

Supporting Information

The Synthesis of Cycloalka[*b*]furans via an Au(I)-Catalyzed Tandem Reaction of 3-Yne-1,2-diols

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1. General information:

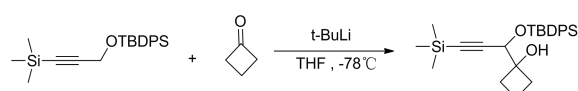
All reactions under standard conditions were carried out under air or argon, dry atmosphere and monitored by thin-layer chromatography (TLC) on gel F254 plates. All products were purified through silica gel chromatography (200~300 mesh). Column chromatography was carried out with light petroleum ether (bp. 60~90 °C), ethyl acetate and dichloromethane as eluent. ^1H and ^{13}C NMR spectra were recorded in CDCl_3 , acetone- d_6 or C_6D_6 on 400 MHz instruments and spectral data were reported in ppm. High-resolution mass spectral analysis (HRMS) data were measured on the Apex II by means of the ESI technique. Melting point was measured with SGW-X4B instrument.

2. Preparation and characterization of substrates

Substrates **1a**, **1b**, **1e**, **1q** were prepared using a known strategy.^[1] However, the other Substrates were prepared by the improved way showing below.



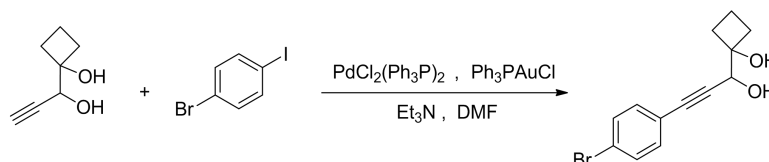
To a solution of trimethylsilylacetylene (5.56 g, 8 mL, 57 mmol) in dry tetrahydrofuran was added *n*-butyllithium (2.5 M in *n*-hexane) (25 mL, 62.7 mmol) at -78 °C under argon atmosphere. Then transferred the reaction flask to room temperature. After the reaction mixture was stirred for 1 h, Paraformaldehyde (2.7 g, 91.2 mmol) was added. Then raised the temperature to 45 °C and stirred the reaction mixture for another 4 h, the reaction mixture would be very thickness. At this time, TBDPSCl (19.8 g, 17 mL, 68.4 mmol) and imidazole (7.6 g, 119.7 mmol) was added. Stirred for 15 min. The aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product (16.7 g, 82%).



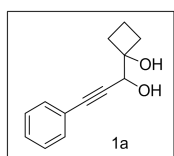
To a solution of tert-butyldiphenyl ((3-(trimethylsilyl) prop-2-yn-1-yl)oxy) silane (3.5 g, 5.41 mmol) in dry tetrahydrofuran was added tert-butyllithium (*t*-BuLi) (1.3 M in *n*-hexane) (8.3 mL, 10.81 mmol) at -78 °C under argon atmosphere. After the reaction mixture was stirred for 2 h at the same temperature, cyclobutanone (1.61 mL, 21.63 mmol) was added dropwise. The reaction mixture was stirred for another 2 h. Upon completion of the reaction (monitored by TLC). The reaction mixture was quenched by slow addition of aqueous solution of saturated ammonium chloride solution. The aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product (1.90 g, 80% yield).



To a stirred solution of 1-(1-((tert-butyldiphenylsilyl)oxy)-3-phenylprop-2-yn-1-yl) cyclobutanol (1.90 g, 4.31 mmol) in dry tetrahydrofuran (30 mL) was added tetrabutylammonium fluoride (2.26 g, 8.64 mmol) at room temperature under argon atmosphere. The reaction mixture was stirred for 1 h. Upon completion of the reaction (monitored by TLC), the reaction mixture was quenched by slow addition of water. The aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product **1r** (0.77 g, 88% yield) as a colorless oil.

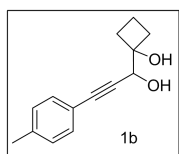


This reaction was referenced the strategy designed by Panda.^[2] To a 20 mL test tube equipped with a magnetic stir bar was added 1-bromo-4-iodobenzene (247 mg, 0.87 mmol), **1r** (100 mg, 0.79 mmol), dissolved with 1 mL DMF. Then the tube was filled with argon, PdCl₂(Ph₃P)₂ (11 mg, 0.16 mmol), Ph₃PAuCl (7.8 mg, 0.16 mmol) and Et₃N (330 µL, 2.38 mmol). The reaction mixture was stirred for 4 h. After 4 h, the reaction was quenched by 3 mL water and the aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product (**1l**) 1-(3-(4-bromophenyl)-1-hydroxyprop-2-yn-1-yl) cyclobutanol as a colorless amorphous solid.



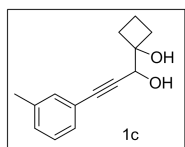
1-(3-phenylprop-2-yn-1-yl)cyclobutanol (**1a**)

1a as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.48–7.42 (m, 2H), 7.33–7.27 (m, 3H), 4.57 (s, 1H), 2.81(s, 2H), 2.40–2.24 (m, 2H), 2.20–2.06 (m, 2H), 1.92–1.80 (m, 1H), 1.68 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 131.80, 128.58, 128.27, 122.20, 86.71, 85.61, 68.09, 32.07, 31.69, 11.98; HRMS (ESI) calcd for C₁₃H₁₄O₂ [M+NH₄]⁺: 220.1332, found 220.1329.



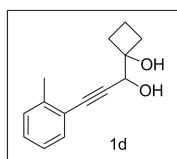
1-(3-(p-tolyl)prop-2-yn-1-yl)cyclobutanol (**1b**)

1b as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.34 (d, *J* = 8.1 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 4.57 (s, 1H), 3.38 (s, 2H), 2.39–2.24 (m, 5H), 2.20–2.06 (m, 2H), 1.90–1.75 (m, 1H), 1.73–1.54 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 138.73, 131.80, 129.09, 119.28, 86.24, 85.72, 76.78, 68.14, 32.06, 31.85, 21.52, 12.15; HRMS (ESI) calcd for C₁₄H₁₆O₂ [M+NH₄]⁺: 234.1489, found 234.1486.



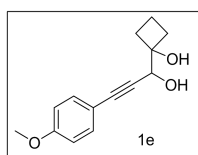
1-(1-hydroxy-3-(m-tolyl)prop-2-yn-1-yl)cyclobutanol (1c)

1c as a colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.27–7.14 (m, 4H), 4.56 (s, 1H), 2.47 (s, 2H), 2.38–2.30 (m, 5H), 2.19–2.09 (m, 2H), 1.93–1.83 (m, 1H), 1.75–1.72 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.07, 132.42, 129.57, 128.91, 128.24, 121.96, 86.20, 85.93, 76.64, 68.21, 32.19, 31.62, 21.18, 11.98; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{16}\text{O}_2$ $[\text{M}+\text{Na}]^+$: 239.1043, found 239.1039.



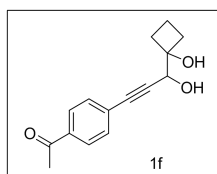
1-(1-hydroxy-3-(o-tolyl)prop-2-yn-1-yl)cyclobutanol (1d)

1d as a colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 (d, J = 8.0 Hz, 1H), 7.26 – 7.19 (m, 2H), 7.14 (d, J = 6.6 Hz, 1H), 4.61 (s, 1H), 2.49 – 2.39 (m, 4H), 2.38 – 2.33 (m, 2H), 2.26 – 2.11 (m, 3H), 1.89 (d, J = 10.8 Hz, 1H), 1.71 – 1.69 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, Acetone) δ 140.90, 132.73, 130.35, 129.17, 126.55, 123.88, 94.40, 83.40, 77.28, 68.75, 32.67, 29.65, 20.84, 12.81; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{16}\text{O}_2$ $[\text{M}+\text{NH}_4]^+$: 234.1489, found 234.1488.



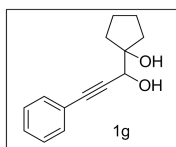
1-(1-hydroxy-3-(4-methoxyphenyl)prop-2-yn-1-yl)cyclobutanol (1e)

1e as a colorless oil. $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.50–7.33 (m, 2H), 6.90–6.75 (m, 2H), 4.55 (s, 1H), 3.80 (s, 3H), 2.71 (s, 2H), 2.39–2.24 (m, 2H), 2.21–2.04 (m, 2H), 1.93–1.80 (m, 1H), 1.73–1.61 (m, 1H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 159.77, 133.28, 114.20, 113.90, 85.61, 85.20, 68.16, 55.26, 32.13, 31.61, 11.96; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{16}\text{O}_3$ $[\text{M}+\text{H}]^+$: 233.1172, found 233.1169.



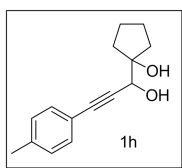
1-(4-(3-hydroxy-3-(1-hydroxycyclobutyl)prop-1-yn-1-yl)phenyl)ethanone (1f)

1f as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.84 (d, J = 8.4 Hz, 2H), 7.48 (d, J = 8.0 Hz, 2H), 4.59 (s, 1H), 3.27 (s, 1H), 3.07 (s, 1H), 2.56 (s, 3H), 2.36–2.29 (m, 2H), 2.17–2.096 (m, 2H), 1.90–1.80 (m, 1H), 1.71–1.64 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 197.60, 136.41, 131.96, 128.21, 127.25, 90.42, 84.63, 76.61, 68.05, 32.14, 32.03, 26.62, 12.04; **HRMS** (ESI) calcd for $\text{C}_{15}\text{H}_{16}\text{O}_3$ $[\text{M}+\text{Na}]^+$: 267.0993, found 267.0992.



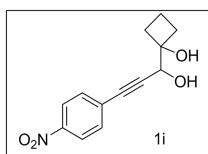
1-(1-hydroxy-3-phenylprop-2-yn-1-yl)cyclopentanol (1g)

1g as a colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.43–7.41 (m, 2H), 7.29–7.24 (m, 3H), 4.53 (s, 1H), 3.72 (s, 1H), 2.88 (s, 1H), 2.00–1.64 (m, 8H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 131.66, 128.41, 128.18, 122.26, 87.74, 85.56, 84.89, 69.32, 36.61, 36.32, 24.59, 24.47; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{16}\text{O}_2$ $[\text{M}+\text{Na}]^+$: 239.1047, found 239.1043.



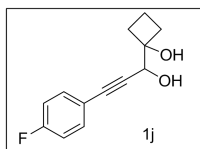
1-(1-hydroxy-3-(p-tolyl)prop-2-yn-1-yl)cyclopentanol (1h)

1h as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.32 (d, J = 8.0 Hz, 2H), 7.10 (d, J = 8.0 Hz, 2H), 4.50 (s, 1H), 3.19 (s, 1H), 2.53 (s, 1H), 2.34 (s, 3H), 2.01–1.91 (m, 1H), 1.90–1.79 (m, 3H), 1.78–1.62 (m, 4H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.73, 131.51, 128.85, 119.23, 86.95, 85.92, 85.13, 69.76, 36.52, 25.76, 24.50, 24.20, 21.59; **HRMS** (ESI) calcd for $\text{C}_{15}\text{H}_{18}\text{O}_2$ $[\text{M}+\text{Na}]^+$: 253.1202, found 253.1199.



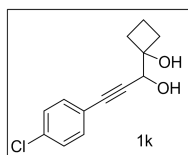
1-(1-hydroxy-3-(4-nitrophenyl)prop-2-yn-1-yl)cyclobutanol (1i)

1i as a orange colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.19 (d, J = 8.2 Hz, 2H), 7.59 (d, J = 8.0 Hz, 2H), 4.62 (s, 1H), 2.55 (d, J = 6.0 Hz, 1H), 2.39–2.34 (m, 3H), 2.17–2.15 (m, 2H), 1.91–1.1.89 (m, 1H), 1.72–1.65 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 147.35, 132.63, 129.11, 123.59, 92.17, 83.72, 76.54, 68.07, 32.32, 32.15, 11.94; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{13}\text{NO}_4$ $[\text{M}+\text{Na}]^+$: 226.0839, found 226.0838.



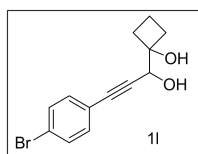
1-(3-(4-fluorophenyl)-1-hydroxyprop-2-yn-1-yl)cyclobutanol (1j)

1j as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.44–7.39 (m, 2H), 7.01–6.95 (m, 2H), 4.55 (s, 1H), 2.99 (s, 2H), 2.36–2.27 (m, 2H), 2.17–2.08 (m, 2H), 1.90–1.80 (m, 1H), 1.71–1.62 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 161.30(d, J = 12.0 Hz), 133.73(d, J = 8.4 Hz), 118.27(d, J = 3.6 Hz), 115.57(d, J = 22.0 Hz), 86.60, 84.71, 67.70, 32.24, 31.48, 11.96; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{13}\text{FO}_2$ $[\text{M}+\text{Na}]^+$: 243.0798, found 243.0792.



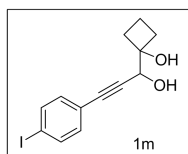
1-(3-(4-chlorophenyl)-1-hydroxyprop-2-yn-1-yl)cyclobutanol (**1k**)

1k as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.37 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 8.8 Hz, 2H), 4.56 (s, 1H), 2.62 (s, 2H), 2.35–2.29 (m, 2H), 2.15–2.09 (m, 2H), 1.92–1.82 (m, 1H), 1.73–1.66 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 134.75, 133.07, 128.69, 120.71, 87.71, 84.56, 76.59, 68.11, 32.19, 31.85, 11.97; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{13}\text{ClO}_2$ $[\text{M}+\text{Na}]^+$: 254.0496, found 259.0494.



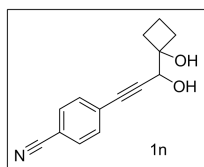
1-(3-(4-bromophenyl)-1-hydroxyprop-2-yn-1-yl)cyclobutanol (**1l**)

1l as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 4.54 (s, 1H), 2.98 (s, 2H), 2.33–2.29 (m, 2H), 2.12 (dd, J = 18.4, 9.6 Hz, 2H), 1.90–1.80 (m, 1H), 1.71–1.59 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 133.21, 131.55, 122.88, 121.14, 87.95, 84.49, 76.56, 68.00, 32.07, 31.85, 11.96; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{13}\text{BrO}_2$ $[\text{M}+\text{Na}]^+$: 302.9991, found 302.9989.



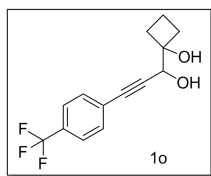
1-(1-hydroxy-3-(4-iodophenyl)prop-2-yn-1-yl)cyclobutanol (**1m**)

1m as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.67 (d, J = 8.4 Hz, 2H), 7.18 (d, J = 8.4 Hz, 2H), 4.57 (s, 1H), 2.66 (s, 2H), 2.35–2.29 (m, 2H), 2.17–2.09 (m, 2H), 1.92–1.83 (m, 1H), 1.72–1.63 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 137.53, 133.32, 121.71, 94.67, 88.17, 84.77, 76.57, 68.12, 32.21, 31.86, 11.98; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{13}\text{IO}_2$ $[\text{M}+\text{Na}]^+$: 350.9852, found 350.9850.



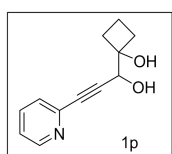
4-(3-hydroxy-3-(1-hydroxycyclobutyl)prop-1-yn-1-yl)benzonitrile (**1n**)

1n as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.60–7.57 (m, 2H), 7.52–7.50 (m, 2H), 4.58 (s, 1H), 2.89 (s, 1H), 2.70 (s, 1H), 2.35–2.29 (m, 2H), 2.18–2.10 (m, 2H), 1.92–1.82 (m, 1H), 1.70–1.62 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 132.37, 132.03, 127.25, 118.30, 111.97, 91.46, 83.86, 76.55, 68.01, 32.23, 32.12, 11.97; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_2$ $[\text{M}+\text{Na}]^+$: 250.0838, found 250.0836.



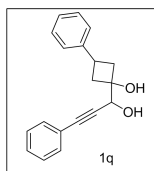
1-(1-hydroxy-3-(4-(trifluoromethyl)phenyl)prop-2-yn-1-yl)cyclobutanol (1o)

1o as a colorless amorphous solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.54 (s, 4H), 4.59 (s, 1H), 2.89 (d, J = 22.2 Hz, 2H), 2.37–2.30 (m, 2H), 2.18–2.11 (m, 2H), 1.93–1.83 (m, 1H), 1.73–1.68 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 132.08, 130.40(q, J = 33.0 Hz), 127.85, 126.04, 125.31, 125.27, 125.24, 125.20, 125.15, 122.44, 89.30, 84.24, 76.62, 68.03, 32.18, 31.98, 11.98; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{13}\text{F}_3\text{O}_2$ $[\text{M}+\text{Na}]^+$: 293.0760, found 293.0758.



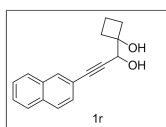
1-(1-hydroxy-3-(pyridin-2-yl)prop-2-yn-1-yl)cyclobutanol (1p)

1p as a colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.56 (d, J = 4.6 Hz, 1H), 7.67–7.62 (m, 1H), 7.43 (d, J = 7.8 Hz, 1H), 7.28–7.22 (m, 1H), 4.61 (s, 1H), 4.43 (s, 2H), 2.38–2.31 (m, 2H), 2.24–2.12 (m, 2H), 2.07–1.83 (m, 1H), 1.71–1.64 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 149.53, 142.46, 136.54, 127.33, 123.19, 88.62, 83.99, 76.55, 67.85, 32.12, 31.83, 12.18; **HRMS** (ESI) calcd for $\text{C}_{12}\text{H}_{13}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 204.1021, found 204.1019.



1-(1-hydroxy-3-phenylprop-2-yn-1-yl)-3-phenylcyclobutanol (1q)

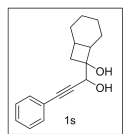
1q as a colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.49–7.46 (m, 2H), 7.35–7.18 (m, 8H), 4.70 (s, 1H), 3.29–3.18 (m, 1H), 2.86–2.83 (m, 2H), 2.65–2.55 (m, 2H), 2.35–2.26 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 144.92, 131.80, 128.64, 128.31, 126.56, 126.01, 122.08, 86.62, 85.80, 72.74, 68.09, 40.15, 39.65, 29.98; **HRMS** (ESI) calcd for $\text{C}_{19}\text{H}_{18}\text{O}_2$ $[\text{M}+\text{NH}_4]^+$: 296.1645, found 296.1642.



1-(1-hydroxy-3-(naphthalen-2-yl)prop-2-yn-1-yl)cyclobutanol (1r)

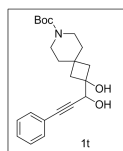
1r as a colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 (s, 1H), 7.86 – 7.69 (m, 3H), 7.56 – 7.40 (m, 3H), 4.66 (s, 1H), 3.37 (s, 1H), 3.15 (s, 1H), 2.39 (m, 2H), 2.26 – 2.12 (m, 2H), 1.93 – 1.82 (m, 1H), 1.71 (m, 1H); $^{13}\text{C NMR}$ (101 MHz,) δ 132.78, 132.72, 131.80, 128.31, 127.91, 127.65, 126.73,

126.50, 119.41, 87.03, 85.87, 76.66, 68.08, 32.06, 31.79, 12.02; **HRMS** (ESI) calcd for C₁₇H₁₆O₂ [M+H]⁺ : 253.1223, found 253.1213.



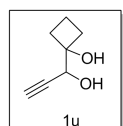
7-(1-hydroxy-3-phenylprop-2-yn-1-yl)bicyclo[4.2.0]octan-7-ol (1s)

1s as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.43 (m, 2H), 7.34–7.27 (m, 3H), 4.59 (d, J = 6.8 Hz, 1H), 2.49 – 2.42 (m, 2H), 2.31 – 2.21 (m, 1H), 2.02 – 1.97 (m, 1H), 1.81 – 1.50 (m, 8H); ¹³C NMR (101 MHz, CDCl₃) δ 131.81, 128.62, 128.32, 122.37, 86.99, 85.86, 75.88, 68.63, 39.08, 33.61, 26.17, 24.09, 22.27, 21.57, 21.03; **HRMS** (ESI) calcd for C₁₇H₂₀O₂ [M+H]⁺ : 257.1536, found 257.1526.



tert-butyl 2-hydroxy-2-(1-hydroxy-3-phenylprop-2-yn-1-yl)-7-azaspiro[3.5]nonane-7-carboxylate (1t)

1t as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.42 (m, 2H), 7.35 – 7.26 (m, 3H), 4.44 (d, J = 6.4 Hz, 1H), 3.38 – 3.35 (m, 2H), 3.31 – 3.29 (m, 2H), 2.44 – 2.39 (m, 2H), 2.23 – 2.17 (m, 2H), 1.92–1.89 (m, 2H), 1.86–1.58 (m, 2H), 1.45 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ 154.97, 131.77, 128.82, 128.40, 121.97, 86.59, 86.50, 79.35, 72.86, 70.05, 41.32, 41.12, 38.19, 37.27, 29.59, 28.47; **HRMS** (ESI) calcd for C₂₂H₂₉NO₄ [M+H]⁺ : 372.2169, found 372.2159.



1-(1-hydroxyprop-2-yn-1-yl)cyclobutanol (1u)

1u as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 4.30 (s, 1H), 3.85 (s, 2H), 2.44 (d, J = 2.0 Hz, 1H), 2.24–2.17 (m, 2H), 2.08–2.01 (m, 2H), 1.82–1.73 (m, 1H), 1.62–1.51 (m, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 81.87, 76.45, 73.77, 67.19, 31.72, 11.97; **HRMS** (ESI) calcd for C₇H₁₀O₂ [M+Na]⁺ : 149.0573, found 149.0571.

3. General experimental procedure for products

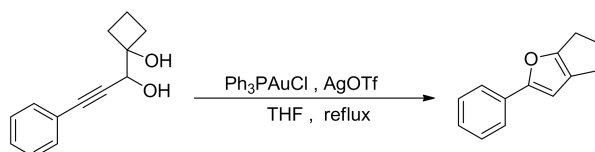
3.1 Optimization of the tandem reaction conditions

Entry	Catalyst (mol%)	Solvent	T (°C)	Yield ^b (%)
1	Ph ₃ PAuCl+AgOTf (10)	<i>n</i> -hexane	80	8
2	Ph ₃ PAuCl+AgOTf (10)	DCE	80	30
3	Ph ₃ PAuCl+AgOTf (10)	CHCl ₃	reflux	26

4	Ph ₃ PAuCl+AgOTf (10)	THF	reflux	41
5	Ph ₃ PAuCl+AgOTf (10)	THF	r.t.	trace
6	Ph ₃ PAuCl+AgOTf (10)	THF	40	27
7	Ph ₃ PAuCl+AgOTf (5)	THF	reflux	46
8	Ph ₃ PAuCl+AgOTf (1)	THF	reflux	56
9	Ph ₃ PAuCl+AgOTf (0.5)	THF	reflux	78
10	Ph ₃ PAuCl+AgOTf (0.2)	THF	reflux	73
11	Ph ₃ PAuCl+AgOTf (0.1)	THF	reflux	24
12	RuCl ₃ (10)	THF	reflux	trace
13	FeCl ₃ (10)	THF	reflux	^e n.d.
14	AgNO ₃ (10)	THF	reflux	^e n.d.
15 ^f	Ph ₃ PAuCl+AgOTf (0.5)	THF	reflux	12
16	AgO (10)	THF	reflux	11
17	IrCl ₃ (10)	THF	reflux	trace
18	Cu(OAc) ₂ (10)	THF	reflux	^e n.d.
19	CuCl (10)	THF	reflux	trace
20	Cu(OTf) ₂ (10)	THF	reflux	trace
21	AgOAc (10)	THF	reflux	^e n.d.
22	AgNO ₂ (10)	THF	reflux	^e n.d.
23	Yb(OTf) ₃ (10)	THF	reflux	trace
24	AuCl (0.5)	THF	reflux	10
25	Au[P(t-Bu) ₂ (o-biphenyl)]Cl+AgOTf (0.5)	THF	reflux	21
26	AuCl+AgSbF ₆ (0.5)	THF	reflux	trace
27	IPrAuCl+AgSbF ₆ (0.5)	THF	reflux	12
28	IPrAuCl+AgOTf (0.5)	THF	reflux	^e n.d.
29	Ph ₃ PAuCl (0.5)	THF	reflux	^e n.d.
30	AgOTf (0.5)	THF	reflux	^e n.d.
31 ^c	Ph ₃ PAuCl+AgNTf ₂ (0.5)	THF	reflux	17
32 ^c	Ph ₃ PAuCl+AgPF ₆ (0.5)	THF	reflux	^e n.d.
33 ^c	Ph ₃ PAuCl+AgBF ₄ (0.5)	THF	reflux	21
34 ^c	Ph ₃ PAuCl+AgOTf (0.5)	THF	reflux	82
35 ^{cd}	Ph ₃ PAuCl+AgOTf (0.5)	THF	reflux	86

^aReaction conditions: without other notification, all of the reactions were performed with 1-(1-hydroxy-3-phenylprop-2-yn-1-yl)cyclobutanol (0.5 mmol), catalyst in solvent (1.5 mL) under Ar for 4 h. ^bIsolated yield. ^cThe amount of solvent is 3 mL. ^dunder air. ^en.d. = not detected. ^f Reaction condition is MW, 70 °C, 0.5h.

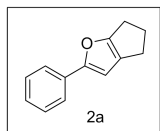
3.2. General experimental procedure for products 2a-2q using 2a as an example



To a stirred solution of 1-(1-hydroxy-3-phenylprop-2-yn-1-yl) cyclobutanol (100 mg, 0.5 mmol) in THF (3 mL) was successively added PPh₃AuCl (2.5 mg, 0.005 mmol) and AgOTf (1.3 mg, 0.005

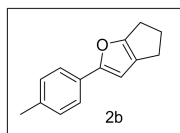
mmol) at 70 °C under air. The reaction would be stirred for 4 h, then quenched by water (2 mL). The aqueous layer was extracted three times with EtOAc and the combined organic layers were washed with brine, dried over sodium sulfate, and evaporated to dryness and purified by column chromatography to afford pure desired product **2a** (78 mg, 86%) as a white solid.

4. Characterization of products 2a-2q



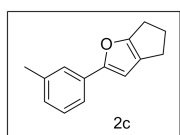
2-phenyl-5,6-dihydro-4H-cyclopenta[*b*]furan (**2a**)

2a (78 mg) was obtained through the general procedure in 86% yield as a white solid. mp 53.8–54.2 °C; ¹H NMR (400 MHz, acetone-*d*₆) δ 7.64 (d, *J* = 7.2 Hz, 2H), 7.42–7.31 (m, 2H), 7.27–7.16 (m, 1H), 6.69 (s, 1H), 2.74–2.70 (m, 2H), 2.57–2.53 (m, 2H), 2.48–2.43 (m, 2H); ¹³C NMR (101 MHz, acetone-*d*₆) δ 160.46, 158.49, 132.93, 129.69, 128.71, 127.68, 123.86, 104.81, 28.47, 25.29, 24.13; HRMS (ESI) calcd for C₁₃H₁₂O [M+H]⁺ : 185.0963, found 185.0961.



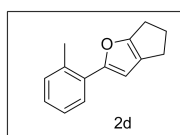
2-(p-tolyl)-5,6-dihydro-4H-cyclopenta[*b*]furan(**2b**)

2b (76 mg) was obtained through the general procedure in 83% yield as a white solid. mp 56.6–57.2 °C; ¹H NMR (400 MHz, acetone-*d*₆) δ 7.53 (d, *J* = 8.2 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 6.61 (s, 1H), 2.73–2.69 (m, 2H), 2.54–2.52 (m, 2H), 2.48–2.41(m, 2H), 2.31 (s, 3H); ¹³C NMR (101 MHz, acetone-*d*₆) δ 159.96, 158.76, 137.36, 130.32, 128.60, 123.92, 104.05, 28.49, 25.30, 24.17, 21.34; HRMS (ESI) calcd for C₁₄H₁₄O [M+H]⁺ : 199.1117, found 199.1110.



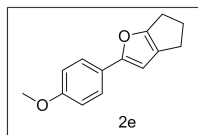
2-(m-tolyl)-5,6-dihydro-4H-cyclopenta[*b*]furan (**2c**)

2c (47 mg) was obtained through the general procedure in 51% yield as a colorless oil; ¹H NMR (400 MHz, acetone-*d*₆) δ 7.47–7.42 (m, 2H), 7.24 (t, *J* = 8.0 Hz, 1H), 7.04 (d, *J* = 7.6 Hz, 1H), 6.66 (s, 1H), 2.74–2.70 (m, 2H), 2.57–2.53 (m, 2H), 2.49–2.34(m,2H), 2.34 (s, 3H); ¹³C NMR (101 MHz, acetone-*d*₆) δ 160.26, 158.63, 139.12, 132.85, 129.58, 128.61, 128.42, 124.40, 121.07, 104.63, 28.42, 25.24, 24.10, 21.58; HRMS (ESI) calcd for C₁₄H₁₄O [M+H]⁺ : 199.1117, found 199.1115.



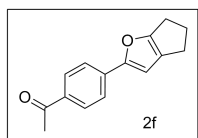
2-(o-tolyl)-5,6-dihydro-4H-cyclopenta[*b*]furan (**2d**)

2d (71 mg) was obtained through the general procedure in 78% yield as a colorless oil. $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.66–7.60 (m, 1H), 7.26–7.15 (m, 3H), 6.51 (s, 1H), 2.75–2.72 (m, 2H), 2.58–2.56 (m, 2H), 2.50–2.46 (m, 5H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 155.54, 153.51, 130.19, 127.66, 127.60, 124.04, 123.39, 122.82, 122.48, 104.01, 24.05, 20.82, 19.74, 17.83; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{14}\text{O}$ $[\text{M}+\text{K}]^+$: 237.0688, found 237.0686.



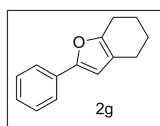
2-(4-methoxyphenyl)-5,6-dihydro-4H-cyclopenta[b]furan (2e)

2e (64 mg) was obtained through the general procedure in 70% yield as a colorless amorphous solid; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.59–7.55 (m, 2H), 6.96–6.92 (m, 2H), 6.53 (s, 1H), 3.81 (s, 3H), 2.72–2.68 (m, 2H), 2.56–2.51 (m, 2H), 2.48–2.41 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 159.92, 159.49, 158.66, 128.53, 125.95, 125.37, 115.15, 103.12, 55.73, 28.47, 25.27, 24.17; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{14}\text{O}_2$ $[\text{M}+\text{H}]^+$: 215.1089, found 215.1087.



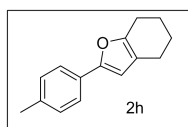
1-(4-(5,6-dihydro-4H-cyclopenta[b]furan-2-yl)phenyl)ethanone (2f)

2f (68 mg) was obtained through the general procedure in 73% yield as a colorless amorphous solid; $^1\text{H NMR}$ (400 MHz, C_6D_6) δ 7.89 (d, J = 8.8 Hz, 2H), 7.65 (d, J = 8.2 Hz, 2H), 7.23 (s, 3H), 6.45 (s, 1H), 2.47 (t, J = 7.6 Hz, 2H), 2.38 (t, J = 6.8 Hz, 2H), 2.19 (s, 3H), 2.17–2.03 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, C_6D_6) δ 195.63, 161.32, 157.11, 135.93, 135.63, 129.32, 122.91, 106.29, 27.88, 26.07, 24.88, 23.55; **HRMS** (ESI) calcd for $\text{C}_{15}\text{H}_{14}\text{O}_2$ $[\text{M}+\text{H}]^+$: 227.1067, found 227.1058.



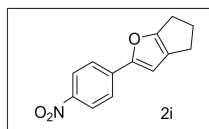
2-phenyl-4,5,6,7-tetrahydrobenzofuran (2g)

2g (40 mg) was obtained through the general procedure in 44% yield as a colorless oil; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.70–7.60 (m, 2H), 7.40–7.31 (m, 2H), 7.25–7.15 (m, 1H), 6.62 (s, 1H), 2.64–2.60 (m, 2H), 2.46–2.42 (m, 2H), 1.89–1.81 (m, 2H), 1.77–1.70 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 152.81, 151.95, 132.88, 130.04, 128.02, 124.40, 120.46, 107.64, 24.35, 24.30, 23.23; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{14}\text{O}$ $[\text{M}+\text{H}]^+$: 199.1117, found 199.1110.



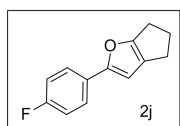
2-(p-tolyl)-4,5,6,7-tetrahydrobenzofuran (2h)

2h (35 mg) was obtained through the general procedure in 38% yield as a colorless oil; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.53 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 6.54 (s, 1H), 2.63–2.59 (m, 2H), 2.45–2.41 (m, 2H), 2.31 (s, 3H), 1.90–1.79 (m, 2H), 1.78–1.68 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 152.62, 151.05, 137.25, 130.24, 129.85, 124.02, 119.91, 106.45, 23.96, 23.92, 23.88, 22.83, 21.27; **HRMS** (ESI) calcd for $\text{C}_{15}\text{H}_{16}\text{O}$ $[\text{M}+\text{H}]^+$: 213.1274, found 213.1265.



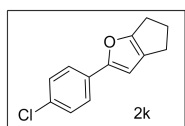
2-(4-nitrophenyl)-5,6-dihydro-4H-cyclopenta[b]furan (2i)

2i (72 mg) was obtained through the general procedure in 77% yield as a orange colorless amorphous solid; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 8.20 (d, J = 8.8 Hz, 2H), 7.68 (d, J = 8.8 Hz, 2H), 6.74 (s, 1H), 2.81–2.77 (m, 2H), 2.63–2.59 (m, 2H), 2.53–2.46 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 162.69, 155.31, 145.70, 137.40, 128.75, 124.36, 122.86, 107.75, 27.69, 24.84, 23.35; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{11}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 203.0812, found 203.0808.



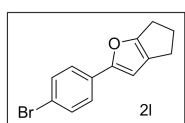
2-(4-fluorophenyl)-5,6-dihydro-4H-cyclopenta[b]furan (2j)

2j (80 mg) was obtained through the general procedure in 87% yield as a white solid. mp 57.8–58.4 °C; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.68–7.64 (m, 2H), 7.16–7.12 (m, 2H), 6.65 (s, 1H), 2.73–2.69 (m, 2H), 2.56–2.53 (m, 2H), 2.49–2.41 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 162.74(d, J = 243 Hz), 160.50, 157.59, 129.58(d, J = 3 Hz), 128.80, 125.84(d, J = 8 Hz), 116.56(d, J = 22 Hz), 116.45, 104.72, 104.71, 28.49, 25.29, 24.13; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{11}\text{FO}$ $[\text{M}+\text{H}]^+$: 203.0867, found 203.0858.



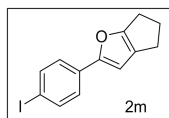
2-(4-chlorophenyl)-5,6-dihydro-4H-cyclopenta[b]furan (2k)

2k (85 mg) was obtained through the general procedure in 92% yield as a white solid. mp 73.1–74.6 °C; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.65–7.62 (m, 2H), 7.38–7.37 (m, 2H), 6.73 (s, 1H), 2.74–2.70 (m, 2H), 2.57–2.54 (m, 2H), 2.50–2.42(m,2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 160.98, 157.27, 132.63, 131.65, 129.78, 128.94, 125.34, 105.56, 28.44, 25.25, 24.05; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{11}\text{ClO}$ $[\text{M}+\text{H}]^+$: 219.0571, found 219.0564.



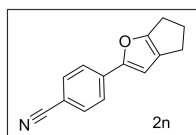
2-(4-bromophenyl)-5,6-dihydro-4*H*-cyclopenta[*b*]furan (**2l**)

2l (77 mg) was obtained through the general procedure in 82% yield as a white solid. mp 84.6–85.7 °C; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.59–7.50 (m, 4H), 6.74 (s, 1H), 2.75–2.68 (m, 2H), 2.58–2.51 (m, 2H), 2.50–2.41 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 161.08, 157.30, 132.76, 132.03, 128.99, 125.63, 120.68, 105.69, 28.47, 25.29, 24.08; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{11}\text{BrO}$ $[\text{M}+\text{H}]^+$: 263.0066, found 263.0057.



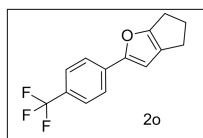
2-(4-iodophenyl)-5,6-dihydro-4*H*-cyclopenta[*b*]furan (**2m**)

2m (76 mg) was obtained through the general procedure in 80% yield as a white solid. mp 100.3–101.1 °C; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.73 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 6.76 (s, 1H), 2.73–2.7 (m, 2H), 2.57–2.53 (m, 2H), 2.49–2.42 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 161.09, 157.32, 138.73, 132.41, 128.94, 125.70, 105.73, 91.82, 28.41, 25.24, 24.02; **HRMS** (ESI) calcd for $\text{C}_{13}\text{H}_{11}\text{IO}$ $[\text{M}+\text{H}]^+$: 310.9927, found 310.9918.



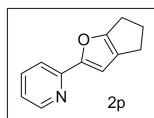
4-(5,6-dihydro-4*H*-cyclopenta[*b*]furan-2-yl)benzonitrile (**2n**)

2n (68 mg) was obtained through the general procedure in 81% yield as a colorless amorphous solid; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.79–7.77 (m, 2H), 7.74–7.72 (m, 2H), 6.94 (s, 1H), 2.77–2.73 (m, 2H), 2.59–2.55 (m, 2H), 2.51–2.44 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 162.72, 156.55, 136.50, 133.59, 129.52, 124.04, 119.62, 110.28, 108.21, 28.39, 25.25, 23.92; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{11}\text{NO}$ $[\text{M}+\text{H}]^+$: 210.0913, found 210.0905.



