

Supporting Information

Metal-free synthesis of nitriles from aldehydes using *N*-Boc-*O*-tosylhydroxylamine as nitrogen source

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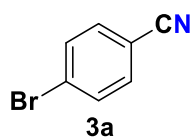
EXPERIMENTAL SECTION

1. General Information:

Unless otherwise stated, all the reactions were carried out using oven dried glassware under an open atmosphere in a round bottom flask with magnetic stirring bar at room temperature. Aldehydes were used as received without further purification. The aminating reagents were also prepared by following reported literature. TLC was carried out on pre-coated plates (Merck silica gel 60, F₂₅₄) and the spots were visualized with UV light or by charring the plates dipped in PMA or Ninhydrin or DNP solution. The compounds were purified by flash column chromatography using silica gel (100-200 mesh) with distilled solvents (EtOAc:Hexane) as mobile phase otherwise mentioned. ¹H and ¹³C NMR spectra were recorded at 400 or 800 MHz and 100 or 200 MHz instruments respectively in CDCl₃. Chemical shifts (δ) are given in ppm. The residual solvent signals were used as references (CDCl₃: δ H = 7.26 ppm). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, sext = sextet, sep = septet.

2. General Procedure for preparation of nitriles from aldehydes: To a stirring solution of aldehydes (0.5 mmol, 1.0 equiv.) in TFE (0.5 mL) at room temperature in an open round bottom flask, aminating agent (0.75 mmol, 1.5 equiv.) was added. The reaction mixture was stirred at the room temperature for 2-3h. After formation of aldoxime (monitor by TLC), Cs₂CO₃ (1.5 equiv.) was added and reaction mixture was further stirred at room temperature for given time. After complete consumption of aldoxime, the reaction mixture was diluted with ethyl acetate (15 mL) and the organic layer was washed with brine solution (5 mL) and dried over anhydrous Na₂SO₄. The organic layer was evaporated under reduced pressure and crude obtained was purified through silica gel column chromatography using ethyl acetate/hexane as eluent.

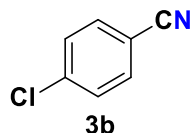
3. Characterization data of the products:



4-bromobenzonitrile (3a): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (84 mg, 92% yield; mp 107–110 °C) whose spectral data were consistent with the literature values.¹

¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H).

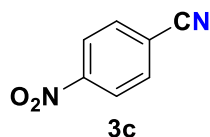
¹³C NMR (200 MHz, CDCl₃) δ 133.16, 132.33, 127.70, 117.80, 110.90.



4-chlorobenzonitrile (3b): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (58 mg, 84% yield; mp 86–90 °C) whose spectral data were consistent with the literature values.¹

¹H NMR (400 MHz, CDCl₃) δ 7.59 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H).

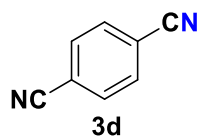
¹³C NMR (100 MHz, CDCl₃) δ 139.65, 133.48, 129.80, 118.05, 110.90.



4-nitrobenzonitrile (3c): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1, v/v) afforded the title compound as yellow solid (73 mg, 98% yield; mp 144–147 °C) whose spectral data were consistent with the literature values.²

¹H NMR (400 MHz, CDCl₃) δ 8.36 (d, *J* = 8.6 Hz, 2H), 7.89 (d, *J* = 8.6 Hz, 2H).

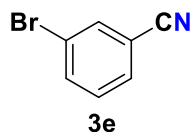
¹³C NMR (100 MHz, CDCl₃) δ 150.13, 133.60, 124.40, 118.43, 116.92.



Terephthalonitrile (3d): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (61 mg, 95% yield; mp 220–222 °C) whose spectral data were consistent with the literature values.²

¹H NMR (400 MHz, CDCl₃) δ 7.79 (s, 4H).

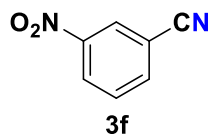
¹³C NMR (100 MHz, CDCl₃) δ 132.91, 117.13, 116.83.



3-bromobenzonitrile (3e): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (79 mg, 87% yield; mp 35–38 °C) whose spectral data were consistent with the literature values.¹

¹H NMR (400 MHz, CDCl₃) δ 7.80 (s, 1H), 7.75 (d, *J* = 8.2 Hz, 1H), 7.61 (d, *J* = 7.7 Hz, 1H), 7.37 (t, *J* = 7.9 Hz, 1H).

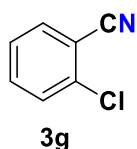
¹³C NMR (200 MHz, CDCl₃) δ 135.71, 134.22, 130.36, 130.33, 122.41, 116.88, 113.67.



3-nitrobenzonitrile (3f): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1, v/v) afforded the title compound as yellow solid (71 mg, 96% yield; mp 112–114 °C) whose spectral data were consistent with the literature values.³

¹H NMR (400 MHz, CDCl₃) δ 8.52 (s, 1H), 8.47 (d, *J* = 8.3 Hz, 1H), 8.01 (d, *J* = 7.7 Hz, 1H), 7.75 (t, *J* = 8.0 Hz, 1H).

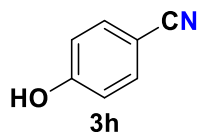
¹³C NMR (100 MHz, CDCl₃) δ 148.30, 137.71, 130.78, 127.61, 127.28, 116.62, 114.17.



2-chlorobenzonitrile (3g): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 20:1, v/v) afforded the title compound as white solid (63 mg, 91% yield; mp 36–40 °C) whose spectral data was consistent with the literature values.⁴

¹H NMR (400 MHz, CDCl₃) δ 7.67 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.59 – 7.47 (m, 2H), 7.42 – 7.33 (m, 1H).

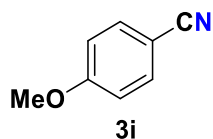
¹³C NMR (100 MHz, CDCl₃) δ 136.95, 134.11, 133.99, 130.15, 127.26, 116.05, 113.48.



4-hydroxybenzonitrile (3h): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 4:1, v/v) afforded the title compound as brown solid (41 mg, 69% yield; mp 110–112 °C) whose spectral data were consistent with the literature values.²

¹H NMR (400 MHz, CDCl₃) δ 7.55 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 2H).

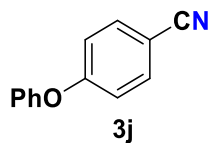
¹³C NMR (100 MHz, CDCl₃) δ 160.28, 134.46, 119.38, 116.59, 103.23.



4-methoxybenzonitrile (3i): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (52 mg, 78% yield; mp 55–58 °C) whose spectral data were consistent with the literature values.¹

¹H NMR (400 MHz, CDCl₃) δ 7.56 – 7.52 (m, 2H), 6.93 – 6.90 (m, 2H), 3.83 (s, 3H).

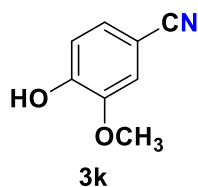
^{13}C NMR (100 MHz, CDCl_3) δ 162.86, 133.94, 119.23, 114.77, 103.86, 55.55.



4-phenoxybenzonitrile (3j): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 95:5, v/v) afforded the title compound as colorless oil (80 mg, 82% yield) whose spectral data were consistent with the literature values.³

^1H NMR (800 MHz, CDCl_3) δ 7.60 (d, J = 8.0 Hz, 2H), 7.43 (t, J = 8.0 Hz, 2H), 7.25 (t, J = 8.0 Hz, 1H), 7.08 (d, J = 8.0 Hz, 2H), 7.01 (d, J = 8.0 Hz, 2H).

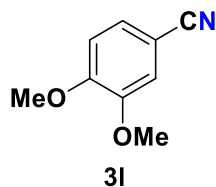
^{13}C NMR (200 MHz, CDCl_3) δ 161.44, 154.56, 133.93, 130.06, 124.97, 120.22, 118.66, 117.69, 105.54.



4-hydroxy-3-methoxybenzonitrile (3k): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (50 mg, 67% yield; mp 84–86 °C) whose spectral data were consistent with the literature values.⁵

^1H NMR (400 MHz, CDCl_3) δ 7.22 (d, J = 7.2 Hz, 1H), 7.08 (s, 1H), 6.95 (d, J = 8.2 Hz, 1H), 6.19 (br s, 1H), 3.92 (s, 3H).

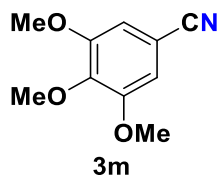
^{13}C NMR (100 MHz, CDCl_3) δ 150.04, 146.77, 127.10, 119.37, 115.34, 113.88, 103.35, 56.34.



3,4-dimethoxybenzonitrile (3l): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (59 mg, 72% yield; mp 65–67 °C) whose spectral data were consistent with the literature values.⁴

^1H NMR (400 MHz, CDCl_3) δ 7.30 – 7.24 (m, 1H), 7.06 (d, J = 1.9 Hz, 1H), 6.89 (d, J = 8.4 Hz, 1H), 3.91 (s, 3H), 3.88 (s, 3H).

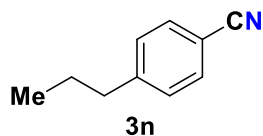
^{13}C NMR (100 MHz, CDCl_3) δ 152.96, 149.28, 126.55, 119.31, 114.02, 111.33, 103.98, 56.22, 56.18.



3,4,5-trimethoxybenzonitrile (3m): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (88 mg, 91% yield; mp 92–94 °C) whose spectral data were consistent with the literature values.⁶

¹H NMR (400 MHz, CDCl₃) δ 6.83 (s, 2H), 3.86 (s, 3H), 3.84 (s, 6H).

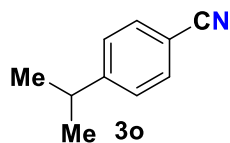
¹³C NMR (100 MHz, CDCl₃) δ 153.58, 142.34, 118.99, 109.47, 106.72, 61.05, 56.41.



4-propylbenzonitrile (3n): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 95:5, v/v) afforded the title compound as yellow oil (54 mg, 75% yield) whose spectral data were consistent with the literature values.⁷

¹H NMR (800 MHz, CDCl₃) δ 7.56 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 2.65 (t, *J* = 8.0 Hz, 2H), 1.65 (sext, *J* = 8.0 Hz, 2H), 0.95 (t, *J* = 8.0 Hz, 3H).

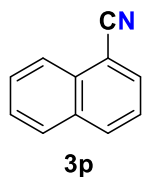
¹³C NMR (200 MHz, CDCl₃) δ 148.21, 131.92, 129.12, 119.06, 109.33, 37.94, 23.96, 13.52.



4-isopropylbenzonitrile (3o): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 98:2, v/v) afforded the title compound as colorless oil (61 mg, 84% yield) whose spectral data were consistent with the literature values.⁵

¹H NMR (800 MHz, CDCl₃) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 2.96 (sep, *J* = 8.0 Hz, 1H), 1.26 (d, *J* = 8.0 Hz, 6H).

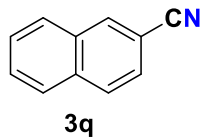
¹³C NMR (200 MHz, CDCl₃) δ 154.22, 132.05, 127.16, 119.02, 109.40, 34.21, 23.36.



1-naphthonitrile (3p): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as colorless oil (66 mg, 86% yield) whose spectral data were consistent with the literature values.¹

^1H NMR (400 MHz, CDCl_3) δ 8.24 (d, $J = 8.3$ Hz, 1H), 8.08 (d, $J = 8.3$ Hz, 1H), 7.92 (t, $J = 6.5$ Hz, 2H), 7.72 – 7.67 (m, 1H), 7.65 – 7.59 (m, 1H), 7.58 – 7.50 (m, 1H).

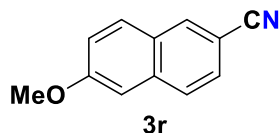
^{13}C NMR (100 MHz, CDCl_3) δ 133.39, 133.03, 132.74, 132.46, 128.77, 128.71, 127.66, 125.25, 125.03, 117.93, 110.29.



2-naphthonitrile (3q): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 95:5, v/v) afforded the title compound as light pink solid (67 mg, 88% yield) whose spectral data were consistent with the literature values.³

^1H NMR (800 MHz, CDCl_3) δ 8.16 (s, 1H), 7.87 (d, $J = 8$ Hz, 1H), 7.86 (d, $J = 8$ Hz, 1H), 7.84 (d, $J = 8$ Hz, 1H), 7.66-7.62 (m, 1H), 7.61-7.58 (m, 1H), 7.57 (dd, $J = 8.5, 1.5$ Hz, 1H).

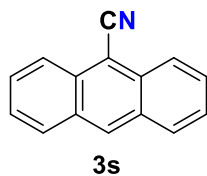
^{13}C NMR (200 MHz, CDCl_3) δ 134.34, 133.87, 131.92, 128.94, 128.83, 128.15, 127.81, 127.43, 126.03, 119.07, 109.02.



6-methoxy-2-naphthonitrile (3r): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 95:5, v/v) afforded the title compound as white solid (84 mg, 92% yield) whose spectral data were consistent with the literature values.³

^1H NMR (800 MHz, CDCl_3) δ 8.07 (s, 1H), 7.74 (d, $J = 8.0$ Hz, 1H), 7.72 (d, $J = 8.0$ Hz, 1H), 7.52 (dd, $J = 8.5, 1.5$ Hz, 1H), 7.23 (dd, $J = 8.9, 2.4$ Hz, 1H), 7.12 (d, $J = 2.2$ Hz, 1H), 3.94 (s, 3H).

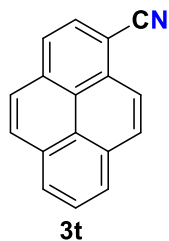
^{13}C NMR (200 MHz, CDCl_3) δ 150.79, 136.19, 133.50, 129.74, 127.60, 127.46, 126.80, 120.48, 119.46, 106.39, 105.67, 55.31.



Anthracene-9-carbonitrile (3s): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane) afforded the title compound as colorless oil (63 mg, 62% yield) whose spectral data were consistent with the literature values.⁵

^1H NMR (800 MHz, CDCl_3) δ 8.66 (s, 1H), 8.41 (dd, $J = 8.6, 0.6$ Hz, 2H), 8.07 (d, $J = 8$ Hz, 2H), 7.74-7.70 (m, 2H), 7.62-7.57 (m, 2H).

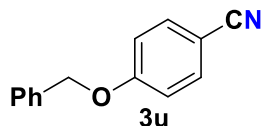
^{13}C NMR (200 MHz, CDCl_3) δ 133.24, 132.72, 130.55, 128.92, 128.91, 126.31, 125.22, 117.25, 105.32.



Pyrene-1-carbonitrile (3t): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 97:3, v/v) afforded the title compound as yellow gum (69 mg, 60% yield) whose spectral data were consistent with the literature values.⁹

¹H NMR (800 MHz, CDCl₃) δ 8.37 (d, *J* = 8 Hz, 1H), 8.27 (dd, *J* = 6.9, 5.1 Hz, 2H), 8.22 (d, *J* = 8.0 Hz, 1H), 8.18 (t, *J* = 8.0 Hz, 2H), 8.09 (t, *J* = 8.0 Hz, 2H), 8.02 (d, *J* = 8.0 Hz, 1H).

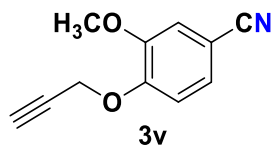
¹³C NMR (200 MHz, CDCl₃) δ 134.08, 132.85, 130.73, 130.42, 130.40, 130.40, 129.45, 126.99, 126.91, 126.89, 126.77, 124.32, 123.85, 123.82, 123.38, 118.80, 105.45.



4-(benzyloxy)benzonitrile (3u): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as white solid (76 mg, 73% yield; mp 85–87 °C) whose spectral data were consistent with the literature values.³

¹H NMR (400 MHz, CDCl₃) δ 7.58 (d, *J* = 8.2 Hz, 2H), 7.50 – 7.30 (m, 5H), 7.02 (d, *J* = 8.2 Hz, 2H), 5.12 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 162.07, 135.80, 134.12, 128.88, 128.53, 127.58, 119.28, 115.70, 104.32, 70.39.

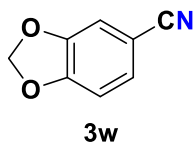


3-methoxy-4-(prop-2-yn-1-yloxy)benzonitrile (3v): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1, v/v) afforded the title compound as white solid (72 mg, 77% yield; mp 64–67 °C).

¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, *J* = 8.3 Hz, 1H), 7.21 – 7.01 (m, 2H), 4.83 (s, 2H), 3.90 (s, 3H), 2.57 (s, 1H).

¹³C NMR (100 MHz, CDCl₃) δ 150.65, 149.76, 126.15, 119.10, 114.55, 113.67, 105.17, 77.35, 76.94, 56.70, 56.26.

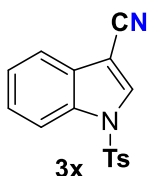
HRMS (ESI) *m/z* [M + H]⁺ calcd for [C₁₁H₁₀NO₂]⁺ 188.0706, found 188.0712.



Benzo[*d*][1,3]dioxole-5-carbonitrile (3w): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 4:1, v/v) afforded the title compound as white solid (61 mg, 83% yield; mp 88-90 °C) whose spectral data were consistent with the literature values.⁵

¹H NMR (400 MHz, CDCl₃) δ 7.24 – 7.17 (m, 1H), 7.03 (s, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.06 (s, 2H).

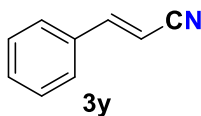
¹³C NMR (100 MHz, CDCl₃) δ 151.65, 148.14, 128.34, 119.01, 111.52, 109.25, 105.05, 102.33.



1-tosyl-1H-indole-3-carbonitrile (3x): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1, v/v) afforded the title compound as white solid (135 mg, 91% yield; mp 155–157 °C) whose spectral data were consistent with the literature values.¹

¹H NMR (400 MHz, CDCl₃) δ 8.10 (s, 1H), 8.00 (d, *J* = 8.3 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 2H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.30 (d, *J* = 8.1 Hz, 2H), 2.38 (s, 3H).

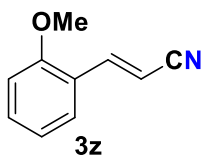
¹³C NMR (100 MHz, CDCl₃) δ 146.50, 134.25, 133.79, 133.27, 130.52, 128.47, 127.36, 126.65, 124.91, 120.42, 113.92, 113.61, 93.80, 21.81.



Cinnamitrile (3y): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as colourless oil (43 mg, 67% yield) whose spectral data were consistent with the literature values.³

¹H NMR (400 MHz, CDCl₃) δ 7.46 – 7.37 (m, 6H), 5.87 (d, *J* = 16.7 Hz, 1H).

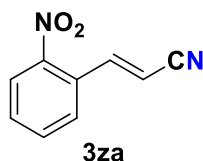
¹³C NMR (100 MHz, CDCl₃) δ 150.64, 133.58, 131.29, 129.19, 127.43, 118.24, 96.40.



(E)-3-(2-methoxyphenyl)acrylonitrile (3z): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 3:1, v/v) afforded the title compound as yellow solid (51 mg, 64% yield; mp 88–90 °C) whose spectral data were consistent with the literature values.⁹

¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, *J* = 16.8 Hz, 1H), 7.42 – 7.34 (m, 2H), 6.99 – 6.92 (m, 2H), 6.05 (d, *J* = 16.8 Hz, 1H), 3.88 (s, 3H).

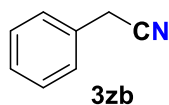
¹³C NMR (100 MHz, CDCl₃) δ 158.28, 146.51, 132.41, 128.99, 122.54, 120.88, 119.09, 111.36, 96.96, 55.60.



(E)-3-(2-nitrophenyl)acrylonitrile (3za): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 3:1, v/v) afforded the title compound as yellowish solid (60 mg, 69% yield; mp 88–90 °C) whose spectral data were consistent with the literature values.³

¹H NMR (400 MHz, CDCl₃) δ 8.11 (d, *J* = 7.4 Hz, 1H), 7.94 (d, *J* = 16.3 Hz, 1H), 7.71 – 7.57 (m, 3H), 5.86 (d, *J* = 16.3 Hz, 1H).

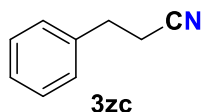
¹³C NMR (100 MHz, CDCl₃) δ 147.52, 146.69, 134.16, 131.43, 129.67, 128.81, 125.36, 117.04, 101.57.



2-phenylacetonitrile (3zb): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 96:4, v/v) afforded the title compound as yellow oil (38 mg, 65% yield) whose spectral data were consistent with the literature values.¹

¹H NMR (800 MHz, CDCl₃) δ 7.40–7.37 (m, 2H), 7.35–7.32 (m, 3H), 3.76 (s, 2H).

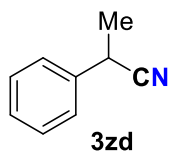
¹³C NMR (200 MHz, CDCl₃) δ 129.83, 129.11, 128.02, 127.88, 117.86, 23.58.



3-phenylpropanenitrile (3zc): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as colorless oil (50 mg, 76% yield) whose spectral data were consistent with the literature values.²

¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.32 (m, 2H), δ 7.32 – 7.29 (m, 3H), 2.95 (t, *J* = 7.4 Hz, 2H), 2.60 (t, *J* = 7.4 Hz, 2H).

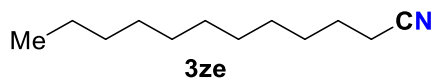
^{13}C NMR (100 MHz, CDCl_3) δ 138.15, 128.96, 128.35, 127.32, 119.23, 31.65, 19.42.



2-phenylpropanenitrile (3zd): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 96:4, v/v) afforded the title compound as colorless oil (54 mg, 80% yield) whose spectral data were consistent with the literature values.²

^1H NMR (800 MHz, CDCl_3) δ 7.42 (t, $J = 8.0$ Hz, 2H), 7.38 (d, $J = 8.0$ Hz, 2H), 7.36 (t, $J = 8.0$ Hz, 1H), 3.92 (q, $J = 8.0$ Hz, 1H), 1.66 (d, $J = 8.0$ Hz, 3H).

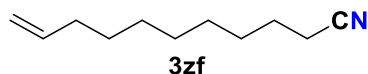
^{13}C NMR (200 MHz, CDCl_3) δ 136.94, 129.02, 127.91, 126.58, 121.52, 31.09, 21.34.



Dodecanenitrile (3ze): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as colorless oil (57 mg, 63% yield) whose spectral data were consistent with the literature values.¹

^1H NMR (400 MHz, CDCl_3) δ 2.32 (t, $J = 7.1$ Hz, 2H), 1.64 (p, $J = 7.2$ Hz, 2H), 1.49 – 1.37 (m, 2H), 1.35 – 1.17 (m, 14H), 0.87 (t, $J = 6.8$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 119.93, 31.97, 29.63, 29.58, 29.38, 28.85, 28.74, 25.46, 22.75, 17.19, 14.17.



Undec-10-enenitrile (3zf): Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 9:1, v/v) afforded the title compound as colorless oil (49 mg, 59% yield) whose spectral data were consistent with the literature values.⁷

^1H NMR (400 MHz, CDCl_3) δ 5.87 – 5.72 (m, 1H), 5.05 – 4.87 (m, 2H), 2.33 (t, $J = 7.1$ Hz, 2H), 2.10 – 1.97 (m, 2H), 1.71 – 1.60 (m, 2H), 1.47 – 1.28 (m, 10H).

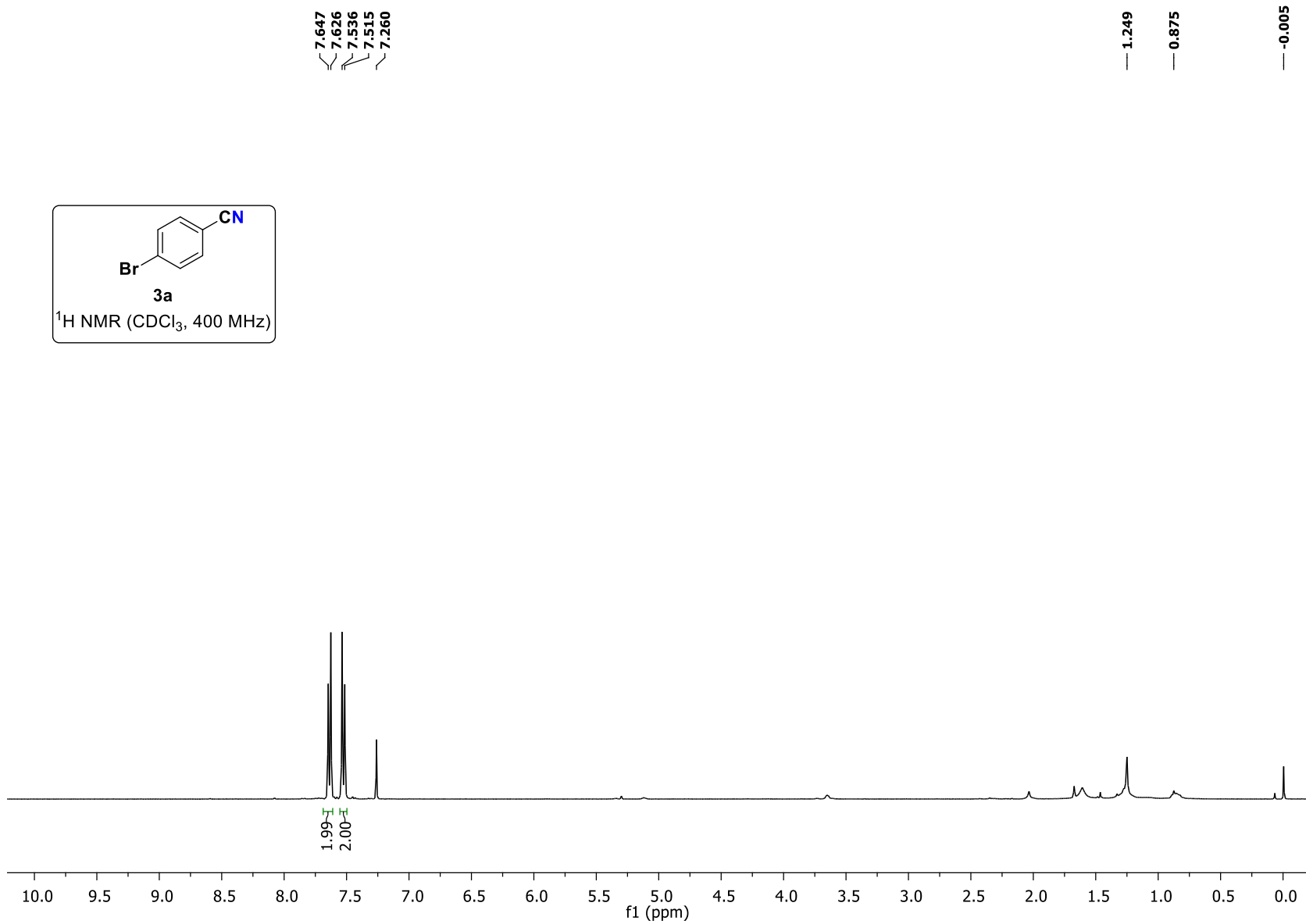
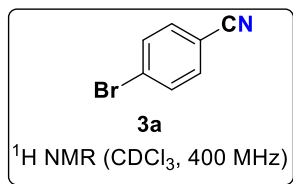
^{13}C NMR (100 MHz, CDCl_3) δ 139.17, 119.97, 114.36, 33.85, 29.25, 29.06, 28.94, 28.83, 28.76, 25.47, 17.24.

