Supporting Information

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

Puneet Kumar,[a] Vikram Singh,[b] Jawahar L. Jat[†*a] and Bhoopendra Tiwari[‡*b]

[†]Department of Chemistry, Babasaheb Bhimrao Ambedkar University, Vidya Vihar, Raebareli Road, Lucknow, India, Email: jawaharlj@bbau.ac.in, jatjawahar@gmail.com

[‡]Division of Molecular Synthesis & Drug Discovery, Centre of Biomedical Research, SGPGIMS-Campus, Raebareli Road, Lucknow, India, Email: btiwari@cbmr.res.in

List of Contents:

1. General Information……..……...……SI-2
2. General procedure for the preparation of nitriles from aldehydes…….…….…….SI-2
3. Characterization data of the products.……..…….…….…….....….SI-2
4. References……...……...…….……...…….SI-11
5. ^{1}H NMR Spectra of the products …….……..……..…...……...SI-13

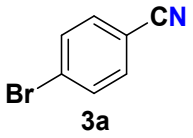
EXPERIMENTAL SECTION

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, F_{254}) 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. ^{1}H and ^{13}C NMR spectra were recorded at 400 or 800 MHz and 100 or 200 MHz instruments respectively in $CDCl_3$. Chemical shifts (δ) are given in ppm. The residual solvent signals were used as references ($CDCl_3$: 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), Cs_2CO_3 (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 Na_2SO_4. 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:

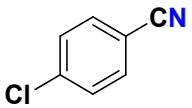


3a

4-bromobenzonitrile (3a): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H).

^{13}C NMR (200 MHz, $CDCl_3$) δ 133.16, 132.33, 127.70, 117.80, 110.90.

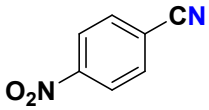


3b

4-chlorobenzonitrile (3b): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, J = 8.4 Hz, 2H), 7.46 (d, J = 8.4 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.65, 133.48, 129.80, 118.05, 110.90.

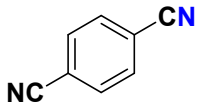


3c

4-nitrobenzonitrile (3c): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (d, J = 8.6 Hz, 2H), 7.89 (d, J = 8.6 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 150.13, 133.60, 124.40, 118.43, 116.92.

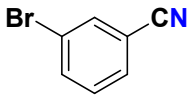


3d

Terephthalonitrile (3d): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (s, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 132.91, 117.13, 116.83.

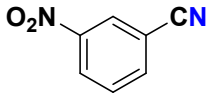


3e

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

^{1}H NMR (400 MHz, $CDCl_3$) 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).

^{13}C NMR (200 MHz, $CDCl_3$) δ 135.71, 134.22, 130.36, 130.33, 122.41, 116.88, 113.67.

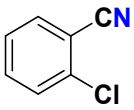


3f

3-nitrobenzonitrile (3f): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) 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).

^{13}C NMR (100 MHz, $CDCl_3$) δ 148.30, 137.71, 130.78, 127.61, 127.28, 116.62, 114.17.

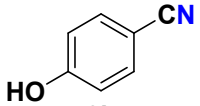


3g

2-chlorobenzonitrile (3g): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.59 – 7.47 (m, 2H), 7.42 – 7.33 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.95, 134.11, 133.99, 130.15, 127.26, 116.05, 113.48.

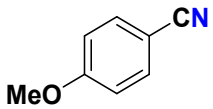


3h

4-hydroxybenzonitrile (3h): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 160.28, 134.46, 119.38, 116.59, 103.23.

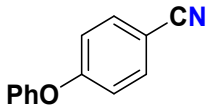


3i

4-methoxybenzonitrile (3i): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) 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.

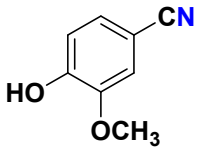


3j

4-phenoxybenzonitrile (3j): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.

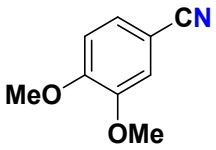


3k

4-hydroxy-3-methoxybenzonitrile (3k): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.

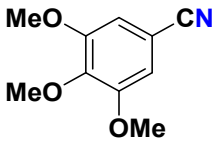


3l

3,4-dimethoxybenzonitrile (3l): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.

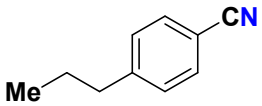


3m

3,4,5-trimethoxybenzonitrile (3m): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.83 (s, 2H), 3.86 (s, 3H), 3.84 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.58, 142.34, 118.99, 109.47, 106.72, 61.05, 56.41.

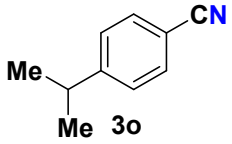


3n

4-propylbenzonitrile (**3n**): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (800 MHz, $CDCl_3$) 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).

^{13}C NMR (200 MHz, $CDCl_3$) δ 148.21, 131.92, 129.12, 119.06, 109.33, 37.94, 23.96, 13.52.

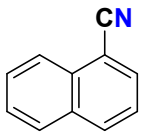


3o

4-isopropylbenzonitrile (**3o**): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (800 MHz, $CDCl_3$) 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).

^{13}C NMR (200 MHz, $CDCl_3$) δ 154.22, 132.05, 127.16, 119.02, 109.40, 34.21, 23.36.

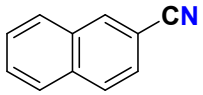


3p

1-naphthonitrile (**3p**): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.

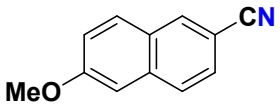


3q

2-naphthonitrile (**3q**): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.

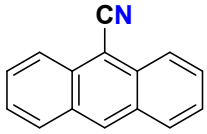


3r

6-methoxy-2-naphthonitrile (**3r**): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.

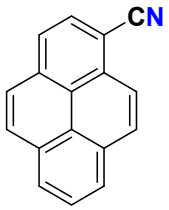


3s

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

^{1}H 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.

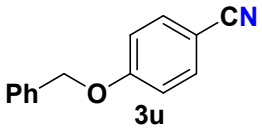


3t

Pyrene-1-carbonitrile (**3t**): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (800 MHz, $CDCl_3$) 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).

^{13}C NMR (200 MHz, $CDCl_3$) 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.

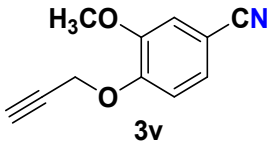


3u

4-(benzyloxy)benzonitrile (3u): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) 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).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.07, 135.80, 134.12, 128.88, 128.53, 127.58, 119.28, 115.70, 104.32, 70.39.



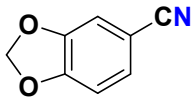
3v

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

^{1}H NMR (400 MHz, $CDCl_3$) 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).

^{13}C NMR (100 MHz, $CDCl_3$) 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 $[C_{11}H_{10}NO_2]^+$ 188.0706, found 188.0712.

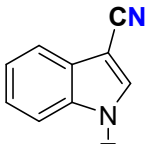


3w

Benzo[*d*][1,3]dioxole-5-carbonitrile (3w): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.17 (m, 1H), 7.03 (s, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.06 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 151.65, 148.14, 128.34, 119.01, 111.52, 109.25, 105.05, 102.33.

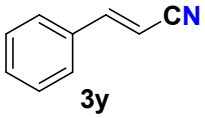


3x

1-tosyl-1*H*-indole-3-carbonitrile (3x): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) 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).

^{13}C NMR (100 MHz, $CDCl_3$) 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.

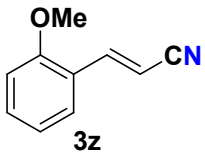


3y

Cinnamonitrile (3y): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.37 (m, 6H), 5.87 (d, *J* = 16.7 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 150.64, 133.58, 131.29, 129.19, 127.43, 118.24, 96.40.

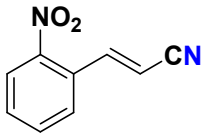


3z

(*E*)-3-(2-methoxyphenyl)acrylonitrile (3z): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) 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).

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.28, 146.51, 132.41, 128.99, 122.54, 120.88, 119.09, 111.36, 96.96, 55.60.

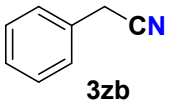


3za

(*E*)-3-(2-nitrophenyl)acrylonitrile (3za): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) 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).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.52, 146.69, 134.16, 131.43, 129.67, 128.81, 125.36, 117.04, 101.57.

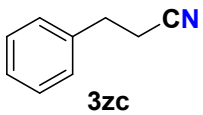


3zb

2-phenylacetonitrile (zab): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (800 MHz, $CDCl_3$) δ 7.40-7.37 (m, 2H), 7.35-7.32 (m, 3H), 3.76 (s, 2H).

^{13}C NMR (200 MHz, $CDCl_3$) δ 129.83, 129.11, 128.02, 127.88, 117.86, 23.58.

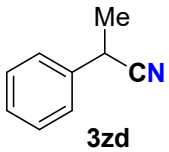


3zc

3-phenylpropanenitrile (3zc): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H NMR (400 MHz, $CDCl_3$) 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.

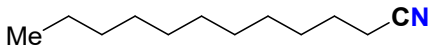


3zd

2-phenylpropanenitrile (3zd): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.

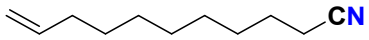


3ze

Dodecanenitrile (3ze): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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.



3zf

Undec-10-enenitrile (3zf): Prepared according to general procedure and crude was purified by silica gel column choromatography (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]

^{1}H 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).

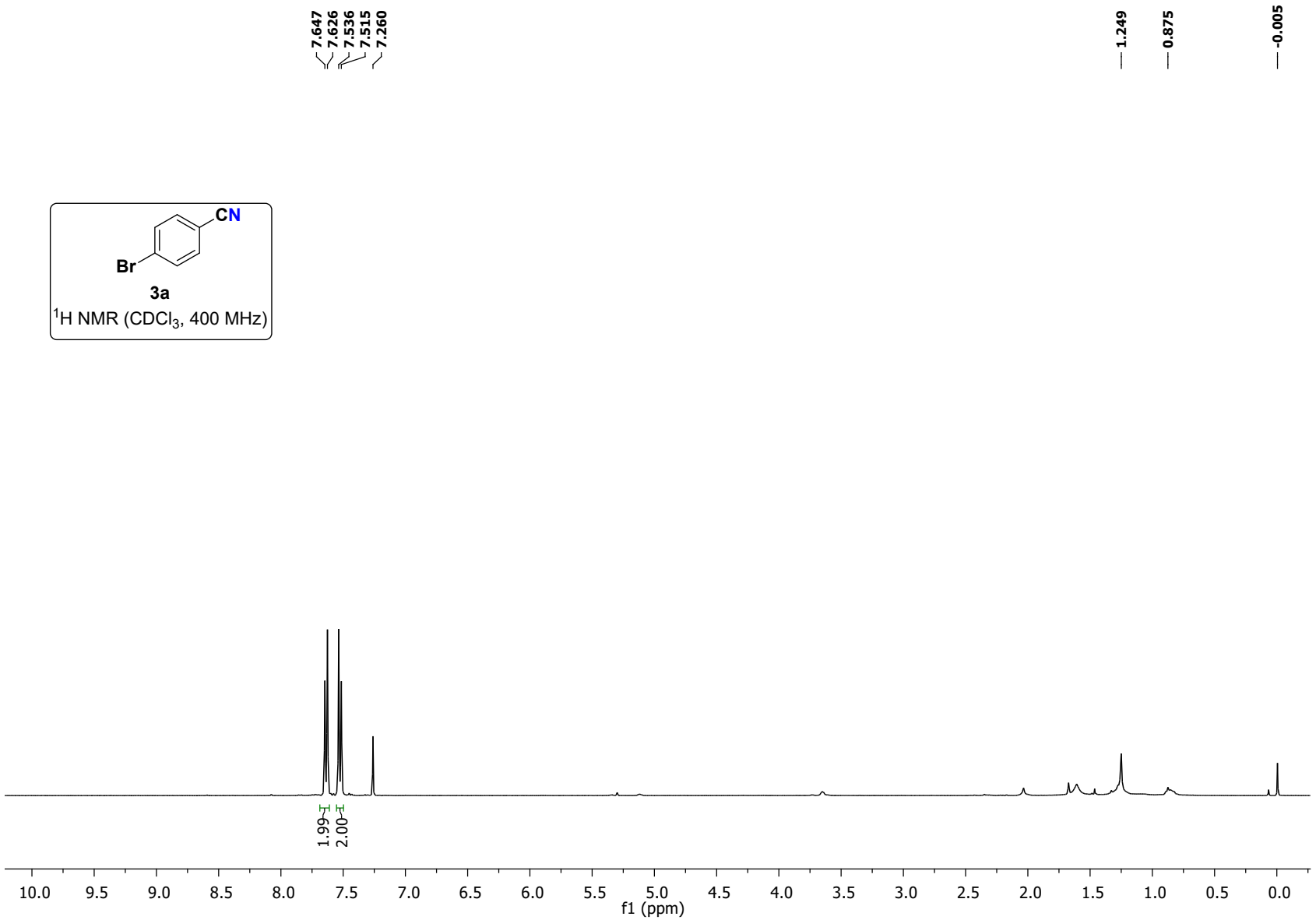
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.17, 119.97, 114.36, 33.85, 29.25, 29.06, 28.94, 28.83, 28.76, 25.47, 17.24.

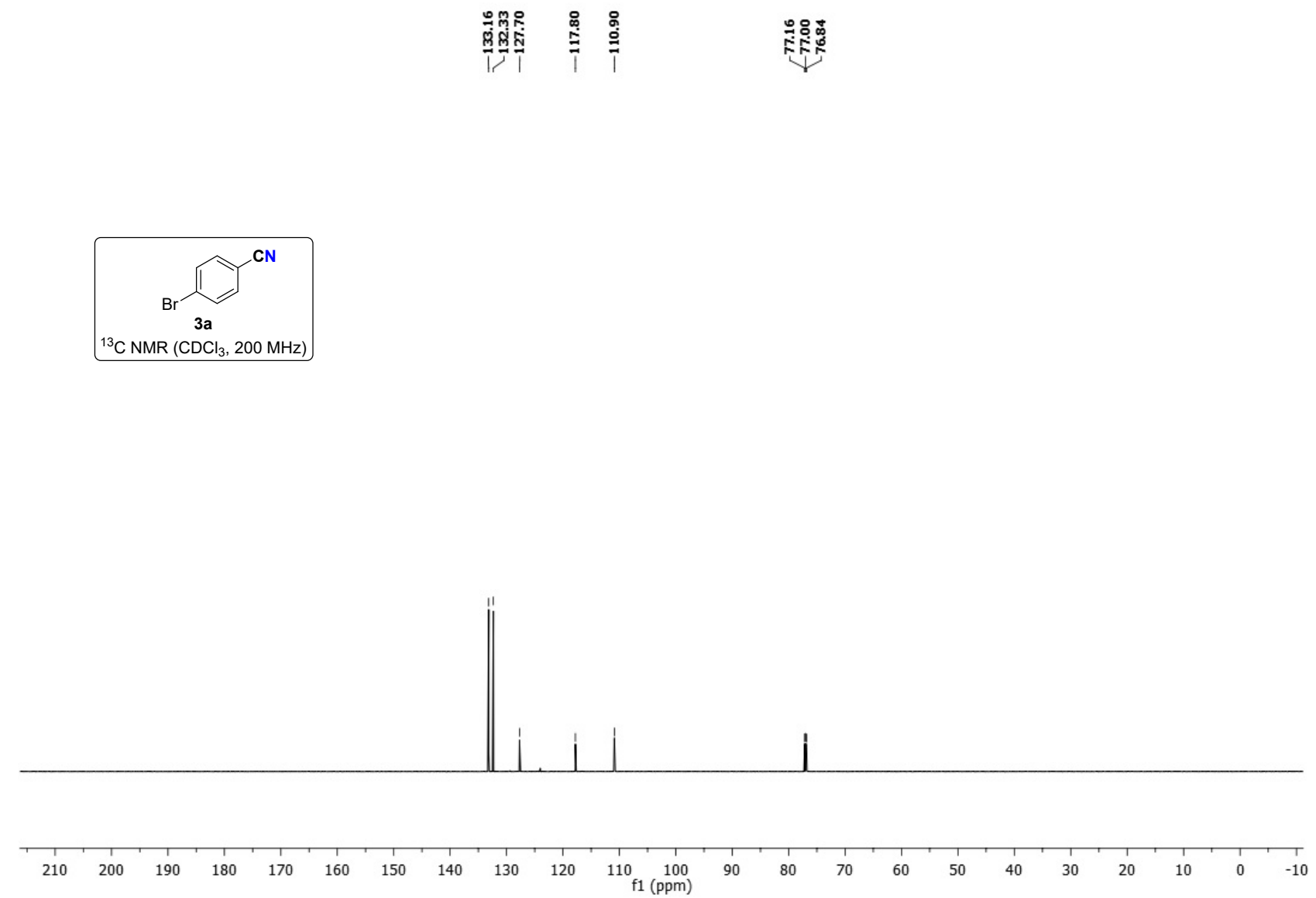
4. References:

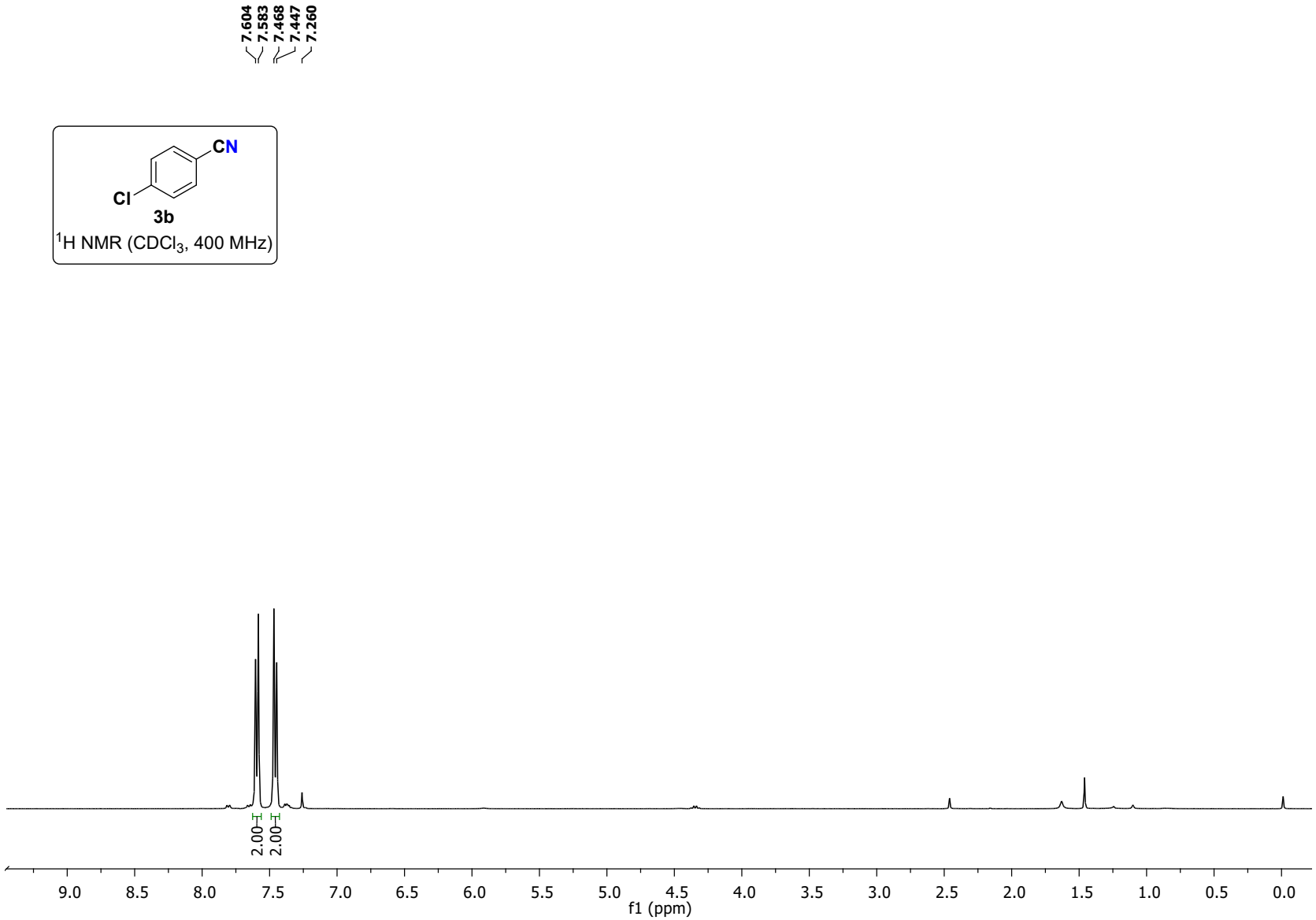
1. W.-Y. Fang, H.-L. Qin, *J. Org. Chem.* **2019**, *84*, 5803–5812.
2. K. Hyodo, K. Togashi, N. Oishi, G. Hasegawa, K. Uchida, *Org. Lett*. **2017**, *19*, 3005−3008.
3. Q. Wu, Y. Luo, A. Lei, J.You, *J. Am. Chem. Soc.* **2016**, *138*, 2885−2888.
4. C. Fang, M. Li, X. Hu, W. Mo, B. Hu, N. Sun, L. Jin, Z. Shen, *RSC Adv.* **2017**, *7*, 1484−1489.
5. S. R. Mudshinge, C. S. Potnis, B. Xu, G. B. Hammonda, *Green Chem.* **2020**, *22*, 4161–4164.
6. Y. Luo, Q. Wen, Z. Wu, J. Jin, P. Lu, Y. Wang, *Tetrahedron* **2013**, *69*, 8400−8404.

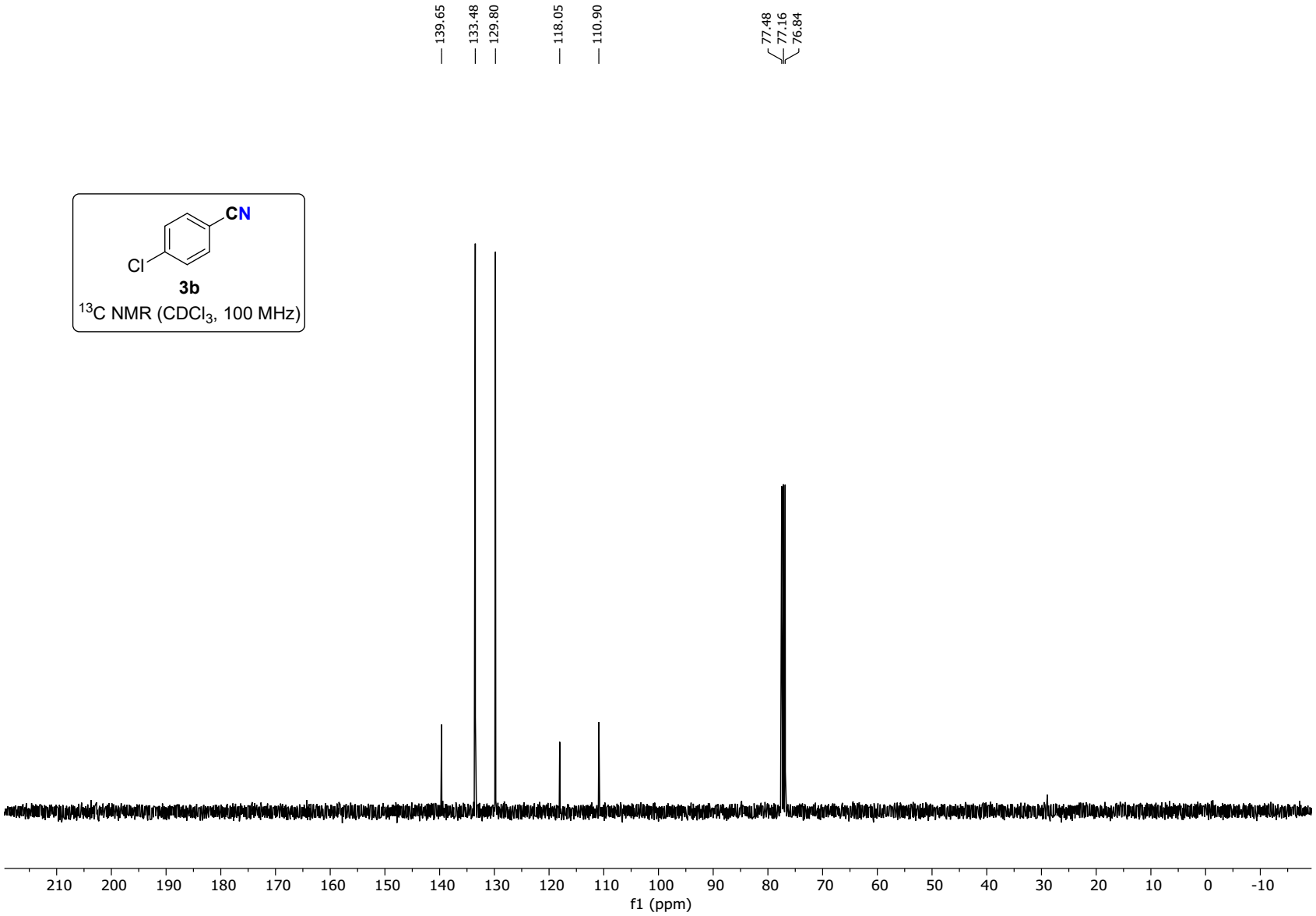
7. B. Chatterjee, S. Jena, V. Chugh, T. Weyhermüller, C. Werlé, *ACS Catal.* **2021**, *11*, 7176−7185.
8. X.-D. An, Y. Shouyun *Org. Lett.* **2015**, *17*, 5064–5067.
9. Y.-P. Han, X.-R. Song, Y.-F. Qiu, X.-H. Hao, J. Wang, X.-X. Wu, X.-Y. Liu, Y.-M. Liang, *J. Org. Chem.* **2015**, *80*, 9200–9207.

