Oxidative Cleavage of Allyl Ethers by an Oxoammonium Salt

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Supporting Information

Key to Abbreviated Terms:

$CDCl_3$ – Deuterated chloroform
DCM – Dichloromethane
DMF– *N*, *N*-Dimethylformamide
Et_2O – Diethyl ether
EtOAc – Ethyl acetate
Hex – Hexanes
THF – Tetrahydrofuran
TLC – Thin Layer Chromatography
TMG - 1,1,3,3-Tetramethylguanidine

General Considerations:

General:

All chemical transformations requiring inert atmospheric conditions or vacuum distillation utilized Schlenk line techniques with a 3- or 4-port dual-bank manifold. Nitrogen was used to provide such an atmosphere. NMR Spectra (^{1}H, ^{13}C, ^{19}F) were performed at 298 K on either a Brüker DRX-400 MHz NMR, or Brüker Avance 500 MHz NMR. ^{1}H-NMR Spectra obtained in $CDCl_3$ were referenced to residual non-deuterated chloroform (7.26 ppm) in the deuterated solvent. ^{13}C-NMR Spectra obtained in $CDCl_3$ were referenced to chloroform (77.3 ppm). ^{19}F-NMR spectra were referenced to hexafluorobenzene (–164.9 ppm)[1]. Reactions were monitored by an Agilent Technologies 7820A Gas Chromatograph attached to a 5975 Mass Spectrometer, ^{1}H-NMR, and/or by TLC on silica gel plates (60Å porosity, 250 μm thickness). High-resolution mass spectra were performed on either a JEOL AccuTOF-DART SVP 100 in positive direct analysis in real time (DART) ionization method, using PEG as the internal standard. TLC analysis was performed using Hex/EtOAc as the eluent and visualized using permanganate stain, *p*-anisaldehyde stain, Seebach's Stain, and/or UV light. Flash chromatography and silica plugs utilized Dynamic Adsorbants Inc. flash silica gel (60Å porosity, 32-63 μm) or a Teledyne Isco Combiflash® Rf 200 using Redisep Rf Gold® silica gel cartridges.

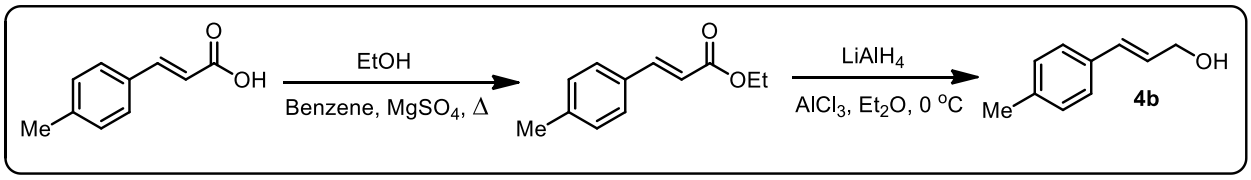
Chemicals:

Deuterated NMR solvents ($CDCl_3$) were purchased from Cambridge Isotope Laboratories. $CDCl_3$ stored over 4Å molecular sieves and K_2CO_3. Sodium sulfate, sodium hydride, aluminum trichloride, sodium carbonate, THF (reagent grade), CH_2Cl_2, EtOH, Et_2O (ACS Grade and reagent grade), and TBAF (1 M in THF) were purchased from Sigma-Aldrich. Trifluoromethyltrimethylsilane and hexafluorobenzene were purchased from Synquest Laboratories and/or Oakwood Chemicals. Substituted propionic acid derivatives were either purchased commercially or prepared in house by Knoevenagel protocol from malonic acid and their corresponding aldehydes.[2] The oxoammonium salt 4-acetamido-2,2,6,6-tetramethyl-1-oxopiperidin-1-ium tetrafluoroborate **1a** was prepared according to our recently published protocol.[3]

[1] Ravikumar, I.; Saha, S.; Ghosh, P. *Chem. Commun*. **2011**, *47*, 4721.
[2] See Hamlin, T. A.; Kelly, C. B.; Leadbeater, N. E. Eur. J. Org. Chem., **2013**, 3658 for a representative example of this protocol.
[3] Mercadante, M. A.; Kelly, C. B.; Bobbitt, J. M.; Tilley, L. J.; Leadbeater, N. E. *Nat. Protoc.* **2013**, *8*, 666.

Synthesis of Allyl Alcohols and their Precursors:

Synthesis of (*E*)-3-(p-tolyl)prop-2-en-1-ol (4b)



trans-ethyl 4-methylcinnamate[4]

To a 250 mL round bottom flask was added *trans*-4-methylcinnamic acid, (16.5 g, 0.102 mol, 1 equiv.), absolute ethanol (21.1 g, 0.459 mol, 4.5 equiv.) and benzene (50 mL, 5 M in *trans*-cinnamic acid). The flask was equipped with a stir bar, a Soxhlet with cellulose membrane insert (containing 25 g of $MgSO_4$), and a reflux condenser. The flask was heated to ≈ 40 °C in oil bath and concentrated H_2SO_4 (1.05 mL, 0.0194 mol, 0.19 equiv.) was added all at once via a syringe. The solution was heated to reflux (oil bath at 120 °C) and allowed to stir overnight. The solution was then diluted with ether (≈100 mL). The mixture was transferred to a separatory funnel and washed with 3 X 100 mL of a 10% w.t. sodium bicarbonate solution (***CAUTION***: CO_2 gas is evolved). The organic layer was then washed with deionized water (≈100 mL), brine (≈100 mL), and dried with Na_2SO_4. The solvent was removed *in vacuo* by rotary evaporation to afford the ethyl 4-methylcinnamate as a clear, pale yellow oil (14.87 g, 77%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.33 (t, *J*=7.20 Hz, 3 H) 2.36 (s, 3 H) 4.26 (q, *J*=7.12 Hz, 2 H) 6.39 (d, *J*=15.91 Hz, 1 H) 7.18 (d, *J*=7.98 Hz, 2 H) 7.41 (d, *J*=8.13 Hz, 2 H) 7.66 (d, *J*=16.06 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 14.54 (CH_3) 21.62 (CH_3) 60.56 (CH_2) 117.39 (CH) 128.23 (CH) 129.80 (CH) 131.95 (C) 140.78 (C) 144.76 (CH) 167.34 (C)
GC-MS (EI) 190 ($[M]^+$, 57%) 162 (21%) 145 (90%) 131 (16%) 118 (52%) 115 (100%) 102 (11%) 91 (47%) 65 (23%) 63 (12%) 39 (10%)

(*E*)-3-(p-tolyl)prop-2-en-1-ol[5] (4b)

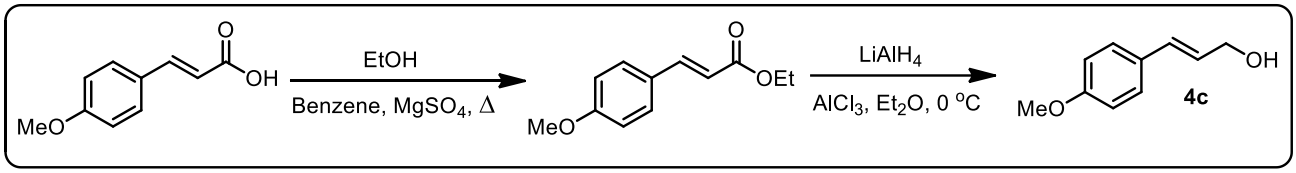
To a flamed-dried 1000 mL flask equipped with a large stir bar was added anhydrous Et_2O (392 mL) and $LiAlH_4$ (1.49 g, 0.0392 mol, 1.12 equiv). The flask was sealed with a rubber septum and placed under an N_2 atmosphere using a N_2 inlet needle. The mixture was cooled 0 °C in a large ice bath for 10 minutes and after this time anhydrous $AlCl_3$ (1.76 g, 0.0132 mol, 0.377 equiv) was added rapidly to the flask. The solution was allowed to stir for five minutes and gradually transitioned from grey to white. At this time, ethyl 4-methylcinnamate (6. 66 g, 0.035 mol, 1 equiv) dissolved in 21 mL of anhydrous Et_2O was added to the flask over five minutes. After complete addition, the solution was allowed to stir at 0 °C for 0.5 h.[6] After this time, the septum was removed and the solution was *carefully* quenched with 1 M HCl (200 mL). **CAUTION!** *Large excess of hydrogen gas evolved*. The biphasic solution was allowed to stir for 10 minutes, and gradually

[4] Wadhwa, K.; Verkade, J. G. *J. Org. Chem.*, **2009**, *74*, 4368.
[5] Miyamura, H.; Choo, G. C. Y.; Yasukawa, T.; Yoo, W.-J.; Kobayashi, S. *Chem. Commun.*, **2013** , *49*, 9917.
[6] It is imperative that the reaction mixture be quenched 30 minutes after addition of the ester. Longer reaction times lead to unwanted over reduction of the double bond.

became clear. The quenched reaction mixture was transferred to a separatory funnel and the phases were separated. The aqueous layer was extracted with Et_2O (2 X 75 mL) and the combined organic layers were washed with saturated aqueous $NaHCO_3$ (2 X 125 mL), deionized water (125 mL), and brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the pure alcohol **4b** (4.54 g, 88%) as a powdery white solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 2.34 (s, 3 H) 4.31 (t, *J*=4.89 Hz, 2 H) 6.32 (dt, *J*=15.87, 5.86 Hz, 1 H) 6.59 (d, *J*=15.86 Hz, 1 H) 7.13 (d, *J*=7.98 Hz, 2 H) 7.29 (d, *J*=8.13 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 21.47 (CH_3) 64.15 (CH_2) 126.67 (CH) 127.72 (CH) 129.58 (CH) 131.51 (CH) 134.15 (C) 137.85 (C)
GC-MS (EI) 148 ([M]$^+$, 54%) 133 (27%) 119 (21%) 115 (66%) 105 (100%) 91 (88%) 77 (34%) 65 (28%) 63 (24%) 55(29%) 51 (22%) 39 (24%)

Synthesis of (*E*)-3-(4-methoxyphenyl)prop-2-en-1-ol (4c)



***trans*-ethyl 4-methoxycinnamate**[7]

Synthesis of ethyl 4-methoxycinnamate (12.43 g, 60%) was accomplished using the procedure for the preparation of ethyl 4-methylcinnamate, with the following modification: The reaction was conducted using *trans*-4-methoxycinnamic acid (17.82 g, 0.100 mol). The ester was obtained as a tan solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.32 (t, *J*=7.13 Hz, 3 H) 3.82 (s, 3 H) 4.24 (q, *J*=7.12 Hz, 2 H) 6.30 (d, *J*=15.96 Hz, 1 H) 6.87 - 6.91 (m, 2 H) 7.35 (s, 1 H) 7.44 - 7.48 (m, 2 H) 7.63 (d, *J*=15.96 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) 14.59 (CH_3) 55.58 (CH_3) 60.53 (CH_2) 114.55 (CH) 116.01 (CH) 127.45 (CH) 129.91 (C) 144.45 (CH) 161.57 (C) 167.54 (C) δ ppm
GC-MS (EI) 206 ([M]$^+$, 65%) 186 (8%) 161 (100%) 158 (23%) 134 (22%) 129 (66%) 104 (13%) 80 (11%) 68 (31%)

(*E*)-3-(4-methoxyphenyl)prop-2-en-1-ol[8] **(4c)**

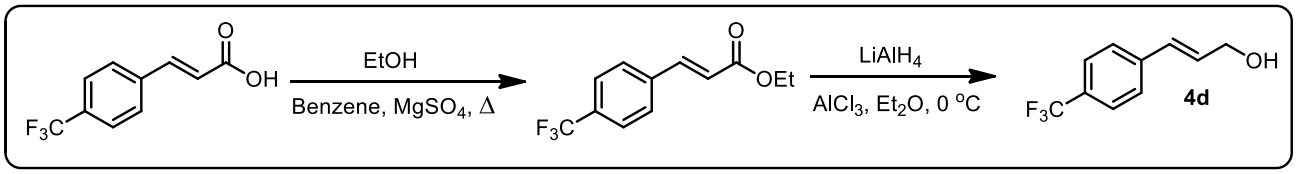
Synthesis of (*E*)-3-(4-methoxyphenyl)prop-2-en-1-ol (3.49 g, 61%) was accomplished using the procedure for the preparation of (*E*)-3-(p-tolyl)prop-2-en-1-ol, **4b**, with the following modification: The reaction was conducted using *trans*-4-methoxycinnamate (7.21 g, 0.035 mol). The alcohol was obtained as a powdery, pale yellow solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 3.81 (s, 3 H) 4.30 (t, *J*=5.81 Hz, 2 H) 6.24 (dt, *J*=15.81, 5.94 Hz, 1 H) 6.56 (d, *J*=15.91 Hz, 1 H) 6.86 (d, *J*=8.66 Hz, 2 H) 7.33 (d, *J*=8.66 Hz, 2 H)

[7] West, T. H.; Daniels, D. S. B.; Slawin, A. M. Z.; Smith, A. D. *J. Am. Chem. Soc.*, **2014**, *136*, 4476.
[8] Zimmer, L. E.; Charette, A. B. *J. Am. Chem. Soc.*, **2009**, *131*, 15624.

^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 55.56 (CH_3) 64.19 (CH_2) 114.29 (CH) 126.54 (CH) 127.94 (CH) 129.70 (CH) 131.23 (C) 159.59 (C)
GC-MS (EI) 164 ([M]$^+$, 24%) 131 (7%) 121 (100%) 108 (45%) 103 (17%) 91 (30%) 77 (29%) 65 (14%) 63 (15%) 55(12%) 51 (11%) 39 (9%)

Synthesis of (*E*)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-ol (4d)



***trans*-ethyl 4-(trifluoromethyl)cinnamate**[7]

Synthesis of ethyl 4-(trifluoromethyl)cinnamate (5.23 g, 86%) was accomplished using the procedure for the preparation of ethyl 4-methylcinnamate, with the following modification: The reaction was conducted using *trans*-4-(trifluoromethyl)cinnamic acid (5.40 g, 0.025 mol). The ester was obtained as a powdery, white solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.34 (t, *J*=7.13 Hz, 3 H) 4.28 (q, *J*=7.15 Hz, 2 H) 6.50 (d, *J*=16.06 Hz, 1 H) 7.35 (s, 1 H) 7.59 - 7.66 (m, 4 H) 7.69 (d, *J*=16.01 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 14.53 (CH_3) 61.06 (CH_2) 124.10 (q, $J_{C\text{-}F}$ =272.00 Hz, CF_3) 121.15 (CH) 126.12 (q, $J_{C\text{-}C\text{-}C\text{-}F}$ =3.70 Hz, CH) 128.42 (CH) 131.99 (q, $J_{C\text{-}C\text{-}F}$ =32.80 Hz, C) 138.14 (C) 142.95 (CH) 166.66 (C)
^{19}F NMR ($CDCl_3$, 377 MHz) δ ppm -66.00 (s)
GC-MS (EI) 244 ([M]$^+$, 11%) 216 (17%) 199 (83%) 171 (39%) 151 (100%) 145 (10%) 135 (13%) 131 (12%) 102 (34%) 75 (22%) 69 (10%) 51 (11%) 43 (9%)

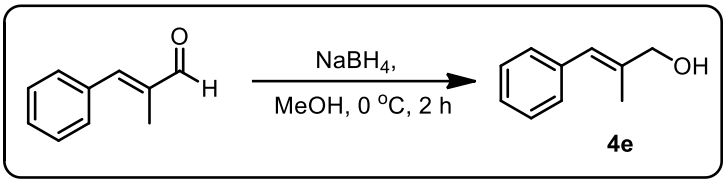
(*E*)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-ol[9] **(4d)**

Synthesis of (*E*)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-ol (2.75 g, 68%) was accomplished using the procedure for the preparation of (*E*)-3-(p-tolyl)prop-2-en-1-ol, **4b**, with the following modification: The reaction was conducted using *trans*-ethyl 4-(trifluoromethyl)cinnamate (4.89 g, 0.020 mol). The alcohol was obtained as a pale yellow solid

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 2.39 (s, 1 H) 4.35 (dd, *J*=5.35, 1.27 Hz, 2 H) 6.43 (dt, *J*=16.01, 5.40 Hz, 1 H) 6.64 (d, *J*=15.96 Hz, 1 H) 7.44 (d, *J*=8.17 Hz, 2 H) 7.55 (d, *J*=8.22 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 63.49 (CH_2) 124.44 (q, $J_{C\text{-}F}$ = 271.50 Hz, CF_3) 125.80 (q, $J_{C\text{-}C\text{-}C\text{-}F}$ = 3.85 Hz, CH) 126.82 (CH) 129.69 (q, $J_{C\text{-}C\text{-}F}$ = 32.80 Hz, C) 129.57 (CH) 131.47 (CH) 140.46 (C)
^{19}F NMR ($CDCl_3$, 377 MHz) δ ppm -65.67 (s)
GC-MS (EI) 202 ([M]$^+$, 31%) 183 (31%) 173 (11%) 160 (83%) 151 (45%) 146 (23%) 133 (100%) 127 (36%) 115 (59%) 109 (15%) 105 (28%) 91 (51%) 77 (23%) 75 (28%) 69 (29%) 63 (19%) 55 (37%) 51 (21%) 39 (13%)

[9] Vyas, D. J.; Oestreich, M. Chem. Commun., **2010**, *46*, 568.

Synthesis of (*E*)-2-methyl-3-phenylprop-2-en-1-ol (4e)

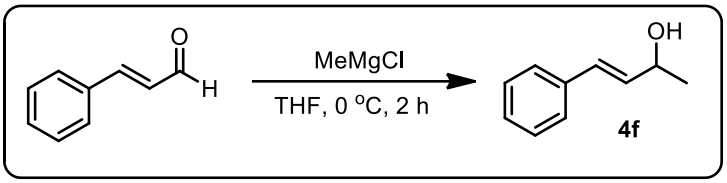


(*E*)-2-methyl-3-phenylprop-2-en-1-ol[10] (4e)

To a one-neck 250 mL round bottom flask equipped with stirbar was added methanol (100 mL, 0.5 M in the aldehyde) and α-methylcinnamaldehyde (7.31 g, 0.050 mol, 1 equiv). The mixture was placed in an ice bath and allowed to cool while stirring for 10 minutes. At this time, sodium borohydride (3.78 g, 0.100 mol, 2 equiv) was added very slowly portion wise. The reaction mixture was stirred at 0 °C for 2 hours. Once complete, the contents of the reaction flask were transferred to a separatory funnel and diluted with of deionized water (≈ 200 mL) and ether (≈ 300 mL). The aqueous layer was extracted with ether (3 X 100 mL). The organic layers were combined and washed with deionized water (≈ 200 mL) and brine (≈ 200 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation affording the crude alcohol. Further purification was accomplished by vacuum distillation (72-74 @ 0.1 mmHg) to give the pure alcohol **4e** (5.77 g, 77%) as a clear, colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.68 (br. s., 1 H) 1.91 (s, 3 H) 4.20 (s, 2 H) 6.54 (s, 1 H) 7.20 - 7.25 (m, 1 H) 7.27 - 7.37 (m, 4 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 15.55 (CH_3) 69.27 (CH_2) 125.32 (CH) 126.71 (CH) 128.41 (CH) 129.14 (CH) 137.83 (C) 137.92 (C)
GC-MS (EI) 148 ($[M]^+$, 43%) 133 (21%) 129 (19%) 115 (72%) 105 (39%) 91 (100%) 78 (30%) 65 (14%) 63 (15%) 55 (13%) 51 (17%) 39 (14%)

Synthesis of (*E*)-4-phenylbut-3-en-2-ol (4f)



(*E*)-4-phenylbut-3-en-2-ol[11] (4f)

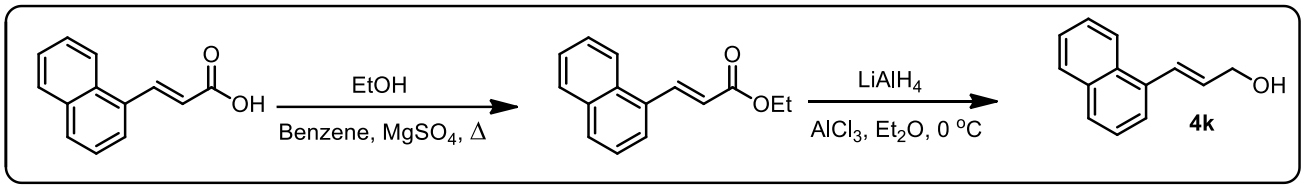
To a flamed dried 250 mL round bottom flask equipped with a stir bar, rubber septum, and nitrogen inlet needle was added anhydrous THF (83 mL) and *trans*-cinnamaldehyde (4.17 g, 0.030 mol). The solution was placed under nitrogen and cooled to 0 °C in an ice-water bath. After cooling for ten minutes, a 3 M solution of methylmagnesium chloride in THF (12.5 mL, 0.0375 mol, 1.25 equiv) was added to the flask dropwise *via* a syringe. After complete addition, the solution was allowed to stir at 0 °C for 2 h. After this time, the flask was equipped with a vent needle and the reaction mixture was quenched with an aqueous 1 M HCl solution (75 mL). **CAUTION**!

[10] Bausch, C. C.; Patman, R. L.; Breit, B.; Krische, M. J. Angew. Chem. Int. Ed., 2011, 50, 5687.
[11] Langlois, J.-B.; Alexakis, A. *Angew. Chem. Int. Ed.*, **2011**, *50*, 1877.

Exothermic, Evolves Methane Gas. The quenched reaction mixture was transferred to a separatory funnel and diluted with Et_2O (≈ 150 mL). The layers were separated and the aqueous layer was extracted with Et_2O (3 × 50 mL). The combined organic layers were washed with saturated aqueous bicarbonate (≈ 100 mL), deionized water (≈ 150 mL), and brine (≈ 100 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude alcohol. Further purification was accomplished by vacuum distillation (85-87 ºC @ 0.1 mmHg) to give the pure alcohol **4f** (3.76 g, 85%) as a clear, colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.38 (d, *J*=6.37 Hz, 3 H) 1.61 (br. s., 1 H) 4.50 (quin, *J*=6.28 Hz, 1 H) 6.27 (dd, *J*=15.91, 6.37 Hz, 1 H) 6.57 (d, *J*=15.57 Hz, 1 H) 7.21 - 7.25 (m, 1 H) 7.29 - 7.35 (m, 2 H) 7.36 - 7.41 (m, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) 23.71 (CH_3) 69.23 (CH) 126.74 (CH) 127.92 (CH) 128.86 (CH) 129.69 (CH) 133.85 (CH) 136.98 (C) δ ppm
GC-MS (EI) 148 ([M]$^+$, 42%) 133 (29%) 131 (12%) 115 (43%) 105 (100%) 91 (66%) 77 (57%) 65 (16%) 63 (22%) 55 (37%) 51 (40%) 43 (77%) 39 (22%)

Synthesis of (*E*)-3-(naphthalen-1-yl)prop-2-en-1-ol (4k)



(*E*)-ethyl 3-(naphthalen-1-yl)acrylate[12]

Synthesis of (*E*)-ethyl 3-(naphthalen-1-yl)acrylate (15.17 g, 78%) was accomplished using the procedure for the preparation of ethyl 4-methylcinnamate, with the following modification: The reaction was conducted using (*E*)-3-(naphthalen-1-yl)acrylic acid (16.97 g, 0.0856 mol). The ester was obtained as a light brown solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.39 (t, *J*=7.10 Hz, 3 H) 4.34 (q, *J*=7.20 Hz, 2 H) 6.54 (d, *J*=15.76 Hz, 1 H) 7.48 (t, *J*=7.71 Hz, 1 H) 7.51 - 7.61 (m, 2 H) 7.75 (d, *J*=7.20 Hz, 1 H) 7.86 - 7.91 (m, 2 H) 8.21 (d, *J*=8.47 Hz, 1 H) 8.54 (d, *J*=15.81 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 14.63 (CH_3) 60.84 (CH_2) 121.20 (CH) 123.66 (CH) 125.24 (CH) 125.70 (CH) 126.45 (CH) 127.08 (CH) 128.96 (CH) 130.69 (CH) 131.66 (C) 132.09 (C) 133.92 (C) 141.85 (CH) 167.13 (C)
GC-MS (EI) 226 ([M]$^+$, 12%) 181 (14%) 153 (100%) 126 (9%) 76 (11%)

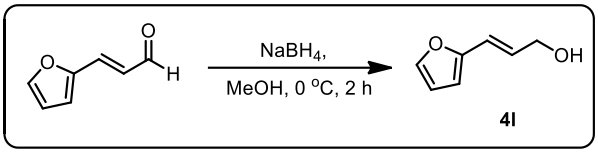
(E)-3-(naphthalen-1-yl)prop-2-en-1-ol[13] (4k)

Synthesis of (*E*)-3-(naphthalen-1-yl)prop-2-en-1-ol (5.95 g, 92%) was accomplished using the procedure for the preparation of (*E*)-2-methyl-3-phenylprop-2-en-1-ol, **4e**, with the following modification: The reaction was conducted using (*E*)-ethyl 3-(naphthalen-1-yl)acrylate (7.92 g, 0.035 mol). The alcohol was obtained as a yellow solid.

[12]Blakemore, P. R.; Ho, D. K. H.; Nap, W. M. *Org. Biomol. Chem.*, **2005**, *3*, 1365.
[13] Bouziane, A.; Helou, M.; Carboni, B.; Carreaux, F.; Demerseman, B.; Bruneau, C.; Renaud, J.-L. *Chem. Eur. J.*, **2008**, *14*, 5630.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 2.12 (br. s., 1 H) 4.43 (dd, *J*=5.55, 1.27 Hz, 2 H) 6.40 (dt, *J*=15.62, 5.60 Hz, 1 H) 7.33 - 7.56 (m, 4 H) 7.60 (d, *J*=7.10 Hz, 1 H) 7.80 (d, *J*=8.13 Hz, 1 H) 7.87 (*apparent doublet*, *J*=7.98 Hz, 1 H) 8.14 (*apparent doublet*, *J*=7.70 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 64.06 (CH_2) 123.97 (CH) 124.12 (CH) 125.84 (CH) 126.01 (CH) 126.28 (CH) 128.23 (CH) 128.30 (CH) 128.76 (CH) 131.39 (CH) 132.02 (C) 133.83 (C) 134.68 (C)
GC-MS (EI) 184 ([M]$^+$, 45%) 165 (100%) 153 (83%) 141 (96%) 128 (82%) 115 (32%) 77 (13%) 63 (18%) 51 (11%) 39 (9%)

Synthesis of (*E*)-3-(furan-2-yl)prop-2-en-1-ol (4l)

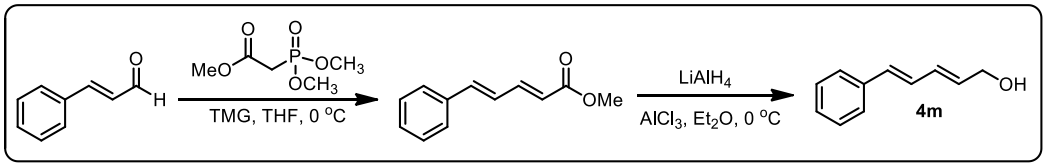


(*E*)-3-(furan-2-yl)prop-2-en-1-ol[14] (4l)

Synthesis of (*E*)-3-(furan-2-yl)prop-2-en-1-ol (4.11 g, 81%) was accomplished using the procedure for the preparation of (*E*)-2-methyl-3-phenylprop-2-en-1-ol, **4e**, with the following modification: (a) The reaction was conducted using (*E*)-3-(furan-2-yl)acrylaldehyde (5.00 g, 0.041 mol). (b) Dichloromethane was used as the extraction solvent. The alcohol **4l** was obtained as a clear, light orange oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 4.30 (d, *J*=4.82 Hz, 2 H) 6.24 (d, *J*=3.26 Hz, 1 H) 6.30 (dt, *J*=15.81, 5.50 Hz, 1 H) 6.37 (dd, *J*=3.26, 1.85 Hz, 1 H) 6.45 (s, 1 H) 7.35 (d, *J*=1.12 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 63.57 (CH_2) 108.28 (CH) 111.55 (CH) 119.59 (CH) 127.48 (CH) 142.32 (CH) 152.66 (C)
GC-MS (EI) 124 ([M]$^+$, 52%) 95 (29%) 81 (73%) 77 (45%) 68 (100%) 65 (30%) 51 (26%) 41 (23%) 39 (53%)

Synthesis of (2E,4E)-5-phenylpenta-2,4-dien-1-ol (4m)



(2*E*,4*E*)-methyl 5-phenylpenta-2,4-dienoate[15]

The following procedure is a modification of the protocol outlined by Tius.[16] To a 100 mL round bottom flask equipped with a stir bar was added THF (16 mL) followed by dimethyl methoxycarbonylmethanephosphonate (8.01 g, 0.044 mol, 1.1 equiv). The flask was allowed to cool to 0 °C in an ice water bath for five minutes. After this time, TMG (5.07 g, 0.044 mol, 1.1

[14] Charette, A. B.; Molinaro, C.; Brochu, C. *J. Am. Chem. Soc.*, **2001**, *123*, 12168.
[15] Huang, Y.; Fananas-Mastral, M.; Minnaard, A. J.; Feringa, B. L. *Chem. Commun.*, **2013**, *49*, 3309.
[16] Barrow, R. A.; Hemscheidt, T.; Liang, J.; Paik, S.; Moore, R. E.; Tius, M. A. *J. Am. Chem. Soc.* **1995**, *117*, 2479.

equiv) was added to the flask dropwise over five minutes. The mixture was allowed to stir at 0 °C for 30 minutes. After this time, the *trans*-cinnamaldehyde (5.29 g, 0.040 mol, 1 equiv) dissolved in 2.6 mL of THF was added dropwise rapidly, turning the solution bright yellow. After five minutes the ice bath was removed and the solution was allowed to stir overnight at room temperature. After this time, the solution was quenched with 25 mL of deionized water. The solution was transferred to a separatory funnel and diluted with deionized water (100 mL) and Et_2O (100 mL). The phases were separated and the aqueous layer was extracted with Et_2O (3 X 50 mL). The combined organic layers were washed with aqueous 1 M HCl (2 X 100 mL), deionized water (100 mL), and brine (150 mL). The organic layer was dried with sodium sulfate and the solvent was removed *in vacuo* by rotary evaporation. The pure ester (7.40 g, 98%) was obtained as a powdery, white solid.

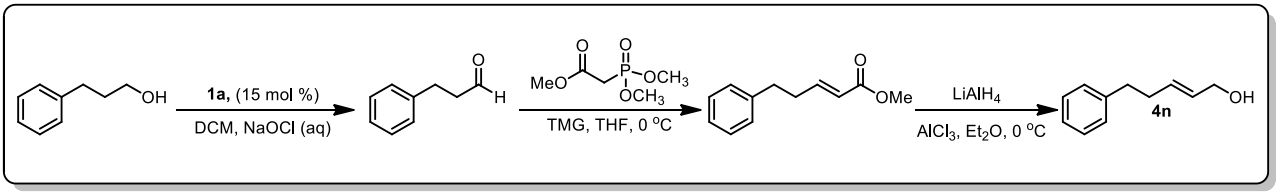
^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 3.77 (s, 3 H) 6.00 (d, *J*=15.28 Hz, 1 H) 6.83 - 6.94 (m, 2 H) 7.28 - 7.38 (m, 3 H) 7.42 - 7.50 (m, 3 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 51.80 (CH_3) 121.08 (CH) 126.44 (CH) 127.45 (CH) 129.06 (CH) 129.31 (CH) 136.25 (C) 140.79 (CH) 145.06 (CH) 167.70 (C)
GC-MS (EI) 188 ([M]$^+$, 16%) 157 (17%) 129 (100%) 102 (13%) 51 (6%)

(2E,4E)-5-phenylpenta-2,4-dien-1-ol[7] (4m)

Synthesis of (2*E*,4*E*)-5-phenylpenta-2,4-dien-1-ol (3.85 g, 80%) was accomplished using the procedure for the preparation of (*E*)-3-(p-tolyl)prop-2-en-1-ol, **4b**, with the following modification: The reaction was conducted using (2*E*,4*E*)-methyl 5-phenylpenta-2,4-dienoate (5.65 g, 0.030 mol). The alcohol was obtained as a light orange, semi-solid material.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.64 (br. s, 1 H) 4.28 (dd, *J*=5.89, 1.36 Hz, 2 H) 5.99 (dt, *J*=15.22, 5.92 Hz, 1 H) 6.45 (dd, *J*=15.08, 10.61 Hz, 1 H) 6.59 (d, *J*=15.67 Hz, 1 H) 6.82 (dd, *J*=15.62, 10.70 Hz, 1 H) 7.25 (tt, *J*=7.30, 1.80 Hz, 1 H) 7.34 (t, *J*=7.80 Hz, 2 H) 7.42 (d, *J*=7.44 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 63.74 (CH_2) 126.68 (CH) 127.92 (CH) 128.42 (CH) 128.90 (CH) 131.95 (CH) 132.75 (CH) 133.10 (CH) 137.41 (C)
GC-MS (EI) 160 ([M]$^+$, 22%) 141 (15%) 128 (39%) 117 (27%) 115 (52%) 104 (100%) 91 (59%) 77 (22%) 65 (11%) 63 (15%) 51 (21%) 39 (14%)

Synthesis of (2E,4E)-5-phenylpenta-2,4-dien-1-ol (4m)



3-Phenylpropanal[17]

[17] Tunge, J. A.; Jana, R. *Org. Lett.* **2009**, *11*, 971

To a 500 mL round bottom flask equipped with a stir bar, was added 3-phenyl-1-propanol (13.62 g, 0.100 mol, 1 equiv) and DCM (200 mL). The oxoammonium salt **1a** (4.50 g, 0.015 mol, 0.15 equiv) was added to the solution. While stirring, commercial bleach (8.25% w/w) (90.18 g, 0.100 mol, 1 equiv) was added all at once. The solution turned from a bright yellow to bright red color. The solution was allowed to stir vigorously at room temperature for 2 h and monitor by ^{1}H NMR to assess reaction completion. The reaction was judged to be complete at this time. The reaction mixture was transferred to a separatory funnel and diluted with mL of deionized water and extracted with DCM (3 × 75 mL). The combined organic extractions were washed with 150 mL of deionized water followed by 150 mL of brine. The organic layer was then dried with Na_2SO_4. The solvent was removed *in vacuo* by rotary evaporation. The crude aldehyde was then adhered to silica gel by mixing it with 1.5 weight equivalents silica gel (relative to the theoretical yield), dissolving it in CH_2Cl_2 and removing the solvent *in vacuo* by rotary evaporation. A plug of silica was then assembled. This was done by adding 3-4 weight equivalents of silica (again relative to the theoretical yield) to a 300 mL coarse-porosity fritted glass funnel. An appropriately sized piece of filter paper relative to the size of the funnel was used to the top of the dry silica gel layer and this layer was pre-wet with hexanes. The dry packed material was gentle added evenly atop the filter paper. Another piece of appropriately sized filter paper was added atop this layer. The desired aldehyde was eluted off the plug *via* a 95:5 by volume mixture of Hex:EtOAc (3 column volumes) followed by 9:1 by volume mixture of Hex:EtOAc (3 column volumes) . The solvent was removed *in vacuo* by rotary evaporation to afford the pure aldehyde (9.88 g, 74%) as clear, pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 2.79 (t, *J*=8.00 Hz, 2 H) 2.97 (t, *J*=7.60 Hz, 2 H) 7.17 - 7.24 (m, 3 H) 7.30 (*apparent triplet*, *J*=7.00 Hz, 2 H) 9.83 (t, *J*=1.46 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 28.38 (CH_2) 45.54 (CH_2) 126.57 (CH) 128.56 (CH) 128.87 (CH) 140.61 (C) 201.80 (C)
GC-MS (EI) 134 ($[M]^+$, 61%) 133 ($[M-1]^+$, 10%) 105 (33%) 103 (16%) 92 (72%) 91 (100%) 78 (47%) 65 (16%)

(*E*)-methyl 5-phenylpent-2-enoate[18]

Synthesis of (*E*)-methyl 5-phenylpent-2-enoate (6.00 g, 79%) was accomplished using the procedure for the preparation of (2*E*,4*E*)-methyl 5-phenylpenta-2,4-dienoate with the following modification: The reaction was conducted using 3-phenylpropanal (5.37 g, 0.040 mol). The ester was obtained as a clear, colorless oil.

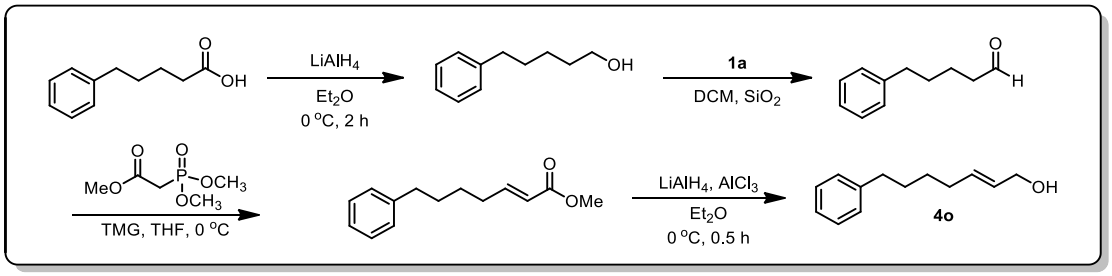
^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 2.54 (*apparent quartet*, *J*=7.50 Hz, 2 H) 2.78 (t, *J*=7.50 Hz, 2 H) 3.73 (s, 3 H) 5.86 (dt, *J*=15.67, 1.48 Hz, 1 H) 7.02 (dt, *J*=15.62, 6.86 Hz, 1 H) 7.17 - 7.24 (m, 3 H) 7.30 (t, *J*=7.20 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 34.12 (CH_2) 34.57 (CH_2) 51.66 (CH_3) 121.71 (CH) 126.42 (CH) 128.57 (CH) 128.73 (CH) 140.98 (CH) 148.58 (C) 167.22 (C)
GC-MS (EI) ($[M]^+$, %) 190 ($[M]^+$, 1%) 172 (5%) 144 (11%) 130 (9%) 117 (19%) 104 (46%) 91 (100%) 79 (10%) 77 (12%) 65 (21%) 57 (15%) 51 (12%) 39 (12%)
(*E*)-5-phenylpent-2-en-1-ol[19] (4n)

[18] Poeylaut-Palena, A. A.; Testero, S. A.; Mata, E. G. *J. Org. Chem.*, **2008**, *73*, 2024.
[19] Race, N. J.; Bower, J. F. *Org. Lett.*, **2013**, *15*, 4616.

