

Supplementary information for

A Copper Catalyzed Azidation and Peroxidation of β -Naphthol Derivatives via Dearomatization Strategy

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Contents

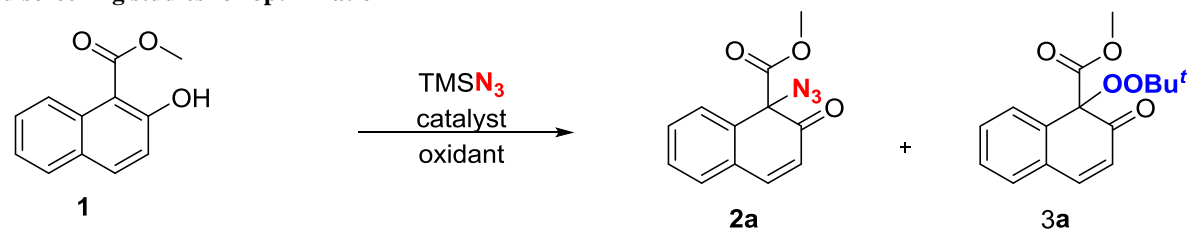
1	General Experimental	3
2	Table 1- Detailed screening studies for optimization	4
3	Tentative Mechanistic Proposal	5
4	Typical Experimental procedure for synthesis of azido compounds	6
5	Typical Experimental procedure for synthesis of peroxy compounds	6
6	Characterization data for azido compounds	7
7	Characterization data for peroxy compounds	11
8	References	15
8	^1H and ^{13}C NMR	16

General experimental

NMR spectra were recorded on 400 MHz spectrometer in CDCl₃, Tetramethylsilane (TMS; $\delta = 0.00$ ppm) served as an internal standard for ¹H NMR. The corresponding residual non-deuterated solvent signal (CDCl₃; $\delta = 77.00$ ppm) was used as internal standard for ¹³C NMR. IR spectra were measured using a FT/IR-410. Mass spectra were measured with Micromass Q-ToF (ESI-HRMS). Column chromatography was carried out on Silica gel 230-400 mesh (commercial suppliers) and thin-layer chromatography was carried out using SILICA GEL GF-254. For column purification, automatic flash column was also used. Melting points were measured using open glass capillary and values are uncorrected. Enantiomeric ratios were determined by HPLC analysis by using stationary phase chiral columns (25 cm \times 0.46 cm) in comparison with authentic racemic materials. All the starting materials were prepared by following the literature procedure.^{1,2}

Caution!!!!

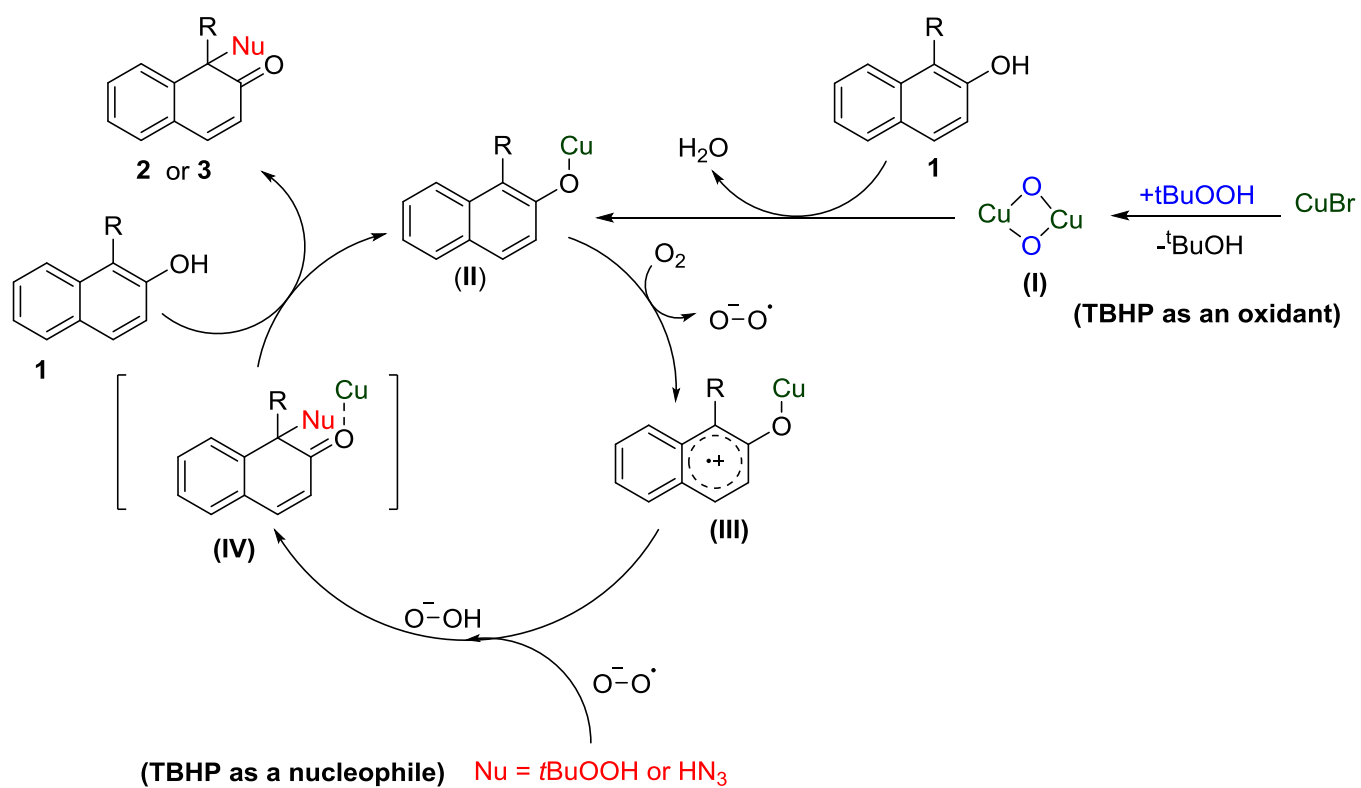
Proper safety precautions should be followed. This reaction should be carried out behind a blast shield and only on a small scale. It should be noted that while no incident occurred during this study, azides are potentially hazardous compounds and adequate safety measures should be taken.³

Table 1. Detailed screening studies for optimization^a

Entry	Catalyst (5 mol%)	Oxidant (1 equiv)	Solvent (1 mL)	Yield (%) ^b	
				2a	3a
1 ^c	TBAI	aq TBHP	1,4-dioxane	ND	
2	CuBr	aq TBHP	1,4-dioxane	18	18
3	CuBr	aq TBHP	MeOH	56	ND
4	CuBr	aq TBHP	CH ₃ CN	84	7
5	CuBr	aq TBHP	Toluene	ND	10
6	CuBr	aq TBHP	EtOAc	25	10
7	CuBr	aq TBHP	H ₂ O	5	ND
8	CuBr	DBP	CH ₃ CN	63	ND
9	CuBr	O ₂	CH ₃ CN	ND	
10	CuBr	DTBP	CH ₃ CN	ND	
11	CuBr	aq H ₂ O ₂	CH ₃ CN	ND	
12	CuBr	DCP	CH ₃ CN	ND	
13	CuBr ₂	aq TBHP	CH ₃ CN	86	14
14	CuCl	aq TBHP	CH ₃ CN	76	23
15	CuI	aq TBHP	CH ₃ CN	11	ND
16	Cu(OAc) ₂	aq TBHP	CH ₃ CN	8	ND
17	NiCl ₂ ·6H ₂ O	aq TBHP	CH ₃ CN	trace	67
18	FeCl ₃ ·6H ₂ O	aq TBHP	CH ₃ CN	19	59
19	CoCl ₂ ·6H ₂ O	aq TBHP	CH ₃ CN	16	48
20 ^d	CuBr	aq TBHP	CH ₃ CN	trace	92
21 ^e	CuBr	aq TBHP	CH ₃ CN	95	5
22 ^f	CuBr	aq TBHP	CH ₃ CN	97	3
23	none	aq TBHP	CH ₃ CN	ND	
24	CuBr	none	CH ₃ CN	ND	
25 ^g	CuBr	aq. TBHP	CH ₃ CN	ND	98

^a Reaction conditions: **1a** (0.25 mmol), TMSN₃ (0.5 mmol), aq. TBHP (70% in water, 1 equiv.) CuBr (5 mol%), CH₃CN (1 mL), at rt, 24 h. ^b NMR yields, calculated using terephthalaldehyde as an internal standard, ^c TBAI (10 mol %), ^d NaN₃ was used instead of TMSN₃, ^e 0.5 equiv of aq TBHP was used, ^f 0.25 equiv. of aq. TBHP was used, ^g TMSN₃ was not used. ND = not detected.

Scheme 1. Tentative mechanistic proposal for azidation reaction



Typical experimental procedure

(a) For the synthesis of azido compounds

TMSN₃ (2 equiv, 0.50mmol) was added to a well-stirred solution of β-naphthol derivative (0.25 mmol), CuBr (0.05 equiv, 0.013 mol) in acetonitrile (1 mL), after that, aq TBHP (70% in water) (0.25 equiv, 0.06 mmol, 8 μL) was added dropwise to the reaction mixture and it was stirred at room temperature for 3-24 h (monitored by TLC). After completion of the reaction (monitored by TLC) reaction mixture was passed through the short pad of Celite and extracted with 50% ethyl acetate and pet ether (3x20mL). The combined organic layer was dried over anhyNa₂SO₄ and concentrated under reduced pressure. The crude product was purified on a silica gel flash column chromatography using hexane/ EtOAc to get the pure product.

(b) For the synthesis of peroxy compounds

Aq TBHP (70% in water, 1 equiv, 0.25 mmol, 35 μL) was added to a well-stirred solution of β -naphthol derivative (0.25 mmol), CuBr (0.05 equiv, 0.013 mol) in acetonitrile (1 mL), after that, the reaction mixture was stirred at room temperature for 12-24 h. After completion of the reaction (monitored by TLC) reaction mixture was passed through the short pad of Celite and extracted with 50% ethyl acetate and pet ether (3x20 mL). The combined organic layer was dried over anhy Na₂SO₄ and concentrated under reduced pressure. The crude product was purified on a silica gel flash column chromatography using hexane/ EtOAc to get the pure product.

(c) Typical experimental procedure for the synthesis of compounds (4a and 4j)

Azide **2a/2j** (0.2mmol) was dissolved in a 2 mL of methanol. Sodium borohydride (1 equiv, 0.4 mmol) was added to the solution and was stirred at room temperature for 5 min. The reaction mixture was quenched using water 5 mL and the solvent was evaporated and water (15 mL) was added to the reaction mixture and extracted with CH₂Cl₂ (3×20 mL). The combined organic phases were dried over anhyNa₂SO₄ and the solvent was evaporated. The residue was purified by flash chromatography on silica using hexane/ EtOAc to get the pure product.

(d) Typical experimental procedure for the synthesis of compound (5a)

The compound **3a** (0.2mmol) was dissolved in methanol (2 mL). Then was added Pd/C (5 mg) to the solution and the reaction mixture was stirred at room temperature for 5 h. The reaction mixture was passed through aCelitepad and extracted using 50% ethyl acetate and pet ether. The combined organic phases were dried over anhyNa₂SO₄ and the solvent was evaporated. The residue was purified by flash chromatography on silica using hexane/ EtOAc to get the pure product.

(e) Typical experimental procedure for the synthesis of compounds (5j)

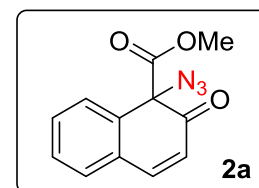
The compound **2j** (0.2mmol) was dissolved in methanol (2 mL). Then was added Pd/C (5 mg) to the solution and the reaction mixture was stirred at room temperature for 5 h. The reaction mixture was passed through a Celite and extracted using 50% ethyl acetate and pet ether. The combined organic phases were dried over anhyNa₂SO₄ and the solvent was evaporated. The residue was purified by flash chromatography on silica using hexane/ EtOAc to get the pure product.

(f) Typical experimental procedure for the synthesis of compounds (6j)

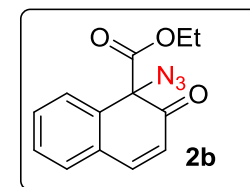
The compound **2j** (0.2 mmol) was dissolved in methanol (2 mL). And was added sodium hydroxide (2 equiv, 0.4 mmol) and aq H₂O₂ (50% in water, 3 equiv) drop wise to the reaction mixture at 0 °C. The reaction mixture was diluted with water and extracted with ethyl acetate (3x20 mL). The combined organic phases were dried over anhydrous Na₂SO₄ and the solvent was evaporated. The residue was purified by flash chromatography on silica using hexane/ EtOAc to get the pure product.

Characterization data for quaternary azidation of β -naphthol derivatives

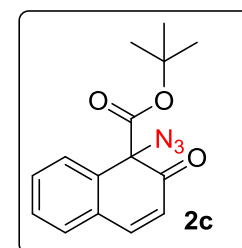
Methyl 1-azido-2-oxo-1,2-dihydronaphthalene-1-carboxylate (2a): Brown solid; Yield - 95%; *mp*: 78°-80°C; R_f(20% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (a). **IR** (neat, cm⁻¹): 2110, 1752, 1670, 1129; **¹H NMR** (400 MHz, CDCl₃): δ 3.70 (s, 3 H) 6.24 (d, *J*=9.77 Hz, 1 H) 7.32 - 7.39 (m, 1 H) 7.39 - 7.55 (m, 4 H); **¹³C NMR** (100 MHz, CDCl₃): δ 53.73, 70.36, 76.68, 77.32, 123.37, 128.24, 129.08, 129.80, 129.99, 130.90, 135.07, 146.75, 167.29, 193.08; **HRESI-MS**(*m/z*): Calculated for C₁₂H₉N₃O₃ (M + Na): 266.0542, found (M + Na): 266.0541.



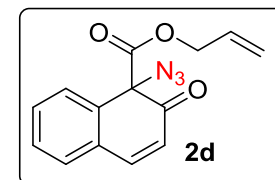
Ethyl 1-azido-2-oxo-1,2-dihydronaphthalene-1-carboxylate (2b): Brown liquid; Yield - 78%; R_f (10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (a). **IR** (neat, cm⁻¹): 2114, 1749, 1671, 1214; **¹H NMR** (400 MHz, CDCl₃): δ 1.14 (t, *J*=7.17 Hz, 3 H) 4.01 - 4.28 (m, 2 H) 6.24 (d, *J*=10.07 Hz, 1 H) 7.32 - 7.38 (m, 1 H) 7.38 - 7.47 (m, 2 H) 7.47 - 7.53 (m, 2 H); **¹³C NMR** (100 MHz, CDCl₃): δ 13.74, 63.14, 70.49, 76.69, 77.32, 123.48, 128.18, 129.17, 129.71, 129.94, 130.85, 135.34, 146.65, 166.73, 193.33; **HRESI-MS**(*m/z*): Calculated for C₁₃H₁₁N₃O₃ (M + Na): 280.0698, found (M + Na): 280.0697.



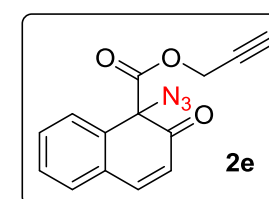
***tert*-Butyl 1-azido-2-oxo-1,2-dihydronaphthalene-1-carboxylate (2c):** Yellow oil; Yield - 91%; R_f (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (a). **IR** (neat, cm⁻¹): 2981, 2116, 1745, 1672; **¹H NMR** (400 MHz, CDCl₃): δ 1.33 (s, 9 H) 6.21 (d, *J*=10.07 Hz, 1 H) 7.30 - 7.37 (m, 1 H) 7.37 - 7.46 (m, 2 H) 7.46 - 7.52 (m, 2 H); **¹³C NMR** (100 MHz, CDCl₃): δ 27.46, 70.94, 76.69, 77.32, 84.67, 123.43, 127.70, 129.16, 129.40, 129.76, 130.62, 135.80, 146.41, 165.24, 193.86; **HRESI-MS**(*m/z*): Calculated for C₁₅H₁₅N₃O₃ (M + Na): 308.1011, found (M + Na): 308.1012.



Allyl 1-azido-2-oxo-1,2-dihydronaphthalene-1-carboxylate (2d): Black oil; Yield - 72%; R_f(10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (a). **IR** (neat, cm⁻¹): 3555, 2112, 1750, 1670; **¹H NMR** (400 MHz, CDCl₃): δ 4.60 (d, *J*=5.49 Hz, 2 H) 5.00 - 5.23 (m, 2 H) 5.72 (ddt, *J*=16.78, 10.99, 5.34, 5.34 Hz, 1 H) 6.25 (d, *J*=10.07 Hz, 1 H) 7.32 - 7.39 (m, 1 H) 7.39 - 7.47 (m, 2 H) 7.47 - 7.56 (m, 2 H); **¹³C NMR** (100 MHz, CDCl₃): δ ppm 67.04, 70.48, 76.69, 77.32, 118.70, 123.46, 128.26, 129.16, 129.79, 129.98, 130.50, 130.88, 135.19, 146.73, 166.43, 193.18; **HRESI-MS**(*m/z*): Calculated for C₁₄H₁₁N₃O₃ (M + Na): 292.0698, found (M + Na): 292.0698.



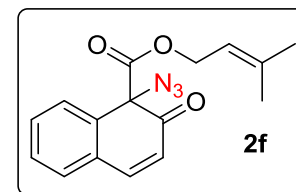
Prop-2-yn-1-yl 1-azido-2-oxo-1,2-dihydronaphthalene-1-carboxylate (2e): Black solid; Yield - 79%; *mp*: 92°-93°C; R_f (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (a). **IR** (neat, cm⁻¹): 3068, 2112, 1755, 1427; **¹H NMR** (400 MHz, CDCl₃): δ 2.43 (t, *J*=2.44 Hz, 1 H) 4.54 - 4.80 (m, 2 H) 6.25 (d, *J*=9.77 Hz, 1



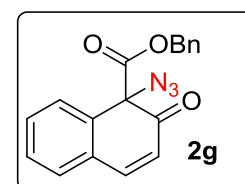
H) 7.34 - 7.40 (m, 1 H) 7.40 - 7.50 (m, 3 H) 7.52 (d, $J=10.07$ Hz, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 54.07, 70.27, 75.88, 76.05, 76.68, 77.31, 123.33, 128.33, 129.11, 129.95, 130.10, 130.93, 134.65, 146.88, 166.14, 192.47; **HRESI-MS**(m/z): Calculated for $\text{C}_{14}\text{H}_9\text{N}_3\text{O}_3$ ($M + \text{Na}$): 290.0542, found ($M + \text{Na}$): 290.0542.

3-Methylbut-2-en-1-yl 1-azido-2-oxo-1,2-dihydronaphthalene-1-carboxylate (2f):

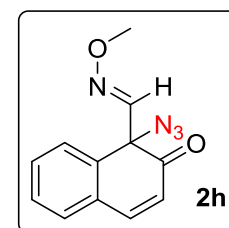
Black oil - 40%; Rf (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 3025, 2113, 1741, 1204; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 1.55 (s, 3 H) 1.66 (s, 3 H) 4.58 (d, $J=7.02$ Hz, 2 H) 5.07 - 5.19 (m, 1 H) 6.22 (d, $J=10.07$ Hz, 1 H) 7.32 - 7.37 (m, 1 H) 7.37 - 7.45 (m, 2 H) 7.45 - 7.52 (m, 2 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 17.93, 25.56, 26.35, 63.81, 70.54, 76.68, 77.31, 117.00, 123.46, 128.18, 129.16, 129.62, 129.88, 130.73, 135.32, 140.76, 146.61, 166.63, 193.31; **HRESI-MS**(m/z): Calculated for $\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_3$ ($M + \text{Na}$): 320.1011, found ($M + \text{Na}$): 320.1011.



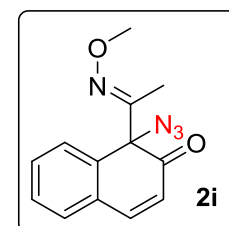
Benzyl 1-azido-2-oxo-1,2-dihydronaphthalene-1-carboxylate (2g): Dirty white solid; Yield - 81%; *mp*: 70°-72°C; Rf (10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2113, 1752, 1672, 1208; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 5.01 - 5.22 (m, 2 H) 6.22 (d, $J=9.77$ Hz, 1 H) 6.97 - 7.09 (m, 2 H) 7.18 - 7.30 (m, 3 H) 7.30 - 7.53 (m, 5 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 68.25, 70.55, 76.69, 77.32, 123.44, 127.33, 127.46, 128.24, 128.34, 128.44, 129.16, 129.73, 129.97, 130.83, 134.48, 135.05, 146.75, 166.53, 193.09; **HRESI-MS**(m/z): Calculated for $\text{C}_{18}\text{H}_{13}\text{N}_3\text{O}_3$ ($M + \text{Na}$): 342.0855, found ($M + \text{Na}$): 342.0851.



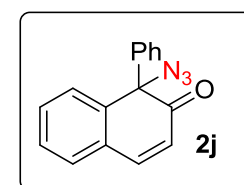
(E)-1-azido-2-oxo-1,2-dihydronaphthalene-1-carbaldehyde O-methyl oxime (2h): White solid; Yield - 58%; *mp*: 93°-95°C; Rf (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2108, 1689, 1033; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 3.85 (s, 3 H) 6.24 (d, $J=9.77$ Hz, 1 H) 7.29 (s, 1 H) 7.31 - 7.36 (m, 1 H) 7.36 - 7.49 (m, 3 H) 7.59 (d, $J=7.63$ Hz, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 62.59, 69.03, 76.68, 77.31, 124.14, 128.19, 129.37, 129.51, 129.81, 130.68, 136.55, 144.79, 146.79, 195.23; **HRESI-MS**(m/z): Calculated for $\text{C}_{12}\text{H}_{10}\text{N}_4\text{O}_2$ ($M + \text{Na}$): 265.0701, found ($M + \text{Na}$): 265.0701.



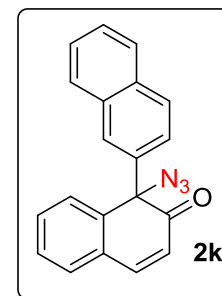
(E)-1-azido-1-(1-(methoxyimino)ethyl)naphthalen-2(1H)-one (2i): Black oil; Yield - 91%; Rf (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2923, 2107, 1668; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 1.63 (s, 3 H) 3.94 (s, 3 H) 6.24 (d, $J=9.77$ Hz, 1 H) 7.31 - 7.36 (m, 1 H) 7.36 - 7.47 (m, 3 H) 7.53 (d, $J=7.63$ Hz, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 11.85, 62.45, 71.20, 76.68, 77.31, 124.56, 128.73, 129.43, 129.81, 129.88, 130.36, 136.54, 144.65, 153.12, 195.20; **HRESI-MS**(m/z): Calculated for $\text{C}_{13}\text{H}_{12}\text{N}_4\text{O}_2$ ($M + \text{Na}$): 279.0858, found ($M + \text{Na}$): 279.0858.



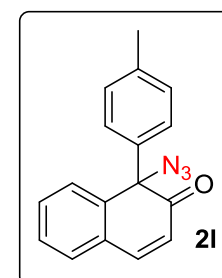
1-Azido-1-phenylnaphthalen-2(1H)-one (2j): Yellow oil; Yield - 71%; Rf (10% EtOAc/Hexane) 0.6; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2108, 1679, 1446, 1235; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 6.15 (d, $J=10.07$ Hz, 1 H) 7.10 - 7.23 (m, 2 H) 7.23 - 7.32 (m, 3 H) 7.34 - 7.50 (m, 4 H) 7.52 - 7.59 (m, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 74.32, 76.68, 77.32, 124.06, 126.79, 128.70, 128.74, 129.07, 129.14, 129.65, 130.10, 130.64, 137.64, 140.35, 144.95, 196.99; **HRESI-MS**(m/z): Calculated for $\text{C}_{16}\text{H}_{11}\text{N}_3\text{O}$ ($M + \text{Na}$): 284.0800, found ($M + \text{Na}$): 284.0808.



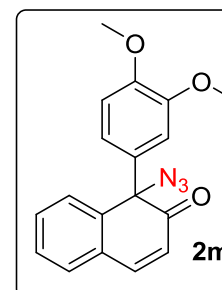
1-Azido-[1,2'-binaphthalen]-2(1H)-one (2k): Brown oil; Yield – 64%; Rf (10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2107, 1677, 1238; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 6.19 (d, $J=10.07$ Hz, 1 H) 7.21 - 7.31 (m, 1 H) 7.37 - 7.53 (m, 6 H) 7.53 - 7.60 (m, 1 H) 7.64 - 7.70 (m, 1 H) 7.70 - 7.82 (m, 3 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 74.34, 76.68, 77.31, 124.13, 124.19, 126.24, 126.49, 126.82, 127.49, 128.43, 128.63, 129.20, 129.35, 129.73, 130.15, 130.75, 132.90, 133.08, 135.12, 140.35, 145.08, 196.81; **HRESI-MS**(m/z): Calculated for $\text{C}_{20}\text{H}_{13}\text{N}_3\text{O}$ (M + Na): 334.0956, found (M + Na): 334.0956.



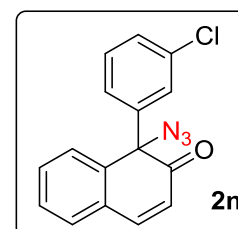
1-Azido-1-(p-tolyl)naphthalen-2(1H)-one (2l): Brown oil; Yield – 78%; Rf (10% EtOAc/Hexane) 0.6; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2108, 1680, 1197; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 2.28 (s, 3 H) 6.13 (d, $J=9.77$ Hz, 1 H) 6.98 - 7.13 (m, 4 H) 7.32 - 7.50 (m, 4 H) 7.52 - 7.60 (m, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 20.98, 74.31, 76.68, 77.31, 124.06, 126.79, 128.97, 129.03, 129.40, 129.62, 130.08, 130.53, 134.49, 138.81, 140.41, 144.75, 197.25; **HRESI-MS**(m/z): Calculated for $\text{C}_{17}\text{H}_{13}\text{N}_3\text{O}$ (M + Na): 298.0956, found (M + Na): 298.0956.



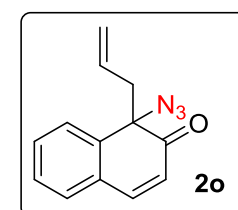
1-Azido-1-(3,4-dimethoxyphenyl)naphthalen-2(1H)-one (2m): Yellow oil; Yield – 66%; Rf (10% EtOAc/Hexane) 0.6; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2106, 1678, 1513; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 3.80 (s, 3 H) 3.82 (s, 3 H) 6.14 (d, $J=9.77$ Hz, 1 H) 6.51 (dd, $J=8.54, 2.14$ Hz, 1 H) 6.71 (d, $J=8.54$ Hz, 1 H) 6.82 (d, $J=2.44$ Hz, 1 H) 7.31 - 7.53 (m, 4 H) 7.57 - 7.66 (m, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 55.79, 55.82, 74.49, 76.69, 77.32, 110.36, 110.66, 119.60, 124.02, 129.00, 129.05, 129.38, 129.62, 130.22, 130.45, 140.21, 144.47, 148.98, 149.52, 197.08; **HRESI-MS**(m/z): Calculated for $\text{C}_{18}\text{H}_{15}\text{N}_3\text{O}_3$ (M + Na): 344.1011, found (M + Na): 344.1010.



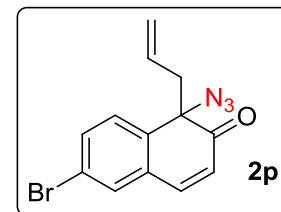
1-Azido-1-(3-chlorophenyl)naphthalen-2(1H)-one (2n): Brown oil; Yield – 65%; Rf (10% EtOAc/Hexane) 0.5; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2108, 1679; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 6.17 (d, $J=10.07$ Hz, 1 H) 7.03 - 7.12 (m, 1 H) 7.14 - 7.31 (m, 3 H) 7.37 - 7.53 (m, 5 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 73.52, 76.68, 77.31, 123.91, 124.94, 126.91, 128.89, 129.19, 129.42, 129.85, 129.94, 130.96, 134.70, 139.73, 139.86, 145.40, 196.10; **HRESI-MS**(m/z): Calculated for $\text{C}_{16}\text{H}_{10}\text{ClN}_3\text{O}$ (M + Na): 318.0410, found (M + Na): 318.0408.



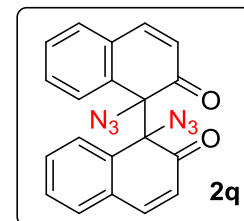
1-Allyl-1-azidonaphthalen-2(1H)-one (2o): Dirty white oil; Yield – 73%; Rf (20% EtOAc/Hexane) 0.8; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2103, 1673, 1267; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 2.69 (dd, $J=13.43, 7.32$ Hz, 1 H) 2.80 (dd, $J=13.43, 7.32$ Hz, 1 H) 4.86 - 5.14 (m, 2 H) 5.45 (ddt, $J=17.05, 9.96, 7.25, 7.25$ Hz, 1 H) 6.17 (d, $J=9.77$ Hz, 1 H) 7.29 - 7.50 (m, 4 H) 7.57 (d, $J=7.63$ Hz, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 46.15, 70.28, 76.68, 77.31, 120.47, 124.07, 127.73, 128.59, 129.57, 129.78, 130.29, 139.85, 145.54, 199.19; **HRESI-MS**(m/z): Calculated for $\text{C}_{13}\text{H}_{11}\text{N}_3\text{O}_3$ (M + Na): 248.0800, found (M + Na): 248.0803.



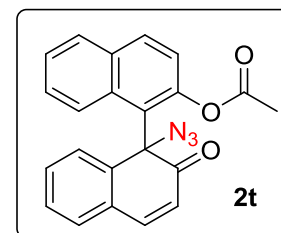
1-Allyl-1-azido-6-bromonaphthalen-2(1H)-one (2p): Brown oil; Yield – 82%; Rf (10% EtOAc/Hexane) 0.6; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2104, 1673; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 2.67 (dd, $J=13.43, 7.32$ Hz, 1 H) 2.78 (dd, $J=13.43, 7.32$ Hz, 1 H) 4.96 (dd, $J=17.09, 1.22$ Hz, 1 H) 5.01 - 5.12 (m, 1 H) 5.44 (ddt, $J=17.05, 9.96, 7.25, 7.25$ Hz, 1 H) 6.21 (d, $J=10.07$ Hz, 1 H) 7.33 (d, $J=10.07$ Hz, 1 H) 7.38 - 7.50 (m, 2 H) 7.57 (dd, $J=8.24, 1.83$ Hz, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 45.99, 69.91, 76.68, 77.31, 120.99, 122.51, 125.29, 129.40, 129.53, 131.42, 132.04, 132.97, 138.61, 143.91, 198.60; **HRESI-MS**(m/z): Calculated for $\text{C}_{13}\text{H}_{10}\text{BrN}_3\text{O}$ (M + Na): 325.9905, found (M + Na): 325.9904.



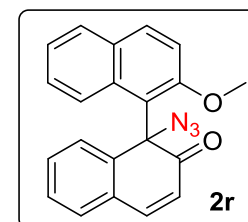
1,1'-Diazido-[1,1'-binaphthalene]-2,2'(1H,1'H)-dione (2q): Black solid; Yield – 72%; **mp**: 111°-113°C; Rf (20% EtOAc/Hexane) 0.5; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2088, 1672, 1011; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 6.09 (d, $J=10$ Hz, 1 H) 6.67 (d, $J=10$ Hz, 1 H) 7.20 (d, $J=11.2$ Hz, 2 H) 7.46-7.29 (m, 4 H) 7.83-7.80 (m, 2 H) 7.95 (d, $J=8.8$ Hz, 1H) 8 (d, $J=6.82$ Hz, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 75.35, 108.06, 112.59, 117.12, 121.52, 123.63, 125.13, 127.65, 127.96, 128.73, 129.17, 129.42, 130.32, 131.07, 131.15, 132.65, 156.92; **HRESI-MS**(m/z): Calculated for $\text{C}_{20}\text{H}_{12}\text{N}_6\text{O}_2$ (M + Na): 391.0919, found (M + Na): 391.0921.



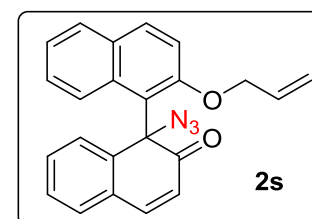
1'-Azido-2'-oxo-1',2'-dihydro-[1,1'-binaphthalen]-2-yl acetate (2r): Yellow oil; Yield – 71%; Rf (30% EtOAc/Hexane) 0.7; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2098, 1766, 1669, 1191; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 2.12 (s, 3 H) 6.46 (d, $J=10.07$ Hz, 1 H) 7.19-7.24 (m, 3 H) 7.33-7.35 (m, 1 H) 7.36-7.45 (m, 3 H) 7.62 (d, $J=10.7$ Hz, 1 H) 7.84-7.89 (m, 2 H) 8.14 (s, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 20.67, 71.84, 122.61, 124.04, 124.75, 125.16, 125.41, 127.33, 128.58, 129.18, 129.45, 129.66, 129.91, 130.78, 131.11, 132.41, 132.51, 139.71, 144.46, 147.66, 168.79, 190.78; **HRESI-MS**(m/z): Calculated for $\text{C}_{22}\text{H}_{15}\text{N}_3\text{O}_3$ (M + Na): 392.1011, found (M + Na): 392.1013.



1-Azido-2'-methoxy-[1,1'-binaphthalen]-2(1H)-one (2s): Brown solid; Yield – 92%; **mp**: 136°-137°C; Rf (30% EtOAc/Hexane) 0.5; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2927, 1668, 1594, 1021; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 3.60 (s, 3 H) 6.40 (d, $J=10.07$ Hz, 1 H) 7.09 - 7.26 (m, 2 H) 7.31 (t, $J=7.48$ Hz, 1 H) 7.34 - 7.47 (m, 3 H) 7.51 (d, $J=10.07$ Hz, 1 H) 7.63 (t, $J=7.78$ Hz, 1 H) 7.86 (d, $J=9.16$ Hz, 2 H) 9.16 (d, $J=8.85$ Hz, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 56.01, 70.71, 76.69, 77.32, 113.98, 122.96, 123.65, 123.71, 124.08, 127.78, 128.76, 129.16, 129.20, 129.21, 129.38, 130.23, 130.29, 131.16, 133.57, 140.15, 143.58, 154.19, 192.28; **HRESI-MS**(m/z): Calculated for $\text{C}_{21}\text{H}_{15}\text{N}_3\text{O}_2$ (M + Na): 364.1062, found (M + Na): 364.1062. **Optical rotation**: $[\alpha]_{\text{D}}^{25} +164.8$ ($c = 1.0, \text{CHCl}_3$) for an enantiomerically enriched sample with 79:21er. The enantiomeric ratio was determined by HPLC analysis using Phenomenex cellulose-1 column (*i*PrOH/*n*-Hexane 10:90, 1.0 mL/min, 20 °C, 254 nm, $\tau_{\text{major}} = 19.4$ min, $\tau_{\text{minor}} = 18.0$ min).



2'-(Allyloxy)-1-azido-[1,1'-binaphthalen]-2(1H)-one (2t): Brown solid; Yield – 92%; **mp**: 130°-133°C; Rf (20% EtOAc/Hexane) 0.5; Prepared as shown in general experimental procedure (a). **IR** (neat, cm^{-1}): 2096, 1668, 1021; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 4.33-4.42 (m, 2 H) 5.03-5.10 (m, 2 H) 5.64-5.74 (m, 1 H) 6.37 (d, $J=10.07$

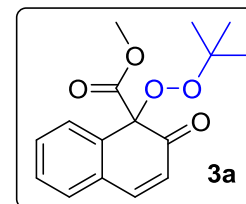


Hz, 1 H) 7.14-7.49 (m, 7 H) 7.59-7.63 (m, 1 H) 7.59-7.63(m, 2 H) 9.15 (d, $J=8.8$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 70.24, 70.96, 114.51, 117.99, 122.44, 123.63, 123.78, 124.67, 127.70, 128.75, 129.10, 129.17, 129.34, 129.43, 130.19, 131.02, 132.57, 133.75, 140.29, 143.52, 153.29; **HRESI-MS**(m/z): Calculated for $\text{C}_{23}\text{H}_{17}\text{N}_3\text{O}_2$ (M + Na): 390.1218, found (M + Na): 390.1217. **Optical rotation**: $[\alpha]_{\text{D}}^{25}$ -50.3 ($c = 1.0$, CHCl_3) for an enantiomerically enriched sample with 79:21er. The enantiomeric ratio was determined by HPLC analysis using Phenomenex cellulose-1 column ($i\text{PrOH}/n\text{-Hexane}$ 5:95, 1.0 mL/min, 20 °C, 254 nm, $\tau_{\text{major}} = 17.2$ min, $\tau_{\text{minor}} = 13.7$ min).

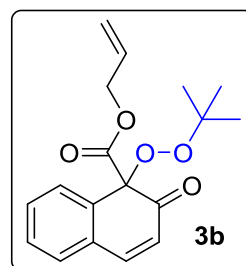
Characterization data for quaternary peroxidation of β -naphthol derivatives

Methyl 1-(tert-butylperoxy)-2-oxo-1,2-dihydronaphthalene-1-carboxylate (3a):

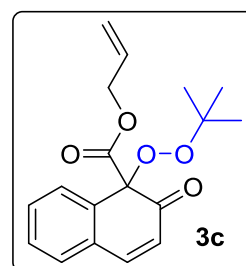
Brown solid; Yield – 95%; mp : 112°-114°C; Rf (30% EtOAc/Hexane) 0.7; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 1759, 1680, 1020; ^1H NMR (400 MHz, CDCl_3): δ 1.13 (s, 9 H) 3.55 - 3.74 (m, 3 H) 6.23 (d, $J=10.07$ Hz, 1 H) 7.31 - 7.39 (m, 1 H) 7.39 - 7.45 (m, 3 H) 7.56 - 7.68 (m, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 26.27, 52.84, 76.68, 77.31, 81.05, 83.96, 124.90, 128.09, 129.34, 129.36, 129.99, 130.58, 137.31, 145.18, 166.22, 193.21; **HRESI-MS**(m/z): Calculated for $\text{C}_{16}\text{H}_{18}\text{O}_5$ (M + Na): 313.1052, found (M + Na): 313.1052.



Allyl 1-(tert-butylperoxy)-2-oxo-1,2-dihydronaphthalene-1-carboxylate (3b): Orange oil; Yield – 98%; Rf (10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 2981, 1757, 1681; ^1H NMR (400 MHz, CDCl_3): δ 1.14 (s, 9 H) 4.39 - 4.64 (m, 2 H) 4.98 - 5.19 (m, 2 H) 5.60 - 5.81 (m, 1 H) 6.24 (d, $J=9.77$ Hz, 1 H) 7.29 - 7.51 (m, 4 H) 7.63 (d, $J=6.71$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 26.31, 65.96, 76.69, 77.32, 81.06, 84.04, 118.19, 124.98, 128.08, 129.32, 129.36, 129.96, 130.64, 130.73, 137.40, 145.11, 165.33, 193.10; **HRESI-MS**(m/z): Calculated for $\text{C}_{18}\text{H}_{20}\text{O}_5$ (M + Na): 339.1208, found (M + Na): 339.1206.

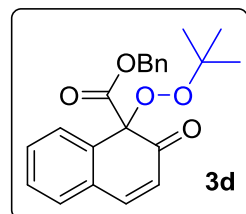


Allyl 1-(tert-butylperoxy)-2-oxo-1,2-dihydronaphthalene-1-carboxylate (3c): Brown solid; Yield – 98%; mp : 68°-70°C; Rf (10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 2932, 1763, 1679, 1195; ^1H NMR (400 MHz, CDCl_3): δ 1.13 (s, 9 H) 2.40 (t, $J=2.44$ Hz, 1 H) 4.61 - 4.67 (m, 2 H) 6.24 (d, $J=10.07$ Hz, 1 H) 7.32 - 7.38 (m, 1 H) 7.38 - 7.48 (m, 3 H) 7.55 - 7.64 (m, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 26.27, 53.13, 75.69, 76.18, 76.68, 77.32, 81.22, 83.85, 124.84, 128.30, 129.47, 129.54, 130.01, 130.63, 136.69, 145.24, 165.05, 192.65; **HRESI-MS**(m/z): Calculated for $\text{C}_{18}\text{H}_{18}\text{O}_5$ (M + Na): 337.1052, found (M + Na): 337.1052.

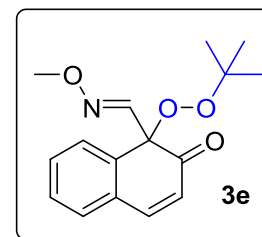


Benzyl 1-(tert-butylperoxy)-2-oxo-1,2-dihydronaphthalene-1-carboxylate (3d):

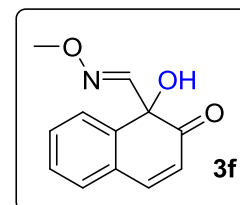
Orange oil; Yield – 78%; Rf (10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 1758, 1680, 1196; ^1H NMR (400 MHz, CDCl_3): δ 1.14 (s, 9 H) 5.09 (s, 2 H) 6.22 (d, $J=9.77$ Hz, 1 H) 6.96 - 7.10 (m, 2 H) 7.18 - 7.28 (m, 2 H) 7.28 - 7.45 (m, 5 H) 7.50 - 7.61 (m, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 26.31, 67.11, 76.68, 77.31, 81.10, 84.14, 124.98, 127.28, 128.03, 128.11, 128.30, 129.29, 129.35, 129.92, 130.64, 134.81, 137.21, 145.13, 165.45, 193.03; **HRESI-MS**(m/z): Calculated for $\text{C}_{22}\text{H}_{22}\text{O}_5$ (M + Na): 389.1365, found (M + Na): 389.1365.



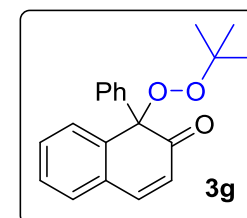
(E)-1-(tert-butylperoxy)-2-oxo-1,2-dihydronaphthalene-1-carbaldehyde O-methyl oxime (3e): Brown oil; Yield – 20%; Rf (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 2933, 2314, 1682, 1573, 1018; **^1H NMR** (400 MHz, CDCl_3): δ 1.18 (s, 9 H) 3.74 (s, 3 H) 6.19 (d, $J=10.07$ Hz, 1 H) 7.30-7.41 (m, 3 H) 7.43 (t, $J=8.1$ Hz, 1 H) 7.62 (d, $J=10.07$ Hz, 1 H); **^{13}C NMR** (100 MHz, CDCl_3): δ 26.49, 62.22, 80.83, 83.39, 125.12, 128.65, 129.11, 129.25, 129.63, 130.71, 139.19, 144.57, 148.02, 195.11; **HRESI-MS**(m/z): Calculated for $\text{C}_{16}\text{H}_{19}\text{NO}_4$ (M + Na): 312.1212, found (M + Na): 312.1212.



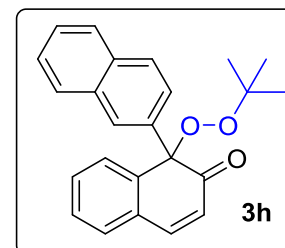
(E)-1-Hydroxy-2-oxo-1,2-dihydronaphthalene-1-carbaldehyde O-methyl oxime (3f): Black oil; Yield – 54%; Rf (10% EtOAc/Hexane) 0.2; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 3351, 1680, 1573, 1012; **^1H NMR** (400 MHz, CDCl_3): δ 3.77 (s, 3 H) 4.22 (s, 1 H) 6.27 (d, $J=9.77$ Hz, 1 H) 7.29 - 7.34 (m, 1 H) 7.34 - 7.50 (m, 4 H) 7.70 (d, $J=7.63$ Hz, 1 H); **^{13}C NMR** (100 MHz, CDCl_3): δ 62.15, 76.69, 77.32, 123.53, 126.84, 127.44, 128.89, 129.27, 129.60, 130.68, 139.49, 145.54, 150.49, 199.89; **HRESI-MS**(m/z): Calculated for $\text{C}_{12}\text{H}_{11}\text{NO}_3$ (M + Na): 240.0637, found (M + Na): 240.0637.



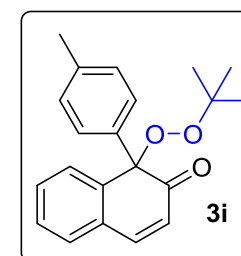
1-(tert-Butylperoxy)-1-phenylnaphthalen-2(1H)-one (3g): Yellow solid; Yield – 64%; **mp**: 98°-99°C; Rf (5% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 3327, 1686, 1399, 1279; **^1H NMR** (400 MHz, CDCl_3): δ 1.27 (s, 9 H) 6.05 (d, $J=10.07$ Hz, 1 H) 7.12 - 7.28 (m, 5 H) 7.32 - 7.46 (m, 4 H) 7.47 - 7.56 (m, 1 H); **^{13}C NMR** (100 MHz, CDCl_3): δ 26.67, 76.68, 77.31, 80.54, 86.79, 124.95, 126.76, 128.22, 128.27, 128.49, 128.78, 129.16, 129.89, 131.34, 138.67, 143.03, 144.38, 197.32; **HRESI-MS**(m/z): Calculated for $\text{C}_{20}\text{H}_{20}\text{O}_3$ (M + Na): 331.1310, found (M + Na): 331.1314.



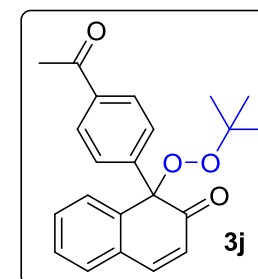
1-(tert-Butylperoxy)-[1,2'-binaphthalen]-2(1H)-one (3h): Yellow oil; Yield – 52%; Rf (10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 2980, 1684, 1196; **^1H NMR** (400 MHz, CDCl_3): δ 1.30 (s, 9 H) 6.09 (d, $J=10.07$ Hz, 1 H) 7.35 - 7.48 (m, 6 H) 7.49 - 7.57 (m, 3 H) 7.66 - 7.77 (m, 3 H); **^{13}C NMR** (100 MHz, CDCl_3): δ 26.72, 76.69, 77.32, 80.67, 86.86, 124.26, 125.07, 126.07, 126.17, 126.52, 127.47, 128.06, 128.35, 128.45, 128.89, 129.37, 129.97, 131.42, 132.85, 133.14, 136.09, 143.02, 144.46, 197.28; **HRESI-MS**(m/z): Calculated for $\text{C}_{24}\text{H}_{22}\text{O}_3$ (M + Na): 381.1467, found (M + Na): 381.1467.



1-(tert-Butylperoxy)-1-(p-tolyl)naphthalen-2(1H)-one (3i): Yellow oil; Yield – 40%; Rf (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 2988, 1690, 1490; **^1H NMR** (400 MHz, CDCl_3): δ 1.27 (s, 9 H) 2.26 (s, 3 H) 6.04 (d, $J=10.07$ Hz, 1 H) 6.88 - 7.17 (m, 4 H) 7.27 - 7.46 (m, 4 H) 7.46 - 7.58 (m, 1 H); **^{13}C NMR** (100 MHz, CDCl_3): δ 21.07, 26.68, 76.69, 77.32, 80.46, 86.91, 124.99, 126.84, 128.15, 128.76, 129.00, 129.11, 129.83, 131.36, 135.60, 138.56, 143.07, 144.22, 197.49; **HRESI-MS**(m/z): Calculated for $\text{C}_{21}\text{H}_{22}\text{O}_3$ (M + Na): 345.1467, found (M + Na): 345.1465.

