Supporting Information

Visible-Light Promoted Intramolecular carboamination of Alkynes for the Synthesis of Oxazolidinone-fused Isoquinolinones

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I. General Information

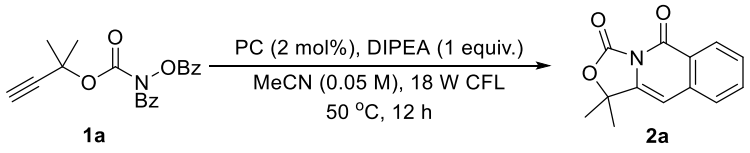
THF, toluene, 1,4-dioxane, and diethyl ether were distilled from sodium benzophenoneketyl prior to use. CH_3CN, iPr$_2$NEt, C_6F_6 and DME were distilled from CaH_2 to use. Sodium hydride (NaH) and methylmagnesium bromide were purchased from Energy and used as received. The other commercially available chemicals were used as received without mentioned. 5 W blue LEDs were used as the light source. NMR spectra were recorded on a Bruker-400 instrument or Oxford instrument. ^{1}H NMR chemical shifts were referenced to tetramethylsilane signal (0 ppm), ^{13}C NMR chemical shifts were referenced to the solvent resonance (77.00 ppm, $CDCl_3$), ^{19}F NMR chemical

shifts were referenced to the solvent resonance. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, m = multiplet, br = broad, q = quadruplet, PE = petroleum ether, EA = ethyl acetate, THF = tetrahydrofuran, DCM = dichloromethane. IR spectra were recorded on a Perkin-Elmer Spectrum One FTIR spectrometer with diamond ATR accessory. High-resolution mass spectra (HRMS) were recorded on LCMS-IT-TOF (ESI-TOF) and EI-TOF (electro-spray ionization-time of flight). Melting points were obtained using an X-4 melting point apparatus (Laboratory Devices, Beijing Taike CO., LTD.).

II. Optimizations of Reaction Conditions.

Optimizations on different photocatalysts were listed in **Table S1**. With most photocatalysts, the reaction gave **2a** in 5-32% yield (entries 1, 2, 3, 5, 7). The *fac*-Ir(ppy)$_3$ led to **2a** in 28% yield with 56% recovery of **1a** (entry 7). The reaction without DIPEA could occur smoothly (entry 8). And photocatalyst was necessary for this reaction (entry 9). So, *fac*-Ir(ppy)$_3$ was chosen as photocatalyst.

Table S1. Optimizations of photocatalysts.[a]

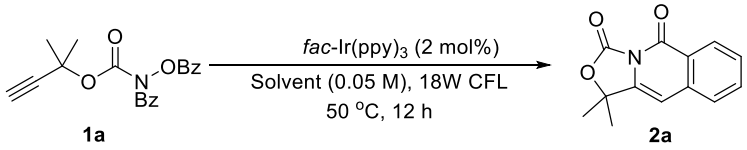


Entry	photocatalyst	2a (%)	Recovery of 1a(%)
1	Ir(dFCF$_3$ppy)$_2$(dtbbpy)(PF$_6$)	14	52
2	Ir(ppy)$_2$(dtbbpy)(PF$_6$)	5	73
3	Ir(dFCF$_3$ppy)$_2$(bpy)(PF$_6$)	32	/
4[b]	Ir(dFCF$_3$ppy)$_2$(bpy)(PF$_6$)	/	99
5	Ru(bpy)$_3$(PF$_6$)$_3$	26	19
6	Eosin Y	/	89
7	*fac*-Ir(ppy)$_3$	28	56
8[b]	*fac*-Ir(ppy)$_3$	20	70
9	/	/	89

[a] Reaction conditions: benzoyloxycarbamate (**1a**) (0.2 mmol), photocatalyst (0.004 mmol), MeCN (4 mL), 50 °C, 12 h. Yields of **2a** and byproducts were determined by ^{1}H NMR using TMSPh as an internal standard. [b] Without DIPEA.

Optimizations on different solvents were listed in **Table S2**. In most solvents, the reaction gave **2a** in 17-57% yield (entries 1-11). The reaction using DMSO as a solvent could afford **2a** in 64% yield. So, DMSO was chosen as solvent.

Table S2. Optimizations of solvents.[a]



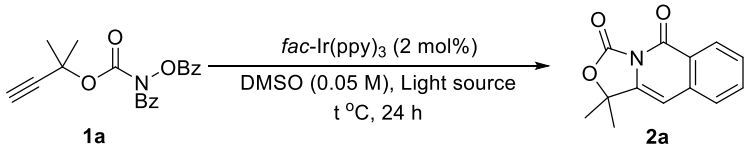
1a **2a**

Entry	Solvent	2a (%)	Recovery of 1a (%)
1	MeCN	20	70
2	THF	17	5
3	dioxane	42	3
4	DME	32	40
5	toluene	34	46
6	PhCl	30	59
7	$PhCF_3$	36	59
8	DCM	27	63
9	DCE	24	67
10	DMF	57	/
11	DMA	48	/
12	DMSO	64	16

[a] Reaction conditions: benzoyloxycarbamate (**1a**) (0.2 mmol), *fac*-$Ir(ppy)_3$ (0.004 mmol), solvent (4 mL), 50 °C, 12 h. Yields of **2a** and byproducts were determined by ^1H NMR using TMSPh as an internal standard.

Optimizations of light source and reaction temperature were listed in **Table S3**. Use of 5 W blue LEDs was better (entry 2). The reaction could deliver **2a** in 88% yield at 60 °C (entry 3). So, 5 W blue LEDs was chosen as light source and 60 °C was chosen as reaction temperature.

Table S3. Optimizations of light source and reaction temperature.[a]



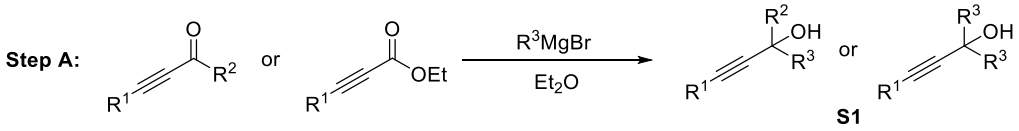
1a **2a**

Entry	Temp. (°C)	Light source	2a (%)	Recovery of 1a (%)
1	50	18W CFL	64	16
2	50	5W blue LEDs	83	/
3	60	5W blue LEDs	88	/
4	80	5W blue LEDs	85	/

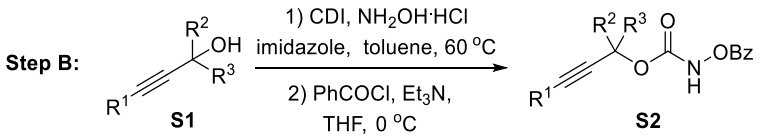
[a] Reaction conditions: benzoyloxycarbamate (**1a**) (0.2 mmol), *fac*-Ir(ppy)$_3$ (0.004 mmol), solvent (4 mL), 50 °C, 12 h. Yields of **2a** and byproducts were determined by ^{1}H NMR using TMSPh as an internal standard.

III. Procedures for the Synthesis of Starting Materials

Starting materials were prepared by the following steps:

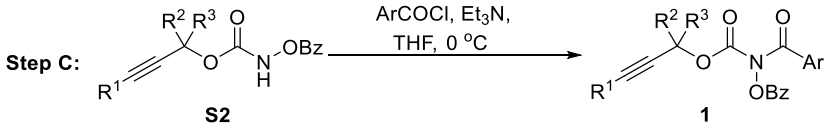


Step A. Substituted propargyl alcohol **S1** were prepared from esters (or ketones) and methylmagnesium bromide： a 500 mL flame-dried three-necked flask was cooled at room temperature under nitrogen, charged with esters (or ketones) (80 mmol) and diethyl ether, added with methylmagnesium bromide (80 mL, 3 M in toluene, 3.0 equiv) dropwisely at 0 °C. The mixture was stirred at room temperature and monitored by TLC. After the **S1** (or ketones) was fully consumed, the reaction was cooled to 0 °C, added dropwisely with saturated ammonium chloride. The resulting suspension was extracted with ether (50 mL x 3). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, concentrated by rotary evaporation and direct used in next step without further purified.



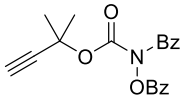
Step B. Alkyl benzoyloxycarbamate **S2** were prepared according to a reported procedure.[1] A 250 mL round-bottom flask was charged with **S1** (60 mmol), toluene (100 mL), *N*,*N*'-Carbonyldiimidazole (78 mmol, 1.3 equiv). The mixture was stirred at 60 °C, after **S2** was fully converted to corresponding intermediate (monitored by TLC), the reaction was concentrated by rotary evaporation. Then the resulting suspension was dissolved in DCM, and washed with water, brine, dried over Na_2SO_4, concentrated by rotary evaporation. Then the resulting suspension was dissolved in MeCN, added with imidazole (120 mmol, 2 equiv) and $NH_2OH{\cdot}HCl$ (180 mmol, 3 equiv). After the intermediate was fully consumed (monitored by TLC), the reaction was concentrated by rotary evaporation. Then the resulting suspension was dissolved in HCl (60 mL, 1 M), the aqueous phase was extracted with extracted with EA (50 mL x 3). The organic layers were combined, washed with brine, dried over Na_2SO_4, concentrated by rotary evaporation, and further purified by flash chromatography on silica gel (PE/EA = 10/1~1/1) to afford the corresponding hydroxylamine.

Hydroxylamine (1 equiv) was dissolved in THF (0.2 M) in a 250 mL round-bottom flask, added with Et_3N (1.0 equiv), then added 4-(trifluoromethyl)benzoyl chloride (1.0 equiv, 1 M in THF) slowly at 0 °C. The mixture was stirred at room temperature for 1~2 hours (monitored by TLC), quenched with H_2O, separated. The aqueous phase was extracted with DCM (40 mL x 3). The organic layers were combined, washed with brine, dried over Na_2SO_4, concentrated by rotary evaporation, and further purified by flash chromatography on silica gel (PE/EA = 10/1~5/1) to afford the corresponding product **S2**.

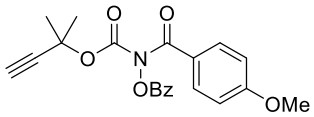


Step C. Hydroxylamine (10 mmol) was dissolved in THF (0.2 M) in a 250 mL round-bottom flask, added with Et_3N (1.0 equiv), then added 4-(trifluoromethyl)benzoyl chloride (1.0 equiv, 1 M in THF) slowly at 0 °C. The mixture was stirred at room temperature for 1-2 hours (monitored by TLC), quenched with H_2O, and separated. The aqueous phase was extracted with DCM (40 mL x 3). The

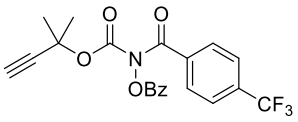
organic layers were combined, washed with brine, dried over Na_2SO_4, concentrated by rotary evaporation, and further purified by flash chromatography on silica gel (PE/EA = 10/1~5/1) to afford the corresponding product **1**.



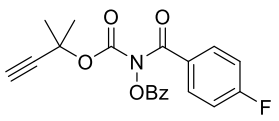
2-methylbut-3-yn-2-yl benzoyl(benzoyloxy)carbamate (1a): 12.02 g, 66% yield, white soild, m.p. 87-88 ºC; IR (neat): 3285, 2923, 1761, 1710, 1325, 1238 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.19-8.13 (m, 2H), 7.79-7.73 (m, 2H), 7.69-7.62 (m, 1H), 7.58-7.42 (m, 5H), 2.59 (s, 1H), 1.45 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.1, 163.9, 148.9, 134.4, 134.2, 132.3, 130.3, 128.7, 128.6, 128.2, 126.3, 82.7, 75.9, 73.9, 28.3; HRMS (ESI) calculated for $[C_{20}H_{18}NO_5]^+$ ($M+H^+$) requires m/z 352.1179, found: m/z 352.1187.



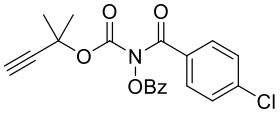
2-methylbut-3-yn-2-yl benzoyloxy(4-methoxybenzoyl)carbamate (1b): 2.58 g, 68% yield, white soild, m.p. 120-121 ºC, IR (neat): 3285, 2924, 1758, 1704, 1368, 1240 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.15 (d, J = 7.8 Hz, 2H), 7.78 (d, J = 7.8 Hz, 2H), 7.70-7.61 (m, 1H), 7.50 (dd, J = 7.8, 7.8 Hz, 2H), 6.94 (d, J = 8.8 Hz, 2H), 3.85 (s, 3H), 2.61 (s, 1H), 1.53 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.2, 164.5, 163.5, 149.0, 134.3, 132.5, 132.2, 128.6, 128.2, 118.3, 114.0, 82.8, 75.8, 73.8, 55.5, 28.3; HRMS (ESI) calculated for $[C_{21}H_{20}NO_6]^+$ ($M+H^+$) requires m/z 382.1285, found: m/z 382.1294.



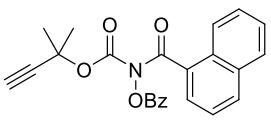
2-methylbut-3-yn-2-yl benzoyloxy(4-(trifluoromethyl)benzoyl)carbamate (1c): 3.50 g, 83% yield, white soild, m.p. 122-125 ºC; IR (neat): 3281, 2924, 1765, 1714, 1323, 1238 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.16 (d, J = 7.6 Hz, 2H), 7.86 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 8.2 Hz, 2H), 7.71-7.64 (m, 1H), 7.56-7.47 (m, 2H), 2.60 (s, 1H), 1.48 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 163.7, 148.6, 137.6, 134.6, 133.6 (q, J = 32.6 Hz), 130.3, 128.8, 128.7, 126.0, 125.3 (q, J = 3.7 Hz), 123.5 (q, J = 271.0 Hz), 82.5, 76.5, 74.2, 28.3; ^{19}F NMR: (376.5 MHz, $CDCl_3$) δ -63.1; HRMS (ESI) calculated for $[C_{21}H_{17}F_3NO_5]^+$ ($M+H^+$) requires m/z 420.1053, found: m/z 420.1060.



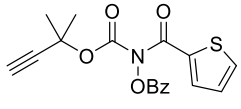
2-Methylbut-3-yn-2-yl (benzoyloxy)(4-fluorobenzoyl)carbamate (1d): 2.83 g, 77% yield, white soild, m.p. 106-108 °C; IR (neat): 3280, 2924, 1762, 1711, 1323, 1236 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.8 Hz, 2H), 7.80 (dd, *J* = 8.0, 5.6 Hz, 2H), 7.67 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.51 (dd, *J* = 7.6, 7.6 Hz, 2H), 7.14 (dd, *J* = 8.6, 8.4 Hz, 2H), 2.60 (s, 1H), 1.52 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 166.4, 166.0, 163.9 (d, *J* = 4.0 Hz), 149.0, 134.4, 131.3 (d, *J* = 8.7 Hz), 130.3, 130.1 (d, *J* = 3.2 Hz), 128.7, 126.2, 115.4 (d, *J* = 22.1 Hz), 82.7, 76.1, 74.0, 28.4; ^{19}F NMR: (376.5 MHz, $CDCl_3$) δ -105.7; HRMS (ESI) calculated for $[C_{20}H_{17}FNO_5]^+$ ($M+H^+$) requires m/z 370.1085, found: m/z 370.1091.



2-methylbut-3-yn-2-yl benzoyloxy(4-chlorobenzoyl)carbamate (1e): 3.35 g, 87% yield, white soild, m.p. = 123-124 °C, IR (neat): 3295, 2924, 1764, 1711, 1325, 1241 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.6 Hz, 2H), 7.71 (d, *J* = 8.6 Hz, 2H), 7.70-7.64 (m, 1H), 7.55-7.47 (m, 2H), 7.44 (d, *J* = 8.6 Hz, 2H), 2.61 (s, 1H), 1.51 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 166.1, 163.8, 148.9, 138.7, 134.5, 132.4, 130.3, 130.1, 128.8, 128.5, 126.1, 82.6, 76.3, 74.1, 28.4; HRMS (ESI) calculated for $[C_{20}H_{17}ClNO_5]^+$ ($M+H^+$) requires m/z 386.0790, found: m/z 386.0796.

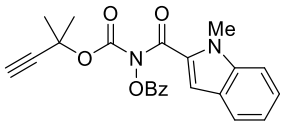


2-methylbut-3-yn-2-yl 1-naphthoyl(benzoyloxy)carbamate (1f): 3.20 g, 80% yield, colorless oil, IR (neat): 3283, 2924, 1762, 1707, 1322, 1238 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.30-8.16 (m, 3H), 7.94 (d, *J* = 8.2 Hz, 1H), 7.86 (d, *J* = 8.2 Hz, 1H), 7.72 (d, *J* = 7.0 Hz, 1H), 7.78-7.56 (m, 2H), 7.56-7.44 (m, 4H), 2.45 (s, 1H), 1.02 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 166.0, 163.8, 147.9, 134.4, 133.2, 133.0, 130.8, 130.3, 129.9, 128.7, 128.2, 127.5, 126.5, 126.2, 125.4, 124.8, 124.6, 82.4, 75.8, 73.8, 27.8; HRMS (ESI) calculated for $[C_{24}H_{20}NO_5]^+$ ($M+H^+$) requires m/z 402.1336, found: m/z 402.1344.

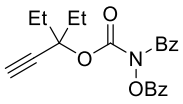


2-methylbut-3-yn-2-yl benzoyloxy(thiophene-2-carbonyl)carbamate (1g): 2.64 g, 74% yield, white soild, m.p. 79-82 °C; IR (neat): 3279, 2924, 1761, 1695, 1357, 1241 cm^{-1}; ^1H NMR: (400 MHz,

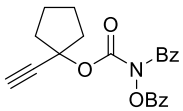
$CDCl_3$) δ 8.14 (d, *J* = 8.0 Hz, 2H), 7.87-7.79 (m, 1H), 7.70-7.58 (m, 2H), 7.54-7.44 (m, 2H), 7.12-7.02 (m, 1H), 2.63 (s, 1H), 1.66 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 163.8, 160.1, 149.0, 134.6, 134.6, 134.4, 133.5, 130.2, 128.7, 127.4, 126.1, 83.0, 76.1, 73.9, 28.5; HRMS (ESI) calculated for $[C_{18}H_{16}NO_5S]^+$ ($M+H^+$) requires m/z 358.0744, found: m/z 358.0754.



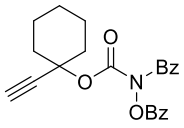
2-methylbut-3-yn-2-yl benzoyloxy(1-methyl-1H-indole-2-carbonyl)carbamate (1h): 2.93 g, 73% yield, white soild, m.p. 107-109 °C; IR (neat): 3065, 2967, 1804, 1693, 1329, 1228 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.20-8.10 (m, 3H), 7.83 (s, 1H), 7.68-7.58 (m, 1H), 7.52-7.44 (m, 2H), 7.36-7.26 (m, 3H), 3.80 (s, 3H), 2.60 (s, 1H), 1.59 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 164.1, 161.5, 150.1, 136.7, 136.5, 134.1, 130.0, 128.6, 126.9, 126.6, 123.0, 122.3, 121.5, 109.8, 108.1, 83.3, 75.1, 73.6, 33.4, 28.5; HRMS (ESI) calculated for $[C_{23}H_{21}N_2O_5]^+$ ($M+H^+$) requires m/z 405.1445, found: m/z 405.1448.



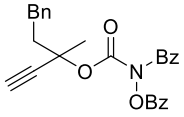
3-Ethylpent-1-yn-3-yl benzoyl(benzoyloxy)carbamate (1i): 3.14 g, 71% yield, colorless oil; IR (neat): 3299, 2922, 1760, 1709, 1317, 1239 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.4 Hz, 2H), 7.77 (d, *J* = 7.4 Hz, 2H), 7.66 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.57-7.47 (m, 3H), 7.47-7.40 (m, 2H), 2.60 (s, 1H), 1.86-1.75 (m, 4H), 0.83 (t, *J* = 7.4 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.1, 163.9, 148.9, 134.3, 134.0, 132.3, 130.2, 128.7, 128.6, 128.2, 126.3, 84.0, 81.1, 75.7, 30.4, 8.0; HRMS (ESI) calculated for $[C_{22}H_{22}NO_5]^+$ ($M+H^+$) requires m/z 380.1492, found: m/z 380.1498.



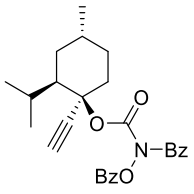
1-ethynylcyclopentyl benzoyl(benzoyloxy)carbamate (1j): 1.94 g, 86% yield, white soild, m.p. 90-92 °C; IR (neat): 3288, 2924, 1761, 1710, 1325, 1239 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.4 Hz, 2H), 7.76 (d, *J* = 7.4 Hz, 2H), 7.70-7.65 (m, 1H), 7.59-7.41 (m, 5H), 2.65 (s, 1H), 2.01-1.88 (m, 4H), 1.70-1.46 (m, 5H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.1, 163.9, 149.2, 134.4, 134.1, 132.3, 130.3, 128.7, 128.6, 128.2, 126.3, 84.5, 82.1, 74.6, 74.6, 40.0, 23.0; HRMS (ESI) calculated for $[C_{22}H_{20}NO_5]^+$ ($M+H^+$) requires m/z 378.1336, found: m/z 378.1346.



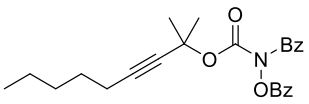
1-ethynylcyclohexyl benzoyl(benzoyloxy)carbamate (1k): 1.53 g, 78% yield, white soild, m.p. 88-90 ºC; IR (neat): 3287, 2927, 1761, 1711, 1323, 1237 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.19-8.13 (m, 2H), 7.81-7.75 (m, 2H), 7.69-7.65 (m, 1H), 7.58-7.41 (m, 5H), 2.67 (s, 1H), 1.90-1.79 (m, 2H), 1.69-1.58 (m, 2H), 1.51-1.36 (m, 5H), 1.31-1.17 (m, 1 H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.1, 163.9, 148.7, 134.3, 134.2, 132.2, 130.3, 128.69, 128.67, 128.2, 126.4, 81.9, 79.6, 75.6, 36.5, 24.6, 22.0; HRMS (ESI) calculated for $[C_{23}H_{22}NO_5]^+$ ($M+H^+$) requires m/z 392.1492, found: m/z 392.1504.



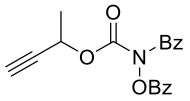
3-methyl-5-phenylpent-1-yn-3-yl benzoyl(benzoyloxy)carbamate (1l): 3.14 g, 71% yield, white soild, m.p. = 98-101 ºC; IR (neat): 3283, 2924, 1761, 1710, 1323, 1239 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.17 (d, J = 7.6 Hz, 2H), 7.79 (d, J = 7.6 Hz, 2H), 7.70-7.62 (m, 1H), 7.56-7.40 (m, 5H), 7.24-7.11 (m, 3H), 6.94 (d, J = 7.2 Hz, 2H), 2.67 (s, 1H), 2.65-2.54 (m, 1H), 2.53-2.41 (m, 1H), 1.97-1.85 (m, 2H), 1.65 (s, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.0, 163.9, 149.0, 140.7, 134.4, 134.0, 132.4, 130.3, 128.8, 128.7, 128.33, 128.27, 128.2, 126.3, 126.0, 81.7, 78.8, 75.2, 43.2, 30.1, 26.1; HRMS (ESI) calculated for $[C_{27}H_{24}NO_5]^+$ ($M+H^+$) requires m/z 442.1649, found: m/z 442.1650.



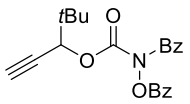
(1R,2R,4R)-1-ethynyl-2-isopropyl-4-methylcyclohexyl benzoyl(benzoyloxy)carbamate (1m): 1.75 g, 93% yield, colorless oil, IR (neat): 3278, 2955, 1773, 1712, 1370, 1243 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.11 (d, J = 7.4 Hz, 2H), 7.78 (d, J = 7.4 Hz, 2H), 7.70-7.63 (m, 1H), 7.56-7.46 (m, 3H), 7.46-7.39 (m, 2H), 2.98-2.87 (m, 1H), 2.65 (s, 1H), 2.47-2.35 (m, 1H), 1.75-1.57 (m, 2H), 1.40-1.29 (m, 2H), 1.29-1.18 (m, 1H), 1.14-1.10 (m, 1H), 0.93-0.82 (m, 7H), 0.64 (d, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.1, 163.8, 149.2, 134.4, 133.5, 132.4, 130.2, 128.8, 128.7, 128.1, 126.2, 82.9, 82.2, 75.5, 51.8, 44.1, 34.3, 27.8, 26.8, 23.7, 21.6, 20.2, 17.9; HRMS (ESI) calculated for $[C_{27}H_{30}NO_5]^+$ ($M+H^+$) requires m/z 448.2118, found: m/z 448.2132.



2-methylnon-3-yn-2-yl benzoyl(benzoyloxy)carbamate (1n): 2.43 g, 57% yield, colorless oil, IR (neat): 3286, 2931, 1751, 1381, 1232 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.17-8.13 (m, 2H), 7.78-7.73 (m, 2H), 7.68-7.61 (m, 1H), 7.57-7.40 (m, 5H), 2.18 (t, *J* = 7.2 Hz, 2H), 1.54-1.45 (m, 2H), 1.40 (s, 6H), 1.37-1.26 (m, 4H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.2, 163.8, 148.8, 134.4, 134.2, 132.0, 130.2, 128.6, 128.5, 128.0, 126.4, 86.5, 79.5, 77.3, 30.8, 28.7, 28.0, 22.0, 18.5, 13.9; HRMS (ESI) calculated for $[C_{25}H_{28}NO_5]^+$ ($M+H^+$) requires m/z 422.1962, found: m/z 422.1966.

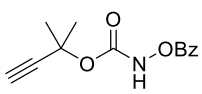


But-3-yn-2-yl benzoyl(benzoyloxy)carbamate (1o): 3.17 g, 94% yield, white soild， m.p. = 81-83 ºC, IR (neat): 3286, 2921, 1757, 1346, 1237 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.18-8.10 (m, 2H), 7.80-7.71 (m, 2H), 7.70-7.61 (m, 1H), 7.58-7.47 (m, 3H), 7.47-7.38 (m, 2H), 5.41-5.30 (m, 1H), 2.49 (d, *J* = 2.2 Hz, 1H), 1.34 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 166.8, 163.8, 150.4, 134.4, 133.5, 132.4, 130.3, 128.7, 128.6, 128.1, 126.1, 80.2, 74.5, 64.2, 20.7; HRMS (ESI) calculated for $[C_{19}H_{15}NNaO_5]^+$ ($M+Na^+$) requires m/z 360.0842, found: m/z 360.0845.



4,4-Dimethylpent-1-yn-3-yl benzoyl(benzoyloxy)carbamate (1p): 2.57 g, 68% yield, white soild， m.p. = 78-80 ºC, IR (neat): 3301, 2923, 1757, 1317, 1240 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.17-8.13 (m, 2H), 7.78-7.73 (m, 2H), 7.68-7.61 (m, 1H), 7.57-7.40 (m, 5H), 2.18 (t, *J* = 7.2 Hz, 2H), 1.54-1.45 (m, 2H), 1.40 (s, 6H), 1.37-1.26 (m, 4H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 166.9, 163.8, 150.7, 134.5, 133.5, 132.5, 130.3, 128.9, 128.8, 128.3, 126.2, 78.2, 76.2, 75.7, 35.0, 25.1; HRMS (ESI) calculated for $[C_{22}H_{21}NO_5Na]^+$ ($M+Na^+$) requires m/z 402.1312, found: m/z 402.1312.

3 were prepared according to **Step A** and **Step B**.



2-Methylbut-3-yn-2-yl (benzoyloxy)carbamate (3): 2.85 g, 77% yield, white soild， m.p. = 110-113ºC, IR (neat): 3287, 2922, 1742, 1452, 1232 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.42 (brs, 1H), 8.14-8.04 (m, 2H),

7.63 (dd, J = 7.4, 7.6 Hz, 1H), 7.48 (dd, J = 8.0, 7.8 Hz, 2H), 2.60 (s, 1H), 1.74 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.2, 129.9, 128.6, 126.7, 83.7, 74.4, 73.2, 28.9; HRMS (ESI) calculated for $[C_{13}H_{13}NO_4Na]^+$ ($M+Na^+$) requires m/z 270.0737, found: m/z 270.0738.

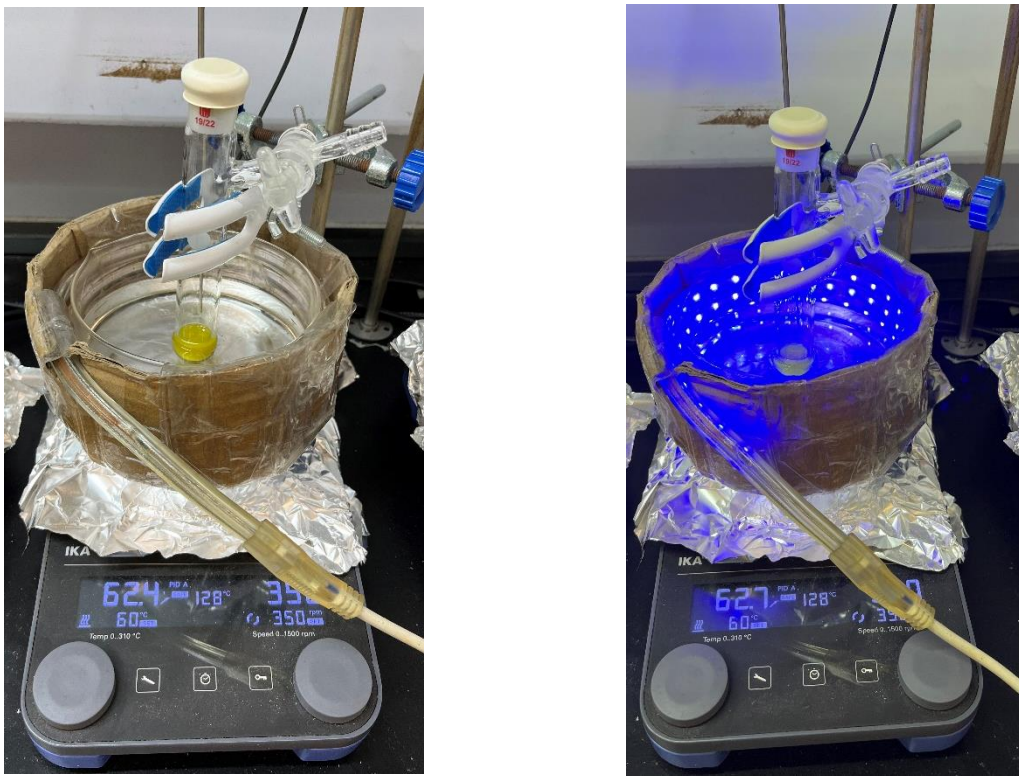
IV. Visible-Light-Promoted Alkyne Carboamination

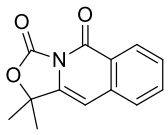
General procedure for visible-light-promoted alkyne carboamination: a 25 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with **1** (0.2 mmol), *fac*-Ir(ppy)$_3$ (0.004 mmol), and DMSO (4 mL). The mixture was degassed for 3 times, and then warmed to room temperature under nitrogen, stirred at 60 °C under 5 W blue LEDs for 24 hours. The reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel to afford the corresponding **2**.

Materials used for set-up:

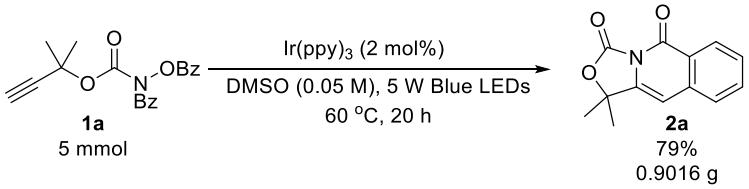
25 mL Schlenk flask. IKA RCT digital. Blue LED lamp strip (455 nm, 5 W/m, 1 m). Oil bath (125 mm). The LED lamp strap was wrapped around the outside of the oil bath.

Fig S1. LED system

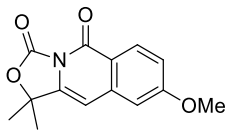




1,1-Dimethyl-1*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2a**): prepared according to the general procedure, using 0.0712 g of benzoyloxycarbamate **1a** (0.20 mmol), 0.0036 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/DCM = 2/1 to DCM), the reaction afforded 0.0492 g, 89% yield, white soild, m.p. = 247-248 °C; IR (neat): 2927, 1804, 1696, 1482, 1283 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1H), 7.72 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.56-7.45 (m, 2H), 6.35 (s, 1H), 1.73 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 158.9, 149.4, 143.5, 136.4 134.2, 129.0, 127.7, 126.4, 125.8, 98.7, 81.6, 27.7; HRMS (ESI) calculated for $[C_{13}H_{12}NO_3]^+$ (M+H$^+$) requires m/z 230.0812, found: m/z 230.0806.

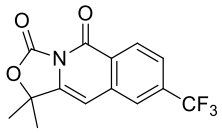


Scaled up reaction: a 250 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with **1a** (1.76 g, 5 mmol), *fac*-Ir(ppy)$_3$ (0.06 g, 0.1 mmol), and DMSO (100 mL). The mixture was degassed for 3 times, and then warmed to room temperature under nitrogen, stirred at 60 °C under 5 W blue LEDs for 20 hours. The reaction was concentrated by rotary evaporation (the solvent could be recovered) and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (monitored by $KMnO_4$) to afford 0.9016 g (79% yield) of **2a**.

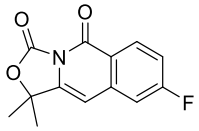


8-Methoxy-1,1-dimethyl-1*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2b**): repared according to the general procedure, using 0.0768 g of benzoyloxycarbamate **1b** (0.20 mmol), 0.0038 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (DCM to DCM/EA = 1/1), the reaction afforded 0.0269 g, 52% yield, colorless oil, IR (neat): 2925, 1806, 1642, 1454, 1289 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 9.0 Hz, 1H), 7.06 (dd, *J* = 9.0,

2.4 Hz, 1H), 6.87 (d, *J* = 2.4 Hz, 1H), 6.27 (s, 1H), 3.93 (s, 3H), 1.72 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 164.3, 158.5, 149.5, 144.4, 138.7, 131.2, 119.1, 116.0, 108.6, 98.6, 81.3, 55.6, 27.7; HRMS (ESI) calculated for $[C_{14}H_{13}NNaO_4]^+$ ($M+Na^+$) requires m/z 282.0737, found: m/z 282.0750.

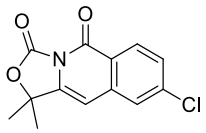


1,1-dimethyl-8-(trifluoromethyl)-1*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2c**): repared according to the general procedure, using 0.2123 g of benzoyloxycarbamate **1c** (0.50 mmol), 0.0065 g of *fac*-Ir(ppy)$_3$ (0.01 mmol), and 10 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 10/1-5/1), the reaction afforded 0.1070 g, 72% yield, colorless oil, IR (neat): 2930, 1698, 1653, 1442, 1285 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.59 (d, *J* = 8.4 Hz, 1H), 7.80 (s, 1H), 7.74 (d, *J* = 8.4 Hz, 1H), 6.42 (s, 1H), 1.76 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 158.0, 148.9, 145.1, 136.6, 136.3 (q, *J* = 156.8 Hz), 130.0, 128.1, 123.9 (q, *J* = 2.9 Hz), 123.7 (q, *J* = 3.6 Hz), 121.9, 98.2, 81.8, 27.7; ^{19}F NMR: (376.5 MHz, $CDCl_3$) δ -63.3; HRMS (ESI) calculated for $[C_{14}H_{11}F_3NO_3]^+$ ($M+H^+$) requires m/z 298.0686, found: m/z 298.0699.

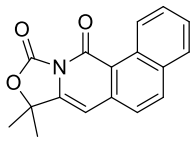


8-Fluoro-1,1-dimethyl-3*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2d**): repared according to the general procedure, using 0.0741 g of benzoyloxycarbamate **1d** (0.20 mmol), 0.0037 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1 to DCM), the reaction afforded 0.0326 g, 66% yield, white soild, m.p. = 297-298 ºC, IR (neat): 2925, 1804, 1691, 1462, 1292 cm^{-1}; ^{1}H NMR: (400 MHz, CD_2Cl_2) δ 8.44 (dd, *J* = 8.6, 5.8 Hz, 1H), 7.29-7.14 (m, 2H), 6.33 (s, 1H), 1.71 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 159.7, 150.7, 146.8, 140.8 (d, *J* = 10.9 Hz), 133.4 (d, *J* = 10.2 Hz), 123.9, 117.4 (d, *J* = 23.3 Hz), 113.6 (d, *J* = 22.6 Hz), 99.7 (d, *J* = 2.9 Hz), 83.2, 31.2, 28.9; ^{19}F NMR: (376.5 MHz, CD_2Cl_2) δ -102.2; HRMS (ESI) calculated for $[C_{13}H_{10}FNNaO_3]^+$ ($M+Na^+$)

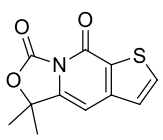
requires m/z 270.0537, found: m/z 270.0550.



8-chloro-1,1-dimethyl-1*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2e**): repared according to the general procedure, using 0.1975 g of benzoyloxycarbamate **1e** (0.20 mmol), 0.0065 g of *fac*-Ir(ppy)$_3$ (0.01 mmol), and 10 mL of DMSO. After reaction at 60 ^{o}C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 10/1-5/1 to DCM), the reaction afforded 0.0811 g, 62% yield, colorless oil, IR (neat): 2925, 1813, 1689, 1463, 1283 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 8.4 Hz, 1H), 7.51-7.43 (m, 2H), 6.27 (s, 1H), 1.74 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 158.2, 149.1, 145.0, 140.9, 137.7, 130.7, 128.2, 125.9, 124.1, 97.7, 81.6, 27.7; HRMS (ESI) calculated for $[C_{13}H_{11}ClNO_3]^+$ (M+H$^+$) requires m/z 264.0422, found: m/z 264.0435.

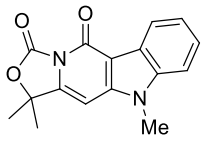


8,8-dimethyl-8*H*-benzo[*h*]oxazolo[*3,4-b*]isoquinoline-10,12-dione (**2f**): repared according to the general procedure, using 0.0802 g of benzoyloxycarbamate **1f** (0.20 mmol), 0.0037 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 ^{o}C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1 to DCM), the reaction afforded 0.0294 g, 53% yield, colorless oil, IR (neat): 2926, 1814, 1696, 1462, 1290 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 10.04 (d, *J* = 8.8 Hz, 1H), 8.11 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.77 (dd, *J* = 7.2, 7.6 Hz, 1H), 7.64 (dd, *J* = 7.2, 7.6 Hz, 1H), 7.50 (d, *J* = 8.8 Hz, 1H), 6.49 (s, 1H), 1.77 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 159.4, 149.7, 144.9, 138.9, 136.0, 132.6, 132.0, 129.5, 128.5, 127.0, 126.9, 124.4, 119.3, 99.4, 81.1, 27.6; HRMS (ESI) calculated for $[C_{17}H_{14}NO_3]^+$ (M+H$^+$) requires m/z 280.0968, found: m/z 280.0988.

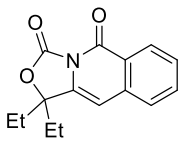


5,5-dimethyl-5*H*-oxazolo[*3,4-a*]thieno[*3,2-d*]pyridine-7,9-dione (**2g**): repared according to the general procedure, using 0.0717 g of benzoyloxycarbamate **1g** (0.20 mmol), 0.0036 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 ^{o}C for 12 hours, the reaction was

concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (DCM), the reaction afforded 0.0207 g, 44% yield, white soild, m.p. = 238-240 °C; IR (neat): 2926, 1806, 1689, 1462, 1268 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 5.0 Hz, 1H), 7.22 (d, *J* = 5.0 Hz, 1H), 6.54 (s, 1H), 1.73 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 154.6, 149.2, 146.3, 145.4, 136.6, 129.8, 124.5, 95.9, 81.5, 27.7; HRMS (ESI) calculated for $[C_{11}H_{10}NO_3S]^+$ ($M+H^+$) requires m/z 236.0376, found: m/z 236.0387.

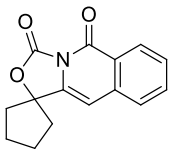


1,1,10-trimethyloxazolo[*3',4':1,6*]pyrido[*4,3-b*]indole-3,5(1*H*,10*H*)-dione (**2h**): repared according to the general procedure, using 0.2024 g of benzoyloxycarbamate **1h** (0.20 mmol), 0.0091 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 1/1 to EA), the reaction afforded 0.0618 g, 44% yield, colorless oil, IR (neat): 2933, 1803, 1690, 1368, 1285 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.36 (d, *J* = 7.6 Hz, 1H), 7.44-7.30 (m, 3H), 6.38 (s, 1H), 3.83 (s, 3H), 1.75 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 155.4, 149.6, 149.0, 144.6, 138.9, 124.7, 124.4, 122.6, 122.0, 109.2, 106.6, 87.5, 81.1, 29.9, 27.7; HRMS (ESI) calculated for $[C_{16}H_{15}N_2O_3]^+$ ($M+H^+$) requires m/z 283.1077, found: m/z 283.1087.

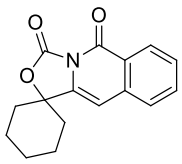


1,1-Diethyl-3H-oxazolo[3,4-b]isoquinoline-3,5(1H)-dione (**2i**): repared according to the general procedure, using 0.0760 g of benzoyloxycarbamate **1l** (0.20 mmol), 0.0036 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 10/1-5/1), the reaction afforded 0.0435 g, 85% yield, white soild, m.p. = 210-211 °C, IR (neat): 2926, 1804, 1692, 1463, 1282 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.52-8.45 (m, 1H), 7.77-7.68 (m, 1H), 7.57-7.48 (m, 2H), 6.30 (s, 1H), 2.17-2.03 (m, 2H), 1.97-1.84 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 159.0, 150.1, 141.0, 136.3, 134.2,

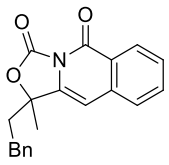
129.0, 127.7, 126.5, 125.7, 99.3, 87.6, 32.0, 7.2; HRMS (ESI) calculated for $[C_{15}H_{16}NO_3]^+$ (M+H$^+$) requires m/z 258.1125, found: m/z 258.1134.



spiro[cyclopentane-1,1'-oxazolo[*3,4-a*]pyridine]-3',5'-dione (**2j**): repared according to the general procedure, using 0.0758 g of benzoyloxycarbamate **1i** (0.20 mmol), 0.0035 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1), the reaction afforded 0.0383 g, 75% yield, white soild, m.p. = 207-209 ºC; IR (neat): 2953, 1806, 1774, 1481, 1234 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 8.0 Hz, 1H), 7.75-7.66 (m, 1H), 7.56-7.44 (m, 2H), 6.38 (s, 1H), 2.36-2.22 (m, 2H), 2.15-1.90 (m, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 158.9, 149.6, 142.4, 136.4, 134.2, 129.0, 127.6, 126.4, 125.7, 98.9, 91.3, 40.8, 24.4; HRMS (ESI) calculated for $[C_{15}H_{13}NNaO_3]^+$ (M+Na$^+$) requires m/z 278.0788, found: m/z 278.0800.

