

SUPPORTING INFORMATION

Gold(III)-catalyzed azide-yne cyclization/O-H insertion cascade reaction for the expeditious construction of 3-alkoxy-4-quinolinone frameworks

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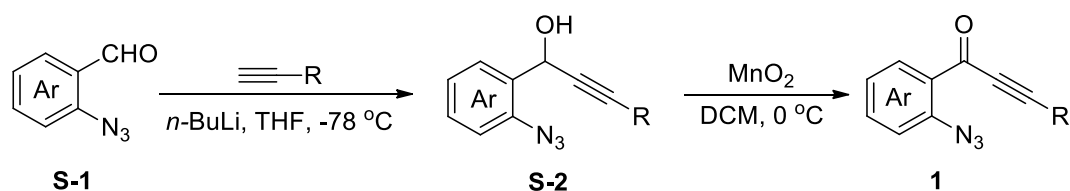
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General Information

All reactions were carried out in oven-dried glassware under an atmosphere of dry argon. Metal catalysts used in this reaction were purchased from commercial sources and used directly. Flash column chromatography was performed using silica gel (300-400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 200-300 mesh silica gel impregnated with a fluorescent indicator (254 nm). ^1H NMR and ^{13}C NMR spectra were recorded on a 400 MHz spectrometer in CDCl_3 or $\text{DMSO}-d_6$; chemical shifts are reported in ppm with the solvent signals as reference, and coupling constants (J) are given in Hertz. The peak information is described as: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, comp = composite. High-resolution mass spectra (HRMS) were recorded on a commercial apparatus (CI Source, GCT PremierTM and BRUKER micrOTF-Q III).

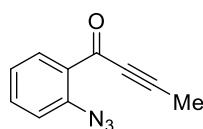
General Procedure for the Synthesis of Azide Alkynes **1**.¹



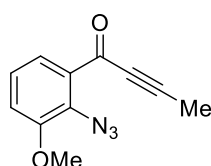
Synthesis of S-2: To a 50-mL oven-dried flask containing a magnetic stirring bar, and alkyne (11.0 mmol) in 15 mL of THF, was added $n\text{-BuLi}$ (4.4 mL, 2.5 M, 11.0 mmol, 1.1 equiv) slowly at $-78\text{ }^\circ\text{C}$ under argon atmosphere. The reaction mixture was stirred for additional 30 mins, then **S-1** (10.0 mmol) in THF (5.0 mL) was added to the above reaction mixture, and the reaction mixture was stirred for 8 h under these conditions. The reaction was quenched with saturated NH_4Cl (20 mL). The organic phase was separated, and the aqueous layer was extracted with DCM ($3 \times 20\text{ mL}$). The combined organic layer was washed with brine (30 mL), dried over anhydrous MgSO_4 and concentrated in *vacuo* after filtration. The residue was purified by column

chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 5:1) to afford pure products **S-2** in >85% yields.

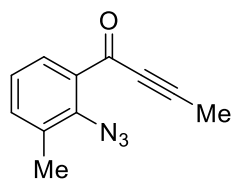
Synthesis of 1: To a 50-mL oven-dried flask containing a magnetic stirring bar, and **S-2** (5.0 mmol) in DCM (10 mL), MnO₂ (6.521 g, 75.0 mmol) was added at room temperature. The resulting reaction mixture was stirred for 5 - 15 mins. After **S-2** was completely consumed (monitored by TLC), the reaction mixture was filtered through a short pad of Celite and the filtrate was concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 30:1) to give **1** in >80% yields. For **1j–1l**, **1n–1w**, and **1z**, the physical and spectral properties identical to those earlier reported.¹



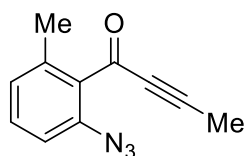
1-(2-Azidophenyl)but-2-yn-1-one (1a). Yellow solid; mp: 39-41 °C. ¹H NMR (400 MHz, CDCl₃) (δ, ppm) 8.09 – 8.07(m, 1H), 7.56 – 7.52 (m, 1H), 7.24 – 7.19 (m, 2H), 2.12 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 176.5, 140.1, 134.2, 133.5, 128.9, 124.5, 120.2, 93.0, 80.4, 4.5. HRMS (TOF MS CI⁺) calculated for C₁₀H₇N₃NaO⁺ [M+Na]⁺: 208.0481, found 208.0487.



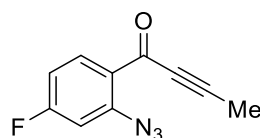
1-(2-Azido-3-methoxyphenyl)but-2-yn-1-one (1b). Yellow solid; mp: 71-73 °C. ¹H NMR (400 MHz, CDCl₃) (δ, ppm) 7.56 – 7.54 (m, 1H), 7.15 (t, *J* = 8.0 Hz, 1H), 7.06 – 7.03 (m, 1H), 3.92 (s, 3H), 2.12 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 177.0, 154.4, 131.2, 128.4, 124.9, 124.0, 115.8, 92.9, 80.9, 56.5, 4.6. HRMS (TOF MS CI⁺) calculated for C₁₁H₉N₃NaO₂⁺ [M+Na]⁺: 238.0587, found 238.0594.



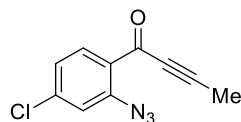
1-(2-Azido-3-methylphenyl)but-2-yn-1-one (1c). Yellow solid; mp: 41-41 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.03 – 8.00 (m, 1H), 7.40 – 7.37 (m, 1H), 7.19 (t, J = 7.7 Hz, 1H), 2.35 (s, 3H), 2.14 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 177.3, 138.7, 135.8, 133.8, 131.5, 130.9, 125.0, 92.7, 80.3, 18.2, 4.5. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_9\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 222.0638, found 222.0635.



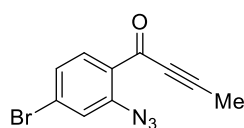
1-(2-Azido-6-methylphenyl)but-2-yn-1-one (1d). Yellow solid; mp: 67-69 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 7.31 (t, J = 7.9 Hz, 1H), 7.03 (d, J = 8.1 Hz, 1H), 6.99 – 6.97 (m, 1H), 2.31 (s, 3H), 2.07 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 181.1, 137.5, 137.0, 132.3, 130.7, 127.2, 116.2, 94.1, 81.6, 19.2, 4.7. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_9\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 222.0638, found 222.0647.



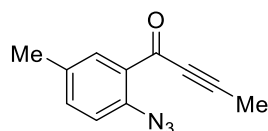
1-(2-Azido-4-fluorophenyl)but-2-yn-1-one (1e). Yellow solid; mp: 94-96 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.12 – 8.02 (m, 1H), 6.88 – 6.80 (m, 2H), 2.05 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.7, 165.6 (d, J = 257.7 Hz), 142.6 (d, J = 9.8 Hz), 136.0 (d, J = 10.6 Hz), 125.1 (d, J = 3.0 Hz), 111.8 (d, J = 21.8 Hz), 107.4 (d, J = 25.2 Hz), 93.0, 79.9, 4.3. ^{19}F NMR (376 MHz, CDCl_3) δ -102.5. HRMS (TOF MS CI^+) calculated for $\text{C}_{10}\text{H}_6\text{FN}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 226.0387, found 226.0381.



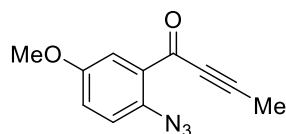
1-(2-Azido-4-chlorophenyl)but-2-yn-1-one (1f). Yellow solid; mp: 91-93 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.03 (d, $J = 8.4$ Hz, 1H), 7.23 – 7.16 (m, 2H), 2.13 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.3, 141.6, 140.4, 134.7, 127.3, 124.9, 120.4, 93.5, 80.2, 4.6. HRMS (TOF MS CI^+) calculated for $\text{C}_{10}\text{H}_6\text{ClN}_3\text{NaO}^+ [\text{M}+\text{Na}]^+$: 242.0092, found 242.0095.



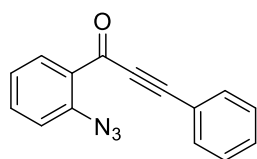
1-(2-Azido-4-bromophenyl)but-2-yn-1-one (1g). Yellow solid; mp: 92-94 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 7.84 (d, $J = 8.3$ Hz, 1H), 7.28 – 7.21 (m, 2H), 2.02 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.4, 141.4, 134.6, 128.7, 127.9, 127.7, 123.3, 93.5, 80.2, 4.6. HRMS (TOF MS CI^+) calculated for $\text{C}_{10}\text{H}_6\text{BrN}_3\text{NaO}^+ [\text{M}+\text{Na}]^+$: 285.9586, found 285.9587.



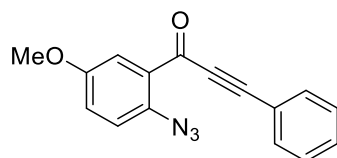
1-(2-Azido-5-methylphenyl)but-2-yn-1-one (1h). Yellow solid; mp: 56-58 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 7.64 (d, $J = 2.3$ Hz, 1H), 7.14 – 7.11 (m, 1H), 6.89 – 6.84 (m, 1H), 2.17 (s, 3H), 1.94 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.7, 136.6, 134.4, 134.0, 133.1, 128.0, 120.0, 92.2, 79.8, 20.1, 3.8. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_9\text{N}_3\text{NaO}^+ [\text{M}+\text{Na}]^+$: 222.0638, found 222.0639.



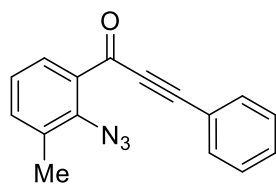
1-(2-Azido-5-methoxyphenyl)but-2-yn-1-one (1i). Yellow solid; mp: 68-70 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.12 (d, $J = 8.8$ Hz, 1H), 6.77 – 6.63 (m, 2H), 3.88 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.1, 164.4, 142.5, 136.4, 122.1, 110.2, 105.7, 91.8, 80.2, 55.9, 4.5. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_9\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 238.0587, found 238.0582.



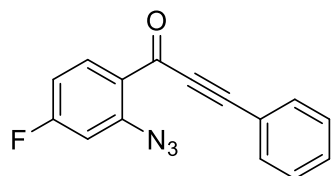
1-(2-Azidophenyl)-3-phenylprop-2-yn-1-one (1j). Yellow solid; mp: 62-65 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.19 – 8.13 (m, 1H), 7.67 – 7.62 (m, 2H), 7.61 – 7.56 (m, 1H), 7.49 – 7.45 (m, 1H), 7.44 – 7.37 (m, 2H), 7.30 – 7.23 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.2, 140.3, 134.4, 133.22, 133.18, 131.0, 129.2, 129.0, 128.8, 124.7, 120.3, 93.5, 88.4. HRMS (TOF MS CI^+) calculated for $\text{C}_{15}\text{H}_9\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 270.0638, found 270.0642.



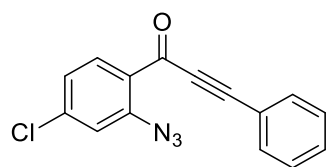
1-(2-Azido-5-methoxyphenyl)-3-phenylprop-2-yn-1-one (1k). Yellow solid; mp: 68-70 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 7.65 – 7.59 (comp, 3H), 7.48 – 7.42 (m, 1H), 7.41 – 7.36 (m, 2H), 7.18 – 7.09 (m, 2H), 3.83 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.7, 156.5, 133.1, 132.7, 131.0, 129.6, 128.7, 121.5, 120.9, 120.2, 116.6, 93.7, 88.5, 55.8. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{11}\text{N}_3\text{NaO}_2^+$ $[\text{M}+\text{Na}]^+$: 300.0743, found 300.0750.



1-(2-Azido-3-methylphenyl)-3-phenylprop-2-yn-1-one (1l). Yellow solid; mp: 70-72 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.10 (d, $J = 7.8, 1.0$ Hz, 1H), 7.66 – 7.61 (m, 2H), 7.46 – 7.44 (m, 1H), 7.41 – 7.35 (m, 3H), 7.22 (t, $J = 7.7$ Hz, 1H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.8, 138.6, 135.9, 133.7, 133.1, 131.3, 130.9, 130.7, 128.7, 125.1, 120.0, 93.0, 88.0, 18.1. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{12}\text{N}_3\text{O}^+$ $[\text{M}+\text{H}]^+$: 262.0975, found 262.0981.

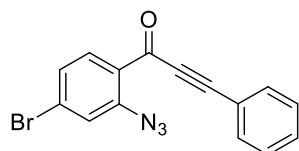


1-(2-Azido-4-fluorophenyl)-3-phenylprop-2-yn-1-one (1m). Yellow solid; mp: 58-60 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.21 (m, $J = 9.2, 6.3$ Hz, 1H), 7.66 – 7.60 (m, 2H), 7.51 – 7.45 (m, 1H), 7.41 – 7.39 (m, 2H), 7.00 – 6.90 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.7, 166.0 (d, $J = 258.0$ Hz), 143.0 (d, $J = 9.5$ Hz), 135.8 (d, $J = 10.5$ Hz), 133.2, 131.0, 128.8, 125.5, 120.1, 112.2 (d, $J = 21.7$ Hz), 107.7 (d, $J = 25.1$ Hz), 93.6, 88.2. ^{19}F NMR (376 MHz, CDCl_3) δ -102.0. HRMS (TOF MS CI^+) calculated for $\text{C}_{15}\text{H}_8\text{FN}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 288.0544, found 288.0540.

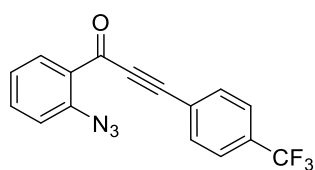


1-(2-Azido-4-chlorophenyl)-3-phenylprop-2-yn-1-one (1n). Yellow solid; mp: 73-75 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.09 (d, $J = 8.3$ Hz, 1H), 7.65 – 7.60 (m, 2H), 7.50 – 7.45 (m, 1H), 7.41 (t, $J = 7.4$ Hz, 2H), 7.25 – 7.20 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.9, 141.6, 140.5, 134.3, 133.2, 131.1, 128.8, 127.3, 125.0,

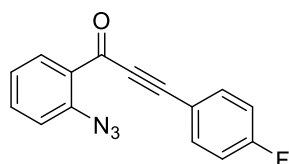
120.4, 120.0, 93.9, 88.2. HRMS (TOF MS Cl^+) calculated for $\text{C}_{15}\text{H}_8\text{ClN}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 304.0248, found 304.0249.



1-(2-Azido-4-bromophenyl)-3-phenylprop-2-yn-1-one (1o). Yellow solid; mp: 91-93 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.00 (d, $J = 8.2$ Hz, 1H), 7.65 – 7.60 (m, 2H), 7.50 – 7.44 (m, 1H), 7.43 – 7.35 (comp, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.1, 141.6, 134.3, 133.3, 131.2, 129.0, 128.9, 128.1, 127.8, 123.4, 120.1, 94.0, 88.3. HRMS (TOF MS Cl^+) calculated for $\text{C}_{15}\text{H}_8\text{BrN}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 347.9743, found 347.9745.

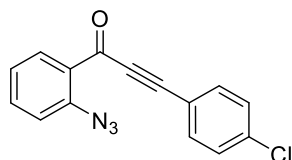


1-(2-Azidophenyl)-3-(4-(trifluoromethyl)phenyl)prop-2-yn-1-one (1p). Yellow oil. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.16 – 8.10 (m, 1H), 7.74 (d, $J = 8.1$ Hz, 2H), 7.65 (d, $J = 8.2$ Hz, 2H), 7.63 – 7.57 (m, 1H), 7.30 – 7.23 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.6, 140.5, 136.0, 134.7, 133.21, 133.15, 132.3 (q, $J = 33.0$ Hz), 128.5, 125.8 (q, $J = 3.7$ Hz), 124.8 (q, $J = 237.1$ Hz), 124.7, 120.3, 90.8, 89.5. ^{19}F NMR (376 MHz, CDCl_3) δ -63.1. HRMS (TOF MS Cl^+) calculated for $\text{C}_{16}\text{H}_8\text{F}_3\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 338.0512, found 338.0500.

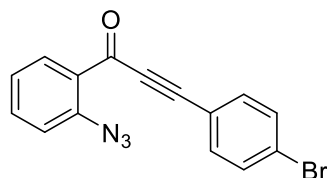


1-(2-Azidophenyl)-3-(4-fluorophenyl)prop-2-yn-1-one (1q). Yellow solid; mp: 82-84 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.16 – 8.10 (m, 1H), 7.66 – 7.55

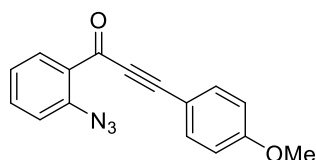
(comp, 3H), 7.30 – 7.23 (m, 2H), 7.13 – 7.06 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.0, 164.1 (d, $J = 254.0$ Hz), 140.3, 135.4 (d, $J = 8.9$ Hz), 134.4, 133.1, 128.9, 124.7, 120.3, 116.4 (d, $J = 3.6$ Hz), 116.3 (d, $J = 22.4$ Hz), 92.3, 88.4. ^{19}F NMR (376 MHz, CDCl_3) δ -105.9. HRMS (TOF MS CI^+) calculated for $\text{C}_{15}\text{H}_8\text{FN}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 288.0544, found 288.0552.



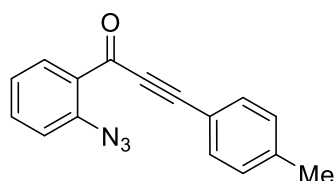
1-(2-Azidophenyl)-3-(4-chlorophenyl)prop-2-yn-1-one (1r). Yellow solid; mp: 86-88 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.16 – 8.09 (m, 1H), 7.61 – 7.53 (comp, 3H), 7.38 (d, $J = 8.5$ Hz, 2H), 7.26 (t, $J = 8.4$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.9, 140.3, 137.3, 134.5, 134.3, 133.1, 129.2, 128.8, 124.7, 120.3, 118.7, 92.0, 89.1. HRMS (TOF MS CI^+) calculated for $\text{C}_{15}\text{H}_8\text{ClN}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 304.0248, found 304.0254.



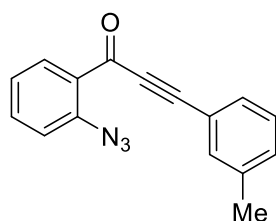
1-(2-Azidophenyl)-3-(4-bromophenyl)prop-2-yn-1-one (1s). Yellow solid; mp: 85-88 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.17 – 8.09 (m, 1H), 7.63 – 7.56 (m, 1H), 7.56 – 7.52 (m, 2H), 7.51 – 7.46 (m, 2H), 7.30 – 7.23 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.7, 140.2, 134.4, 134.3, 133.1, 132.1, 128.6, 125.7, 124.6, 120.2, 119.1, 91.9, 89.1. HRMS (TOF MS CI^+) calculated for $\text{C}_{15}\text{H}_8\text{BrN}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 347.9743, found 347.9744.



1-(2-Azidophenyl)-3-(4-methoxyphenyl)prop-2-yn-1-one (1t). Yellow solid; mp: 78-81 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.16 (d, $J = 7.7$ Hz, 1H), 7.63 – 7.58 (comp, 3H), 7.28 (t, $J = 7.9$ Hz, 2H), 6.94 (d, $J = 8.5$ Hz, 2H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.2, 161.9, 140.1, 135.3, 134.1, 133.0, 129.3, 124.7, 120.2, 114.5, 112.0, 94.8, 88.5, 55.6. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{11}\text{N}_3\text{NaO}_2^+$ $[\text{M}+\text{Na}]^+$: 300.0743, found 300.0730.

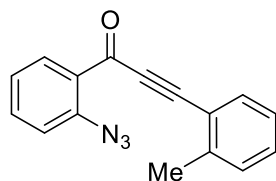


1-(2-Azidophenyl)-3-(p-tolyl)prop-2-yn-1-one (1u). Yellow solid; mp: 76-78 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 7.92 (d, $J = 1.4$ Hz, 1H), 7.67 – 7.60 (m, 2H), 7.51 – 7.44 (m, 1H), 7.43 – 7.35 (comp, 3H), 7.15 (d, $J = 8.2$ Hz, 1H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.2, 141.7, 140.1, 134.2, 133.2, 133.1, 129.6, 129.1, 124.6, 120.2, 117.1, 94.1, 88.3, 21.8. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{11}\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 284.0794, found 284.0797.

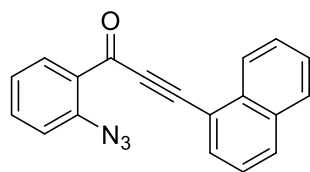


1-(2-Azidophenyl)-3-(m-tolyl)prop-2-yn-1-one (1v). Yellow oil. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.16 – 8.11 (m, 1H), 7.58 – 7.51 (m, 1H), 7.43 – 7.38 (m, 2H), 7.27 – 7.19 (comp, 4H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.8, 139.9, 138.4, 134.2, 133.4, 133.0, 131.7, 130.1, 128.6, 128.5, 124.5, 120.1, 119.7, 93.6, 88.0, 21.0. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{11}\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 284.0794, found

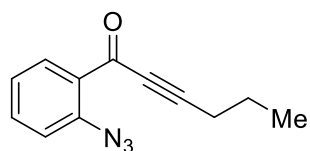
284.0784.



1-(2-Azidophenyl)-3-(o-tolyl)prop-2-yn-1-one (1w). Yellow oil. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.21 – 8.10 (m, 1H), 7.64 – 7.53 (m, 2H), 7.39 – 7.33 (m, 1H), 7.30 – 7.18 (comp, 4H), 2.55 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.2, 142.3, 140.2, 134.2, 133.8, 133.0, 131.0, 130.0, 129.2, 126.0, 124.7, 120.2, 120.1, 92.6, 92.3, 20.8. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{11}\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 284.0794, found 284.0804.

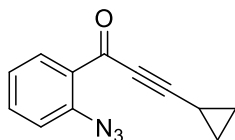


1-(2-Azidophenyl)-3-(naphthalen-1-yl)prop-2-yn-1-one (1x). Yellow solid; mp: 59-61 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.42 (d, J = 8.3 Hz, 1H), 8.24 – 8.21 (m, 1H), 7.96 (d, J = 8.2 Hz, 1H), 7.92 – 7.88 (m, 2H), 7.64 – 7.54 (comp, 3H), 7.49 (t, J = 7.7 Hz, 1H), 7.29 (t, J = 7.8 Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.0, 140.2, 134.3, 133.8, 133.4, 133.1, 132.9, 131.7, 129.2, 128.7, 127.8, 127.0, 125.8, 125.3, 124.7, 120.2, 117.9, 93.3, 92.0. HRMS (TOF MS CI^+) calculated for $\text{C}_{19}\text{H}_{11}\text{N}_3\text{NaO}^+$ $[\text{M}+\text{Na}]^+$: 320.0794, found 320.0783.



1-(2-Azidophenyl)hex-2-yn-1-one (1y). Yellow oil. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.15 – 8.11 (m, 1H), 7.64 – 7.56 (m, 1H), 7.33 – 7.27 (m, 2H), 2.51 (t, J = 7.0 Hz, 2H), 1.74 (p, J = 7.2 Hz, 2H), 1.12 (t, J = 7.4 Hz, 3H). ^{13}C NMR (100 MHz,

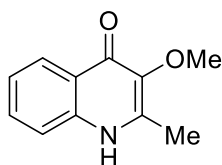
CDCl₃) δ 176.5, 140.1, 134.1, 133.3, 129.2, 124.6, 120.3, 97.2, 81.4, 21.43, 21.39, 13.7. HRMS (TOF MS CI⁺) calculated for C₁₂H₁₁N₃NaO⁺ [M+Na]⁺: 236.0794, found 236.0783.



1-(2-Azidophenyl)-3-cyclopropylprop-2-yn-1-one (1z). Yellow oil. ¹H NMR (400 MHz, CDCl₃) (δ , ppm) 7.99 – 7.91 (m, 1H), 7.51 – 7.41 (m, 1H), 7.19 – 7.10 (m, 2H), 1.49 – 1.40 (m, 1H), 1.01 – 0.89 (comp, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 175.9, 139.6, 133.8, 132.9, 128.9, 124.4, 120.0, 101.5, 76.9, 9.9. HRMS (TOF MS CI⁺) calculated for C₁₂H₉N₃NaO⁺ [M+Na]⁺: 234.0638, found 234.0654.

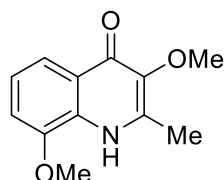
General Procedure for the Reaction of Azide Alkynes **1** with **2a**.

To a 10-mL oven-dried vial containing a magnetic stirring bar, azide alkynes **1** (0.2 mmol), AuCl₃ (2.3 mg, 5.0 mol %), and methanol **2a** (1.0 mL) were added in sequence at room temperature under argon atmosphere. The resulting reaction mixture was stirred for 8 h under these conditions. When the reaction was completed (monitored by TLC), the solvent was evaporated *in vacuo* and the residue was purified by flash column chromatography on silica gel without additional treatment (DCM/methanol = 80:1 to 20:1) to afford the pure products **3** in good to high yields.

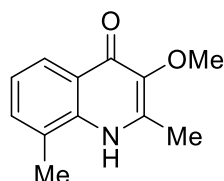


3-Methoxy-2-methylquinolin-4(1H)-one (3a).² 35.9 mg, 95% yield. Yellow solid; mp: 240-242 °C. ¹H NMR (400 MHz, DMSO-*d*₆) (δ , ppm) 11.62 (s, 1H), 8.11 – 8.09 (m, 1H), 7.59 – 7.55 (m, 1H), 7.51 (d, *J* = 8.3 Hz, 1H), 7.28 – 7.21 (m, 1H), 3.74 (s,

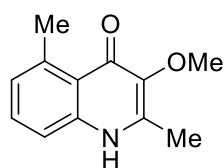
3H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 171.0, 141.9, 139.6, 138.3, 130.9, 125.5, 124.8, 122.1, 117.7, 58.9, 14.4. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_{11}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 212.0682, found 212.0687.



3,8-Dimethoxy-2-methylquinolin-4(1H)-one (3b). 35.9 mg, 82% yield. Yellow solid; mp: 227-229 °C. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) (δ , ppm) 10.97 (s, 1H), 7.65 (d, J = 3.0 Hz, 1H), 7.18 (d, J = 6.6 Hz, 2H), 3.98 (s, 3H), 3.72 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 170.7, 148.2, 141.9, 140.1, 129.2, 126.5, 122.1, 116.1, 110.2, 58.8, 56.1, 14.1. HRMS (TOF MS CI^+) calculated for $\text{C}_{12}\text{H}_{13}\text{NNaO}_3^+$ $[\text{M}+\text{Na}]^+$: 242.0788, found 242.0784.

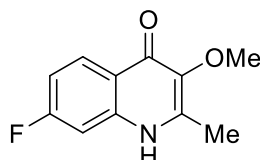


3-Methoxy-2,8-dimethylquinolin-4(1H)-one (3c). 17.5 mg, 43% yield. Yellow solid; mp: 225-227 °C. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) (δ , ppm) 10.29 (s, 1H), 8.05 – 7.89 (m, 1H), 7.47 – 7.40 (m, 1H), 7.17 – 7.13 (m, 1H), 3.73 (s, 3H), 2.52 (s, 3H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 171.2, 142.2, 139.6, 137.1, 131.8, 125.8, 125.7, 122.9, 121.9, 58.8, 17.6, 14.2. HRMS (TOF MS CI^+) calculated for $\text{C}_{12}\text{H}_{14}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 204.1019, found 204.1017.

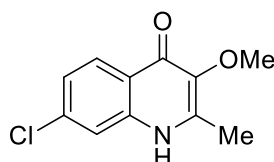


3-Methoxy-2,5-dimethylquinolin-4(1H)-one (3d). 27.6 mg, 68% yield. Yellow solid;

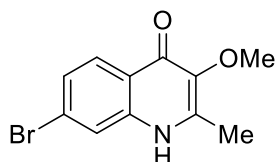
mp: 228-230 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) (δ , ppm) 11.28 (s, 1H), 7.38 – 7.34 (m, 1H), 7.30 (d, $J = 8.9$ Hz, 1H), 6.91 – 6.89 (m, 1H), 3.69 (s, 3H), 2.82 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 173.7, 140.7, 140.01, 139.96, 139.1, 130.0, 124.4, 123.6, 115.8, 58.8, 23.2, 14.0. HRMS (TOF MS CI^+) calculated for $\text{C}_{12}\text{H}_{13}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 226.0838, found 226.0831.



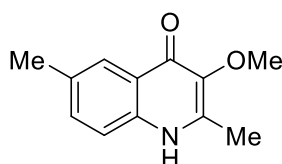
7-Fluoro-3-methoxy-2-methylquinolin-4(1H)-one (3e). 32.3 mg, 78% yield. Yellow solid; mp: 174-176°C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) (δ , ppm) 11.61 (s, 1H), 8.14 – 8.10 (m, 1H), 7.30 – 6.99 (m, 2H), 3.72 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 170.8, 163.5 (d, $J = 246.9$ Hz), 142.3, 139.7, 139.4 (d, $J = 12.6$ Hz), 128.2 (d, $J = 10.6$ Hz), 122.5, 111.2 (d, $J = 23.6$ Hz), 102.6 (d, $J = 25.0$ Hz), 59.0, 14.4. ^{19}F NMR (376 MHz, $\text{DMSO}-d_6$) δ -109.1. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_{10}\text{FNNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 230.0588, found 230.0583.



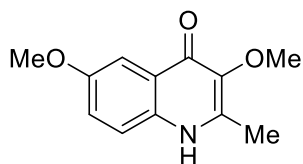
7-Chloro-3-methoxy-2-methylquinolin-4(1H)-one (3f). 38.9 mg, 87% yield. Yellow solid; mp: 262-264 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) (δ , ppm) 11.64 (s, 1H), 8.07 (d, $J = 8.7$ Hz, 1H), 7.50 (d, $J = 2.0$ Hz, 1H), 7.26 – 7.24 (m, 1H), 3.73 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 170.7, 142.4, 140.0, 138.9, 135.5, 127.2, 124.1, 122.6, 116.7, 59.0, 14.4. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_{11}\text{ClNO}_2^+$ $[\text{M}+\text{H}]^+$: 224.0473, found 224.0480.



7-Bromo-3-methoxy-2-methylquinolin-4(1H)-one (3g). 44.5 mg, 83% yield. Yellow solid; mp: 262-264 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.64 (s, 1H), 8.01 – 7.99 (d, J = 8.5 Hz, 1H), 7.67 (d, J = 1.7 Hz, 1H), 7.38 – 7.35 (m, 1H), 3.73 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 170.6, 142.3, 139.9, 139.1, 127.2, 125.2, 124.3, 124.2, 119.8, 58.9, 14.4. HRMS (TOF MS CI^+) calculated for $\text{C}_{11}\text{H}_{11}\text{BrNO}_2^+$ $[\text{M}+\text{H}]^+$: 267.9968, found 267.9974.

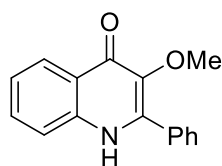


3-Methoxy-2,6-dimethylquinolin-4(1H)-one (3h). 31.7 mg, 78% yield. Yellow solid; mp: 192-194 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.46 (s, 1H), 7.87 (s, 1H), 7.40 (d, J = 1.4 Hz, 2H), 3.72 (s, 3H), 2.37 (d, J = 12.0 Hz, 6H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 170.8, 141.4, 139.4, 136.4, 132.3, 131.3, 125.5, 124.0, 117.6, 58.9, 20.7, 14.3. HRMS (TOF MS CI^+) calculated for $\text{C}_{12}\text{H}_{14}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 204.1019, found 204.1026.

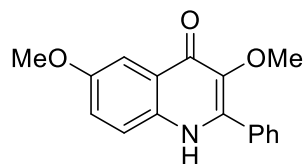


3,6-Dimethoxy-2-methylquinolin-4(1H)-one (3i). 32.9 mg, 75% yield. Yellow solid; mp: 187-189 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.36 (s, 1H), 8.02 – 7.92 (m, 1H), 6.85 (d, J = 7.2 Hz, 2H), 3.83 (s, 3H), 3.71 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 170.9, 161.3, 141.0, 140.0, 139.2, 126.6, 119.8, 112.5, 98.3, 59.0, 55.34 14.3. HRMS (TOF MS CI^+) calculated for $\text{C}_{12}\text{H}_{13}\text{NNaO}_3^+$ $[\text{M}+\text{Na}]^+$:

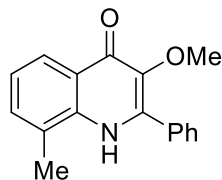
242.0788, found 242.0793.



3-Methoxy-2-phenylquinolin-4(1H)-one (3j). 47.7 mg, 95% yield. Yellow solid; mp: 173-175 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.69 (s, 1H), 8.18 – 8.15 (m, 1H), 7.72 – 7.60 (comp, 4H), 7.60 – 7.53 (comp, 3H), 7.32 – 7.28 (m, 1H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 172.1, 142.4, 138.9, 138.6, 132.2, 131.1, 129.5, 129.0, 128.2, 125.4, 124.6, 122.4, 118.3, 59.2. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{14}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 252.1019, found 252.1020.

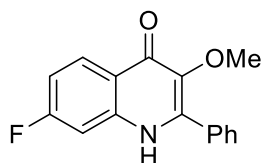


3,6-Dimethoxy-2-phenylquinolin-4(1H)-one (3k). 41.0 mg, 73% yield. Yellow solid; mp: 191-193 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.64 (s, 1H), 7.69 – 7.62 (comp, 3H), 7.59 – 7.53 (comp, 4H), 7.30 – 7.27 (m, 1H), 3.85 (s, 3H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 171.3, 155.1, 141.7, 138.4, 133.5, 132.4, 129.5, 129.1, 128.3, 126.6, 122.1, 120.2, 103.6, 59.2, 55.3. HRMS (TOF MS CI^+) calculated for $\text{C}_{17}\text{H}_{16}\text{NO}_3^+$ $[\text{M}+\text{H}]^+$: 282.1125, found 282.1129.

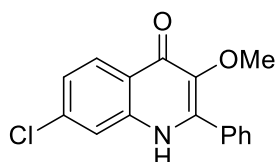


3-Methoxy-8-methyl-2-phenylquinolin-4(1H)-one (3l). 41.3mg, 78% yield. White solid; mp: 232-234 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 10.39 (s, 1H), 8.06 (d, J = 8.1 Hz, 1H), 7.68 – 7.66 (m, 2H), 7.56 – 7.53 (comp, 3H), 7.47 (d, J = 7.1 Hz,

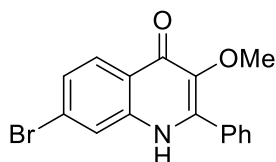
1H), 7.22 (t, $J = 7.6$ Hz, 1H), 3.64 (s, 3H), 2.56 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 172.5, 142.8, 139.1, 137.4, 132.4, 132.2, 129.5, 129.4, 128.1, 126.8, 125.9, 122.8, 122.4, 59.2, 17.6. HRMS (TOF MS Cl^+) calculated for $\text{C}_{17}\text{H}_{15}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 288.0995, found 288.1008.



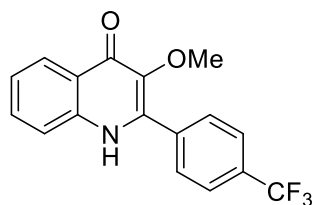
7-Fluoro-3-methoxy-2-phenylquinolin-4(1H)-one (3m). 44.1 mg, 82% yield. White solid; mp: 245-247 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.69 (s, 1H), 8.22 – 8.18 (m, 1H), 7.68 – 7.65 (m, 2H), 7.61 – 7.52 (comp, 3H), 7.39 – 7.36 (m, 1H), 7.18 – 7.13 (m, 1H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 171.9, 163.6 (d, $J = 247.3$ Hz), 142.7, 139.9 (d, $J = 12.7$ Hz), 139.1, 132.1, 129.8, 129.1, 128.4, 128.1 (d, $J = 10.7$ Hz), 122.6, 111.6 (d, $J = 23.8$ Hz), 103.2 (d, $J = 25.0$ Hz), 59.3. ^{19}F NMR (376 MHz, DMSO- d_6) δ -108.35. HRMS (TOF MS Cl^+) calculated for $\text{C}_{16}\text{H}_{13}\text{FNO}_2^+$ $[\text{M}+\text{H}]^+$: 270.0925, found 270.0920.



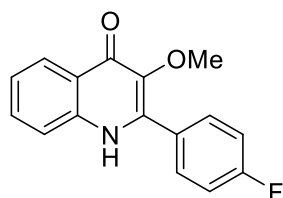
7-Chloro-3-methoxy-2-phenylquinolin-4(1H)-one (3n). 48.5 mg, 85% yield. Yellow solid; mp: 240-242 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.72 (s, 1H), 8.16 – 8.14 (m, 1H), 7.72 – 7.65 (comp, 3H), 7.59 – 7.57 (comp, 3H), 7.33 – 7.31 (m, 1H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 171.8, 142.7, 139.4, 139.3, 135.9, 132.0, 129.8, 129.1, 128.5, 127.2, 124.1, 122.9, 117.4, 59.3. HRMS (TOF MS Cl^+) calculated for $\text{C}_{16}\text{H}_{13}\text{ClNO}_2^+$ $[\text{M}+\text{H}]^+$: 286.0629, found 286.0632.



7-Bromo-3-methoxy-2-phenylquinolin-4(1H)-one (3o). 57.9 mg, 88% yield. Yellow solid; mp: 251-253 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.73 (s, 1H), 8.08 – 8.05 (m, 1H), 7.88 – 7.87 (m, 1H), 7.70 – 7.65 (m, 2H), 7.61 – 7.54 (comp, 3H), 7.43 – 7.40 (m, 1H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 171.8, 142.7, 139.5, 139.4, 132.0, 129.8, 129.1, 128.4, 127.1, 125.5, 124.6, 124.3, 120.6, 59.3. HRMS (TOF MS CI^+) calculated for $\text{C}_{16}\text{H}_{13}\text{BrNO}_2^+$ $[\text{M}+\text{H}]^+$: 330.0124, found 330.0118.

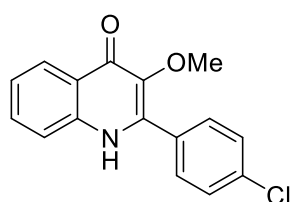


3-Methoxy-2-(4-(trifluoromethyl)phenyl)quinolin-4(1H)-one (3p). 19.1 mg, 30% yield. White solid; mp: 178-180 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.79 (s, 1H), 8.19 – 8.17 (m, 1H), 7.97 – 7.89 (comp, 4H), 7.71 – 7.61 (m, 2H), 7.34 – 7.30 (m, 1H), 3.71 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 172.4, 141.1, 139.3, 138.8, 136.3, 131.6, 130.3, 130.0 (q, $J = 32.1$ Hz), 128.0 (q, $J = 237.1$ Hz), 125.7, 125.31 (q, $J = 3.6$ Hz), 124.9, 122.8, 118.5, 59.5. ^{19}F NMR (376 MHz, DMSO- d_6) δ -61.3. HRMS (TOF MS CI^+) calculated for $\text{C}_{17}\text{H}_{12}\text{F}_3\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 342.0712, found 342.0714.

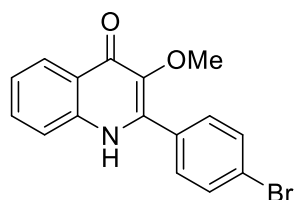


2-(4-Fluorophenyl)-3-methoxyquinolin-4(1H)-one (3q). 44.1 mg, 82% yield. Yellow solid; mp: 209-245 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.86 (s,

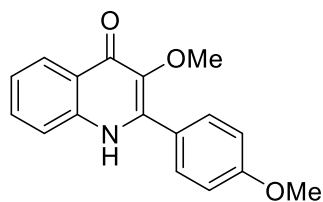
1H), 8.16 – 8.13 (m, 1H), 7.83 – 7.70 (comp, 3H), 7.68 – 7.60 (m, 1H), 7.44 – 7.36 (m, 2H), 7.32 – 7.28 (m, 1H), 3.66 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 172.2, 162.7 (d, *J* = 247.2 Hz), 141.5, 139.0, 138.7, 131.7 (d, *J* = 8.5 Hz), 131.2, 128.5 (d, *J* = 3.2 Hz), 125.5, 124.7, 122.5, 118.5, 115.3 (d, *J* = 21.6 Hz), 59.2. ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -111.4. HRMS (TOF MS Cl⁺) calculated for C₁₆H₁₃FNO₂⁺ [M+H]⁺: 270.0925, found 270.0921.



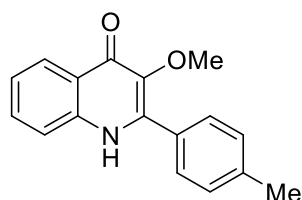
2-(4-Chlorophenyl)-3-methoxyquinolin-4(1H)-one (3r). 48.5 mg, 85% yield. Yellow solid; mp: 254-256 °C. ¹H NMR (400 MHz, DMSO-*d*₆) 11.69 (s, 1H), 8.17 – 8.14 (m, 1H), 7.73 – 7.67 (m, 2H), 7.67 – 7.61 (m, 4H), 7.32 – 7.28 (m, 1H), 3.67 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 172.6, 141.6, 139.5, 139.1, 134.9, 131.8, 131.5, 131.4, 128.8, 126.0, 125.2, 123.0, 118.8, 59.7. HRMS (TOF MS Cl⁺) calculated for C₁₆H₁₃ClNO₂⁺ [M+H]⁺: 286.0629, found 286.0628.



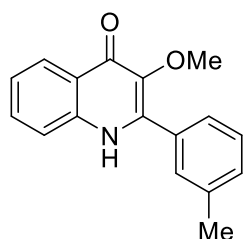
2-(4-Bromophenyl)-3-methoxyquinolin-4(1H)-one (3s). 53.3 mg, 81% yield. Yellow solid; mp: 221-223 °C. ¹H NMR (400 MHz, DMSO-*d*₆) (δ, ppm) 11.77 (s, 1H), 8.17 – 8.15 (m, 1H), 7.79 – 7.73 (m, 2H), 7.72 – 7.60 (comp, 4H), 7.33 – 7.29 (m, 1H), 3.67 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 172.0, 141.4, 139.0, 138.7, 131.5, 131.3, 129.2, 128.4, 125.5, 124.7, 123.2, 122.7, 118.5, 59.4. HRMS (TOF MS Cl⁺) calculated for C₁₆H₁₃BrNO₂⁺ [M+H]⁺: 330.0124, found 330.0129.



3-Methoxy-2-(4-methoxyphenyl)quinolin-4(1H)-one (3t). 51.7 mg, 92% yield. Yellow solid; mp: 226-228 °C. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) (δ , ppm) 11.54 (s, 1H), 8.15 – 8.12 (m, 1H), 7.71 – 7.56 (comp, 4H), 7.31 – 7.27 (m, 1H), 7.18 – 7.07 (m, 2H), 3.85 (s, 3H), 3.65 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 172.0, 160.3, 142.2, 138.9, 138.7, 131.0, 130.6, 125.4, 124.7, 124.3, 122.3, 118.3, 113.8, 59.1, 55.3. HRMS (TOF MS Cl^+) calculated for $\text{C}_{17}\text{H}_{15}\text{NNaO}_3^+$ $[\text{M}+\text{Na}]^+$: 304.0944, found 304.0941.

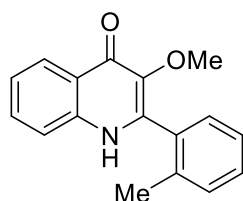


3-Methoxy-2-(p-tolyl)quinolin-4(1H)-one (3u). 45.1 mg, 85% yield. Yellow solid; mp: 262-264 °C. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) (δ , ppm) 11.61 (s, 1H), 8.17 – 8.15 (m, 1H), 7.70 – 7.68 (m, 1H), 7.64 – 7.54 (m, 3H), 7.38 – 7.35 (m, 2H), 7.33 – 7.25 (m, 1H), 3.66 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100MHz, $\text{DMSO-}d_6$) δ 172.2, 142.5, 139.3, 139.0, 138.7, 131.2, 129.4, 129.0, 128.9, 125.5, 124.8, 122.4, 118.4, 59.2, 21.0. HRMS (TOF MS Cl^+) calculated for $\text{C}_{17}\text{H}_{15}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 288.0995, found 288.0989.