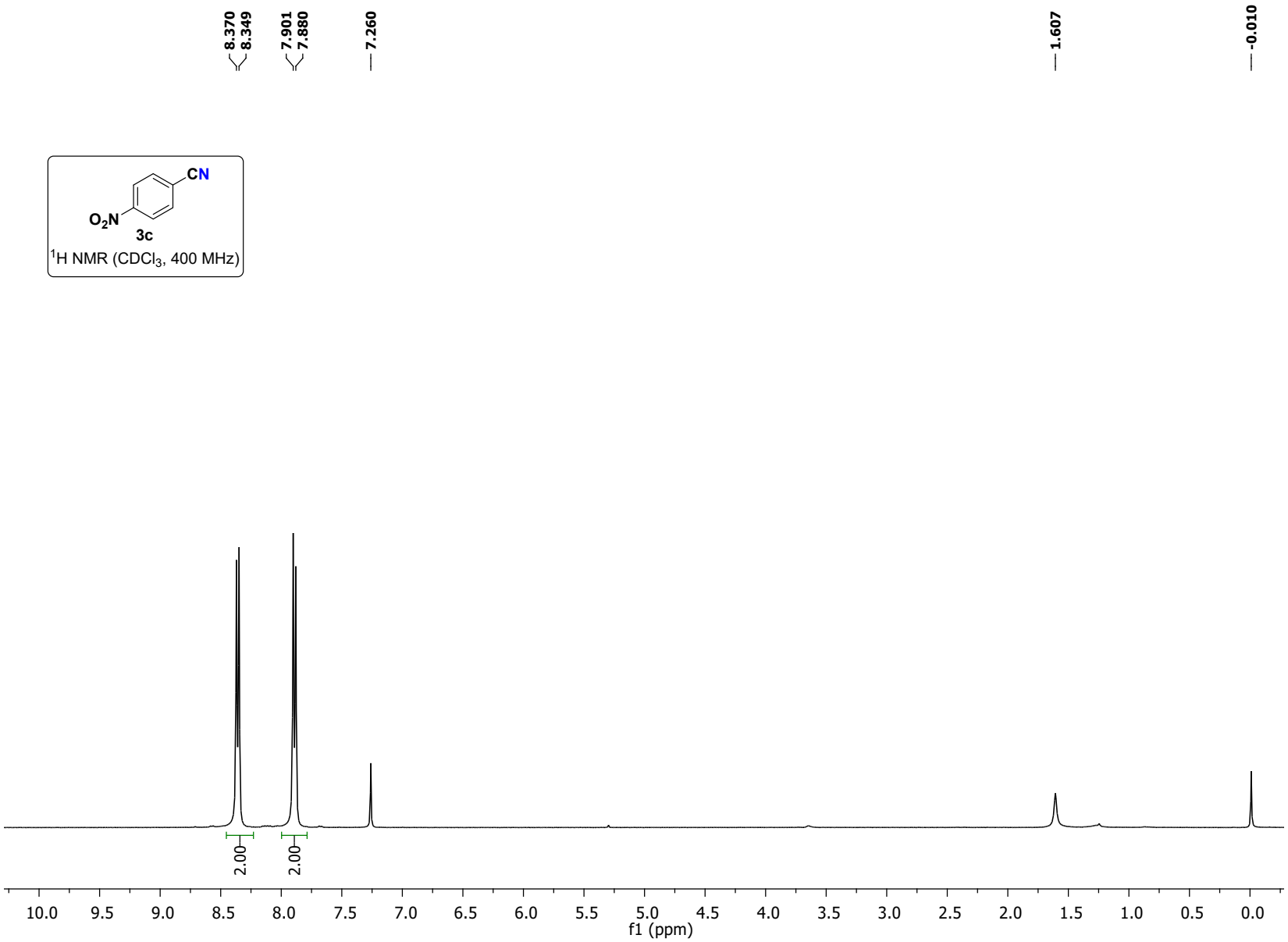
5. NMR Spectra of the products

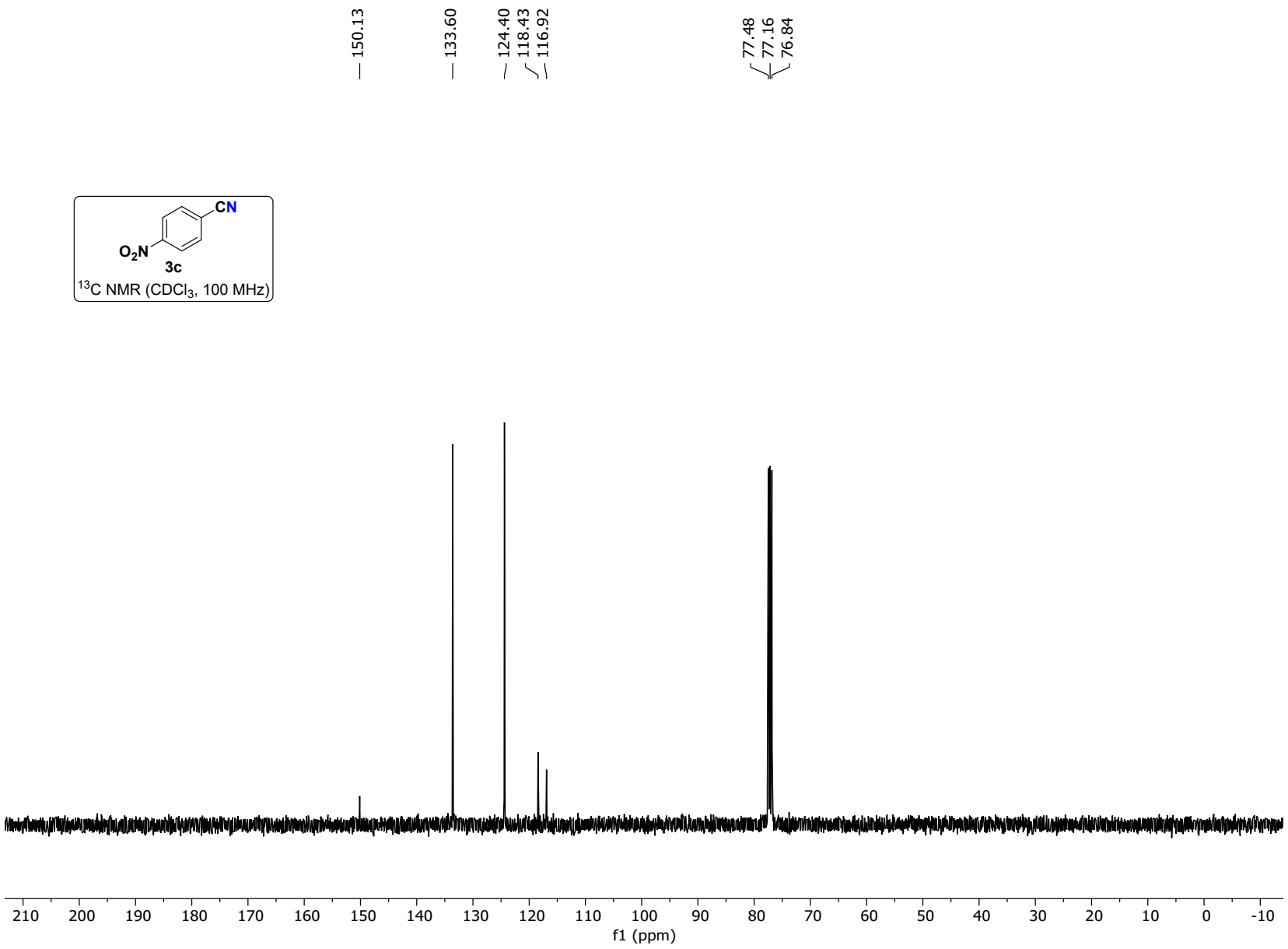


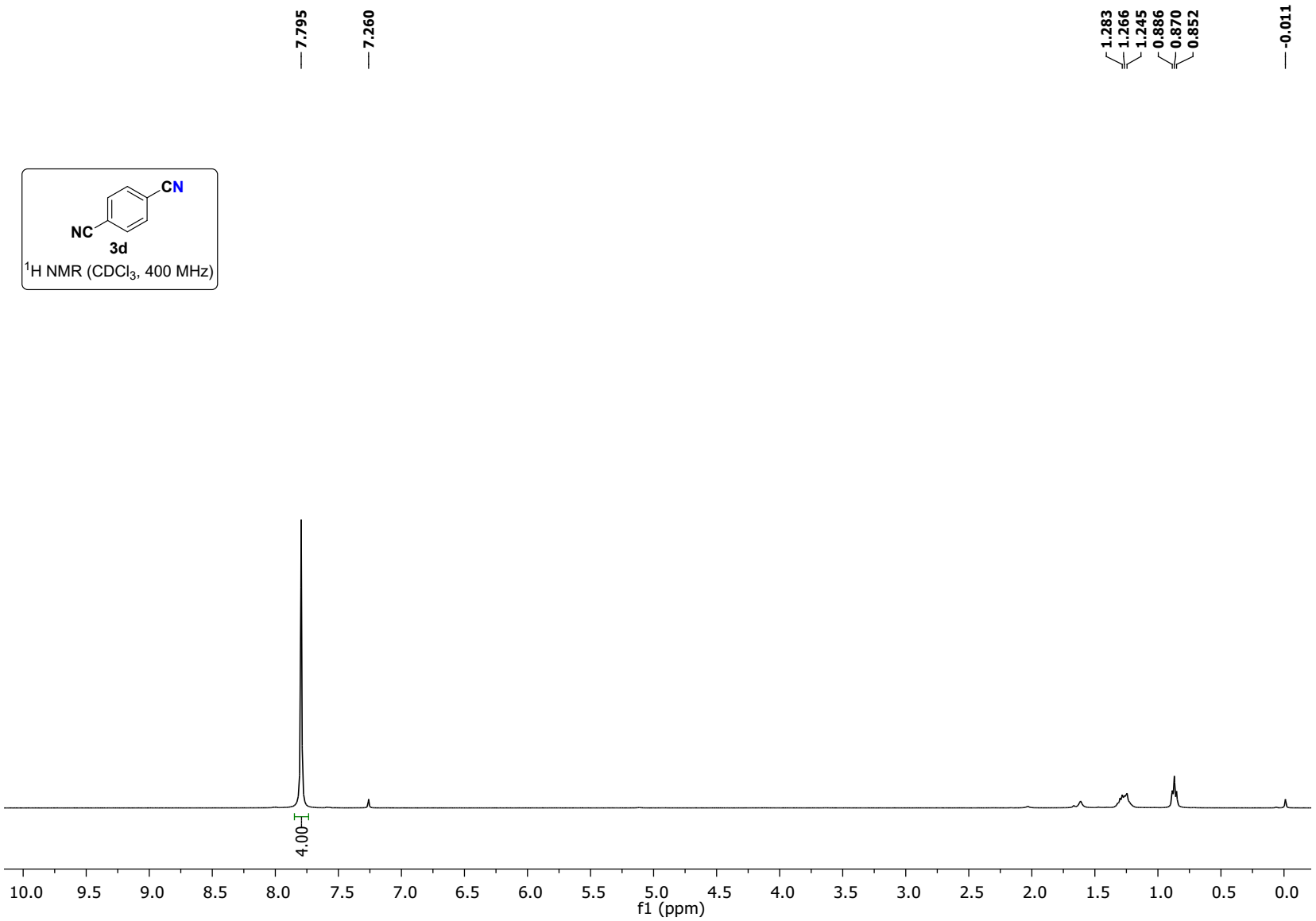

133.16
132.33
127.70
117.80
110.90
77.16
77.00
76.84
CN
Br
3a
13C NMR (CDCl3, 200 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

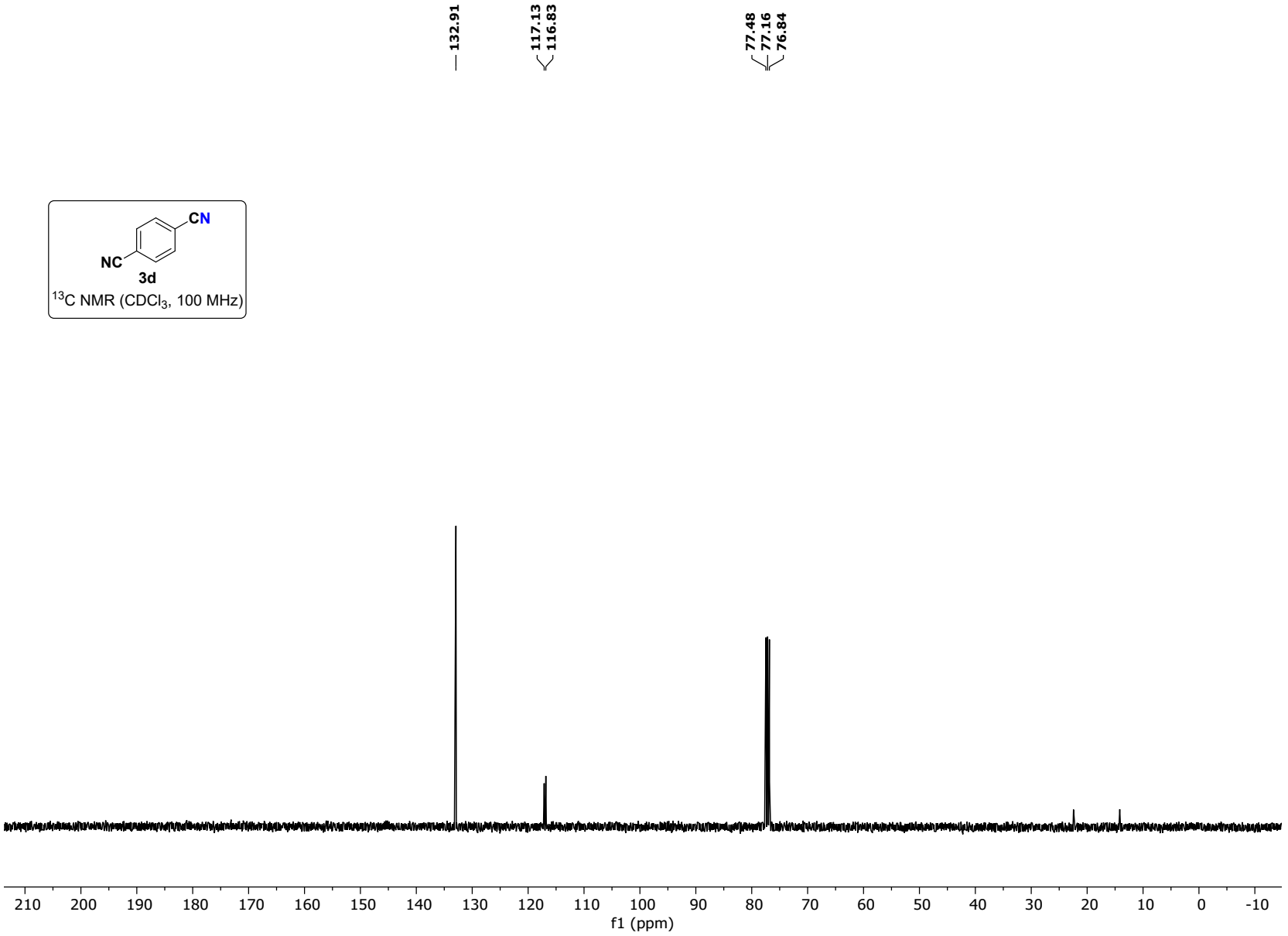

7.604
7.583
7.468
7.447
7.260
CN
Cl
3b
^{1}H NMR ($CDCl_3$, 400 MHz)
2.00
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

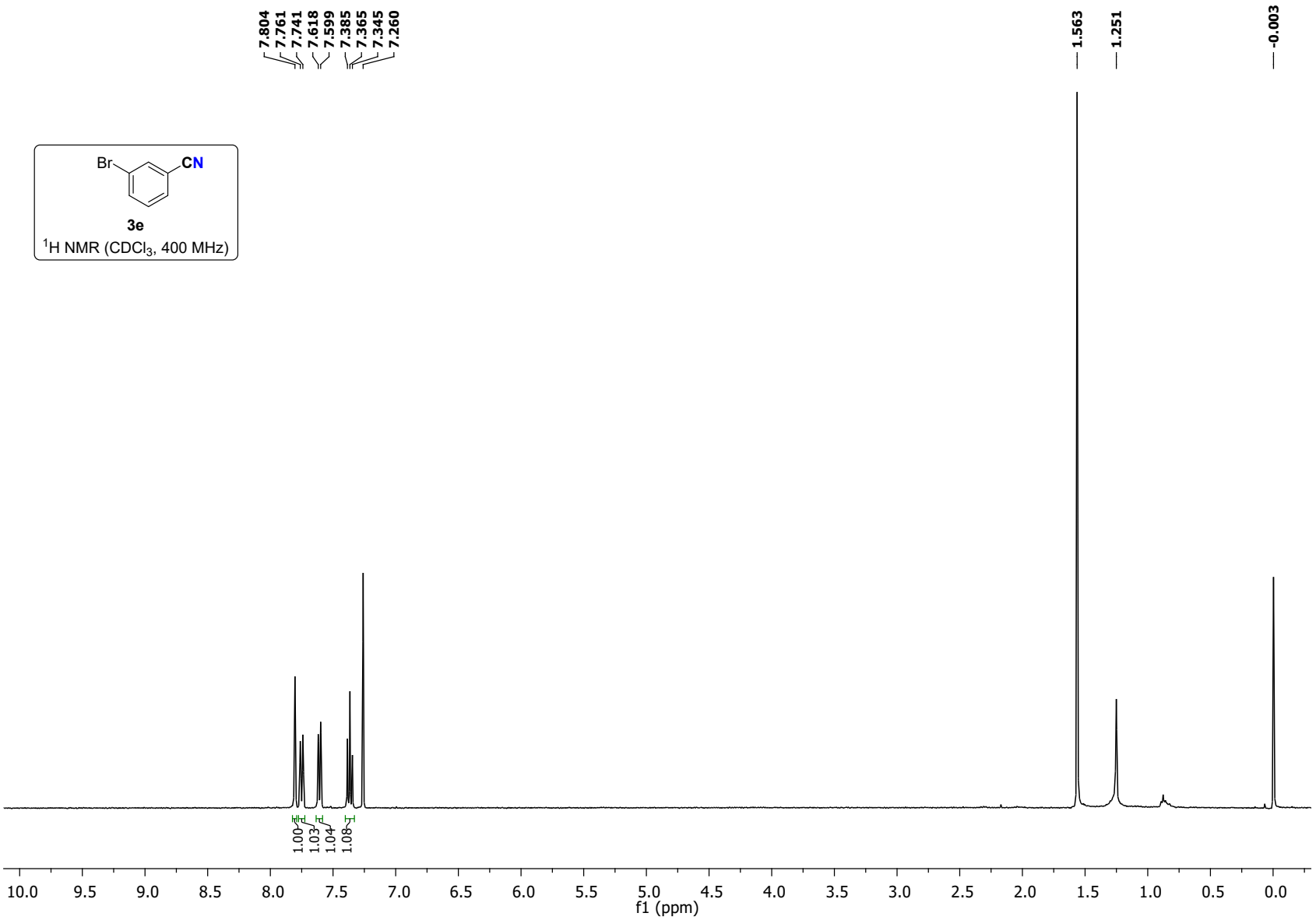

139.65
133.48
129.80
118.05
110.90
77.48
77.16
76.84
CN
Cl
3b
13C NMR (CDCl3, 100 MHz)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

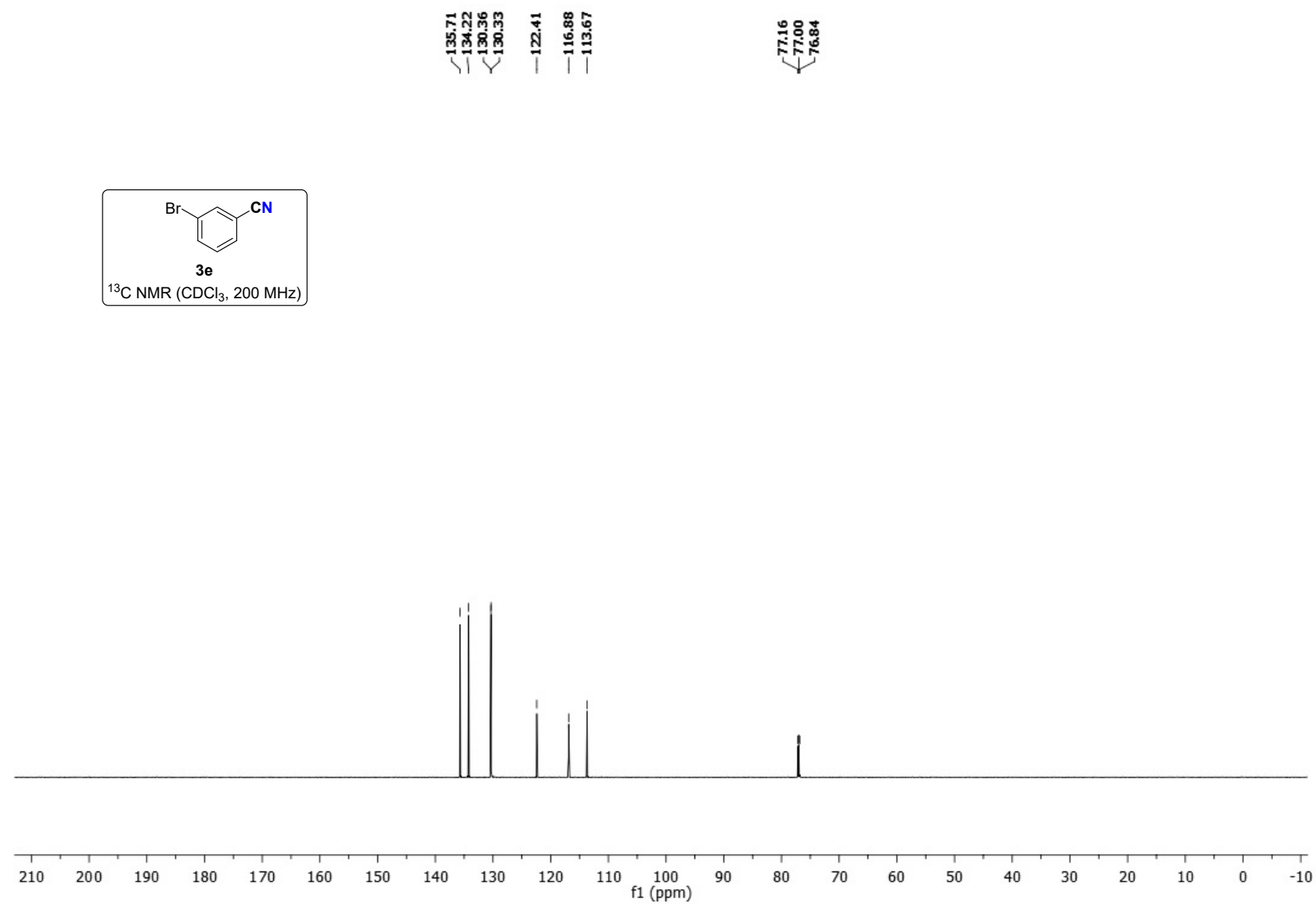

8.370
8.349
7.901
7.880
7.260
1.607
-0.010
CN
O2N
3c
1H NMR (CDCl3, 400 MHz)
2.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

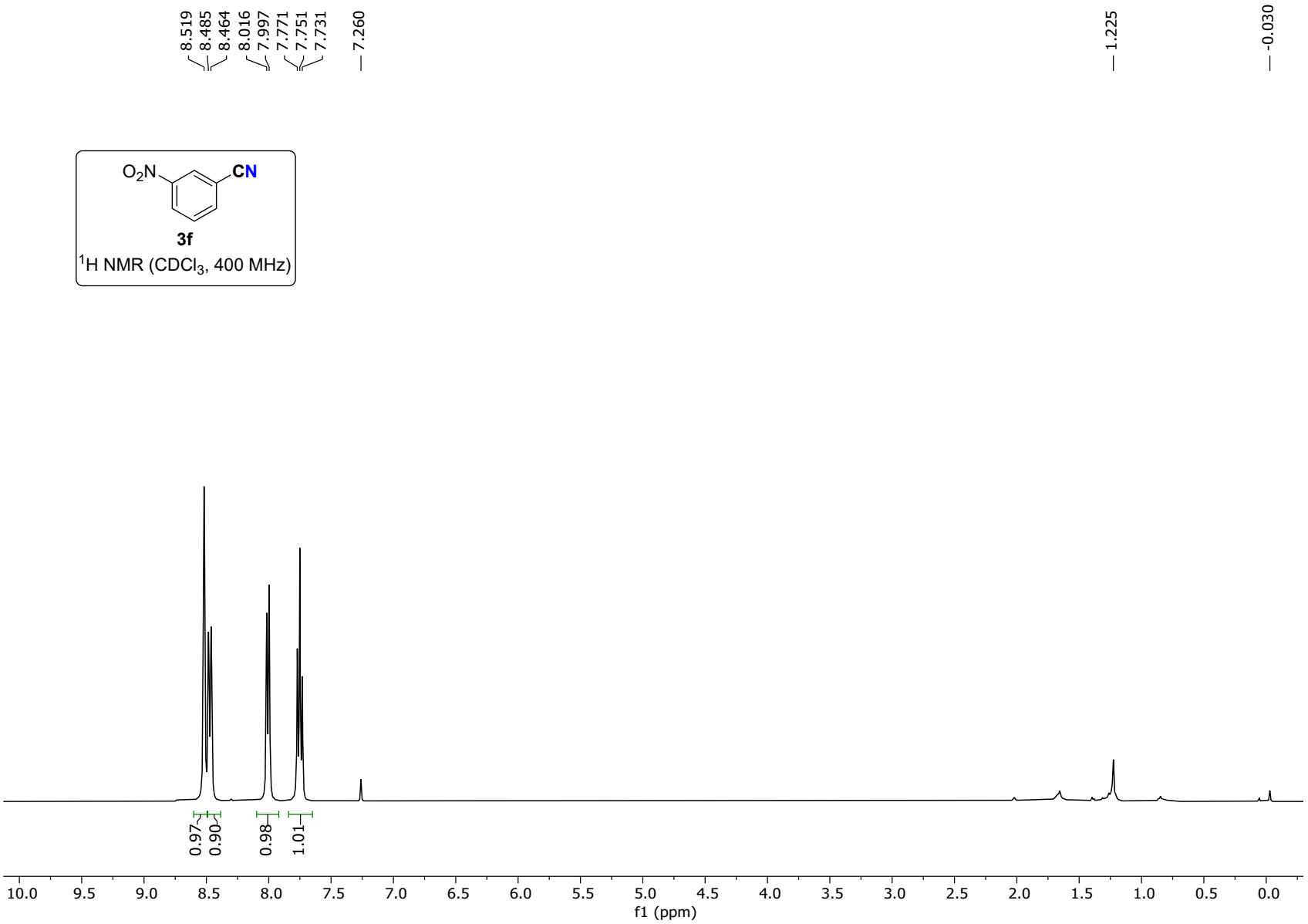

150.13
133.60
124.40
118.43
116.92
77.48
77.16
76.84
CN
O2N
3c
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

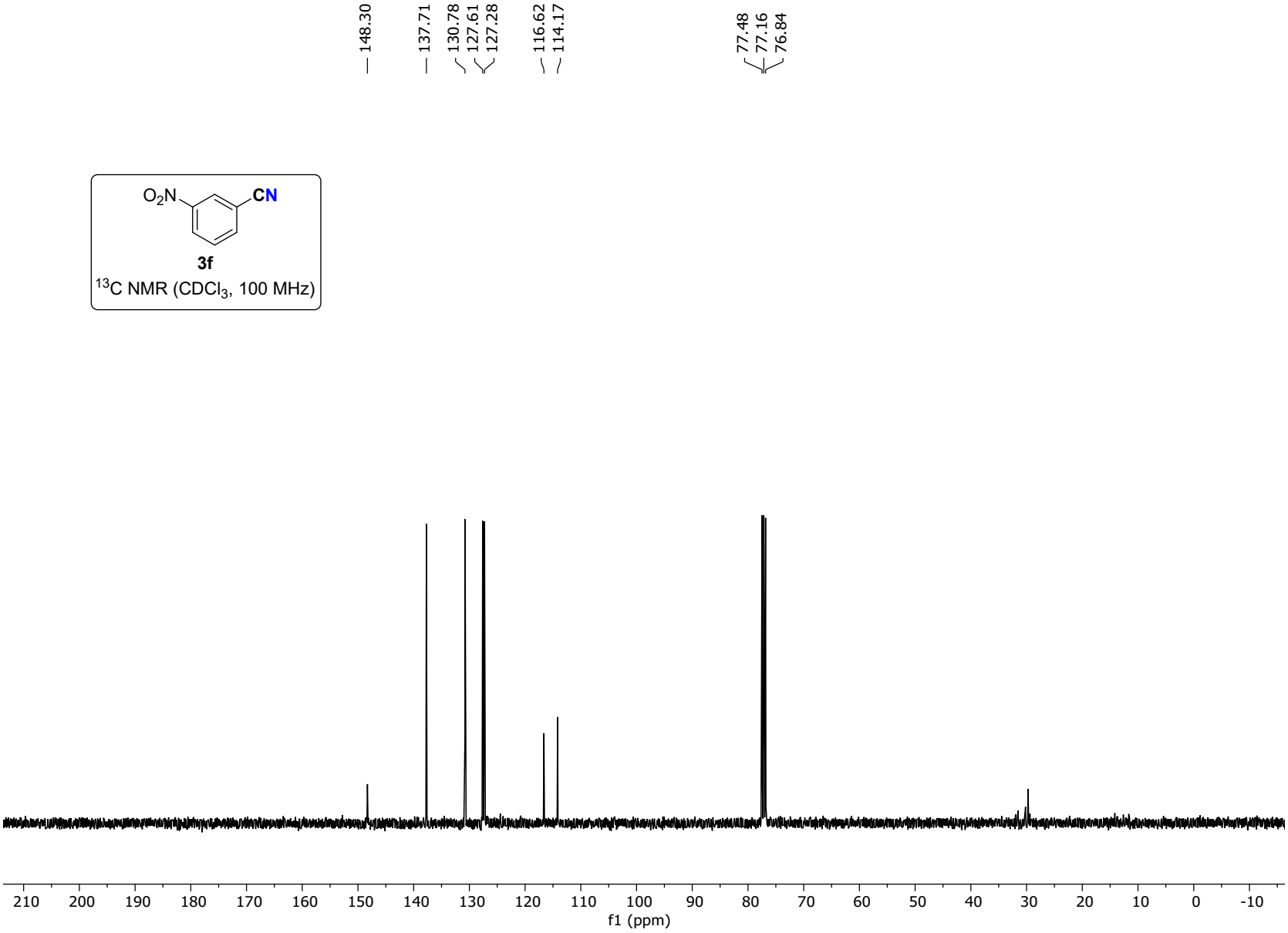

7.795
7.260
1.283
1.266
1.245
0.886
0.870
0.852
-0.011
CN
NC
3d
^{1}H NMR ($CDCl_3$, 400 MHz)
4.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

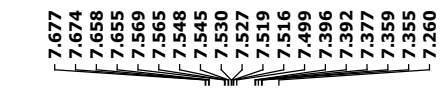

132.91
117.13
116.83
77.48
77.16
76.84
CN
NC
3d
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.804
7.761
7.741
7.618
7.599
7.385
7.365
7.345
7.260
1.563
1.251
-0.003
Br
CN
3e
1H NMR (CDCl3, 400 MHz)
1.00
1.03
1.04
1.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

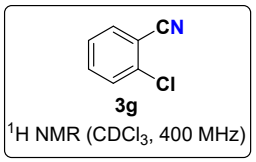

135.71
134.22
130.36
130.33
122.41
116.88
113.67
77.16
77.00
76.84
Br
CN
3e
13C NMR (CDCl3, 200 MHz)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

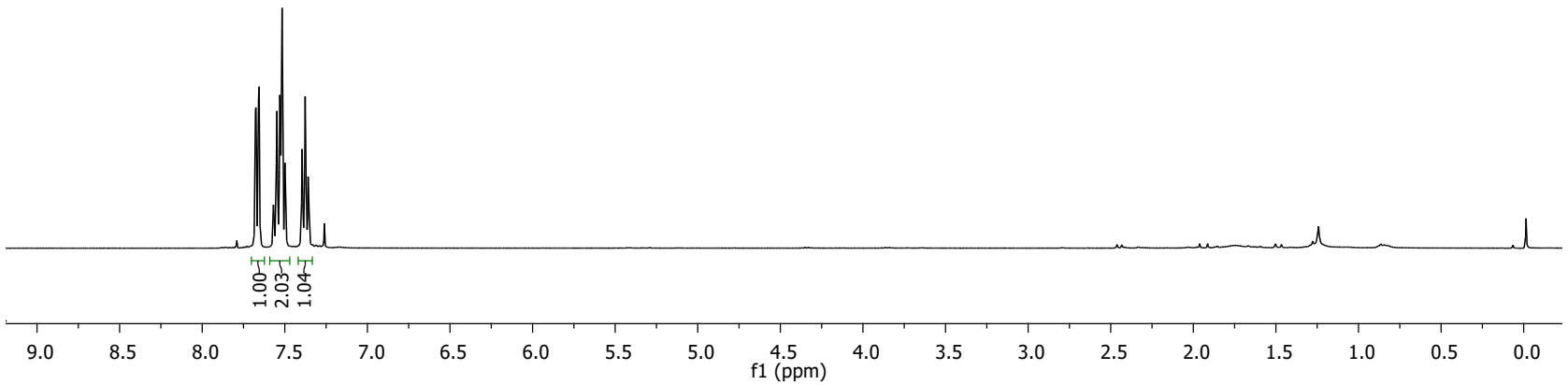

8.519
8.485
8.464
8.016
7.997
7.771
7.751
7.731
7.260
1.225
-0.030
O2N
CN
3f
1H NMR (CDCl3, 400 MHz)
0.97
0.90
0.98
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

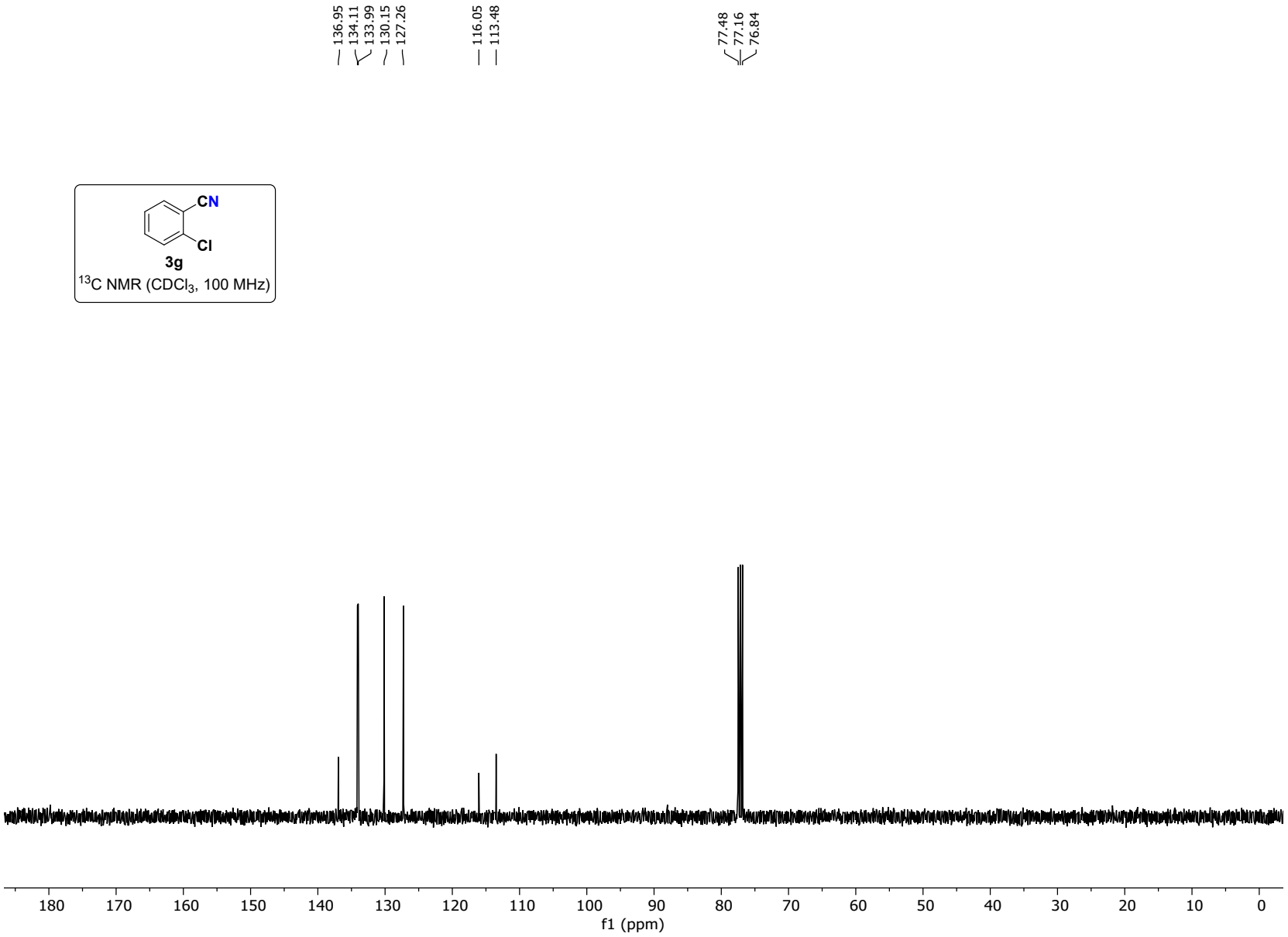

148.30
137.71
130.78
127.61
127.28
116.62
114.17
77.48
77.16
76.84
O2N
CN
3f
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