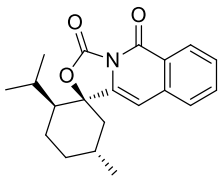


spiro[cyclohexane-1,1'-oxazolo[*3,4-b*]isoquinoline]-3',5'-dione (**2k**): repared according to the general procedure, using 0.1198 g of benzoyloxycarbamate **1j** (0.50 mmol), 0.0068 g of *fac*-Ir(ppy)$_3$ (0.01 mmol), and 10 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1), the reaction afforded 0.0493 g, 49% yield, white soild, m.p. = 217-219 ºC; IR (neat): 2927, 1805, 1698, 1482, 1280 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 7.8 Hz, 1H), 7.76-7.66 (m, 1H), 7.55-7.46 (m, 2H), 6.34 (s, 1H), 2.08-1.93 (m, 2H), 1.90-1.73 (m, 7H), 1.47-1.31 (m, 1H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 159.1, 149.7, 143.5, 136.4, 134.2, 129.0, 127.6, 126.5, 125.8, 99.1, 83.3, 36.6, 24.3, 21.5; HRMS (ESI) calculated for $[C_{16}H_{15}NNaO_3]^+$ (M+Na$^+$) requires m/z 292.0944, found: m/z 292.0955.

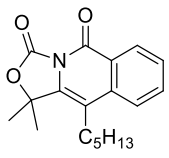


1-methyl-1-phenethyl-1*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2l**): repared according to the general procedure, using 0.2225 g of benzoyloxycarbamate **1m** (0.50 mmol), 0.0067 g of *fac*-Ir(ppy)$_3$ (0.01

mmol), and 10 mL of C_6F_6. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 4/1-2/1), the reaction afforded 0.0930 g, 58% yield, colorless oil, IR (neat): 2931, 1810, 1697, 1483, 1278 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1H), 7.76-7.68 (m, 1H), 7.56-7.45 (m, 2H), 7.25-7.18 (m, 2H), 7.16-7.08 (m, 3H), 6.35 (s, 1H), 2.83-2.71 (m, 1H), 2.63-2.50 (m, 1H), 2.43-2.30 (m, 1H), 2.26-2.14 (m, 1H), 1.76 (s, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 158.9, 149.6, 142.1, 139.7, 136.2, 134.3, 129.0, 128.5, 128.1, 127.8, 126.5, 126.3, 125.7, 99.3, 83.6, 42.1, 29.4, 26.6; HRMS (ESI) calculated for $[C_{20}H_{17}NNaO_3]^+$ ($M+Na^+$) requires m/z 342.1101, found: m/z 342.1111.

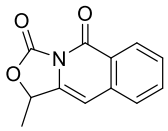


(1*S*,2*S*,5*R*)-2-isopropyl-5-methylspiro[cyclohexane-1,1'-oxazolo[*3,4-b*]isoquinoline]-3',5'-dione (**2m**): repared according to the general procedure, using 0.0900 g of benzoyloxycarbamate **1k** (0.20 mmol), 0.0036 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/DCM = 10/1-2/1), the reaction afforded 0.0300 g, 46% yield, white soild, m.p. = 260-263 ºC; IR (neat): 2927, 1796, 1693, 1480, 1278 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1H), 7.76-7.66 (m, 1H), 7.56-7.46 (m, 2H), 6.31 (s, 1H), 2.01-1.85 (m, 3H), 1.81-1.56 (m, 5H), 1.54-1.41 (m, 1H), 1.20-1.04 (m, 1H), 0.95 (d, *J* = 6.0 Hz, 3H), 0.90-0.78 (m, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 159.0, 149.8, 143.0, 136.5, 134.2, 129.0, 127.5, 126.5, 125.7, 98.7, 87.8, 49.6, 47.5, 34.1, 28.3, 27.1, 23.7, 21.7, 21.4, 17.8; HRMS (ESI) calculated for $[C_{20}H_{24}NO_3]^+$ ($M+H^+$) requires m/z 326.1751, found: m/z 326.1756.

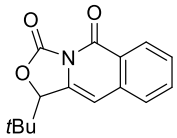


1,1-dimethyl-10-pentyl-1*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2n**): repared according to the general procedure, using 0.2110 g of benzoyloxycarbamate **1n** (0.20 mmol), 0.0091 g of *fac*-Ir(ppy)$_3$ (0.004 mmol), and 10 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further

purified by flash chromatography on silica gel (PE/EA = 10/1-2/1), the reaction afforded 0.1158 g, 77% yield, white soild, m.p. = 150-152 ºC; IR (neat): 2955, 1821, 1695, 1484, 1278 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.0 Hz, 1H), 7.81-7.73 (m, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.58-7.50 (m, 1H), 2.73-2.64 (m, 2H), 1.81 (s, 6H), 1.69-1.57 (m, 2H), 1.55-1.46 (m, 2H), 1.46-1.36 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 158.9, 149.4, 137.4, 136.7, 134.0, 129.2, 127.4, 126.5, 123.2, 111.2, 82.1, 32.2, 30.1, 26.9, 26.5, 22.4, 14.0; HRMS (ESI) calculated for $[C_{18}H_{23}NNaO_3]^+$ ($M+Na^+$) requires m/z 324.1570, found: m/z 324.1584.



1-methyl-3*H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2o**): repared according to the general procedure, using 0.1699 g of benzoyloxycarbamate **1o** (0.50 mmol), 0.0068 g of *fac*-$Ir(ppy)_3$ (0.01 mmol), and 10 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^1H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 4/1 to DCM), the reaction afforded 0.0278 g, 26% yield, colorless oil; IR (neat): 2919, 1813, 1651, 1463, 1279 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 7.6 Hz, 1H), 7.77-7.68 (m, 1H), 7.58-7.47 (m, 2H), 6.40 (s, 1H), 5.53-5.43 (m, 1H), 1.73 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 158.9, 150.1, 139.4, 136.3, 134.3, 129.0, 127.8, 126.5, 125.8, 99.9, 73.5, 20.6; HRMS (ESI) calculated for $[C_{12}H_9NNaO_3]^+$ ($M+Na^+$) requires m/z 238.0475, found: m/z 238.0473.

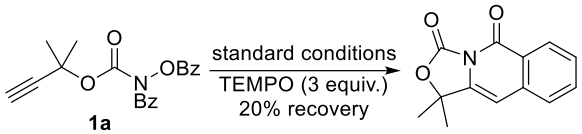


1-(*tert*-butyl)-*3H*-oxazolo[*3,4-b*]isoquinoline-3,5-dione (**2p**): repared according to the general procedure, using 0.0762 g of benzoyloxycarbamate **1p** (0.20 mmol), 0.0029 g of *fac*-$Ir(ppy)_3$ (0.004 mmol), and 4 mL of DMSO. After reaction at 60 ºC for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^1H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 4/1 to DCM), the reaction afforded 0.0171 g, 33% yield, colorless oil; IR (neat): 2920, 1806, 1695, 1462, 1280 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1H), 7.79-7.67 (m, 1H), 7.60-7.46 (m, 2H), 6.47 (s, 1H), 5.05-4.97 (m, 1H), 1.10 (s, 9H); ^{13}C NMR: (100 MHz,

$CDCl_3$) δ 159.0, 150.3, 136.3, 136.1, 134.3, 128.9, 127.9, 126.6, 125.8, 102.3, 84.3, 36.2, 24.6; HRMS (ESI) calculated for $[C_{15}H_{15}NNaO_3]^+$ ($M+Na^+$) requires m/z 280.0944, found: m/z 280.0945.

V. Mechanistic Studies

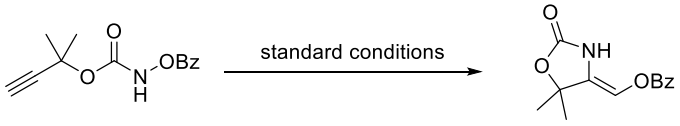
1. Radical inhibitor control experiments



1a **2a** 9%

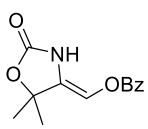
According to the general procedure Conditions A, a 25 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with 0.0786 g (0.2 mmol) of **1a**, 0.0036 g (0.004 mmol) of *fac*-Ir(ppy)$_3$, 5 mL of DMSO, and 0.0938 g (0.6 mmol) of TEMPO. The mixture was degassed for 3 times, then warmed to room temperature under nitrogen, stirred at 60 °C under blue LED for 12 hours. The mixture was concentrated by rotary evaporation, 20% recovery of **1a** and 9% yield of **2a** was observed (NMR yield, using Trimethylphenylsilane as an internal standard).

2. Alkyne oxyamidation



3 **4** 15%

A 25 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with **3** (0.2 mmol), *fac*-Ir(ppy)$_3$ (0.004 mmol), and DMSO (4 mL). The mixture was degassed for 3 times, and then warmed to room temperature under nitrogen, stirred at 60 °C under 5 W blue LEDs for 24 hours. The reaction yields were detected by NMR. 15% NMR yield of **4** was detected.



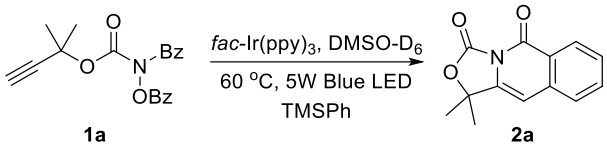
(*Z*)-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)methyl benzoate (**4**): white soild, m.p. = 137-138 °C; IR (neat): 2922, 1757, 1712, 1460, 1269 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.0-8.7 (br, 1H), 8.20 (d, *J* = 7.6 Hz, 2H), 7.63 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.51 (dd, *J* = 7.6, 7.4 Hz, 2H), 6.96 (s, 1H), 1.64 (s,

6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.8, 155.9, 133.8, 130.6, 130.1, 128.7, 128.5, 111.2, 82.9, 28.3; HRMS (ESI) calculated for $[C_{13}H_{13}NNaO_4]^+$ ($M+Na^+$) requires m/z 270.0737, found: m/z 270.0737.

3. Light on and off experiments.

A 25 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with **1a** (0.2 mmol), *fac*-Ir(ppy)$_3$ (0.004 mmol), DMSO-D_6 (4 mL), and trimethylphenylsilane (10 μL). The mixture was degassed for 3 times, and then warmed to room temperature under nitrogen, stirred at 60 ºC under 5 W blue LEDs. The reaction yields were detected by NMR.

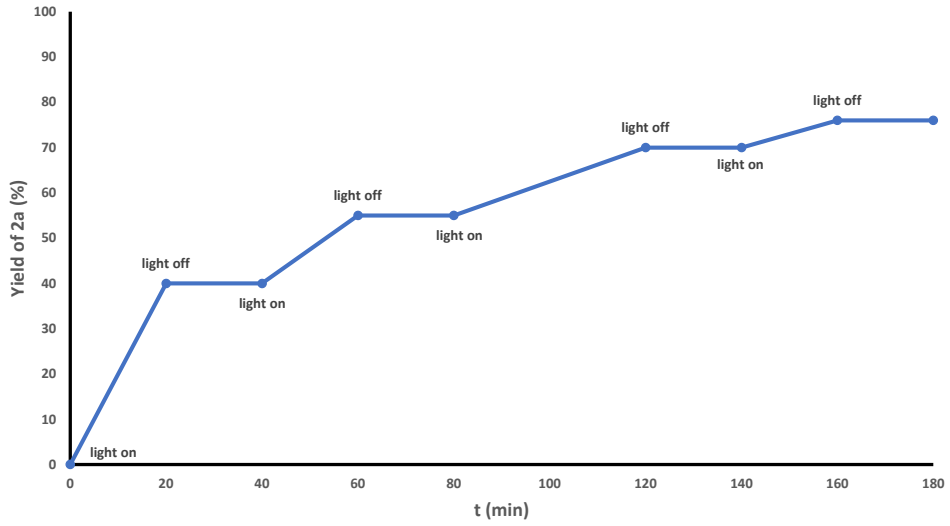
Table S4. Light on and off experiments.



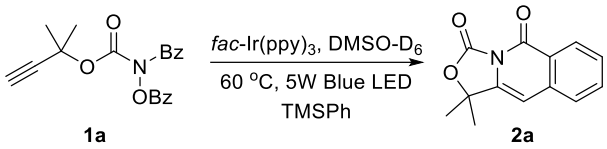
1a **2a**

Entry	Time (min)	Light	2a (%)	Recovery of 1a (%)
1	0	On	0	100
2	20	Off	40	60
3	40	On	40	60
4	60	Off	55	45
5	80	On	55	45
6	100	Off	70	30
7	120	On	70	30
8	140	Off	76	24
9	160	/	76	24

Fig. S2 Light on and off experiments.



4. Quantum yield experiments.



1a **2a**

A 25 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with **1a** (0.2 mmol), *fac*-Ir(ppy)$_3$ (0.004 mmol), DMSO-D$_6$ (4 mL), and trimethylphenylsilane (10 μL). The mixture was degassed for 3 times, and then warmed to room temperature under nitrogen, stirred at 60 °C under 5 W blue LEDs for 1200 s. The reaction yields were detected by NMR.

The quantum yield is calculated by:[2]

$$\Phi = \frac{\text{moles of product formed}}{\text{moles of light absorbed}} = \frac{\text{moles of product formed}}{\text{moles of photon} \cdot f}$$

The fraction of light absorbed (f) is calculated by:

The molar absorption coefficient ε of *fac*-(Ir(ppy)$_3$) in 455 nm was 2800 $L \cdot mol^{-1} \cdot cm^{-1}$.[3] The diameter of the 25 mL Schlenk flask is 2 cm. The *fac*-(Ir(ppy)$_3$) concentration is 0.001 mol/L. So the absorbance $A = \varepsilon \cdot c \cdot l = 2800 \times 2 \times 0.001 = 5.6$. So the fraction of light absorbed $f = 1\text{-}10^{-A} > 0.9999$.

The moles of photon is calculated by:

$$\text{moles of photon} = \frac{\text{total number of photons}}{NA} = \frac{\frac{E_{total}}{h\nu}}{NA} = \frac{\frac{P \cdot t}{h\nu}}{NA} = \frac{\frac{p \cdot S \cdot t}{\frac{hc}{\lambda}}}{NA}$$

NA is Avogadro constant. h is Planck constant. c is lightspeed. λ is light wavelength. t is reaction time. p is light intensity. S is the illumination area. The diameter of the 25 mL Schlenk flask is 2 cm. So when the reaction solution is 4 mL, the solution height is about 4/π cm. And the illumination area S is about 8 cm^2.

Determination of the light intensity at the 455 nm LED systems. The light intensity was detected by Sanwa laser power meter LP1. The instrument probe was placed in the center of the oil bath, and the results were as follows.

Table S5. The light intensity at the 455 nm LED systems

Entry	**Light intensity (mW/cm^2)**
1	2.139
2	2.171
3	2.204
4	2.171
5	2.237
Average	2.184

Experiment: 71.2 mg (0.2 mmol) **1a**, 2.6 mg (0.004 mmol) *fac*-$Ir(ppy)_3$, 4.0 mL (0.05 M) DMSO-D6 after 1200 s yielded 40% of **2a**.

Φ(40%) = 1.0035.

Sample quantum yield calculation:

$$\Phi = \frac{8 \times 10^{-5}\ \text{mol}}{\frac{2.184 \times 8 \times 10^{-3} \times 1200}{\frac{6.626 \times 10^{-34} \times 3 \times 10^{8}}{455 \times 10^{-9}}}} \times \frac{1.00}{6.02 \times 10^{23}} = 1.0035$$

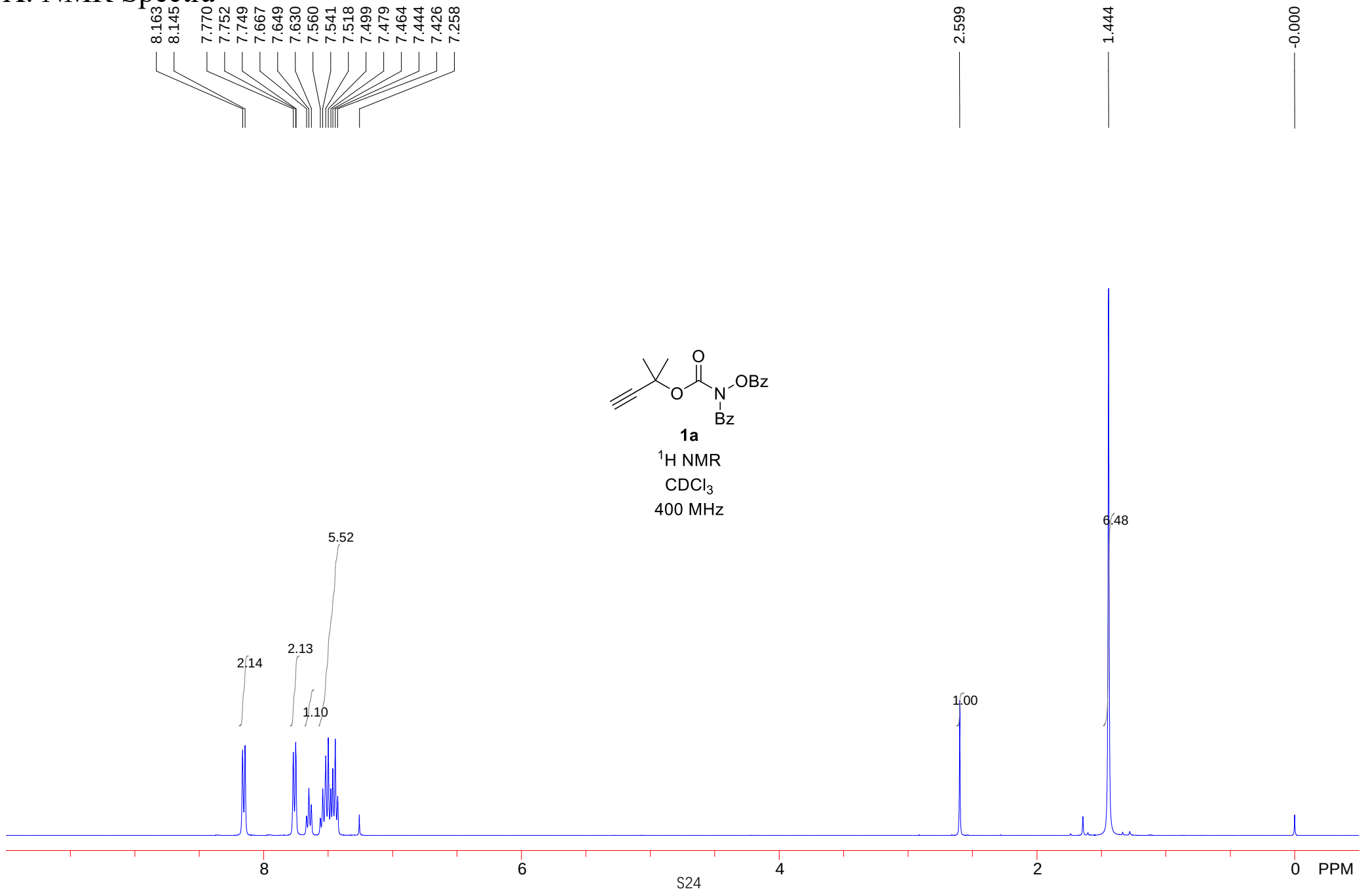
IX. References

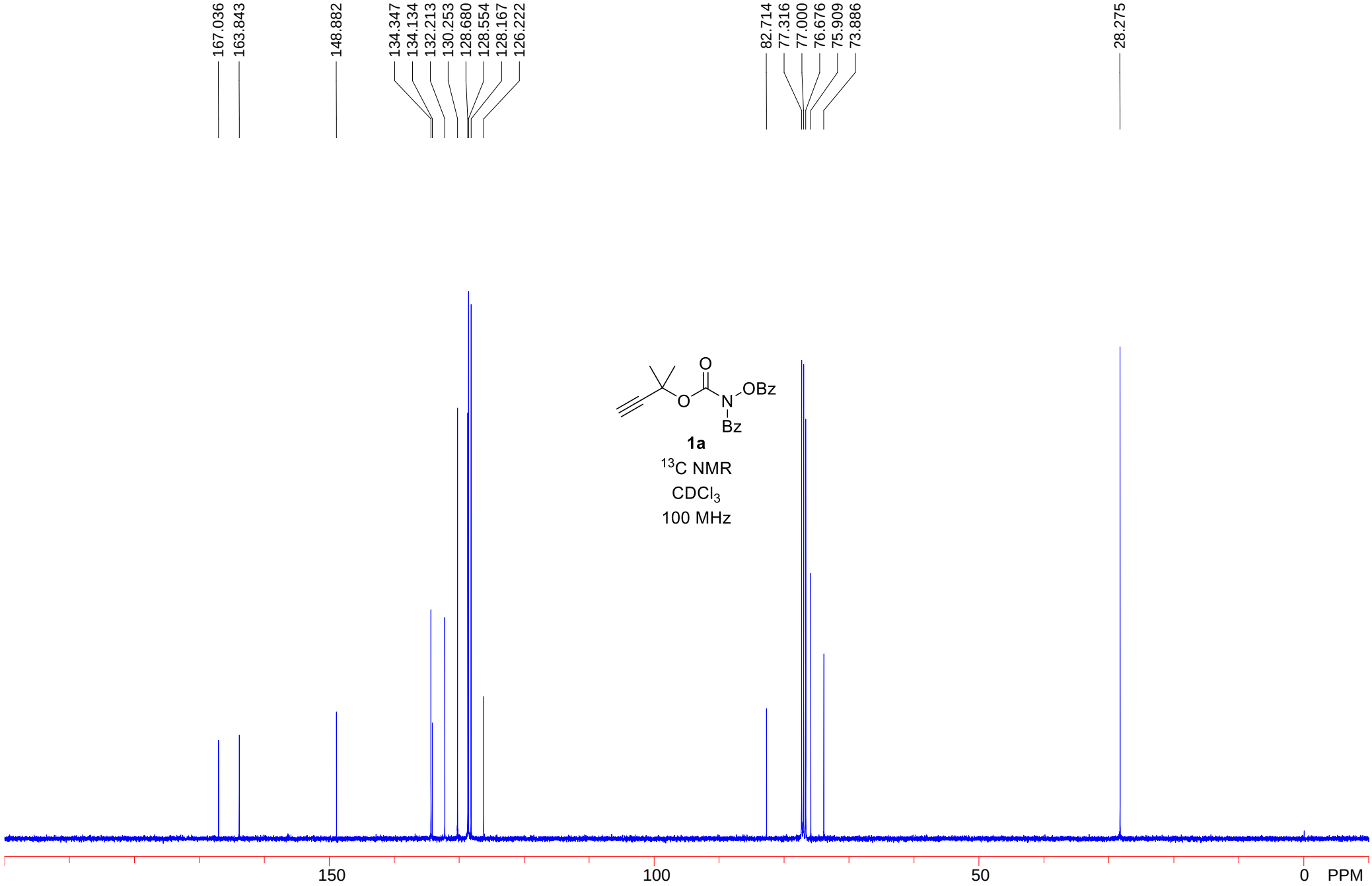
[1] Q. Guo, X. Ren, Z. Lu, *Org. Lett.* **2019**, *21*, 880.

[2] M. A. Cismesia, T. P. Yoon, *Chem. Sci.* **2015**, *6*, 5426.

[3] A. B. Tamayo, B. D. Alleyne, P. I. Djurovich, S. Lamansky, I. Tsyba, N. N. Ho, R. Bau, M. E. Thompson, *J. Am. Chem. Soc.* **2003**, *125*, 7377.

X. NMR Spectra

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76.676
75.909
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N
OBz
Bz
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CDCl3
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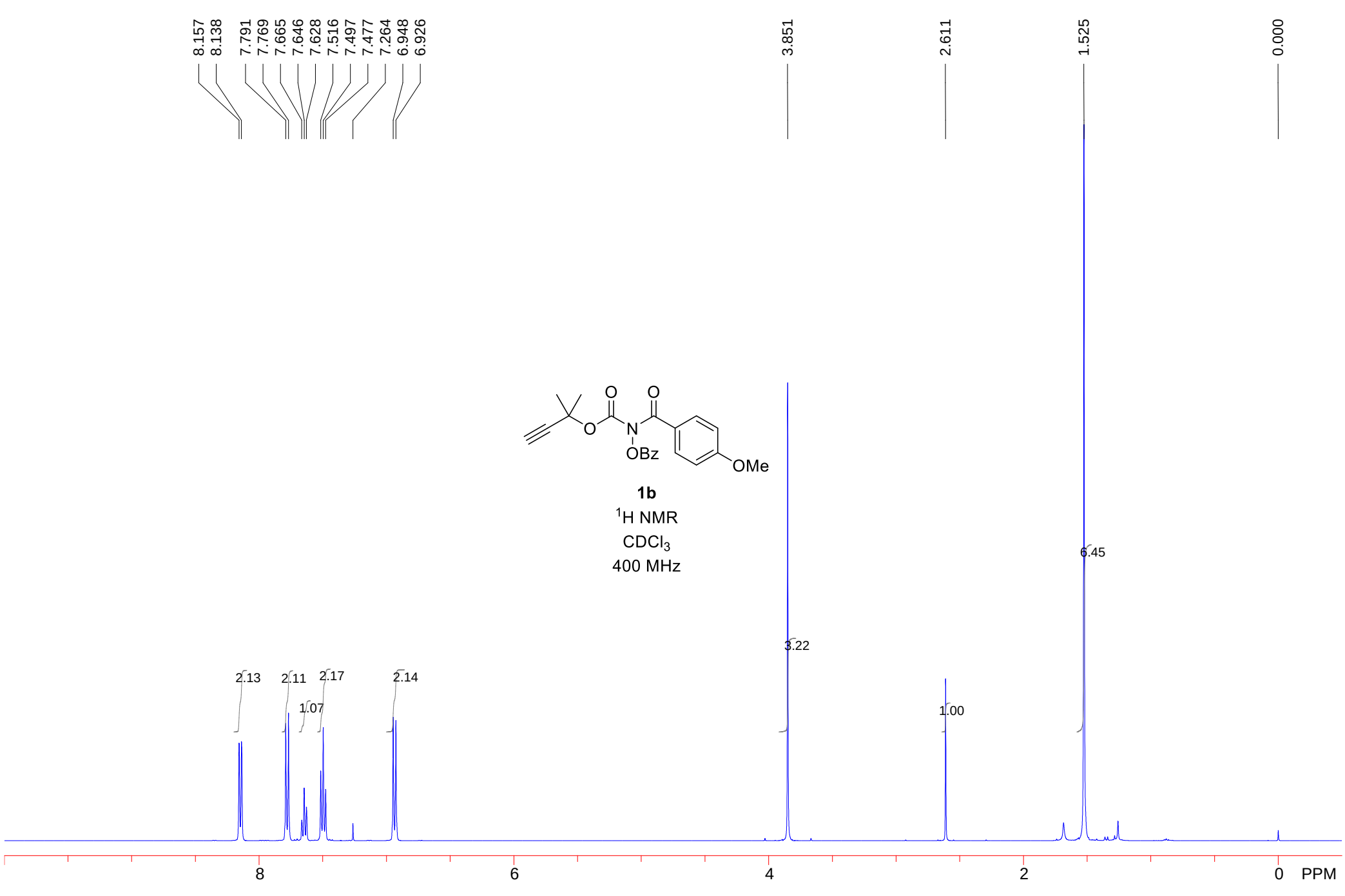
PPM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6.926
3.851
2.611
1.525
0.000
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O
O
N
OBz
OMe
1b
1H NMR
CDCl3
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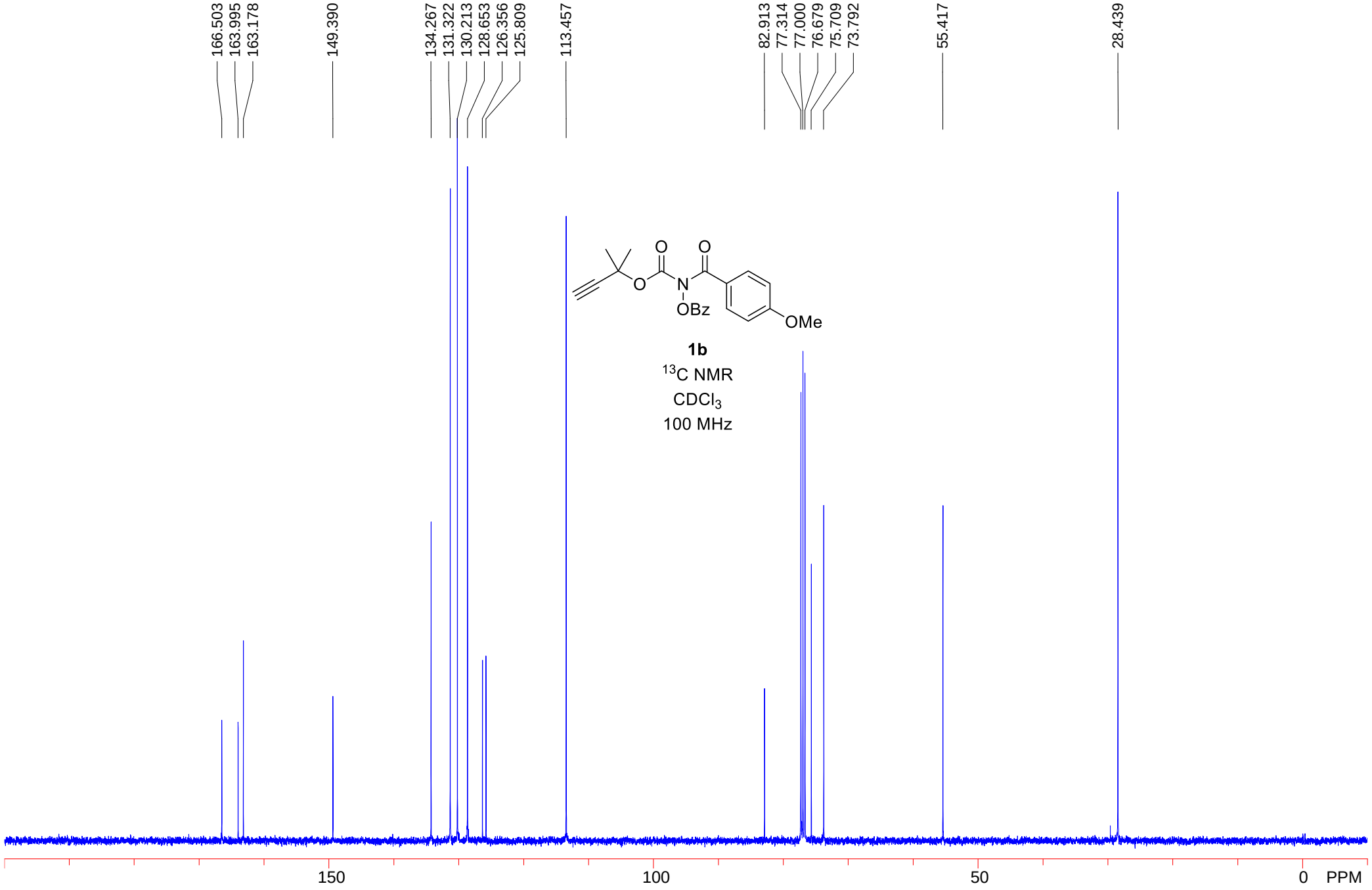
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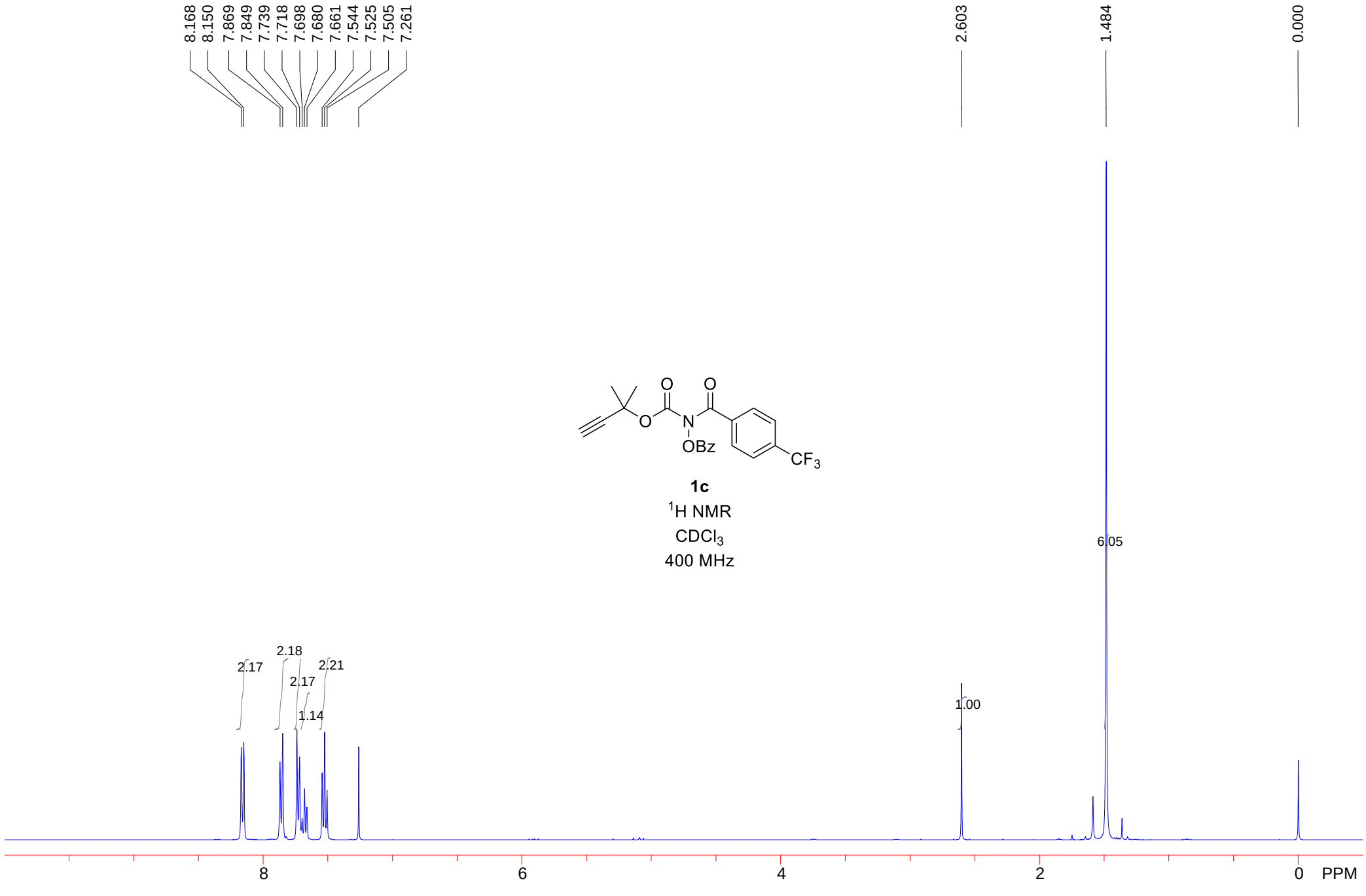
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CF3
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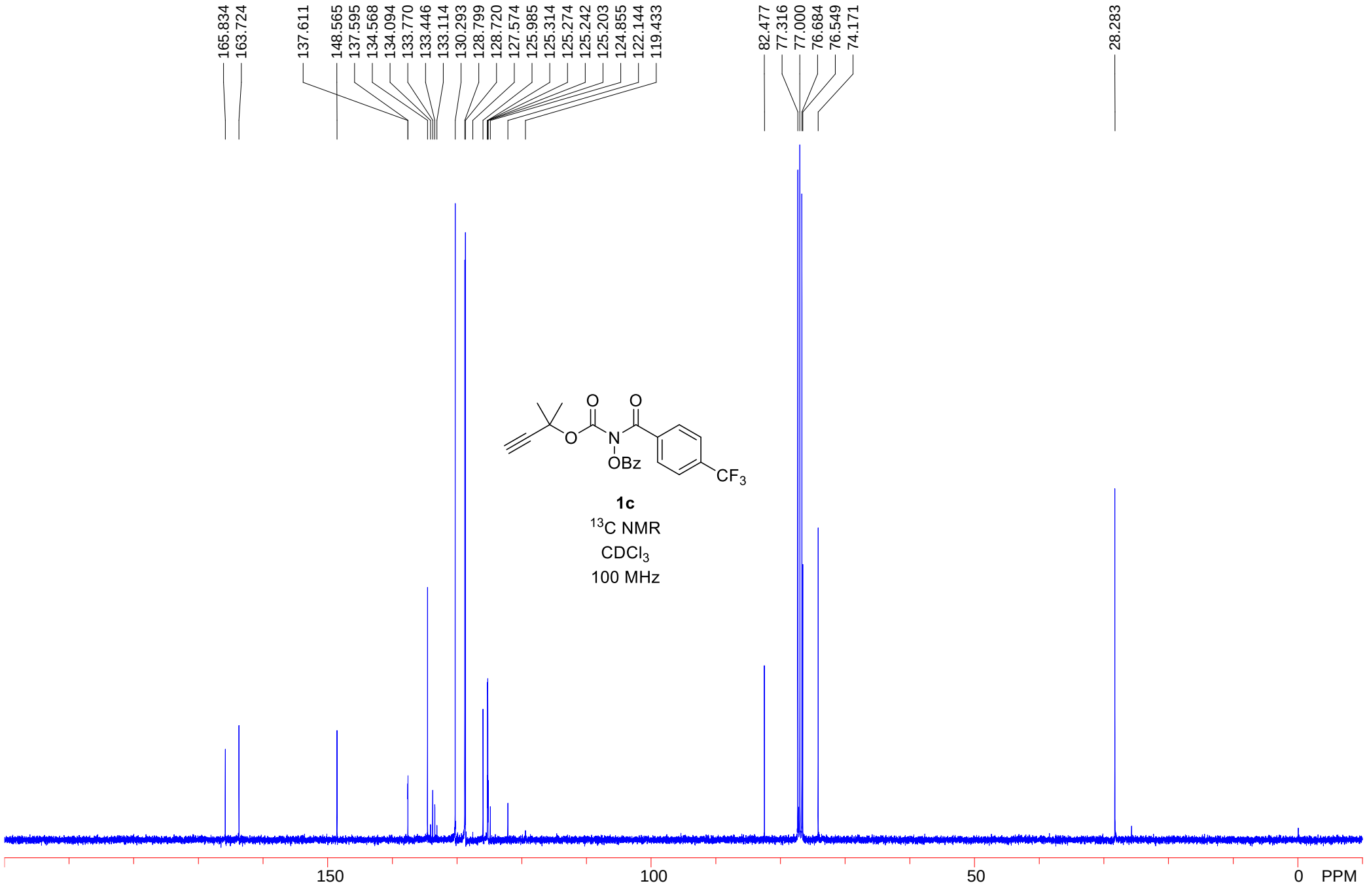
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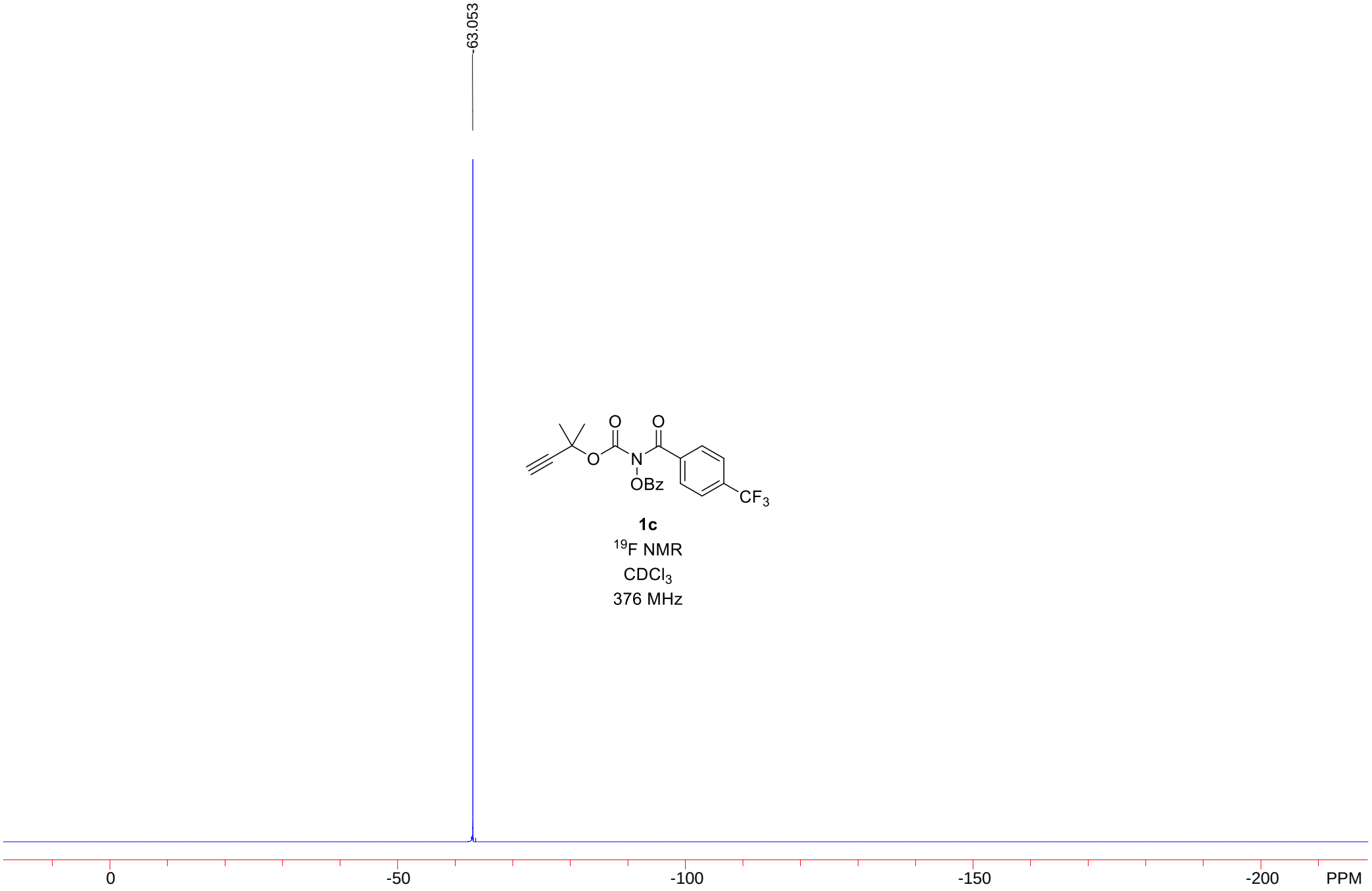
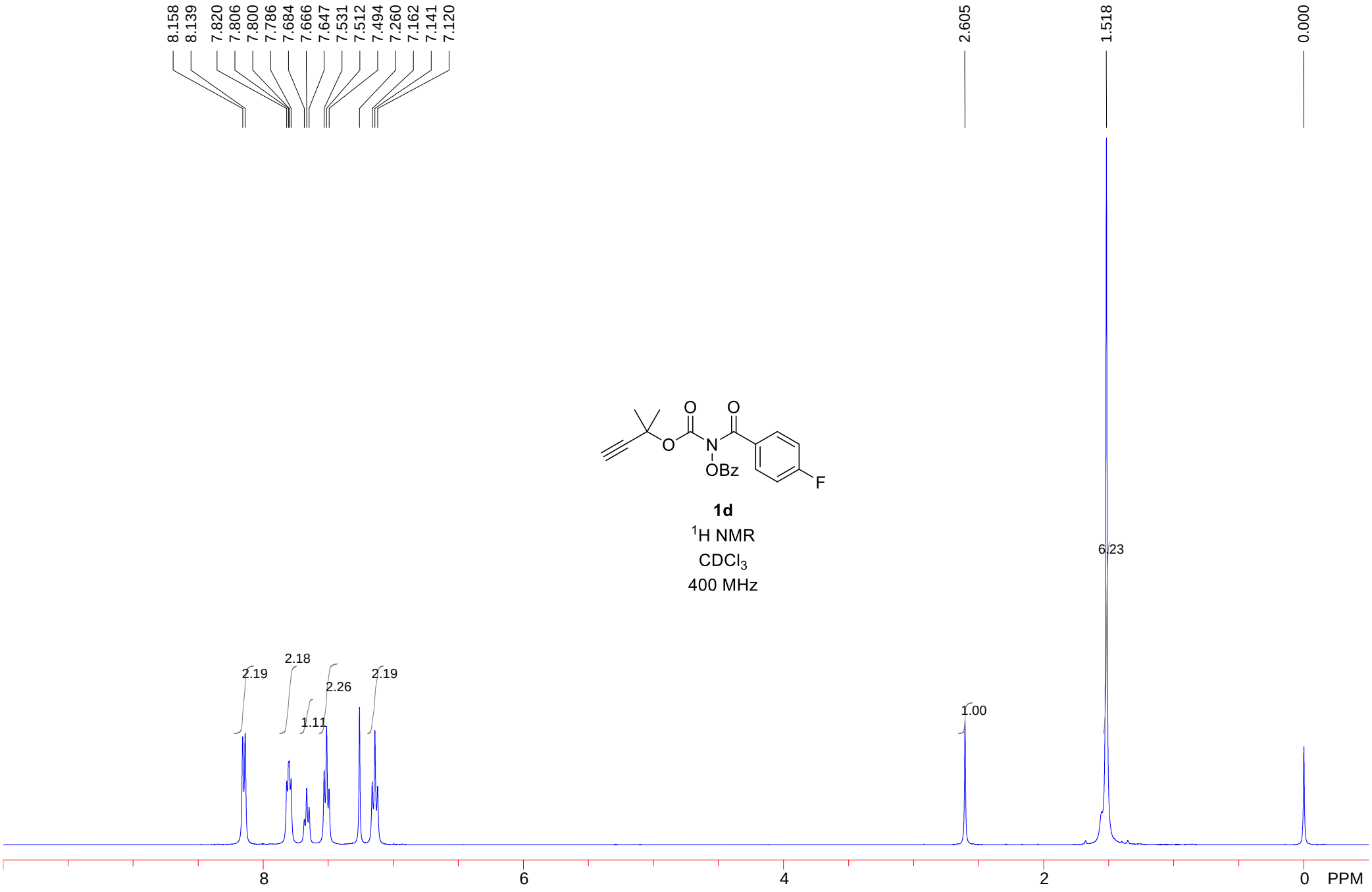


