Iron(III)-Mediated Bicyclization of 1,2-Allenyl Aryl Ketones: Assembly of Indanone-Fused Polycyclic Scaffolds and Dibenzo[*a,e*]pentalene Derivatives

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Supporting Information

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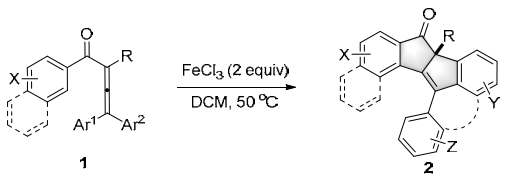
1. General Methods.

NMR spectra were recorded on a Bruker AV-400 MHz spectrometer. The ^{1}H NMR (400 MHz) chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) in $CDCl_3$ and solvent signals (2.50 ppm) in DMSO-d_6. The coupling constants, *J* values were reported in Hertz (Hz). The ^{13}C NMR (100 MHz) chemical shifts were referenced to the internal solvent signals (77.0 ppm in $CDCl_3$, 39.52 ppm in DMSO-d_6). High-resolution mass spectra (HRMS) were recorded on a Waters TOFMS GCT Premier using ESI ionization. UV-Vis spectra were taken on a HITACHI U3010 Spectrophotometer. Fluorescence measurements were performed on an Agilent Cary Eclipse Fluorescence Spectrophotometer. Melting points were measured with WRR digital point apparatus and not corrected. All commercial reagents were used without additional purification and solvents were dried by standard methods when necessary. The anhydrous $FeCl_3$ (AR) was purchased from Aladdin Reagent Company, Inc. Petroleum ether referred to the fraction with boiling point in the range 60−90 °C. All reactions were monitored by TLC with GF 254 silica gel coated plates. Flash column chromatography was carried out using 200−300 mesh silica gel. The materials 1,3,3-triarylpropyne[1], 2-methyl-1,4,4-triphenylbuta-2,3-dien-1-one **2o**[2] and 4-(4-*iso*butylphenyl)-2-methyl-1-phenylpenta-2,3-dien-1-one **2z**[2] was prepared according to the literature procedure.

2. Procedure and experiment data for indanone-fused polycyclic compounds 2

(a) General procedure:

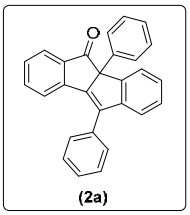


The solution of *tetra*-substituted allenyl ketones **1** (0.2 mmol, 1.0 equiv) in 2 mL of DCM was added

$FeCl_3$ (0.4 mmol, 2.0 equiv) in the open air. Then the vessel was sealed and submerged in an oil bath preheated to 50 ^oC for 12 h. After completion of the reaction, the mixture was quenched by adding 10 mL of H_2O and extracted with EtOAc (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford indanone-fused polycycles **2**.

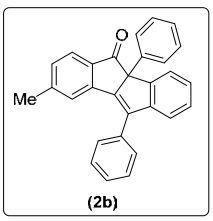
(b) Experiment data:

4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2a)



The reaction of 1,2,4,4-tetraphenylbuta-2,3-dien-1-one **1a** (74.8 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2a** (64.7 mg, 87%) as a yellow solid; **5.580 g (15 mmol) scale reaction**: The reaction of 1,2,4,4-tetraphenylbuta-2,3-dien-1-one **1a** (5.580 g, 15 mmol, 1.0 equiv), $FeCl_3$ (4.860 g, 30 mmol, 2.0 equiv), in 150 mL of DCM at 50 ^oC for 12 h afforded **2a** (4.456 g, 80%) as a yellow solid; m.p. 167-168 ^oC (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.77-7.72 (m, 3H), 7.71-7.67 (m, 3H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.57 (t, *J* = 7.4 Hz, 2H), 7.51-7.47 (m, 1H), 7.43-7.38 (m, 2H), 7.29-7.24 (m, 4H), 7.22 (s, 1H), 7.20-7.17 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.9, 146.5, 145.6, 145.2, 144.5, 144.1, 139.9, 139.1, 134.9, 133.8, 129.6, 128.9, 128.7, 128.6, 128.1, 127.8, 127.7, 126.9, 126.2, 125.0, 124.6, 122.7, 121.9, 74.1; HRMS (ES^+-TOF) calcd for $C_{28}H_{19}O$ ($[M+H]^+$): 371.1430, found 371.1433.

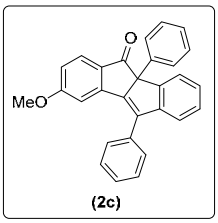
8-methyl-4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2b)



The reaction of 2,4,4-triphenyl-1-(*p*-tolyl)buta-2,3-dien-1-one **1b** (77.2 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2b** (72.6 mg, 94%) as a yellow solid; m.p. 194-196 ^oC

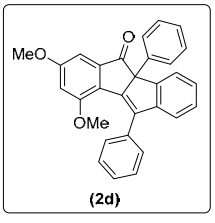
(Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76-7.71 (m, 3H), 7.68-7.63 (m, 3H), 7.58 (t, *J* = 7.6 Hz, 2H), 7.51-7.49 (m, 1H), 7.43 (s, 1H), 7.38-7.37 (m, 1H), 7.26-7.22 (m, 4H), 7.19-7.18 (m, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.4, 146.6, 146.0, 145.6, 145.3, 144.4, 142.3, 140.2, 138.7, 134.0, 129.6, 129.3, 128.9, 128.7, 128.5, 127.7, 127.5, 126.8, 126.2, 124.8, 124.6, 123.2, 121.8, 74.4, 22.2; HRMS (ES$^+$-TOF) calcd for $C_{29}H_{21}O$ ($[M+H]^+$): 385.1587, found 385.1595.

8-methoxy-4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2c)



The reaction of 1-(4-methoxyphenyl)-2,4,4-triphenylbuta-2,3-dien-1-one **1c** (80.9 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2c** (75.0 mg, 92%) as a yellow solid; m.p. 197-198 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.26 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76-7.74 (m, 1H), 7.72-7.67 (m, 5H), 7.56 (t, *J* = 7.2 Hz, 2H), 7.50-7.46 (m, 1H), 7.39-7.37 (m, 1H), 7.28-7.22 (m, 4H), 7.20-7.16 (m, 1H), 7.08 (d, *J* = 2.0 Hz, 1H), 6.75 (dd, J_1 = 4.4 Hz, J_2 = 1.0 Hz, 1H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.2, 165.1, 146.5, 146.4, 145.5, 145.4, 140.3, 139.2, 137.6, 133.9, 129.6, 128.85, 128.76, 128.5, 127.7, 127.5, 127.0, 126.7, 126.2, 124.7, 121.9, 114.8, 107.0, 74.2, 55.5; HRMS (ES$^+$-TOF) calcd for $C_{29}H_{21}O_2$ ($[M+H]^+$): 401.1536, found 401.1546.

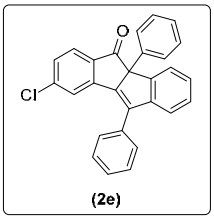
7,9-dimethoxy-4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2d)



The reaction of 1-(3,5-dimethoxyphenyl)-2,4,4-triphenylbuta-2,3-dien-1-one **1d** (86.5 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2d** (40.8 mg, 47%) as a yellow solid; m.p. 156-158 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 5/1); ^{1}H NMR (400 MHz,

$CDCl_3$): δ 7.74-7.71 (m, 1H), 7.68-7.66 (m , 2H), 7.48-7.38 (m, 5H), 7.23-7.14 (m, 6H), 6.87 (d, *J* = 1.0 Hz, 1H), 6.55 (d, *J* = 1.0 Hz, 1H), 3.79 (s, 3H), 3.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 161.4, 156.3, 147.4, 147.1, 144.0, 143.8, 140.4, 135.4, 134.8, 129.1, 128.9, 127.9, 127.7, 127.6, 127.4, 126.2, 125.9, 124.0, 121.5, 106.1, 99.2, 75.3, 55.8, 55.0; HRMS (ES^+-TOF) calcd for $C_{30}H_{23}O_3$ ($[M+H]^+$): 431.1642, found 431.1646.

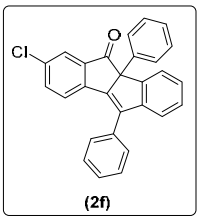
8-chloro-4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2e)



(2e)

The reaction of 1-(4-chlorophenyl)-2,4,4-triphenylbuta-2,3-dien-1-one **1e** (81.4 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2e** (73.0 mg, 89%) as a yellow solid; m.p. 188-189 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.73 (m, 1H), 7.70-7.68 (m, 2H), 7.67-7.64 (m, 3H), 7.61-7.57 (m, 3H), 7.53-7.50 (m, 1H), 7.40-7.38 (m, 1H), 7.29-7.23 (m, 4H), 7.21-7.18 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.6, 145.3, 144.8, 142.8, 141.4, 140.6, 139.5, 133.4, 129.5, 129.1, 129.0, 128.8, 128.4, 127.9, 127.8, 127.3, 126.1, 126.0, 124.7, 122.8, 122.2, 74.4; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}ClO$ ($[M+H]^+$): 405.1041, found 405.1049.

7-chloro-4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2f)

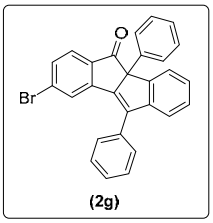


(2f)

The reaction of 1-(3-chlorophenyl)-2,4,4-triphenylbuta-2,3-dien-1-one **1f** (81.4 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2f** (71.2 mg, 87%) as a yellow solid; m.p. 140-142 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.73 (m, 1H), 7.71-7.68 (m, 3H), 7.66-7.64 (m, 2H), 7.59-7.55 (m, 3H), 7.52-7.50 (m, 1H), 7.40-7.34 (m. 2H), 7.29-7.20 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.7, 145.7, 145.4, 145.1,

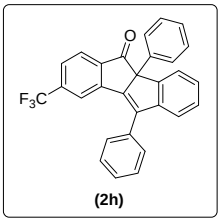
145.0, 142.2, 139.8, 139.3, 134.8, 134.5, 133.6, 129.5, 129.1, 128.9, 128.7, 127.91, 127.87, 127.2, 126.2, 125.0, 124.6, 123.6, 122.1, 74.5; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}ClO$ ($[M+H]^+$): 405.1041, found 405.1042.

8-bromo-4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2g)



The reaction of 1-(4-bromophenyl)-2,4,4-triphenylbuta-2,3-dien-1-one **1g** (90.2 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2g** (80.5 mg, 93%) as a yellow solid; m.p. 213-215 ^oC (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.76-7.73 (m, 2H), 7.70-7.68 (m, 2H), 7.65-7.58 (m, 5H), 7.54-7.50 (m, 1H), 7.40-7.36 (m, 2H), 7.30-7.26 (m, 3H), 7.23-7.20 (m, 2H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.8, 145.4, 145.2, 144.7, 143.2, 140.6, 139.4, 133.4, 131.3, 130.2, 129.5, 129.1, 129.0, 128.8, 127.9, 127.8, 127.3, 126.14, 126.10, 125.8, 124.7, 122.3, 74.3; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}BrO$ ($[M+H]^+$): 449.0536, found 449.0544.

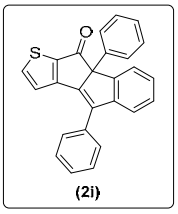
4b,10-diphenyl-8-(trifluoromethyl)indeno[2,1-a]inden-5(4bH)-one (2h)



The reaction of 2,4,4-triphenyl-1-(4-(trifluoromethyl)phenyl)buta-2,3-dien-1-one **1h** (88.1 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2g** (74.0 mg, 83%) as a yellow solid; m.p. 214-216 ^oC (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.87-7.82 (m, 2H), 7.76-7.71 (m, 3H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.60 (t, *J* = 7.4 Hz, 2H), 7.55-7.48 (m, 2H), 7.44-7.42 (m, 1H), 7.32-7.21 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.0, 146.9, 145.2, 144.6, 144.2, 141.1, 139.0, 136.0 (q, $^2J_{C\text{-}F}$ = 32.0 Hz), 133.2, 129.4, 129.3, 129.1, 128.8, 128.0, 127.5, 126.2, 125.4, 124.9 (q, $^3J_{C\text{-}F}$ = 1.7 Hz), 124.7, 123.3 (q, $^1J_{C\text{-}F}$ = 275.0 Hz), 122.4, 119.5

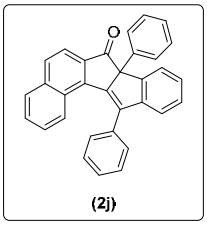
(broad), 74.6; HRMS (ES^+-TOF) calcd for $C_{29}H_{18}F_3O$ ($[M+H]^+$): 439.1304, found 439.1311.

4,8b-diphenylbenzo[4,5]pentaleno[2,1-b]thiophen-9(8bH)-one (2i)



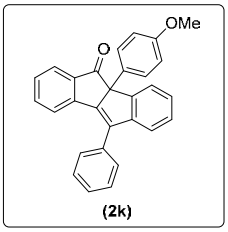
The reaction of 2,4,4-triphenyl-1-(thiophen-2-yl)buta-2,3-dien-1-one **1i** (75.7 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2i** (43.1 mg, 57%) as a yellow solid; m.p. 174-176 ^oC (Petroleum ether/EtOAc); R*f* = 0.50 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.79-7.62 (m, 6H), 7.59-7.41 (m, 4H), 7.32-7.15 (m, 5H), 7.08 (d, *J* = 4.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 186.5, 157.1, 146.5, 145.4, 144.7, 143.3, 140.8, 140.4, 135.7, 133.8, 129.0, 128.83, 128.78, 128.7, 127.8, 127.6, 127.2, 126.1, 124.6, 122.1, 121.2, 77.9; HRMS (ES^+-TOF) calcd for $C_{26}H_{17}OS$ ($[M+H]^+$): 377.0995, found 377.0998.

7a,12-diphenylbenzo[4,5]pentaleno[1,2-a]naphthalen-7(7aH)-one (2j)



The reaction of 1-(naphthalen-2-yl)-2,4,4-triphenylbuta-2,3-dien-1-one **1j** (84.6 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2j** (60.6 mg, 72%) as a yellow solid; m.p. 238-240 ^oC (Petroleum ether/EtOAc); R*f* = 0.50 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.84-7.79 (m, 3H), 7.75-7.70 (m, 3H), 7.51-7.45 (m, 2H), 7.40-7.34 (m, 5H), 7.23-7.14 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.7, 146.9, 145.6, 145.5, 144.6, 143.4, 140.3, 137.1, 136.7, 134.5, 129.04, 128.98, 128.9, 128.5, 128.2, 127.9, 127.6, 127.5, 126.8, 126.5, 126.1, 124.4, 122.0, 120.5, 76.2; HRMS (ES^+-TOF) calcd for $C_{32}H_{21}O$ ($[M+H]^+$): 421.1587, found 421.1591.

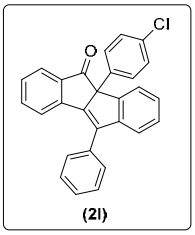
4b-(4-methoxyphenyl)-10-phenylindeno[2,1-a]inden-5(4bH)-one (2k)



The reaction of 2-(4-methoxyphenyl)-1,4,4-triphenylbuta-2,3-dien-1-one **1k** (80.4 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of

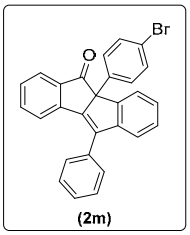
DCM at 50 °C for 12 h afforded **2k** (75.6 mg, 94%) as a yellow solid; m.p. 151-153 °C (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77-7.66 (m, 4H), 7.65-7.51 (m, 5H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.40-7.32 (m, 2H), 7.27-7.16 (m, 3H), 6.76 (d, *J* = 8.8 Hz, 2H), 3.65 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.9, 159.0, 146.6, 145.5, 145.4, 144.4, 143.9, 138.9, 134.7, 133.8, 131.8, 129.5, 128.6, 128.5, 128.1, 127.6, 127.3, 126.8, 124.9, 124.5, 122.6, 121.8, 114.3, 73.4, 55.1; HRMS (ES^+-TOF) calcd for $C_{29}H_{21}O_2$ ($[M+H]^+$): 401.1536, found 401.1540.

4b-(4-chlorophenyl)-10-phenylindeno[2,1-a]inden-5(4bH)-one (2l)



The reaction of 2-(4-chlorophenyl)-1,4,4-triphenylbuta-2,3-dien-1-one **1l** (81.4 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 °C for 12 h afforded **2l** (62.7 mg, 77%) as a yellow solid; m.p. 169-171 °C (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76-7.69 (m, 4H), 7.64-7.55 (m, 5H), 7.52-7.48 (m, 1H), 7.45-7.38 (m, 2H), 7.31-7.27 (m, 3H), 7.21 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 146.1, 145.5, 144.9, 144.3, 144.0, 139.5, 138.4, 135.1, 133.6, 129.6, 129.1, 128.85, 128.77, 128.6, 128.3, 128.0, 127.6, 127.1, 125.1, 124.6, 122.7, 122.1, 73.5; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}ClO$ ($[M+H]^+$): 405.1041, found 405.1038.

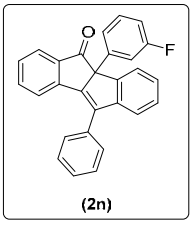
4b-(4-bromophenyl)-10-phenylindeno[2,1-a]inden-5(4bH)-one (2m)



The reaction of 2-(4-bromophenyl)-1,4,4-triphenylbuta-2,3-dien-1-one **1m** (90.2 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 °C for 12 h afforded **2m** (82.0 mg, 91%) a yellow solid; m.p. 188-190 °C (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 7.8 Hz, 1H), 7.72-7.69 (m, 3H), 7.63 (d, *J* = 13.6 Hz, 1H), 7.69-7.48 (m, 5H), 7.45-7.35 (m, 4H),

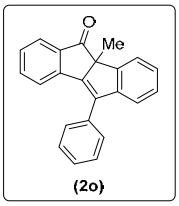
7.31-7.25 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 146.0, 145.5, 144.8, 144.3, 143.9, 139.5, 139.0, 135.1, 133.6, 132.0, 129.5, 128.9, 128.6, 128.3, 128.0, 127.1, 125.1, 124.5, 122.7, 122.1, 121.8, 73.5; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}BrO$ ($[M+H]^+$): 449.0536, found 449.0539.

4b-(3-fluorophenyl)-10-phenylindeno[2,1-a]inden-5(4bH)-one (2n)



The reaction of 2-(3-fluorophenyl)-1,4,4-triphenylbuta-2,3-dien-1-one **1n** (78.0 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2n** (45.8 mg, 58%) as a yellow solid; m.p. 152-154 ^oC (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77-7.70 (m, 4H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.58 (t, *J* = 7.8 Hz, 2H), 7.52-7.47 (m, 2H), 7.45-7.35 (m, 3H), 7.31-7.25 (m, 3H), 7.23-7.21 (m, 1H), 6.90-6.88 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 162.9 (d, $^1J_{C\text{-}F}$ = 245.2 Hz), 146.0, 145.6, 144.7, 144.3, 144.1, 142.2 (d, $^3J_{C\text{-}F}$ = 7.4 Hz) , 139.6, 135.1, 133.6, 130.3 (d, $^3J_{C\text{-}F}$ = 8.1 Hz), 129.6. 128.9, 128.6, 128.3, 128.0, 127.1, 125.1, 124.6, 122.8, 122.1, 122.0, 114.7 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 113.4 (d, $^2J_{C\text{-}F}$ = 22.9 Hz), 73.7; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}FO$ ($[M+H]^+$): 389.1336, found 389.1340.

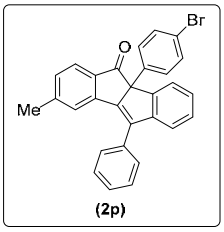
4b-methyl-10-phenylindeno[2,1-a]inden-5(4bH)-one (2o)



The reaction of 2-methyl-1,4,4-triphenylbuta-2,3-dien-1-one **1o** (62.1 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2o** (46.8 mg, 75%) as a yellow solid; m.p. 112-114 ^oC (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, *J* = 7.8 Hz, 1H), 7.78-7.76 (m, 1H), 7.64-7.60 (m, 3H), 7.53 (t, *J* = 7.4 Hz, 2H), 7.47-7.44 (m, 2H), 7.42-7.40 (m, 1H), 7.34-7.32 (m, 3H), 1.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.8, 149.1, 145.40, 145.37, 143.6, 143.4, 136.9, 134.8, 134.1, 129.5, 128.5, 128.0, 127.7, 126.7, 125.1, 123.7,

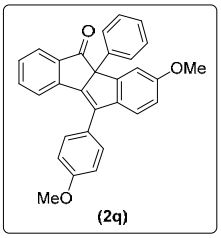
122.9, 121.9, 66.5, 26.9; HRMS (ES^+-TOF) calcd for $C_{23}H_{17}O$ ($[M+H]^+$): 309.1274, found 309.1279.

4b-(4-bromophenyl)-8-methyl-10-phenylindeno[2,1-a]inden-5(4bH)-one (2p)



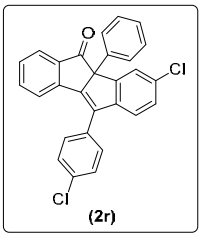
The reaction of 2-(4-bromophenyl)-4,4-diphenyl-1-(*p*-tolyl)buta-2,3-dien-1-one **1p** (93.1 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2p** (82.2 mg, 88%) as a yellow solid; m.p. 193-195 ^oC (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.71-7.65 (m, 3H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.59-7.50 (m, 5H), 7.42 (s, 1H), 7.39-7.34 (m, 3H), 7.28-7.24 (m, 2H), 7.08-7.06 (d, *J* = 8.0 Hz, 1H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.1, 146.3, 146.1, 145.5, 144.8, 144.2, 142.0, 139.3, 139.0, 133.7, 132.0, 129.6, 129.5, 128.8, 128.6, 127.9, 127.0, 124.9, 124.5, 123.2, 122.0, 121.6, 73.7, 22.3; HRMS (ES^+-TOF) calcd for $C_{29}H_{20}BrO$ ($[M+H]^+$): 463.0692, found 463.0693.

3-methoxy-10-(4-methoxyphenyl)-4b-phenylindeno[2,1-a]inden-5(4bH)-one (2q)



The reaction of 4,4-bis(4-methoxyphenyl)-1,2-diphenylbuta-2,3-dien-1-one **1q** (86.6 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2q** (19.8 mg, 23%) as a yellow solid; m.p. 197-199 ^oC (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 5/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 7.8 Hz, 1H), 7.66-7.64 (m, 4H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.42-7.38 (m, 1H), 7.61-7.28 (m, 2H), 7.24-7.19 (m, 4H), 7.09 (d, *J* = 8.8 Hz, 2H), 6.80 (dd, J_1 = 8.4 Hz, J_2 = 2.4 Hz, 1H), 3.92 (s, 3H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.4, 160.0, 159.4, 147.2, 144.8, 144.0, 143.8, 140.2, 139.0, 138.6, 124.9, 130.9, 128.9, 127.6, 127.5, 126.4, 126.2, 125.0, 122.4, 122.3, 114.0, 113.2, 111.2, 73.6, 55.6, 55.4; HRMS (ES^+-TOF) calcd for $C_{30}H_{23}O_3$ ($[M+H]^+$): 431.1642, found 431.1642.

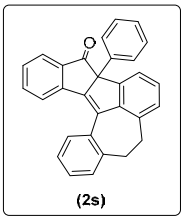
3-chloro-10-(4-chlorophenyl)-4b-phenylindeno[2,1-a]inden-5(4bH)-one (2r)



(2r)

The reaction of 4,4-bis(4-chlorophenyl)-1,2-diphenylbuta-2,3-dien-1-one **1r** (88.4 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2r** (82.8 mg, 95%) as a yellow solid; m.p. 207-209 ^oC (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.2 Hz, 1H), 7.73-7.70 (m, 1H), 7.65-7.51 (m, 7H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.32-7.20 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.8, 147.2, 146.5, 144.4, 143.7, 143.5, 139.0, 137.0, 135.1, 134.8, 133.2, 131.9, 130.7, 129.1, 129.0, 128.6, 128.1, 128.0, 126.1, 125.3, 122.7, 122.3, 74.0; HRMS (ES^+-TOF) calcd for $C_{28}H_{17}Cl_2O$ ($[M+H]^+$): 439.0651, found 439.0651.

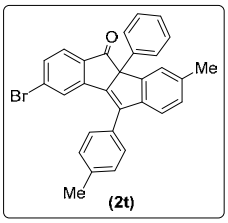
9b-phenyl-6,9b-dihydrodibenzo[cd,h]indeno[1,2-a]azulen-10(5H)-one (2s)



(2s)

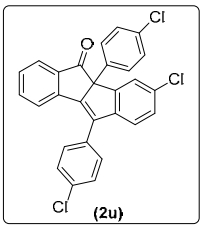
The reaction of 3-(10,11-dihydro-5*H*-dibenzo[*a*,*d*][7]annulen-5-ylidene)-1,2-diphenylprop-2-en-1-one **1s** (79.7 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2s** (62.2 mg, 78%) as a yellow solid; m.p. 205-206 ^oC (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 8.03 (s, 2H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.64-7.58 (m, 3H), 7.50-7.36 (m, 3H), 7.30-7.24 (m, 2H), 7.21-7.17 (m, 3H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.01 (d, *J* = 7.6 Hz, 1H), 3.12-2.97 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.4, 145.1, 144.7, 144.4, 143.0, 142.5, 140.4, 138.6, 134.8, 133.2, 133.0, 129.6, 129.3, 128.9, 128.6, 128.3, 127.5, 126.4, 126.2, 125.7, 125.1, 122.4, 122.2, 77.2, 36.3, 34.1; HRMS (ES^+-TOF) calcd for $C_{30}H_{21}O$ ($[M+H]^+$): 397.1587, found 397.1591.

8-bromo-3-methyl-4b-phenyl-10-(p-tolyl)indeno[2,1-a]inden-5(4bH)-one (2t)



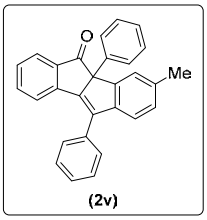
The reaction of 1-(4-bromophenyl)-2-phenyl-4,4-di-*p*-tolylbuta-2,3-dien-1-one **1t** (95.9 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2t** (51.0 mg, 55%) as a yellow solid; m.p. 232-234 ^oC (Petroleum ether/EtOAc); R*f* = 0.35 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* = 1.6 Hz, 1H), 7.64-7.54 (m, 5H), 7.39 (d, *J* = 7.6 Hz, 2H), 7.36-7.33 (m, 1H), 7.29-7.20 (m, 5H), 7.08 (d, *J* = 8.0 Hz, 1H), 2.49 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.4, 145.7, 145.5, 143.3, 142.9, 142.8, 140.8, 139.7, 139.1, 137.5, 130.9, 130.5, 130.2, 129.4, 129.0, 128.6, 127.7, 126.2, 126.0, 125.7, 125.5, 122.0, 74.0, 21.6, 21.5; HRMS (ES^+-TOF) calcd for $C_{30}H_{22}BrO$ ($[M+H]^+$): 477.0849, found 477.0859.

3-chloro-4b,10-bis(4-chlorophenyl)indeno[2,1-a]inden-5(4bH)-one (2u)



The reaction of 2,4,4-tris(4-chlorophenyl)-1-phenylbuta-2,3-dien-1-one **1u** (95.2 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC for 12 h afforded **2u** (76.1 mg, 80%) as a yellow solid; m.p. 243-245 ^oC (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.6 Hz, 1H), 7.68-7.67 (m, 1H), 7.61-7.54 (m, 7H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.32-7.27 (m, 1H), 7.25-7.23 (m, 3H), 7.22-7.21 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 146.8, 146.2, 144.1, 143.6, 143.4, 137.5, 137.4, 135.3, 135.0, 134.0, 133.4, 131.6, 130.7, 129.3, 129.0, 128.7, 128.3, 127.5, 125.3, 125.2, 122.7, 122.5, 73.3; HRMS (ES^+-TOF) calcd for $C_{28}H_{16}Cl_3O$ ($[M+H]^+$): 473.0261, found 473.0276.

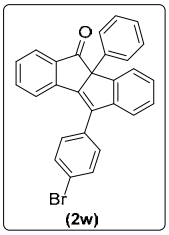
2-methyl-4b,10-diphenylindeno[2,1-a]inden-5(4bH)-one (2v)



The reaction of 1,2,4-triphenyl-4-(*p*-tolyl)buta-2,3-dien-1-one **1v** (77.2 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^oC

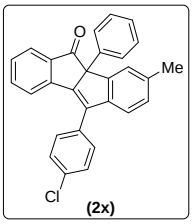
for 12 h afforded **2v** (72.2 mg, 94%) as a yellow solid; m.p. 164-166 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.67 (m, 5H), 7.63-7.54 (m, 4H), 7.50-7.48 (m, 1H), 7.41-7.37 (m, 2H), 7.28-7.25 (m, 2H), 7.23-7.19 (m, 2H), 7.07 (d, *J* = 7.6 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.2, 145.7, 145.5, 144.4, 144.3, 143.0, 140.1, 139.2, 137.1, 134.8, 129.6, 129.5, 129.3, 128.9, 128.6, 128.5, 127.9, 127.6, 126.2, 125.6, 125.0, 122.6, 121.6, 74.0, 21.6; HRMS (ES^+-TOF) calcd for $C_{29}H_{21}O$ ($[M+H]^+$): 385.1587, found 385.1594.

10-(4-bromophenyl)-4b-phenylindeno[2,1-a]inden-5(4bH)-one (2w)



The reaction of 4-(4-bromophenyl)-1,2,4-triphenylbuta-2,3-dien-1-one **1w** (90.3 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2w** (80.5 mg, 89%) as a yellow solid; m.p. 211-213 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76-7.74 (m, 2H), 7.70-7.64 (m, 4H), 7.59-7.56 (m, 3H), 7.43-7.39 (m, 1H), 7.32-7.30 (m, 1H), 7.28-7.25 (m, 2H), 7.24-7.15 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 147.1, 145.2, 145.1, 144.6, 143.7, 139.6, 137.8, 134.9, 132.8, 131.8, 131.1, 129.5, 129.1, 129.0, 128.7, 128.3, 127.9, 127.8, 127.7, 127.1, 126.2, 125.1, 124.7, 122.8, 122.6, 121.6, 74.3; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}BrO$ ($[M+H]^+$): 449.0536, found 449.0534.

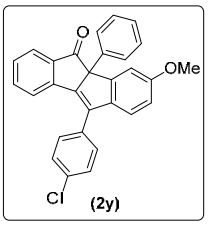
10-(4-chlorophenyl)-2-methyl-4b-phenylindeno[2,1-a]inden-5(4bH)-one (2x)



The reaction of 4-(4-chlorophenyl)-1,2-diphenyl-4-(*p*-tolyl)buta-2,3-dien-1-one **1x** (84.2 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2x** (71.0 mg, 85%) as a yellow solid; m.p. 181-183 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz,

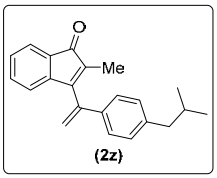
$CDCl_3$): δ 7.73 (d, *J* = 7.6 Hz, 1H), 7.65-7.61 (m, 4H), 7.56 (t, *J* = 8.8 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.24-7.16 (m, 5H), 7.06 (d, *J* = 7.6 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 146.2, 145.4, 144.4, 144.0, 142.6, 139.8, 137.9, 137.3, 134.9, 134.5, 132.5, 130.8, 128.9, 128.8, 128.6, 128.1, 127.6, 126.2, 125.7, 125.1, 122.5, 121.3, 74.0, 21.6; HRMS (ES^+-TOF) calcd for $C_{29}H_{20}ClO$ ($[M+H]^+$): 419.1197, found 419.1194.

10-(4-chlorophenyl)-2-methoxy-4b-phenylindeno[2,1-a]inden-5(4bH)-one (2y)



The reaction of 4-(4-chlorophenyl)-4-(4-methoxyphenyl)-1,2-diphenylbuta-2,3-dien-1-one **1y** (87.4 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2y** (55.1 mg, 63%) as a yellow solid; m.p. 169-172 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.6 Hz, 1H), 7.64-7.62 (m, 4H), 7.56-7.51 (m, 3H), 7.42-7.38 (m, 1H), 7.32 (d, *J* = 2.4 Hz, 1H), 7.25-7.16 (m, 5H), 6.81-6.78 (m, 1H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 159.6, 147.1, 145.2, 144.22, 144.17, 139.8, 138.0, 137.9, 134.9, 134.5, 132.5, 130.8, 129.0, 128.8, 127.9, 127.7, 126.1, 125.1, 122.3, 122.2, 113.3, 111.3, 73.8, 55.6; HRMS (ES^+-TOF) calcd for $C_{29}H_{20}ClO_2$ ($[M+H]^+$): 435.1146, found 435.1146.

3-(1-(4-isobutylphenyl)vinyl)-2-methyl-1H-inden-1-one (2z)

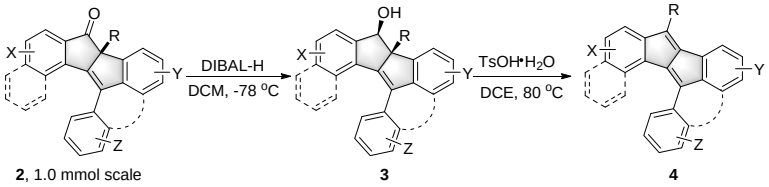


The reaction of 4-(4-*iso*butylphenyl)-2-methyl-1-phenylpenta-2,3-dien-1-one **1z** (60.9 mg, 0.2 mmol, 1.0 equiv), $FeCl_3$ (65 mg, 0.4 mmol, 2.0 equiv), in 2 mL of DCM at 50 ^{o}C for 12 h afforded **2z** (43.0 mg, 70%) as an orange liquid; R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, *J* = 6.8 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.18-7.10 (m, 4H), 6.65 (d, *J* = 6.8 Hz, 1H), 5.91 (s, 1H), 5.41 (s, 1H), 2.47 (d, *J* = 7.2 Hz, 2H), 1.88-1.85 (m, 1H), 1.79 (s, 3H), 0.90 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ

198.3, 155.4, 146.1, 142.2, 141.2, 135.3, 133.3, 132.6, 130.8, 129.4, 127.8, 126.4, 122.2, 120.9, 116.3, 45.1, 30.1, 22.3, 8.7; HRMS (ES^+-TOF) calcd for $C_{22}H_{23}O$ ($[M+H]^+$): 303.1743, found 303.1749.

3. Access to unsymmetrical dibenzo[*a*,*e*]pentalenes 4 from 2

(a) General procedure:

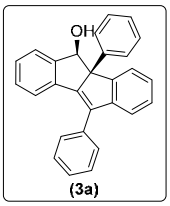


Step one: To a solution of indanone-fused polycyclic compounds **2** (1.0 mmol, 1.0 equiv) in 5 mL of dry DCM at -78 °C under N_2 was added dropwise DIBAL-H (1.5 M solution in toluene, 1.05 mmol, 1.05 equiv). After the solution was stirred at -78 °C for 1.0 h, the reaction was quenched by adding 10 mL of cold water and kept stirring for another 0.5 h. Then the mixture was allowed to warm up to room temperature and extracted with DCM (3×20 mL). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo.* The residue was purified by flash silica gel chromatography to afford reduction products **3** with high regioselectivity.

Step two: The solution of **3** (0.3 mmol, 1.0 equiv) in 3 mL of DCE was added $TsOH{\cdot}H_2O$ (0.6 mmol, 2.0 equiv). This mixture was allowed to stir at 80 ^oC for 12 h. After completion, the reaction was quenched by adding 10 mL of H_2O and extracted with DCM (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford dibenzo[*a*,*e*]pentalenes **4**.

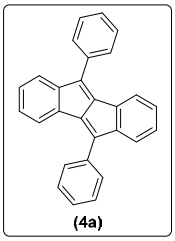
(b) Experiment data for 3 and 4:

cis-4b,10-diphenyl-4b,5-dihydroindeno[2,1-a]inden-5-ol (3a)



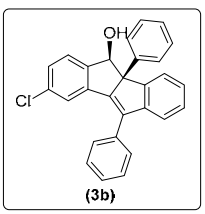
The solution of 4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2a** (370.4 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3a** (357.0 mg, 96%) as a white solid; m.p. 97-98 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, CDCl3): δ 7.90 (d, *J* = 6.8 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.61-7.57 (m, 3H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.35-7.33 (m, 2H), 7.29-7.25 (m, 2H), 7.22-7.15 (m, 5H), 5.11 (d, *J* = 12.0 Hz, 1H), 2.11-2.07 (m, 1H); ^{13}C NMR (100 MHz, CDCl3): δ 152.9, 150.7, 148.8, 146.7, 138.8, 136.1, 135.7, 134.3, 129.4, 129.2, 128.42, 128.35, 128.07, 128.05, 127.4, 127.2, 125.5, 124.5, 124.2, 121.7, 121.4, 77.3, 74.6; HRMS (ES^{+}-TOF) calcd for $C_{28}H_{21}O$ ([M+H]$^{+}$): 373.1587, found 373.1596.

5,10-diphenylindeno[2,1-a]indene (4a)



The solution of *cis*-4b,10-diphenyl-4b,5-dihydroindeno[2,1-*a*]inden-5-ol **3a** (111.8 mg, 0.3 mmol, 1.0 equiv) and TsOH·H_2O (114 mg, 0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded **4a** (86.4 mg, 78%) as a red solid; m.p. 237-238 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.35 (Petroleum ether/EtOAc = 20/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68-7.66 (m, 2H), 7.52 (t, *J* = 7.4 Hz, 2H), 7.46-7.42 (m, 1H), 7.21 (d, *J* = 7.2 Hz, 1H), 7.02 (d, *J* = 7.2 Hz, 1H), 6.92-6.88 (m, 1H), 6.86-6.82 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7, 143.2, 140.7, 135.2, 133.9, 128.8, 128.6, 128.5, 127.8, 127.4, 122.5, 121.9; HRMS (ES^{+}-TOF) calcd for $C_{28}H_{19}$ ([M+H]$^{+}$): 355.1481, found 355.1483. (Spectroscopic data match that previously reported[3])

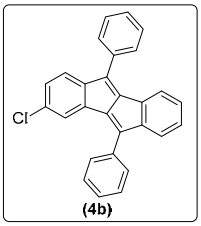
cis-8-chloro-4b,10-diphenyl-4b,5-dihydroindeno[2,1-a]inden-5-ol (3b)



The solution of 8-chloro-4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2e** (409.8 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3b** (342.0 mg, 85%) as a white solid; m.p. 176-177 ^{o}C

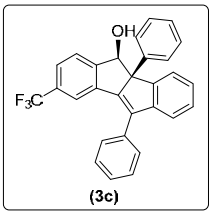
(Petroleum ether/EtOAc); R*f* = 0.20 (Petroleum ether/EtOAc = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.83 (m, 1H), 7.65-7.63 (m, 2H), 7.57-7.50 (m, 5H) 7.46-7.42 (m, 1H), 7.36-7.35 (m, 2H), 7.31-7.26 (m, 2H), 7.24-7.18 (m, 4H), 5.12 (d, *J* = 11.2 Hz, 1H), 2.06-2.03 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.6, 150.3, 148.4, 146.7, 137.5, 136.4, 135.4, 134.1, 133.3, 130.4, 129.3, 128.52, 128.48, 128.4, 128.2, 127.6, 125.7, 124.4, 124.1, 121.9, 121.6, 77.2, 74.2; HRMS (ES^+-TOF) calcd for $C_{28}H_{20}ClO$ ($[M+H]^+$): 407.1197, found 407.1184.

3-chloro-5,10-diphenylindeno[2,1-a]indene (4b)



The solution of *cis*-8-chloro-4b,10-diphenyl-4b,5-dihydroindeno[2,1-*a*]inden-5-ol **3b** (123.3 mg, 0.3 mmol, 1.0 equiv) and $TsOH{\cdot}H_2O$ (114 mg, 0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded **4b** (106.7 mg, 85%) a red solid; m.p. 219-222 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 20/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 7.2 Hz, 2H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.52-7.42 (m, 5H), 7.19 (d, *J* = 6.8 Hz, 1H), 7.14 (d, *J* = 8.8 Hz, 1H), 7.01 (d, *J* = 7.2 Hz, 1H), 6.94 (d, *J* = 7.2 Hz, 1H), 6.89 (t, *J* = 7.2 Hz, 2H), 6.83 (d, *J* = 7.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7, 149.3, 143.5, 143.0, 141.1, 139.1, 135.0, 134.9, 134.5, 133.7, 132.4, 129.8, 129.0, 128.9, 128.7, 128.5, 128.0, 127.8, 127.6, 122.6, 122.1, 122.0, 121.8; HRMS (ES^+-TOF) calcd for $C_{28}H_{17}ClNa$ ($[M+Na]^+$): 411.0911, found 411.0898.

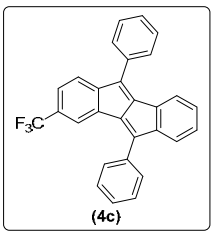
cis-4b,10-diphenyl-8-(trifluoromethyl)-4b,5-dihydroindeno[2,1-a]inden-5-ol (3c)



The solution of 4b,10-diphenyl-8-(trifluoromethyl)indeno[2,1-*a*]inden-5(4b*H*)-one **2h** (442.5 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3c** (404.0 mg, 90%) as a white solid; m.p. 92-95 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91-7.89 (m, 1H), 7.79 (s, 1H), 7.6-7.64 (m, 2H), 7.58-7.53 (m, 4H), 7.48-7.44 (m, 3H),

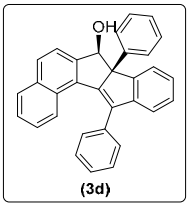
7.39-7.37 (m, 1H), 7.32-7.24 (m, 4H), 7.23-7.17 (m, 1H), 5.11 (d, *J* = 12.0 Hz, 1H), 2.22 (d, *J* = 12.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.3, 148.7, 146.3, 138.2, 138.1, 136.2, 133.6, 130.6 (q, $^2J_{C\text{-}F}$ = 31.2 Hz), 129.2, 129.0, 128.63, 128.58, 127.7, 127.6, 126.1, 124.9 (q, $^3J_{C\text{-}F}$ = 3.6 Hz), 124.6, 124.5, 124.0 (q, $^1J_{C\text{-}F}$ = 270.8 Hz), 121.9, 118.3 (broad), 77.1, 74.8; HRMS (ES^+-TOF) calcd for $C_{29}H_{20}F_3O$ ($[M+H]^+$): 441.1461, found 441.1448.

5,10-diphenyl-3-(trifluoromethyl)indeno[2,1-a]indene (4c)



The solution of *cis*-4b,10-diphenyl-8-(trifluoromethyl)-4b,5-dihydroindeno[2,1-*a*]inden-5-ol **3c** (133.4 mg, 0.3 mmol, 1.0 equiv) and $TsOH{\cdot}H_2O$ (114 mg, 0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded **4c** (96.0 mg, 72%) as a red solid; m.p. 207-210 ^oC (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 20/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67-7.64 (m, 4H), 7.57-7.47 (m, 6H), 7.38 (s, 1H), 7.22 (d, *J* = 7.2 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 1H), 7.11-7.05 (m, 2H), 6.96-6.92 (m, 1H), 6.90-6.86 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.7, 149.4, 145.3, 142.8, 141.6, 139.3, 135.5, 134.9, 133.3 (q, $^3J_{C\text{-}F}$ = 2.4 Hz), 129.7, 129.4, 129.1, 128.9, 128.8, 128.4, 128.0, 124.9 (q, $^3J_{C\text{-}F}$ = 3.7 Hz), 124.3 (q, $^1J_{C\text{-}F}$ = 270.2 Hz), 123.1, 121.9, 118.1 (broad); HRMS (ES^+-TOF) calcd for $C_{29}H_{17}F_3Na$ ($[M+Na]^+$): 445.1175, found 445.1186.

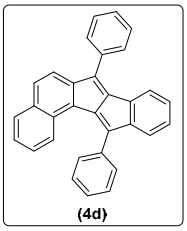
cis-7a,12-diphenyl-7,7a-dihydrobenzo[4,5]pentaleno[1,2-a]naphthalen-7-ol (3d)



The solution of 7a,12-diphenylbenzo[4,5]pentaleno[1,2-*a*]naphthalen-7(7a*H*)-one **2j** (420.5 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3d** (336.0 mg, 80%) as a white solid; m.p. 207-208 ^oC (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81-7.76 (m, 2H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.30 (t, *J* = 7.4 Hz, 1H),

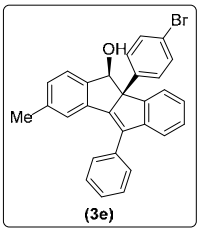
7.23-7.12 (m, 6H), 7.05-6.96 (m, 4H), 6.93-6.90 (m, 2H), 6.86-6.73 (m, 2H), 5.68-5.64 (m, 1H) ,4.48 (d, *J* = 6.4 Hz, 1H), 13.4 (d, *J* = 11.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.1, 143.7, 142.5, 139.7, 138.9, 138.1, 137.8, 133.9, 130.3, 130.1, 129.5, 128.7, 128.2, 127.7, 127.3, 127.03, 126.99, 126.8, 126.5, 125.3, 125.1, 121.8, 76.5, 64.6; HRMS (ES^+-TOF) calcd for $C_{32}H_{23}O$ ($[M+H]^+$): 423.1743, found 423.1746.

7,12-diphenylbenzo[4,5]pentaleno[1,2-a]naphthalene (4d)



The solution of *cis*-7a,12-diphenyl-7,7a-dihydrobenzo[4,5]pentaleno[1,2-*a*] naphthalen-7-ol **3d** (126.8 mg, 0.3 mmol, 1.0 equiv) and $BF_3{\cdot}Et_2O$ (0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded **4d** (103.9 mg, 82%) as a red solid; m.p. 218-220 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 20/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72-7.62 (m, 5H), 7.45-7.31 (m, 8H), 7.25-7.17 (m, 4H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.75 (t, *J* = 7.6 Hz, 1H), 6.50 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.6, 143.5, 142.6, 142.4, 141.6, 136.0, 133.2, 132.7, 132.6, 131.8, 131.4, 129.3, 129.2, 128.9, 128.8, 128.7, 128.6, 128.5, 128.4, 128.3, 128.12, 128.06, 127.7, 127.5, 126.2, 124.9, 123.5, 119.5; HRMS (ES^+-TOF) calcd for $C_{32}H_{21}$ ($[M+H]^+$): 405.1638, found 405.1643.

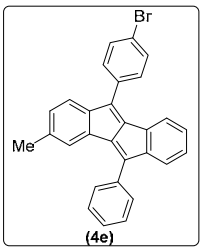
cis-4b-(4-bromophenyl)-8-methyl-10-phenyl-4b,5-dihydroindeno[2,1-a]inden-5-ol (3e)



The solution of 4b-(4-bromophenyl)-8-methyl-10-phenylindeno[2,1-*a*]inden-5(4b*H*)-one **2p** (444.5 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3e** (414.0 mg, 93%) as a white solid; m.p. 168-170 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 20/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.81 (m, 1H), 7.64 (d, *J* = 7.2 Hz, 2H), 7.53 (t, *J* = 7.6 Hz, 2H), 7.47-7.42 (m, 3H), 7.36-7.32 (m, 4H),7.29-7.26 (m, 2H), 7.24-7.22 (m, 1H), 7.03 (d, *J* = 7.6 Hz, 1H),

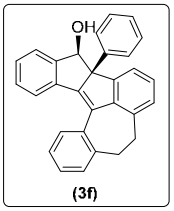
5.08 (d, *J* = 11.6 Hz, 1H), 2.30 (s, 3H), 2.01-1.97 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.3, 149.9, 148.3, 146.7, 138.4, 138.2, 136.0, 135.5, 134.2, 131.3, 130.8, 129.4, 129.1, 128.5, 128.2, 127.6, 125.6, 124.3, 123.8, 122.5, 121.5, 121.4, 74.4, 21.6; HRMS (ES^+-TOF) calcd for $C_{29}H_{21}BrNaO$ ($[M+Na]^+$): 487.0668, found 487.0666.

10-(4-bromophenyl)-3-methyl-5-phenylindeno[2,1-a]indene (4e)



The solution of *cis*-4b-(4-bromophenyl)-8-methyl-10-phenyl-4b,5-dihydroindeno [2,1-*a*]inden-5-ol **3e** (133.8 mg, 0.3 mmol, 1.0 equiv) and $TsOH{\cdot}H_2O$ (114 mg, 0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded **4e** (84.8 mg, 63%) as a red solid; m.p. 197-199 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 20/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66-7.63 (m, 4H), 7.54-7.51 (m, 4H), 7.45 (t, *J* = 7.2 Hz, 1H), 7.14 (d, *J* = 6.8 Hz, 1H), 7.01 (d, *J* = 6.8 Hz, 2H), 6.91-6.82 (m, 3H), 6.70 (d, *J* = 7.6 Hz, 1H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.6, 146.6, 143.0, 142.9, 140.7, 139.5, 137.6, 135.3, 135.0, 133.9, 133.0. 131.9, 130.1, 128.8, 128.7, 128.5, 128.2, 127.8, 127.4, 123.2, 122.7, 122.5, 122.0, 121.7, 21.4; HRMS (ES^+-TOF) calcd for $C_{29}H_{20}Br$ ($[M+H]^+$): 447.0743, found 447.0753.

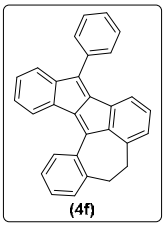
cis-9b-phenyl-5,6,9b,10-tetrahydrodibenzo[cd,h]indeno[1,2-a]azulen-10-ol (3f)



The solution of 9b-phenyl-6,9b-dihydrodibenzo[*cd*,*h*]indeno[1,2-*a*]azulen-10(5*H*)-one **2s** (400.5 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3f** (352.0 mg, 88%) as a white solid; m.p. 153-156 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04-7.66 (m, 3H), 7.61-7.46 (m, 2H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.28-7.07 (m, 9H), 7.02 (d, *J* = 7.6 Hz, 1H), 5.12 (s, 1H), 3.30-2.83 (m, 4H), 2.30-2.03 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.6 (broad), 149.0 (broad), 143.5, 142.5, 139.3, 138.0, 136.1, 133.8, 129.3, 129.1, 128.6, 128.5,

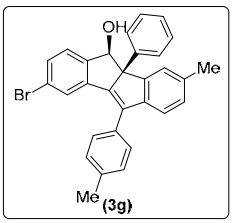
128.3, 128.2, 127.1, 125.6, 124.9, 124.1, 121.9 (broad), 77.3, 35.6 (broad), 34.1; HRMS (ES^+-TOF) calcd for $C_{30}H_{23}O$ ($[M+H]^+$): 399.1743, found 399.1740.

10-phenyl-5,6-dihydrodibenzo[cd,h]indeno[1,2-a]azulene (4f)



The solution of *cis*-9b-phenyl-5,6,9b,10-tetrahydrodibenzo[*cd*,*h*]indeno[1,2-*a*]azulen-10-ol **3f** (119.6 mg, 0.3 mmol, 1.0 equiv) and $TsOH{\cdot}H_2O$ (114 mg, 0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded **4f** (108.0 mg, 91%) as a red solid; m.p. 185-186 ^oC (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 20/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.98-7.92 (m, 1H), 7.66-7.63 (m, 2H), 7.52-7.46 (m, 3H), 7.42 (t, *J* = 7.2 Hz, 1H), 7.32-7.28 (m, 2H), 7.23-7.18 (m, 1H), 7.06-6.99 (m, 2H), 6.92-6.85 (m, 2H), 6.74-6.63 (m, 2H), 3.40-3.92 (m, 2H), 2.90-2.83 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.9, 146.5, 143.4, 143.2, 142.5, 142.2, 139.8, 139.1, 135.5, 135.3, 133.9, 133.7, 131.2, 129.6, 129.5, 129.4, 128.6, 127.6, 127.4, 127.0, 126.0, 122.3, 119.5, 35.6, 33.4; HRMS (ES^+-TOF) calcd for $C_{30}H_{21}$ ($[M+H]^+$): 381.1638, found 381.1627.

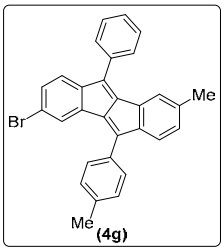
cis-8-bromo-3-methyl-4b-phenyl-10-(p-tolyl)-4b,5-dihydroindeno[2,1-a]inden-5-ol (3g)



The solution of 8-bromo-3-methyl-4b-phenyl-10-(*p*-tolyl)indeno[2,1-*a*]inden-5(4b*H*)-one **2t** (476.5 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3g** (444.0 mg, 93%) as a white solid; m.p. 226-228 ^oC (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 5/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.70-7.68 (m, 2H), 7.57 (d, *J* = 7.2 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.29-7.24 (m, 4H), 7.20-7.16 (m, 2H), 7.09 (d, *J* = 7.6 Hz, 1H), 5.01 (d, *J* = 8.0 Hz, 1H), 2.46 (s, 3H), 2.43 (s, 3H), 2.06 (d, *J* = 12.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.5, 148.9, 147.5, 143.9, 138.6, 138.3, 137.8, 137.7, 135.8, 130.9, 130.5, 129.3, 129.2, 129.0, 128.5, 128.2, 127.4, 126.4, 125.52, 125.49, 124.5, 122.3, 121.5, 74.3, 21.7, 21.4; HRMS (ES^+-TOF)

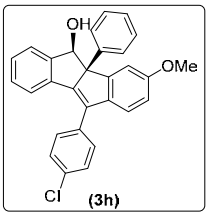
calcd for $C_{30}H_{24}BrO$ ($[M+H]^+$): 479.1005, found 479.0992.

8-bromo-2-methyl-5-phenyl-10-(p-tolyl)indeno[2,1-a]indene (4g)



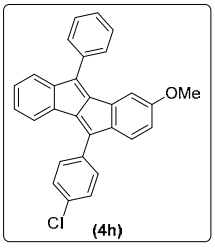
The solution of *cis*-8-bromo-3-methyl-4b-phenyl-10-(*p*-tolyl)-4b,5-dihydroindeno [2,1-a]inden-5-ol **3g** (143.5 mg, 0.3 mmol, 1.0 equiv) and $TsOH{\cdot}H_2O$ (114 mg, 0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded **4g** (107.6 mg, 75%) as a red solid; m.p. 227-229 ^oC (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 20/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 7.2 Hz, 2H), 7.54-7.50 (m, 4H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.35-7.43 (m, 3H), 7.01-7.00 (m, 2H), 6.91 (d, *J* = 7.6 Hz, 1H), 6.85 (d, *J* = 7.6 Hz, 1H), 6.71 (d, *J* = 7.6 Hz, 1H), 2.46 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 146.7, 143.3, 142.8, 140.7, 139.3, 139.2, 137.9, 137.2, 135.5, 133.7, 130.6, 129.8, 129.5, 128.8, 128.7, 128.4, 128.31, 128.27, 124.6, 123.20, 123.16, 122.7, 121.4, 21.5, 21.4; HRMS (ES^+-TOF) calcd for $C_{30}H_{22}Br$ ($[M+H]^+$): 461.0899, found 461.0885.

cis-10-(4-chlorophenyl)-2-methoxy-4b-phenyl-4b,5-dihydroindeno[2,1-a]inden-5-ol (3h)



The solution of 10-(4-chlorophenyl)-2-methoxy-4b-phenylindeno[2,1-*a*]inden-5(4b*H*)-one **2x** (434.9 mg, 1.0 mmol) and DIBAL-H (1.5 M solution in toluene, 0.7 mL, 1.05 mmol) in 5 mL of dry DCM afforded **3h** (365.3 mg, 84%) as a white solid; m.p. 107-109 ^oC (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 5/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.58-7.54 (m, 4H), 7.51-7.47 (m, 4H), 7.36-7.34 (m, 1H), 7.24-7.16 (m, 6H), 6.81 (dd, J_1 = 8.4 Hz, J_2 = 2.4 Hz, 1H), 5.12 (d, *J* = 7.6 Hz, 1H), 3.87 (s, 3H), 2.07 (d, *J* = 12.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.5, 152.5, 150.5, 149.4, 139.4, 138.7, 135.6, 134.7, 133.8, 133.0, 130.6, 129.0, 128.7, 128.4, 127.9, 127.3, 124.2, 121.6, 121.3, 112.2, 112.1, 74.4, 55.7; HRMS (ES^+-TOF) calcd for $C_{29}H_{22}ClO_2$ ($[M+H]^+$): 437.1303, found 437.1311.

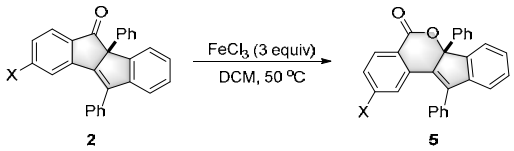
10-(4-chlorophenyl)-2-methoxy-5-phenylindeno[2,1-a]indene (4h)



The solution of *cis*-10-(4-chlorophenyl)-2-methoxy-4b-phenyl-4b,5-dihydroindeno[2,1-*a*]inden-5-ol **3h** (131.1 mg, 0.3 mmol, 1.0 equiv) and $TsOH{\cdot}H_2O$ (114 mg, 0.6 mmol, 2.0 equiv) in 3 mL of DCE afforded 4**h** (78.7 mg, 60%) as red solid; m.p. 204-206 ^oC (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.66-7.59 (m, 4H), 7.53-7.43(m, 5H), 7.15 (d, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 7.6 Hz, 1H), 6.92-6.84 (m, 4H), 6.39-6.36 (m, 1H), 3.71 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.2, 149.2, 142.4, 142.2, 142.1, 140.9, 139.8, 137.0, 135.0, 134.5, 133.8, 132.5, 129.8, 129.0, 128.8, 128.7, 128.5, 128.4, 127.5, 122.7, 122.6, 121.4, 110.6, 110.4, 55.4; HRMS (ES^+-TOF) calcd for $C_{29}H_{20}ClO$ ($[M+H]^+$): 419.1197, found 419.1183.

4. Diverse transformations of 2 for the synthesis of 3D-polycyclic compounds 5-10

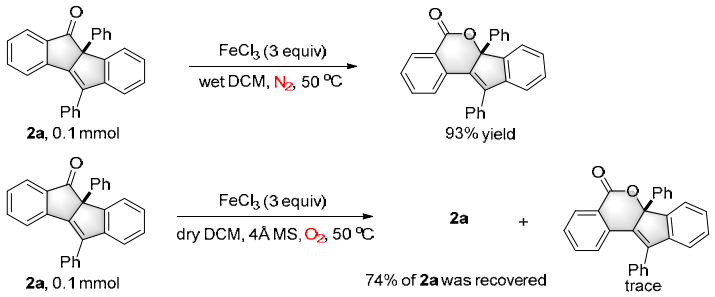
(a) General procedure for the synthesis of compounds 5:



The solution of indanone-fused polycyclic compounds **2** (0.2 mmol, 1.0 equiv) in 2 mL of DCM was added $FeCl_3$ (97 mg, 0.6 mmol, 3.0 equiv). Then the vessel was sealed and submerged in an oil bath preheated to 50 ^oC for 2.5 h. After completion, the mixture was quenched by adding 10 mL of H_2O and extracted with DCM (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford polycyclic compounds **5**.

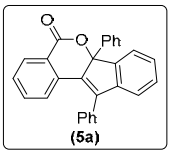
Controlled experiments were carried out: (1) **2a** could be transformed to lactam **5a** in 93% yield

under N_2 protection condition (without O_2). (2) Most of starting material **2a** was recovered when the reaction was conducted in dry DCM, 4Å MS and O_2 balloon condition. These results indicated that the O of **5a** might from H_2O in solvent.



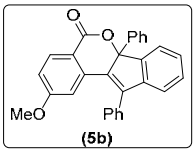
(b) Experiment data for 5:

6a,11-diphenylindeno[1,2-c]isochromen-5(6aH)-one (5a)



The solution of 4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2a** (74.0 mg, 0.2 mmol, 1.0 equiv) and $FeCl_3$ (97 mg, 0.6 mmol, 3.0 equiv) in 2 mL of DCM at 50 ^oC for 2.5 h afford **5a** (66.8 mg, 86%) as a white solid; m.p. 223-226 ^oC (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, *J* = 7.6 Hz, 1H), 7.55-7.45 (m, 7H), 7.41 (d, *J* = 6.8 Hz, 1H), 7.31-7.27 (m, 3H), 7.23-7.17 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.2, 146.5, 143.4, 142.6, 139.8, 135.7, 133.6, 133.3, 132.8, 131.1, 129.3, 129.1, 128.9, 128.7, 128.5, 128.0, 127.7, 125.5, 124.6, 124.1, 123.7, 121.8, 91.6; HRMS (ES^+-TOF) calcd for $C_{28}H_{19}O_2$ ($[M+H]^+$): 387.1380, found 387.1385.

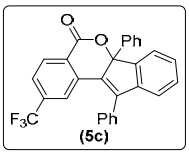
2-methoxy-6a,11-diphenylindeno[1,2-c]isochromen-5(6aH)-one (5b)



The solution of 8-methoxy-4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2c** (80.2 mg, 0.2 mmol, 1.0 equiv) and $FeCl_3$ (97 mg, 0.6 mmol, 3.0 equiv) in 2 mL of DCM at 50 ^oC for 2.5 h afford **5b** (74.2 mg, 89%) as a white solid; m.p. 169-171 ^oC (Petroleum ether/EtOAc); R*f* = 0.4 (Petroleum ether/EtOAc = 5/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, *J* =

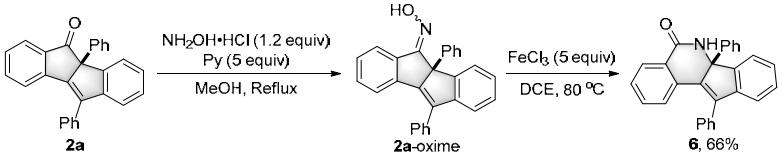
8.8 Hz, 1H), 7.55-7.47 (m, 7H), 7.40 (d, *J* = 6.8 Hz, 1H), 7.27-7.14 (m, 6H), 6.76-6.73 (m, 1H), 6.61 (d, *J* = 2.4 Hz, 1H), 3.53 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.0, 163.4, 146.7, 143.2, 142.7, 139.9, 136.1, 135.1, 133.2, 132.8, 129.2, 129.0, 128.9, 128.8, 127.9, 127.7, 124.6, 124.0, 121.8, 116.3, 115.3, 109.4, 91.5, 55.1; HRMS (ES^+-TOF) calcd for $C_{29}H_{21}O_3$ ($[M+H]^+$): 417.1485, found 417.1494.

6a,11-diphenyl-2-(trifluoromethyl)indeno[1,2-c]isochromen-5(6aH)-one (5c)



The solution of 4b,10-diphenyl-8-(trifluoromethyl)indeno[2,1-*a*]inden-5(4b*H*)-one **2h** (87.6 mg, 0.2 mmol, 1.0 equiv) and $FeCl_3$ (97 mg, 0.6 mmol, 3.0 equiv) in 2 mL of DCM at 50 °C for 2.5 h afford **5c** (47.2 mg, 52%) as a white solid; m.p. 209-211 °C (Petroleum ether/EtOAc); R*f* = 0.35 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (d, *J* = 8.0 Hz, 1H), 7.56-7.54 (m, 5H), 7.50-7.48 (m, 3H), 7.43-7.42 (m, 2H), 7.31-7.27 (m, 3H), 7.25-7.20 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.0, 146.5, 144.6, 142.9, 139.2, 135.0 (q, $^2J_{C\text{-}F}$ = 32.7 Hz), 134.3, 134.0, 131.9, 131.8, 129.6, 129.5, 129.3, 129.1, 128.5, 128.33, 128.30, 126.4, 124.9 (q, $^3J_{C\text{-}F}$ = 3.2 Hz), 123.0 (q, $^1J_{C\text{-}F}$ = 271.6 Hz), 122.5 (q, $^3J_{C\text{-}F}$ = 3.1 Hz), 122.3, 91.8; HRMS (ES^+-TOF) calcd for $C_{29}H_{18}F_3O_2$ ($[M+H]^+$): 455.1253, found 455.1257.

Synthesis of compound 6:

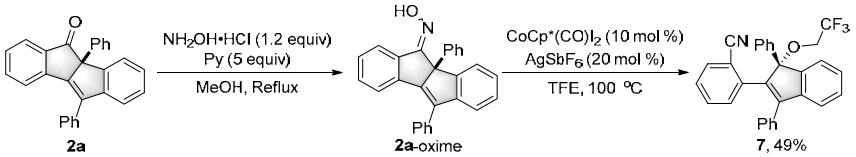


6a,11-diphenyl-6,6a-dihydro-5H-indeno[1,2-c]isoquinolin-5-one (6)

The solution of 4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2a** (74 mg, 0.2 mmol, 1.0 equiv) in 5 mL of MeOH was added pyridine (79 mg, 1.0 mmol, 5.0 equiv) and hydroxylamine hydrochloride (18 mg, 0.24 mmol, 1.2 equiv). Then the vessel was sealed and submerged in an oil bath preheated to

80 °C for 24 h. After completion of the reaction, the solvent was removed under reduced pressure. 10 mL of H_2O was added to the residual solid and extracted with DCM (3×10 mL). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo.* The residue was purified with flash silica gel chromatography to afford **2a**-oxime as a solid, which was used for the next step**.** To a solution of **2a**-oxime in 16 mL of DCE were added $FeCl_3$ (162 mg, 1.0 mmol, 5.0 equiv). Then the mixture was stirred at 80 °C for 5 h. After completion of the reaction, the mixture was quenched by adding 10 mL of H_2O and extracted with DCM (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford **6** (50.9 mg, over all yield: 66%) as a red solid; m.p. 277-279 °C (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 3/1); ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.89 (s, 1H), 7.89-7.87 (m, 1H), 7.57-7.51 (m, 6H), 7.37 (d, *J* = 7.6 Hz, 2H), 7.30-7.21 (m, 6H), 7.15-7.08 (m, 2H), 7.01-7.00 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 166.1, 148.0, 143.6, 142.4, 140.0, 139.5, 132.8, 132.1, 132.0, 129.1, 128.8, 128.7, 128.6, 128.4, 128.3, 128.2, 127.7, 127.2, 127.0, 125.4, 124.8, 123.9, 120.9, 70.7; HRMS (ES^+-TOF) calcd for $C_{28}H_{20}NO$ ($[M+H]^+$): 386.1539, found 386.1541.

Synthesis of compound 7:

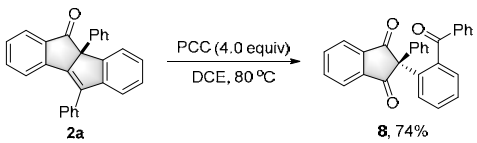


2-(1,3-diphenyl-1-(2,2,2-trifluoroethoxy)-1H-inden-2-yl)benzonitrile (7)

The solution of 4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2a** (74 mg, 0.2 mmol, 1.0 equiv) in 5 mL of MeOH was added pyridine (79 mg, 1.0 mmol, 5.0 equiv) and hydroxylamine hydrochloride (18 mg, 0.24 mmol, 1.2 equiv). Then the vessel was sealed and submerged in an oil bath preheated to

80 ^{o}C for 24 h. After completion of the reaction, the solvent was removed under reduced pressure. 10 mL of H_2O was added to the residual solid and extracted with DCM (3×10 mL). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo.* The residue was purified with flash silica gel chromatography to afford **2a**-oxime as a solid, which was used for next step. To a solution of **2a**-oxime in 6 mL of TFE were added CoCp*(CO)I_2 (9.5 mg, 0.02 mmol, 10 mol %) and $AgSbF_6$ (13.7 mg, 0.04 mmol, 20 mol %). Then the vessel was sealed and submerged in an oil bath preheated to 100 ^{o}C for 4 h. After completion of the reaction, the mixture was quenched by adding 10 mL of H_2O and extracted with DCM (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford **7** (46.2 mg, over all yield: 49%) as a colorless liquid; R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, *J* = 7.6 Hz, 1H), 7.42-7.33 (m, 11H), 7.29-7.26 (m, 4H), 7.23-7.21 (m,1H), 6.99 (s, 1H), 4.01-3.92 (m, 1H), 3.75-3.66 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9, 145.5, 142.8, 140.4, 138.5, 138.1, 133.2, 132.7, 132.1, 132.0, 129.6, 129.5, 128.6, 128.4,. 128.1, 127.9, 127.8, 127.2, 126.9, 125.9, 124.6 (q, *J* = 275.6 Hz), 122.1, 118.2, 113.6, 92.7, 62.3 (q, *J* = 34.3 Hz); HRMS (ES^{+}-TOF) calcd for $C_{30}H_{20}F_3NNaO$ ([M+Na]$^{+}$): 490.1389, found 490.1399.

Synthesis of compound 8:

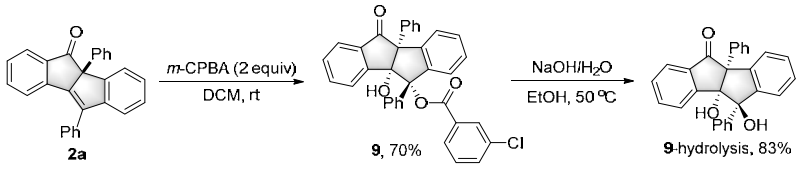


2-(2-benzoylphenyl)-2-phenyl-1H-indene-1,3(2H)-dione (8)

The solution of 4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2a** (74.4 mg, 0.2 mmol, 1.0 equiv) in 4 mL of DCE was added PCC (172 mg, 0.8 mmol, 4.0 equiv). Then the mixture was stirred at 80 ^{o}C

for 4 h. After completion of the reaction, the mixture was quenched by adding 10 mL of H_2O and extracted with EtOAc (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford **8** (59.8 mg, 74%) as a white solid; m.p. 220-222 ^oC (Petroleum ether/EtOAc); R*f* = 0.35 (Petroleum ether/EtOAc = 5/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.92-7.90 (m, 2H), 7.75-7.73 (m, 4H), 7.56-7.54 (m, 1H), 7.46-7.38 (m, 5H), 7.38-7.29 (m, 5H), 6.93 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.6, 198.4, 140.9, 138.5, 137.3, 137.1, 136.6, 135.2, 134.2, 132.8, 131.3, 130.6, 130.4, 129.2, 129.1, 128.3, 128.1, 126.7, 123.5, 69.3; HRMS (ES^+-TOF) calcd for $C_{28}H_{19}O_3$ ($[M+H]^+$): 403.1329, found 403.1338.

Synthesis of compound 9 and its hydrolysis:



(4bR,5R,9bR)-4b-hydroxy-10-oxo-5,9b-diphenyl-4b,5,5a,9a,9b,10-hexahydroindeno[2,1-a]inden-5-yl 3-chlorobenzoate (9)

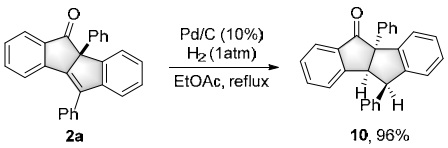
The solution of 4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2a** (72.4 mg, 0.2 mmol, 1.0 equiv) in 2 mL of DCM was added *m*-CPBA (64.8 mg, 0.4 mmol, 2.0 equiv) in the open air. Then the mixture was stirred at 25 ^oC for 48 h. After completion of the reaction, the mixture was quenched by adding 10 mL of H_2O and extracted with EtOAc (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford **9** (76.0 mg, 70%) as a white solid; m.p. 148-150 ^oC (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 5/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.93-7.91 (m, 1H), 7.74-7.70 (m, 1H), 7.61-7.58 (m, 1H), 7.51-7.29 (m, 13H), 7.22-7.15 (m, 2H), 7.03 (d, *J* =

7.6 Hz, 2H), 6.93 (s, 1H), 5.74 (d, *J* = 8.0 Hz, 1H), 3.27 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.0, 162.1, 150.0, 143.8, 138.6, 138.3, 136.0, 135.8, 134.5, 134.1, 133.2, 131.6, 131.4, 130.9, 130.5, 130.1, 129.5, 128.8, 128.7, 128.6, 128.5, 128.4, 128.1, 127.8, 126.5, 123.7, 93.3, 91.3, 73.0; HRMS (ES^+-TOF) calcd for $C_{35}H_24ClO_4$ ($[M+H]^+$): 543.1358, found 543.1364.

(4bS,9bR,10R)-9b,10-dihydroxy-4b,10-diphenyl-9b,10-dihydroindeno[2,1-a]inden-5(4bH)-one (9-hydrolysis)

The solution of (4bR,5R,9bR)-4b-hydroxy-10-*oxo*-5,9b-diphenyl-4b,5,9b,10-tetrahydroindeno[2,1-*a*] inden-5-yl 3-chlorobenzoate **9** (0.1 mmol, 54.3 mg, 1.0 equiv) in 2 mL of EtOH was added NaOH (1 M solution in H_2O, 1 mL) with stiring at 50 ^oC for 4 h. After completion of the reaction, the mixture was quenched by adding 10 mL of H_2O and extracted with EtOAc (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford **9**-hydrolysis. (33.8 mg, 83%) as a white solid; m.p. 170-172 ^oC (Petroleum ether/EtOAc); R*f* = 0.30 (Petroleum ether/EtOAc = 3/1); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.60 (d, *J* = 7.6 Hz, 1 H), 7.43-7.16 (m, 12H), 6.62-6.90 (m, 2H), 6.72 (s, 1H), 5.95 (d, *J* = 7.6 Hz, 1H), 5.09 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 204.2, 153.0, 145.2, 142.7, 141.4, 140.5, 135.2, 134.0, 130.3, 129.6, 129.2, 128.9, 128.3, 128.1, 127.6, 127.3, 126.6, 126.5, 125.9, 122.5, 90.8, 84.3, 73.4; HRMS (ES^+-TOF) calcd for $C_{28}H_{20}NaO_3$ ($[M+Na]^+$): 427.1305, found 427.1314.

Synthesis of compound 10:



2a **10**, 96%

(4bS,9bS,10R)-4b,10-diphenyl-9b,10-dihydroindeno[2,1-a]inden-5(4bH)-one

The solution of 4b,10-diphenylindeno[2,1-*a*]inden-5(4b*H*)-one **2a** (74.4 mg, 0.2 mmol, 1.0 equiv) in 5 mL of EA was added Pd/C (10%, 7.4 mg) with refluxing for 12 h under a hydrogen atmosphere. After completion of the reaction, the mixture was quenched by adding 10 mL of H_2O and extracted with EtOAc (3×10 mL). The combined organic phase was washed with H_2O (3×10 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford **10** (71.4 mg, 96%) a white solid; m.p. 184-187 ^oC (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1); ^1H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.2 Hz, 1H), 7.65-7.63 (m, 1H), 7.32-7.24 (m, 11H), 7.18-7.13 (m, 1H), 7.09-7.07 (m, 2H), 7.04-7.02 (m, 1H), 6.01 (d, *J* = 7.6 Hz, 1H), 5.18 (d, *J* = 9.6 Hz, 1H), 4.32 (d, *J* = 9.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.7, 152.2, 145.3, 143.0, 142.4, 139.4, 136.7, 133.7, 130.4, 128.8, 128.6, 128.5, 128.1, 127.8, 127.7, 127.5, 127.2, 127.0, 126.3, 125.9, 124.0, 70.8, 60.2, 53.0; HRMS (ES^+-TOF) calcd for $C_{28}H_{21}O$ ($[M+H]^+$): 373.1587, found 373.1593.