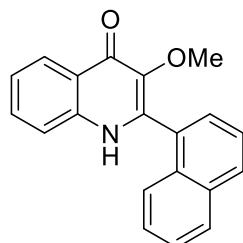


3-Methoxy-2-(m-tolyl)quinolin-4(1H)-one (3v). 45.1 mg, 85% yield. Yellow solid; mp: 194-196 °C. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) (δ , ppm) 11.66 (s, 1H), 8.18 – 8.16

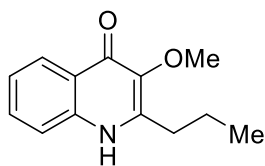
(m, 1H), 7.70 (d, $J = 8.4$ Hz, 1H), 7.65 – 7.59 (m, 1H), 7.43 – 7.48 (comp, 3H), 7.35 (d, $J = 6.7$ Hz, 1H), 7.32 – 7.26 (m, 1H), 3.68 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 172.3, 142.7, 139.1, 138.7, 137.6, 132.3, 131.2, 130.2, 129.5, 128.3, 126.4, 125.5, 124.8, 122.5, 118.4, 59.3, 21.0. HRMS (TOF MS CI^+) calculated for $\text{C}_{17}\text{H}_{16}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 266.1176, found 266.1182.



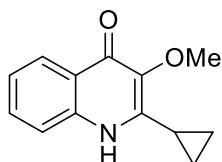
3-Methoxy-2-(o-tolyl)quinolin-4(1H)-one (3w). 43.5 mg, 82% yield. White solid; mp: 271-273 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.73 (s, 1H), 8.18 (d, $J = 8.2$ Hz, 1H), 7.66 – 7.57 (m, 2H), 7.46 – 7.28 (comp, 5H), 3.57 (s, 3H), 2.21 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 172.0, 142.7, 139.2, 138.6, 136.3, 132.4, 131.2, 130.0, 129.4, 129.3, 125.7, 125.7, 124.8, 122.5, 118.3, 59.1, 19.2. HRMS (TOF MS CI^+) calculated for $\text{C}_{17}\text{H}_{15}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 288.0995, found 288.0996.



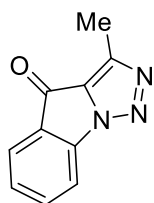
3-Methoxy-2-(naphthalen-1-yl)quinolin-4(1H)-one (3x). 40.9 mg, 68% yield. Yellow solid; mp: 202-204 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.90 (s, 1H), 8.27 – 8.22 (m, 1H), 8.15 – 8.12 (m, 1H), 8.08 – 8.06 (m, 1H), 7.71 – 7.67 (m, 2H), 7.66 – 7.57 (comp, 4H), 7.57 – 7.52 (m, 1H), 7.37 – 7.33 (m, 1H), 3.55 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 172.0, 141.5, 140.1, 138.7, 133.0, 131.6, 130.8, 130.3, 129.6, 128.3, 127.4, 127.0, 126.4, 125.9, 125.3, 125.1, 124.9, 122.5, 118.3, 59.2. HRMS (TOF MS CI^+) calculated for $\text{C}_{20}\text{H}_{16}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 302.1176, found 302.1181.



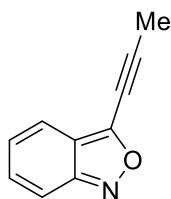
3-Methoxy-2-propylquinolin-4(1H)-one (3y). 36.0 mg, 83% yield. Yellow solid; mp: 166–168 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.55 (s, 1H), 8.18 – 8.09 (m, 1H), 7.64 – 7.55 (m, 2H), 7.25 – 7.25 (m, 1H), 3.80 (s, 3H), 2.76 – 2.67 (m, 2H), 1.77 – 1.65 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 171.2, 145.3, 139.4, 138.3, 130.9, 125.4, 124.8, 122.1, 117.8, 59.2, 30.2, 21.9, 13.7. HRMS (TOF MS CI^+) calculated for $\text{C}_{13}\text{H}_{16}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 218.1176, found 218.1179.



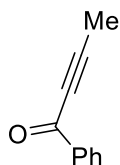
2-Cyclopropyl-3-methoxyquinolin-4(1H)-one (3z). 30.5 mg, 71% yield. Yellow solid; mp: 159–162 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) δ 10.58 (s, 1H), 8.09 – 8.07 (m, 1H), 7.68 (d, J = 8.4 Hz, 1H), 7.57 – 7.53 (m, 1H), 7.29 – 7.20 (m, 1H), 3.77 (s, 3H), 2.39 – 2.32 (m, 1H), 1.12 (comp, 4H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 169.4, 147.6, 140.1, 138.2, 130.9, 124.6, 124.4, 122.7, 118.2, 59.6, 9.9, 8.2. HRMS (TOF MS CI^+) calculated for $\text{C}_{13}\text{H}_{14}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 216.1019, found 216.1025.



3-Methyl-4H-[1,2,3]triazolo[1,5-a]indol-4-one (4a). Yellow solid; mp: 138–140 °C. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 7.73 – 7.67 (m, 2H), 7.65 – 7.61 (m, 1H), 7.39 – 7.35 (m, 1H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.6, 143.4, 140.6, 135.7, 131.3, 130.0, 128.8, 125.8, 112.8, 11.2. HRMS (TOF MS CI^+) calculated for $\text{C}_{10}\text{H}_8\text{N}_3\text{O}^+$ $[\text{M}+\text{H}]^+$: 186.0667, found 186.0667.



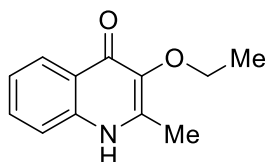
3-(Prop-1-yn-1-yl)benzo[c]isoxazole (5a). Yellow oil. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 7.55 – 7.47 (m, 2H), 7.26 – 7.22 (m, 1H), 6.99 – 6.95 (m, 1H), 2.21 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 157.0, 149.9, 131.0, 124.7, 119.8, 119.6, 115.5, 101.9, 67.1, 5.1. HRMS (TOF MS CI^+) calculated for $\text{C}_{10}\text{H}_8\text{NO}^+$ $[\text{M}+\text{H}]^+$: 158.0600, found 158.0591.



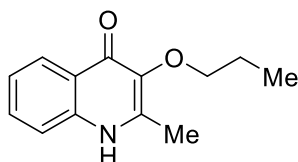
1-Phenylbut-2-yn-1-one (6a). Yellow oil. ^1H NMR (400 MHz, CDCl_3) (δ , ppm) 8.15 – 8.12 (m, 2H), 7.62 – 7.56 (m, 1H), 7.49 – 7.45 (m, 2H), 2.15 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 178.4, 136.9, 134.1, 129.7, 128.6, 92.6, 79.1, 4.5. HRMS (TOF MS CI^+) calculated for $\text{C}_{10}\text{H}_9\text{O}^+$ $[\text{M}+\text{H}]^+$: 145.0648, found 145.0647.

General Procedure for the Reaction of **1a** with Alcohols **2**.

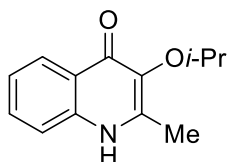
To a 10-mL oven-dried vial containing a magnetic stirring bar, azide alkynes **1a** (37.0 mg, 0.2 mmol), AuCl_3 (2.3 mg, 5.0 mol %), alcohols **2** (0.4 mol, 2.0 equiv.), and DCE (1.0 mL) were added in sequence in at room temperature under argon atmosphere. The resulting reaction mixture was stirred for 8 h under these conditions. When the reaction was completed (monitored by TLC). Then the solvent was evaporated in vacuo and the residue was purified by flash column chromatography on silica gel without additional treatment (DCM/methanol = 80:1 to 20:1) to afford the pure products **7** in good to high yields.



3-Ethoxy-2-methylquinolin-4(1H)-one (7a). 27.6 mg, 68% yield. Yellow solid; mp: 222-224 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.53 (s, 1H), 8.09 – 8.07 (m, 1H), 7.59 – 7.55 (m, 1H), 7.50 – 7.48 (m, 1H), 7.26 – 7.22 (m, 1H), 4.03 (q, J = 7.0 Hz, 2H), 2.37 (s, 3H), 1.24 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 171.2, 141.9, 138.3, 138.3, 130.8, 125.4, 124.9, 122.0, 117.6, 66.3, 15.5, 14.5. HRMS (TOF MS Cl^+) calculated for $\text{C}_{12}\text{H}_{14}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 204.1019, found 204.1013.

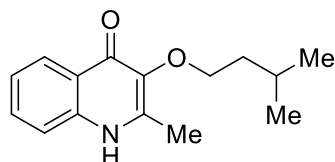


2-Methyl-3-propoxyquinolin-4(1H)-one (7b). 26.9 mg, 62% yield. Yellow solid; mp: 231-233 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.52 (s, 1H), 8.09 – 8.07 (m, 1H), 7.59 – 7.56 (m, 1H), 7.50 – 7.47 (m, 1H), 7.25 – 7.21 (m, 1H), 3.92 (t, J = 6.6 Hz, 2H), 2.37 (s, 3H), 1.70 – 1.61 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 171.2, 141.7, 138.6, 138.2, 130.8, 124.9, 122.0, 117.6, 72.5, 23.0, 14.5, 10.6. HRMS (TOF MS Cl^+) calculated for $\text{C}_{13}\text{H}_{15}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 240.0995, found 240.0994.

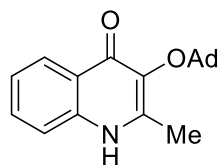


3-Isopropoxy-2-methylquinolin-4(1H)-one (7c). 31.2 mg, 72% yield. Yellow solid; mp: 163-165 °C. ^1H NMR (400 MHz, DMSO- d_6) (δ , ppm) 11.53 (s, 1H), 8.09 – 8.07 (m, 1H), 7.59 – 7.54 (m, 1H), 7.50 – 7.48 (m, 1H), 7.25 – 7.21 (m, 1H), 4.71 (hept, J = 6.2 Hz, 1H), 2.36 (s, 3H), 1.18 (d, J = 6.2 Hz, 6H). ^{13}C NMR (100 MHz, DMSO- d_6)

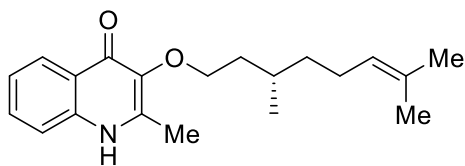
δ 171.4, 142.0, 138.2, 137.0, 130.7, 125.3, 124.9, 122.0, 117.5, 71.3, 22.4, 14.8. HRMS (TOF MS Cl^+) calculated for $\text{C}_{13}\text{H}_{15}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 240.0995, found 240.1000.



3-(Isopentyloxy)-2-methylquinolin-4(1H)-one (7d). 26.5mg, 54% yield. Yellow solid; mp: 213-215 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) (δ , ppm) 11.52 (s, 1H), 8.09 – 8.07 (m, 1H), 7.59 – 7.55 (m, 1H), 7.50 – 7.48 (m, 1H), 7.22 (d, $J = 7.4$ Hz, 1H), 3.99 (t, $J = 6.7$ Hz, 2H), 2.37 (s, 3H), 1.82 – 1.73 (m, 1H), 1.55 (q, $J = 6.8$ Hz, 2H), 0.92 (d, $J = 6.7$ Hz, 6H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 171.2, 141.7, 138.6, 138.2, 130.8, 125.4, 124.8, 122.0, 117.6, 69.2, 38.6, 24.5, 22.6, 14.4. HRMS (TOF MS Cl^+) calculated for $\text{C}_{15}\text{H}_{19}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 268.1308, found 268.1312.

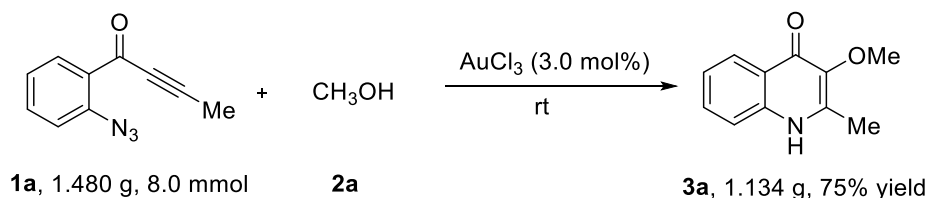


3-(((3R)-Adamantan-1-yl)oxy)-2-methylquinolin-4(1H)-one (7e). 32.1 mg, 52% yield. Yellow solid; mp: 254-256 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) (δ , ppm) 11.43 (s, 1H), 8.11 – 8.08 (m, 1H), 7.56 – 7.53 (m, 1H), 7.48 – 7.46 (m, 1H), 7.22 – 7.19 (m, 1H), 2.38 (s, 3H), 2.06 (s, 3H), 1.91 (d, $J = 3.0$ Hz, 6H), 1.54 (d, $J = 3.2$ Hz, 6H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 173.4, 143.7, 138.2, 135.2, 130.7, 125.26, 125.2, 121.8, 117.4, 79.0, 54.9, 42.6, 35.8, 30.5, 16.1. HRMS (TOF MS Cl^+) calculated for $\text{C}_{20}\text{H}_{23}\text{NNaO}_2^+$ $[\text{M}+\text{Na}]^+$: 332.1621, found 332.1612.



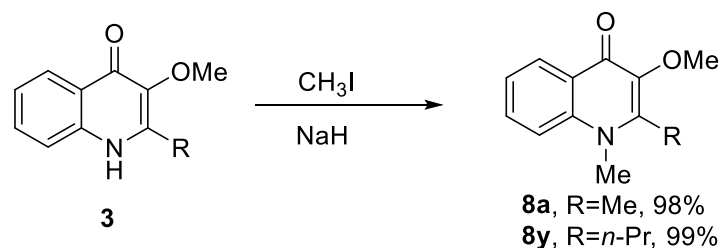
(S)-3-((3,7-Dimethyloct-6-en-1-yl)oxy)-2-methylquinolin-4(1H)-one (7f). 30.0 mg, 48% yield. Yellow solid; mp: 70-72 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) (δ , ppm) 11.53 (s, 1H), 8.09 – 8.07 (m, 1H), 7.59– 7.55 (m, 1H), 7.50 – 7.47 (m, 1H), 7.25– 7.21 (m, 1H), 5.13 – 5.04 (m, 1H), 4.02– 3.98 (m, 2H), 2.36 (s, 3H), 2.02– 2.19 (m, 2H), 1.74 – 1.64 (m, 2H), 1.63 (d, J = 1.4 Hz, 3H), 1.56 (d, J = 1.4 Hz, 3H), 1.50 – 1.28 (m, 3H), 1.24 – 1.11 (m, 2H), 0.91 (d, J = 6.5 Hz, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 171.2, 141.7, 138.6, 138.2, 130.8, 130.5, 125.4, 124.9, 124.7, 122.0, 117.6, 69.0, 36.8, 36.7, 28.8, 25.5, 25.0, 19.4, 17.5, 14.4. HRMS (TOF MS Cl^+) calculated for $\text{C}_{20}\text{H}_{28}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 314.2115, found 314.2119.

General Procedure for the Scale Up.

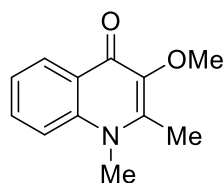


To a 50-mL oven-dried round-bottom flask with a magnetic stirring bar, **1a** (1.480 g, 8.0 mmol), AuCl_3 (72.8 mg, 3.0 mol %), and methanol **2a** (20.0 mL) were added in sequence under atmosphere of argon at room temperature, and the reaction mixture was stirred for 8 h under these conditions. When the reaction was completed (monitored by TLC), Then the solvent was evaporated in vacuo and the residue was purified by flash column chromatography on silica gel without additional treatment ($\text{DCM}/\text{methanol}$ = 80:1 to 20:1) to afford the 1.134 g pure product **3a** in 75% yield.

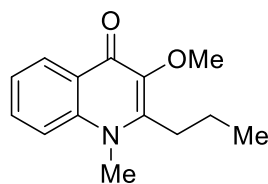
Derivatizations:



Synthesis of 8: A solution of **3** (0.20 mmol) in THF (1.0 ml) was added to a stirred suspension solution of NaH (16.0 mg, 0.40 mmol, 2.0 equiv.) in THF (1.0 ml) at 0° for 5 min, and CH₃I (142 mg, 2.0 mmol, 10.0 equiv.) was added to the mixture. The mixture was stirred overnight at ambient temperature and quenched with saturated NH₄Cl solution. The mixture was adjusted to pH 10 with 10% NaOH and extracted with CHCl₃. The extract was washed with 10% NaOH followed by drying Na₂SO₄. Then the solvent was evaporated in vacuo and the residue was purified by flash column chromatography on silica gel without additional treatment (DCM/ methanol = 80:1 to 20:1) to afford the pure product **8a** in 98% yield and the pure product **8y** (**leiokinine A**) in 99% yield.



3-Methoxy-1,2-dimethylquinolin-4(1H)-one (8a). 39.8 mg, 98% yield. Yellow solid; mp: 246-248 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.22 – 8.20 (m, 1H), 7.74 (d, *J* = 8.7 Hz, 1H), 7.69 – 7.64 (m, 1H), 7.34 – 7.30 (m, 1H), 3.74 (s, 3H), 3.71 (s, 3H), 2.50 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 170.2, 145.3, 140.2, 139.9, 131.5, 126.1, 125.4, 122.4, 116.5, 59.1, 35.0, 13.9. HRMS (TOF MS ESI⁺) calculated for C₁₂H₁₃NNaO₂⁺ [M+Na]⁺: 226.0838, found 226.0837.

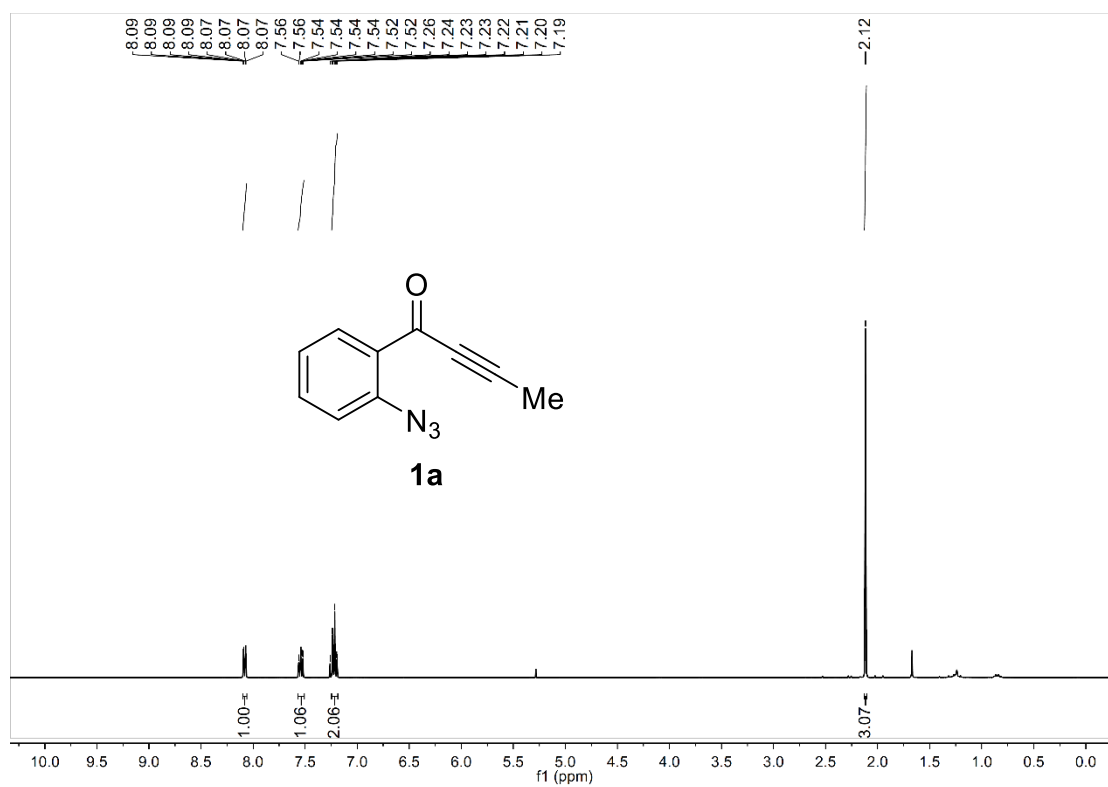


3-Methoxy-1-methyl-2-propylquinolin-4(1H)-one (8y).³ 45.7 mg, 99% yield. Yellow solid; mp: 63-65 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.24 – 8.21 (m, 1H), 7.76 – 7.74 (m, 1H), 7.74 – 7.68 (m, 1H), 7.36 – 7.32 (m, 1H), 3.38 – 7.37 (m, 6H), 2.95 – 2.86 (m, 2H), 1.64 – 1.55 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 170.3, 148.1, 140.2, 140.0, 131.4, 126.2, 125.3, 122.1, 116.7, 59.2, 34.9, 28.63, 21.7, 13.9. HRMS (TOF MS ESI⁺) calculated for C₁₄H₁₇NNaO₂⁺ [M+Na]⁺: 254.1151, found 254.1162.

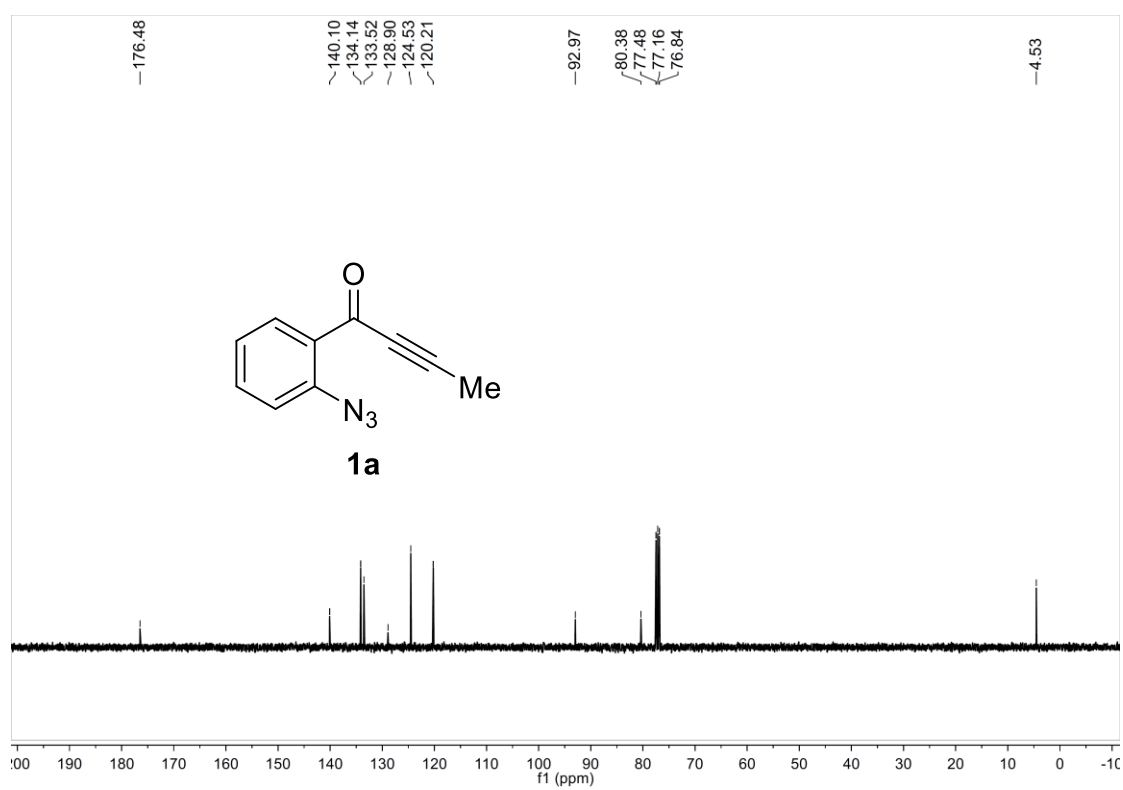
References:

- 1 (a) H. Su, M. Bao, J.-J. Huang, L.-H. Qiu and X.-F. Xu, *Adv. Synth. Catal.*, 2019, **361**, 826; (b) H. Su, M. Bao, C. Pei, W.-H. Hu, L.-H. Qiu and X.-F. Xu, *Org. Chem. Front.*, 2019, **6**, 2404.
- 2 A. A. A. Aguilar, S. J. B. Avila, T. S. Kaufman and E. L. Larghi, *Org. Lett.*, 2018, **20**, 5058.
- 3 M. Sakai, K. Chatani, K. Saito, Y. Yamagiwa and T. Kamikawa, *J. Nat. Prod.*, 1990, **53**, 1508.

