids are drawn at 50% probability level. The crystal of suitable quality was obtained from slow evaporation of solution of **3aa'** in ethyl acetate, and was analyzed by single crystal diffractometer. Atomic coordinates, bond lengths, bond angles, and thermal parameters for this compound have been deposited at the Cambridge Crystallographic Data Centre. **CCDC 1942930** contains supplementary crystallographic data.



Bond precision: C-C = 0.0019 Å Wavelength: 0.71073

Cell: a= 11.7409(3) b= 6.8805(2) c= 13.6599(4)

alpha= 90 beta= 90 gamma= 90

Temperature: 100K

	Calculated	Reported
Volume	1103.49 (5)	1103.49 (5)
Space group	P n m a	P n m a
Hall group	-P 2ac 2n	-P 2ac 2n
Moiety formula	C ₁₄ H ₁₂ O ₃	
Sum formula	C ₁₄ H ₁₂ O ₃	C ₇ H ₆ O ₁
Mr	228.24	114.12
Dx, g cm ⁻³	1.374	1.374
Z	4	8
Mu (mm ⁻¹)	0.096	0.096
F000	480.0	480.0
F000'	480.26	
h, k, lmax	18, 8, 17	18, 8, 17
Nref	1367	1367
Tmin, Tmax		0.855, 1.000

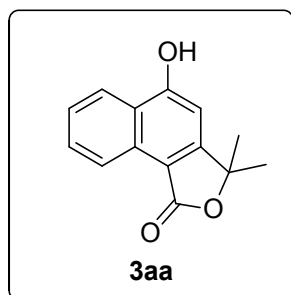
Correction method= #reported T Limits: Tmin=0.855 Tmax=1.000

AbsCorr = MULTI-SCAN

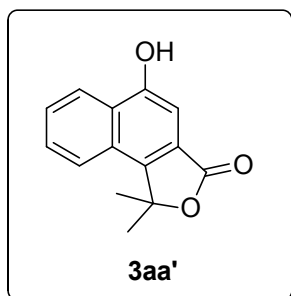
Data completeness= 0.998 Theta (max)= 27.483

R (reflections) = 0.0388(1243) wR2 (reflections) = 0.1083(1364)

S = 1.071 Npar= 103.

Characterization data**5-Hydroxy-3,3-dimethylnaphtho[1,2-c]furan-1(3H)-one (3aa):**

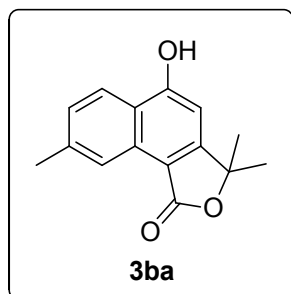
Prepared as shown in general procedure A. White solid, 67% (31 mg); R_f (30% EtOAc-Pet. ether) = 0.4; mp 249-251 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 8.72 (d, J = 8.3 Hz, 1H), 8.27 (d, J = 8.4 Hz, 1H), 7.74 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.60 (ddd, J = 8.3, 6.9, 1.2 Hz, 1H), 6.95 (s, 1H), 1.62 (s, 5H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.2, 160.3, 159.4, 130.1, 129.4, 125.9, 124.6, 123.3, 122.4, 108.5, 100.2, 83.4, 26.7; IR (KBr, cm^{-1}): 3210, 2976, 1707, 1572; HRMS (ESI) (m/z): Calculated for $\text{C}_{14}\text{H}_{12}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 229.0865, found $[\text{M}+\text{H}]^+$: 229.0865.

5-Hydroxy-1,1-dimethylnaphtho[1,2-c]furan-3(1H)-one (3aa)':

Prepared as shown in general procedure A. White solid, 4% (2 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 208-210 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 10.89 (brs, 1H), 8.37 – 8.32 (m, 1H), 8.15 – 8.10 (m, 1H), 7.75 – 7.69 (m, 2H), 7.04 (s, 1H), 1.80 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.4, 155.0, 143.7, 128.2, 128.1, 127.6, 126.8, 124.5, 123.8, 123.0, 100.2, 85.6,

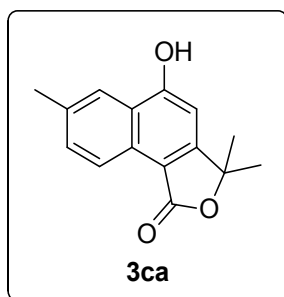
26.9; IR (KBr, cm^{-1}): 3273, 3085, 1718, 1588; HRMS (ESI) (m/z): Calculated for $\text{C}_{14}\text{H}_{12}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 229.0865, found $[\text{M}+\text{H}]^+$: 229.0862.

5-Hydroxy-3,3,8-trimethylnaphtho[1,2-c]furan-1(3H)-one (3ba):



Prepared as shown in general procedure A. White solid, 58% (28 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp above 260 °C; ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 8.45 (s, 1H), 8.12 (d, $J = 8.6$ Hz, 1H), 7.41 (dd, $J = 8.6, 1.5$ Hz, 1H), 6.87 (s, 1H), 2.48 (s, 3H), 1.58 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO}-d_6$): δ 169.9, 160.5, 160.1, 139.9, 130.8, 128.4, 123.6, 123.0, 121.9, 108.3, 99.8, 84.0, 27.1, 22.0; IR (KBr, cm^{-1}): 3399, 3060, 3046, 1704, 1577, 1242; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{14}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 243.1021, found $[\text{M}+\text{H}]^+$: 243.1022.

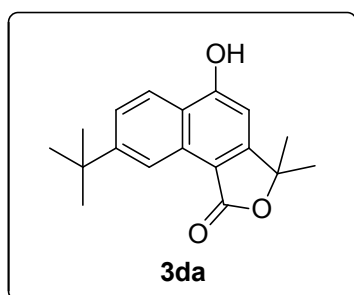
5-Hydroxy-3,3,7-trimethylnaphtho[1,2-c]furan-1(3H)-one (3ca):



Prepared as shown in general procedure A. White solid, 74% (36 gm); mp 206-208 °C; R_f (30% EtOAc-Pet. ether) = 0.4; ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 11.52 (brs, 1H), 8.61 (d, $J = 8.4$ Hz, 1H), 8.06 (s, 1H), 7.57 (dd, $J = 8.5, 1.6$ Hz, 1H), 6.92 (s, 1H), 2.50 (s, 3H), 1.62 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$

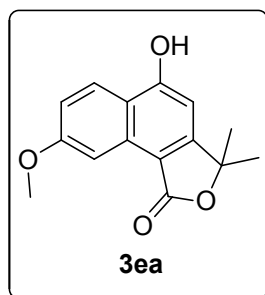
NMR (101 MHz, DMSO- d_6): δ 169.3, 159.8, 158.5, 135.2, 131.3, 128.2, 124.8, 122.3, 122.3, 108.5, 100.1, 83.4, 26.7, 21.5; IR (KBr, cm^{-1}): 3060, 3024, 1582, 1219, 1112; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{14}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 243.1021, found $[\text{M}+\text{H}]^+$: 243.1021.

8-(*tert*-Butyl)-5-hydroxy-3,3-dimethylnaphtho[1,2-*c*]furan-1(3H)-one (3da):



Prepared as shown in general procedure A. White solid, 67% (38 mg); R_f (30% EtOAc-Pet. ether) = 0.6; mp above 260 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 8.74 (d, $J = 1.7$ Hz, 1H), 8.20 (d, $J = 8.9$ Hz, 1H), 7.68 (dd, $J = 8.9, 1.8$ Hz, 1H), 6.89 (s, 1H), 1.61 (s, 6H), 1.37 (s, 9H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.0, 159.8, 159.1, 151.6, 130.0, 124.1, 122.7, 122.4, 117.2, 108.18, 99.4, 83.0, 34.6, 30.6, 26.4; IR (KBr, cm^{-1}): 3448, 2960, 1702, 1575, 1210; HRMS (ESI) (m/z): Calculated for $\text{C}_{18}\text{H}_{20}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 285.1491, found $[\text{M}+\text{H}]^+$: 285.1489.

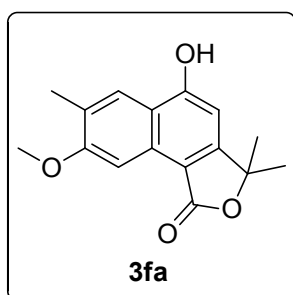
5-Hydroxy-8-methoxy-3,3-dimethylnaphtho[1,2-*c*]furan-1(3H)-one (3ea):



Prepared as shown in general procedure A. White solid, 62% (32 mg); R_f (30% EtOAc-Pet. ether) = 0.3; mp above 260 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 11.50 (brs, 1H), 8.17 (d, $J = 9.2$ Hz, 1H), 8.10 (d, $J = 2.6$ Hz, 1H), 7.21 (dd, $J = 9.2, 2.6$ Hz, 1H), 6.79 (s, 1H), 3.90 (s, 3H), 1.60 (s,

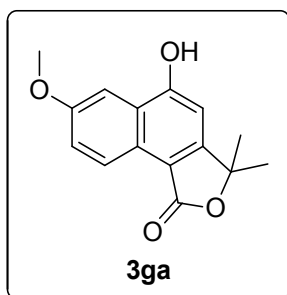
6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.4 (s), 160.4, 160.2, 160.0, 132.1, 125.1, 119.3, 117.49, 107.5, 101.7, 98.5, 83.3, 55.3, 26.8; IR (KBr, cm^{-1}): 3246, 2923, 1706, 1580, 1198; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{14}\text{O}_4\text{H} [\text{M}+\text{H}]^+$: 259.0970, found $[\text{M}+\text{H}]^+$: 259.0970.

5-Hydroxy-8-methoxy-3,3,7-trimethylnaphtho[1,2-c]furan-1(3H)-one (3fa):



Prepared as shown in general procedure A. White solid, 66% (36 mg); R_f (30% EtOAc-Pet. ether) = 0.3; mp. above 250 °C charred; ^1H NMR (400 MHz, DMSO- d_6): δ 11.38 (s, 1H), 8.05 (s, 1H), 8.01 (s, 1H), 6.77 (s, 1H), 3.93 (s, 3H), 2.33 (s, 3H), 1.60 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.5, 160.0, 159.1, 159.0, 130.8, 127.0, 124.3, 119.1, 107.5, 100.2, 98.6, 83.3, 55.5, 26.7, 16.9; IR (KBr, cm^{-1}): 3244, 2925, 1698, 1578; HRMS (ESI) (m/z): Calculated for $\text{C}_{16}\text{H}_{16}\text{O}_4\text{H} [\text{M}+\text{H}]^+$: 273.1127, found $[\text{M}+\text{H}]^+$: 273.1128.

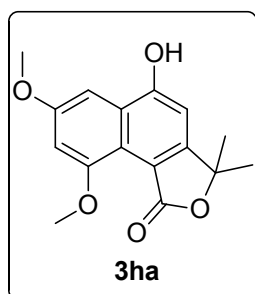
5-Hydroxy-7-methoxy-3,3-dimethylnaphtho[1,2-c]furan-1(3H)-one (3ga):



Prepared as shown in general procedure A. White solid, 40% (21 mg); R_f (30% EtOAc-Pet. ether) = 0.3; mp above 250 °C charred; ^1H NMR (400 MHz, DMSO- d_6): δ 11.51 (s, 1H), 8.62 (d, $J = 9.1$ Hz, 1H), 7.59 (d, $J = 2.5$ Hz, 1H), 7.40 (dd, $J = 9.1, 2.6$ Hz, 1H), 6.93 (s, 1H), 3.89 (s, 3H),

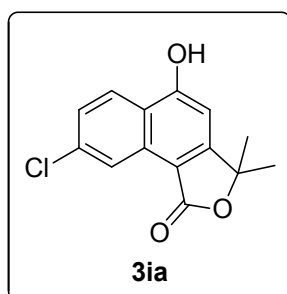
1.61 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.3, 159.1, 157.3, 156.9, 126.0, 125.0, 124.1, 121.2, 108.7, 102.2, 100.5, 83.5, 55.3, 26.8; IR (KBr, cm^{-1}): 3214, 2974, 1706, 1575, 1222; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{14}\text{O}_4\text{H}$ $[\text{M}+\text{H}]^+$: 259.0970, found $[\text{M}+\text{H}]^+$: 259.0968.

5-Hydroxy-7,9-dimethoxy-3,3-dimethylnaphtho[1,2-c]furan-1(3H)-one (3ha):



Prepared as shown in general procedure A. White solid, 39% (23 mg); R_f (40% EtOAc-Pet. ether) = 0.3; mp Charred at 230-232 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 11.42 (brs, 1H), 7.20 (s, 1H), 6.88 (s, 1H), 6.78 (s, 1H), 3.87 (s, 6H), 1.55 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): 166.3, 158.6, 157.9, 157.9, 157.6, 127.6, 118.0, 109.3, 101.4, 100.9, 94.3, 80.5, 55.6, 55.2, 27.3; IR (KBr, cm^{-1}): 3255, 2982, 1742, 1553; HRMS (ESI) (m/z): Calculated for $\text{C}_{16}\text{H}_{16}\text{O}_5\text{H}$ $[\text{M}+\text{H}]^+$: 289.1076, found $[\text{M}+\text{H}]^+$: 289.1076.

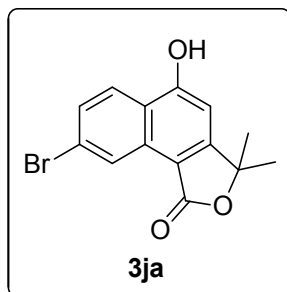
8-Chloro-5-hydroxy-3,3-dimethylnaphtho[1,2-c]furan-1(3H)-one (3ia):



Prepared as shown in general procedure A. White solid, 58% (30 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp above 260 °C; ^1H NMR (400 MHz, DMSO- d_6) δ : 8.59 (s, 1H), 8.22 (d, $J = 8.9$ Hz,

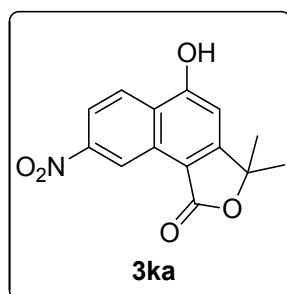
1H), 7.56 (d, $J = 7.4$ Hz, 1H), 6.96 (s, 1H), 1.59 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.5, 161.1, 160.6, 135.2, 131.1, 126.9, 126.1, 123.4, 121.6, 108.2, 101.0, 84.5, 26.9; IR (KBr, cm^{-1}): 3321, 2957, 1703, 1575, 1121; HRMS (ESI) (m/z): Calculated for $\text{C}_{14}\text{H}_{11}\text{ClO}_3\text{H}$ $[\text{M}+\text{H}]^+$: 263.0475, found $[\text{M}+\text{H}]^+$: 263.0472.

8-Bromo-5-hydroxy-3,3-dimethylnaphtho[1,2-c]furan-1(3H)-one (3ja):



Prepared as shown in general procedure A. White solid, 70% (43 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp above 260 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 8.72 (s, 1H), 8.09 (d, $J = 8.9$ Hz, 1H), 7.62 (d, $J = 8.9$ Hz, 1H), 6.95 (s, 1H), 1.58 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.45, 160.9, 160.7, 131.3, 129.3, 126.0, 124.7, 124.1, 123.5, 108.0, 101.1, 84.4, 26.9; IR (KBr, cm^{-1}): 3176, 2952, 1698, 1569; HRMS (ESI) (m/z): Calculated for $\text{C}_{14}\text{H}_{11}\text{BrO}_3\text{H}$ $[\text{M}+\text{H}]^+$: 306.9970, found $[\text{M}+\text{H}]^+$: 306.9969.

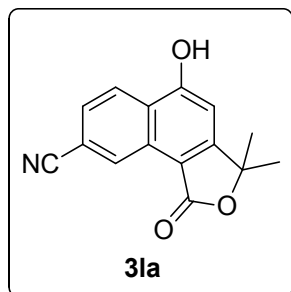
5-Hydroxy-3,3-dimethyl-8-nitronaphtho[1,2-c]furan-1(3H)-one (3ka):



Prepared as shown in general procedure A. yellow solid, 67% (37 mg); R_f (50% EtOAc-Pet. ether) = 0.3; mp above 260 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 12.15 (s, 1H), 9.41 (s, 1H),

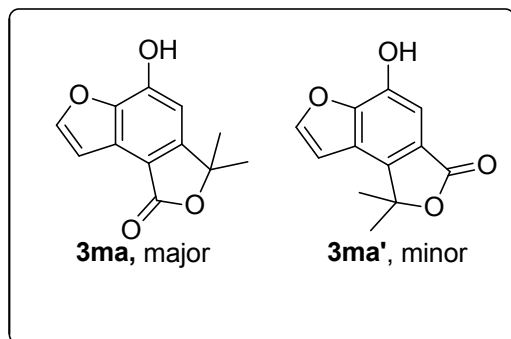
8.41 (d, $J = 6.6$ Hz, 1H), 8.25 (d, $J = 7.2$ Hz, 1H), 7.14 (s, 1H), 1.66 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 168.2, 160.9, 159.9, 147.4, 128.8, 127.2, 125.4, 118.7, 118.0, 109.9, 103.2, 83.8, 26.1; IR (KBr, cm^{-1}): 3223, 3113, 1700, 1541, 1312, 1196; HRMS (ESI) (m/z): Calculated for $\text{C}_{14}\text{H}_{11}\text{NO}_5\text{H}$ $[\text{M}+\text{H}]^+$: 274.0715, found $[\text{M}+\text{H}]^+$: 274.0716.

5-Hydroxy-3,3-dimethyl-1-oxo-1,3-dihydronaphtho[1,2-c]furan-8-carbonitrile (3la):



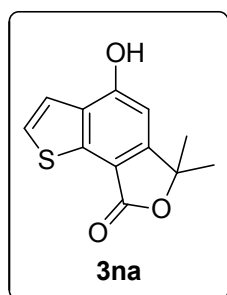
Prepared as shown in general procedure A. White solid, 61% (31 mg); R_f (50% EtOAc-Pet. ether) = 0.4; mp above 250 °C charred; ^1H NMR (400 MHz, DMSO- d_6): δ 9.03 (s, 1H), 8.41 (d, $J = 8.7$ Hz, 1H), 7.90 (d, $J = 8.7$ Hz, 1H), 7.12 (s, 1H), 1.65 (s, 6H); ^{13}C NMR (101 MHz, DMSO- d_6) δ : 168.6, 160.7, 160.1, 128.9, 127.8, 126.8, 126.2, 125.0, 118.7, 111.8, 108.7, 102.9, 84.2, 26.4; IR (KBr, cm^{-1}): 3433, 3045, 2228, 1745, 1573, 1242; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{11}\text{NO}_3\text{H}$ $[\text{M}+\text{H}]^+$: 254.0817, found $[\text{M}+\text{H}]^+$: 254.0817.

8-Bromo-5-hydroxy-3,3-dimethylnaphtho[1,2-c]furan-1(3H)-one (3ma):



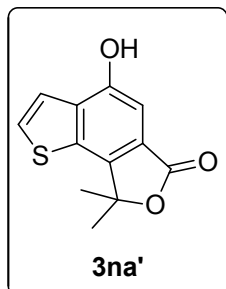
Prepared as shown in general procedure A. Both regio-isomers formed (72:28) as inseparable mixture. White solid, 59% (26 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 222 °C charred; ^1H NMR (400 MHz, $\text{DMSO-}d_6$): δ 11.44 (s, 1H), 8.22 (d, $J = 2.1$ Hz, 1H), 7.15 (d, $J = 2.1$ Hz, 1H), 6.88 (s, 1H), 1.59 (s, 6H) are peaks corresponding to major isomer, 10.73 (s, 0.40H), 7.37 (d, $J = 2.2$ Hz, 0.41H), 7.01 (s, 0.40H), 1.67 (s, 2.57H) are peaks corresponding to minor isomer; $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO-}d_6$): δ 169.4, 168.3, 154.7, 149.1, 148.8, 148.1, 147.4, 144.3, 144.0, 140.0, 125.2, 121.9, 120.2, 107.2, 105.3, 105.1, 102.3, 84.7, 84.2, 27.2, 26; IR (KBr, cm^{-1}): 3229, 2983, 1704, 1598, 1337, 1189; HRMS (ESI) (m/z): Calculated for $\text{C}_{12}\text{H}_{10}\text{O}_4\text{H}$ $[\text{M}+\text{H}]^+$: 219.0657, found $[\text{M}+\text{H}]^+$: 219.0656.

4-Hydroxy-6,6-dimethylthieno[2,3-*e*]isobenzofuran-8(6H)-one (3na):



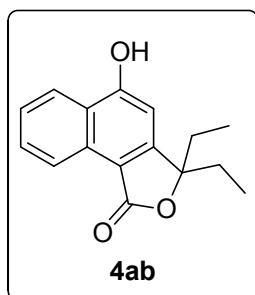
Prepared as shown in general procedure A. White solid, 31% (15 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 243-245 °C; ^1H NMR (400 MHz, $\text{DMSO-}d_6$): δ 11.40 (s, 1H), 7.78 (s, 1H), 7.62 (s, 1H), 6.89 (s, 1H), 1.63 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO-}d_6$): δ 168.12, 158.7, 156.6, 136.4, 130.9, 126.9, 120.7, 109.5, 101.5, 85.4, 27.1.; IR (KBr, cm^{-1}): 3382, 1702, 1634; HRMS (ESI) (m/z): Calculated for $\text{C}_{12}\text{H}_{10}\text{O}_3\text{SH}$ $[\text{M}+\text{H}]^+$: 235.0429, found $[\text{M}+\text{H}]^+$: 235.0431.

4-Hydroxy-8,8-dimethylthieno[2,3-*e*]isobenzofuran-6(8H)-one (3na'):

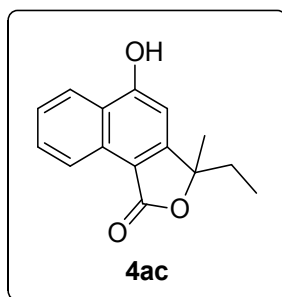


Prepared as shown in general procedure A. White solid, 30% (15 mg); R_f (30% EtOAc-Pet. ether) = 0.6; mp above 250 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 10.78 (s, 3H), 8.03 (d, J = 5.1 Hz, 4H), 7.70 (d, J = 5.2 Hz, 4H), 7.01 (s, 4H), 1.69 (s, 24H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.2, 153.9, 140.8, 135.0, 131.8, 130.2, 122.2, 121.9, 102.6, 84.1, 25.6; IR (KBr, cm^{-1}): 3236, 1707; HRMS (ESI) (m/z): Calculated for $\text{C}_{12}\text{H}_{10}\text{O}_3\text{SH}$ $[\text{M}+\text{H}]^+$: 235.0429, found $[\text{M}+\text{H}]^+$: 235.0428

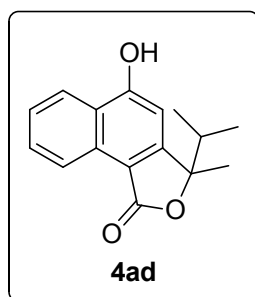
3,3-Diethyl-5-hydroxynaphtho[1,2-c]furan-1(3H)-one (4ab):



Prepared as shown in general procedure A. White solid, 55% (28 mg); R_f (30% EtOAc-Pet. ether) = 0.4; mp 192-194 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 8.68 (d, J = 8.2 Hz, 1H), 8.25 (d, J = 8.3 Hz, 1H), 7.76 – 7.68 (m, 1H), 7.62 – 7.55 (m, 1H), 6.86 (s, 1H), 2.02 (dq, J = 14.6, 7.3 Hz, 2H), 1.90 (dq, J = 14.7, 7.2 Hz, 2H), 0.56 (t, J = 7.3 Hz, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 170.6, 160.6, 156.6, 130.3, 130.0, 126.5, 125.0, 123.7, 122.8, 111.2, 100.6, 89.2, 30.8, 7.7; IR (Neat, cm^{-1}): 3134, 2930, 2839, 1698, 1571; HRMS (ESI) (m/z): Calculated for $\text{C}_{16}\text{H}_{16}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 257.1178, found $[\text{M}+\text{H}]^+$: 257.1177.

3-Ethyl-5-hydroxy-3-methylnaphtho[1,2-c]furan-1(3H)-one (4ac):

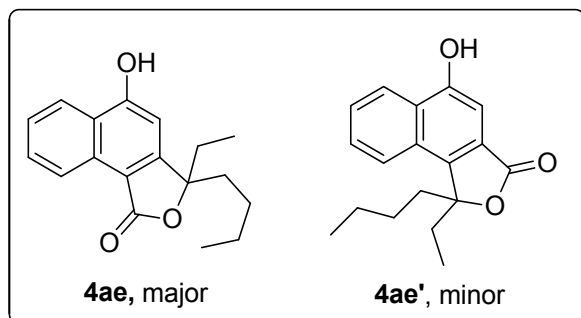
Prepared as shown in general procedure A. White solid, 69% (33 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 210-212 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 11.62 (s, 1H), 8.73 (d, J = 8.2 Hz, 1H), 8.28 (d, J = 8.3 Hz, 1H), 7.83 – 7.68 (m, 1H), 7.59 (ddd, J = 8.2, 7.0, 1.1 Hz, 1H), 6.90 (s, 1H), 2.09 – 1.86 (m, 2H), 1.60 (s, 3H), 0.64 (t, J = 7.4 Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.5, 160.3, 157.9, 130.0, 129.4, 125.9, 124.7, 123.3, 122.4, 109.5, 100.2, 85.8, 31.5, 25.3, 7.7.; IR (KBr, cm^{-1}): 3212, 3071, 1705, 1576; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{14}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 243.1021, found $[\text{M}+\text{H}]^+$: 243.1019.

5-Hydroxy-3-isopropyl-3-methylnaphtho[1,2-c]furan-1(3H)-one (4ad):

Prepared as shown in general procedure A. White solid, 54% (28 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 184-186 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 11.60 (s, 1H), 8.73 (d, J = 8.3 Hz, 1H), 8.27 (d, J = 8.4 Hz, 1H), 7.74 (t, J = 7.6 Hz, 1H), 7.59 (t, J = 7.6 Hz, 1H), 6.91 (s, 1H), 2.20 (dq, J = 13.6, 6.8 Hz, 1H), 0.98 (d, J = 6.7 Hz, 3H), 0.67 (d, J = 6.8 Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.7, 160.1, 156.0, 130.0, 129.4, 125.9, 124.6, 123.3, 122.4, 109.5, 100.6,

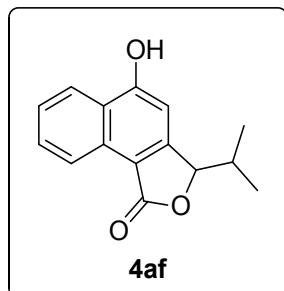
87.7, 35.1, 23.7, 16.8; IR (KBr, cm^{-1}): 3255, 3071, 1703, 1577; HRMS (ESI) (m/z): Calculated for $\text{C}_{16}\text{H}_{16}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 257.1178, found $[\text{M}+\text{H}]^+$: 257.1179.

3-Butyl-3-ethyl-5-hydroxynaphtho[1,2-c]furan-1(3H)-one (4ae):



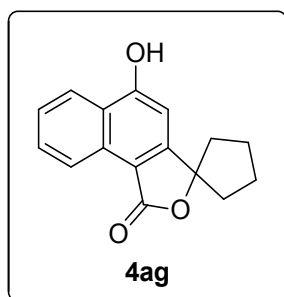
Prepared as shown in general procedure A. **4ae** and **4ae'** formed in inseparable mixture (88:12) White solid, 47% (27 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp above 250 °C charred; ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ Major isomer peaks: 11.60 (s, 1H), 8.72 (d, $J = 8.3$ Hz, 1H), 8.27 (d, $J = 8.3$ Hz, 1H), 7.60 (t, $J = 7.5$ Hz, 1H), 6.87 (s, 1H), 2.09 – 1.88 (m, 4H), 0.59 (t, $J = 7.3$ Hz, 3H), 0.44 (t, $J = 7.2$ Hz, 1H). Minor isomer peak: 10.90 (s, 0.25H), 8.35 (dd, $J = 6.4, 3.2$ Hz, 0.27H), 8.08 (dd, $J = 6.2, 3.1$ Hz, 0.27H), 7.04 (s, 0.26H), 2.33 – 2.11 (m, 1.18H), 0.44 (t, $J = 7.2$ Hz, 0.80H). merged peaks: 7.81 – 7.66 (m, 1.52H), 0.82 – 0.54 (m, 8.4H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO}-d_6$): δ 169.8, 160.3, 156.2, 129.9, 129.4, 125.9, 124.7, 123.3, 122.4, 110.6, 100.3, 88.2, 37.1, 30.7, 24.9, 22.1, 13.8, 7.3; IR (KBr, cm^{-1}): 3244, 3071, 1731, 1694; HRMS (ESI) (m/z): Calculated for $\text{C}_{18}\text{H}_{20}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 285.1491, found $[\text{M}+\text{H}]^+$: 285.1494.

5-Hydroxy-3-isopropyl-naphtho[1,2-c]furan-1(3H)-one (4af):



Prepared as shown in general procedure A. yellow solid, 38% (18 mg); R_f (30% EtOAc-Pet. ether) = 0.6; mp 183-185 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 10.98 (s, 1H), 8.27 – 8.36 (m, 1H), 8.00 – 8.13 (m, 1H), 7.56 – 7.85 (m, 2H), 7.03 (s, 1H), 5.93 (d, J = 2.3 Hz, 1H), 2.66 (dd, J = 6.8, 2.3 Hz, 1H), 1.27 (d, J = 6.8 Hz, 4H), 1.27 (d, J = 6.8 Hz, 4H), 0.30 (d, J = 6.8 Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 171.1, 155.0, 139.4, 128.2, 128.0, 127.6, 124.5, 123.8, 123.7, 100.1, 84.8, 31.6, 20.3, 13.5; IR (KBr, cm^{-1}): 3496, 3066, 1757, 1602; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{14}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 243.1021, found $[\text{M}+\text{H}]^+$: 243.1020.

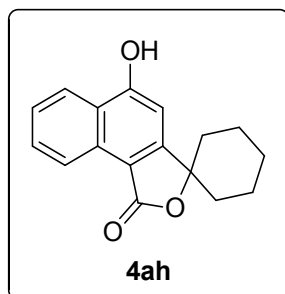
5'-Hydroxy-1'-H-spiro[cyclopentane-1,3'-naphtho[1,2-c]furan]-1'-one (4ag):



Prepared as shown in general procedure A. White solid, 51% (26 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp above 250 °C; ^1H NMR (400 MHz, DMSO- d_6): δ 11.62 (brs, 1H), 8.71 (d, J = 8.2 Hz, 1H), 8.27 (d, J = 8.3 Hz, 1H), 7.79 – 7.68 (m, 1H), 7.60 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 6.93 (s, 1H), 2.15 (d, J = 8.7 Hz, 2H), 1.96 (t, J = 5.5 Hz, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.1, 160.3, 156.6, 129.9, 129.4, 125.9, 124.7, 123.3, 109.5, 100.2, 100.1, 93.3, 38.8, 24.6; IR

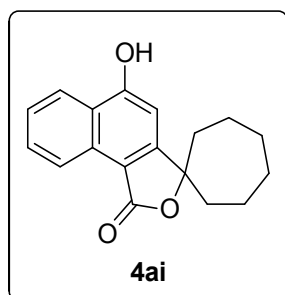
(KBr, cm^{-1}): 3129, 2960, 1701, 1574; HRMS (ESI) (m/z): Calculated for $\text{C}_{16}\text{H}_{14}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 255.1021, found $[\text{M}+\text{H}]^+$: 255.1021.