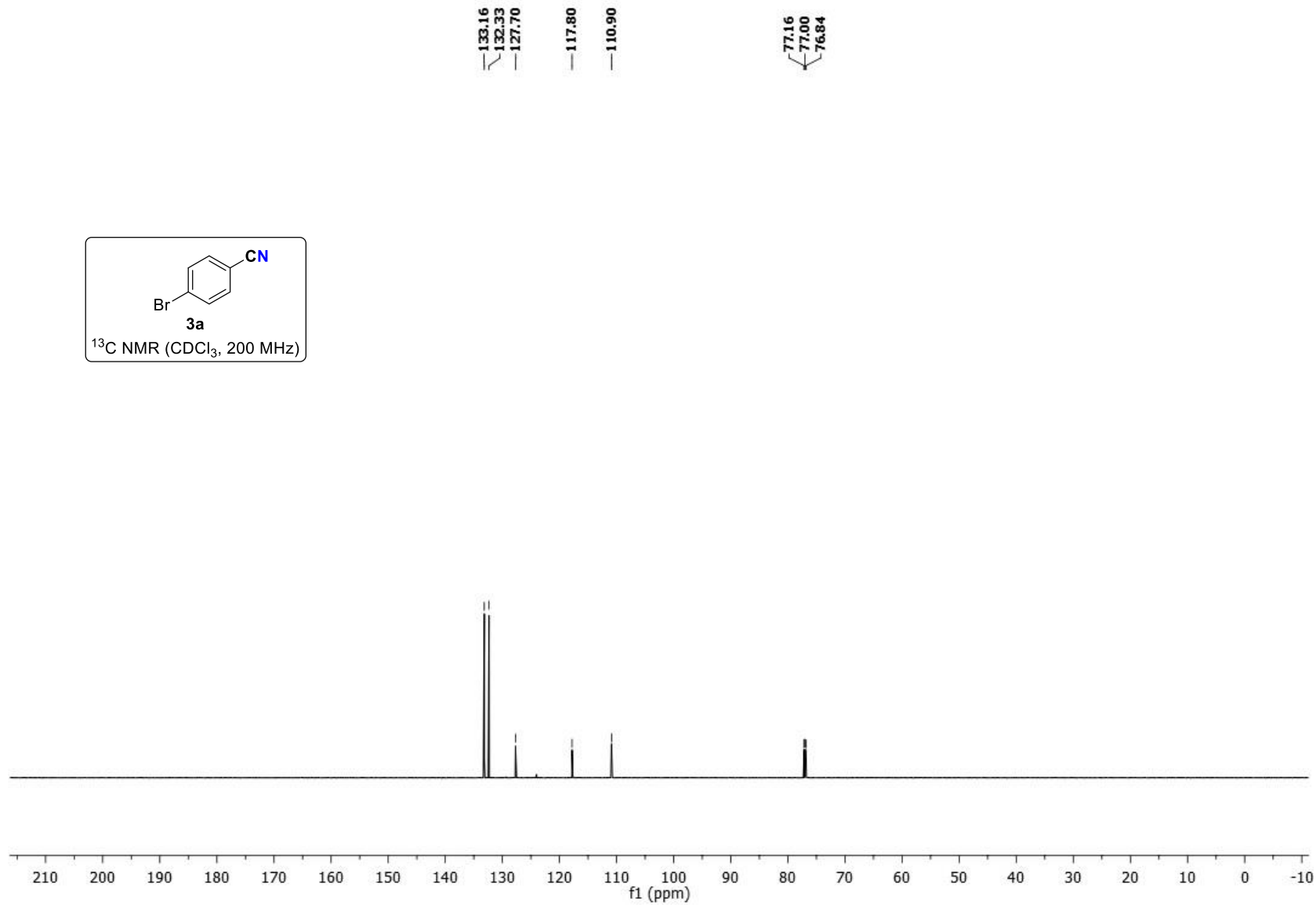
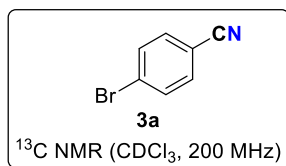
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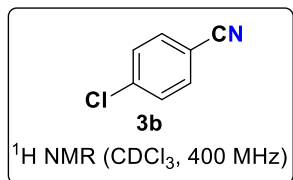
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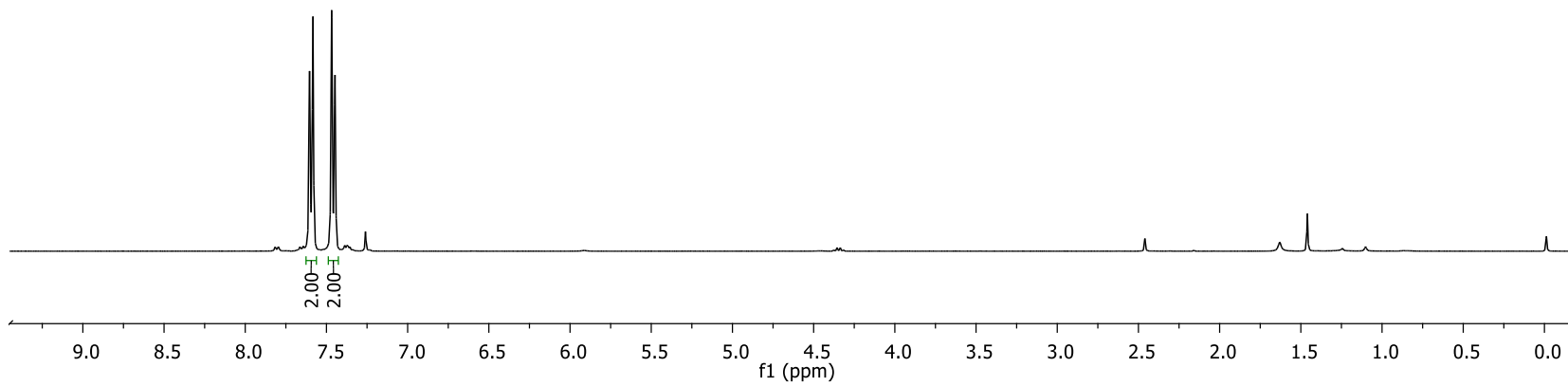
5. NMR Spectra of the products

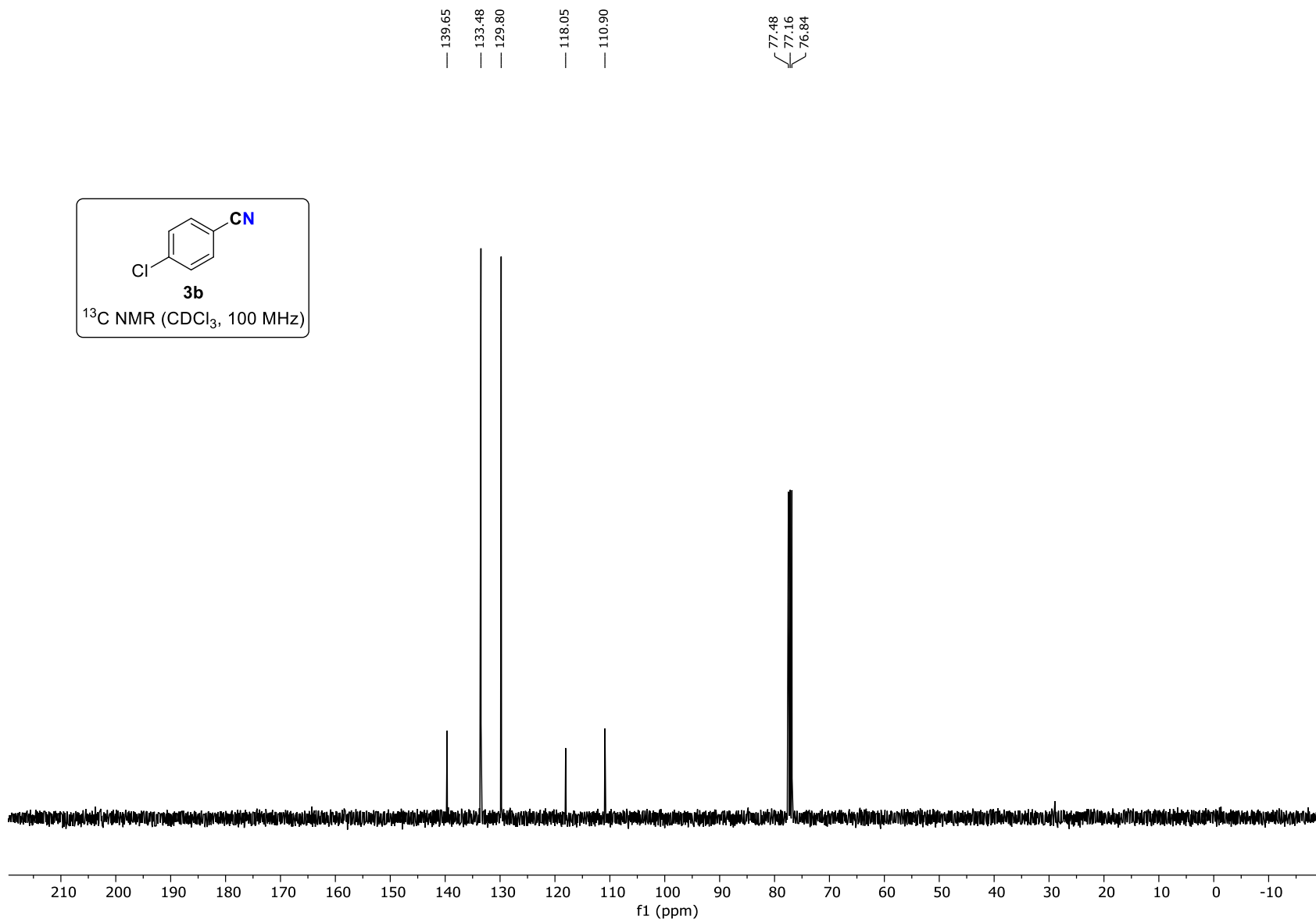
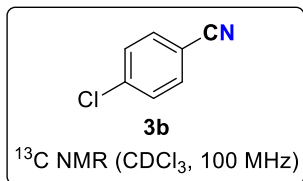


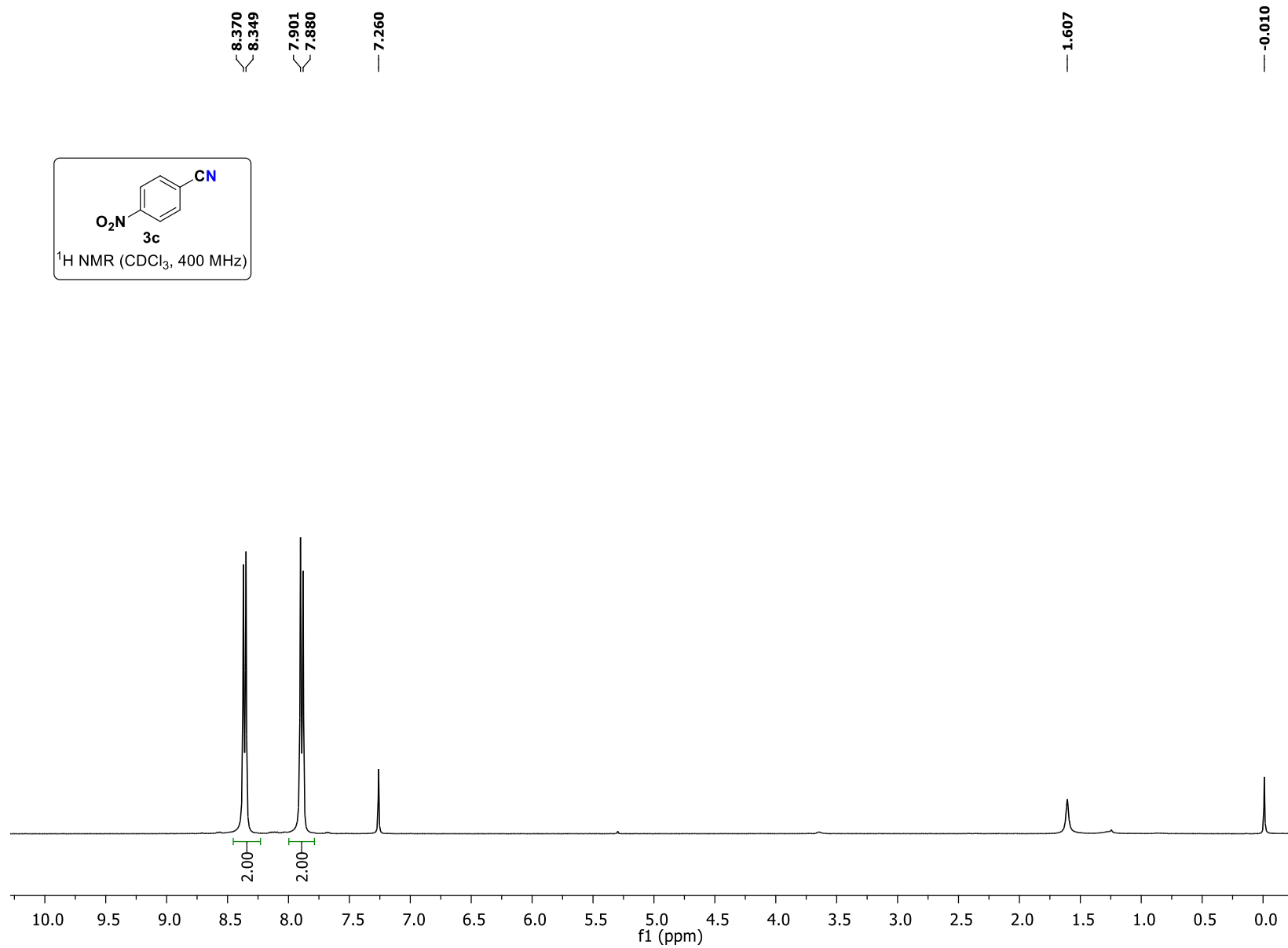
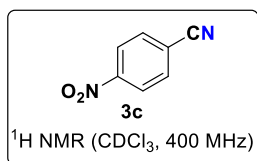


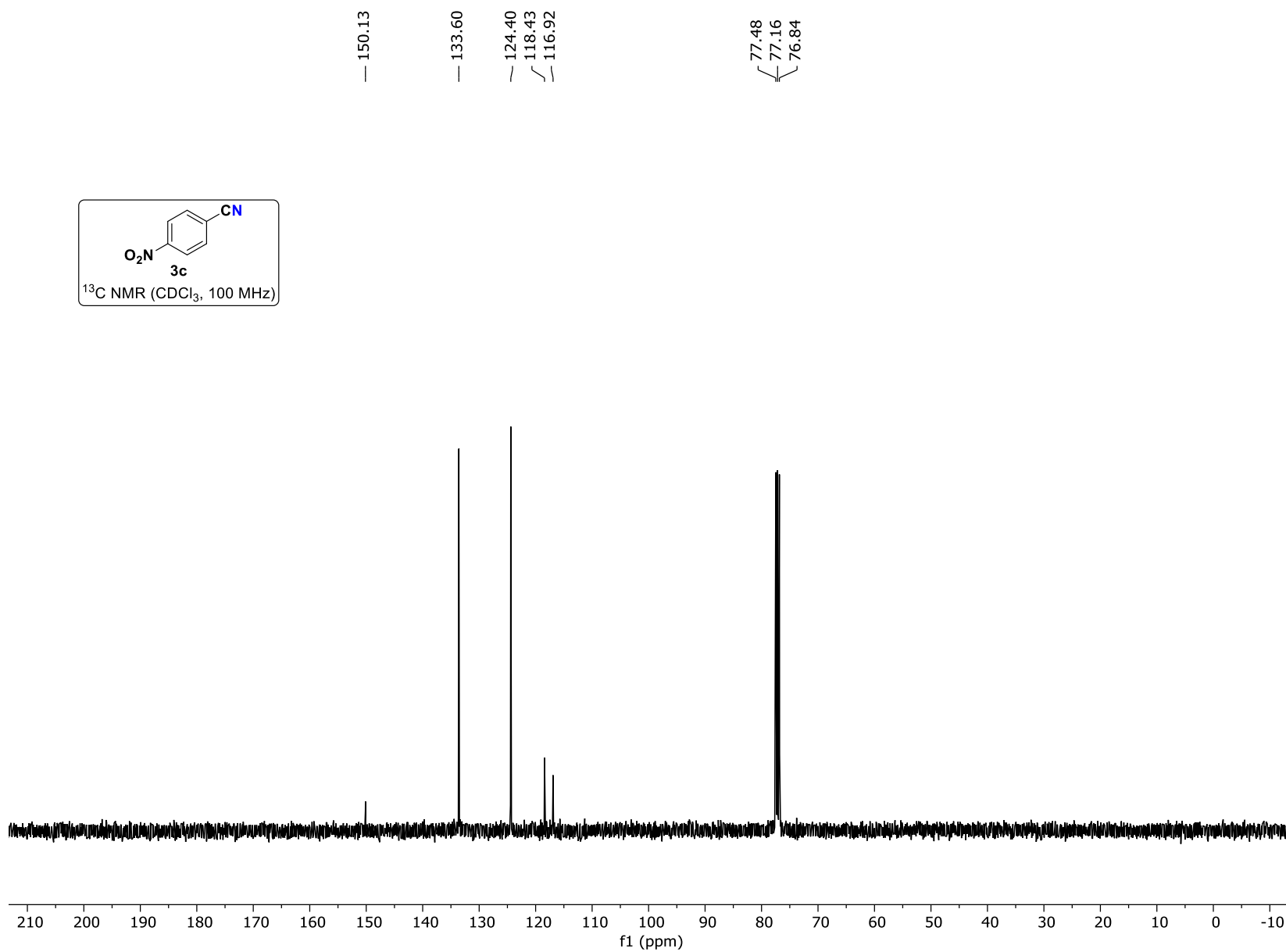
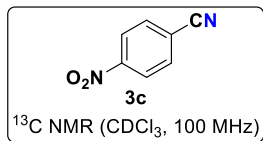


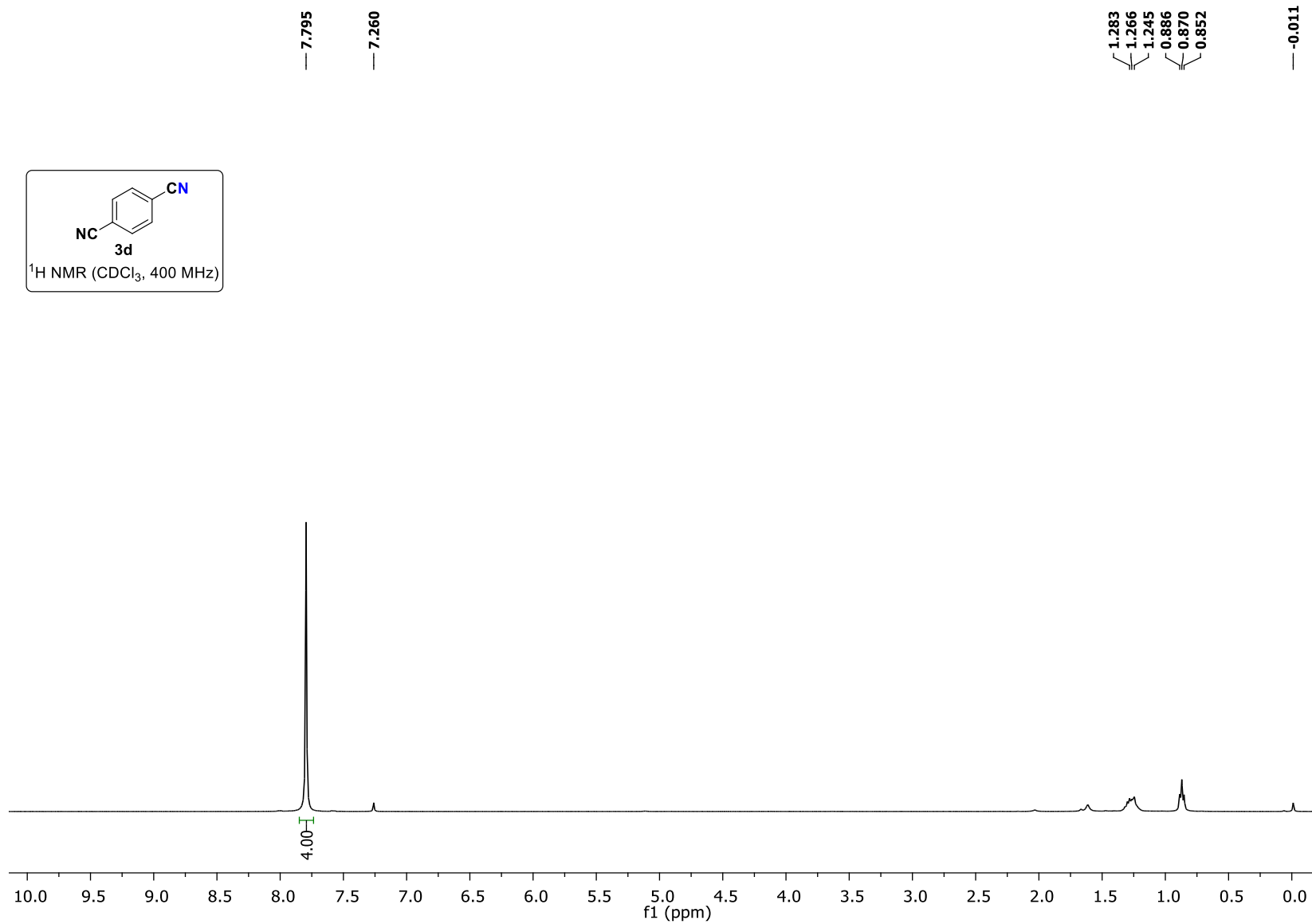
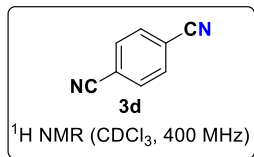
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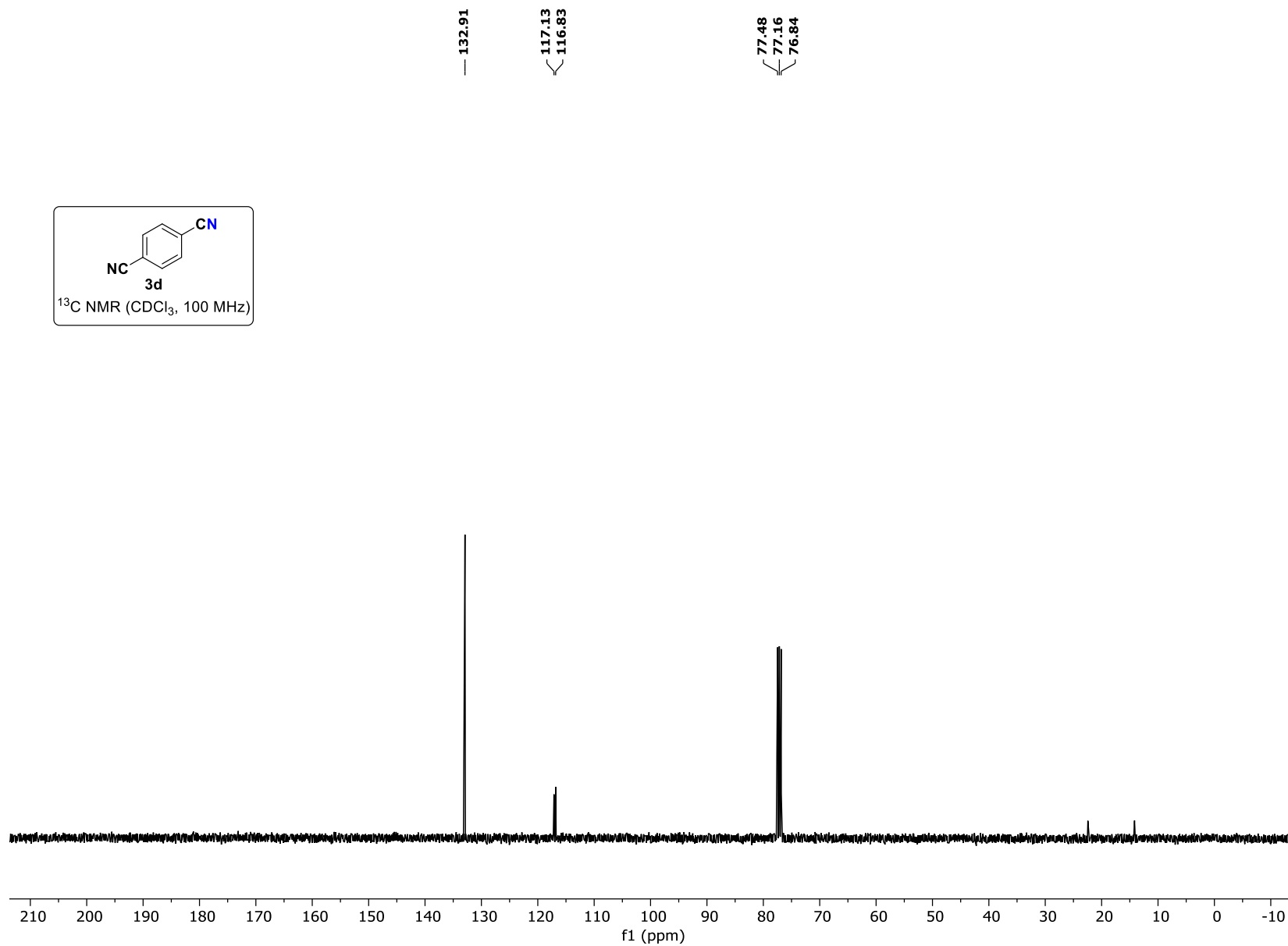
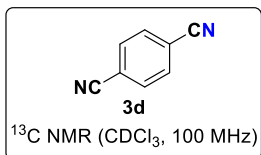


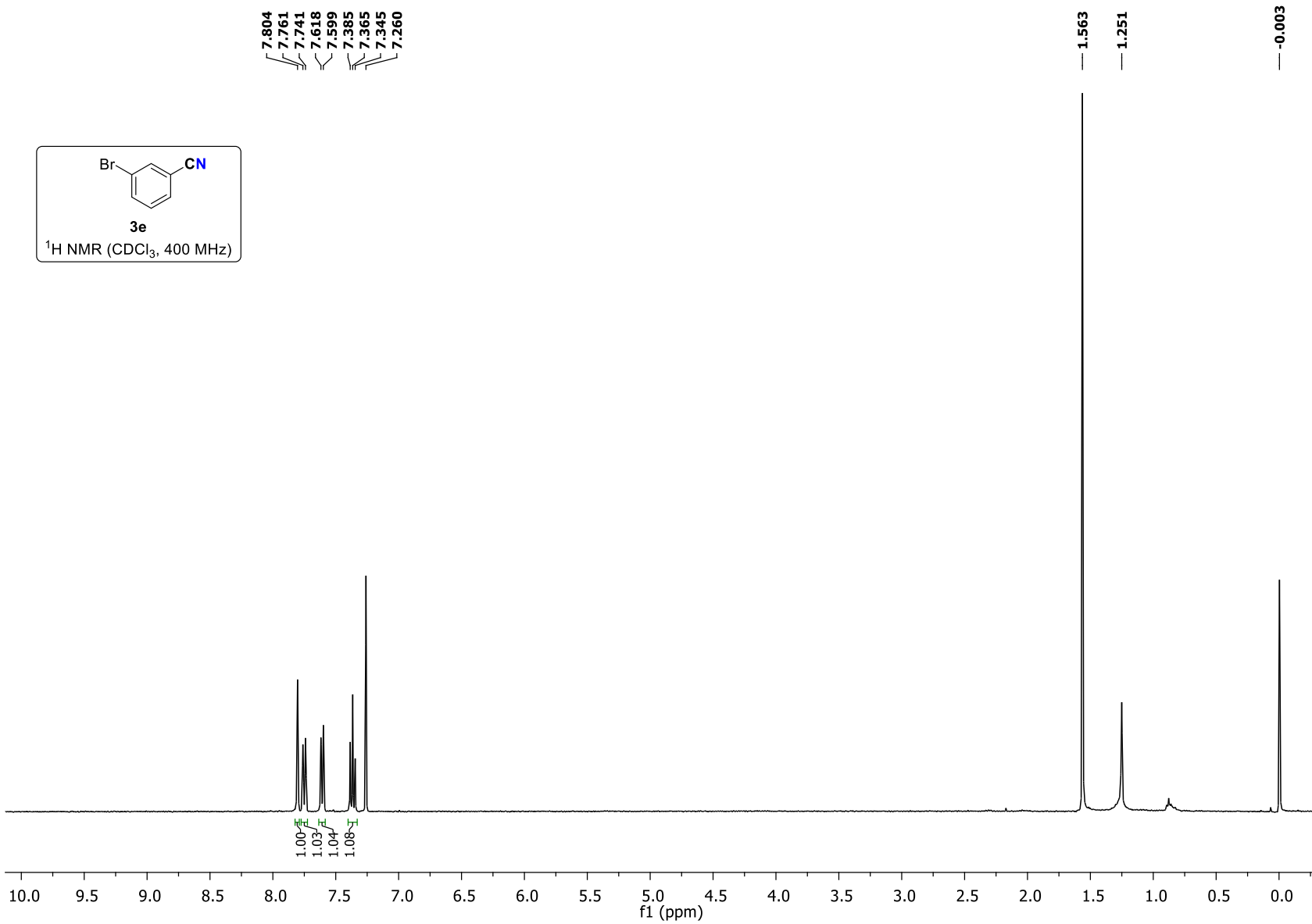
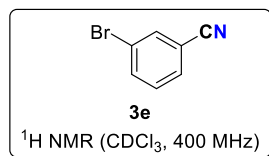


