Supporting Information

Metal-free stereoselective intramolecular oxyamination of alkynes using a robotic and rapid photocatalytic synthesis and screening system

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I. General Information and Method of the Robotic System.

THF and toluene were distilled from sodium benzophenoneketyl prior to use. MeCN [Extra Dry, with molecular sieves, Water≤50 ppm (by K.F.)], MeCN, EA, HCl, $NH_2OH{\cdot}HCl$, iPr_2NEt, Neutral Red (NR), Sodium sulfate (Na_2SO_4), Triethylamine (Et_3N), Imidazole and *N*,*N*'-Carbonyldiimidazole (CDI) 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 for the scale-up reaction. NMR spectra were recorded on a Bruker-400 instrument or Oxford instrument. ^1H 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.).

General Information of the Robotic System

The light source module consisted of 4 lasers (5.5 W, λ_{max} = 450 nm, Oxlasers, Shanghai, China) and 4 optical fibers (400-μm diameter, Lanwin Technology, Zhongshan, China) for conducting the light into the LCW photocatalytic microreactor. The reaction mixture was prepared using the liquid handling module with a syringe pump (20738325 Cavro XCalibur Pump, Tecan, Männedorf, Switzerland) and a 10-port selective valve (C5-3000EUTF, VICI, Schenkkan, Switzerland), connected with a PFA capillary (1512L, IDEX, Northbrook, USA). The LCW photocatalytic microreactor was designed as a cannula configuration with an inner fused-silica capillary (530 μm i.d. and 690 μm o.d., Refined Chromatography Co., Yongnian, China) and an outer glass capillary (1.5 mm i.d. and 2.5 mm o.d., Jingke, Guangzhou, China), installed with two polypropylene tees (3.2 mm, 1/8, Jieliante Technology Co., Beijing, China). The reaction channel and the PFA capillary were connected using unions (P-702, IDEX, Northbrook, USA) or tees (P-712, IDEX, Northbrook,

USA). In order to perform the synthesis at the set temperatures, the cooling solution was circulated in the outer capillary using a diaphragm pump (KVP004, Kamoer Fluid Technology Co., Shanghai, China), and the temperature of the circulating cooling solution was monitored in real time by a digital temperature controller (XH-W3002, Xinghe Electronic Technology Co., Suqian, China), which could control the on and off of the semiconductor chilling plate (TEC1-12706, Xinghe Electronic Technology Co., Suqian, China). The self-developed control program was written with LabView (8.0, National Instruments, Austin, USA).

Basic Operation of the Robotic System

Utilizing the self-developed control program, the operation of the 10-port selective valve, the syringe pump, the LCW photocatalytic microreactor, and the lasers was automatedly controlled, and different photocatalyst species and solvents were selected. For the optimization of photocatalysts, the desired volumes of the stock solutions (0.2 M substrate, 0.2 M internal standard, 0.02 M photocatalyst and 0.004 M DIPEA in MeCN) stored in argon were aspirated using the 10-port selective valve and the syringe pump to prepare the reaction mixtures before synthesis. After fast and homogeneous mixing, the reaction mixture was delivered into the reaction channel of the LCW photocatalytic microreactor for rapid synthesis. For the optimization of solvents, the reaction mixtures were prepared and stored in argon, and then delivered to the LCW photocatalytic microreactor for synthesis as well. Unless otherwise mentioned, the difunctionalization of alkynes were performed in the LCW photocatalytic microreactor with 2.5 min residence time under automated temperature control (e.g., 40 ± 2 °C) and uniform light irradiation from the 3 W 450 nm lasers. A parallel photoreactor (PL-SX100A, Beijing Pulinsaisi Technology Co., Beijing, China) was used for the synthesis of different substrates. After synthesis, the reaction mixture was collected, diluted and filtered. Then, the product yields of the reaction mixture were obtained using the HPLC (Vanquish Core, C_{18} column, Thermo Fisher Scientific, Waltham, USA) analysis method with a UV-Vis detector at the detection wavelength of 220 nm. During the HPLC analysis, the mobile phase A was the ultrapure water obtained from a water purification system (Barnstead MicroPure, Thermo Fisher Scientific, Waltham, USA), and the mobile phase B was MeCN.

The quantitative results of the condition screening were obtained by HPLC analysis method. For the difunctionalization of alkynes, the HPLC gradient elution method was optimized (from 30%B to 90%B) and the detection time for each sample was 15 min, which could match the time

consumed for condition screening of 14 min (**Fig. S1A**). The standard curves of the model substrate and its corresponding product were made, respectively, using 1,3,5-trimethylbenzene as the internal standard substance (**Fig. S1B, S1C**). When screening the conditions of the model substrate, only a small amount of reaction mixture (c.a. 5 μL) was collected, diluted and then analyzed by the HPLC system to obtain information such as product yields and substrate recovery.

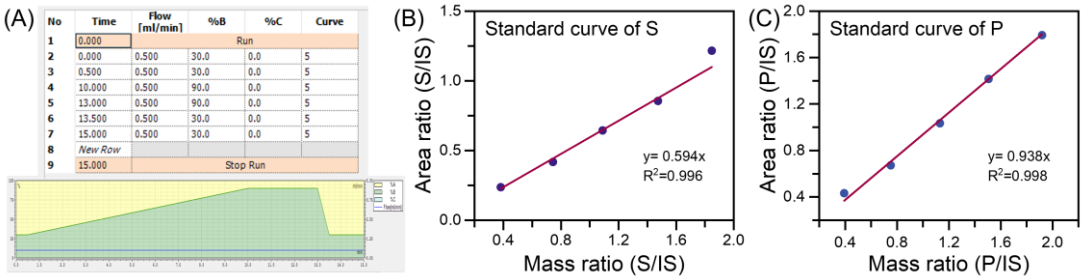


Fig. S1. Quantitative analysis using HPLC method. (A) Optimized HPLC gradient elution method. (B) Standard curve of the model substrate. (C) Standard curves of the model product.

II. Optimizations of Reaction Conditions.

It took only 14 min to complete each condition screening rapidly, among which only 2.5 min residence time was needed for synthesis, 2 min was for sample collection after synthesis, 5.5 min was for rinsing and replacing sample of the reaction channel, and 4 min was for sample preparation, including reactants introducing, diluting, mixing, and so on.

Utilizing the self-developed system for automated photocatalytic synthesis and screening, the optimized conditions and results on different photocatalysts are shown in **Figure 2**. The optimized conditions and results on different solvents are shown in **Figure S2**. In most solvents, such as MeOH, THF, DCM, DMF, acetone, dioxane, and so on, the reaction gave **2a** in 8-73% yield. The reaction using MeCN as a solvent could afford **2a** in 81% yield, so MeCN was chosen as the optimal solvent. The optimization results of the amount of DIPEA and reaction temperature are also shown in **Figure S2**. The reaction could give **2a** in 76% yield with 2 mol% DIPEA, in 45% with 1 e.q. DIPEA, and in 60% without DIPEA. What's more, the reaction could deliver **2a** in 59% yield at 20 °C. As a result, 10 mol% was chosen as the appropriate DIPEA amount, and 40 °C was chosen as the optimal reaction temperature. The reaction was also carried out using MeCN (SCR) and the yield of **2a** was

74%. Addition of 10 e.q. of water to the reaction mixture slightly decreased the yield of **2a** to 59%, suggesting that the synthesis strategy can tolerate the presence of water to some extent.

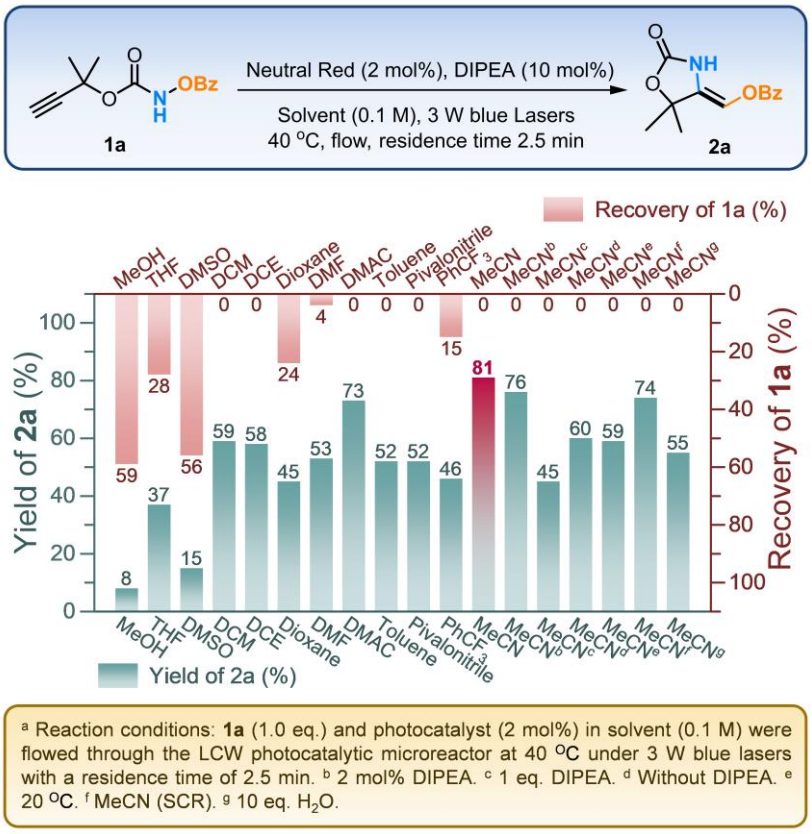
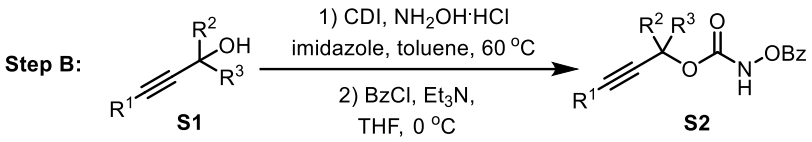


Fig. S2. Optimizations of solvents, DIPEA amount and reaction temperature. [a]

III. Procedures for the Synthesis of Starting Materials

Starting materials were prepared by the following steps:

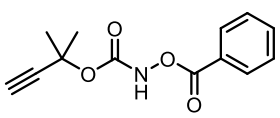
Step A. Substituted propargyl alcohol **S1** were prepared according to the previously reported procedures.[1]



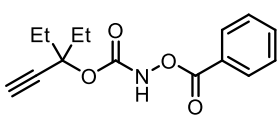
Step B. Alkyl benzoyloxycarbamate **S2** were prepared according to a reported procedure.[1] A 250

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 mixture 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 mixture was concentrated by rotary evaporation. Then the resulting suspension was dissolved in HCl (60 mL, 1 M), the aqueous phase was 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 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 **S2**.

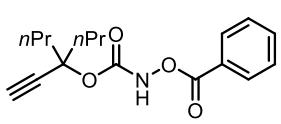


2-Methylbut-3-yn-2-yl (benzoyloxy)carbamate (1a): 2.85 g, 77% yield, white soild; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.43 (brs, 1H), 8.14-8.04 (m, 2H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 2.60 (s, 1H), 1.74 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.2, 129.9, 128.6, 126.7, 83.7, 74.4, 73.2, 28.9; The characterization data of the product is consistent with the reported literature.[1]

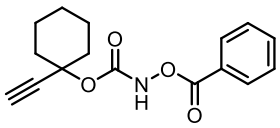


3-Ethylpent-1-yn-3-yl (benzoyloxy)carbamate (1b): 4.46 g, 85% yield, white soild, m.p. = 95-96 °C, IR (neat): 3300, 2980, 1746, 1456, 1231 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.43 (brs, 1H), 8.15-8.02 (m, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.47 (dd, *J* = 7.6 Hz, 2H), 2.62 (s, 1H), 2.17-2.04 (m, 2H), 2.04-1.91

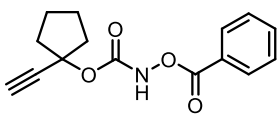
(m, 2H), 1.01 (t, J = 7.4 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.1, 129.8, 128.6, 126.7, 82.2, 81.9, 74.9, 30.9, 8.1; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1049.



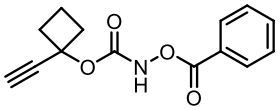
4-Ethynylheptan-4-yl (benzoyloxy)carbamate (1c): 4.22 g, 70% yield, white soild, m.p. = 63-66 ºC, IR (neat): 3300, 2923, 1747, 1453, 1233 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.31 (brs, 1H), 8.10 (d, J = 7.4 Hz, 2H), 7.63 (t, J = 7.6 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 2.62 (s, 1H), 2.12-1.98 (m, 2H), 1.96-1.84 (m, 2H), 1.56-1.42 (m, 4H), 0.94 (t, J = 7.4 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.1, 129.9, 128.6, 126.7, 82.4, 81.4, 74.8, 40.5, 17.2, 13.9; HRMS (ESI) calculated for $[C_{17}H_{21}NNaO_4]^+$ ($M+Na^+$) requires m/z 326.1363, found: m/z 326.1364.



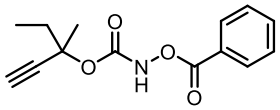
1-Ethynylcyclohexyl (benzoyloxy)carbamate (1d): 4.05 g, 64% yield, white soild, m.p. = 135-137 ºC, IR (neat): 3294, 2936, 2860, 1743, 1451, 1227 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.38 (brs, 1H), 8.13-8.27 (m, 2H), 7.67-6.60 (m, 1H), 7.48 (t, J = 7.6 Hz, 2H), 2.67 (s, 1H), 2.21-2.09 (m, 2H), 2.22-1.89 (m, 2H), 1.72-1.57 (m, 5H), 1.55-1.46 (m, 1H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.4, 134.2, 129.9, 128.6, 126.7, 82.6, 77.9, 75.0, 36.9, 24.8, 22.3; HRMS (ESI) calculated for $[C_{16}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 310.1050, found: m/z 310.1051.



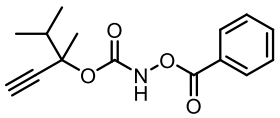
1-Ethynylcyclopentyl (benzoyloxy)carbamate (1e): 3.17 g, 77% yield, white soild, m.p. = 110-112 ºC, IR (neat): 3291, 2957, 1744, 1452, 1234 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.31 (brs, 1H), 8.10 (d, J = 7.6 Hz, 2H), 7.64 (t, J = 7.6 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 2.64 (s, 1H), 2.46-2.26 (m, 2H), 2.23-2.13 (m, 2H), 1.90-1.62 (m, 4H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.8, 134.2, 129.9, 128.7, 126.6, 83.0, 82.9, 73.9, 40.3, 23.1; HRMS (ESI) calculated for $[C_{15}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 296.0893, found: m/z 296.0894.



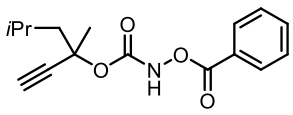
1-Ethynylcyclobutyl (benzoyloxy)carbamate (1f): 3.74 g, 41% yield, white soild, m.p. = 100-102 °C, IR (neat): 3271, 1740, 1453, 1229, 1103 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.39 (brs, 1H), 8.13-8.07 (m, 2H), 7.67-7.60 (m, 1H), 7.53-7.45 (m, 2H), 2.66 (s, 1H), 2.65-2.57 (m, 2H), 2.56-2.45 (m, 2H), 2.04-1.89 (m, 2H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.7, 154.4, 134.3, 129.9, 128.7, 126.5, 82.9, 73.6, 73.5, 36.4, 14.1; HRMS (ESI) calculated for $[C_{14}H_{13}NNaO_4]^+$ ($M+Na^+$) requires m/z 282.0737, found: m/z 282.0738.



3-Methylpent-1-yn-3-yl (benzoyloxy)carbamate (1g): 0.88 g, 90% yield, white soild, m.p. = 90-91 °C, IR (neat): 3289, 2976, 1744, 1455, 1235 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.31 (brs, 1H), 8.10 (d, *J* = 7.2 Hz, 2H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 2.62 (s, 1H), 2.10-1.97 (m, 1H), 1.97-1.84 (m, 1H), 1.75 (s, 3H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.2, 129.9, 128.7, 126.7, 82.7, 78.3, 74.1, 34.4, 26.0, 8.4; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 284.2893, found: m/z 284.0895.

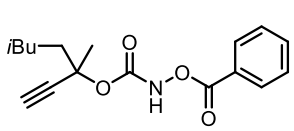


3,4-Dimethylpent-1-yn-3-yl (benzoyloxy)carbamate (1h): 0.44 g, 77% yield, white soild, m.p. = 88-90 °C, IR (neat): 3274, 2973, 1746, 1455, 1236 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.30 (brs, 1H), 8.13-8.06 (m, 1H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 2.61 (s, 1H), 2.30-2.18 (m, 1H), 1.74 (s, 3H), 1.05 (d, *J* = 6.8 Hz, 3H), 1.02 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.9, 154.6, 134.2, 129.9, 128.7, 126.7, 81.8, 81.5, 74.8, 37.4, 23.3, 17.3, 17.0; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1050.

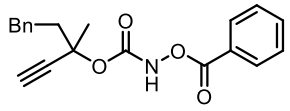


3,5-Dimethylhex-1-yn-3-yl (benzoyloxy)carbamate (1i): 1.33 g, 74% yield, white soild, m.p. = 71-73 °C, IR (neat): 3302, 2957, 1747, 1457, 1234 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.28 (brs, 1H), 8.10 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 2.63 (s, 1H), 2.06-1.87 (m, 2H), 1.83-1.74 (m, 4H), 0.98 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.2, 129.9, 128.6, 126.7, 83.1, 77.9, 74.4, 49.4, 27.3, 24.8, 24.0, 23.7; HRMS (ESI) calculated for

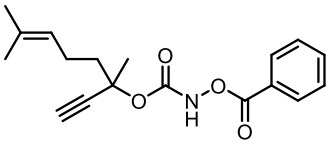
$[C_{16}H_{19}NNaO_4]^+$ (M+Na^+) requires m/z 312.1206, found: m/z 312.1208.



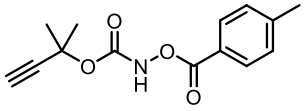
3,6-Dimethylhept-1-yn-3-yl (benzoyloxy)carbamate (1j): 0.23 g, 59% yield, white soild, m.p. = 49-52 °C, IR (neat): 3290, 2956, 1748, 1459, 1234 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.29 (brs, 1H), 8.10 (d, *J* = 7.2 Hz, 2H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 2.61 (s, 1H), 2.05-1.93 (m, 1H), 1.90-1.80 (m, 1H), 1.76 (s, 3H), 1.60-1.47 (m, 1H), 1.44-1.31 (m, 2H), 0.92-0.85 (m, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.2, 129.9, 128.7, 126.7, 83.0, 77.9, 74.1, 39.4, 32.8, 27.9, 26.5, 22.5, 22.4; HRMS (ESI) calculated for $[C_{17}H_{21}NNaO_4]^+$ (M+Na^+) requires m/z 326.1363, found: m/z 326.1361.



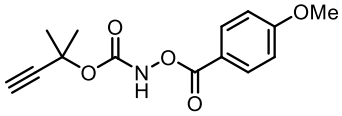
3-Methyl-5-phenylpent-1-yn-3-yl (benzoyloxy)carbamate (1k): 7.16 g, 85% yield, white soild, m.p. = 102-103 °C, IR (neat): 3300, 2922, 1747, 1453, 1233 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.36 (brs, 1H), 8.15-8.04 (m, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.26 (t, *J* = 7.6 Hz, 2H), 7.21-7.10 (m, 3H), 2.83 (t, *J* = 8.4 Hz, 2H), 2.68 (s, 1H), 2.35-2.22 (m, 1H), 2.20-2.07 (m, 1H), 1.82 (s, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 141.0, 134.2, 129.9, 128.7, 128.39, 128.37, 126.6, 126.0, 82.5, 77.3, 74.6, 43.4, 30.5, 26.6; HRMS (ESI) calculated for $[C_{20}H_{19}NNaO_4]^+$ (M+Na^+) requires m/z 360.1206, found: m/z 360.1204.



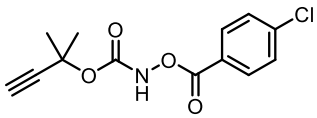
3,7-Dimethyloct-6-en-1-yn-3-yl (benzoyloxy)carbamate (1l): 5.09 g, 78% yield, white soild, m.p. = 55-57 °C, IR (neat): 3295, 2924, 1747, 1452, 1234 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.31 (brs, 1H), 8.10 (d, *J* = 8.0 Hz, 2H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 5.09 (t, *J* = 6.8 Hz, 1H), 2.63 (s, 1H), 2.25-2.13 (m, 2H), 2.07-1.96 (m, 1H), 1.92-1.81 (m, 1H), 1.77 (s, 3H), 1.68 (s, 3H), 1.59 (s, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.8, 154.5, 134.2, 132.5, 129.9, 128.7, 126.7, 122.8, 82.7, 77.6, 74.3, 41.4, 26.5, 25.6, 22.8, 17.6; HRMS (ESI) calculated for $[C_{18}H_{21}NNaO_4]^+$ (M+Na^+) requires m/z 338.1363, found: m/z 338.1366.



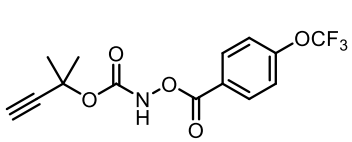
2-Methylbut-3-yn-2-yl ((4-methylbenzoyl)oxy)carbamate (1m): 0.47 g, 60% yield, white soild, m.p. = 74-76 °C, IR (neat): 3287, 1741, 1611, 1465, 1237 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.31 (brs, 1H), 7.99 (d, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 7.6 Hz, 2H), 2.60 (s, 1H), 2.43 (s, 3H), 1.75 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.9, 154.5, 145.2, 130.0, 129.4, 123.8, 83.7, 74.4, 73.2, 28.9, 21.8; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 284.0893, found: m/z 284.0895.



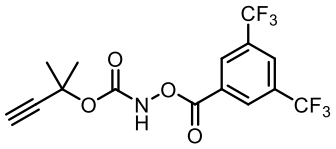
2-Methylbut-3-yn-2-yl ((4-methoxybenzoyl)oxy)carbamate (1n): 0.48 g, 58% yield, colorless oil, IR (neat): 3275, 1739, 1606, 1511, 1463, 1242 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.32 (brs, 1H), 8.05 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 3.88 (s, 3H), 2.60 (s, 1H), 1.75 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.5, 164.3, 154.6, 132.1, 118.8, 114.0, 83.8, 74.4, 73.1, 55.5, 28.9; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_5]^+$ ($M+Na^+$) requires m/z 300.0842, found: m/z 300.0843.



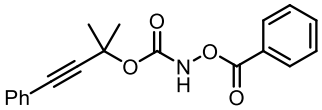
2-Methylbut-3-yn-2-yl ((4-chlorobenzoyl)oxy)carbamate (1o): 0.76 g, 83% yield, white soild, m.p. = 76-77 °C, IR (neat): 3300, 1745, 1596, 1483, 1239 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.30 (brs, 1H), 8.04 (d, *J* = 7.2 Hz, 2H), 7.47 (d, *J* = 7.2 Hz, 2H), 2.61 (s, 1H), 1.75 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.0, 154.4, 140.9, 131.3, 129.1, 125.1, 83.6, 74.7, 73.3, 28.9; HRMS (ESI) calculated for $[C_{13}H_{12}ClNNaO_4]^+$ ($M+Na^+$) requires m/z 304.0347, found: m/z 304.0348.



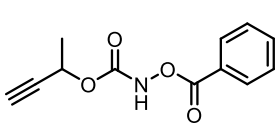
2-Methylbut-3-yn-2-yl ((4-(trifluoromethoxy)benzoyl)oxy)carbamate (1p): 0.86 g, 89% yield, white soild, m.p. = 76-78 °C, IR (neat): 3299, 1749, 1607, 1471, 1241 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.30 (brs, 1H), 8.16 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 2.61 (s, 1H), 1.75 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 164.7, 154.4, 153.5, 132.1, 124.9, 120.4, 120.2 (q, *J* = 259.5 Hz), 83.6, 74.7, 73.3, 28.9; ^{19}F NMR: (376 MHz, $CDCl_3$) δ -57.6; HRMS (ESI) calculated for $[C_{14}H_{12}F_3NNaO_5]^+$ ($M+Na^+$) requires m/z 356.0560, found: m/z 356.0561.



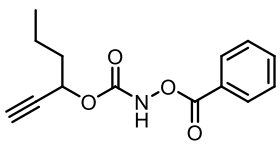
2-Methylbut-3-yn-2-yl ((3,5-bis(trifluoromethyl)benzoyl)oxy)carbamate (1q): 0.63 g, 82% yield, white soild, m.p. = 75-77 °C, IR (neat): 3274, 1752, 1382, 1282, 1222 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.55 (s, 2H), 8.40 (brs, 1H), 8.15 (s, 1H), 2.63 (s, 1H), 1.77 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 163.4, 154.1, 132.3 (q, *J* = 34.2 Hz), 130.1(q, *J* = 3.0 Hz), 129.0, 127.5 (q, *J* = 3.7 Hz), 122.6 (q, *J* = 271.2 Hz), 83.4, 75.1, 73.5, 28.8; ^{19}F NMR: (376 MHz, $CDCl_3$) δ -63.0; HRMS (ESI) calculated for $[C_{15}H_{11}F_6NNaO_4]^+$ ($M+Na^+$) requires m/z 406.0484, found: m/z 406.0482.



2-Methyl-4-phenylbut-3-yn-2-yl (benzoyloxy)carbamate (1r): 1.55 g, 48% yield, white soild, m.p. = 111-114 °C, IR (neat): 2921, 2855, 1748, 1456, 1235 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.36 (brs, 1H), 8.14-8.08 (m, 2H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.52-7.42 (m, 4H), 7.33-7.25 (m, 3H), 1.84 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.9, 154.5, 134.2, 131.9, 129.9, 128.7, 128.5, 128.1, 126.7, 122.3, 89.1, 84.8, 75.5, 29.1; HRMS (ESI) calculated for $[C_{19}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 346.1050, found: m/z 346.1051.

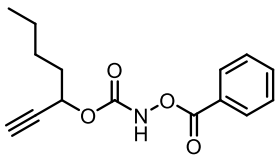


But-3-yn-2-yl (benzoyloxy)carbamate (1s): 6.00 g, 74% yield, white soild, m.p. = 82-84 °C, IR (neat): 3275, 2925, 1743, 1454, 1232 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.50 (brs, 1H), 8.13-8.06 (m, 2H), 7.68-7.60 (m, 1H), 7.52-7.44 (m, 2H), 5.53-5.45 (m, 1H), 2.54 (d, *J* = 2.0 Hz, 1H), 1.57 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.6, 155.3, 134.3, 129.9, 128.7, 126.4, 81.0, 74.0, 62.9, 21.2; HRMS (ESI) calculated for $[C_{12}H_{11}NNaO_4]^+$ ($M+Na^+$) requires m/z 256.0580, found: m/z 256.0581.

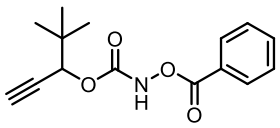


Hex-1-yn-3-yl (benzoyloxy)carbamate (1t): 1.62 g, 69% yield, white soild, m.p. = 63-66 °C, IR (neat): 3292, 2963, 1743, 1456, 1232 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.41 (brs, 1H), 8.10 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.2 Hz, 2H), 5.41 (t, *J* = 6.4 Hz, 1H), 2.52 (s, 1H), 1.90-1.75 (m, 2H), 1.66-1.42 (m, 2H), 0.95 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.7, 155.5, 134.3, 130.0, 128.7, 126.5, 80.2, 74.6, 66.5, 36.6, 18.0, 13.5; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_4]^+$

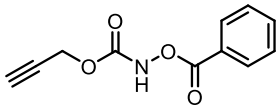
($M+Na^+$) requires m/z 284.0893, found: m/z 284.0892.



Hept-1-yn-3-yl (benzoyloxy)carbamate (1u): 18.29 g, 84% yield, white soild, m.p. = 49-52 ºC, IR (neat): 3291, 2926, 1741, 1456, 1230 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.52 (brs, 1H), 8.13-8.06 (m, 2H), 7.52-7.44 (m, 1H), 7.52-7.45 (m, 2H), 5.40 (td, *J* = 6.8, 2.0 Hz, 1H), 2.54 (d, *J* = 2.0 Hz, 1H), 1.91-1.76 (m, 2H), 1.50-1.28 (m, 4H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.7, 155.5, 134.3, 129.9, 128.7, 126.5, 80.2, 74.6, 66.6, 34.3, 26.7, 22.1, 13.8; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1052.



4,4-Dimethylpent-1-yn-3-yl (benzoyloxy)carbamate (1v): 4.23 g, 77% yield, white soild, m.p. = 50-53 ºC, IR (neat): 3296, 2965, 1741, 1456, 1370, 1231 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.59 (brs, 1H), 8.12-8.07 (m, 2H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 5.14 (d, *J* = 2.4 Hz, 1H), 2.51 (d, *J* = 2.0 Hz, 1H), 1.03 (s, 9H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.7, 155.8, 134.2, 129.9, 128.7, 126.5, 78.9, 75.2, 74.3, 35.2, 25.3; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1052.



Prop-2-yn-1-yl (benzoyloxy)carbamate (1w): 3.59 g, 82% yield, white soild, m.p. = 63-65 ºC, IR (neat): 3294, 2920, 1744, 1453, 1233 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.55 (br, 1H), 8.12-8.05 (m, 2H), 7.68-7.61 (m, 1H), 7.53-7.44 (m, 2H), 4.82 (d, *J* = 2.4 Hz, 2H), 2.55 (t, *J* = 2.4 Hz, 1H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.5, 155.5, 134.3, 129.9, 128.7, 126.3, 75.9, 54.0; HRMS (ESI) calculated for $[C_{11}H_9NNaO_4]^+$ ($M+Na^+$) requires m/z 242.0424, found: m/z 242.0423.

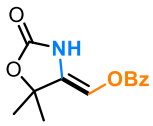
IV. Visible-Light-Promoted Intramolecular Oxyamidation of Alkynes

Materials used for set-up: 10 mL Schlenk flask. IKA RCT digital. Blue LED lamp (450 nm, 5

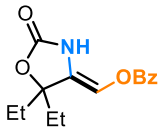
W/m, 1 m). Water bath. The LED lamp was set aside the Schlenk flask.

General procedure for visible-light-promoted alkyne carboamination: a 10 mL flame-dried Schlenk flask was cooled at room temperature under argon, charged with **1** (0.2 mmol), NR (0.004 mmol), DIPEA (0.02 mmol) and MeCN (2 mL). The mixture was degassed for 3 times, and then warmed to room temperature under argon, stirred at 40 °C under 5 W blue LEDs in the parallel batch photoreactor for 12 hours (**Fig. S3**). 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 product **2**.

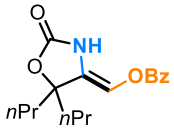
Fig. S3. Photograph of the parallel batch photoreactor.



(*Z*)-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)methyl benzoate (2a): repared according to the general procedure, using 0.0496 g of benzoyloxycarbamate **1a** (0.20 mmol), 0.0014g of NR (0.002 mmol), 0.0026 g of DIPEA (0.02 mmol, 3.4 uL) and 2 mL of MeCN. After reaction at 40 °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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0366 g, 74% yield, white soild; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.99 (brs, 1H), 8.21 (d, J = 8.0 Hz, 2H), 7.62 (t, J = 7.6 Hz, 1H), 7.50 (t, J = 7.6 Hz, 2H), 6.95 (s, 1H), 1.64 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.8, 156.3, 133.7, 130.6, 130.2, 128.7, 128.4, 111.2, 82.9, 28.3; The characterization data of the product is consistent with the reported literature.[1]



(*Z*)-(5,5-diethyl-2-oxooxazolidin-4-ylidene)methyl benzoate (2b): repared according to the general procedure, using 0.0554 g of benzoyloxycarbamate **1b** (0.20 mmol), 0.0012 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0428 g, 78% yield, colorless oil; IR (neat): 2977, 1763, 1717, 1373, 1251 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.27 (brs, 1H), 8.24 (d, *J* = 7.6 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 6.90 (s, 1H), 2.01-1.89 (m, 2H), 1.78-1.67 (m, 2H), 1.01 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.9, 157.1, 133.6, 130.2, 128.6, 128.5, 127.3, 111.5, 88.7, 32.6, 7.2; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1051.



(*Z*)-(2-oxo-5,5-dipropyloxazolidin-4-ylidene)methyl benzoate (2c): repared according to the general procedure, using 0.0611 g of benzoyloxycarbamate **1c** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0468 g, 77% yield, white soild, m.p. = 174-177 °C; IR (neat): 2961, 1762, 1718, 1454, 1251 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.70 (brs, 1H), 8.20 (d, *J* = 8.0, 2H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 6.89 (s, 1H), 1.94-1.82 (m, 2H), 1.71-1.59 (m, 2H), 1.56-1.38 (m, 4H), 0.95 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.9, 156.7, 133.7, 130.1, 128.6, 128.5, 128.0, 111.5, 88.1, 42.2, 16.1, 13.9; HRMS (ESI) calculated for $[C_{17}H_{21}NNaO_4]^+$ ($M+Na^+$) requires m/z 326.1363, found: m/z 326.1363.

(CCDC: 2329040)

Bond precision:	C-C = 0.0019 Å	Wavelength=0.71073	
Cell:	a/ Å =12.2627(5)	b/ Å =8.4254(4)	c/ Å =16.0986(6)
	alpha/°=90	beta/°=101.590(1)	gamma/°=90

Temperature: 170 K

	Calculated	Reported
Volume	1629.36(12)	1629.36(12)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	$C_{17}H_{21}NO_4$	$C_{17}H_{21}NO_4$
Sum formula	$C_{17}H_{21}NO_4$	$C_{17}H_{21}NO_4$
Mr	303.35	303.35
Dx,g cm^{-3}	1.237	1.237
Z	4	4
Mu (mm^{-1})	0.088	0.088
F000	648.0	648.0
F000’	648.33	
h,k,lmax	16,11,21	16,11,21
Nref	4073	4056
Tmin,Tmax	0.967,0.983	0.680,0.746
Tmin’	0.958	

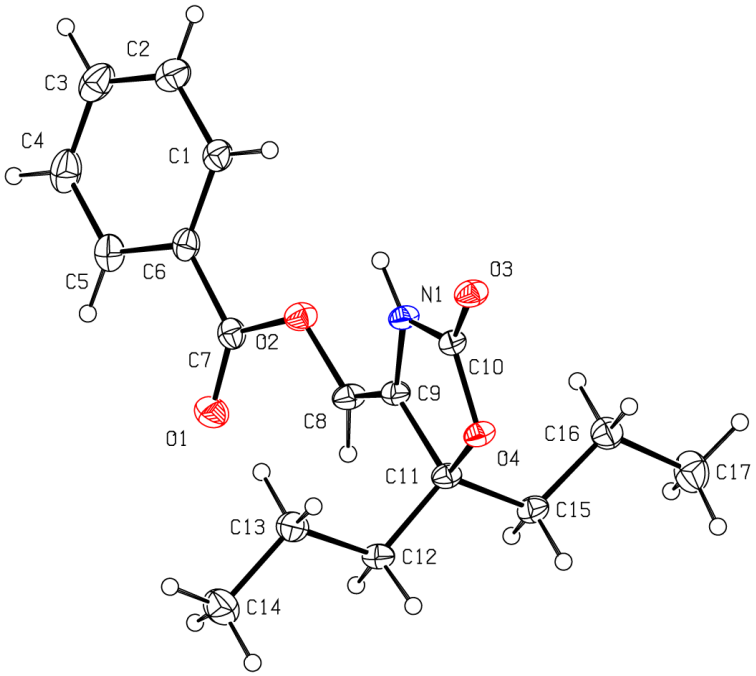
Correction method= # Reported T Limits: Tmin=0.680 Tmax=0.746

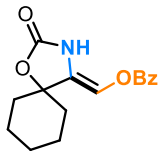
AbsCorr = MULTI-SCAN

Data completeness= 0.996 Theta(max)= 28.342

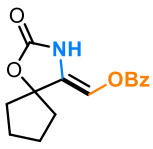
R(reflections)= 0.0456(3342) wR2(reflections)= 0.1046(4056)

S = 1.092 Npar= 201



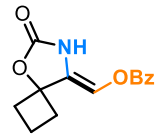


(*Z*)-(2-oxo-1-oxa-3-azaspiro[4.5]decan-4-ylidene)methyl benzoate (2d): repared according to the general procedure, using 0.0576 g of benzoyloxycarbamate **1d** (0.20 mmol), 0.0014 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 ºC for 12 hours, the reaction 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, 1% Et_3N), the reaction afforded 0.0389 g, 68% yield, white soild, m.p. = 260-262 ºC; IR (neat): 2935, 1754, 1710, 1274, 1142 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.04 (brs, 1H), 8.42 (d, *J* = 8.0 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.51 (t, *J* = 7.2 Hz, 2H), 6.95 (s, 1H), 2.28 (d, *J* = 13.6 Hz, 2H), 1.82-1.66 (m, 5H), 1.64-1.54 (m, 2H), 1.40-1.30 (m, 1H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.9, 156.4, 133.7, 130.7, 130.1, 128.6, 128.5, 111.5, 84.6, 37.3, 24.5, 21.6; HRMS (ESI) calculated for $[C_{16}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 310.1050, found: m/z 310.1051.

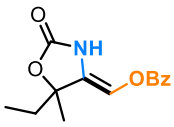


(*Z*)-(2-oxo-1-oxa-3-azaspiro[4.4]nonan-4-ylidene)methyl benzoate (2e): repared according to the general procedure, using 0.0549 g of benzoyloxycarbamate **1e** (0.20 mmol), 0.0014 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 ºC for 12 hours, the reaction 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, 1% Et_3N), the reaction afforded 0.0396 g, 72% yield, light yellow soild, m.p. = 184-186 °C; IR (neat): 3271, 2952, 1719, 1367, 1259, 1126 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.92 (brs, 1H), 8.20 (d, *J* = 7.2 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 2H), 6.97 (s, 1H), 2.38-2.24 (m, 2H), 2.00-1.78 (m, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.8, 156.6, 133.7, 130.2, 129.5, 128.6, 128.5, 111.2, 92.6, 41.3, 24.1; HRMS (ESI) calculated for $[C_{15}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 296.0893, found: m/z 296.0895.

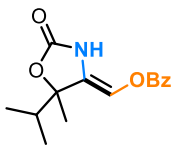


(*Z*)-(6-oxo-5-oxa-7-azaspiro[3.4]octan-8-ylidene)methyl benzoate (2f): repared according to the general procedure, using 0.0518 g of benzoyloxycarbamate **1f** (0.20 mmol), 0.0015 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0243 g, 47% yield, white soild, m.p. = 182-184 °C; IR (neat): 3271, 1763, 1720, 1327, 1256, 1133 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.53 (brs, 1H), 8.19 (d, *J* = 7.6 Hz, 2H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.26 (s, 1H), 2.82-2.70 (m, 2H), 2.52-2.42 (m, 2H), 2.14-2.00 (m, 1H), 1.94-1.78 (m, 1H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.8, 156.3, 133.8, 130.4, 130.2, 128.7, 128.4, 111.6, 84.1, 36.8, 12.8; HRMS (ESI) calculated for $[C_{14}H_{13}NNaO_4]^+$ ($M+Na^+$) requires m/z 282.0737, found: m/z 282.0735.

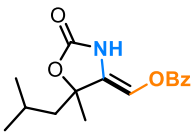


(*Z*)-(5-ethyl-5-methyl-2-oxooxazolidin-4-ylidene)methyl benzoate (2g): repared according to the general procedure, using 0.0525 g of benzoyloxycarbamate **1g** (0.20 mmol), 0.0014 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0395 g, 76% yield, white soild, m.p. = 176-178 °C; IR (neat): 3219, 2983, 1764, 1719, 1256, 1131 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.27 (brs, 1H), 8.23 (d, *J* = 7.6 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 6.92 (s, 1H), 1.98-1.87 (m, 1H), 1.86-1.73 (m, 1H), 1.60 (s, 3H), 1.02 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.9, 156.7, 133.7, 130.2, 129.2, 128.6, 128.5, 111.4, 85.7, 34.0, 26.8, 7.5; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 284.0893, found:

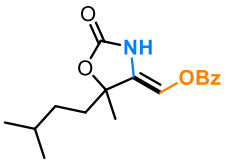
m/z 284.0892.



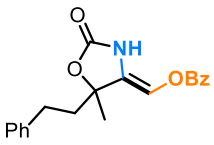
(*Z*)-(5-isopropyl-5-methyl-2-oxooxazolidin-4-ylidene)methyl benzoate (2h): repared according to the general procedure, using 0.0555 g of benzoyloxycarbamate **1h** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 ºC for 12 hours, the reaction 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, 1% Et_3N), the reaction afforded 0.0394 g, 72% yield, white soild, m.p. = 209-212 ºC; IR (neat): 2924, 1761, 1717, 1375, 1258, 1128 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.15 (brs, 1H), 8.23 (d, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 6.92 (s, 1H), 2.01-1.88 (m, 1H), 1.59 (s, 3H), 1.09-1.02 (m, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.9, 156.7, 133.7, 130.2, 129.0, 128.6, 128.4, 111.6, 88.0, 37.5, 25.0, 16.4, 16.2; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1048.



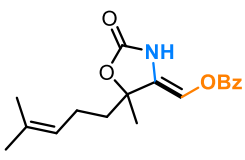
(*Z*)-(5-isobutyl-5-methyl-2-oxooxazolidin-4-ylidene)methyl benzoate (2i): repared according to the general procedure, using 0.0580 g of benzoyloxycarbamate **1i** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 ºC for 12 hours, the reaction 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, 1% Et_3N), the reaction afforded 0.0415 g, 72% yield, light yellow soild, m.p. = 159-162 ºC; IR (neat): 2958, 1762, 1718, 1256, 1128 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.33 (brs, 1H), 8.24 (d, *J* = 8.0 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 6.93 (s, 1H), 1.97-1.78 (m, 2H), 1.74-1.65 (m, 1H), 1.60 (s, 3H), 0.99 (d, *J* = 5.2 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.9, 156.7, 133.7, 130.2, 130.1, 128.6, 128.5, 111.5, 85.6, 49.3, 27.8, 24.3, 24.1, 24.0; HRMS (ESI) calculated for $[C_{16}H_{19}NNaO_4]^+$ ($M+Na^+$) requires m/z 312.1206, found: m/z 312.1206.



(*Z*)-(5-isopentyl-5-methyl-2-oxooxazolidin-4-ylidene)methyl benzoate (2j): repared according to the general procedure, using 0.0620 g of benzoyloxycarbamate **1j** (0.20 mmol), 0.0014 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0463 g, 76% yield, colorless oil; IR (neat): 3214, 2955, 1759, 1718, 1257, 1127 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.28 (brs, 1H), 8.24 (d, *J* = 7.2 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 6.94 (s, 1H), 1.94-1.84 (m, 1H), 1.77-1.67 (m, 1H), 1.61 (s, 3H), 1.59-1.49 (m, 1H), 1.40-1.24 (m, 2H), 0.90 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.8, 156.8, 133.7, 130.2, 129.4, 128.6, 128.4, 111.4, 85.5, 39.0, 31.7, 27.9, 27.2, 22.5, 22.4; HRMS (ESI) calculated for $[C_{17}H_{21}NNaO_4]^+$ ($M+Na^+$) requires m/z 326.1363, found: m/z 326.1363.

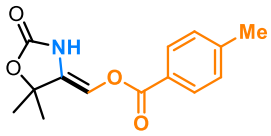


(*Z*)-(5-methyl-2-oxo-5-phenethyloxazolidin-4-ylidene)methyl benzoate (2k): repared according to the general procedure, using 0.0681 g of benzoyloxycarbamate **1k** (0.20 mmol), 0.0014 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0472 g, 70% yield, colorless oil; IR (neat): 2923, 1761, 1719, 1454, 1256 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.42 (brs, 1H), 8.18 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.33-7.26 (m, 2H), 7.23-7.14 (m, 3H), 6.97 (s, 1H), 2.86-2.68 (m, 2H), 2.28-2.16 (m, 1H), 2.10-1.97 (m, 1H), 1.66 (s, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.8, 156.5, 140.5, 133.8, 130.2, 129.1, 128.7, 128.5, 128.4, 128.3, 126.2, 111.6, 85.0, 43.0, 29.5, 27.3; HRMS (ESI) calculated for $[C_{20}H_{19}NNaO_4]^+$ ($M+Na^+$) requires m/z 360.1206, found: m/z 360.1206.

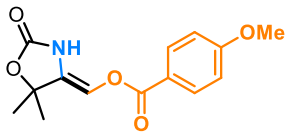


(*Z*)-(5-methyl-5-(4-methylpent-3-en-1-yl)-2-oxooxazolidin-4-ylidene)methyl benzoate (2l): repared according to the general procedure, using 0.0662 g of benzoyloxycarbamate **1l** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12

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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0249 g, 38% yield, colorless oil; IR (neat): 2980, 1725, 1454, 1378, 1269 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.99 (brs, 1H), 8.21 (d, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 6.93 (s, 1H), 5.09 (t, *J* = 6.8 Hz, 1H), 2.22-2.02 (m, 2H), 1.98-1.86 (m, 1H), 1.80-1.68 (m, 1H), 1.67 (s, 3H), 1.64-1.57 (m, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.8, 156.4, 133.7, 132.8, 130.1, 129.3, 128.6, 128.4, 122.4, 111.4, 85.2, 40.9, 27.3, 25.6, 21.9, 17.7; HRMS (ESI) calculated for $[C_{18}H_{21}NNaO_4]^+$ ($M+Na^+$) requires m/z 338.1363, found: m/z 338.1364.

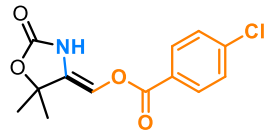


***(Z)*-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)methyl 4-methylbenzoate (2m):** repared according to the general procedure, using 0.0522 g of benzoyloxycarbamate **1m** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0320 g, 61% yield, light yellow soild, m.p. = 277-280 °C; IR (neat): 2991, 1760, 1714, 1417, 1267 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.99 (brs, 1H), 8.09 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.94 (s, 1H), 2.43 (s, 3H), 1.63 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.9, 156.4, 144.6, 130.3, 130.2, 129.4, 125.6, 111.2, 82.9, 28.3, 21.7; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 284.0893, found: m/z 284.0894.

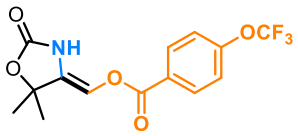


***(Z)*-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)methyl 4-methoxybenzoate (2n):** repared according to the general procedure, using 0.0536 g of benzoyloxycarbamate **1n** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0277 g, 50% yield, light yellow soild, m.p. = 174-177 °C; IR (neat): 2987, 1755, 1606, 1513, 1257 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.97 (brs, 1H), 8.16 (d, *J* = 8.8 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 6.93 (s, 1H), 3.89 (s, 3H), 1.63 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 164.0, 162.6,

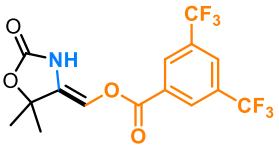
156.5, 132.3, 130.1, 120.7, 113.9, 111.3, 83.0, 55.5, 28.3; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 300.0842, found: m/z 300.0842.



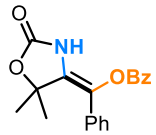
(*Z*)-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)methyl 4-chlorobenzoate (2o): repared according to the general procedure, using 0.0564 g of benzoyloxycarbamate **1o** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0184 g, 33% yield, white soild, m.p. = 225-228 °C; IR (neat): 3529, 2921, 1716, 1263, 1178, 1124 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.33 (brs, 1H), 8.18 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 6.95 (s, 1H), 1.64 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 162.0, 156.5, 140.3, 131.6, 130.8, 129.0, 126.8, 111.1, 83.1, 28.3; HRMS (ESI) calculated for $[C_{13}H_{12}ClNNaO_4]^+$ ($M+Na^+$) requires m/z 304.0347, found: m/z 304.0345.



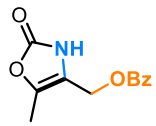
***(Z)*-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)methyl 4-(trifluoromethoxy)benzoate (2p):** repared according to the general procedure, using 0.0665 g of benzoyloxycarbamate **1p** (0.20 mmol), 0.0014 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0322 g, 49% yield, white soild, m.p. = 218-220 °C; IR (neat): 2926, 1755, 1712, 1271, 1174 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 9.46 (brs, 1H), 8.31 (d, *J* = 7.6 Hz, 2H), 7.34 (d, *J* = 7.6 Hz, 2H), 6.96 (s, 1H), 1.65 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 161.7, 156.6, 153.2, 132.3, 130.9, 126.8, 120.4, 120.3, (q, *J* = 258.1 Hz), 111.2, 83.1, 28.3; ^{19}F NMR: (376 MHz, $CDCl_3$) δ -57.5; HRMS (ESI) calculated for $[C_{14}H_{12}F_3NNaO_5]^+$ ($M+Na^+$) requires m/z 354.0560, found: m/z 354.0561.



***(Z)*-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)methyl 3,5-bis(trifluoromethyl)benzoate (2q):** repared according to the general procedure, using 0.0769 g of benzoyloxycarbamate **1q** (0.20 mmol), 0.0012 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0465 g, 61% yield, white soild, m.p. = 256-258 °C; IR (neat): 2957, 1723, 1282, 1244, 1139 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 10.30 (brs, 1H), 8.75 (s, 2H), 8.12 (s, 1H), 6.99 (s, 1H), 1.66 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 160.5, 157.2, 132.4 (q, J = 33.4 Hz),132.3, 130.8, 130.5, 127.1-126.9 (m), 122.8 (q, J = 271.6 Hz), 111.0, 83.1, 28.1; ^{19}F NMR: (376 MHz, $CDCl_3$) δ -62.8; HRMS (ESI) calculated for $[C_{15}H_{11}F_6NNaO_4]^+$ ($M+Na^+$) requires m/z 406.0484, found: m/z 406.0483.

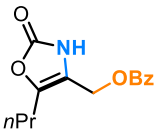


(*Z*)-(5,5-dimethyl-2-oxooxazolidin-4-ylidene)(phenyl)methyl benzoate (2r): repared according to the general procedure, using 0.0649 g of benzoyloxycarbamate **1r** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0431 g, 67% yield, white soild, m.p. = 210-213 °C; IR (neat): 3352, 2981, 1747, 1390, 1277 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.11 (d, J = 8.0 Hz, 2H), 7.65 (t, J = 6.8 Hz, 2H), 7.51 (t, J = 7.6 Hz, 2H), 7.47-7.35 (m, 4H), 7.32-7.25 (m, 1H), 1.69 (s, 6H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 165.2, 155.5, 134.0, 133.6, 132.6, 130.0, 129.1, 128.8, 128.7, 128.5, 126.1, 124.0, 84.9, 25.7; HRMS (ESI) calculated for $[C_{19}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 346.1050, found: m/z 346.1051.

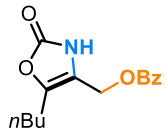


(5-Methyl-2-oxo-2,3-dihydrooxazol-4-yl)methyl benzoate (2s): repared according to the general procedure, using 0.0466 g of benzoyloxycarbamate **1s** (0.20 mmol), 0.0012 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA

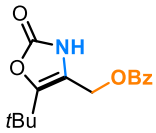
= 5/1-2/1, 1% Et_3N), the reaction afforded 0.0159 g, 34% yield, yellow soild, m.p. = 96-99 °C; IR (neat): 3266, 2960, 1723, 1452, 1271 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.47 (br, 1H), 8.03 (d, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 5.03 (s, 2H), 2.18 (s, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.0, 155.7, 137.4, 133.5, 129.8, 129.1, 128.5, 116.2, 55.3, 10.1; HRMS (ESI) calculated for $[C_{12}H_{11}NNaO_4]^+$ ($M+Na^+$) requires m/z 256.0580, found: m/z 256.0578.



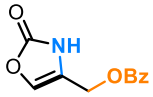
(2-Oxo-5-propyl-2,3-dihydrooxazol-4-yl)methyl benzoate (2t): repared according to the general procedure, using 0.0526 g of benzoyloxycarbamate **1t** (0.20 mmol), 0.0012 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^1H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0192 g, 37% yield, colorless oil; IR (neat): 3352, 2972, 1722, 1452, 1382, 1270 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.55 (brs, 1H), 8.03 (d, *J* = 7.2 Hz, 2H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 5.04 (t, 2H), 2.48 (t, *J* = 7.2 Hz, 2H), 1.70-1.58 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.1, 155.6, 141.1, 133.6, 129.8, 129.0, 128.5, 116.3, 55.2, 26.3, 20.7, 13.4; HRMS (ESI) calculated for $[C_{14}H_{15}NNaO_4]^+$ ($M+Na^+$) requires m/z 284.0893, found: m/z 284.0894.



(5-Butyl-2-oxo-2,3-dihydrooxazol-4-yl)methyl benzoate (2u): repared according to the general procedure, using 0.0549 g of benzoyloxycarbamate **1u** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C 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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0170 g, 31% yield, colorless oil; IR (neat): 3355, 2974, 1722, 1382, 1272 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$) δ 8.73 (brs, 1H), 8.04 (d, *J* = 8.0 Hz, 2H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 5.04 (s, 2H), 2.50 (t, *J* = 7.2 Hz, 2H), 1.68-1.54 (m, 2H), 1.44-1.22 (m, 2H), 0.92 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 167.0, 155.8, 141.2, 133.6, 129.8, 129.1, 128.5, 116.0, 55.2, 29.4, 24.1, 21.9, 13.6; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1050.



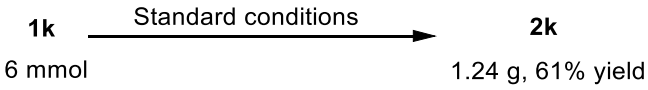
(5-(*Tert*-butyl)-2-oxo-2,3-dihydrooxazol-4-yl)methyl benzoate (2v): repared according to the general procedure, using 0.0551 g of benzoyloxycarbamate **1v** (0.20 mmol), 0.0013 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °C for 12 hours, the reaction was concentrated by rotary evaporation and monitored by ^{1}H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0276 g, 50% yield, colorless oil; IR (neat): 3283, 2977, 1722, 1453, 1270, 1113 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.68 (brs, 1H), 8.05 (d, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 7.2 Hz, 2H), 5.18 (s, 2H), 1.34 (s, 9H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 166.7, 155.5, 147.3, 133.5, 129.8, 129.2, 128.5, 114.3, 56.1, 32.9, 28.8; HRMS (ESI) calculated for $[C_{15}H_{17}NNaO_4]^+$ ($M+Na^+$) requires m/z 298.1050, found: m/z 298.1051.



(2-Oxo-2,3-dihydrooxazol-4-yl)methyl benzoate (2w): repared according to the general procedure, using 0.0443 g of benzoyloxycarbamate **1w** (0.20 mmol), 0.0014 g of NR (0.004 mmol), 0.0026 g of DIPEA (0.02 mmol) and 2 mL of MeCN. After reaction at 40 °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 = 5/1-2/1, 1% Et_3N), the reaction afforded 0.0101 g, 23% yield, IR (neat): 2923, 2853, 1727, 1457, 1375, 1273 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 8.37 (brs, 1H), 8.04 (d, *J* = 7.2 Hz, 2H), 7.60 (t, *J* = 6.8 Hz, 1H), 7.46 (t, *J* = 6.8 Hz, 2H), 6.89 (s, 1H), 5.05 (s, 2H). HRMS (ESI) calculated for $[C_{11}H_{10}NO_4]^+$ ($M+H^+$) requires m/z 220.0604, found: m/z 220.0605. The characterization data of the product is consistent with the reported literature.[2]

V. Synthetic Applications and Mechanistic Studies

a) Gram-scale reaction and synthetic applications:



Materials used for set-up: 100 mL Schlenk flask. IKA RCT digital. Blue LED lamp strip (454 nm, 5 W/m, 1 m). Water bath (95 mm). The LED lamp strap was wrapped around the outside of the

oil bath.

A 100 mL flame-dried Schlenk flask was cooled at room temperature under argon, then charged with **1k** (2.0244 g, 6.0 mmol), NR (0.0346 g, 0.12 mmol), DIPEA (0.0776 g, 0.6 mmol) and MeCN (60 mL). The mixture was degassed for three times, warmed to room temperature under argon, and stirred at 40 °C while being irradiated with 5 W blue LEDs for 12 hours. The reaction mixture 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, 1% Et_3N) to afford 1.24 g **2k** in 61% yield.

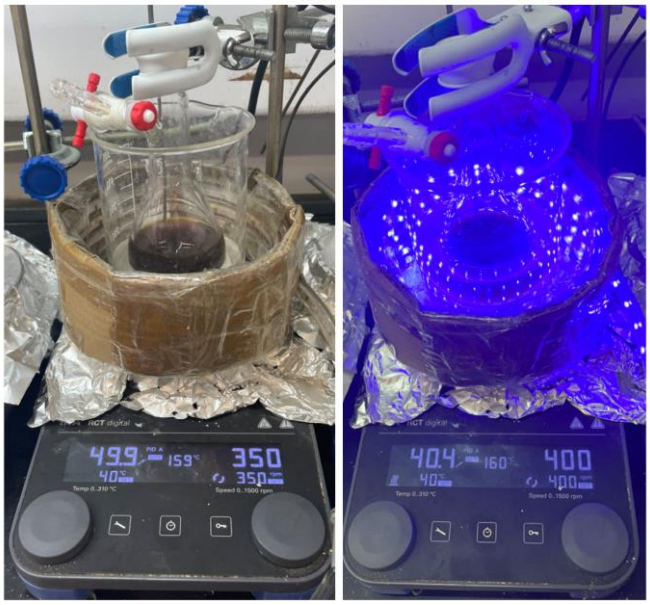


Fig. S4. Photograph of the scale-up reaction.