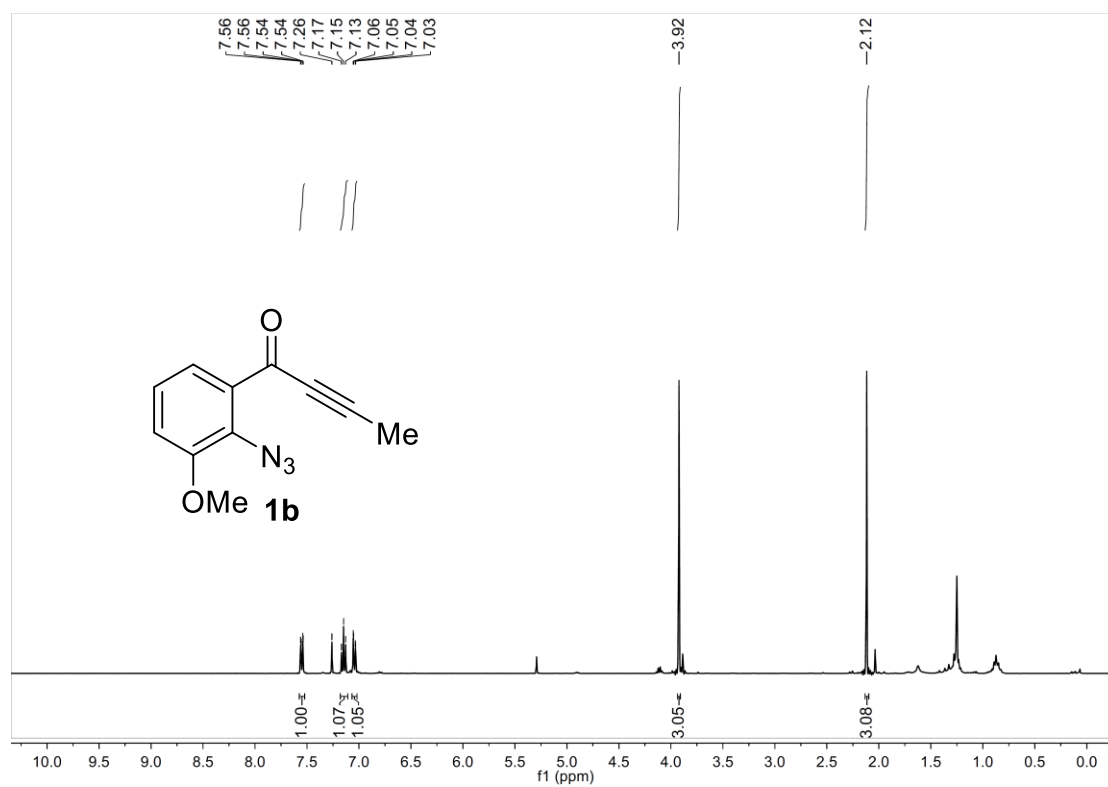
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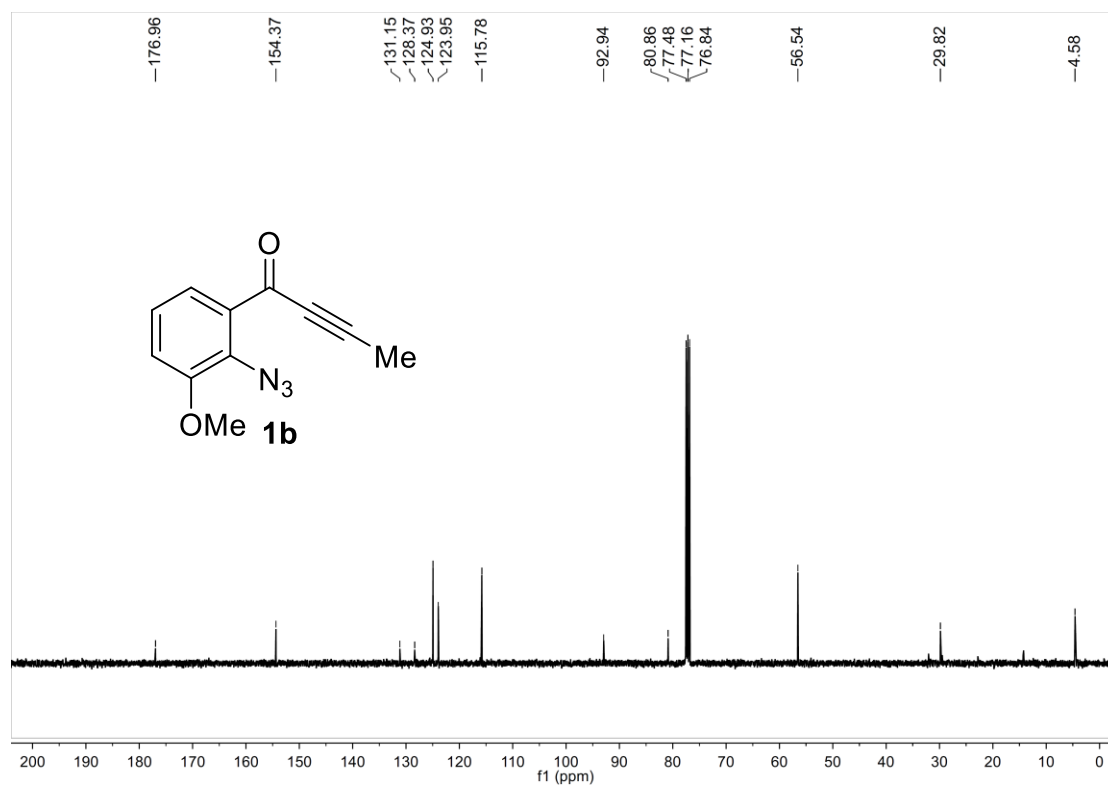
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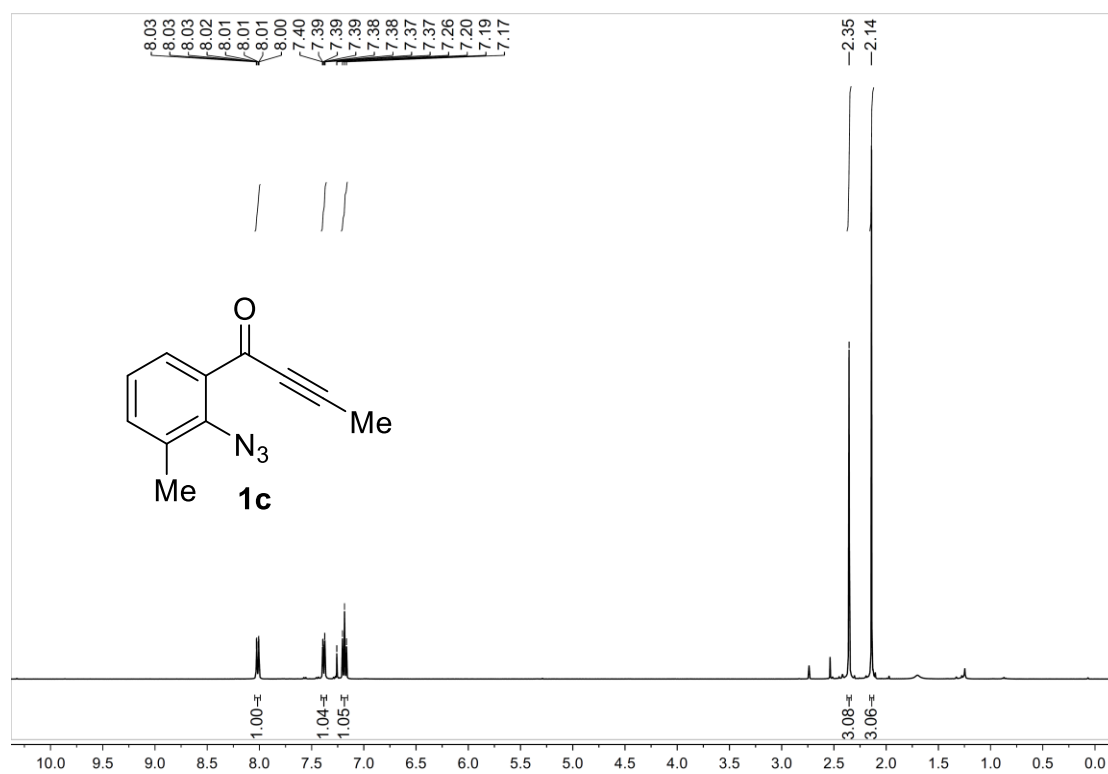
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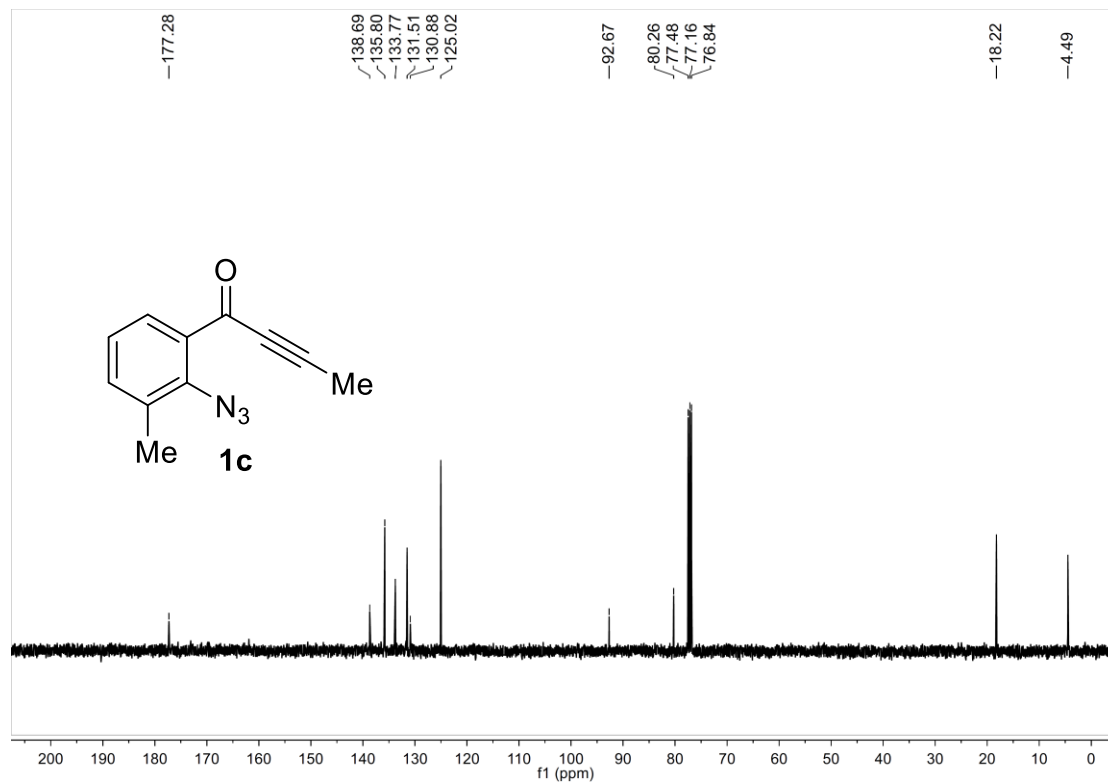
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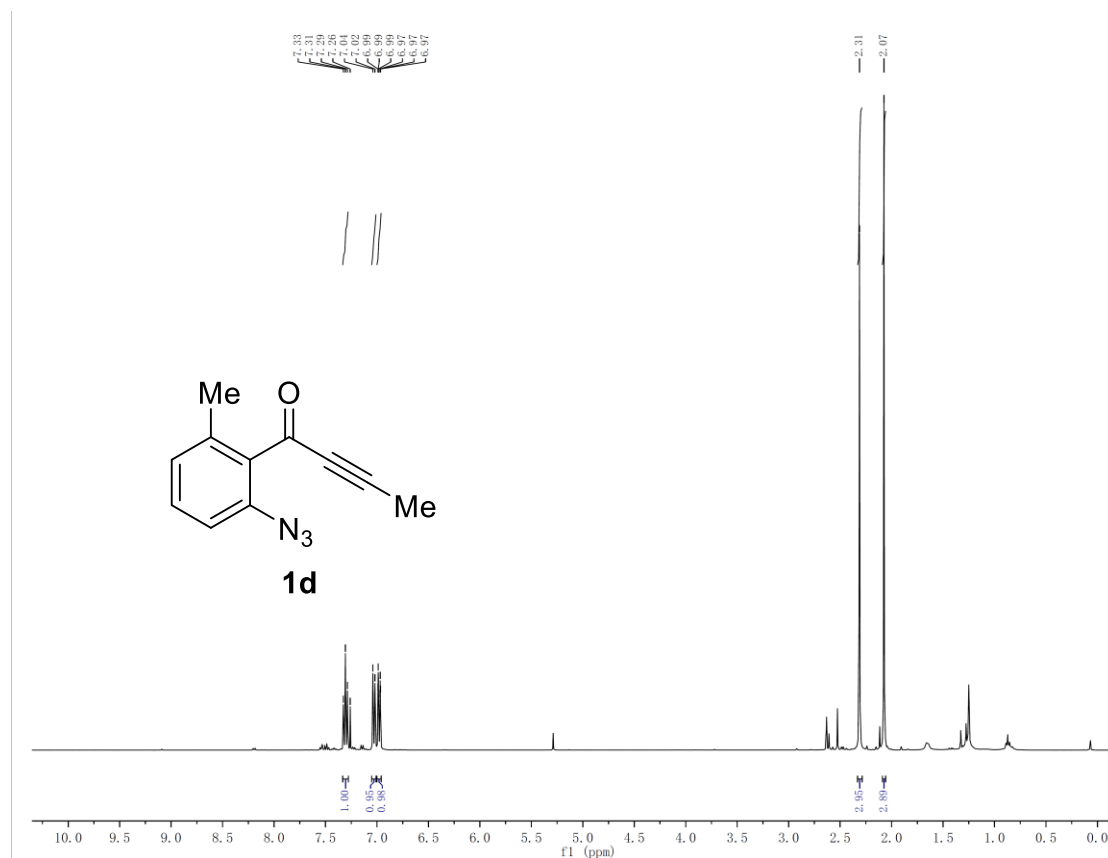
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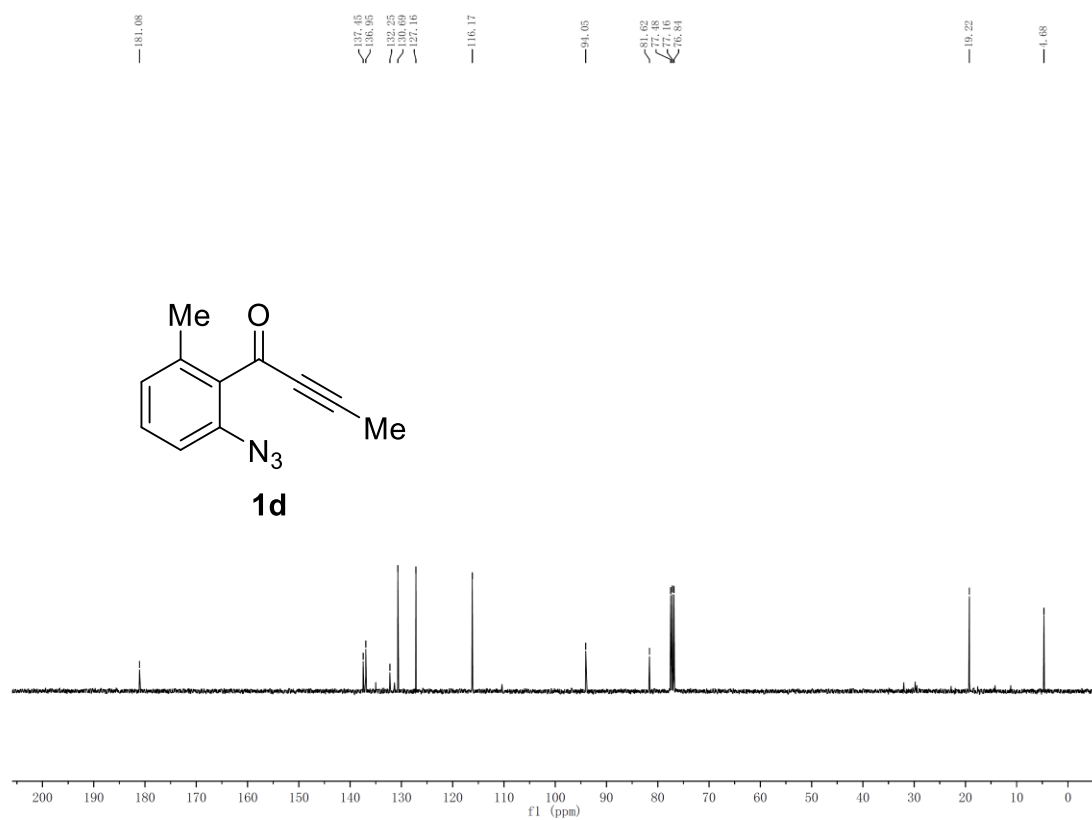
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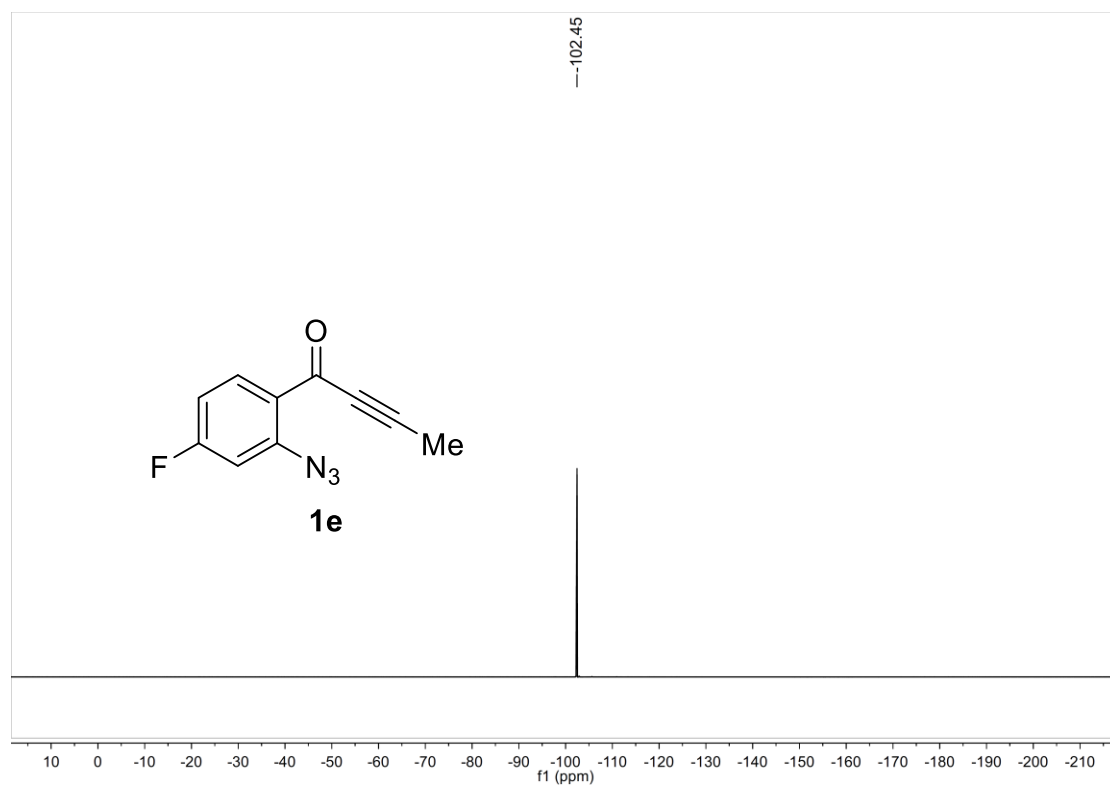
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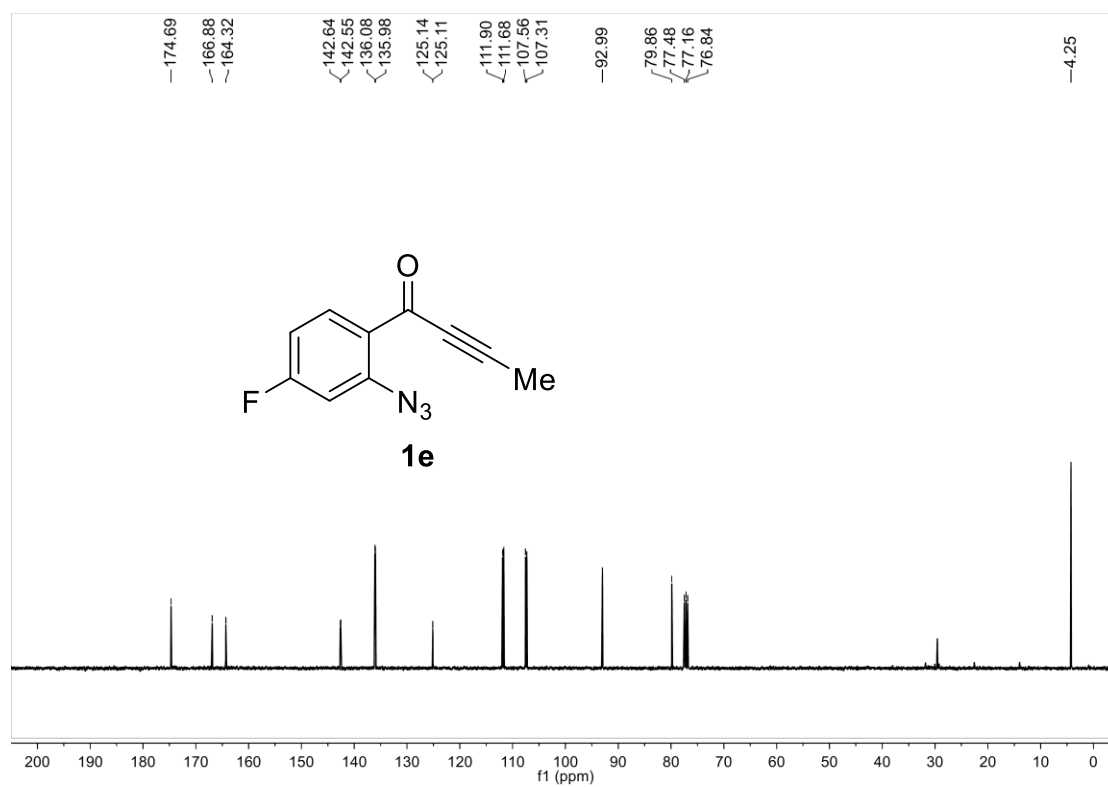
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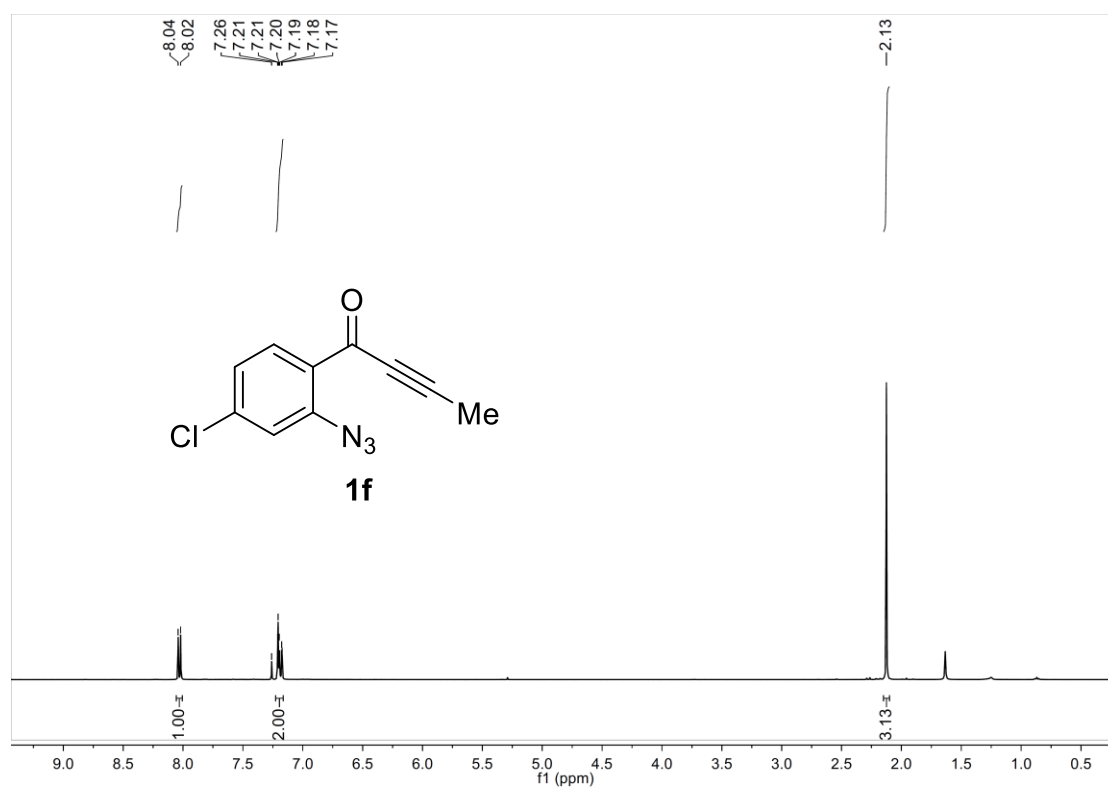
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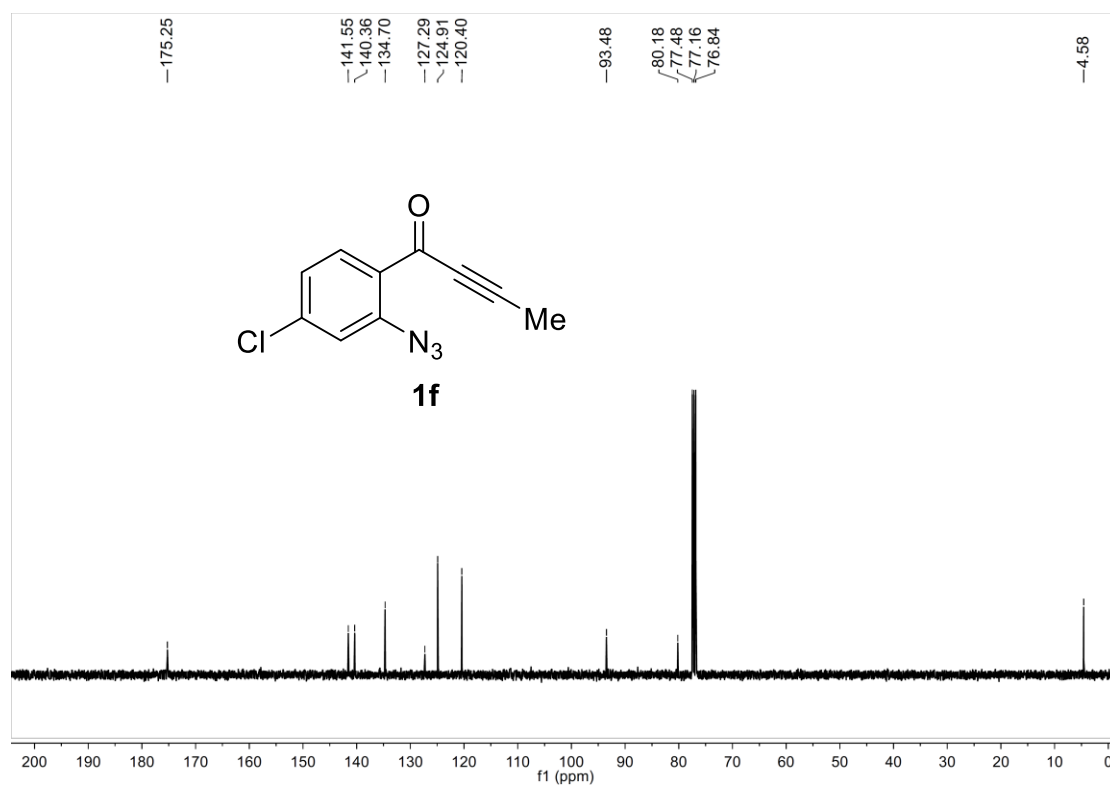
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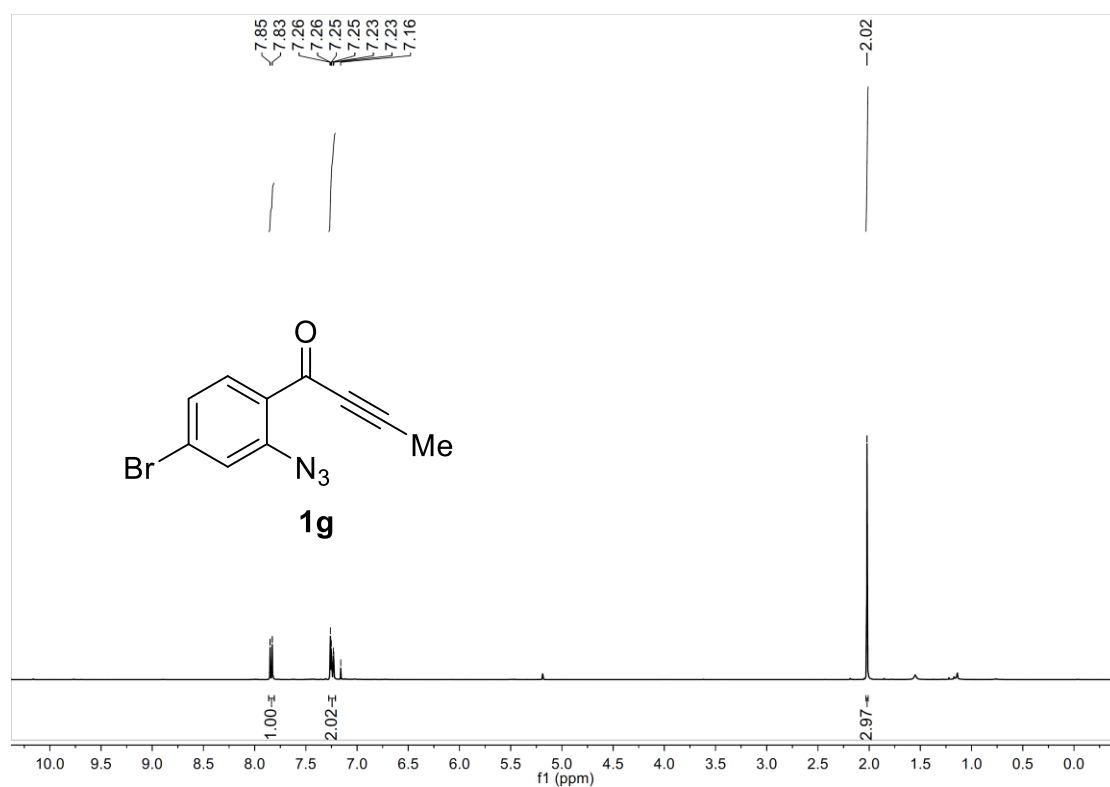
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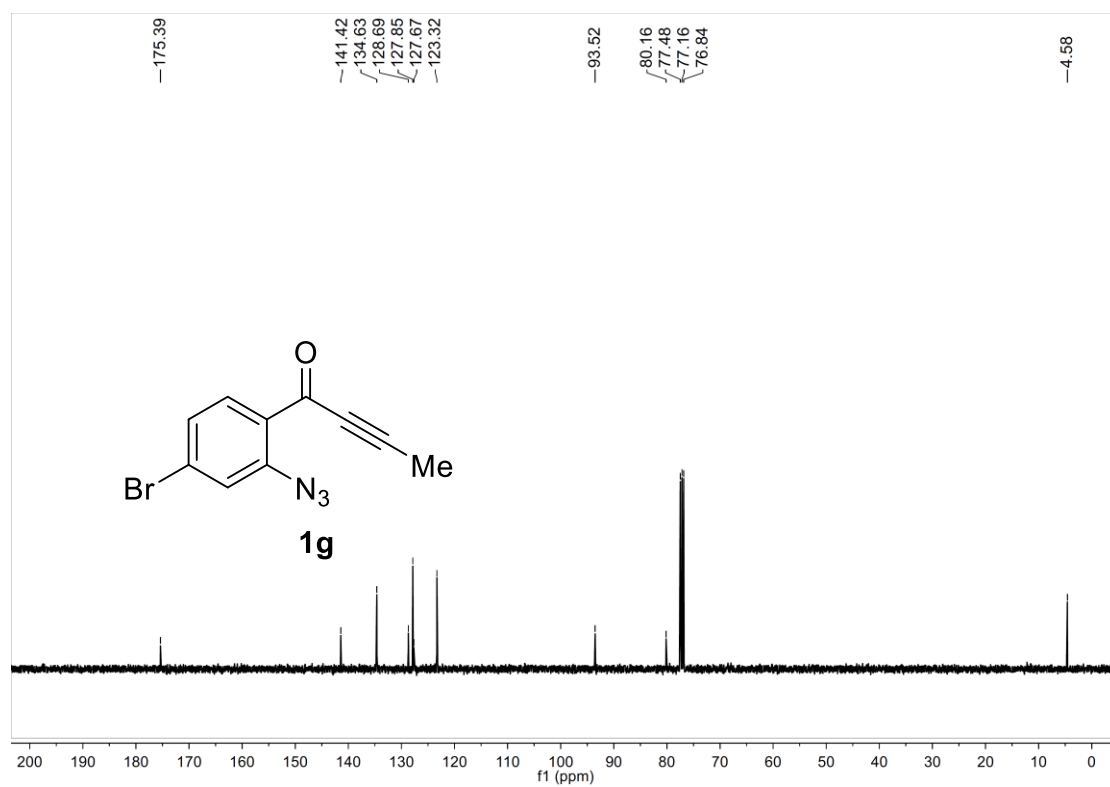
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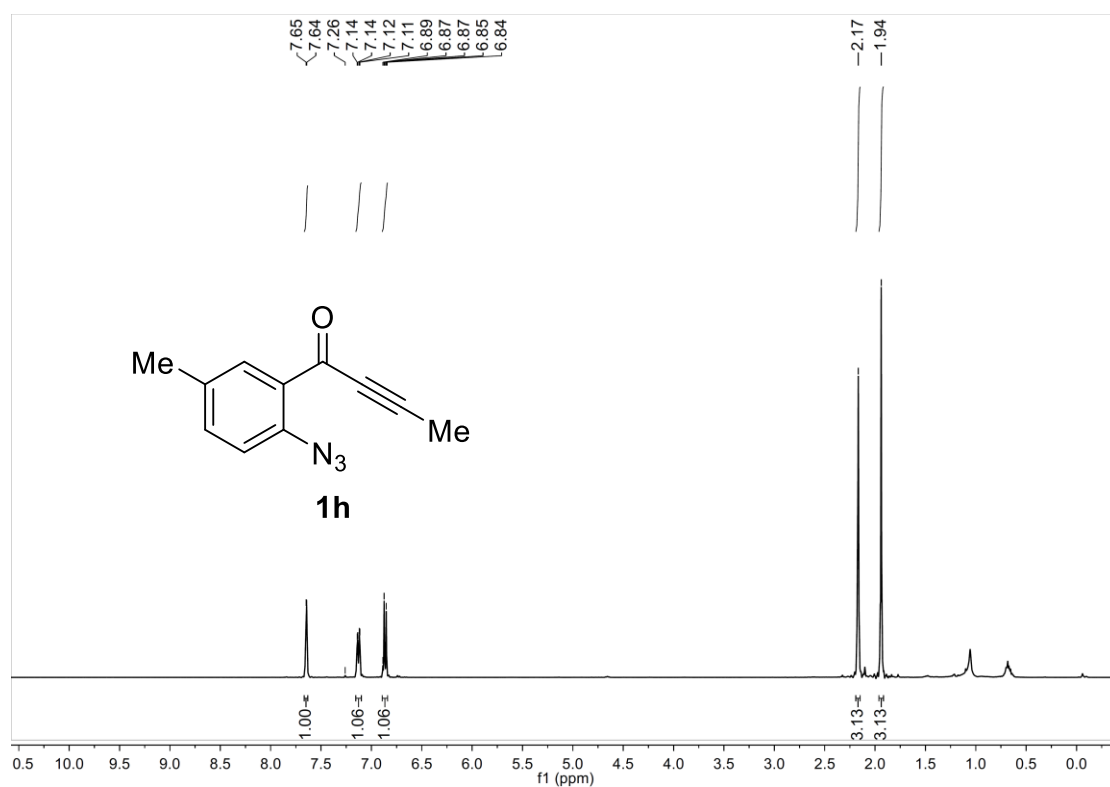
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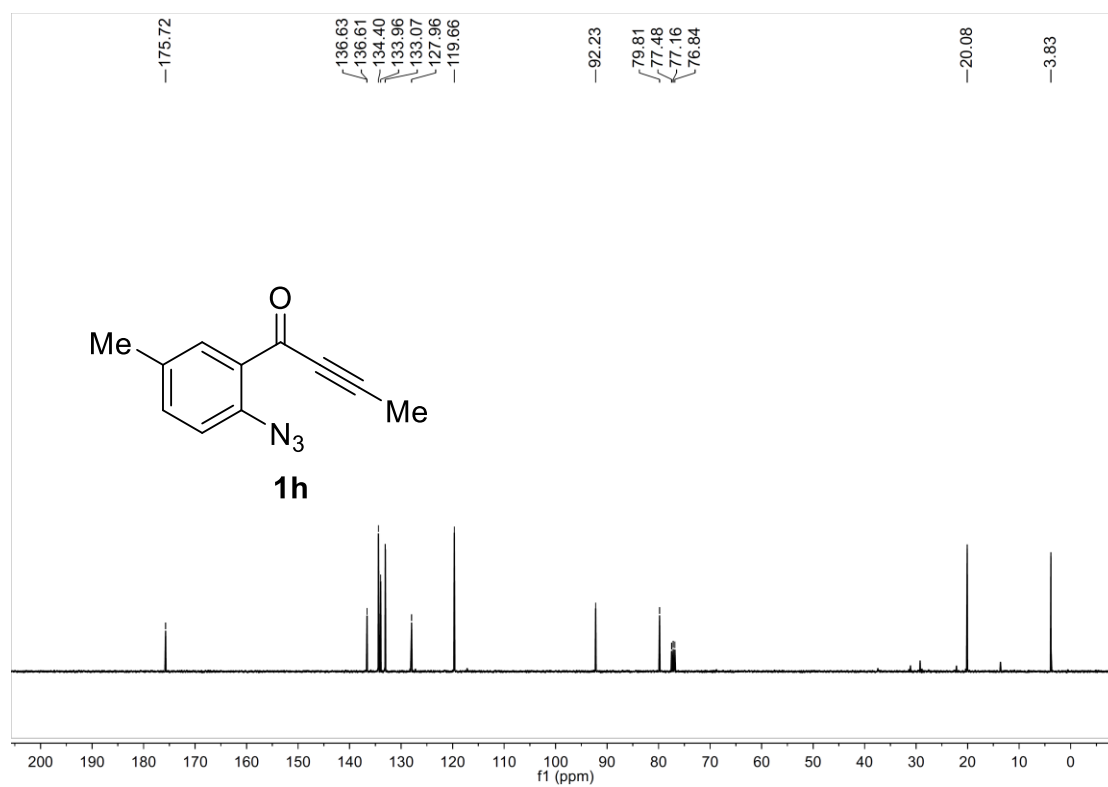
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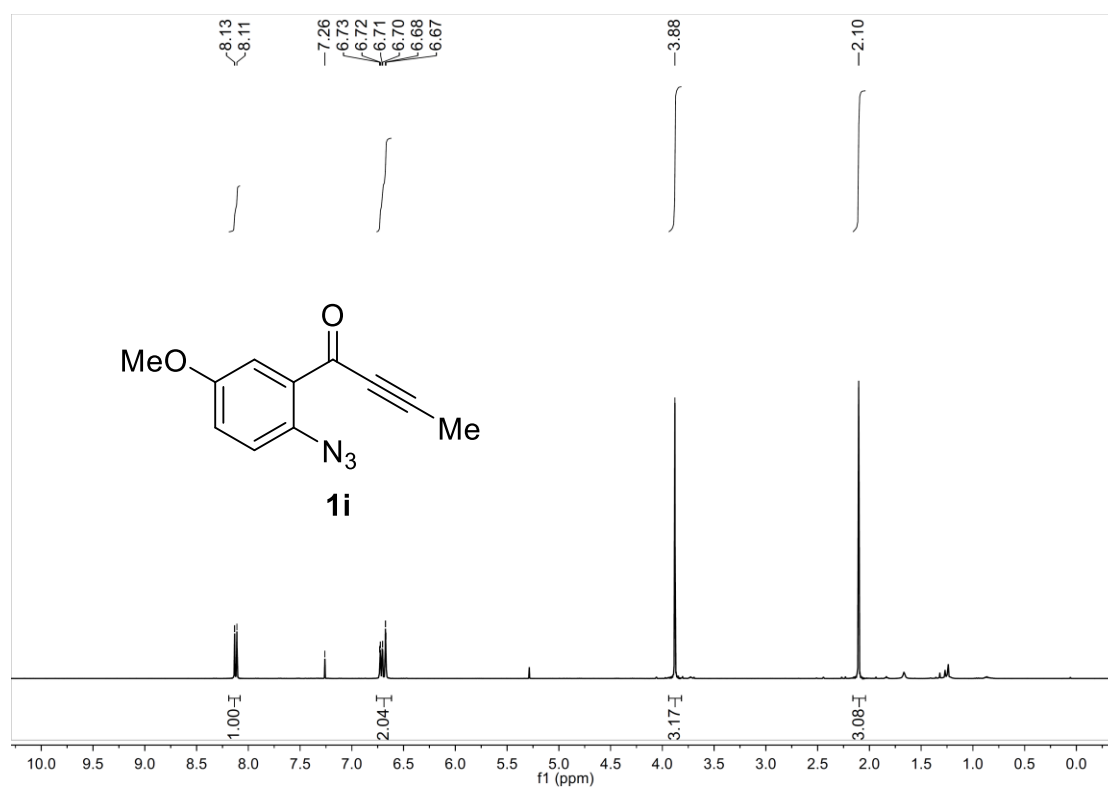
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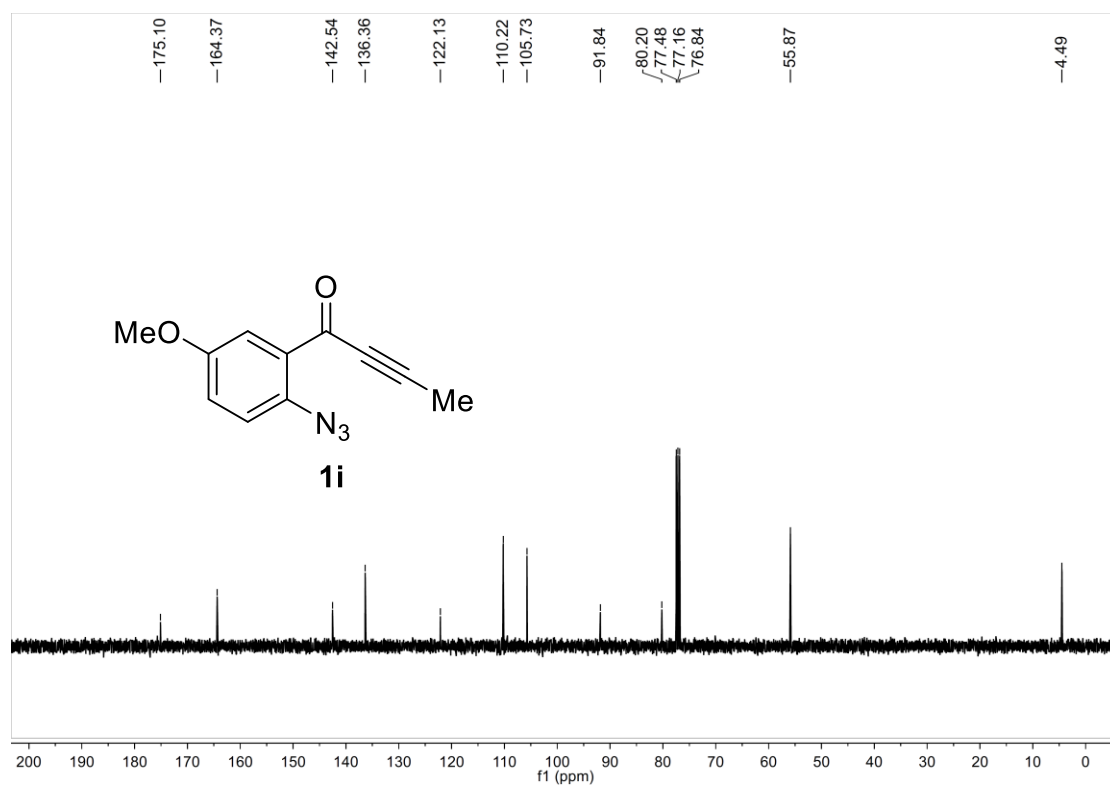