5'-Hydroxy-1'H-spiro[cyclohexane-1,3'-naphtho[1,2-c]furan]-1'-one (4ah):



Prepared as shown in general procedure A. White solid, 58% (31 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 233-235 °C; ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 8.78 – 8.69 (m, 1H), 8.27 (d, $J = 8.1$ Hz, 1H), 7.73 (ddd, $J = 8.3, 6.9, 1.3$ Hz, 1H), 7.59 (ddd, $J = 8.3, 6.9, 1.2$ Hz, 1H), 6.92 (s, 1H), 1.98 – 1.90 (m, 2H), 1.68 (dt, $J = 27.8, 13.4$ Hz, 8H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, $\text{DMSO}-d_6$): δ 169.2, 160.1, 159.1, 130.1, 129.3, 125.8, 124.7, 123.2, 122.4, 108.7, 100.4, 84.6, 35.3, 24.0, 22.1; IR (KBr, cm^{-1}): 3222, 3075, 1699, 1571; HRMS (ESI) (m/z): Calculated for $\text{C}_{17}\text{H}_{16}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 269.1178, found $[\text{M}+\text{H}]^+$: 269.1179.

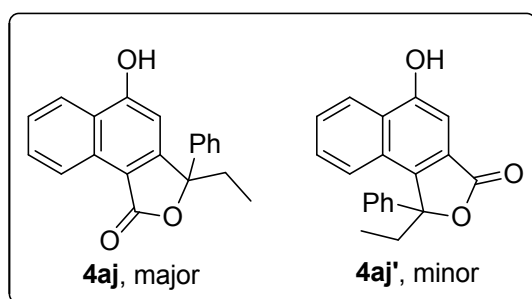
5'-Hydroxy-1'H-spiro[cycloheptane-1,3'-naphtho[1,2-c]furan]-1'-one (4ai):



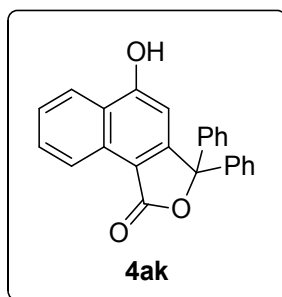
Prepared as shown in general procedure A. White solid, 66% (37 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 229-231 °C; ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 11.60 (s, 1H), 8.72 (d, $J = 8.3$ Hz, 1H),

8.26 (d, $J = 8.4$ Hz, 1H), 7.73 (t, $J = 7.5$ Hz, 1H), 7.58 (t, $J = 7.6$ Hz, 1H), 6.96 (s, 1H), 2.04 (dd, $J = 14.5, 9.8$ Hz, 2H), 1.79 (ddd, $J = 49.7, 26.5, 10.7$ Hz, 10H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.4, 160.4, 160.3, 130.1, 129.4, 125.8, 124.6, 123.2, 122.5, 108.0, 100.3, 87.9, 39.0, 28.4, 22.6; IR (KBr, cm^{-1}): 3099, 2917, 1701, 1574; HRMS (ESI) (m/z): Calculated for $\text{C}_{18}\text{H}_{18}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 283.1334, found $[\text{M}+\text{H}]^+$: 283.1338.

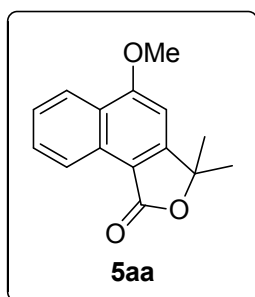
3-Ethyl-5-hydroxy-3-phenylnaphtho[1,2-c]furan-1(3H)-one (**4aj**):



Prepared as shown in general procedure A. White solid, 46% (28 mg) **4aj** and **4aj'** regio isomers obtained as inseparable mixture 80:20 ratio; R_f (30% EtOAc-Pet. ether) = 0.4; mp Charred at 200-202 °C; ^1H NMR (400 MHz, DMSO- d_6): δ major isomer peaks 11.66 (s, 0.83H), 8.76 (d, $J = 8.3$ Hz, 1H), 8.25 (d, $J = 8.4$ Hz, 1H), 7.77 (t, $J = 7.3$ Hz, 1H), 7.71 – 7.49, 6.95 (s, 1H), 0.73 (t, $J = 7.1$ Hz, 3.3H), minor isomer peaks 11.05 (s, 0.29H), 8.33 (d, $J = 8.3$ Hz, 0.39H), 7.14 (s, 0.4H), 0.52 (t, $J = 7.0$ Hz, 1.1H), merged peaks 7.71 – 7.49 (m, 4.68H), 7.36 (ddd, $J = 13.9, 12.7, 7.3$ Hz, 5.36H), 2.93 – 2.69 (m, 0.8H), 2.59 – 2.47 (m, 3.0H), 2.38 (dd, $J = 14.3, 7.3$ Hz, 1.1H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 170.3, 169.8, 160.6, 157.1, 155.6, 140.6, 140.4, 139.4, 129.9, 129.7, 128.9, 128.7, 128.3, 128.1, 128.0, 127.1, 126.5, 126.2, 125.0, 124.7, 124.6, 124.3, 123.9, 123.3, 122.5, 108.8, 101.0, 100.1, 90.2, 88.3, 30.8, 28.6, 7.94, 7.91, 7.6; IR (KBr, cm^{-1}): 3278, 3065, 1718, 1576; HRMS (ESI) (m/z): Calculated for $\text{C}_{20}\text{H}_{16}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 305.1178, found $[\text{M}+\text{H}]^+$: 305.1178.

5-Hydroxy-3,3-diphenylnaphtho[1,2-c]furan-1(3H)-one (4ak):

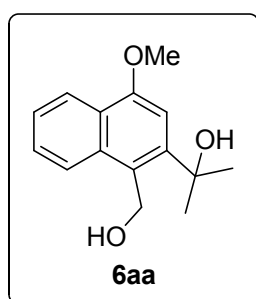
Prepared as shown in general procedure A. brown solid, 41% (29 mg); R_f (30% EtOAc-Pet. ether) = 0.4; mp above 250 °C ^1H NMR (400 MHz, DMSO- d_6): δ 11.23 (s, 0.49H) 8.35 (d, J = 8.4 Hz, 1H), 7.65 (ddd, J = 8.3, 6.5, 1.6 Hz, 1H), 7.59 – 7.52 (m, 2H), 7.41 – 7.36 (m, 6H), 7.27 – 7.22 (m, 4H), 7.19 (s, 1H); ^{13}C $\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6): δ 169.9, 156.2, 140.8, 139.1, 128.9, 128.6, 128.4, 128.32, 128.3, 128.0, 125.2, 124.0, 123.8, 100.5, 92.2; IR (KBr, cm^{-1}): 3278, 3065, 1718, 1576; HRMS (ESI) (m/z): Calculated for $\text{C}_{20}\text{H}_{16}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 305.1178, found $[\text{M}+\text{H}]^+$: 305.1178.

5-Methoxy-3,3-dimethylnaphtho[1,2-c]furan-1(3H)-one (5aa):

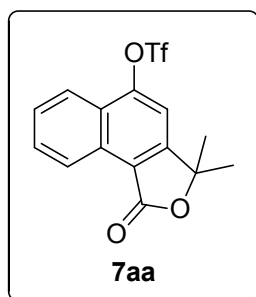
Naphthol derivative **3aa** (0.4 mmol) and potassium carbobate (0.8 mmol) are stirred in a 10 ml round bottom flask for 30 min. Added iodomethane (1.2 mmol) and stirred for 4 h. On completion of reaction (monitored by TLC), reaction mixture is filtered through celite bed. Filtrate is distilled and purified by flash column chromatography. White solid, 83% (92 mg); R_f (10% EtOAc-Pet. ether) = 0.6; mp 202-204 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.93 (d, J = 8.3

Hz, 1H), 8.32 (d, $J = 8.5$ Hz, 1H), 7.77 – 7.66 (m, 1H), 7.58 (ddd, $J = 8.3, 7.0, 1.2$ Hz, 1H), 6.69 (s, 1H), 4.12 (s, 3H), 1.71 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 161.7, 158.9, 130.4, 129.6, 126.5, 125.7, 123.7, 123.0, 111.5, 95.6, 83.8, 56.3, 27.2; IR (Neat, cm^{-1}): 2978, 1793, 1578; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{14}\text{O}_3\text{H}$ $[\text{M}+\text{H}]^+$: 243.1021, found $[\text{M}+\text{H}]^+$: 243.1022.

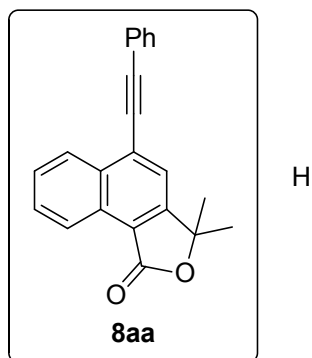
2-(1-(Hydroxymethyl)-4-methoxynaphthalen-2-yl)propan-2-ol (6aa):



Prepared using the following procedure. LAH is added (0.4 mmol) to dry THF (2 ml) at 0 °C and stirred for five minute under argon condition. Added solution of **5aa**(0.2 mmol in 2 ml dry THF) drop wise at 0 °C. Reaction mixture is stirred for 2 h at rt under argon condition. On completion of reaction (monitored by TLC), reaction mixture is quenched with 10% of sodium hydroxide (2 ml) and filtered. Filtrate is extracted with ethylacetate (10 ml 2 times), dried with anhydrous sodium sulfate and distilled. The crude product is purified by flash column chromatography. Yellow liiquid, 85% (42 mg); R_f (30% EtOAc-Pet. ether) = 0.5; mp 145-147 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.29 – 8.15 (m, 2H), 7.53 (t, $J = 7.5$ Hz, 1H), 7.44 (t, $J = 7.5$ Hz, 1H), 6.76 (d, $J = 10.3$ Hz, 1H), 5.26 (s, 2H), 3.97 (s, 3H), 2.93 (brs, 2H), 1.70 (s, 6H); ^{13}C NMR (101 MHz, CDCl_3) δ : ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$) δ 155.0, 144.8, 134.6, 127.4, 125.8, 125.1, 125.0, 124.1, 122.1, 102.2, 75.0, 58.2, 55.4, 32.9; IR (Neat, cm^{-1}): 3415, 2973, 1593; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{18}\text{O}_3\text{Na}$ $[\text{M}+\text{Na}]^+$: 269.1154, found $[\text{M}+\text{Na}]^+$: 269.1154.

3,3-Dimethyl-1-oxo-1,3-dihydronaphtho[1,2-c]furan-5-yl trifluoromethanesulfonate (7aa):

Prepared using following procedure. Naphthol **3aa** (0.2 mmol) was dissolved in DCM (5.0 mL) and then cooled to $-78\text{ }^{\circ}\text{C}$. Triethylamine (3 mmol) was added, followed by addition of trifluoromethanesulfonic anhydride (TF_2O , 208.0 μL , 1.5 mmol) within 5 minutes via a syringe. The resulting solution was gradually warmed to room temperature and stirred for 2 h until **3aa** had been completely consumed as determined by TLC. The solvent was then removed under reduced pressure to get the crude product, which was purified by flash column chromatography. White solid, 90% (65%); R_f (10% EtOAc-Pet. ether) = 0.5; mp $202\text{--}204\text{ }^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3) δ 9.10 (d, $J = 8.3\text{ Hz}$, 1H), 8.17 (d, $J = 8.4\text{ Hz}$, 1H), 7.90 – 7.82 (m, 1H), 7.82 – 7.75 (m, 1H), 7.46 (s, 1H), 1.76 (s, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 168.7, 156.0, 150.2, 130.9, 130.7, 129.0, 126.3, 124.3, 121.8, 119.3, 118.8 (q, $J = 320.5\text{ Hz}$), 110.2 (d, $J = 4.0\text{ Hz}$), 84.5, 27.0; IR (Neat, cm^{-1}): 2984, 1756, 1640; HRMS (ESI) (m/z): Calculated for $\text{C}_{15}\text{H}_{11}\text{F}_3\text{O}_5\text{SH}$ $[\text{M}+\text{H}]^+$: 361.0358, found $[\text{M}+\text{H}]^+$: 361.0356.

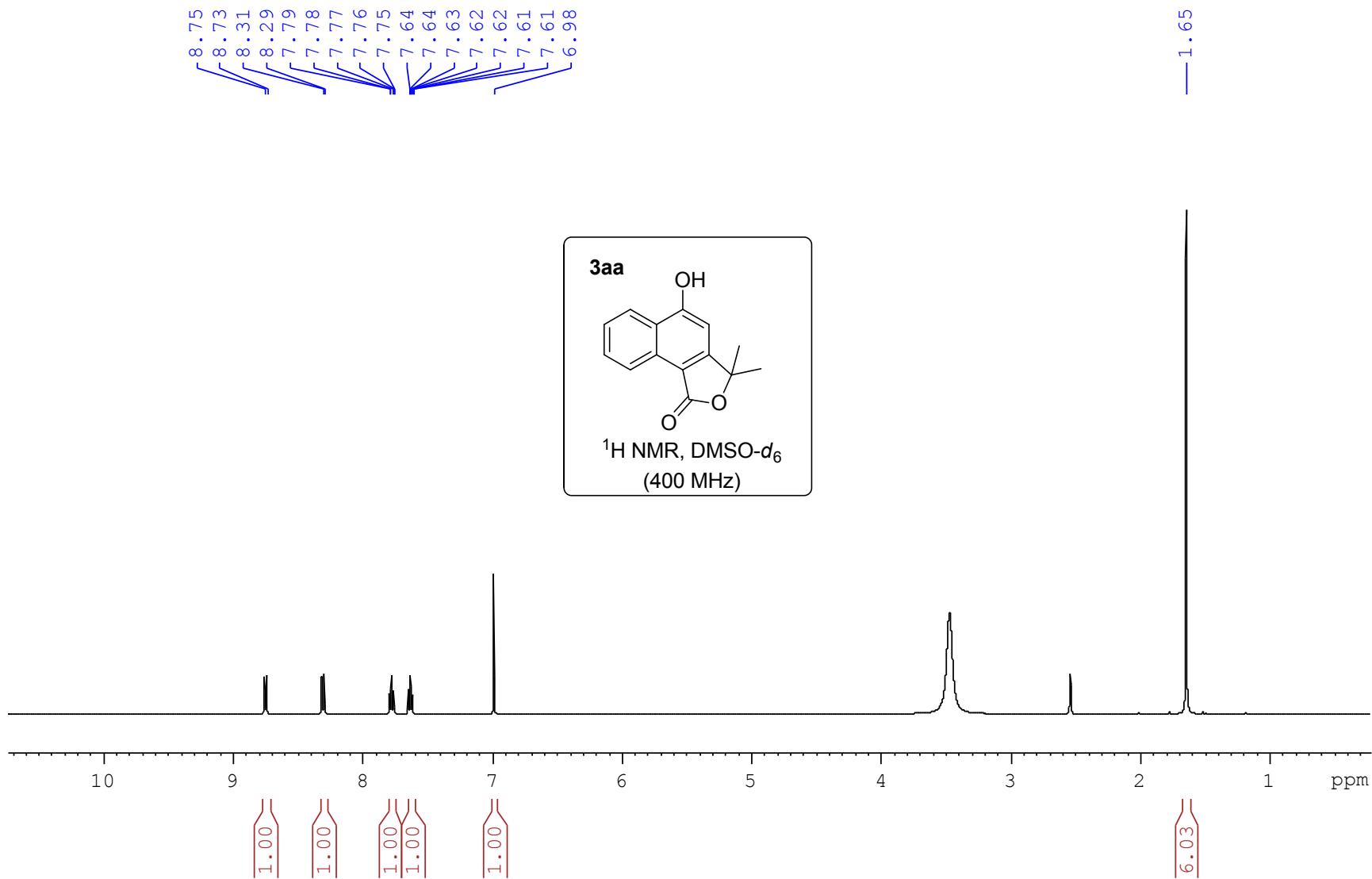
3,3-Dimethyl-5-(phenylethynyl)naphtho[1,2-c]furan-1(3H)-one (8aa):

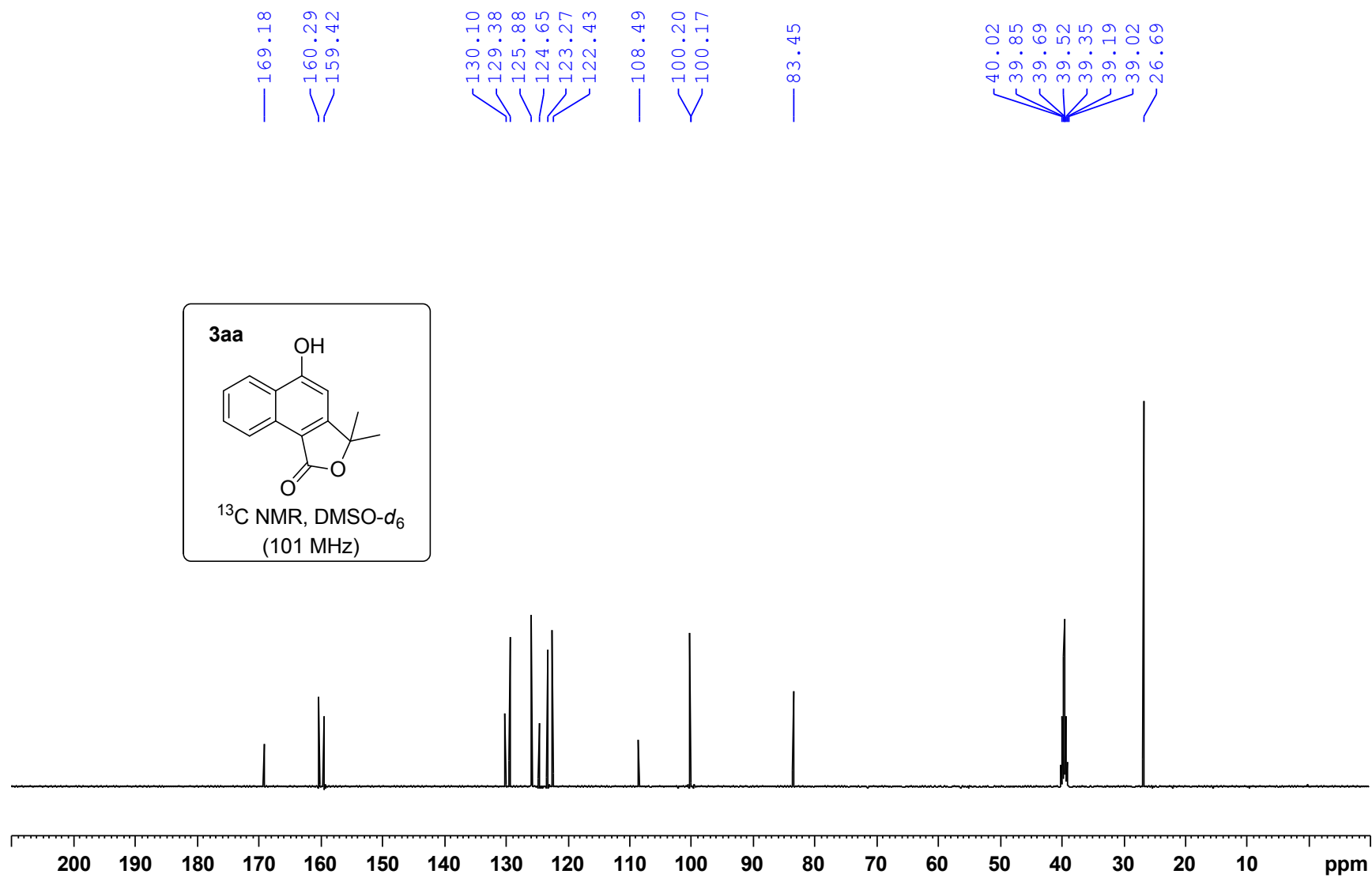
Prepared by the following procedure. **7aa** (0.1 mmol), phenylacetylene (0.3 mmol), bis(triphenylphosphine)palladium(II)dichloride (0.01 mmol), copper(I) bromide (0.005 mmol), diisopropylethylamine (0.3 mmol), and DMF (1.0 mL) were added to a screw cap tube. The reaction mixture was heated to 80 °C under N₂ condition for 5 h until **7aa** on completion of reaction (checked by TLC), the reaction mixture was cooled down to room temperature and then diluted with ethyl acetate (30 mL). The organic phase was washed with brine (4x10 mL) and dried with NaSO₄. The solvent was removed under reduced pressure to get the crude product, which was purified by flash column chromatography. Yellow liquid, 90% (28 mg); *R_f* (10% EtOAc-Pet. ether) = 0.8; ¹H NMR (400 MHz, CDCl₃) δ 9.10 – 9.03 (m, 1H), 8.54 (d, *J* = 7.9 Hz, 1H), 7.80 – 7.70 (m, 3H), 7.68 (dd, *J* = 6.7, 3.0 Hz, 2H), 7.47 – 7.40 (m, 3H), 1.74 (s, 6H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ: 169.7, 155.5, 133.2, 132.0, 131.9, 129.3, 128.7, 128.6, 127.9, 126.9, 124.1, 122.4, 121.8, 121.7, 119.0, 98.1, 86.9, 84.2, 27.1, 27.1; IR (Neat, cm⁻¹): 3062, 2980, 2206, 1756, 1580; HRMS (ESI) (*m/z*): Calculated for C₂₂H₁₆O₂H [M+H]⁺: 313.1229, found [M+H]⁺: 313.1229.

References:

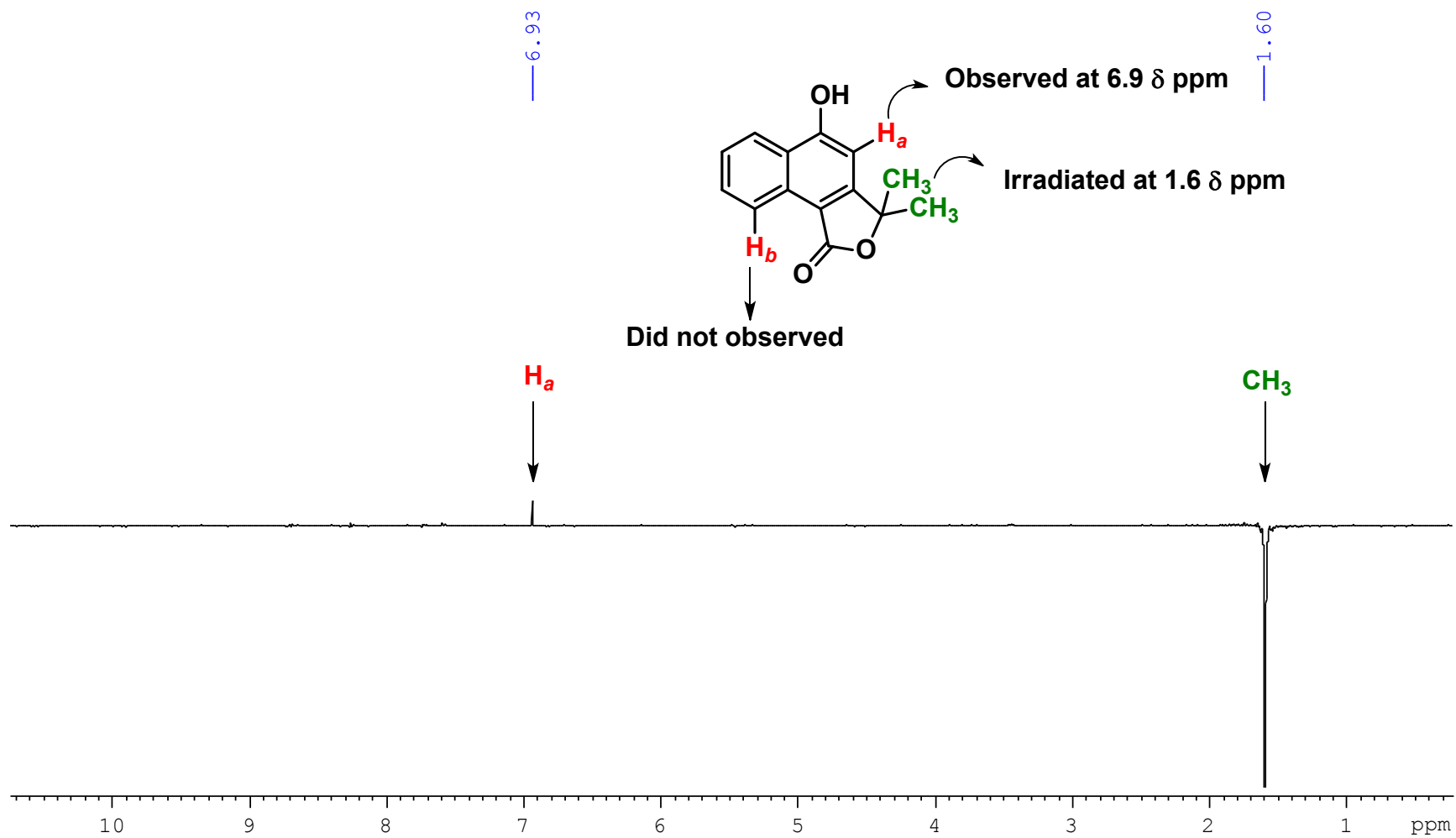
- 1) Liao, G.; Song, H.; Yin, X.-S.; Shi, B.-F. Expeditious Synthesis of Pyrano[2,3,4-de]Quinolines via Rh(III)-Catalyzed Cascade C–H Activation/Annulation/Lactonization of Quinolin-4-Ol with Alkynes. *Chem. Commun.* **2017**, *53*, 7824–7827.
- 2) Xu, Y.; Yang, X.; Zhou, X.; Kong, L.; Li, X. Rhodium(III)-Catalyzed Synthesis of Naphthols via C–H Activation of Sulfoxonium Ylides. *Org. Lett.* **2017**, *19*, 4307–4310.

¹H and ¹³C Spectra





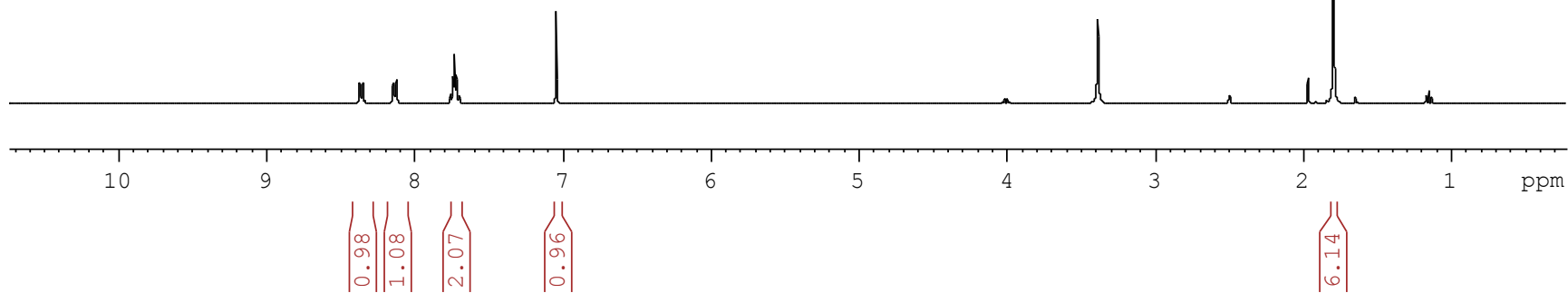
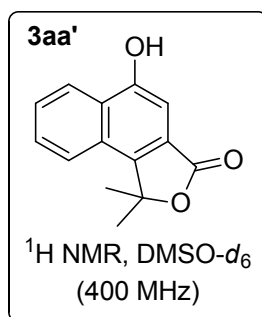
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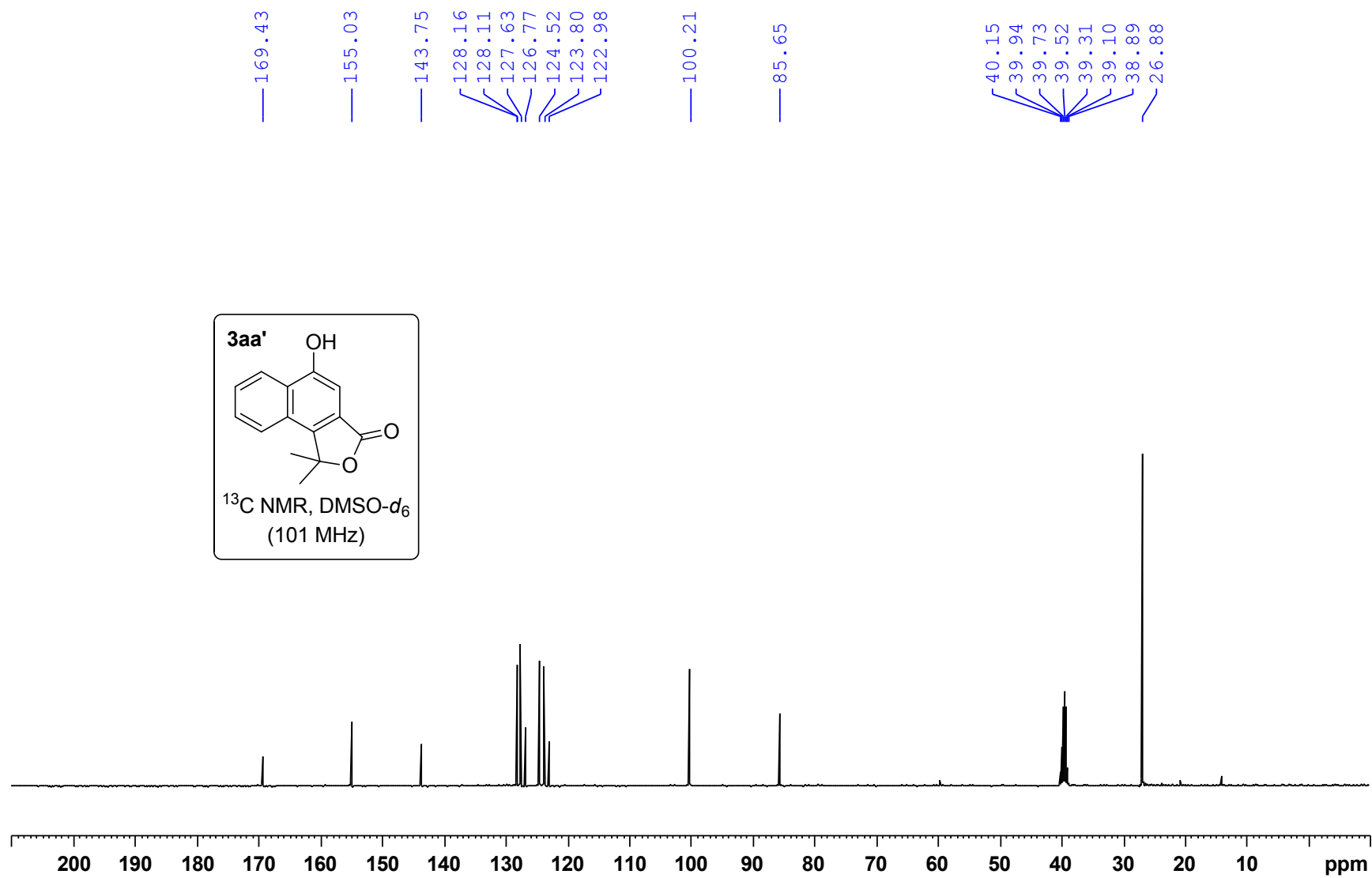