The E-factor takes into account waste byproducts, leftover reactants, solvent losses, spent catalysts and catalyst supports, and anything else that can be regarded as a waste.[3] Since the solvent MeCN can be recovered, it is not regarded as the waste. The $m_{product}$ is the mass of the product purified by flash chromatography. The $m_{byproduct}$ is calculated as the difference between the mass of the product in 100% theoretical yield and the mass of the purified product. The m_{NR} and m_{DIPEA} are the mass of the NR and DIPEA used in the reaction, respectively. Herein, the E-factor of the **Gram-scale reaction** is calculated by: [3]

$$\mathrm{E} = \frac{\text{Total mass of waste from process}}{\text{Total mass of product}} = \frac{m_{byproduct} + m_{NR} + m_{DIPEA}}{m_{product}}$$

$$= \frac{0.7884 + 0.0346 + 0.0776\ \mathrm{g}}{1.2368\ \mathrm{g}} = 0.728$$

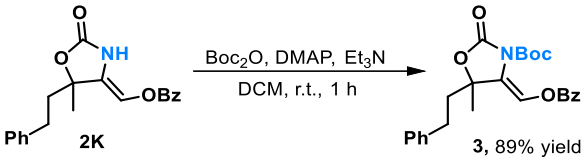
The atom utilization (AU) measures the conversion of atoms in the feedstock to the target product, regardless of factors such as yield (all assumed to be 100%) and selectivity, as well as the various types of catalysts and additives used in the synthesis process. Herein, the $A_{substrate}$ and the $A_{product}$ are the relative molecular masses of the substrate **1k** and product **2k**, respectively. The AU of the **Gram-scale reaction** is calculated by: [3]

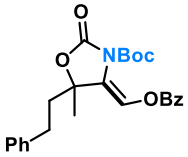
$$AU\ (\%)=\frac{A_{product}}{A_{substrate}}\times 100\ \%=\frac{337.4}{337.4}\times 100\ \%=100\ \%$$

The reaction mass efficiency (RME) is also a measure of feedstock utilization, and refers to how much of the feedstock mass is converted into the product. Herein, the mass of the product is the mass of product **2k** purified by flash chromatography, and the total mass of reactants is the mass of substrate **1k**. The RME of the **Gram-scale reaction** is calculated by: [3]

$$RME\ (\%)=\frac{\text{Mass of product}}{\text{Total mass of reactants}}\times 100\ \%=\frac{1.2368\ g}{2.0252\ g}\times 100\ \%=61\ \%$$

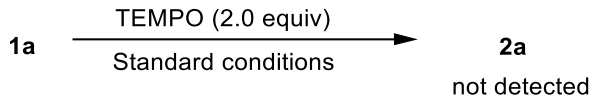
Synthetic applications





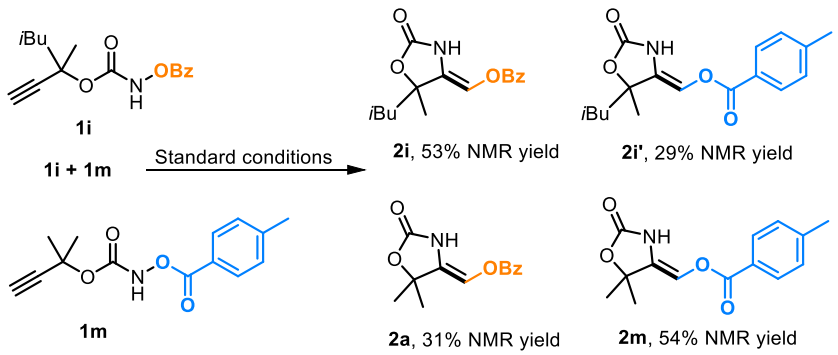
***Tert*-butyl (*Z*)-4-((benzoyloxy)methylene)-5-methyl-2-oxo-5-phenethyloxazolidine-3-carboxylate (3):** repared according to the general procedure,[4] a mixture of **2k** (0.169 g, 0.5 mmol), Boc_2O (0.160 g, 0.7 mmol), Et_3N (0.076 g, 105 μL, 0.75 mmol) and a catalytic amount of DMAP (0.0072 g, 0.05 mmol) in CH_2Cl_2 (10 mL) was stirred at room temperature for 1 hour, the reaction was concentrated by rotary evaporation and monitored by ^1H NMR, and then further purified by flash chromatography on silica gel (PE/EA = 5/1), the reaction afforded 0.196 g, 89% yield, colorless oil; ^1H NMR: (600 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.8 Hz, 2H), 7.61 (t, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.32-7.26 (m, 2H), 7.21-7.16 (m, 3H), 7.08 (s, 1H), 2.80-2.70 (m, 2H), 2.25-2.15 (m, 1H), 2.11-2.02 (m, 1H), 1.66 (s, 3H), 1.57 (s, 9H); ^{13}C NMR: (150 MHz, $CDCl_3$) δ 162.7, 150.7, 147.1, 140.4, 133.9, 130.2, 128.6, 128.5, 128.3, 128.3, 126.2, 124.4, 117.2, 85.2, 83.2, 42.9, 29.5, 27.8, 26.5; HRMS (ESI) calculated for $[C_{25}H_{27}NNaO_6]^+$ ($M+Na^+$) requires m/z 460.1731, found: m/z 460.1732.

b) Radical-inhibition experiments:



According to the general procedure, a 10 mL flame-dried Schlenk flask was cooled at room temperature under argon, then charged with 0.0742 g (0.3 mmol) of **1a**, 0.0017 g (0.006 mmol) of NR, 0.0039 g (0.03 mmol) of DIPEA and 3 mL of MeCN, and 0.0937 g (0.6 mmol) of TEMPO. The mixture was degassed for 3 times, then warmed to room temperature under argon, stirred at 40 °C under blue LED for 12 hours. The mixture was concentrated by rotary evaporation, and no product **2a** was monitored by ^{1}H NMR.

c) Cross-over reaction:



According to the general procedure, a 10 mL flame-dried Schlenk flask was cooled at room temperature under argon, then charged with a 1:1 N-acyloxycarbamates mixture of 0.0586 g **1i** (0.2 mmol) and 0.0526 g **1m** (0.2 mmol), 0.0024 g (0.008 mmol) of NR, 0.0052 g (0.04 mmol) of DIPEA and 4 mL of MeCN. The mixture was degassed for 3 times, then warmed to room temperature under argon, stirred at 40 °C under blue LED for 12 hours. The mixture was concentrated by rotary evaporation, and 4 cross-over products were observed, including **2i** (53%), **2i'** (29%), **2a** (31%), **2m** (54%) (NMR yield, using Trimethylphenylsilane as an internal standard).

d) Stern-Volmer quenching experiments

Spectrum Data of NR:

Experimental Details: All fluorescence data was acquired on a Shimadzu RF6000. Stock solutions of $5x10^{-5}$ M NR, and $5x10^{-5}$ M 1:5 mixture of NR and DIPEA were prepared in HPLC-grade MeCN. After preparing these solutions in separate vials, the volume was transferred to a 1 cm cuvette, which was fitted with an appropriate septum, and sparged for 10 minutes prior to data acquisition. Absorption experiment samples were excited in the 300-700 nm wavelength range, then collected at 590 nm. Emission experiment samples were excited at 450 nm, then collected in the 460-700 nm wavelength range.

The commercially available hydrochloride form of NR is a good acid-base indicator, which appears red in acetonitrile solution. When DIPEA is added to the solution, the solution becomes alkaline and the color changes to yellow. The data unveiled that the pinnacle of absorption wavelength for NR stood at 478.4 and 559.9 nm, while the maximum emission wavelength reached 614 nm. In the case of NR+DIPEA (1:5), the maximum absorption wavelength settled at 444.2 nm, with the maximum emission wavelength at 570 nm. The results of fluorescence spectroscopy imply that the maximum absorption and emission wavelength of NR exhibit fluctuations contingent upon the acidity and alkalinity of the solution. Considering the alkaline solution of our reaction, it follows that the maximum absorption wavelength of NR should approximately hover around 444.2 nm, while the maximum emission wavelength should manifest at approximately 570 nm. The fluorescence intensity of NR is significantly enhanced with the addition of DIPEA (**Fig. S5**).

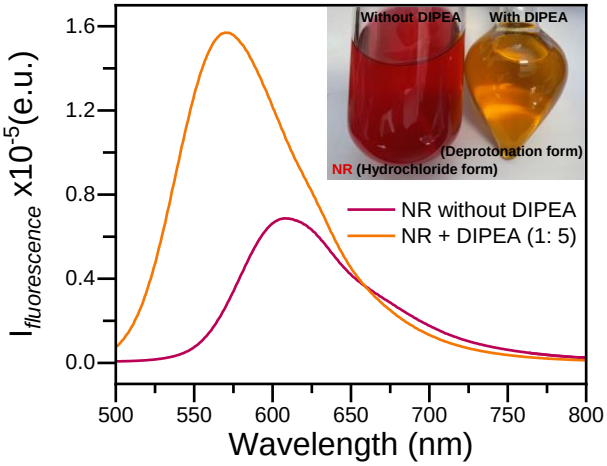


Fig. S5. Emission data of NR, NR+DIPEA (1:5). The color change of NR in acetonitrile at different pH values.

Fluorescence Quenching Data

Table S1. Sample, contents: $5x10^{-5}$ M NR+DIPEA (1:5), indicated concentration **1b**

Entry	Concentration, mM	Intensity	Average	I_0/I
blank1	0	164769		
blank2	0	156982	159579	1
blank3	0	156987		
S1-1	5	153194		
S1-2	5	149432	151851	1.05089
S1-3	5	152928		
S2-1	10	148452		
S2-2	10	150042	146994	1.08562
S2-3	10	142488		
S3-1	15	141843		
S3-2	15	142063	142307	1.12137
S3-3	15	143015		
S4-1	20	139484		
S4-2	20	140941	139229	1.14616
S4-3	20	137263		
S5-1	25	134419		
S5-2	25	136015	134272	1.18848
S5-3	25	132383		

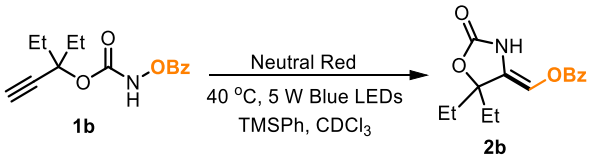
Experimental Details: All fluorescence data was acquired on a Shimadzu RF6000. Stock solutions of a 1:5 mixture of NR+DIPEA, and **1b** were prepared in HPLC-grade MeCN. Stock solutions were combined by volume to give 4.0 mL samples which each contained $5x10^{-5}$ M NR+DIPEA (1:5), as well as variable concentrations (5, 10, 15, 20, 25 mM) of **1b** indicated in the "concentration" column of the table. After preparing these solutions in separate vials, the volume was transferred to a 1 cm cuvette, which was fitted with an appropriate septum, and sparged for 10

minutes prior to data acquisition. Samples were excited at 450 nm, then collected in the 460-700 nm wavelength range. Intensities were recorded at 570 nm (λmax of NR in alkaline solution), repeated three times, and then averaged. The intensity of the emission peak at 570 nm (λex = 444 nm) expressed as the ratio I_0/I, where I_0 is the emission intensity of NR at 570 nm in the absence of a quencher and I is the observed intensity, as a function of the quencher concentration was measured. The Stern-Volmer emission spectra and plot of NR (0.05 mM) at different concentrations of **1b** are shown in **Scheme 3d**, the data are shown in **Table S1**.

e) Light on and off experiments

A 25 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with **1b** (0.2 mmol), NR (0.002 mmol), DIPEA (0.02 mmol) and $CDCl_3$ (4 mL). The mixture was degassed for 3 times, and then warmed to room temperature under nitrogen, stirred at 40 °C under 5 W blue LEDs. The reaction yields were detected by NMR (**Table S2, Fig. S6**).

Table S2. Light on and off experiments.



Entry	Time (min)	Light	2b (%)	Recovery of 1b (%)
1	0	On	0	100
2	10	off	26	61
3	20	On	27	61
4	30	off	42	42
5	40	On	42	42
6	50	off	52	36
7	60	/	52	36

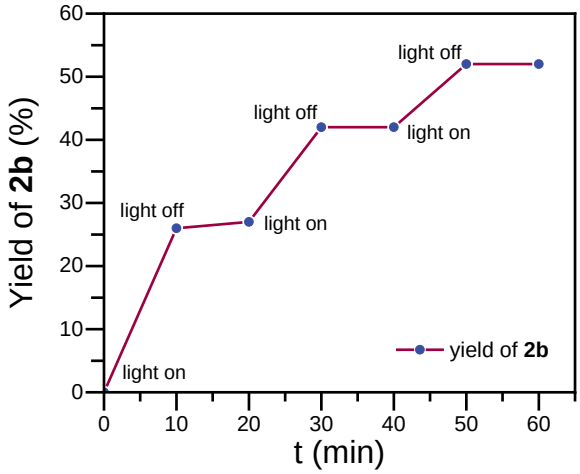


Fig. S6. Light on and off experiments.

f) Quantum yield experiments

A 25 mL flame-dried Schlenk flask was cooled at room temperature under nitrogen, charged with **1b** (0.2 mmol), NR (0.002 mmol), DIPEA (0.02 mmol) and $CDCl_3$ (4 mL). The mixture was degassed for 3 times, and then warmed to room temperature under nitrogen, stirred at 40 °C under 5 W blue LEDs for 1200 s. The reaction yields were detected by NMR.

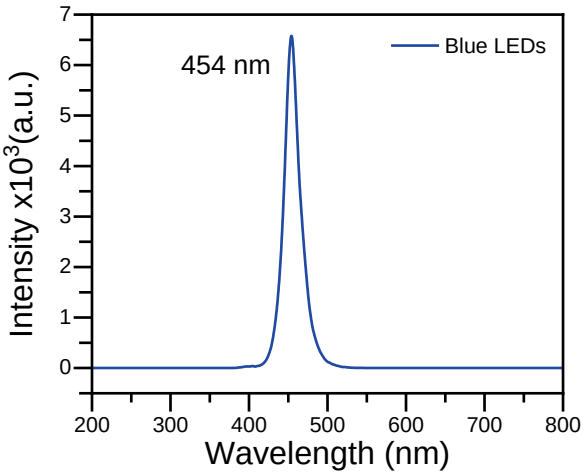


Fig. S7. The emission spectra of the light source (5 W blue LEDs, λmax = 454 nm).

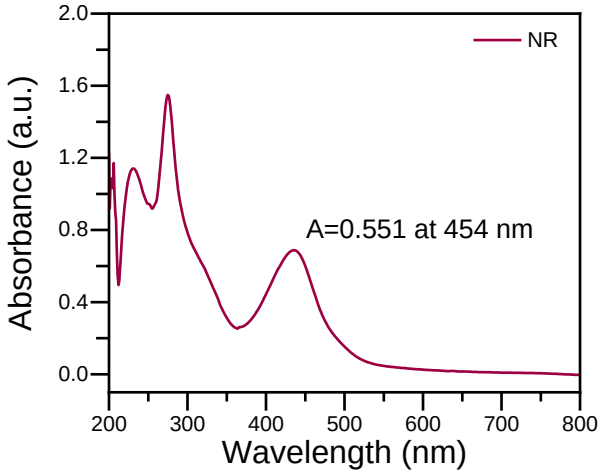


Fig. S8. The absorbance spectra of NR (5×10^{-5} mol/L) in the presence of DIPEA (2.5×10^{-4} mol/L).

The quantum yield is calculated by: [5]

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

The absorbance (A) of NR ($c=5\times10^{-5}$ mol/L) at 454 nm is 0.551 (**Fig. S7, Fig. S8**), and the optical length of the cuvette (l) is 1 cm, so the molar absorption coefficient ε of NR in 454 nm is calculated by:

$$\varepsilon = \frac{A}{c \cdot l} = \frac{0.551}{5\times10^{-5}\times1} = 11020 \ \text{L·mol}^{-1}\text{·cm}^{-1}$$

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

The molar absorption coefficient ε of NR in 454 nm is 11020 $\text{L·mol}^{-1}\text{·cm}^{-1}$. The diameter of the 25 mL Schlenk flask is 2 cm. The NR concentration is 0.0005 mol/L. So the absorbance $A = \varepsilon \cdot c \cdot l = 11020\times2\times0.0005 = 11.02$, and the fraction of light absorbed $f = 1\text{-}10^{-A} > 0.9999$. The moles of photon is calculated by:

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

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

Determination of the light intensity at the 454 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 S3**).

Table S3. The light intensity at the 454 nm LED systems

Entry	Light intensity (mW/cm^2)
1	2.412
2	2.342
3	2.425
4	2.372
5	2.217
6	2.435
Average	**2.367**

Experiment: 55.4 mg (0.2 mmol) **1b**, 1.4 mg (0.002 mmol) NR, 2.6 mg of DIPEA (0.02 mmol, 3.4 μL), 4.0 mL (0.05 M) $CDCl_3$ after 1200 s yielded 42% of **2b**.

Φ(42%) = 0.9743.

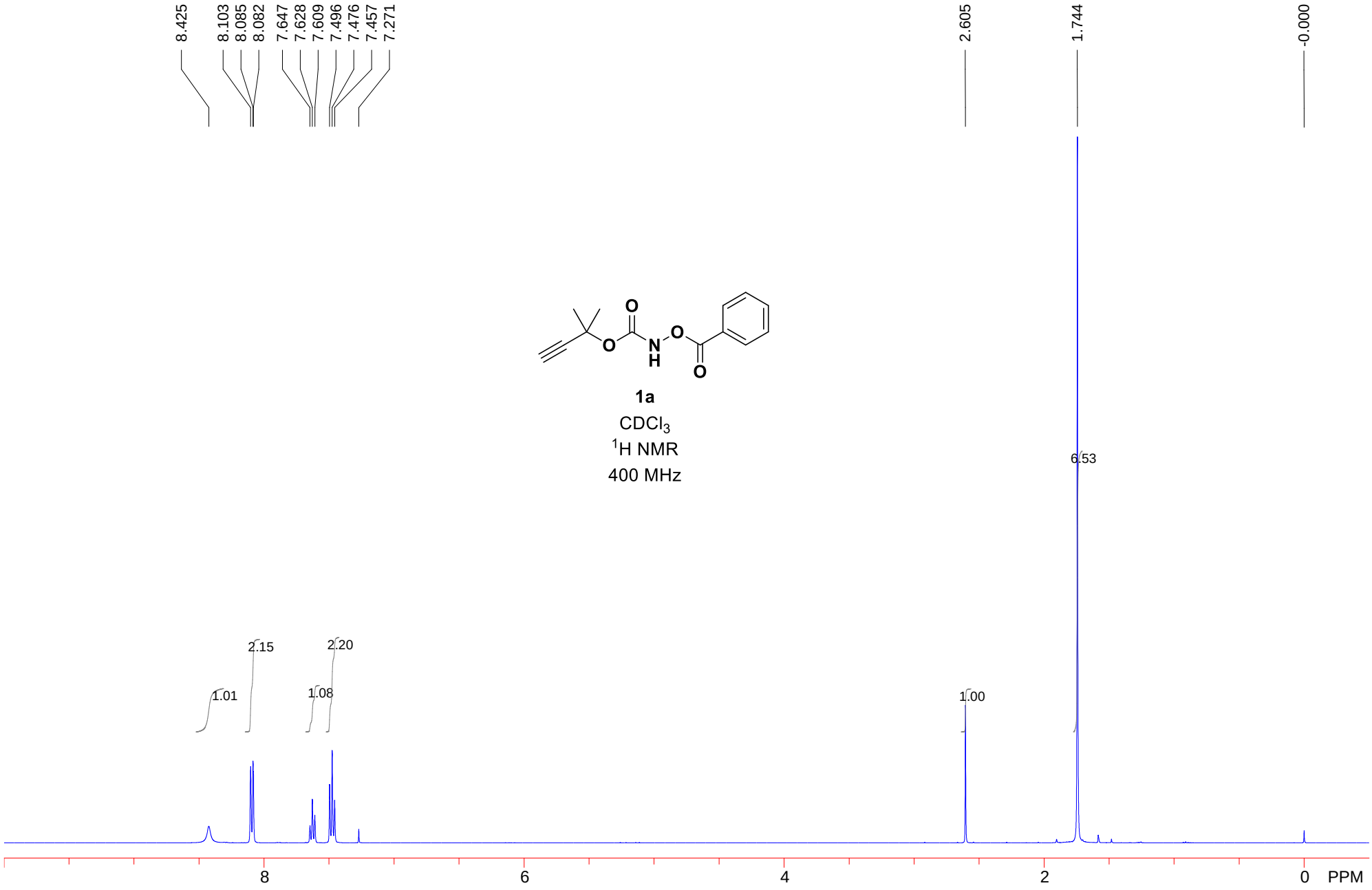
Sample quantum yield calculation:

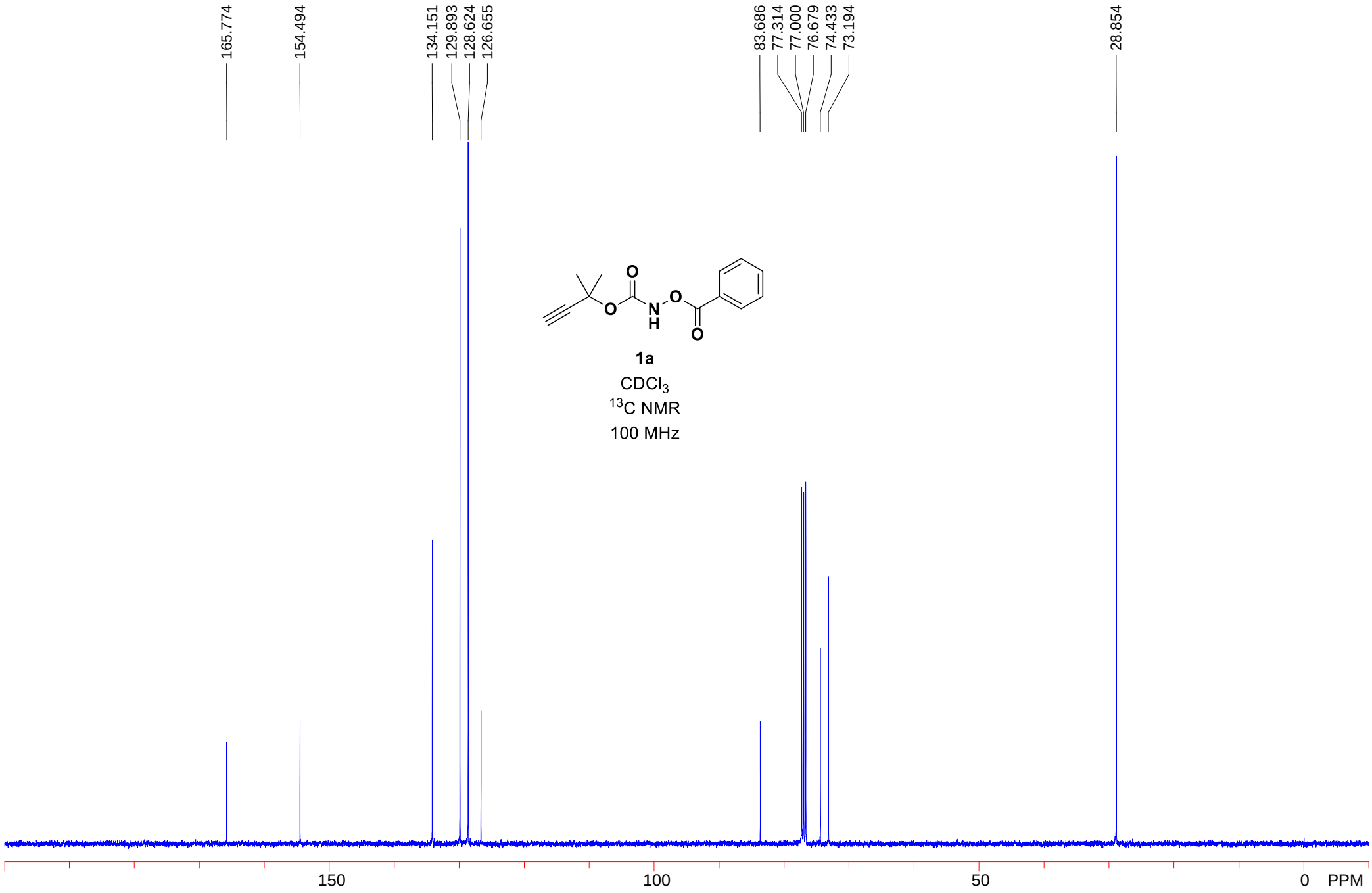
$$\Phi = \frac{8.4 \times 10^{-5}\ \text{mol}}{\frac{2.367 \times 8 \times 10^{-3} \times 1200}{\frac{6.626 \times 10^{-34} \times 3 \times 10^{8}}{\frac{454 \times 10^{-9}}{6.02 \times 10^{23}}} \times 1.00}} = 0.9743$$

VII. References

1. Q. Guo, D. Lu, Y. Mao and Z. Lu, *Chem. Commun.*, 2023, **59**, 1979-1982.
2. S. Su, T. Wu, Y. Xia, D. J. Wink and D. Lee, *Chem. - Eur. J.*, 2023, **29**, e202203371.
3. R. A. Sheldon, *ACS Sustainable Chem. Eng.*, 2017, **6**, 32-48.
4. Y. Tan, F. Han, M. Hemming, J. Wang, K. Harms, X. Xie, and E. Meggers, *Org. Lett.,* 2020, **22**, 16, 6653-6656.
5. M. A. Cismesia, T. P. Yoon, *Chem. Sci.*, 2015, **6**, 5426-5434.

IX. NMR Spectra

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H
O
O
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CDCl3
13C NMR
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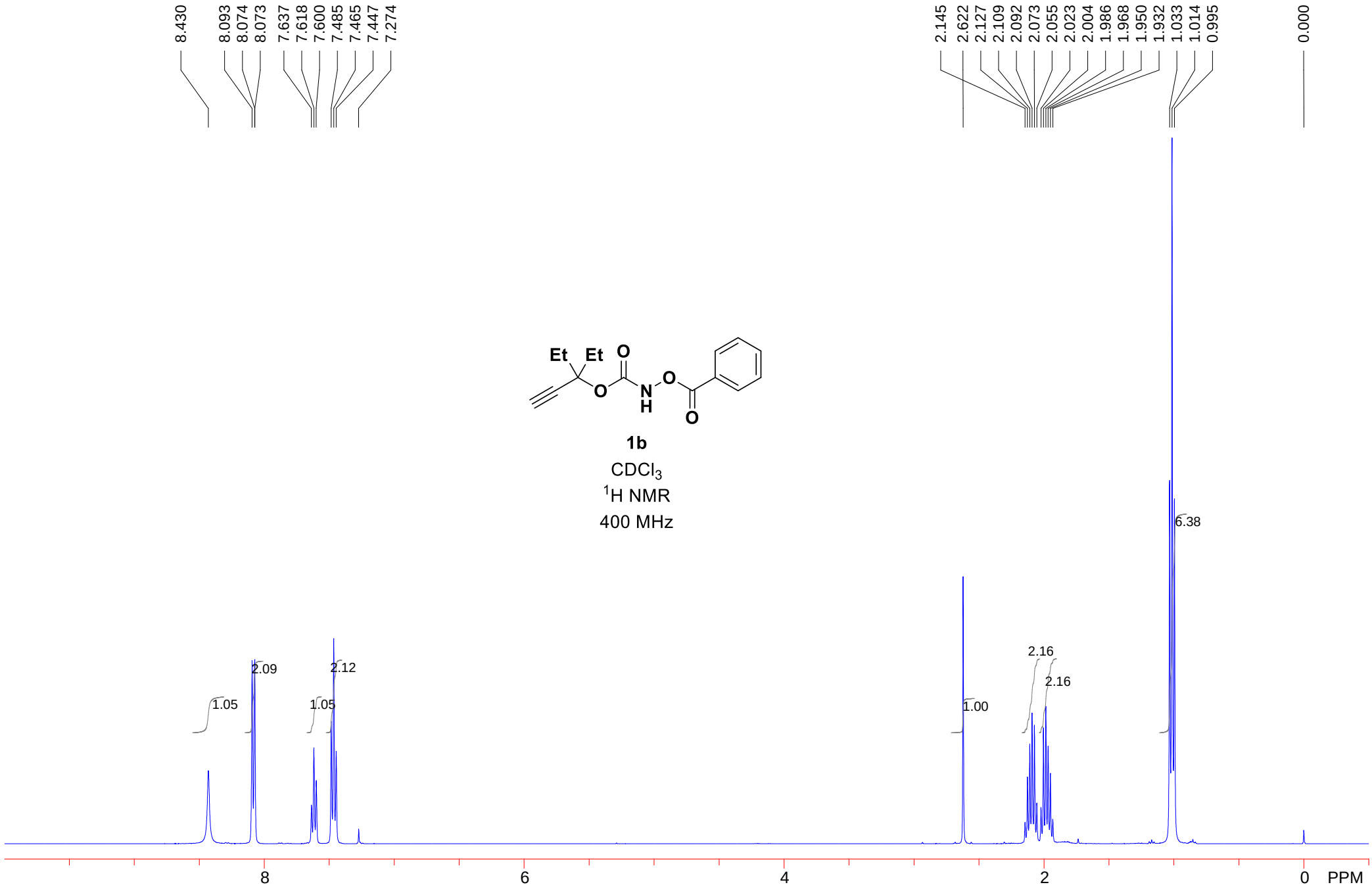
150
100
50
0
PPM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2.622
2.127
2.109
2.092
2.073
2.055
2.023
2.004
1.986
1.968
1.950
1.932
1.033
1.014
0.995
0.000
Et
Et
O
O
N
H
O
O
1b
CDCl3
1H NMR
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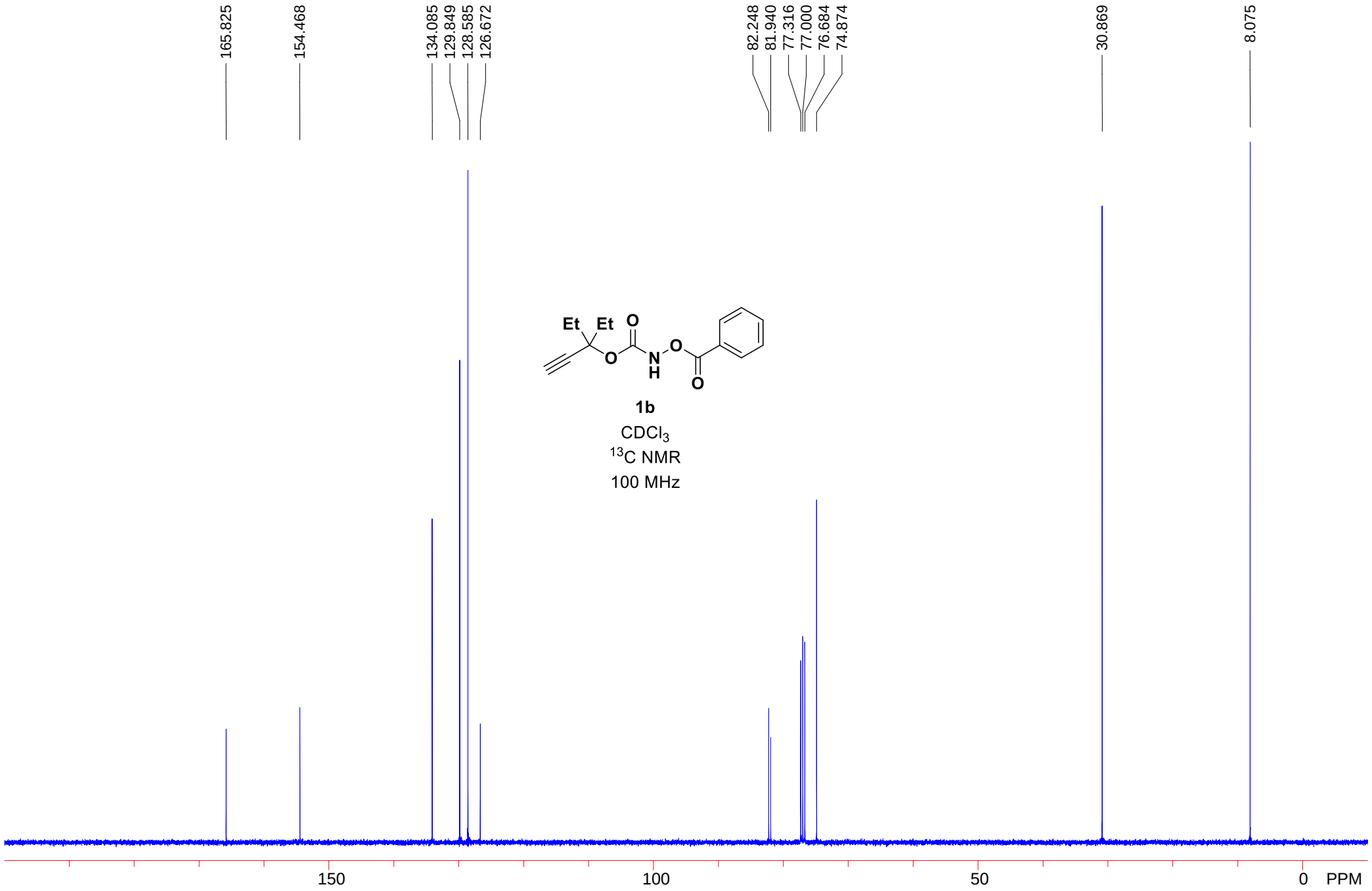
1.05
2.12
1.00
2.16
2.16
6.38
8
6
4
2
0
PPM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30.869
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O
N
H
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CDCl3
13C NMR
100 MHz
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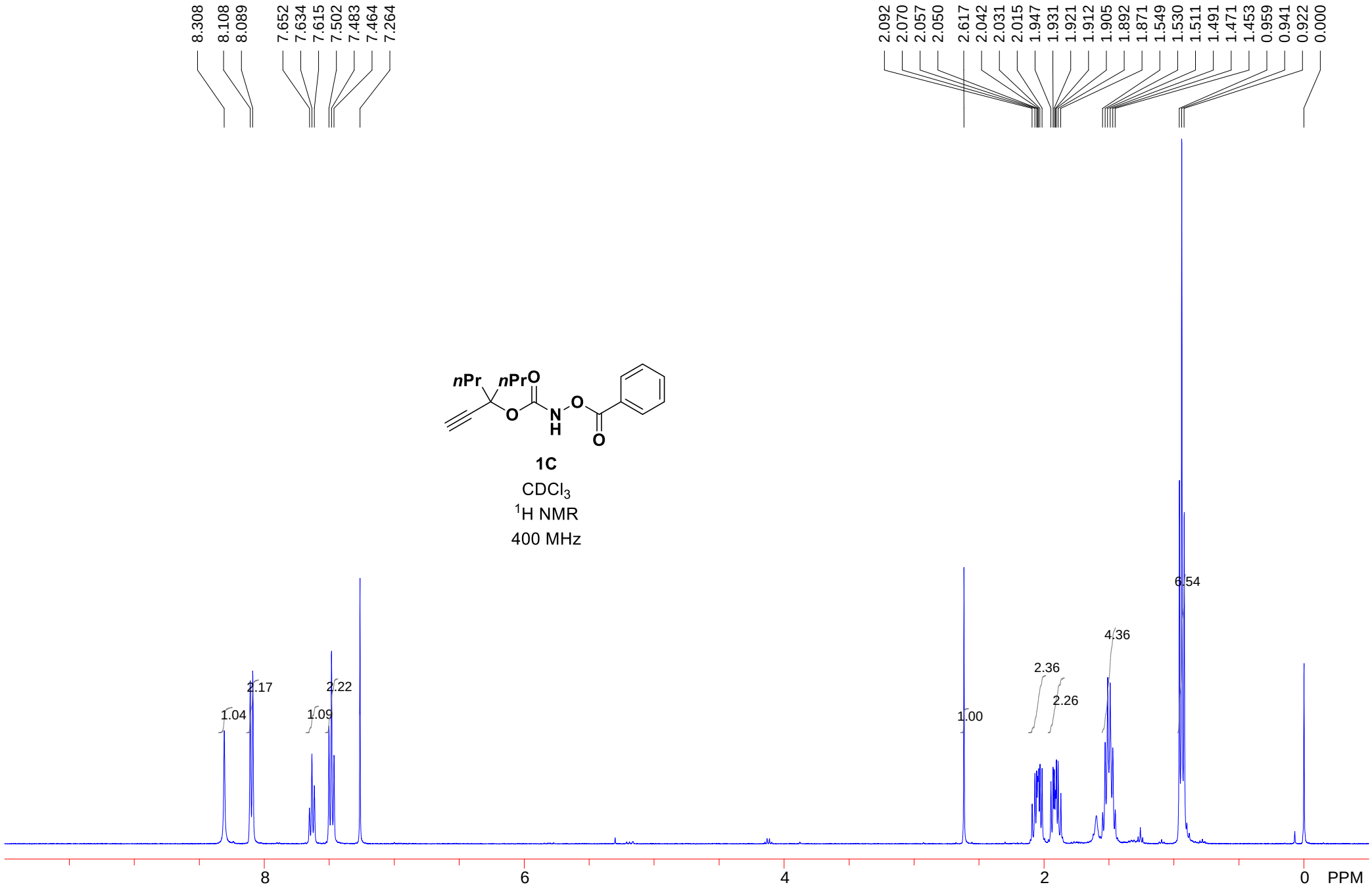
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2.050
2.617
2.042
2.031
2.015
1.947
1.931
1.921
1.912
1.905
1.892
1.871
1.549
1.530
1.511
1.491
1.471
1.453
0.959
0.941
0.922
0.000
nPr
nPr
O
O
N
H
O
O
1C
CDCl3
1H NMR
400 MHz
1.04
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1.00
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4.36
6.54
8
6
4
2
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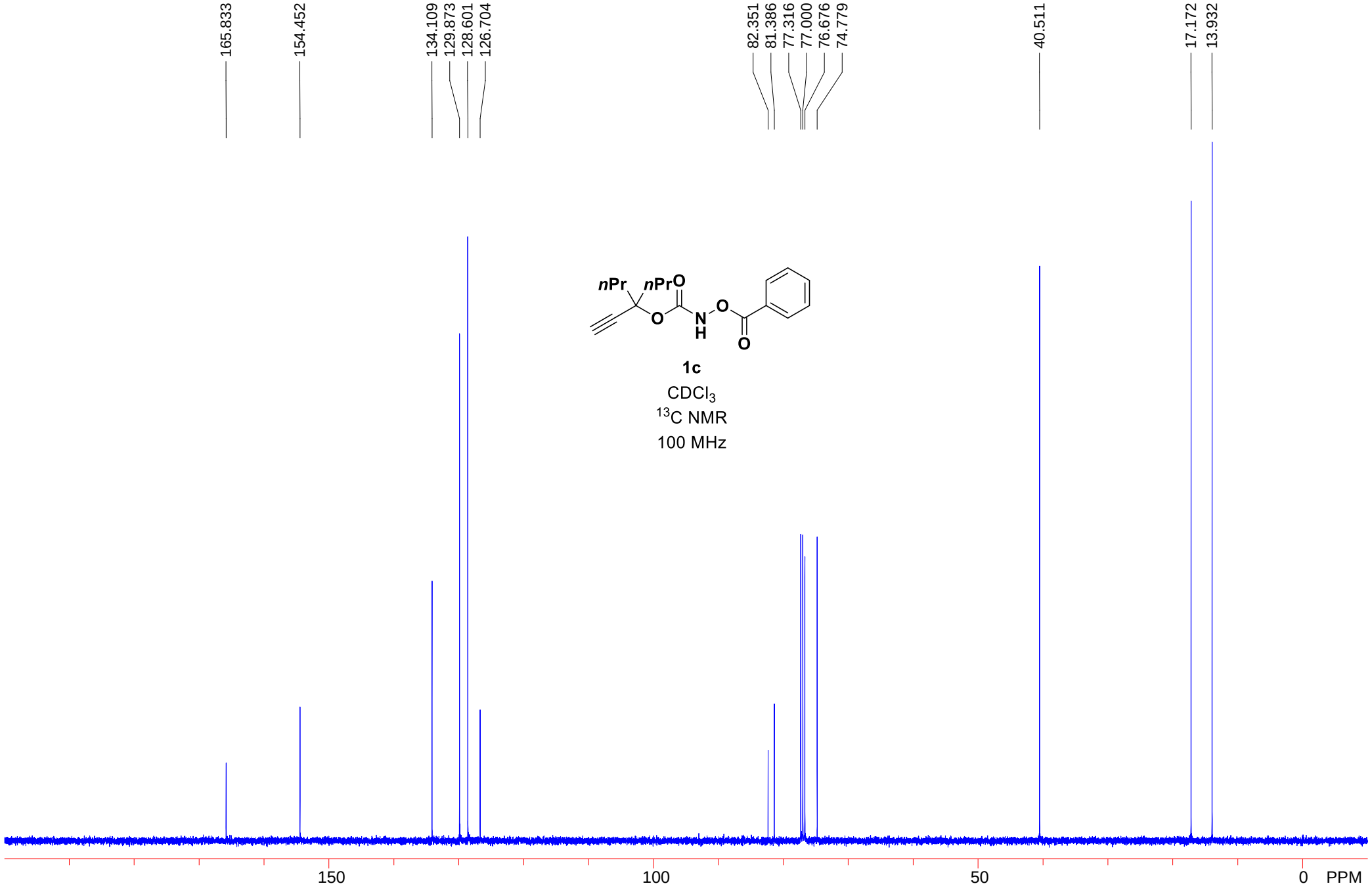
PPM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13.932
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100
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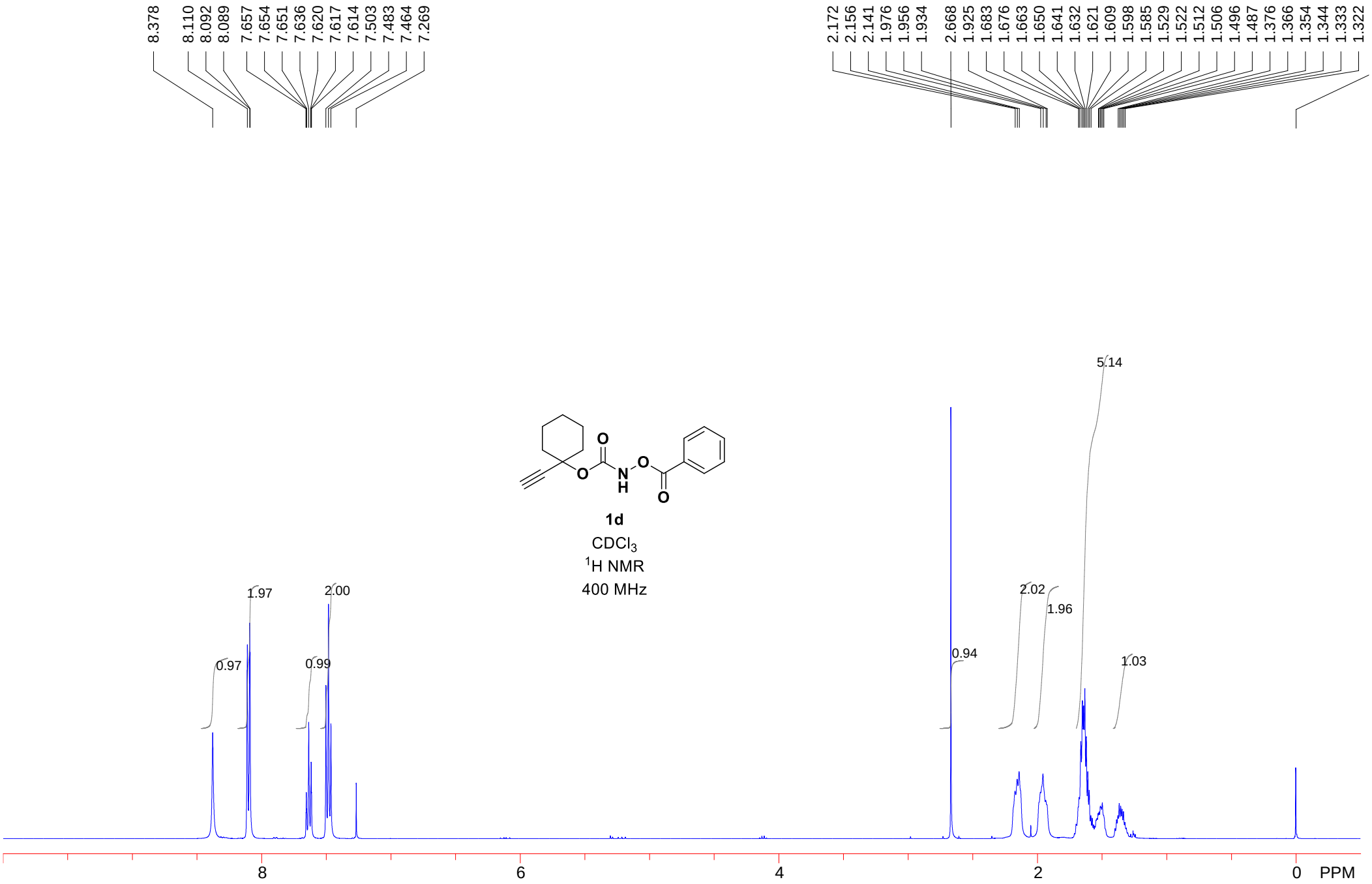
PPM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1.956
1.934
2.668
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1.663
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1.641
1.632
1.621
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1.585
1.529
1.522
1.512
1.506
1.496
1.487
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1.366
1.354
1.344
1.333
1.322
O
O
N
H
O
O
1d
CDCl3
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400 MHz
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6
4
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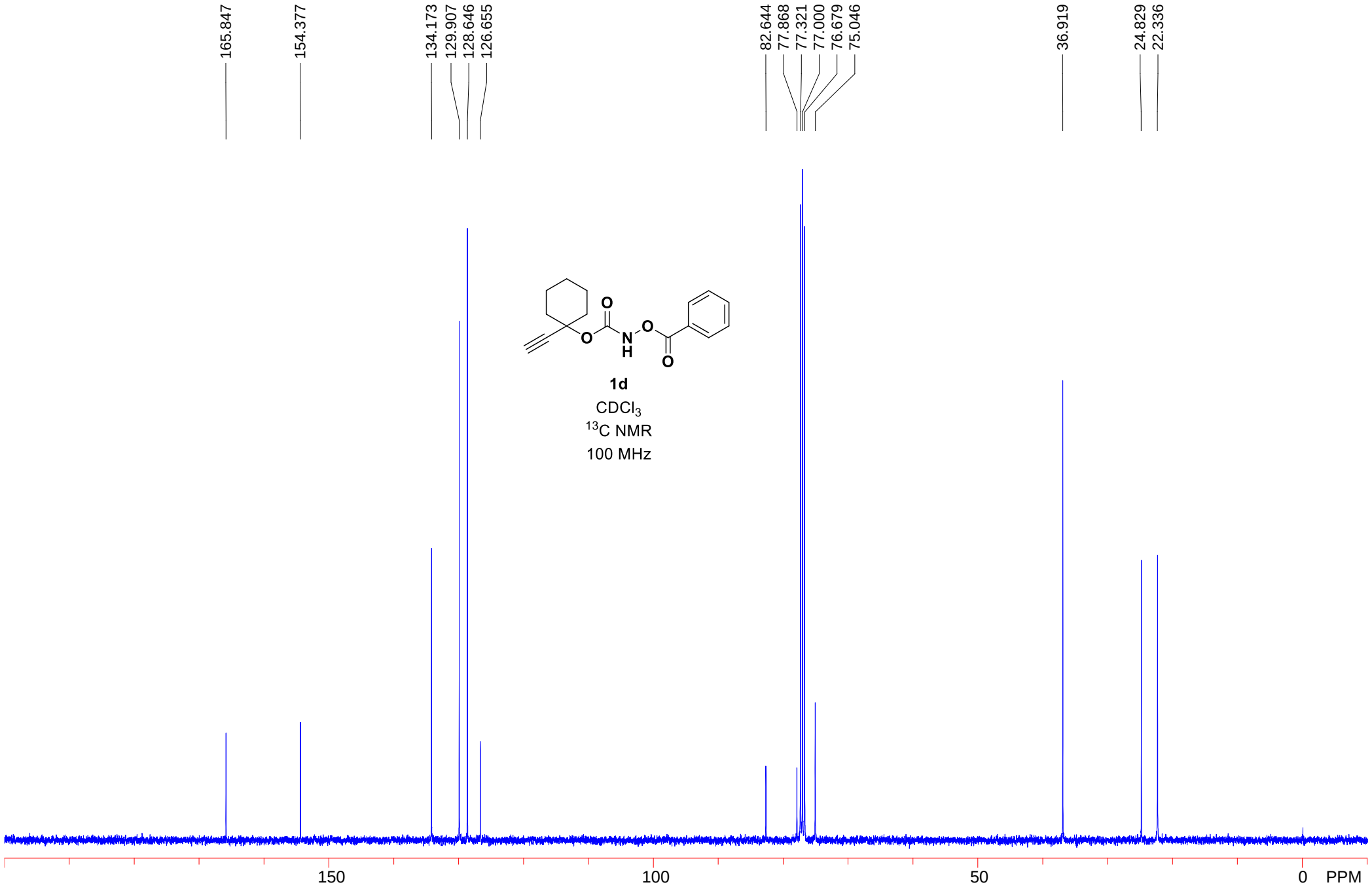
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100
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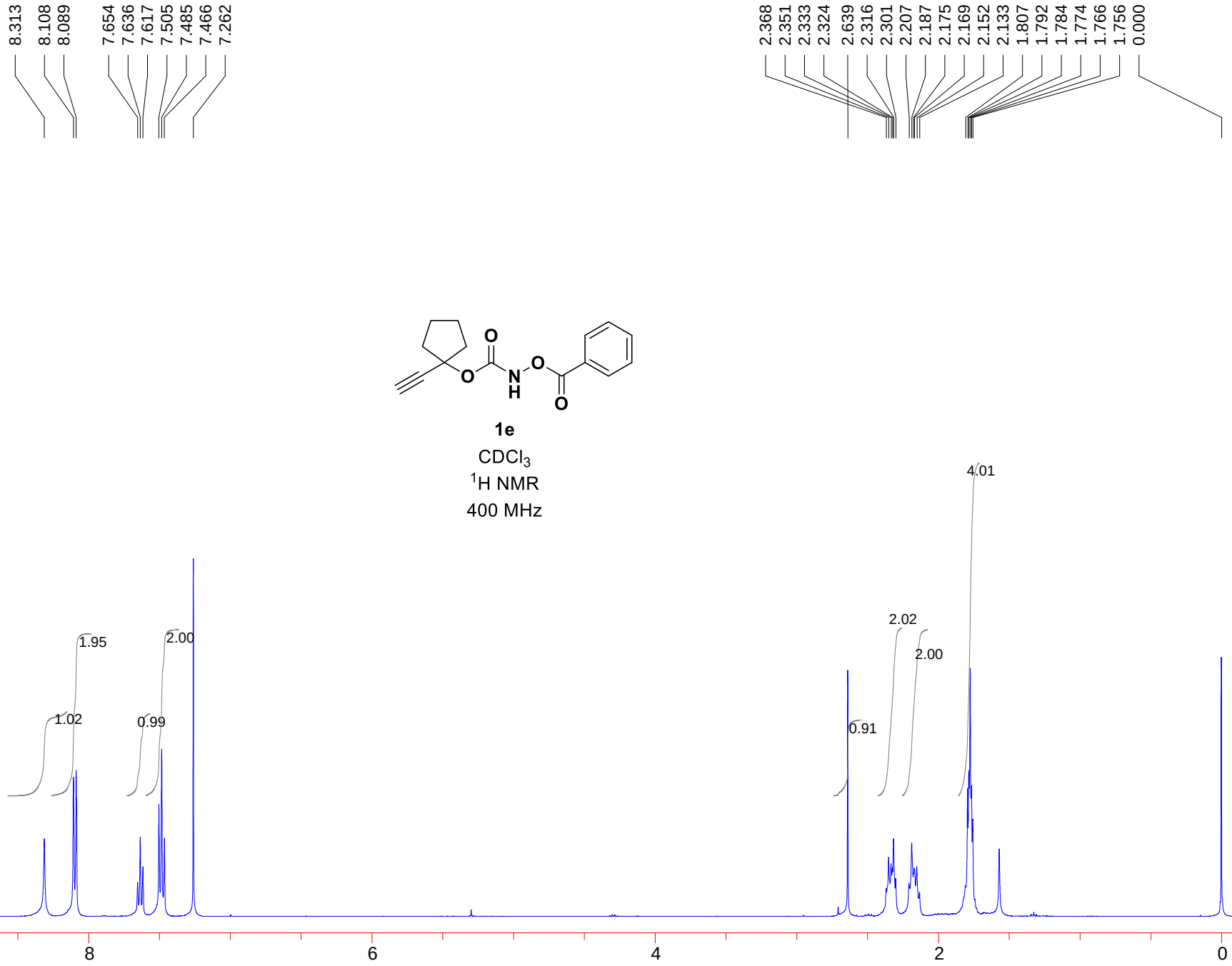
PPM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2.301
2.207
2.187
2.175
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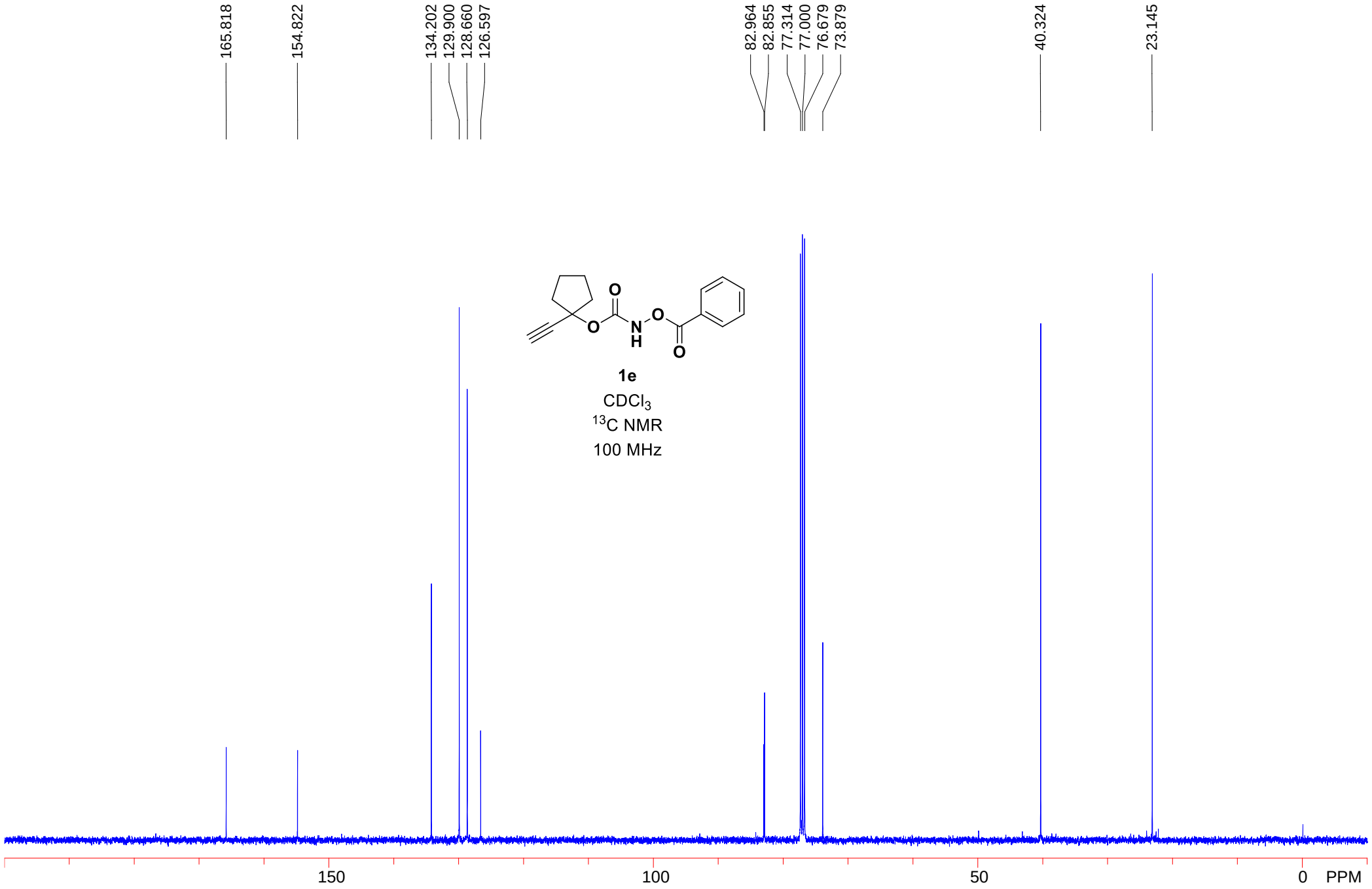
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100
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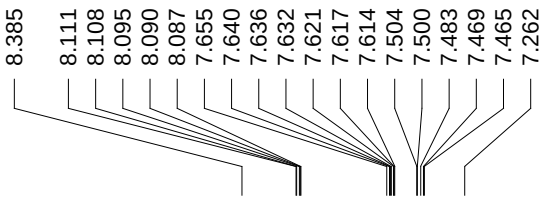
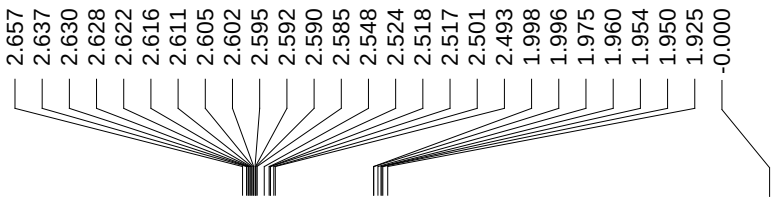
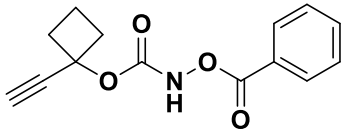
PPM