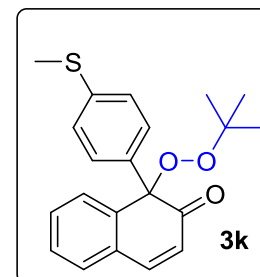


1-(4-Acetylphenyl)-1-(tert-butylperoxy)naphthalen-2(1H)-one (3j): Yellow solid; Yield – 72%; **mp**: 83°-85°C; Rf(30% EtOAc/Hexane) 0.2; Prepared as shown in general

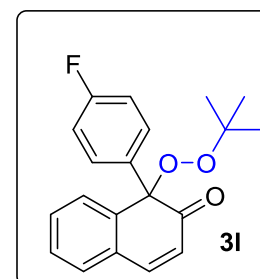


experimental procedure (b). **IR** (neat, cm^{-1}): 1685, 1603, 1266; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 1.26 (s, 9 H) 2.54 (s, 3 H) 6.10 (d, $J=10.07$ Hz, 1 H) 7.21 - 7.33 (m, 2 H) 7.34 - 7.48 (m, 5 H) 7.76 - 7.86 (m, 2 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 26.59, 26.61, 76.69, 77.32, 80.86, 86.16, 124.81, 126.82, 128.27, 128.59, 129.04, 129.20, 130.15, 131.18, 136.79, 142.45, 143.74, 144.86, 196.54, 197.58; **HRESI-MS**(m/z): Calculated for $\text{C}_{22}\text{H}_{22}\text{O}_4(\text{M} + \text{Na})$: 373.1416, found (M + Na): 373.1419.

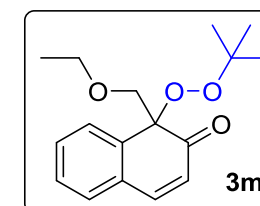
1-(tert-Butylperoxy)-1-(4-(methylthio)phenyl)naphthalen-2(1H)-one (3k): Yellow oil; Yield – 48%; Rf (10% EtOAc/Hexane) 0.5; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 2980, 1684, 1195; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 1.26 (s, 9 H) 2.41 (s, 3 H) 6.05 (d, $J=10.07$ Hz, 1 H) 7.03 - 7.13 (m, 4 H) 7.31 - 7.47 (m, 4 H) 7.51 (d, $J=7.32$ Hz, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 15.44, 26.66, 76.68, 77.20, 77.31, 80.56, 86.62, 123.48, 124.97, 126.04, 127.41, 127.73, 128.30, 128.83, 129.14, 129.90, 130.20, 131.36, 135.12, 139.48, 142.72, 144.27, 197.18; **HRESI-MS**(m/z): Calculated for $\text{C}_{21}\text{H}_{22}\text{O}_3\text{S}(\text{M} + \text{Na})$: 377.1187, found (M + Na): 377.1186.



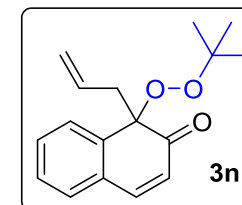
1-(tert-Butylperoxy)-1-(4-fluorophenyl)naphthalen-2(1H)-one (3l): Yellow solid; Yield – 79%; mp: 70°-72°C; Rf(10% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 2980, 1685, 1500; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 1.26 (s, 9 H) 6.05 (d, $J=10.07$ Hz, 1 H) 6.84 - 6.96 (m, 2 H) 7.09 - 7.22 (m, 2 H) 7.32 - 7.46 (m, 4 H) 7.46 - 7.55 (m, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 26.64, 76.68, 77.31, 80.66, 86.23, 115.09, 115.30, 124.87, 128.43, 128.86, 128.91, 128.94, 129.17, 129.99, 131.32, 134.38, 134.40, 142.64, 144.42, 161.54, 164.01, 197.18; **HRESI-MS**(m/z): Calculated for $\text{C}_{20}\text{H}_{19}\text{FO}_3(\text{M} + \text{Na})$: 349.1216, found (M + Na): 349.1219.



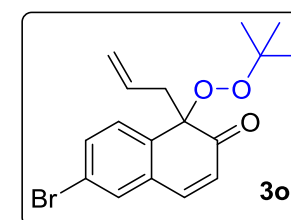
1-(tert-Butylperoxy)-1-(ethoxymethyl)naphthalen-2(1H)-one (3m): Black oil; Yield – 30%; Rf (20% EtOAc/Hexane) 0.7; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 3436, 2979, 1679; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 1.04 (t, $J=7.02$ Hz, 3 H) 1.18 (s, 9 H) 3.32 - 3.54 (m, 2 H) 3.59 - 3.75 (m, 2 H) 6.17 (d, $J=10.07$ Hz, 1 H) 7.19 - 7.46 (m, 4 H) 7.65 (d, $J=7.63$ Hz, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 14.86, 26.56, 68.01, 75.67, 76.68, 77.31, 80.13, 86.48, 125.45, 128.02, 128.04, 129.00, 129.32, 131.09, 140.77, 144.59, 197.46; **HRESI-MS**(m/z): Calculated for $\text{C}_{17}\text{H}_{22}\text{O}_4(\text{M} + \text{Na})$: 313.1416, found (M + Na): 313.1415.



1-Allyl-1-(tert-butylperoxy)naphthalen-2(1H)-one (3n): Orange oil; Yield – 44%; Rf(20% EtOAc/Hexane) 0.8; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 1680, 1195; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 1.16 (s, 9 H) 2.50 (dd, $J=12.97$, 6.87 Hz, 1 H) 2.69 (dd, $J=12.97$, 7.78 Hz, 1 H) 4.74 - 4.97 (m, 2 H) 5.22 - 5.45 (m, 1 H) 6.14 (d, $J=10.07$ Hz, 1 H) 7.20 - 7.38 (m, 3 H) 7.43 (t, $J=7.48$ Hz, 1 H) 7.63 (d, $J=7.93$ Hz, 1 H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 26.53, 45.14, 76.69, 77.32, 80.12, 84.68, 119.58, 125.80, 127.45, 127.86, 128.84, 129.52, 129.56, 130.98, 142.61, 144.70, 198.88; **HRESI-MS**(m/z): Calculated for $\text{C}_{17}\text{H}_{20}\text{O}_3(\text{M} + \text{Na})$: 295.1310, found (M + Na): 295.1307.

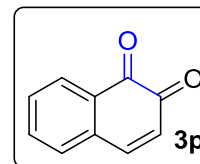


1-Allyl-6-bromo-1-(tert-butylperoxy)naphthalen-2(1H)-one (3o): Orange oil; Yield – 42%; Rf (10% EtOAc/Hexane) 0.7; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 3424, 1643, 1437; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 1.15 (s, 9 H) 2.46 (dd, $J=12.97$, 6.87 Hz, 1 H) 2.67 (dd, $J=12.97$, 7.78 Hz, 1 H) 4.79 - 4.90

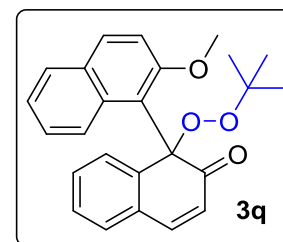


(m, 1 H) 4.92 (d, $J=10.07$ Hz, 1 H) 5.34 (ddt, $J=17.17, 9.92, 7.36, 7.36$ Hz, 1 H) 6.18 (d, $J=10.07$ Hz, 1 H) 7.25 (d, $J=10.07$ Hz, 1 H) 7.43 (d, $J=1.83$ Hz, 1 H) 7.49 (d, $J=8.24$ Hz, 1 H) 7.55 (dd, $J=8.39, 1.98$ Hz, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 26.49, 44.92, 76.68, 77.31, 80.37, 84.48, 120.05, 121.67, 126.93, 129.07, 129.16, 131.33, 132.31, 132.86, 141.40, 143.01, 198.16; **HRESI-MS**(m/z): Calculated for $\text{C}_{17}\text{H}_{19}\text{BrO}_3(\text{M} + \text{Na})$: 373.0415, found (M + Na): 373.0412.

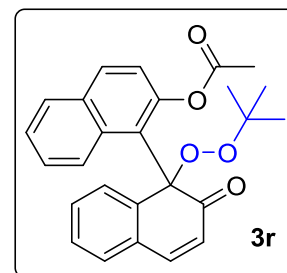
Naphthalene-1,2-dione (3p): Yellow solid; Yield – 56%; *mp*: 145°-146°C; *Rf* (30% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 3382, 1661, 1287; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 6.44 (d, $J=10.38$ Hz, 1 H) 7.37 (d, $J=7.32$ Hz, 1 H) 7.45 (d, $J=10.38$ Hz, 1 H) 7.52 (t, $J=7.48$ Hz, 1 H) 7.66 (t, $J=7.63$ Hz, 1 H) 8.11 (d, $J=7.63$ Hz, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 76.69, 77.32, 127.94, 129.83, 130.21, 130.85, 131.63, 134.78, 135.81, 145.32, 178.89, 180.86; **HRESI-MS**(m/z): Calculated for $\text{C}_{10}\text{H}_6\text{O}_2(\text{M} + \text{Na})$: 181.0265, found (M + Na): 181.0263.



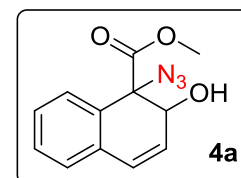
1-(tert-Butylperoxy)-2'-methoxy-[1,1'-binaphthalen]-2(1H)-one (3q): Brown solid; Yield – 80%; *mp*: 138°-139°C; *Rf* (30% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 1682, 1595, 1019; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 1.09 (s, 9 H) 3.51 (s, 3 H) 6.40 (d, $J=9.77$ Hz, 1 H) 6.98 - 7.20 (m, 2 H) 7.20 - 7.33 (m, 3 H) 7.33 - 7.44 (m, 3 H) 7.53 (ddd, $J=8.70, 7.02, 1.37$ Hz, 1 H) 7.78 (dd, $J=8.39, 5.04$ Hz, 2 H) 9.12 (d, $J=8.85$ Hz, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 26.50, 55.76, 76.68, 77.32, 80.56, 86.67, 113.97, 123.48, 125.75, 126.63, 126.73, 127.91, 128.07, 128.74, 129.09, 129.74, 130.36, 130.59, 131.53, 133.68, 141.58, 142.30, 153.68, 196.89; **HRESI-MS**(m/z): Calculated for $\text{C}_{25}\text{H}_{24}\text{O}_4(\text{M} + \text{Na})$: 411.1572, found (M + Na): 411.1570.



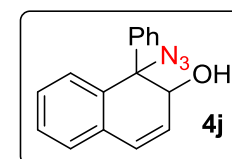
1'-(tert-Butylperoxy)-2'-oxo-1',2'-dihydro-[1,1'-binaphthalen]-2-yl acetate (3r): Brown liquid; Yield – 98%; *Rf* (30% EtOAc/Hexane) 0.7; Prepared as shown in general experimental procedure (b). **IR** (neat, cm^{-1}): 1768, 1682, 1186; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 1.09 (s, 9 H) 1.89 (s, 3 H) 6.43 (d, $J=10.07$ Hz, 1 H) 7.07 (d, $J=8.8$ Hz, 1 H) 7.15-7.32 (m, 7 H) 7.82-7.85 (m, 2 H) 9.08 (s, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 20.59, 26.42, 81.03, 86.74, 121.65, 125.20, 126.45, 126.63, 127.45, 127.83, 128.25, 128.71, 128.99, 129.92, 130.16, 130.76, 130.81, 132.48, 133.59, 140.80, 142.49, 145.92, 167.67, 195.33; **HRESI-MS**(m/z): Calculated for $\text{C}_{26}\text{H}_{24}\text{O}_5(\text{M} + \text{Na})$: 439.1521, found (M + Na): 439.1519.



Methyl 1-azido-2-hydroxy-1,2-dihydronaphthalene-1-carboxylate (4a): Yellow oil; Yield – 42%; *Rf* (30% EtOAc/Hexane) 0.7; Prepared as shown in general experimental procedure (c). **IR** (neat, cm^{-1}): 3471 (br) 2114, 1737, 1253; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 3.29 (s, 1 H) 3.77 (s, 3 H) 4.83 (d, $J=7.6$ Hz, 1 H) 6.01 (dd, $J_1=10$ Hz, $J_2=2.8$ Hz, 1 H) 6.44 (dd, $J_1=10$ Hz, $J_2=2.0$ Hz, 1 H) 7.12 (d, $J=7.2$ Hz, 1 H) 7.28-7.34 (m, 2 H) 7.51 (d, $J=6.8$ Hz, 1 H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 53.19, 72.24, 73.73, 127.28, 127.33, 127.71, 128.27, 129.59, 129.90, 130.59, 132.42, 169.86; **HRESI-MS**(m/z): Calculated for $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_3(\text{M} + \text{Na})$: 268.0698, found (M + Na): 268.0698.

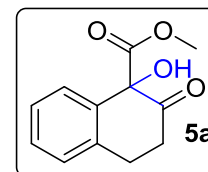


1-Azido-1-phenyl-1,2-dihydronaphthalen-2-ol (4j): Yellow oil; Yield – 95%; *Rf* (20% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (c). **IR** (neat, cm^{-1}): 3409, 2104, 1447; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 4.37 (br. s., 1 H) 5.98 - 6.07 (m, 1 H) 6.58 (d, $J=9.77$ Hz, 1 H) 7.21 - 7.28 (m, 3 H) 7.28 - 7.39 (m, 6 H) 7.39 - 7.49 (m, 3 H); ^{13}C

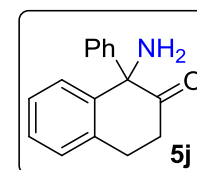


NMR (100 MHz, CDCl₃): δ 72.07, 72.81, 73.22, 74.55, 75.59, 76.68, 77.31, 126.40, 127.38, 127.62, 127.79, 128.00, 128.07, 128.14, 128.19, 128.27, 128.33, 128.37, 128.45, 128.73, 128.76, 128.86, 128.94, 129.04, 129.40, 129.84, 130.52, 132.97, 133.17, 134.03, 134.31, 136.64, 140.01; **HRESI-MS**(*m/z*): Calculated for C₁₆H₁₃N₃O(M + Na): 286.0956, found (M + Na): 286.0956.

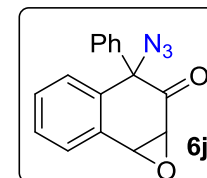
Methyl 1-hydroxy-2-oxo-1,2,3,4-tetrahydronaphthalene-1-carboxylate (5a): Yellow oil; Yield – 41%; R_f (20% EtOAc/Hexane) 0.8; Prepared as shown in general experimental procedure (d). **IR** (neat, cm⁻¹): 3446, 1722, 1453; **¹H NMR** (400 MHz, CDCl₃): δ 2.57 - 2.77 (m, 1 H) 2.96 - 3.17 (m, 2 H) 3.24 - 3.44 (m, 1 H) 3.72 (s, 3 H) 4.54 (s, 1 H) 7.14 - 7.26 (m, 1 H) 7.29 - 7.40 (m, 2 H) 7.57 - 7.70 (m, 1 H); **¹³C NMR** (100 MHz, CDCl₃): δ 27.40, 34.50, 53.38, 76.68, 77.31, 79.60, 126.44, 127.46, 127.95, 128.89, 134.51, 135.91, 169.77, 206.76; **HRESI-MS**(*m/z*): Calculated for C₁₂H₁₂O₄(M + Na): 243.0633, found (M + Na): 243.0633.



1-Amino-1-phenyl-3,4-dihydronaphthalen-2(1H)-one (5j): Yellow oil; Yield – 72%; R_f (20% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (e). **IR** (neat, cm⁻¹): 3378, 3300, 1715, 1219; **¹H NMR** (400 MHz, CDCl₃): δ 2.45 - 2.61 (m, 2 H) 2.61 - 2.84 (m, 6 H) 6.96 - 7.04 (m, 2 H) 7.13 - 7.22 (m, 2 H) 7.22 - 7.30 (m, 3 H) 7.33 (t, *J*=7.48 Hz, 1 H) 7.40 (t, *J*=7.63 Hz, 1 H) 7.87 (d, *J*=7.63 Hz, 1 H); **¹³C NMR** (100 MHz, CDCl₃): δ 27.31, 34.98, 69.03, 76.68, 77.31, 126.55, 127.14, 127.25, 127.71, 127.81, 127.87, 128.89, 137.11, 139.14, 141.54, 211.32; **HRESI-MS**(*m/z*): Calculated for C₁₆H₁₅NO(M + H): 238.1232, found (M + H): 238.1231.



3-Azido-3-phenyl-3,7b-dihydronaphtho[1,2-b]oxiren-2(1aH)-one (6j): Yellow solid; Yield – 53%; *mp*: 119°-121°C; R_f (10% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure (f). **IR** (neat, cm⁻¹): 2106, 1723, 1219; **¹H NMR** (400 MHz, CDCl₃): δ 3.85 (d, *J*=3.97 Hz, 1 H) 4.23 (d, *J*=3.97 Hz, 1 H) 7.01 (dd, *J*=6.71, 2.75 Hz, 2 H) 7.27 - 7.42 (m, 3 H) 7.42 - 7.54 (m, 1 H) 7.54 - 7.66 (m, 2 H) 7.68 - 7.79 (m, 1 H); **¹³C NMR** (100 MHz, CDCl₃): δ 53.40, 59.23, 75.96, 76.69, 77.32, 126.85, 128.65, 128.76, 129.09, 129.64, 129.72, 130.11, 130.92, 136.80, 137.66, 202.15; **HRESI-MS**(*m/z*): Calculated for C₁₆H₁₁N₃O₂(M + Na): 300.0749, found (M + Na): 300.0743.



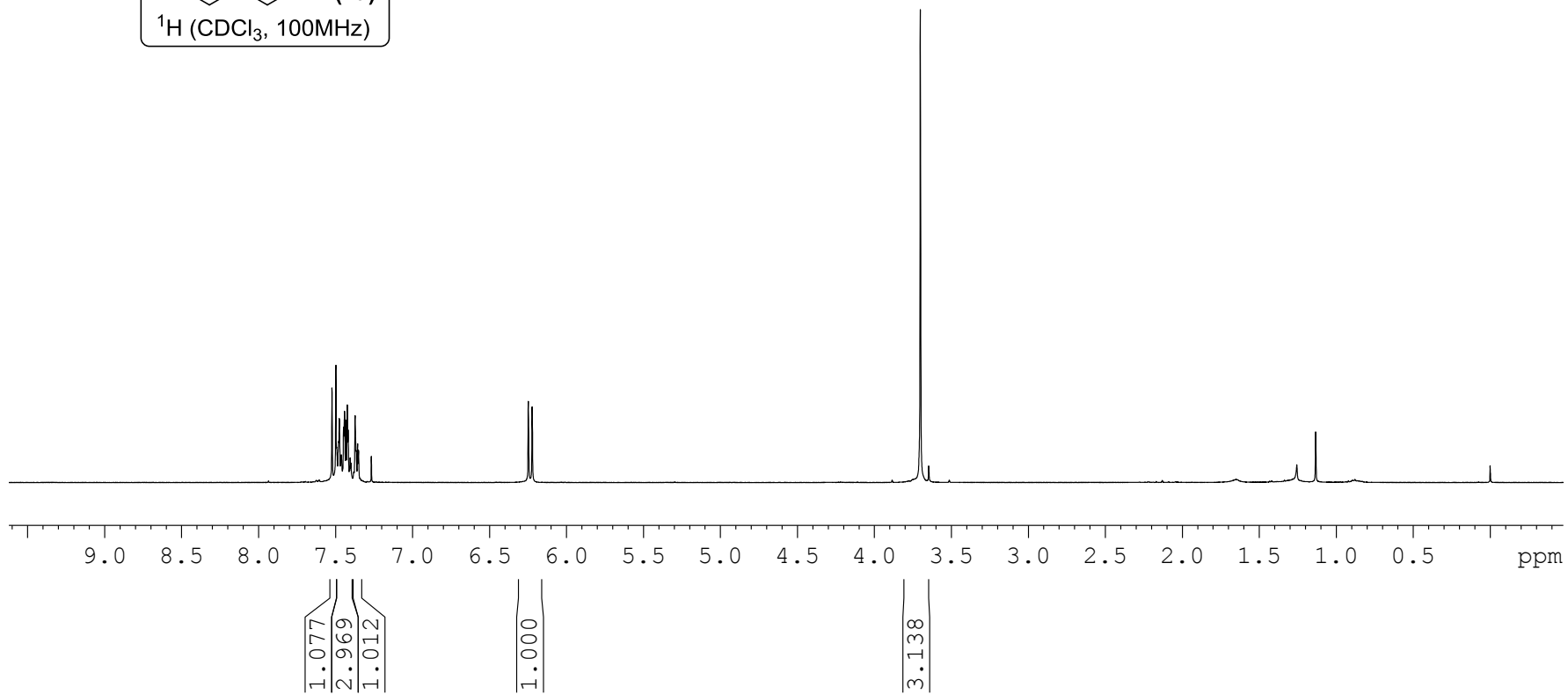
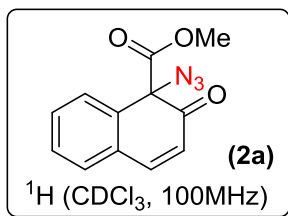
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1. W. Guo, J. Li, N. Fan, W. Wu, P. Zhou, C. Xia, *Synth. Commun.* 2005, **35**, 145.
2. C. Liu, Y. Zhang, N. Liu, J. Qiu, *Green Chem.* 2012, **14**, 2999.
3. F. Gonzalez-Bobes, N. Kopp, L. Li, J. Deerberg, P. Sharma, S. Leung, M. Davies, J. Bush, J. Hamm, M. Hrytsak, *Org. Process Res. Dev.* 2012, **16**, 2051.

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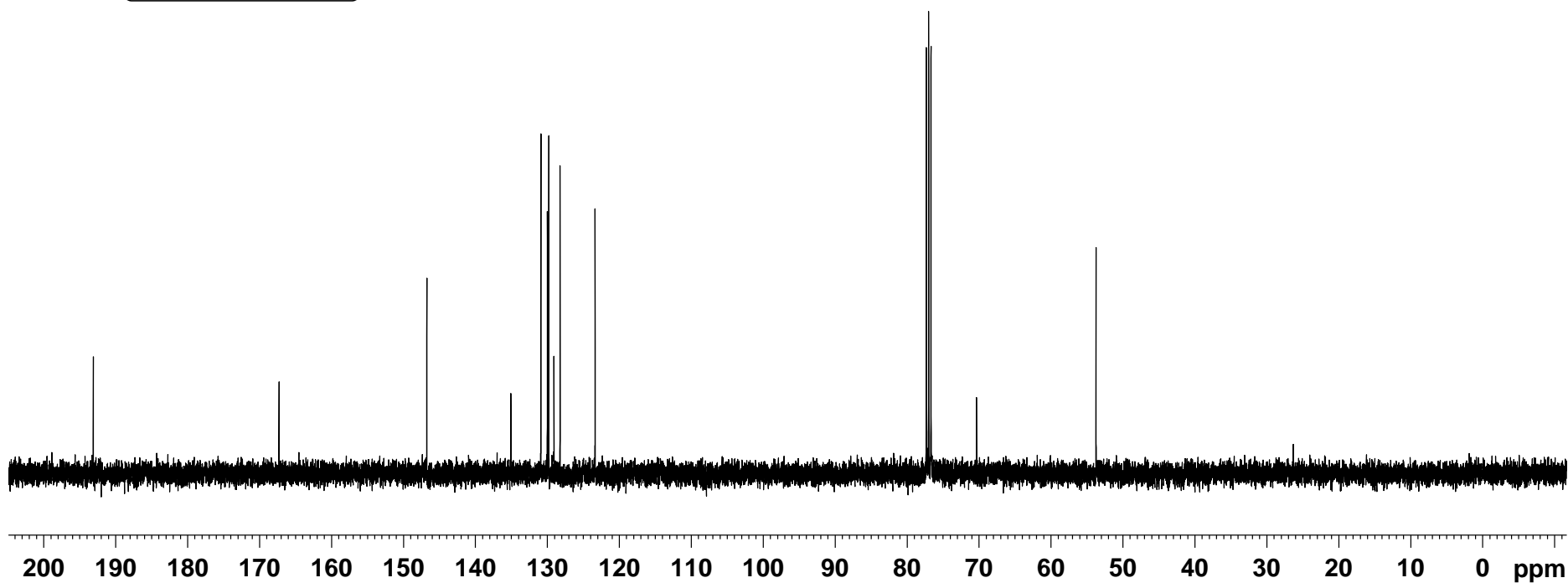
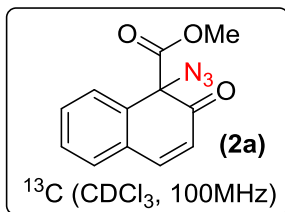
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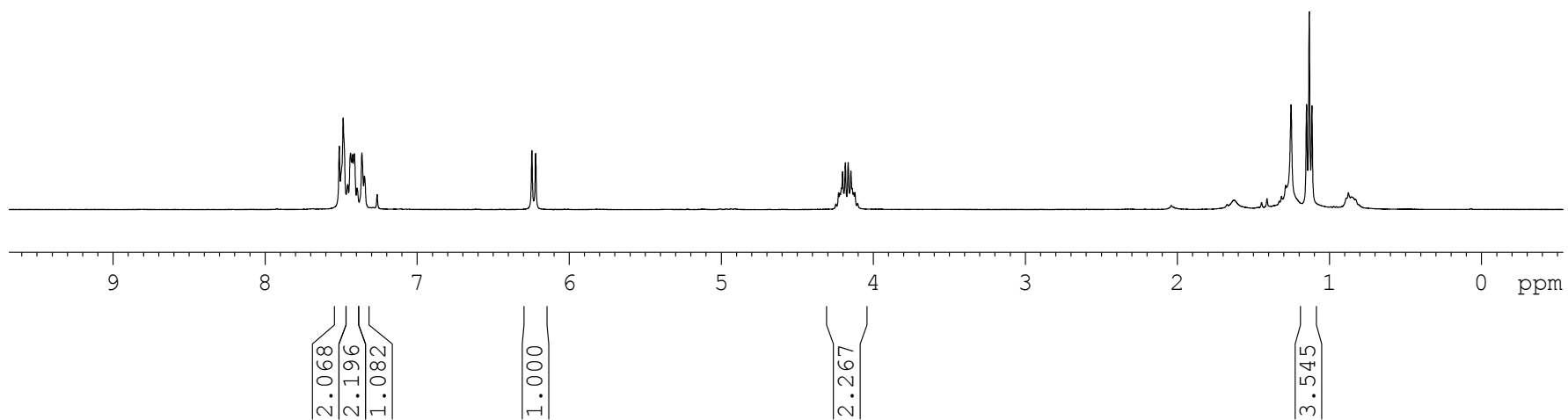
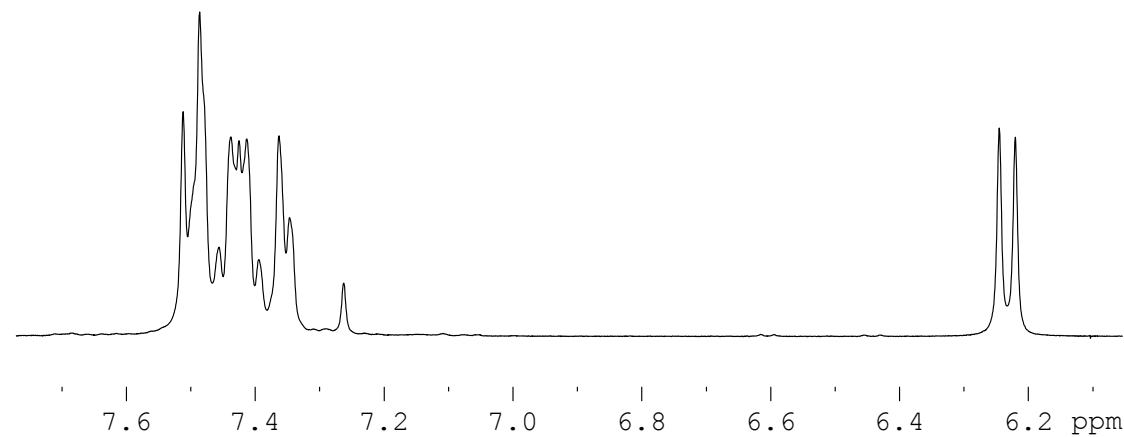
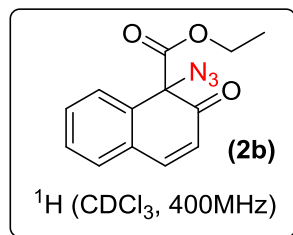
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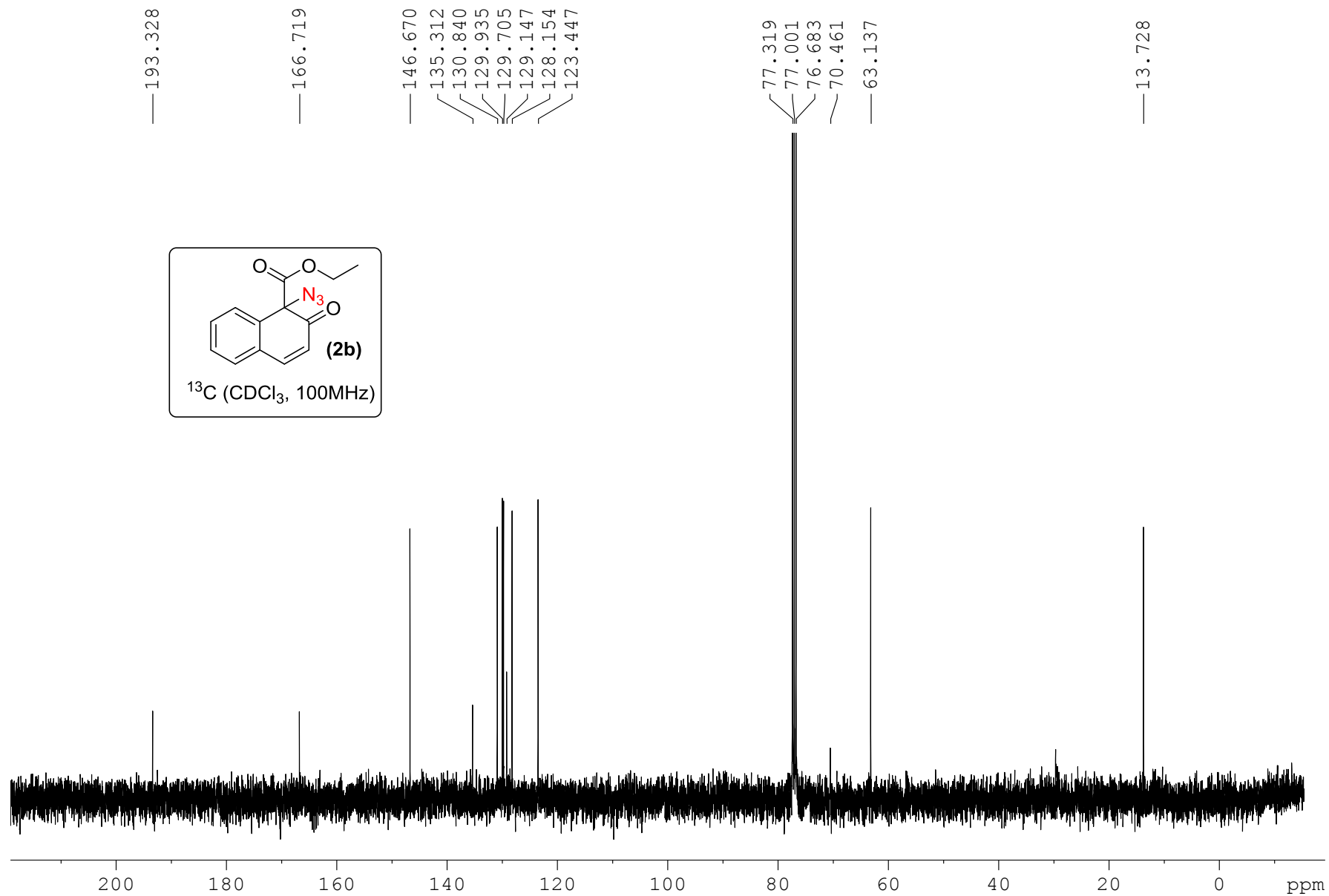


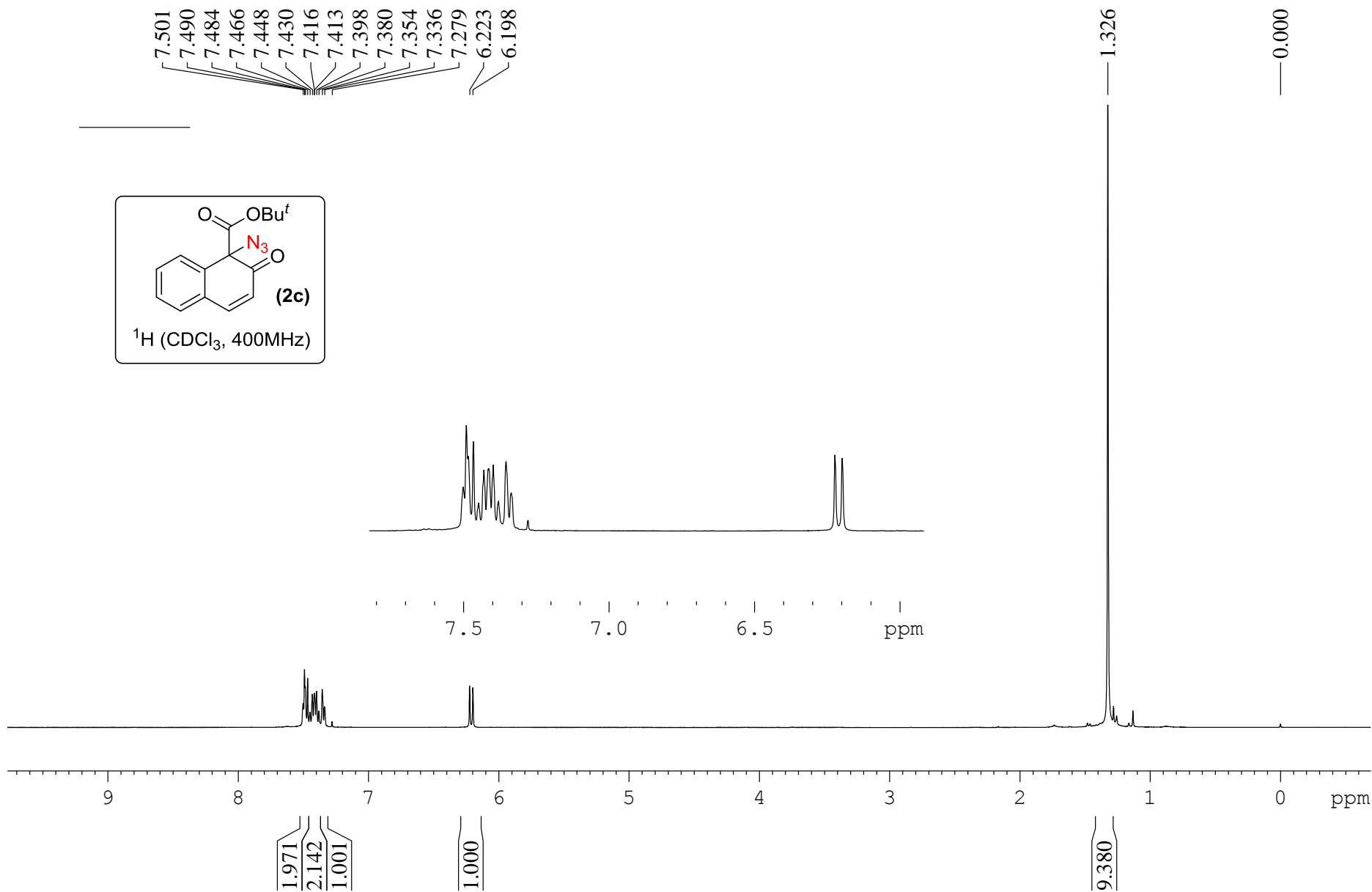
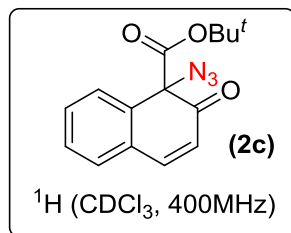
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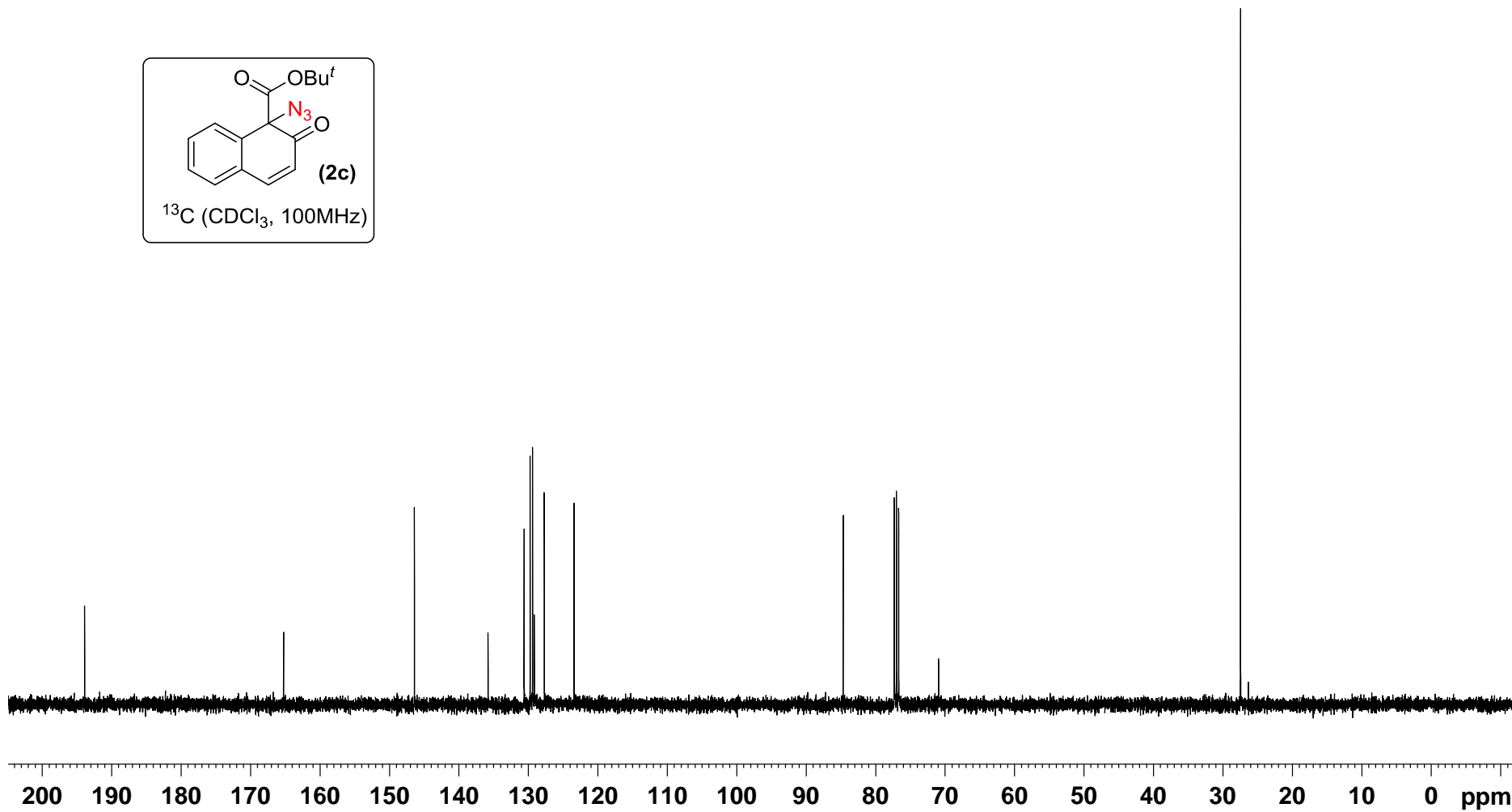
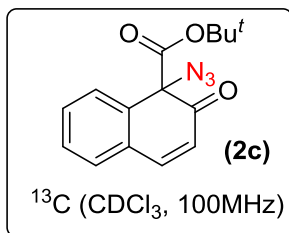
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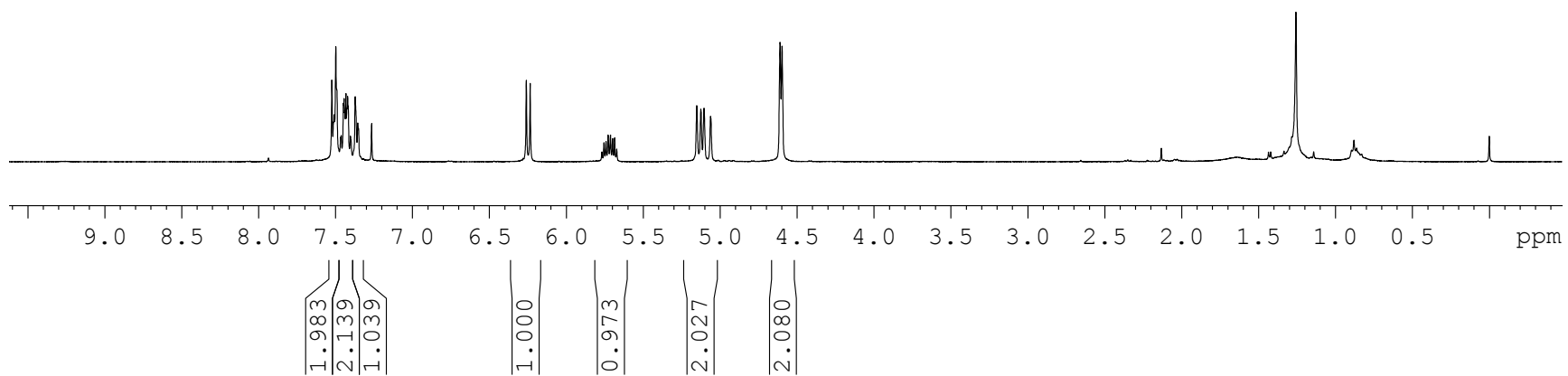
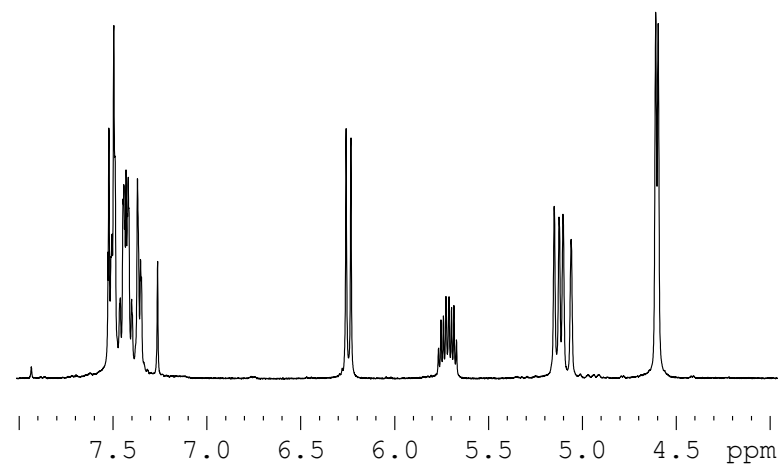
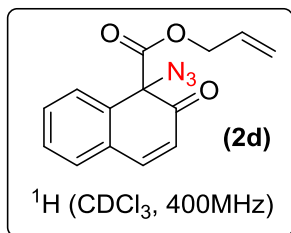
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7.424
7.420
7.401
7.370
7.354
7.349
7.263
6.258
6.233
5.766
5.753
5.740
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5.670
5.150
5.124
5.103
5.060
4.609
4.595

0.000

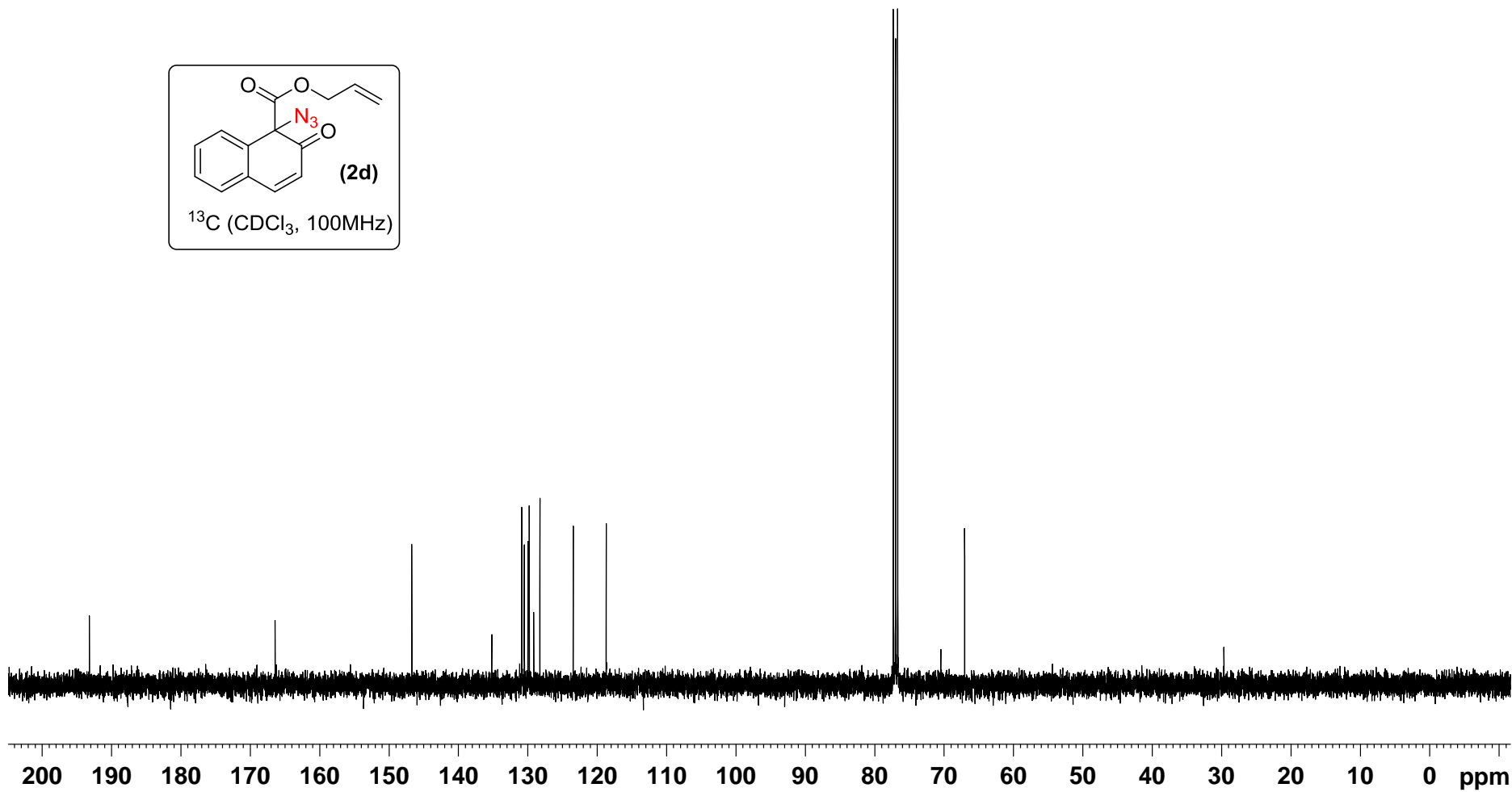
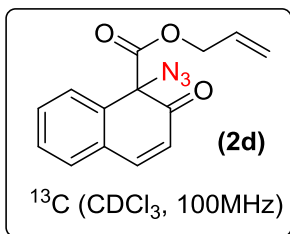