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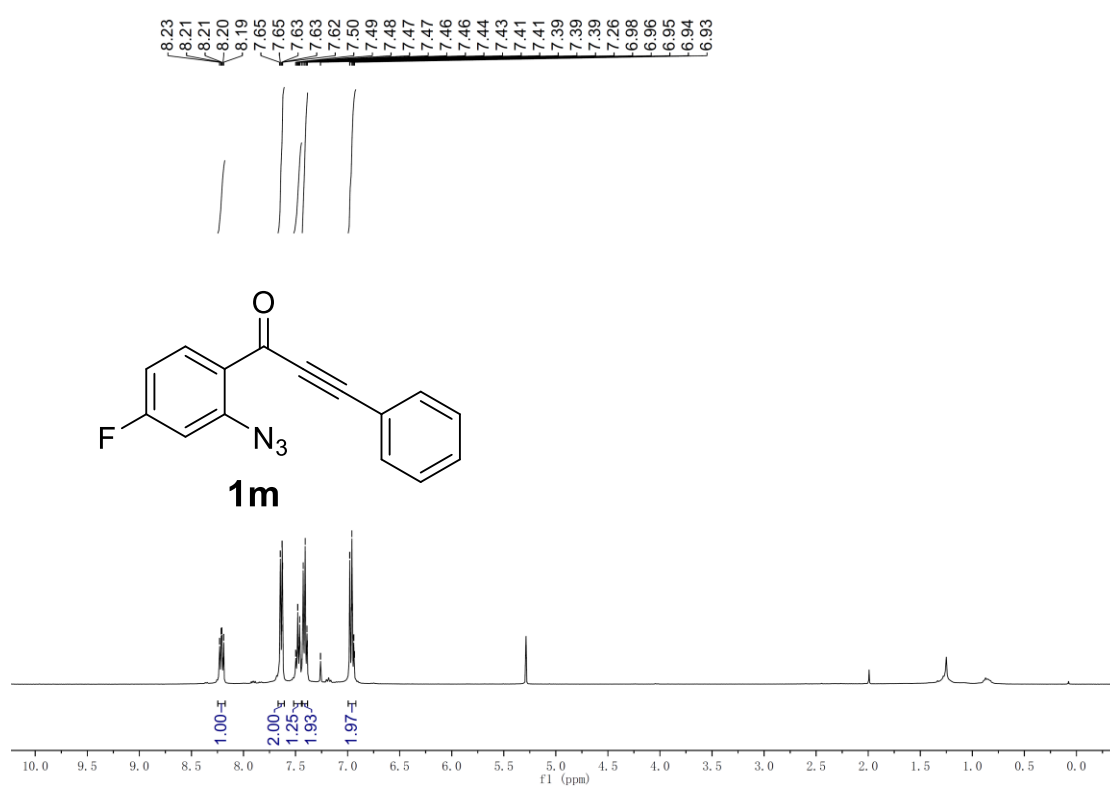
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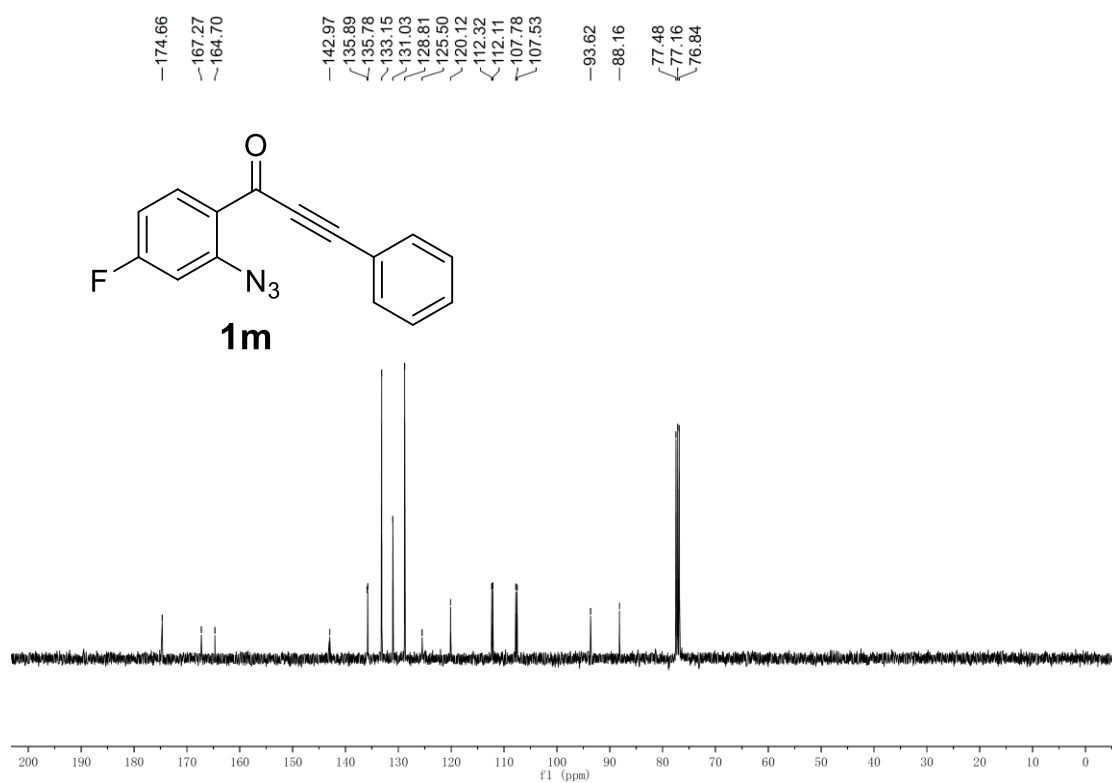
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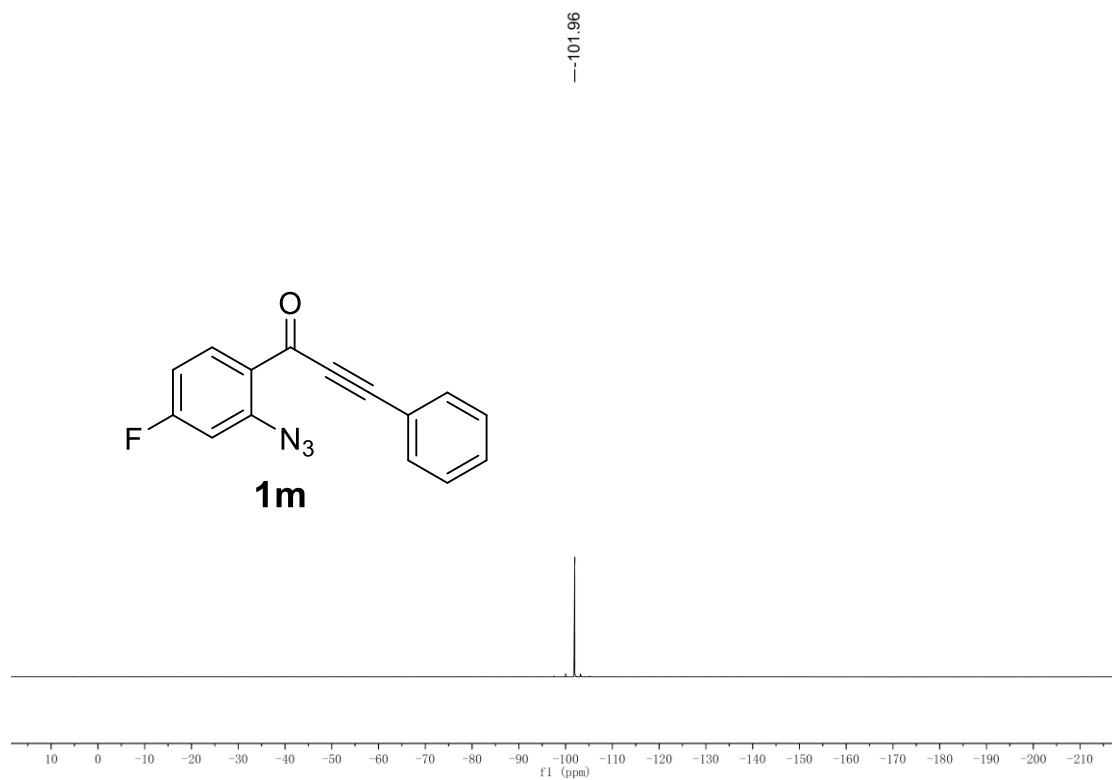
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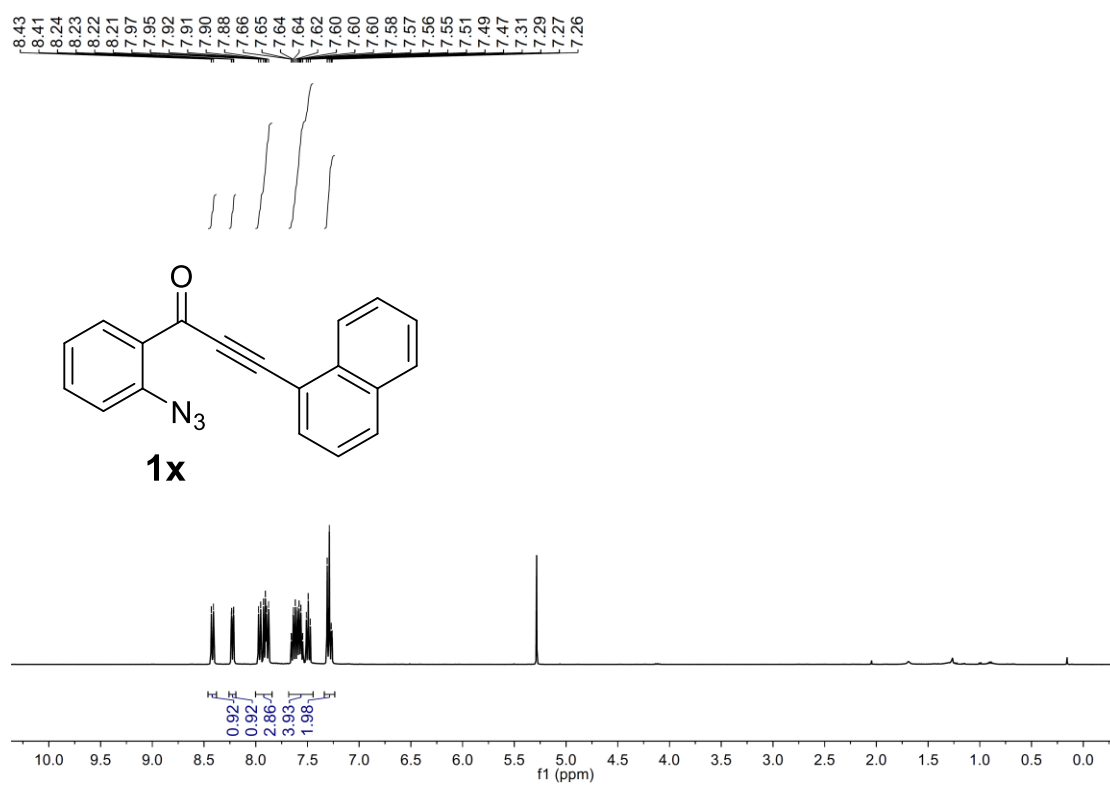
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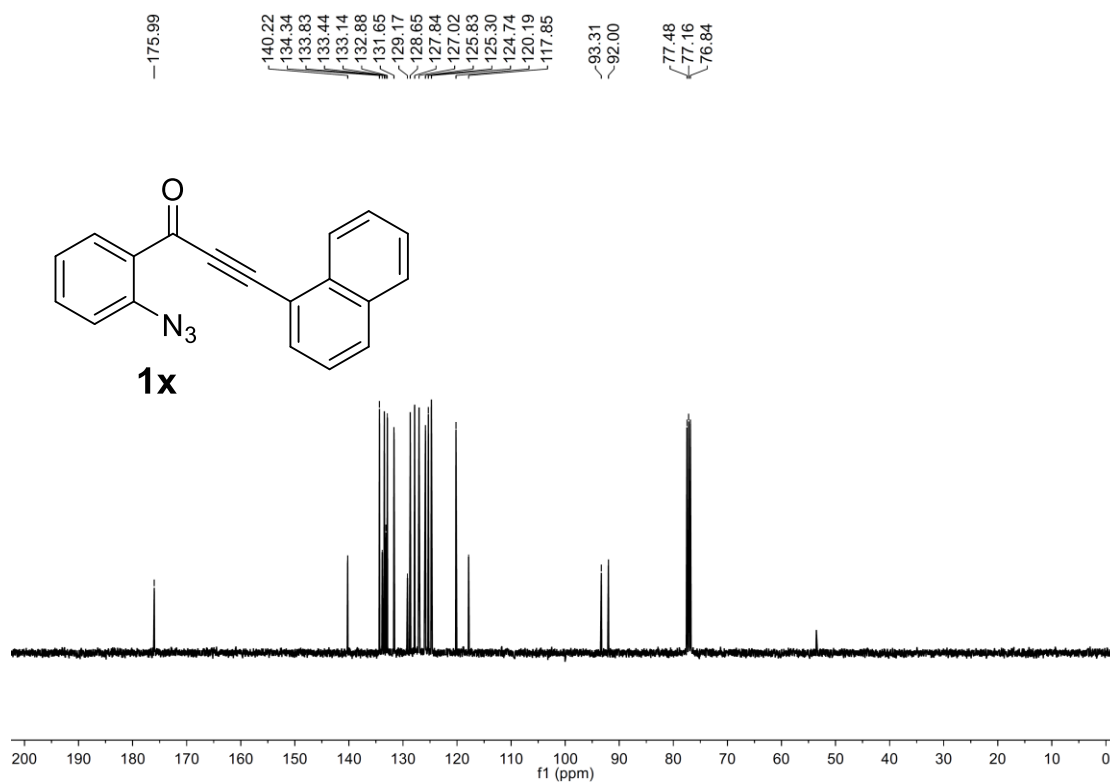
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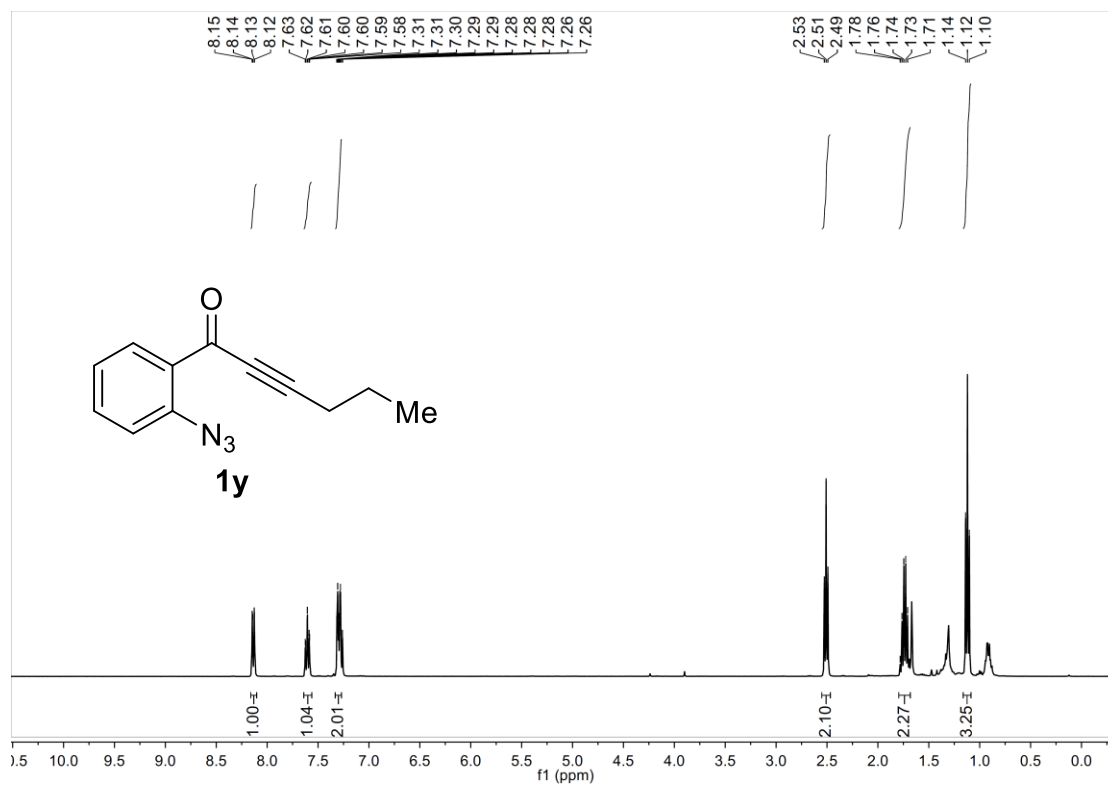
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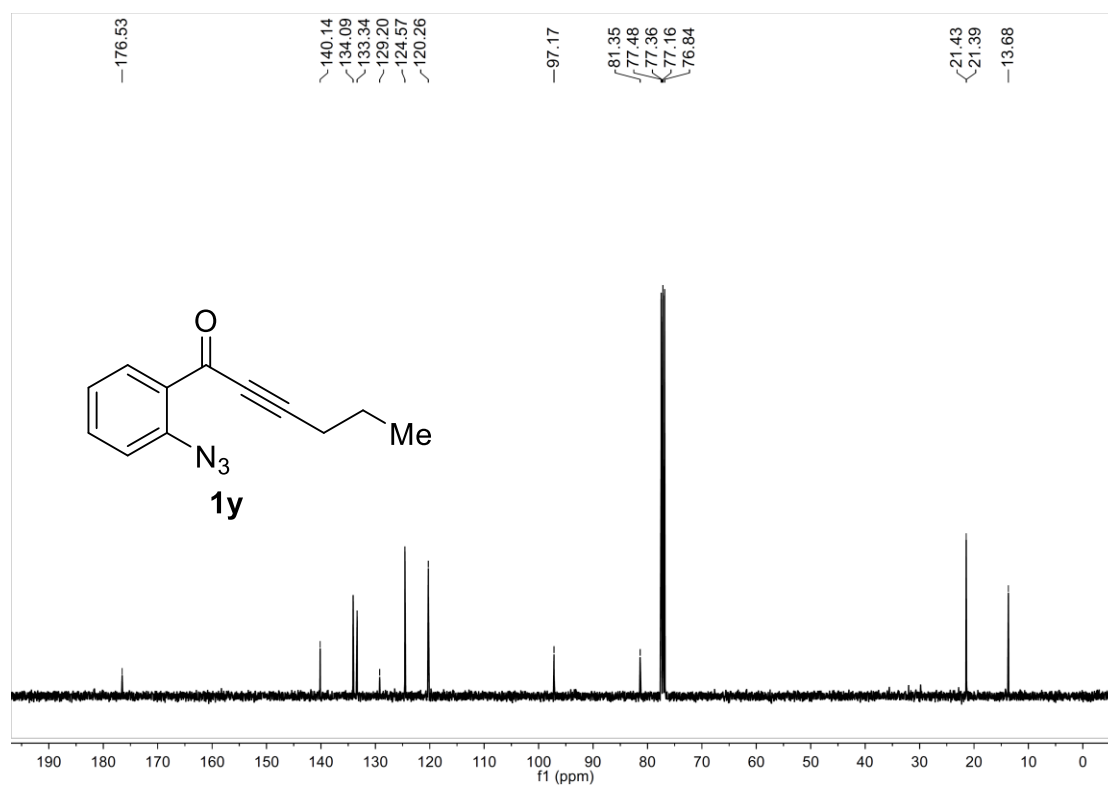
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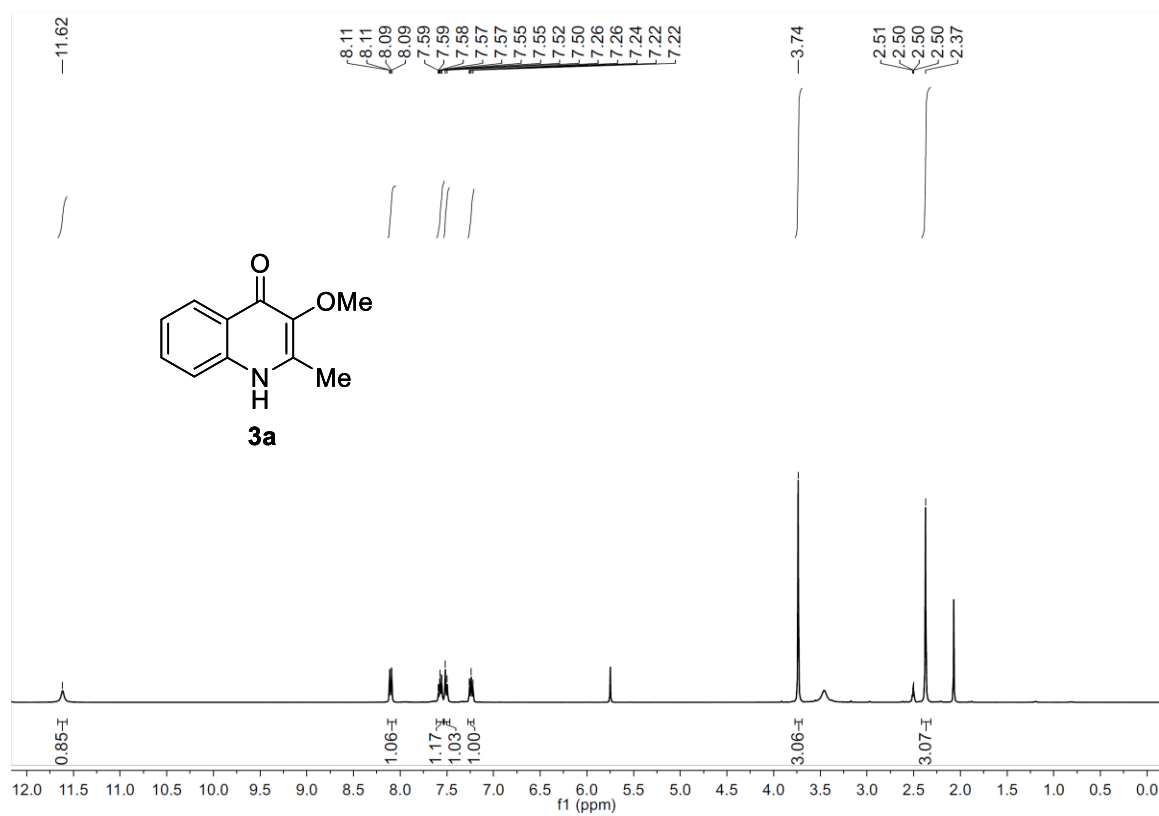
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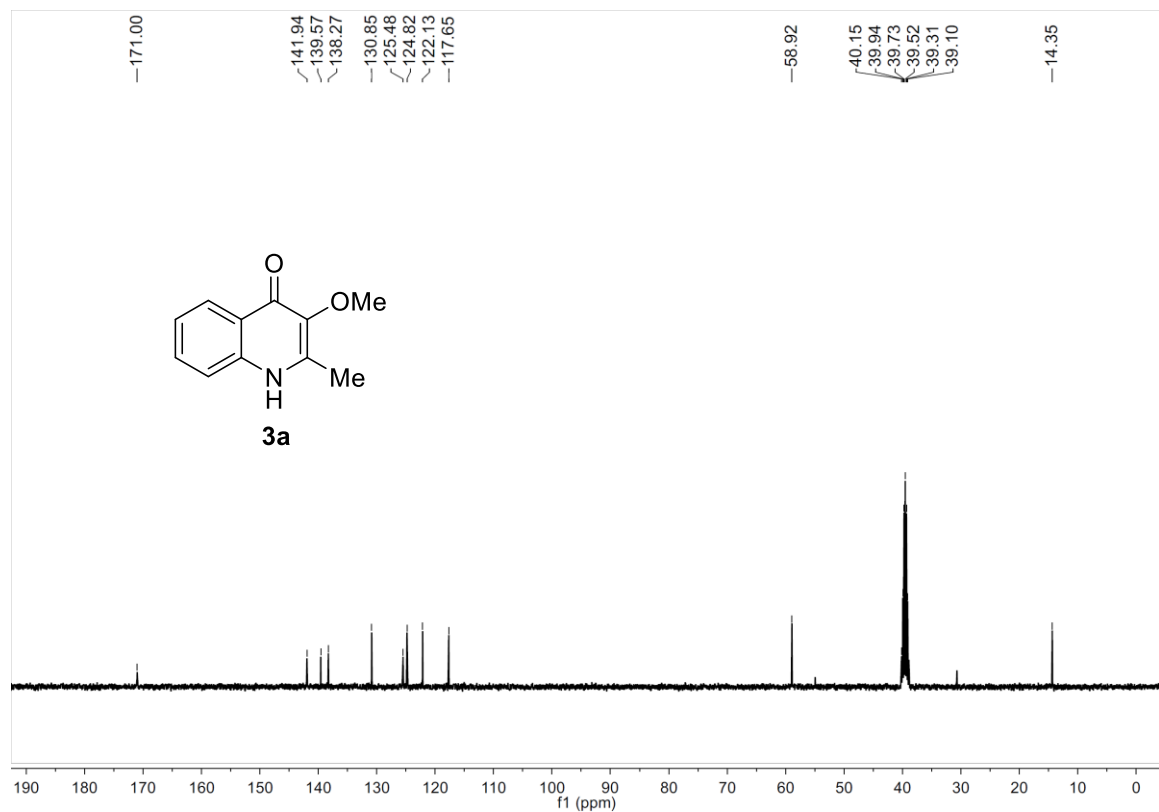
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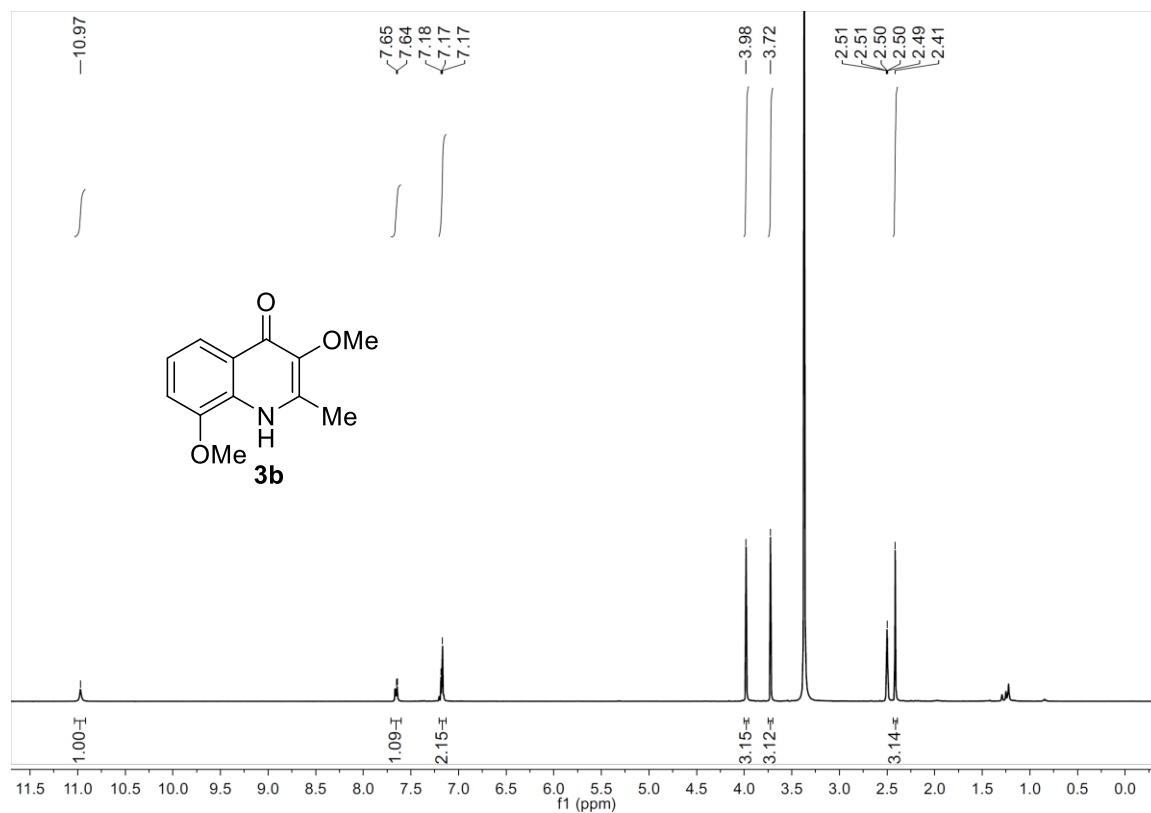
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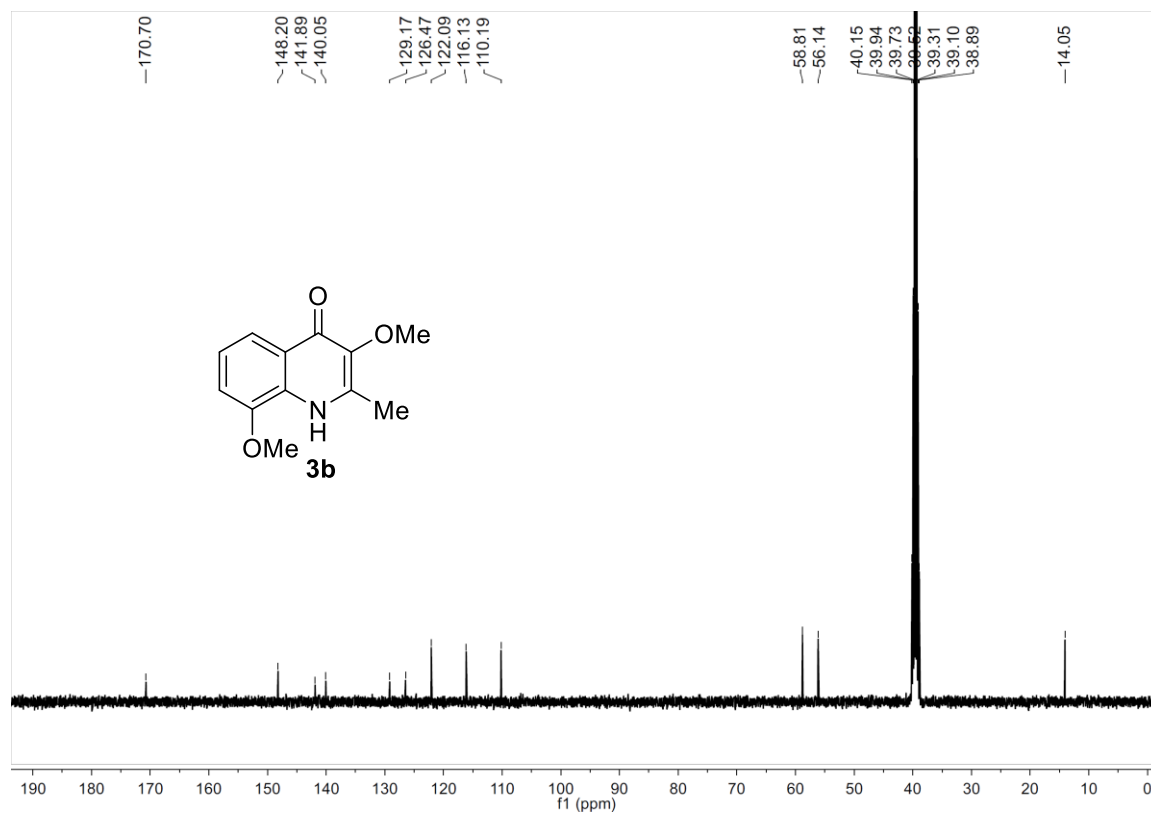
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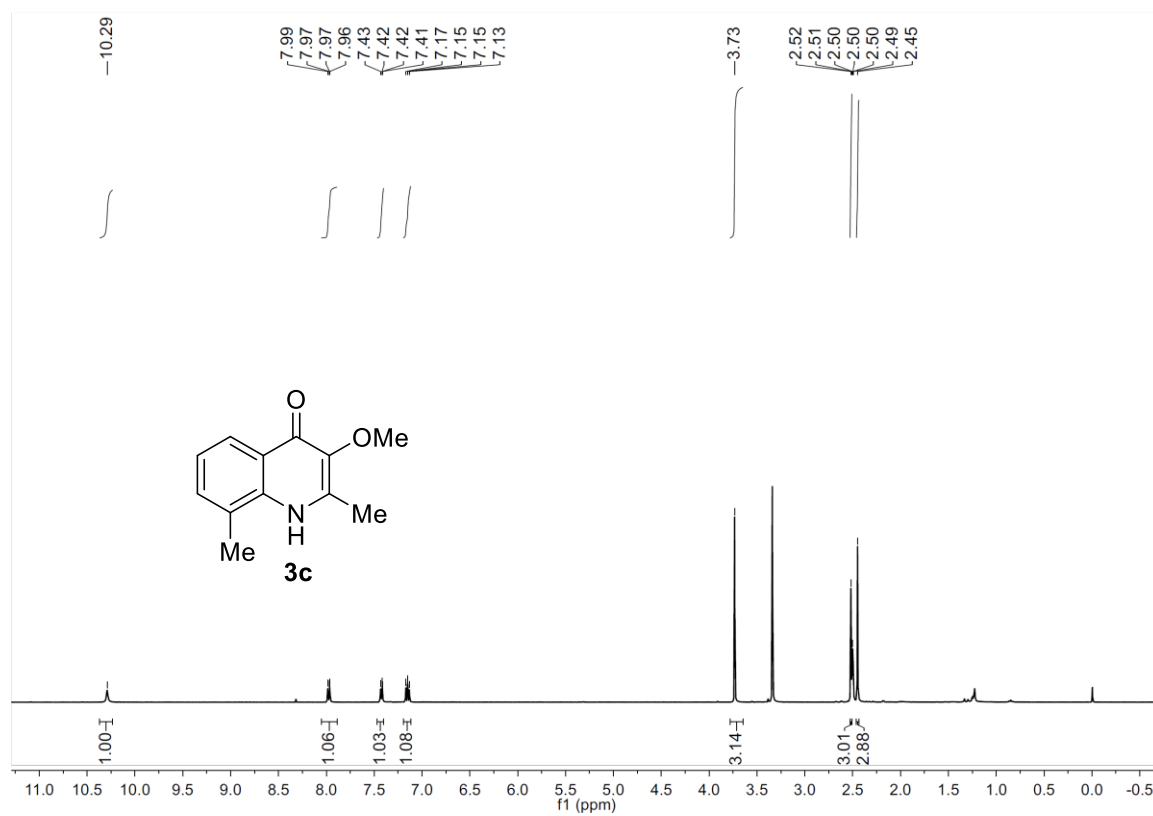
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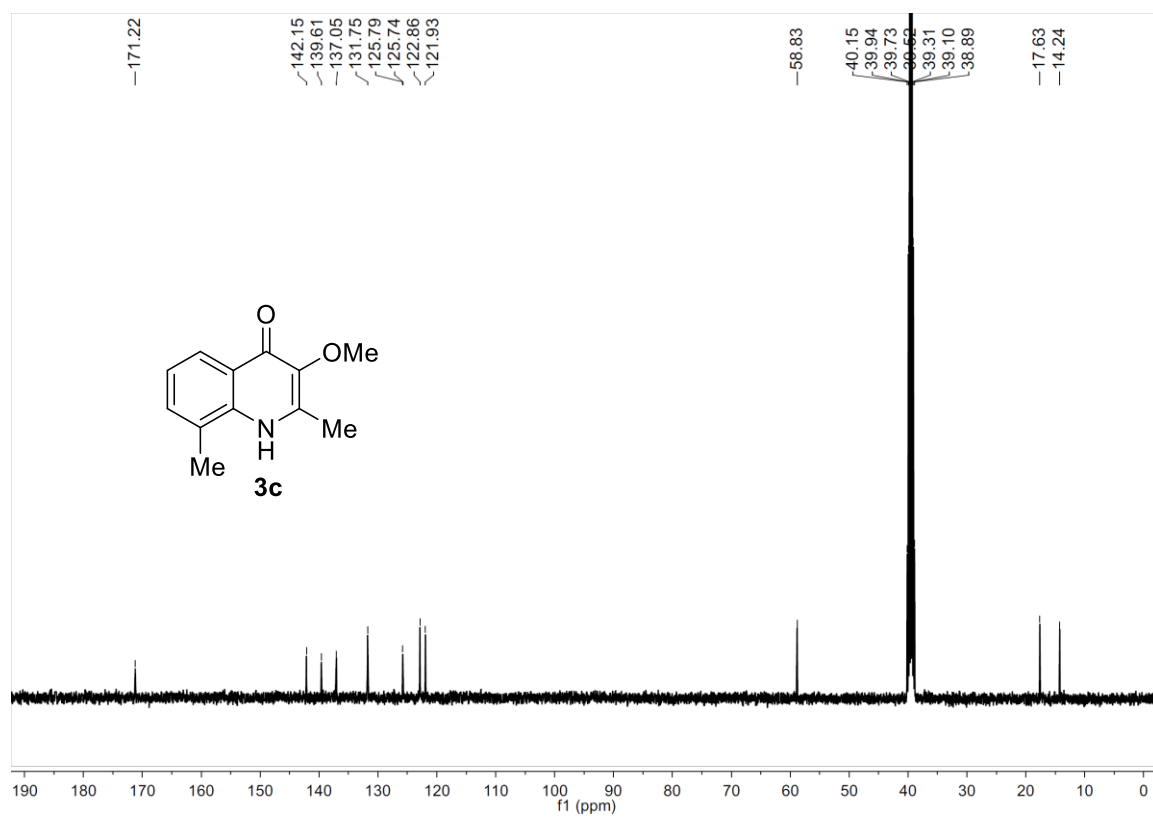
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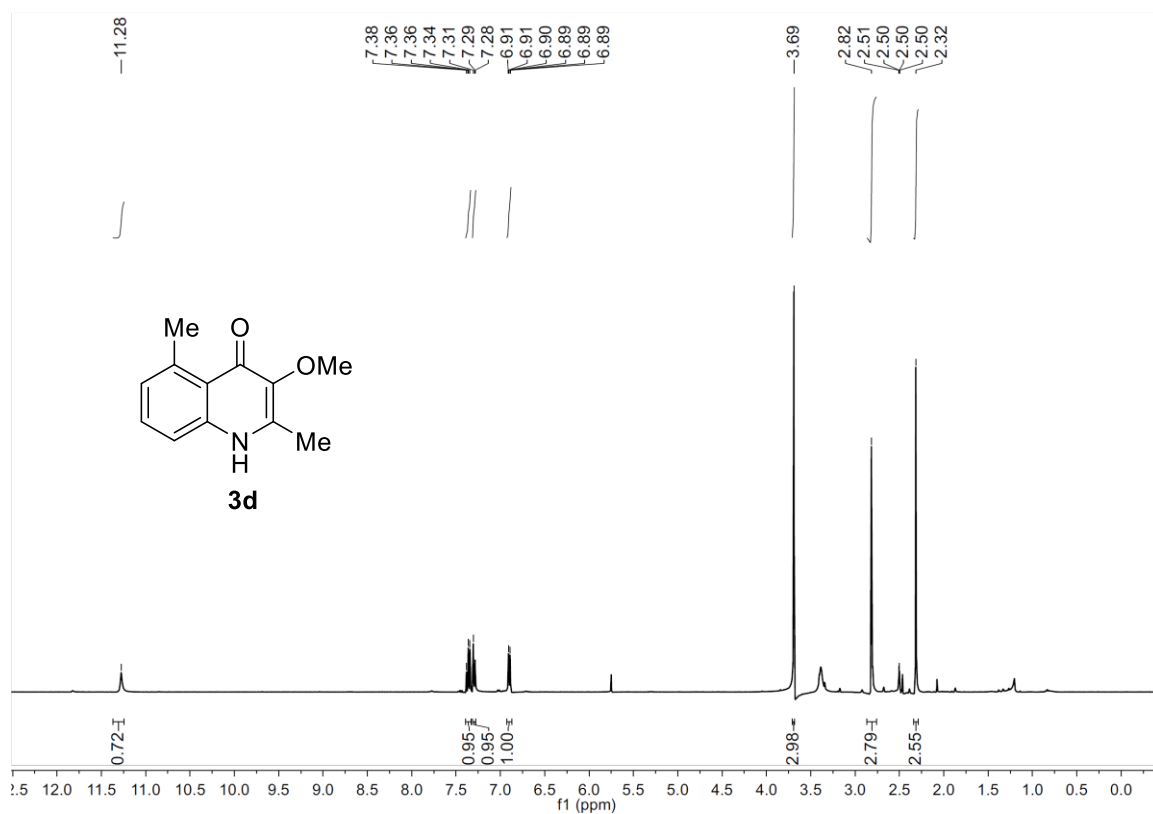
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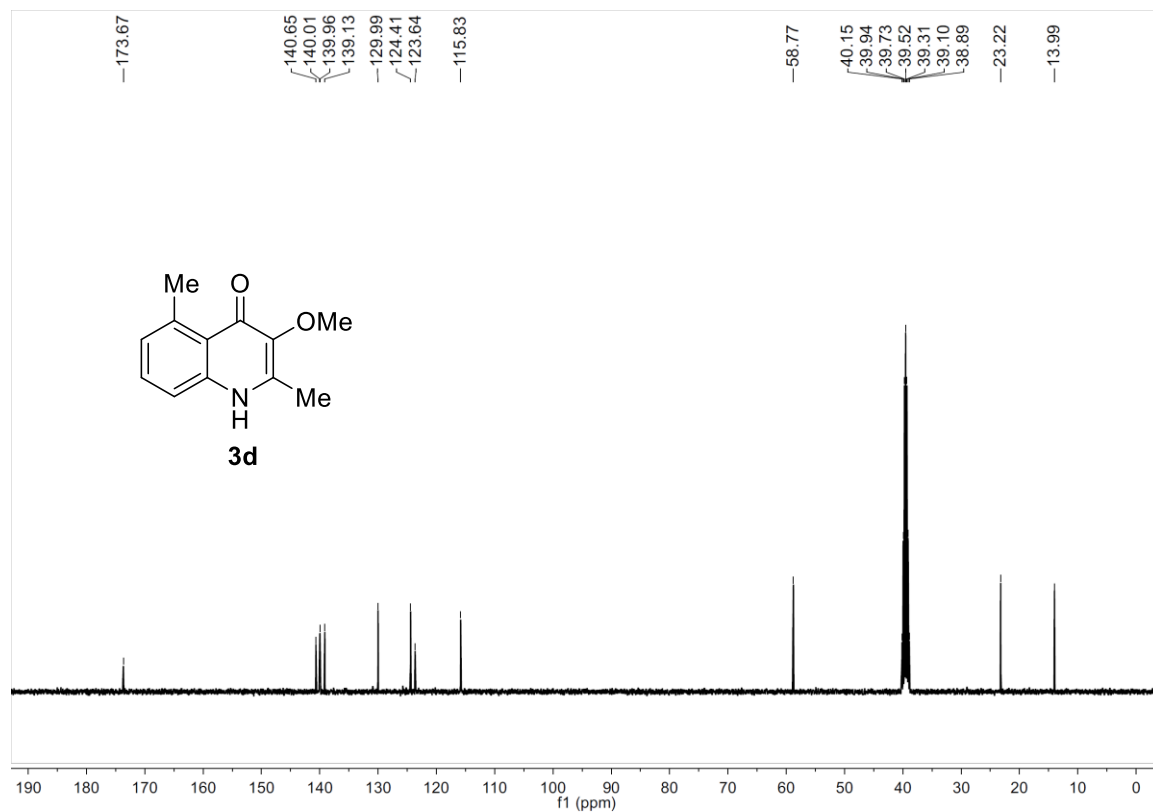
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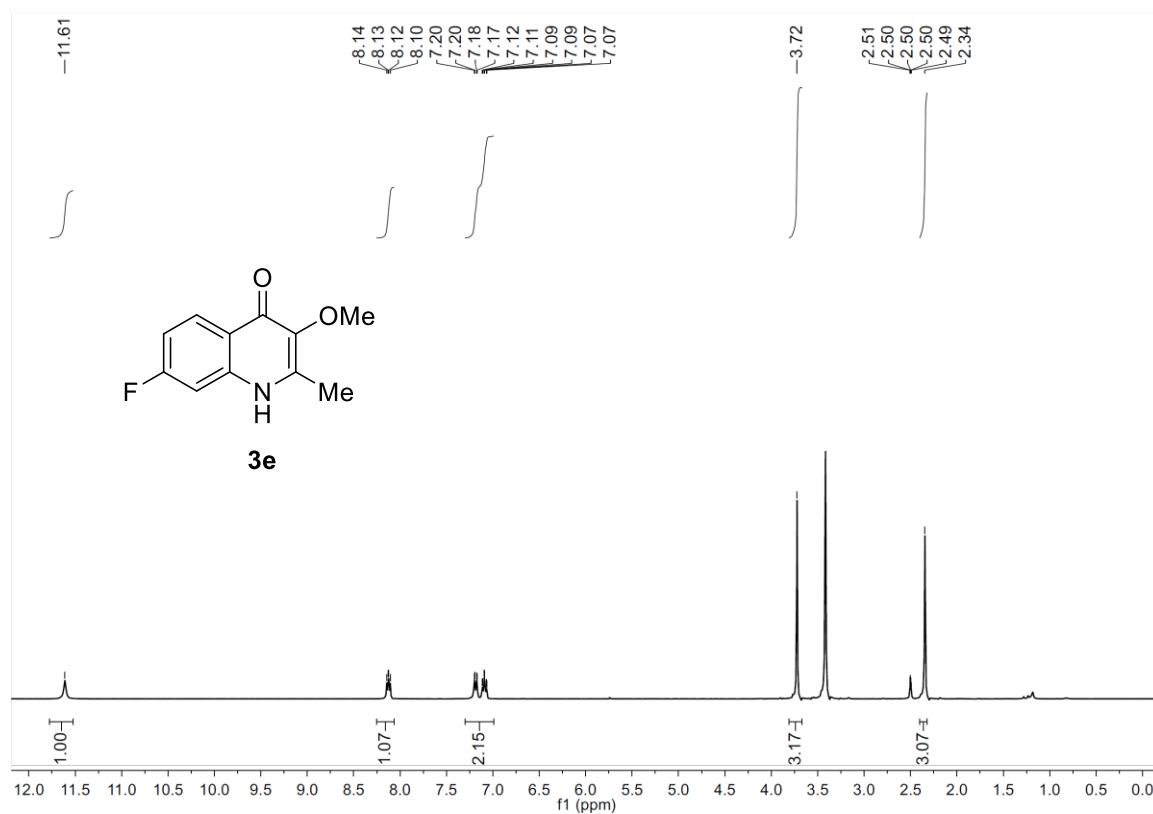