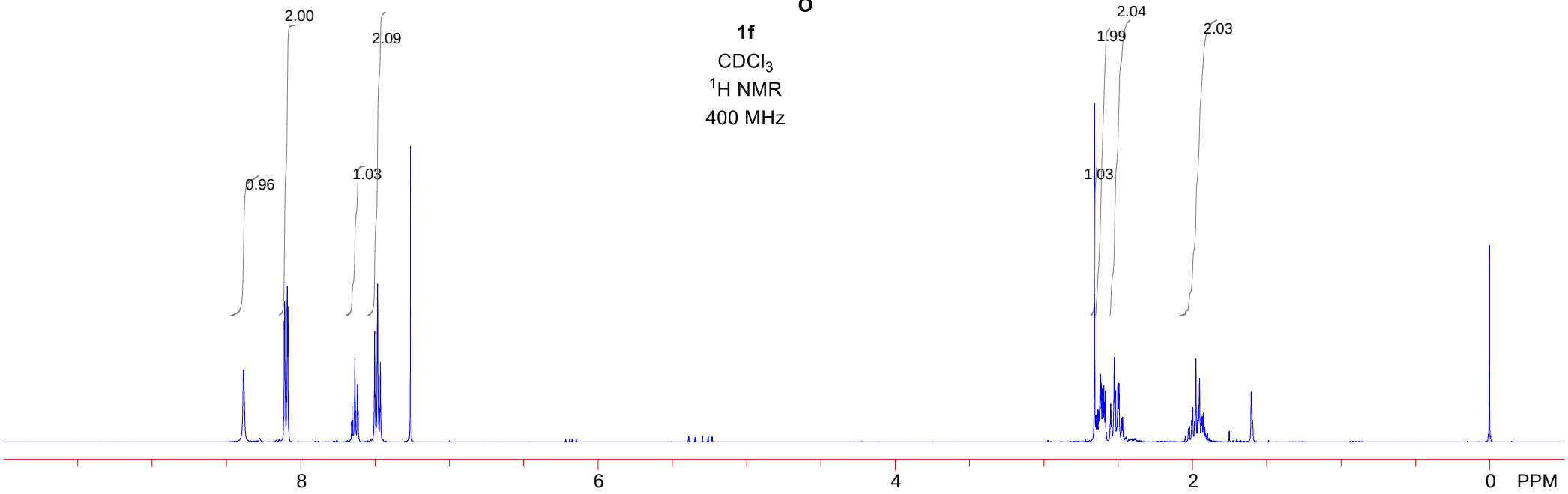


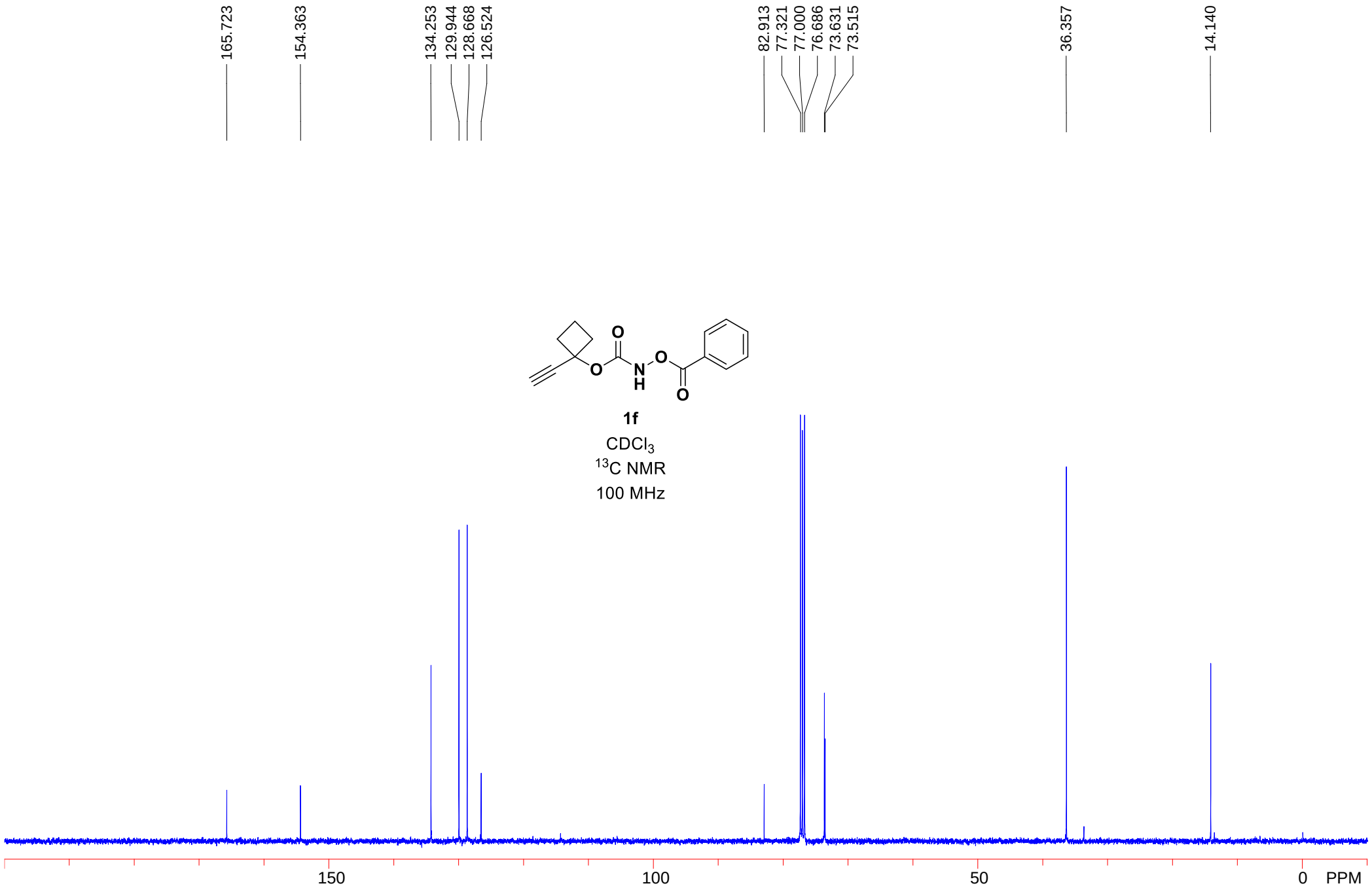
1f

$CDCl_3$

^1H NMR

400 MHz

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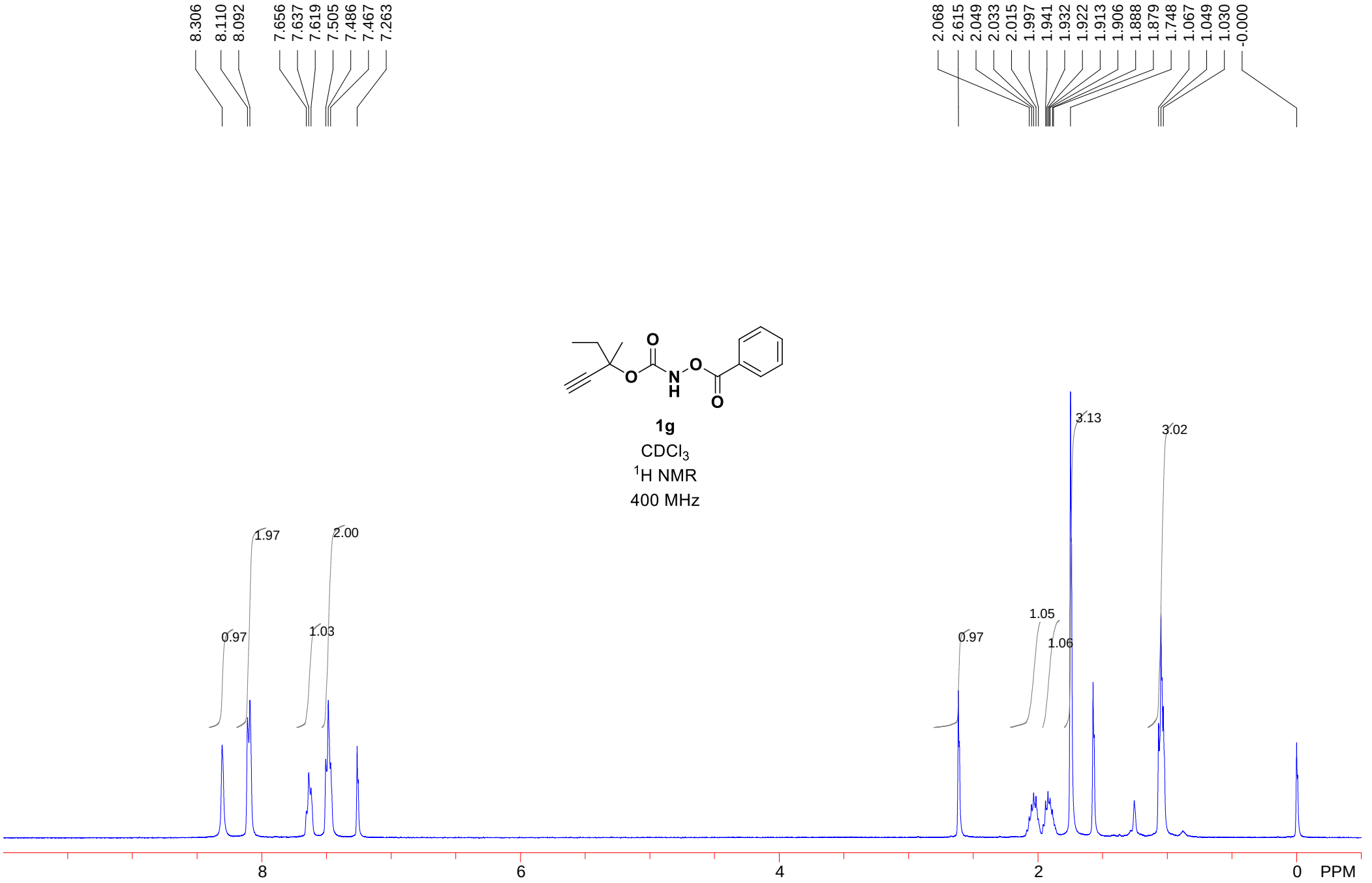
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1H NMR
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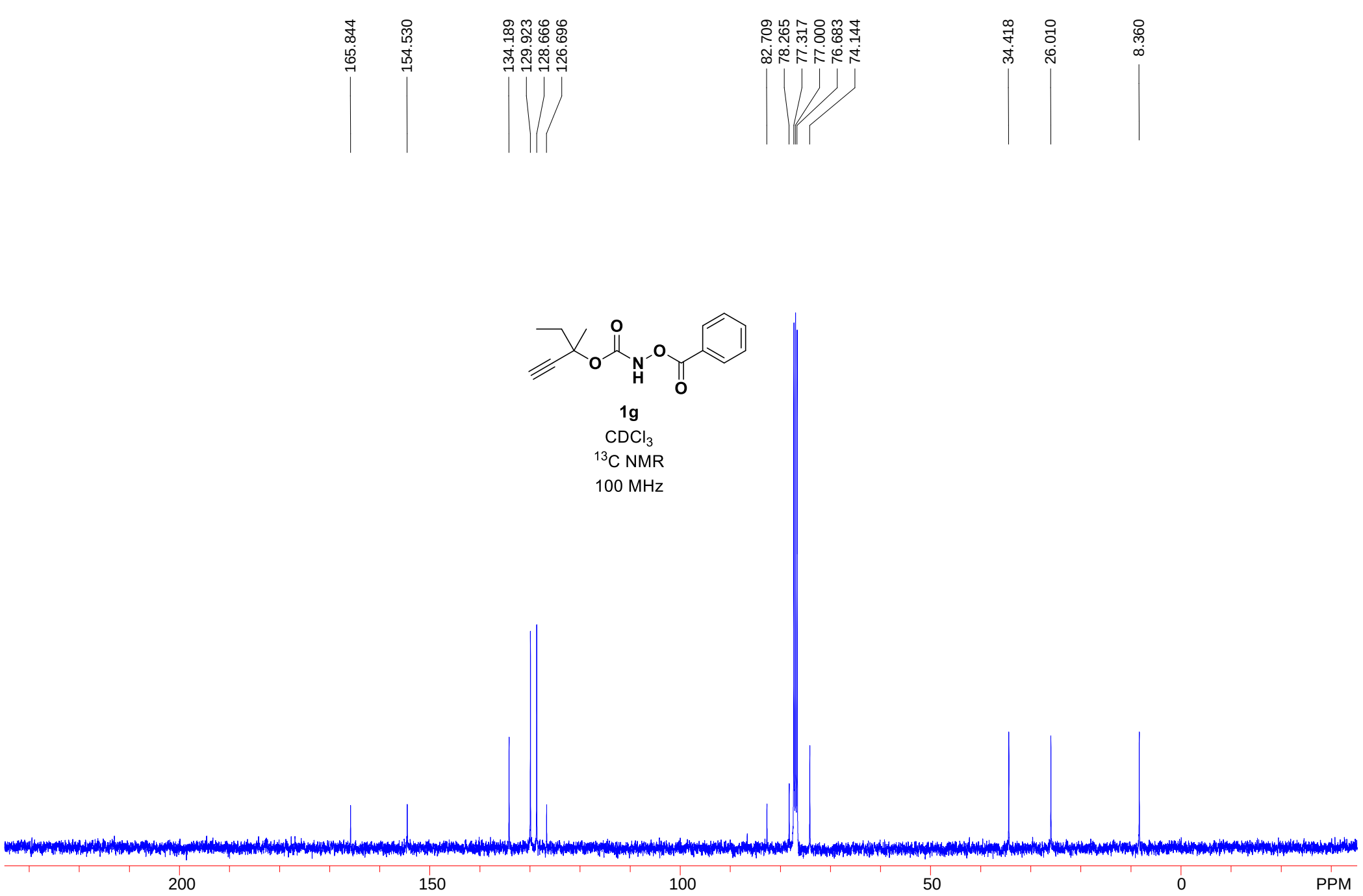
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200
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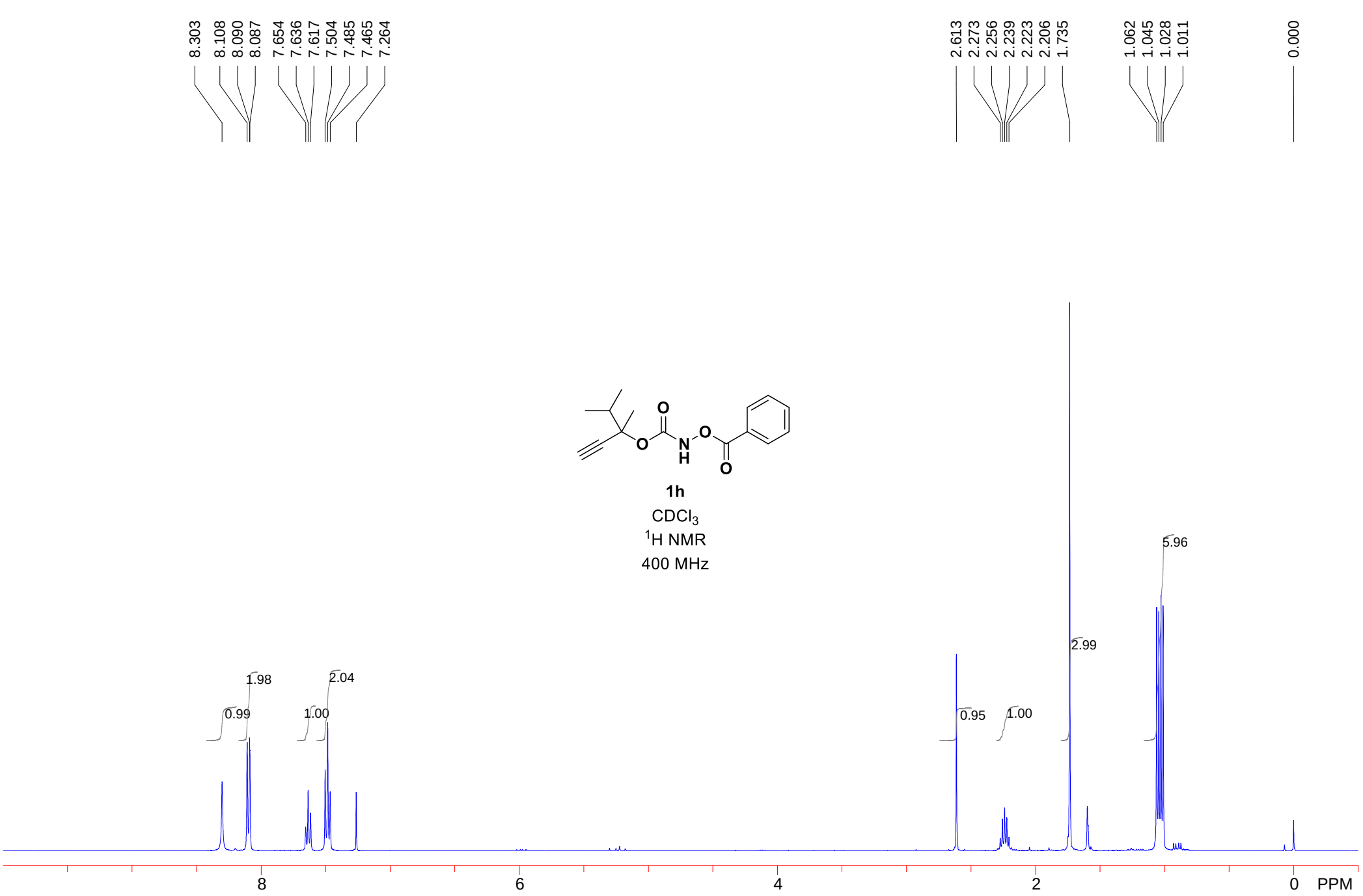
PPM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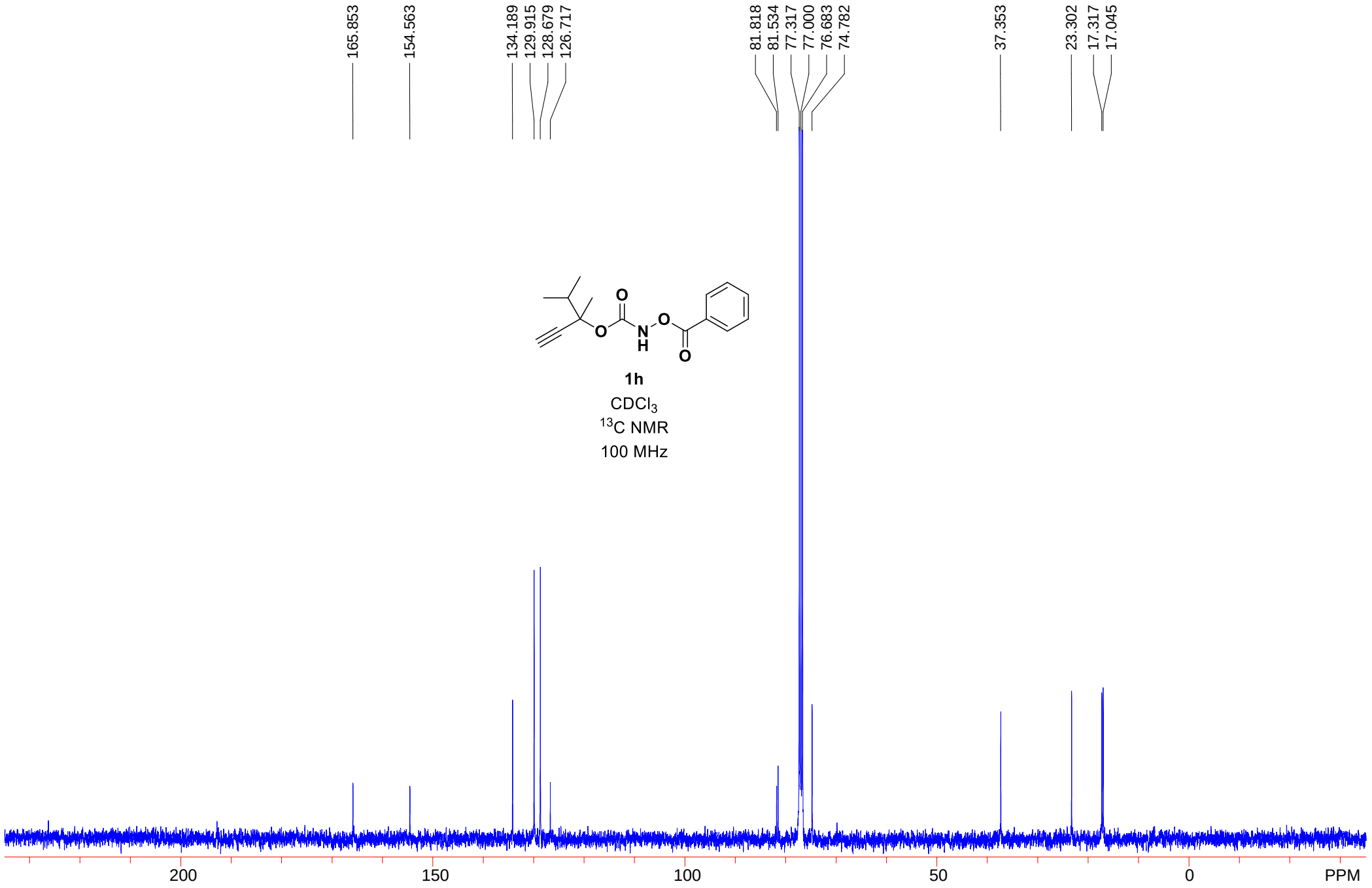
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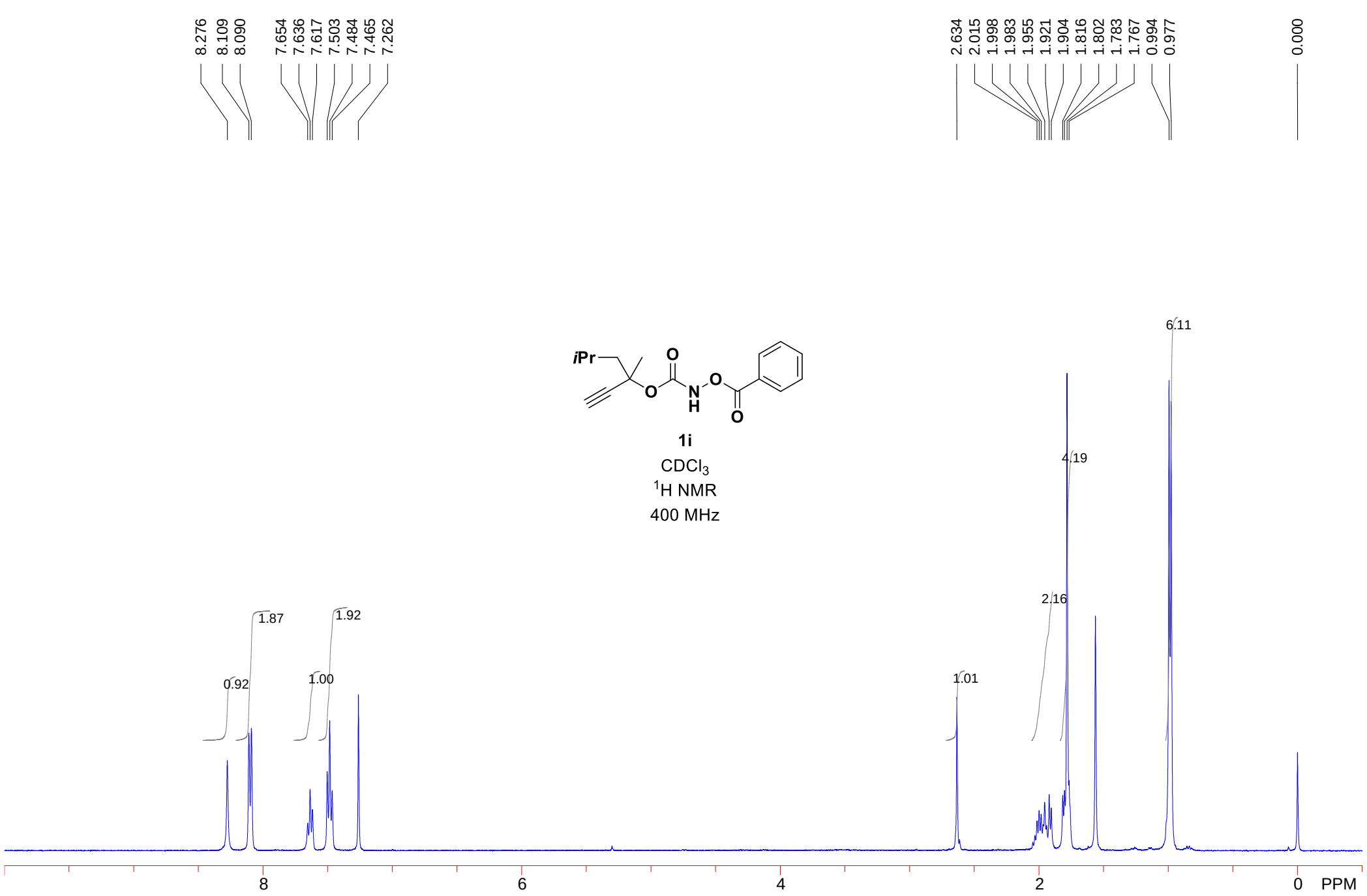
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H
O
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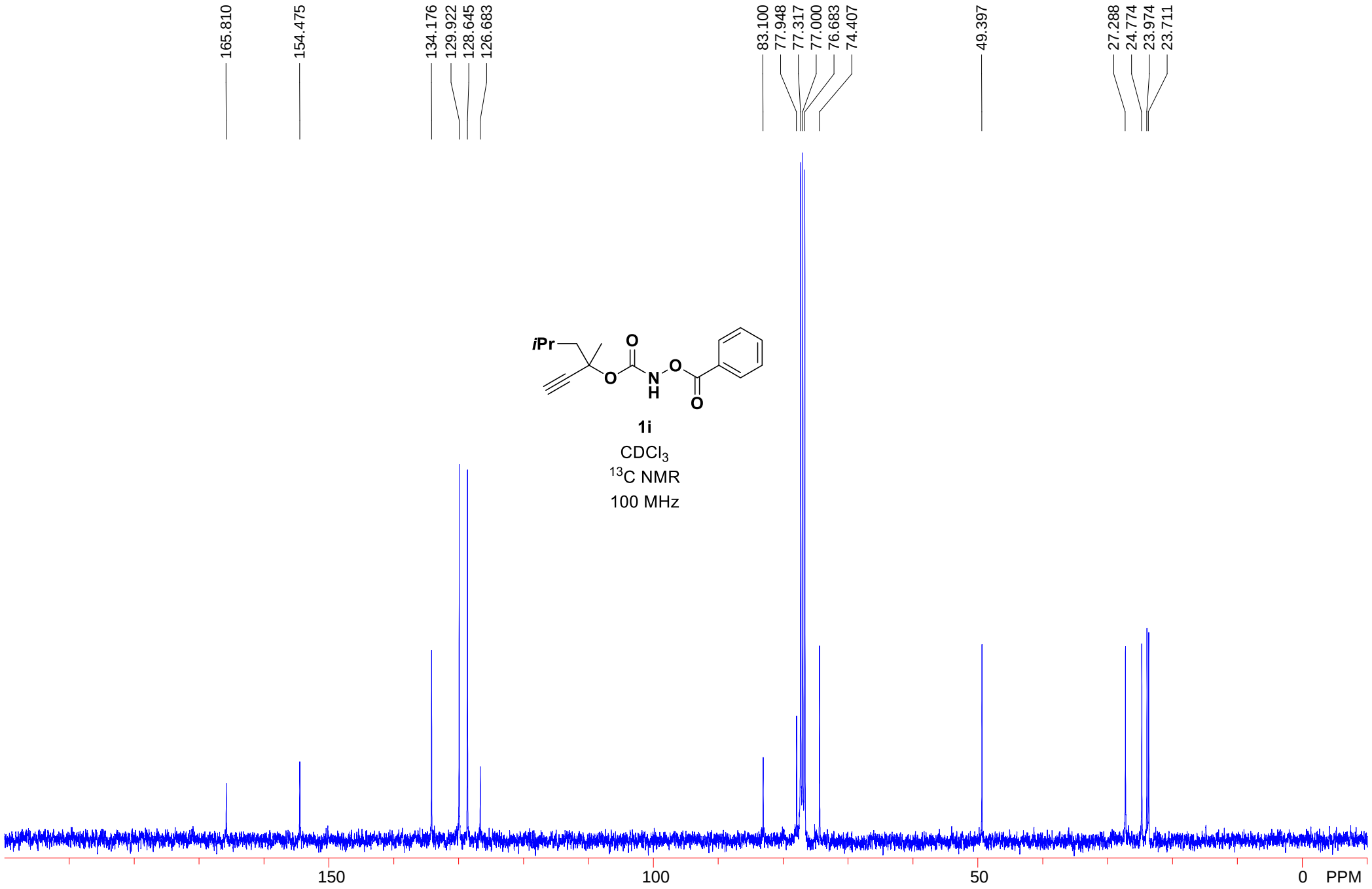
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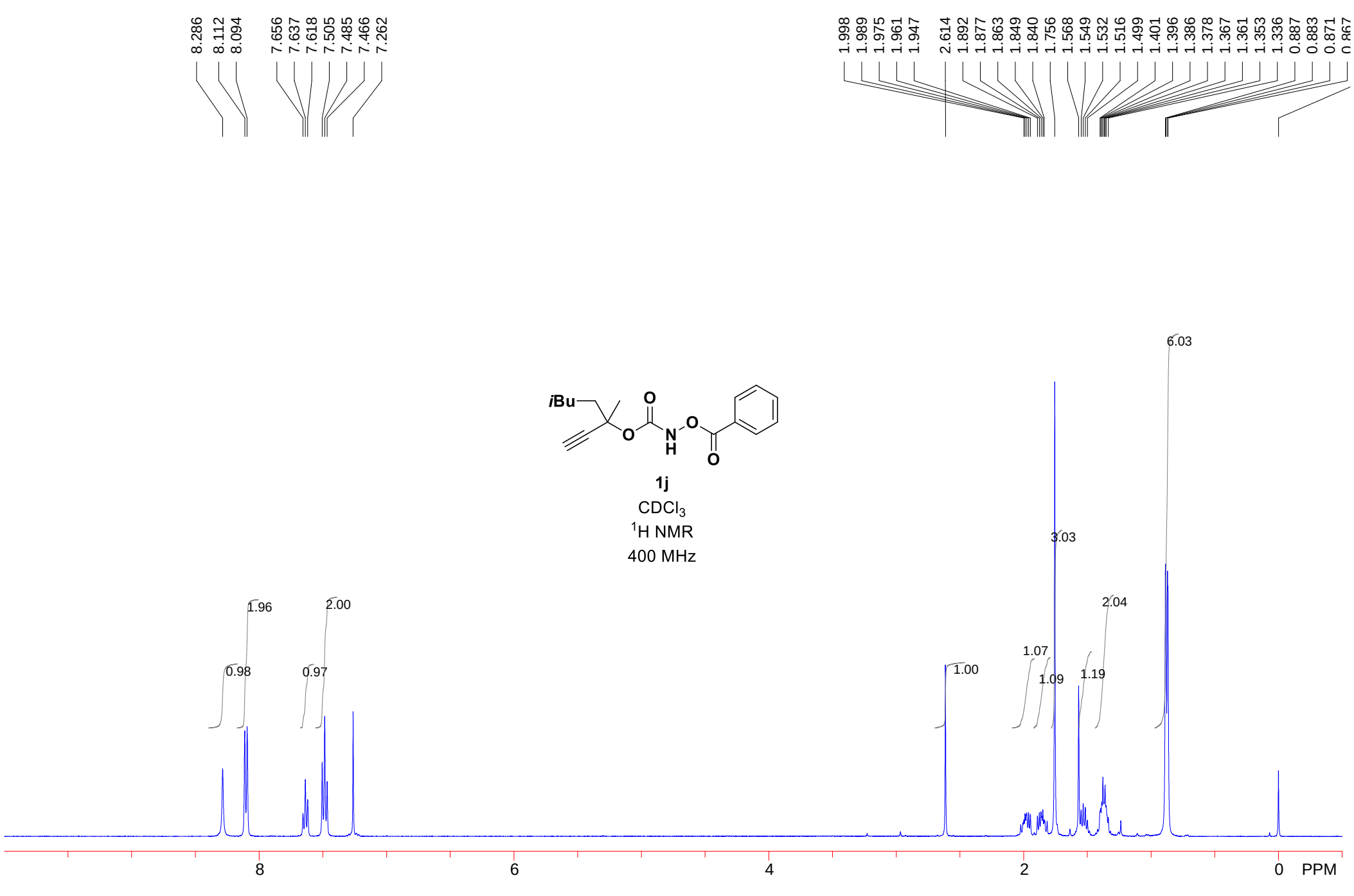
PPM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2
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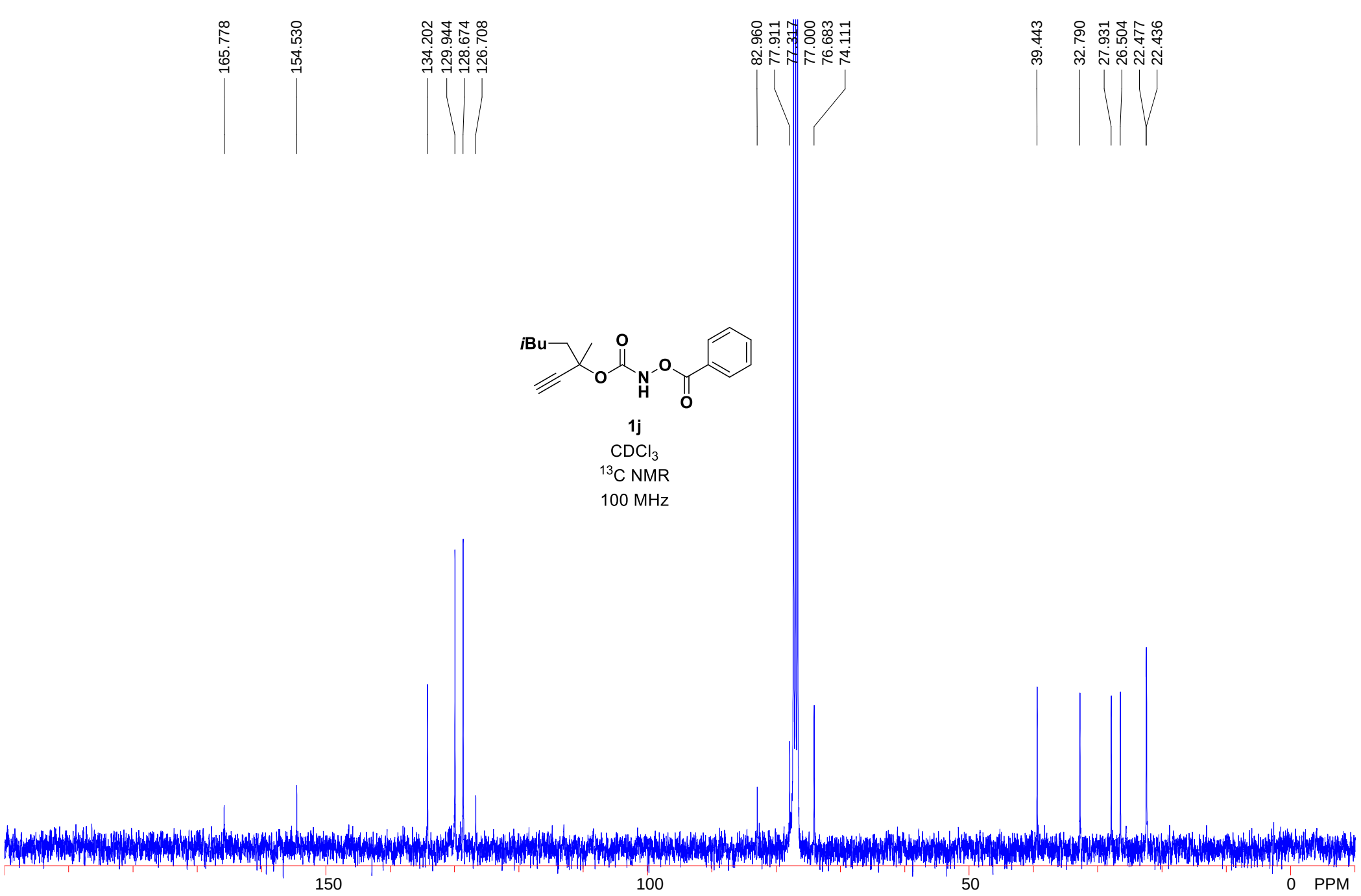
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77.000
76.683
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39.443
32.790
27.931
26.504
22.477
22.436
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O
N
H
O
O
1j
CDCl3
13C NMR
100 MHz
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100
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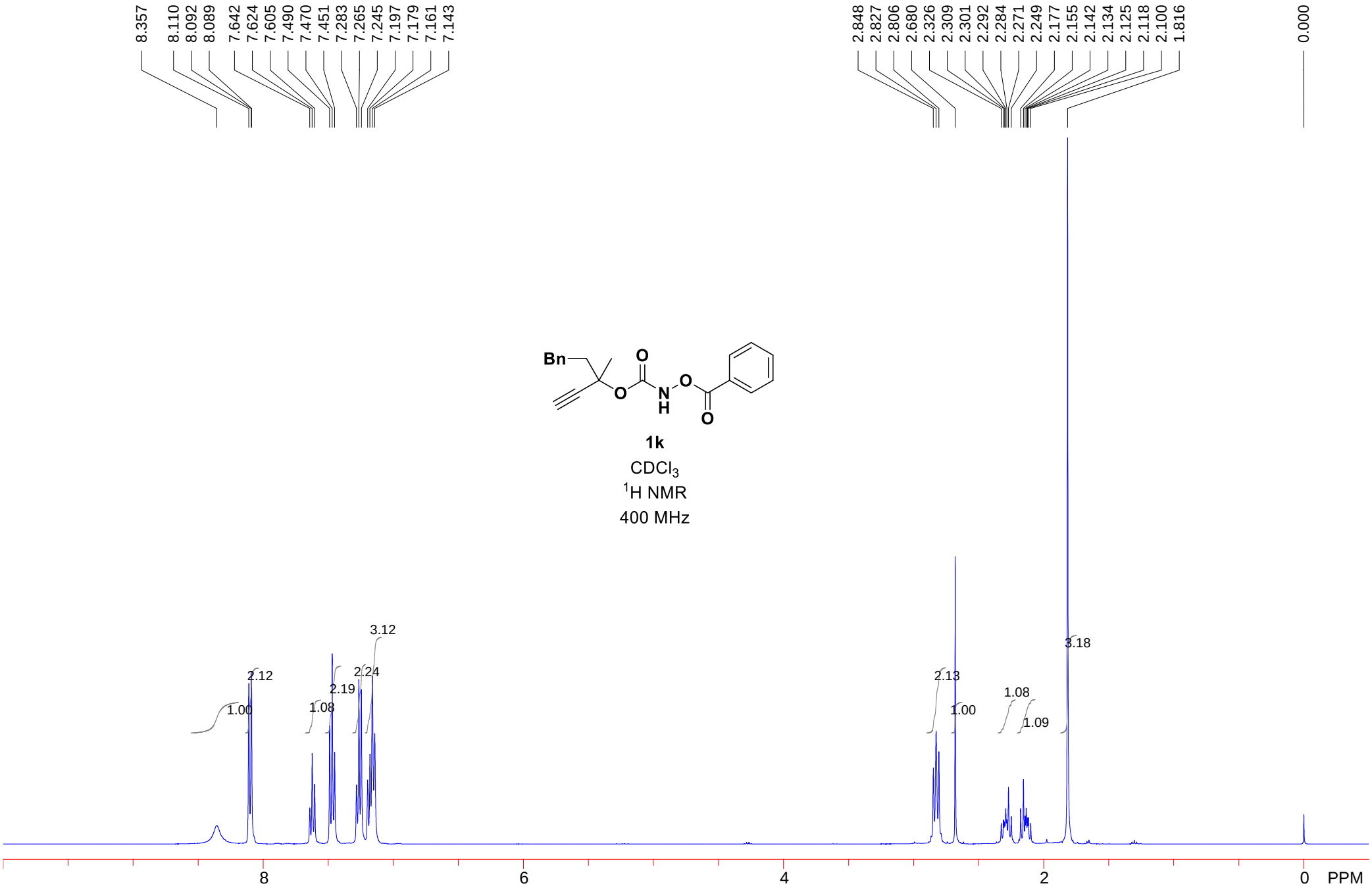
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7.490
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7.245
7.197
7.179
7.161
7.143
2.848
2.827
2.806
2.680
2.326
2.309
2.301
2.292
2.284
2.271
2.249
2.177
2.155
2.142
2.134
2.125
2.118
2.100
1.816
0.000
Bn
O
O
N
H
O
O
1k
CDCl3
1H NMR
400 MHz
1.00
2.12
1.08
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3.12
2.13
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1.09
3.18
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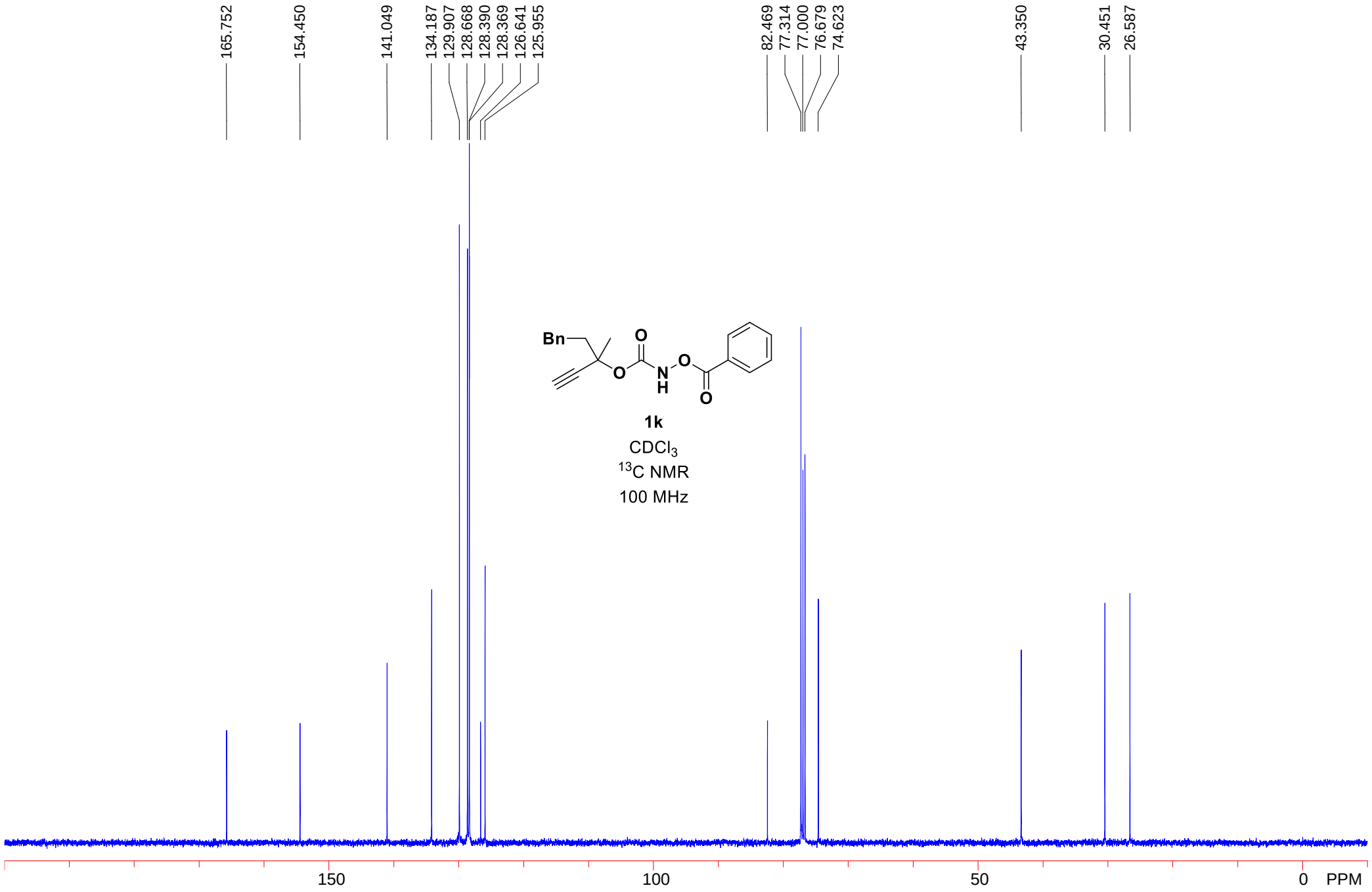
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76.679
74.623
43.350
30.451
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O
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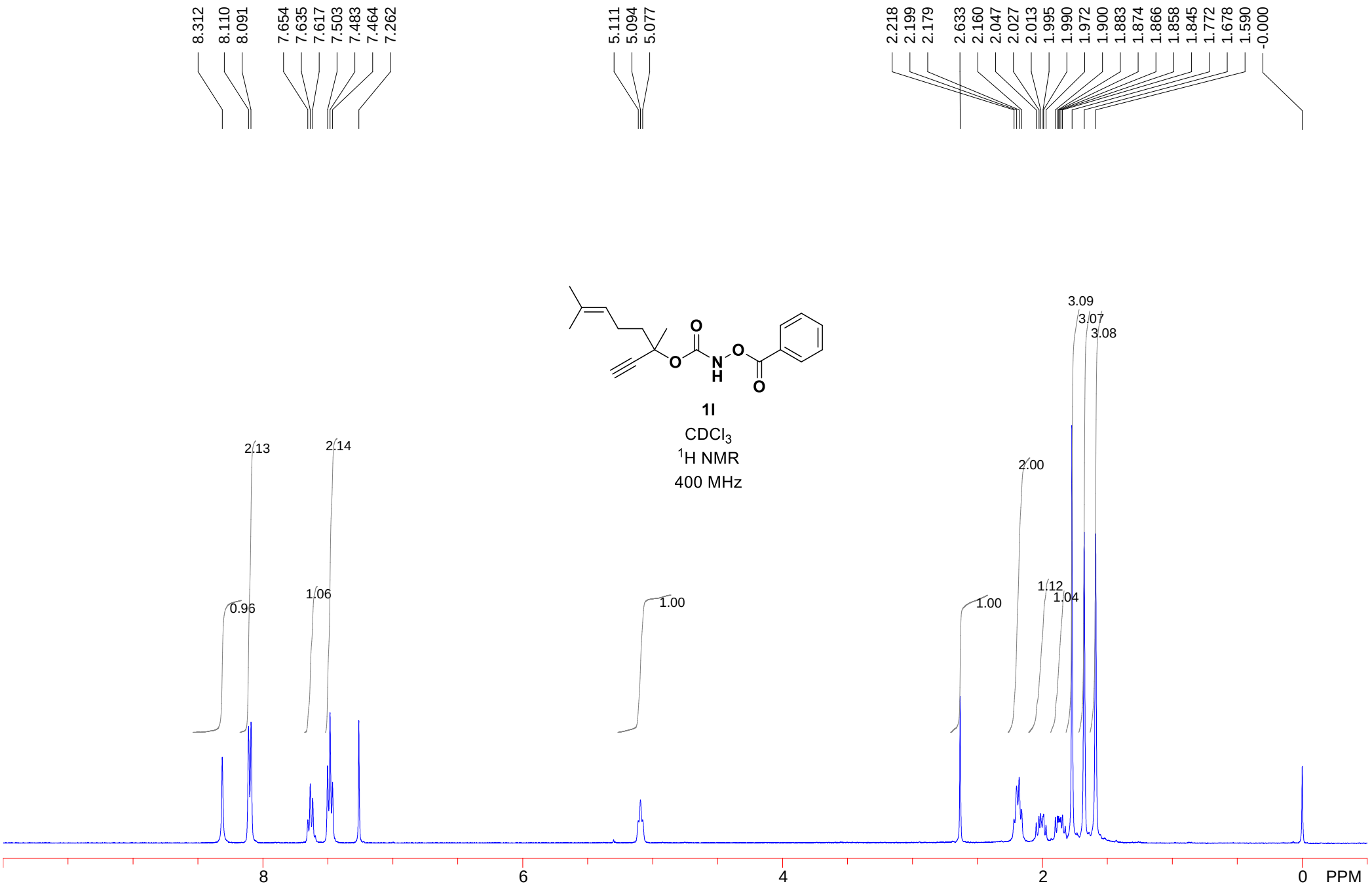
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7.483
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5.111
5.094
5.077
2.218
2.199
2.179
2.633
2.160
2.047
2.027
2.013
1.995
1.990
1.972
1.900
1.883
1.874
1.866
1.858
1.845
1.772
1.678
1.590
-0.000
O
O
N
H
O
O
1l
CDCl3
1H NMR
400 MHz
2.13
2.14
0.96
1.06
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1.04
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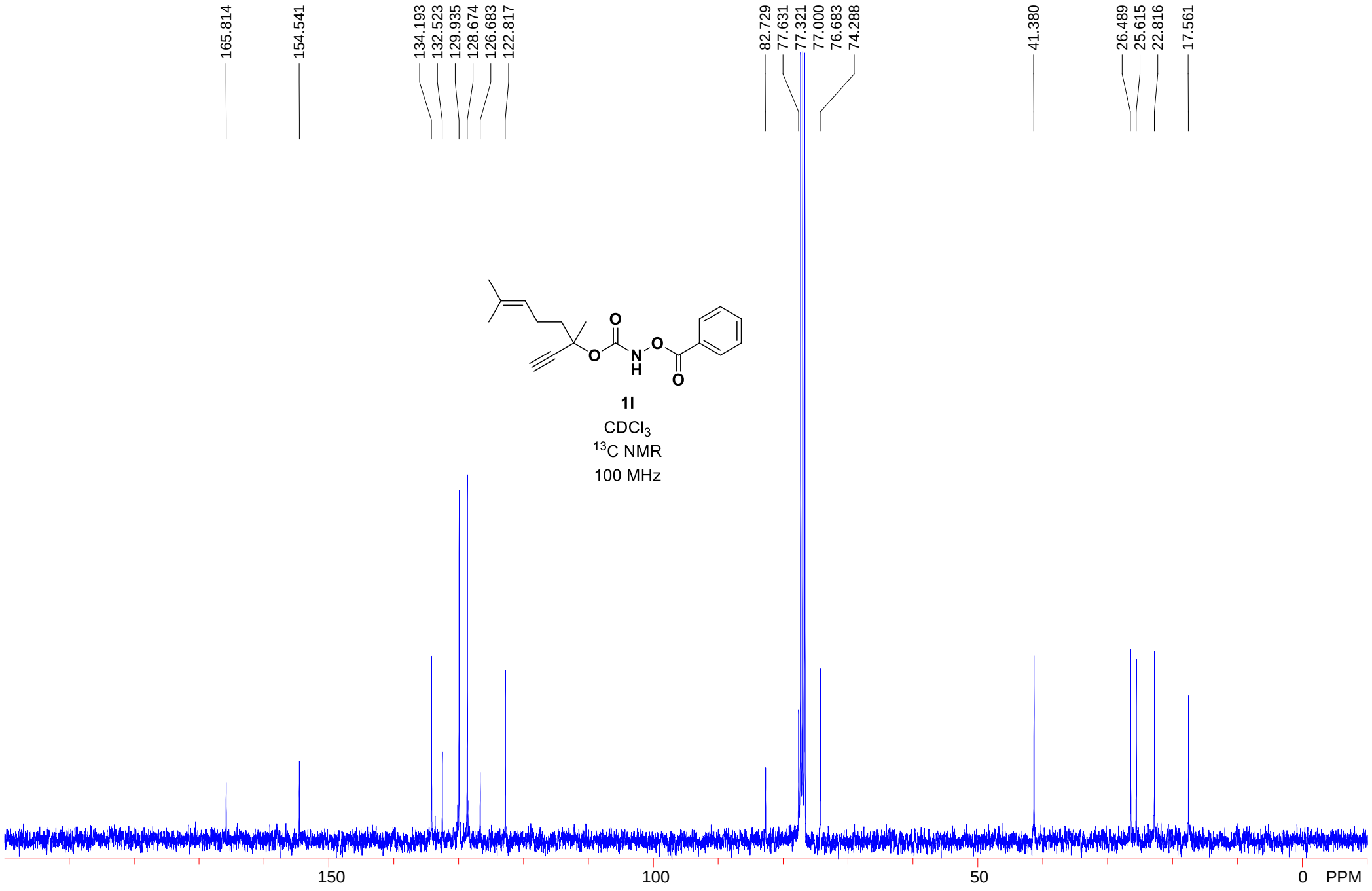
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76.683
74.288
41.380
26.489
25.615
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O
N
H
O
O
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CDCl3
13C NMR
100 MHz
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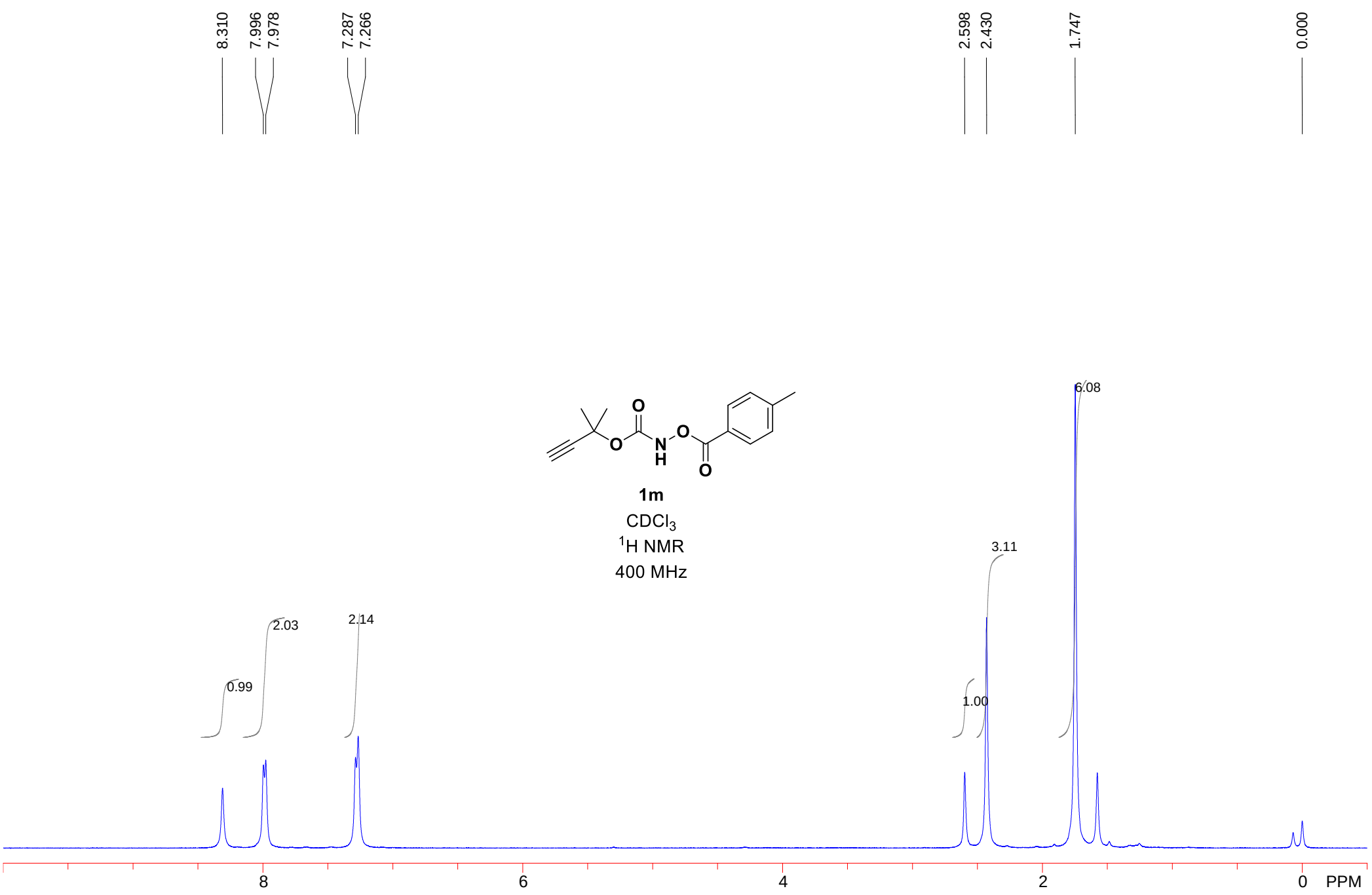
PPM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7.978
7.287
7.266
2.598
2.430
1.747
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1m
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0.99
2.03
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6.08
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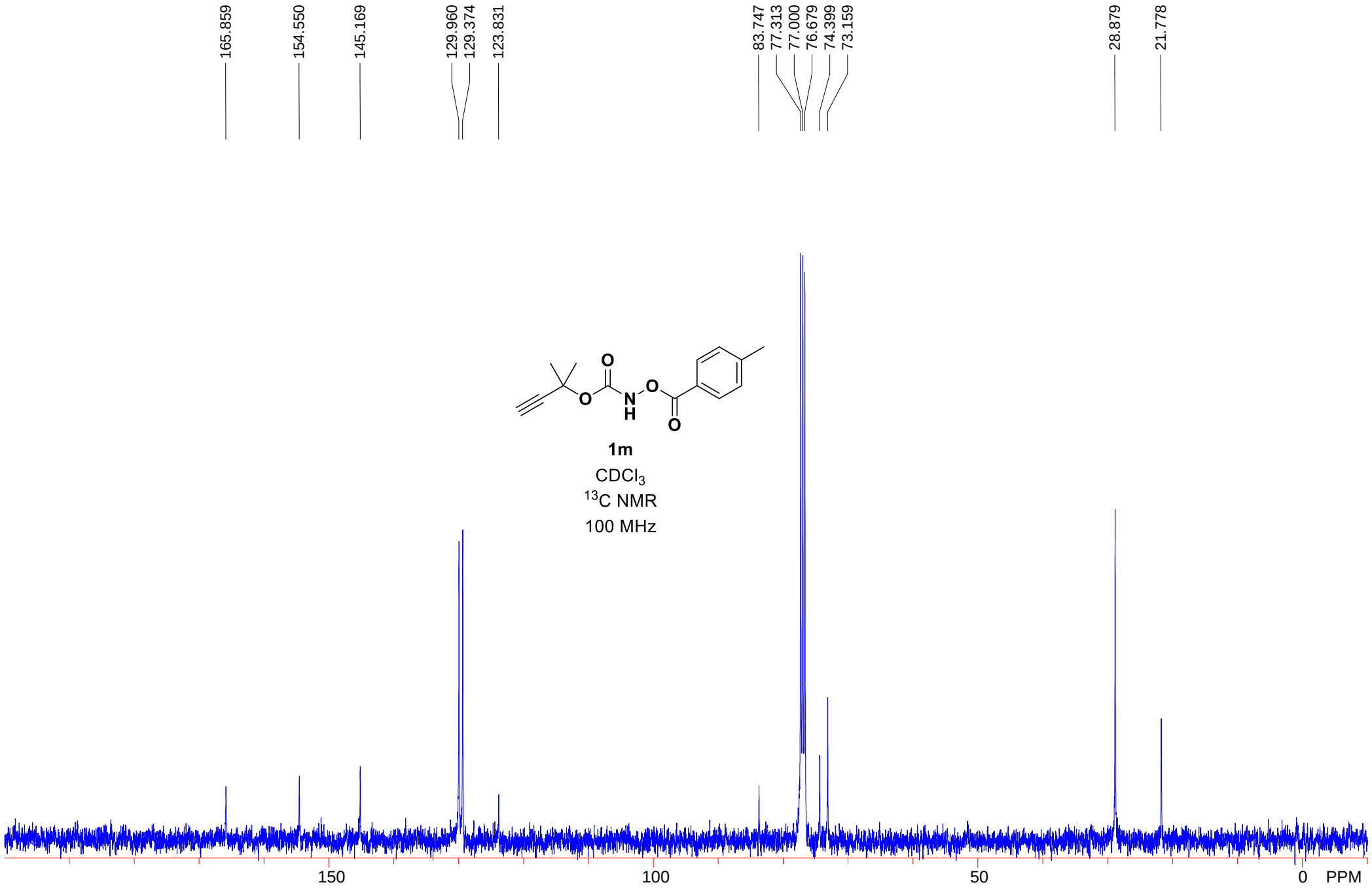
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77.313
77.000
76.679
74.399
73.159
28.879
21.778
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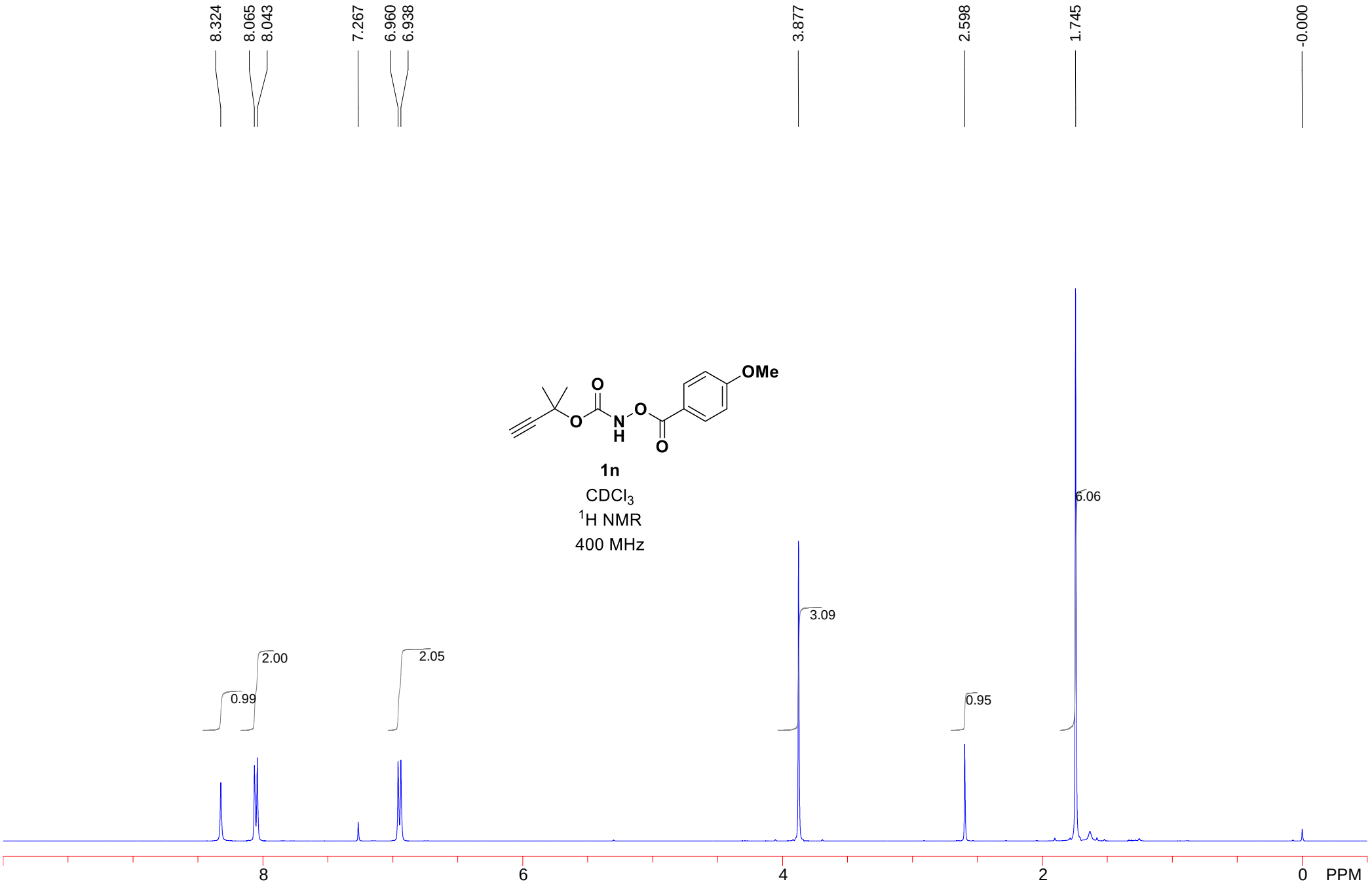
O
N
H
O
O
1m
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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7.267
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6.938
3.877
2.598
1.745
-0.000
OMe
O
O
N
H
O
O
1n
CDCl3
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400 MHz
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2.00
2.05
3.09
0.95
6.06
8
6
4
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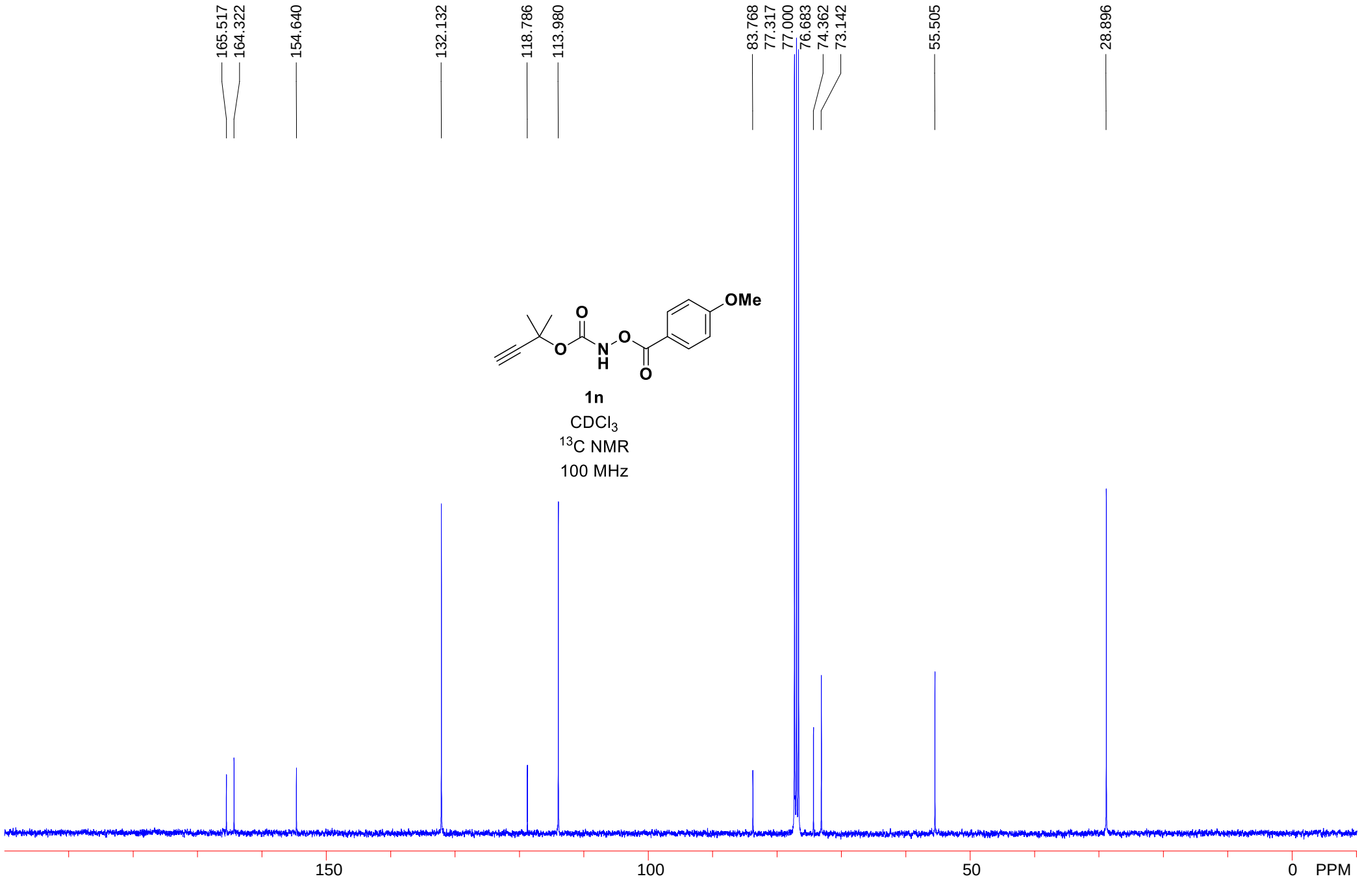
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154.640
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113.980
83.768
77.317
77.000
76.683
74.362
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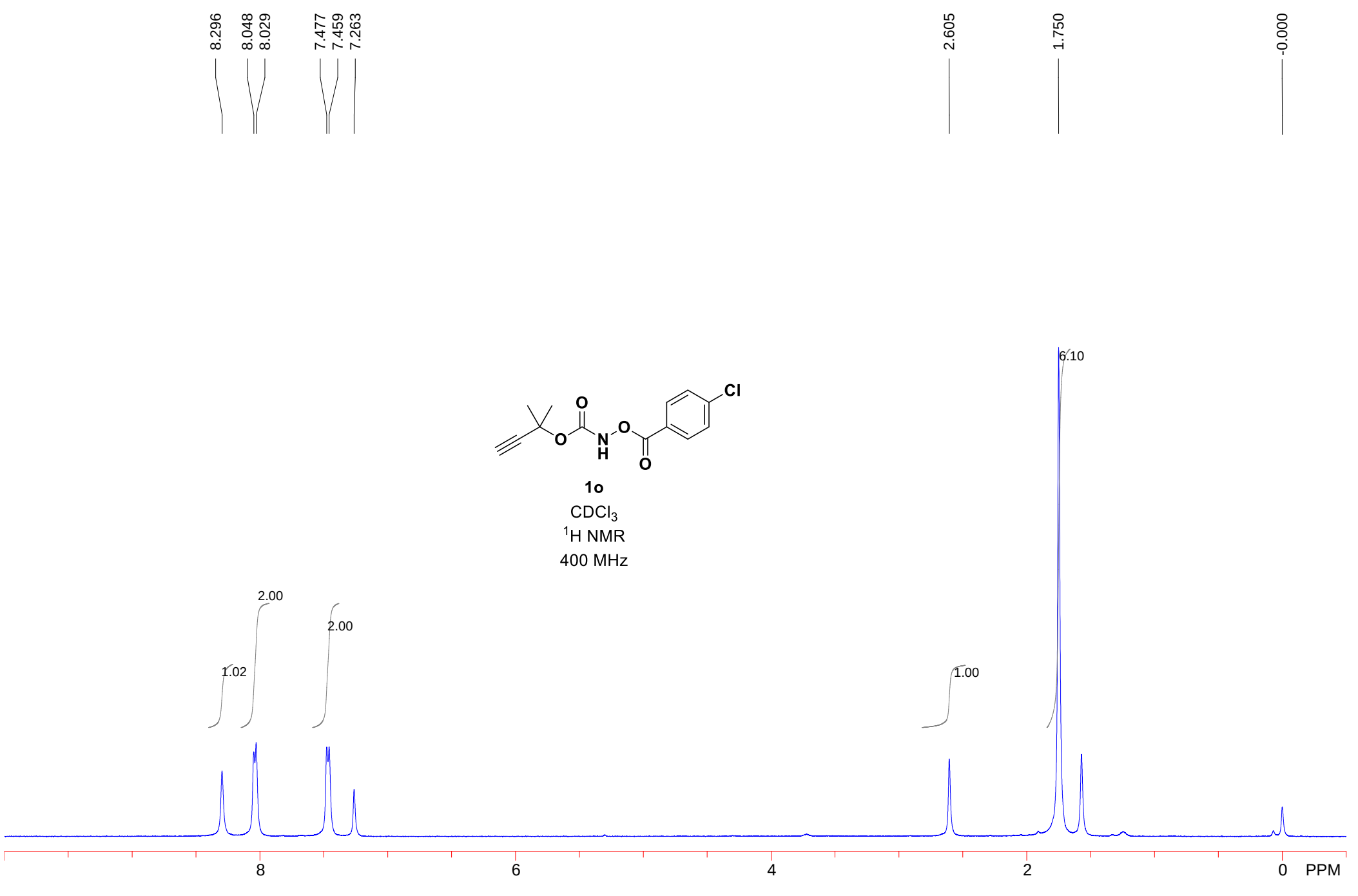
55.505
28.896
OMe
O
O
N
H
O
O
O
1n
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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7.263
2.605
1.750
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Cl
O
O
N
H
O
O
1o
CDCl3
1H NMR
400 MHz
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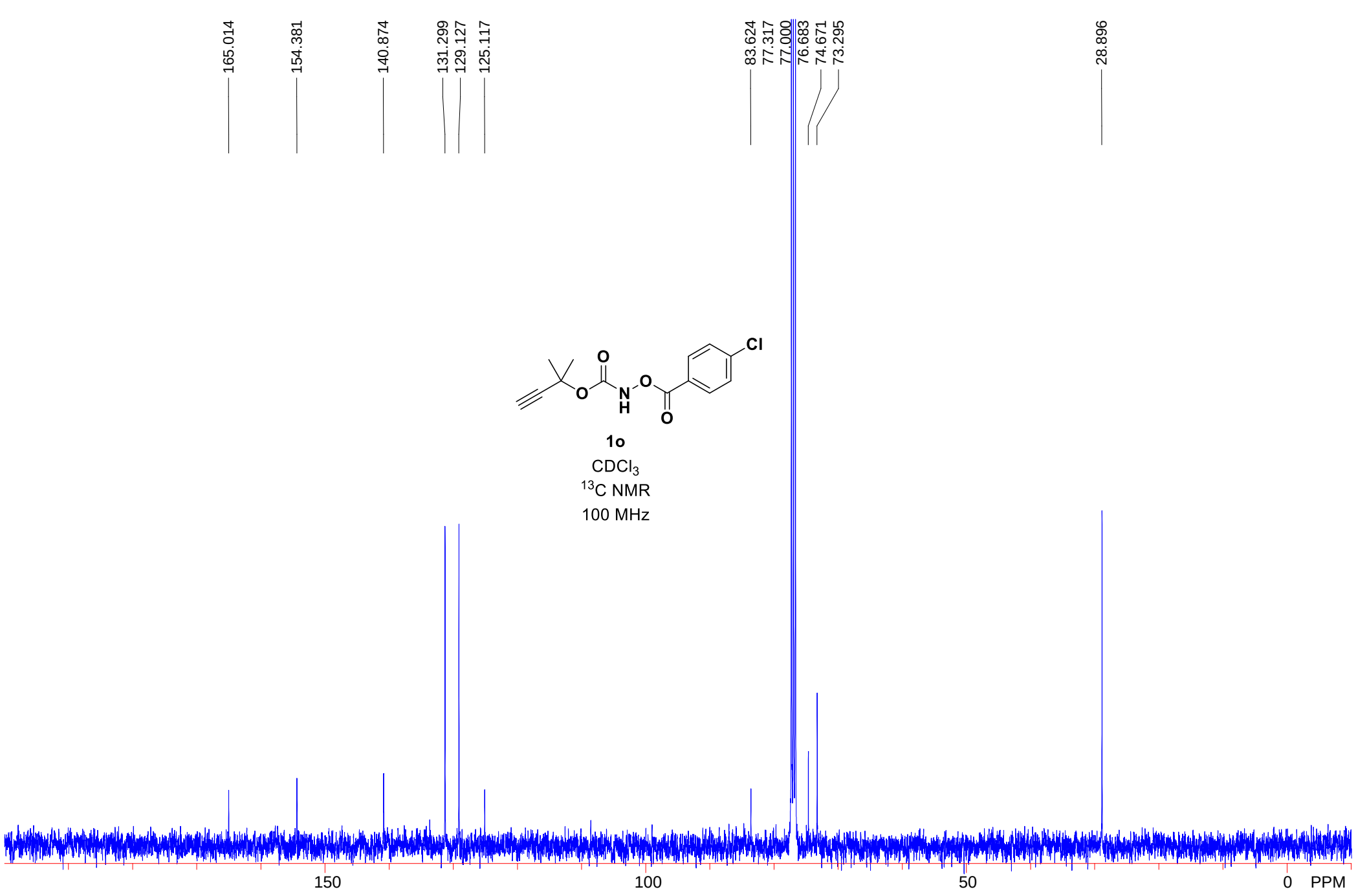
2.00
1.02
1.00
8
6
4
2
0
PPM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140.874
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77.317
77.000
76.683
74.671
73.295
28.896
Cl
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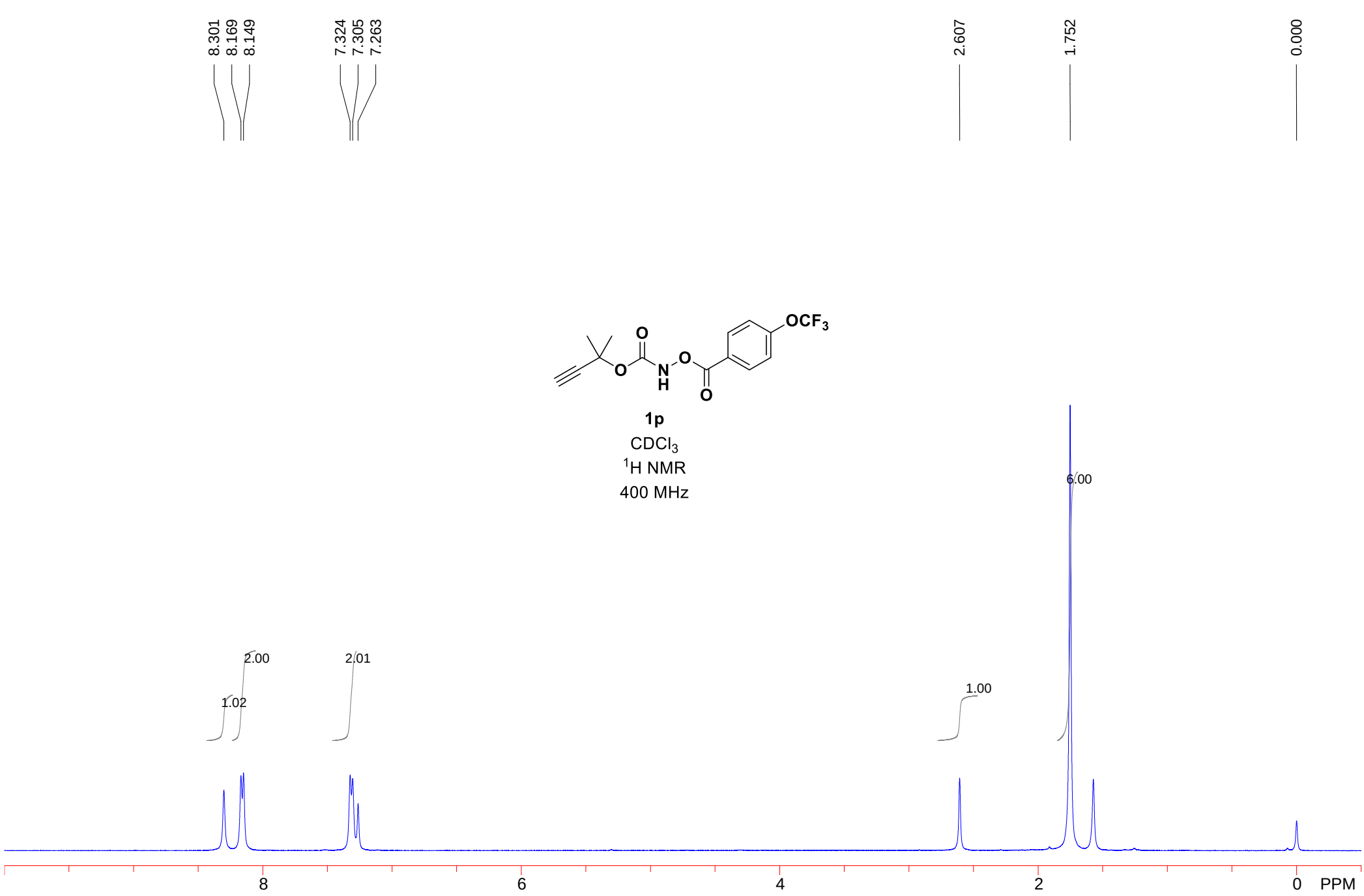
O
N
H
O
O
1o
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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2.607
1.752
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OCF3
O
O
N
H
O
O
1p
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1H NMR
400 MHz
6.00
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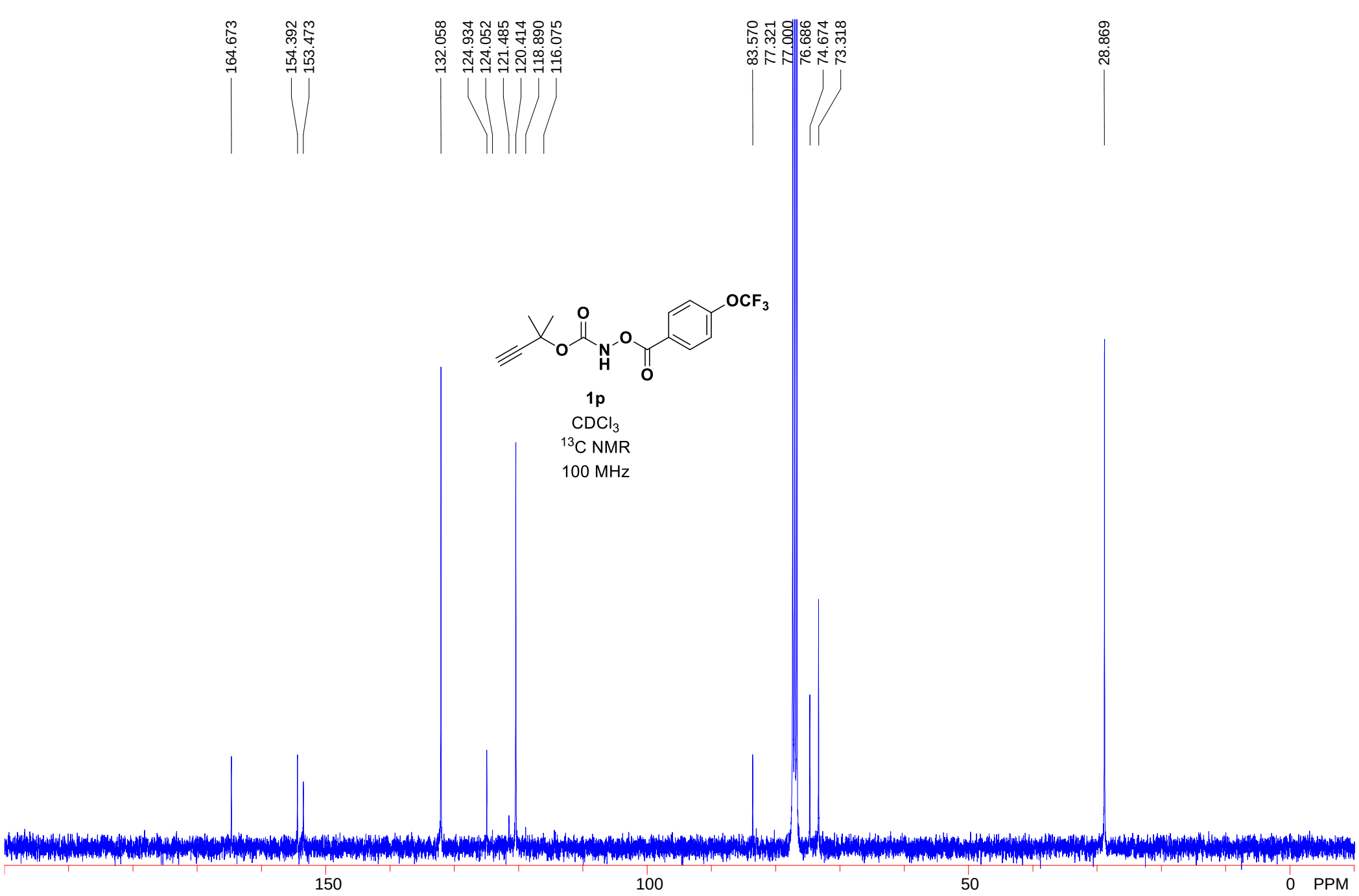
1.02
1.00
8
6
4
2
0
PPM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77.000
76.686
74.674
73.318
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N
H
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13C NMR
100 MHz
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100
50
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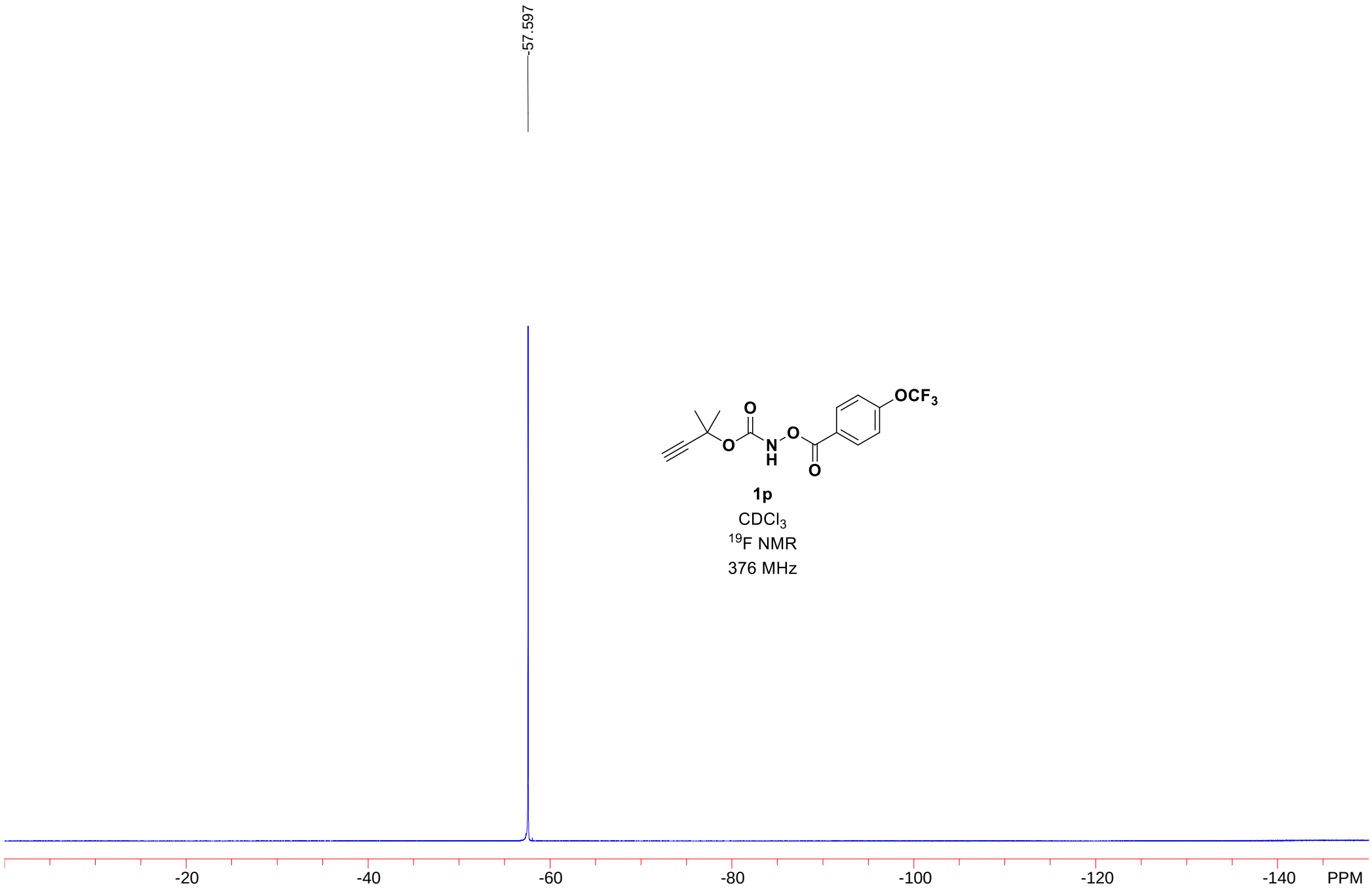
PPM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O
O
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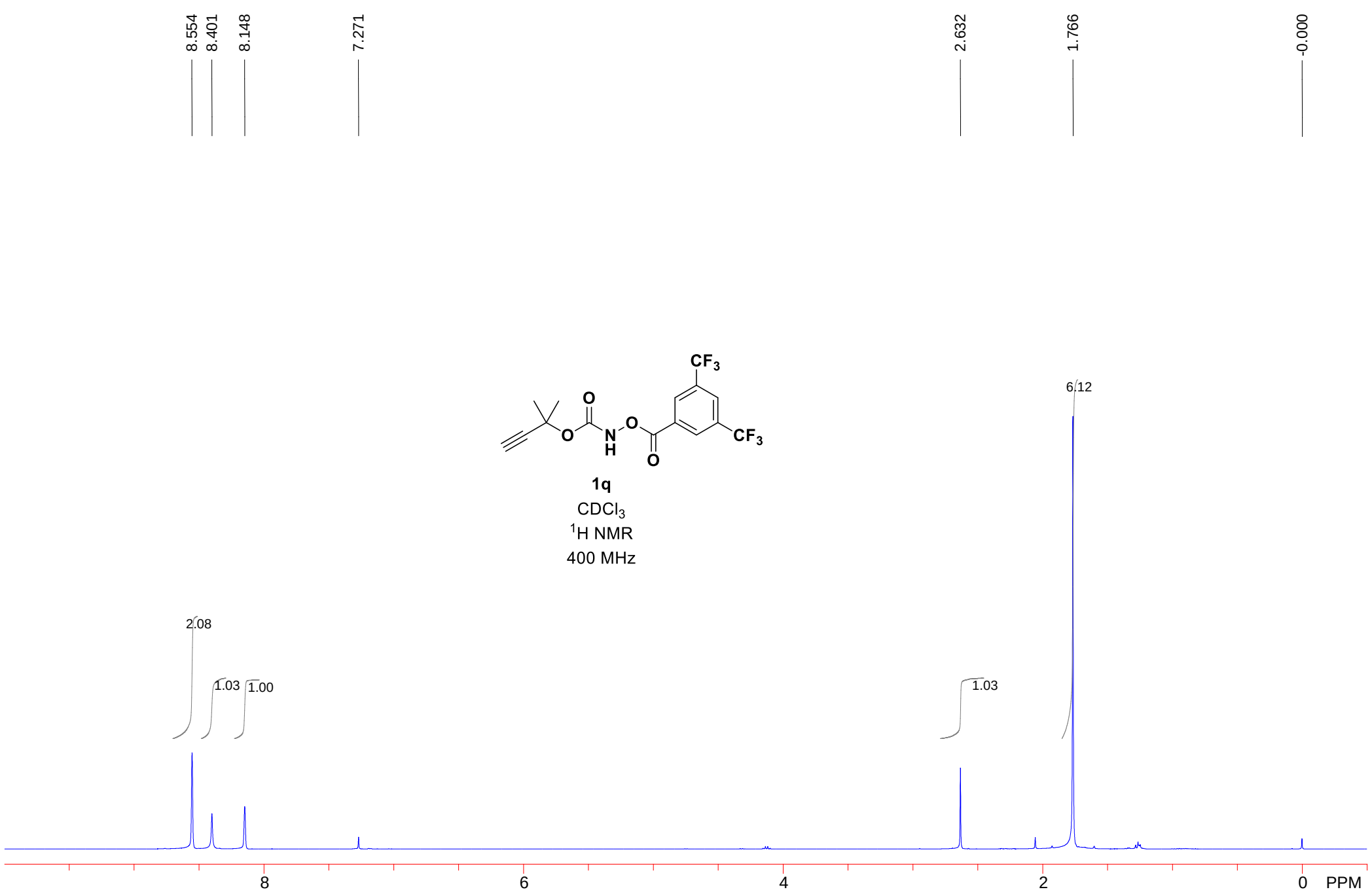
1p
CDCl3
19F NMR
376 MHz
-20
-40
-60
-80
-100
-120
-140
PPM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2.632
1.766
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CF3
CF3
O
O
N
H
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O
O
1q
CDCl3
1H NMR
400 MHz
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1.03
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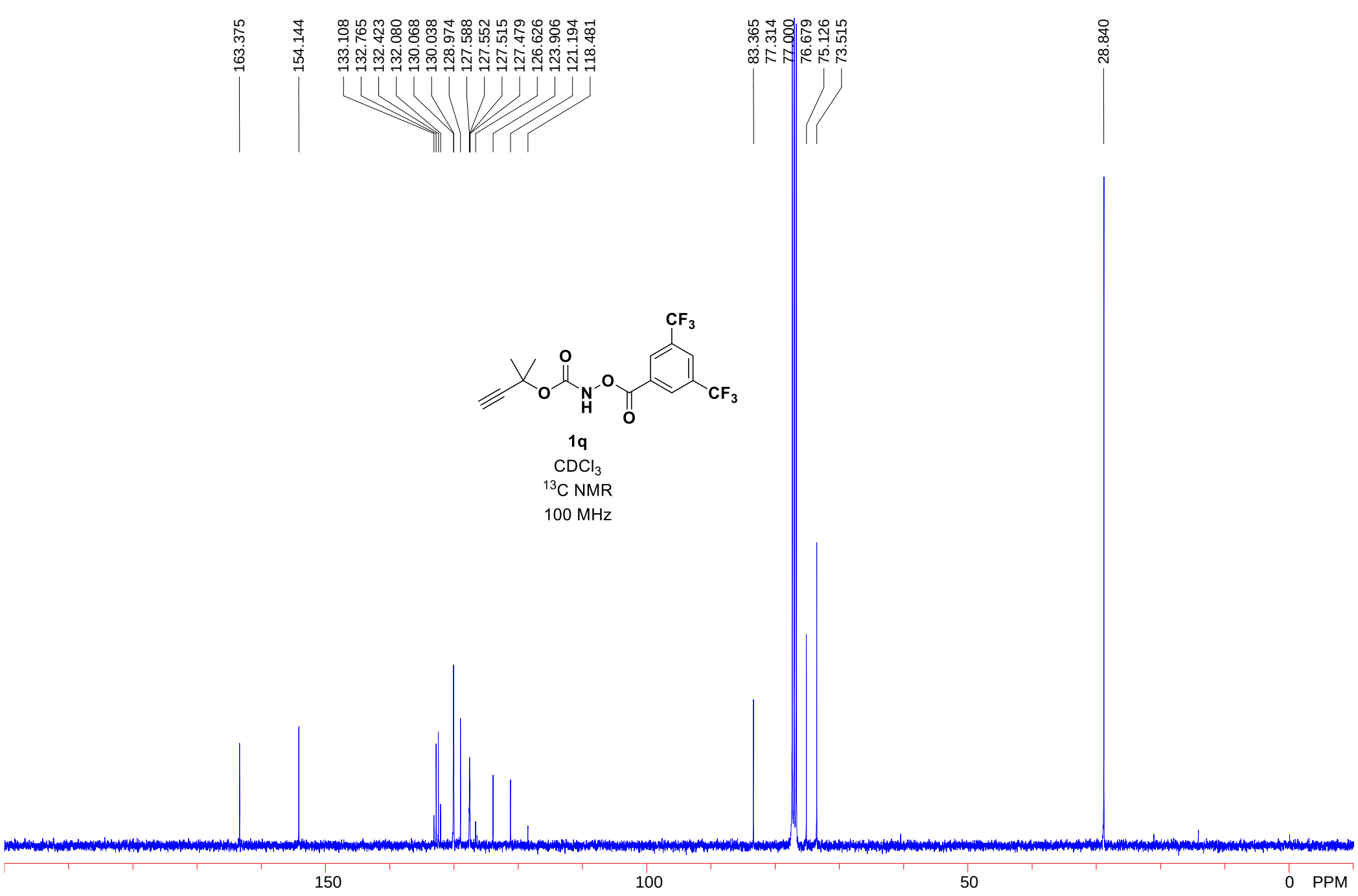
8
6
4
2
0
PPM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130.038
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127.515
127.479
126.626
123.906
121.194
118.481
83.365
77.314
77.000
76.679
75.126
73.515
28.840
CF3
CF3
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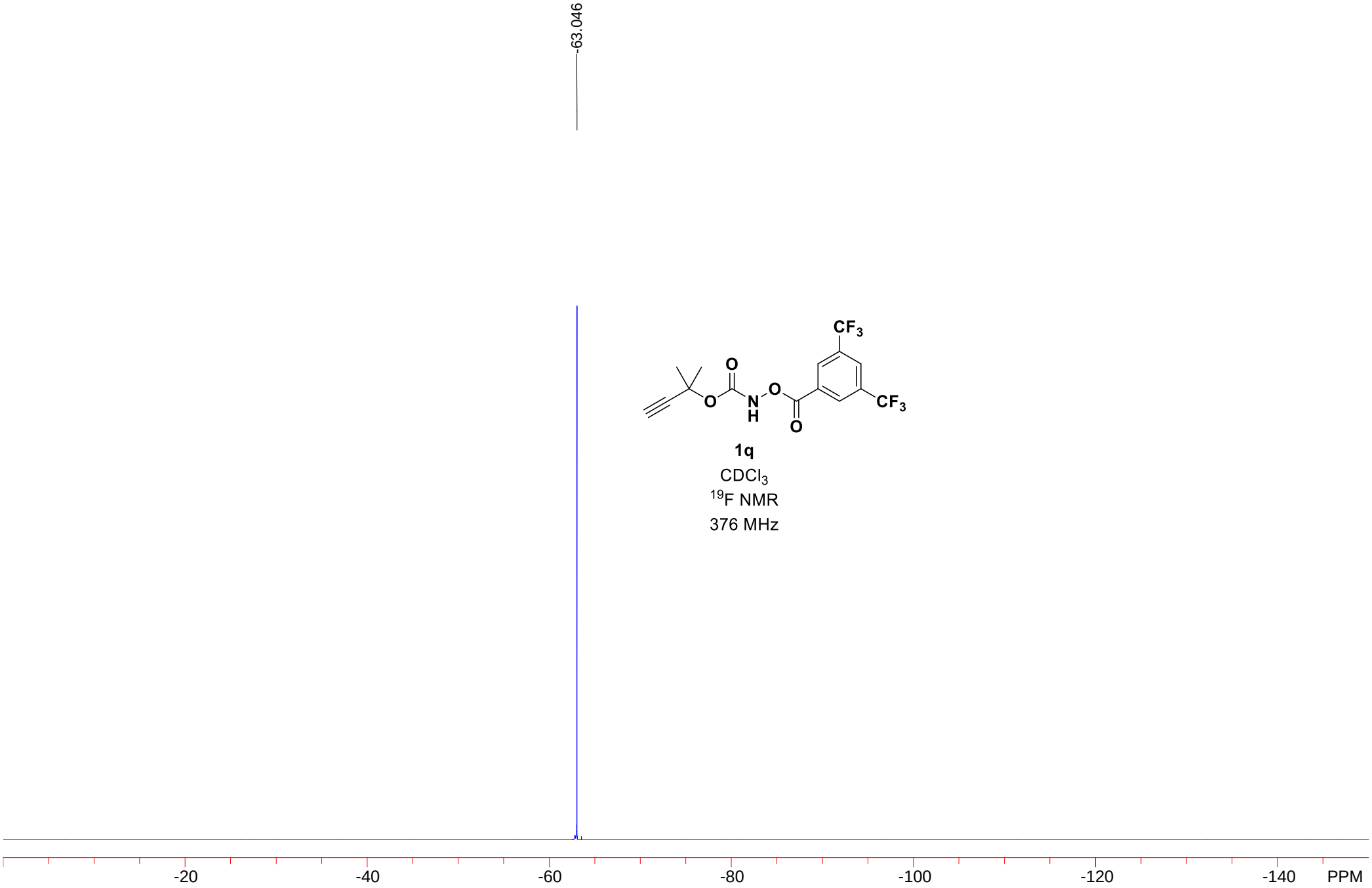
N
H
O
O
1q