— 193.177

— 166.425

146.732
135.187
130.881
130.501
129.980
129.788
129.156
128.258
123.459
118.703

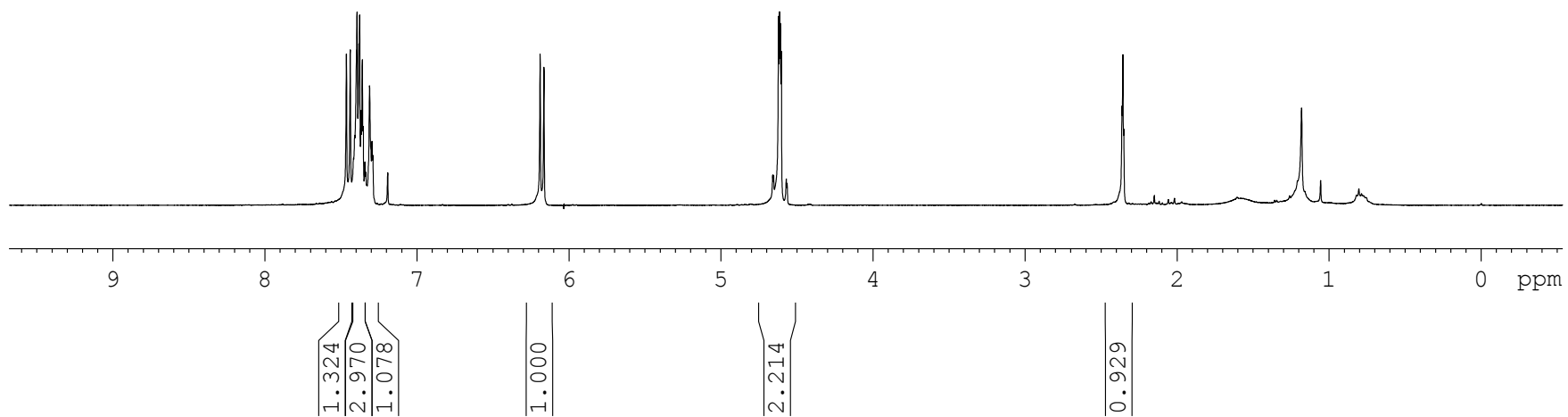
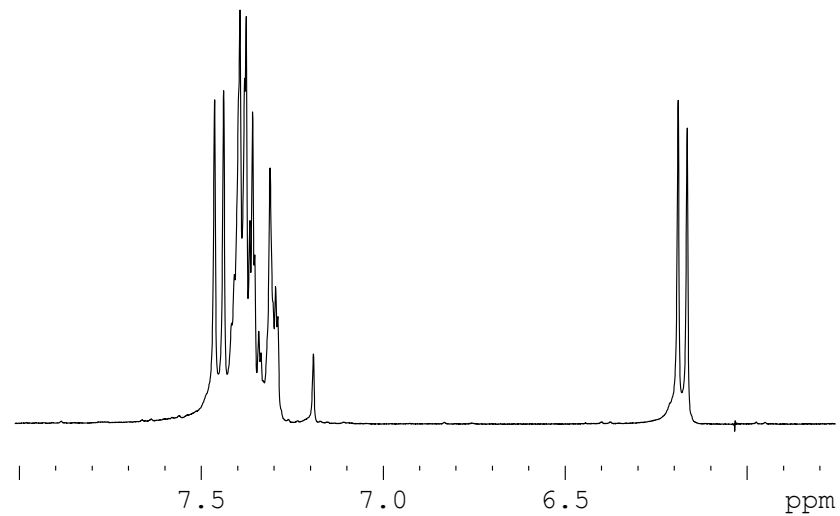
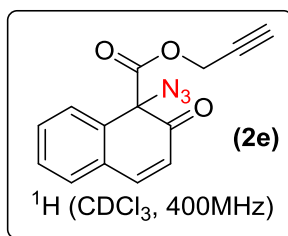
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77.001
76.683
70.481
67.031



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7.417
7.409
7.394
7.381
7.377
7.367
7.359
7.353
7.342
7.336
7.311
7.296
7.290
7.192
6.189
6.164

4.659
4.654
4.621
4.615
4.609
4.603
4.570

2.361
2.356



—192.475

—166.145

—146.887

134.653

130.937

130.098

129.951

129.114

128.326

123.333

77.318

77.000

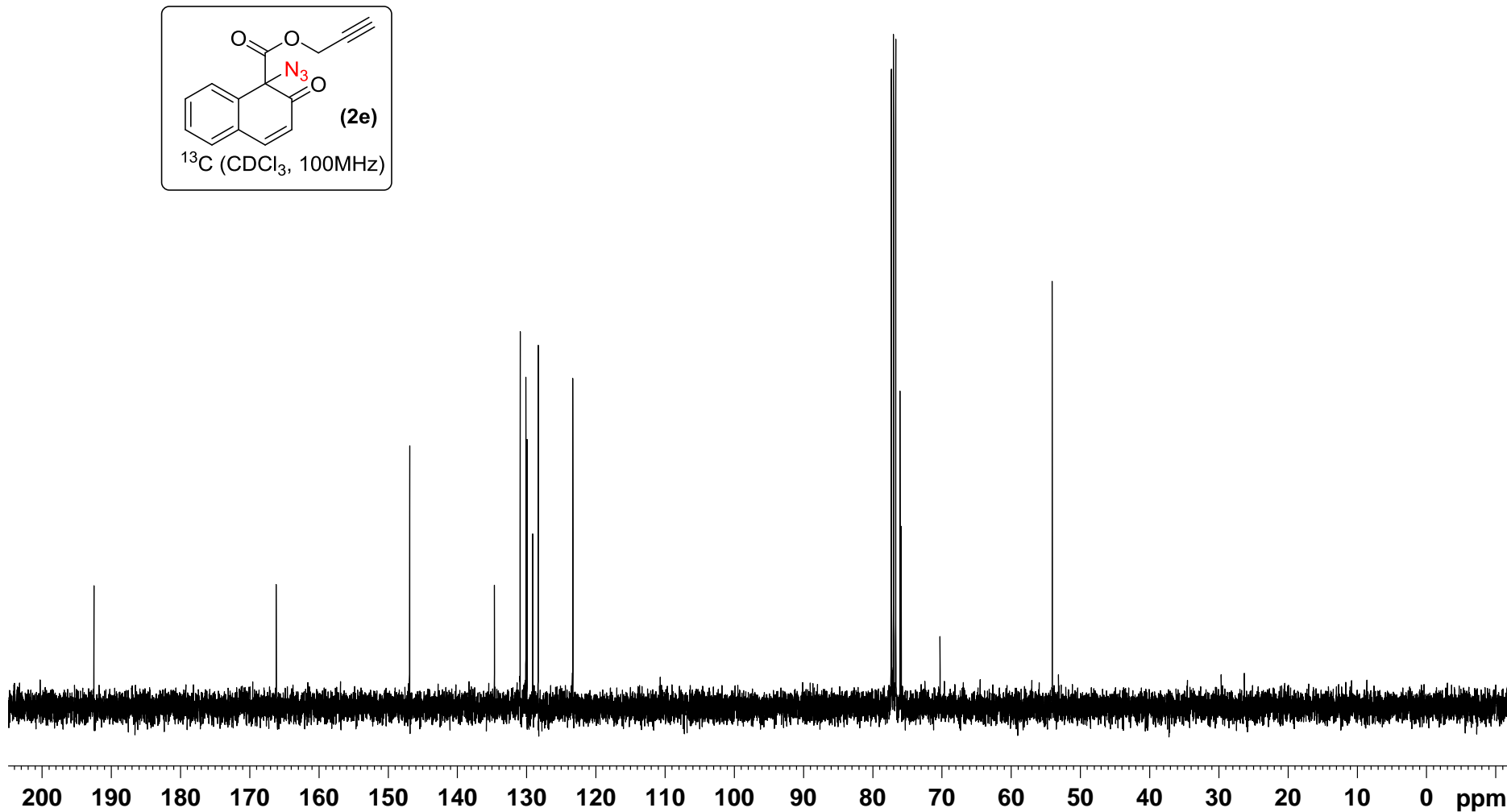
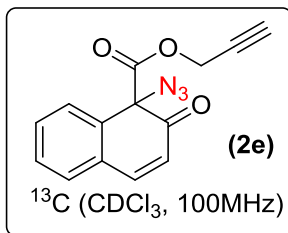
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76.055

75.879

70.272

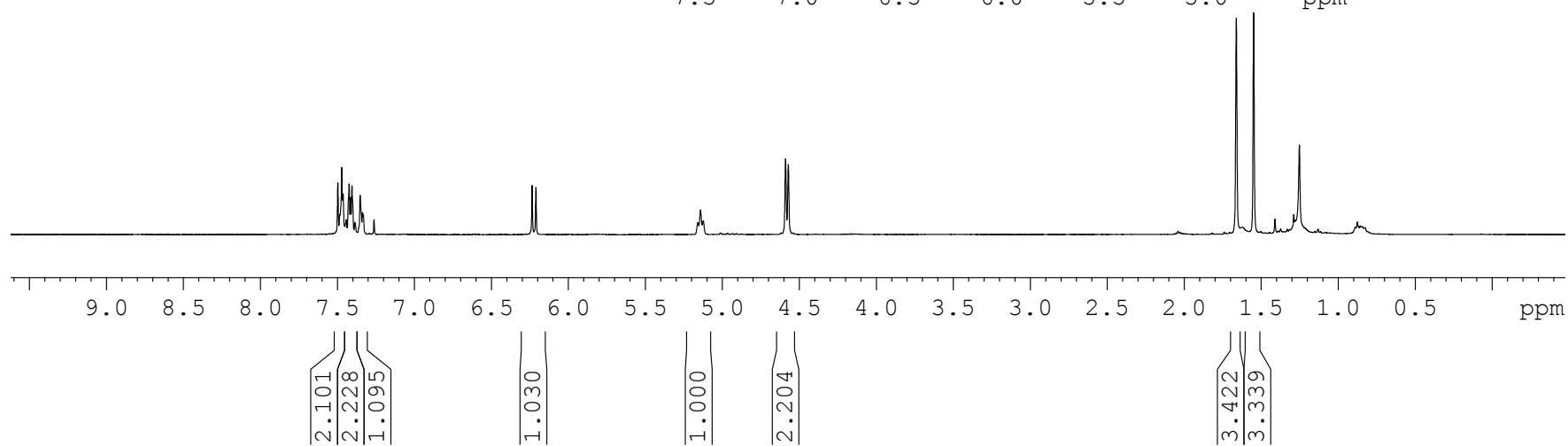
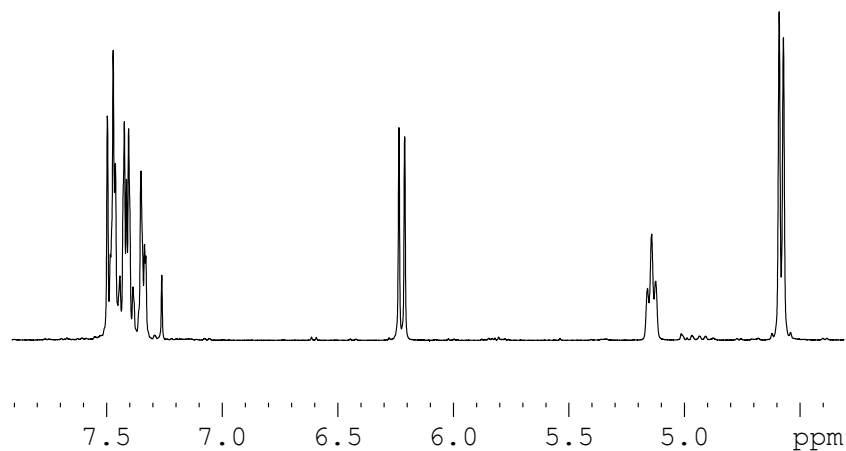
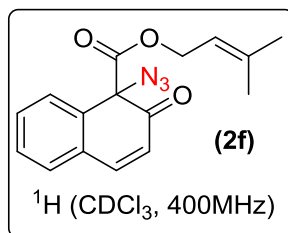
—54.068



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7.463
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7.423
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7.405
7.386
7.352
7.336
7.330
7.261
6.235
6.210

5.160
5.142
5.125
4.590
4.572

1.663
1.550



— 193.313

— 166.635

146.610

140.764

135.318

130.733

129.891

129.625

129.161

128.184

123.467

117.008

77.317

77.000

76.682

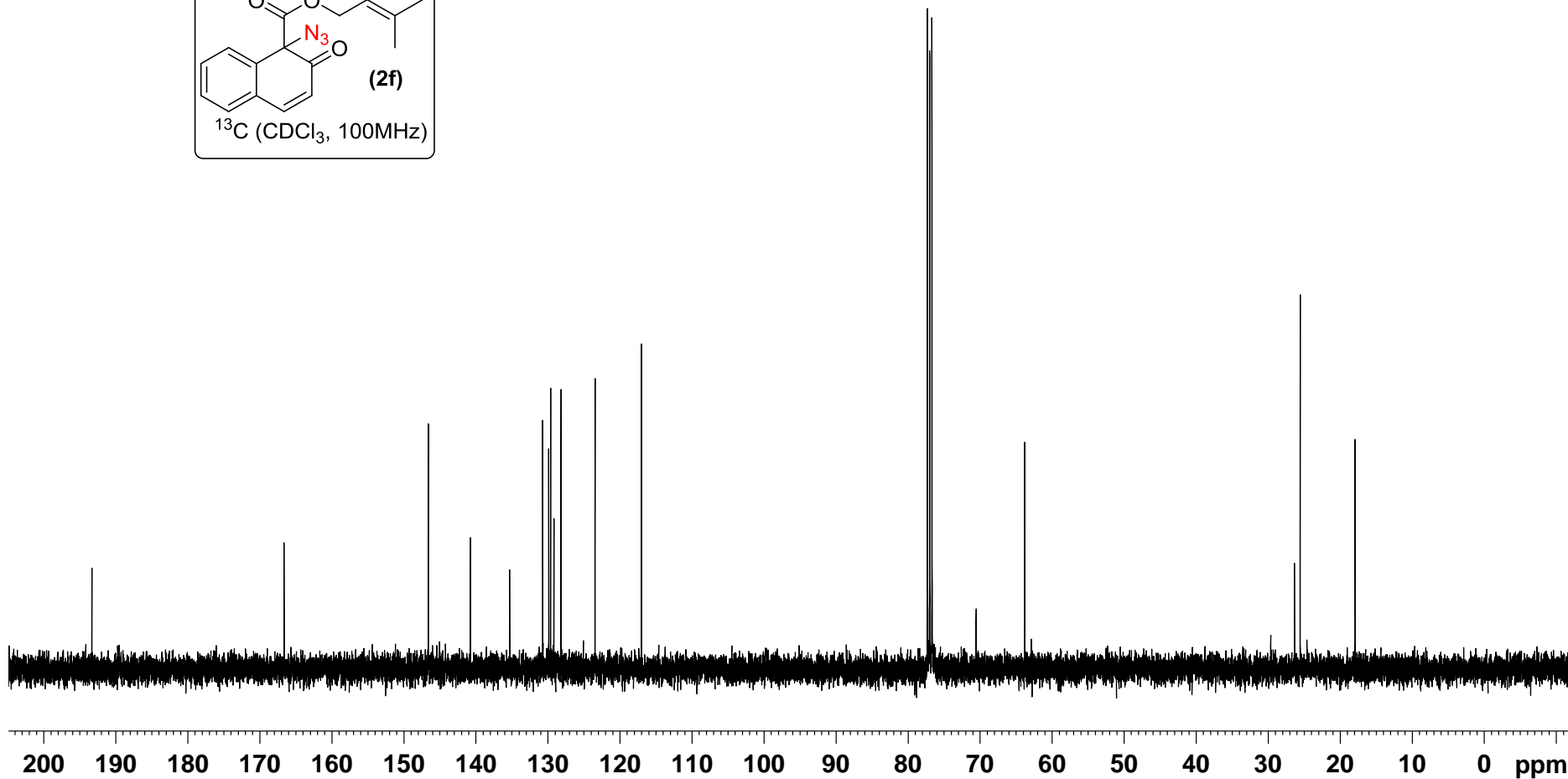
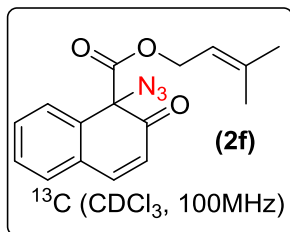
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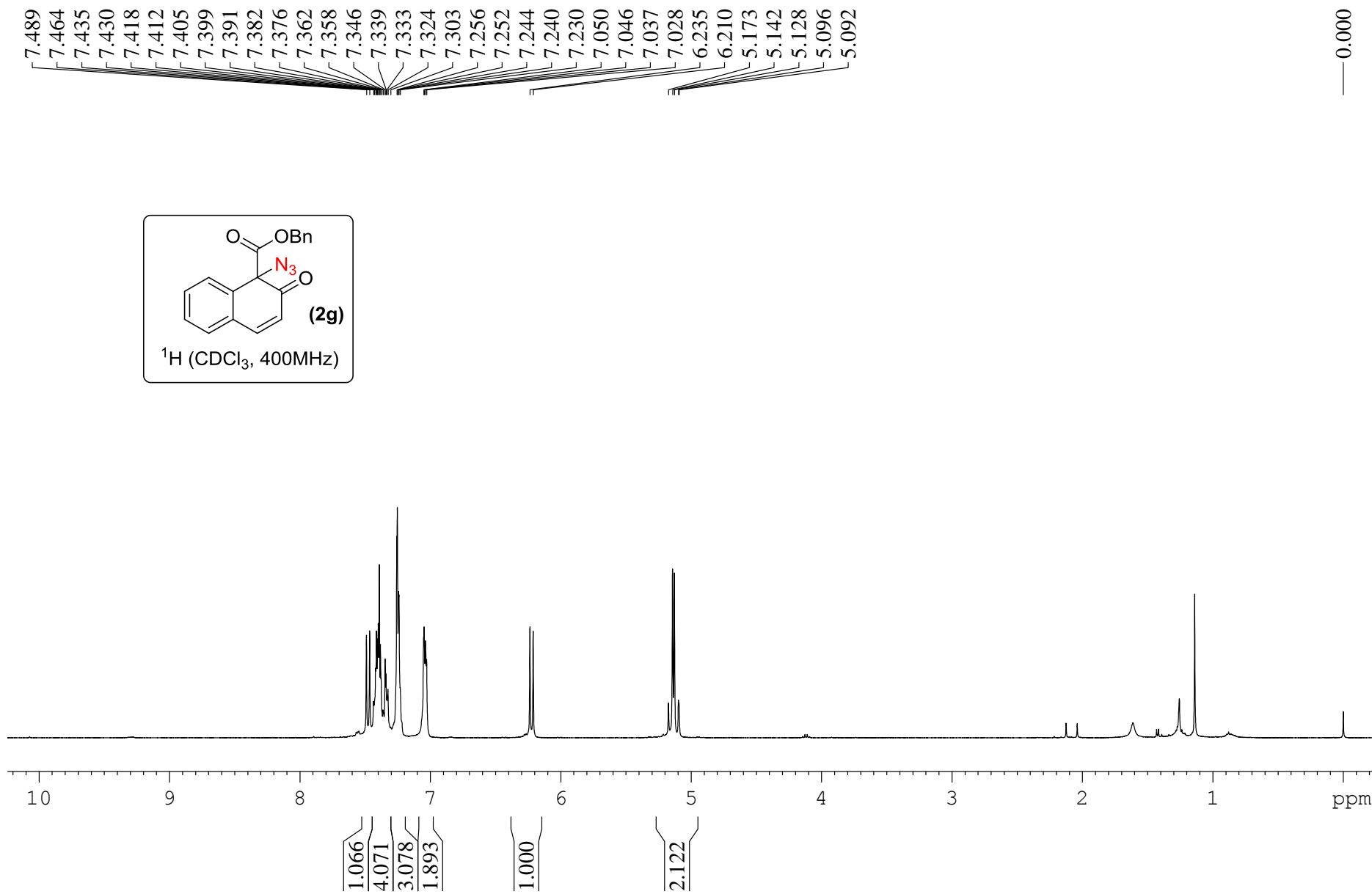
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25.562

17.935





193.091

166.528

146.752

135.047

134.475

130.826

129.964

129.732

129.162

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128.339

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127.463

127.323

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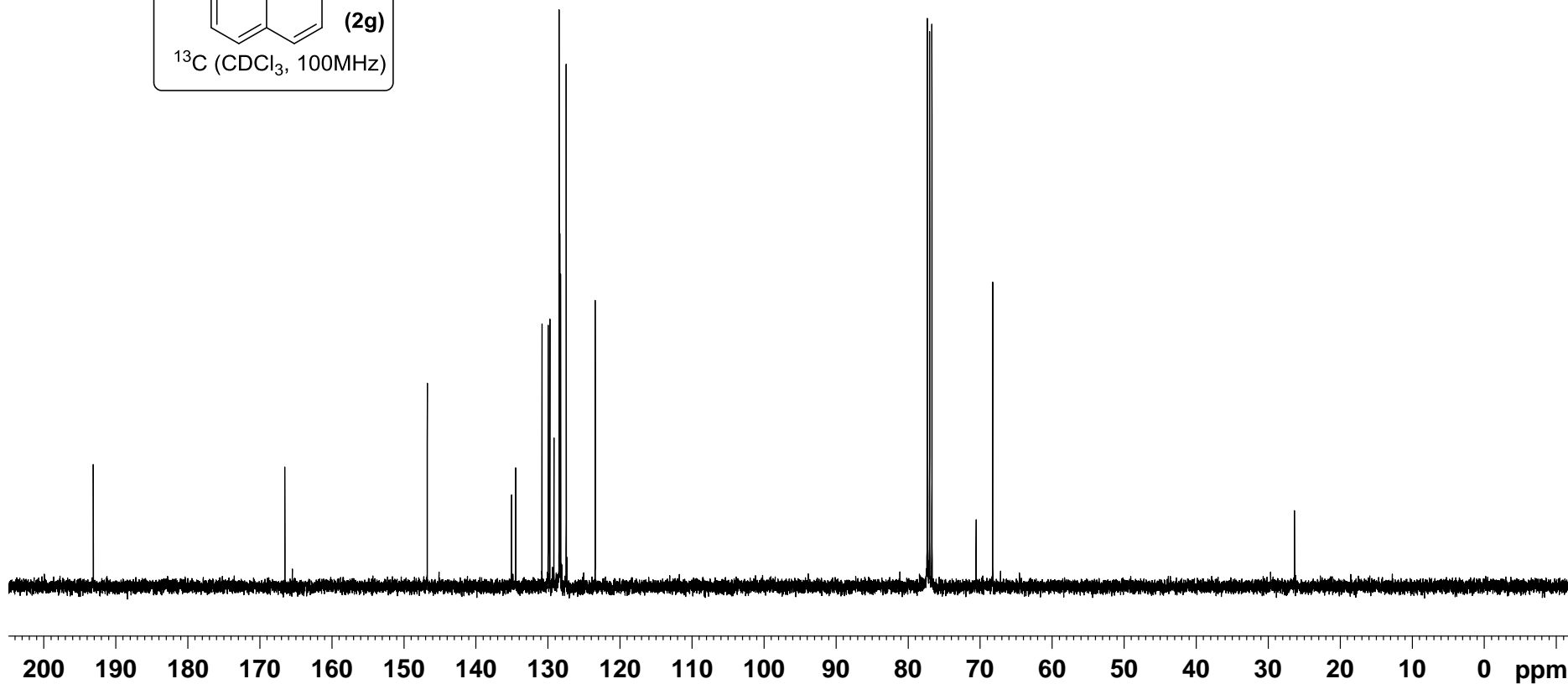
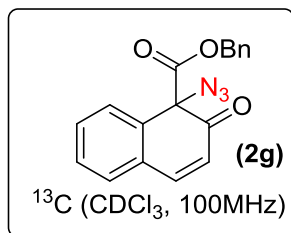
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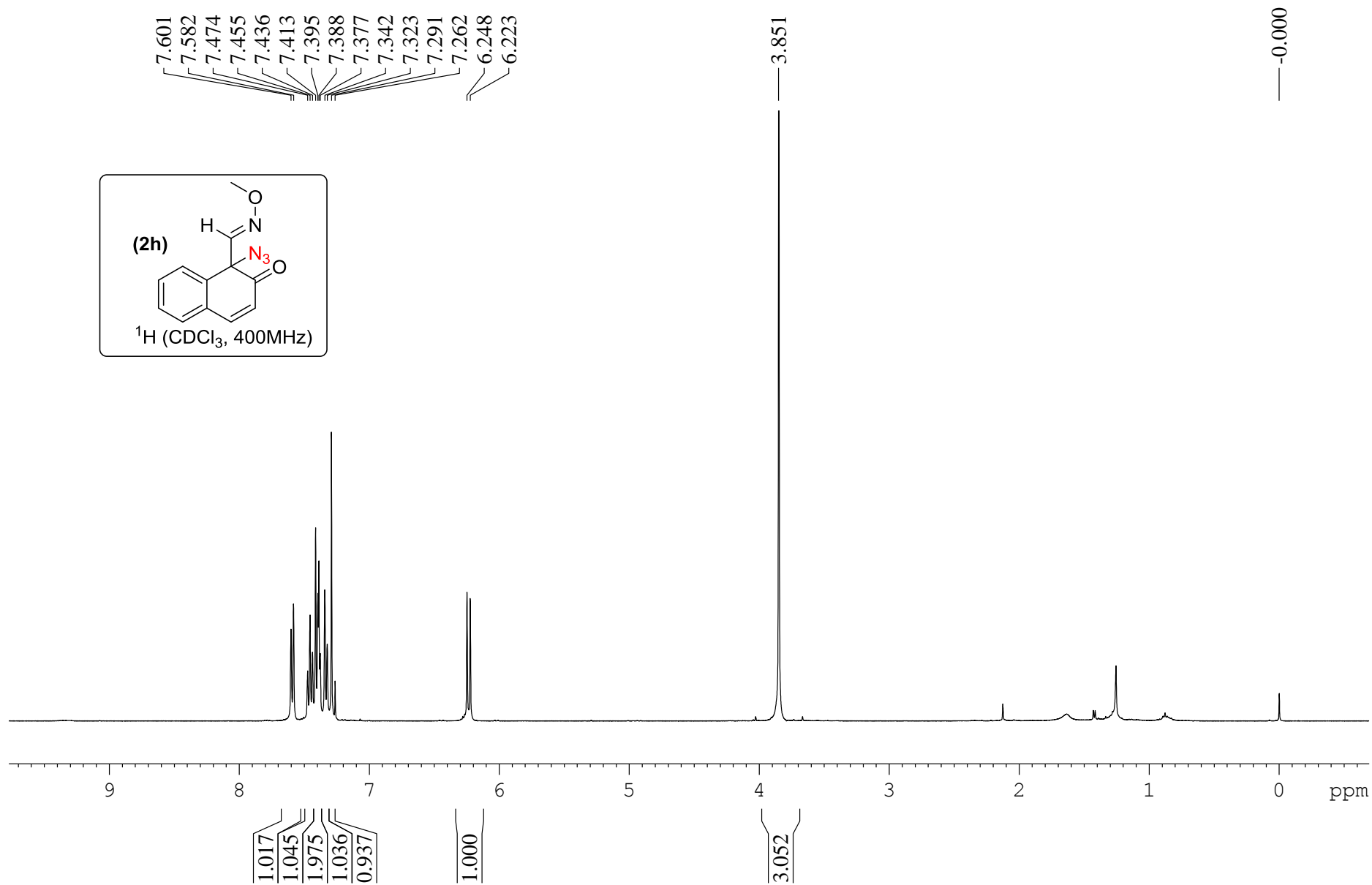
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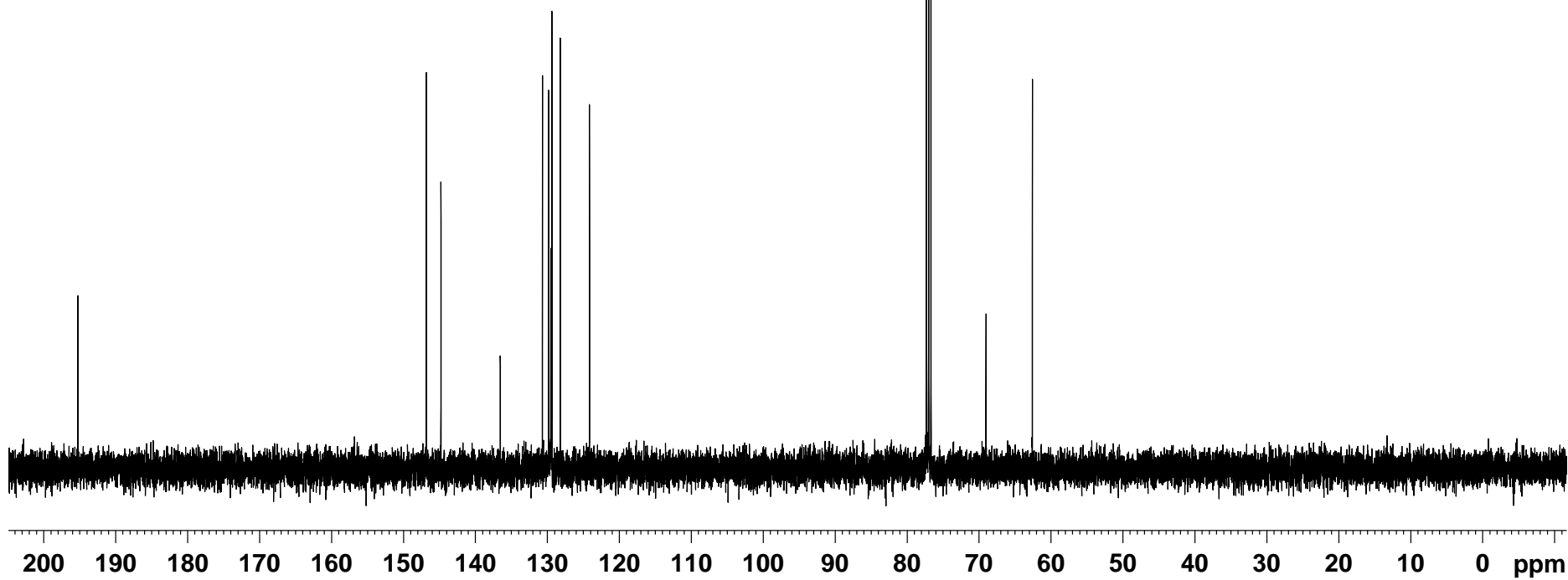
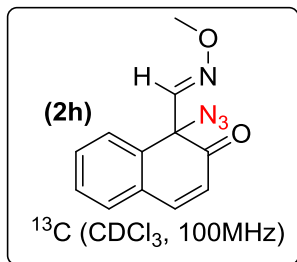




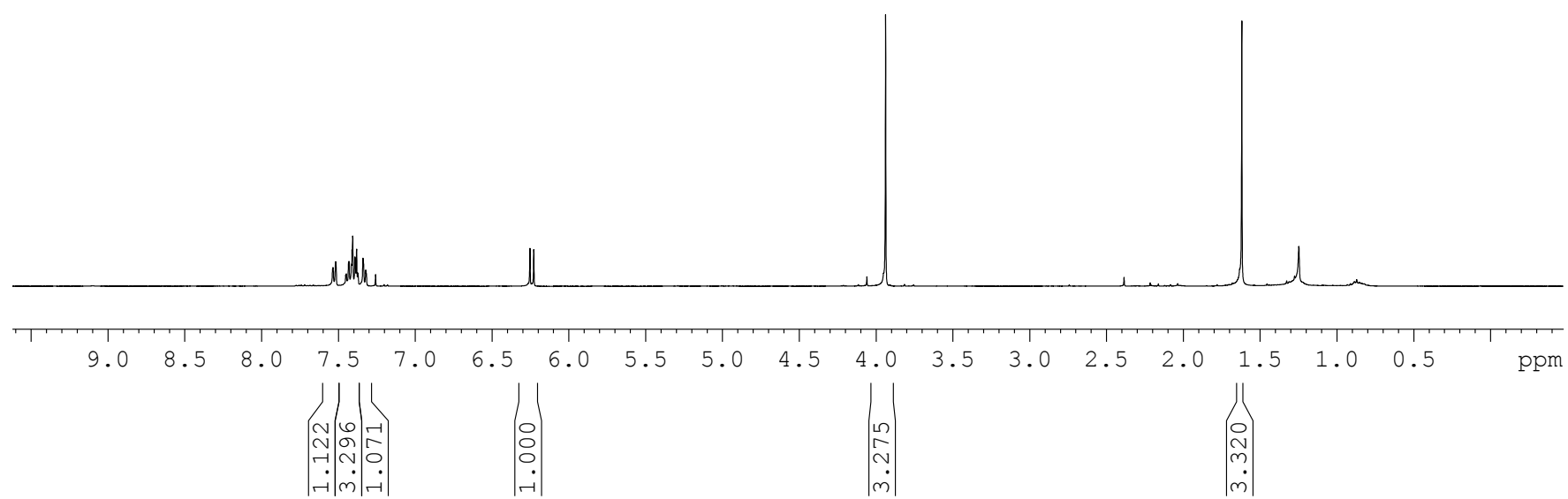
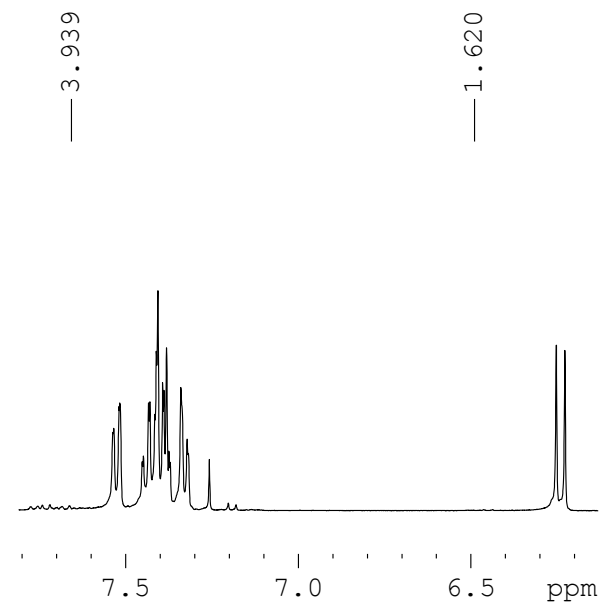
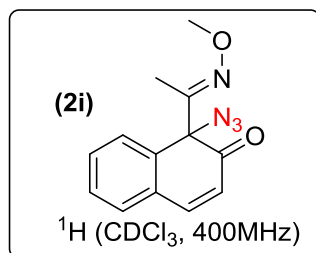
195.237

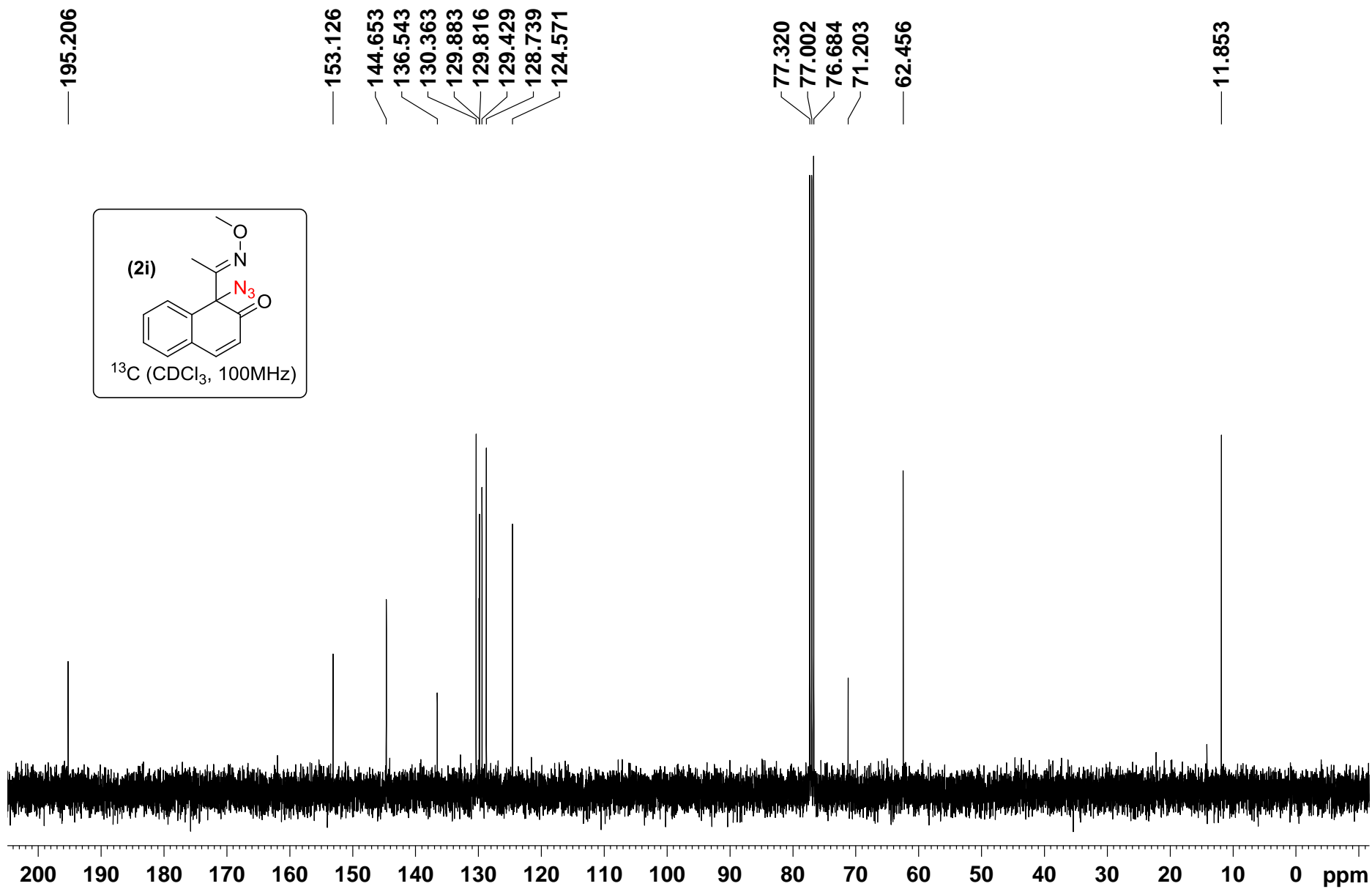
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129.516
129.369
128.196
124.143

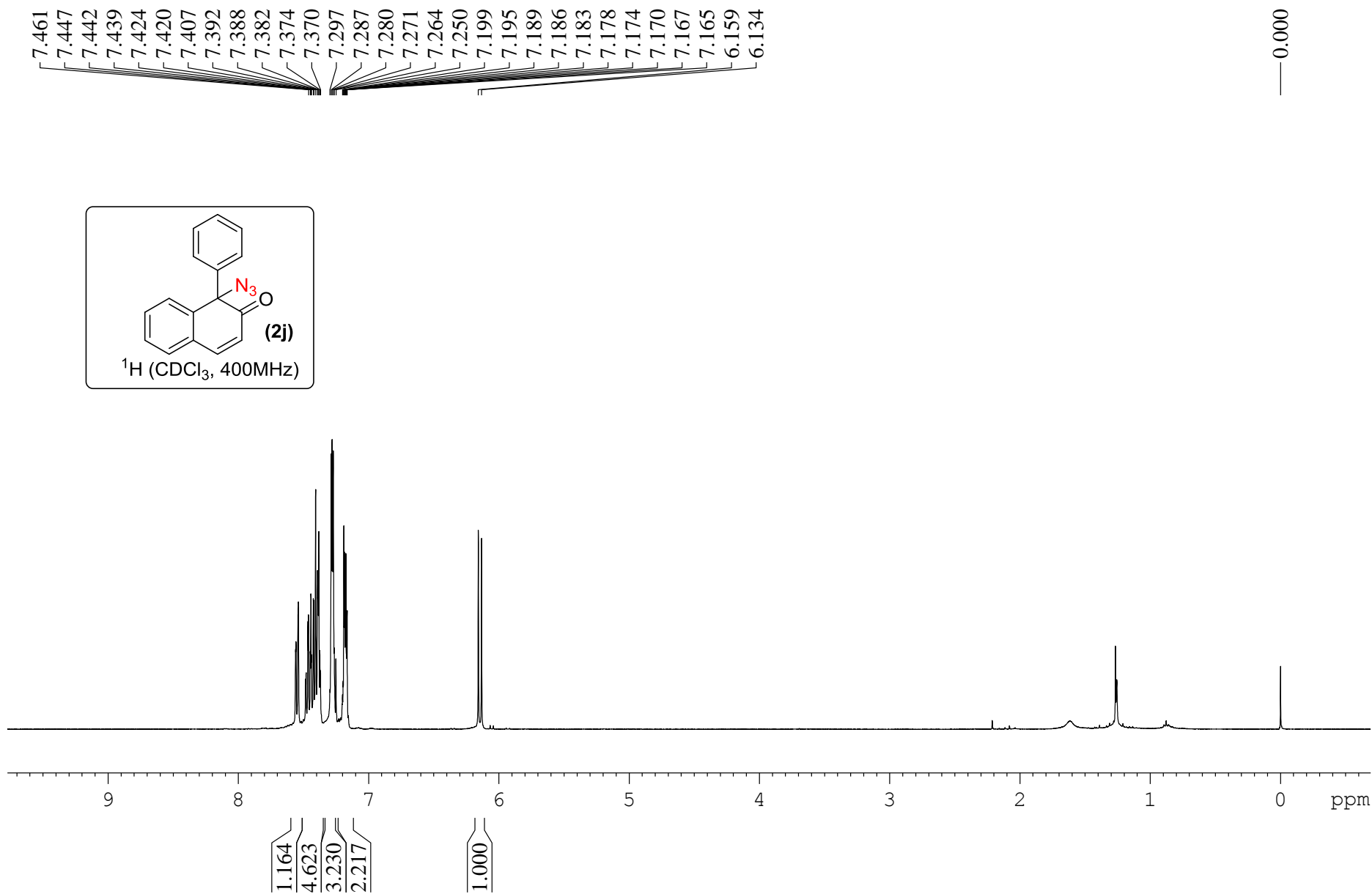
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76.681
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62.591

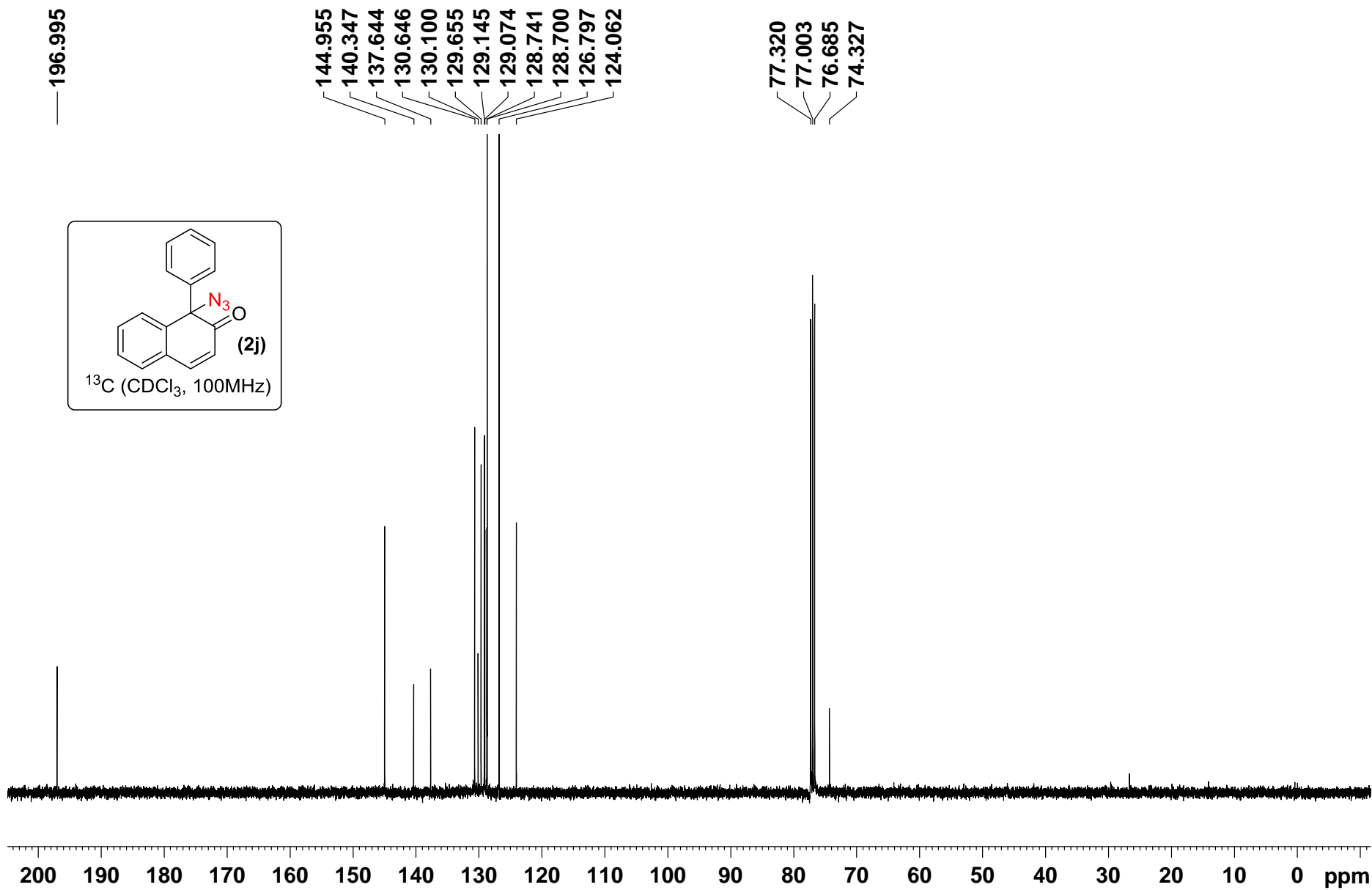


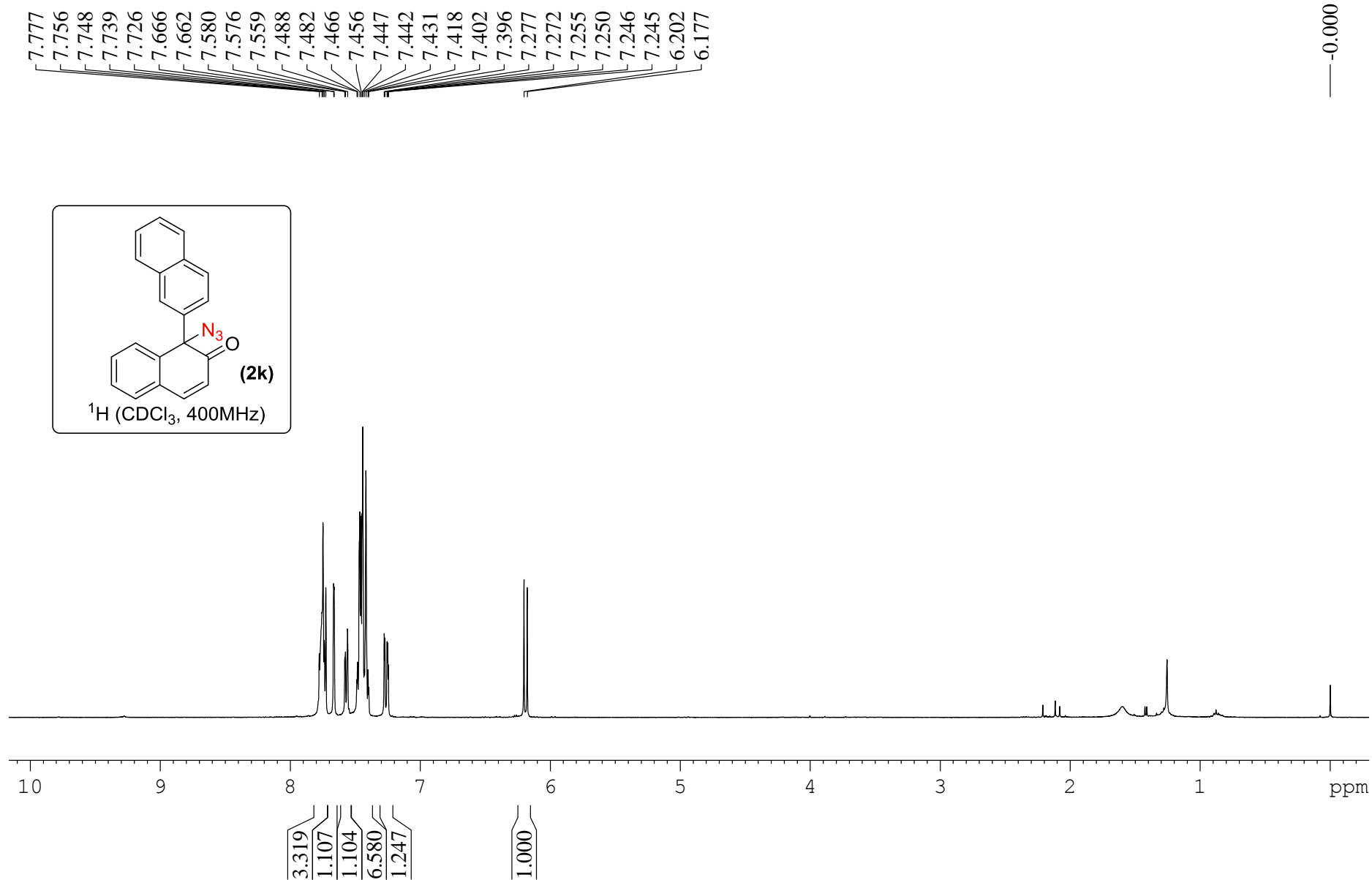
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7.534
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7.430
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7.411
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7.389
7.381
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7.370
7.340
7.322
7.318
7.257
6.253
6.228