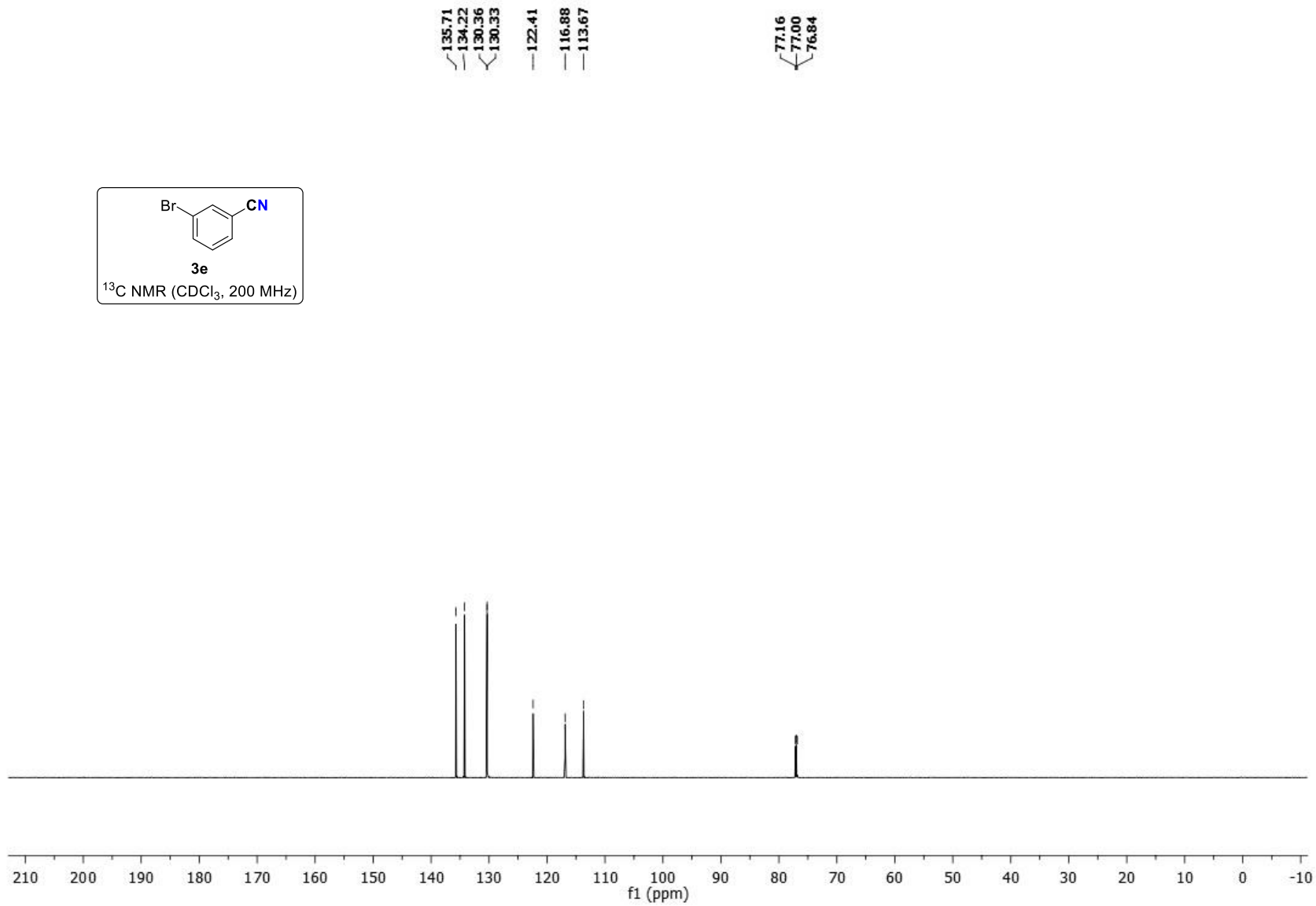
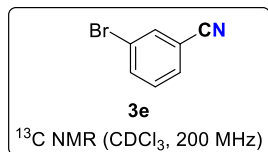


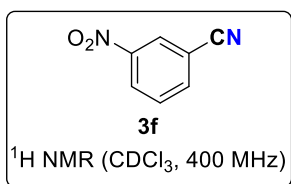








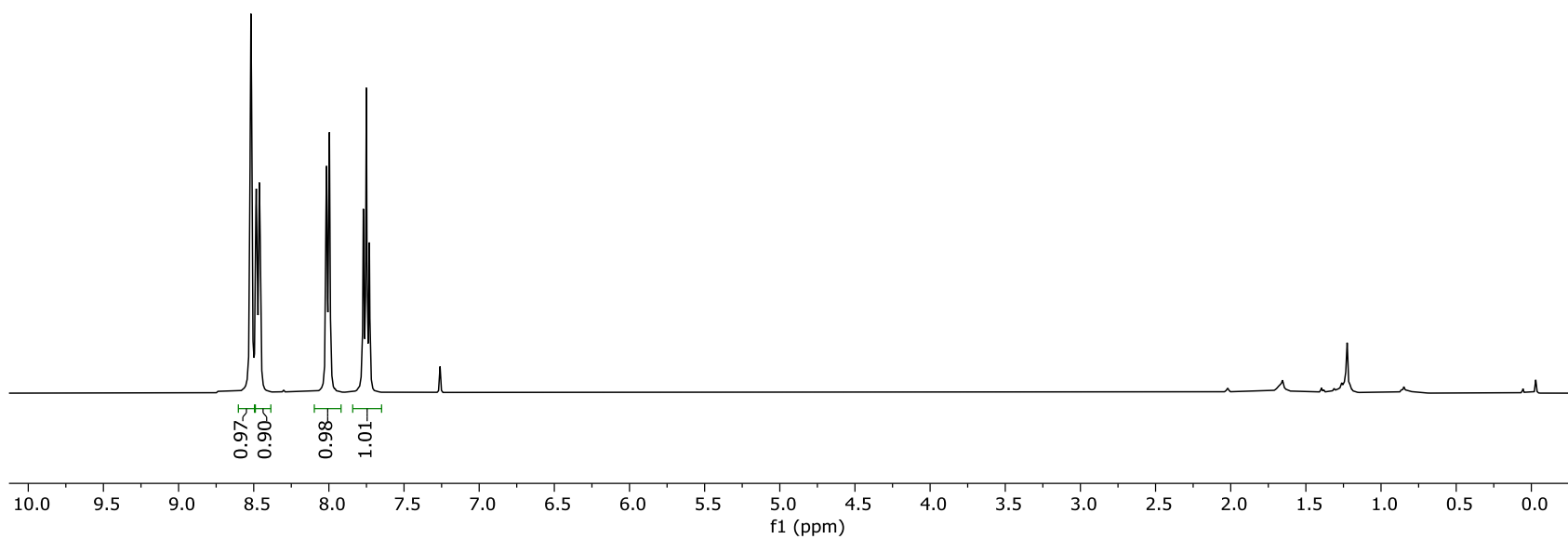


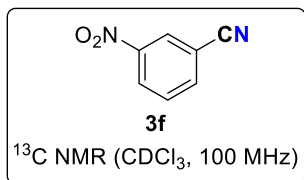


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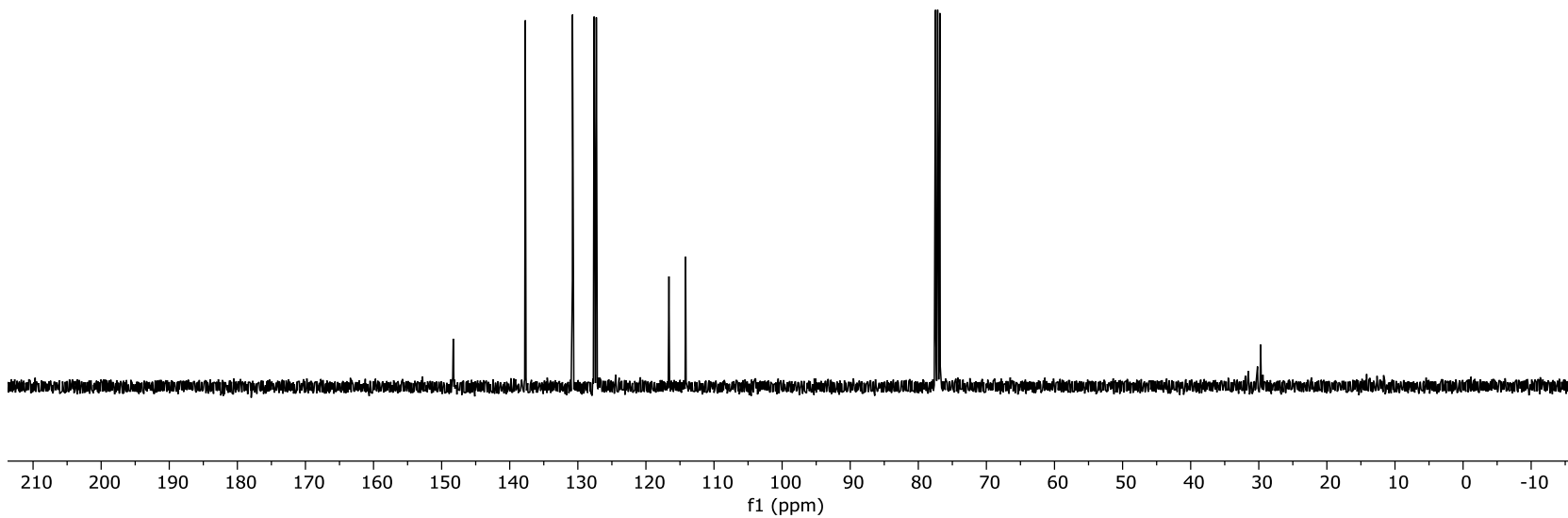
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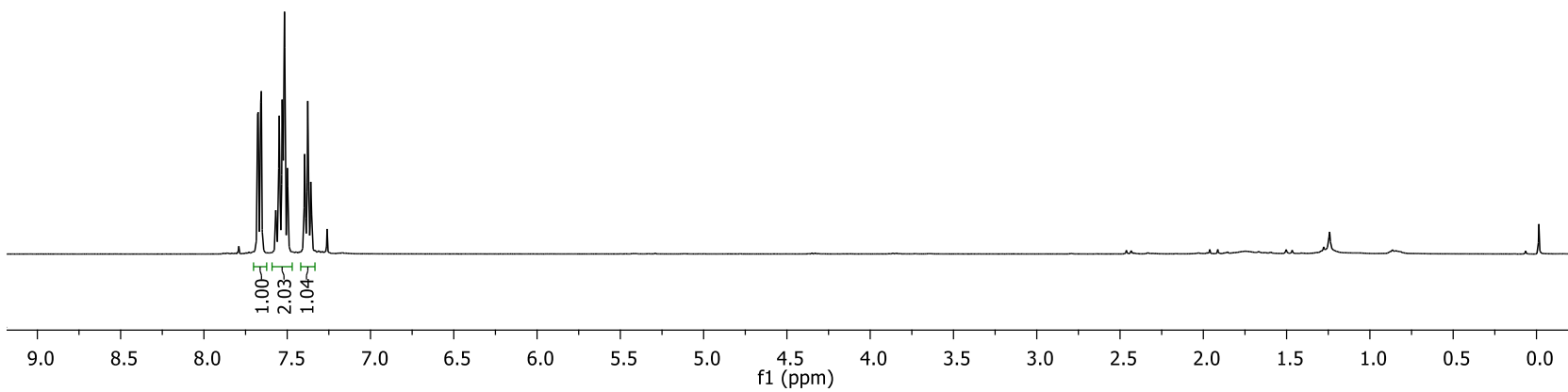
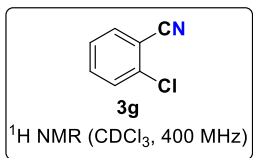


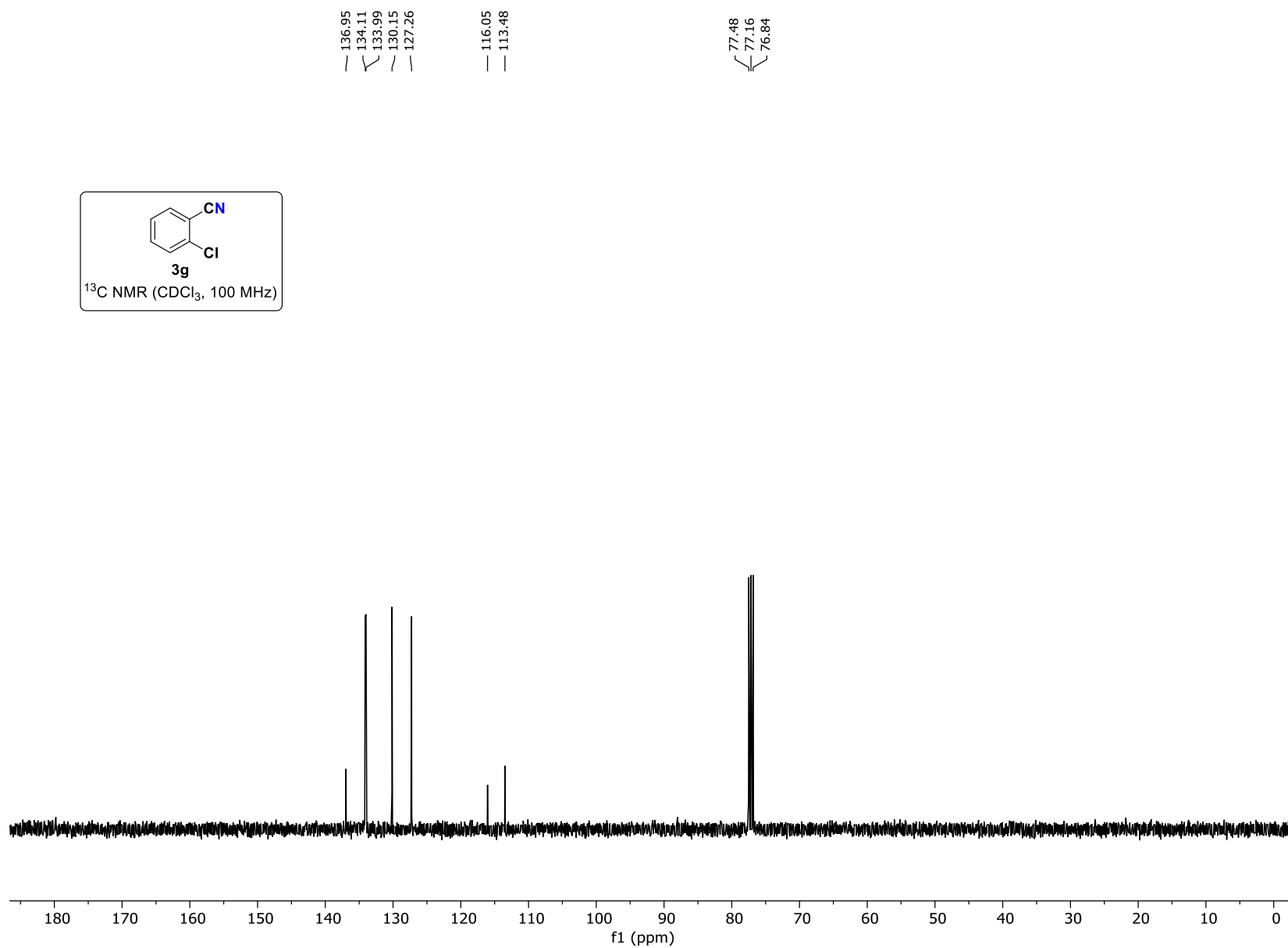
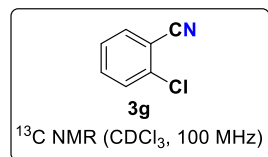


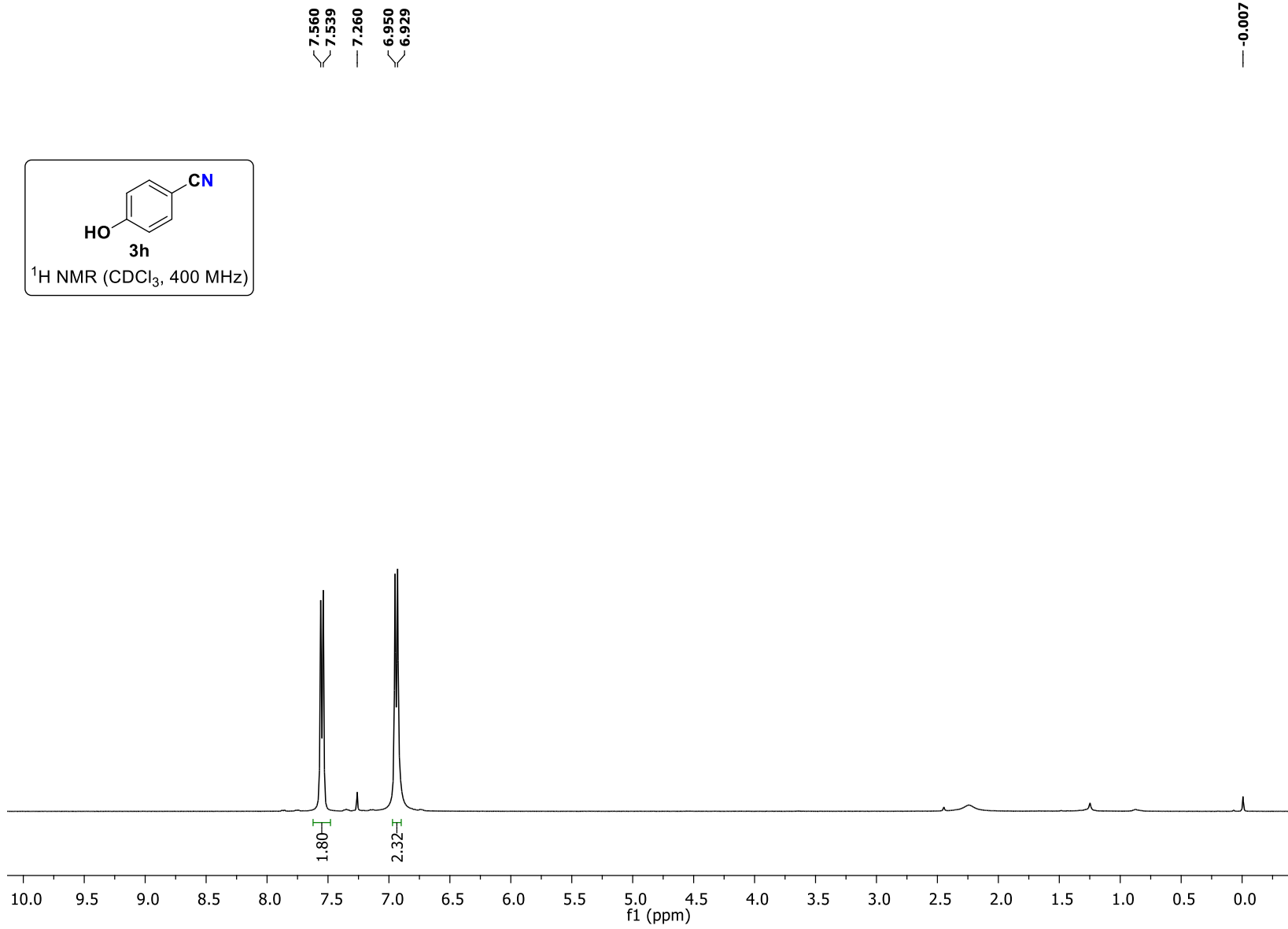
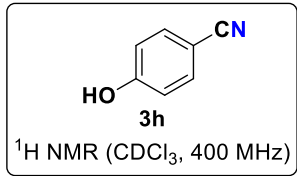
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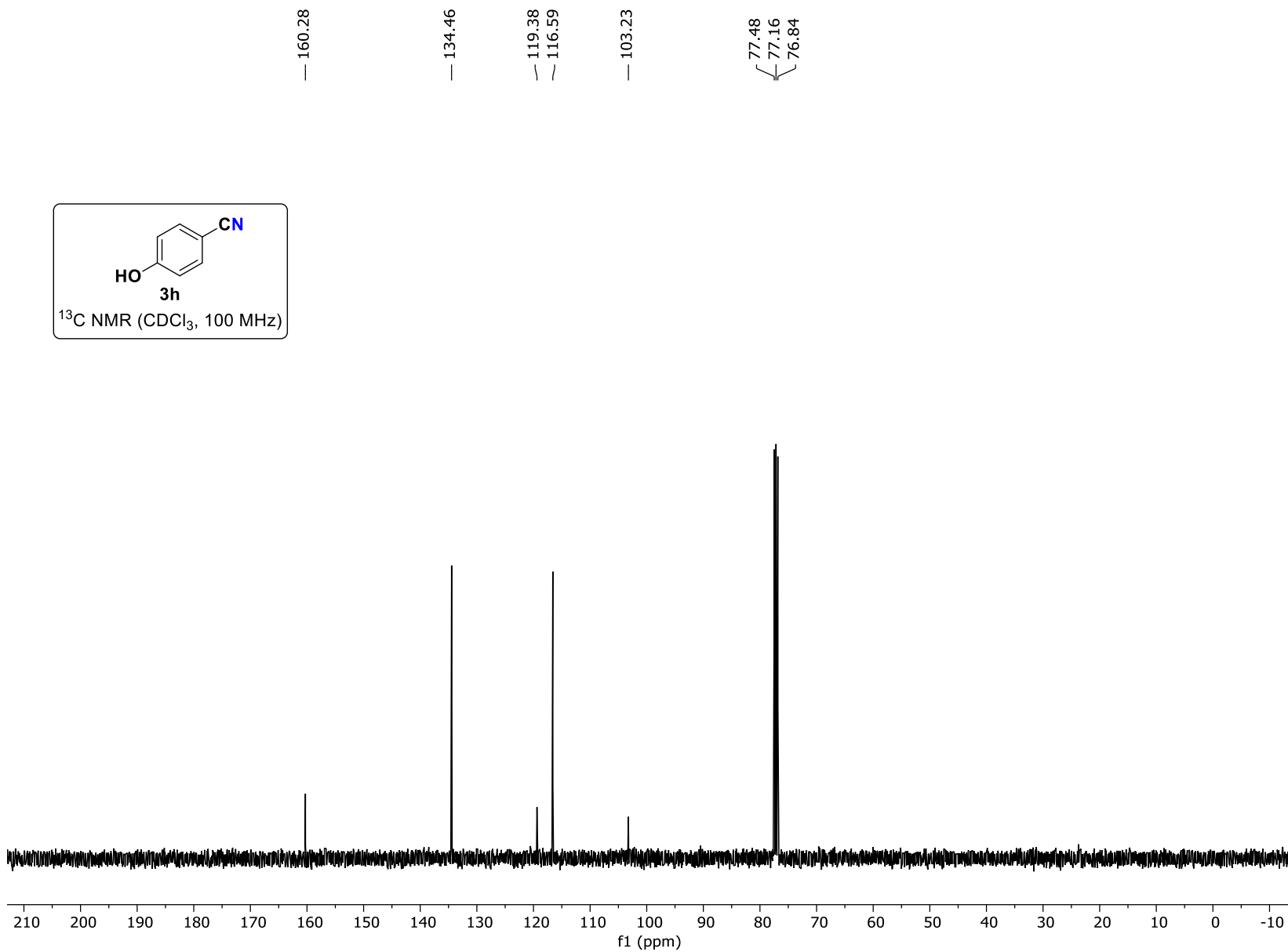
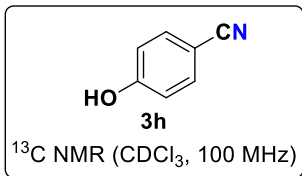


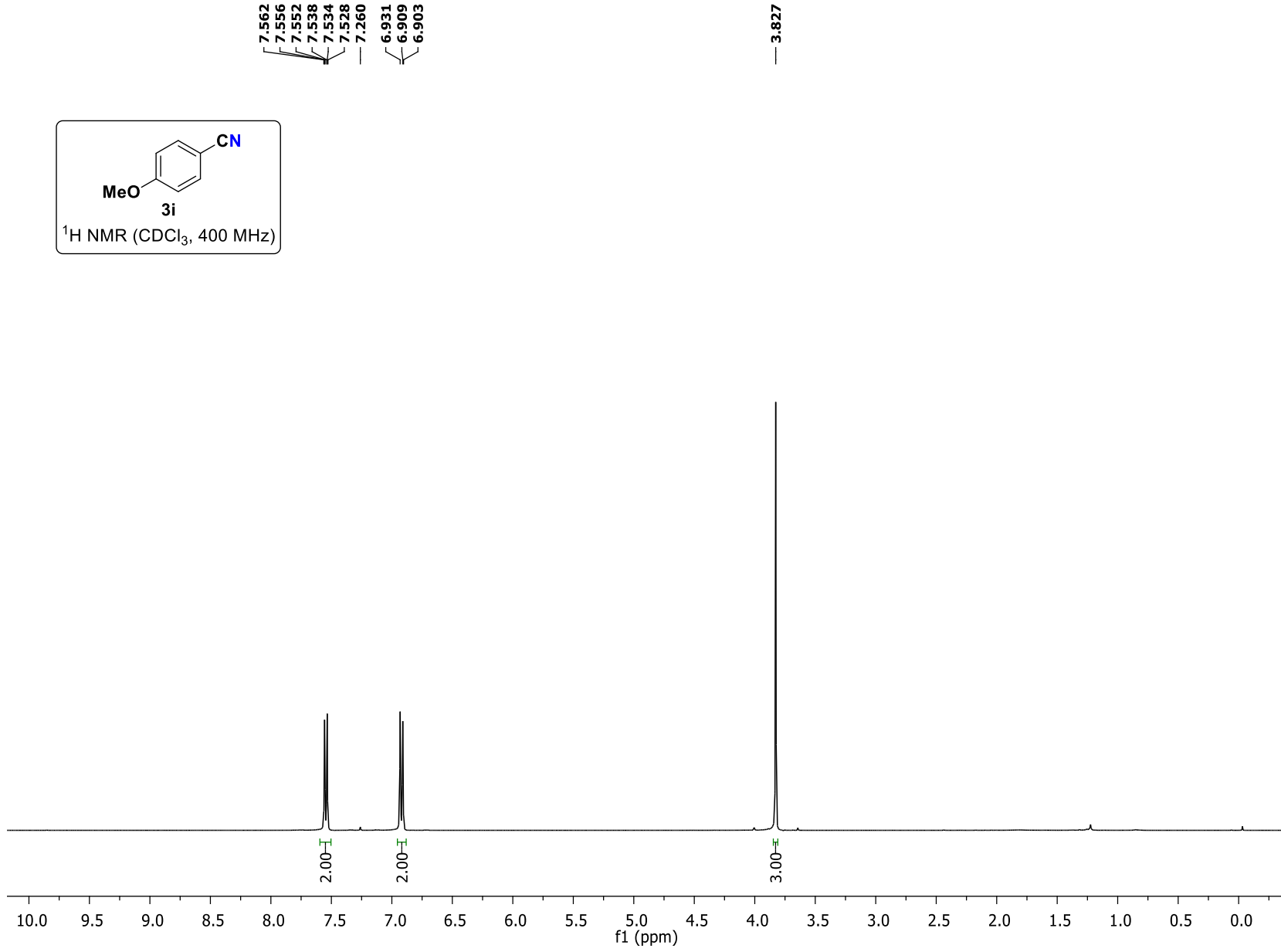
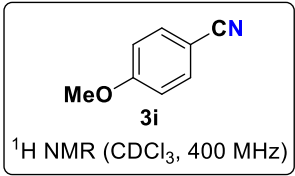
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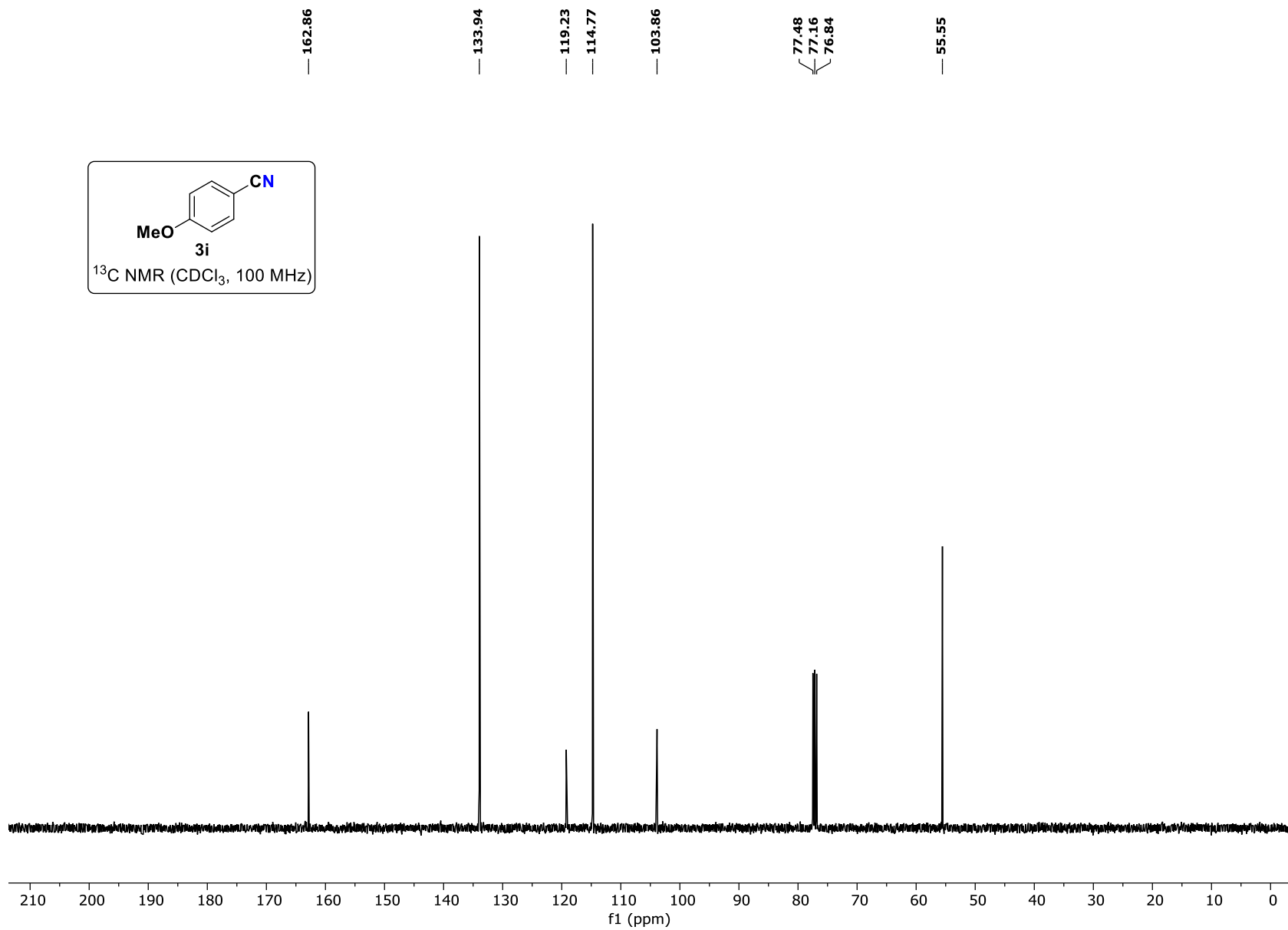