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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CF3
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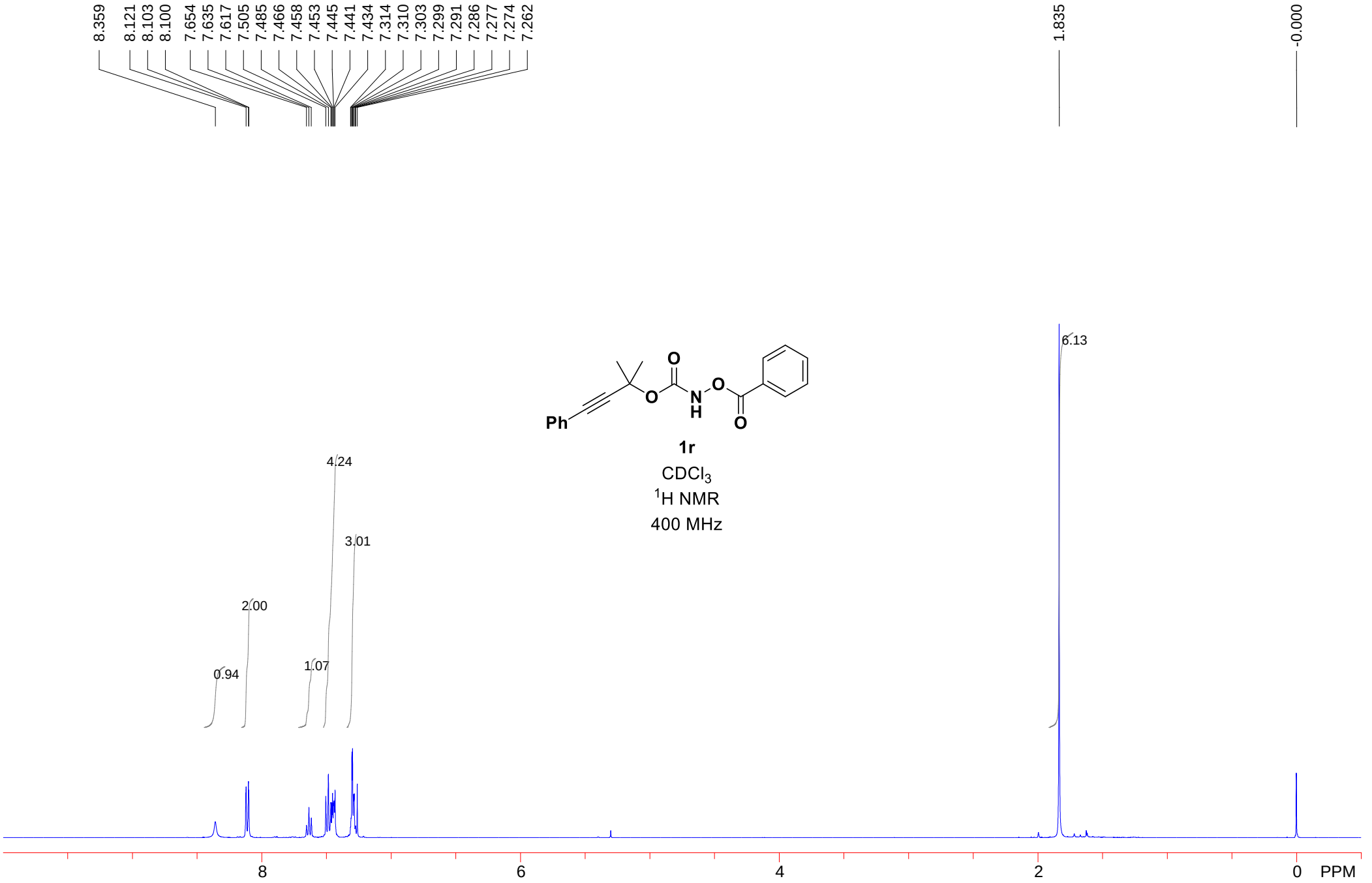
PPM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7.441
7.434
7.314
7.310
7.303
7.299
7.291
7.286
7.277
7.274
7.262
1.835
-0.000
Ph
O
O
N
H
O
O
1r
CDCl3
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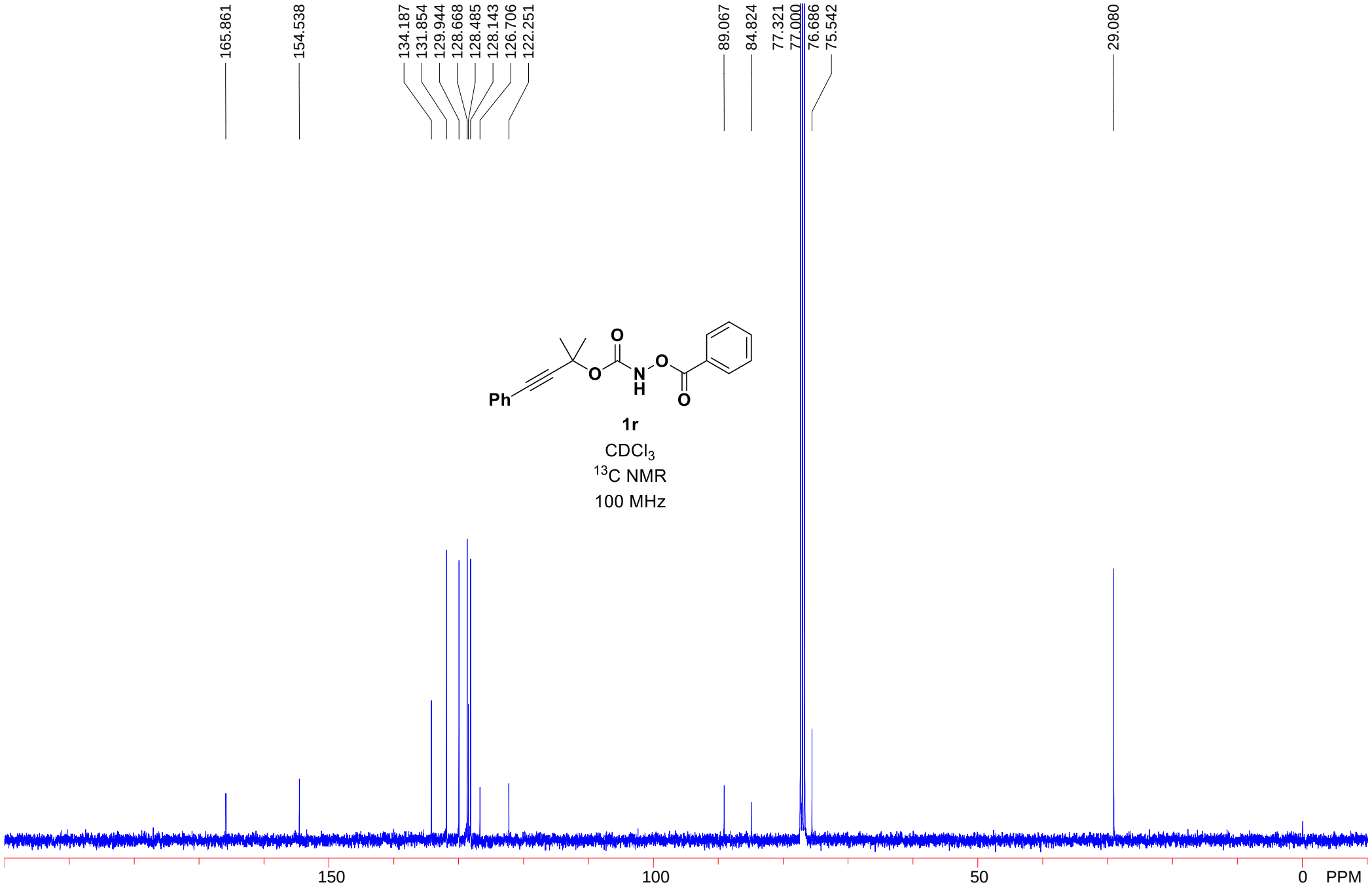
PPM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77.321
77.000
76.686
75.542
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O
O
Ph
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100 MHz
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100
50
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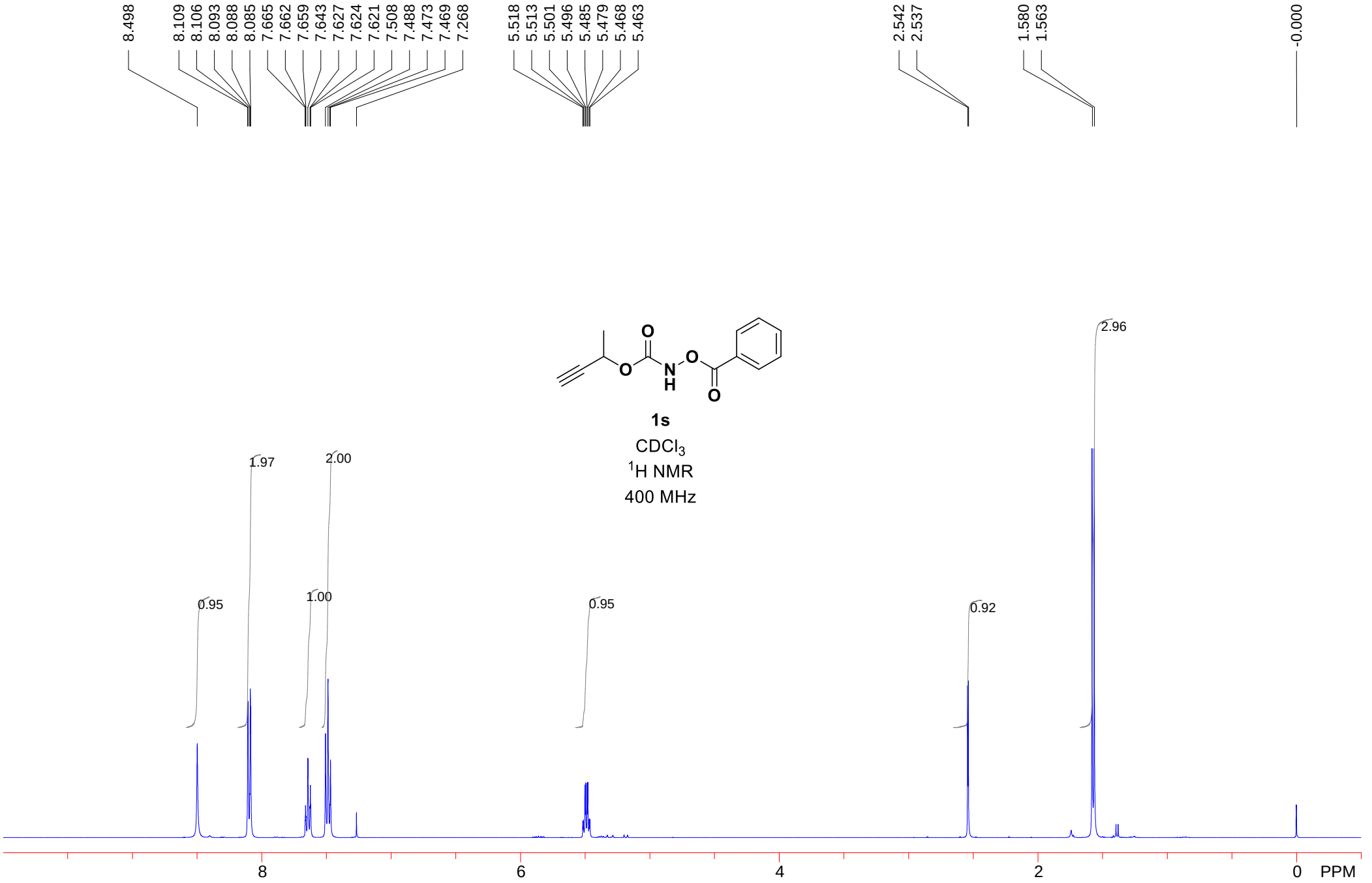
PPM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7.659
7.643
7.627
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7.621
7.508
7.488
7.473
7.469
7.268
5.518
5.513
5.501
5.496
5.485
5.479
5.468
5.463
2.542
2.537
1.580
1.563
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1s
CDCl3
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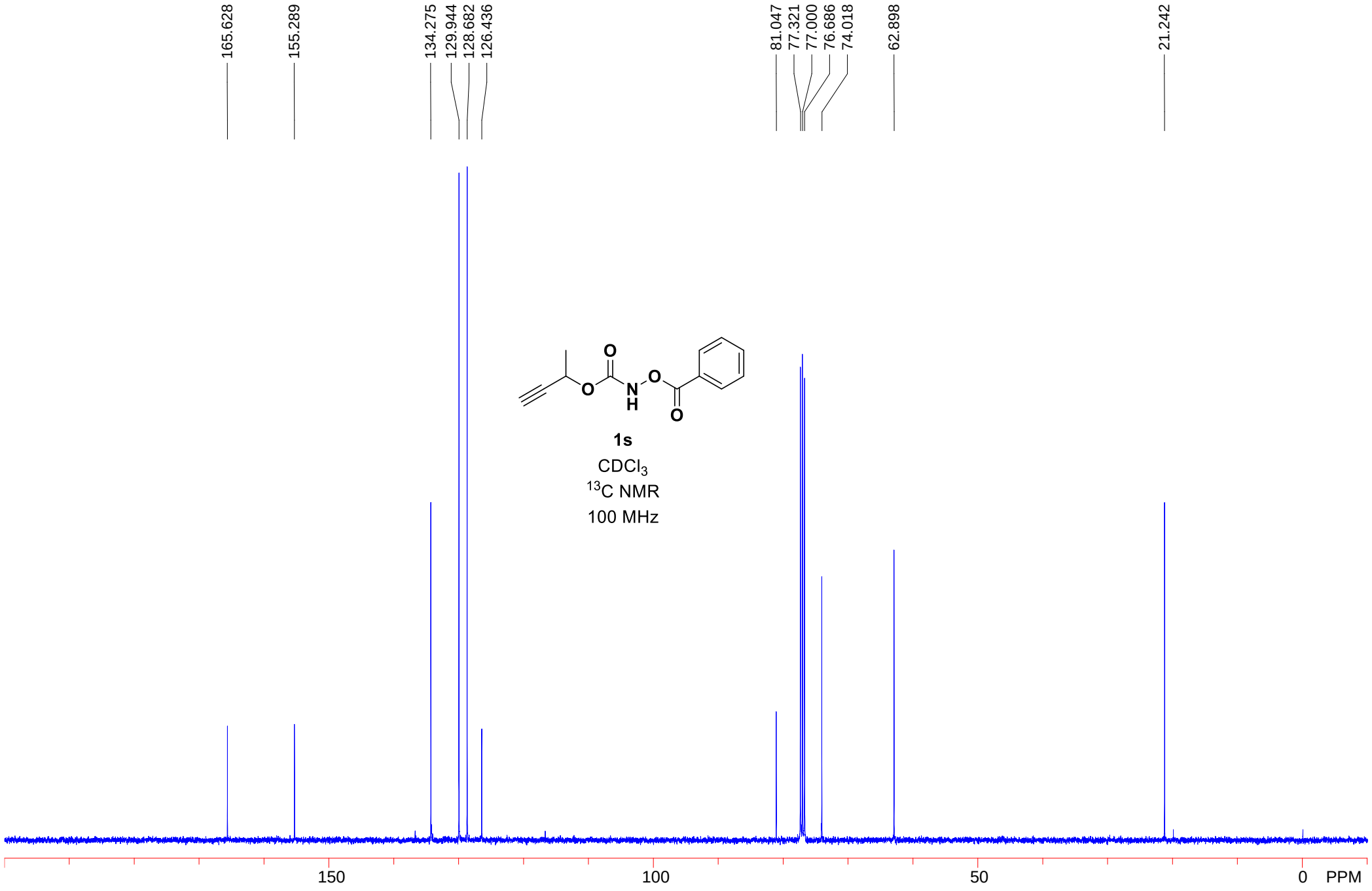
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74.018
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H
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CDCl3
13C NMR
100 MHz
150
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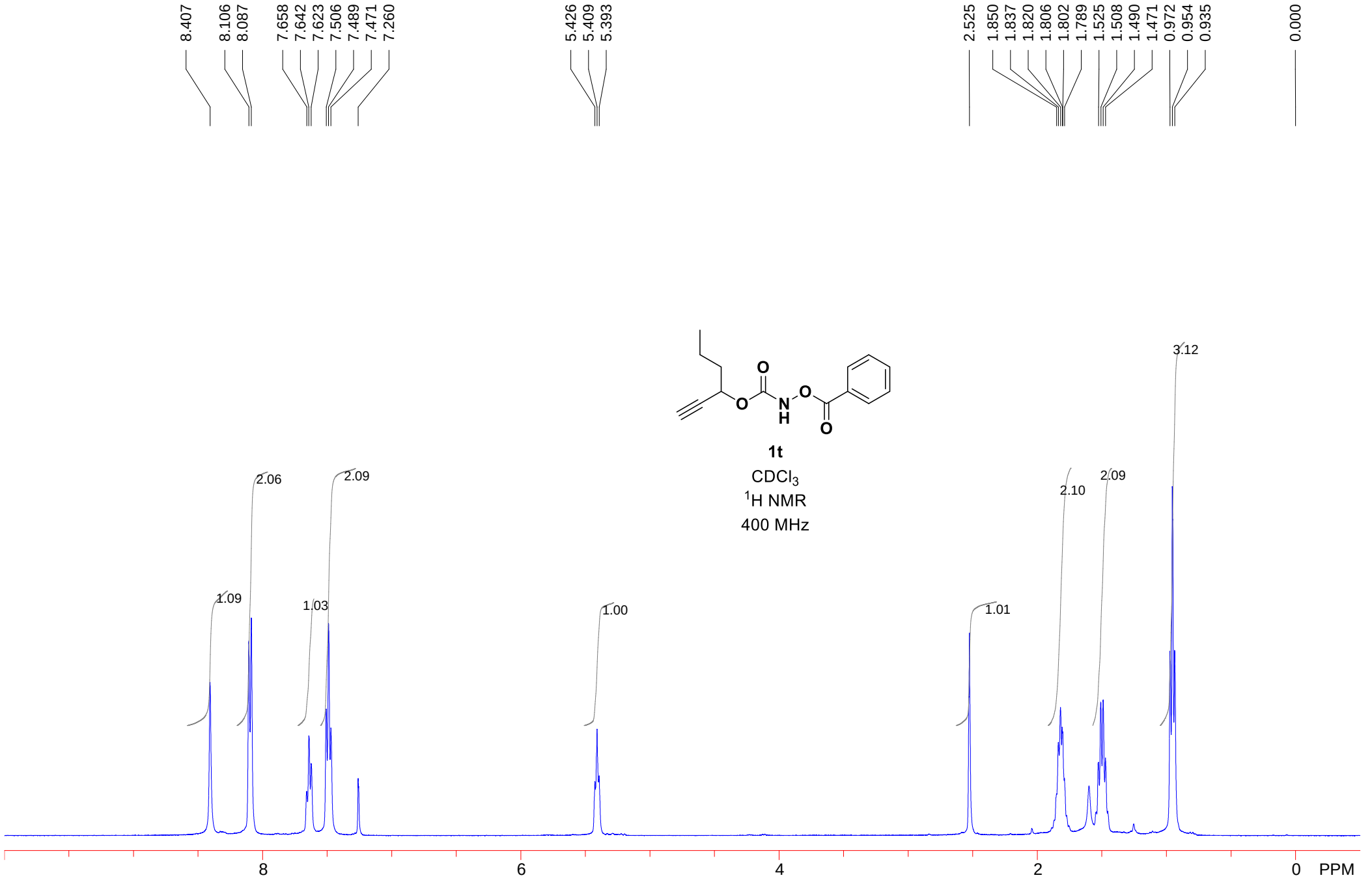
PPM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5.409
5.393
2.525
1.850
1.837
1.820
1.806
1.802
1.789
1.525
1.508
1.490
1.471
0.972
0.954
0.935
0.000
1t
CDCl3
1H NMR
400 MHz
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3.12
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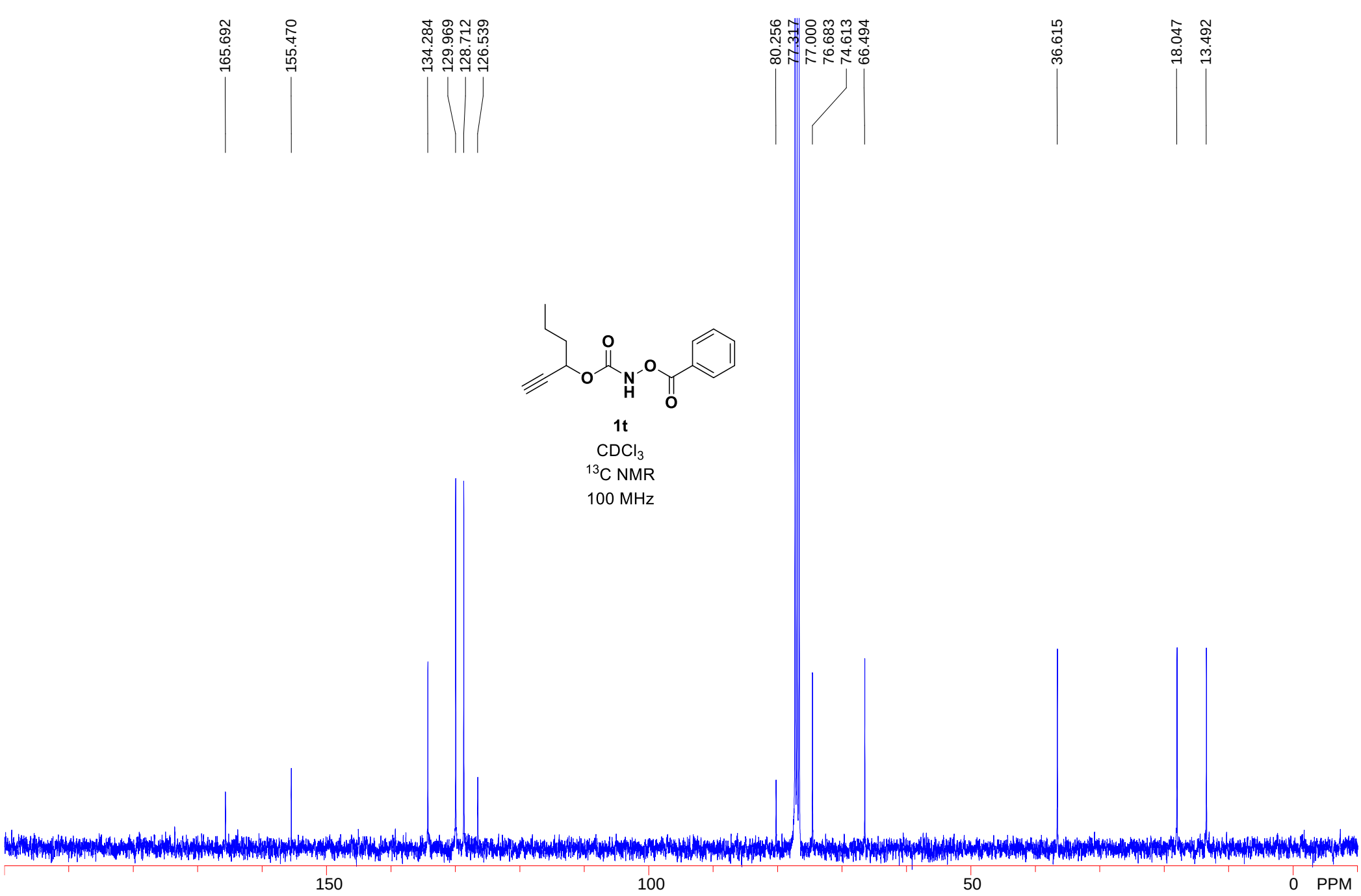
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36.615
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13C NMR
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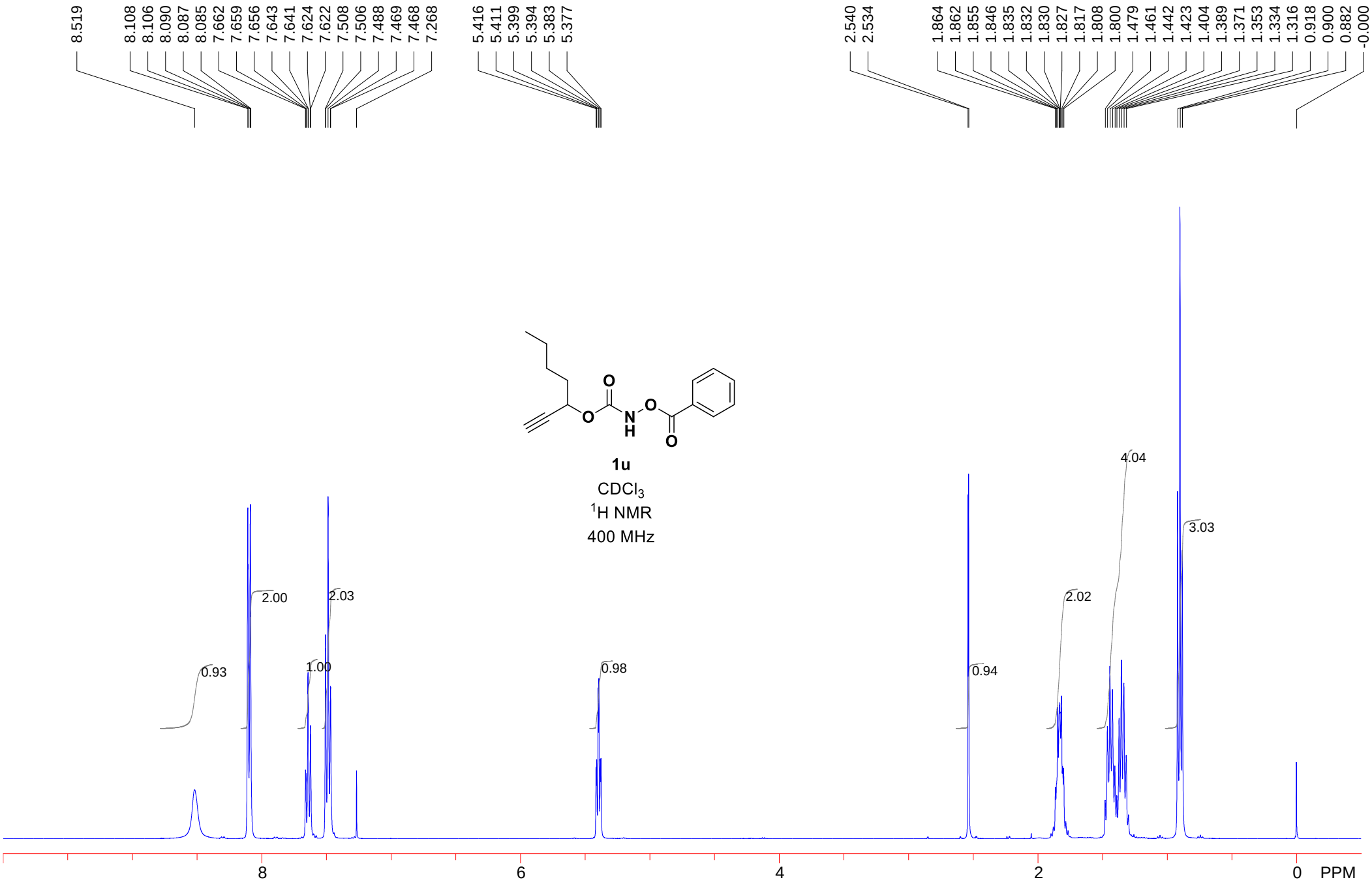
PPM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7.622
7.508
7.506
7.488
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7.468
7.268
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5.383
5.377
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2.534
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1.855
1.846
1.835
1.832
1.830
1.827
1.817
1.808
1.800
1.479
1.461
1.442
1.423
1.404
1.389
1.371
1.353
1.334
1.316
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0.900
0.882
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O
N
H
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CDCl3
1H NMR
400 MHz
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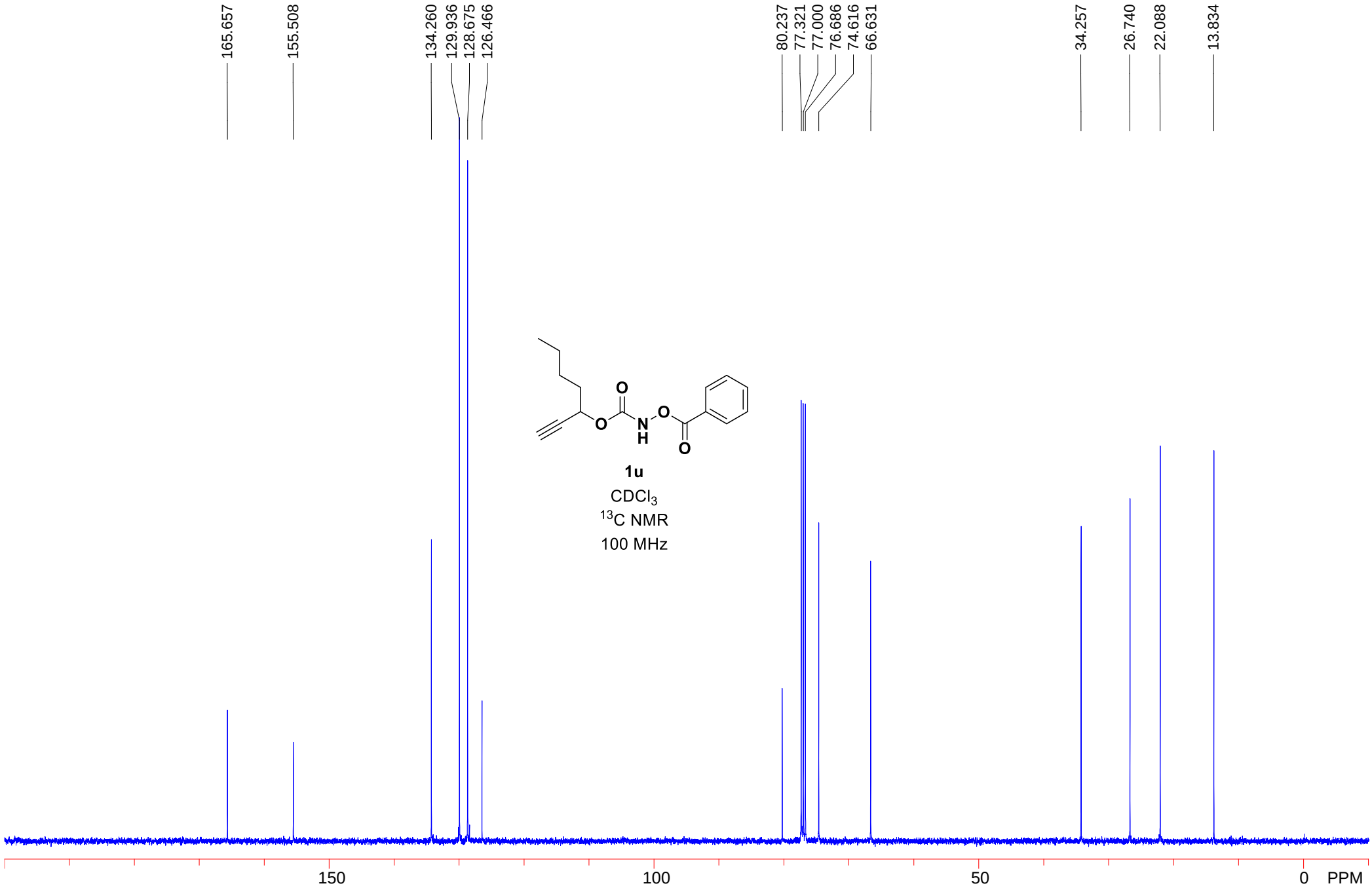
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26.740
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H
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13C NMR
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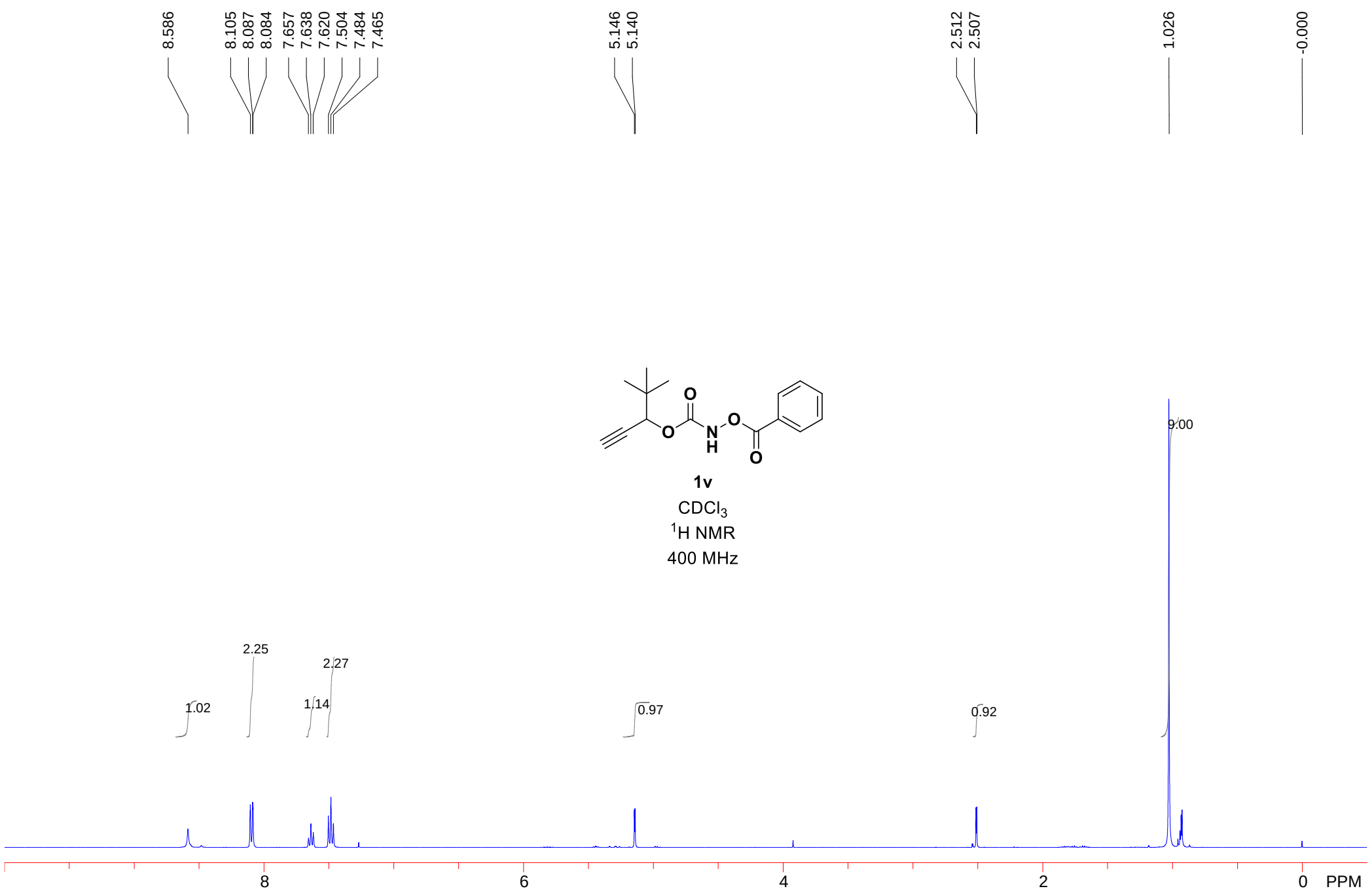
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2.507
1.026
-0.000
1v
CDCl3
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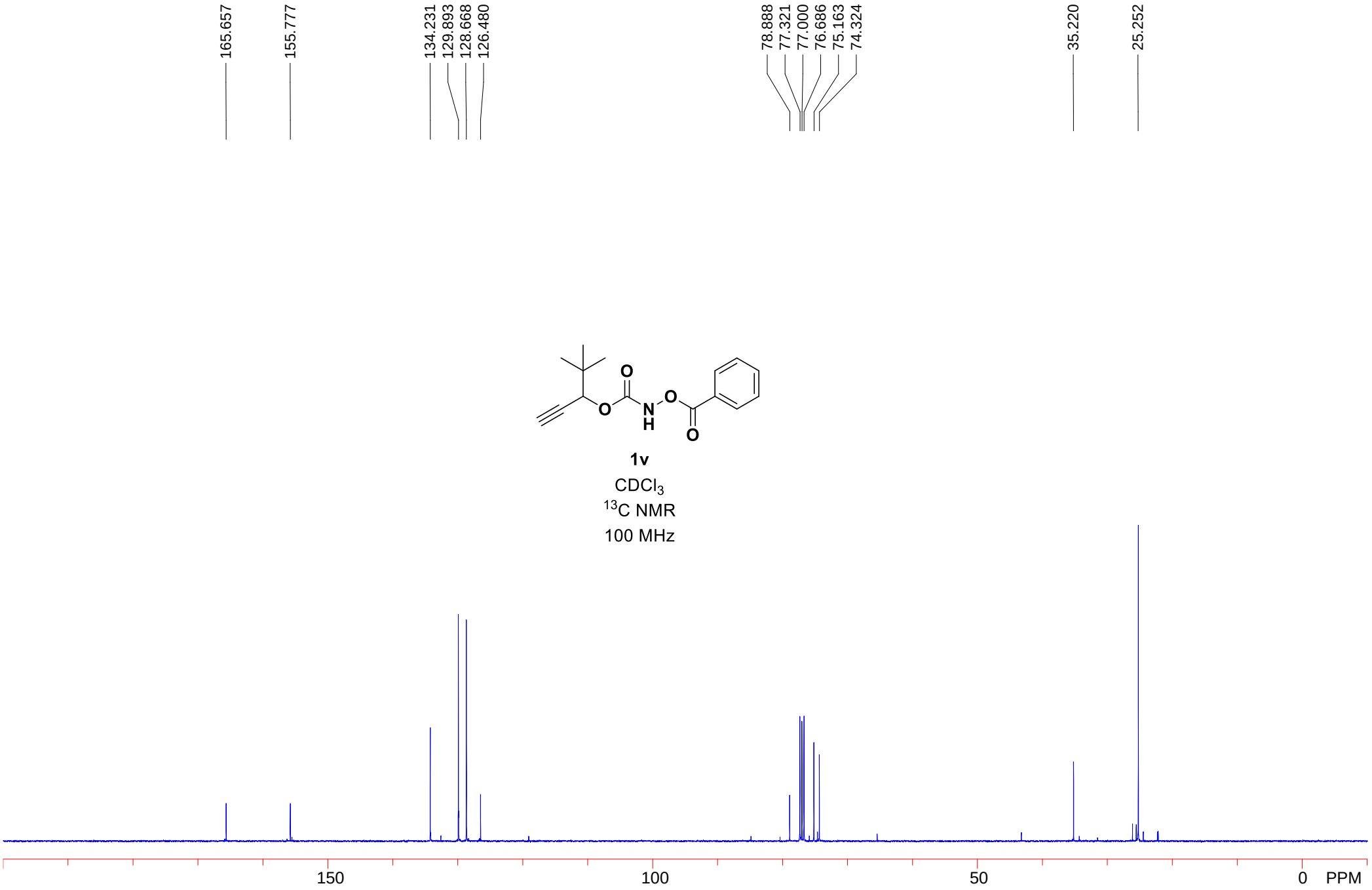
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O
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13C NMR
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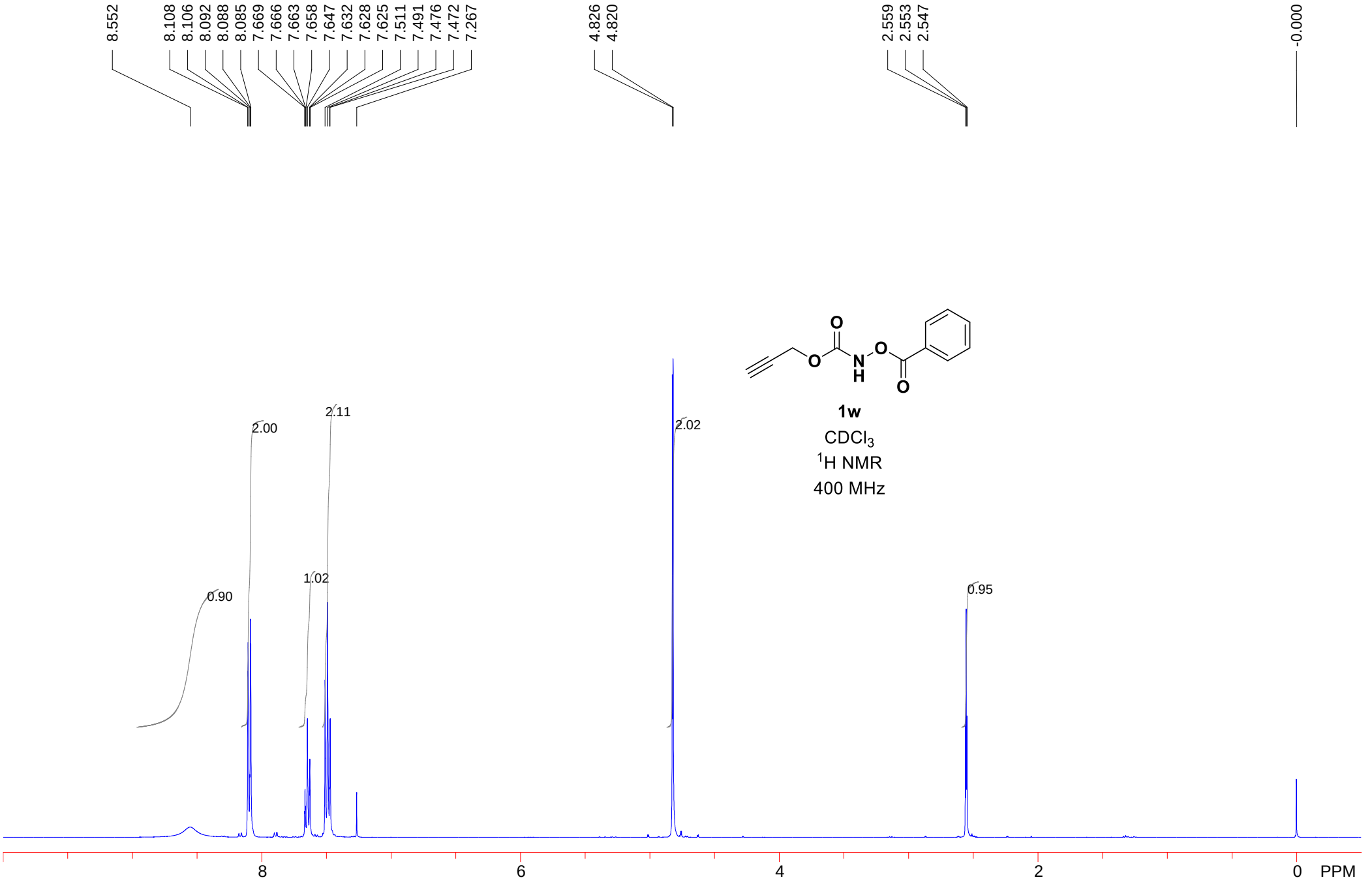
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4.826
4.820
2.559
2.553
2.547
-0.000
1w
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2.00
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2.02
0.95
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6
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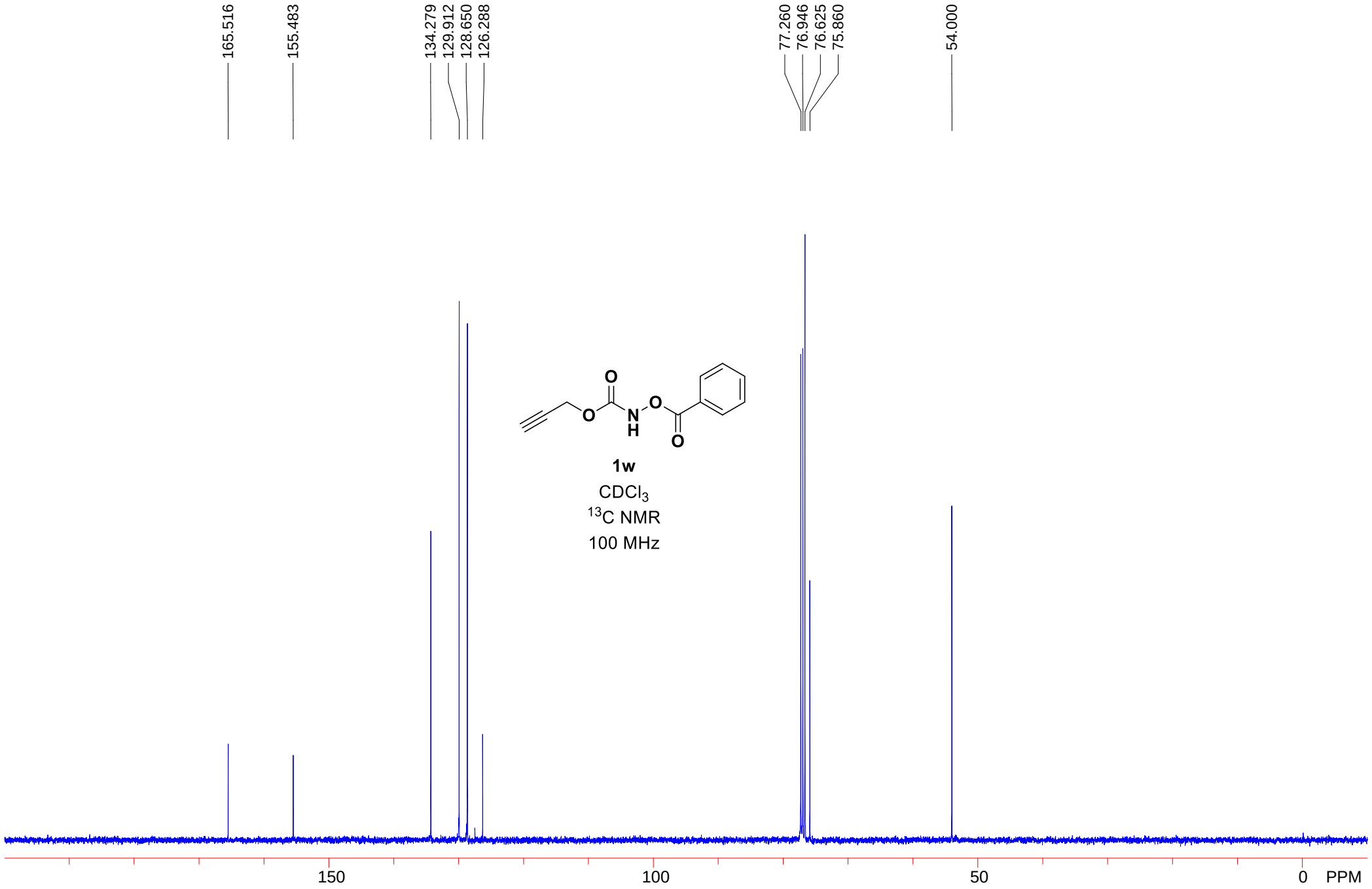
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O
N
H
O
O
1w
CDCl3
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100 MHz
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100
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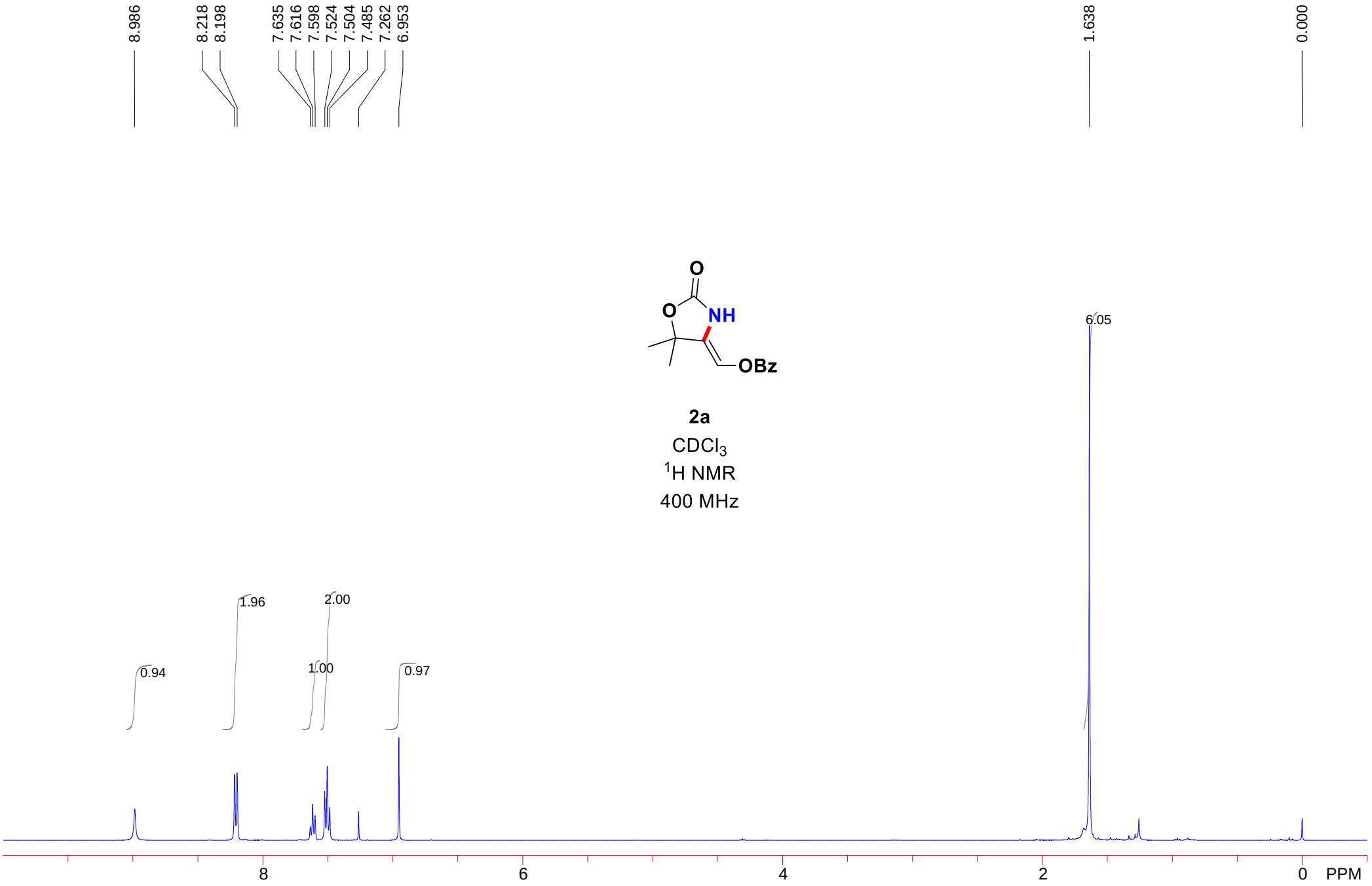
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8.198
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7.504
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7.262
6.953
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0.000
O
O
NH
OBz
2a
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0.97
8
6
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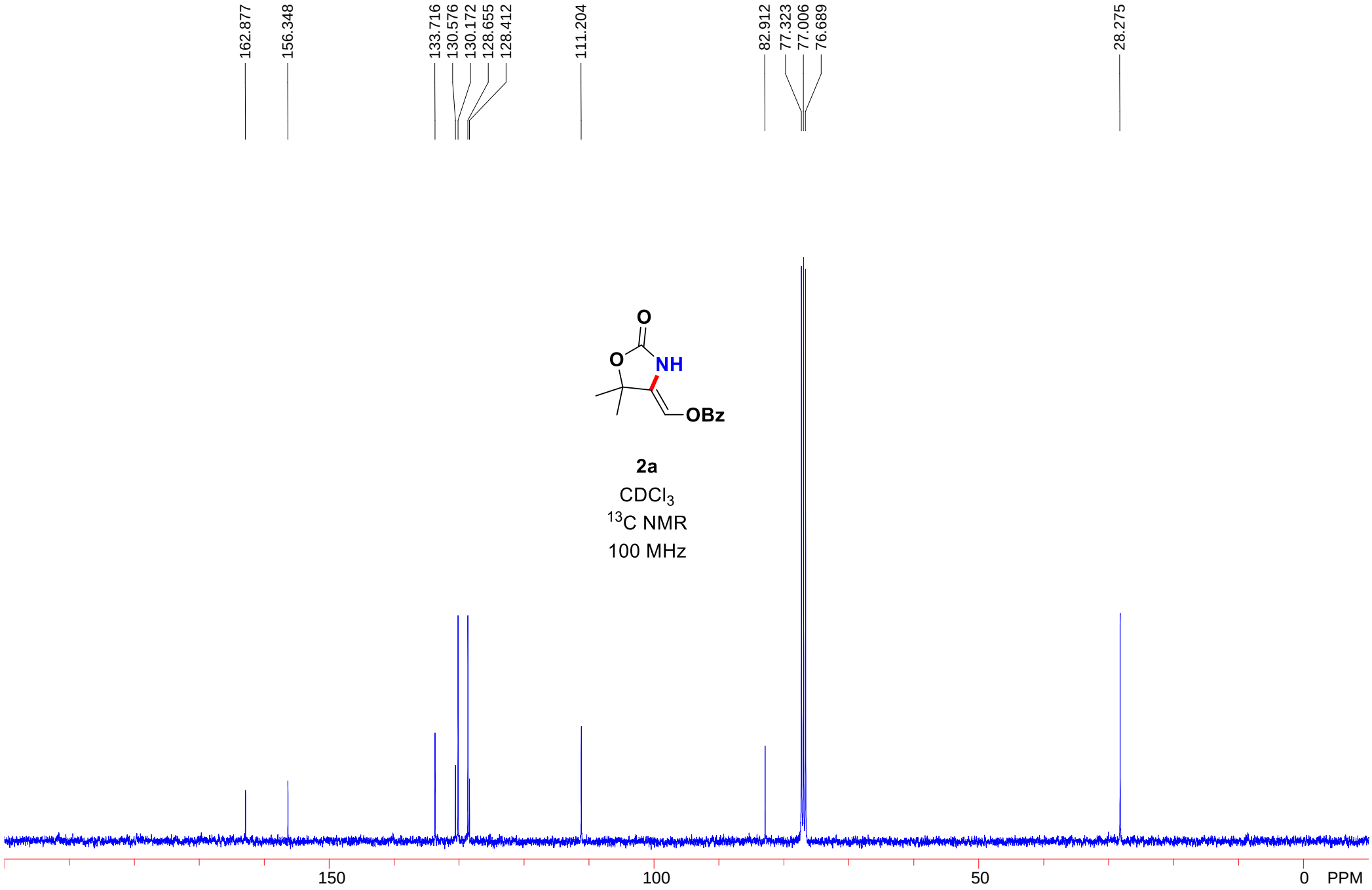
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111.204
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O
O
NH
OBz
2a
CDCl3
13C NMR
100 MHz
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100
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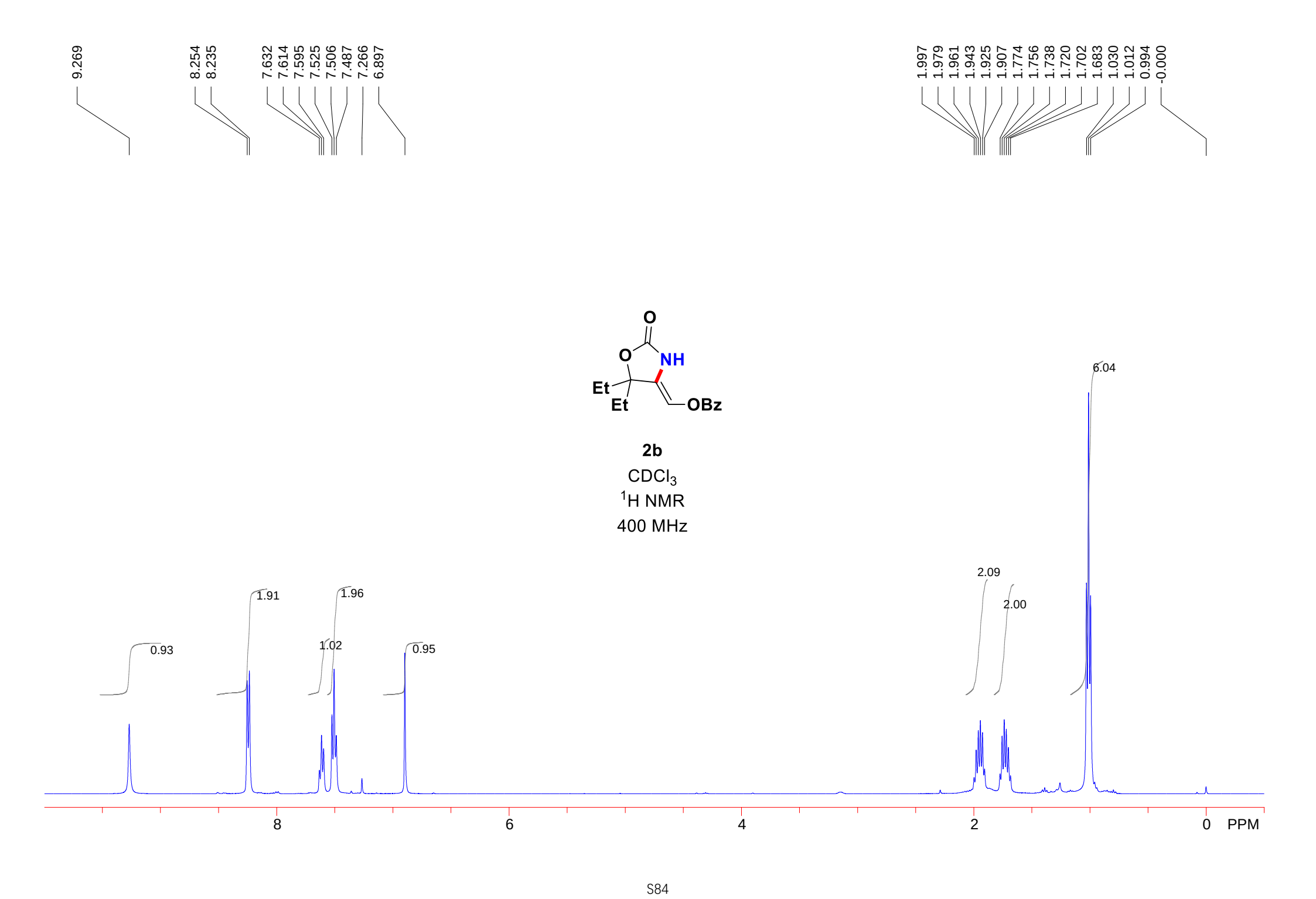
PPM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8.254
8.235
7.632
7.614
7.595
7.525
7.506
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7.266
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1.979
1.961
1.943
1.925
1.907
1.774
1.756
1.738
1.720
1.702
1.683
1.030
1.012
0.994
-0.000
O
O
NH
Et
Et
OBz
2b
CDCl3
1H NMR
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8
6
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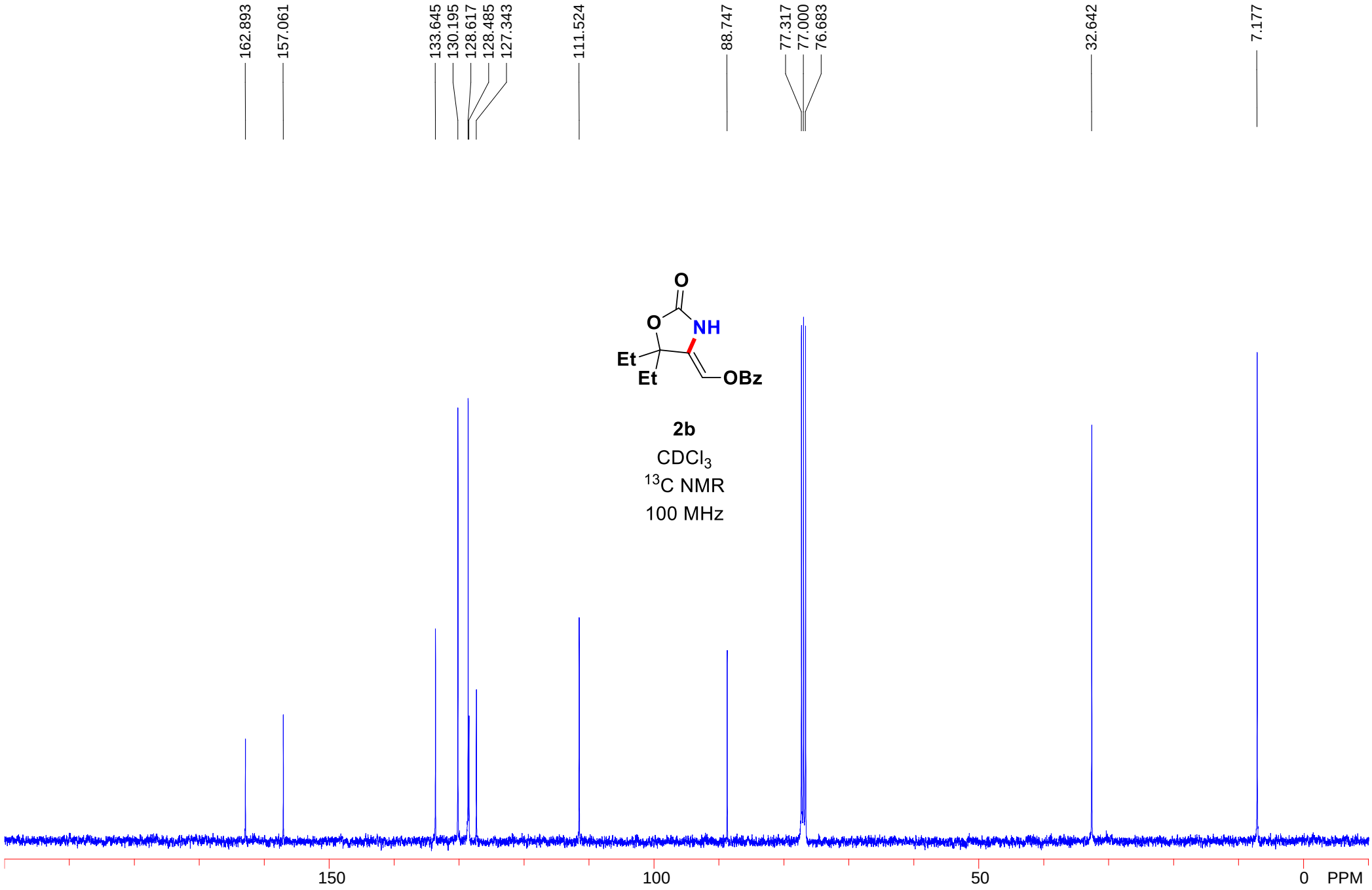
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76.683
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NH
Et
Et
OBz
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13C NMR
100 MHz
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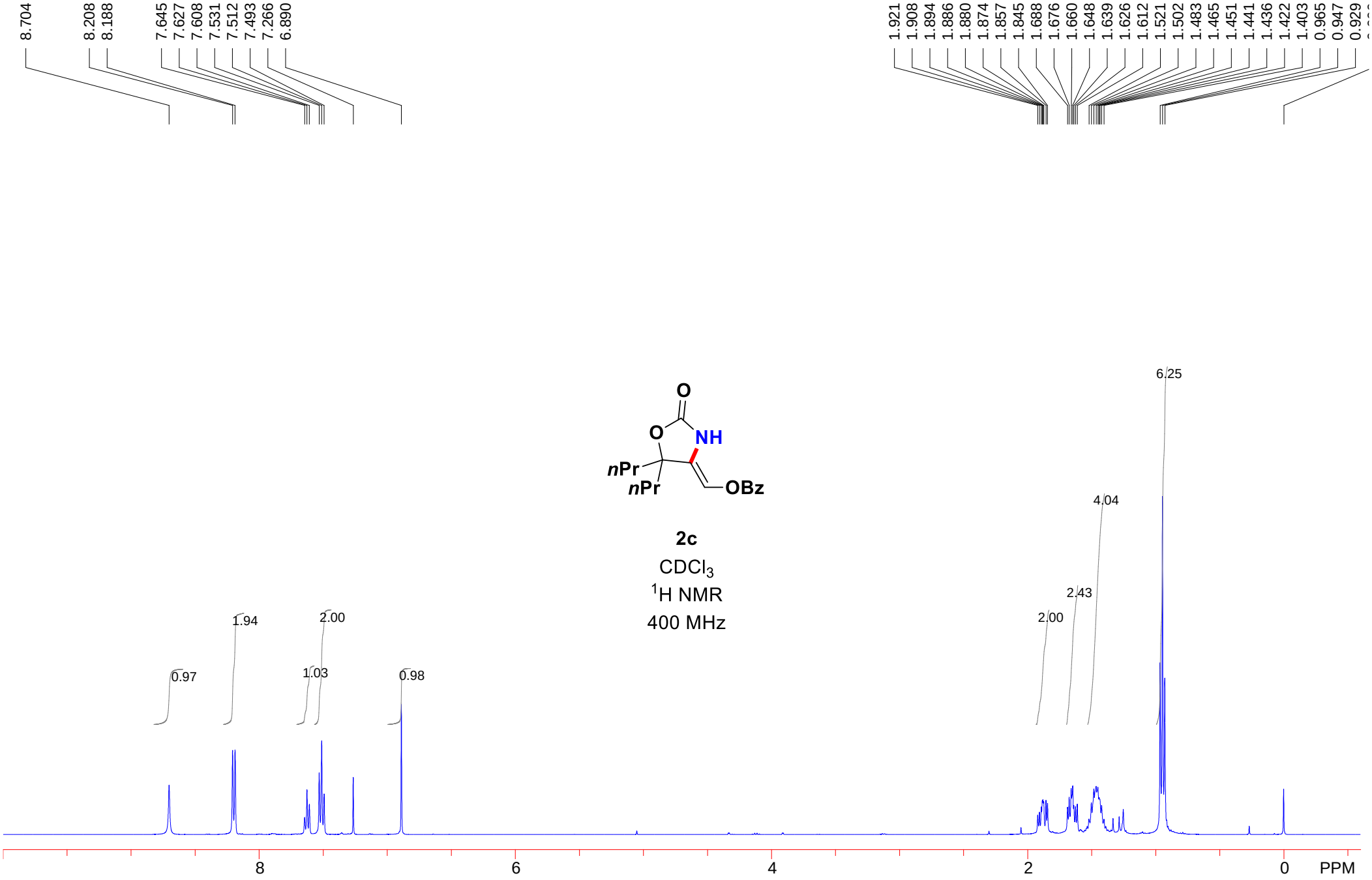
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8.208
8.188
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7.627
7.608
7.531
7.512
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7.266
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1.921
1.908
1.894
1.886
1.880
1.874
1.857
1.845
1.688
1.676
1.660
1.648
1.639
1.626
1.612
1.521
1.502
1.483
1.465
1.451
1.441
1.436
1.422
1.403
0.965
0.947
0.929
O
O
NH
nPr
nPr
OBz
2c
CDCl3
1H NMR
400 MHz
6.25
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0.97
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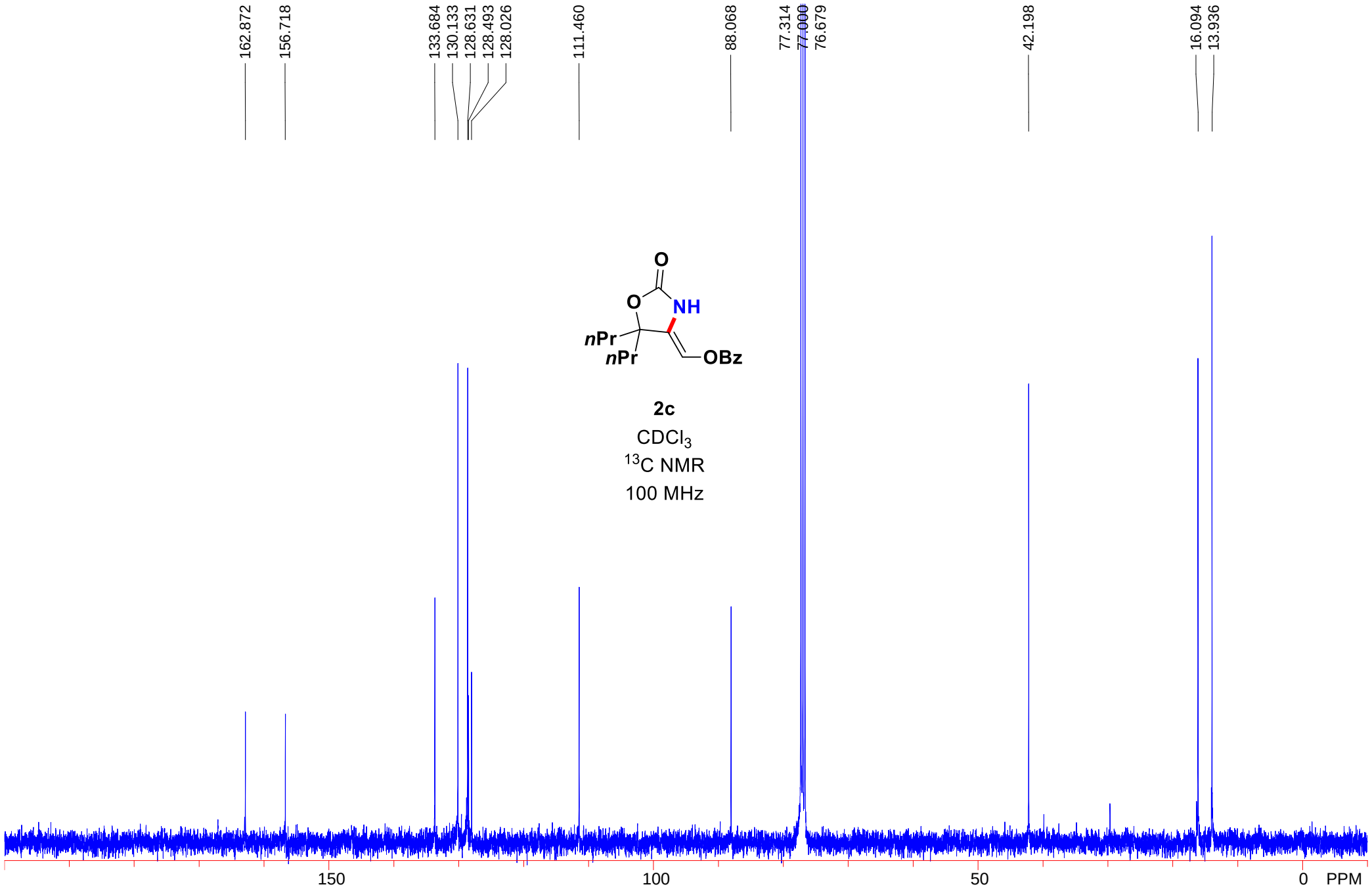
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76.679
42.198
16.094
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nPr
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13C NMR
100 MHz
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100
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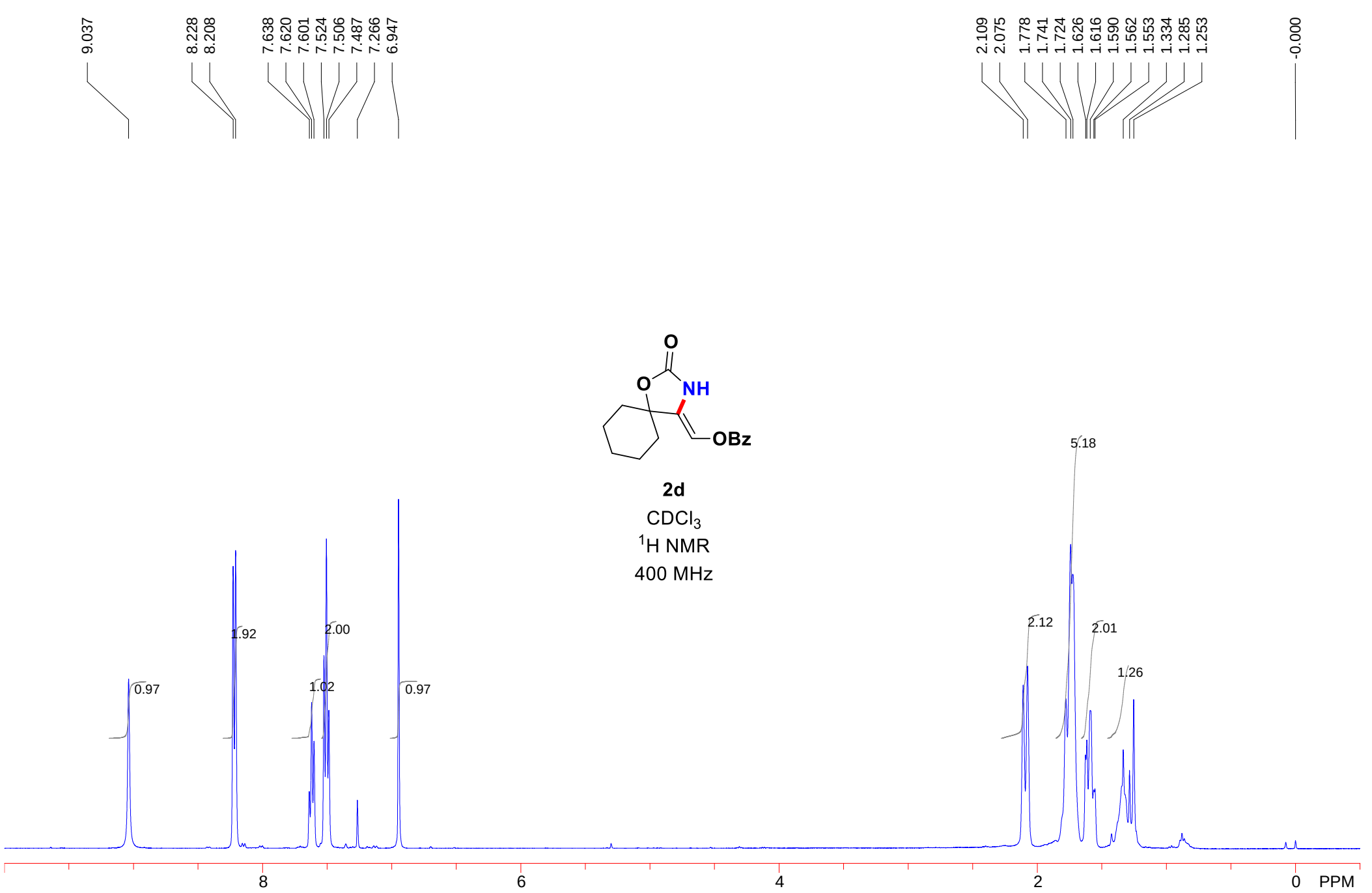
PPM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8.228
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7.638
7.620
7.601
7.524
7.506
7.487
7.266
6.947
2.109
2.075
1.778
1.741
1.724
1.626