Synthesis of (*E*)-5-phenylpent-2-en-1-ol (4.42 g, 91%) was accomplished using the procedure for the preparation of (*E*)-3-(p-tolyl)prop-2-en-1-ol, **4b**, with the following modification: The reaction was conducted using (*E*)-methyl 5-phenylpent-2-enoate (5.71 g, 0.030 mol). The alcohol was obtained as a clear, colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.29 (br. s, 1 H) 2.38 (*apparent quartet*, *J*=7.40 Hz, 2 H) 2.72 (*apparent triplet*, *J*=8.20 Hz, 2 H) 4.08 (t, *J*=5.06 Hz, 2 H) 5.62 - 5.79 (m, 2 H) 7.16 - 7.23 (m, 3 H) 7.29 (*apparent triplet*, *J*=7.20 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 34.23 (CH_2) 35.81 (CH_2) 63.97 (CH_2) 126.15 (CH) 128.60 (CH) 128.70 (CH) 129.88 (CH) 132.54 (CH) 141.97 (C)
GC-MS (EI) 162 ([M]$^+$, 1%) 144 (32%) 108 (11%) 91 (100%) 65 (21%) 41 (12%)

Synthesis of (E)-7-phenylhept-2-enal (4o)



5-phenylpentan-1-ol[20]

To an oven-dried 500 mL flask equipped with a stirbar was added $LiAlH_4$ (4.17 g, 0.110 mol, 1.1 equiv). The flask was sealed with a rubber septum and placed under a N_2 atmosphere *via* an inlet needle. The flask was then charged with anhydrous Et_2O (100 mL) and was cooled to 0 °C *via* an ice-water bath. After stirring for ten minutes, 5-phenylpentanoic acid (17.82 g, 0.100 mol, 1 equiv) in dry Et_2O (200 mL, 0.5 M in the acid) was added dropwise to the flask. The mixture was stirred at 0 °C for 3 h. After this time, the reaction was quenched at 0 °C by *very careful* addition of deionized H_2O (4.2 mL), 2 M NaOH (8.4 mL), and additional deionized H_2O (12.6 mL). **Note:** *Due to the amount of hydrogen gas evolved, it is advisable to add several vent needles to the septum.* The quenched solution was allowed to stir for about ten minutes, resulting in the formation of an off-white precipitate. The solids were removed by filtration and the filtrate was diluted with Et_2O (100 mL). The filtrate was transferred to a separatory funnel and washed with saturated aqueous sodium bicarbonate (200 mL), deionized H_2O (200 mL) and brine (200 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation affording the pure alcohol (14.39 g, 88%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.37 (t, *J* = 5.11 Hz, 1 H) 1.44 (quin, *J* = 7.80 Hz, 2 H) 1.66 (s, 4 H) 2.66 (t, *J* = 7.80 Hz, 2 H) 3.66 (q, *J* = 5.50 Hz, 2 H) 7.17 - 7.25 (m, 3 H) 7.31 (t, *J* = 7.70 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 25.66 (CH_2) 31.51 (CH_2) 32.89 (CH_2) 36.16 (CH_2) 63.17 (CH_2) 125.92 (CH) 128.52 (CH) 128.65 (CH) 142.82 (C)

[20] Szostak, M.; Collins, K. D.; Fazakerley, N. J.; Spain, M.; Procter, D. J. *Org. Biomol. Chem.*, **2012**, *10*, 5820.

GC-MS (EI) 164 ([M]$^+$, 3%) 146 (33%) 117 (51%) 104 (93%) 91 (100%) 77 (17%) 65 (24%) 39 (9%)

5-phenylpentanal[21]

To a 1000 mL flask equipped with stirbar was added 5-phenylpentan-1-ol (13.50 g, 0.0822 mol, 1 equiv) and DCM (550 mL, ≈ 0.15 M in the alcohol). After mixing for a few minutes, the oxoammonium salt **1a** (27.13 g, 0.0904 mol, 1.1 equiv) was added followed by 13.50 g of SiO_2 (1 mass equiv to substrate). The flask was sealed with a rubber septa and the mixture was allowed to stir vigorously for 48 h at room temperature. Upon reaction completion (confirmed by GC/MS analysis), the slurry was filtered through Celite® *via* a coarse porosity fritted funnel. The solids were rinsed thoroughly with Et_2O (≈ 400 mL). The filtrate was then adhered to silica gel by mixing it with 1.5 weight equivalents silica gel and removing the solvent *in vacuo* by rotary evaporation. A plug of silica was then assembled. This was done by adding 3-4 weight equivalents of silica (again relative to the theoretical yield) to a 600 mL coarse-porosity fritted glass funnel. An appropriately sized piece of filter paper relative to the size of the funnel was used to the top of the dry silica gel layer and this layer was pre-wet with hexanes. The dry packed material was gentle added evenly atop the filter paper. Another piece of appropriately sized filter paper was added atop this layer. The plug was eluted with a 95:5 by volume mixture of Hex:EtOAc (3 column volumes) followed by 9:1 (3 column volumes). The solvent was removed *in vacuo* by rotary evaporation to afford the pure aldehyde (11.25 g, 84%) as a clear, colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.71 (*apparent septet*, *J* = 3.90 Hz, 4 H) 2.42 - 2.52 (m, 2 H) 2.67 (s, 2 H) 7.21 (t, *J* = 8.20 Hz, 3 H) 7.31 (t, *J* = 7.80 Hz, 2 H) 9.79 (t, *J* = 1.70 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 21.80 (CH_2) 30.99 (CH_2) 35.77 (CH_2) 43.83 (CH_2) 125.97 (CH) 128.49 (CH) 128.51 (CH) 142.09 (C) 202.51 (C)
GC-MS (EI) 162 ([M]$^+$, 20%) 144 (13%) 129 (20%) 117 (25%) 105 (13%) 91 (100%) 77 (13%) 65 (22%) 51 (10%) 39 (11%)

(*E*)-methyl 7-phenylhept-2-enoate[22]

Synthesis of (*E*)-methyl 7-phenylhept-2-enoate (12.83 g, 85%) was accomplished using the procedure for the preparation of (2*E*,4*E*)-methyl 5-phenylpenta-2,4-dienoate with the following modification: The reaction was conducted using 5-phenylpentanal (11.25 g, 0.0694 mol). The ester was obtained as a clear, pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.53 (quin, *J* = 8.00 Hz, 2 H) 1.68 (quin, *J* = 7.40 Hz, 2 H) 2.26 (qd, *J* = 7.27, 1.56 Hz, 2 H) 2.65 (t, *J* = 7.59 Hz, 2 H) 3.75 (s, 3 H) 5.85 (dt, *J* = 15.57, 1.56 Hz, 1 H) 6.99 (dt, *J* = 15.57, 7.01 Hz, 1 H) 7.21 (t, *J* = 8.20 Hz, 3 H) 7.31 (t, *J* = 7.80 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 27.86 (CH_2) 31.14 (CH_2) 32.31 (CH_2) 35.92 (CH_2) 51.62 (CH_3) 121.29 (CH) 126.02 (CH) 128.56 (CH) 128.62 (CH) 142.45 (C) 149.60 (CH) 167.35 (C)
GC-MS (EI) 218 ([M]$^+$, 4%) 186 (6%) 158 (18%) 144 (7%) 130 (10%) 117 (16%) 113 (12%) 104 (15%) 91 (100%) 77 (9%) 68 (20%) 65 (18%) 39 (11%)

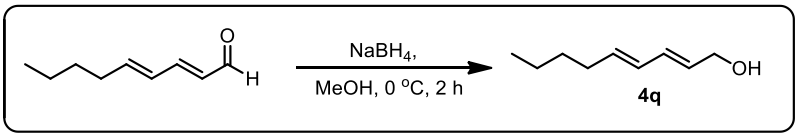
[21] Anderson, C. D.; Shea, K. J.; Rychnovsky, S. D. *Org. Lett.*, **2005**, *7*, 4879.
[22] De Esch, I. J. P.; Gaffar, A.; Menge, W. M. P. B.; Timmerman, H. *Bioorg. Med. Chem.* **1999**, *7*, 3003.

(*E*)-7-phenylhept-2-en-1-ol (4o)[23]

Synthesis of (*E*)-7-phenylhept-2-en-1-ol (6.78 g, 71%) was accomplished using the procedure for the preparation of (*E*)-3-(p-tolyl)prop-2-en-1-ol, **4b**, with the following modification: The reaction was conducted using (*E*)-methyl 7-phenylhept-2-enoate (10.92 g, 0.050 mol). The alcohol was obtained as a clear, colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.36 (br. s., 1 H) 1.46 (quin, *J* = 7.55 Hz, 2 H) 1.67 (quin, *J* = 7.60 Hz, 2 H) 2.11 (q, *J* = 6.80 Hz, 2 H) 2.64 (t, *J* = 7.50 Hz, 2 H) 4.11 (d, *J* = 4.96 Hz, 2 H) 5.58 - 5.80 (m, 2 H) 7.21 (s, 3 H) 7.30 (t, *J* = 7.40 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 29.01 (CH_2) 31.24 (CH_2) 32.33 (CH_2) 36.06 (CH_2) 64.05 (CH_2) 125.92 (CH) 128.53 (CH) 128.67 (CH) 129.35 (CH) 133.39 (CH) 142.86 (C)
GC-MS (EI) 190 ([M]$^+$, 0.1%) 159 (5%) 130 (12%) 91 (100%) 77 (5%) 65 (14%) 63 (4%) 51 (5%)

Synthesis of (2*E*,4*E*)-nona-2,4-dien-1-ol (4o)



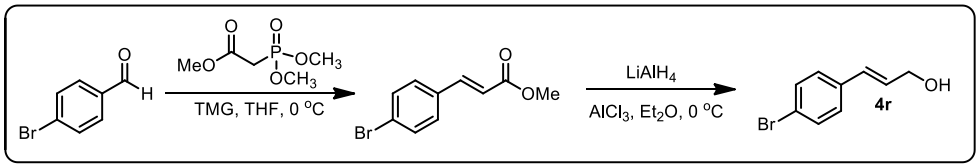
(2*E*,4*E*)-nona-2,4-dien-1-ol[24] **(4q)**

Synthesis of (2*E*,4*E*)-nona-2,4-dien-1-ol (2.79 g, 79%) was accomplished using the procedure for the preparation of (*E*)-2-methyl-3-phenylprop-2-en-1-ol, **4e**, with the following modification: The reaction was conducted using (2*E*,4*E*)-nona-2,4-dienal (3.46 g, 0.025 mol). The alcohol **4q** was obtained as a clear, pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 0.89 (t, *J*=6.90 Hz, 3 H) 1.23 - 1.46 (m, 6 H) 2.08 (q, *J*=6.97 Hz, 2 H) 3.48 (s, 1 H) 4.15 (d, *J*=6.08 Hz, 2 H) 5.66 - 5.77 (m, 2 H) 6.04 (dd, *J*=15.18, 10.56 Hz, 1 H) 6.21 (dd, *J*=15.18, 10.56 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 14.19 (CH_3) 22.50 (CH_2) 31.64 (CH_2) 32.57 (CH_2) 63.84 (CH_2) 129.59 (2 × CH) 132.43 (CH) 136.10 (CH)
GC-MS (EI) 154 ([M]$^+$, 7%) 97 (100%) 84 (13%) 81 (27%) 79 (48%) 77 (27%) 71 (14%) 67 (59%) 55 (15%) 45 (35%) 46 (41%) 39 (26%)

Synthesis of (*E*)-3-(4-bromophenyl)prop-2-en-1-ol (4r)

[23] Narsaiah, A. V.; Ghogare, R. S. *Synthesis*, **2011**, 3271.
[24] Kazmaier, U. *Tetrahedron*, **1998**, *54*, 1491.



***trans*-methyl 4-bromocinnamate**[7]

Synthesis of *trans*-methyl 4-bromocinnamate (17.52 g, 97%) was accomplished using the procedure for the preparation of (2*E*,4*E*)-methyl 5-phenylpenta-2,4-dienoate with the following modification: The reaction was conducted using 4-bromobenzaldehyde (13.88 g, 0.075 mol). The ester was obtained as a powdery, white solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 3.80 (s, 3 H) 6.42 (d, *J*=16.01 Hz, 1 H) 7.38 (dt, *J*=8.51, 1.60 Hz, 2 H) 7.52 (dt, *J*=8.37, 1.90 Hz, 2 H) 7.62 (d, *J*=16.01 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 52.06 (CH_3) 118.79 (CH) 124.82 (C) 129.71 (CH) 132.42 (CH) 133.58 (C) 143.74 (CH) 167.41 (C)
GC-MS (EI) 242 ($[M]^+$, ^{81}Br, 34%) 240 ($[M]^+$, ^{79}Br, 34%) 211 (^{81}Br, 58%) 209 (^{79}Br, 61%) 183 (^{81}Br, 17%) 181 (^{79}Br, 17%) 130 (11%) 102 (100%) 76 (28%) 63 (12%) 51 (26%)

(E)-3-(4-bromophenyl)prop-2-en-1-ol[7] **(4r)**

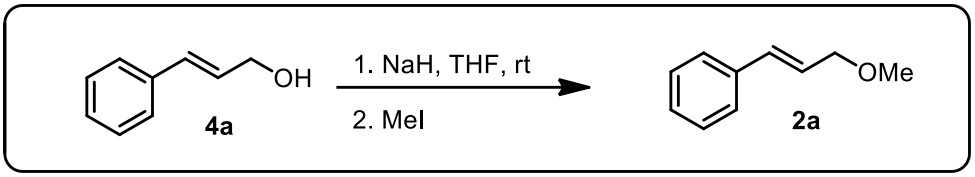
Synthesis of (*E*)-3-(4-bromophenyl)prop-2-en-1-ol (7.00 g, 66%) was accomplished using the procedure for the preparation of (*E*)-3-(p-tolyl)prop-2-en-1-ol, **4b**, with the following modification: The reaction was conducted using *trans*-methyl 4-bromocinnamate (12.06 g, 0.050 mol). The alcohol was obtained as a powdery, white solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.51 (t, *J*=5.86 Hz, 1 H) 4.34 (td, *J*=5.72, 1.51 Hz, 2 H) 6.38 (dt, *J*=15.86, 5.50 Hz, 1 H) 6.59 (d, *J*=15.91 Hz, 1 H) 7.27 (d, *J*=9.24 Hz, 2 H) 7.46 (dt, *J*=8.61, 2.50 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 63.80 (CH_2) 121.73 (C) 128.26 (CH) 129.61 (CH) 130.07 (CH) 131.99 (CH) 135.92 (C)
GC-MS (EI) 214 ($[M]^+$, ^{81}Br, 42%) 212 ($[M]^+$, ^{79}Br, 42%) 171 (^{81}Br, 49%) 169 (^{79}Br, 49%) 158 (^{81}Br, 24%) 156 (^{79}Br, 25%) 133 (100%) 115 (86%) 103 (54%) 91 (78%) 77 (76%) 65 (13%) 63 (38%) 55 (43%) 51 (46%) 39 (21%)

Synthesis of Allyl Ethers

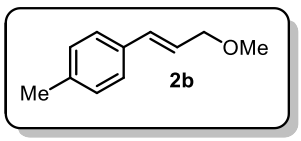
General Procedure A: Methylation of Allyl Alcohols with Methyl Iodide

(*E*)-(3-methoxyprop-1-en-1-yl)benzene[25] (2a)



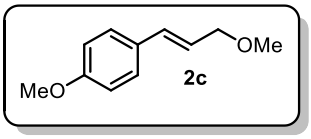
The following procedure is modification of the protocol outlined by Jamison.[25] To a flame-dried 250 mL round bottom flask equipped with a stir bar was added NaH[26] (2.40 g, 0.100 mol, 2 equiv) followed by anhydrous THF (125 mL). The flask was sealed with a rubber septum and placed under an N_2 atmosphere *via* an N_2 inlet needle. The alcohol **4a** (6.71 g, 0.050 mol, 1 equiv) dissolved in a minimum amount of THF (≈ 10 mL) was added to the flask dropwise over ten minutes. **CAUTION!** *Mildly exothermic, evolves H_2 gas!* The reaction mixture was allowed to stir at room temperature for 1.5 h during which time the solution transitioned from a pale yellow to a orangish-red. After this time, MeI (21.30 g, 0.150 mol, 3 equiv) was added to the flask and the solution was allowed to stir for six hours.[27] The reaction mixture was then *carefully* quenched with deionized water (≈ 30 mL) and transferred to a separatory funnel. The mixture was diluted with deionized water (150 mL) and Et_2O (150 mL) and the layers were separated. The aqueous layer was extracted with Et_2O (3 × 75 mL) and the combined organic layers were washed with deionized water (2 × 100 mL) followed by brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude ether. Further purification was accomplished by SiO_2 plug (95:5 to 9:1 Hex:EtOAc) to give the pure ether **2a** (5.74 g, 77%) as a clear, pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 3.45 (s, 3 H) 4.15 (dd, *J*=5.98, 1.22 Hz, 2 H) 6.36 (dt, *J*=15.94, 5.95 Hz, 1 H) 6.68 (d, *J*=16.01 Hz, 1 H) 7.30 (t, *J*=7.10 Hz, 1 H) 7.38 (t, *J*=7.52 Hz, 2 H) 7.46 (d, *J*=7.69 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 58.0 (CH_3), 73.1 (CH_2), 126.1(CH), 126.6 (CH), 127.8 (CH), 128.7 (CH), 132.5 (CH), 136.9 (C)
GC-MS (EI) 148 ([M]$^+$, 13%) 131 (100%) 115 (30%) 103 (88%) 91 (14%) 77 (81%) 63 (25%) 51 (61%) 39 (17%)

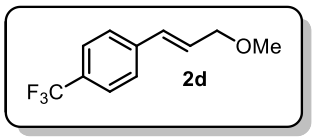


(*E*)-1-(3-methoxyprop-1-en-1-yl)-4-methylbenzene,[25] 2b (2.15 g, 74%) was prepared according to General Procedure A from **4b** (2.67 g, 0.018 mol) as a clear, colorless oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 2.37 (s, 3 H) 3.42 (s, 3 H) 4.11 (dd, *J*=5.90, 1.36 Hz, 2 H) 6.28 (dt, *J*=15.89, 5.90 Hz, 1 H) 6.62 (d, *J*=15.89 Hz, 1 H) 7.16 (d, *J*=8.17 Hz, 2 H) 7.33 (d, *J*=7.72 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 21.35 (CH_3) 58.05 (CH_3) 73.37 (CH_2) 125.06 (CH) 126.59 (CH) 129.44 (CH) 132.64 (CH) 134.14 (C) 137.64 (C) **GC-MS** (EI) 162 ([M]$^+$, 44%) 147 (66%) 132 (61%) 129 (44%) 119 (33%) 115 (100%) 103 (18%) 91 (77%) 77 (28%) 65 (21%) 63 (21%) 51 (22%)

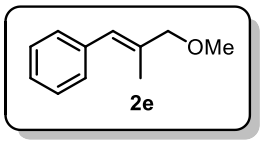
[25] Mastsubara, R.; Jamison, T. F. *J. Am. Chem. Soc.*, **2010**, *132*, 6880.
[26] NaH was pre-washed with pentane five times prior to use to remove the mineral oil. The now whitish solid was dried under light vacuum.
[27] The alkylation process can be dramatically accelerated by heating to 40-45 ºC without any detrimental effects



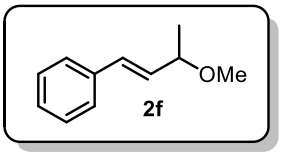
(*E*)-1-methoxy-4-(3-methoxyprop-1-en-1-yl)benzene,[28] **2c** (3.23 g, 87%) was prepared according to General Procedure A from **4c** (3.40 g, 0.0207 mol) as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 3.38 (s, 3 H) 3.78 (s, 3 H) 4.07 (d, *J*=6.20 Hz, 2 H) 6.16 (dt, *J*=15.90, 6.16 Hz, 1 H) 6.56 (d, *J*=15.91 Hz, 1 H) 6.86 (d, *J*=8.90 Hz, 2 H) 7.33 (d, *J*=8.71 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 55.42 (CH_3) 58.06 (CH_3) 73.50 (CH_2) 114.23 (CH) 123.94 (CH) 127.93 (CH) 129.75 (C) 132.40 (CH) 159.58 (C) **GC-MS** (EI) 178 ([M]$^+$, 45%) 147 (100%) 135 (31%) 131 (30%) 121 (12%) 115 (40%) 103 (53%) 91 (68%) 77 (45%) 65 (18%) 63 (21%) 51 (23%)



(*E*)-1-(3-methoxyprop-1-en-1-yl)-4-(trifluoromethyl)benzene,[25] **2d** (1.98 g, 74%) was prepared according to General Procedure A from **4d** (2.50 g, 0.0124 mol) as a clear, colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 3.41 (s, 3 H) 4.12 (dd, *J*=5.64, 1.22 Hz, 2 H) 6.38 (dt, *J*=16.00, 5.65 Hz, 1 H) 6.65 (d, *J*=16.01 Hz, 1 H) 7.48 (d, *J*=8.27 Hz, 2 H) 7.57 (d, *J*=8.32 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 58.53 (CH_3) 72.98 (CH_2) 124.49 (q, $J_{C\text{-}F}$ = 270.50 Hz, CF_3) 125.80 (q, $J_{C\text{-}C\text{-}C\text{-}F}$ = 3.85 Hz, 18 C) 126.86 (CH) 129.12 (CH) 129.71 (q, $J_{C\text{-}C\text{-}F}$ = 32.40 Hz, C) 130.82 (CH) 140.54 (q, $J_{C\text{-}C\text{-}C\text{-}C\text{-}C\text{-}F}$ =1.30 Hz, C) **^{19}F NMR** ($CDCl_3$, 377 MHz) δ ppm -65.69 (s) **GC-MS** (EI) 216 ([M]$^+$, 51%) 201 (12%) 197 (16%) 189 (21%) 185 (26%) 173 (16%) 166 (48%) 159 (13%) 153 (26%) 151 (33%) 148 (64%) 133 (59%) 127 (18%) 115 (100 %) 103 (17%) 75 (1%) 69 (14%) 45 (21)

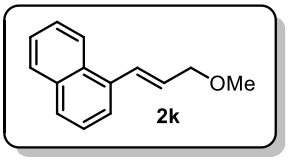


(*E*)-(3-methoxy-2-methylprop-1-en-1-yl)benzene,[29] **2e** (3.52 g, 64%) was prepared according to General Procedure A from **4e** (5.00 g, 0.0337mol) as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 1.97 (s, 3 H) 3.44 (s, 3 H) 4.04 (s, 2 H) 6.59 (s, 1 H) 7.28 (t, *J*=6.10 Hz, 1 H) 7.33 - 7.45 (m, 4 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 15.50 (CH_3) 57.86 (CH_3) 78.84 (CH_2) 126.60 (CH) 127.05 (CH) 128.26 (CH) 129.07 (CH) 135.23 (C) 137.72 (C) **GC-MS** (EI) 162 ([M]$^+$, 31%) 147 (62%) 131 (28%) 129 (35%) 115 (100%) 103 (11%) 91 (85%) 77 (20%) 65 (14%) 63 (16%) 51 (16%) 45 (17%) 39 (16%)

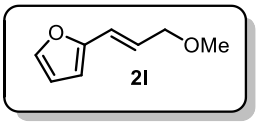


(*E*)-(3-methoxybut-1-en-1-yl)benzene,[30] **2f** (1.79 g, 79%) was prepared according to General Procedure A from **4f** (2.07 g, 0.0140 mol) as a clear, colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 1.43 (d, *J*=6.18 Hz, 3 H) 3.40 (s, 3 H) 3.96 (quin, *J*=6.64 Hz, 1 H) 6.18 (dd, *J*=15.96, 7.59 Hz, 1 H) 6.61 (d, *J*=15.96 Hz, 1 H) 7.31 (t, *J*=7.10 Hz, 1 H) 7.39 (t, *J*=7.57 Hz, 2 H) 7.48 (d, *J*=7.88 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 21.53 (CH_3) 56.13 (CH_2) 78.22 (CH_2) 126.60 (CH) 127.78 (CH) 128.71 (CH) 131.49 (CH) 131.62 (C) 136.82 (C) **GC-MS** (EI) 162 ([M]$^+$, 42%) 147 (100%) 131 (50%) 129 (34%) 115 (97%) 103 (29%) 91 (77%) 77 (42%) 65 (12%) 63 (17%) 50 (29%) 41 (59%)

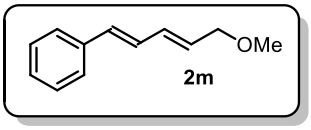
[28] Engler, T. A.; LaTessa, K. O.; Iyengar, R.; Chai, W.; Agrios, K. *Bioorg. Med. Chem.*, **1996**, *4*, 1755.
[29] Lu, T,-J.; Lin, C.-K. *J. Org. Chem.*, **2008**, *73*, 9527.
[30] Malkov, A. V.; Baxendale, I. R.; Dvořák, D.; Mansfield, D. J.; Kočovský, P. *J. Org. Chem.*, **1999**, *64*, 2737.



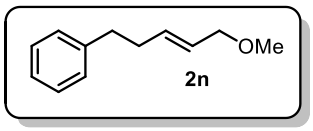
(*E*)-1-(3-methoxyprop-1-en-1-yl)naphthalene,[31] **2k** (2.29 g, 77%) was prepared according to General Procedure A from **4k** (2.76 g, 0.015 mol) as a clear, light brown oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 3.56 (s, 3 H), 4.28 (dd, *J* = 5.90, 1.36 Hz, 2 H), 6.44 (dt, *J* = 15.89, 5.90 Hz, 1 H), 7.50 (d, *J* = 15.44 Hz, 1 H), 7.55 (t, *J* = 7.72 Hz, 1 H), 7.61 (quint, *J* = 8.20, 1.40 Hz, 2 H), 7.73 (d, *J* = 7.27 Hz, 1 H), 7.89 (d, *J* = 8.17 Hz, 1 H), 7.95 (d, *J* = 7.72 Hz, 1 H), 8.27 (d, *J* = 8.17 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 58.14 (CH_3) 73.30 (CH_2) 123.94 (CH) 124.04 (CH) 125.72 (CH) 125.86 (CH) 126.12 (CH) 128.10 (CH) 128.63 (CH) 129.38 (CH) 129.48 (CH) 131.31 (C) 133.76 (C) 134.67 (C) **GC-MS** (EI) 198 ([M]$^+$, 11%) 165 (100%) 153 (51%) 128 (13%) 115 (14%) 82 (7%) 77 (7%) 63 (7%) 45 (8%)



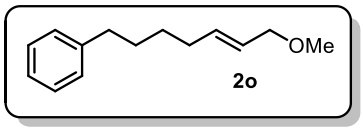
(*E*)-2-(3-methoxyprop-1-en-1-yl)furan[32]**, 2l** (3.86 g, 99%) was prepared according to General Procedure A from **4l** (3.50 g, 0.01819 mol) as a clear, light orange oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 3.40 (s, 3 H) 4.09 (dd, *J*=5.84, 1.07 Hz, 2 H) 6.23 (dt, *J*=15.81, 5.80 Hz, 1 H) 6.26 (d, *J*=3.45 Hz, 1 H) 6.39 (dd, *J*=3.16, 1.85 Hz, 1 H) 6.45 (d, *J*=15.91 Hz, 1 H) 7.37 (s, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 58.05 (CH_3) 72.70 (CH_2) 108.06 (CH) 111.38 (CH) 120.58 (CH) 124.83 (CH) 142.13 (CH) 152.58 (C) **GC-MS** (EI) 138 ([M]$^+$, 50%) 124 (14%) 110 (16%) 107 (58%) 96 (29%) 80 (94%) 78 (100%) 67 (37%) 65 (24%) 51 (30%) 39 (36%)



((1E,3E)-5-methoxypenta-1,3-dien-1-yl)benzene,[25] **2m** (1.61 g, 93%) was prepared according to General Procedure A from **4m** (1.60 g, 0.010 mol) as a clear, bright yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 3.39 (s, 3 H) 4.05 (d, *J*=6.03 Hz, 2 H) 5.91 (dt, *J*=15.24, 6.12 Hz, 1 H) 6.44 (dd, *J*=15.23, 10.51 Hz, 1 H) 6.58 (d, *J*=15.67 Hz, 1 H) 6.82 (dd, *J*=15.96, 11.00 Hz, 1 H) 7.25 (t, *J*=7.30 Hz, 1 H) 7.34 (t, *J*=7.59 Hz, 2 H) 7.42 (d, *J*=7.35 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 58.14 (CH_3) 72.98 (CH_2) 126.60 (CH) 127.78 (CH) 128.44 (CH) 128.80 (CH) 130.21 (CH) 132.90 (CH) 133.07 (CH) 137.37 (C) **GC-MS** (EI) 174 ([M]$^+$, 35%) 142 (63%) 131 (11%) 128 (100%) 115 (82%) 102 (15%) 91 (54%) 77 (19%) 65 (17%) 63 (20%) 51 (22%) 45 (7%) 39 (15%)



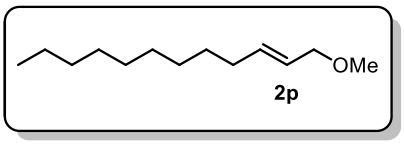
(*E*)-(5-methoxypent-3-en-1-yl)benzene,[25] **2n** (3.16 g, 90%) was prepared according to General Procedure A from **4n** (3.25 g, 0.025 mol) as a clear, colorless oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 2.42 (q, *J*=7.30 Hz, 2 H) 2.75 (t, *J*=8.17 Hz, 2 H) 3.33 (s, 2 H) 3.90 (d, *J*=5.90 Hz, 2 H) 5.63 (dt, *J*=15.44, 6.80 Hz, 1 H) 5.79 (dt, *J*=15.44, 7.70 Hz, 1 H) 7.22 (d, *J*=8.17 Hz, 3 H) 7.32 (t, *J*=7.30 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 34.35 (CH_2) 35.80 (CH_2) 57.95 (CH_3) 73.40 (CH_2) 126.11 (CH) 127.10 (CH) 128.58 (CH) 128.67 (CH) 134.00 (CH) 142.02 (C) **GC-MS** (EI) 176 ([M]$^+$, 0.1%) 144 (31%) 129 (20%) 115 (7%) 104 (4%) 91 (100%) 85 (15%) 71 (15%) 65 (23%) 56 (17%) 45 (14%)



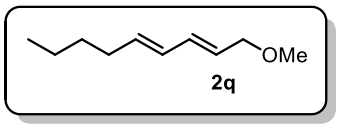
(*E*)-(7-methoxyhept-5-en-1-yl)benzene,[25] **2o** (4.54 g, 89%) was prepared according to General Procedure A from **4o** (4.76 g, 0.025 mol) as a clear, colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 1.46 (quin, *J*=7.55 Hz, 2 H) 1.66 (quin, *J*=7.60 Hz, 2 H) 2.11 (q, *J*=7.22

[31] Pérez, M.; Fañanás-Mastral, M.; Hornillos, V.; Rudolph, A.; Bos, P. H.; Harutyunyan, S. R.; Feringa, B. L. *Chem. Eur. J.*, **2012**, *18*, 11880.
[32] Hayashi, Y.; Mukaiyama, T. *Chem. Lett.*, **1987**, 181.

Hz, 2 H) 2.63 (t, *J*=7.71 Hz, 2 H) 3.34 (s, 3 H) 3.88 (d, *J*=6.13 Hz, 2 H) 5.57 (dtt, *J*=15.47, 6.30, 6.30, 1.30, 1.30 Hz, 1 H) 5.72 (dt, *J*=15.70, 6.60 Hz, 1 H) 7.17 - 7.22 (m, 3 H) 7.27 - 7.32 (m, 2 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 28.83 (CH_2) 31.10 (CH_2) 32.27 (CH_2) 35.92 (CH_2) 57.72 (CH_3 73.31 (CH_2) 125.75 (CH) 126.52 (CH) 128.35 (CH) 128.49 (CH) 134.50 (CH) 142.64 (C) **GC-MS** (EI) 204 ([M]$^+$, 0.1%) 172 (14%) 144 (13%) 129 (13%) 117 (22%) 104 (71%) 91 (100%) 81 (15%) 77 (15%) 71 (46%) 65 (25%) 53 (8%) 45 (19%) 41 (18%) 39 (16%)



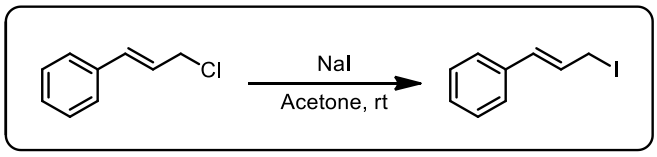
(*E*)-1-methoxydodec-2-ene,[25] **2p** (3.93 g, 99%) was prepared according to General Procedure A from **4p** (3.69 g, 0.020 mol) as a clear, colorless oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 0.90 (t, *J*=7.30 Hz, 3 H) 1.21 - 1.35 (m, 12 H) 1.39 (quin, *J*=7.30 Hz, 2 H) 2.06 (q, *J*=6.80 Hz, 2 H) 3.29 - 3.34 (m, 3 H) 3.87 (d, *J*=5.90 Hz, 2 H) 5.55 (dt, *J*=14.53, 5.90 Hz, 1 H) 5.71 (dt, *J*=15.44, 5.90 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 14.31 (CH_3) 22.94 (CH_2) 29.40 (CH_2) 29.47 (CH_2) 29.61 (CH_2) 29.78 (CH_2) 29.85 (CH_2) 31.87 (CH_2) 32.19 (CH_2) 57.81 (CH_3) 73.56 (CH_2) 126.32 (CH) 135.22 (CH) **GC-MS** (EI) 198 ([M]$^+$, 1%) 139 (2%) 124 (2%) 113 (2%) 110 (5%) 96 (11%) 85 (20%) 82 (21%) 71 (100%) 67 (18%) 58 (15%) 55 (25%) 45 (16%) 41 (43%) 39 (13%)



(2*E*,4*E*)-1-methoxynona-2,4-diene,[25] **2q** (2.14 g, 93%) was prepared according to General Procedure A from **4p** (2.10 g, 0.015 mol) as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 0.89 (t, *J*=7.20 Hz, 3 H) 1.24 - 1.41 (m, 4 H) 2.08 (q, *J*=6.68 Hz, 2 H) 3.31 (s, 3 H) 3.92 (d, *J*=6.37 Hz, 2 H) 5.55 - 5.77 (m, 2 H) 6.04 (t, *J*=13.00 Hz, 1 H) 6.20 (dd, *J*=14.48, 11.17 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 14.05 (CH_3) 22.41 (CH_2) 31.59 (CH_2) 32.49 (CH_2) 57.86 (CH_3) 73.09 (CH_2) 126.93 (CH) 129.68 (CH) 133.56 (CH) 135.66 (CH) **GC-MS** (EI) 154 ([M$^+$], 1%) 140 (9%) 122 (4%) 98 (8%) 96 (13%) 83 (72%) 79 (42%) 77 (29%) 69 (33%) 55 (76%) 41 (100%) 39 (50%)

General Procedure B: Alkylation of Alcohols with Cinnamyl Iodide

Finkelstein Reaction to Prepare Requisite Iodide



Cinnamyl Iodide[33]

The following procedure is a modification of the protocol outlined by Kaiser.[33] To a 250 mL round bottom flask equipped with a large stir bar was added NaI (16.79 g, 0.072 mol, 1.2 equiv) and acetone (60 mL). After stirring for five minutes, cinnamyl chloride (9.17 g 0.060 mol, 1 equiv) was added all at once turning the solution light yellow. The heterogeneous reaction mixture was allowed to stir vigorously for 1.5 h.[34] After this time, the solvent was removed *in vacuo* by rotary evaporation in a 20 °C water bath.[35] The resulting slurry was taken up in anhydrous Et_2O and

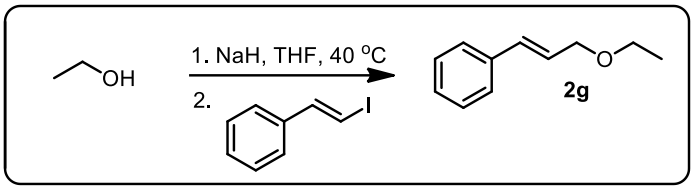
[33] Kaiser, C.; Burger, A.; Zirngibl, L.; Davis, C. S.; Zirkle, C. L. *J. Org. Chem.*, **1962**, *27*, 768.
[34] Longer reaction times lead to significant decomposition.
[35] This compound is thermally unstable and thus it is *imperative* that a room temperature or cooler bath be used during solvent removal.

filtered through a coarse fritted funnel. The filtrate was collected and the solvent was removed *in vacuo* by rotary evaporation in a 20 °C water bath giving the desired iodide[36] (13.44 g, 92 %) as a off-white powdery solid.

^{1}H NMR ($CDCl_3$, 300 MHz) δ ppm 4.17 (d, *J*=7.94 Hz, 2 H) 6.51 (dt, *J*=15.75, 7.90 Hz, 1 H) 6.66 (d, *J*=15.55 Hz, 1 H) 7.29 - 7.55 (m, 5 H)
^{13}C NMR ($CDCl_3$, 75 MHz) δ ppm 7.39 (CH_2) 126.69 (CH) 126.96 (CH) 128.24 (CH) 128.72 (CH) 133.13 (CH) 135.91 (C)
GC-MS (EI) 244 ([M^+], 56%), 167 (43%), 127 (31%), 117 (100%), 106 (11%), 91 (19%), 77 (9%), 57 (23%), 43 (25%)

General Alkylation Procedure using Cinnamyl Iodide

(*E*)-(3-methoxyprop-1-en-1-yl)benzene[25] (2g)

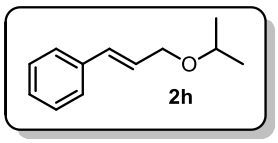


To a flame-dried 250 mL round bottom flask equipped with a stir bar was added NaH[37] (5.04 g, 0.210 mol, 6 equiv) followed by anhydrous THF (88 mL). The flask was sealed with a rubber septum and placed under an N_2 atmosphere *via* an N_2 inlet needle. Absolute ethanol (4.83 g, 0.105 mol, 3 equiv) was added to the flask dropwise over ten minutes. **CAUTION!** *Mildly exothermic, evolves H_2 gas!* The reaction mixture was allowed to stir at room temperature for 1.5 h during which time the solution transitioned from a pale yellow to a light orange. After this time, cinnamyl iodide (8.54 g, 0.035 mol, 1 equiv) dissolved in a minimum amount of THF (≈ 12 mL) was added to the flask. After five minutes, the flask was heated to 40 ° C and the solution was allowed to stir for six hours. The reaction mixture was then *carefully* quenched with deionized water (≈ 50 mL) and transferred to a separatory funnel. The mixture was diluted with deionized water (150 mL) and Et_2O (150 mL) and the layers were separated. The aqueous layer was extracted with Et_2O (3 × 75 mL) and the combined organic layers were washed with deionized water (2 × 100 mL) followed by brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude ether. Further purification was accomplished by SiO_2 plug (95:5 to 9:1 Hex:EtOAc) to give the pure ether **2g** (5.17 g, 91%) as a clear, yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.32 (t, *J*=7.20 Hz, 3 H) 3.60 (q, *J*=7.01 Hz, 2 H) 4.19 (d, *J*=5.98 Hz, 2 H) 6.37 (dt, *J*=15.91, 6.01 Hz, 1 H) 6.67 (d, *J*=15.96 Hz, 1 H) 7.28 (t, *J*=7.30 Hz, 1 H) 7.36 (t, *J*=7.54 Hz, 2 H) 7.45 (d, *J*=7.64 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 15.38 (CH_3) 65.76 (CH_2) 71.28 (CH_2) 126.45 (CH) 126.57 (CH) 127.69 (CH) 128.64 (CH) 132.23 (CH) 136.90 (C)

[36] This compound is very unstable and cannot be stored neat at room temperature for extended periods. However, storage at -20 °C dramatically prolongs its lifetime and is useable up to two weeks. After this time significant decomposition is observed.
[37] NaH was pre-washed with pentane five times prior to use to remove the mineral oil. The now whitish solid was dried under light vacuum.