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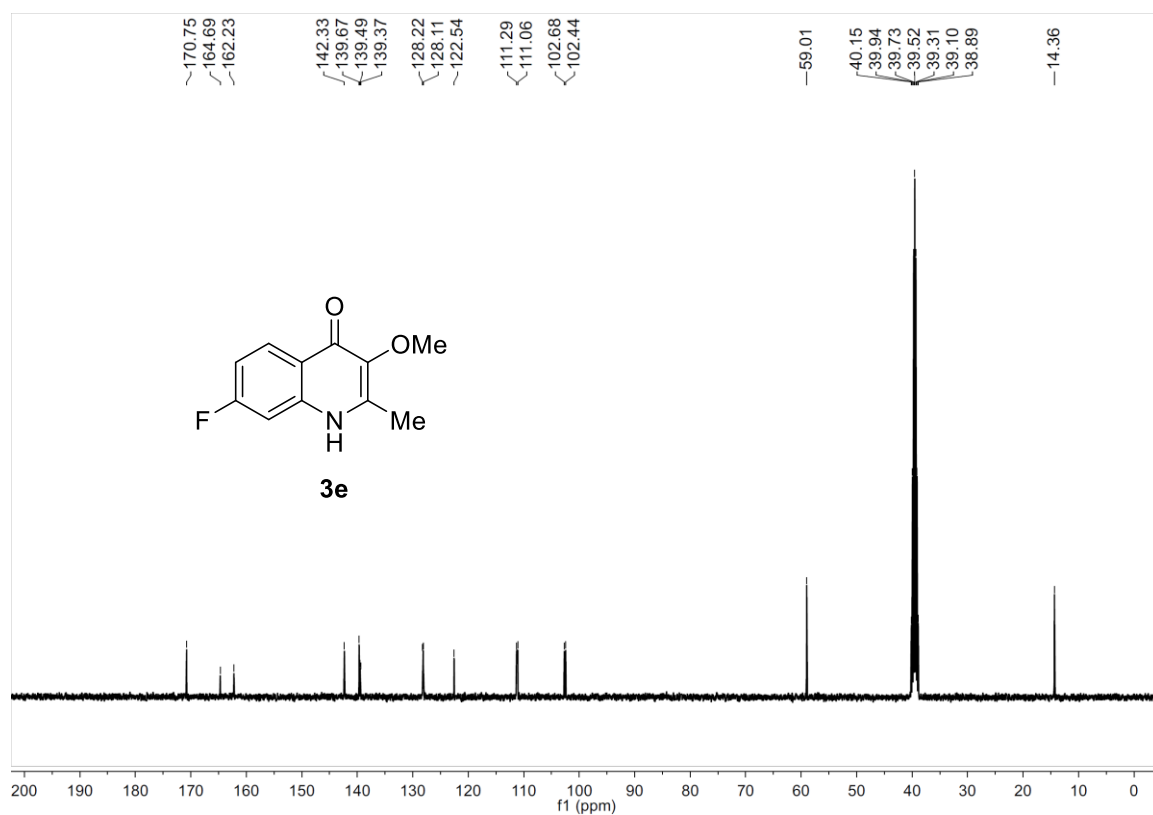