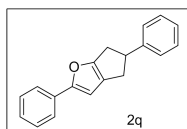
2-(4-(trifluoromethyl)phenyl)-5,6-dihydro-4*H*-cyclopenta[*b*]furan (**2o**)

2o (58 mg) was obtained through the general procedure in 62% yield as a yellow solid. mp 81.2–81.9 °C; $^1\text{H NMR}$ (400 MHz, acetone- d_6) δ 7.83 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.4 Hz, 2H), 6.90 (s, 1H), 2.77–2.74 (m, 2H), 2.60–2.56 (m, 2H), 2.52–2.44 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, acetone- d_6) δ 162.11, 156.87, 136.30, 129.27, 128.55 (q, J = 33.0 Hz), 126.71 (q, J = 4.0 Hz), 125.58 (q, J = 270.0 Hz), 124.03, 107.30, 28.45, 25.28, 24.00; **HRMS** (ESI) calcd for $\text{C}_{14}\text{H}_{11}\text{F}_3\text{O}$ $[\text{M}+\text{H}]^+$: 253.0835, found 253.0826.



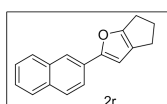
2-(5,6-dihydro-4*H*-cyclopenta[*b*]furan-2-yl)pyridine (**2p**)

2p (38 mg) was obtained through the general procedure in 42% yield as a colorless crystal. mp 85.9-86.3 °C; ¹H NMR (400 MHz, acetone-*d*₆) δ 8.51–8.49 (m, 1H), 7.78–7.74 (m, 1H), 7.63–7.61 (m, 1H), 7.18–7.15 (m, 1H), 6.95 (s, 1H), 2.76–2.72 (m, 2H), 2.59–2.55 (m, 2H), 2.51–2.43(m,2H); ¹³C NMR (101 MHz, acetone-*d*₆) δ 161.87, 158.72, 150.98, 150.55, 137.51, 129.01, 122.37, 118.20, 107.92, 28.43, 25.27, 24.00; HRMS (ESI) calcd for C₁₂H₁₁NO [M+H]⁺ : 186.0913, found 186.0906.



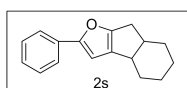
2,5-diphenyl-5,6-dihydro-4*H*-cyclopenta[*b*]furan (**2q**)

2q (41 mg) was obtained through the general procedure in 78% yield as colorless amorphous solid. ¹H NMR (400 MHz, acetone-*d*₆) δ 7.68 (d, *J* = 7.4 Hz, 2H), 7.44–7.17 (m, 8H), 6.75 (s, 1H), 4.17–4.04 (m, 1H), 3.23 (dd, *J* = 15.6, 9.2 Hz, 1H), 3.06 (dd, *J* = 14.8, 8.8 Hz, 1H), 2.91–2.78 (m, 1H), 2.67–2.62 (m, 1H); ¹³C NMR (101 MHz, acetone-*d*₆) δ 158.63, 158.51, 147.23, 132.80, 129.73, 129.56, 127.84, 127.81, 127.58, 127.31, 123.93, 104.73, 49.18, 34.60, 33.82; HRMS (ESI) calcd for C₁₉H₁₆O [M+H]⁺ : 261.1274, found 261.1264.



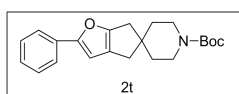
2-(naphthalen-2-yl)-5,6-dihydro-4*H*-cyclopenta[*b*]furan (**2r**)

2r (67 mg) was obtained through the general procedure in 72% yield as a white solid. mp 88.9-91.3 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.06 (s, 1H), 7.81-7.78 (m, 3H), 7.70 (d, *J* = 10.4 Hz, 1H), 7.45-7.41(m, 2H), 6.64 (s, 1H), 2.79 – 2.50 (m, 6H); ¹³C NMR (101 MHz, CDCl₃) δ 160.03, 157.64, 133.67, 132.35, 129.18, 128.23, 128.80, 127.80, 127.69, 126.35, 125.49, 121.97, 120.96, 104.28, 27.75, 24.85, 23.56; HRMS (ESI) calcd for C₁₇H₁₄O [M+H]⁺ : 235.1117, found 235.1111.



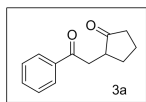
2-phenyl-4,5,6,7,7a,8-hexahydro-3*bH*-indeno[2,1-*b*]furan (**2s**)

2s (80 mg) was obtained through the general procedure in 86% yield as a colorless amorphous solid; ¹H NMR (400 MHz, CDCl₃) δ 7.60 (dd, *J* = 0.8, 1.2 Hz, 2H), 7.35 – 7.31 (m, 2H), 7.25 – 7.16 (m, 1H), 6.52 (s, 1H), 2.87 – 2.73 (m, 3H), 2.51 – 2.46 (m, 1H), 1.68-1.63 (m, 1H), 1.54 – 1.45 (m, 2H), 1.44 – 1.43 (m, 4H), 1.42 – 1.41 (m, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 158.96, 156.58, 131.98, 131.93, 128.56, 126.44, 122.99, 102.90, 42.71, 36.87, 30.81, 29.21, 28.73, 22.86, 22.79; HRMS (ESI) calcd for C₁₇H₁₈O [M+H]⁺ : 239.1430, found 239.1421.



tert-butyl 2-phenyl-4,6-dihydrospiro[cyclopenta[*b*]furan-5,4'-piperidine]-1'-carboxylate (**2t**)

2t (69 mg) was obtained through the general procedure in 72% yield as a colorless amorphous solid; **¹H NMR** (400 MHz, CDCl₃) δ 7.60 – 7.58 (m, 2H), 7.36 – 7.32 (m, 2H), 7.22 – 7.18 (m, 1H), 6.50 (s, 1H), 3.55-3.50 (m, 2H), 3.44 – 3.37 (m, 2H), 2.68 (s, 2H), 2.48 (s, 2H), 1.72-1.70 (m, 4H), 1.47 (s, 9H); **¹³C NMR** (101 MHz, CDCl₃) δ 157.56, 156.48, 154.89, 131.66, 128.60, 126.72, 124.76, 123.07, 103.69, 79.42, 47.44, 38.25, 37.55, 36.86, 28.48; **HRMS** (ESI) calcd for C₂₂H₂₇NO₃ [M+H]⁺ : 354.2064, found 354.2049.



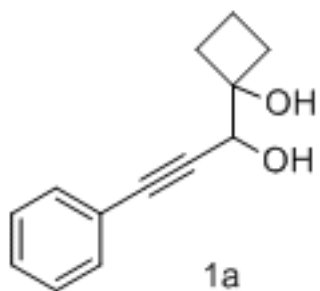
2-(2-oxo-2-phenylethyl)cyclopentanone (**3a**)

3a (56 mg) was obtained through the general procedure in 56% yield as white amorphous solid; **¹H NMR** (400 MHz, CDCl₃) δ 8.00–7.91 (m, 2H), 7.58–7.55 (m, 1H), 7.46 (m, 2H), 3.53 (dd, *J* = 16.0, 4.0 Hz, 1H), 3.05 (dd, *J* = 16.0, 8.0 Hz, 1H), 2.72–2.58 (m, 1H), 2.47–2.21 (m, 3H), 2.14–2.04 (m, 1H), 1.93–1.79 (m, 1H), 1.63–1.59 (m, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 220.32, 197.95, 136.62, 133.23, 128.62, 128.04, 45.09, 38.65, 37.56, 29.72, 20.85; **HRMS** (ESI) calcd for C₁₃H₁₄O₂ [M+H]⁺ : 203.1077, found 203.1067.

5. References

- [1] X.-Q. Mou, Z.-L. Xu, S.-H. Wang, D.-Y. Zhu, J. Wang, W. Bao, S.-J. Zhou, C. Yang, D. Zhang, *Chemical Communications* **2015**, 51, 12064-12067.
- [2] B. Panda, T. K. Sarkar, *Synthesis* **2013**, 45, 817-829.

6. ¹H NMR and ¹³C NMR spectra for substrates and products



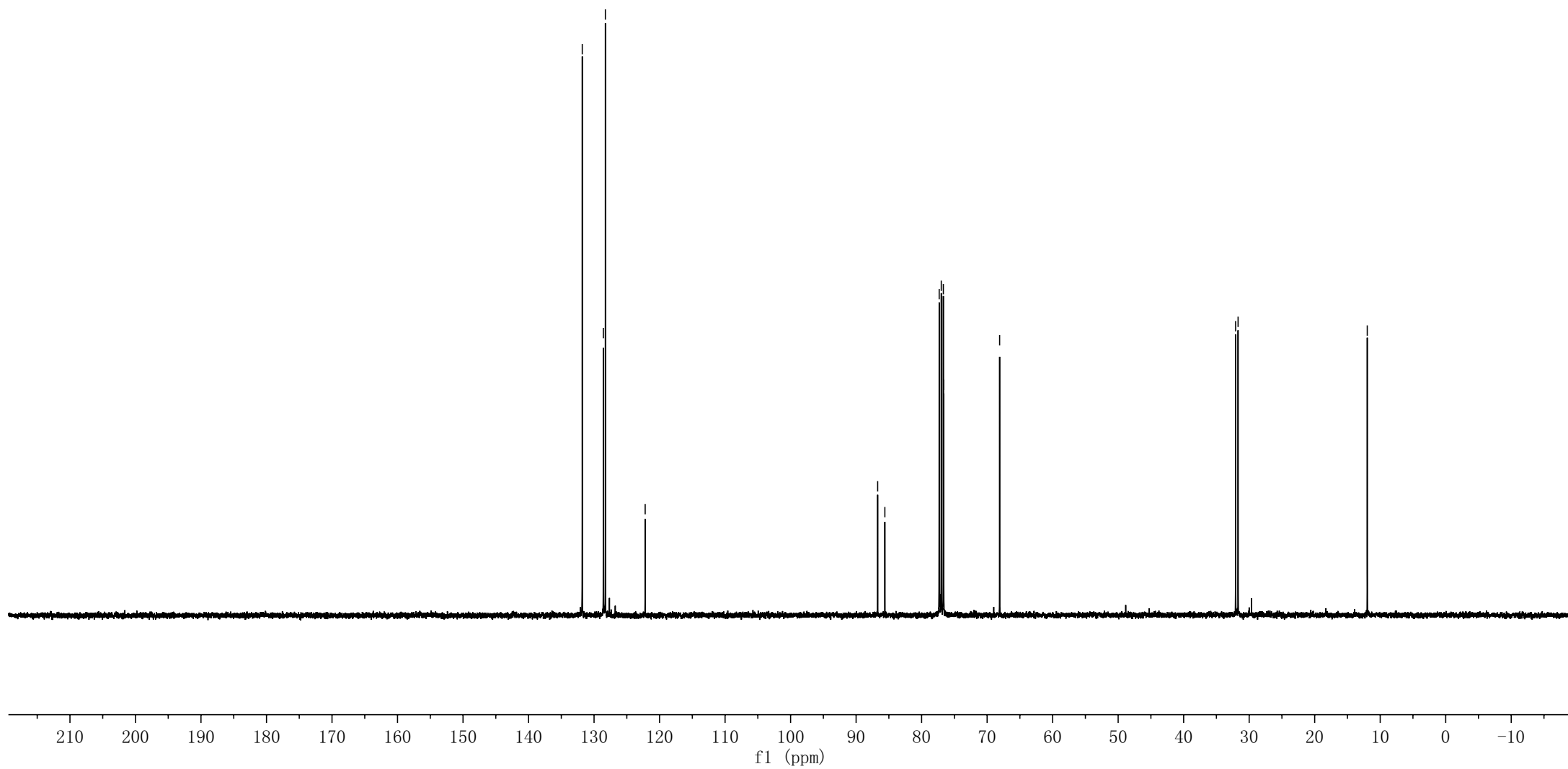
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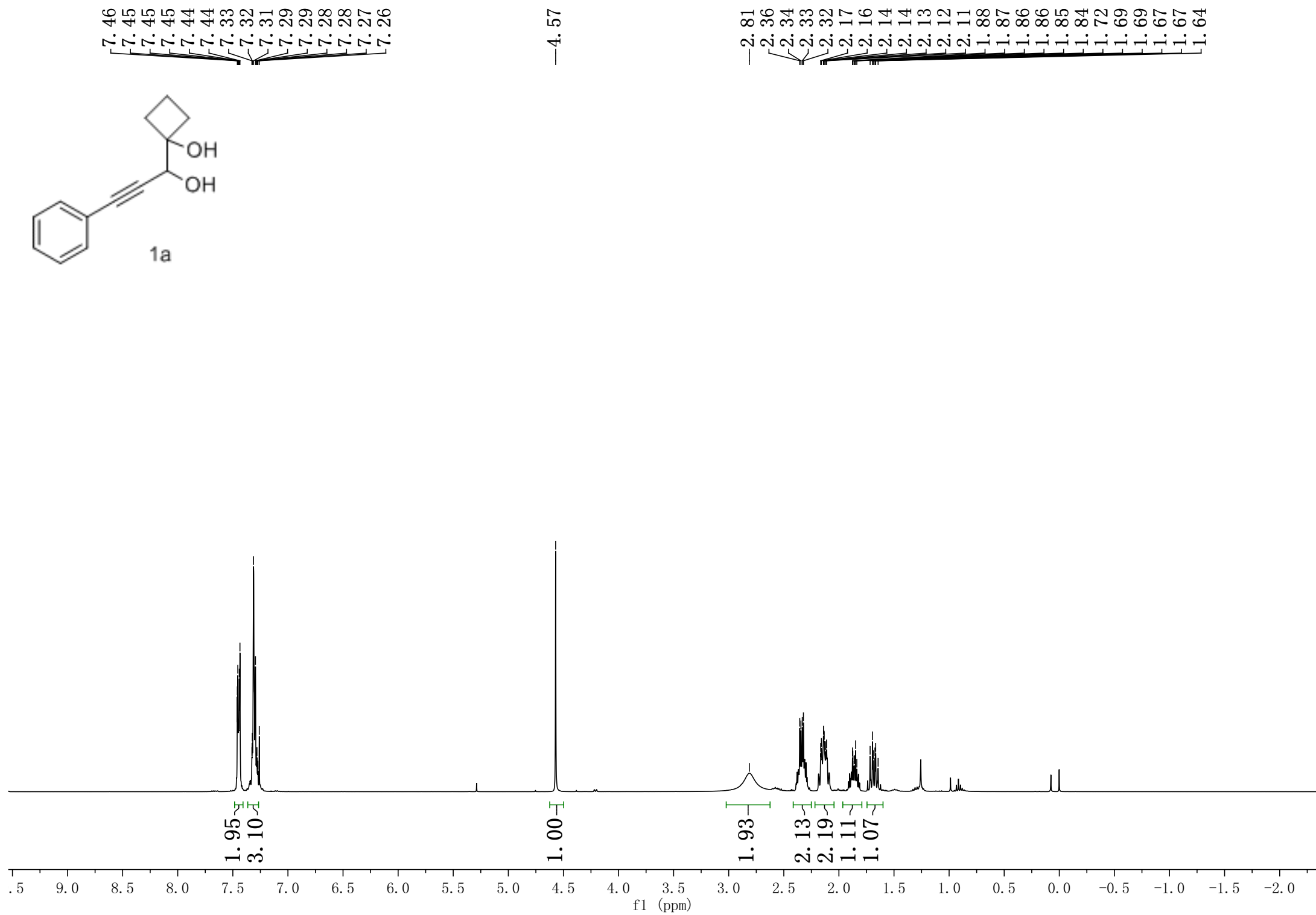
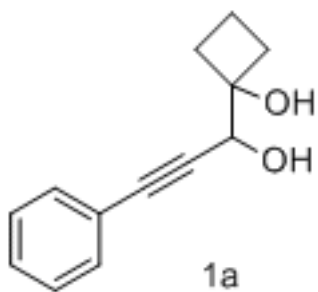
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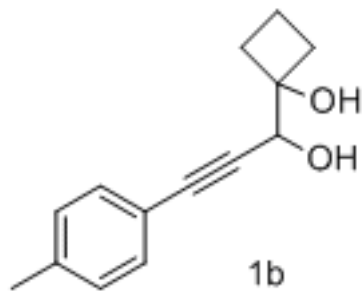
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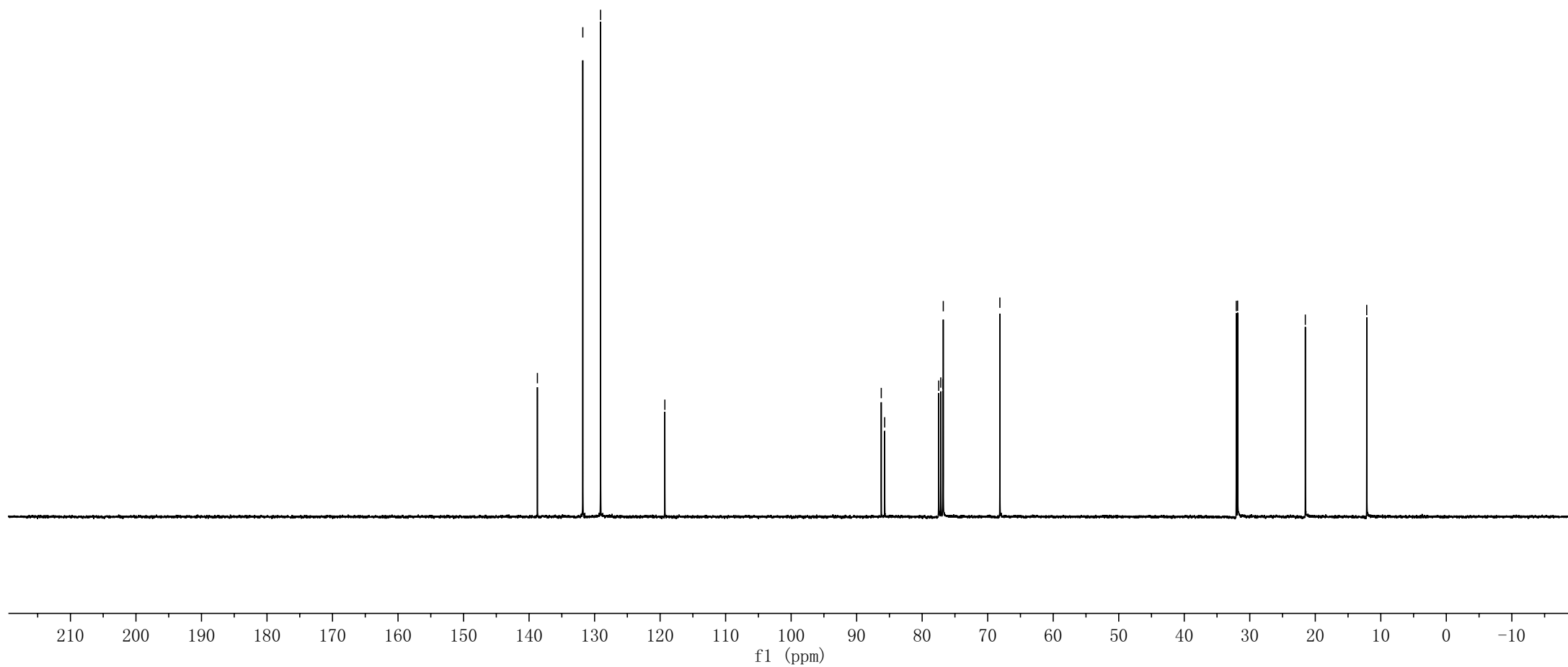
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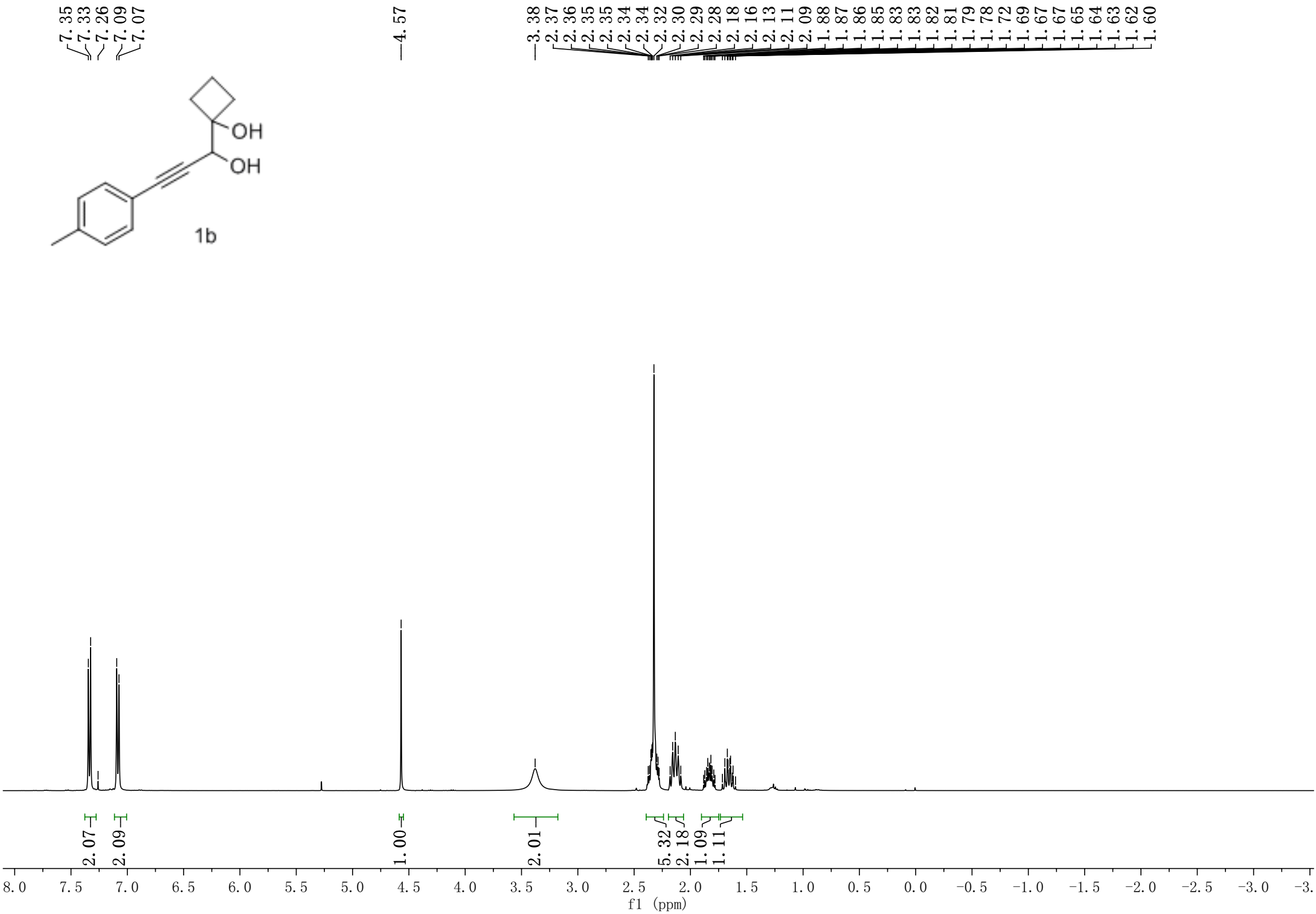


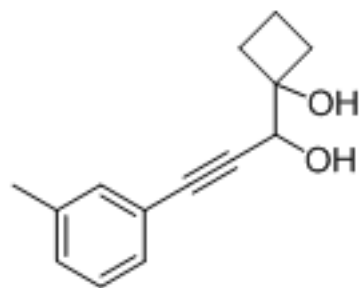




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 —129.09
 —119.28
 —86.24
 —85.72
 —77.16
 —76.84
 —76.18
 —75.18
 —32.06
 —31.85
 —21.52
 —12.15





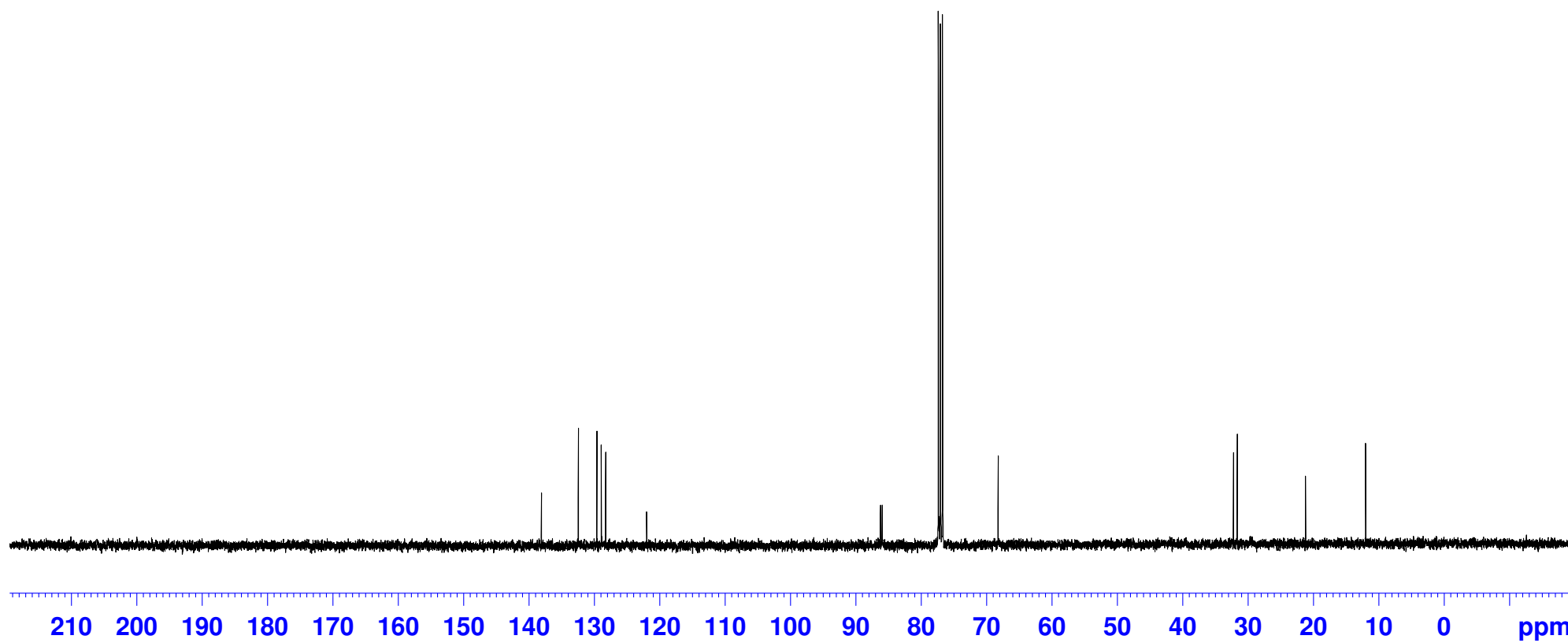


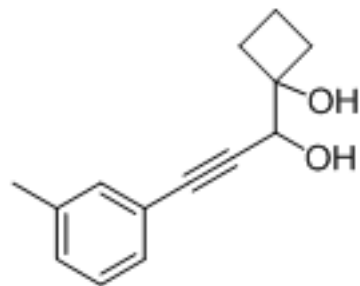
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 — 132.42
 — 129.57
 — 128.91
 — 128.24
 — 121.96

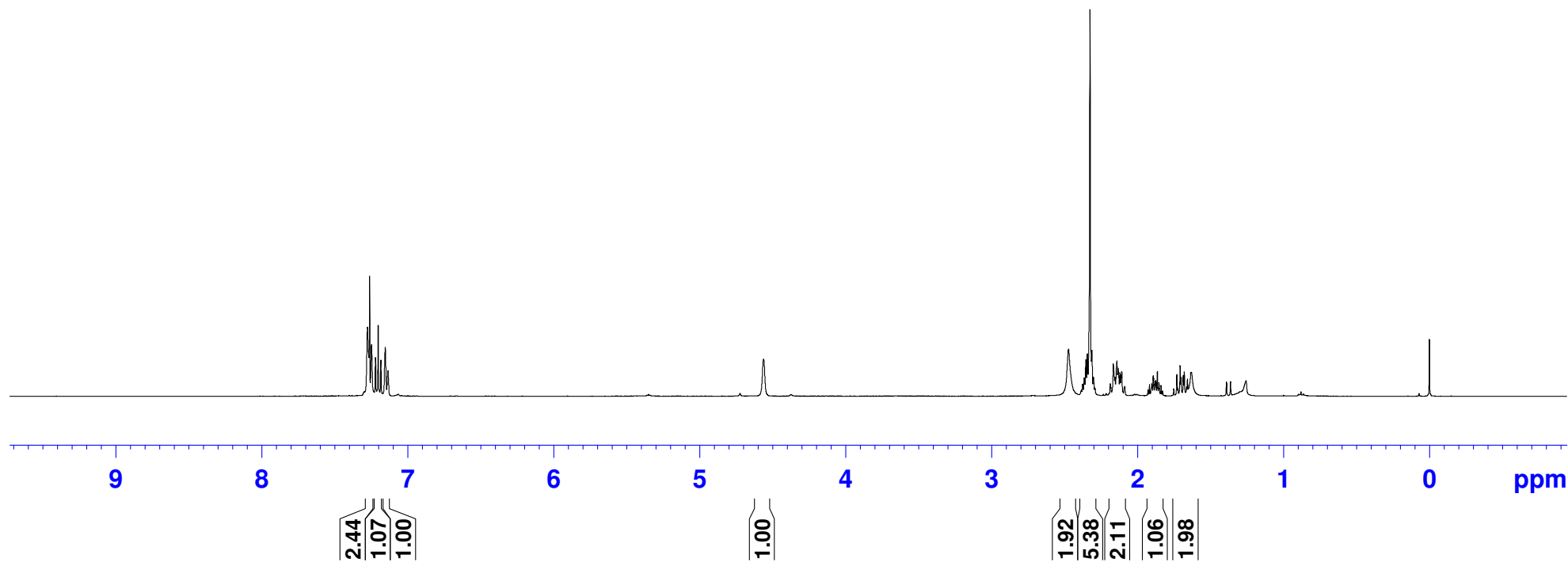
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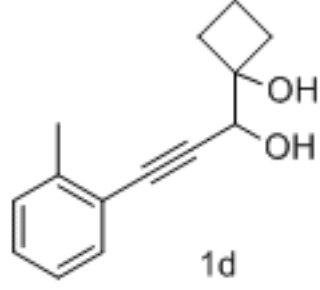
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1c