GC-MS (EI) 162 ([M]$^+$, 23%) 133 (27%) 117 (47%) 115 (62%) 105 (100%) 91 (38%) 77 (34%) 63 (12%) 51 (15%)

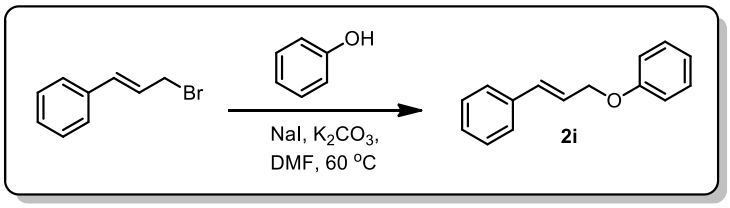


(*E*)-(3-isopropoxyprop-1-en-1-yl)benzene,[38] **2h** (3.18 g, 52%) was prepared according to General Procedure B from cinnamyl iodide (8.54 g, 0.035 mol) with the following modification: anhydrous isopropanol (6.31 g, 0.105 mol) was used in place of ethanol. The ether **2h** was obtained as a clear, pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.28 (d, *J*=6.13 Hz, 6 H) 3.74 (dt, *J*=12.21, 6.11 Hz, 1 H) 4.19 (d, *J*=5.94 Hz, 2 H) 6.37 (dt, *J*=15.90, 5.92 Hz, 1 H) 6.67 (d, *J*=15.96 Hz, 1 H) 7.28 (t, *J*=7.00 Hz, 1 H) 7.36 (t, *J*=7.52 Hz, 2 H) 7.45 (d, *J*=7.69 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 22.3 (CH_3) 68.8 (CH) 71.1 (CH_2) 126.6 (CH) 127.1 (CH) 127.6 (CH) 128.6 (CH) 131.8 (CH) 137.1 (C)
GC-MS (EI) 176 ([M]$^+$, 15%) 134 (30%) 117 (57%) 115 (71) 105 (100%) 103 (20%) 91 (47%) 77 (30%) 51 (12%)

Route C: Other Methods for Preparation of Substrates (2l and 2j)

(cinnamyloxy)benzene



(cinnamyloxy)benzene[39]

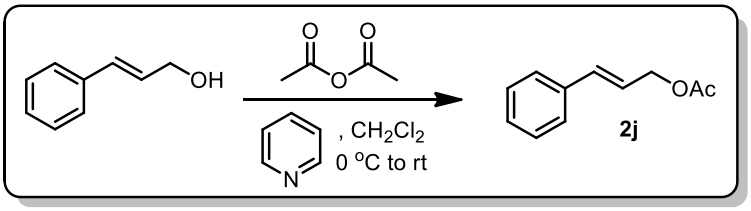
This procedure is a modification of the procedure outlined by McQuaid et al.[40]**Error! Bookmark ot defined.** To a 250 mL flask equipped with a large stir bar was added K_2CO_3 (12.62 g, 0.0913 mol, 3 equiv), DMF (20 mL), and NaI (0.456 g, 0.00305 mol, 0.1 equiv). At this time, phenol (3.01 g, 0.0320 mol, 1.05 equiv) was added all at once turning the solution pale yellow. Cinnamyl bromide (6.00 g, 0.0305 mol, 1 equiv) was then added all at once. The flask was equipped with an air-cooled condenser and heated to 60 °C in an oil bath. The solution was stirred at this temperature for 12 h. At this time, the solids of the now red-brown solution were removed by filtration through a fritted funnel, washing thoroughly with Et_2O (≈ 250 mL). The cloudy filtrate was transferred to a separatory funnel and washed with deionized H_2O (3 × 150 mL). The organic layer was washed with 2 M NaOH (75 mL), deionized water (100 mL), brine (150 mL) and dried with Na_2SO_4. The solvent was removed *in vacuo* by rotary evaporation affording the pure ether (3.92 g, 61%) as a flaky, off-white crystalline solid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 4.76 (d, *J*=5.79 Hz, 2 H) 6.48 (dt, *J*=15.96, 5.77 Hz, 1 H) 6.79 (d, *J*=16.06 Hz, 1 H) 7.02 (t, *J*=6.70 Hz, 3 H) 7.26 - 7.41 (m, 5 H) 7.47 (d, *J*=7.78 Hz, 2 H)

[38] Kim, H.; Lee, C. *Org. Lett.*, **2002**, *4*, 4369.
[39] Hajra, S.; Sinha, D. *J. Org. Chem.*, **2011**, *76*, 7334.
[40] McQuaid, K. M.; Long, J. Z.; Sames, D. *Org. Lett.* **2009**, *11*, 2972.

^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 68.82 (CH_2) 115.07 (CH) 121.09 (CH) 124.80 (CH) 126.85 (CH) 128.15 (CH) 128.86 (CH) 129.84 (CH) 133.22 (CH) 136.74 (C) 158.90 (C)
GC-MS (EI) 210 ([M]$^+$, 1%) 117 (100%) 116 (12%) 115 (51%) 102 (3%) 91 (25%) 77 (7%) 65 (22%) 51 (8%) 39 (14%)

Synthesis of Cinnamyl Acetate (2j)



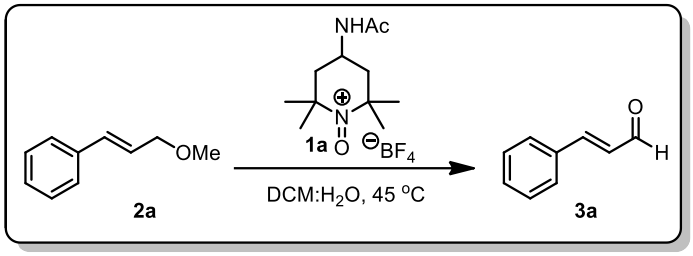
Cinnamyl acetate[41] (2j)

To a 100 mL round bottom flask equipped with a stir bar was added cinnamyl alcohol (6.71 g, 0.050 mol, 1 equiv) and CH_2Cl_2 (10 mL). The flask was cooled to 0 °C in an ice bath for ten minutes. After this time, acetic anhydride (17.1 g, 0.167 mol, 3.4 equiv) was added followed by dropwise addition of pyridine (13.21 g, 0.167 mol, 3.4 equiv). The solution was allowed to stir for 30 minutes at 0 °C. After this time, the solution was allowed to warm to room temperature and was stirred overnight. The solution was transferred to a separatory funnel and diluted with 150 mL of CH_2Cl_2 and 150 mL of aqueous 2 M HCl. The layers were separated and the acid layer was extracted with CH_2Cl_2 (2 × 50mL). The combined organic layers were washed with 2 M HCl (100 mL), saturated aqueous $NaHCO_3$ (2 ×100 mL), deionized H_2O (100 mL), and brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude acetate. Further purification was accomplished by vacuum distillation (93-95 °C @ 0.3 mmHg) to give the pure acetate **2j** (7.03 g, 80 %) as a clear, colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 2.12 (s, 3 H) 4.76 (d, *J*=6.42 Hz, 2 H) 6.32 (dt, *J*=15.90, 6.43 Hz, 1 H) 6.68 (d, *J*=15.91 Hz, 1 H) 7.29 (t, *J*=7.20 Hz, 1 H) 7.35 (t, *J*=7.47 Hz, 2 H) 7.42 (d, *J*=7.15 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 21.05 (CH_3) 65.14 (CH_2) 123.33 (CH) 126.73 (CH) 128.18 (CH) 128.72 (CH) 134.27 (CH) 136.34 (C) 170.84 (C)
GC-MS (EI) 176 ([M]$^+$, 14%) 134 (25%) 117 (36%) 116 (38%) 115 (100%) 105 (40%) 103 (21%) 91 (30%) 77 (36%) 63 (13%) 51 (18%) 43 (90%)

General Oxidative Cleavage Protocol of Allyl Ethers using 1a

[41] Henderson, W. H.; Check, C. T.; Proust, N.; Stambuli, J. P. *Org. Lett.*, **2010**, *12*, 824.

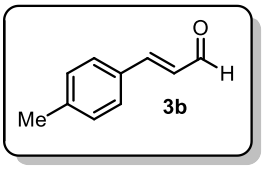


Cinnamaldehyde[42] (from 2a)

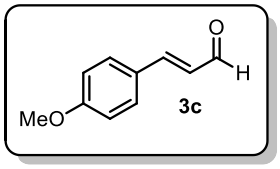
To a 100 mL round bottom flask equipped with a stir bar was added the ether **2a** (1.48 g, 0.010 mol, 1 equiv). CH_2Cl_2 (40 mL), and deionized water (10 mL). After stirring for five minutes, the oxoammonium salt **1a** (6.302 g, 0.021 mol, 2.1 equiv)was added to the flask and the flask was equipped with a reflux condenser. The flask was heated to 45 °C and allowed to stir overnight. The reaction mixture gradually became orange during this time. Reaction progress was monitored by GC/MS and/or ^{1}H NMR. After 12 h the reaction was judged to be complete.[43,4445] At this time the reaction mixture was cooled to room temperature and transferred to a separatory funnel. The mixture was diluted with deionized water (100 mL) and Et_2O (150 mL) the layers were separated. The aqueous layer was extracted with Et_2O (5 × 50 mL). The combined organic layers were washed with deionized water (2 × 100 mL) and brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude aldehyde. The crude aldehyde was then adhered to silica gel by mixing it with 1.5 weight equivalents silica gel (relative to the theoretical yield), dissolving it in CH_2Cl_2 and removing the solvent *in vacuo* by rotary evaporation. A plug of silica was then assembled. This was done by adding 3-4 weight equivalents of silica (again relative to the theoretical yield) to a 60 mL coarse-porosity fritted glass funnel. An appropriately sized piece of filter paper relative to the size of the funnel was used to the top of the dry silica gel layer. The dry packed material was gentle added evenly atop the filter paper. Another piece of appropriately sized filter paper was added over this layer. The desired aldehyde was eluted off the plug *via* a 95:5 by volume mixture of Hex:EtOAc (3 column volumes) followed by 9:1 by volume mixture of Hex:EtOAc (3 column volumes) . The solvent was removed *in vacuo* by rotary evaporation to afford the pure aldehyde (1.23 g, 93%) as clear, pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 6.75 (dd, *J*=15.97, 7.71 Hz, 1 H) 7.43 - 7.50 (m, 3 H) 7.52 - 7.64 (m, 3 H) 9.74 (d, *J*=7.68 Hz, 1 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 128.66 (CH) 128.76 (CH) 129.27 (CH) 131.43 (CH) 134.18 (C) 152.89 (CH) 193.80 (C)
GC-MS (EI) 132 ([M]$^+$, 58%) 131 (100%) 103 (62%) 77 (51%) 63 (12%) 51 (38%)

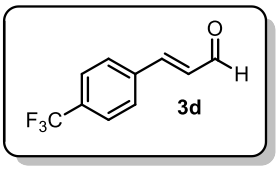
[42] Park, C. P.; Kim, D.-P. *J. Am. Chem. Soc.*, **2010**, *132*, 10102.
[43] The rate at which oxidative cleavage occurs dramatically varies between substrates. In several cases, we noted that the reaction would stall even after 24 h (usually at 90% conversion). To alleviate this, more **1a** was added (typically in 0.2 equiv intervals) and subsequently checked after 2 h. This process was repeated until complete or near complete cleavage was observed. Separation of aldehydes from their corresponding allyl ethers is difficult but can be accomplished using SiO_2 chromatography.
[44] If the reaction does stall and the goal is deprotection, it is advisable to simply carry the crude material to the reduction step
[45] We are unsure why in some cases the reaction stalls but we believe that it may relate to the oxoammonium salt function in two roles: One as a phase-transfer catalyst and one as the oxidant. Towards the end of the reaction, much of the oxidant is consumed in oxidation of the substrate and methanol thus limiting its ability to fulfill both roles. Another possibility is the competitive over-oxidation of methanol to CO_2 by way of formic acid.



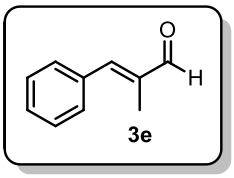
trans-4-methylcinnamaldehyde,[46] **3b** (1.154 g, 79%) was prepared according to general cleavage protocol from **2b** (1.622 g, 0.010 mol) as a clear, bright yellow. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 2.41 (s, 3 H) 6.69 (dd, *J*=15.91, 7.71 Hz, 1 H) 7.25 (d, *J*=8.13 Hz, 2 H) 7.40 - 7.52 (m, 3 H) 9.69 (d, *J*=7.74 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 21.61 (CH_3) 127.73 (CH) 128.60 (CH) 129.91 (CH) 131.41 (C) 141.99 (C) 152.93 (CH) 193.73 (C) **GC-MS** (EI) 146 ([M]$^+$, 17%) 131 (100%) 117 (30%) 115 (43%) 103 (23%) 91 (33%) 89 (12%) 77 (7%) 65 (14%) 63 (15%) 51 (10%) 39 (11%)



trans-4-methoxycinnamaldehyde,[47] **3c** (1.105 g, 54%) was prepared according to general cleavage protocol from **2c** (1.782 g, 0.010 mol) as a clear, bright yellow. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 3.89 (s, 3 H) 6.64 (dd, *J*=15.83, 7.76 Hz, 1 H) 6.93 - 7.00 (m, 2 H) 7.45 (d, *J*=15.89 Hz, 1 H) 7.50 - 7.59 (m, 2 H) 9.68 (d, *J*=7.70 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 55.71 (CH_3) 114.83 (CH) 126.80 (CH) 127.05 (C) 130.59 (CH) 152.91 (CH) 162.46 (C) 193.90 (C) **GC-MS** (EI) 162 ([M]$^+$, 100%) 147 (20%) 134 (32%) 131 (88%) 121 (13%) 119 (56%) 108 (45%) 103 (32%) 91 (77%) 77 (37%) 65 (48%) 63 (48%) 51 (25%)

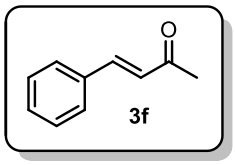


trans-4-(trifluoromethyl)cinnamaldehyde,[48] **3d** (1.297 g, 90%) was prepared according to general cleavage protocol from **2d** (1.55 g, 0.007 mol) with the following modifications: 1) The reaction was conducted over 72 h 2) An additional 1.2 equiv of **1a** was added to the reaction in 0.2 equiv increments during the course of the 72 h. The aldehyde **3d** was obtained as a clear, bright yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 6.78 (dd, *J*=16.06, 7.49 Hz, 1 H) 7.51 (d, *J*=16.06 Hz, 1 H) 7.63 - 7.74 (m, 4 H) 9.76 (d, *J*=7.49 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 123.90 (q, $J_{C\text{-}F}$ = =272.40 Hz, CF_3) 126.14 (q, $J_{C\text{-}C\text{-}C\text{-}F}$ = 3.85 Hz, CH) 128.73 (CH) 130.66 (CH) 132.60 (q, $J_{C\text{-}C\text{-}F}$ = 32.80 Hz, C) 137.57 (C) 150.36 (CH) 193.27 (C) **^{19}F NMR** ($CDCl_3$, 377 MHz) δ ppm -66.14 (s) **GC-MS** (EI) 200 ([M]$^+$, 12%) 181 (11%) 171 (15%) 151 (61%) 145 (10%) 131 (100%) 125 (8%) 103 (44%) 77 (13%) 75 (21%) 69 (12%) 51 (13%)

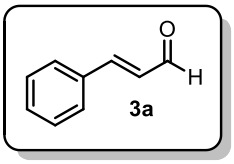


α-methylcinnamaldehyde,[49] **3e** (1.448 g, 99%) was prepared according to general cleavage protocol from **2e** (1.622 g, 0.010 mol) with the following modifications: 1) The reaction was conducted over 36 h 2) An additional 1.2 equiv of **1a** was added to the reaction in 0.2 equiv increments during the course of the 36 h. The aldehyde **3e** was obtained as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 2.11 (s, 3 H) 7.31 (s, 1 H) 7.43 (t, *J*=7.80 Hz, 1 H) 7.49 (t, *J*=7.27 Hz, 2 H) 7.57 (d, *J*=7.27 Hz, 2 H) 9.62 (s, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 11.18 (CH_3) 128.96 (CH) 129.80 (C) 130.26 (CH) 135.40 (C) 138.64 (C) 150.02 (CH) 195.77 (C) **GC-MS** (EI) 146 ([M]$^+$, 51%) 145 (100%) 128 (12%) 117 (96%) 115 (71%) 103 (25%) 89 (19%) 78 (28%) 65 (18%) 63 (19%) 51 (25%) 39 (26%)

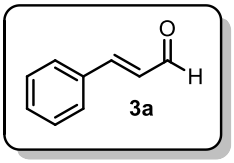
[46] Mueller, C. A.; Pfaltz, A. *Angew. Chem. Int. Ed.*, **2008**, *47*, 3363.
[47] Chavhan, S. W.; Cook, M. J. *Chem. Eur. J.*, **2014**, *20*, 4891.
[48] Hayashi, Y.; Itoh, T.; Ishikawa, H. *Adv. Synth. Catal.*, **2013**, *355*, 3661.
[49] Pelletier, G.; Bechara, W. S.; Charette, A. N. *J. Am. Chem. Soc.*, **2010**, *132*, 12817.



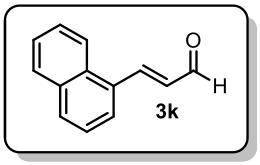
(*E*)-4-phenylbut-3-en-2-one,[50] **3f** (1.189 g, 83%) was prepared according to general cleavage protocol from **2f** (1.433 g, 0.00883 mol) with the following modifications: 1) The reaction was conducted over 36 h 2) An additional 0.8 equiv of **1a** was added to the reaction in 0.2 equiv increments during the course of the 36 h. The ketone **3f** was obtained as a clear, yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 2.41 (s, 3 H) 6.74 (d, *J*=16.32 Hz, 1 H) 7.37 - 7.46 (m, 3 H) 7.49 - 7.61 (m, 3 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 27.79 (CH_3) 127.45 (CH) 128.53 (CH) 129.25 (CH) 130.79 (CH) 134.72 (C) 143.68 (CH) 198.63 (C) **GC-MS** (EI) 146 ([M]$^+$, 40%) 131 (80%) 103 (100%) 77 (64%) 63 (13%) 51 (40%) 43 (30%) 39 (10%)



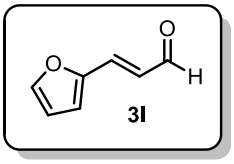
***trans*-cinnamaldehyde**[42] **(from 2g),** **3a** (1.226 g, 93%) was prepared according to general cleavage protocol from **2g** (1.622 g, 0.010 mol) as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 6.75 (dd, *J*=15.97, 7.71 Hz, 1 H) 7.43 - 7.50 (m, 3 H) 7.52 - 7.64 (m, 3 H) 9.74 (d, *J*=7.68 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 128.66 (CH) 128.76 (CH) 129.27 (CH) 131.43 (CH) 134.18 (C) 152.89 (CH) 193.80 (C) **GC-MS** (EI) 132 ([M]$^+$, 58%) 131 (100%) 103 (62%) 77 (51%) 63 (12%) 51 (38%)



***trans*-cinnamaldehyde**[42] **(from 2h),** **3a** (1.163 g, 88%) was prepared according to general cleavage protocol from **2h** (1.763 g, 0.010 mol) as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 6.75 (dd, *J*=15.97, 7.71 Hz, 1 H) 7.43 - 7.50 (m, 3 H) 7.52 - 7.64 (m, 3 H) 9.74 (d, *J*=7.68 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 128.66 (CH) 128.76 (CH) 129.27 (CH) 131.43 (CH) 134.18 (C) 152.89 (CH) 193.80 (C) **GC-MS** (EI) 132 ([M]$^+$, 58%) 131 (100%) 103 (62%) 77 (51%) 63 (12%) 51 (38%)

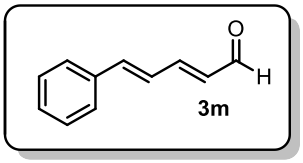


(*E*)-3-(naphthalen-1-yl)acrylaldehyde,[51] **3k** (1.610 g, 90%) was prepared according to general cleavage protocol from **2k** (1.945 g, 0.00981 mol) as a clear, yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 6.88 (dd, *J*=16.00, 7.38 Hz, 1 H) 7.53 - 7.69 (m, 3 H) 7.85 (d, *J*=7.38 Hz, 1 H) 7.94 (d, *J*=8.00 Hz, 1 H) 7.99 (d, *J*=8.00 Hz, 1 H) 8.22 (d, *J*=8.61 Hz, 1 H) 8.36 (d, *J*=16.00 Hz, 1 H) 9.89 (d, *J*=7.38 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 123.08 (CH) 125.78 (CH) 126.02 (CH) 126.72 (CH) 127.57 (CH) 129.28 (CH) 131.25 (CH) 131.28 (C) 131.50 (C) 131.90 (CH) 134.06 (C) 149.57 (CH) 193.89 (C) **GC-MS** (EI) 182 ([M]$^+$, 56%) 181 (98%) 153 (100%) 128 (19%) 102 (5%) 98 (5%) 76 (14%) 63 (10%) 51 (7%) 39 (3%)

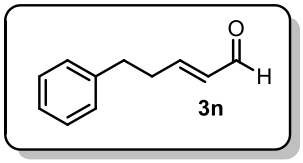


(*E*)-3-(furan-2-yl)acrylaldehyde,[48] **3l** (0.168 g, 14%) was prepared according to general cleavage protocol[52] from **2l** (1.382 g, 0.010 mol) as a clear, orange oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 6.56 (dd, *J*=3.41, 1.80 Hz, 1 H) 6.61 (dd, *J*=15.69, 7.91 Hz, 1 H) 6.79 (d, *J*=3.45 Hz, 1 H) 7.24 (d, *J*=15.67 Hz, 1 H) 7.55 - 7.61 (m, 1 H) 9.64 (dd, *J*=7.88, 0.83 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 113.14 (CH) 116.93 (CH) 126.35 (CH) 138.04 (CH) 146.18 (CH) 150.92 (C) 193.14 (C) **GC-MS** (EI) 122 ([M]$^+$, 64%) 94 (79%) 68 (25%) 65 (100%) 63 (36%) 51 (10%)

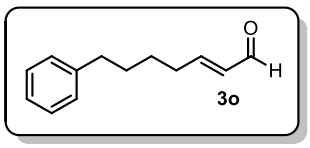
[50] Mueller, T.; Djanashvili, K.; Arends, I. W. C. E.; Peters, J. A.; Hanefeld, U. *Chem. Commun.*, **2013**, *49*, 361.
[51] Kim, E.; Lee, S.; Park, S. B. *Chem. Commun.*, **2012**, *48*, 2331.
[52] Extensive polymerization was observed during the course of the reaction and the reactions turned dark brown.



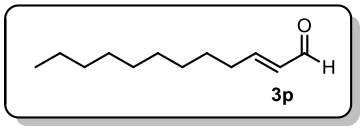
(2*E*,4*E*)-5-phenylpenta-2,4-dienal,[48] 3m (1.402 g, 89%) was prepared according to general cleavage protocol from **2m** (1.740 g, 0.010 mol) as a clear, yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 6.30 (dd, *J*=15.17, 7.96 Hz, 1 H) 6.95 - 7.12 (m, 2 H) 7.29 - 7.58 (m, 6 H) 9.65 (d, *J*=7.96 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 126.46 (CH) 127.81 (CH) 129.21 (CH) 129.96 (CH) 131.90 (CH) 135.85 (CH) 142.70 (CH) 152.30 (CH) 193.85 (C) **GC-MS** (EI) 158 ([M]$^+$, 56%) 129 (100%) 115 (43%) 102 (18%) 77 (21%) 63 (13%) 51 (22%) 39 (10%)



(*E*)-5-phenylpent-2-enal,[53] 3m (1.150 g, 72%) was prepared according to general cleavage protocol from **2m** (1.763 g, 0.010 mol) with the following modifications: 1) The reaction was conducted over 36 h 2) An additional 0.4 equiv of **1a** was added to the reaction in 0.2 equiv increments during the course of the 36 h. The aldehyde **3n** was obtained as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 2.66 - 2.75 (m, 2 H) 2.87 (t, *J*=7.80 Hz, 2 H) 6.16 (ddt, *J*=15.64, 7.85, 1.47, 1.47 Hz, 1 H) 6.88 (dt, *J*=15.63, 6.68 Hz, 1 H) 7.17 - 7.27 (m, 3 H) 7.30 - 7.37 (m, 2 H) 9.52 (d, *J*=7.88 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 34.23 (CH_2) 34.37 (CH_2) 126.53 (CH) 128.49 (CH) 128.75 (CH) 133.54 (CH) 140.45 (C) 157.44 (CH) 194.05 (C) **GC-MS** (EI) 160 ([M]$^+$, 1%) 116 (17%) 91 (100%) 65 (18%) 51 (7%) 39 (10%)



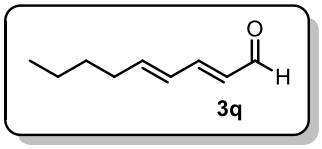
(*E*)-7-phenylhept-2-enal,[54] 3o (1.619 g, 86%) was prepared according to general cleavage protocol from **2o** (2.043 g, 0.010 mol) with the following modifications: 1) The reaction was conducted over 48 h 2) An additional 1.6 equiv of **1a** was added to the reaction in 0.2 equiv increments during the course of the 48 h. The aldehyde **3o** was obtained as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 1.53 - 1.64 (m, 2 H) 1.66 - 1.78 (m, 2 H) 2.39 (qd, *J*=7.18, 1.51 Hz, 2 H) 2.67 (t, *J*=7.52 Hz, 2 H) 6.14 (ddt, *J*=15.61, 7.88, 1.49, 1.49 Hz, 1 H) 6.85 (dt, *J*=15.59, 6.78 Hz, 1 H) 7.16 - 7.25 (m, 3 H) 7.28 - 7.36 (m, 2 H) 9.52 (d, *J*=7.88 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 27.67 (CH_2) 31.16 (CH_2) 32.85 (CH_2) 35.89 (CH_2) 126.14 (CH) 128.65 (2 × CH) 133.38 (CH) 142.29 (C) 158.73 (CH) 194.30 (C) **GC-MS** (EI) 188 ([M]$^+$, 3%) 170 (6%) 144 (9%) 129 (8%) 117 (18%) 104 (15%) 91 (100%) 77 (12%) 65 (23%) 41 (12%) 39 (18%)



(*E*)-dodec-2-enal,[55] 3p (1.334 g, 73%) was prepared according to general cleavage protocol from **2p** (1.984 g, 0.010 mol) with the following modifications: 1). The reaction was conducted over 48 h 2) An additional 1.0 equiv of **1a** was added to the reaction in 0.2 equiv increments during the course of the 48 h. The aldehyde **3p** was obtained as a clear, pale yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 0.87 (t, *J*=6.71 Hz, 3 H) 1.18 - 1.37 (m, 12 H) 1.50 (quin, *J*=7.19 Hz, 2 H) 2.32 (q, *J*=6.90 Hz, 2 H) 6.10 (ddt, *J*=15.52, 7.80, 1.50, 1.50 Hz, 1 H) 6.84 (dt, *J*=15.51, 6.84 Hz, 1 H) 9.49 (d, *J*=7.88 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 14.28 (CH_3) 22.86 (CH_2) 28.06 (CH_2) 29.35 (CH_2) 29.47 (CH_2) 29.56 (CH_2) 29.67 (CH_2) 32.06 (CH_2)

[53] Schmidt, A.; Hilt, G. *Org. Lett.*, **2013**, *15*, 2708.
[54] Das, B.; Veeranjaneyulu, B.; Balasubramanyam, P.; Srilatha, M. *Tetrahedron: Asymmetry*, **2010**, *21*, 2762.
[55] Patterson, J. W.; Pfister, J. R.; Wagner, P. J.; Murthy, D. V. K. *J. Org. Chem.*, **1983**, *48*, 2572.

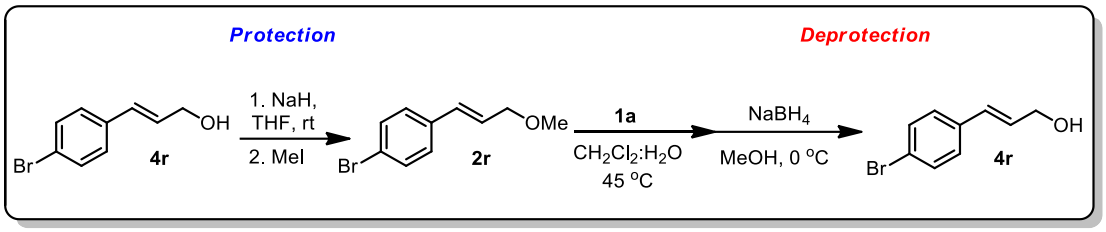
32.93 (CH_2) 133.19 (CH) 159.11 (CH) 194.21 (C) **GC-MS** (EI) 182 ($[M]^+$, 0.1%) 138 (3%) 135 (6%) 121 (9%) 111 (8%) 97 (19%) 83 (42%) 79 (14%) 70 (56%) 67 (24%) 55 (62%)43 (60%) 41 (100%) 39 (44%)



(2*E*,4*E*)-nona-2,4-dienal,[56] 3q (1.151 g, 83%) was prepared according to general cleavage protocol from **2q** (1.542 g, 0.010 mol) as a clear, yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 0.91 (t, *J*=7.18 Hz, 3 H) 1.35 (quin, *J*=7.30 Hz, 2 H) 1.44 (quin, *J*=7.80 Hz, 2 H) 2.21 (q, *J*=6.93 Hz, 2 H) 6.06 (dd, *J*=15.28, 7.95 Hz, 1 H) 6.20 - 6.38 (m, 2 H) 7.07 (dd, *J*=15.25, 9.63 Hz, 1 H) 9.52 (d, *J*=7.95 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 14.03 (CH_3) 22.44 (CH_2) 30.87 (CH_2) 33.07 (CH_2) 128.84 (CH) 130.22 (CH) 147.53 (CH) 153.02 (CH) 194.04 (C) **GC-MS** (EI) 138 ($[M]^+$, 7%) 95 (8%) 81 (100%) 67 (22%) 65 (11%) 54 (10%) 41 (30%) 39 (26%)

Protection/Deprotection Sequence Involving Oxidative Cleavage

[56] Dabdoub, M. J.; Dabdoub V. B.; Baroni, A. C. M.; Hurtado, G. R.; Barbosa, S. L. *Tetrahedron Lett.*, **2010**, *51*, 1666.



***Protection:* Synthesis of (E)-1-bromo-4-(3-methoxyprop-1-en-1-yl)benzene[57] (2r)**

To a flame-dried 250 mL round bottom flask equipped with a stir bar was added NaH[58] (1.55 g, 0.0648 mol, 2 equiv) followed by anhydrous THF (81 mL). The flask was sealed with a rubber septum and placed under an N_2 atmosphere *via* an N_2 inlet needle. The alcohol **4r** (6.874 g, 0.0324 mol, 1 equiv) dissolved in a minimum amount of THF (≈ 9 mL) was added to the flask dropwise over ten minutes. **CAUTION!** *Mildly exothermic, evolves H_2 gas!* The reaction mixture was allowed to stir at room temperature for 1.5 h during which time the solution transitioned from a pale yellow to an orangish-red. After this time, MeI (13.79 g, 0.972 mol, 3 equiv) was added to the flask and the solution was allowed to stir for six hours.[59] The reaction mixture was then *carefully* quenched with deionized water (≈ 20 mL) and transferred to a separatory funnel. The mixture was diluted with deionized water (150 mL) and Et_2O (150 mL) and the layers were separated. The aqueous layer was extracted with Et_2O (3 × 75 mL) and the combined organic layers were washed with deionized water (2 × 100 mL) followed by brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude ether. Further purification was accomplished by SiO_2 plug (95:5 to 9:1 Hex:EtOAc) to give the pure ether **2a** (6.381 g, 87%) as a clear, pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 3.42 (s, 3 H) 4.10 (dd, *J*=5.84, 1.36 Hz, 2 H) 6.29 (dt, *J*=15.95, 5.84 Hz, 1 H) 6.57 (d, *J*=15.96 Hz, 1 H) 7.27 (d, *J*=9.05 Hz, 2 H) 7.46 (dt, *J*=8.42, 1.90 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 58.43 (CH_3) 73.17 (CH_2) 121.71 (C) 127.14 (CH) 128.27 (CH) 131.32 (CH) 131.96 (CH) 135.98 (C)
GC-MS (EI) 228 ([M]$^+$, ^{81}Br, 20%) 226 ([M]$^+$, ^{79}Br, 20%) 147 (86%) 131 (14%) 115 (100%) 104 (35%) 91 (10%) 89 (15%) 77 (15%) 75 (10%) 63 (14%) 39 (6%)

***Deprotection:* Synthesis of (E)-3-(4-bromophenyl)prop-2-en-1-ol[60] (4r)**

Stage One: Oxidative Cleavage

To a 100 mL round bottom flask equipped with a stir bar was added the ether **2r** (2.271 g, 0.010 mol, 1 equiv). CH_2Cl_2 (40 mL), and deionized water (10 mL). After stirring for five minutes, the oxoammonium salt **1a** (6.302 g, 0.021 mol, 2.1 equiv)was added to the flask and the flask was equipped with a reflux condenser. The flask was heated to 45 °C and allowed to stir overnight. The reaction mixture gradually became orange during this time. Reaction progress was monitored by GC/MS and/or ^{1}H NMR. After 24 h the reaction was judged to be incomplete and additional 1a (0.600 g, 0.002 mol 0.2 equiv) was added and the reaction was heated to 55°C. After an additional

[57] Bolte, B.; Odabachian, Y.; Gagosz, F. *J. Am. Chem. Soc*., **2010** , *132*, 7294.
[58] NaH was pre-washed with pentane five times prior to use to remove the mineral oil. The now whitish solid was dried under light vacuum.
[59] The alkylation process can be dramatically accelerated by heating to 40-45 °C without any detrimental effects
[60] Vyas, D. J.; Oestreich, M. *Chem. Commun.*, **2010**, *46*, 568.

12 h, the reaction was judged to be complete. At this time the reaction mixture was cooled to room temperature and transferred to a separatory funnel. The mixture was diluted with deionized water (100 mL) and Et_2O (150 mL) the layers were separated. The aqueous layer was extracted with Et_2O (5 × 50 mL). The combined organic layers were washed with deionized water (2 × 100 mL) and brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude aldehyde which was used directly in the next step.

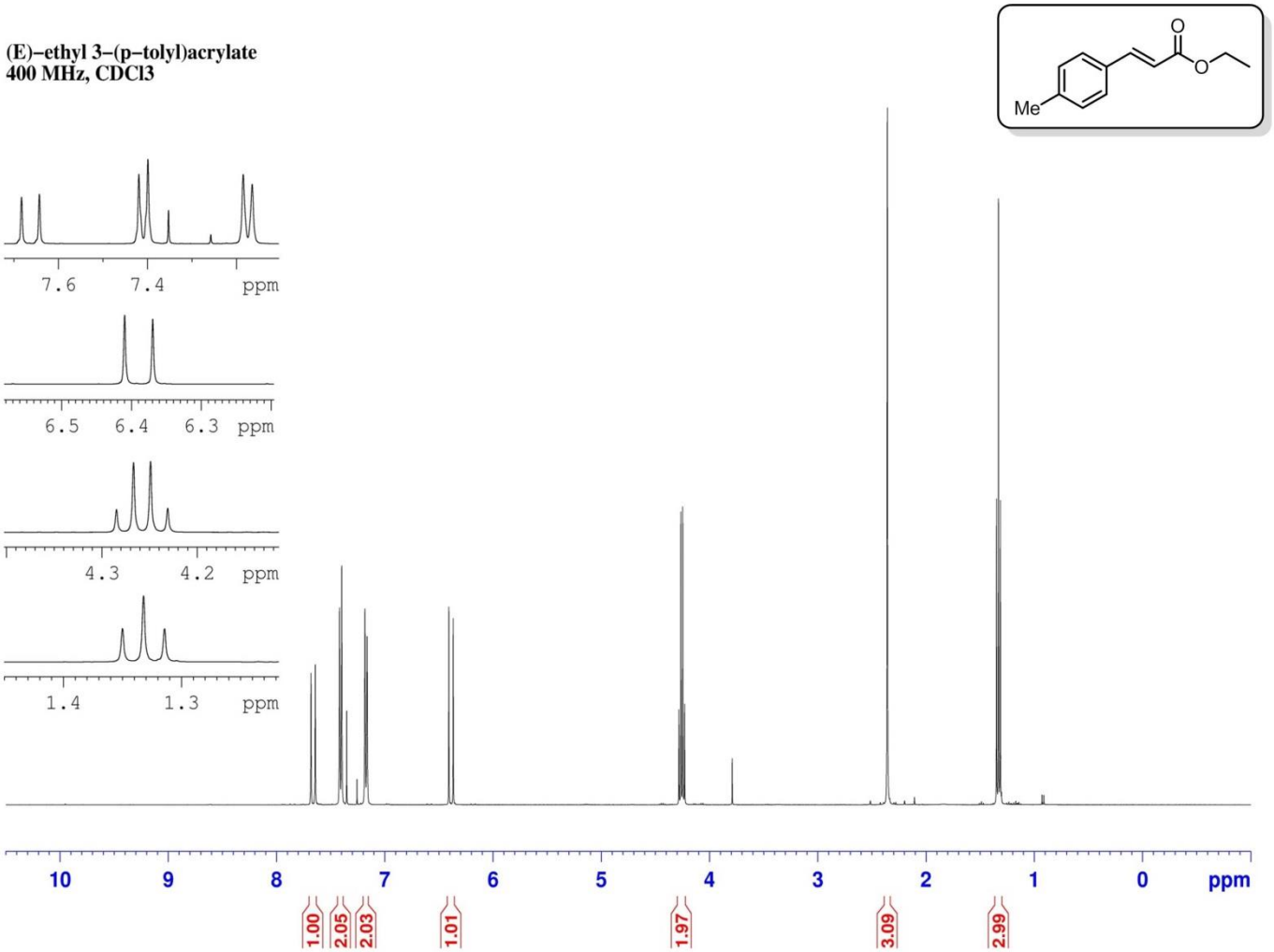
Stage Two: Reduction

The crude aldehyde **3r** was dissolved in methanol (20 mL) and transferred to a 100 mL round bottom flask equipped with a stir bar. The flask was cooled to 0 °C *via* an ice-water bath. After stirring at 0 °C for 10 minutes, $NaBH_4$ (0.757 g, 0.020 mol, 2 equiv[61]) was added slowly portion-wise to the flask. **CAUTION!** *Exothermic, evolves H_2 gas*. The reaction mixture was allowed to stir at 0 °C for 30 minutes after complete addition of $NaBH_4$. The flask was then warmed to room temperature and allowed to stir for 90 minutes. After this time, the reaction was *carefully* quenched with deionized H_2O (30 mL). The quenched reaction mixture was transferred to a separatory funnel and diluted with deionized water (50 mL) and Et_2O (100 mL). The layers were separated and the aqueous layer was extracted with Et_2O (3 × 50 mL). The combined organic layers were washed with deionized H_2O (150 mL) and brine (150 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo* by rotary evaporation to give the crude alcohol. Further purification was accomplished by SiO_2 plug. The crude alcohol was adhered to silica using 1-2 weight equivalents (relative to theoretical yield) silica and DCM. The DCM was removed *in vacuo* to provide the adhered alcohol. A plug of silica was then assembled. This was done by adding 3-4 weight equivalents of silica (again relative to the theoretical yield) to a 150 ml coarse-porosity fritted glass funnel. An appropriately sized piece of filter paper relative to the size of the funnel was placed on top of the dry silica gel layer. The dry packed material was gently added evenly over the filter paper. Another piece of appropriately sized filter paper was added over this layer. The plug was first washed with 400 ml of a 95:5 by volume mixture of Hex:EtOAc. This layer was discarded and the desired alcohol was eluted off using 8:2 Hex: EtOAc (2 column volumes) followed by 7:3 Hex:EtOAc (3 column volumes). The solvent was removed *in vacuo* by rotary evaporation to afford the pure alcohol (1.864 g, 88%) as a powdery white solid.