1.616
1.590
1.562
1.553
1.334
1.285
1.253
-0.000
O
O
NH
OBz
2d
CDCl3
1H NMR
400 MHz
0.97
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0.97
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5.18
2.01
1.26
8
6
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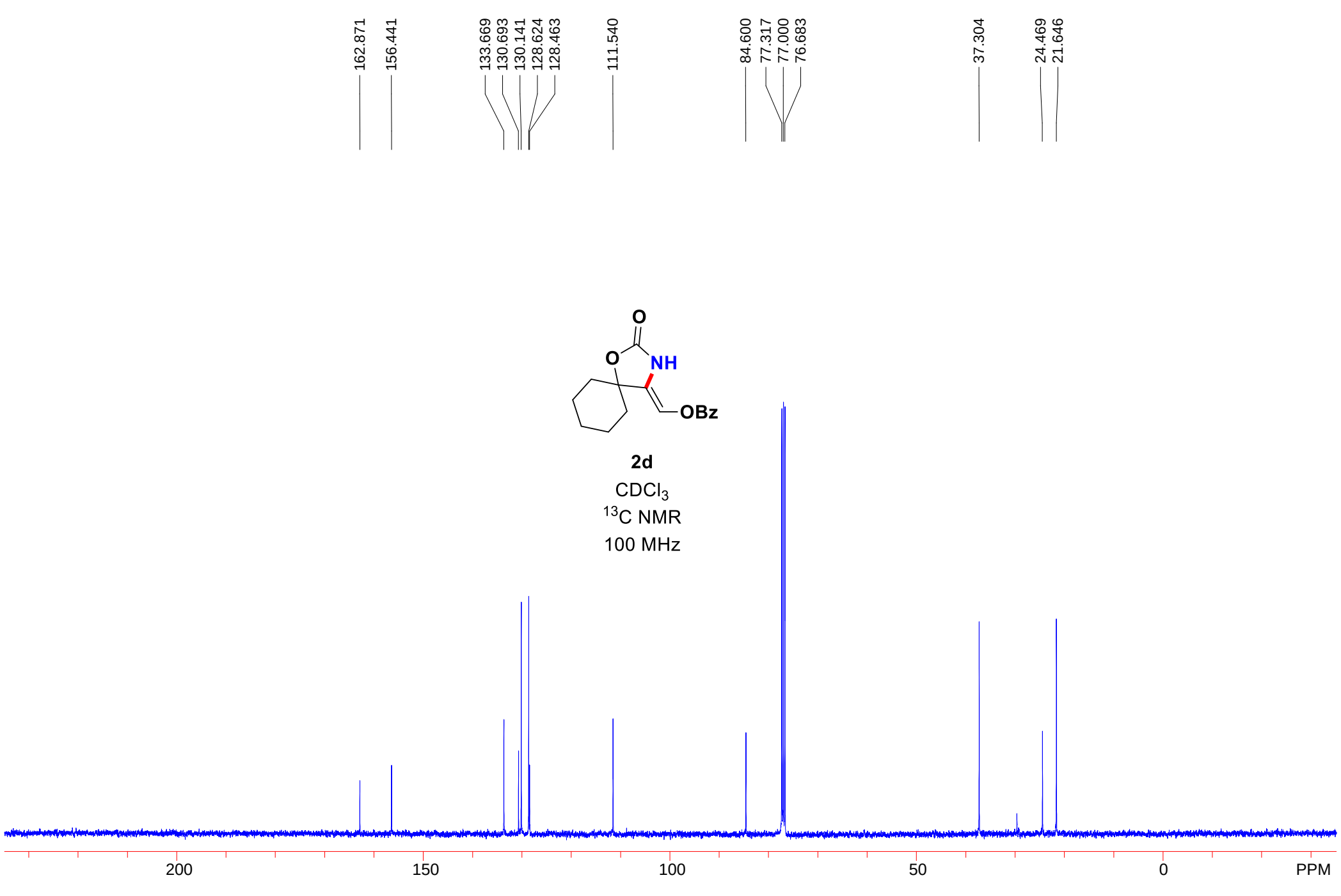
PPM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128.463
111.540
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77.000
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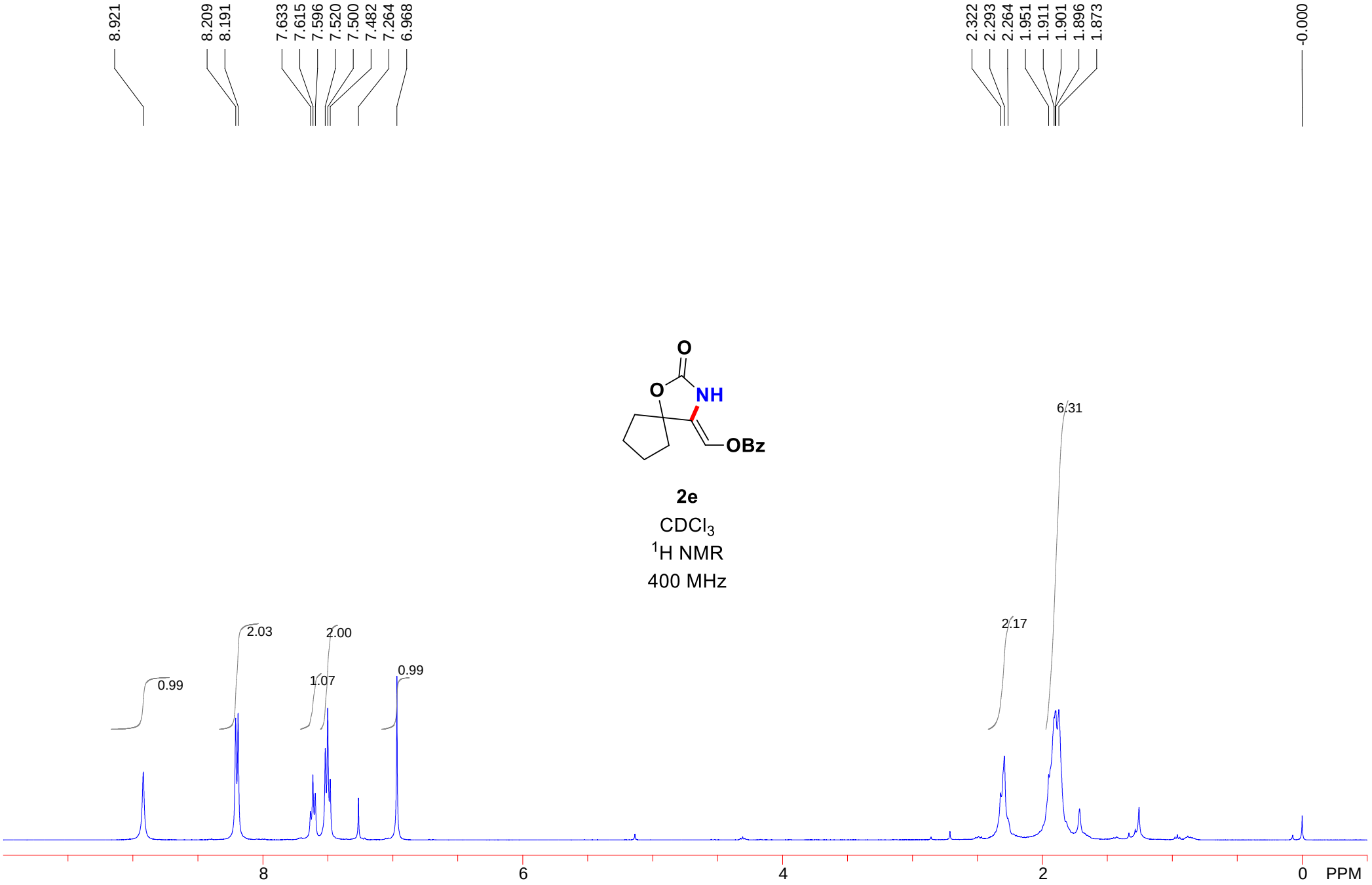
37.304
24.469
21.646
O
O
NH
OBz
2d
CDCl3
13C NMR
100 MHz
200
150
100
50
0
PPM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8.191
7.633
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7.596
7.520
7.500
7.482
7.264
6.968
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2.264
1.951
1.911
1.901
1.896
1.873
-0.000
O
O
NH
OBz
2e
CDCl3
1H NMR
400 MHz
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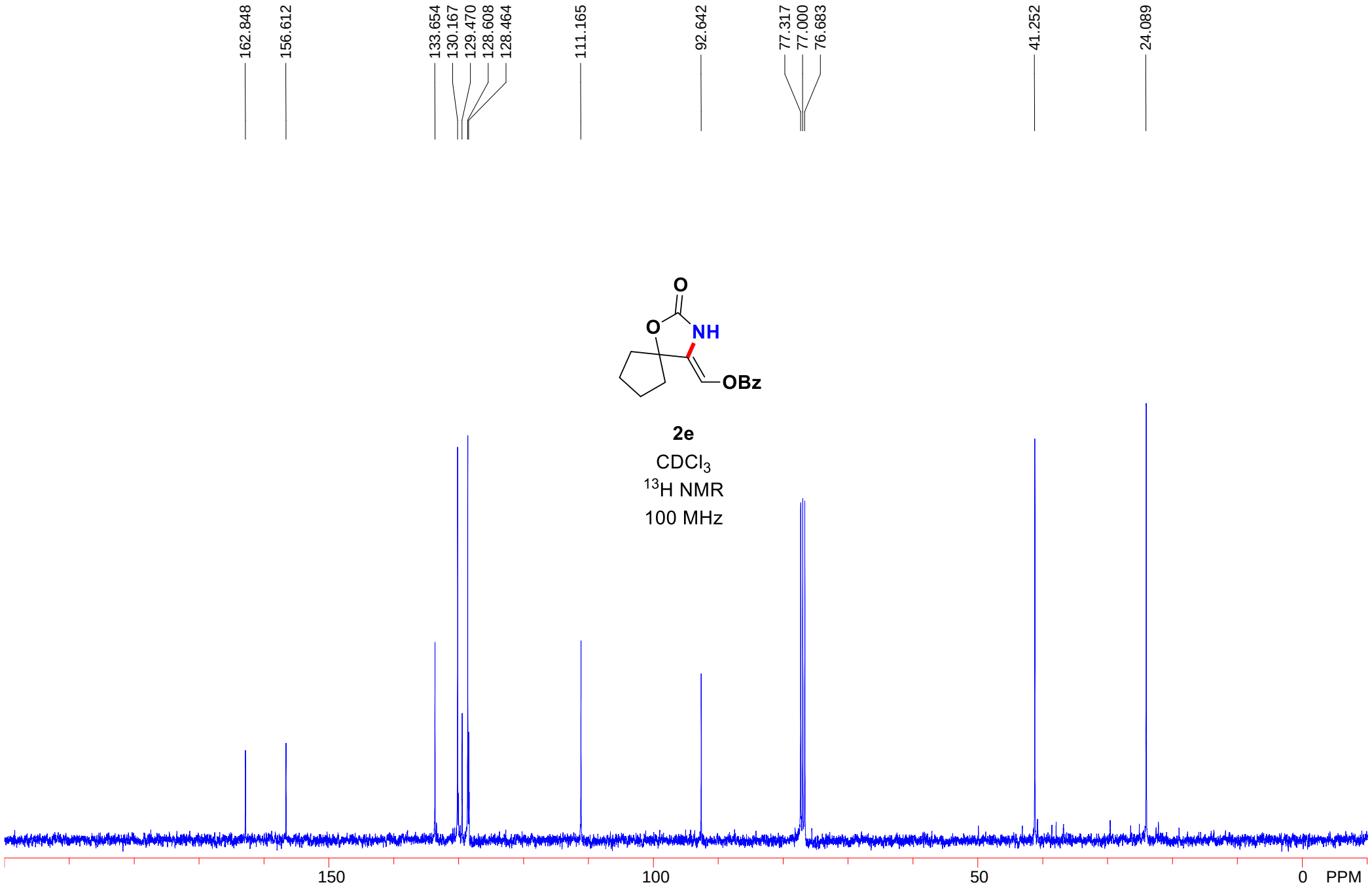
2.03
2.00
1.07
0.99
2.17
6.31
8
6
4
2
0
PPM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133.654
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128.608
128.464
111.165
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77.317
77.000
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O
O
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OBz
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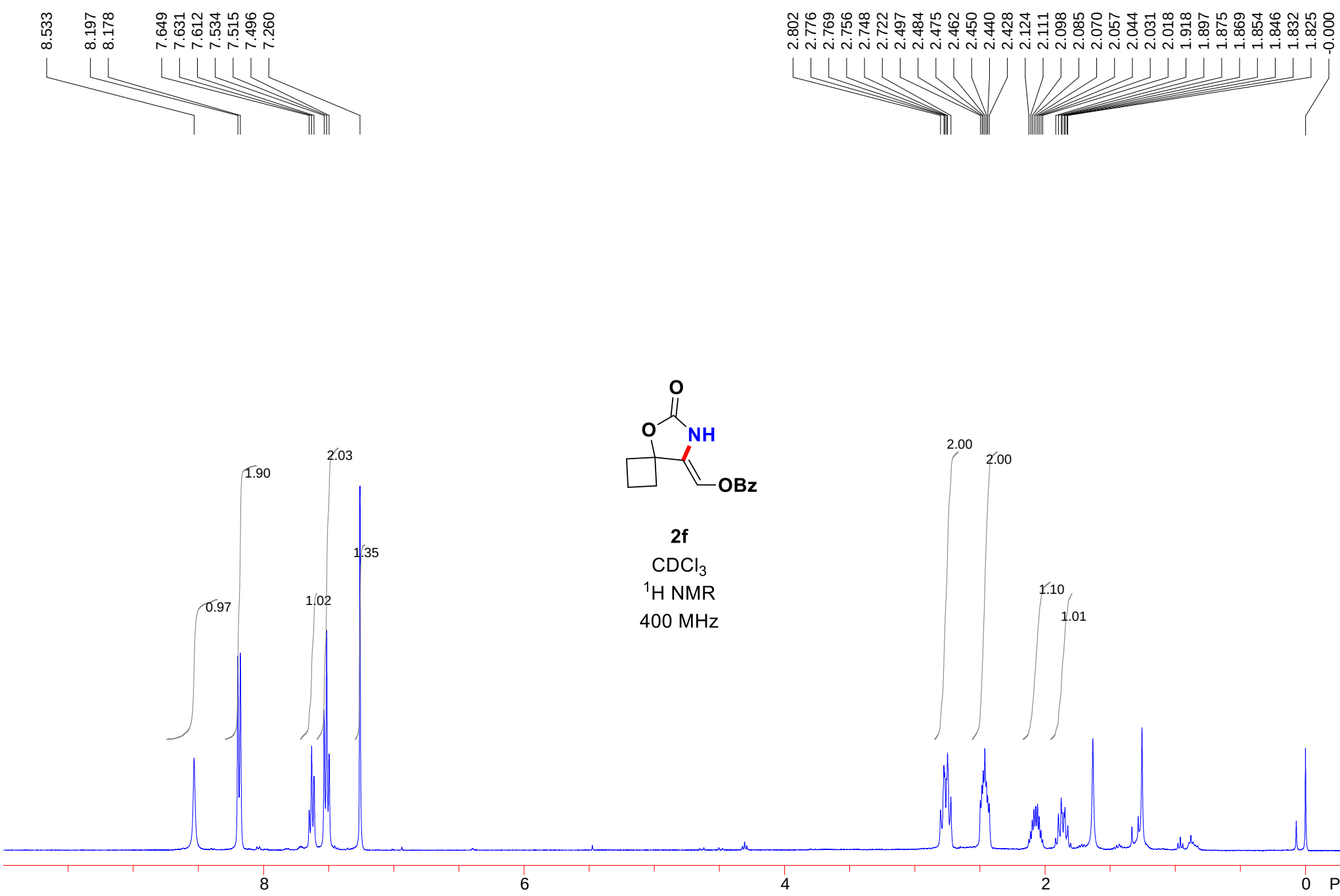
CDCl3
13H NMR
100 MHz
150
100
50
0
PPM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2.769
2.756
2.748
2.722
2.497
2.484
2.475
2.462
2.450
2.440
2.428
2.124
2.111
2.098
2.085
2.070
2.057
2.044
2.031
2.018
1.918
1.897
1.875
1.869
1.854
1.846
1.832
1.825
-0.000
O
O
NH
OBz
2f
CDCl3
1H NMR
400 MHz
0.97
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1.01
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6
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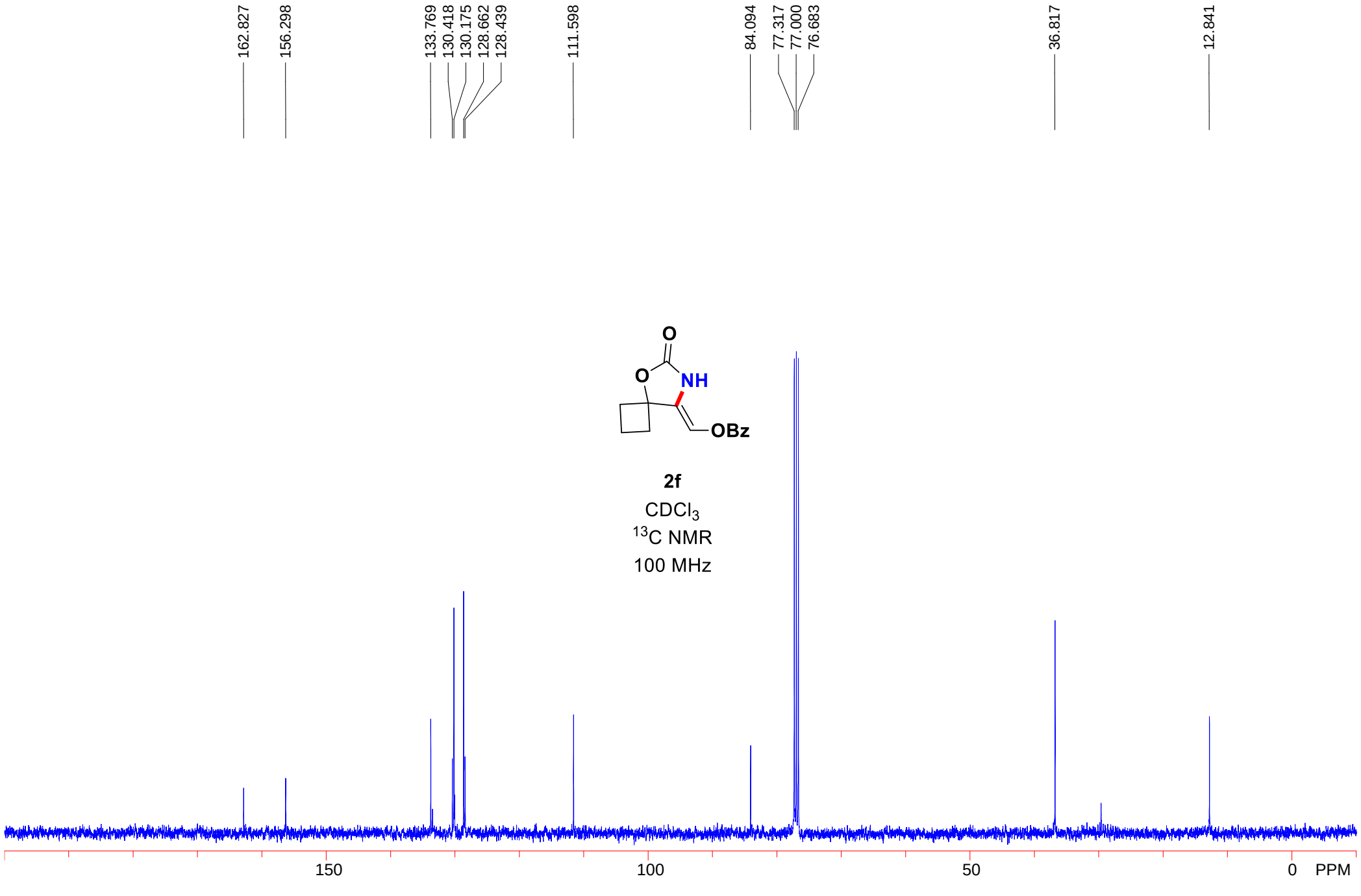
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77.000
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O
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100
50
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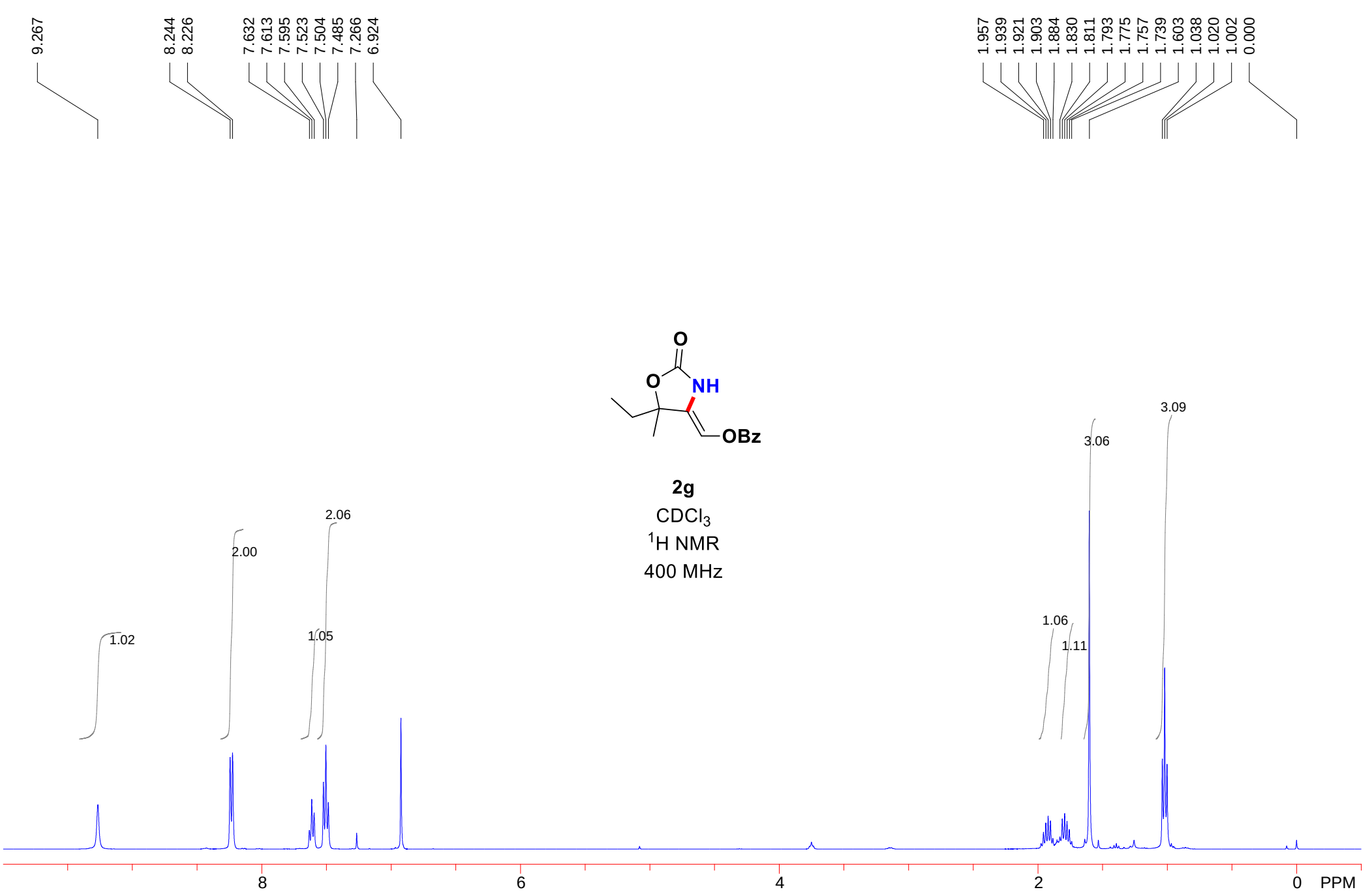
PPM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7.613
7.595
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7.504
7.485
7.266
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1.921
1.903
1.884
1.830
1.811
1.793
1.775
1.757
1.739
1.603
1.038
1.020
1.002
0.000
O
O
NH
OBz
2g
CDCl3
1H NMR
400 MHz
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1.05
1.06
1.11
3.06
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6
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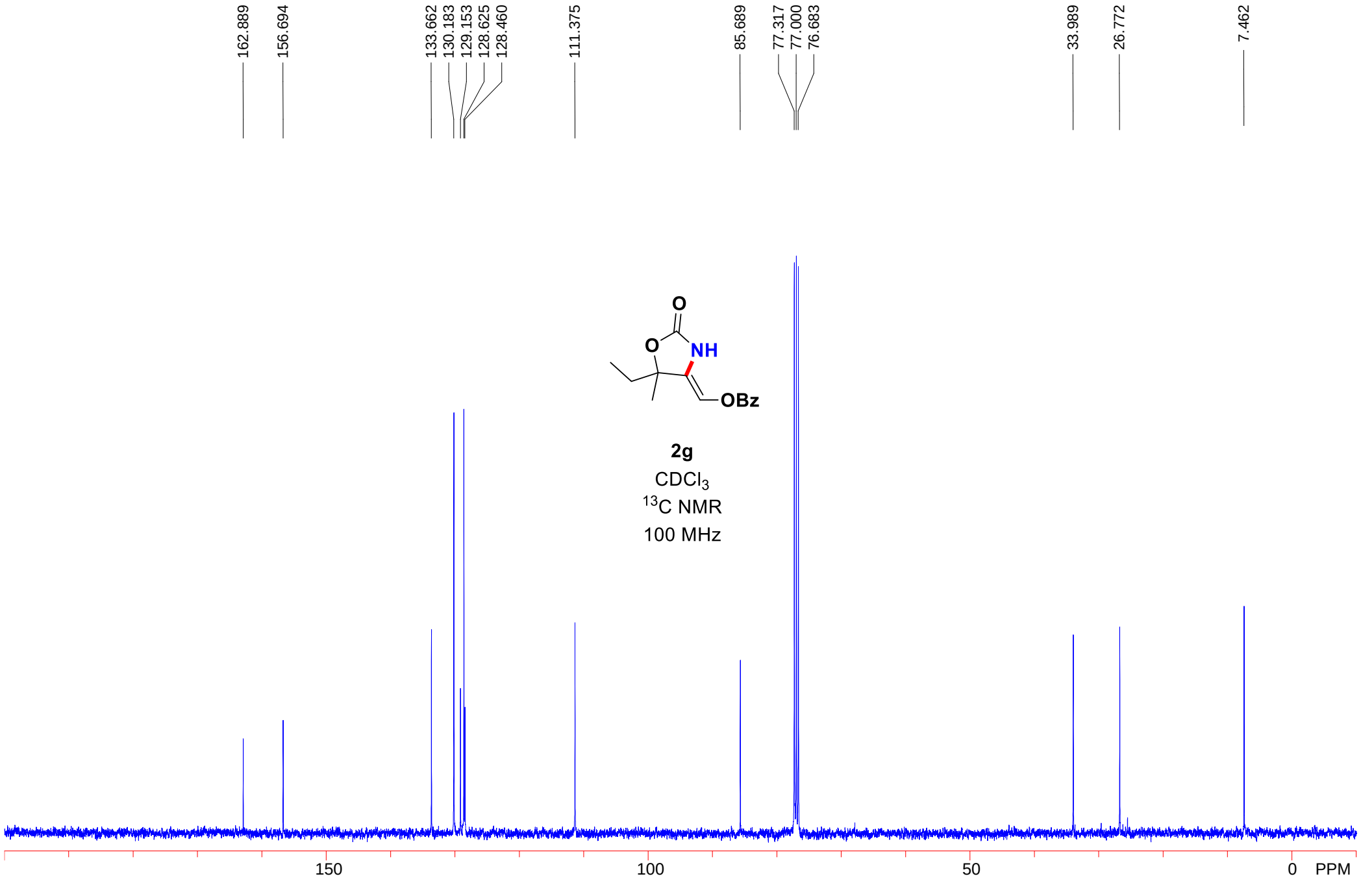
PPM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77.000
76.683
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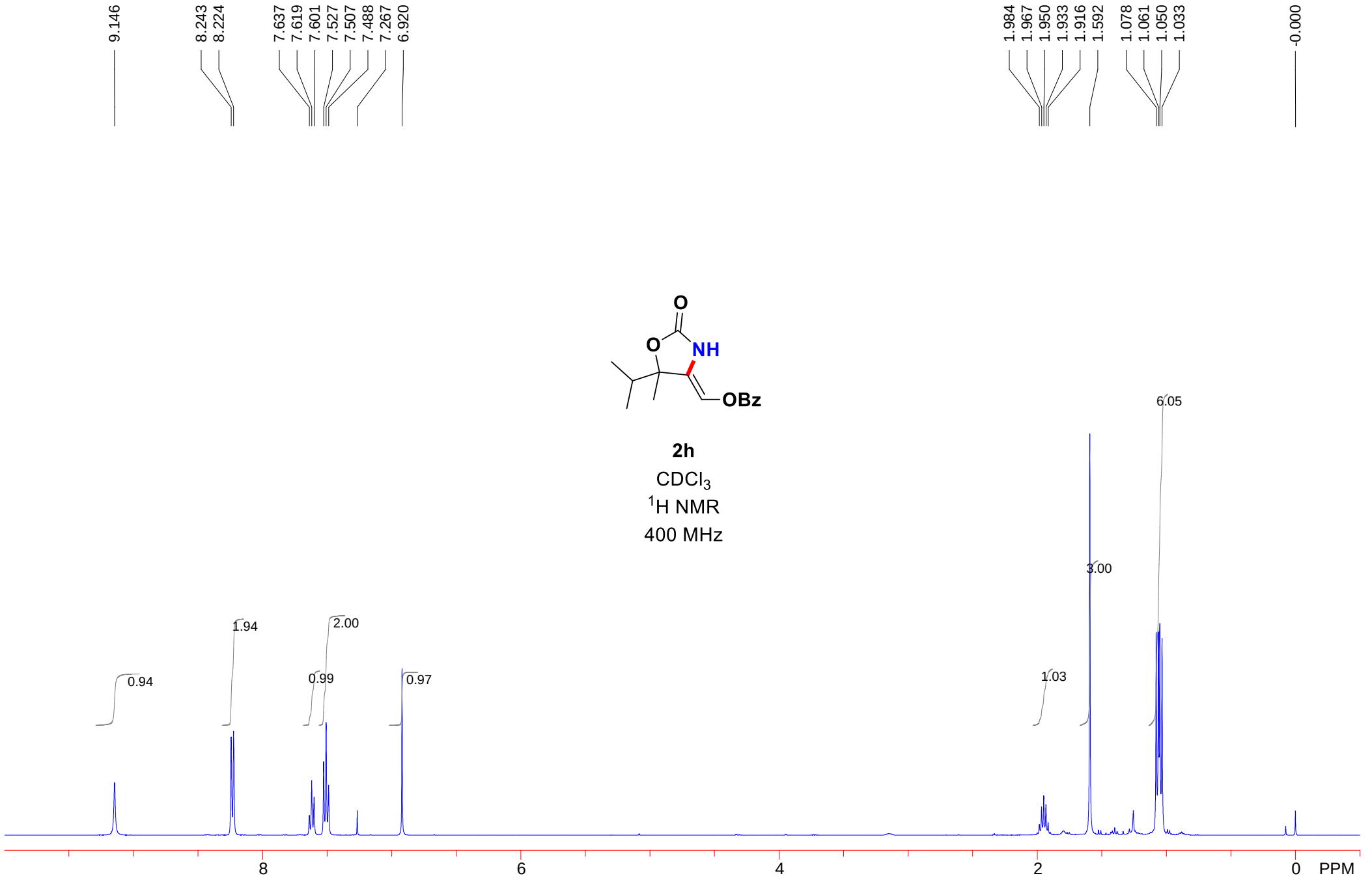
O
O
NH
OBz
2g
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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7.527
7.507
7.488
7.267
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1.967
1.950
1.933
1.916
1.592
1.078
1.061
1.050
1.033
-0.000
O
O
NH
OBz
2h
CDCl3
1H NMR
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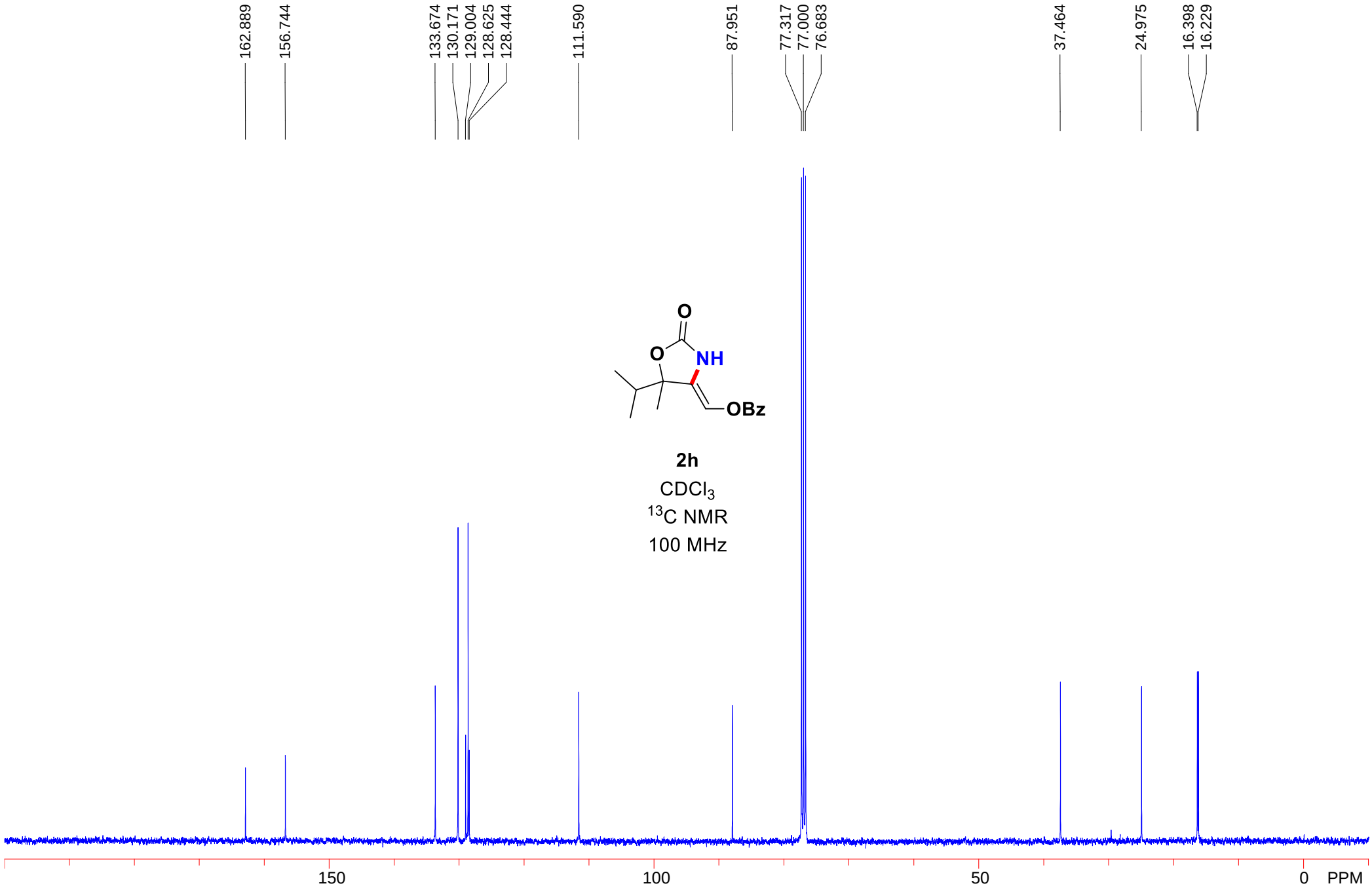
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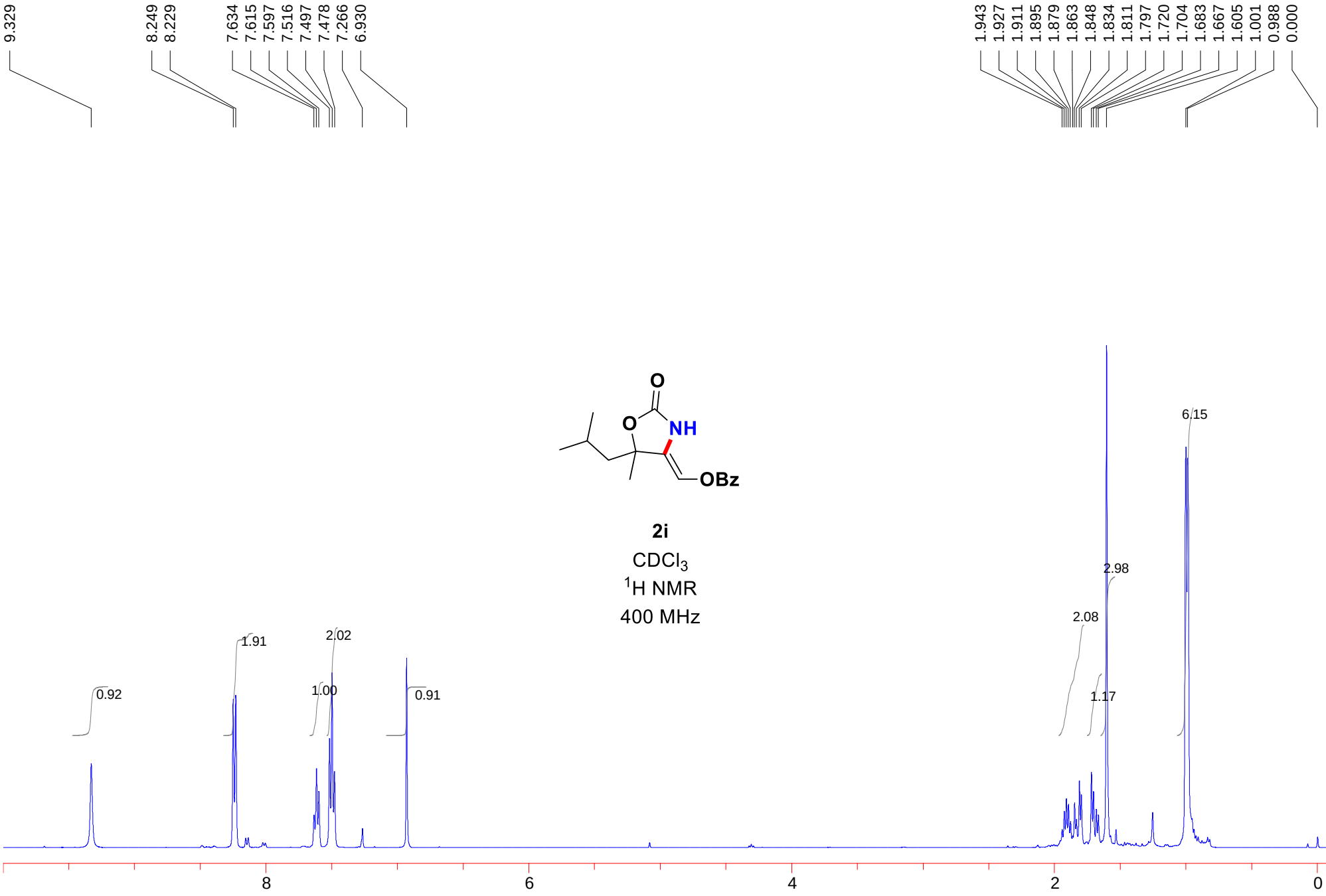
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1.927
1.911
1.895
1.879
1.863
1.848
1.834
1.811
1.797
1.720
1.704
1.683
1.667
1.605
1.001
0.988
0.000
O
O
NH
OBz
2i
CDCl3
1H NMR
400 MHz
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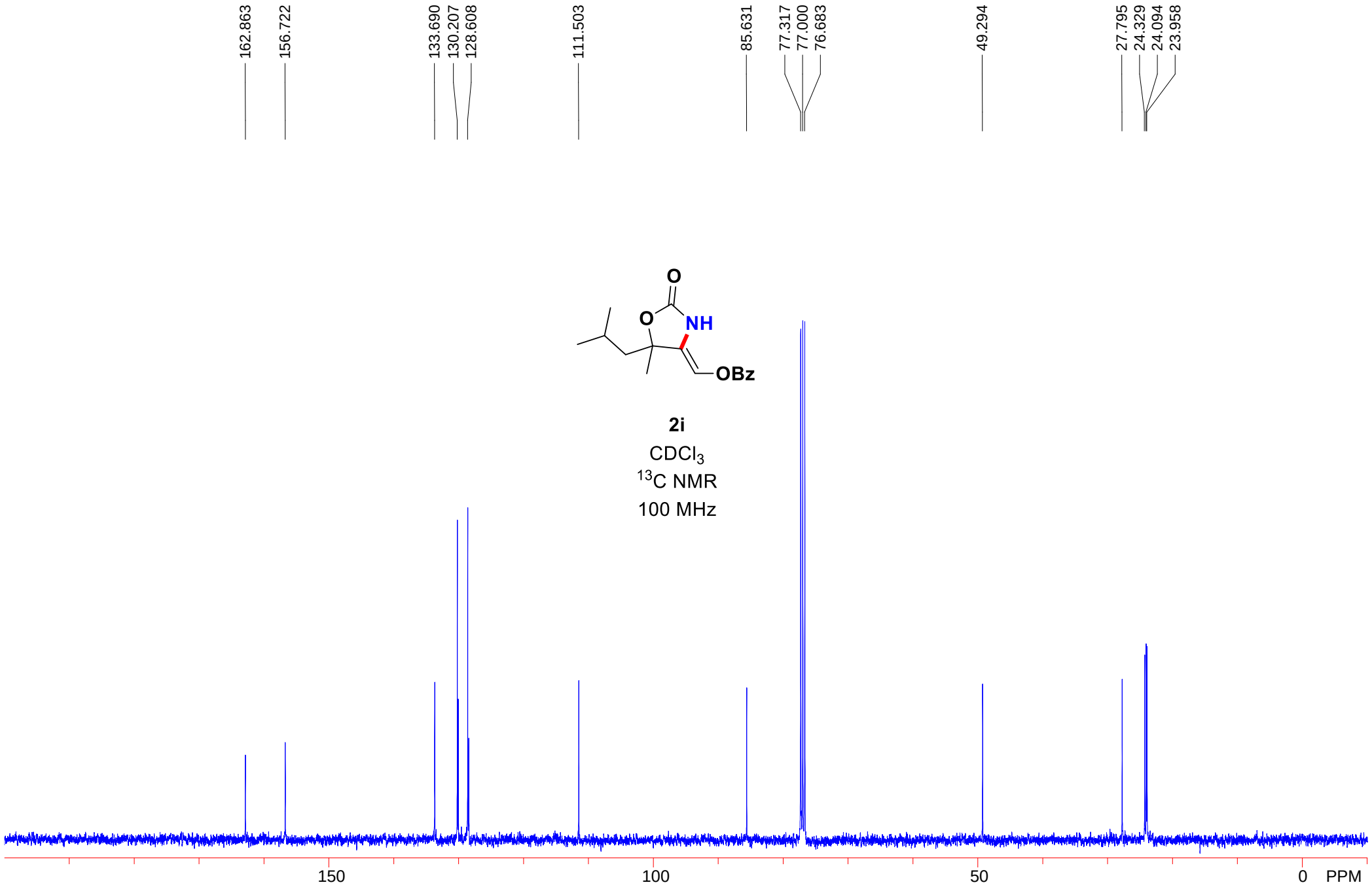
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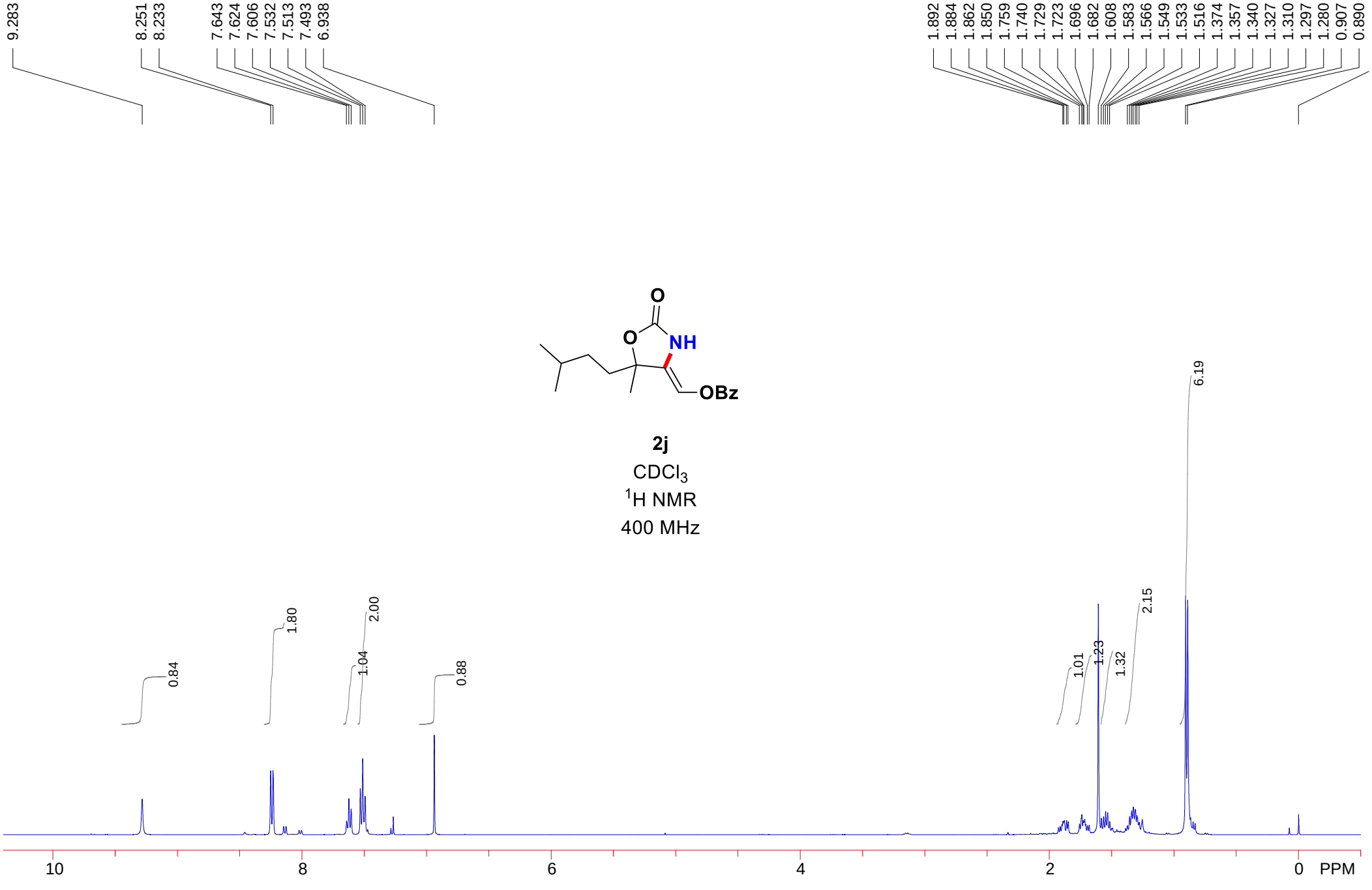
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1.850
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1.740
1.729
1.723
1.696
1.682
1.608
1.583
1.566
1.549
1.533
1.516
1.374
1.357
1.340
1.327
1.310
1.297
1.280
0.907
0.890
O
O
NH
OBz
2j
CDCl3
1H NMR
400 MHz
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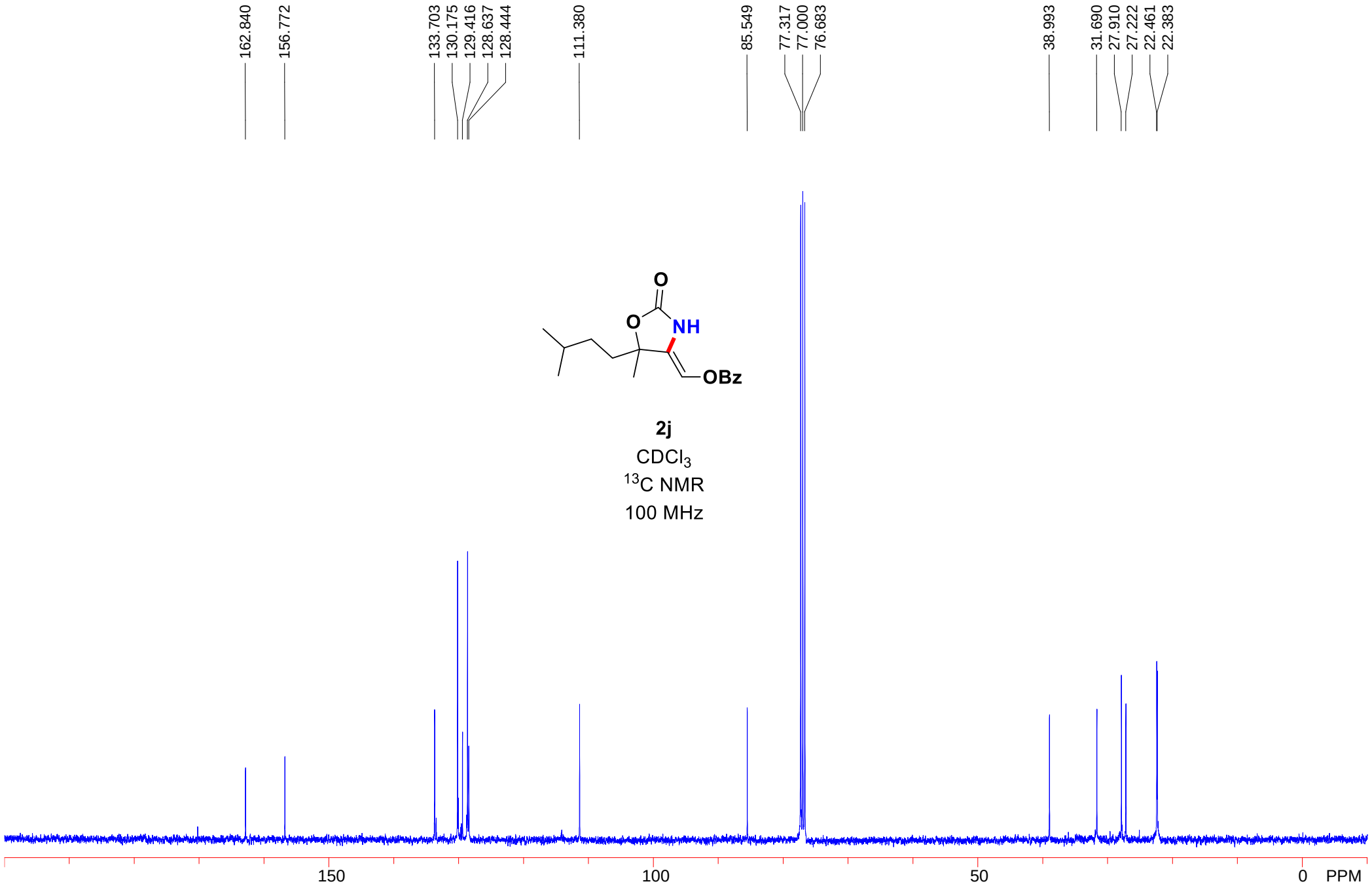
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76.683
38.993
31.690
27.910
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OBz
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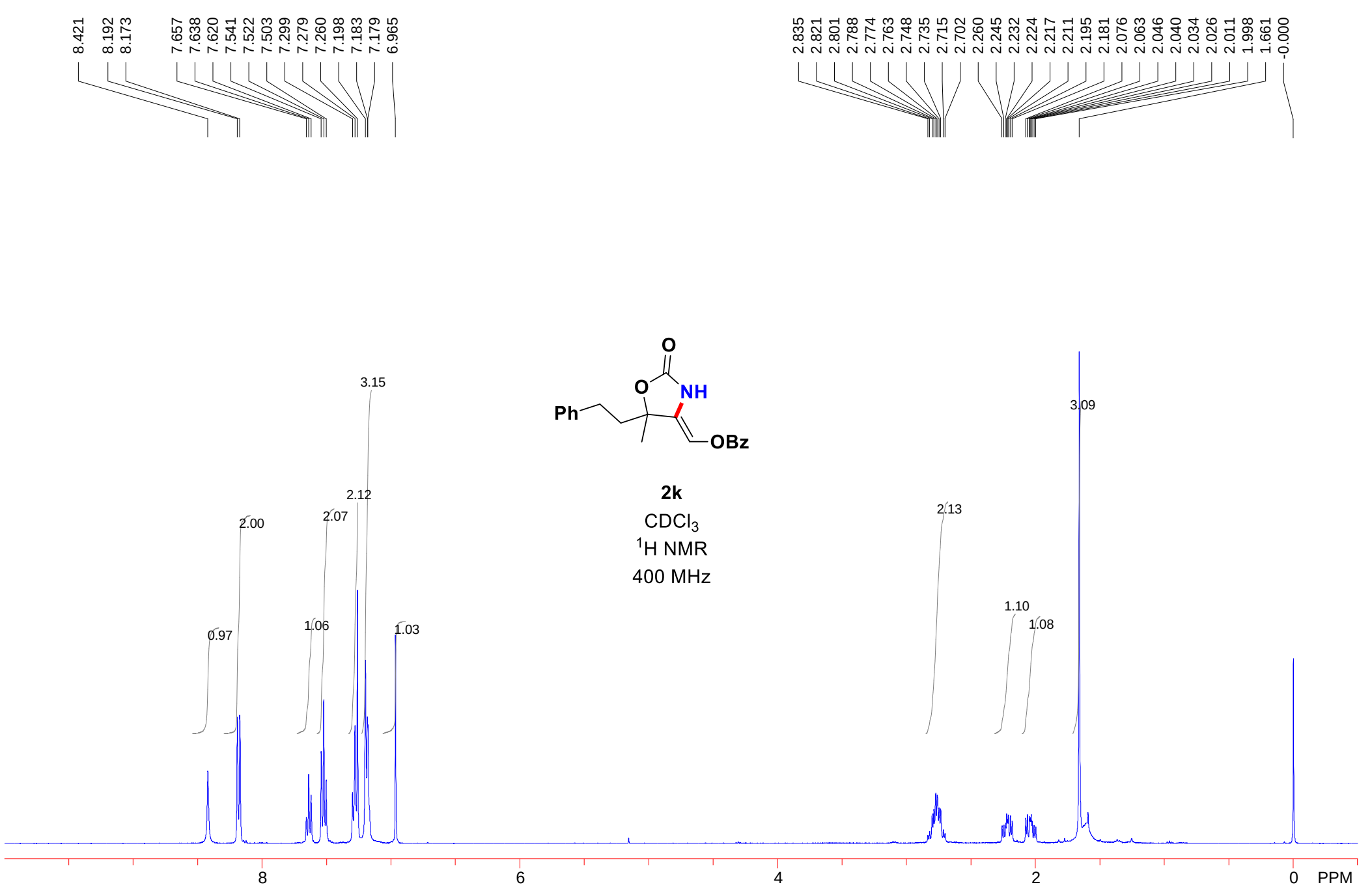
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2.821
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2.788
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2.763
2.748
2.735
2.715
2.702
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2.245
2.232
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Ph
OBz
2k
CDCl3
1H NMR
400 MHz
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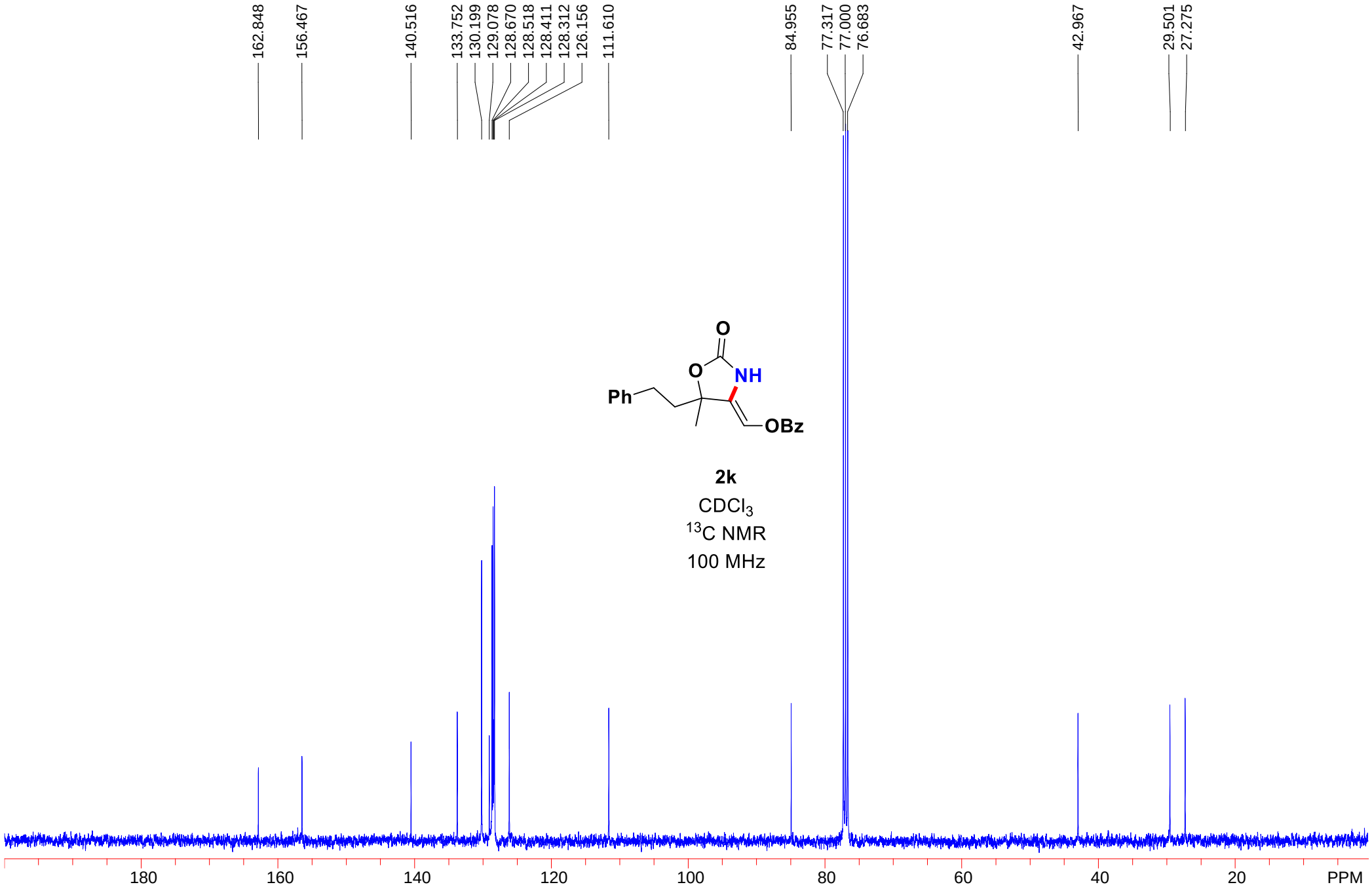
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84.955
77.317
77.000
76.683
42.967
29.501
27.275
O
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120
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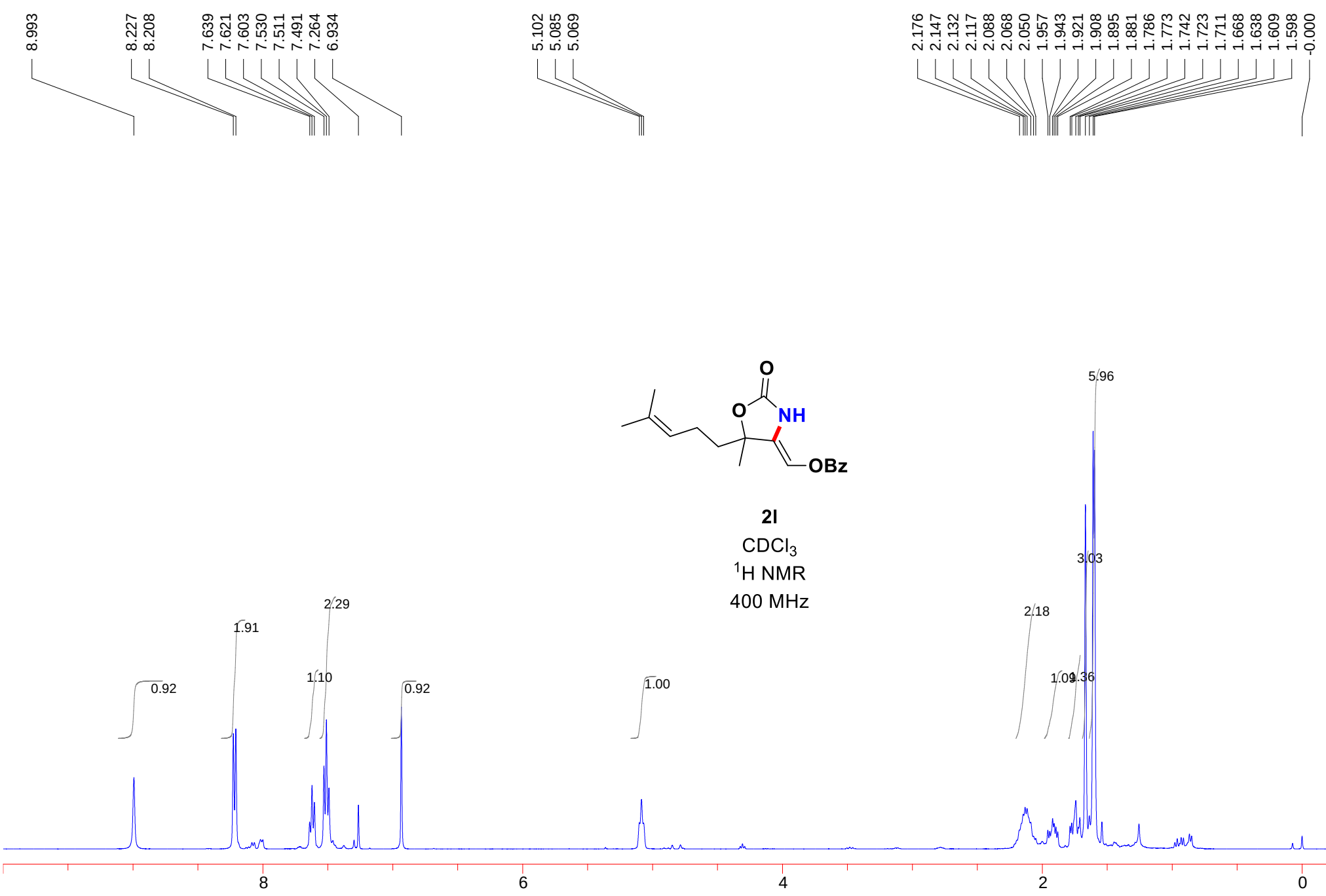
PPM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2l
CDCl3
1H NMR
400 MHz
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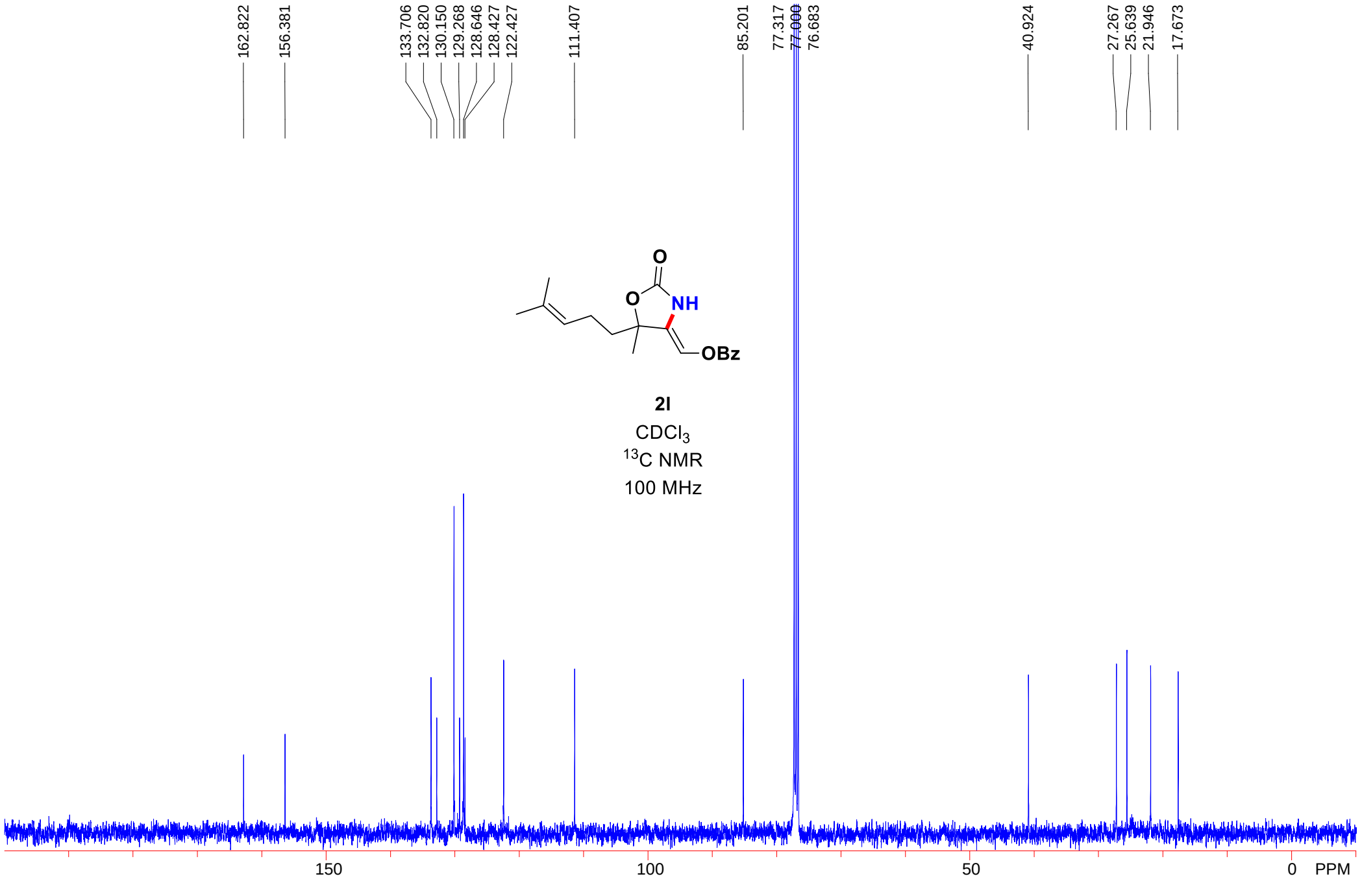
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77.000
76.683
40.924
27.267
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13C NMR
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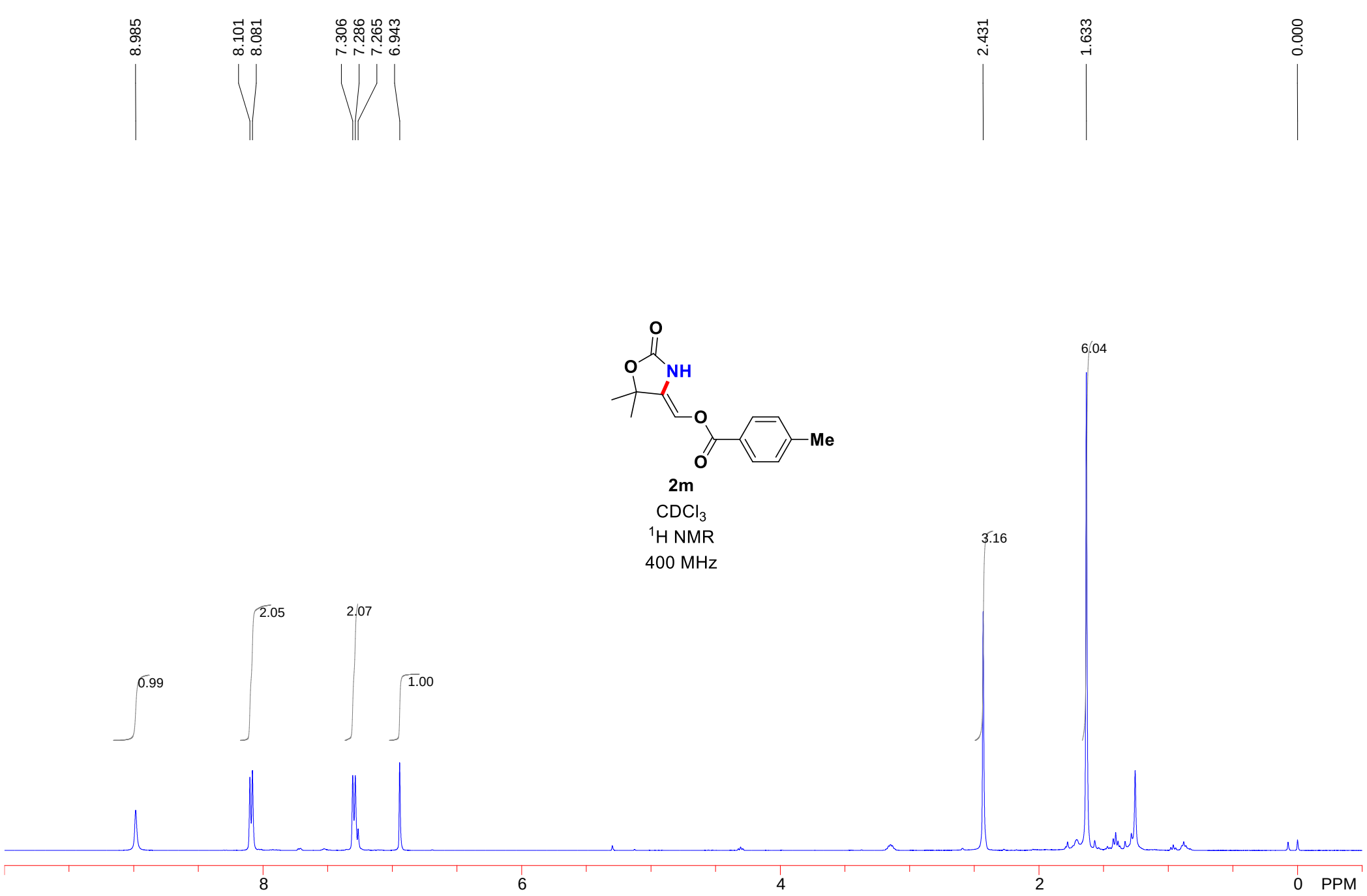
PPM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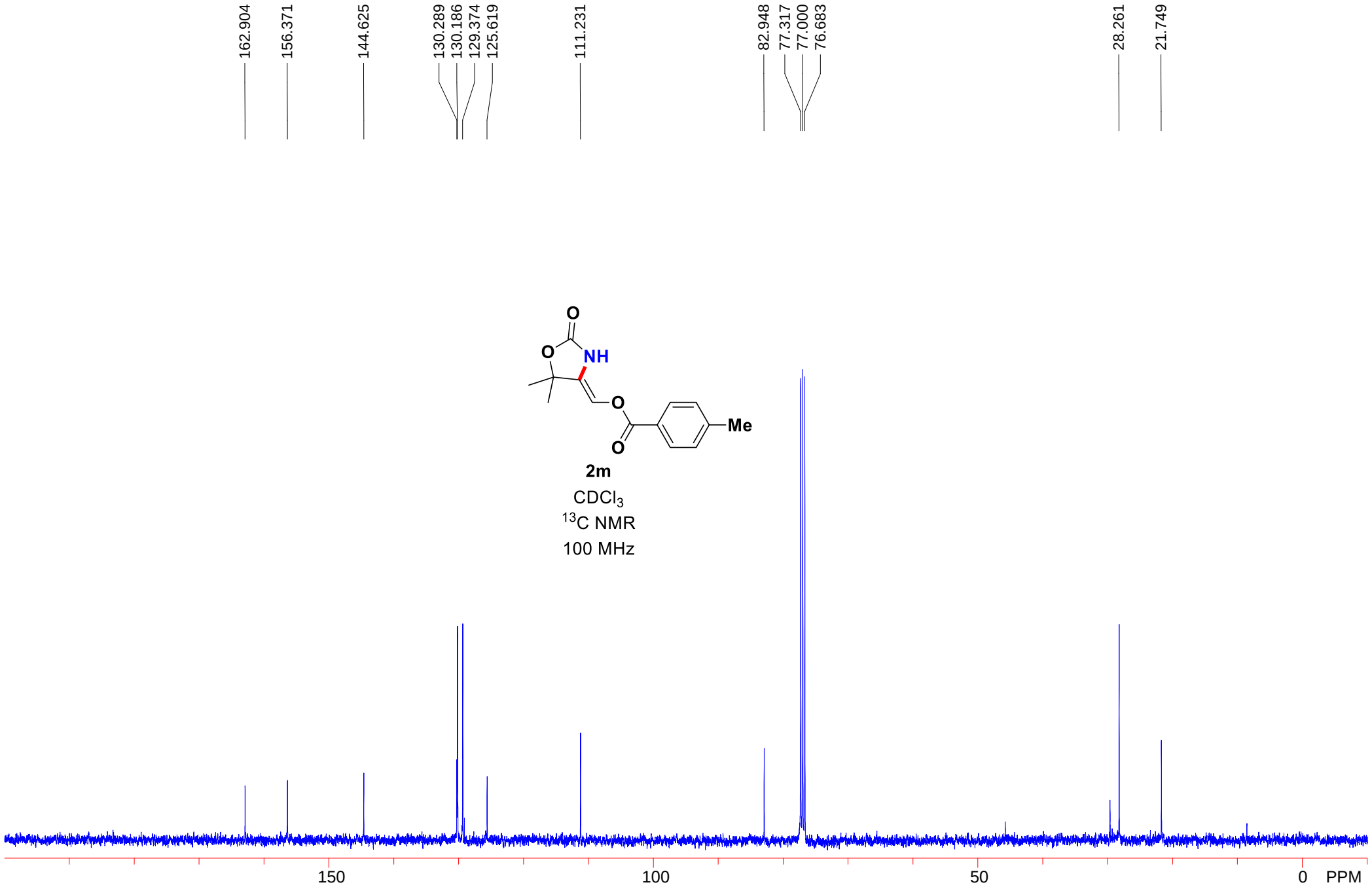
7.306
7.286
7.265
6.943
2.431
1.633
0.000
O
O
NH
O
O
Me
2m
CDCl3
1H NMR
400 MHz
0.99
2.05
2.07
1.00
3.16
6.04
8
6
4
2
0
PPM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156.371
144.625
130.289
130.186
129.374
125.619
111.231
82.948
77.317
77.000
76.683
28.261
21.749
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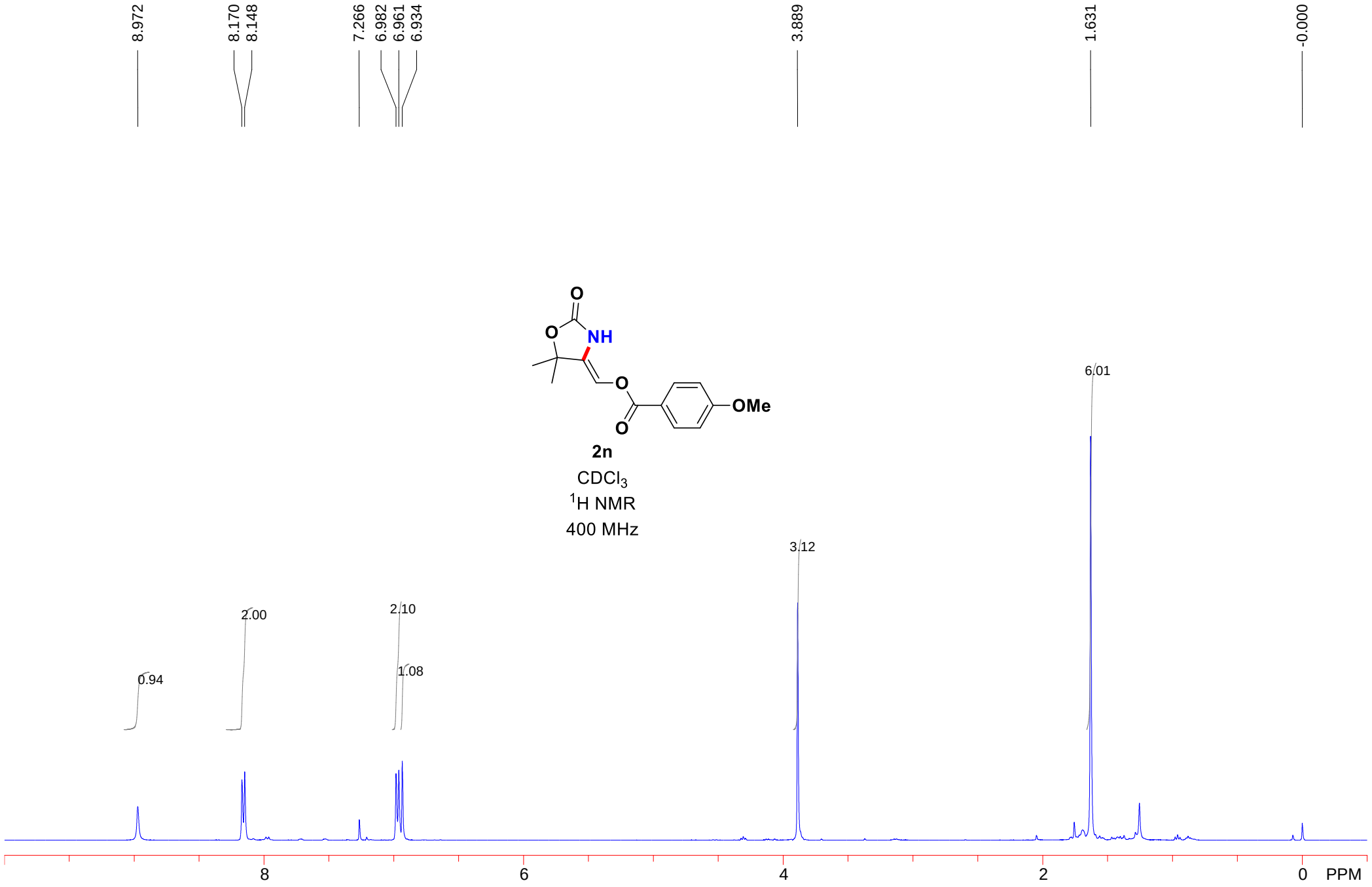
O
NH
O
O
Me
2m
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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8.170
8.148
7.266
6.982
6.961
6.934
3.889
1.631
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O
NH
O
O
OMe
2n
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8
6
4
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0
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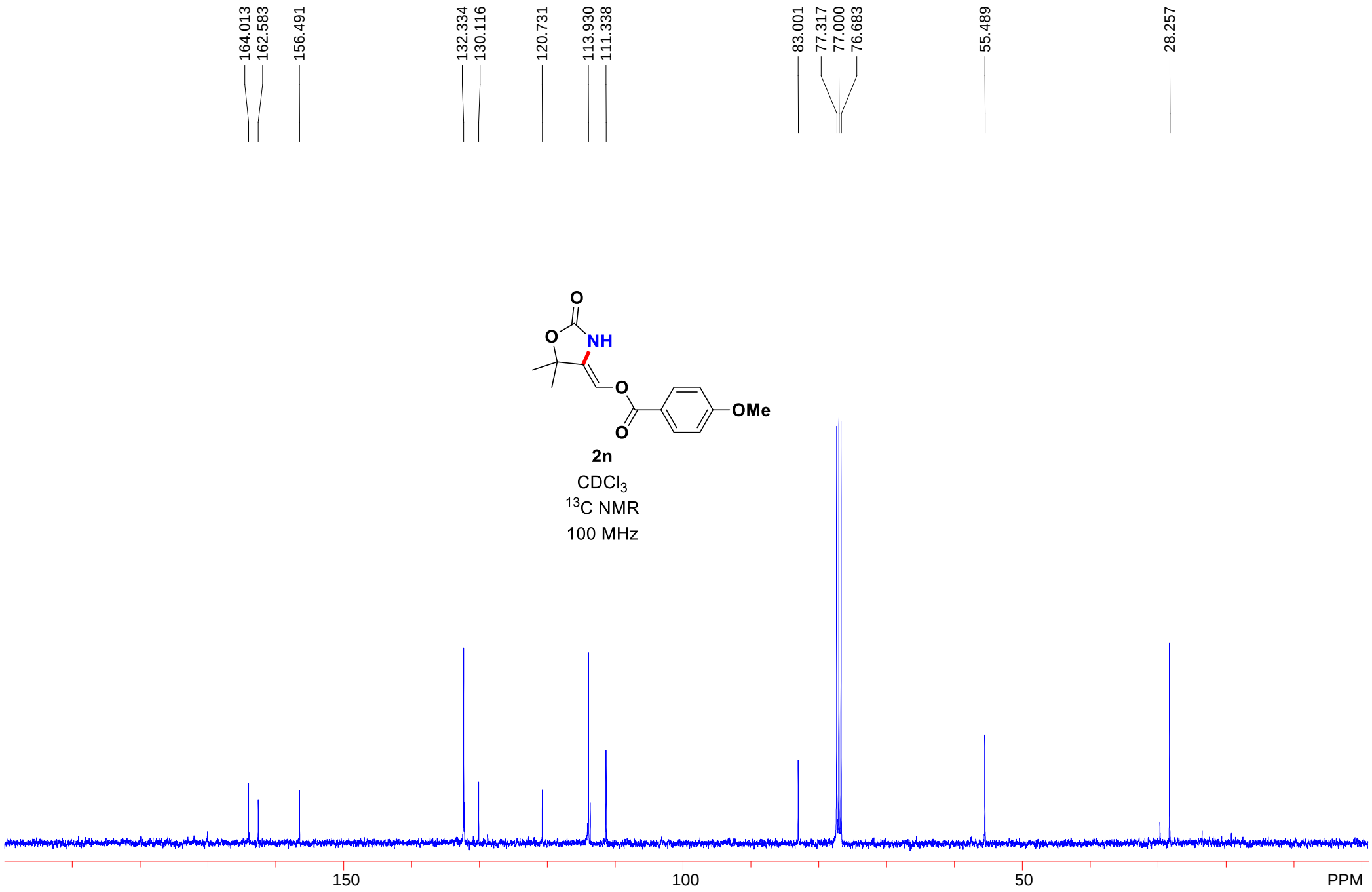
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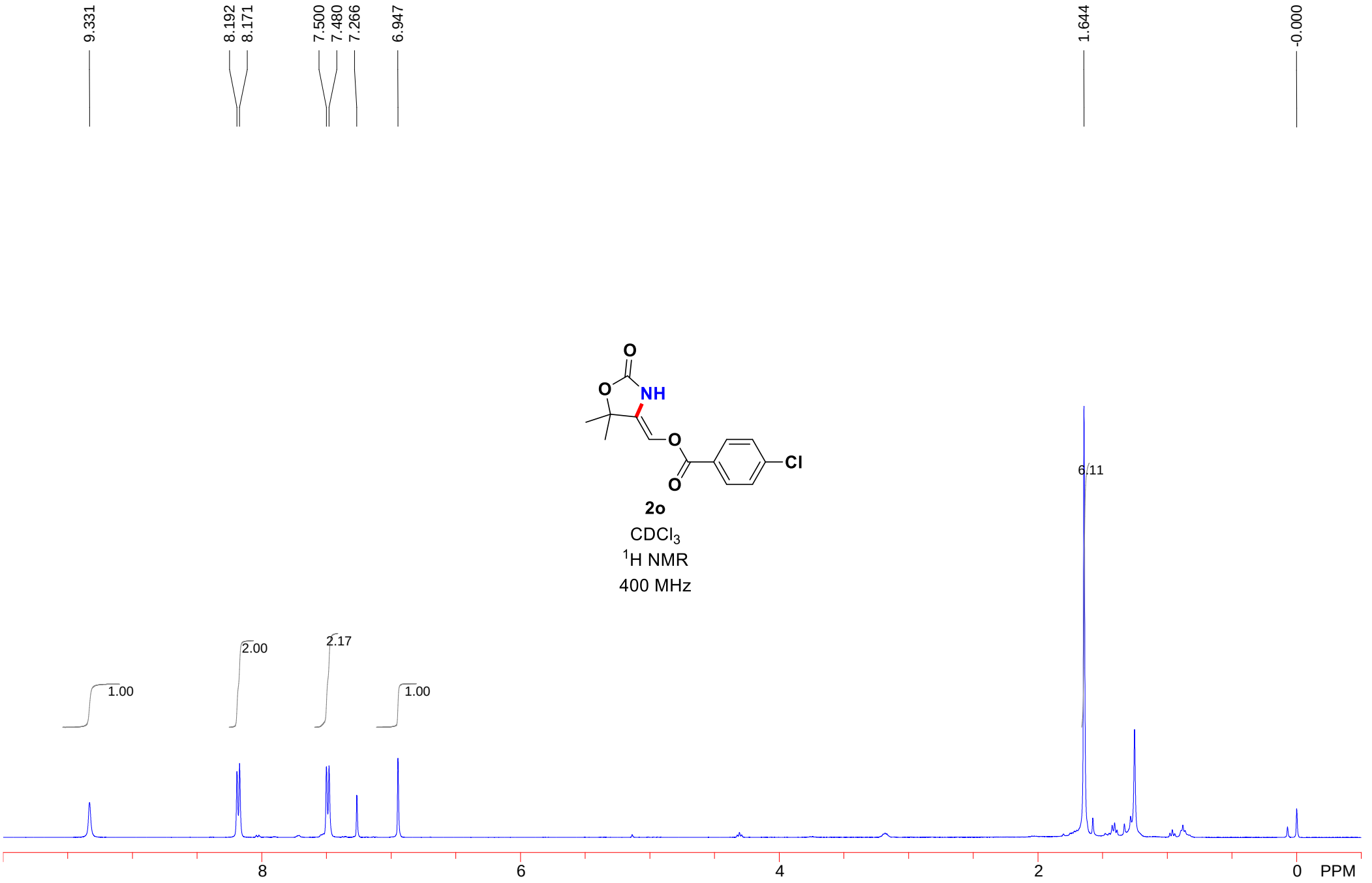
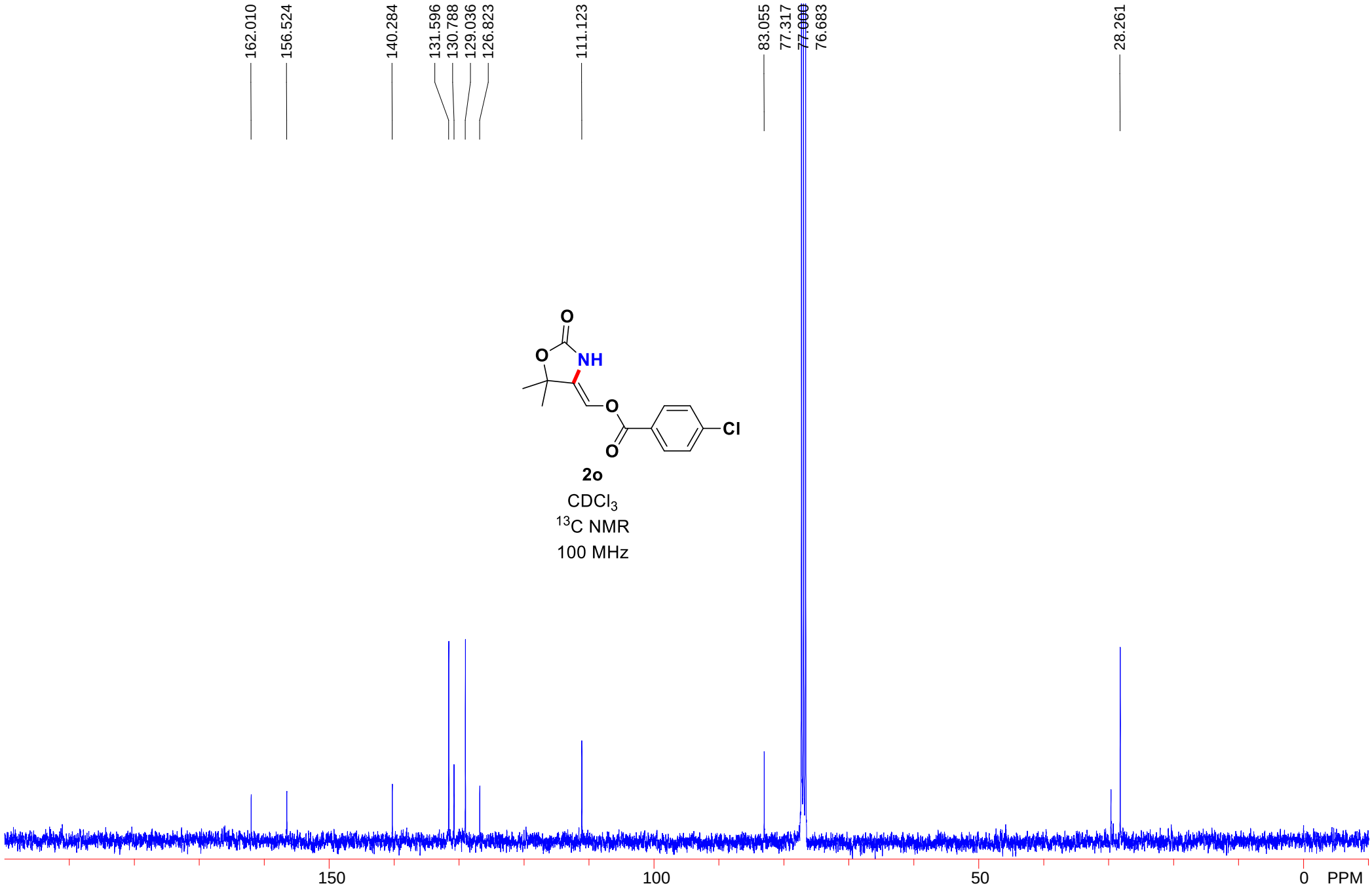
162.583
156.491
132.334
130.116
120.731
113.930
111.338
83.001
77.317
77.000
76.683
55.489
28.257
O
O
NH
O
O
OMe
2n
CDCl3
13C NMR
100 MHz
150
100
50
PPM