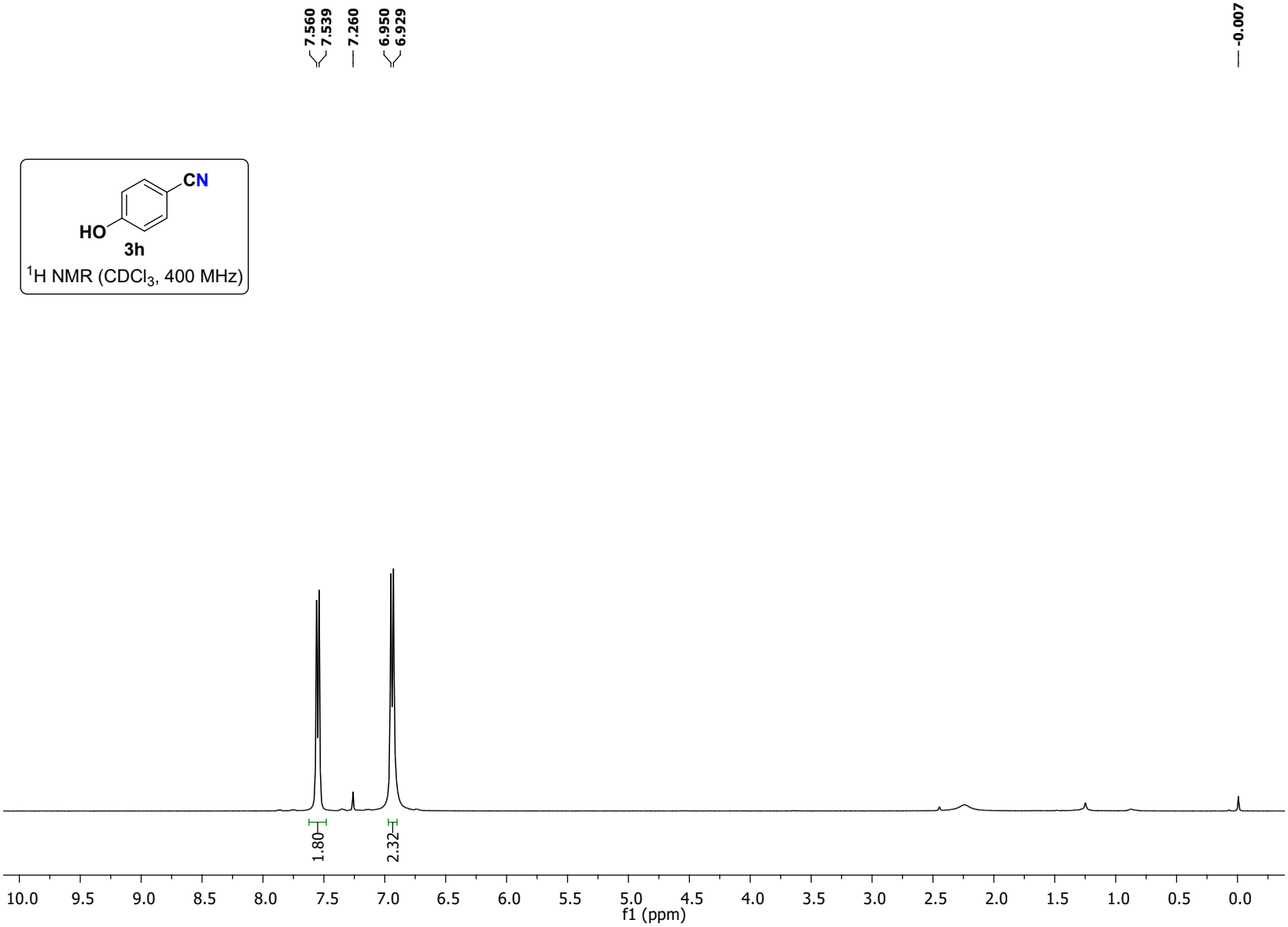

7.677
7.674
7.658
7.655
7.569
7.565
7.548
7.545
7.530
7.527
7.519
7.516
7.499
7.396
7.392
7.377
7.359
7.355
7.260

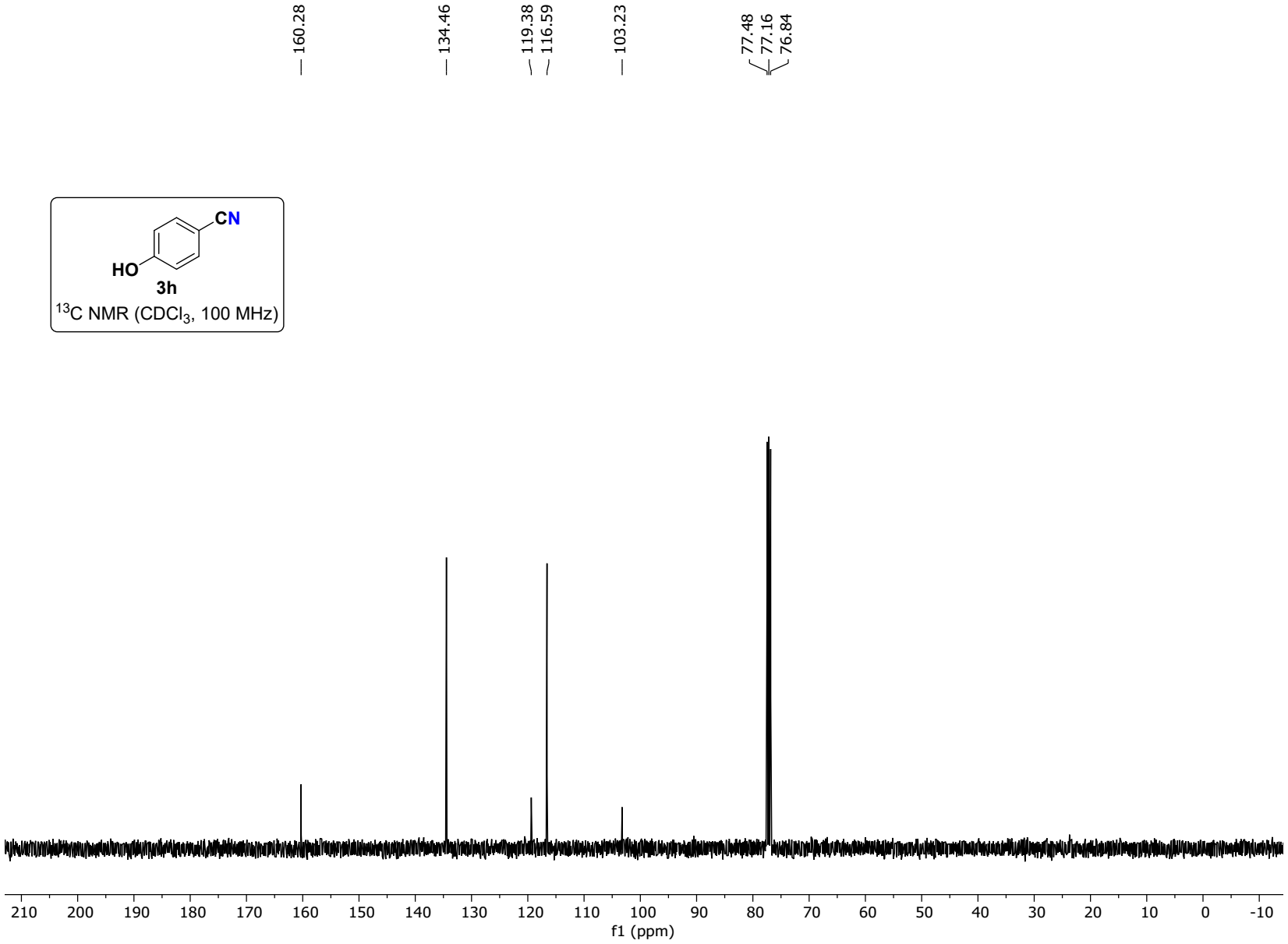

-0.014

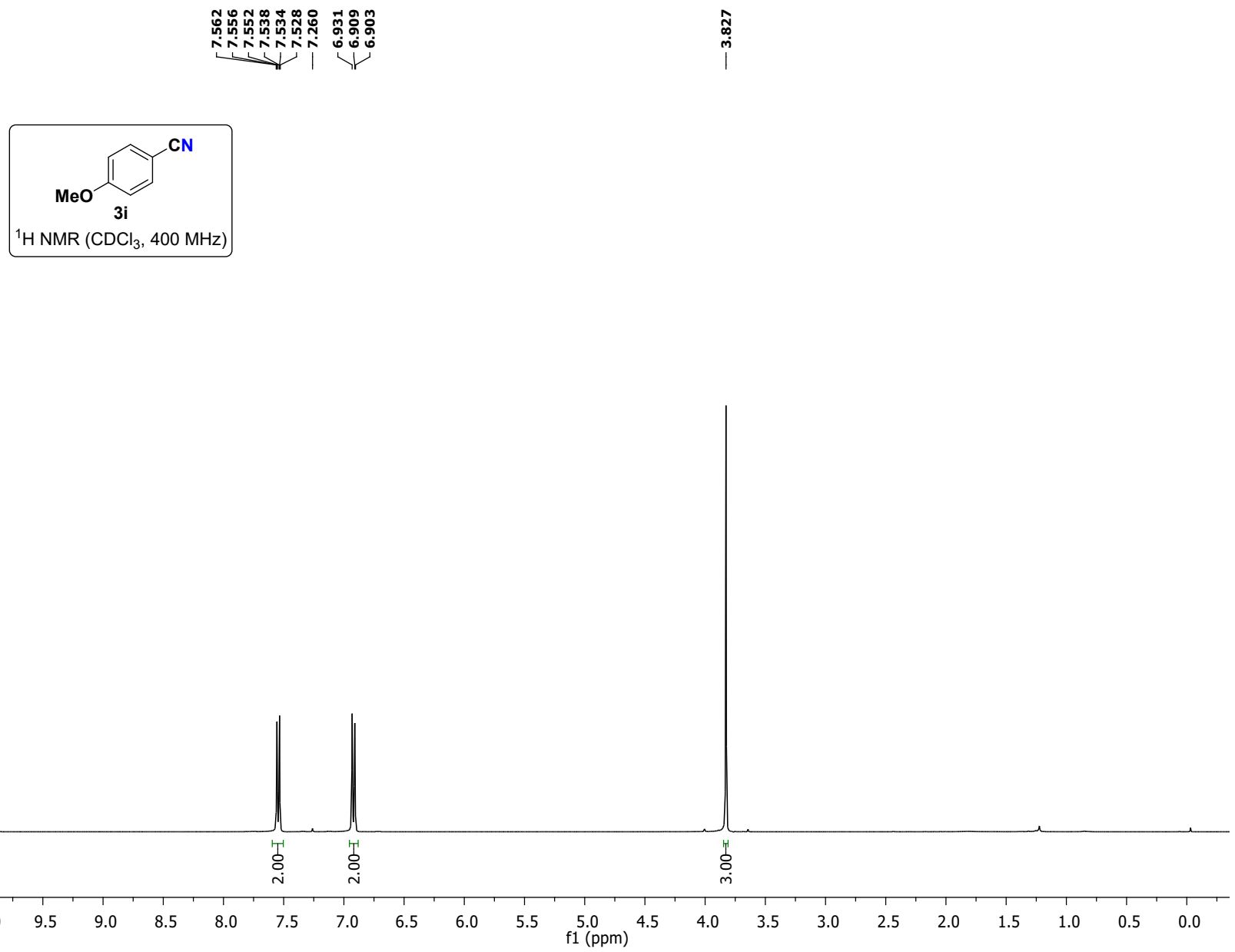

CN
Cl
3g
^{1}H NMR ($CDCl_3$, 400 MHz)

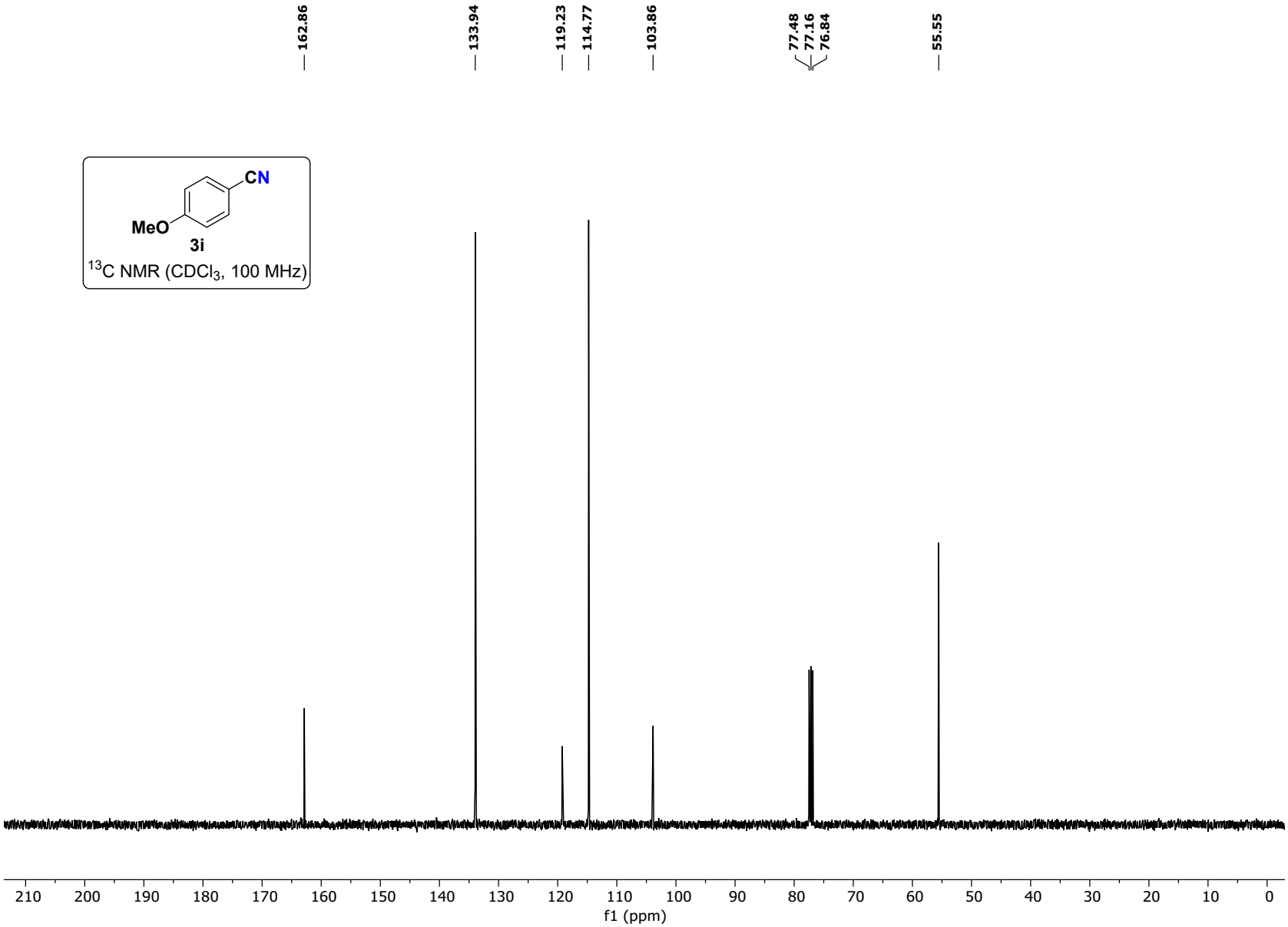

1.00
2.03
1.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


136.95
134.11
133.99
130.15
127.26
116.05
113.48
77.48
77.16
76.84
CN
Cl
3g
13C NMR (CDCl3, 100 MHz)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

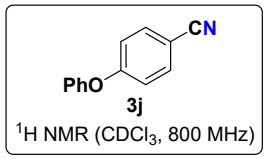

7.560
7.539
7.260
6.950
6.929
-0.007
CN
HO
3h
^{1}H NMR ($CDCl_3$, 400 MHz)
1.80
2.32
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

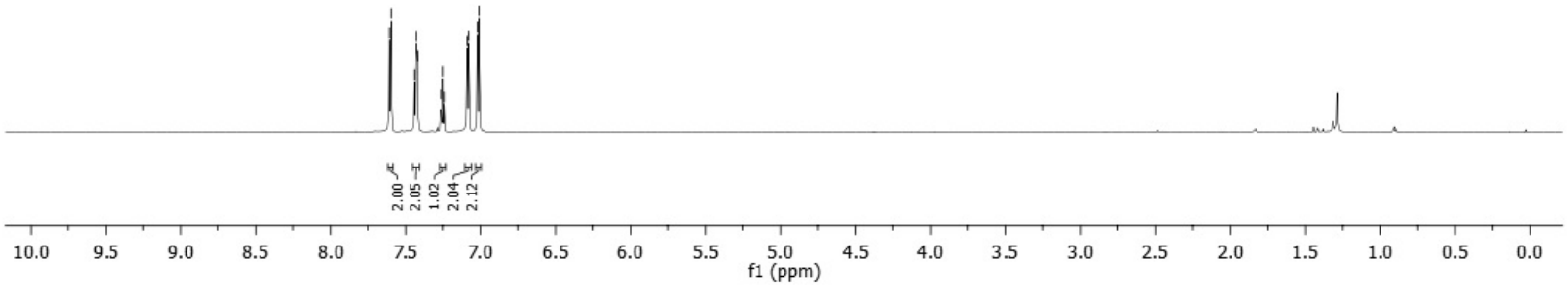

160.28
134.46
119.38
116.59
103.23
77.48
77.16
76.84
CN
HO
3h
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

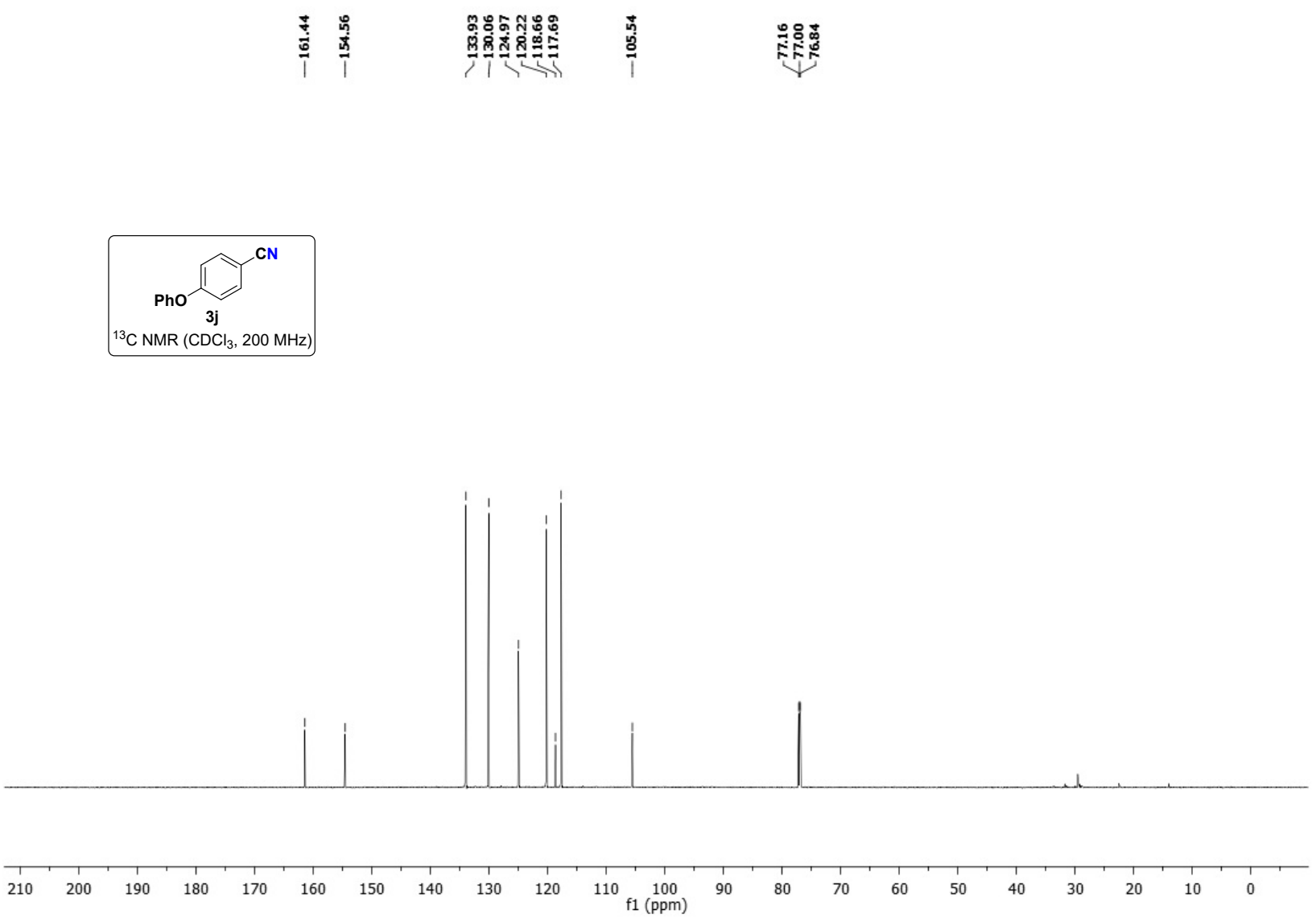

7.562
7.556
7.552
7.538
7.534
7.528
7.260
6.931
6.909
6.903
3.827
CN
MeO
3i
^{1}H NMR ($CDCl_3$, 400 MHz)
2.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

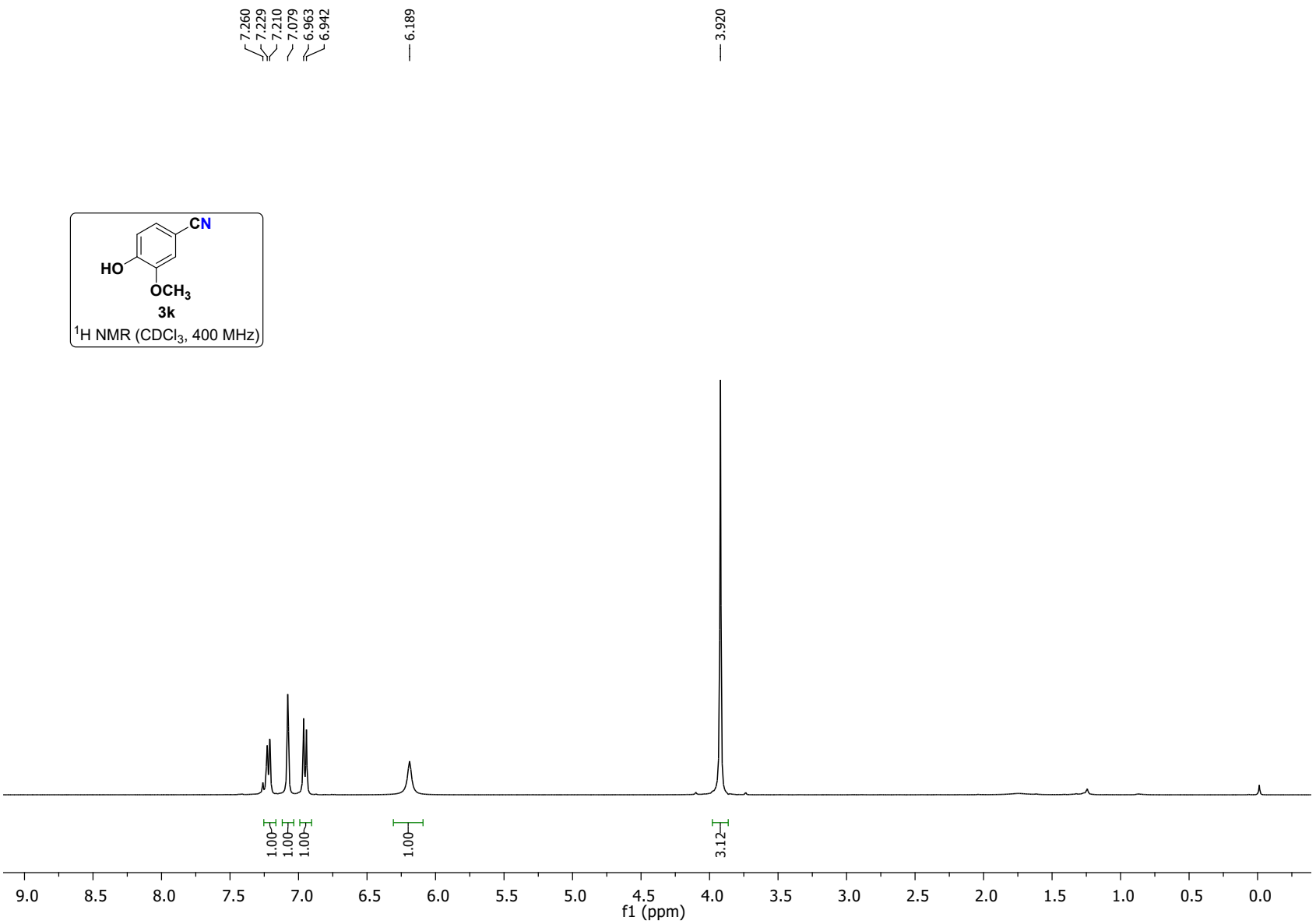

162.86
133.94
119.23
114.77
103.86
77.48
77.16
76.84
55.55
CN
MeO
3i
13C NMR (CDCl3, 100 MHz)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

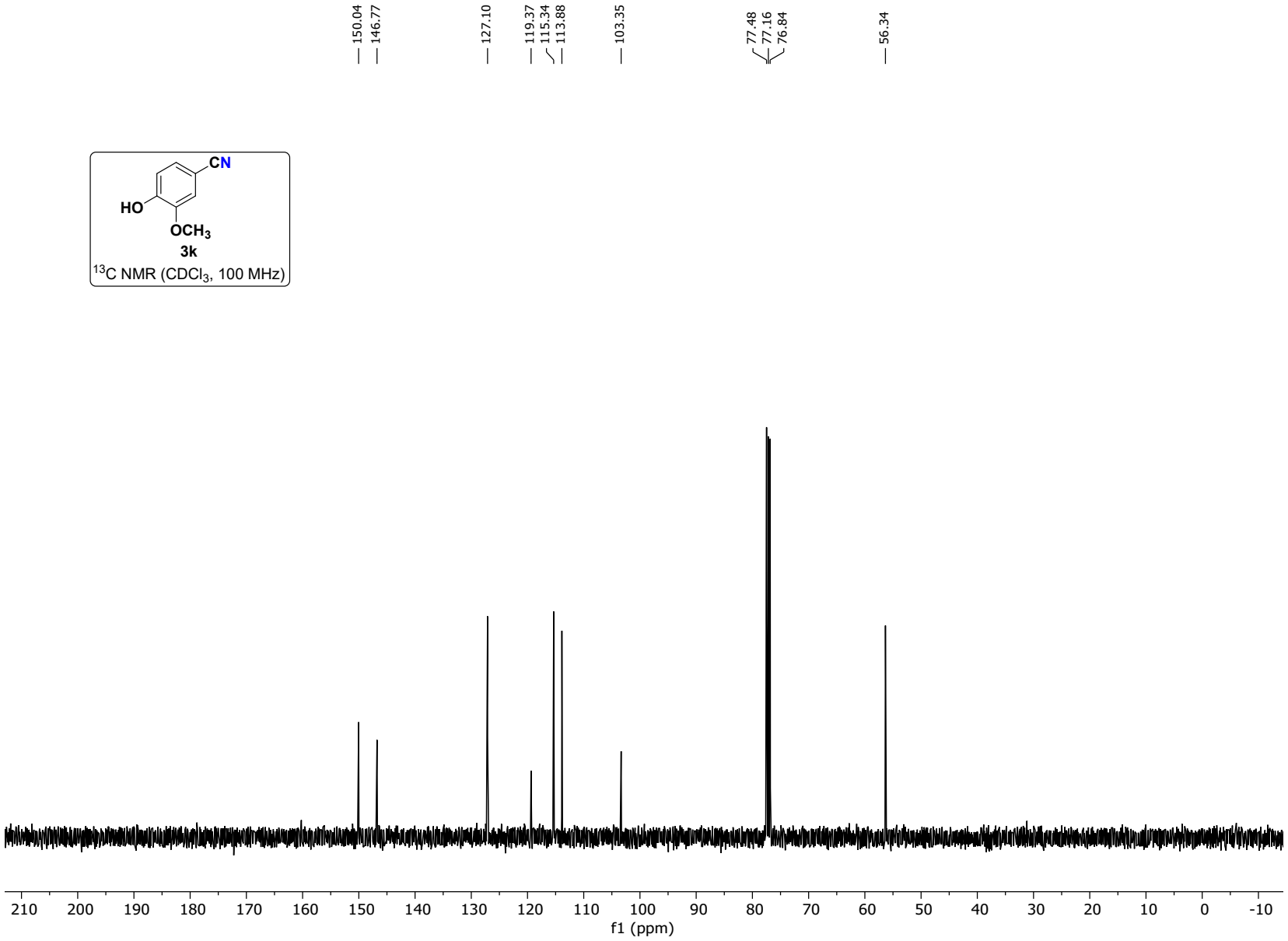

7.61
7.60
7.44
7.43
7.42
7.26
7.25
7.24
7.09
7.08
7.02
7.01

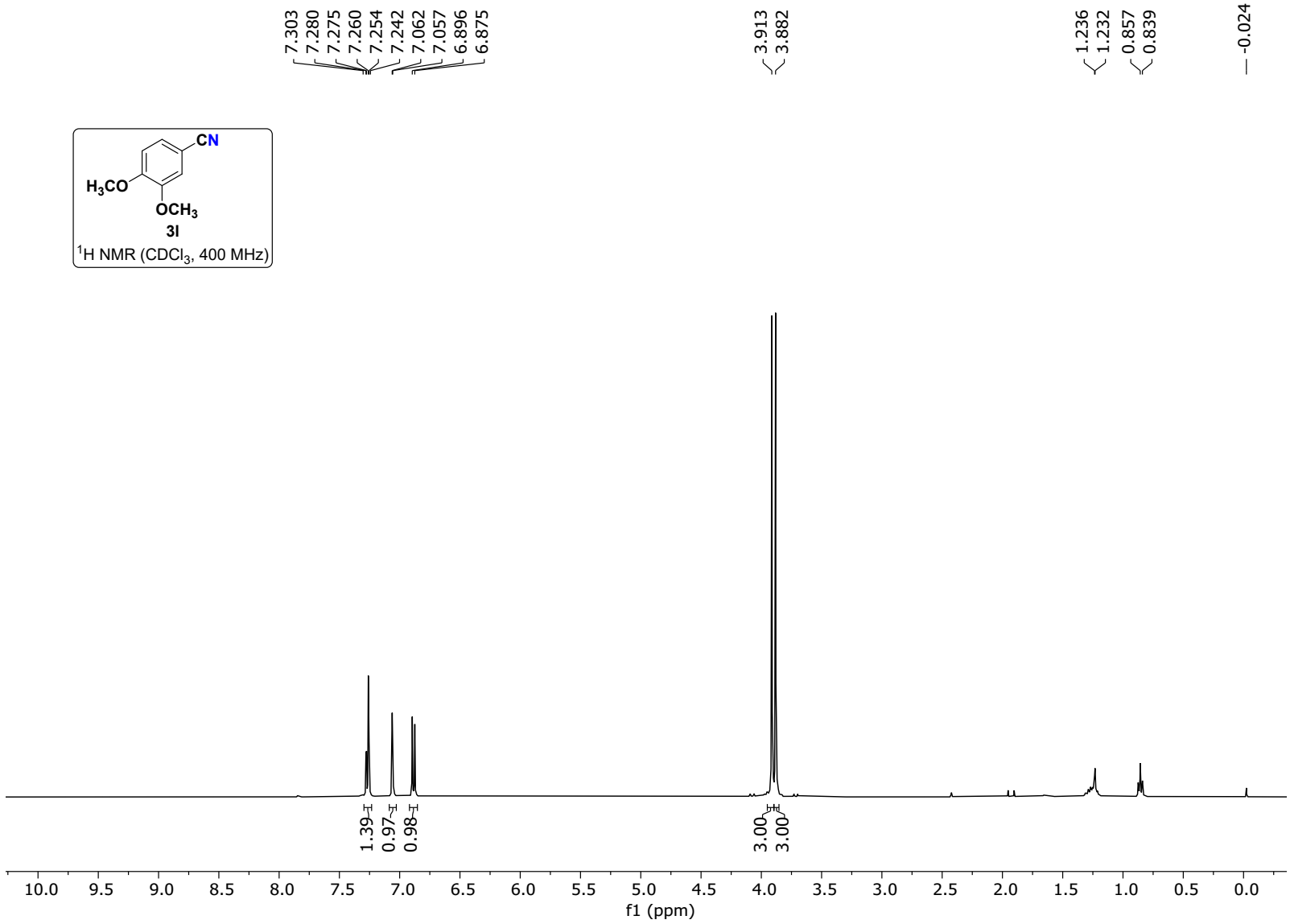

CN
PhO
3j
^{1}H NMR ($CDCl_3$, 800 MHz)