5. Emission spectra and density functional theory (DFT) bandgap calculation

The cyclopenta-fused skeletons of compounds **2** and **3** might be intriguing units for new functional materials, including organic light emitting diodes, electroluminescence materials, and organic transistors. As a consequence, the fluorescent spectroscopic properties of a series of compounds **2** and **4** were checked as showed. Fluorescence spectra of **2** in both dilute dichloromethane (DCM) solution and solid state were measured (***Figure S1*** and ***S2***). All of tested compounds **2** covered the light ranging from 400 to 650 nm with a maximum at around 490 nm in its DCM solution. Obviously, the nature of substituents attaching to the carbonyl aromatic ring greatly affected the fluorescent intensity (See ***Figure S1***: examples **2b**, **2d**, **2e** and **2h**). For examples, **2d** with strong

electron-donating group 7,9-*di*MeO displayed highest fluorescent intensity while **2h** with strong electron-withdrawing group 8-CF_3 showed the lowest one. The substituents on the benzene ring at the 4-position of compounds **2** had little effect on the fluorescence (See ***Figure S1***: examples **2a**, **2k** and **2l**). Functional groups tethered to indene units remarkably changed the fluorescent emission bands (See ***Figure S1***: examples **2a**, **2q**, **2r** and **2s**). Luminescence wavelength of compound **2q** (about 510 nm) had a remarkable bathochromic shift together with increasing fluorescent intensity comparing with substrates **2a**, **2r** and **2s** because of its donor-acceptor architecture. Moreover, all of the employed compounds also showed fluorescent emission in its solid state (***Figure S2***). Interestingly, compounds **2i** and **2s** exhibited highly emissive in solid state probably due to the influence of S-atom in π-conjugated system in the case of **2i** and the rotation restriction of the phenyl ring in the C11-position of **2s**, respectively. Although no fluorescence of compounds **4** was observed in solid state due to the planar structure of **4** with the existence of intermolecular π–π stacking, these compounds were strongly luminescent in dilute DCM, with maximum emissions at 410 nm and 430 nm, respectively (***Figure S3***). The substituents affected the π-conjugated systems in similar fashions. For examples, the strong electron-donating group on the aromatic ring (**4e**, **4f**, and **4h**) could pronouncedly increase fluorescent intensity and the electron-withdrawing group (**4b** and **4c**) led to decrease of fluorescent intensity. Furthermore, cuvette images of the compounds **2b**, **2d**, **2j**, **2q**, **4e** and **4h** were taken under a handheld UV-lamp (λ_{ex} = 365 nm), **2b**, **2j**, **4e**, **4h** showed blue color and **2d, 2q** displayed light green color (***Figure S4***). Besides, the fluorescent quantum yield of selected compounds **2d** and **2h** was found to be Φ_{2d} = 0.45 and Φ_{4h} = 0.43 based on the standard of quinine sulfate (Φ = 0.55 in 0.1 M H_2SO_4, See ***Figure S5***). Finally, the density functional theory (DFT) bandgap calculation shows that the HOMO-LUMO gap of compounds **2b, 2d, 2h, 2k, 2l** and **2q**

decreases (see ***Figure S6***) with the increase of push-pull electron effect (see ***Figure S1***), which is consistent with their fluorescence response.

Emission spectra:

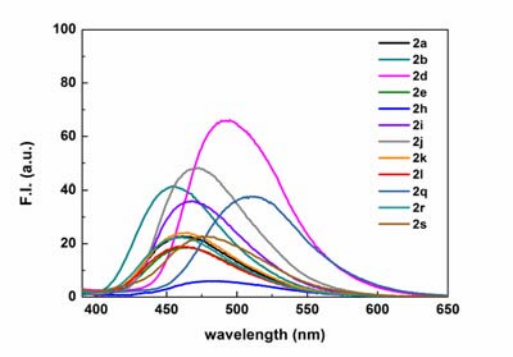


Figure S1. Emission spectra of **2a**–**b**, **2d**–**e**, **2h**–**l**, and **2q**–**s** in DCM (c = 10^{-5} mol/L).

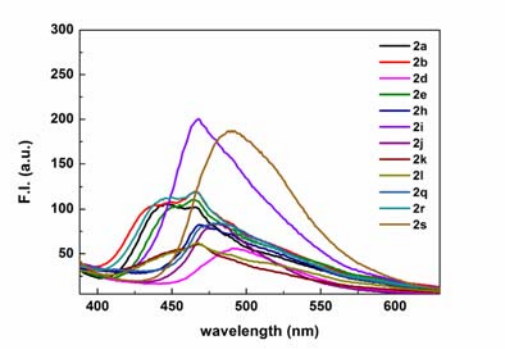


Figure S2. Solid emission spectra of **2a**–**b**, **2d**–**e**, **2h**–**l**, and **2q**–**s**

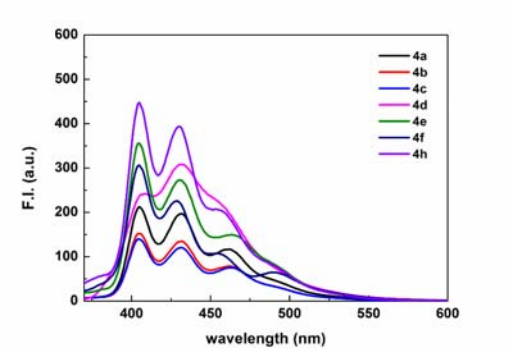


Figure S3. Emission spectra of **4a**–**f**, and **4h** in DCM (c = 10^{-5} mol/L)

Figure S4. Cuvette images of the compounds **2b**, **2d**, **2j**, **2q**, **4e** and **4h** taken under a handheld UV-lamp (λ_{ex} = 365 nm).

Table S1. Emission and excitation wavelength of all compounds in DCM

Compounds	Emission wavelength/nm	Excitation wavelength/nm	Stokes Shift/nm
2a	384	463	79
2b	395	455	60
2d	401	492	91
2e	351	464	113
2h	410	484	74
2i	387	467	80
2j	393	473	80
2k	324	464	140
2l	355	461	106
2q	363	511	148
2r	341	458	117
2s	414	480	66
4a	315	431	116
4b	318	432	114
4c	320	433	113
4d	340	432	92
4e	337	430	93
4f	326	428	102
4h	316	430	114

Sample preparation:

2d UV Absorbance 0.022 in DCM

4h UV Absorbance 0.033 in DCM

Quinine sulfate UV Absorbance 0.024 in 0.1 M H_2SO_4

Quantum yield determination: The quantum yields of samples were determined based on quinine sulfate 0.1 M H_2SO_4 in deionized water.

(Φ_{2d} = 0.458253).

(Φ_{4h} = 0.433607).

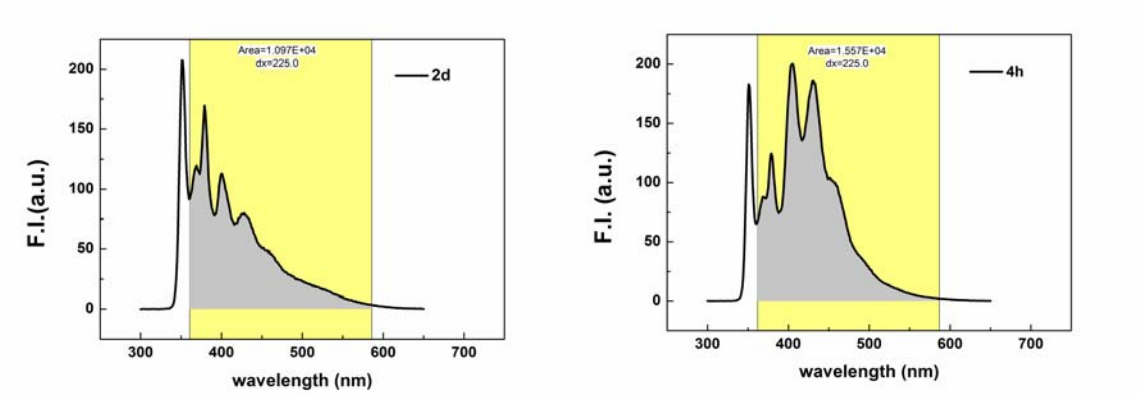
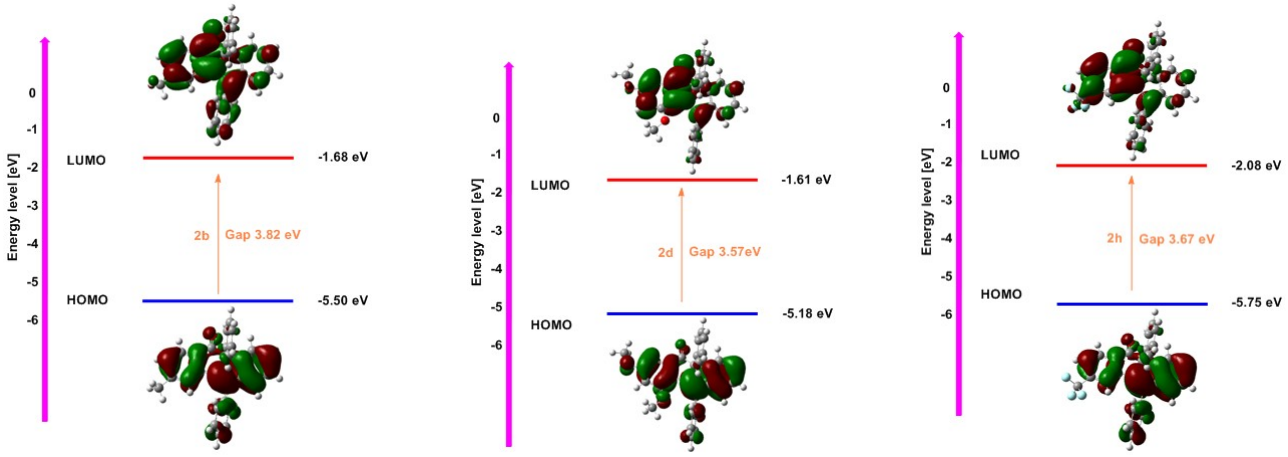


***Figure S5*. UV/FL spectra and quantum yields of 2d and 4h:**

DFT calculations at the B3LYP-D3/6-311G(d,p) level of theory were carried out to optimize the structure and for the calculation of vibrational frequencies. DFT calculations are based on a Gaussian 09 program.

Density functional theory (DFT) calculation:



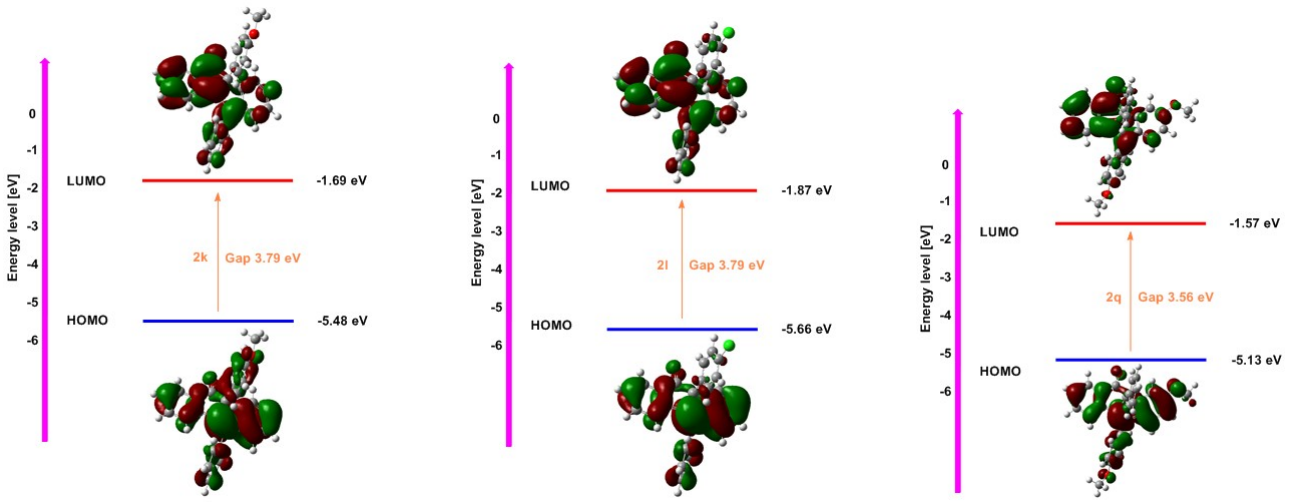


Figure S6. Energy gaps (Δ*E* L/H), HOMO and LUMO energy levels of the frontier molecular orbitals of compounds **2b**, **2d**, **2h**, **2k**, **2l** and **2q**

Cartesian coordinates for all relevant optimized structures

Structure 2b

E(RPBE1PBE) = -229.85041052554658 a.u.

0 1			
C	-0.97384700	4.07157000	-1.29267300
C	-1.89413100	3.54881000	-0.36180500
C	-1.70659200	2.25905500	0.15243300
C	-0.60154900	1.50420300	-0.25143800
C	0.30995200	2.05404300	-1.18397800
C	0.12593400	3.32957300	-1.71271900
C	-0.18633200	0.12528800	0.01061500
C	1.26184400	-0.04343600	-0.43421000
C	1.37815800	1.07608800	-1.50861800
C	-0.83314500	-1.07560000	0.06342200
C	0.10481900	-2.11801000	-0.40912600
C	1.32098100	-1.51209600	-0.80172600
O	2.20315600	1.15050700	-2.39911100
C	-0.06252600	-3.49914300	-0.53579000
C	0.97897100	-4.25657700	-1.07971400
C	2.17350400	-3.64967600	-1.47763400
C	2.35934400	-2.26982600	-1.32235800
C	2.30097600	0.26960900	0.67108400
C	-2.25459100	-1.30725900	0.38085200
C	-2.82591400	-0.75659900	1.54116000

C	-4.17156200	-0.96191000	1.84404600
C	-4.97284500	-1.72462700	0.99338500
C	-4.41770800	-2.28265400	-0.16022400
C	-3.07205900	-2.08198000	-0.46181400
C	1.97586000	0.12340400	2.02571000
C	2.93293900	0.34837600	3.01471800
C	4.23115700	0.72356500	2.66633600
C	4.56349800	0.86853000	1.31971500
C	3.60710900	0.64402000	0.32813800
C	-3.06564200	4.38795700	0.08950800
H	-1.13473700	5.07073200	-1.68881400
H	-2.42372800	1.85348700	0.85786400
H	0.83021600	3.71964500	-2.44121500
H	-0.98363000	-3.97804800	-0.21891700
H	0.85887800	-5.33040100	-1.19047600
H	2.96786100	-4.25369700	-1.90603800
H	3.29348200	-1.80092600	-1.61409800
H	-2.19690400	-0.18613100	2.21814800
H	-4.59132200	-0.53328400	2.74958300
H	-6.02041400	-1.88697000	1.22903300
H	-5.03563900	-2.87378400	-0.82992600
H	-2.65186500	-2.50629000	-1.36798600
H	0.97062700	-0.17279300	2.30603800
H	2.66125800	0.23043200	4.05998300
H	4.97536400	0.90169700	3.43707000
H	5.57003800	1.16142500	1.03474400
H	3.87258000	0.76976600	-0.71610800
H	-3.52459600	4.91652800	-0.75197200
H	-3.83510500	3.77766400	0.56934400
H	-2.74719900	5.14871000	0.81285500

Structure 2d

E(RPBE1PBE) = -229.85041052554658 a.u.

0 1			
C	3.80986000	-1.06160600	0.63928400
C	3.06698200	-1.95801300	-0.15688600
C	1.72748100	-1.72144200	-0.46279400
C	1.10623300	-0.55368700	0.03170000
C	1.88507800	0.32620300	0.80813300
C	3.22119500	0.10125200	1.13646700
C	-0.26400500	-0.04148600	-0.01334400

C	-0.21624600	1.44092200	0.36070000
C	1.06334300	1.49232000	1.23287200
C	-1.52348500	-0.54349900	0.14460700
C	-2.37123900	0.52851100	0.71445300
C	-1.59143500	1.68408000	0.94325400
O	1.36239100	2.33548700	2.05648000
C	-3.72266200	0.51387700	1.06697900
C	-4.27482700	1.65198700	1.66238200
C	-3.49590000	2.78921100	1.89335300
C	-2.14596200	2.81732700	1.51849600
C	0.02283100	2.38109200	-0.84742600
C	-2.02283900	-1.92541200	-0.04325000
C	-2.01977500	-2.53251000	-1.30888700
C	-2.53550400	-3.81558700	-1.48525600
C	-3.06268400	-4.51786000	-0.39942500
C	-3.07547000	-3.92457400	0.86361300
C	-2.56837700	-2.63697400	1.03853700
C	-0.31702900	1.99092100	-2.14870500
C	-0.15484000	2.87069300	-3.21881400
C	0.35051400	4.15394600	-3.00645200
C	0.69149700	4.54988200	-1.71326100
C	0.53058500	3.67109100	-0.64120000
O	0.96935500	-2.54057700	-1.23402200
C	1.53260800	-3.75833700	-1.70027400
O	5.10087500	-1.43545100	0.85848000
C	5.91706700	-0.58108700	1.64951200
H	3.58316700	-2.83689500	-0.52150700
H	3.74636400	0.81687400	1.75582200
H	-4.33118800	-0.36711400	0.88858400
H	-5.32312600	1.65219900	1.94721100
H	-3.94075700	3.66072000	2.36455200
H	-1.54449200	3.70491600	1.68596800
H	-1.61012900	-1.98888500	-2.15315800
H	-2.53446400	-4.26479200	-2.47470700
H	-3.46566700	-5.51697600	-0.53806400
H	-3.48259500	-4.46265400	1.71491400
H	-2.58221800	-2.17879000	2.02294600
H	-0.71397900	0.99674700	-2.32290000
H	-0.42442300	2.54987900	-4.22118500
H	0.47851300	4.83736300	-3.84085100
H	1.08835800	5.54516700	-1.53411000
H	0.81040000	3.98353600	0.35946100
H	1.87312200	-4.38688800	-0.86866400
H	2.37091500	-3.57768100	-2.38456300

H	0.73021700	-4.26969000	-2.23258400
H	6.89553700	-1.06050300	1.69601700
H	5.51775200	-0.47099900	2.66508300
H	6.01975300	0.41134900	1.19402200

Structure 2h

E(RPBE1PBE) = -229.85041052554658 a.u.

0 1			
C	2.74256000	-2.37097200	-1.33185000
C	3.09931700	-1.37261100	-0.41135800
C	2.15690200	-0.49006400	0.12167700
C	0.82197100	-0.61851000	-0.27404400
C	0.47190300	-1.62621700	-1.20405100
C	1.41692400	-2.49623300	-1.73957900
C	-0.37890700	0.16759200	0.00566300
C	-1.60126100	-0.62886200	-0.43637500
C	-0.98331700	-1.55526600	-1.51956300
C	-0.65079400	1.50385000	0.06813200
C	-2.04351400	1.70484300	-0.38791400
C	-2.59193200	0.46352100	-0.78610000
O	-1.56637300	-2.13503800	-2.41337900
C	-2.80136400	2.87350600	-0.49469900
C	-4.09121700	2.79229500	-1.02671100
C	-4.62220600	1.56438100	-1.43172800
C	-3.87954500	0.38447500	-1.29515700
C	-2.18539000	-1.55224000	0.66242900
C	0.29609200	2.58911100	0.38325100
C	1.10432200	2.52033400	1.53124000
C	2.01275900	3.53558900	1.82874900
C	2.12908800	4.64047800	0.98433000
C	1.32833700	4.72502400	-0.15662300
C	0.41659700	3.71383700	-0.45291900
C	-1.96926700	-1.28652700	2.02025200
C	-2.54893500	-2.09046700	3.00163200
C	-3.35300600	-3.17251000	2.64168800
C	-3.57389300	-3.44268900	1.29123800
C	-2.99416300	-2.64043300	0.30756300
H	3.50396800	-3.04124600	-1.71491600
H	2.46457200	0.27009500	0.82791300
H	1.11338200	-3.25011900	-2.45893100
H	-2.39792900	3.82775900	-0.17142400
H	-4.68899600	3.69396100	-1.12250700

H	-5.62320000	1.52281300	-1.85066600
H	-4.30046000	-0.57048900	-1.59204500
H	0.99397000	1.67544800	2.20463500
H	2.62427500	3.46592400	2.72350100
H	2.83604700	5.43172800	1.21491400
H	1.41714800	5.57953400	-0.82105100
H	-0.19028500	3.77924800	-1.35024400

Structure 2k

E(RPBE1PBE) = -229.85041052554658 a.u.

0 1

C	0.76773700	4.45132400	1.29664500
C	1.55550600	4.12686700	0.18264100
C	1.60411600	2.82768100	-0.32736400
C	0.83871400	1.83305200	0.29077100
C	0.04542500	2.17209600	1.41434600
C	0.00811500	3.46612400	1.92678600
C	0.72739600	0.38775200	0.09084500
C	-0.50134800	-0.11849500	0.83823200
C	-0.65065800	0.96716700	1.94021400
C	1.62014800	-0.61940000	-0.13753700
C	1.09319900	-1.84684500	0.49959600
C	-0.11673300	-1.54637200	1.16716400
O	-1.25022000	0.86482500	2.99309100
C	1.61491900	-3.14212900	0.54410300
C	0.93487000	-4.11699300	1.27973500
C	-0.25386300	-3.81015600	1.94802800
C	-0.79901300	-2.52167400	1.87905100
C	-1.80112600	-0.08763300	-0.00184800
C	2.94765600	-0.50471600	-0.76936900
C	4.09479900	-1.04240100	-0.15803400
C	5.34634900	-0.91591300	-0.75766800
C	5.47824400	-0.26020200	-1.98358500
C	4.34768500	0.27026700	-2.60641100
C	3.09520300	0.14760100	-2.00574100
C	-1.76394900	-0.18130100	-1.40198800
C	-2.93215600	-0.21482300	-2.15047000
C	-4.18088300	-0.15709800	-1.51497800
C	-4.23622900	-0.06724000	-0.12100500
C	-3.05132900	-0.03289600	0.61985800
O	-5.26909300	-0.19242900	-2.33908600

C	-6.55800700	-0.13977400	-1.74858400
H	0.75480800	5.47156700	1.66764900
H	2.14382200	4.90479000	-0.29587500
H	2.22674400	2.59954200	-1.18461200
H	-0.60018900	3.68342300	2.79942900
H	2.53116400	-3.39033300	0.01783200
H	1.33363000	-5.12616000	1.32840800
H	-0.76317900	-4.57912800	2.52137300
H	-1.73170100	-2.28775200	2.38208900
H	4.00316600	-1.53925300	0.80238700
H	6.22214700	-1.32714200	-0.26407100
H	6.45400000	-0.16737700	-2.45108200
H	4.43835400	0.77237400	-3.56530500
H	2.21211800	0.53472200	-2.50533800
H	-0.80734100	-0.23293600	-1.91104800
H	-2.90365200	-0.28556100	-3.23289600
H	-5.18504400	-0.01869300	0.39987600
H	-3.11069000	0.04694700	1.70022800
H	-7.26984700	-0.18183100	-2.57425600
H	-6.73236800	-0.99152800	-1.07872200
H	-6.71015900	0.79194400	-1.18871000

Structure 2l

E(RPBE1PBE) = -229.85041052554658 a.u.

0 1			
C	0.82721200	4.46501900	1.26563100
C	1.58281800	4.11909500	0.13576700
C	1.60105600	2.81485900	-0.36310900
C	0.83748100	1.83678200	0.28291900
C	0.07728200	2.19727200	1.42291000
C	0.06999700	3.49671000	1.92371700
C	0.70288500	0.39115700	0.09878300
C	-0.51156100	-0.09226600	0.88322200
C	-0.62447000	1.00967600	1.97585500
C	1.57838400	-0.62915800	-0.13860700
C	1.05357200	-1.84273400	0.52599500
C	-0.13504000	-1.52108600	1.22150400
O	-1.20846400	0.92530600	3.03867000
C	1.56237700	-3.14290000	0.57370000
C	0.89142100	-4.10084500	1.33937200
C	-0.27540700	-3.77262100	2.03519700
C	-0.80858000	-2.47909900	1.96437900

C	-1.83170400	-0.06931600	0.07661400
C	2.89239900	-0.53627900	-0.80136400
C	4.04708900	-1.07833700	-0.20840900
C	5.28642400	-0.97087900	-0.83639200
C	5.39771000	-0.33090300	-2.07262300
C	4.25913700	0.20328900	-2.67738000
C	3.01898400	0.10023400	-2.04821000
C	-1.82756300	-0.16214400	-1.32040600
C	-3.01864900	-0.20125200	-2.04333000
C	-4.23152800	-0.14715000	-1.35967500
C	-4.26469200	-0.05639400	0.02957100
C	-3.06489300	-0.01764400	0.73968600
Cl	-5.74055800	-0.19121600	-2.26445400
H	0.83716400	5.48875700	1.62669100
H	2.16964400	4.88429400	-0.36445600
H	2.19913500	2.57032800	-1.23315700
H	-0.51397600	3.73086300	2.80847400
H	2.46200400	-3.40744200	0.02725800
H	1.28038600	-5.11361000	1.39043400
H	-0.77737300	-4.52836000	2.63190400
H	-1.72412300	-2.22956200	2.49078900
H	3.97182400	-1.56278200	0.75972200
H	6.16881000	-1.38465800	-0.35699700
H	6.36389100	-0.25307900	-2.56211000
H	4.33385900	0.69312400	-3.64390100
H	2.12924500	0.49072500	-2.53319500
H	-0.88405400	-0.20915400	-1.85314400
H	-3.00585600	-0.27238700	-3.12523000
H	-5.21465800	-0.01225600	0.55043000
H	-3.09156800	0.06490500	1.82096200

Structure 2q

E(RPBE1PBE) = -229.85041052554658 a.u.

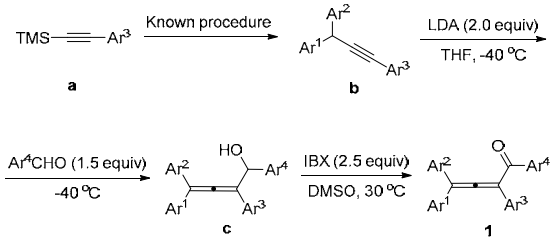
0 1			
C	0.62575500	-4.48274200	-2.05956800
C	1.68822100	-4.03819700	-1.25905800
C	1.61409900	-2.84508700	-0.53746400
C	0.44379000	-2.08081300	-0.61459900
C	-0.62317300	-2.54028600	-1.42674600
C	-0.54143700	-3.72561500	-2.15271800
C	0.06985200	-0.77005500	-0.08581800

C	-1.43807400	-0.59400700	-0.23510300
C	-1.74590000	-1.56692100	-1.40739200
C	0.70303100	0.43737600	0.01318600
C	-0.31426300	1.49769700	-0.13522700
C	-1.57831100	0.90806300	-0.38780000
O	-2.72472800	-1.55674500	-2.12899100
C	-0.20327900	2.88603800	-0.09543000
C	-1.33081300	3.67953900	-0.33649200
C	-2.57041800	3.08131900	-0.60259200
C	-2.70098000	1.67919900	-0.60869000
C	-2.25429800	-1.07901200	0.98900800
C	2.15274100	0.67719200	0.10877900
C	2.80166200	1.61445600	-0.72130100
C	4.16864100	1.82817100	-0.63356100
C	4.93837400	1.11747200	0.30076200
C	4.31527100	0.18901300	1.14216800
C	2.93847500	-0.01908100	1.03797000
C	-1.67863300	-1.12646300	2.26486100
C	-2.43591600	-1.50601700	3.37287700
C	-3.78111000	-1.84558800	3.22387300
C	-4.36203400	-1.79991300	1.95665800
C	-3.60608200	-1.42040100	0.84659900
O	6.27077600	1.40598200	0.31130100
C	7.10190500	0.71504500	1.23171300
O	-3.71899400	3.77127800	-0.86191400
C	-3.66647800	5.18854700	-0.88165400
H	0.71757400	-5.41554400	-2.60739500
H	2.59293500	-4.63680500	-1.19824200
H	2.45210200	-2.52030400	0.06818400
H	-1.37556900	-4.03460200	-2.77531700
H	0.74835700	3.36128600	0.12078300
H	-1.23270400	4.75796800	-0.31052000
H	-3.67518000	1.24183900	-0.79553600
H	2.22473200	2.16067100	-1.46044600
H	4.66980800	2.53885300	-1.28238300
H	4.88181000	-0.36333800	1.88213900
H	2.45790100	-0.72008000	1.71393800
H	-0.63465600	-0.85997800	2.39062100
H	-1.97118700	-1.53655700	4.35446500
H	-4.36977600	-2.14390200	4.08652500
H	-5.40767900	-2.06389100	1.82604800
H	-4.06408300	-1.39948400	-0.13665100
H	8.11237300	1.09016800	1.06448700
H	6.81023400	0.91554400	2.27050700

H	7.08560000	-0.36825200	1.05728100
H	-4.67722100	5.52354000	-1.11886900
H	-3.37105700	5.59751500	0.09314500
H	-2.97444600	5.55764800	-1.64939300

6. Preparation of allenyl ketones 1

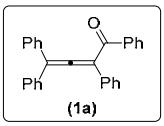
Synthesis of starting materials *1a–1n*, *1p–1y* by revised method, and ^{1}H and ^{13}C NMR spectra data for the reported ones showed good agreement with the literature data[4] (H. Ren, M. Miao, H. Xu, Y. Luo, M. Jin, Z. Chen, J. Xu, *Synthesis* **2017,** *50*, 349). Belows are new general procedure and the summarized characterization data for the newly synthesized allenyl ketones **1**.



To a solution of 1,3,3-triarylpropyne **b**, which was synthesized according to the reported procedure[1] (5.0 mmol, 1.0 equiv) in 20 mL of anhydrous THF, was added dropwise to a solution of LDA (2 M in hexane, 10.0 mmol, 2.0 equiv) at -40 ^{o}C under the N_2 atmosphere. The reaction was stirred for 20 min. Then aldehyde (7.5 mmol, 1.5 equiv) dissolving in 5 mL of dry THF was injected to the mixture by a syringe at -40 ^{o}C. After 15 min, the mixture was allowed to warm up to room temperature, quenched by adding 50 mL of H_2O and extracted with EA (3×50 mL). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo.* The residue was purified with flash silica gel chromatography to afford allenyl alcohol **c**. To a solution of the mixture of **c** in 20 mL of DMSO were added 2.5 equiv of IBX with stirring at 30 ^{o}C for 30 min. After completion of the reaction, the mixture was quenched by adding 20 mL of H_2O and extracted with EtOAc (3×50 mL). The combined organic phase was washed with H_2O (3×50 mL), dried over anhydrous Na_2SO_4,

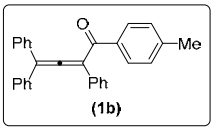
concentrated *in vacuo* and purified by flash silica gel chromatography to give the starting materials **1.**

1,2,4,4-tetraphenylbuta-2,3-dien-1-one (1a)



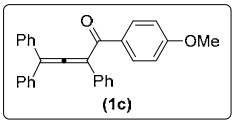
The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.795 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1a** (1.562 g, 84%) as a yellow solid. (Known compound)

2,4,4-triphenyl-1-(p-tolyl)buta-2,3-dien-1-one (1b)



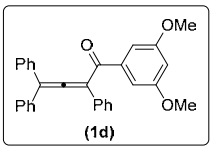
The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 4-methylbenzaldehyde (0.900 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1b** (1.585 g, 82%) as a yellow solid. (Known compound)

1-(4-methoxyphenyl)-2,4,4-triphenylbuta-2,3-dien-1-one (1c)



The reaction of prop-2-yne-1,1,3-triyltribenzene (1.340 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 4-methoxybenzaldehyde (1.022 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1c** (1.610 g, 80%) as a yellow solid. (Known compound)

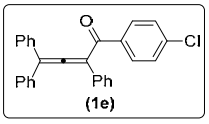
1-(3,5-dimethoxyphenyl)-2,4,4-triphenylbuta-2,3-dien-1-one (1d)



The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 3,5-dimethoxybenzaldehyde (1.247

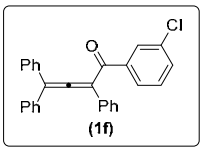
g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1d** (1.758 g, 81%) as a yellow solid. (Known compound)

1-(4-chlorophenyl)-2,4,4-triphenylbuta-2,3-dien-1-one (1e)



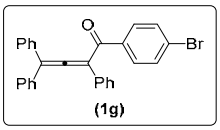
The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 4-chlorobenzaldehyde (1.054 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1e** (1.669 g, 82%) as a yellow solid. (Known compound)

1-(3-chlorophenyl)-2,4,4-triphenylbuta-2,3-dien-1-one (1f)



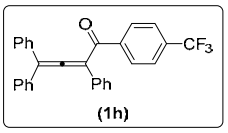
The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 3-chlorobenzaldehyde (1.054 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1f** (1.664 g, 82%) as a yellow solid. (Known compound)

1-(4-bromophenyl)-2,4,4-triphenylbuta-2,3-dien-1-one (1g)



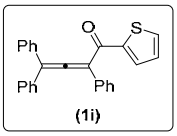
The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 4-bromobenzaldehyde (1.388 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1g** (1.760 g, 78%) as a yellow solid. (Known compound)

2,4,4-triphenyl-1-(4-(trifluoromethyl)phenyl)buta-2,3-dien-1-one (1h)



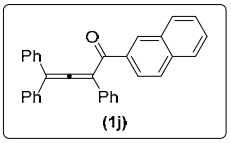
The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 4-(trifluoromethyl)benzaldehyde (1.306 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1h** (1.732 g, 79%) as a yellow solid. (Known compound)

2,4,4-triphenyl-1-(thiophen-2-yl)buta-2,3-dien-1-one (1i)



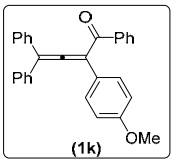
The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 2-thiophenaldehyde (0.841 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1i** (1.325 g, 70%) as a yellow solid. (Known compound)

2,4,4-triphenyl-1-(thiophen-2-yl)buta-2,3-dien-1-one (1j)



The reaction of prop-2-yne-1,1,3-triyltribenzene (1.342 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 2-naphthaldehyde (1.172 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1j** (1.622 g, 77%) as a yellow solid. (Known compound)

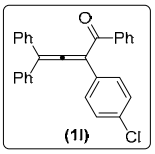
1-(4-methoxyphenyl)-2,4,4-triphenylbuta-2,3-dien-1-one (1k)



The reaction of (3-(4-methoxyphenyl)prop-2-yne-1,1-diyl)dibenzene (1.490g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.795g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1k** (1.208 g, 60%) as a yellow solid; m.p. 66-68 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.25 (Petroleum ether/EtOAc = 10/1);^{1}H NMR (400

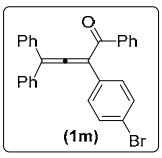
MHz, $CDCl_3$): δ 7.79-7.77 (m, 2H), 7.49-7.45 (m,3H), 7.35-7.32 (m, 6H), 7.24-7.21 (m, 6H), 6.93-6.90 (m, 2H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.7, 193.5, 159.5, 138.2, 135.0, 132.8, 129.31, 129.27, 128.7, 128.5, 128.14, 128.09, 125.0, 115.5, 114.2, 110.9, 55.3; HRMS (ES^+-TOF) calcd for $C_{29}H_{23}O_2$ ($[M+H]^+$): 403.1693, found 403.1687.

2-(4-chlorophenyl)-1,4,4-triphenylbuta-2,3-dien-1-one (1l)



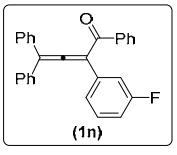
The reaction of (3-(4-chlorophenyl)prop-2-yne-1,1-diyl)dibenzene (1.514g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.795g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1l** (1.607 g, 79%) as a yellow solid; m.p. 72-74 ^oC (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1);^1H NMR (400 MHz, $CDCl_3$): δ 7.77-7.75 (m, 2H), 7.50-7.44 (m, 3H), 7.37-7.34 (m, 8H), 7.24-7.21 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 213.1, 192.9, 137.9, 134.5, 133.9, 133.0, 131.5, 129.4, 129.3, 128.9, 128.8, 128.5, 128.4, 128.2, 116.0, 110.3; HRMS (ES^+-TOF) calcd for $C_{28}H_{20}ClO$ ($[M+H]^+$): 407.1197, found 407.1202.

2-(4-bromophenyl)-1,4,4-triphenylbuta-2,3-dien-1-one (1m)



The reaction of (3-(4-bromophenyl)prop-2-yne-1,1-diyl)dibenzene (1.737 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.796 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1m** (1.625 g, 72%) as a yellow solid. (Known compound)

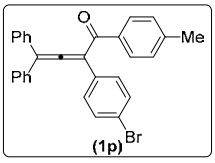
2-(3-fluorophenyl)-1,4,4-triphenylbuta-2,3-dien-1-one (1n)



The reaction of (3-(3-fluorophenyl)prop-2-yne-1,1-diyl)dibenzene (1.431 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.796

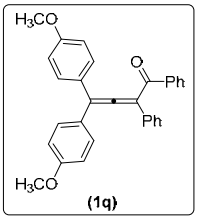
g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1n** (0.918 g, 47%) as a yellow solid. (Known compound)

2-(4-bromophenyl)-4,4-diphenyl-1-(p-tolyl)buta-2,3-dien-1-one (1p)



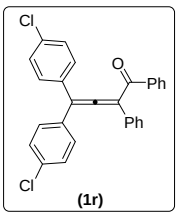
The reaction of (3-(4-bromophenyl)prop-2-yne-1,1-diyl)dibenzene (1.736g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 4-methylbenzaldehyde (0.901g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1p** (1.652 g, 71%) as a yellow solid; m.p. 107-109 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1);^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71-7.69 (m, 2H), 7.50-7.47 (m, 2H), 7.41-7.34 (m, 7H), 7.27-7.24 (m, 5H), 7.03 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.2, 192.3, 144.1, 135.1, 134.6, 132.2, 131.8, 129.6, 128.9, 128.7, 128.5, 128.4, 122.0, 115.9, 110.2, 21.6; HRMS (ES^+-TOF) calcd for $C_{29}H_{22}BrO$ ($[M+H]^+$): 465.0849, found 465.0830.

4,4-bis(4-methoxyphenyl)-1,2-diphenylbuta-2,3-dien-1-one (1q)



The reaction of 4,4'-(3-phenylprop-2-yne-1,1-diyl)bis(methoxybenzene) (1.642 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.796 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1q** (0.908 g, 42%) a yellow solid. (Known compound)

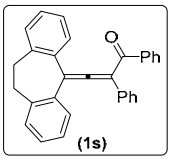
4,4-bis(4-chlorophenyl)-1,2-diphenylbuta-2,3-dien-1-one (1r)



The reaction of 4,4'-(3-phenylprop-2-yne-1,1-diyl)bis(chlorobenzene) (1.886 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.796

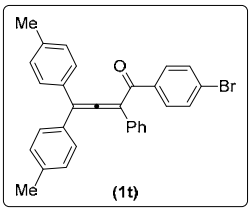
g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1r** (1.368 g, 62%) as a yellow solid. (Known compound)

3-(10,11-dihydro-5H-dibenzo[a,d][7]annulen-5-ylidene)-1,2-diphenylprop-2-en-1-one (1s)



The reaction of 5-(phenylethynyl)-10,11-dihydro-5*H*-dibenzo[*a*,*d*][7]annulene (1.473g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.795g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1s** (1.415 g, 71%) as a yellow solid; m.p. 86-89 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1);^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.6 Hz, 2H), 7.65 (d, *J* = 7.6 Hz, 2H), 7.47-7.34 (m, 6H), 7.23-7.10 (m, 8H), 2.85-2.73 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 213.2, 193.4, 139.1, 138.5, 134.7, 133.1, 132.6, 129.6, 129.4, 129.2, 128.8, 128.4, 128.0, 127.8, 126.5, 117.1, 109.1, 33.1; HRMS (ES^+-TOF) calcd for $C_{30}H_{23}O$ ($[M+H]^+$): 399.1743, found.399.1747.

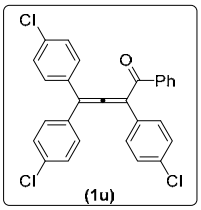
1-(4-bromophenyl)-2-phenyl-4,4-di-p-tolylbuta-2,3-dien-1-one (1t)



The reaction of 4,4'-(3-(4-bromophenyl)prop-2-yne-1,1-diyl)bis(methylbenzene) (1.871 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and 4-bromobenzaldehyde (1.388 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1t** (1.758 g, 71%) as a yellow solid; m.p. 113-115 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1);^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.38-7.35 (m, 4H), 7.32-7.30 (m, 1H), 7.17-7.12 (m, 8H), 2.37 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 213.2, 192.2, 138.3, 136.9, 133.0, 131.7, 131.4, 130.8, 129.5, 128.7, 128.4, 128.1,

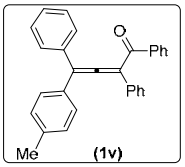
128.0, 127.9, 115.7, 110.8, 21.2; HRMS (ES^+-TOF) calcd for $C_{30}H_{24}BrO$ ($[M+H]^+$): 479.1005, found 479.1015.

2,4,4-tris(4-chlorophenyl)-1-phenylbuta-2,3-dien-1-one (1u)



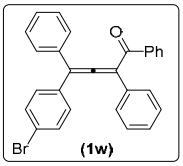
The reaction of 4,4',4''-(prop-2-yne-1,1,3-triyl)tris(chlorobenzene) (1.859 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.796 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.502 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1u** (1.665 g, 70%) as a yellow solid; m.p. 121-122 ^oC (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1);^1H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 7.2 Hz, 2H), 7.50 (t, J = 7.2 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.36-7.33 (m, 6H), 7.29-7.27 (m, 2H), 7.14 (d, *J* = 8.4 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.0, 192.4, 137.7, 134.6, 134.3, 133.3, 132.8, 130.9, 129.6, 129.24, 129.15, 129.1, 128.3, 114.3, 110.9; HRMS (ES^+-TOF) calcd for $C_{28}H_{18}Cl_3O$ ($[M+H]^+$): 475.0418, found 475.0418.

1,2,4-triphenyl-4-(p-tolyl)buta-2,3-dien-1-one (1v)



The reaction of (3-(*p*-tolyl)prop-1-yne-1,3-diyl)dibenzene (1.412 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.796 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1v** (1.353 g, 70%) as a yellow solid. (Known compound)

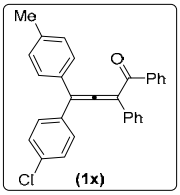
4-(4-bromophenyl)-1,2,4-triphenyl-3l5-buta-2,3-dien-1-one (1w)



The reaction of (3-(4-bromophenyl)prop-1-yne-1,3-diyl)dibenzene (1.736 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.796 g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX

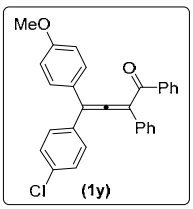
(3.502 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1w** (1.422 g, 63%) as a yellow solid; m.p. 136-137 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.40 (Petroleum ether/EtOAc = 10/1);^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.78 (m, 2H), 7.52-7.46 (m, 5H), 7.38-7.31 (m, 6H) 7.25-7.24 (m, 4H), 7.15-7.12 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.0, 192.9, 137.9, 134.4, 133.9, 133.1, 132.7, 131.9, 130.0, 129.3, 128.8, 128.7, 128.4, 128.2, 127.9, 122.3, 114.9, 111.5, 106.0; HRMS (ES^+-TOF) calcd for $C_{28}H_{20}BrO$ ($[M+H]^+$): 451.0692, found 451.0692.

4-(4-chlorophenyl)-1,2-diphenyl-4-(p-tolyl)buta-2,3-dien-1-one (1x)



The reaction of 1-chloro-4-(3-phenyl-1-(*p*-tolyl)prop-2-yn-1-yl)benzene (1.584g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.795g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1x** (1.368 g, 65%) as a yellow solid; m.p. 118-121^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1);^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.78 (m, 2H), 7.52-7.45 (m, 3H), 7.39-7.35 (m, 2H), 7.32-7.25 (m, 5H), 7.20-7.11 (m, 6H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.1, 193.0, 138.4, 138.0, 134.1, 133.6, 133.0, 132.9, 131.4, 129.7, 129.5, 129.3, 128.9, 128.8, 128.4, 128.3, 128.2, 128.1, 128.0, 114.7, 111.4, 21.2; HRMS (ES^+-TOF) calcd for $C_{29}H_{22}ClO$ ($[M+H]^+$): 421.1354, found 421.1365.

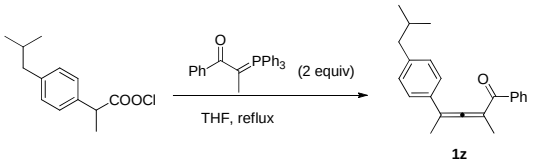
4-(4-chlorophenyl)-4-(4-methoxyphenyl)-1,2-diphenylbuta-2,3-dien-1-one (1y)



The reaction of 1-chloro-4-(1-(4-methoxyphenyl)-3-phenylprop-2-yn-1-yl)benzene (1.664 g, 5.0 mmol, 1.0 equiv), LDA (5 mL, 2.0 M in hexane, 2.0 equiv), and benzaldehyde (0.795g, 7.5 mmol, 1.5 equiv) in 20 mL of THF. The resulted mixture was further oxidized by IBX (3.500 g, 12.5 mmol, 2.5 equiv) in 20 mL of DMSO to afford **1y** (1.529

g, 70%) as a yellow solid; m.p. 89-91 ^{o}C (Petroleum ether/EtOAc); R*f* = 0.45 (Petroleum ether/EtOAc = 10/1);^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.78 (m, 2H), 7.52-7.45 (m, 3H), 7.40-7.36 (m, 2H), 7.32-7.24 (m, 5H), 7.20-7.19 (m, 1H), 7.18-7.15 (m, 3H), 6.90-6.87 (m, 2H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.1, 193.1, 159.8, 138.0, 134.1, 133.7, 133.0, 132.9, 129.7, 129.6, 129.3, 128.9, 128.8, 128.2, 128.1, 127.9, 126.5, 114.5, 114.3, 111.3, 55.3; HRMS (ES^+-TOF) calcd for $C_{29}H_{22}ClO_2$ ($[M+H]^+$): 437.1303, found 437.1305.

4.2 Synthesis of Starting Materials *1z.*



4-(4-isobutylphenyl)-2-methyl-1-phenylpenta-2,3-dien-1-one (1z)

To a solution of 1-phenyl-2-(triphenyl-l5-phosphanylidene)propan-1-one (3.375 g, 8.6 mmol, 2.0 equiv) in dry THF (30 mL) under N_2 was added to hypochlorous 2-(4-isobutylphenyl)propanoic anhydride (0.963 g, 4.3 mmol, 1.0 equiv), then the solution was refluxed for 4.0 h. After completion of the reaction, the mixture was quenched by adding 50 mL of H_2O and extracted with EtOAc (3×50 mL). The combined organic phase was washed with H_2O (3×30 mL), dried over anhydrous Na_2SO_4, concentrated *in vacuo* and purified by flash silica gel chromatography to afford **1z**. (795 mg, 60%) as a red liquid; R*f* = 0.40 (Petroleum ether/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72-7.70 (m, 2H), 7.42-7.39 (m, 1H), 7.28 (d, *J* = 7.6 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 2.45 (d, *J* = 7.2 Hz, 2H), 2.11 (d, *J* = 8.0 Hz, 6H), 1.88-1.81 (m, 1H), 0.90 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 214.6, 195.2, 141.2, 138.6, 132.6, 131.7, 129.3, 128.5, 127.8, 125.7, 104.0, 103.1, 45.0, 30.2, 22.3, 16.5, 15.0; HRMS (ES^+-TOF) calcd for $C_{22}H_{25}O$ ($[M+H]^+$): 305.1900, found 305.1911.

5. X-ray diffraction analysis of 2p, **2y**, **5a**, **9**, and **10**

Crystallographic structure analysis of **2p**, **2y, 5a, 9** and **10**: A suitable single crystal was mounted on a *Xcalibur, Atlas, Gemini ultra* at $T_{(2p)}$ = 150 K, $T_{(2y)}$ = 293 K , $T_{(5a)}$ = 298 K, $T_{(9)}$ = 170 K or $T_{(10)}$ = 298 K using Mo Kα radiation($\lambda_{(2p)}$ = 0.71073 Å, $\lambda_{(2y)}$ = 0.71073 Å, $\lambda_{(5a)}$ = 0.71073 Å, $\lambda_{(9)}$ = 0.71073 Å, $\lambda_{(10)}$ = 0.71073 Å). The intensity data were collected with *CrysAlisPro* program and reduced by *CrysAlisPro* program. The structure was solved by direct methods, expended by difference Fourier syntheses and refined by Full-matrix squares on F^2 using *SHELXL* program packages. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in ideal positions and refined as riding atoms. Details of the X-ray experiments and crystal data are summarized in ***Table S2***, ***Table S3, Table S4, Table S5*** and ***Table S6.***

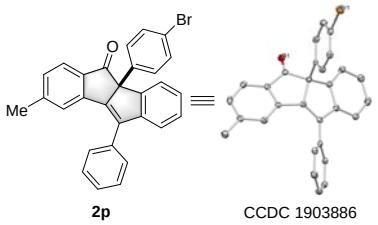


Figure S7. ORTEP drawing of **2p** with ellipsoid contour at 30% probability level

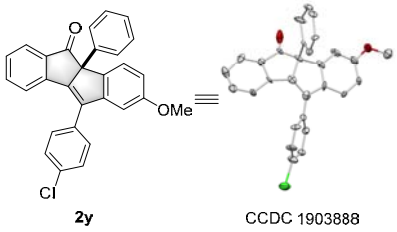


Figure S8. ORTEP drawing of **2y** with ellipsoid contour at 30% probability level

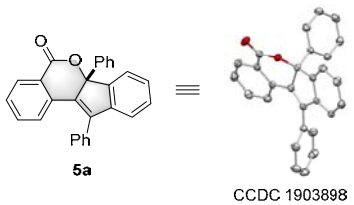


Figure S9. ORTEP drawing of **5a** with ellipsoid contour at 30% probability level

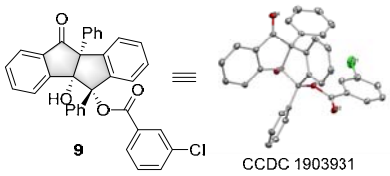


Figure S10. ORTEP drawing of **9** with ellipsoid contour at 30% probability level

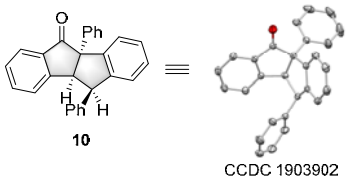


Figure S11. ORTEP drawing of **10** with ellipsoid contour at 30% probability level

Table S2. Crystal data and structure refinement for **2p**.

Empirical formula	$C_{29}H_{19}BrO$	
Formula weight	463.35	
Temperature	150 K	
Wavelength	0.71073 Å	
Crystal system, Space group	trigonal, R -3	
Unit cell dimensions	a= 28.0155(18) Å	α= 90°
	b= 28.0155(18) Å	β= 90°
	c= 14.9771(11) Å	γ= 120°
Volume	10180.1(15) $Å^3$	
Z	18	
Density(calculated)	1.360 Mg/m^3	
Absorption coefficient	1.835 mm^{-1}	
F(000)	4248	
Crystal size	0.42*0.36*0.29 mm^3	
Theta range for data collection	3.197 to 25.349°.	
Index ranges	-18<=h<=33, -33<=k<=28, -17<=l<=14	
Reflections collected	6978 [R(int)= 0.0382]	
Independent reflections	4126	
Completeness to theta= 25.350°	99.6%	

Absorption correction	Multi-scan from equivalents
Refinement method	Full-matrix squares on F^2
Data/restraints/parameters	4126 /0/ 281
Goodness-of-fit on F^2	1.026
Final R indices [I>2sigma(I)]	R_1= 0.0438, wR_2= 0.0924
R indices(all data)	R_1= 0.0656, wR_2= 0.1045
Extinction coefficient	?
Largest diff. peak and hole	0.606 and -0.547 $Å^3$

Table S3. Crystal data and structure refinement for **2y**.

Empirical formula	$C_{29}H_{19}ClO_2$	
Formula weight	434.11	
Temperature	293K	
Wavelength	0.71073 Å	
Crystal system, Space group	monoclinic, P 1 21/m 1	
Unit cell dimensions	a= 11.7014(12) Å	α= 90°
	b= 8.6987(8) Å	β= 94.856(9)°
	c= 21.7780(2) Å	γ= 90°
Volume	2208.7(4) $Å^3$	
Z	2	
Density(calculated)	1.987 Mg/m^3	
Absorption coefficient	1.341 mm^{-1}	
F(000)	1312.0	
Crystal size	?	
Theta range for data collection	5.844 to 45°	
Index ranges	-11<=h<=12, -9<=k<=7, -23<=l<=21	
Reflections collected	6550 [R(int)= 0.0547]	
Independent reflections	3954	

Completeness to theta= 25.350°	99.77%
Absorption correction	Multi-scan from equivalents
Refinement method	Full-matrix squares on F^2
Data/restraints/parameters	3954 /2/579
Goodness-of-fit on F^2	2.246
Final R indices [I>2sigma(I)]	R_1= 0.1747, wR_2= 0.4869
R indices(all data)	R_1= 0.1879, wR_2= 0.5017
Extinction coefficient	?
Largest diff. peak and hole	0.83 and -0.60 $Å^3$

Table S4. Crystal data and structure refinement for **5a**.

Empirical formula	C_{28} H_{18} O_2	
Formula weight	386.45	
Temperature	298 K	
Wavelength	0.71073 Å	
Crystal system, Space group	monoclinic, P 1 21/n 1	
Unit cell dimensions	a= 11.7597(9)Å	α= 90°
	b= 10.1515(7) Å	β= 105.118(7)°
	c= 16.9119(11) Å	γ= 90°
Volume	1949.0(3)$Å^3$	
Z	4	
Density(calculated)	1.3169 Mg/m^3	
Absorption coefficient	0.082 mm^{-1}	
F(000)	808.3831	
Crystal size	?	
Theta range for data collection	3.16 to 29.38°	
Index ranges	-15<=h<=15, -14<=k<=7, -21<=l<=14	
Reflections collected	8659 [R(int)= 0.0291]	

Independent reflections	4522
Completeness to theta= 25.350°	99.76%
Absorption correction	Multi-scan from equivalents
Refinement method	Full-matrix squares on F^2
Data/restraints/parameters	4522 /0/ 270
Goodness-of-fit on F^2	0.9907
Final R indices [I>2sigma(I)]	R_1= 0.0546, wR_2= 0.4869
R indices(all data)	R_1= 0.1155, wR_2= 0.1301
Extinction coefficient	?
Largest diff. peak and hole	0.3932 and -0.4098 $Å^3$

Table S5. Crystal data and structure refinement for **9**.

Empirical formula	$C_{35}H_{23}ClO_4$	
Formula weight	542.13	
Temperature	170 K	
Wavelength	0.71073Å	
Crystal system, Space group	monoclinic, -P 2ybc	
Unit cell dimensions	a= 8.9363(2) Å	α= 90°
	b= 17.2110(4)Å	β= 101.2240(10)°
	c= 20.3755(4)Å	γ= 90°
Volume	3073.87(12) $Å^3$	
Z	4	
Density(calculated)	1.364 Mg/m^3	
Absorption coefficient	0.174mm^{-1}	
F(000)	1320	
Crystal size	?	
Theta range for data collection	2.577 to 25.350°	
Index ranges	-10<=h<=10, -20<=k<=20, -22<=l<=24	
Reflections collected	28853 [R(int)= 0.0368]	

Independent reflections	5636
Completeness to theta= 25.350°	99.6%
Absorption correction	Multi-scan from equivalents
Refinement method	Full-matrix squares on F^2
Data/restraints/parameters	5636 /0/ 418
Goodness-of-fit on F^2	1.015
Final R indices [I>2sigma(I)]	R_1= 0.0546 , wR_2= 0.0925
R indices(all data)	R_1= 0.0451, wR_2= 0.0994
Extinction coefficient	?
Largest diff. peak and hole	0.246 and -0.388$Å^3$

Table S6. Crystal data and structure refinement for **10**.

Empirical formula	C_{28} $H_{20}O$	
Formula weight	372.47	
Temperature	298 K	
Wavelength	0.71073Å	
Crystal system, Space group	triclinic, -P 1	
Unit cell dimensions	a= 10.1558(17) Å	α= 83.002(10)°
	b= 10.2913(8)Å	β= 62.589(17)°
	c= 10.8220(18)Å	γ= 85.427(9)°
Volume	996.3(3)$Å^3$	
Z	2	
Density(calculated)	1.2416 Mg/m^3	
Absorption coefficient	0.074mm^{-1}	
F(000)	392.1694	
Crystal size	?	
Theta range for data collection	2.77 to 29.47°	
Index ranges	-13<=h<=13, -14<=k<=9, -13<=l<=14	
Reflections collected	8182 [R(int)= 0.0279]	

Independent reflections	4621
Completeness to theta= 25.350°	99.80%
Absorption correction	Multi-scan from equivalents
Refinement method	Full-matrix squares on F^2
Data/restraints/parameters	4621 /0/ 261
Goodness-of-fit on F^2	0.9598
Final R indices [I>2sigma(I)]	R_1= 0.0534, wR_2= 0.0925
R indices(all data)	R_1= 0.1029, wR_2= 0.1207
Extinction coefficient	?
Largest diff. peak and hole	0.3016and -0.3609Å^3

Reference

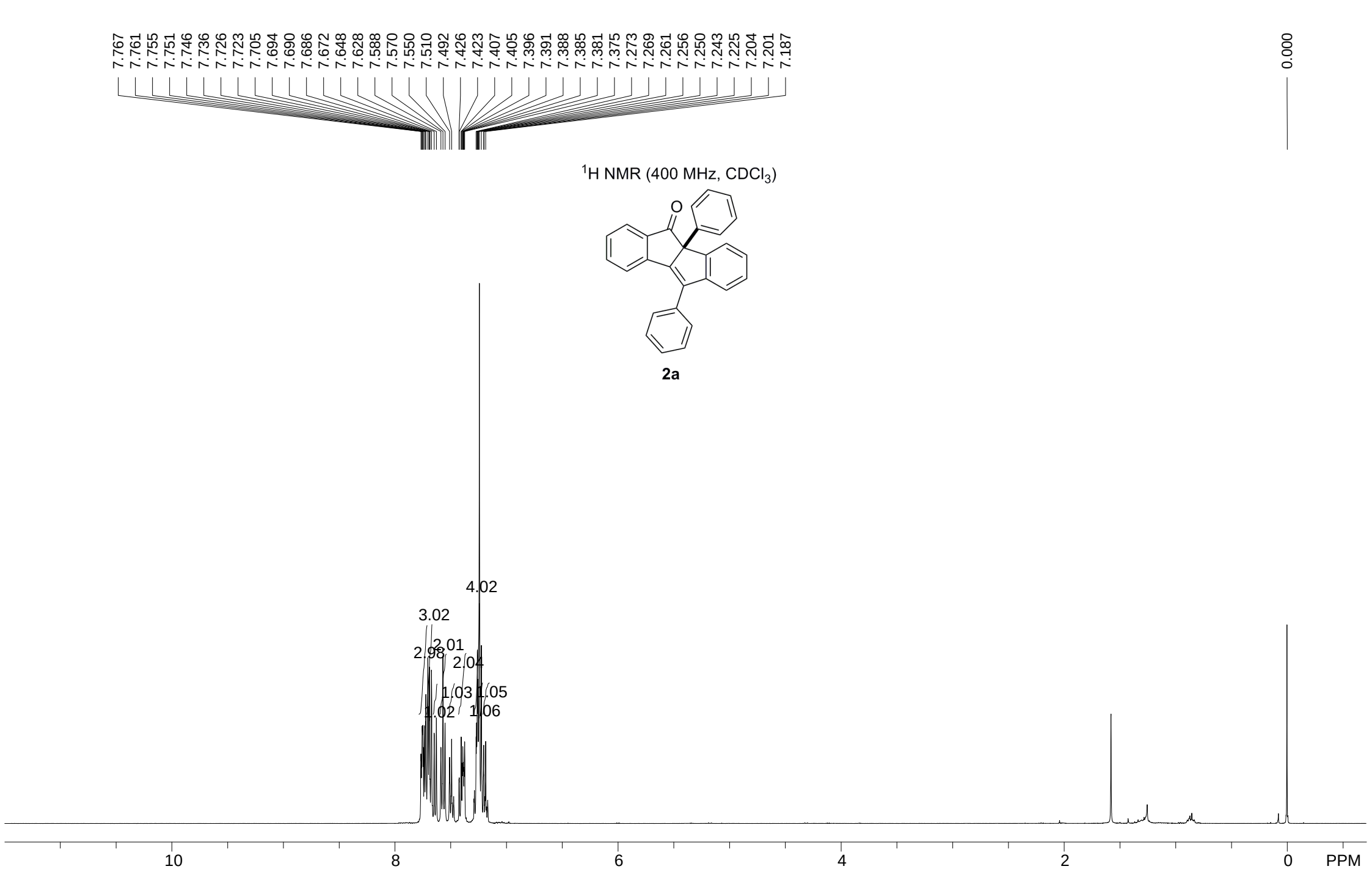
1. M. Yasuda, T. Saito, M. Ueba, A. Baba, *Angew. Chem. Int. Ed.* **2004**, *116*, 1438-1440.

2. T. Johannes, P. Bernd, *Org. Lett.* **2018**, *20*, 2257-2260.

3. J. Shen, D. Yuan, Y. Qiao, X. Shen, Z. Zhang, Y. Zhong, Y. Yi, X. Zhu, *Org. Lett.* **2014**, *16*, 4924-4927.

4. H. Ren, M. Miao, H. Xu, Y. Luo, M. Jin, Z. Chen, J. Xu, *Synthesis* **2017,** *50*, 349-360.

6. Copies of ^{1}H NMR and ^{13}C NMR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7.755
7.751
7.746
7.736
7.726
7.723
7.705
7.694
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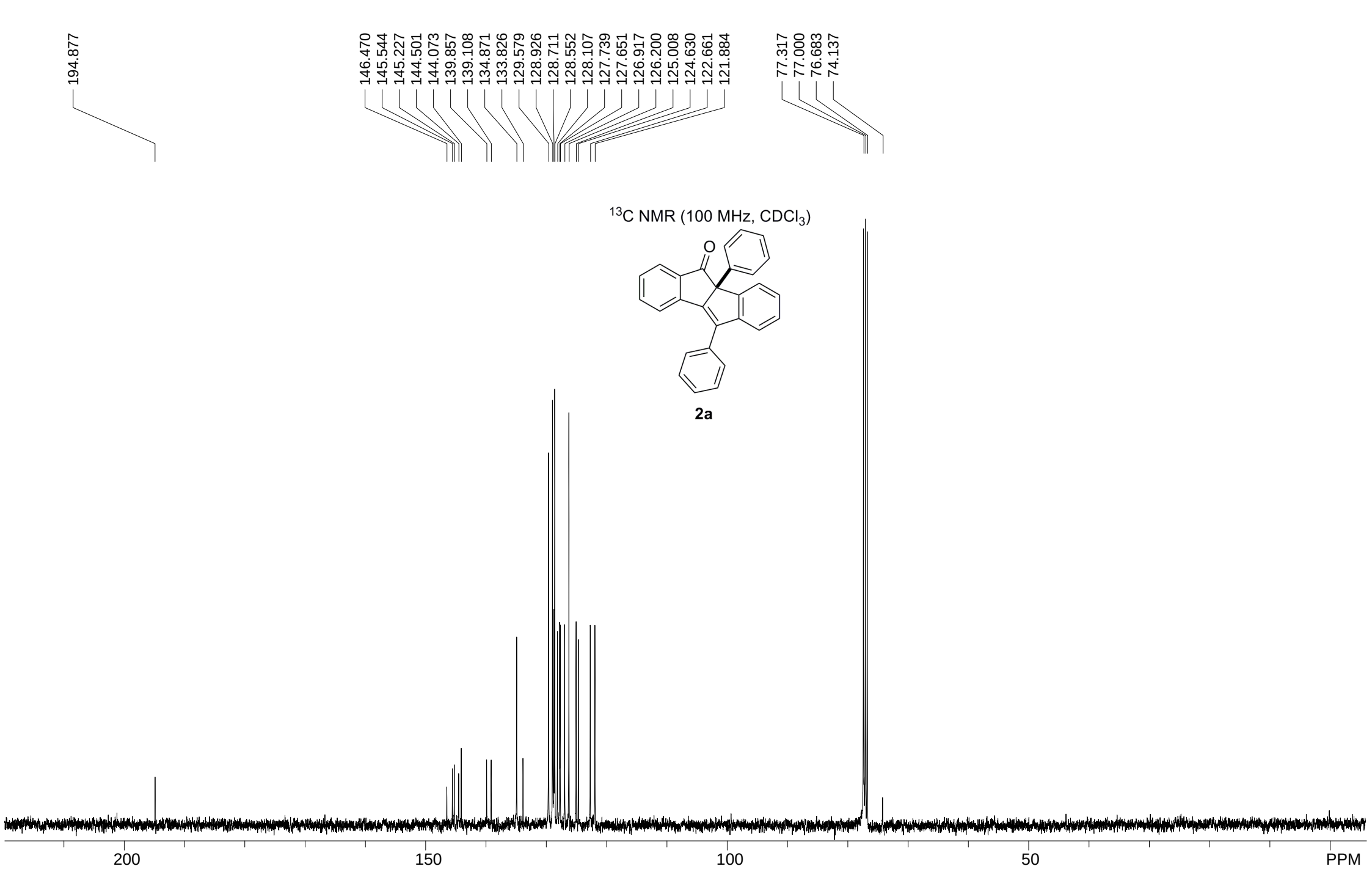
7.672
7.648
7.628
7.588
7.570
7.550
7.510
7.492
7.426
7.423
7.407
7.405
7.396
7.391
7.388
7.385
7.381
7.375
7.273
7.269
7.261
7.256
7.250
7.243
7.225
7.204
7.201
7.187
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
2a
4.02
3.02
2.98
2.01
2.04
1.03
1.05
1.02
1.06
10
8
6
4
2
0
PPM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145.227
144.501
144.073
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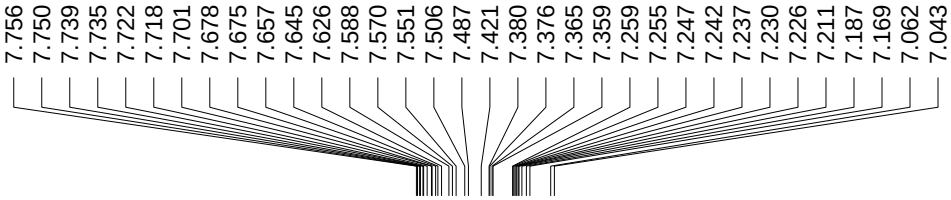
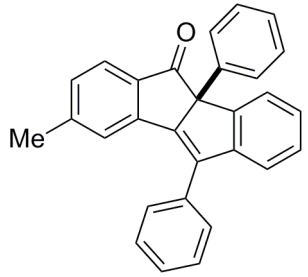
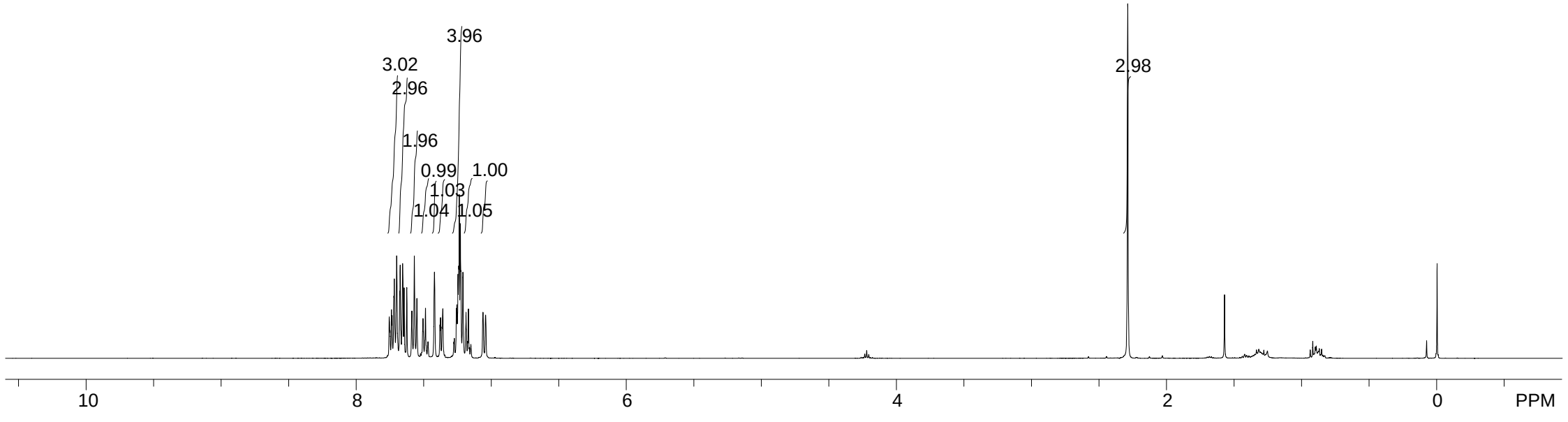
133.826
129.579
128.926
128.711
128.552
128.107
127.739
127.651
126.917
126.200
125.008
124.630
122.661
121.884
77.317
77.000
76.683
74.137
^{13}C NMR (100 MHz, $CDCl_3$)
O
2a
200
150
100
50
PPM



^{1}H NMR (400 MHz, $CDCl_3$)



2b



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145.329
144.380
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140.184
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133.955
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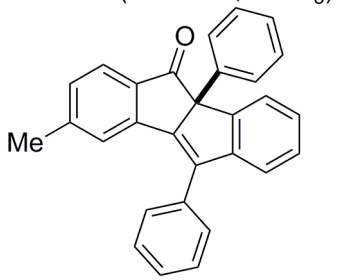
128.663
128.522
127.667
127.547
126.822
126.169
124.840
124.620
123.189
121.806
77.317
77.000
76.683
74.388
22.244

^{13}C NMR (100 MHz, $CDCl_3$)



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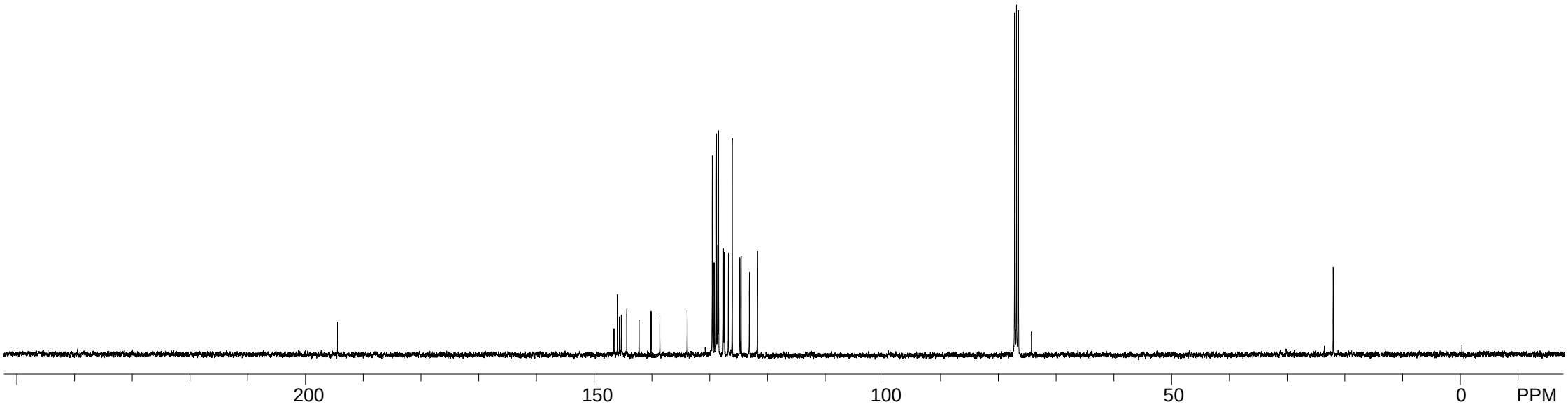