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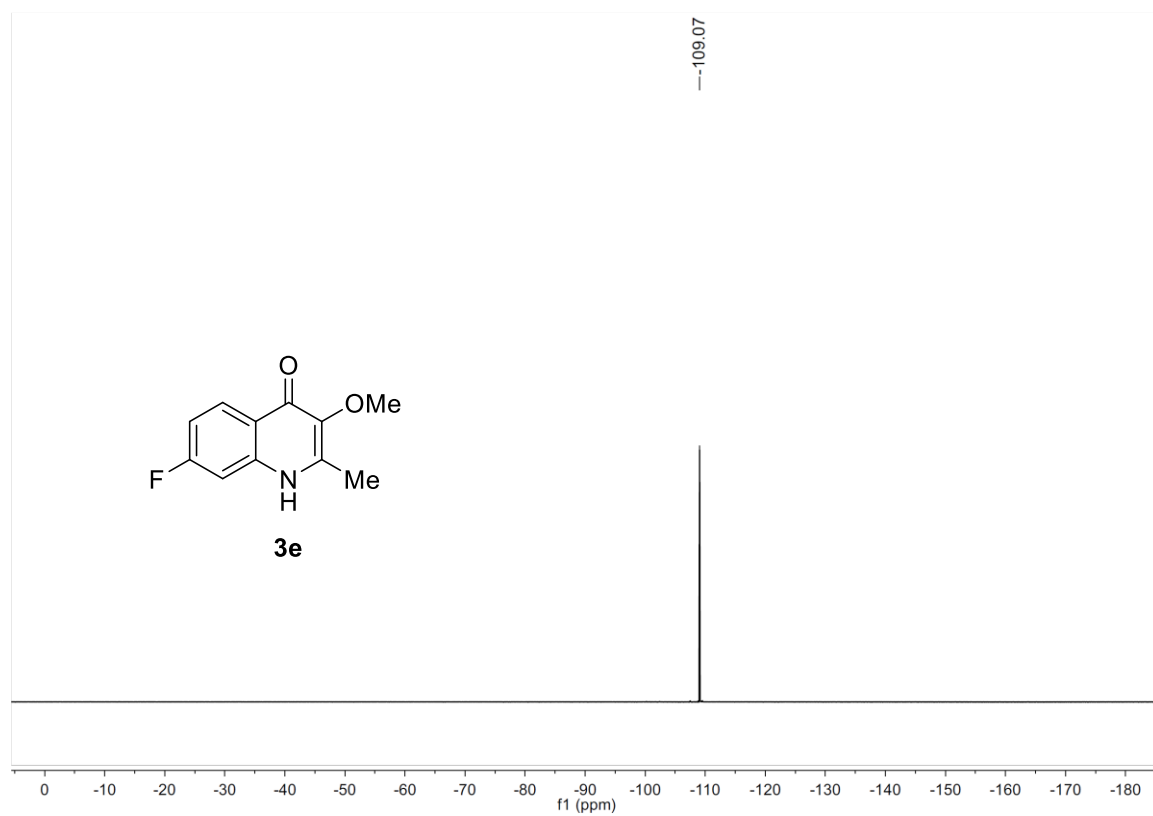
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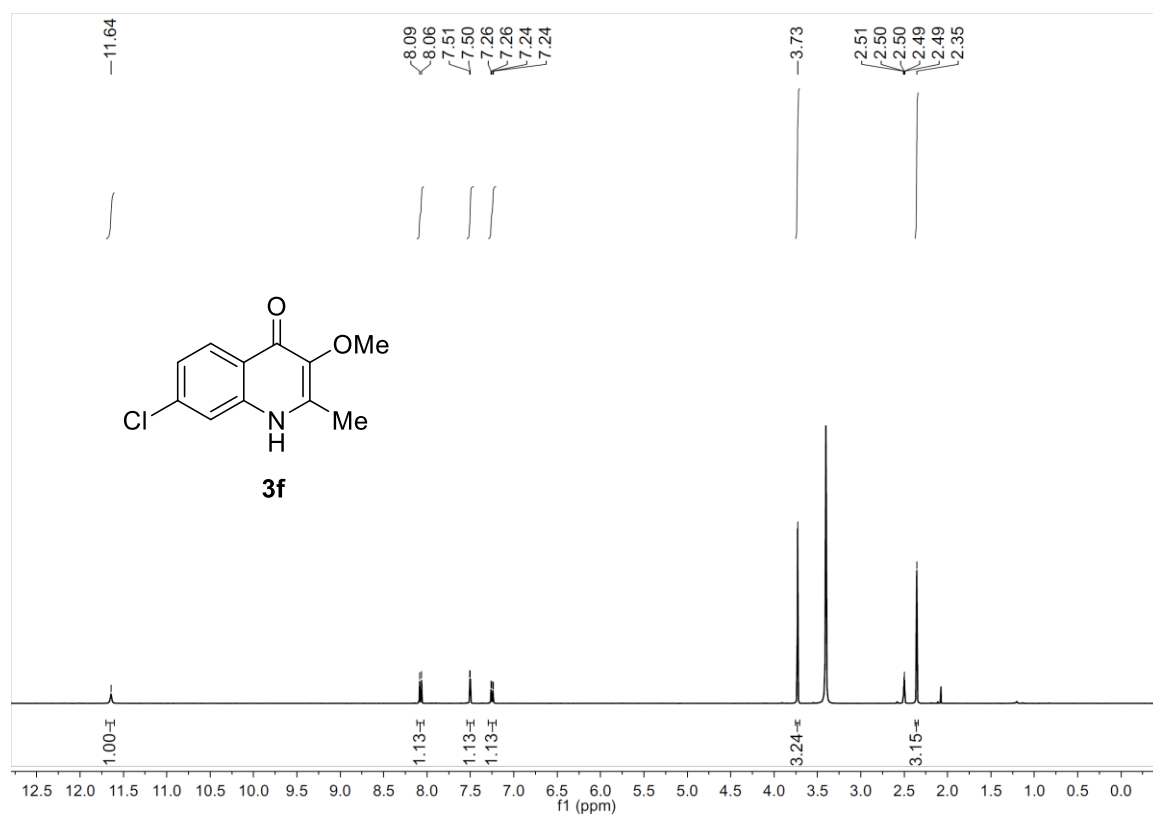
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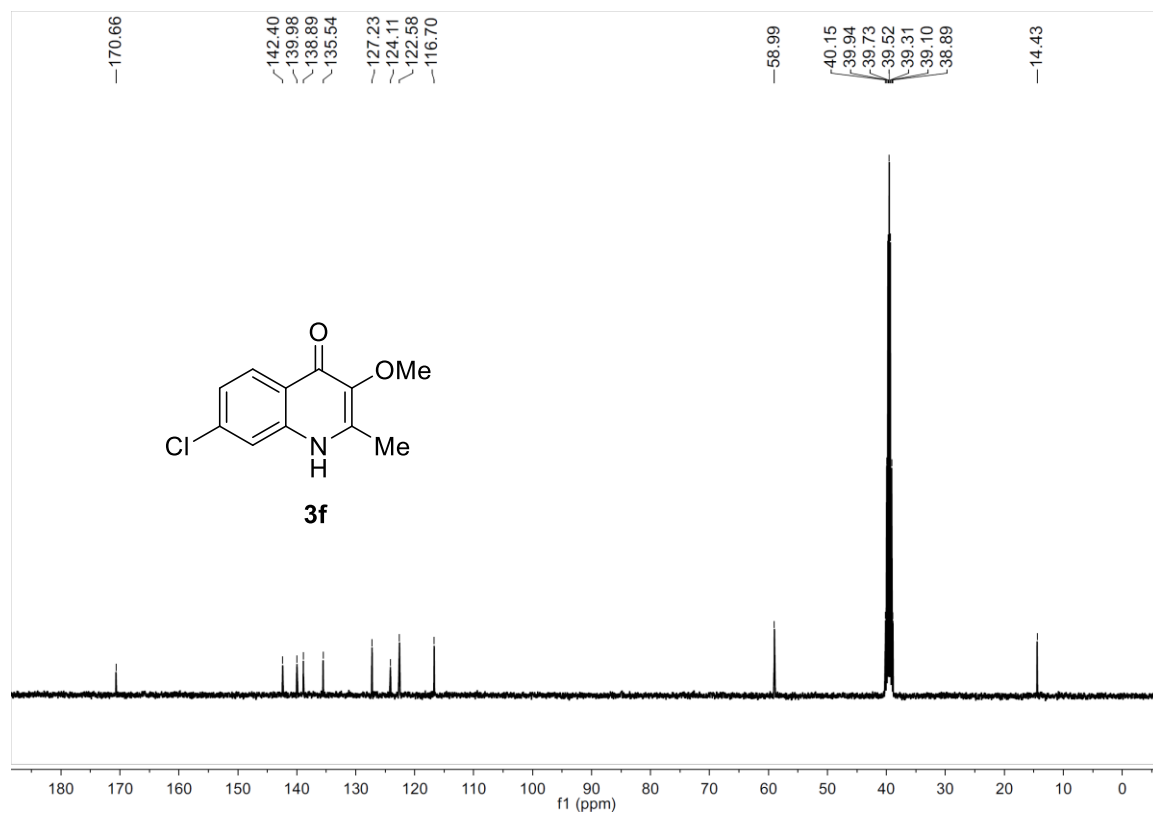
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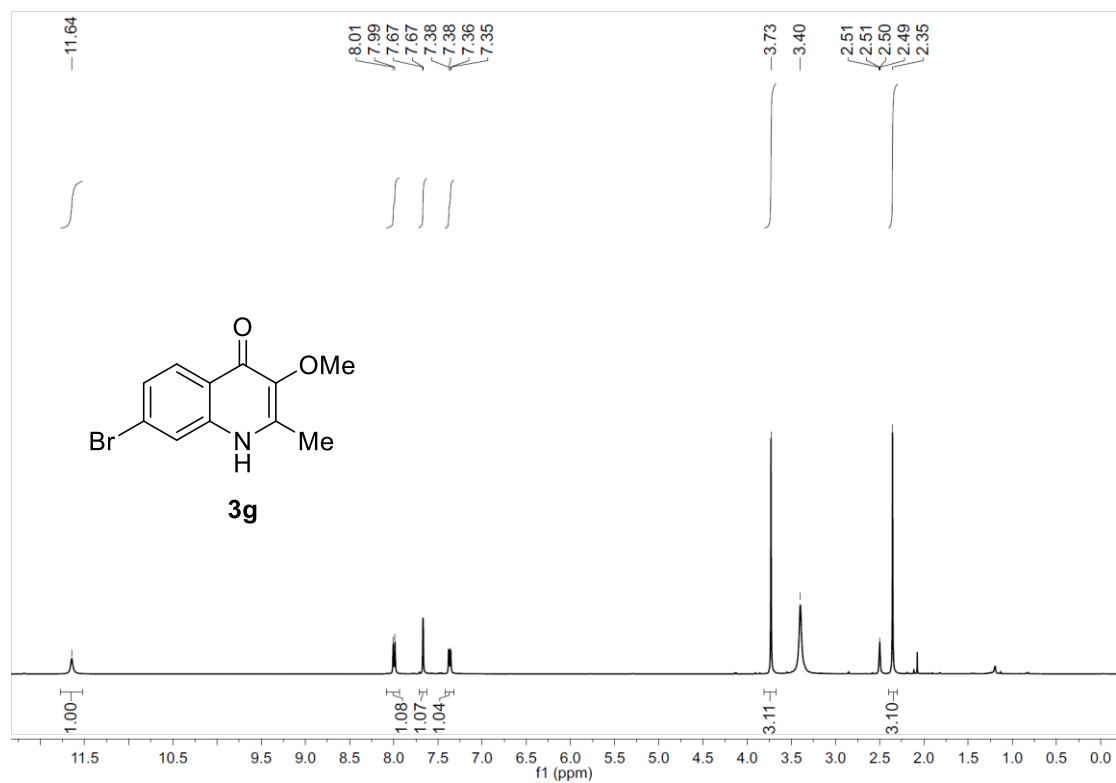
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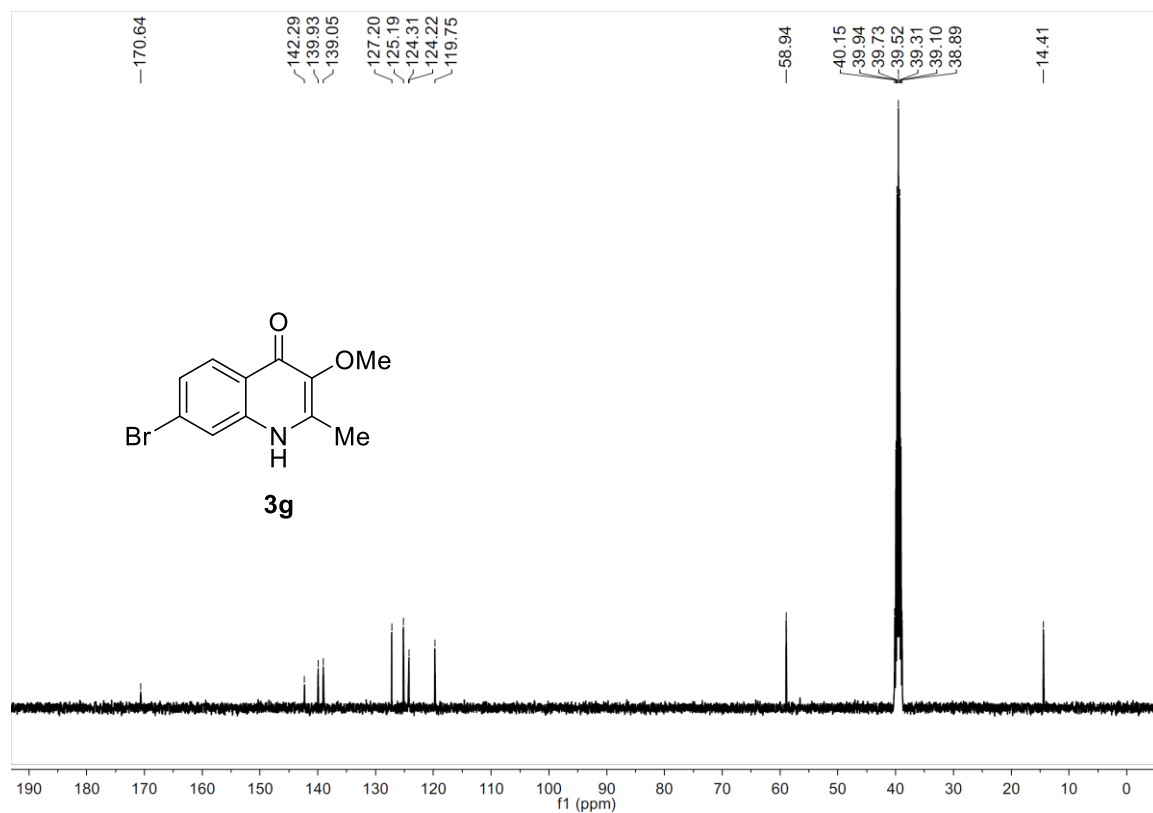
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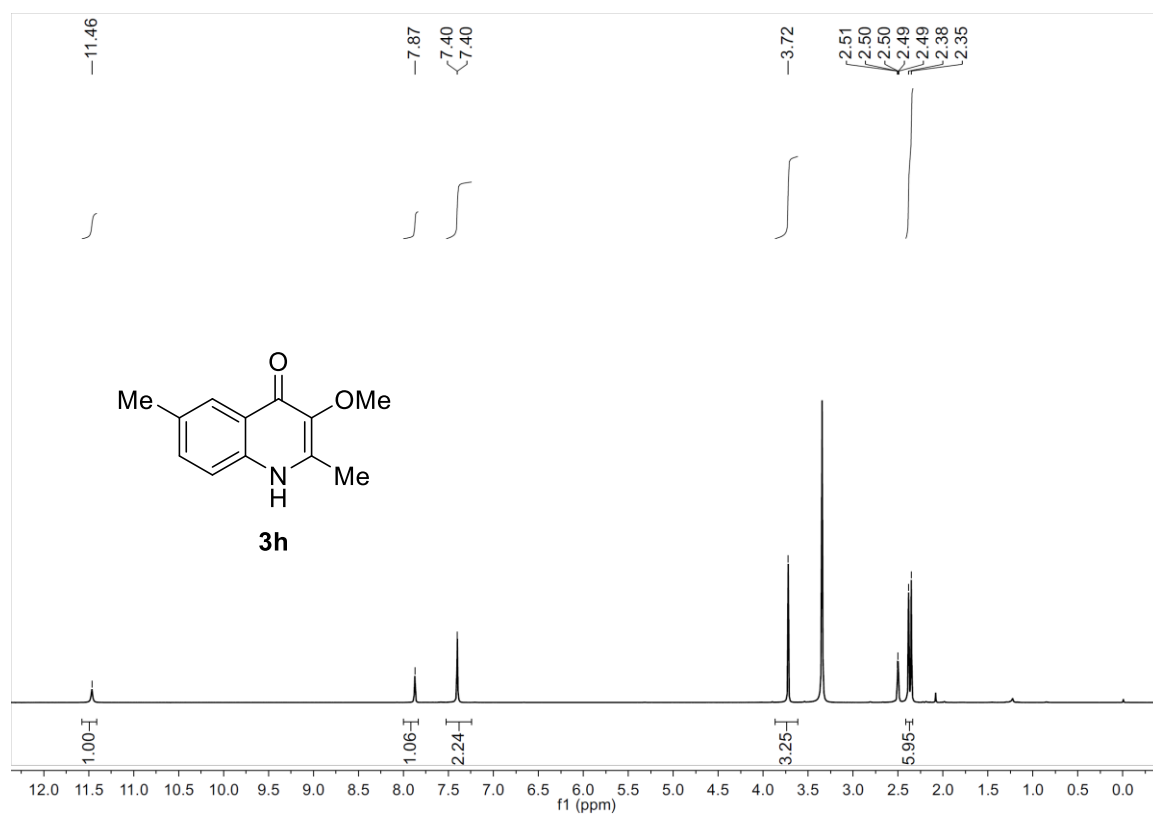
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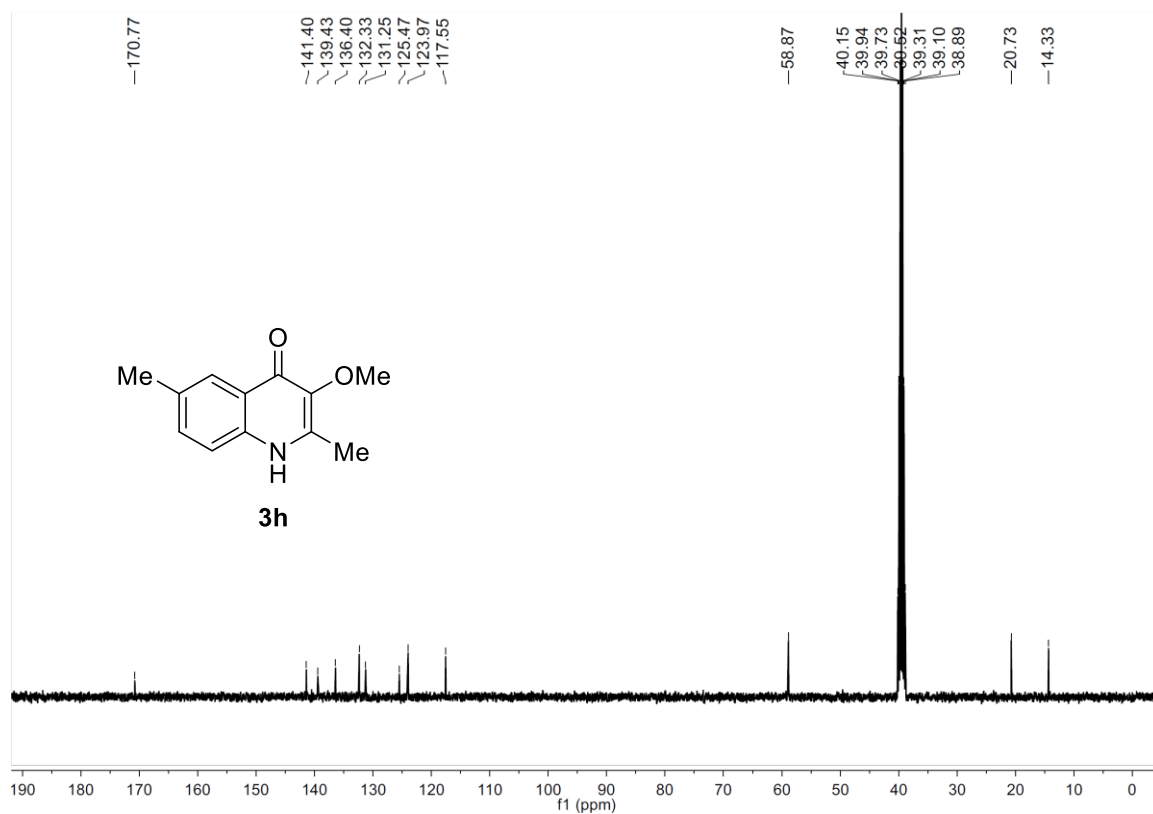
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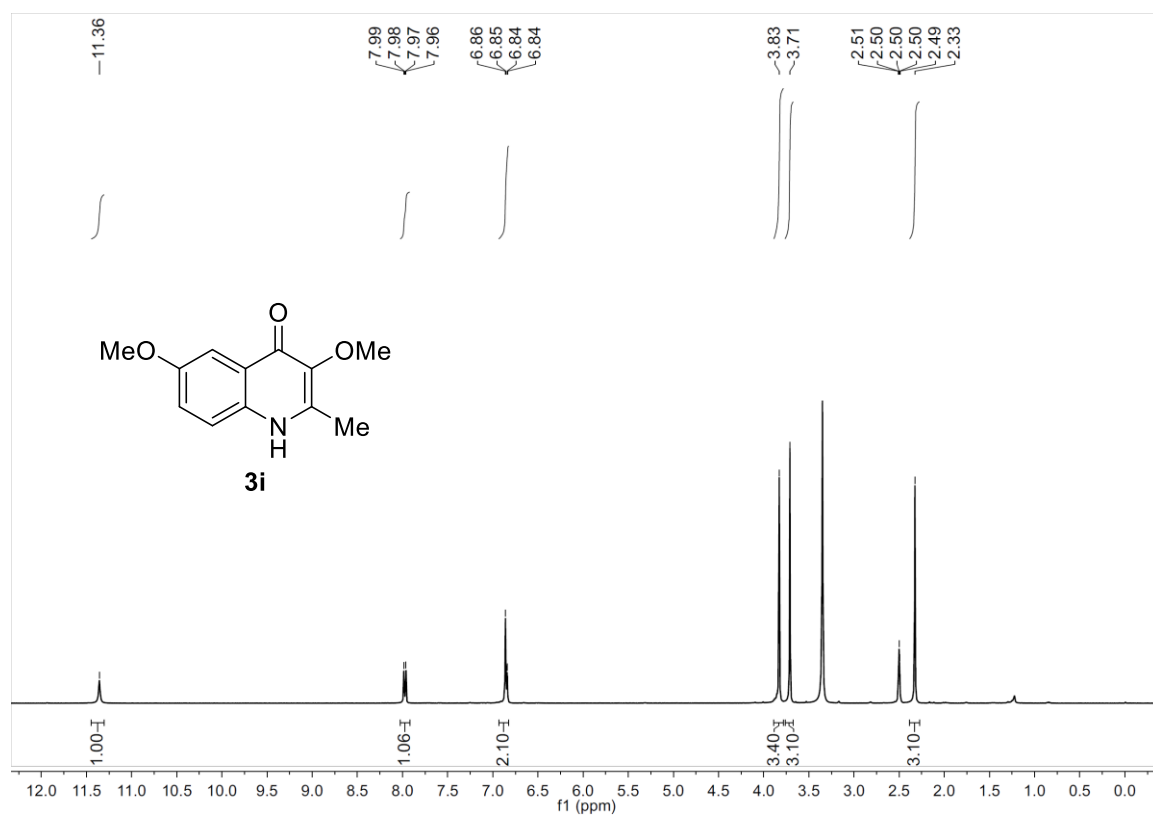
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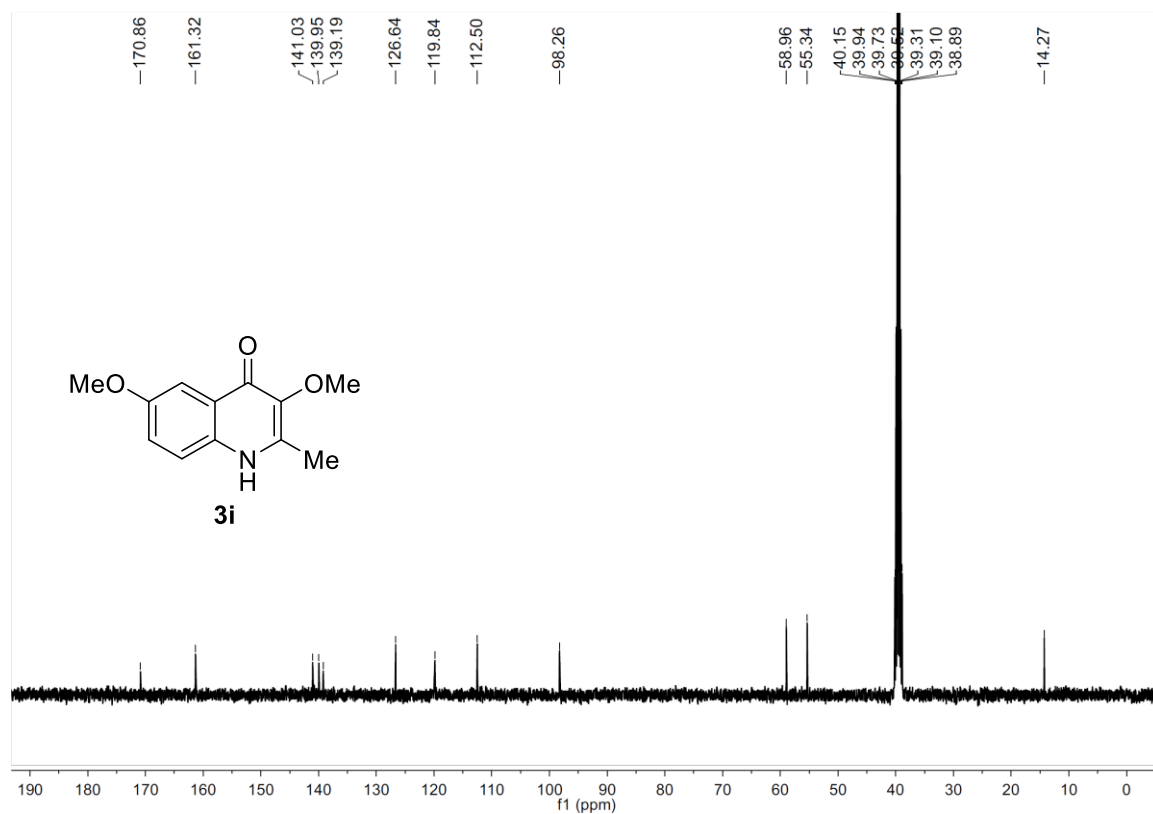
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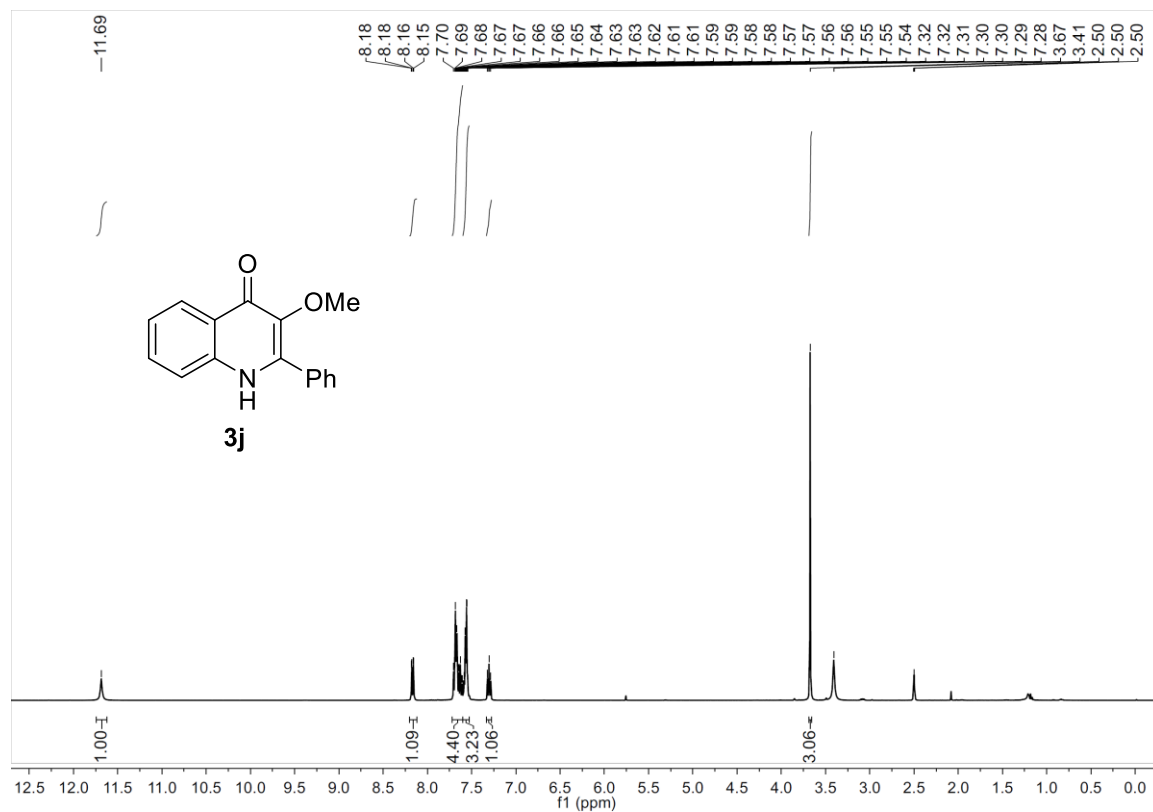
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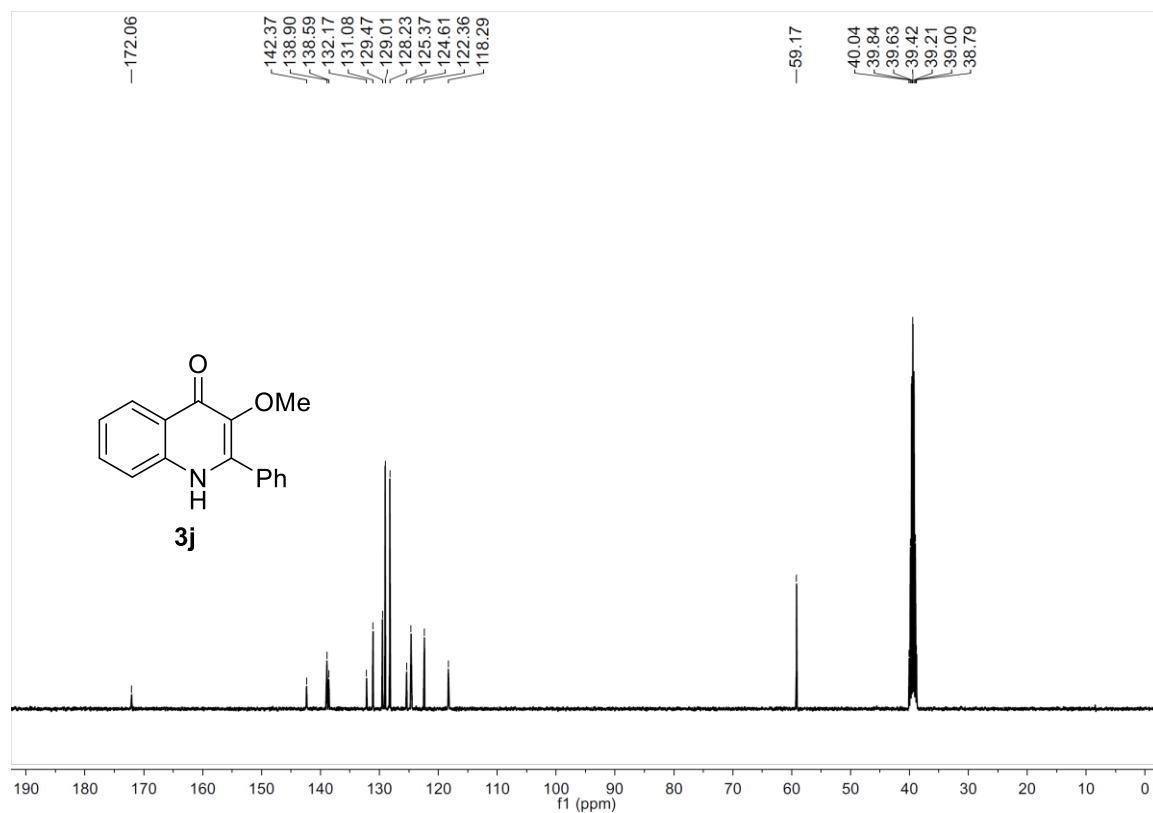
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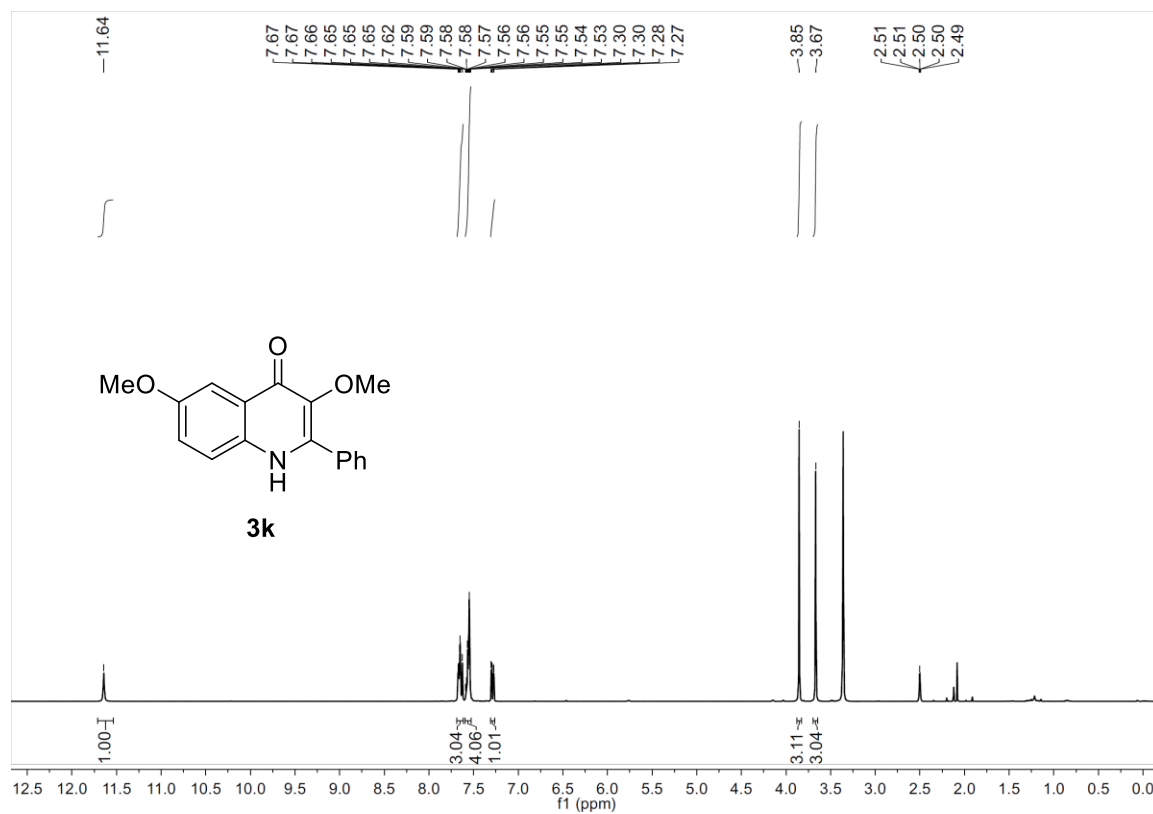
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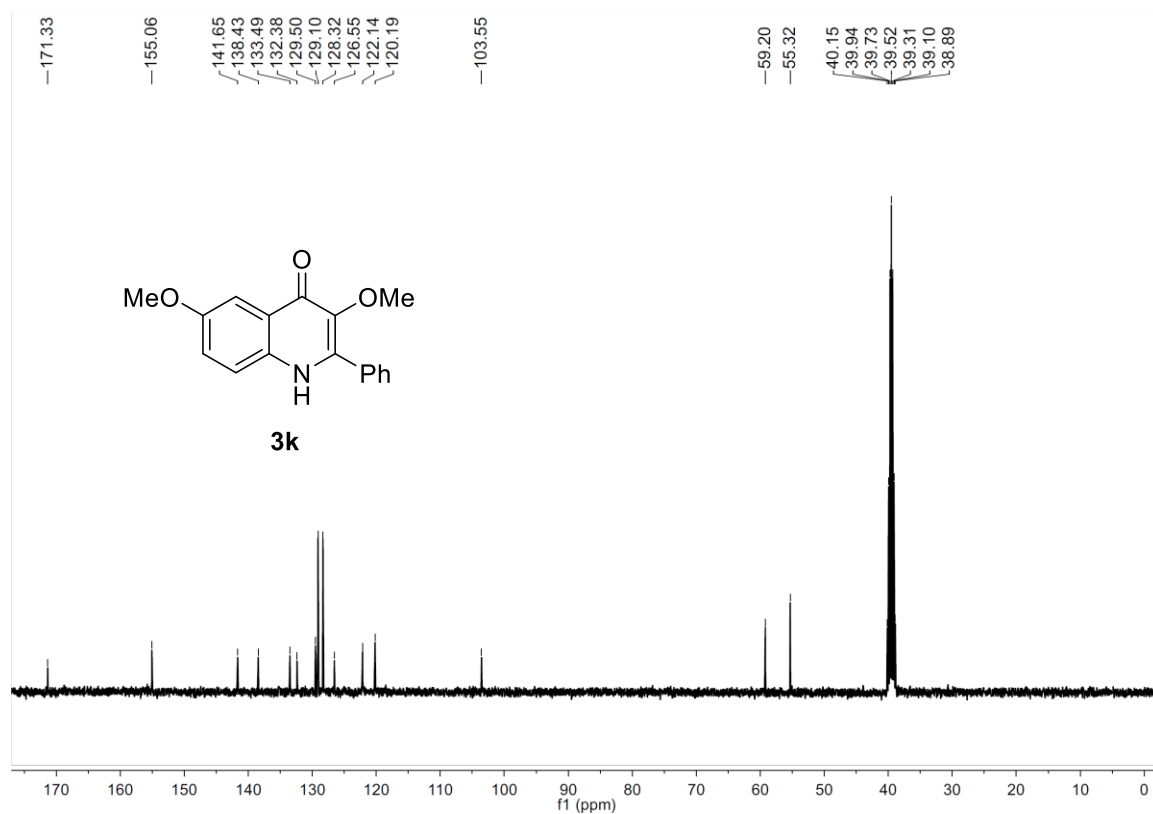
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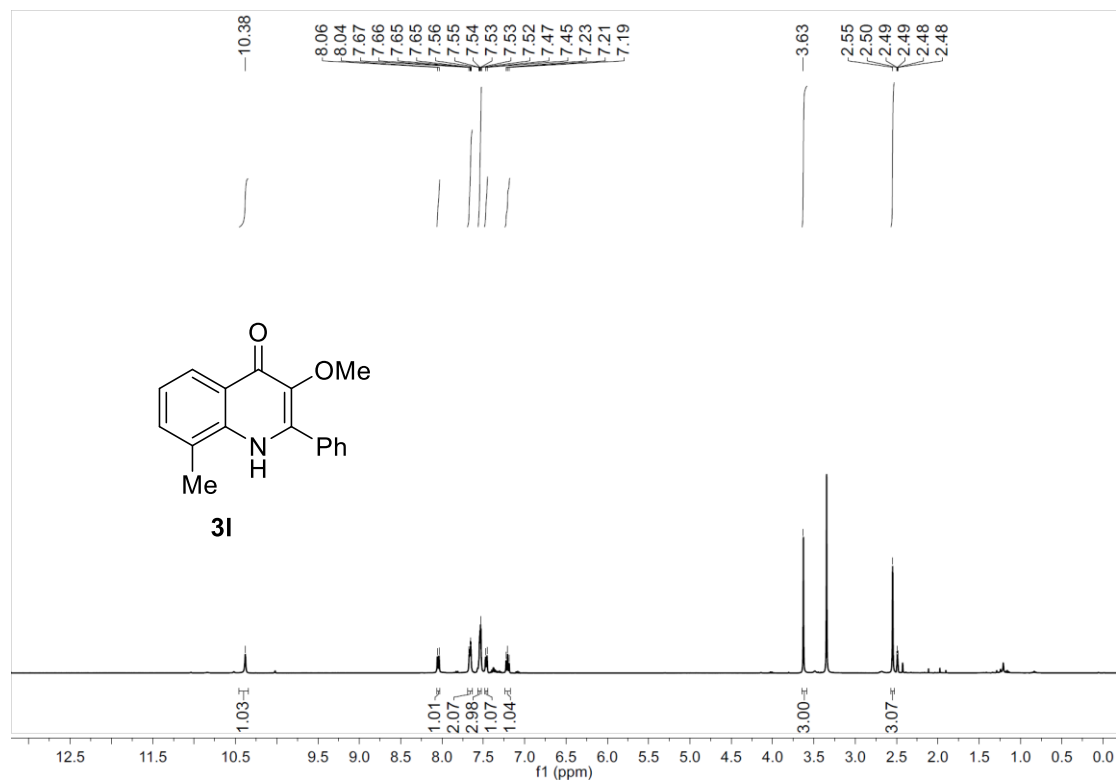
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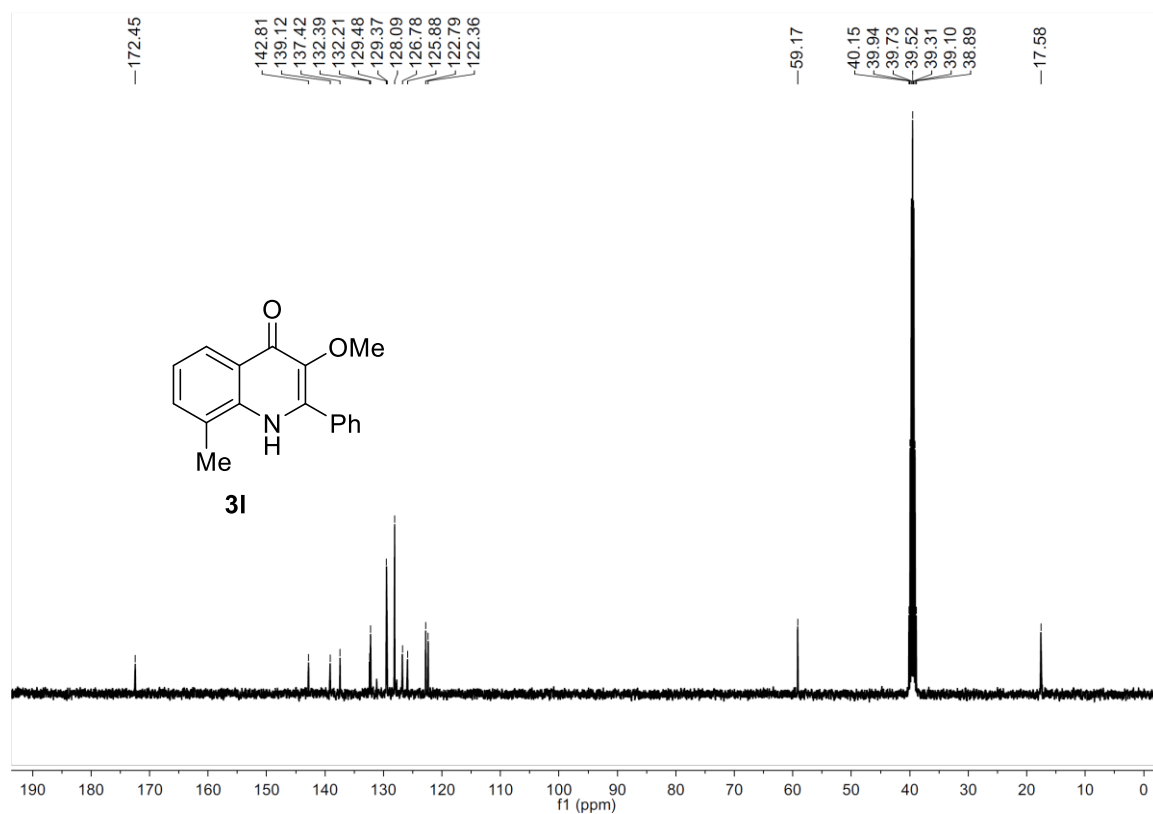
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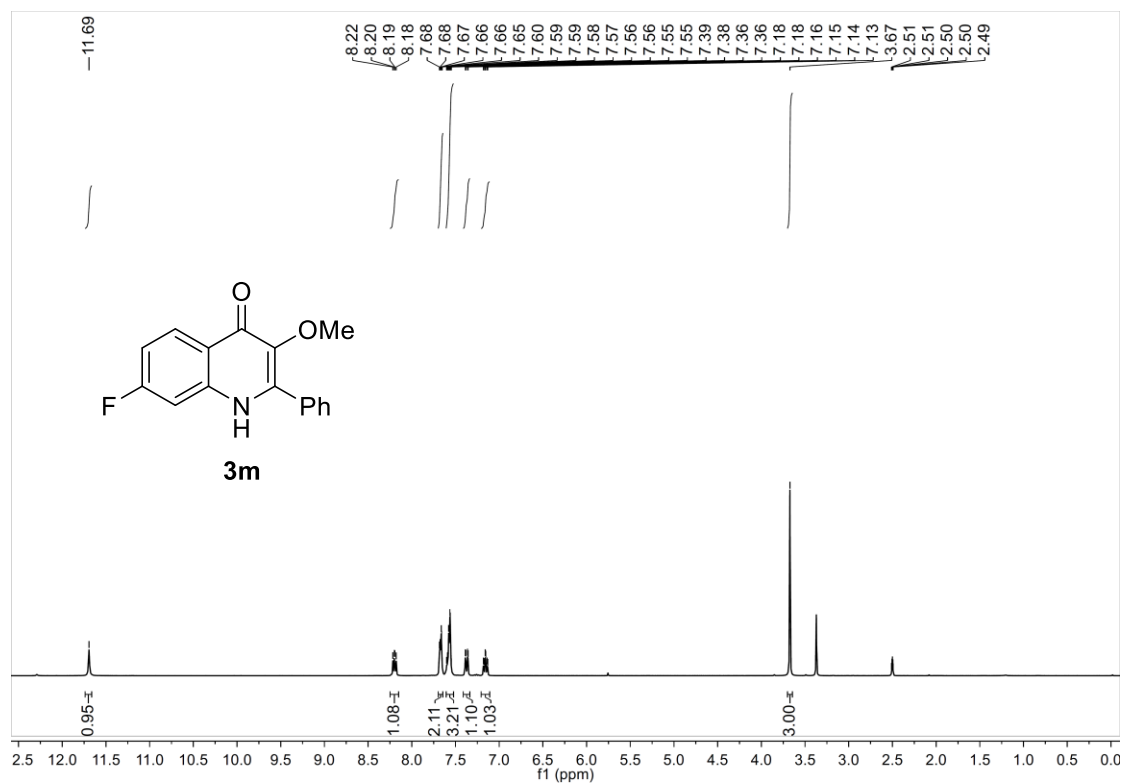
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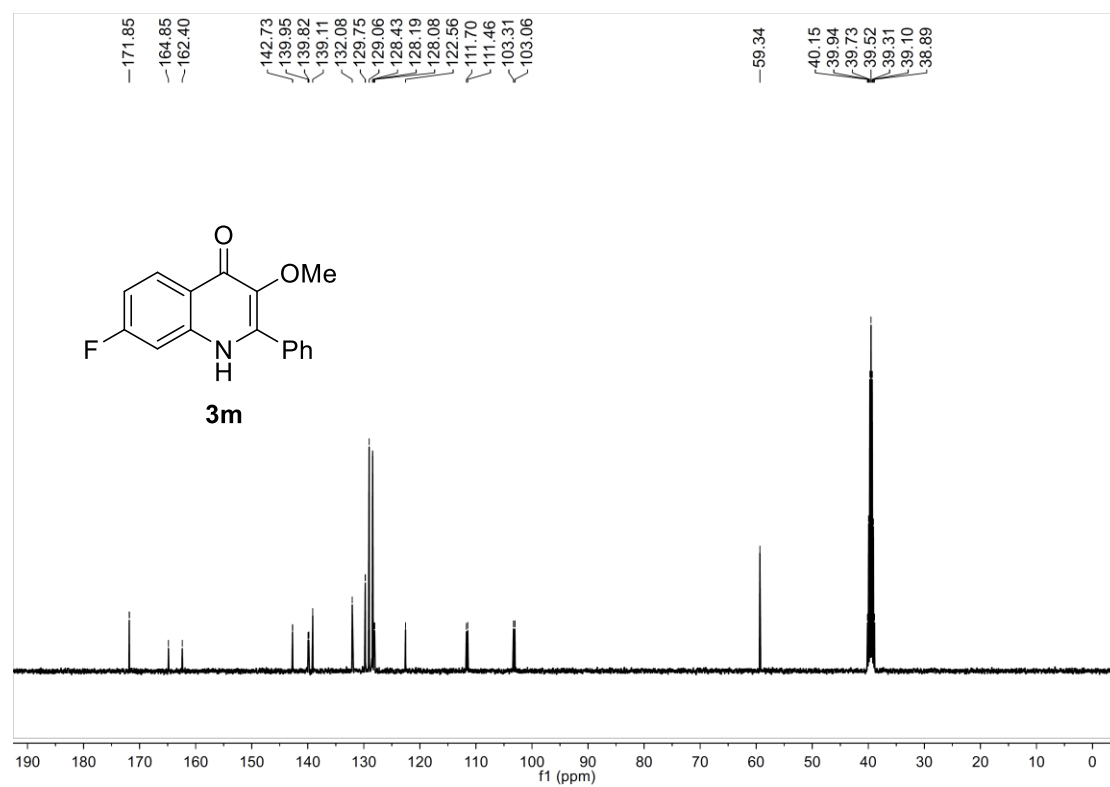
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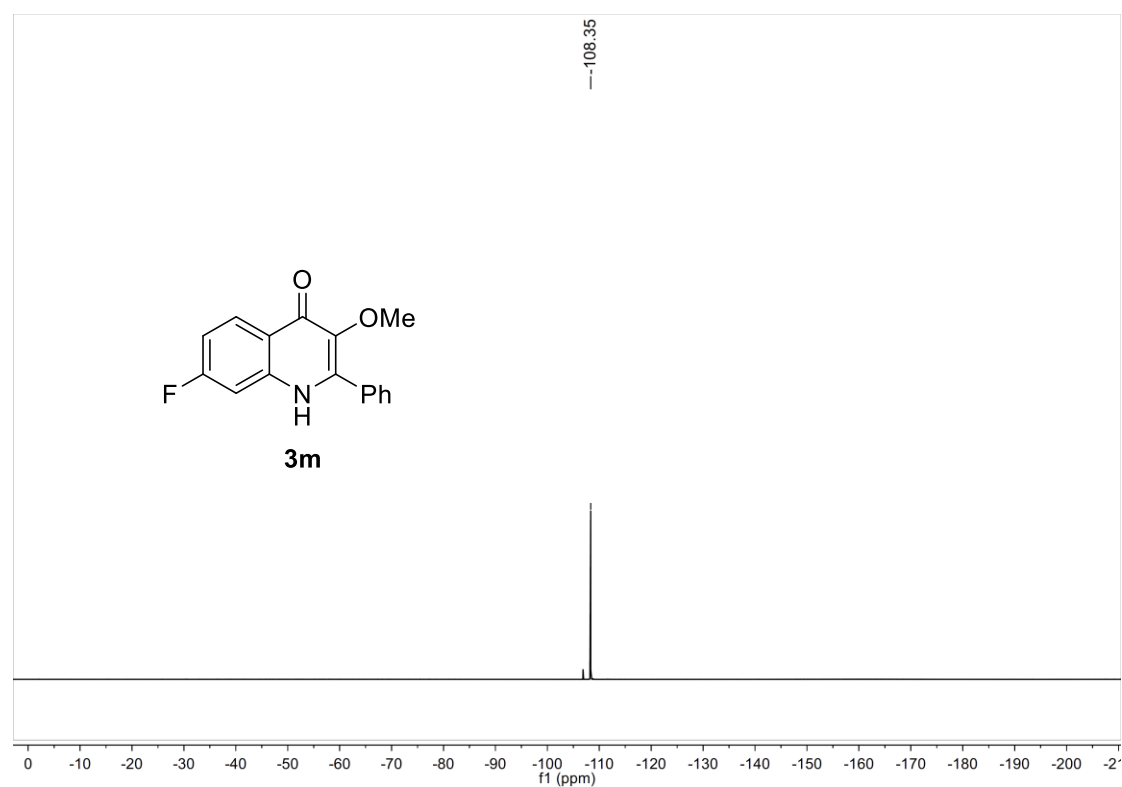
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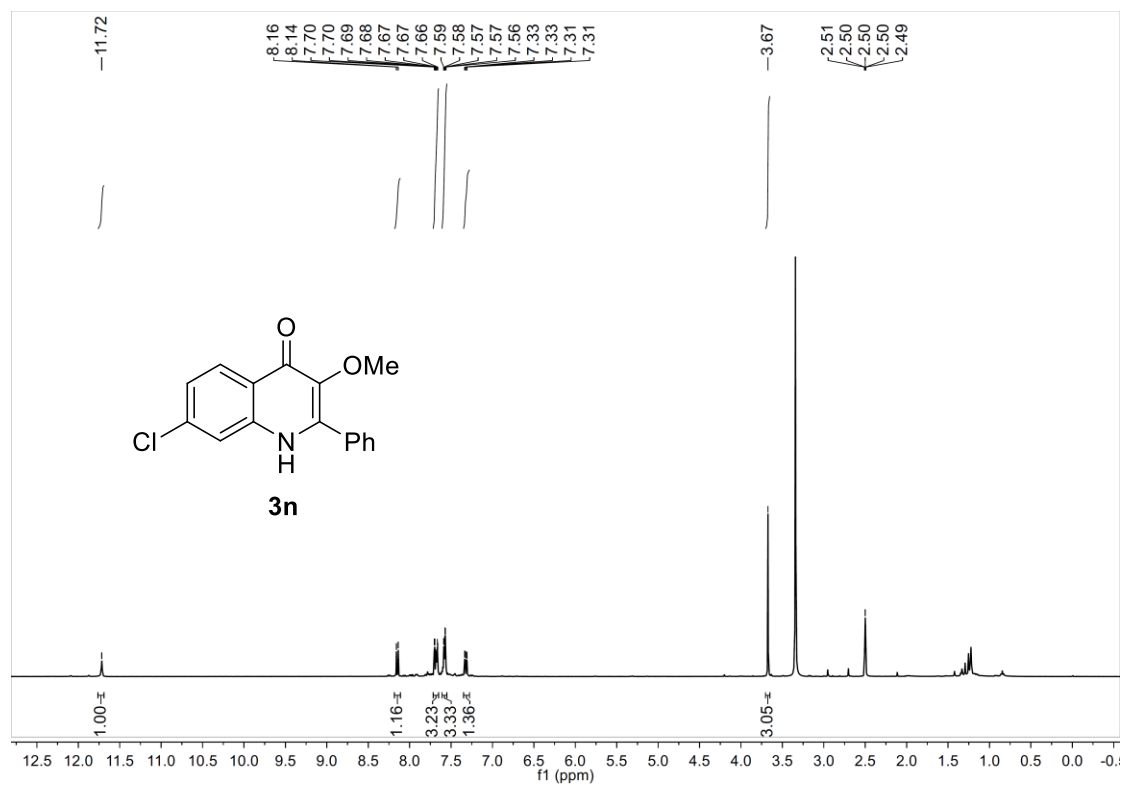
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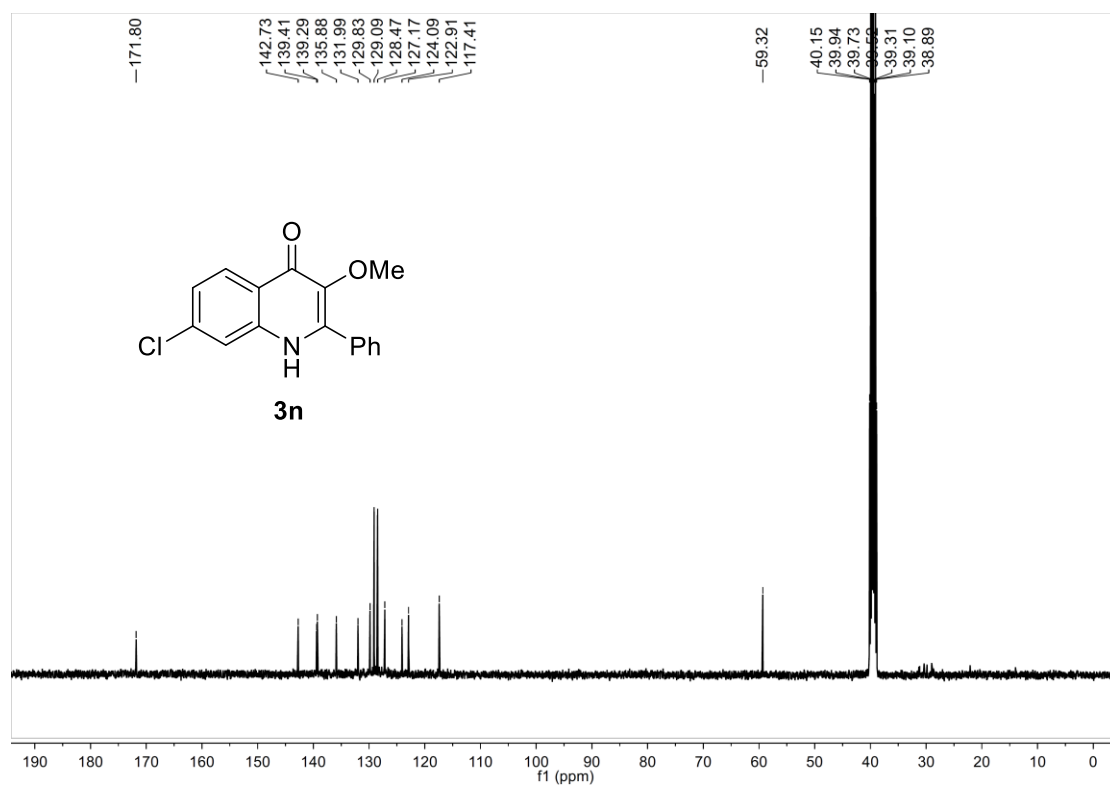
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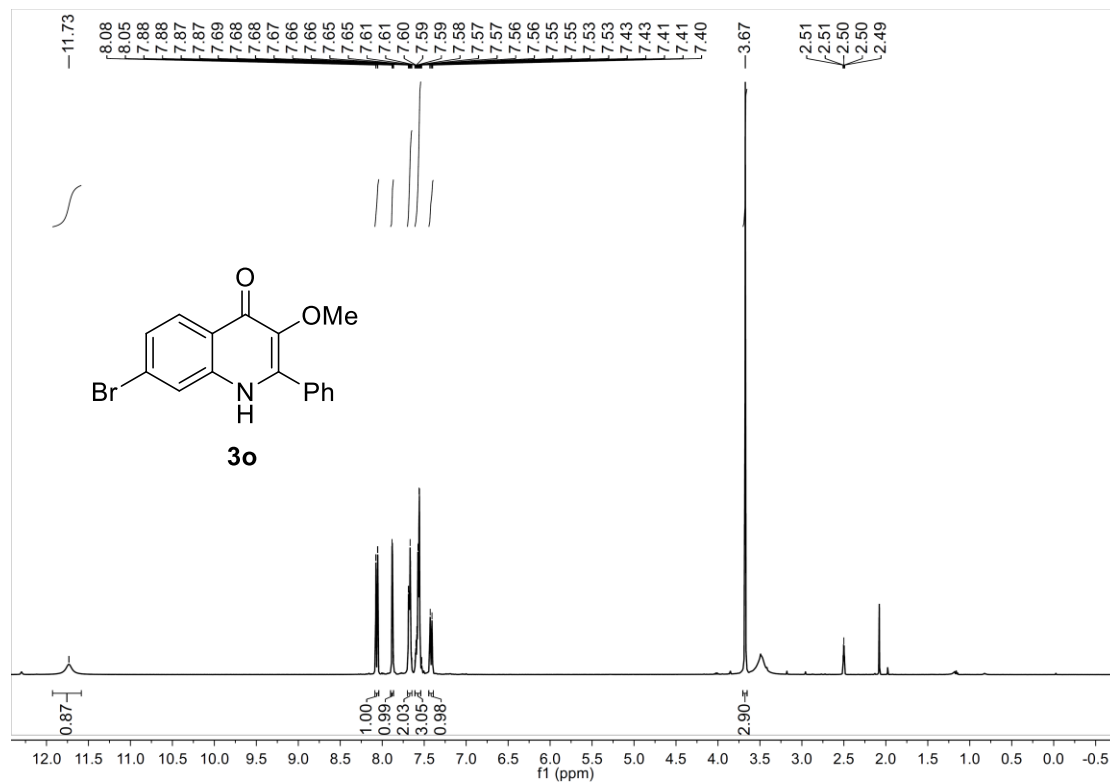