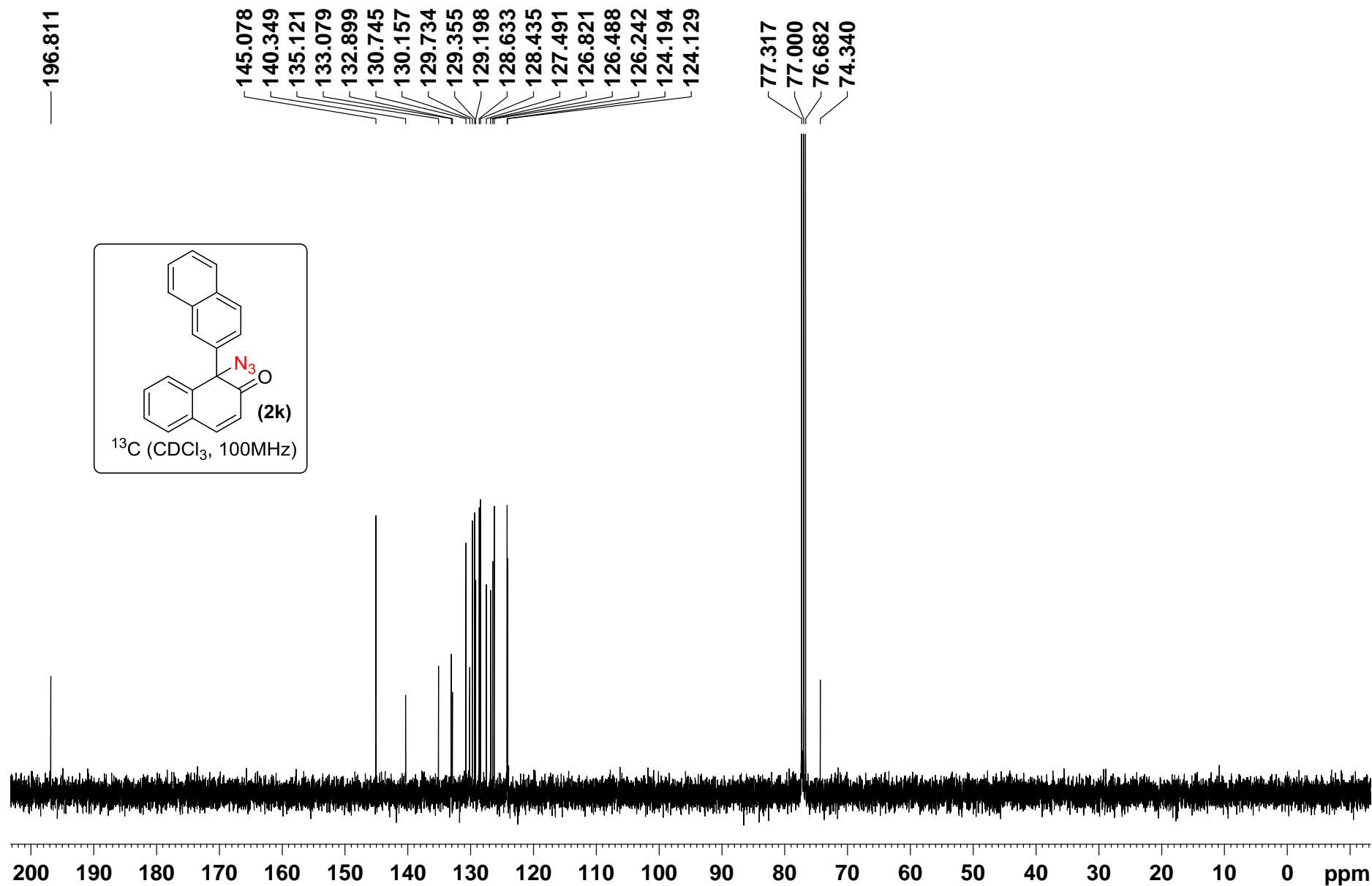








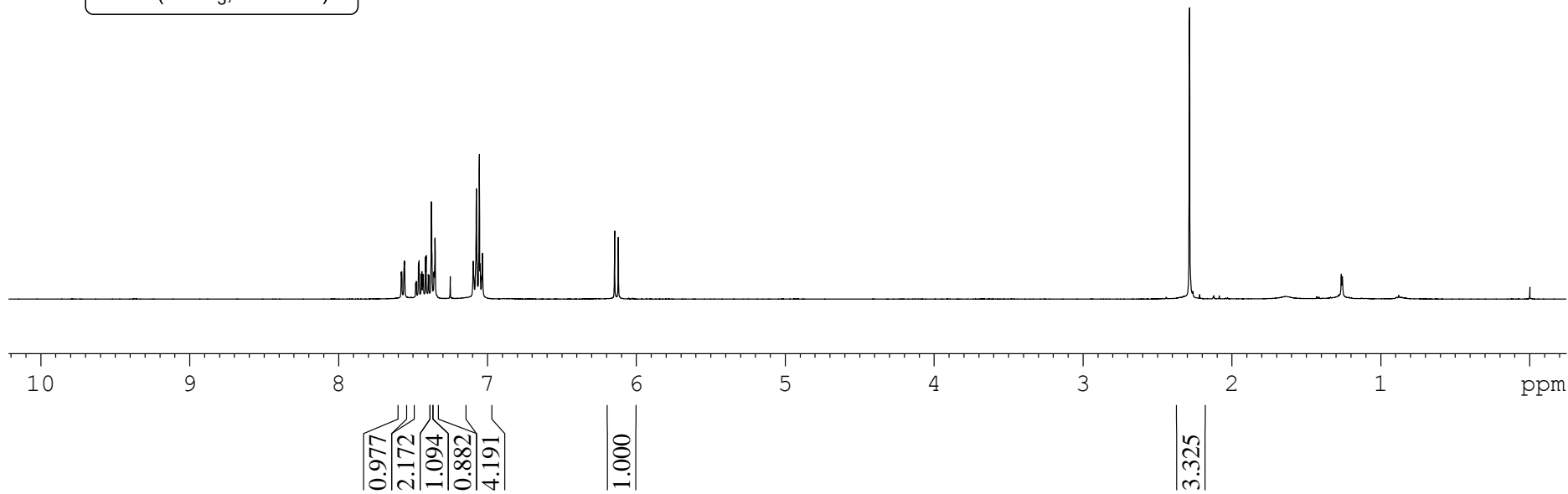
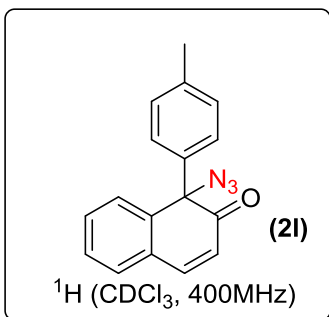




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7.434
7.431
7.416
7.412
7.398
7.394
7.377
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7.095
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7.049
7.039
7.033
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6.121

2.285

0.000

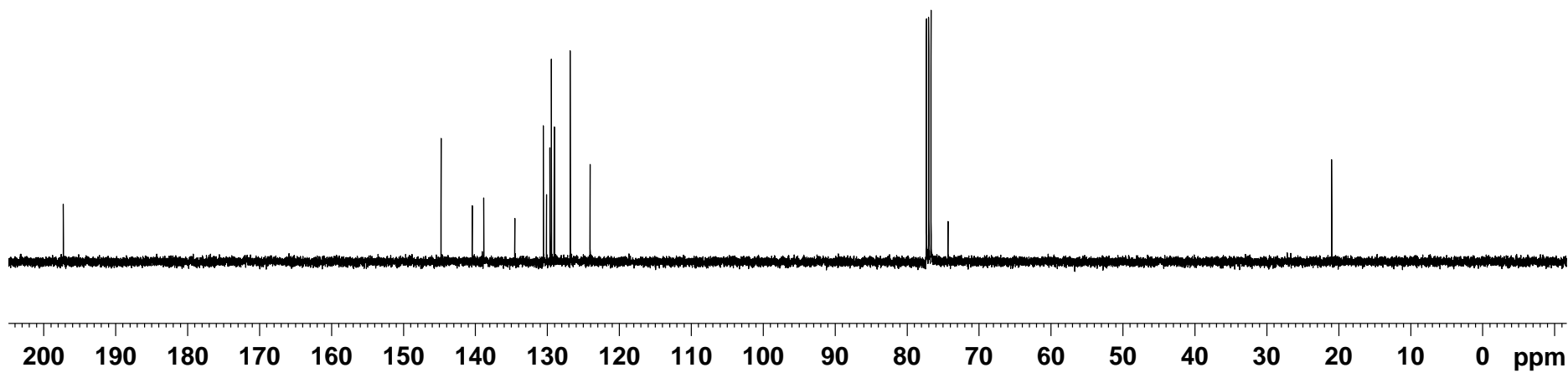
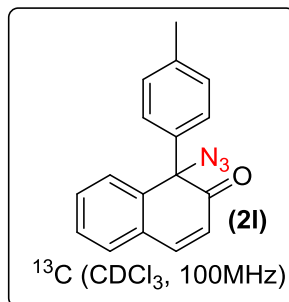


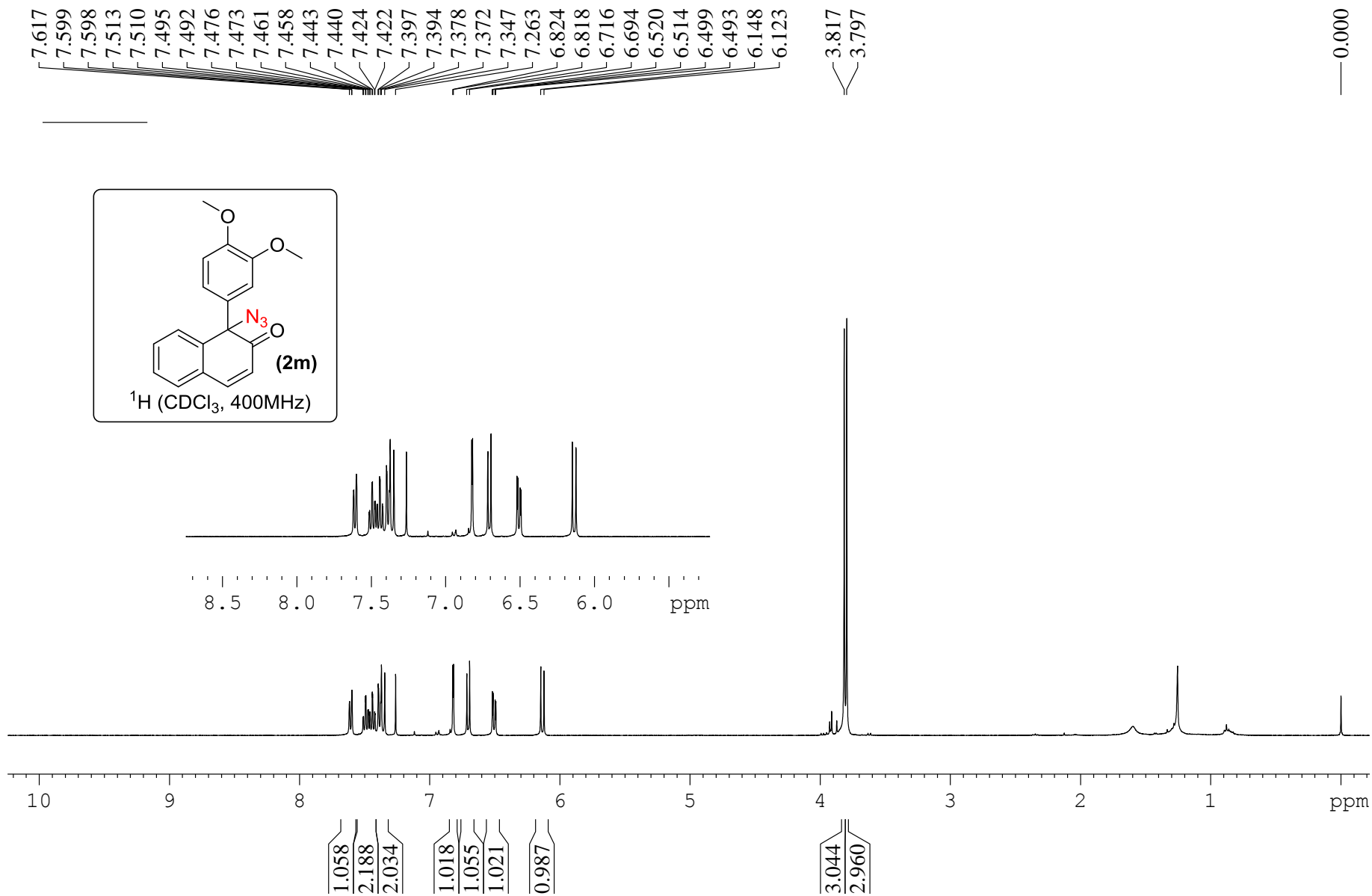
— 197.250

144.752
140.414
138.818
134.493
130.530
130.085
129.619
129.409
129.034
128.970
126.791
124.062

77.318
77.001
76.683
74.314

— 20.983



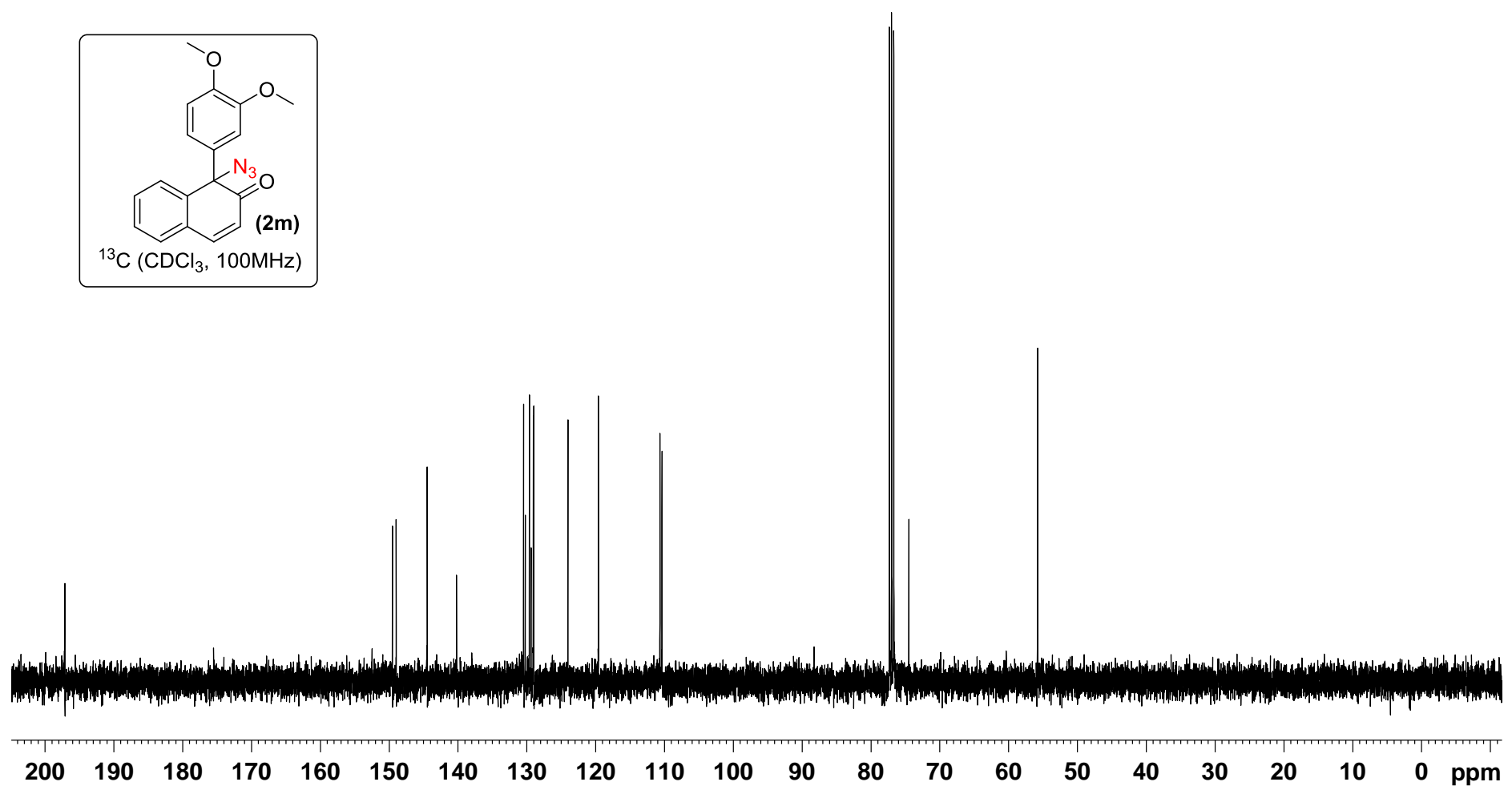
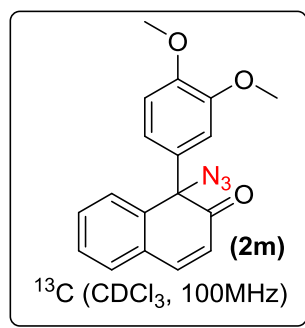


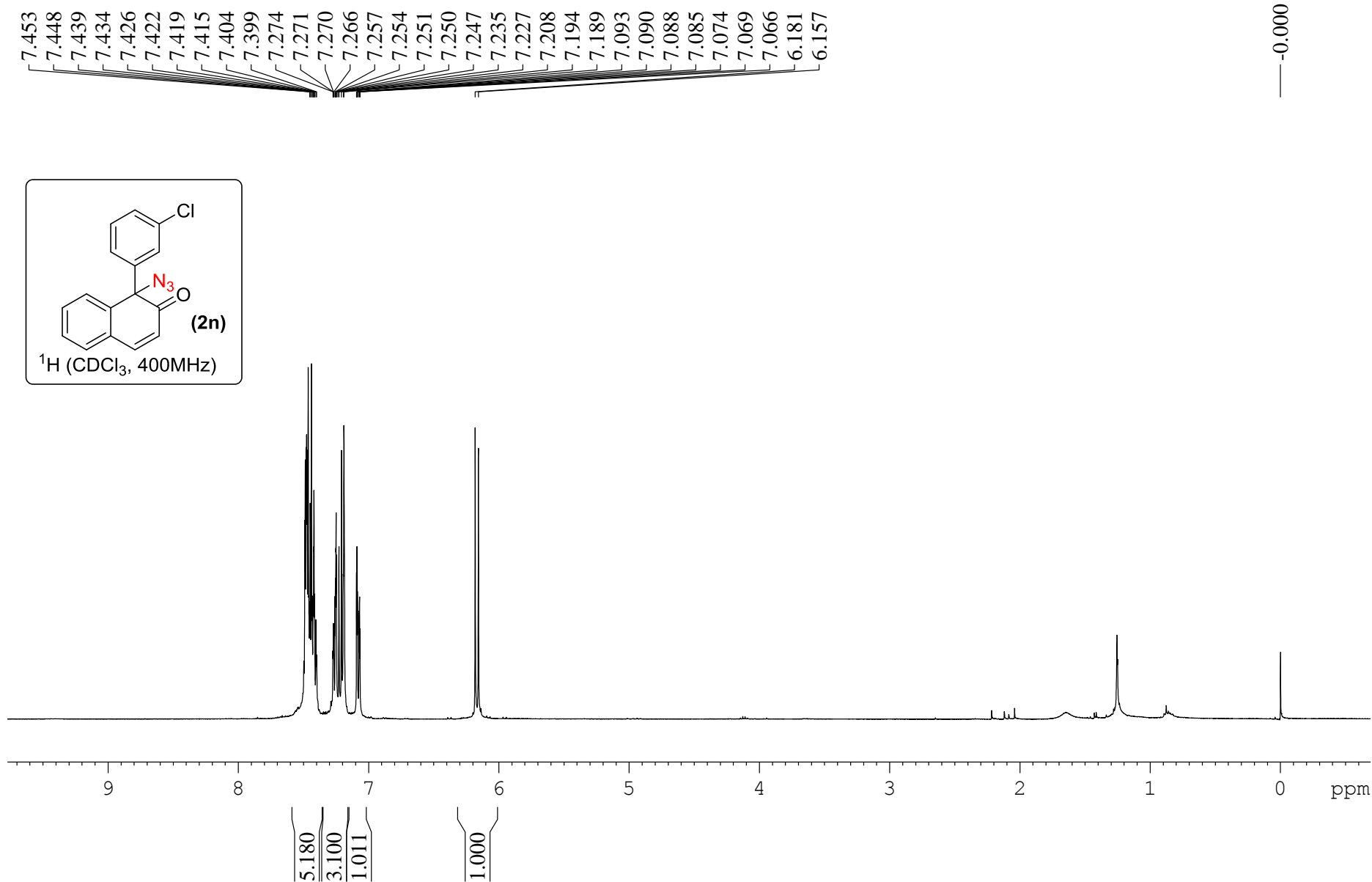
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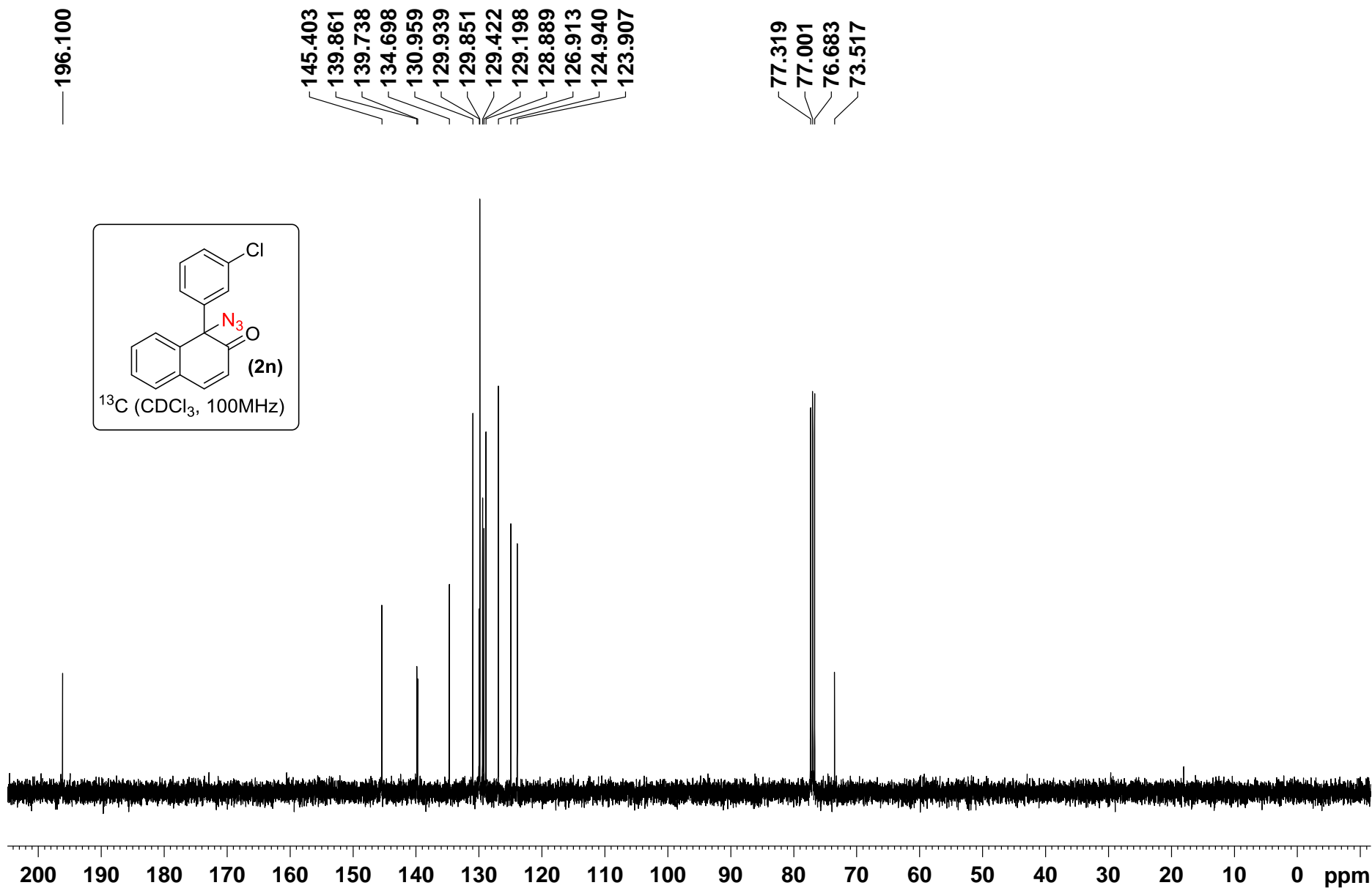
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129.381
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128.995
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110.656
110.357

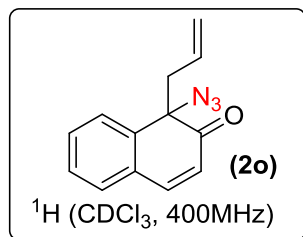
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77.000
76.682
74.483

55.813
55.786



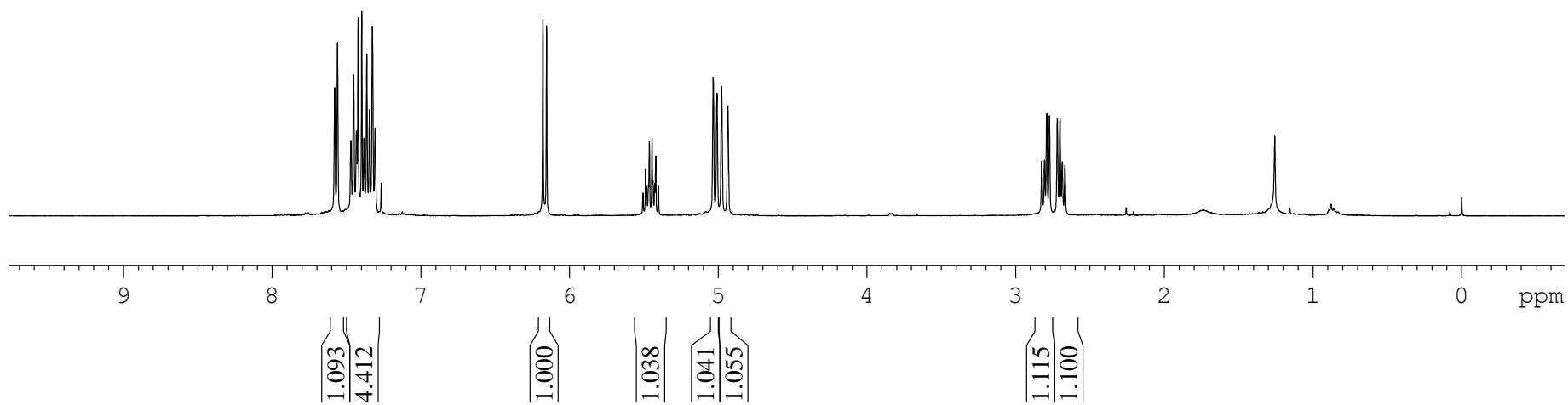


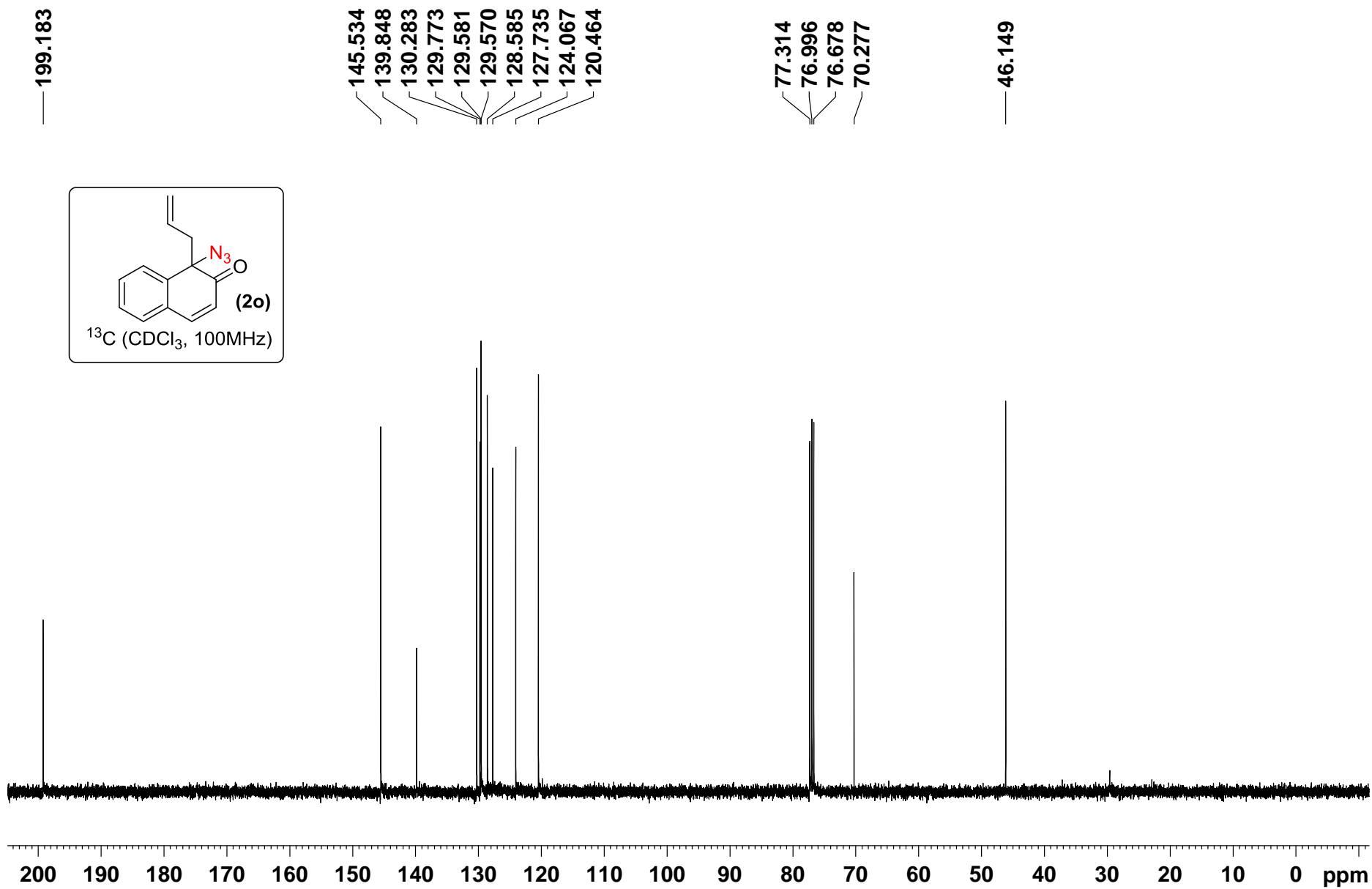


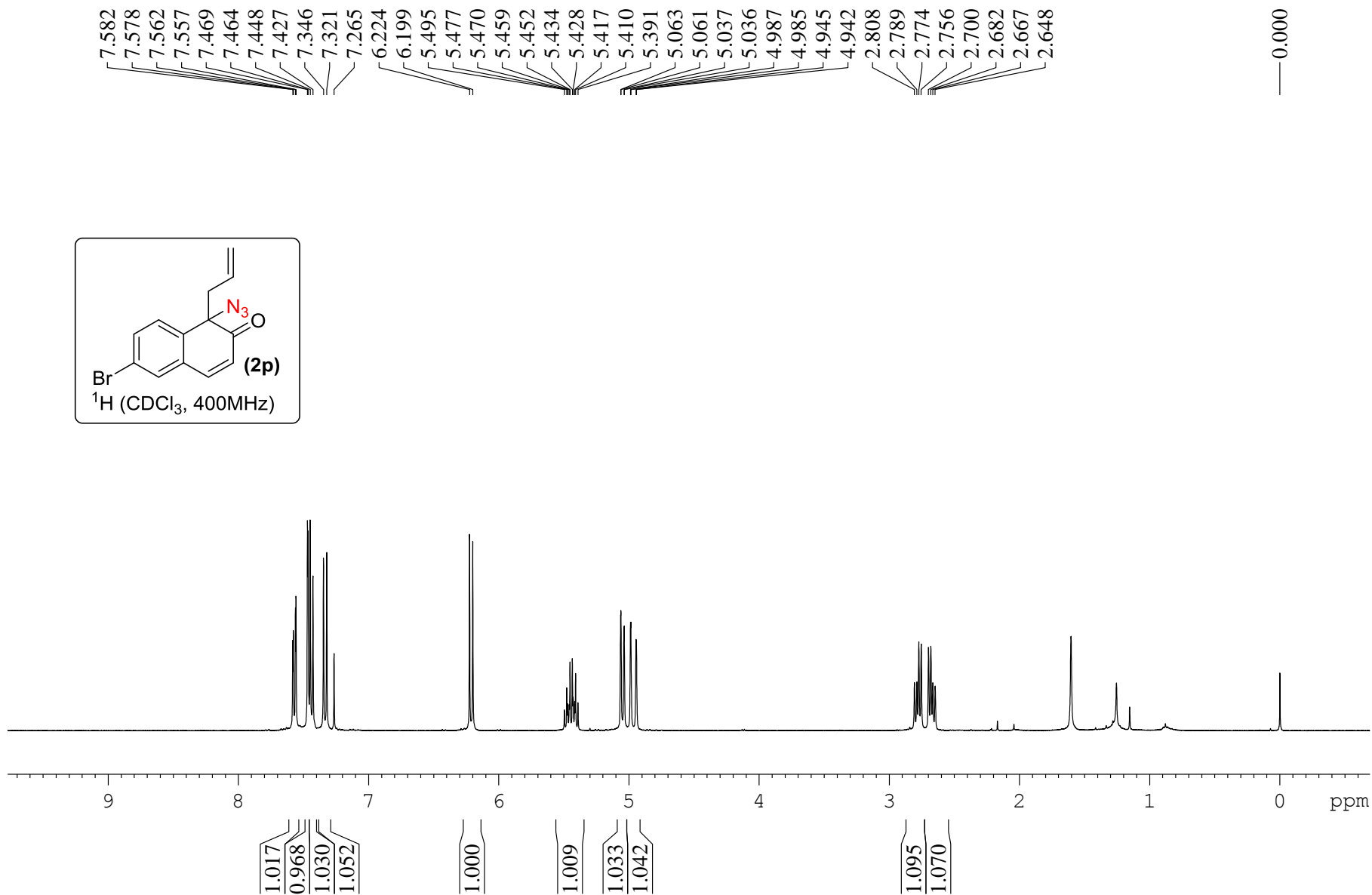
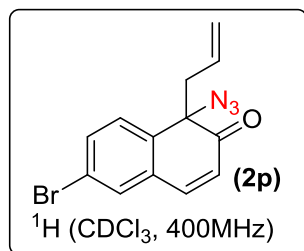


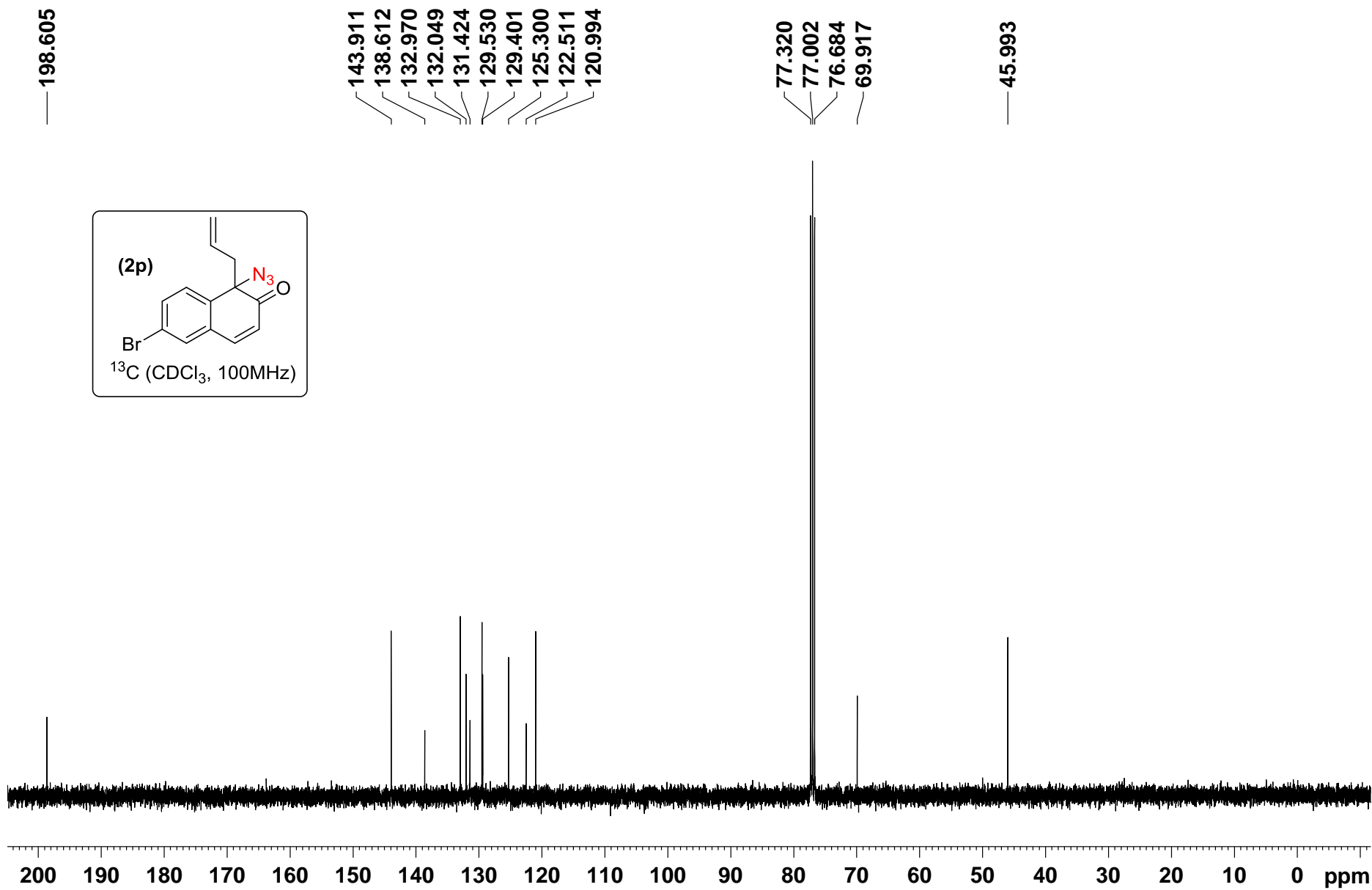
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7.266
6.179
6.154
5.506
5.487
5.480
5.469
5.463
5.445
5.438
5.427
5.420
5.402
5.033
5.008
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2.825
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2.792
2.773
2.720
2.702
2.687
2.669

0.000



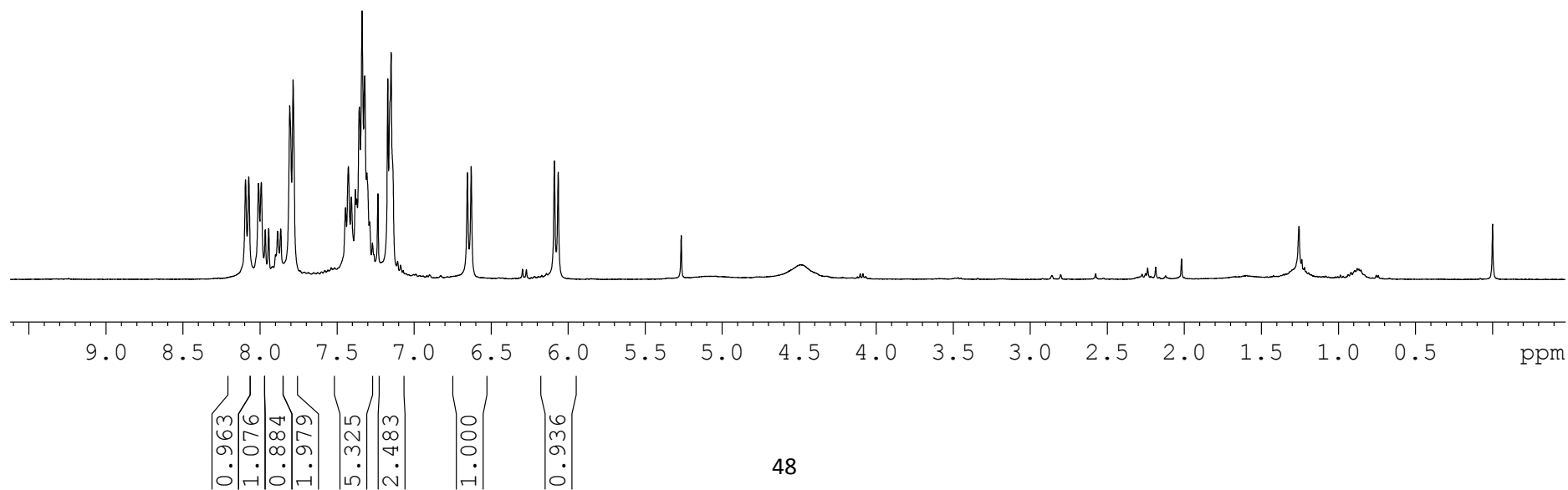
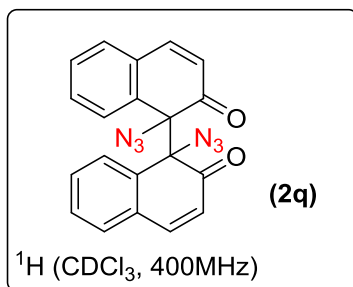


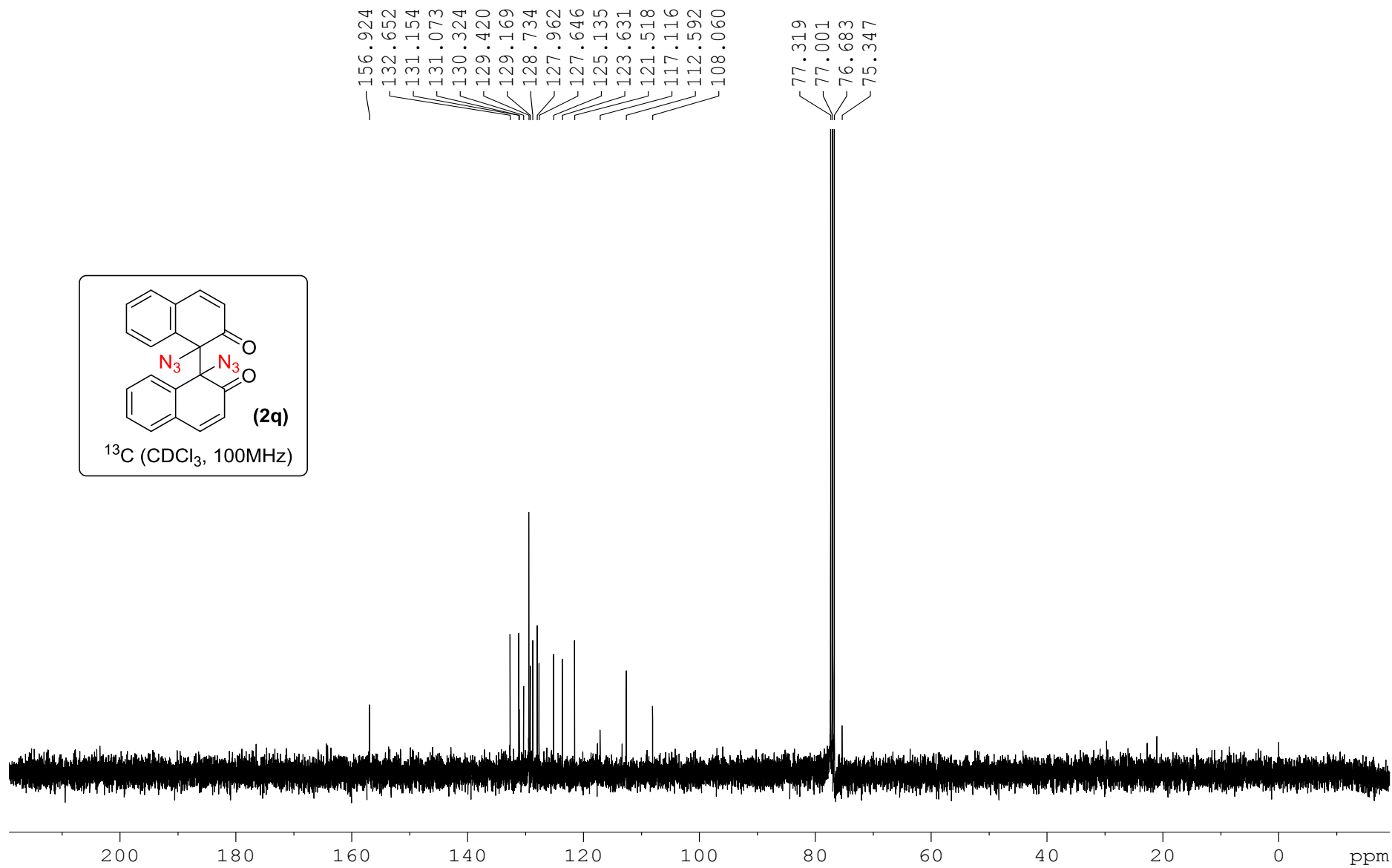
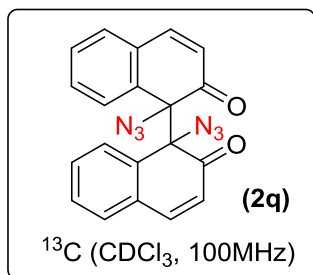