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8.36
8.35
8.35
8.34
8.14
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7.72
7.71
7.71
7.70
7.04

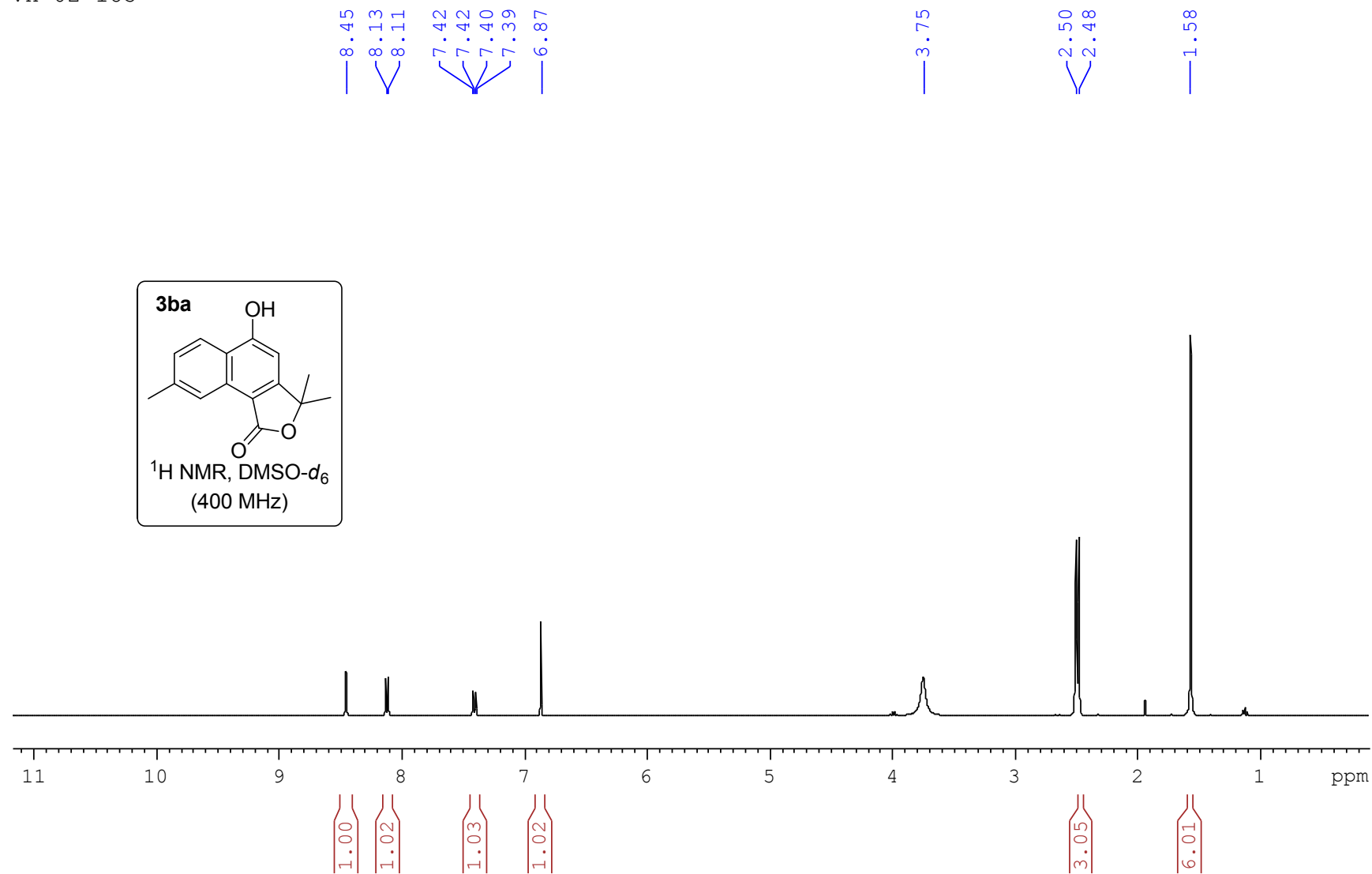
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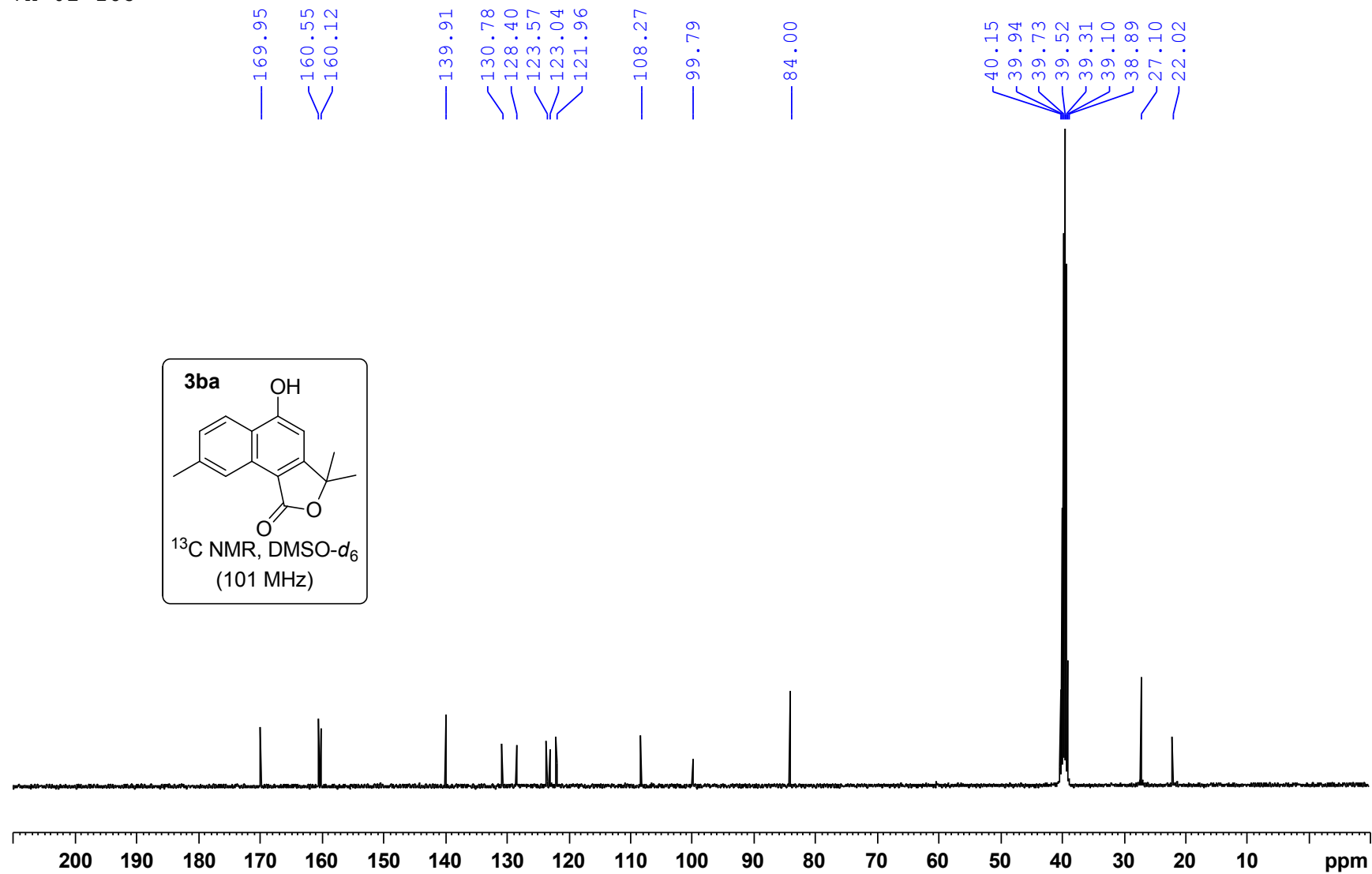




VH-02-185



VH-02-185



VH-02-195

11.52

8.62
8.60

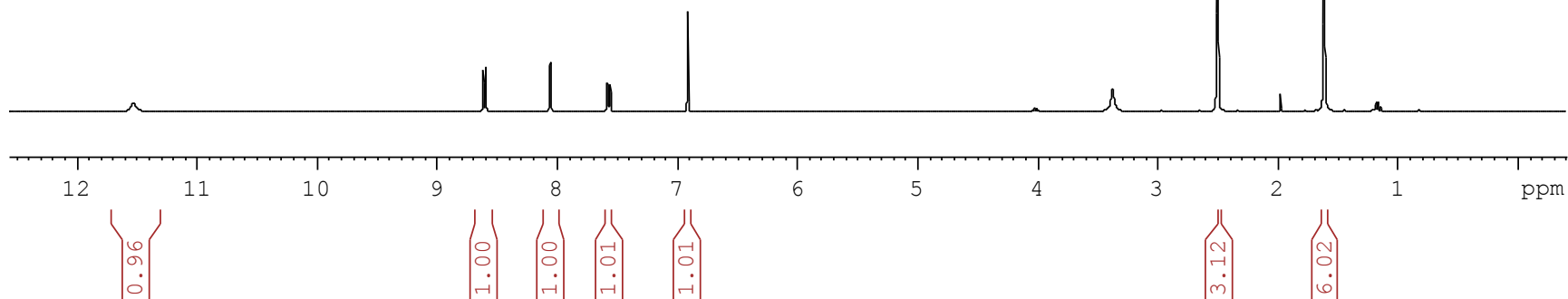
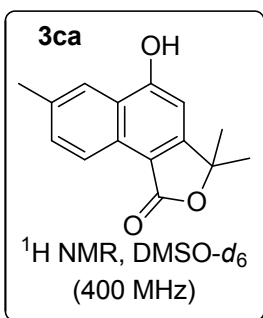
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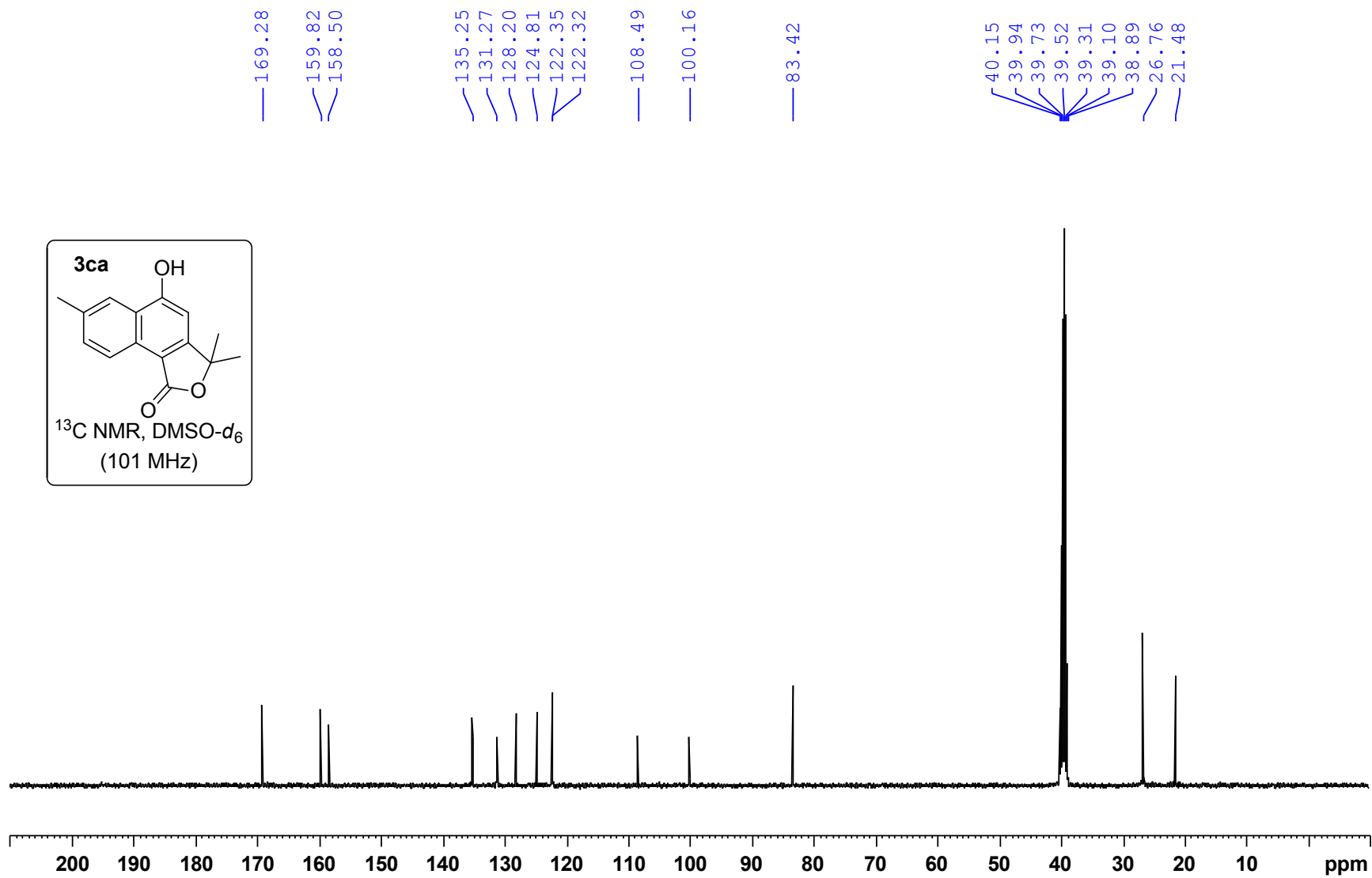
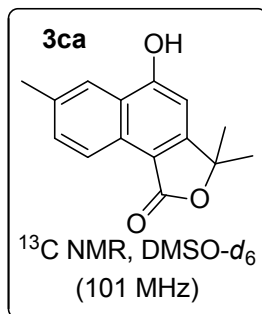
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7.56

6.92

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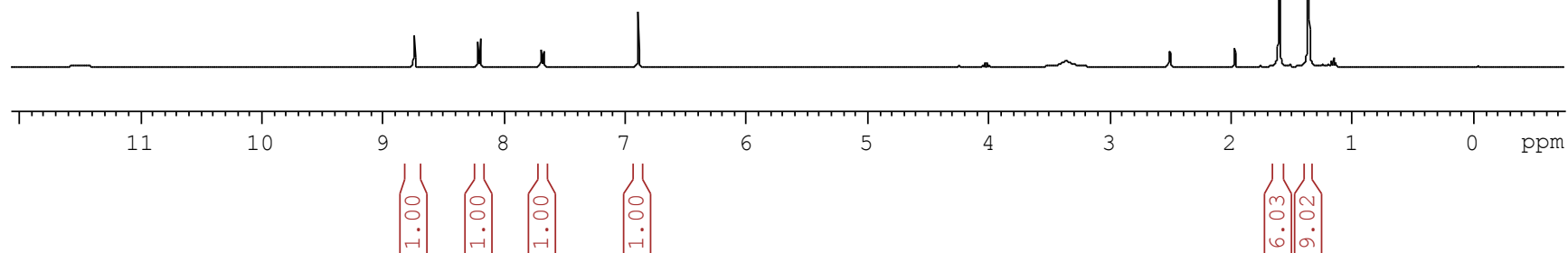
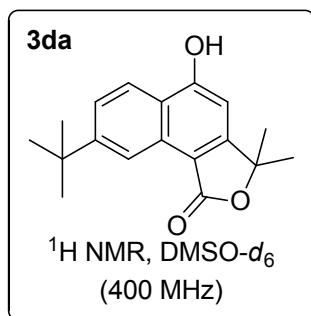
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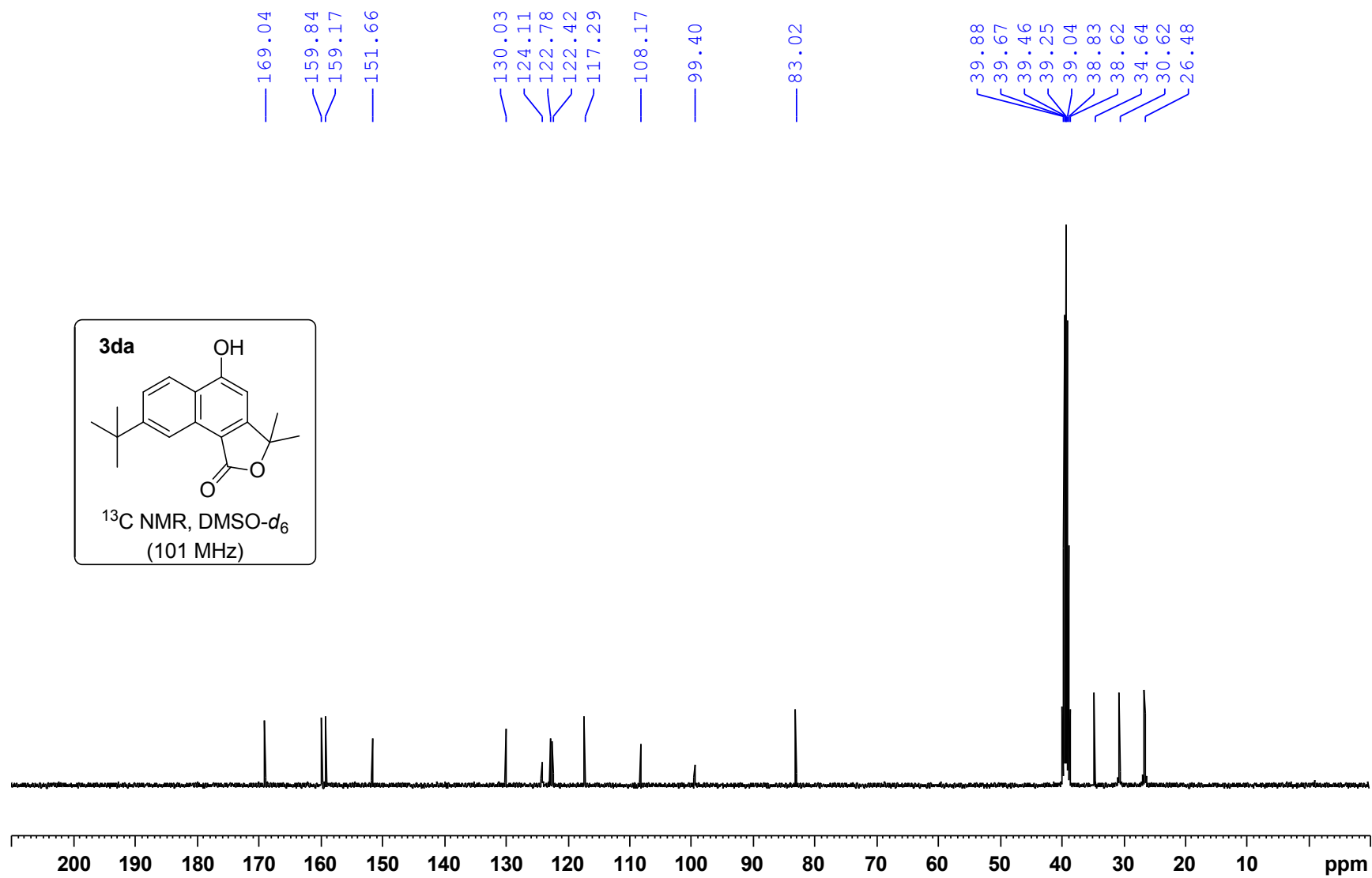
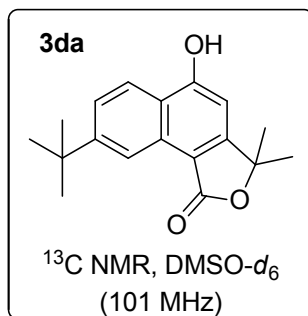




8.74
8.73
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8.19
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7.69
7.67
7.66
6.89

2.50
1.61
1.37





VH-02-183

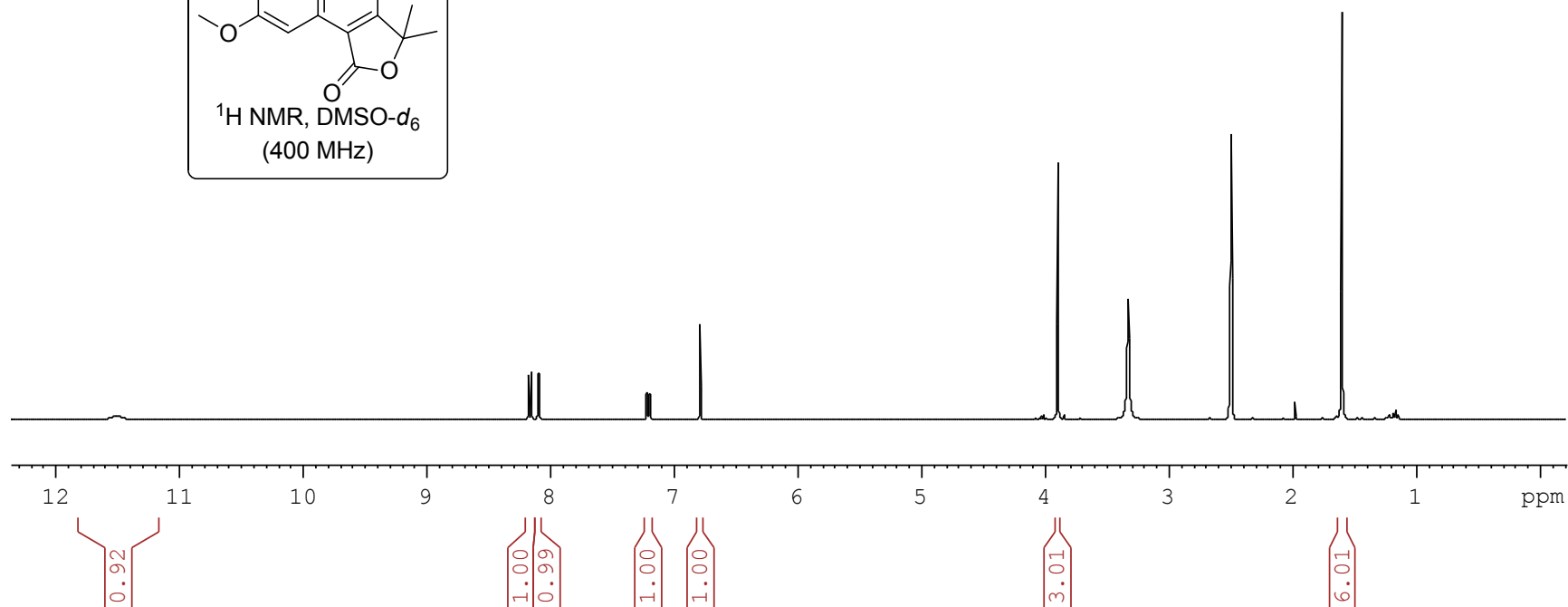
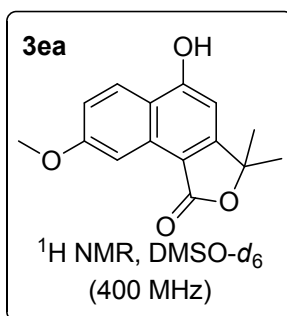
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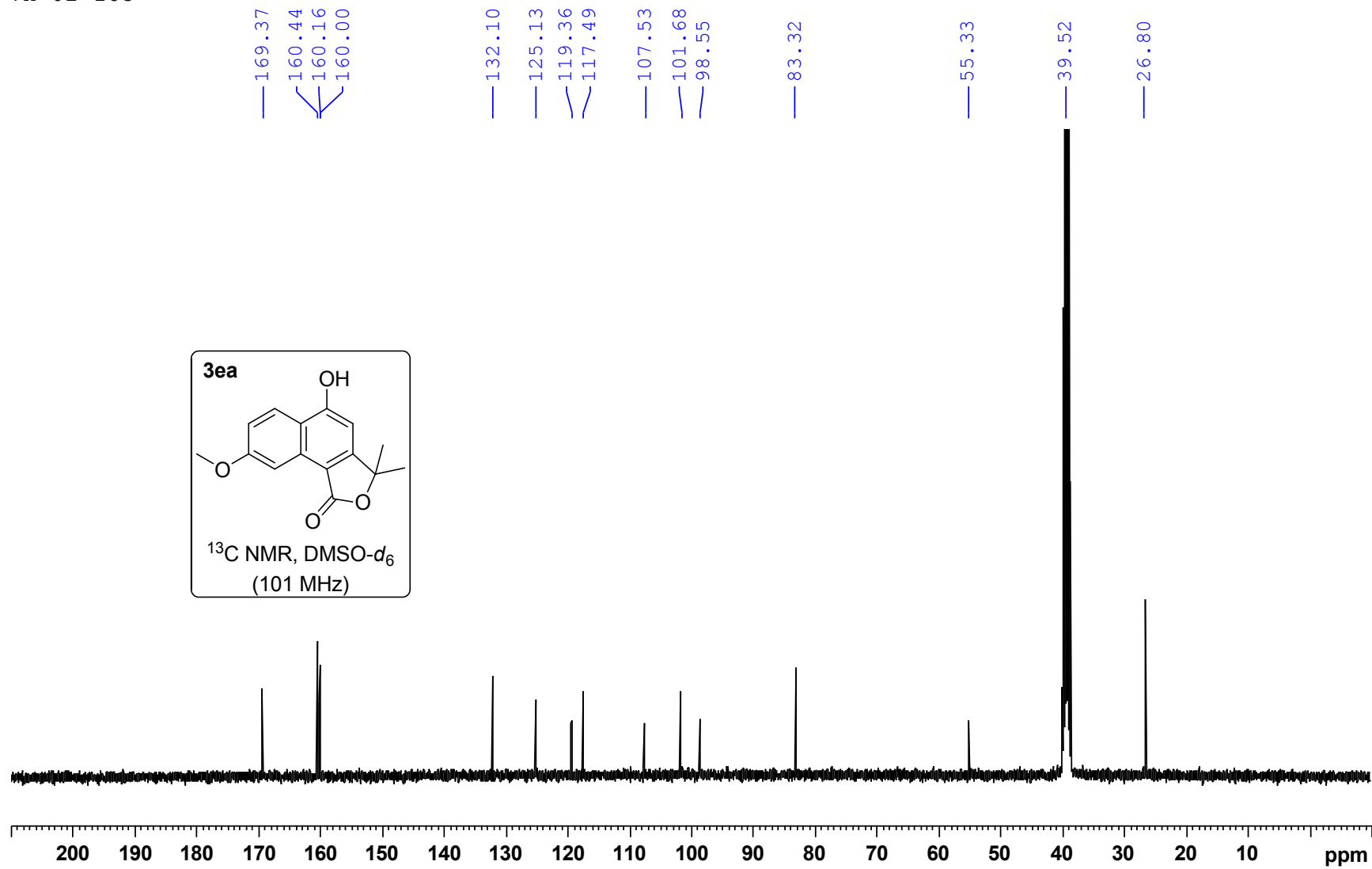
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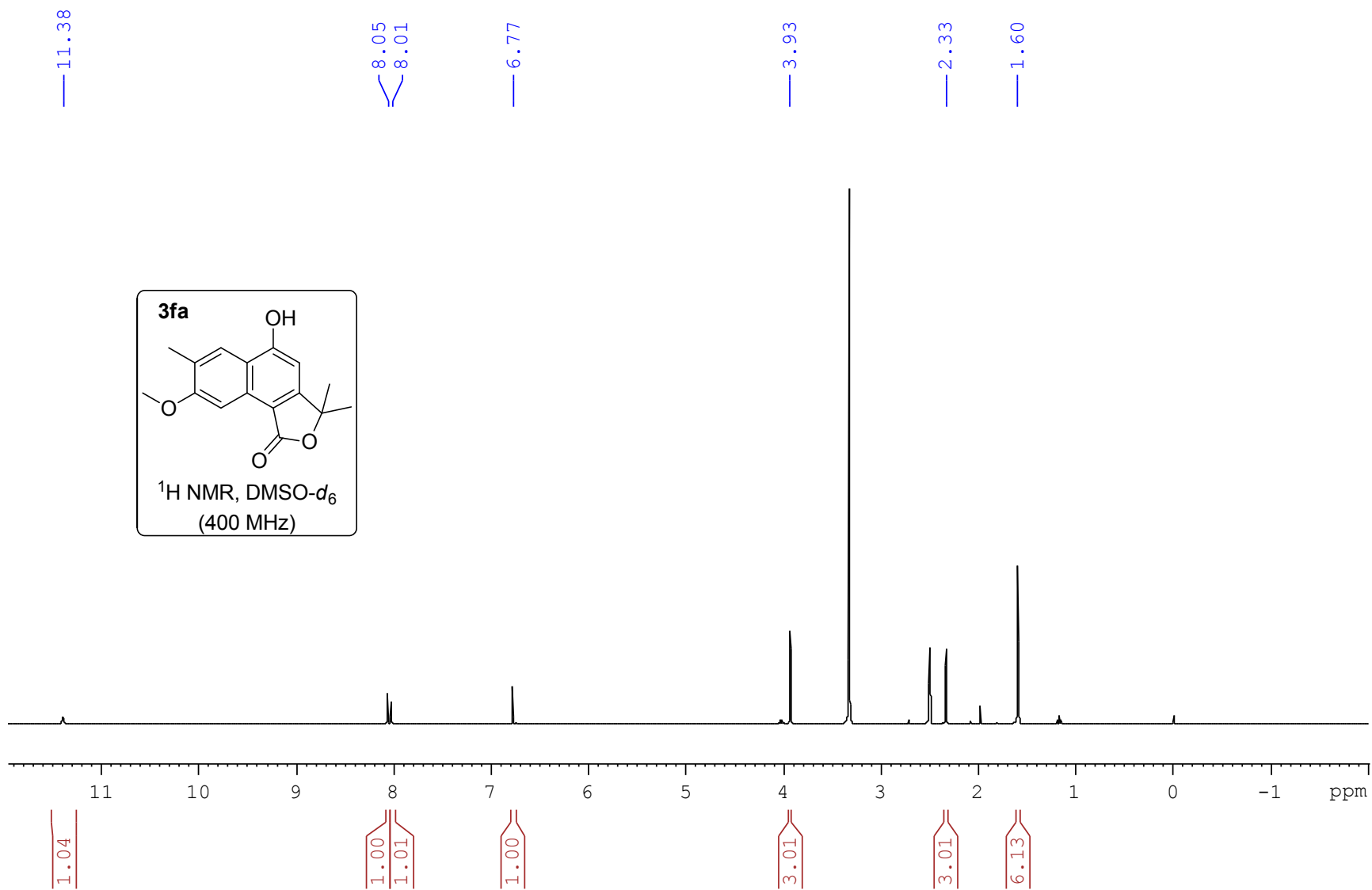
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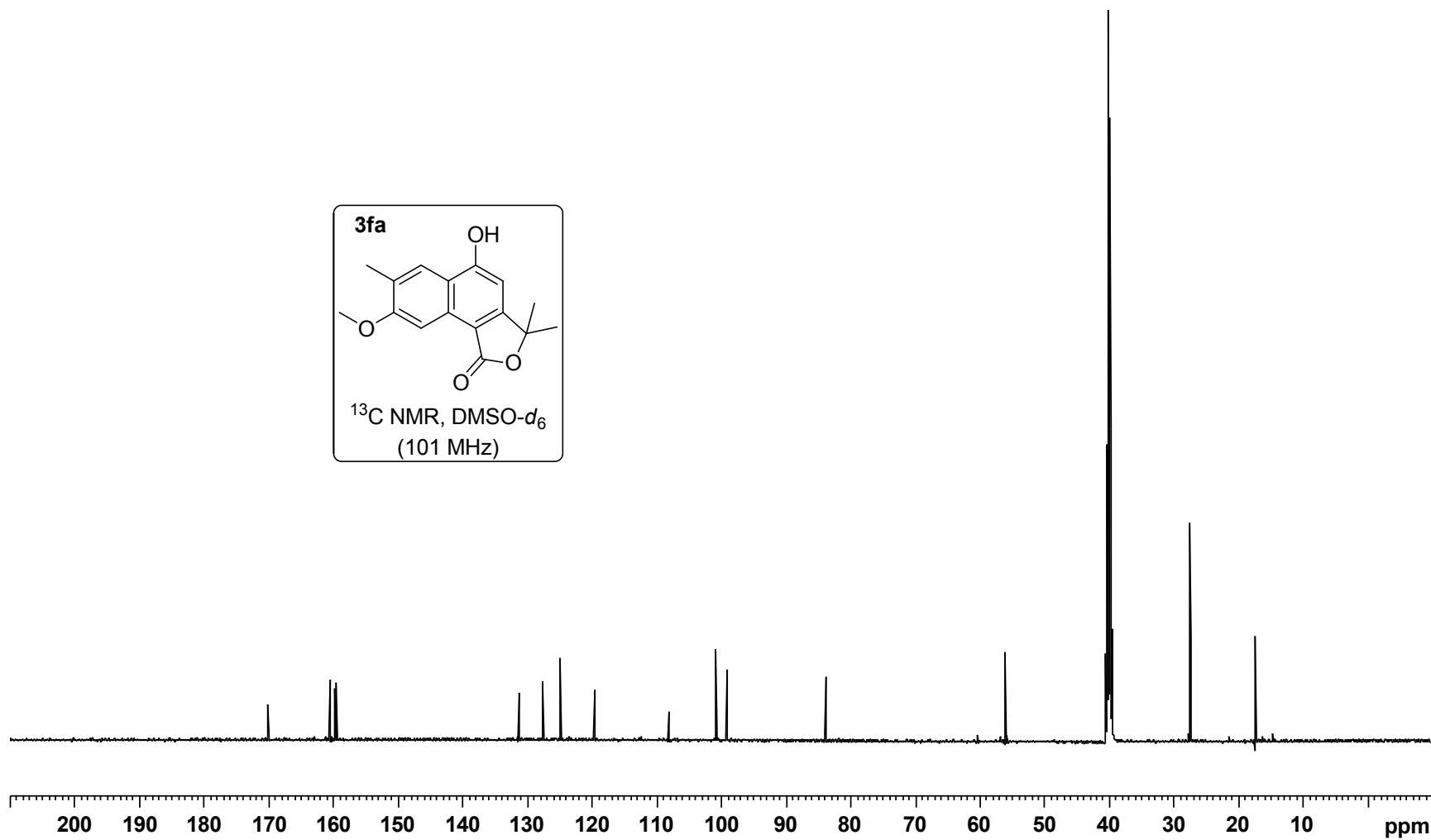
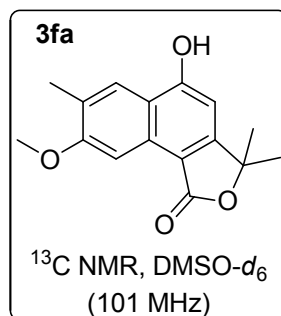
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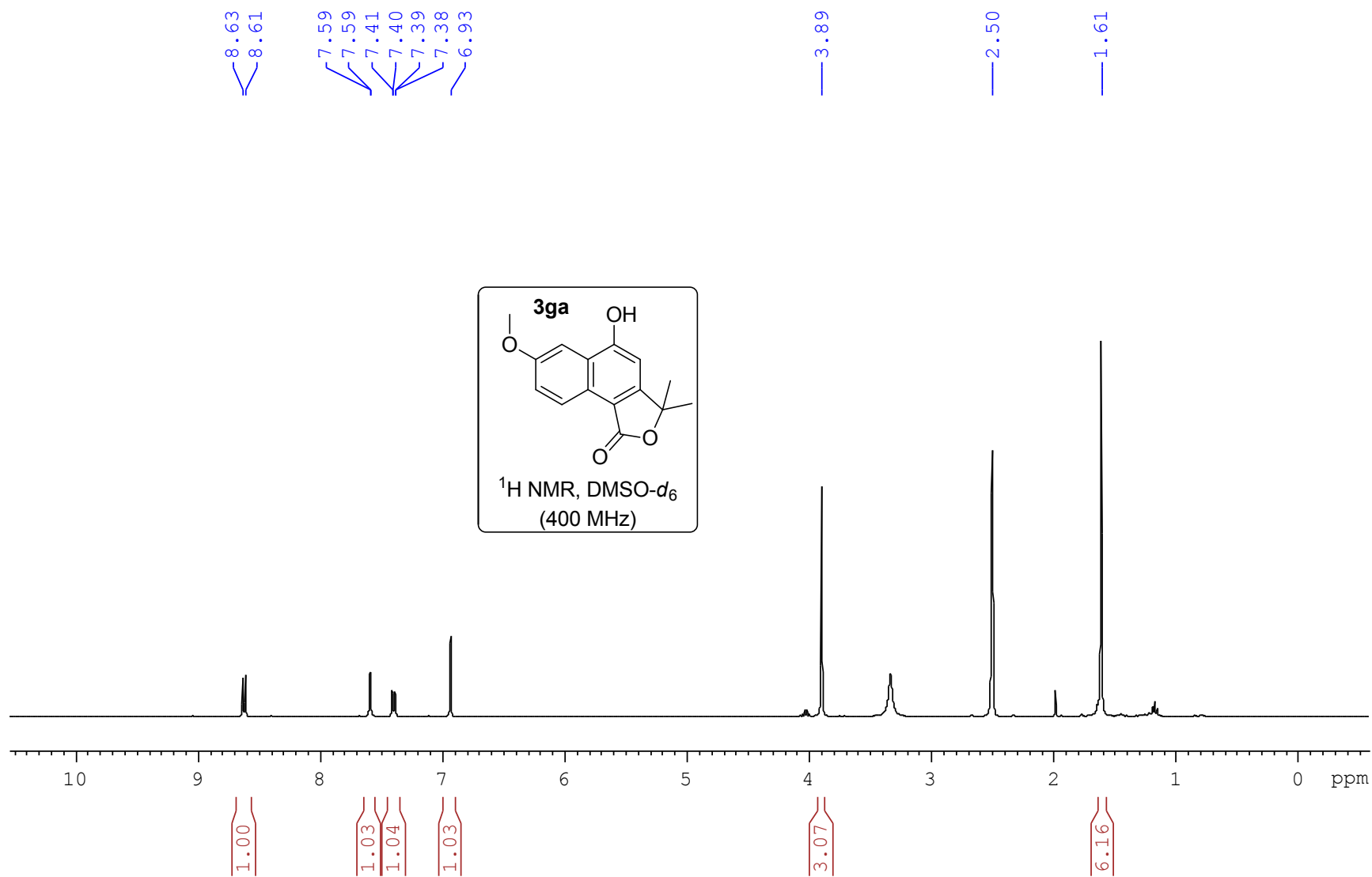