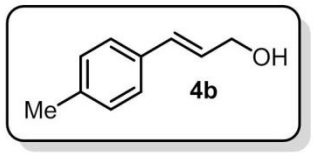
^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm 1.51 (t, *J*=5.86 Hz, 1 H) 4.34 (td, *J*=5.72, 1.51 Hz, 2 H) 6.38 (dt, *J*=15.86, 5.50 Hz, 1 H) 6.59 (d, *J*=15.91 Hz, 1 H) 7.27 (d, *J*=9.24 Hz, 2 H) 7.46 (dt, *J*=8.61, 2.50 Hz, 2 H)
^{13}C NMR ($CDCl_3$, 100 MHz) δ ppm 63.80 (CH_2) 121.73 (C) 128.26 (CH) 129.61 (CH) 130.07 (CH) 131.99 (CH) 135.92 (C)
GC-MS (EI) ($[M]^+$, %) 214 ($[M]^+$, ^{81}Br, 42%) 212 ($[M]^+$, ^{79}Br, 42%) 171 (^{81}Br, 49%) 169 (^{79}Br, 49%) 158 (^{81}Br, 24%) 156 (^{79}Br, 25%) 133 (100%) 115 (86%) 103 (54%) 91 (78%) 77 (76%) 65 (13%) 63 (38%) 55 (43%) 51 (46%) 39 (21%)

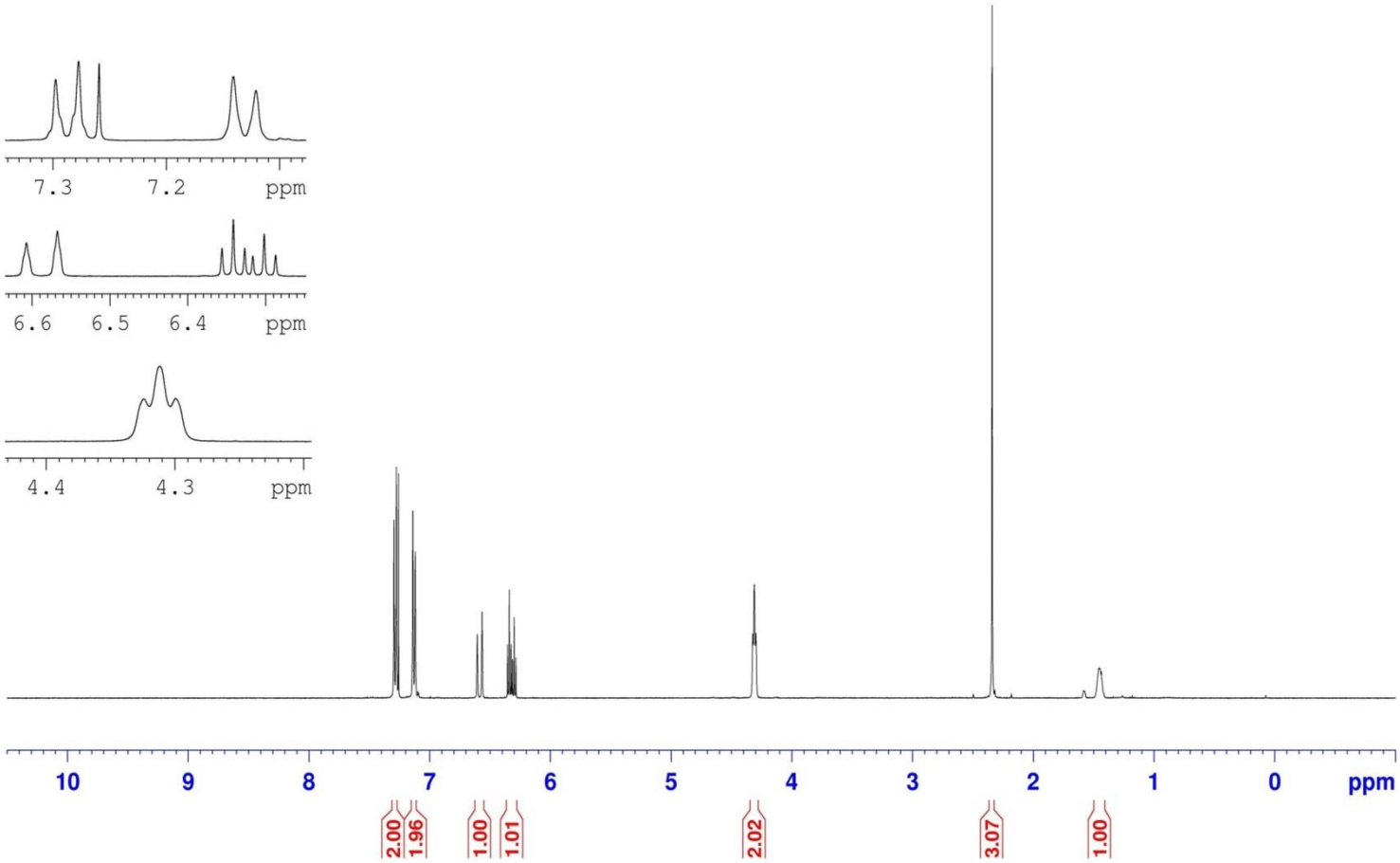
[61] Assumes 100% yield from *Stage One*

^{1}H NMR Spectra of Synthesized Compounds

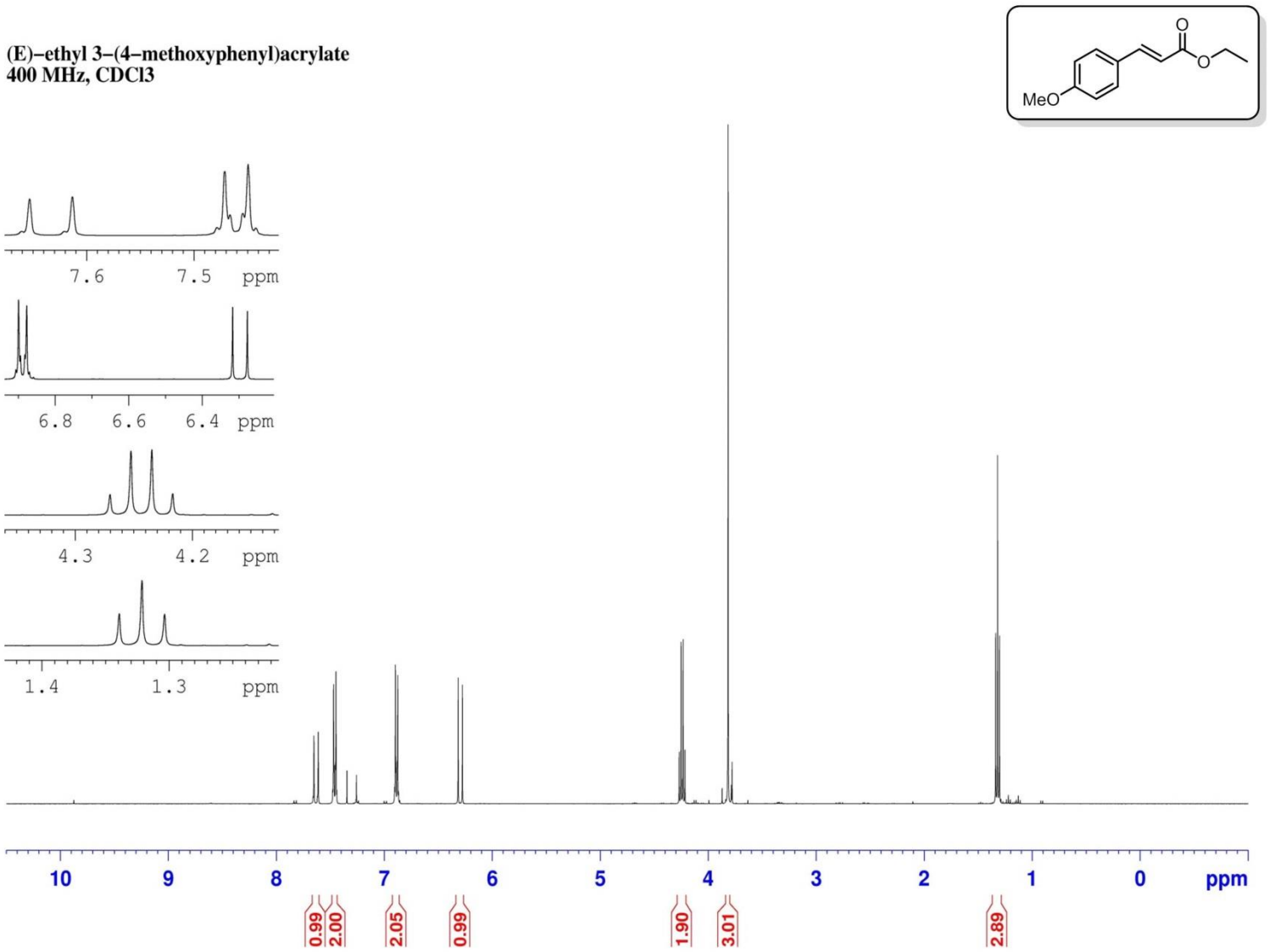


(E)−3−(p−tolyl)prop−2−en−1−ol
400 MHz, CDCl3

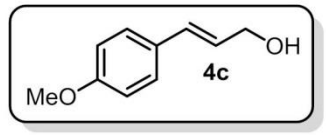


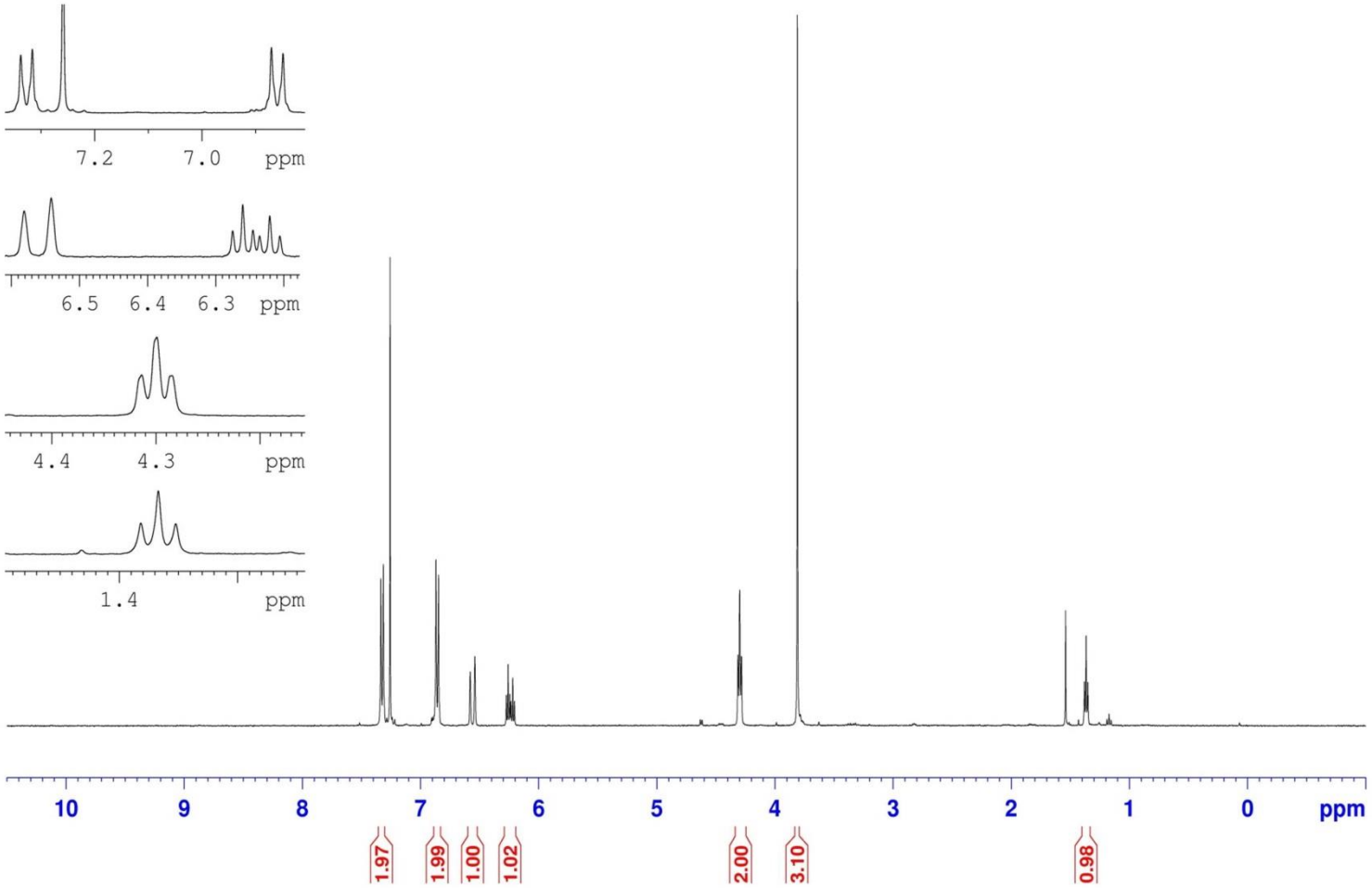


(E)−ethyl 3−(4−methoxyphenyl)acrylate
400 MHz, CDCl3

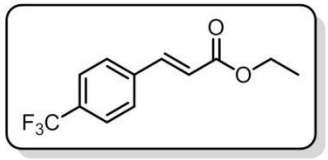


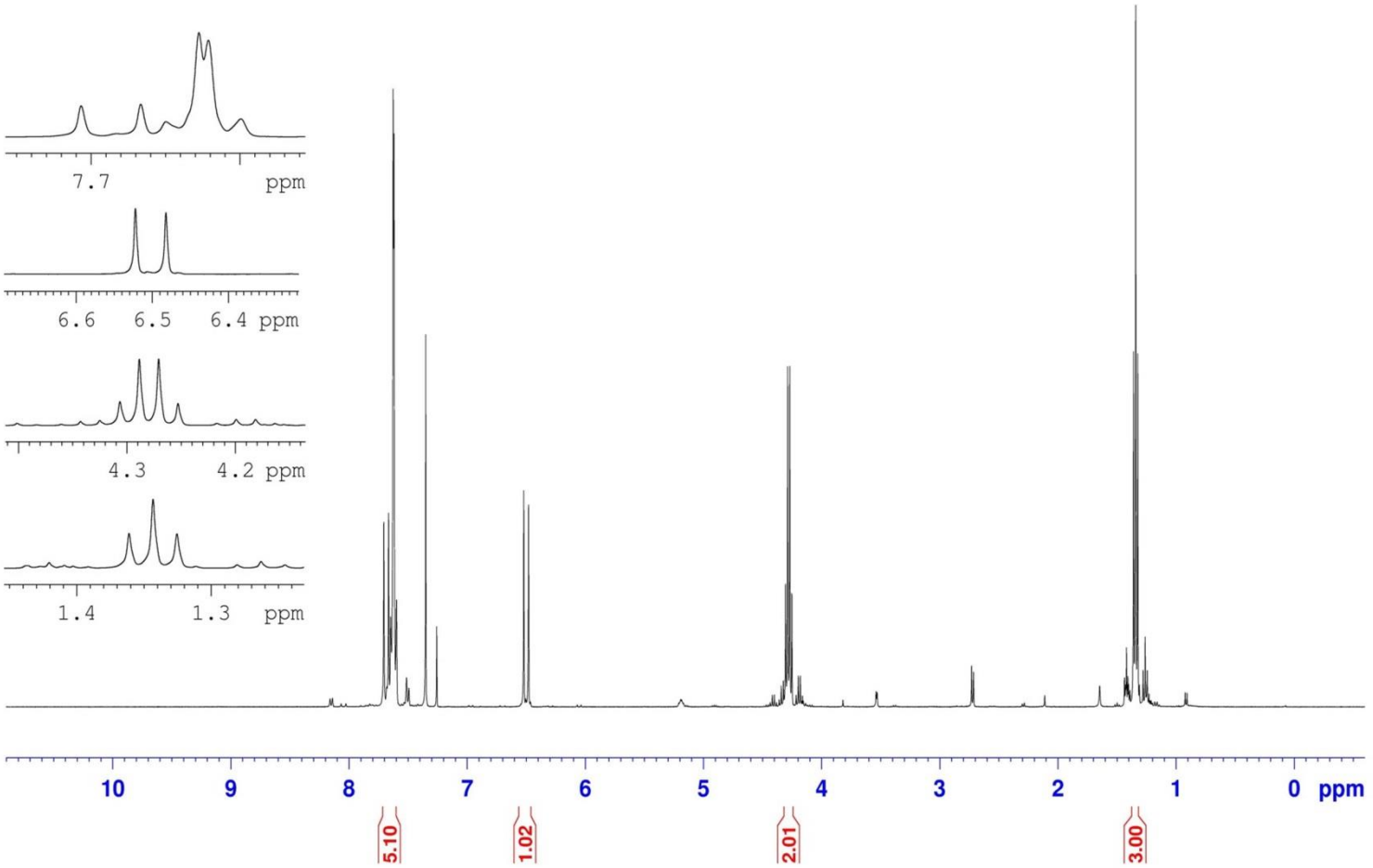
(E)−3−(4−methoxyphenyl)prop−2−en−1−ol
400 MHz, CDCl3



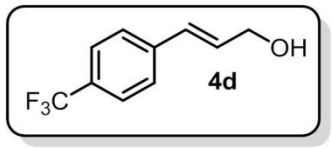


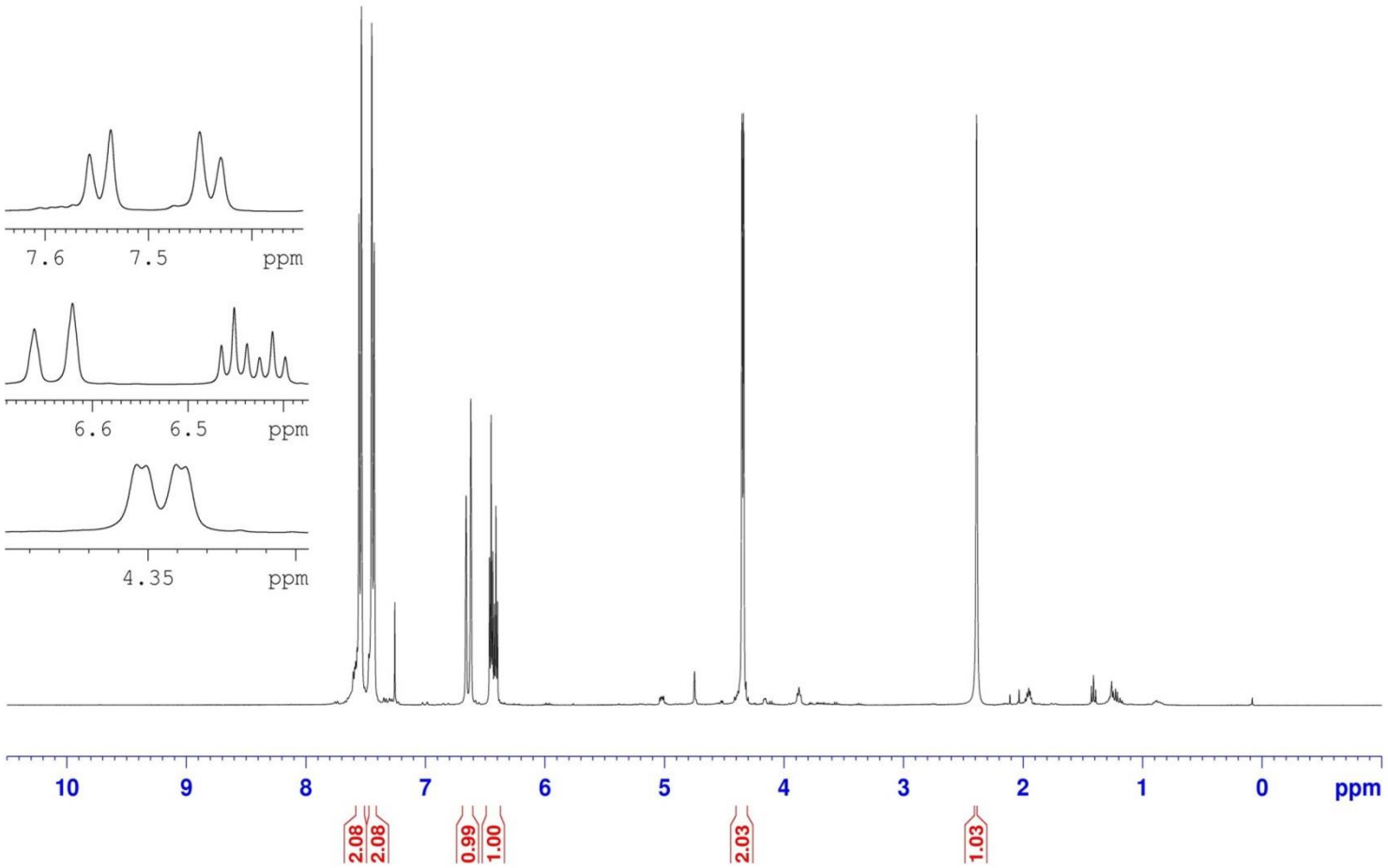
(E)−ethyl 3−(4−(trifluoromethyl)phenyl)acrylate
400 MHz, CDCl3



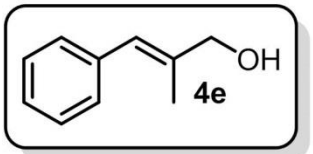


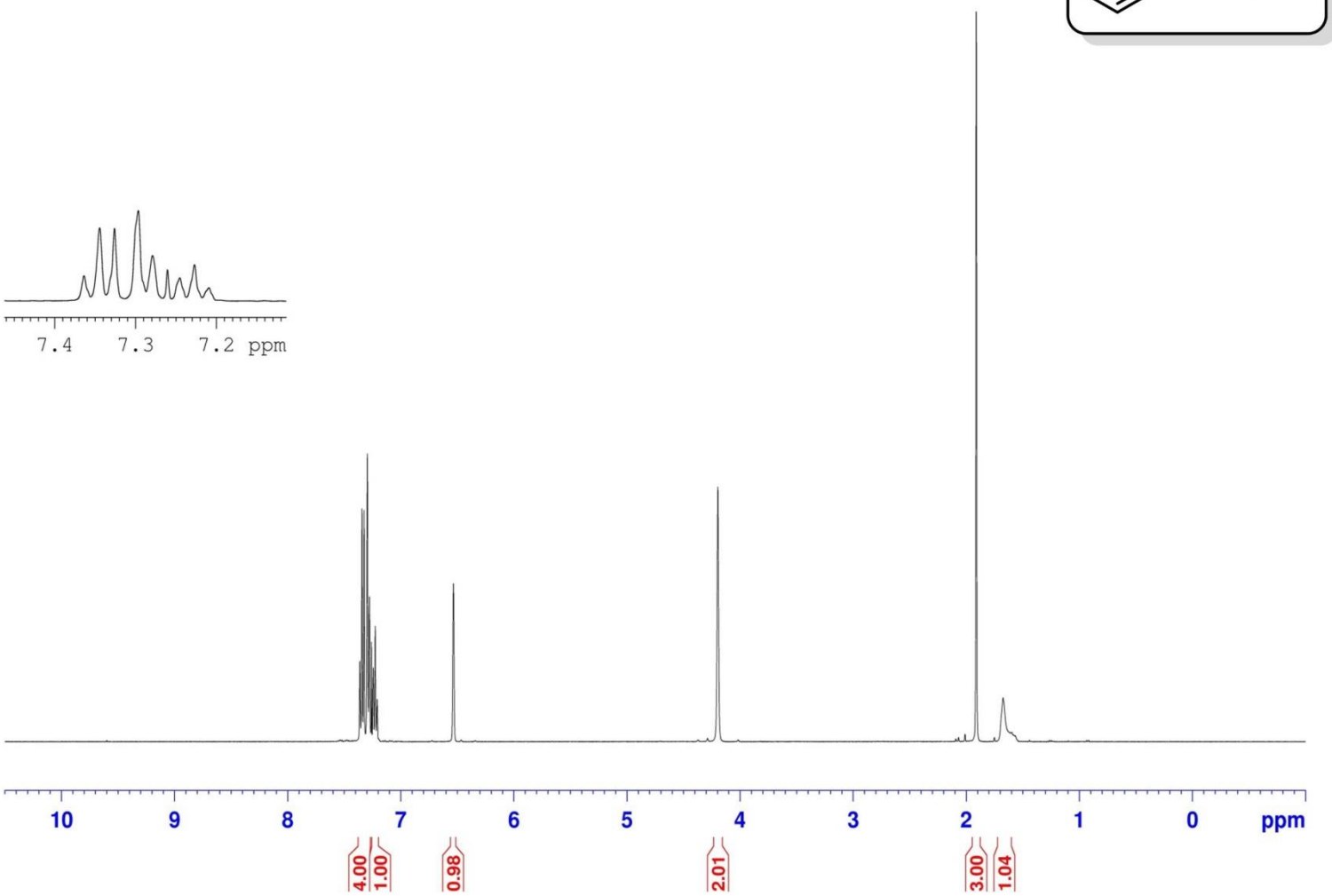
(E)−3−(4−(trifluoromethyl)phenyl)prop−2−en−1−ol
400 MHz, CDCl3



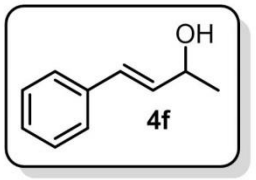


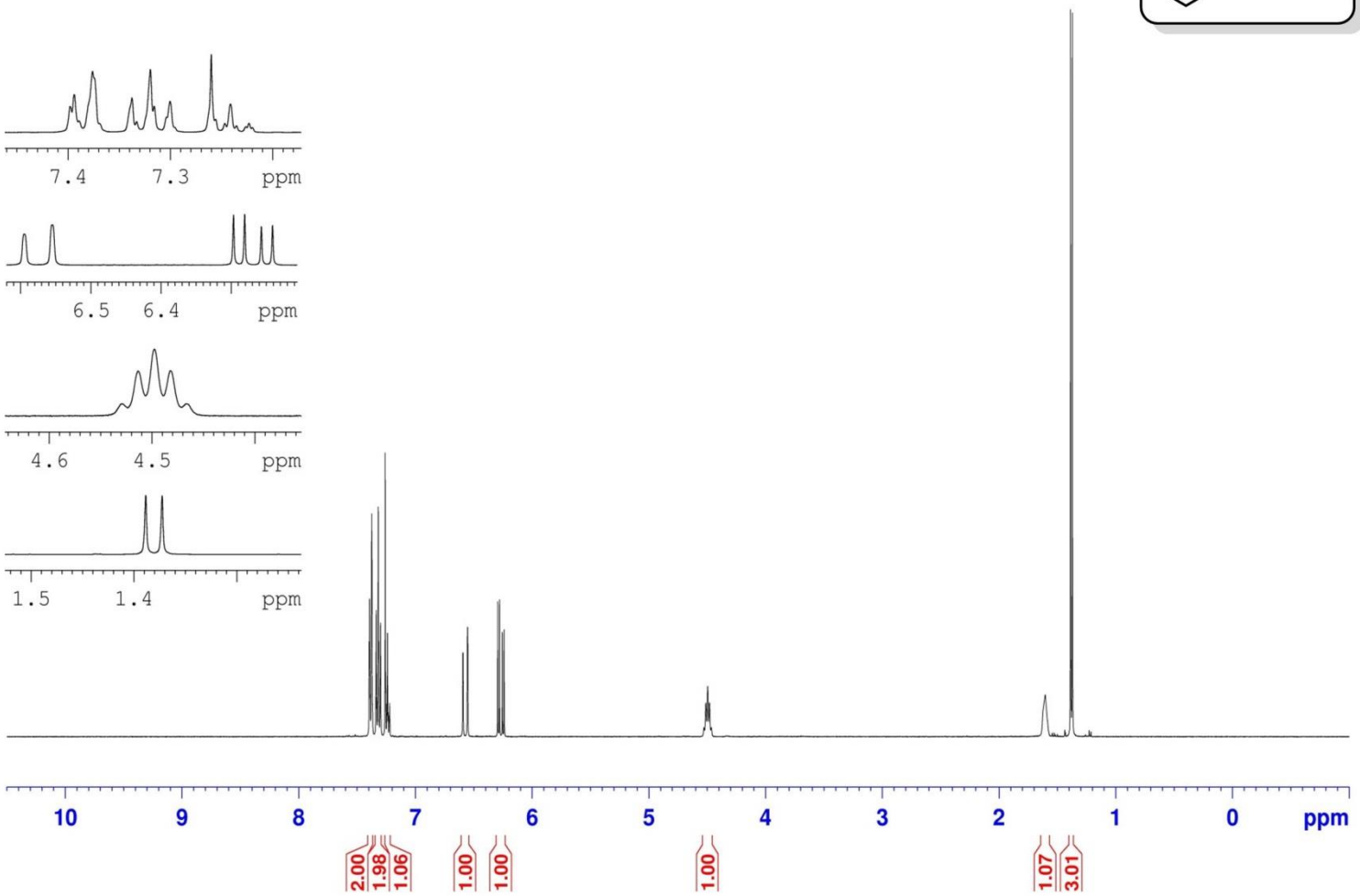
(E)−2−methyl−3−phenylprop−2−en−1−ol
400 MHz, CDCl3



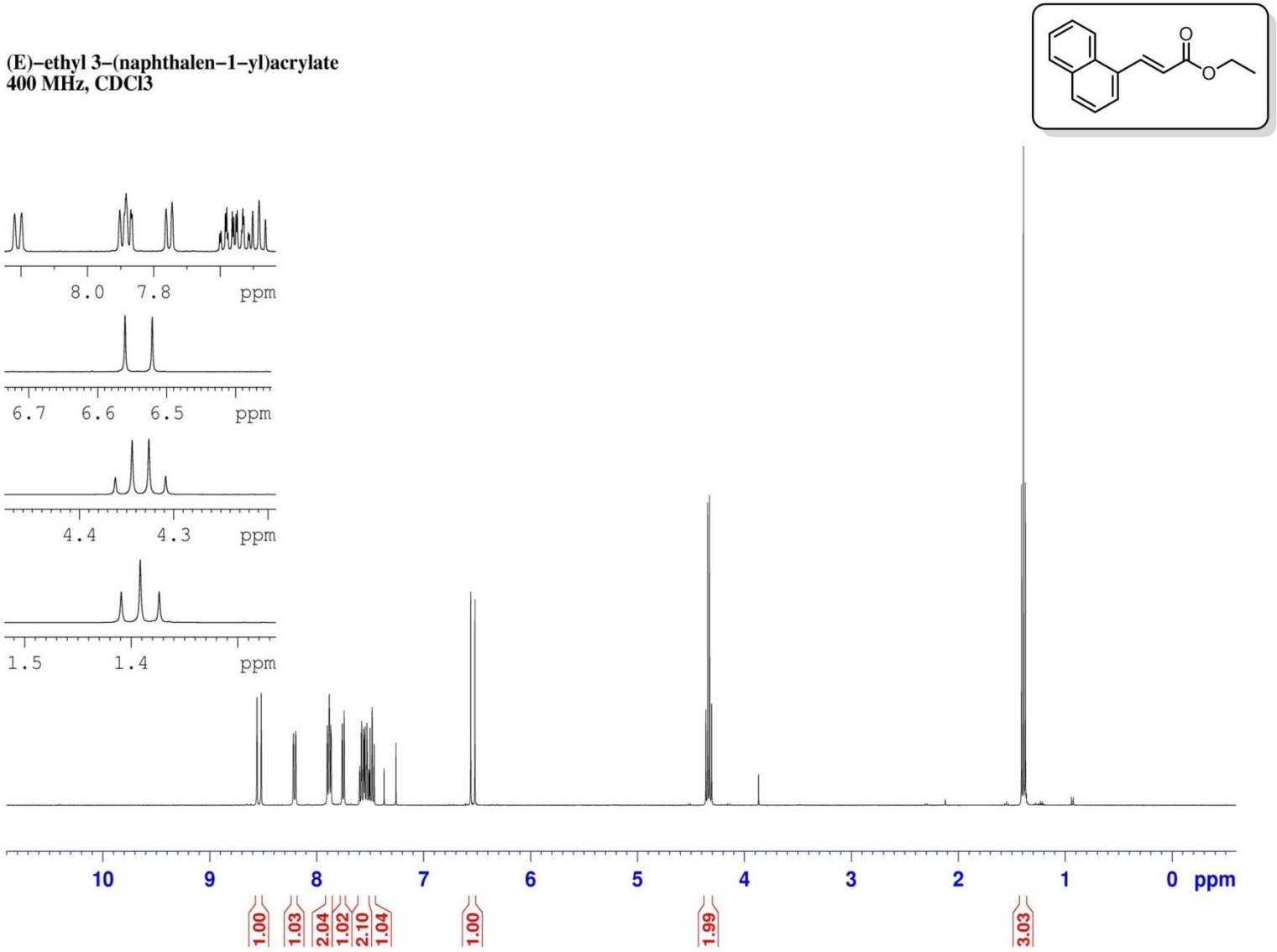


(E)–4–phenylbut–3–en–2–ol
400 MHz, CDCl3

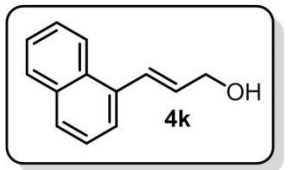


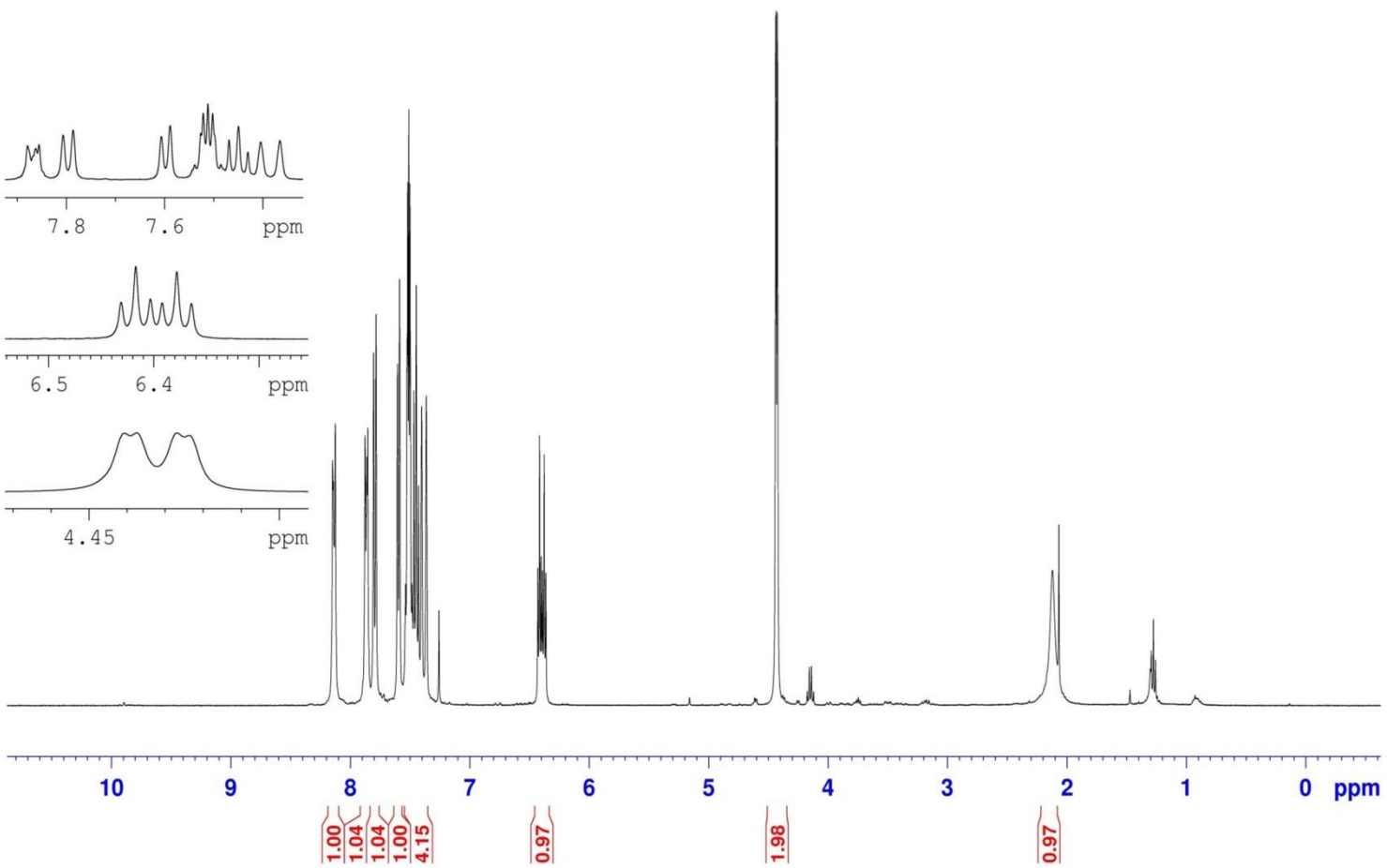


(E)–ethyl 3–(naphthalen–1–yl)acrylate
400 MHz, CDCl3

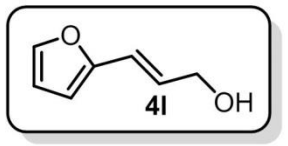


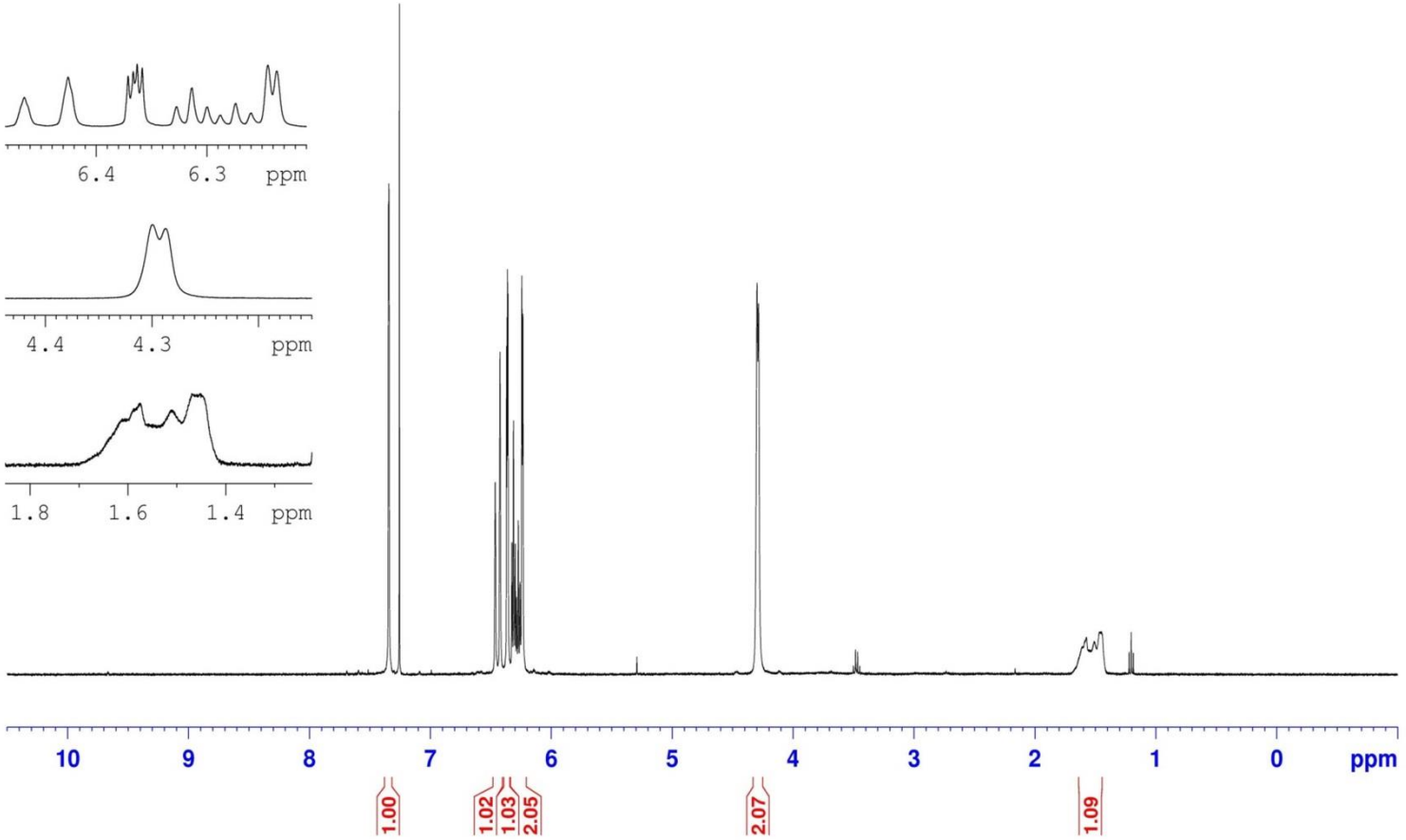
(E)−3−(naphthalen−1−yl)prop−2−en−1−ol
400 MHz, CDCl3