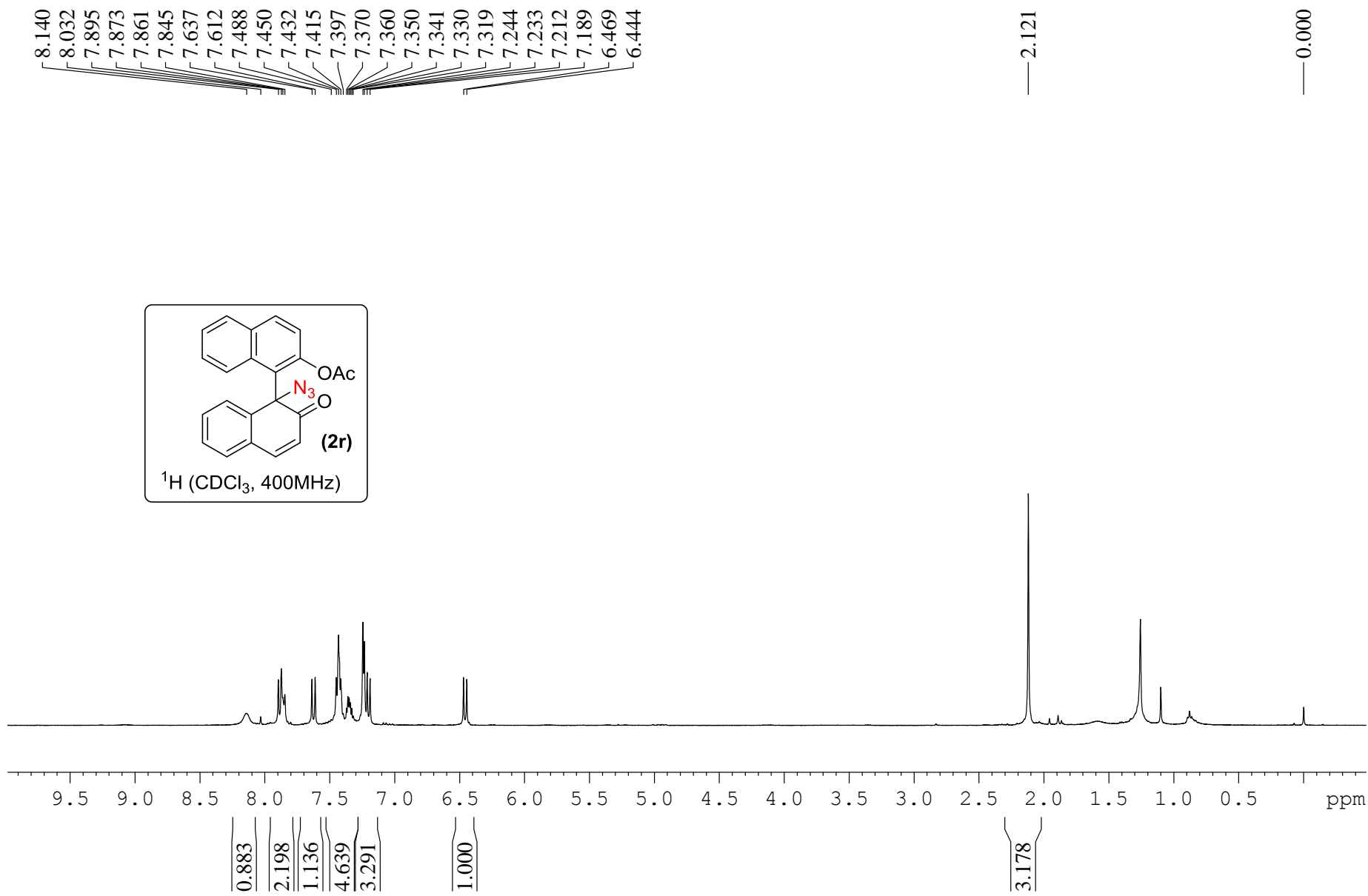


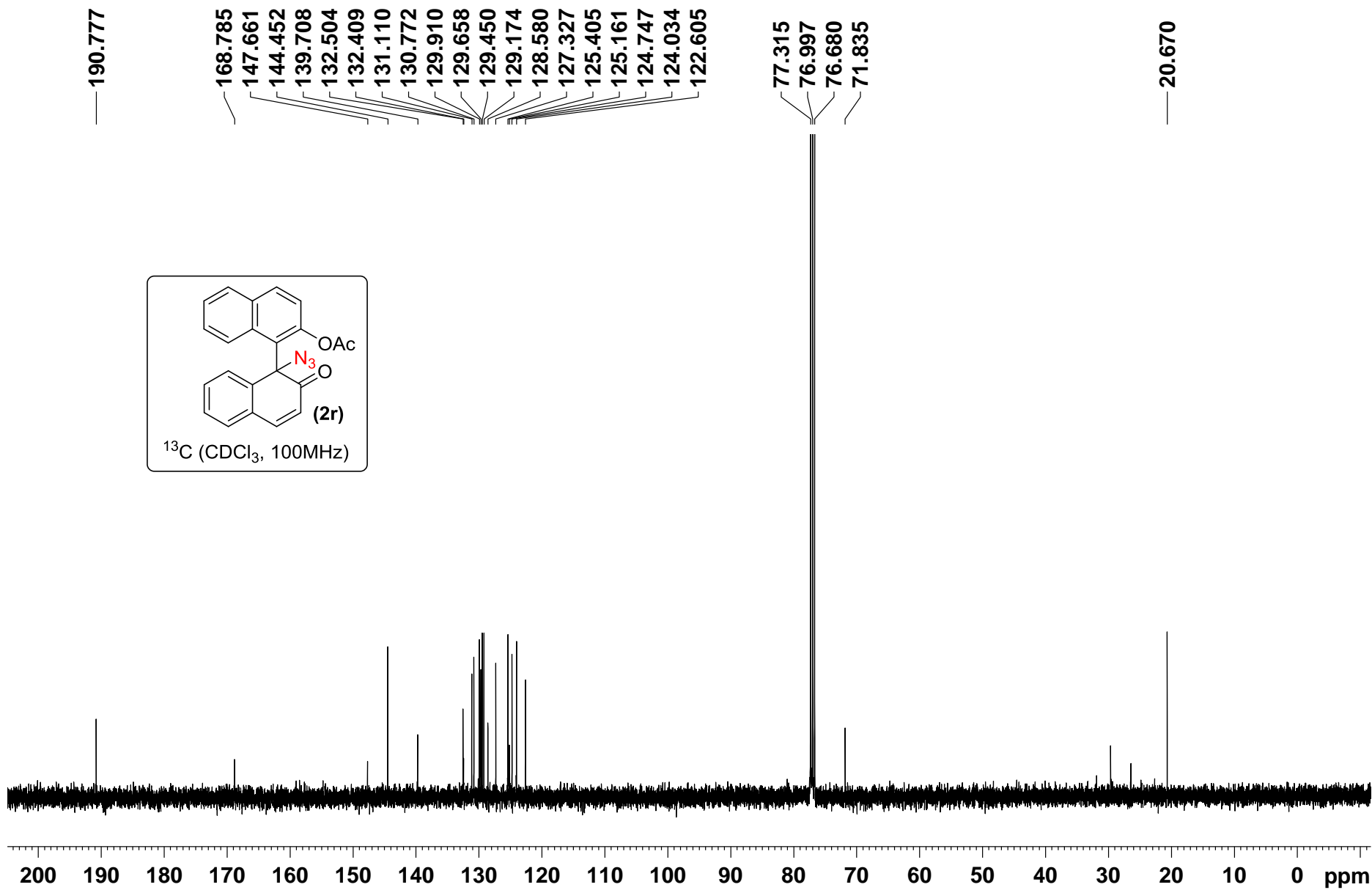
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7.865
7.806
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7.444
7.426
7.407
7.381
7.372
7.354
7.337
7.320
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7.287
7.270
7.234
7.170
7.148
6.653
6.629
6.089
6.064

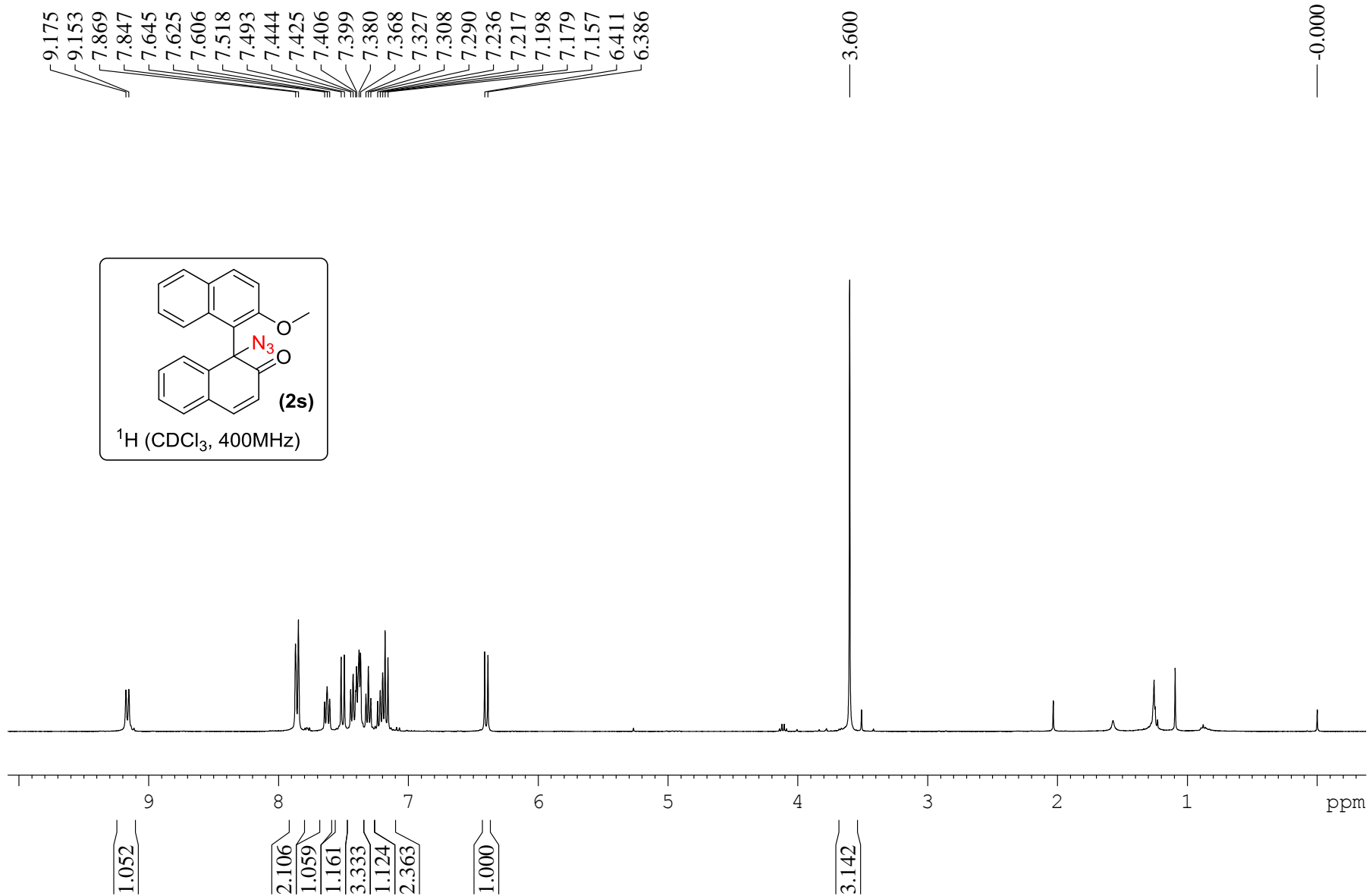
— 0.000

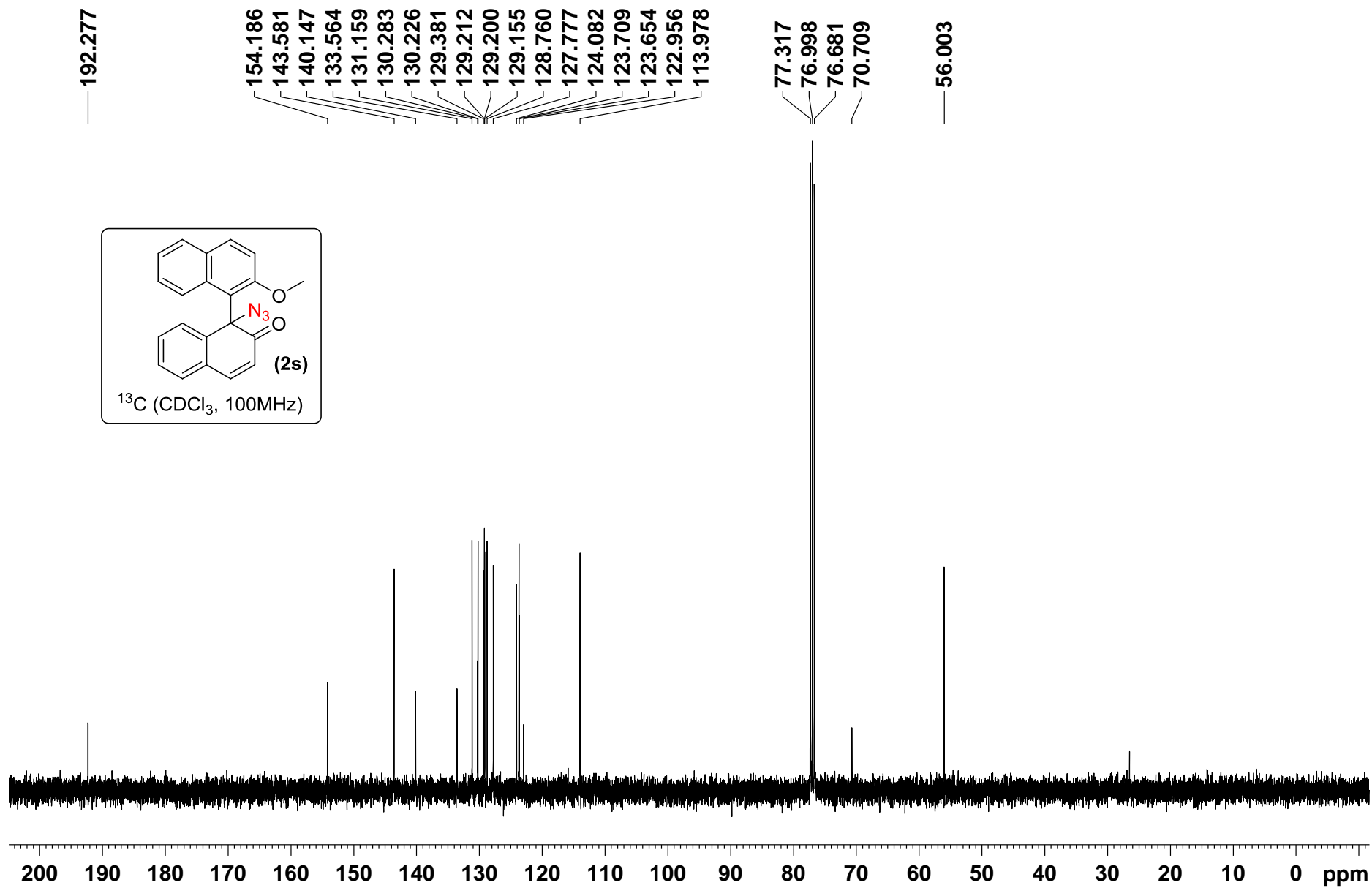


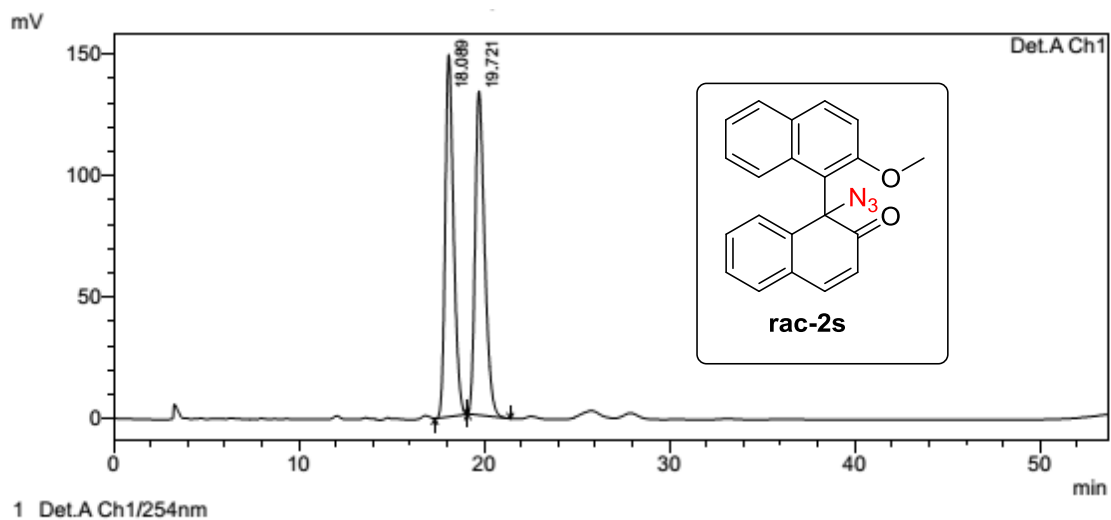










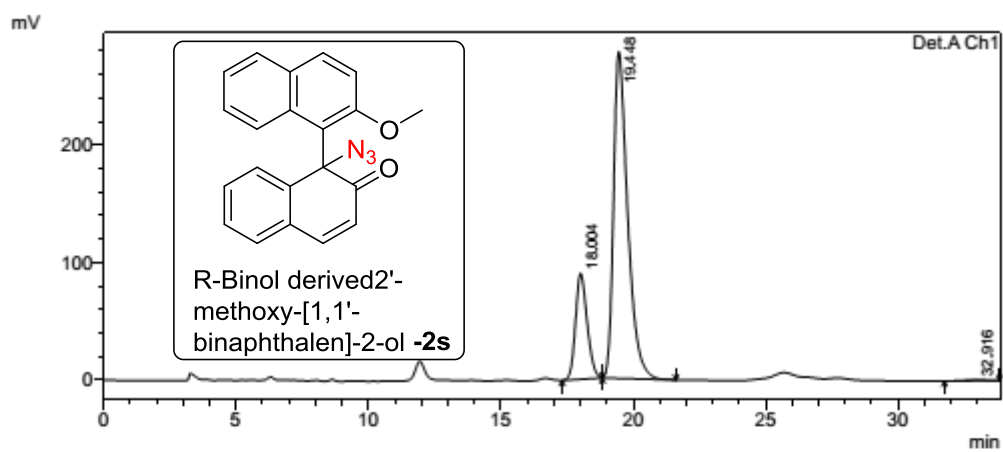


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Area %
1	18.089	4673609	49.548
2	19.721	4758820	50.452
Total		9432429	100.000

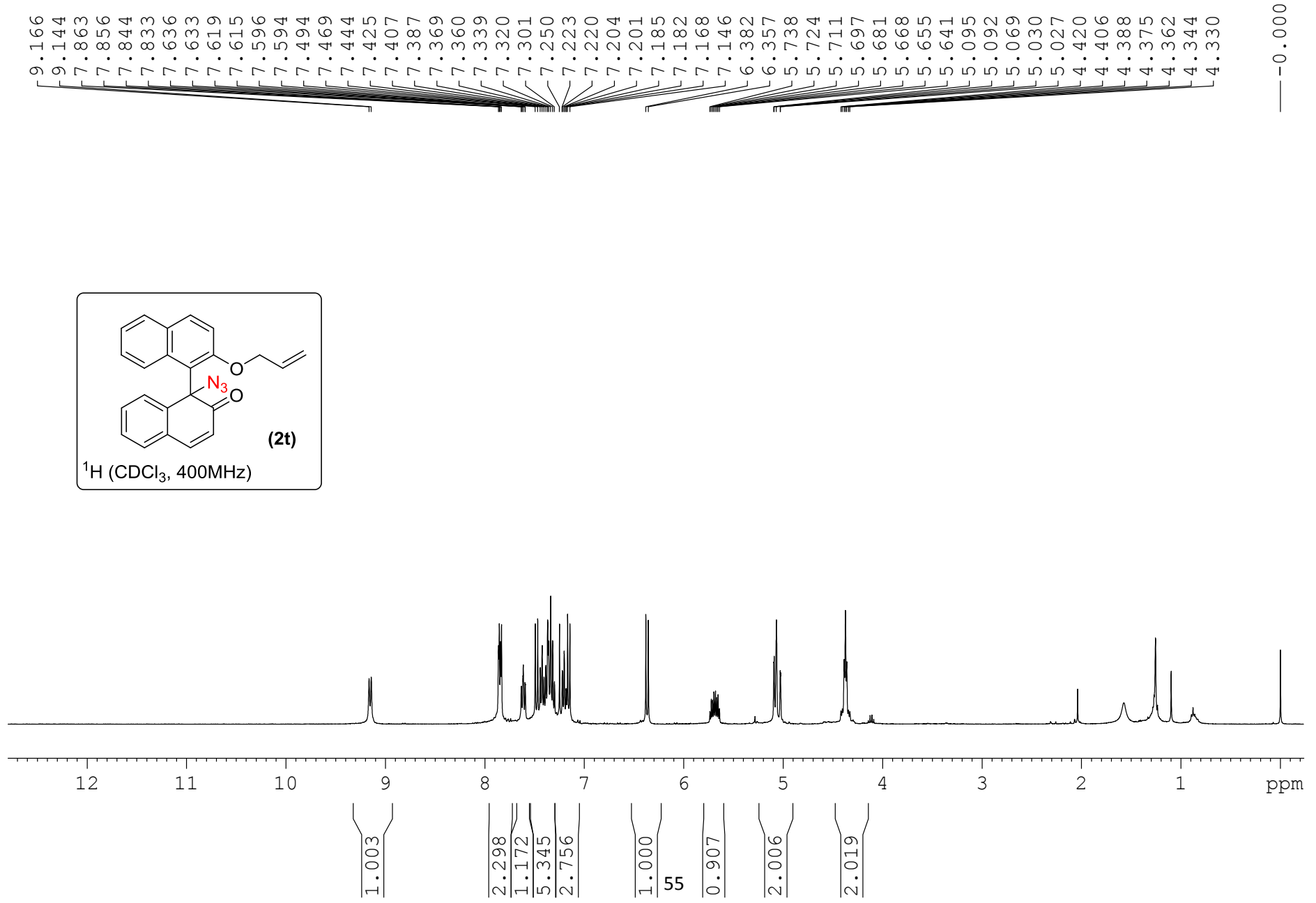
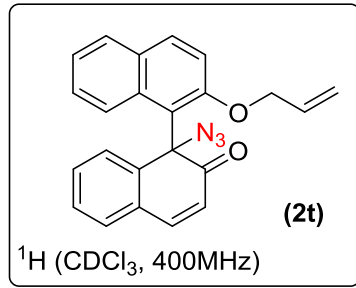


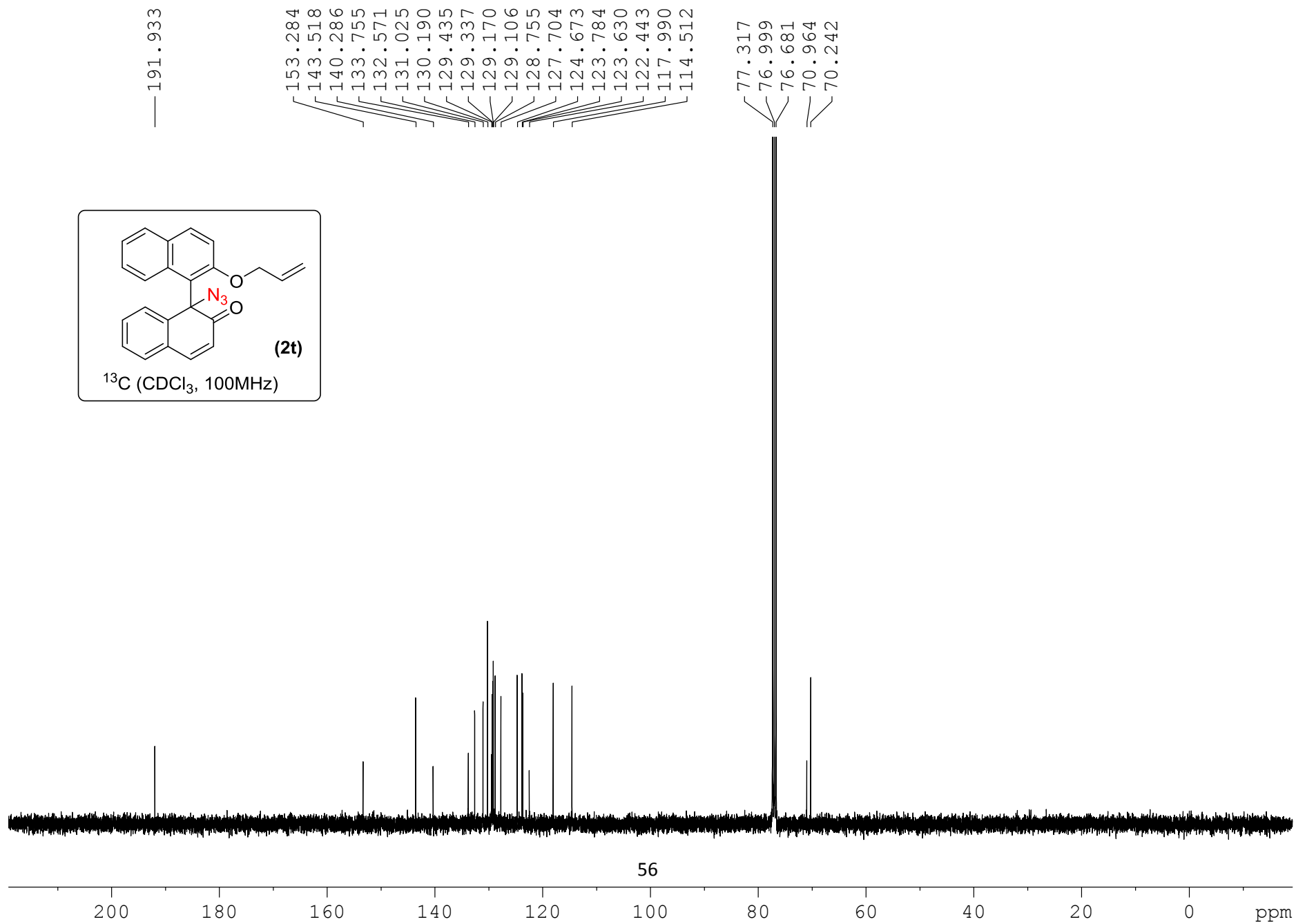
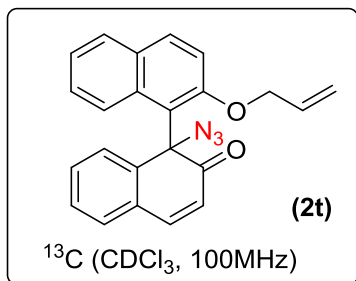
1 Det.A Ch1/254nm

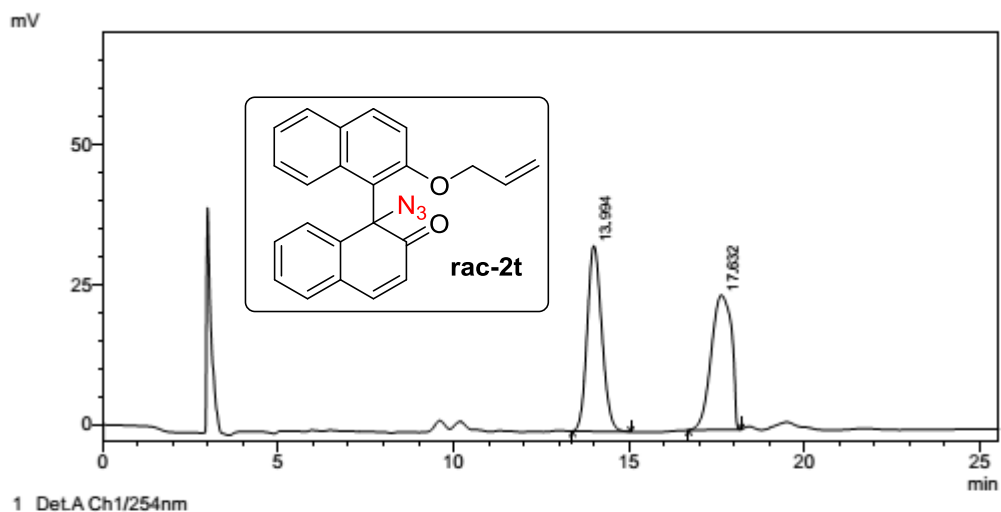
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Area %
1	18.004	2737364	20.962
2	19.448	10274842	78.680
3	32.916	46758	0.358
Total		13058964	100.000



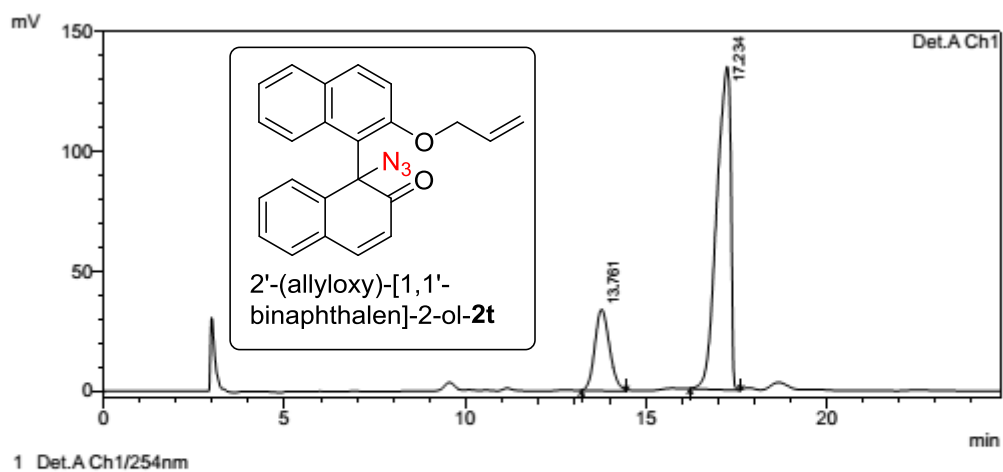




PeakTable

Detector A Ch1 254nm

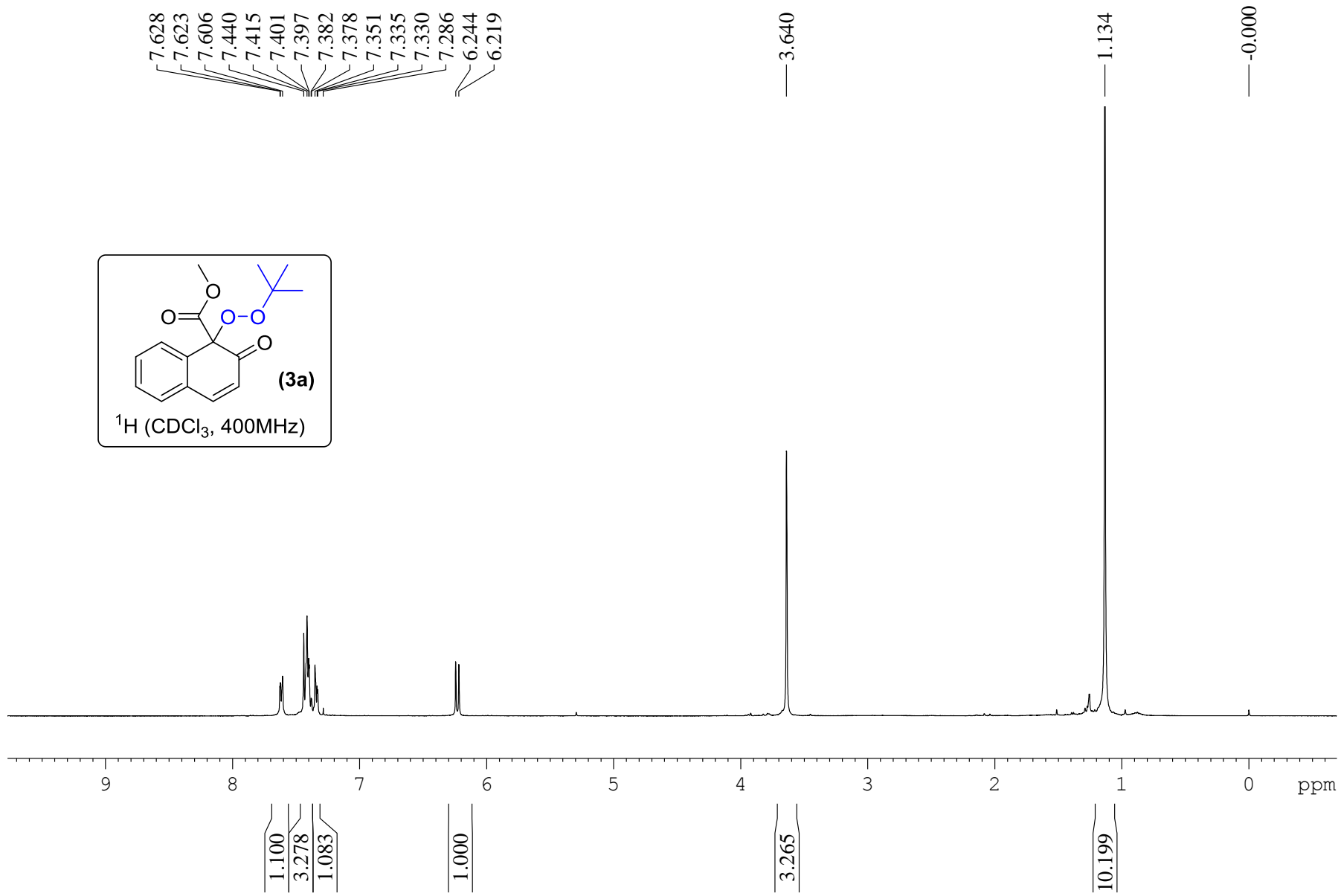
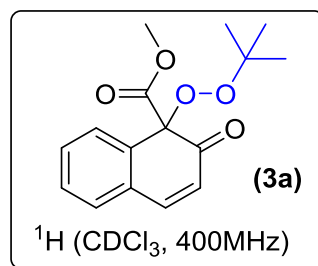
Peak#	Ret. Time	Area	Area %
1	13.994	963917	49.997
2	17.632	964015	50.003
Total		1927932	100.000



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Area %
1	13.761	968056	20.988
2	17.234	3644476	79.012
Total		4612532	100.000



— 193.213

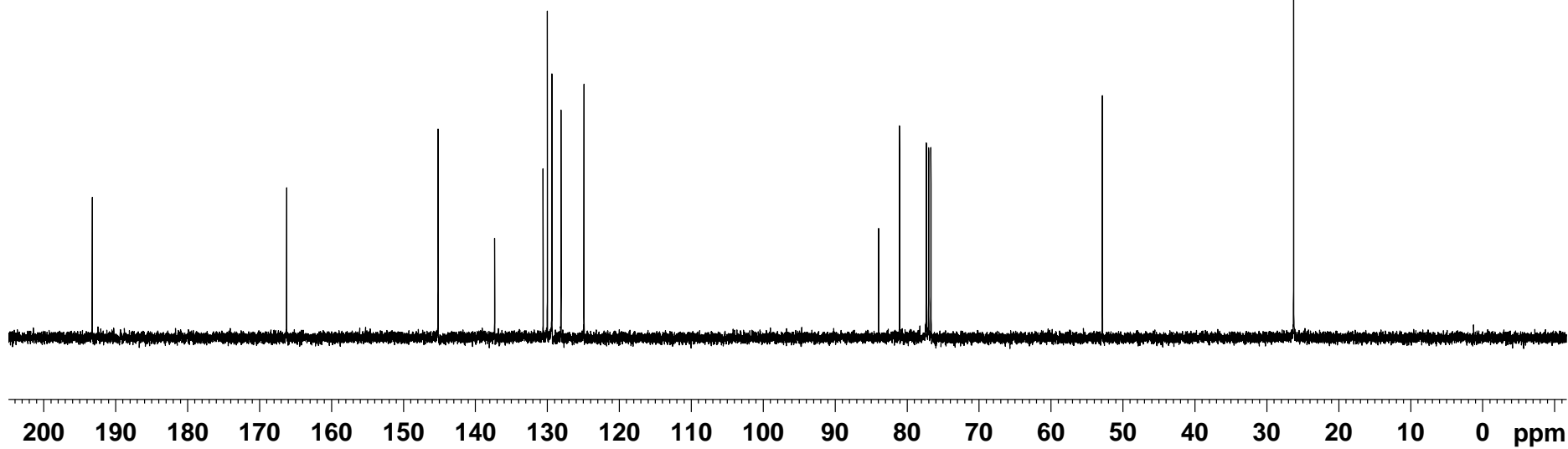
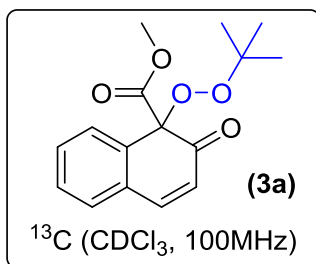
— 166.228

145.183
 137.320
 130.589
 129.999
 129.366
 129.340
 128.099
 124.903

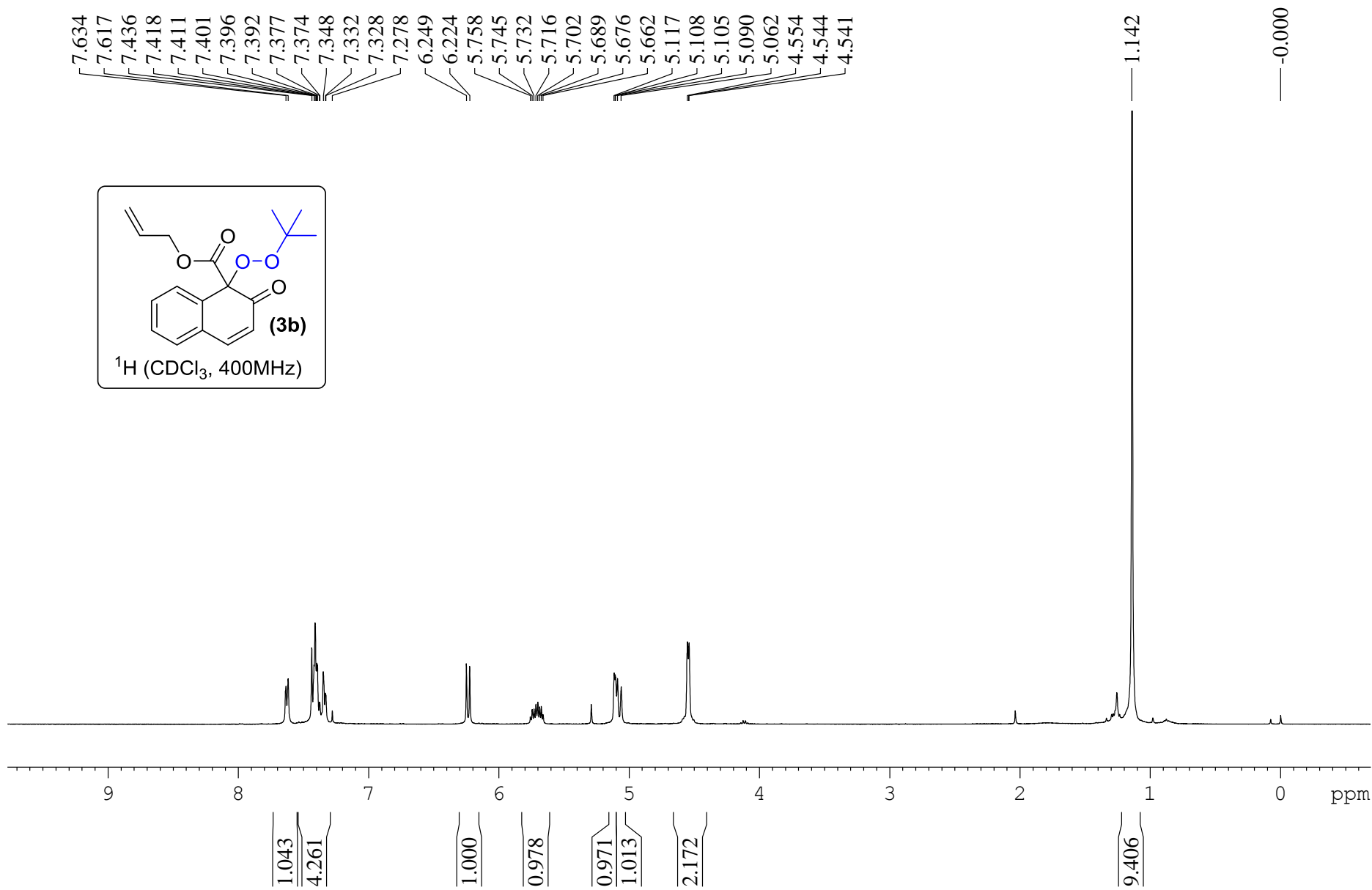
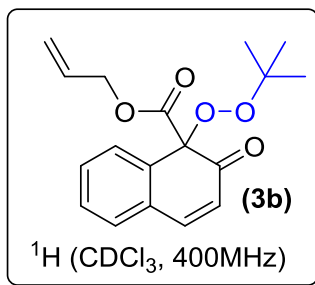
83.966
 81.055
 77.319
 77.001
 76.684

— 52.847

— 26.274



7.634
7.617
7.436
7.418
7.411
7.401
7.396
7.392
7.377
7.374
7.348
7.332
7.328
7.278
6.249
6.224
5.758
5.745
5.732
5.716
5.702
5.689
5.676
5.662
5.117
5.108
5.105
5.090
5.062
4.554
4.544
4.541



193.100

165.326

145.113

137.395

130.730

130.634

129.955

129.357

129.311

128.082

124.977

118.183

84.040

81.058

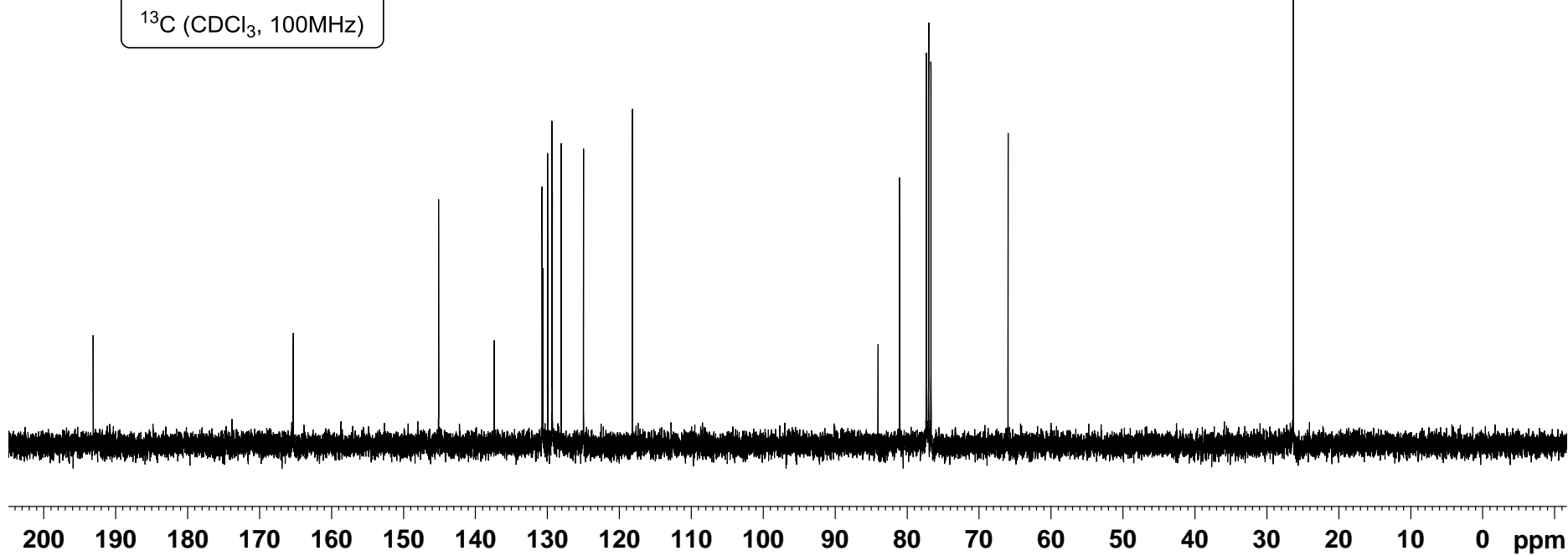
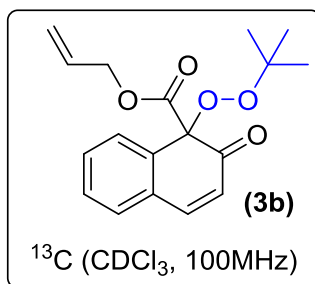
77.316

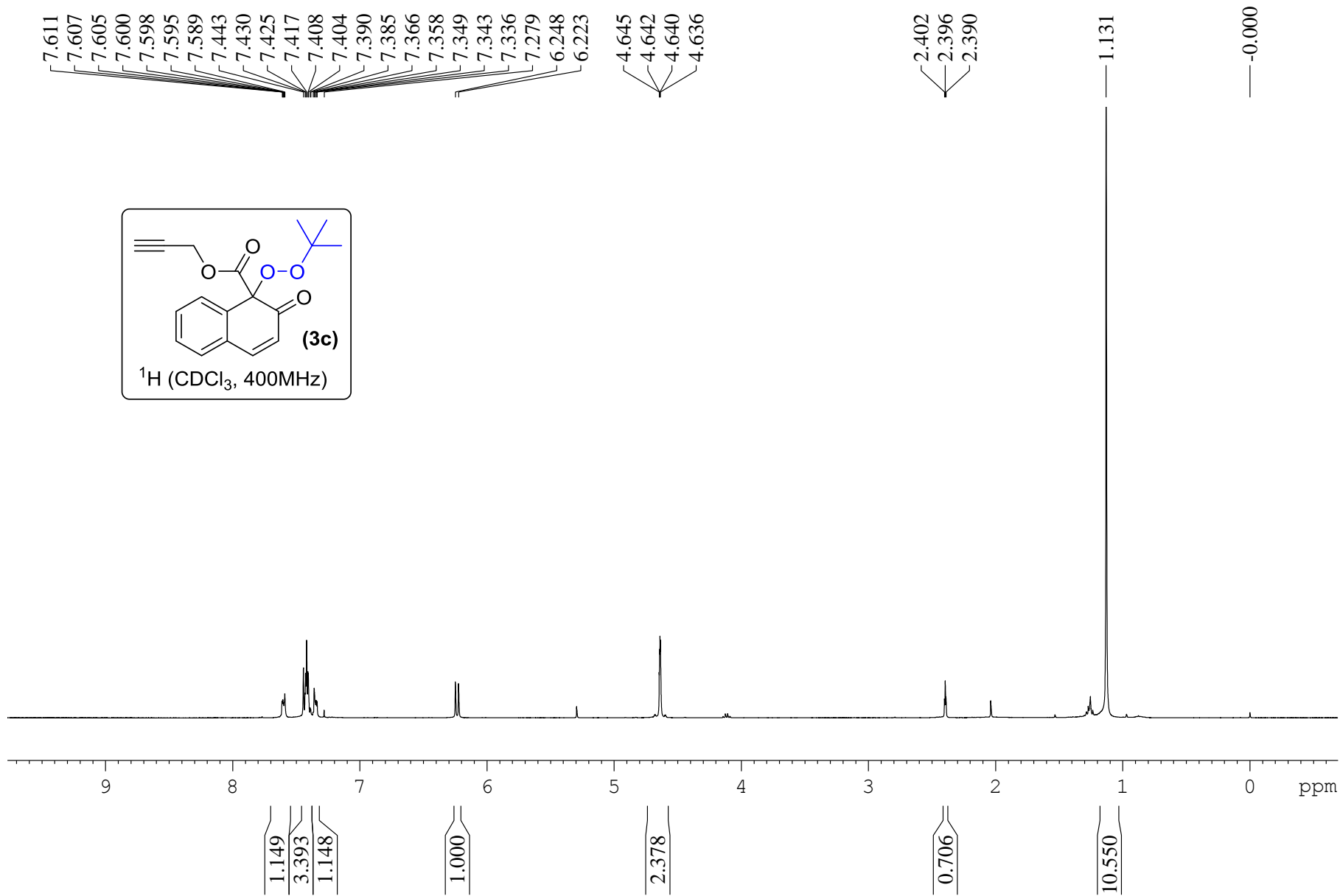
76.998

76.681

65.962

26.305





— 192.659

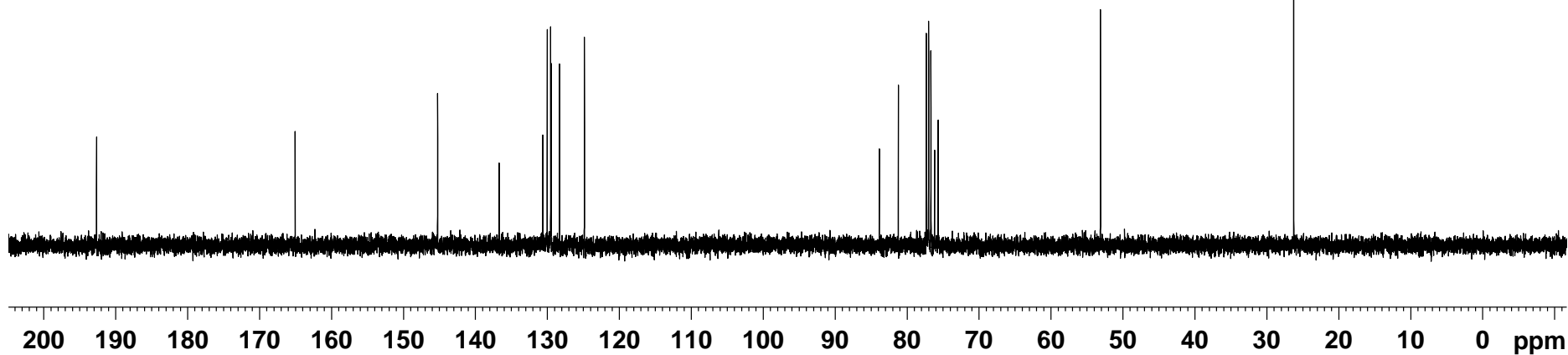
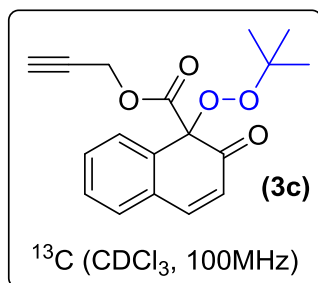
— 165.053