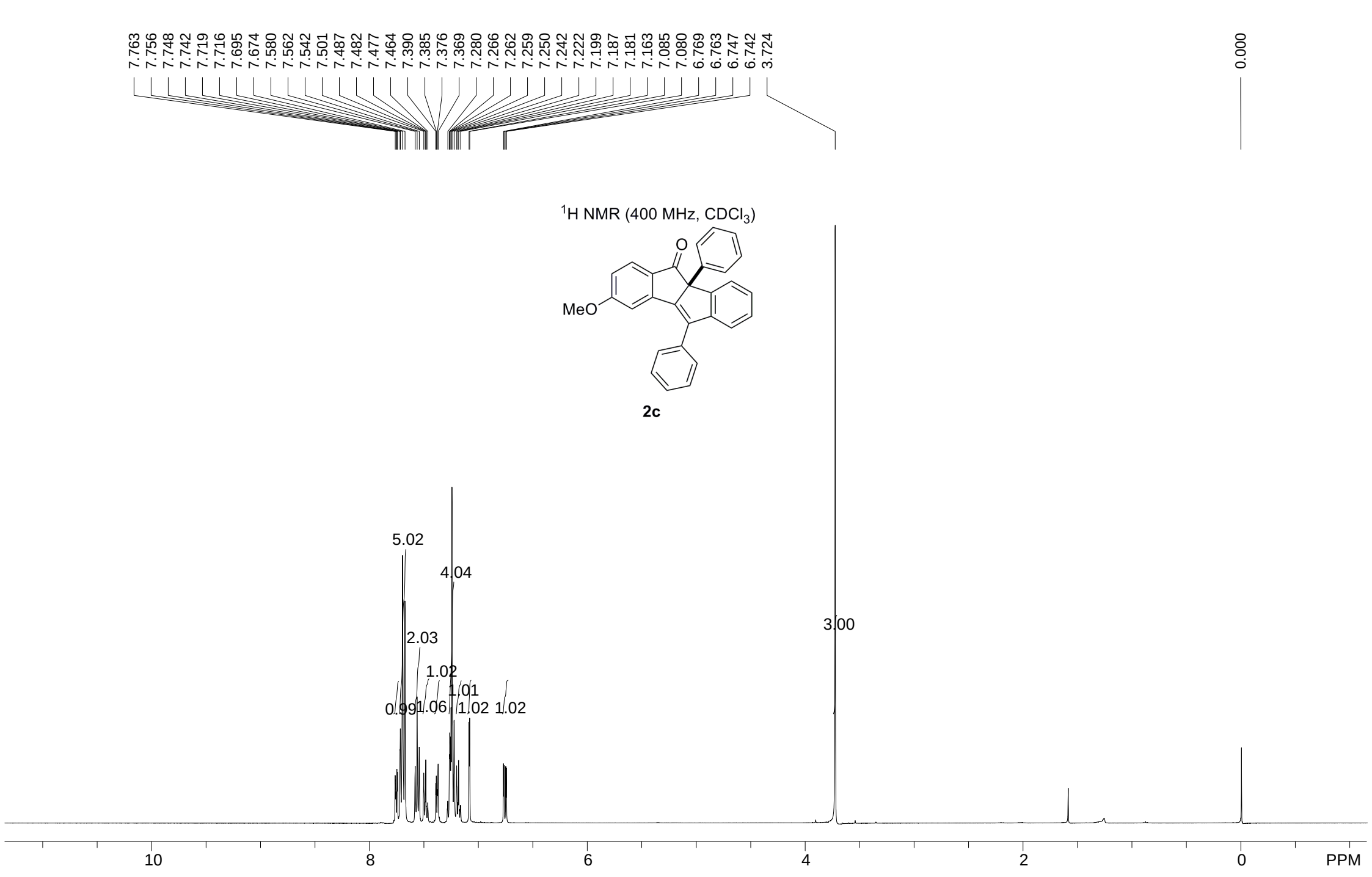
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7.580
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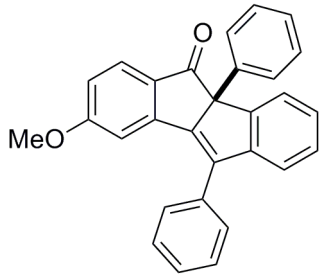
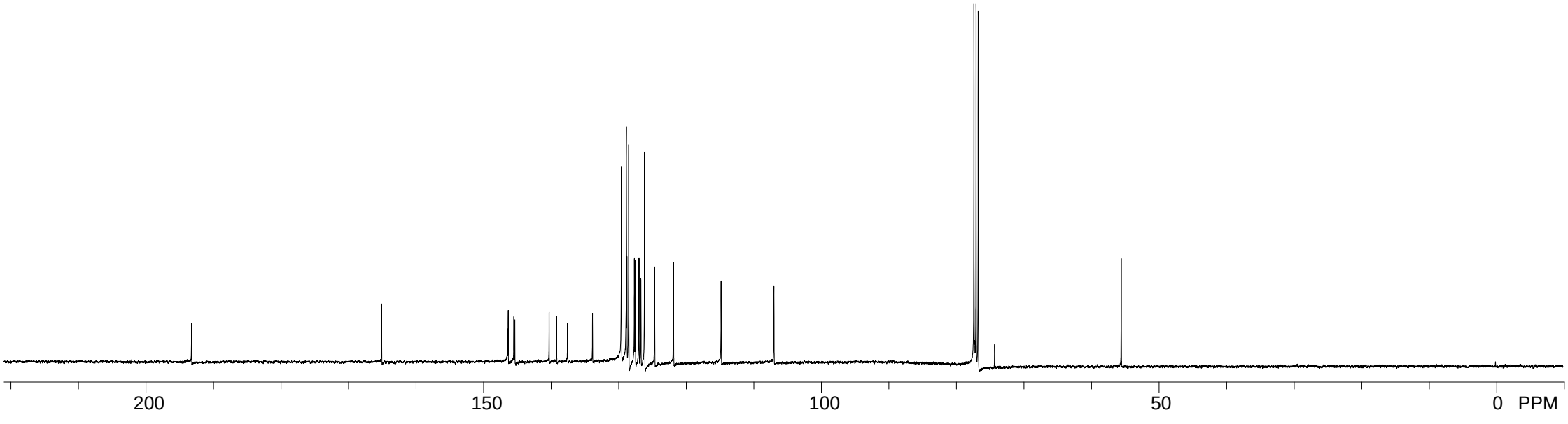
7.542
7.501
7.487
7.482
7.477
7.464
7.390
7.385
7.376
7.369
7.280
7.266
7.262
7.259
7.250
7.242
7.222
7.199
7.187
7.181
7.163
7.085
7.080
6.769
6.763
6.747
6.742
3.724
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
MeO
2c
5.02
4.04
2.03
1.02
1.01
0.99
1.06
1.02
1.02
3.00
10
8
6
4
2
0
PPM

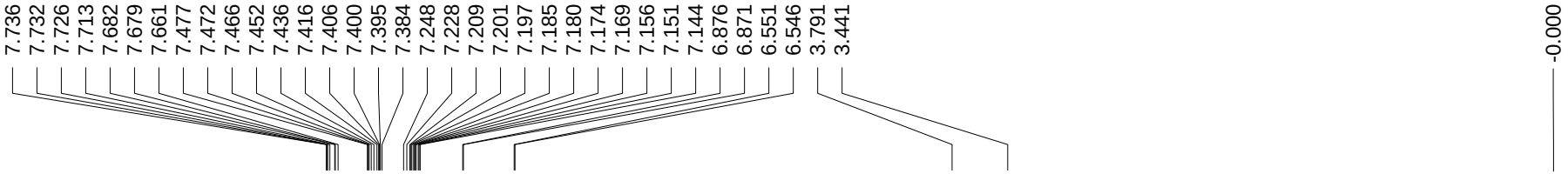
193.246
165.103
146.477
146.355
145.526
145.382
140.300
139.180
137.570
133.861
129.586
128.849
128.761
128.495
127.658
127.522
126.970
126.699
126.150
124.654
121.857
114.803
106.970
77.318
77.000
76.682
74.236
55.477

^{13}C NMR (100 MHz, $CDCl_3$)



2c





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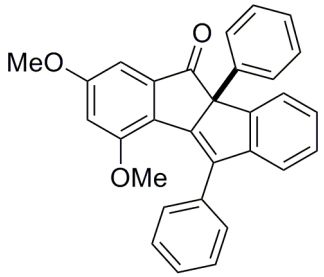
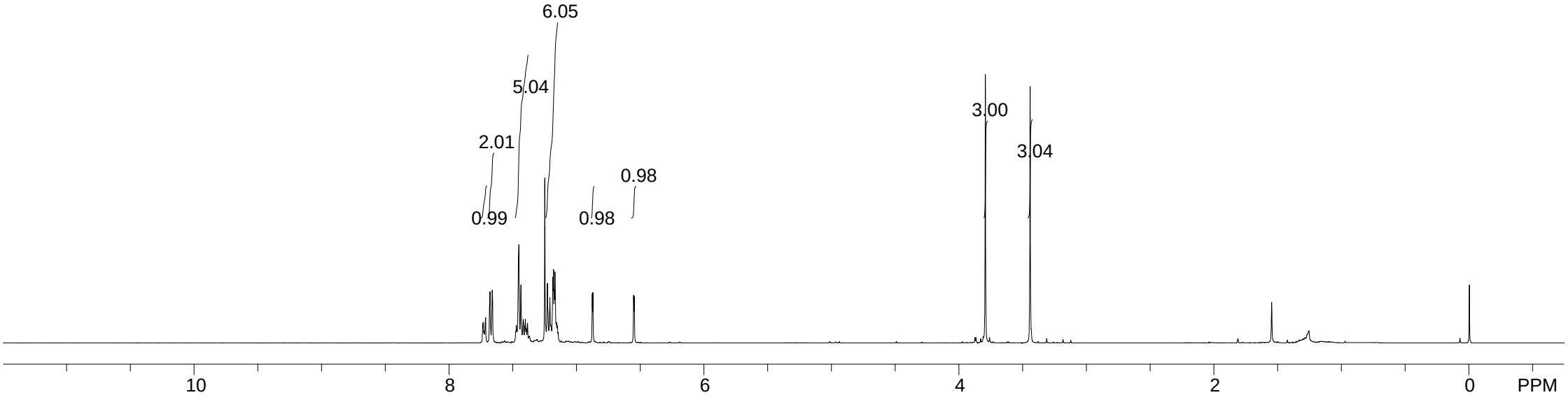
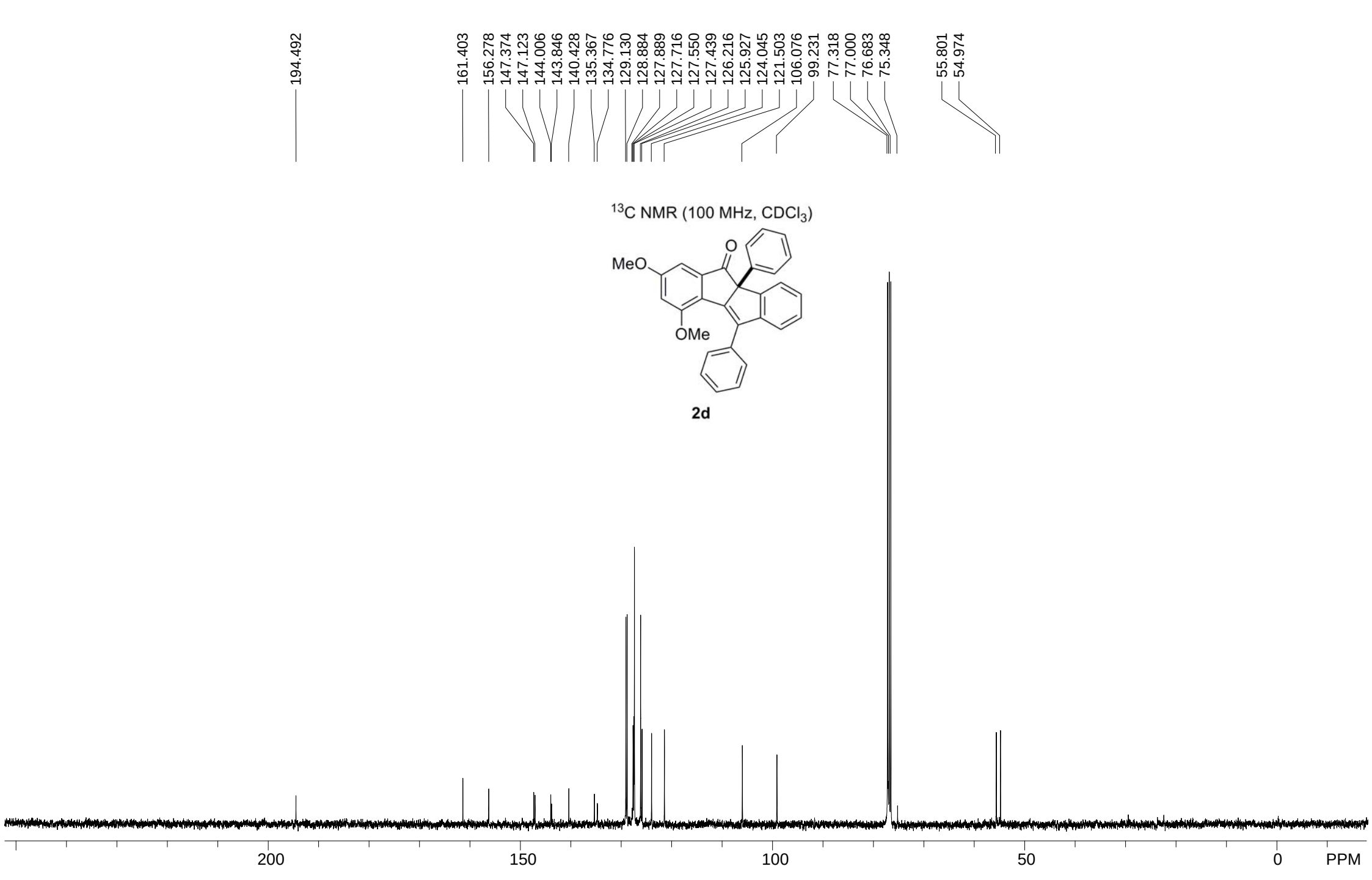


2d

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147.123
144.006
143.846
140.428
135.367
134.776
129.130
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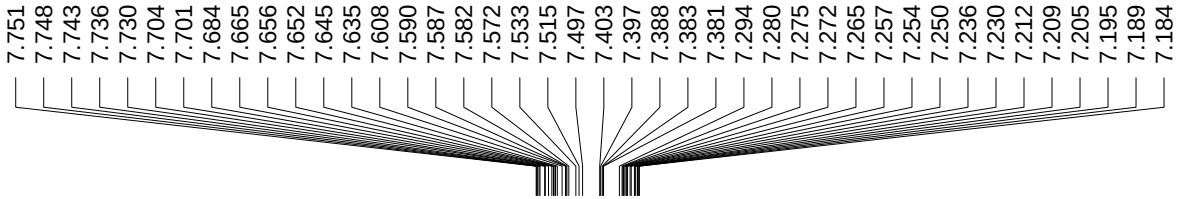
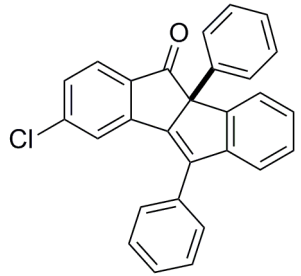
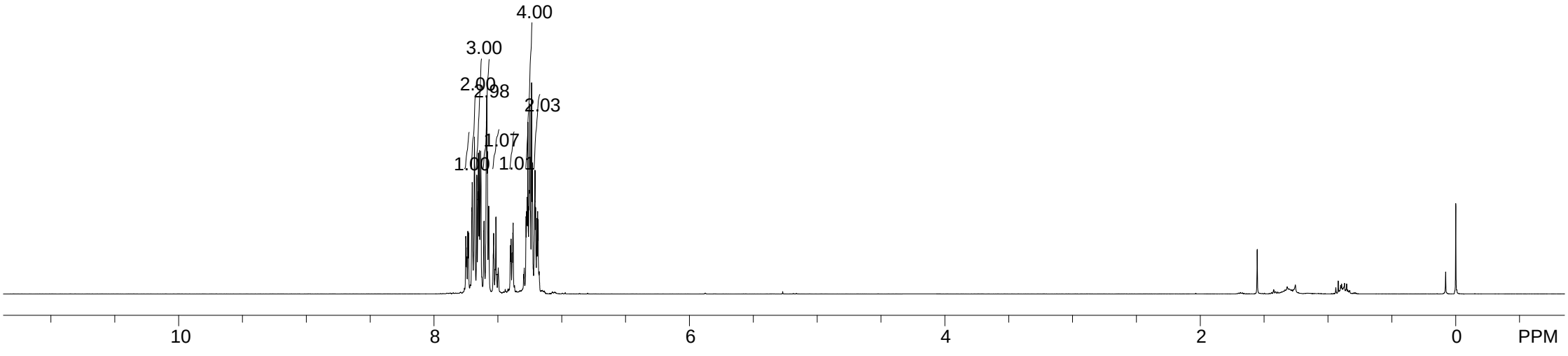
127.716
127.550
127.439
126.216
125.927
124.045
121.503
106.076
99.231
77.318
77.000
76.683
75.348
55.801
54.974
13C NMR (100 MHz, CDCl3)
MeO
O
OMe
2d
200
150
100
50
0
PPM

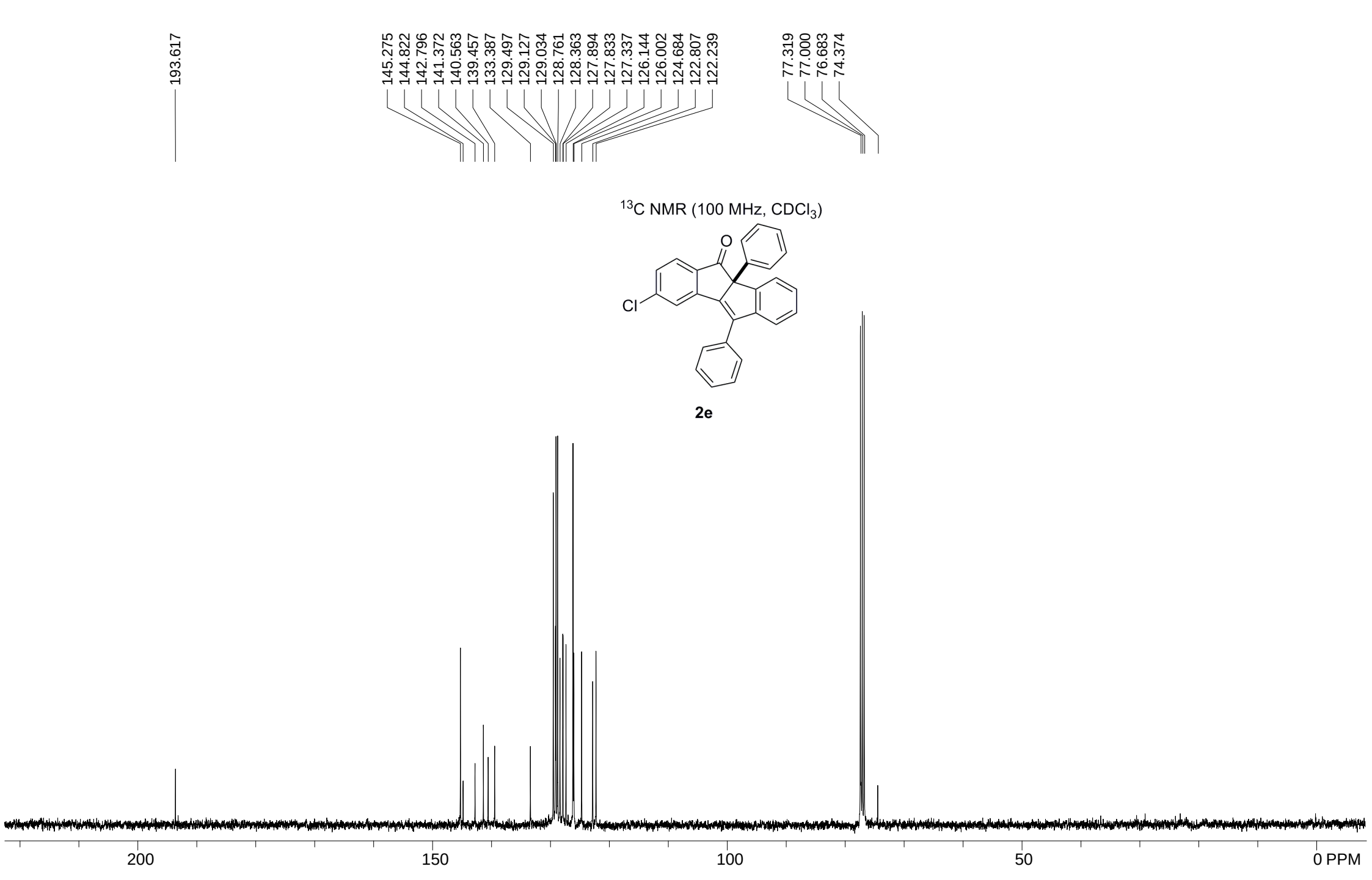


^{1}H NMR (400 MHz, $CDCl_3$)



2e

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133.387
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129.127
129.034
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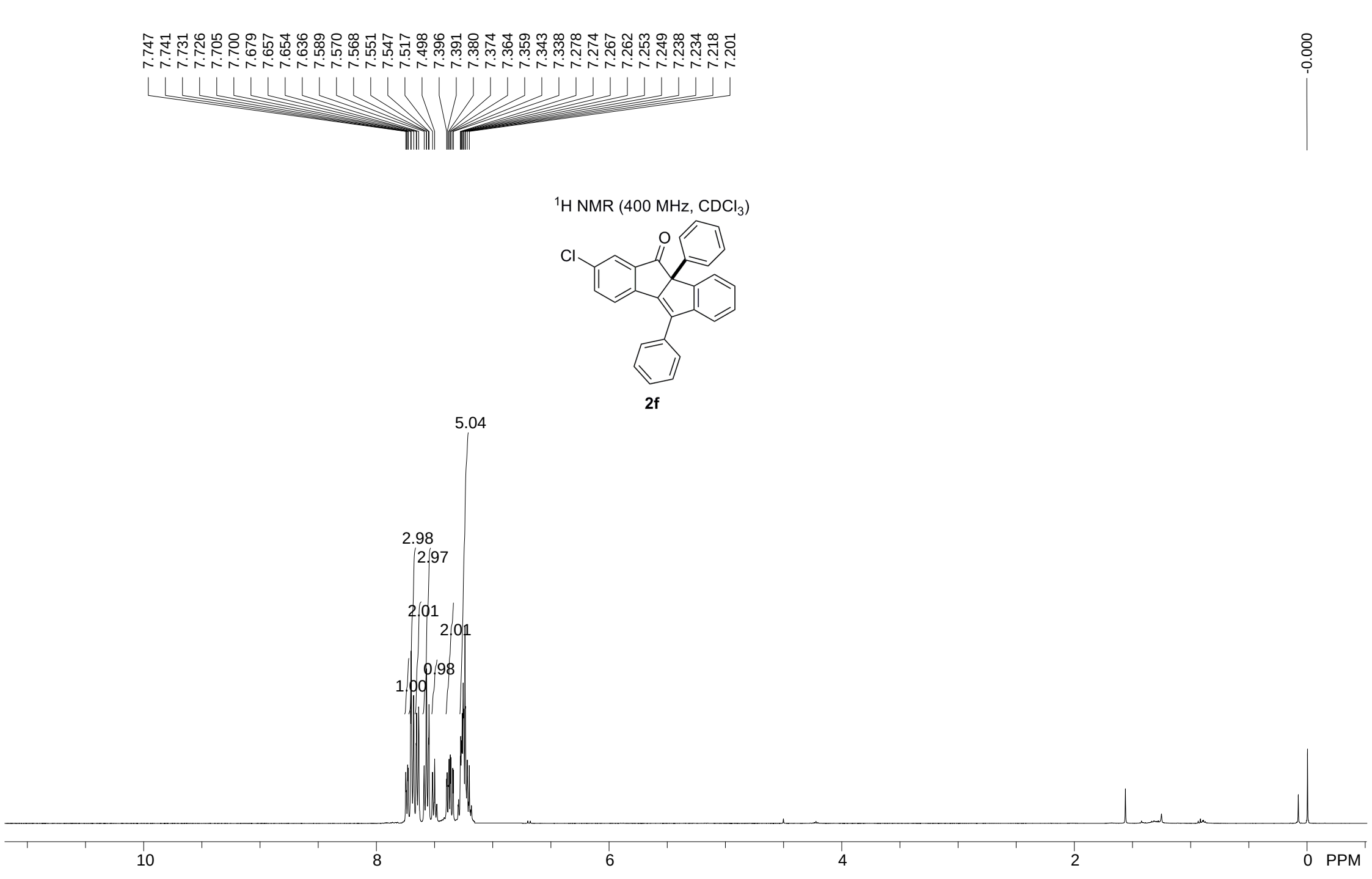
128.363
127.894
127.833
127.337
126.144
126.002
124.684
122.807
122.239
77.319
77.000
76.683
74.374
13C NMR (100 MHz, CDCl3)
O
Cl
2e
200
150
100
50
0 PPM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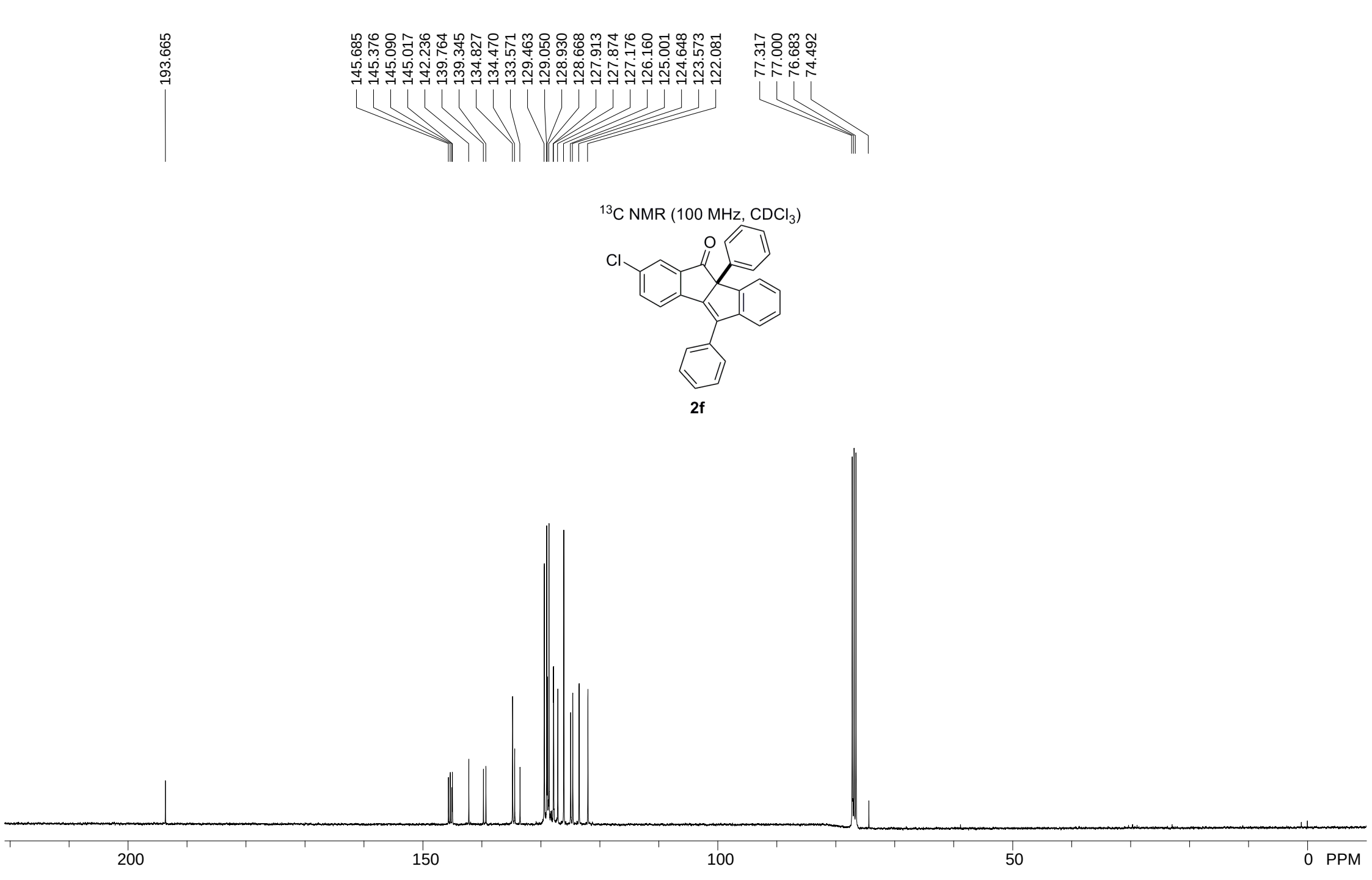
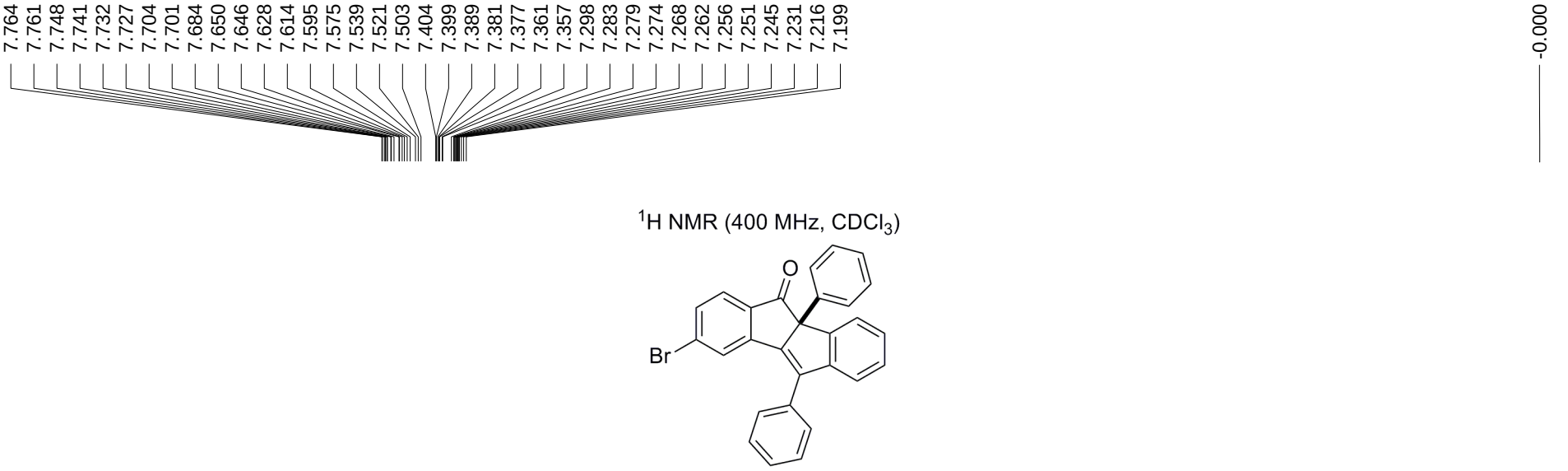
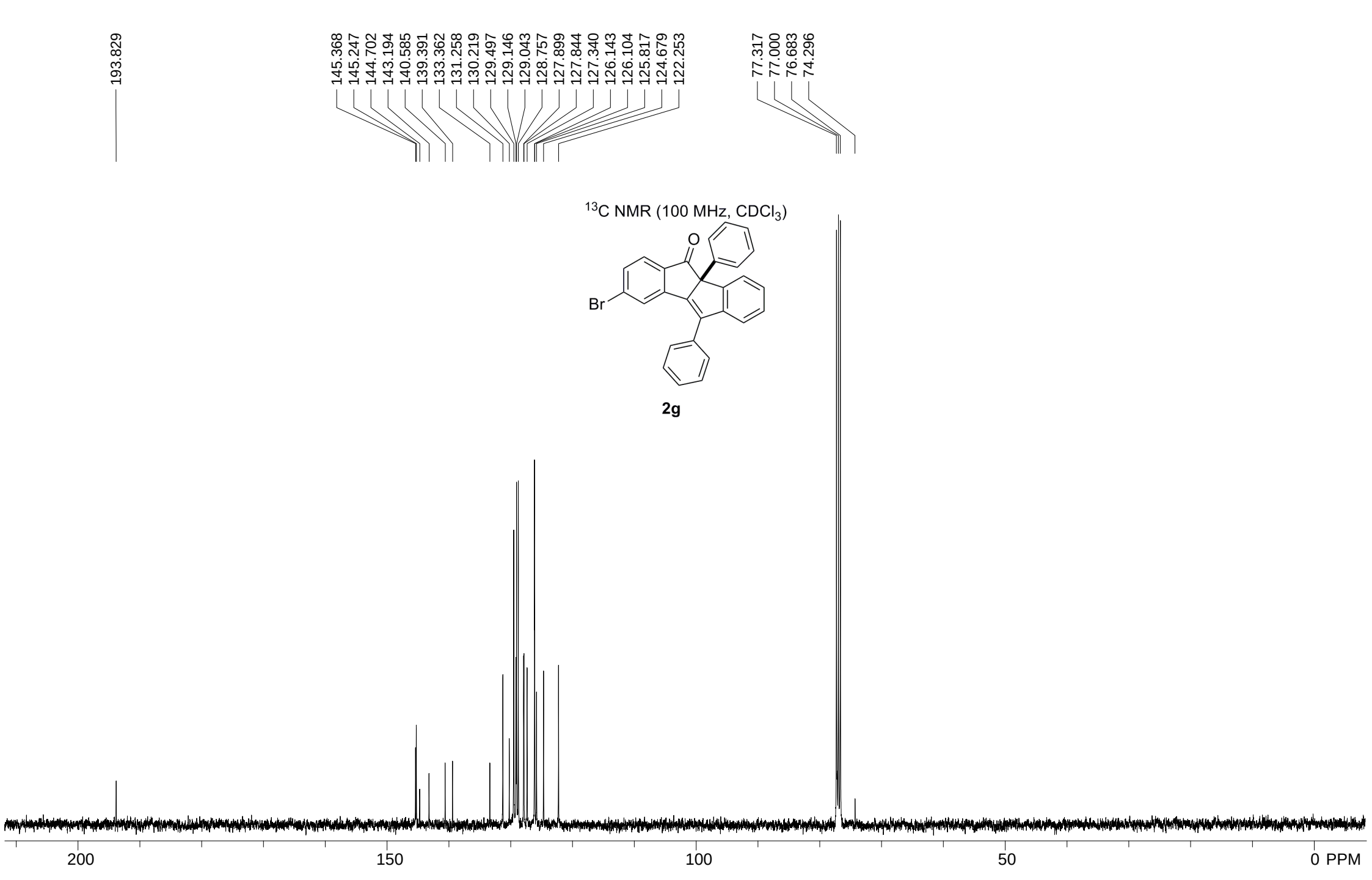
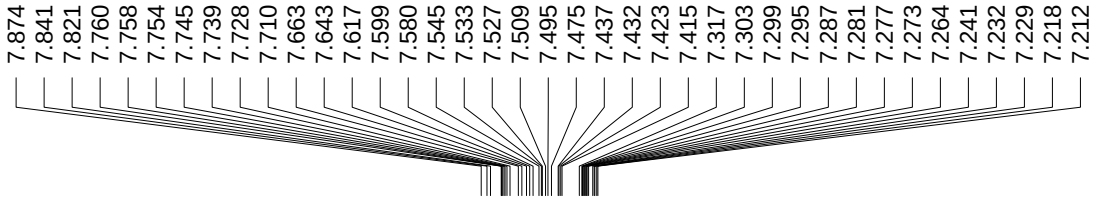
7.741
7.731
7.726
7.705
7.700
7.679
7.657
7.654
7.636
7.589
7.570
7.568
7.551
7.547
7.517
7.498
7.396
7.391
7.380
7.374
7.364
7.359
7.343
7.338
7.278
7.274
7.267
7.262
7.253
7.249
7.238
7.234
7.218
7.201
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
O
2f
5.04
2.98
2.97
2.01
2.01
0.98
1.00
10
8
6
4
2
0
PPM

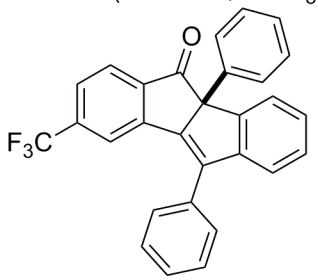

193.665
145.685
145.376
145.090
145.017
142.236
139.764
139.345
134.827
134.470
133.571
129.463
129.050
128.930
128.668
127.913
127.874
127.176
126.160
125.001
124.648
123.573
122.081
77.317
77.000
76.683
74.492
^{13}C NMR (100 MHz, $CDCl_3$)
Cl
O
2f
200
150
100
50
0
PPM


7.764
7.761
7.748
7.741
7.732
7.727
7.704
7.701
7.684
7.650
7.646
7.628
7.614
7.595
7.575
7.539
7.521
7.503
7.404
7.399
7.389
7.381
7.377
7.361
7.357
7.298
7.283
7.279
7.274
7.268
7.262
7.256
7.251
7.245
7.231
7.216
7.199
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
O
Br
2g
5.09
3.07
2.05
2.01
2.02
2.03
1.06
10
8
6
4
2
0
PPM

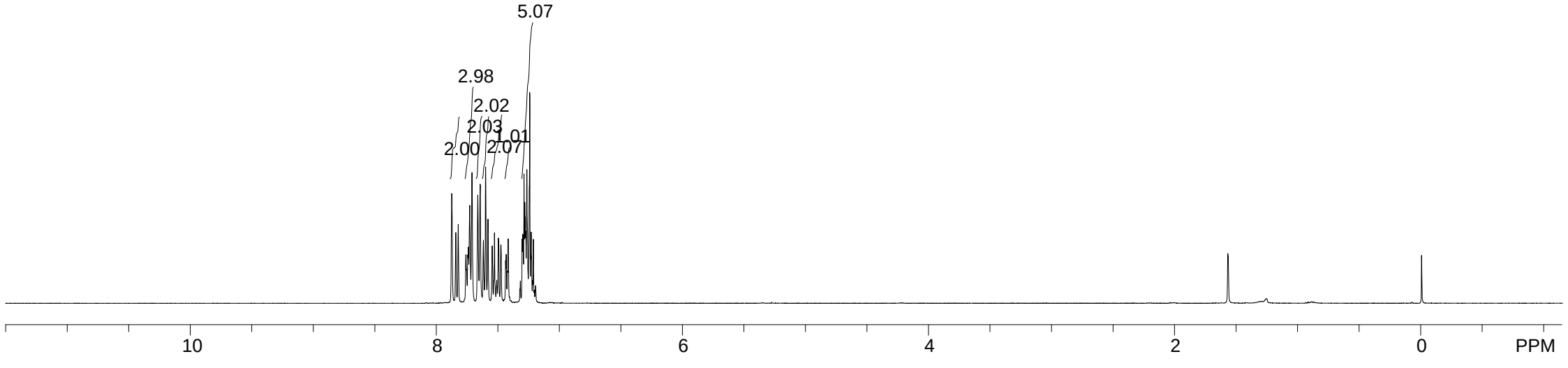

193.829
145.368
145.247
144.702
143.194
140.585
139.391
133.362
131.258
130.219
129.497
129.146
129.043
128.757
127.899
127.844
127.340
126.143
126.104
125.817
124.679
122.253
77.317
77.000
76.683
74.296
13C NMR (100 MHz, CDCl3)
O
Br
2g
200
150
100
50
0 PPM

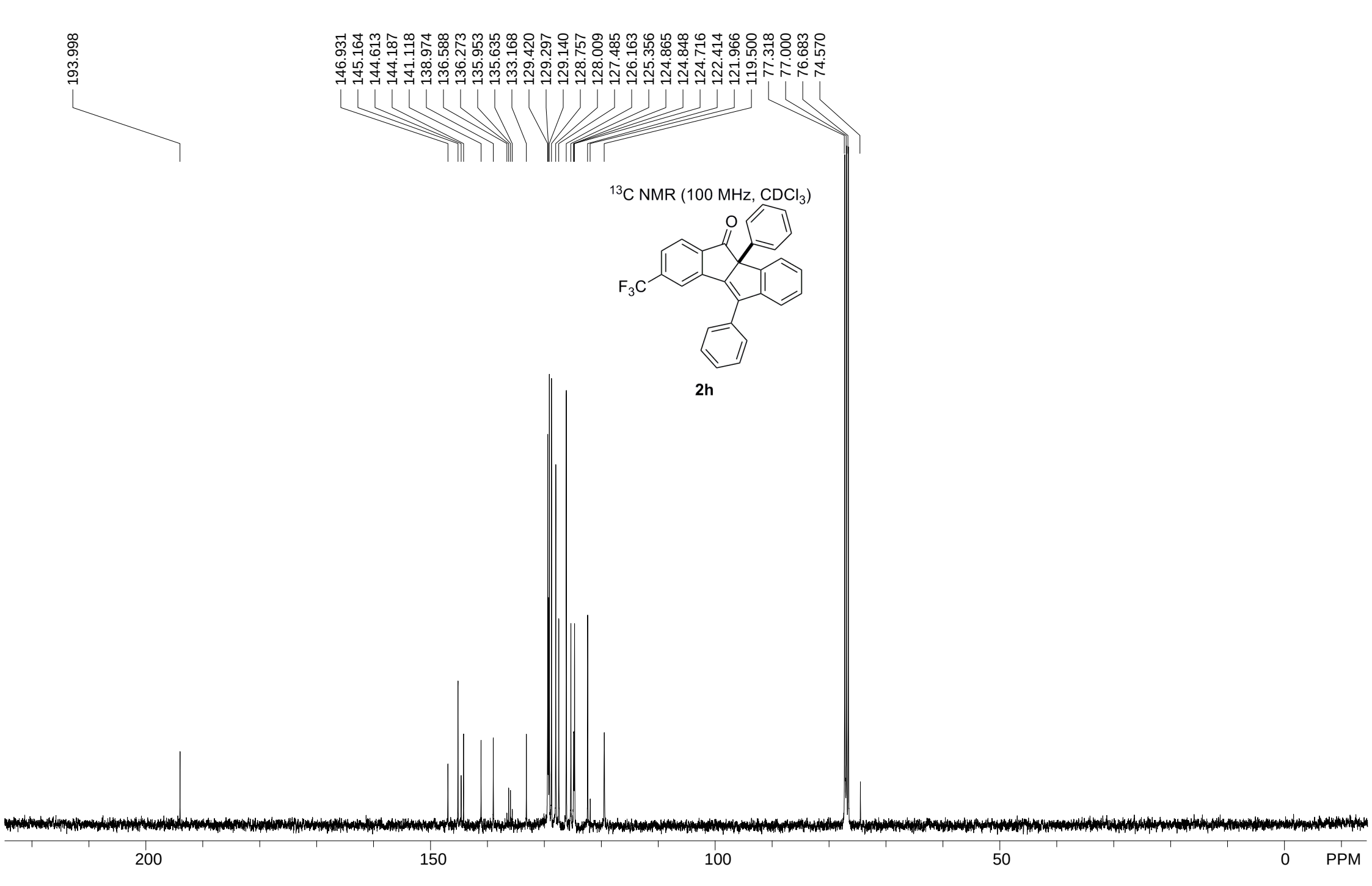


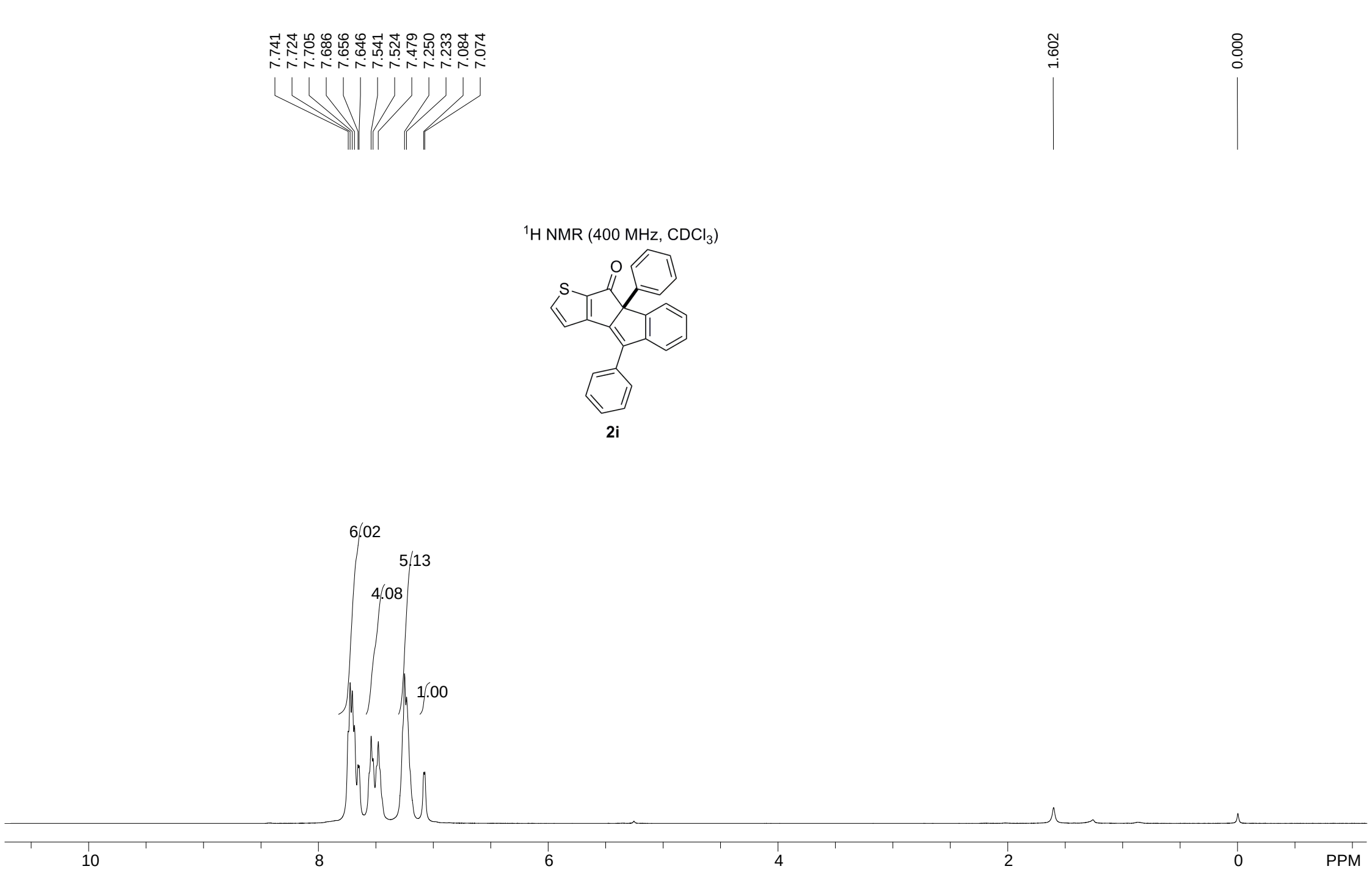
^{1}H NMR (400 MHz, $CDCl_3$)

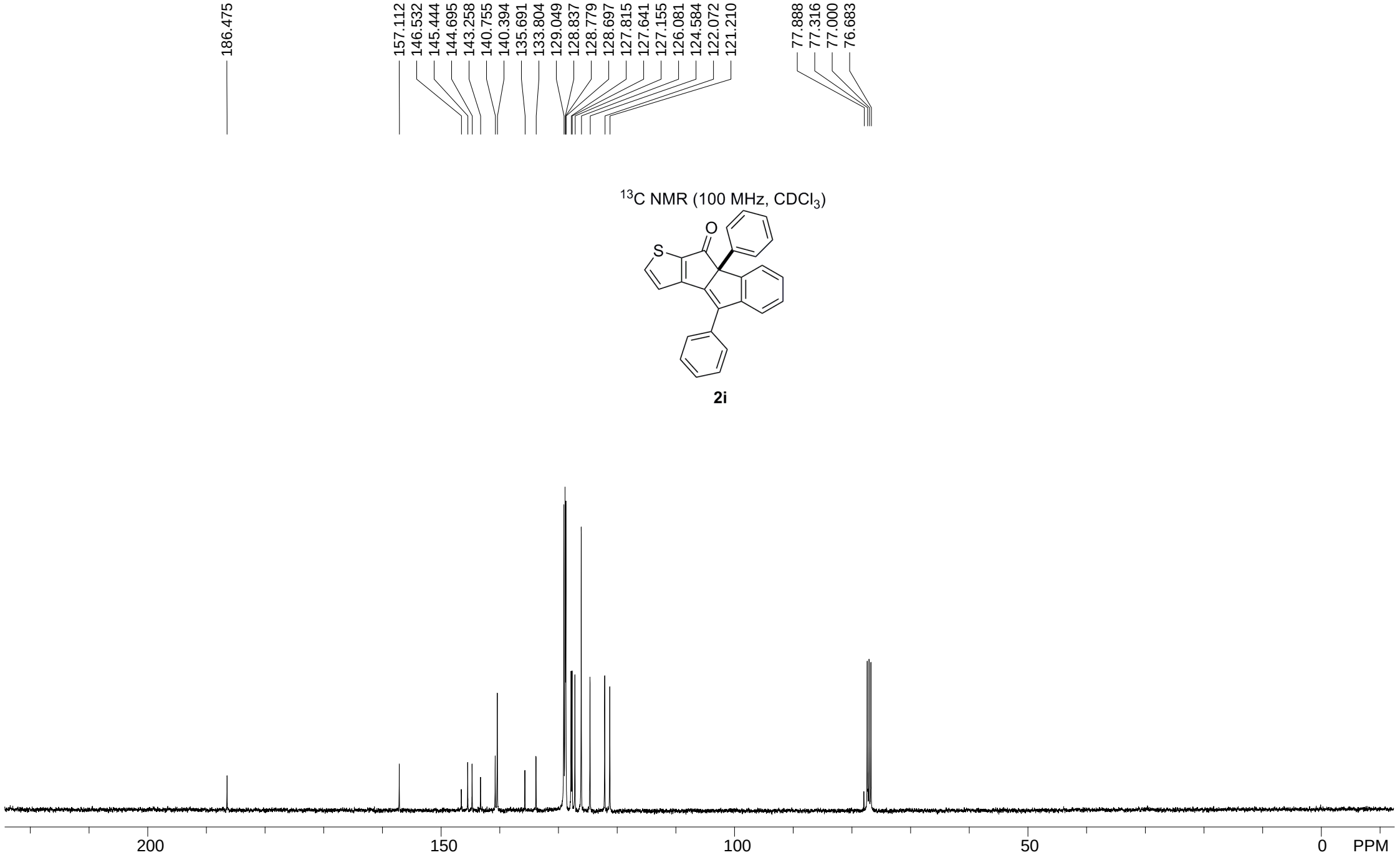


2h

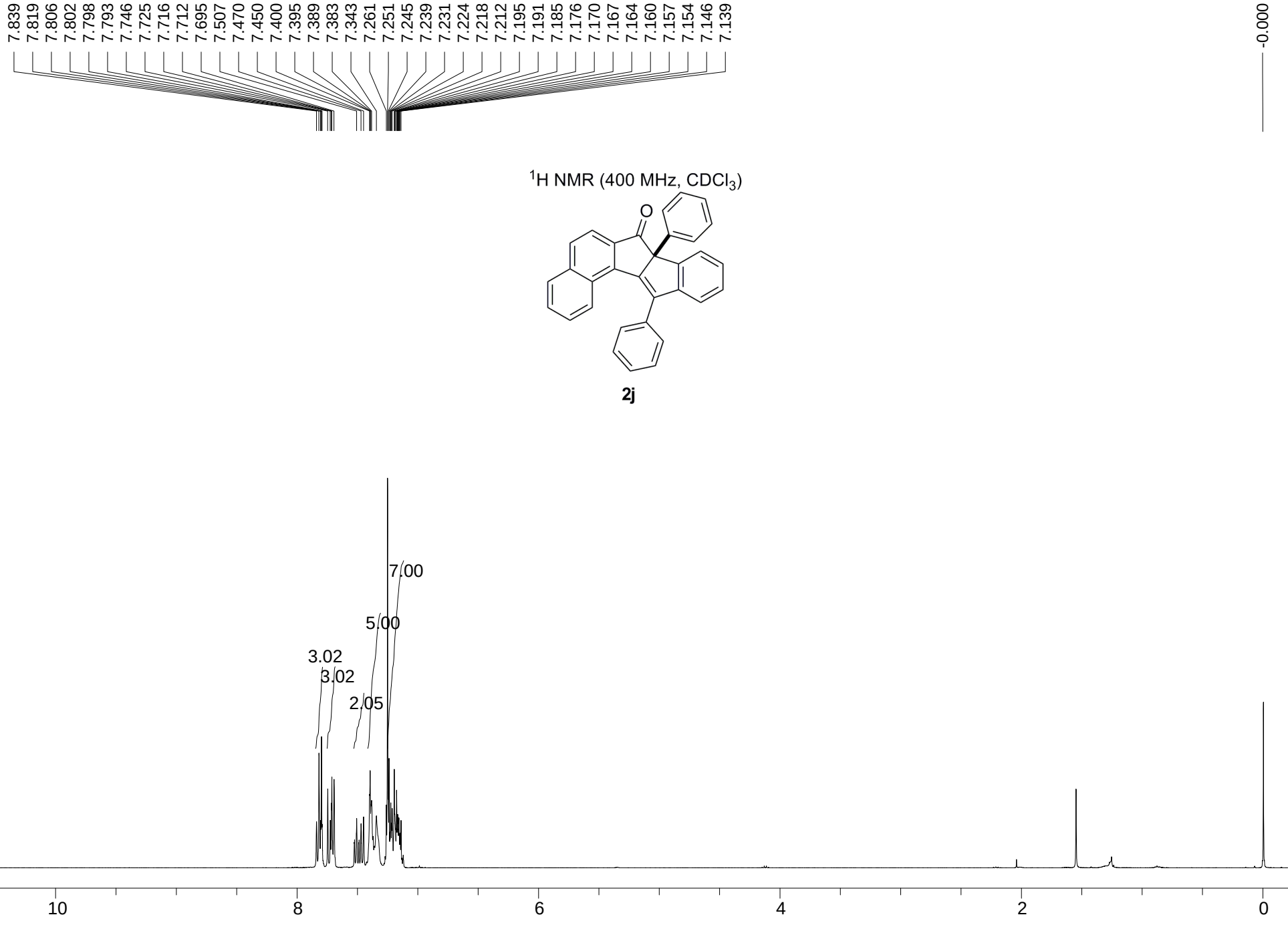


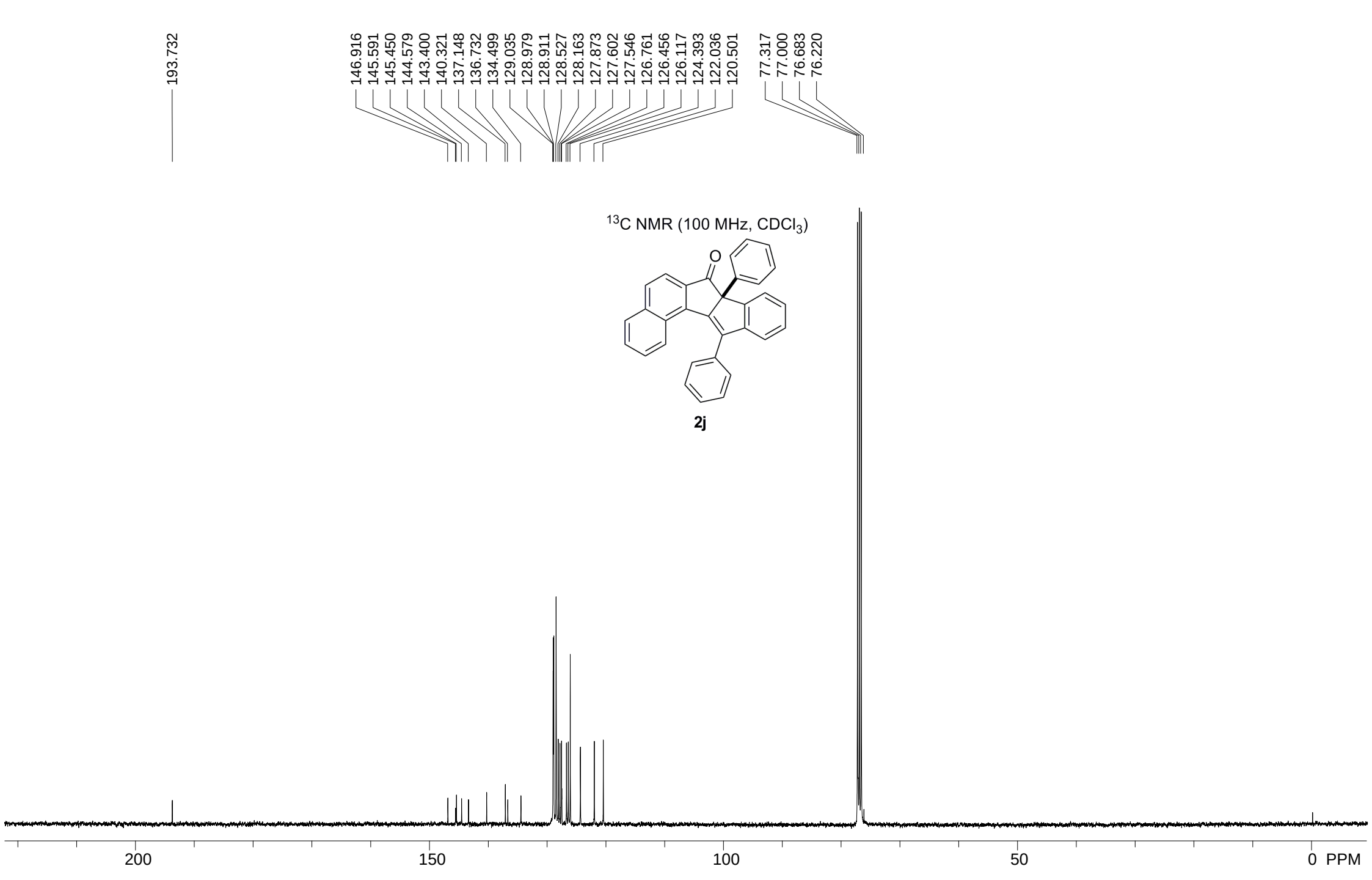

193.998
146.931
145.164
144.613
144.187
141.118
138.974
136.588
136.273
135.953
135.635
133.168
129.420
129.297
129.140
128.757
128.009
127.485
126.163
125.356
124.865
124.848
124.716
122.414
121.966
119.500
77.318
77.000
76.683
74.570
^{13}C NMR (100 MHz, $CDCl_3$)
O
F_3C
2h
200
150
100
50
0
PPM

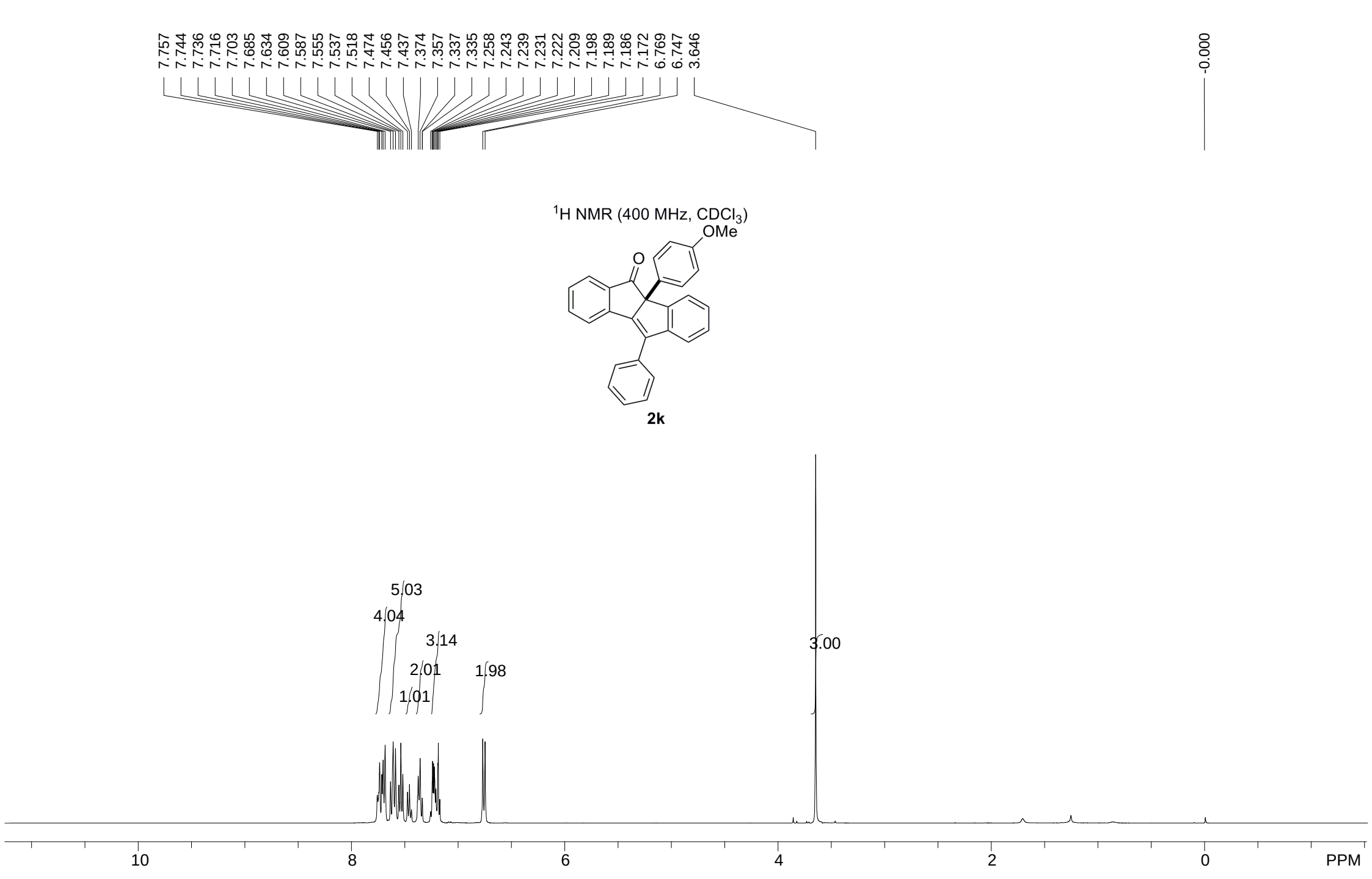

7.741
7.724
7.705
7.686
7.656
7.646
7.541
7.524
7.479
7.250
7.233
7.084
7.074
1.602
0.000
1H NMR (400 MHz, CDCl3)
O
S
2i
6.02
5.13
4.08
1.00
10
8
6
4
2
0
PPM

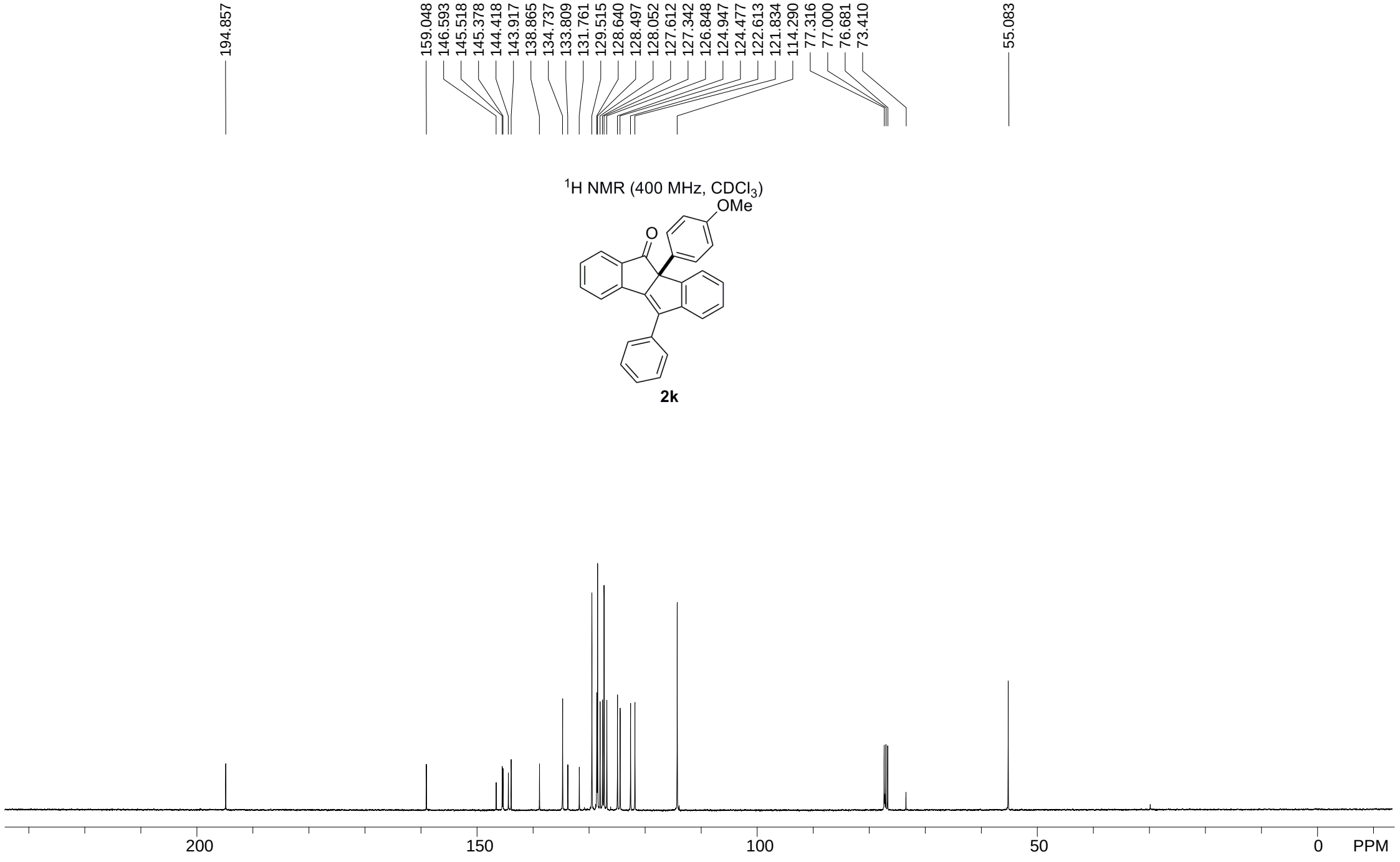

186.475
157.112
146.532
145.444
144.695
143.258
140.755
140.394
135.691
133.804
129.049
128.837
128.779
128.697
127.815
127.641
127.155
126.081
124.584
122.072
121.210
77.888
77.316
77.000
76.683
^{13}C NMR (100 MHz, $CDCl_3$)
O
S
200
150
100
50
0
PPM

2i


7.839
7.819
7.806
7.802
7.798
7.793
7.746
7.725
7.716
7.712
7.695
7.507
7.470
7.450
7.400
7.395
7.389
7.383
7.343
7.261
7.251
7.245
7.239
7.231
7.224
7.218
7.212
7.195
7.191
7.185
7.176
7.170
7.167
7.164
7.160
7.157
7.154
7.146
7.139
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
O
2j
7.00
5.00
3.02
3.02
2.05
10
8
6
4
2
0
PPM

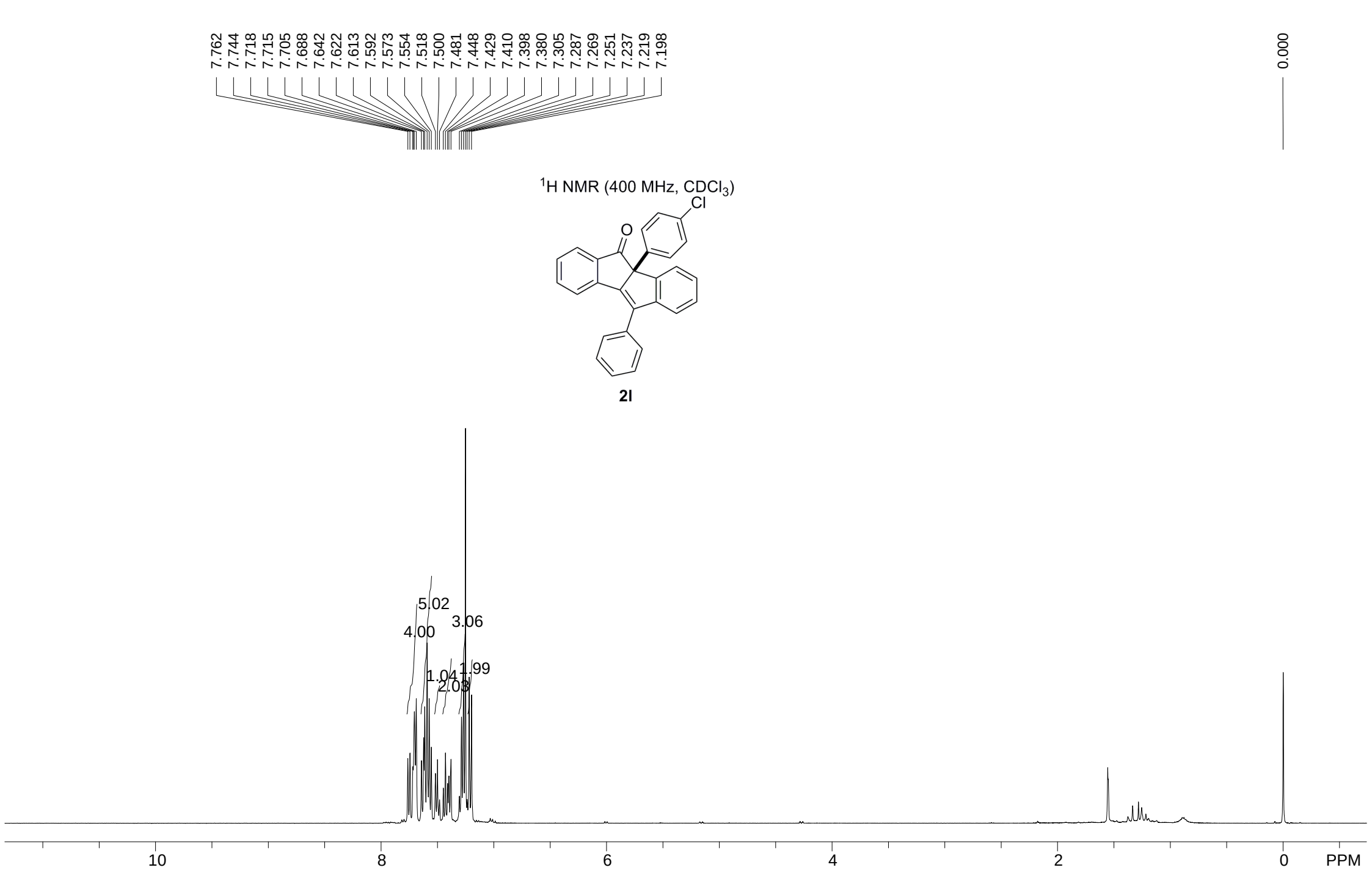

193.732
146.916
145.591
145.450
144.579
143.400
140.321
137.148
136.732
134.499
129.035
128.979
128.911
128.527
128.163
127.873
127.602
127.546
126.761
126.456
126.117
124.393
122.036
120.501
77.317
77.000
76.683
76.220
^{13}C NMR (100 MHz, $CDCl_3$)
O
2j
200
150
100
50
0 PPM


7.757
7.744
7.736
7.716
7.703
7.685
7.634
7.609
7.587
7.555
7.537
7.518
7.474
7.456
7.437
7.374
7.357
7.337
7.335
7.258
7.243
7.239
7.231
7.222
7.209
7.198
7.189
7.186
7.172
6.769
6.747
3.646
-0.000
1H NMR (400 MHz, CDCl3)
OMe
O
2k
5.03
4.04
3.14
2.01
1.98
1.01
3.00
10
8
6
4
2
0
PPM



^{1}H NMR (400 MHz, $CDCl_3$)

2k

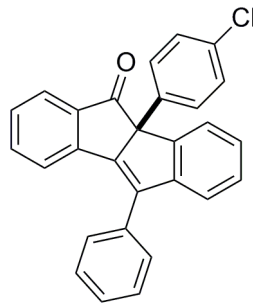

7.762
7.744
7.718
7.715
7.705
7.688
7.642
7.622
7.613
7.592
7.573
7.554
7.518
7.500
7.481
7.448
7.429
7.410
7.398
7.380
7.305
7.287
7.269
7.251
7.237
7.219
7.198
0.000
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
O
2l
5.02
3.06
4.00
1.04
1.99
2.08
10
8
6
4
2
0
PPM

194.606

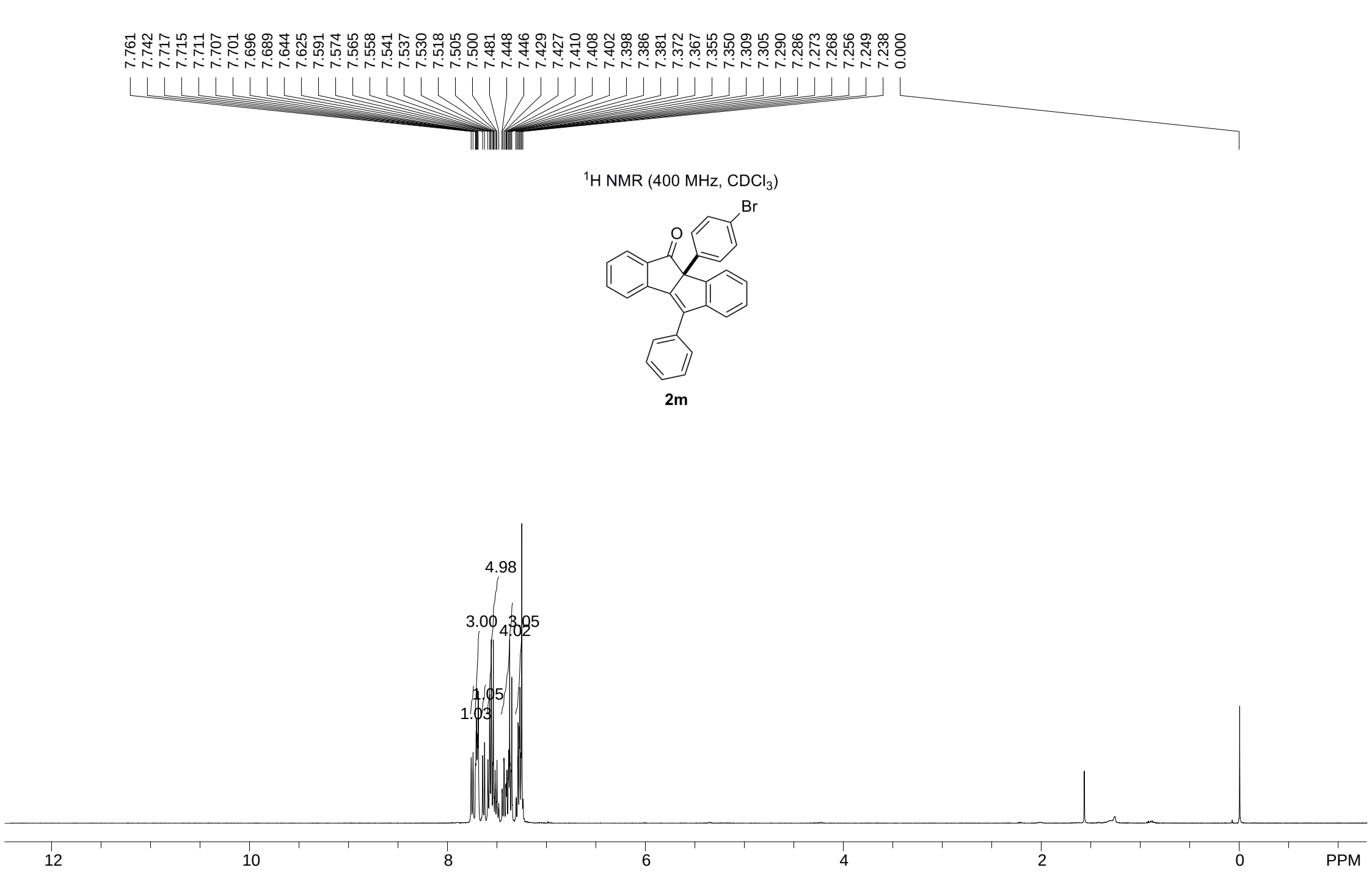
146.079
145.505
144.904
144.321
143.969
139.470
138.432
135.076
133.642
129.551
129.097
128.848
128.772
128.611
128.265
127.961
127.616
127.104
125.089
124.562
122.749
122.080