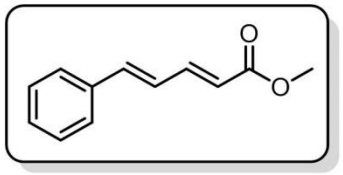


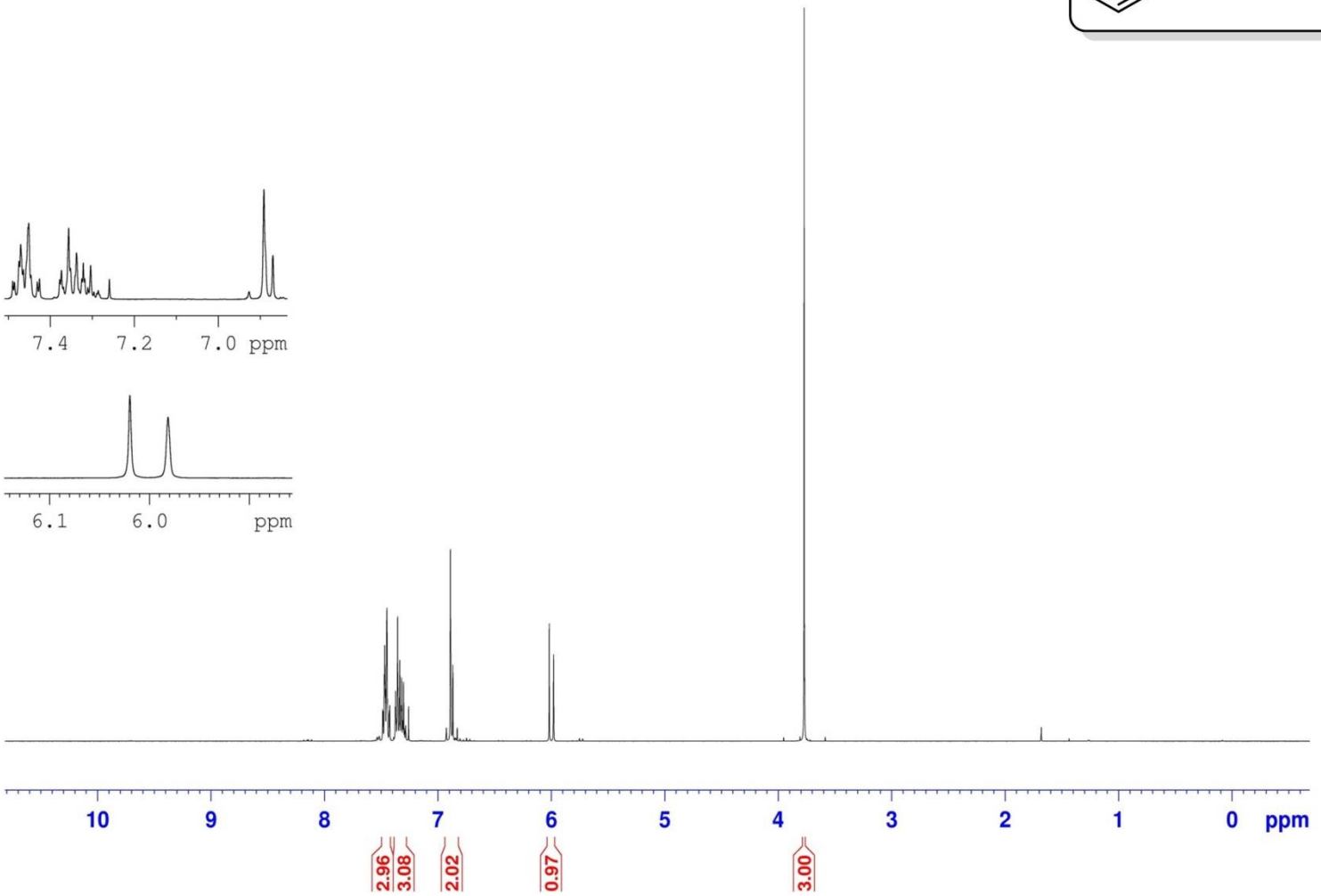
(E)−3−(furan−2−yl)prop−2−en−1−ol
400 MHz, CDCl3



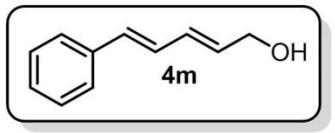


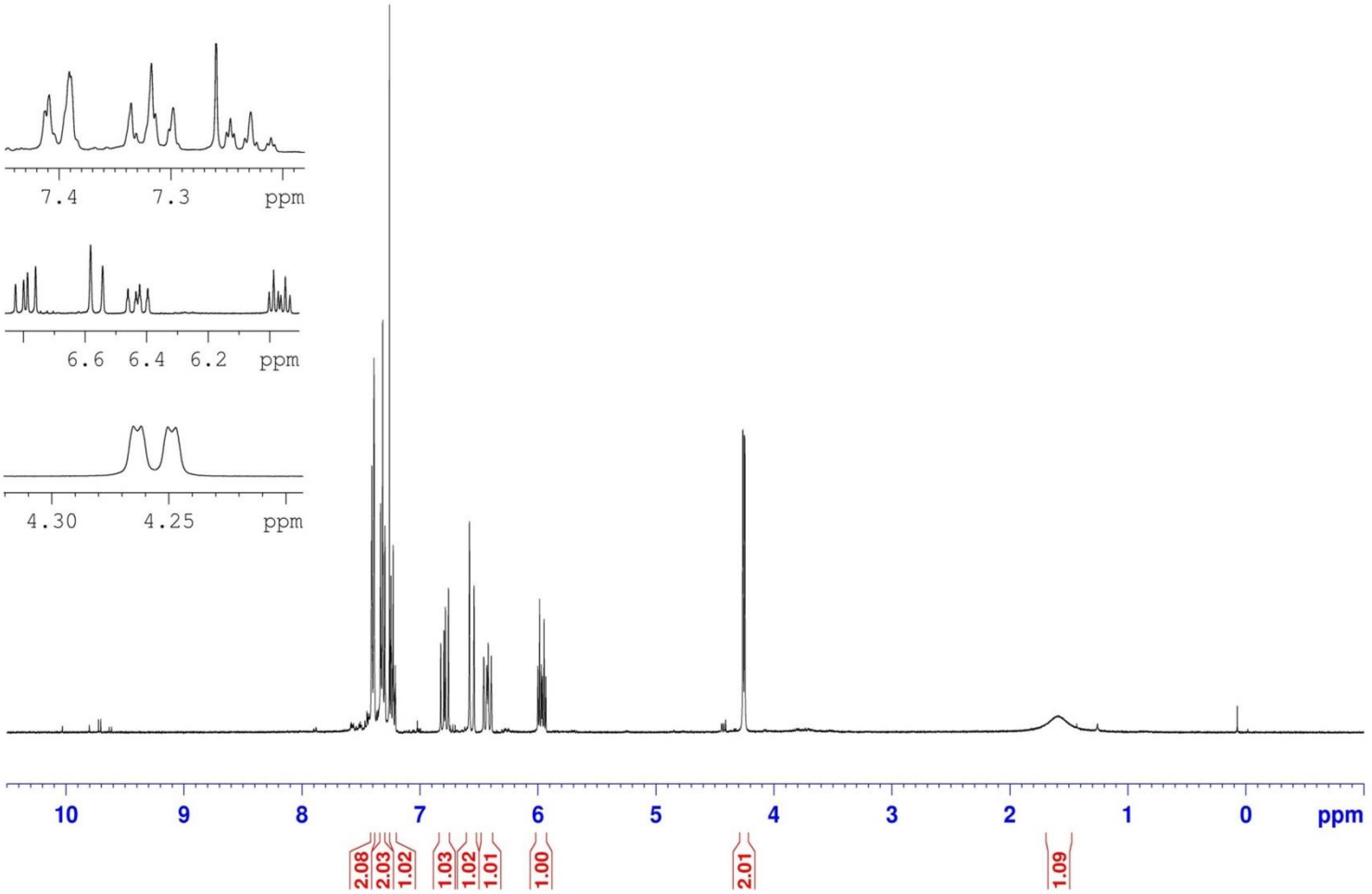
(2E,4E)–methyl 5–phenylpenta–2,4–dienoate
400 MHz, CDCl3



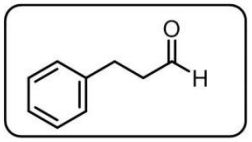


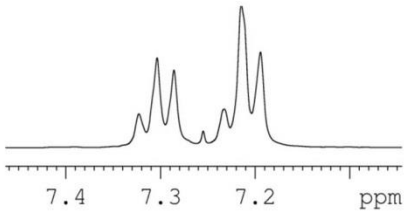
(2E,4E)−5−phenylpenta−2,4−dien−1−ol
400 MHz, CDCl3

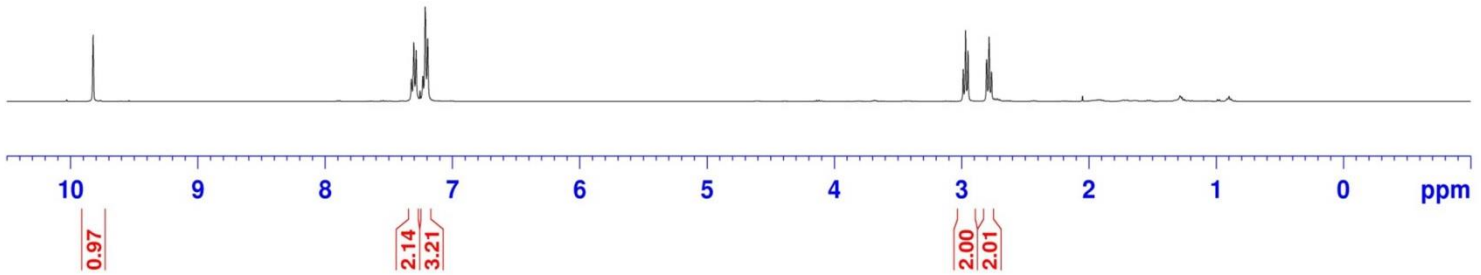




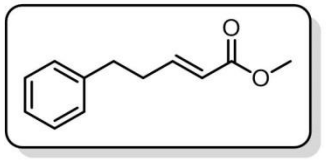
3–phenylpropanal
400 MHz, CDCl3

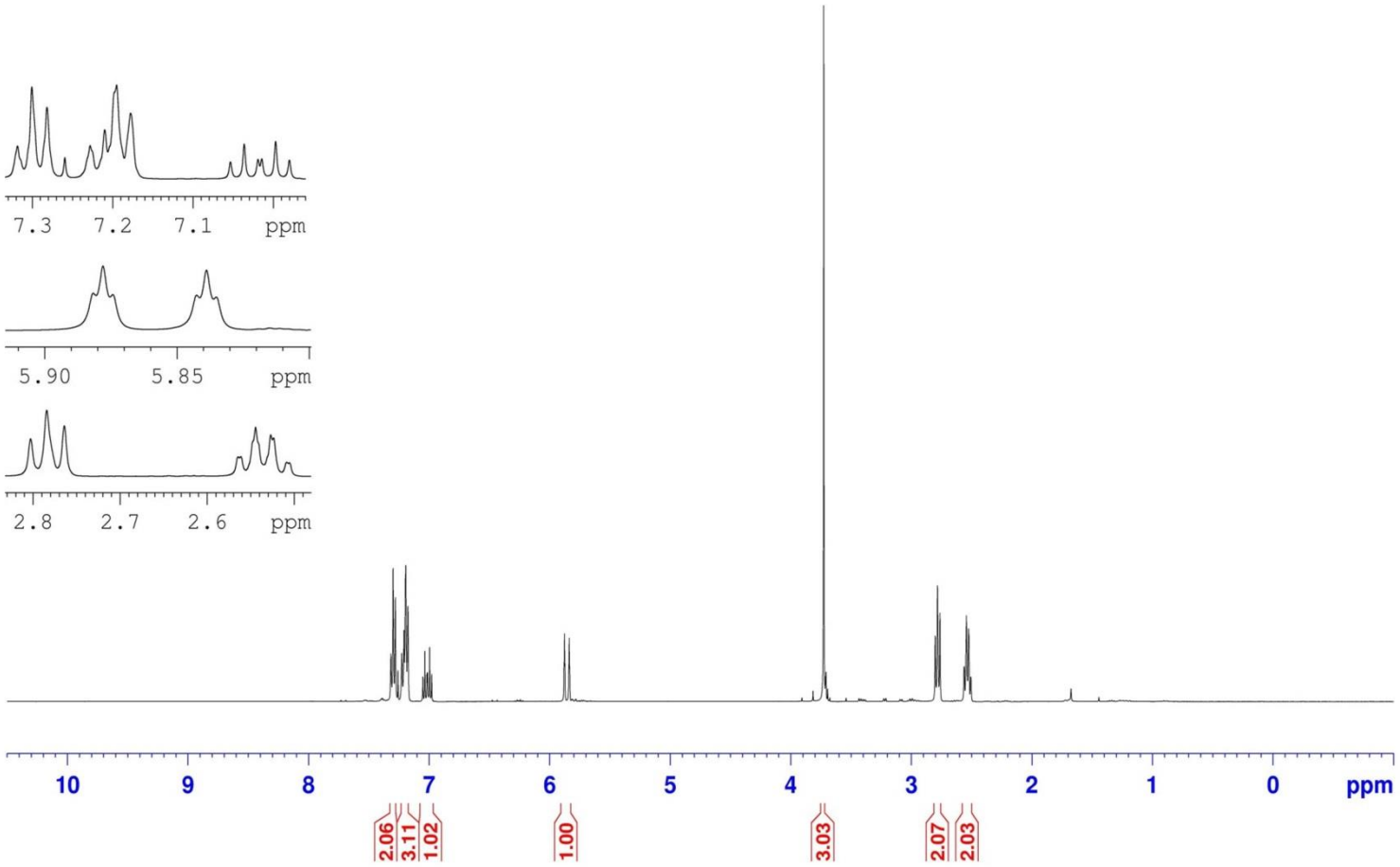




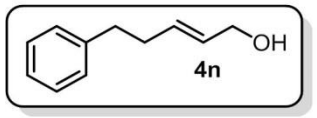


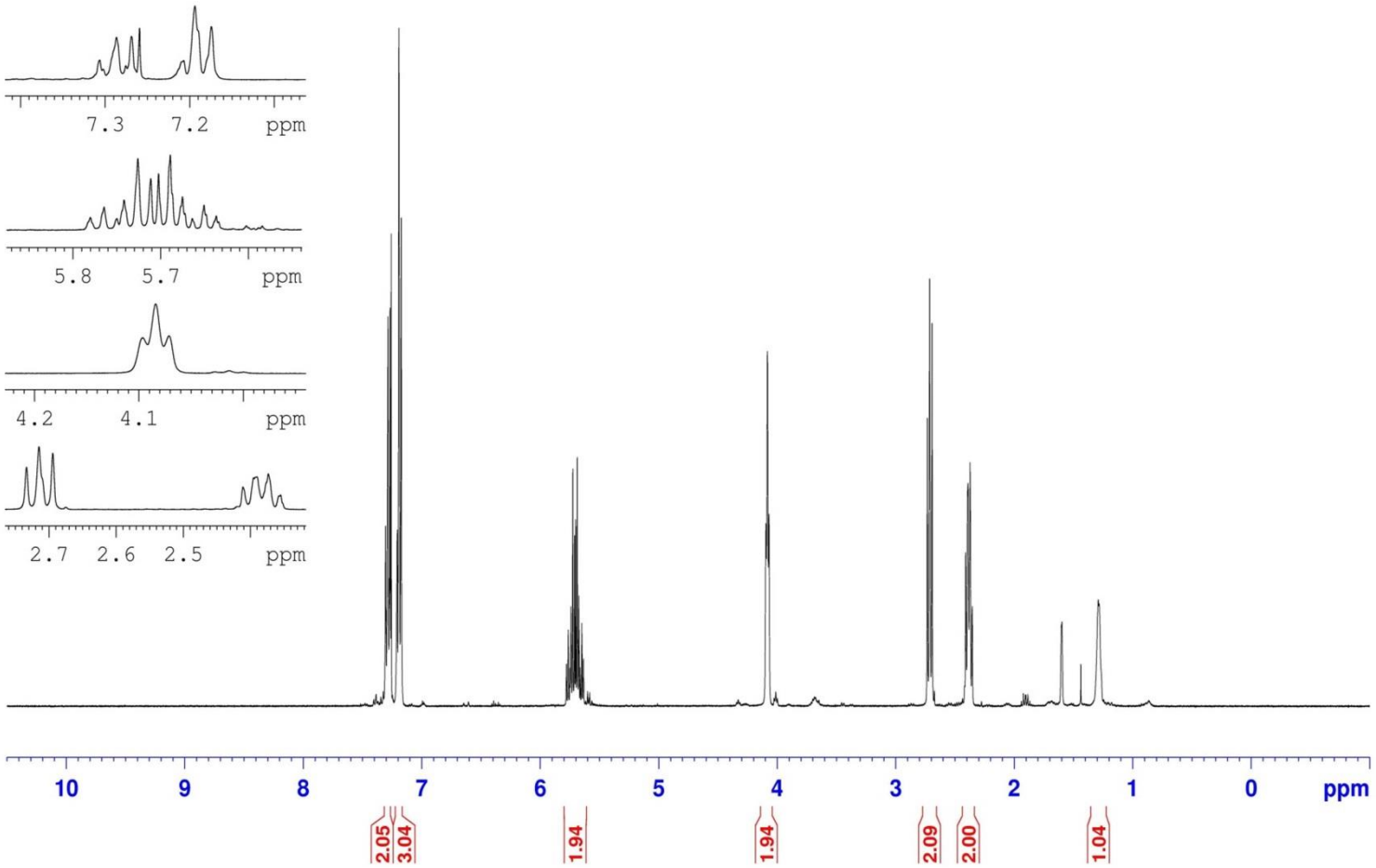
(E)−methyl 5−phenylpent−2−enoate
400 MHz, CDCl3



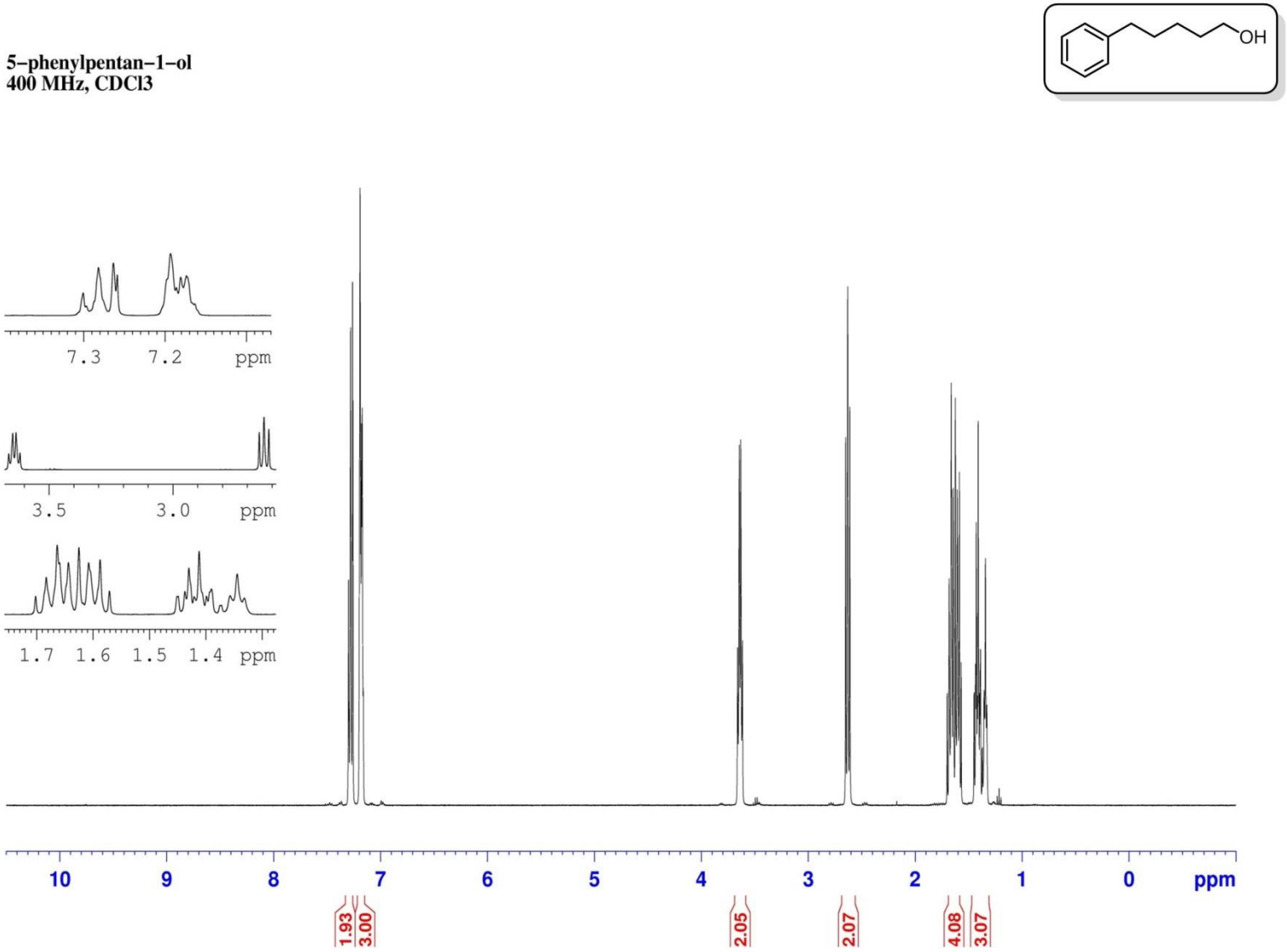


(E)−5−phenylpent−2−en−1−ol
400 MHz, CDCl3

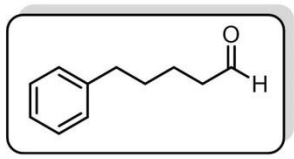


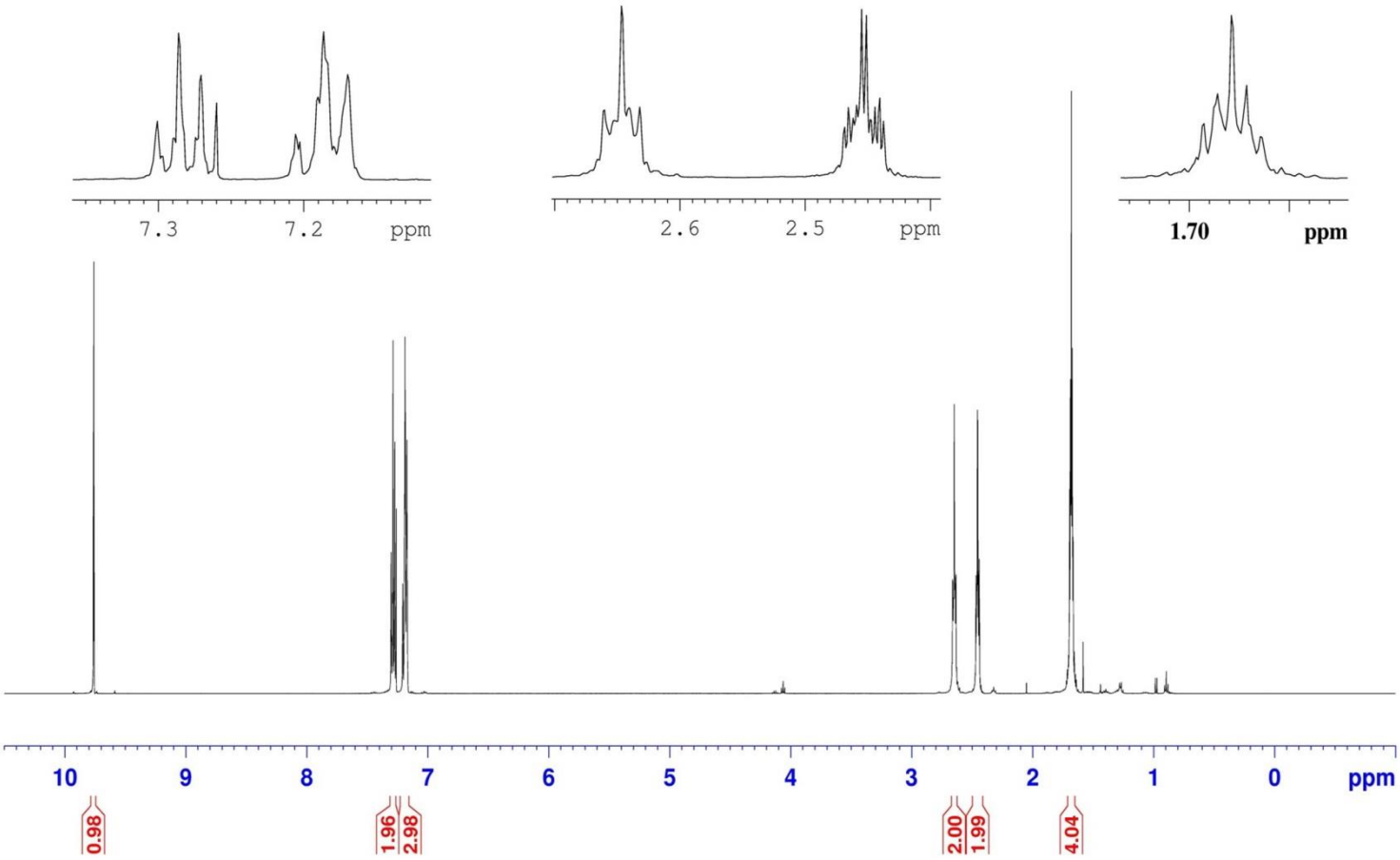


5–phenylpentan–1–ol
400 MHz, CDCl3

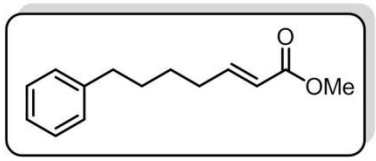


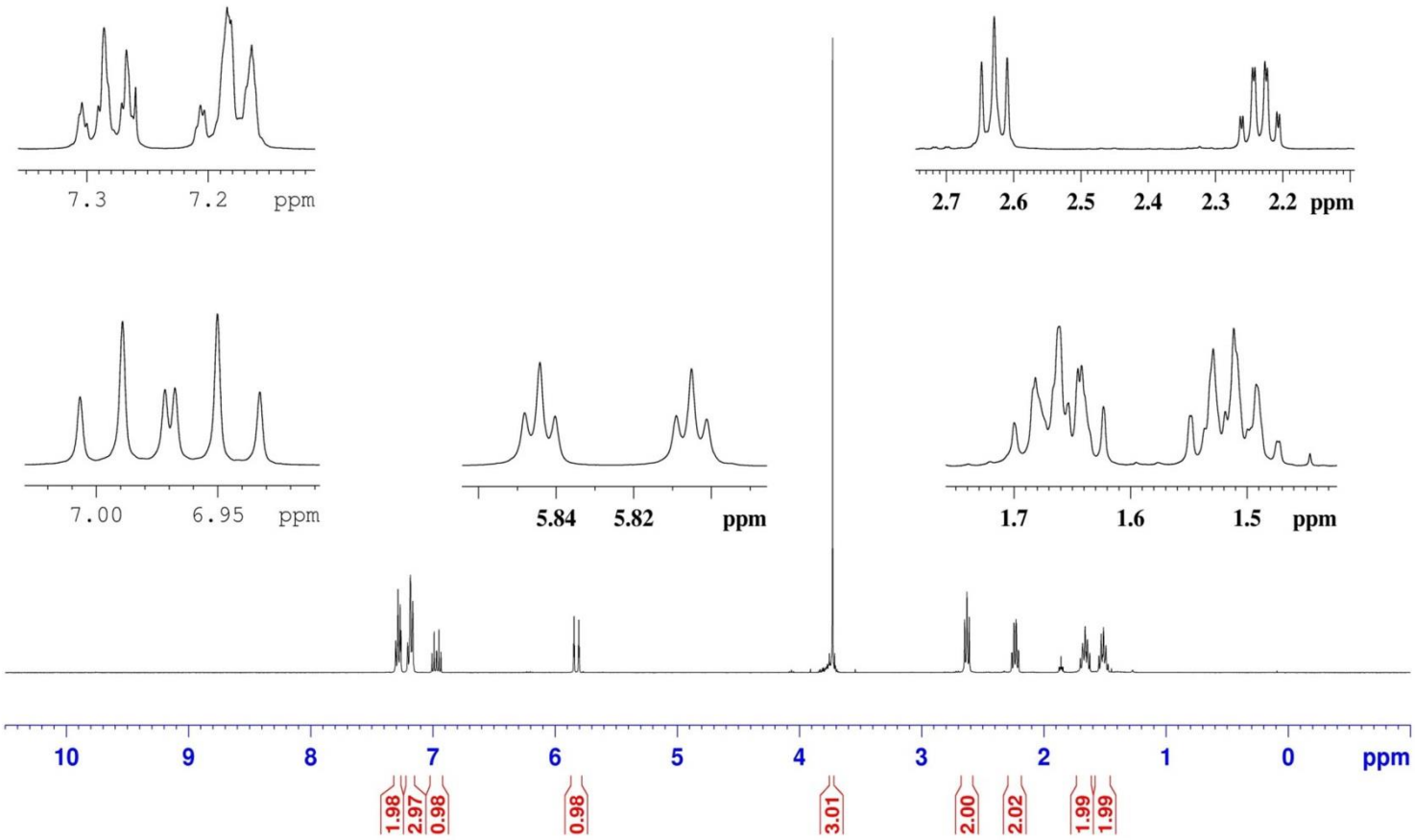
5–phenylpentanal
CDCl3, 400 MHz



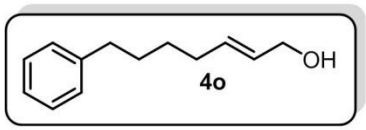


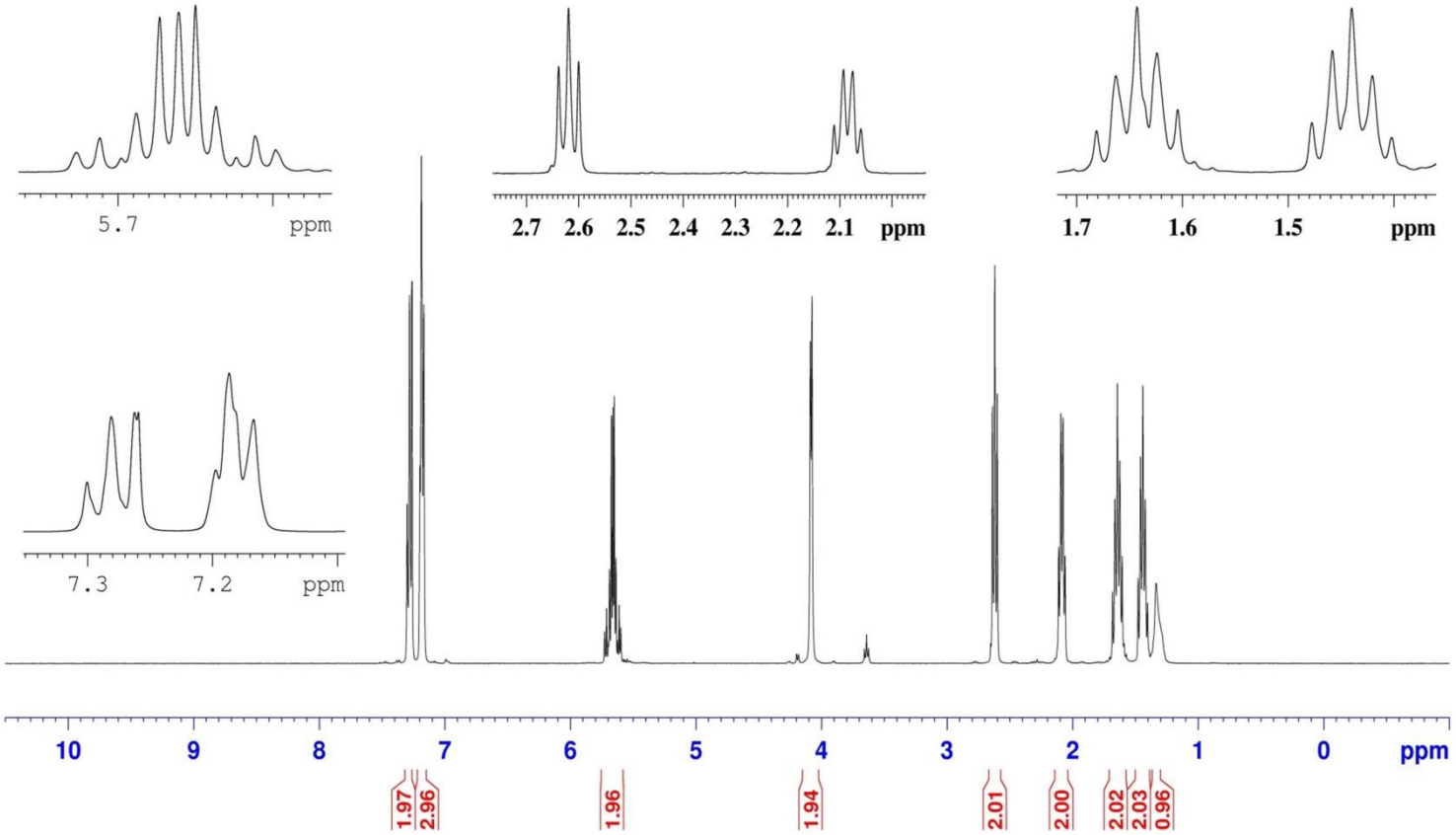
methyl 7–phenylhept–2–enoate
CDCl3, 400 MHz



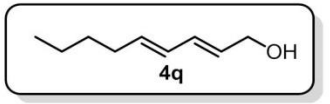


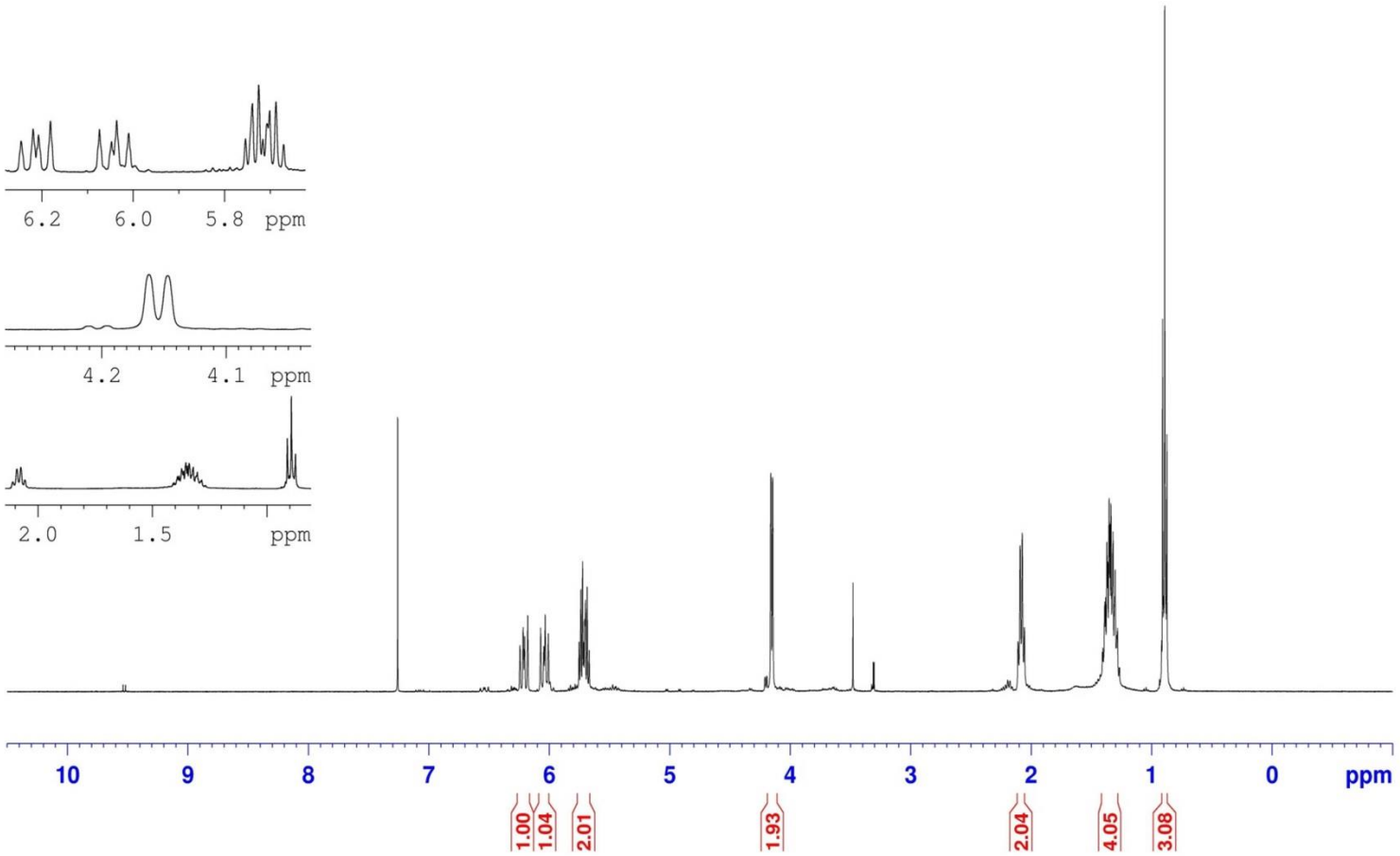
7–phenylhept–2–en–1–ol
CDCl3, 400 MHz



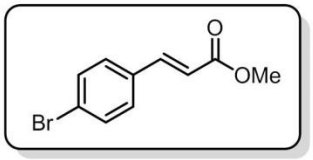


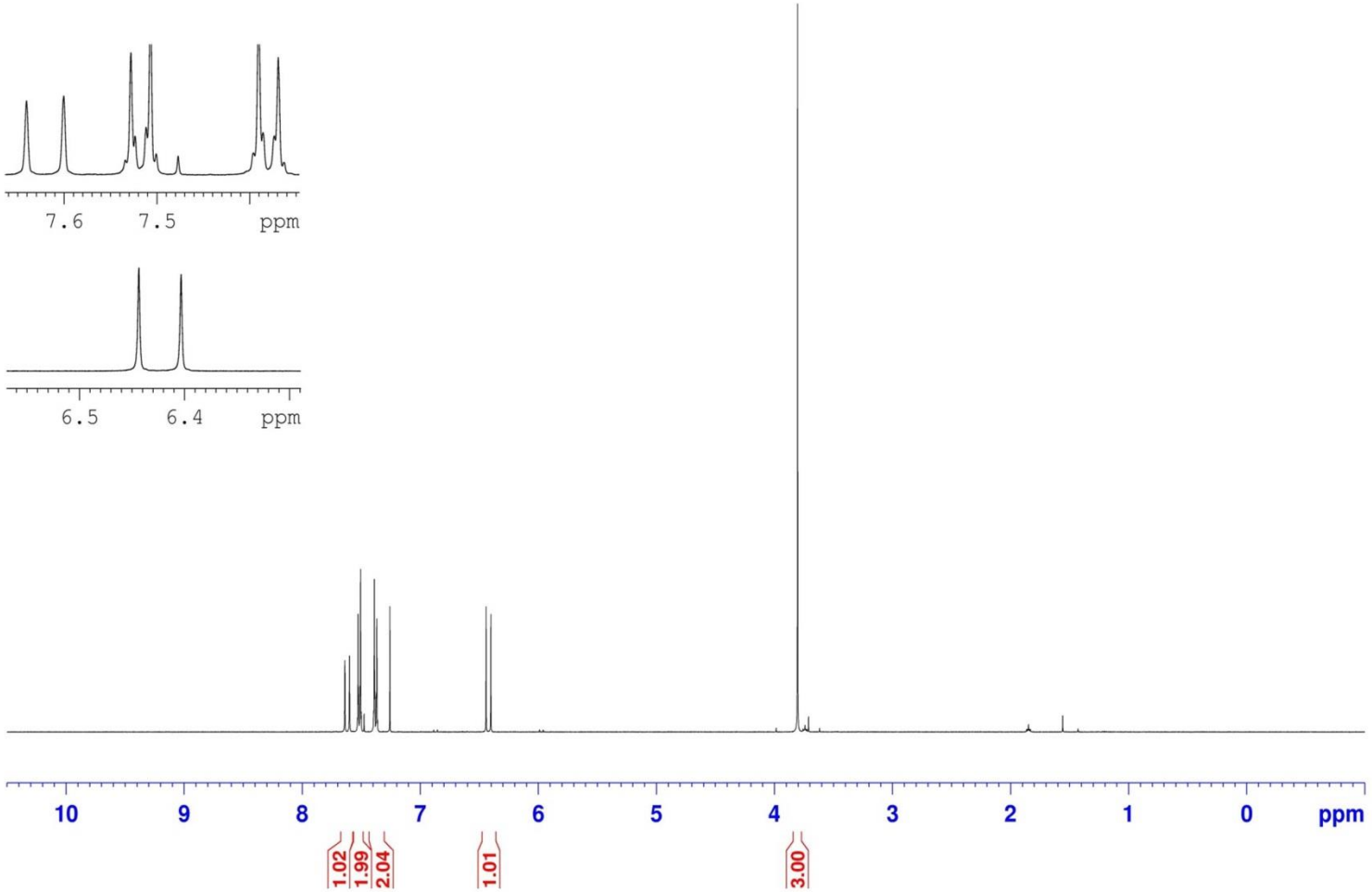
(2E,4E)–nona–2,4–dien–1–ol
400 MHz, CDCl3