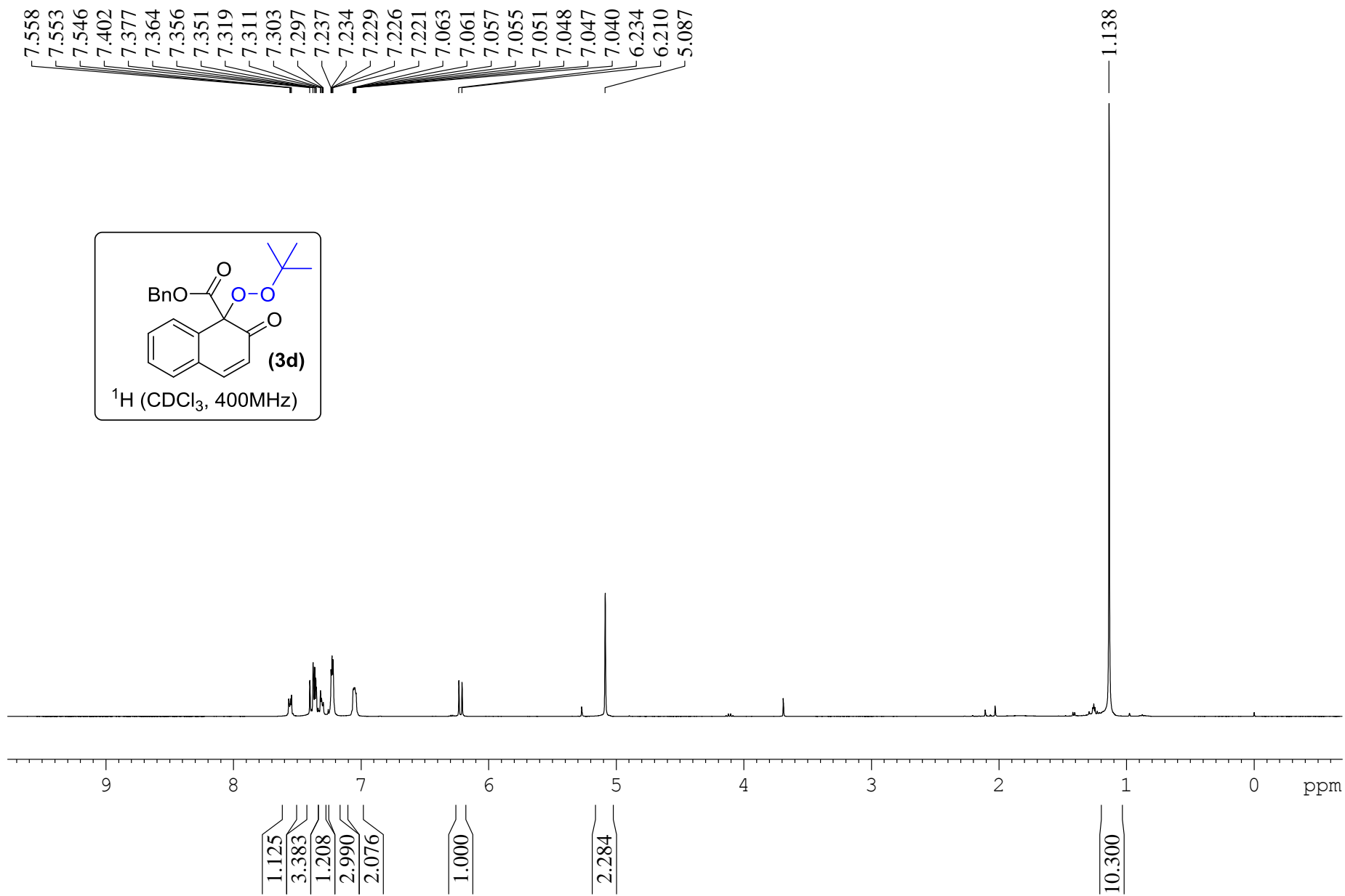
145.248
 136.692
 130.634
 130.014
 129.539
 129.473
 128.303
 124.844

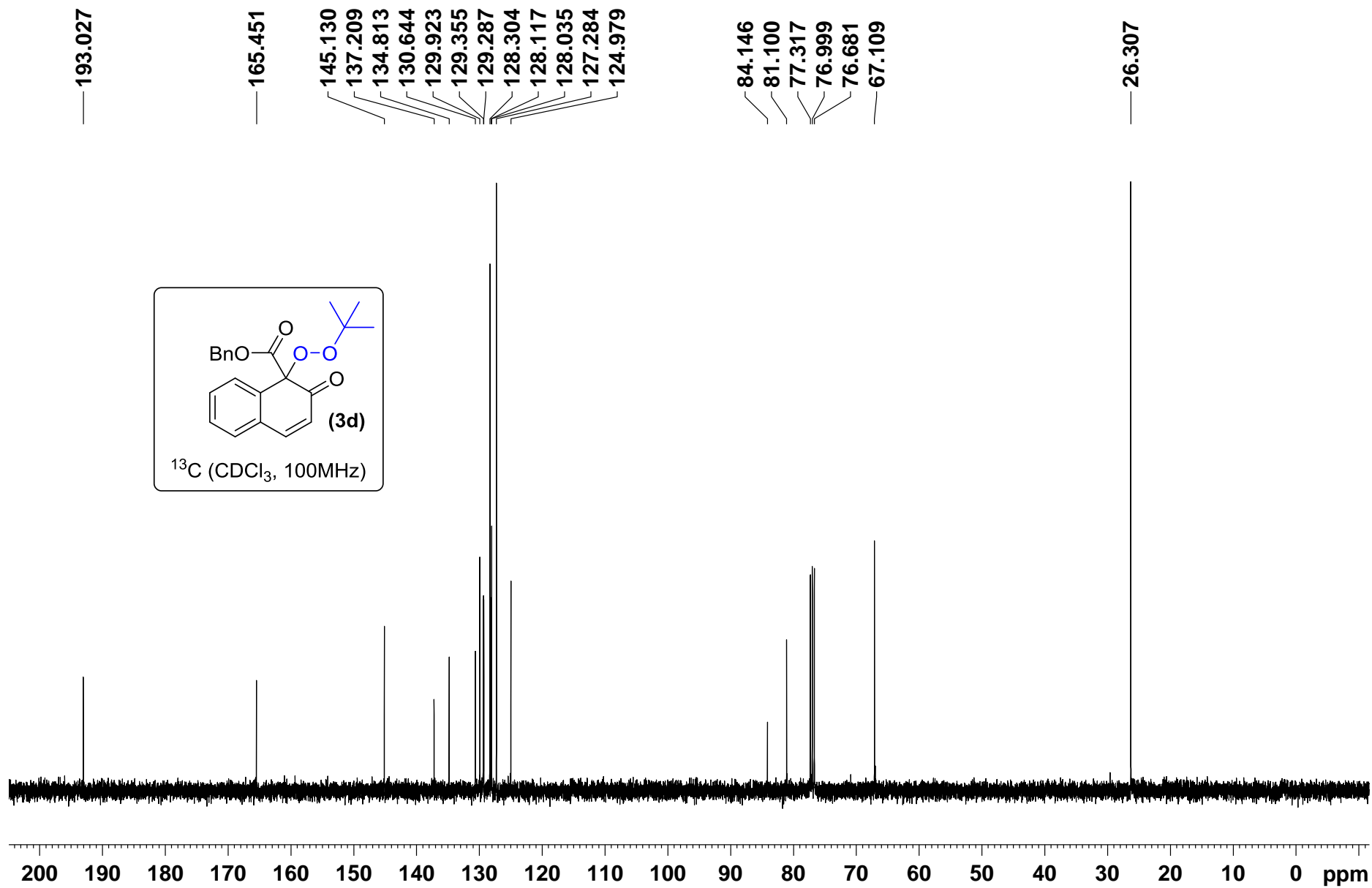
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 77.005
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 76.183
 75.688

— 53.129

— 26.269





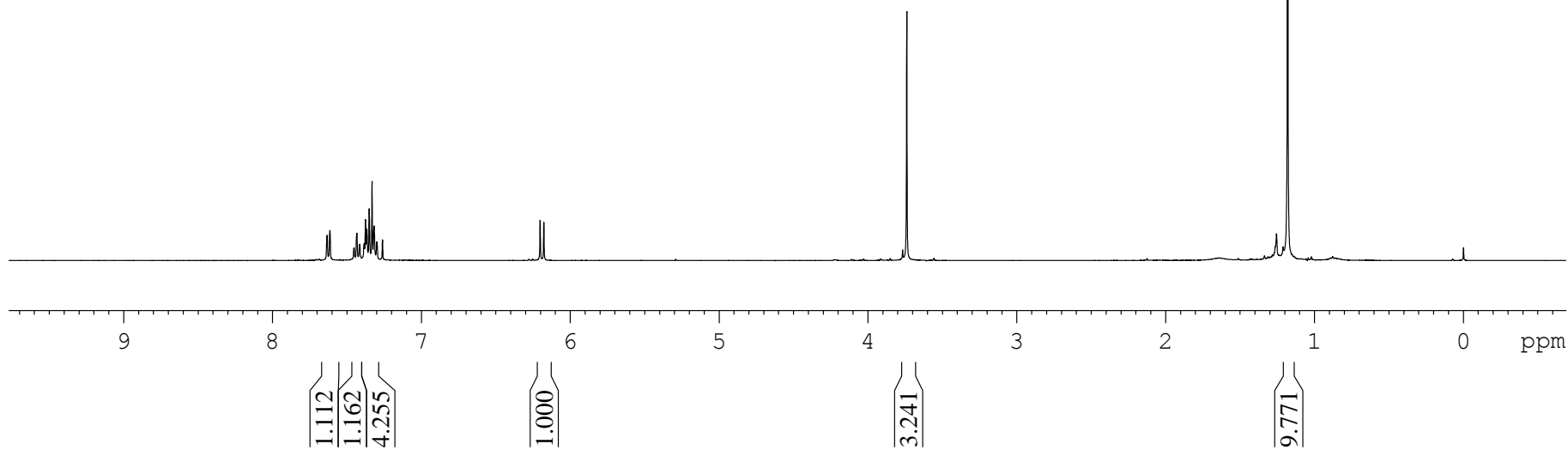
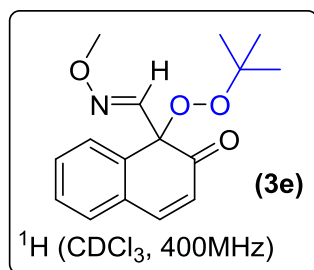


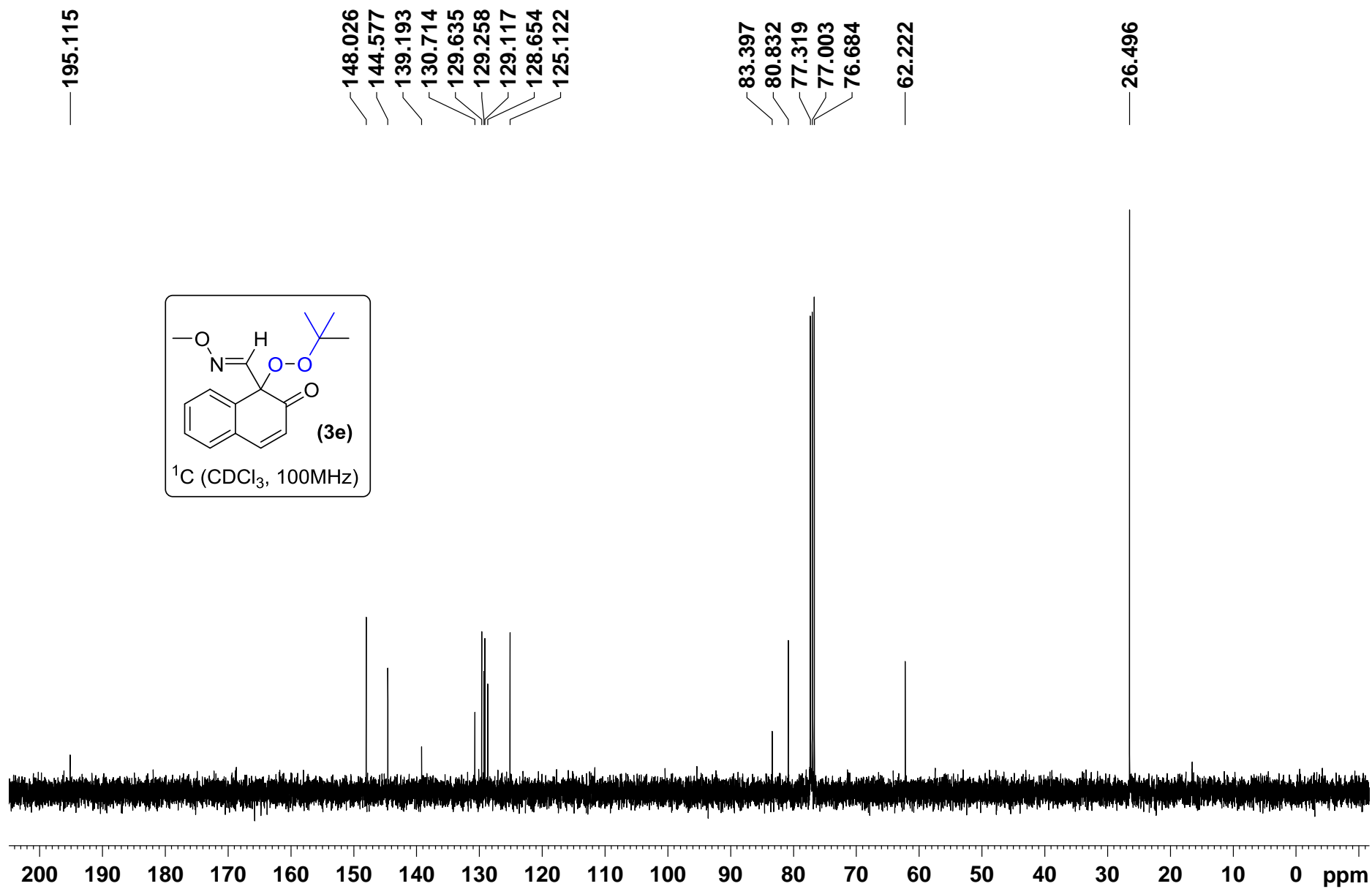
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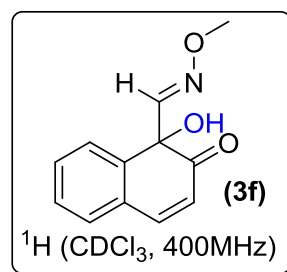
— 3.740

— 1.181

— 0.001



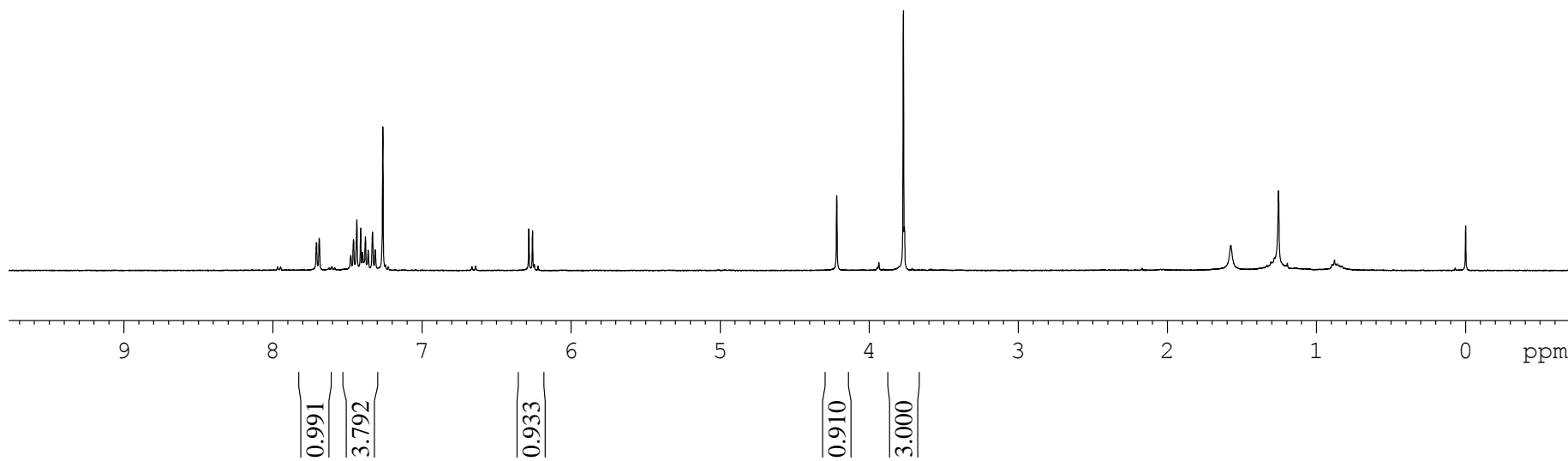


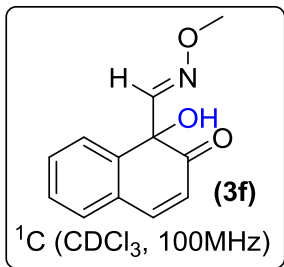
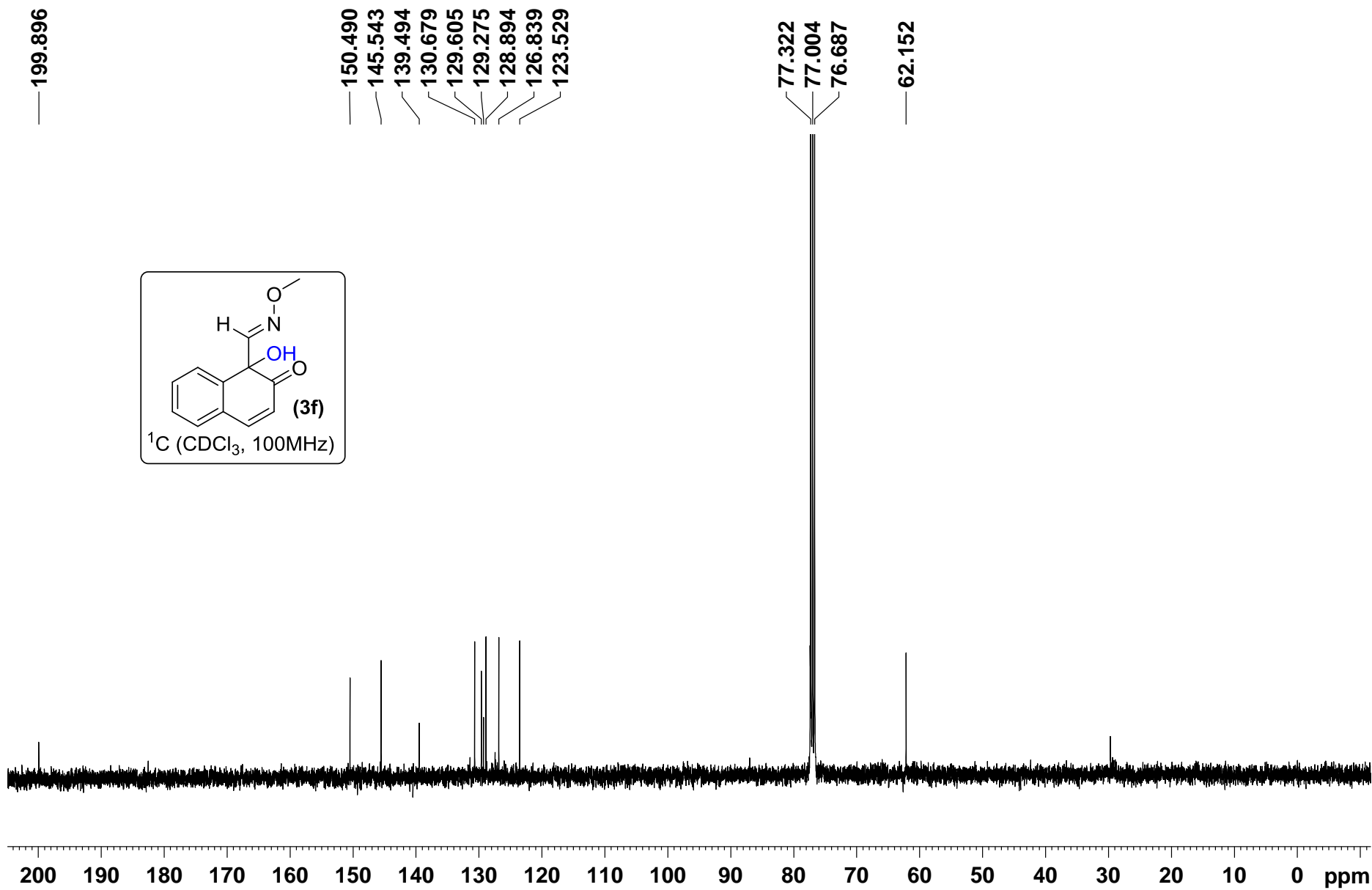


7.707
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 7.379
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 7.313
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 6.258

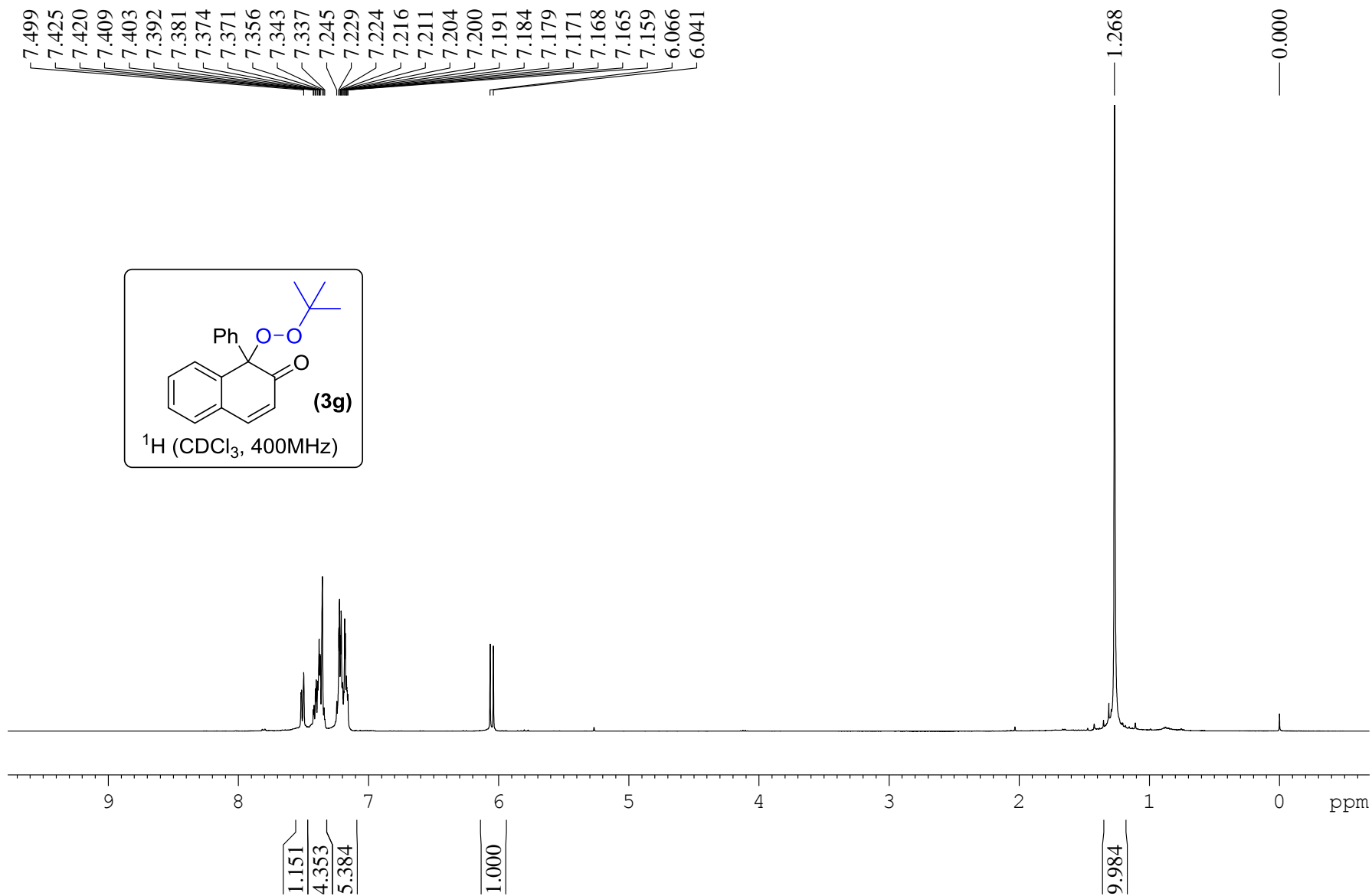
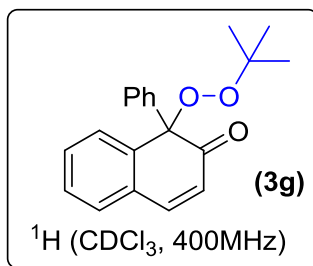
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 — 3.772

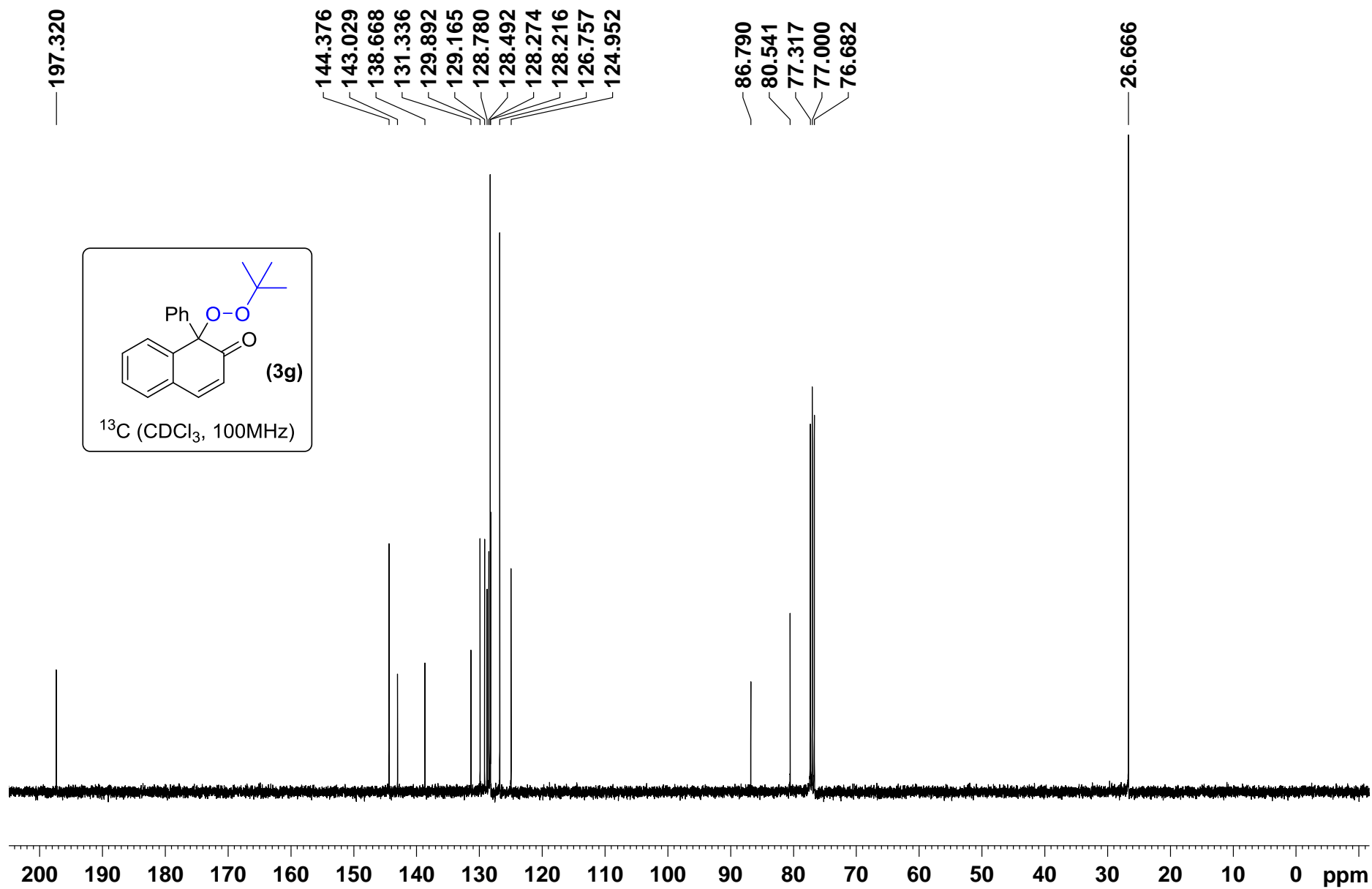
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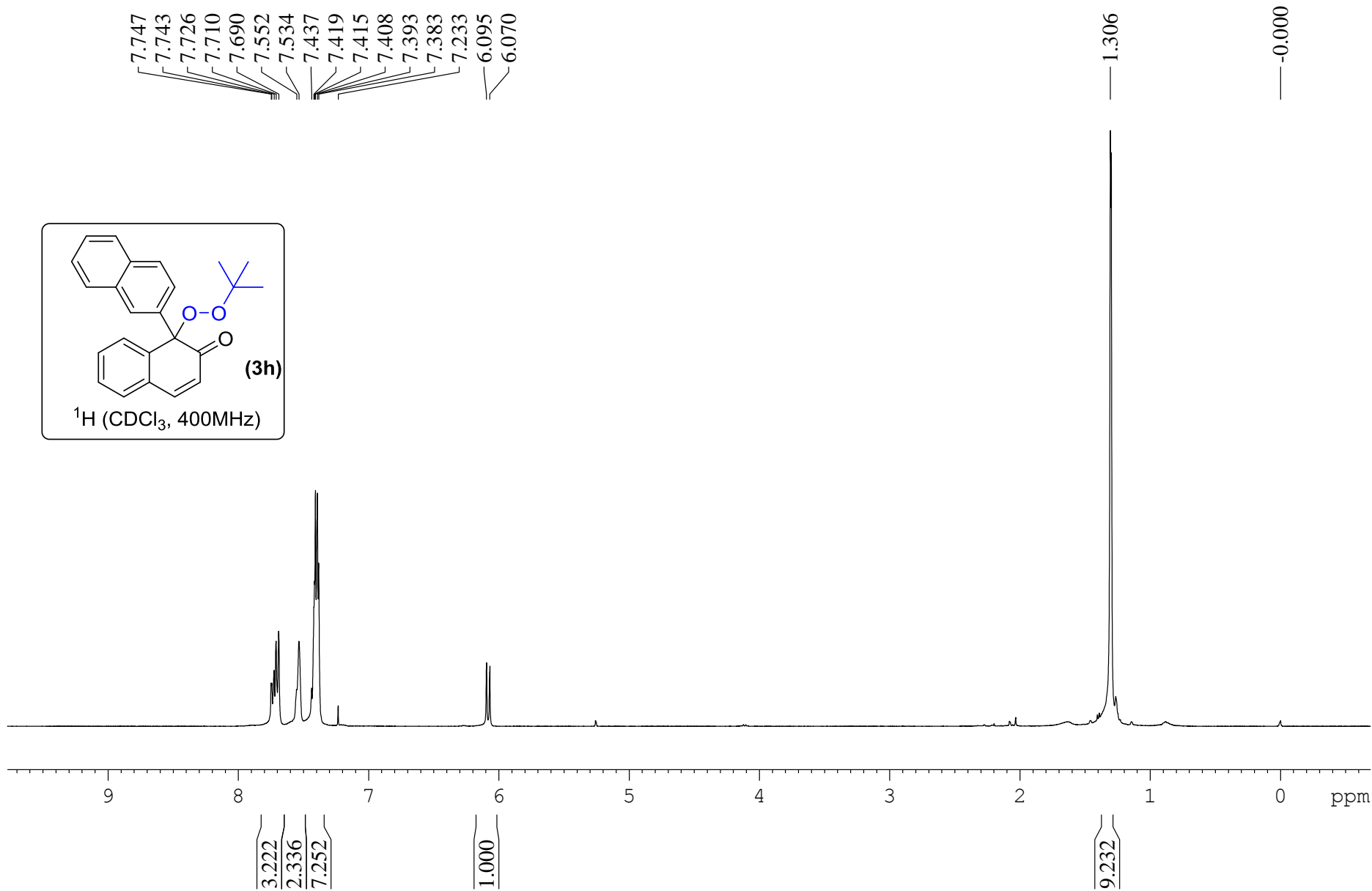


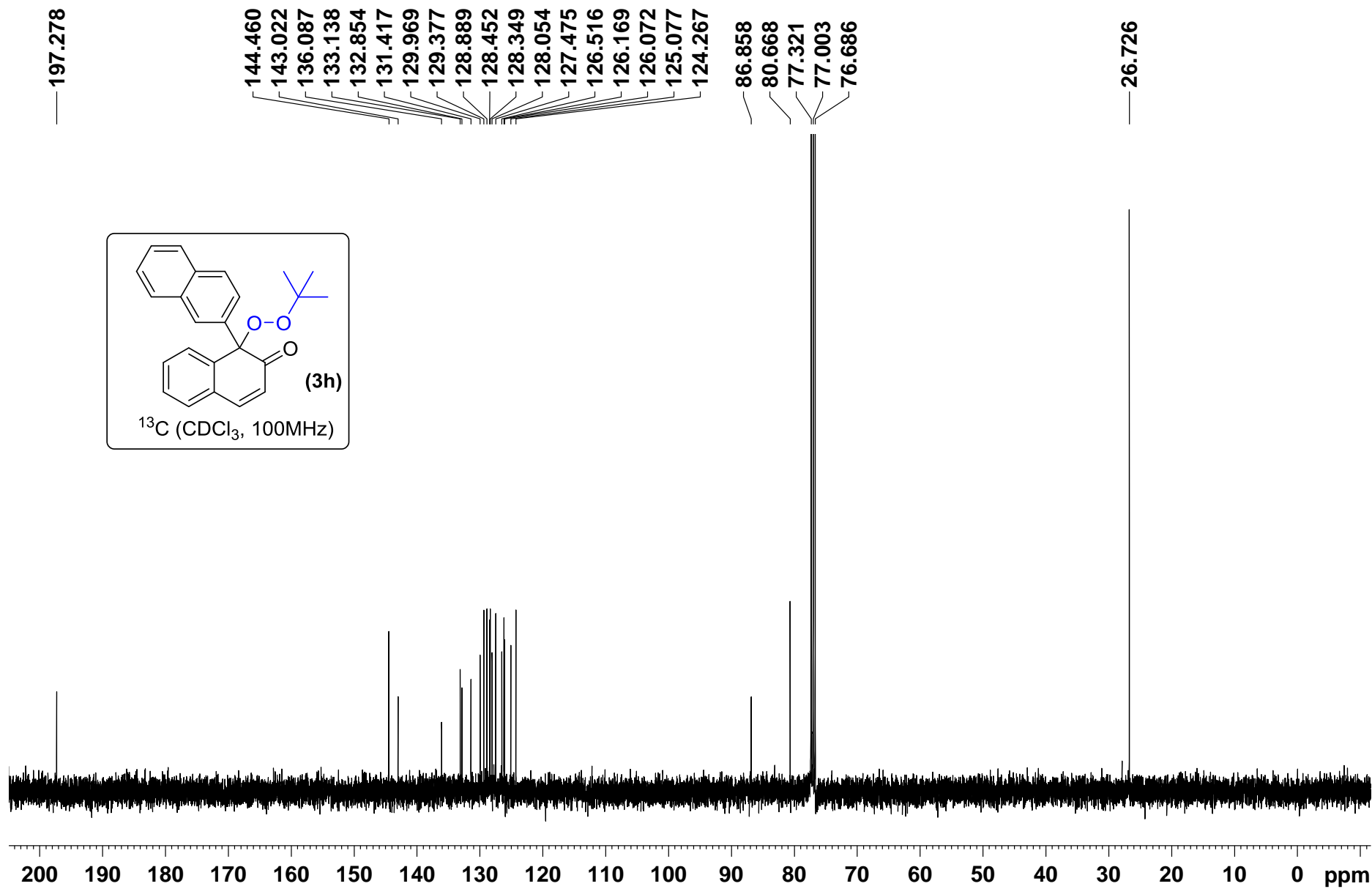


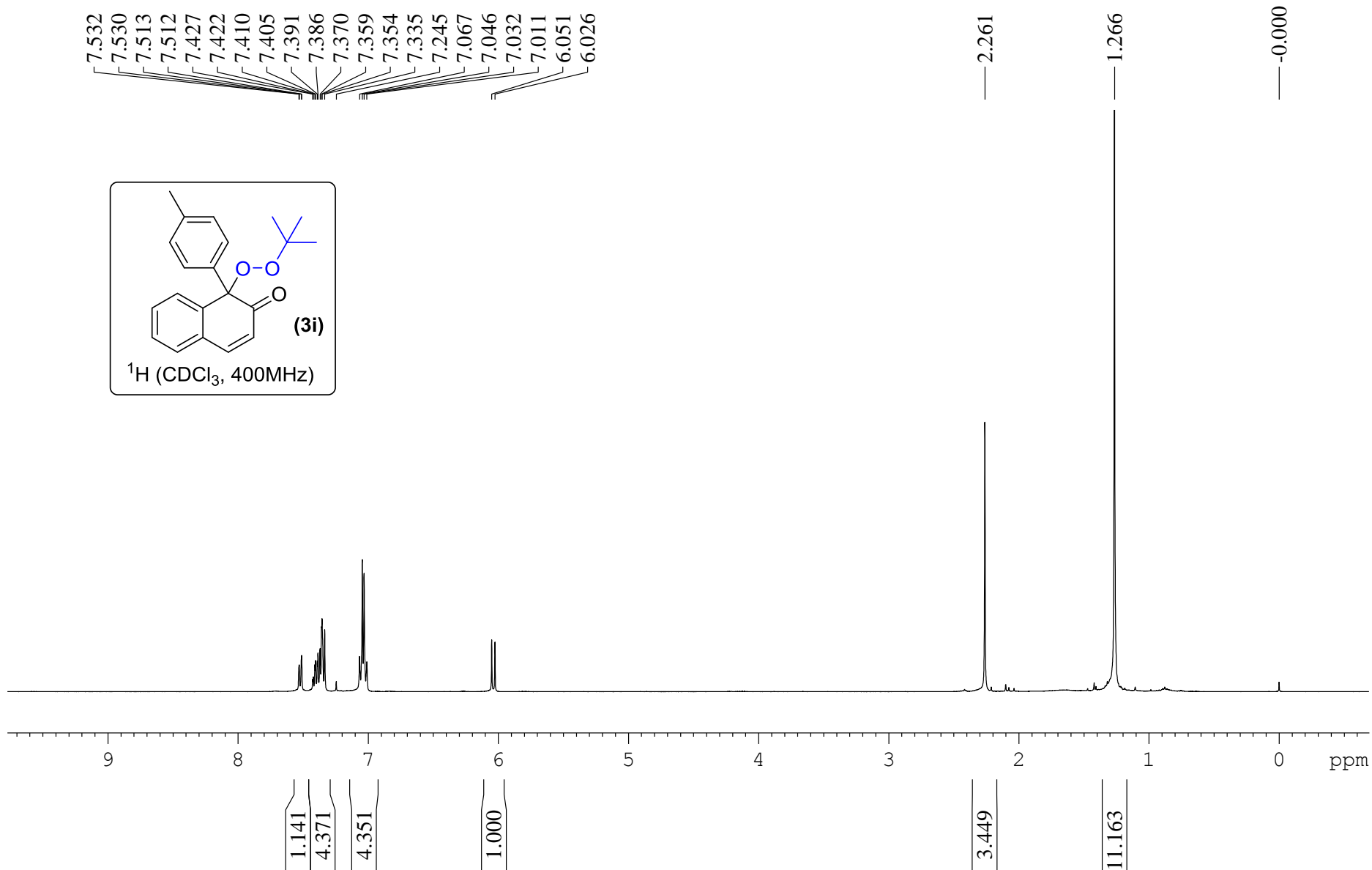
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7.245
7.229
7.224
7.216
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6.041