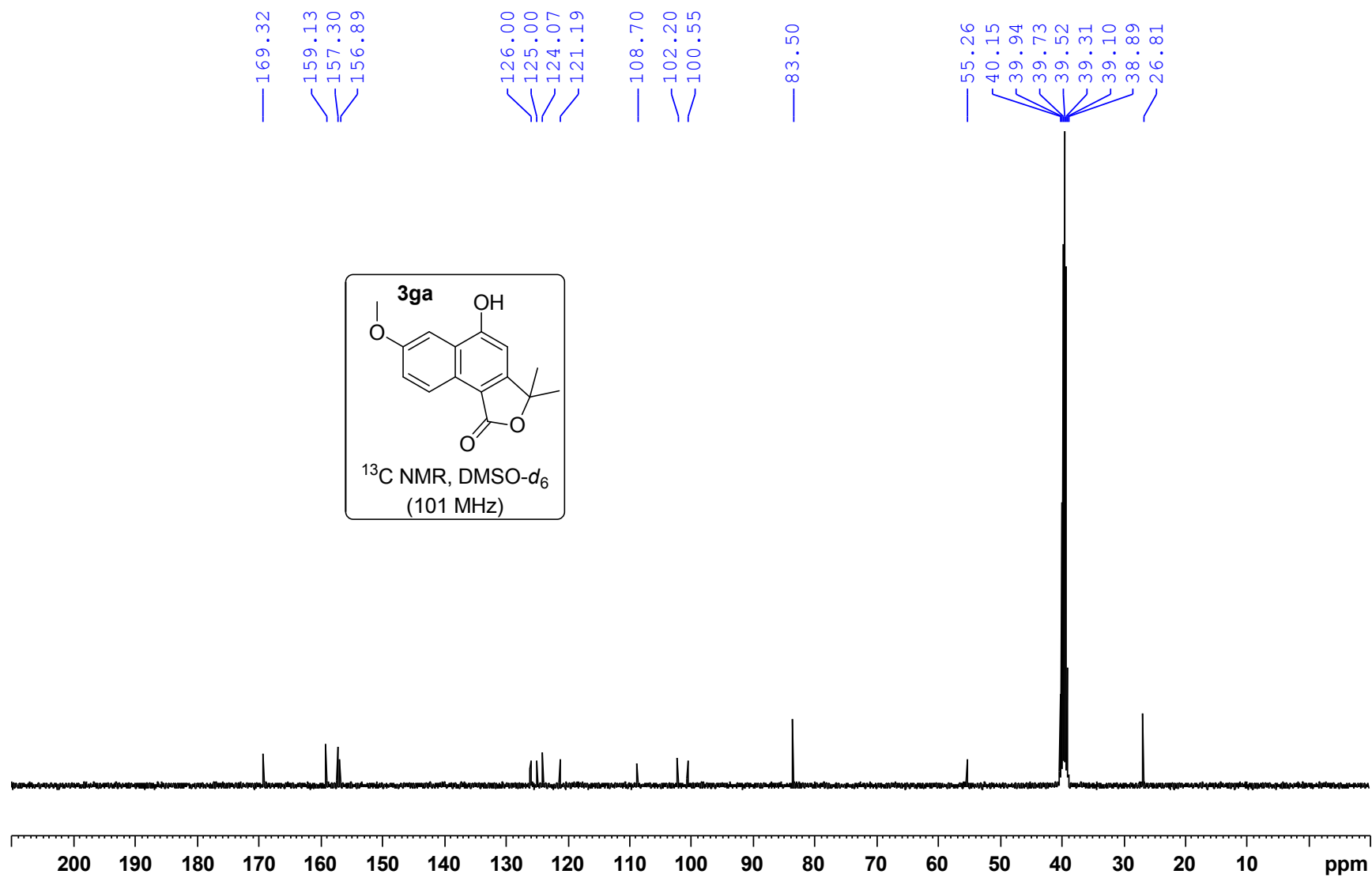
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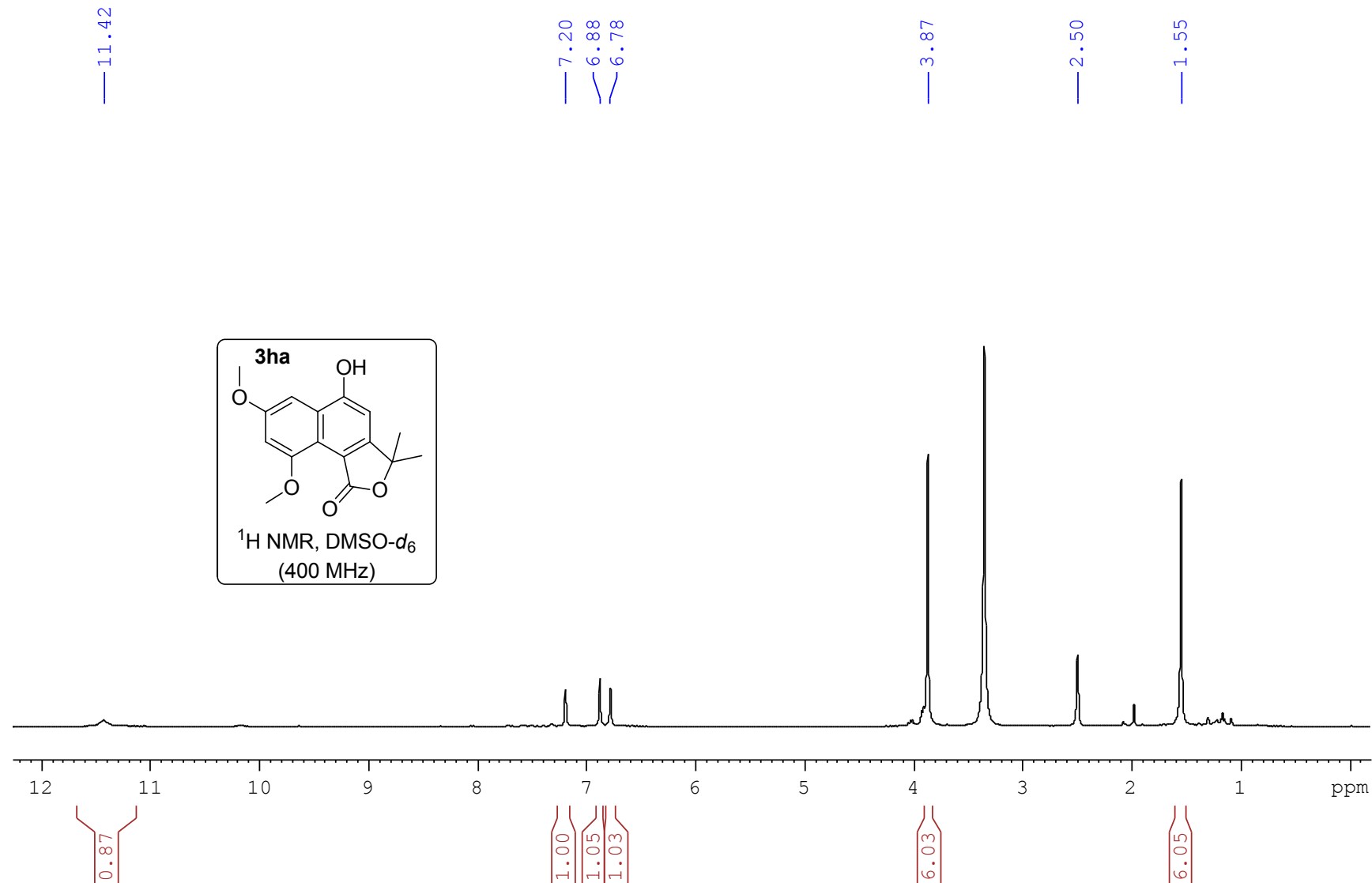




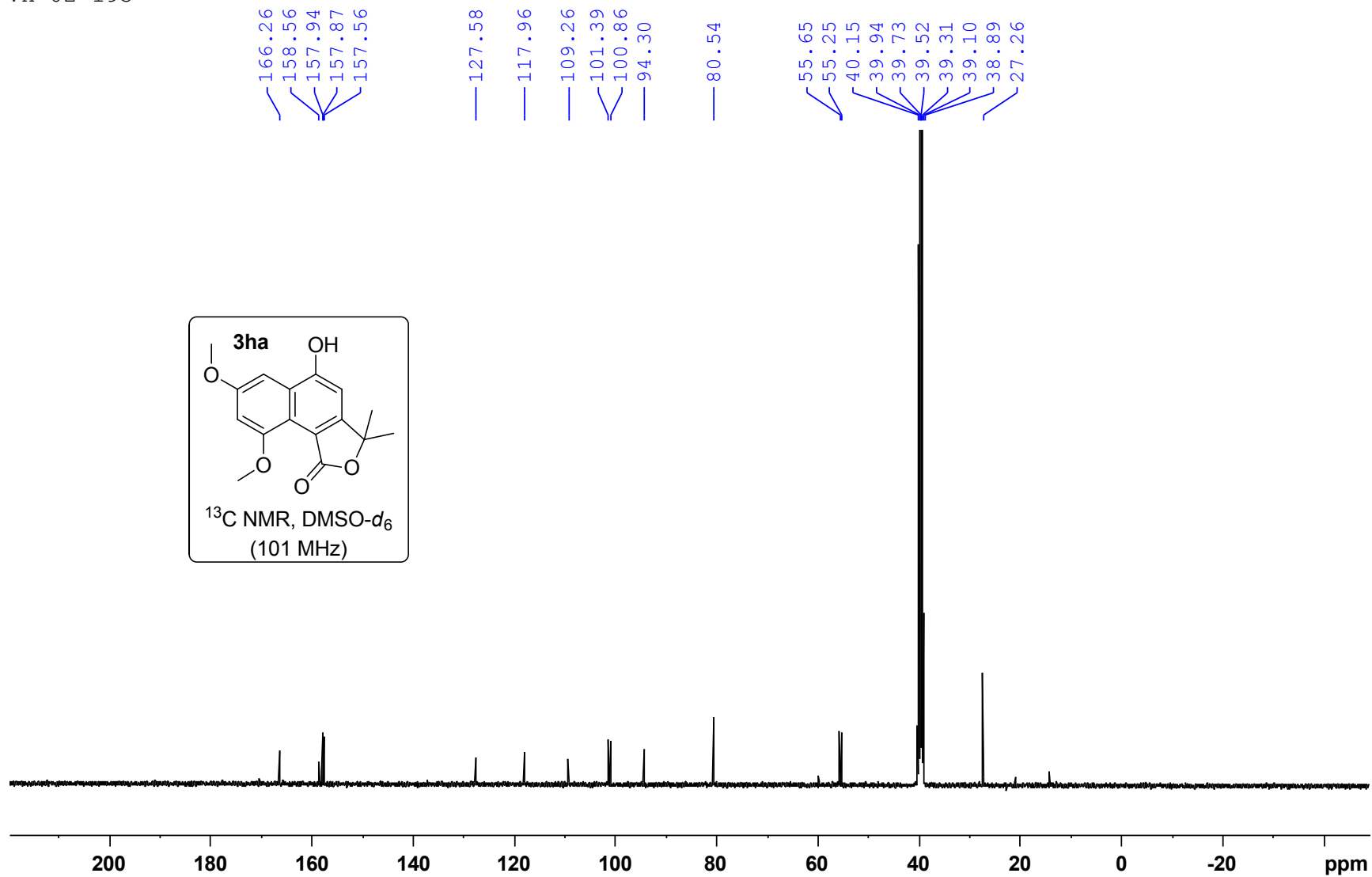




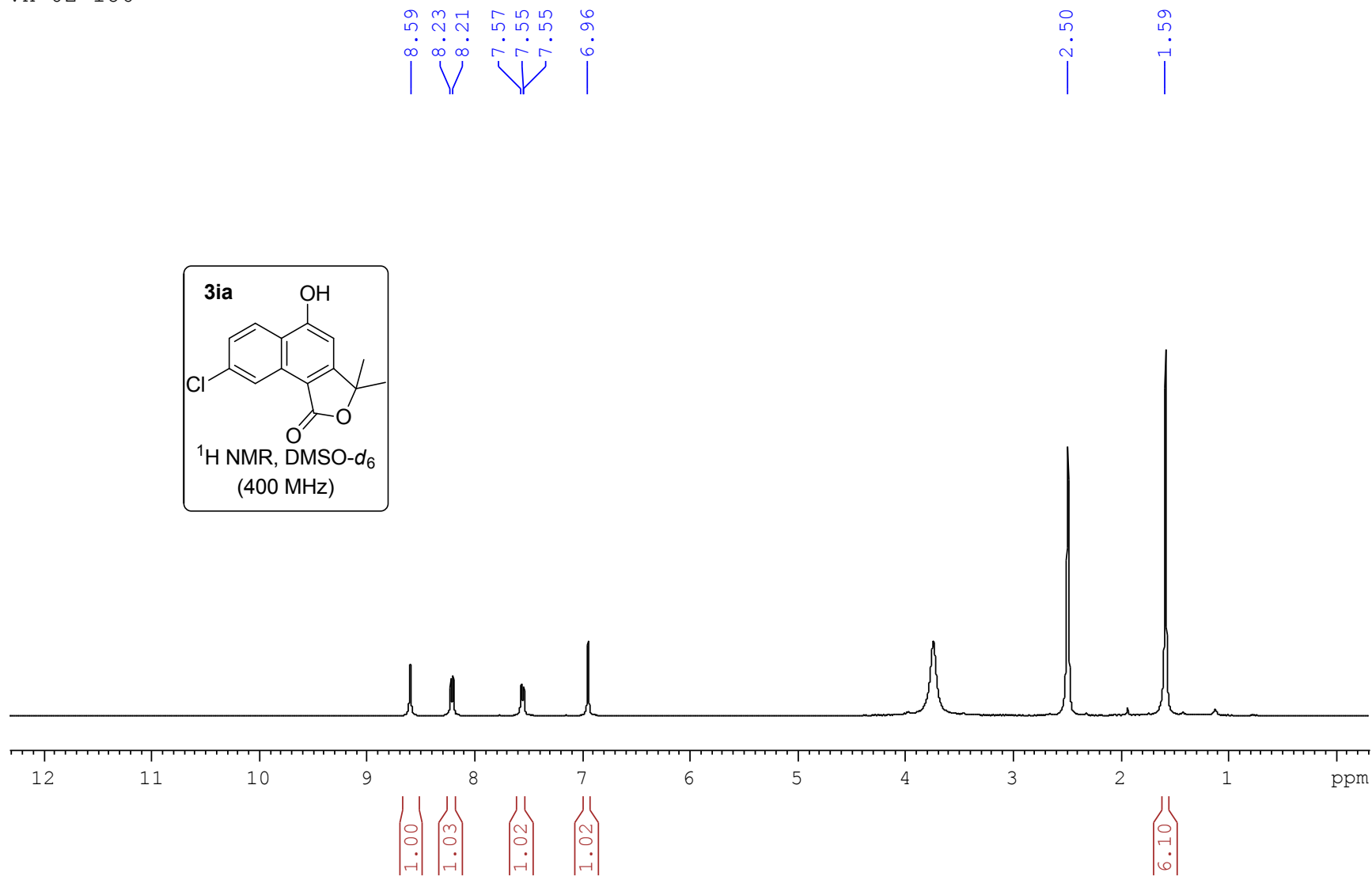
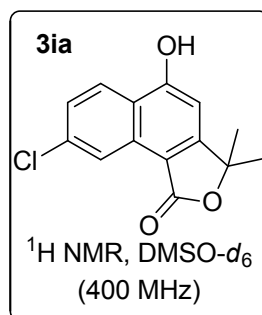
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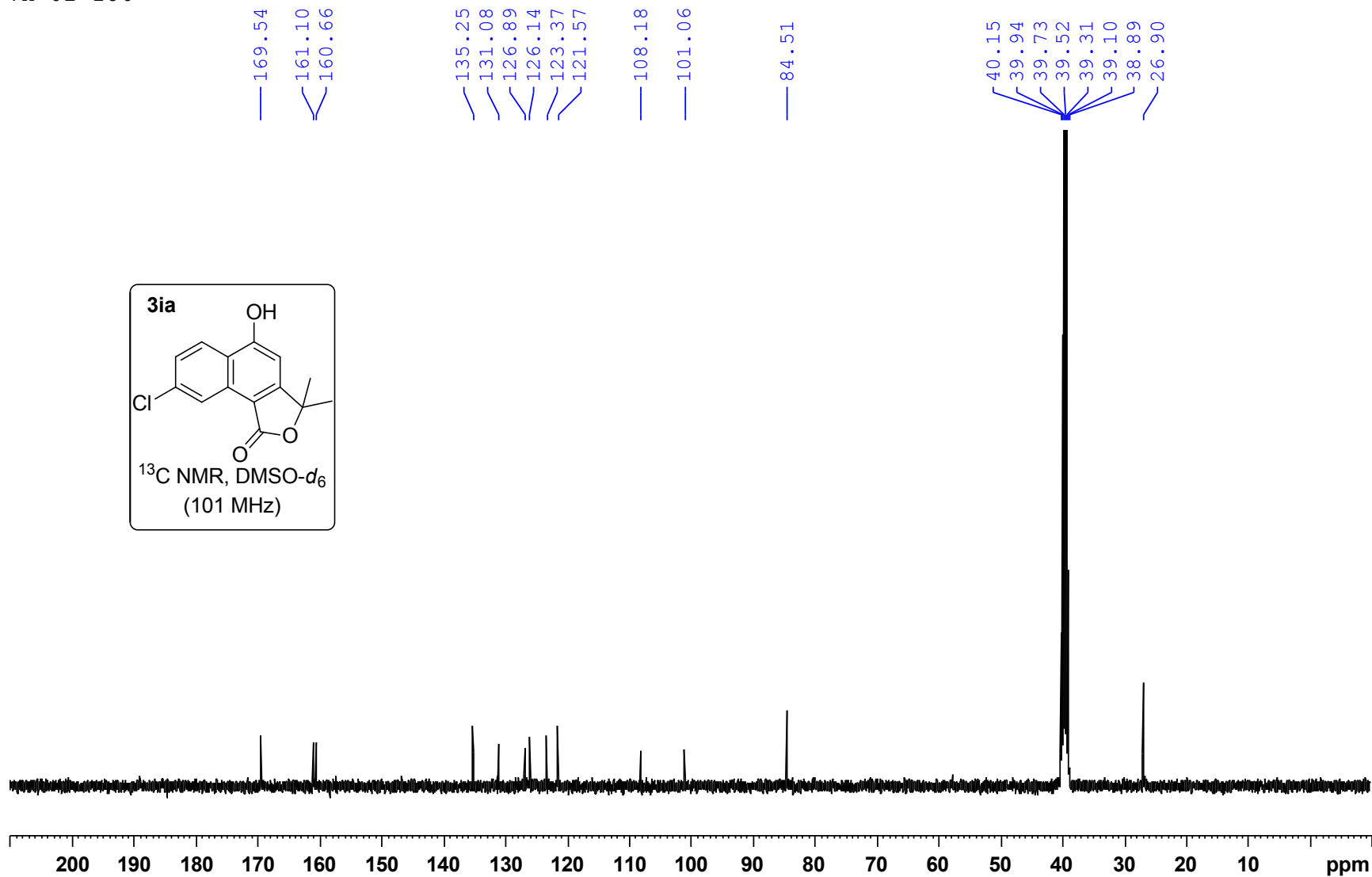
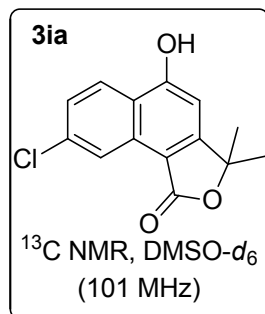
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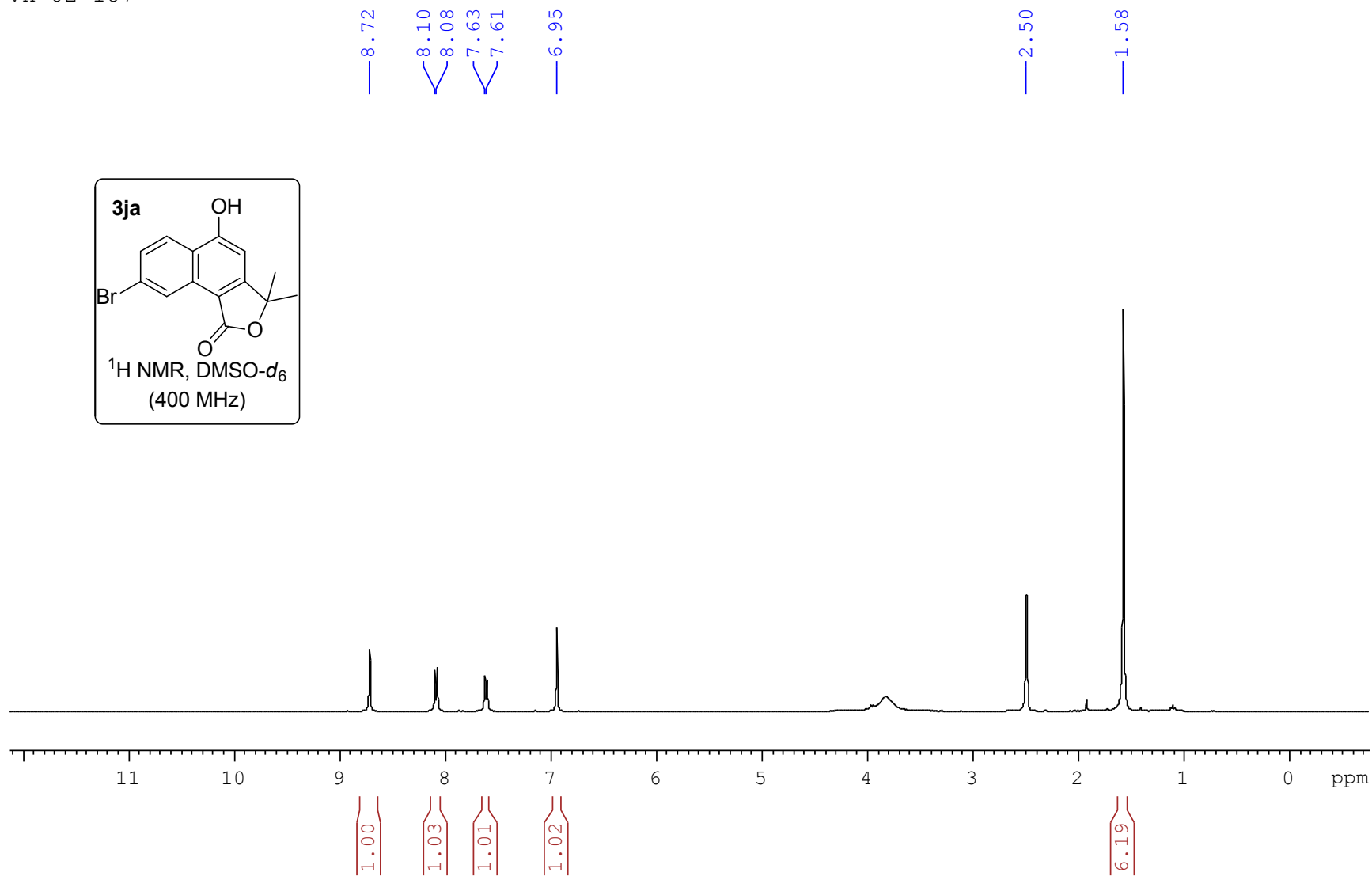
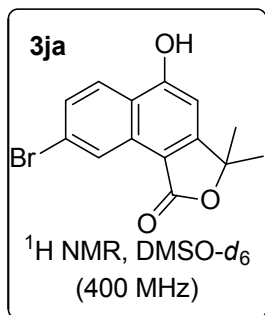
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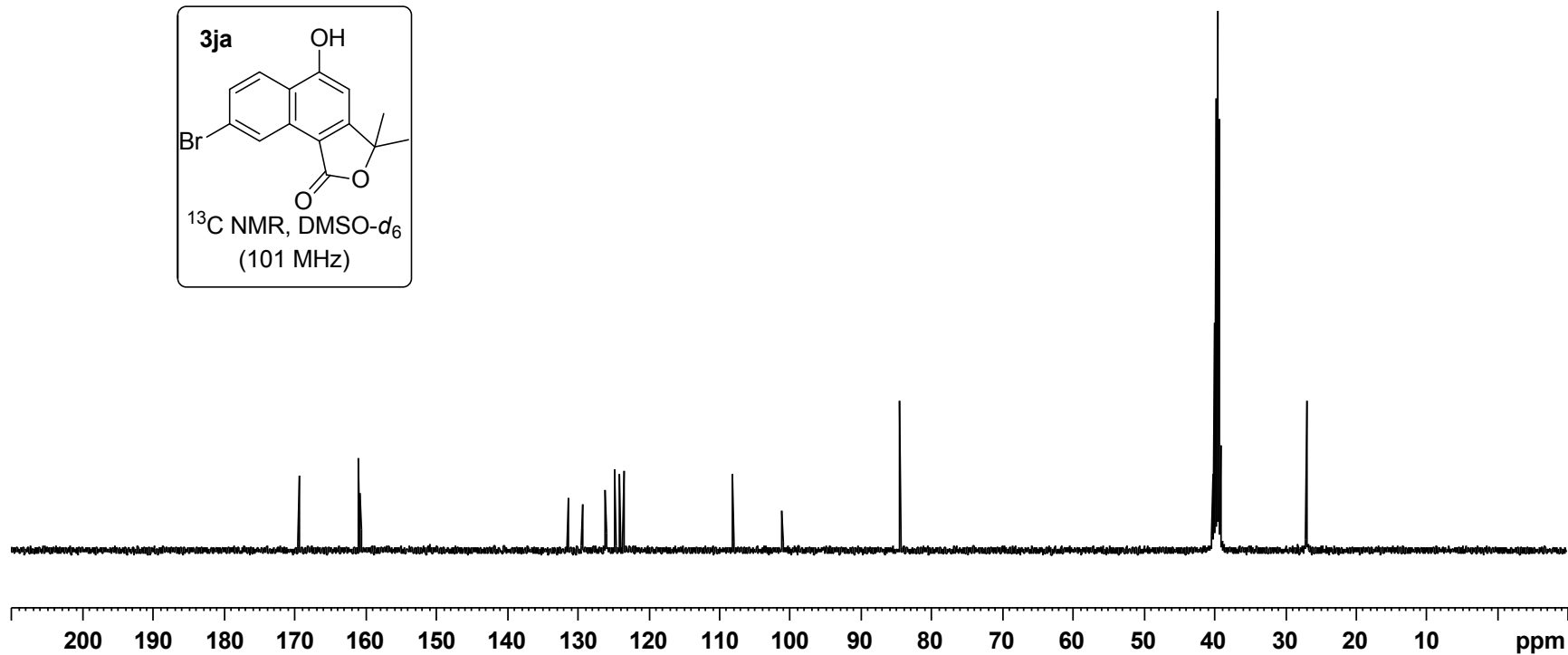
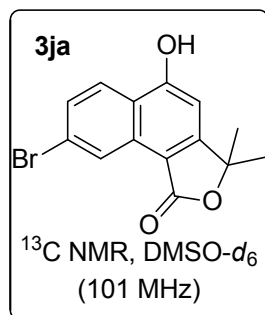
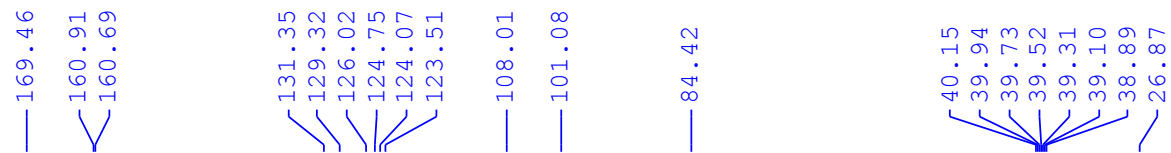
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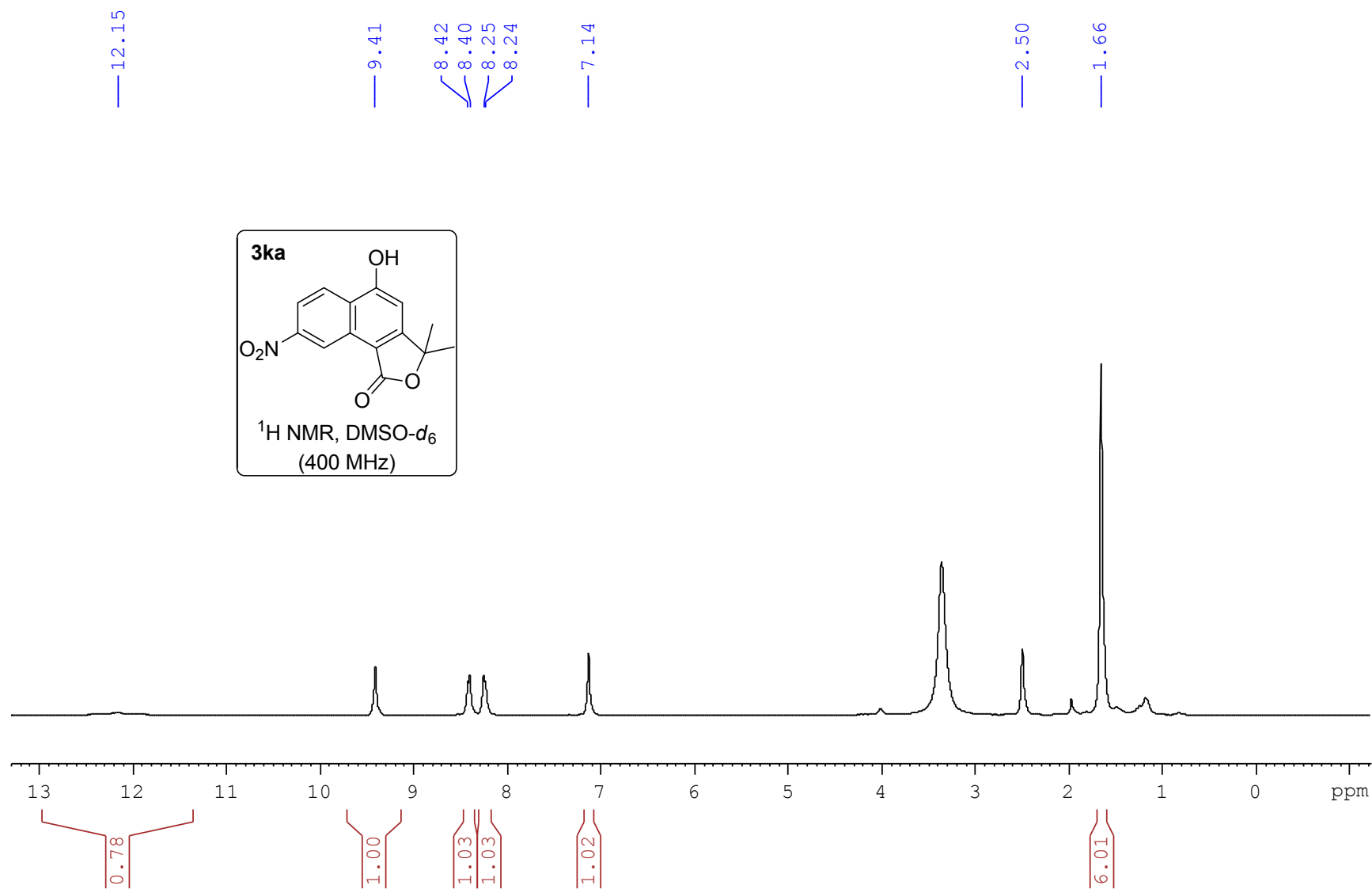