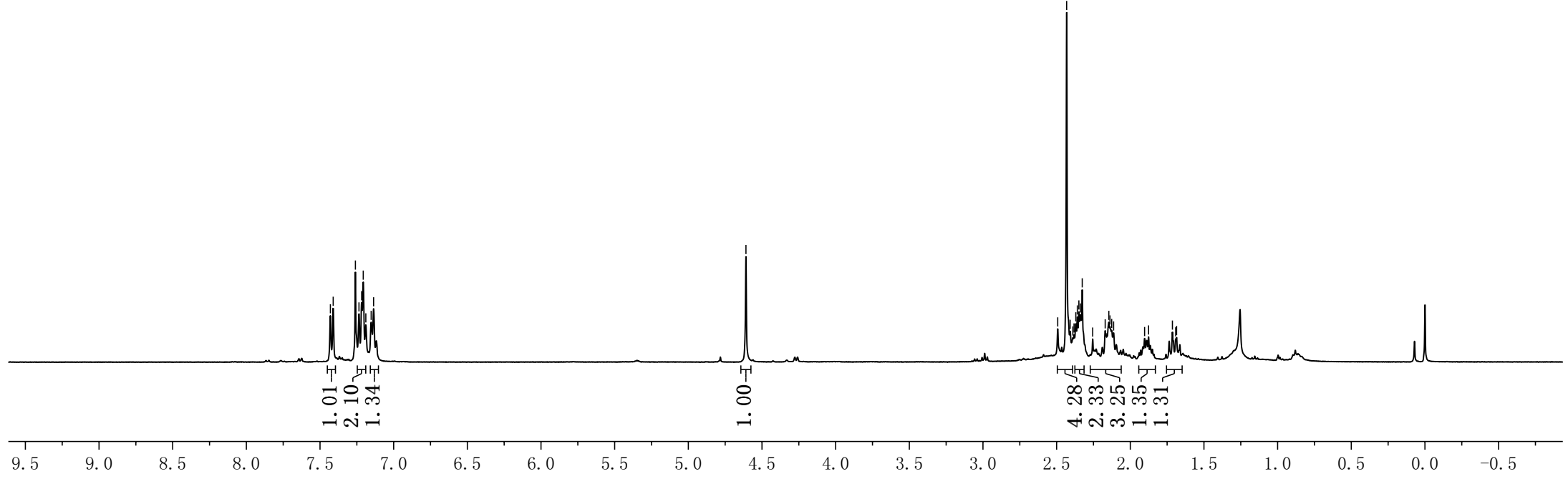


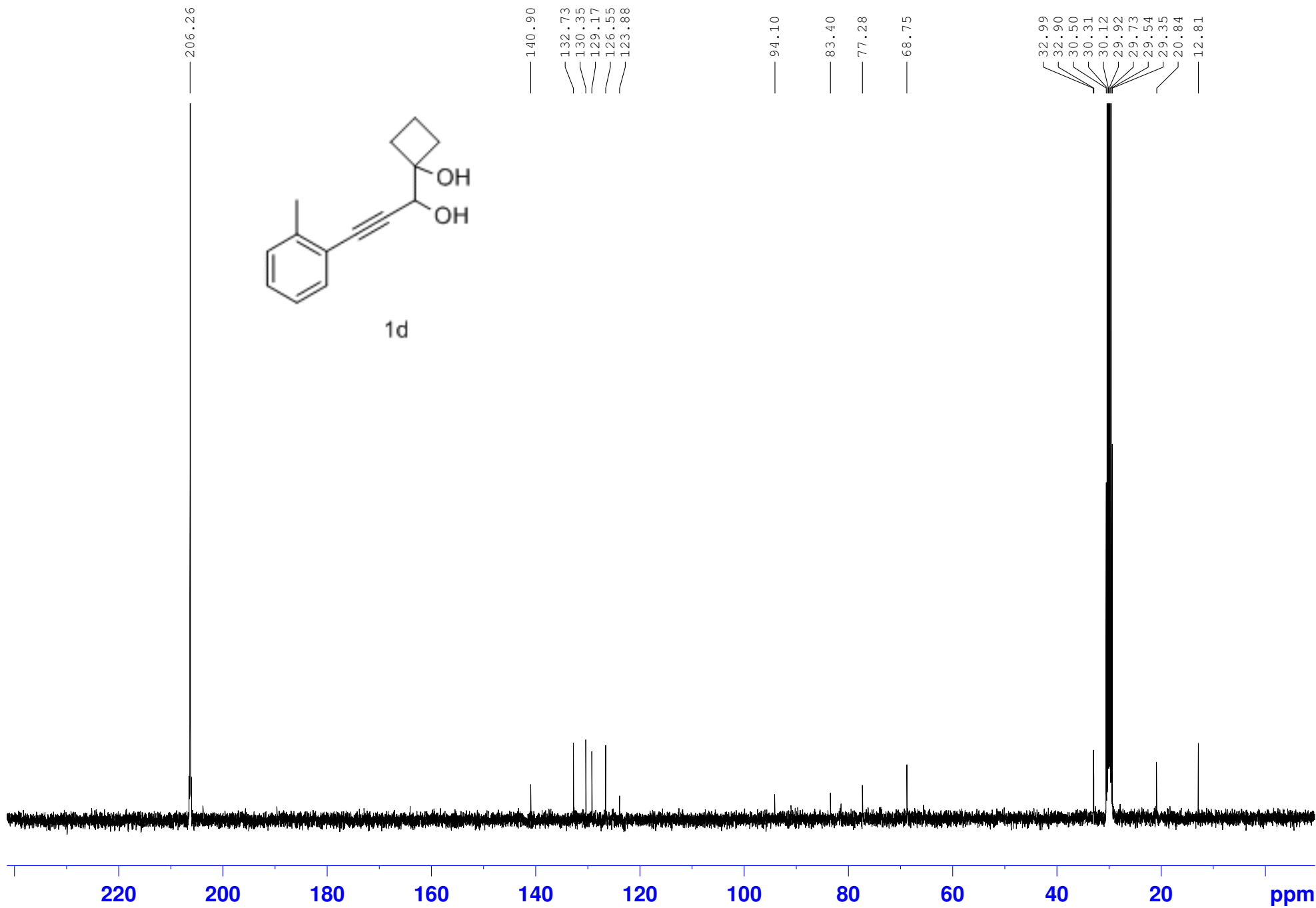


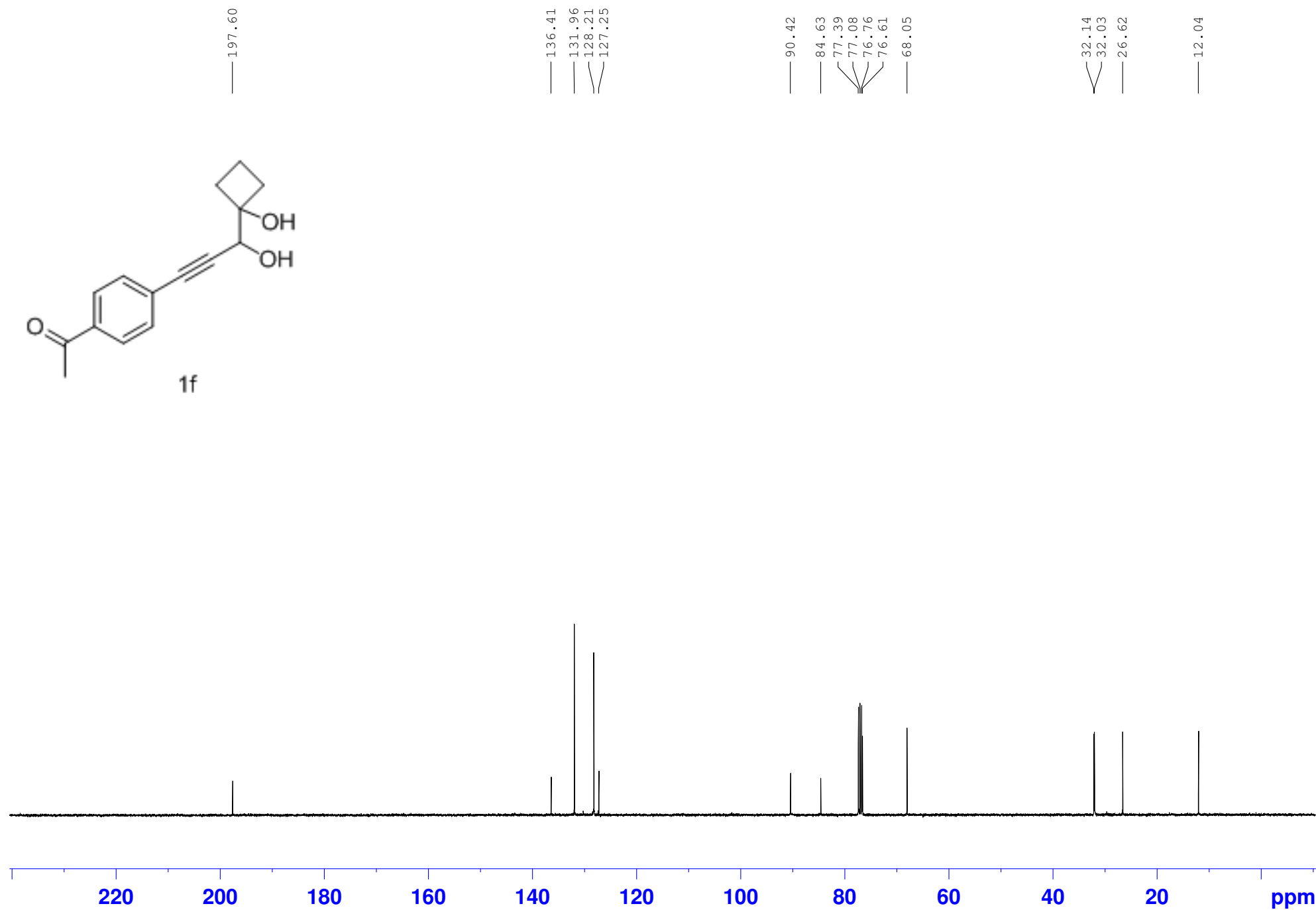
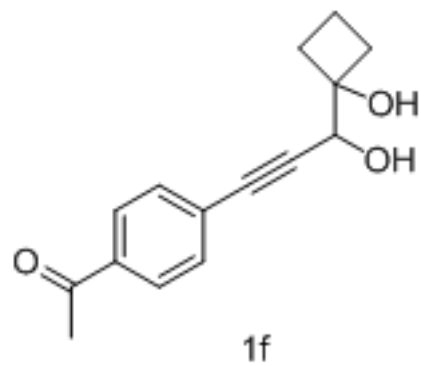
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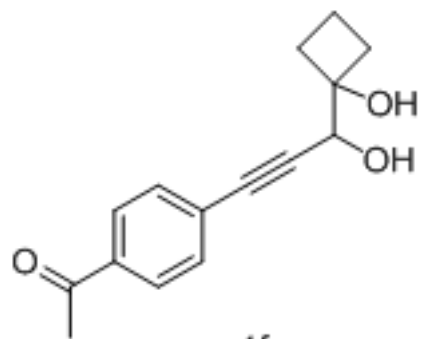
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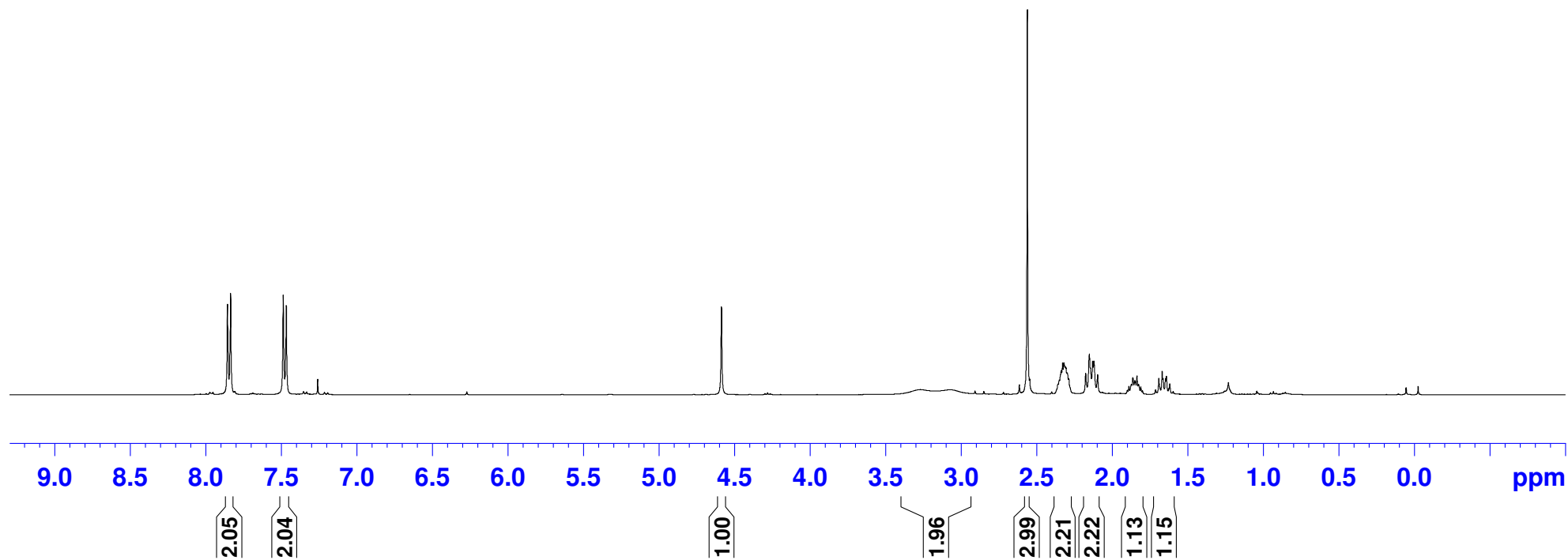






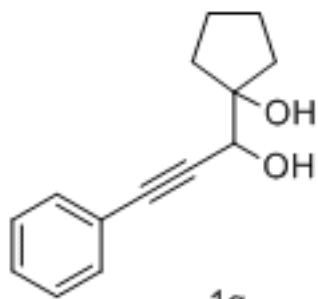


1f



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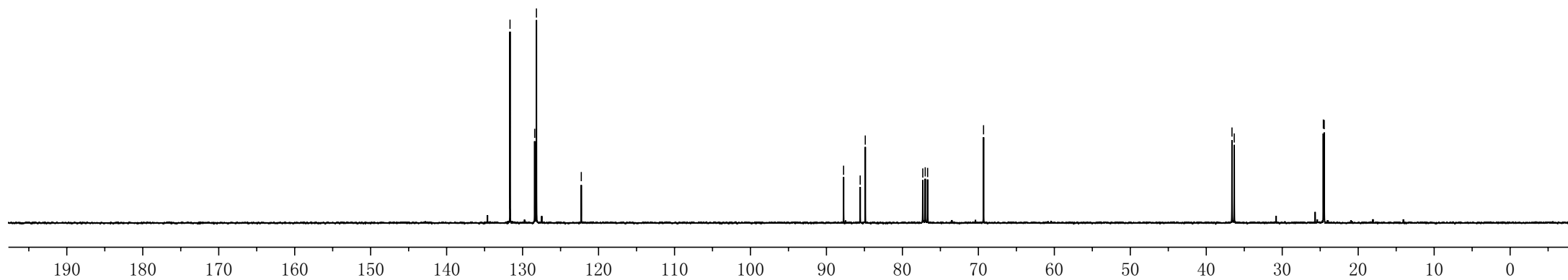
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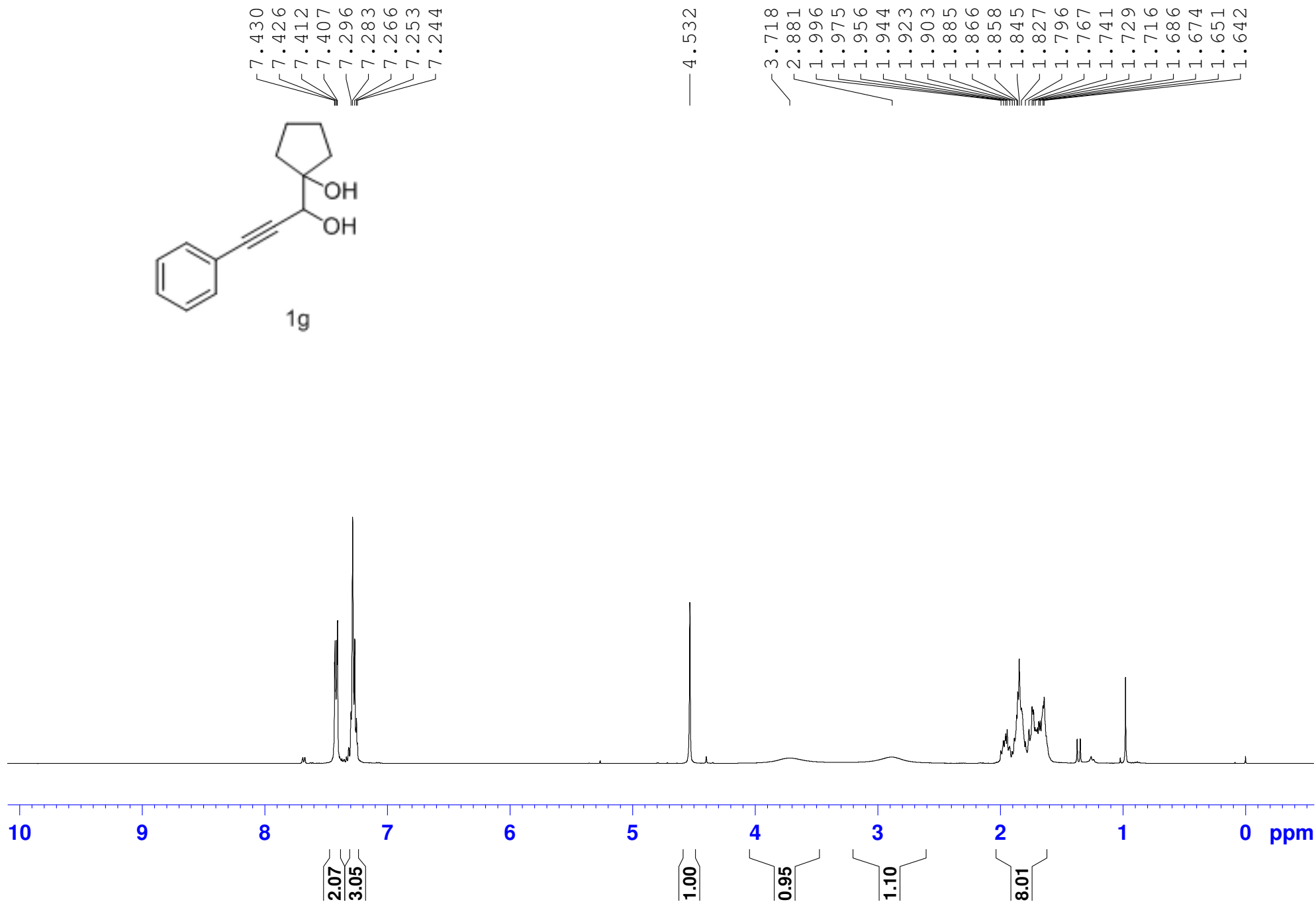
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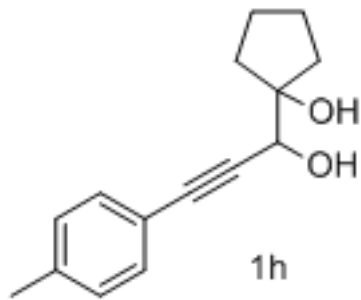
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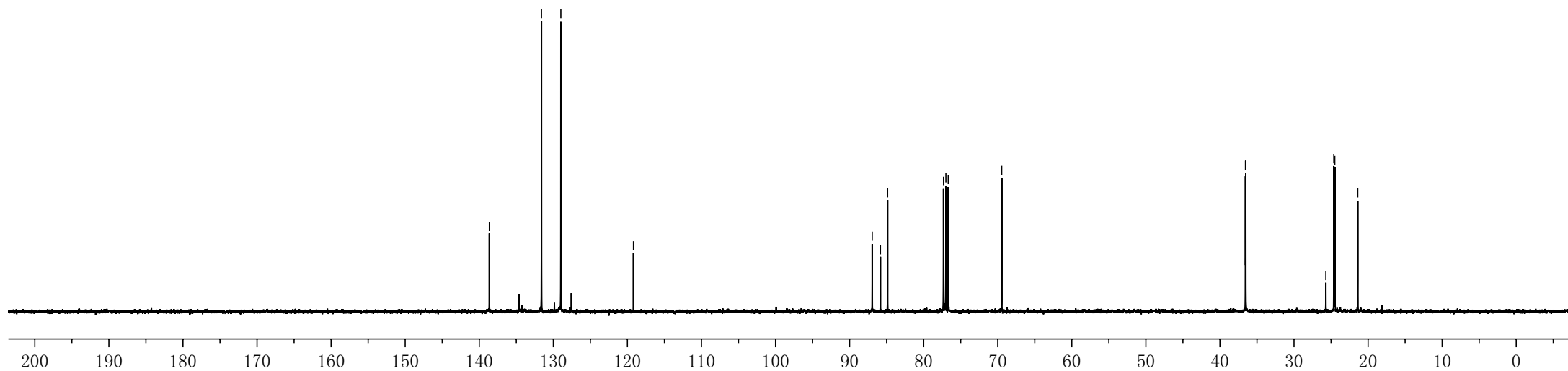
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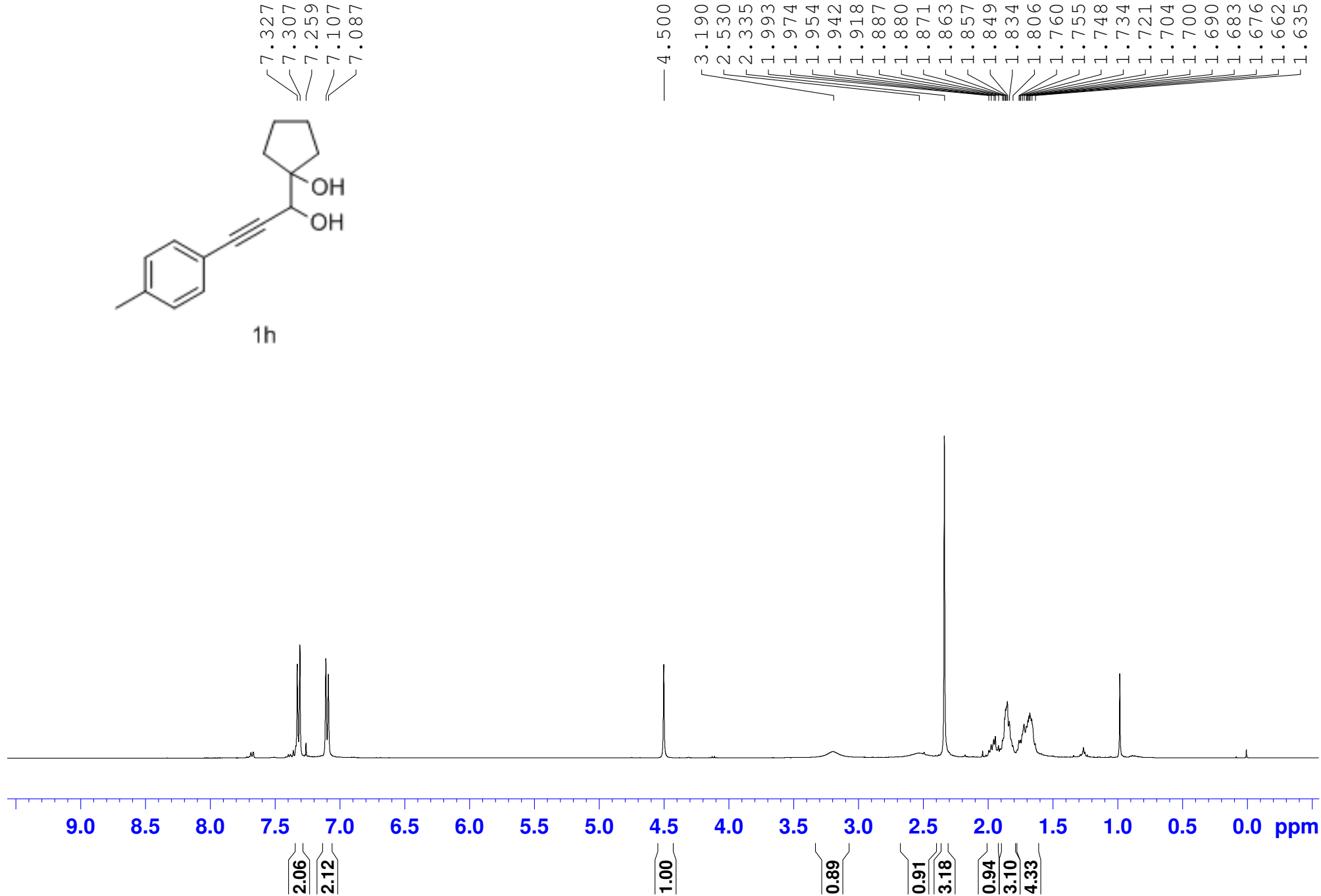
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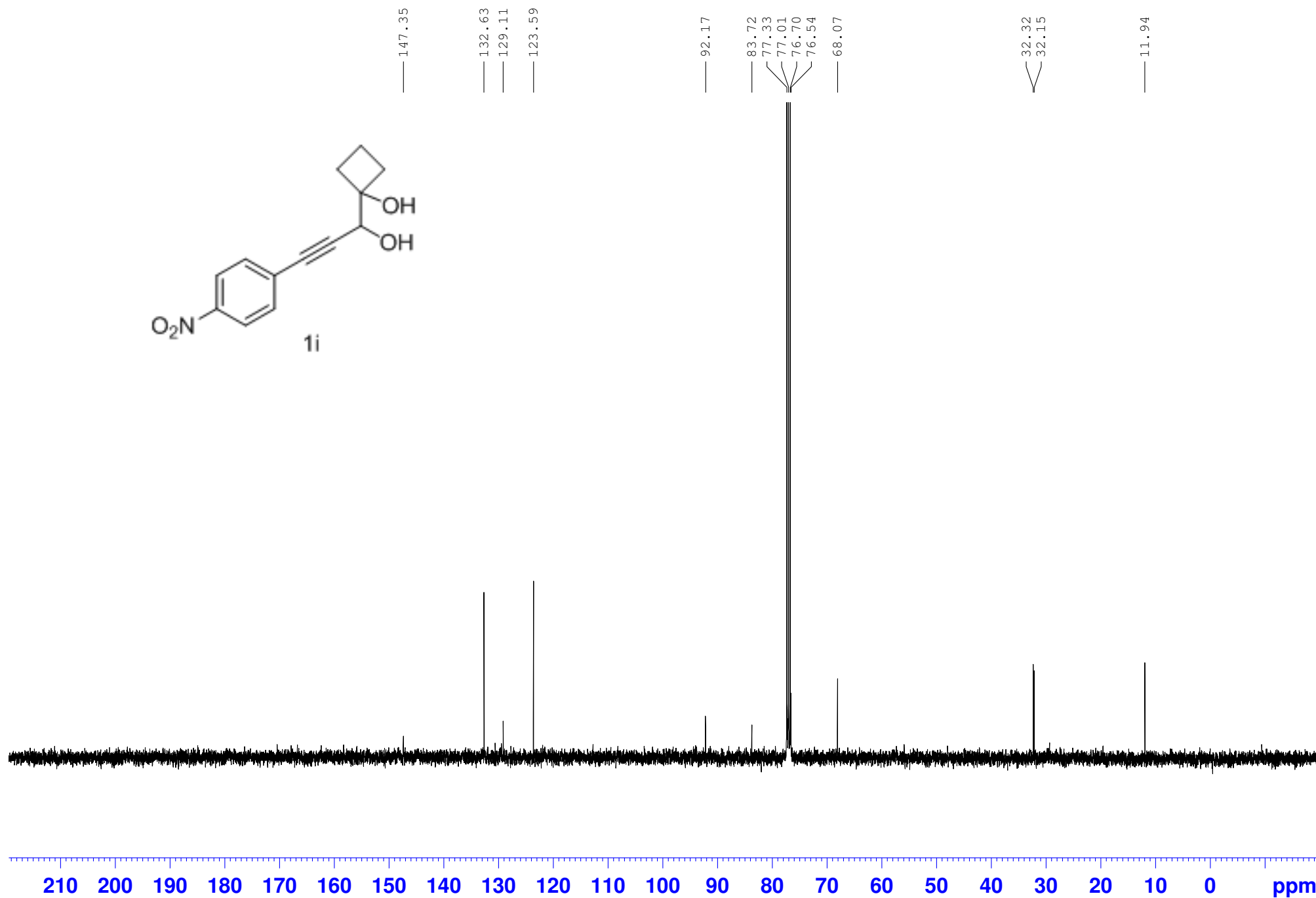
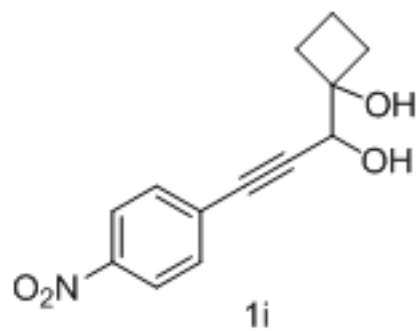
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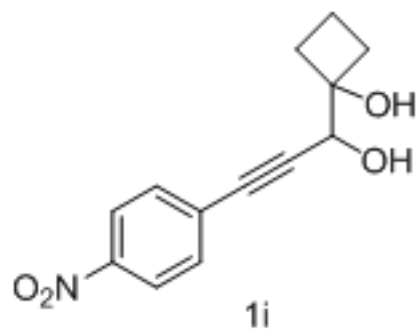
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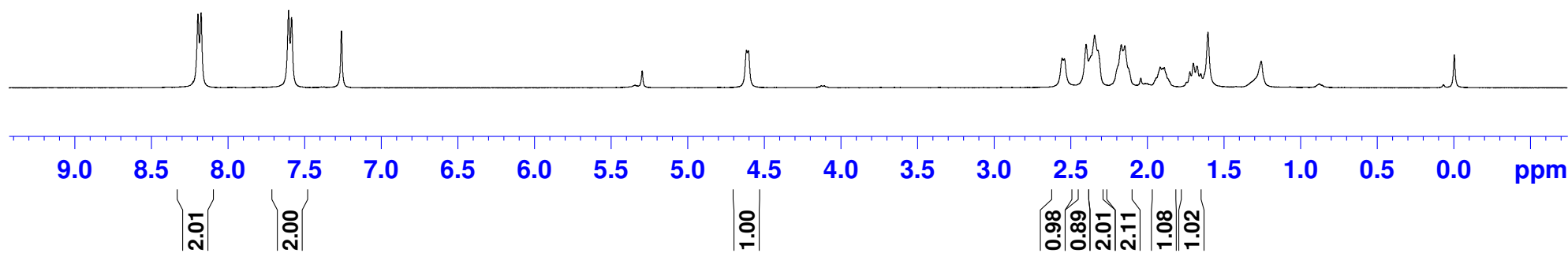
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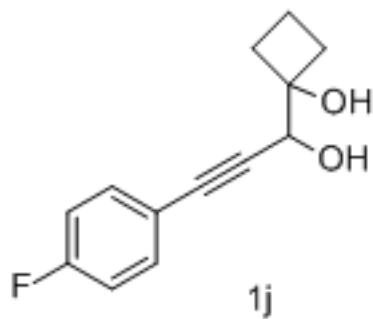
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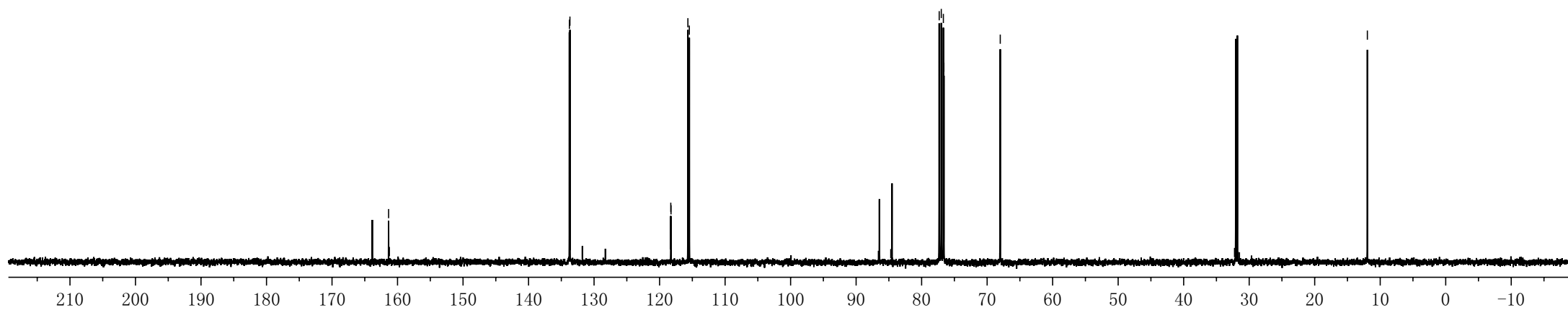
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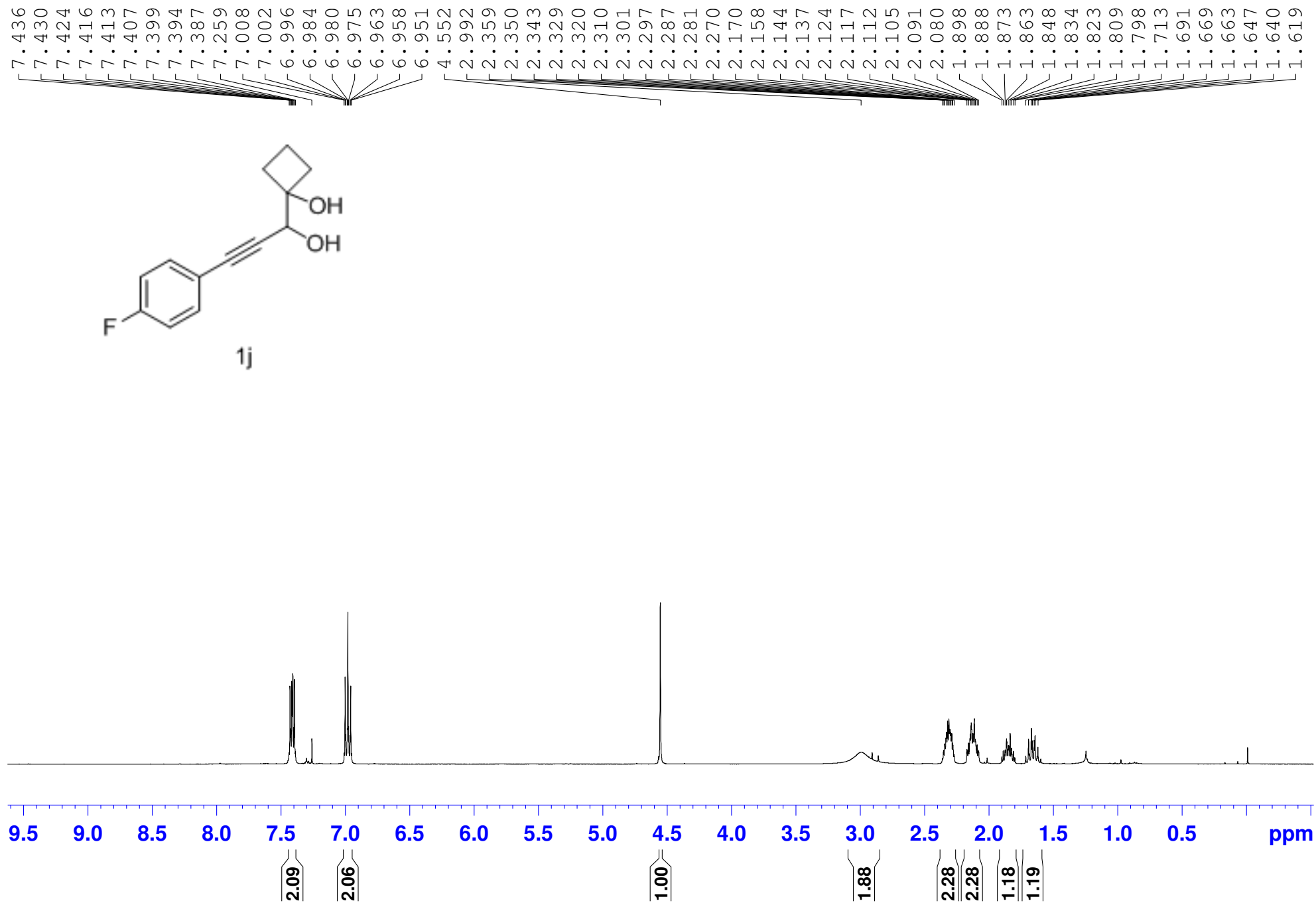
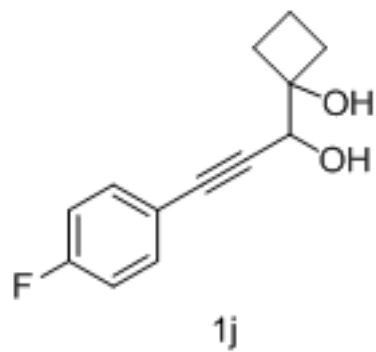
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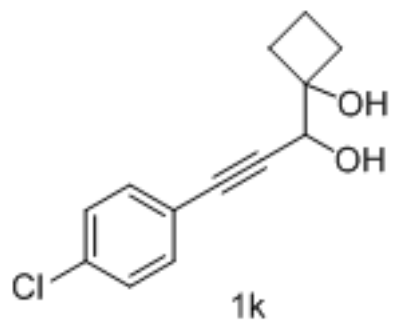
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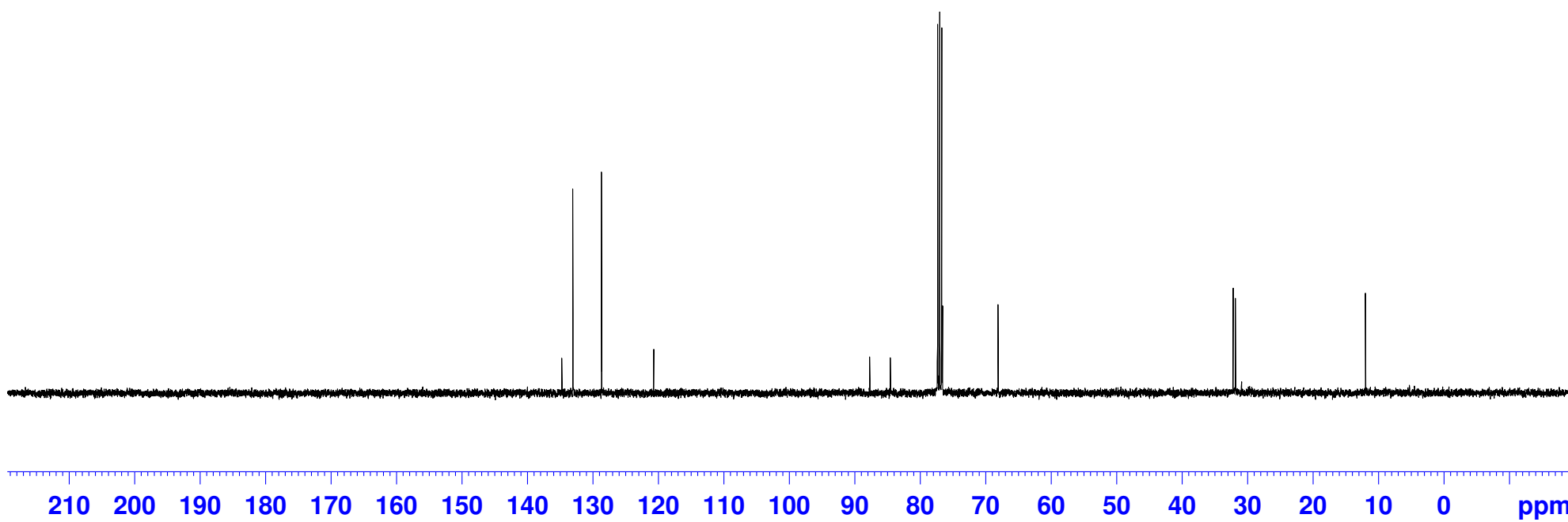


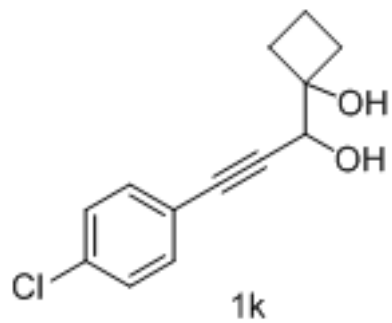
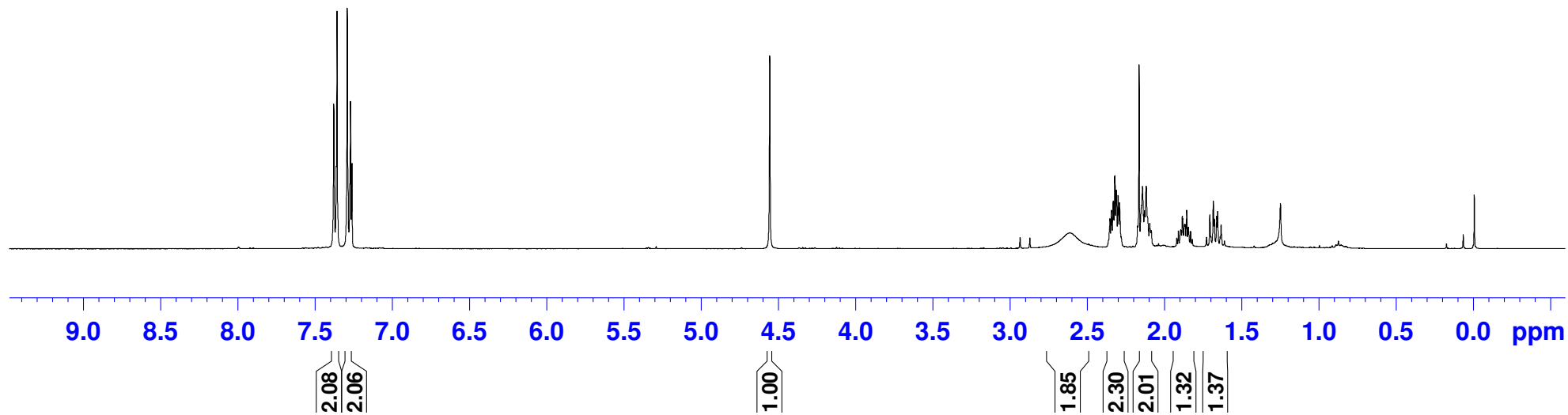
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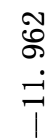
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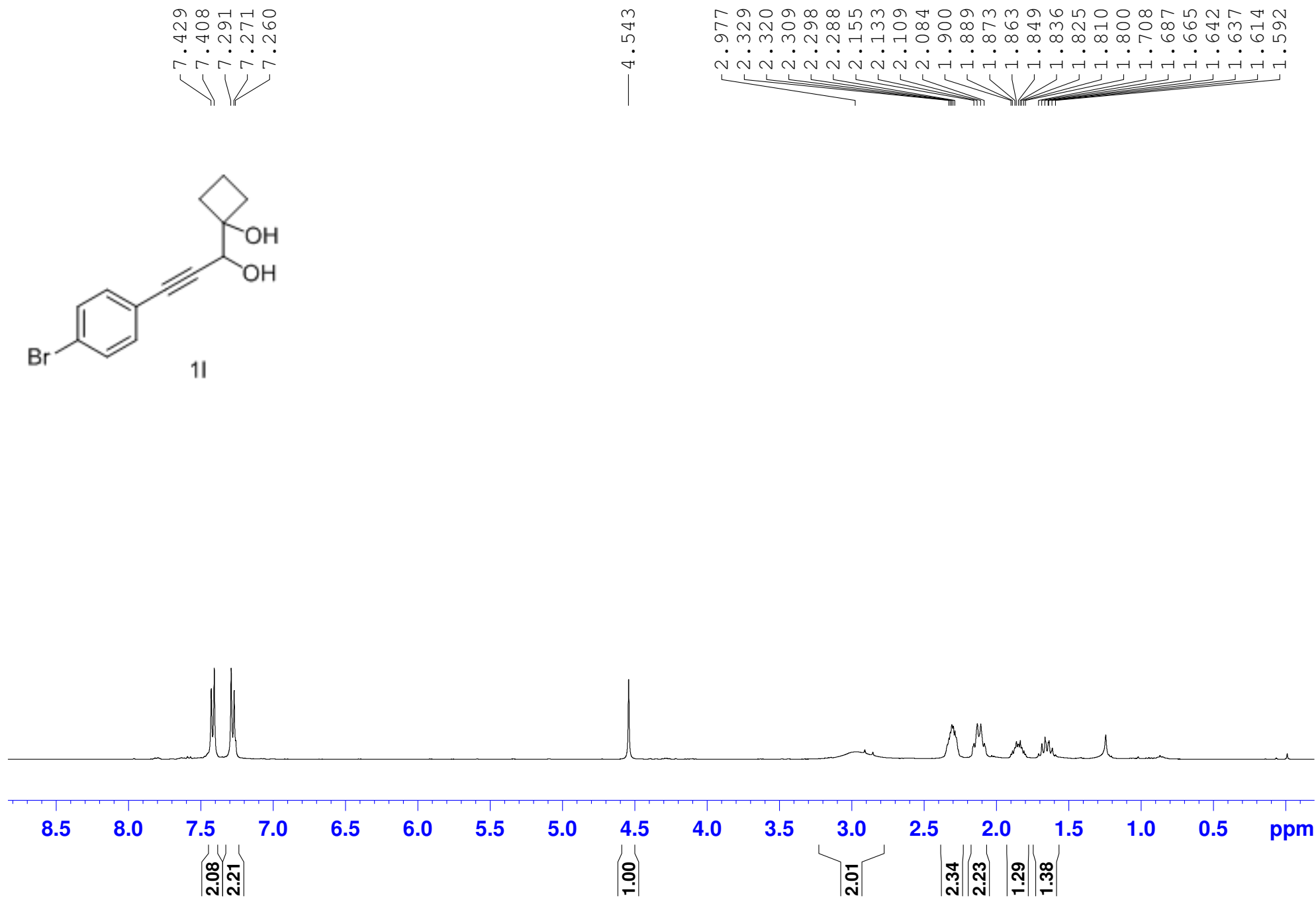