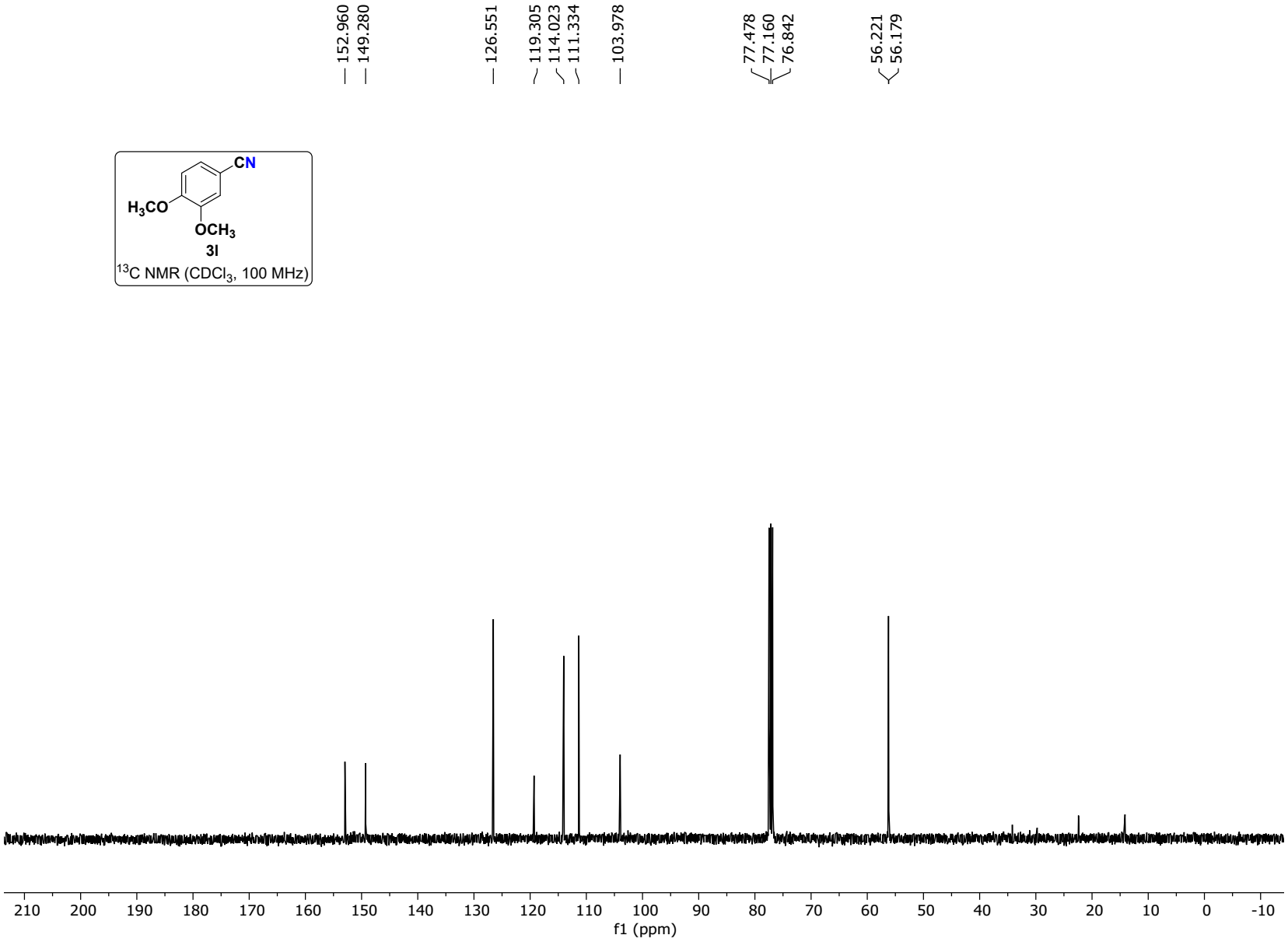

2.00
2.05
1.02
2.04
2.12
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

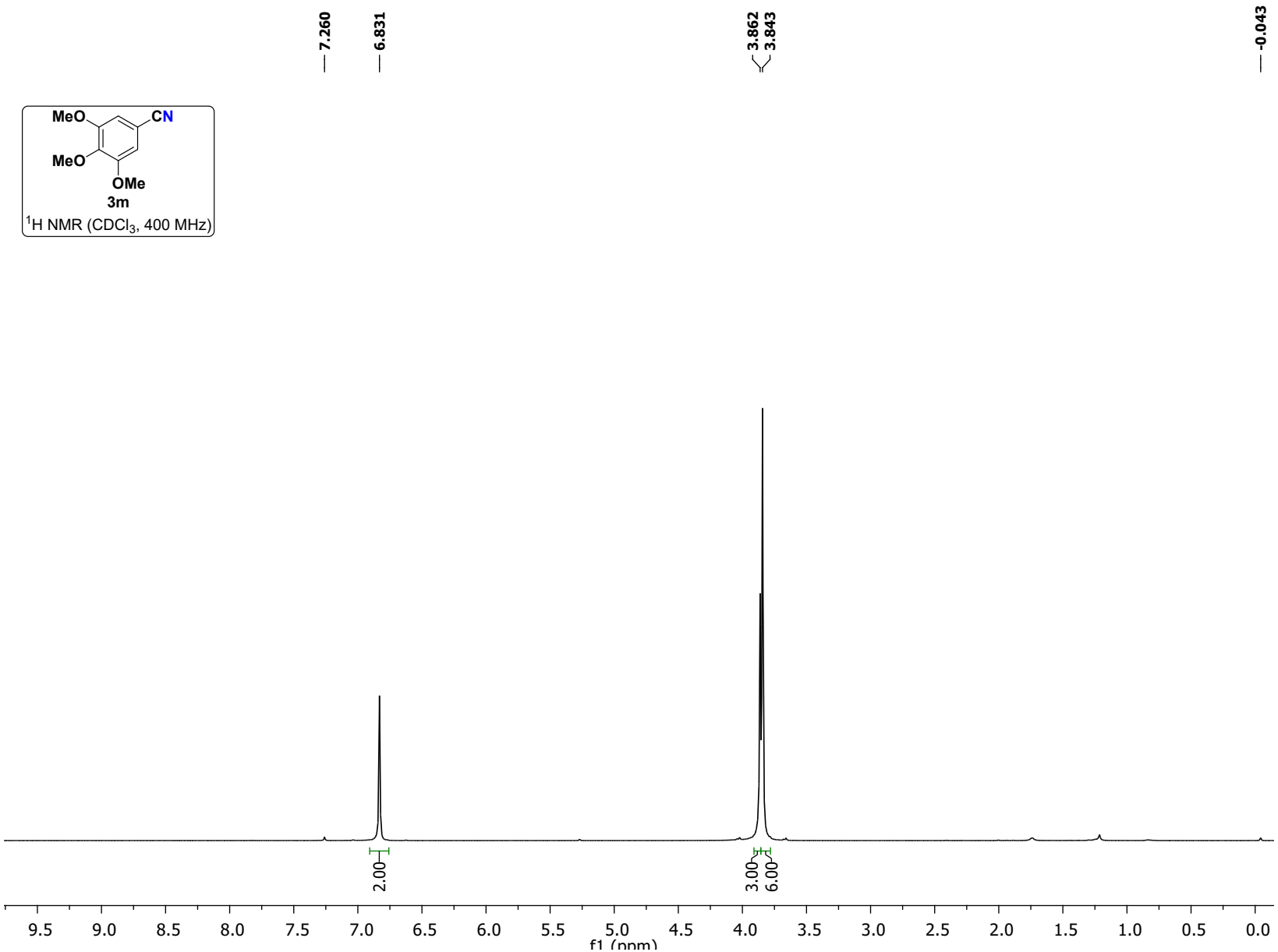

161.44
154.56
133.93
130.06
124.97
120.22
118.66
117.69
105.54
77.16
77.00
76.84
CN
PhO
3j
13C NMR (CDCl3, 200 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

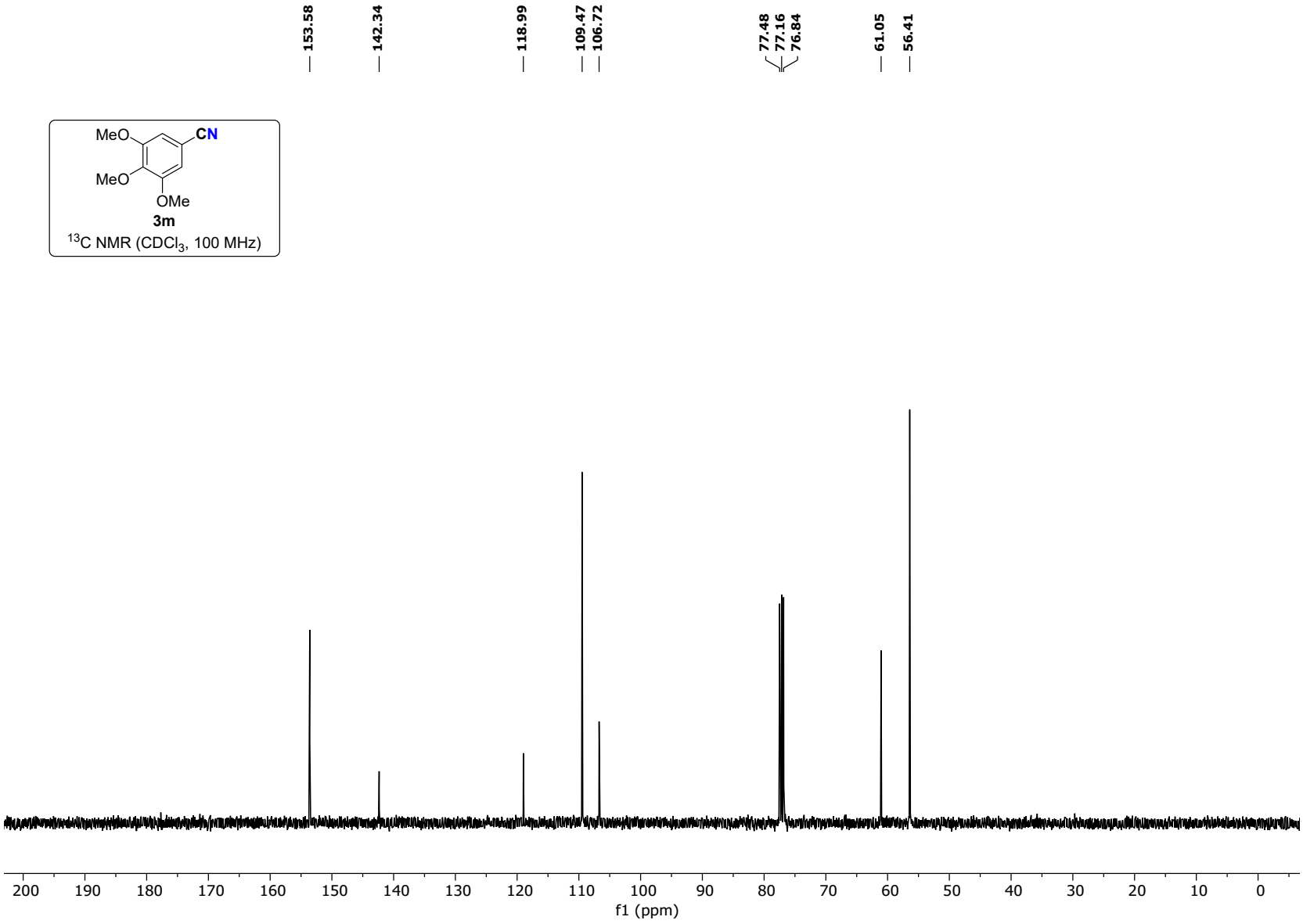

7.260
7.229
7.210
7.079
6.963
6.942
6.189
3.920
CN
HO
OCH3
3k
1H NMR (CDCl3, 400 MHz)
1.00
1.00
1.00
1.00
3.12
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

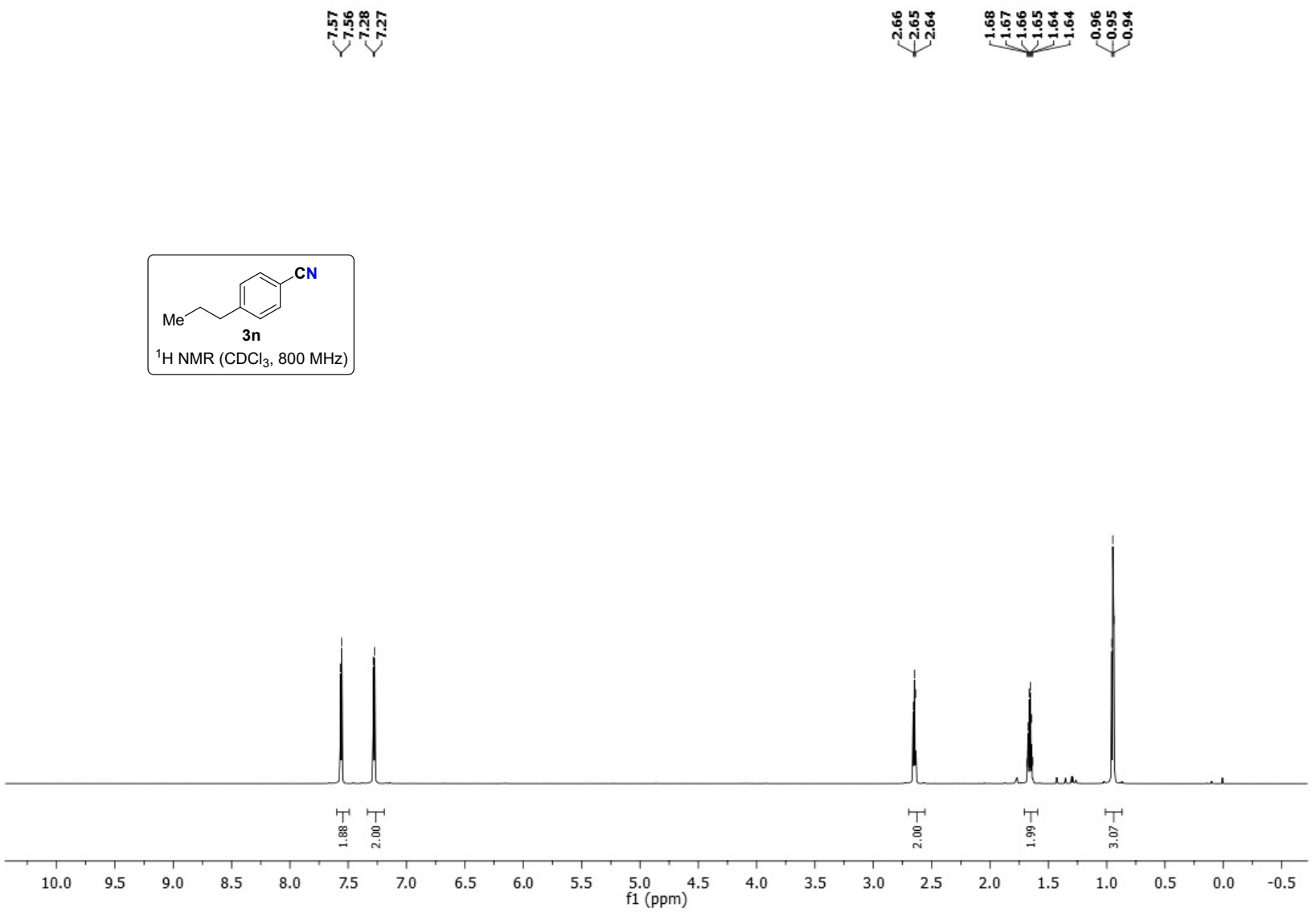

150.04
146.77
127.10
119.37
115.34
113.88
103.35
77.48
77.16
76.84
56.34
CN
HO
OCH3
3k
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

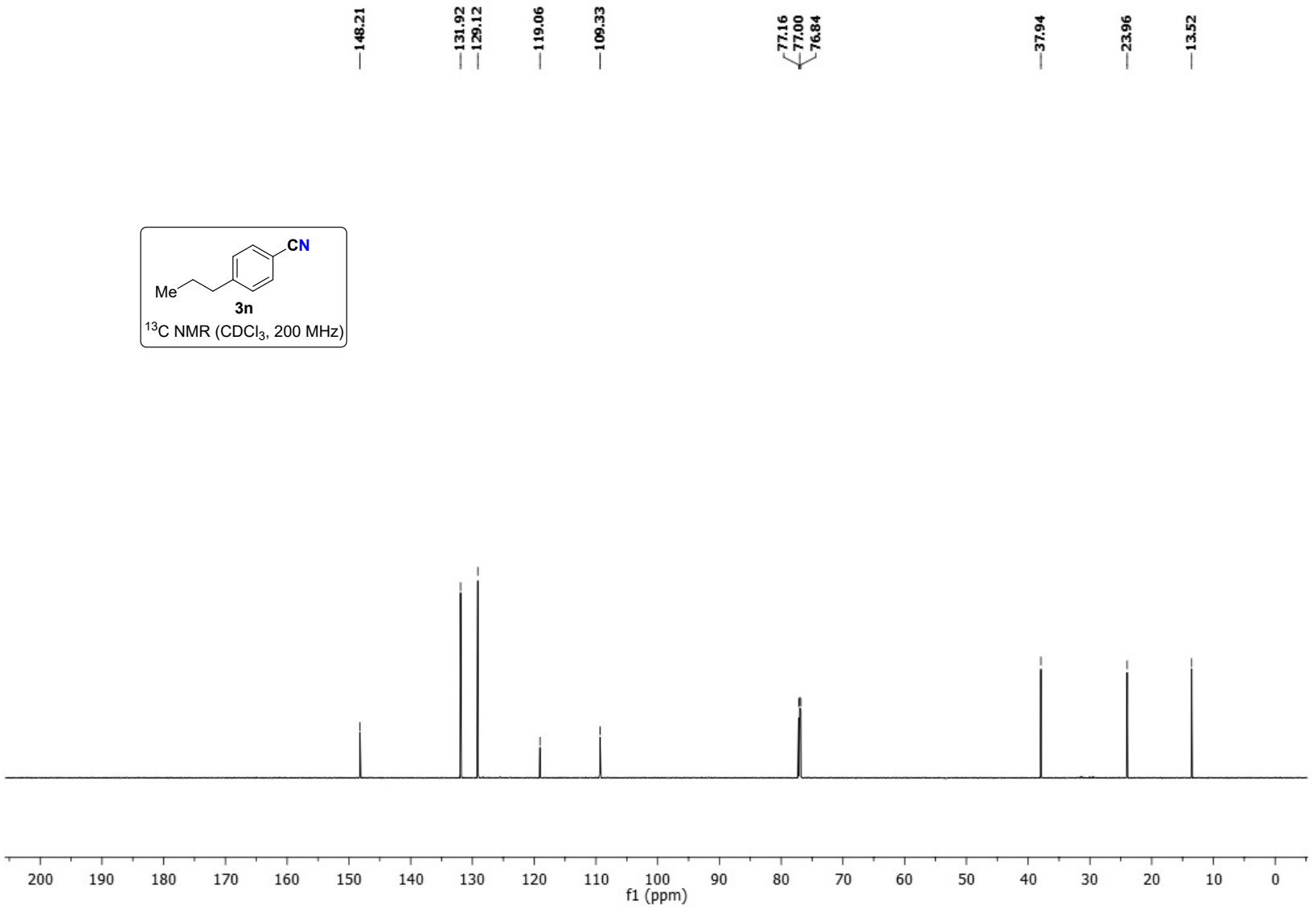

7.303
7.280
7.275
7.260
7.254
7.242
7.062
7.057
6.896
6.875
3.913
3.882
1.236
1.232
0.857
0.839
-0.024
CN
H3CO
OCH3
3l
1H NMR (CDCl3, 400 MHz)
1.39
0.97
0.98
3.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

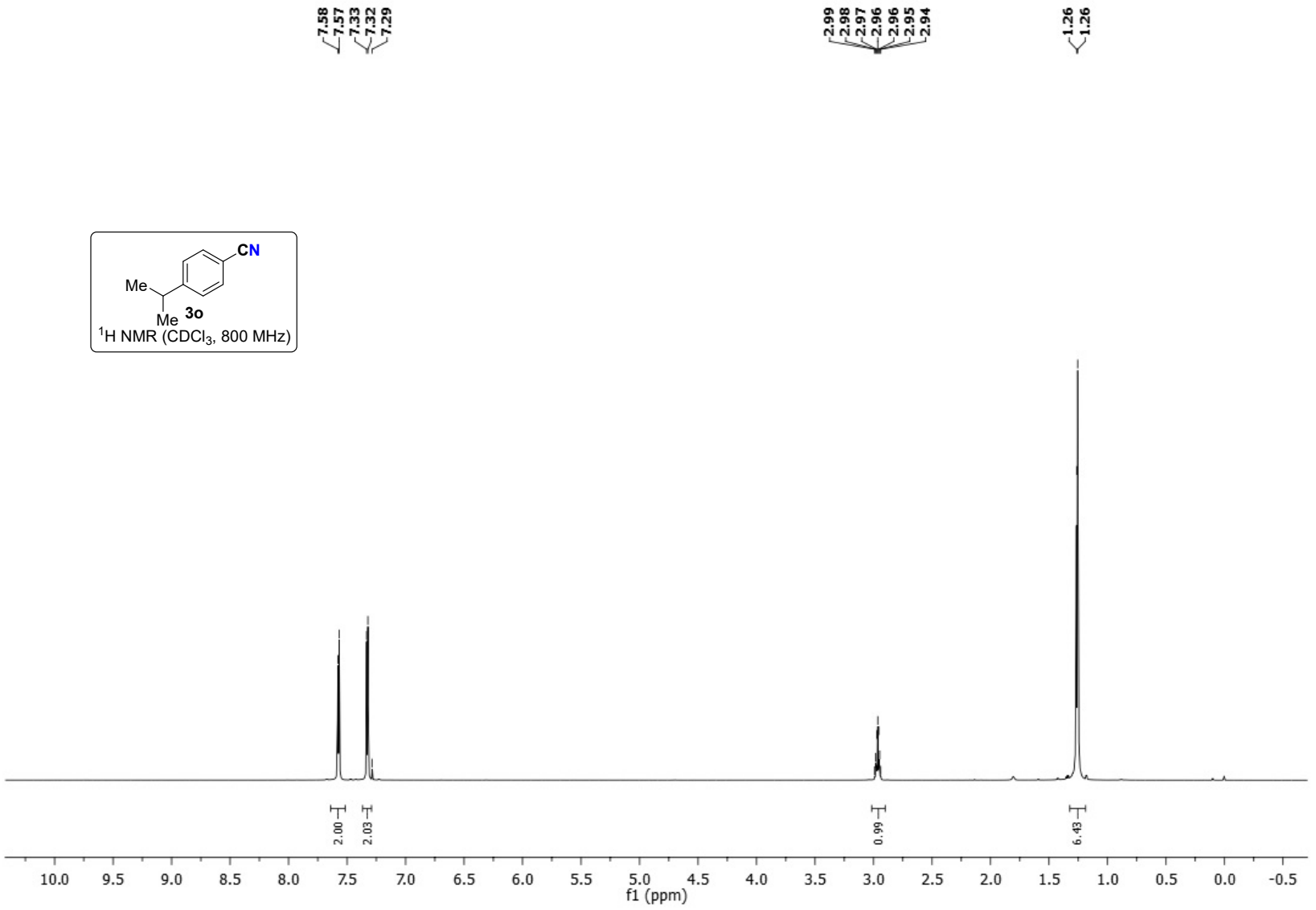

152.960
149.280
126.551
119.305
114.023
111.334
103.978
77.478
77.160
76.842
56.221
56.179
CN
H3CO
OCH3
3l
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

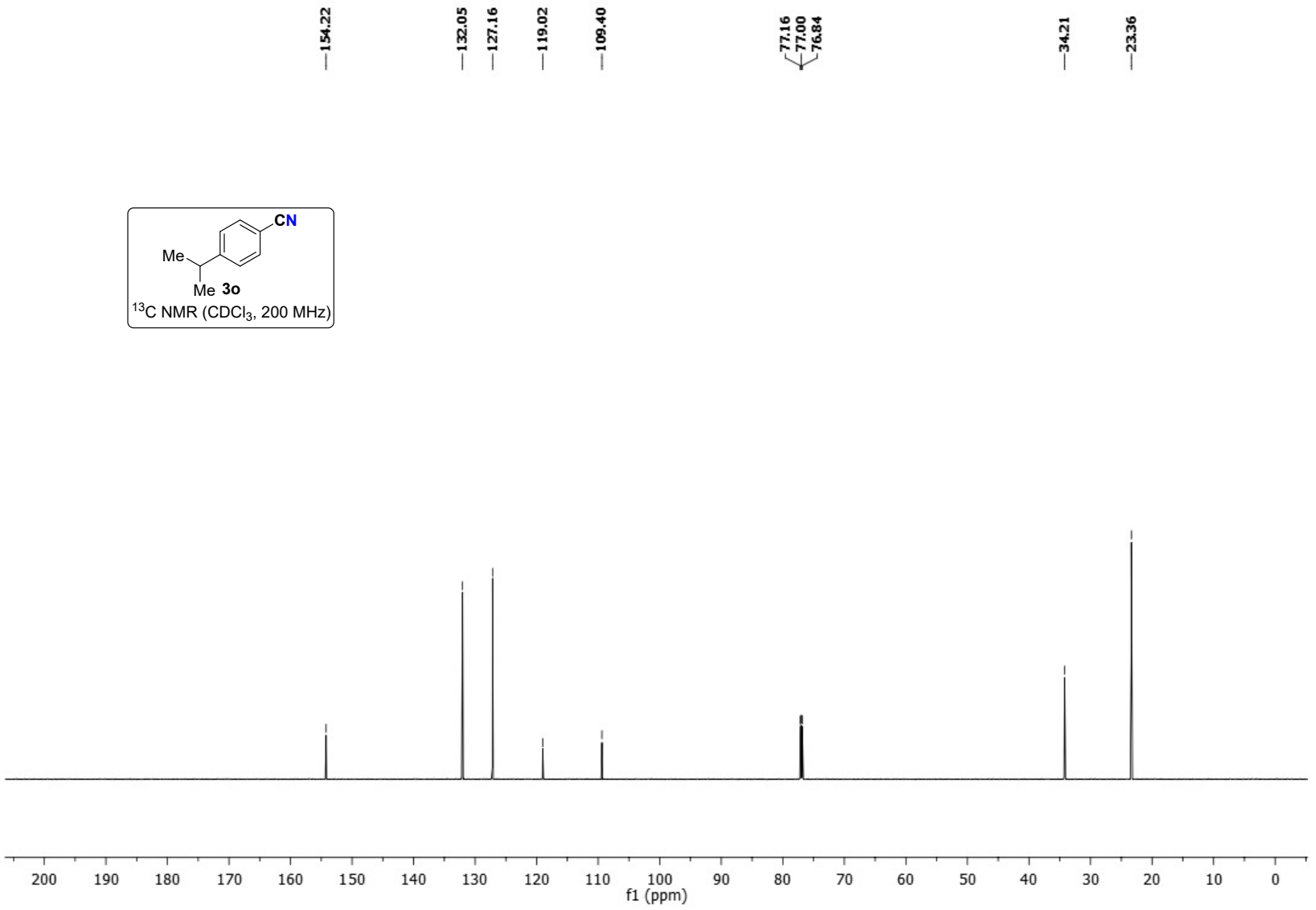

7.260
6.831
3.862
3.843
-0.043
MeO
CN
MeO
OMe
3m
1H NMR (CDCl3, 400 MHz)
2.00
3.00
6.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