9.331
8.192
8.171
7.500
7.480
7.266
6.947
1.644
-0.000
O
O
NH
O
O
Cl
2o
CDCl3
1H NMR
400 MHz
1.00
2.00
2.17
1.00
6.11
8
6
4
2
0
PPM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111.123
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77.000
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28.261
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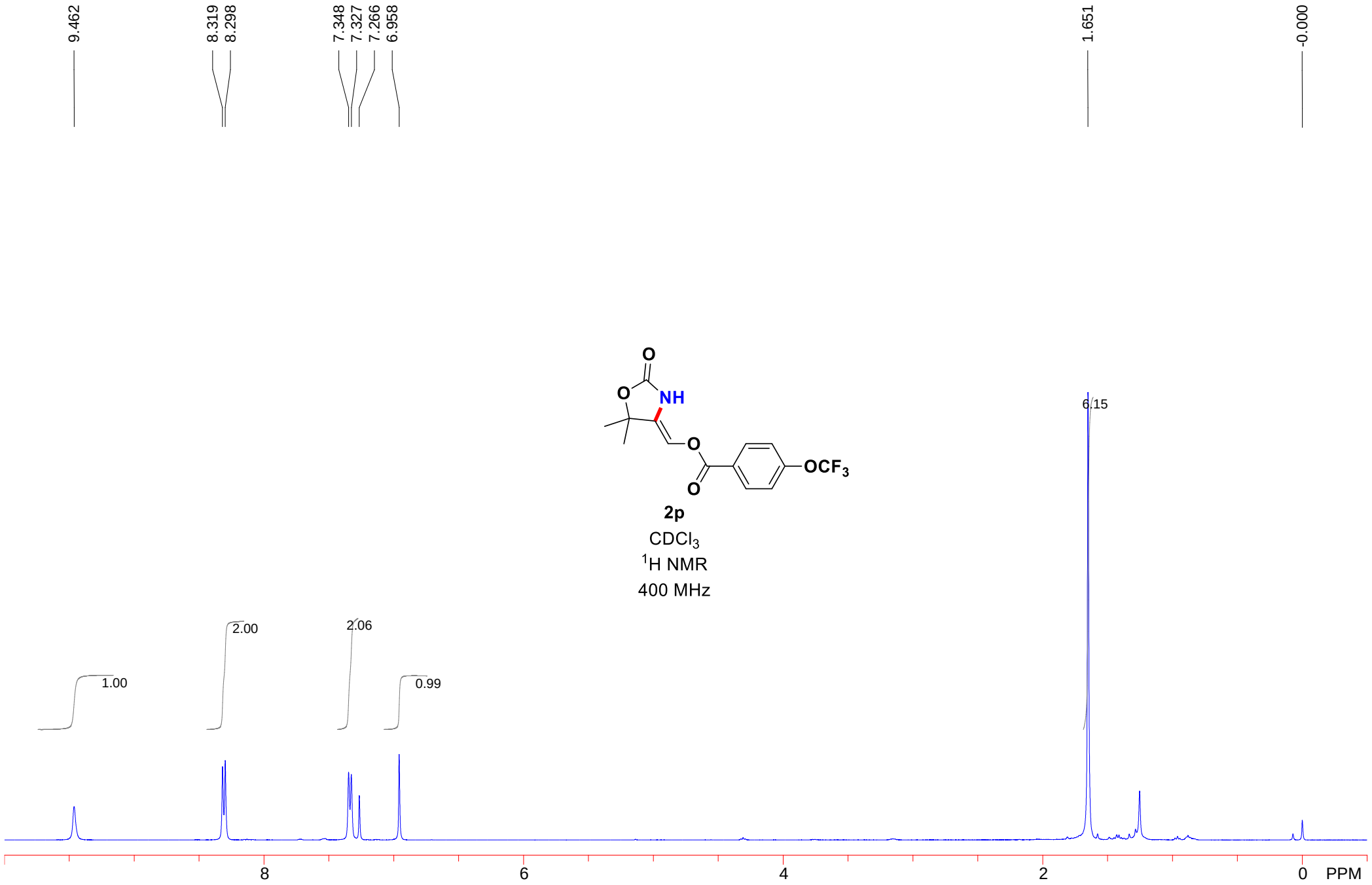
O
NH
O
O
Cl
2o
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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8.319
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7.348
7.327
7.266
6.958
1.651
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O
O
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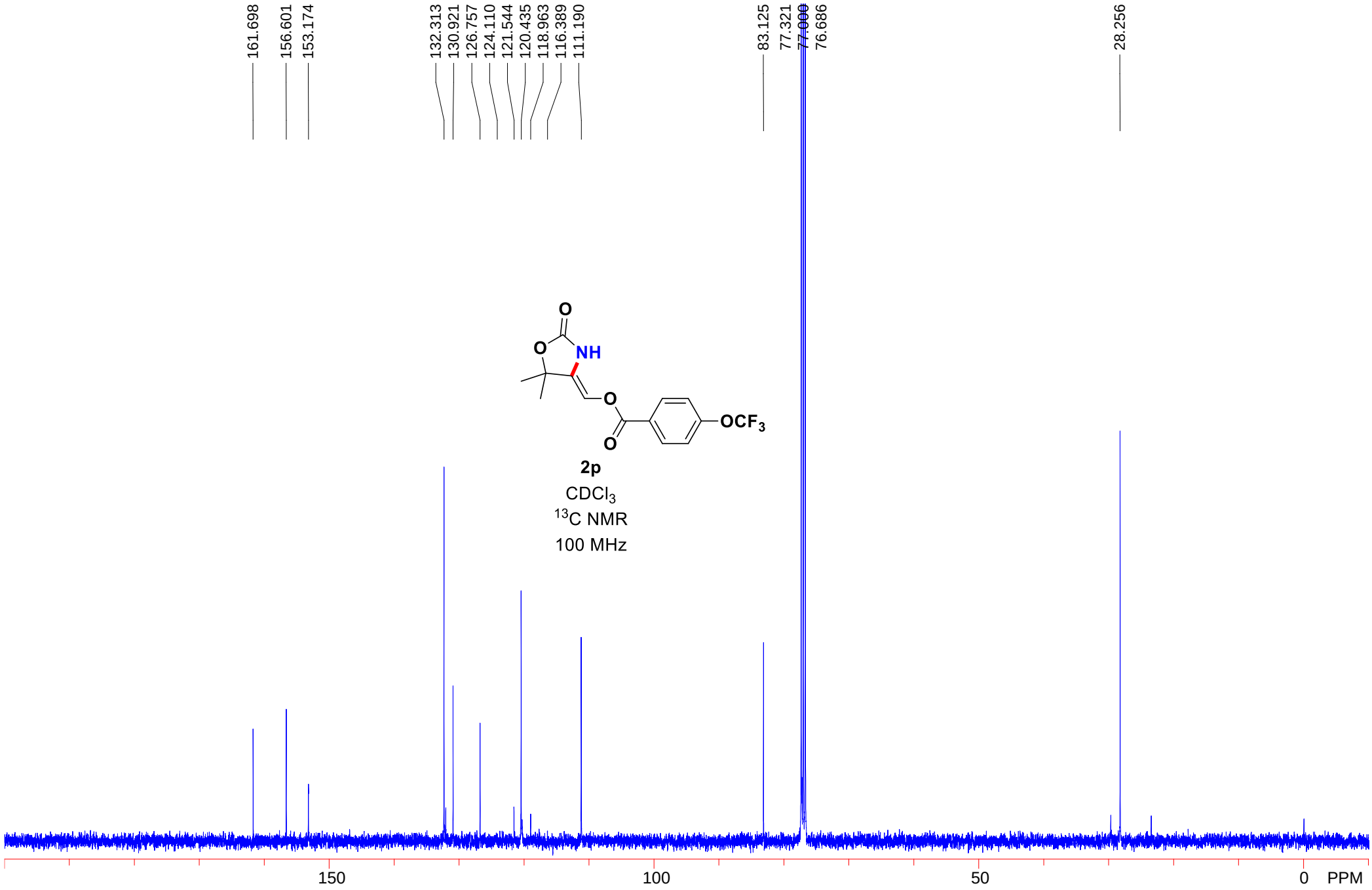
O
O
OCF3
2p
CDCl3
1H NMR
400 MHz
1.00
2.00
2.06
0.99
6.15
8
6
4
2
0
PPM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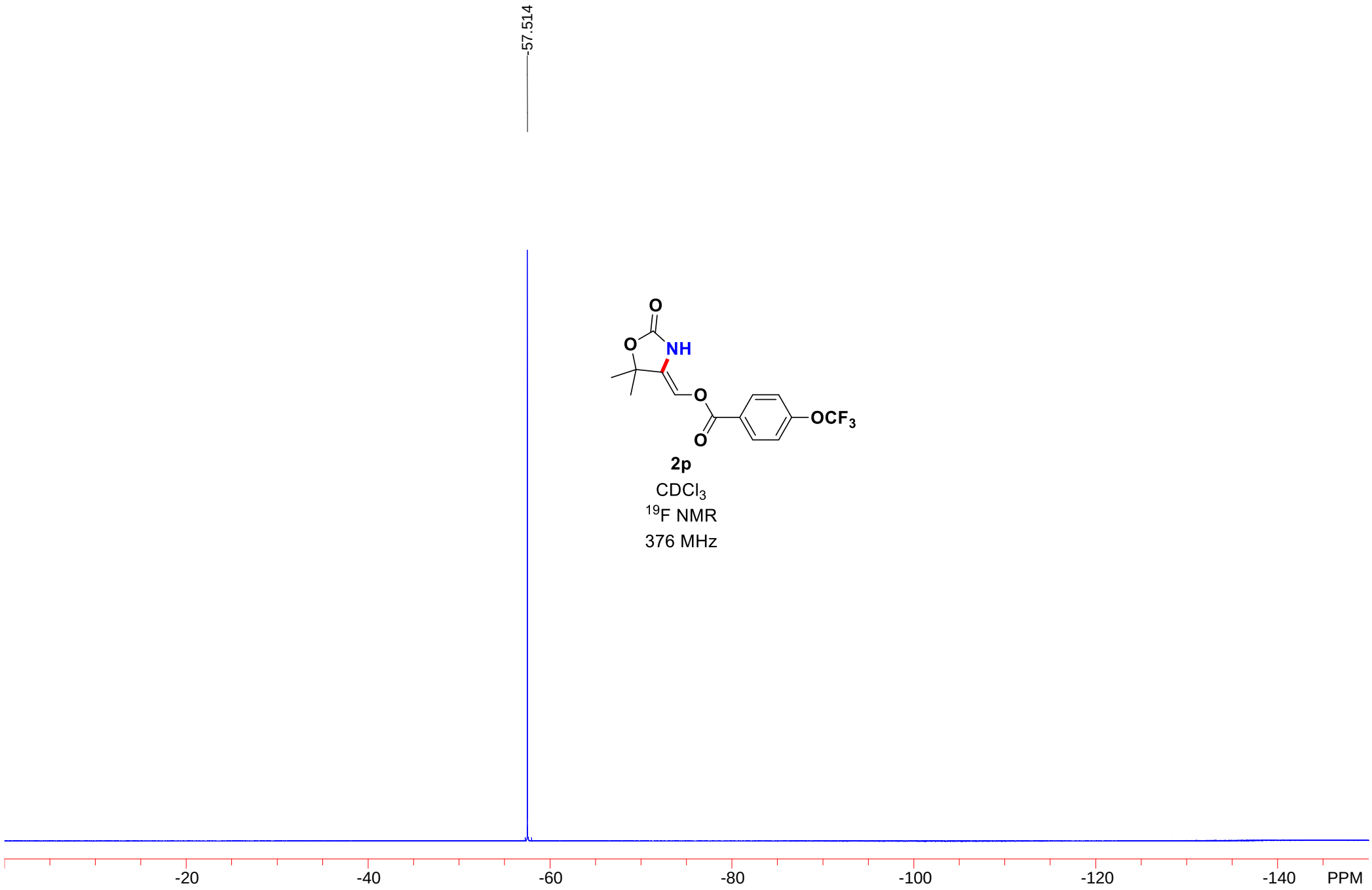
156.601
153.174
132.313
130.921
126.757
124.110
121.544
120.435
118.963
116.389
111.190
83.125
77.321
77.000
76.686
28.256
O
O
NH
O
O
OCF3
2p
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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O
O
NH
O
O
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2p
CDCl3
19F NMR
376 MHz
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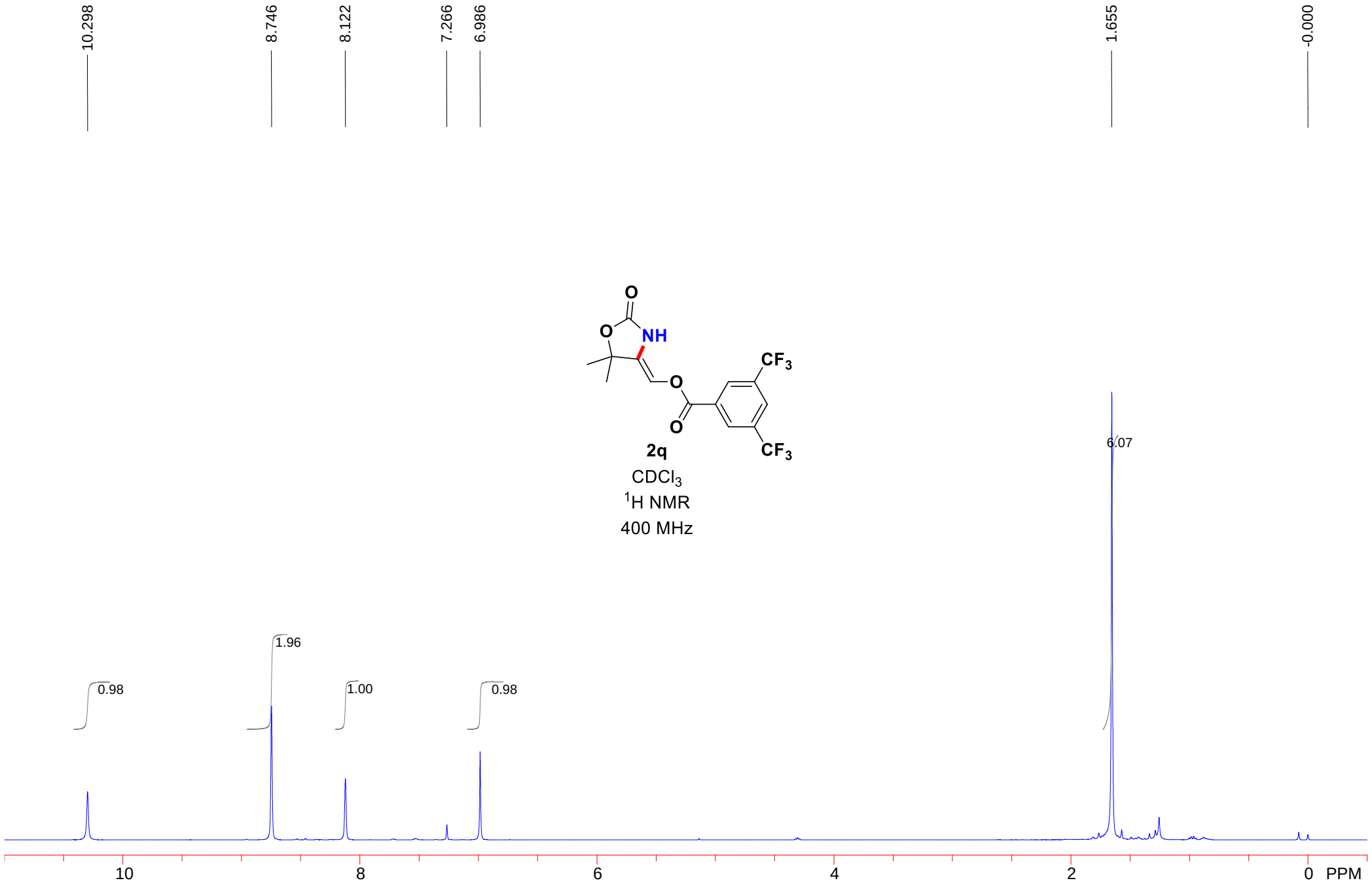
-40
-60
-80
-100
-120
-140
PPM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8.746
8.122
7.266
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-0.000
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O
NH
O
O
CF3
CF3
2q
CDCl3
1H NMR
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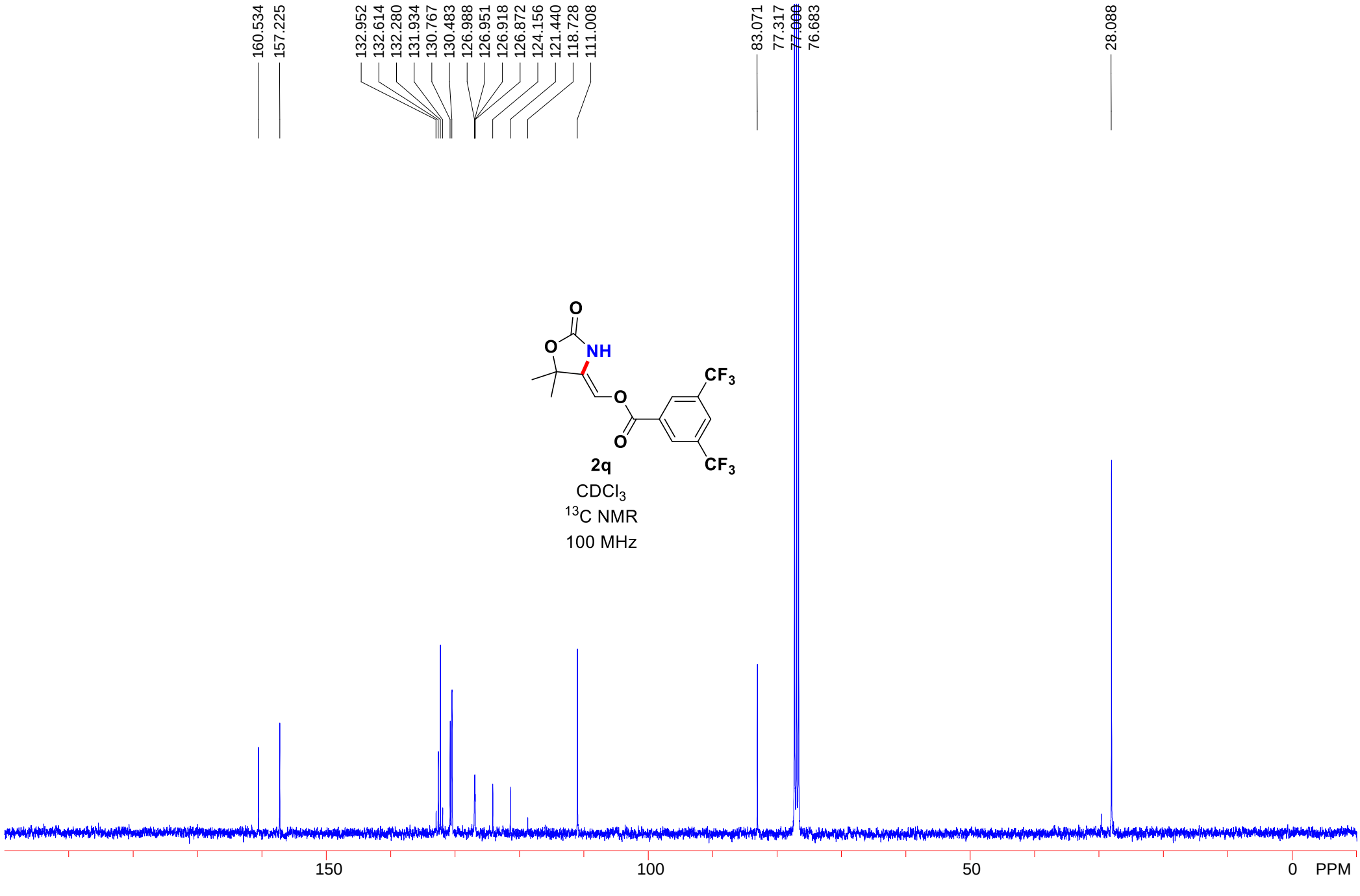
1.00
0.98
6.07
10
8
6
4
2
0
PPM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157.225
132.952
132.614
132.280
131.934
130.767
130.483
126.988
126.951
126.918
126.872
124.156
121.440
118.728
111.008
83.071
77.317
77.000
76.683
28.088
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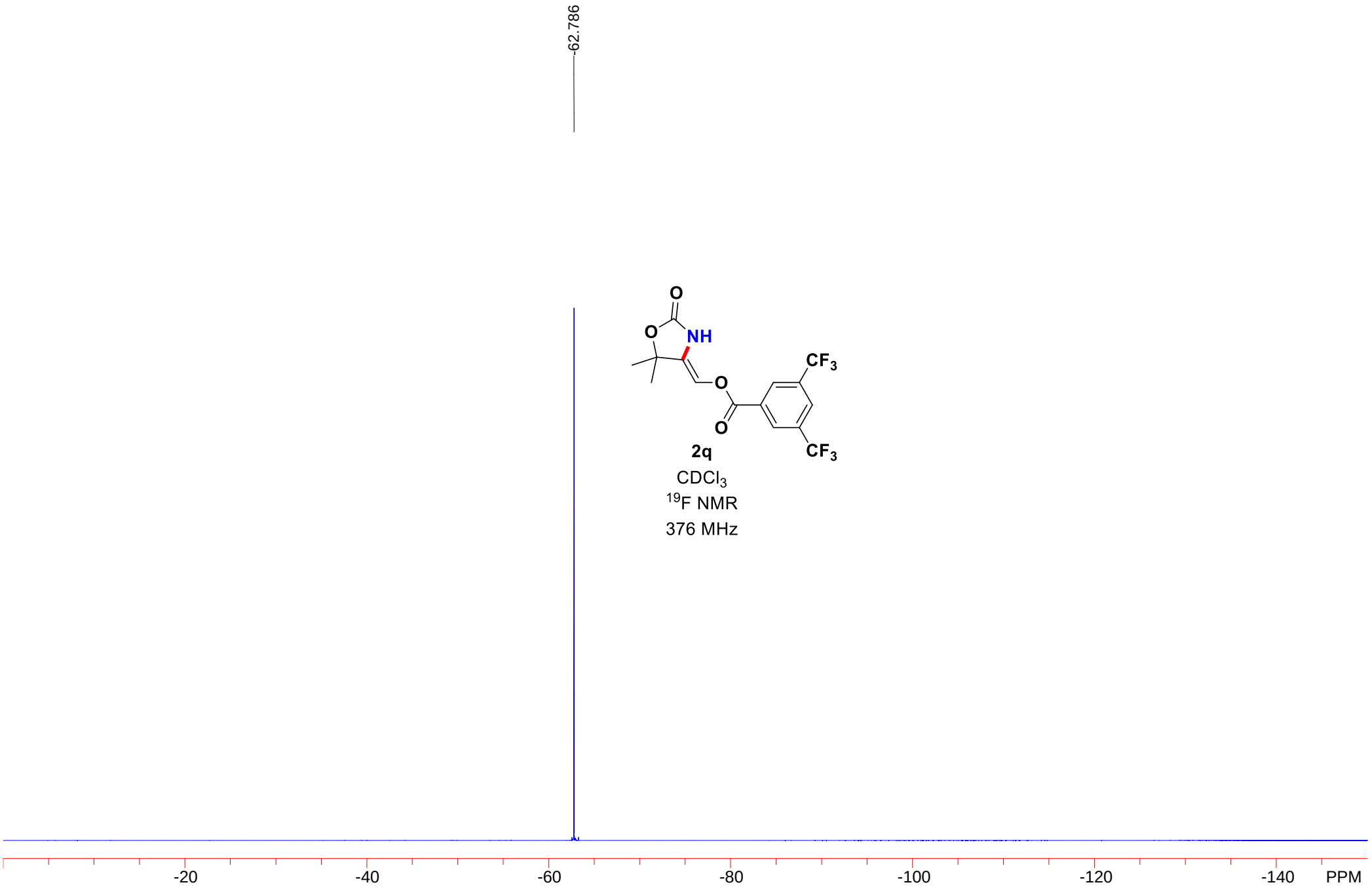
O
NH
O
O
CF3
CF3
2q
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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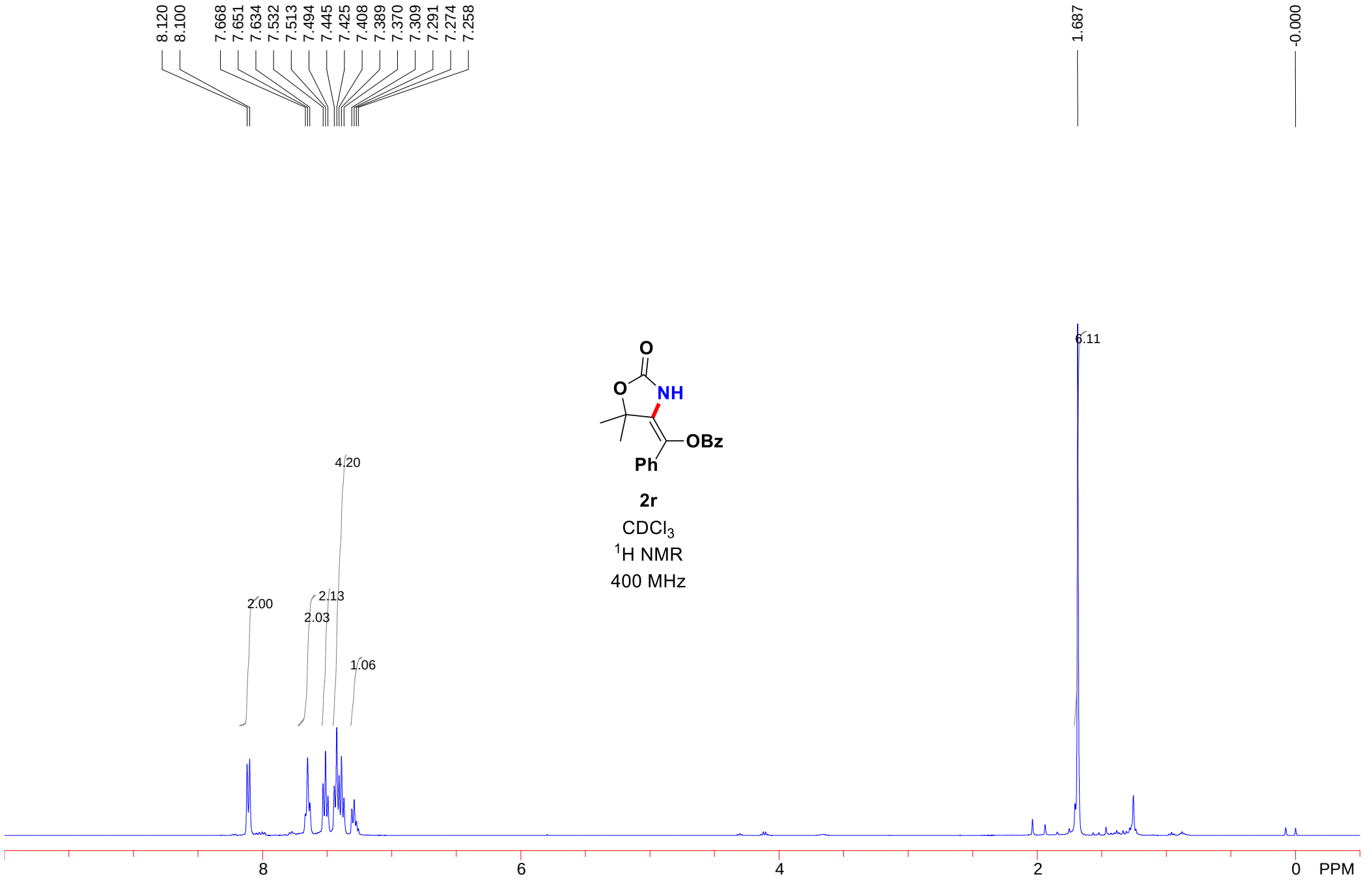
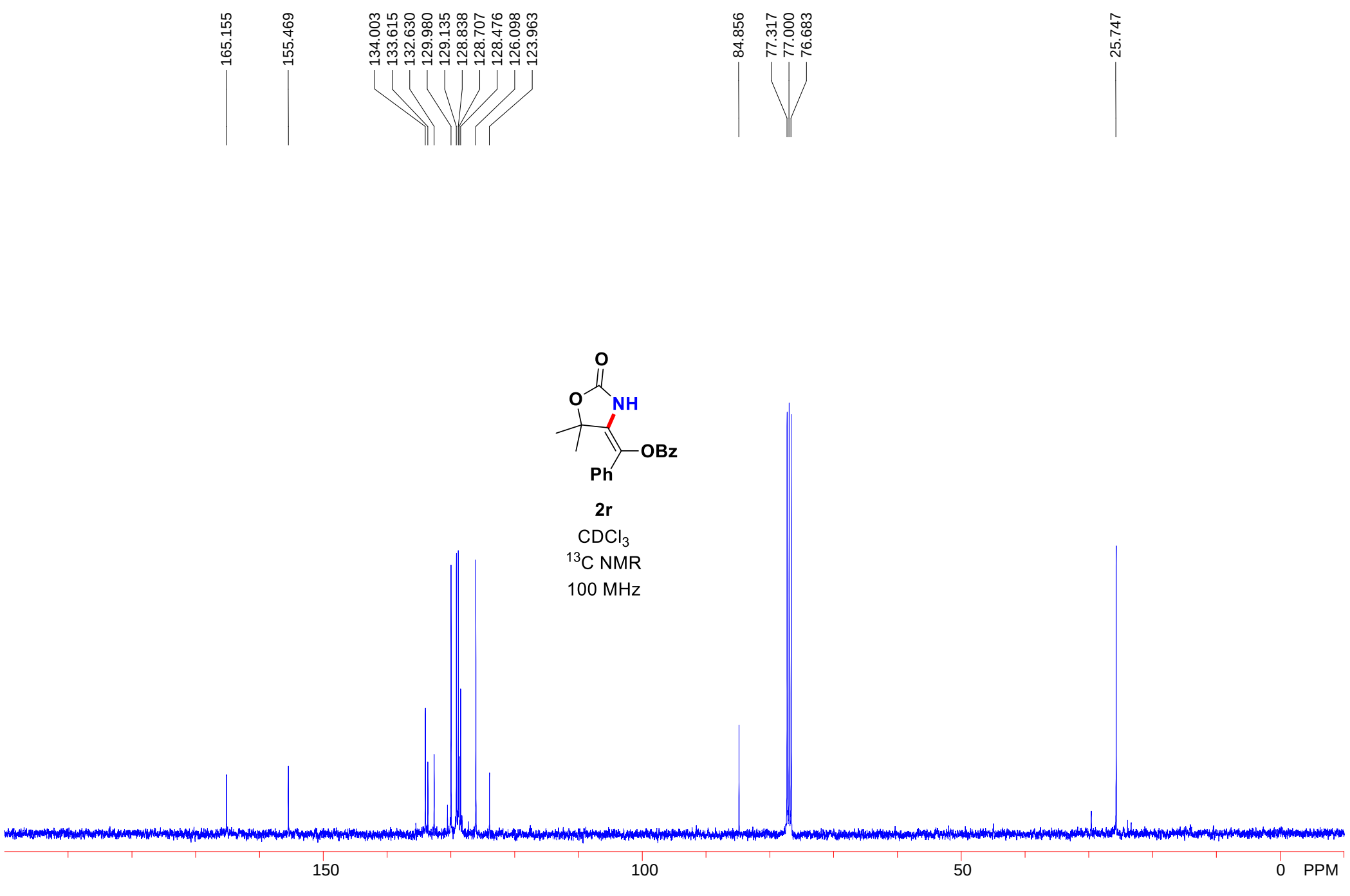
O
O
NH
O
O
CF3
CF3
2q
CDCl3
19F NMR
376 MHz
-20
-40
-60
-80
-100
-120
-140
PPM