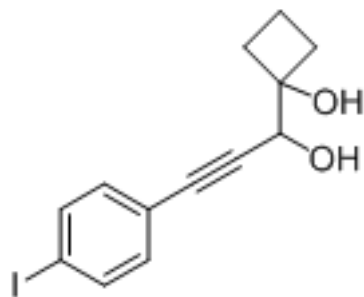


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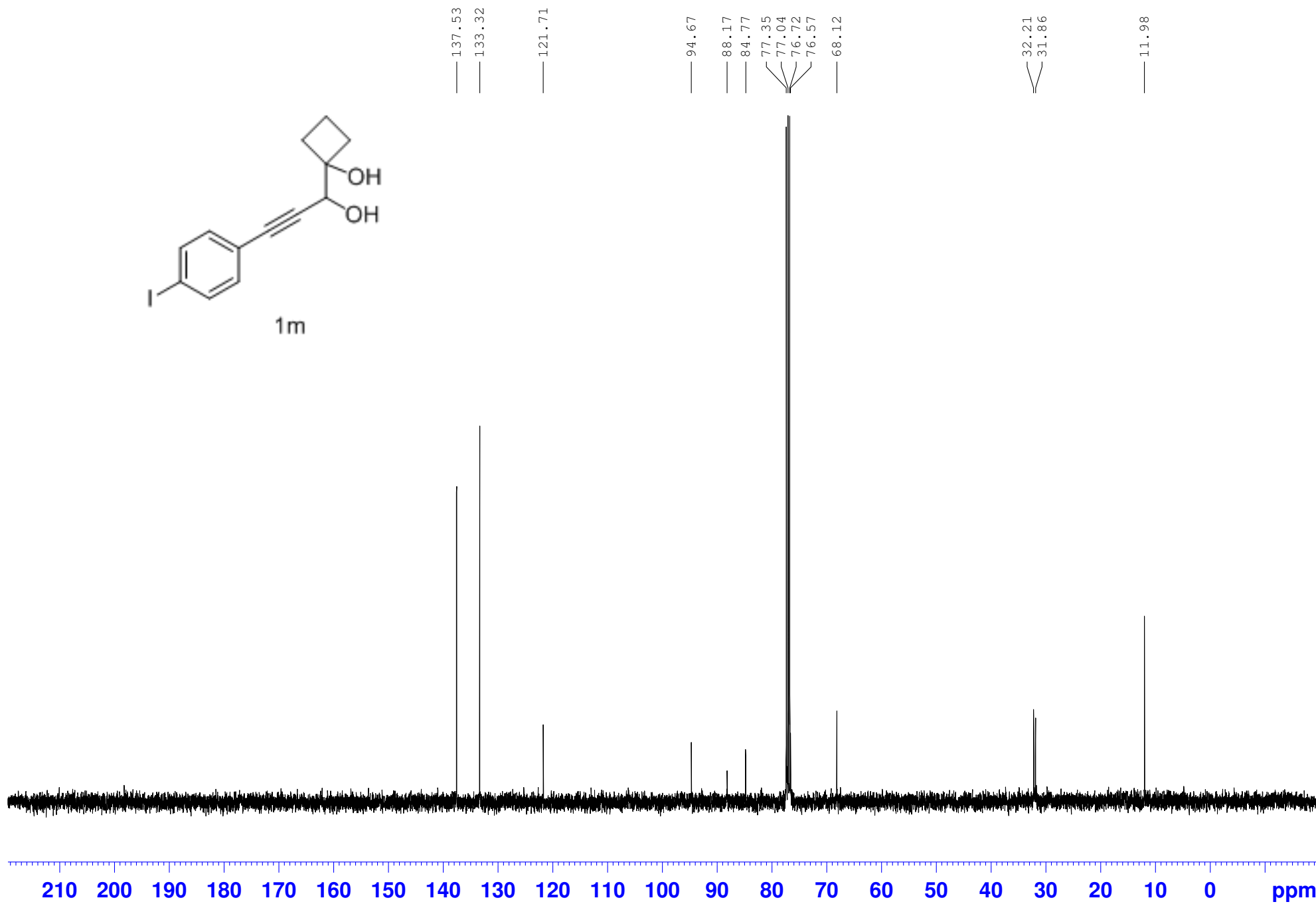
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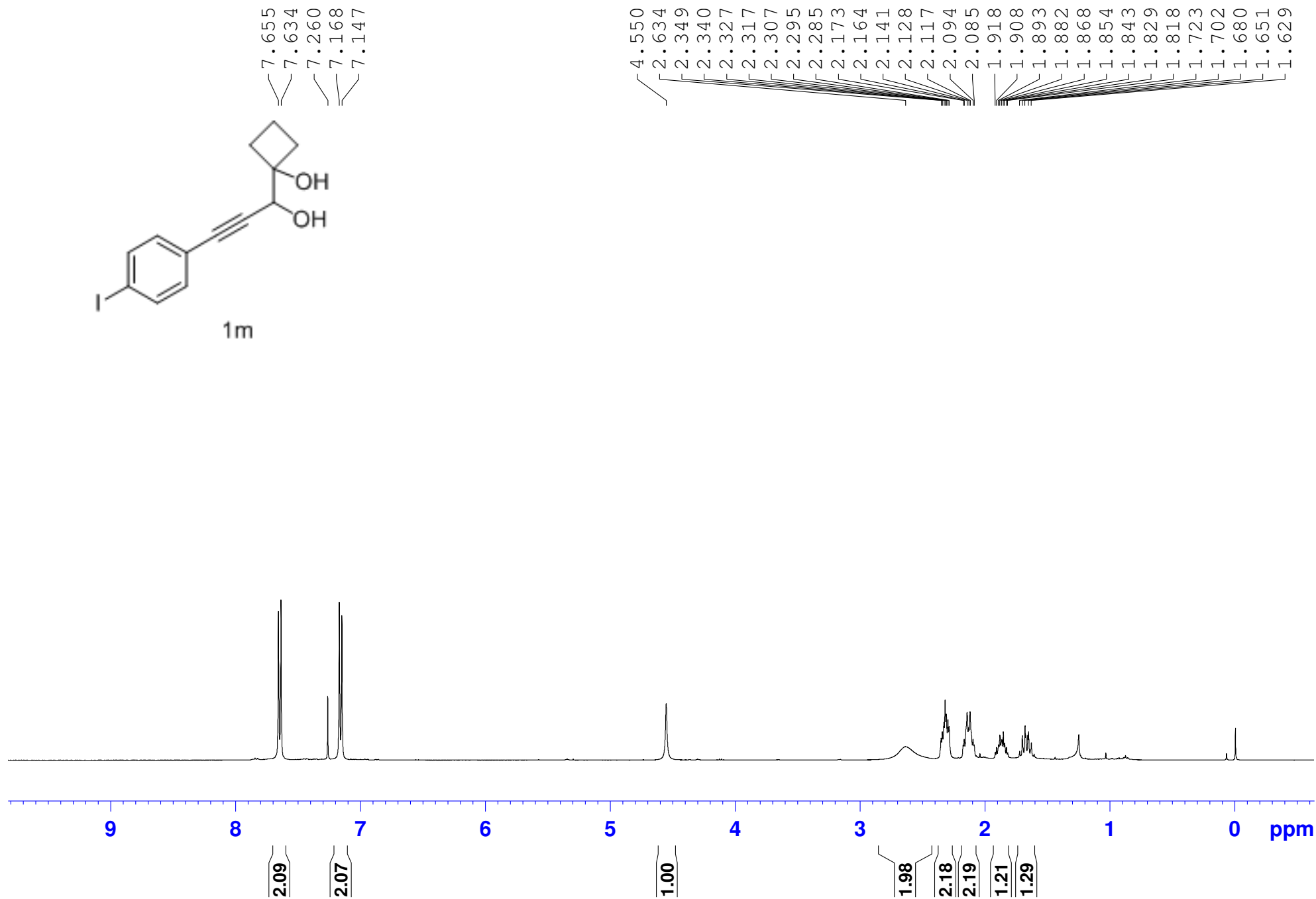


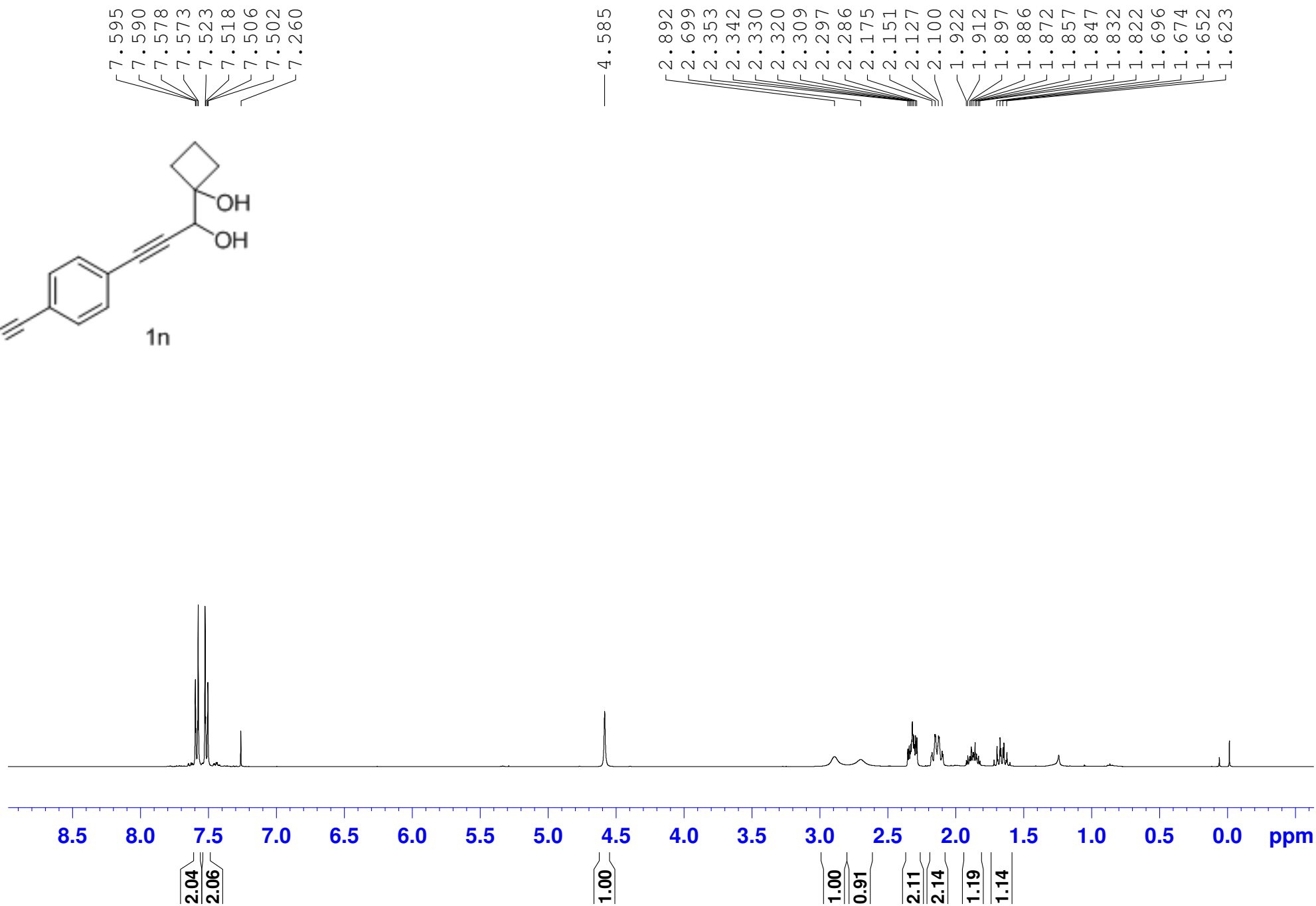
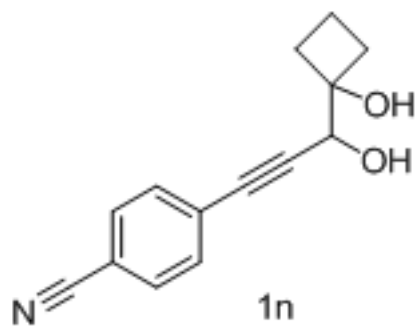


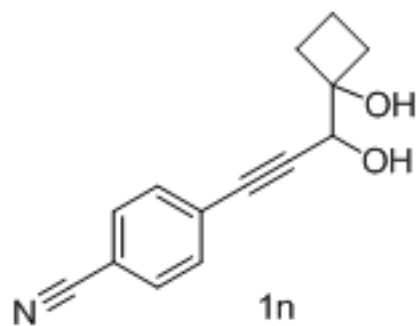


1m









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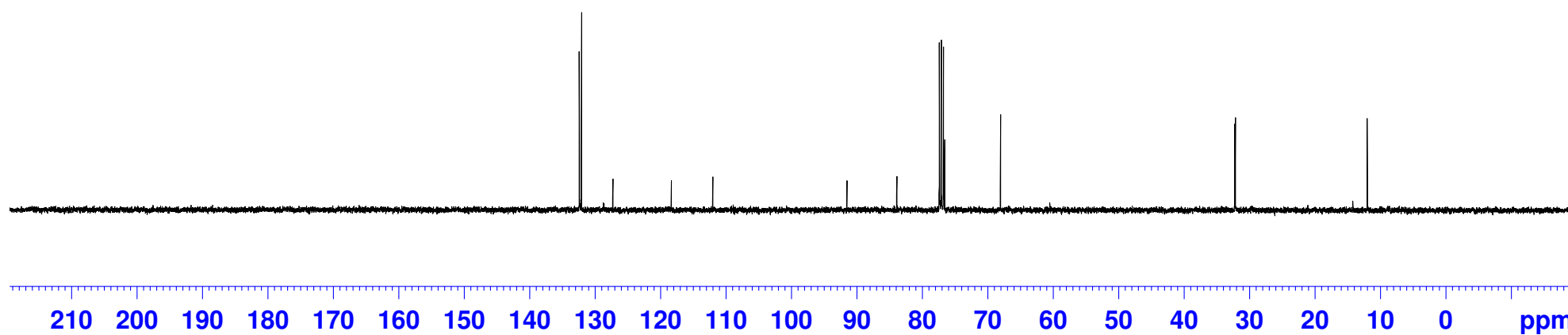
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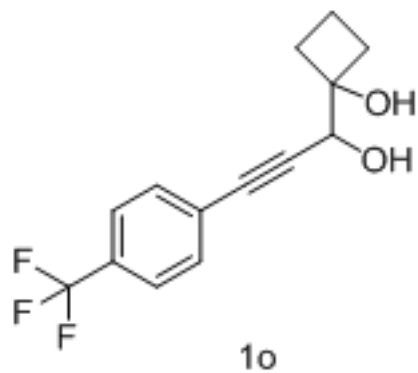
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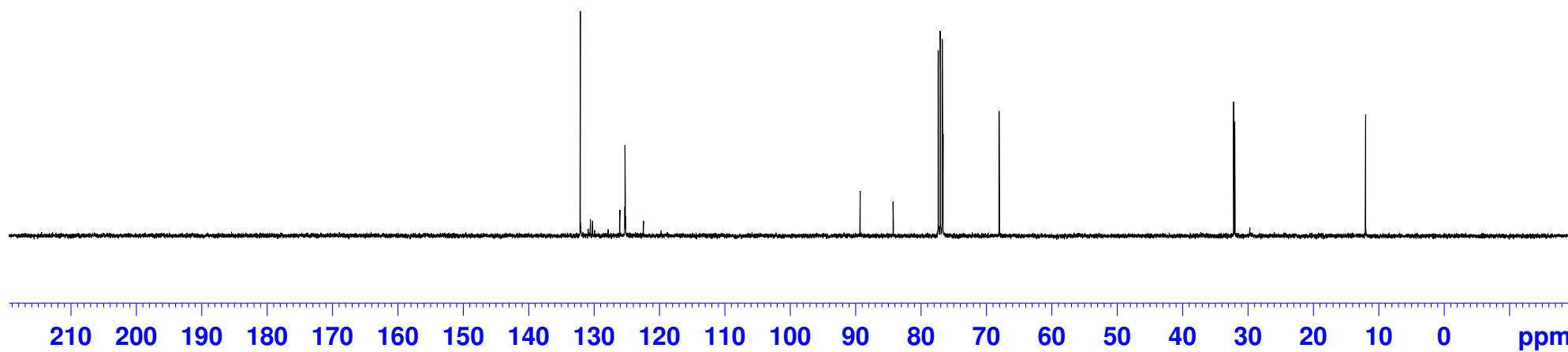


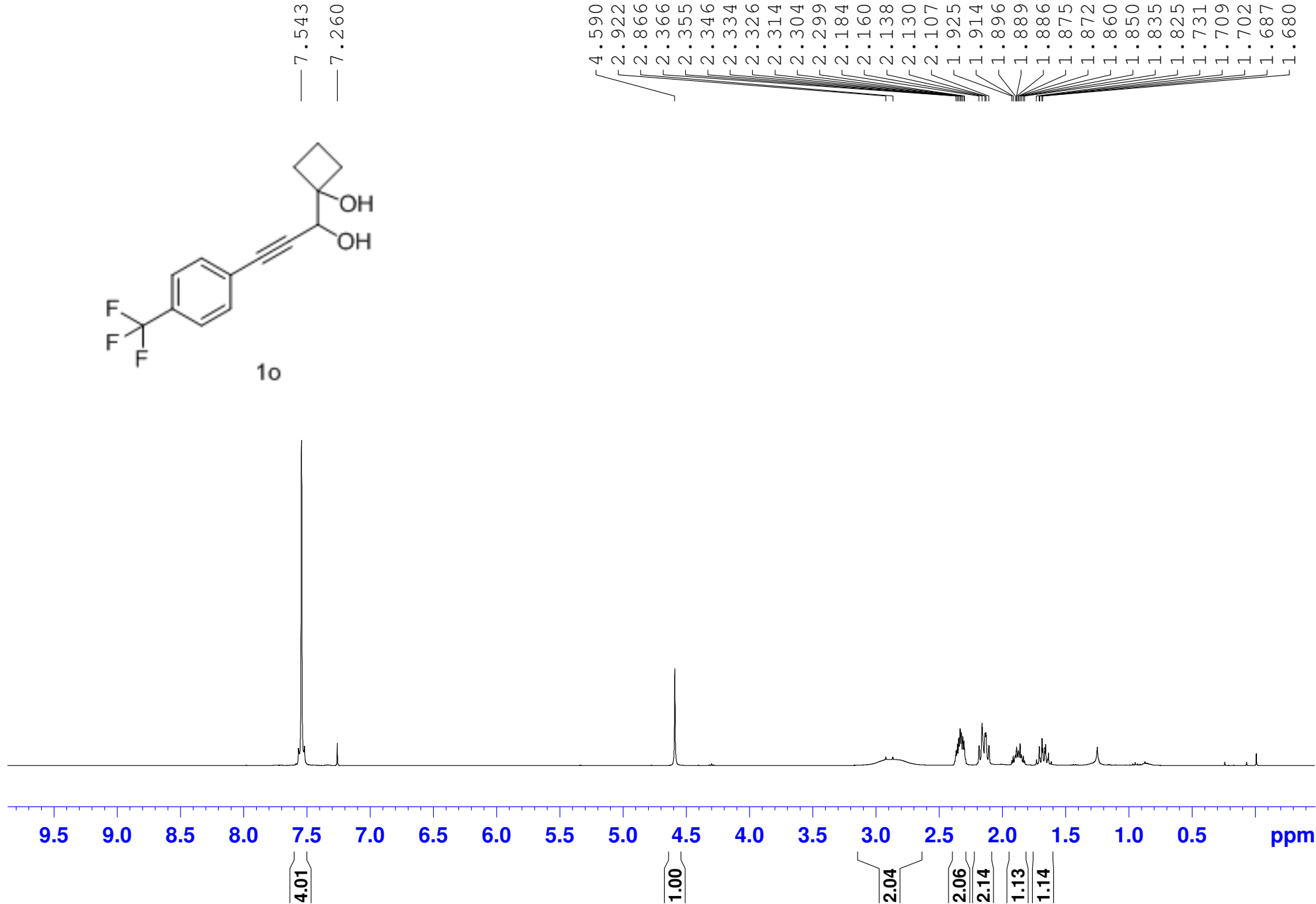
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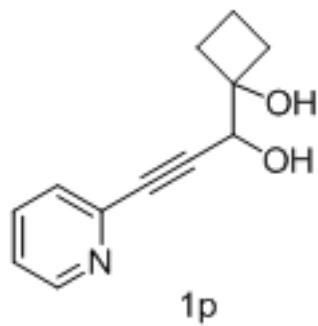
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77.34
77.02
76.71
76.62
68.03

32.18
31.98

11.98





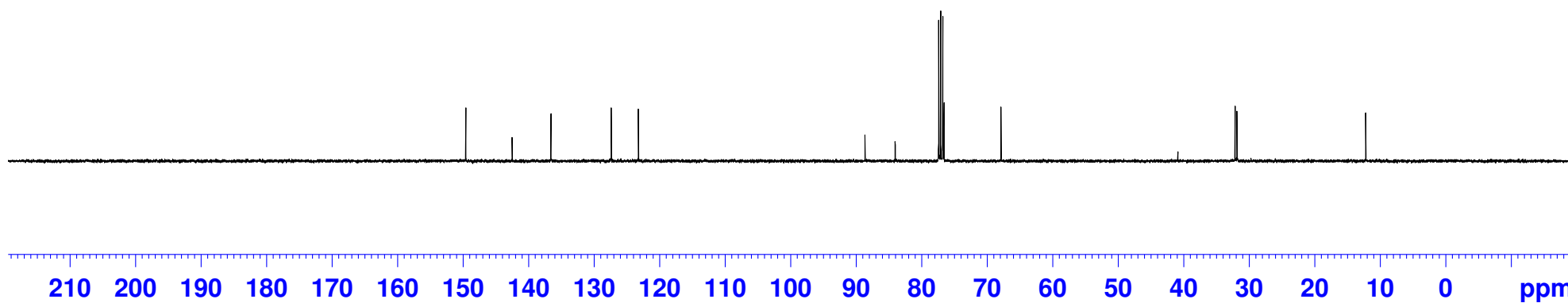


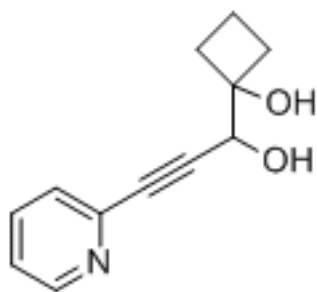
— 149.53
 — 142.46
 — 136.54
 — 127.33
 — 123.19

— 88.62
 — 83.99
 77.36
 77.05
 76.73
 76.55
 — 67.85

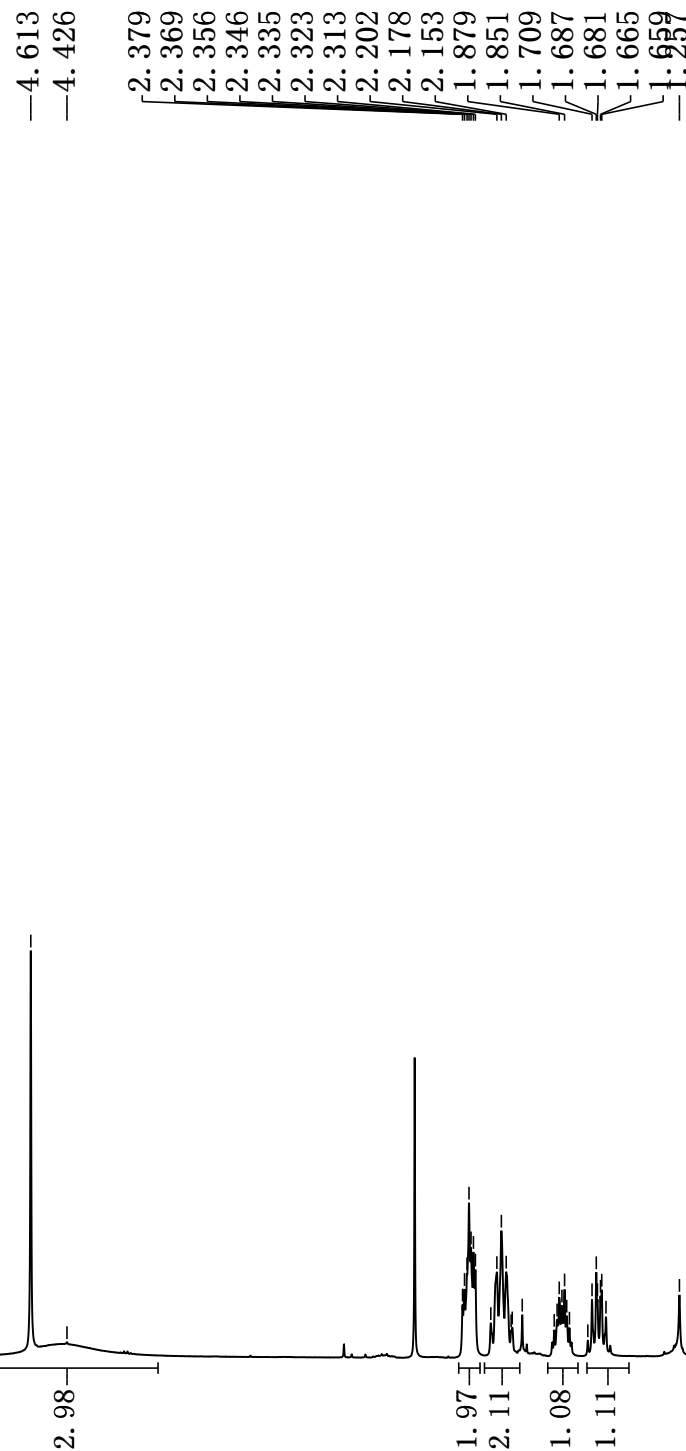
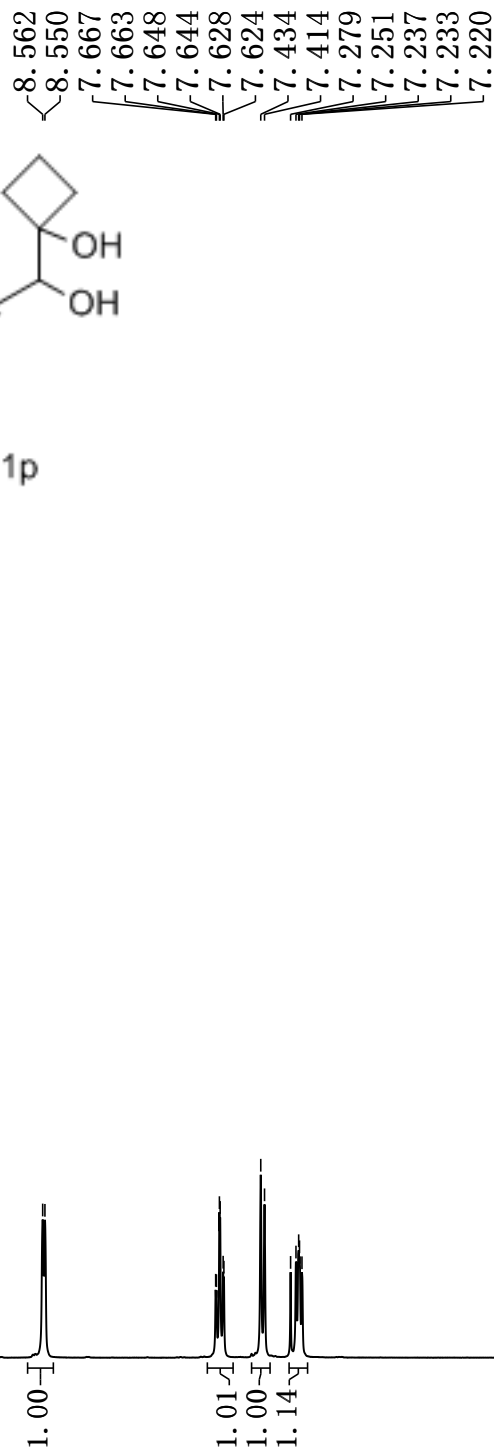
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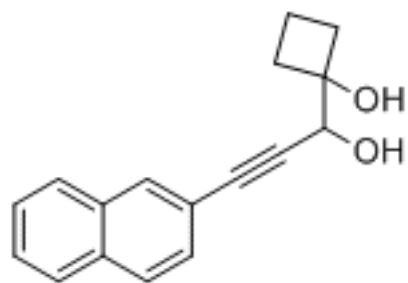
— 12.18





1p





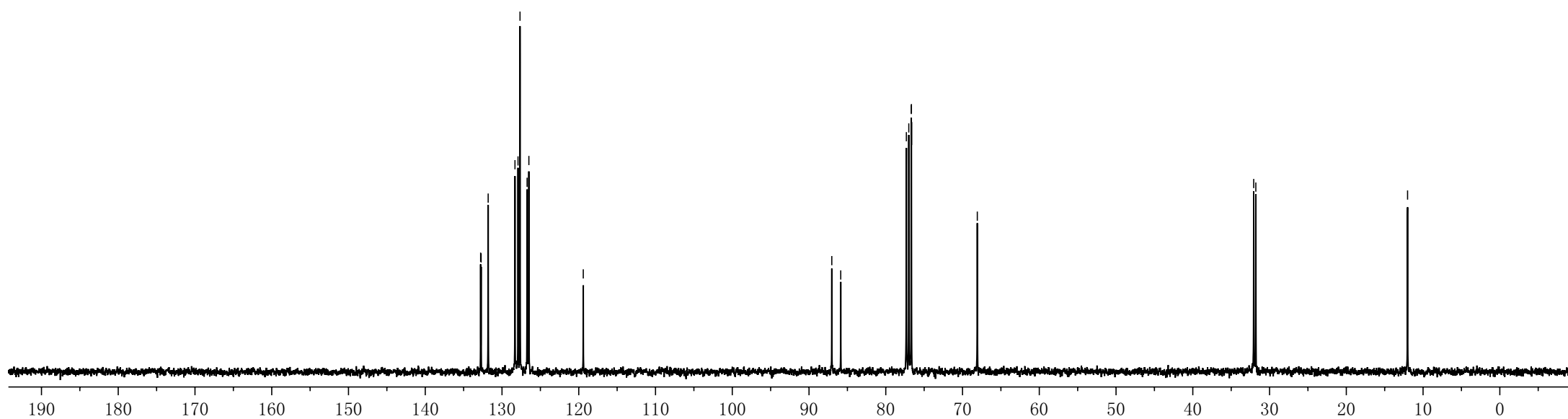
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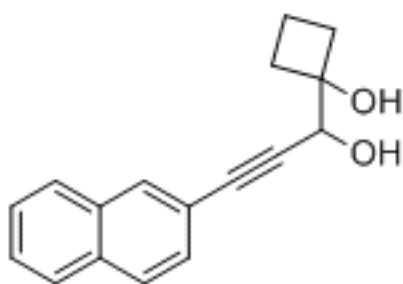
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131.802
128.310
127.913
127.653
126.731
126.499
—119.412

87.032
85.867
77.318
77.000
76.678
76.660
—68.076

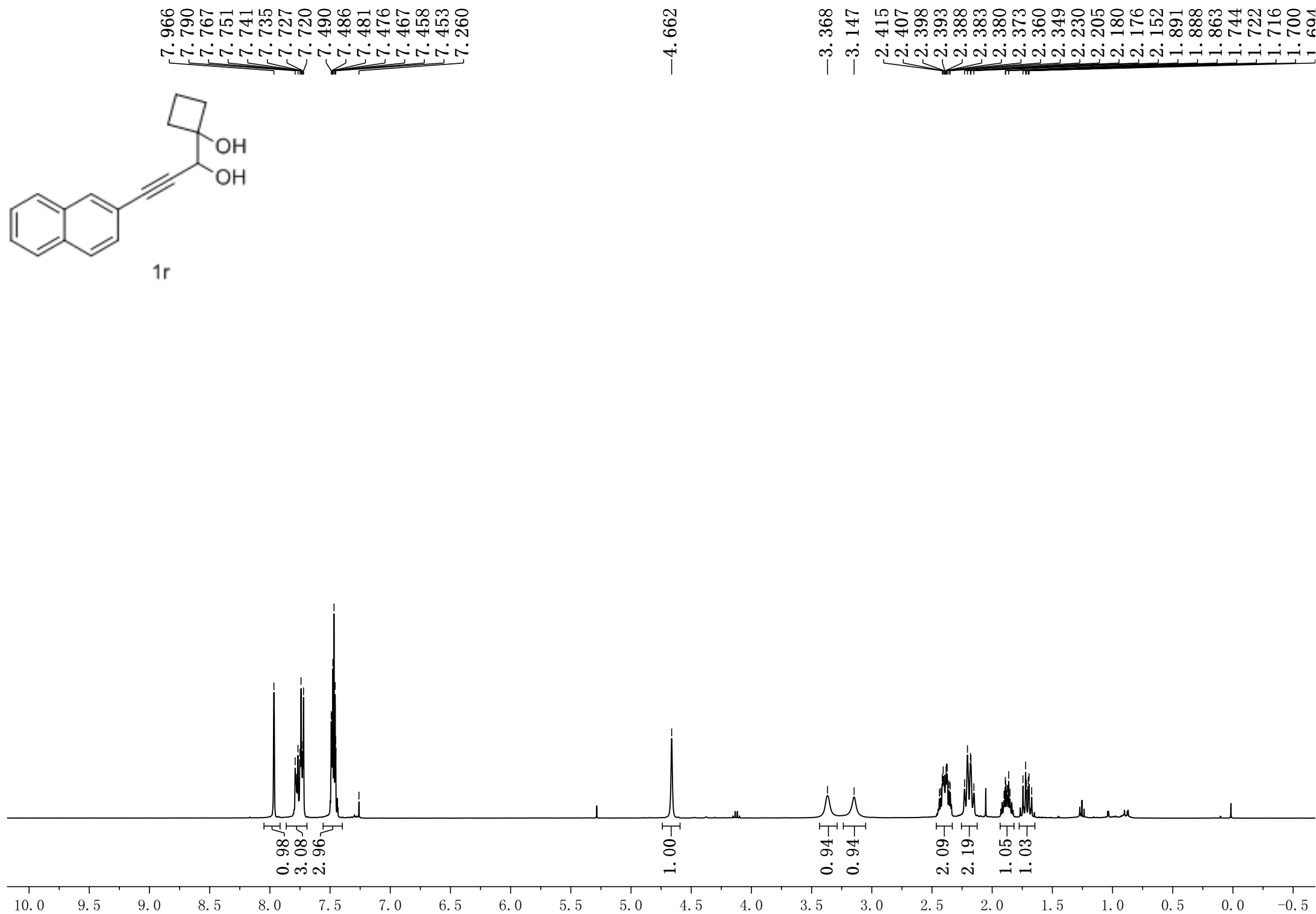
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31.789

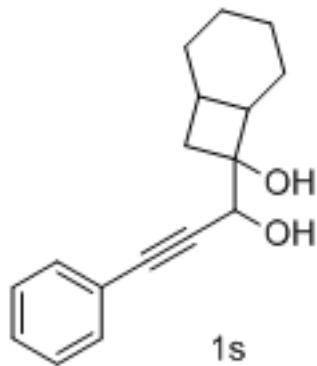
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1r