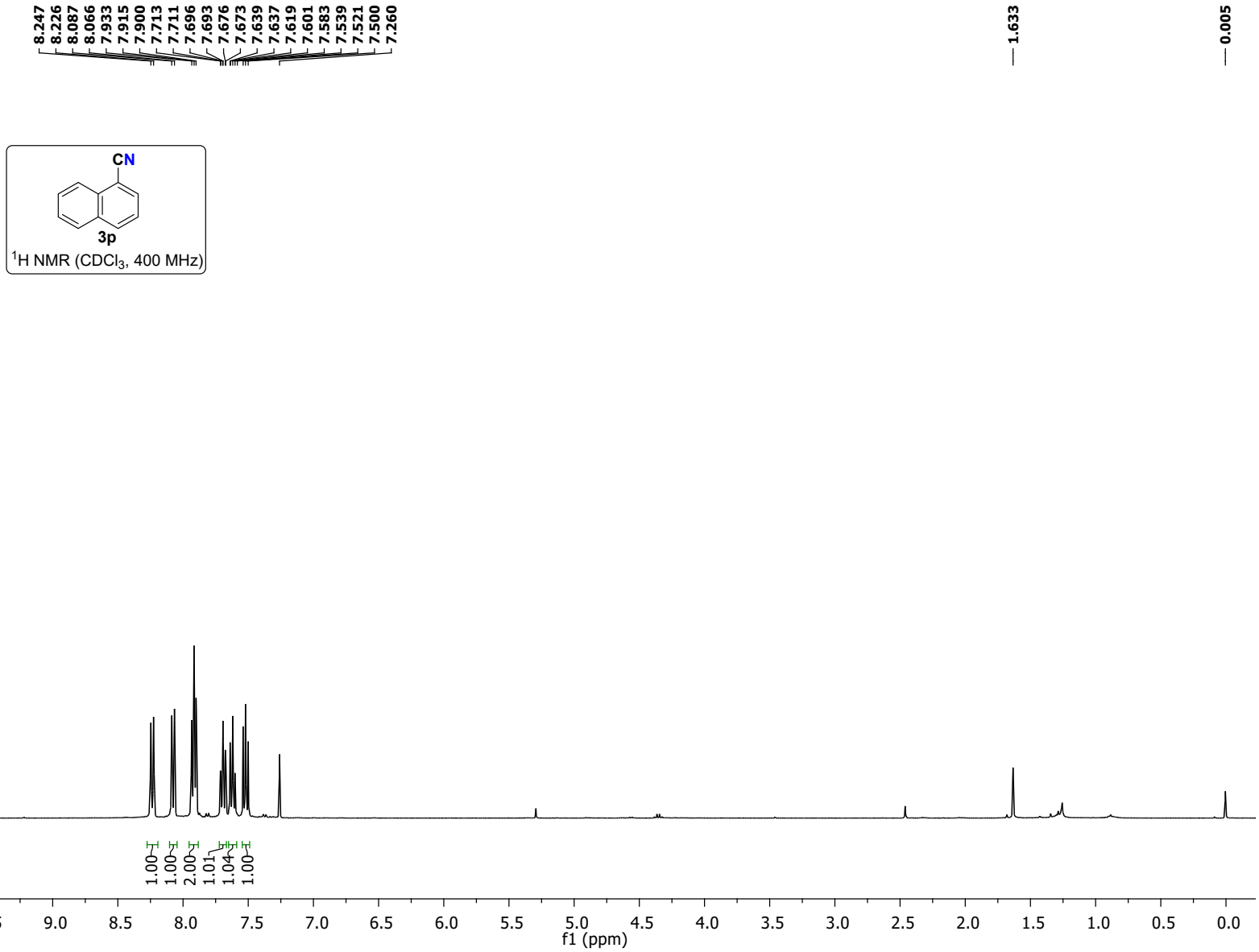

153.58
142.34
118.99
109.47
106.72
77.48
77.16
76.84
61.05
56.41
MeO
CN
MeO
OMe
3m
13C NMR (CDCl3, 100 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

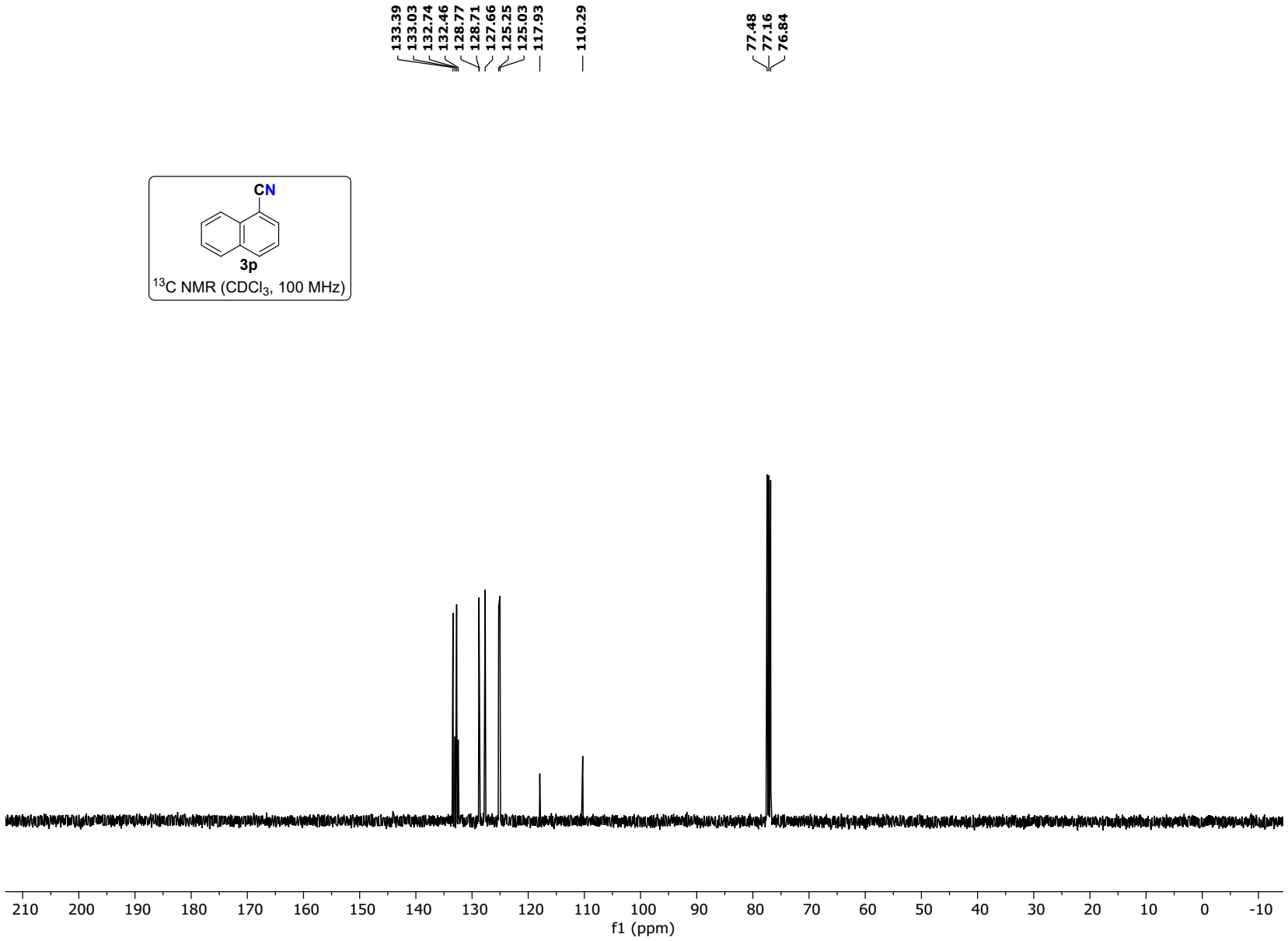

7.57
7.56
7.28
7.27
2.66
2.65
2.64
1.68
1.67
1.66
1.65
1.64
1.64
0.96
0.95
0.94
CN
Me
3n
1H NMR (CDCl3, 800 MHz)
1.88
2.00
2.00
1.99
3.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


148.21
131.92
129.12
119.06
109.33
77.16
77.00
76.84
37.94
23.96
13.52
CN
Me
3n
13C NMR (CDCl3, 200 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

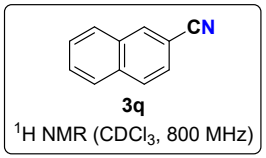

7.58
7.57
7.33
7.32
7.29
2.99
2.98
2.97
2.96
2.96
2.95
2.94
1.26
1.26
CN
Me
Me
3o
1H NMR (CDCl3, 800 MHz)
2.00
2.03
0.99
6.43
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

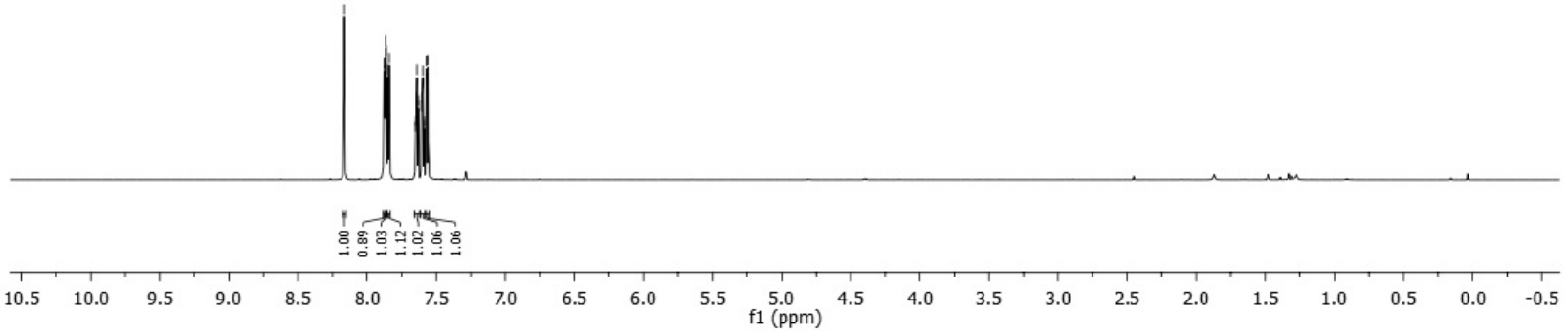

154.22
132.05
127.16
119.02
109.40
77.16
77.00
76.84
34.21
23.36
CN
Me
Me
3o
13C NMR (CDCl3, 200 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

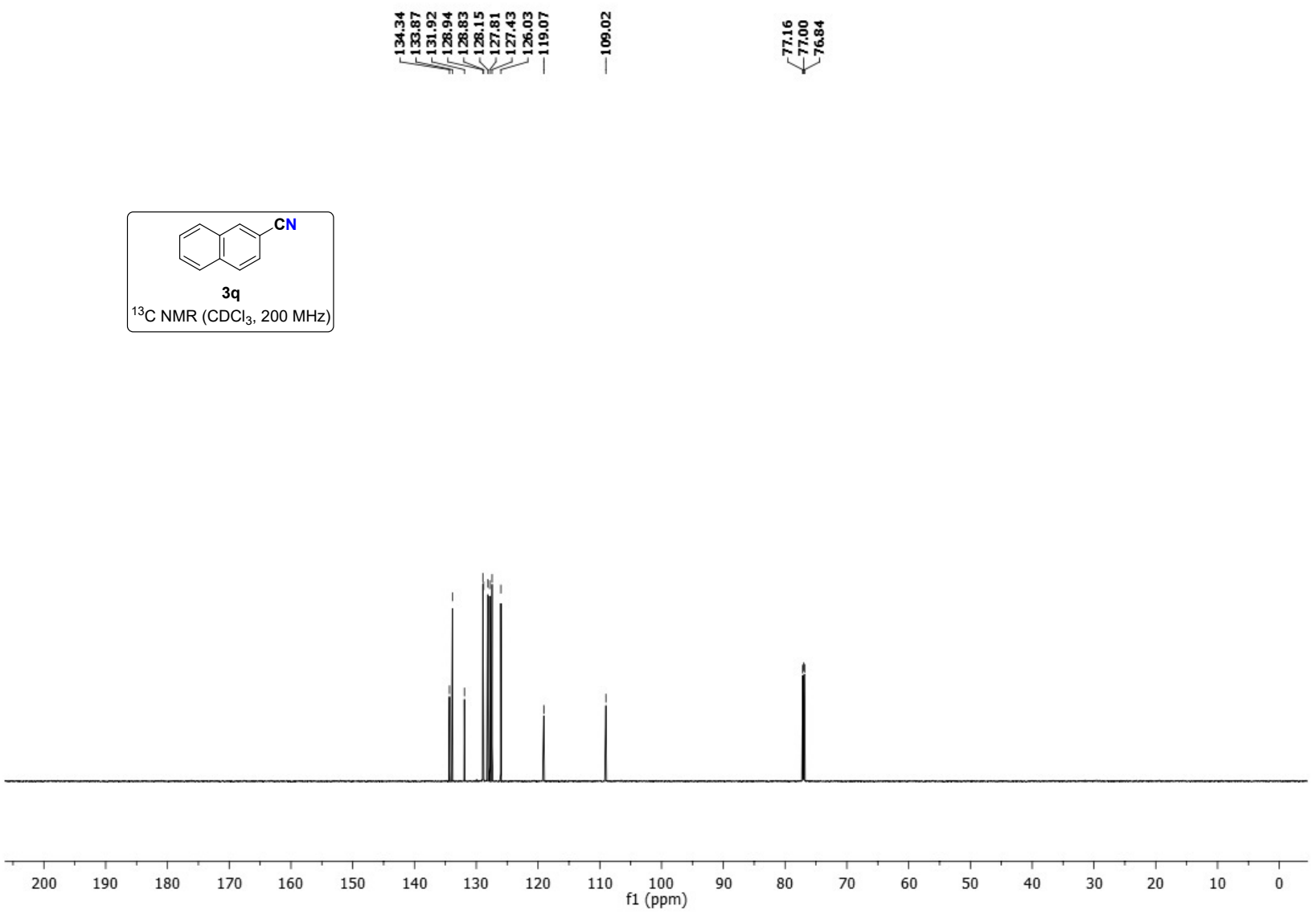

8.247
8.226
8.087
8.066
7.933
7.915
7.900
7.713
7.711
7.696
7.693
7.676
7.673
7.639
7.637
7.619
7.601
7.583
7.539
7.521
7.500
7.260
1.633
0.005
CN
3p
1H NMR (CDCl3, 400 MHz)
1.00
1.00
2.00
1.01
1.04
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

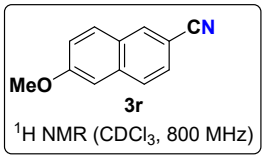

133.39
133.03
132.74
132.46
128.77
128.71
127.66
125.25
125.03
117.93
110.29
77.48
77.16
76.84
CN
3p
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.16
7.88
7.87
7.87
7.86
7.85
7.84
7.65
7.65
7.64
7.64
7.63
7.63
7.61
7.61
7.60
7.60
7.59
7.59
7.57
7.57
7.56
7.56


CN
3q
^{1}H NMR (CDCl$_3$, 800 MHz)

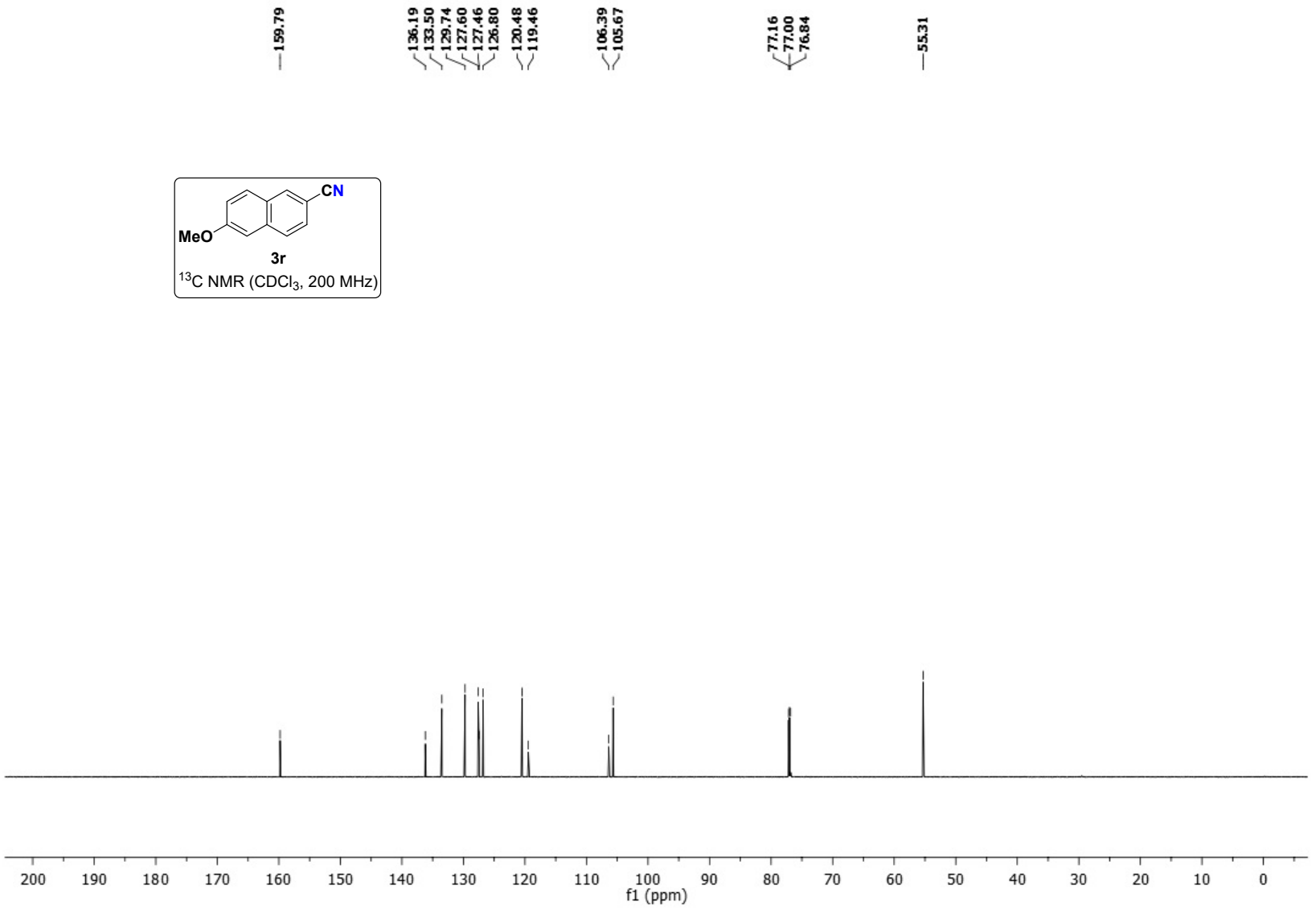

1.00
0.89
1.03
1.12
1.02
1.06
1.06
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


134.34
133.87
131.92
128.94
128.83
128.15
127.81
127.43
126.03
119.07
109.02
77.16
77.00
76.84
CN
3q
13C NMR (CDCl3, 200 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

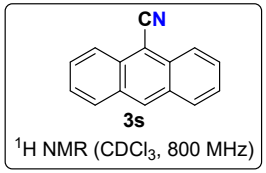


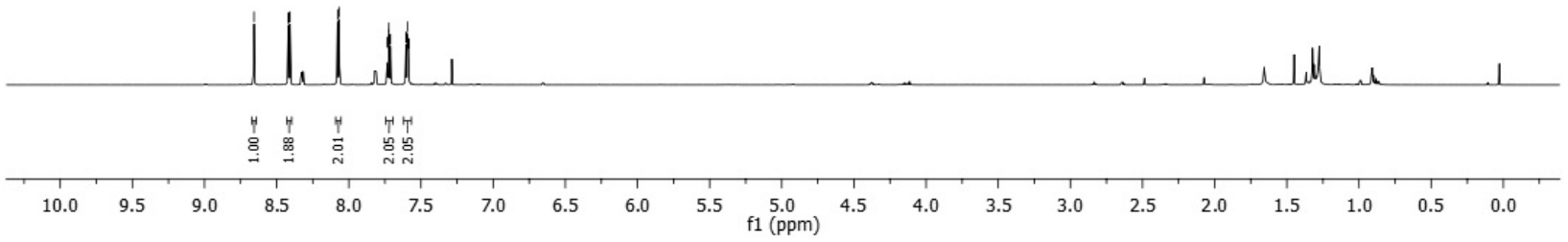
3r

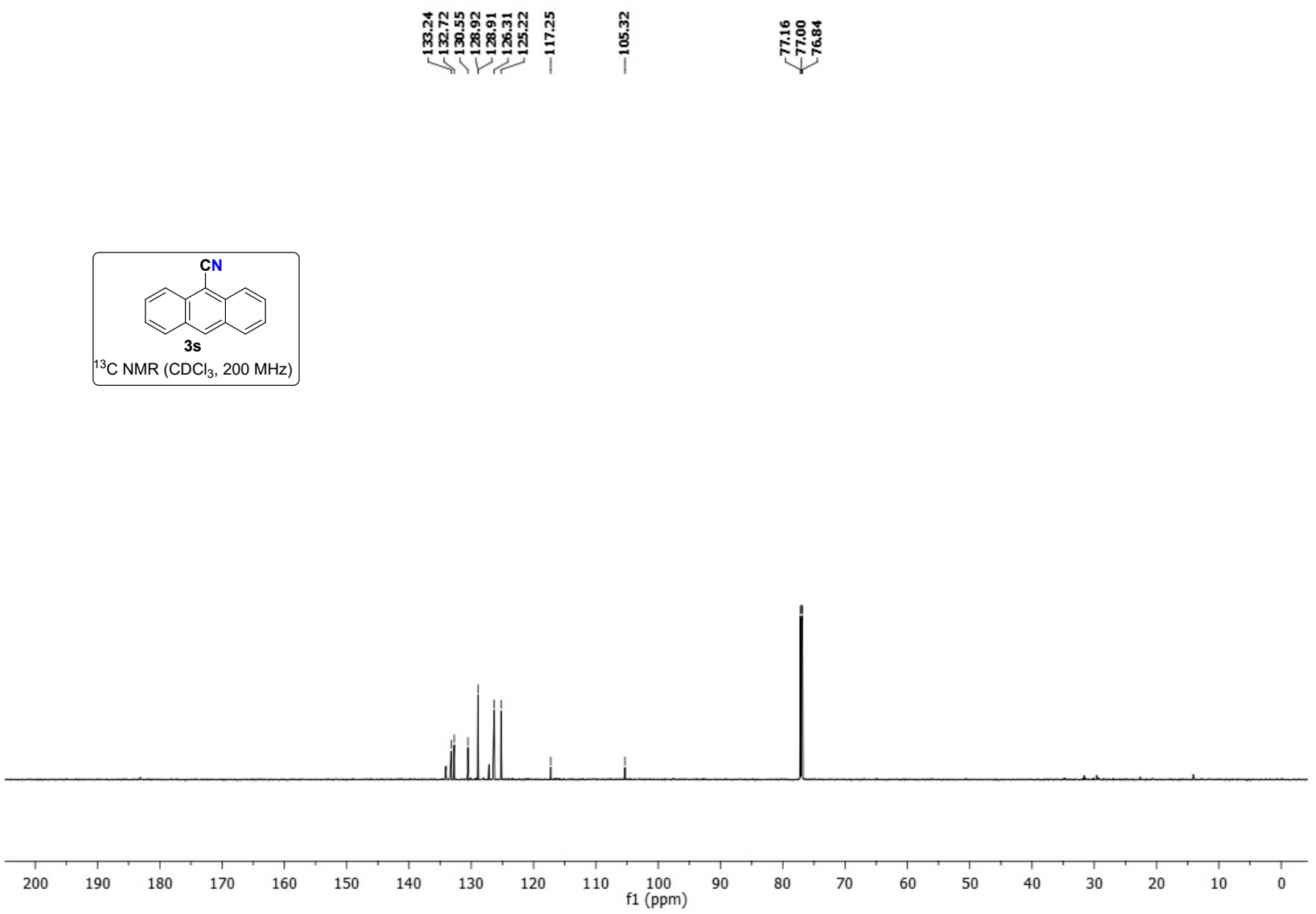
^{1}H NMR ($CDCl_3$, 800 MHz)

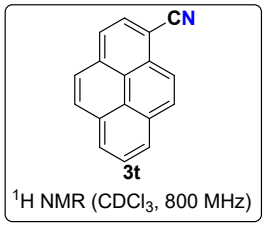

159.79
136.19
133.50
129.74
127.60
127.46
126.80
120.48
119.46
106.39
105.67
77.16
77.00
76.84
55.31
CN
MeO
3r
13C NMR (CDCl3, 200 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


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


CN
3s
^{1}H NMR ($CDCl_3$, 800 MHz)

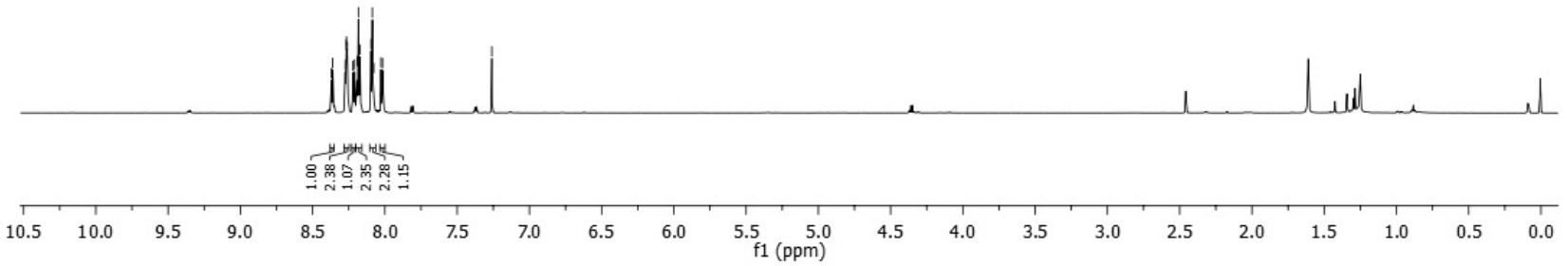

1.00
1.88
2.01
2.05
2.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

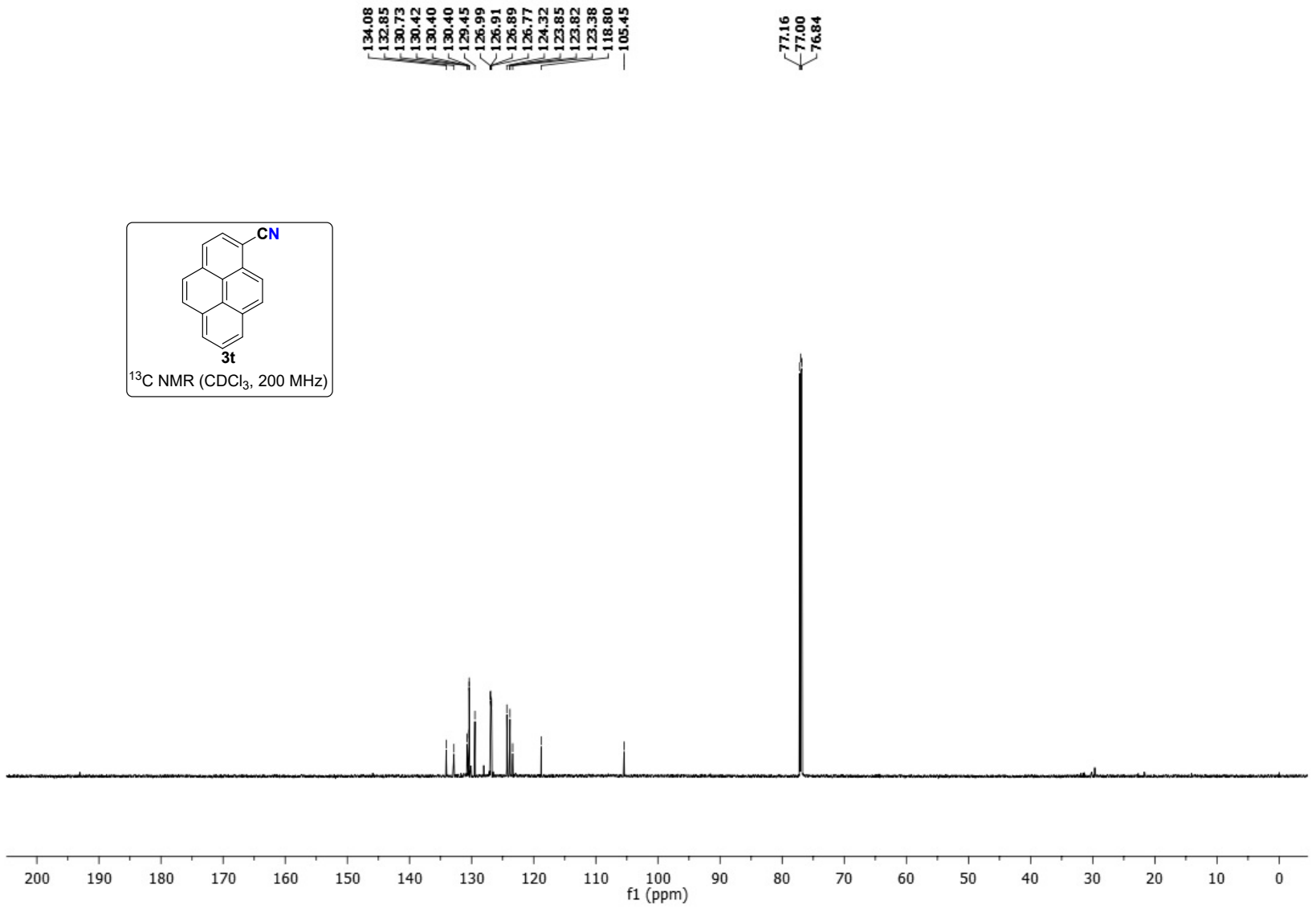

133.24
132.72
130.55
128.92
128.91
126.31
125.22
117.25
105.32
77.16
77.00
76.84
CN
3s
13C NMR (CDCl3, 200 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