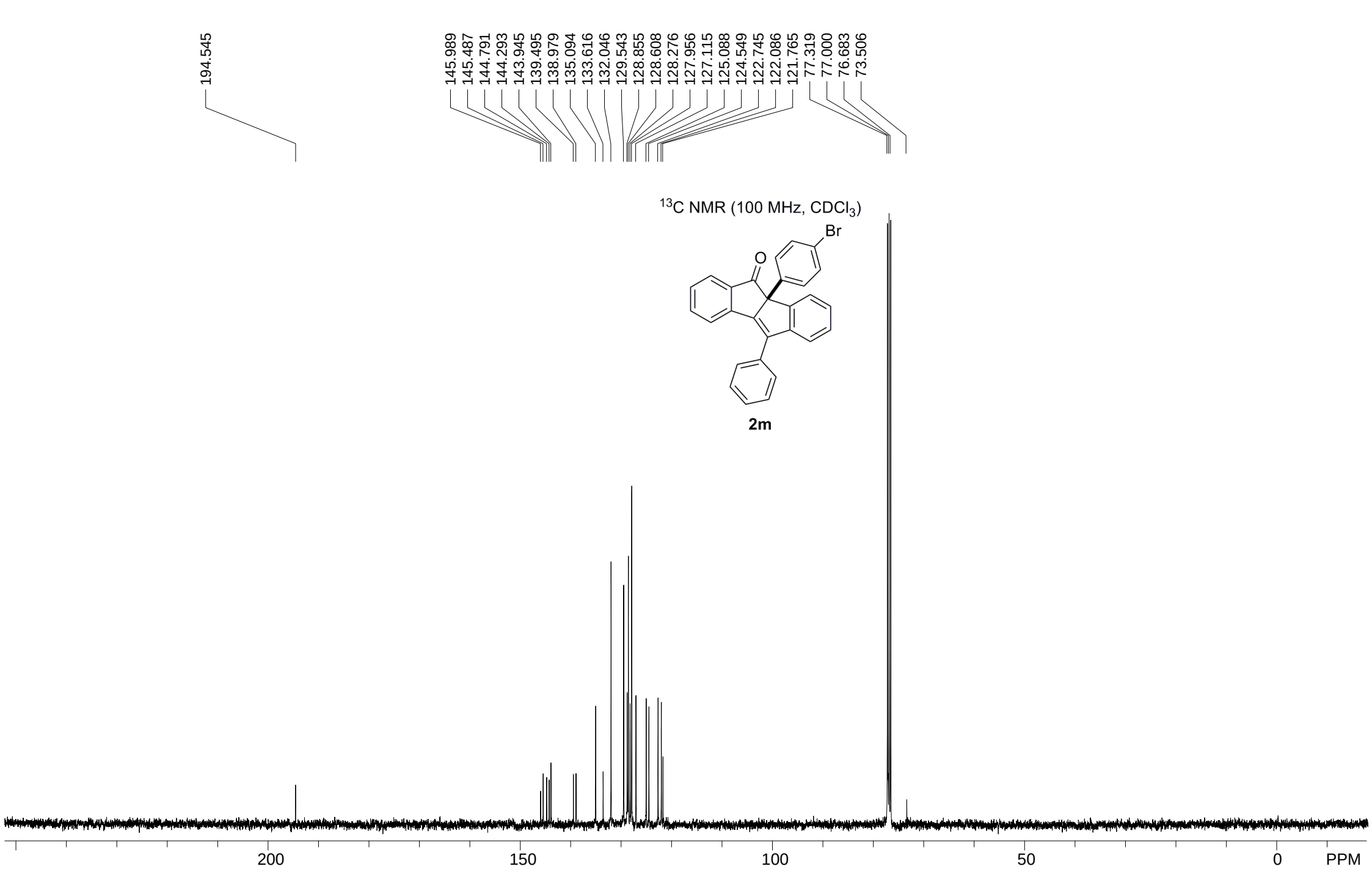
77.317
77.000
76.683
73.466

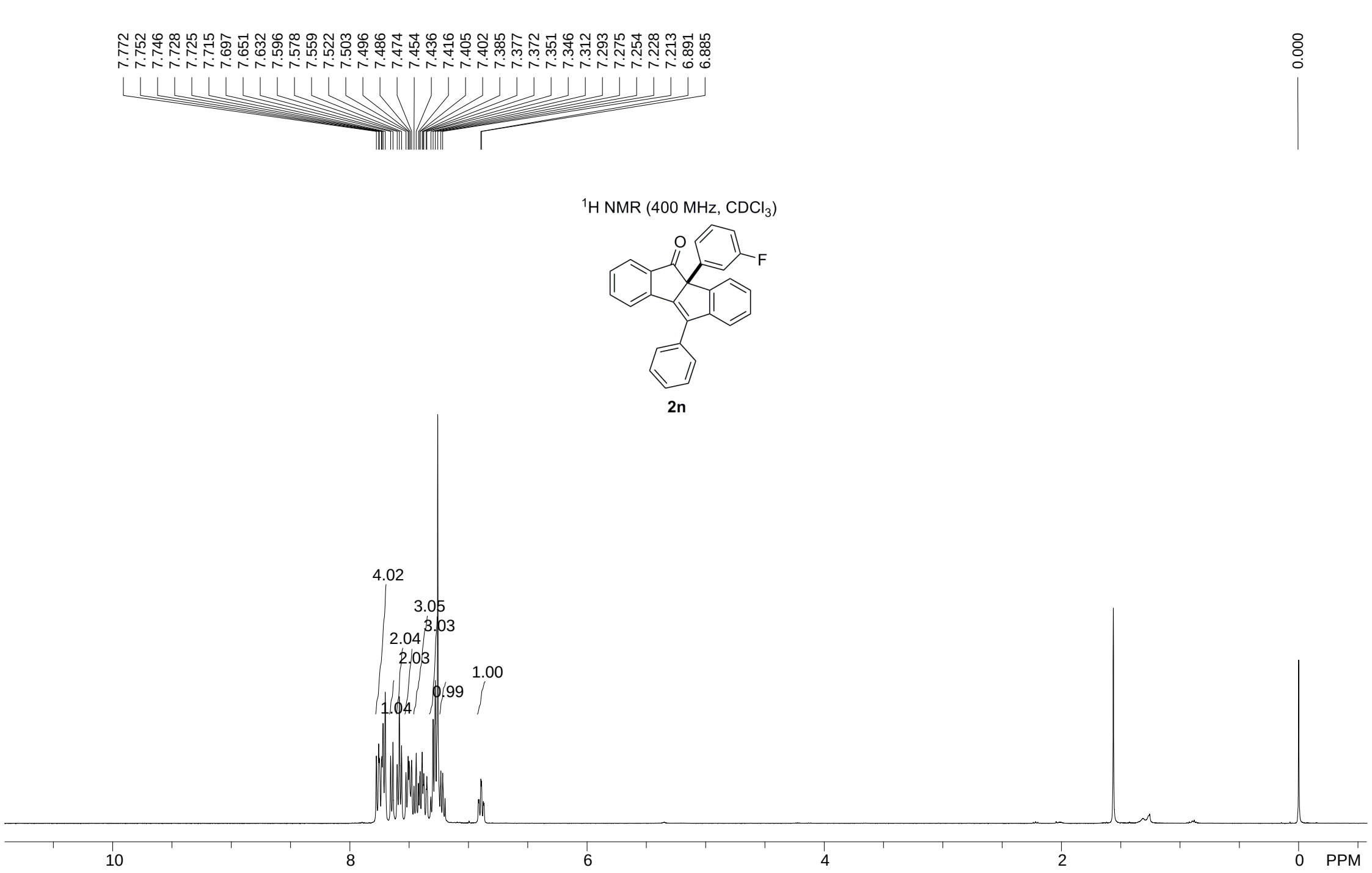
^{13}C NMR (100 MHz, $CDCl_3$)

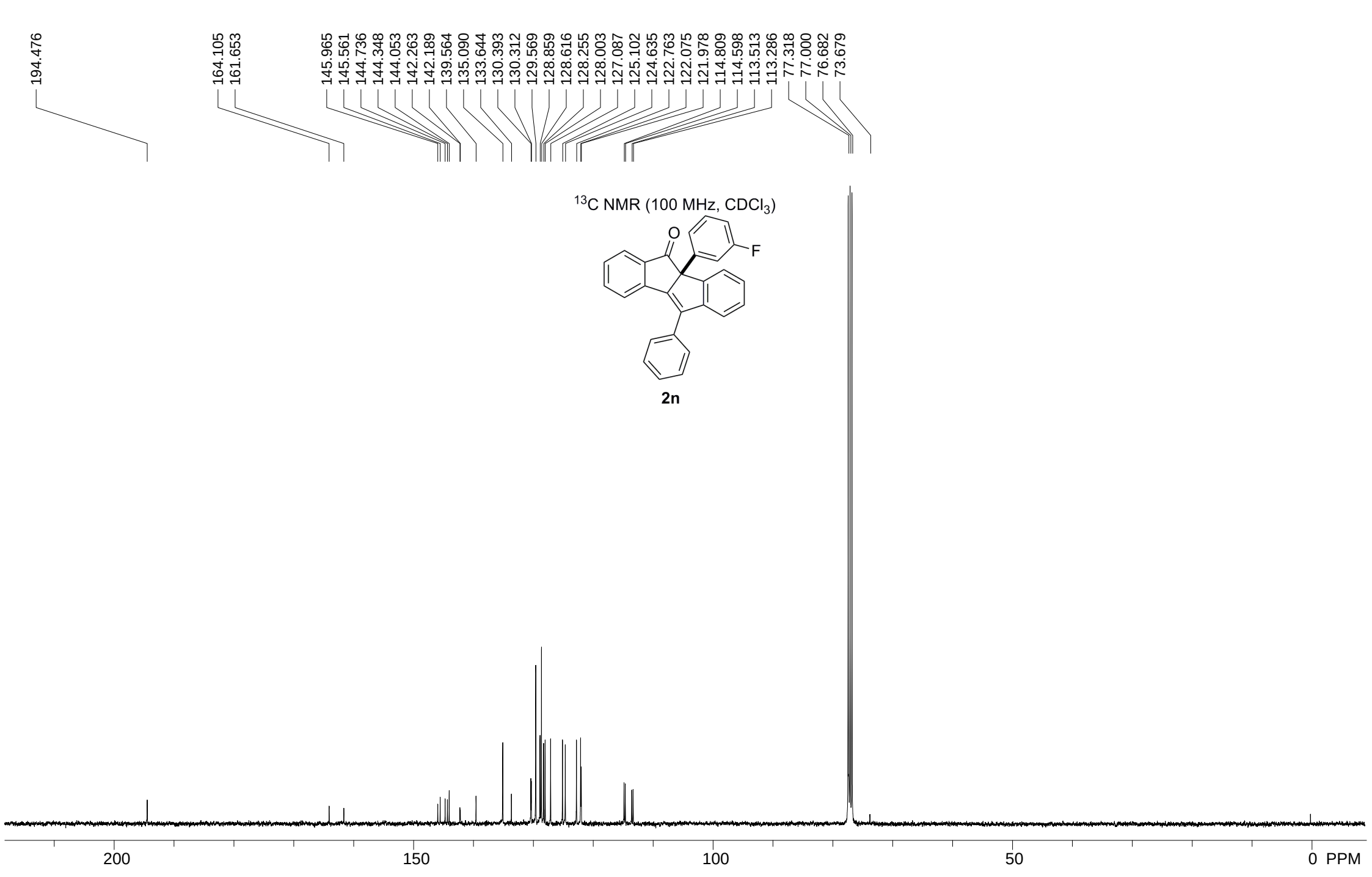


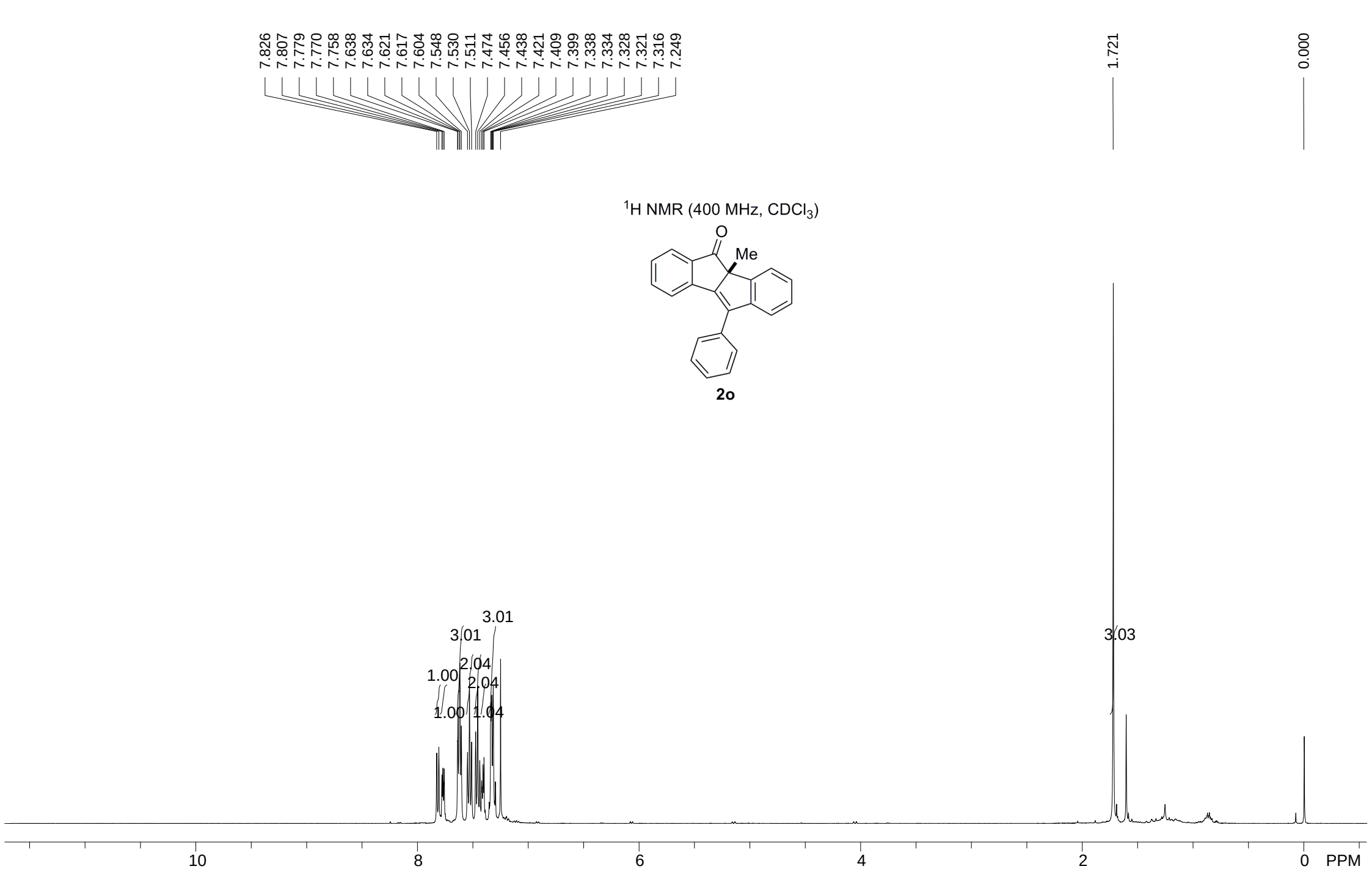
2l

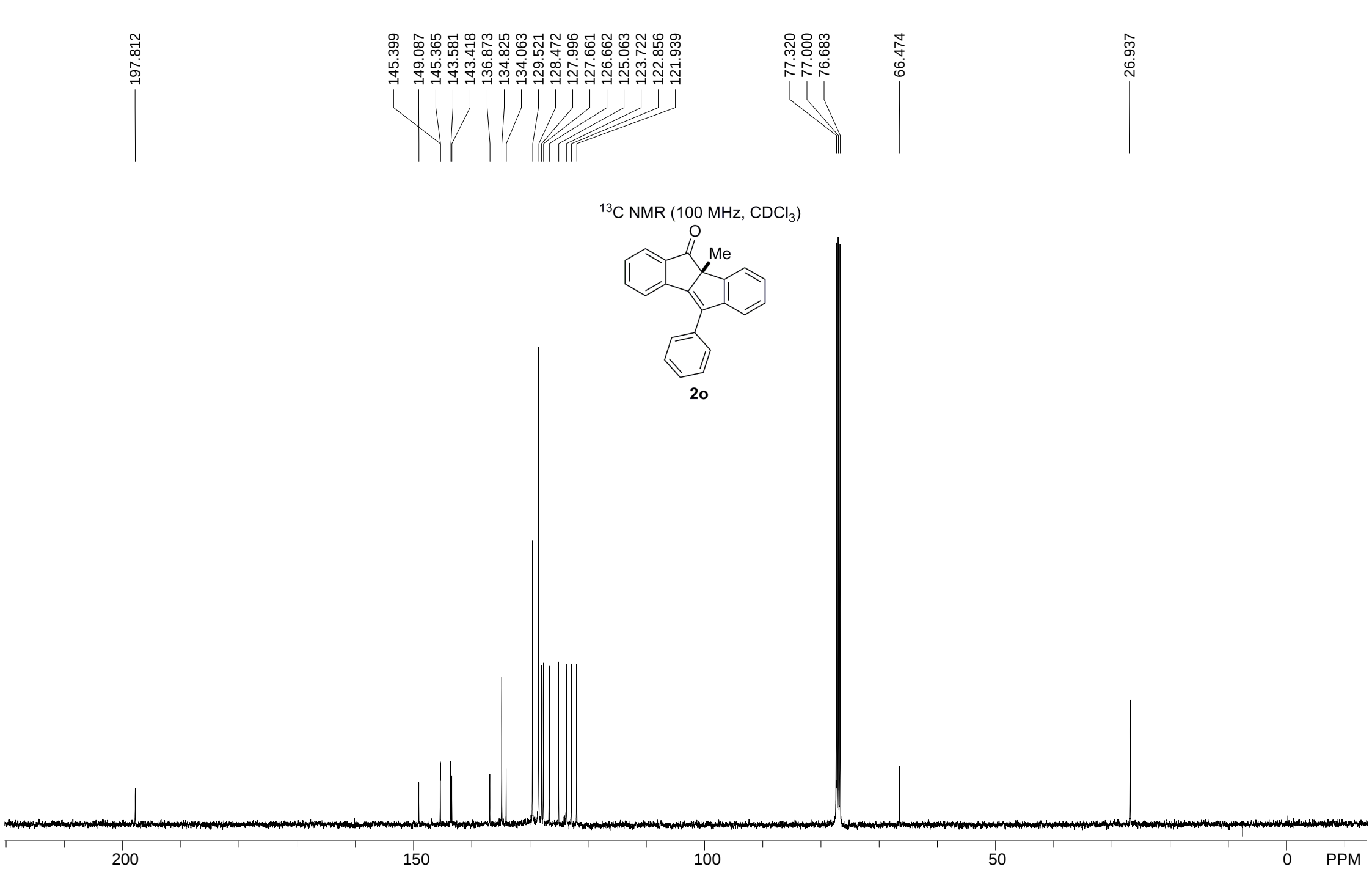

7.761
7.742
7.717
7.715
7.711
7.707
7.701
7.696
7.689
7.644
7.625
7.591
7.574
7.565
7.558
7.541
7.537
7.530
7.518
7.505
7.500
7.481
7.448
7.446
7.429
7.427
7.410
7.408
7.402
7.398
7.386
7.381
7.372
7.367
7.355
7.350
7.309
7.305
7.290
7.286
7.273
7.268
7.256
7.249
7.238
0.000
^{1}H NMR (400 MHz, CDCl$_3$)
Br
O
2m
4.98
3.00
3.05
4.02
1.05
1.03
12
10
8
6
4
2
0
PPM

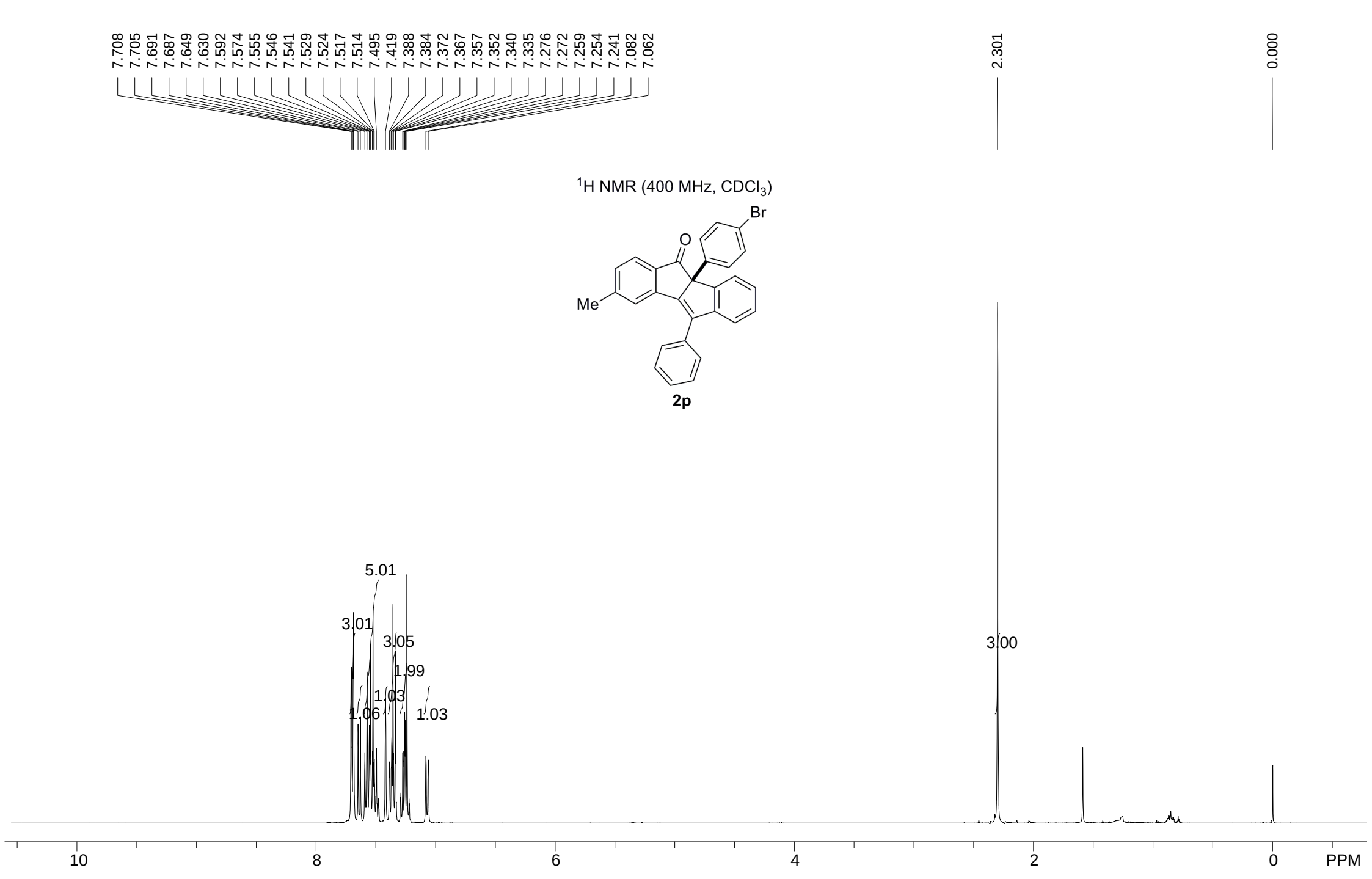

194.545
145.989
145.487
144.791
144.293
143.945
139.495
138.979
135.094
133.616
132.046
129.543
128.855
128.608
128.276
127.956
127.115
125.088
124.549
122.745
122.086
121.765
77.319
77.000
76.683
73.506
^{13}C NMR (100 MHz, $CDCl_3$)
Br
O
2m
200
150
100
50
0
PPM

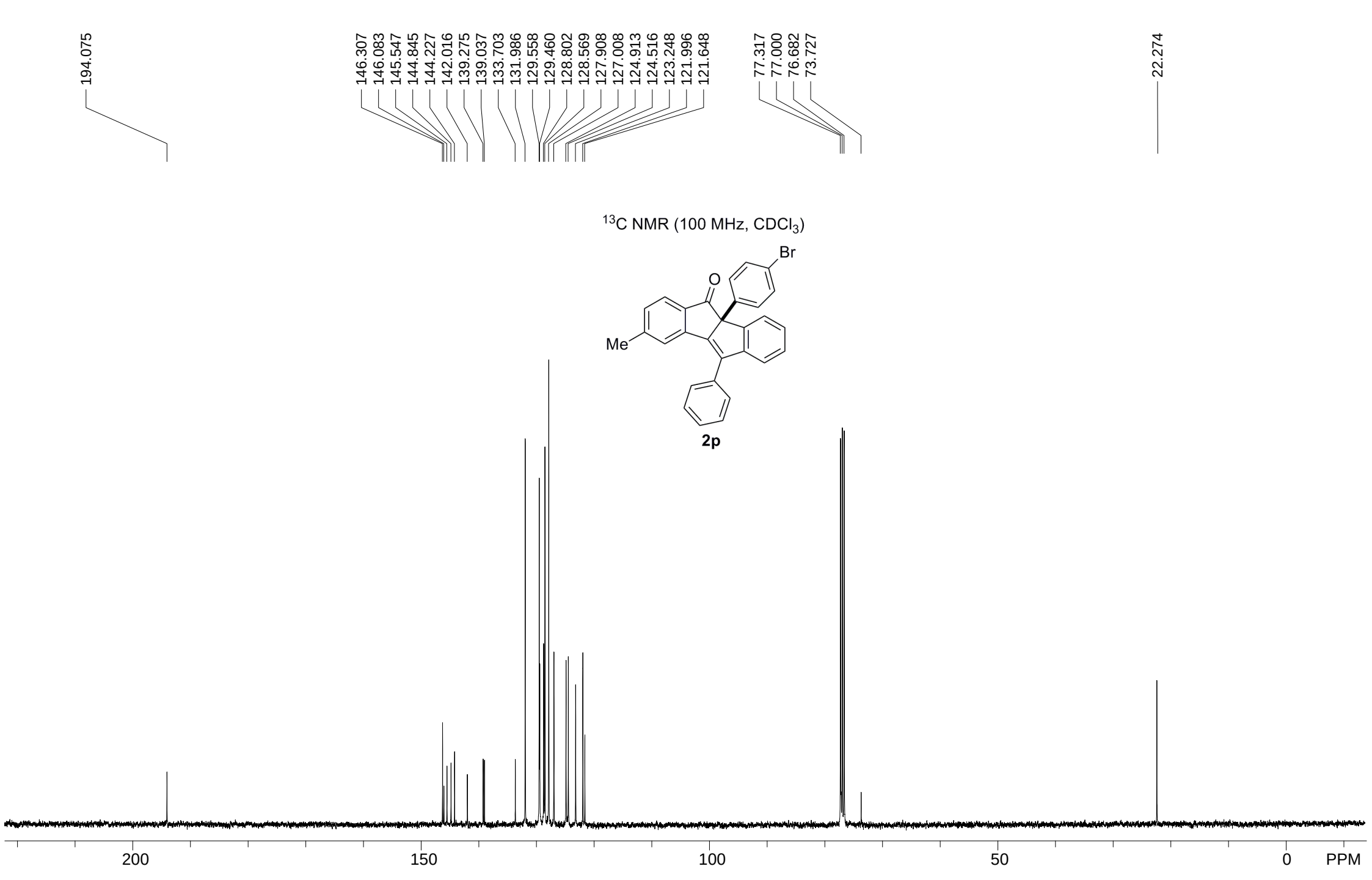

7.772
7.752
7.746
7.728
7.725
7.715
7.697
7.651
7.632
7.596
7.578
7.559
7.522
7.503
7.496
7.486
7.474
7.454
7.436
7.416
7.405
7.402
7.385
7.377
7.372
7.351
7.346
7.312
7.293
7.275
7.254
7.228
7.213
6.891
6.885
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
F
2n
4.02
3.05
3.03
2.04
2.03
1.00
0.99
1.04
10
8
6
4
2
0
PPM

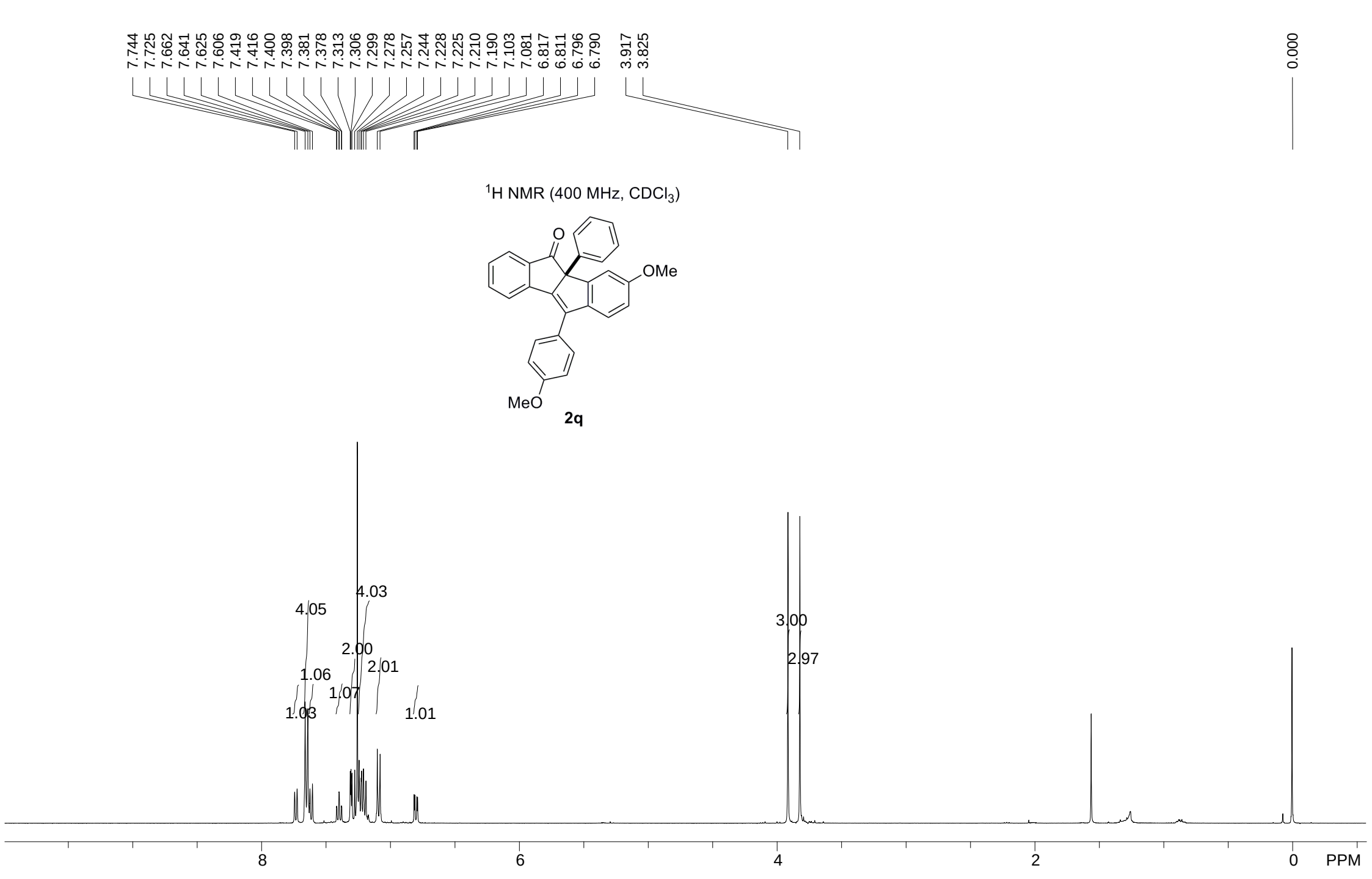

194.476
164.105
161.653
145.965
145.561
144.736
144.348
144.053
142.263
142.189
139.564
135.090
133.644
130.393
130.312
129.569
128.859
128.616
128.255
128.003
127.087
125.102
124.635
122.763
122.075
121.978
114.809
114.598
113.513
113.286
77.318
77.000
76.682
73.679
^{13}C NMR (100 MHz, $CDCl_3$)
O
F
2n
200
150
100
50
0 PPM

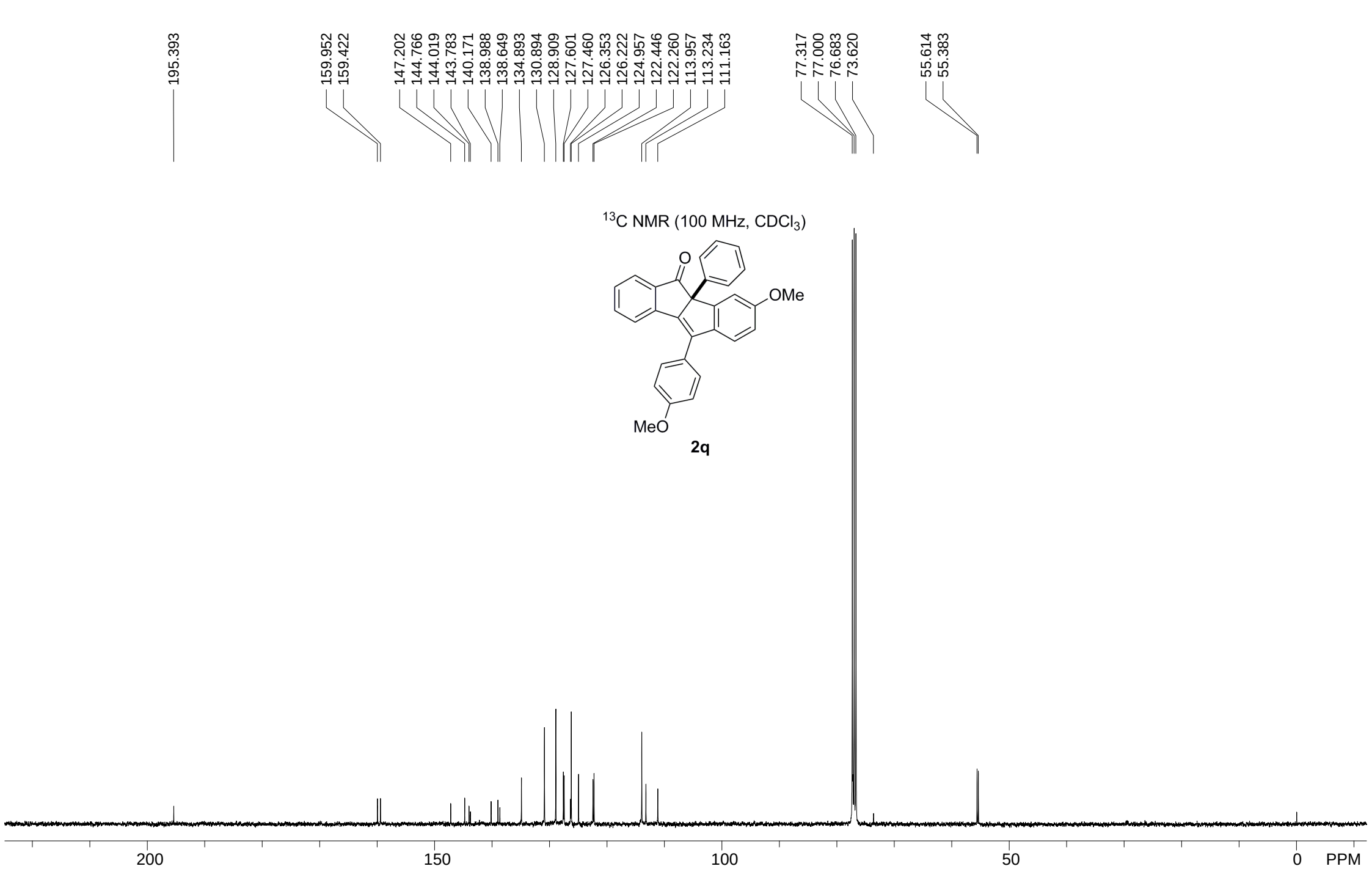

7.826
7.807
7.779
7.770
7.758
7.638
7.634
7.621
7.617
7.604
7.548
7.530
7.511
7.474
7.456
7.438
7.421
7.409
7.399
7.338
7.334
7.328
7.321
7.316
7.249
1.721
0.000
1H NMR (400 MHz, CDCl3)
O
Me
2o
3.01
3.01
2.04
1.00
2.04
1.00
1.04
3.03
10
8
6
4
2
0
PPM


197.812
145.399
149.087
145.365
143.581
143.418
136.873
134.825
134.063
129.521
128.472
127.996
127.661
126.662
125.063
123.722
122.856
121.939
77.320
77.000
76.683
66.474
26.937
13C NMR (100 MHz, CDCl3)
O
Me
2o
200
150
100
50
0
PPM

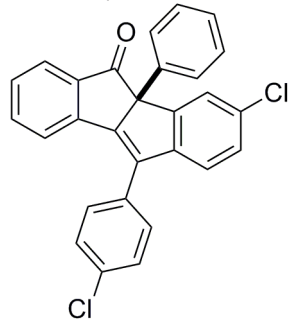

7.708
7.705
7.691
7.687
7.649
7.630
7.592
7.574
7.555
7.546
7.541
7.529
7.524
7.517
7.514
7.495
7.419
7.388
7.384
7.372
7.367
7.357
7.352
7.340
7.335
7.276
7.272
7.259
7.254
7.241
7.082
7.062
2.301
0.000
1H NMR (400 MHz, CDCl3)
Br
O
Me
2p
5.01
3.01
3.05
1.99
1.03
1.06
1.03
3.00
10
8
6
4
2
0
PPM


194.075
146.307
146.083
145.547
144.845
144.227
142.016
139.275
139.037
133.703
131.986
129.558
129.460
128.802
128.569
127.908
127.008
124.913
124.516
123.248
121.996
121.648
77.317
77.000
76.682
73.727
22.274
13C NMR (100 MHz, CDCl3)
Br
O
Me
2p
200
150
100
50
0
PPM

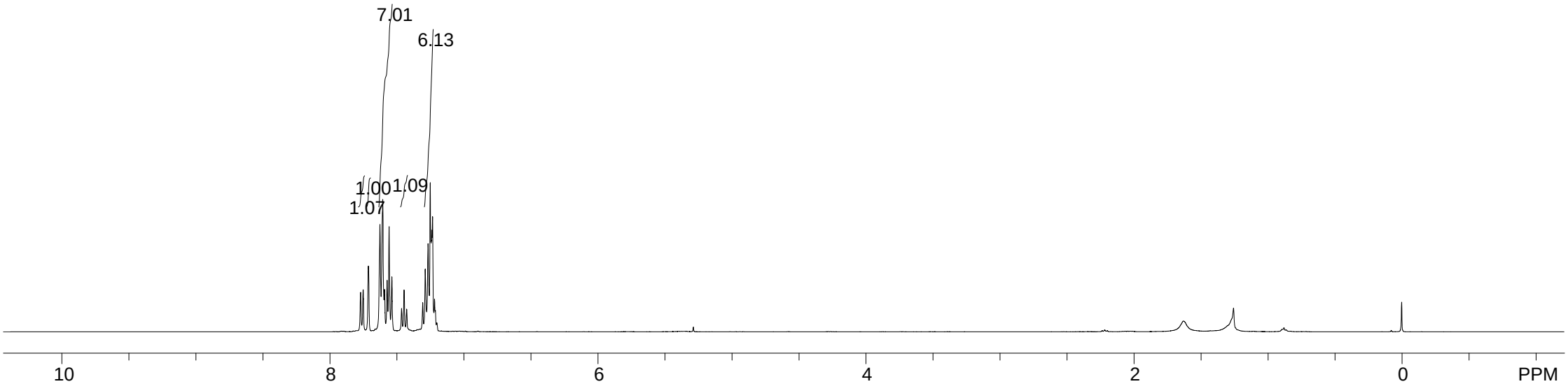

7.744
7.725
7.662
7.641
7.625
7.606
7.419
7.416
7.400
7.398
7.381
7.378
7.313
7.306
7.299
7.278
7.257
7.244
7.228
7.225
7.210
7.190
7.103
7.081
6.817
6.811
6.796
6.790
3.917
3.825
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
OMe
MeO
2q
4.05
4.03
2.00
2.01
1.06
1.07
1.03
1.01
3.00
2.97
8
6
4
2
0
PPM


195.393
159.952
159.422
147.202
144.766
144.019
143.783
140.171
138.988
138.649
134.893
130.894
128.909
127.601
127.460
126.353
126.222
124.957
122.446
122.260
113.957
113.234
111.163
77.317
77.000
76.683
73.620
55.614
55.383
^{13}C NMR (100 MHz, $CDCl_3$)
O
OMe
MeO
2q
200
150
100
50
0
PPM

^{1}H NMR (400 MHz, $CDCl_3$)

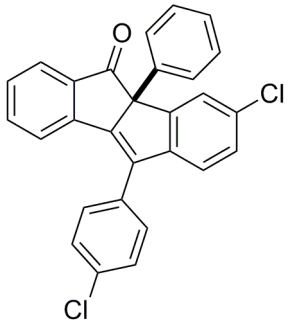


2r

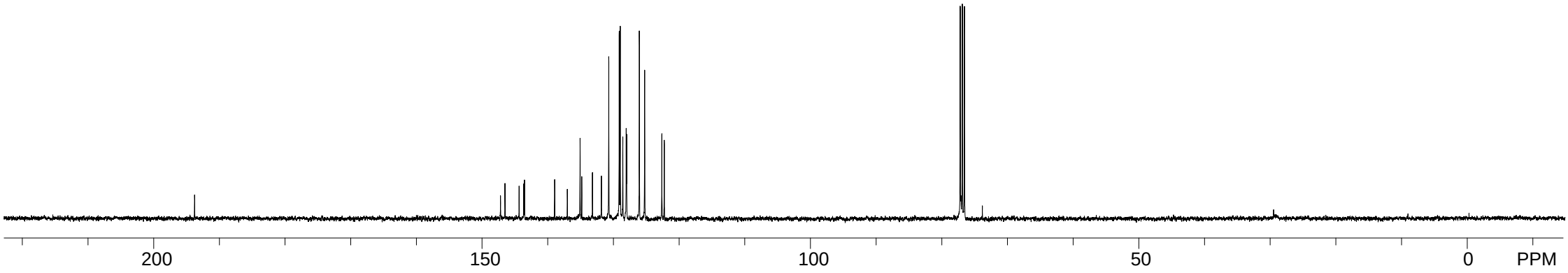


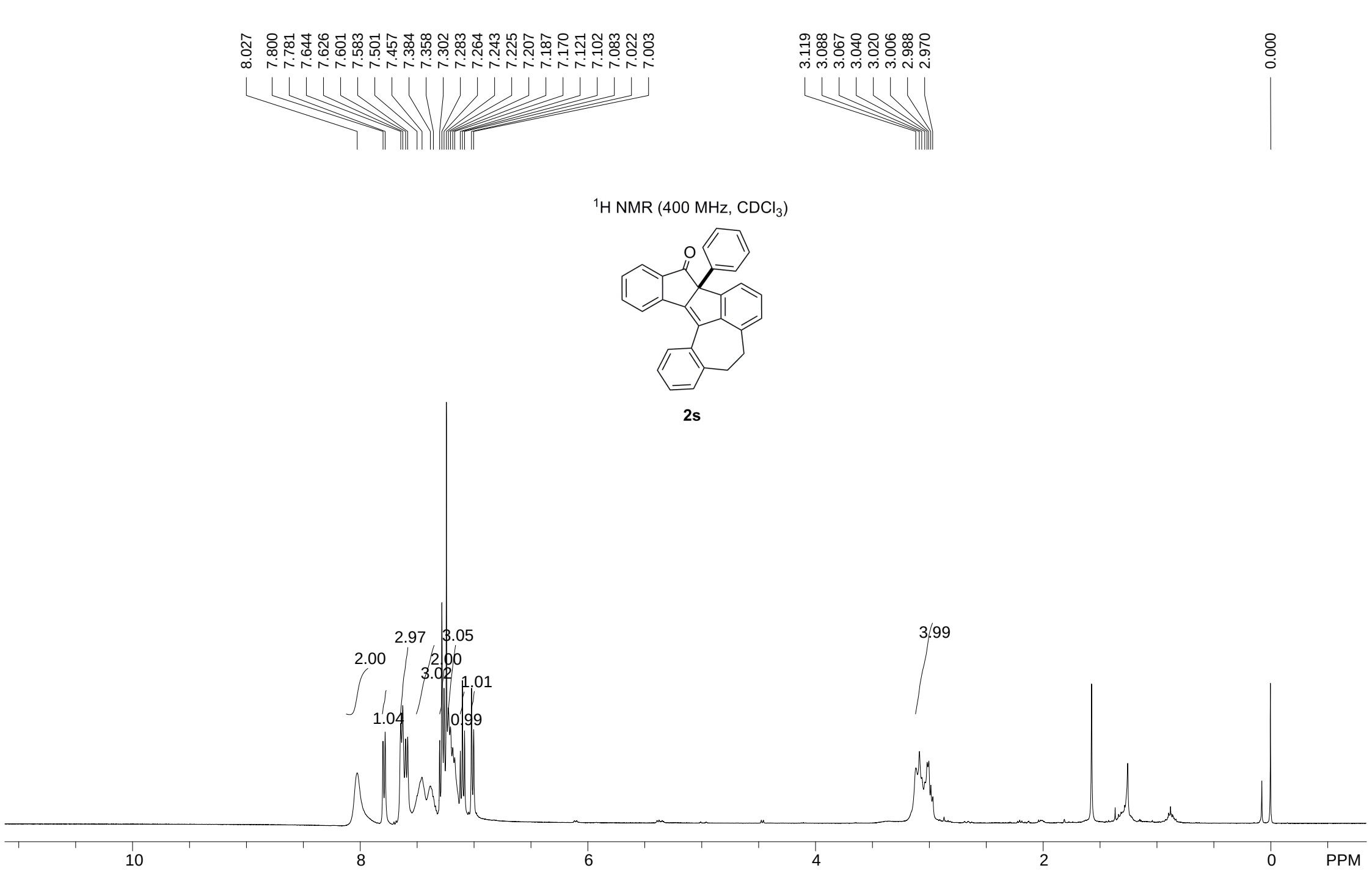
193.765
147.192
146.508
144.362
143.658
143.544
138.959
137.032
135.095
134.839
133.217
131.855
130.736
129.150
128.992
128.601
128.088
128.019
126.091
125.278
122.670
122.299
77.317
77.000
76.681
73.954

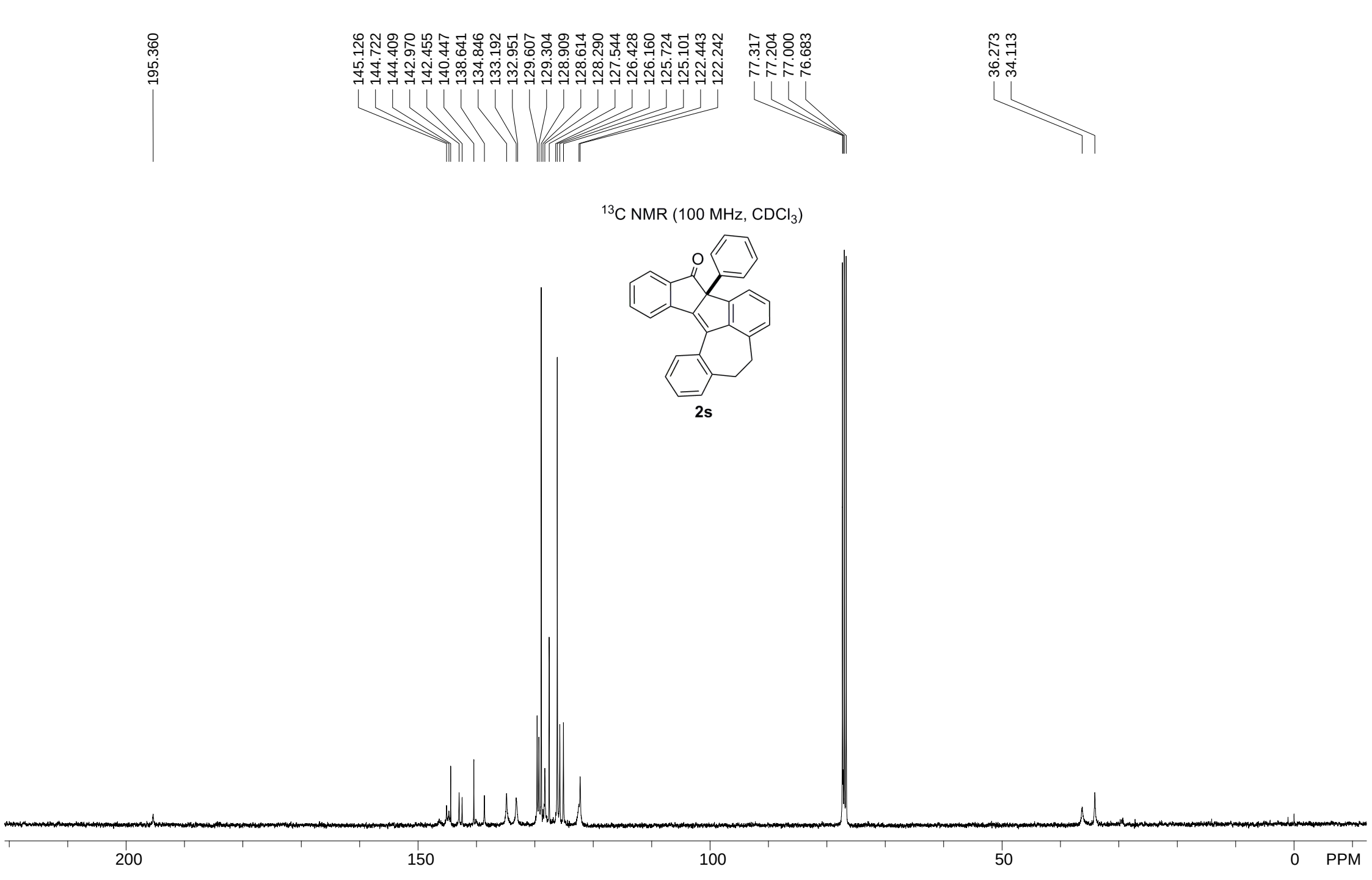
^{13}C NMR (100 MHz, $CDCl_3$)

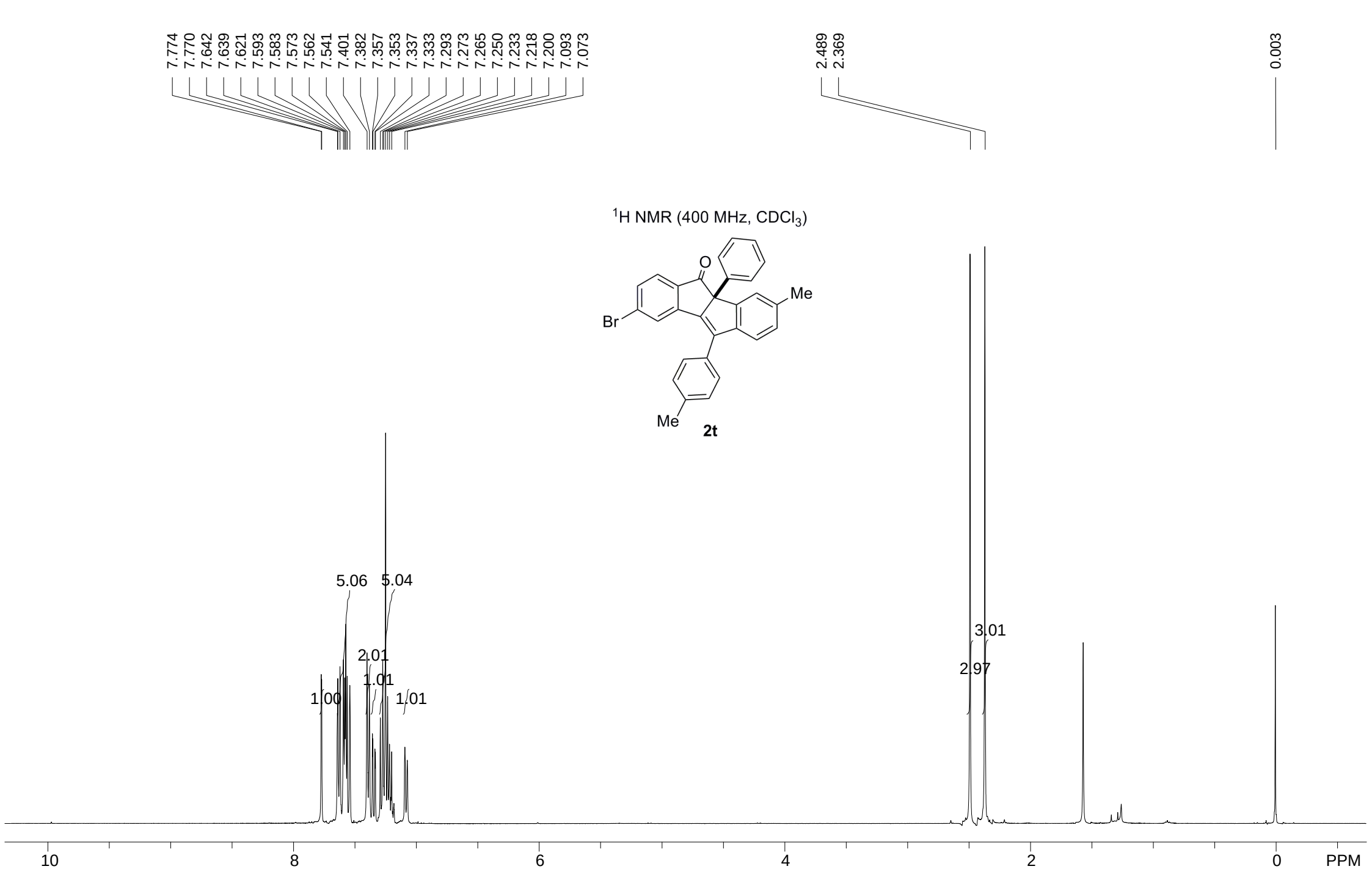


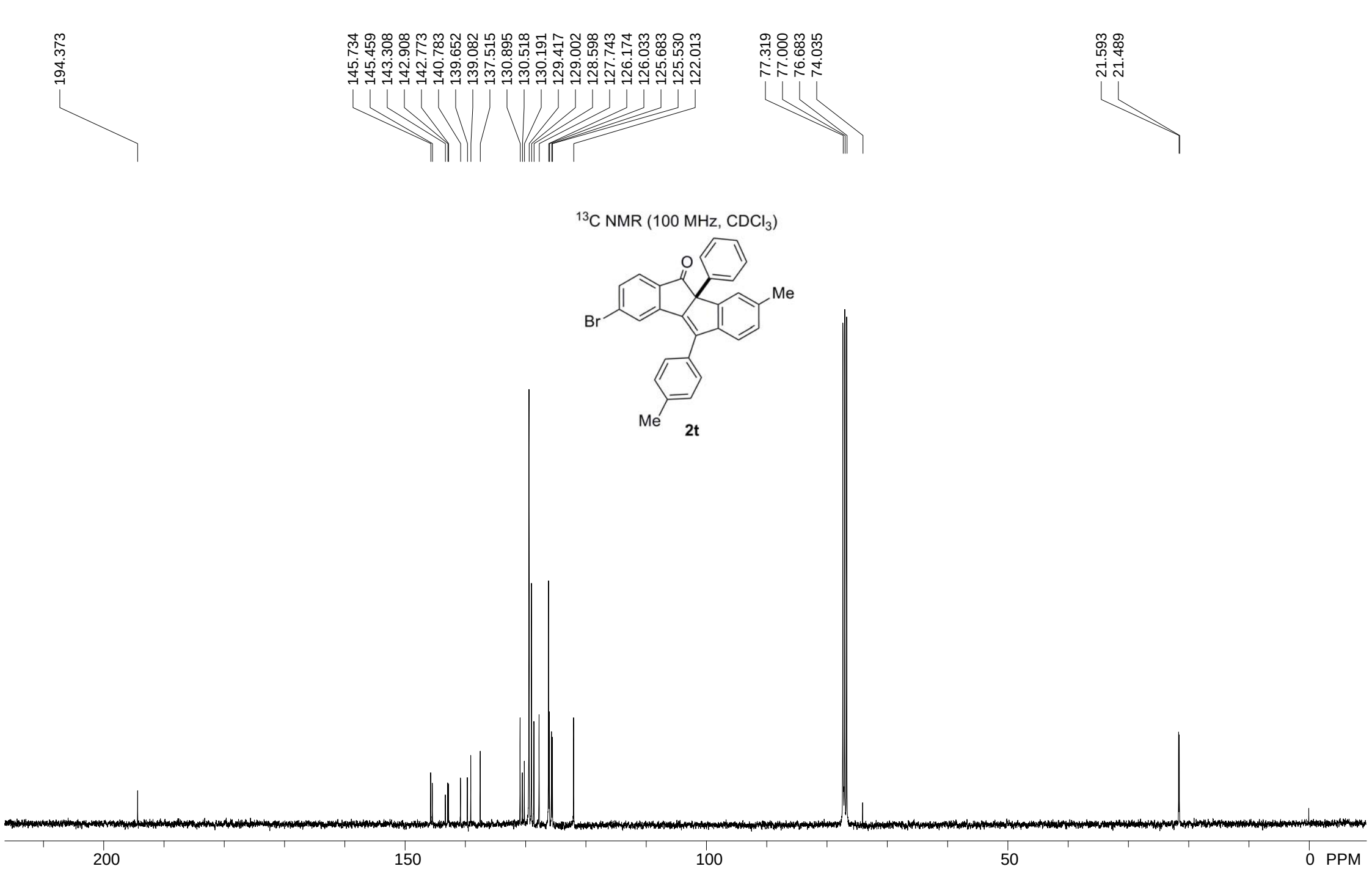
2r

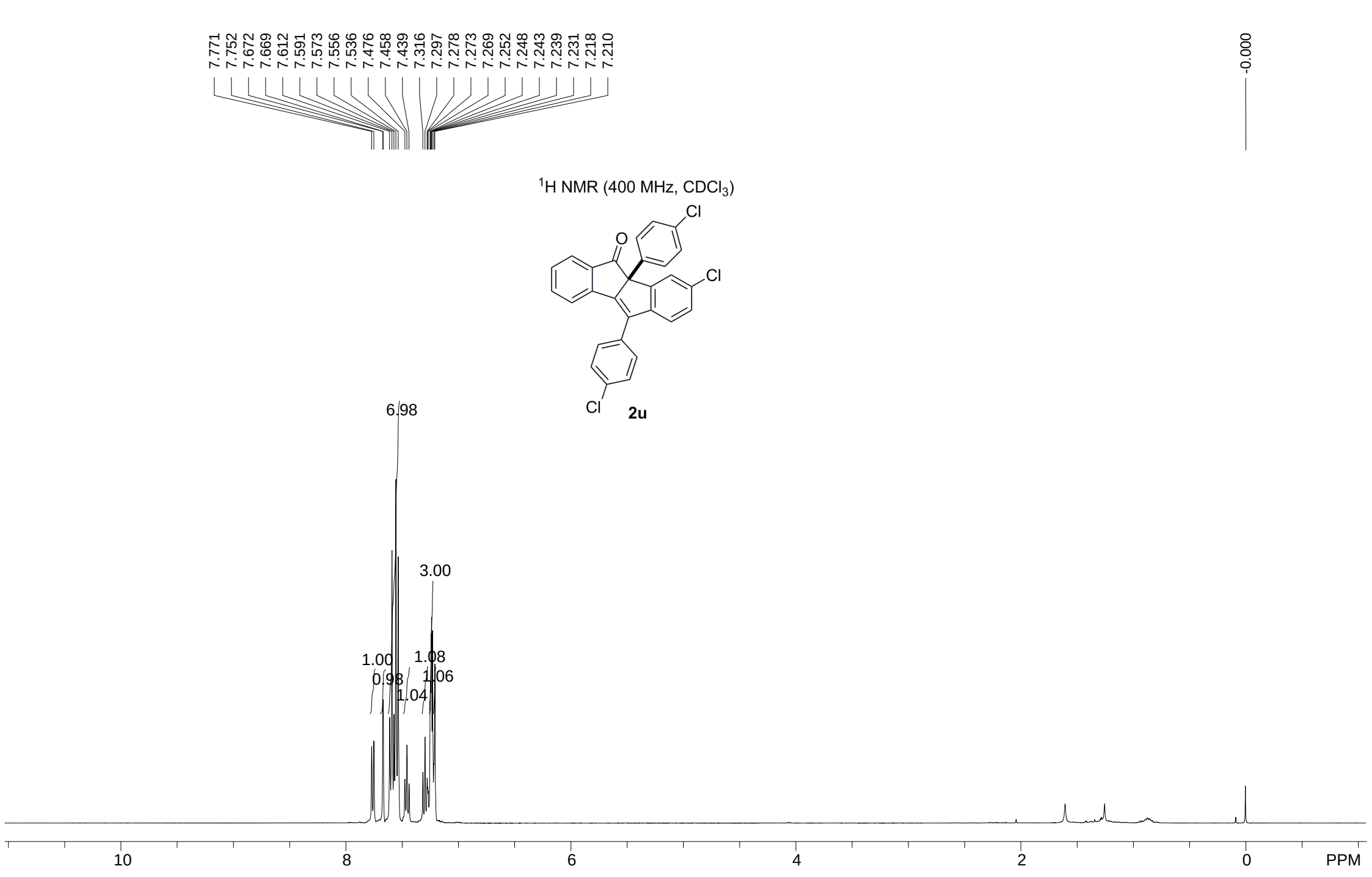



8.027
7.800
7.781
7.644
7.626
7.601
7.583
7.501
7.457
7.384
7.358
7.302
7.283
7.264
7.243
7.225
7.207
7.187
7.170
7.121
7.102
7.083
7.022
7.003
3.119
3.088
3.067
3.040
3.020
3.006
2.988
2.970
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
2s
2.00
2.97
3.05
2.00
3.02
1.01
1.04
0.99
3.99
10
8
6
4
2
0
PPM


195.360
145.126
144.722
144.409
142.970
142.455
140.447
138.641
134.846
133.192
132.951
129.607
129.304
128.909
128.614
128.290
127.544
126.428
126.160
125.724
125.101
122.443
122.242
77.317
77.204
77.000
76.683
36.273
34.113
13C NMR (100 MHz, CDCl3)
O
2s
200
150
100
50
0
PPM

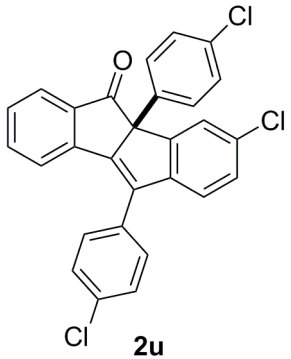

7.774
7.770
7.642
7.639
7.621
7.593
7.583
7.573
7.562
7.541
7.401
7.382
7.357
7.353
7.337
7.333
7.293
7.273
7.265
7.250
7.233
7.218
7.200
7.093
7.073
2.489
2.369
0.003
^{1}H NMR (400 MHz, CDCl$_3$)
O
Br
Me
Me
2t
5.06
5.04
2.01
1.01
1.00
1.01
3.01
2.97
10
8
6
4
2
0
PPM


194.373
145.734
145.459
143.308
142.908
142.773
140.783
139.652
139.082
137.515
130.895
130.518
130.191
129.417
129.002
128.598
127.743
126.174
126.033
125.683
125.530
122.013
77.319
77.000
76.683
74.035
21.593
21.489
13C NMR (100 MHz, CDCl3)
O
Br
Me
Me
2t
200
150
100
50
0
PPM

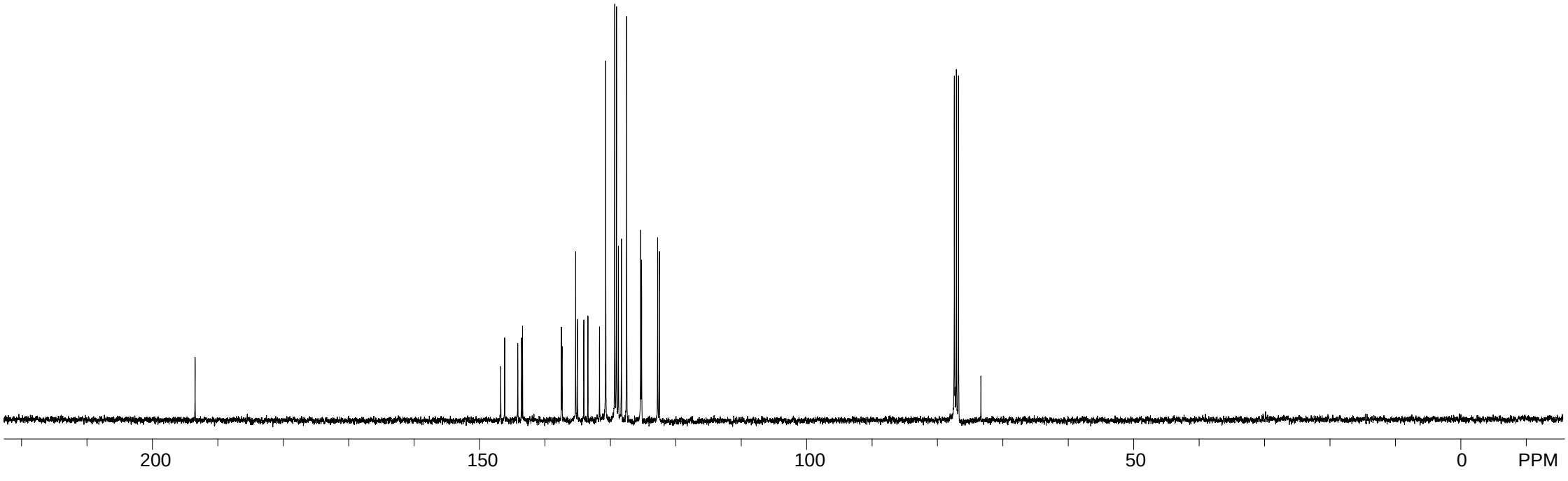

7.771
7.752
7.672
7.669
7.612
7.591
7.573
7.556
7.536
7.476
7.458
7.439
7.316
7.297
7.278
7.273
7.269
7.252
7.248
7.243
7.239
7.231
7.218
7.210
-0.000
1H NMR (400 MHz, CDCl3)
Cl
O
Cl
Cl
2u
6.98
3.00
1.00
0.98
1.04
1.08
1.06
10
8
6
4
2
0
PPM

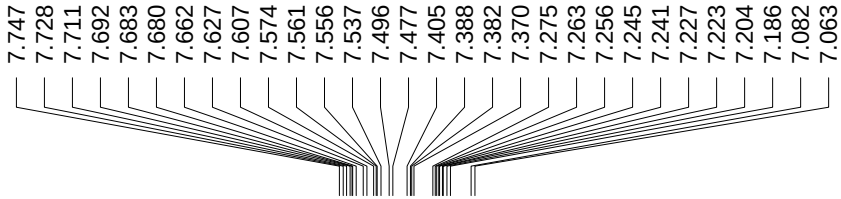
193.470
146.758
146.154
144.148
143.564
143.411
137.467
137.368
135.287
134.980
134.039
133.411
131.636
130.689
129.305
129.034
128.743
128.281
127.479
125.333
125.200
122.740
122.478
77.319
77.000
76.683
73.250

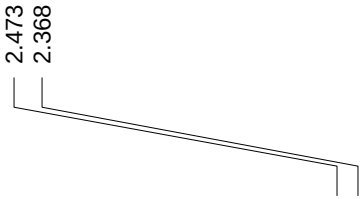
^{13}C NMR (100 MHz, $CDCl_3$)



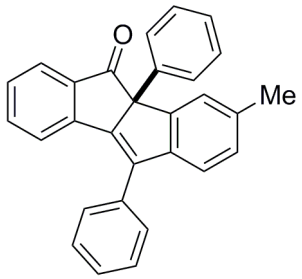
2u



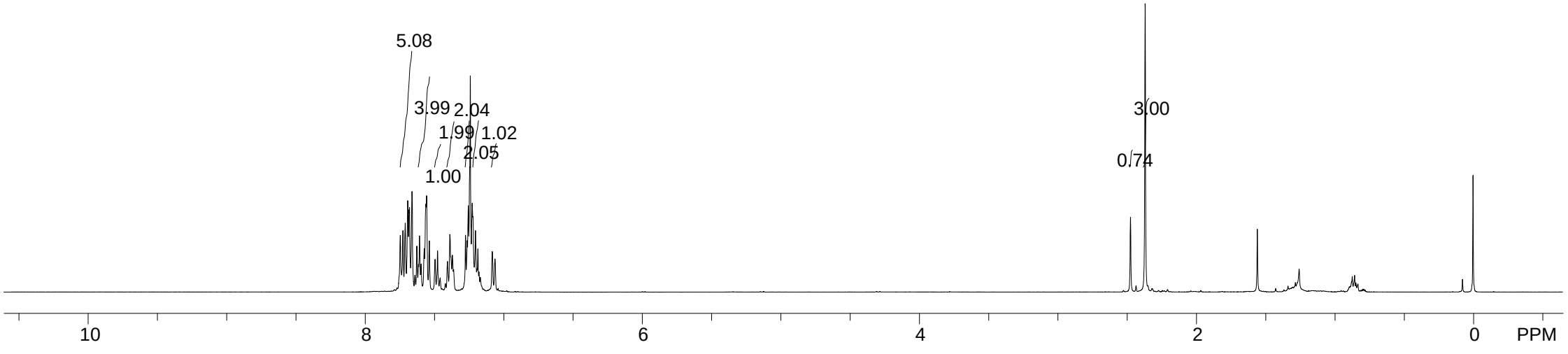


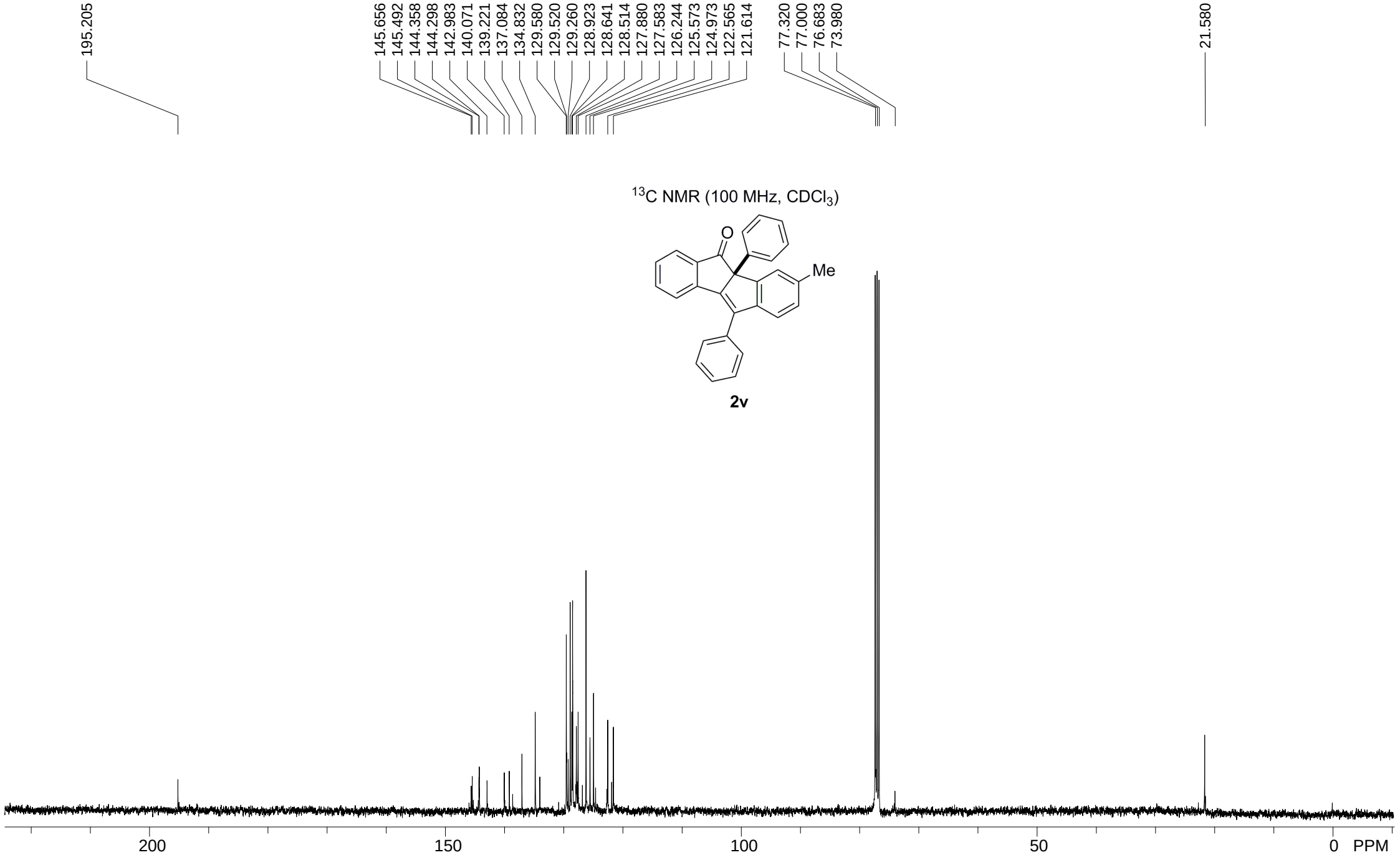


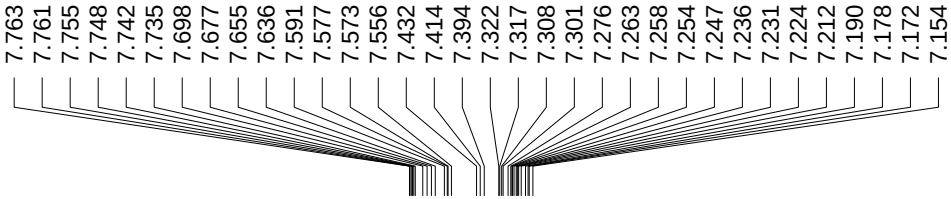
^{1}H NMR (400 MHz, $CDCl_3$)



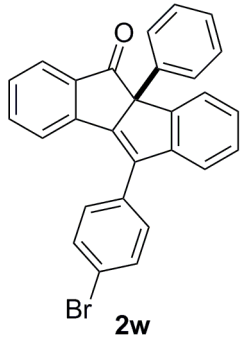
2v



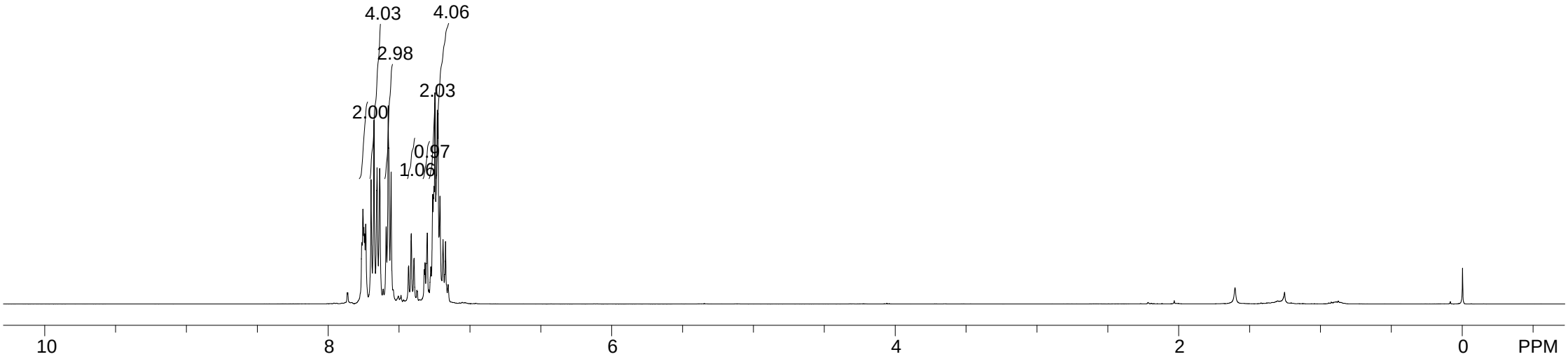

195.205
145.656
145.492
144.358
144.298
142.983
140.071
139.221
137.084
134.832
129.580
129.520
129.260
128.923
128.641
128.514
127.880
127.583
126.244
125.573
124.973
122.565
121.614
77.320
77.000
76.683
73.980
21.580
13C NMR (100 MHz, CDCl3)
O
Me
2v
200
150
100
50
0
PPM