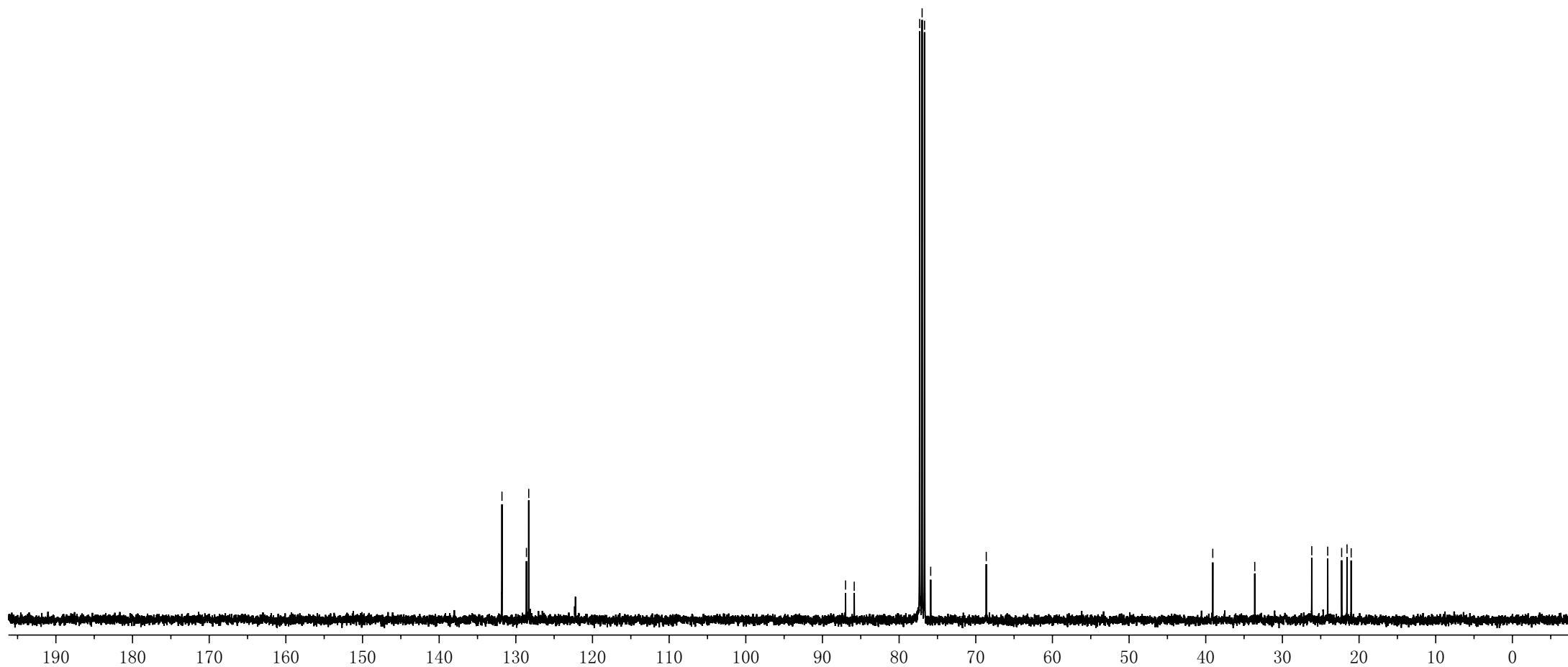


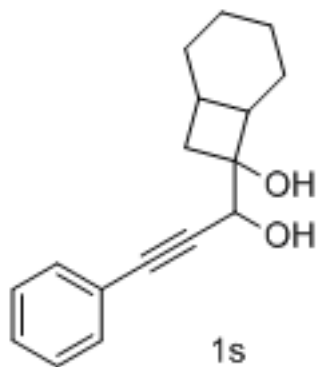


—131.808
—128.617
—128.317
—122.367

—86.989
—85.857
—77.317
—77.000
—76.682
—75.880
—68.632

—39.084
—33.606
—26.170
—24.091
—22.270
—21.572
—21.030

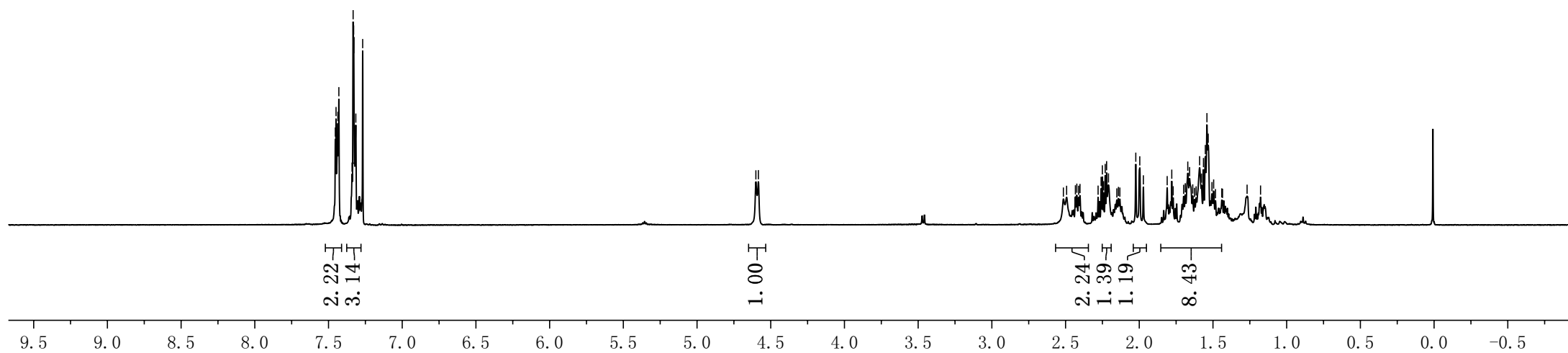




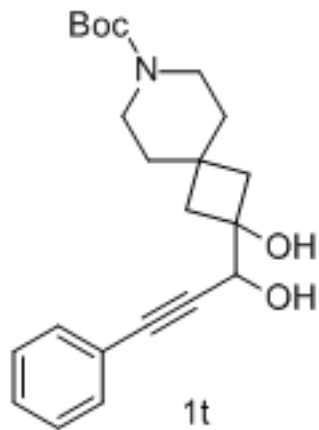
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7.340
7.333
7.329
7.323
7.315
7.268

4.601
4.584

2.493
2.433
2.424
2.411
2.402
2.279
2.259
2.250
2.242
2.231
2.222
2.209
2.024
1.997
1.972
1.811
1.779
1.770
1.699
1.688
1.671
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1.552
1.541
1.532
1.507
1.495
1.269
1.177



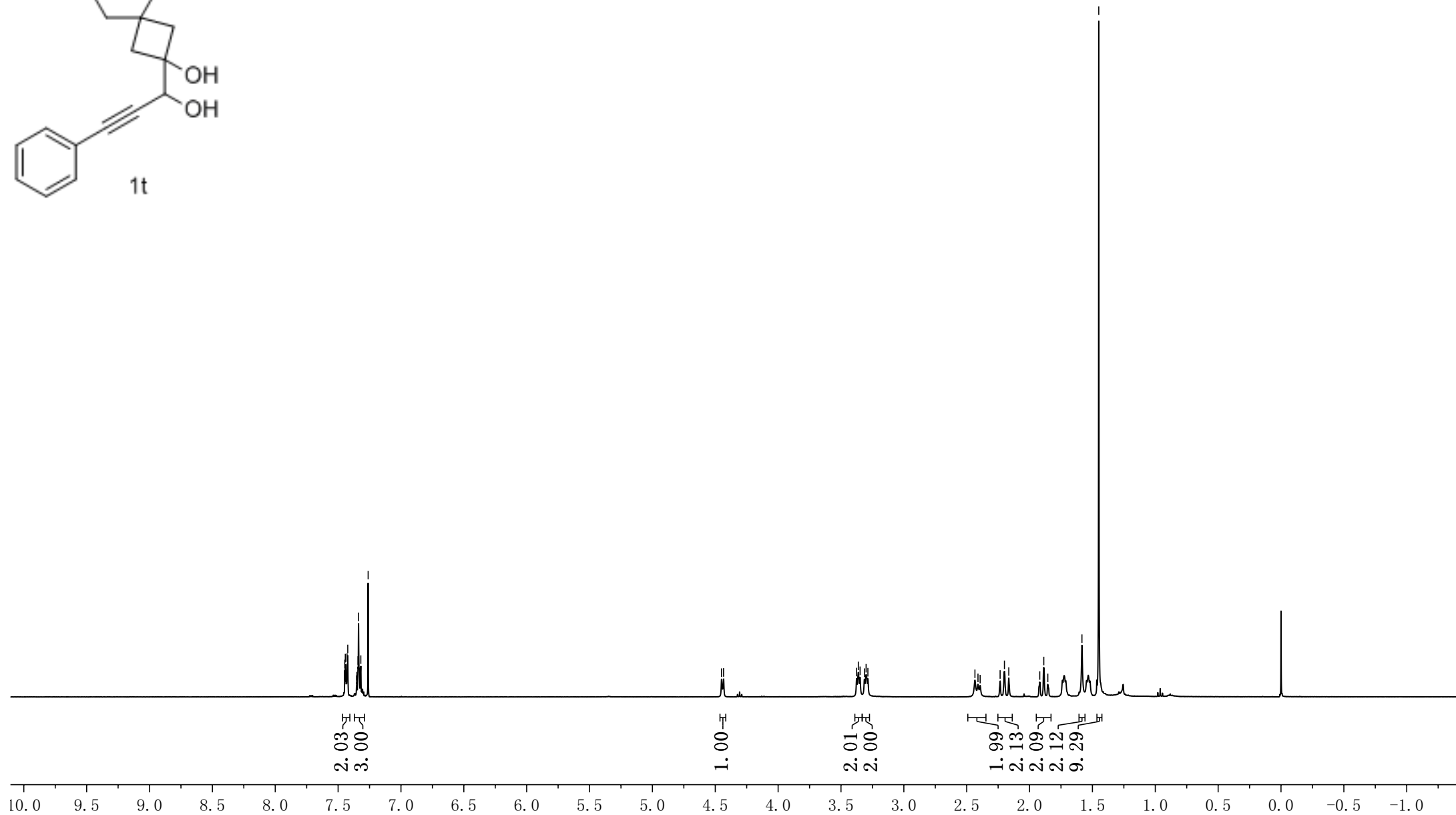


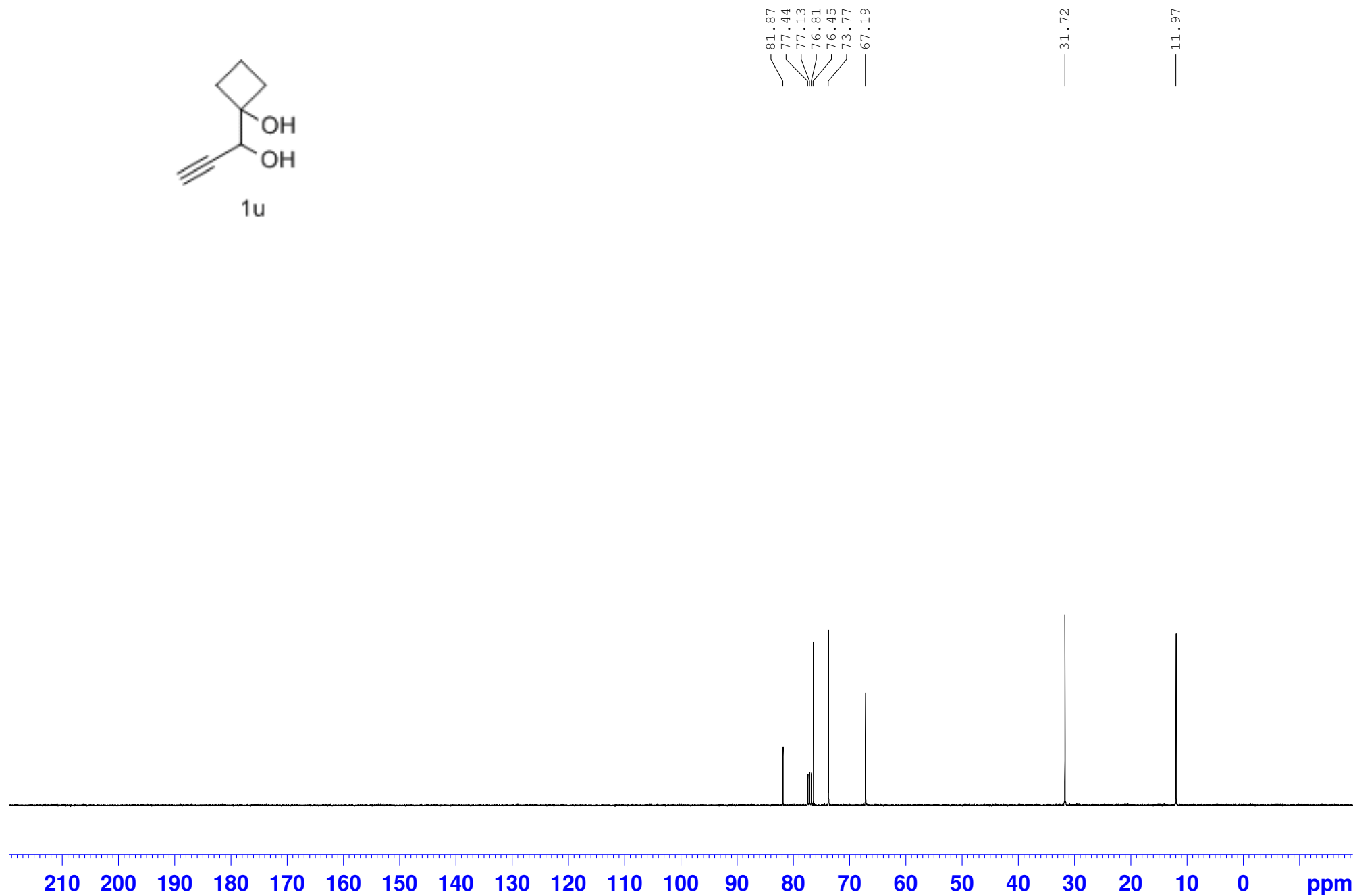
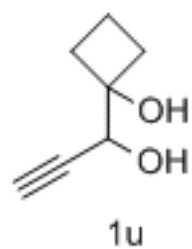


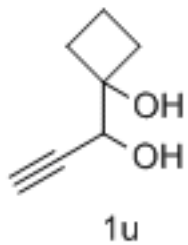
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7.439
7.434
7.429
7.423
7.354
7.351
7.342
7.337
7.319
7.261

4.450
4.434

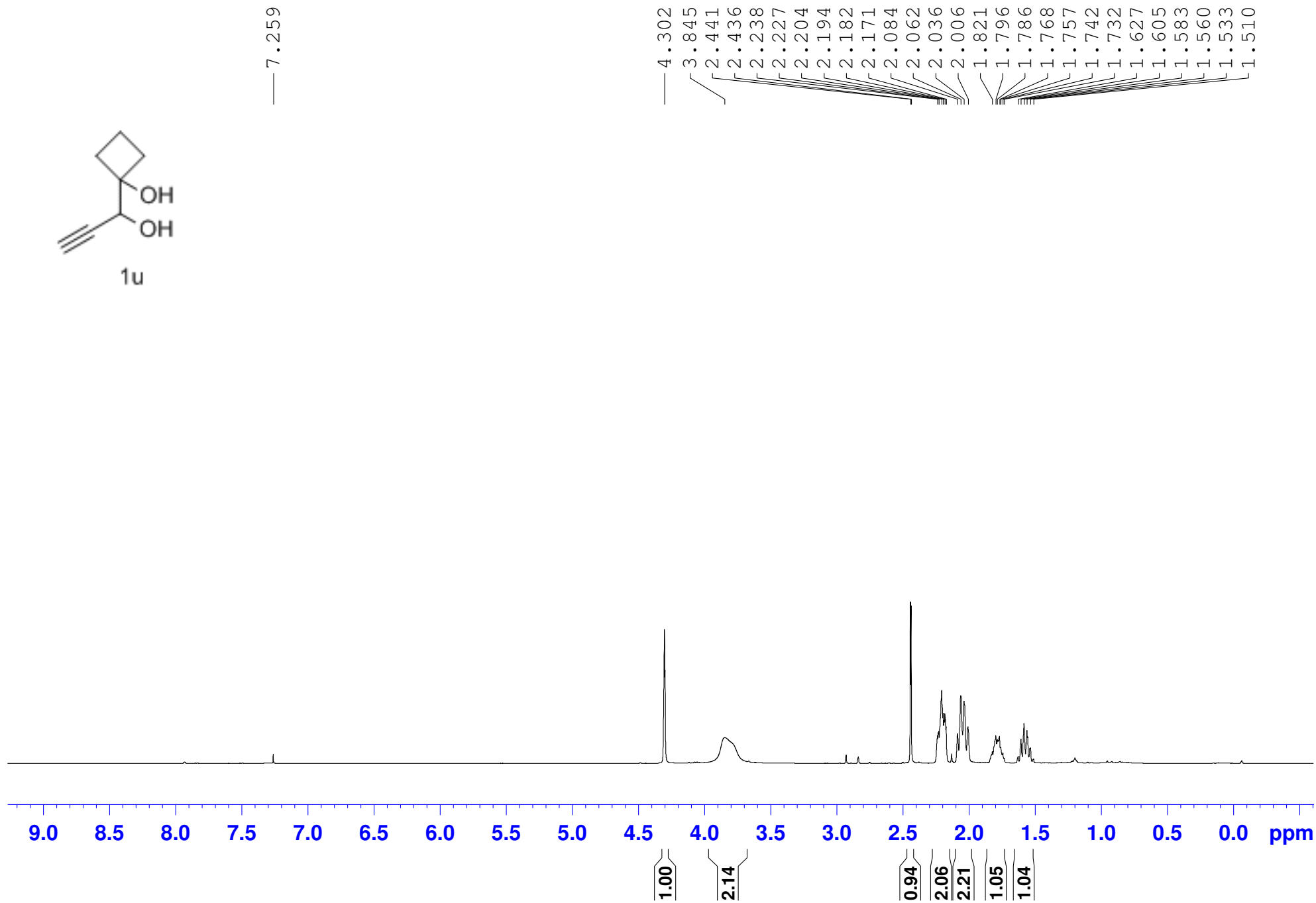
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3.362
3.348
3.314
3.300
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2.435
2.410
2.393
2.234
2.200
2.165
1.919
1.887
1.855
1.583
1.450

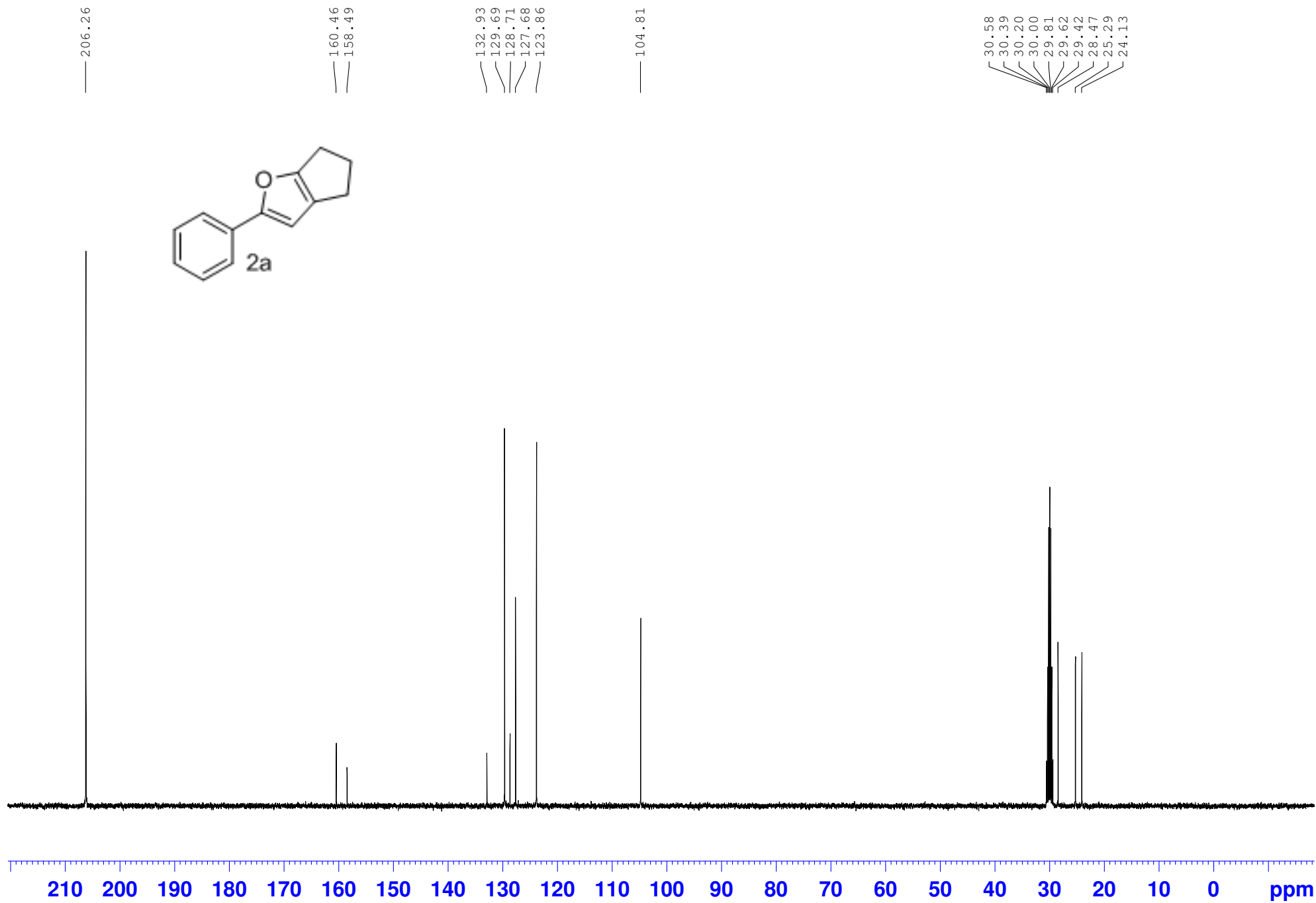
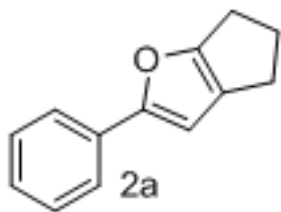


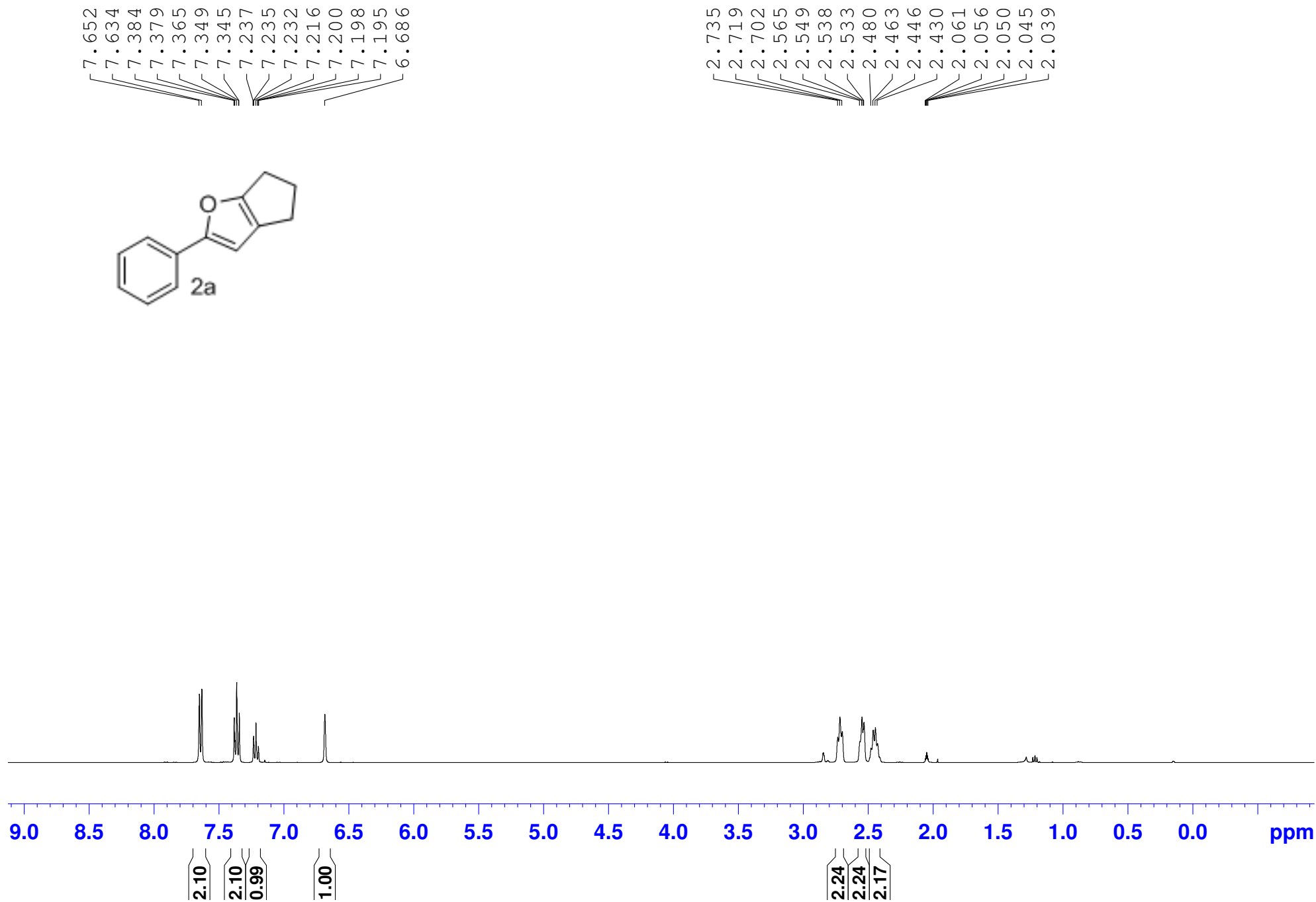
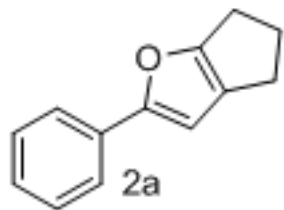


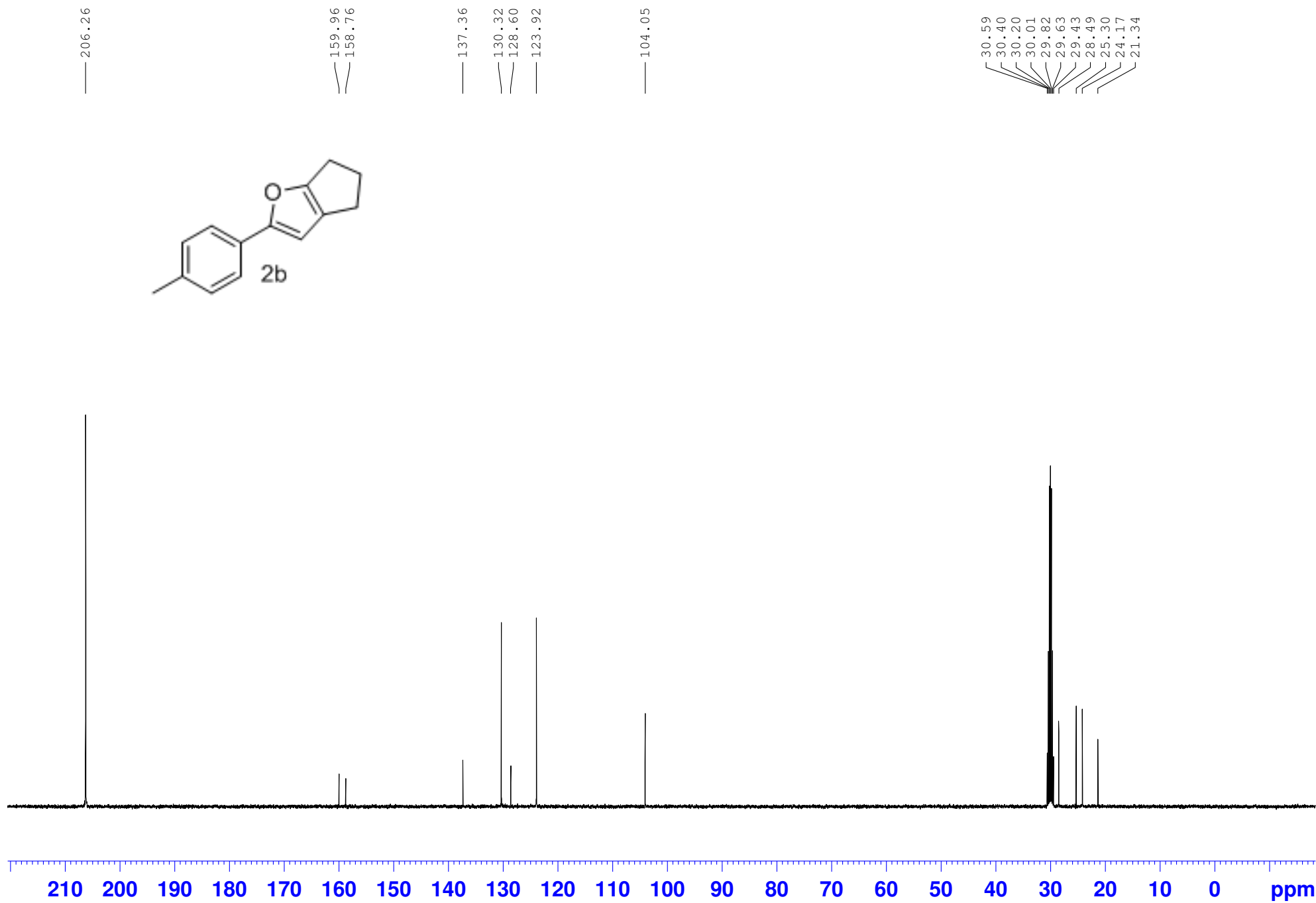
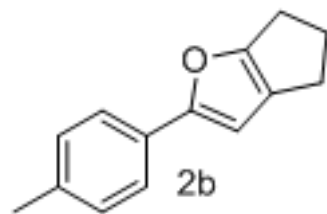


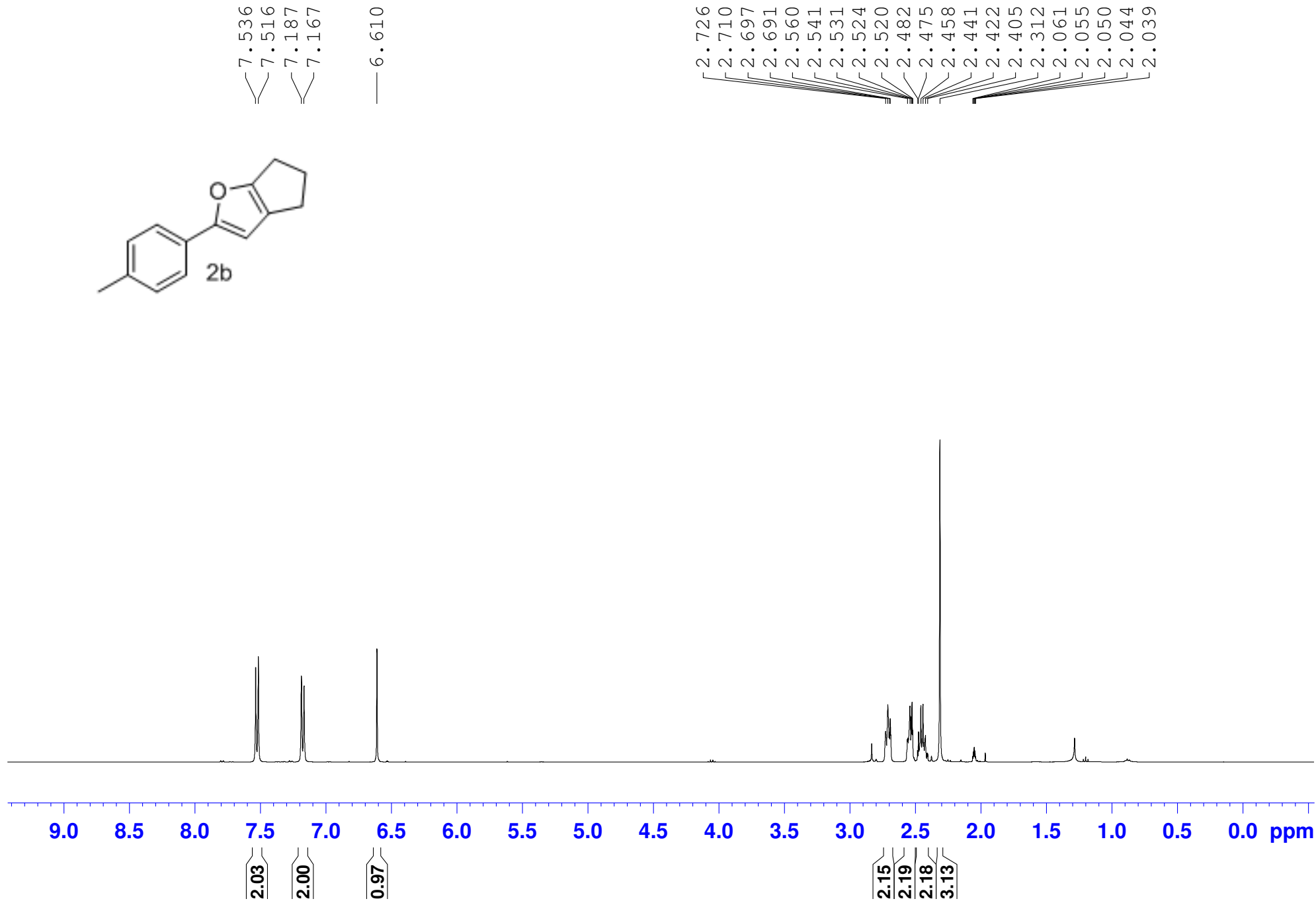
— 7.259

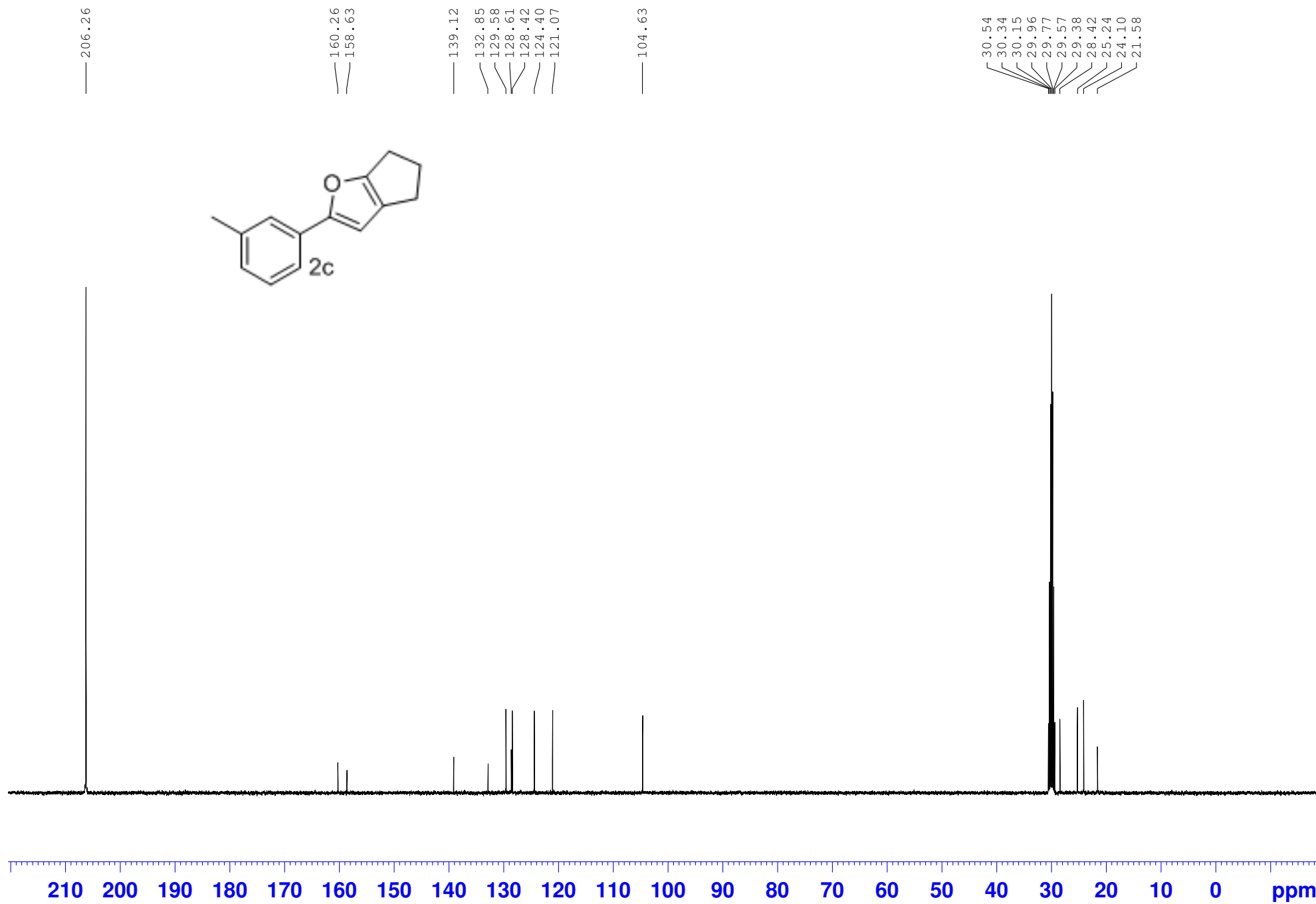
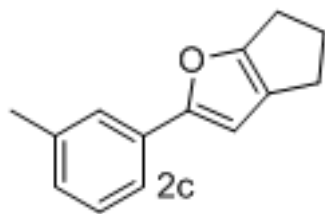






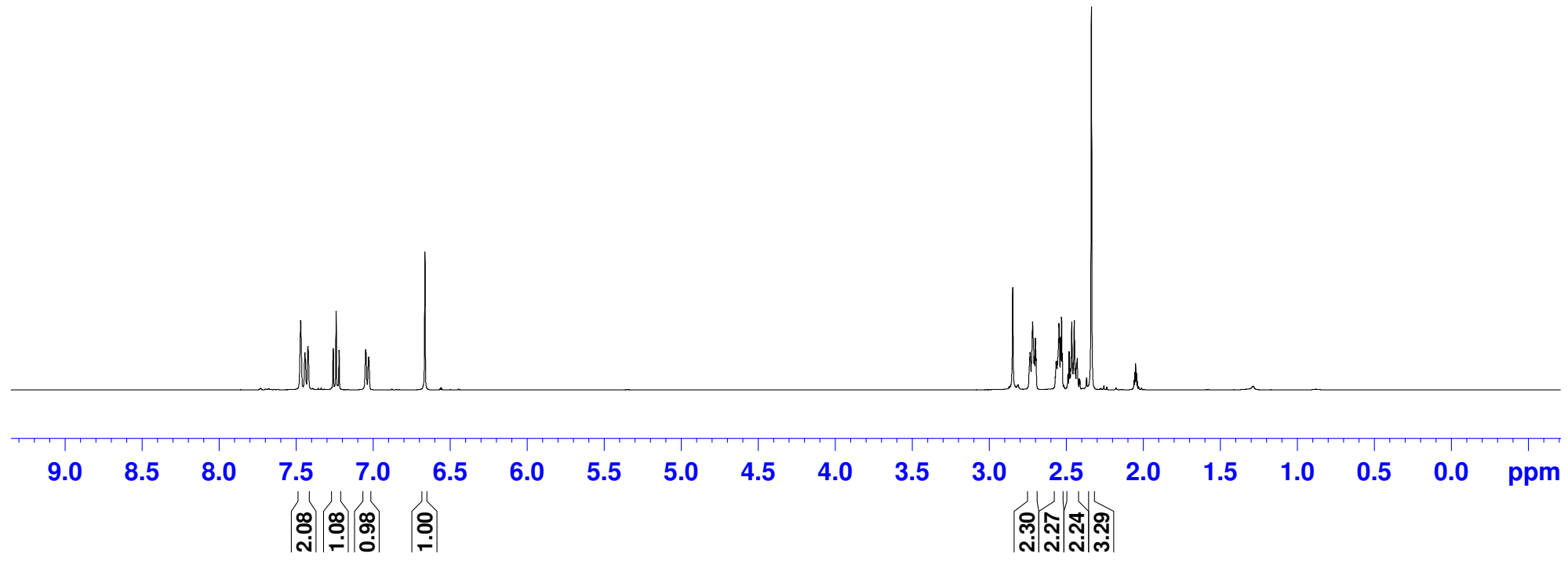
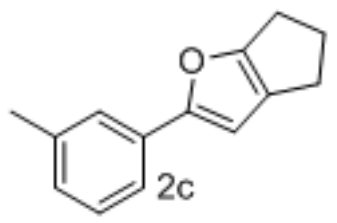