8.120
8.100
7.668
7.651
7.634
7.532
7.513
7.494
7.445
7.425
7.408
7.389
7.370
7.309
7.291
7.274
7.258
1.687
-0.000
O
O
NH
OBz
Ph
2r
CDCl3
1H NMR
400 MHz
4.20
2.13
2.00
2.03
1.06
6.11
8
6
4
2
0
PPM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155.469
134.003
133.615
132.630
129.980
129.135
128.838
128.707
128.476
126.098
123.963
84.856
77.317
77.000
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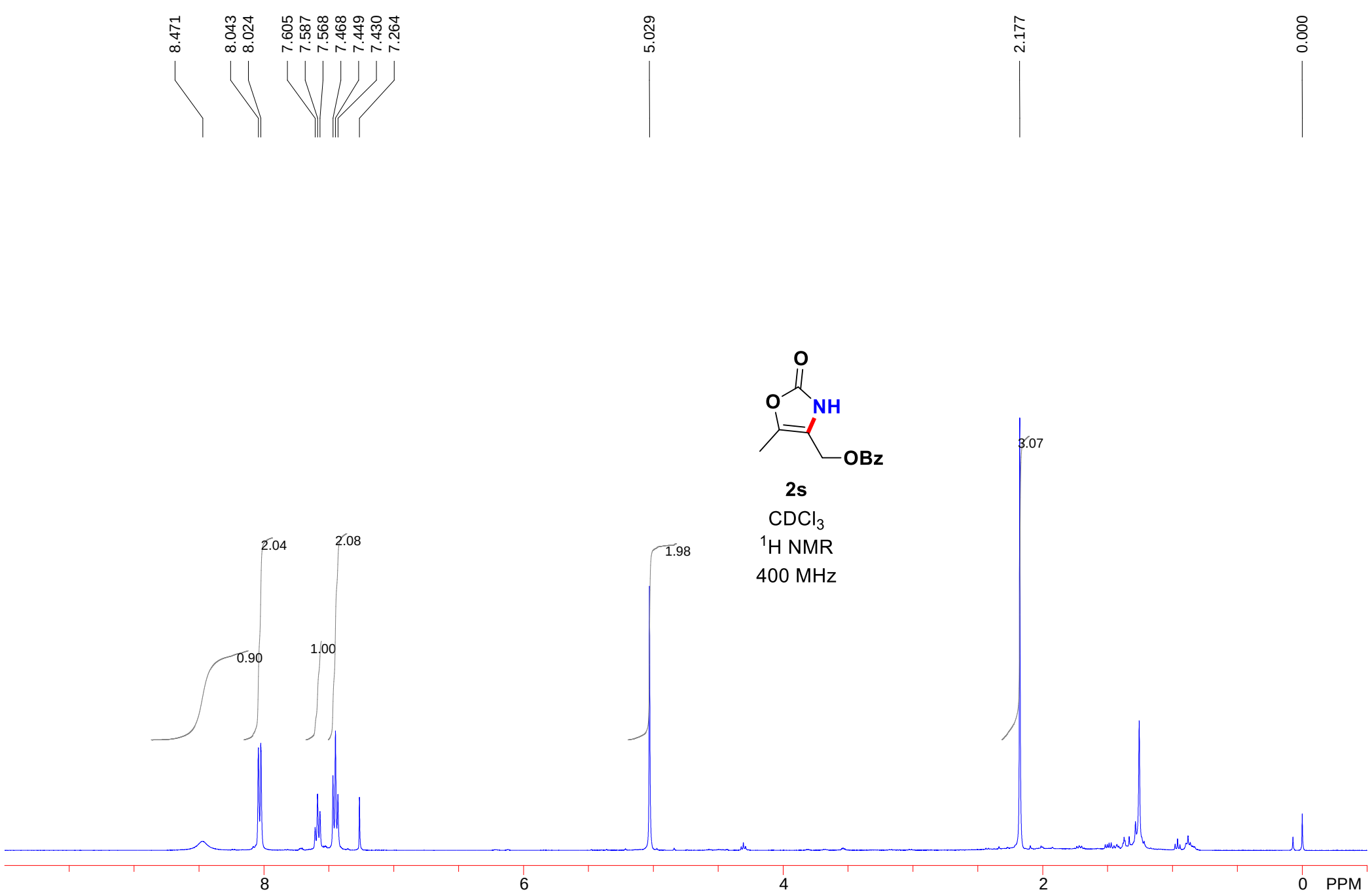
25.747
O
O
NH
OBz
Ph
2r
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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8.043
8.024
7.605
7.587
7.568
7.468
7.449
7.430
7.264
5.029
2.177
0.000
O
O
NH
OBz
2s
CDCl3
1H NMR
400 MHz
2.04
2.08
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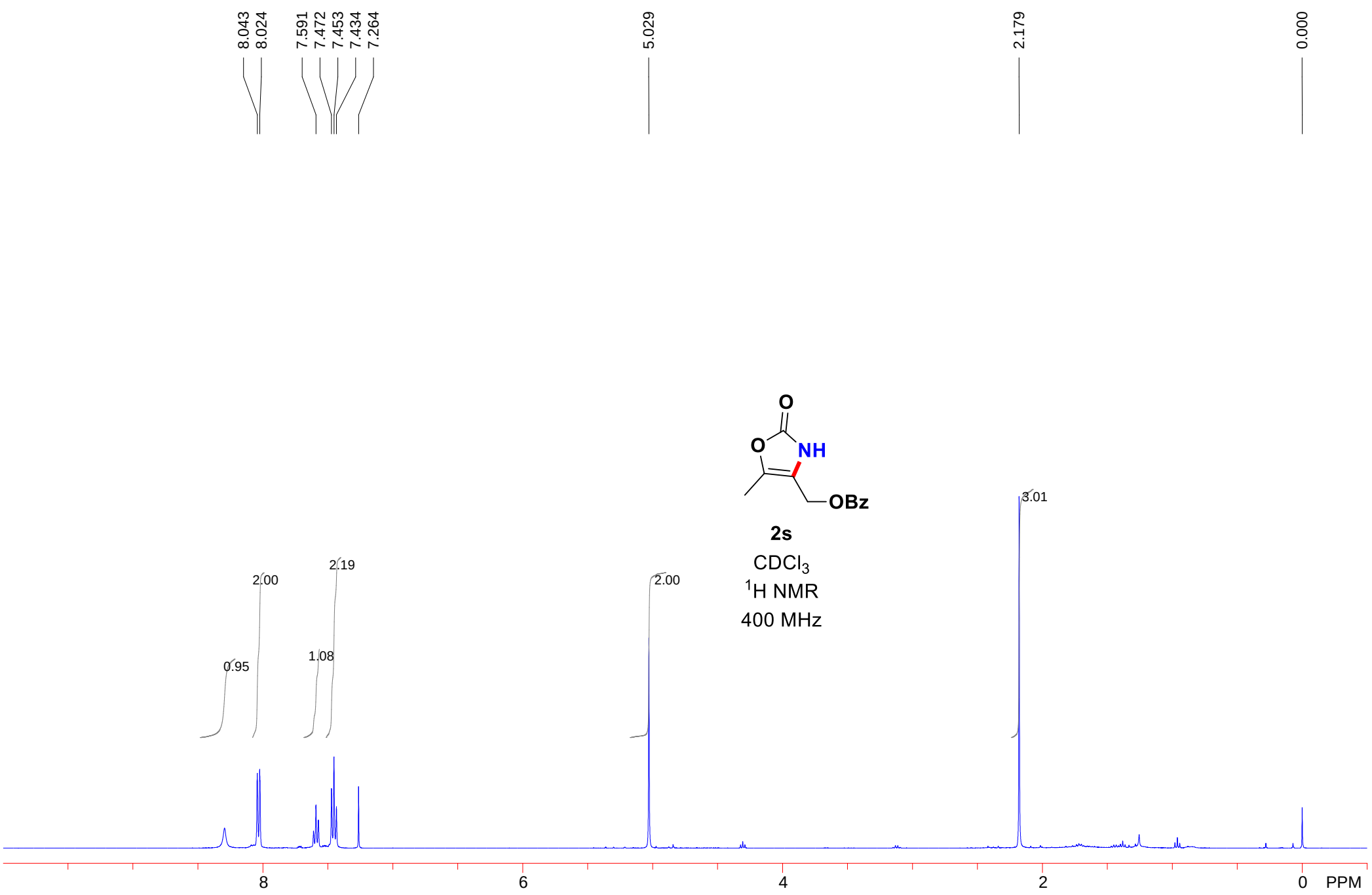
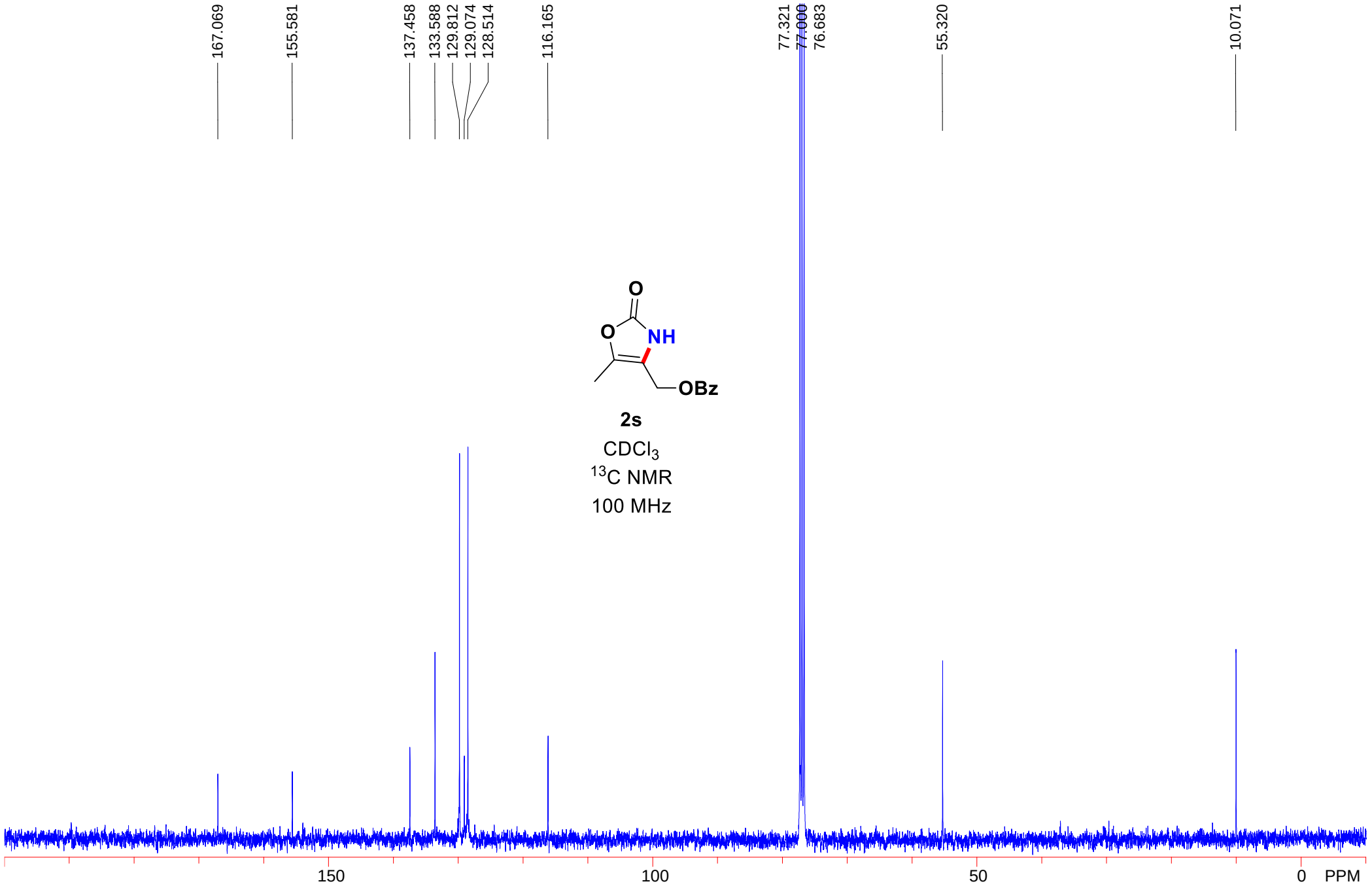
3.07
0.90
1.00
8
6
4
2
0
PPM