^{1}H NMR (400 MHz, $CDCl_3$)



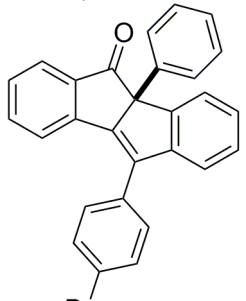
2w



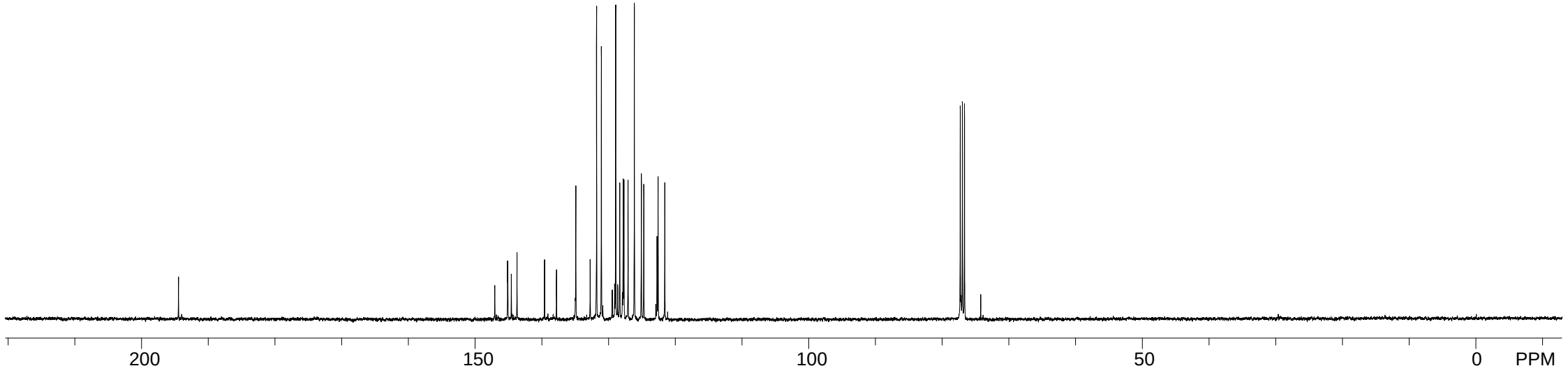
194.462

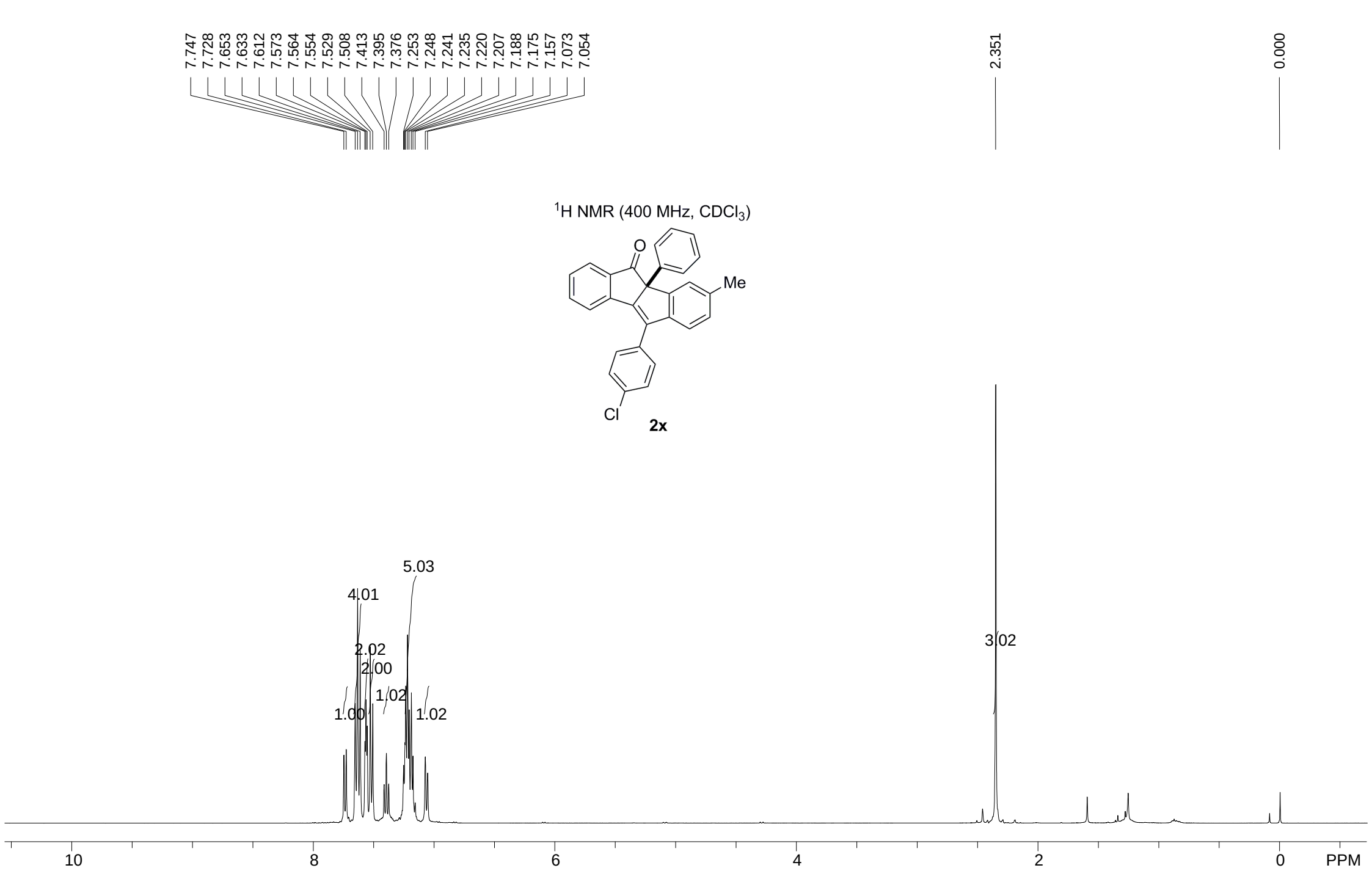
147.070
145.154
145.110
144.590
143.737
139.611
137.819
134.924
132.775
131.809
131.097
129.466
129.109
128.950
128.655
128.340
127.944
127.829
127.712
127.096
126.158
125.105
124.738
122.757
122.595
121.592
77.320
77.000
76.683
74.254

^{13}C NMR (100 MHz, $CDCl_3$)



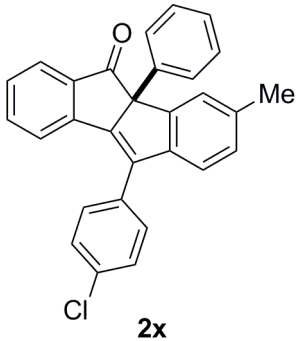
2w



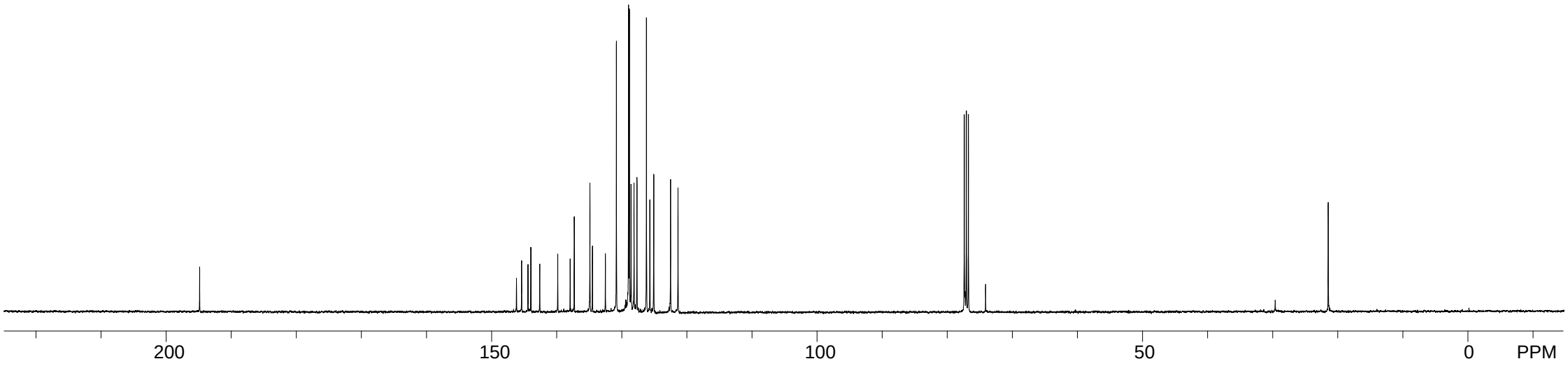

7.747
7.728
7.653
7.633
7.612
7.573
7.564
7.554
7.529
7.508
7.413
7.395
7.376
7.253
7.248
7.241
7.235
7.220
7.207
7.188
7.175
7.157
7.073
7.054
2.351
0.000
1H NMR (400 MHz, CDCl3)
O
Me
Cl
2x
5.03
4.01
2.02
2.00
1.02
1.00
1.02
3.02
10
8
6
4
2
0
PPM

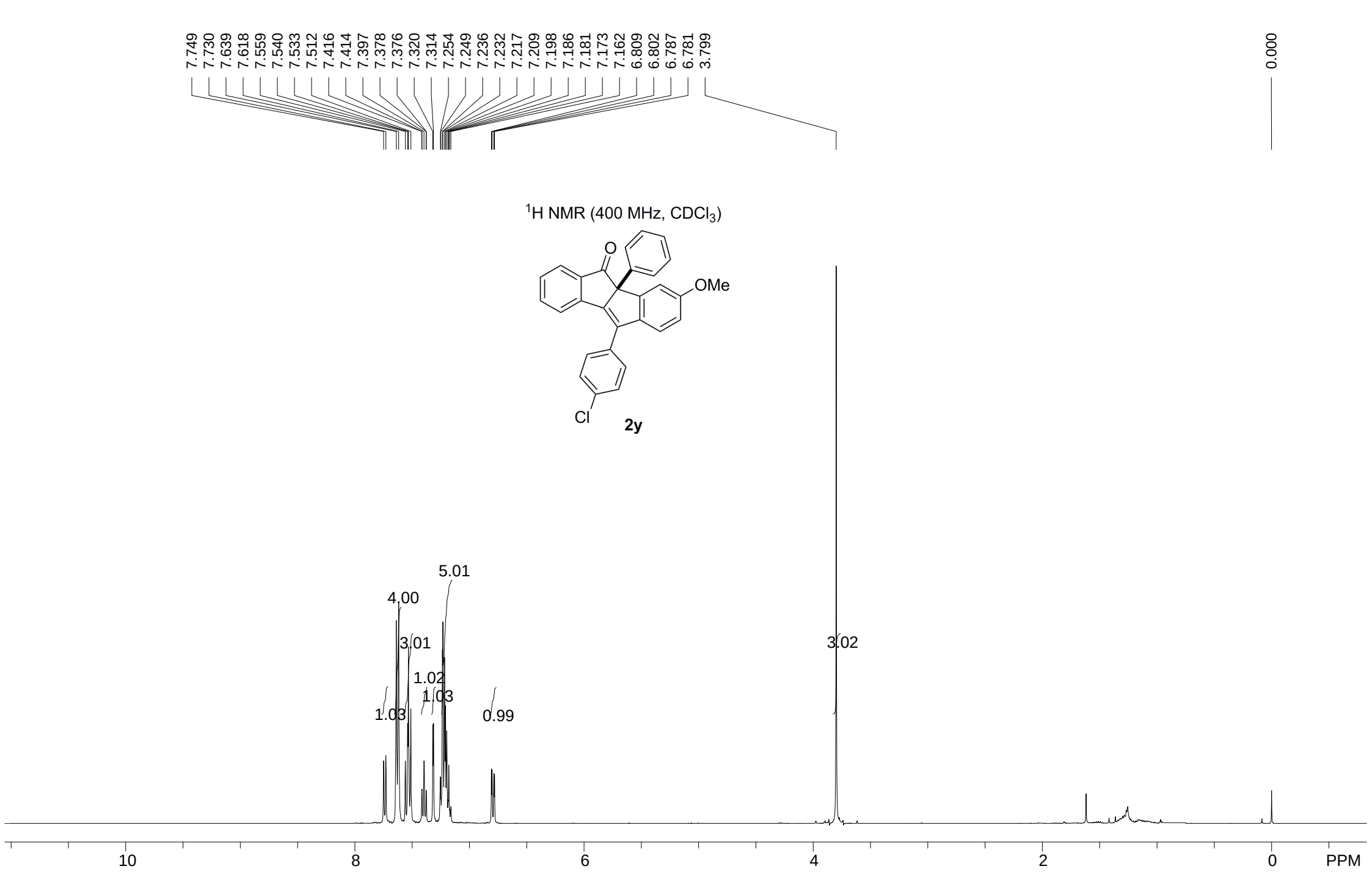
194.847
146.180
145.386
144.409
143.965
142.600
139.830
137.917
137.289
134.880
134.488
132.502
130.807
128.939
128.809
128.552
128.091
127.643
126.183
125.656
125.057
122.470
121.325
77.318
77.000
76.682
74.049
21.551

^{13}C NMR (100 MHz, $CDCl_3$)



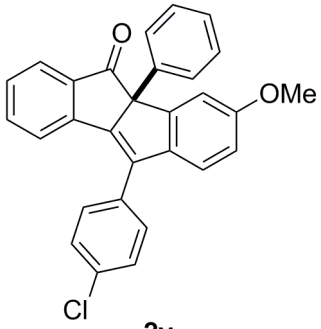
2x



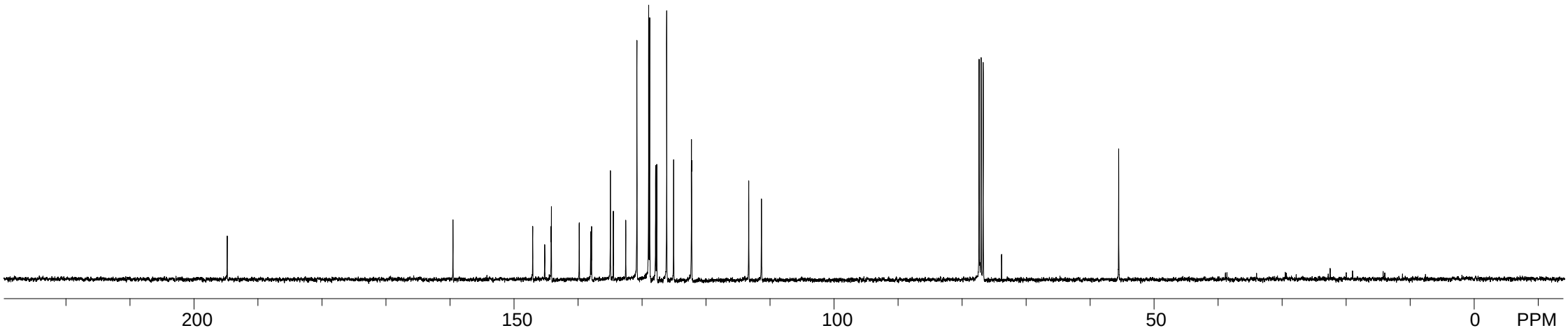

7.749
7.730
7.639
7.618
7.559
7.540
7.533
7.512
7.416
7.414
7.397
7.378
7.376
7.320
7.314
7.254
7.249
7.236
7.232
7.217
7.209
7.198
7.186
7.181
7.173
7.162
6.809
6.802
6.787
6.781
3.799
0.000
1H NMR (400 MHz, CDCl3)
O
OMe
Cl
2y
5.01
4.00
3.01
1.02
1.03
1.03
0.99
3.02
10
8
6
4
2
0
PPM

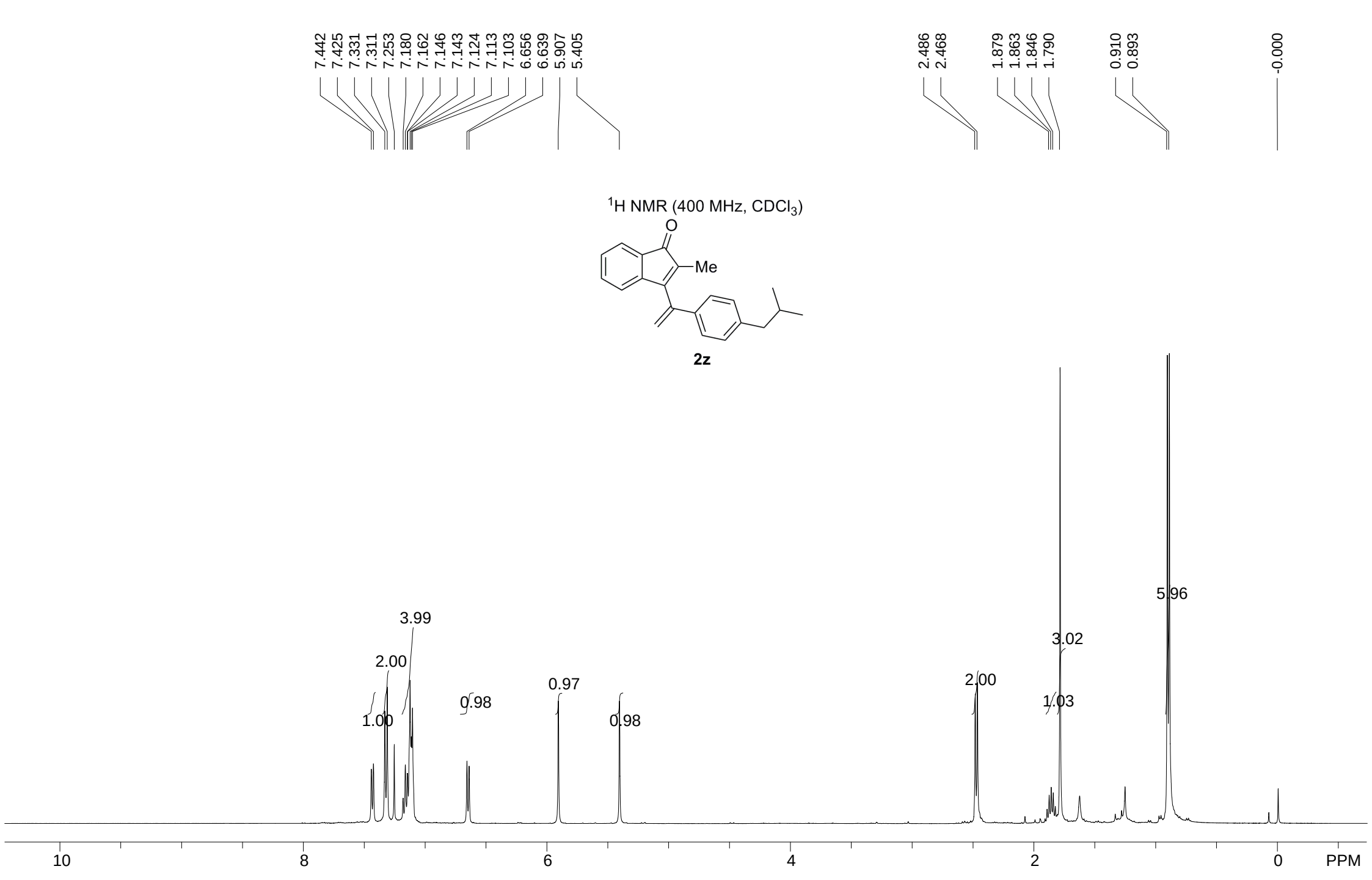
194.800
159.566
147.099
145.220
144.223
144.174
139.818
138.037
137.892
134.943
134.492
132.546
130.797
128.965
128.808
127.877
127.703
126.141
125.058
122.272
122.236
113.313
111.320
77.317
77.000
76.683
73.819
55.581

^{13}C NMR (100 MHz, $CDCl_3$)



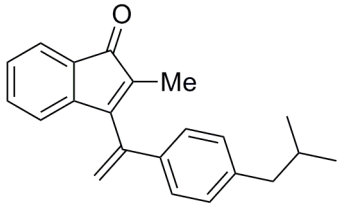
2y



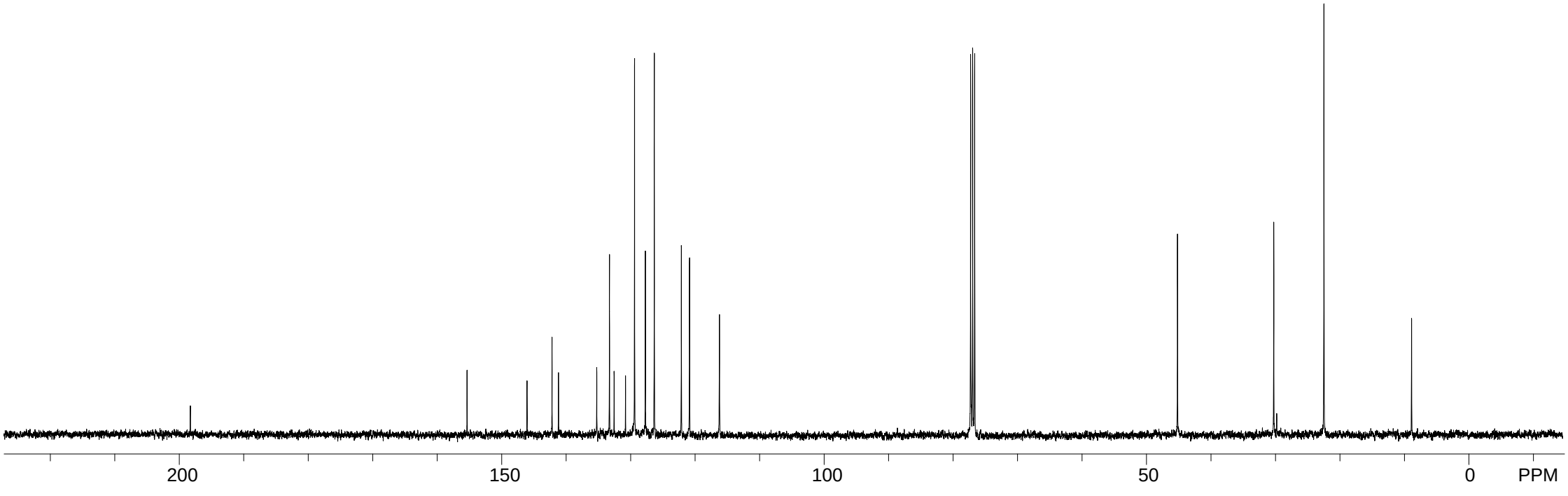

7.442
7.425
7.331
7.311
7.253
7.180
7.162
7.146
7.143
7.124
7.113
7.103
6.656
6.639
5.907
5.405
2.486
2.468
1.879
1.863
1.846
1.790
0.910
0.893
-0.000
1H NMR (400 MHz, CDCl3)
O
Me
2z
3.99
2.00
1.00
0.98
0.97
0.98
2.00
3.02
1.03
5.96
10
8
6
4
2
0
PPM

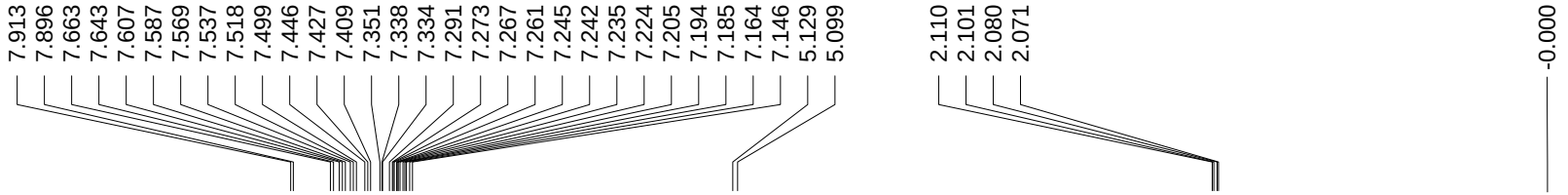
198.277
155.387
146.091
142.218
141.220
135.286
133.316
132.596
130.828
129.433
127.752
126.371
122.176
120.904
116.271
77.318
77.000
76.682
45.117
30.140
22.334
8.695

^{13}C NMR (100 MHz, $CDCl_3$)

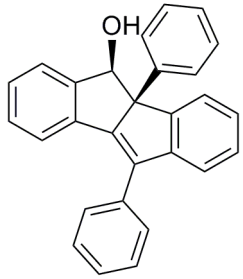


2z

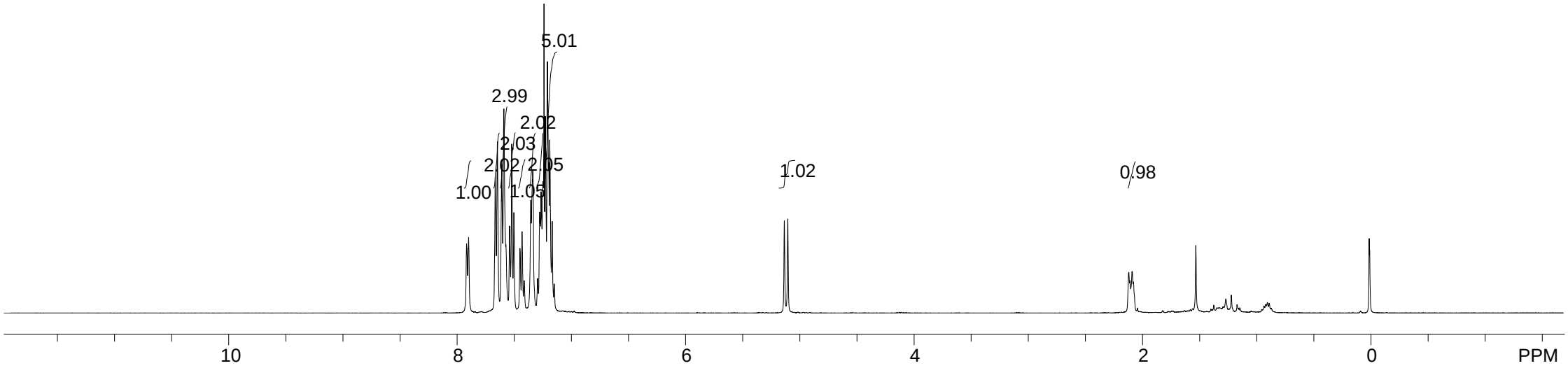


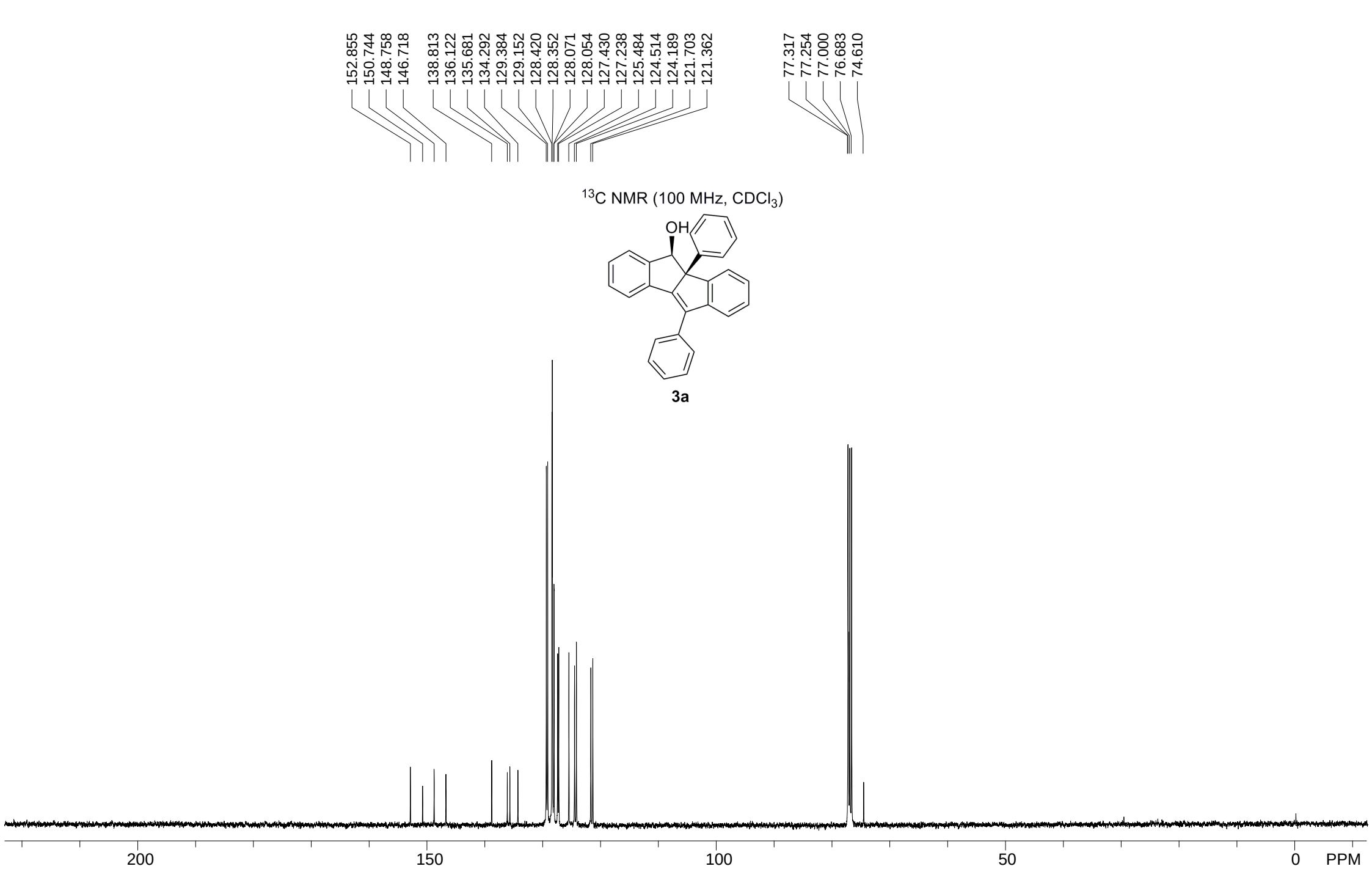


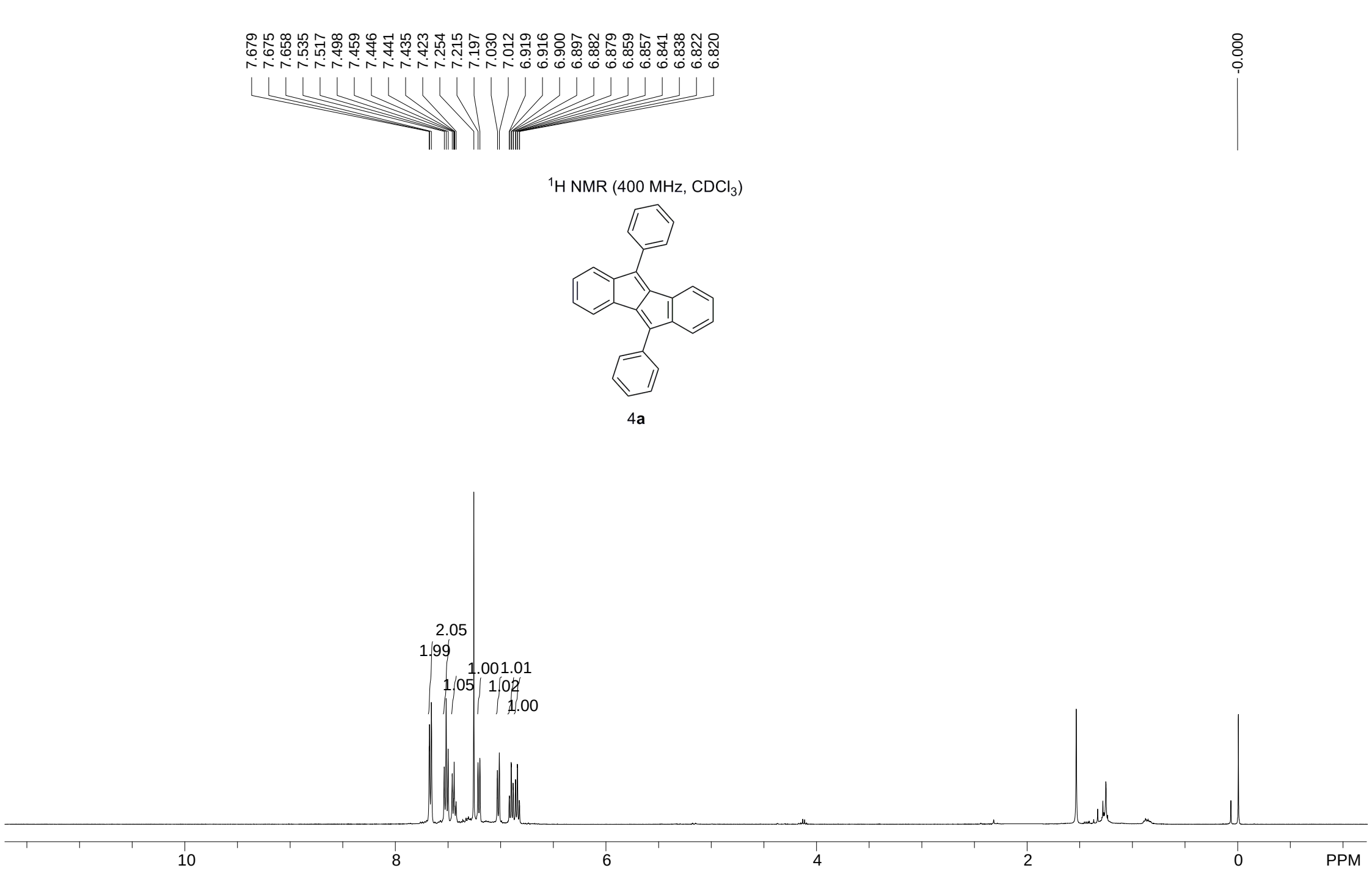
^{1}H NMR (400 MHz, $CDCl_3$)

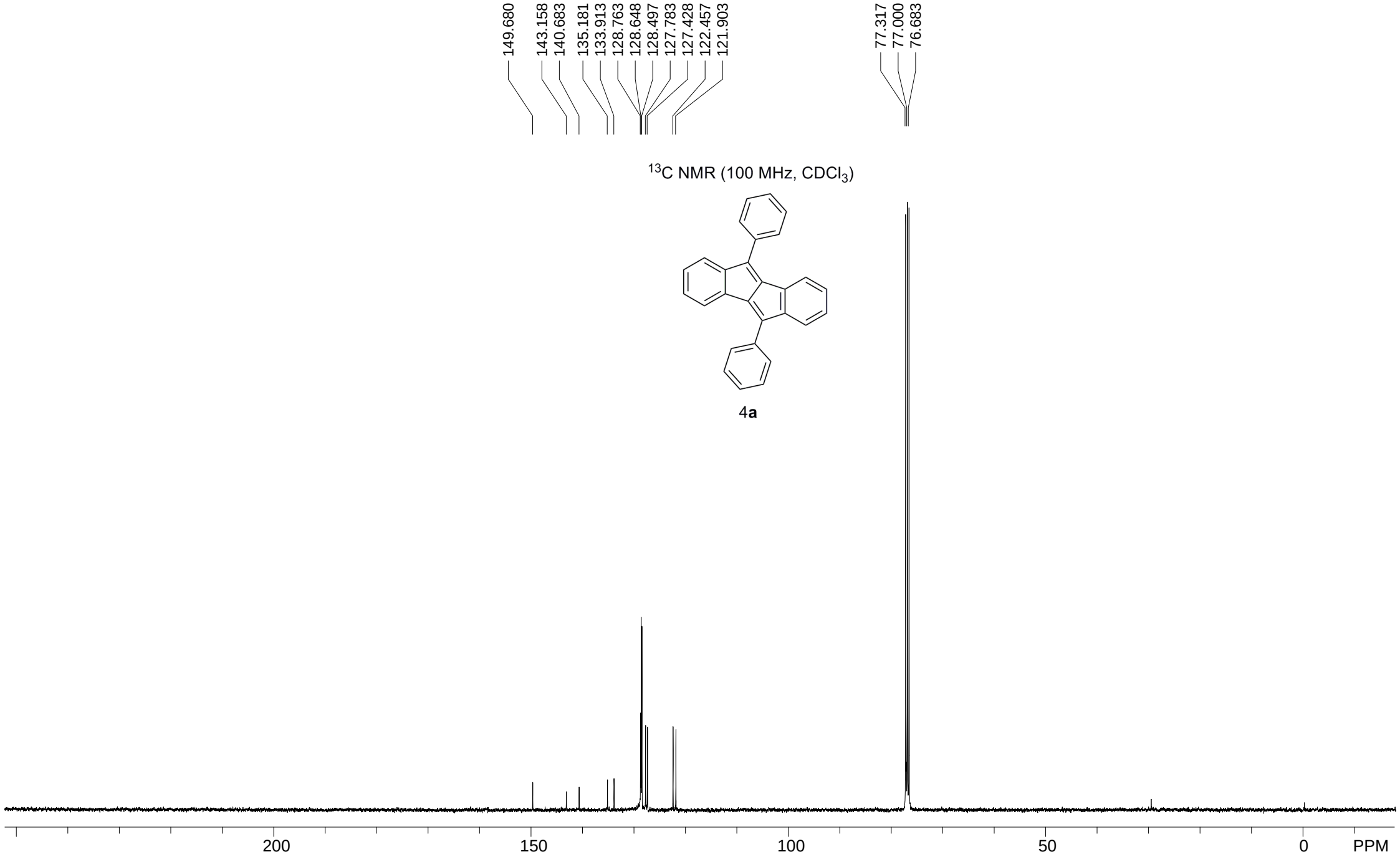


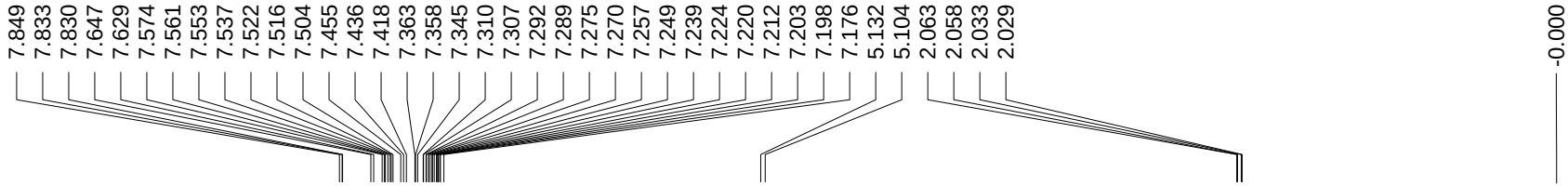
3a



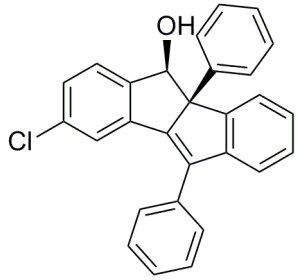

152.855
150.744
148.758
146.718
138.813
136.122
135.681
134.292
129.384
129.152
128.420
128.352
128.071
128.054
127.430
127.238
125.484
124.514
124.189
121.703
121.362
77.317
77.254
77.000
76.683
74.610
13C NMR (100 MHz, CDCl3)
OH
3a
200
150
100
50
0
PPM


7.679
7.675
7.658
7.535
7.517
7.498
7.459
7.446
7.441
7.435
7.423
7.254
7.215
7.197
7.030
7.012
6.919
6.916
6.900
6.897
6.882
6.879
6.859
6.857
6.841
6.838
6.822
6.820
-0.000
^{1}H NMR (400 MHz, $CDCl_3$)
4**a**
2.05
1.99
1.05
1.00
1.01
1.02
1.00
10
8
6
4
2
0
PPM

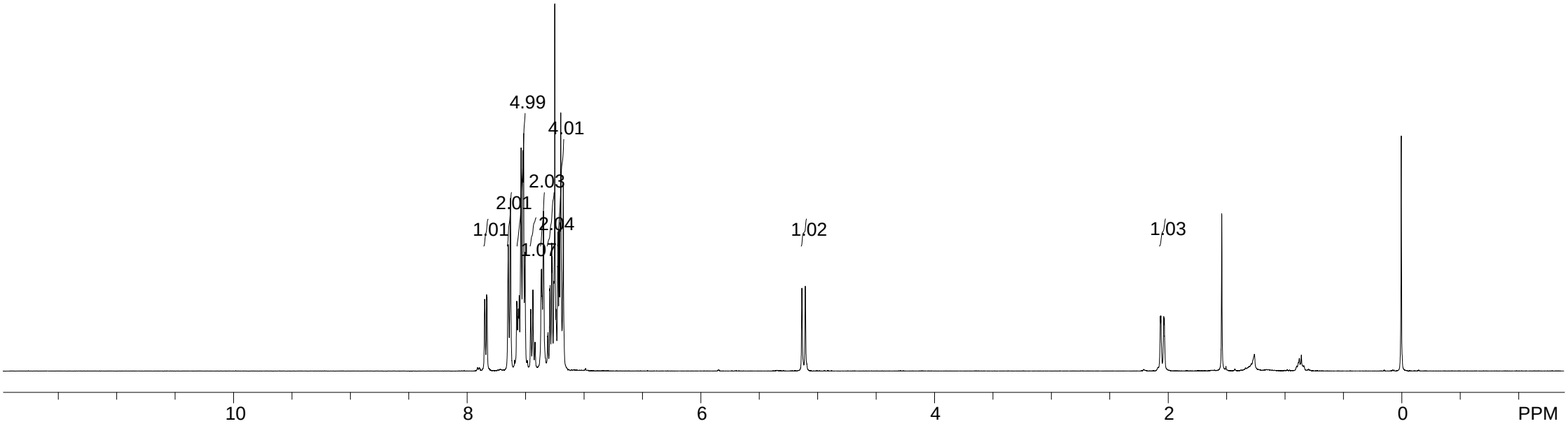

149.680
143.158
140.683
135.181
133.913
128.763
128.648
128.497
127.783
127.428
122.457
121.903
77.317
77.000
76.683
13C NMR (100 MHz, CDCl3)
4a
200
150
100
50
0
PPM

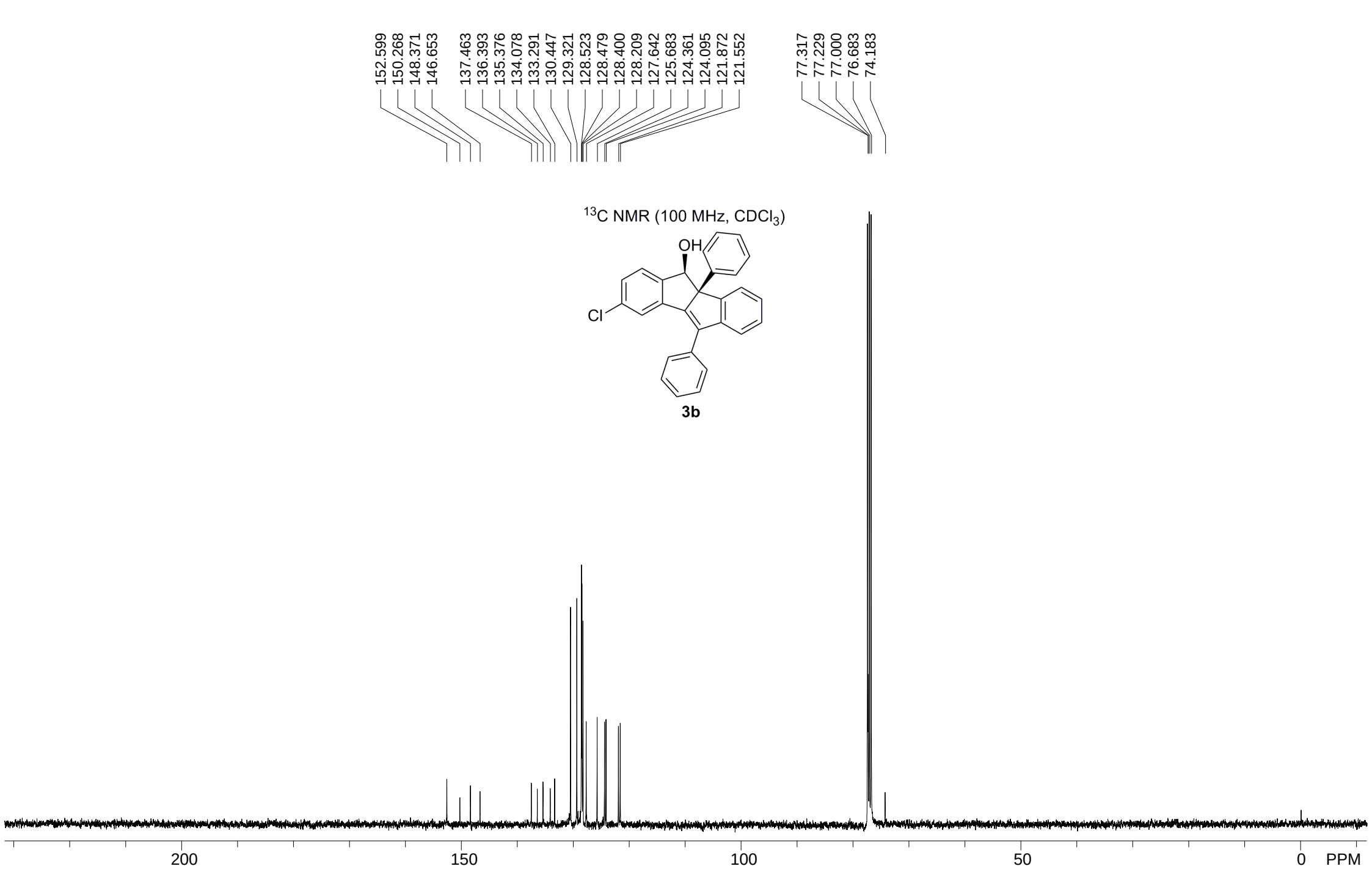


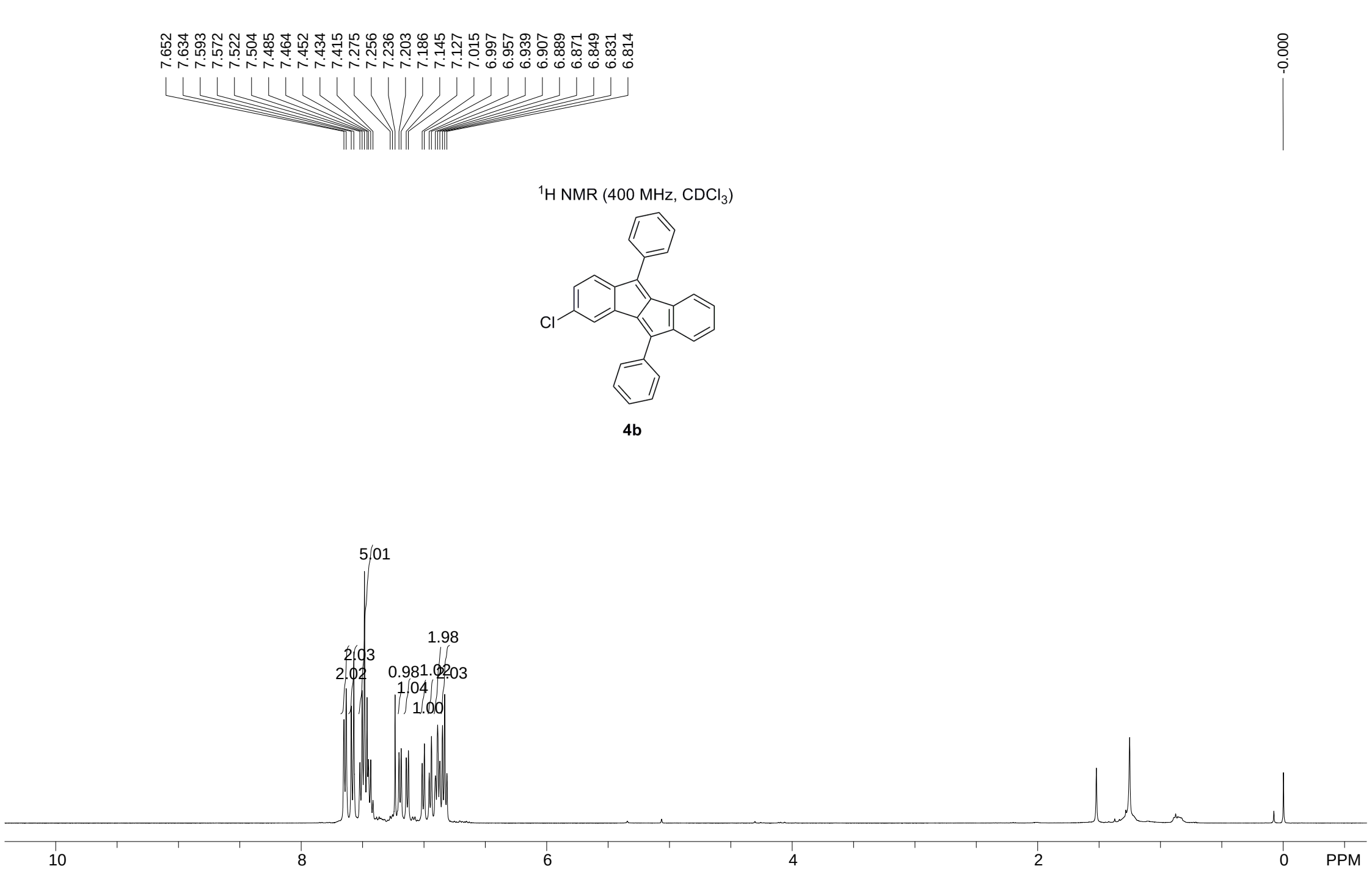
^{1}H NMR (400 MHz, $CDCl_3$)

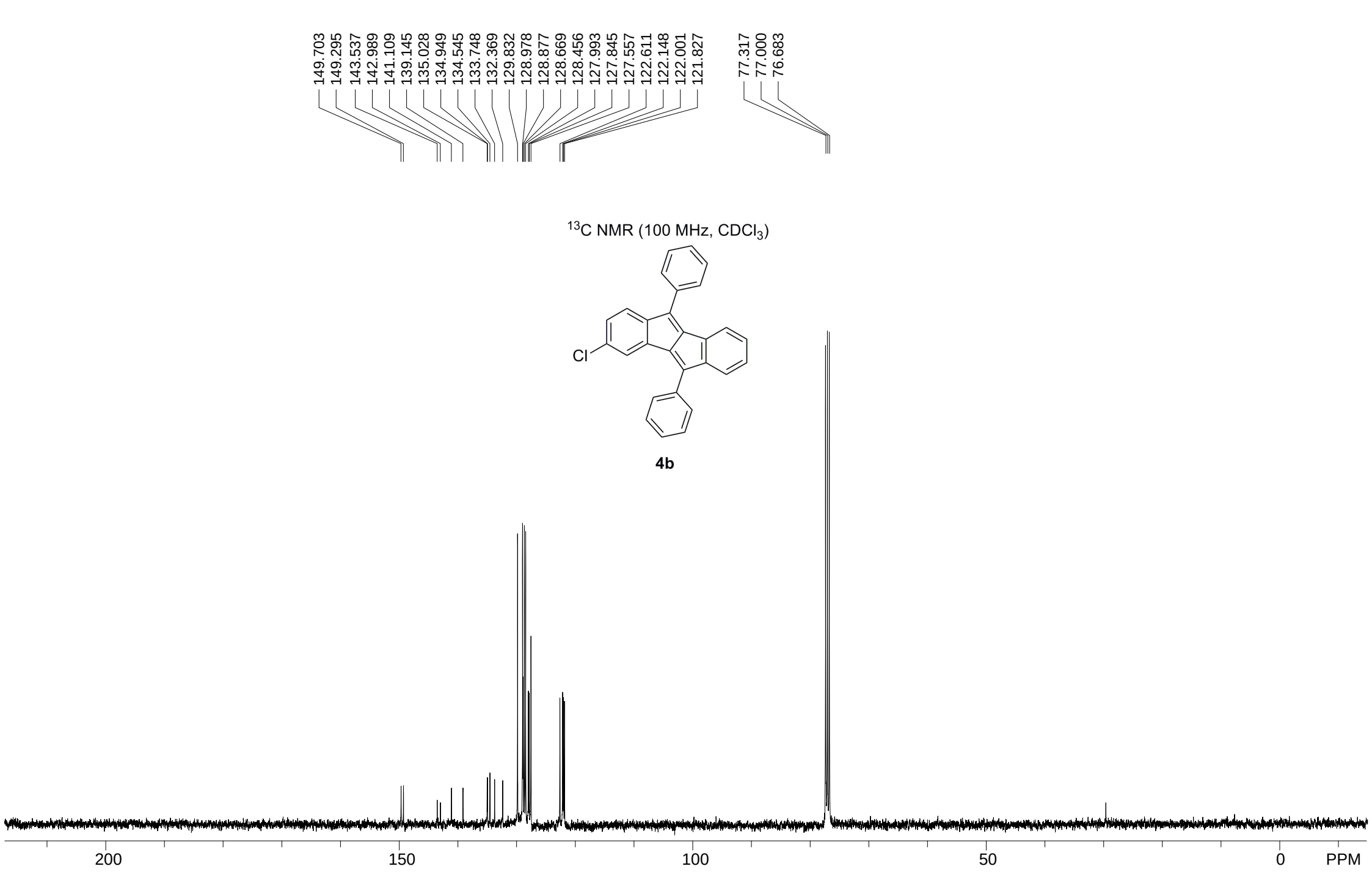


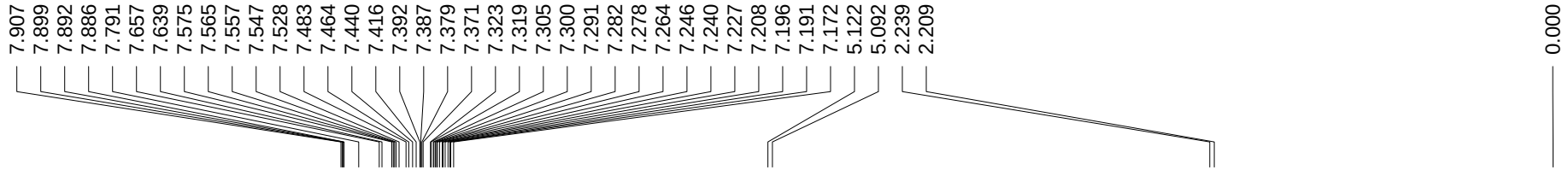
3b



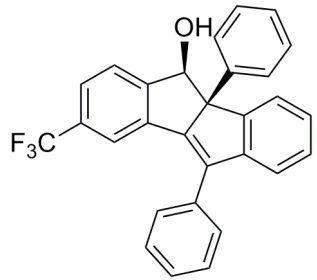

152.599
150.268
148.371
146.653
137.463
136.393
135.376
134.078
133.291
130.447
129.321
128.523
128.479
128.400
128.209
127.642
125.683
124.361
124.095
121.872
121.552
77.317
77.229
77.000
76.683
74.183
13C NMR (100 MHz, CDCl3)
OH
Cl
3b
200
150
100
50
0
PPM


7.652
7.634
7.593
7.572
7.522
7.504
7.485
7.464
7.452
7.434
7.415
7.275
7.256
7.236
7.203
7.186
7.145
7.127
7.015
6.997
6.957
6.939
6.907
6.889
6.871
6.849
6.831
6.814
-0.000
1H NMR (400 MHz, CDCl3)
Cl
4b
5.01
2.03
2.02
0.98
1.04
1.02
1.00
1.98
2.03
10
8
6
4
2
0
PPM

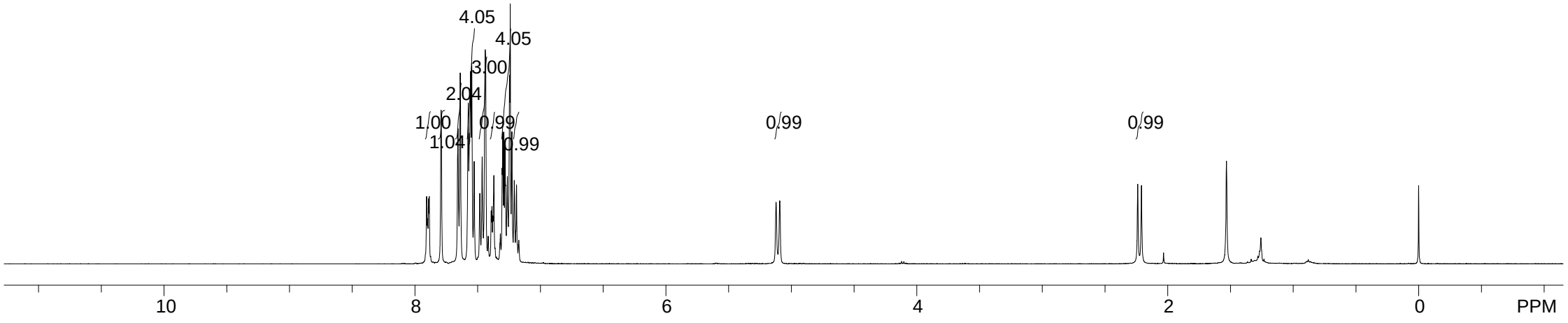

149.703
149.295
143.537
142.989
141.109
139.145
135.028
134.949
134.545
133.748
132.369
129.832
128.978
128.877
128.669
128.456
127.993
127.845
127.557
122.611
122.148
122.001
121.827
77.317
77.000
76.683
^{13}C NMR (100 MHz, $CDCl_3$)
Cl
4b
200
150
100
50
0
PPM

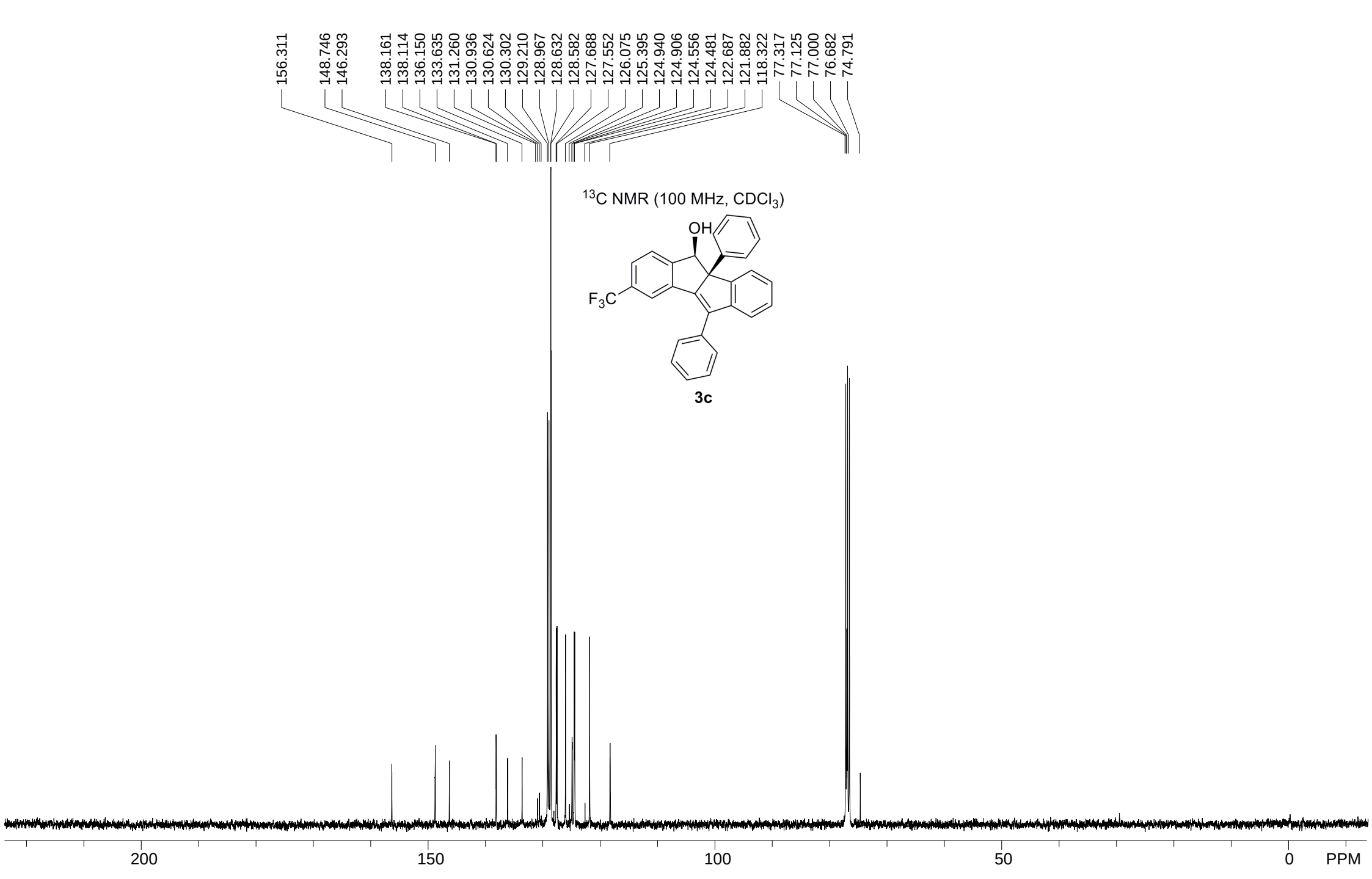


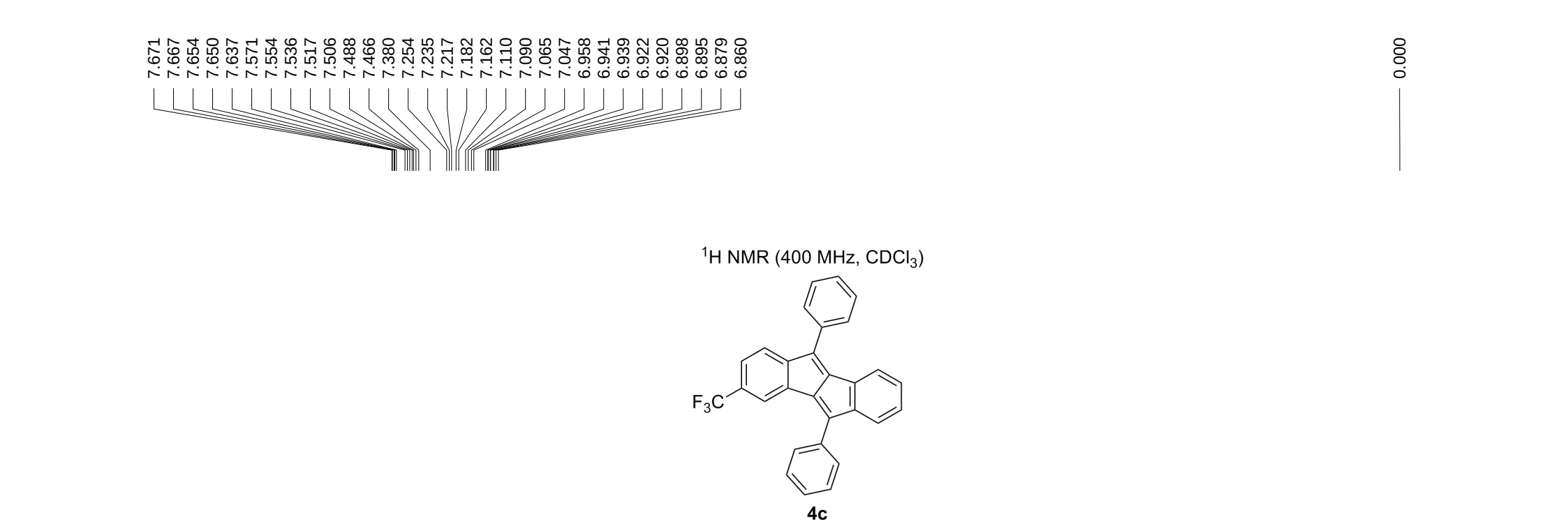
^{1}H NMR (400 MHz, $CDCl_3$)

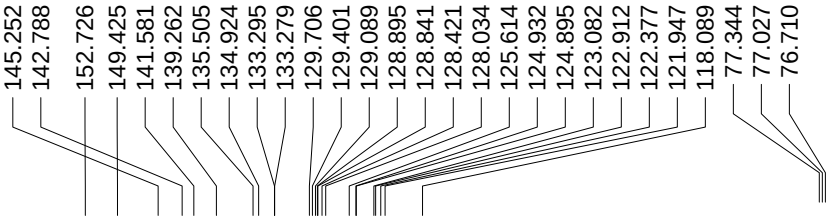


3c

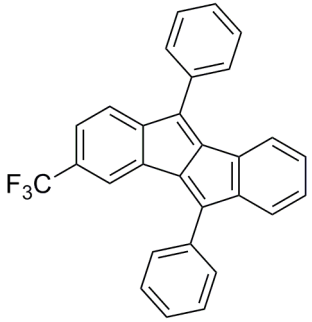



156.311
148.746
146.293
138.161
138.114
136.150
133.635
131.260
130.936
130.624
130.302
129.210
128.967
128.632
128.582
127.688
127.552
126.075
125.395
124.940
124.906
124.556
124.481
122.687
121.882
118.322
77.317
77.125
77.000
76.682
74.791
13C NMR (100 MHz, CDCl3)
OH
F3C
3c
200
150
100
50
0
PPM

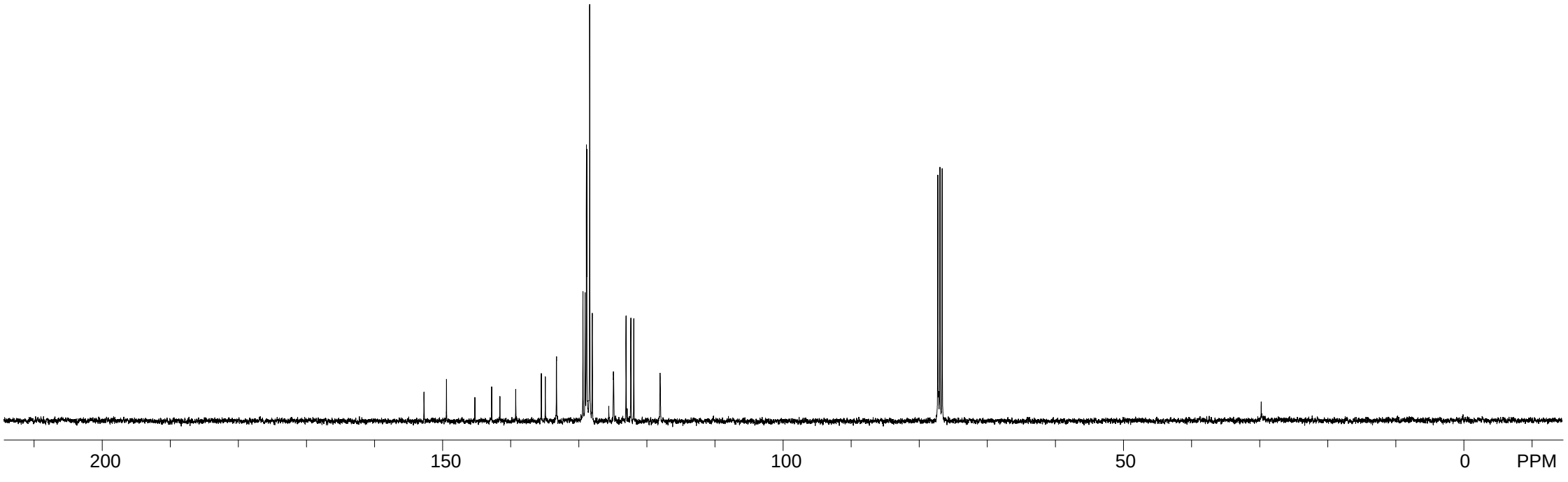

7.671
7.667
7.654
7.650
7.637
7.571
7.554
7.536
7.517
7.506
7.488
7.466
7.380
7.254
7.235
7.217
7.182
7.162
7.110
7.090
7.065
7.047
6.958
6.941
6.939
6.922
6.920
6.898
6.895
6.879
6.860
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
F_3C
4c
5.99
4.00
2.02
1.00
1.02
1.00
1.02
1.01
10
8
6
4
2
0
PPM

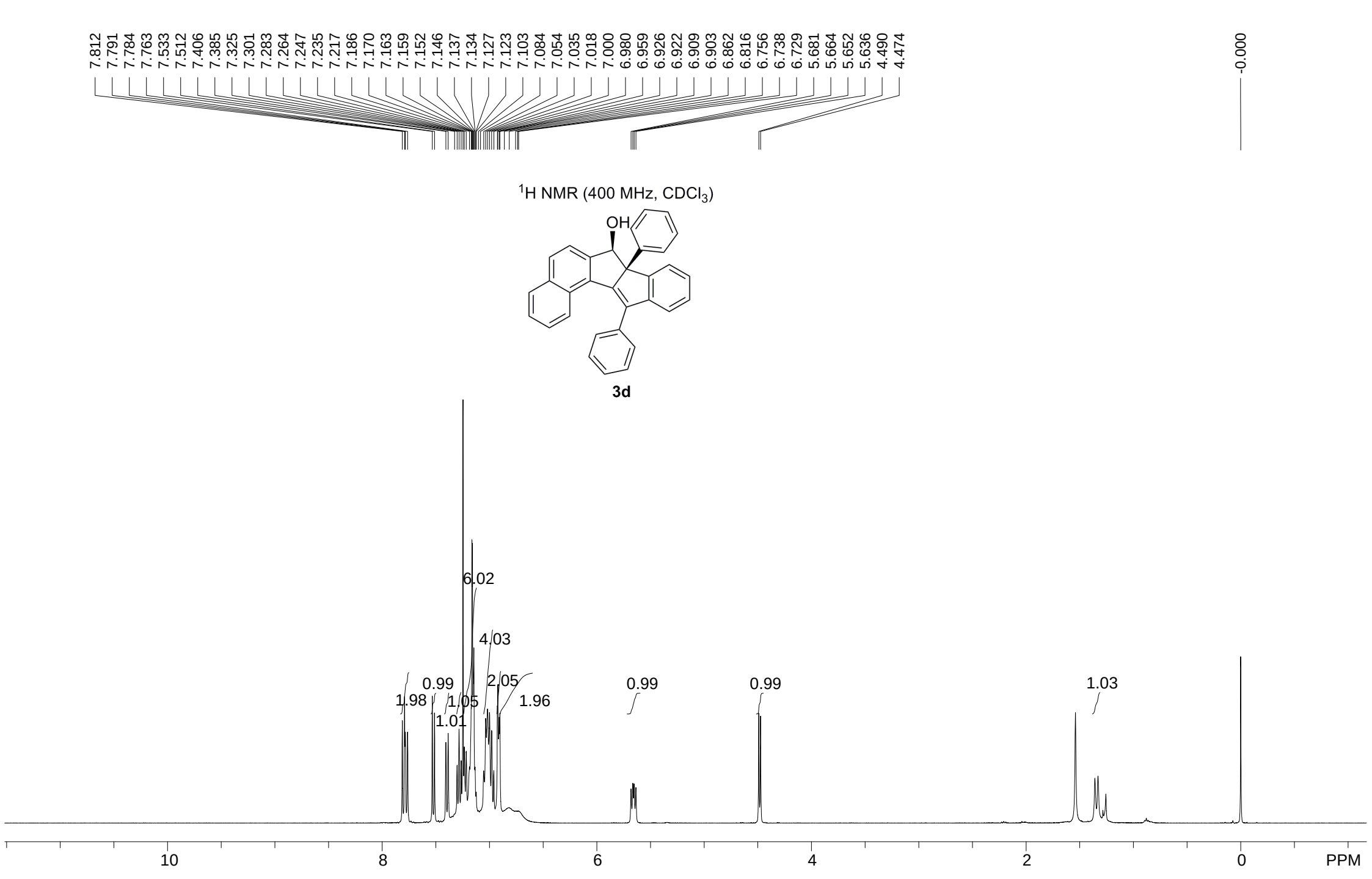


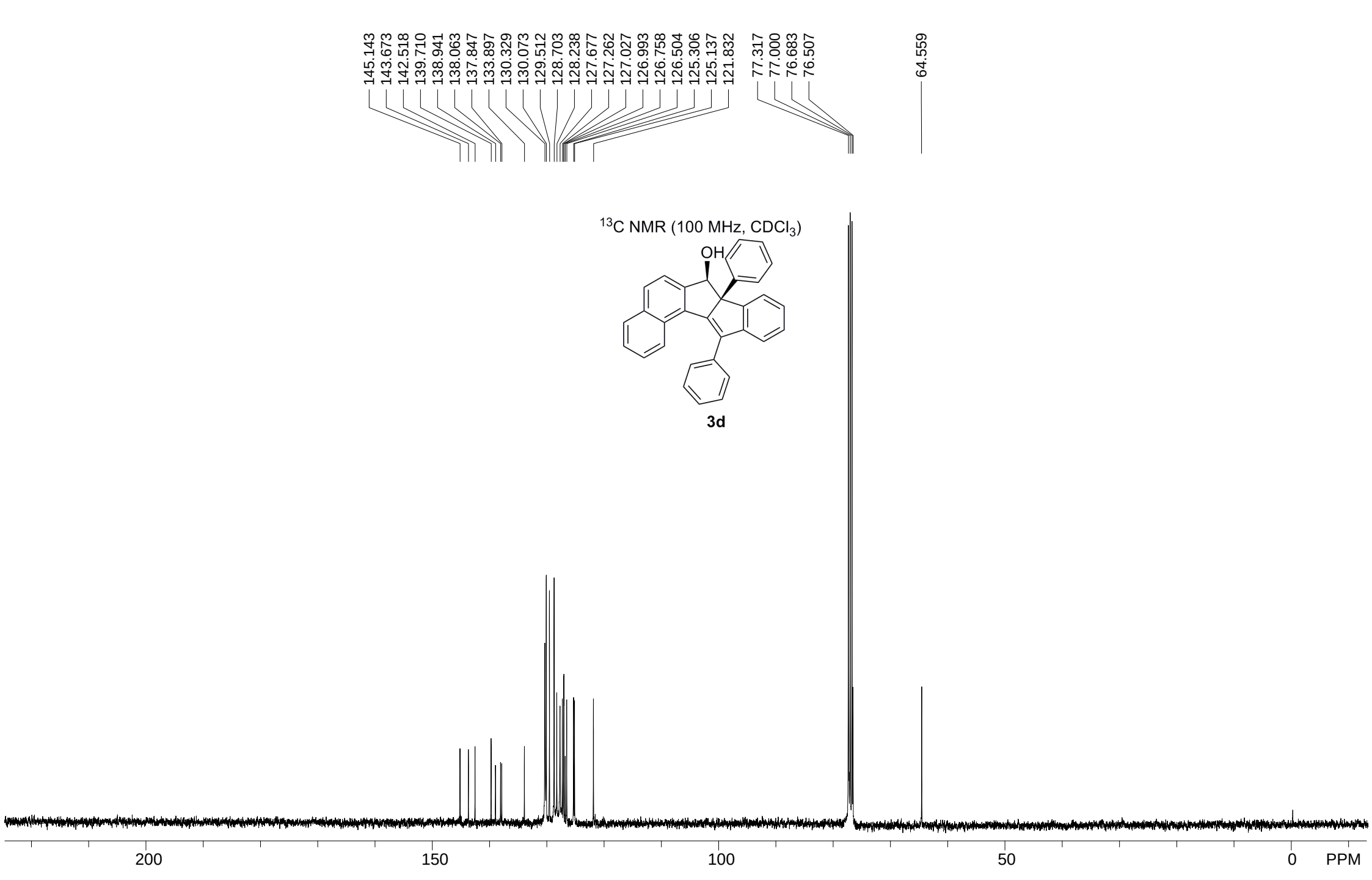
^{13}C NMR (100 MHz, $CDCl_3$)