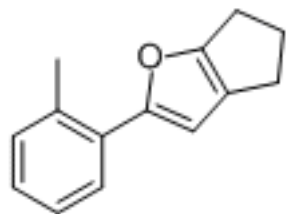




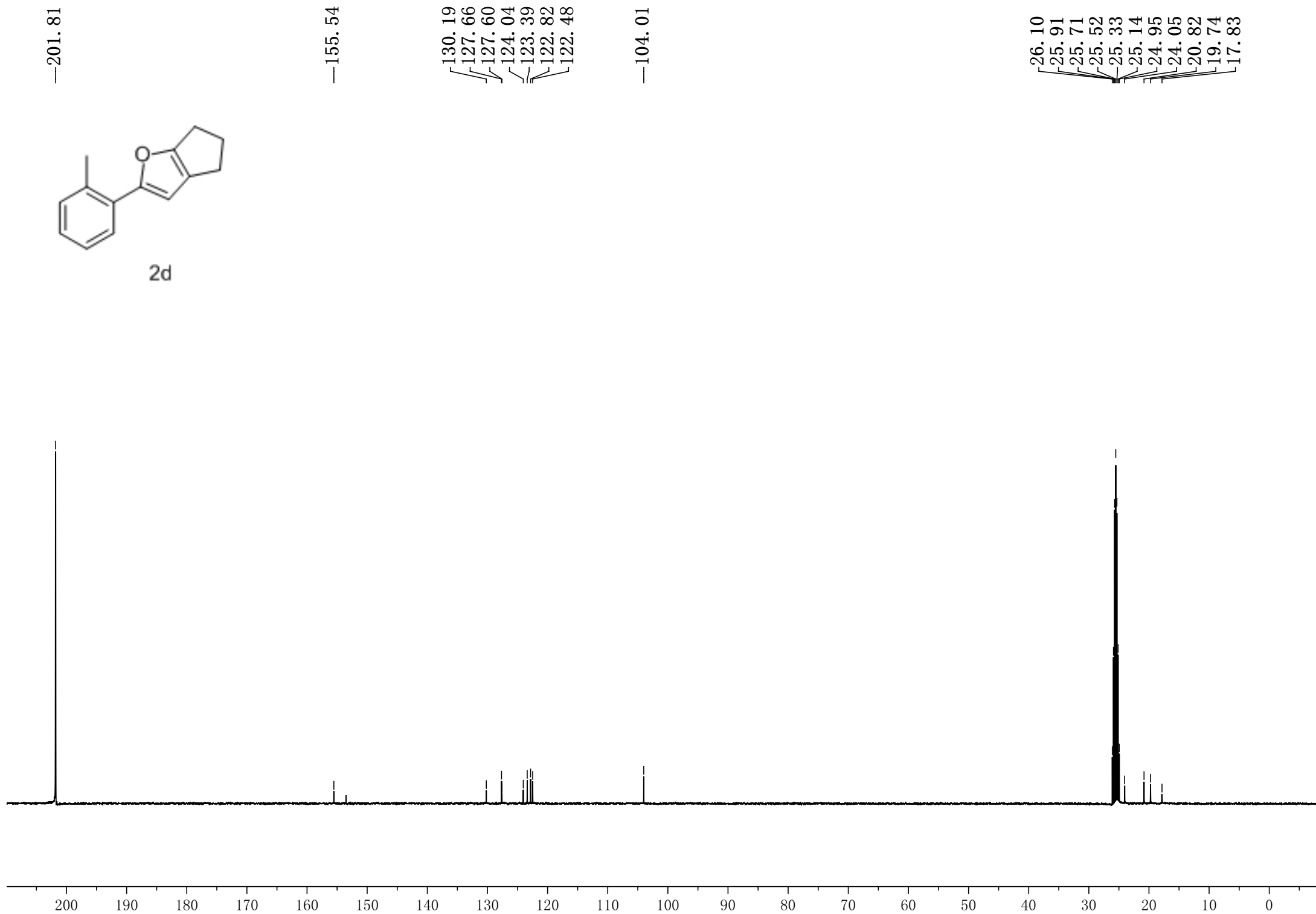
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2.700
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2.547
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2.531
2.526
2.489
2.482
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2.448
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2.411
2.337
2.061
2.055
2.050
2.044
2.039

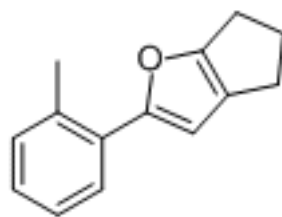
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7.424
7.261
7.241
7.222
7.049
7.030
6.665





2d





2d

7.65
7.64
7.63
7.23
7.23
7.18
7.18
6.51

2.75
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2.72
2.72
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2.56
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2.47
2.46

1.01

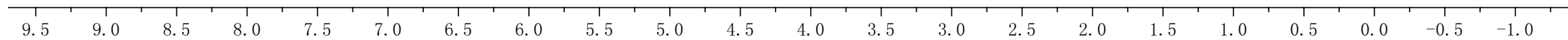
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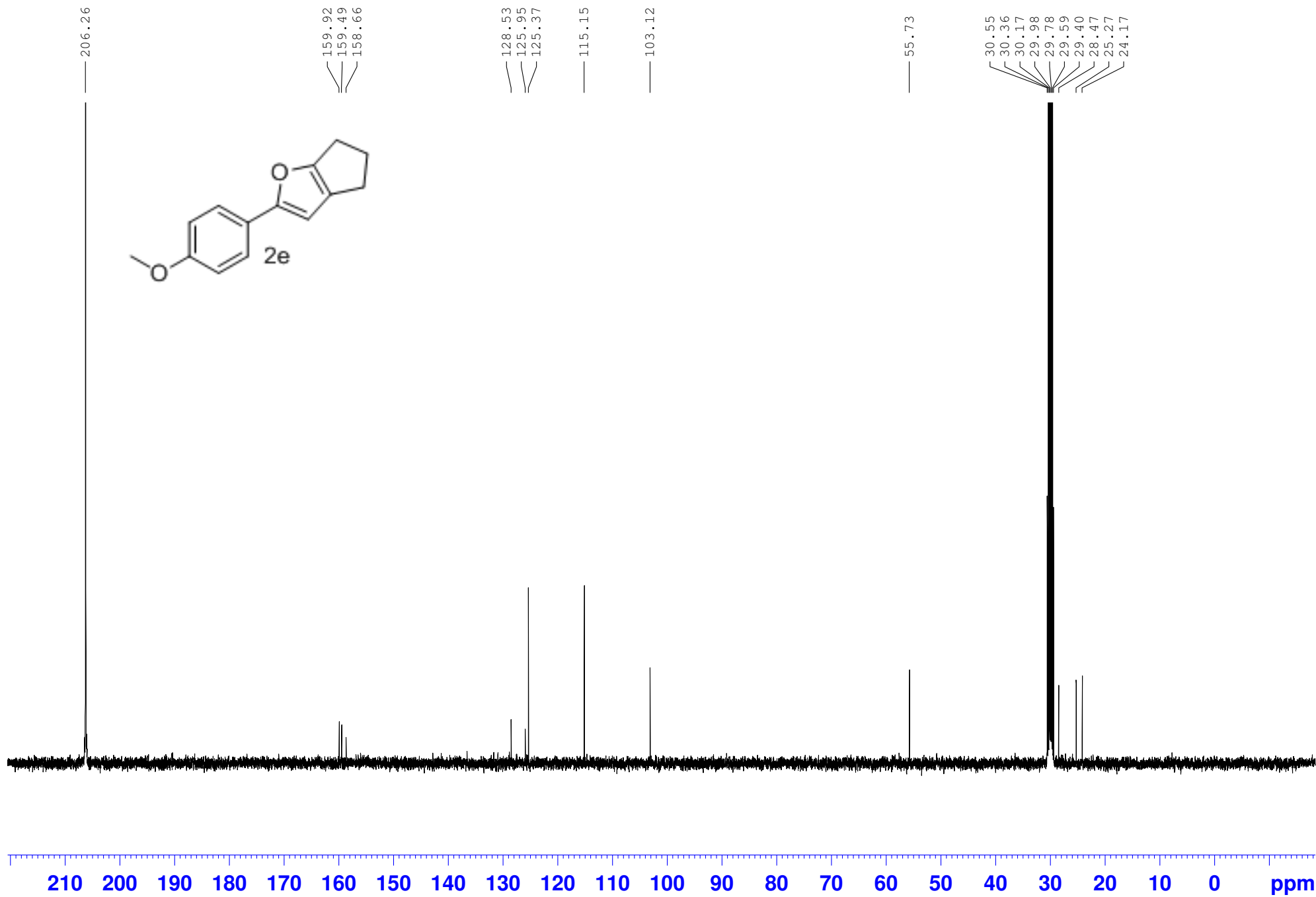
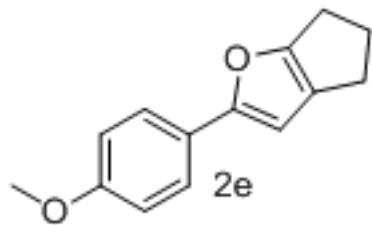
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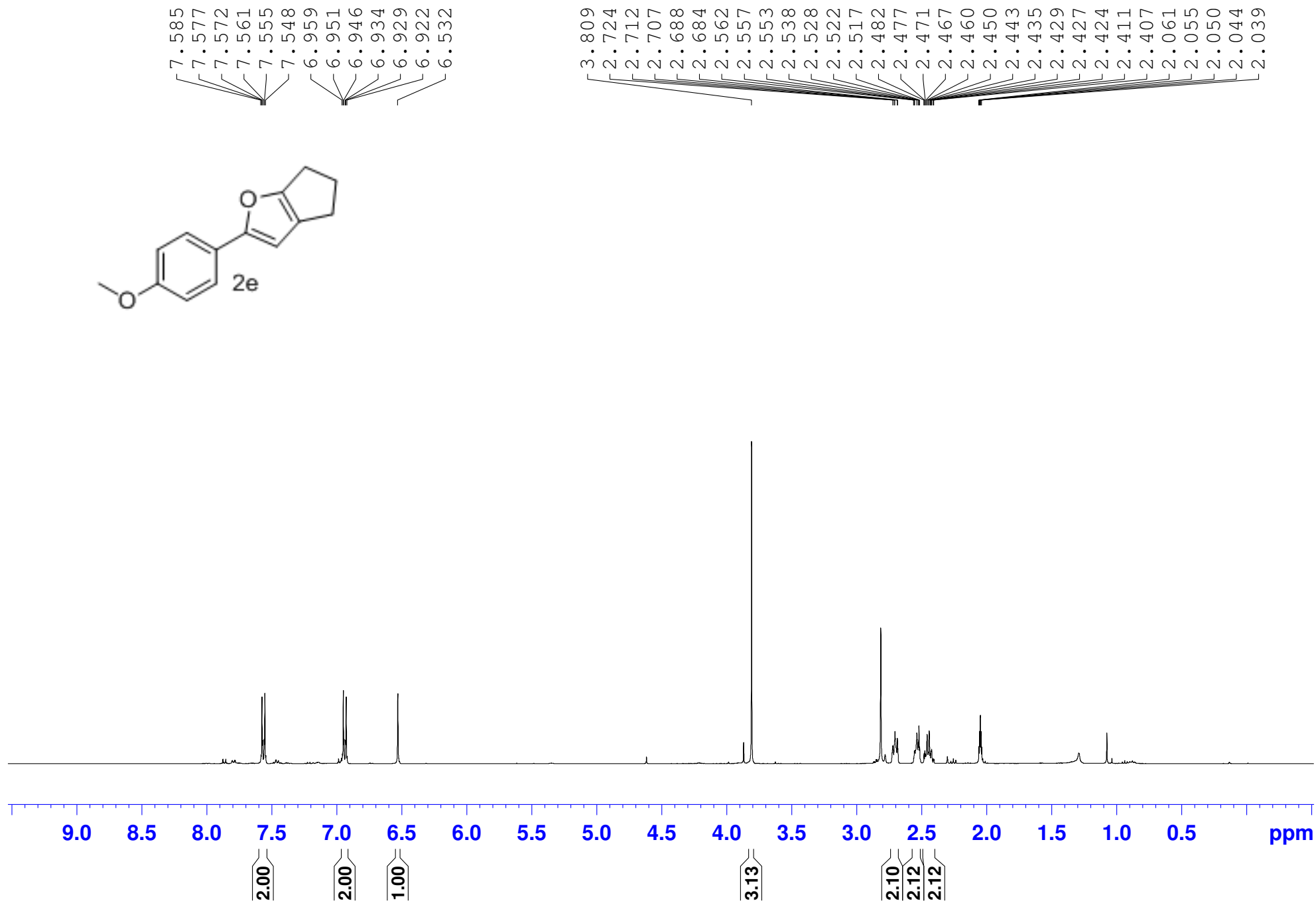
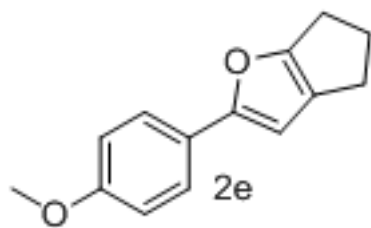
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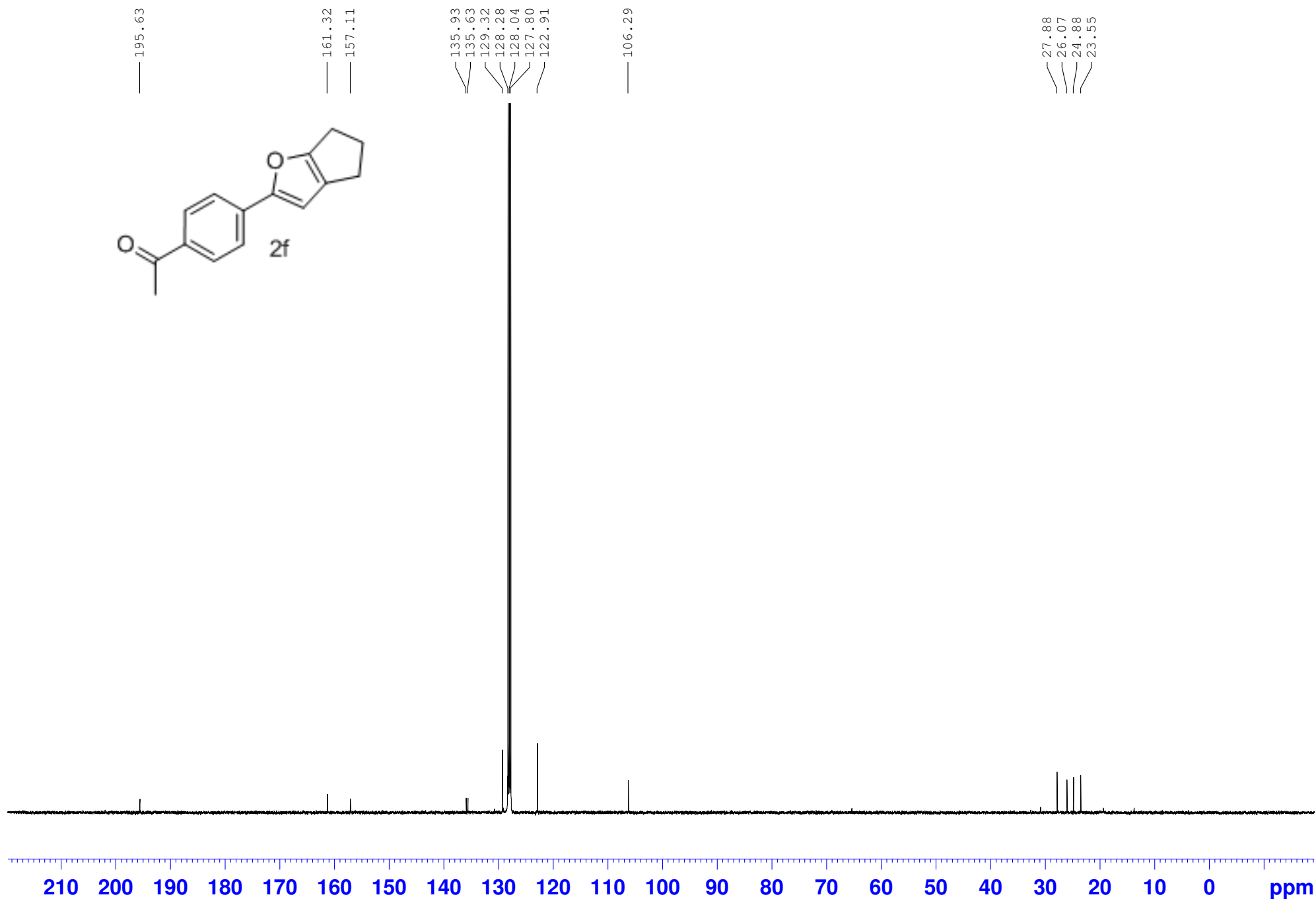
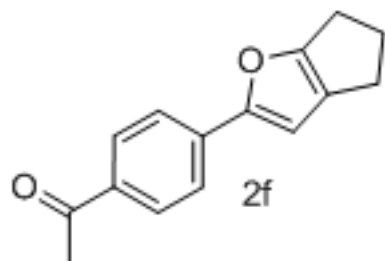
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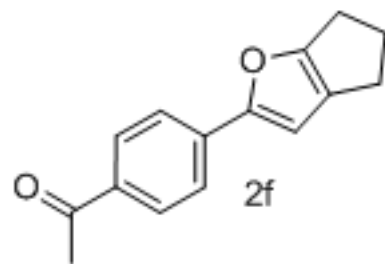
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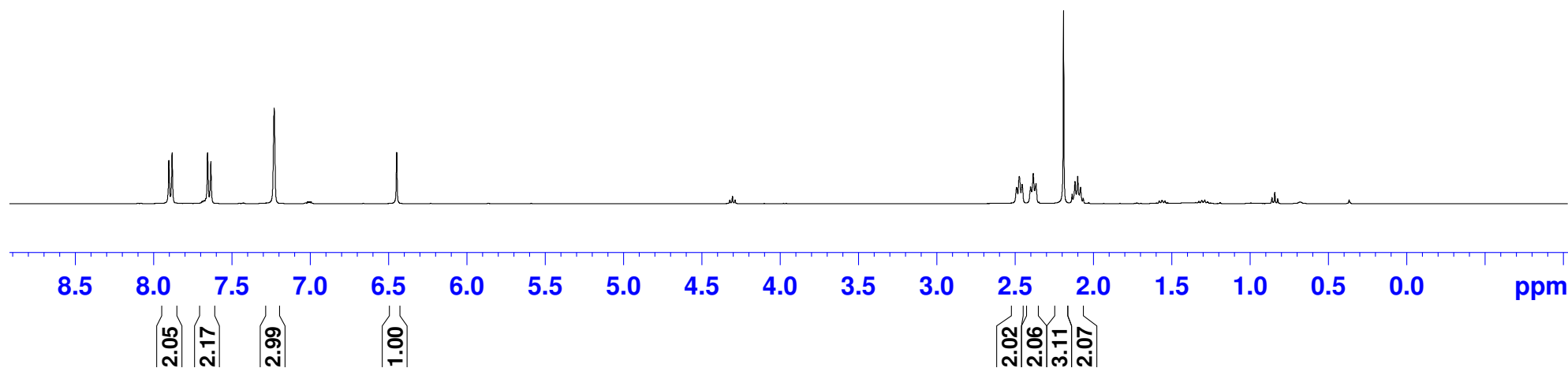


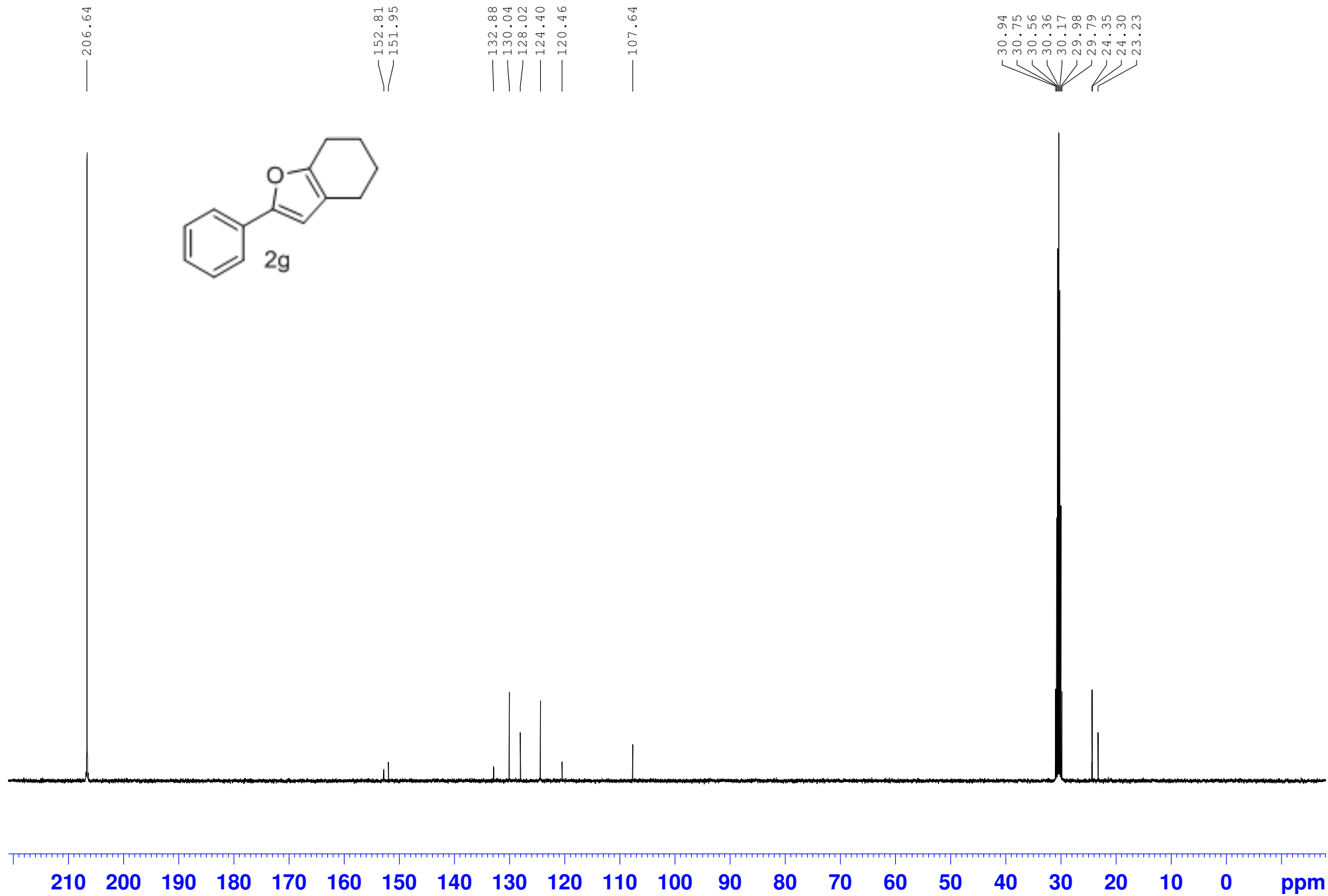


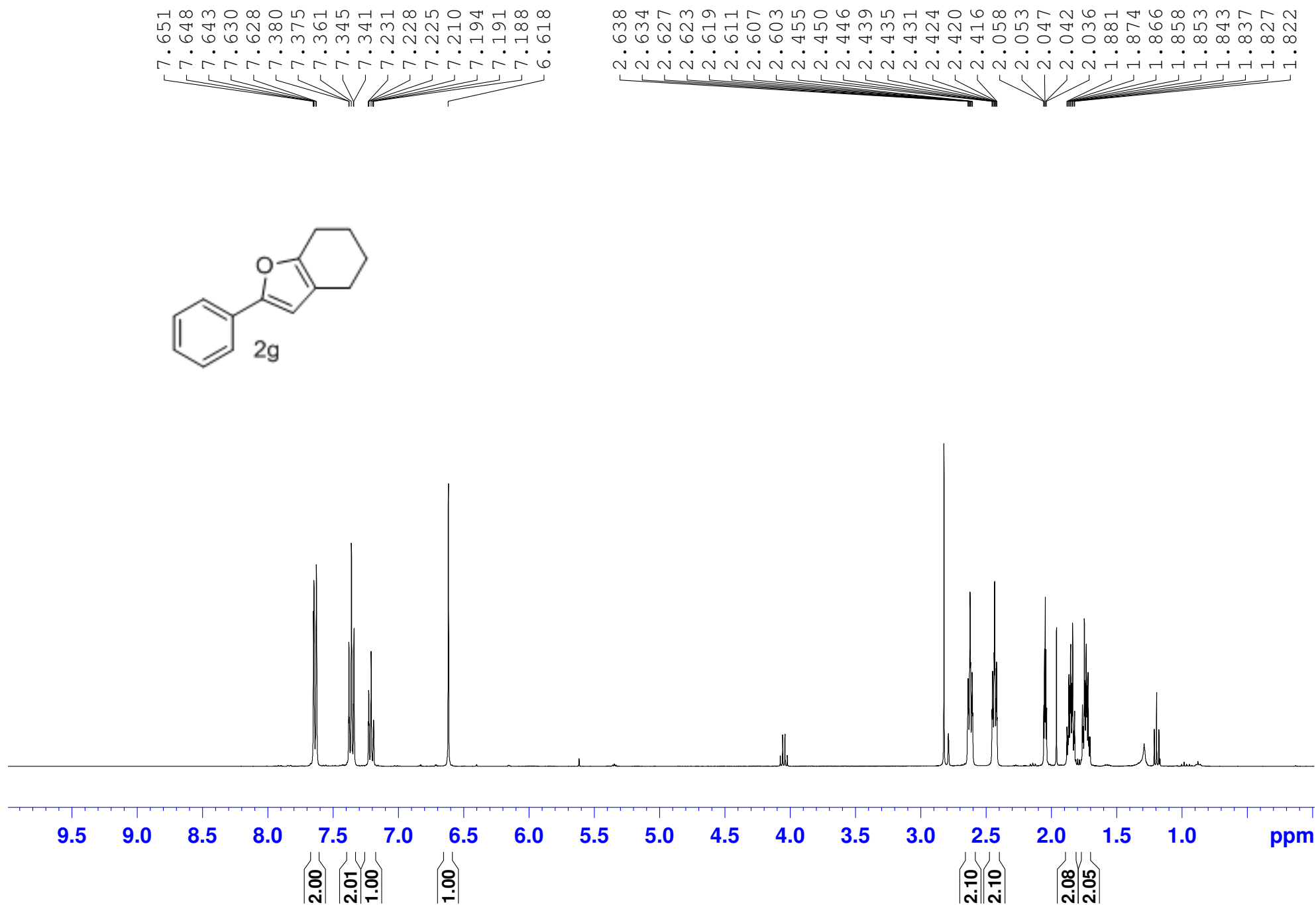
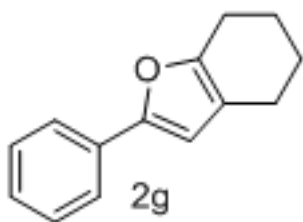


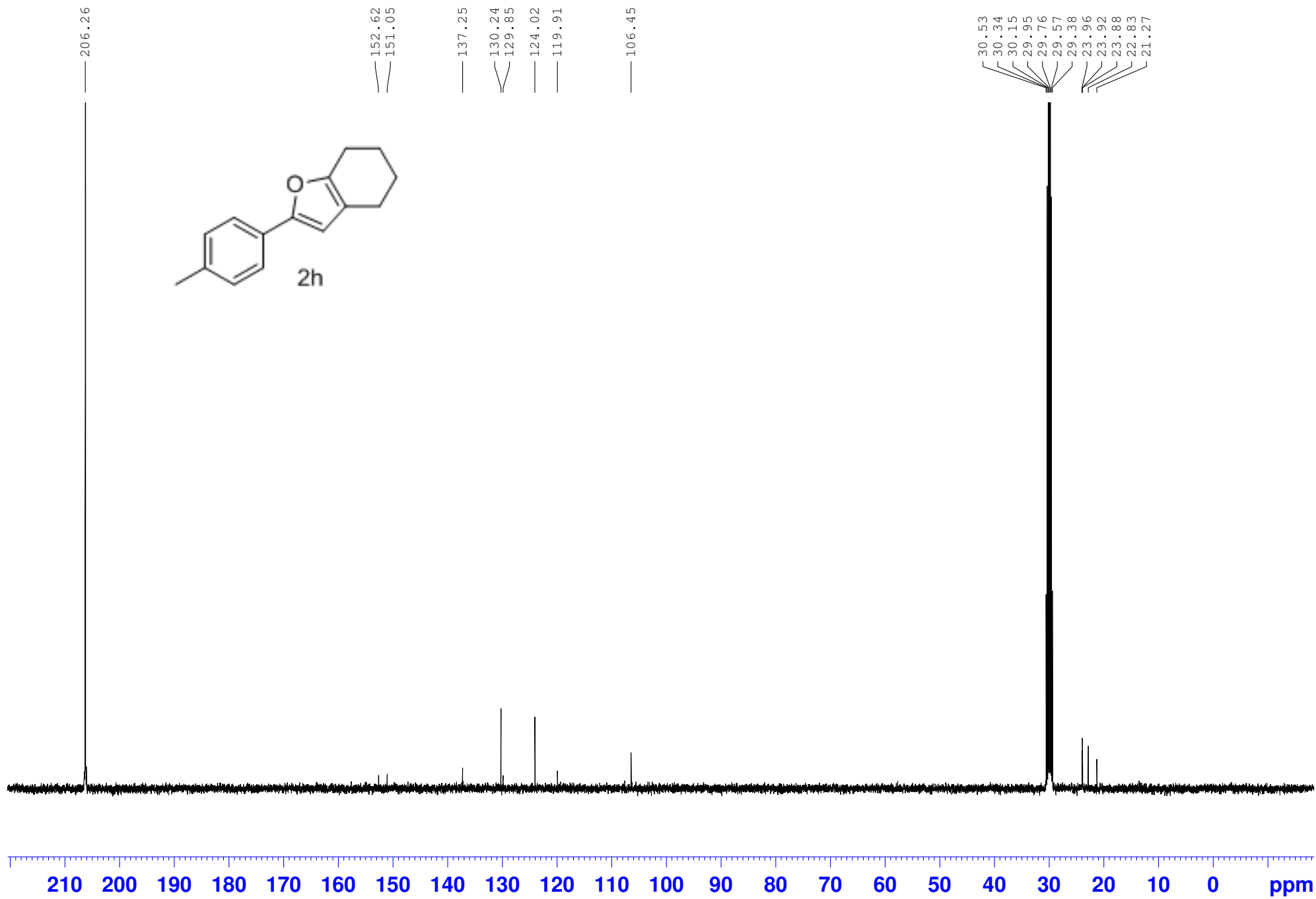
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7.232
6.449

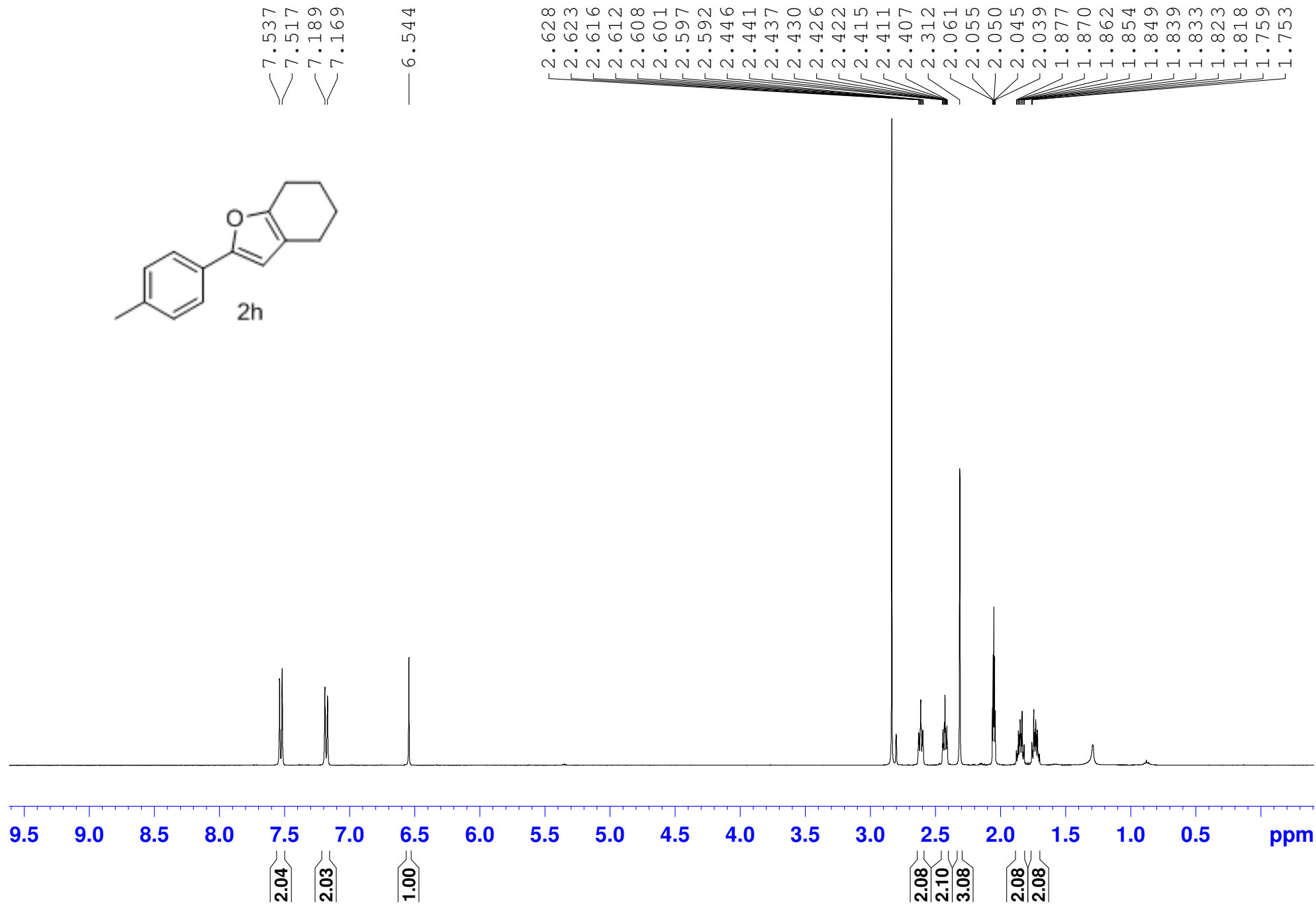
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2.136
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2.100
2.082
2.066

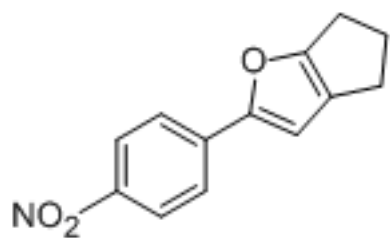












2i

—162.693

—155.310

—145.697

—137.404

—128.750

—124.364

—122.858

—107.747

77.317

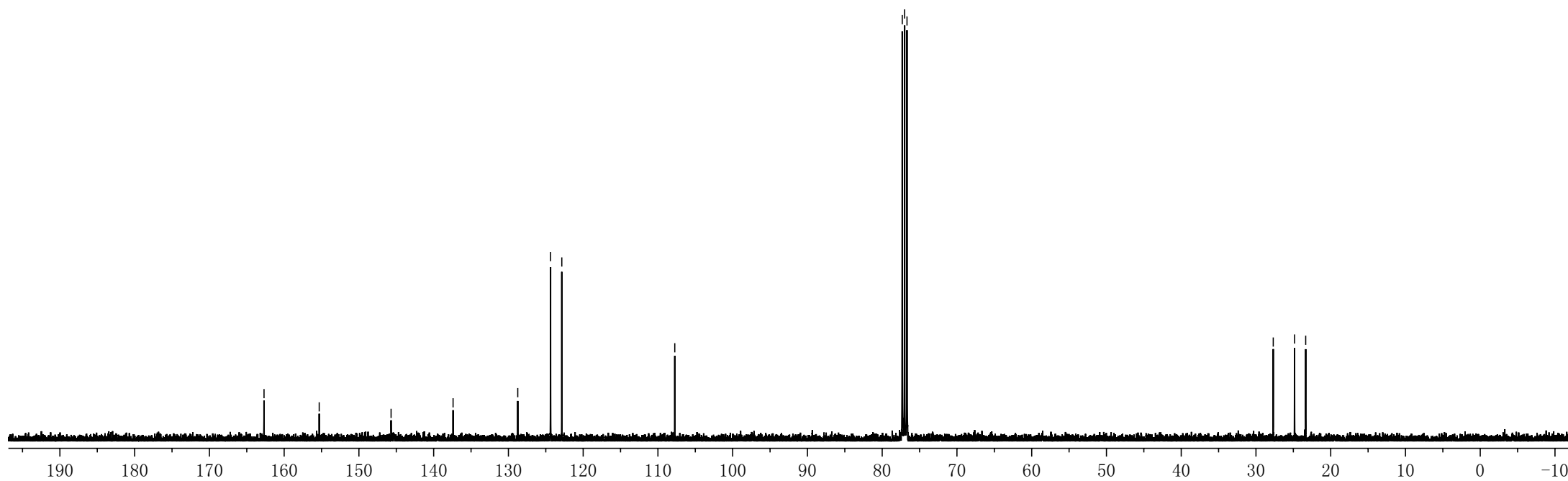
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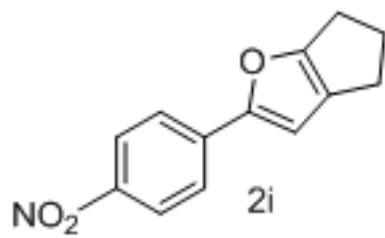
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27.688