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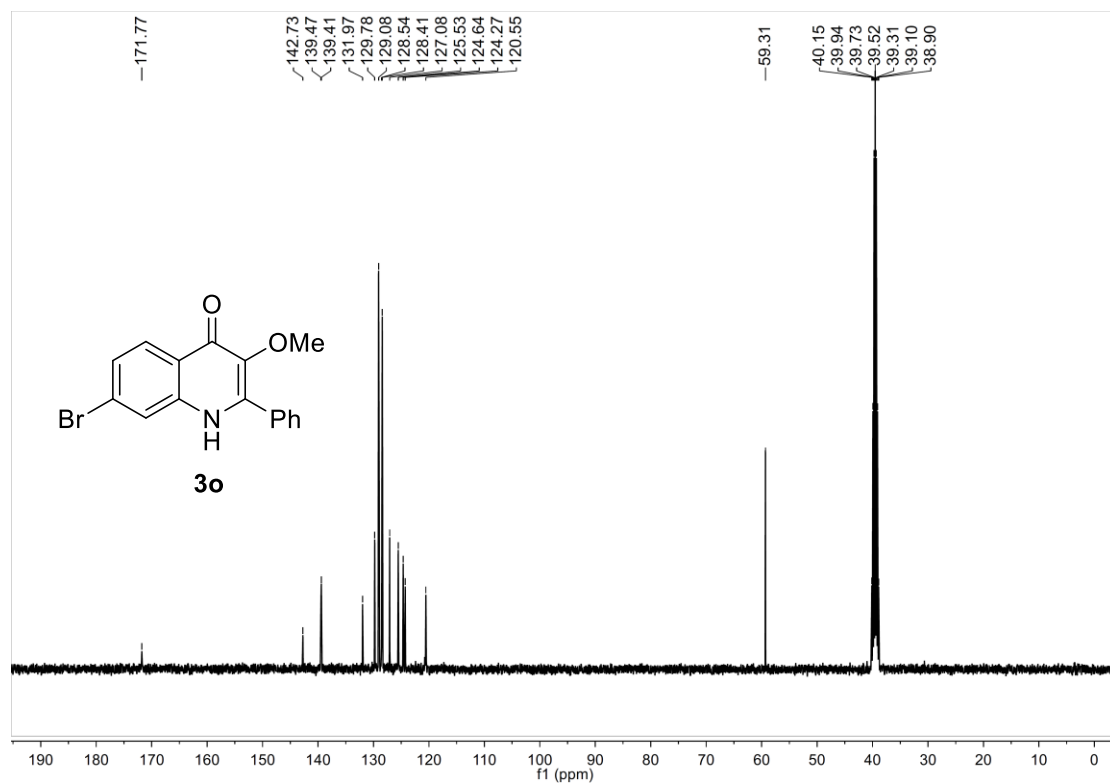
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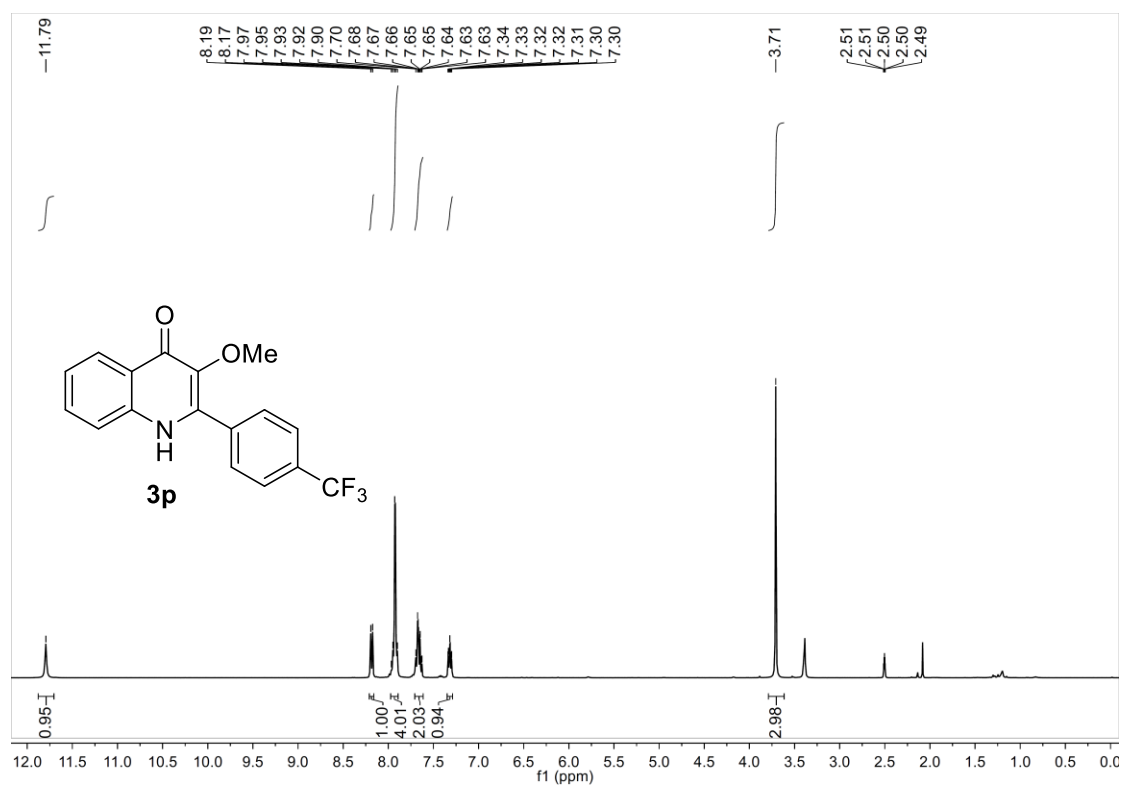
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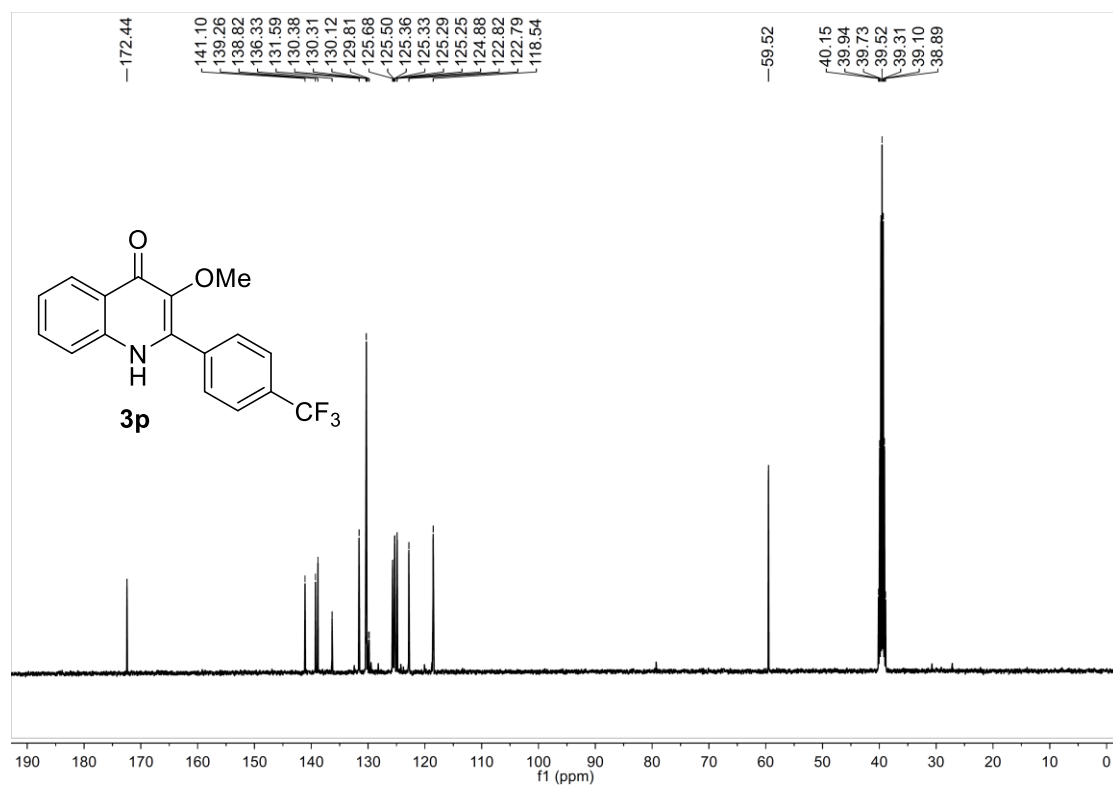
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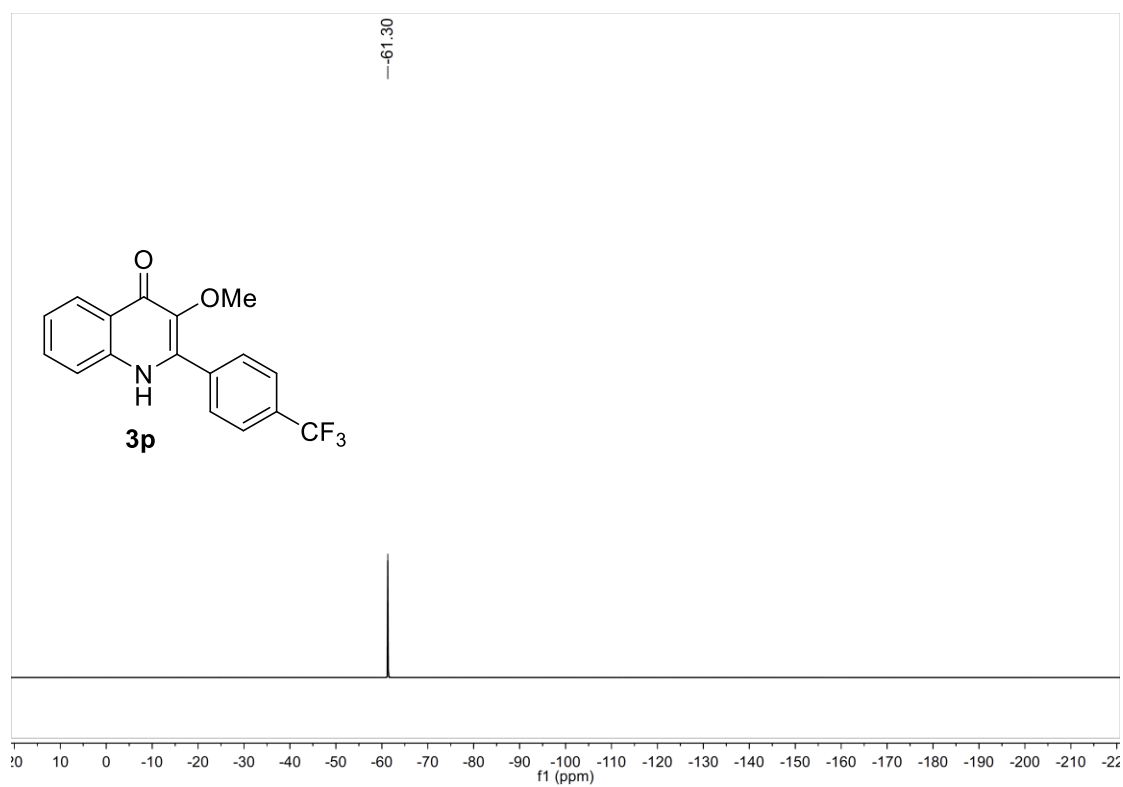
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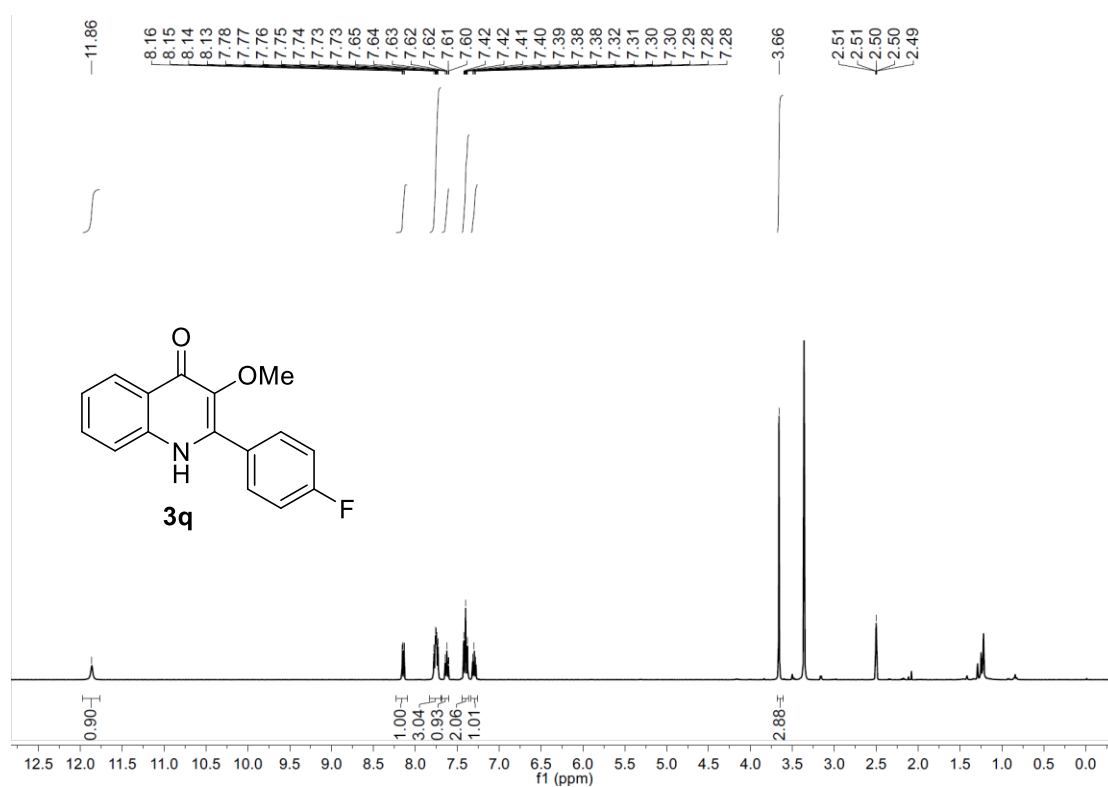
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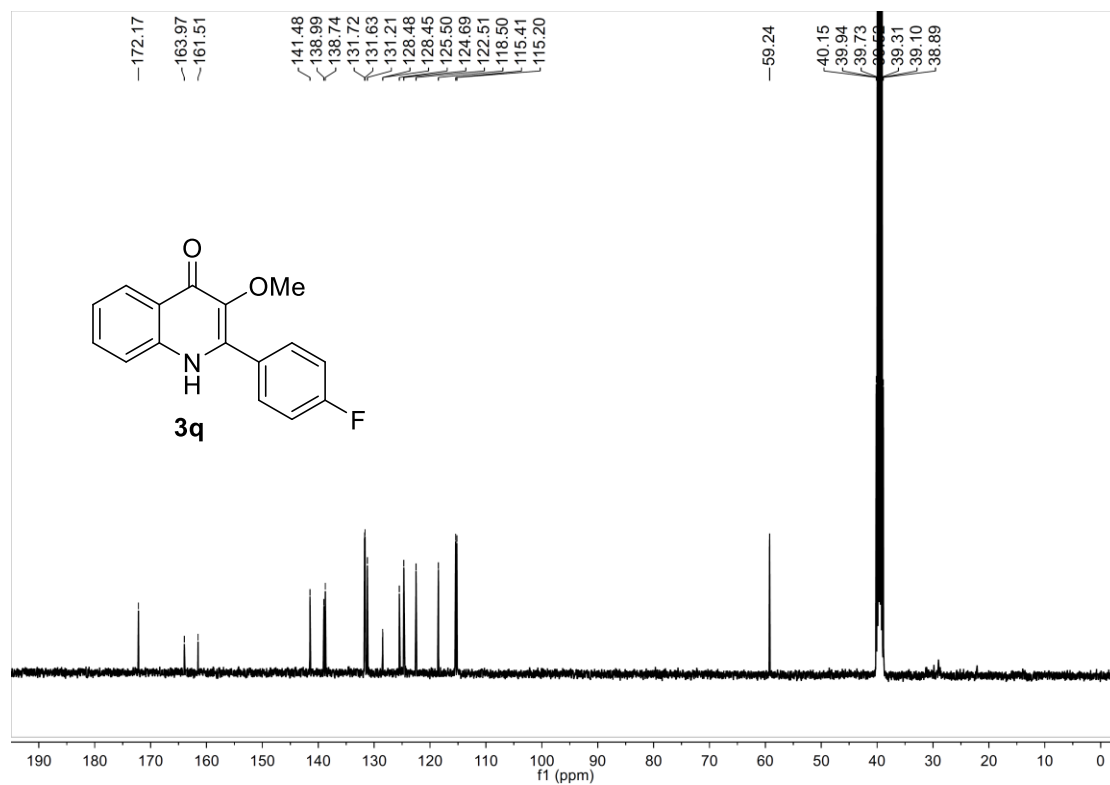
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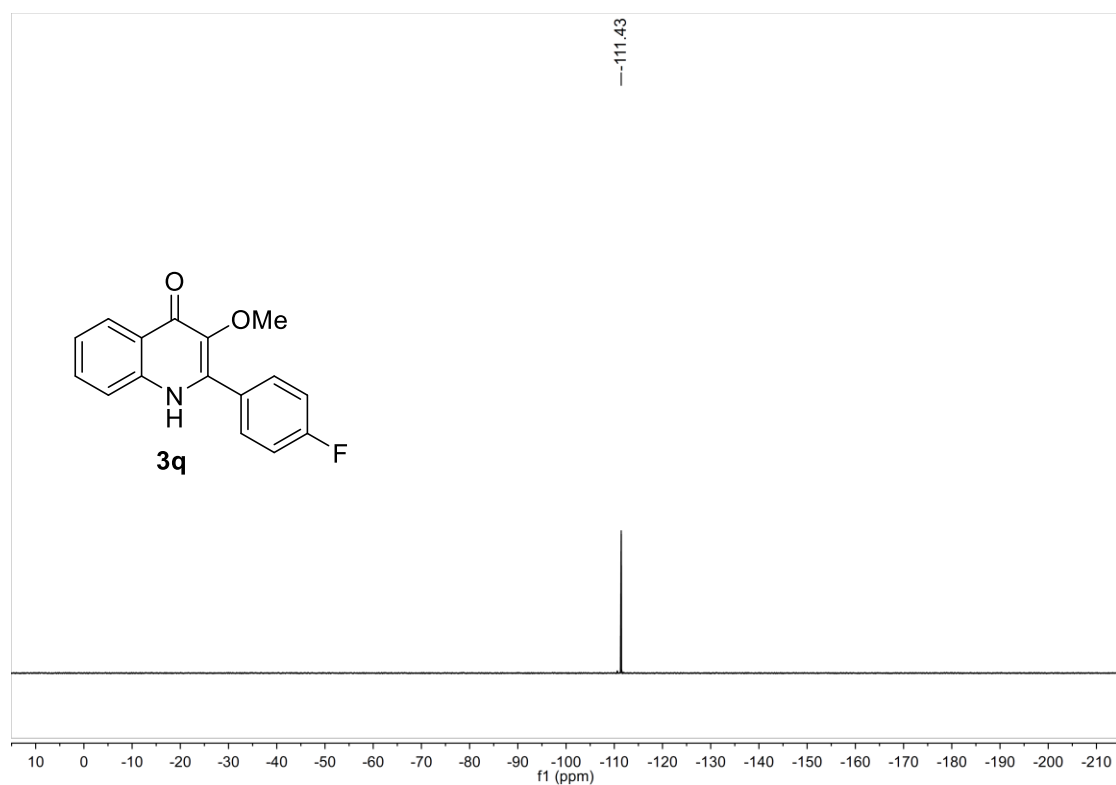
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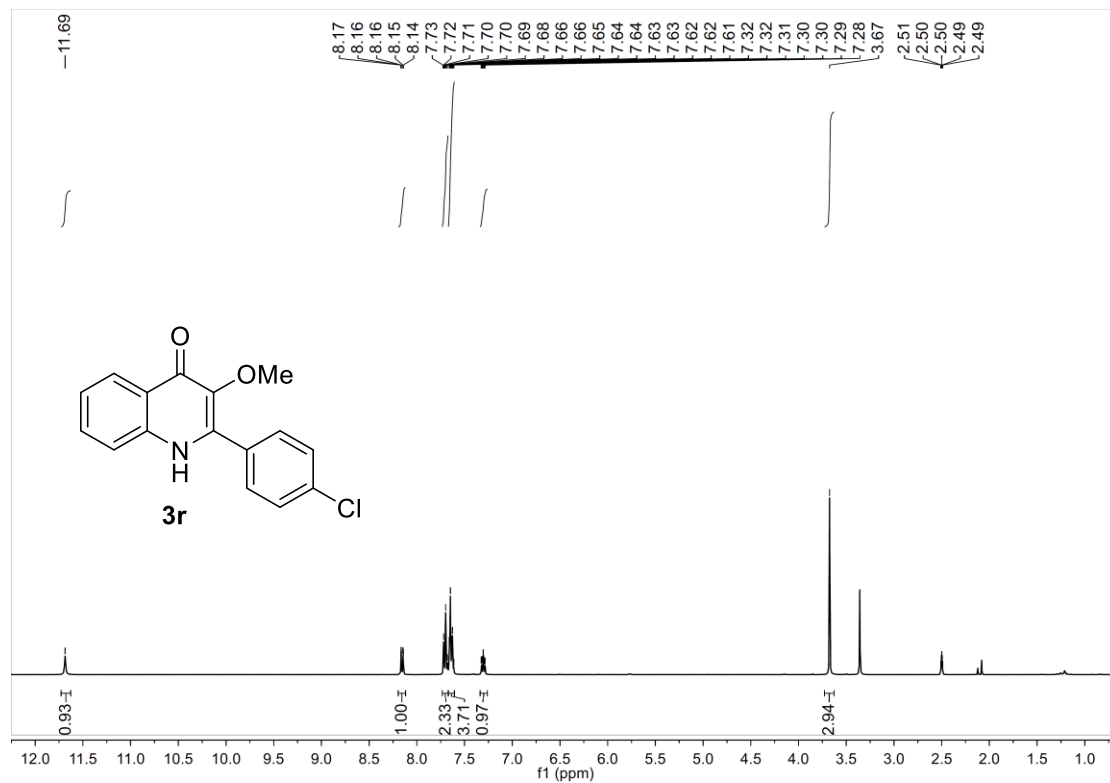
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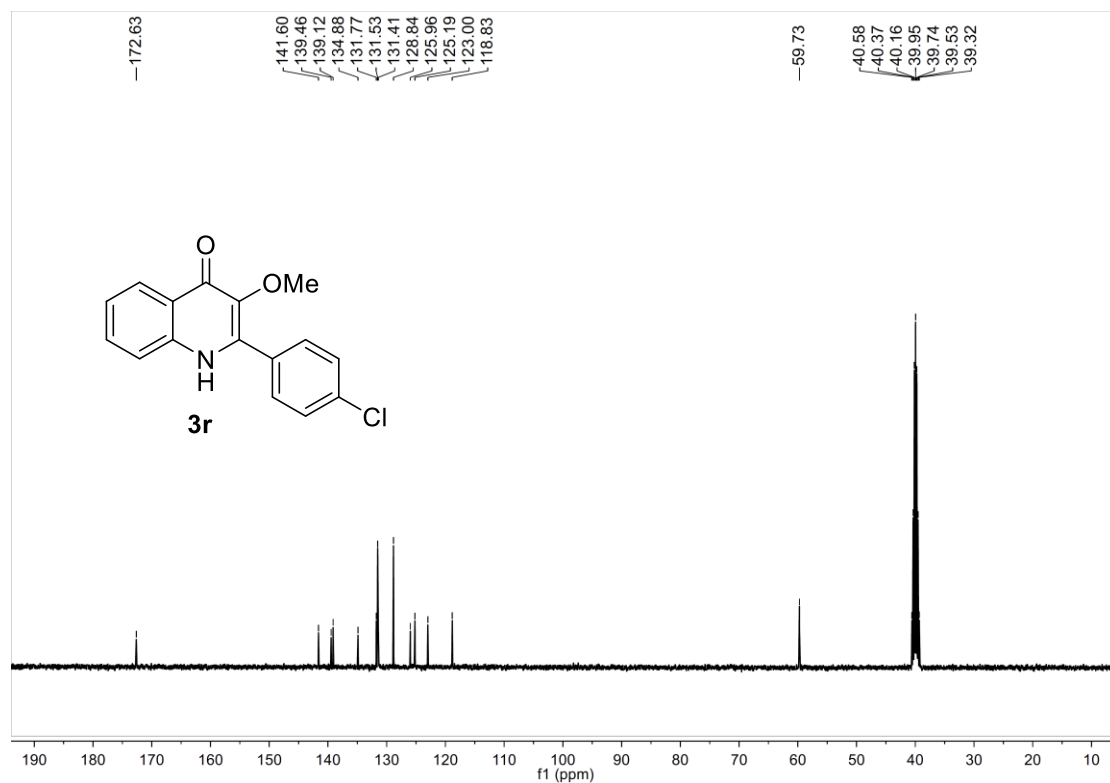
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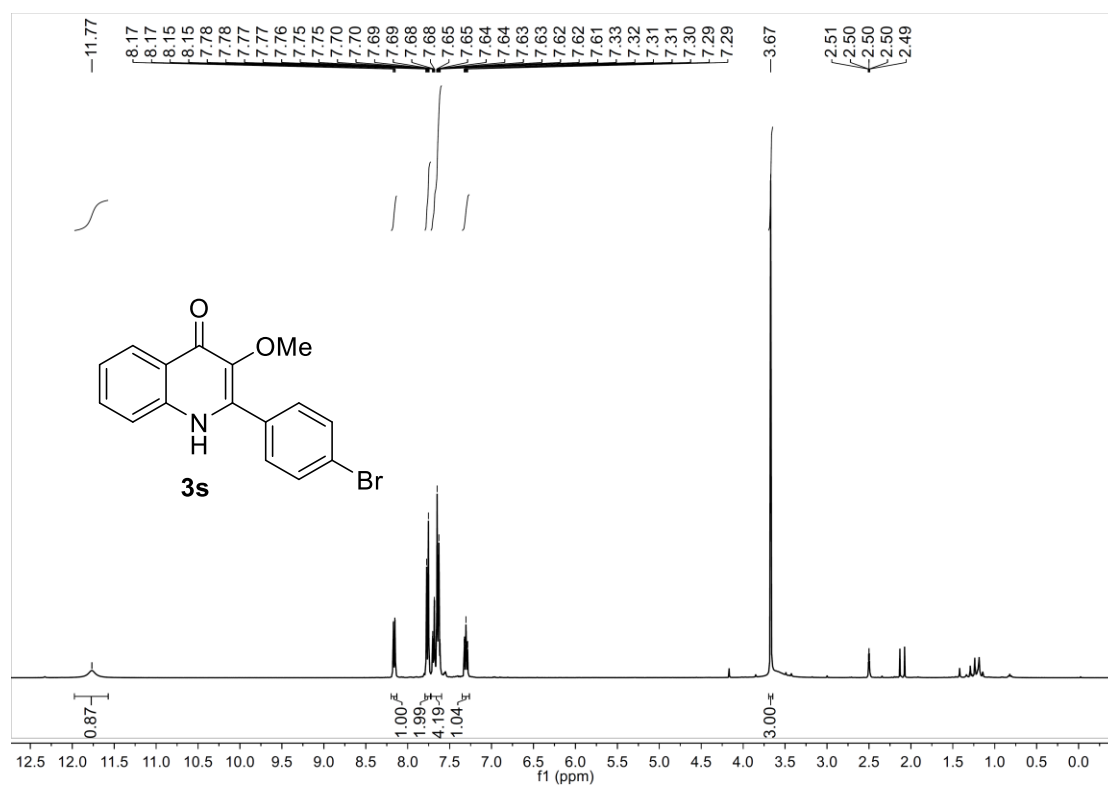
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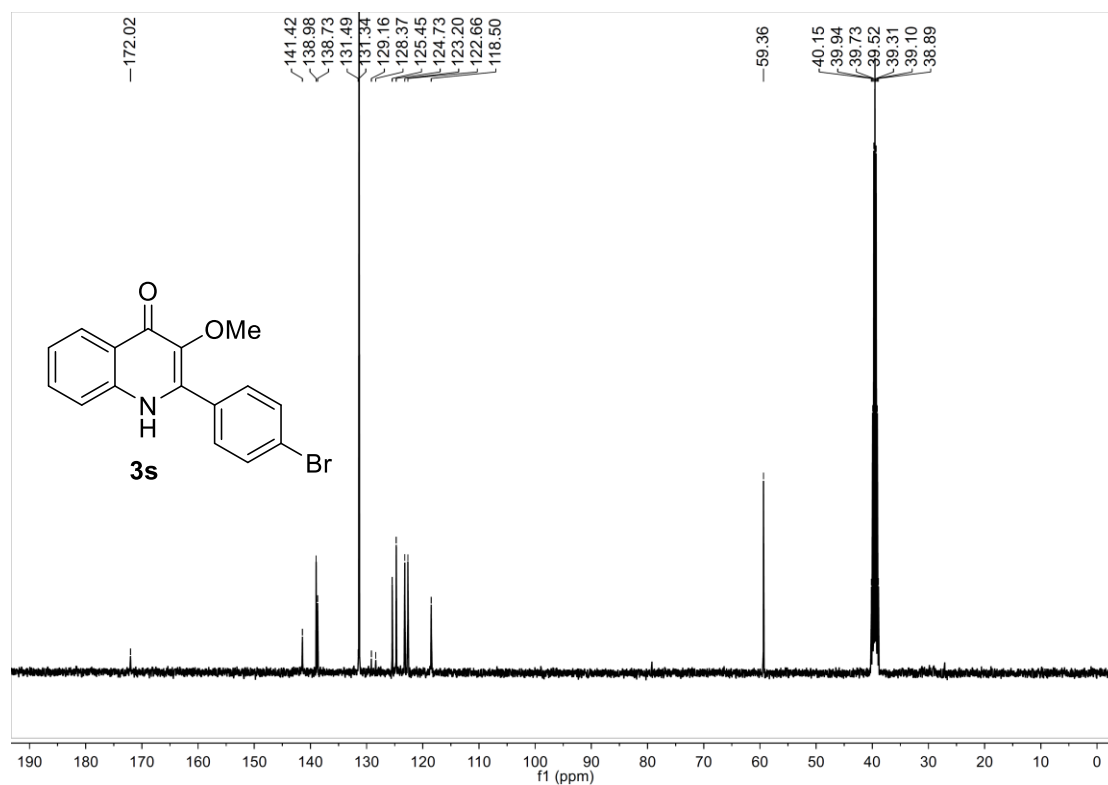
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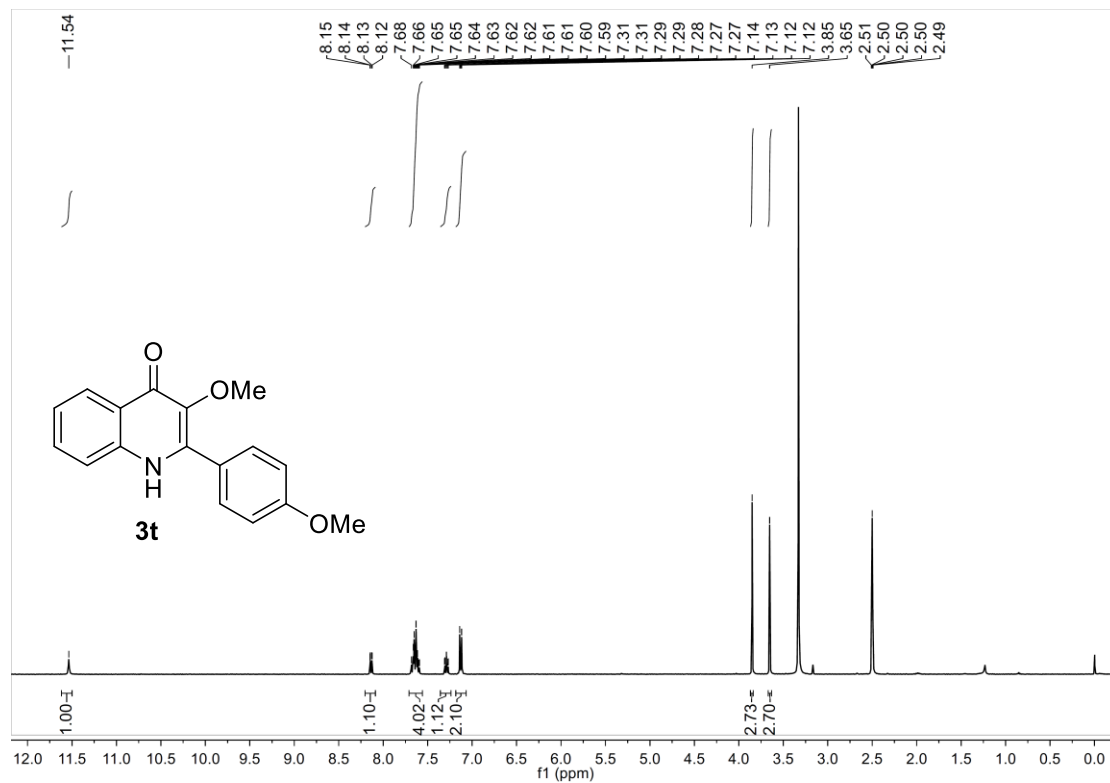
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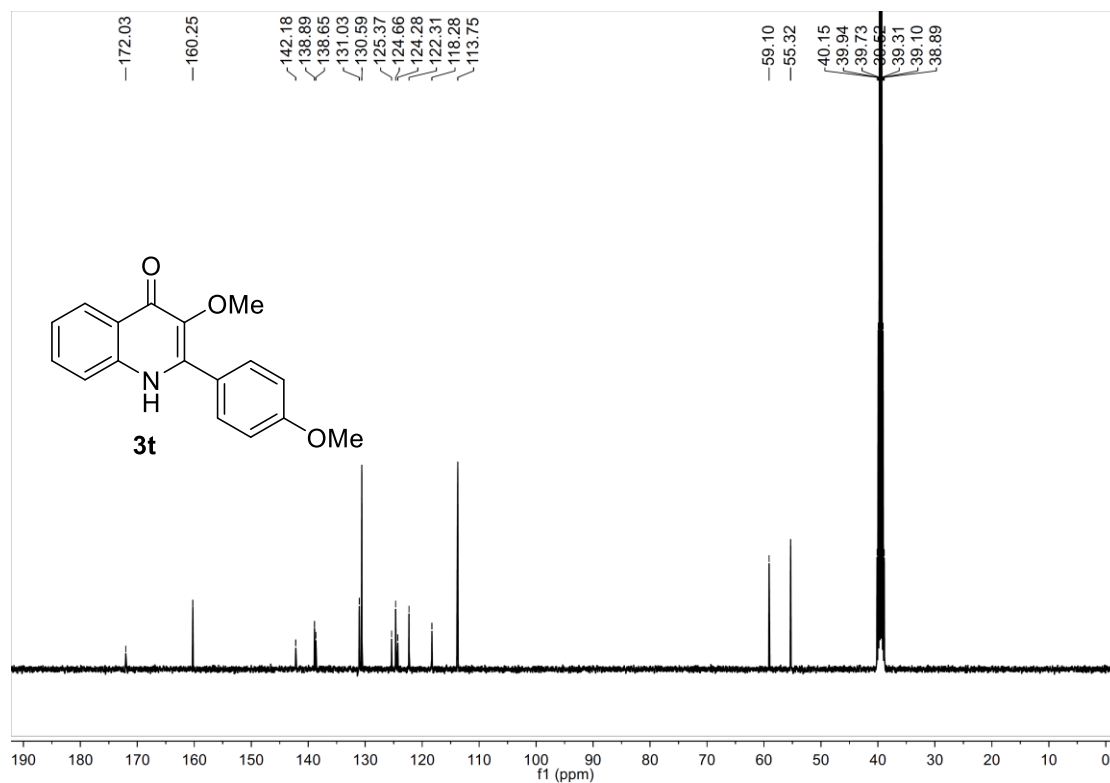
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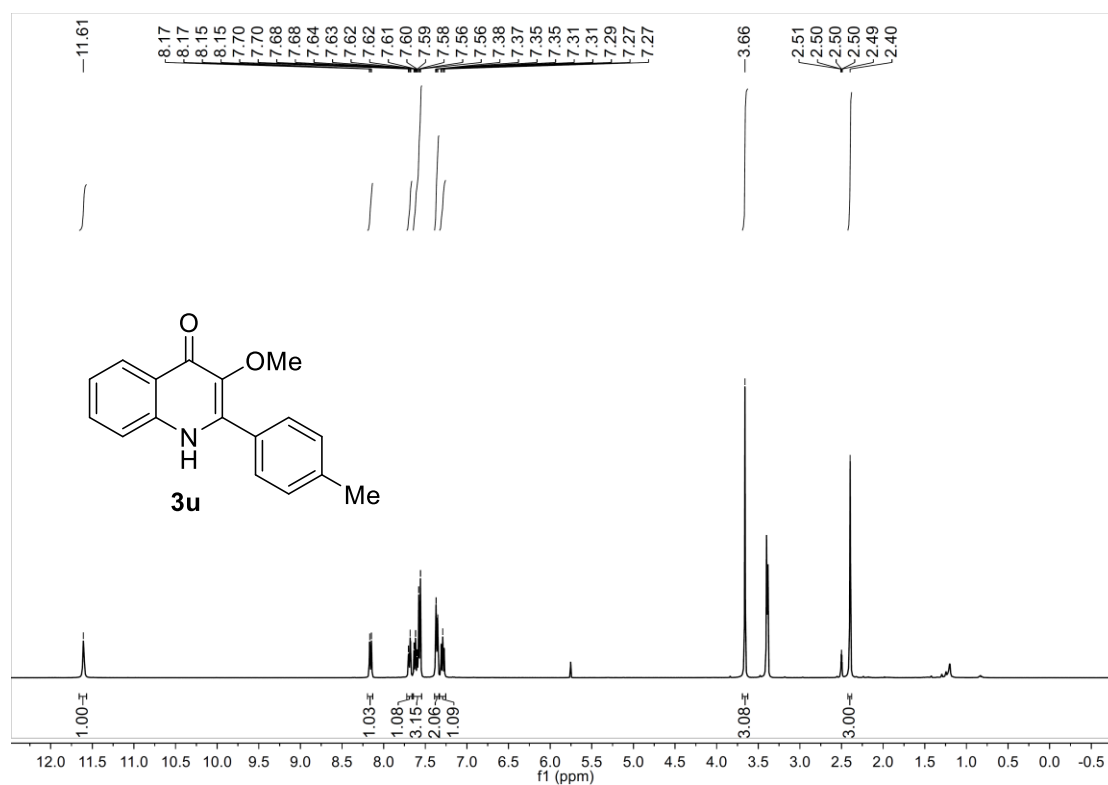
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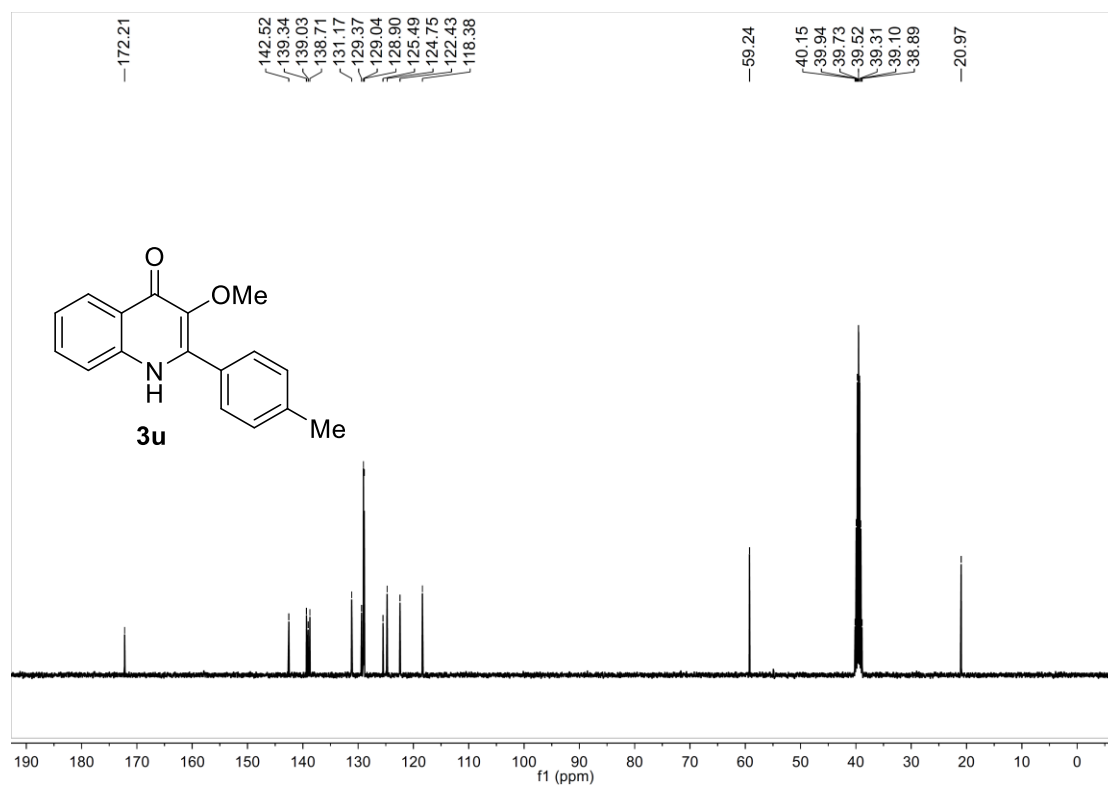
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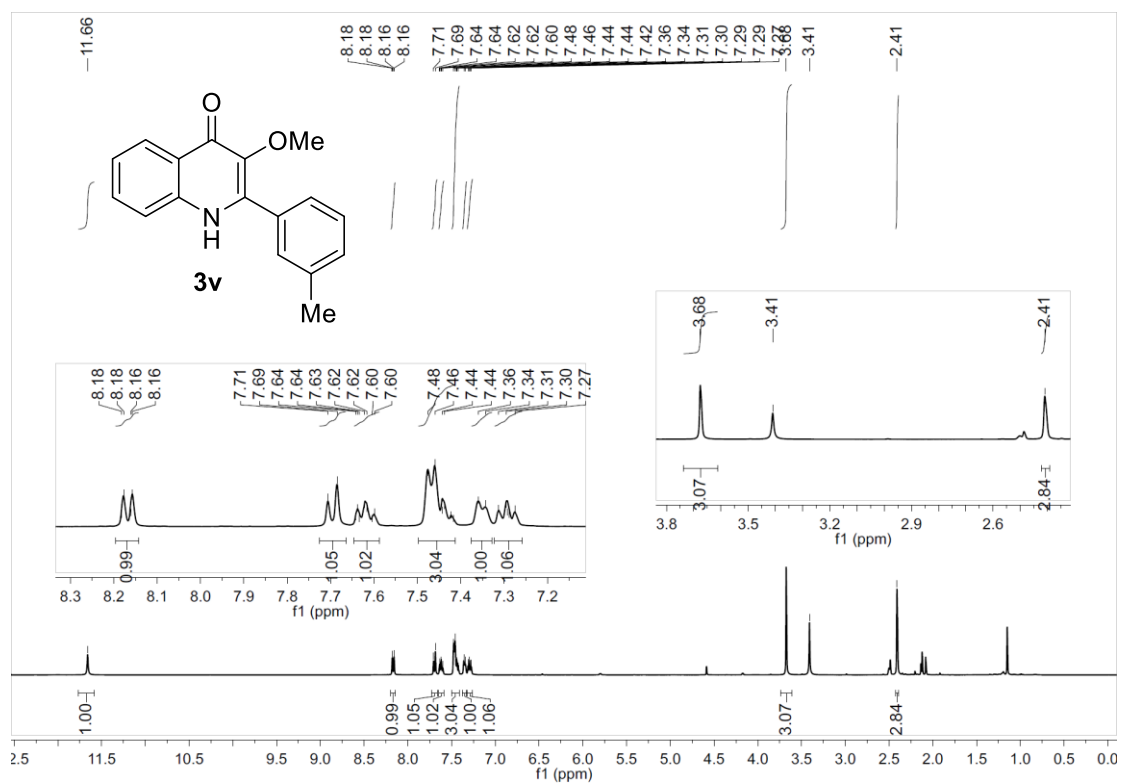
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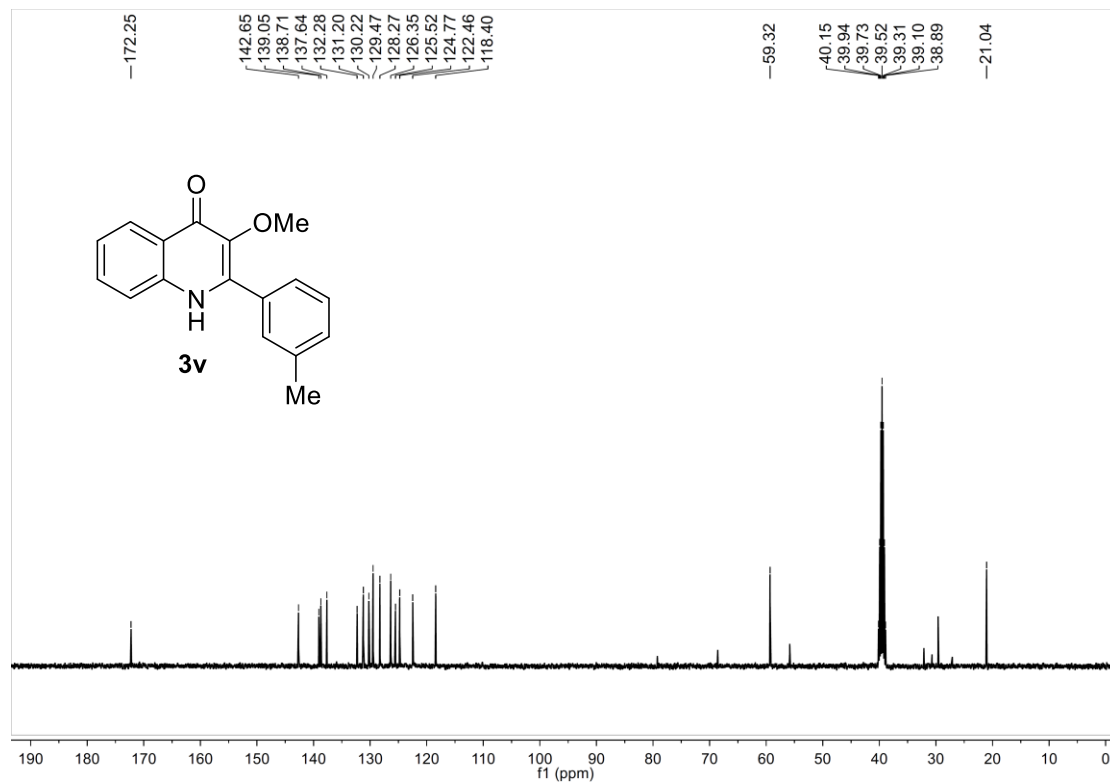
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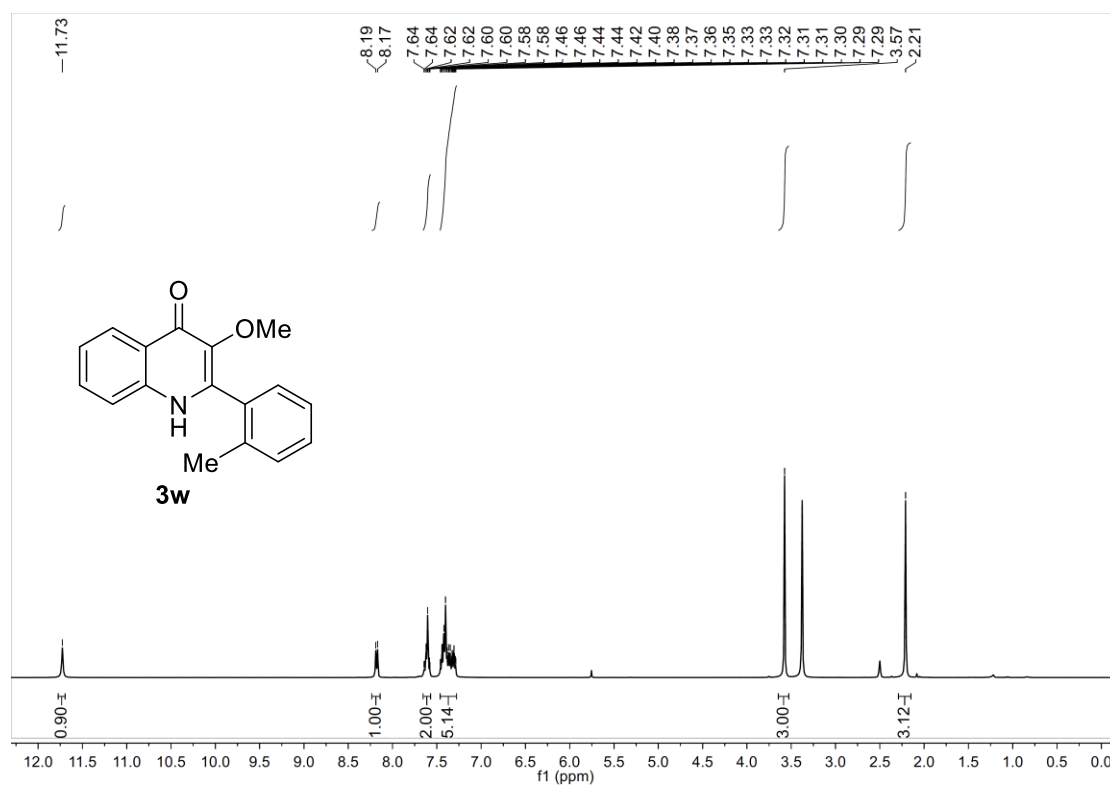
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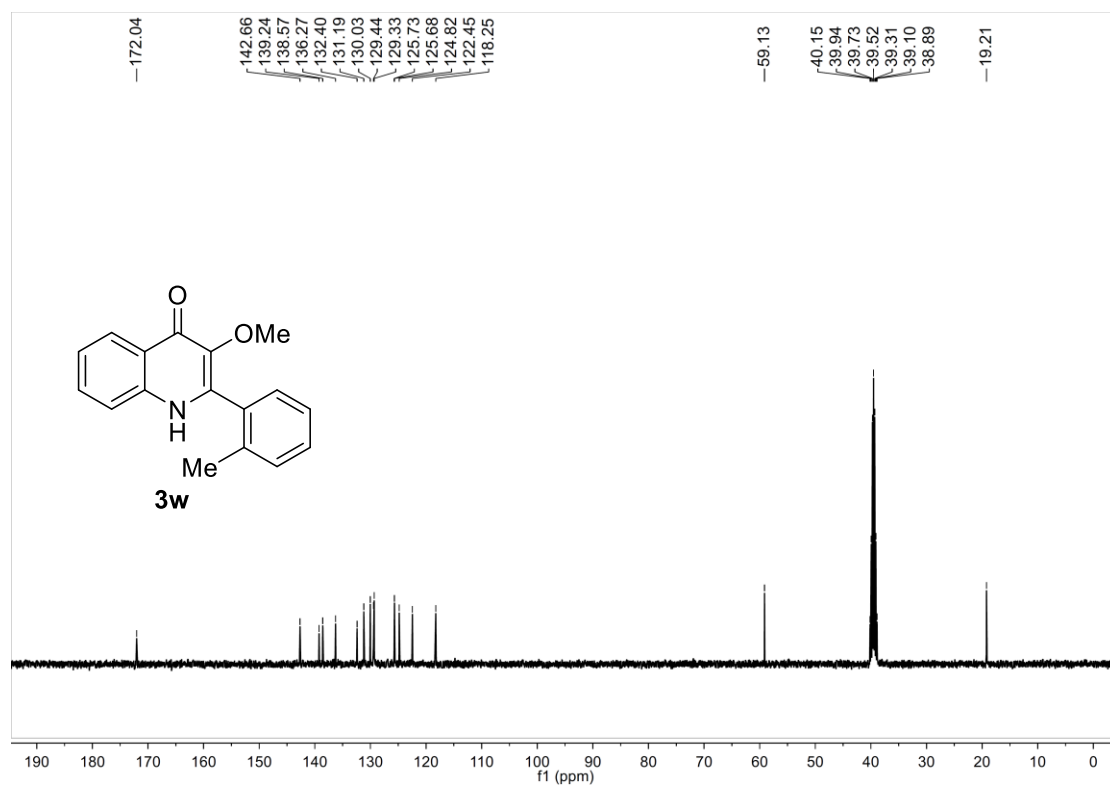
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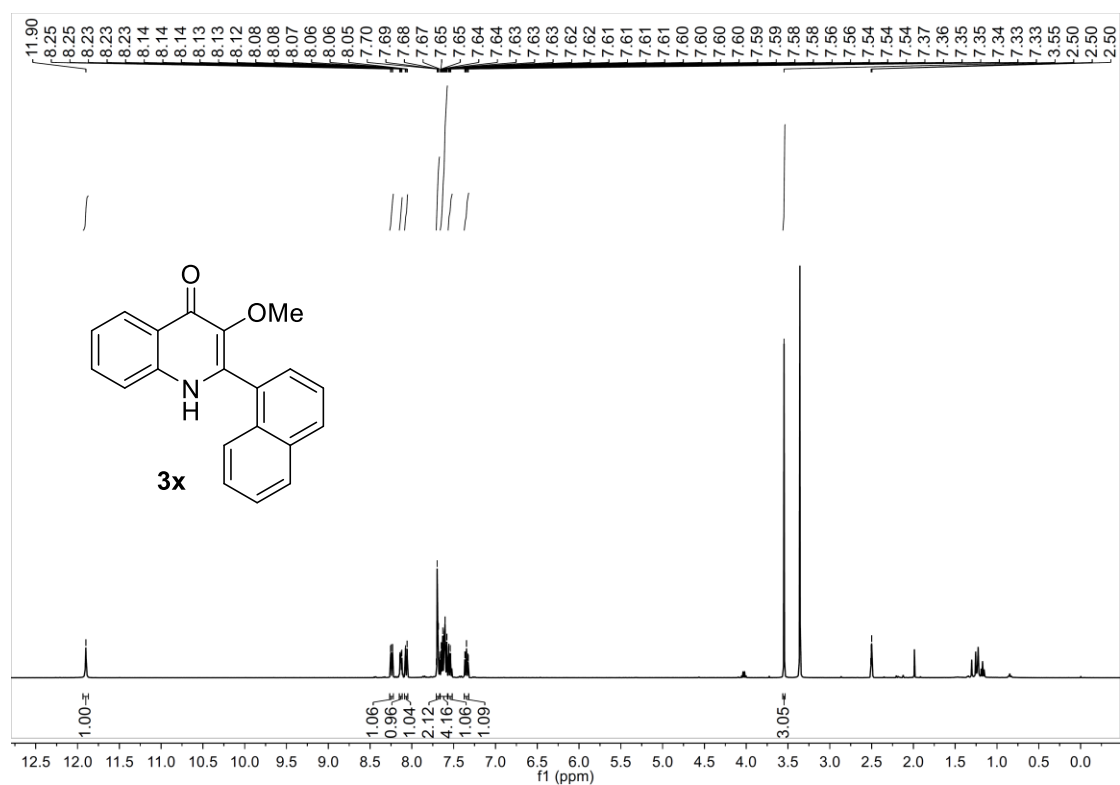
In DMSO-*d*₆