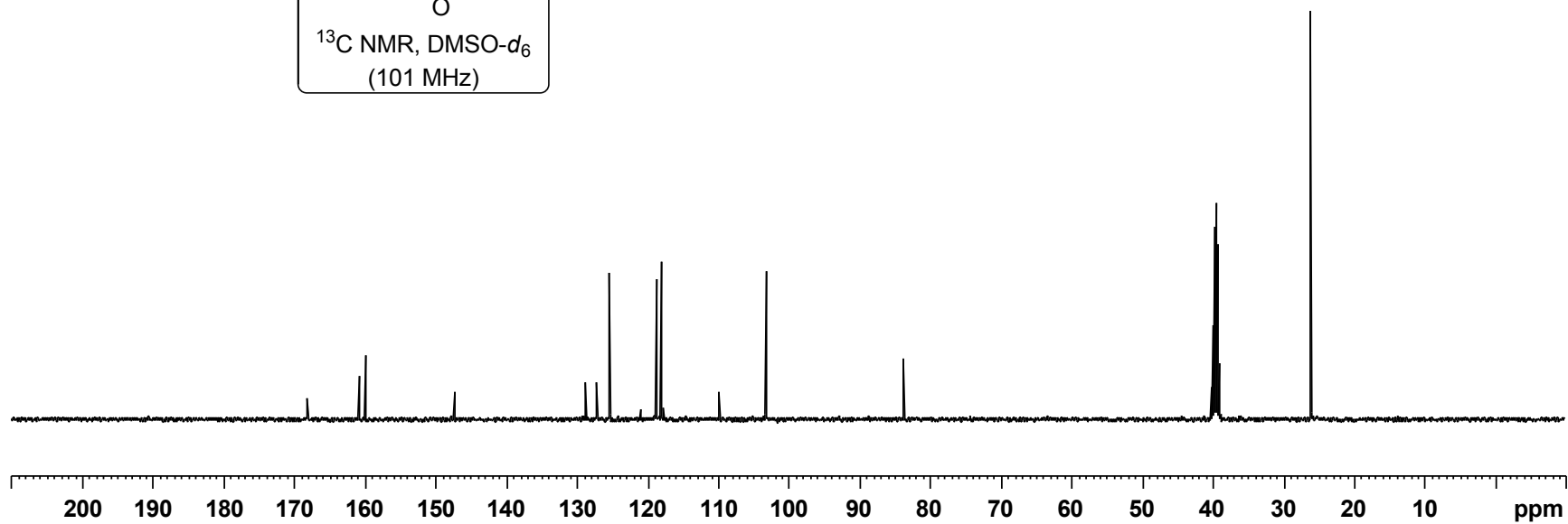
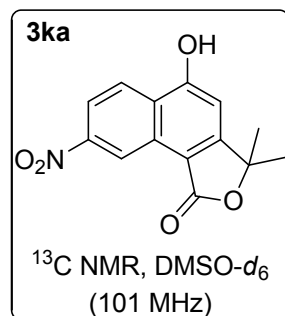
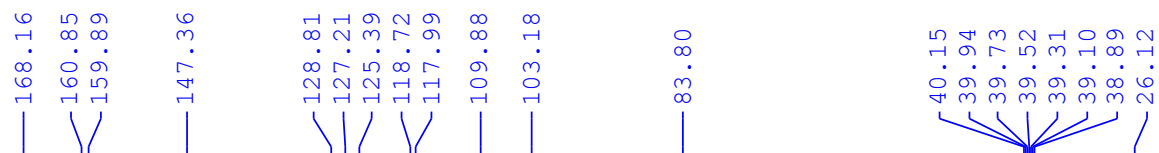
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VH-02-187

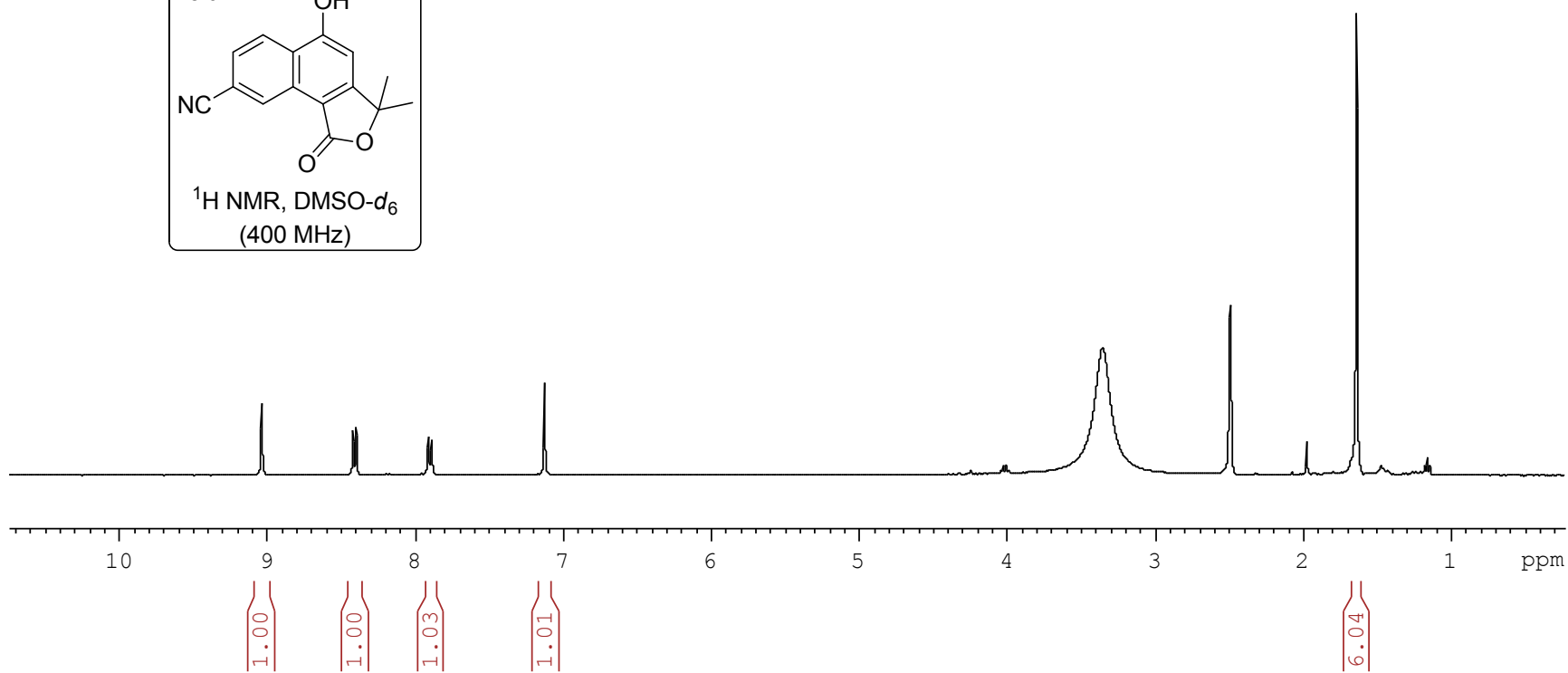
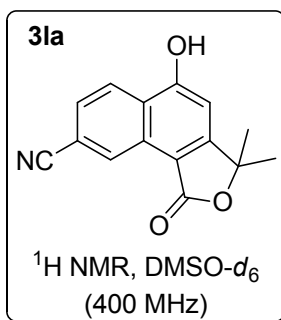




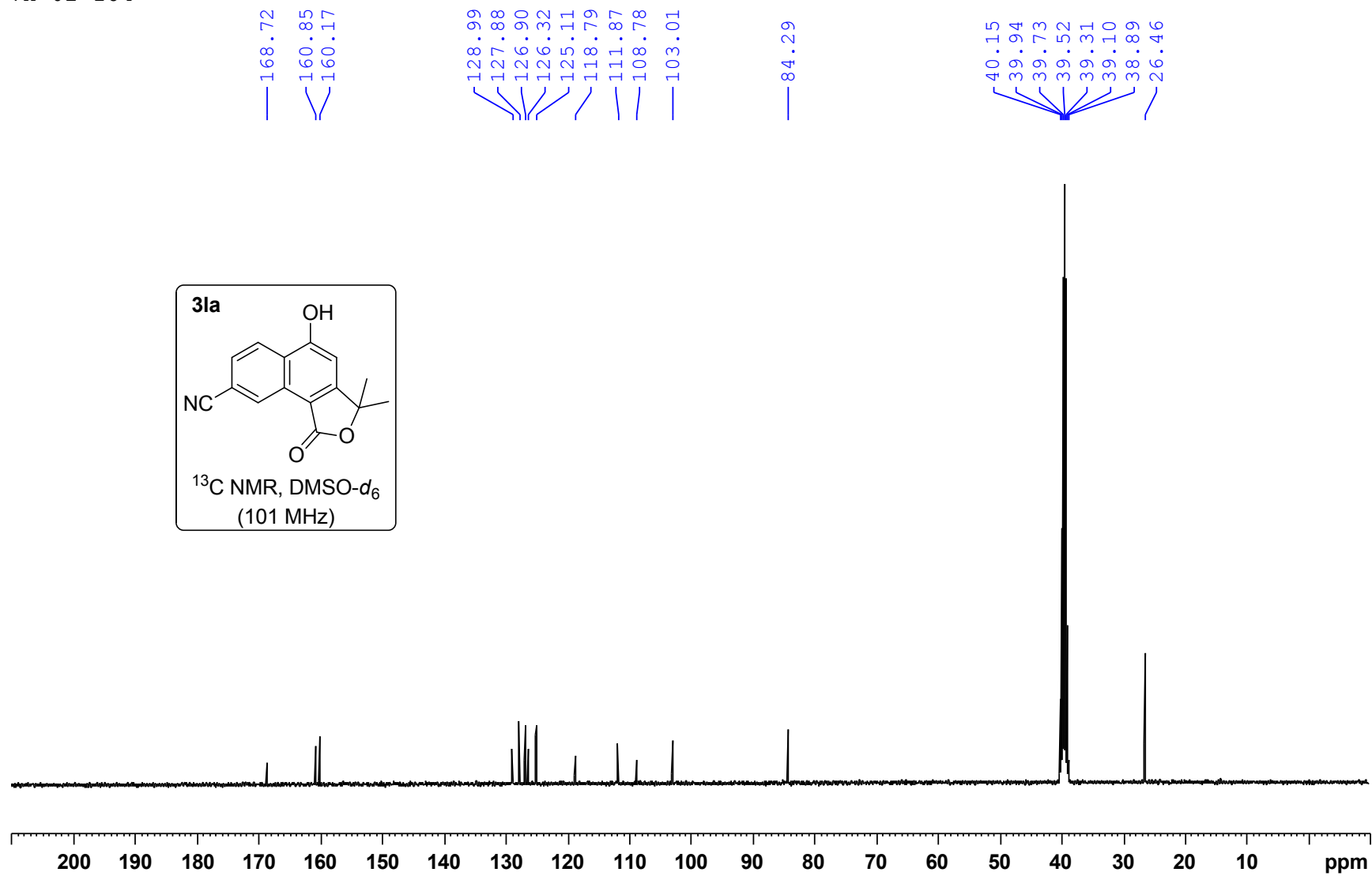


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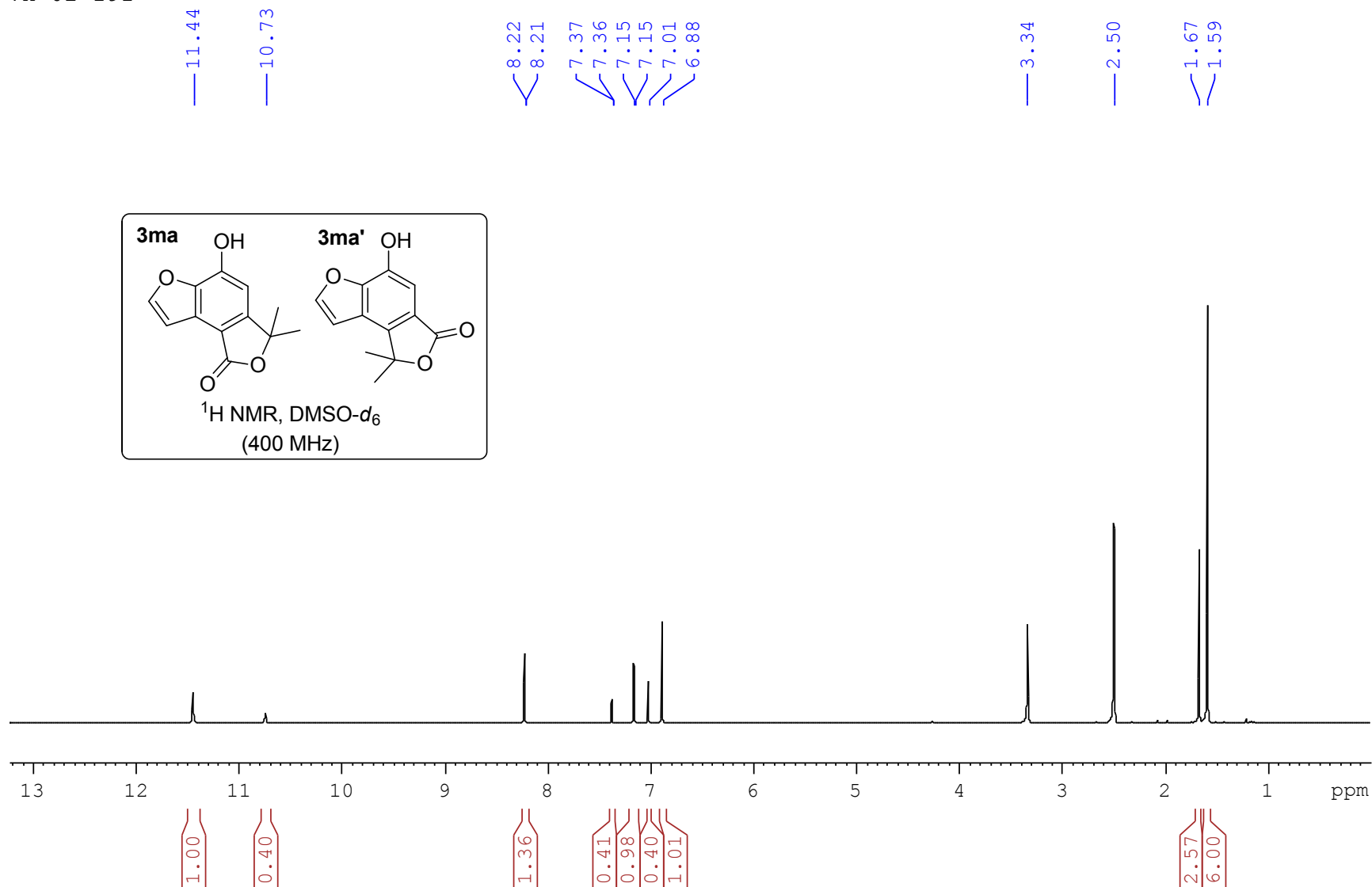
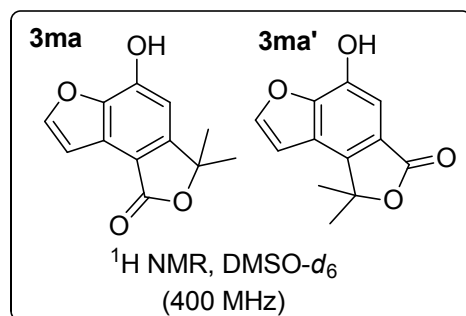
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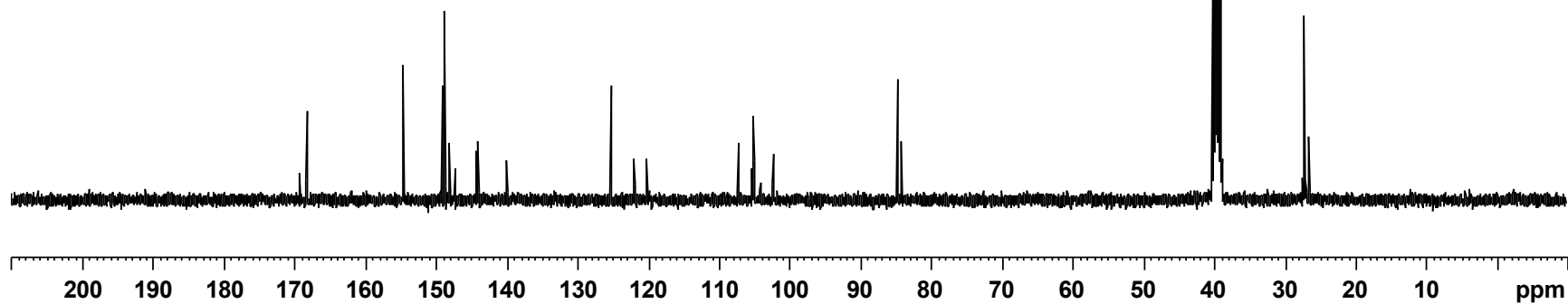
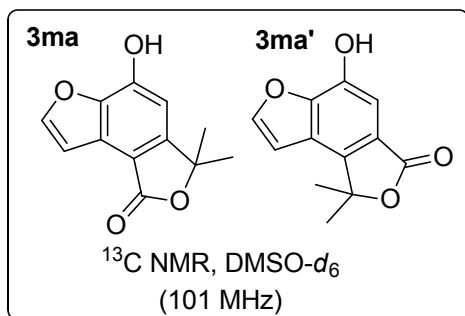


VH-02-191



VH-02-191

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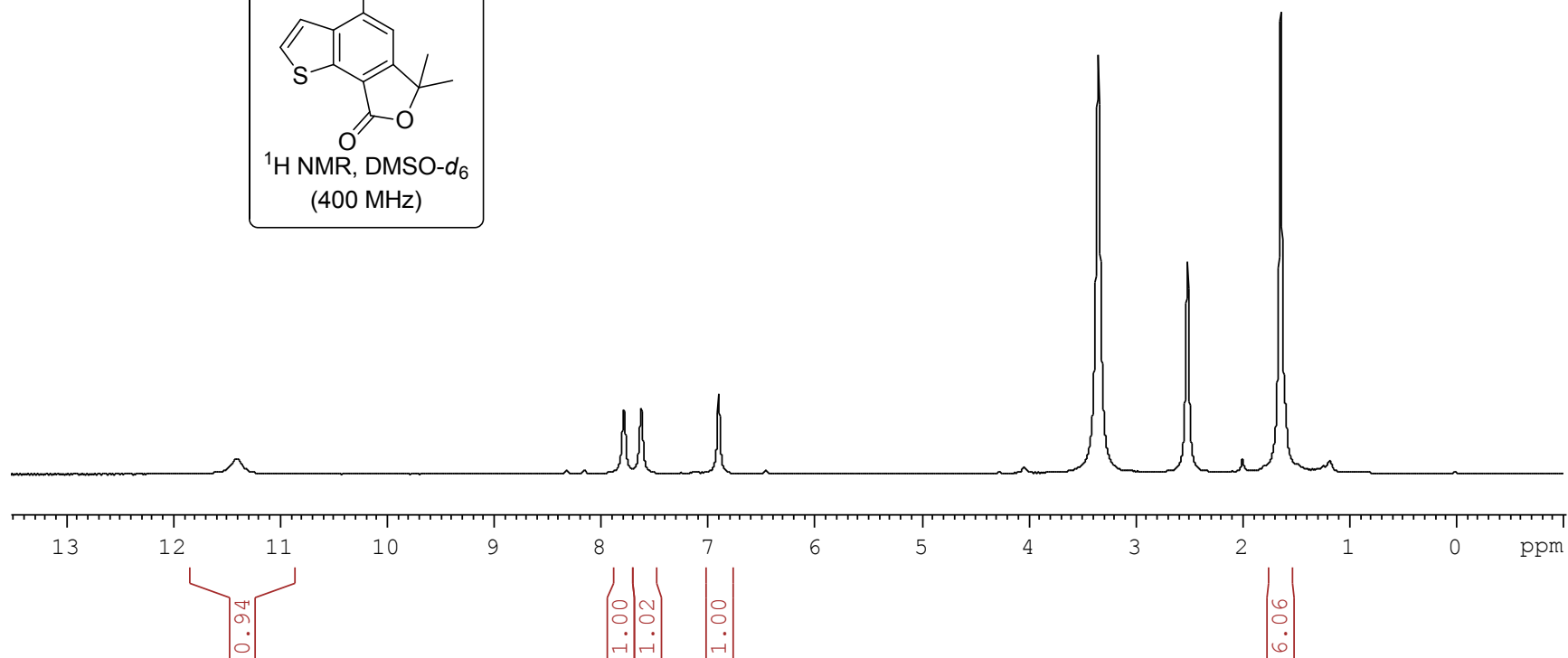
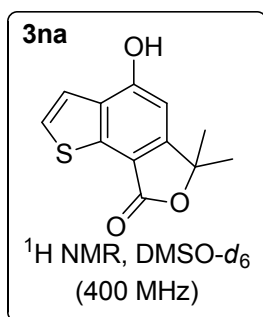
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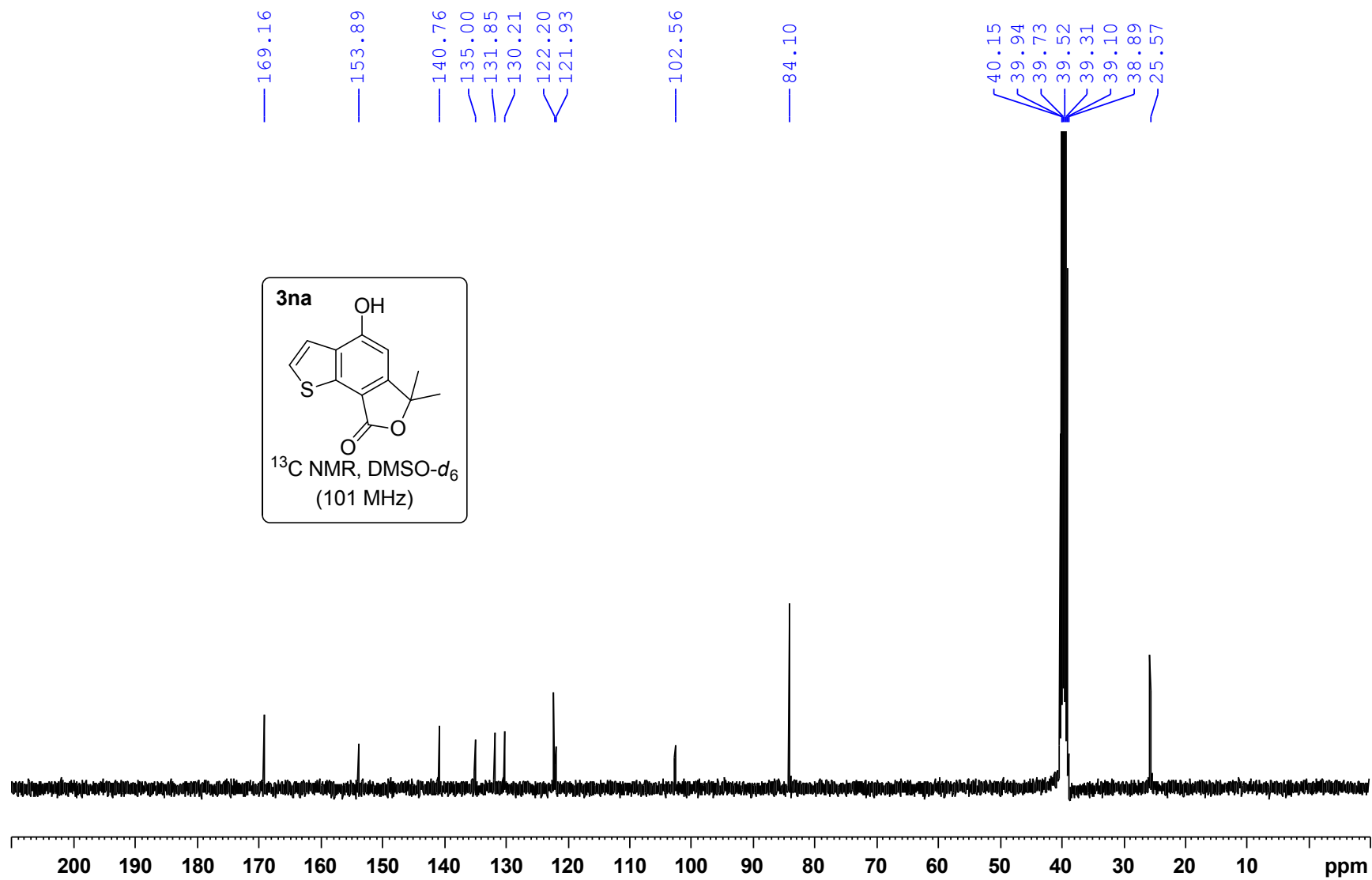
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6.89