24.839

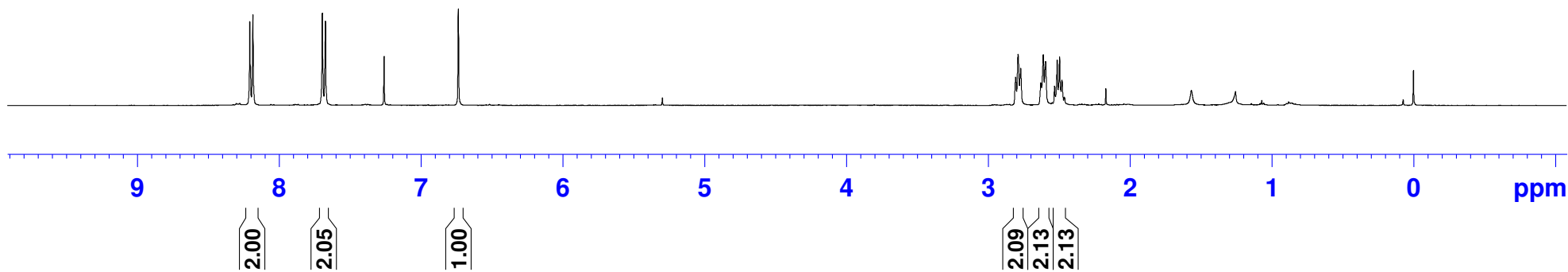
23.347

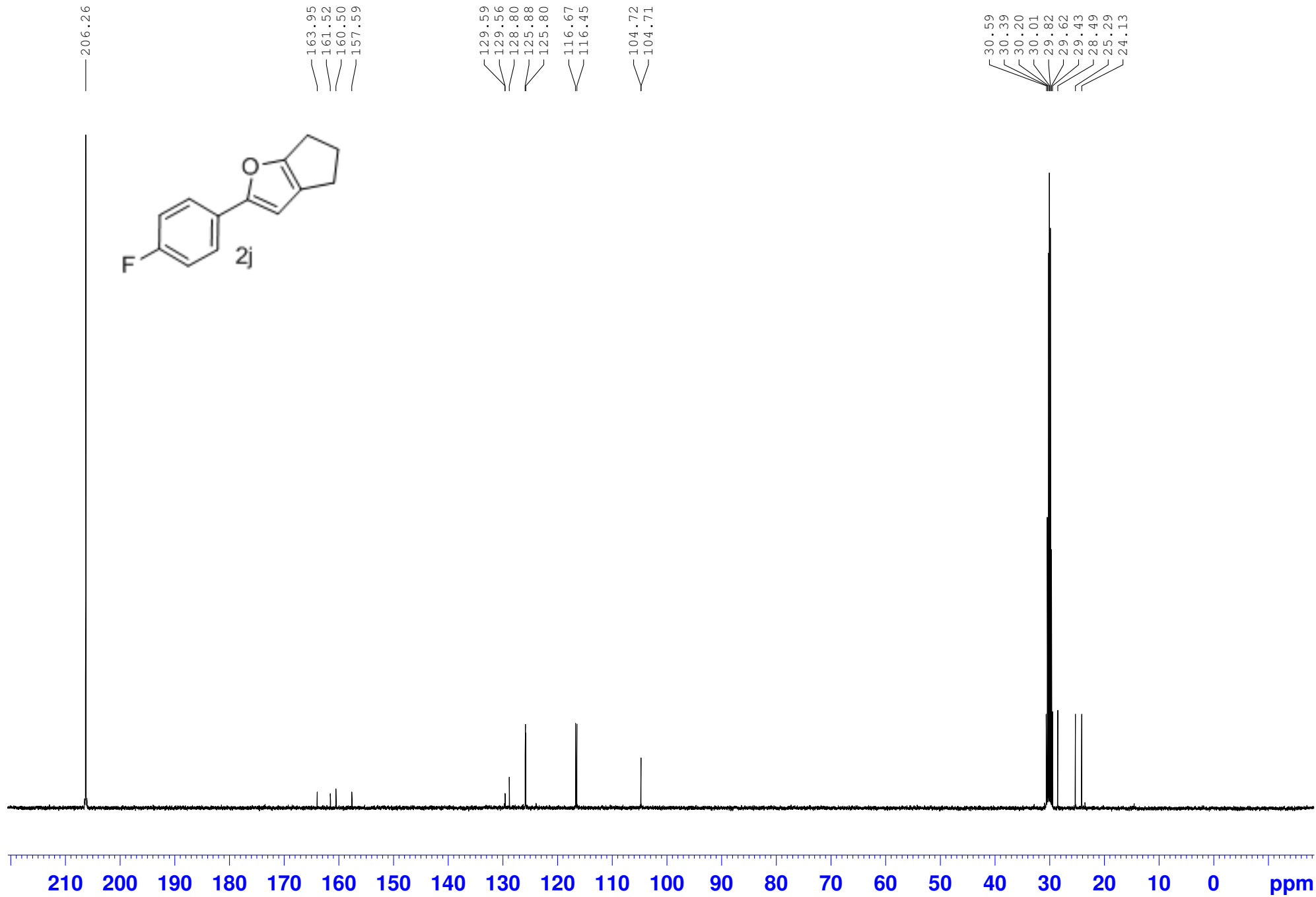




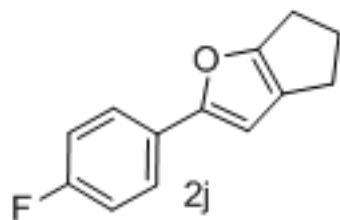
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7.696
7.674
7.260
6.736

2.805
2.789
2.770
2.628
2.611
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2.531
2.513
2.496
2.478
2.462

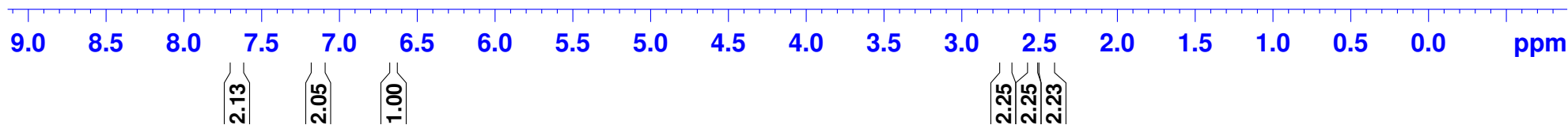


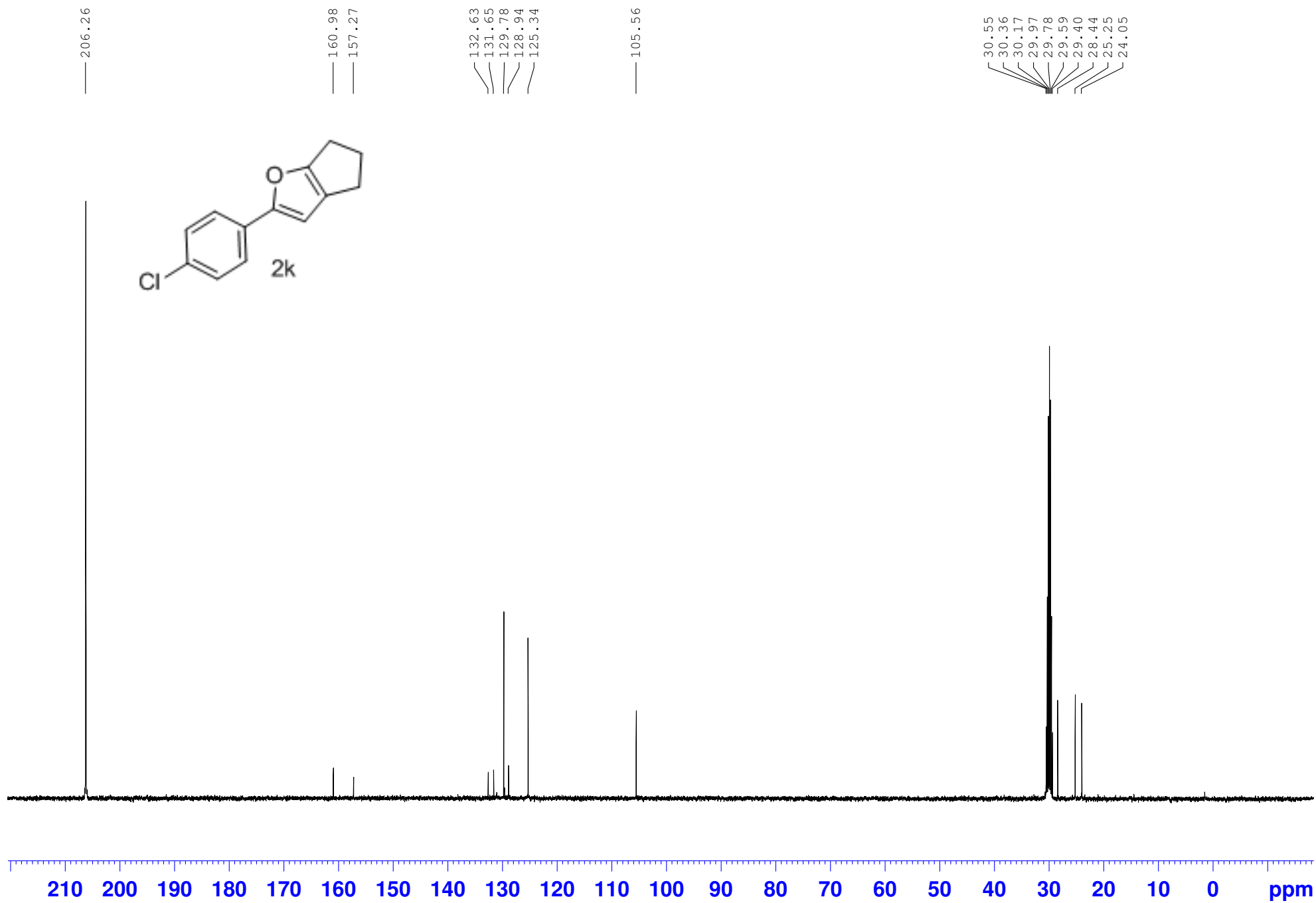
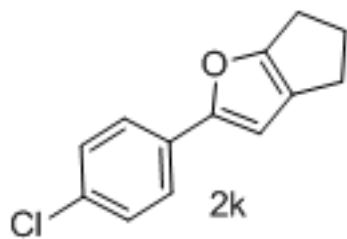


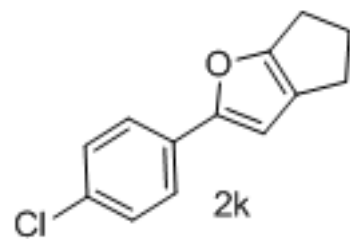
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7.674
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7.657
7.649
7.644
7.160
7.137
7.120
7.115
6.654



2.730
2.714
2.695
2.563
2.560
2.545
2.534
2.528
2.487
2.480
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2.463
2.453
2.447
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2.045
2.039

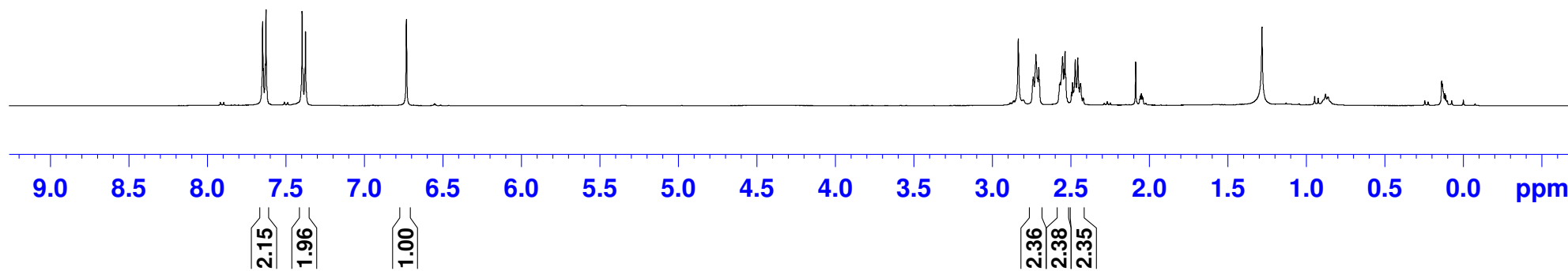


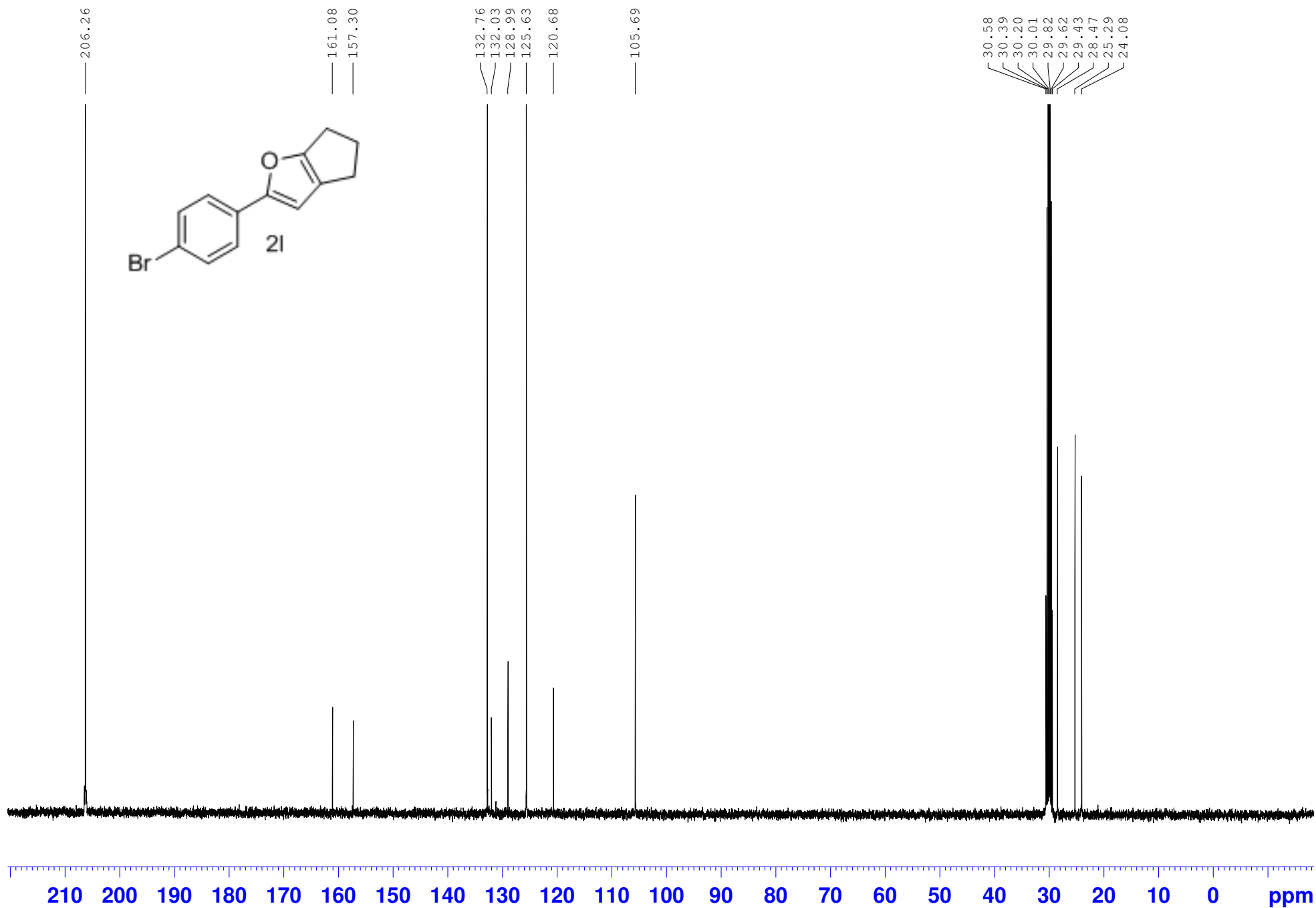
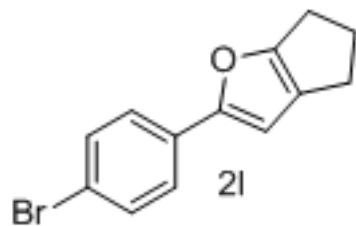


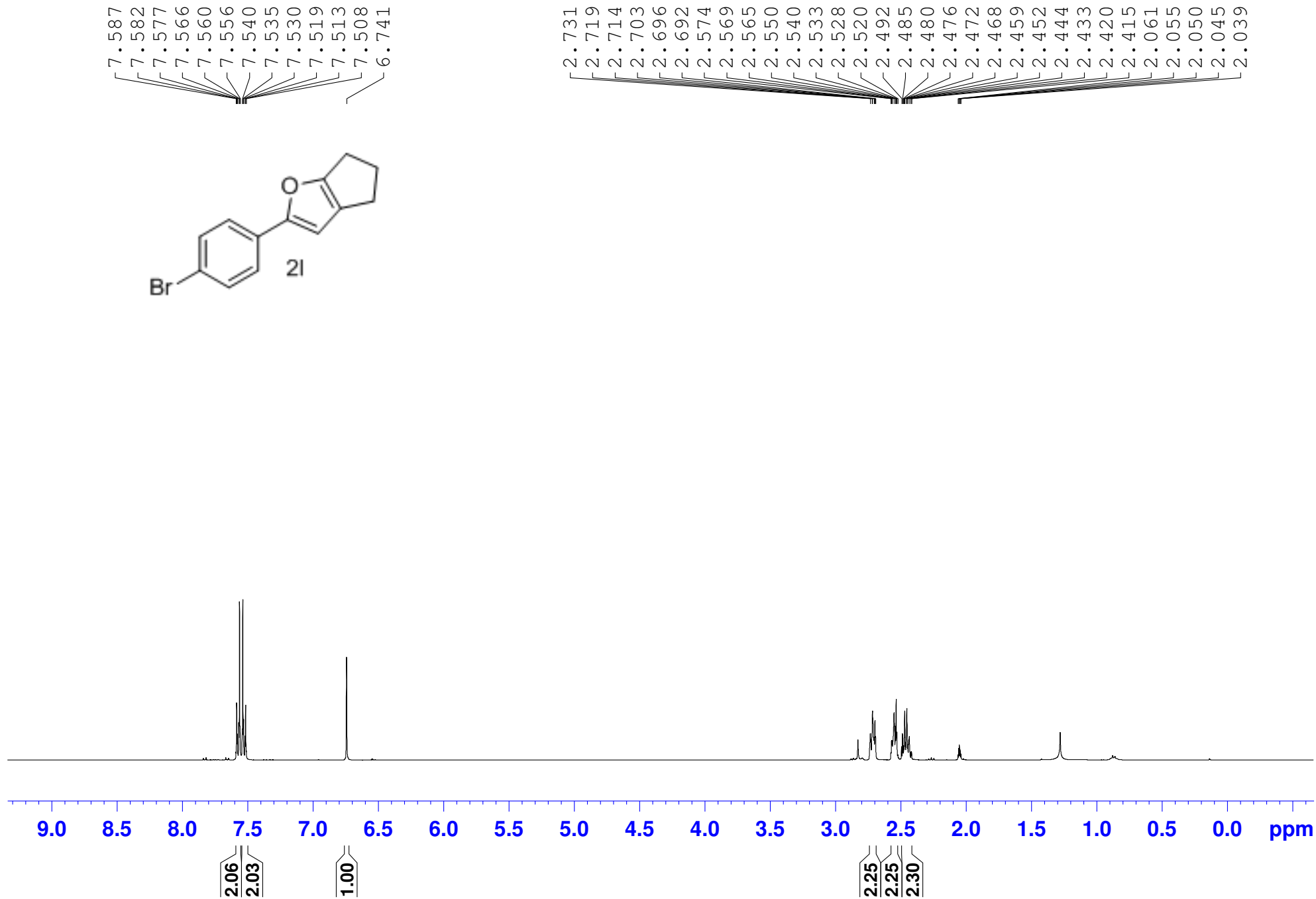


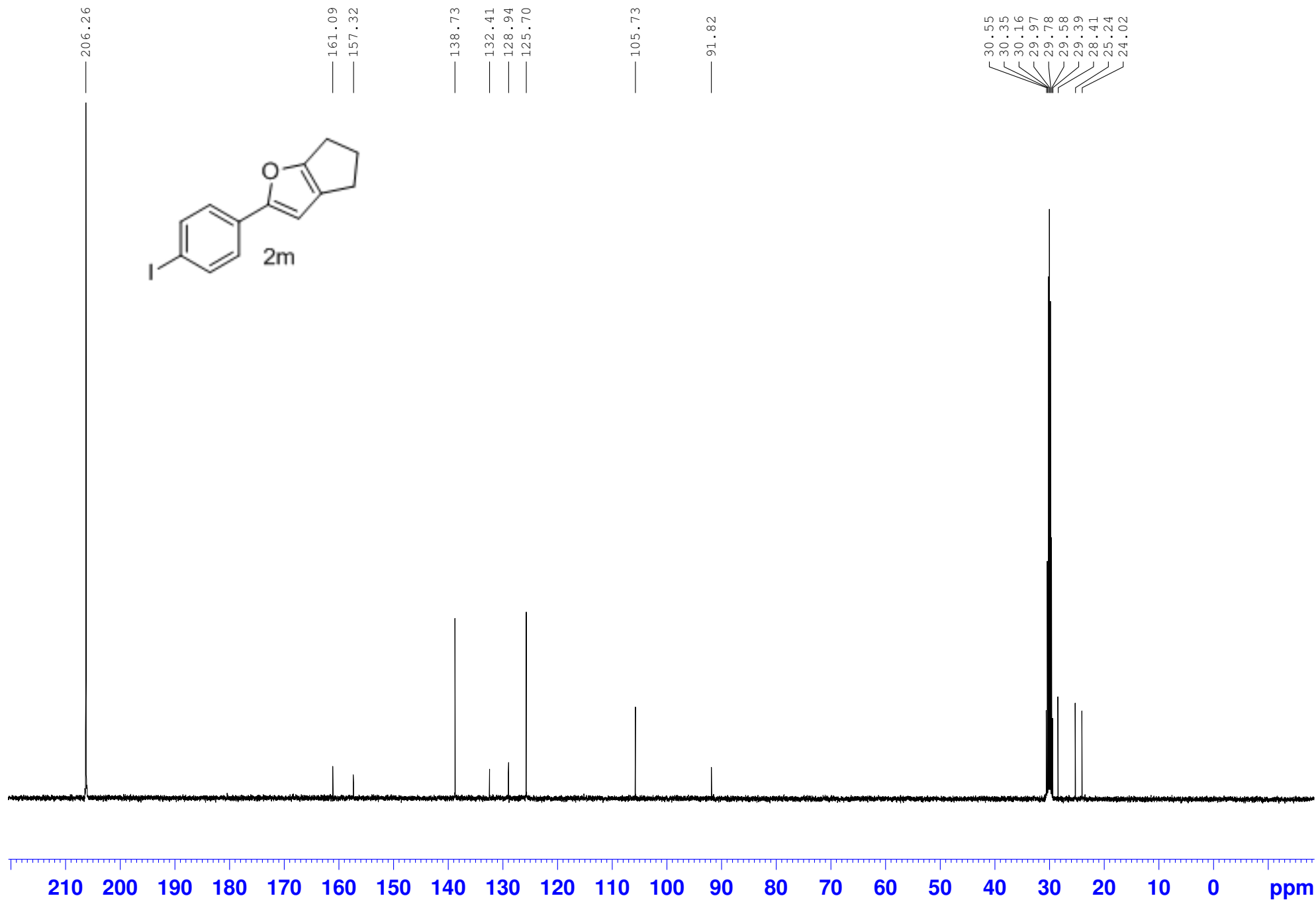
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7.641
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7.624
7.394
7.389
7.377
7.372
— 6.732

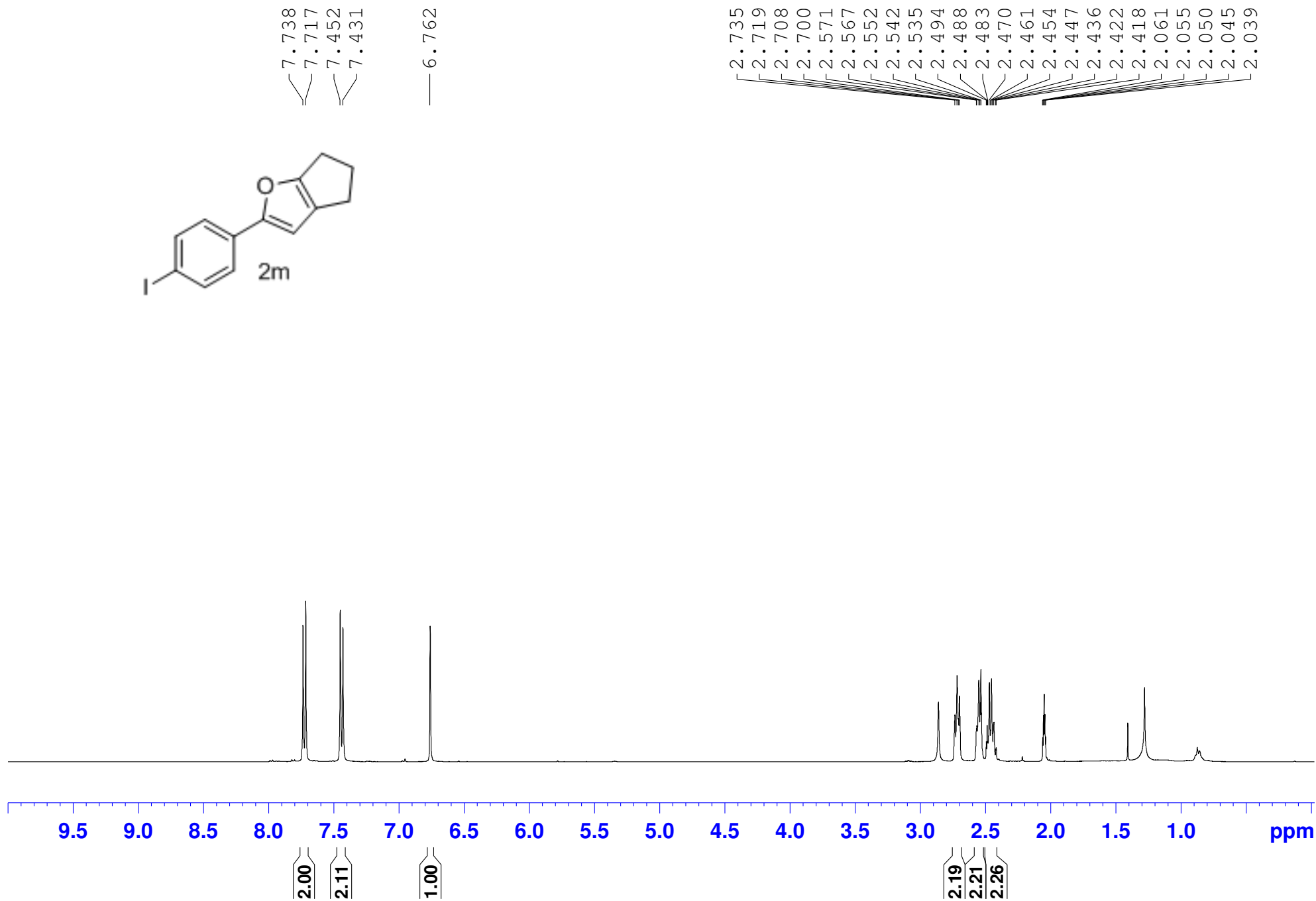
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2.704
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2.552
2.542
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2.436
2.422
2.061
2.055
2.050
2.045
2.039

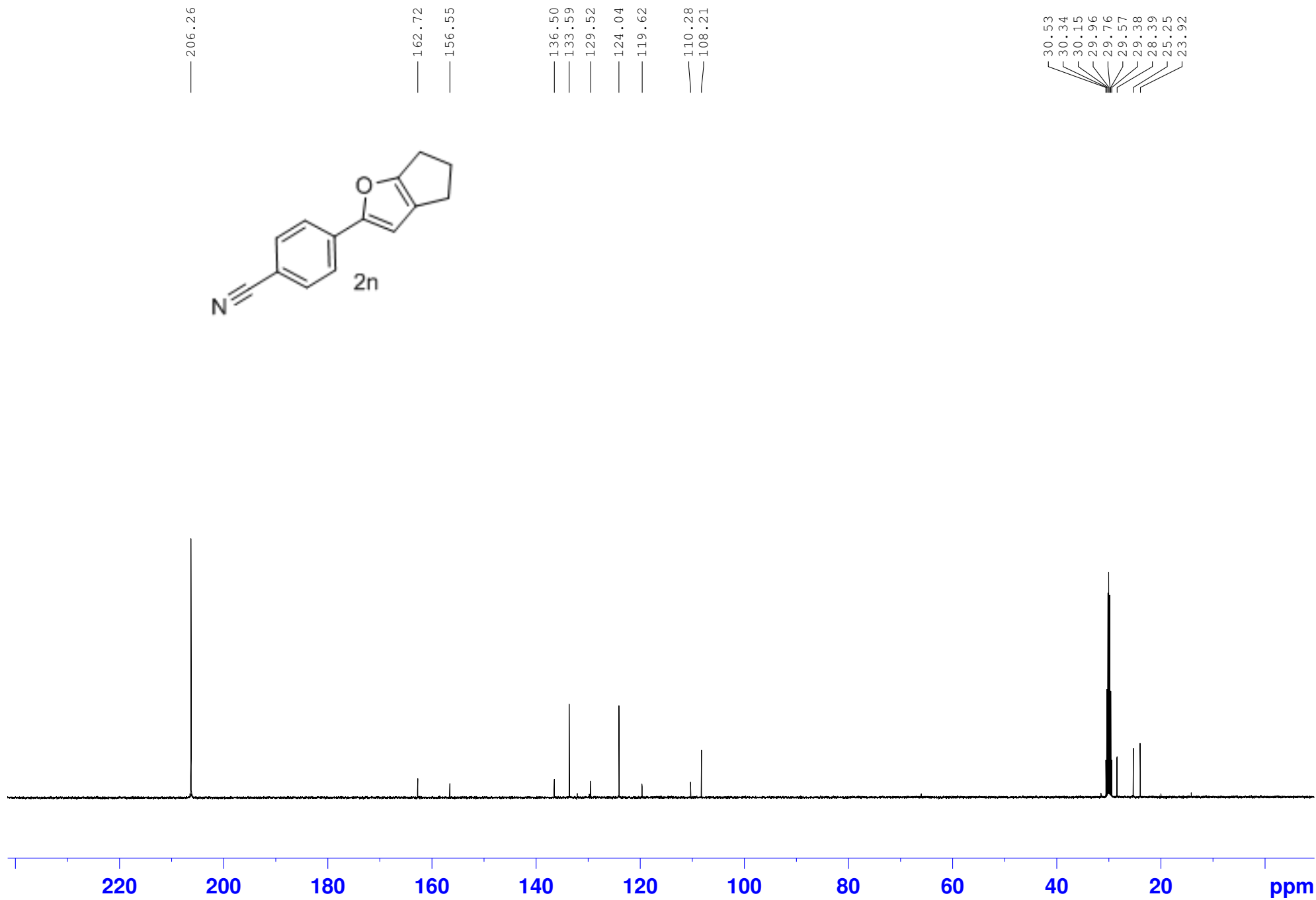
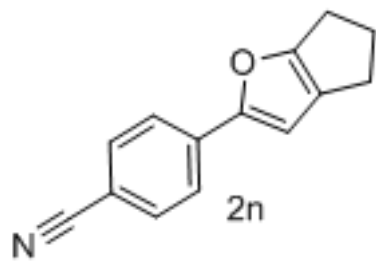




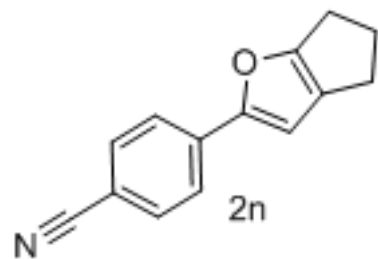




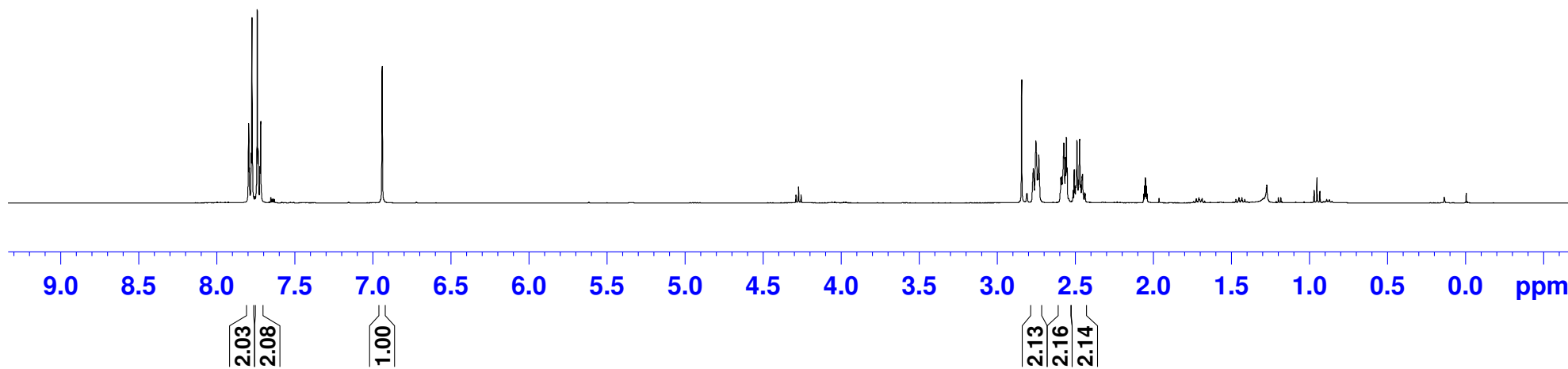


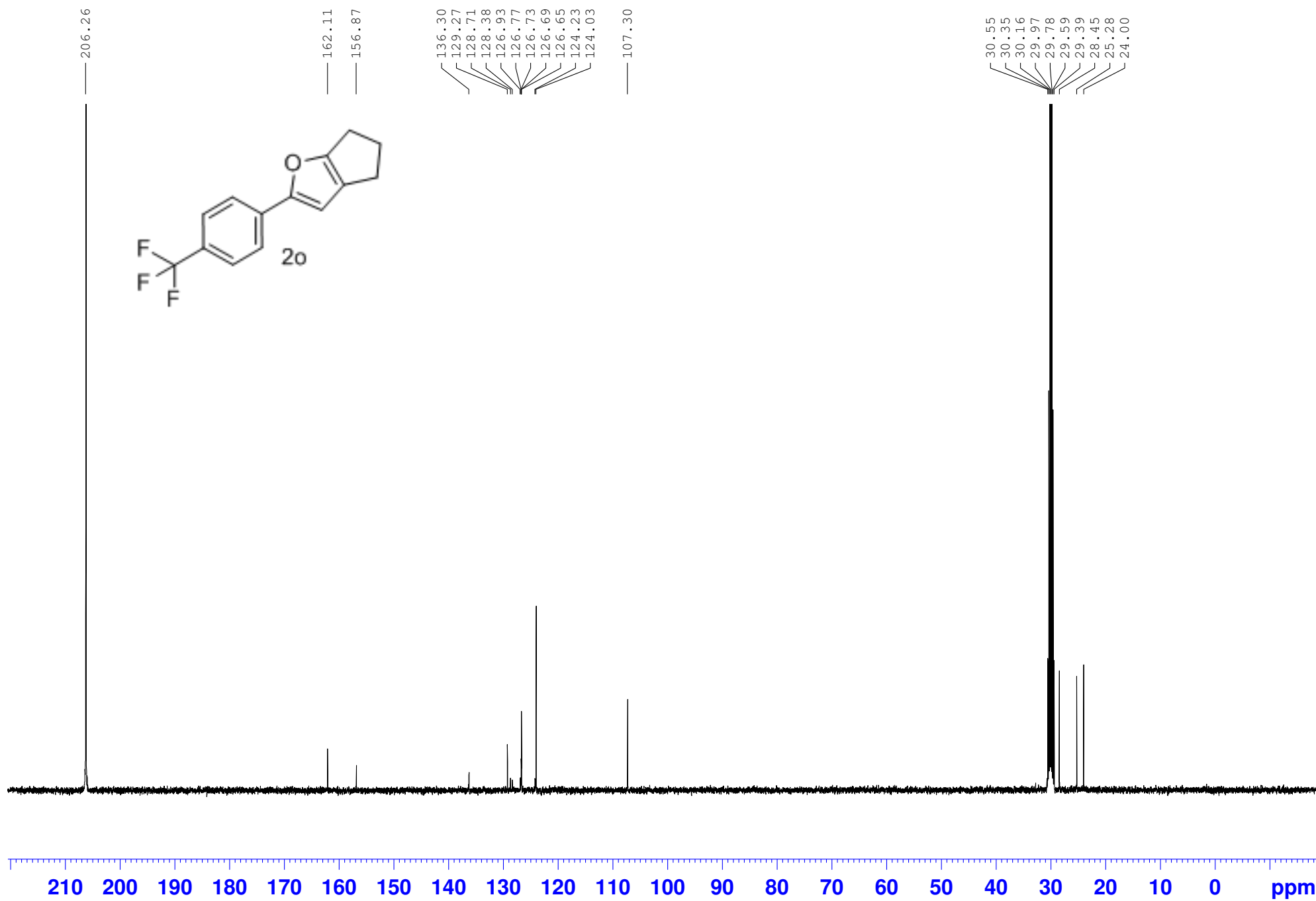


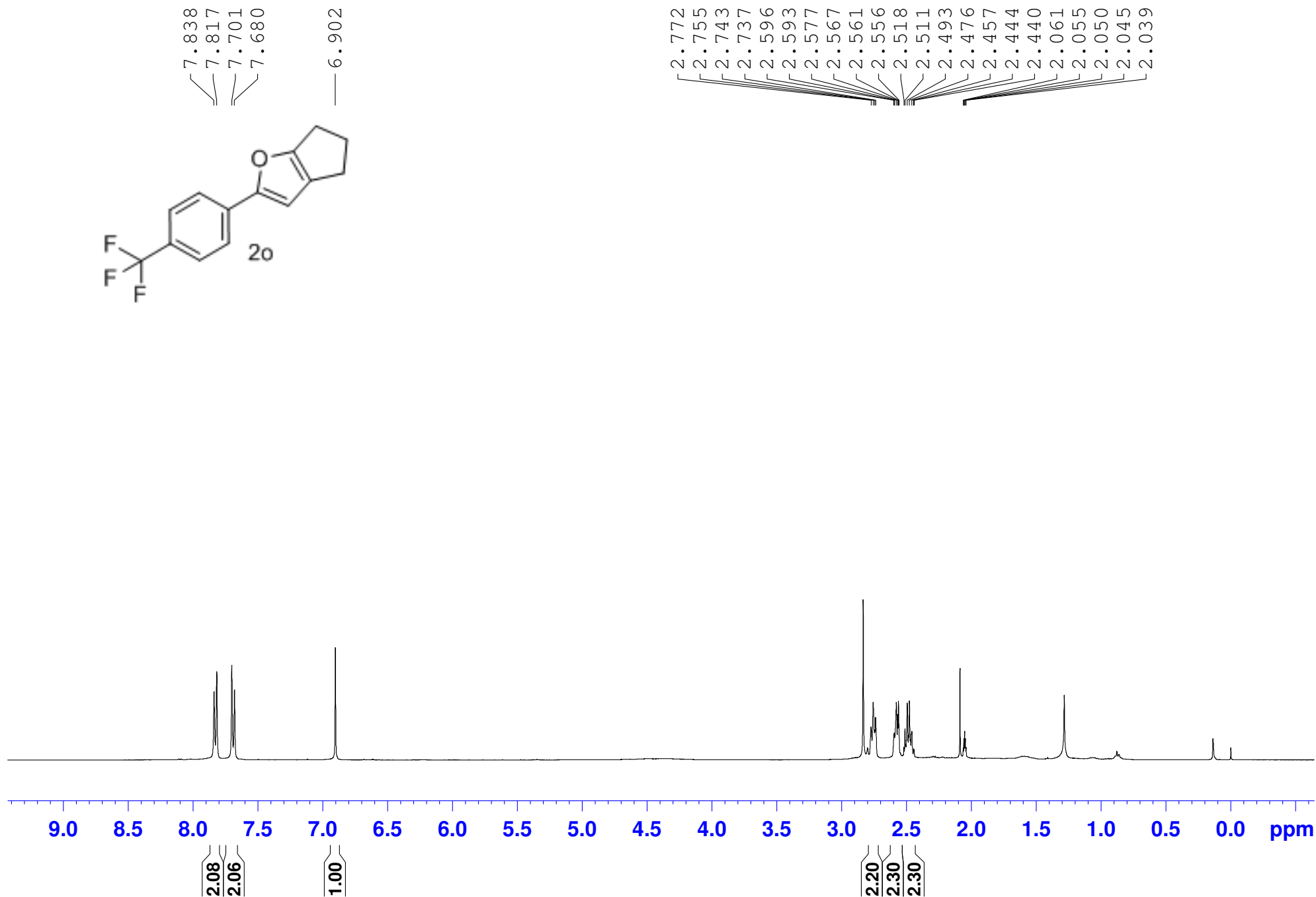
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7.772
7.738
7.733
7.721
7.716
— 6.941