1c

^{19}F NMR

$CDCl_3$

376 MHz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0.000
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O
N
OBz
F
1d
1H NMR
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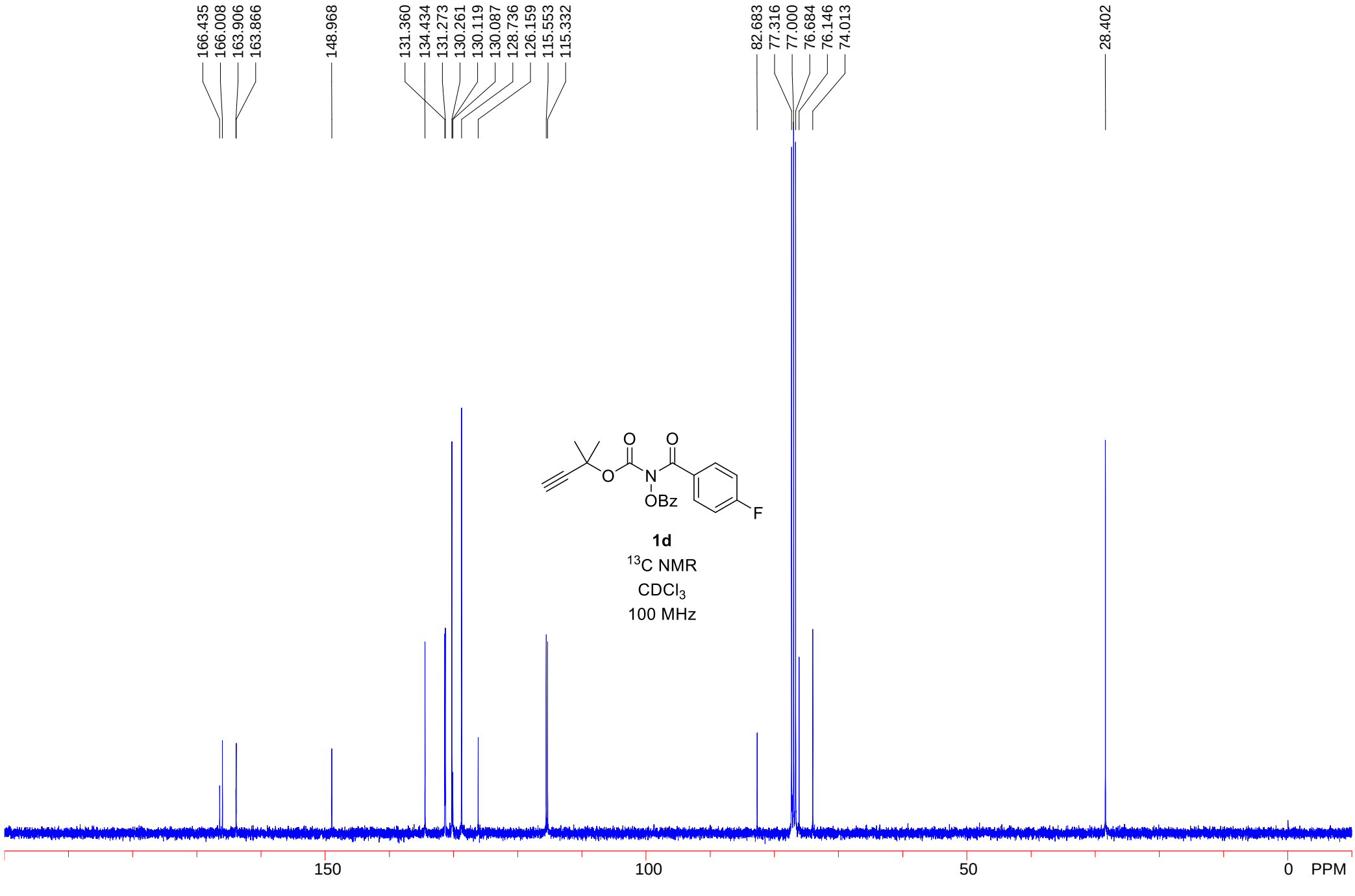
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115.553
115.332
82.683
77.316
77.000
76.684
76.146
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F
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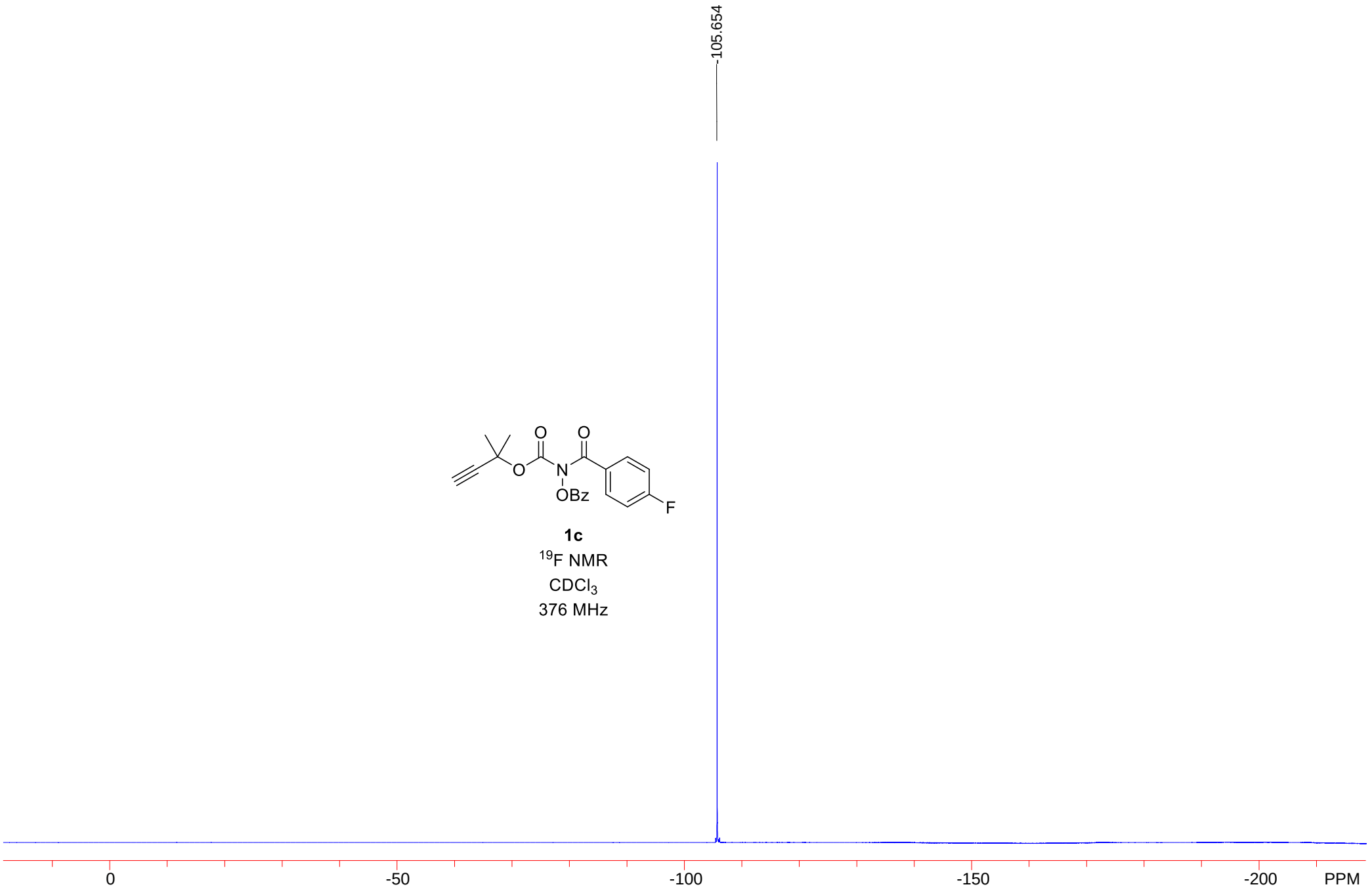
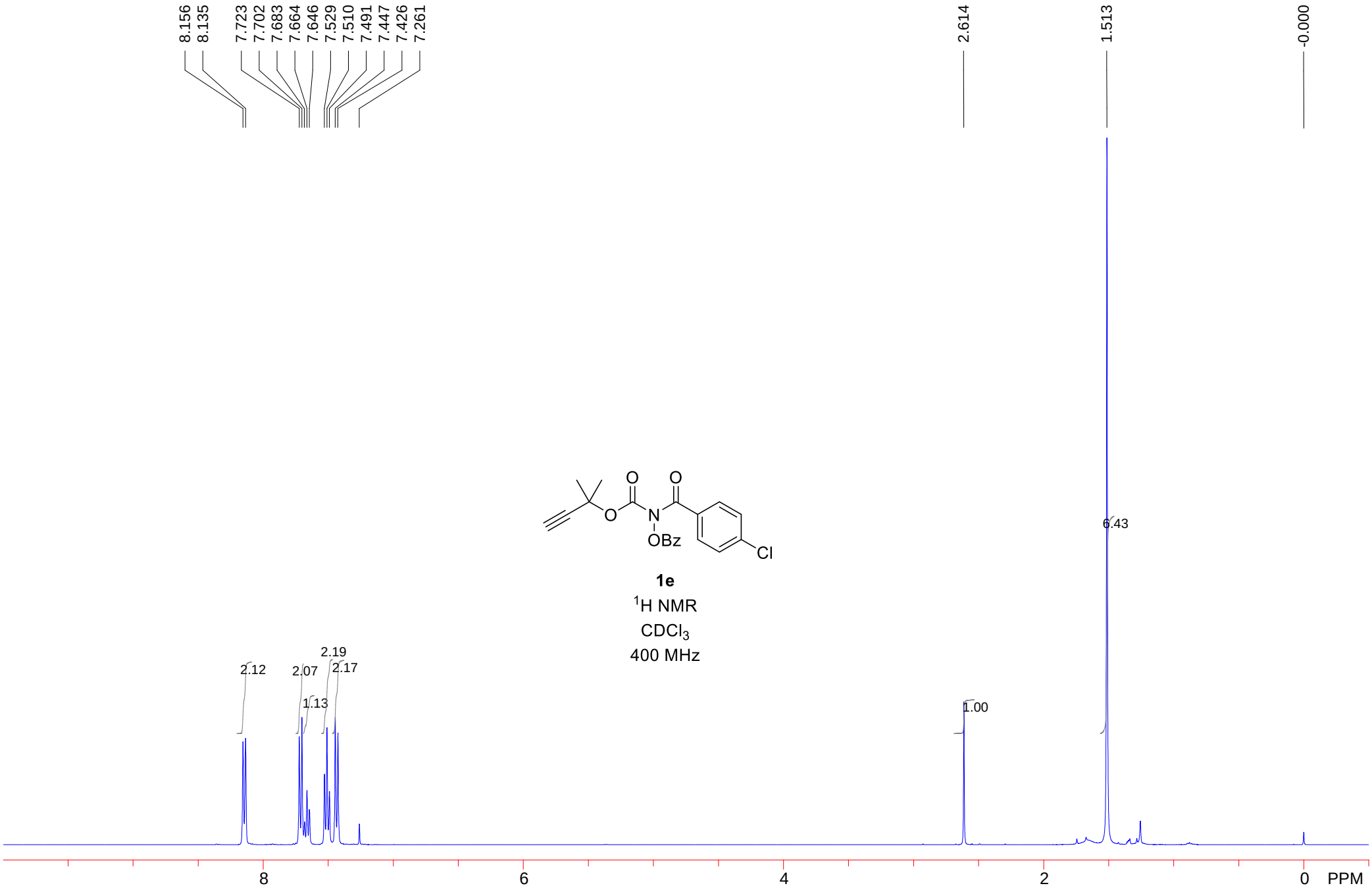