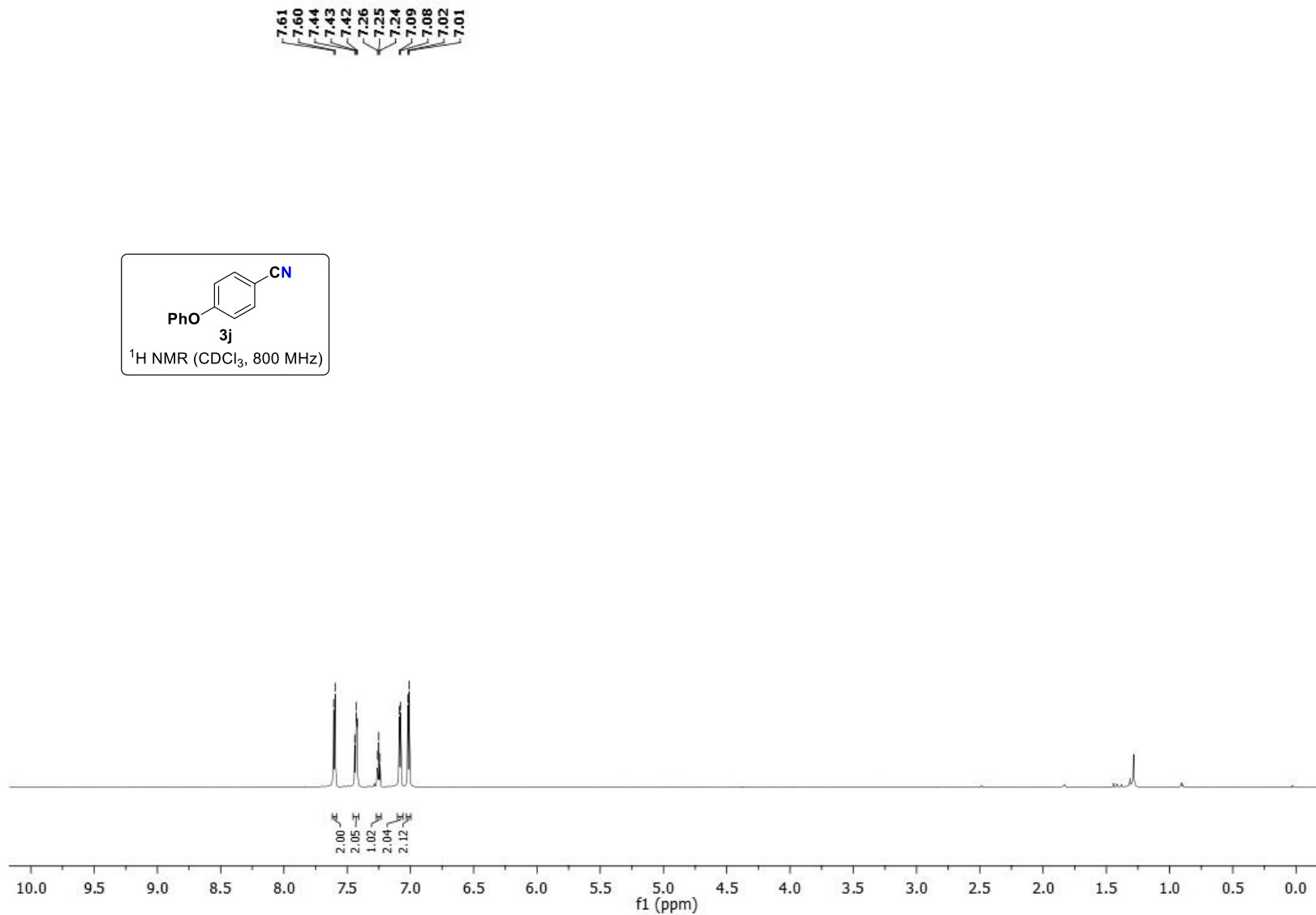
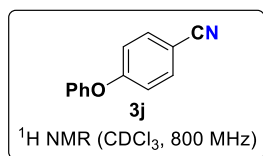


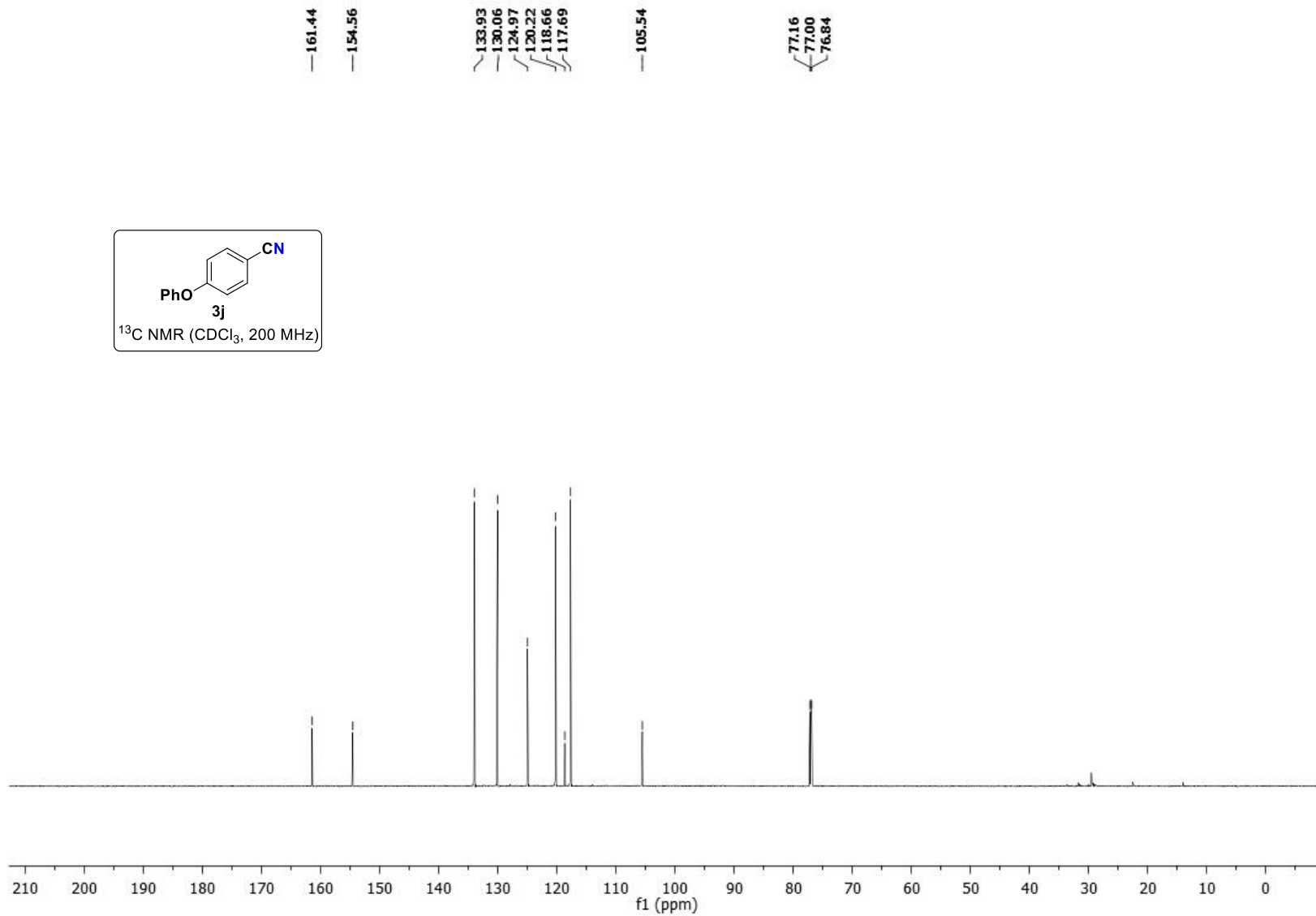
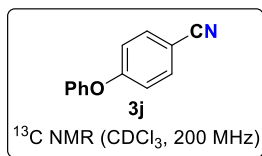


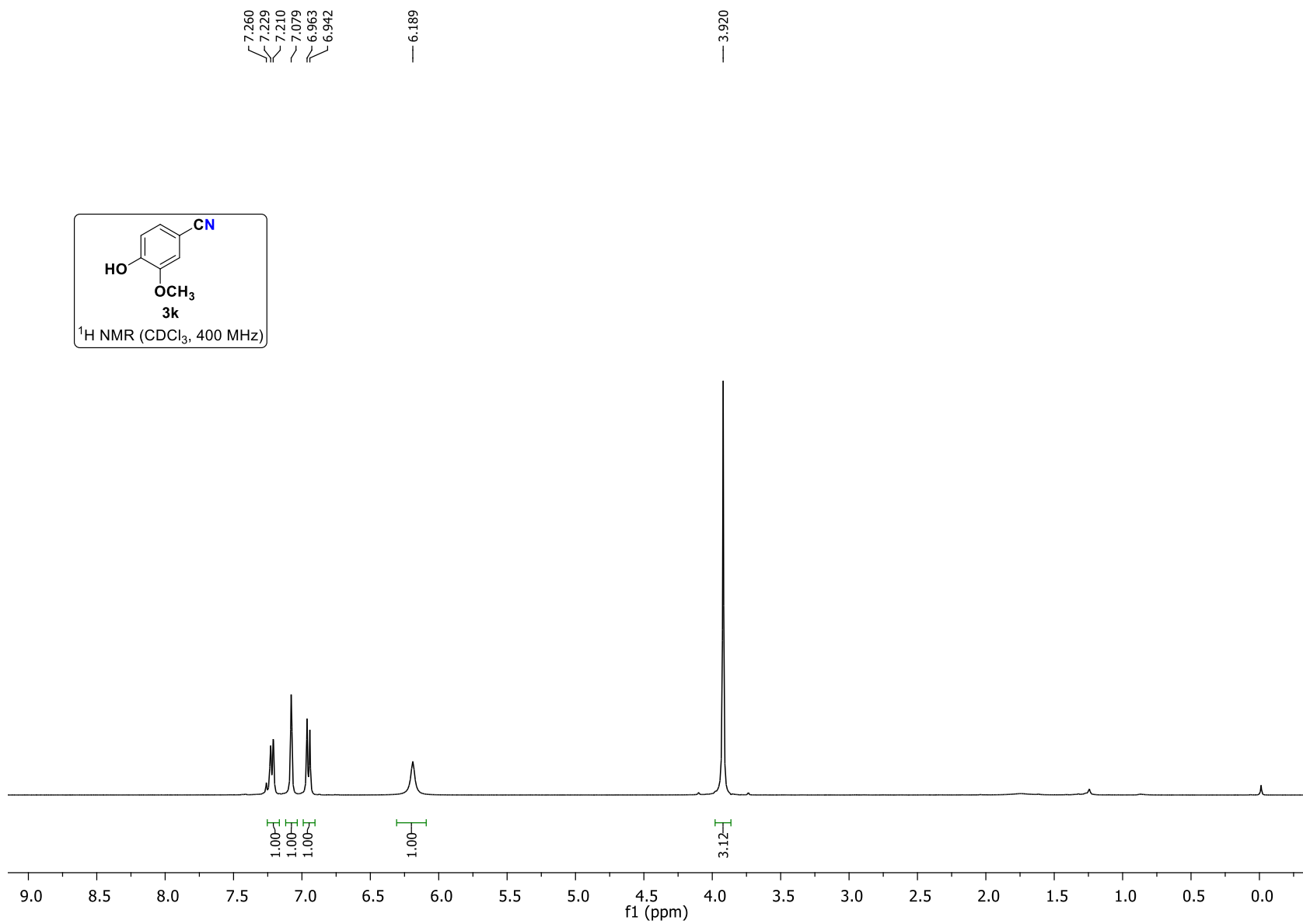
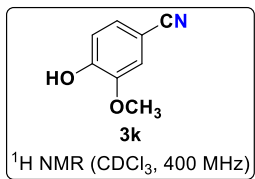


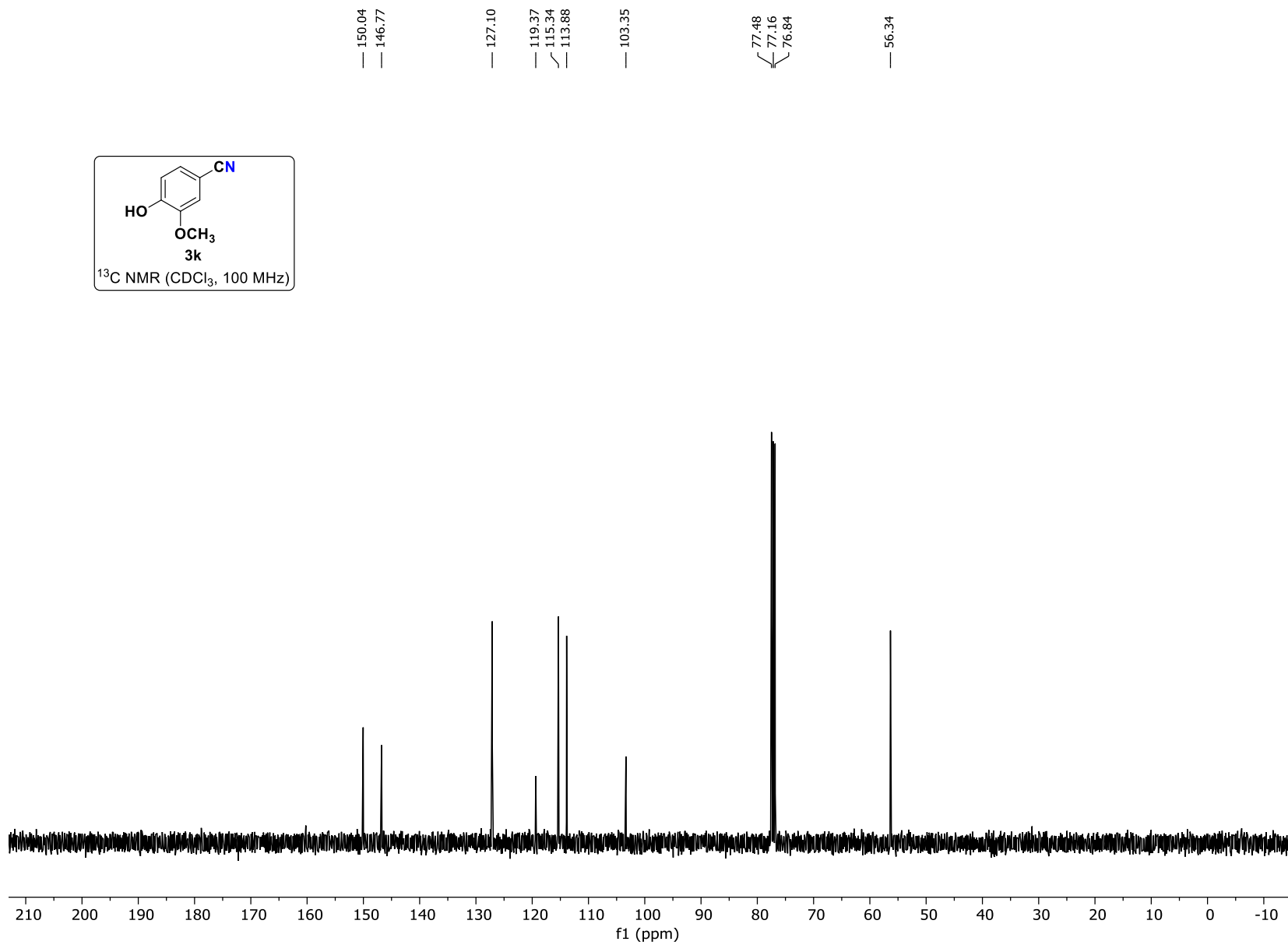
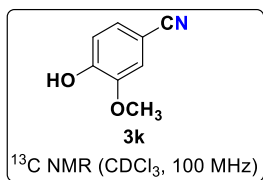


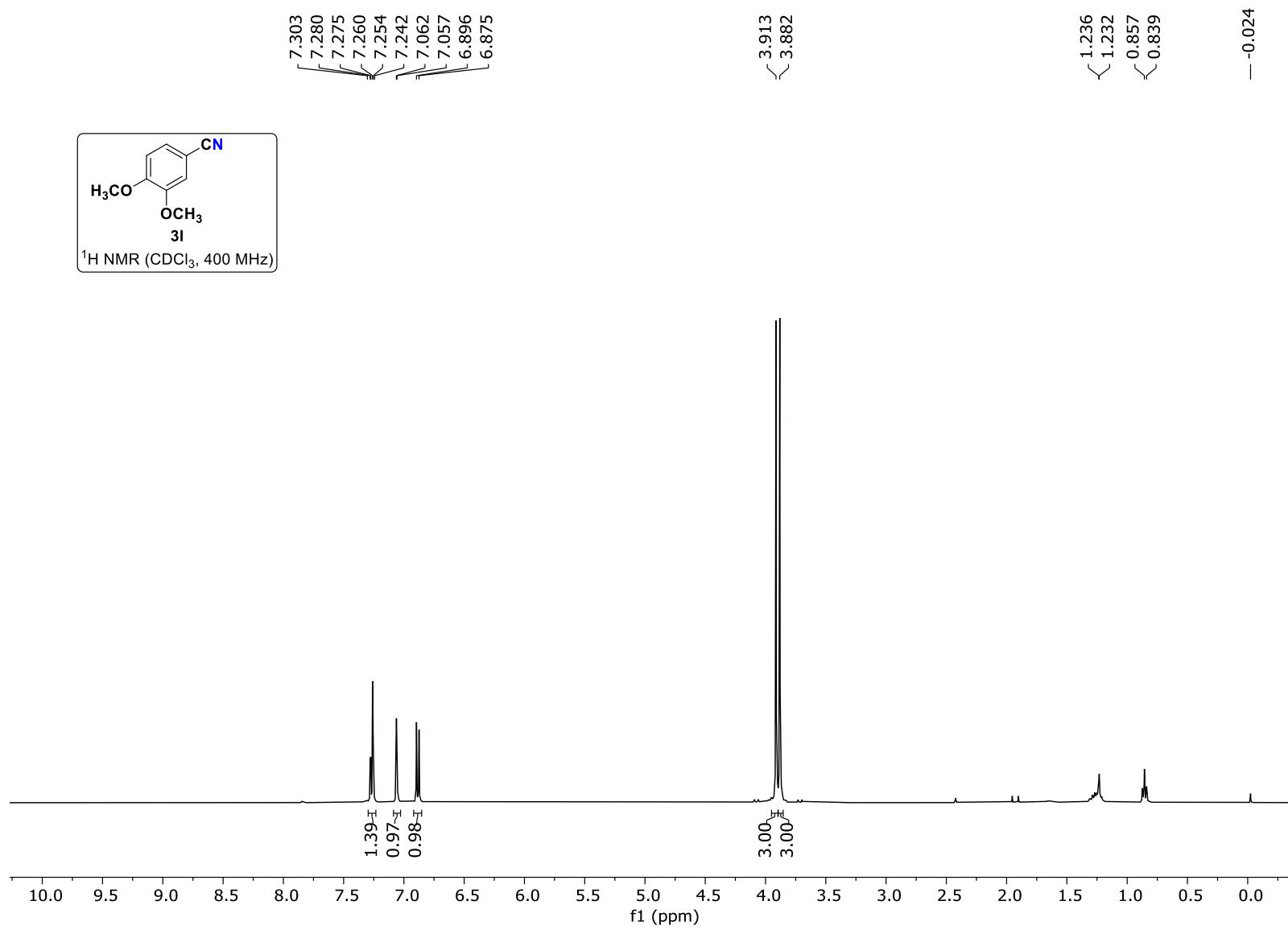
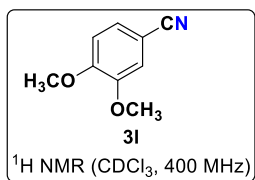


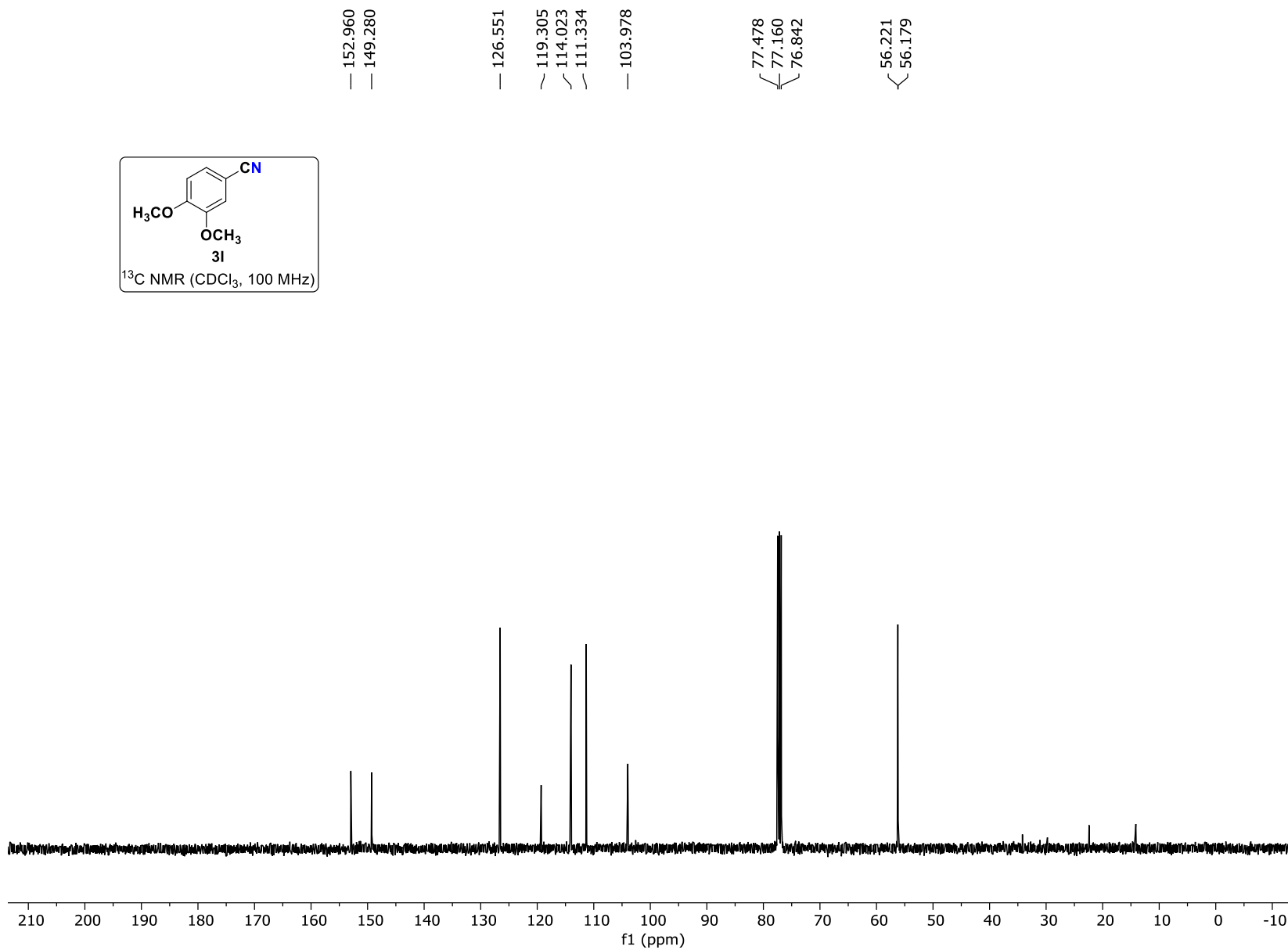
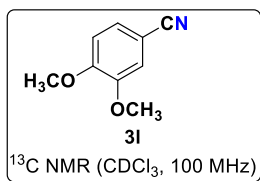