8.043
8.024
7.591
7.472
7.453
7.434
7.264
5.029
2.179
0.000
O
O
NH
OBz
2s
CDCl3
1H NMR
400 MHz
0.95
2.00
1.08
2.19
2.00
3.01
8
6
4
2
0
PPM


167.069
155.581
137.458
133.588
129.812
129.074
128.514
116.165
77.321
77.000
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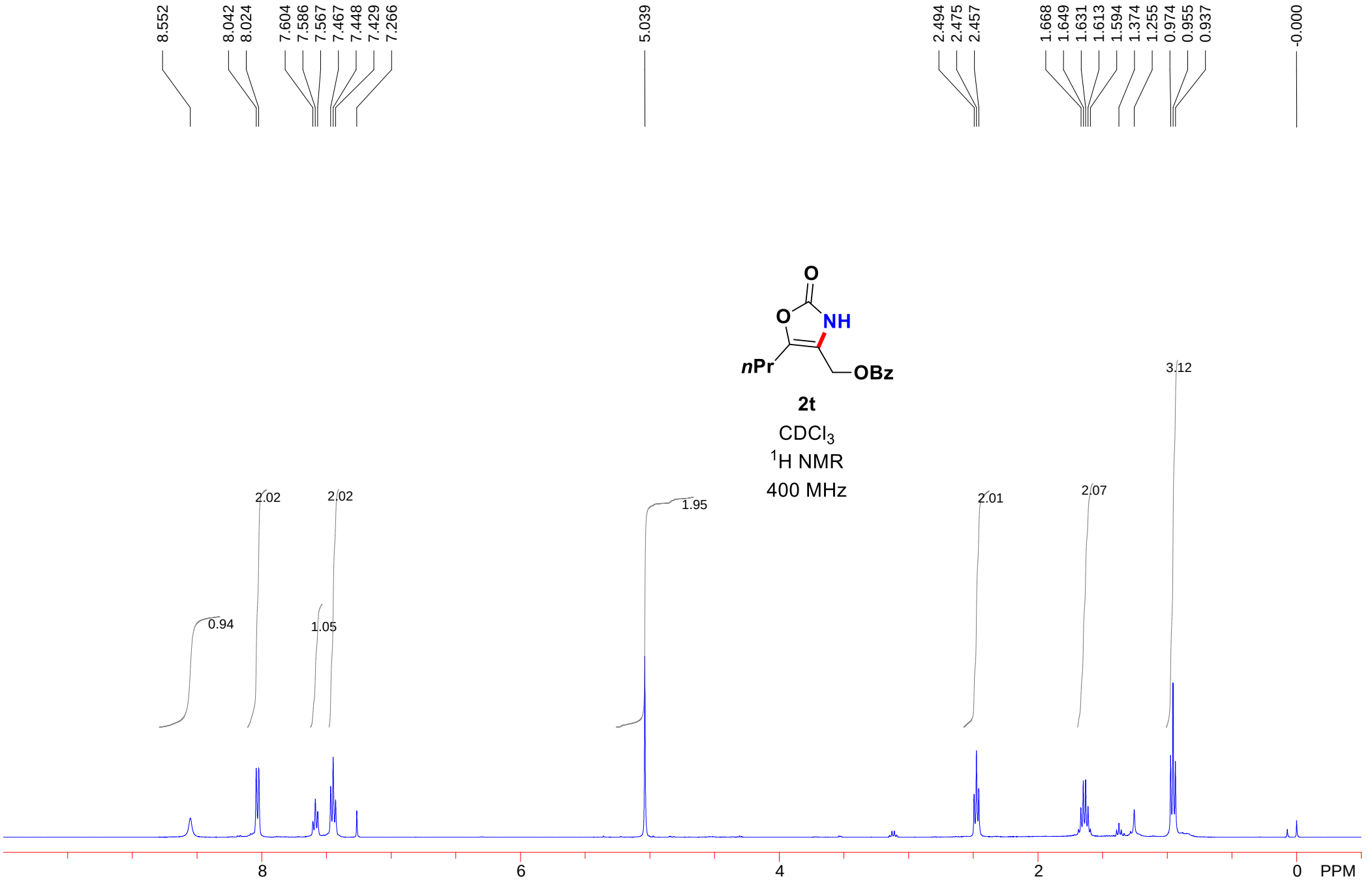
10.071
O
O
NH
OBz
2s
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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7.448
7.429
7.266
5.039
2.494
2.475
2.457
1.668
1.649
1.631
1.613
1.594
1.374
1.255
0.974
0.955
0.937
-0.000
O
O
NH
nPr
OBz
2t
CDCl3
1H NMR
400 MHz
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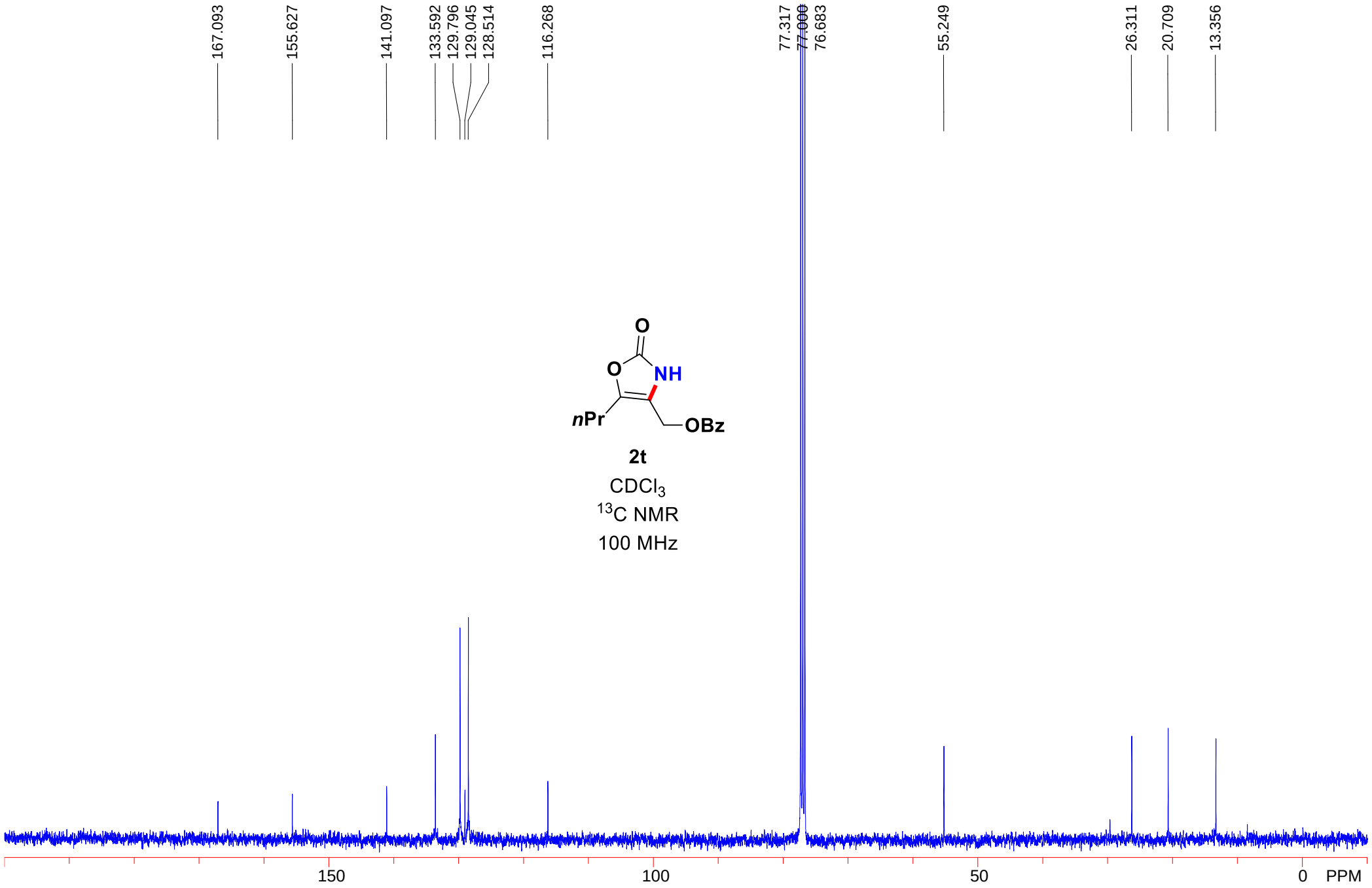
1.05
2.02
1.95
2.01
2.07
3.12
8
6
4
2
0
PPM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128.514
116.268
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77.000
76.683
55.249
26.311
20.709
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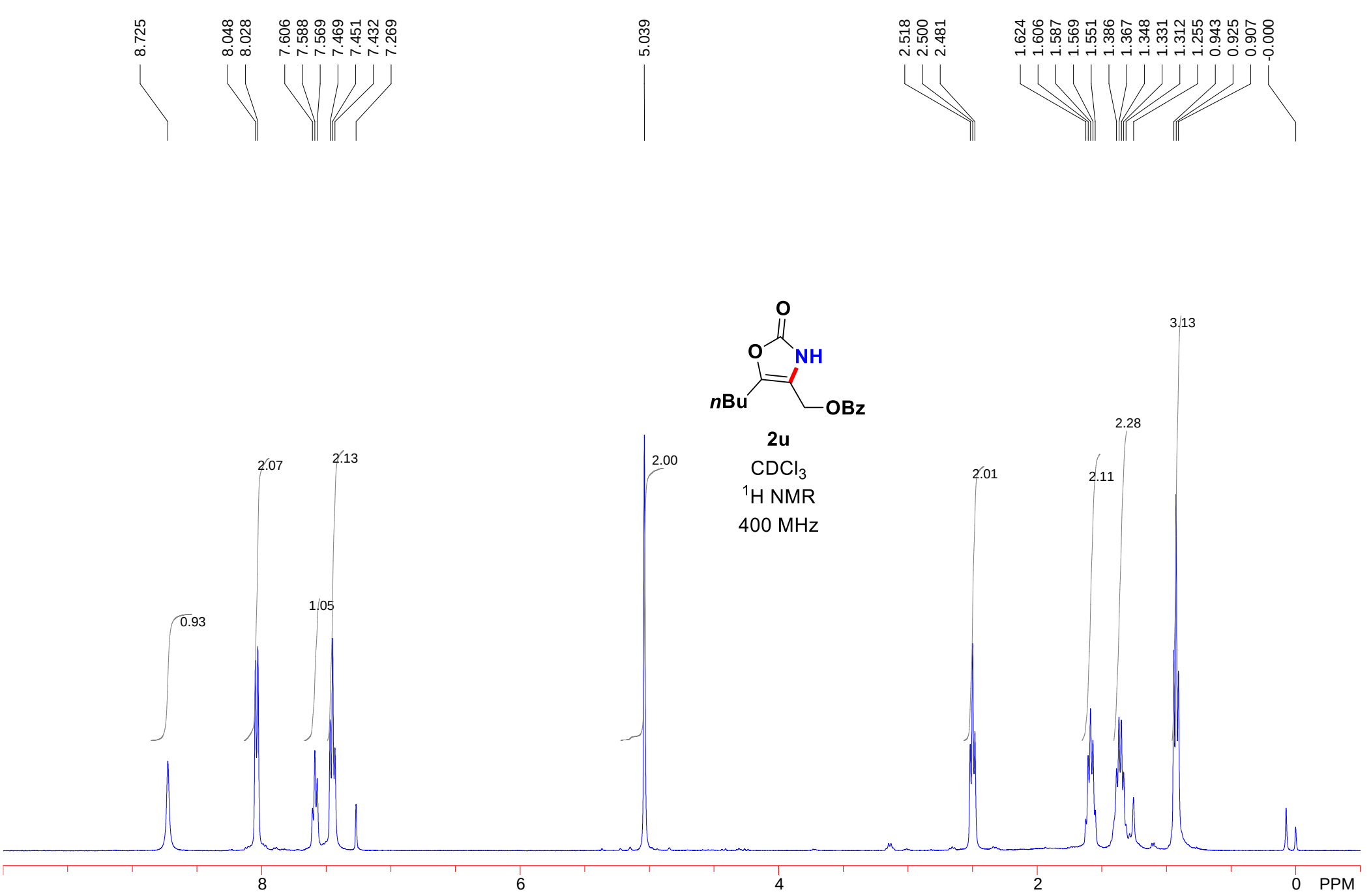
O
NH
nPr
OBz
2t
CDCl3
13C NMR
100 MHz
150
100
50
0
PPM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7.451
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7.269
5.039
2.518
2.500
2.481
1.624
1.606
1.587
1.569
1.551
1.386
1.367
1.348
1.331
1.312
1.255
0.943
0.925
0.907
-0.000
O
O
NH
nBu
OBz
2u
CDCl3
1H NMR
400 MHz
0.93
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6
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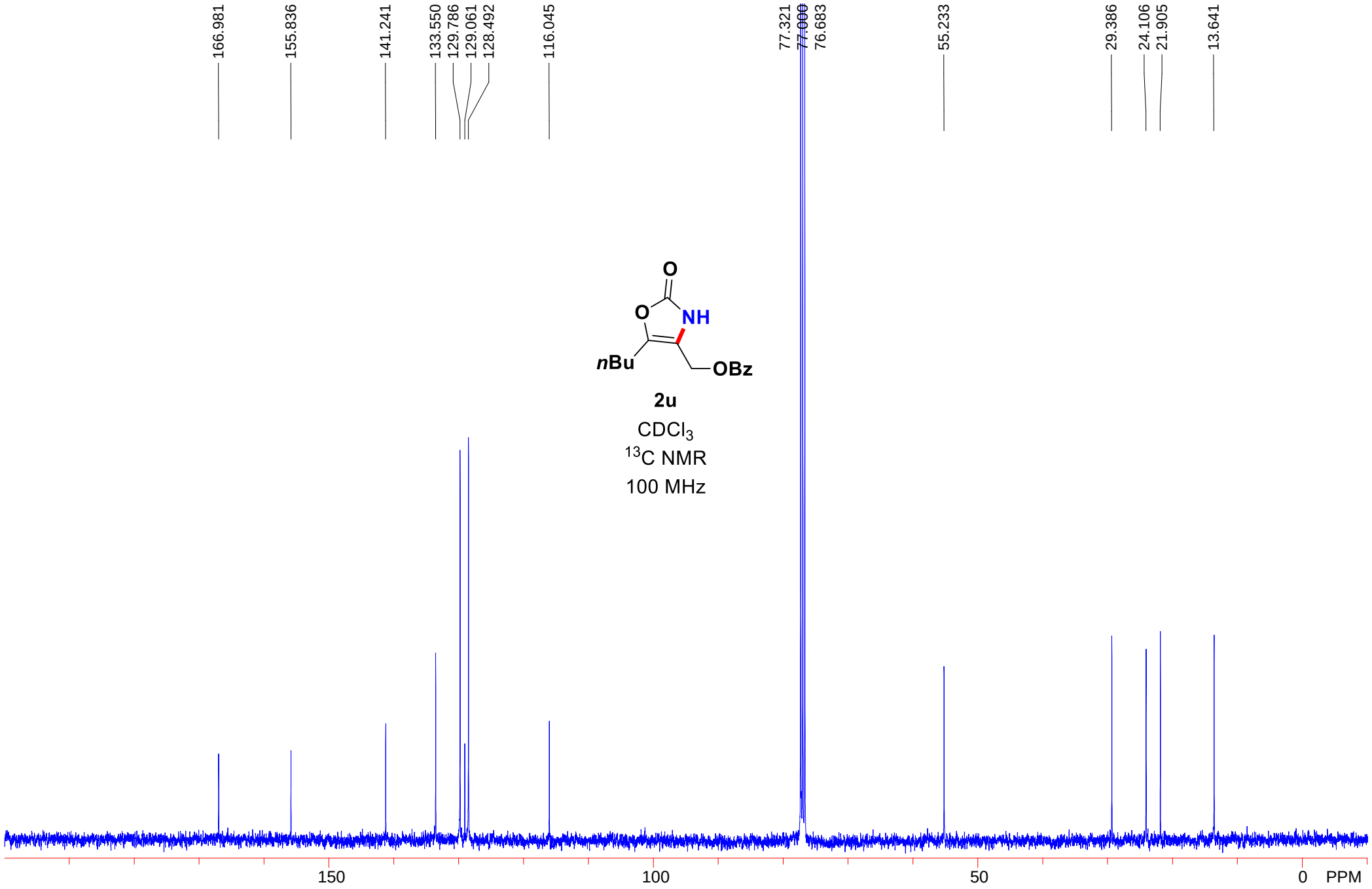
PPM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77.000
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24.106
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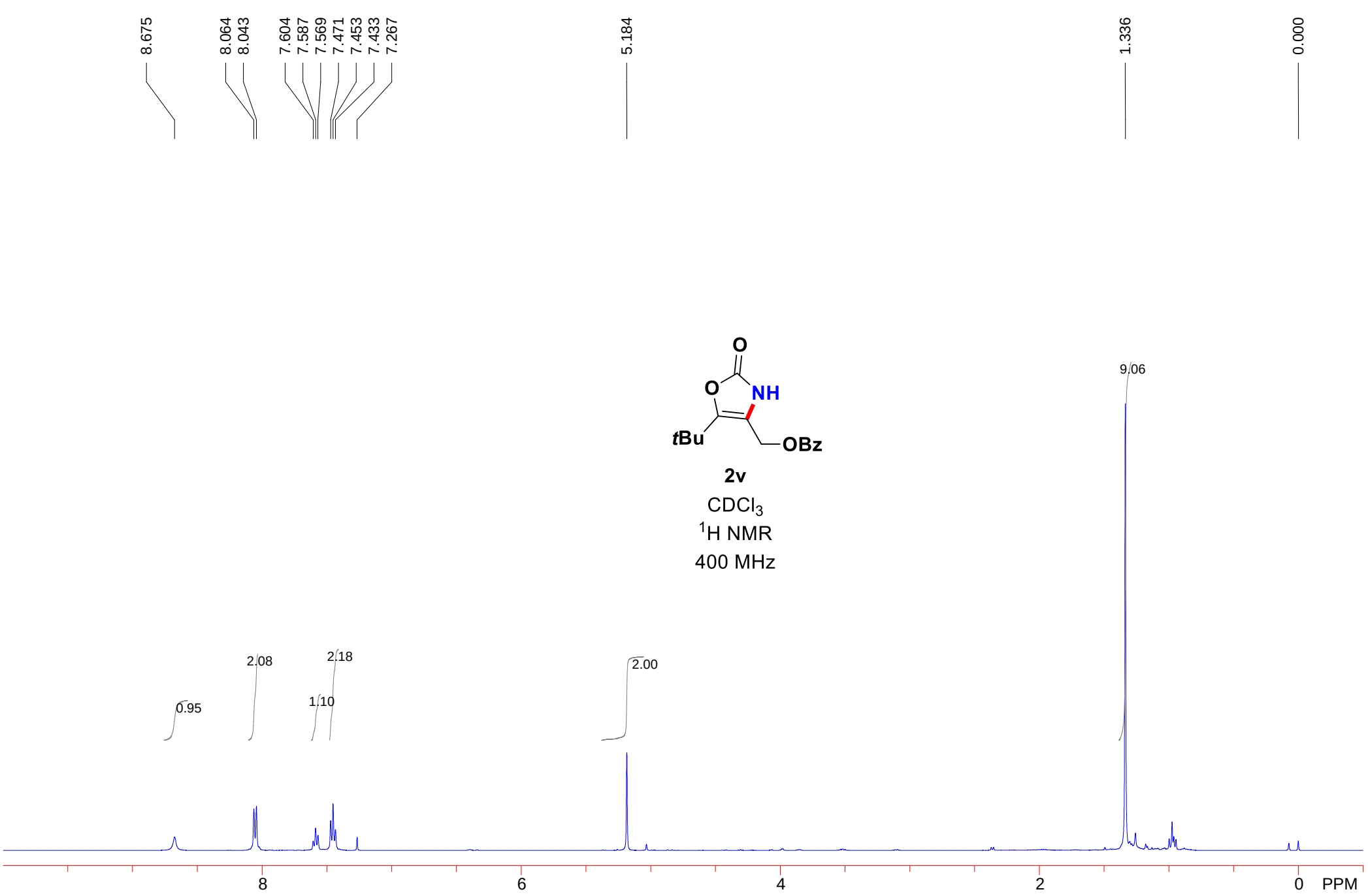
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0.000
O
O
NH
tBu
OBz
2v
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1.10
8
6
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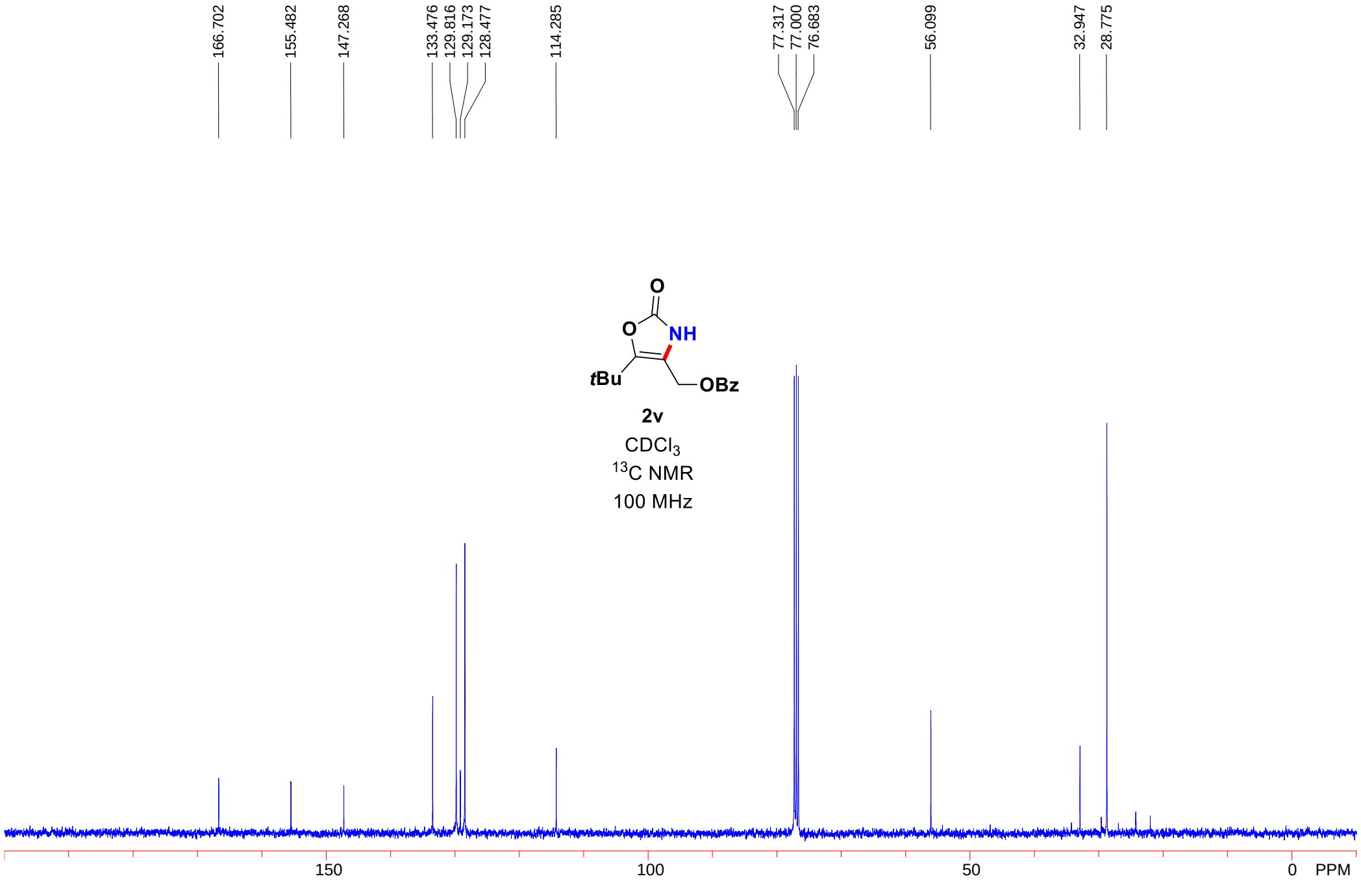
PPM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77.000
76.683
56.099
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O
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13C NMR
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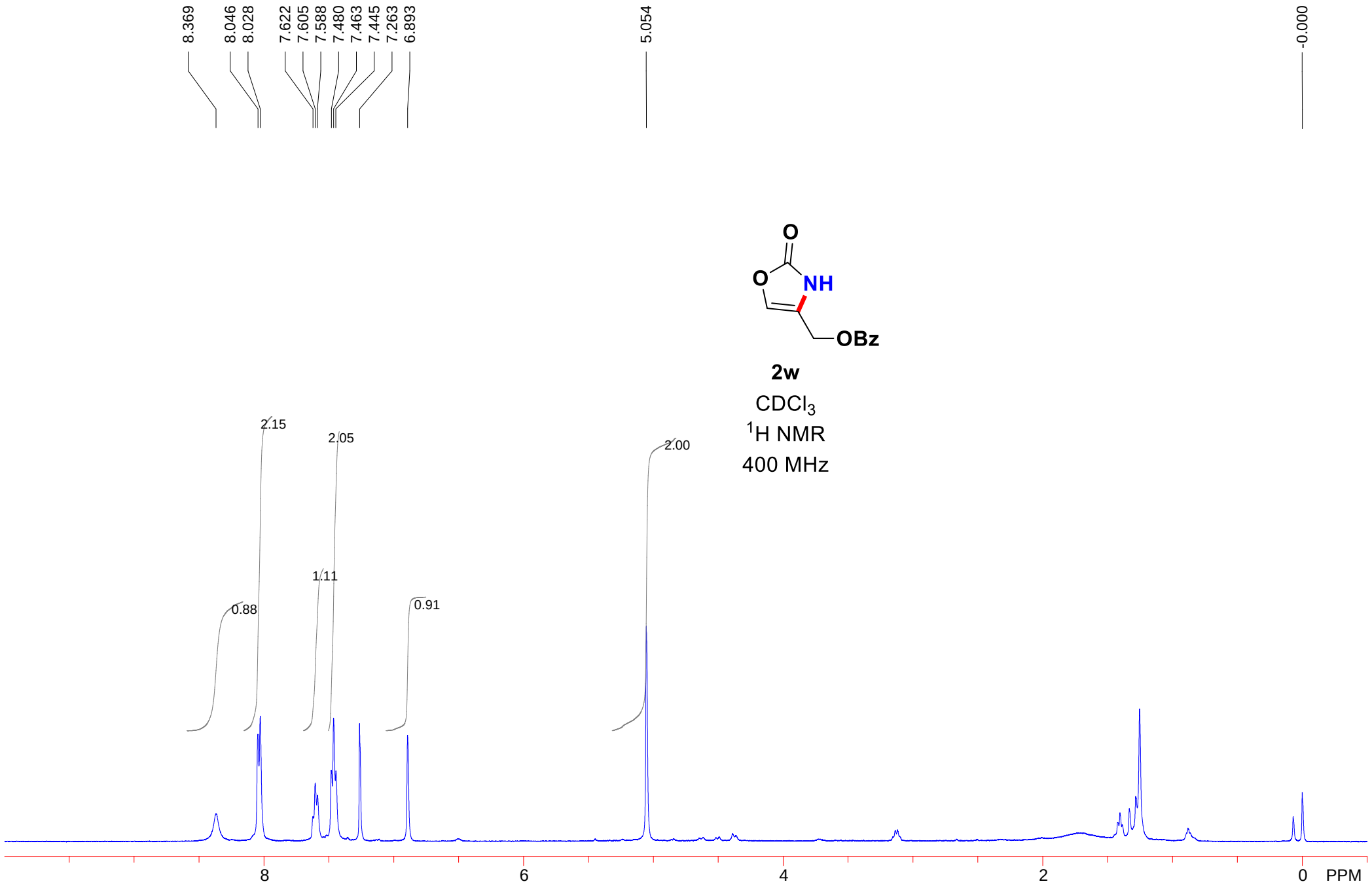
0
PPM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7.263
6.893
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-0.000
O
O
NH
OBz
2w
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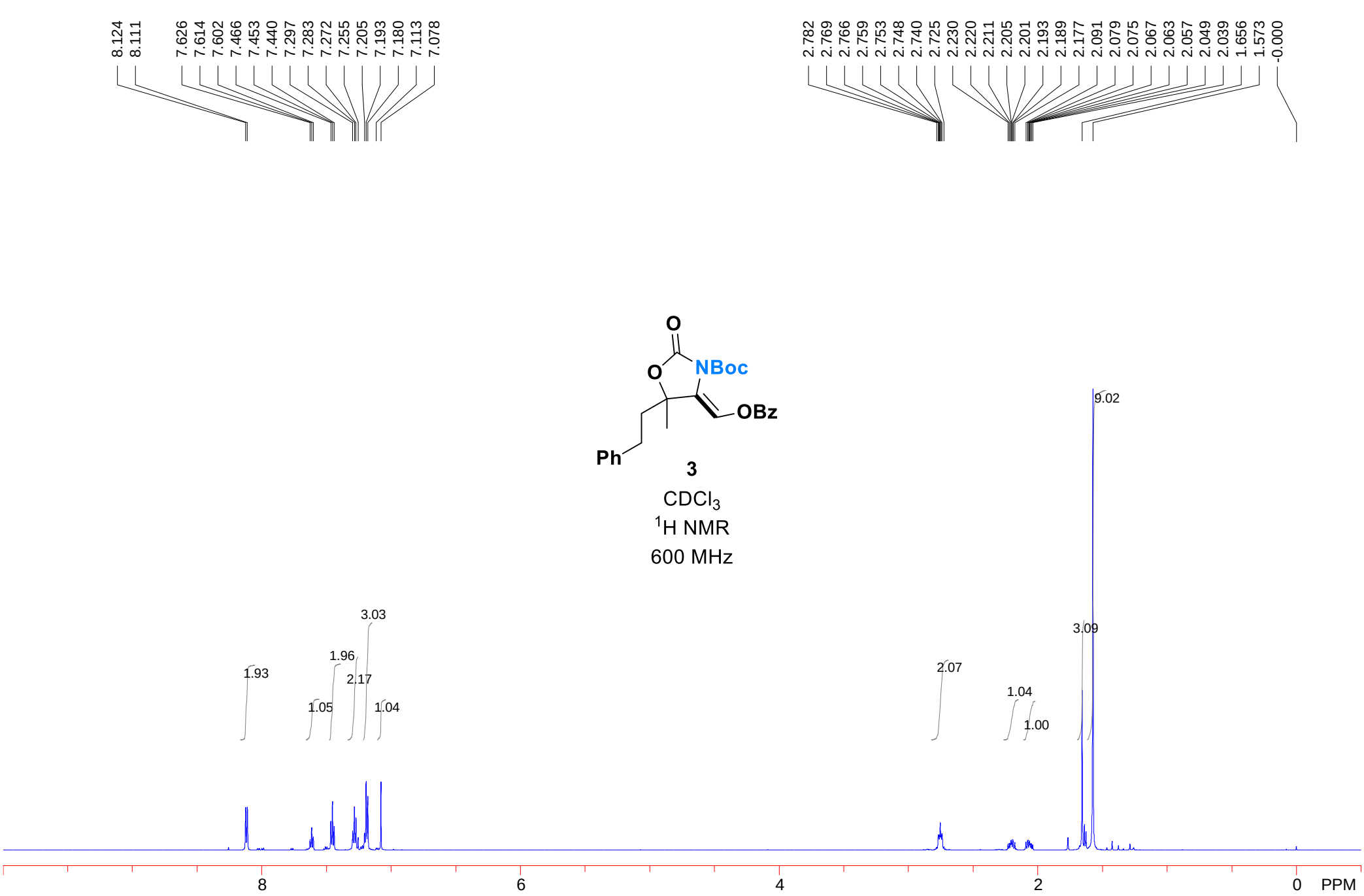
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7.180
7.113
7.078
2.782
2.769
2.766
2.759
2.753
2.748
2.740
2.725
2.230
2.220
2.211
2.205
2.201
2.193
2.189
2.177
2.091
2.079
2.075
2.067
2.063
2.057
2.049
2.039
1.656
1.573
-0.000
O
O
NBoc
OBz
Ph
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CDCl3
1H NMR
600 MHz
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1.96
1.93
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1.00
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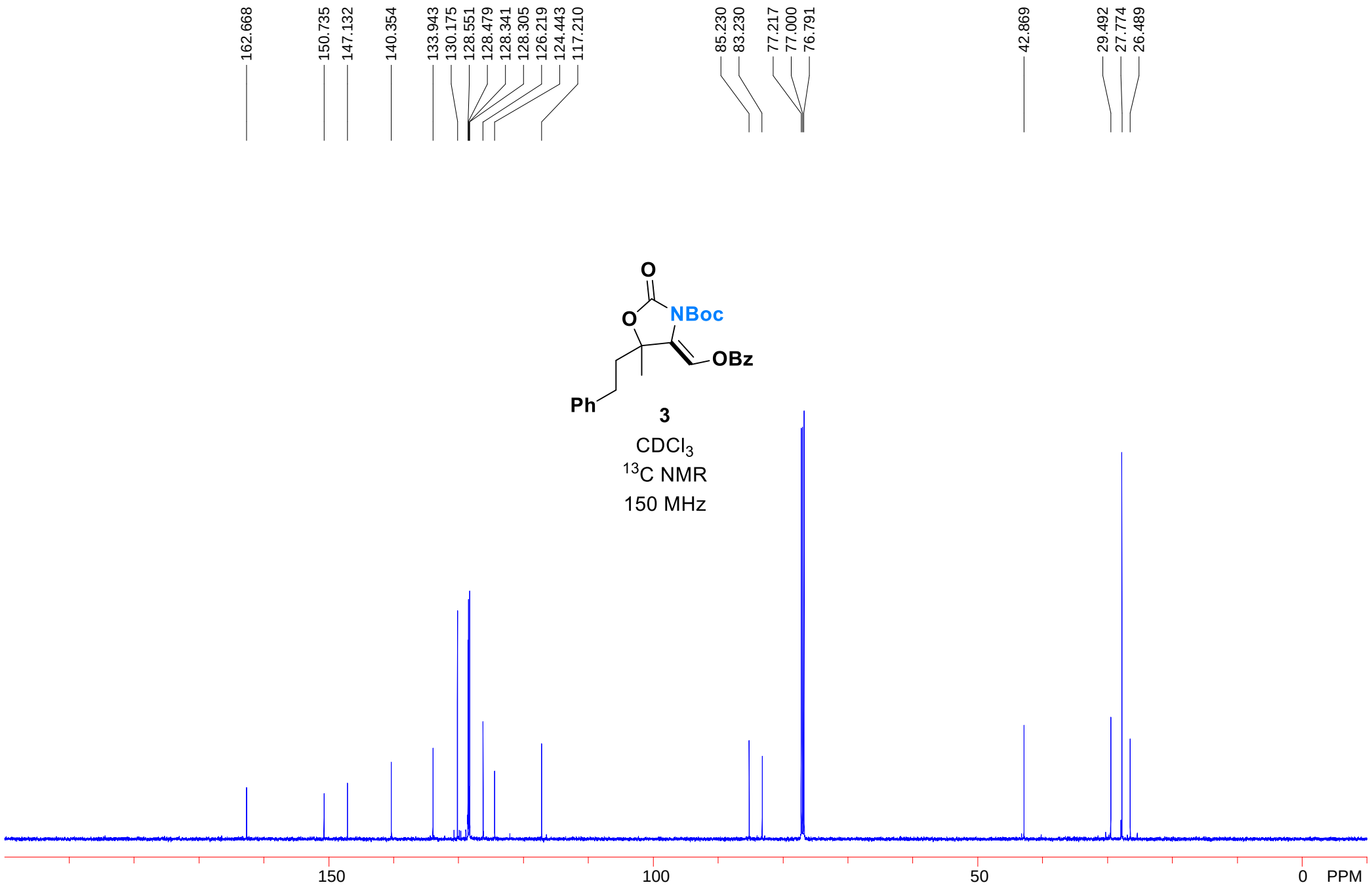
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140.354
133.943
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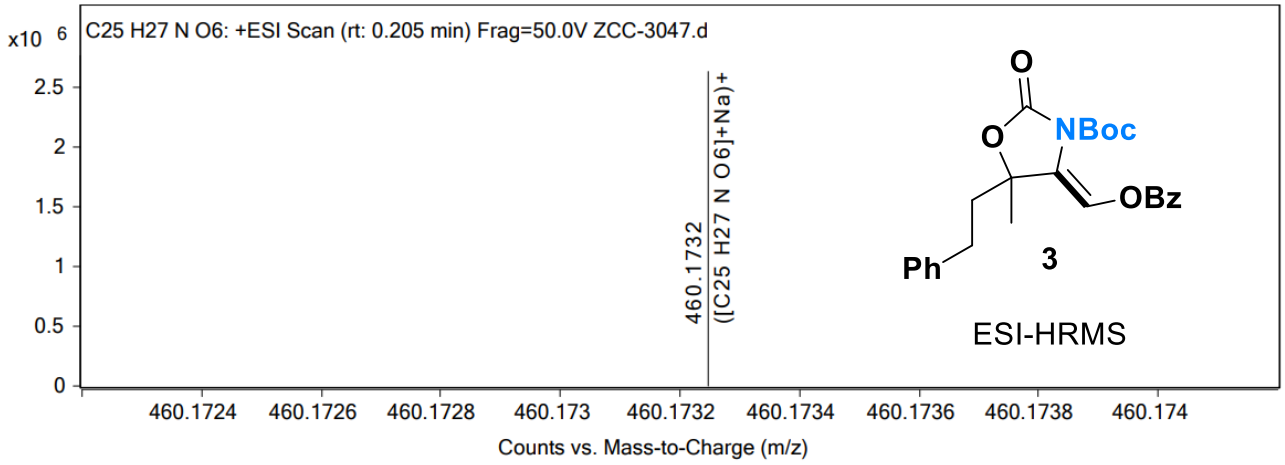
128.551
128.479
128.341
128.305
126.219
124.443
117.210
85.230
83.230
77.217
77.000
76.791
42.869
29.492
27.774
26.489
O
O
NBoc
OBz
Ph
3
CDCl3
13C NMR
150 MHz
150
100
50
0
PPM

Qualitative Analysis Report

Data File	ZCC-3047.d	**Sample Name**	Sample48
Sample Type	Sample	**Position**	P2-E4
Instrument Name	Instrument 1	**User Name**	
Acq Method	a3-50V-100%CH3OH.m	**Acquired Time**	11/22/2024 12:02:31 PM (UTC+08:00)
DA Method	ZJU.m	**Comment**	
Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition Time (Local)**	11/22/2024 12:02:31 PM (UTC+08:00)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.09.00 (B9044.0)	**QTOF Driver Version**	8.00.00
QTOF Firmware Version	25.723	**Tune Mass Range Max.**	3200

Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
50	0	ESI



Formula Calculator Element Limits

Element	Min	Max
C	0	100
H	0	100
O	0	6
N	0	5

Formula Calculator Results

Formula	Best	Measured Mass	Tgt Mass	Diff (ppm)	Score
C25 H27 N Na O6	True	460.1732	460.1731	-0.48	99.79

--- End Of Report ---