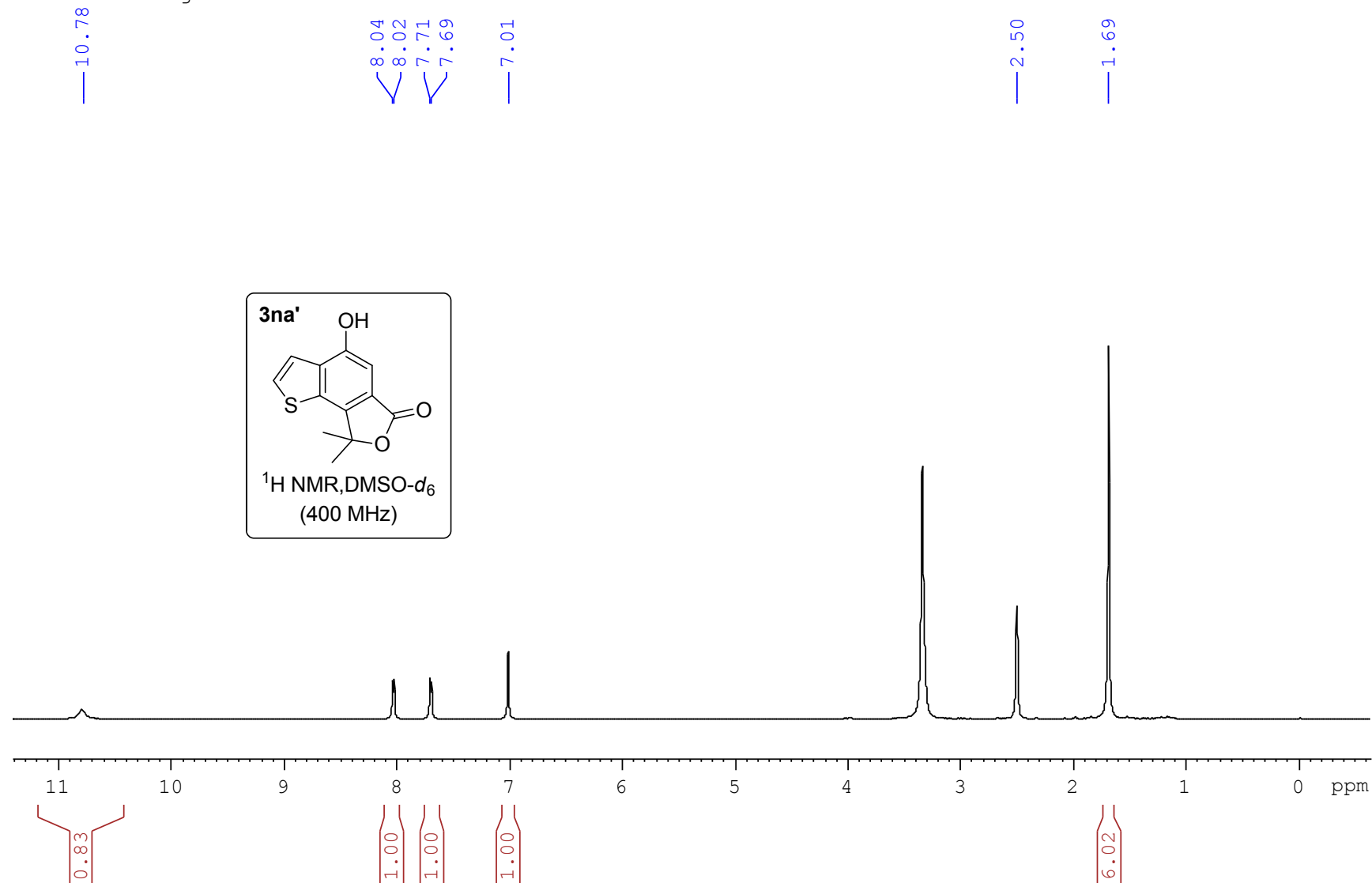
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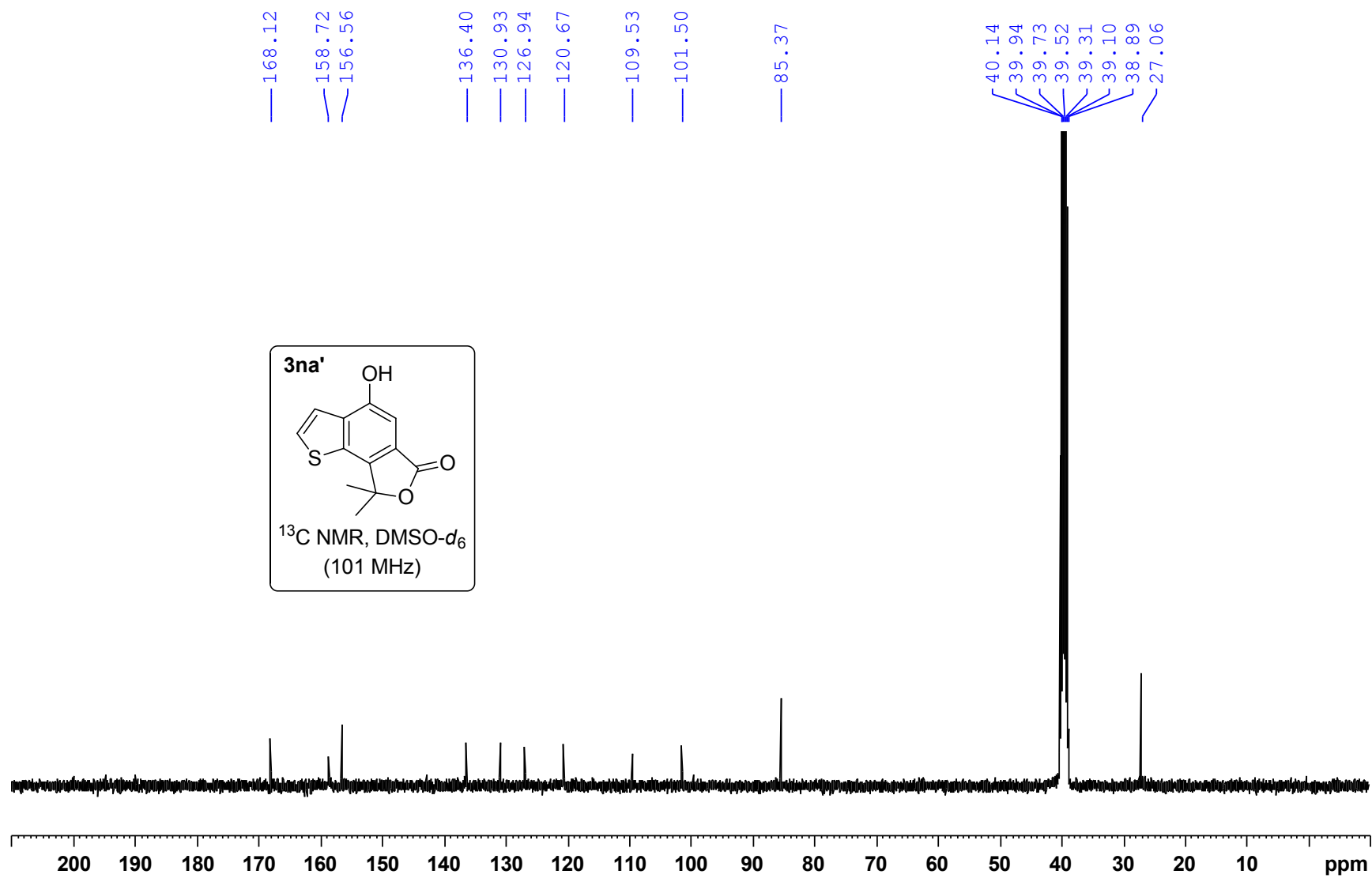
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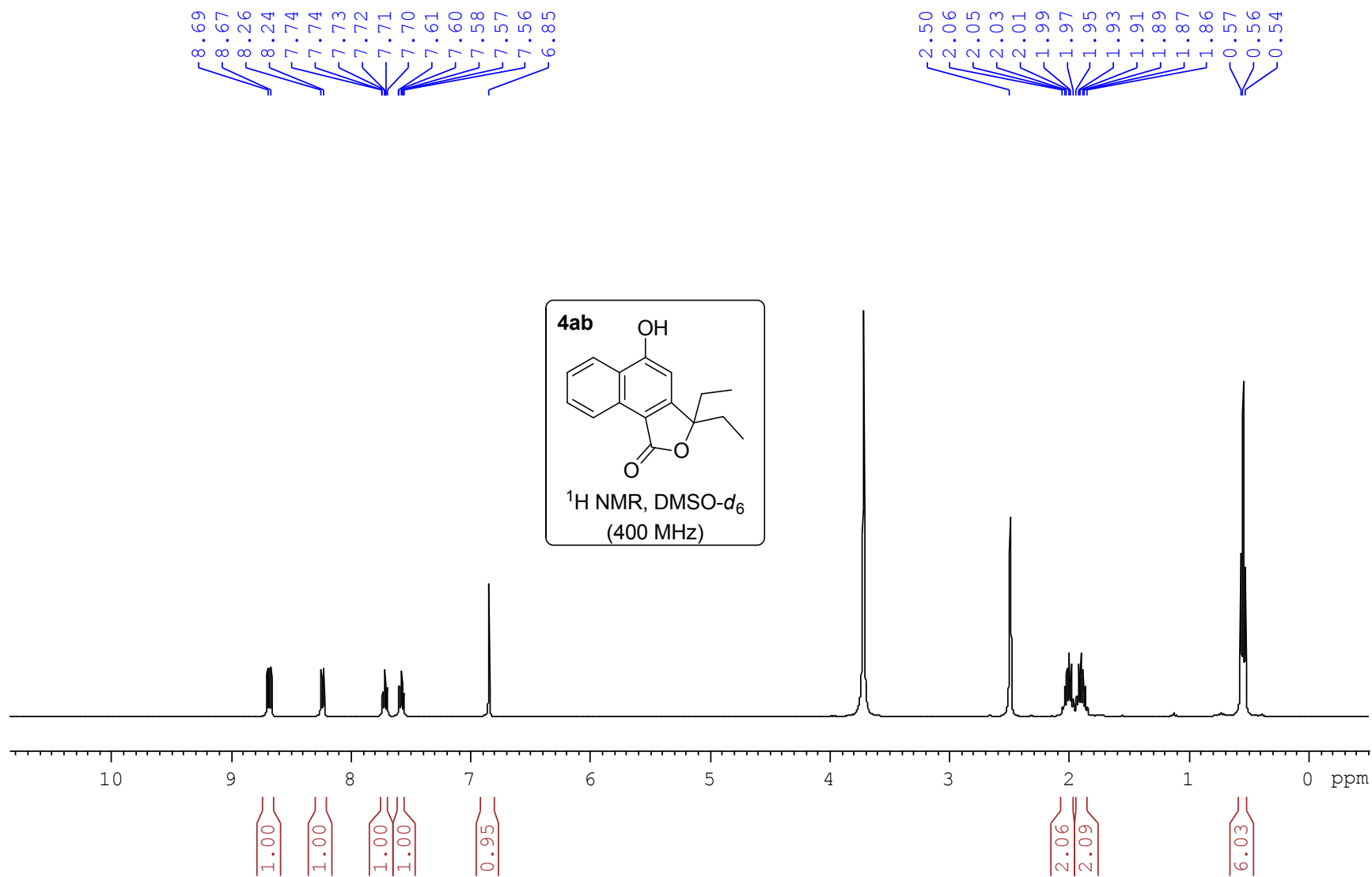


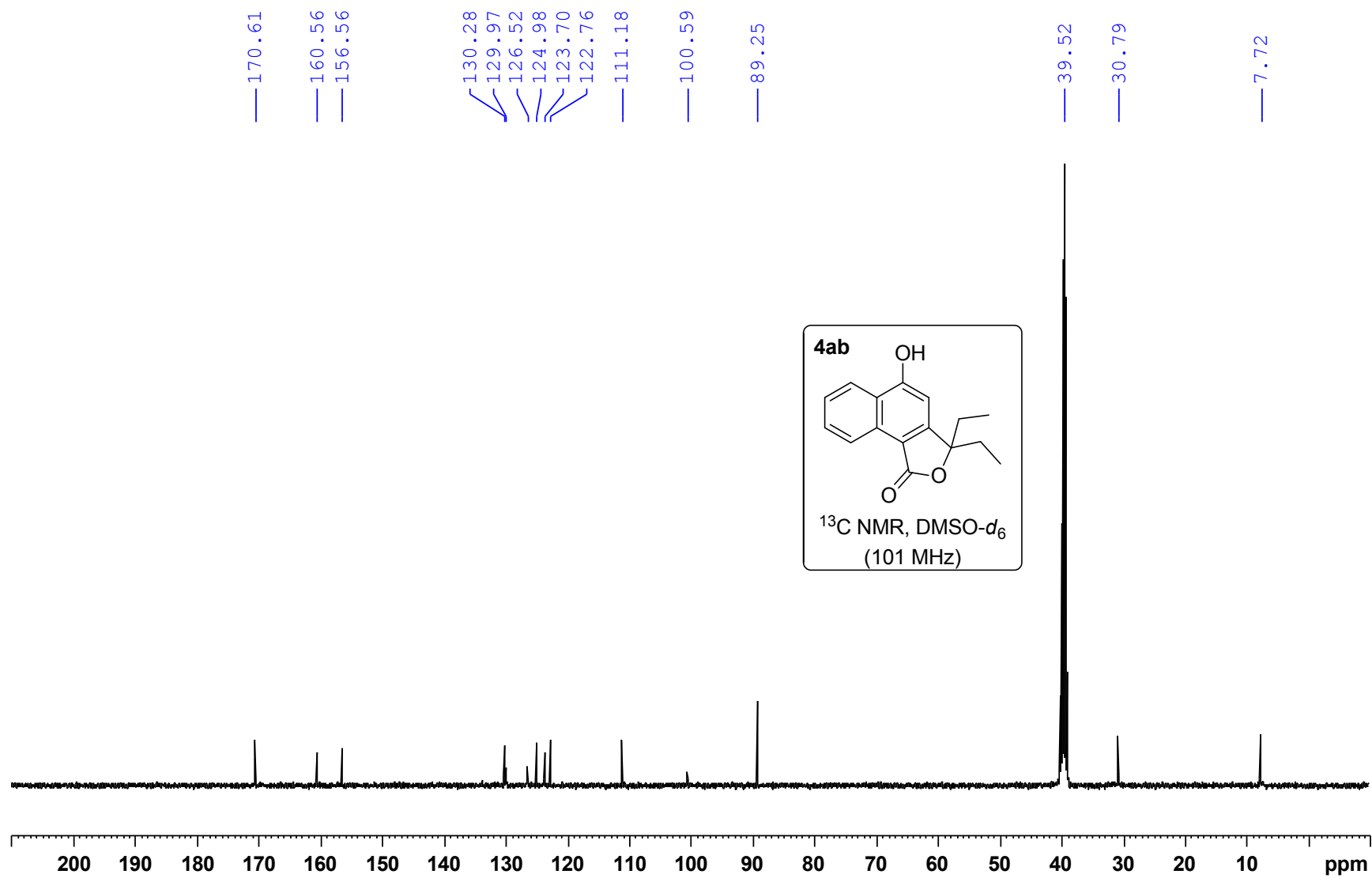


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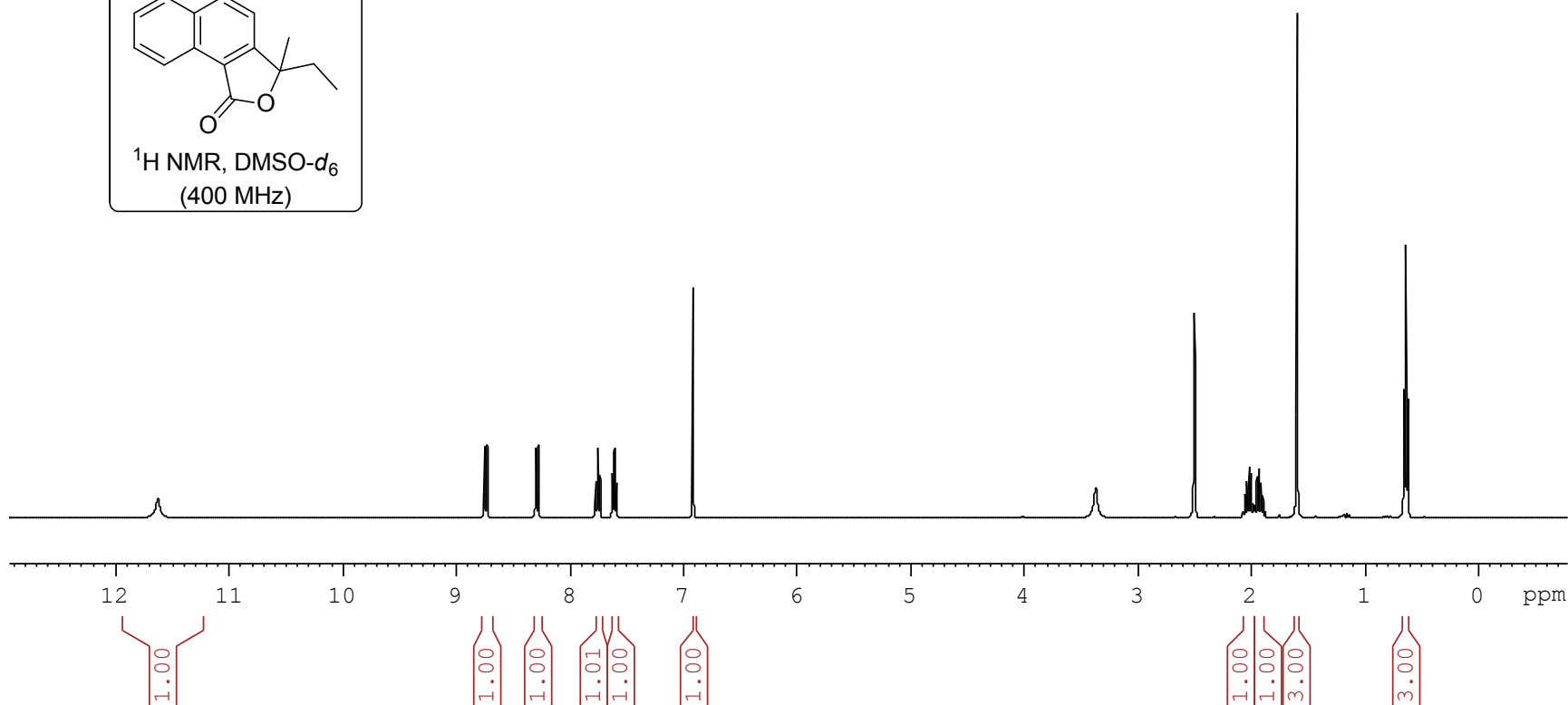
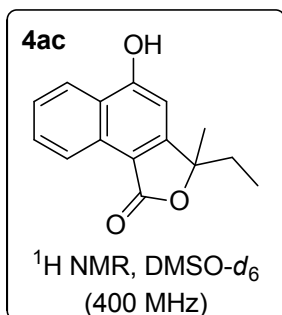


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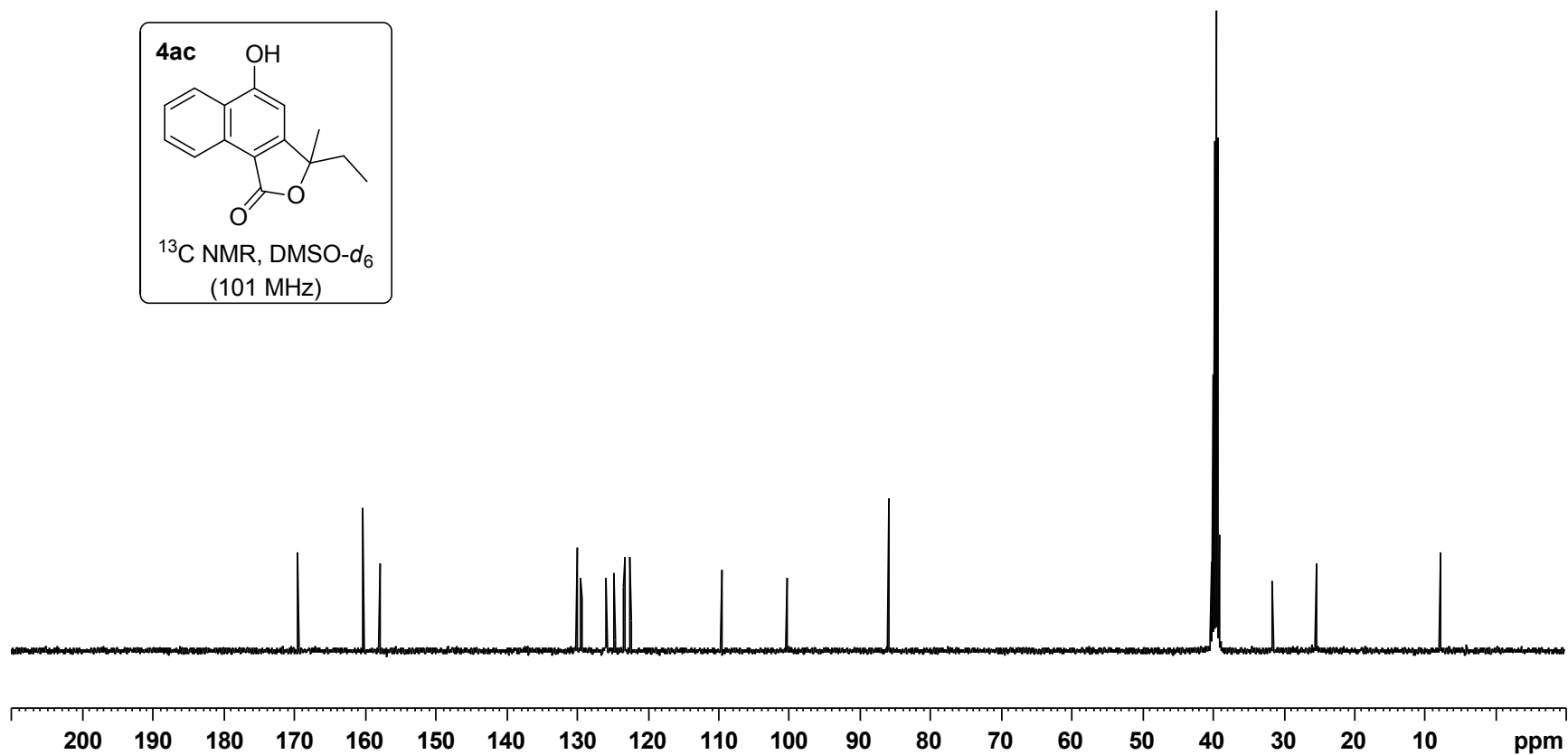
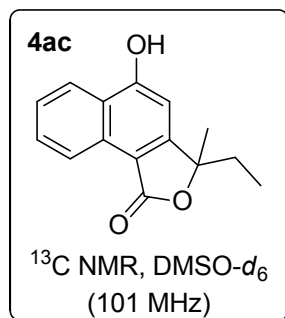
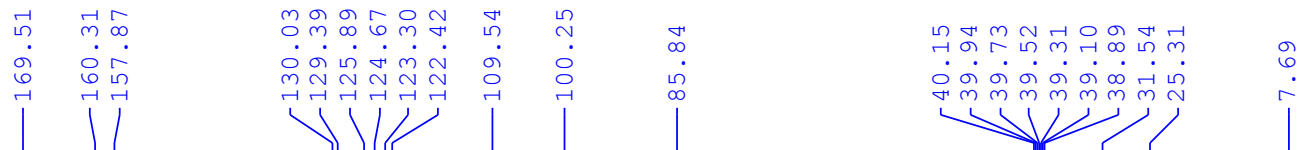
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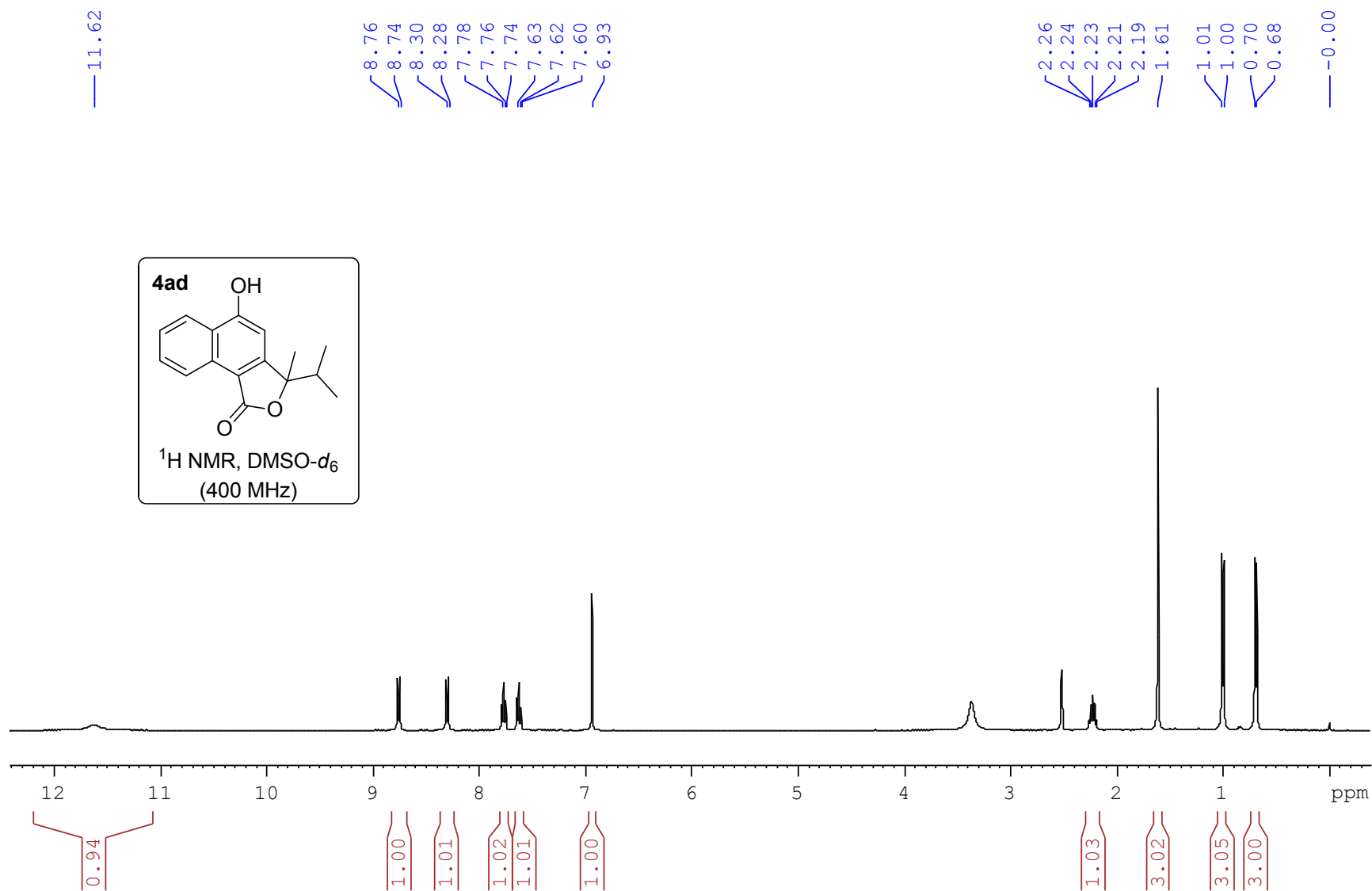
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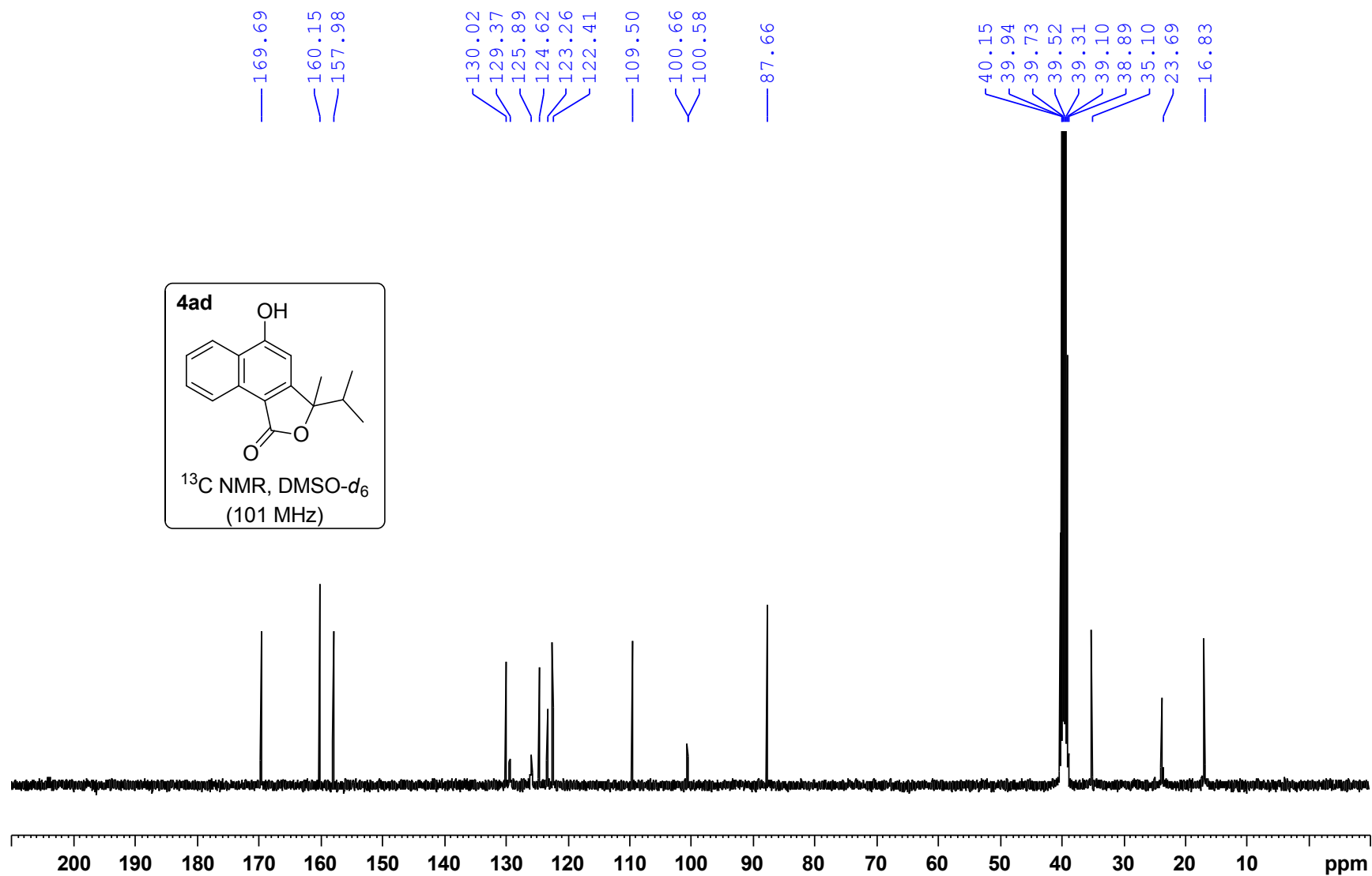
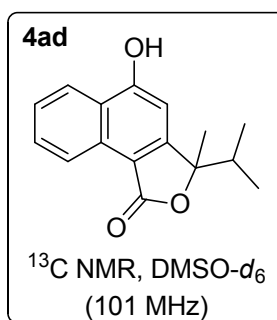
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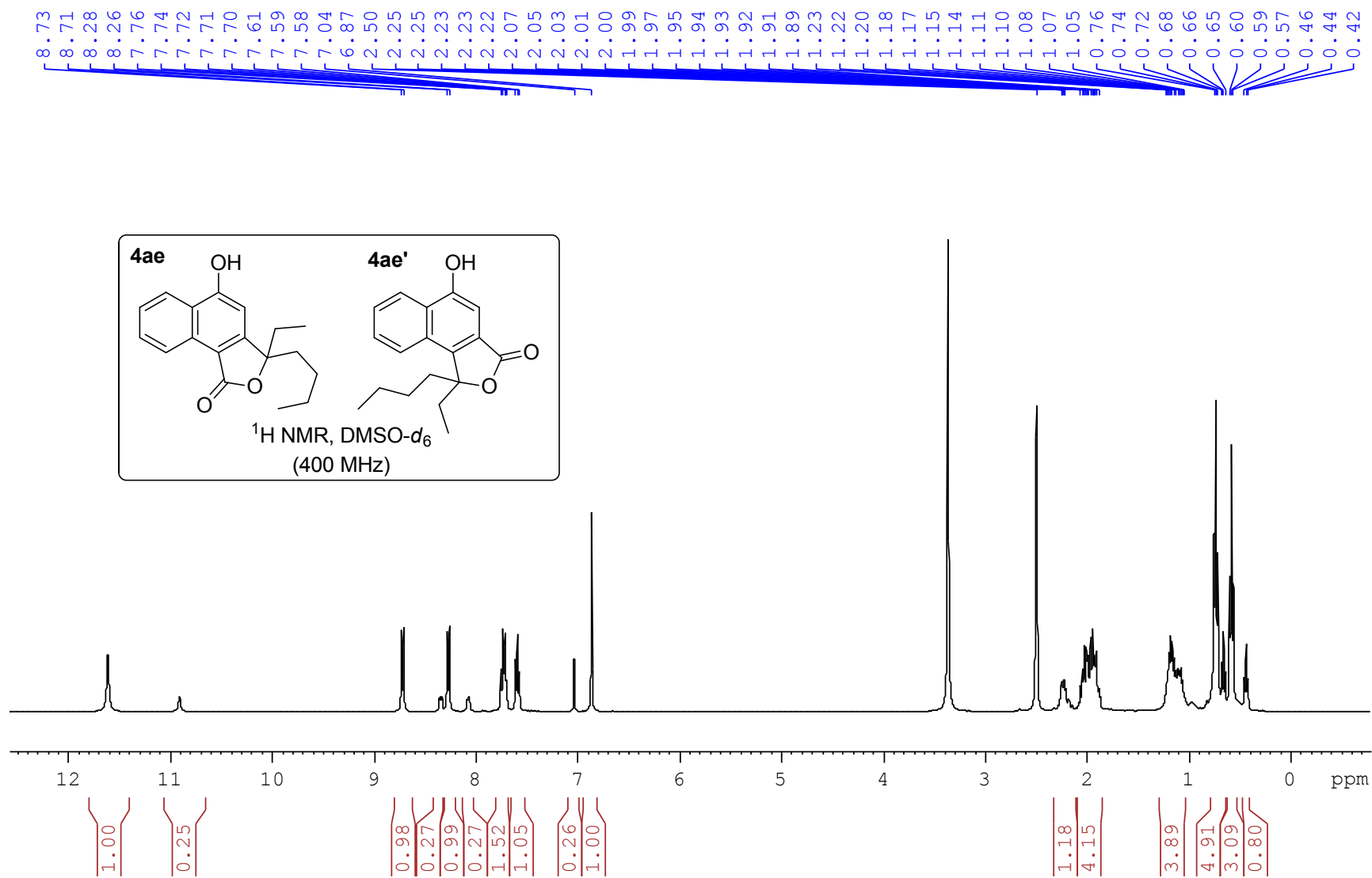