1c

^{19}F NMR

$CDCl_3$

376 MHz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7.261
2.614
1.513
-0.000
O
O
O
N
OBz
Cl
1e
1H NMR
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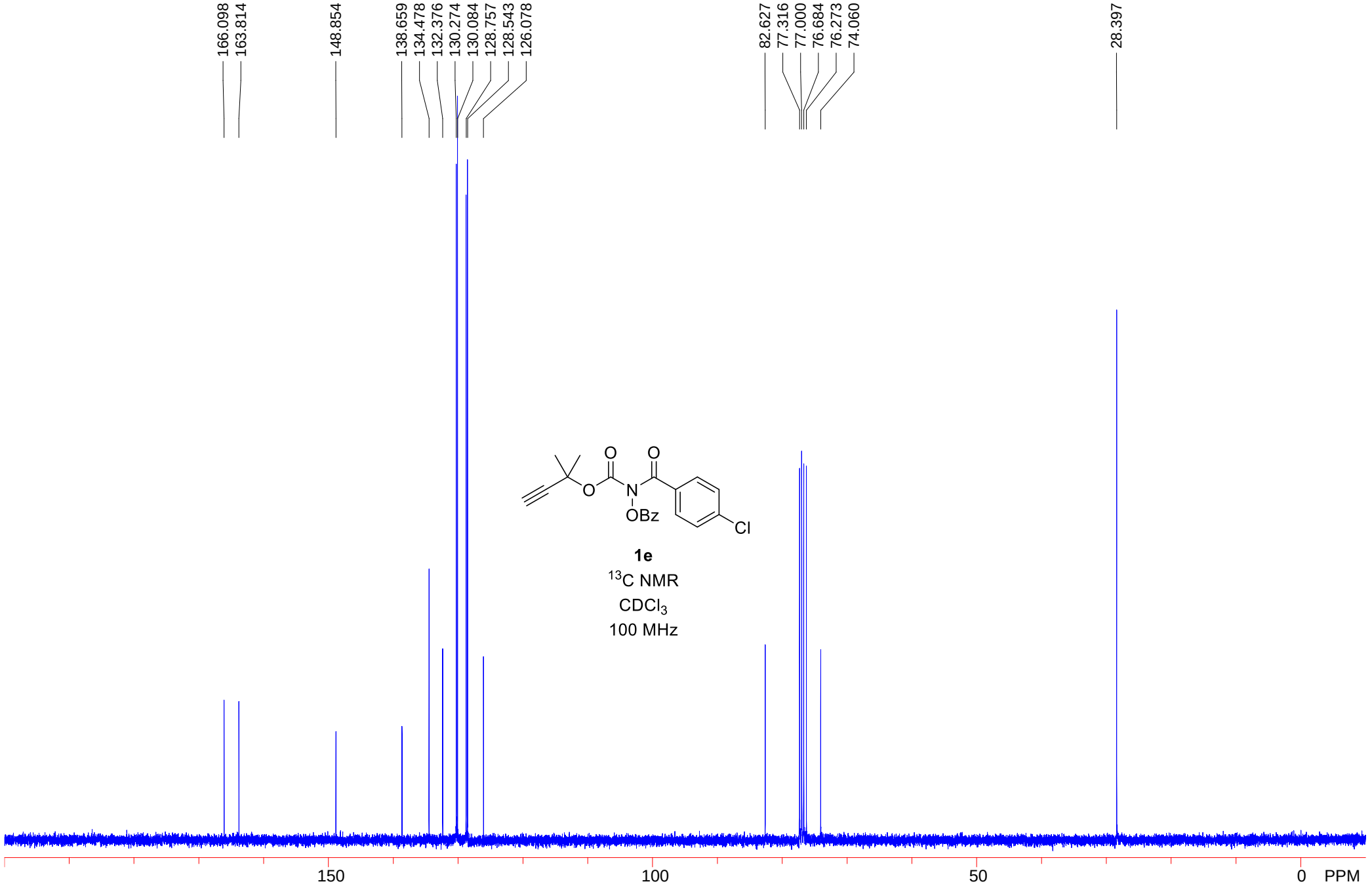
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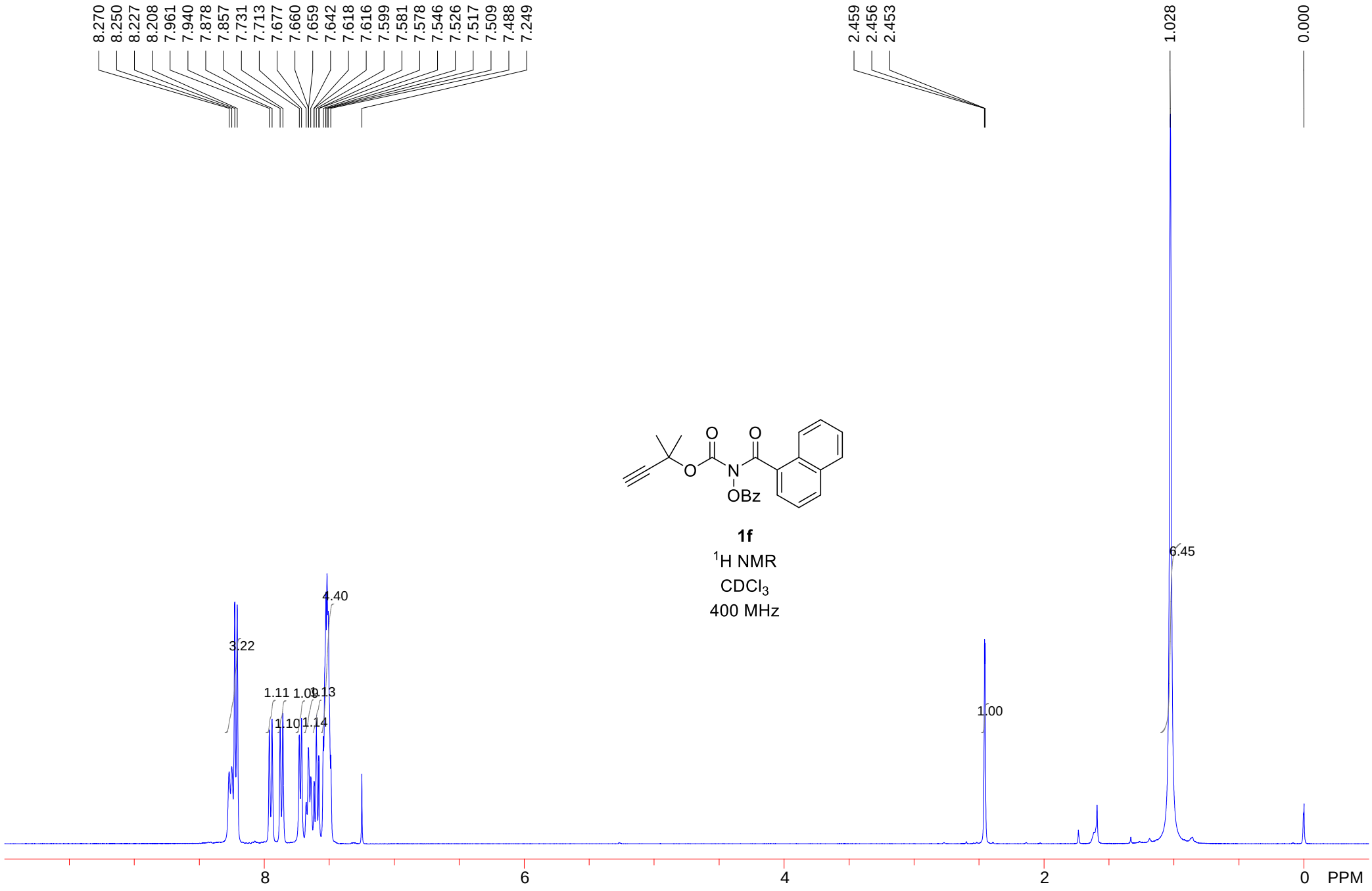
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7.526
7.517
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1f
1H NMR
CDCl3
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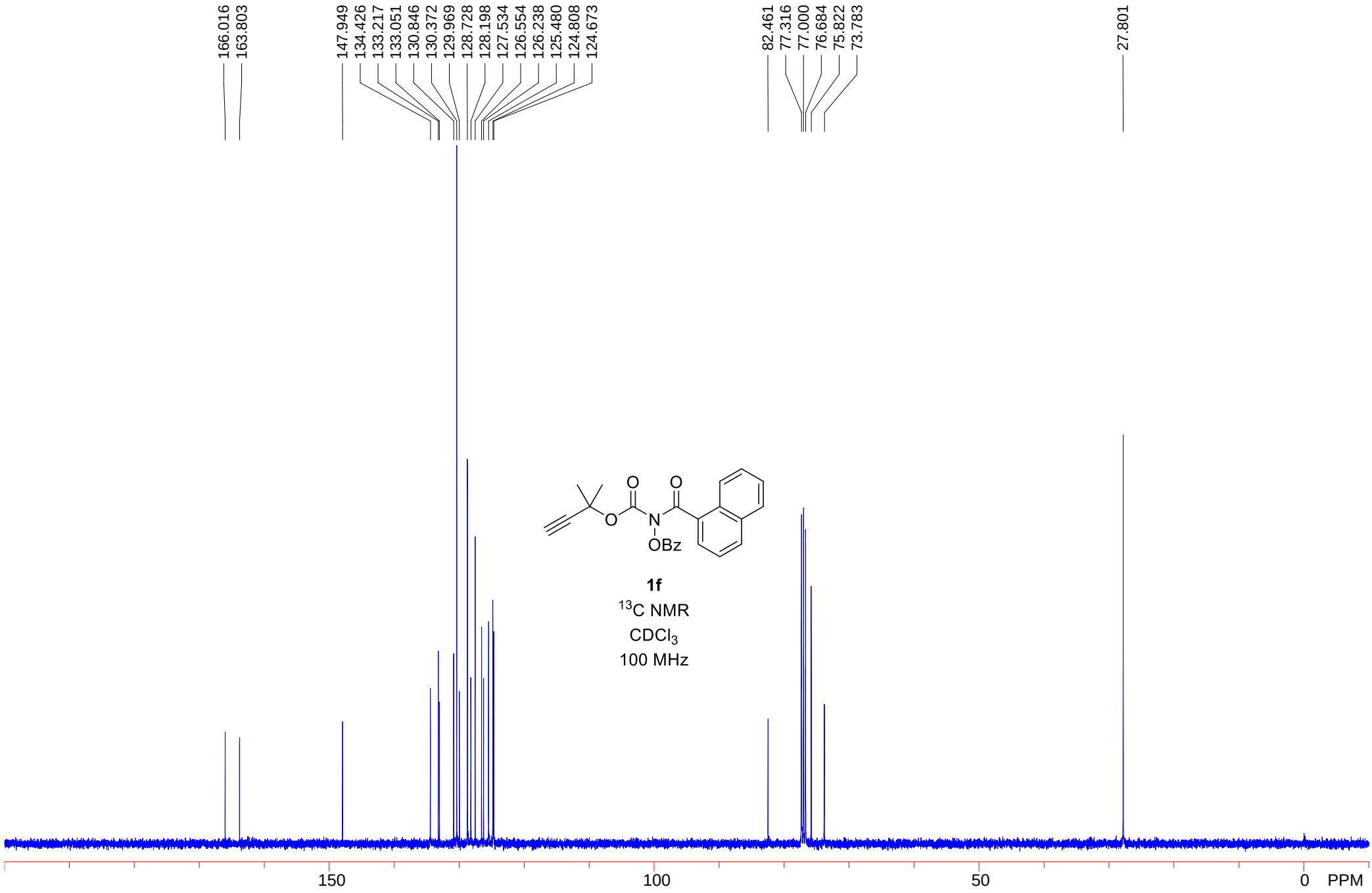
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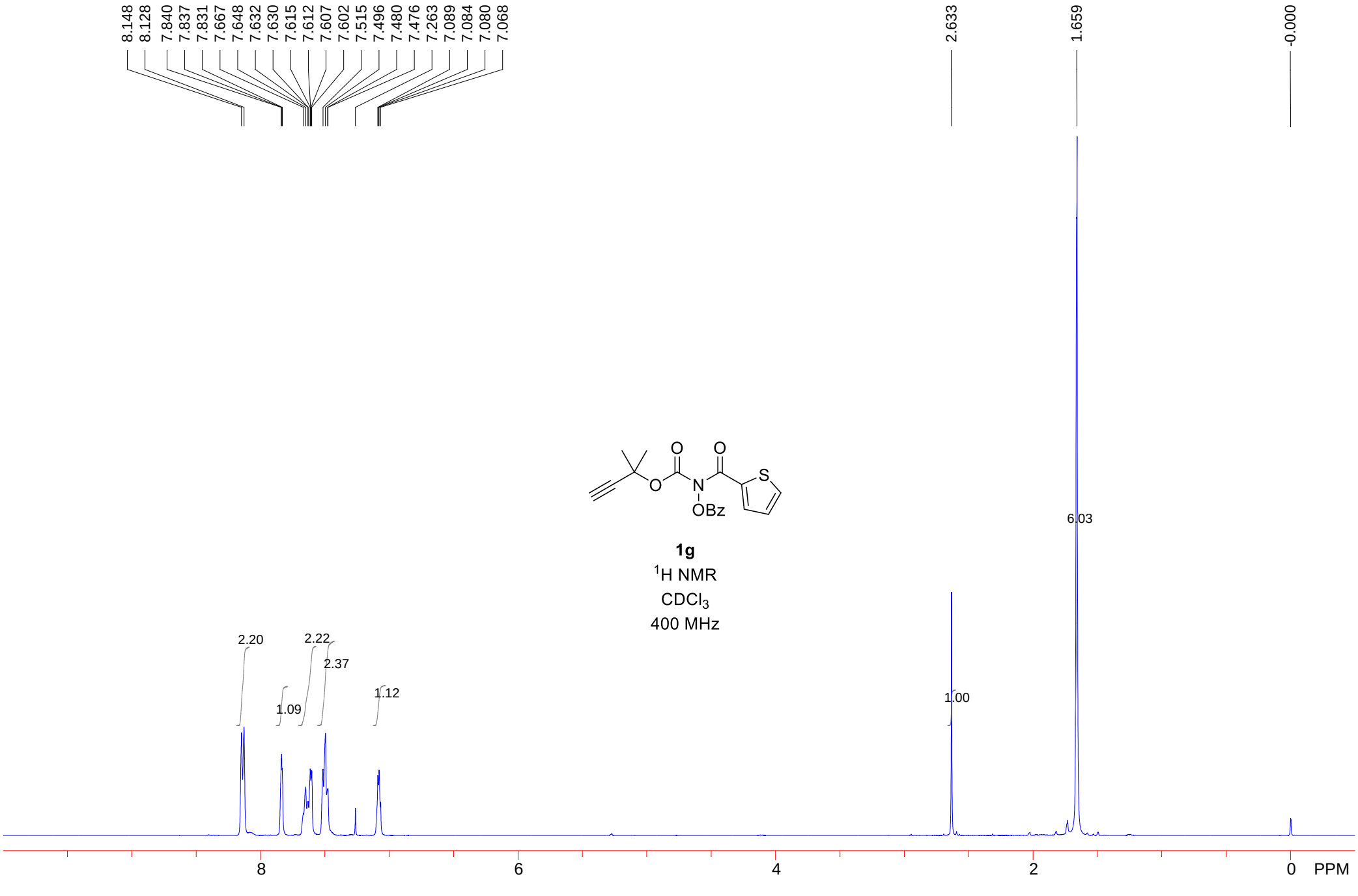
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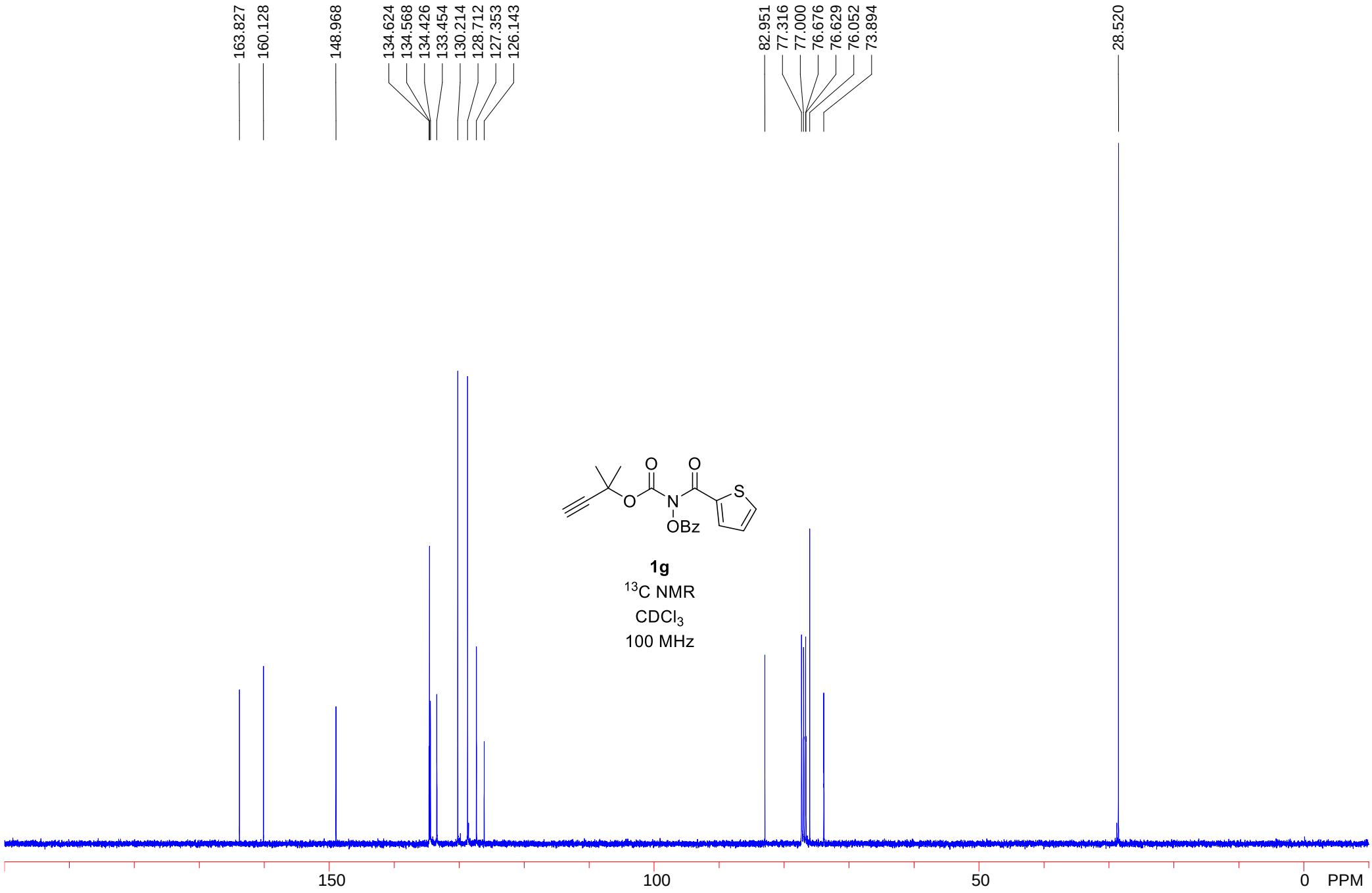
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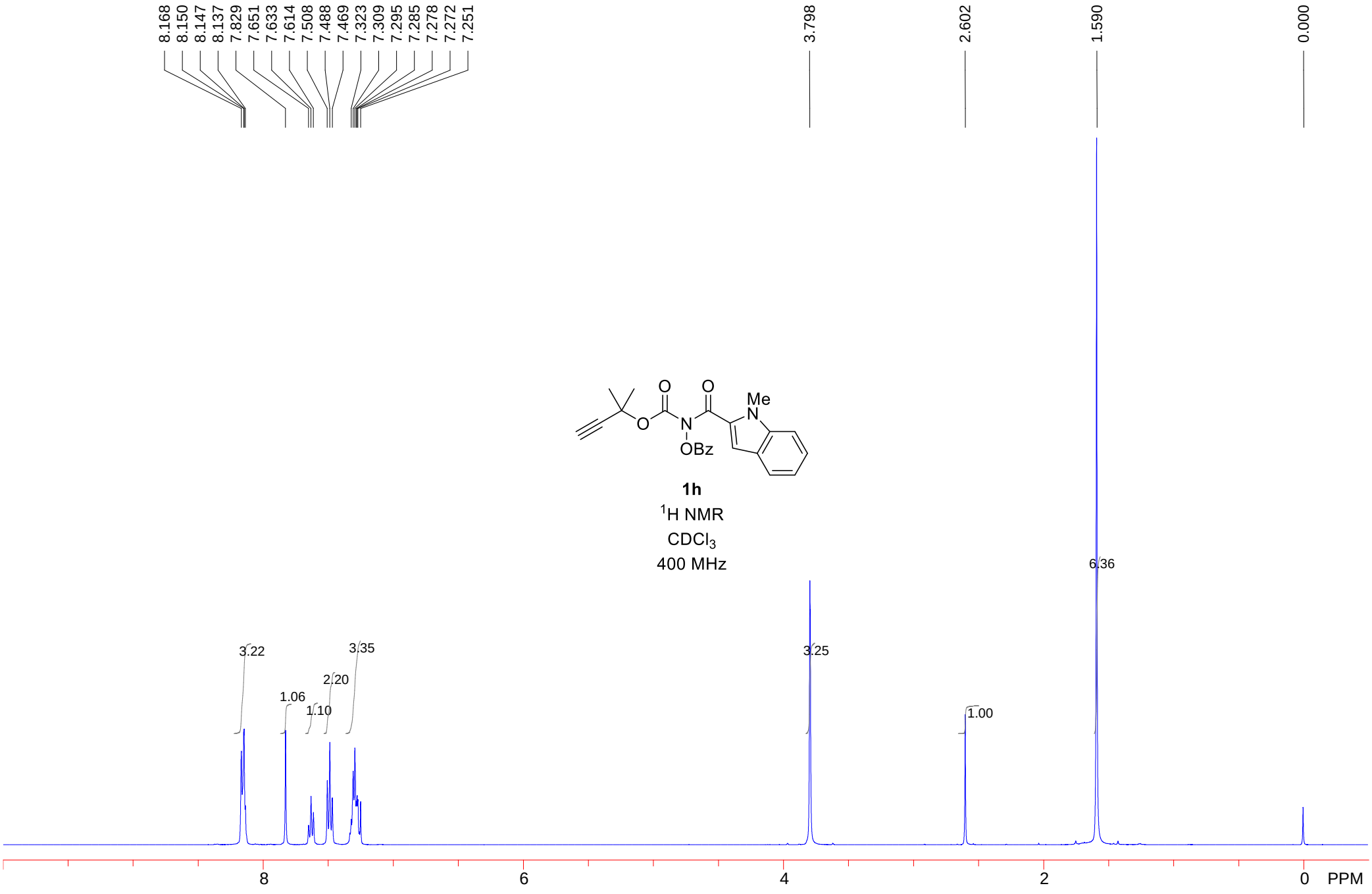
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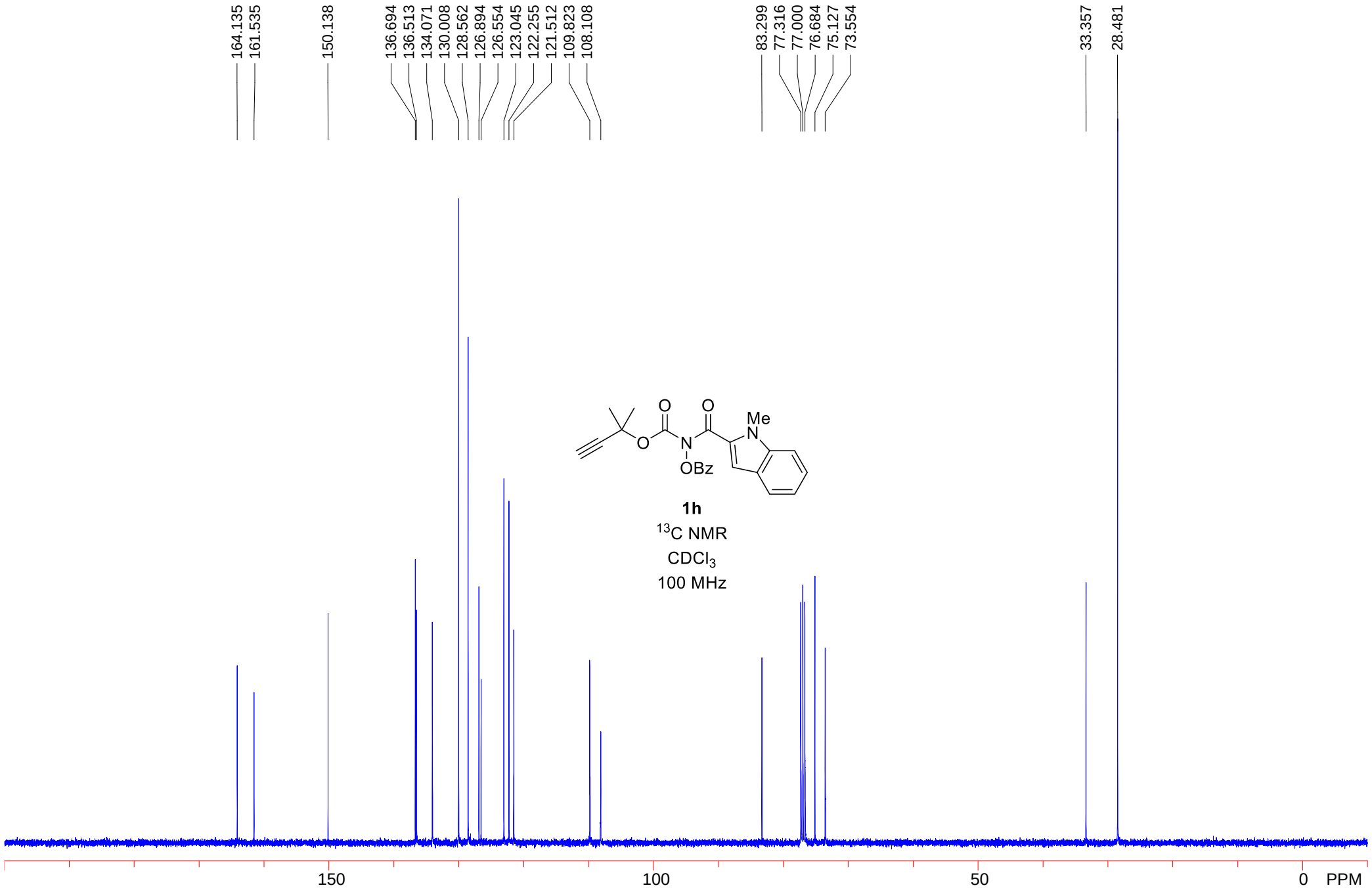
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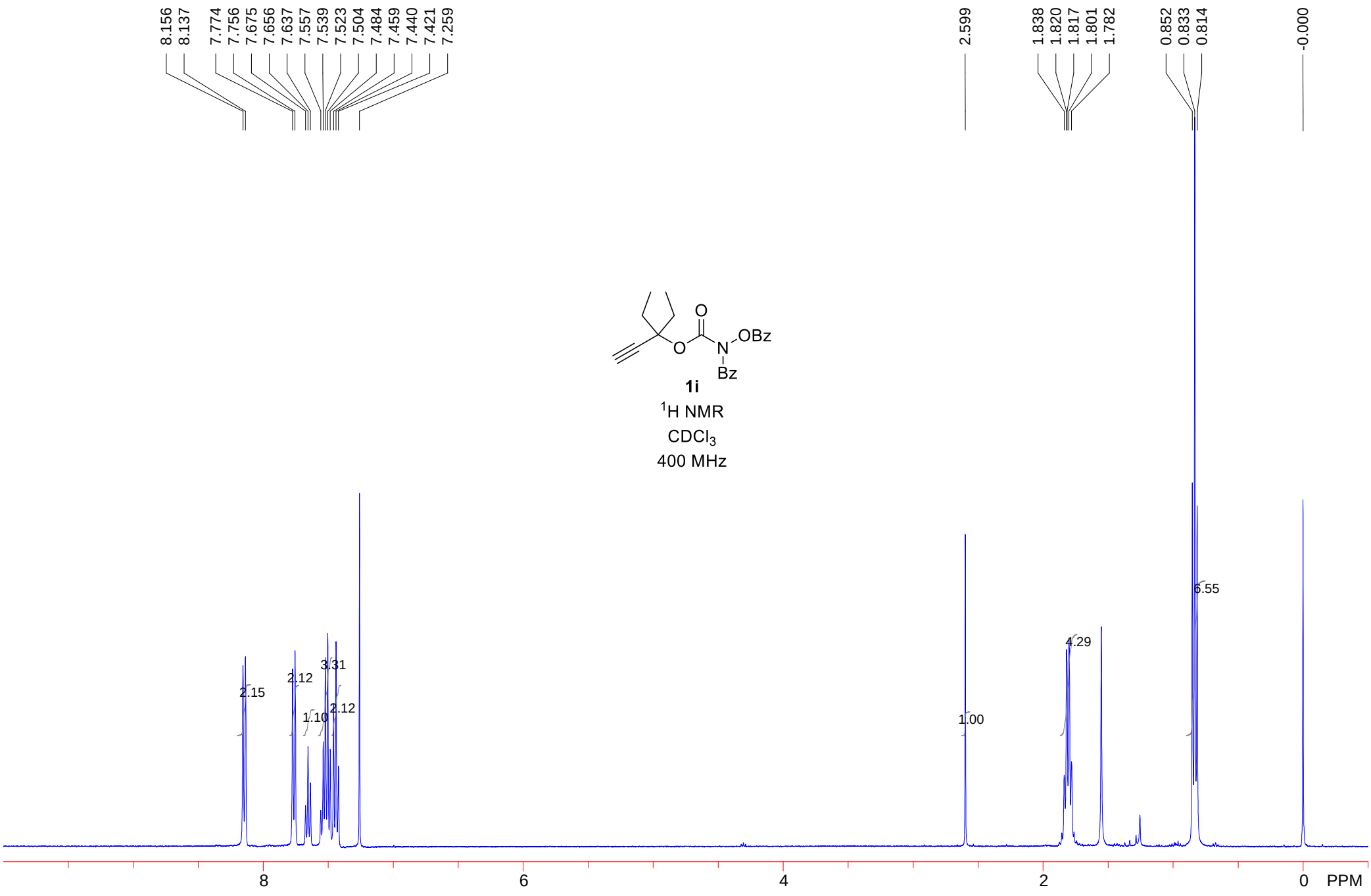
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0.833
0.814
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Bz
1i
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CDCl3
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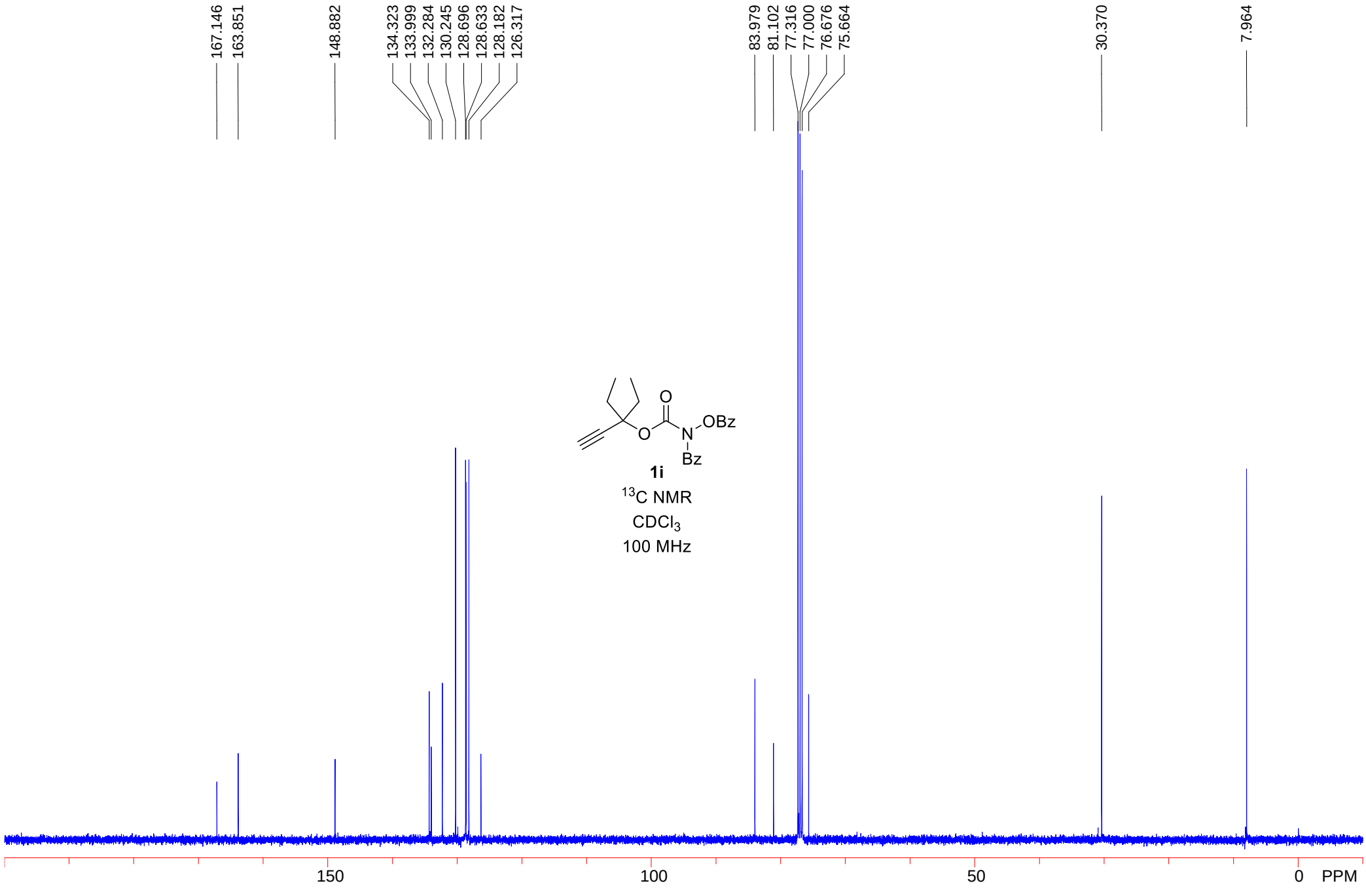
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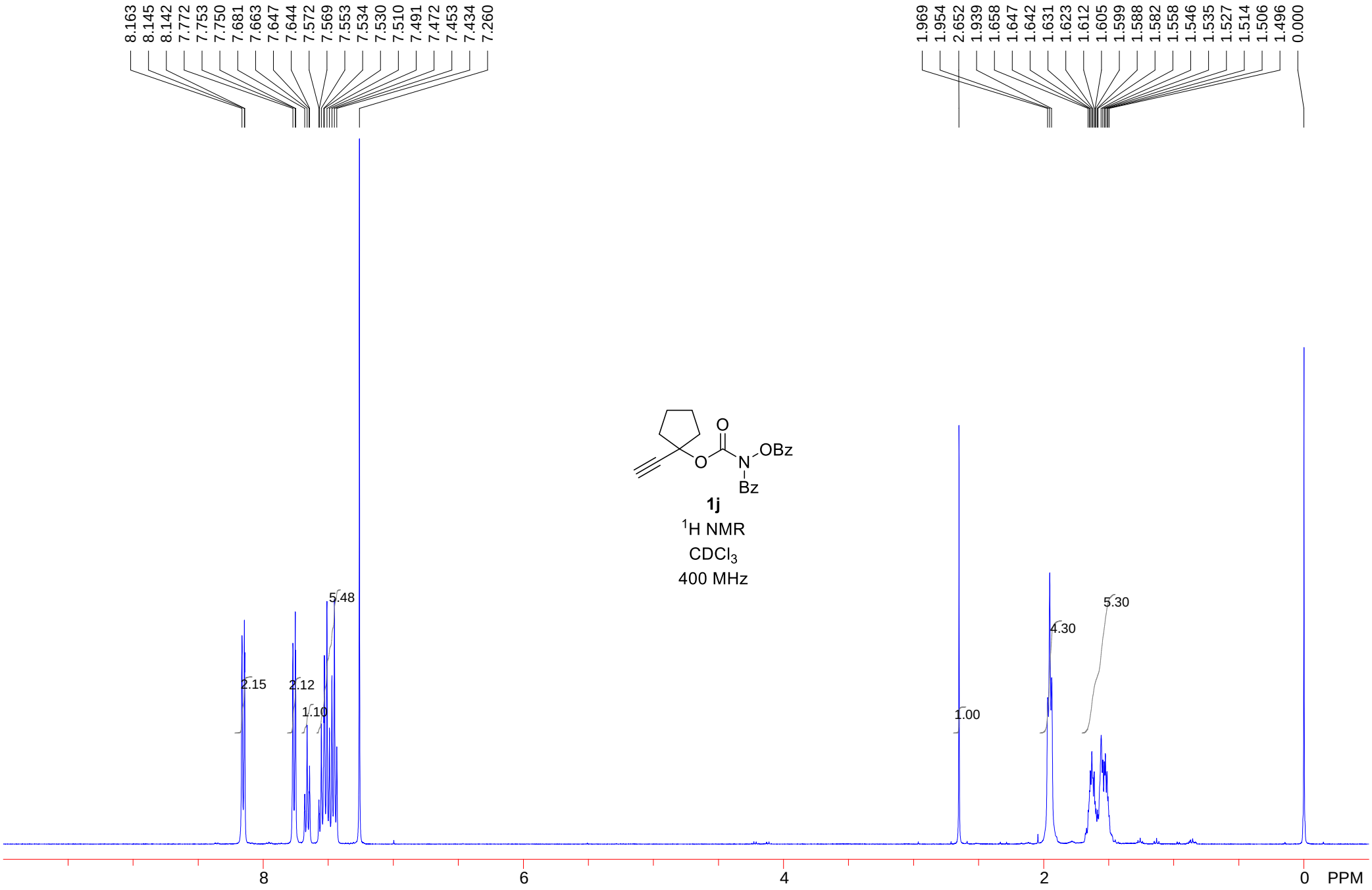
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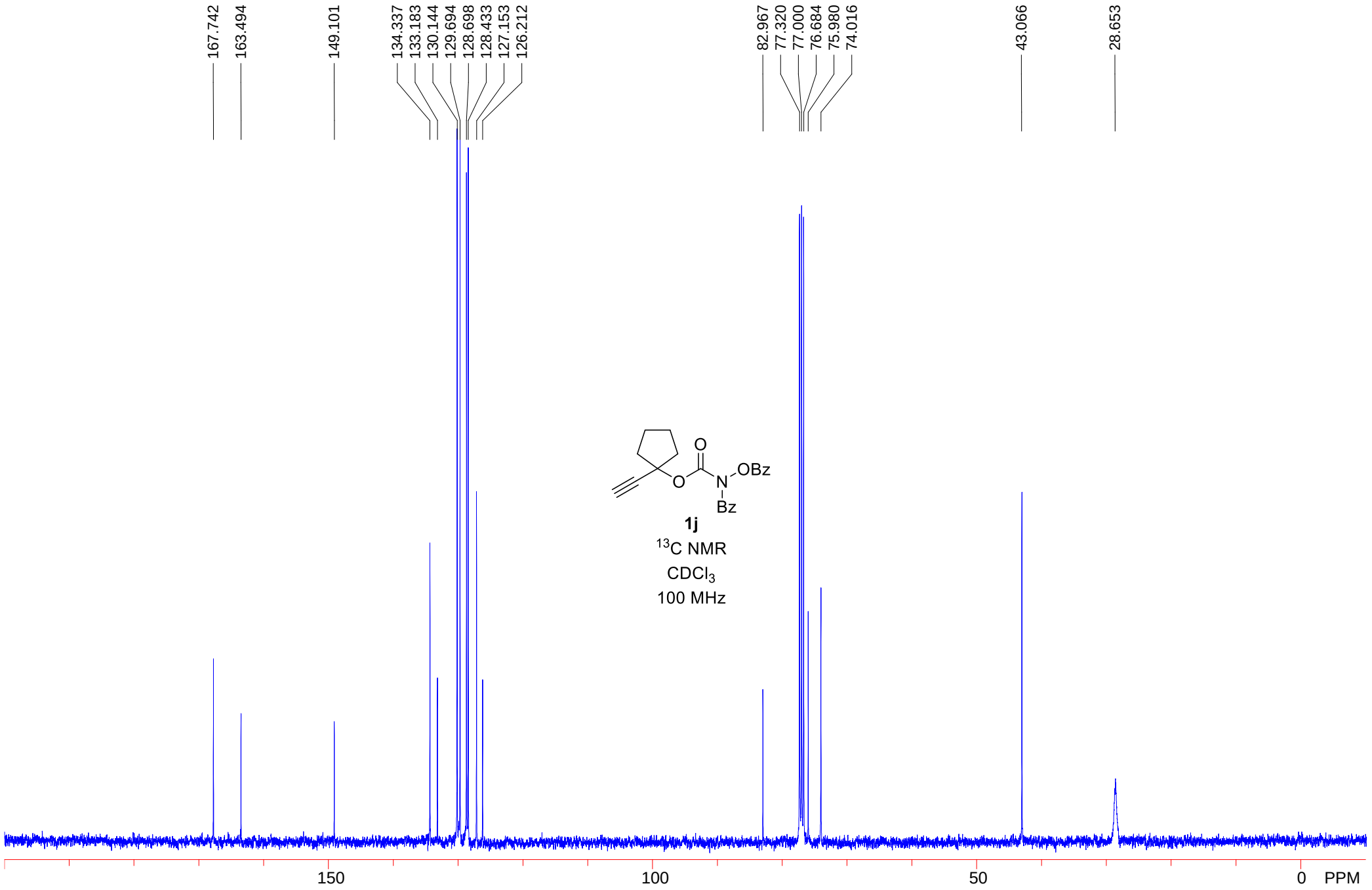
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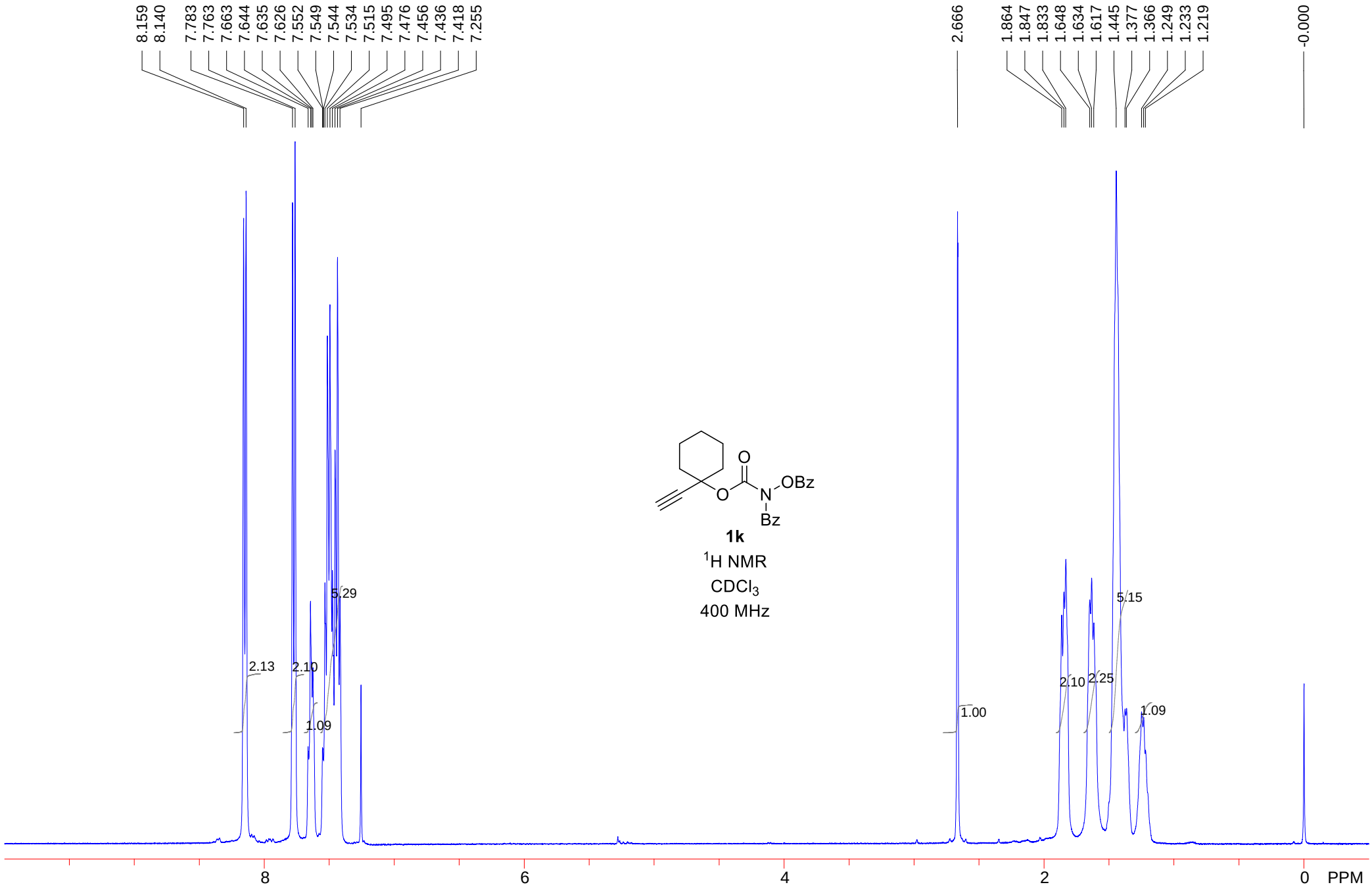
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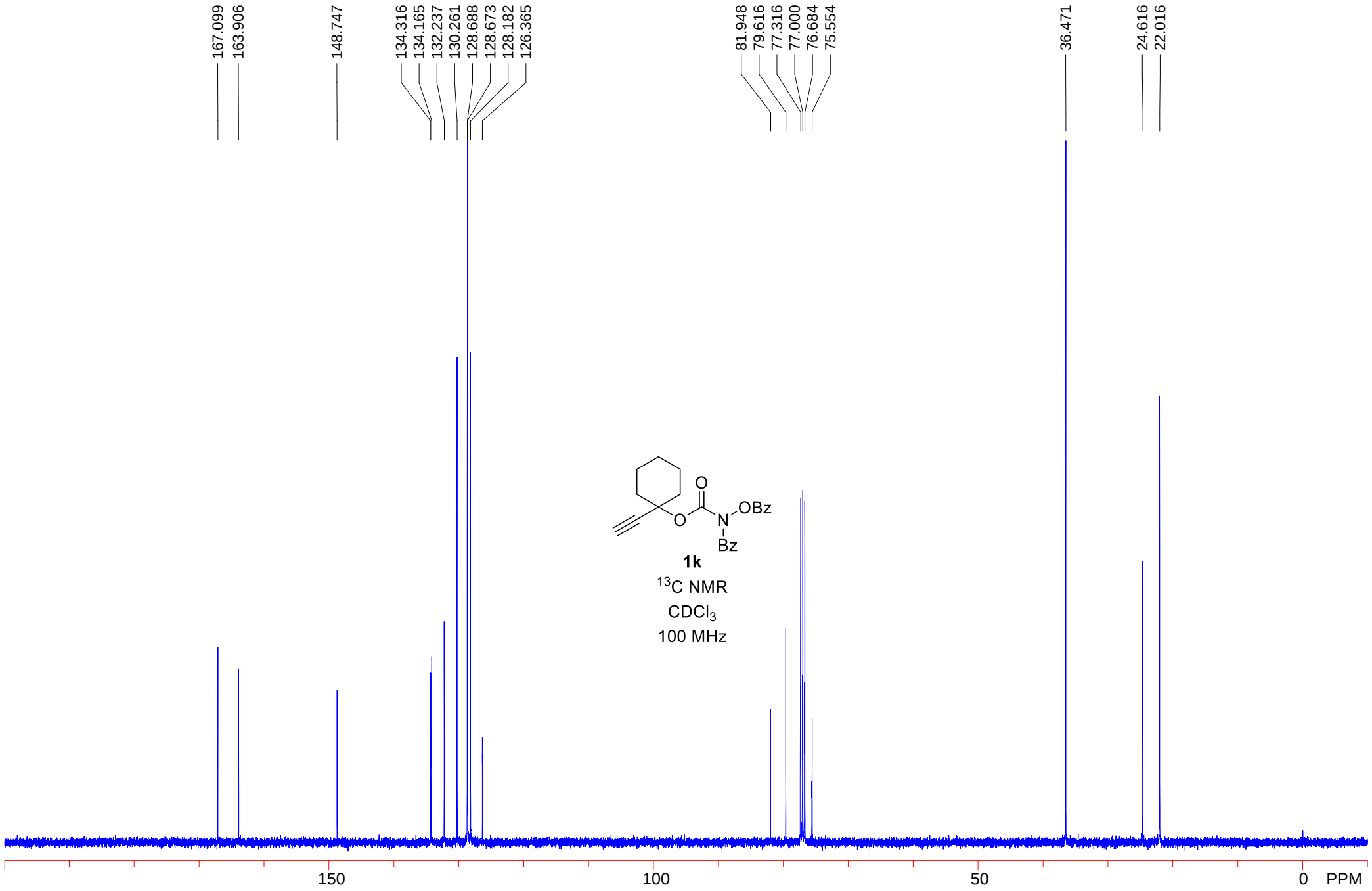
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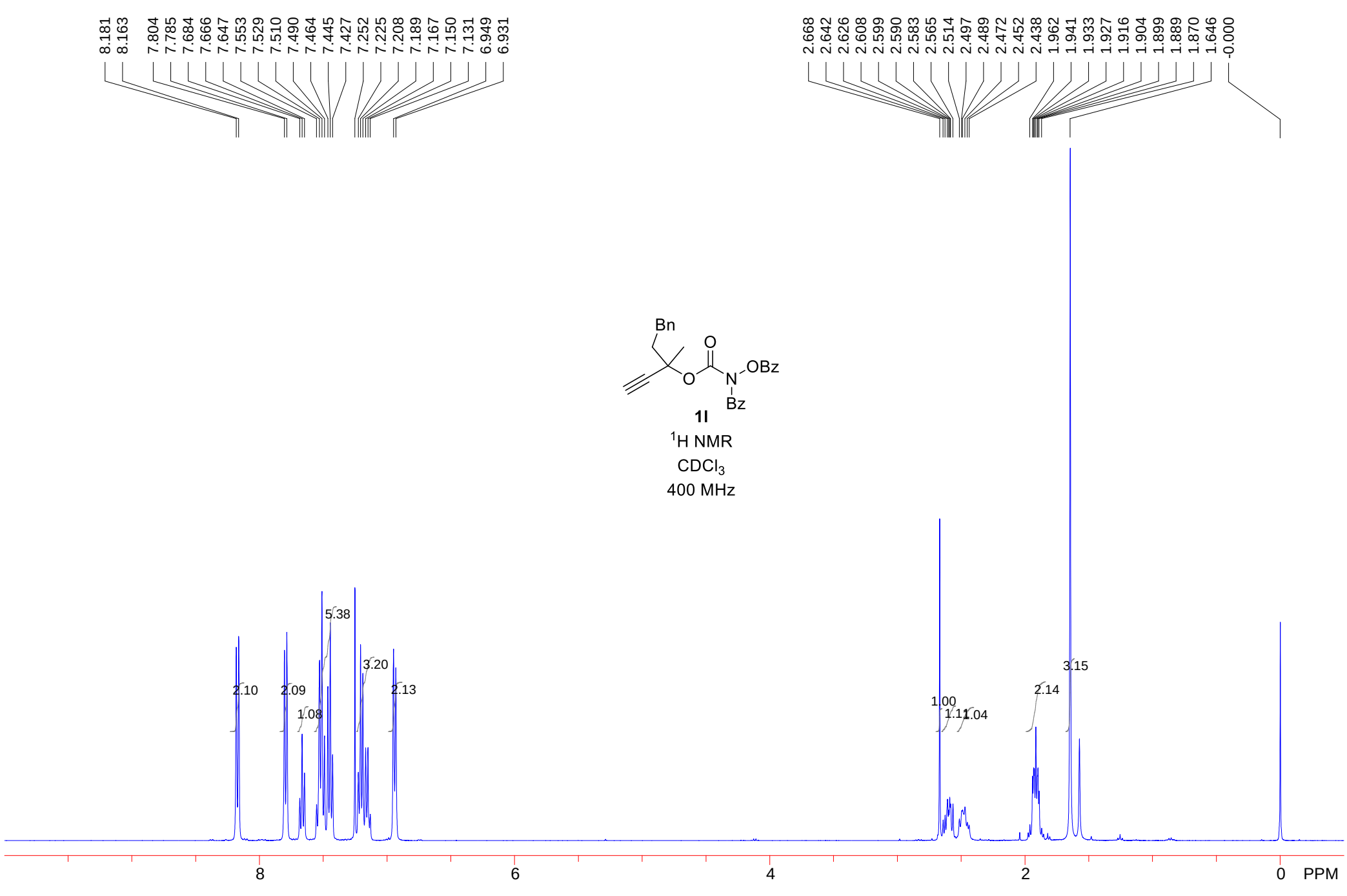
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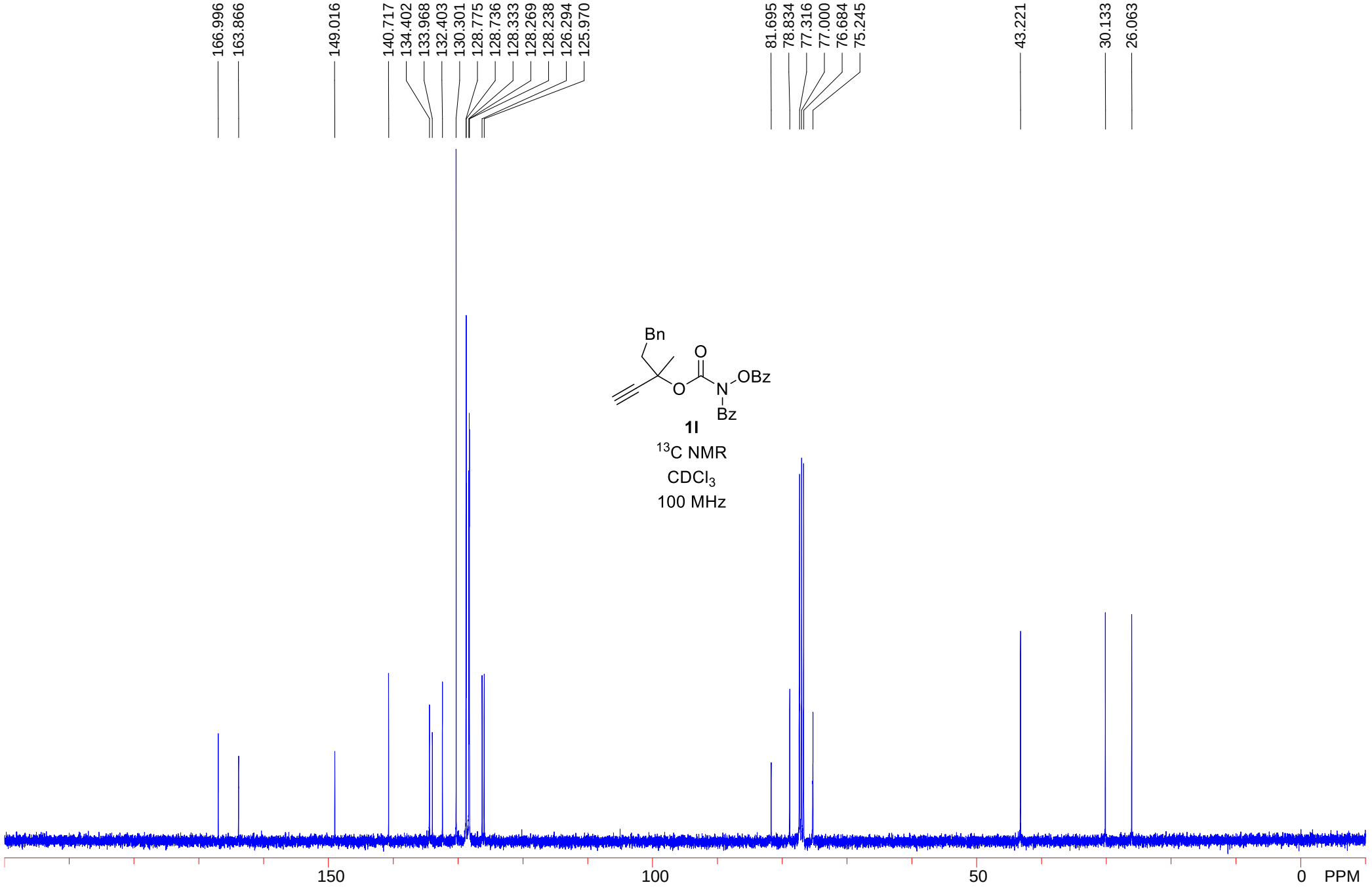
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1l
13C NMR
CDCl3
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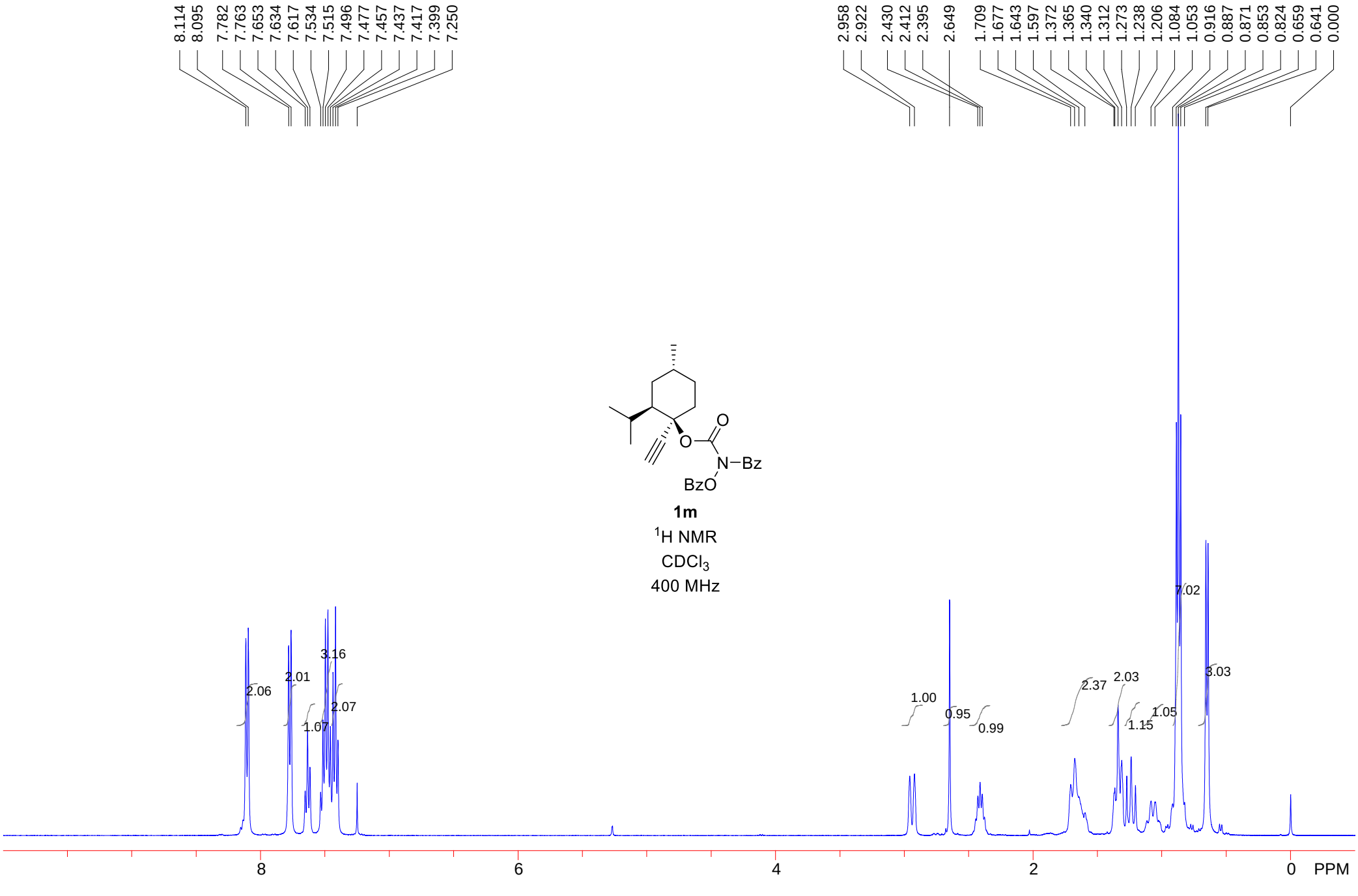
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7.653
7.634
7.617
7.534
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7.496
7.477
7.457
7.437
7.417
7.399
7.250
2.958
2.922
2.430
2.412
2.395
2.649
1.709
1.677
1.643
1.597
1.372
1.365
1.340
1.312
1.273
1.238
1.206
1.084
1.053
0.916
0.887
0.871
0.853
0.824
0.659
0.641
0.000
O
O
N–Bz
BzO
1m
1H NMR
CDCl3
400 MHz
2.06
2.01
1.07
3.16
2.07
1.00
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0.99
2.37
2.03
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1.05
7.02
3.03
8
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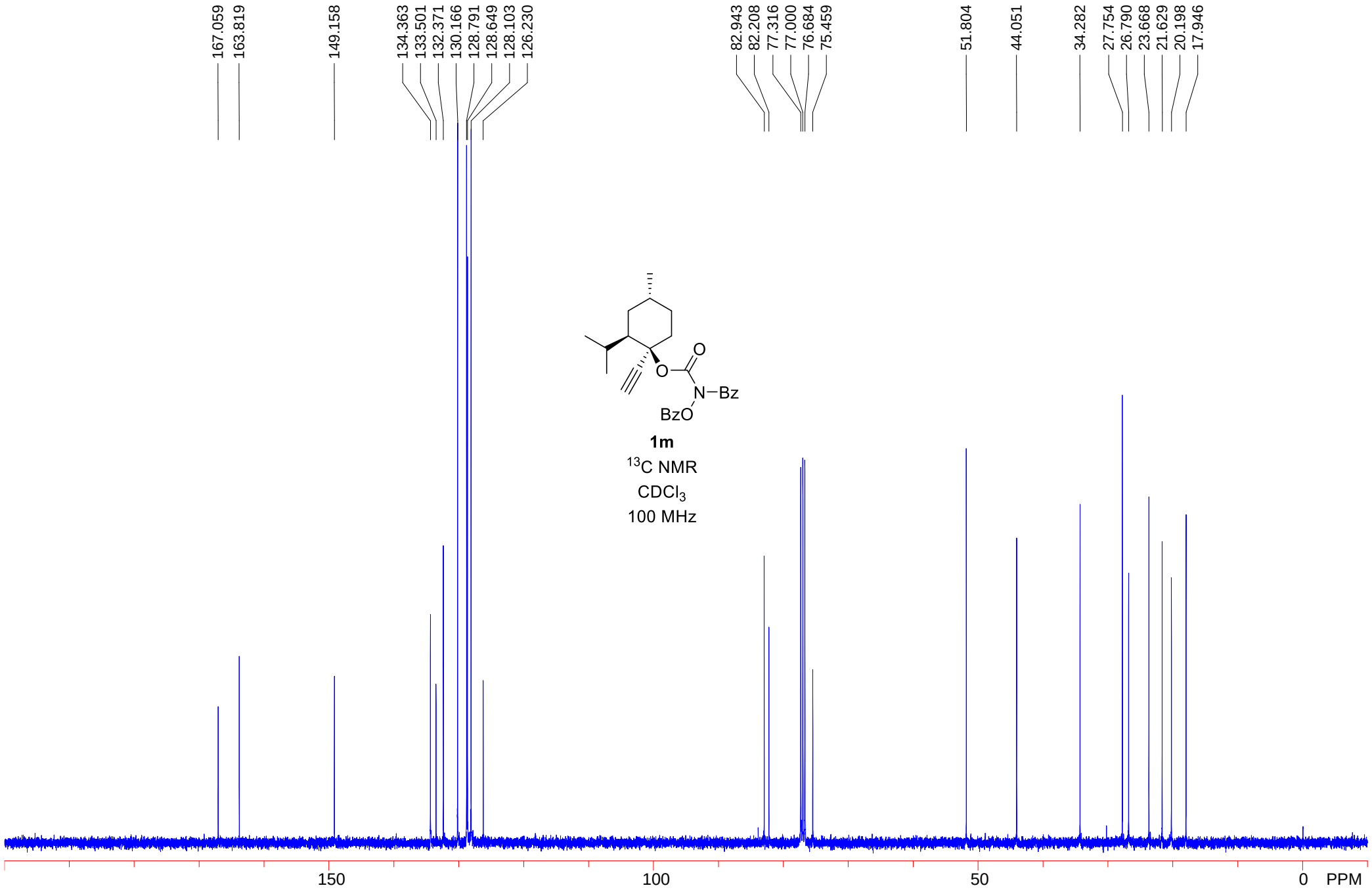
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130.166
128.791
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128.103
126.230
82.943
82.208
77.316
77.000
76.684
75.459
51.804
44.051
34.282
27.754
26.790
23.668
21.629
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O
N−Bz
BzO
1m
13C NMR
CDCl3
100 MHz
150
100
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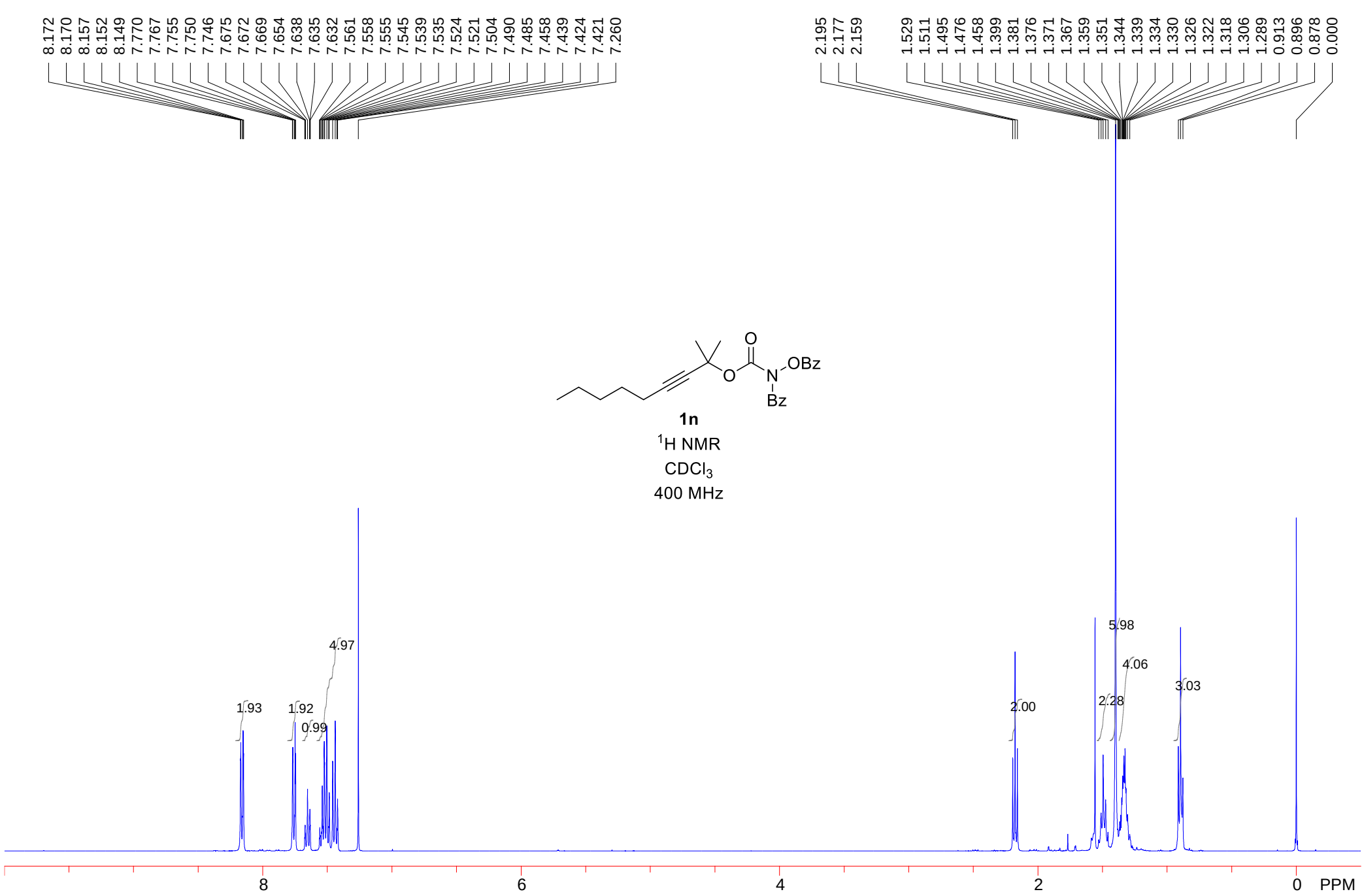
PPM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7.755
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7.746
7.675
7.672
7.669
7.654
7.638
7.635
7.632
7.561
7.558
7.555
7.545
7.539
7.535
7.524
7.521
7.504
7.490
7.485
7.458
7.439
7.424
7.421
7.260
2.195
2.177
2.159
1.529
1.511
1.495
1.476
1.458
1.399
1.381
1.376
1.371
1.367
1.359
1.351
1.344
1.339
1.334
1.330
1.326
1.322
1.318
1.306
1.289
0.913
0.896
0.878
0.000
O
OBz
O
N
Bz
1n
1H NMR
CDCl3
400 MHz
1.93
1.92
0.99
4.97
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4.06
3.03
8
6
4
2
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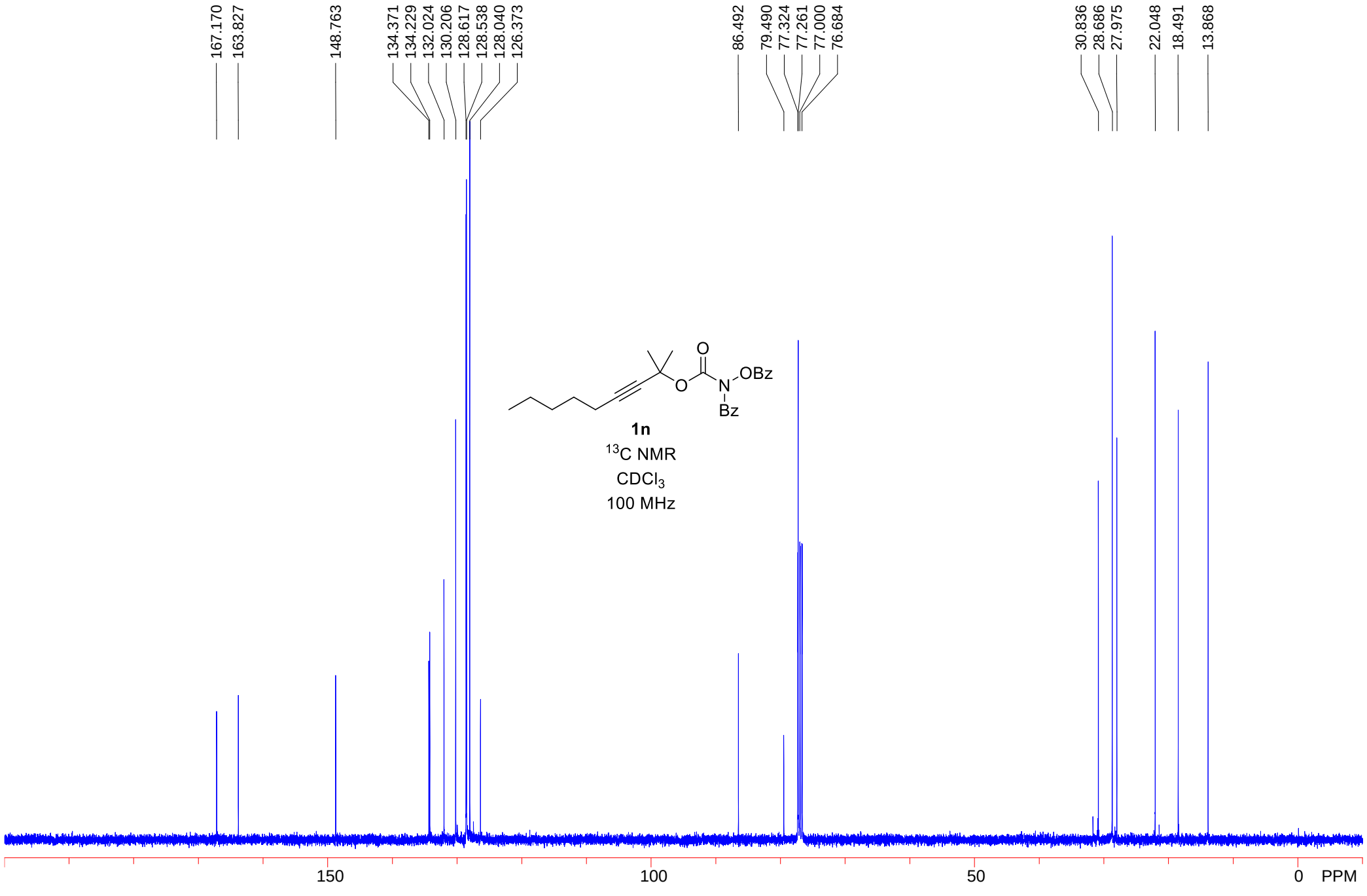
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128.617