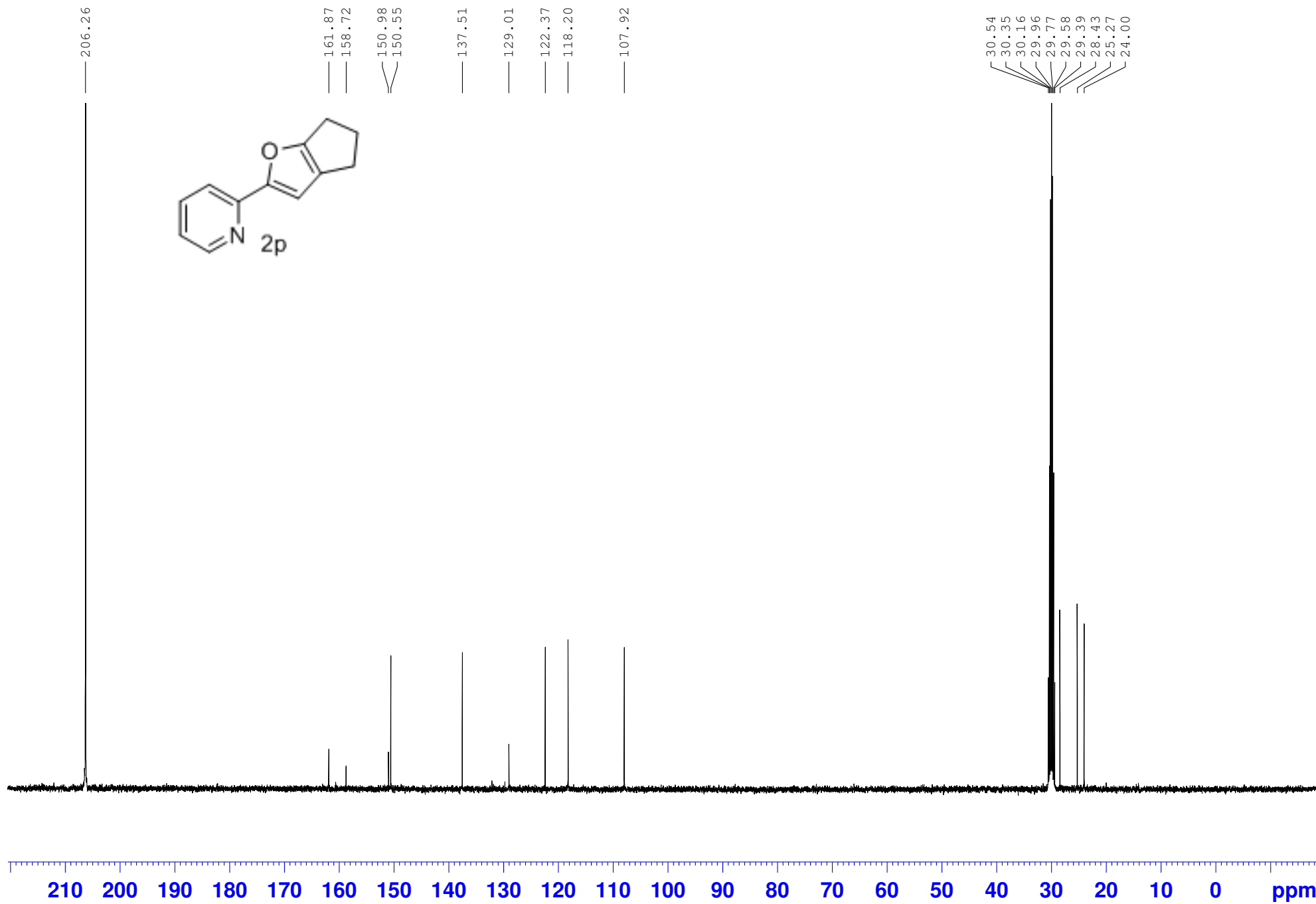


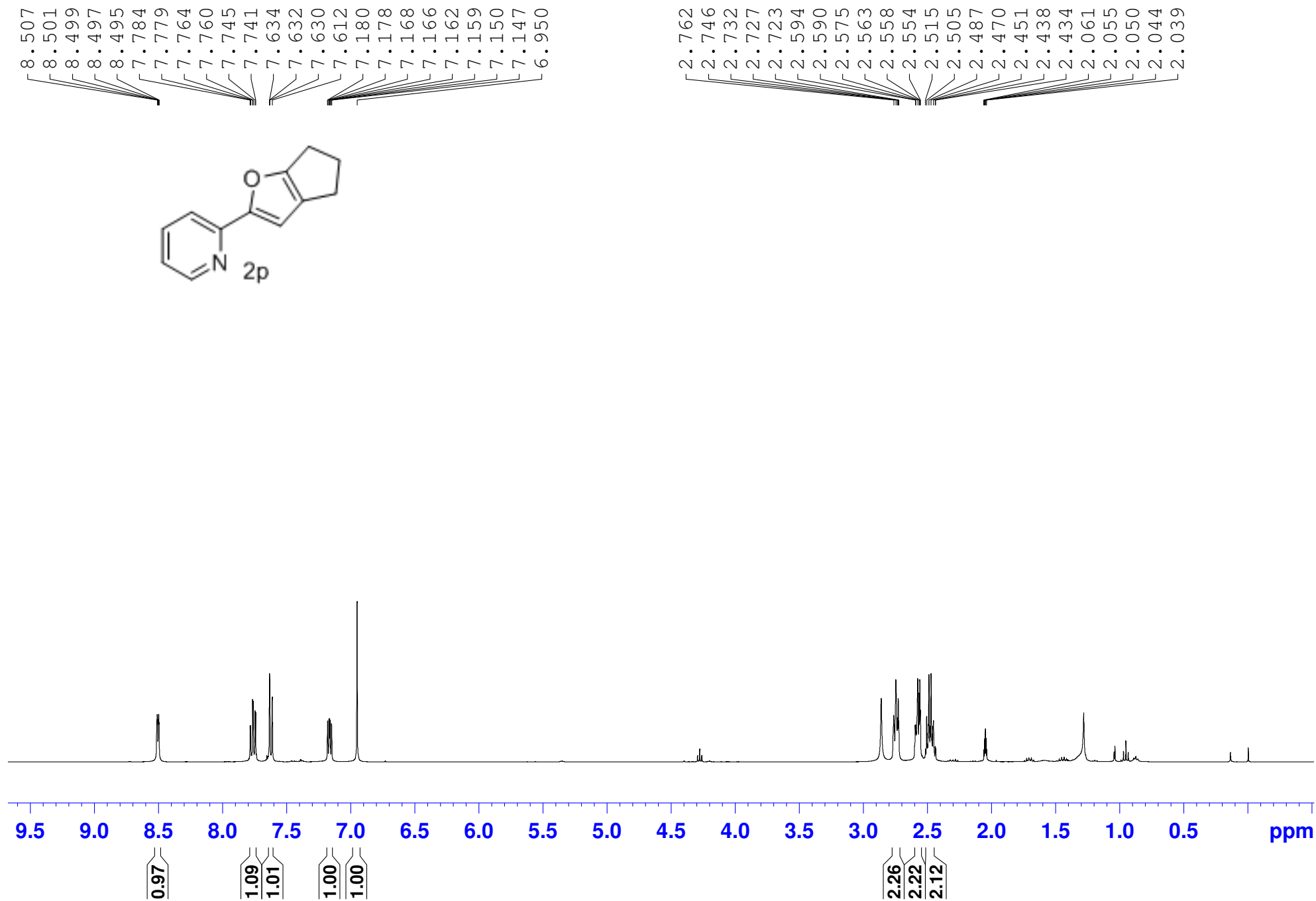
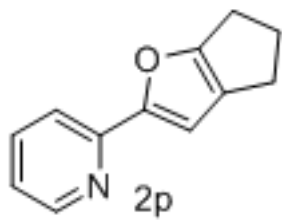
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2.045
2.039

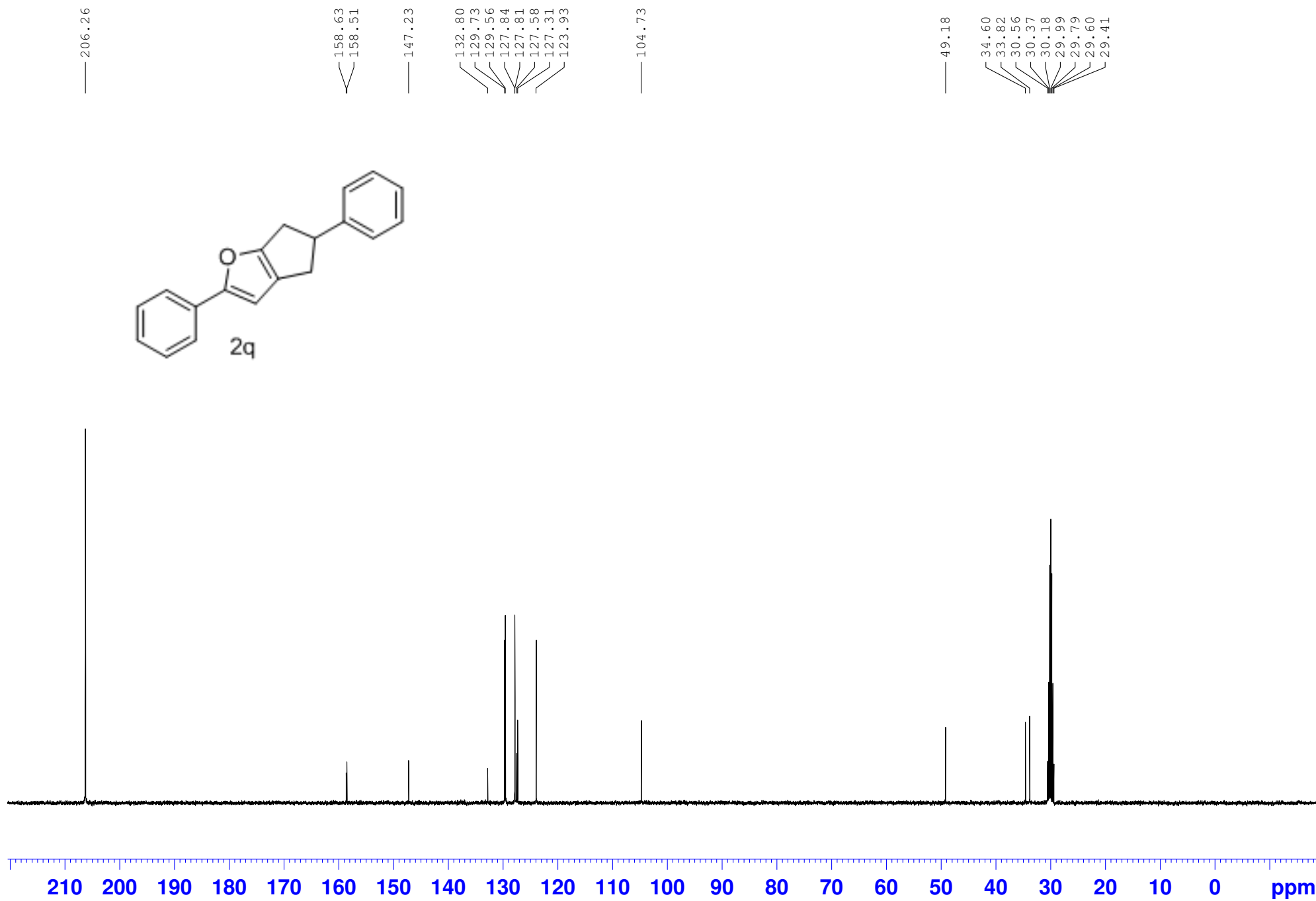
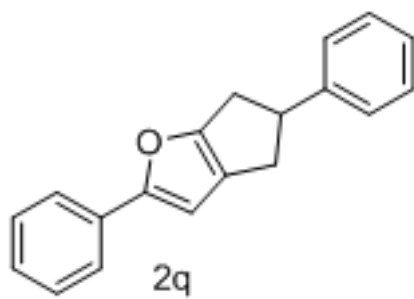


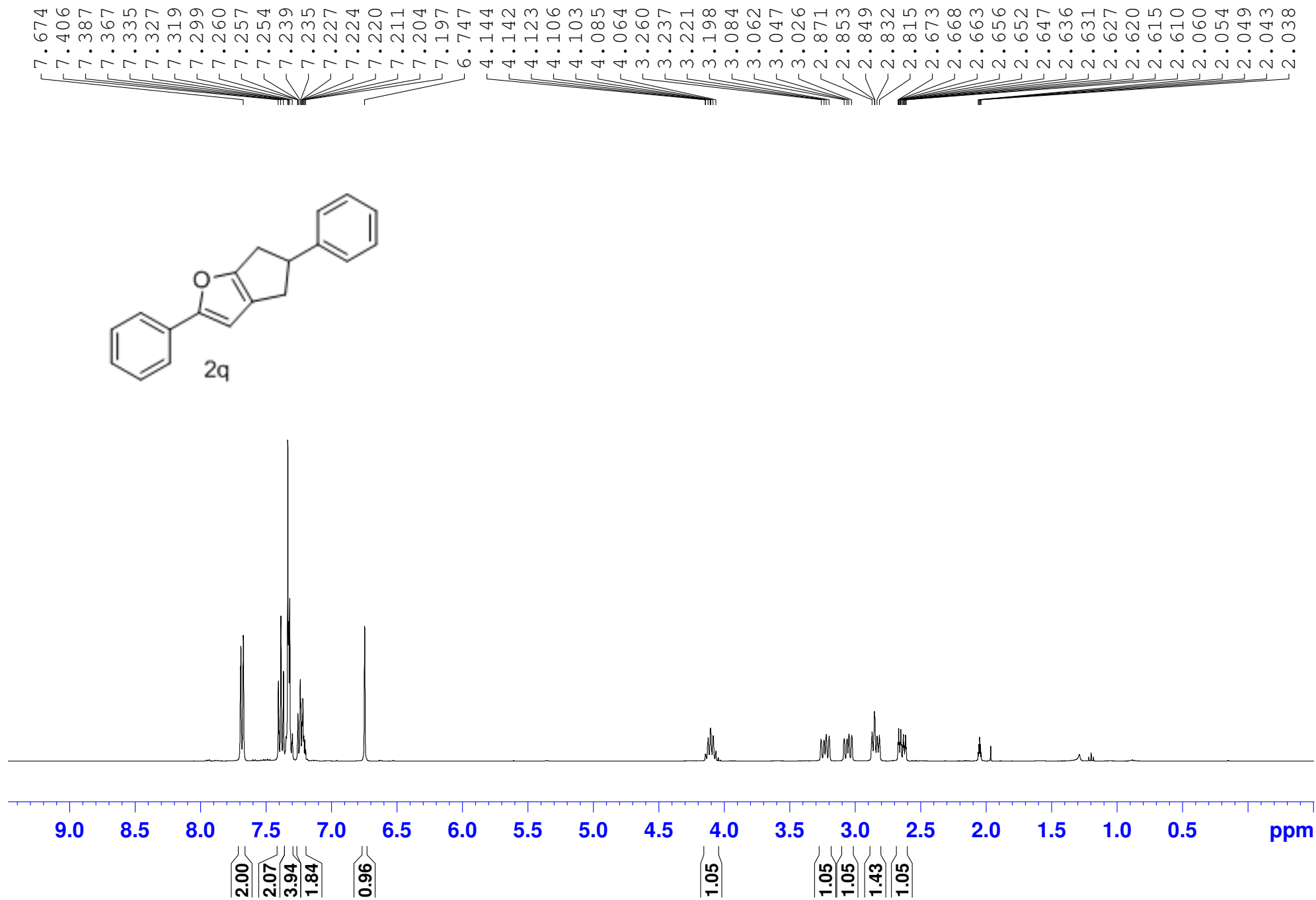


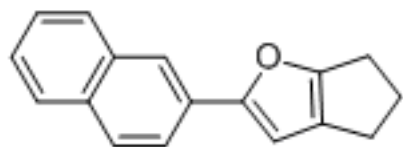












2r

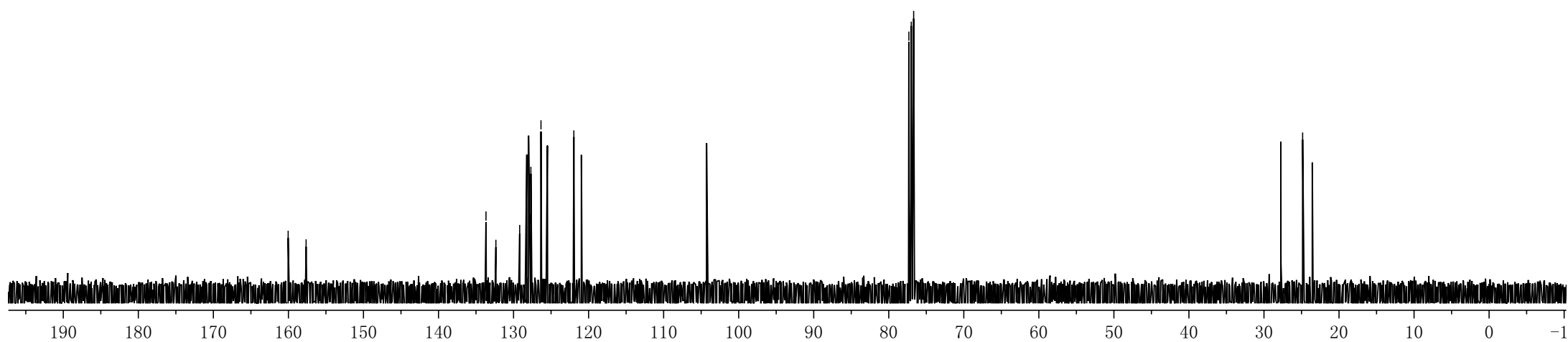
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~157.643

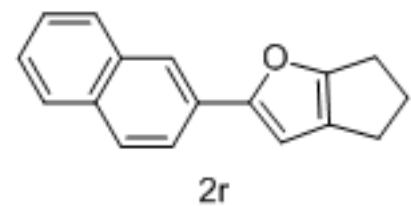
133.673
132.354
129.183
128.232
127.999
127.800
127.692
126.346
125.487
121.965
120.960

—104.279

77.318
77.000
76.682

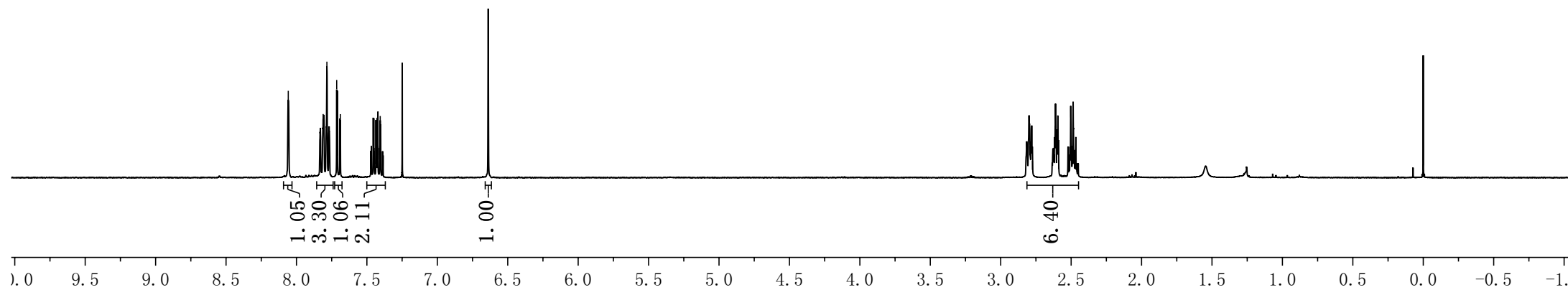
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~24.848
~23.559

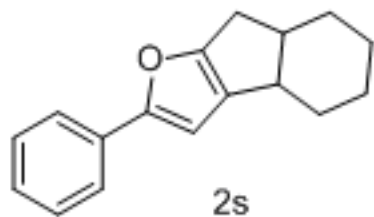




8.058
7.811
7.784
7.714
7.688
7.453
7.448
7.409
7.405
7.249
—6.639

2.793
2.775
2.612
2.597
2.589
2.503





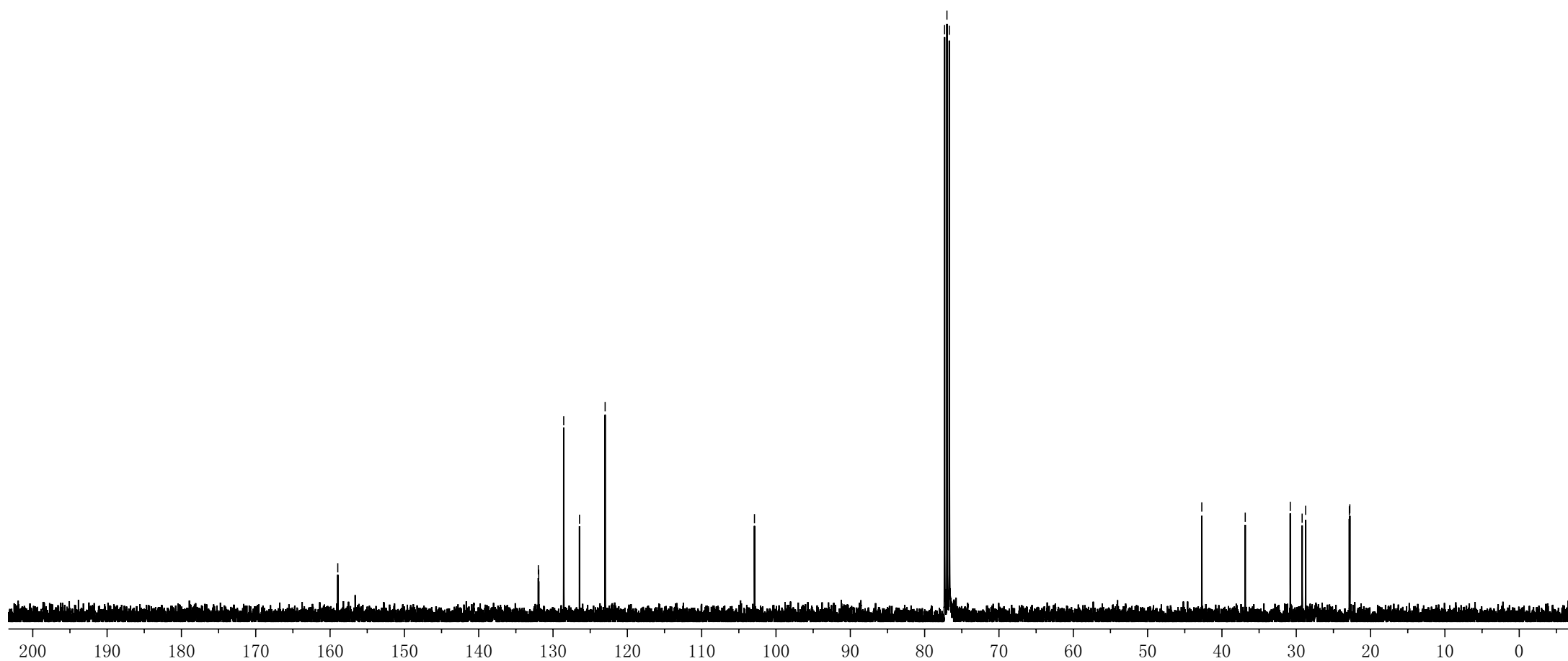
~158.962
~156.576

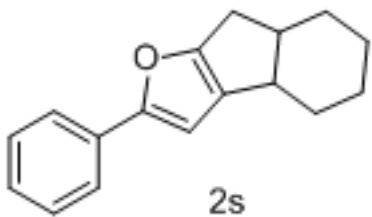
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~128.561
~126.439
~122.990

—102.897

<77.318
<77.000
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~42.709
~36.865
~30.806
~29.210
~28.733
~22.856
~22.792

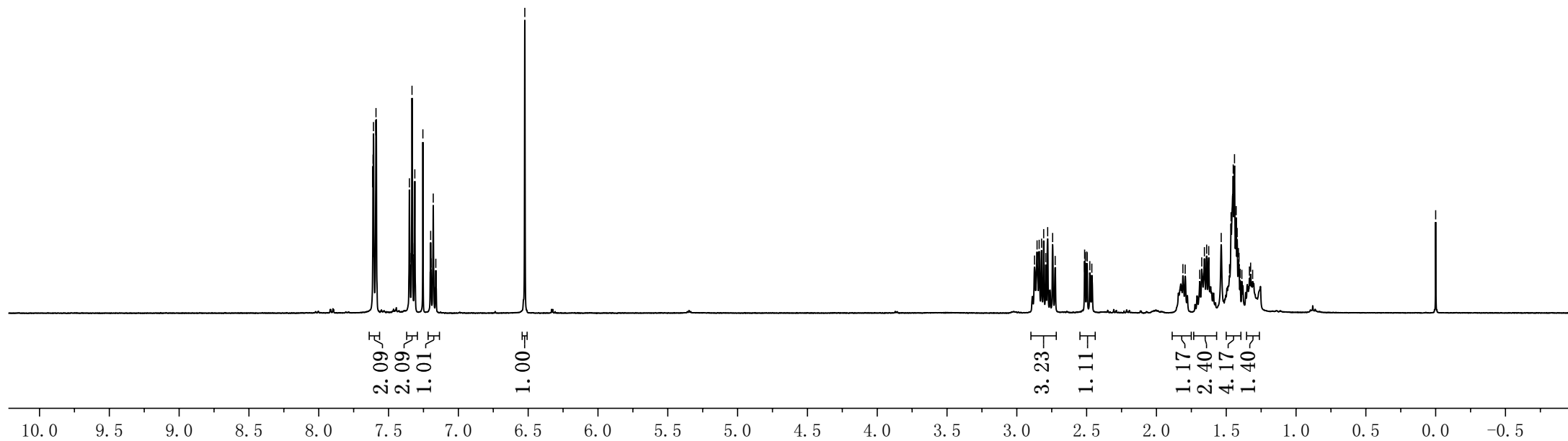


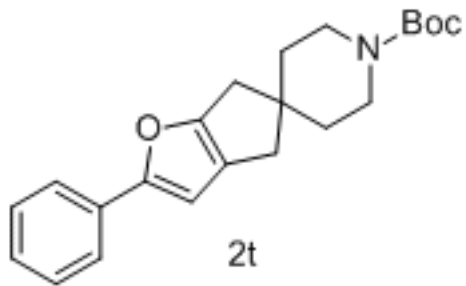


7.611
7.608
7.590
7.588
7.351
7.346
7.332
7.316
7.312
7.254
7.201
7.198
7.196
7.180
7.161
6.524

2.873
2.855
2.840
2.823
2.808
2.792
2.779
2.744
2.725
2.514
2.510
2.499
2.496
2.477
2.463

1.675
1.656
1.640
1.625
1.536
1.466
1.450
1.441
1.429
1.422
1.413
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0.000





157.562
156.484
154.888

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128.599
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124.764
123.070

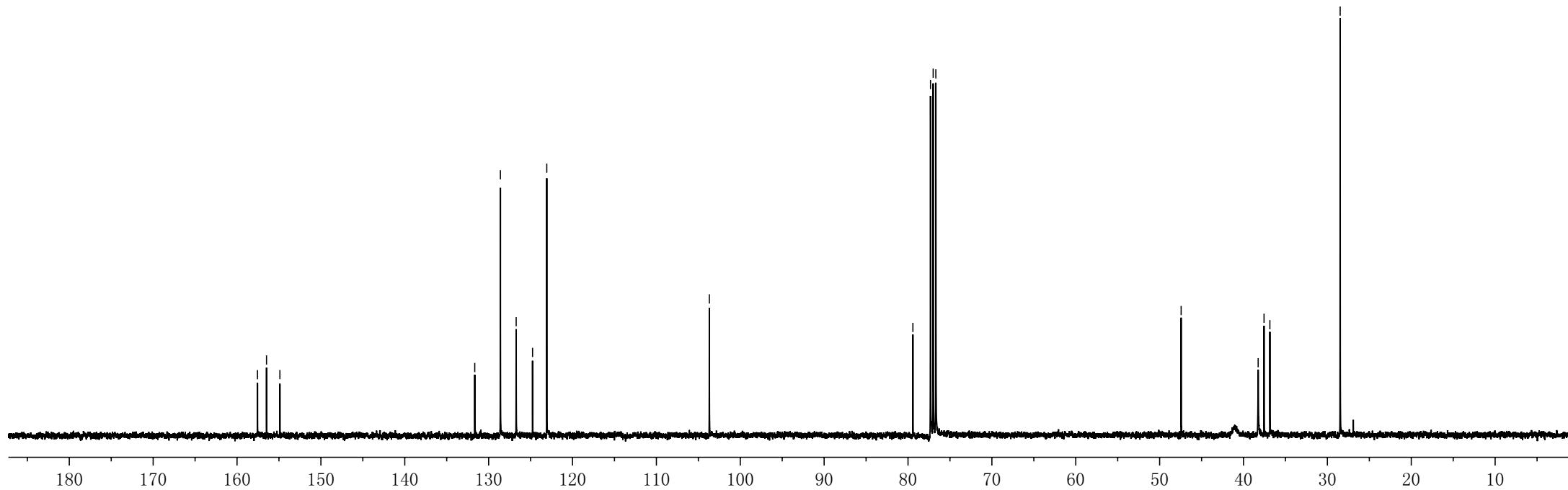
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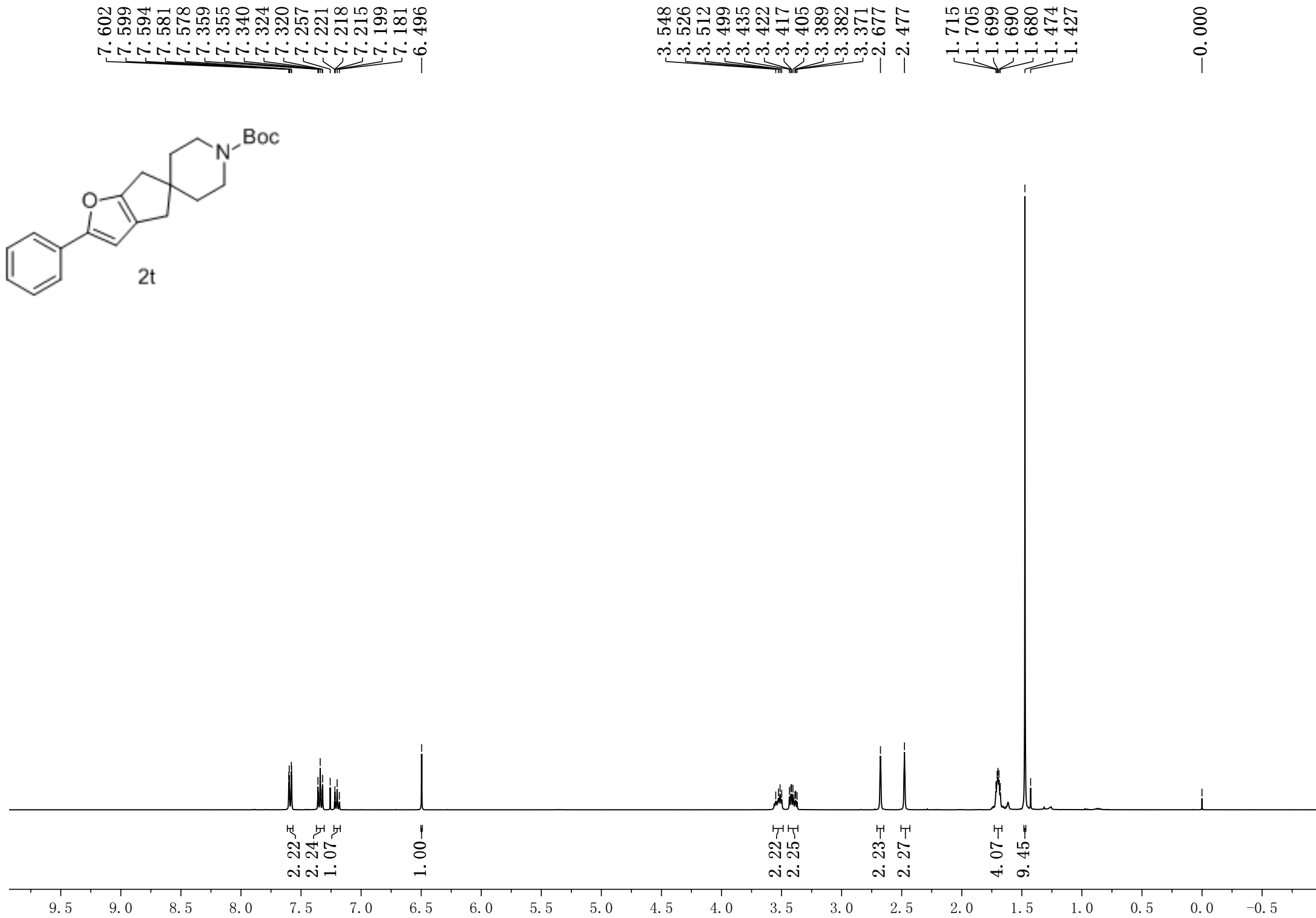
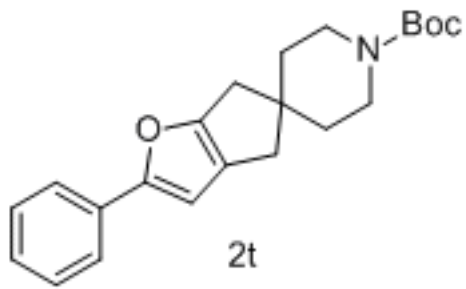
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77.000
76.683

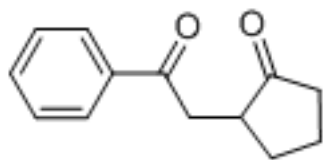
47.435

38.253
37.553
36.856

28.477







—220.32 —197.95

136.62 133.23 128.62 128.04

77.39 77.08 76.76

45.09 38.65 37.56 29.72

—20.85