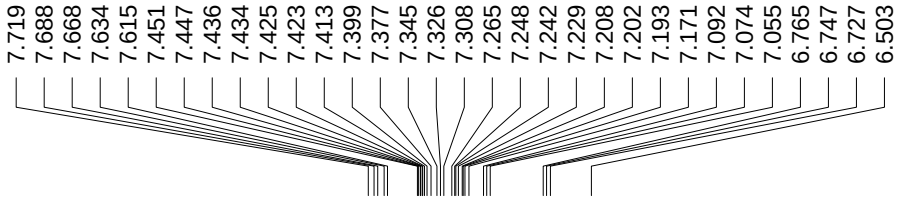


4c

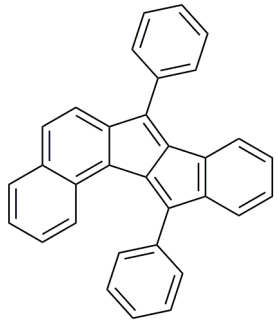



7.812
7.791
7.784
7.763
7.533
7.512
7.406
7.385
7.325
7.301
7.283
7.264
7.247
7.235
7.217
7.186
7.170
7.163
7.159
7.152
7.146
7.137
7.134
7.127
7.123
7.103
7.084
7.054
7.035
7.018
7.000
6.980
6.959
6.926
6.922
6.909
6.903
6.862
6.816
6.756
6.738
6.729
5.681
5.664
5.652
5.636
4.490
4.474
-0.000
^{1}H NMR (400 MHz, $CDCl_3$)
OH
3d
6.02
4.03
2.05
1.98
0.99
1.05
1.01
1.96
0.99
0.99
1.03
10
8
6
4
2
0
PPM

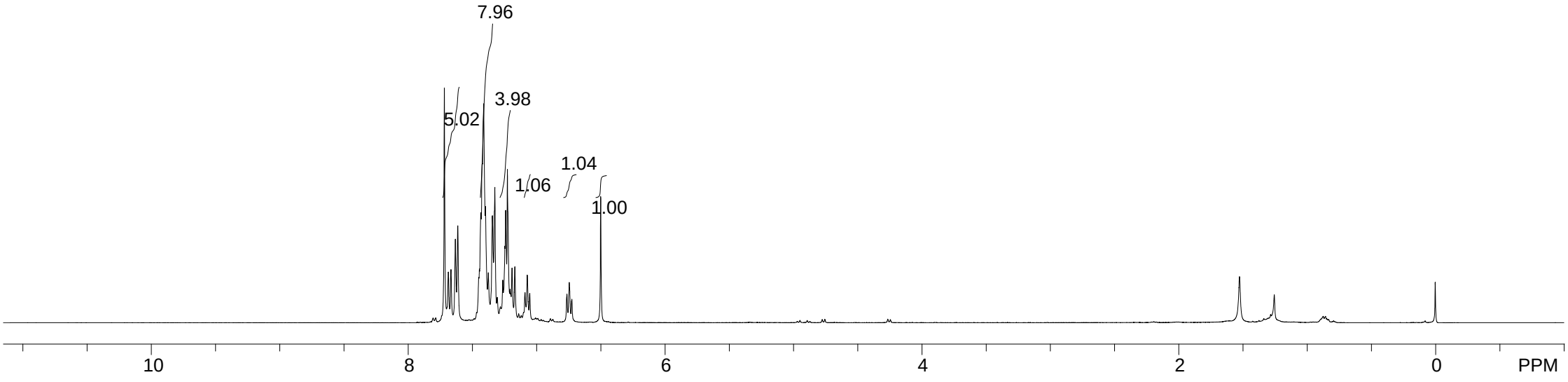

145.143
143.673
142.518
139.710
138.941
138.063
137.847
133.897
130.329
130.073
129.512
128.703
128.238
127.677
127.262
127.027
126.993
126.758
126.504
125.306
125.137
121.832
77.317
77.000
76.683
76.507
64.559
^{13}C NMR (100 MHz, $CDCl_3$)
OH
3d
200
150
100
50
0
PPM

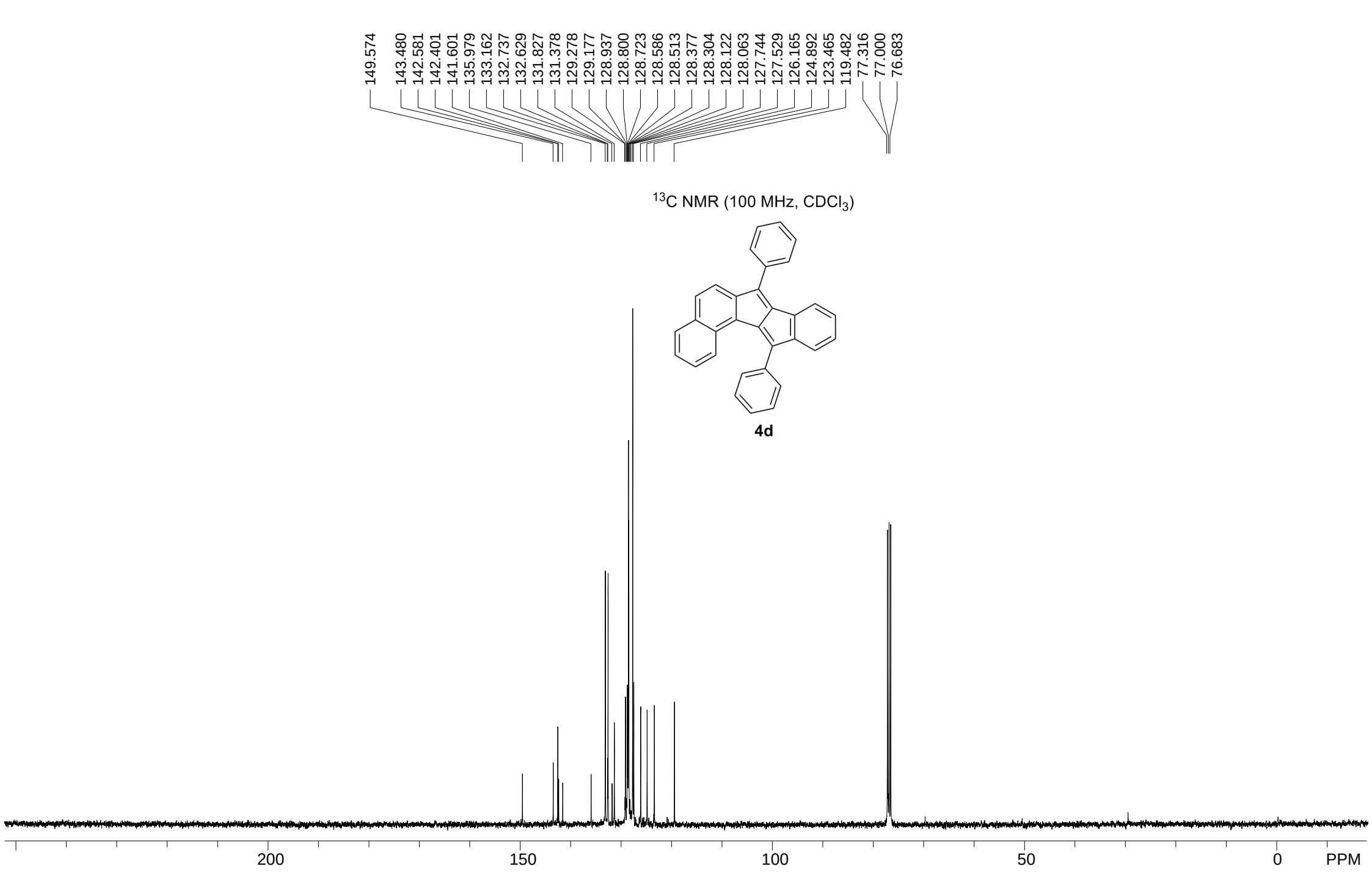


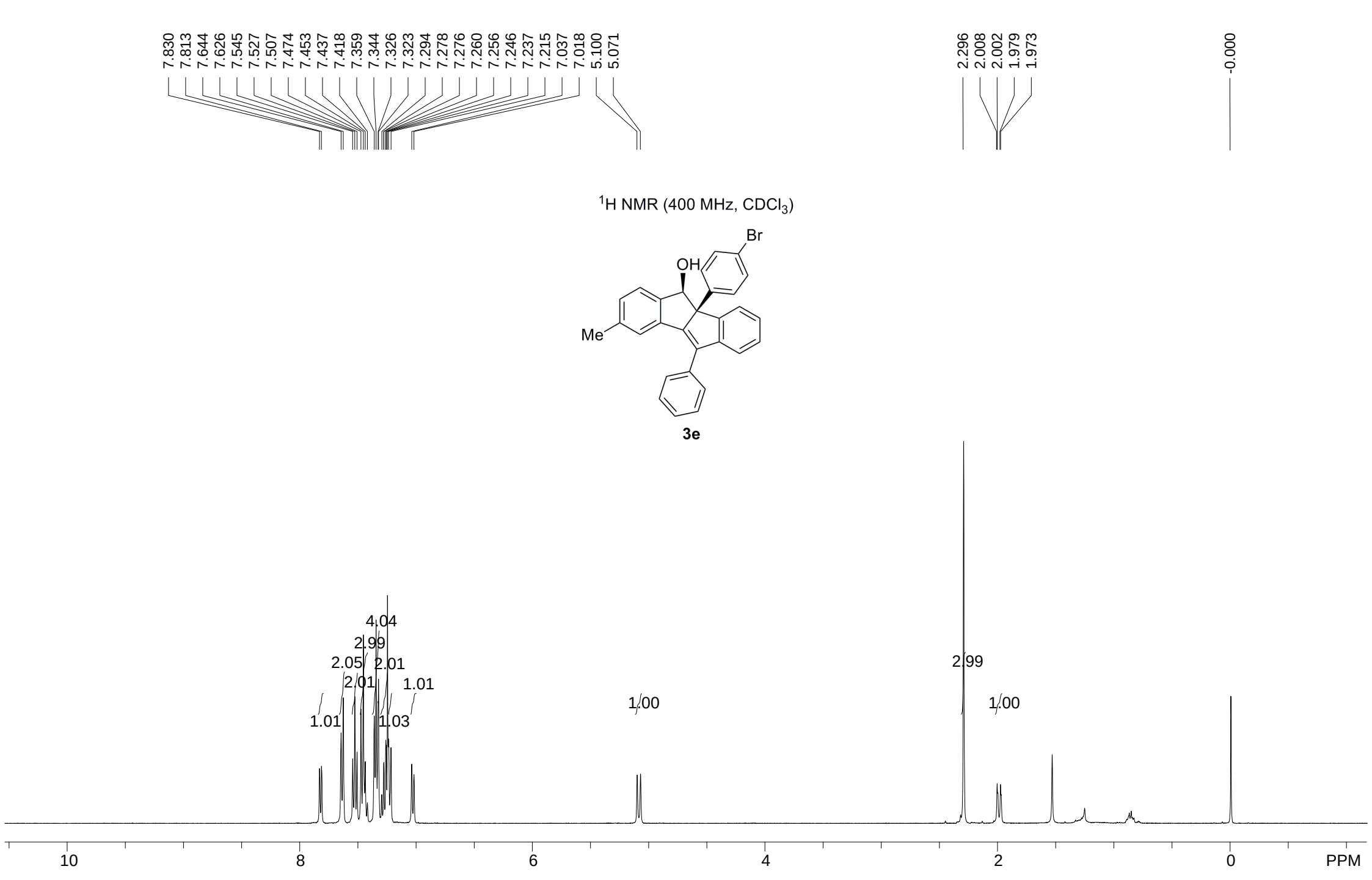
^{1}H NMR (400 MHz, $CDCl_3$)

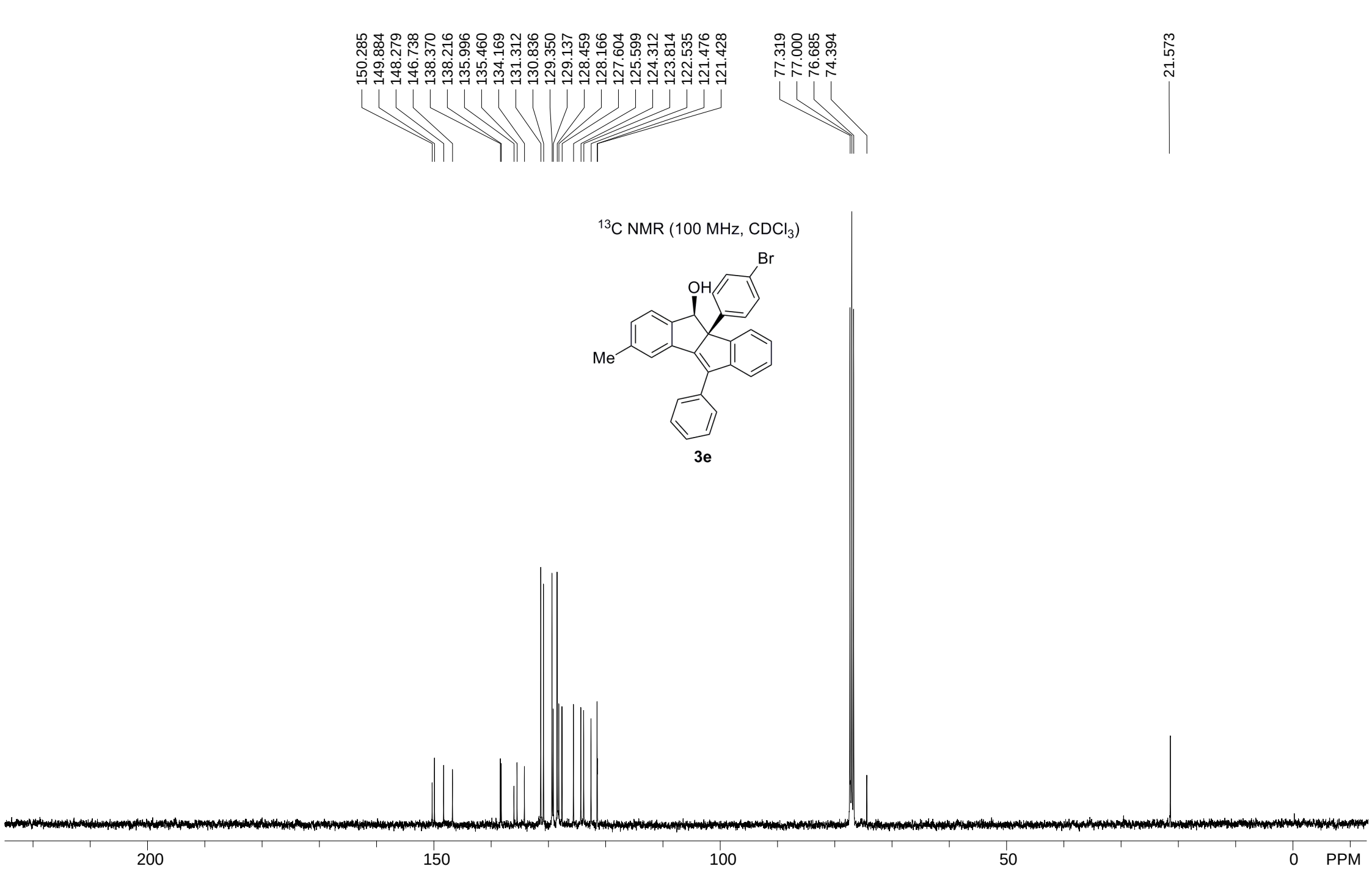


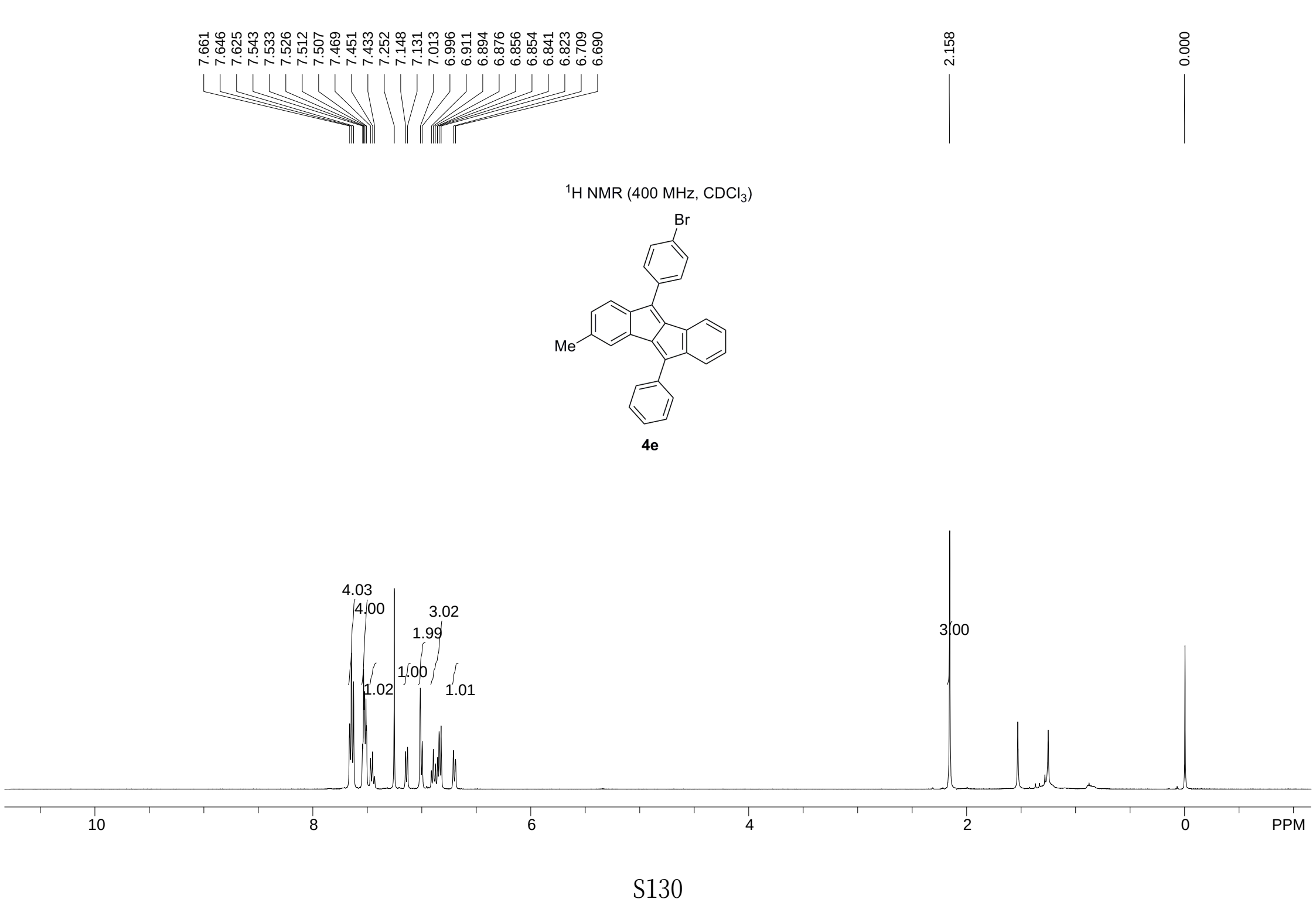
4d

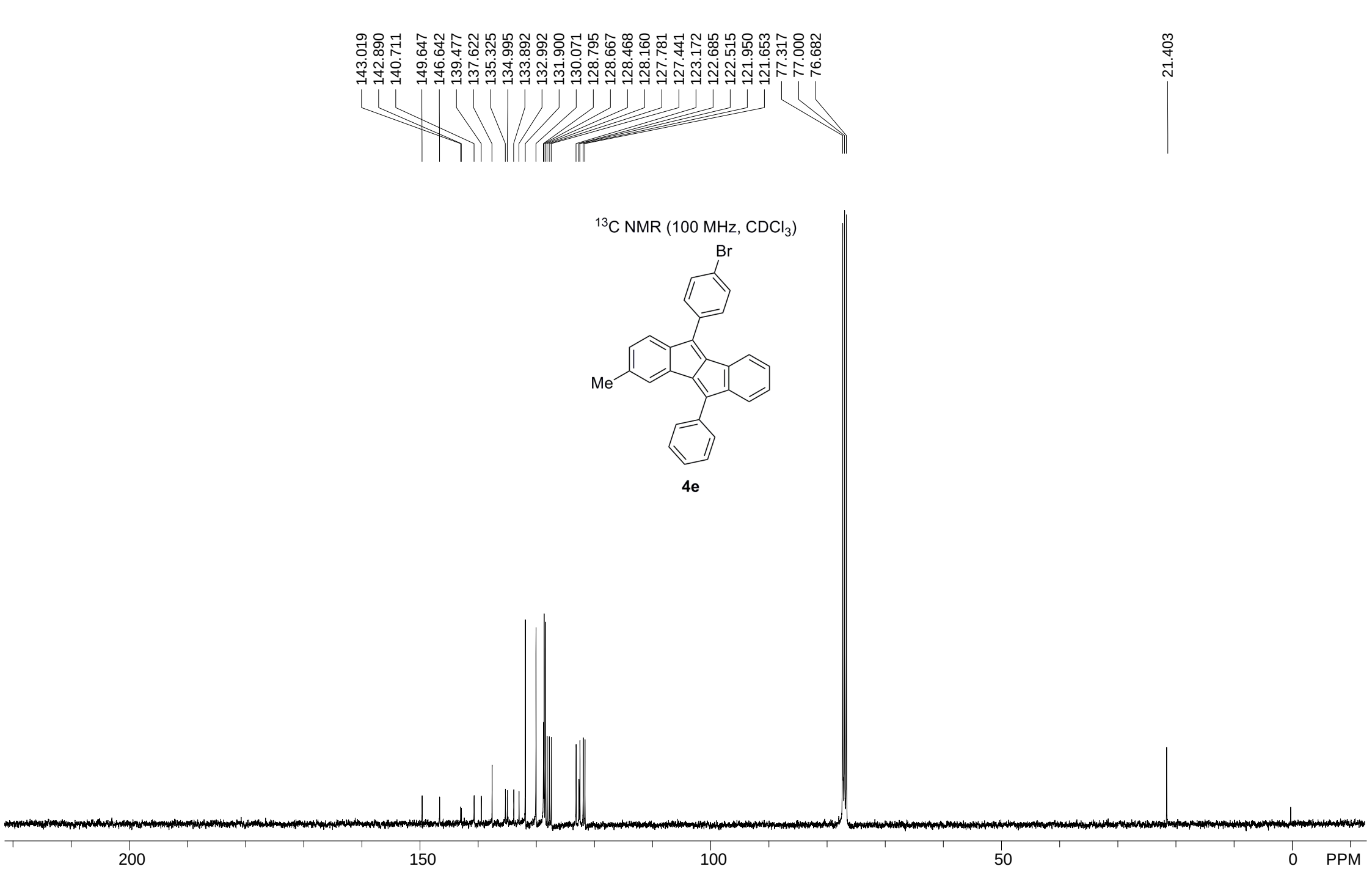


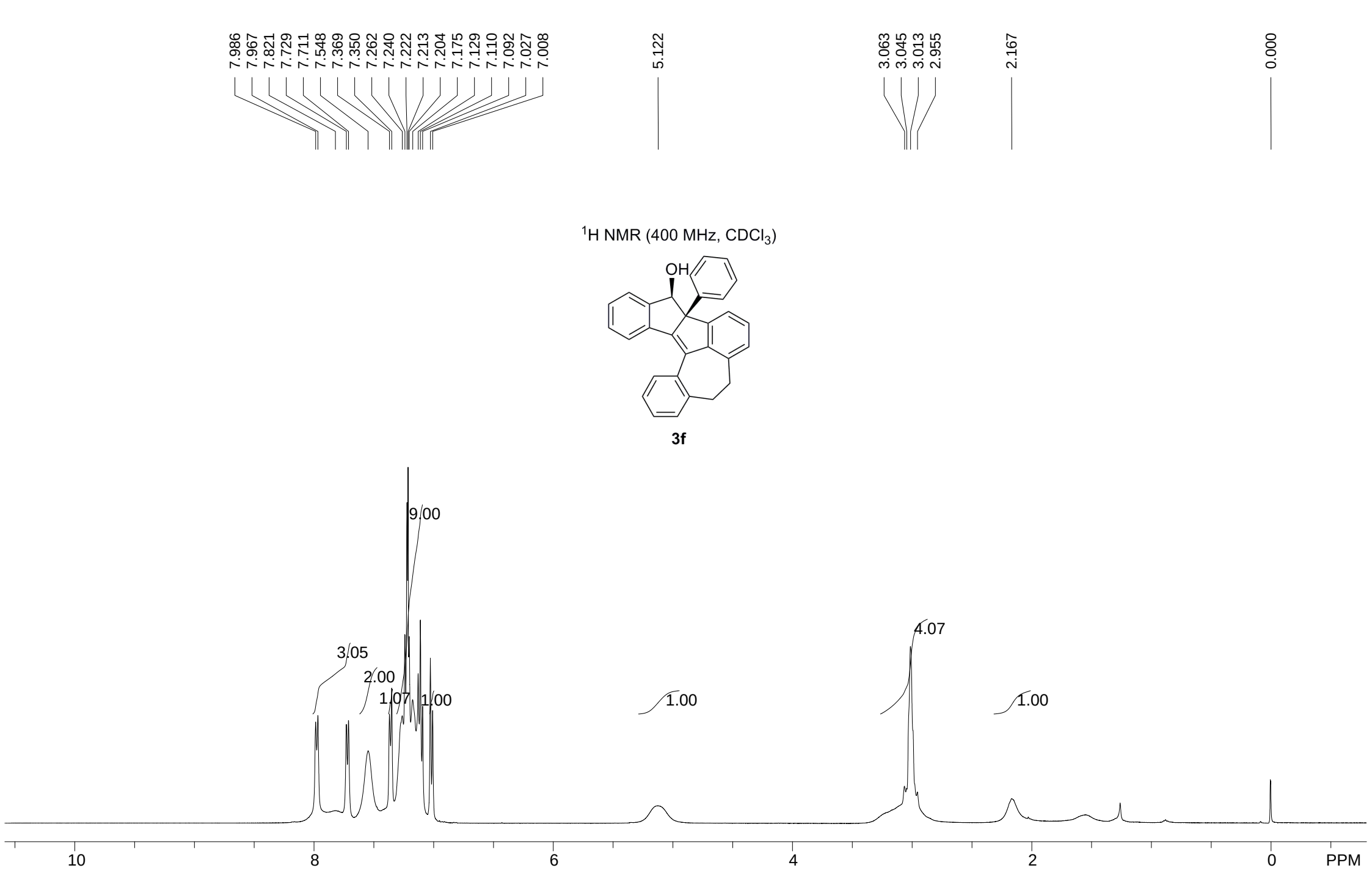

149.574
143.480
142.581
142.401
141.601
135.979
133.162
132.737
132.629
131.827
131.378
129.278
129.177
128.937
128.800
128.723
128.586
128.513
128.377
128.304
128.122
128.063
127.744
127.529
126.165
124.892
123.465
119.482
77.316
77.000
76.683
13C NMR (100 MHz, CDCl3)
4d
200
150
100
50
0
PPM

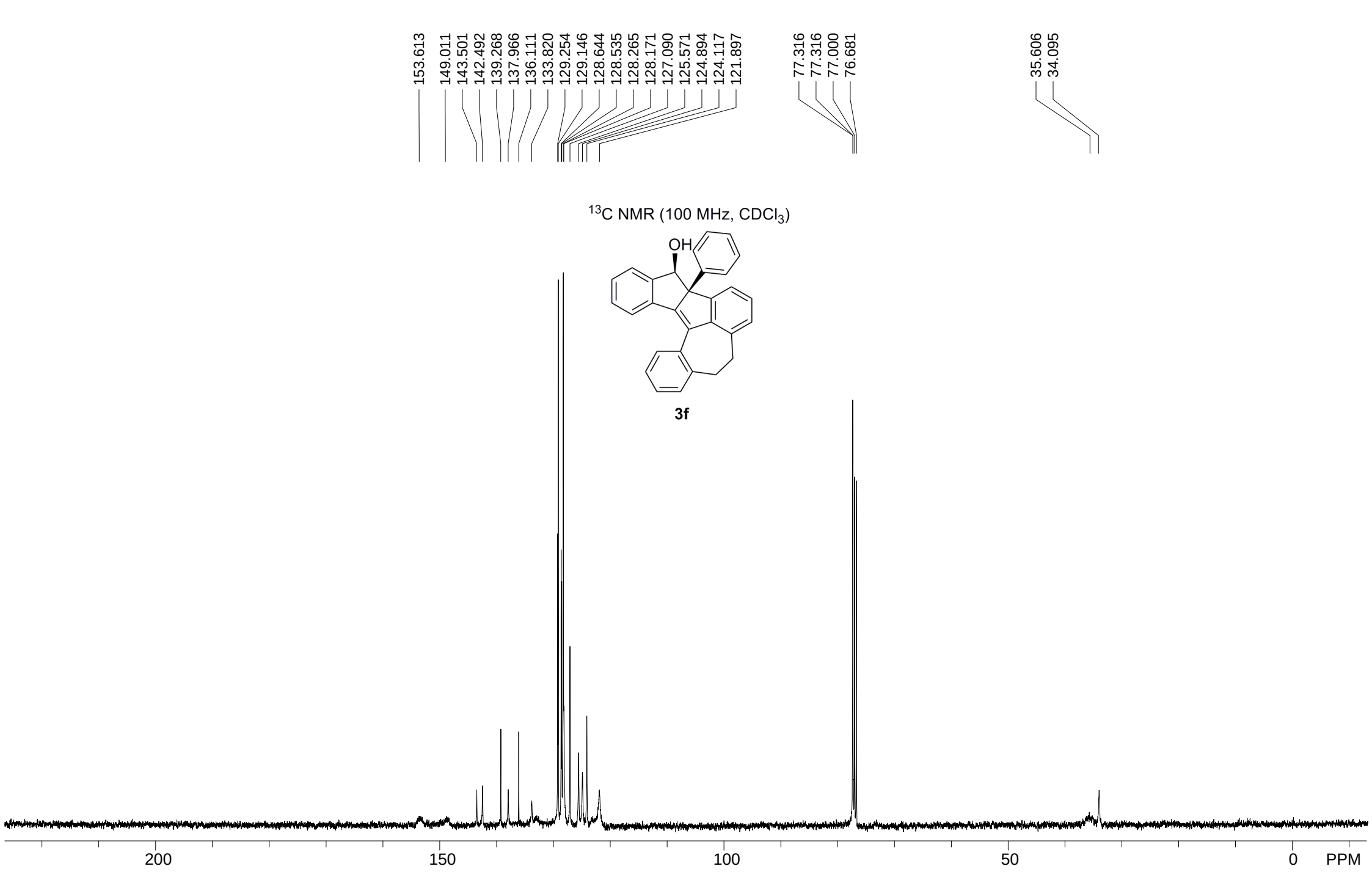

7.830
7.813
7.644
7.626
7.545
7.527
7.507
7.474
7.453
7.437
7.418
7.359
7.344
7.326
7.323
7.294
7.278
7.276
7.260
7.256
7.246
7.237
7.215
7.037
7.018
5.100
5.071
2.296
2.008
2.002
1.979
1.973
-0.000
1H NMR (400 MHz, CDCl3)
Br
OH
Me
3e
4.04
2.99
2.05
2.01
2.01
1.01
1.01
1.03
1.00
2.99
1.00
10
8
6
4
2
0
PPM

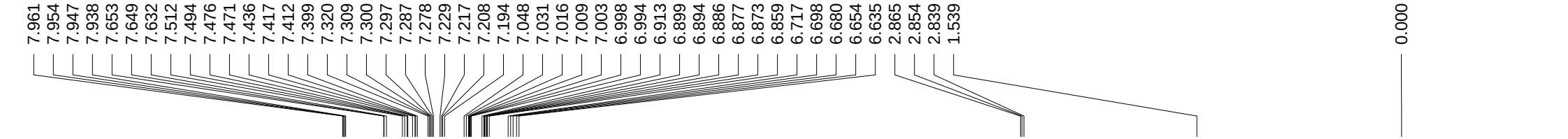

150.285
149.884
148.279
146.738
138.370
138.216
135.996
135.460
134.169
131.312
130.836
129.350
129.137
128.459
128.166
127.604
125.599
124.312
123.814
122.535
121.476
121.428
77.319
77.000
76.685
74.394
21.573
13C NMR (100 MHz, CDCl3)
Br
OH
Me
3e
200
150
100
50
0
PPM


7.661
7.646
7.625
7.543
7.533
7.526
7.512
7.507
7.469
7.451
7.433
7.252
7.148
7.131
7.013
6.996
6.911
6.894
6.876
6.856
6.854
6.841
6.823
6.709
6.690
2.158
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
Br
Me
4e
4.03
4.00
3.02
1.99
1.00
1.02
1.01
3.00
10
8
6
4
2
0
PPM

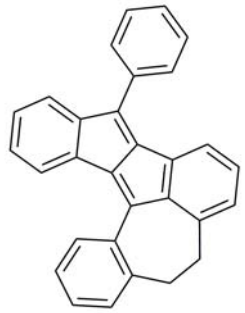

143.019
142.890
140.711
149.647
146.642
139.477
137.622
135.325
134.995
133.892
132.992
131.900
130.071
128.795
128.667
128.468
128.160
127.781
127.441
123.172
122.685
122.515
121.950
121.653
77.317
77.000
76.682
21.403
^{13}C NMR (100 MHz, $CDCl_3$)
Br
Me
4e
200
150
100
50
0
PPM


7.986
7.967
7.821
7.729
7.711
7.548
7.369
7.350
7.262
7.240
7.222
7.213
7.204
7.175
7.129
7.110
7.092
7.027
7.008
5.122
3.063
3.045
3.013
2.955
2.167
0.000
1H NMR (400 MHz, CDCl3)
OH
3f
9.00
3.05
2.00
1.07
1.00
1.00
4.07
1.00
10
8
6
4
2
0
PPM

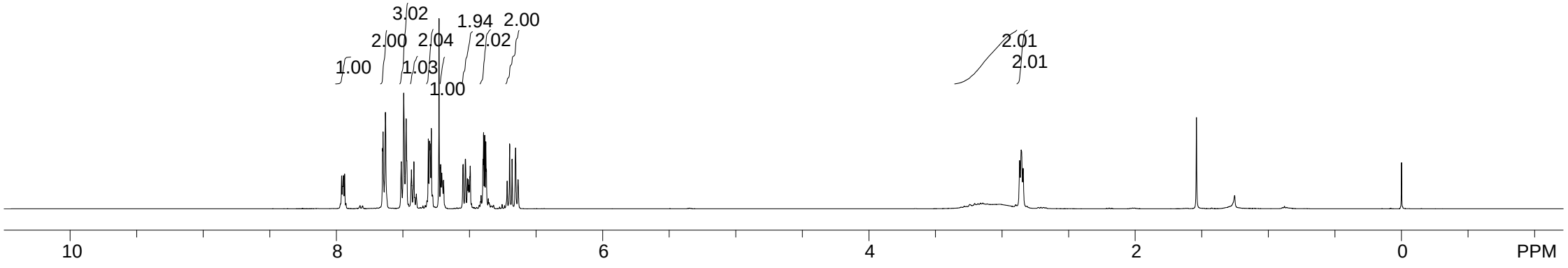

153.613
149.011
143.501
142.492
139.268
137.966
136.111
133.820
129.254
129.146
128.644
128.535
128.265
128.171
127.090
125.571
124.894
124.117
121.897
77.316
77.316
77.000
76.681
35.606
34.095
13C NMR (100 MHz, CDCl3)
OH
3f
200
150
100
50
0
PPM



^{1}H NMR (400 MHz, $CDCl_3$)



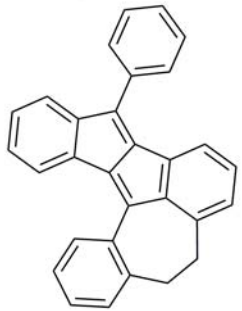
4f



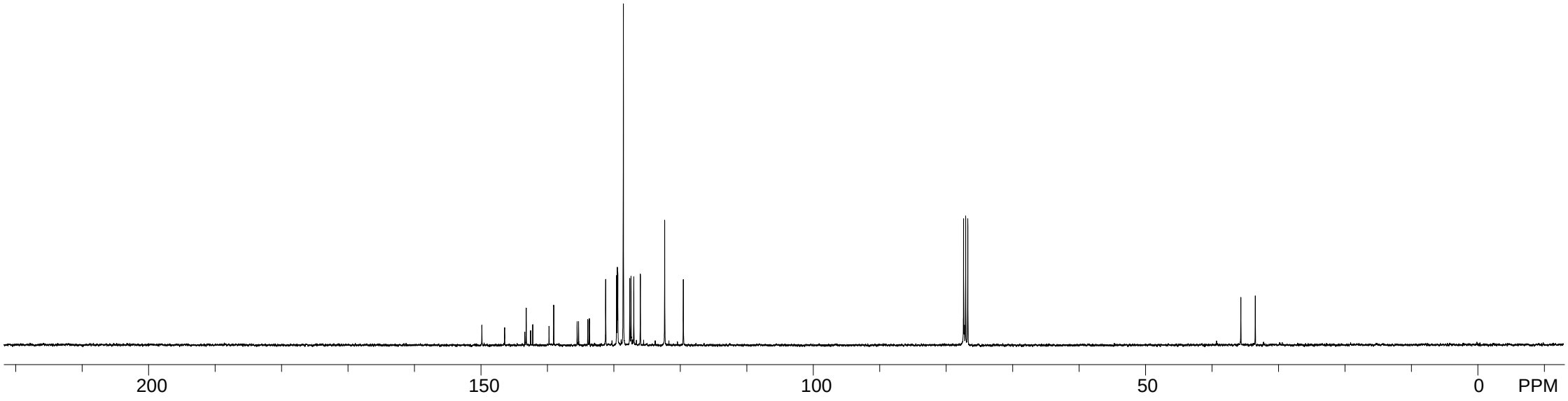
143.418
143.204
142.538
149.886
146.457
142.218
139.767
139.055
135.538
135.315
133.926
133.688
131.235
129.580
129.476
129.403
128.564
127.606
127.415
126.997
126.010
122.345
119.539
77.318
77.000
76.681

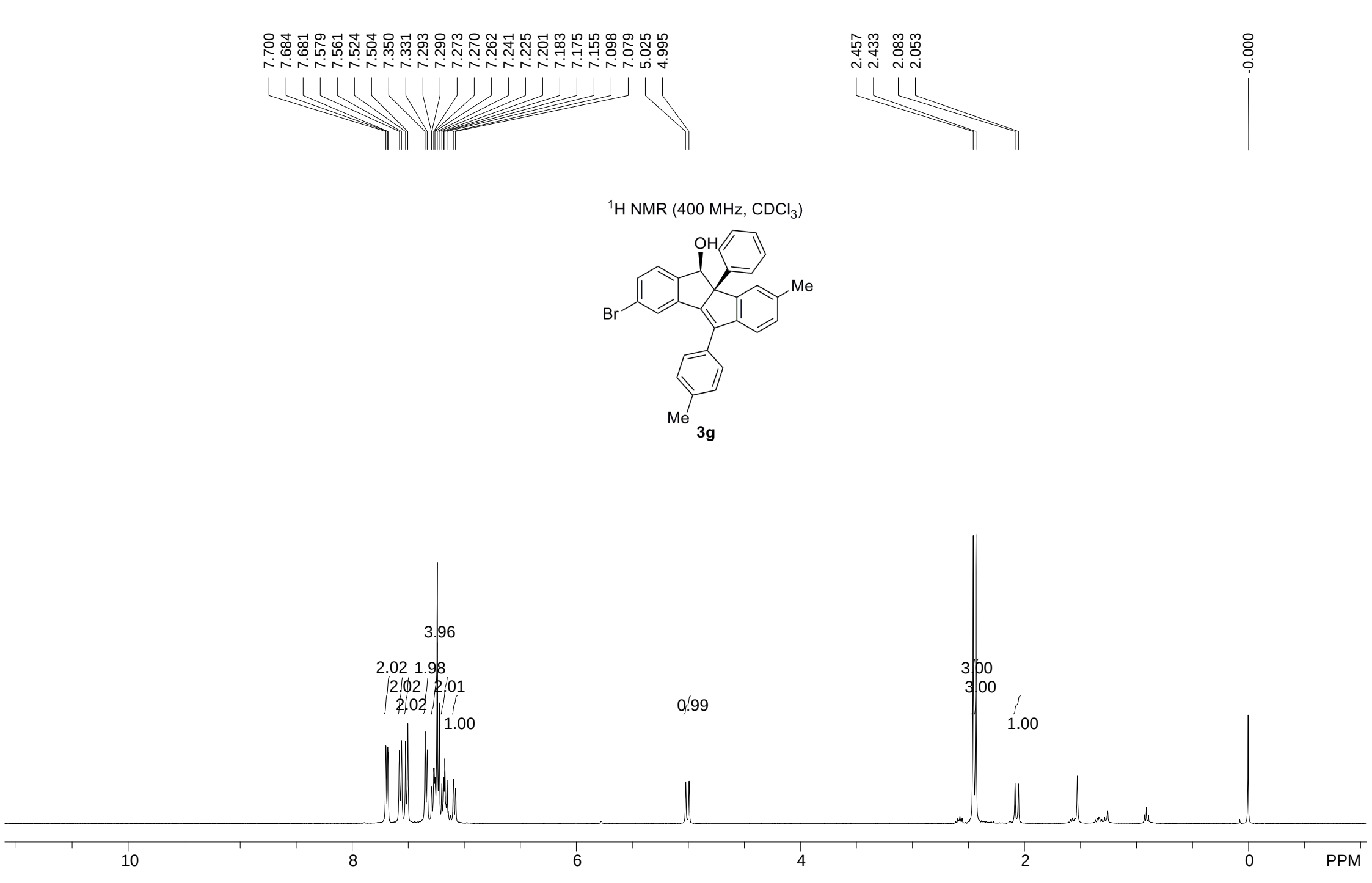
35.552
33.375

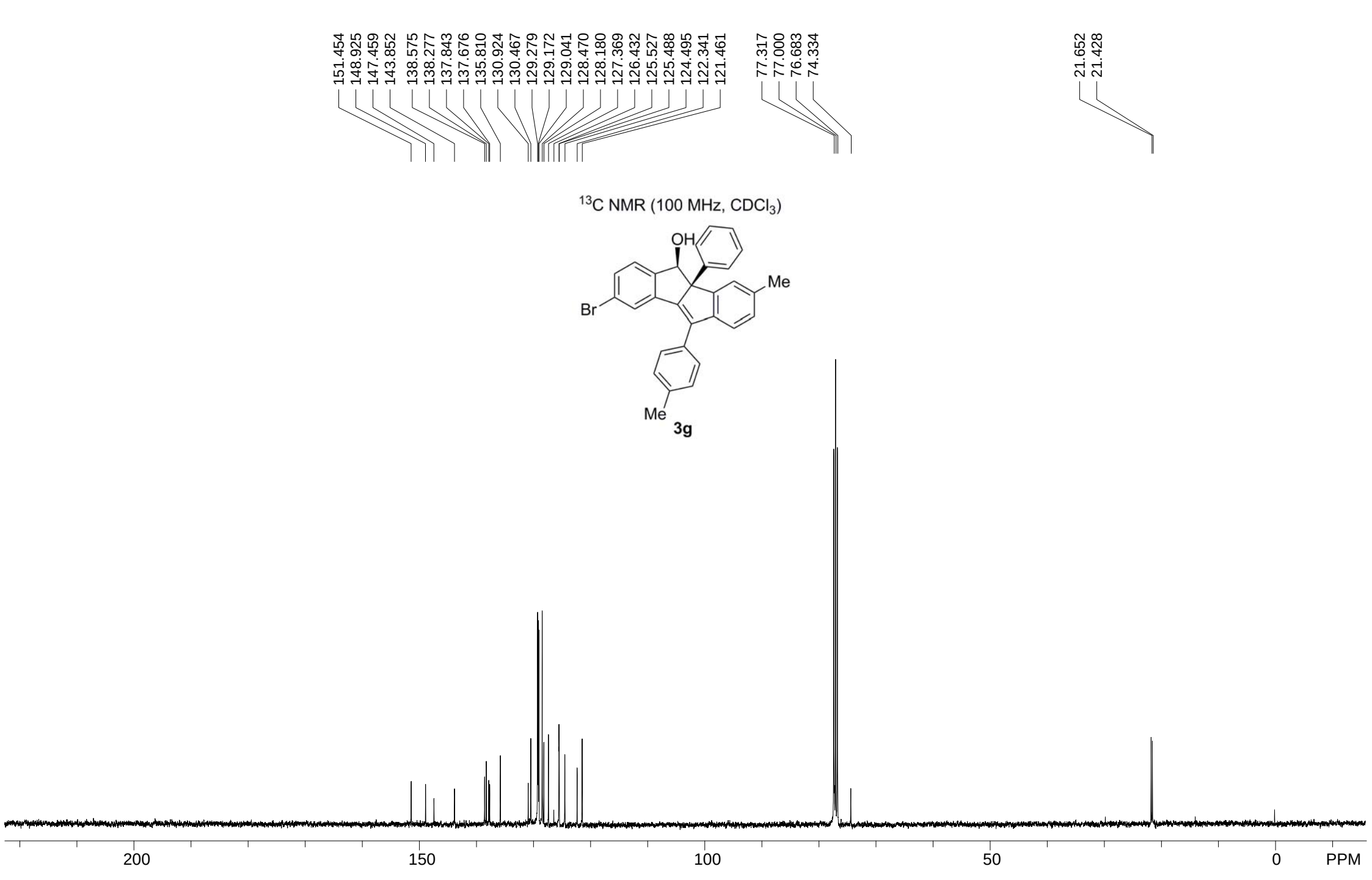
^{13}C NMR (100 MHz, $CDCl_3$)

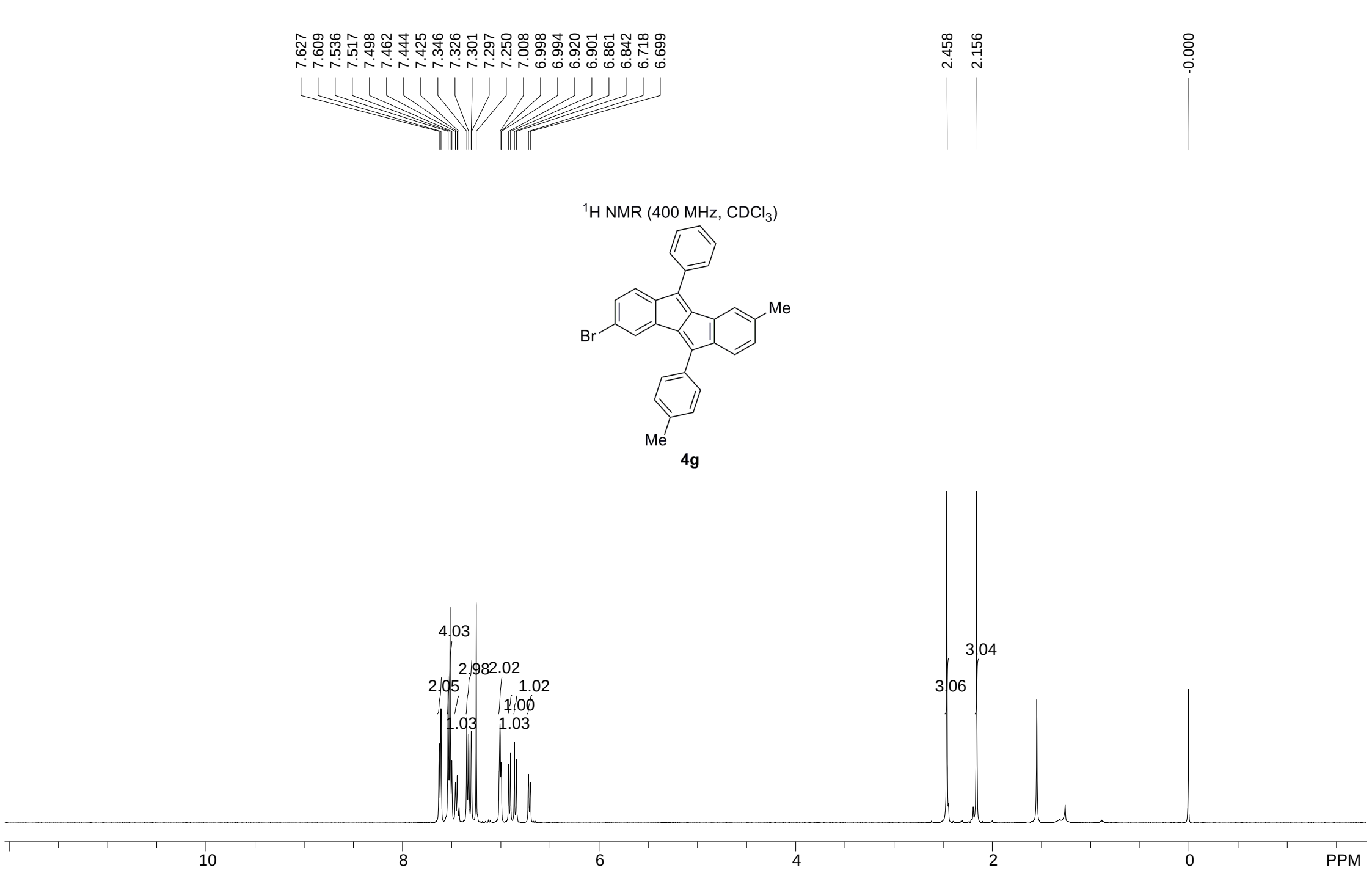


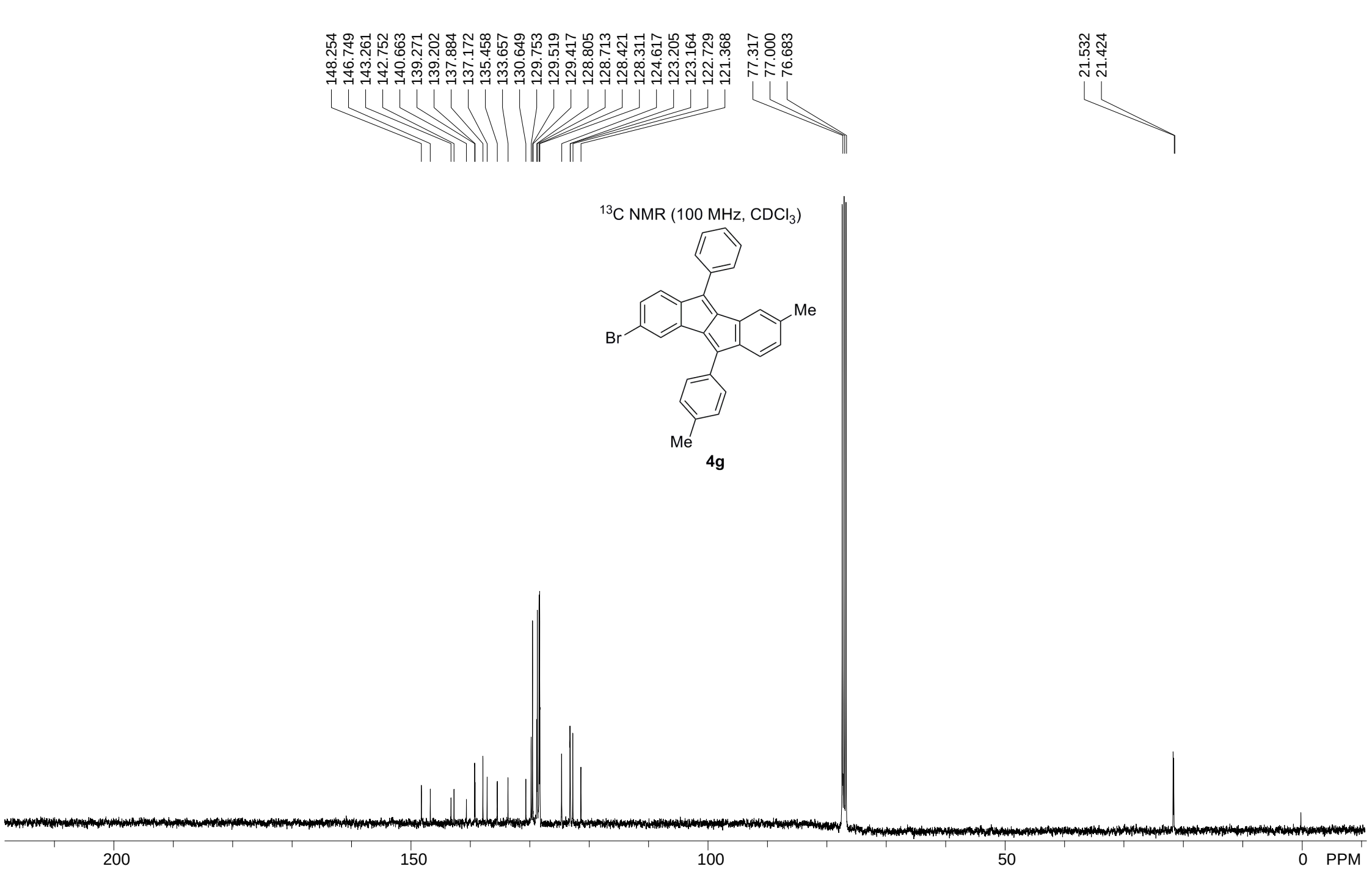
4f

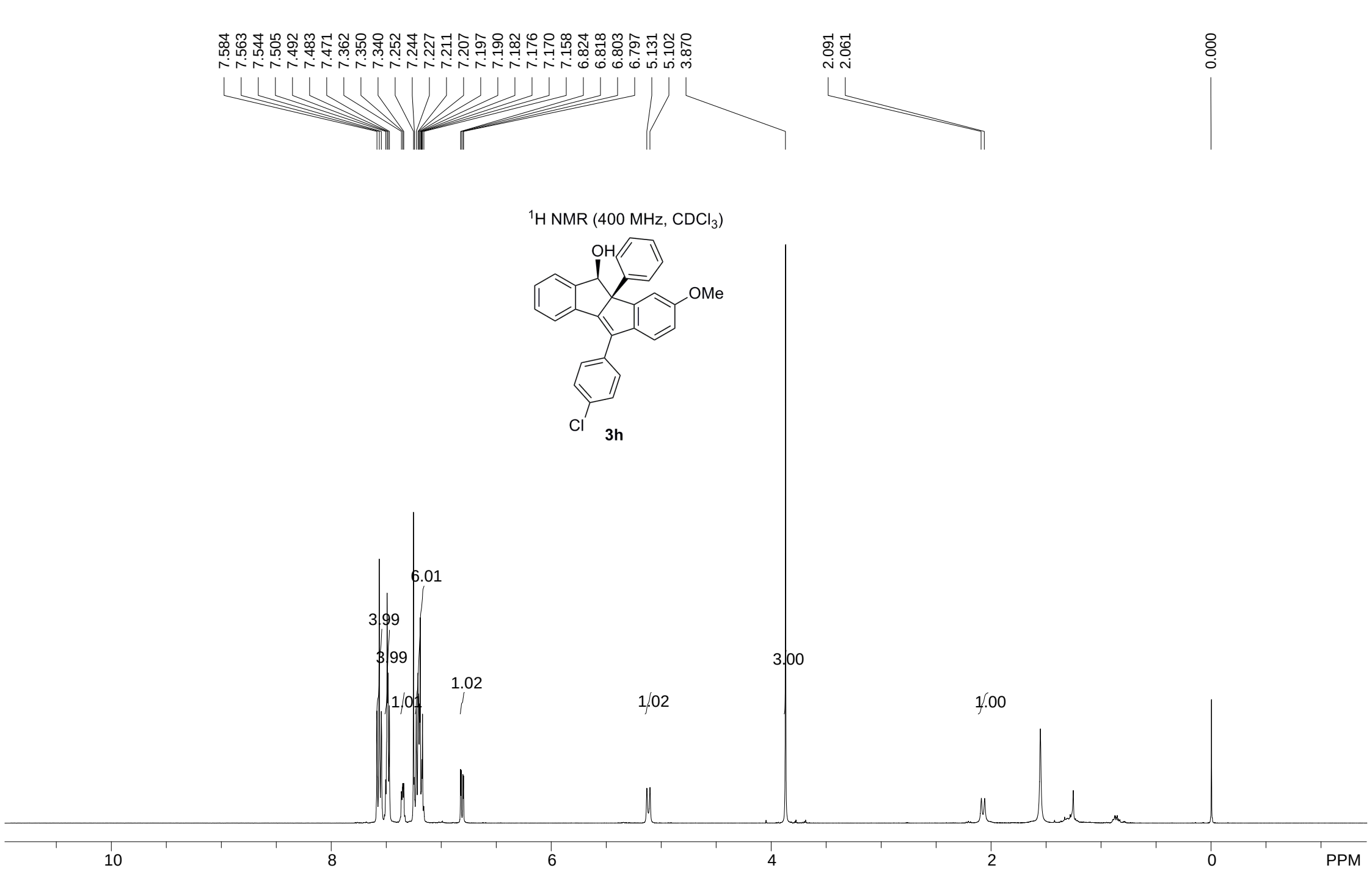


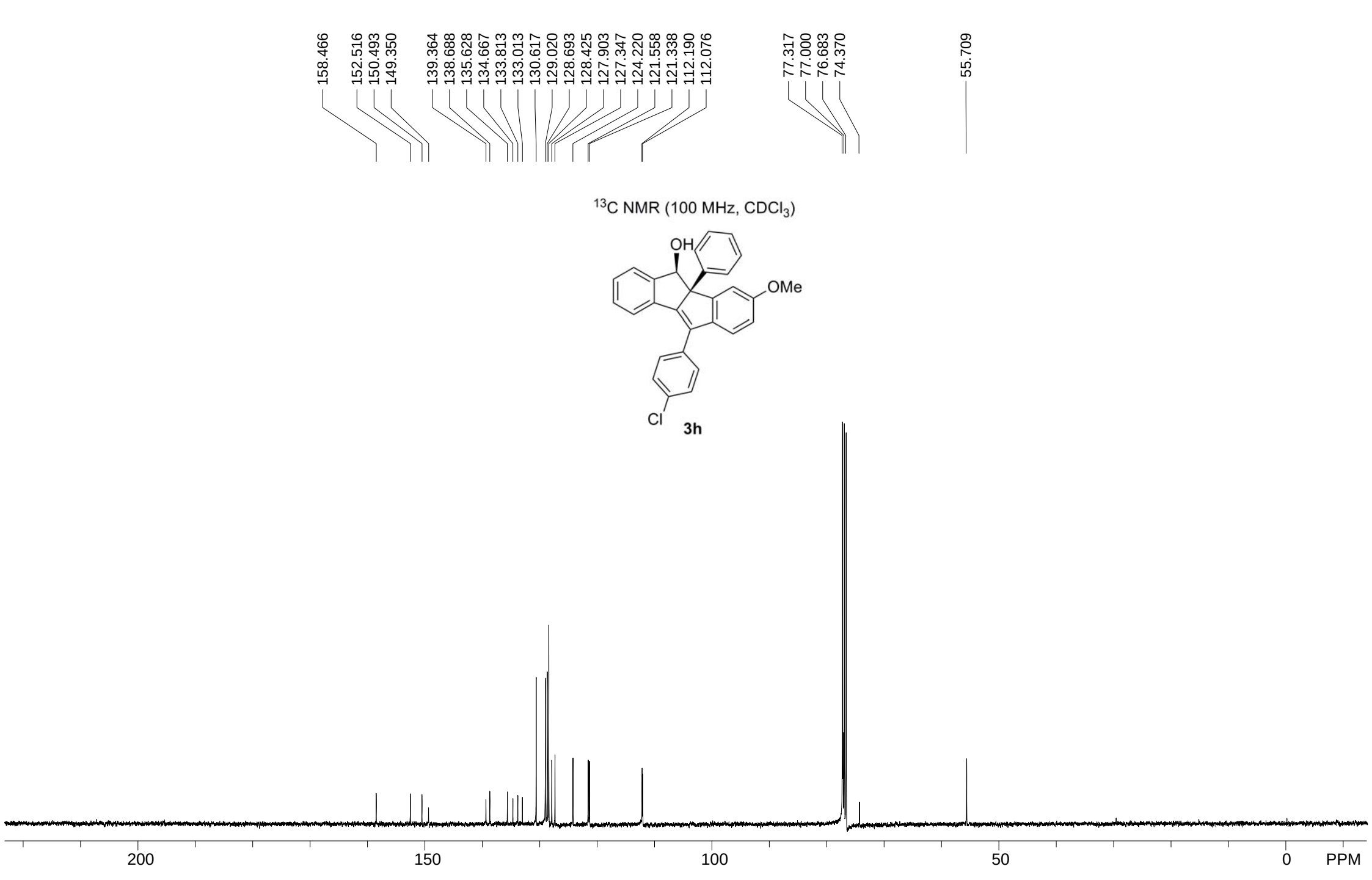

7.700
7.684
7.681
7.579
7.561
7.524
7.504
7.350
7.331
7.293
7.290
7.273
7.270
7.262
7.241
7.225
7.201
7.183
7.175
7.155
7.098
7.079
5.025
4.995
2.457
2.433
2.083
2.053
-0.000
1H NMR (400 MHz, CDCl3)
OH
Me
Br
Me
3g
3.96
2.02
1.98
2.02
2.02
2.01
1.00
0.99
3.00
3.00
1.00
10
8
6
4
2
0
PPM

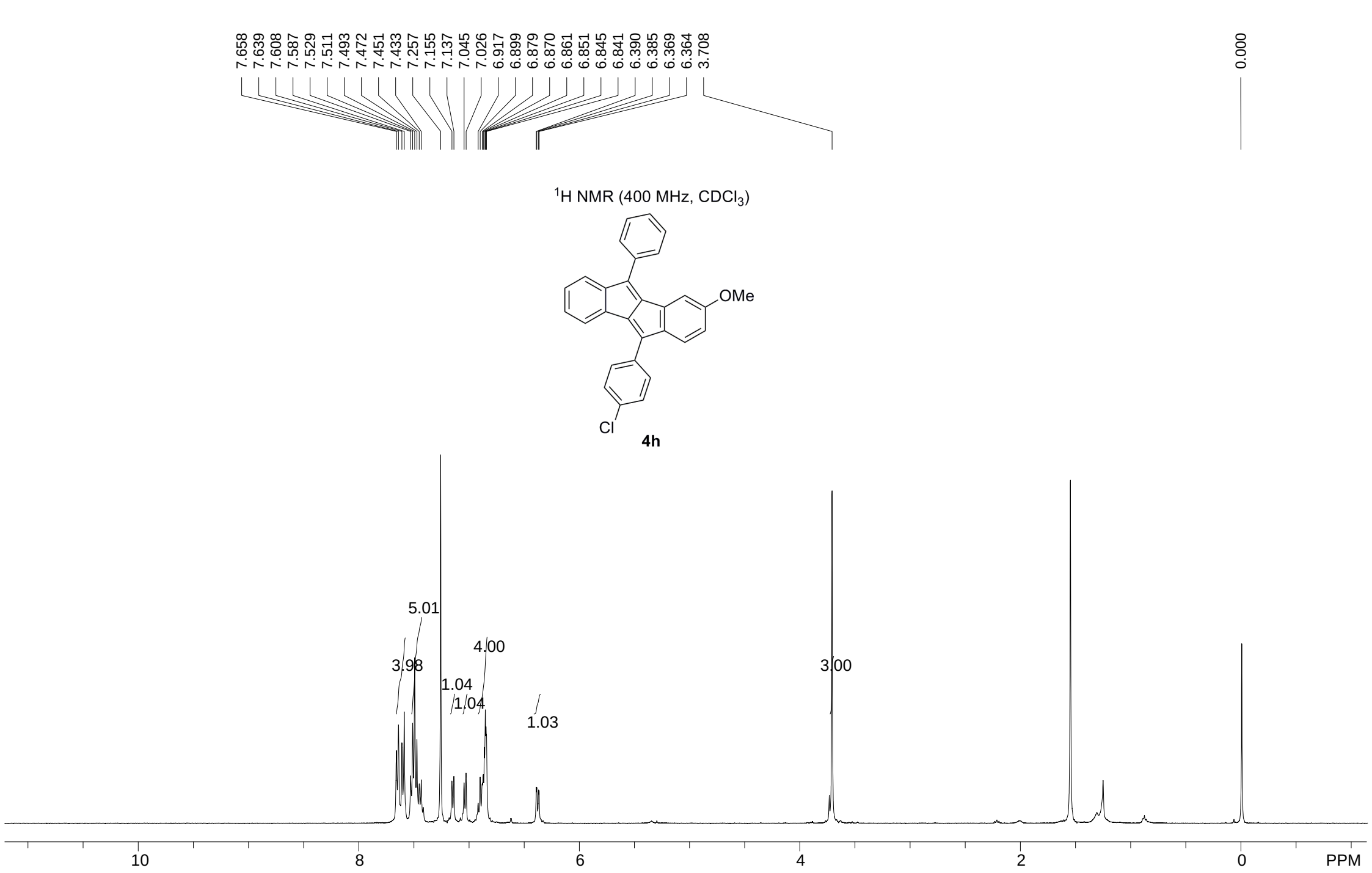

151.454
148.925
147.459
143.852
138.575
138.277
137.843
137.676
135.810
130.924
130.467
129.279
129.172
129.041
128.470
128.180
127.369
126.432
125.527
125.488
124.495
122.341
121.461
77.317
77.000
76.683
74.334
21.652
21.428
^{13}C NMR (100 MHz, $CDCl_3$)
OH
Me
Br
Me
3g
200
150
100
50
0
PPM

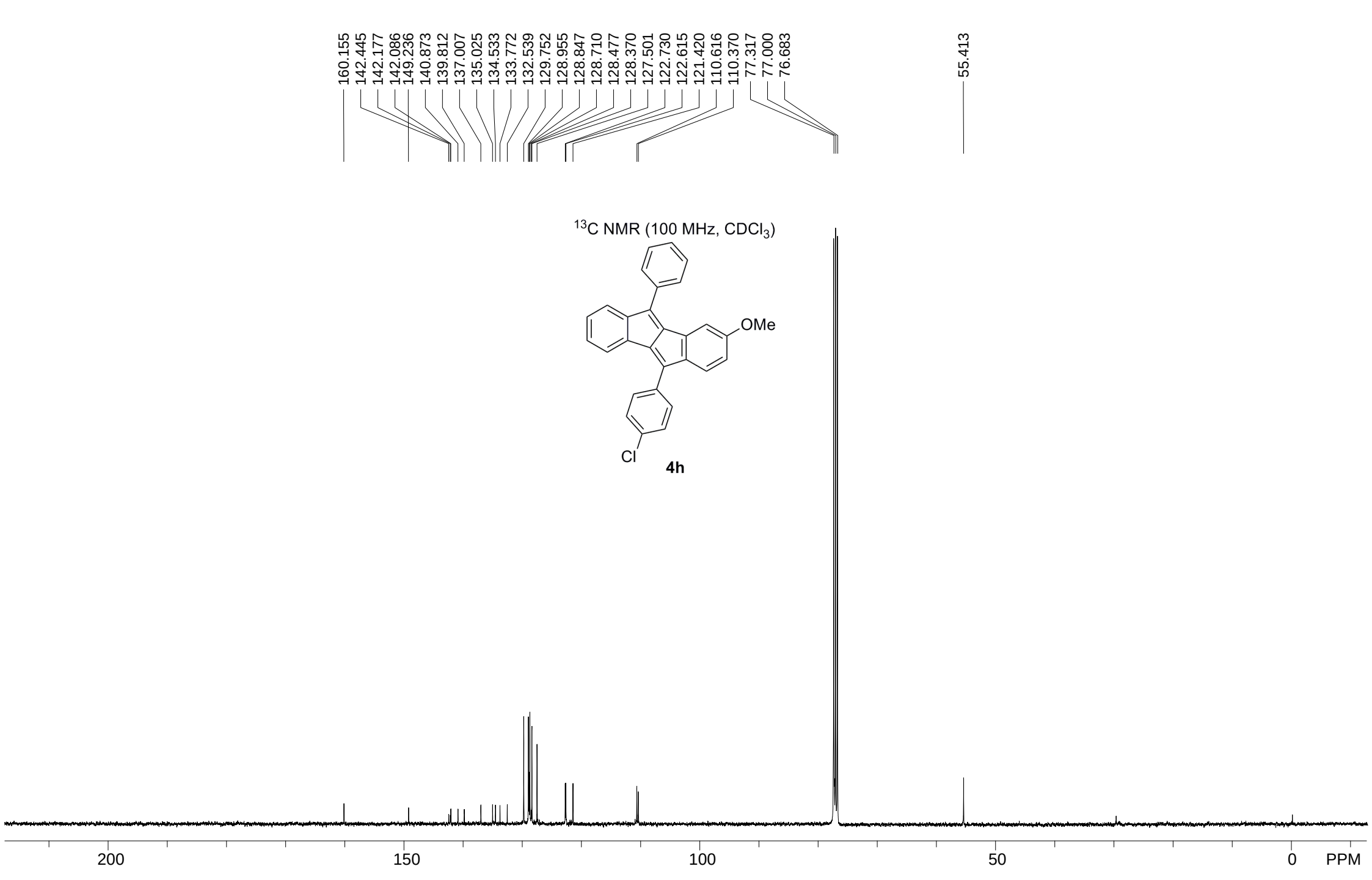

7.627
7.609
7.536
7.517
7.498
7.462
7.444
7.425
7.346
7.326
7.301
7.297
7.250
7.008
6.998
6.994
6.920
6.901
6.861
6.842
6.718
6.699
2.458
2.156
-0.000
1H NMR (400 MHz, CDCl3)
Br
Me
Me
4g
4.03
2.98
2.02
2.05
1.02
1.00
1.03
1.03
3.06
3.04
10
8
6
4
2
0
PPM

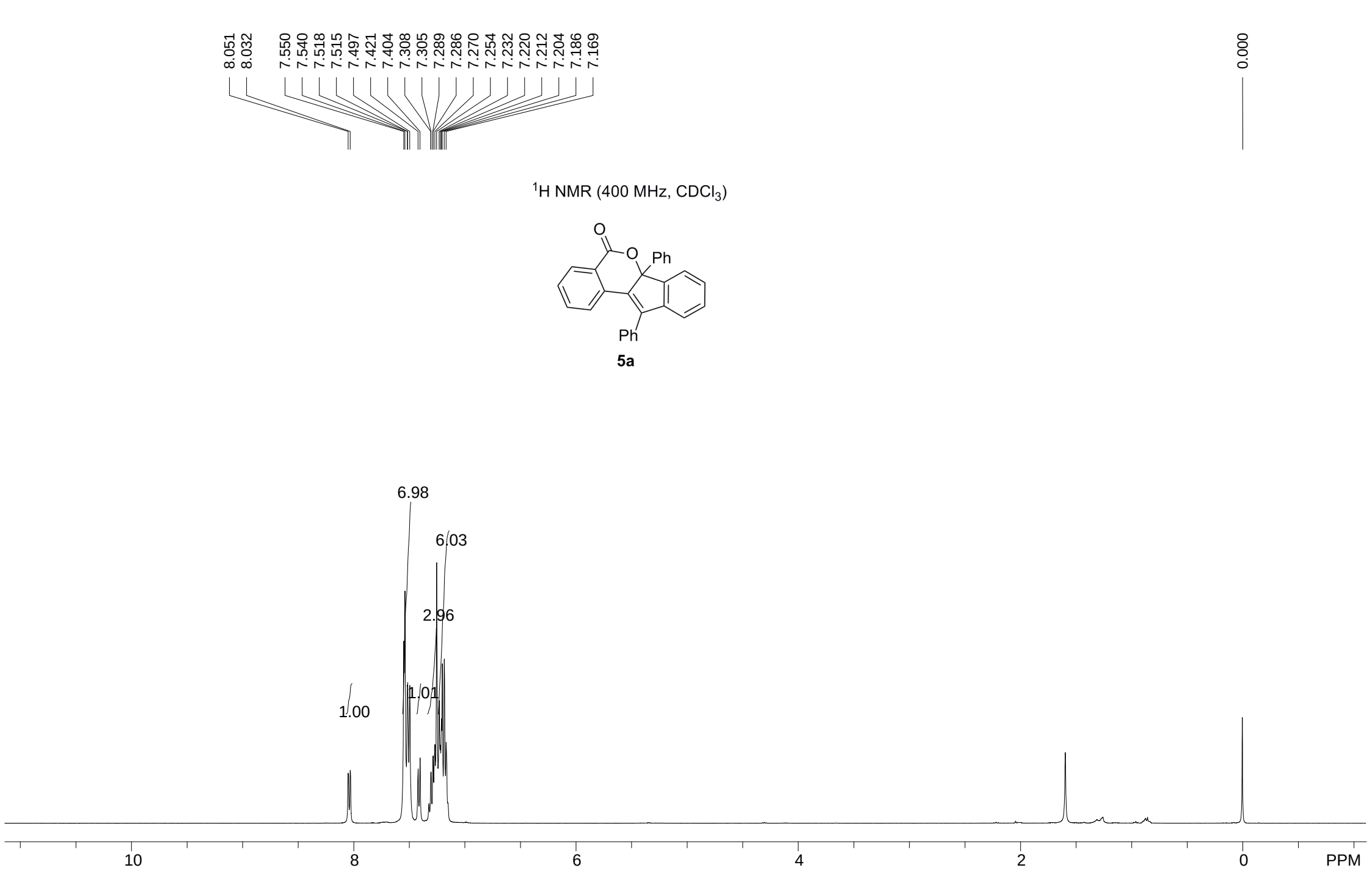

148.254
146.749
143.261
142.752
140.663
139.271
139.202
137.884
137.172
135.458
133.657
130.649
129.753
129.519
129.417
128.805
128.713
128.421
128.311
124.617
123.205
123.164
122.729
121.368
77.317
77.000
76.683
21.532
21.424
13C NMR (100 MHz, CDCl3)
Br
Me
Me
4g
200
150
100
50
0
PPM