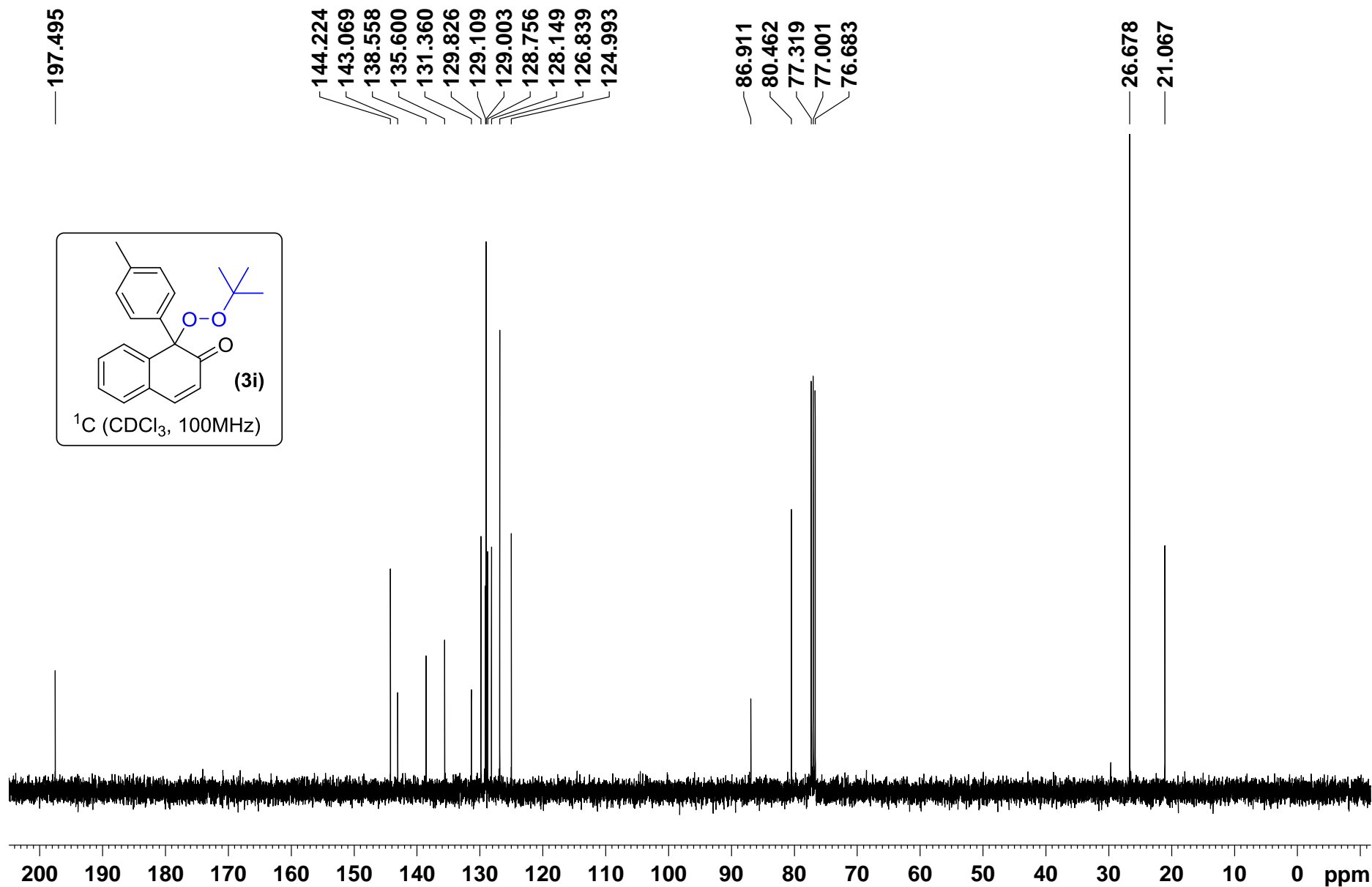


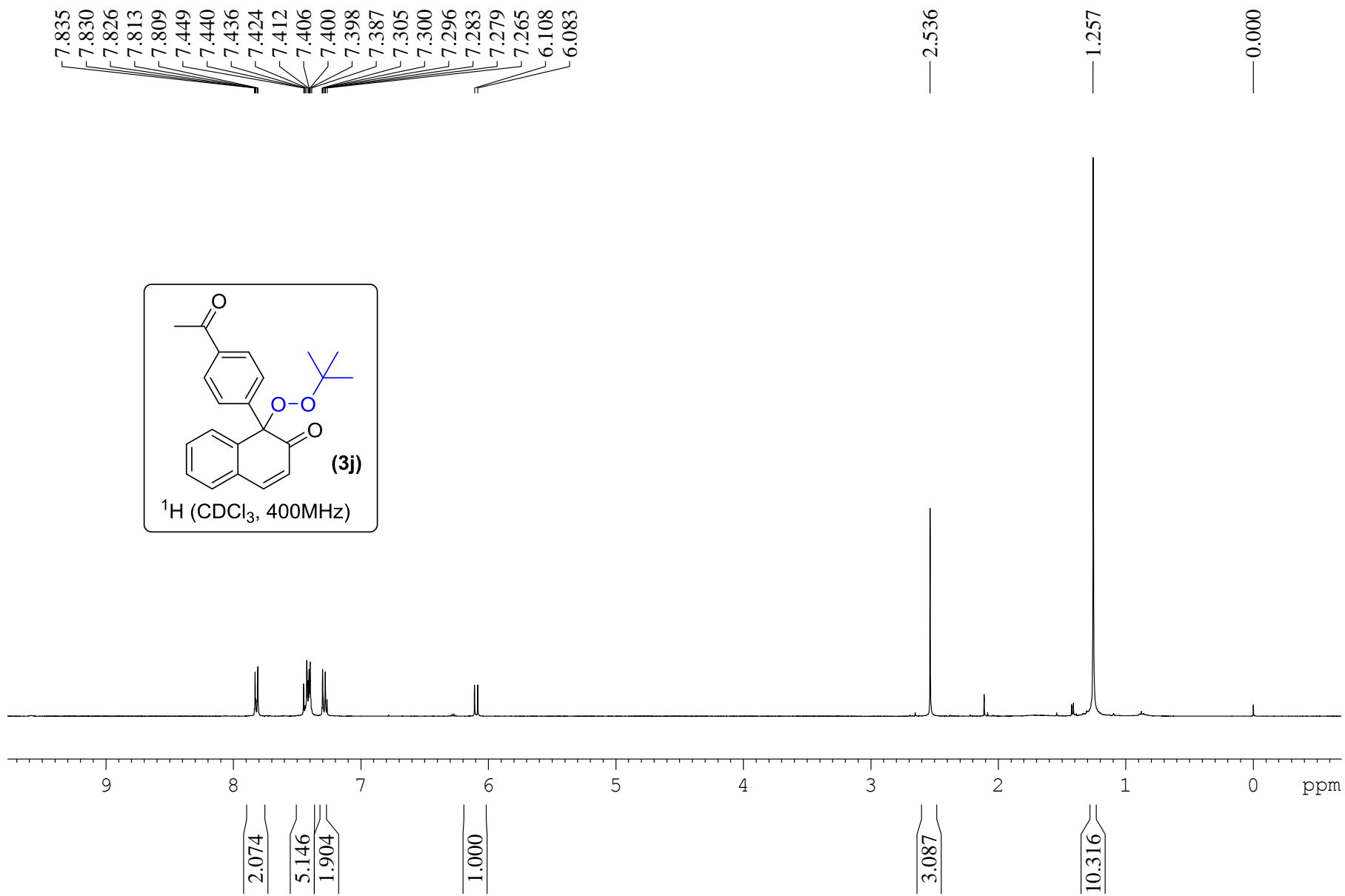


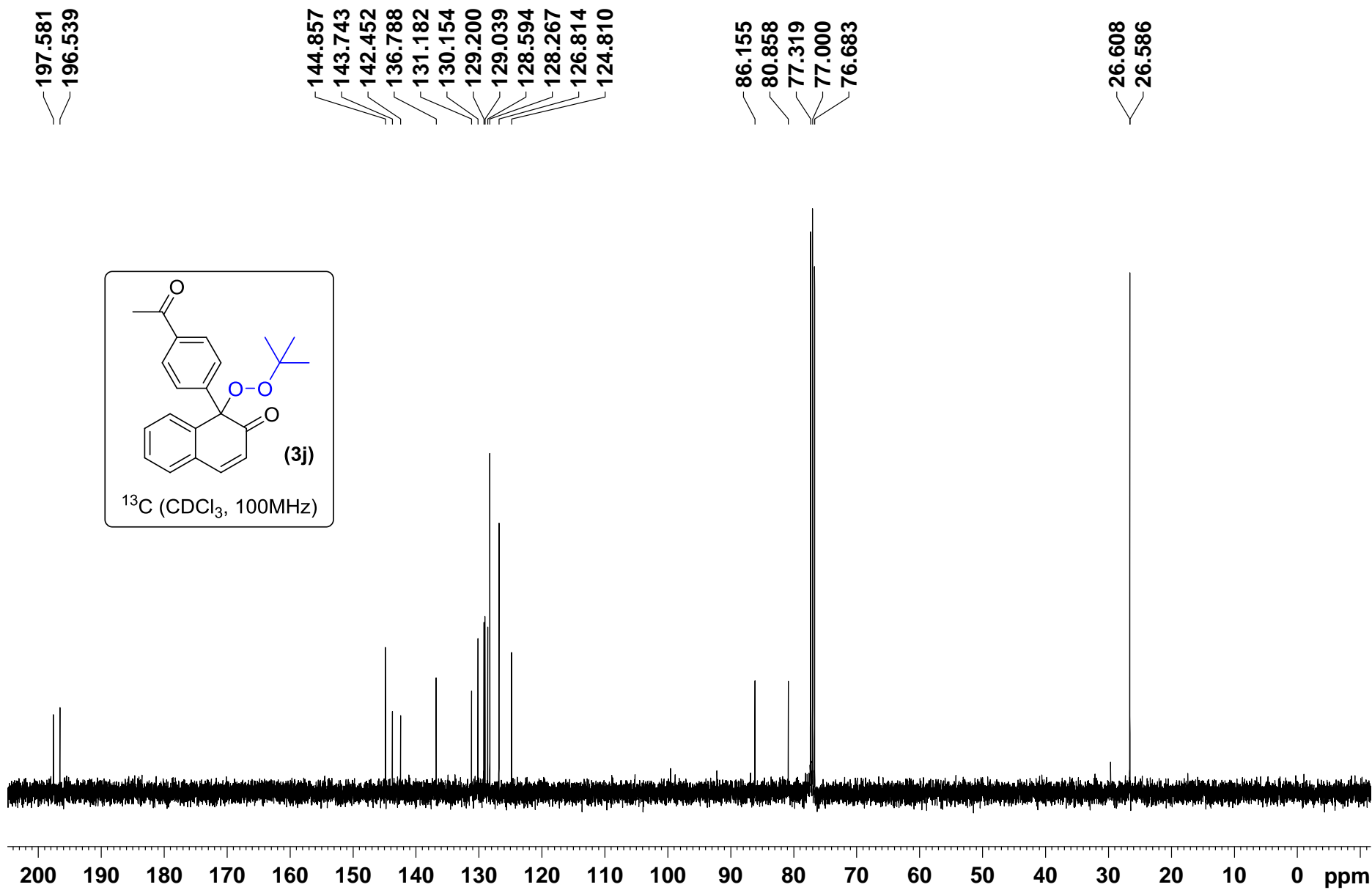




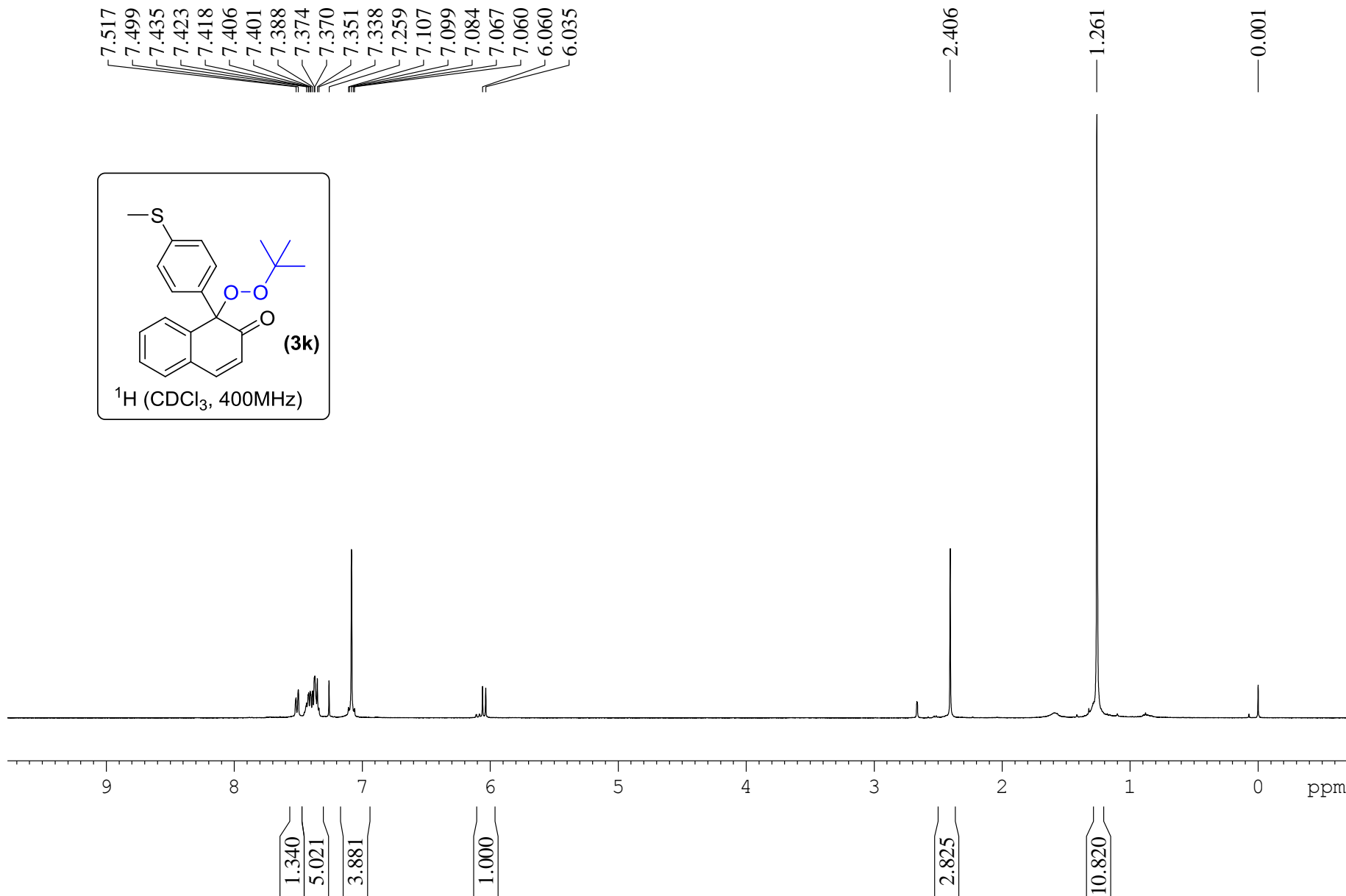
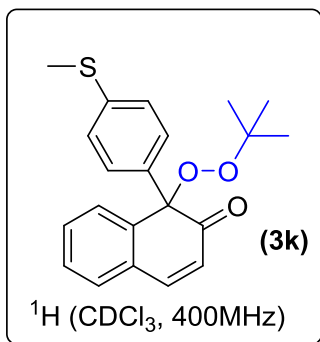


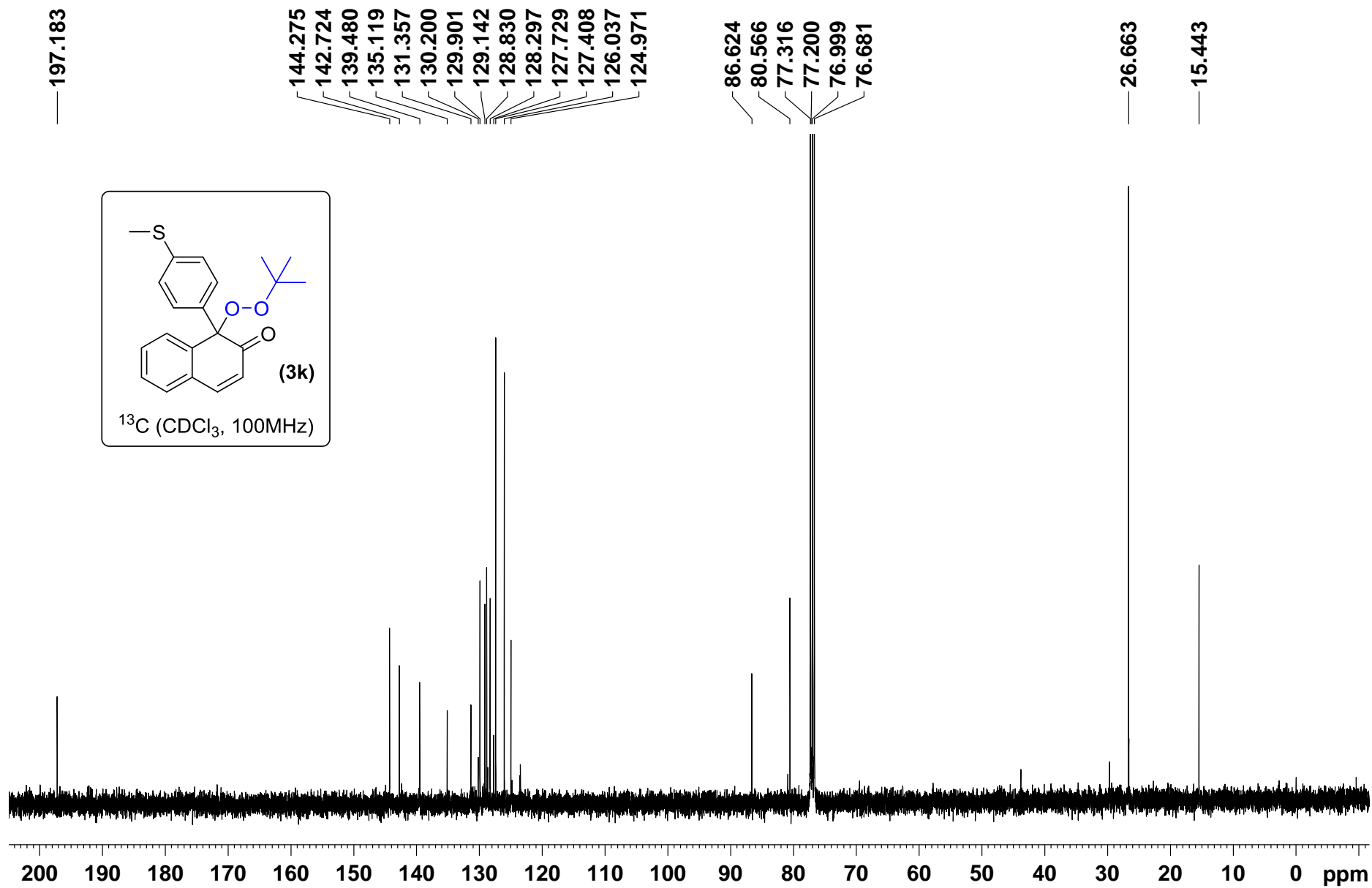




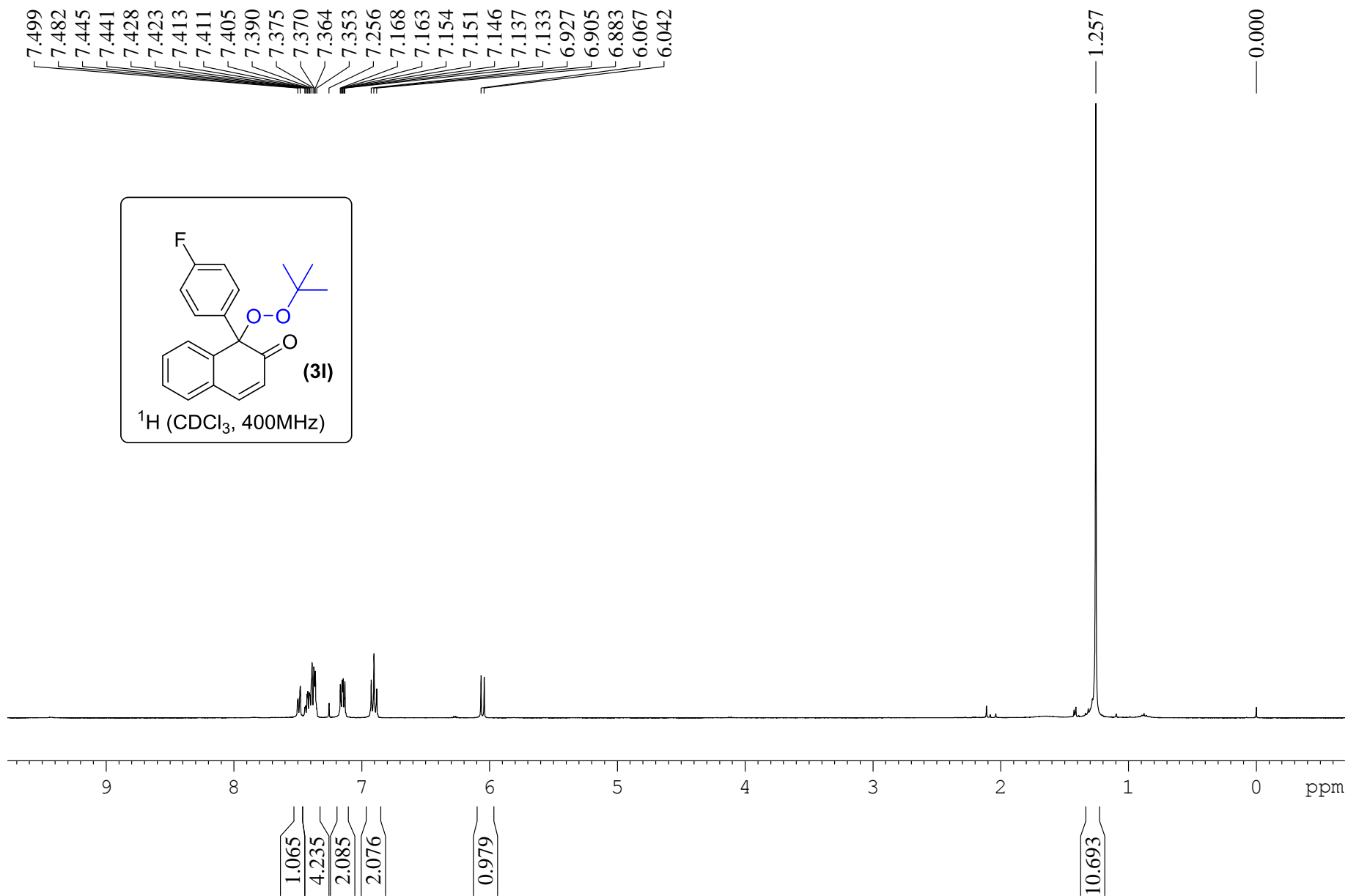
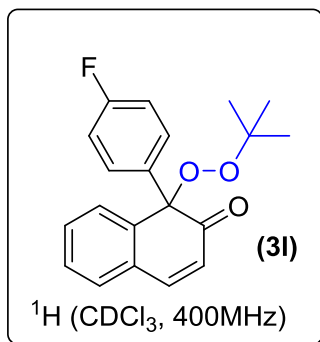


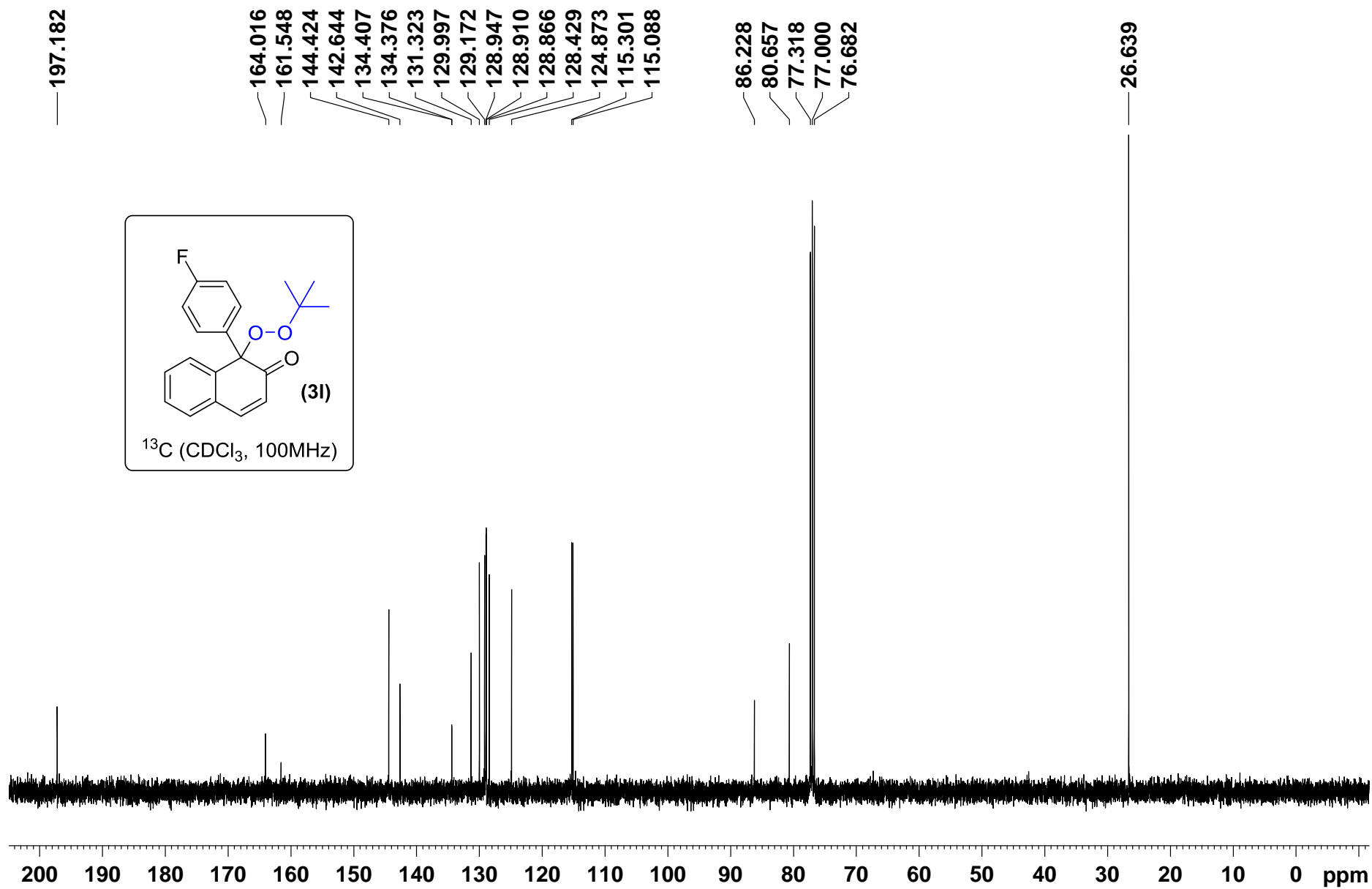
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7.388
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7.107
7.099
7.084
7.067
7.060
6.060
6.035

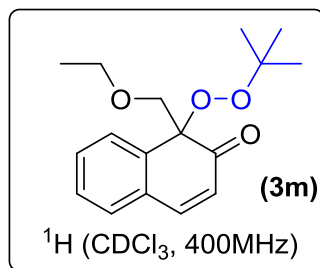




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7.411
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7.390
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7.370
7.364
7.353
7.256
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7.133
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6.905
6.883
6.067
6.042





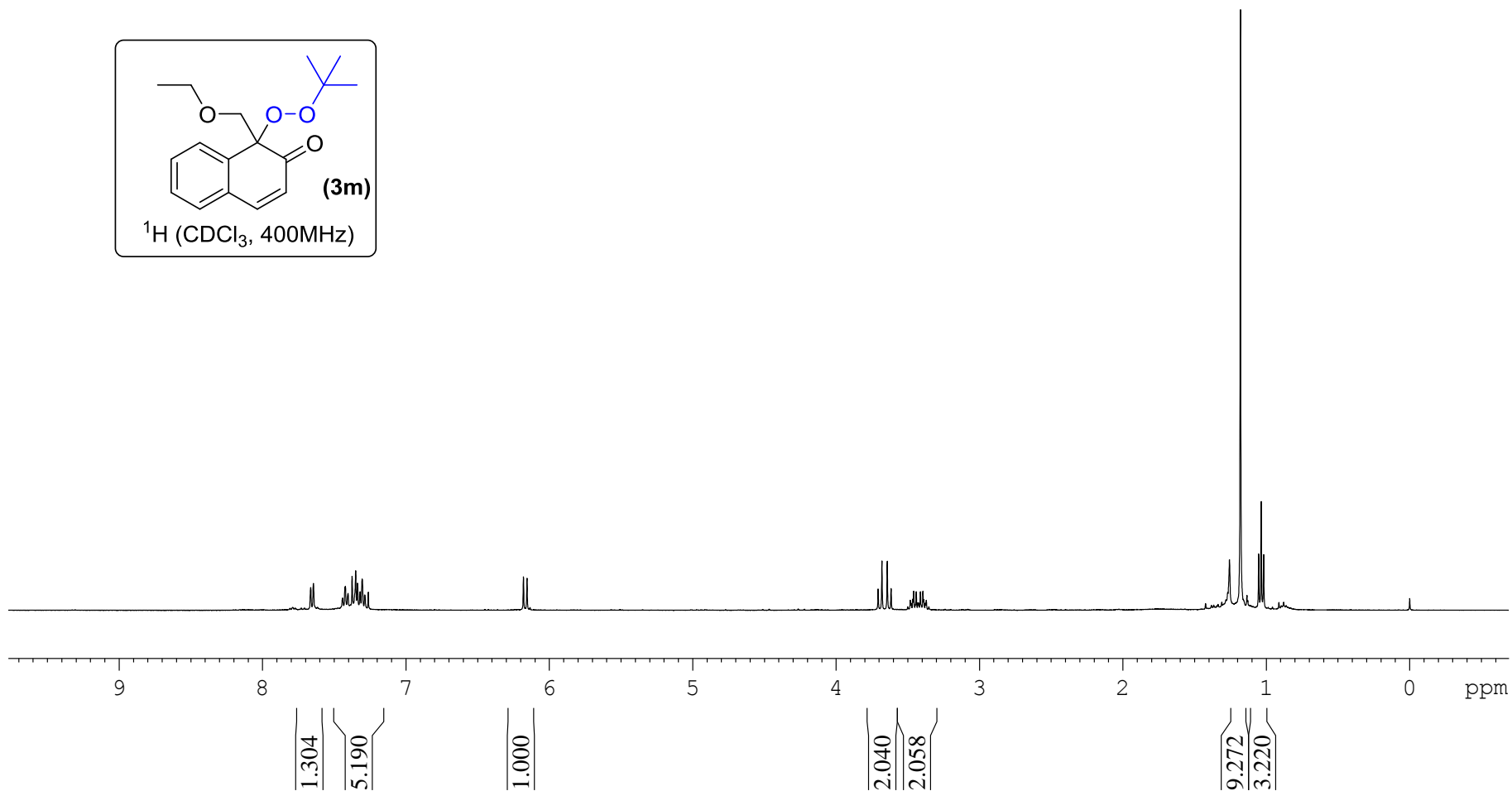


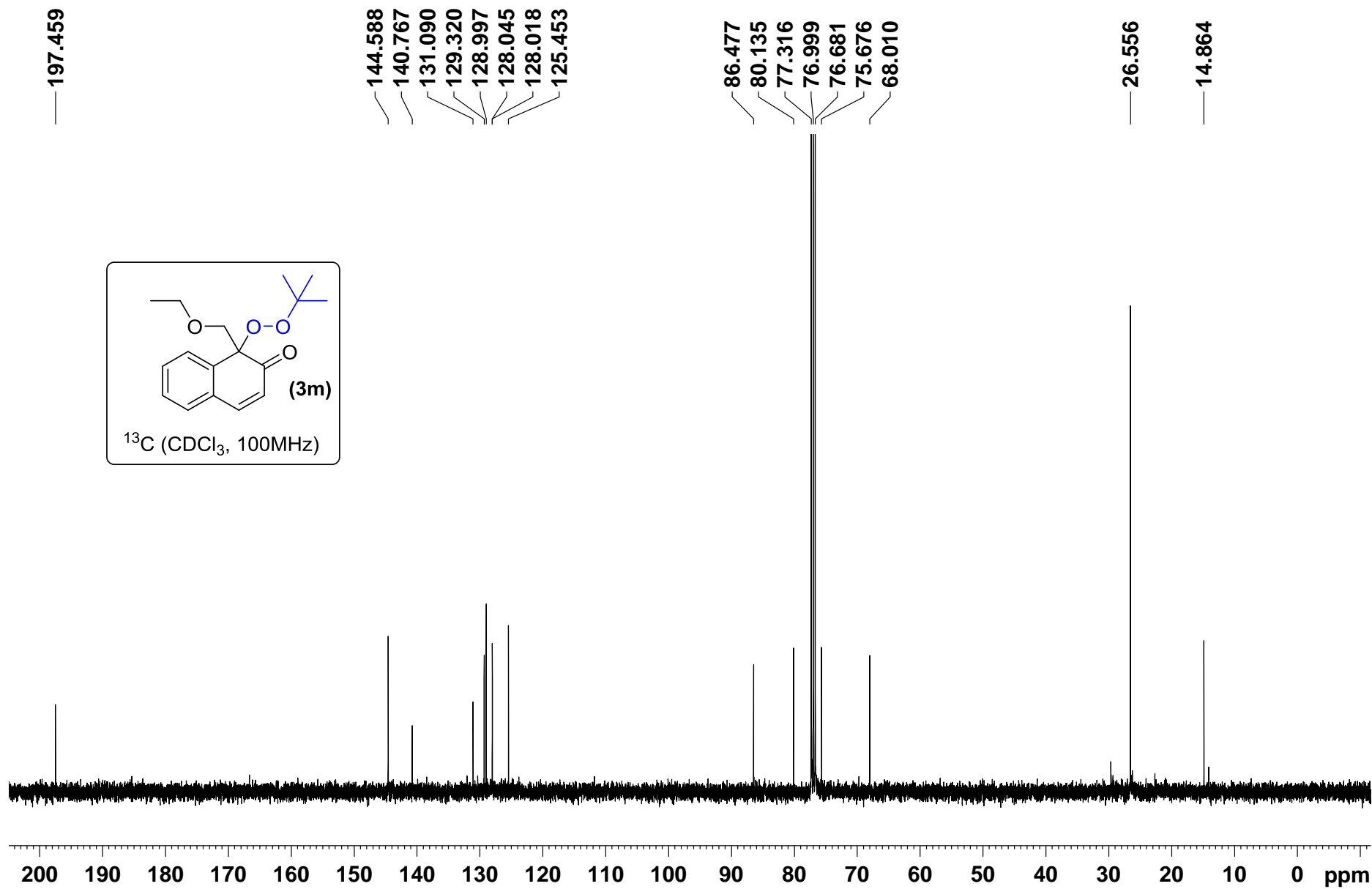
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 6.180
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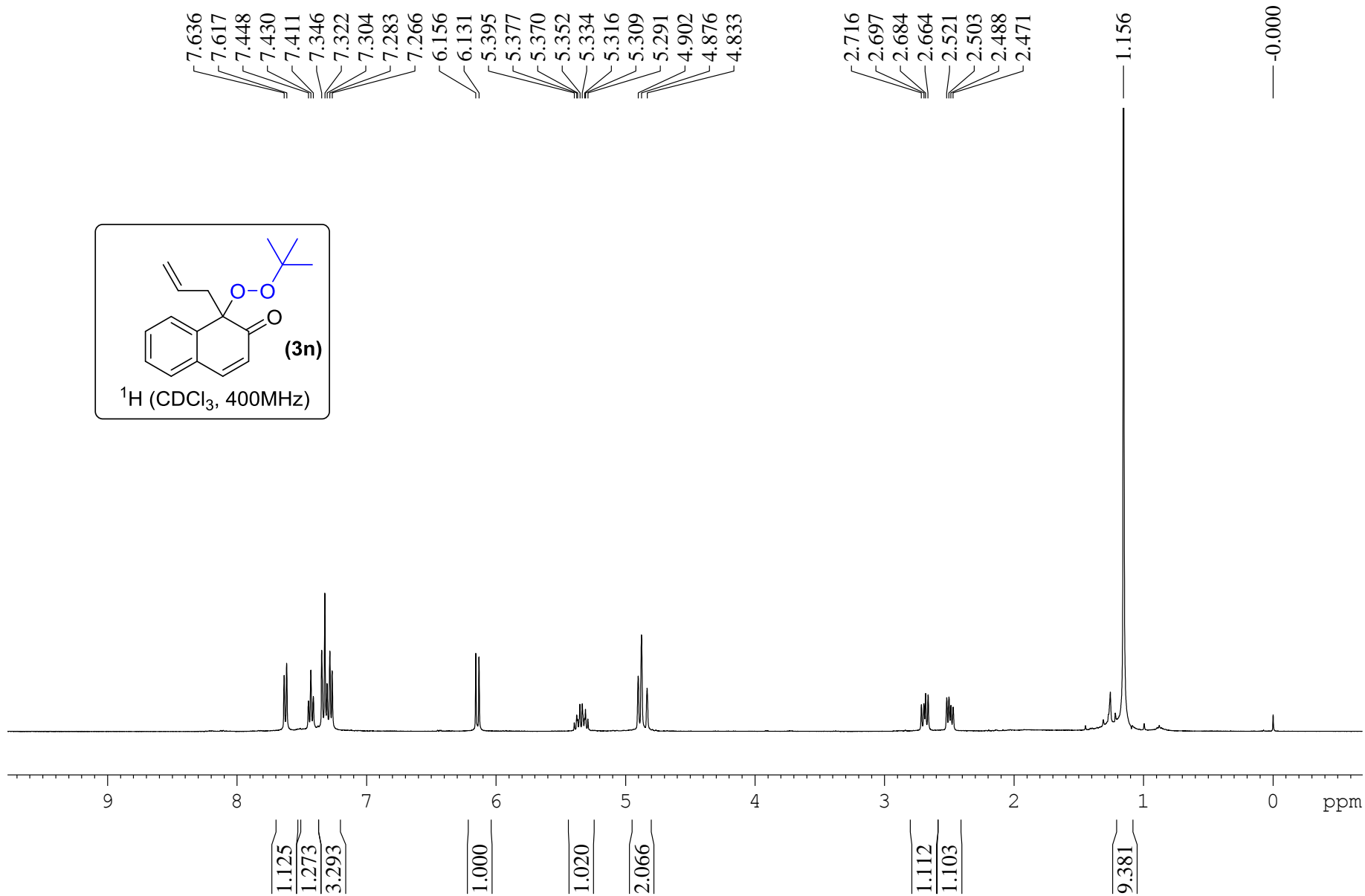
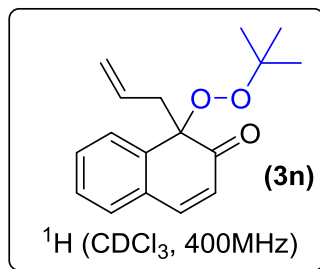
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 3.354

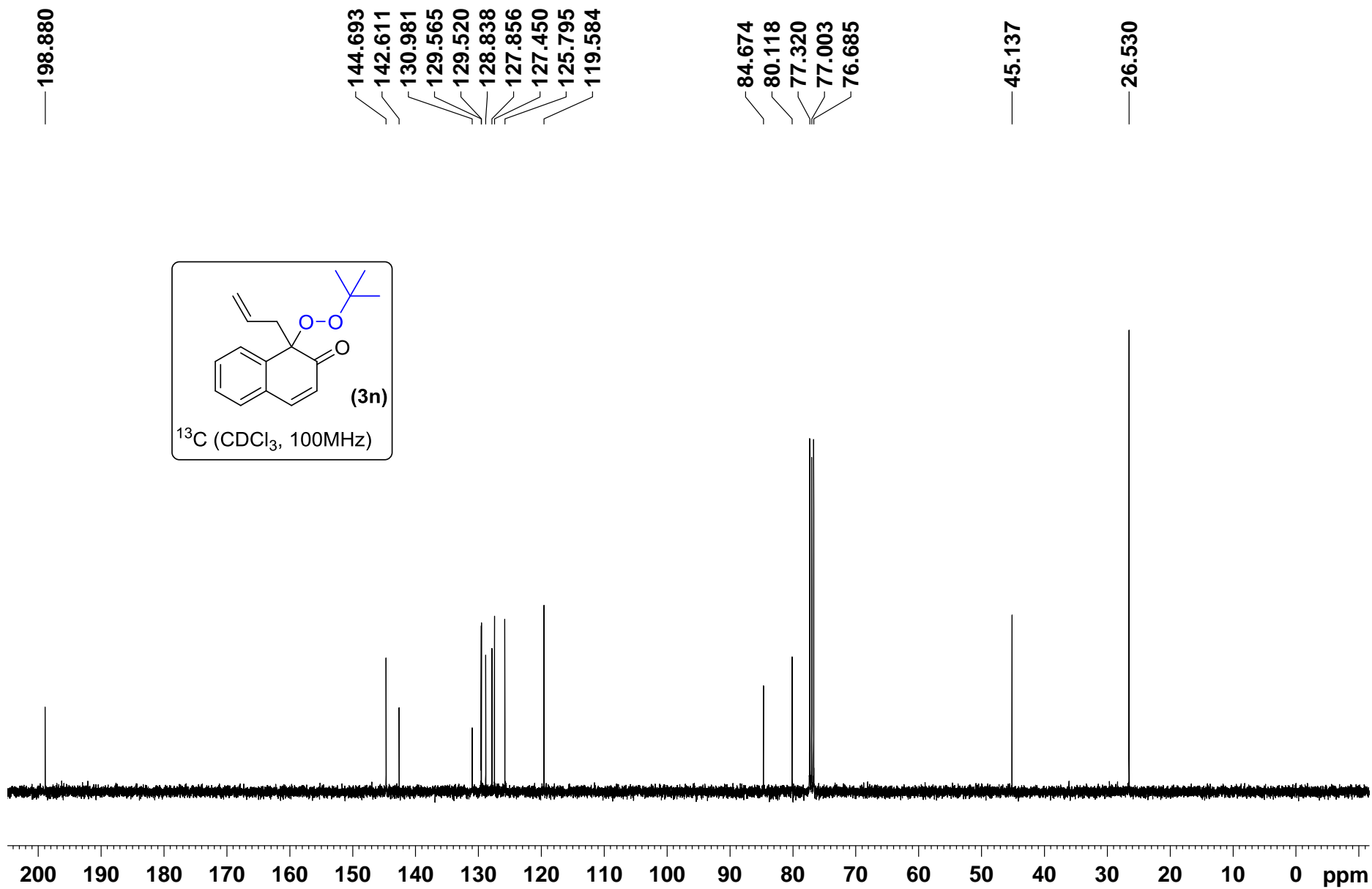
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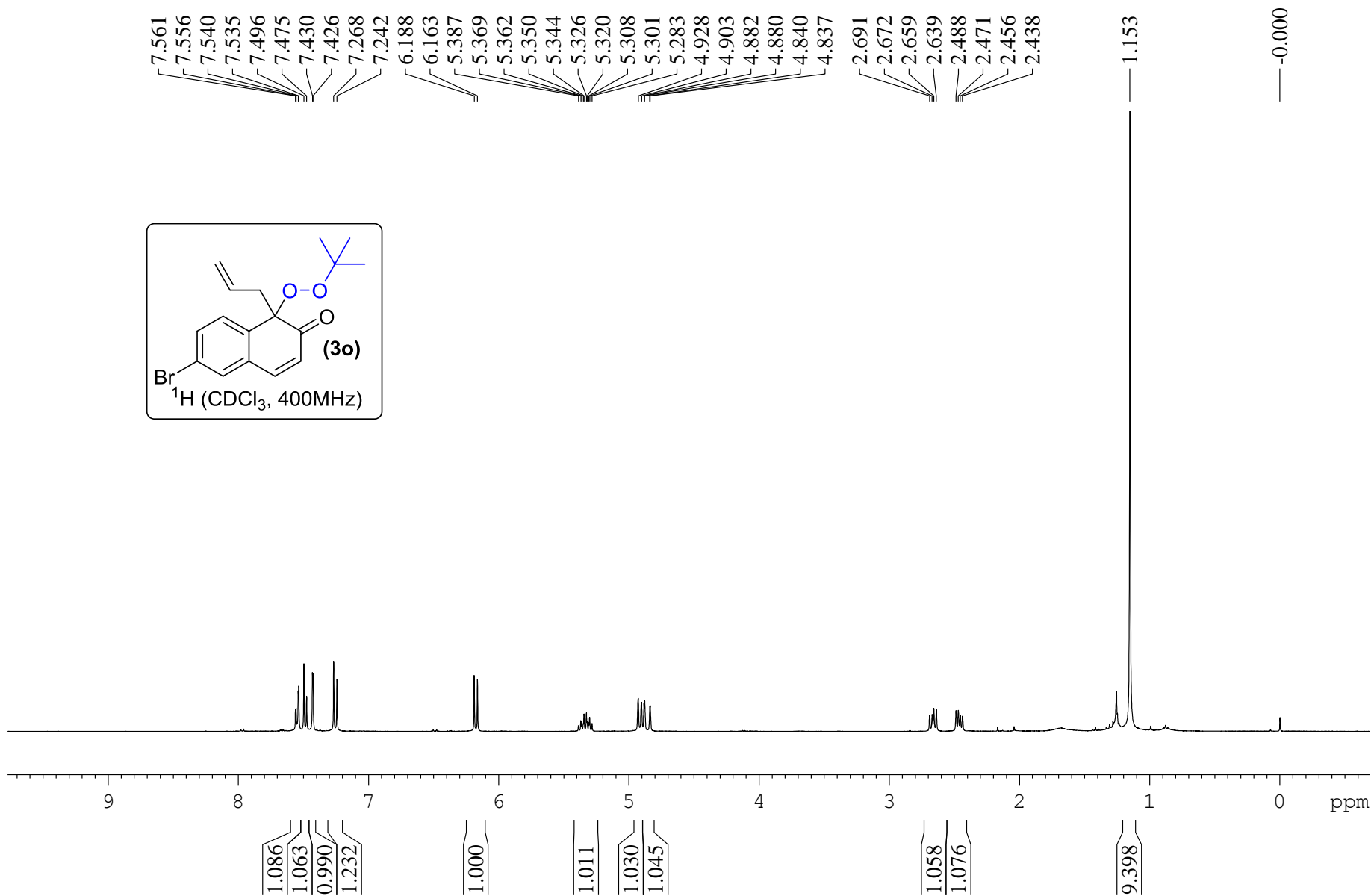
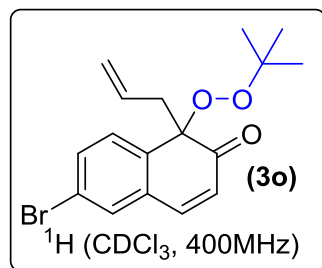
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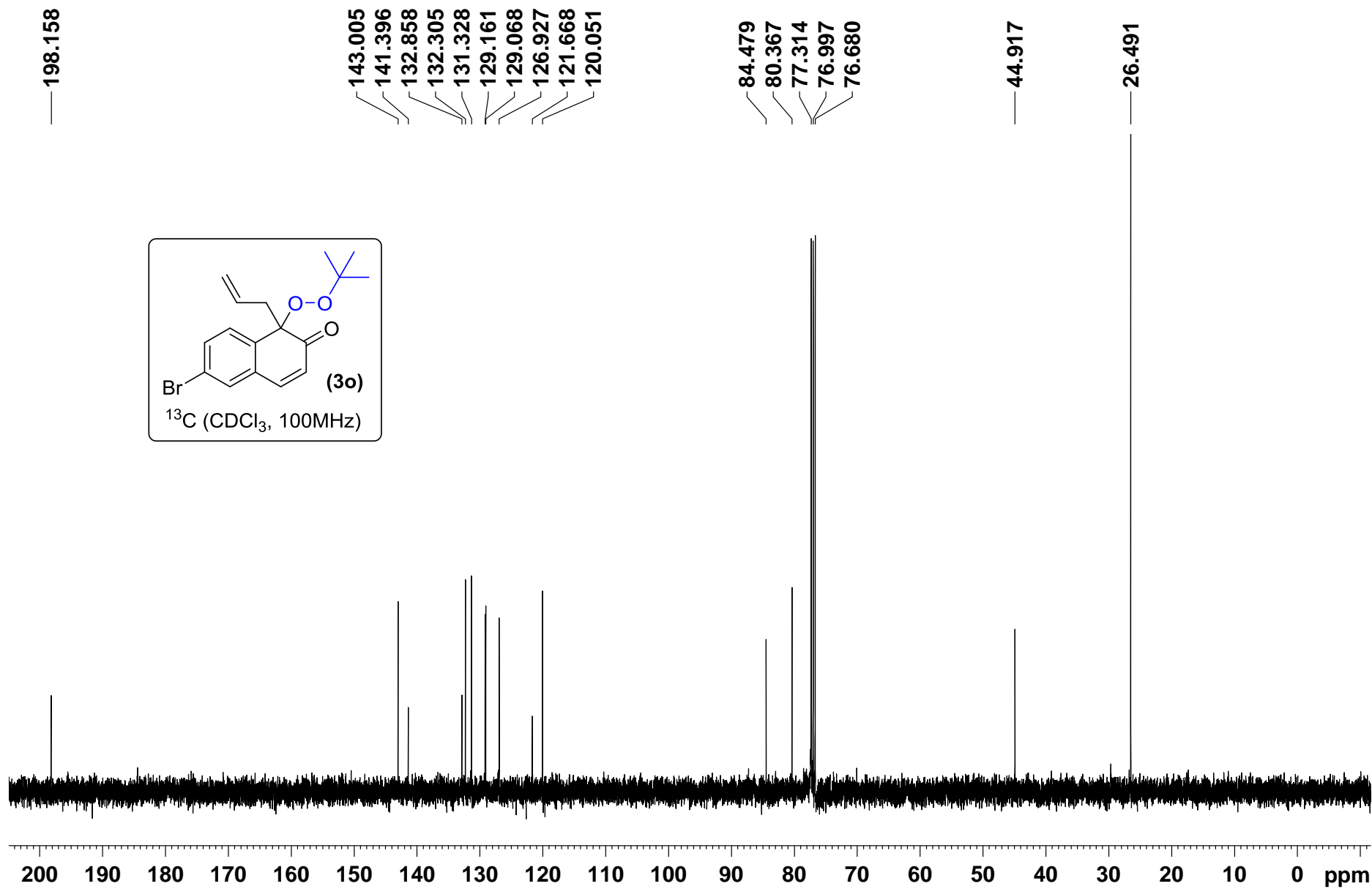






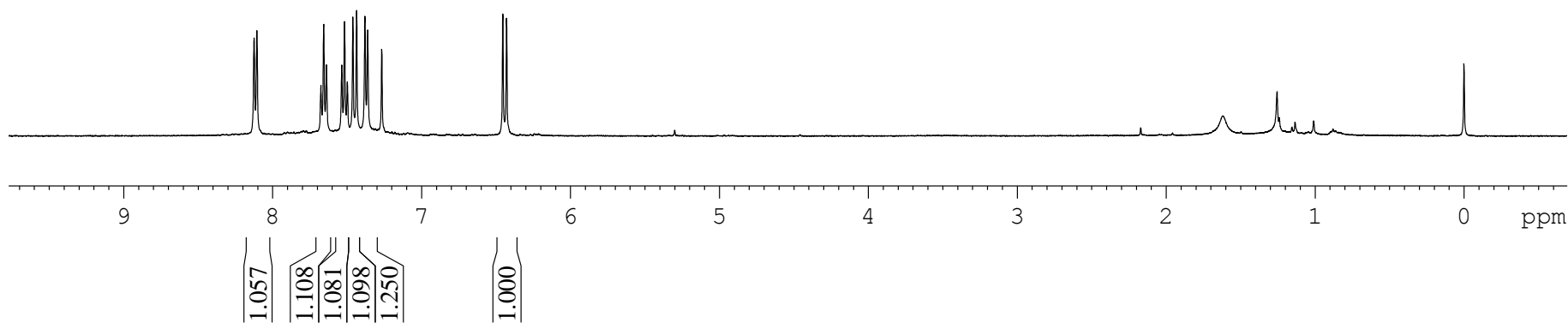
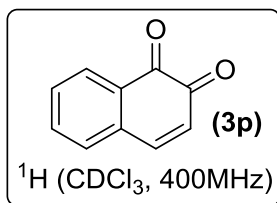