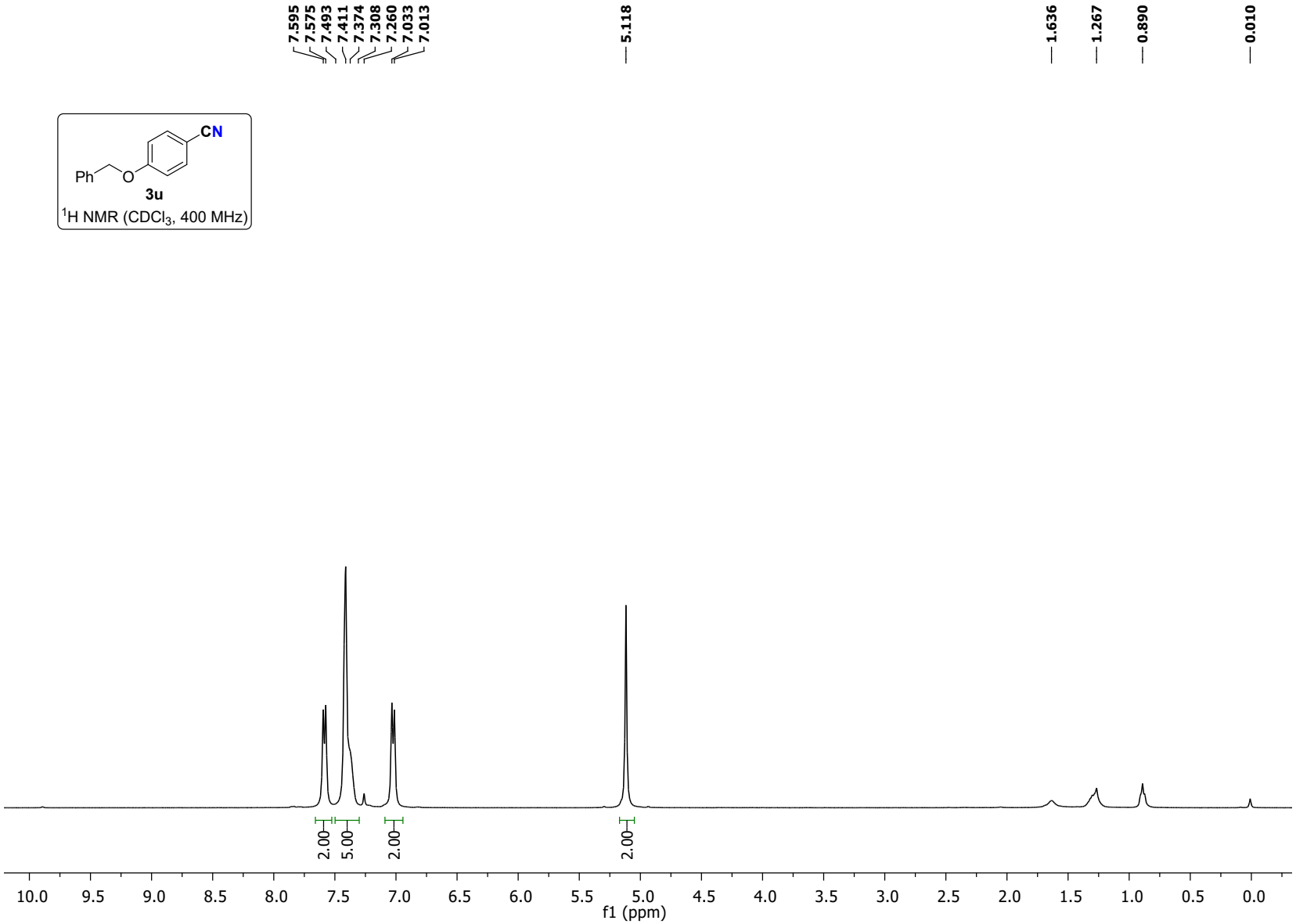


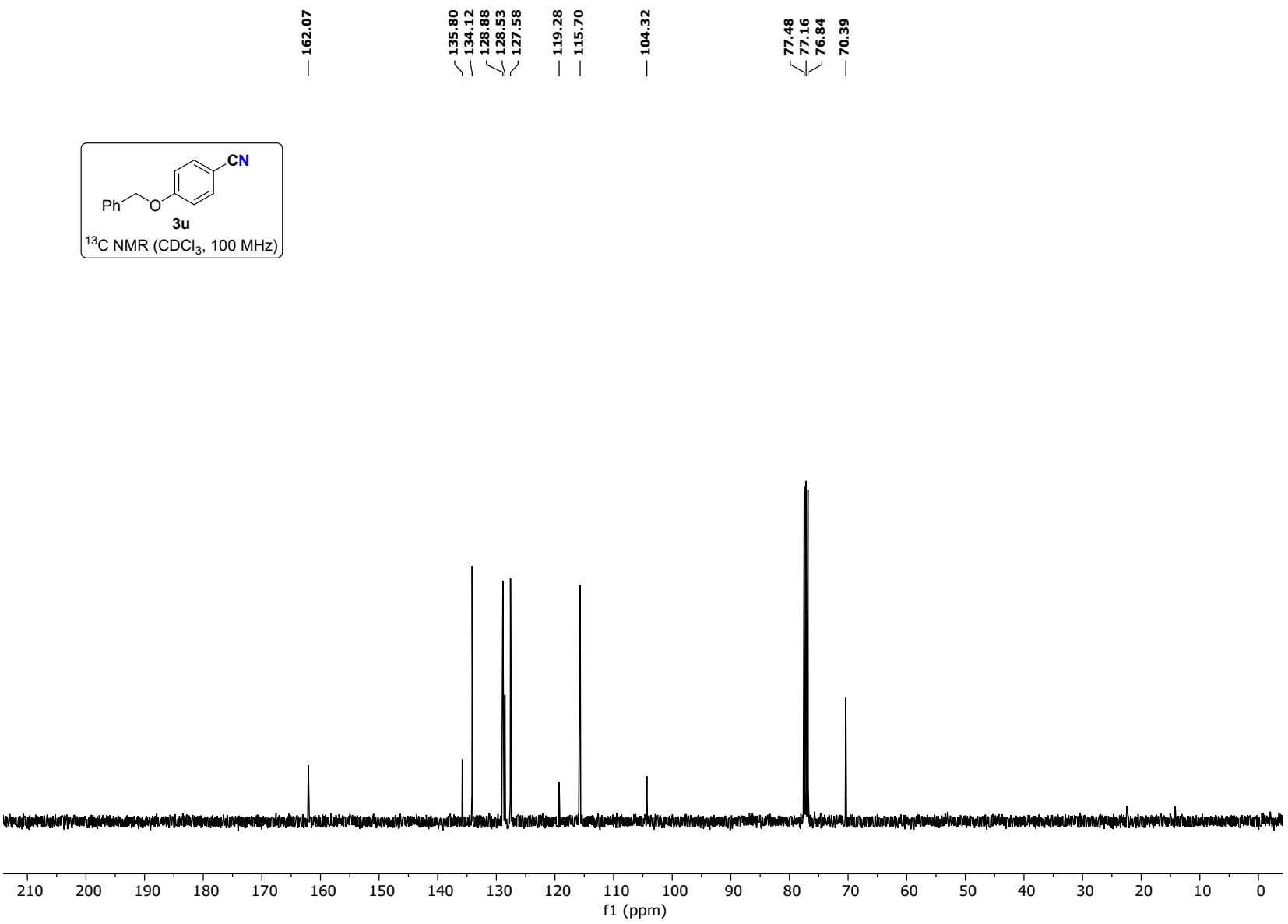
3t

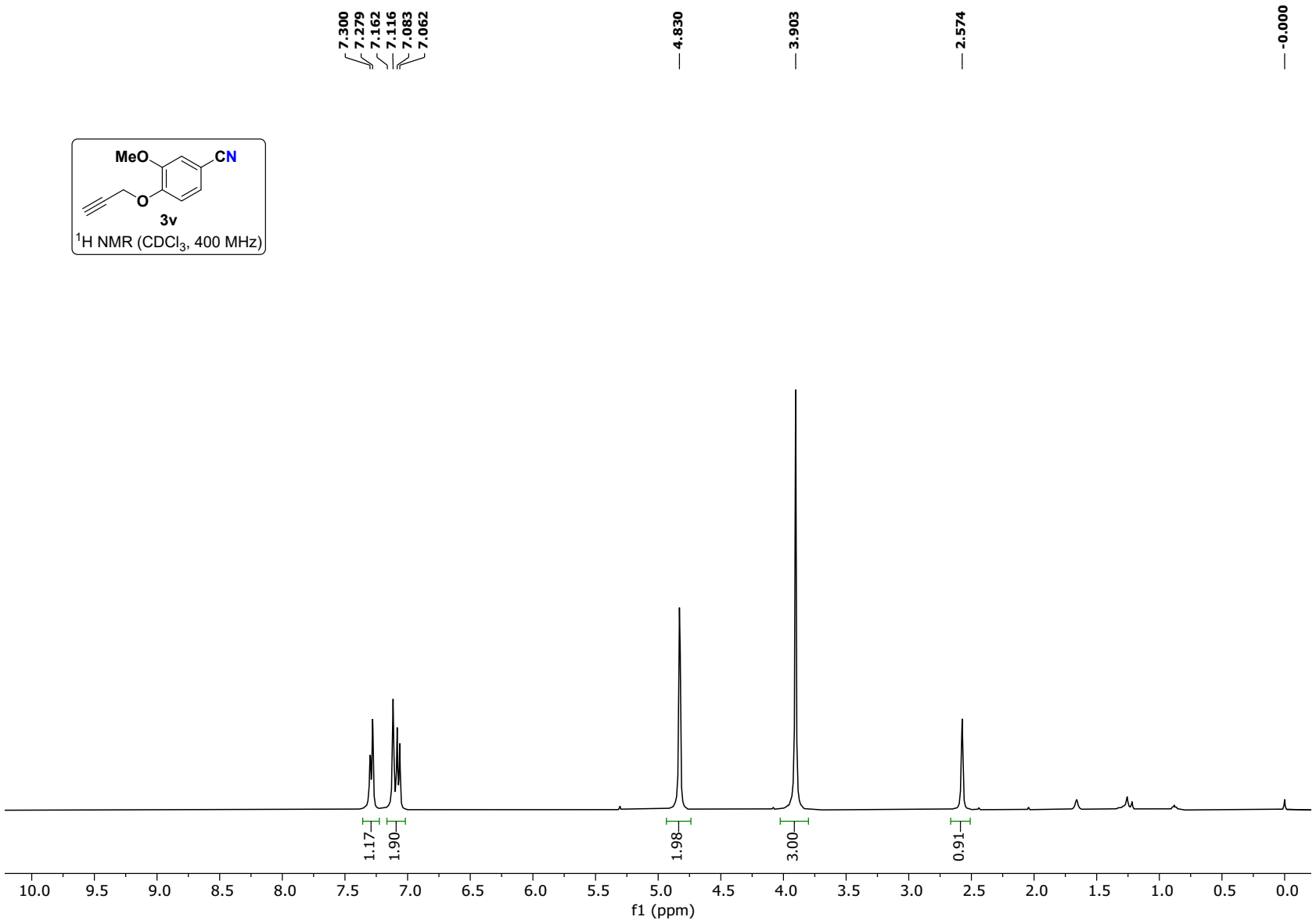
^{1}H NMR ($CDCl_3$, 800 MHz)

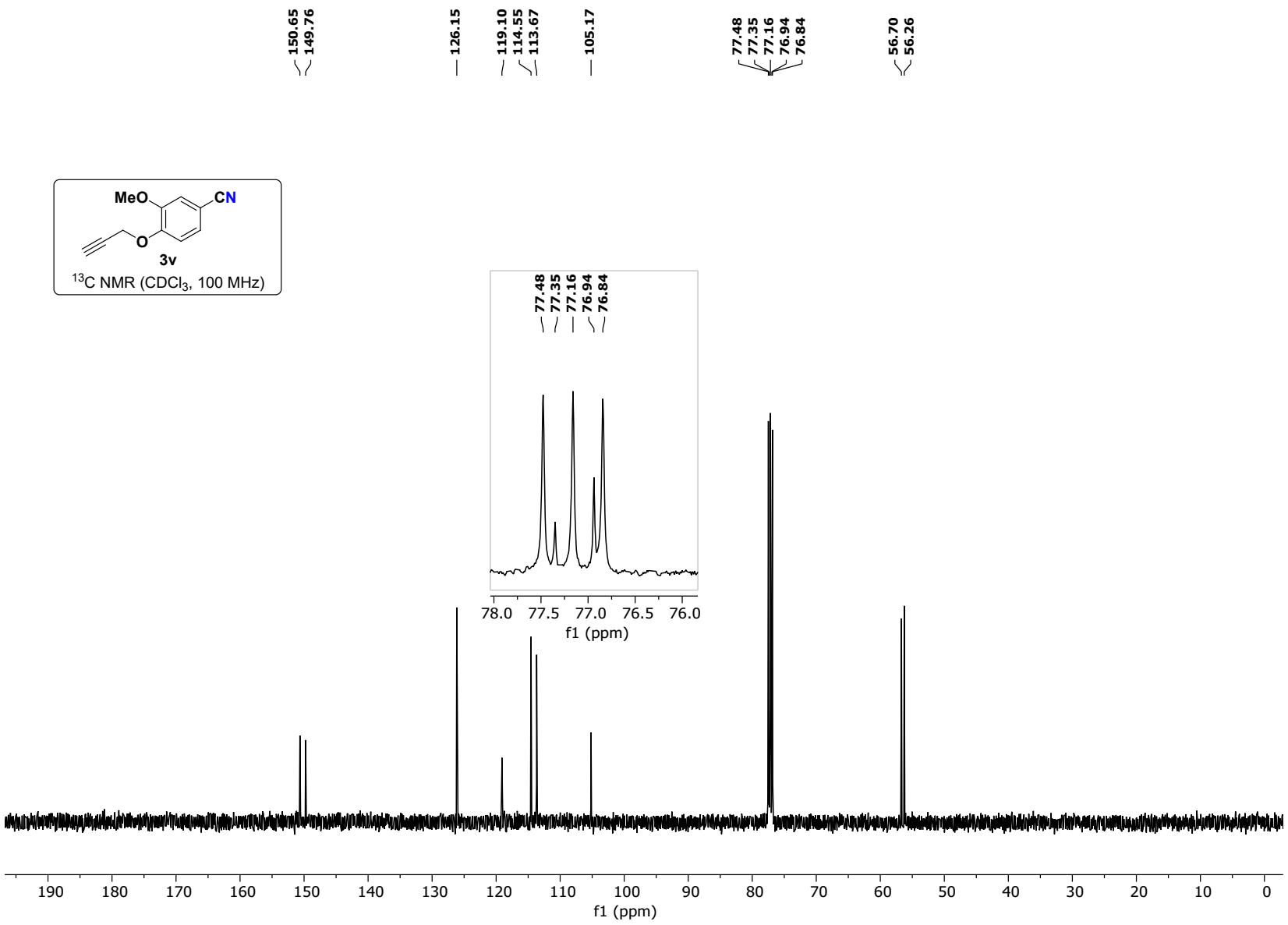


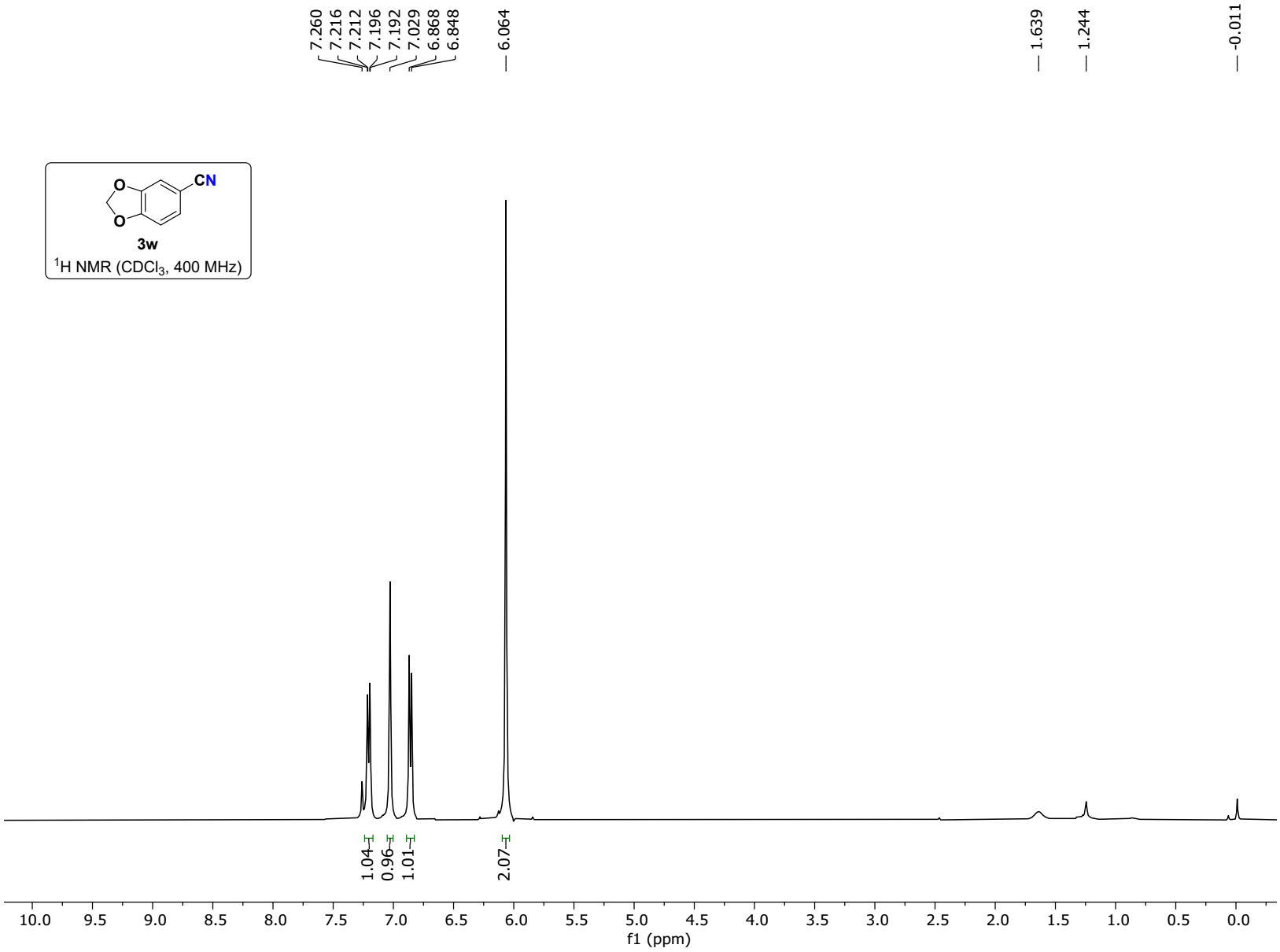

134.08
132.85
130.73
130.42
130.40
130.40
129.45
126.99
126.91
126.89
126.77
124.32
123.85
123.82
123.38
118.80
105.45
77.16
77.00
76.84
CN
3t
13C NMR (CDCl3, 200 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

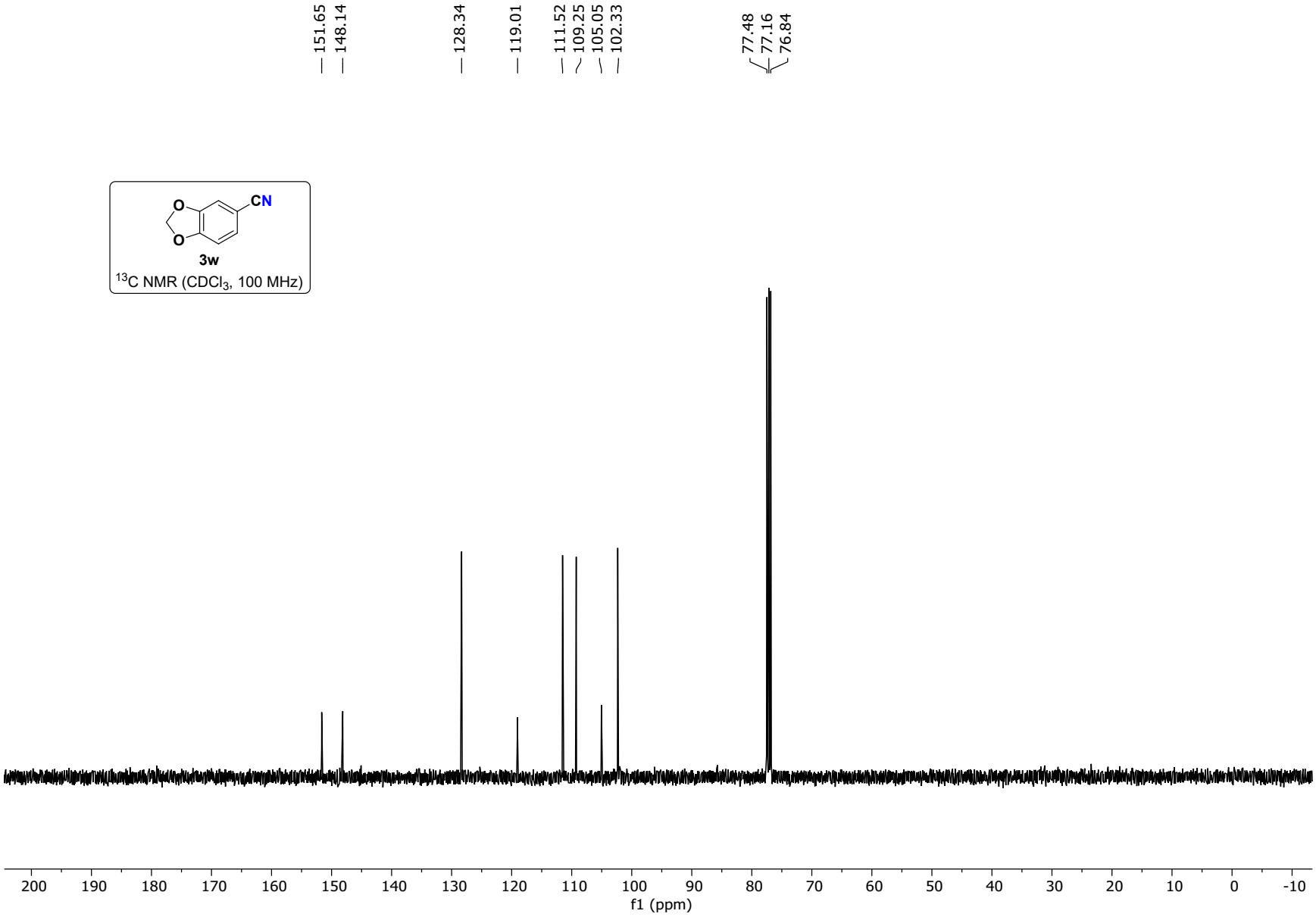

7.595
7.575
7.493
7.411
7.374
7.308
7.260
7.033
7.013
5.118
1.636
1.267
0.890
0.010
CN
Ph
O
3u
^{1}H NMR ($CDCl_3$, 400 MHz)
2.00
5.00
2.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

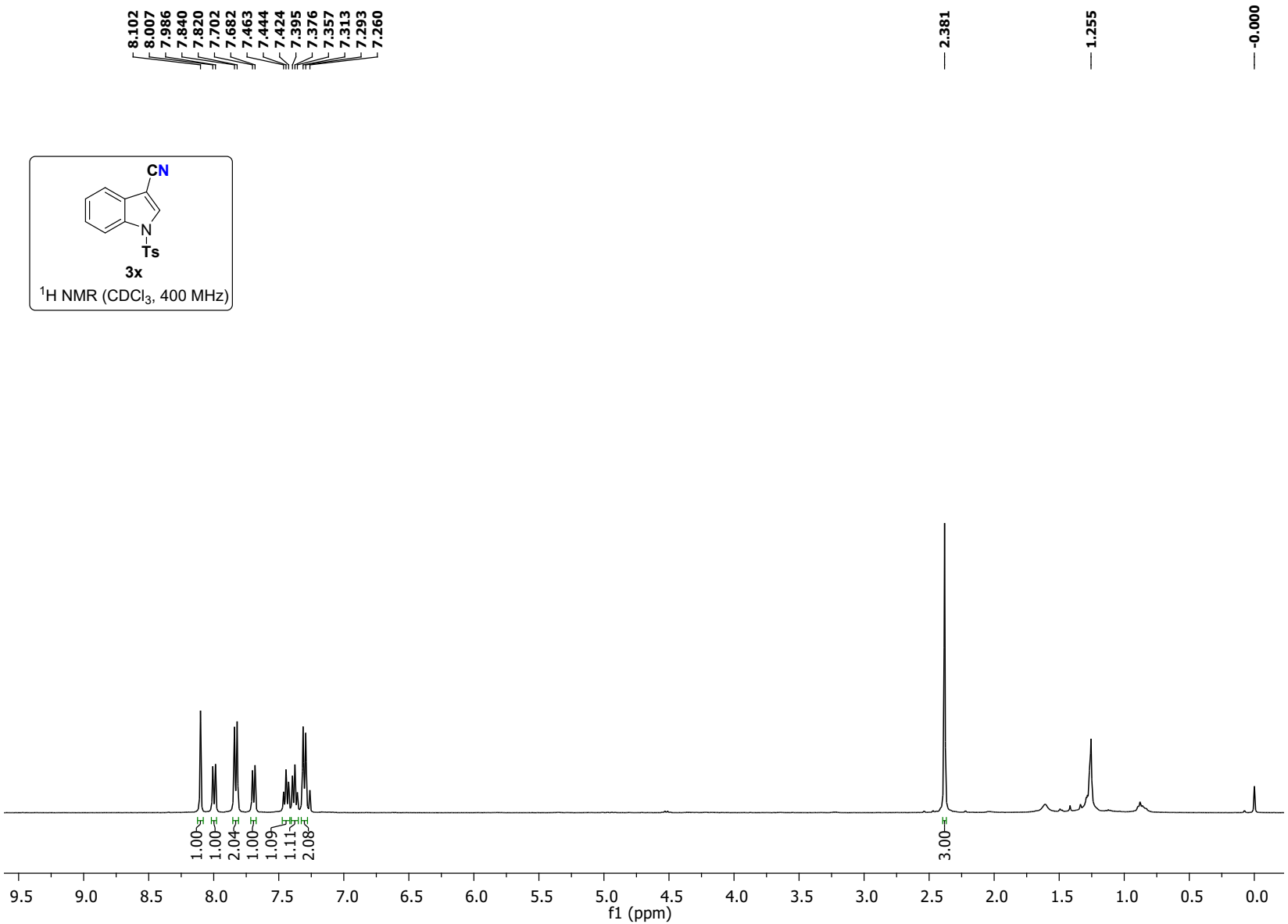

162.07
135.80
134.12
128.88
128.53
127.58
119.28
115.70
104.32
77.48
77.16
76.84
70.39
CN
Ph
O
3u
13C NMR (CDCl3, 100 MHz)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

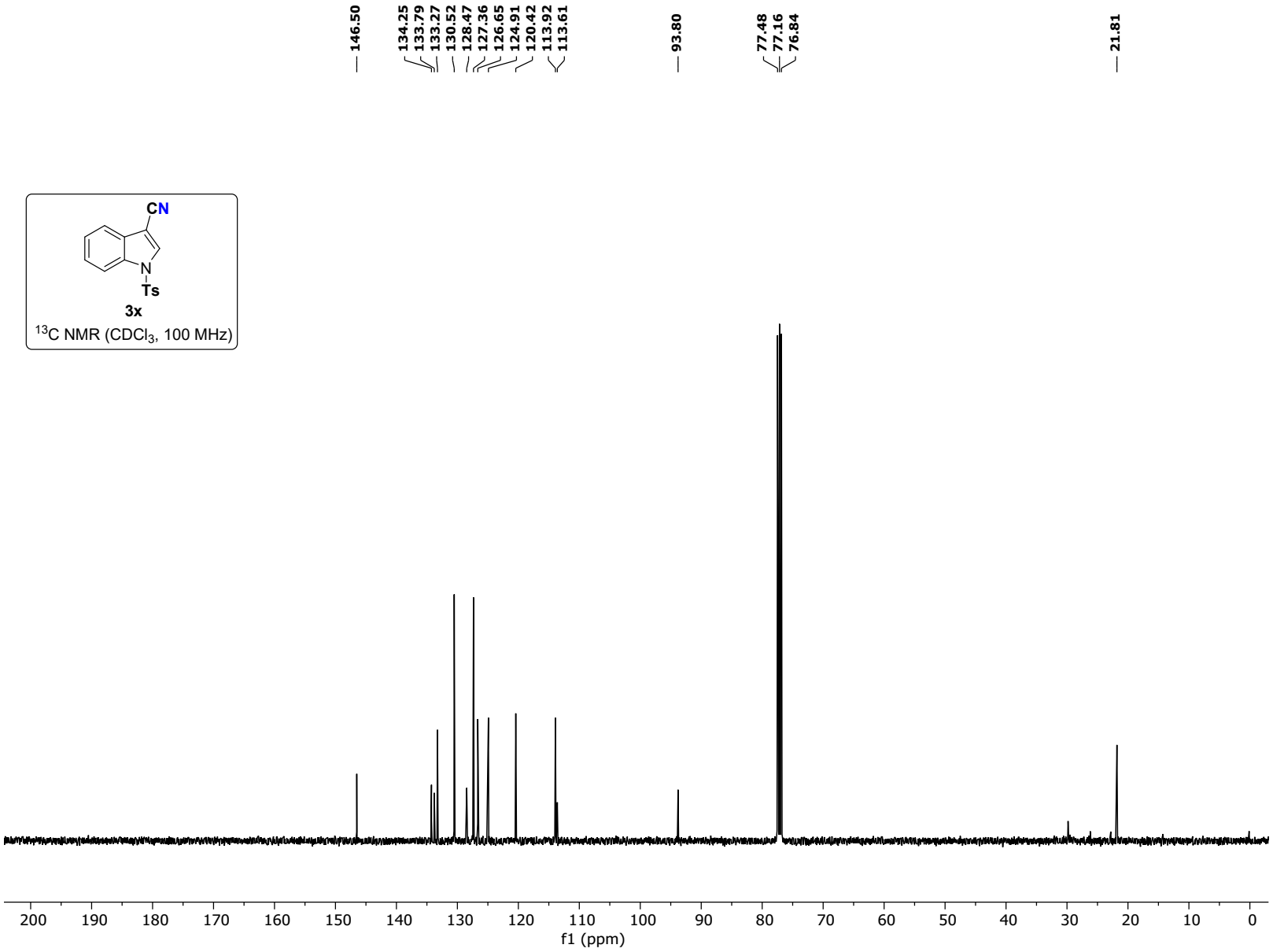

7.300
7.279
7.162
7.116
7.083
7.062
4.830
3.903
2.574
-0.000
MeO
CN
O
3v
^{1}H NMR ($CDCl_3$, 400 MHz)
1.17
1.90
1.98
3.00
0.91
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

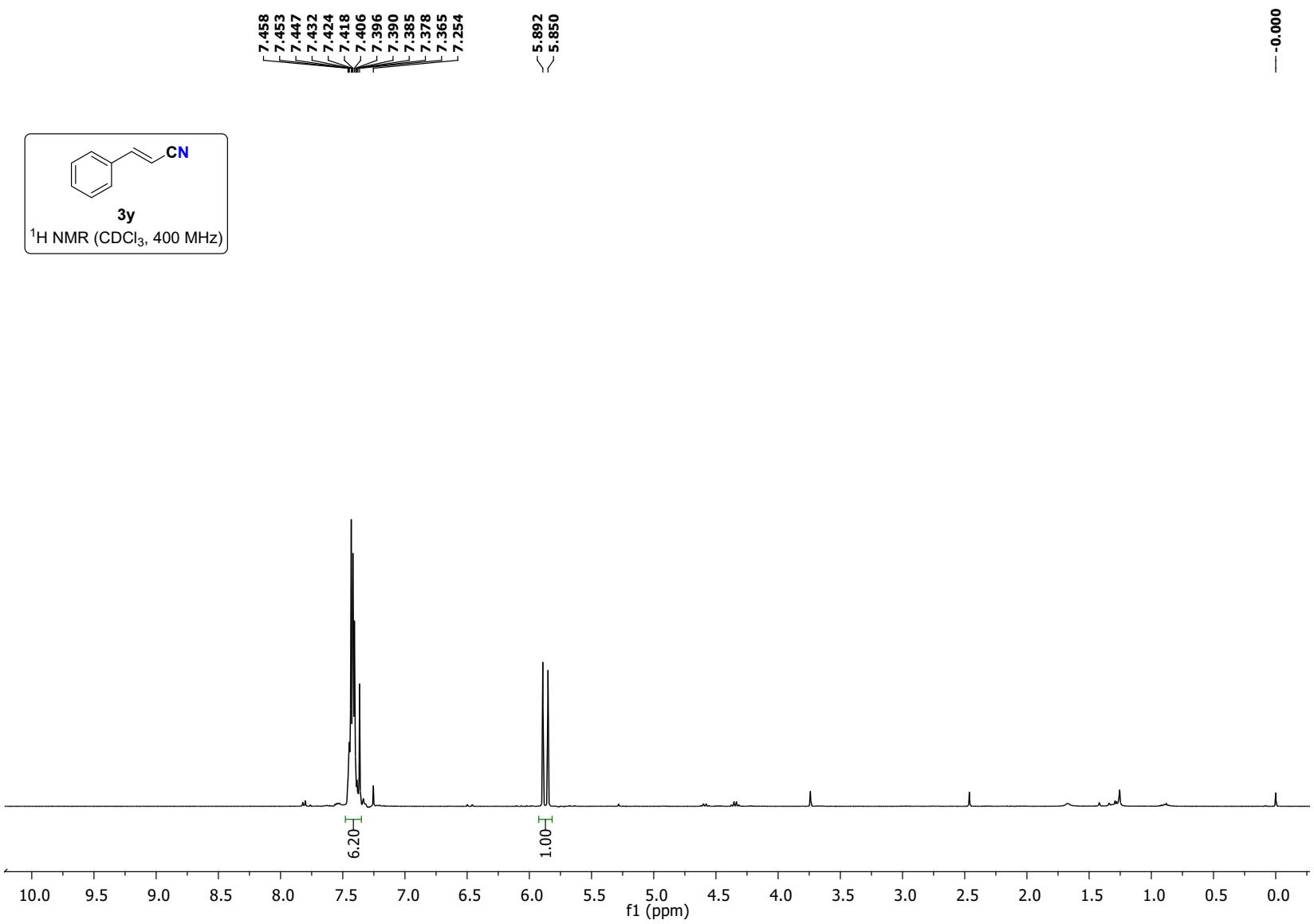

150.65
149.76
126.15
119.10
114.55
113.67
105.17
77.48
77.35
77.16
76.94
76.84
56.70
56.26
MeO
CN
O
3v
13C NMR (CDCl3, 100 MHz)
78.0
77.5
77.0
76.5
76.0
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