7.584
7.563
7.544
7.505
7.492
7.483
7.471
7.362
7.350
7.340
7.252
7.244
7.227
7.211
7.207
7.197
7.190
7.182
7.176
7.170
7.158
6.824
6.818
6.803
6.797
5.131
5.102
3.870
2.091
2.061
0.000
1H NMR (400 MHz, CDCl3)
OH
OMe
Cl
3h
6.01
3.99
3.99
1.02
1.01
1.02
3.00
1.00
10
8
6
4
2
0
PPM


158.466
152.516
150.493
149.350
139.364
138.688
135.628
134.667
133.813
133.013
130.617
129.020
128.693
128.425
127.903
127.347
124.220
121.558
121.338
112.190
112.076
77.317
77.000
76.683
74.370
55.709
^{13}C NMR (100 MHz, CDCl$_3$)
OH
OMe
Cl
3h
200
150
100
50
0
PPM

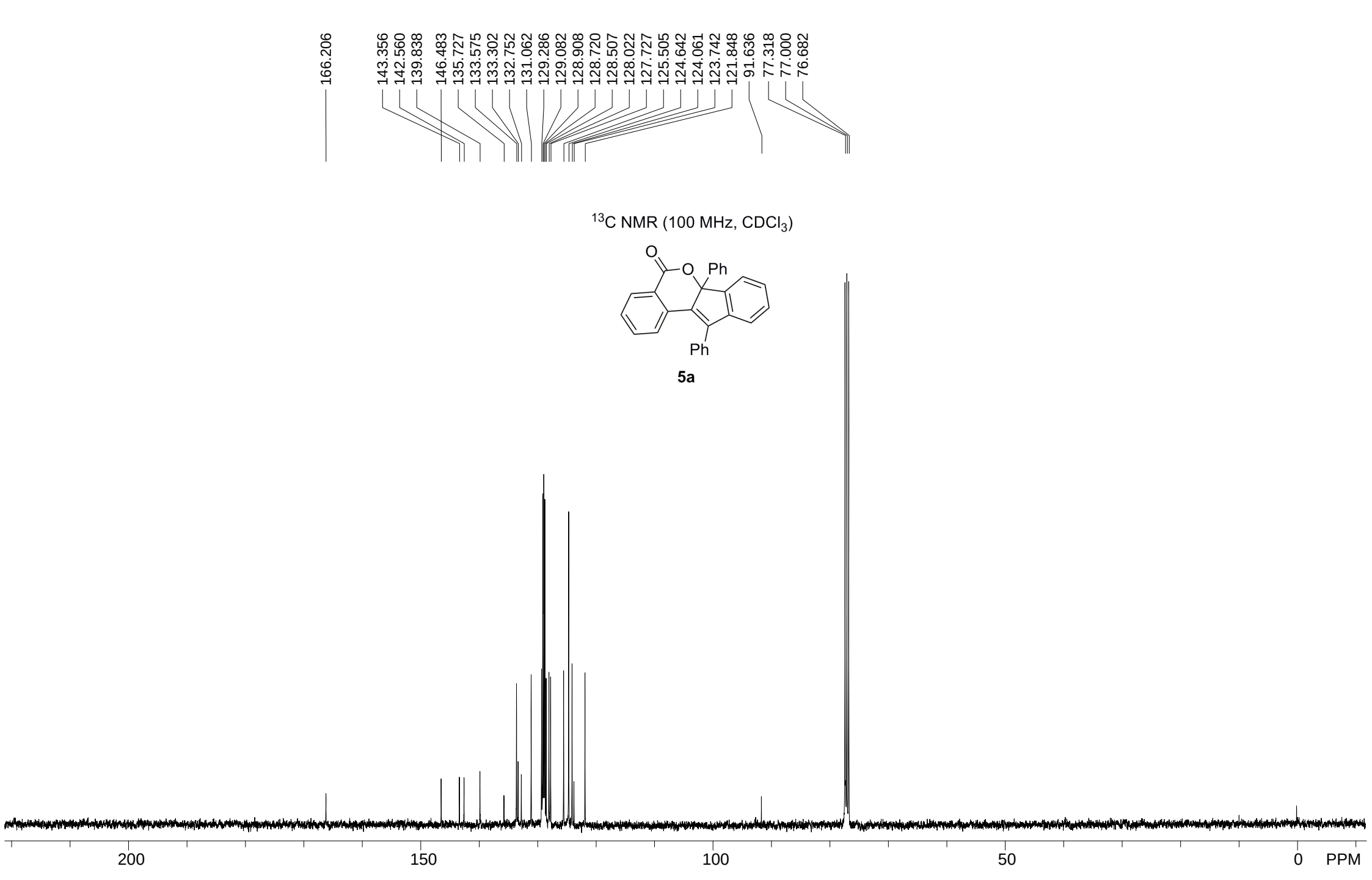

7.658
7.639
7.608
7.587
7.529
7.511
7.493
7.472
7.451
7.433
7.257
7.155
7.137
7.045
7.026
6.917
6.899
6.879
6.870
6.861
6.851
6.845
6.841
6.390
6.385
6.369
6.364
3.708
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
OMe
Cl
4h
5.01
4.00
3.98
1.04
1.04
1.03
3.00
10
8
6
4
2
0
PPM


160.155
142.445
142.177
142.086
149.236
140.873
139.812
137.007
135.025
134.533
133.772
132.539
129.752
128.955
128.847
128.710
128.477
128.370
127.501
122.730
122.615
121.420
110.616
110.370
77.317
77.000
76.683
55.413
13C NMR (100 MHz, CDCl3)
OMe
Cl
4h
200
150
100
50
0
PPM

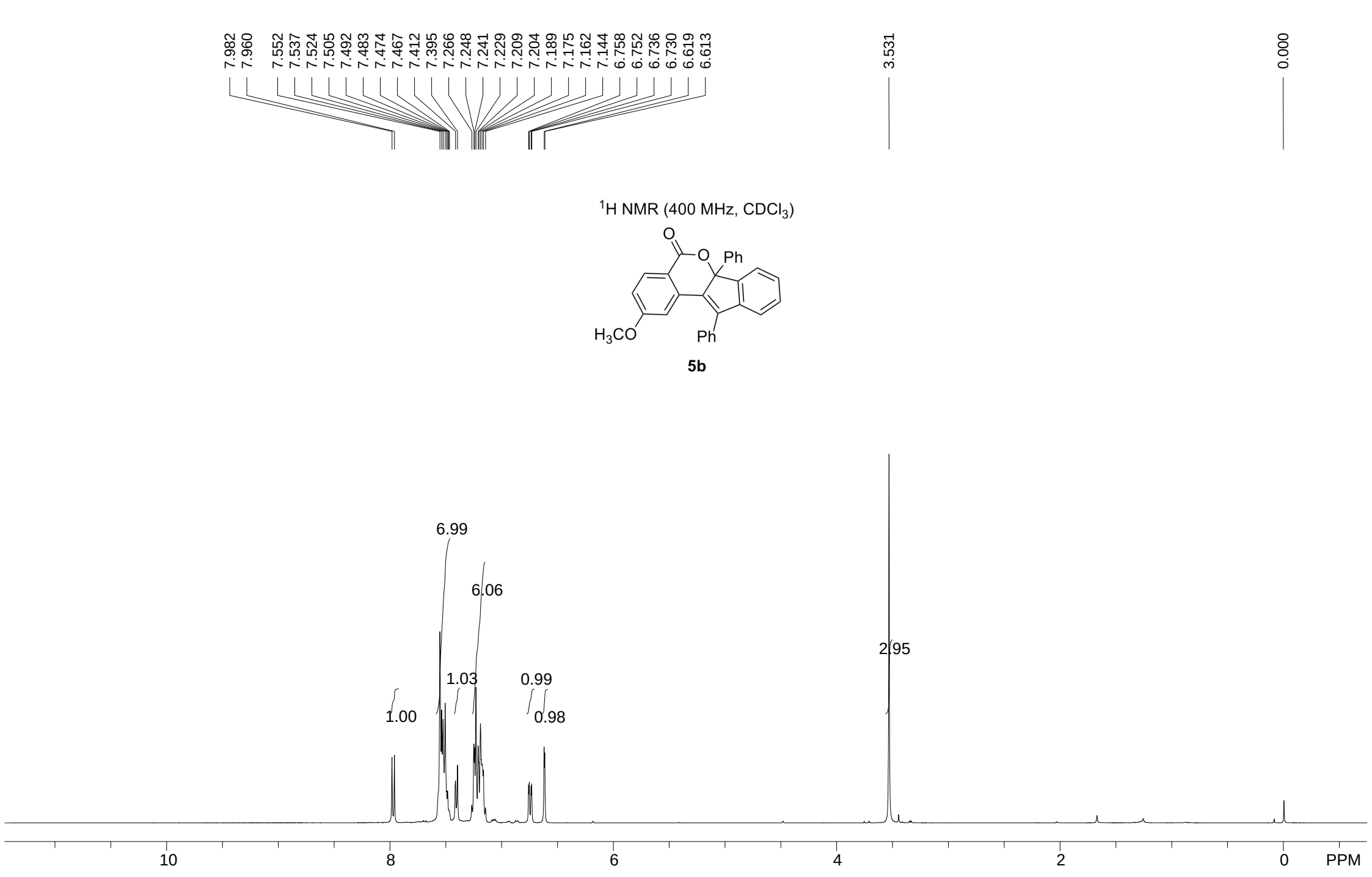


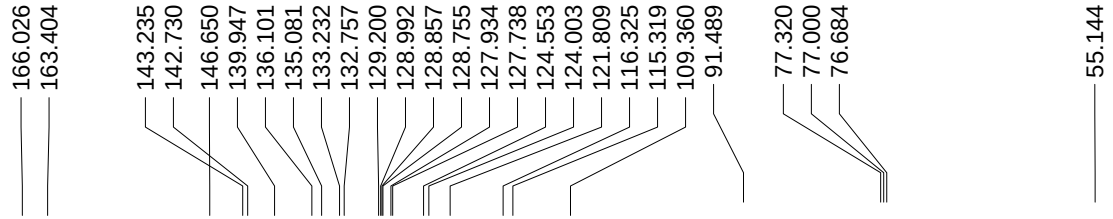
^{1}H NMR (400 MHz, $CDCl_3$)

5a

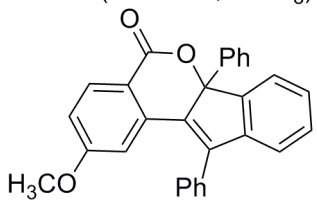

166.206
143.356
142.560
139.838
146.483
135.727
133.575
133.302
132.752
131.062
129.286
129.082
128.908
128.720
128.507
128.022
127.727
125.505
124.642
124.061
123.742
121.848
91.636
77.318
77.000
76.682
^{13}C NMR (100 MHz, $CDCl_3$)
O
O
Ph
Ph
200
150
100
50
0
PPM

5a

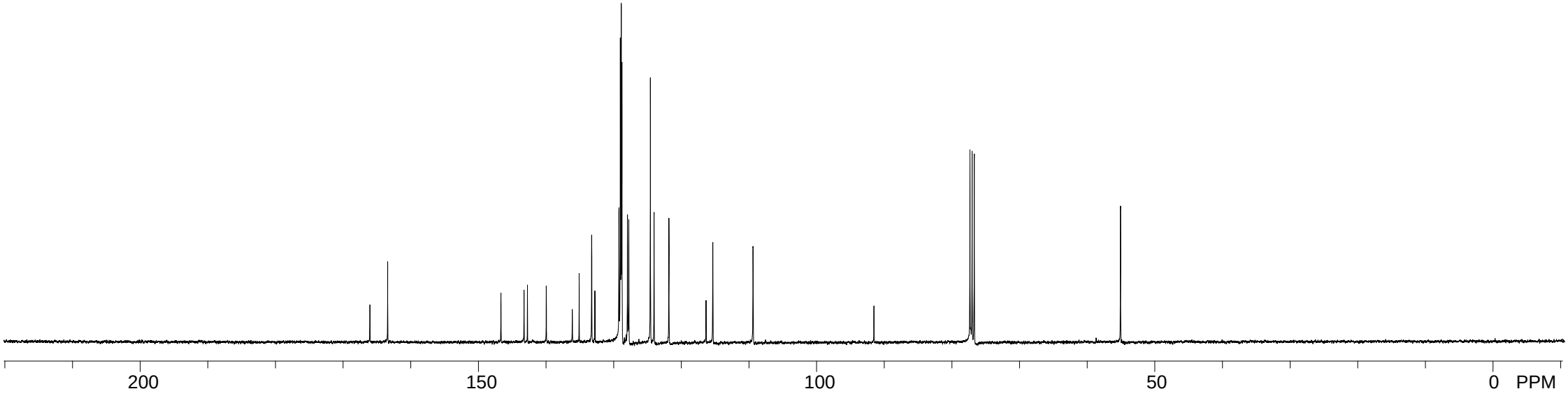

7.982
7.960
7.552
7.537
7.524
7.505
7.492
7.483
7.474
7.467
7.412
7.395
7.266
7.248
7.241
7.229
7.209
7.204
7.189
7.175
7.162
7.144
6.758
6.752
6.736
6.730
6.619
6.613
3.531
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
O
Ph
H_3CO
Ph
5b
6.99
6.06
1.03
0.99
1.00
0.98
2.95
10
8
6
4
2
0
PPM

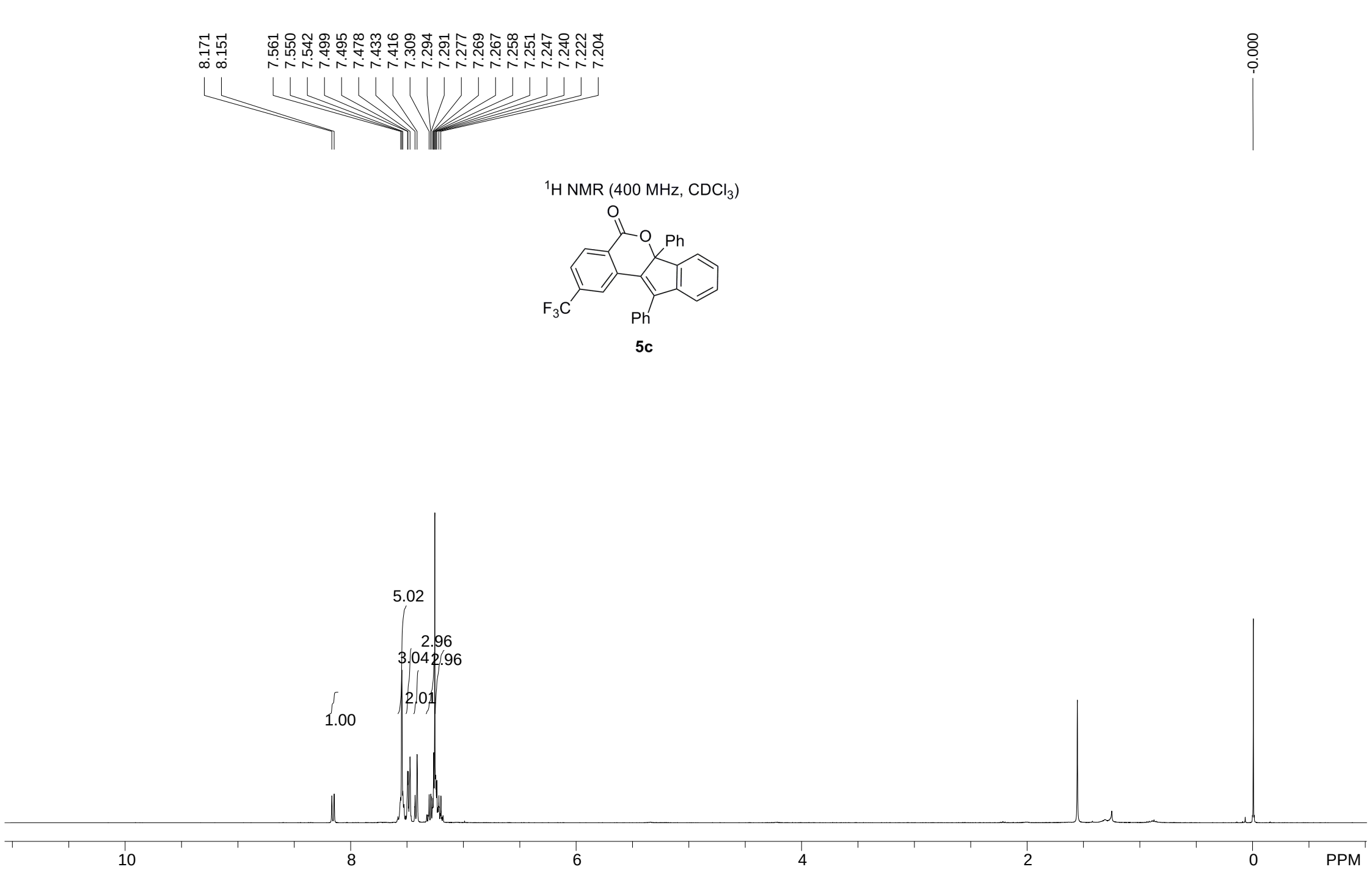


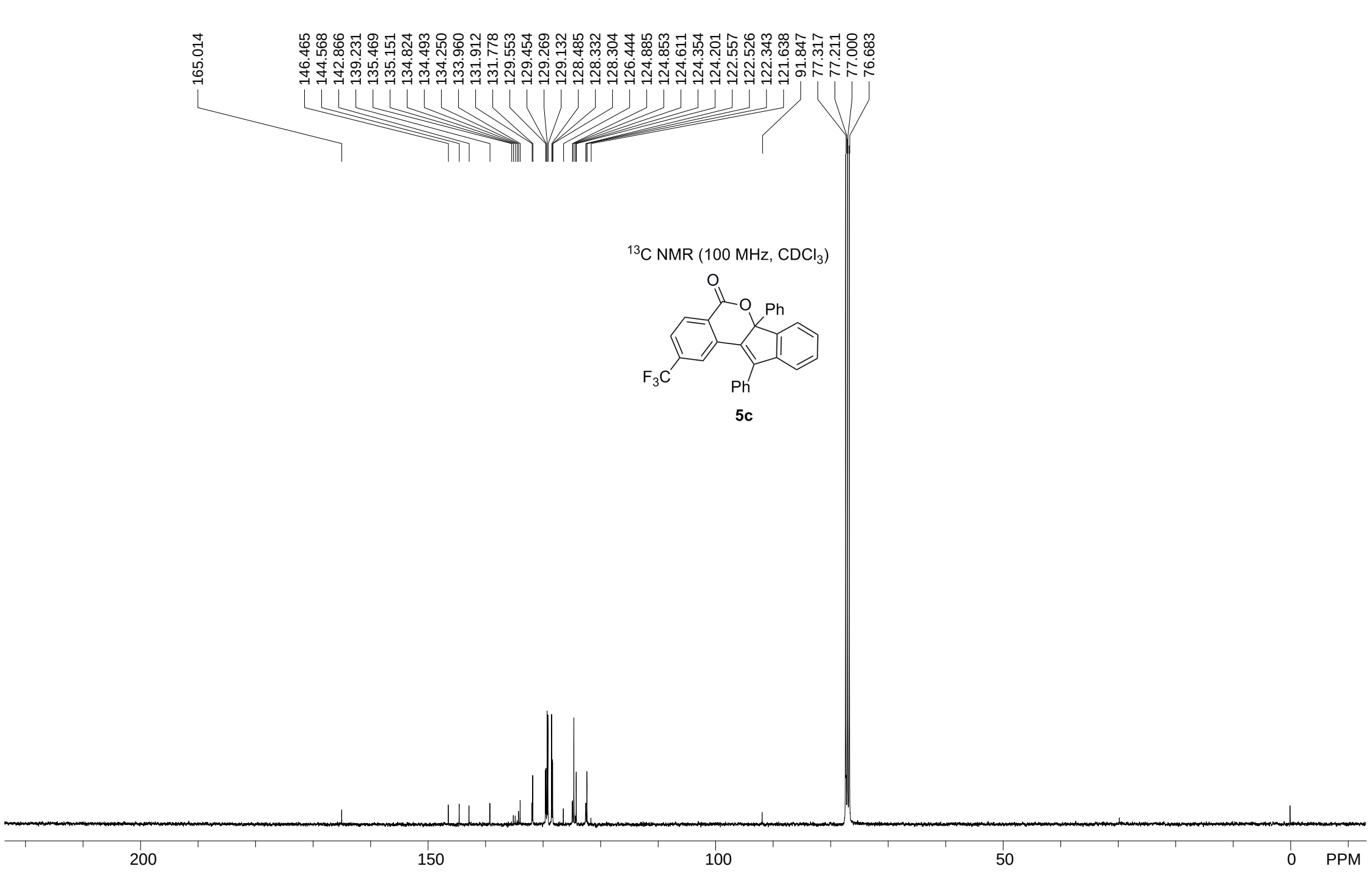
^{13}C NMR (100 MHz, $CDCl_3$)



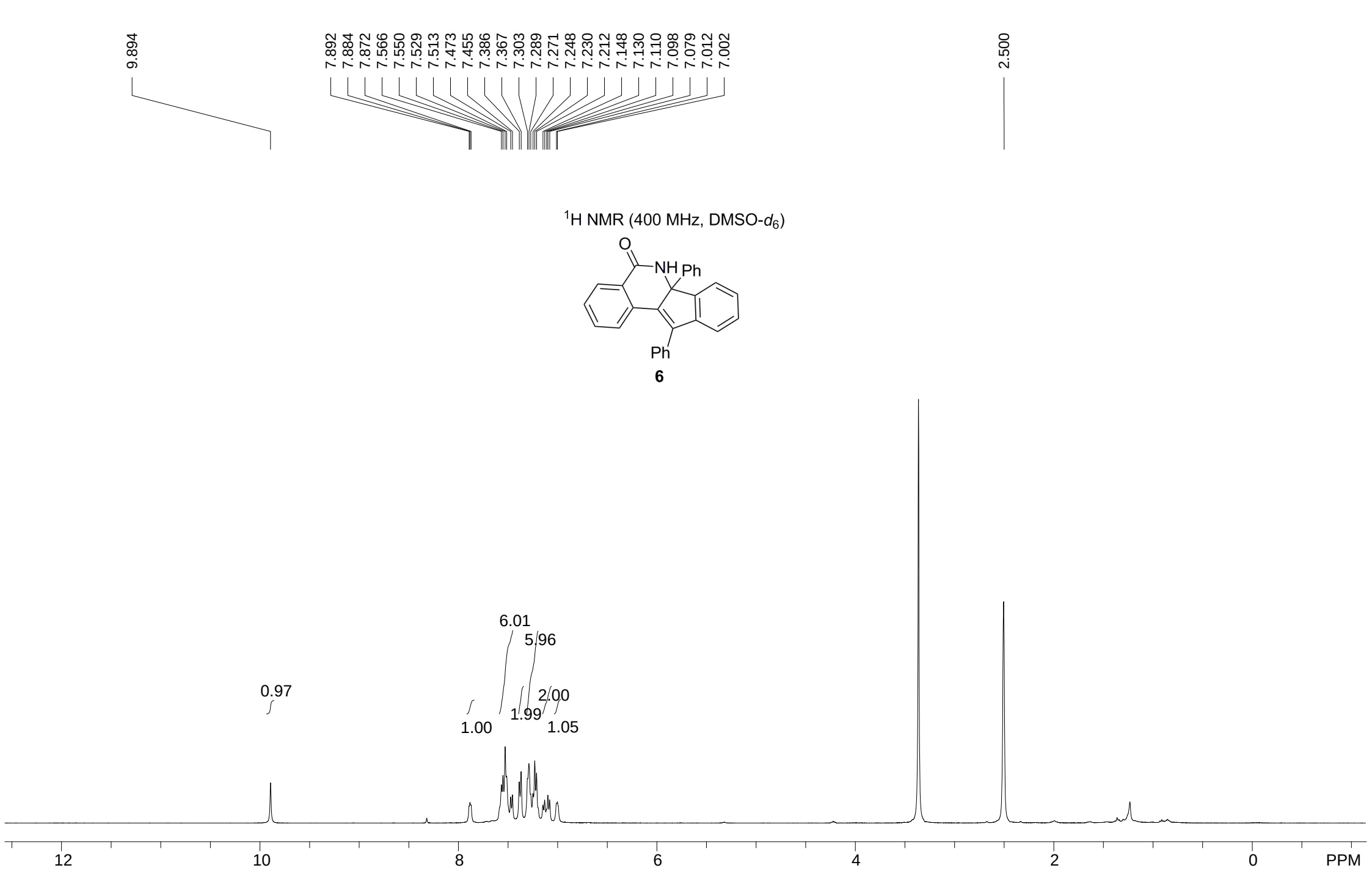
5b

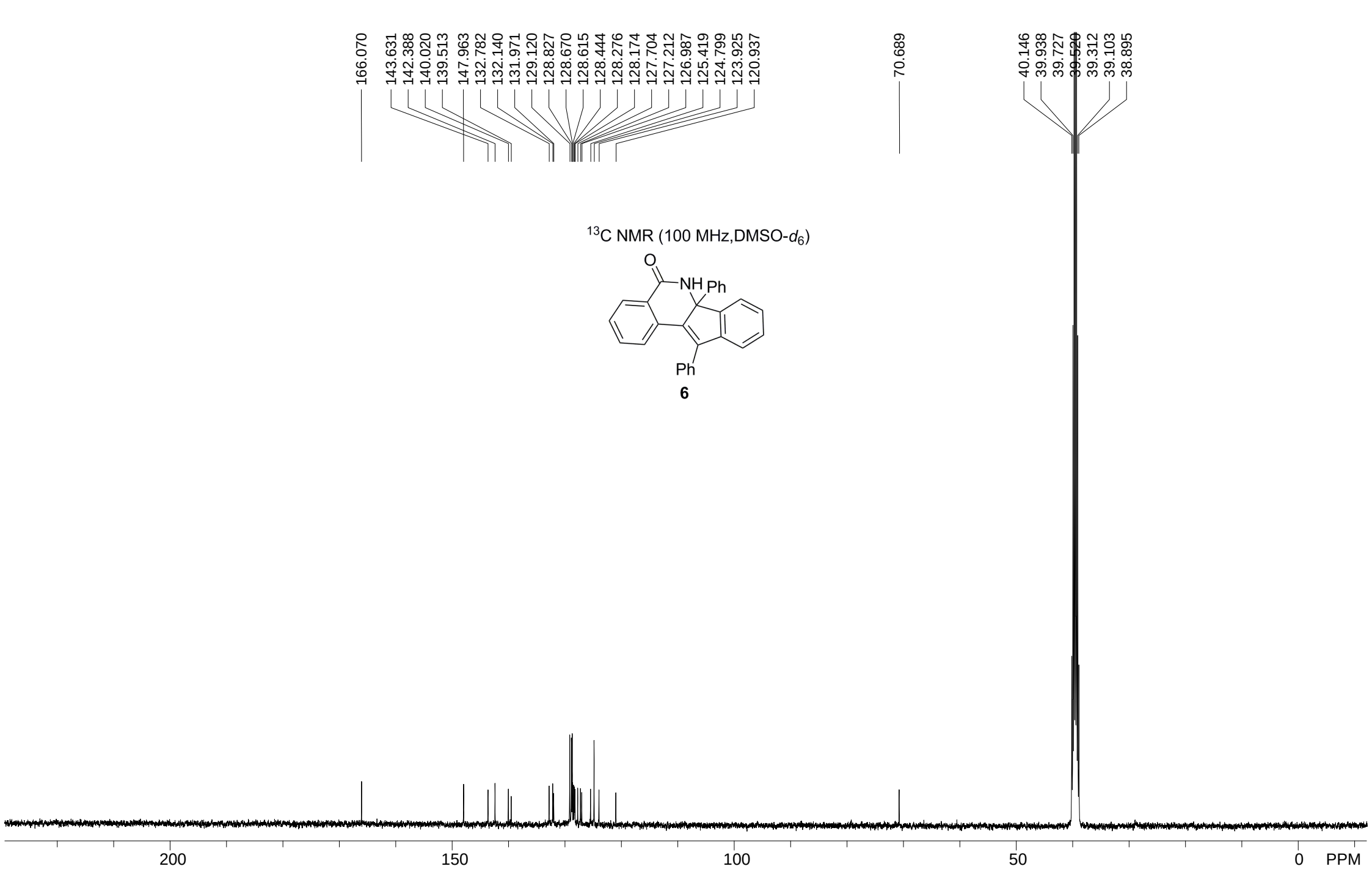



8.171
8.151
7.561
7.550
7.542
7.499
7.495
7.478
7.433
7.416
7.309
7.294
7.291
7.277
7.269
7.267
7.258
7.251
7.247
7.240
7.222
7.204
-0.000
1H NMR (400 MHz, CDCl3)
O
O
Ph
F3C
Ph
5c
5.02
2.96
3.04
2.96
2.01
1.00
10
8
6
4
2
0
PPM

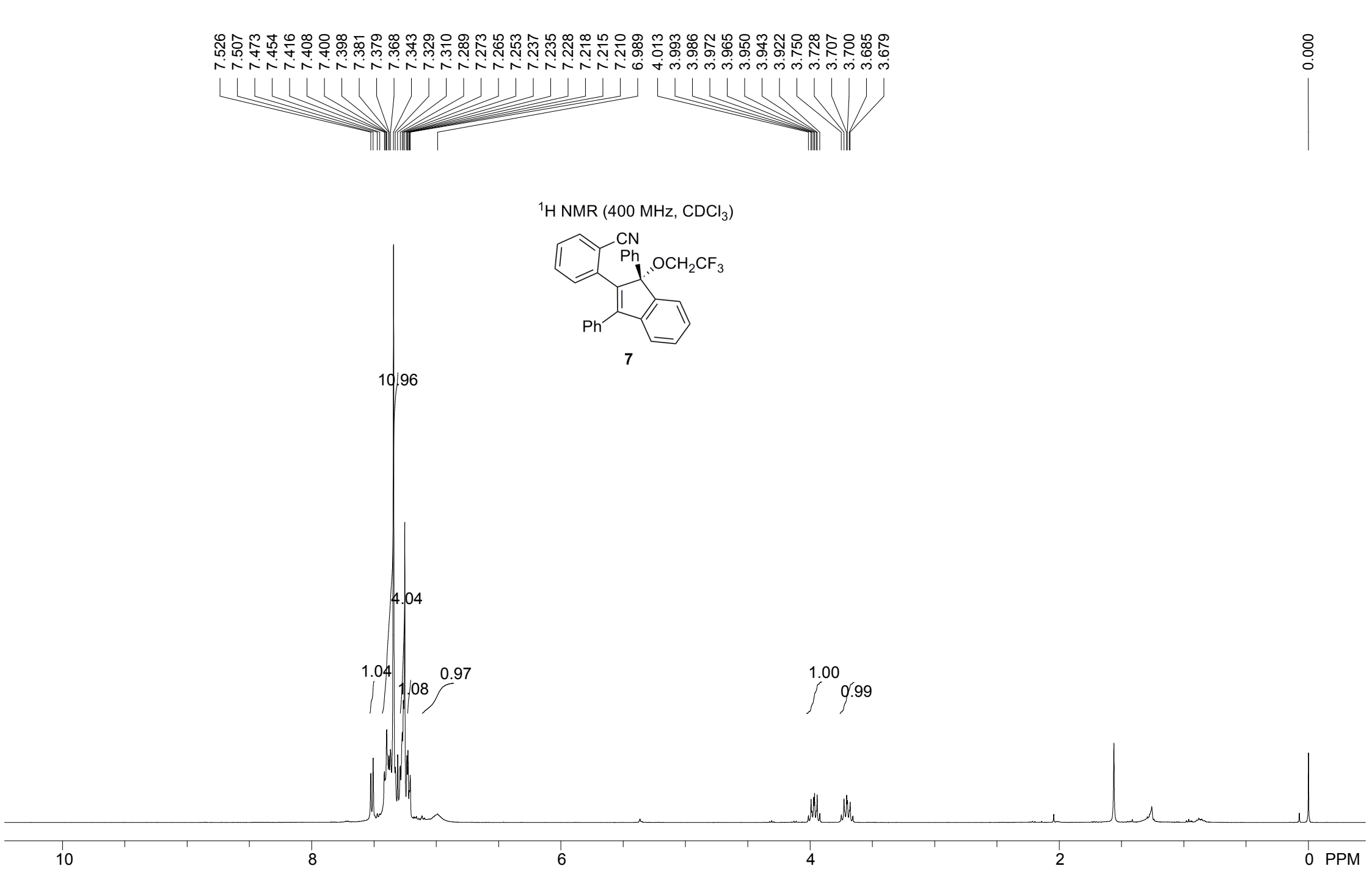

165.014
146.465
144.568
142.866
139.231
135.469
135.151
134.824
134.493
134.250
133.960
131.912
131.778
129.553
129.454
129.269
129.132
128.485
128.332
128.304
126.444
124.885
124.853
124.611
124.354
124.201
122.557
122.526
122.343
121.638
91.847
77.317
77.211
77.000
76.683
^{13}C NMR (100 MHz, $CDCl_3$)
O
O
Ph
F_3C
Ph
200
150
100
50
0
PPM

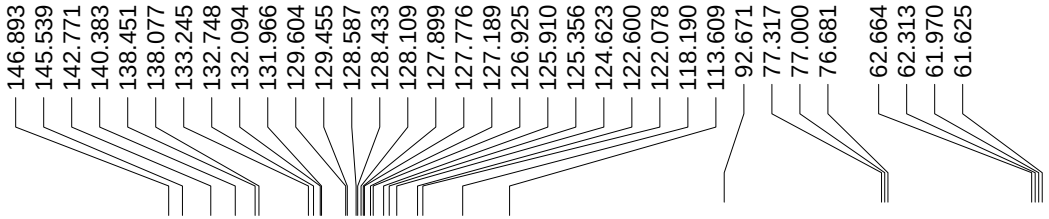
5c


9.894
7.892
7.884
7.872
7.566
7.550
7.529
7.513
7.473
7.455
7.386
7.367
7.303
7.289
7.271
7.248
7.230
7.212
7.148
7.130
7.110
7.098
7.079
7.012
7.002
2.500
^{1}H NMR (400 MHz, DMSO-d_6)
O
NH
Ph
Ph
6
6.01
5.96
0.97
2.00
1.99
1.00
1.05
12
10
8
6
4
2
0
PPM

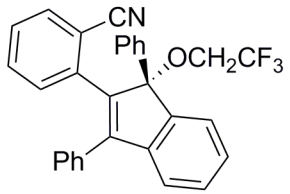

166.070
143.631
142.388
140.020
139.513
147.963
132.782
132.140
131.971
129.120
128.827
128.670
128.615
128.444
128.276
128.174
127.704
127.212
126.987
125.419
124.799
123.925
120.937
70.689
40.146
39.938
39.727
39.520
39.312
39.103
38.895
^{13}C NMR (100 MHz,DMSO-d_6)
O
NH
Ph
Ph
200
150
100
50
0
PPM

6

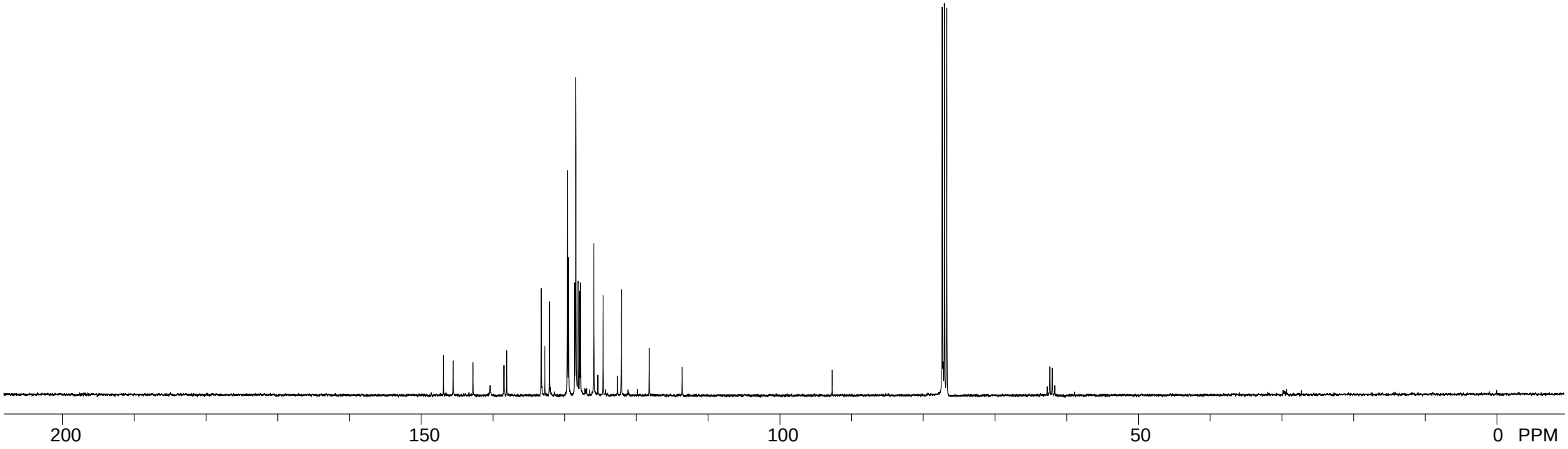

7.526
7.507
7.473
7.454
7.416
7.408
7.400
7.398
7.381
7.379
7.368
7.343
7.329
7.310
7.289
7.273
7.265
7.253
7.237
7.235
7.228
7.218
7.215
7.210
6.989
4.013
3.993
3.986
3.972
3.965
3.950
3.943
3.922
3.750
3.728
3.707
3.700
3.685
3.679
0.000
1H NMR (400 MHz, CDCl3)
CN
Ph
OCH2CF3
Ph
7
10.96
4.04
1.04
1.08
0.97
1.00
0.99
10
8
6
4
2
0
PPM

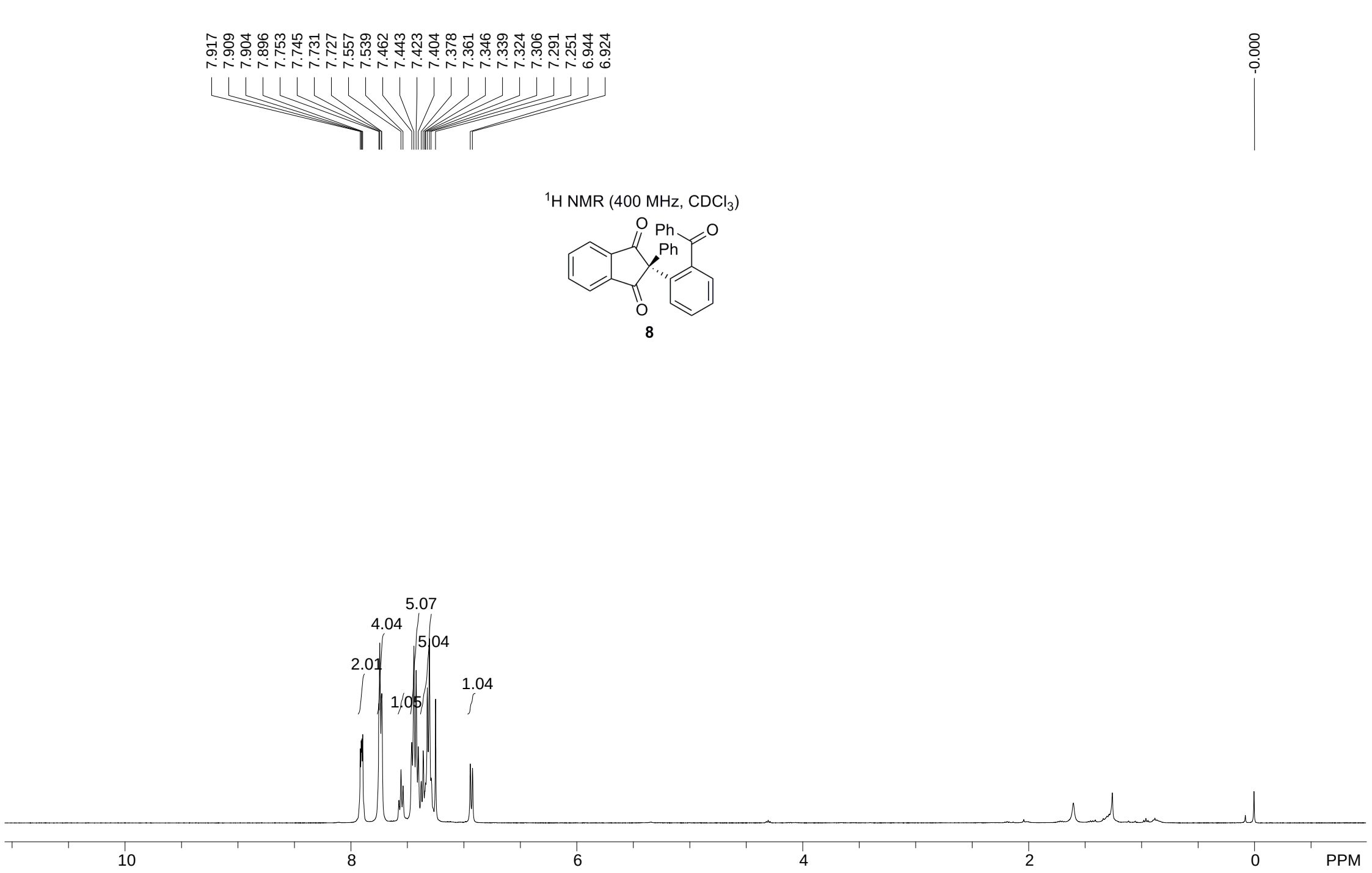


^{13}C NMR (100 MHz, $CDCl_3$)



7



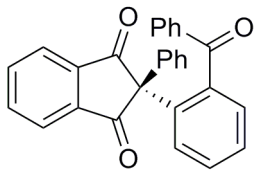

7.917
7.909
7.904
7.896
7.753
7.745
7.731
7.727
7.557
7.539
7.462
7.443
7.423
7.404
7.378
7.361
7.346
7.339
7.324
7.306
7.291
7.251
6.944
6.924
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
O
Ph
O
Ph
O
8
5.07
4.04
5.04
2.01
1.04
1.05
10
8
6
4
2
0
PPM

198.619
198.409

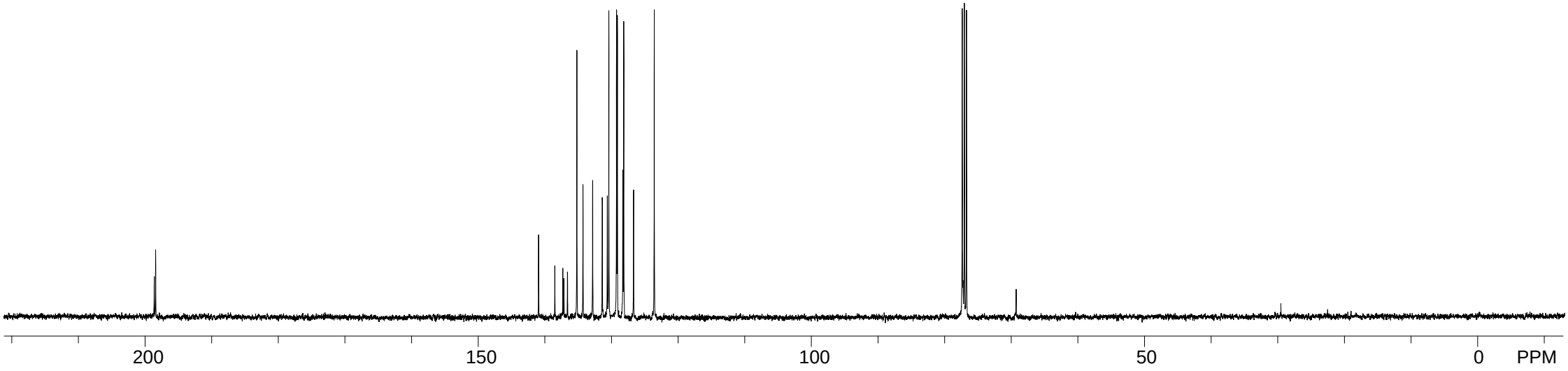
140.913
138.475
137.273
137.114
136.580
135.165
134.244
132.792
131.349
130.591
130.364
129.207
129.082
128.262
128.126
126.653
123.545

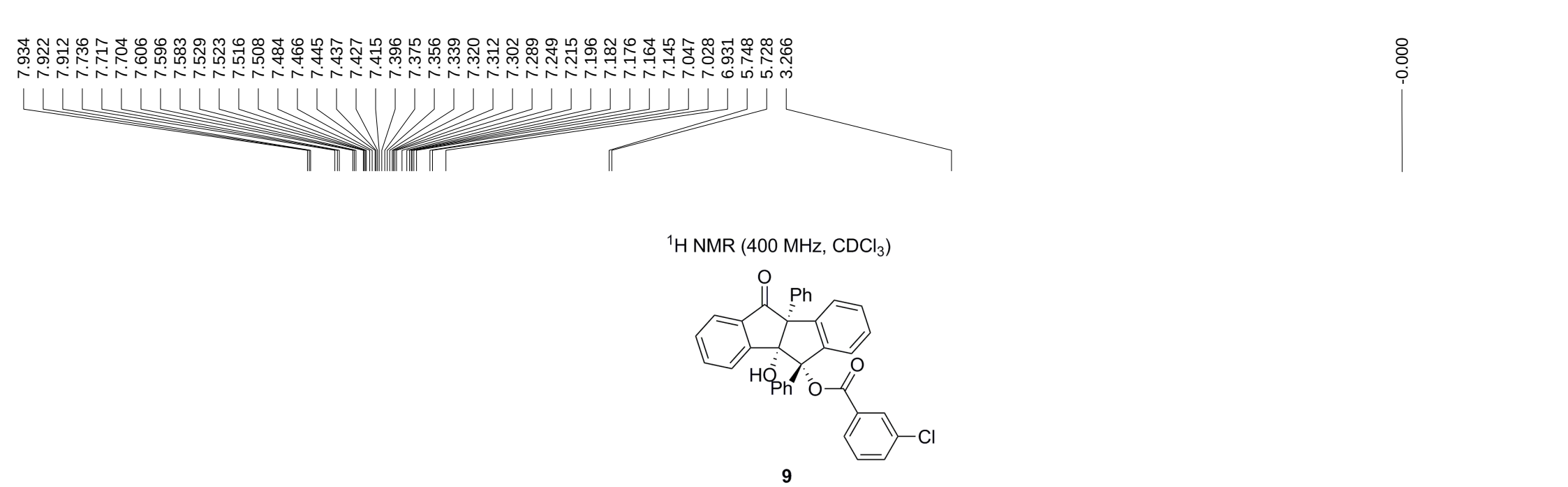
77.318
77.000
76.682
69.254

^{13}C NMR (100 MHz, $CDCl_3$)



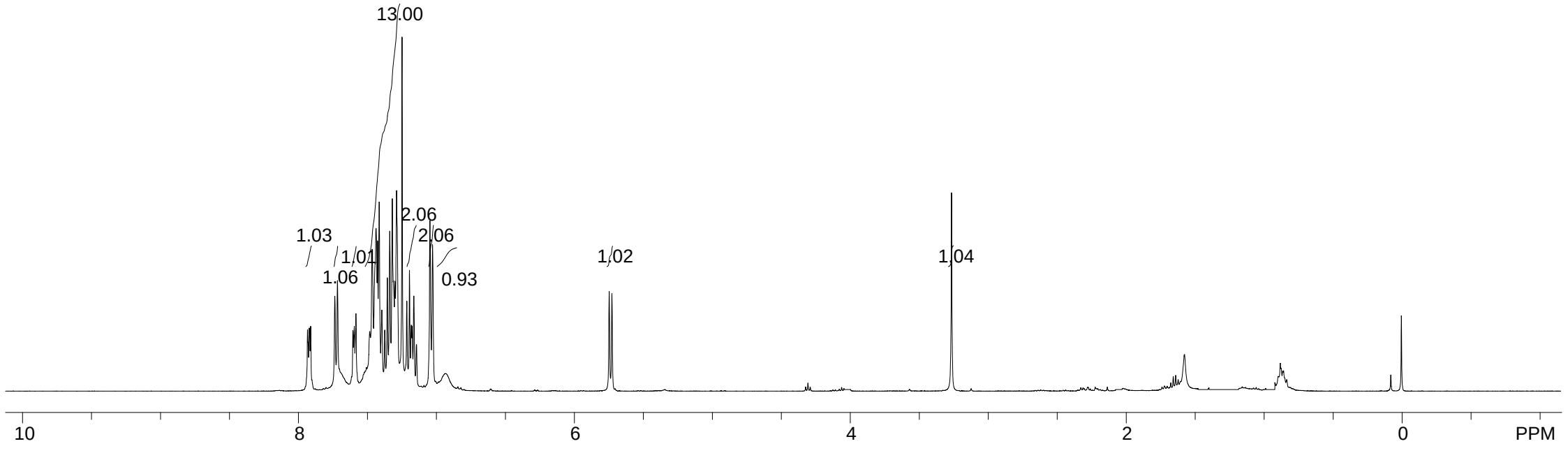
8





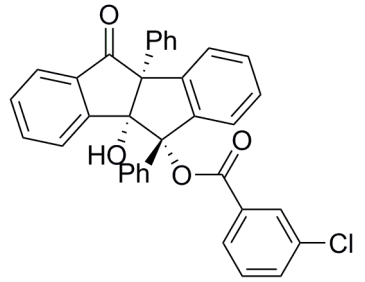
^{1}H NMR (400 MHz, $CDCl_3$)

9

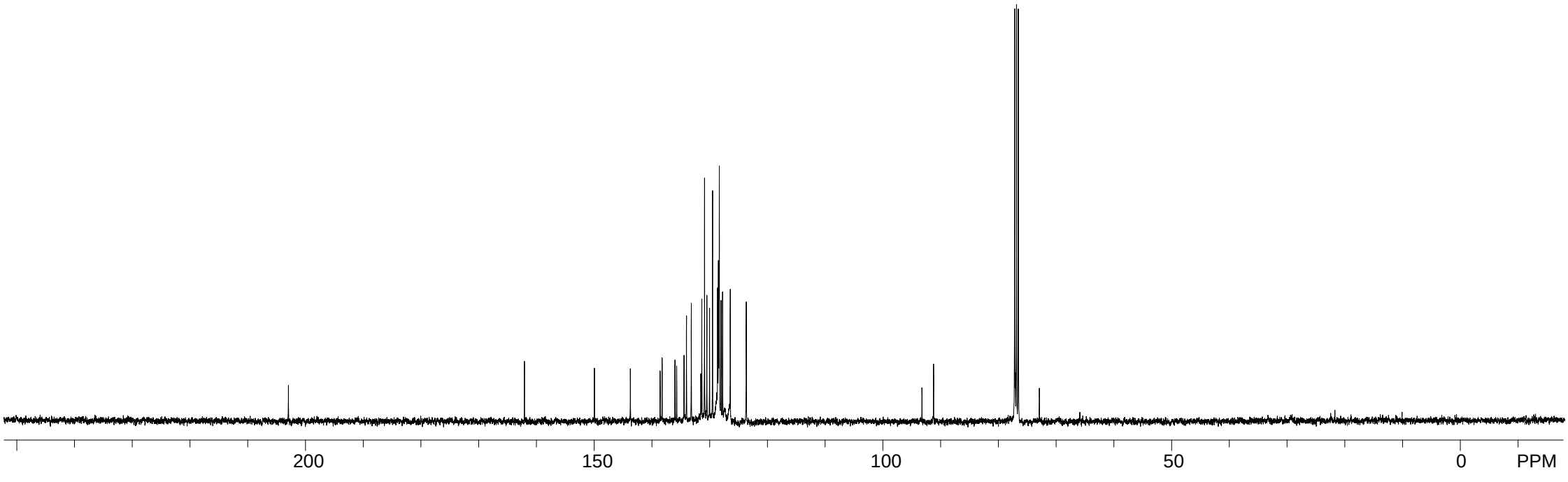


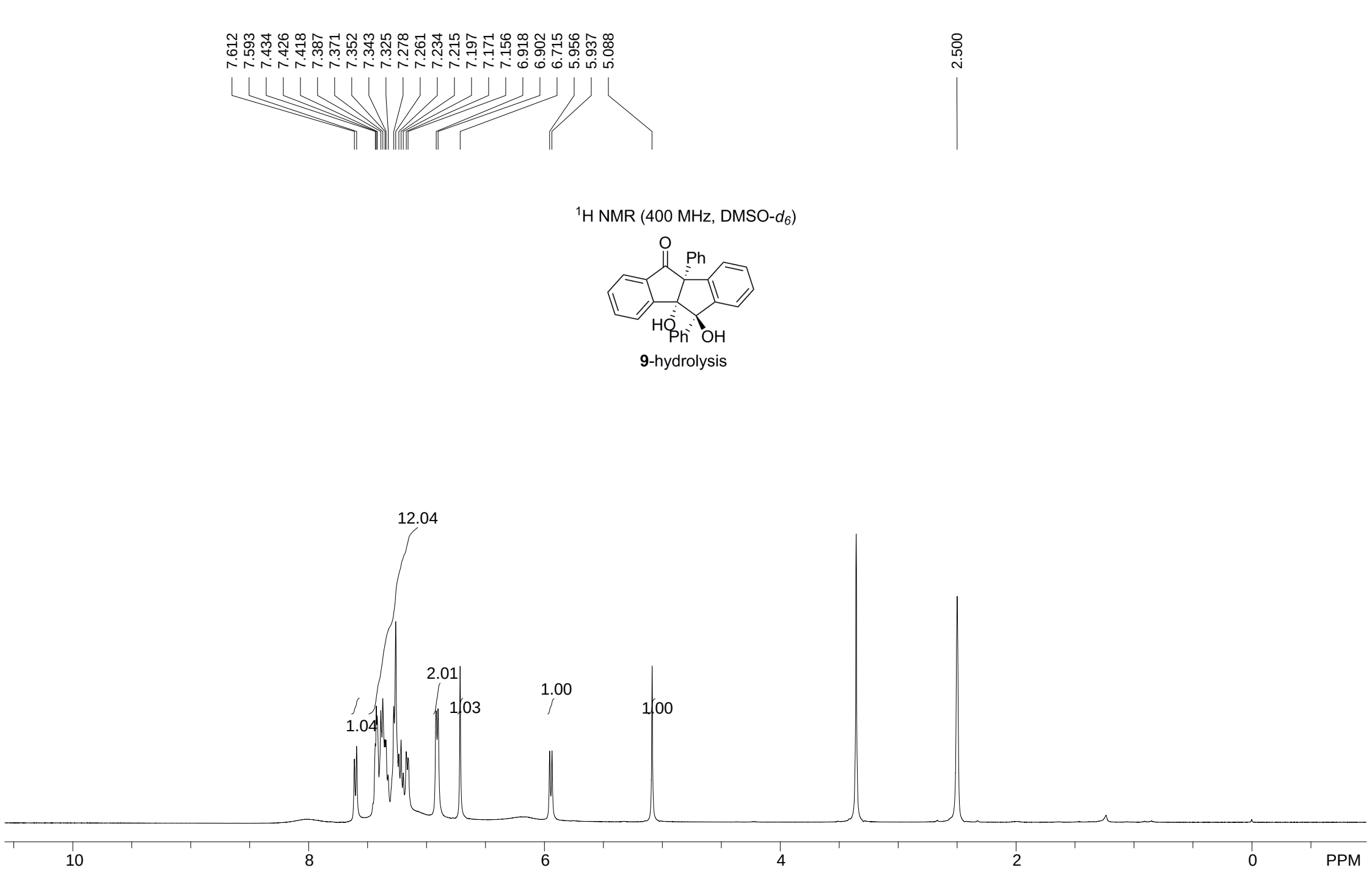
202.951
138.615
162.078
138.270
136.049
135.757
135.684
143.766
134.480
134.059
133.223
131.581
131.408
130.938
130.532
130.068
129.549
128.814
128.682
128.545
128.476
128.382
128.090
127.818
126.489
123.720
93.346
91.336
77.317
77.000
76.683
73.048

^{13}C NMR (100 MHz, $CDCl_3$)



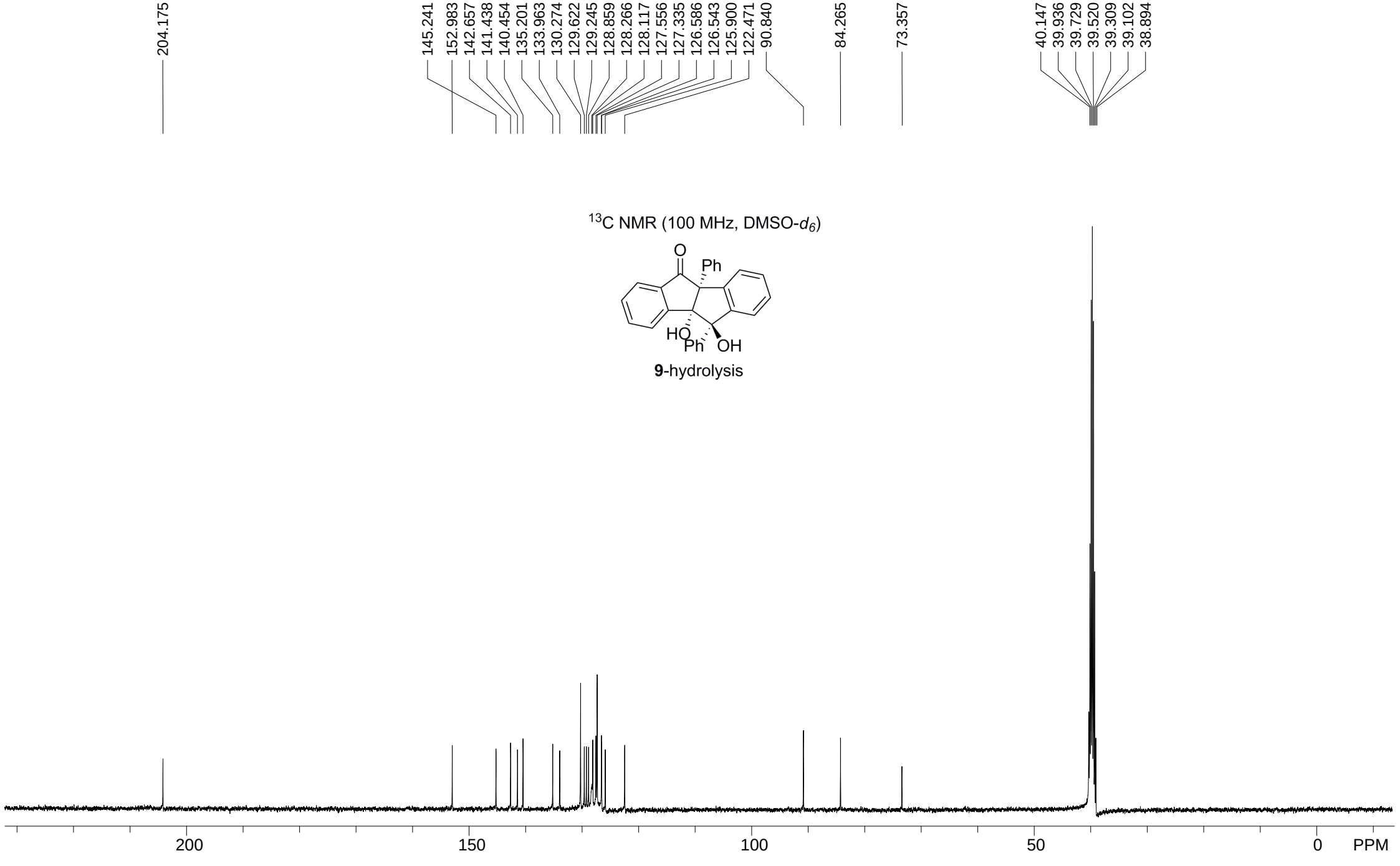
9

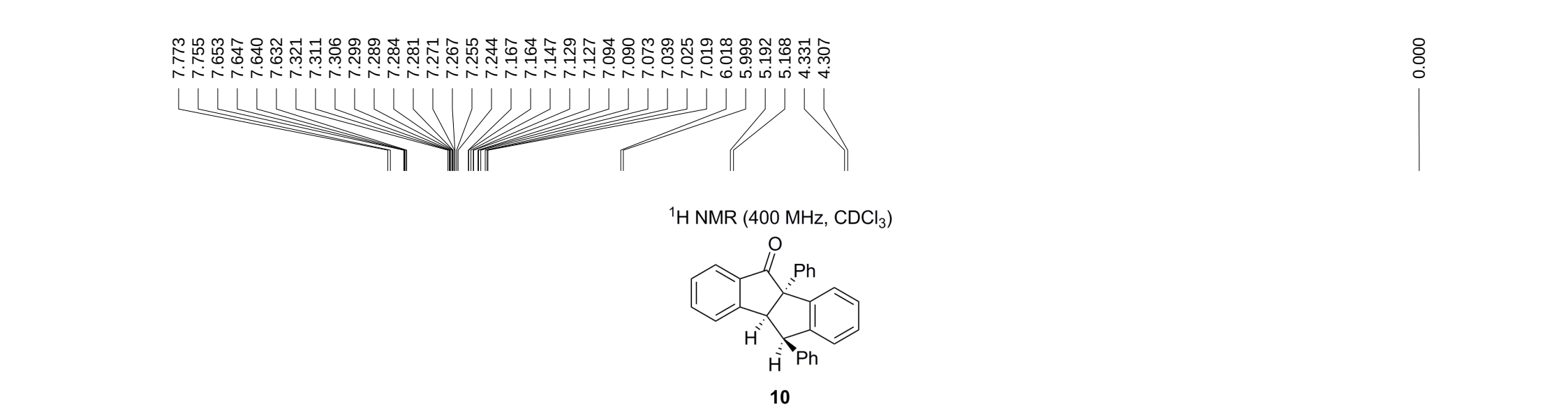




^{1}H NMR (400 MHz, DMSO-d_6)

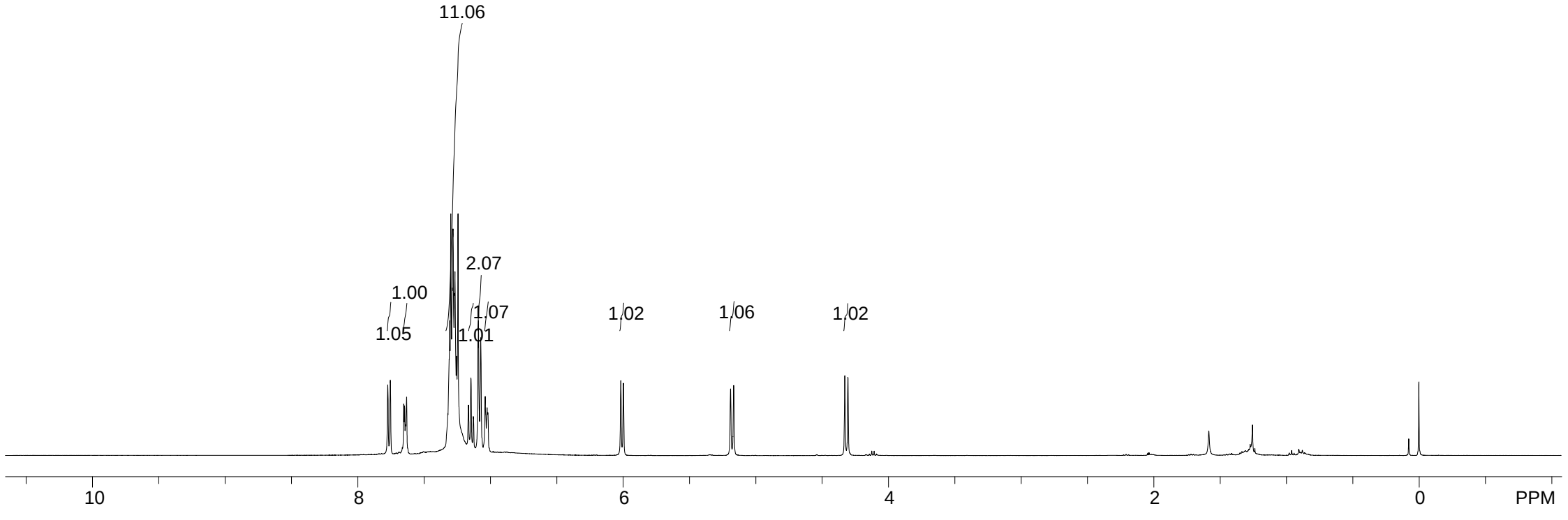
9-hydrolysis


204.175
145.241
152.983
142.657
141.438
140.454
135.201
133.963
130.274
129.622
129.245
128.859
128.266
128.117
127.556
127.335
126.586
126.543
125.900
122.471
90.840
84.265
73.357
40.147
39.936
39.729
39.520
39.309
39.102
38.894
^{13}C NMR (100 MHz, DMSO-d_6)
O
Ph
HO
Ph
OH
9-hydrolysis
200
150
100
50
0
PPM



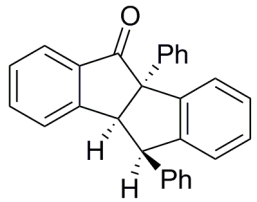
^{1}H NMR (400 MHz, $CDCl_3$)

10



205.747
145.310
152.200
143.027
142.351
139.354
136.675
133.654
130.359
128.846
128.627
128.536
128.111
127.792
127.684
127.457
127.216
126.998
126.347
125.886
123.966
77.317
77.000
76.683
70.762
60.235
53.026

^{13}C NMR (100 MHz, $CDCl_3$)