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79.490
77.324
77.261
77.000
76.684
30.836
28.686
27.975
22.048
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O
N
OBz
Bz
1n
13C NMR
CDCl3
100 MHz
150
100
50
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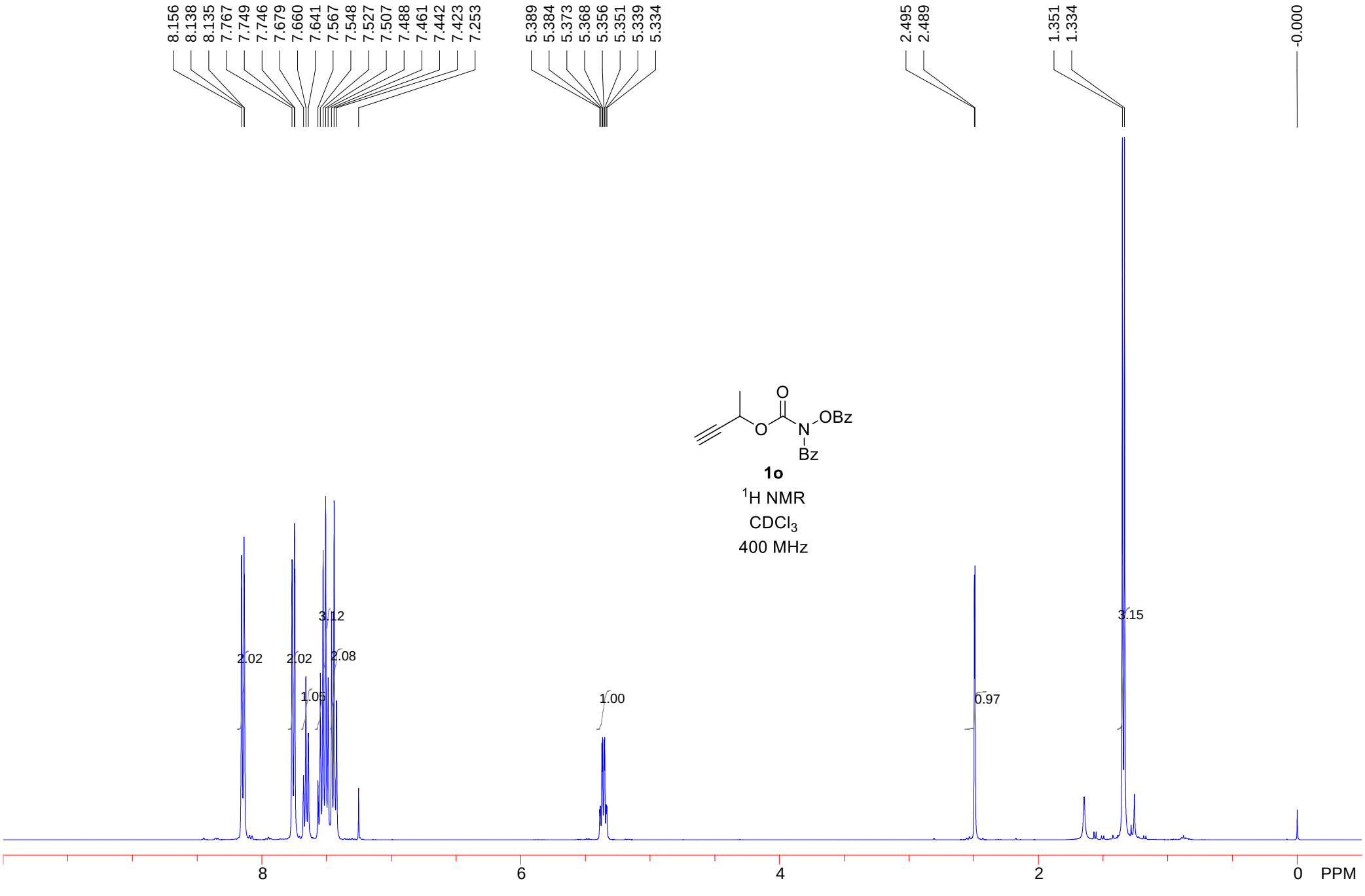
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8.138
8.135
7.767
7.749
7.746
7.679
7.660
7.641
7.567
7.548
7.527
7.507
7.488
7.461
7.442
7.423
7.253
5.389
5.384
5.373
5.368
5.356
5.351
5.339
5.334
2.495
2.489
1.351
1.334
-0.000
O
O
N
OBz
Bz
1o
1H NMR
CDCl3
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6
4
2
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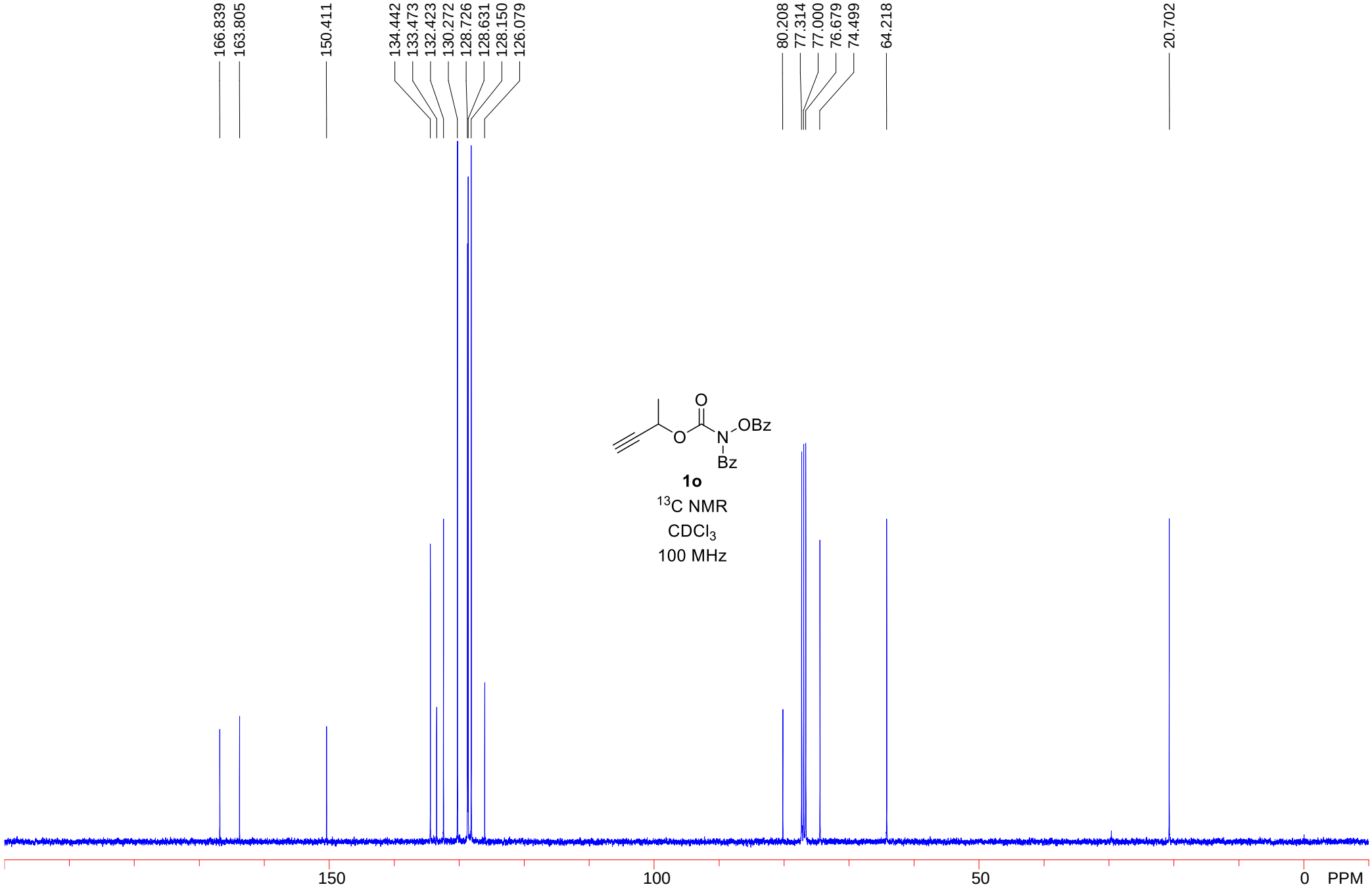
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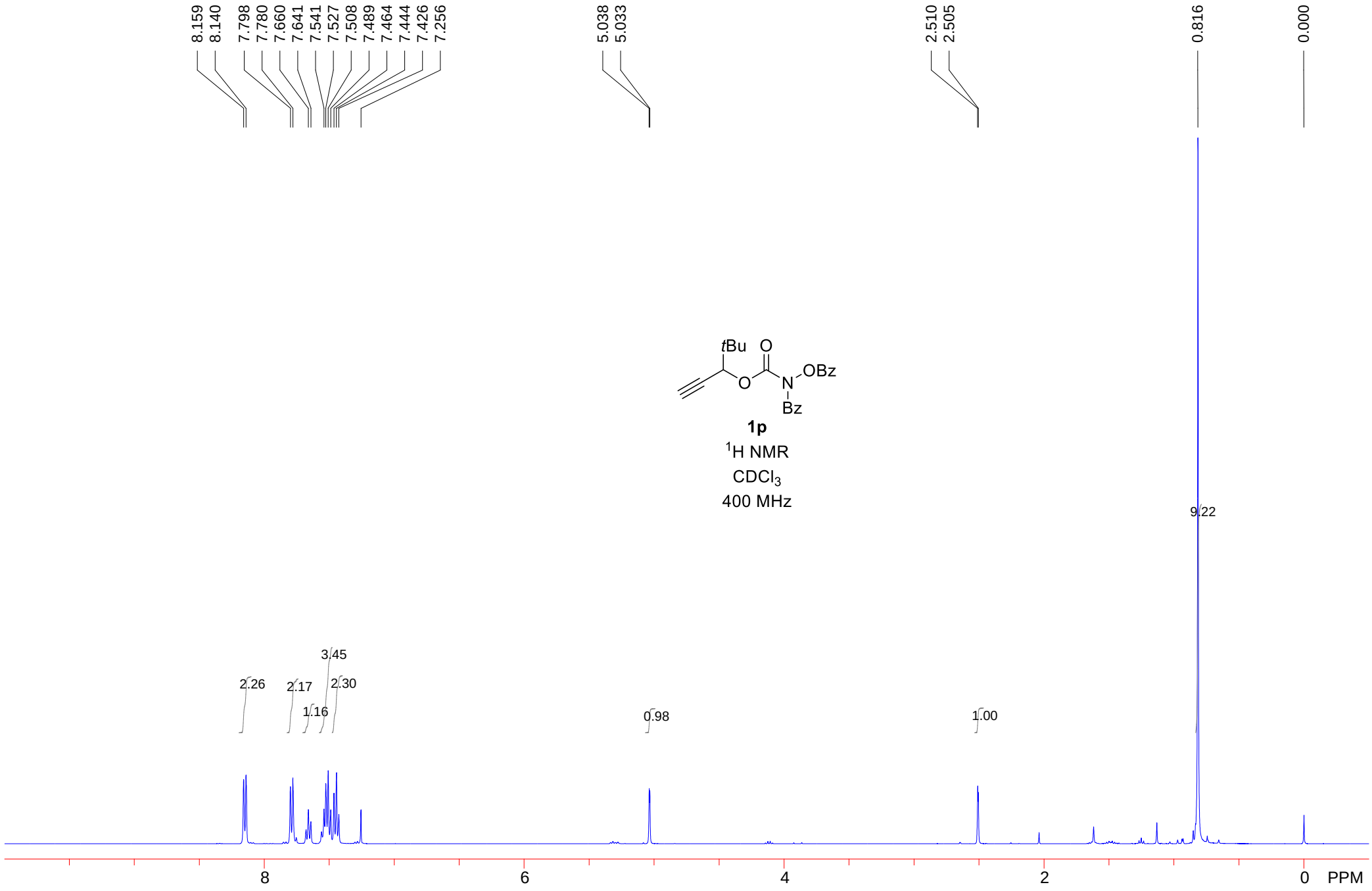
150.411
134.442
133.473
132.423
130.272
128.726
128.631
128.150
126.079
80.208
77.314
77.000
76.679
74.499
64.218
20.702
O
OBz
O
N
Bz
1o
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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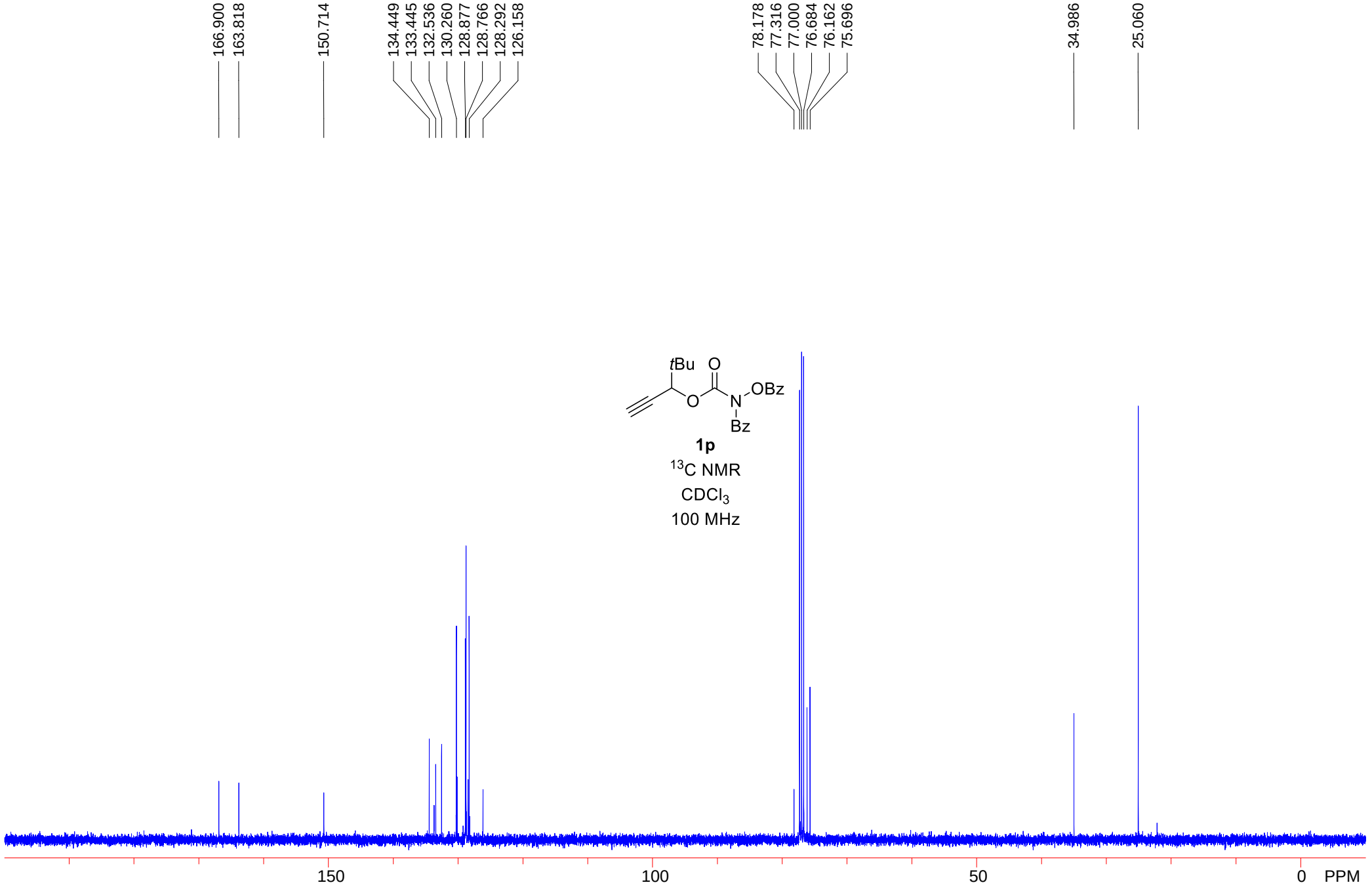
7.798
7.780
7.660
7.641
7.541
7.527
7.508
7.489
7.464
7.444
7.426
7.256
5.038
5.033
2.510
2.505
0.816
0.000
tBu
O
O
N
OBz
Bz
1p
1H NMR
CDCl3
400 MHz
9.22
3.45
2.26
2.17
2.30
1.16
0.98
1.00
8
6
4
2
0
PPM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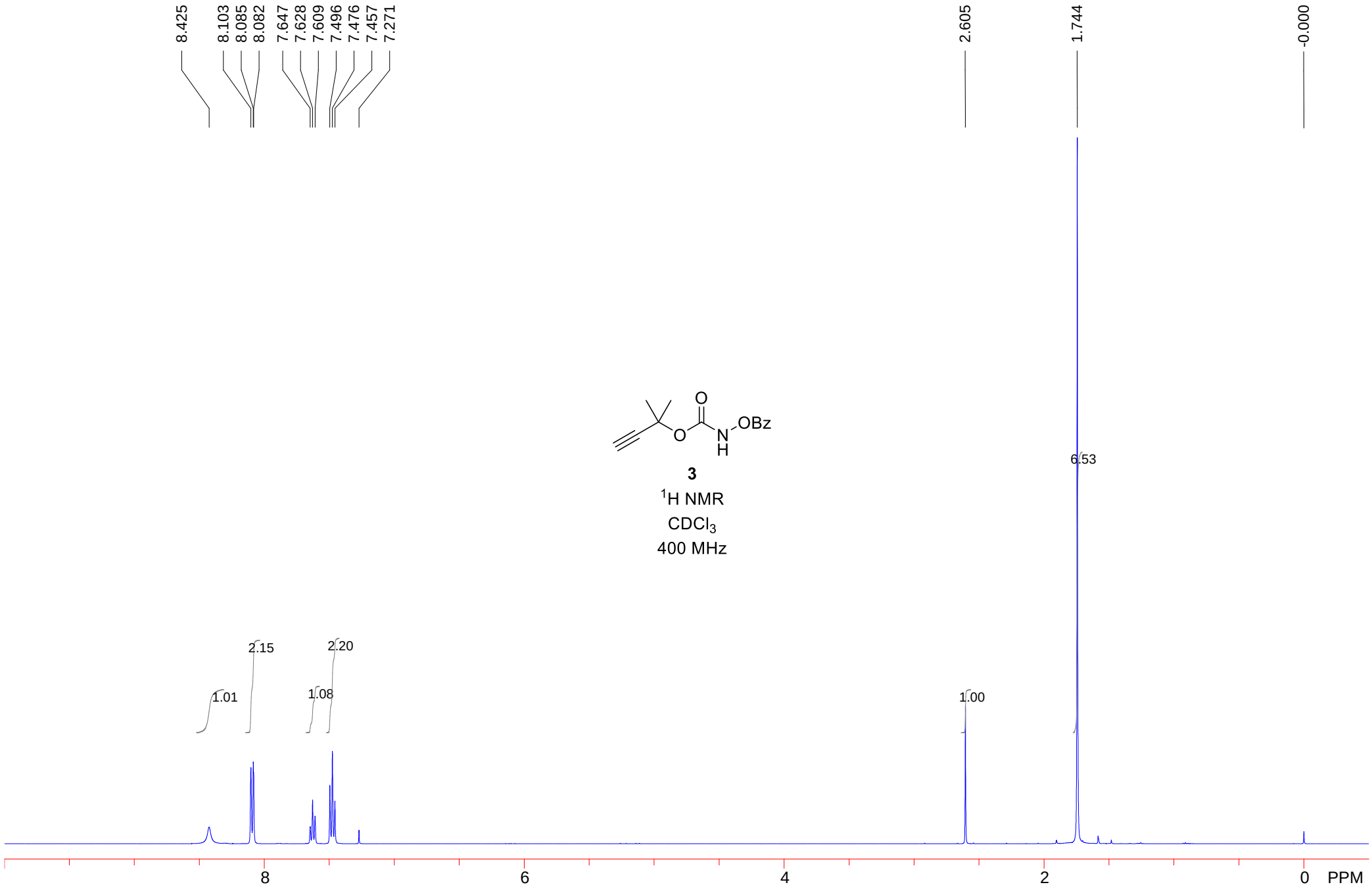
150.714
134.449
133.445
132.536
130.260
128.877
128.766
128.292
126.158
78.178
77.316
77.000
76.684
76.162
75.696
34.986
25.060
tBu
O
O
N
OBz
Bz
1p
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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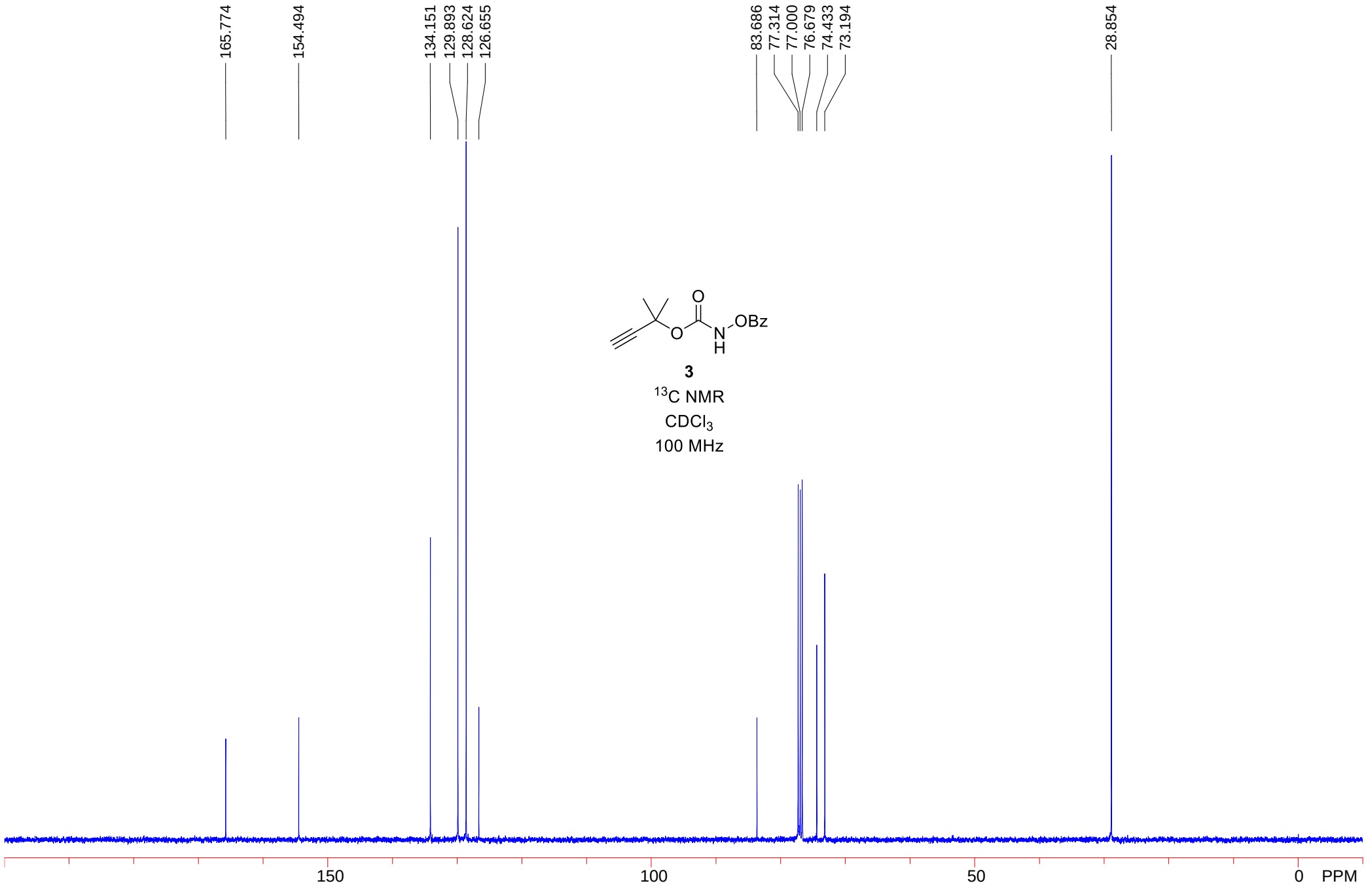
8.082
7.647
7.628
7.609
7.496
7.476
7.457
7.271
2.605
1.744
-0.000
O
O
N
H
OBz
3
1H NMR
CDCl3
400 MHz
6.53
2.15
2.20
1.01
1.08
1.00
8
6
4
2
0
PPM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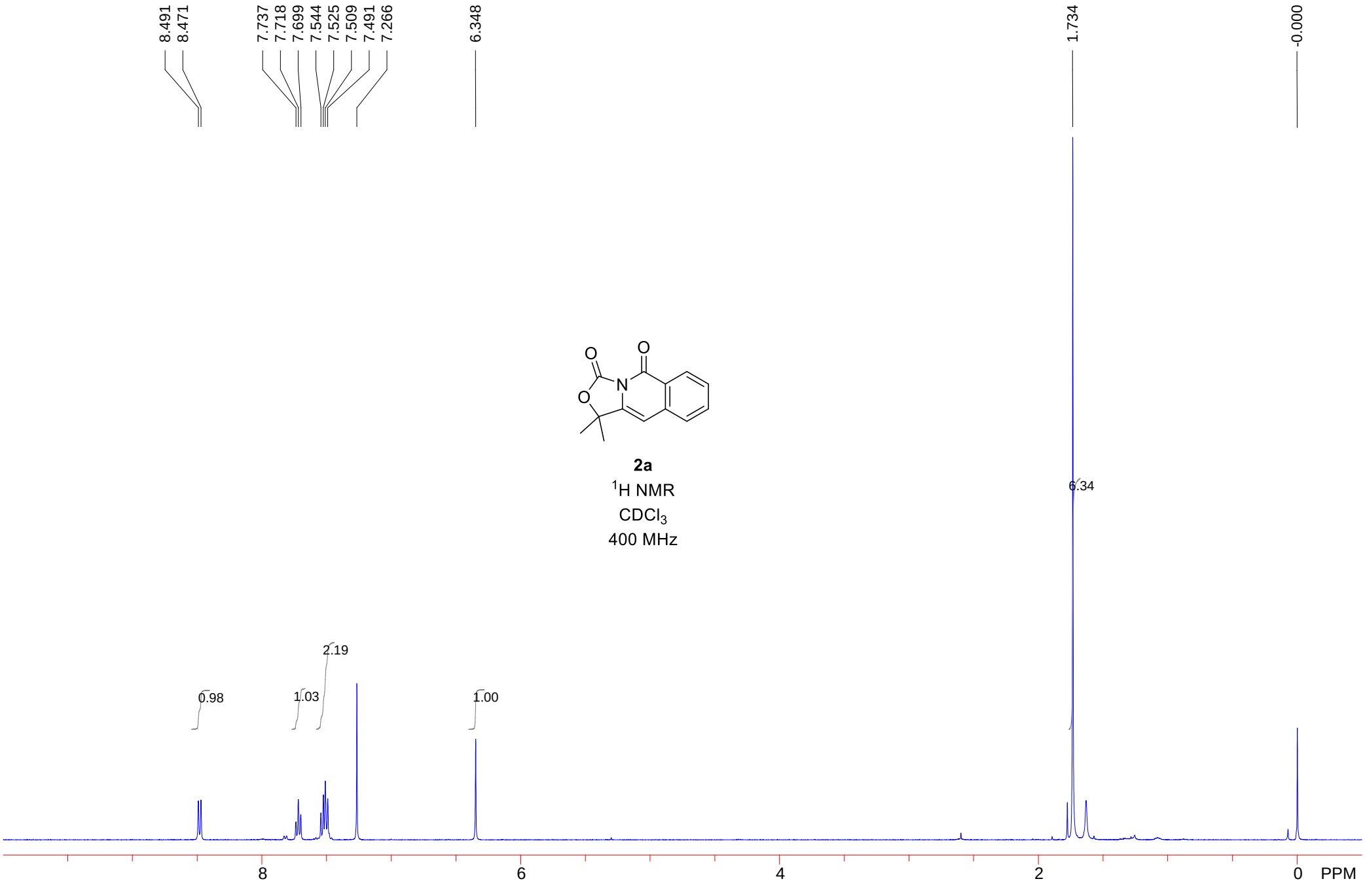
154.494
134.151
129.893
128.624
126.655
83.686
77.314
77.000
76.679
74.433
73.194
28.854
O
O
N
H
OBz
3
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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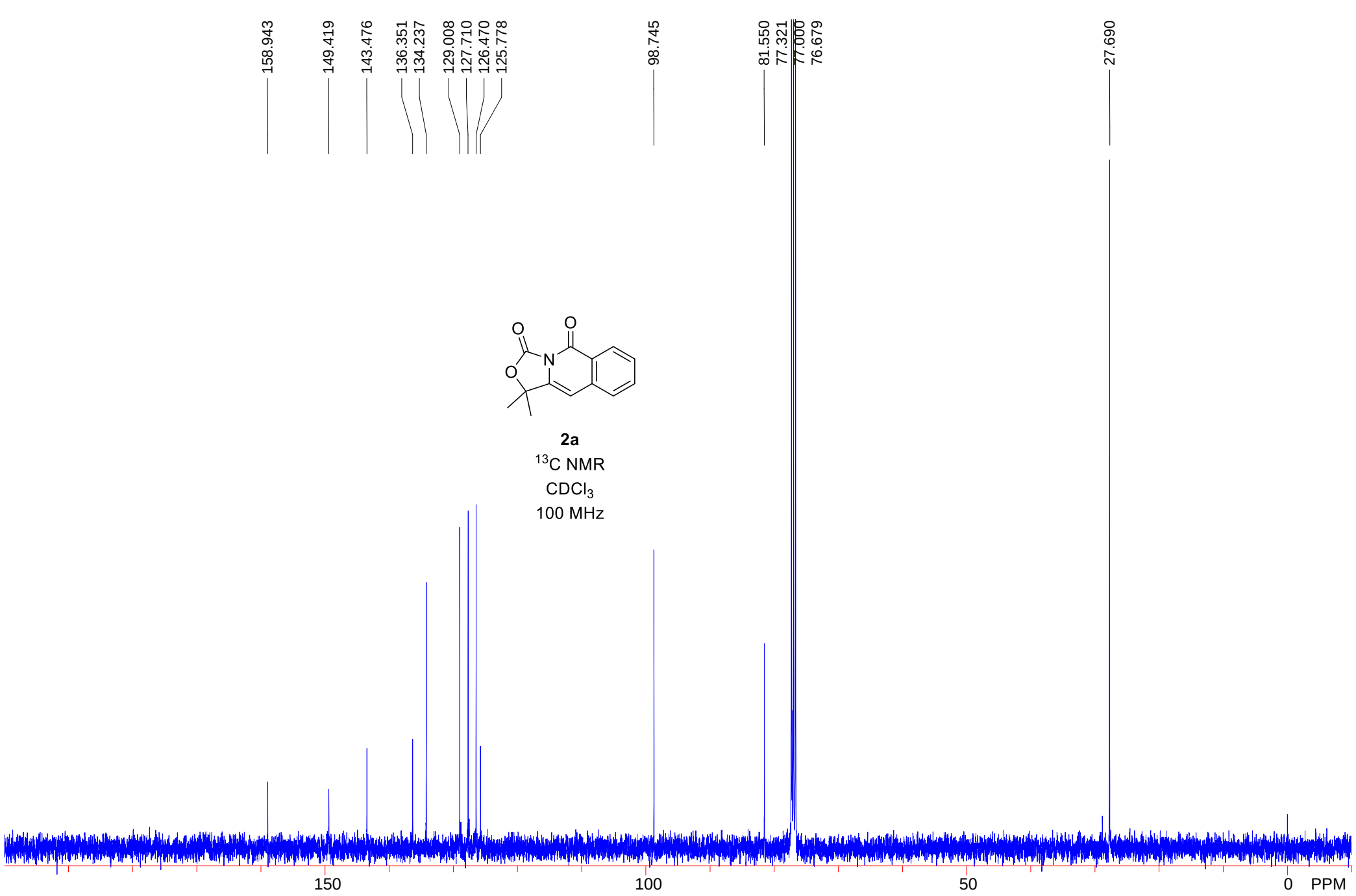
8.471
7.737
7.718
7.699
7.544
7.525
7.509
7.491
7.266
6.348
1.734
-0.000
O
O
N
O
2a
1H NMR
CDCl3
400 MHz
6.34
2.19
0.98
1.03
1.00
8
6
4
2
0
PPM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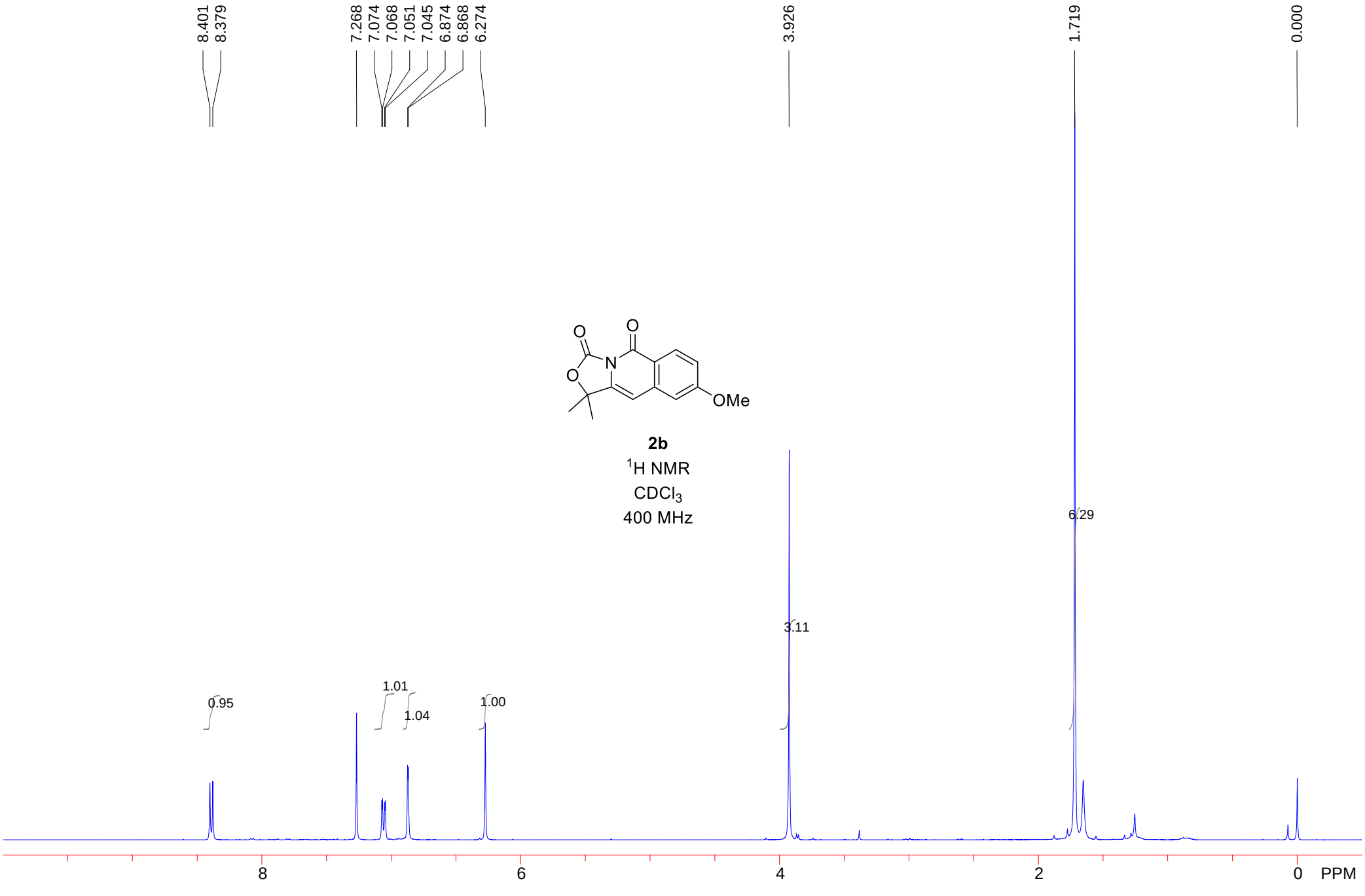
134.237
129.008
127.710
126.470
125.778
98.745
81.550
77.321
77.000
76.679
27.690
O
O
N
O
2a
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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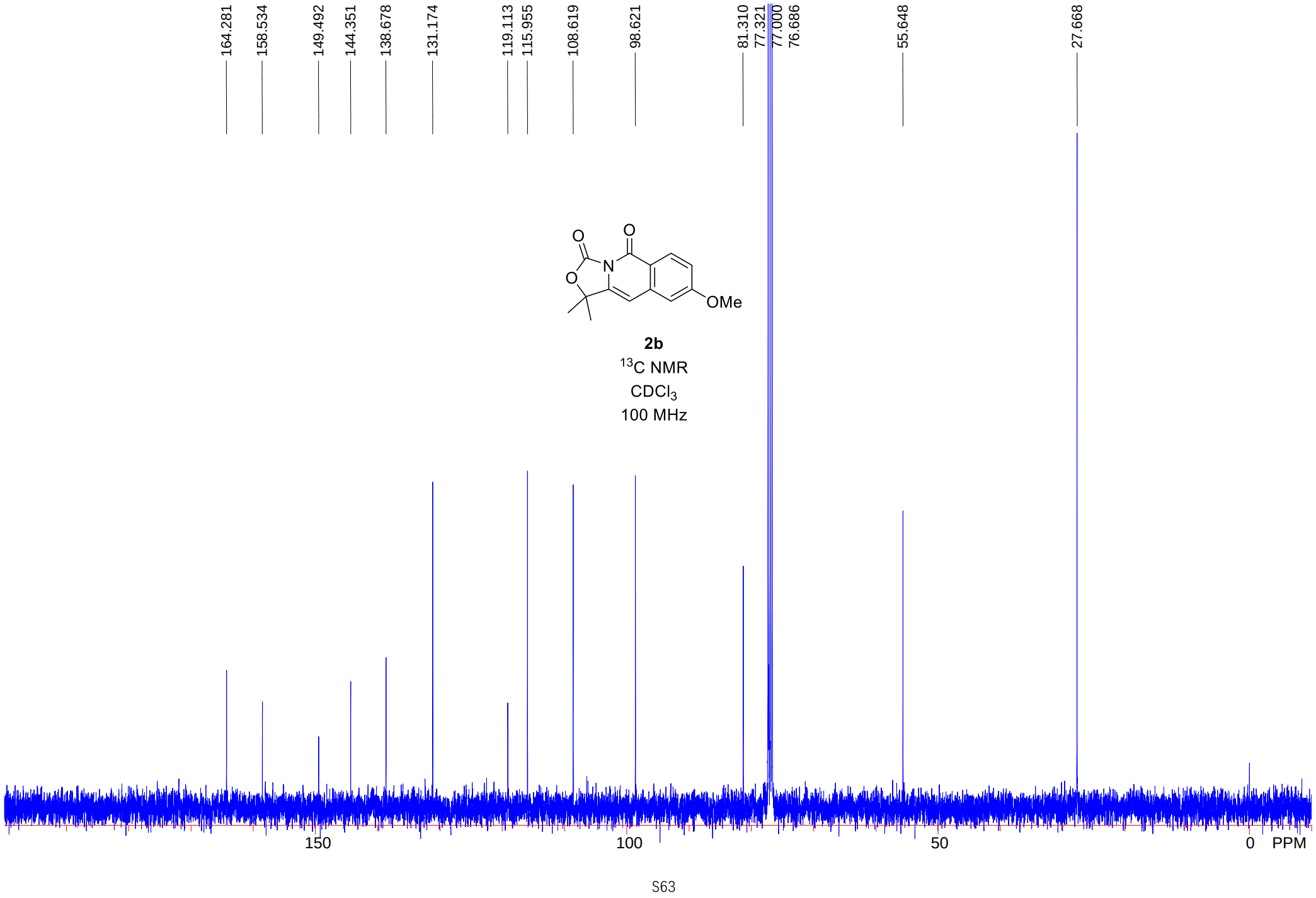
7.268
7.074
7.068
7.051
7.045
6.874
6.868
6.274
3.926
1.719
0.000
O
O
N
O
OMe
2b
1H NMR
CDCl3
400 MHz
0.95
1.01
1.04
1.00
3.11
6.29
8
6
4
2
0
PPM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144.351
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131.174
119.113
115.955
108.619
98.621
81.310
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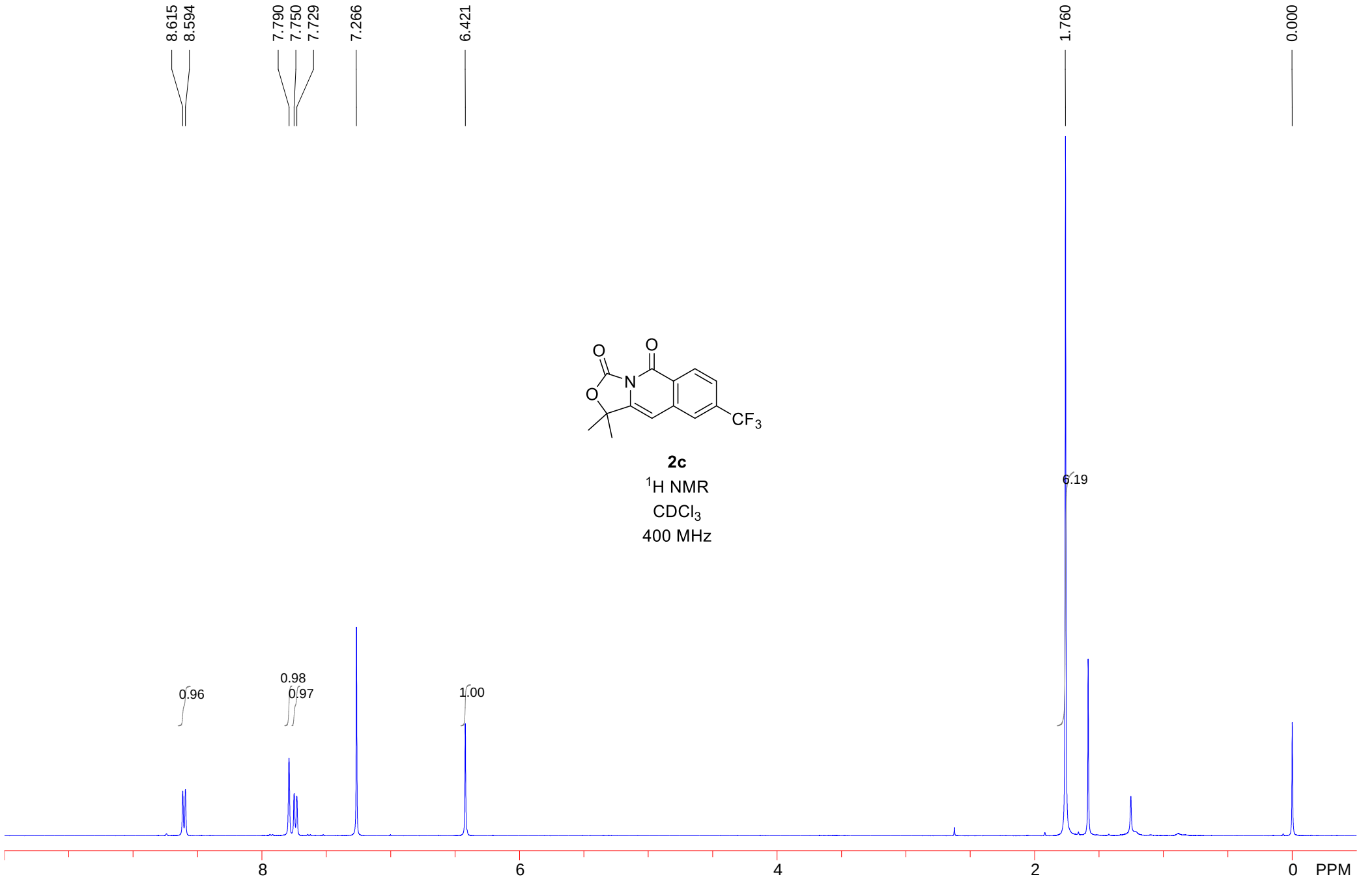
77.000
76.686
55.648
27.668
O
O
N
O
OMe
2b
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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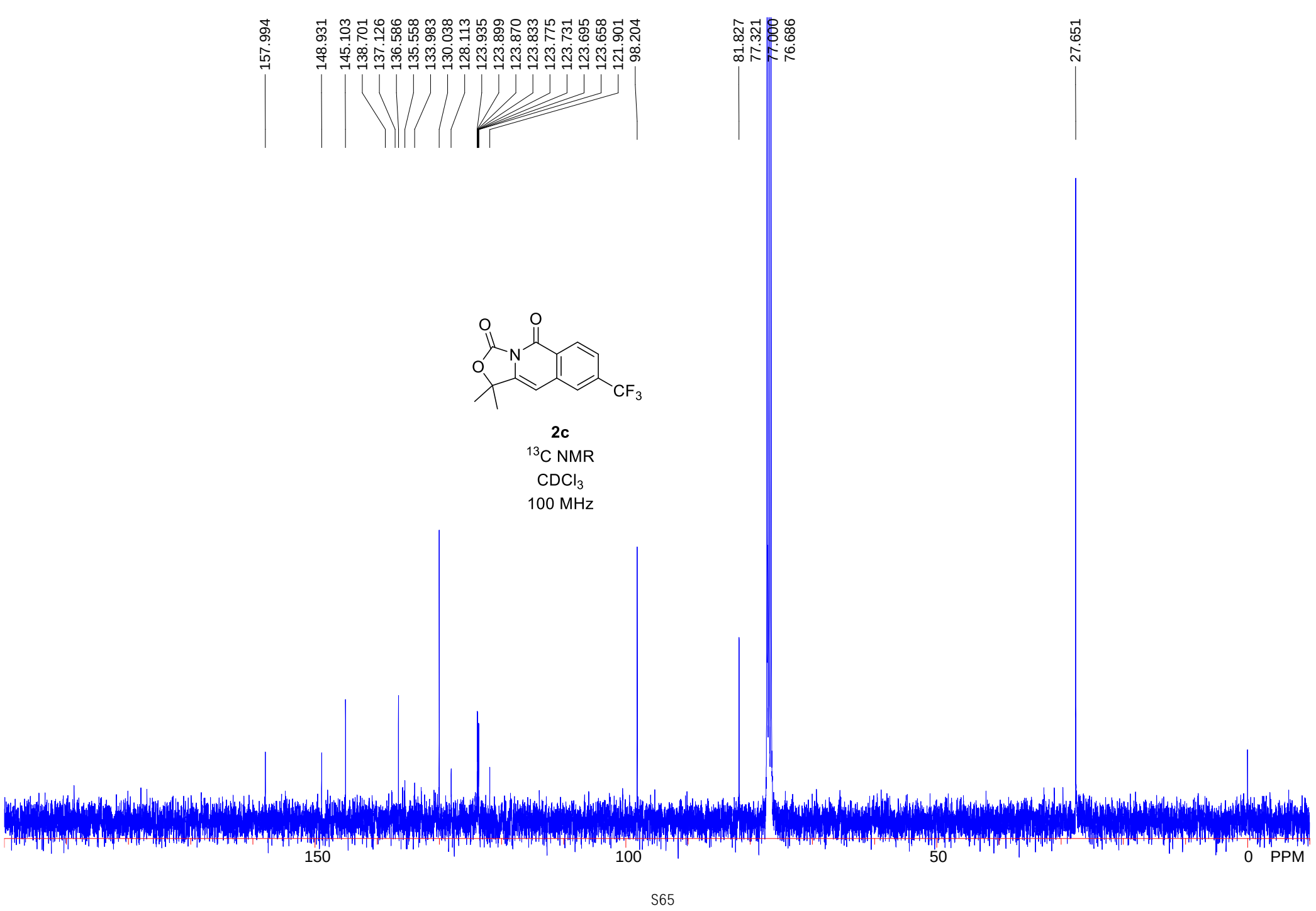
7.790
7.750
7.729
7.266
6.421
1.760
0.000
O
O
N
O
CF3
2c
1H NMR
CDCl3
400 MHz
0.96
0.98
0.97
1.00
6.19
8
6
4
2
0
PPM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145.103
138.701
137.126
136.586
135.558
133.983
130.038
128.113
123.935
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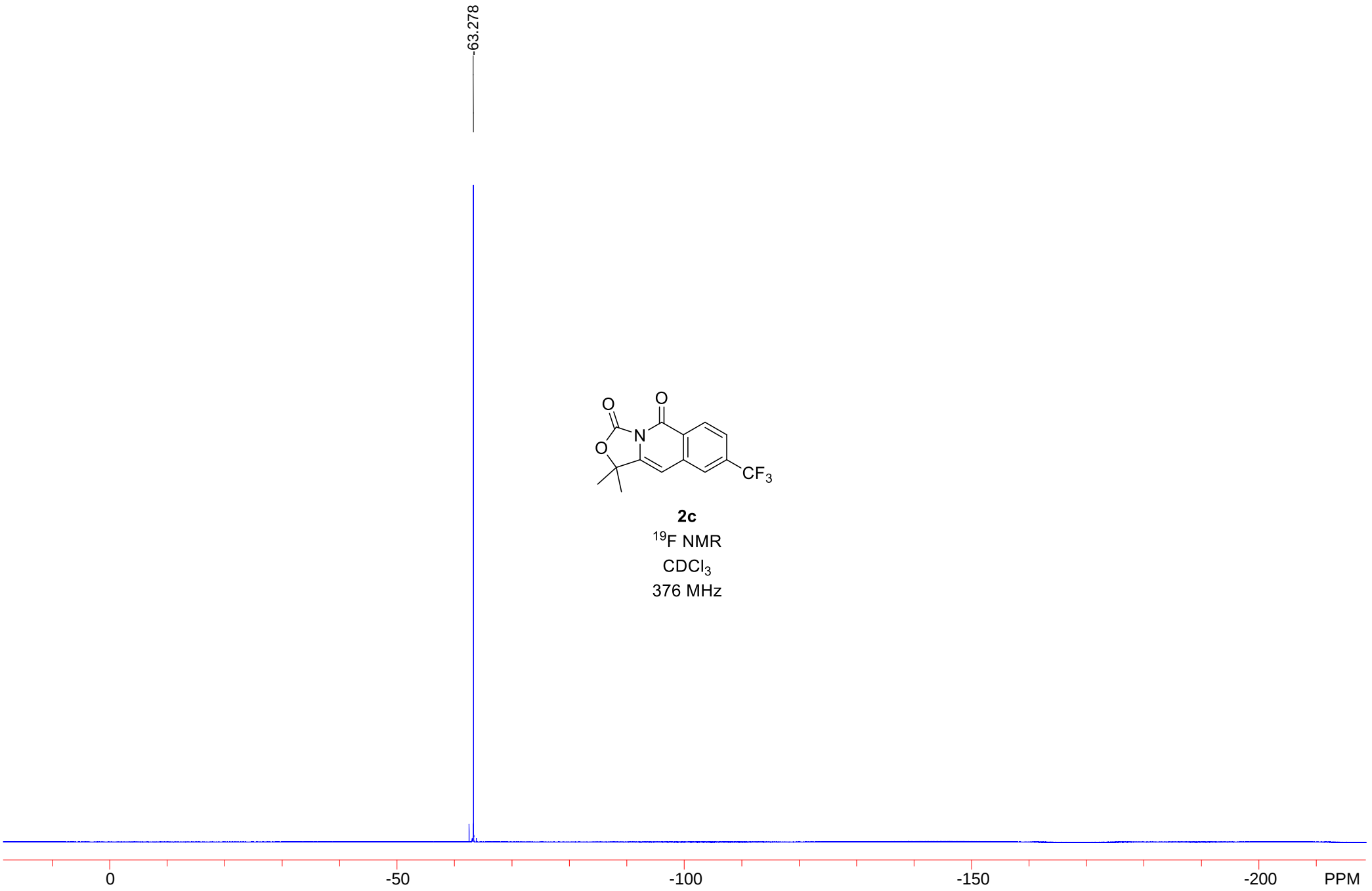
123.870
123.833
123.775
123.731
123.695
123.658
121.901
98.204
81.827
77.321
77.000
76.686
27.651
O
O
N
O
CF3
2c
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM

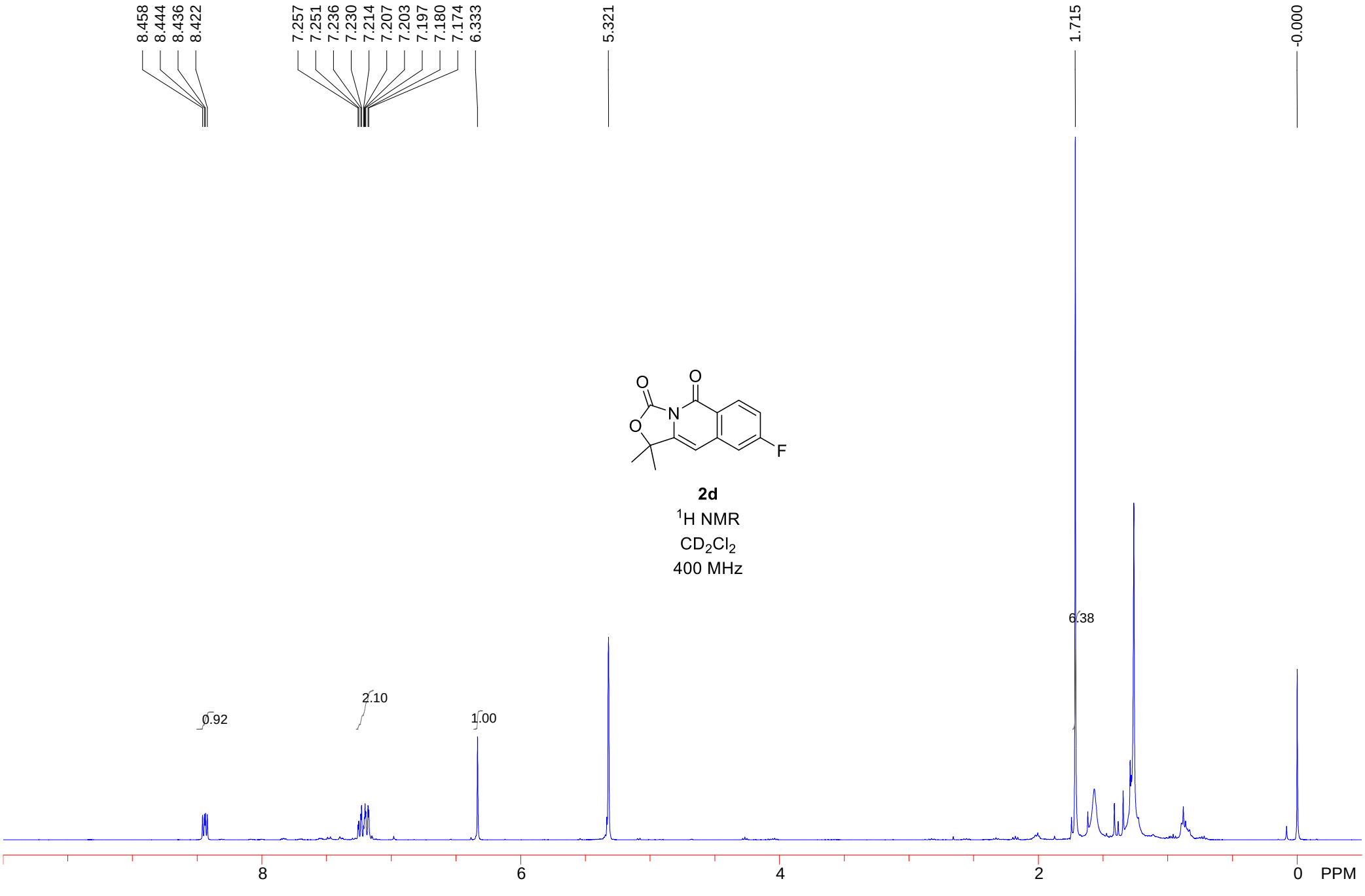


2c

^{19}F NMR

$CDCl_3$

376 MHz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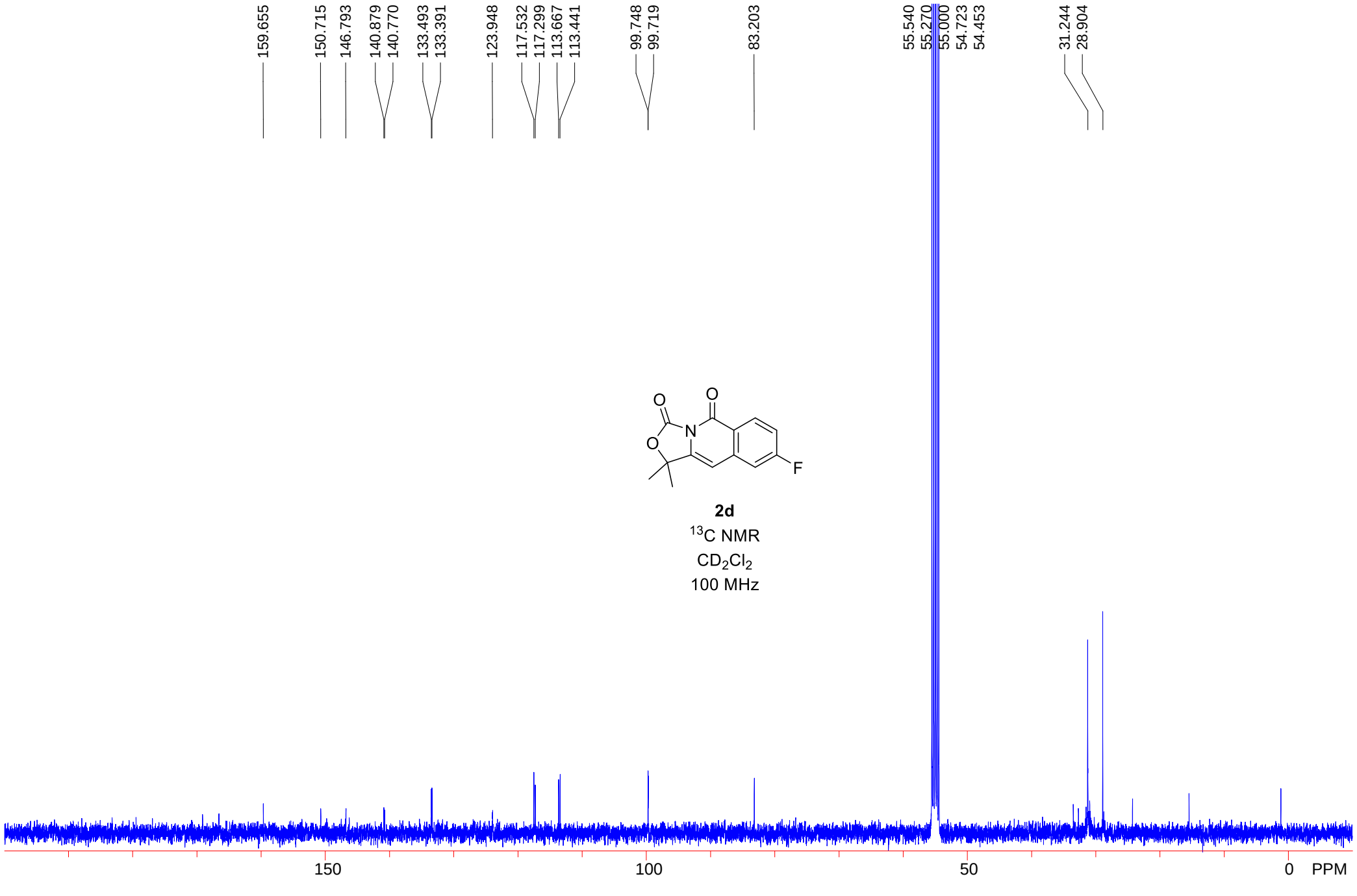
7.257
7.251
7.236
7.230
7.214
7.207
7.203
7.197
7.180
7.174
6.333
5.321
1.715
-0.000
O
O
N
O
F
2d
1H NMR
CD2Cl2
400 MHz
0.92
2.10
1.00
6.38
8
6
4
2
0
PPM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140.879
140.770
133.493
133.391
123.948
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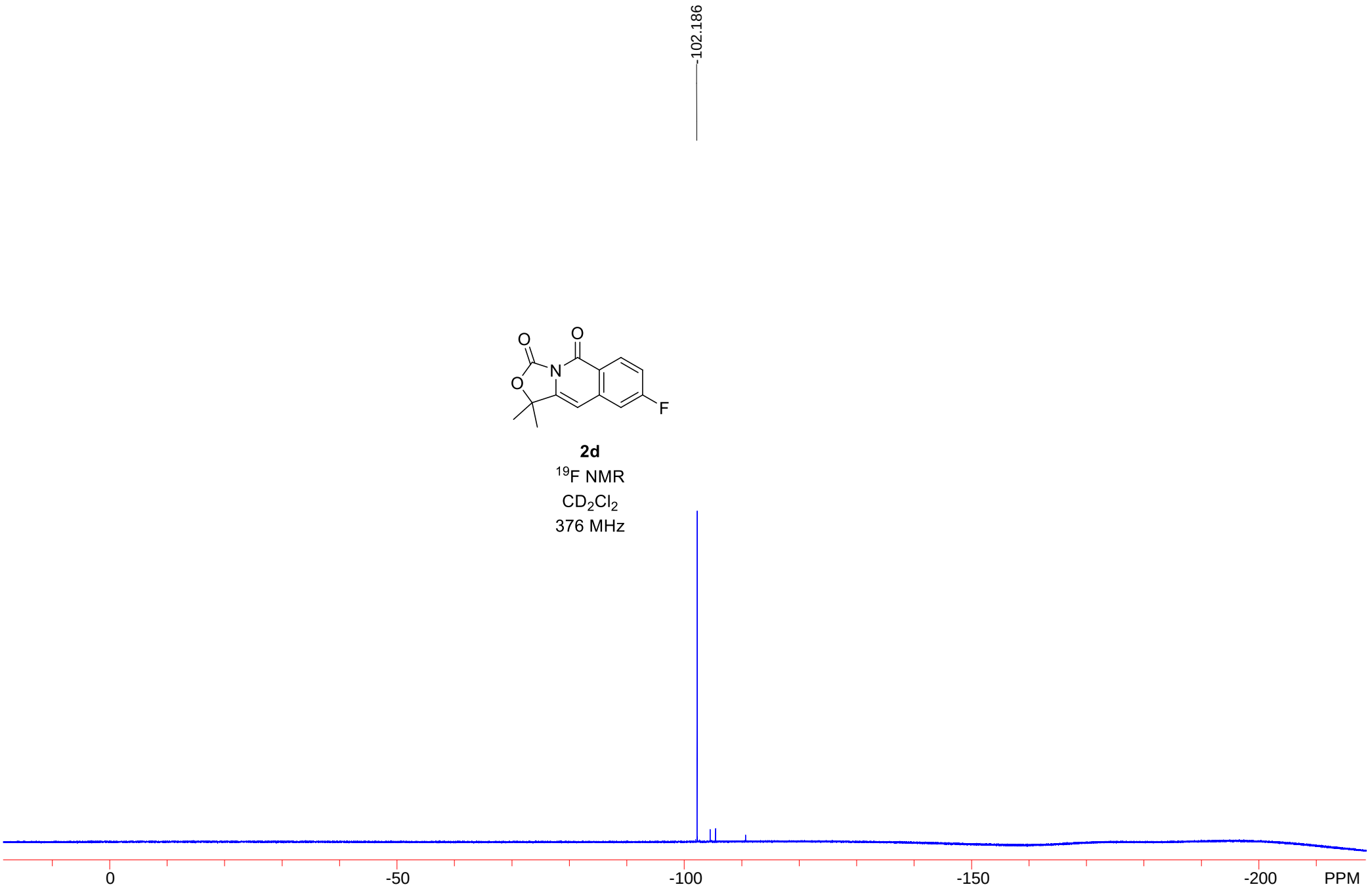
117.299
113.667
113.441
99.748
99.719
83.203
55.540
55.270
55.000
54.723
54.453
31.244
28.904
O
O
N
O
F
2d
13C NMR
CD2Cl2
100 MHz
150
100
50
0
PPM

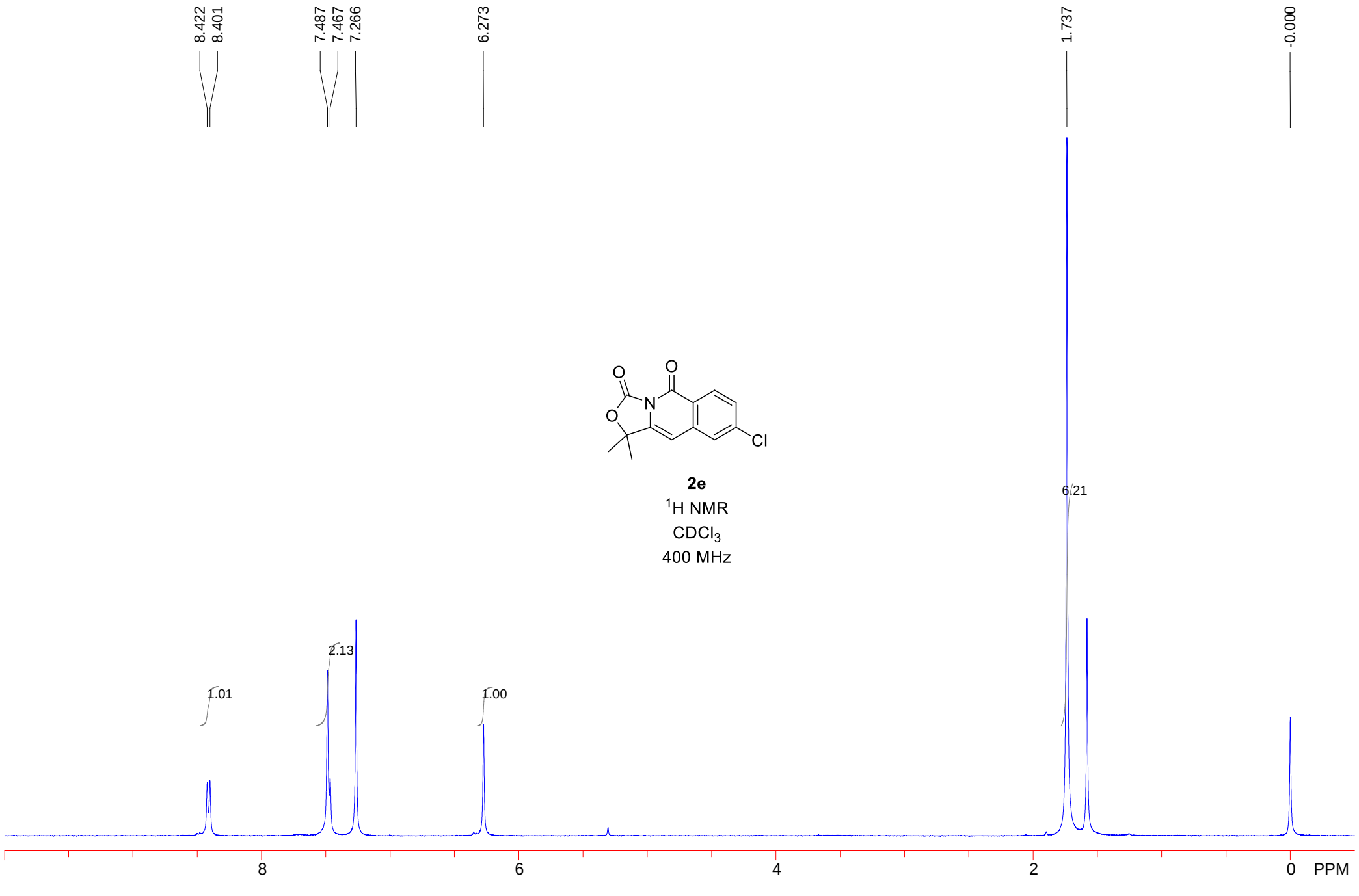


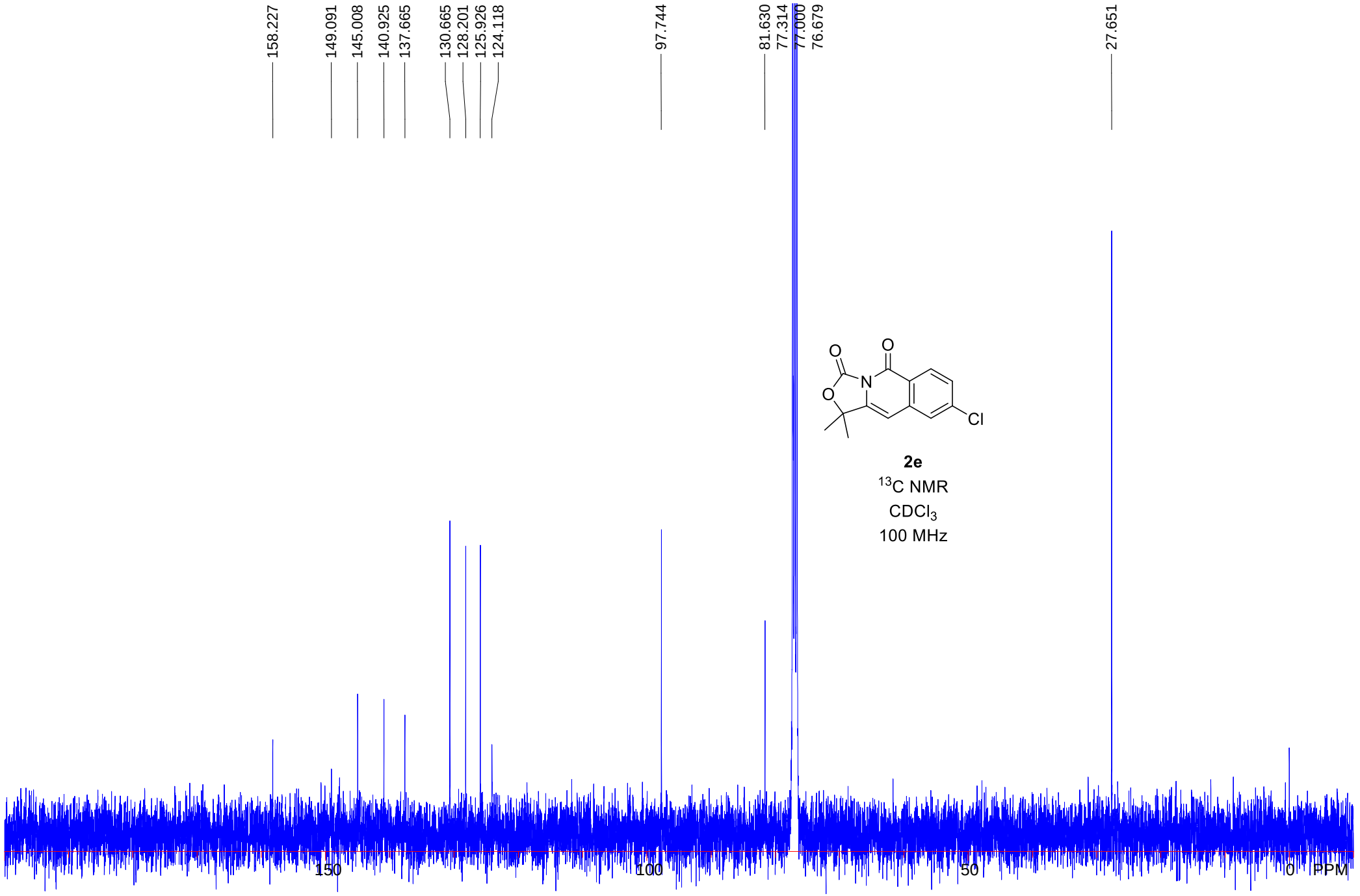
2d

^{19}F NMR

CD_2Cl_2

376 MHz


8.422
8.401
7.487
7.467
7.266
6.273
1.737
-0.000
O
O
N
O
Cl
2e
1H NMR
CDCl3
400 MHz
6.21
2.13
1.01
1.00
8
6
4
2
0
PPM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130.665
128.201
125.926
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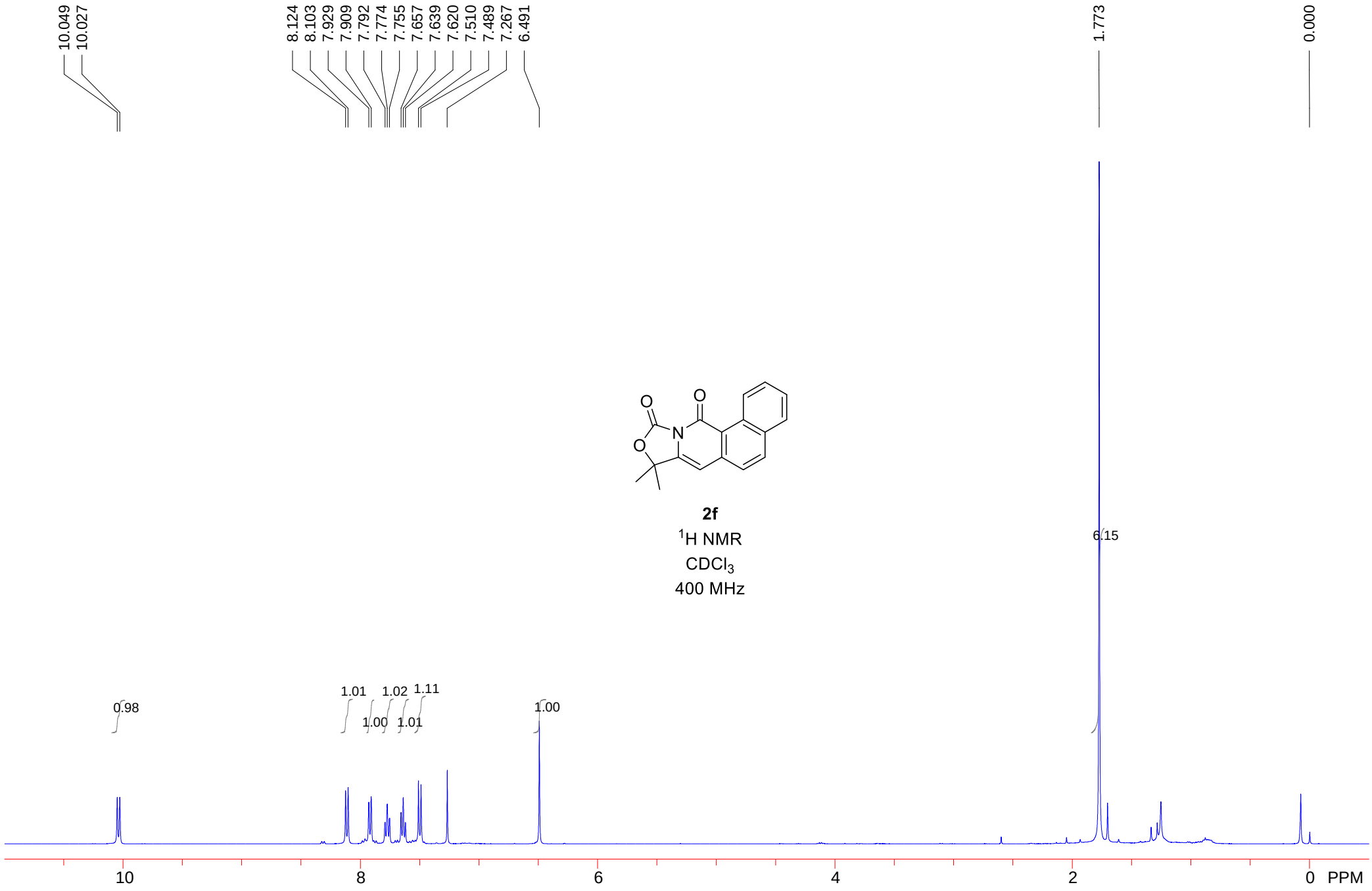
97.744
81.630
77.314
77.000
76.679
27.651
O
O
N
O
Cl
2e
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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8.103
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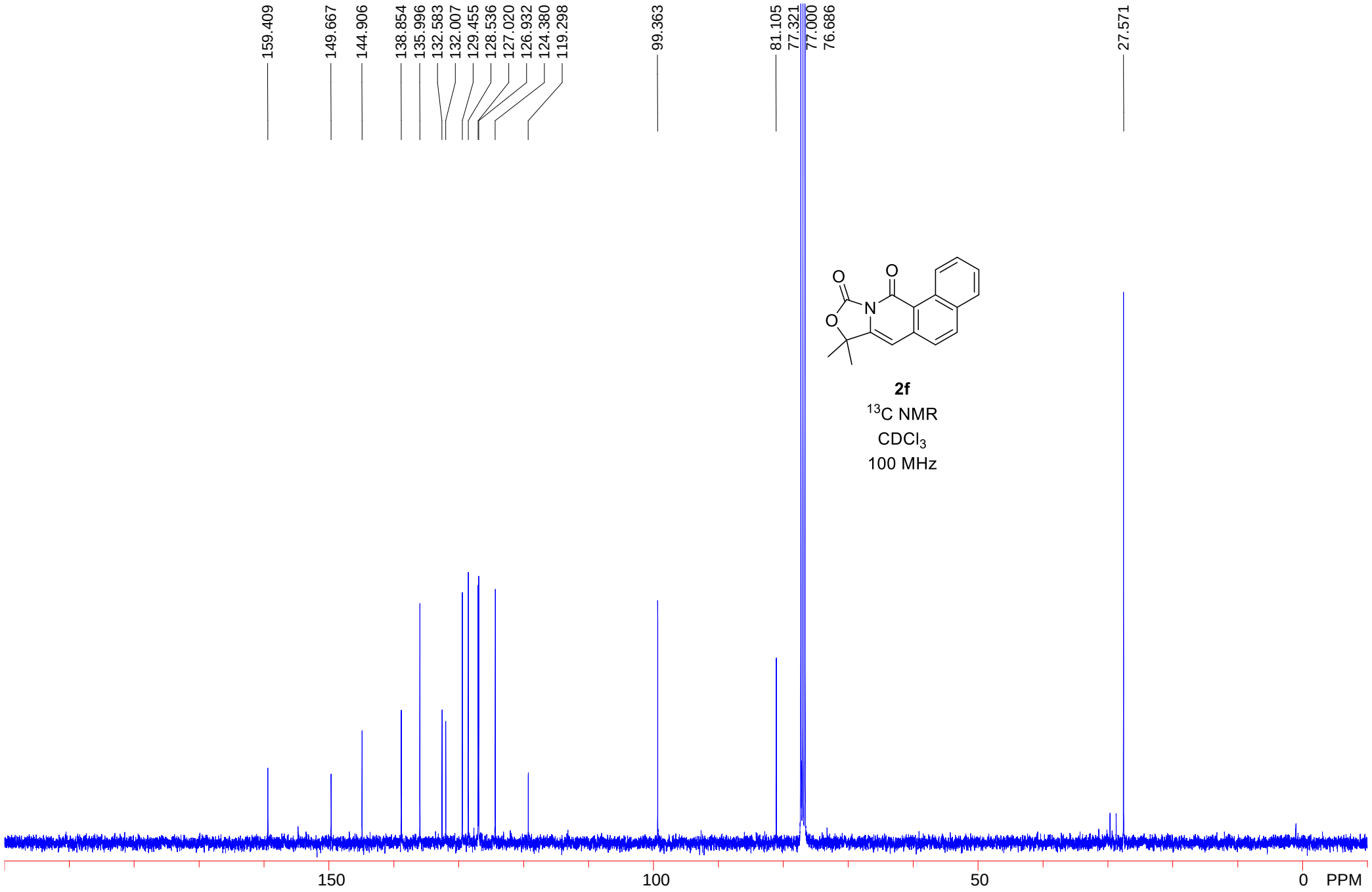
7.909
7.792
7.774
7.755
7.657
7.639
7.620
7.510
7.489
7.267
6.491
1.773
0.000
O
O
N
O
2f
1H NMR
CDCl3
400 MHz
6.15
0.98
1.01
1.00
1.02
1.01
1.11
1.00
10
8
6
4
2
0
PPM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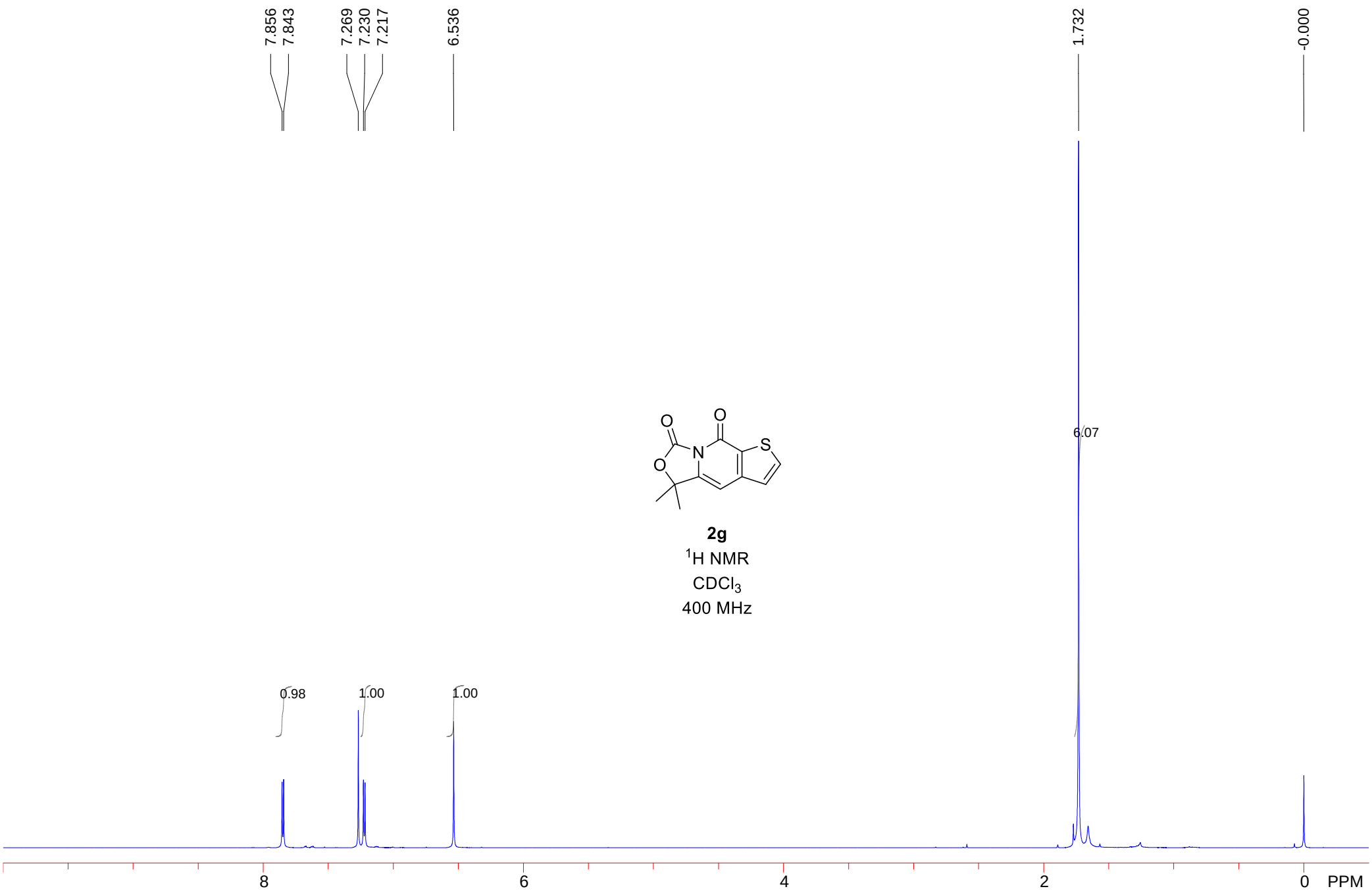
135.996
132.583
132.007
129.455
128.536
127.020
126.932
124.380
119.298
99.363
81.105
77.321
77.000
76.686
27.571
O
O
O
N
2f
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM

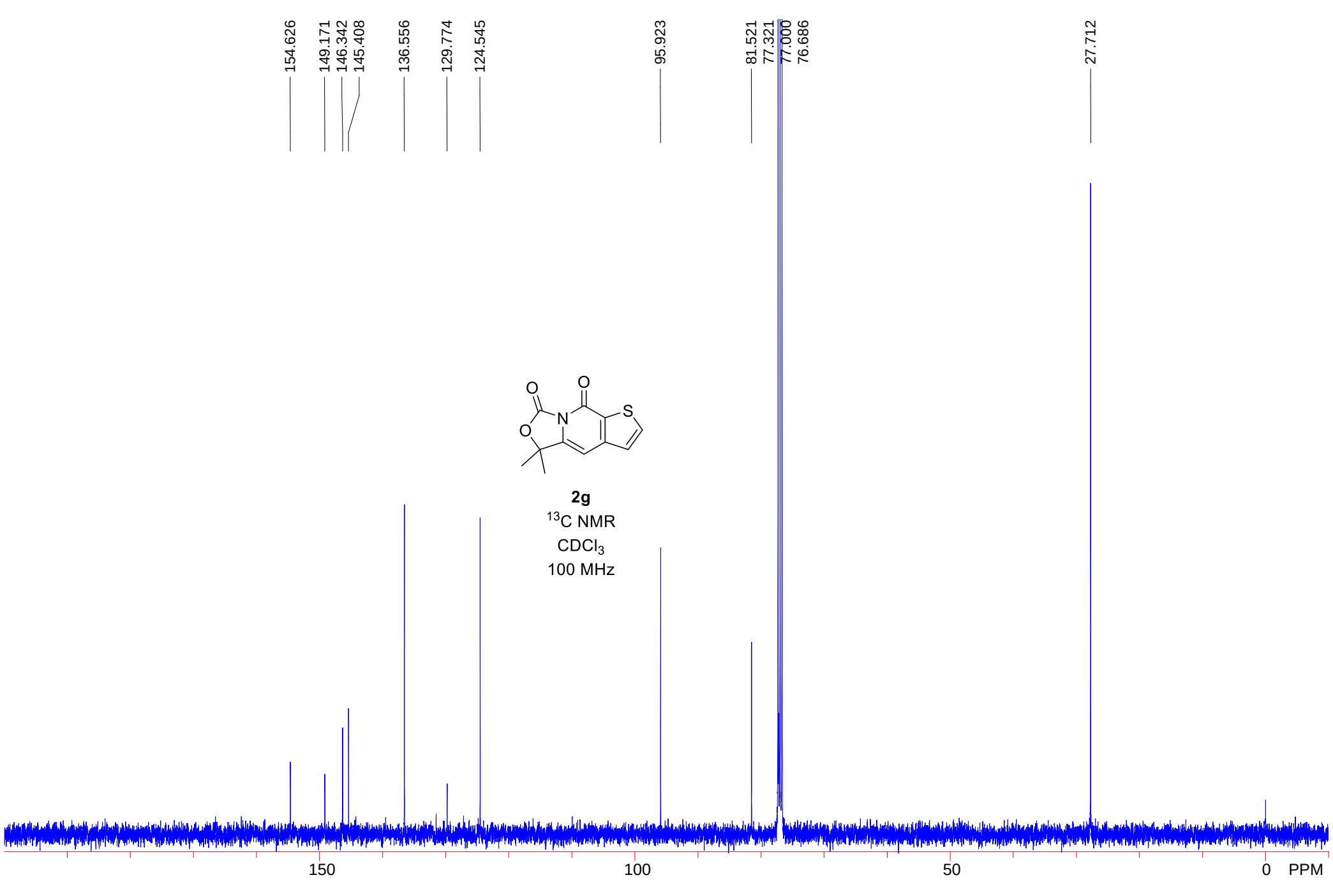


2g

^{1}H NMR

$CDCl_3$

400 MHz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146.342
145.408
136.556
129.774
124.545
95.923
81.521
77.321
77.000
76.686
27.712
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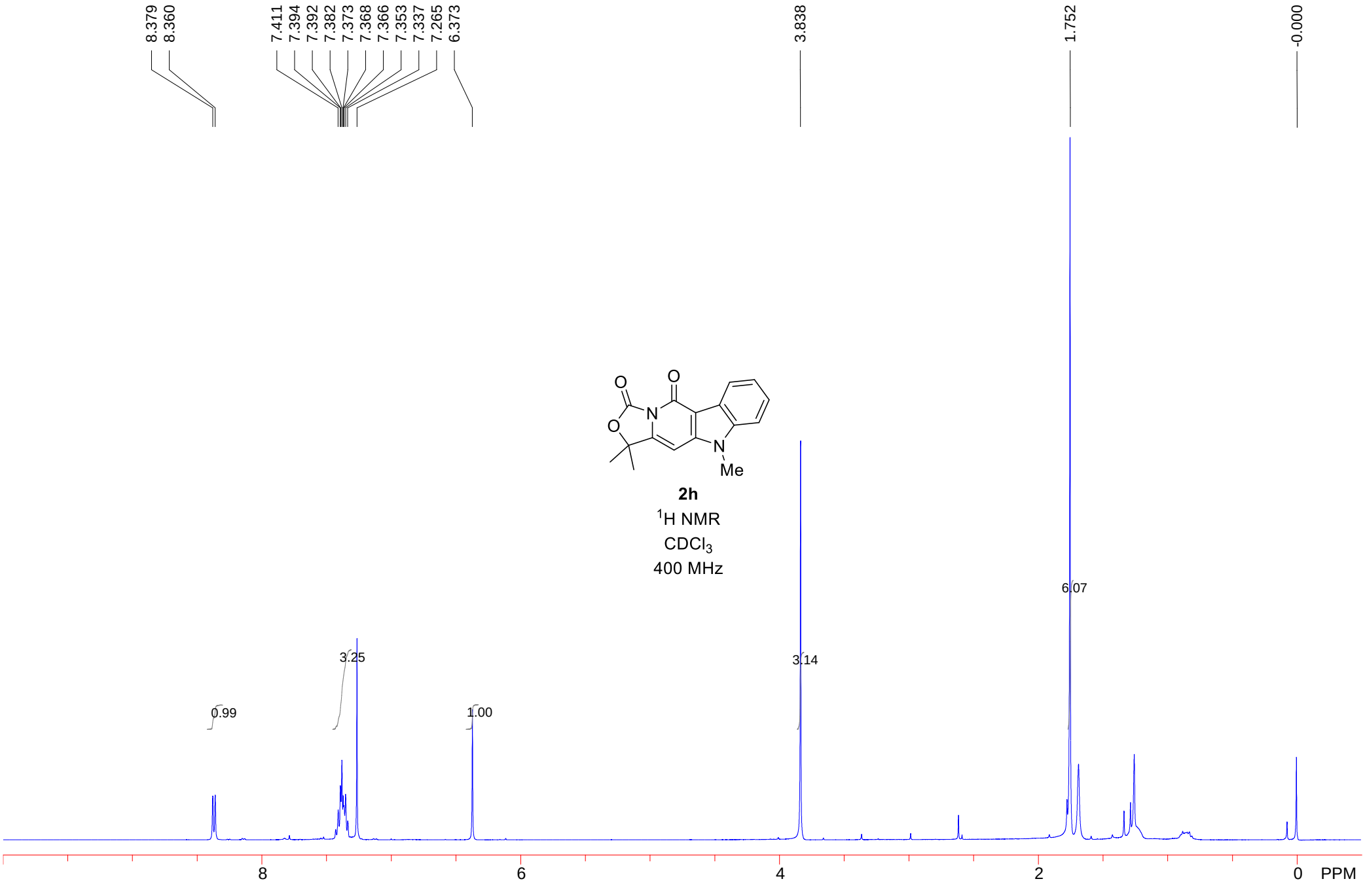
O
N
O
S
2g
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM


8.379
8.360
7.411
7.394
7.392
7.382
7.373
7.368
7.366
7.353
7.337
7.265
6.373
3.838
1.752
-0.000
O
O
N
O
N
Me
2h
1H NMR
CDCl3
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1.00
3.14
6.07
8
6
4
2
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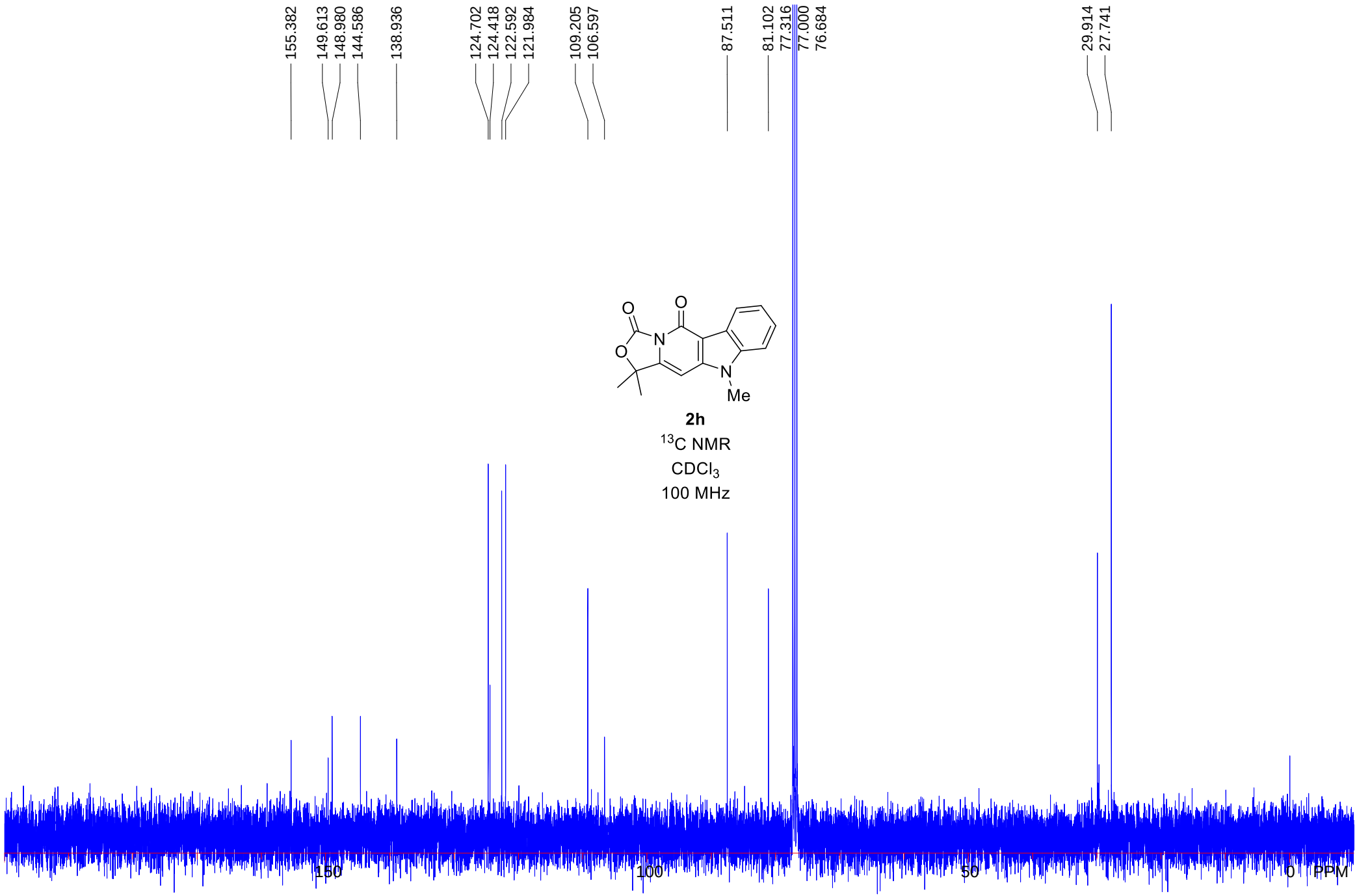
PPM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144.586
138.936
124.702
124.418
122.592
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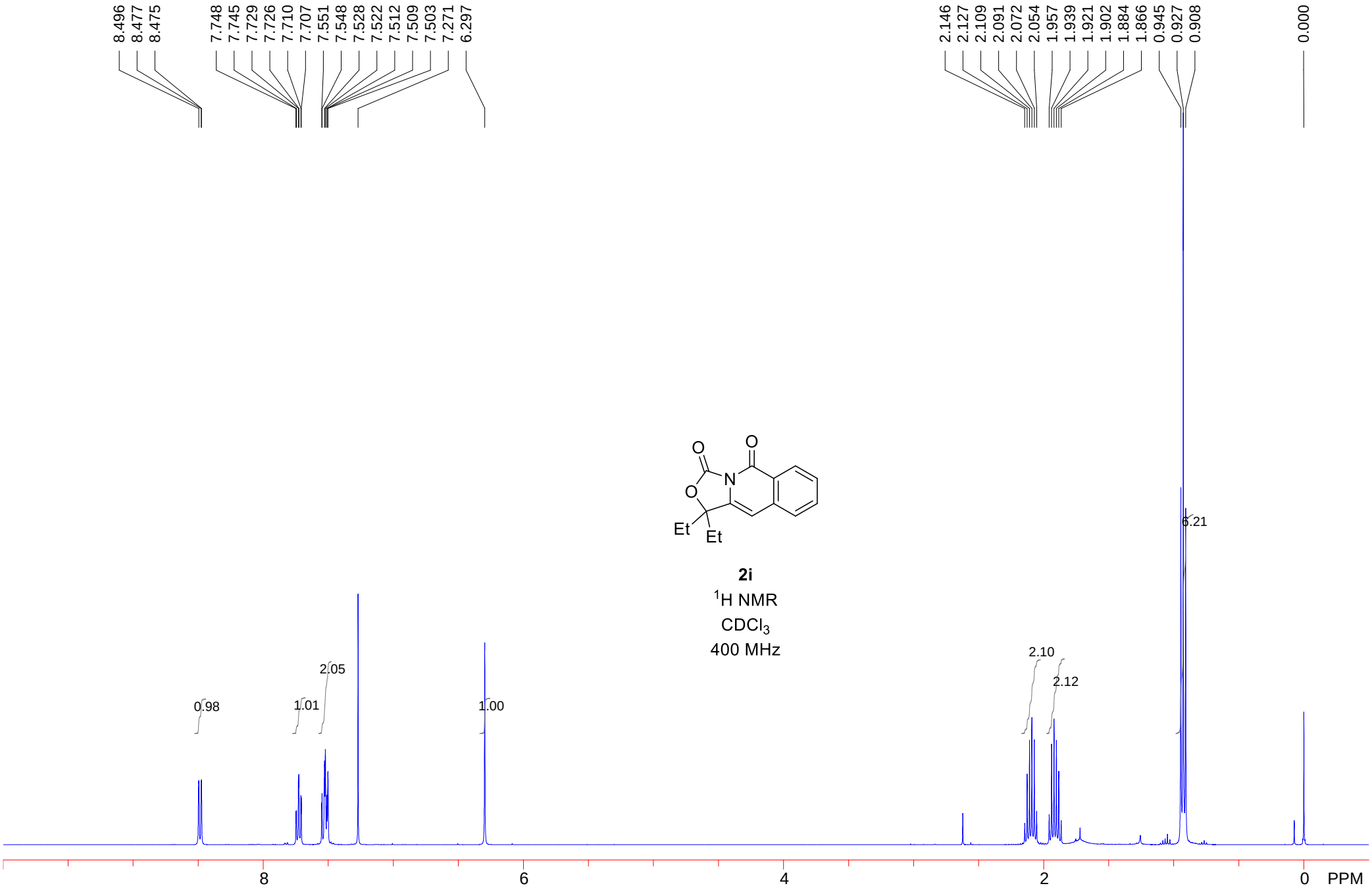
109.205
106.597
87.511
81.102
77.316
77.000
76.684
29.914
27.741
O
O
N
O
N
Me
2h
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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8.475
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7.745
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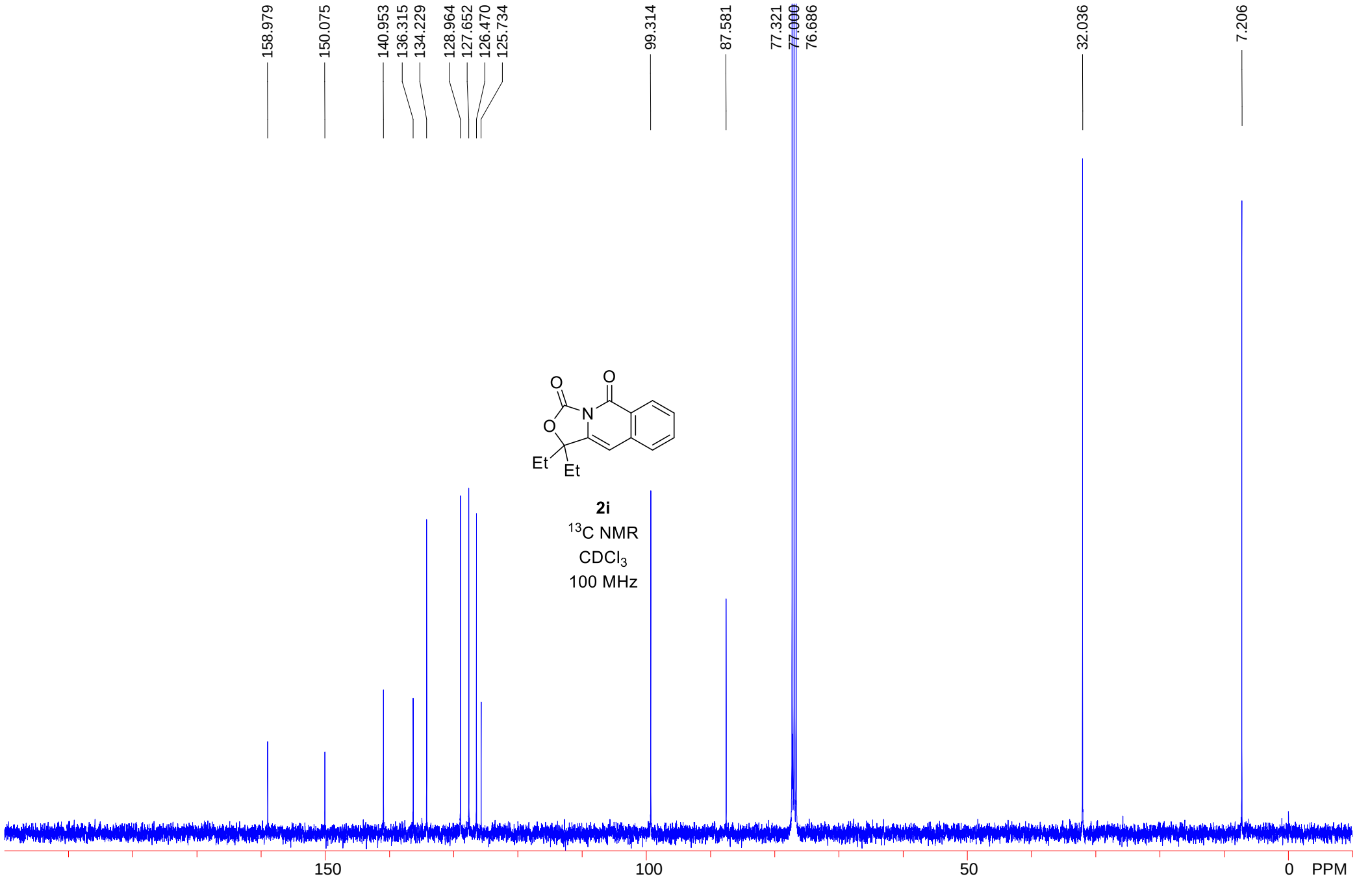
7.726
7.710
7.707
7.551
7.548
7.528
7.522
7.512
7.509
7.503
7.271
6.297
2.146
2.127
2.109
2.091
2.072
2.054
1.957
1.939
1.921
1.902
1.884
1.866
0.945
0.927
0.908
0.000
O
O
N
O
Et
Et
2i
1H NMR
CDCl3
400 MHz
0.98
1.01
2.05
1.00
2.10
2.12
6.21
8
6
4
2
0
PPM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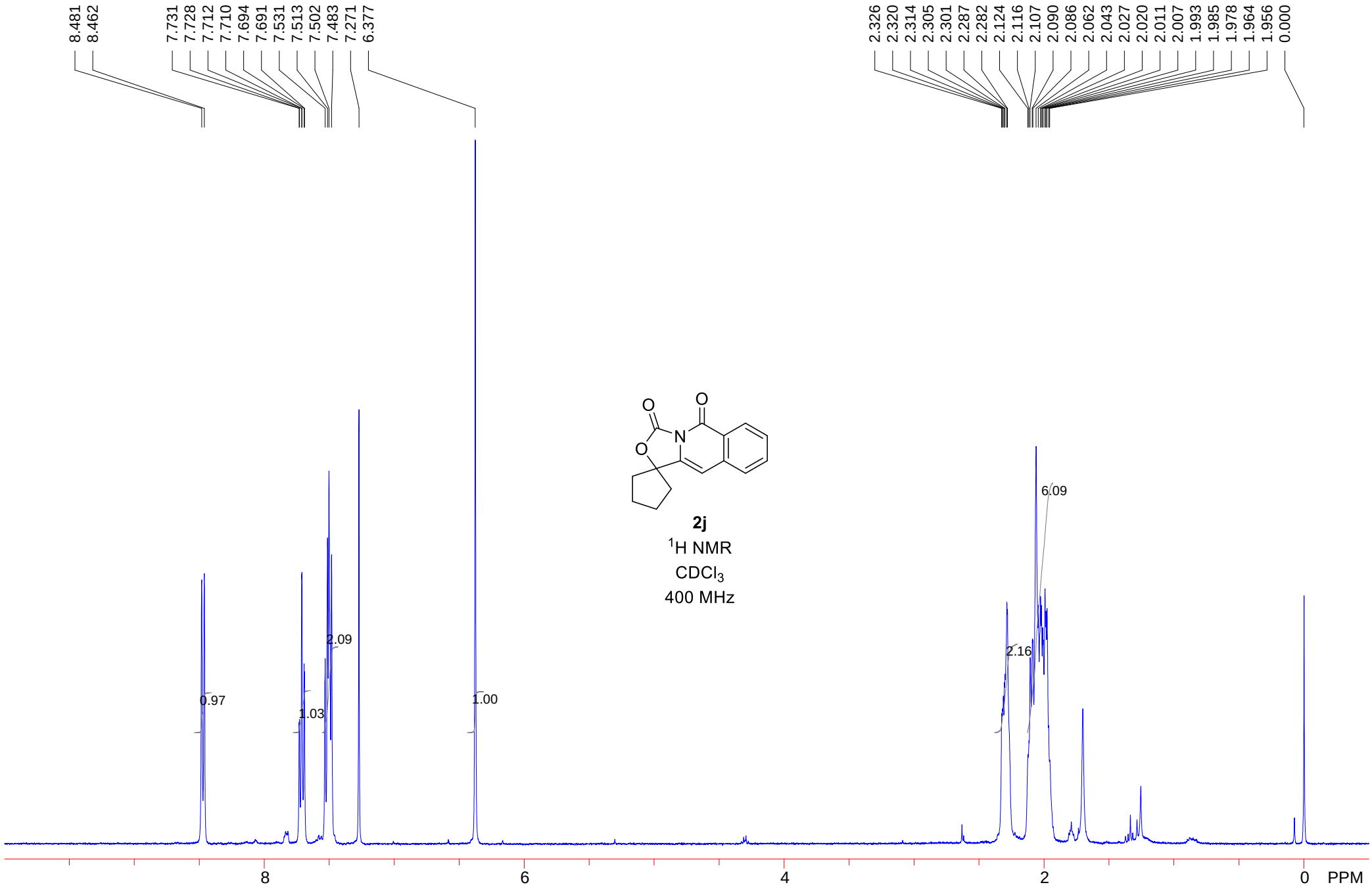
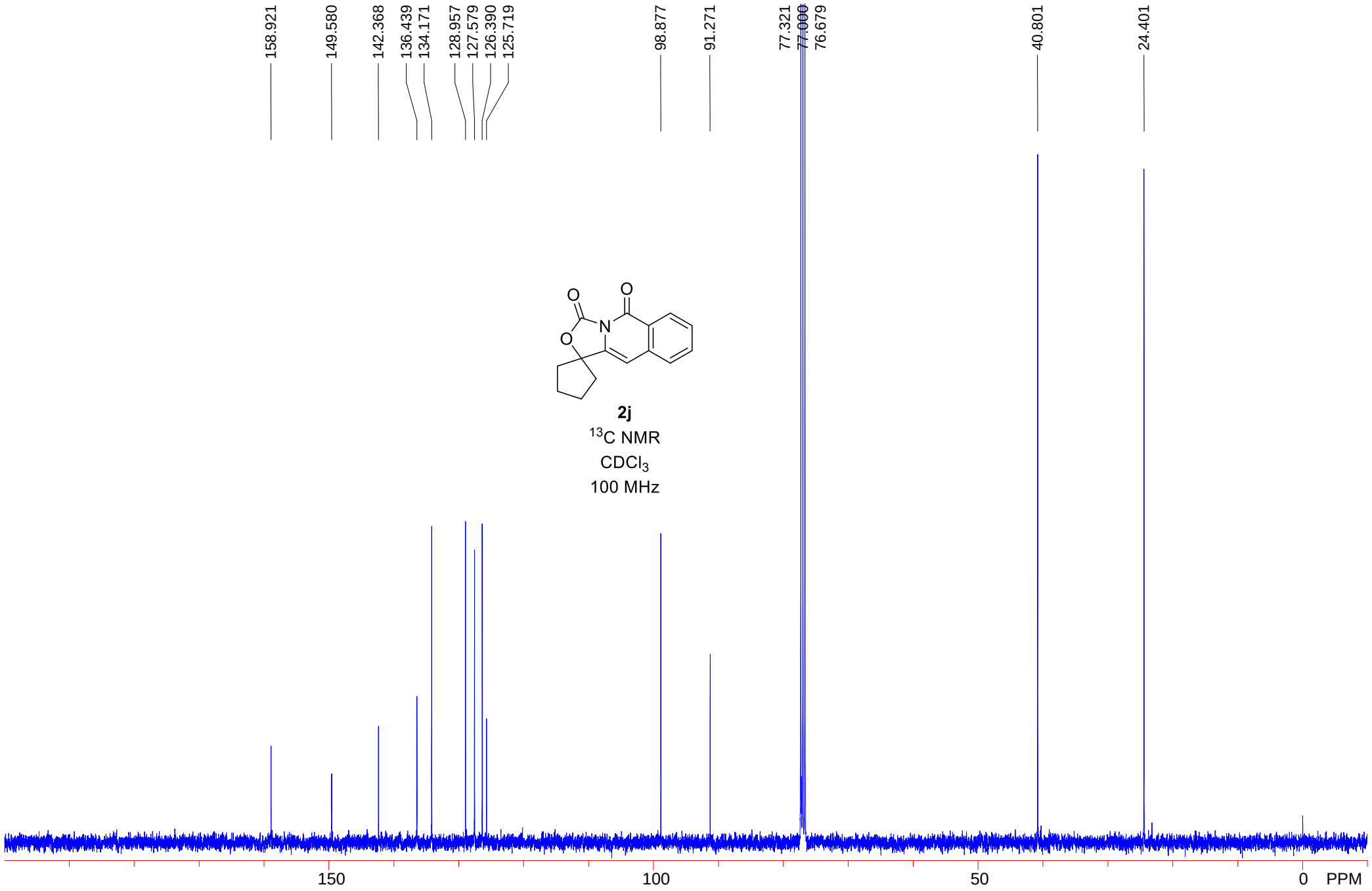
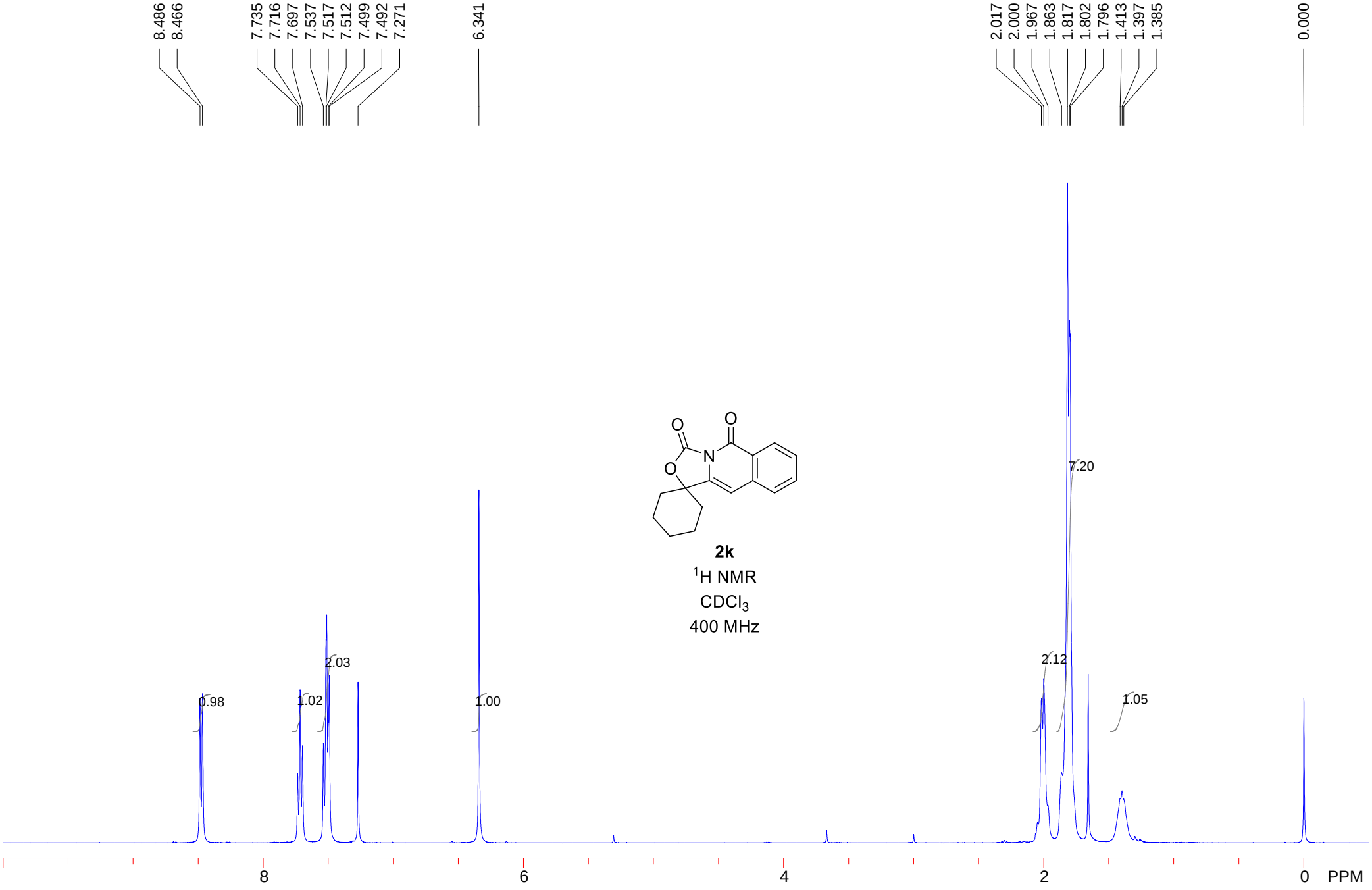
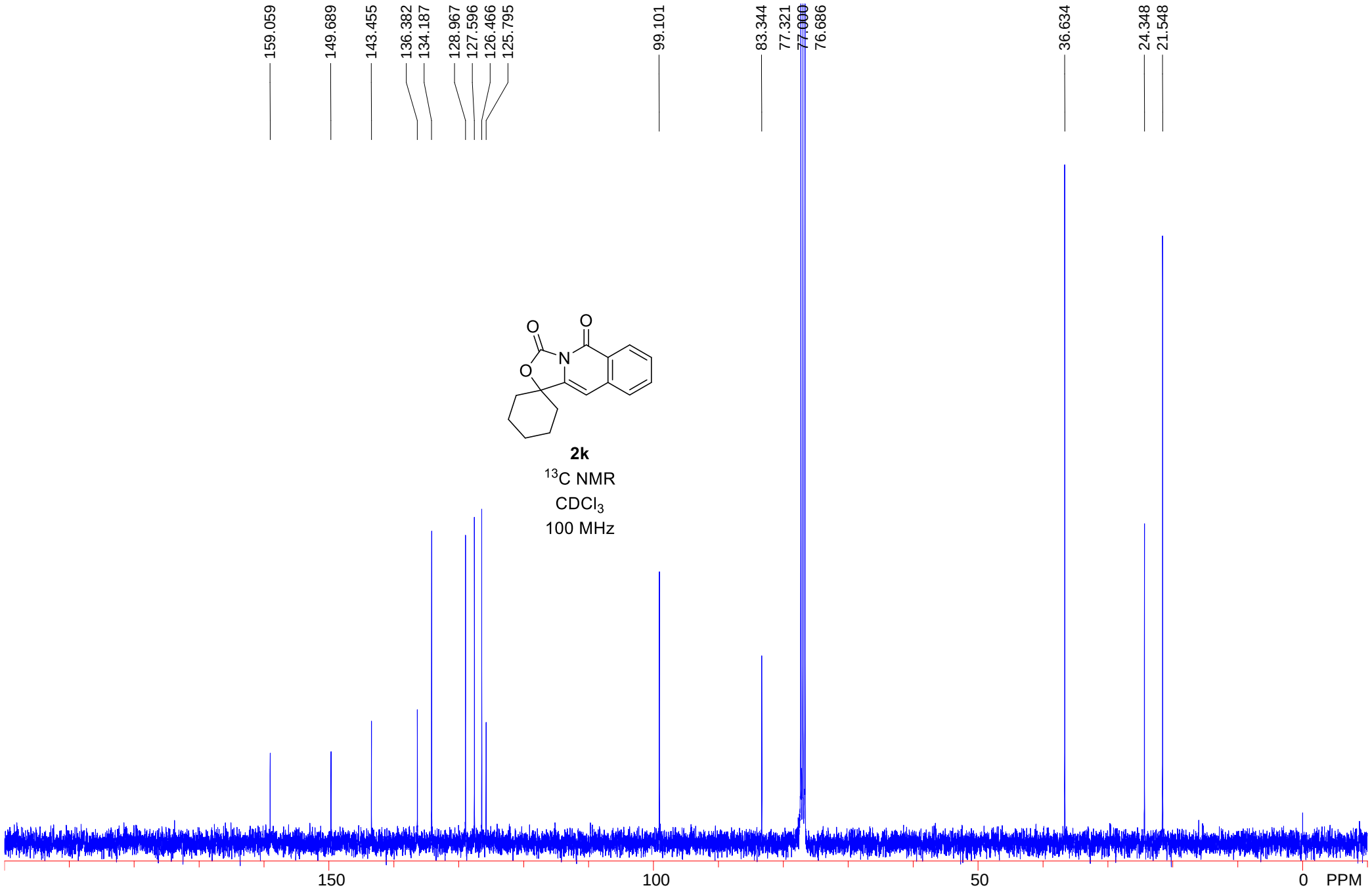
150.075
140.953
136.315
134.229
128.964
127.652
126.470
125.734
99.314
87.581
77.321
77.000
76.686
32.036
7.206
O
O
N
O
Et
Et
2i
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM


8.481
8.462
7.731
7.728
7.712
7.710
7.694
7.691
7.531
7.513
7.502
7.483
7.271
6.377
2.326
2.320
2.314
2.305
2.301
2.287
2.282
2.124
2.116
2.107
2.090
2.086
2.062
2.043
2.027
2.020
2.011
2.007
1.993
1.985
1.978
1.964
1.956
0.000
O
O
N
O
2j
1H NMR
CDCl3
400 MHz
0.97
1.03
2.09
1.00
2.16
6.09
8
6
4
2
0
PPM


158.921
149.580
142.368
136.439
134.171
128.957
127.579
126.390
125.719
98.877
91.271
77.321
77.000
76.679
40.801
24.401
O
O
N
O
2j
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM