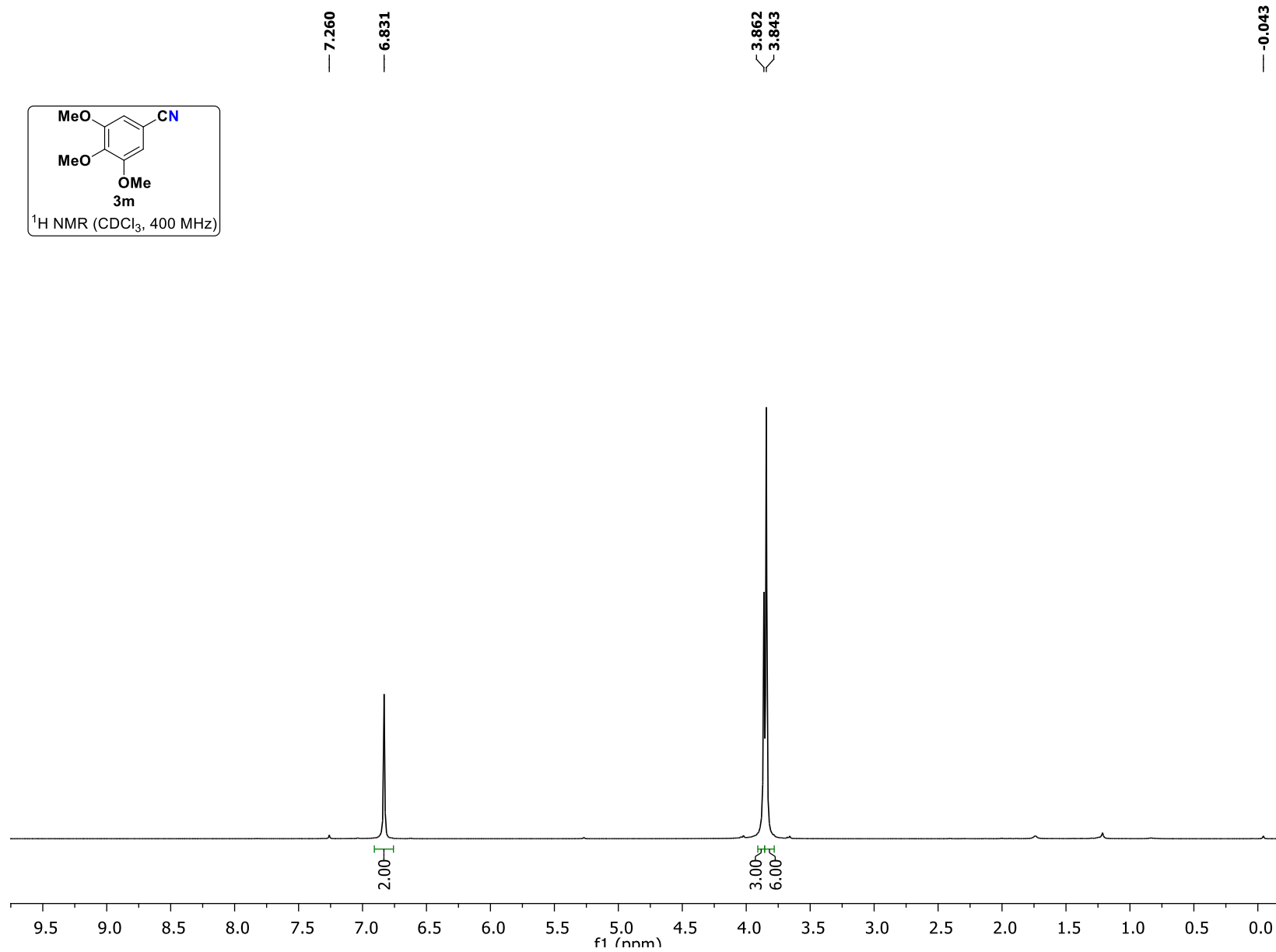
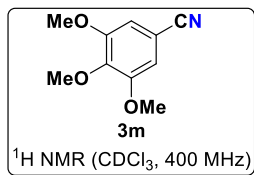


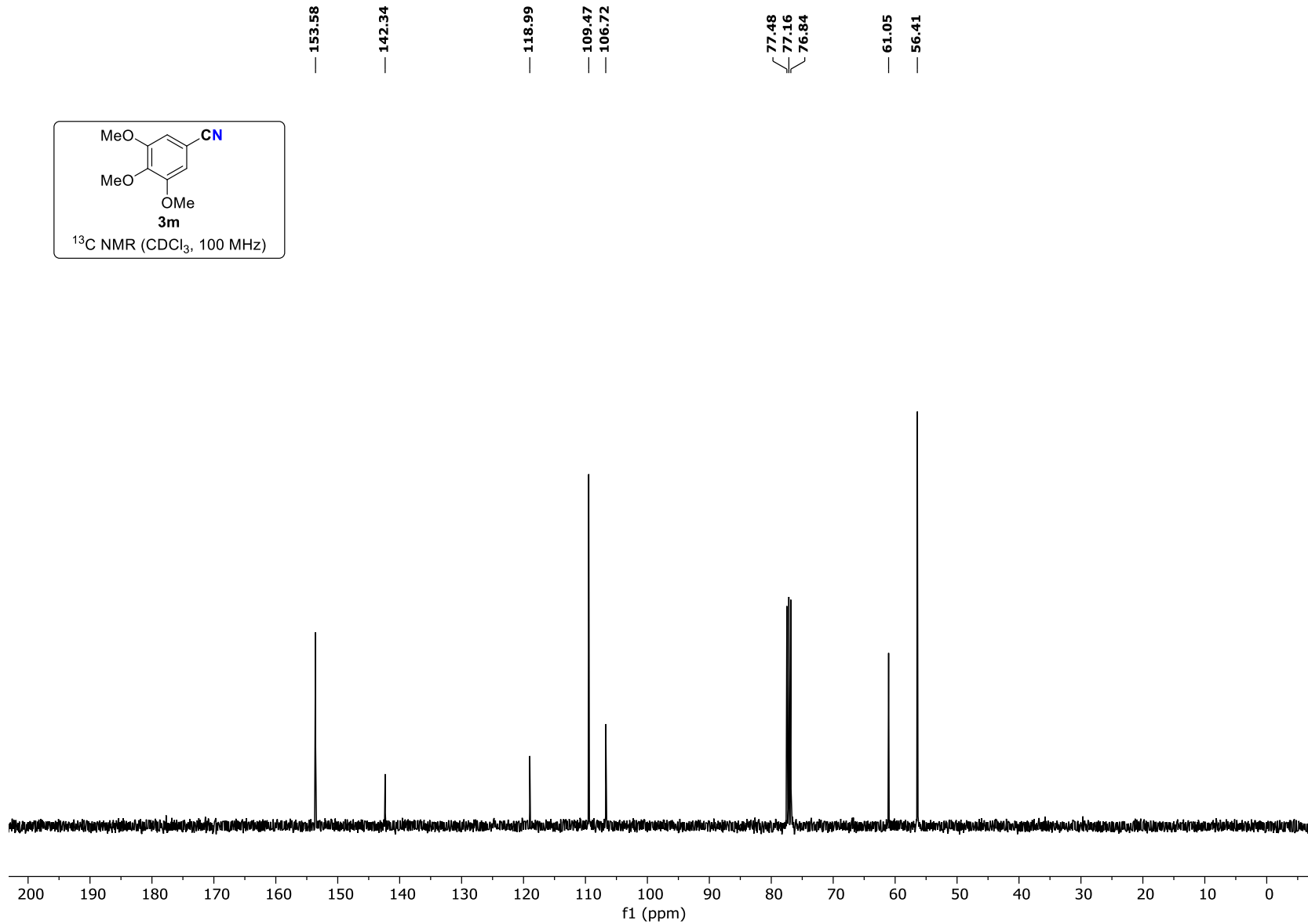
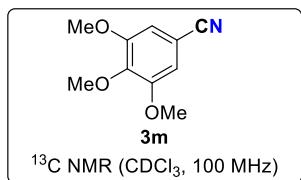


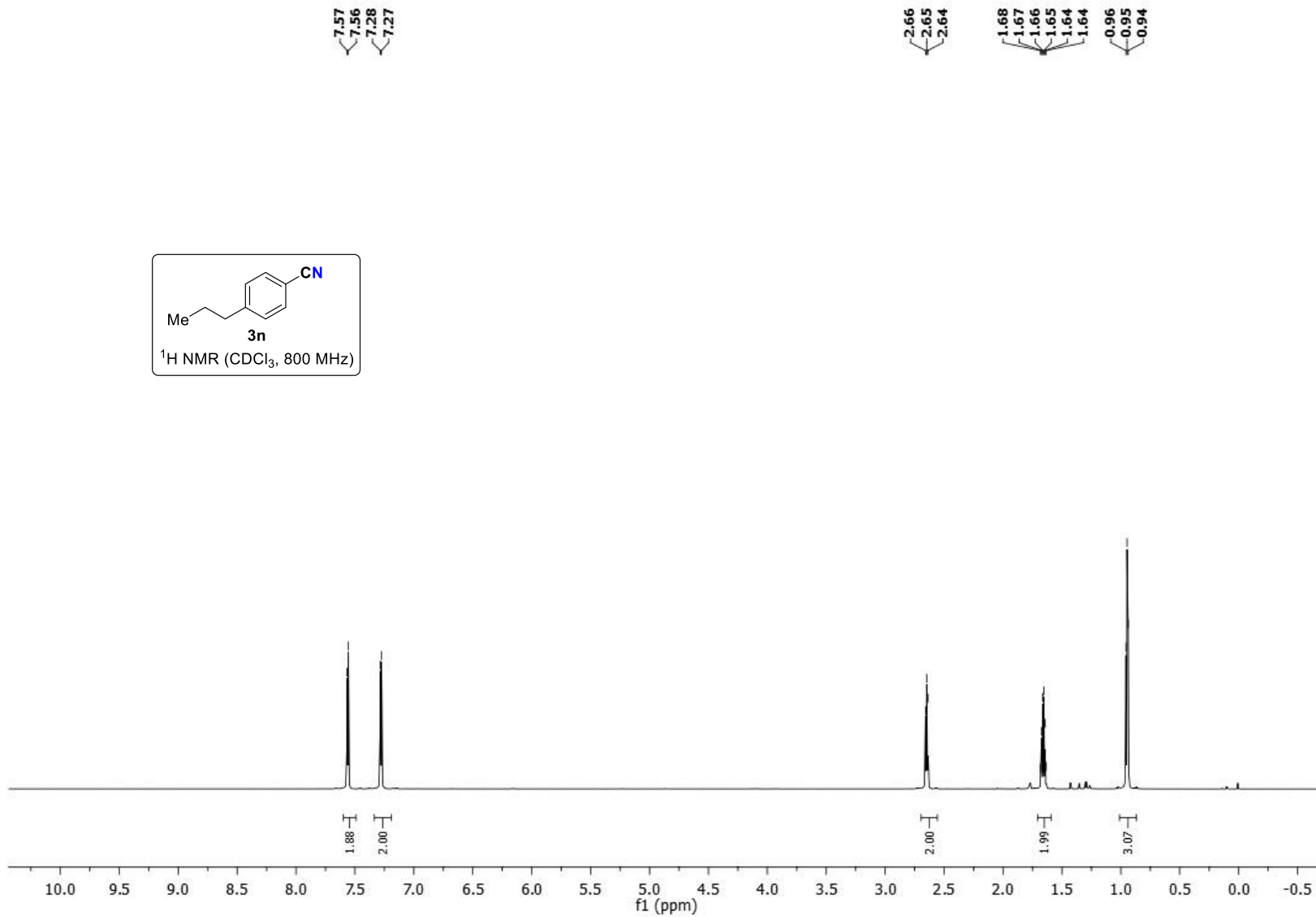
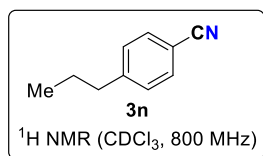


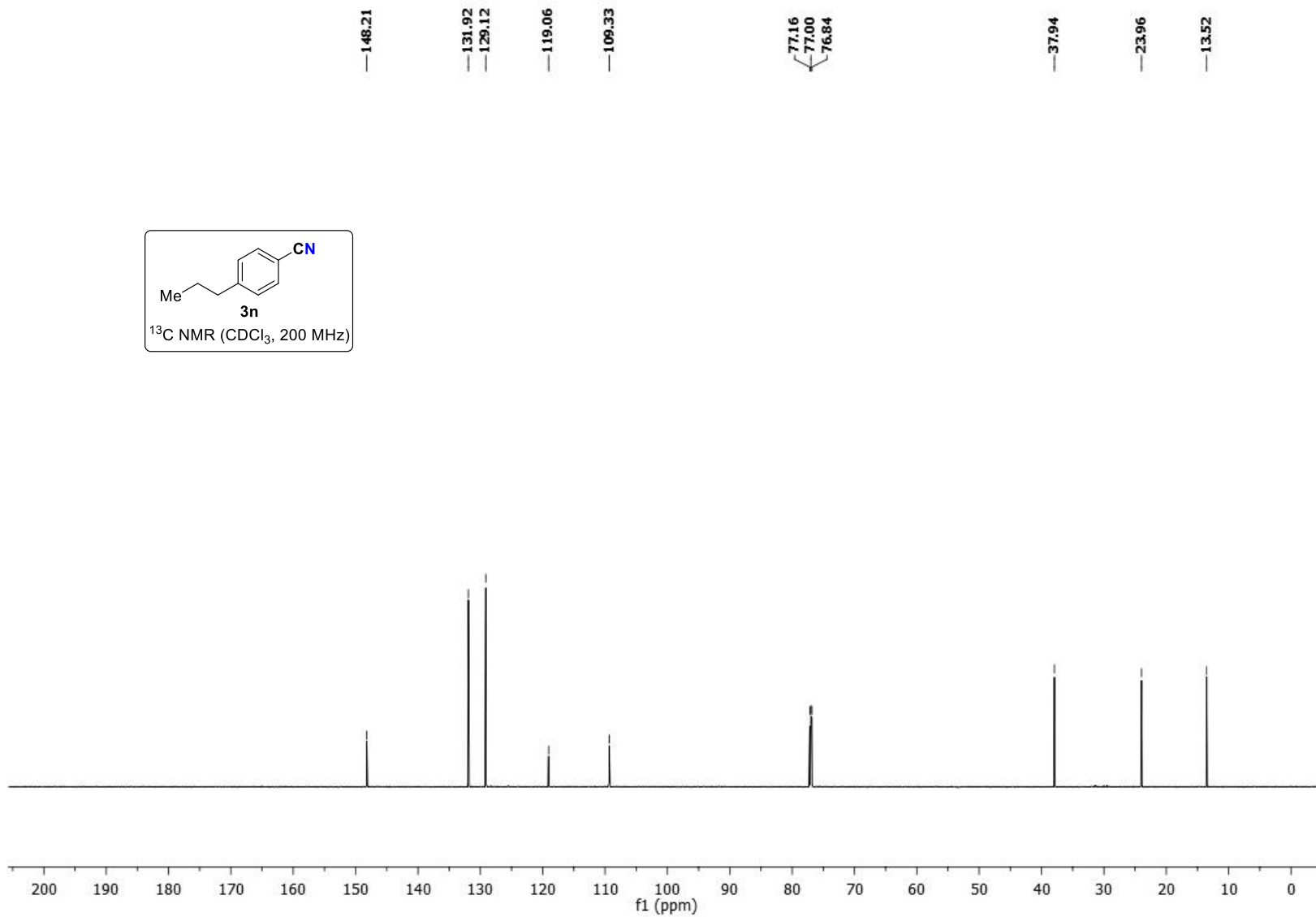
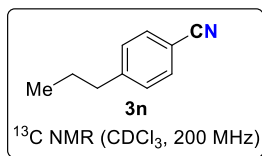


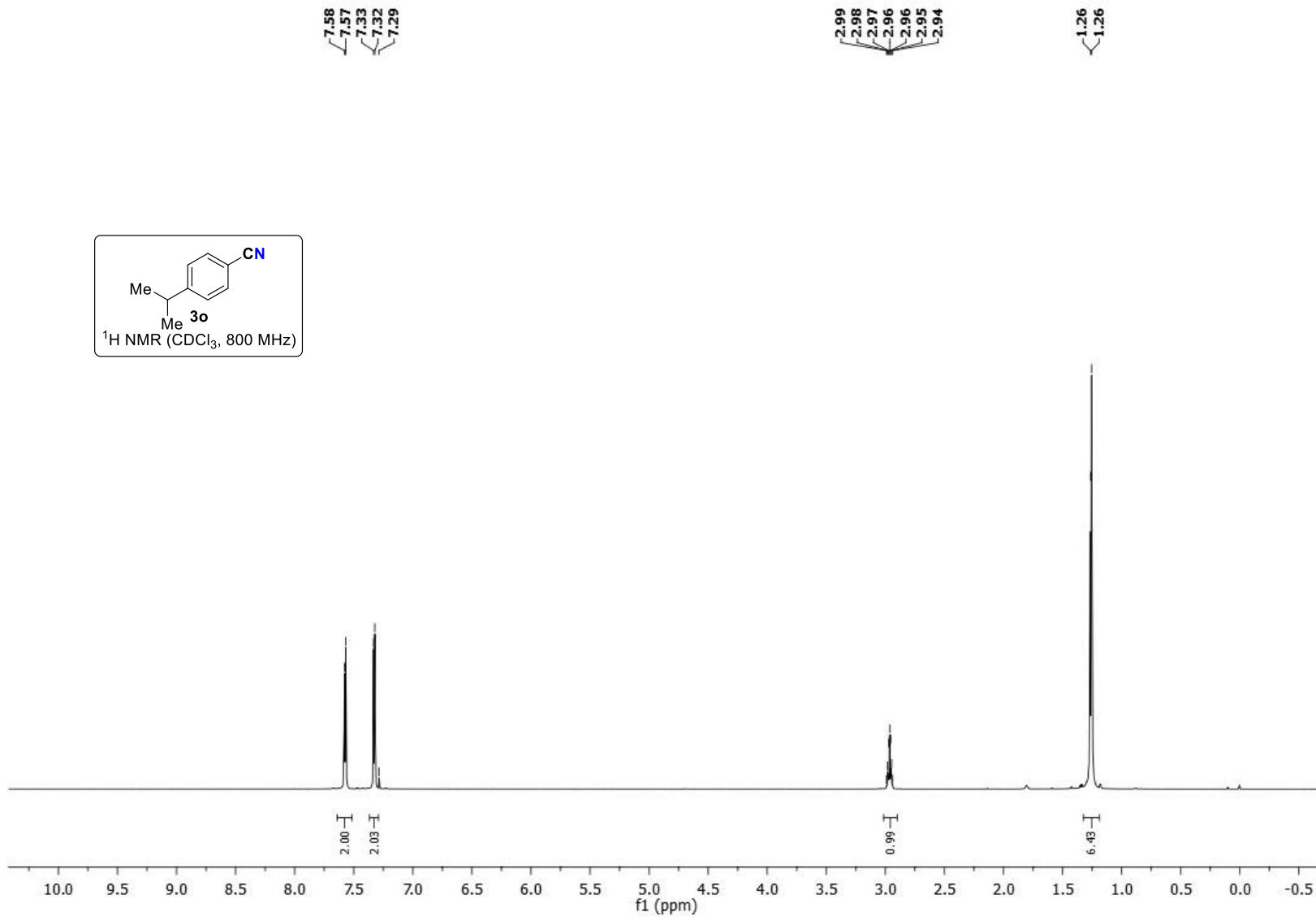
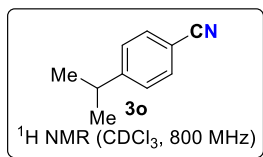


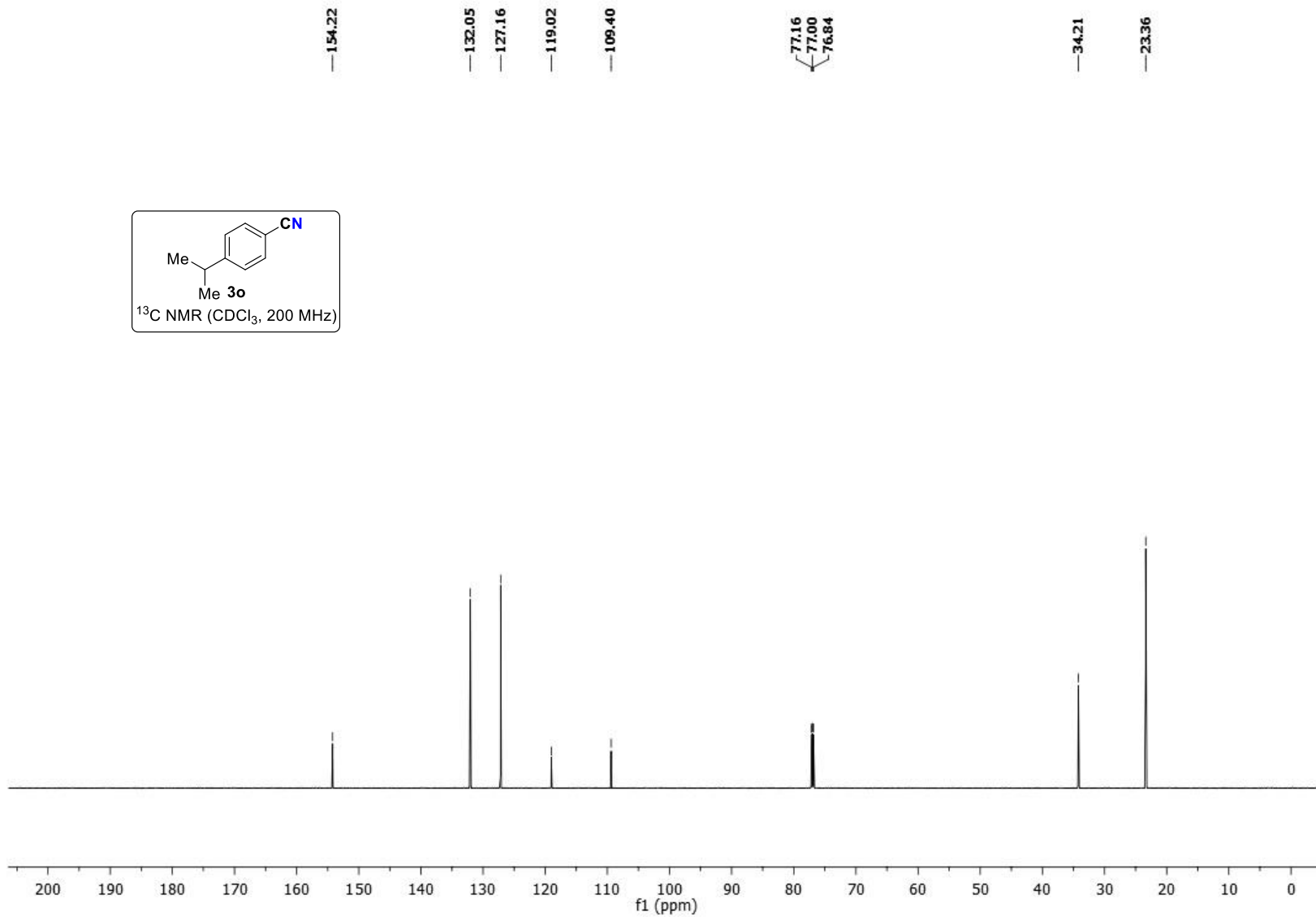
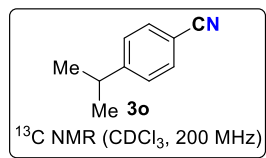


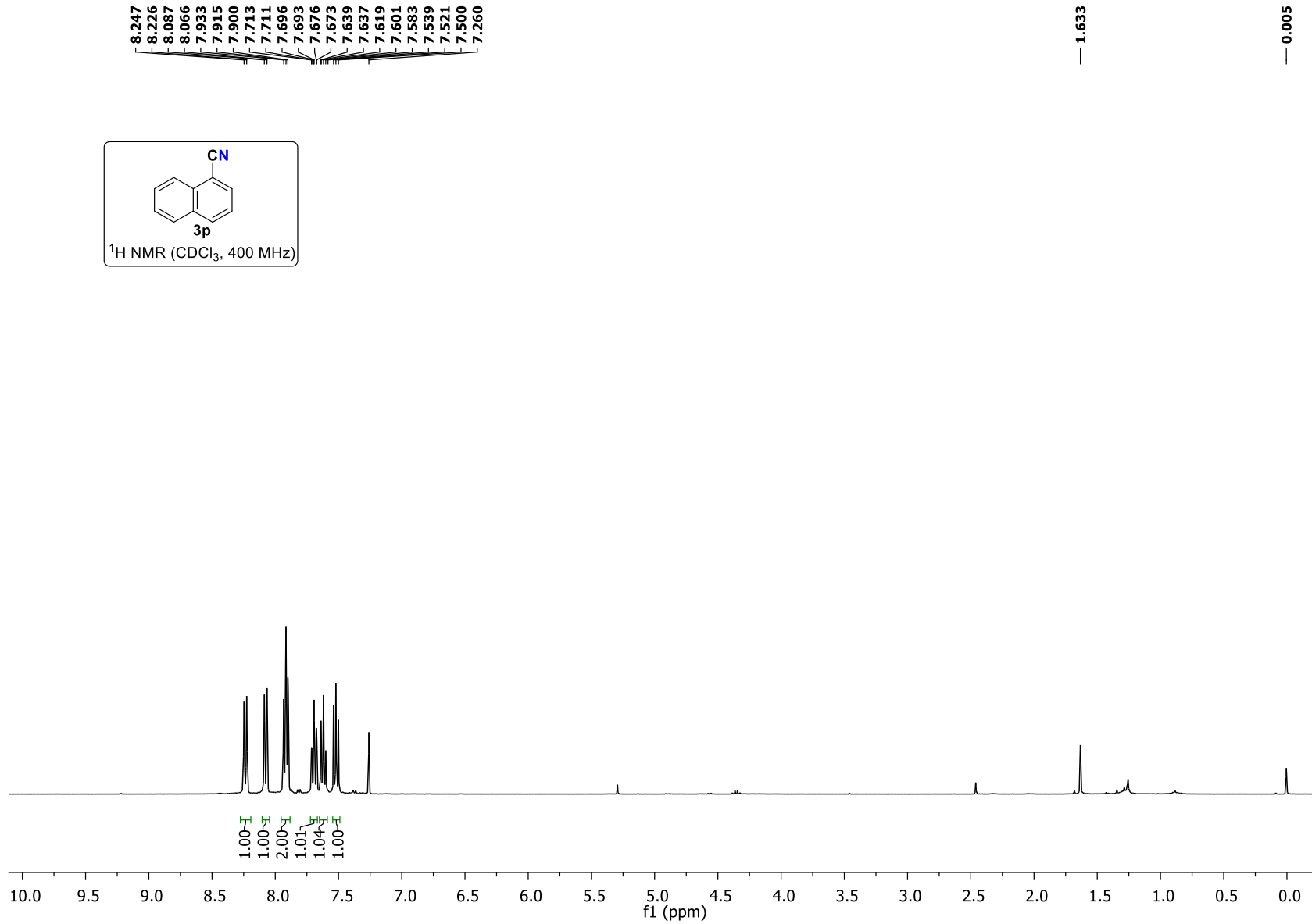


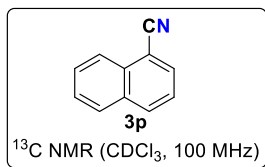






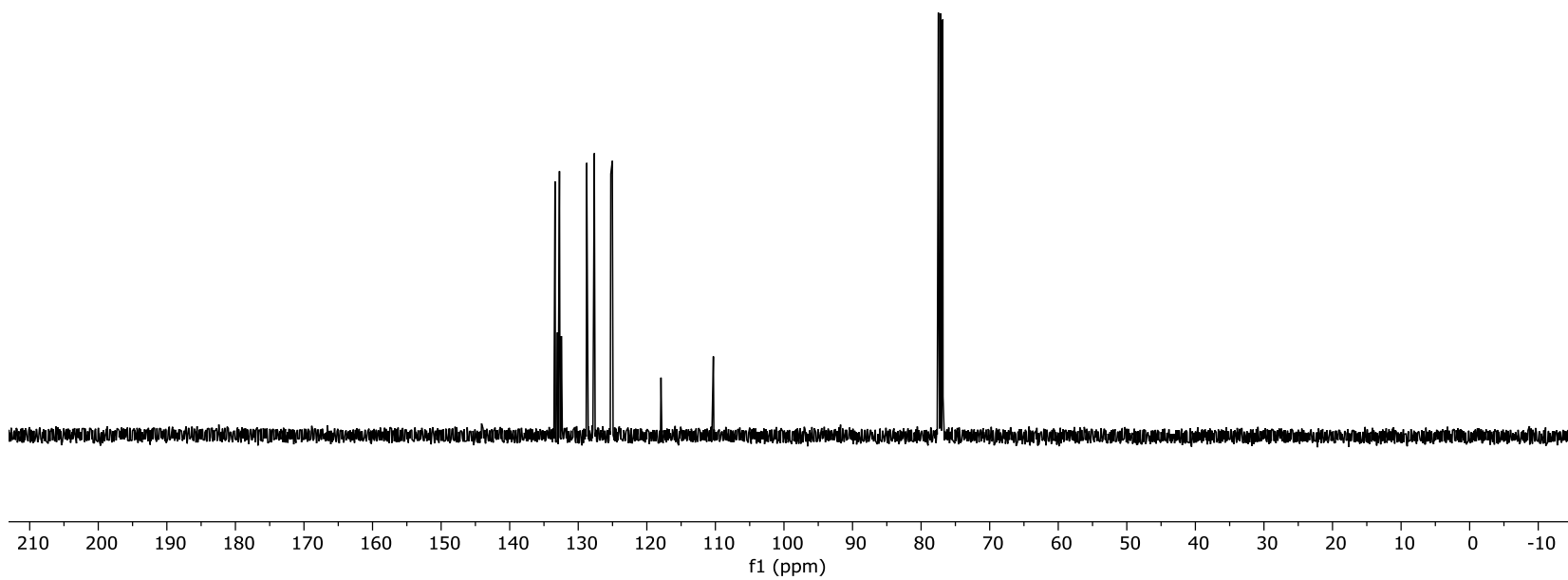




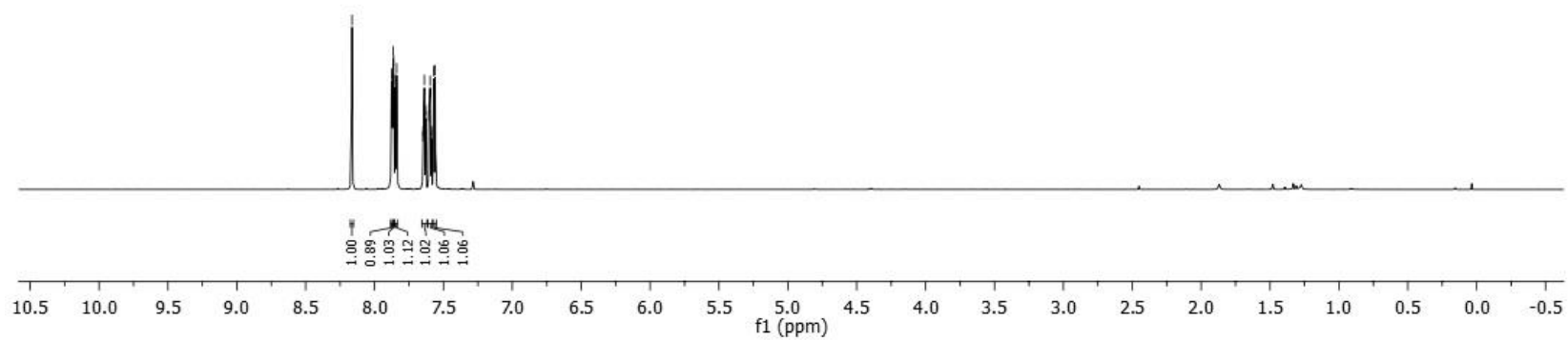
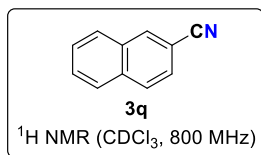