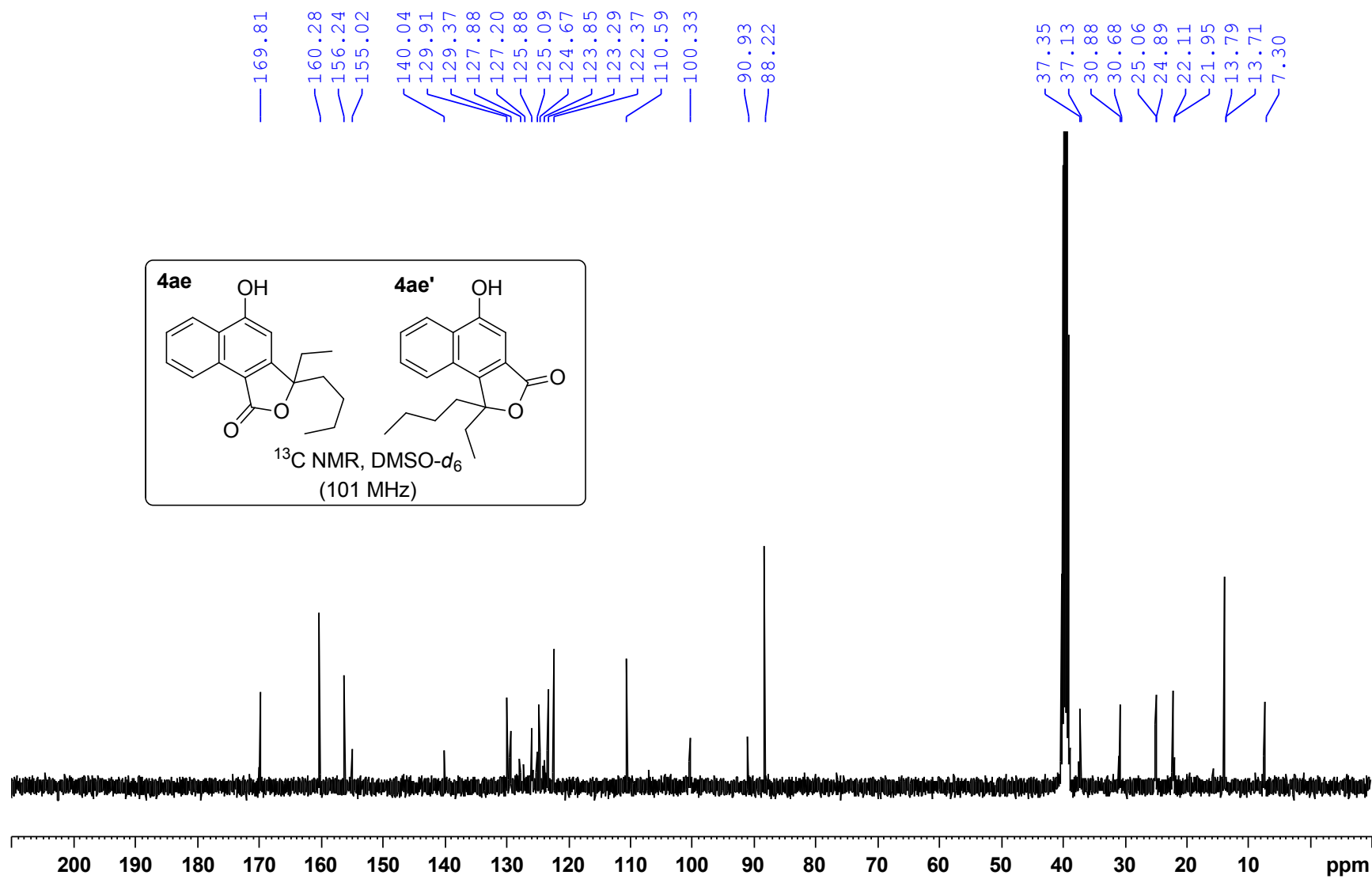
VH-02-AK3

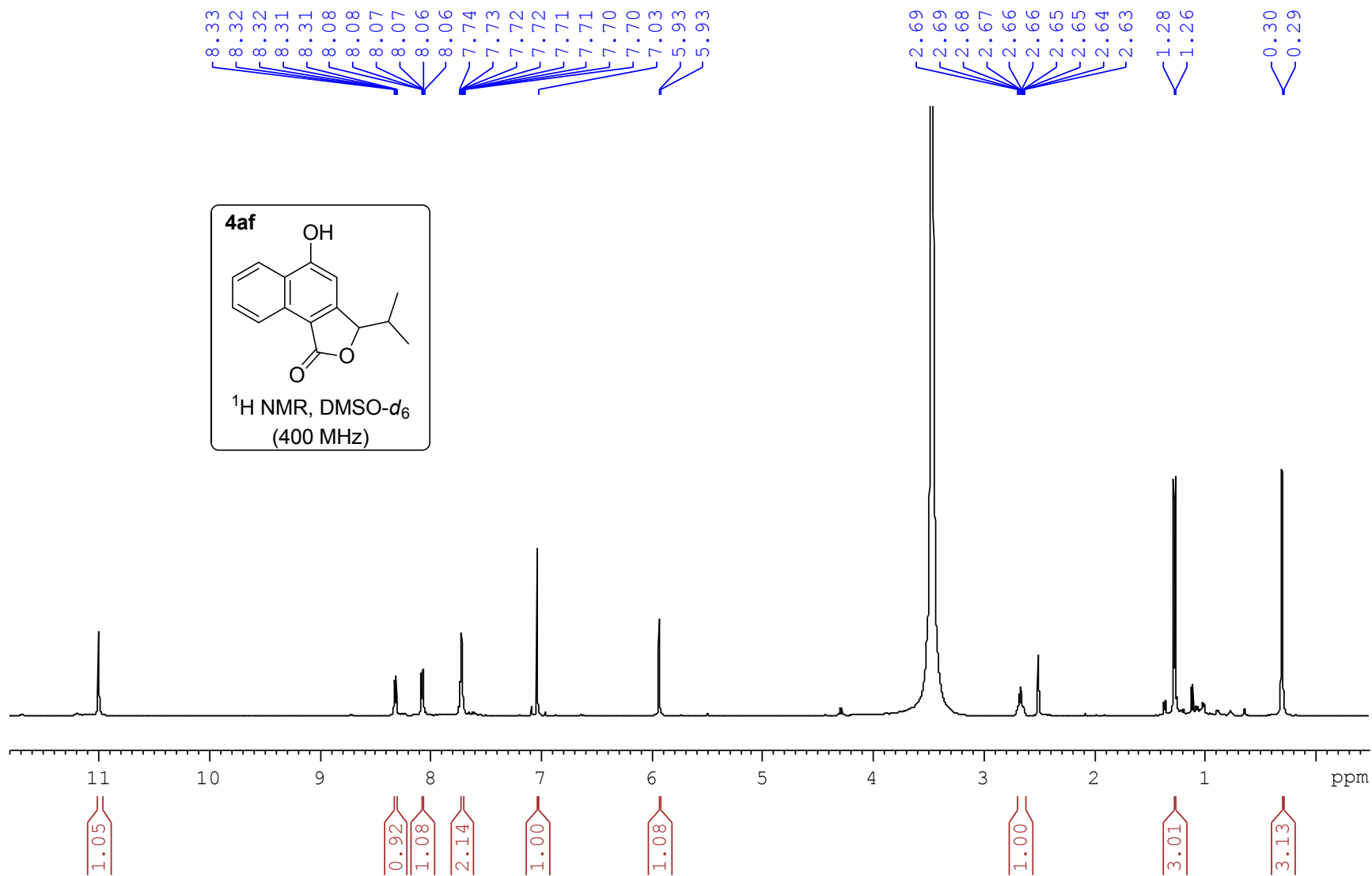


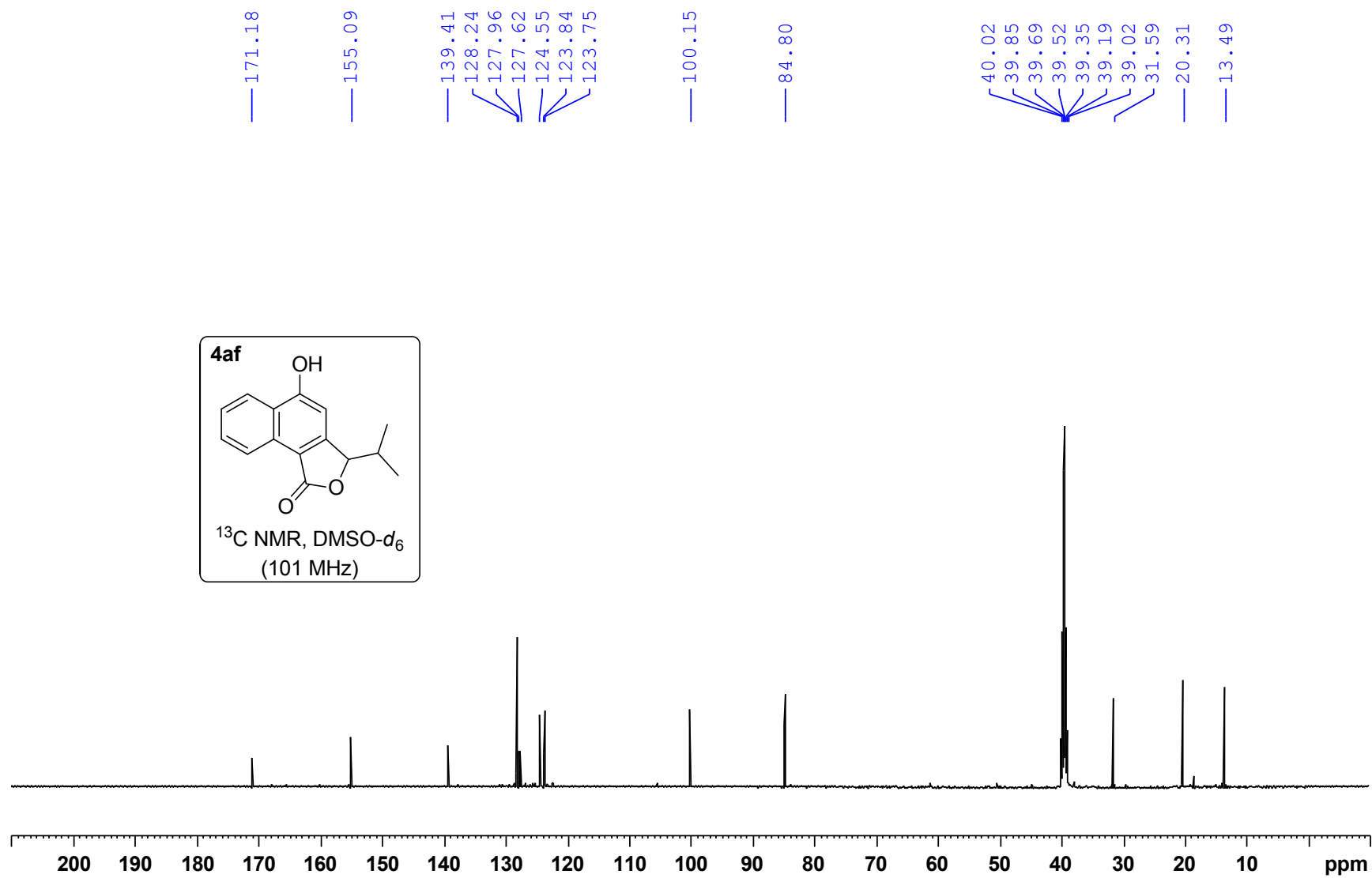








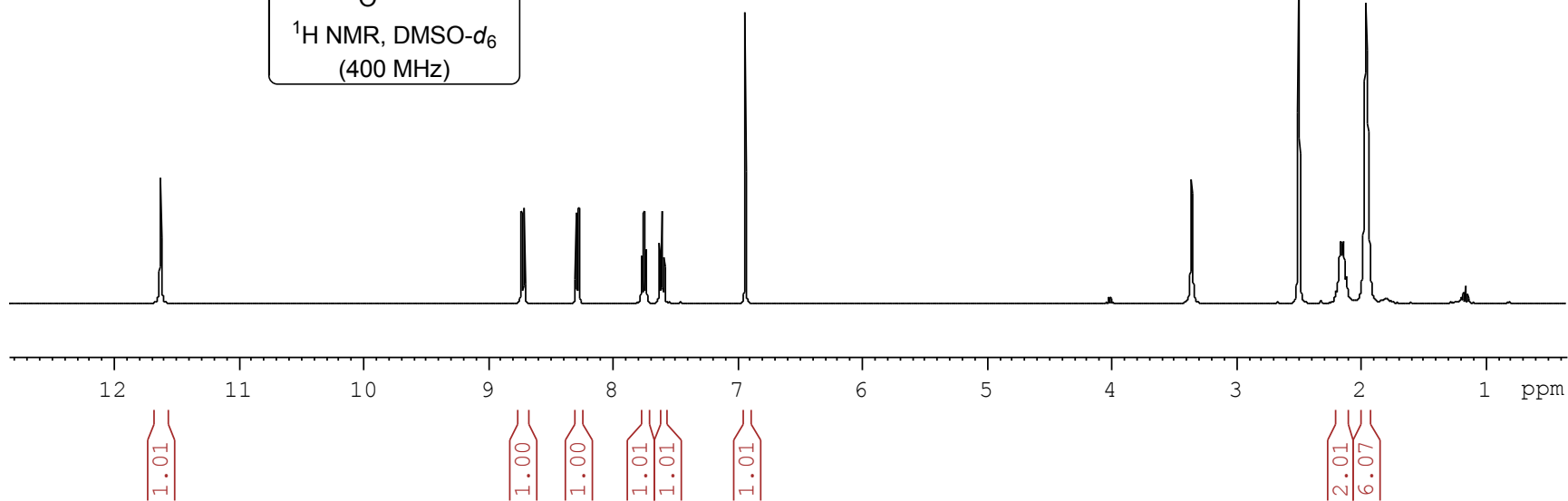
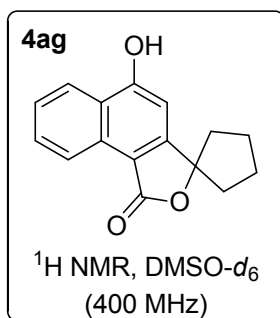


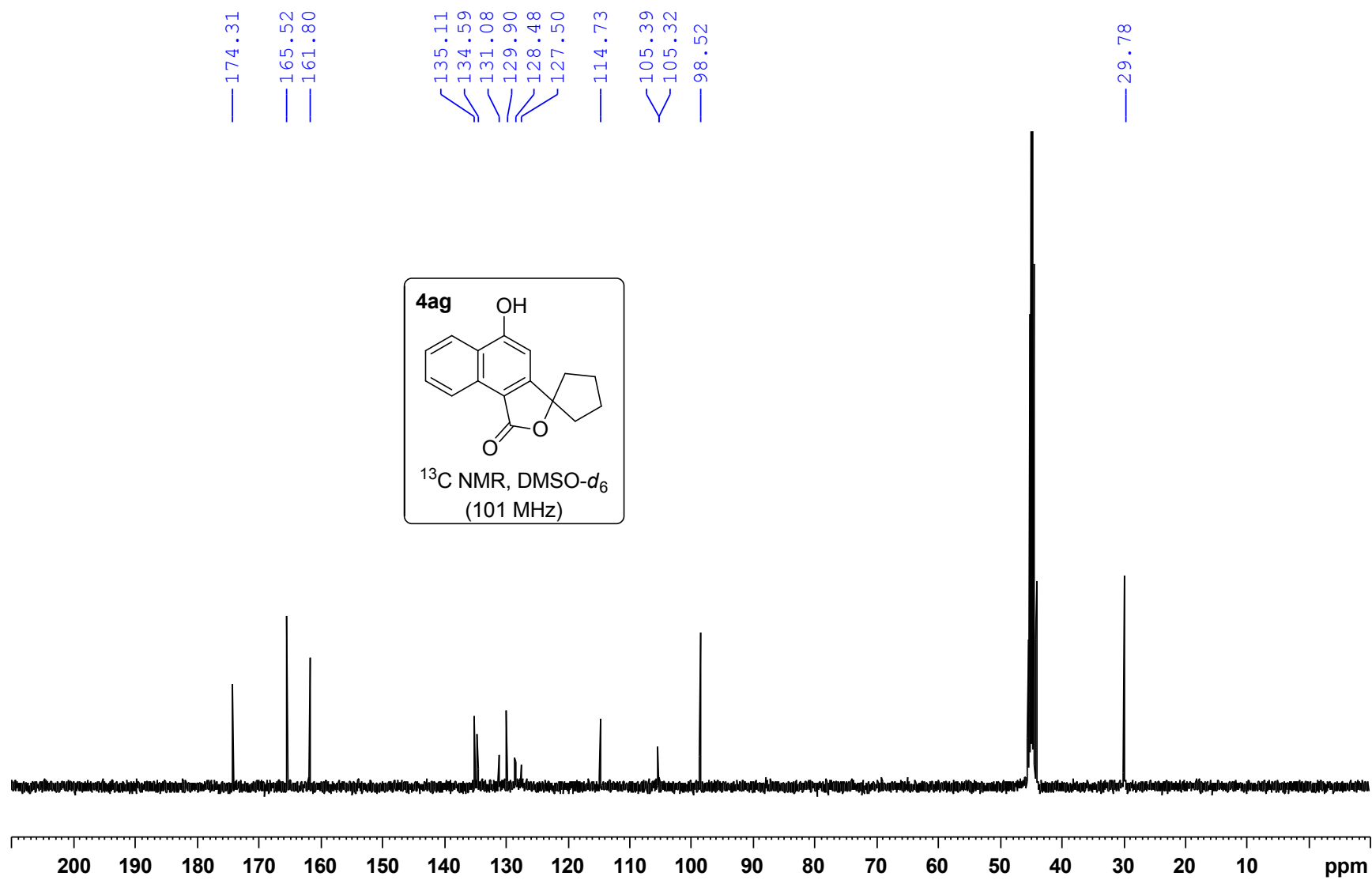


— 11.62

8.72
8.70
8.28
8.26
7.76
7.76
7.74
7.74
7.72
7.72
7.62
7.61
7.60
7.59
7.58
7.58
6.93

2.50
2.20
2.16
2.16
2.14
2.12
1.98
1.96
1.95

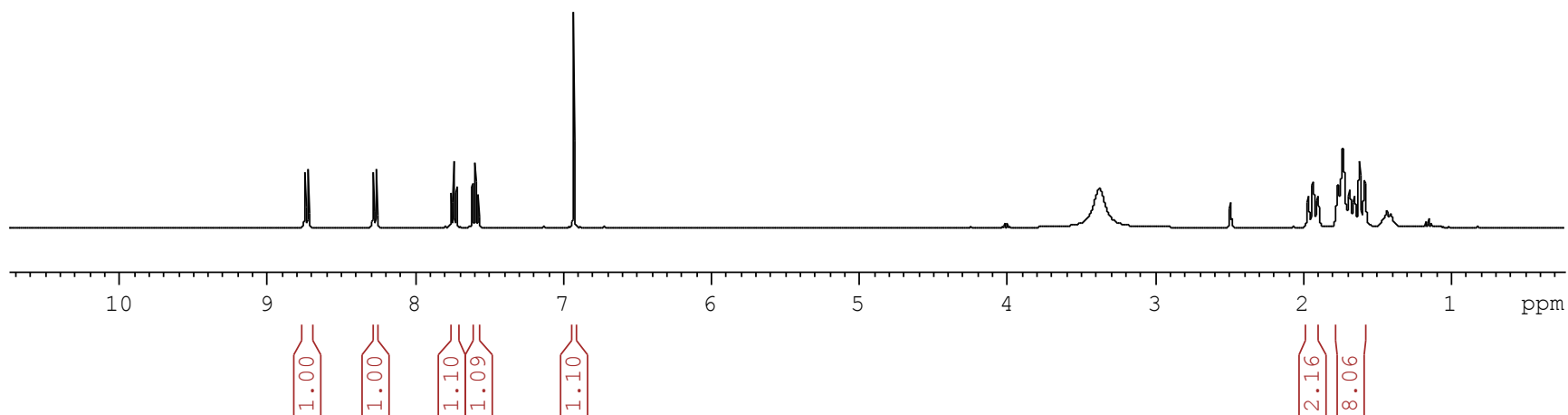
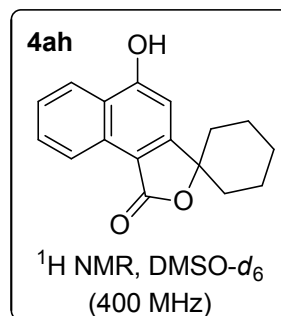




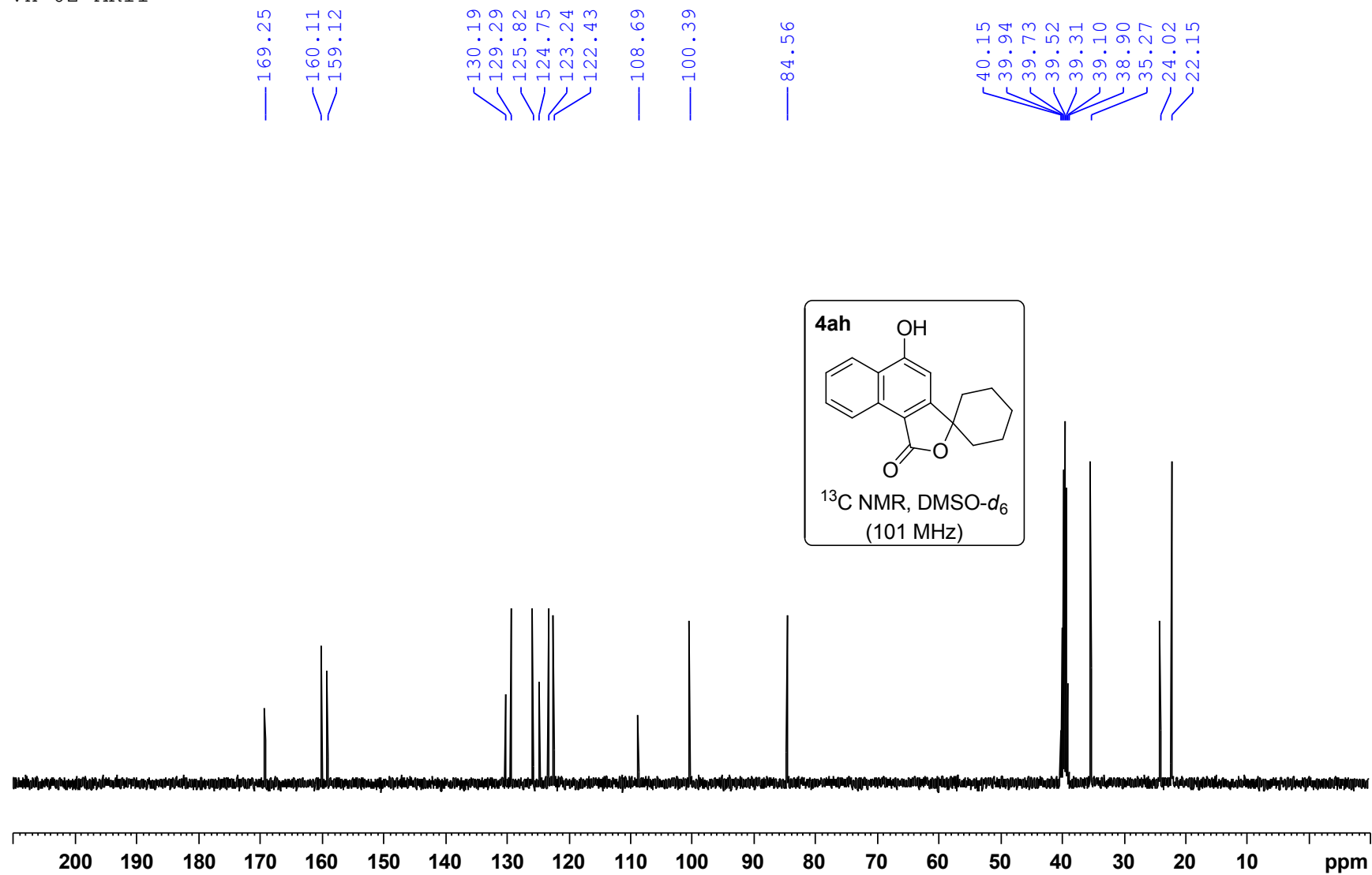
VH-02-AK11

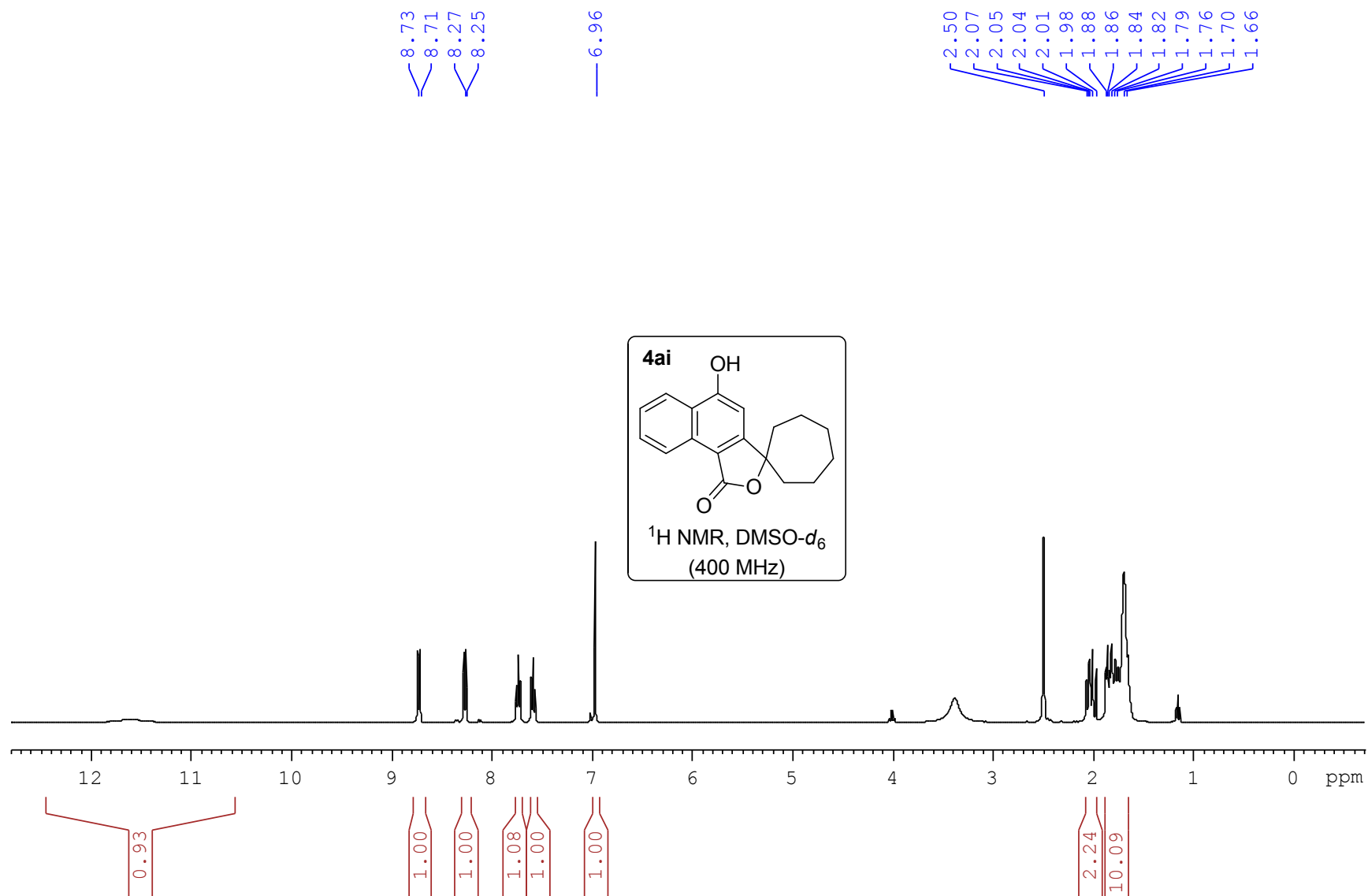
8.74
8.72
8.28
8.26
7.75
7.75
7.74
7.73
7.73
7.72
7.71
7.61
7.61
7.59
7.59
7.59
7.57
7.57
6.92