8.486
8.466
7.735
7.716
7.697
7.537
7.517
7.512
7.499
7.492
7.271
6.341
2.017
2.000
1.967
1.863
1.817
1.802
1.796
1.413
1.397
1.385
0.000
O
O
N
O
2k
1H NMR
CDCl3
400 MHz
0.98
1.02
2.03
1.00
7.20
2.12
1.05
8
6
4
2
0
PPM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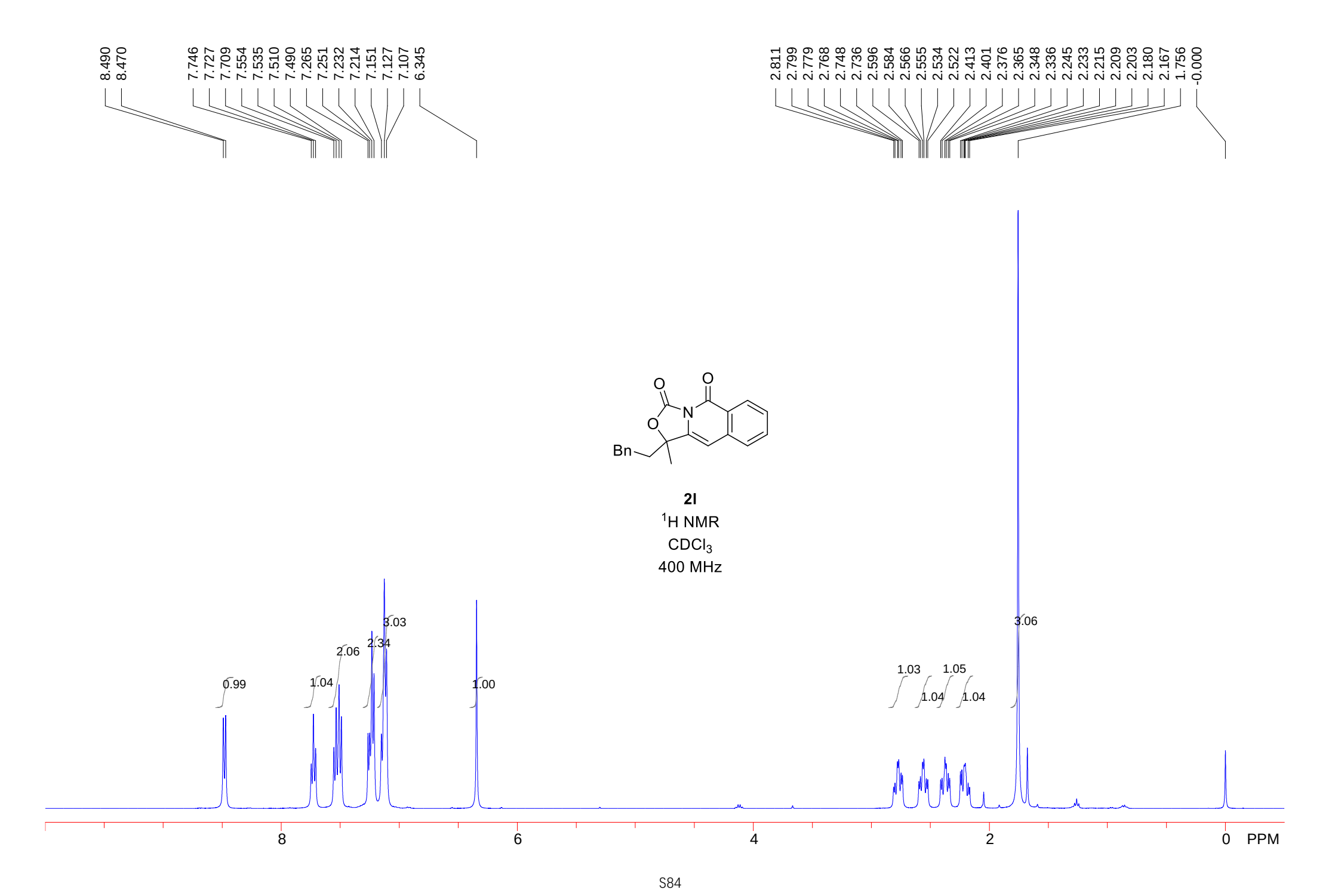
149.689
143.455
136.382
134.187
128.967
127.596
126.466
125.795
99.101
83.344
77.321
77.000
76.686
36.634
24.348
21.548
O
O
N
O
2k
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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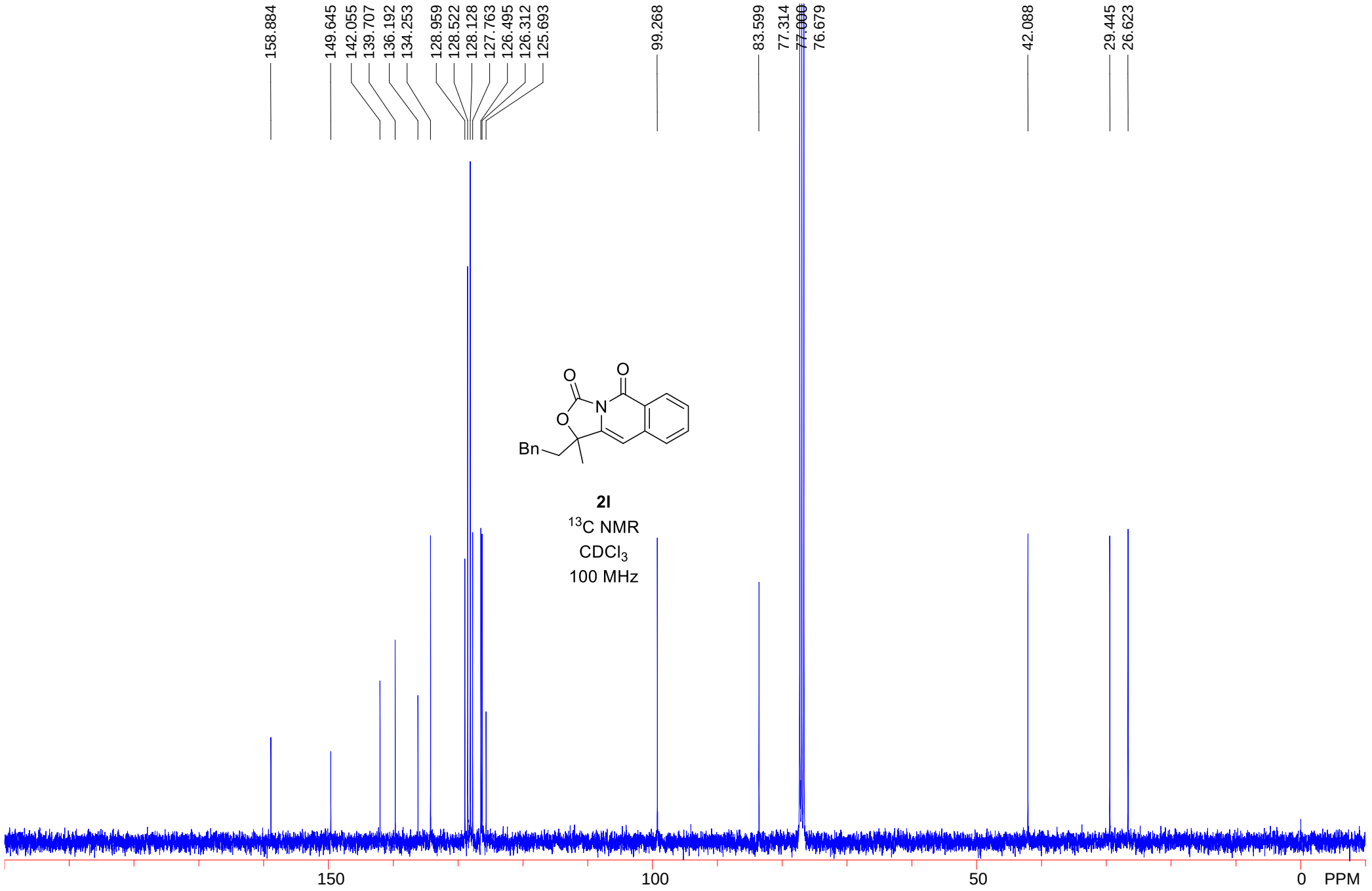
8.470
7.746
7.727
7.709
7.554
7.535
7.510
7.490
7.265
7.251
7.232
7.214
7.151
7.127
7.107
6.345
2.811
2.799
2.779
2.768
2.748
2.736
2.596
2.584
2.566
2.555
2.534
2.522
2.413
2.401
2.376
2.365
2.348
2.336
2.245
2.233
2.215
2.209
2.203
2.180
2.167
1.756
-0.000
O
O
N
O
Bn
2l
1H NMR
CDCl3
400 MHz
0.99
1.04
2.06
2.34
3.03
1.00
1.03
1.04
1.05
1.04
3.06
8
6
4
2
0
PPM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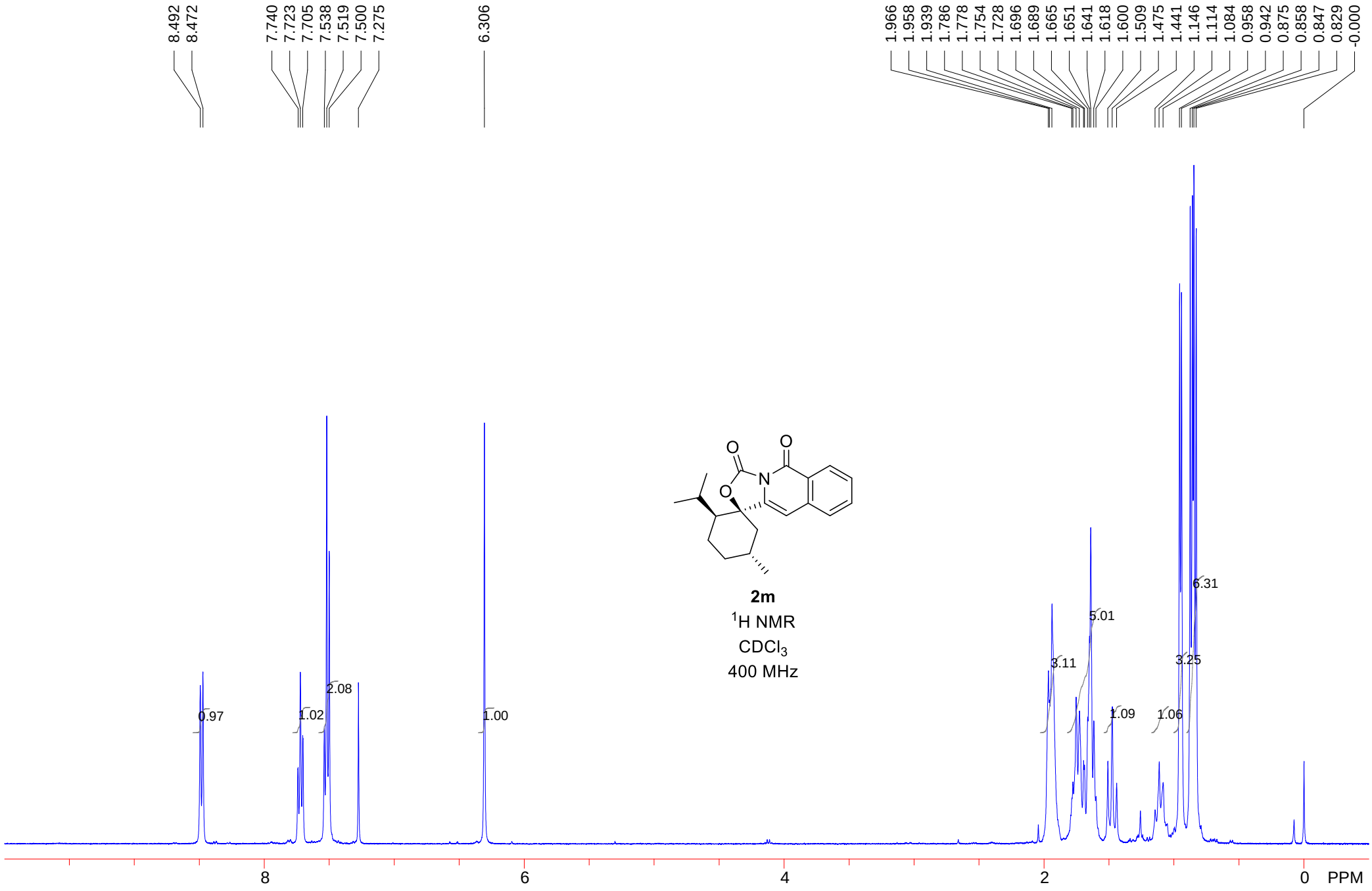
149.645
142.055
139.707
136.192
134.253
128.959
128.522
128.128
127.763
126.495
126.312
125.693
99.268
83.599
77.314
77.000
76.679
42.088
29.445
26.623
O
O
N
O
Bn
2l
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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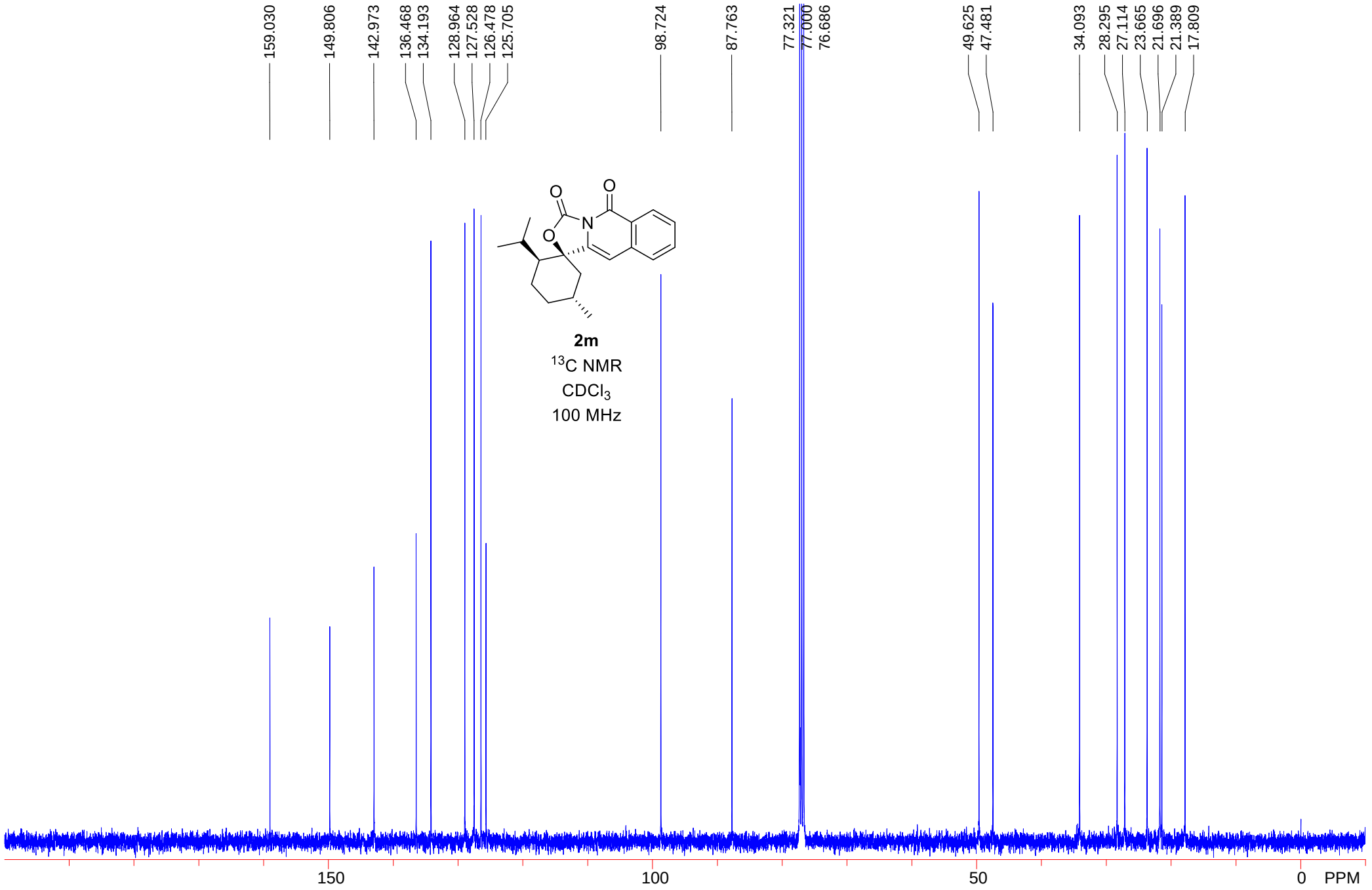
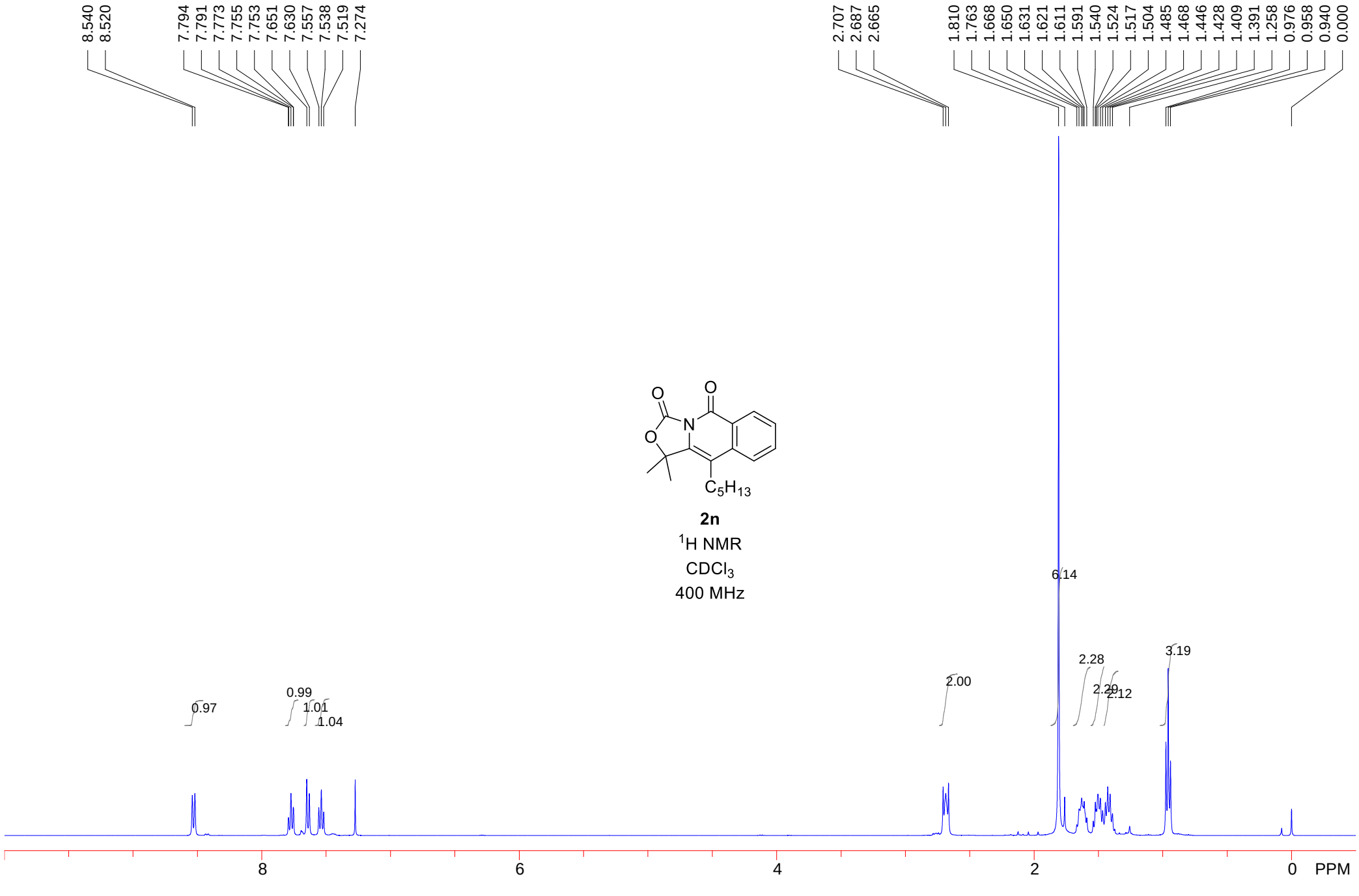
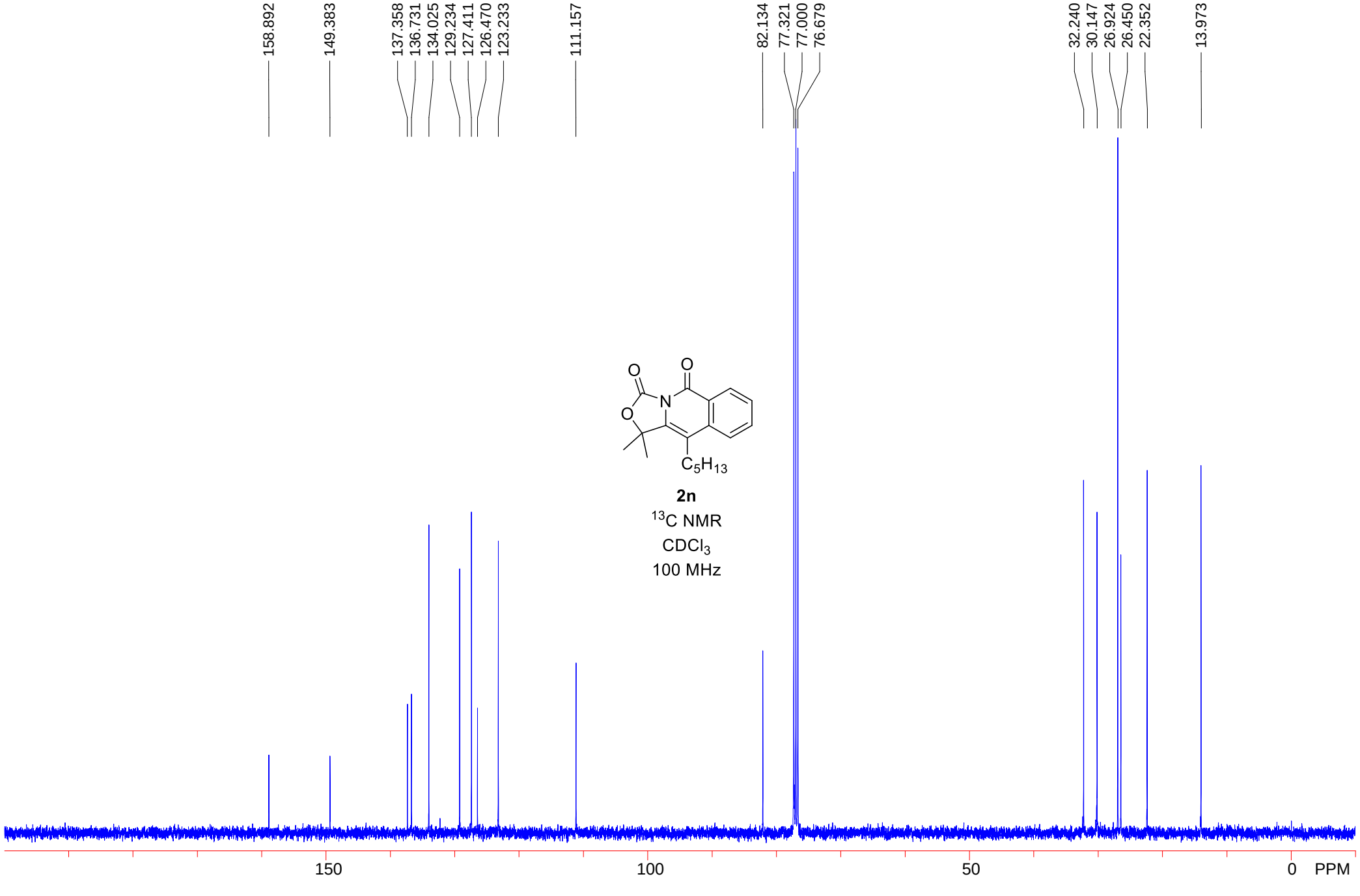
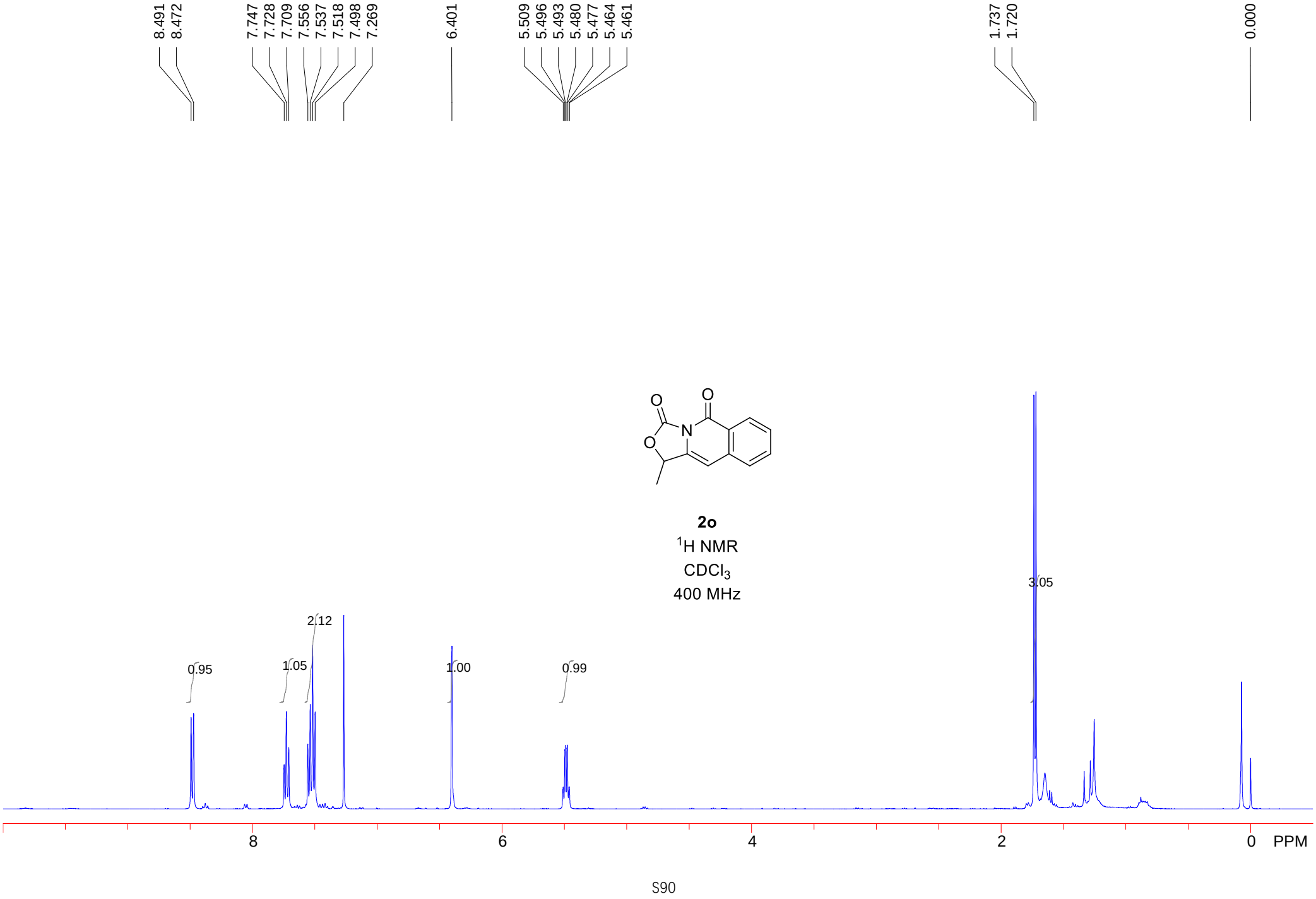
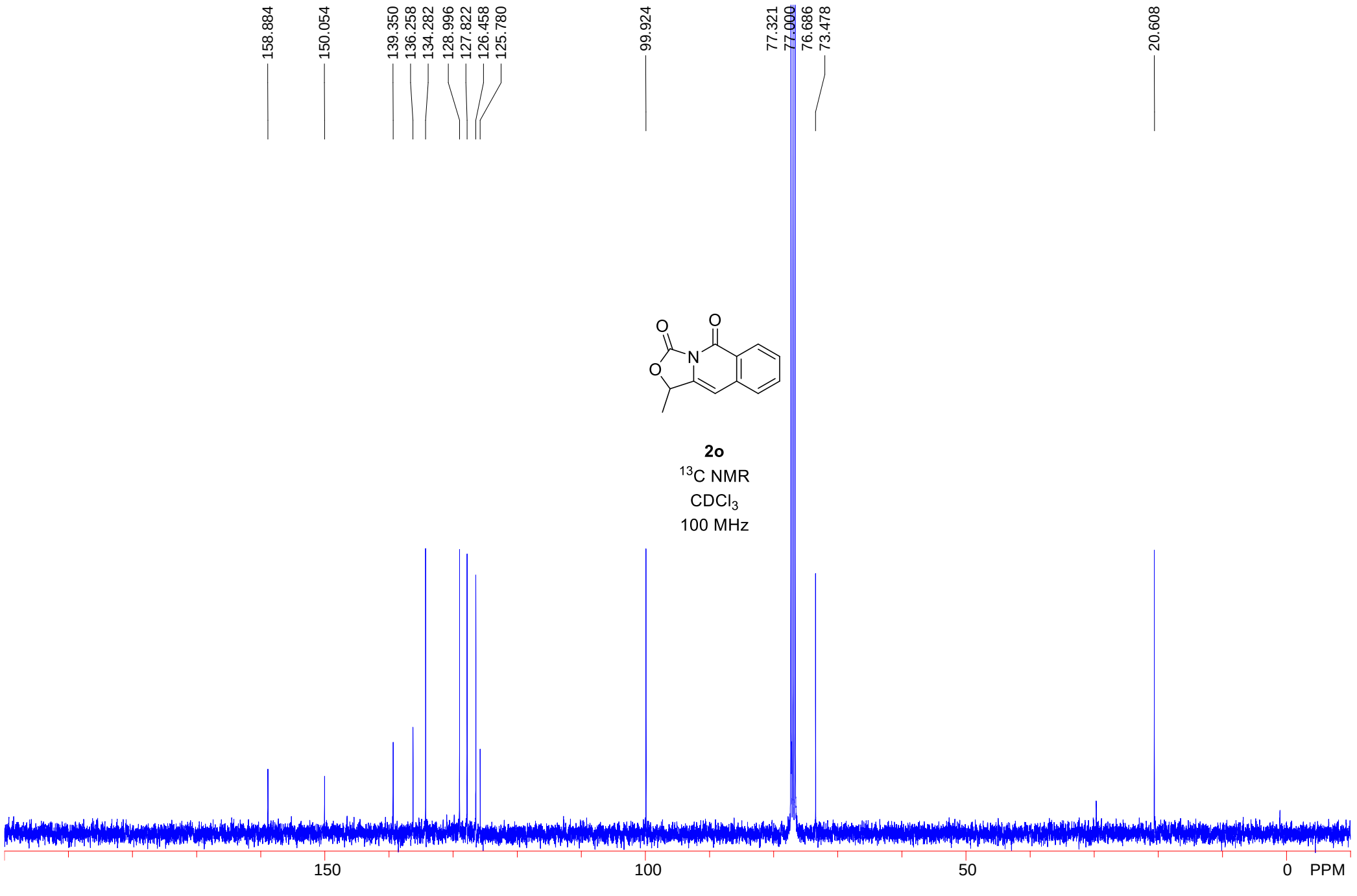
8.472
7.740
7.723
7.705
7.538
7.519
7.500
7.275
6.306
1.966
1.958
1.939
1.786
1.778
1.754
1.728
1.696
1.689
1.665
1.651
1.641
1.618
1.600
1.509
1.475
1.441
1.146
1.114
1.084
0.958
0.942
0.875
0.858
0.847
0.829
-0.000
O
O
N
O
2m
1H NMR
CDCl3
400 MHz
0.97
1.02
2.08
1.00
3.11
5.01
1.09
1.06
3.25
6.31
8
6
4
2
0
PPM

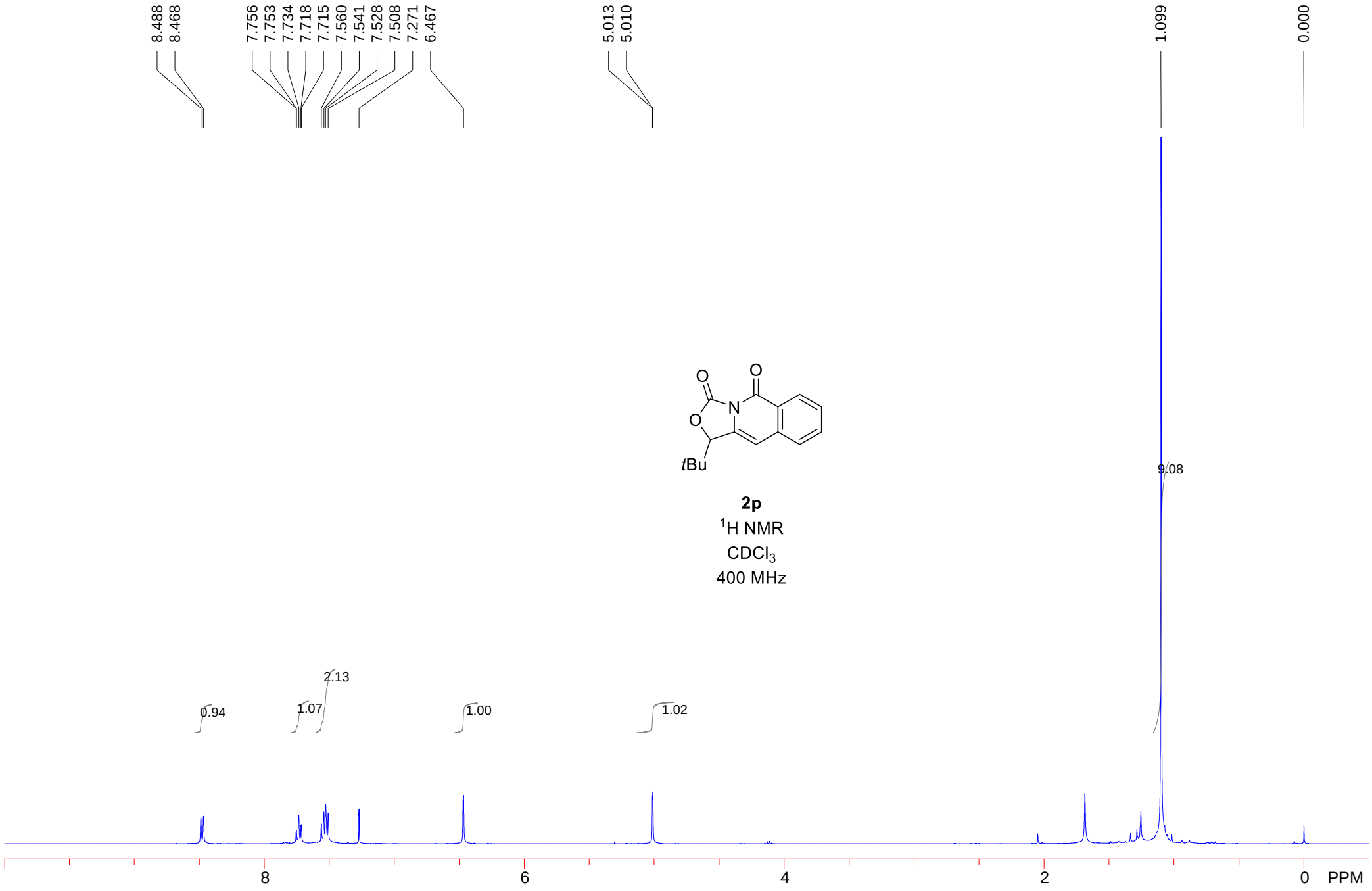

159.030
149.806
142.973
136.468
134.193
128.964
127.528
126.478
125.705
98.724
87.763
77.321
77.000
76.686
49.625
47.481
34.093
28.295
27.114
23.665
21.696
21.389
17.809
O
O
N
O
2m
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM


8.540
8.520
7.794
7.791
7.773
7.755
7.753
7.651
7.630
7.557
7.538
7.519
7.274
2.707
2.687
2.665
1.810
1.763
1.668
1.650
1.631
1.621
1.611
1.591
1.540
1.524
1.517
1.504
1.485
1.468
1.446
1.428
1.409
1.391
1.258
0.976
0.958
0.940
0.000
O
O
N
O
C5H13
2n
1H NMR
CDCl3
400 MHz
0.97
0.99
1.01
1.04
2.00
6.14
2.28
2.29
2.12
3.19
8
6
4
2
0
PPM


158.892
149.383
137.358
136.731
134.025
129.234
127.411
126.470
123.233
111.157
82.134
77.321
77.000
76.679
32.240
30.147
26.924
26.450
22.352
13.973
O
O
N
O
C5H13
2n
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM


8.491
8.472
7.747
7.728
7.709
7.556
7.537
7.518
7.498
7.269
6.401
5.509
5.496
5.493
5.480
5.477
5.464
5.461
1.737
1.720
0.000
O
O
N
O
2o
1H NMR
CDCl3
400 MHz
0.95
1.05
2.12
1.00
0.99
3.05
8
6
4
2
0
PPM


158.884
150.054
139.350
136.258
134.282
128.996
127.822
126.458
125.780
99.924
77.321
77.000
76.686
73.478
20.608
O
O
N
O
2o
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM


8.488
8.468
7.756
7.753
7.734
7.718
7.715
7.560
7.541
7.528
7.508
7.271
6.467
5.013
5.010
1.099
0.000
O
O
N
O
tBu
2p
1H NMR
CDCl3
400 MHz
9.08
2.13
1.07
0.94
1.00
1.02
8
6
4
2
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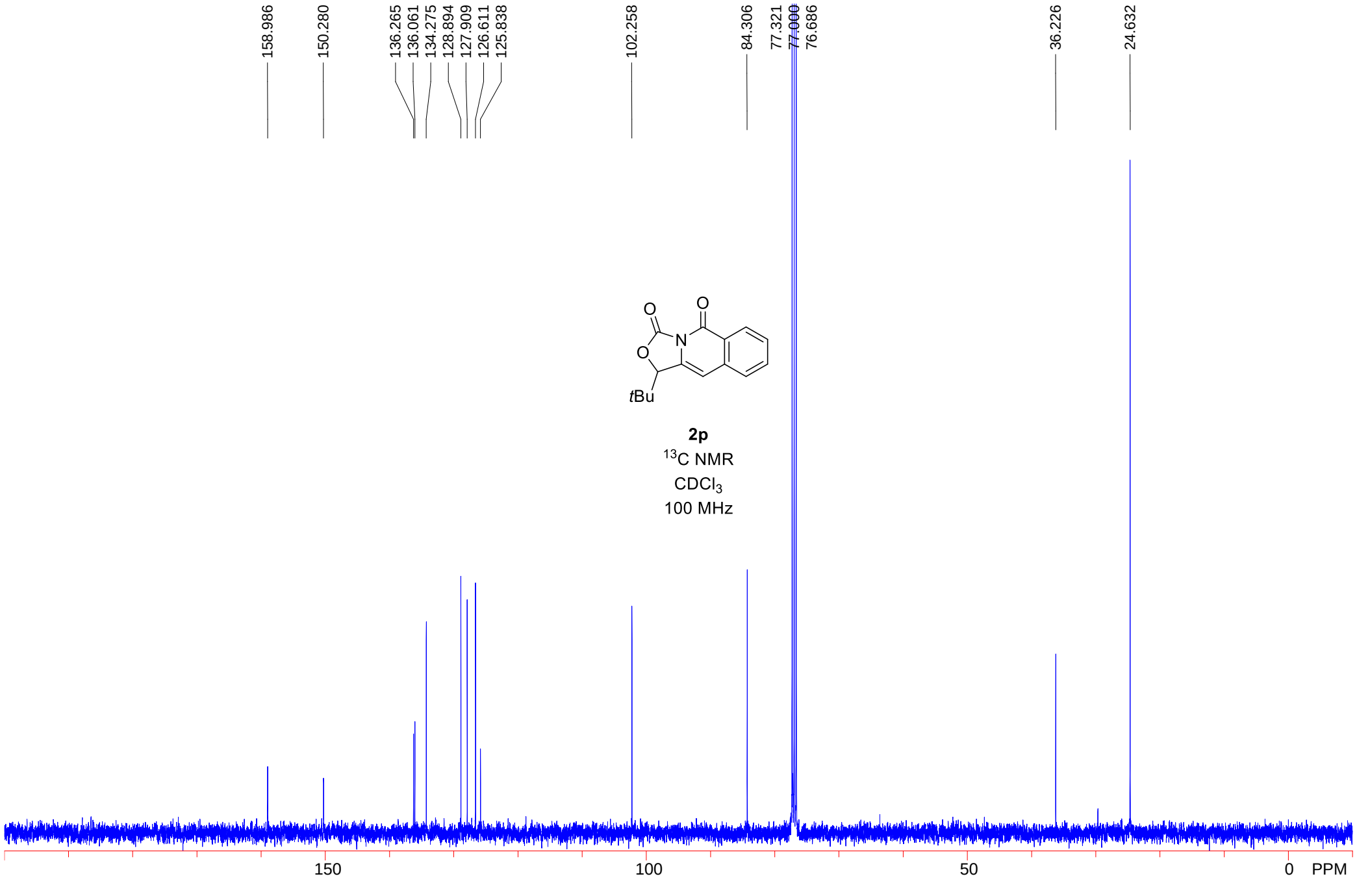
PPM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126.611
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102.258
84.306
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77.000
76.686
36.226
24.632
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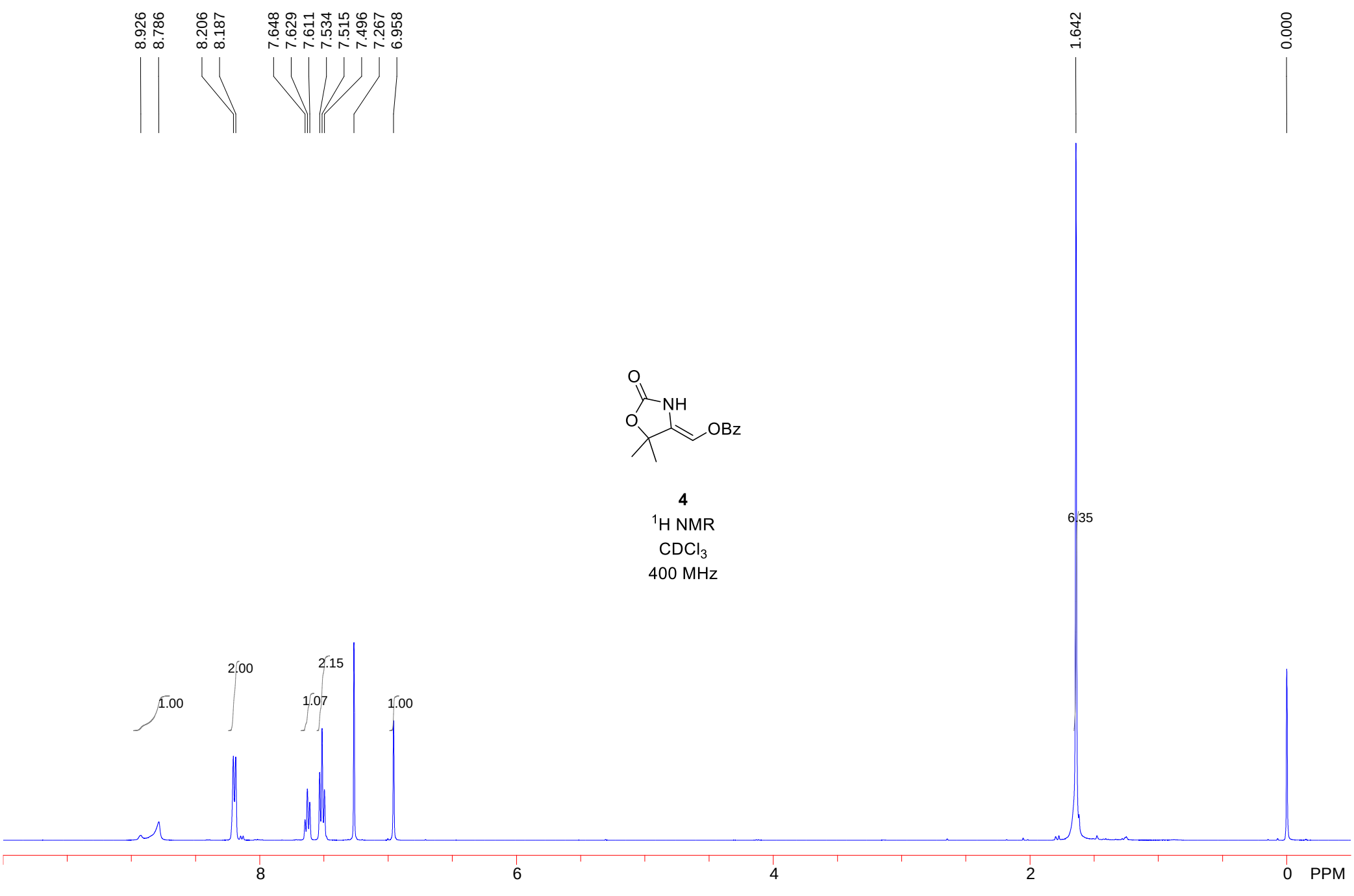
O
N
O
tBu
2p
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM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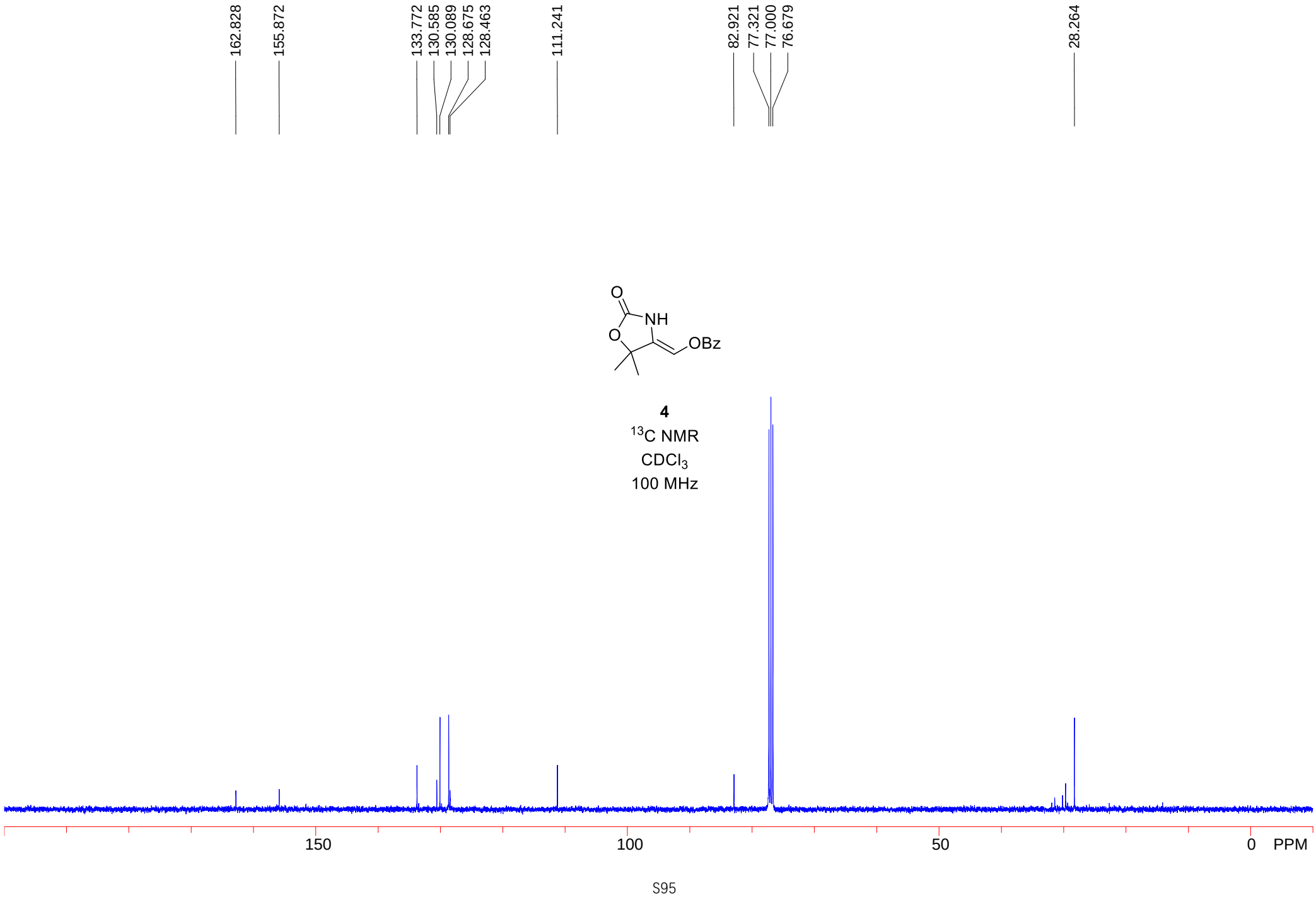
8.206
8.187
7.648
7.629
7.611
7.534
7.515
7.496
7.267
6.958
1.642
0.000
O
NH
O
OBz
4
1H NMR
CDCl3
400 MHz
6.35
1.00
2.00
1.07
2.15
1.00
8
6
4
2
0
PPM


162.828
155.872
133.772
130.585
130.089
128.675
128.463
111.241
82.921
77.321
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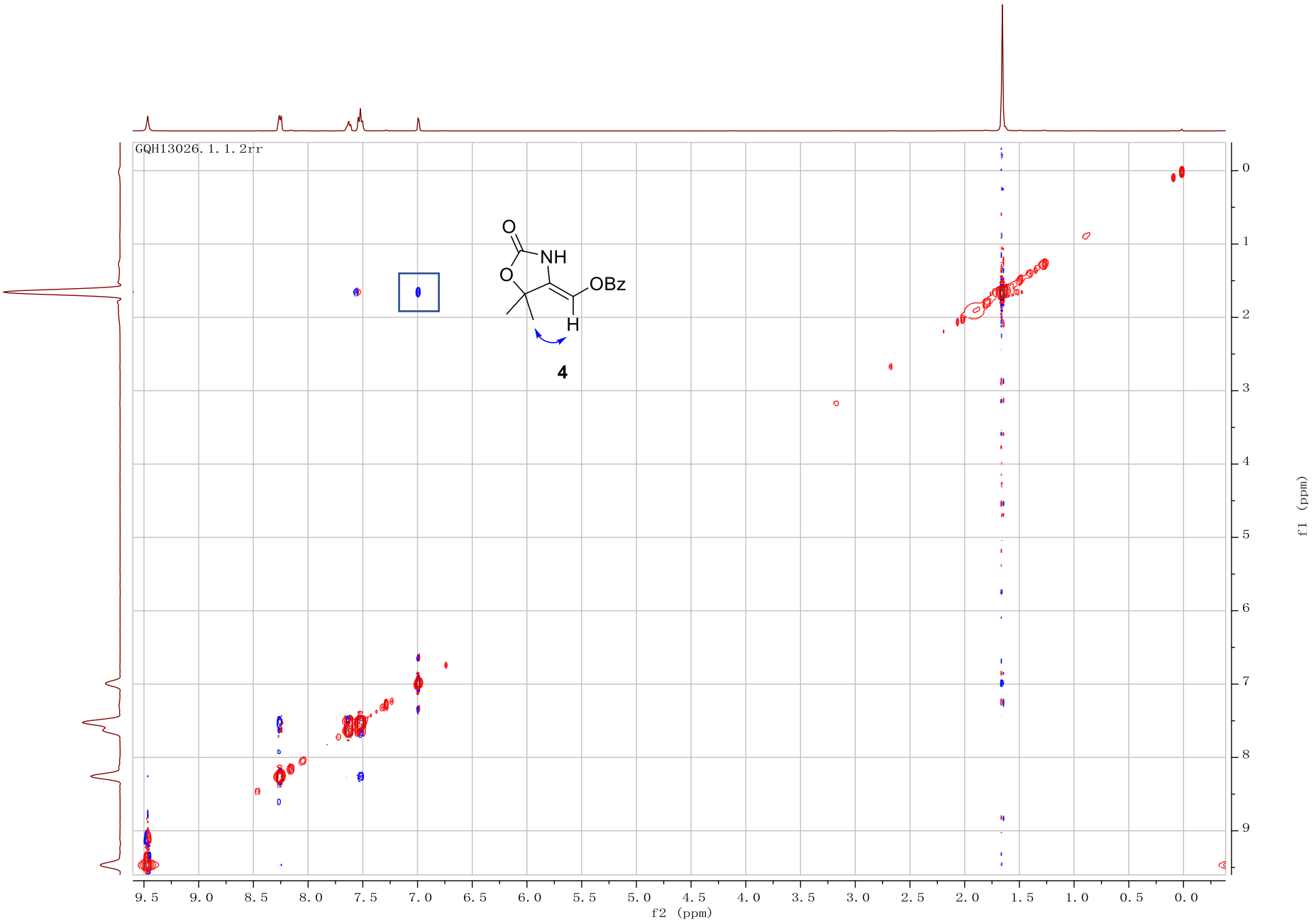
76.679
28.264
O
NH
O
OBz
4
13C NMR
CDCl3
100 MHz
150
100
50
0
PPM


GQH13026. 1. 1. 2rr
O
NH
O
OBz
H
4
f2 (ppm)
f1 (ppm)