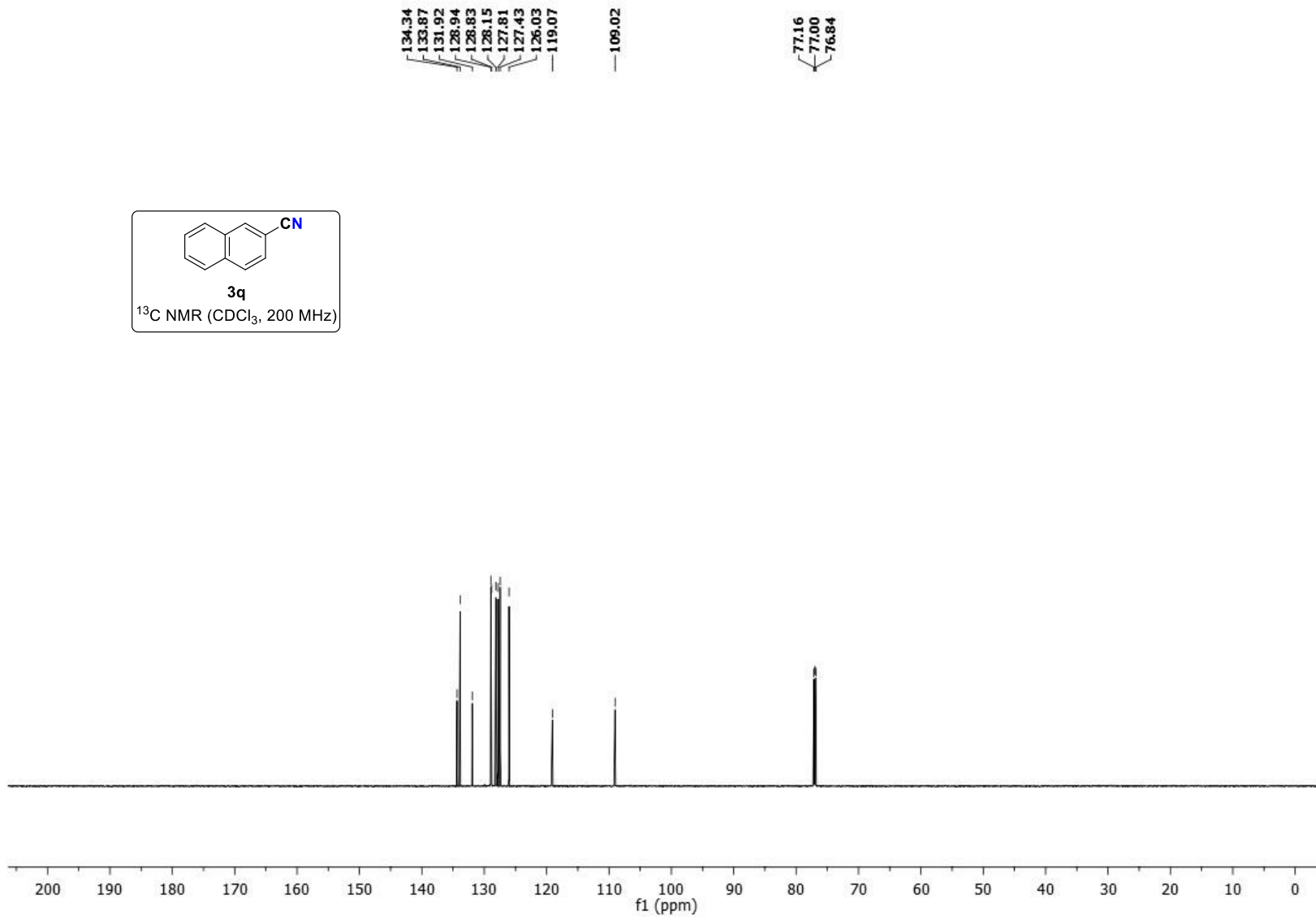
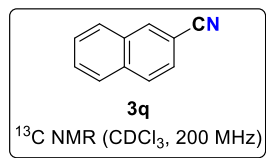
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127.66
125.25
125.03
117.93
110.29

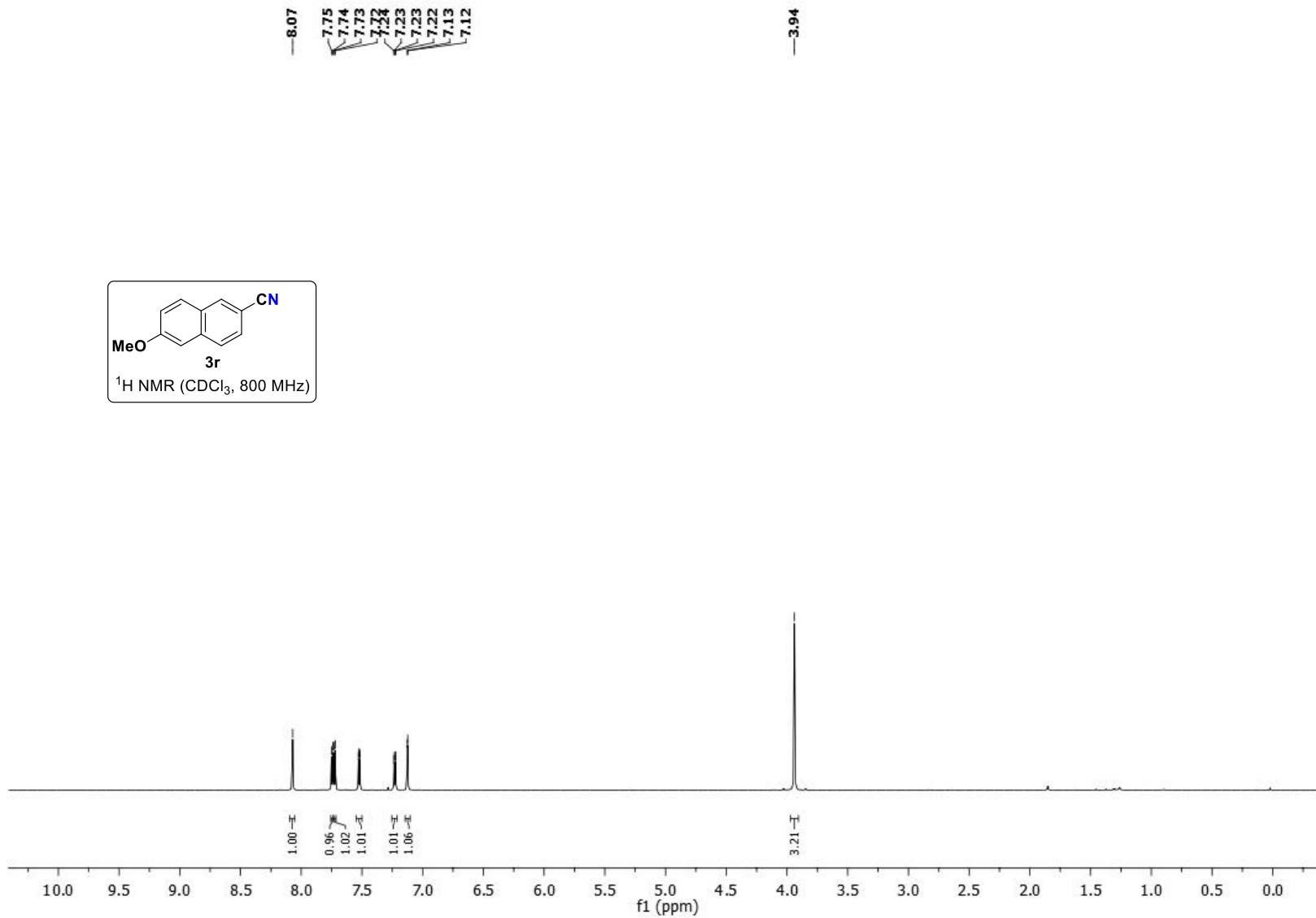
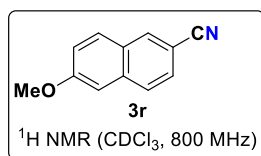
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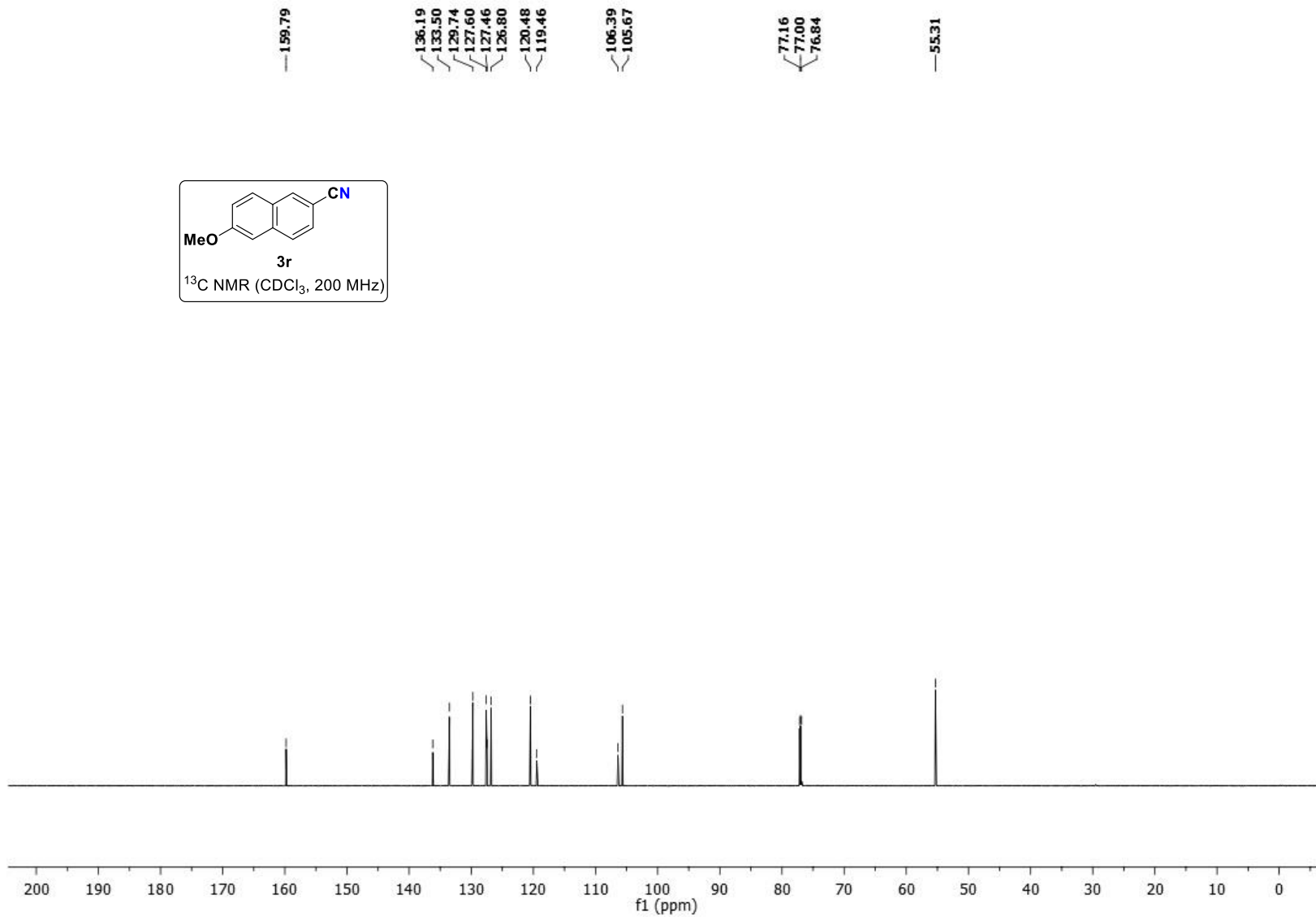
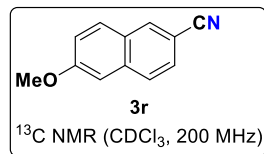


8.16
7.88
7.87
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7.84
7.65
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7.64
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7.56
7.56

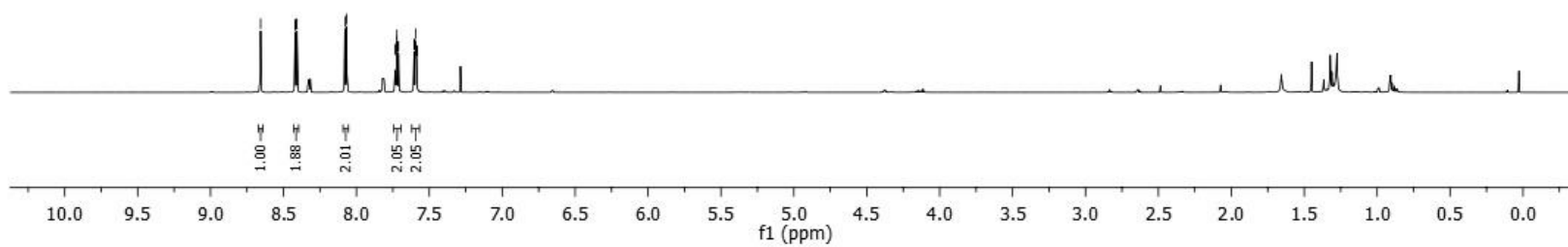
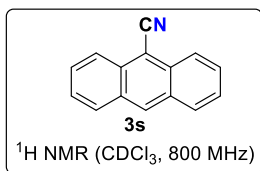


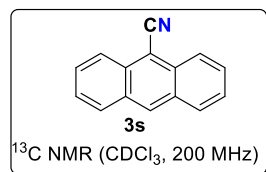






8.66
8.42
8.42
8.41
8.41
8.08
8.07
7.73
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7.72
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7.71
7.71
7.60
7.60
7.59
7.59
7.58

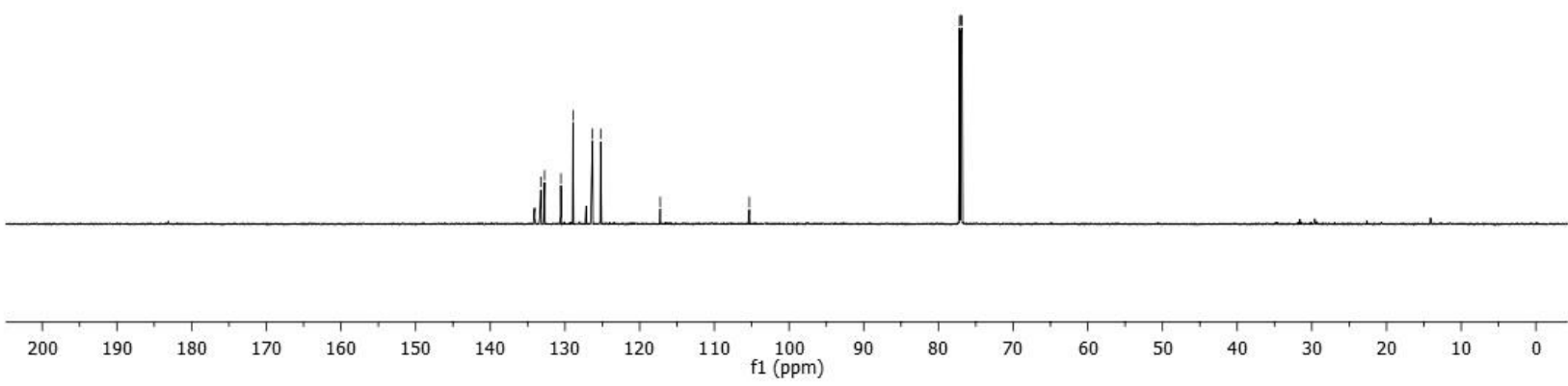




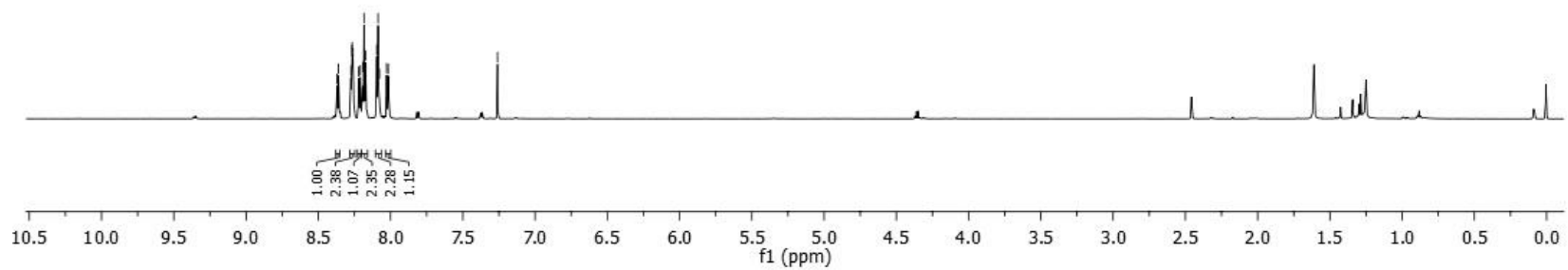
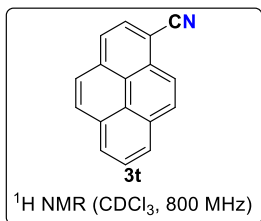
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125.22

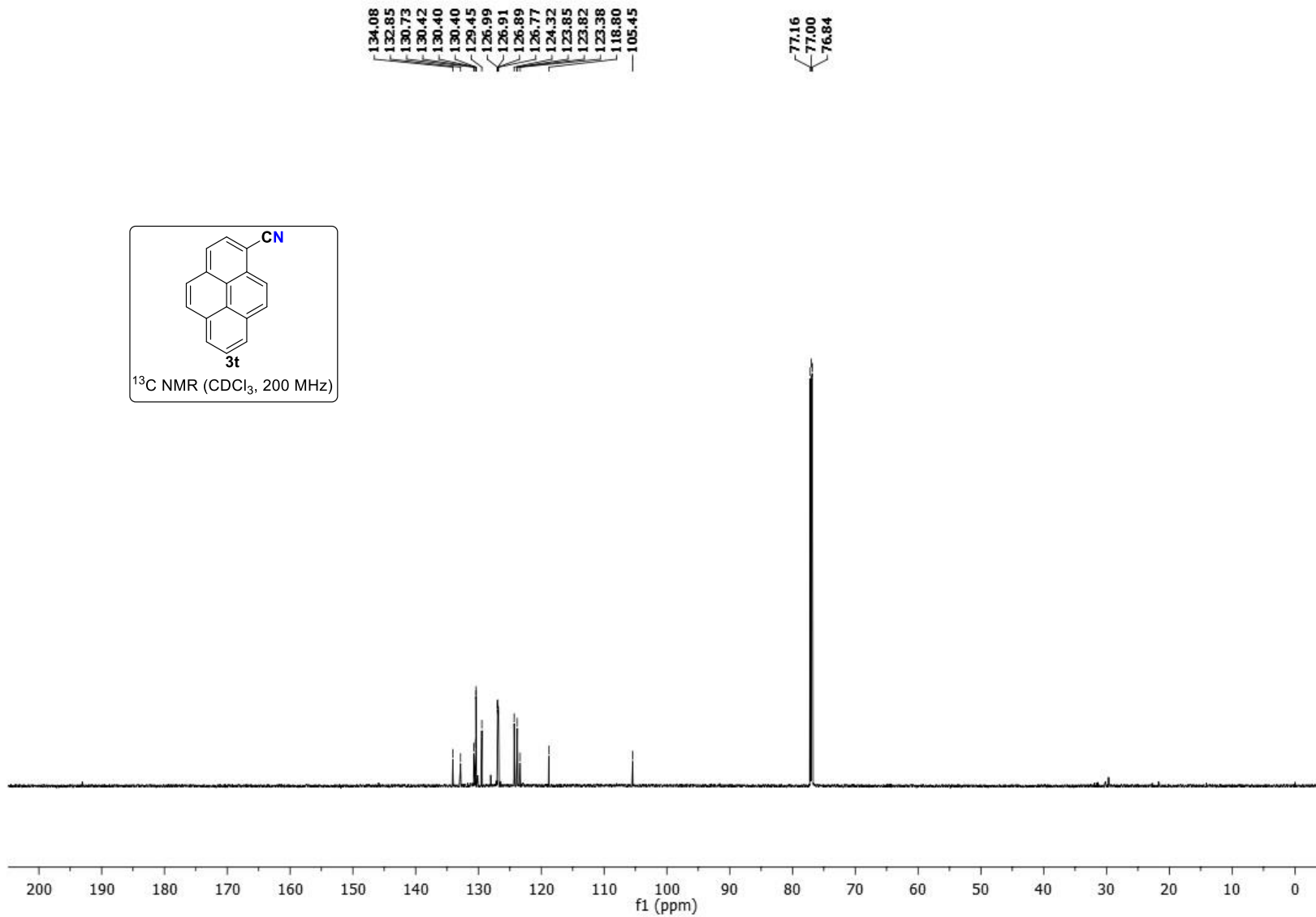
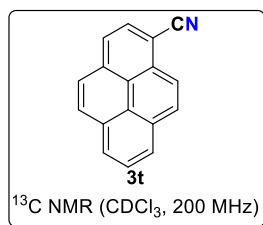
105.32