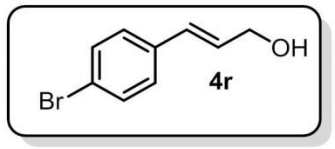


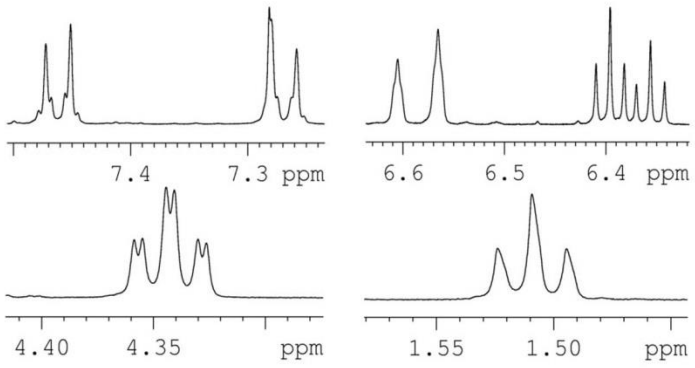
trans–methyl 4–bromocinnamate
400 MHz, CDCl3

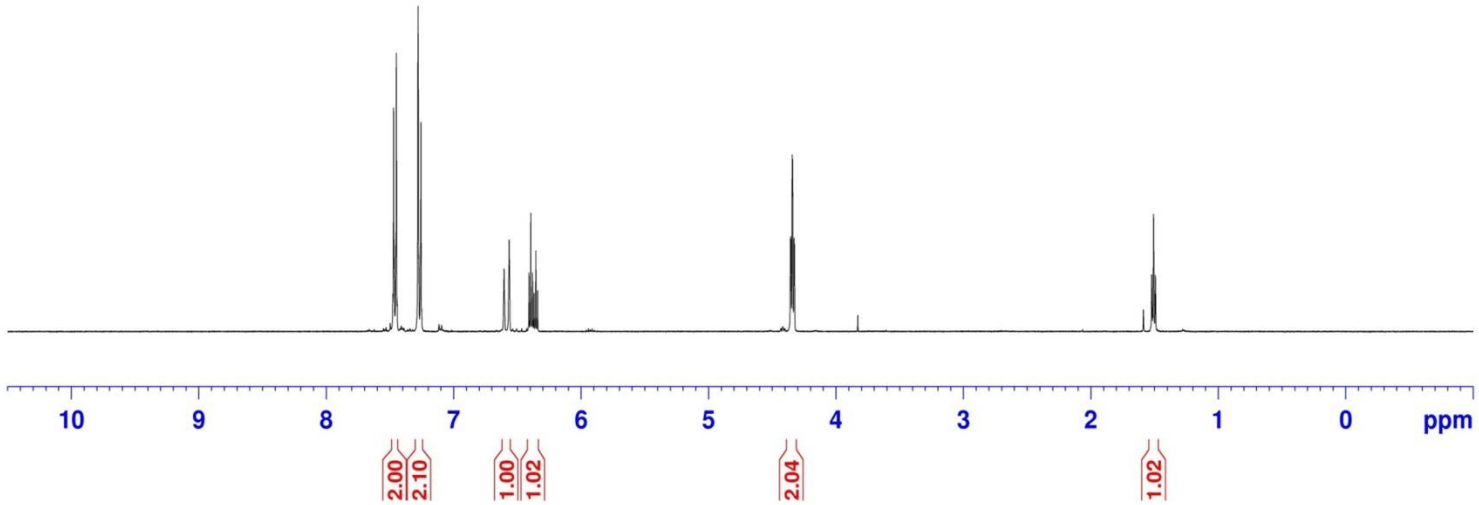




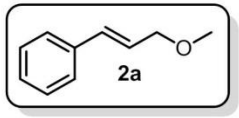
(E)−3−(4−bromophenyl)prop−2−en−1−ol
400 MHz, CDCl3

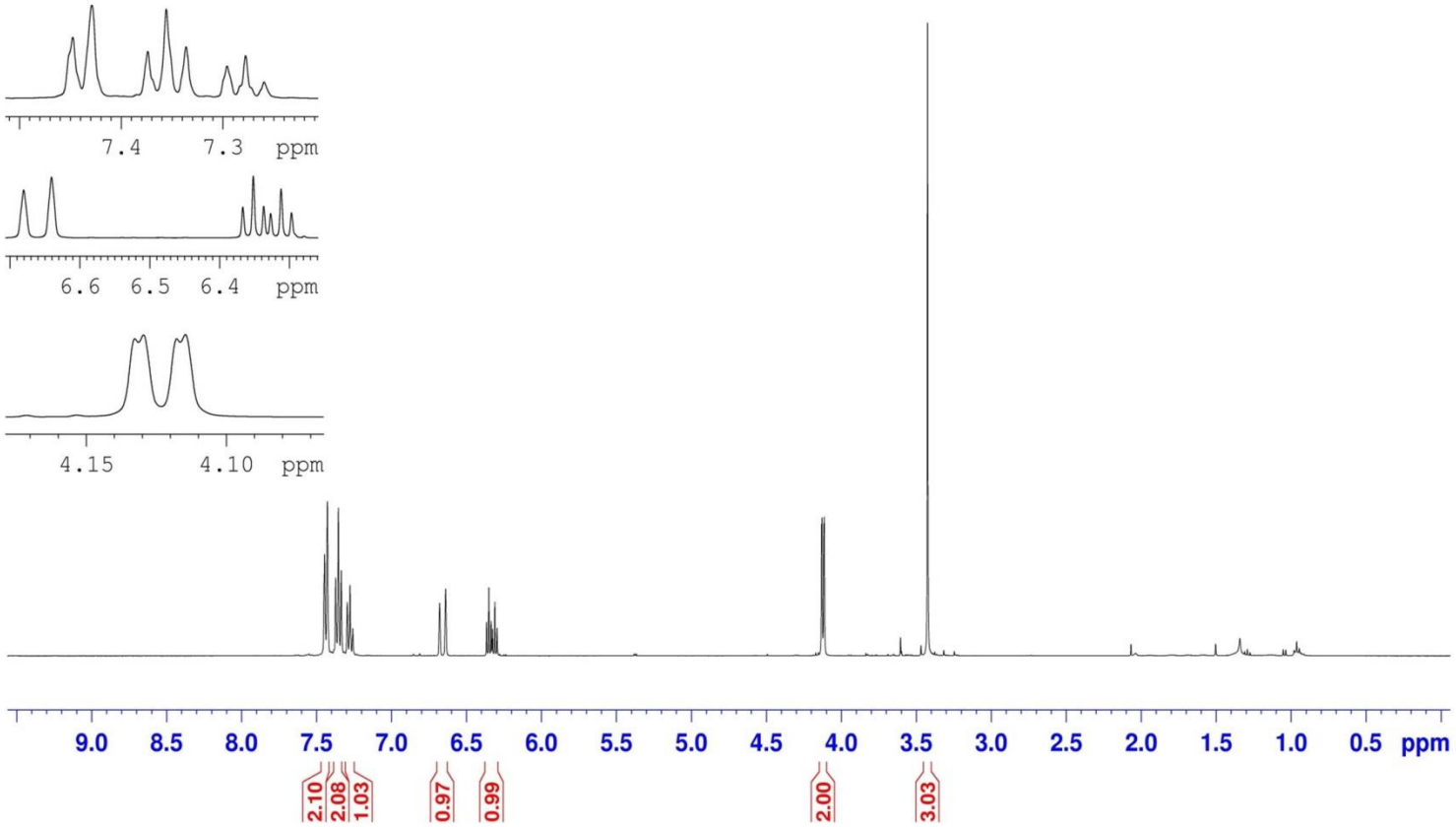




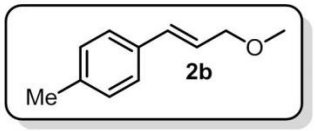


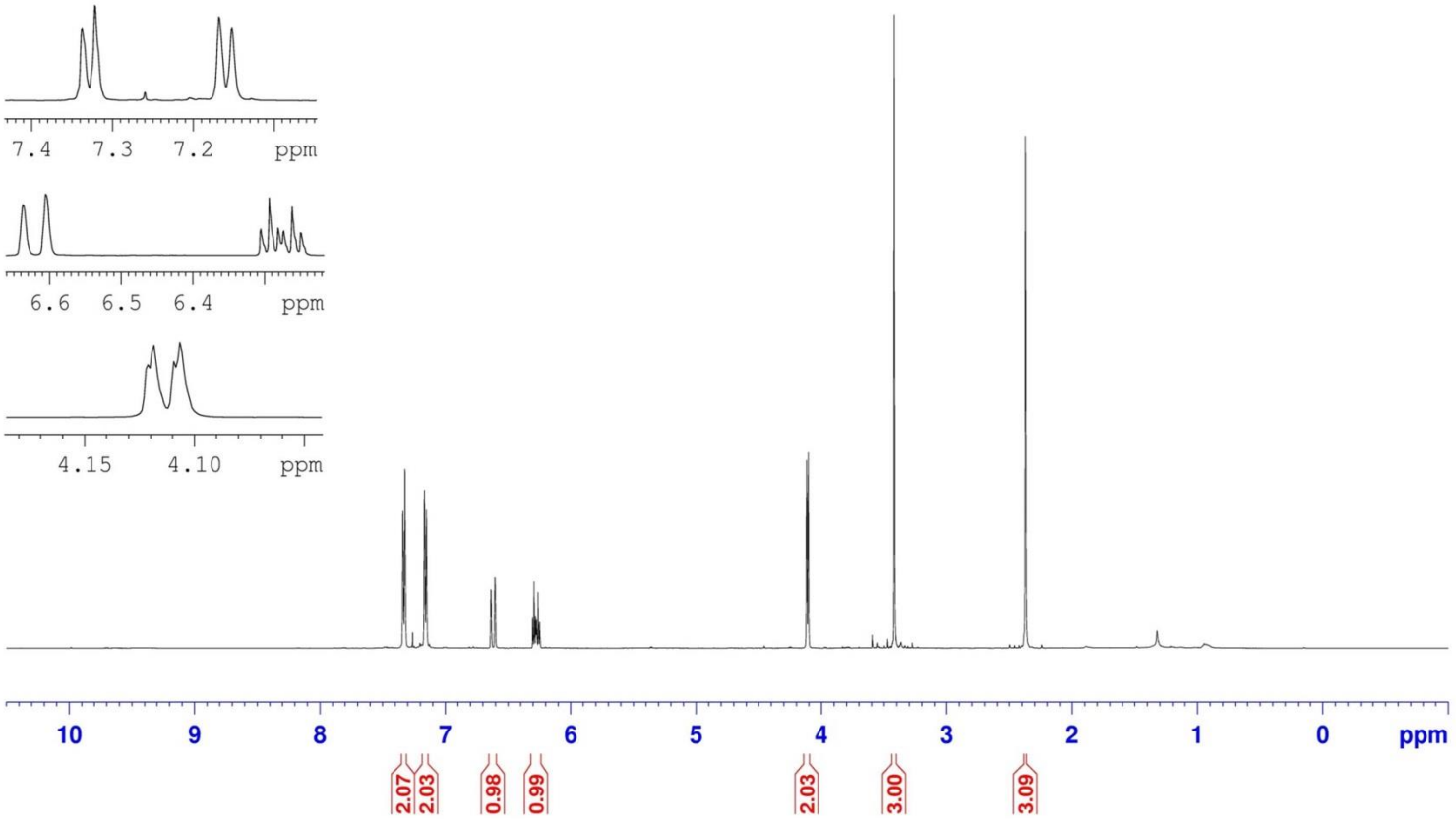
(E)–(3–methoxyprop–1–en–1–yl)benzene
400 MHz, CDCl3



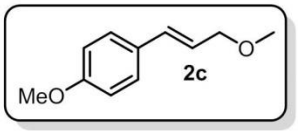


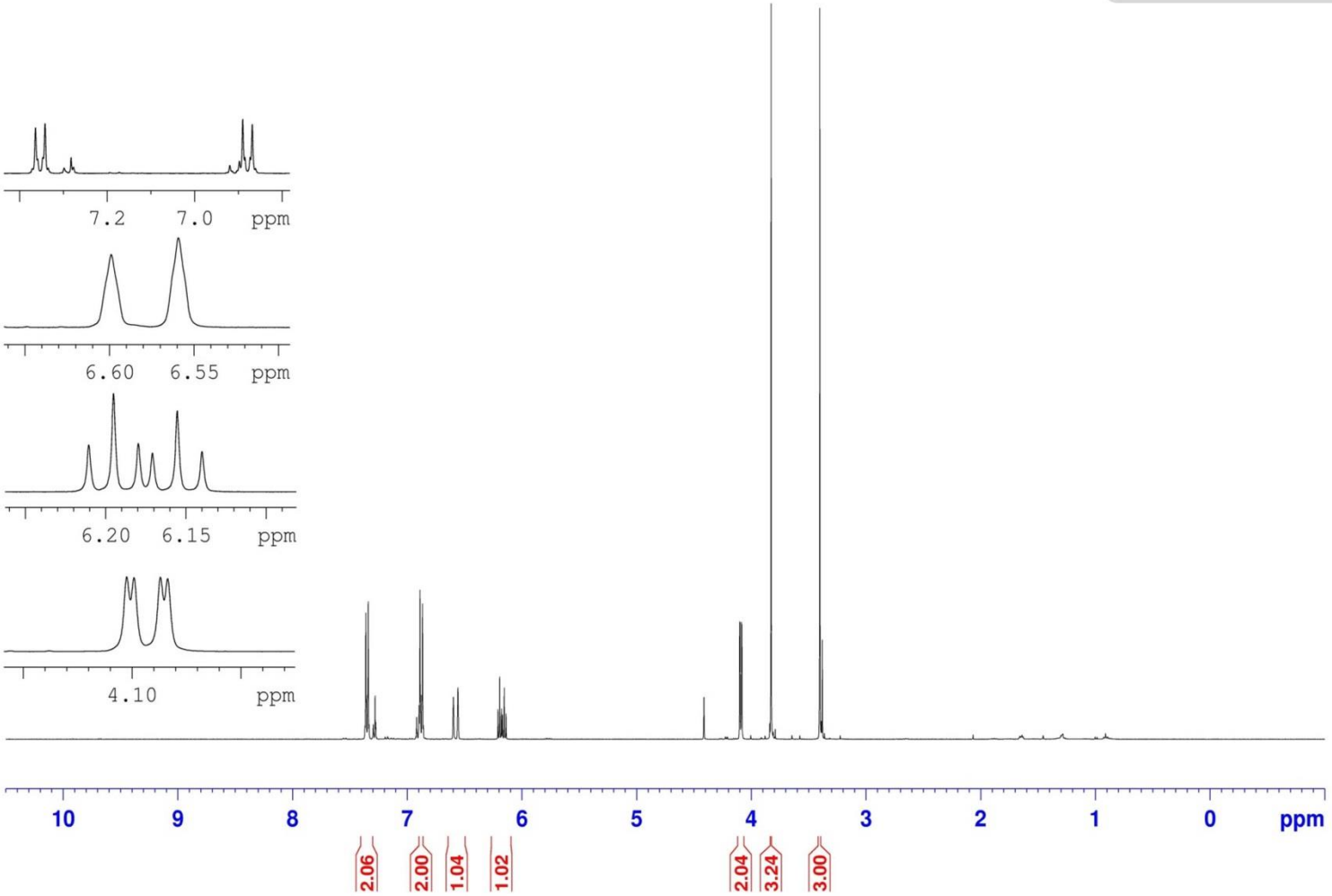
(E)−1−(3−methoxyprop−1−en−1−yl)−4−methylbenzene
500 MHz, CDCl3



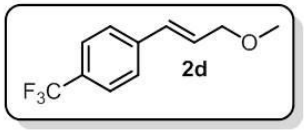


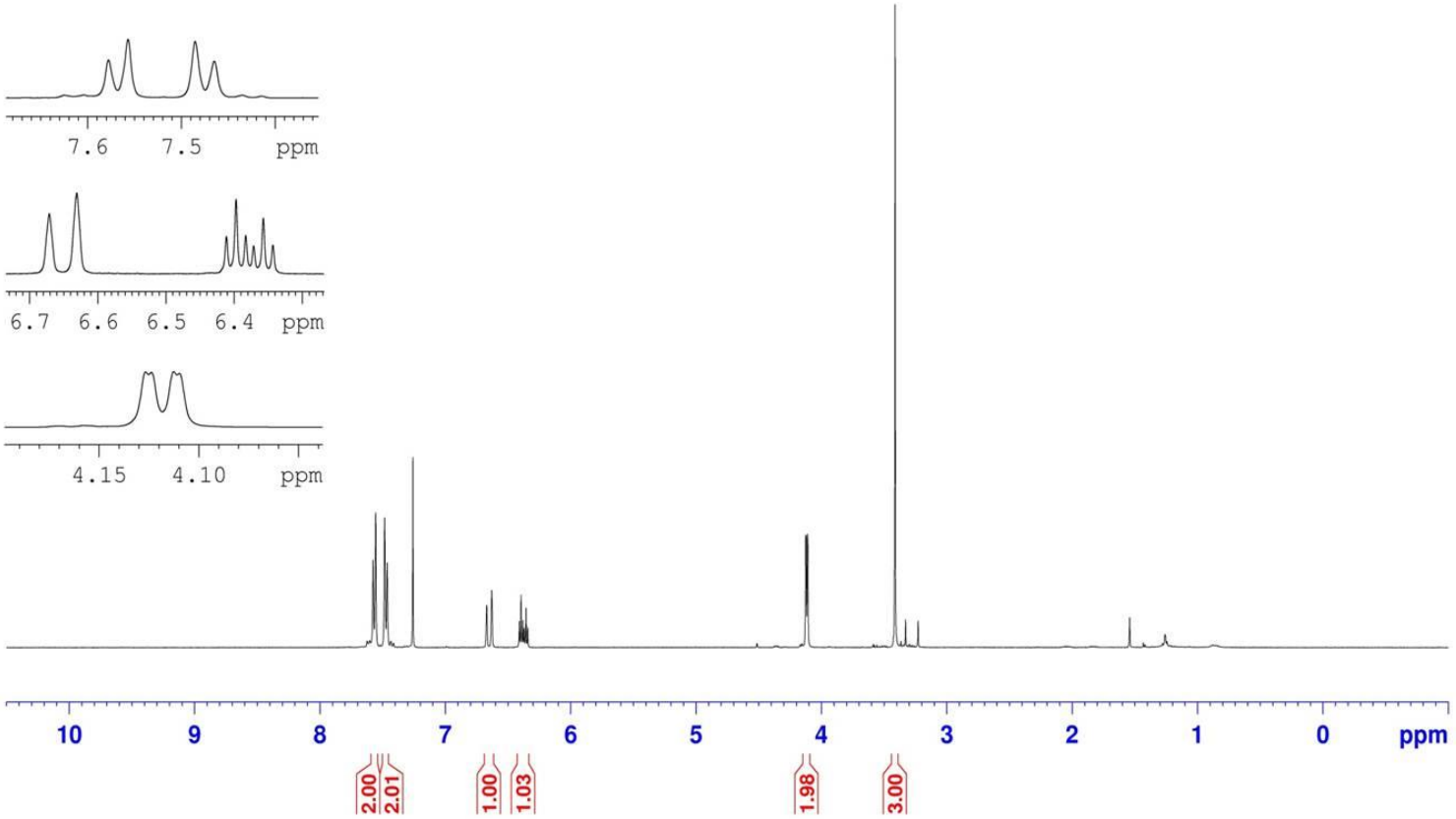
(E)−1−methoxy−4−(3−methoxyprop−1−en−1−yl)benzene
400 MHz, CDCl3



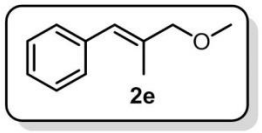


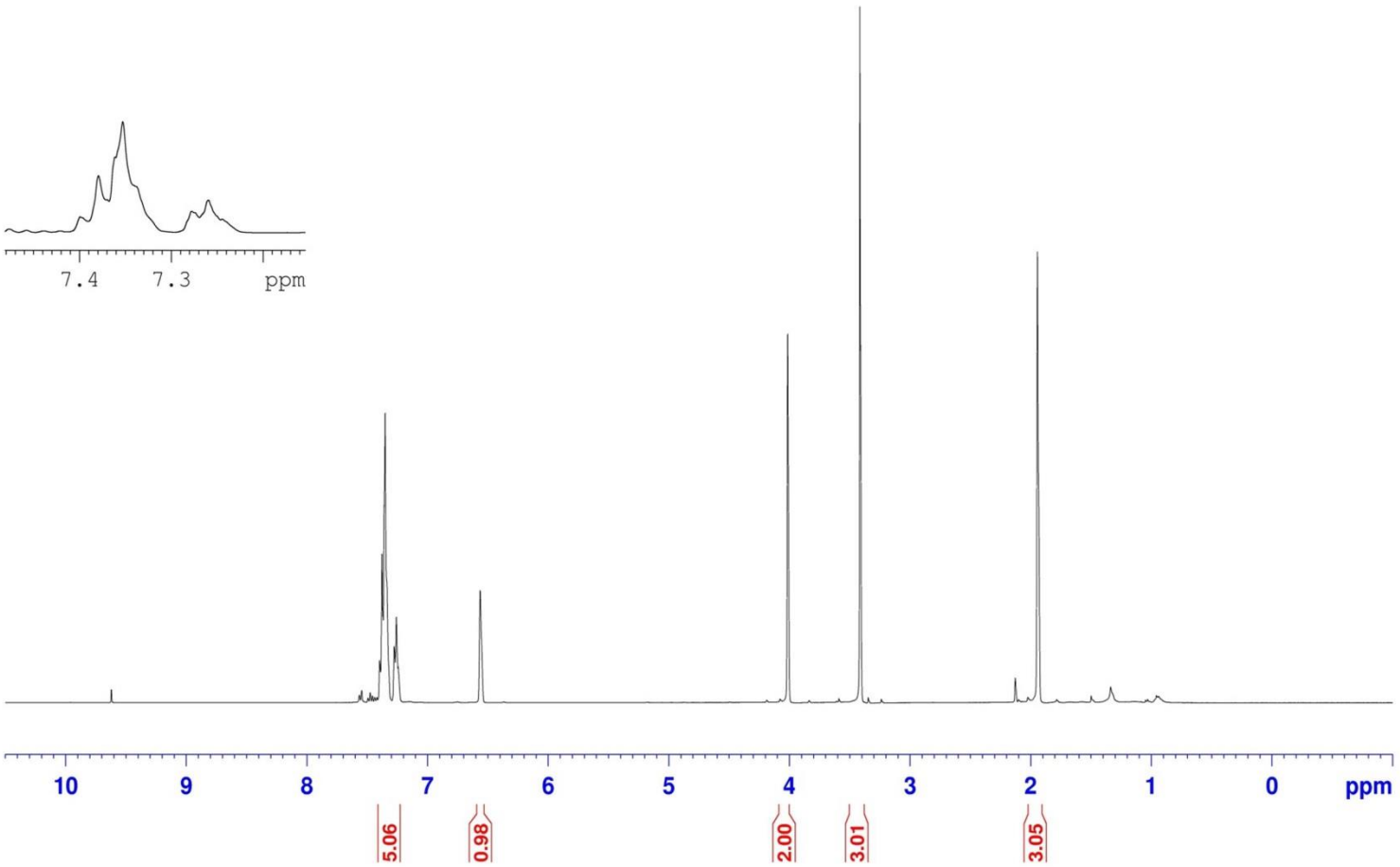
(E)−1−(3−methoxyprop−1−en−1−yl)−4−(trifluoromethyl)benzene
400 MHz, CDCl3



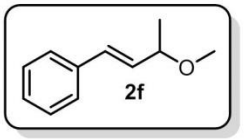


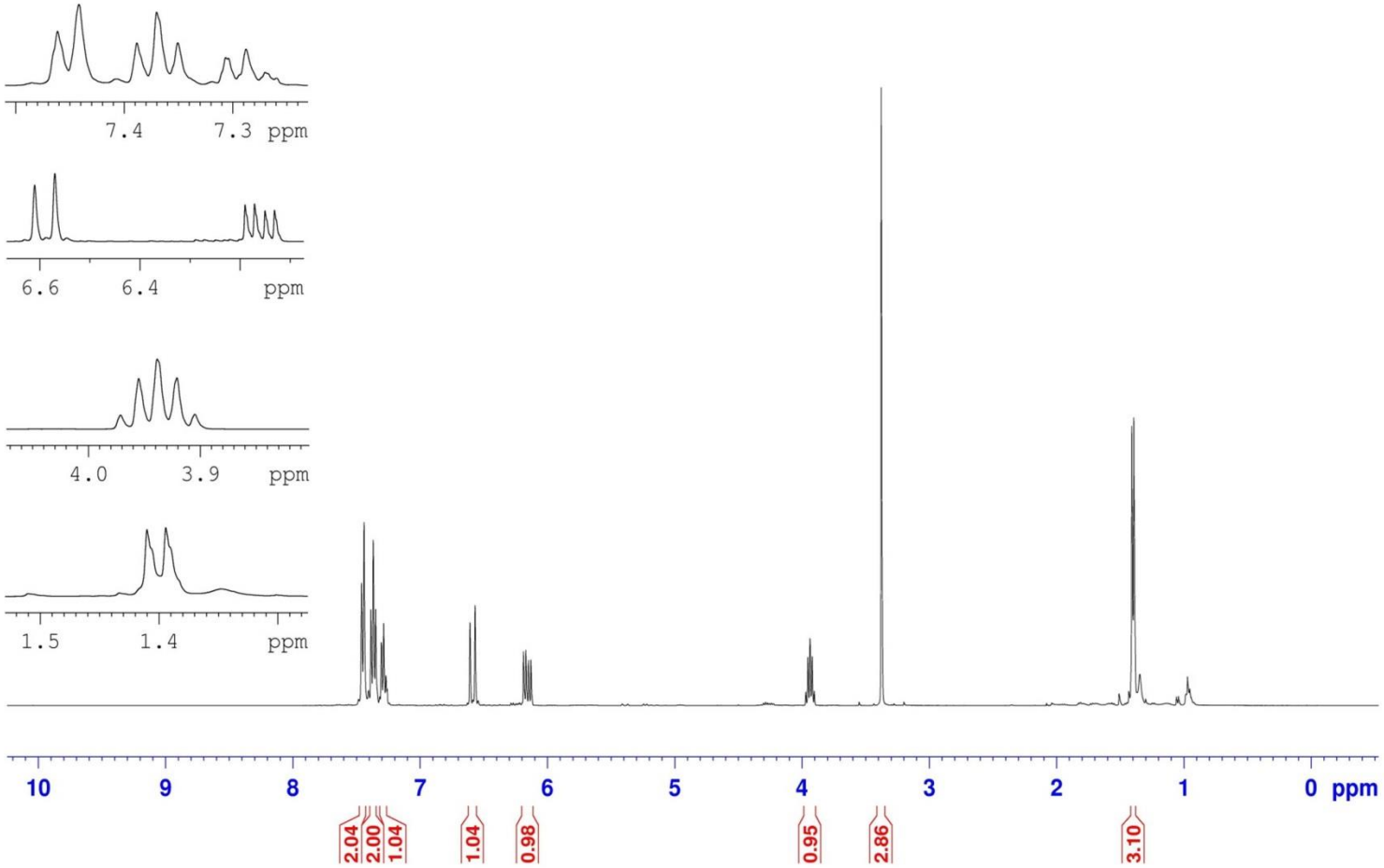
(E)−(3−methoxy−2−methylprop−1−en−1−yl)benzene
400 MHz, CDCl3



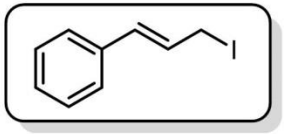


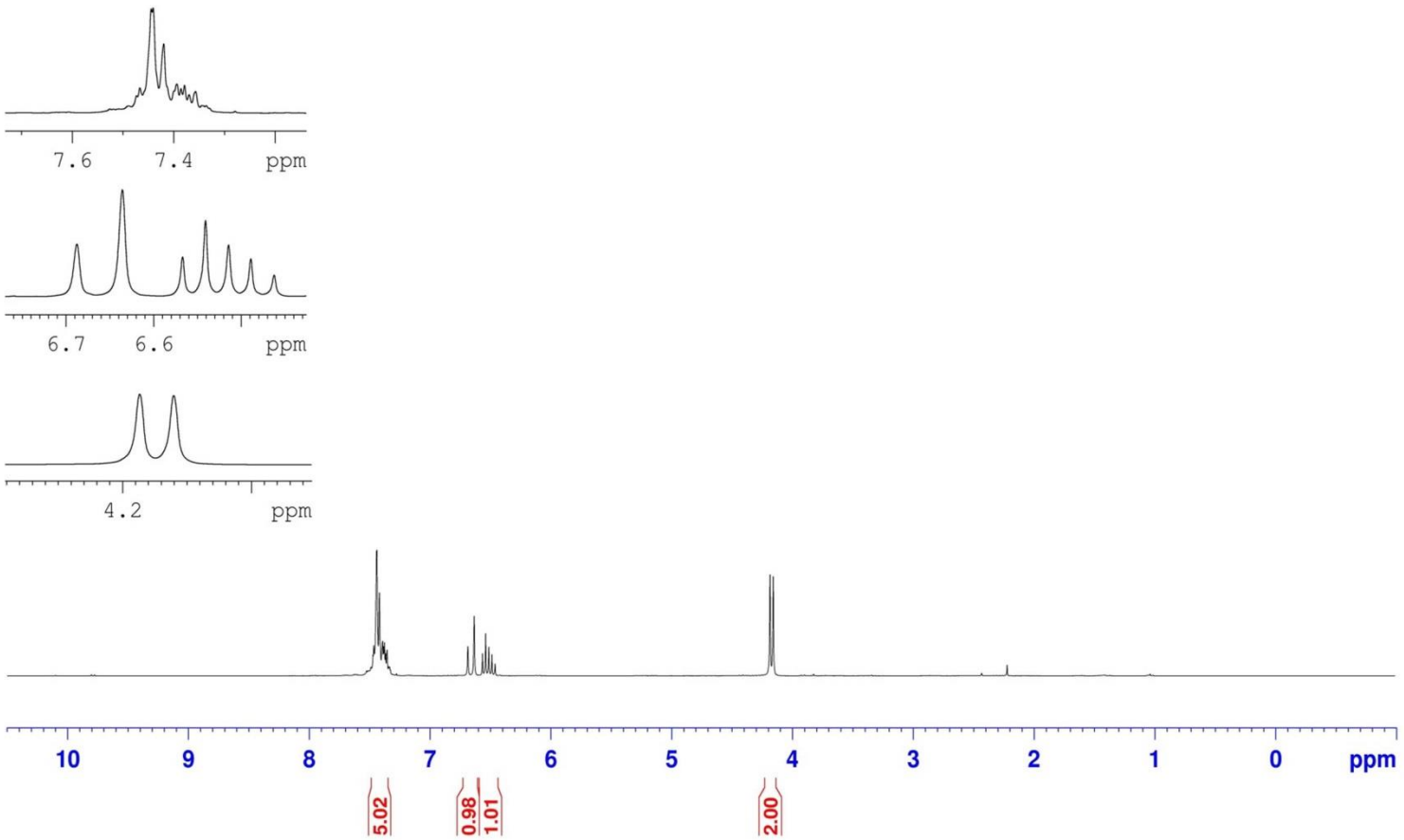
(E)–(3–methoxybut–1–en–1–yl)benzene
400 MHz, CDCl3



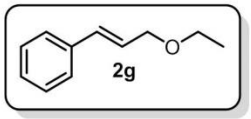


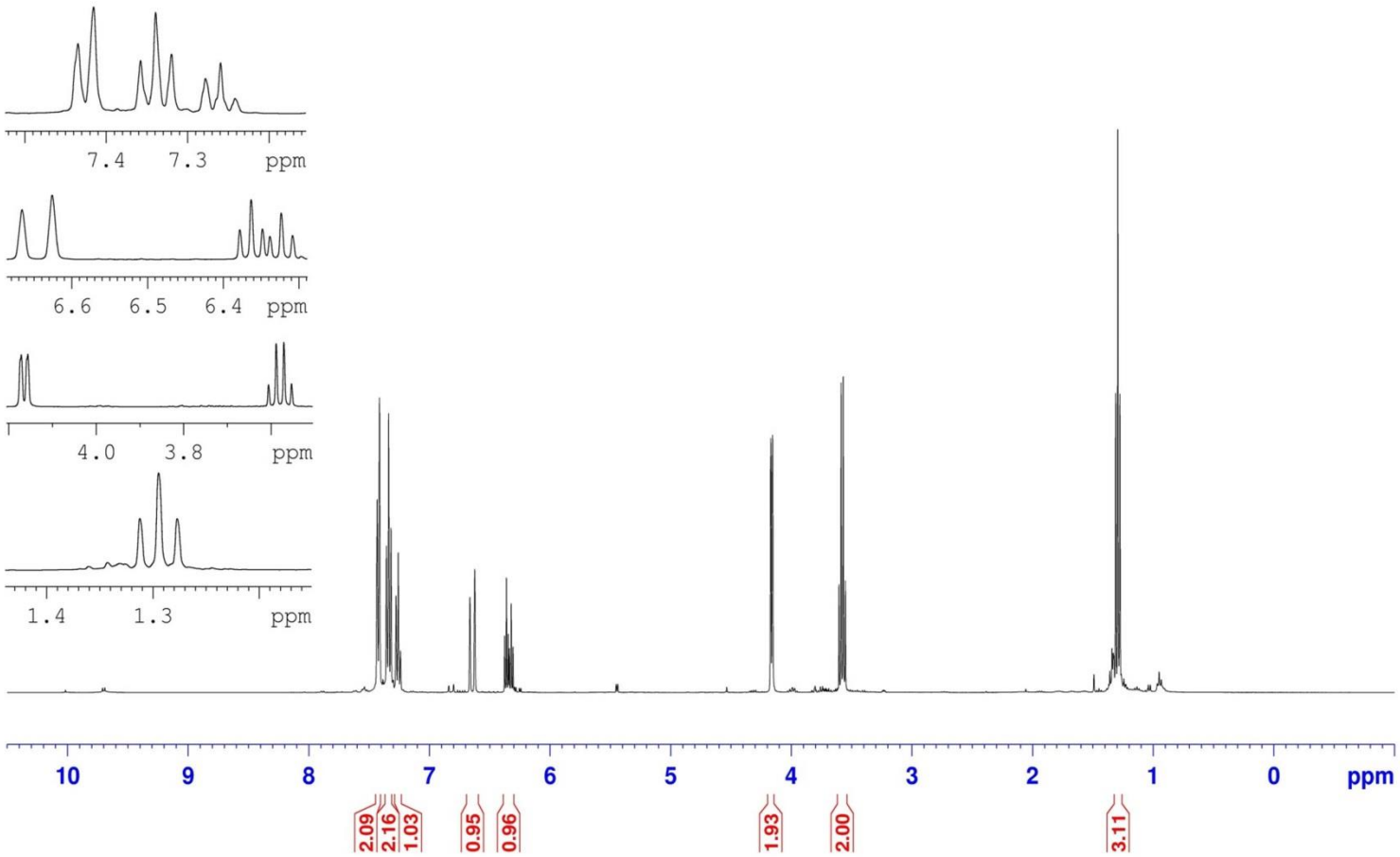
cinnamyl iodide
300 MHz, CDCl3



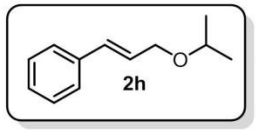


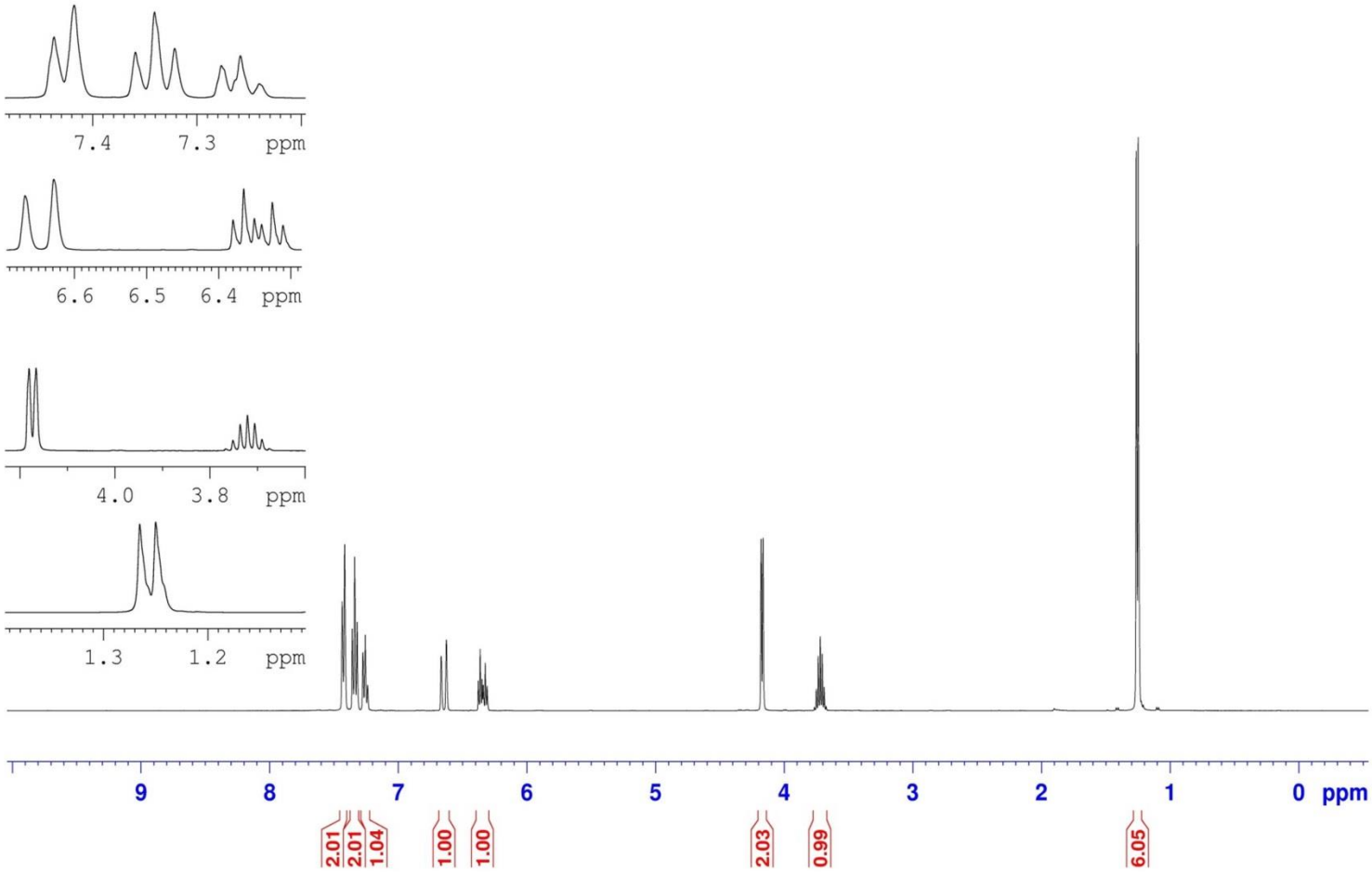
(E)−(3−ethoxyprop−1−en−1−yl)benzene
400 MHz, CDCl3