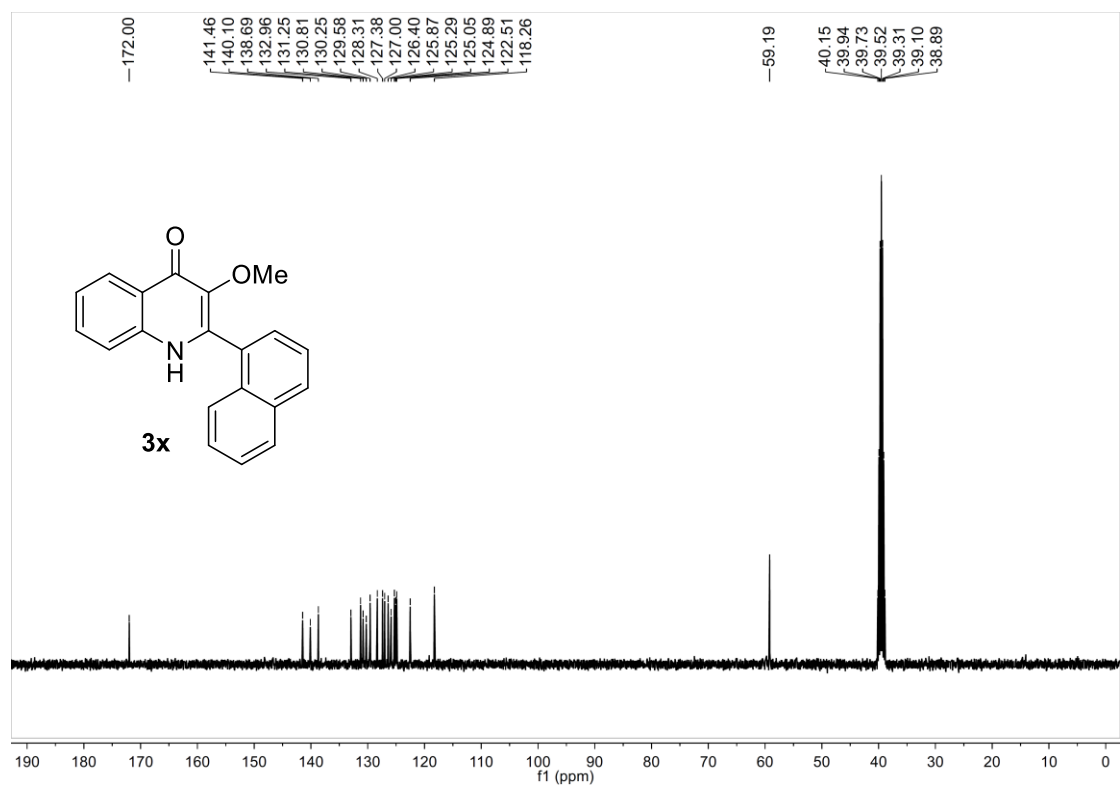
In DMSO-*d*₆



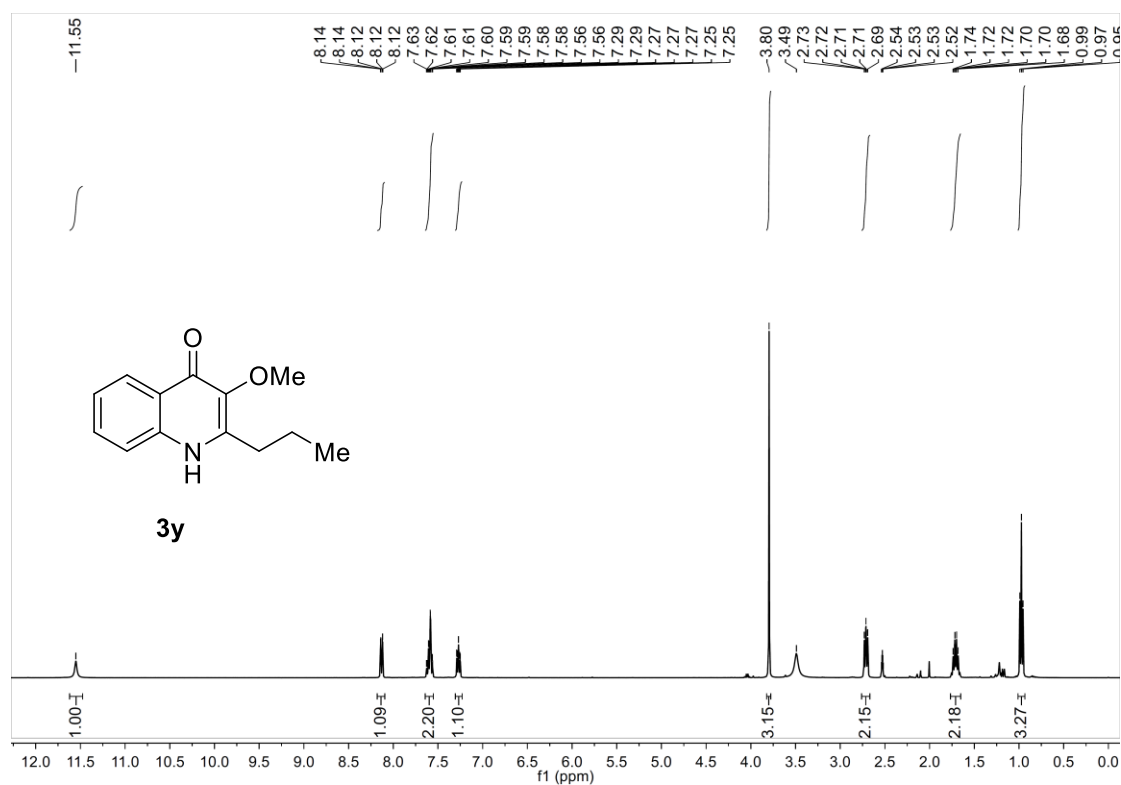
In DMSO-*d*₆



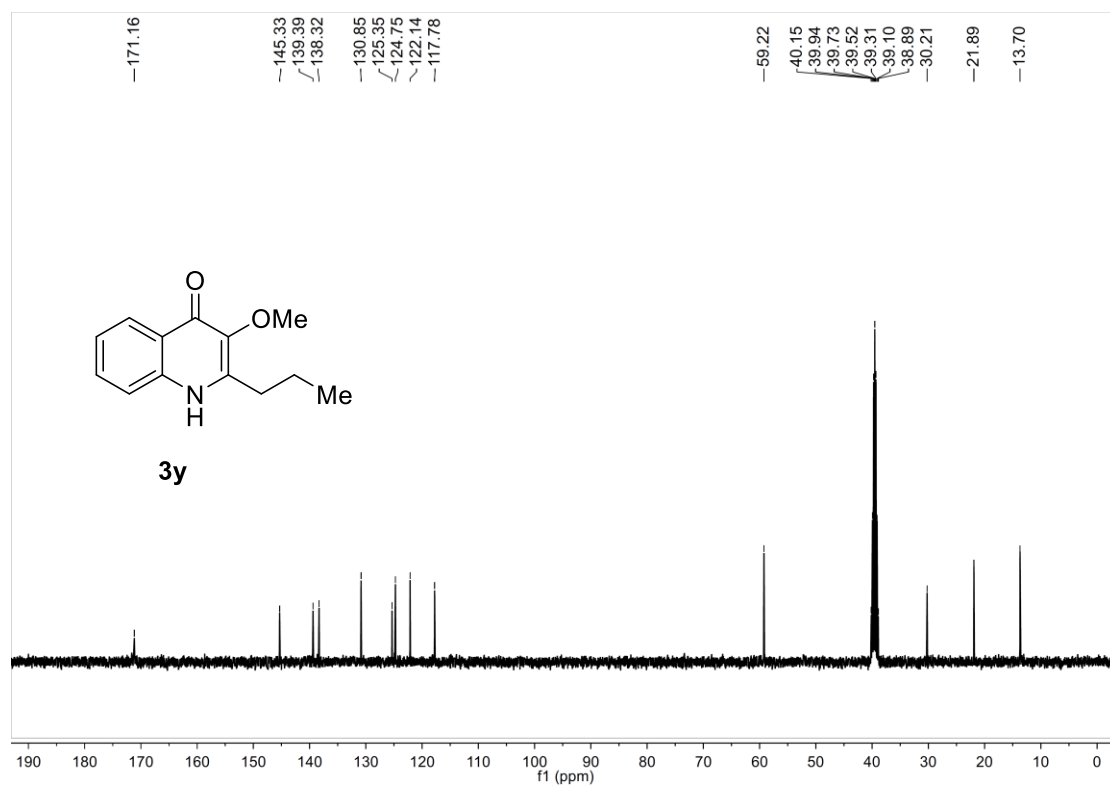
In DMSO-*d*₆



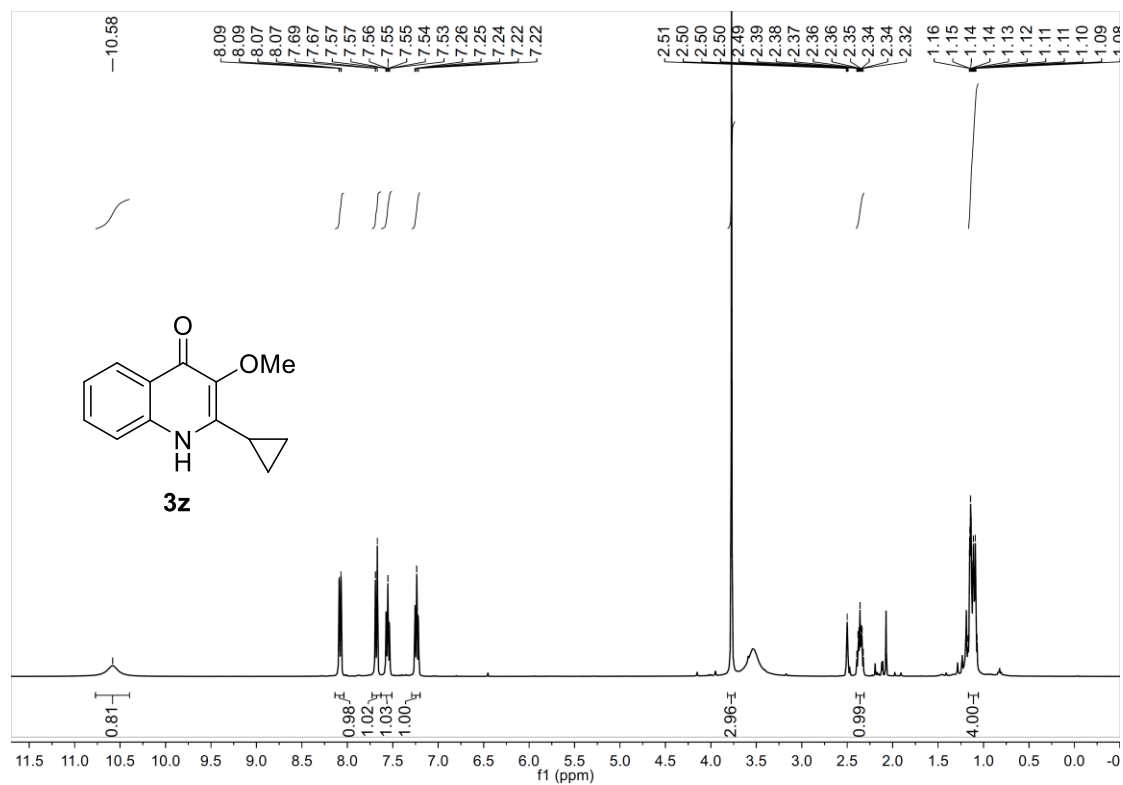
In DMSO-*d*₆



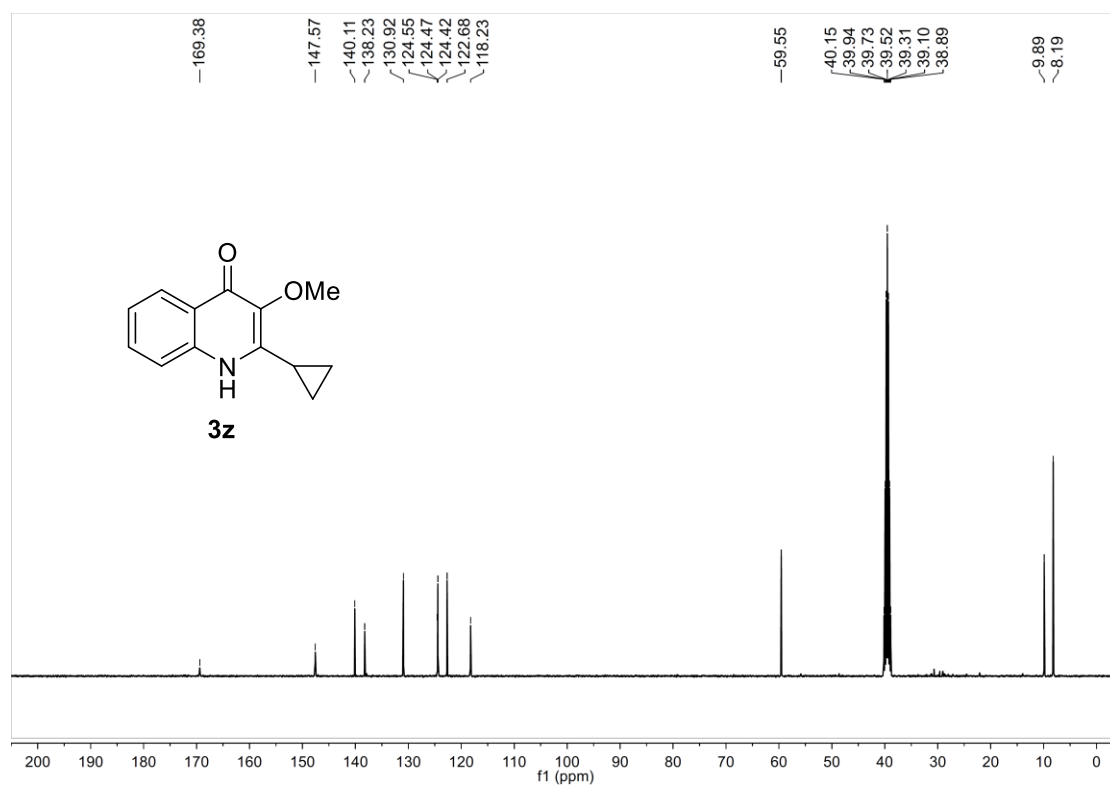
In DMSO-*d*₆



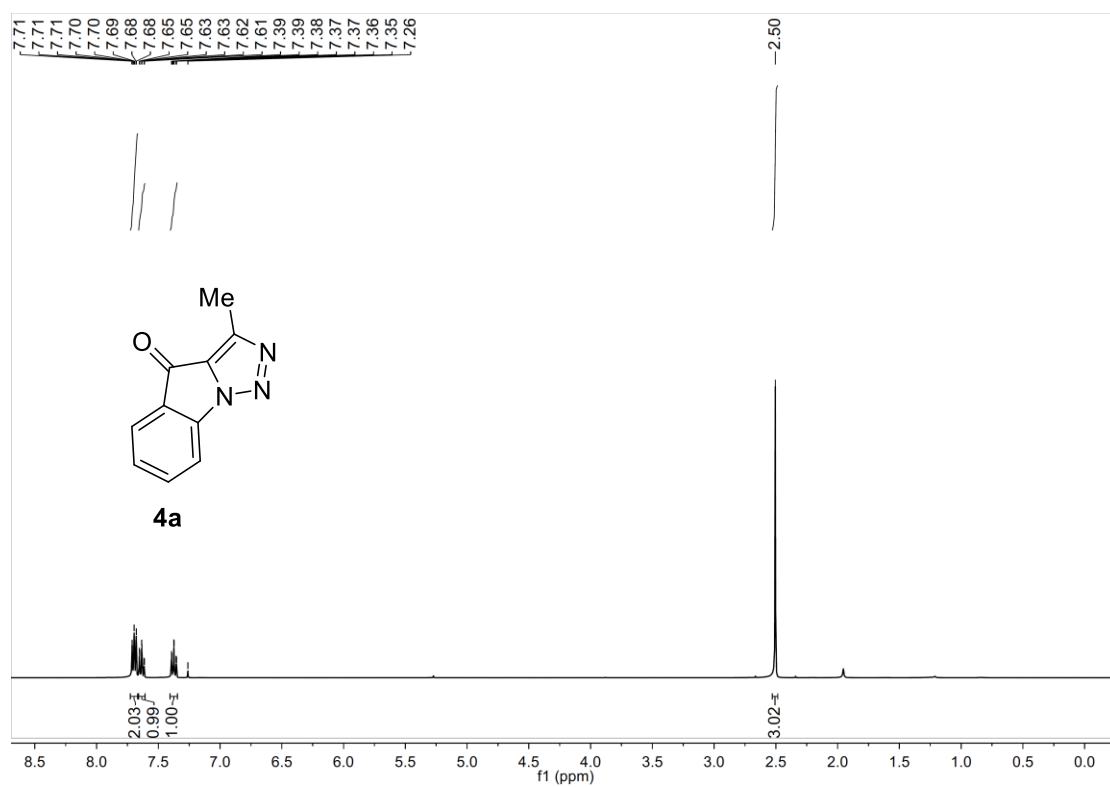
In DMSO-*d*₆



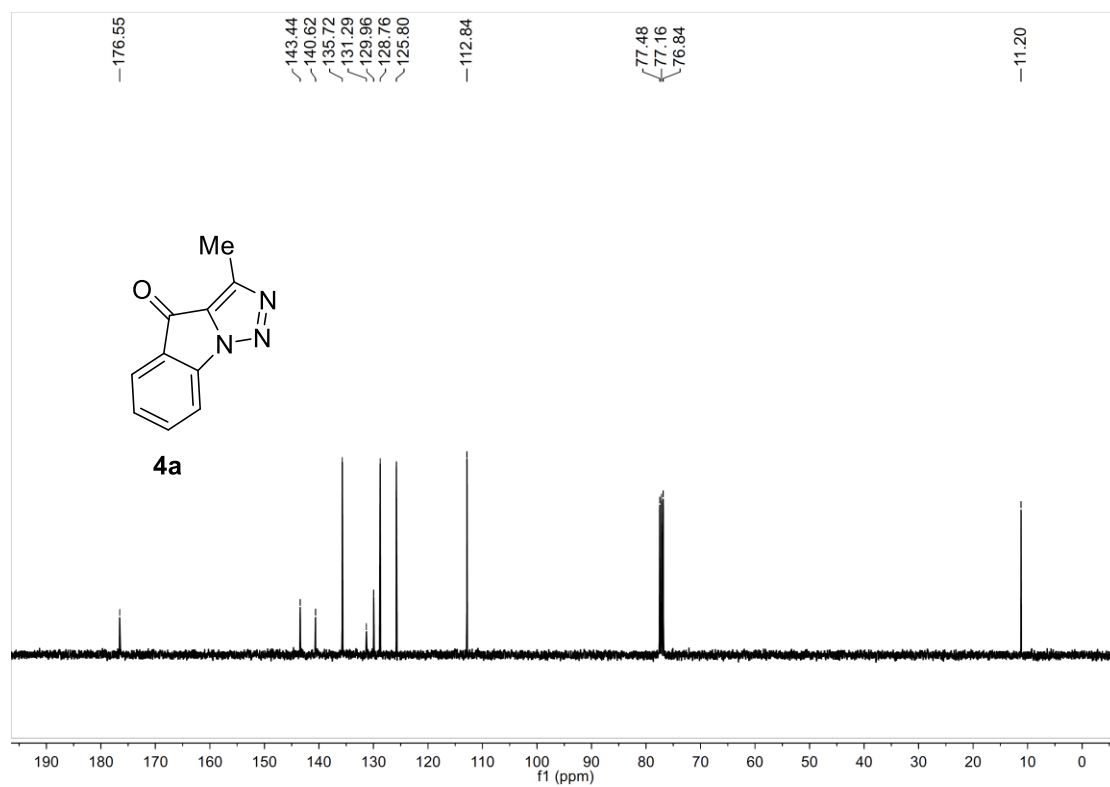
In DMSO-*d*₆



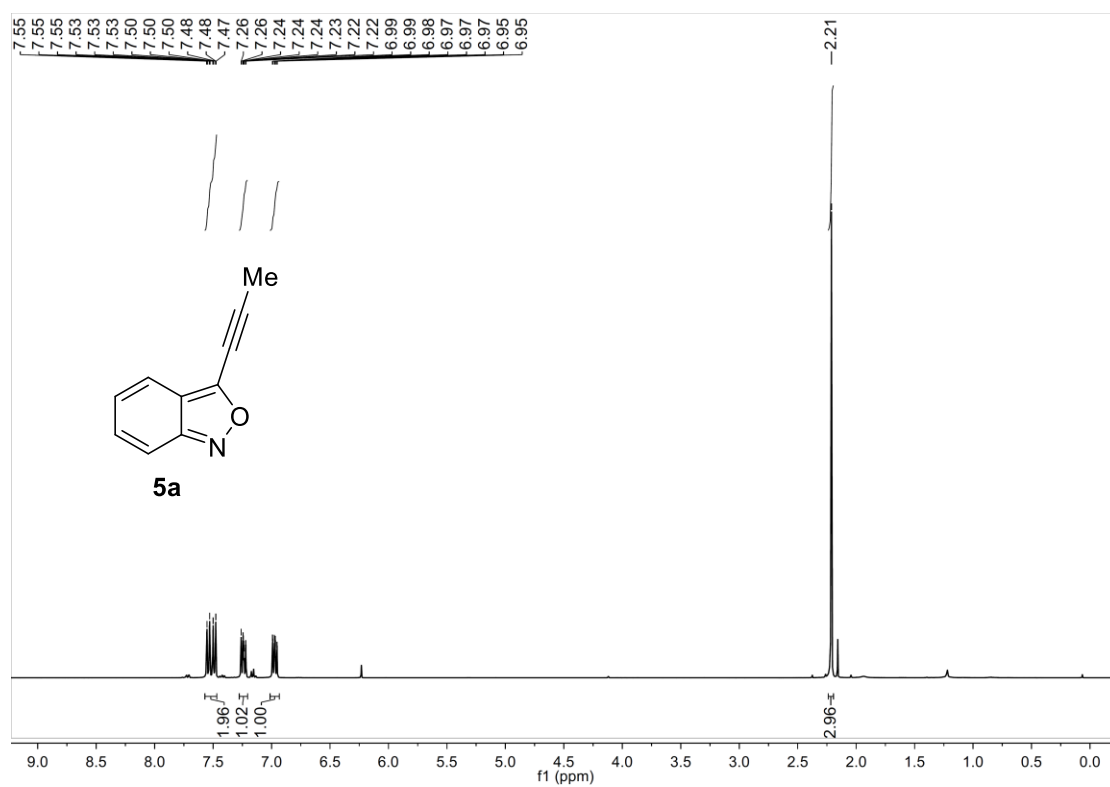
In CDCl₃



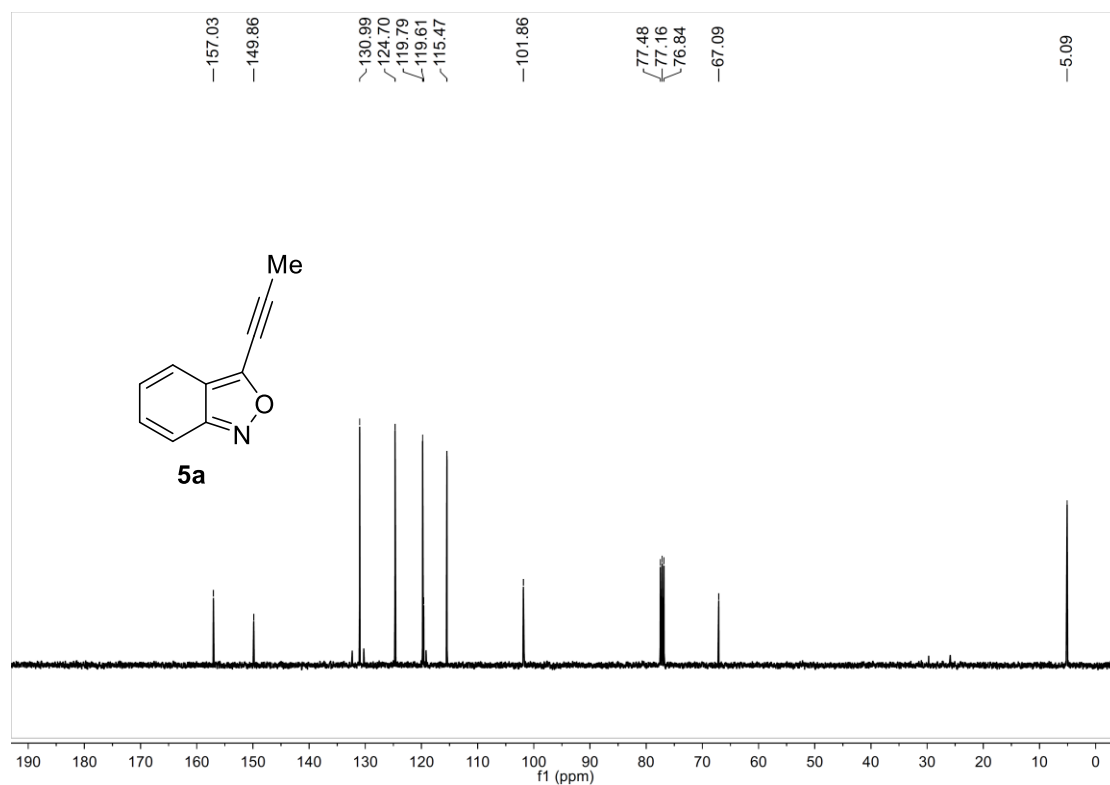
In CDCl₃



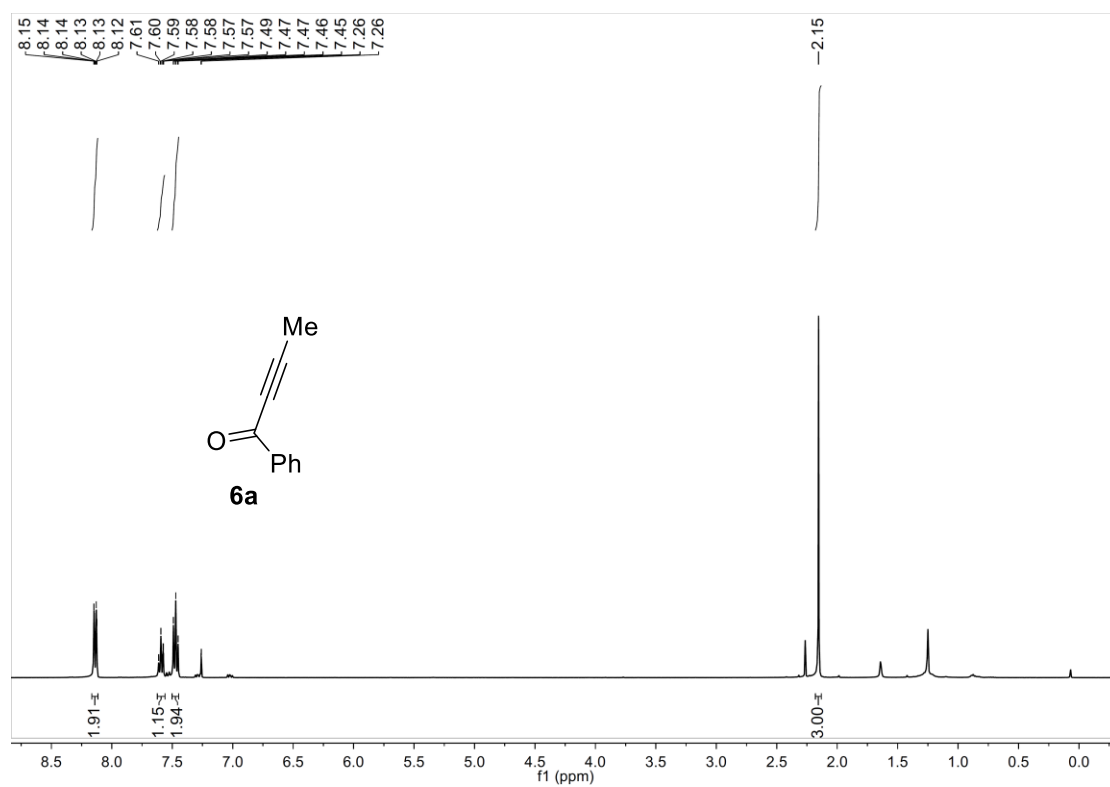
In CDCl₃



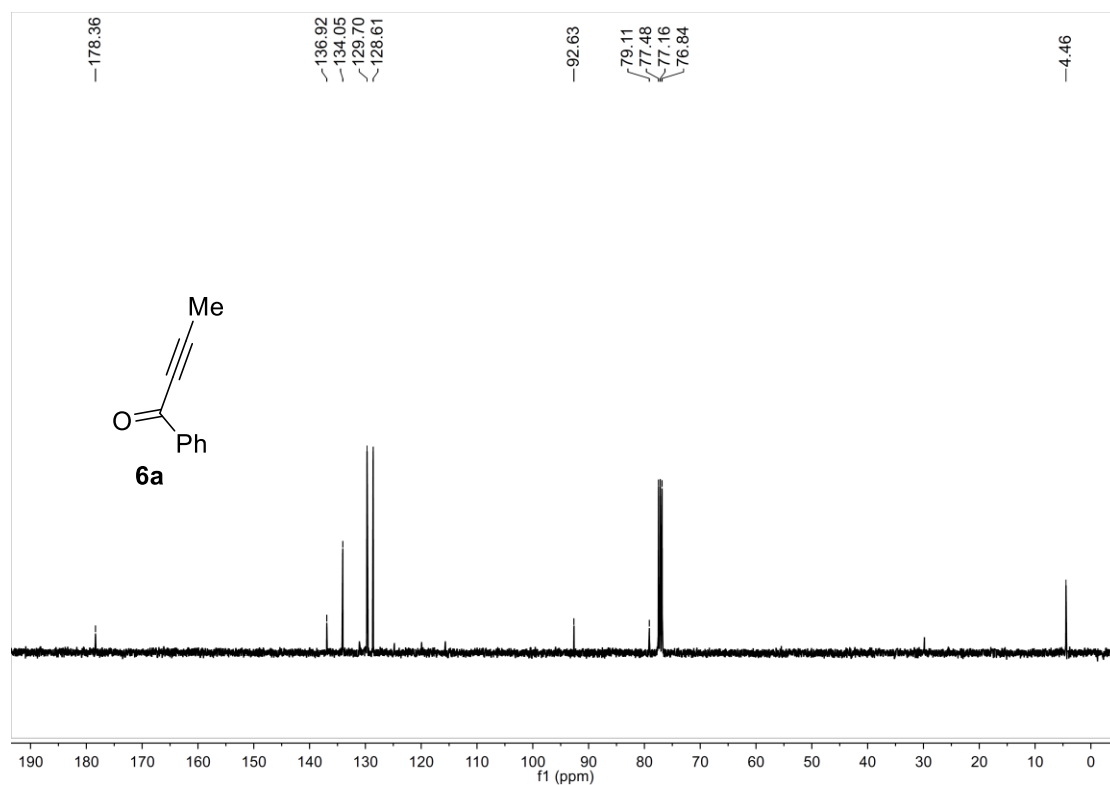
In CDCl₃



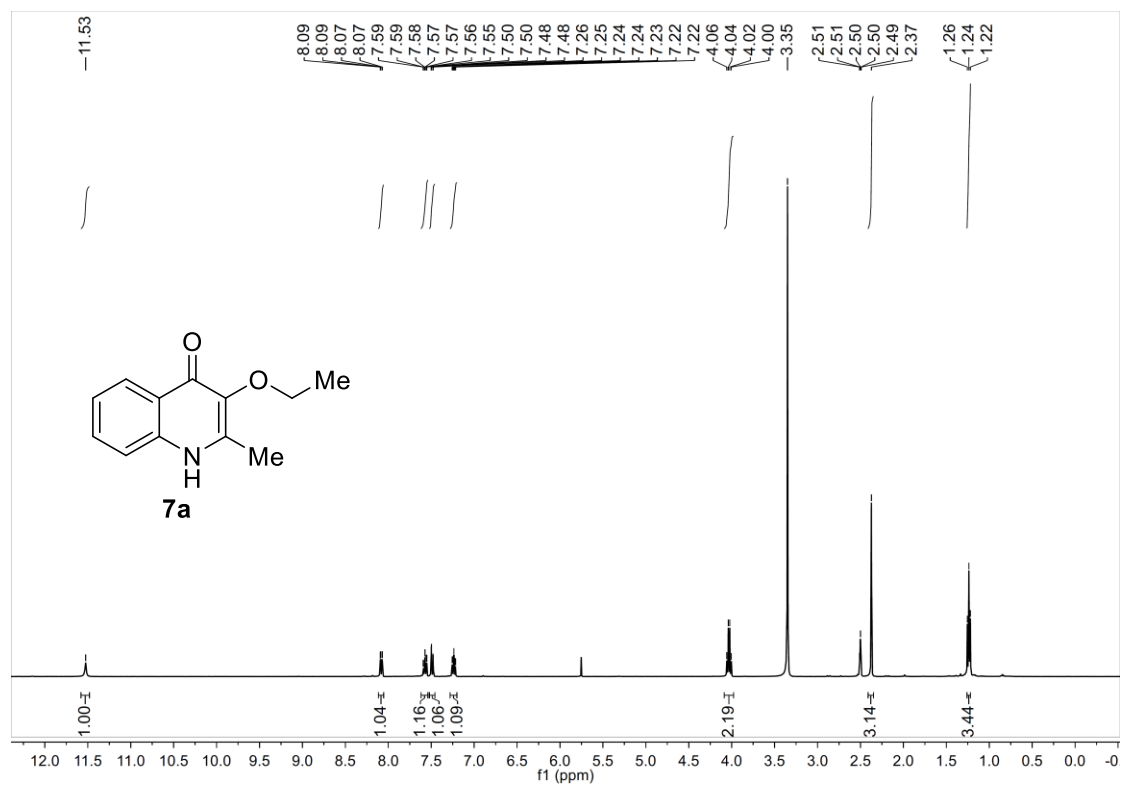
In CDCl₃



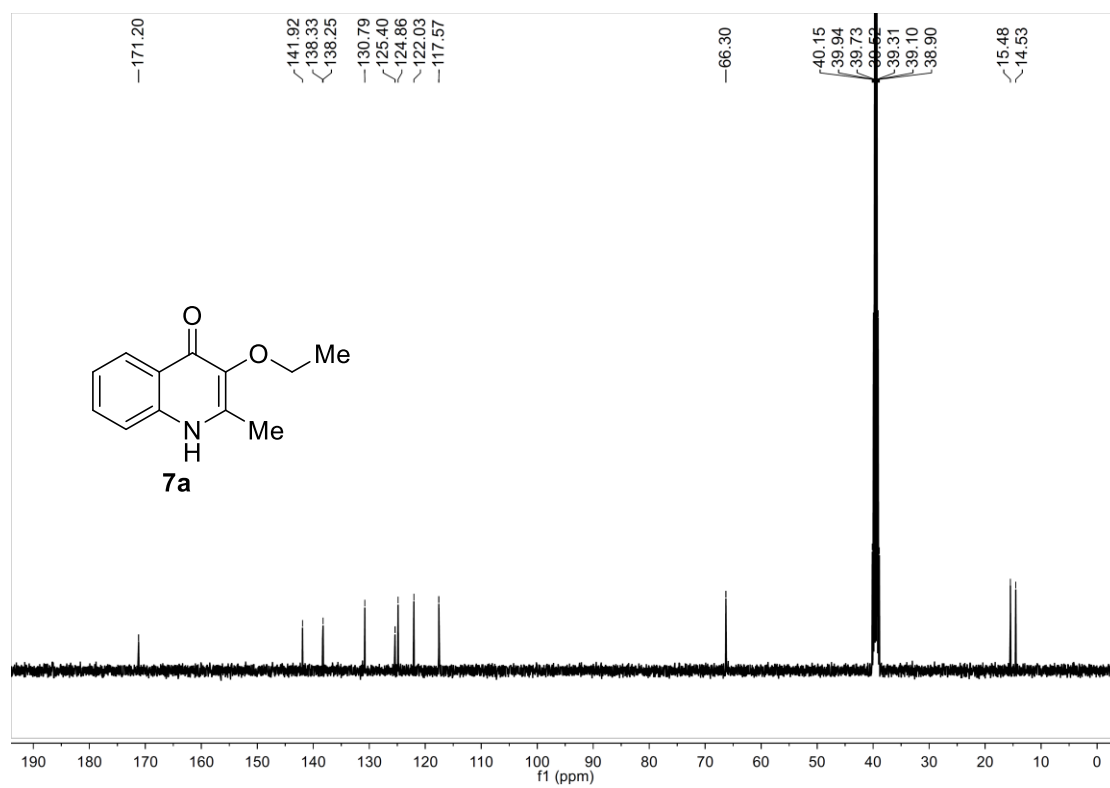
In CDCl₃



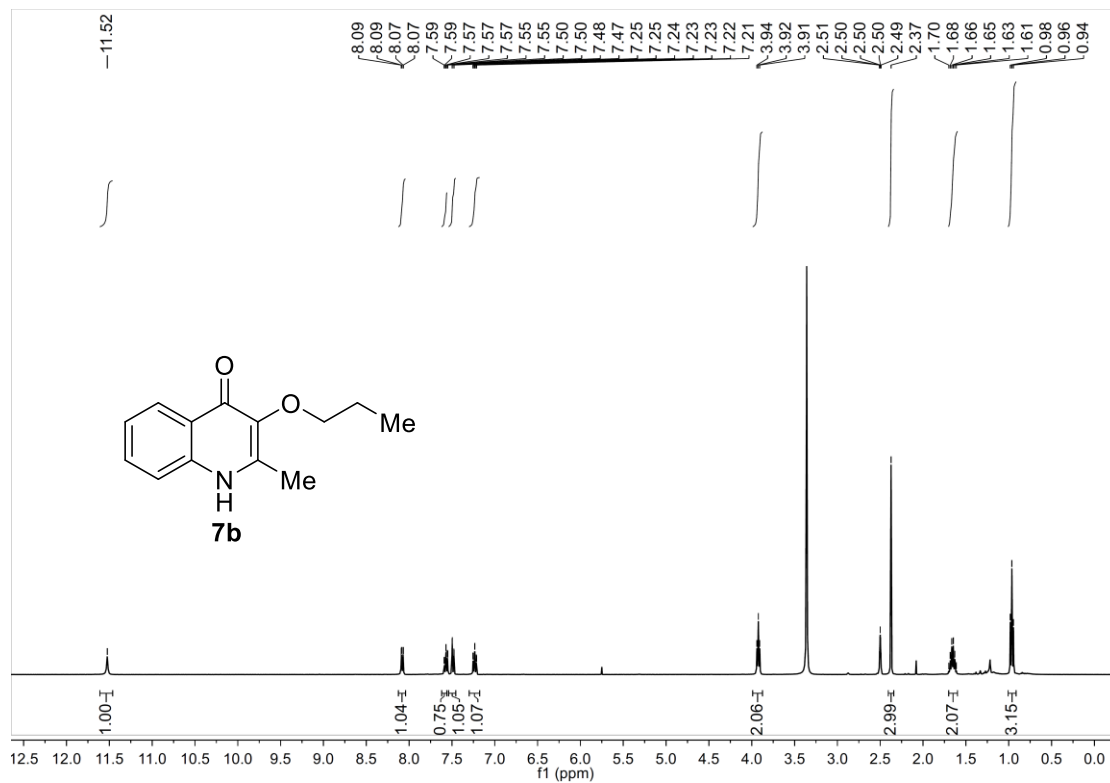
In DMSO-*d*₆



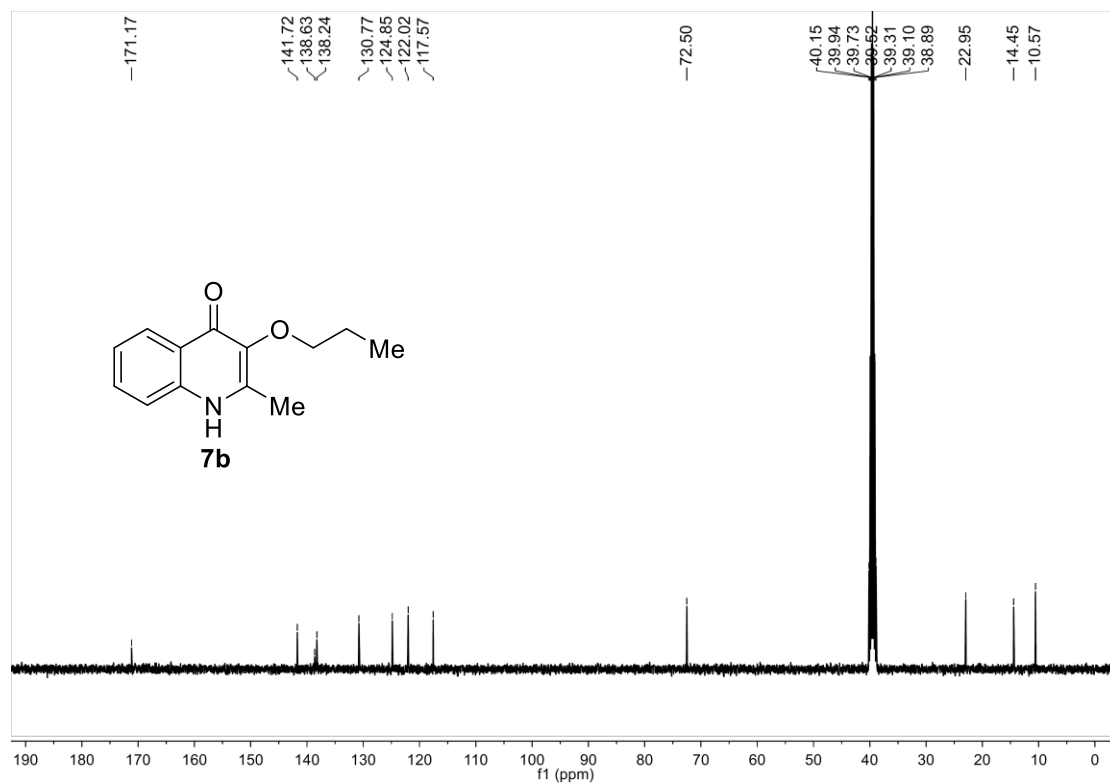
In DMSO-*d*₆



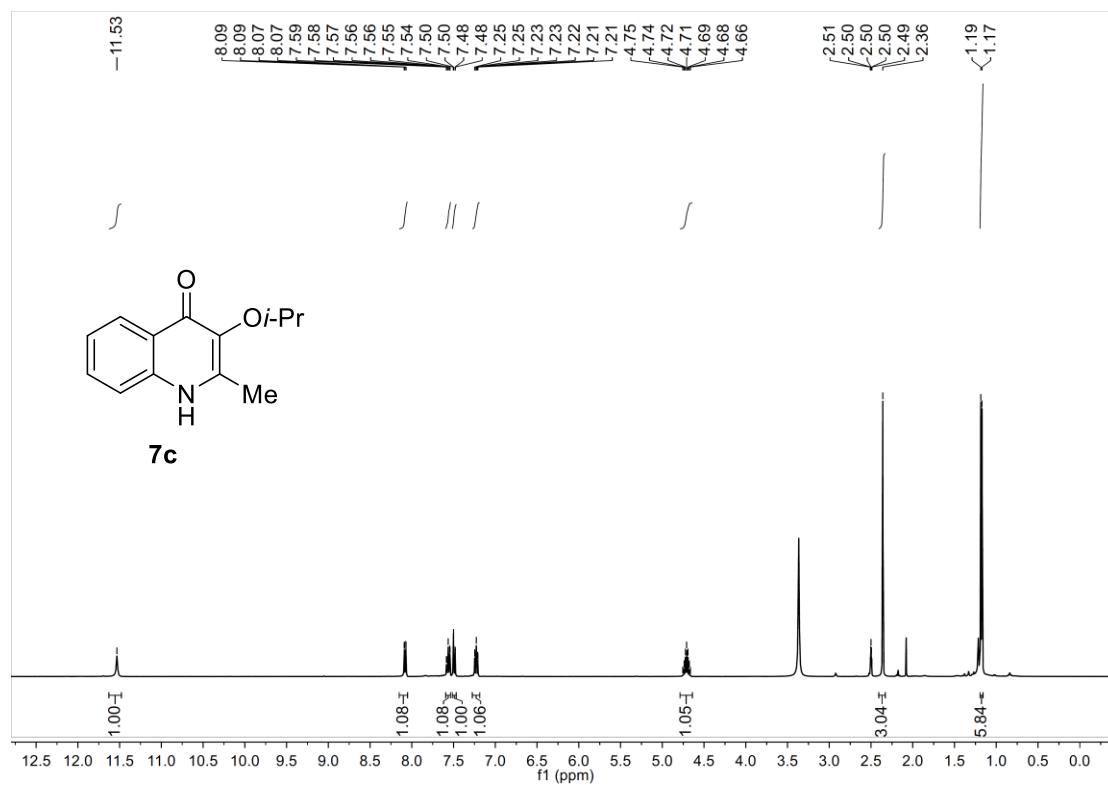
In DMSO-*d*₆



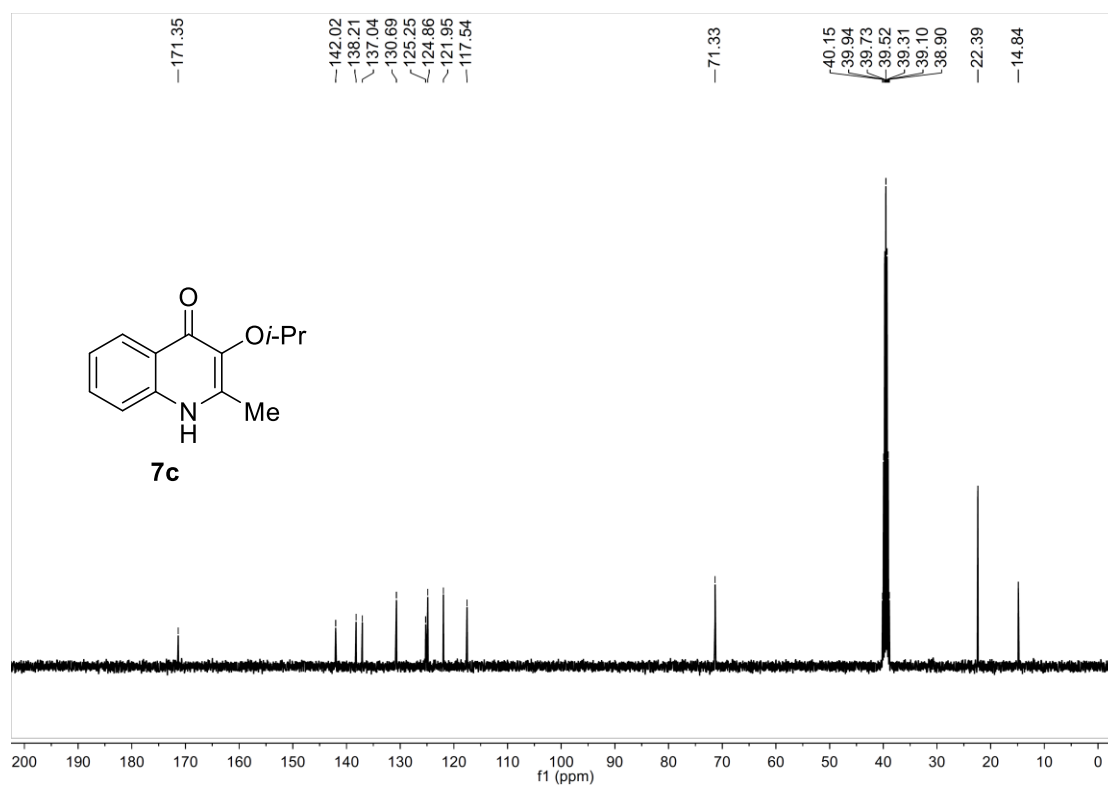
In DMSO-*d*₆



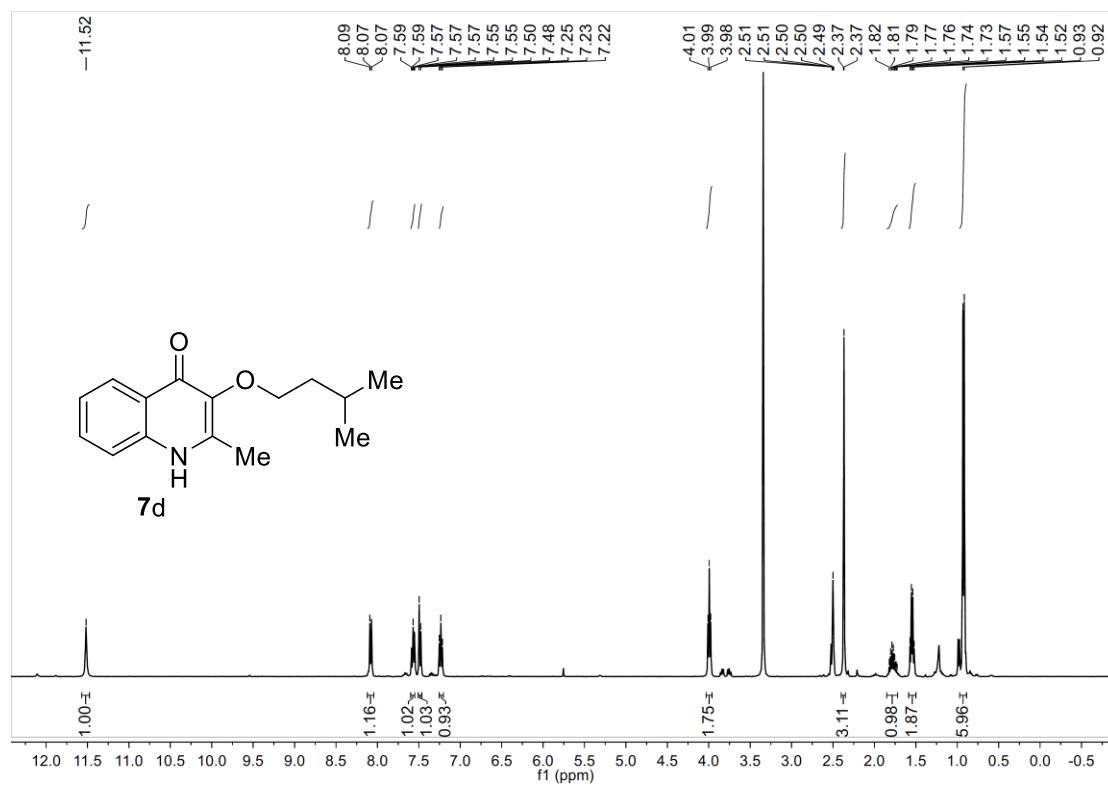
In DMSO-*d*₆



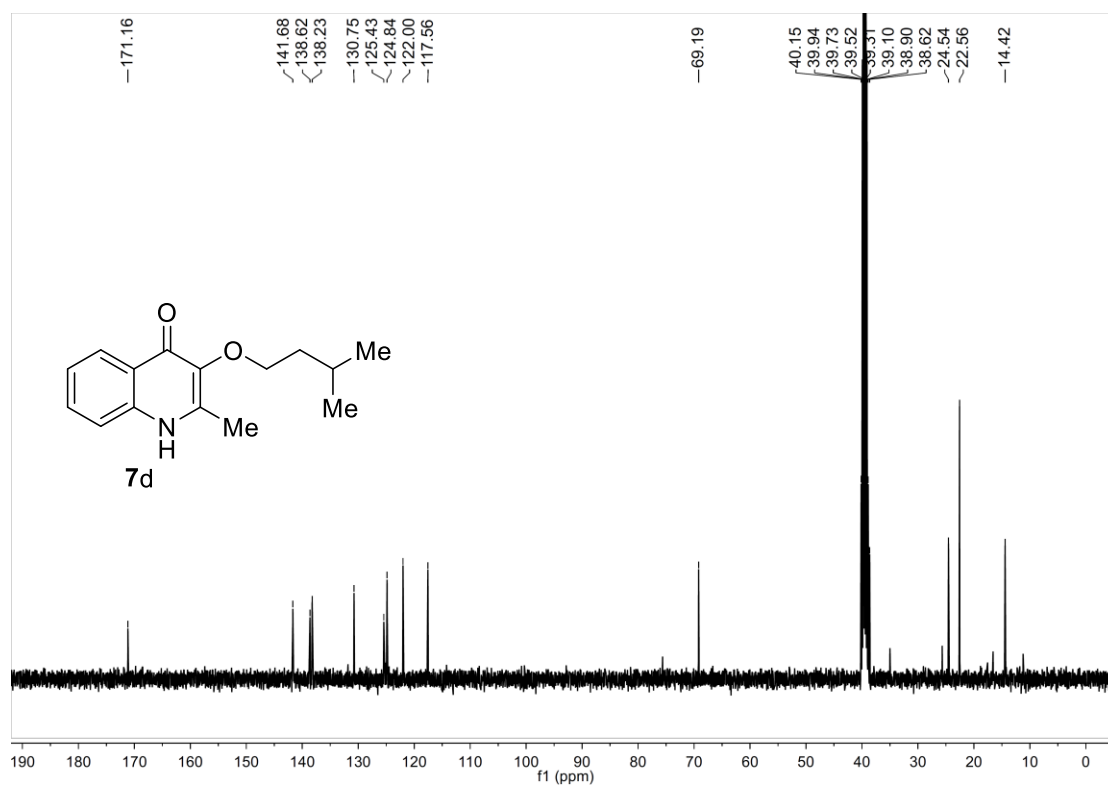
In DMSO-*d*₆



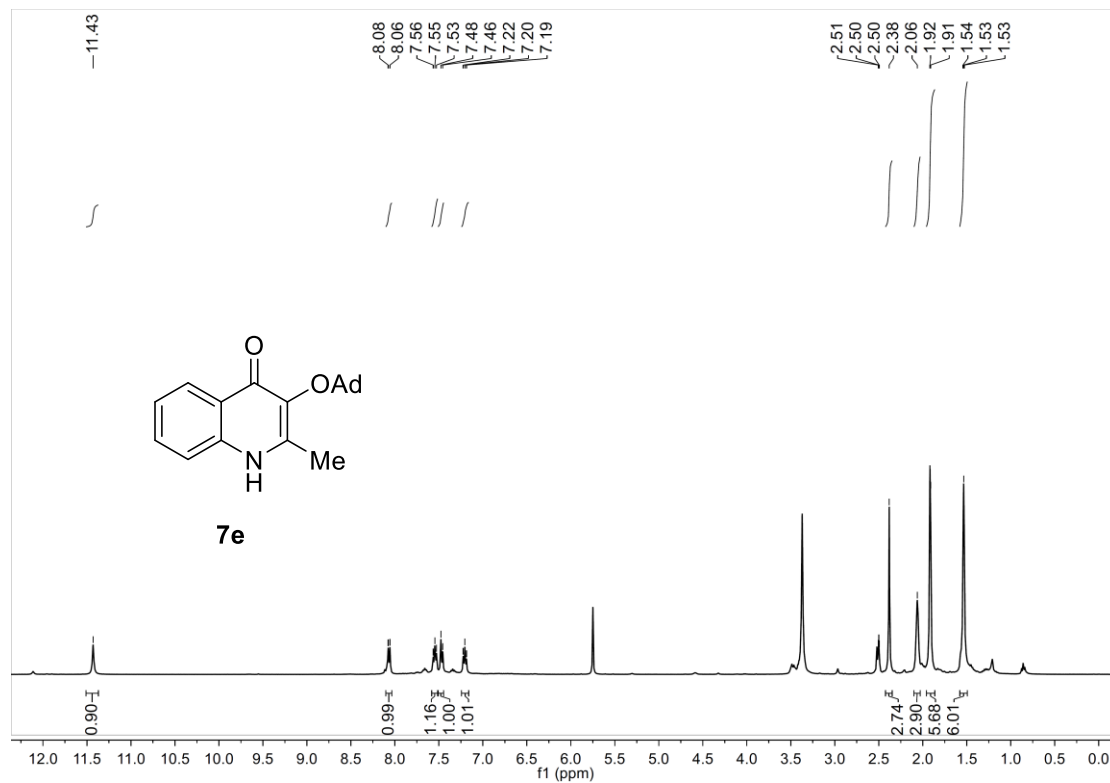
In DMSO-*d*₆



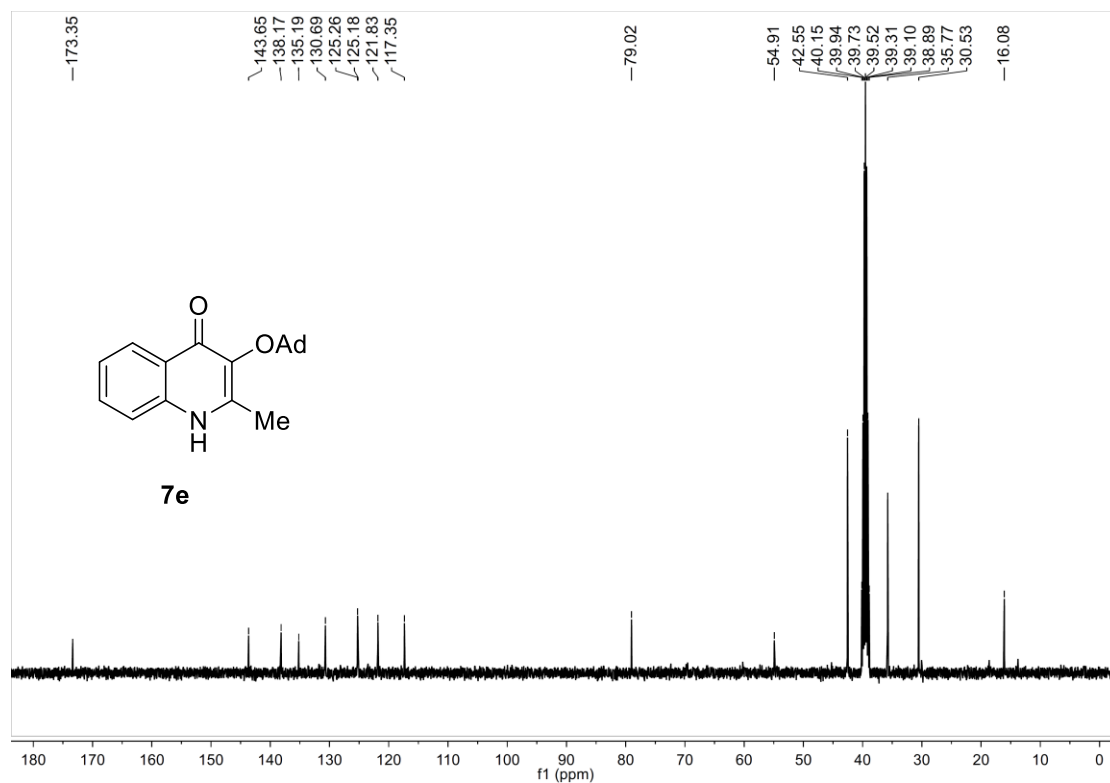
In DMSO-*d*₆



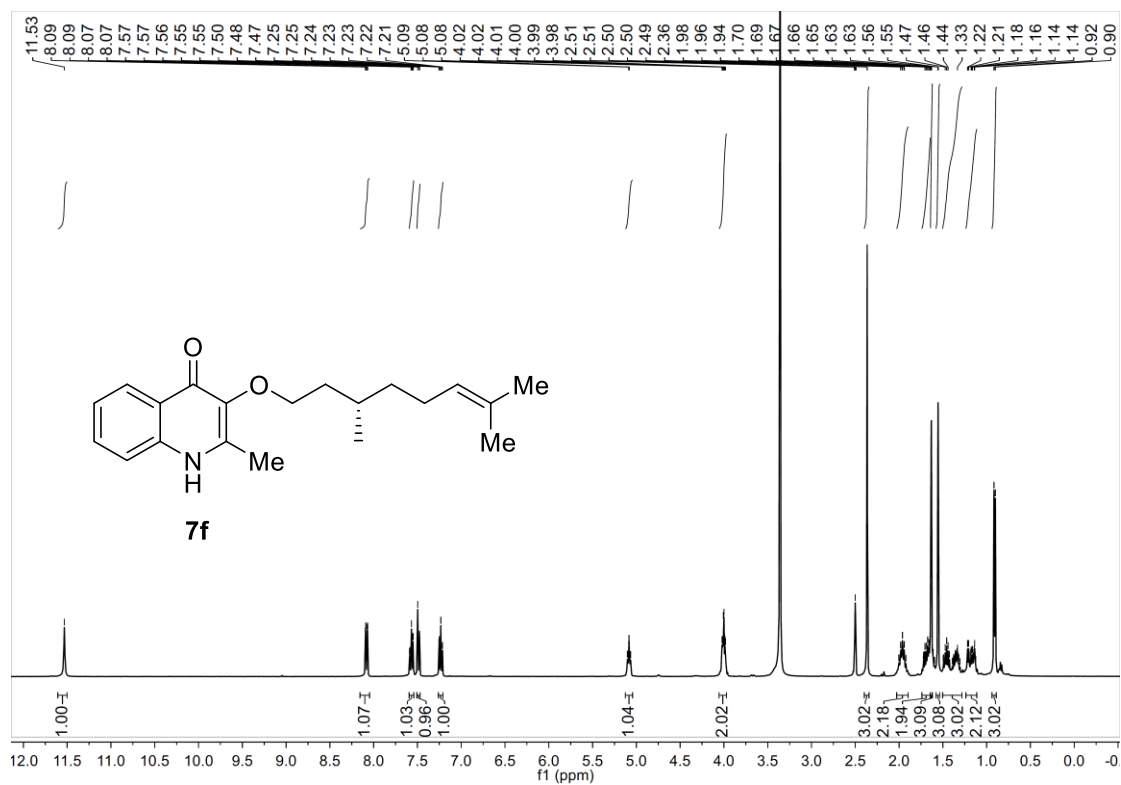
In DMSO-*d*₆



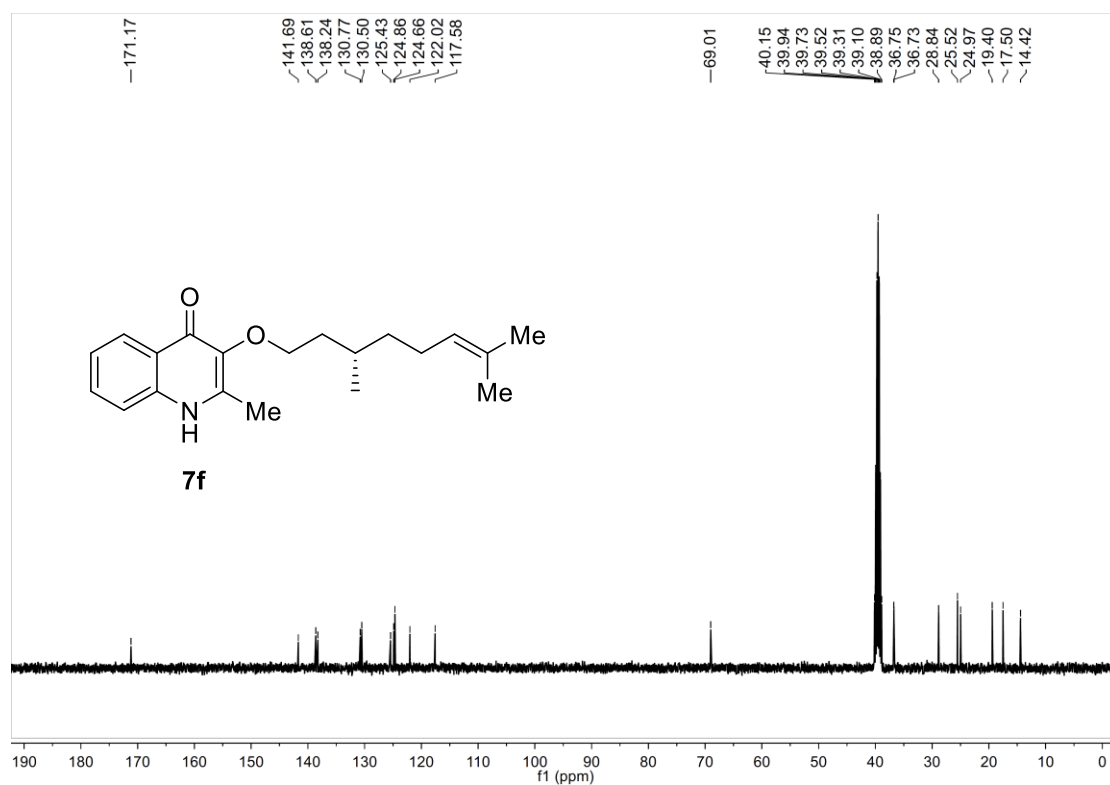
In DMSO-*d*₆



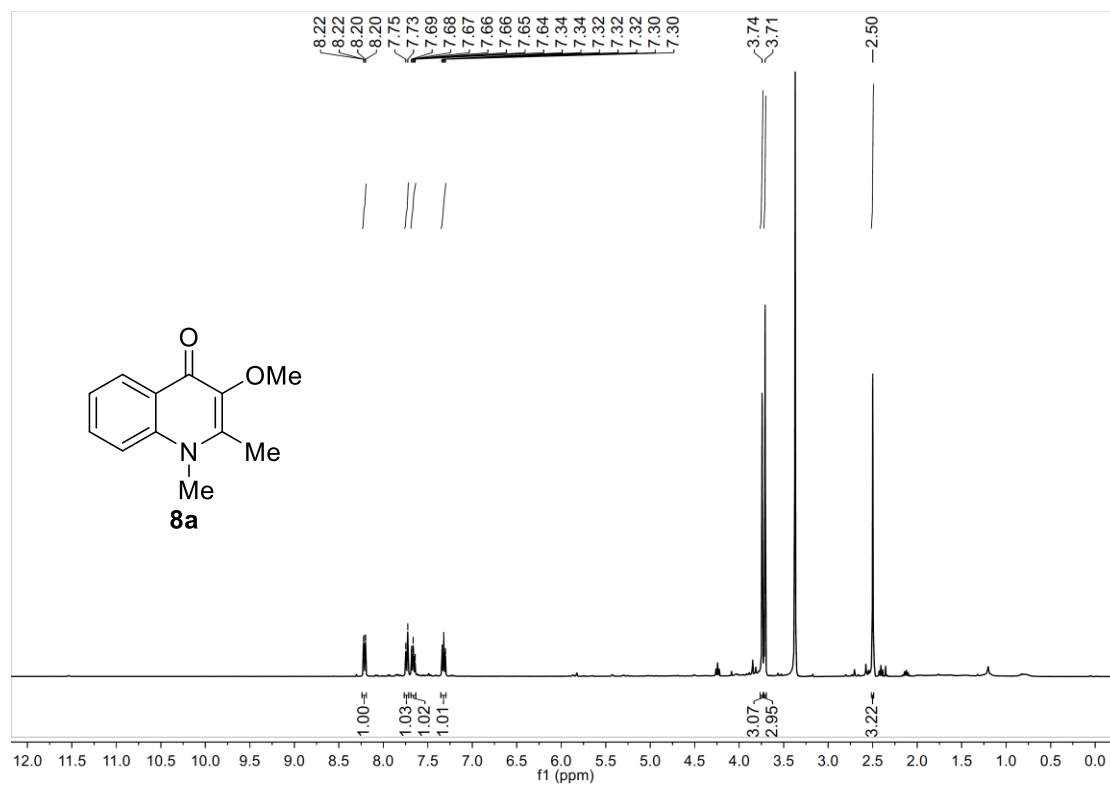
In DMSO-*d*₆



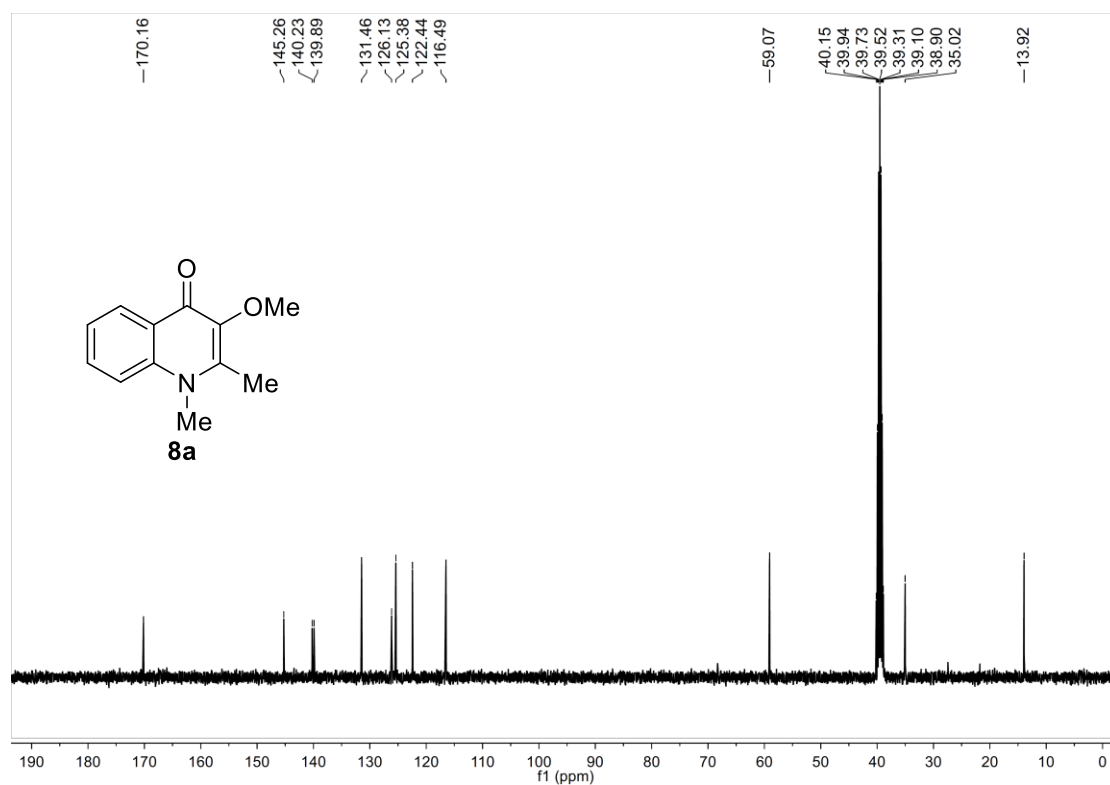
In DMSO-*d*₆



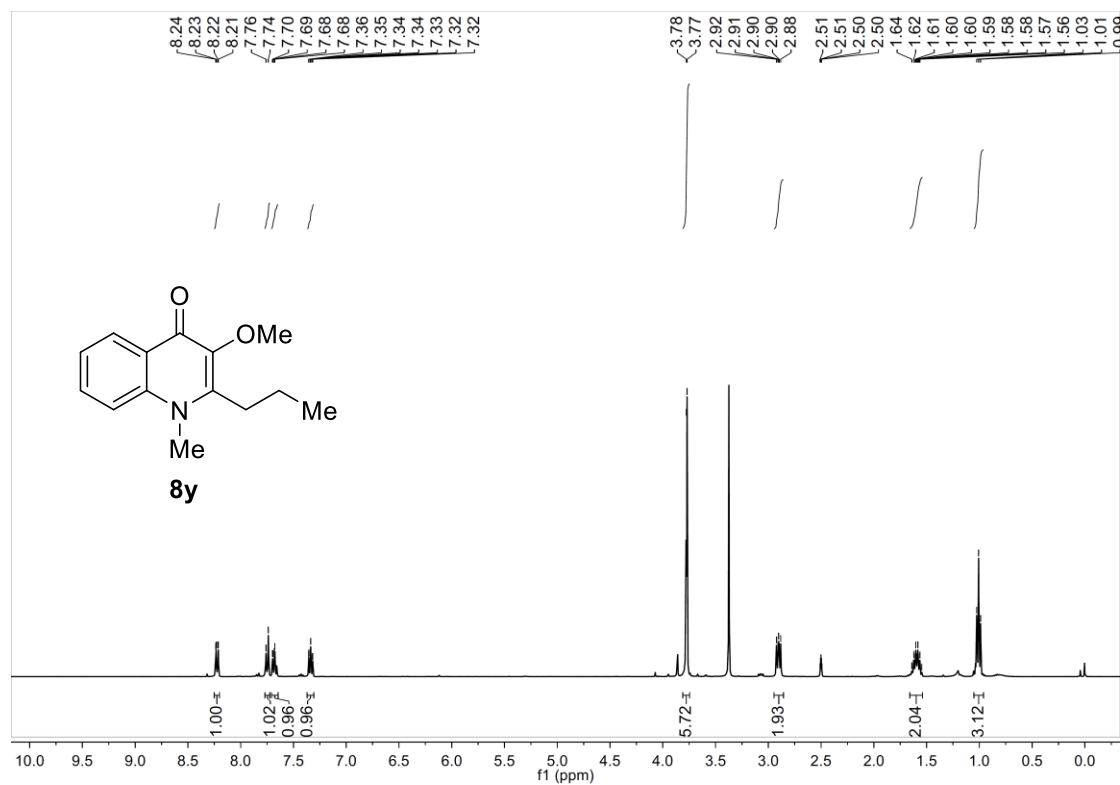
In DMSO-*d*₆



In DMSO-*d*₆



In DMSO- d_6



In DMSO- d_6