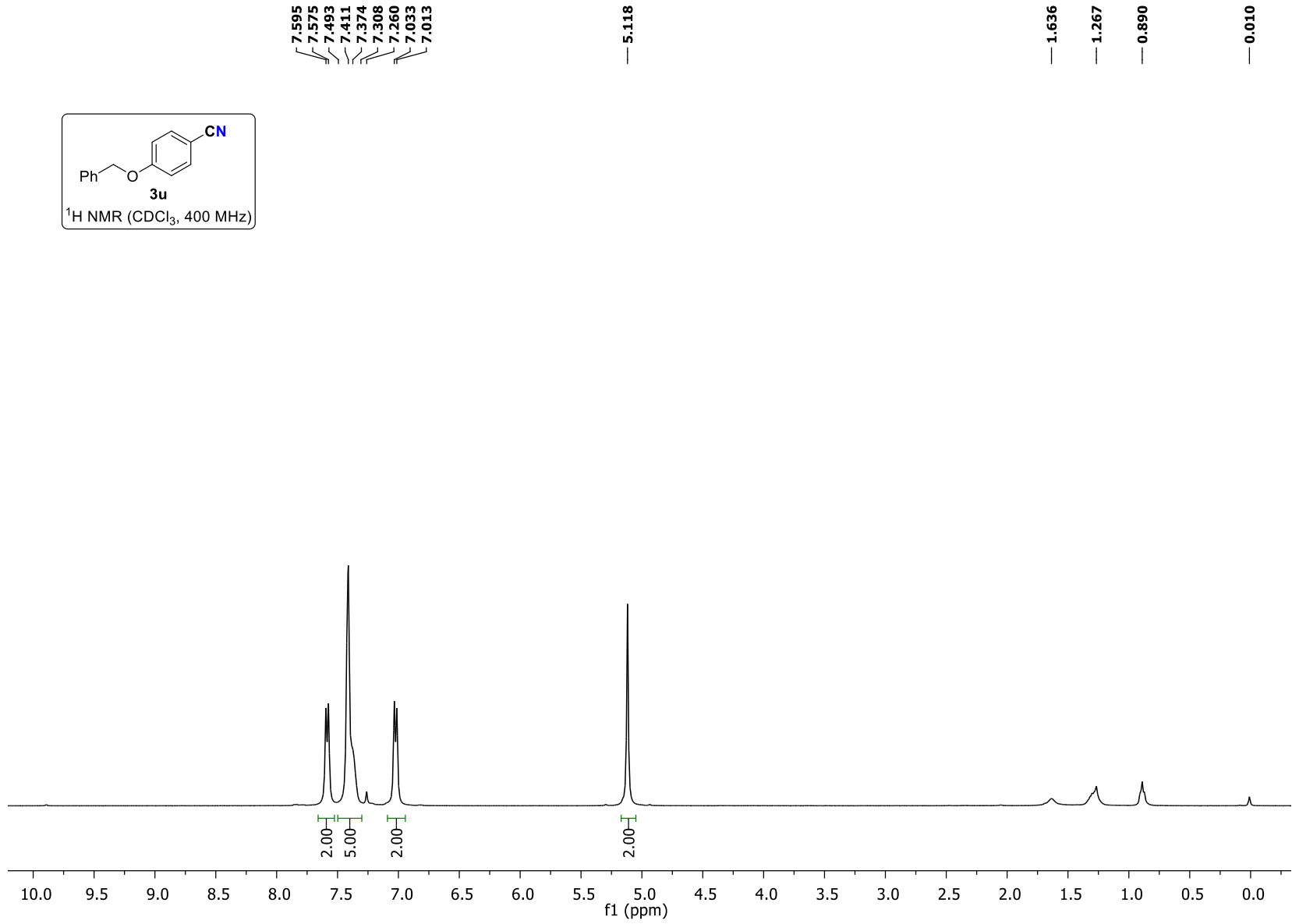
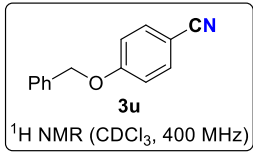
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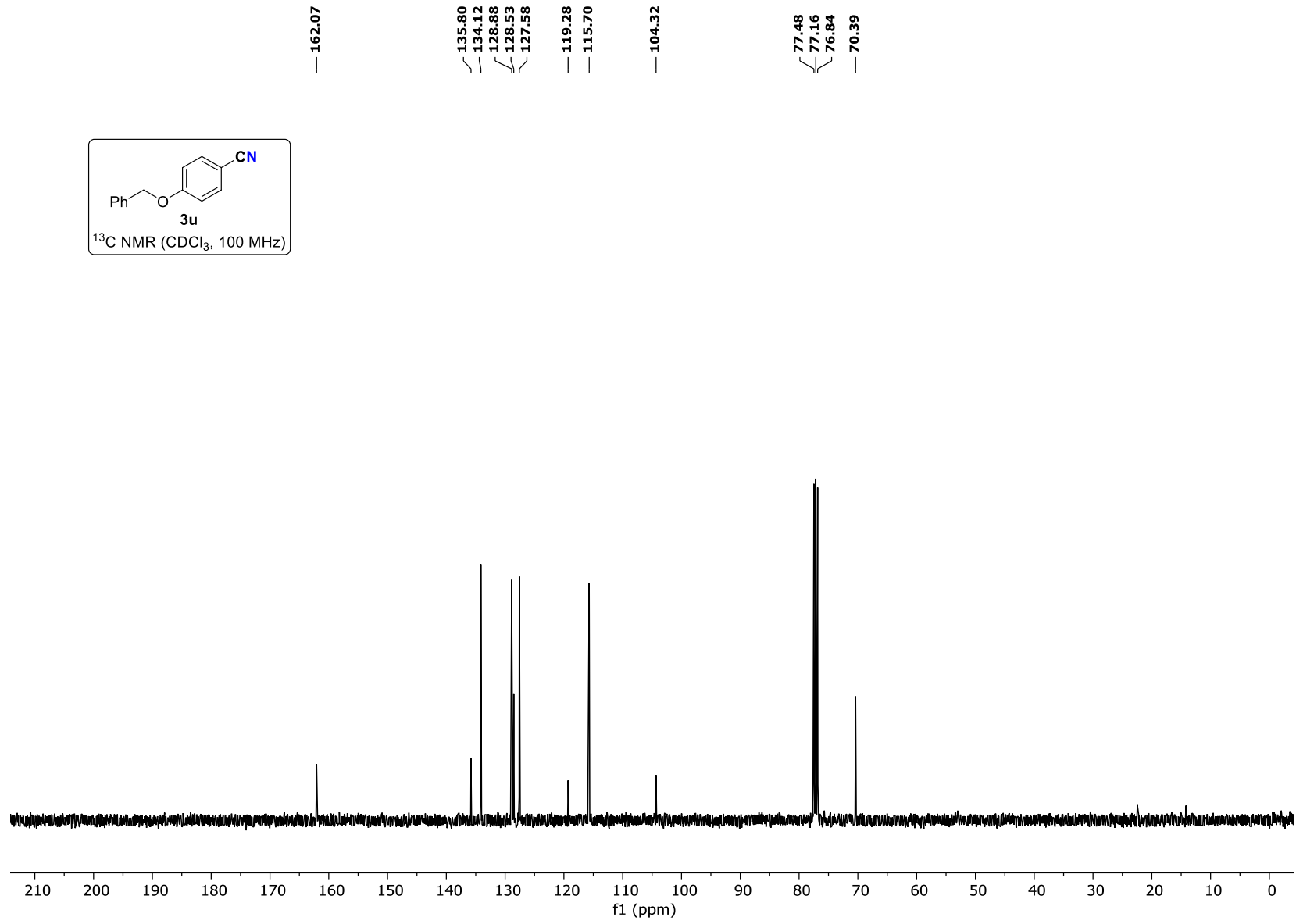
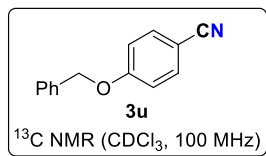


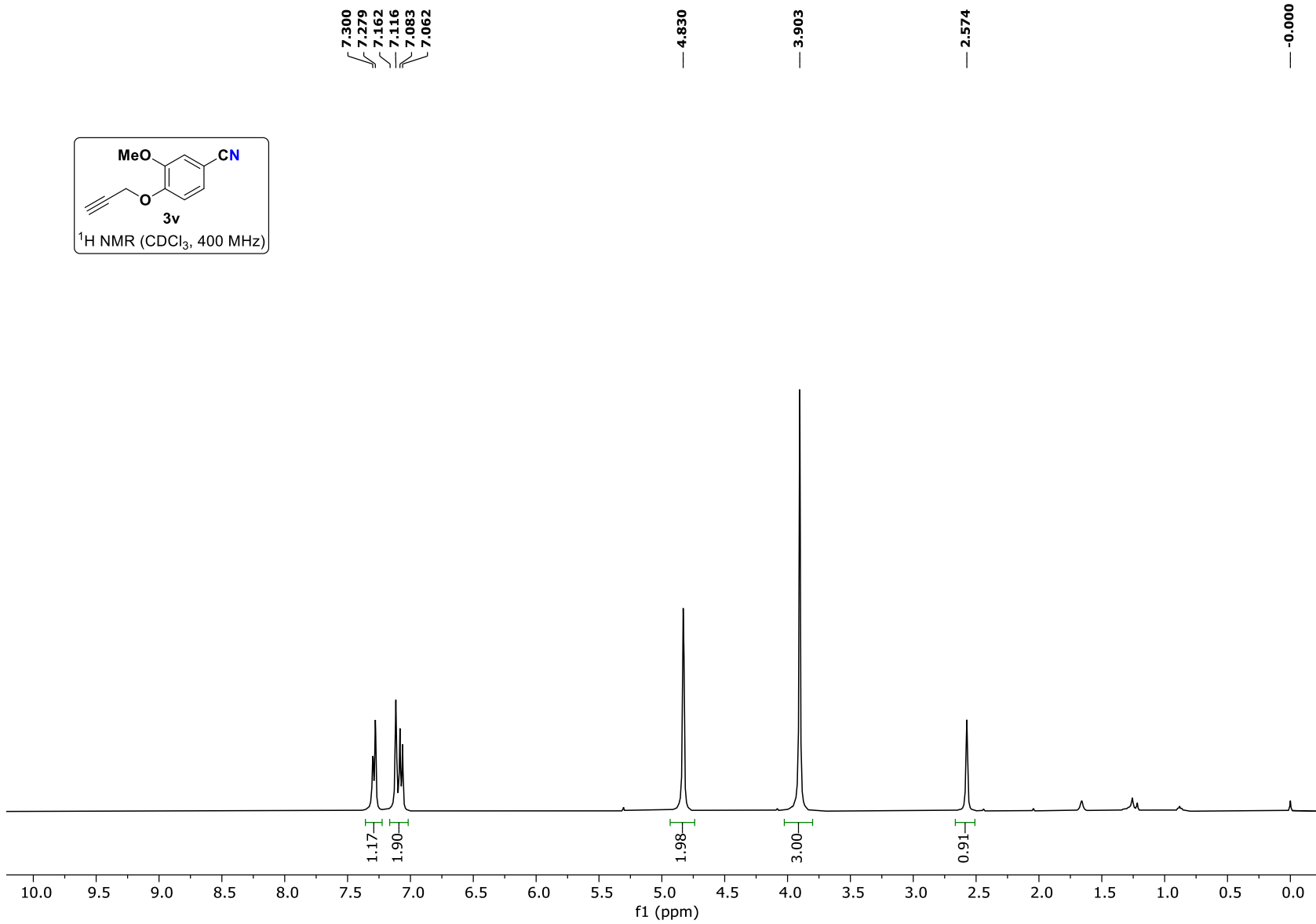
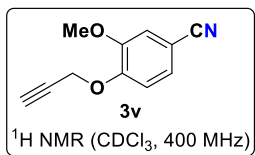
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8.17
8.10
8.09
8.08
8.03
8.02
7.26



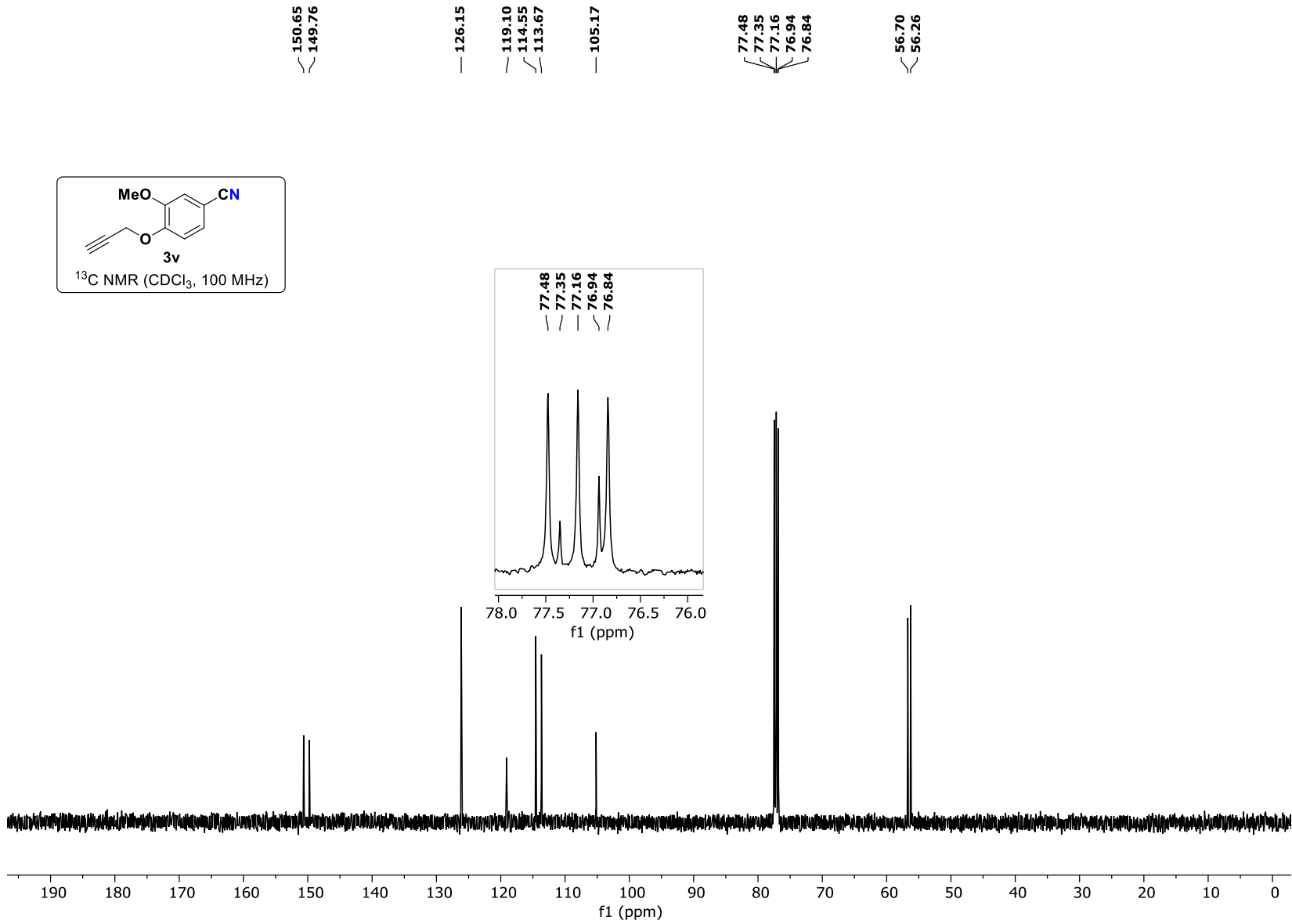


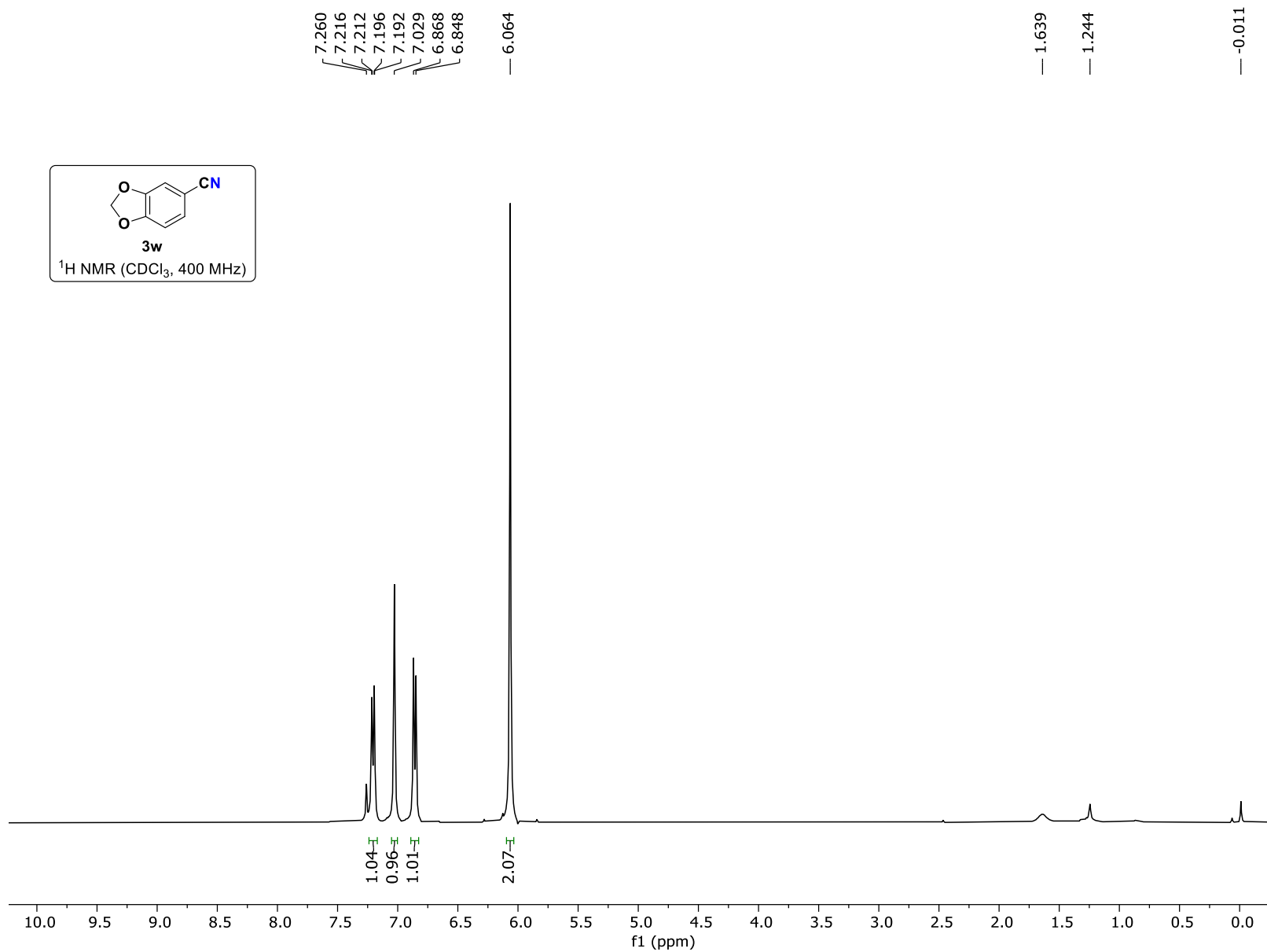
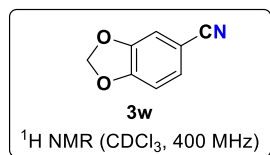


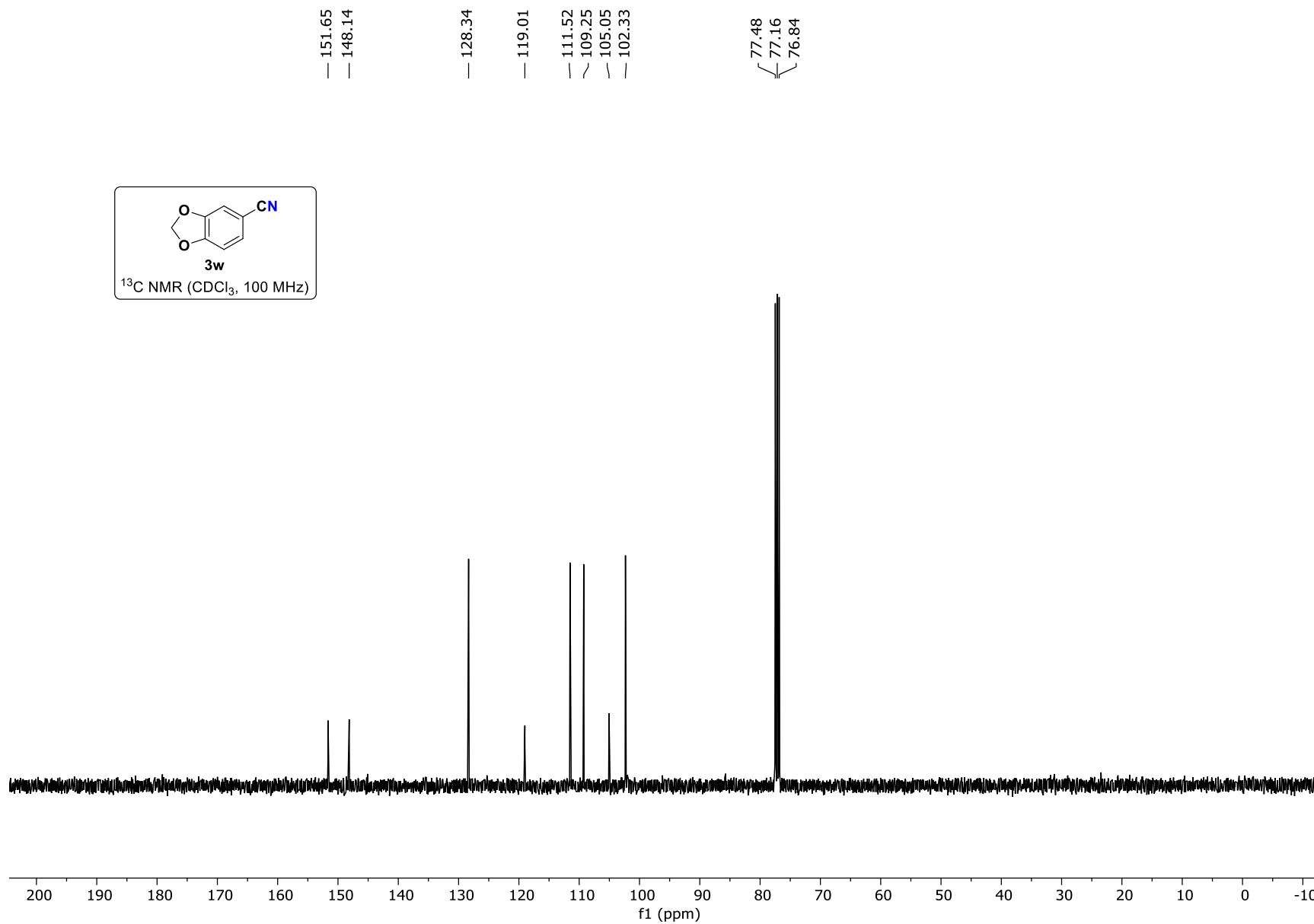
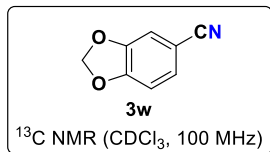




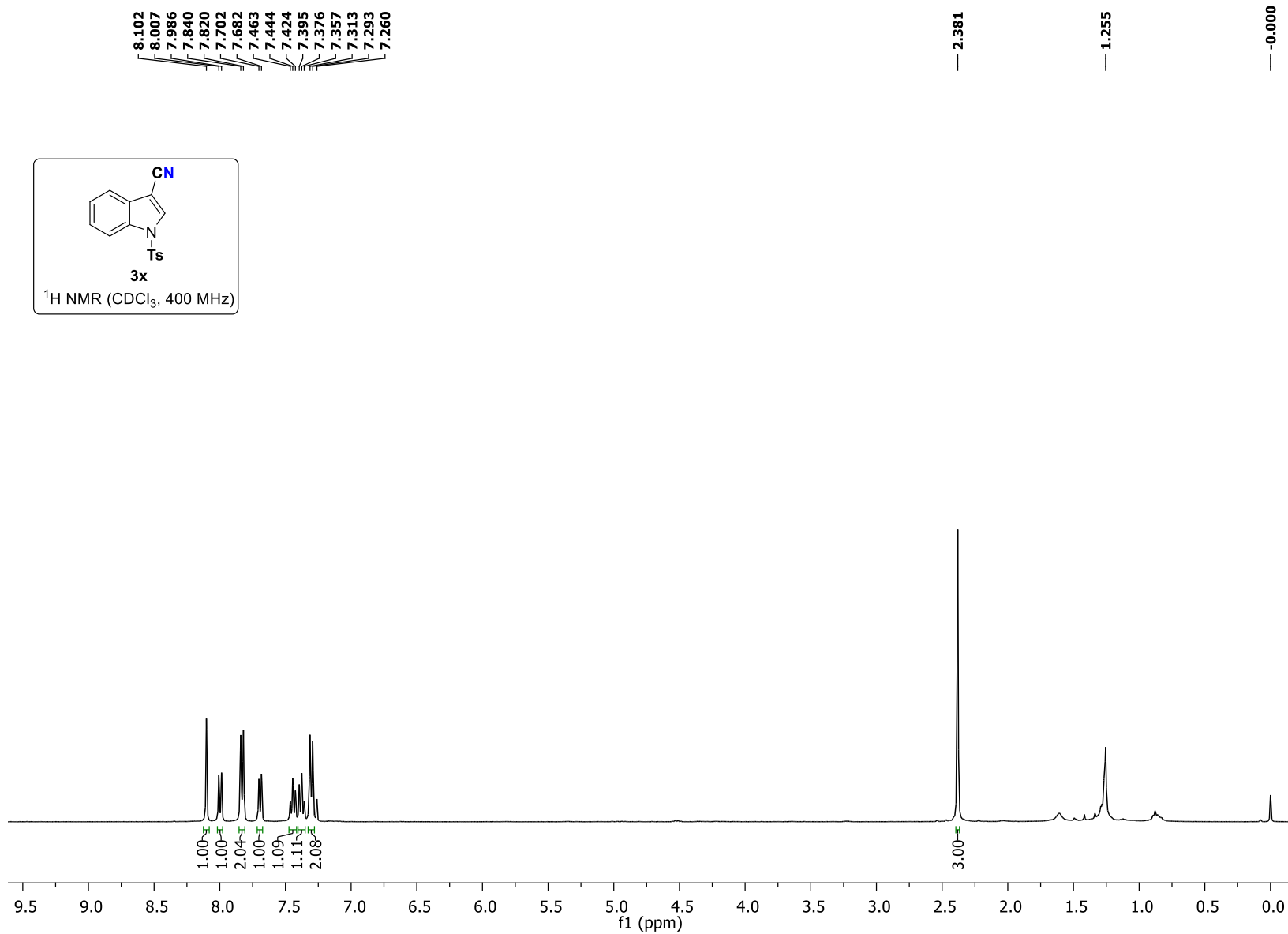
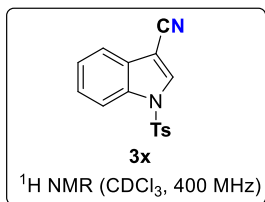
SI-55

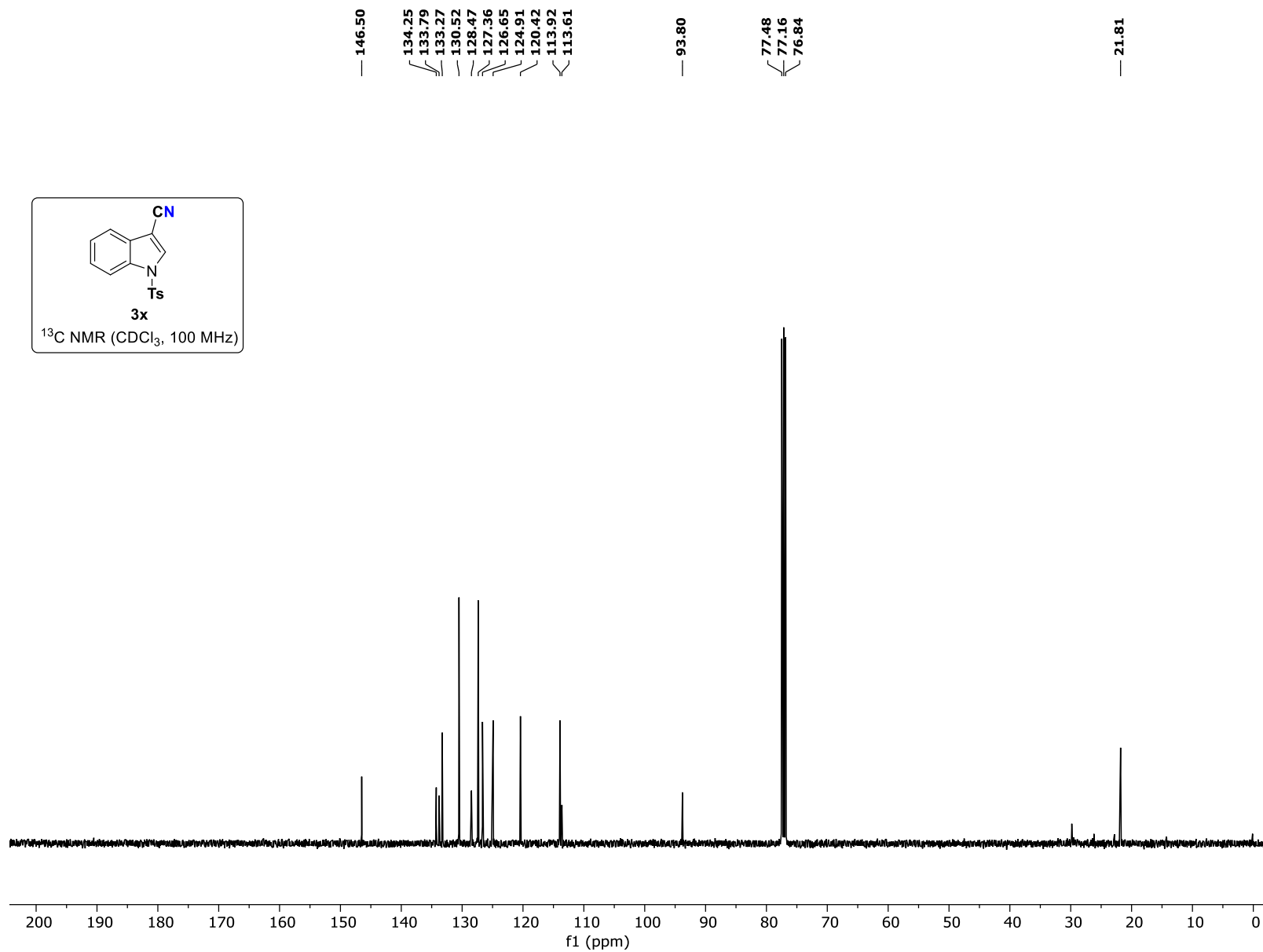
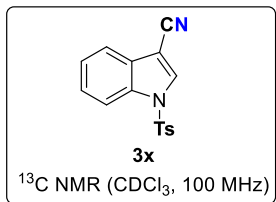