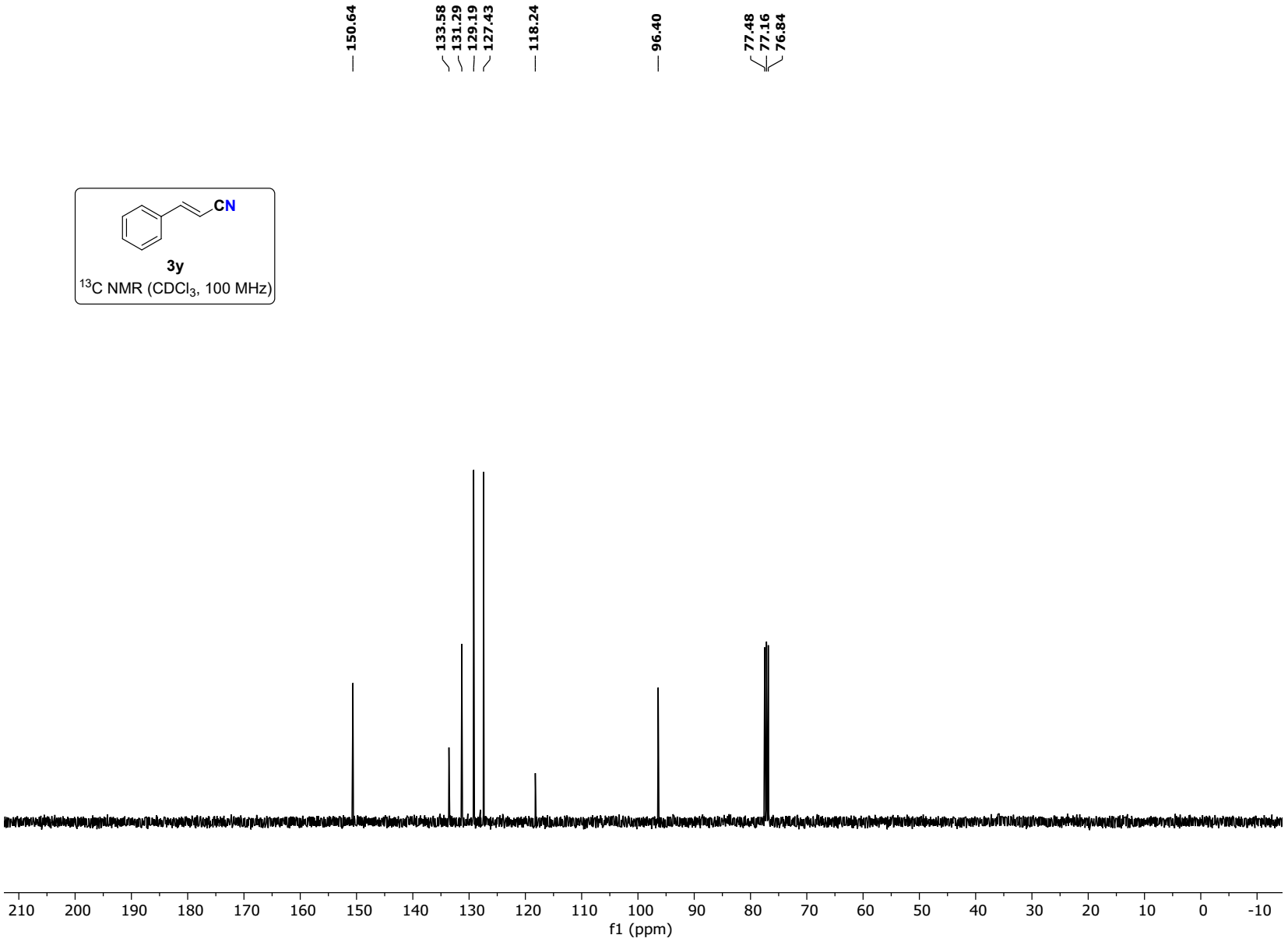

7.260
7.216
7.212
7.196
7.192
7.029
6.868
6.848
6.064
1.639
1.244
-0.011
CN
O
O
3w
1H NMR (CDCl3, 400 MHz)
1.04
0.96
1.01
2.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

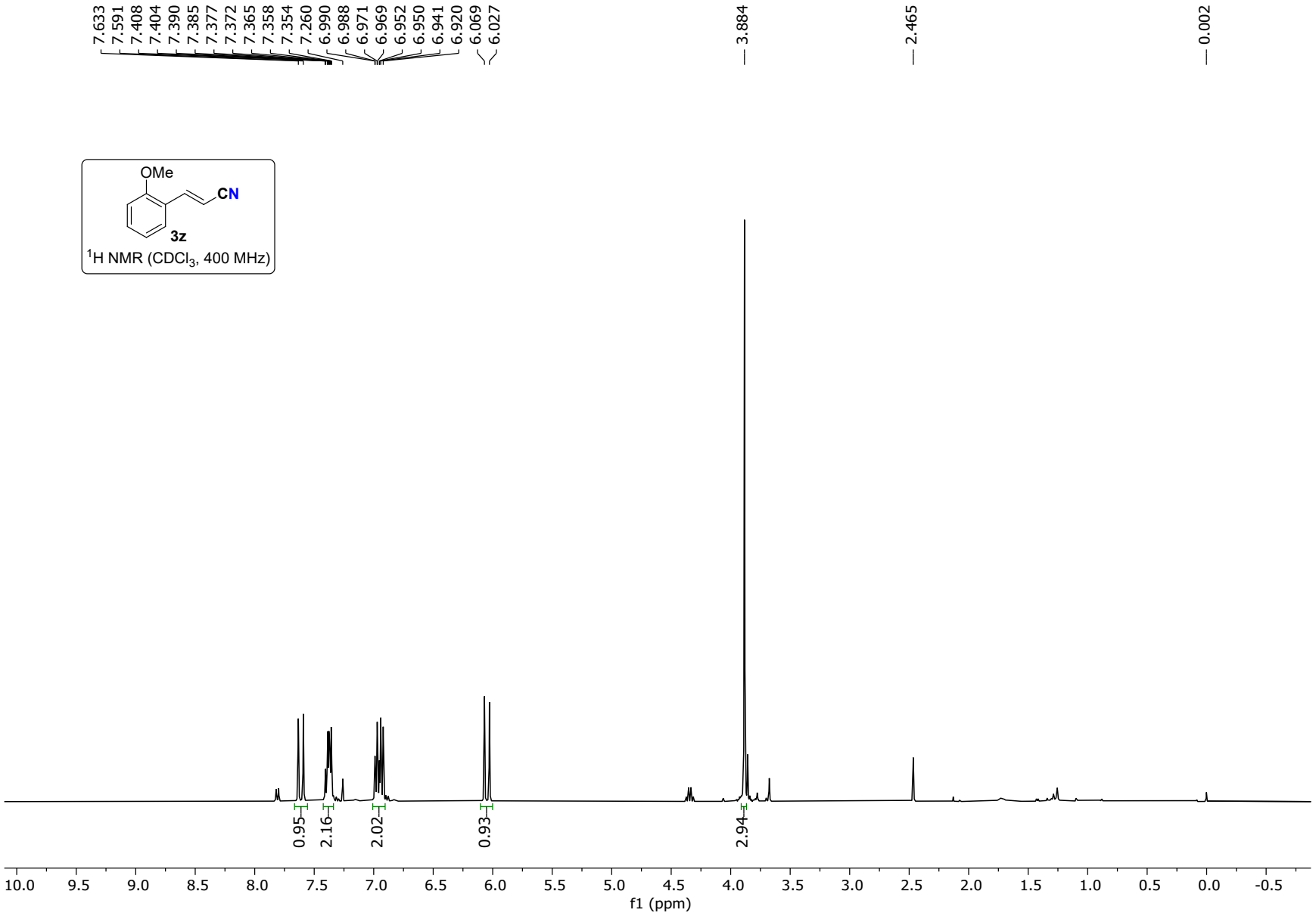

151.65
148.14
128.34
119.01
111.52
109.25
105.05
102.33
77.48
77.16
76.84
CN
O
O
3w
13C NMR (CDCl3, 100 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

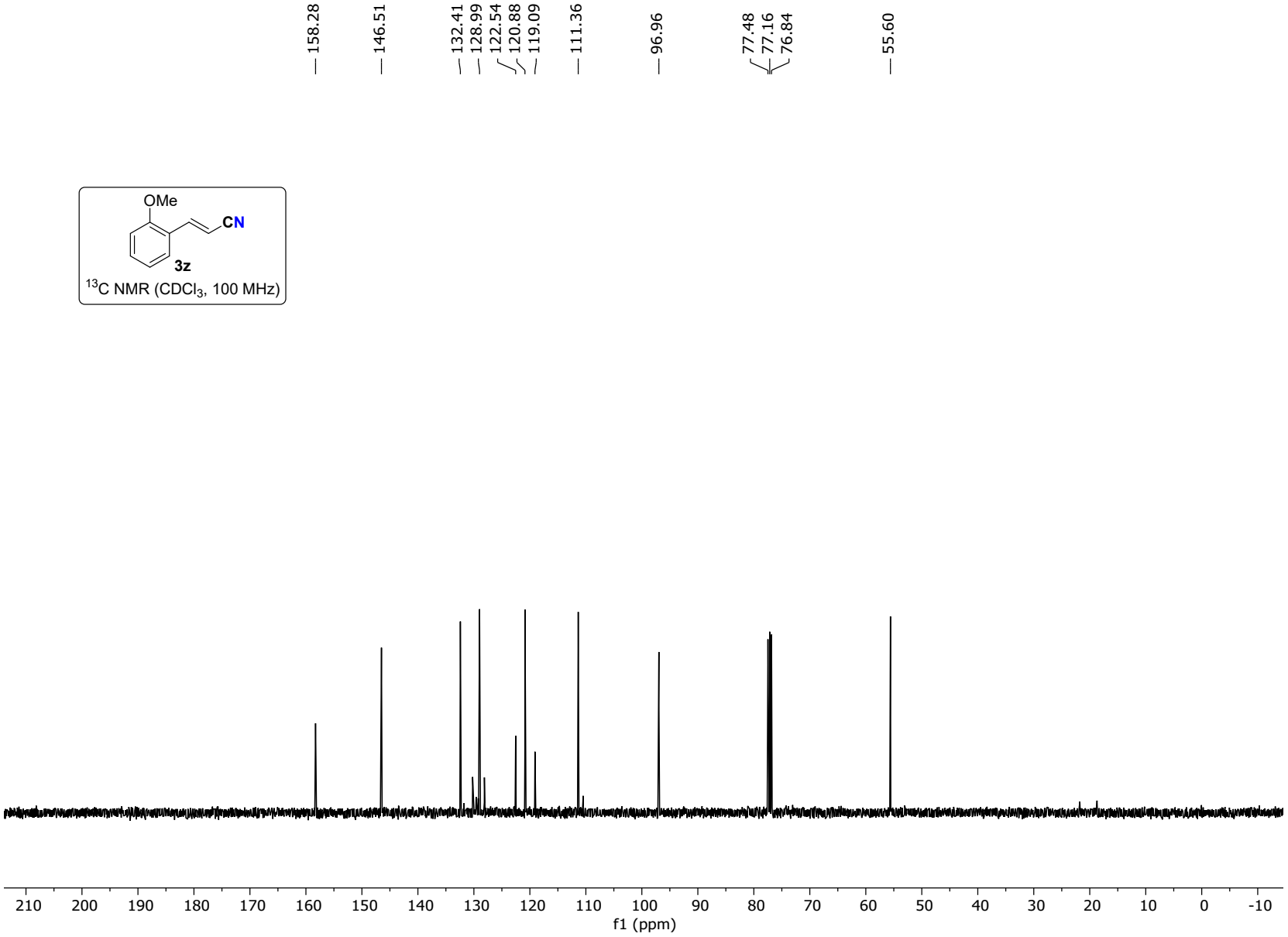

8.102
8.007
7.986
7.840
7.820
7.702
7.682
7.463
7.444
7.424
7.395
7.376
7.357
7.313
7.293
7.260
2.381
1.255
-0.000
CN
N
Ts
3x
^{1}H NMR ($CDCl_3$, 400 MHz)
1.00
1.00
2.04
1.00
1.09
1.11
2.08
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

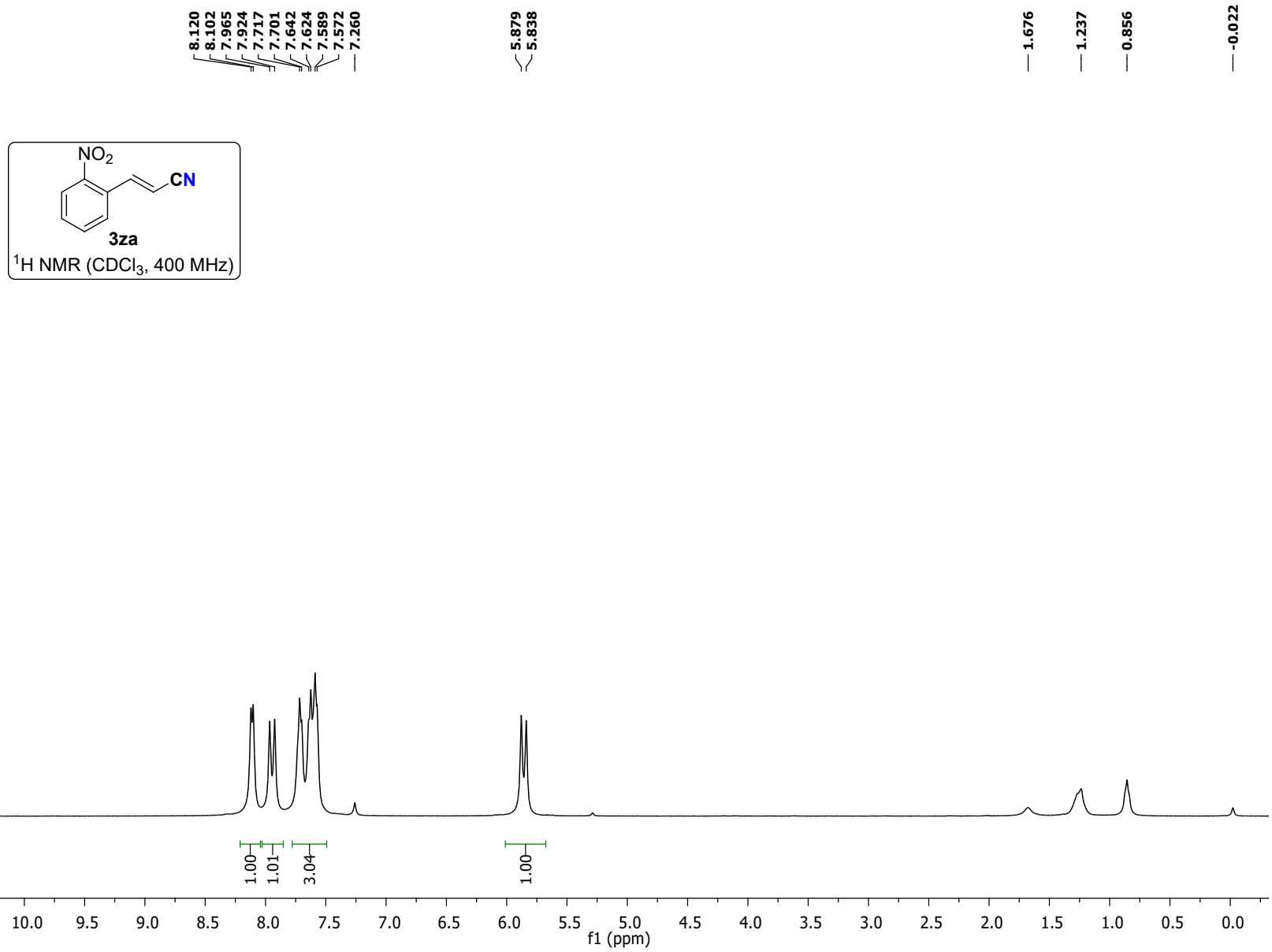

146.50
134.25
133.79
133.27
130.52
128.47
127.36
126.65
124.91
120.42
113.92
113.61
93.80
77.48
77.16
76.84
21.81
CN
N
Ts
3x
13C NMR (CDCl3, 100 MHz)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

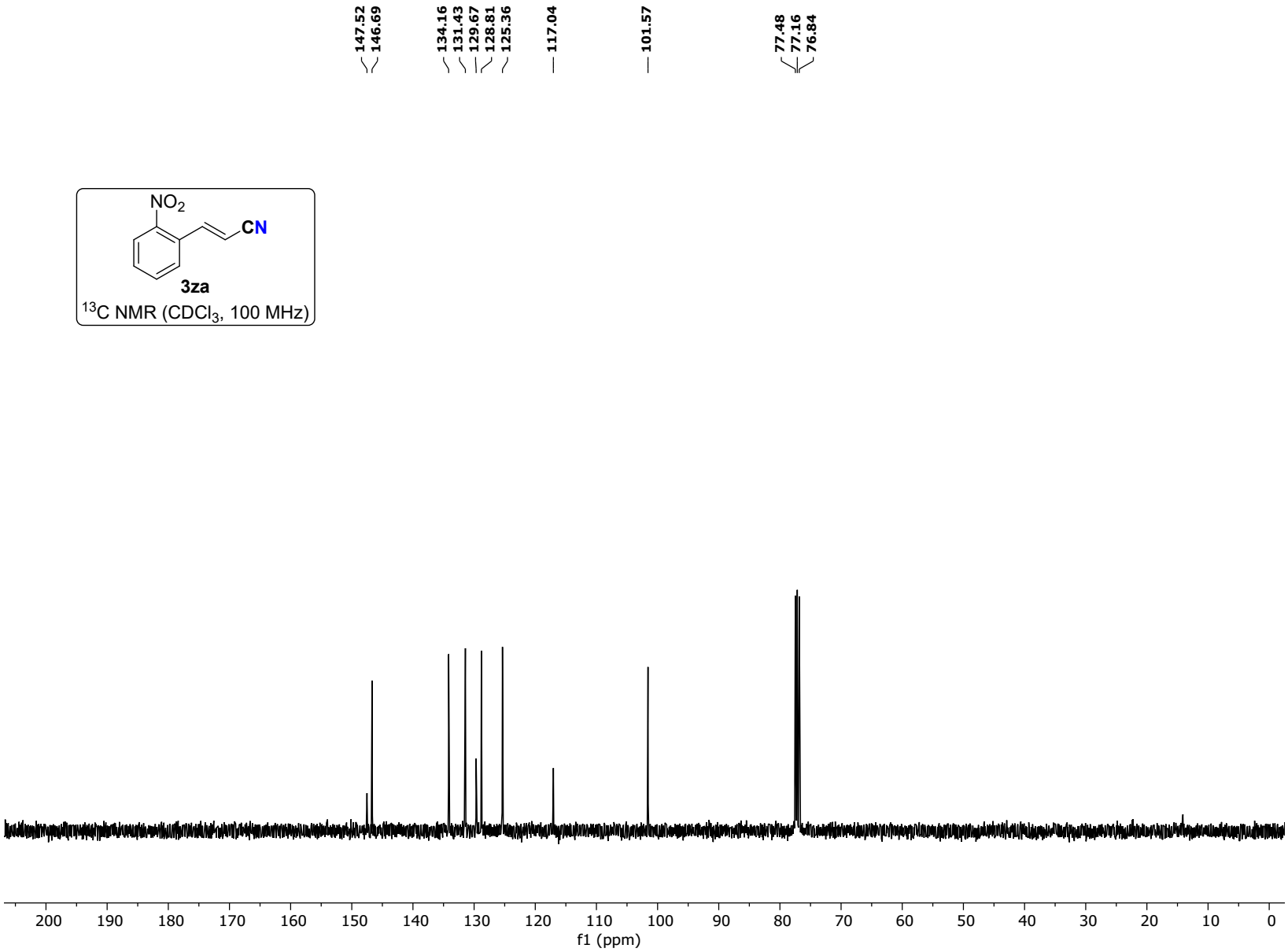

7.458
7.453
7.447
7.432
7.424
7.418
7.406
7.396
7.390
7.385
7.378
7.365
7.254
5.892
5.850
-0.000
CN
3y
^{1}H NMR ($CDCl_3$, 400 MHz)
6.20
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

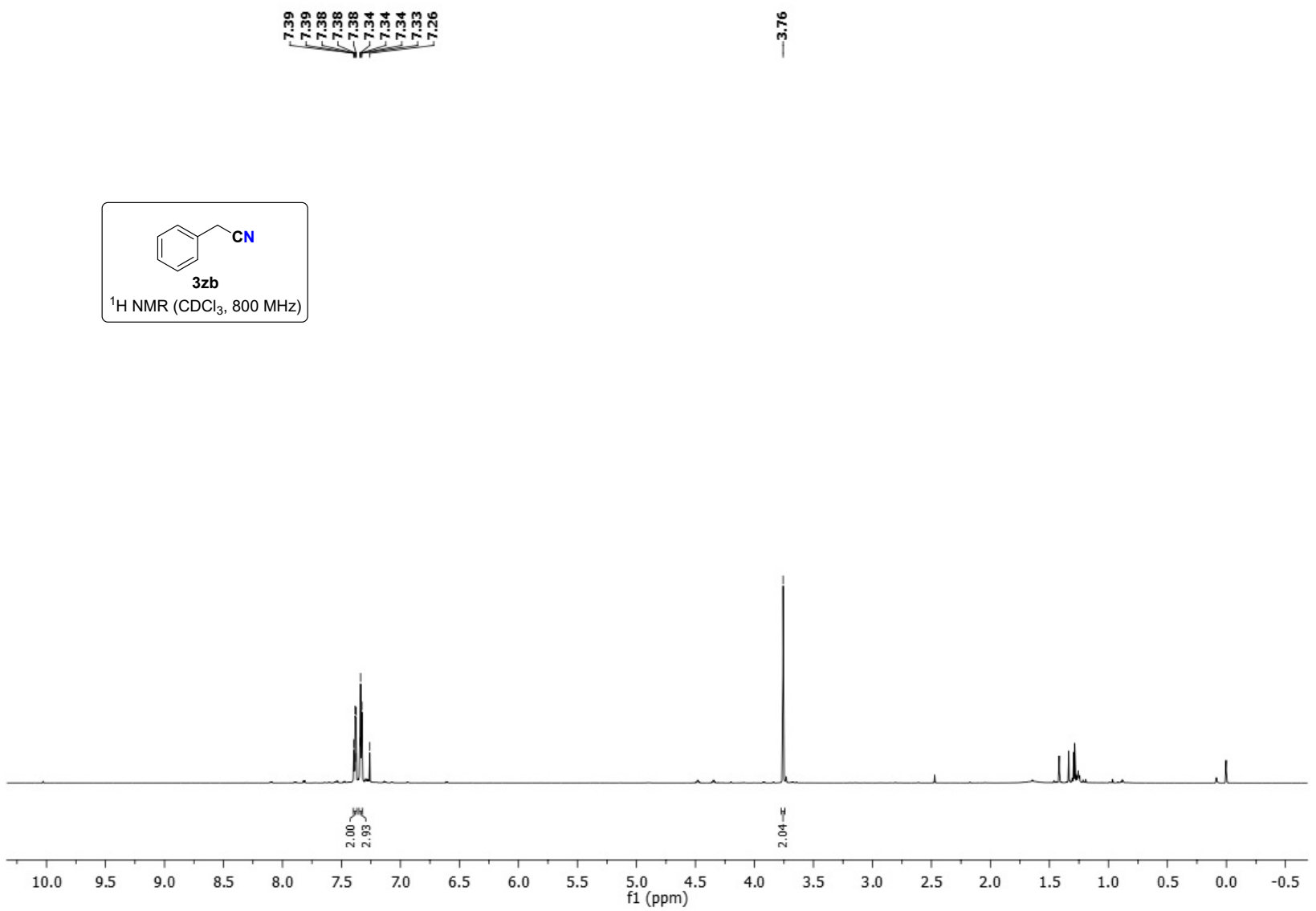

150.64
133.58
131.29
129.19
127.43
118.24
96.40
77.48
77.16
76.84
CN
3y
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

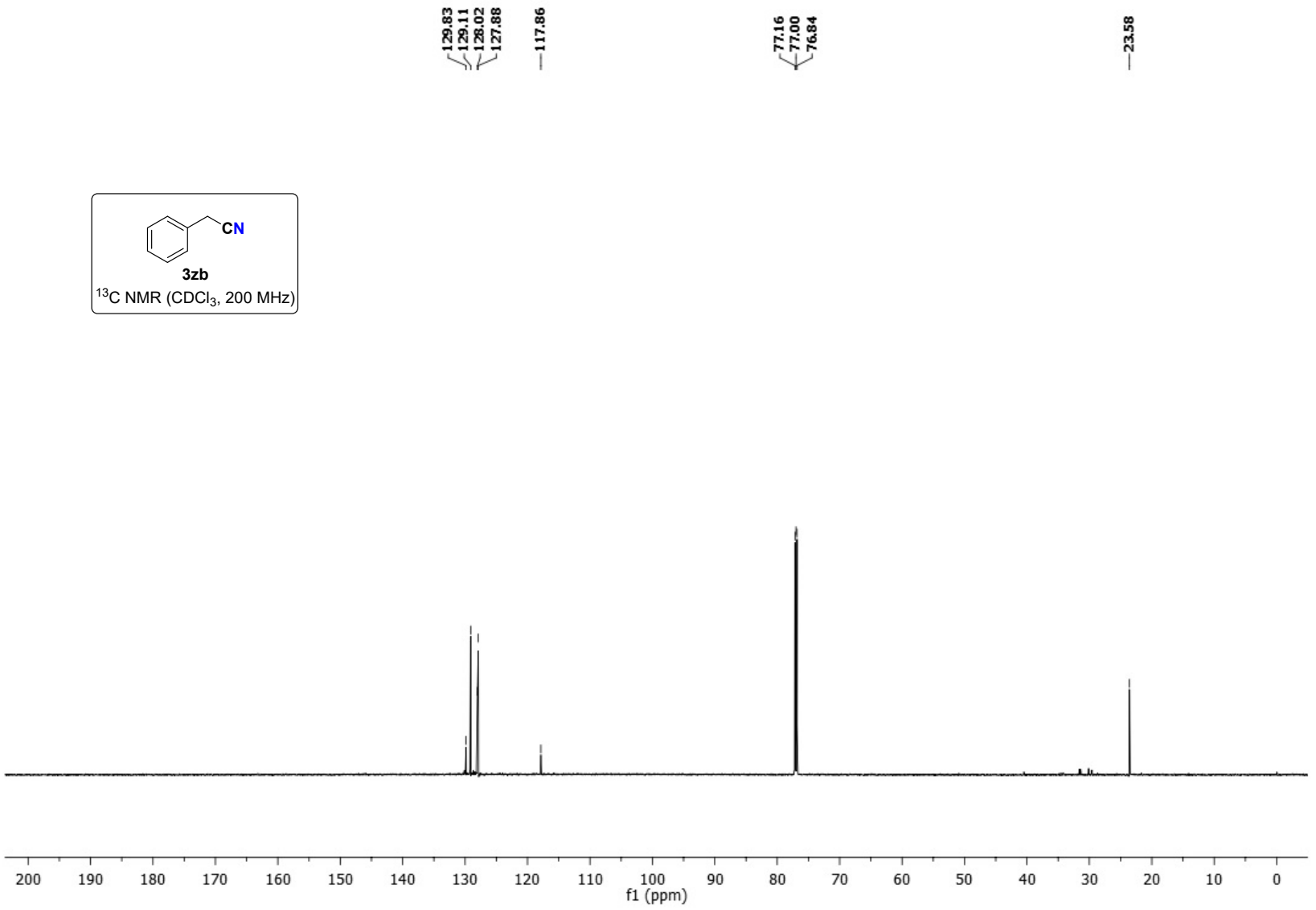

7.633
7.591
7.408
7.404
7.390
7.385
7.377
7.372
7.365
7.358
7.354
7.260
6.990
6.988
6.971
6.969
6.952
6.950
6.941
6.920
6.069
6.027
3.884
2.465
0.002
OMe
CN
3z
1H NMR (CDCl3, 400 MHz)
0.95
2.16
2.02
0.93
2.94
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

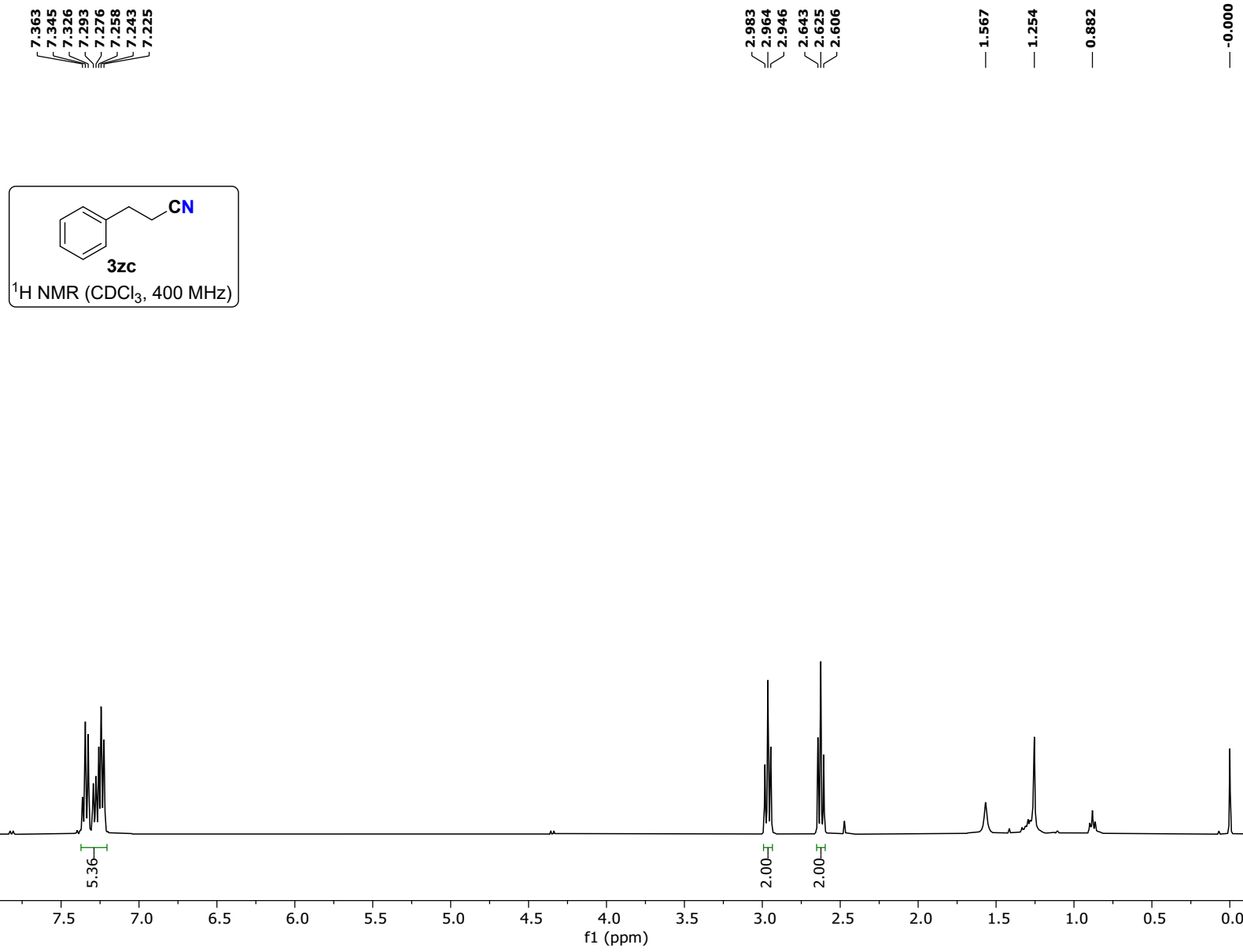

158.28
146.51
132.41
128.99
122.54
120.88
119.09
111.36
96.96
77.48
77.16
76.84
55.60
OMe
CN
3z
13C NMR (CDCl3, 100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