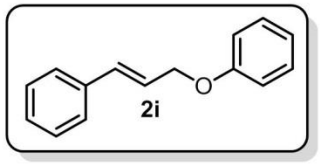


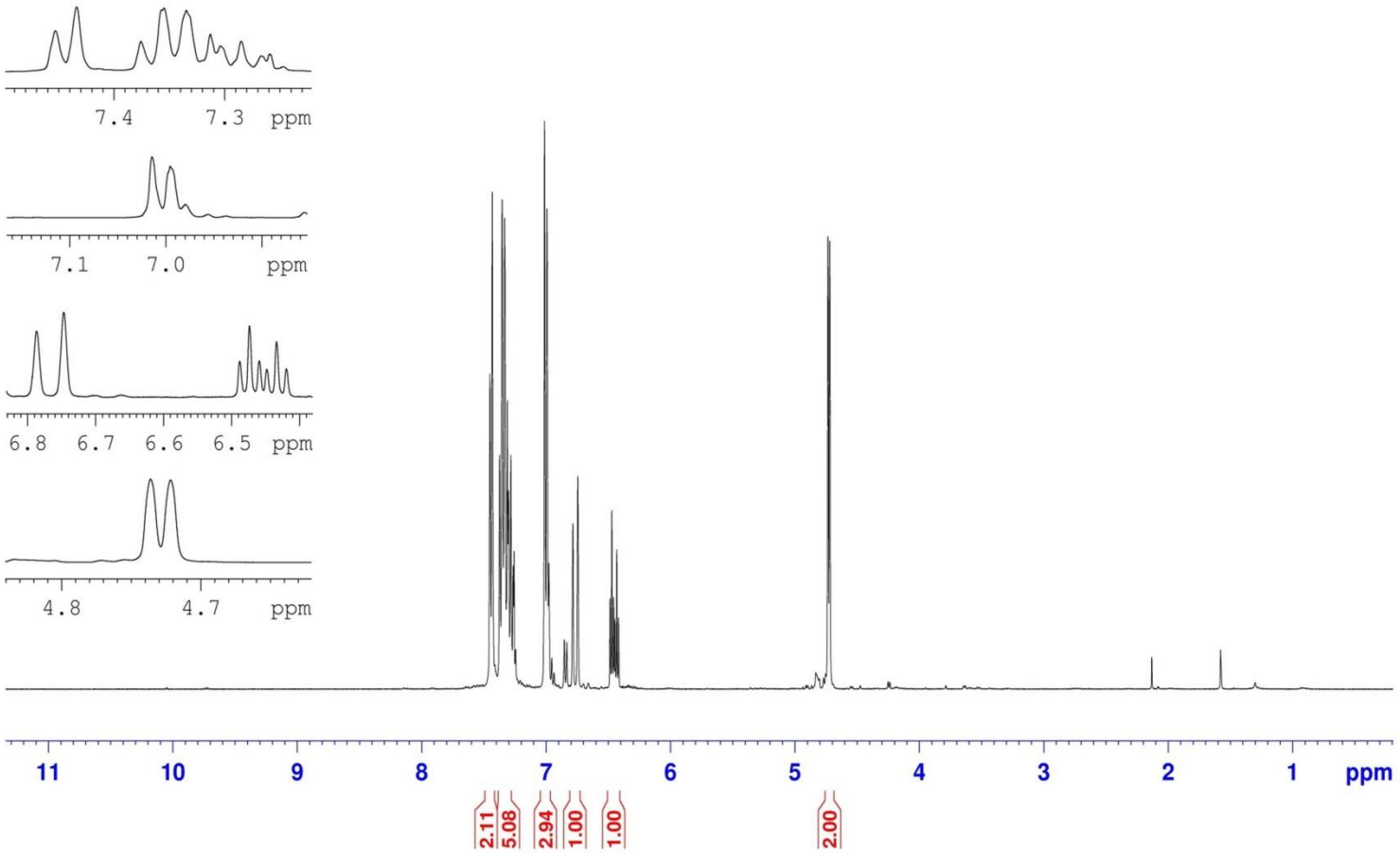
(E)−(3−isopropoxyprop−1−en−1−yl)benzene
400 MHz, CDCl3



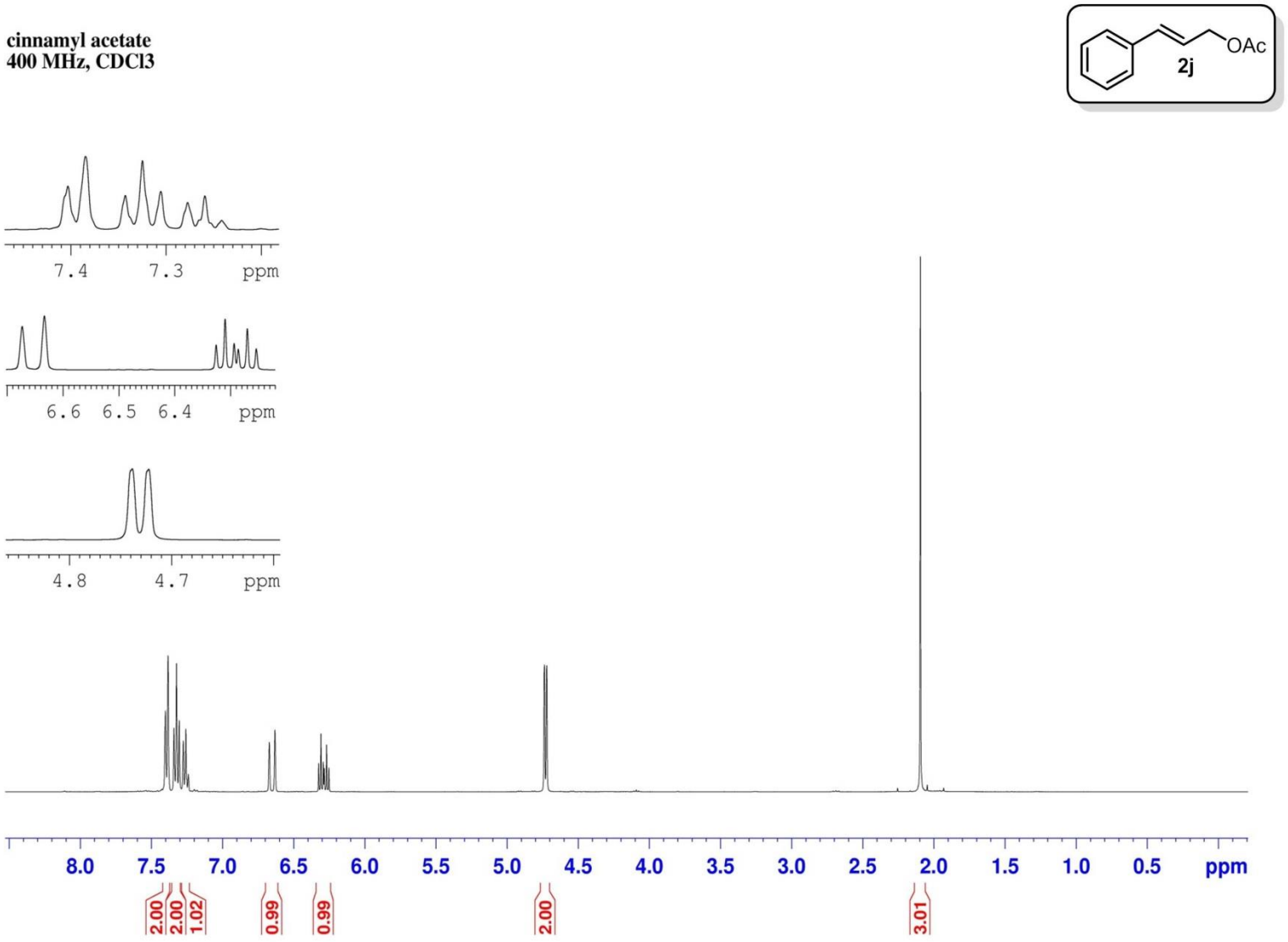


(cinnamyloxy)benzene
400 MHz, CDCl3

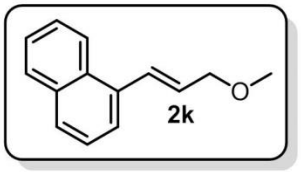


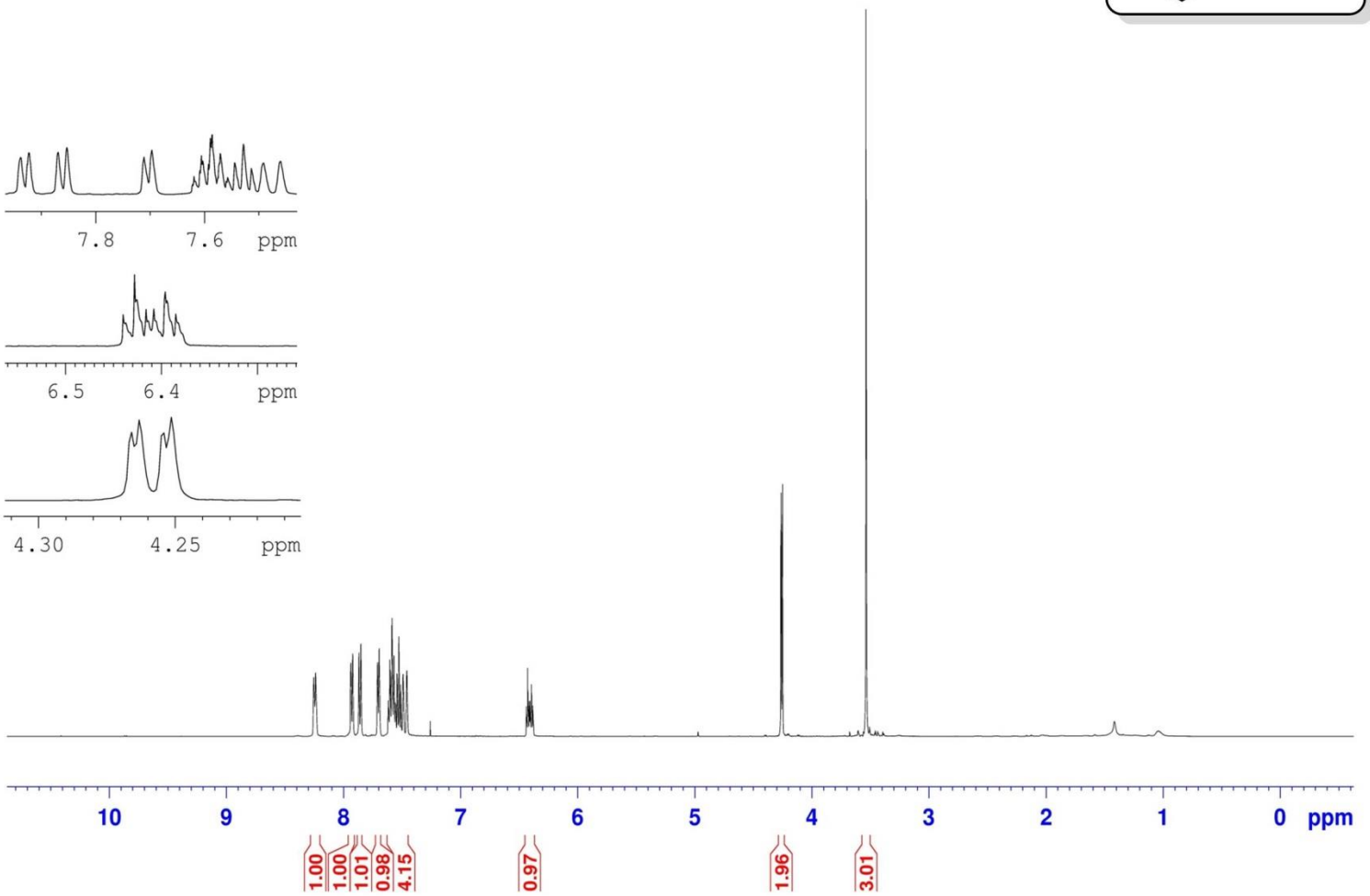


cinnamyl acetate
400 MHz, CDCl3

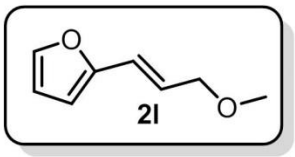


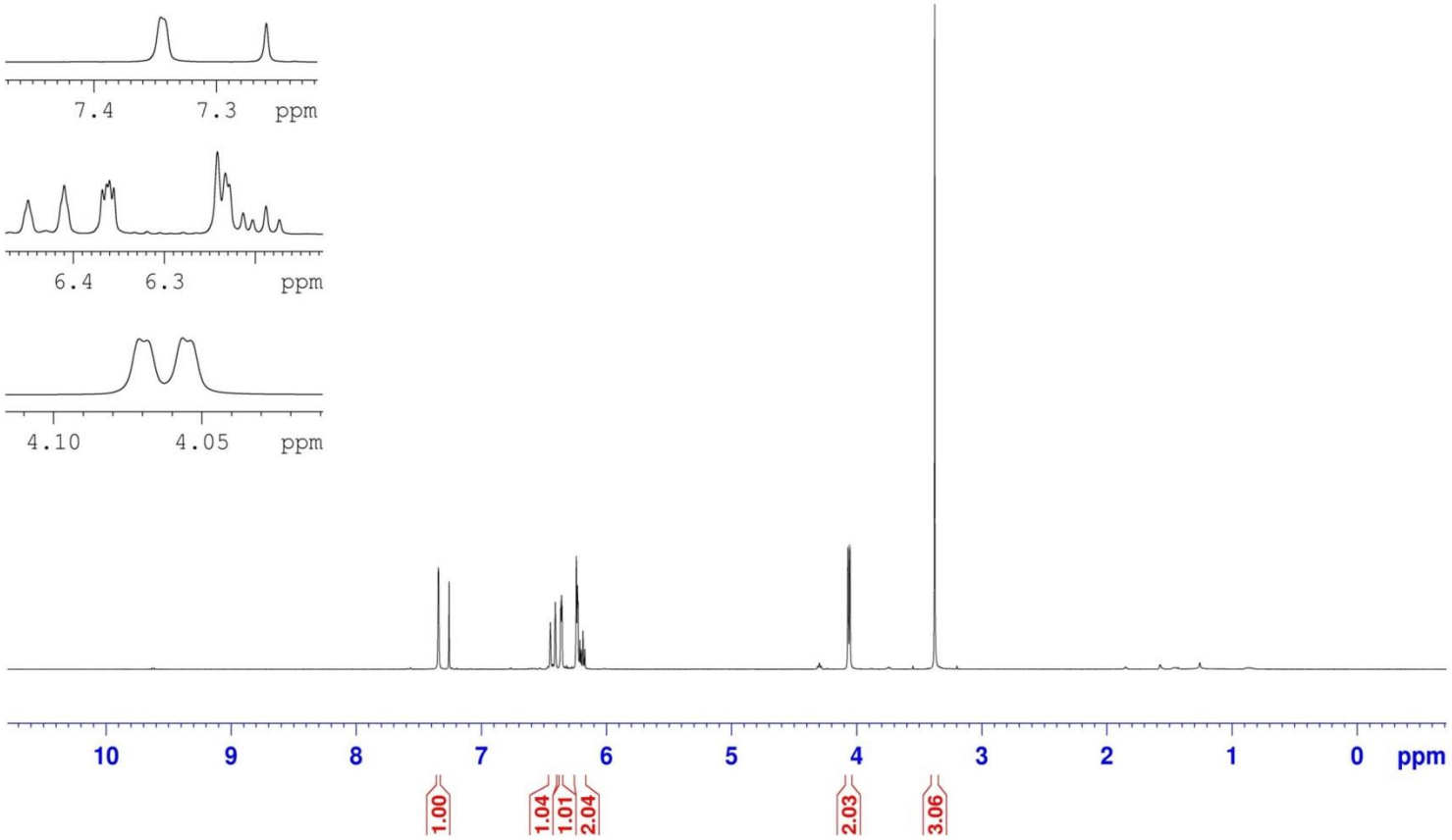
(E)−1−(3−methoxyprop−1−en−1−yl)naphthalene
500 MHz, CDCl3



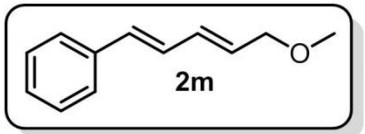


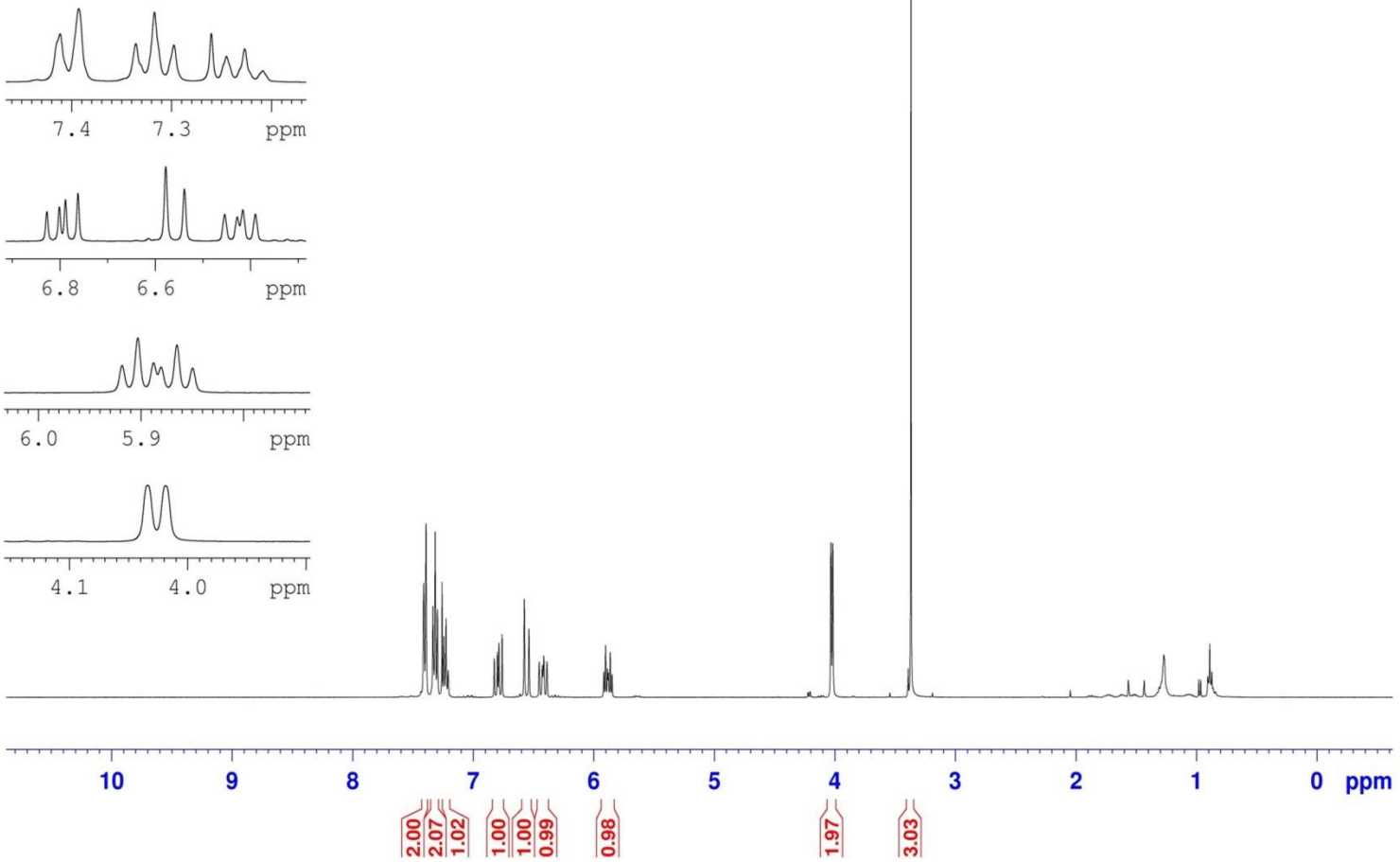
(E)−2−(3−methoxyprop−1−en−1−yl)furan
400 MHz, CDCl3



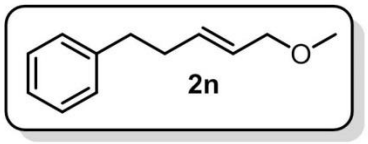


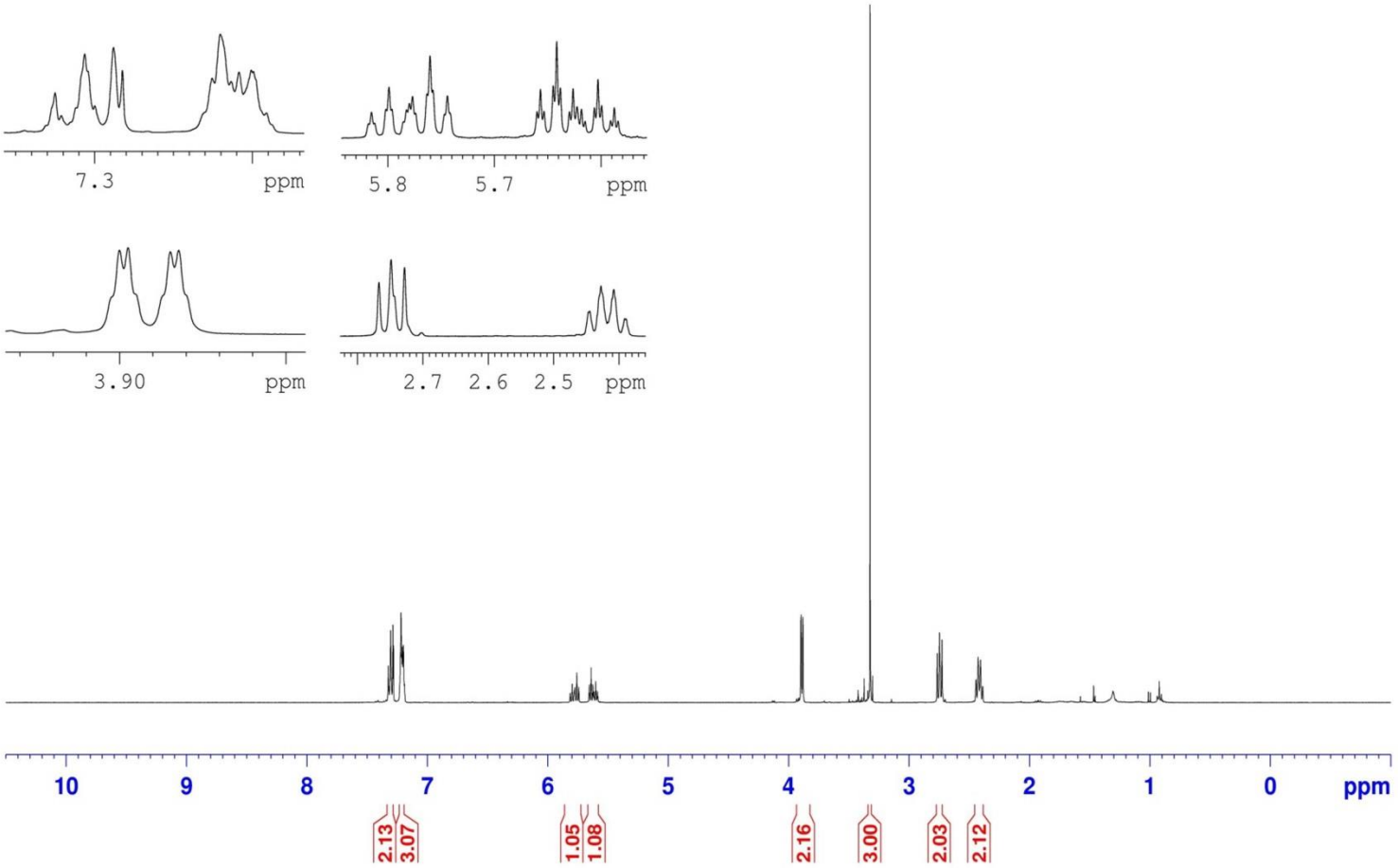
((1E,3E)−5−methoxypenta−1,3−dien−1−yl)benzene
400 MHz, CDCl3



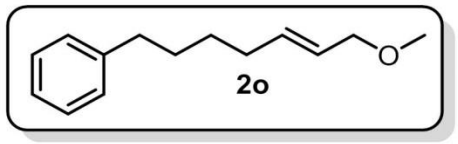


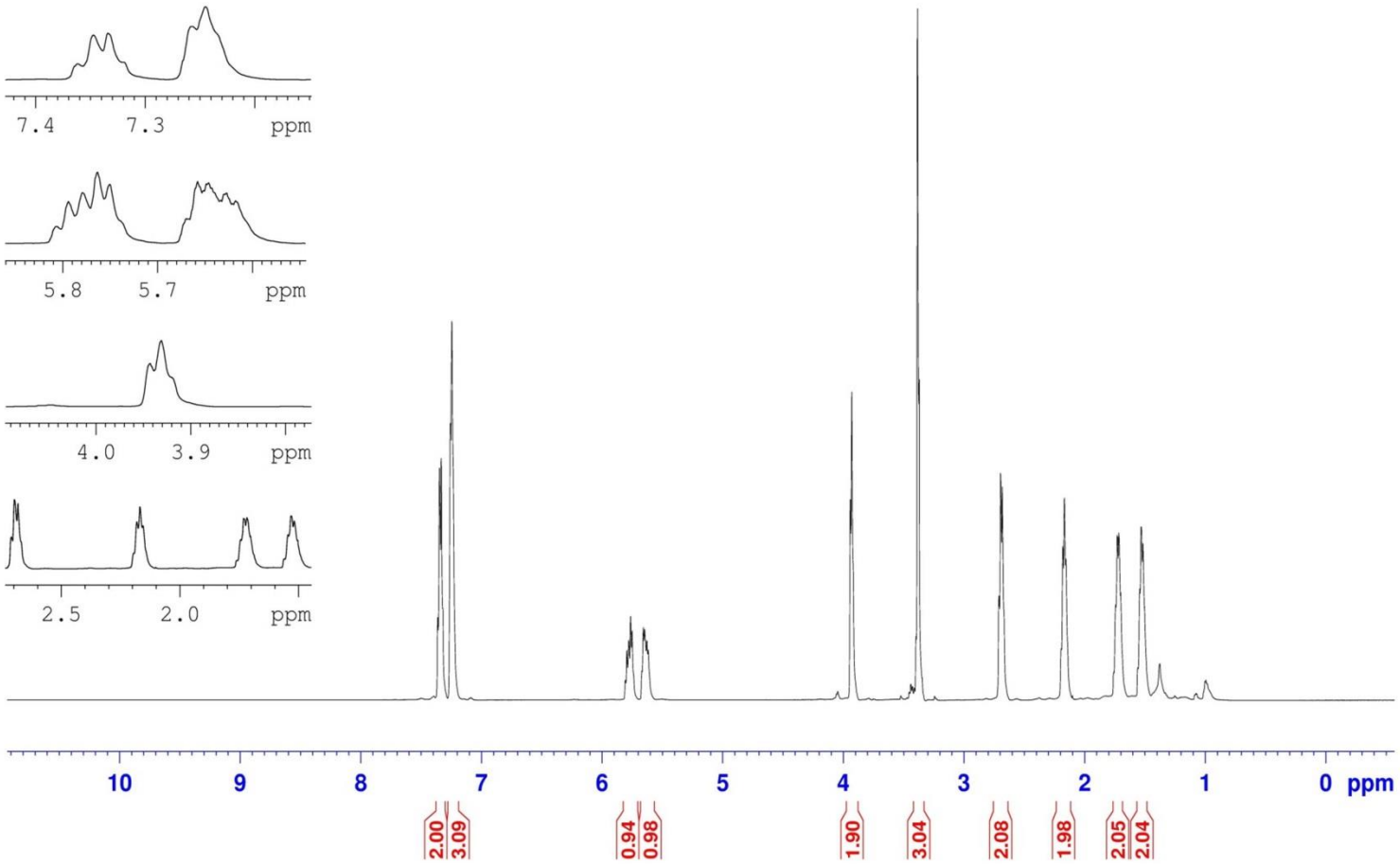
(E)–(5–methoxypent–3–en–1–yl)benzene
400 MHZ, CDCl3



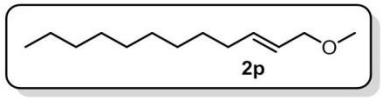


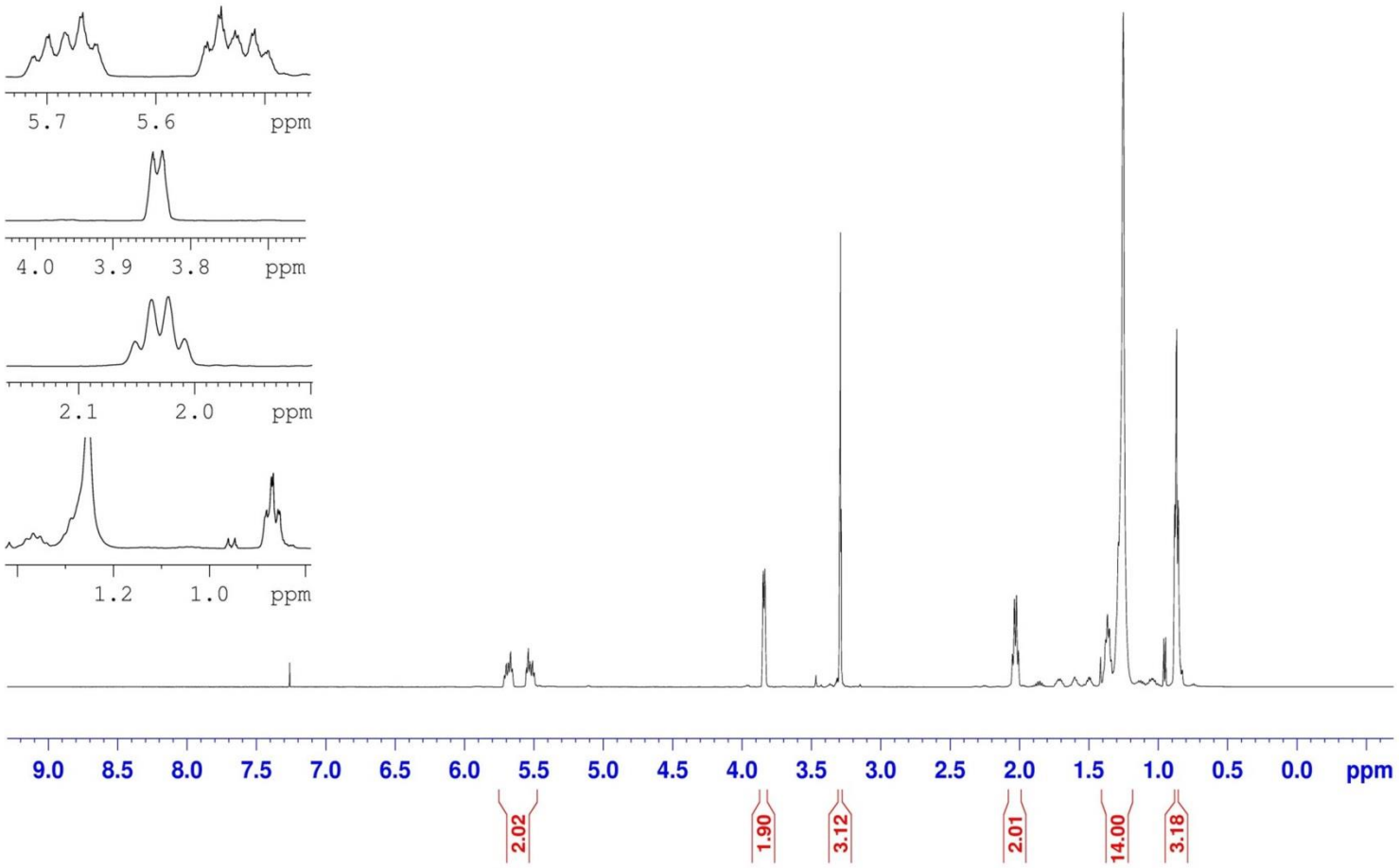
(E)−(7−methoxyhept−5−en−1−yl)benzene
500 MHz, CDCl3



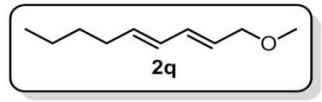


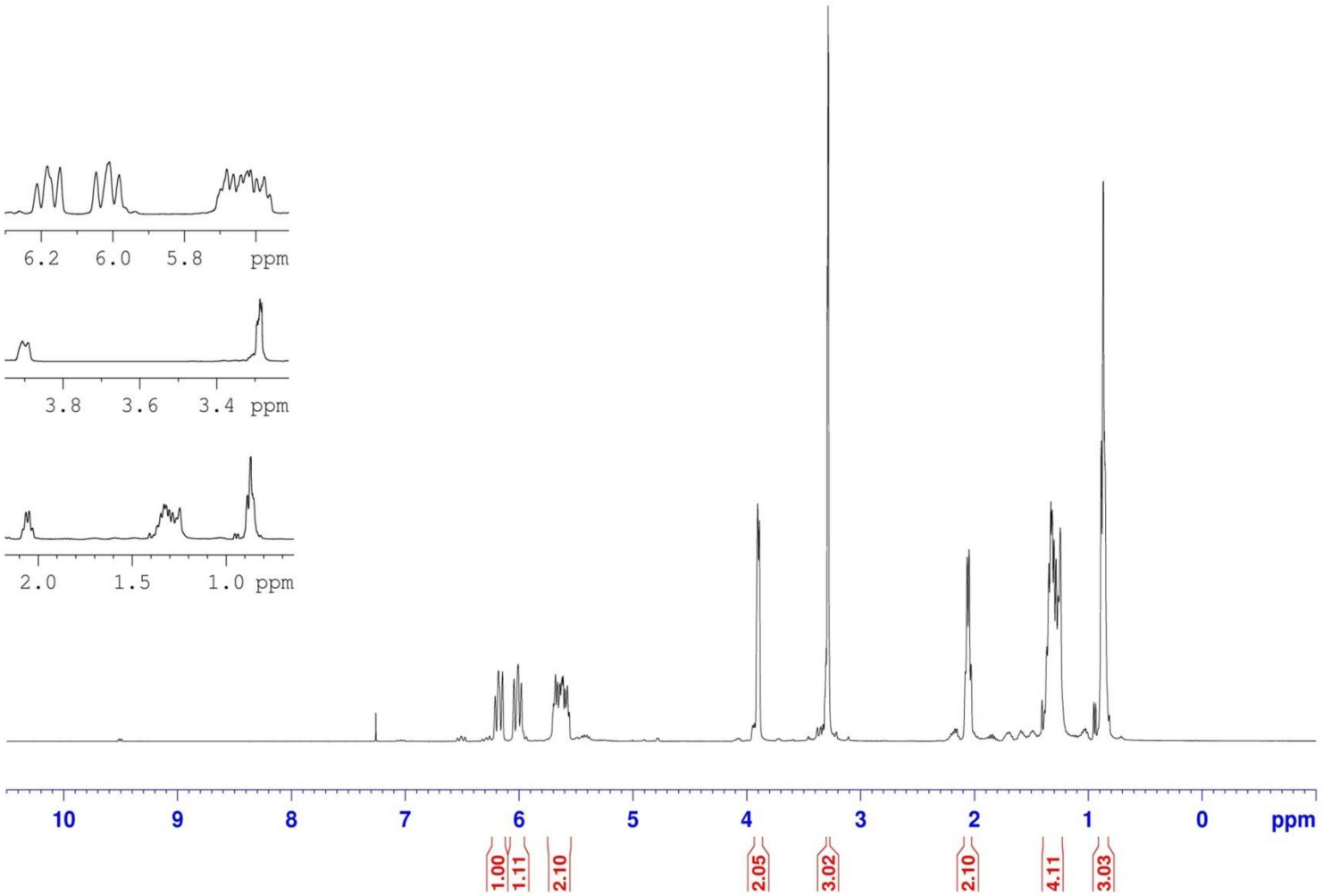
(E)−1−methoxydodec−2−ene
500 MHz, CDCl3



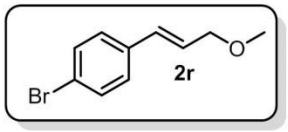


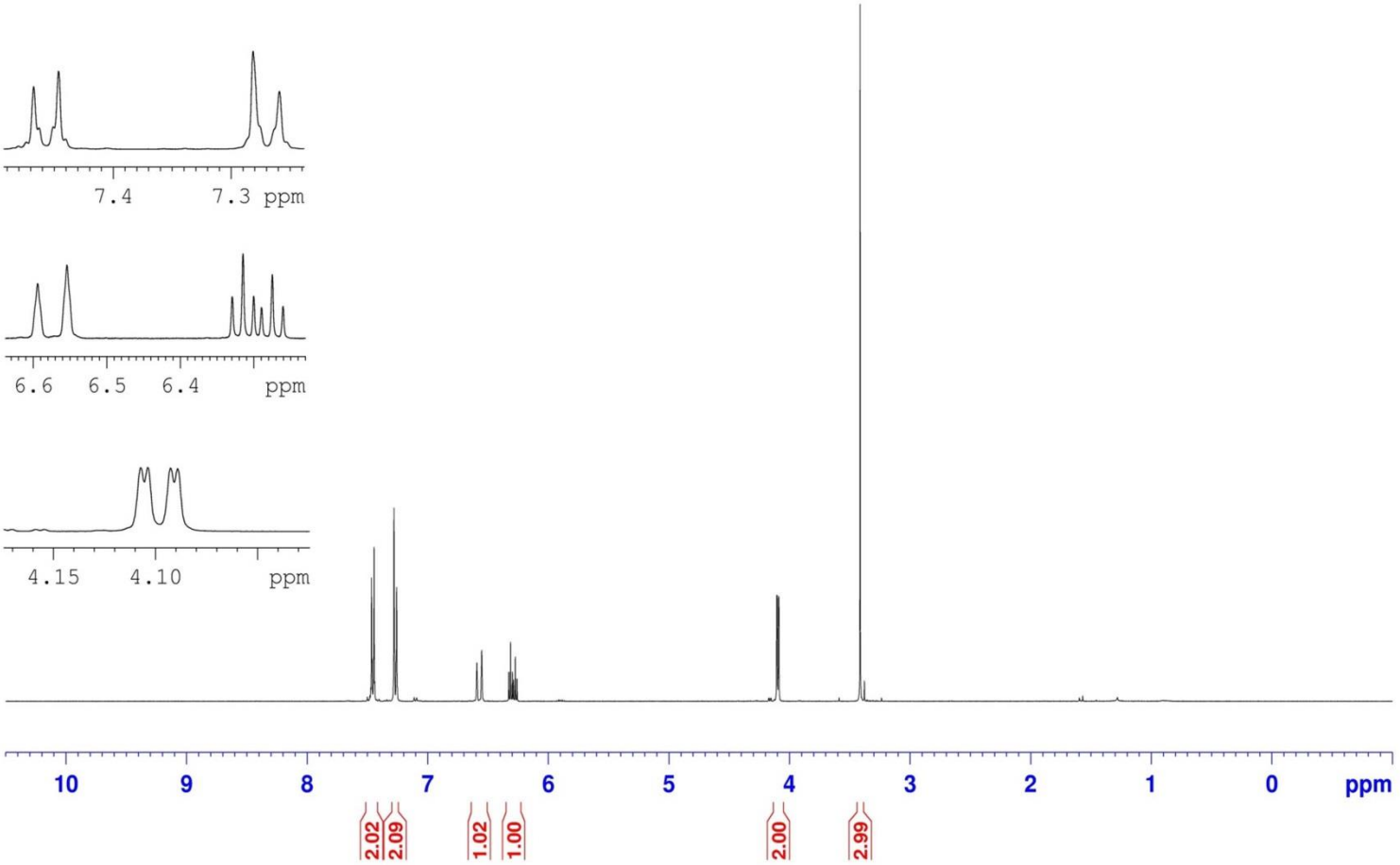
(2E,4E)−1−methoxynona−2,4−diene
400 MHz, CDCl3