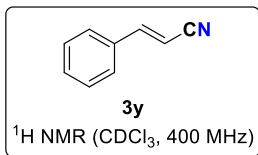




SI-58



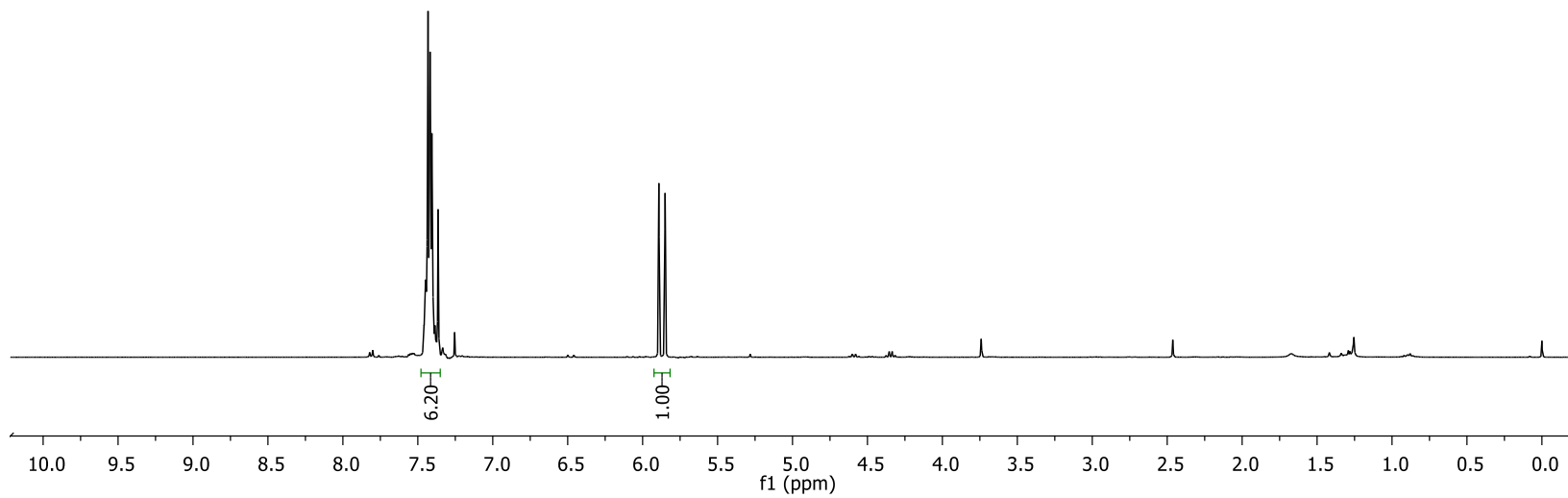


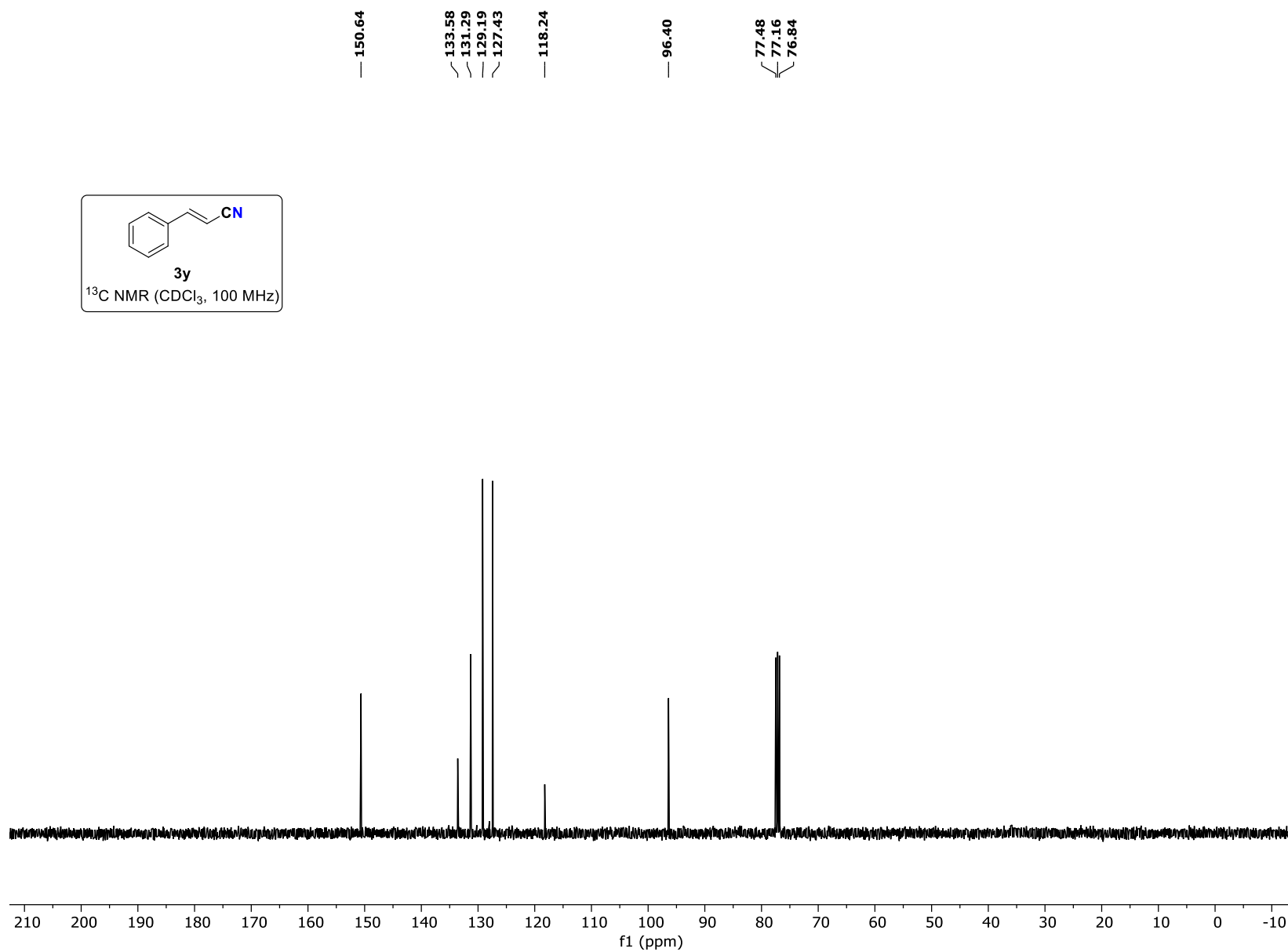
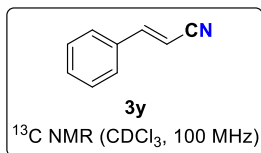


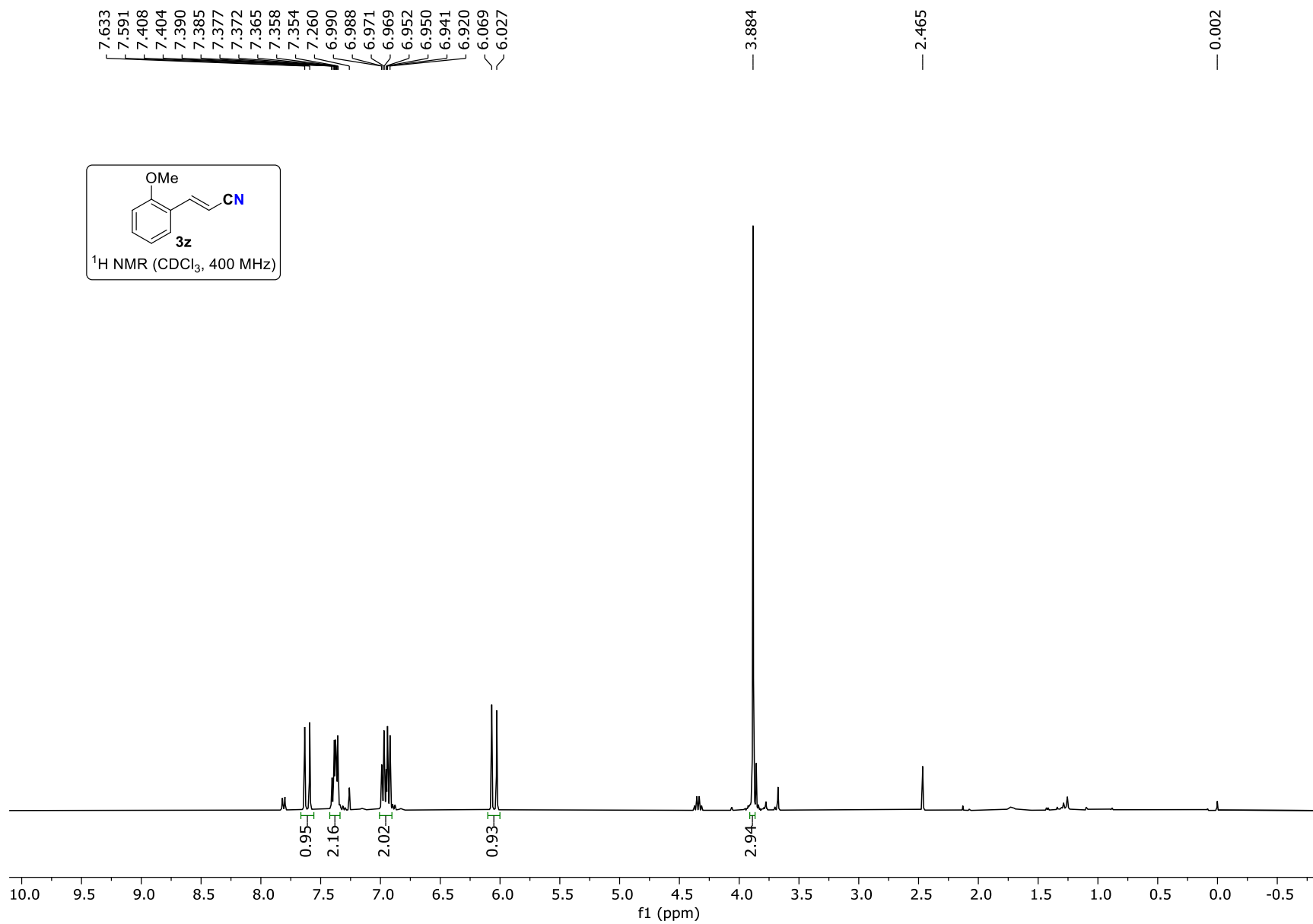
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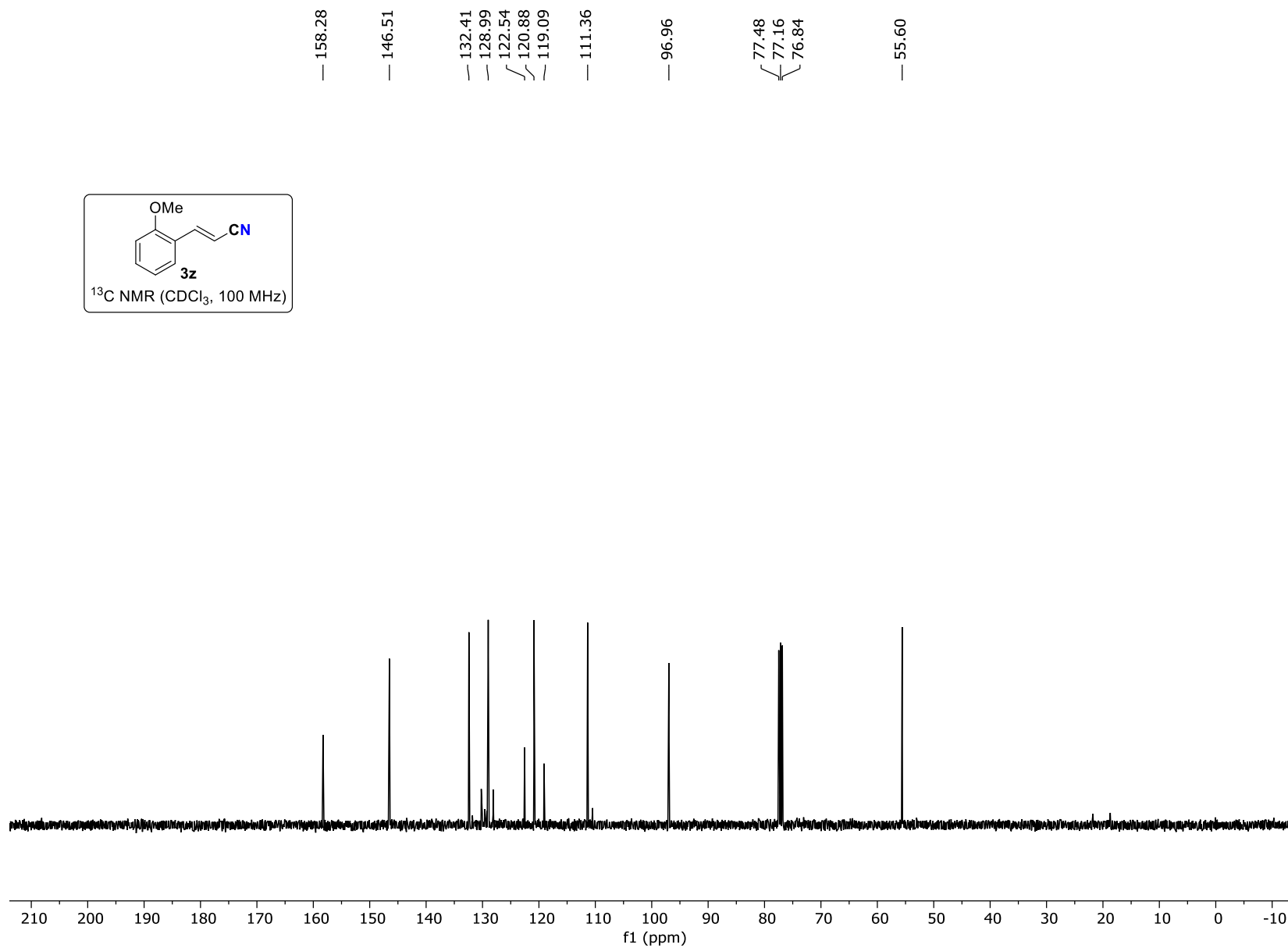
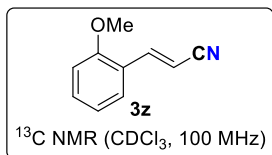
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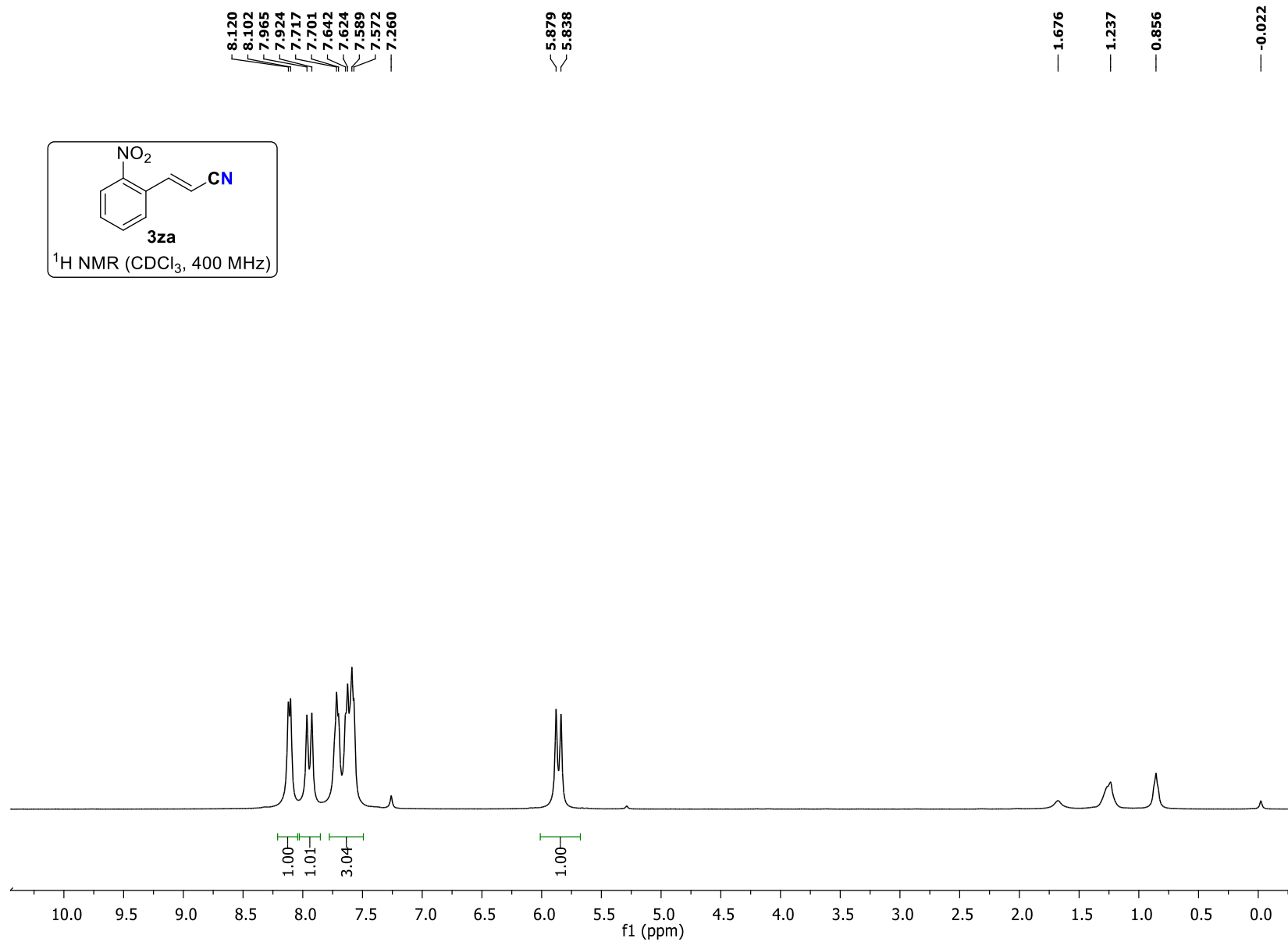
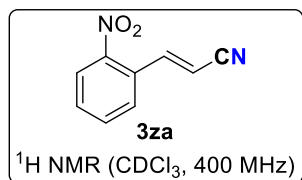


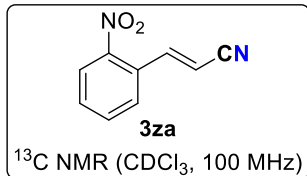




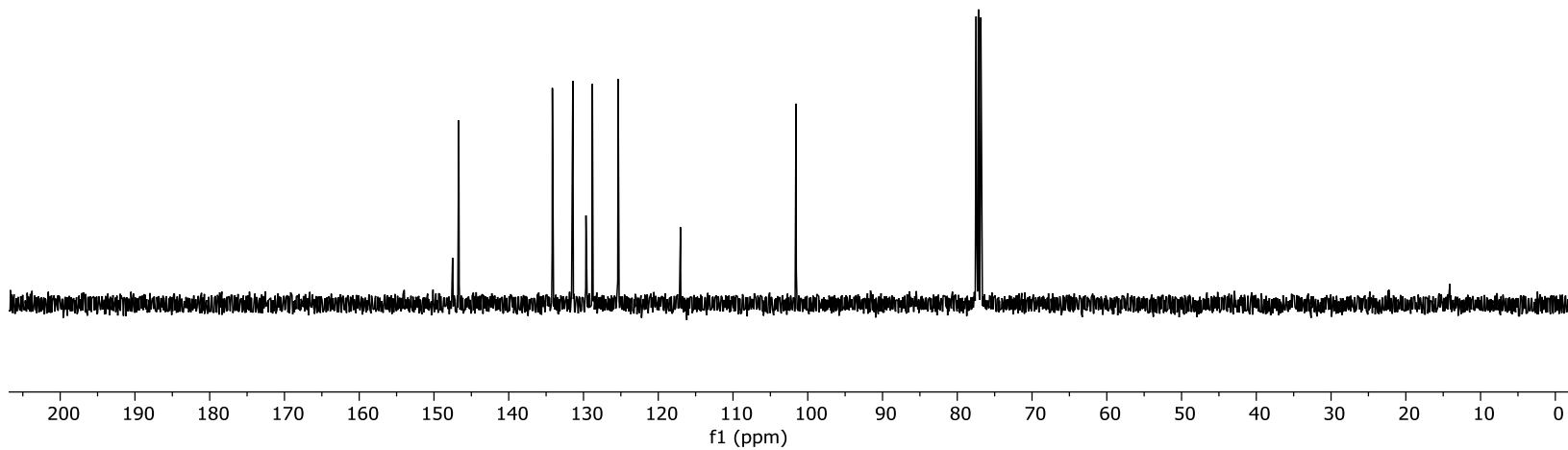
SI-63

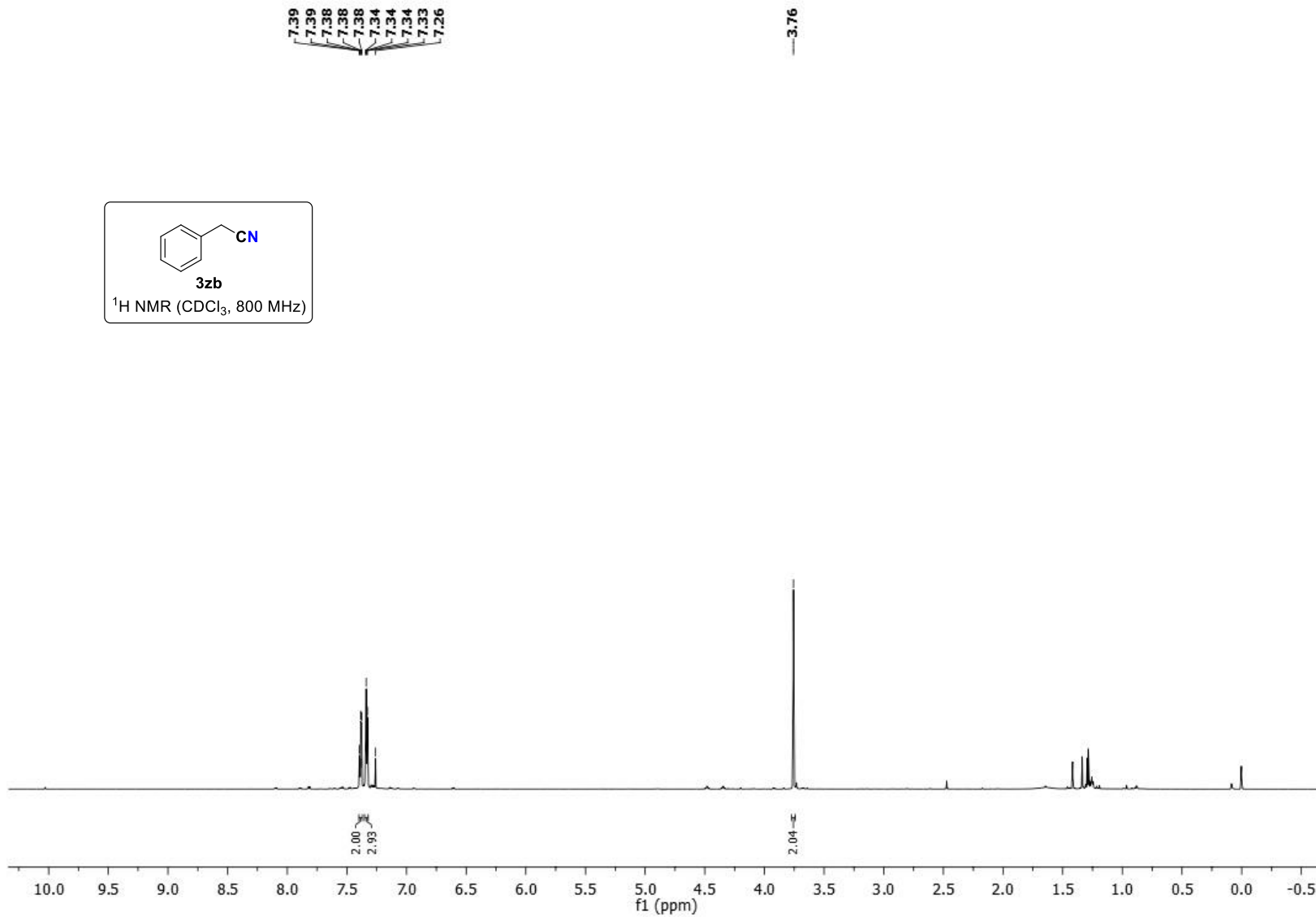
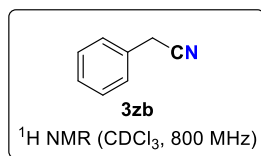


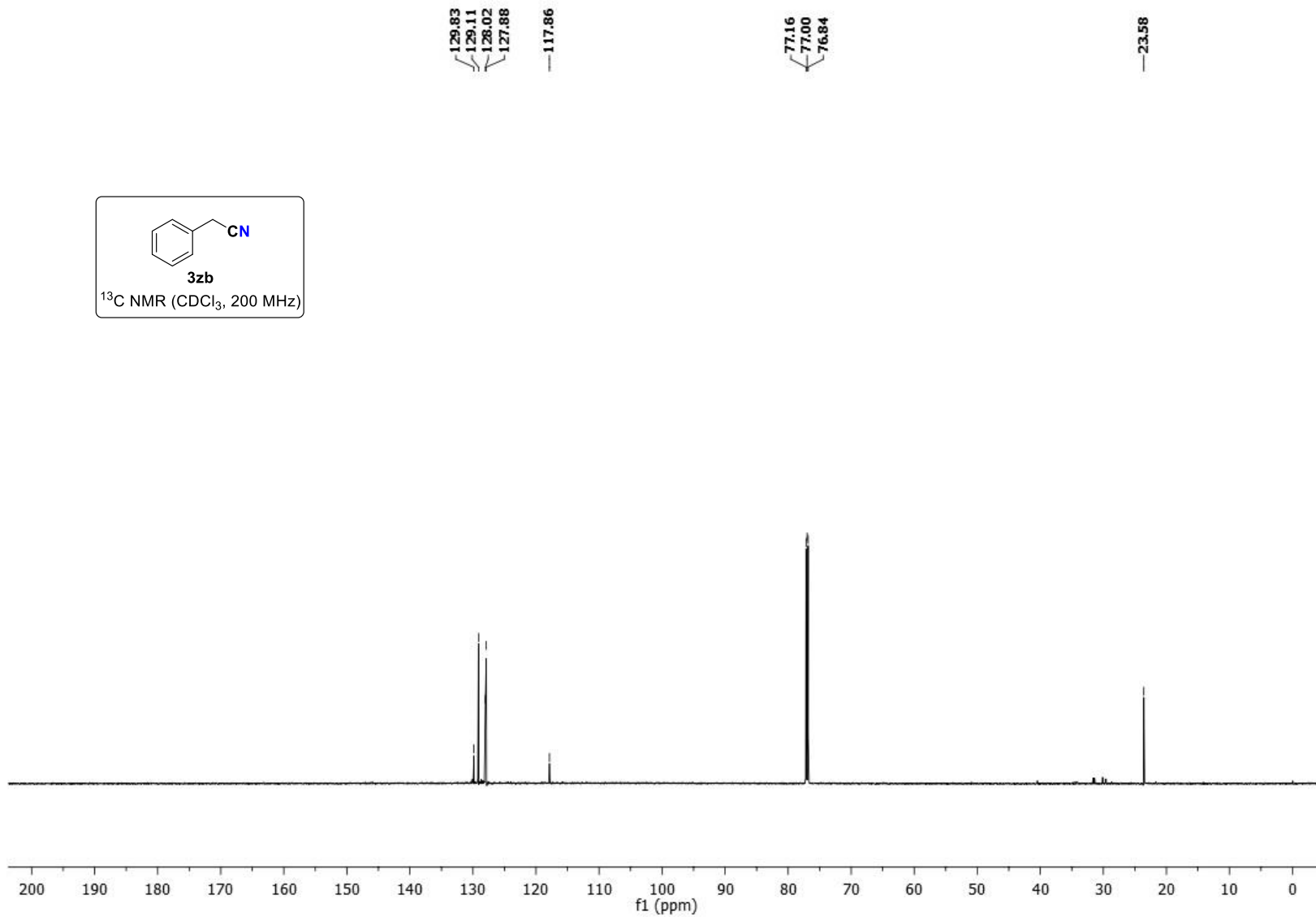
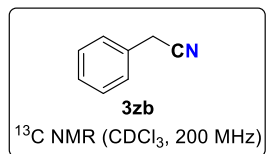




147.52
146.69
134.16
131.43
129.67
128.81
125.36
117.04
101.57
77.48
77.16
76.84







7.363
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7.225

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— 1.567

— 1.254

— 0.882

— -0.000