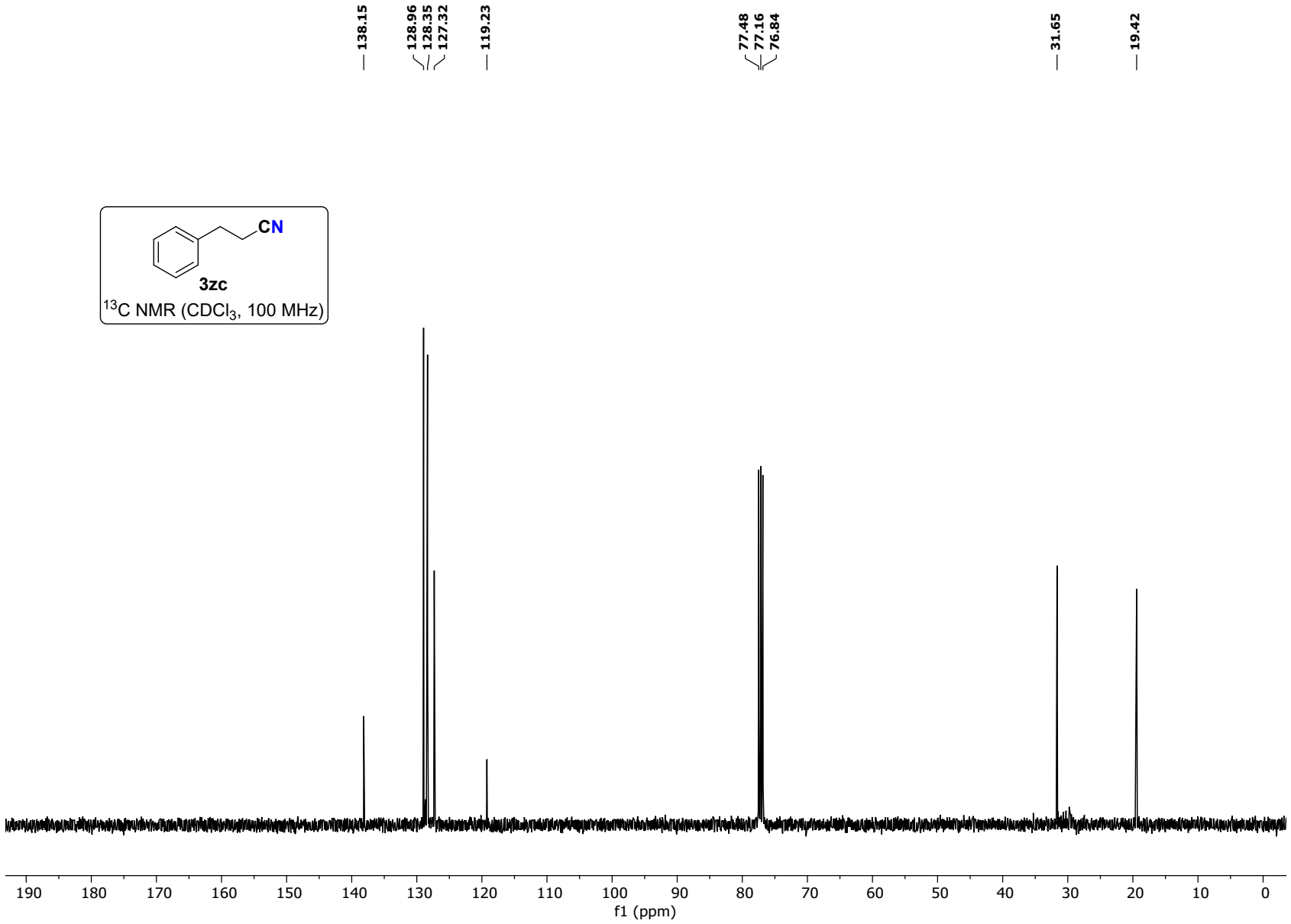

8.120
8.102
7.965
7.924
7.717
7.701
7.642
7.624
7.589
7.572
7.260
5.879
5.838
1.676
1.237
0.856
-0.022
NO2
CN
3za
1H NMR (CDCl3, 400 MHz)
1.00
1.01
3.04
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

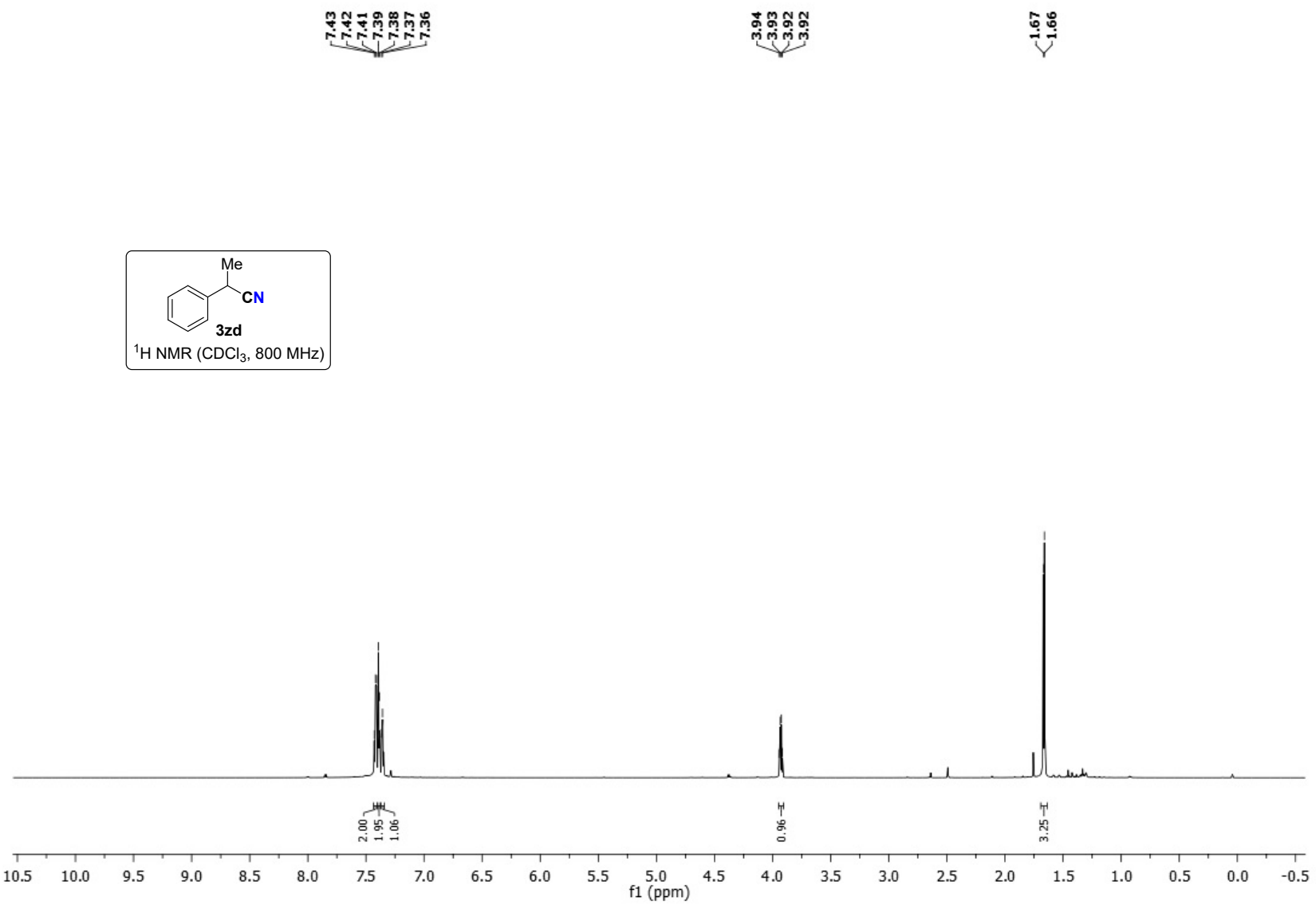

147.52
146.69
134.16
131.43
129.67
128.81
125.36
117.04
101.57
77.48
77.16
76.84
NO2
CN
3za
13C NMR (CDCl3, 100 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

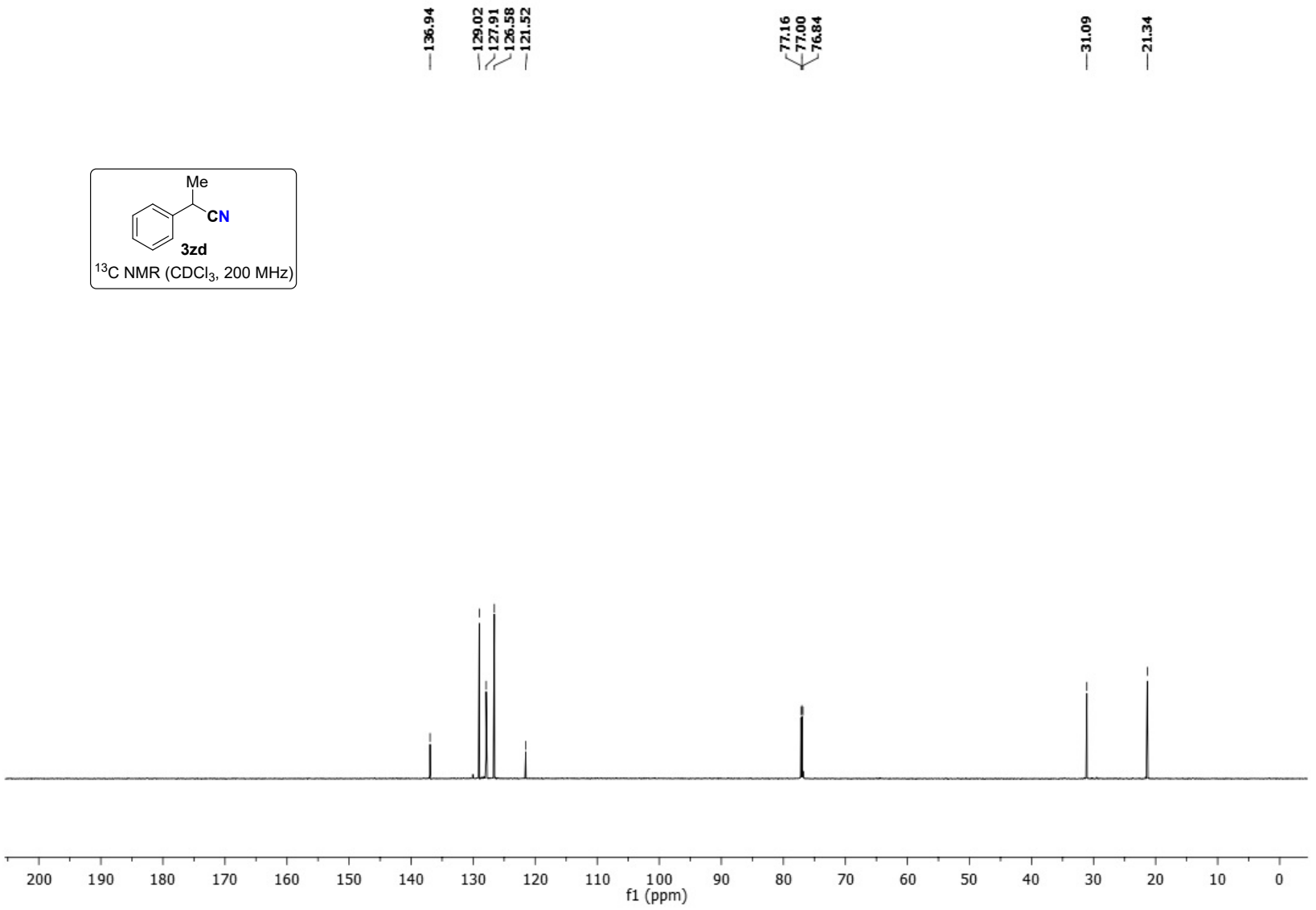

7.39
7.39
7.38
7.38
7.38
7.34
7.34
7.34
7.33
7.26
3.76
CN
3zb
^{1}H NMR ($CDCl_3$, 800 MHz)
2.00
2.93
2.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

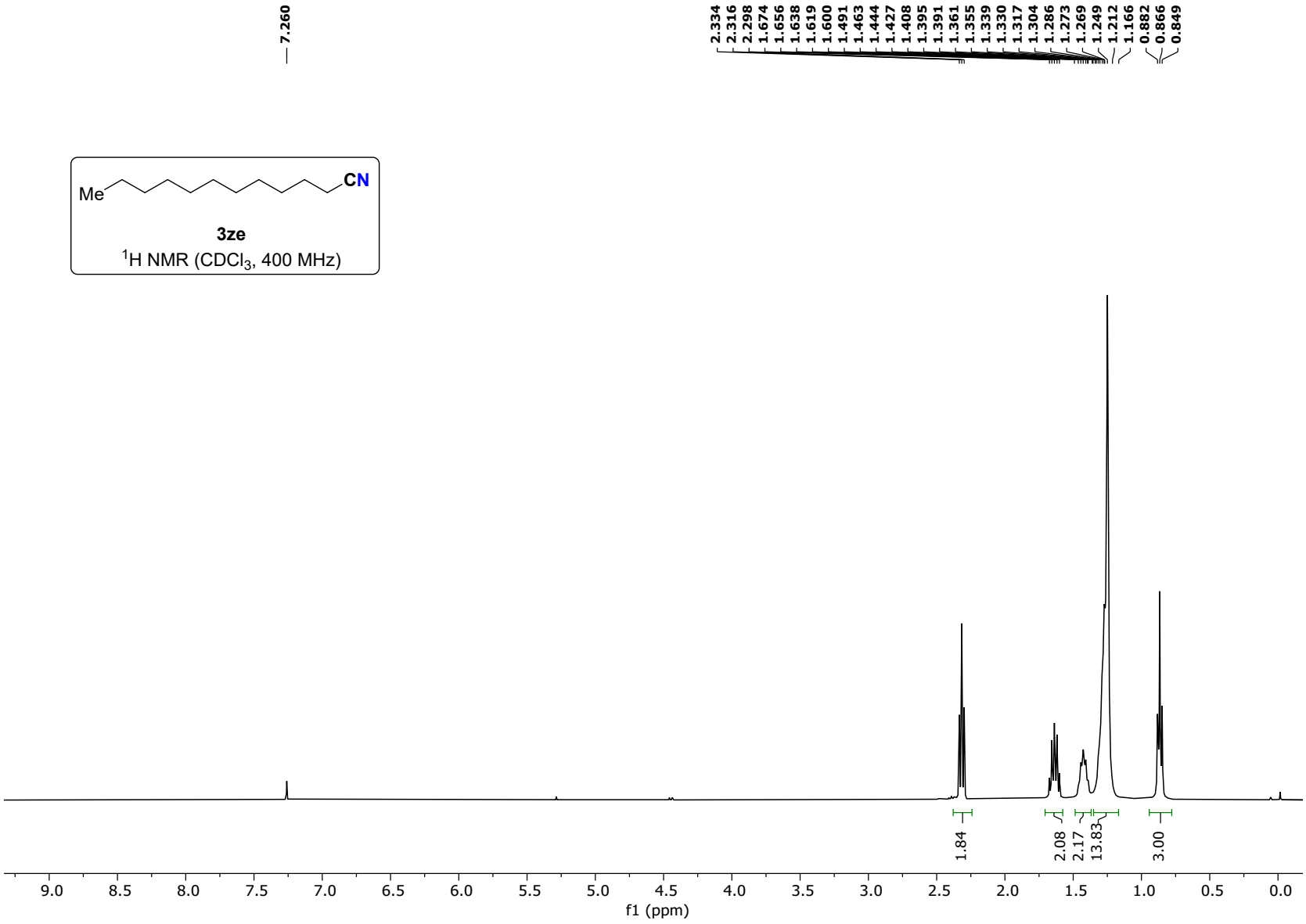

129.83
129.11
128.02
127.88
117.86
77.16
77.00
76.84
23.58
CN
3zb
13C NMR (CDCl3, 200 MHz)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

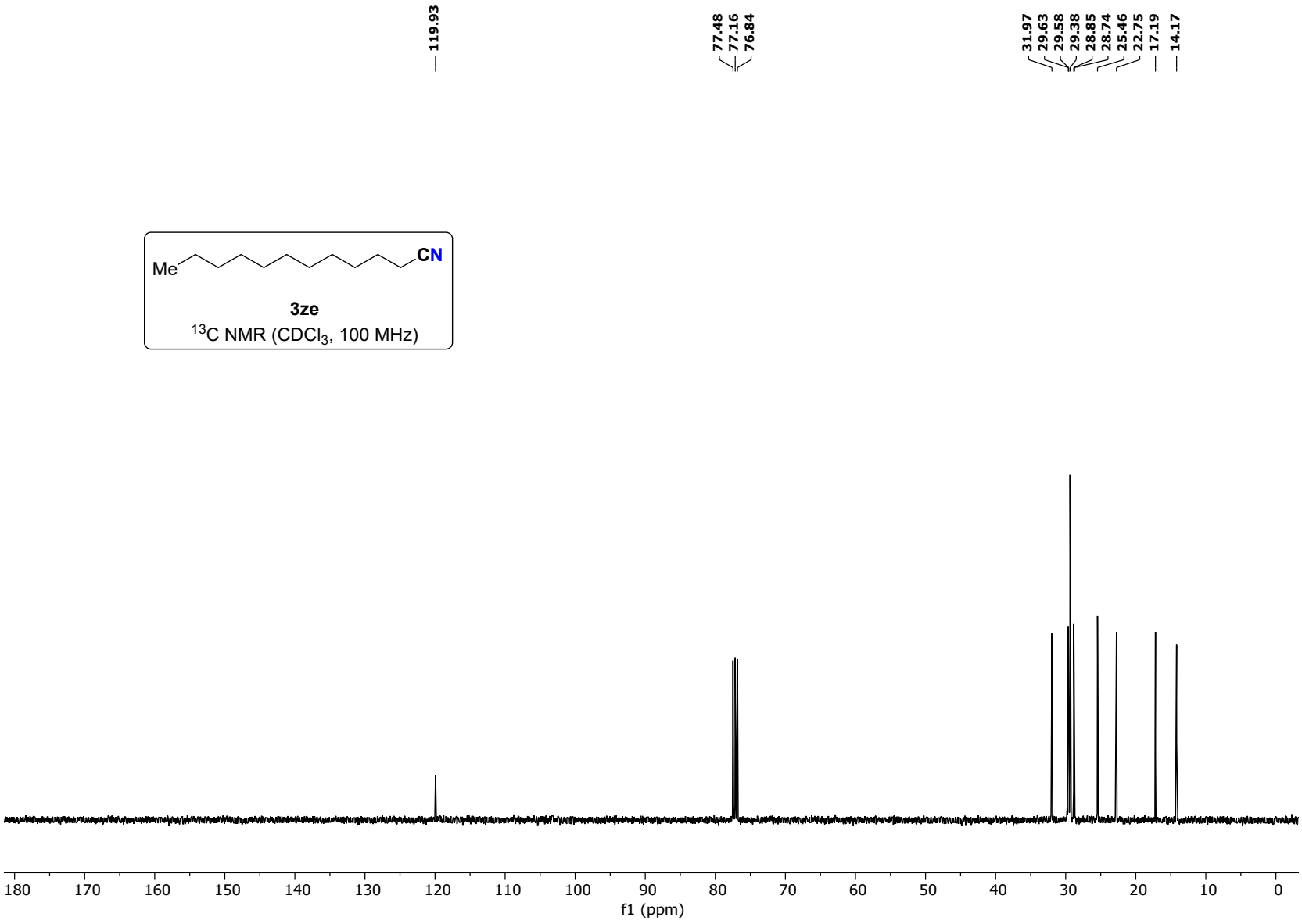

7.363
7.345
7.326
7.293
7.276
7.258
7.243
7.225
2.983
2.964
2.946
2.643
2.625
2.606
1.567
1.254
0.882
-0.000
CN
3zc
1H NMR (CDCl3, 400 MHz)
5.36
2.00
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

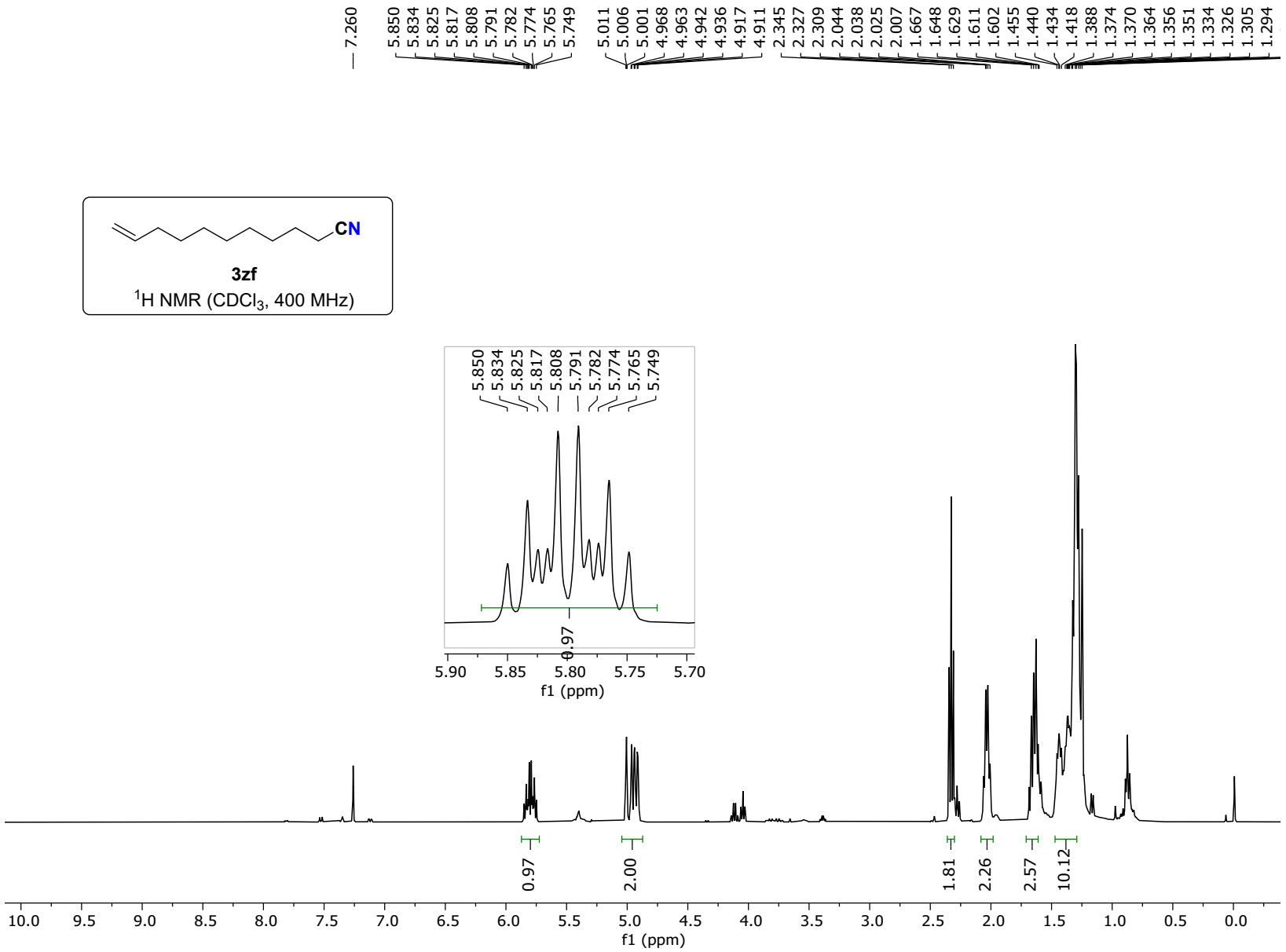

138.15
128.96
128.35
127.32
119.23
77.48
77.16
76.84
31.65
19.42
CN
3zc
13C NMR (CDCl3, 100 MHz)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

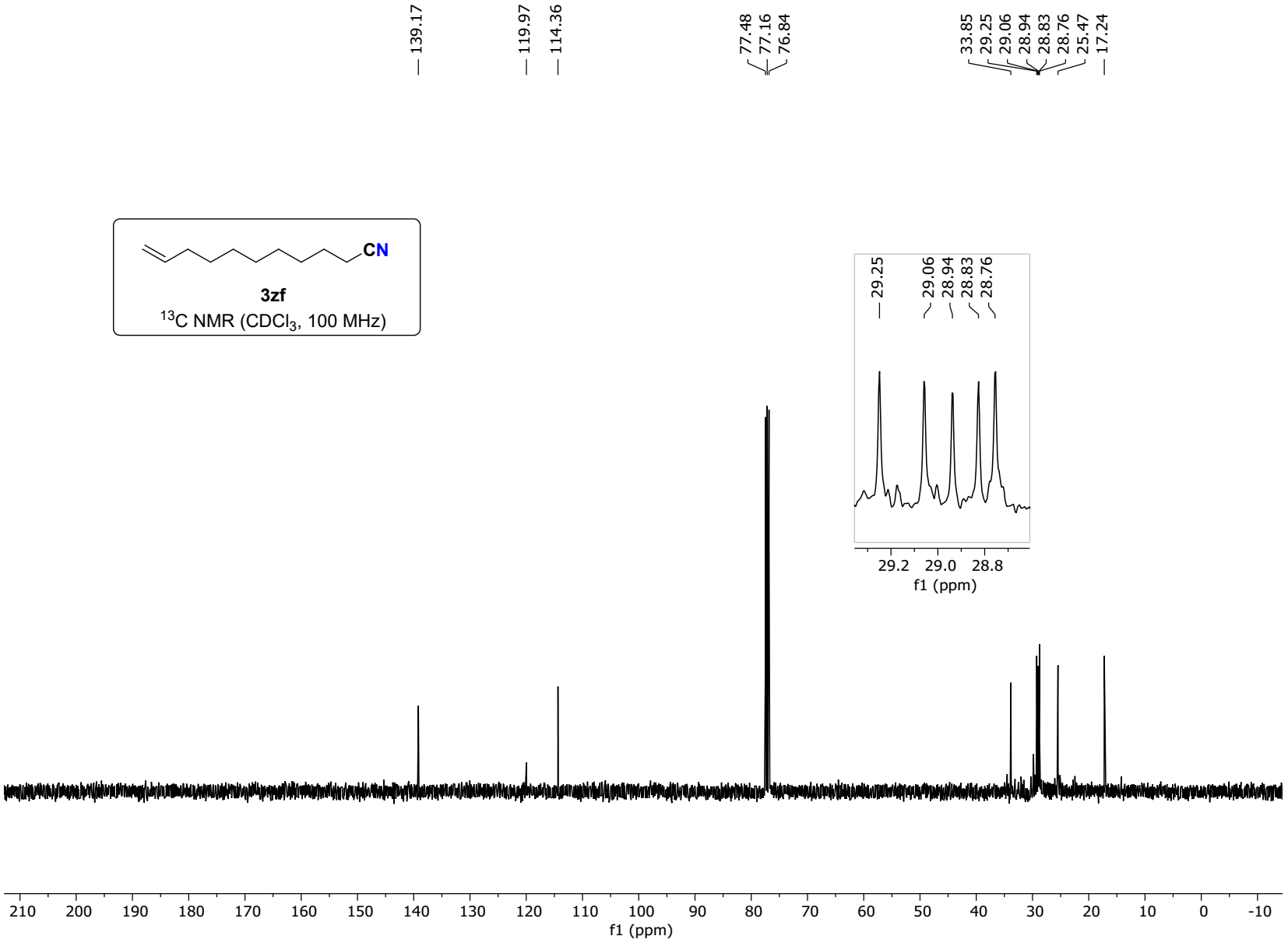

7.43
7.42
7.41
7.39
7.38
7.37
7.36
3.94
3.93
3.92
3.92
1.67
1.66
Me
CN
3zd
^{1}H NMR ($CDCl_3$, 800 MHz)
2.00
1.95
1.06
0.96
3.25
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


136.94
129.02
127.91
126.58
121.52
77.16
77.00
76.84
31.09
21.34
Me
CN
3zd
13C NMR (CDCl3, 200 MHz)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.260
2.334
2.316
2.298
1.674
1.656
1.638
1.619
1.600
1.491
1.463
1.444
1.427
1.408
1.395
1.391
1.361
1.355
1.339
1.330
1.317
1.304
1.286
1.273
1.269
1.249
1.212
1.166
0.882
0.866
0.849
Me
CN
3ze
1H NMR (CDCl3, 400 MHz)
1.84
2.08
2.17
13.83
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


119.93
77.48
77.16
76.84
31.97
29.63
29.58
29.38
28.85
28.74
25.46
22.75
17.19
14.17
Me
CN
3ze
13C NMR (CDCl3, 100 MHz)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.260
5.850
5.834
5.825
5.817
5.808
5.791
5.782
5.774
5.765
5.749
5.011
5.006
5.001
4.968
4.963
4.942
4.936
4.917
4.911
2.345
2.327
2.309
2.044
2.038
2.025
2.007
1.667
1.648
1.629
1.611
1.602
1.455
1.440
1.434
1.418
1.388
1.374
1.370
1.364
1.356
1.351
1.334
1.326
1.305
1.294
CN
3zf
1H NMR (CDCl3, 400 MHz)
0.97
5.90
5.85
5.80
5.75
5.70
f1 (ppm)
0.97
2.00
1.81
2.26
2.57
10.12
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


139.17
119.97
114.36
77.48
77.16
76.84
33.85
29.25
29.06
28.94
28.83
28.76
25.47
17.24
CN
3zf
13C NMR (CDCl3, 100 MHz)
29.25
29.06
28.94
28.83
28.76
29.2
29.0
28.8
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)