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6.429

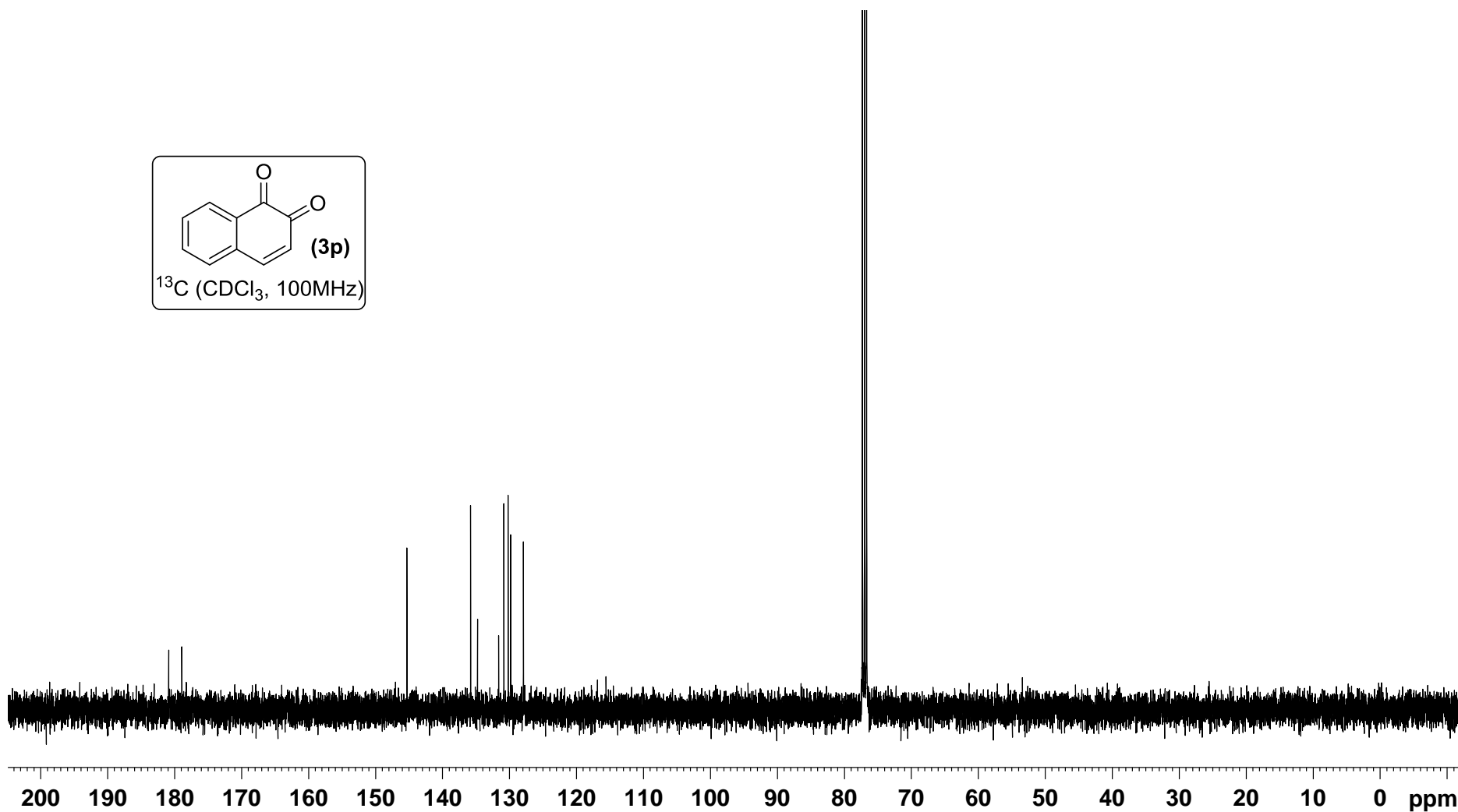
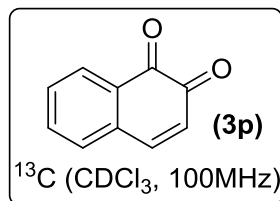
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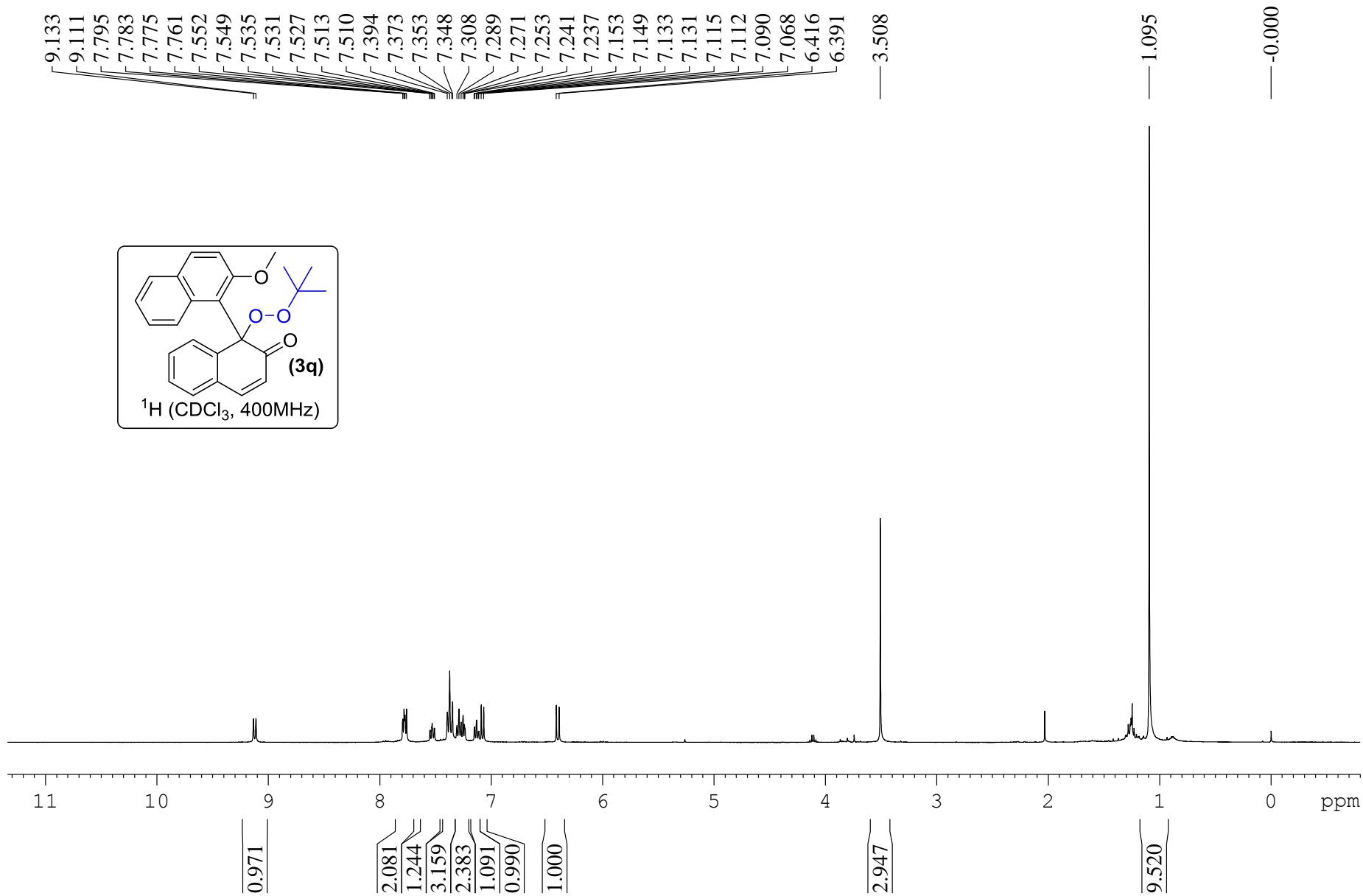


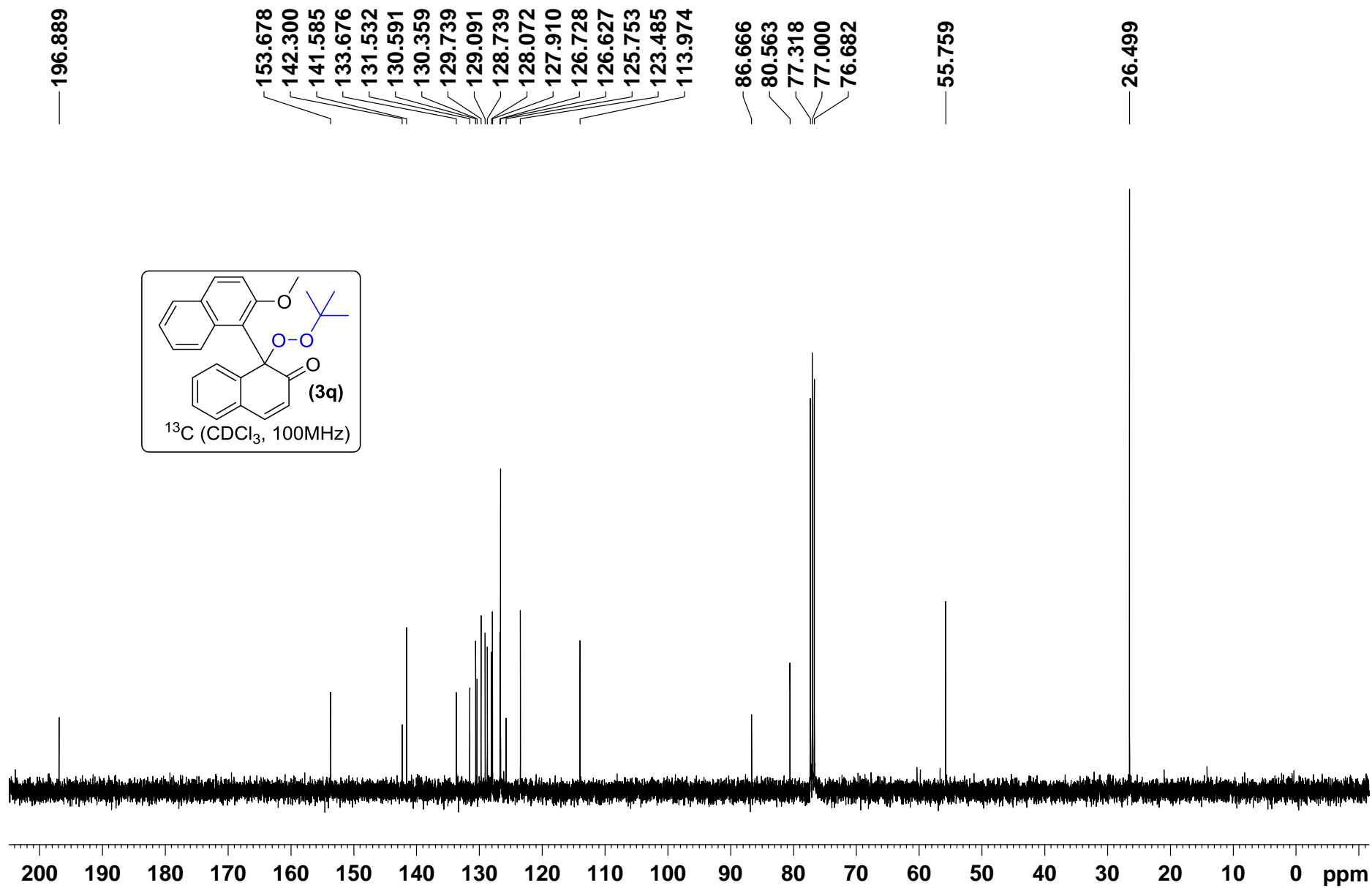
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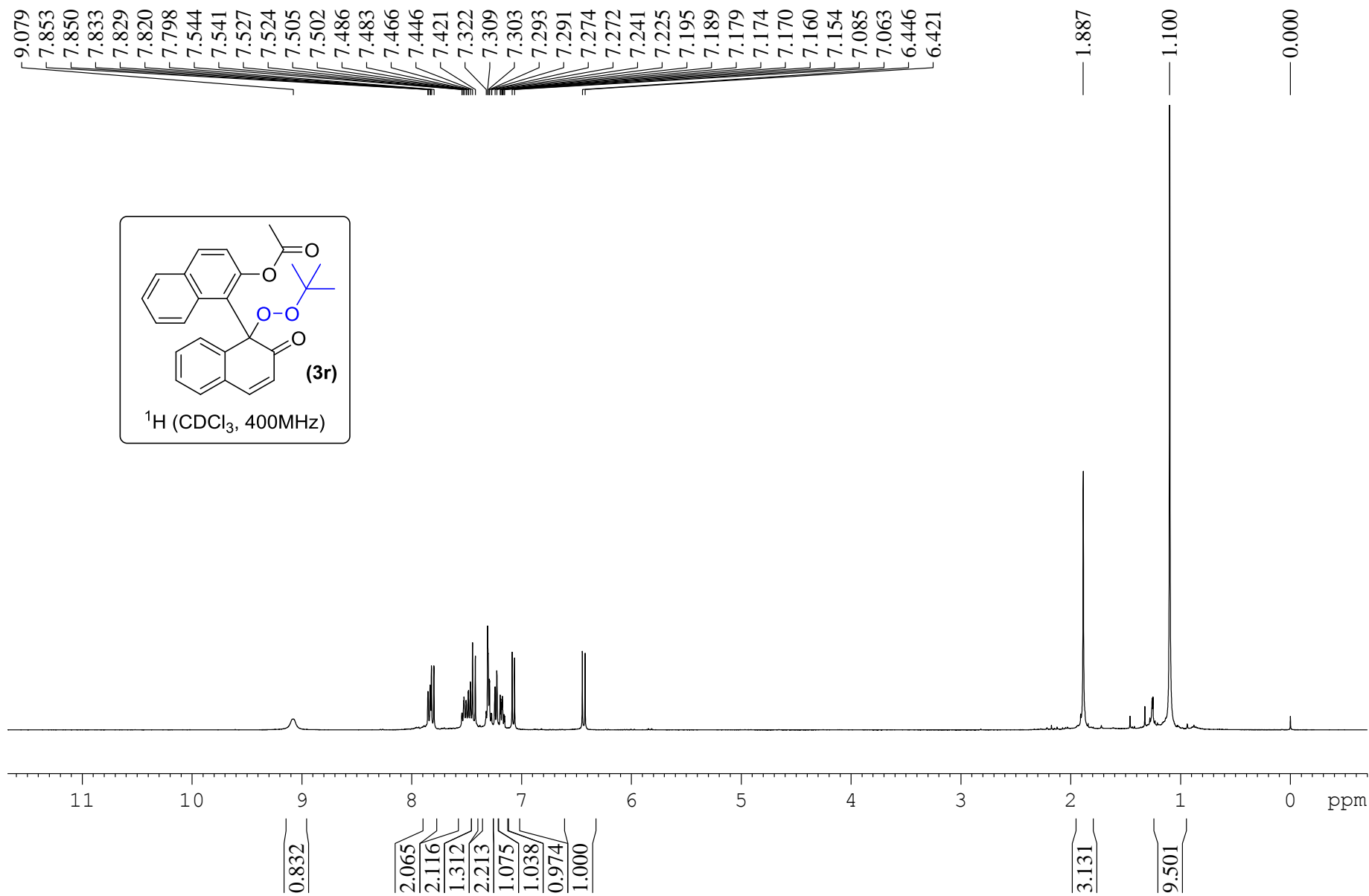
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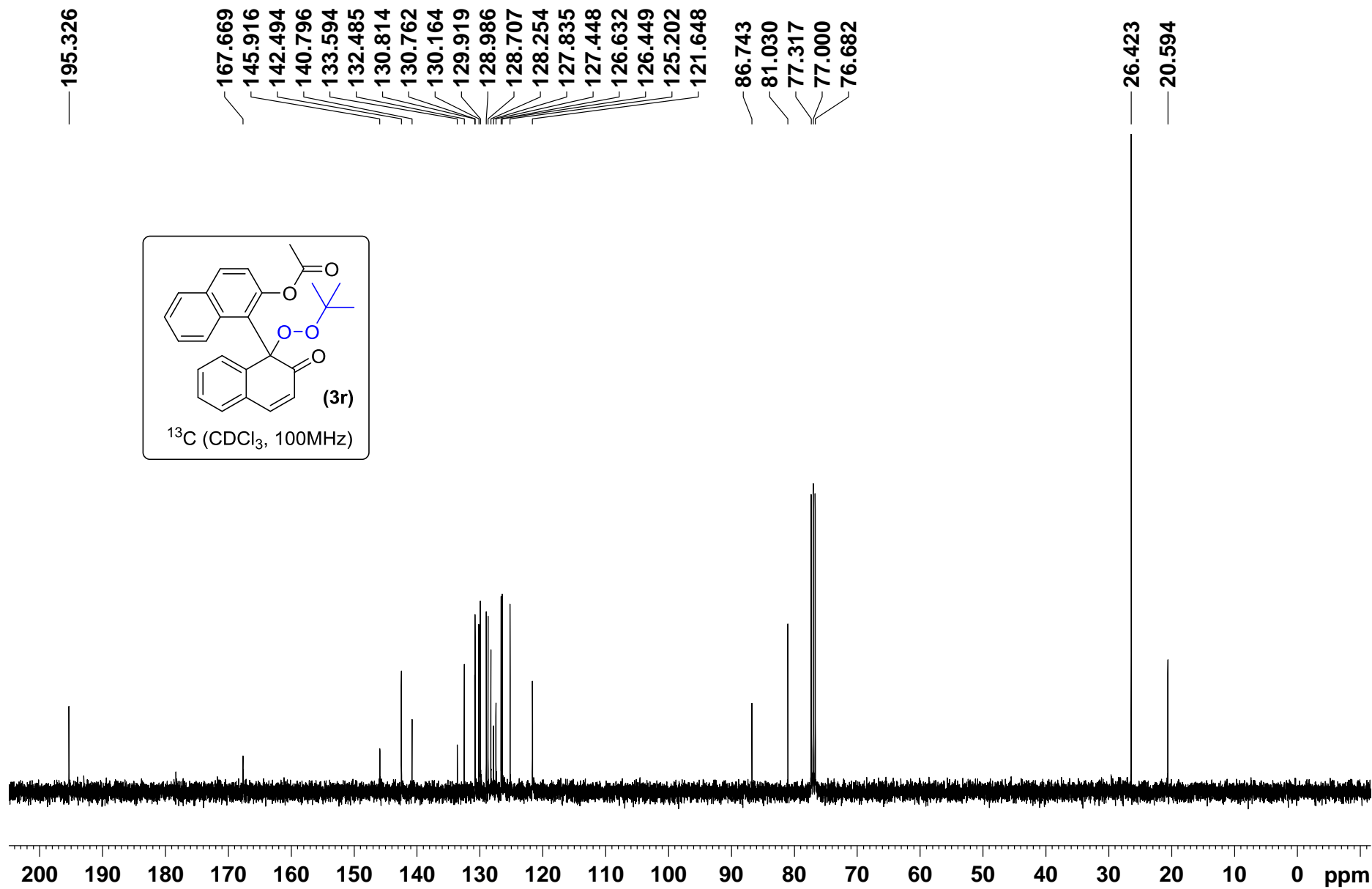
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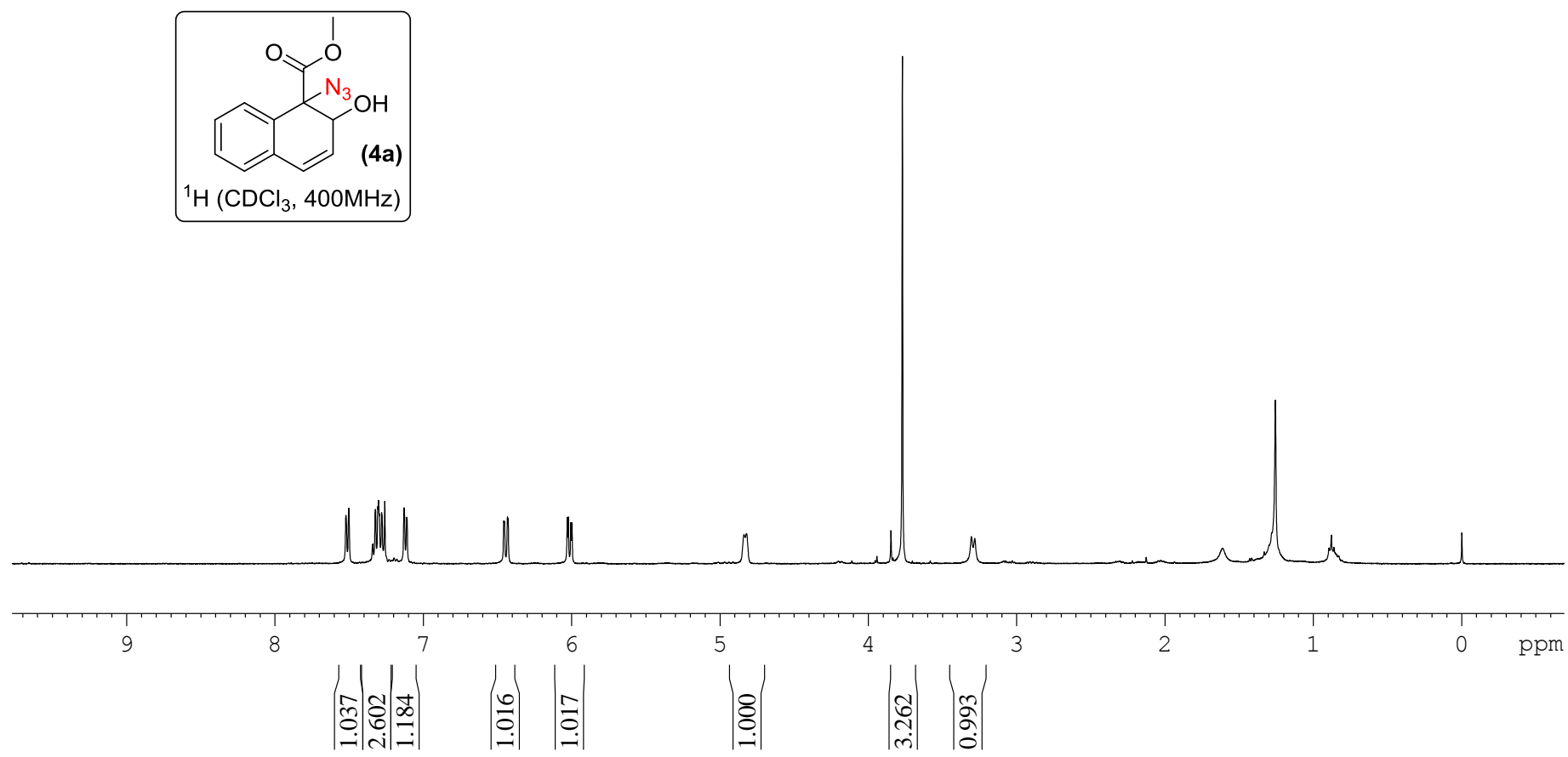
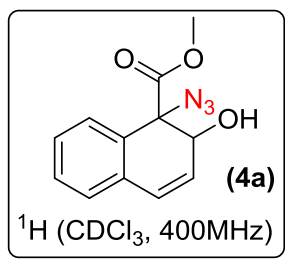


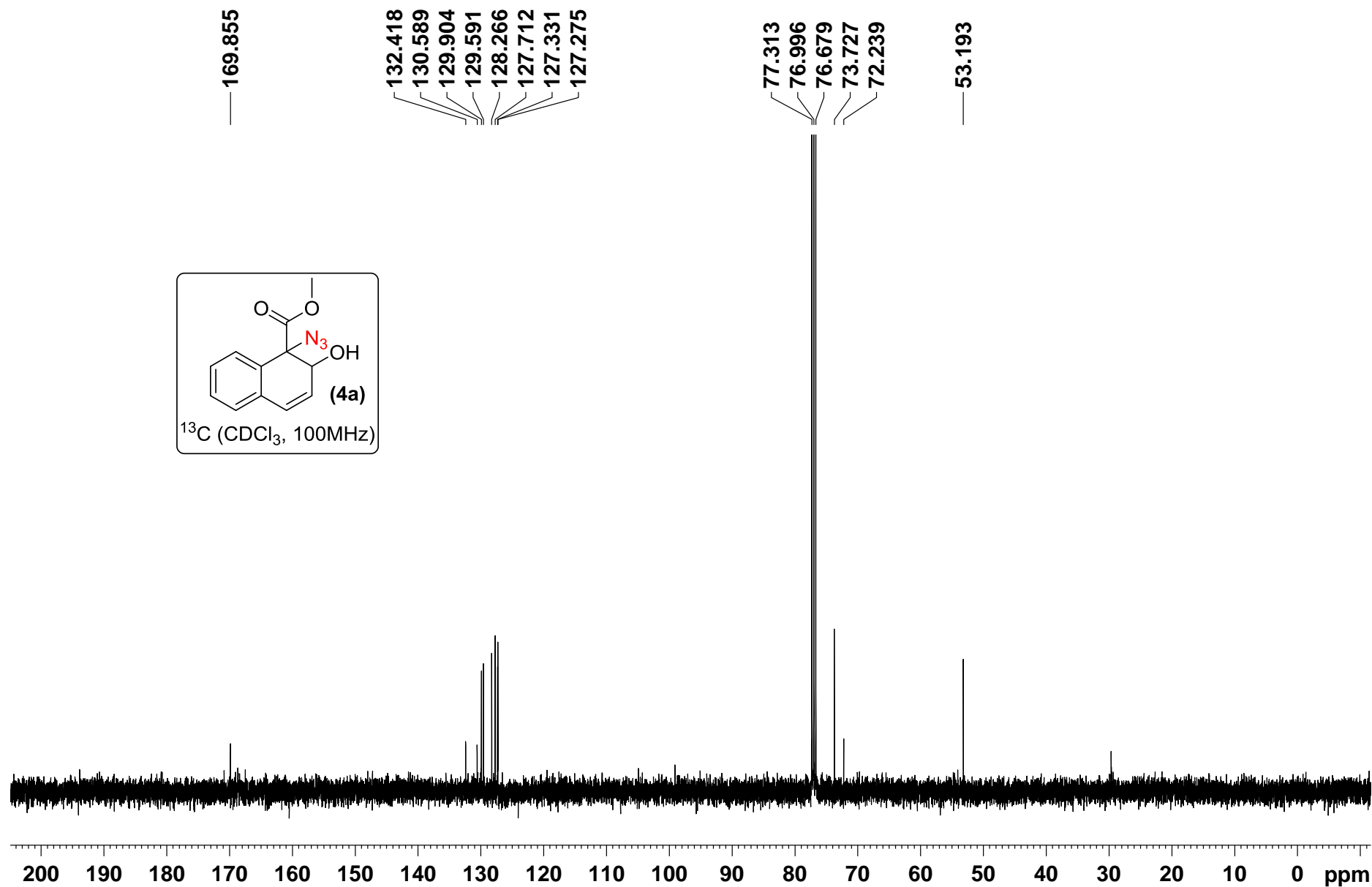


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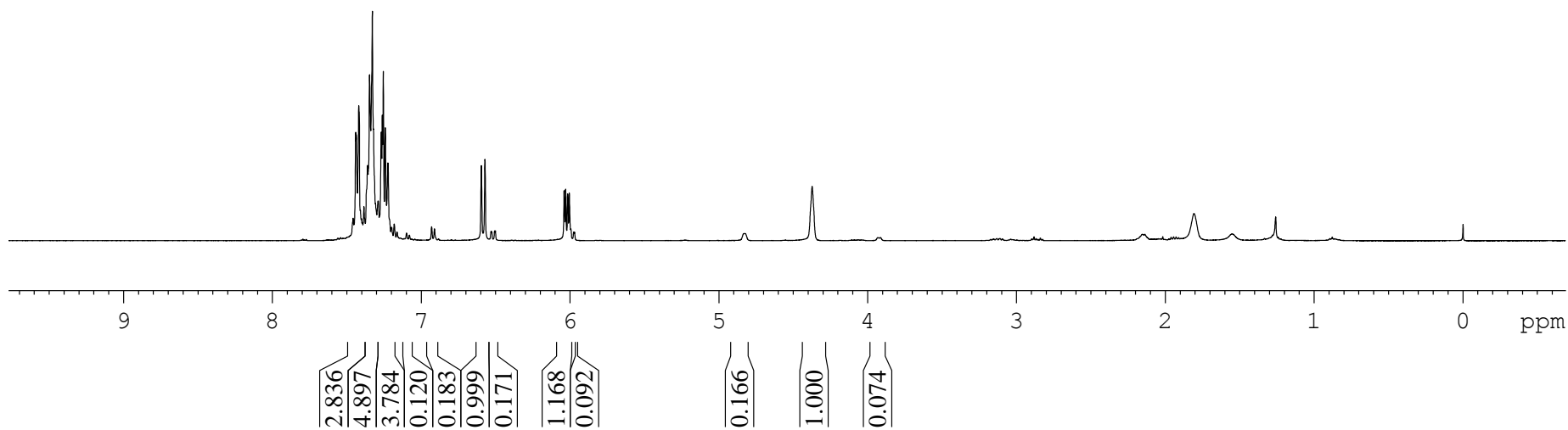
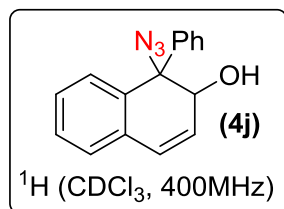
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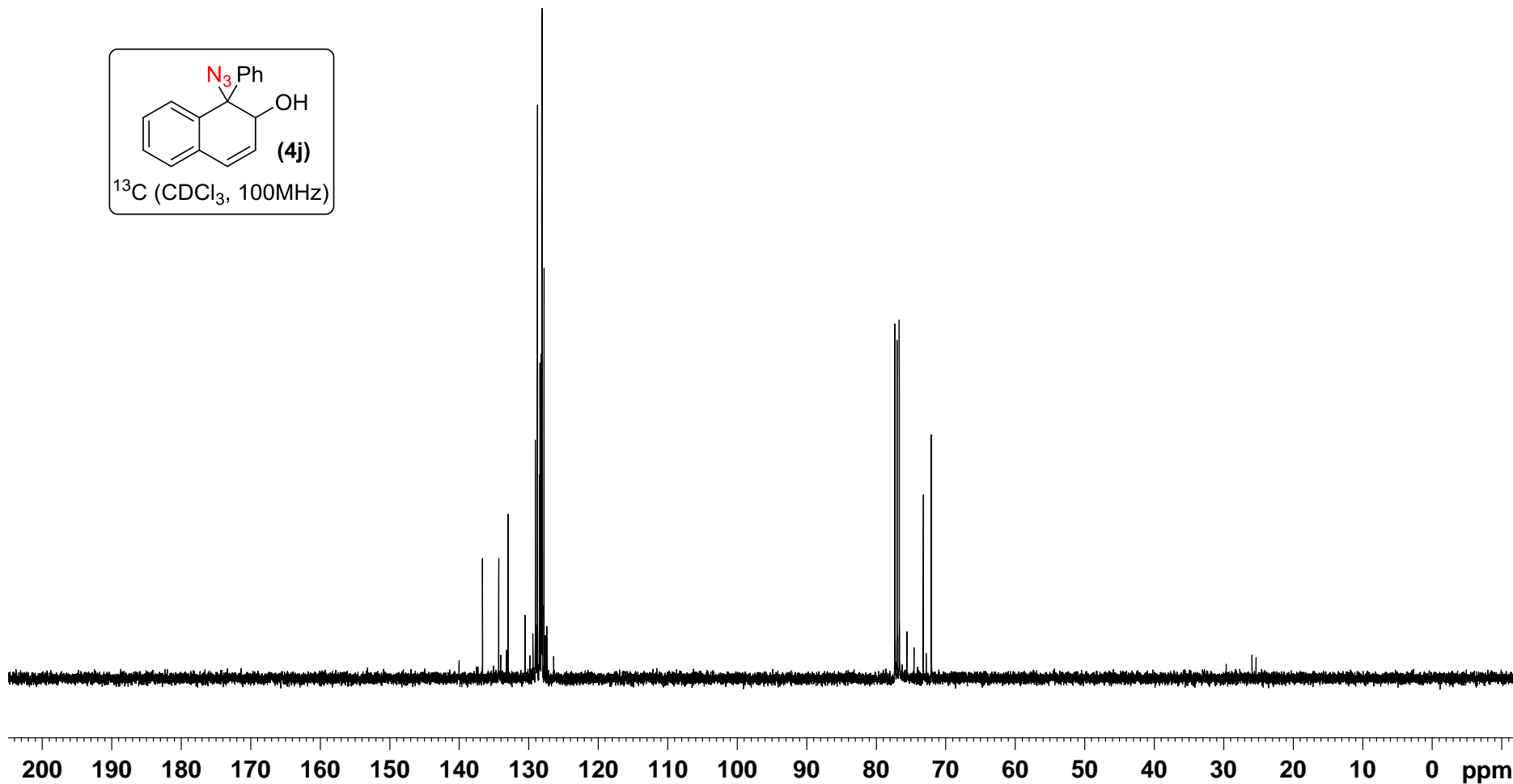
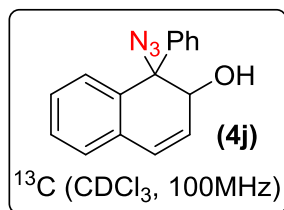


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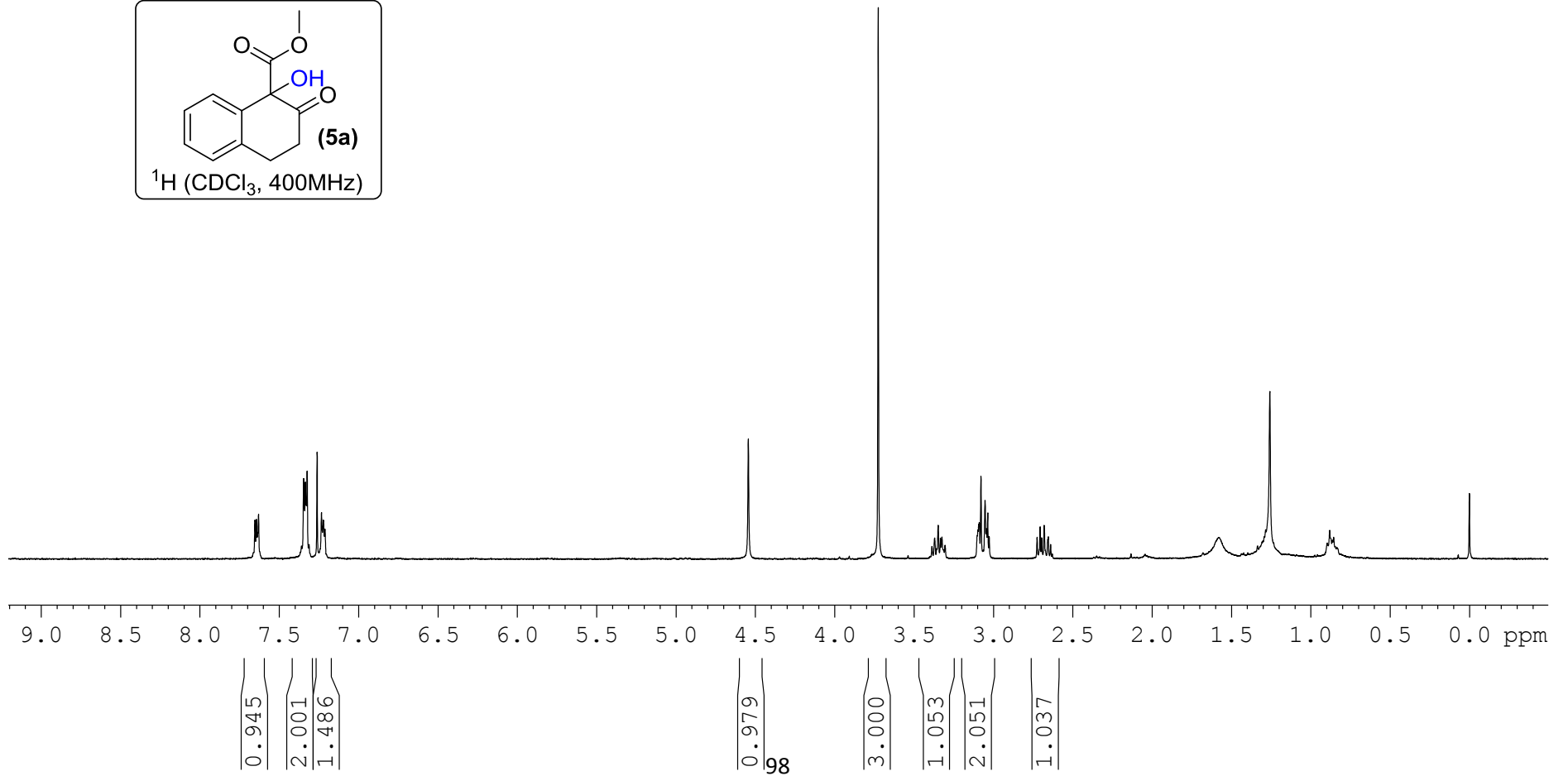
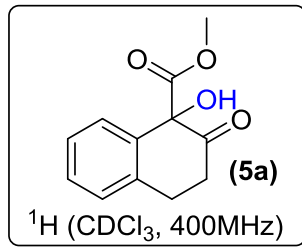
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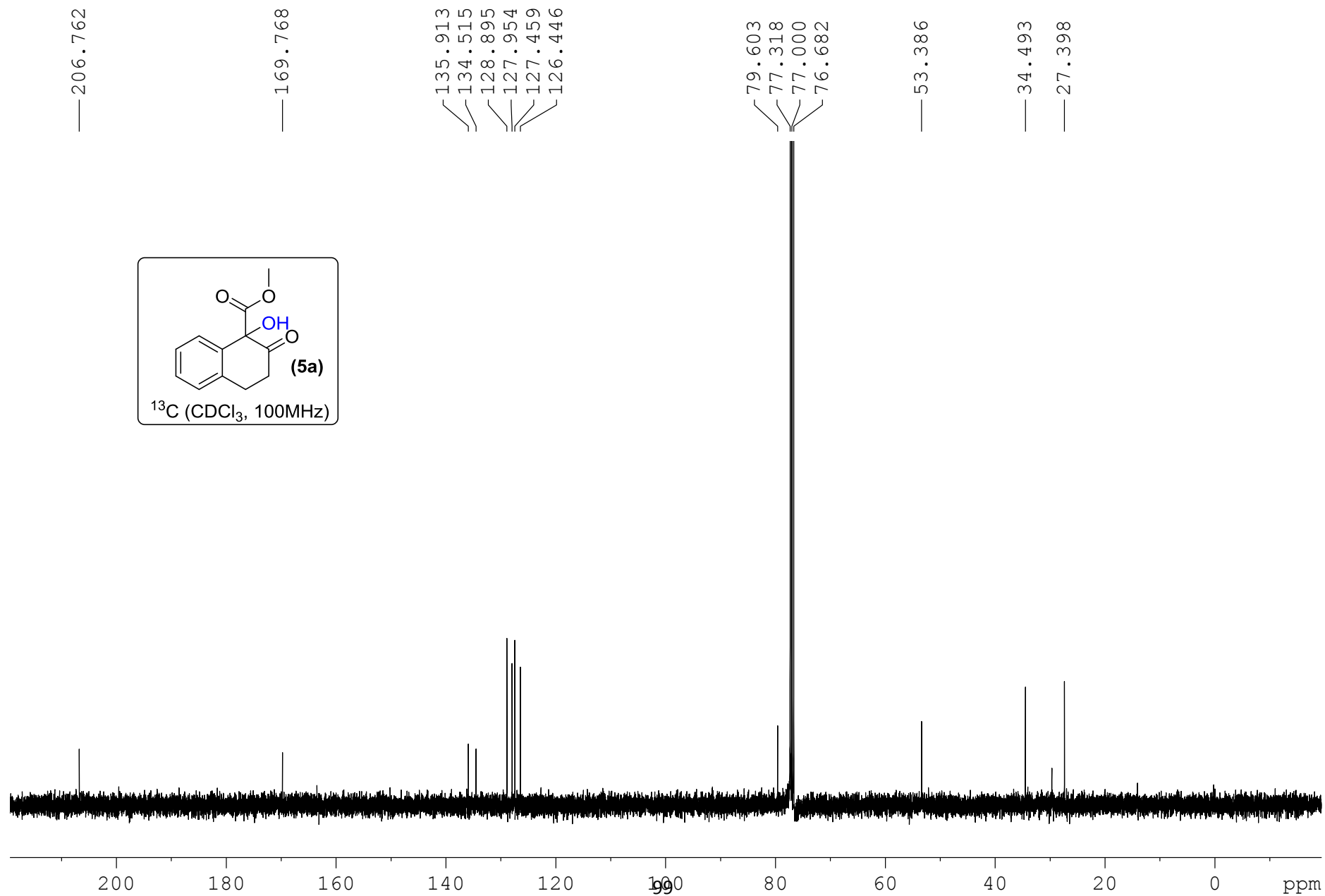
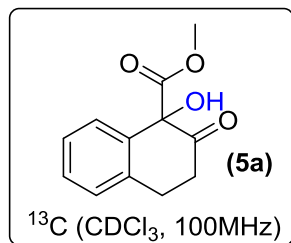


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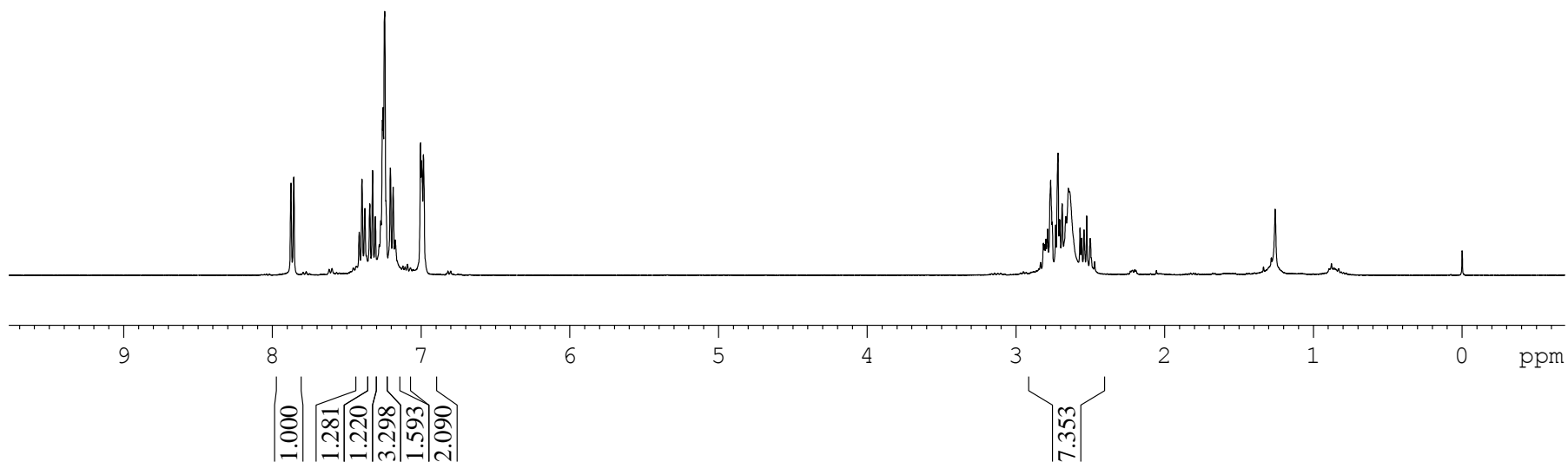
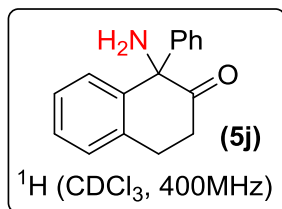


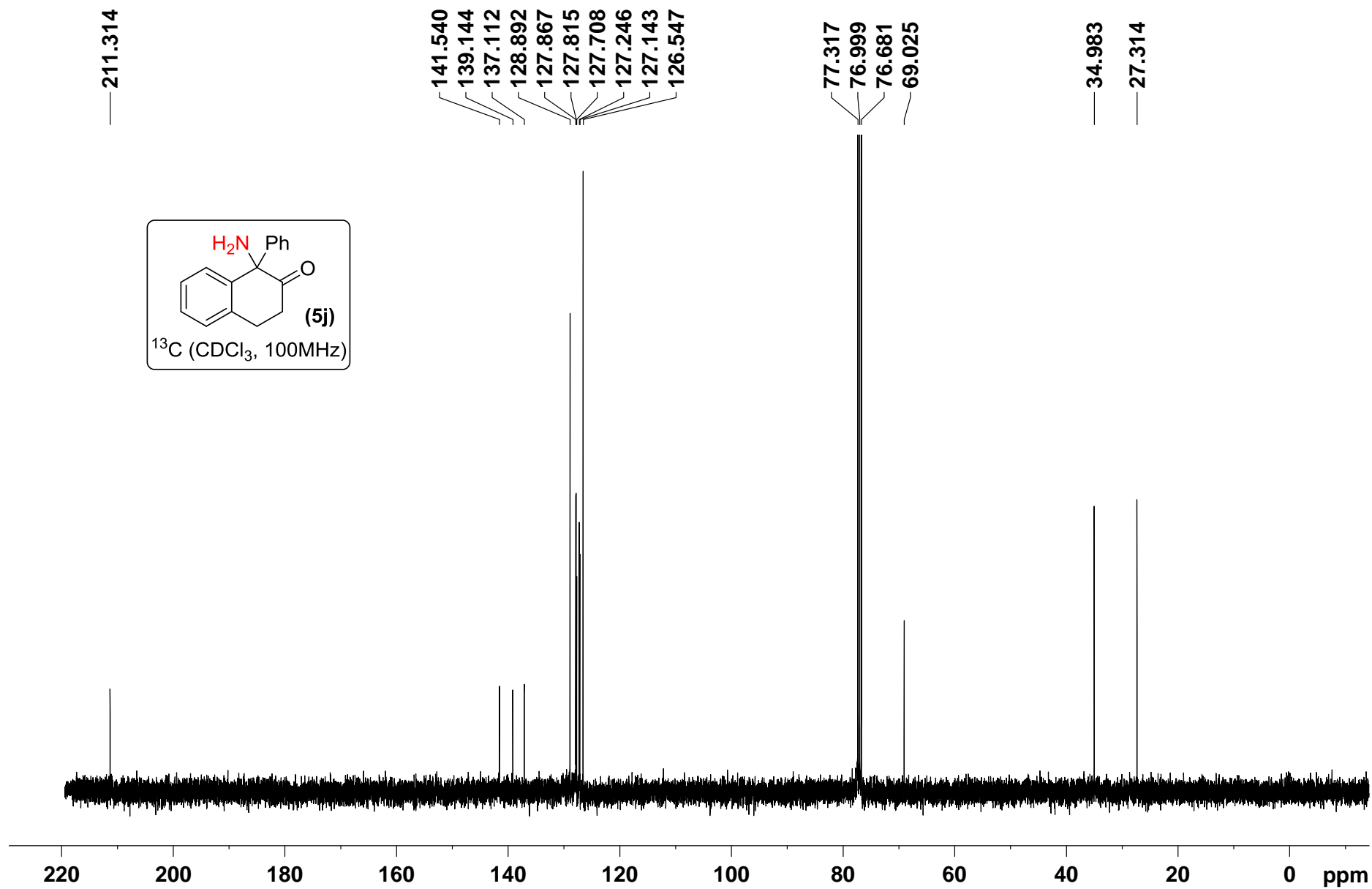


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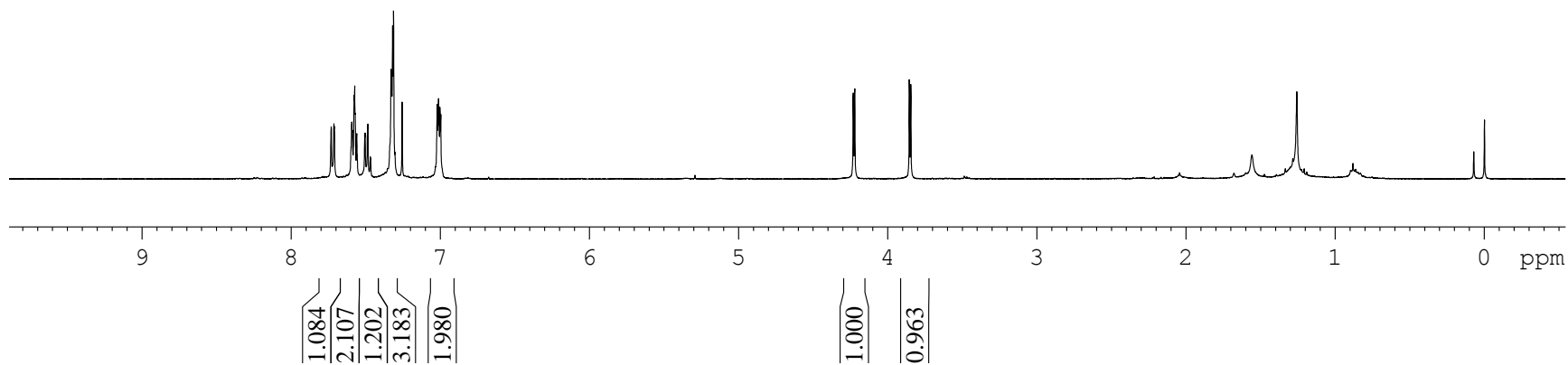
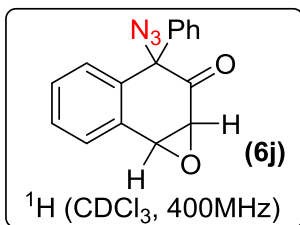




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— 0.000



— 202.152

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126.852

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77.002
76.685
75.958

— 59.229
— 53.402