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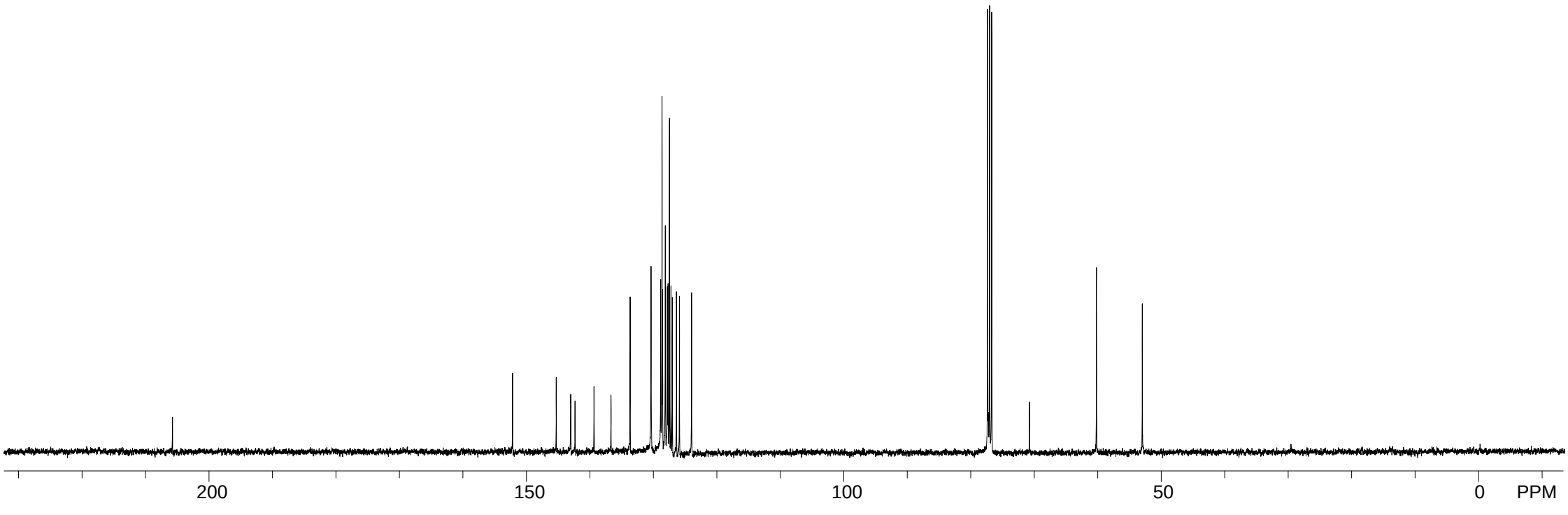
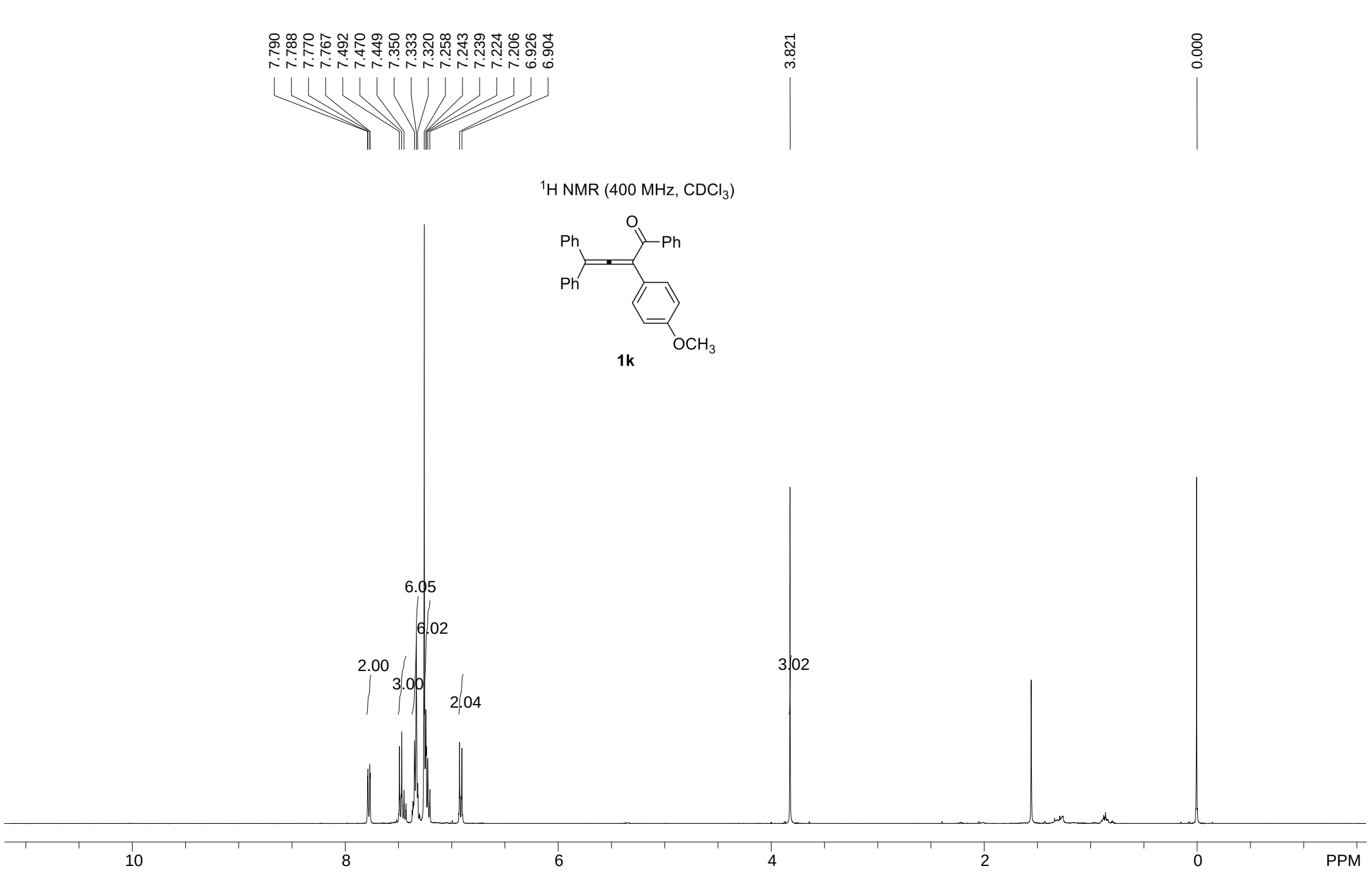
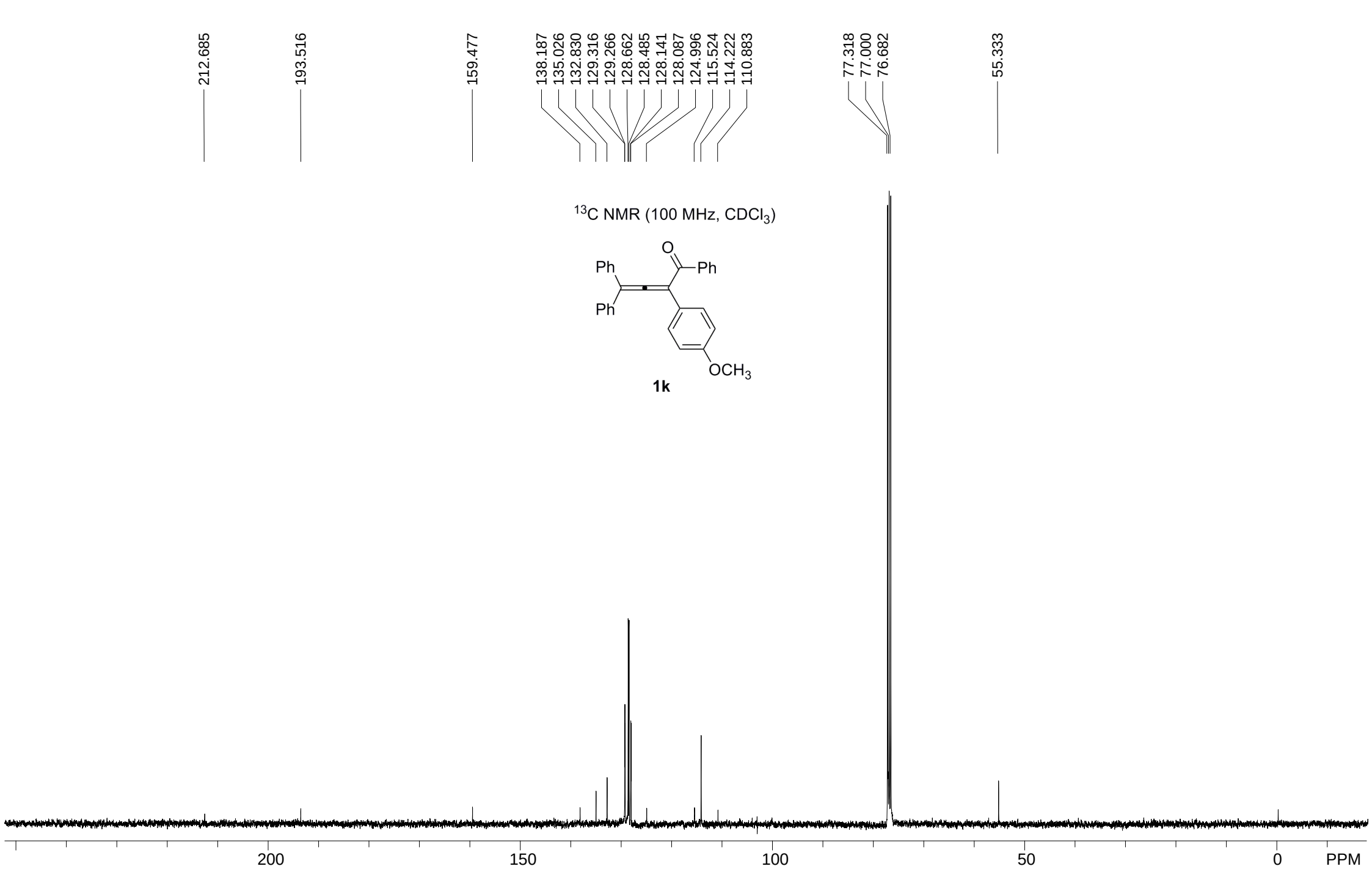
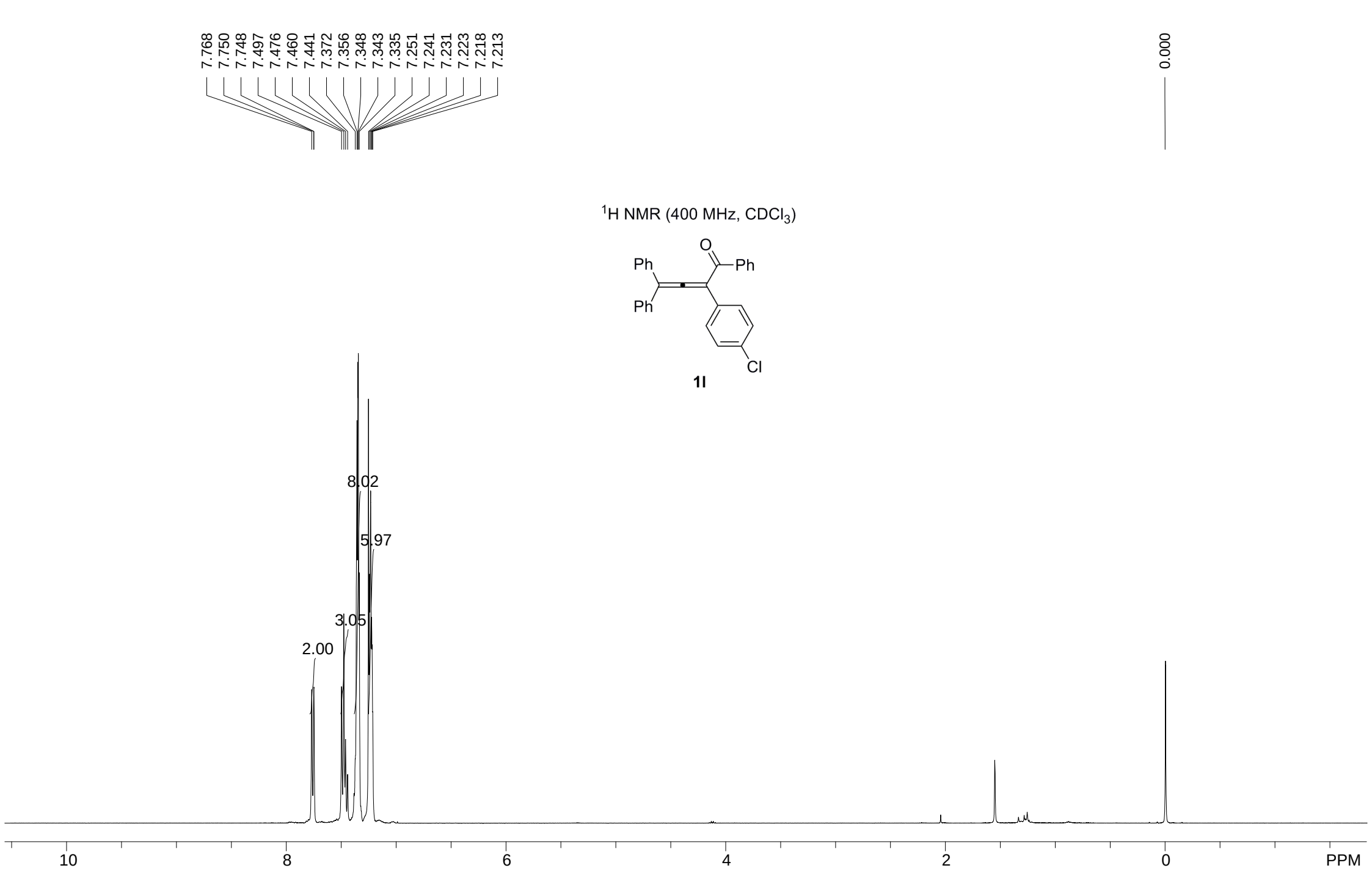
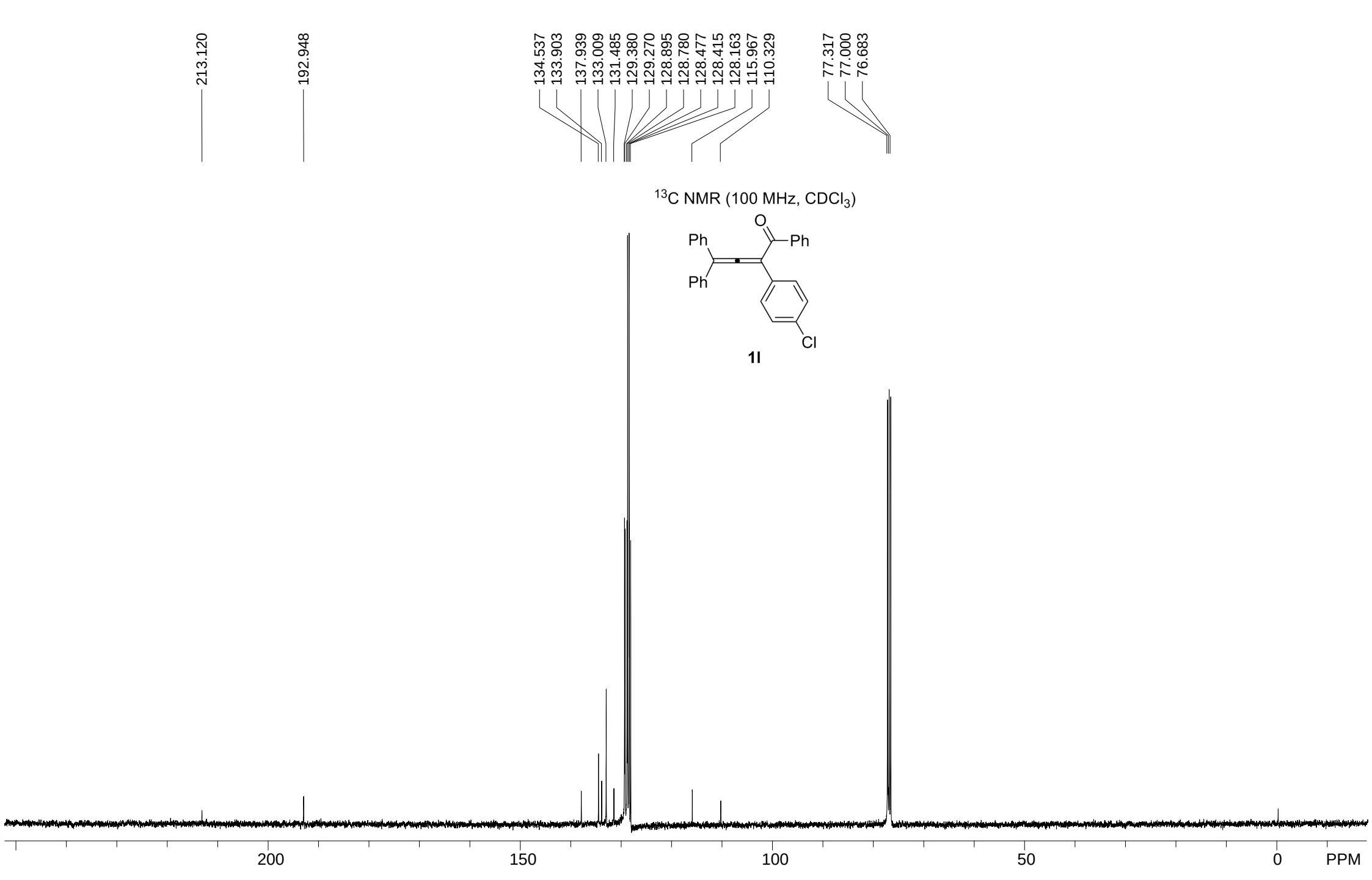
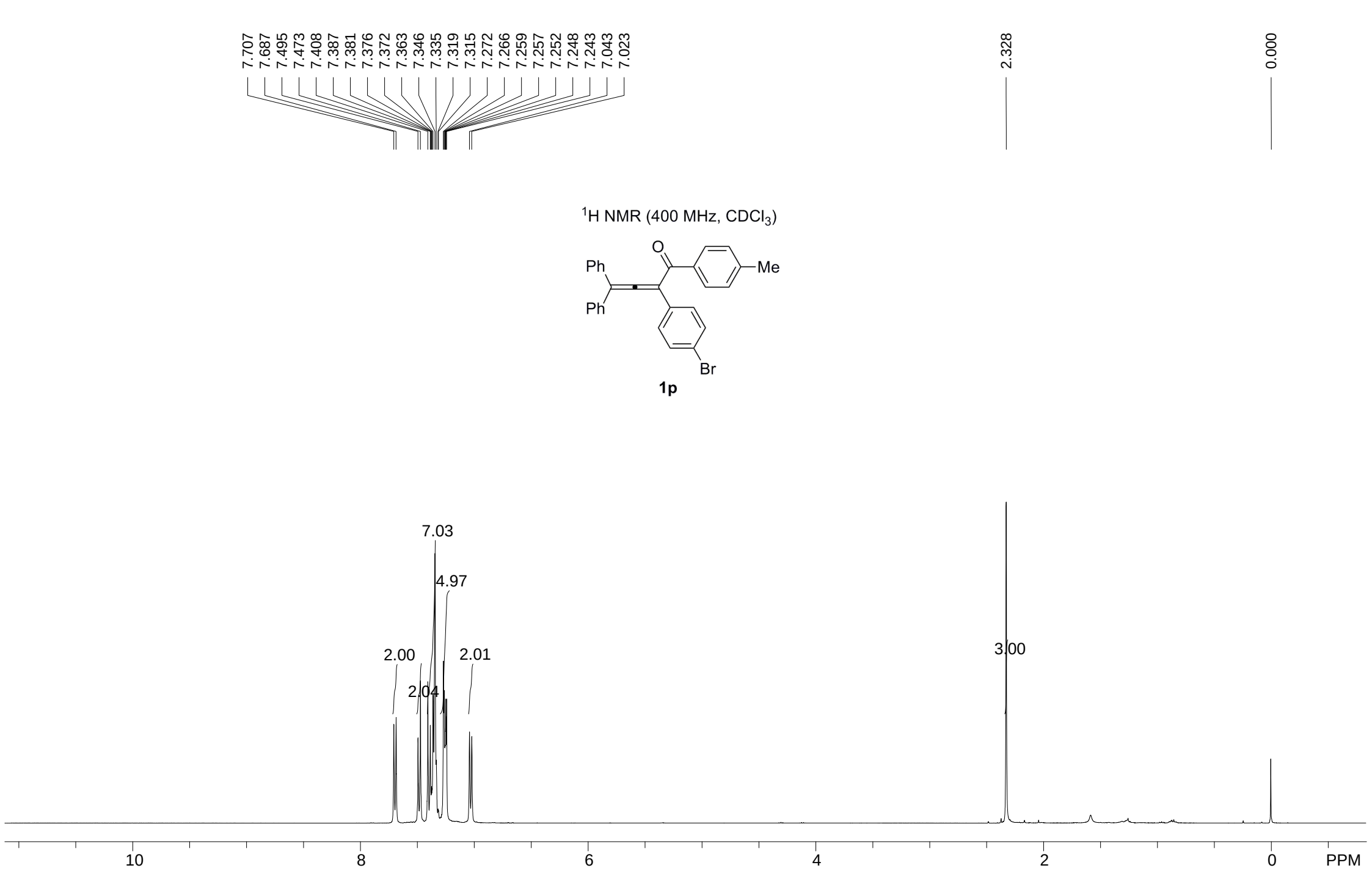


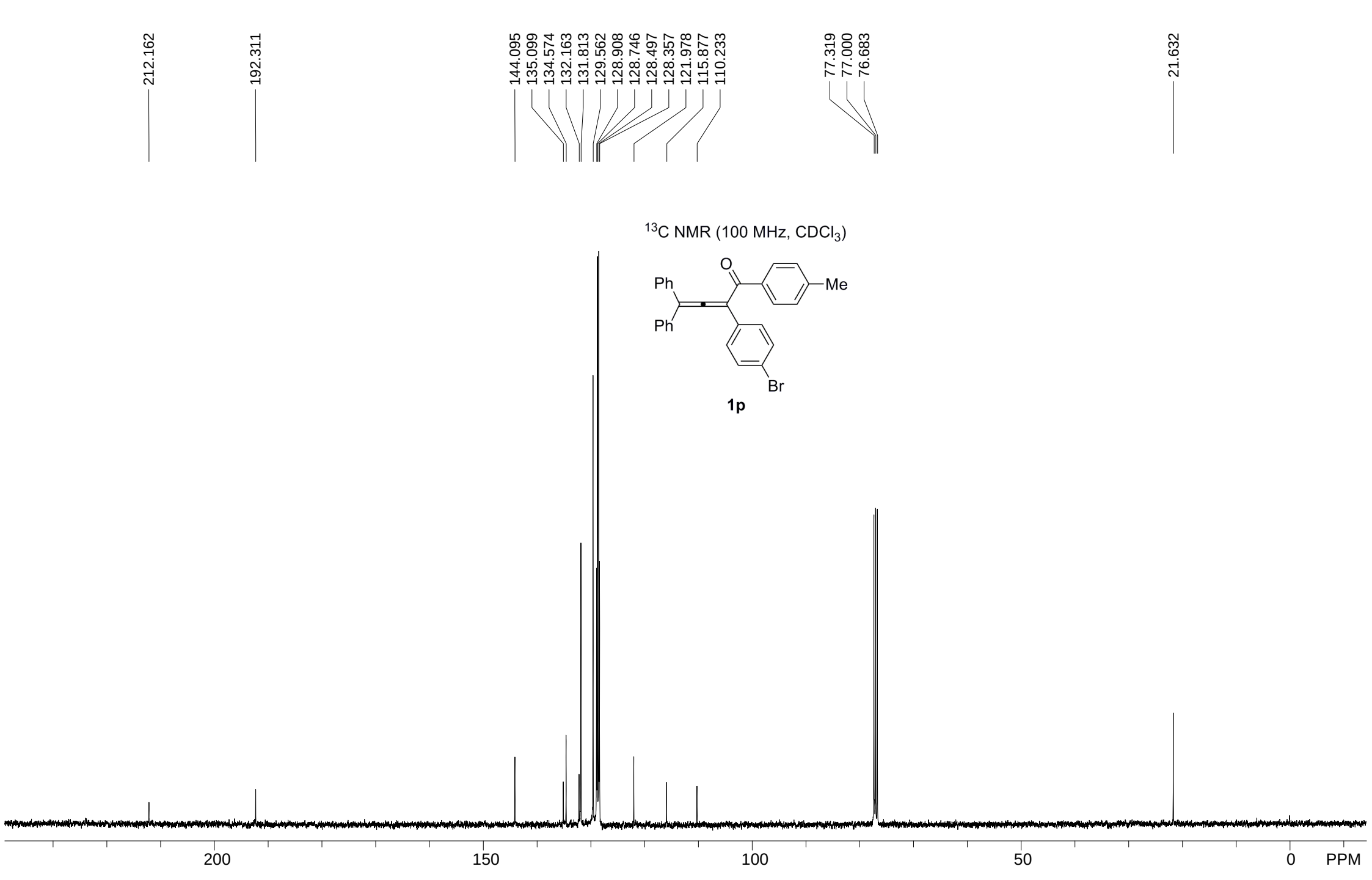

7.790
7.788
7.770
7.767
7.492
7.470
7.449
7.350
7.333
7.320
7.258
7.243
7.239
7.224
7.206
6.926
6.904
3.821
0.000
1H NMR (400 MHz, CDCl3)
O
Ph
Ph
Ph
OCH3
1k
6.05
6.02
2.00
3.00
2.04
3.02
10
8
6
4
2
0
PPM

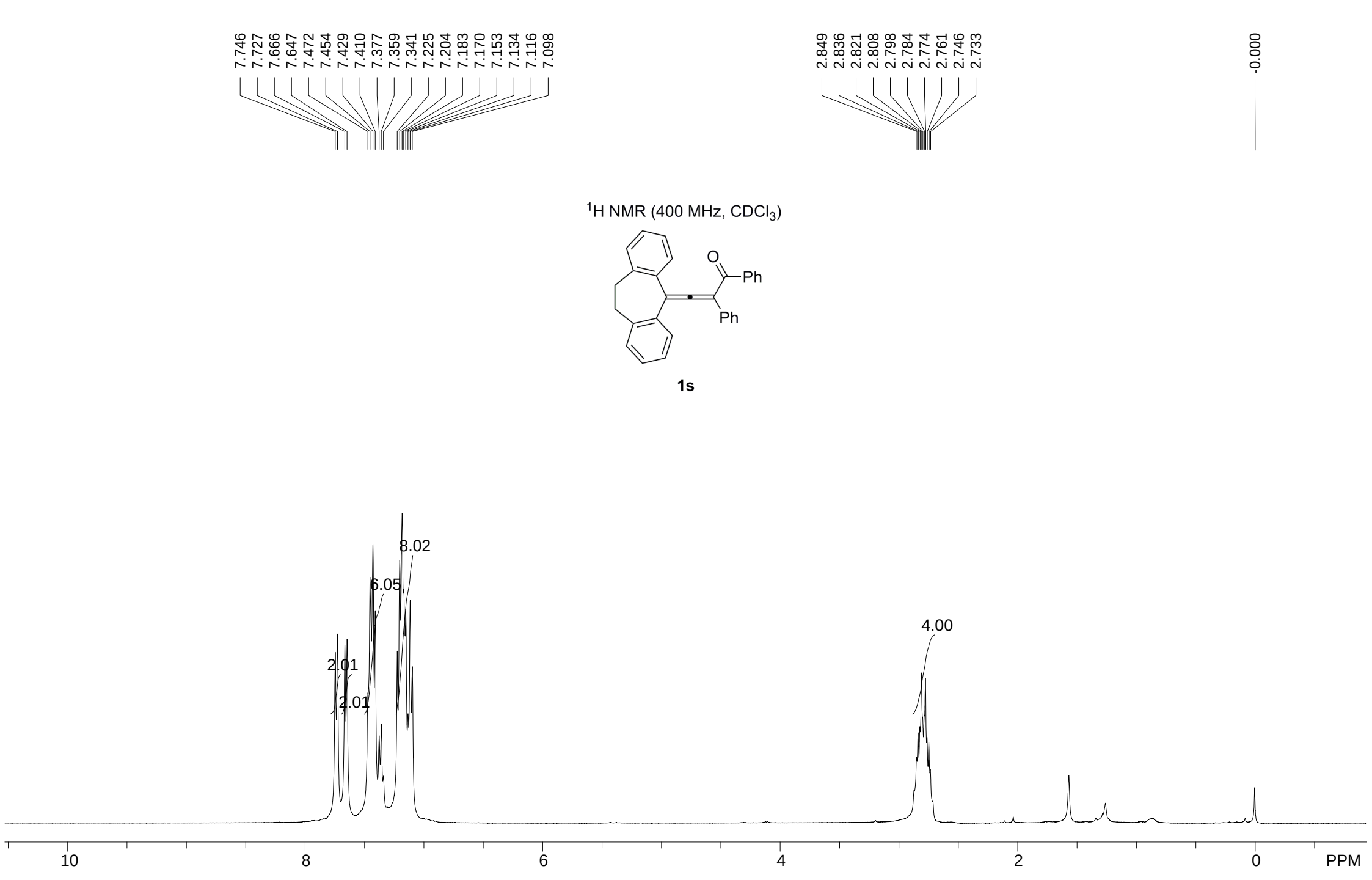

212.685
193.516
159.477
138.187
135.026
132.830
129.316
129.266
128.662
128.485
128.141
128.087
124.996
115.524
114.222
110.883
77.318
77.000
76.682
55.333
13C NMR (100 MHz, CDCl3)
O
Ph
Ph
Ph
OCH3
1k
200
150
100
50
0
PPM

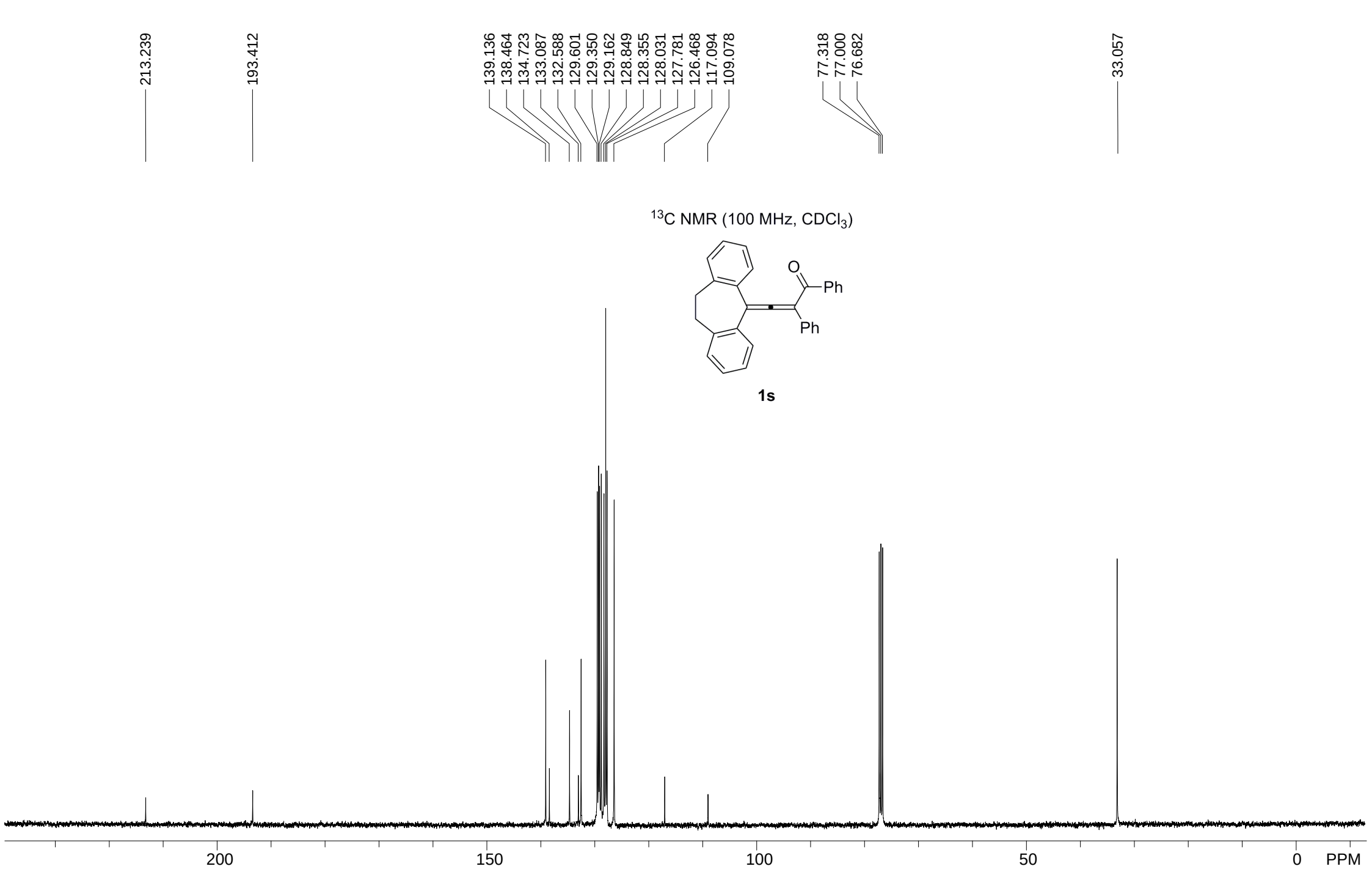

7.768
7.750
7.748
7.497
7.476
7.460
7.441
7.372
7.356
7.348
7.343
7.335
7.251
7.241
7.231
7.223
7.218
7.213
0.000
1H NMR (400 MHz, CDCl3)
O
Ph
Ph
Ph
Cl
1l
8.02
5.97
3.05
2.00
10
8
6
4
2
0
PPM

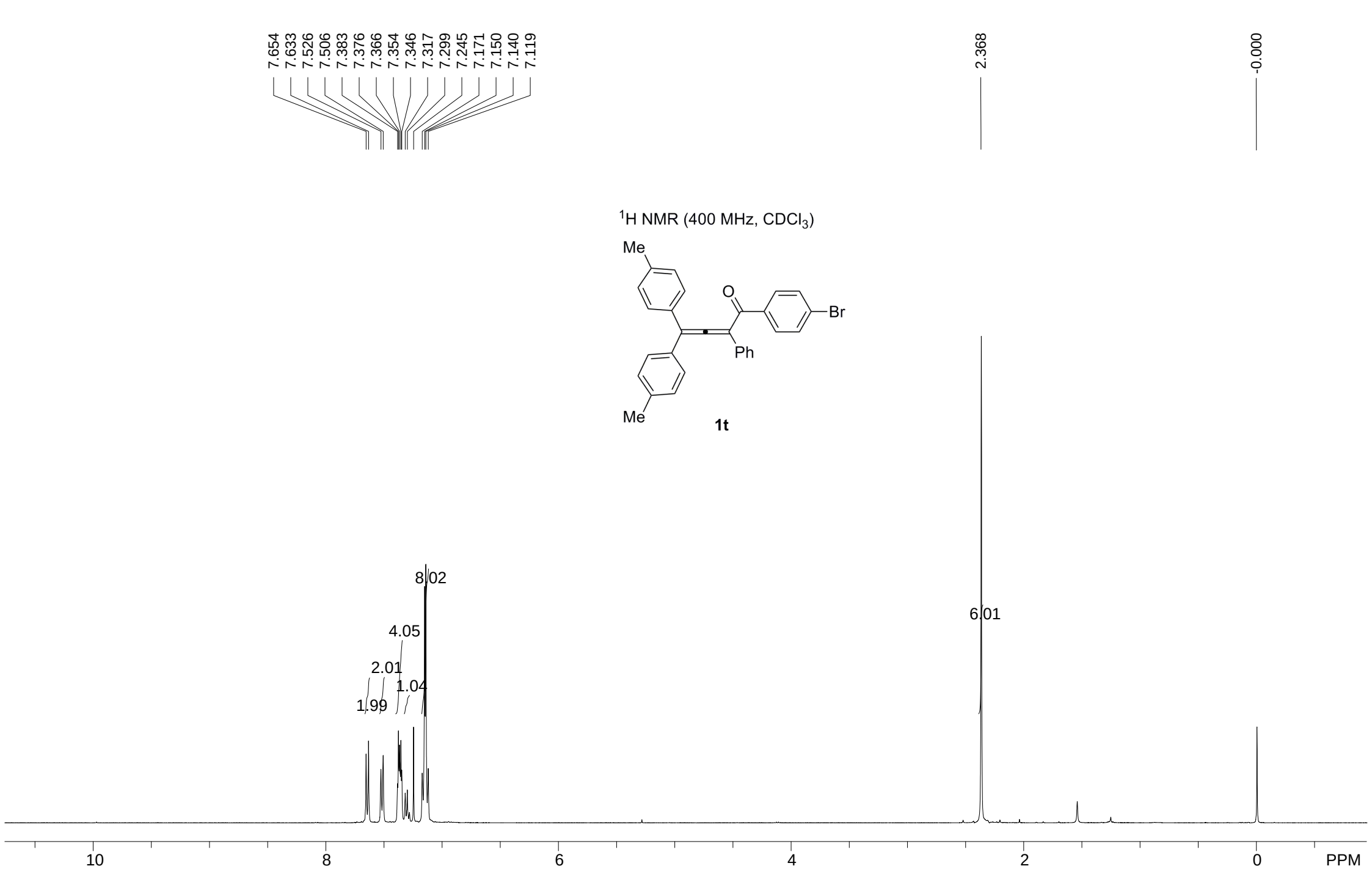

213.120
192.948
134.537
133.903
137.939
133.009
131.485
129.380
129.270
128.895
128.780
128.477
128.415
128.163
115.967
110.329
77.317
77.000
76.683
13C NMR (100 MHz, CDCl3)
O
Ph
Ph
Ph
Cl
1l
200
150
100
50
0
PPM

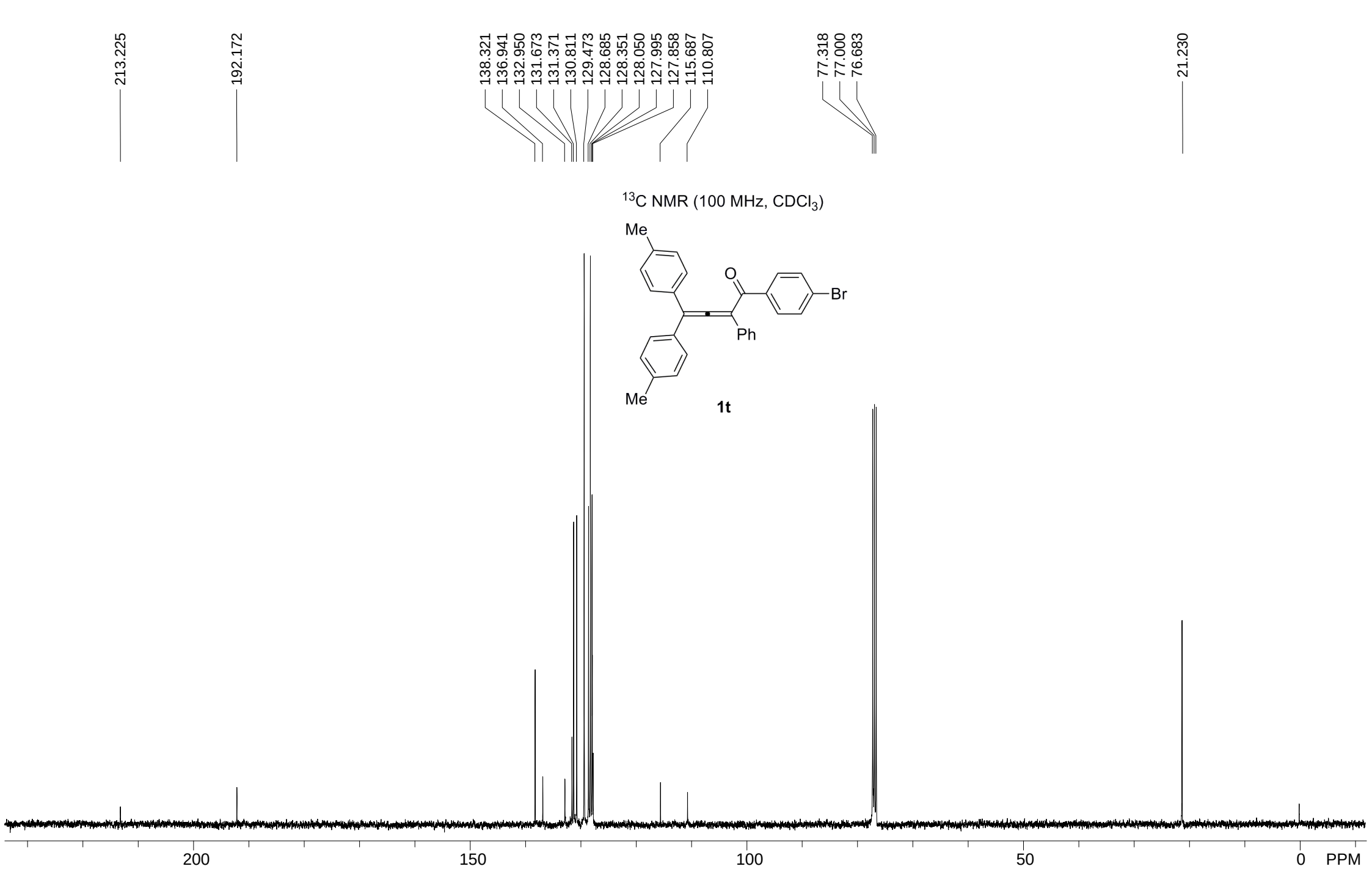

7.707
7.687
7.495
7.473
7.408
7.387
7.381
7.376
7.372
7.363
7.346
7.335
7.319
7.315
7.272
7.266
7.259
7.257
7.252
7.248
7.243
7.043
7.023
2.328
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
Ph
Ph
Me
Br
1p
7.03
4.97
2.00
2.01
2.04
3.00
10
8
6
4
2
0
PPM

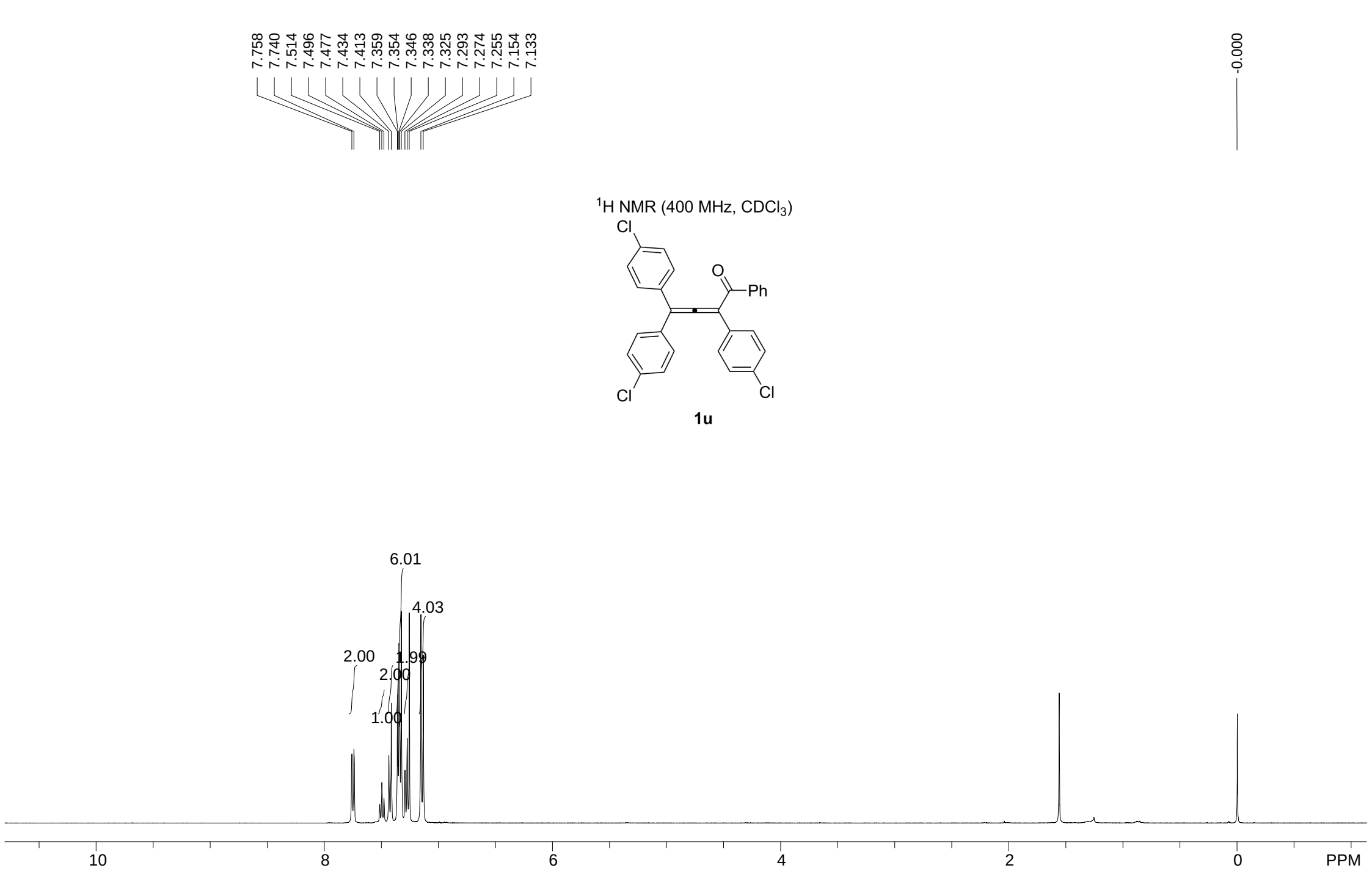

212.162
192.311
144.095
135.099
134.574
132.163
131.813
129.562
128.908
128.746
128.497
128.357
121.978
115.877
110.233
77.319
77.000
76.683
21.632
^{13}C NMR (100 MHz, $CDCl_3$)
O
Ph
Ph
Me
Br
1p
200
150
100
50
0
PPM

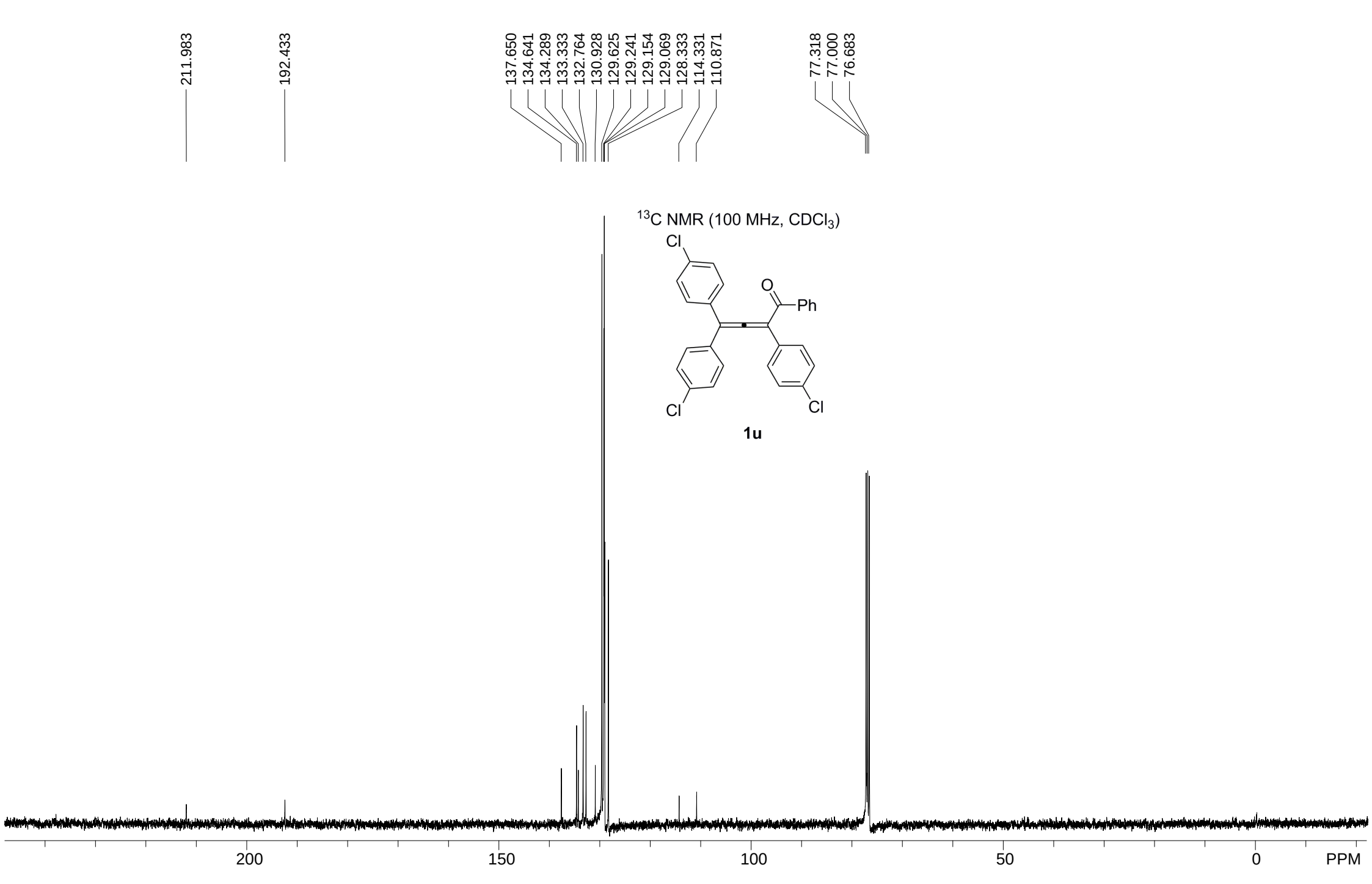

7.746
7.727
7.666
7.647
7.472
7.454
7.429
7.410
7.377
7.359
7.341
7.225
7.204
7.183
7.170
7.153
7.134
7.116
7.098
2.849
2.836
2.821
2.808
2.798
2.784
2.774
2.761
2.746
2.733
-0.000
^{1}H NMR (400 MHz, $CDCl_3$)
O
Ph
Ph
1s
8.02
6.05
2.01
2.01
4.00
10
8
6
4
2
0
PPM

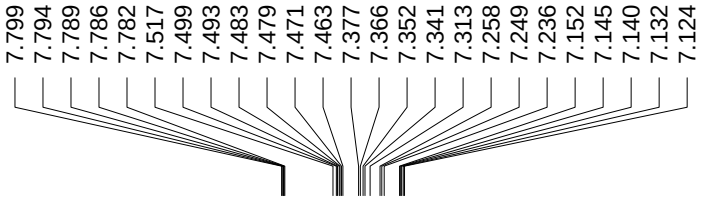

213.239
193.412
139.136
138.464
134.723
133.087
132.588
129.601
129.350
129.162
128.849
128.355
128.031
127.781
126.468
117.094
109.078
77.318
77.000
76.682
33.057
^{13}C NMR (100 MHz, $CDCl_3$)
O
Ph
Ph
1s
200
150
100
50
0
PPM


7.654
7.633
7.526
7.506
7.383
7.376
7.366
7.354
7.346
7.317
7.299
7.245
7.171
7.150
7.140
7.119
2.368
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
Me
O
Br
Ph
Me
1t
8.02
4.05
2.01
1.04
1.99
6.01
10
8
6
4
2
0
PPM

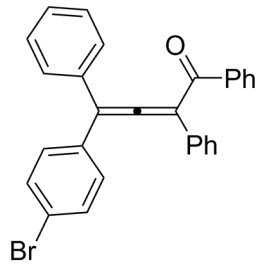

213.225
192.172
138.321
136.941
132.950
131.673
131.371
130.811
129.473
128.685
128.351
128.050
127.995
127.858
115.687
110.807
77.318
77.000
76.683
21.230
^{13}C NMR (100 MHz, $CDCl_3$)
Me
O
Br
Ph
Me
1t
200
150
100
50
0
PPM


7.758
7.740
7.514
7.496
7.477
7.434
7.413
7.359
7.354
7.346
7.338
7.325
7.293
7.274
7.255
7.154
7.133
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
O
Ph
Cl
Cl
1u
6.01
4.03
2.00
1.99
2.00
1.00
10
8
6
4
2
0
PPM

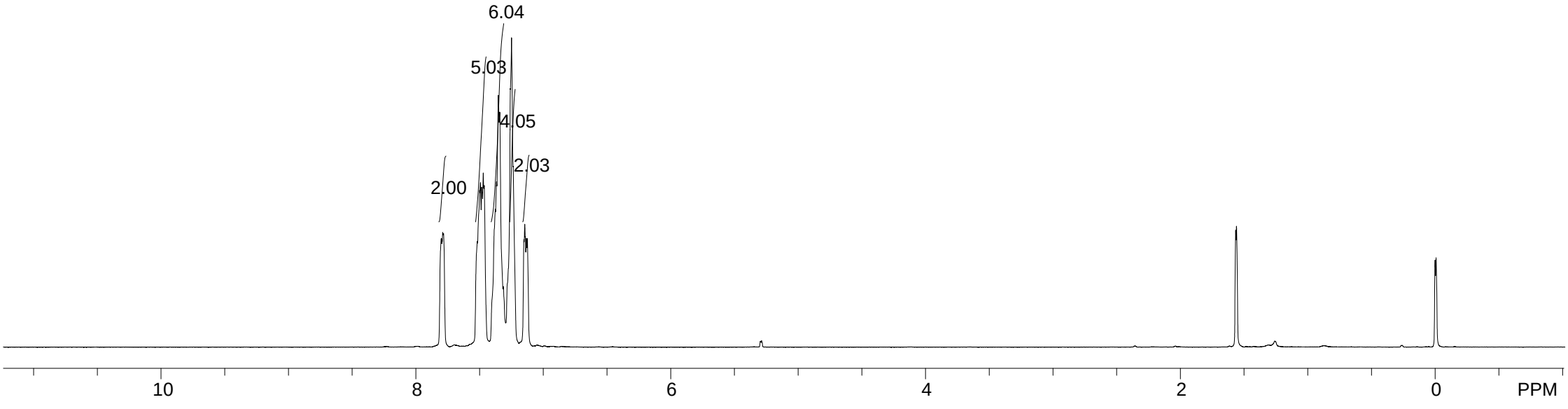

211.983
192.433
137.650
134.641
134.289
133.333
132.764
130.928
129.625
129.241
129.154
129.069
128.333
114.331
110.871
77.318
77.000
76.683
^{13}C NMR (100 MHz, $CDCl_3$)
Cl
O
Ph
Cl
Cl
1u
200
150
100
50
0
PPM

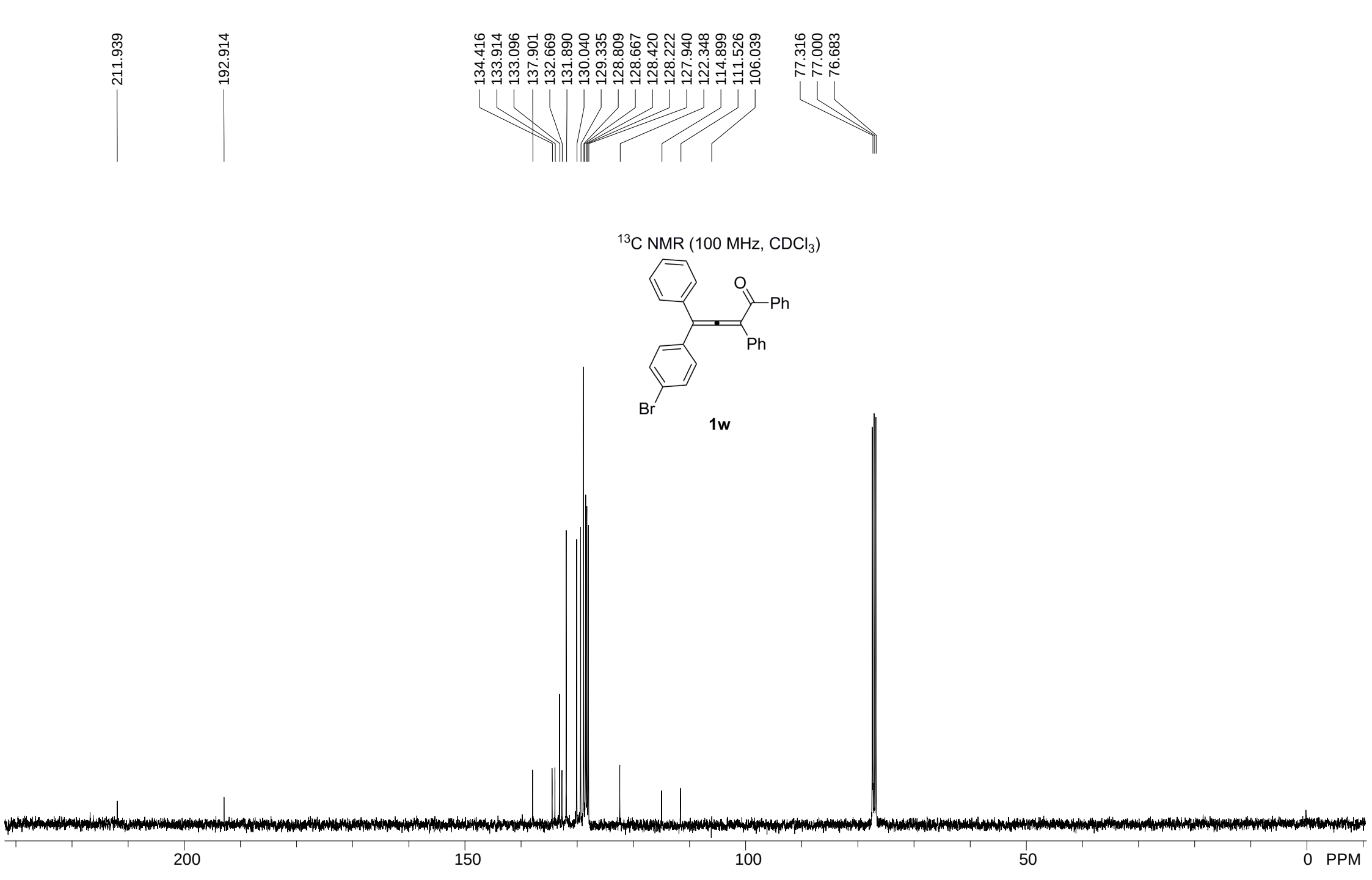


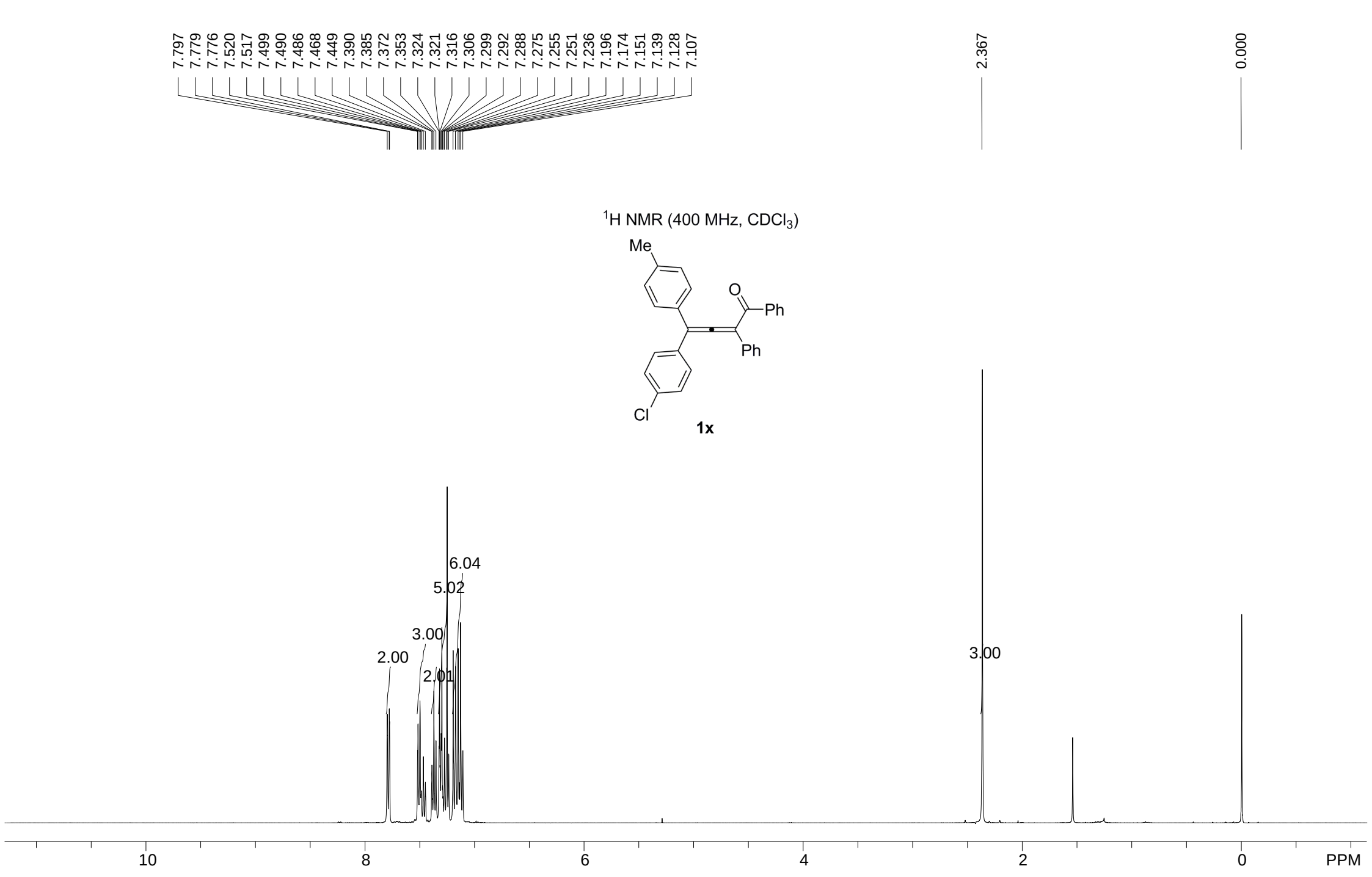
^{1}H NMR (400 MHz, $CDCl_3$)

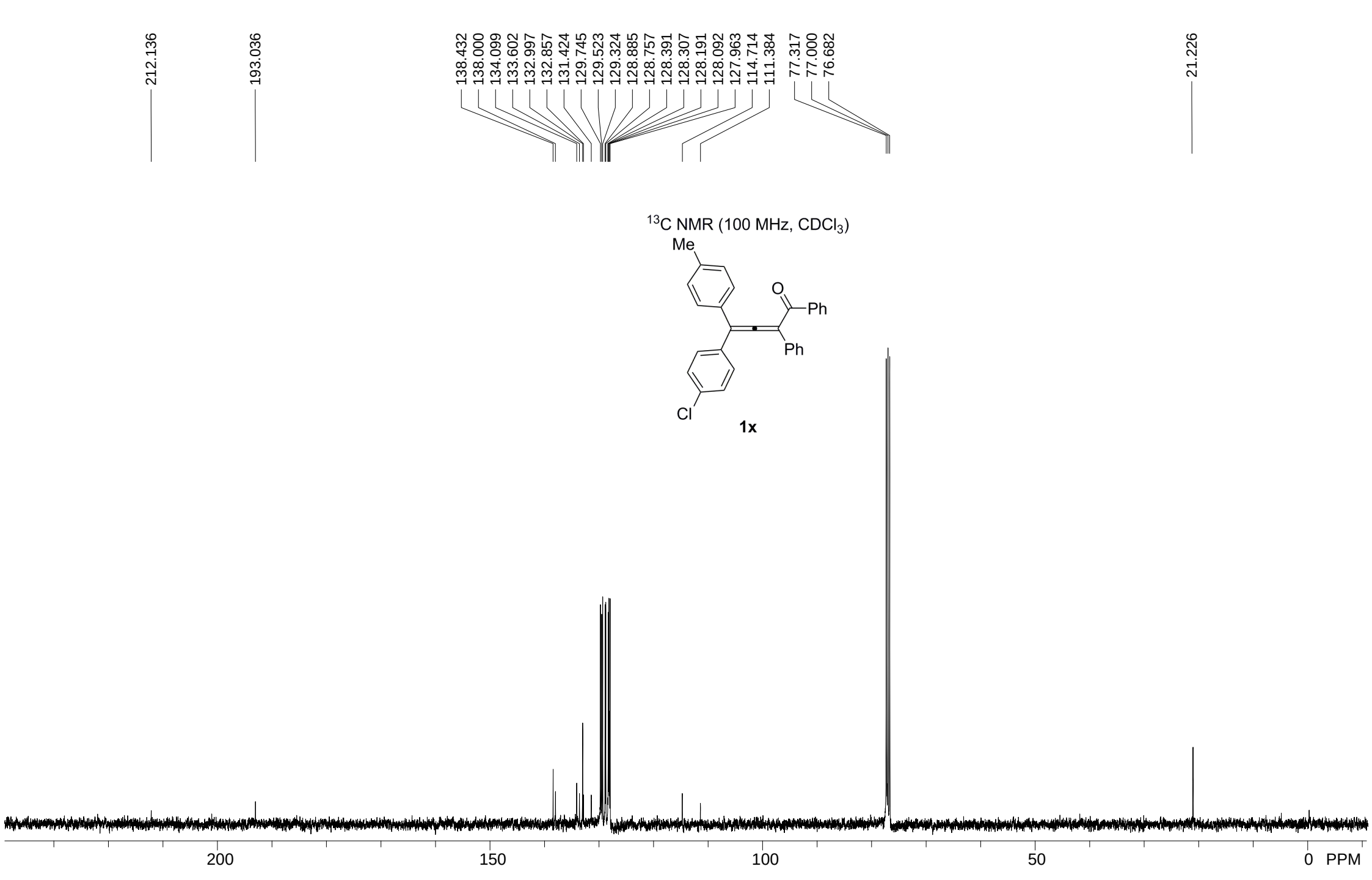


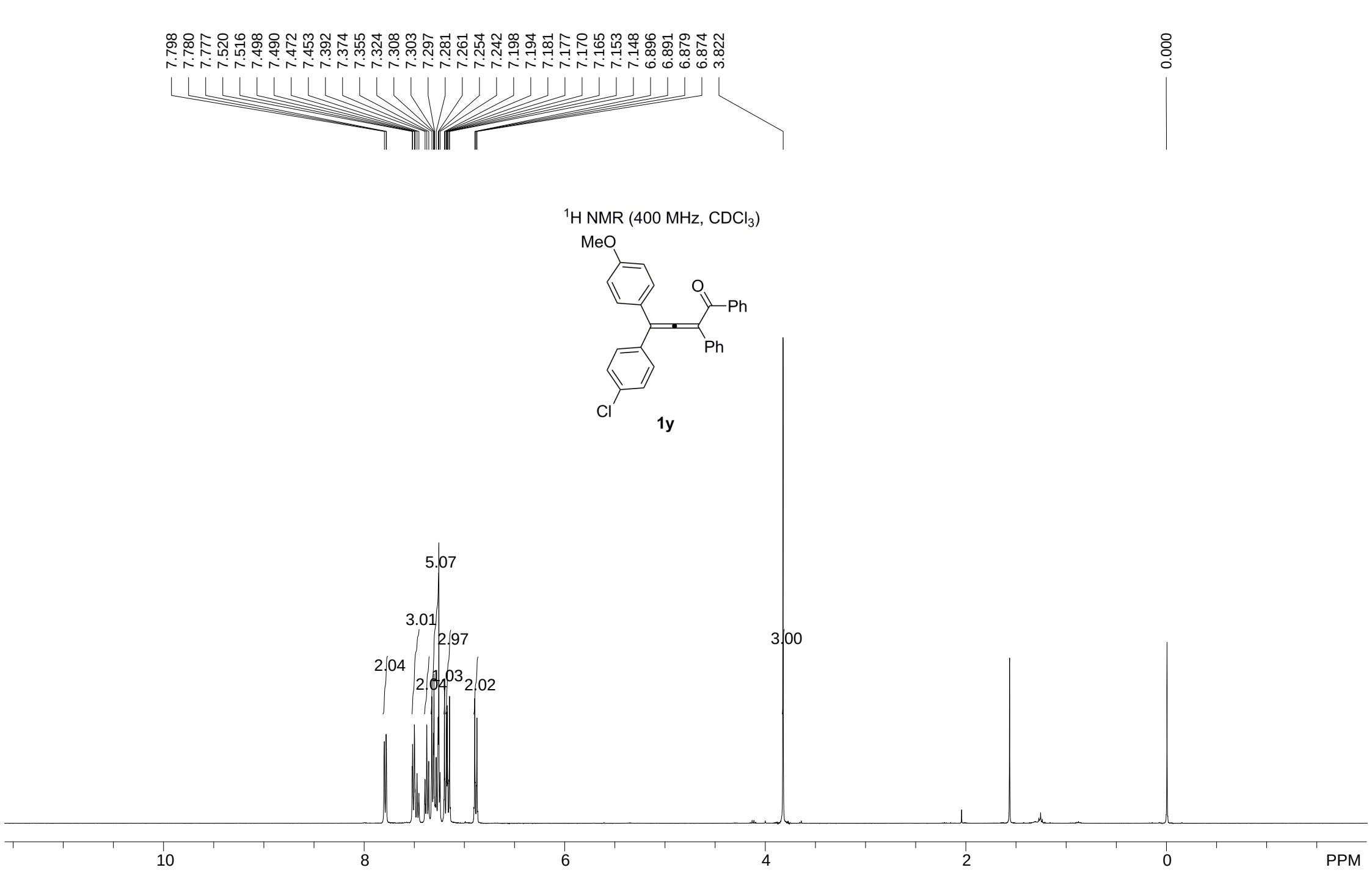
1w

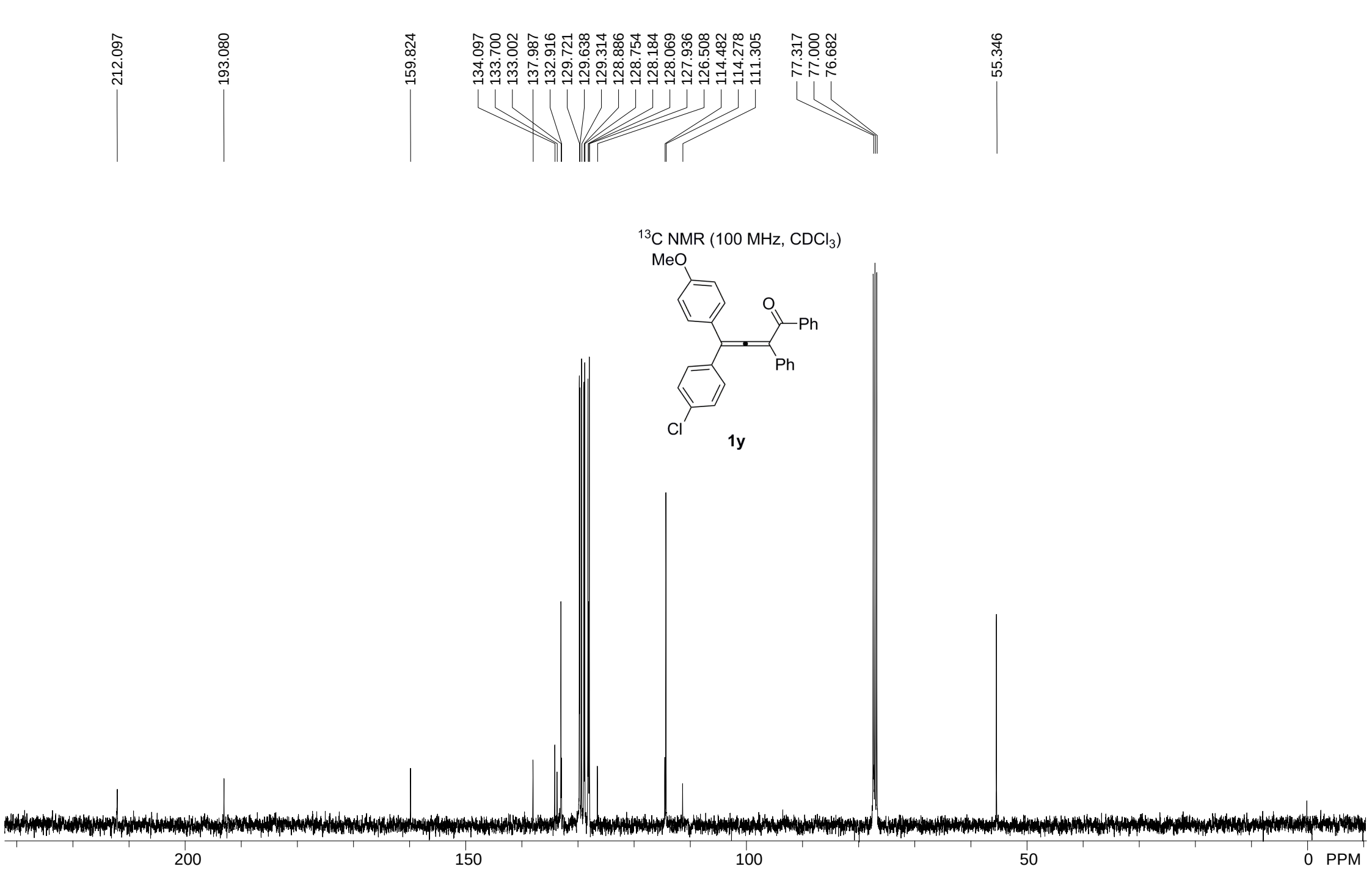


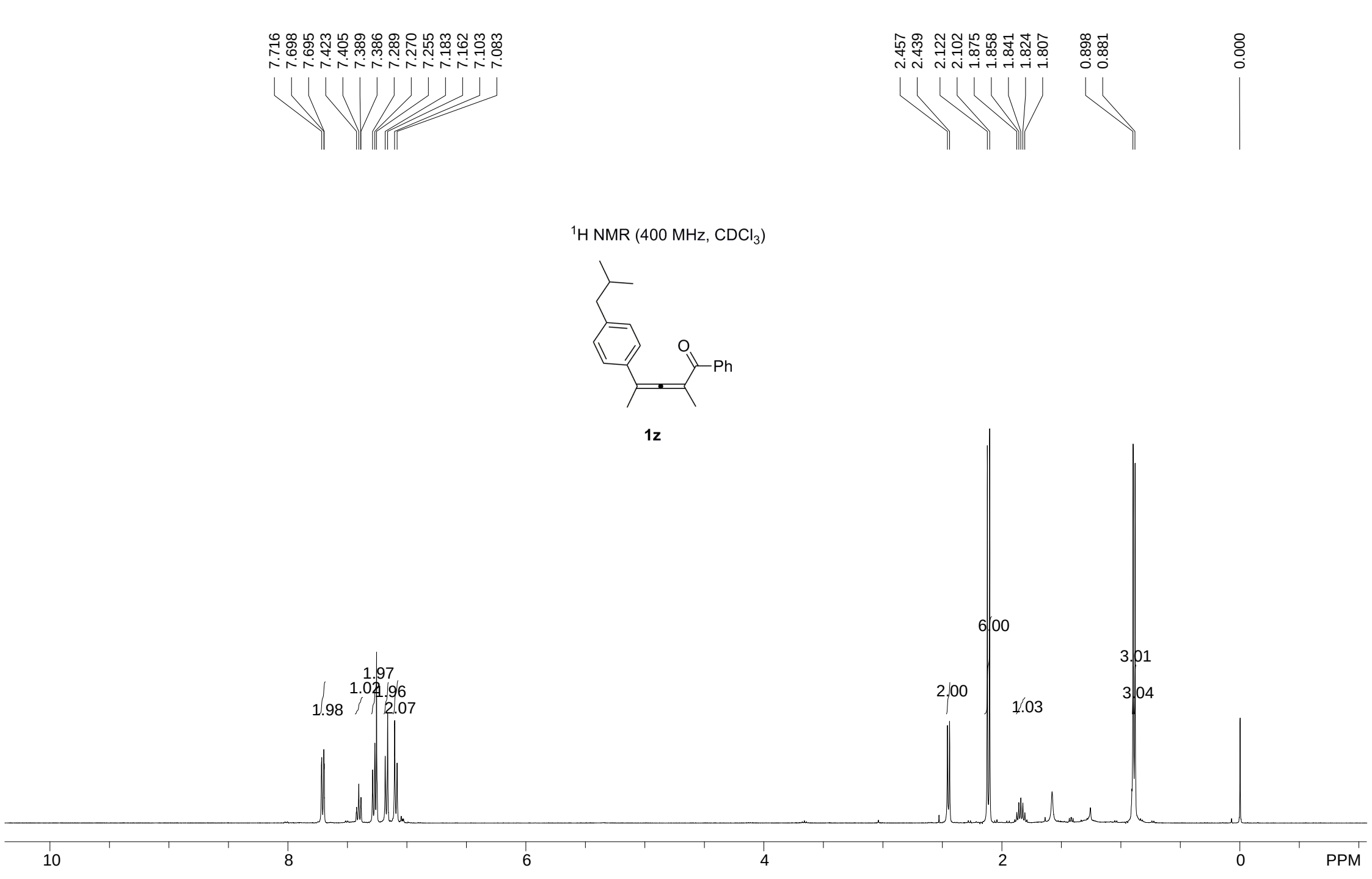

211.939
192.914
134.416
133.914
133.096
137.901
132.669
131.890
130.040
129.335
128.809
128.667
128.420
128.222
127.940
122.348
114.899
111.526
106.039
77.316
77.000
76.683
^{13}C NMR (100 MHz, CDCl$_3$)
O
Ph
Ph
Br
1w
200
150
100
50
0
PPM

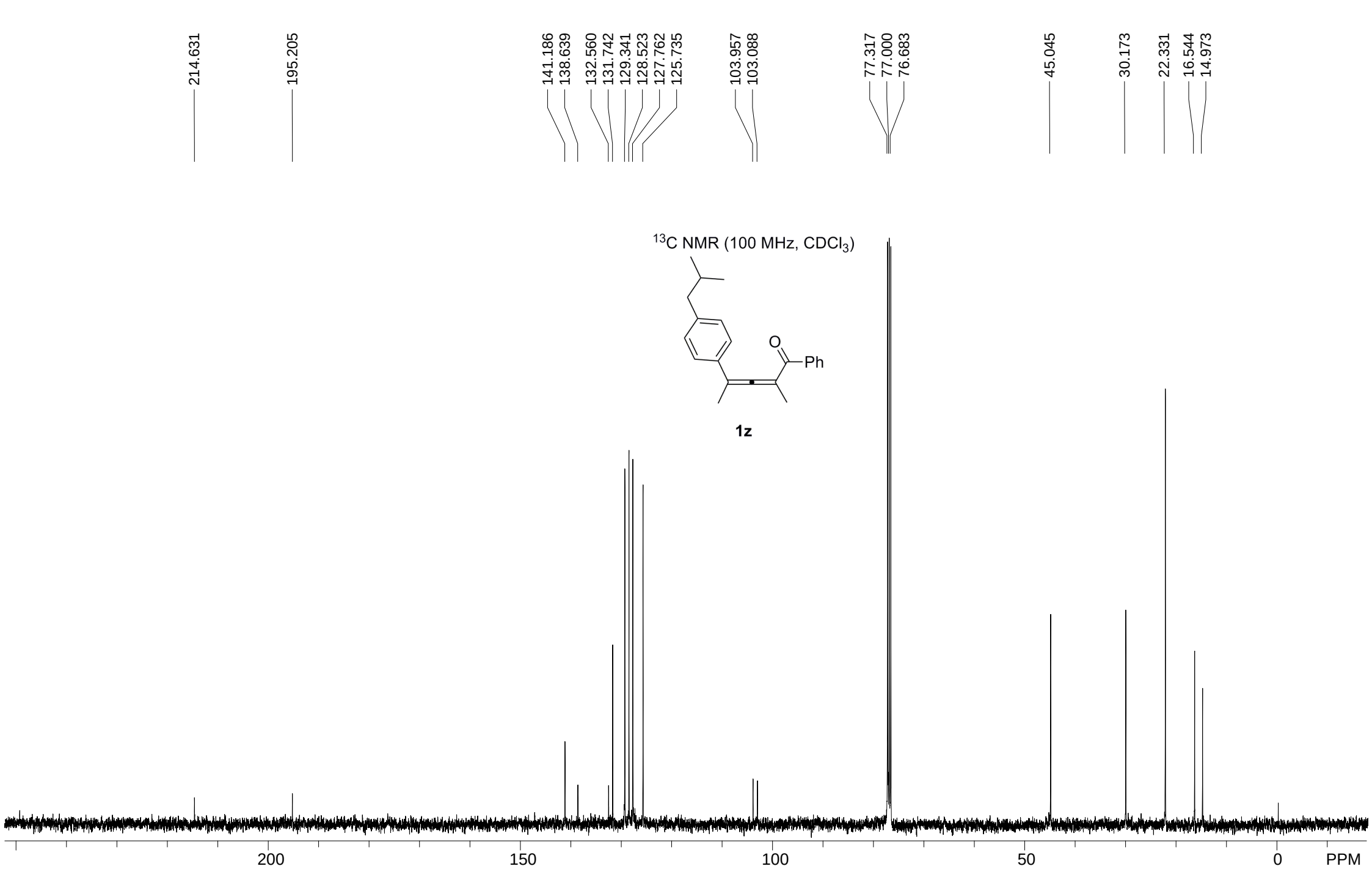

7.797
7.779
7.776
7.520
7.517
7.499
7.490
7.486
7.468
7.449
7.390
7.385
7.372
7.353
7.324
7.321
7.316
7.306
7.299
7.292
7.288
7.275
7.255
7.251
7.236
7.196
7.174
7.151
7.139
7.128
7.107
2.367
0.000
^{1}H NMR (400 MHz, CDCl$_3$)
Me
O
Ph
Ph
Cl
1x
6.04
5.02
3.00
2.00
2.01
3.00
10
8
6
4
2
0
PPM


212.136
193.036
138.432
138.000
134.099
133.602
132.997
132.857
131.424
129.745
129.523
129.324
128.885
128.757
128.391
128.307
128.191
128.092
127.963
114.714
111.384
77.317
77.000
76.682
21.226
^{13}C NMR (100 MHz, $CDCl_3$)
Me
O
Ph
Ph
Cl
1x
200
150
100
50
0
PPM


7.798
7.780
7.777
7.520
7.516
7.498
7.490
7.472
7.453
7.392
7.374
7.355
7.324
7.308
7.303
7.297
7.281
7.261
7.254
7.242
7.198
7.194
7.181
7.177
7.170
7.165
7.153
7.148
6.896
6.891
6.879
6.874
3.822
0.000
^{1}H NMR (400 MHz, $CDCl_3$)
MeO
O
Ph
Ph
Cl
1y
5.07
3.01
2.97
2.04
2.04
1.03
2.02
3.00
10
8
6
4
2
0
PPM


212.097
193.080
159.824
134.097
133.700
133.002
137.987
132.916
129.721
129.638
129.314
128.886
128.754
128.184
128.069
127.936
126.508
114.482
114.278
111.305
77.317
77.000
76.682
55.346
^{13}C NMR (100 MHz, CDCl$_3$)
MeO
O
Ph
Ph
Cl
1y
200
150
100
50
0
PPM


7.716
7.698
7.695
7.423
7.405
7.389
7.386
7.289
7.270
7.255
7.183
7.162
7.103
7.083
2.457
2.439
2.122
2.102
1.875
1.858
1.841
1.824
1.807
0.898
0.881
0.000
1H NMR (400 MHz, CDCl3)
O
Ph
1z
1.98
1.02
1.97
1.96
2.07
2.00
6.00
1.03
3.01
3.04
10
8
6
4
2
0
PPM


214.631
195.205
141.186
138.639
132.560
131.742
129.341
128.523
127.762
125.735
103.957
103.088
77.317
77.000
76.683
45.045
30.173
22.331
16.544
14.973
13C NMR (100 MHz, CDCl3)
O
Ph
1z
200
150
100
50
0
PPM