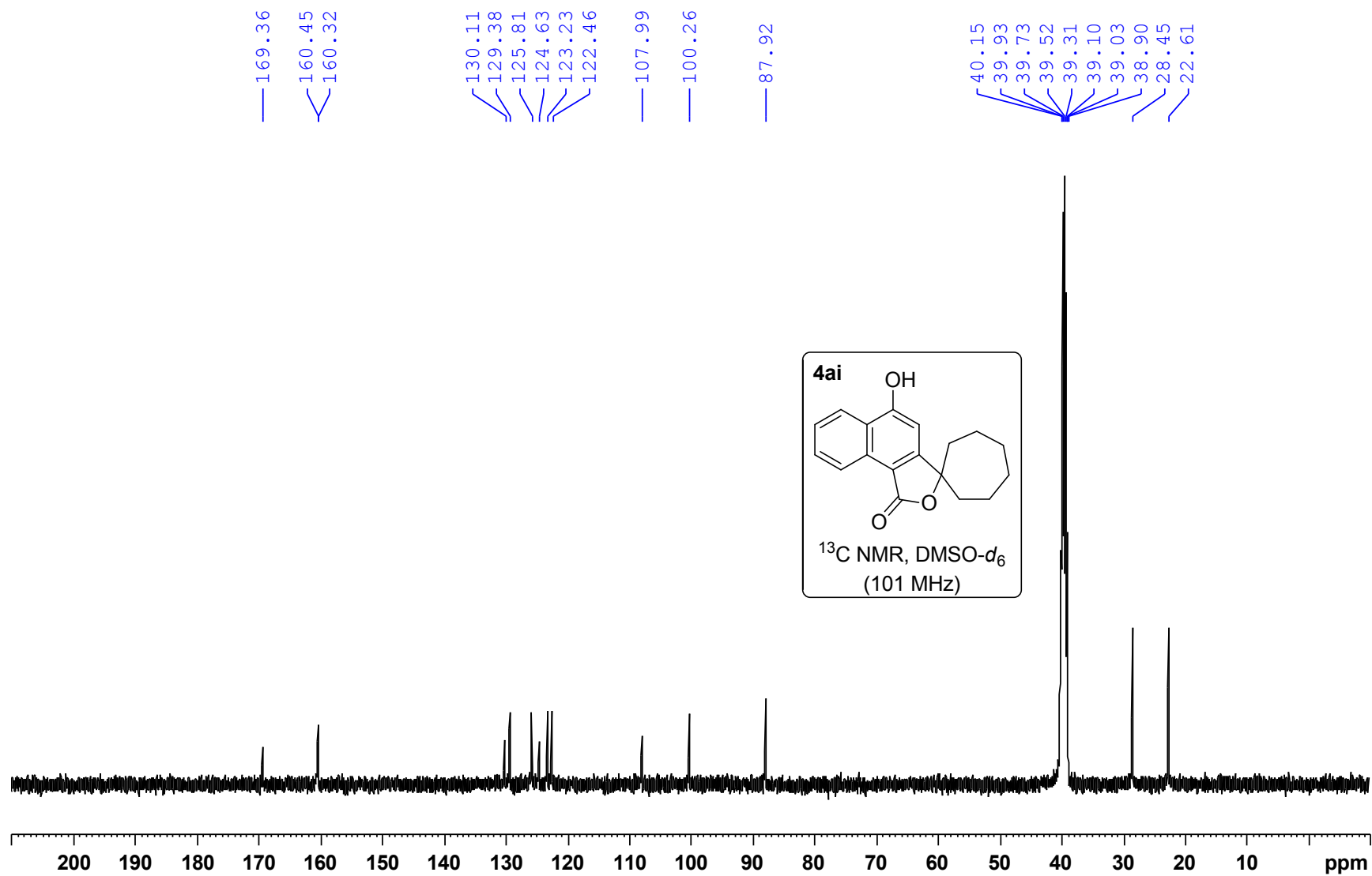
1.98
1.97
1.95
1.94
1.91
1.90
1.77
1.74
1.69
1.66
1.63
1.59

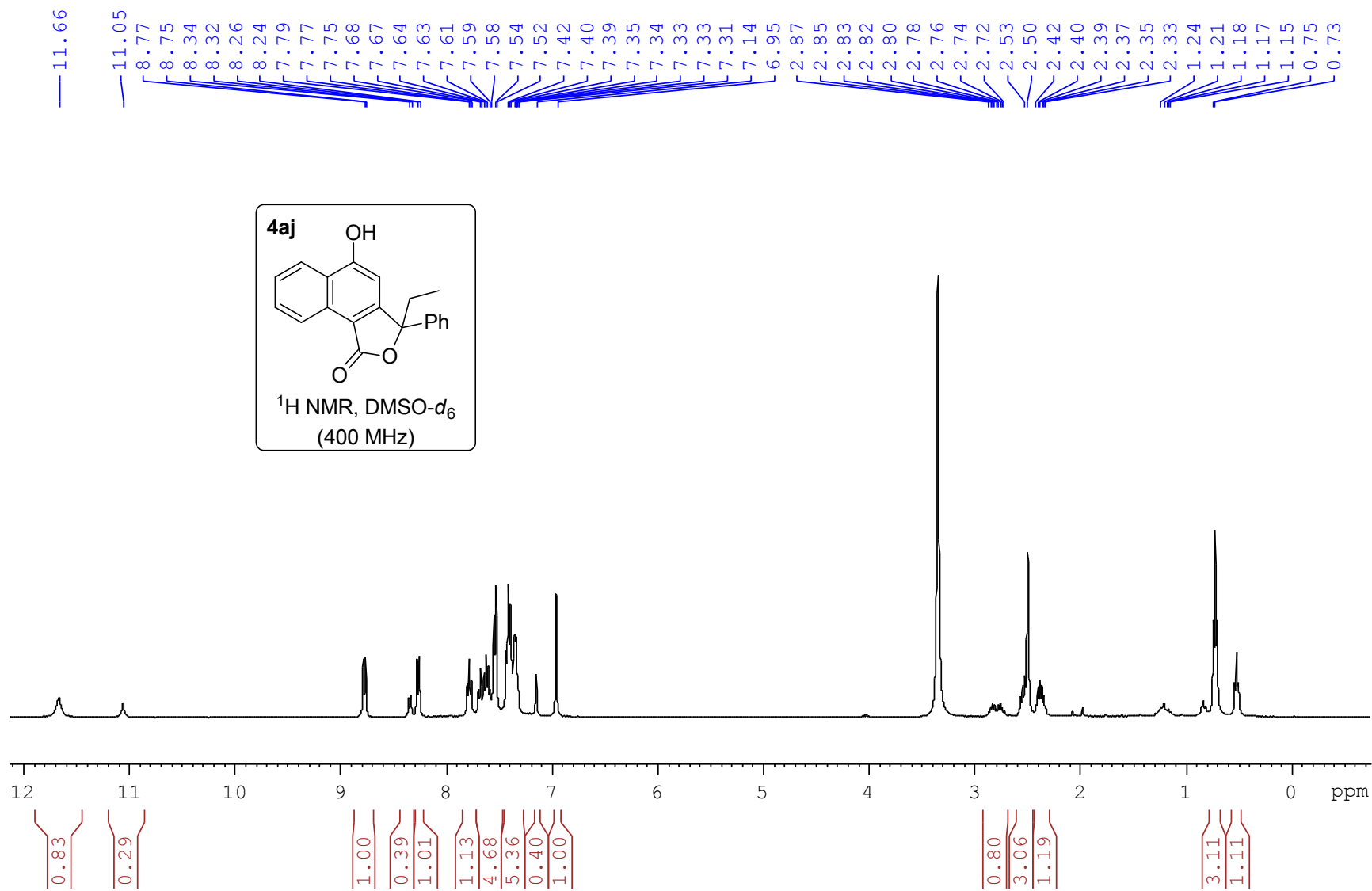


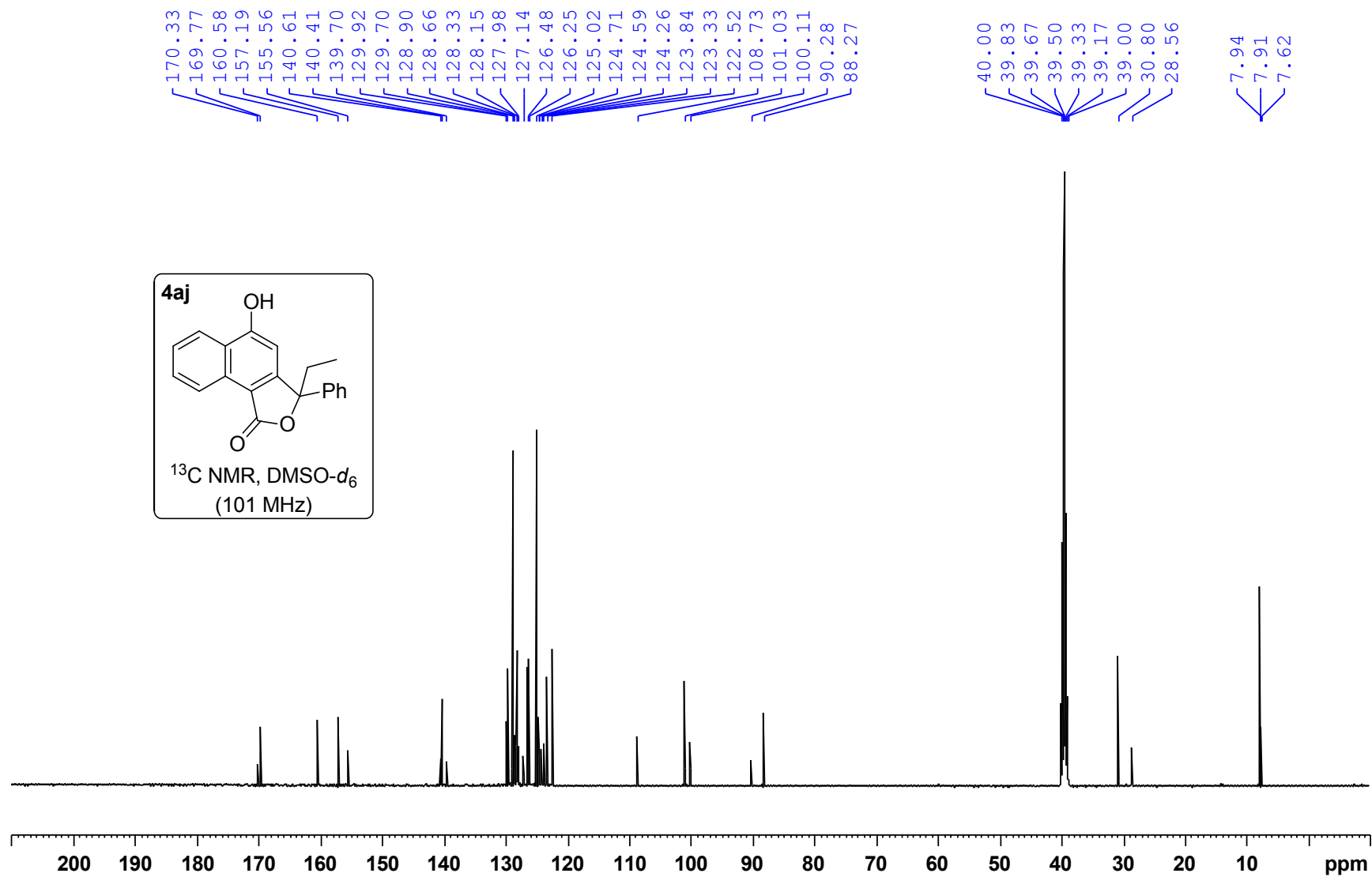
VH-02-AK11



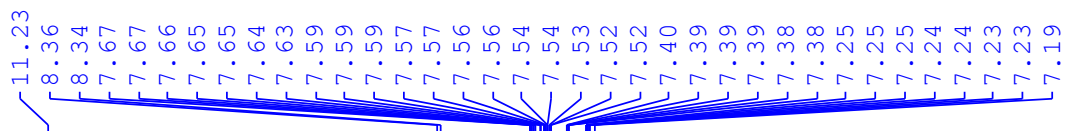




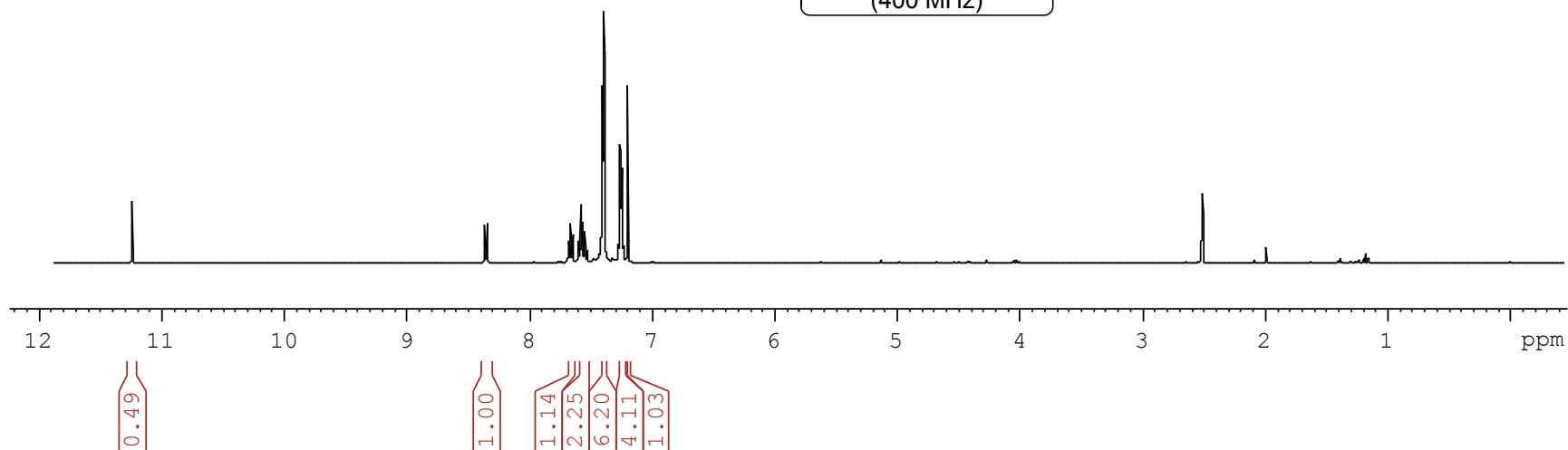
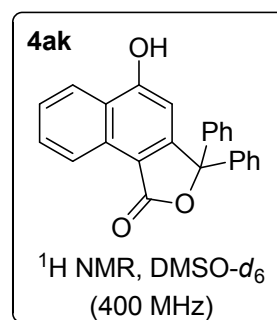


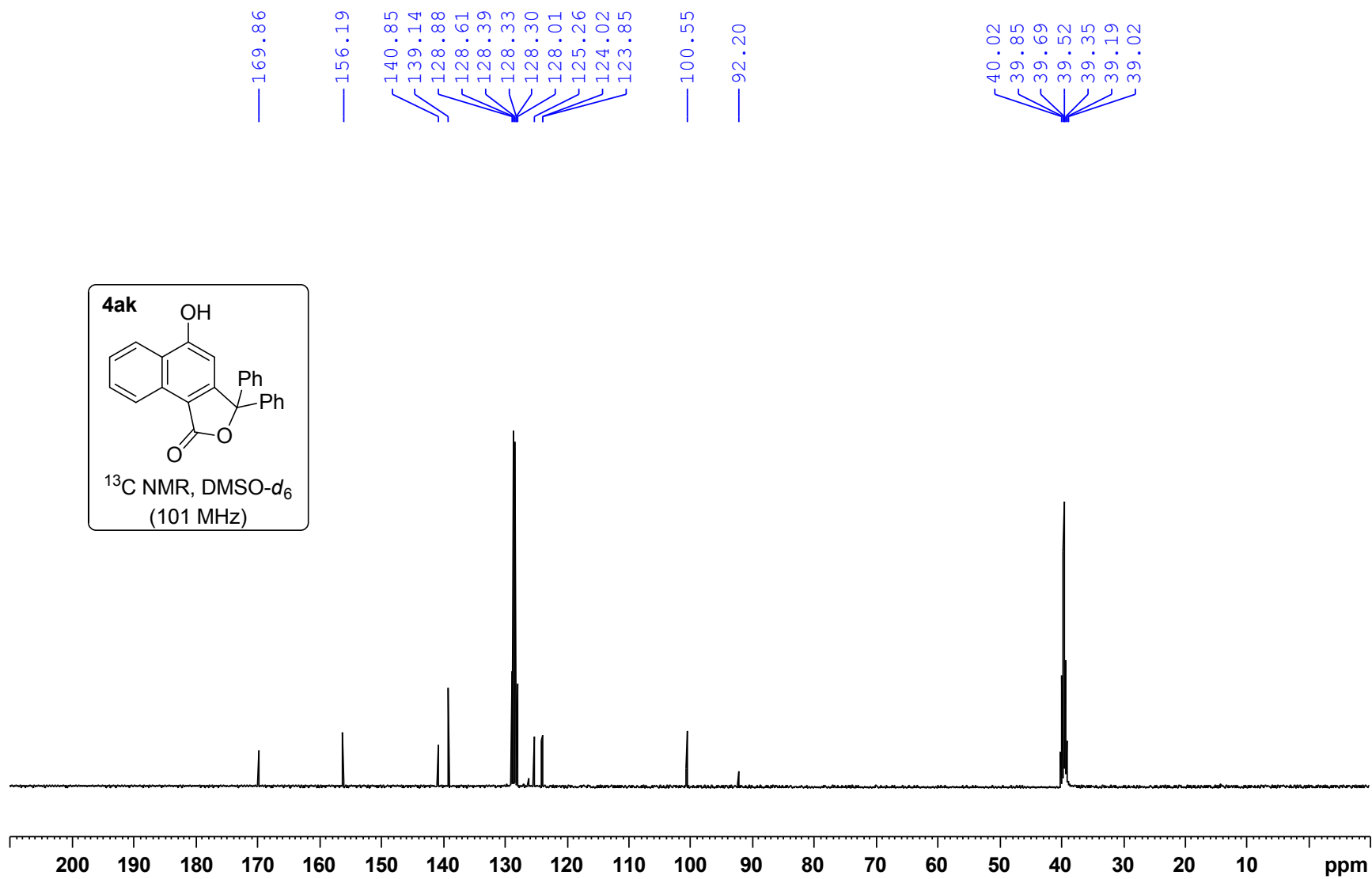
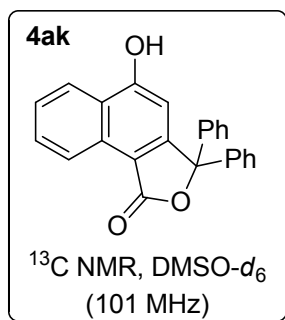


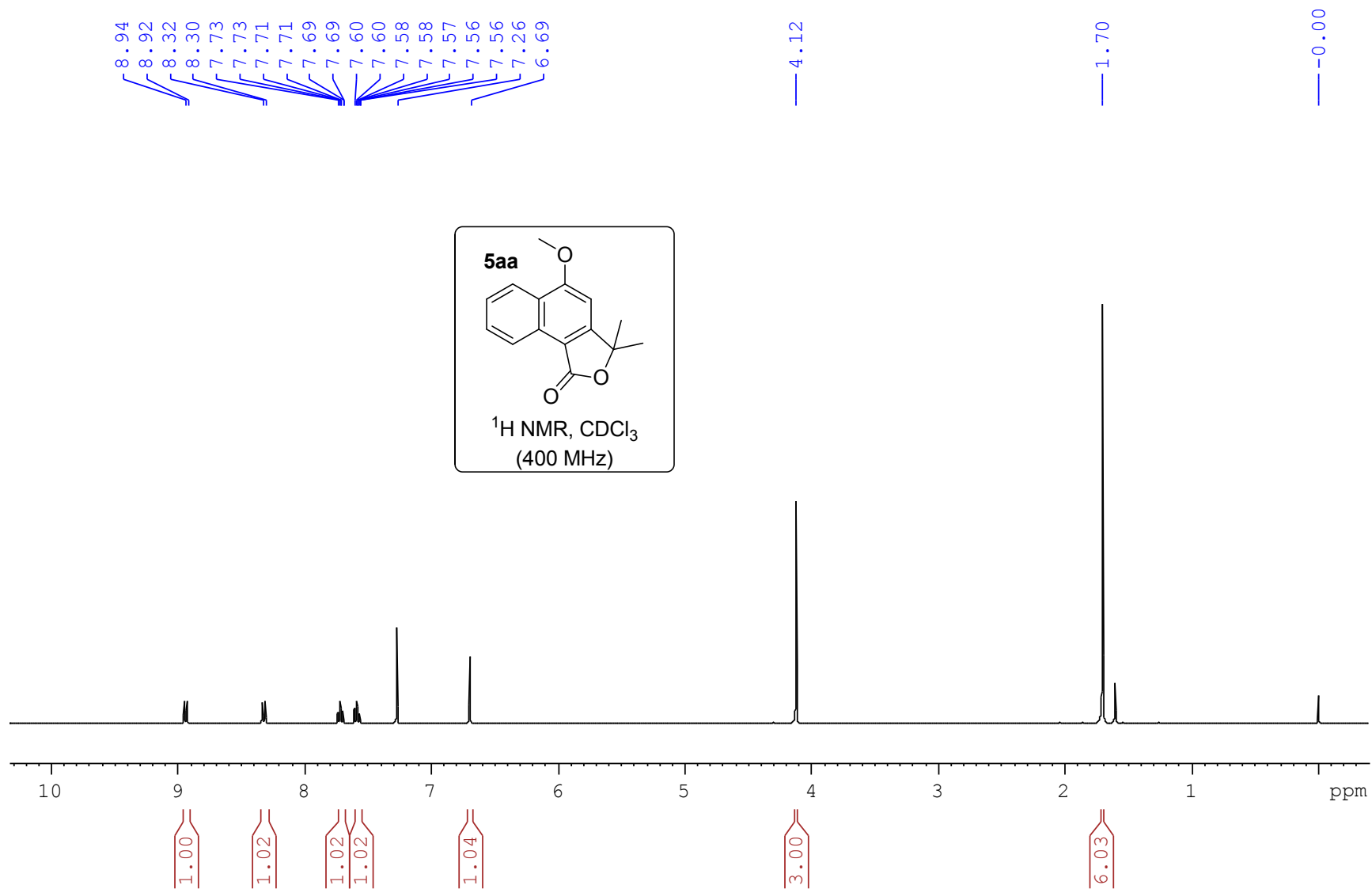
VH-02-AK9

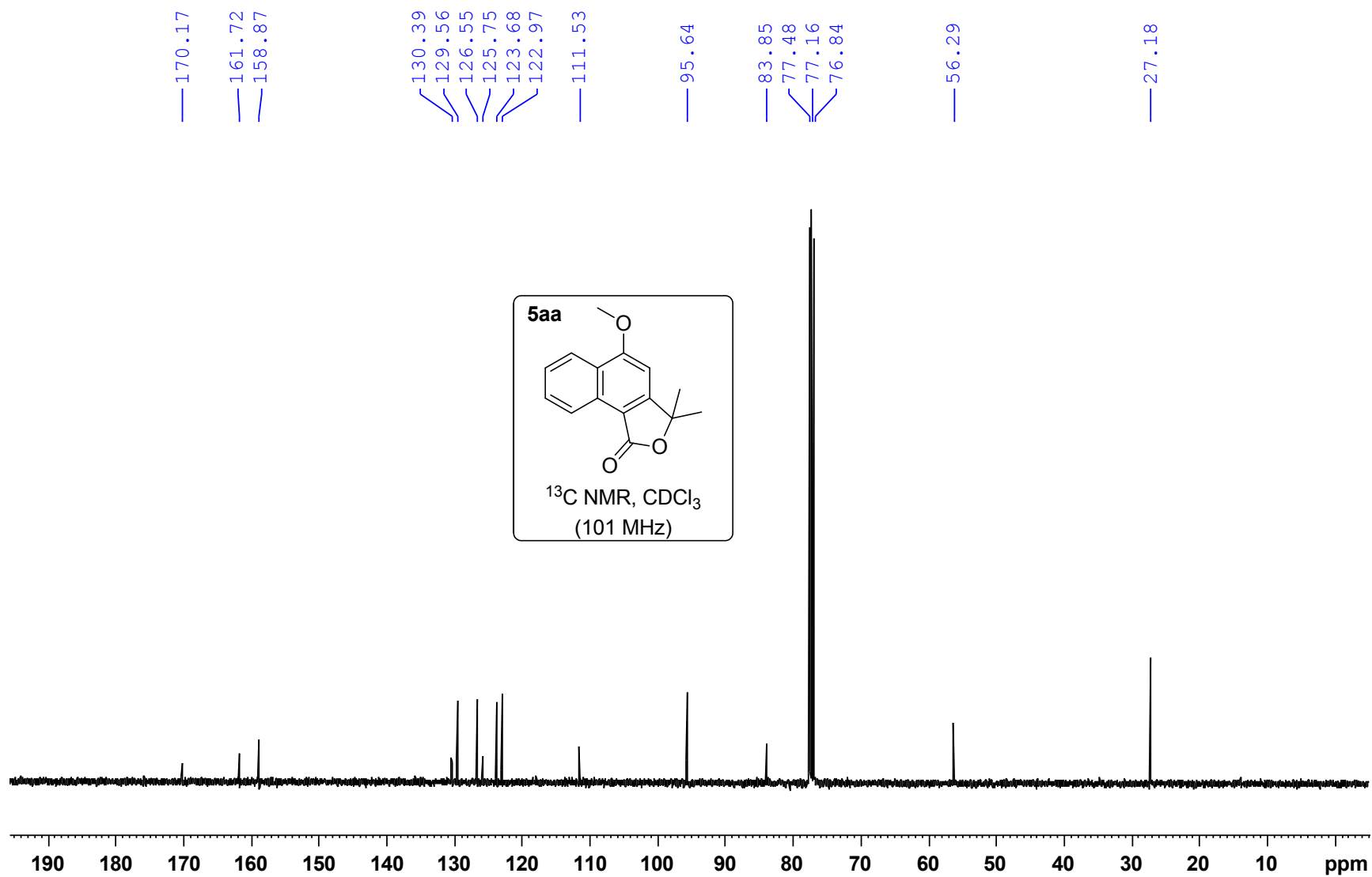


— 2.50

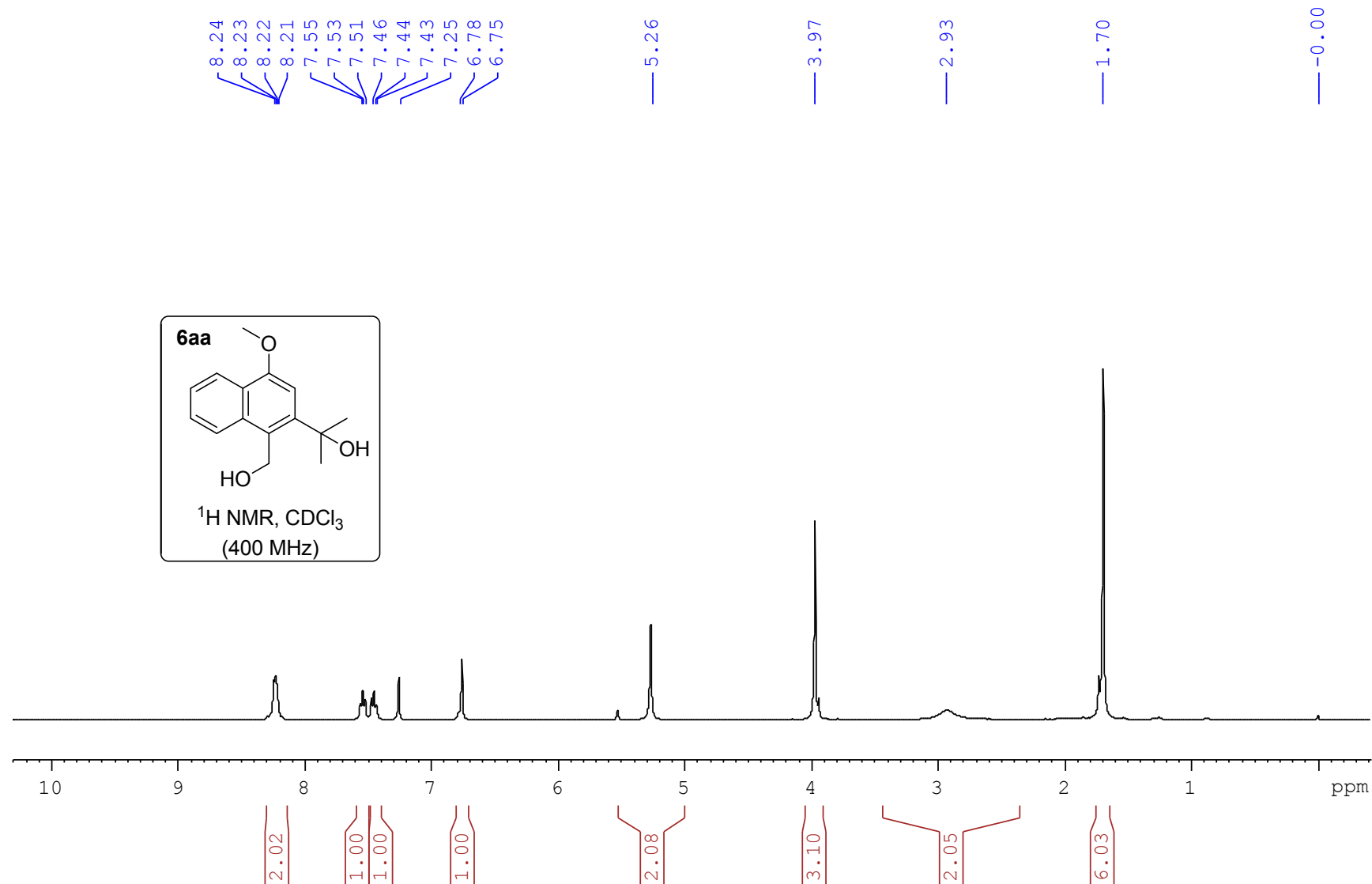








VH-02-206



VH-02-206