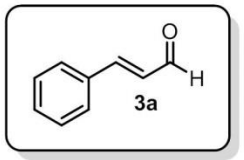


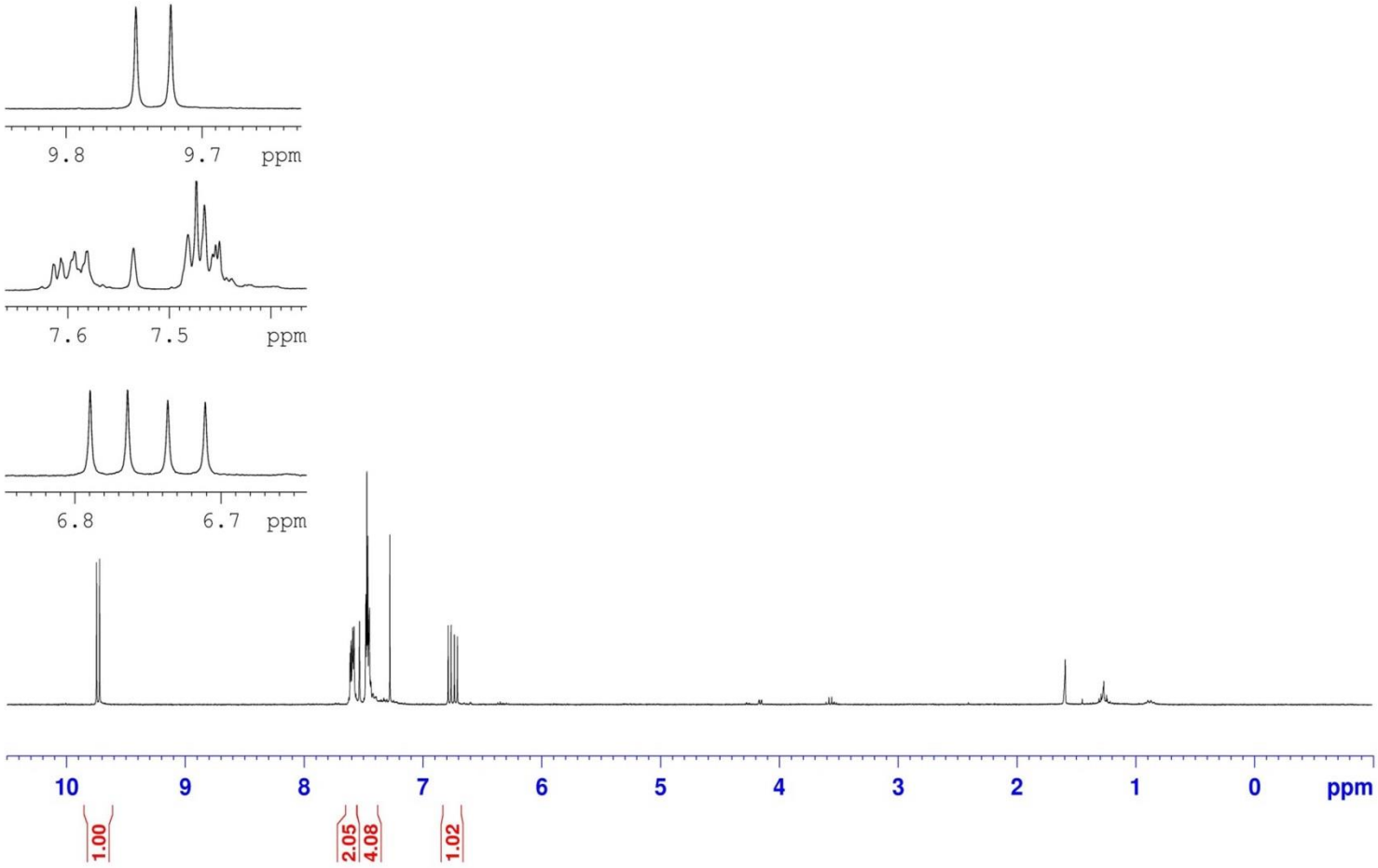
(E)−1−bromo−4−(3−methoxyprop−1−en−1−yl)benzene
400 MHz, CDCl3



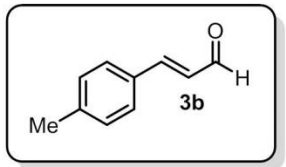


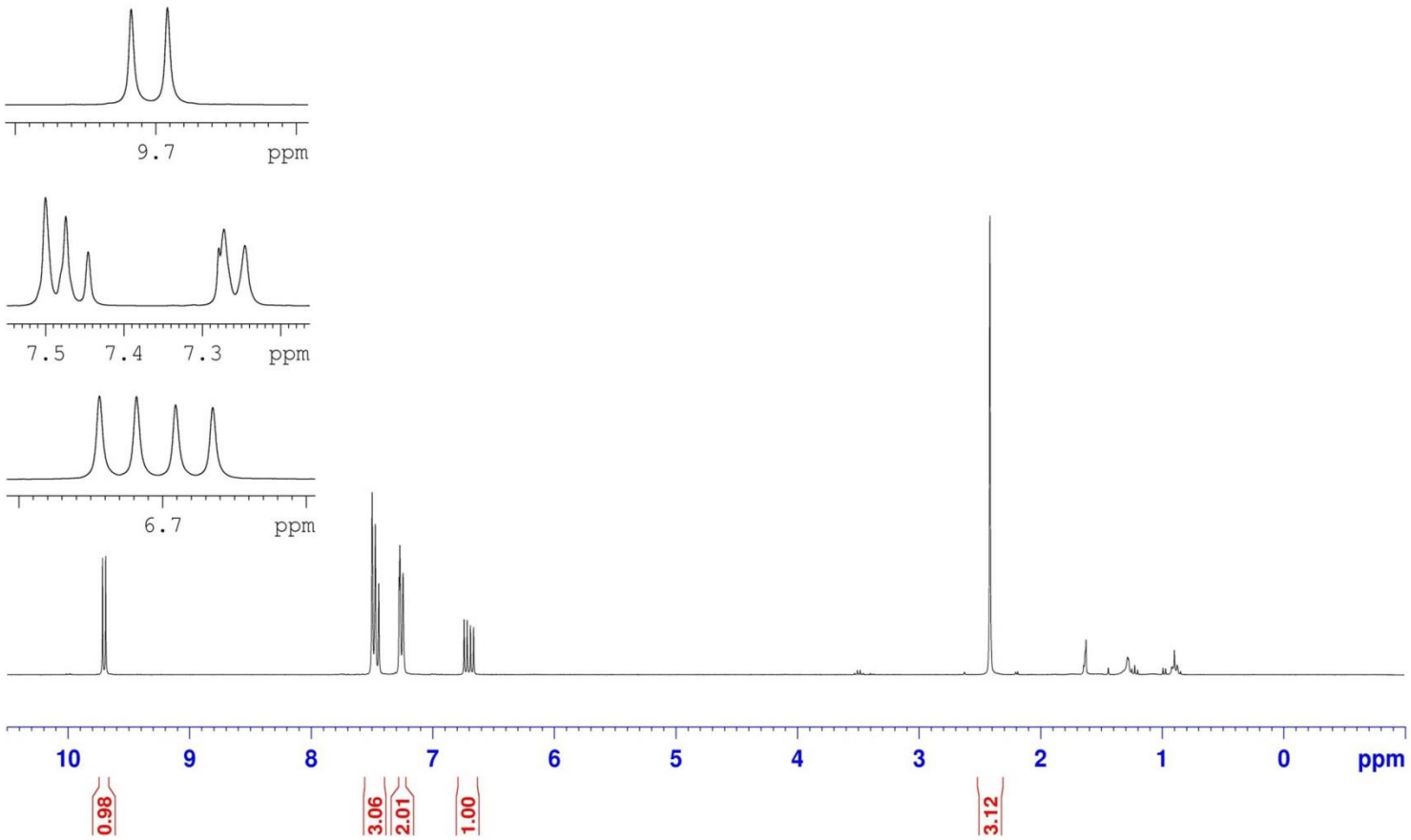
cinnamaldehyde
300 MHz, CDCl3



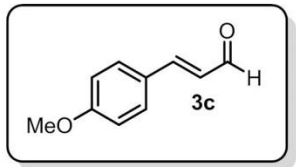


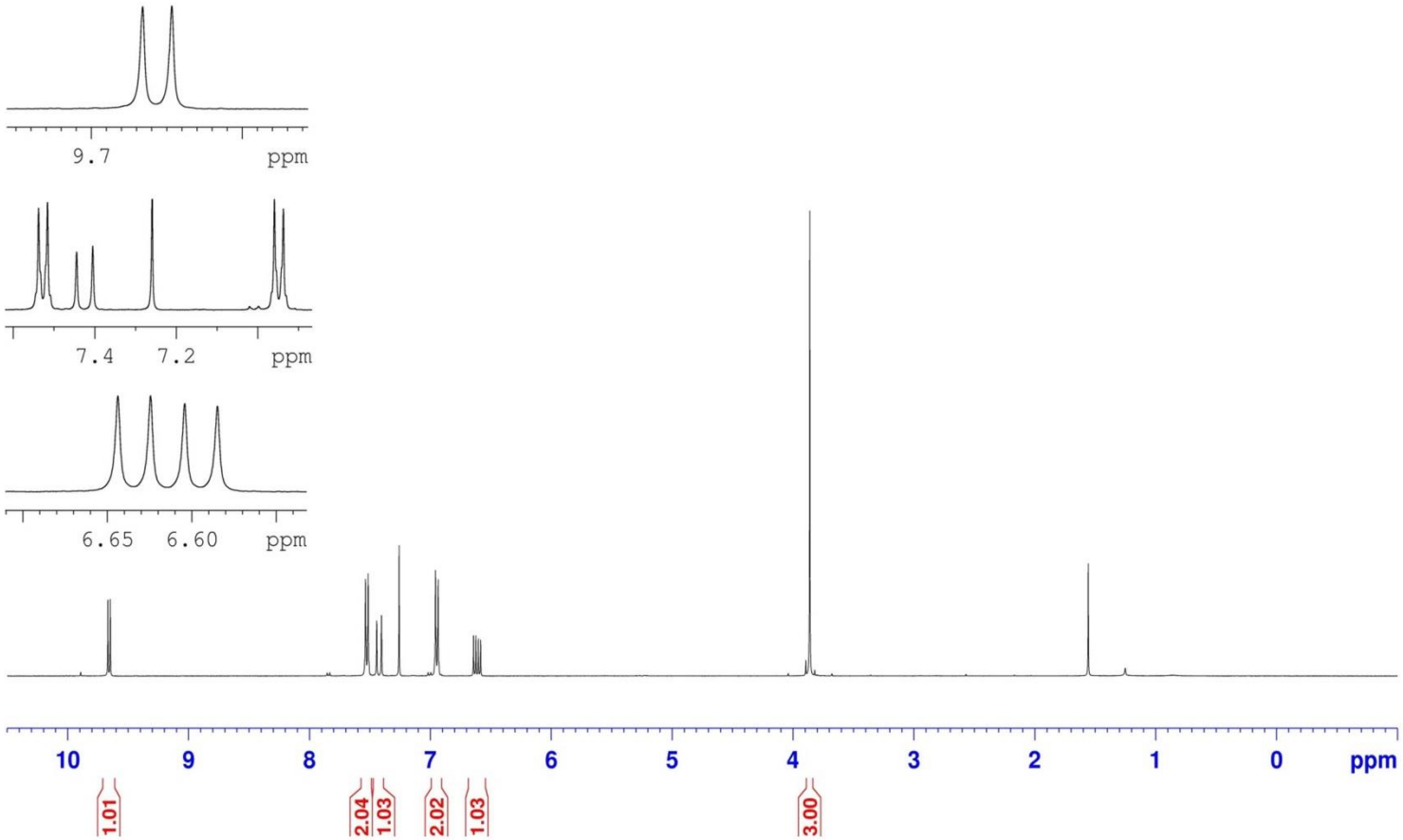
trans–4–methylcinnamaldehyde
300 MHz, CDCl3



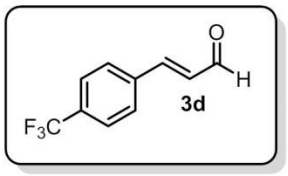


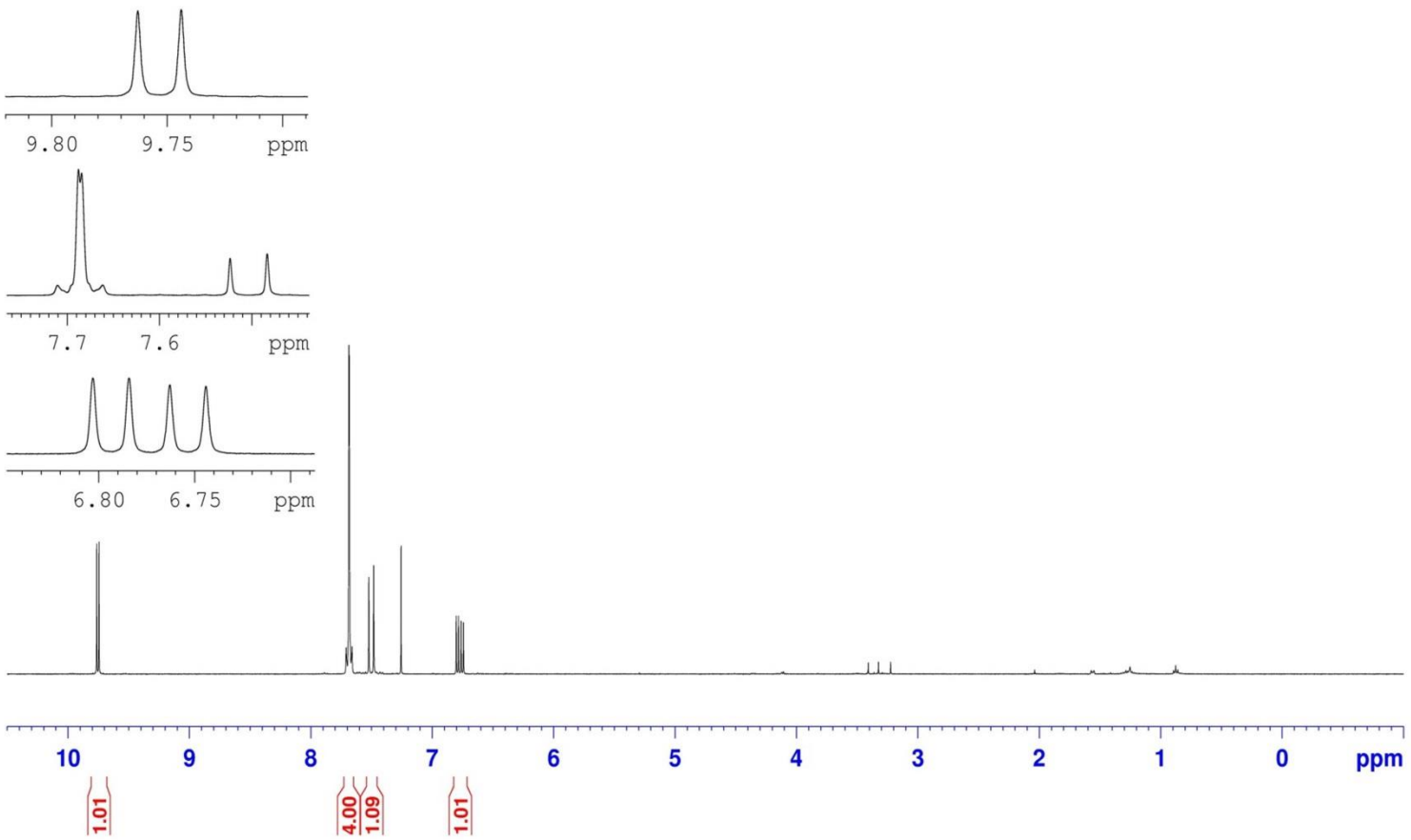
trans–4–methoxycinnamaldehyde
400 MHz, CDCl3



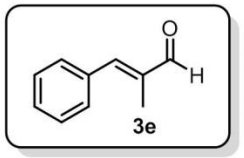


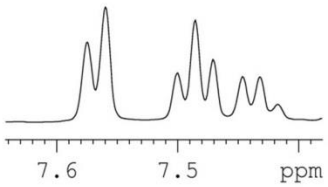
trans−4−(trifluoromethyl)cinnamaldehyde
400 MHz, CDCl3

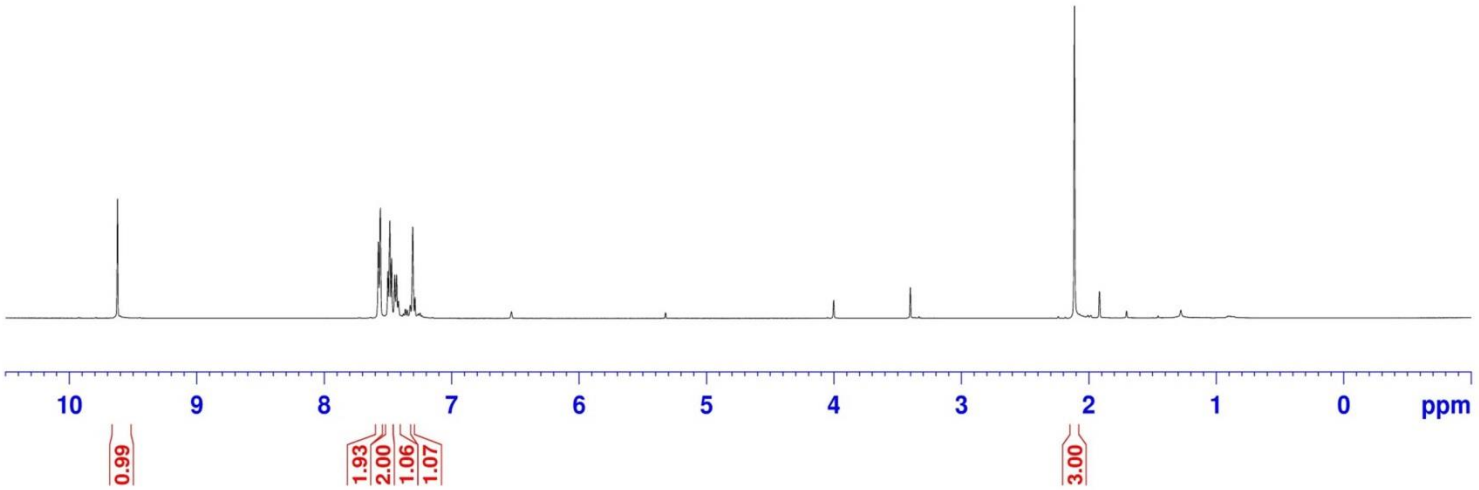




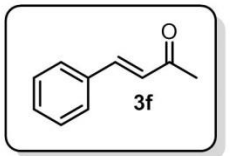
alpha–methylcinnamaldehyde
500 MHz, CDCl3

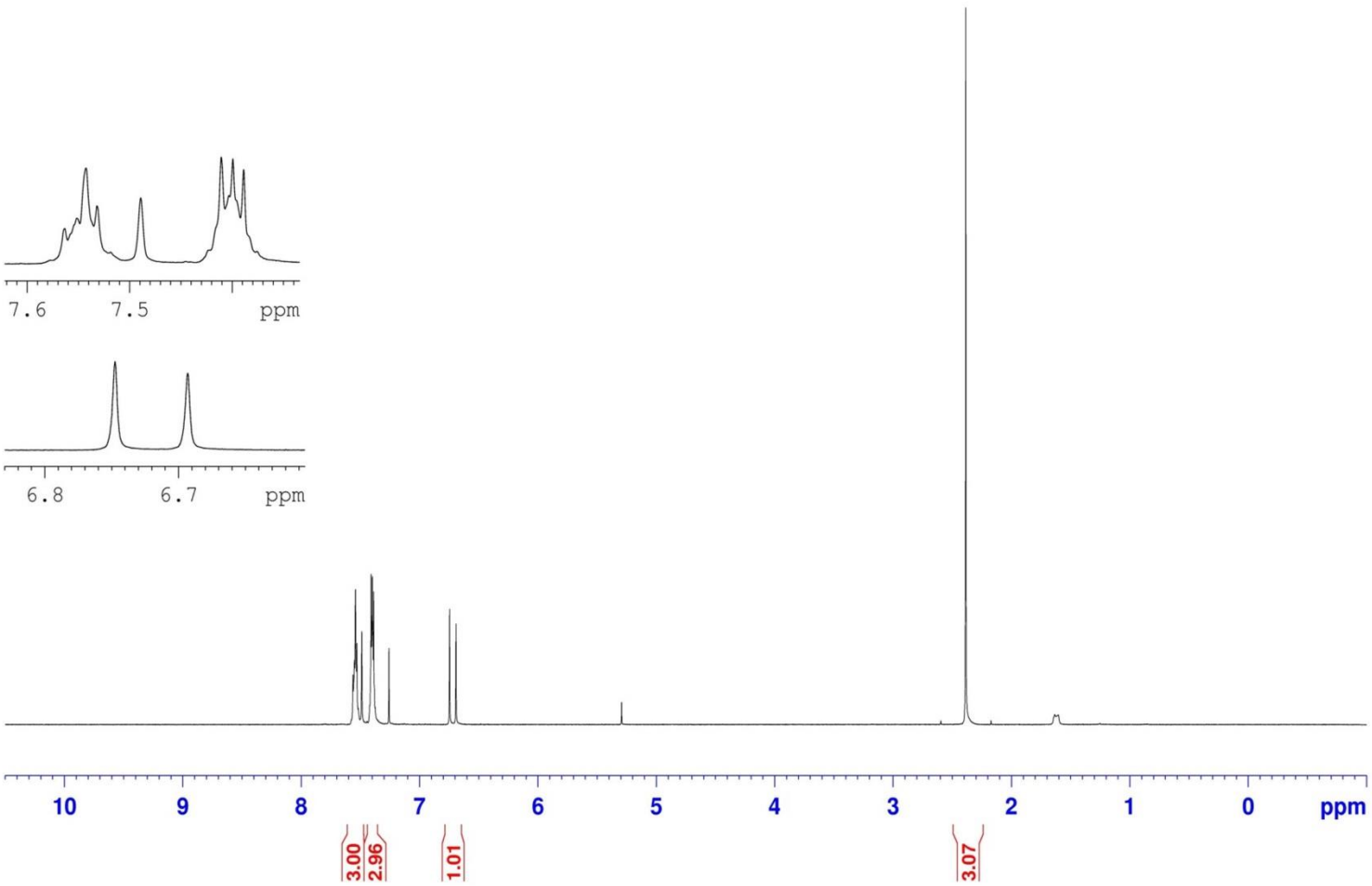




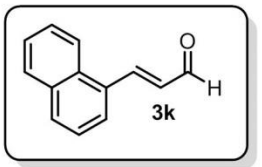


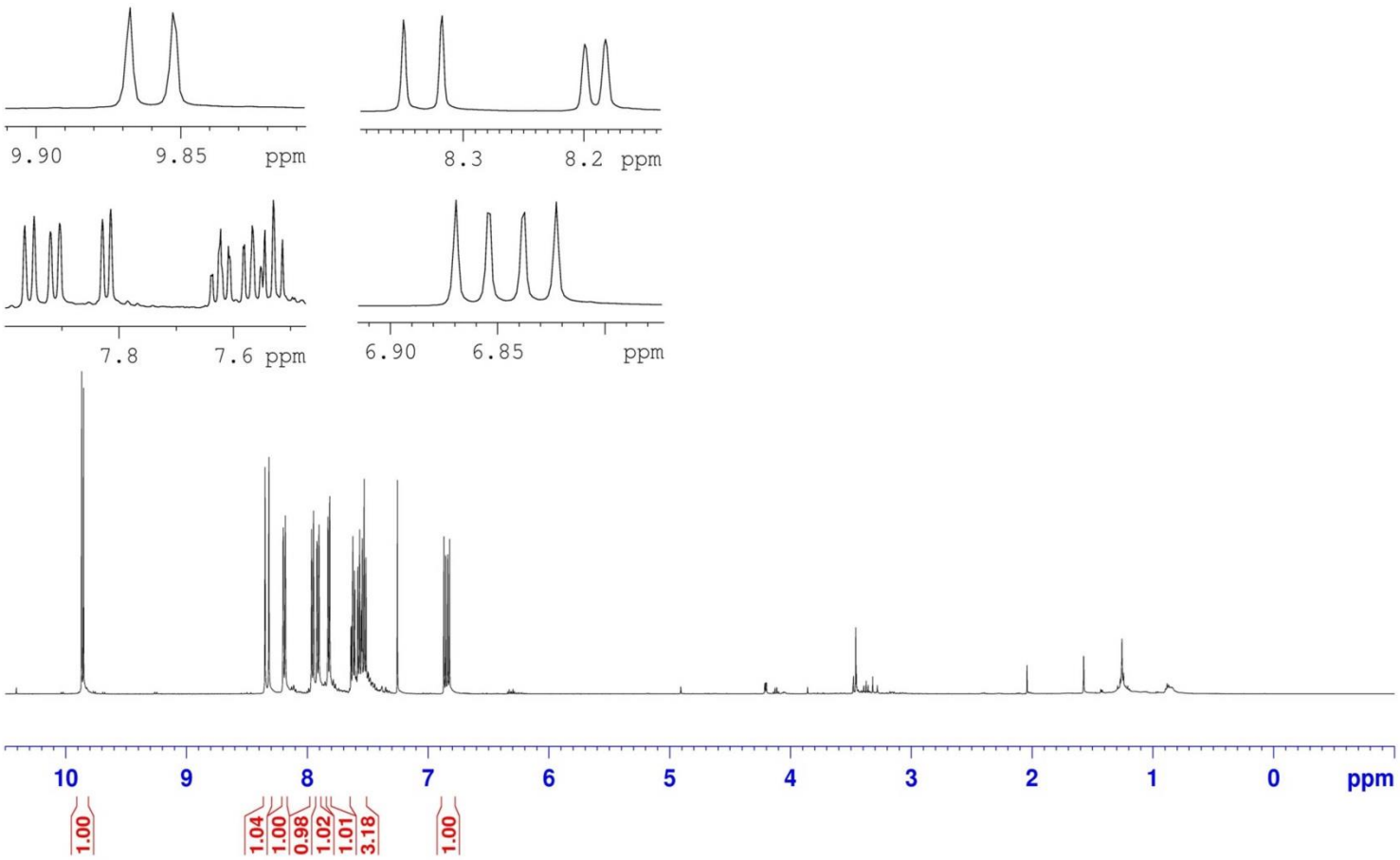
(E)–4–phenylbut–3–en–2–one
300 MHz, CDCl3



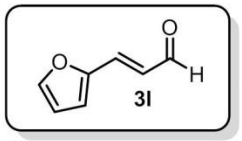


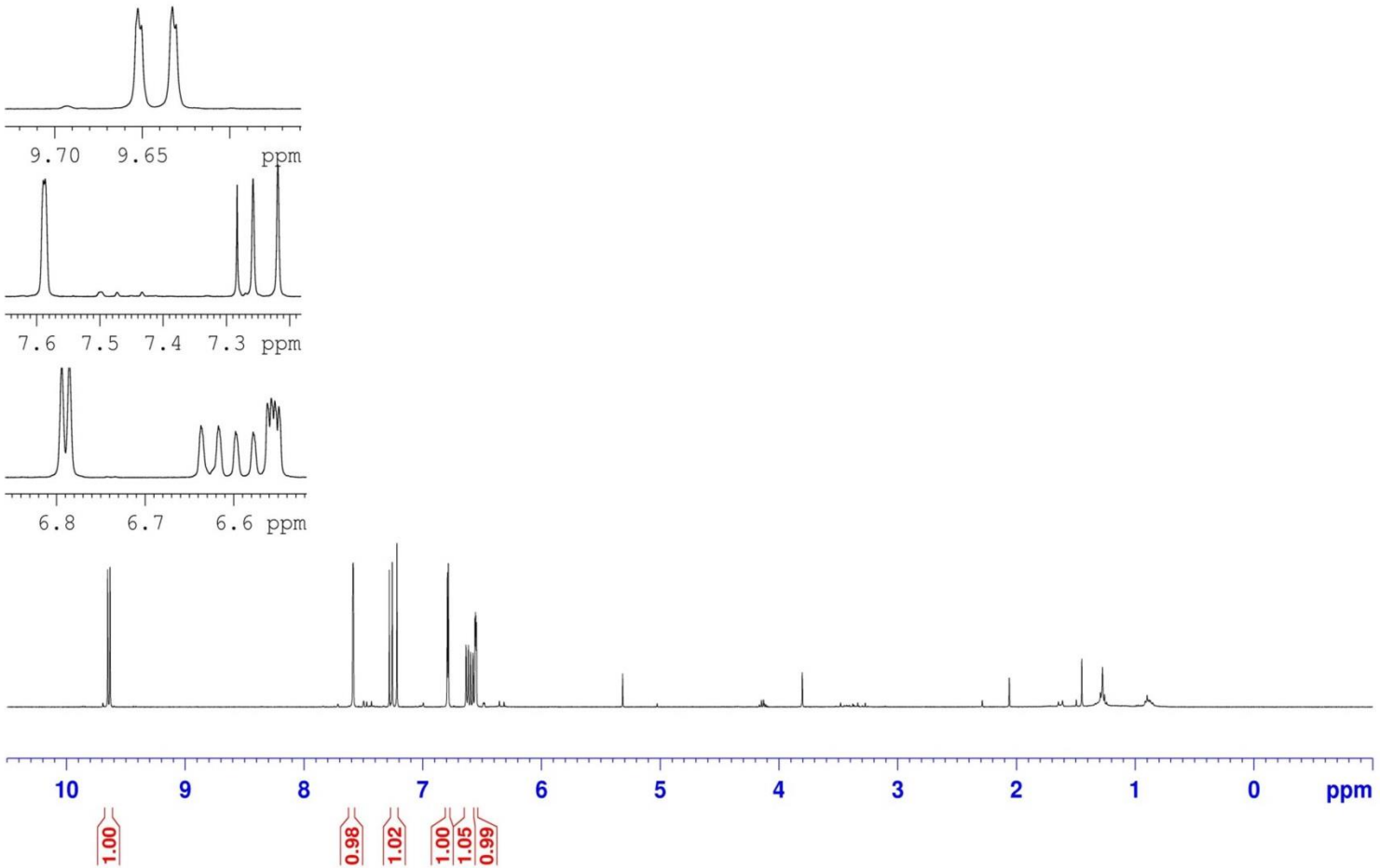
(E)−3−(naphthalen−1−yl)acrylaldehyde
500 MHz, CDCl3



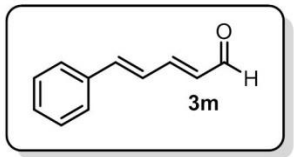


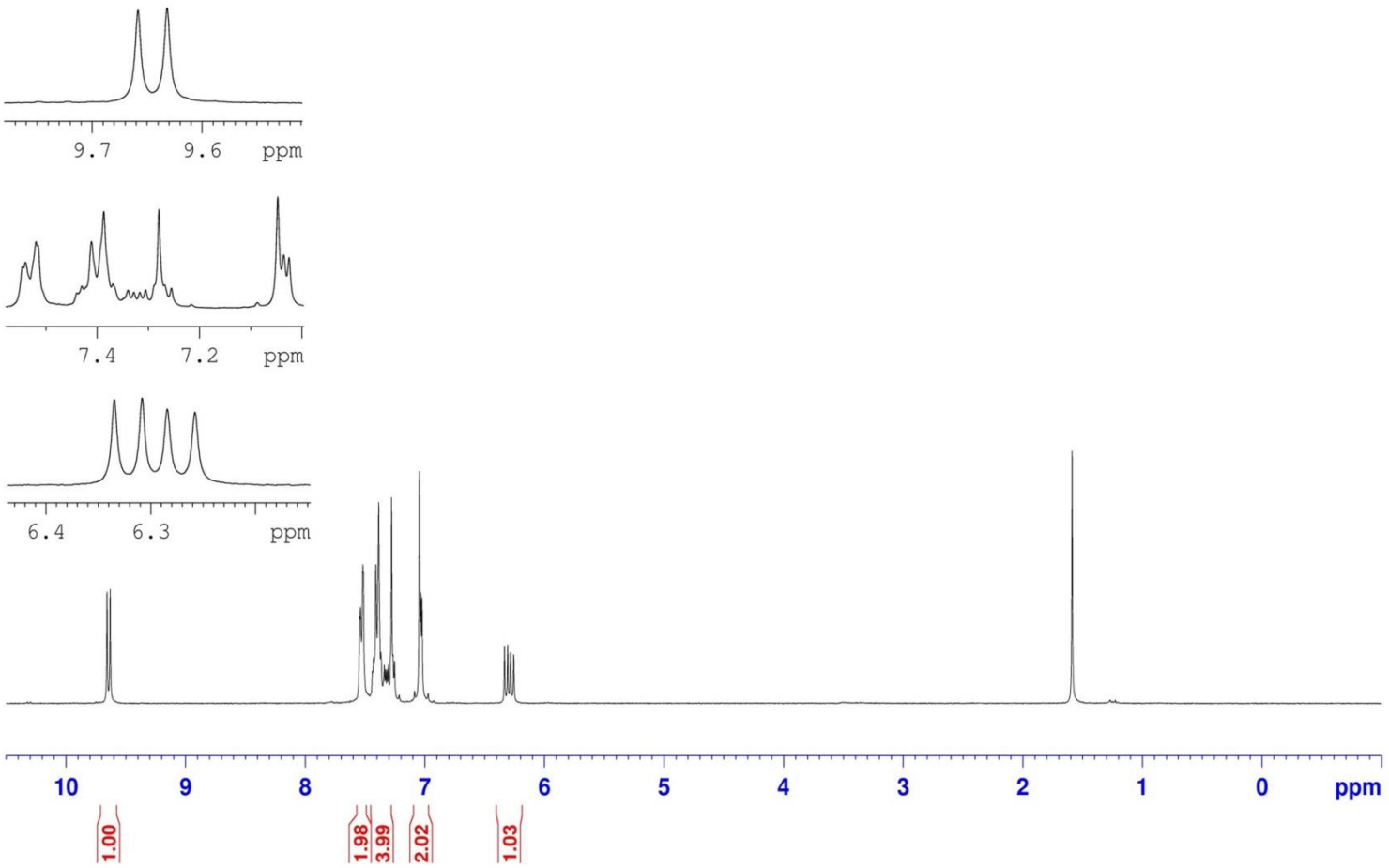
(E)−3−(furan−2−yl)acrylaldehyde
400 MHz, CDCl3



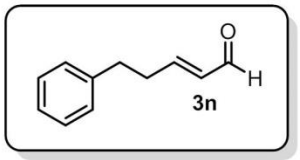


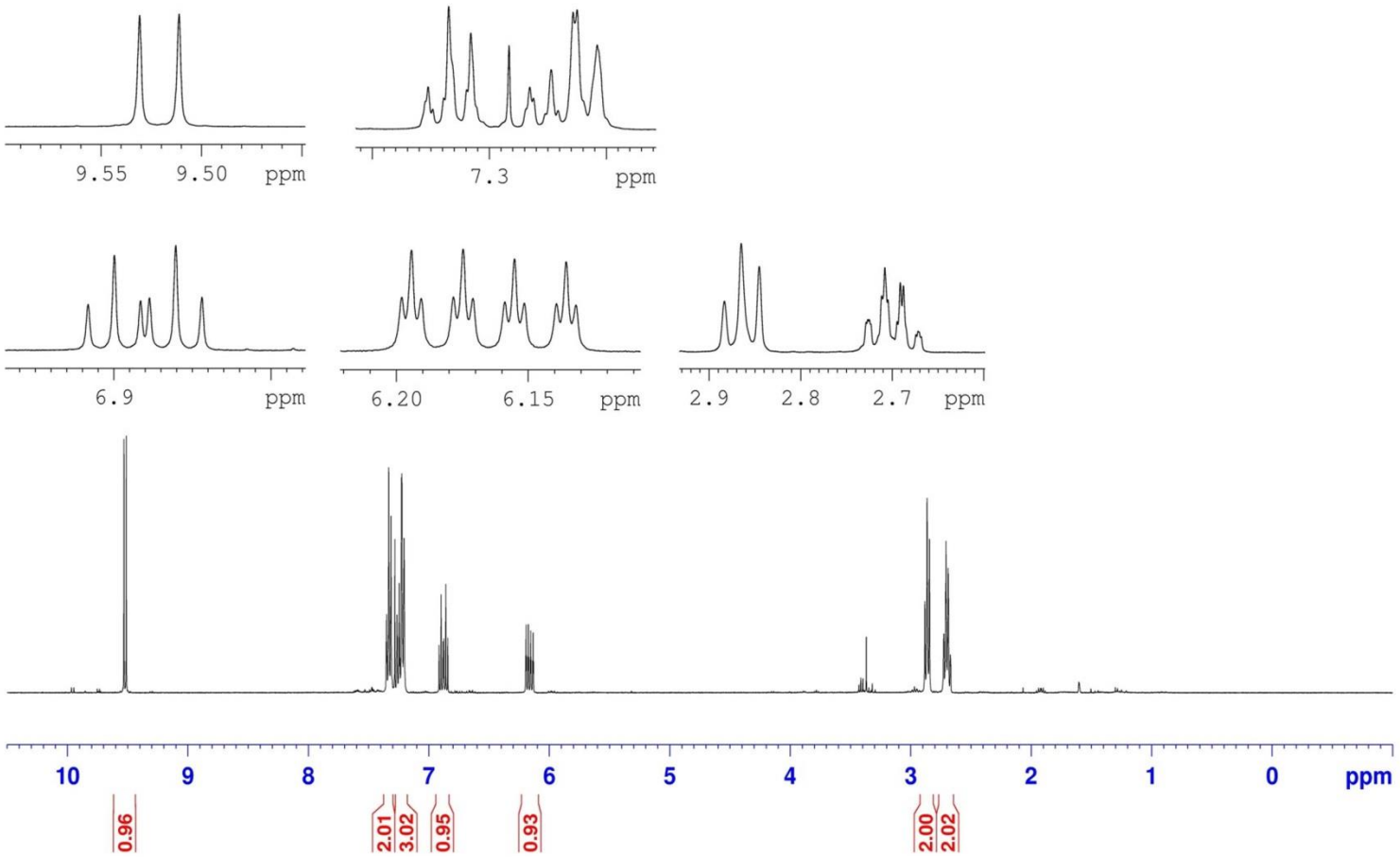
(2E,4E)−5−phenylpenta−2,4−dienal
300 MHZ, CDCl3



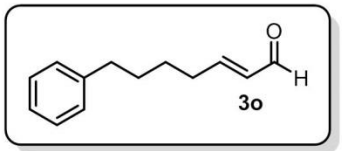


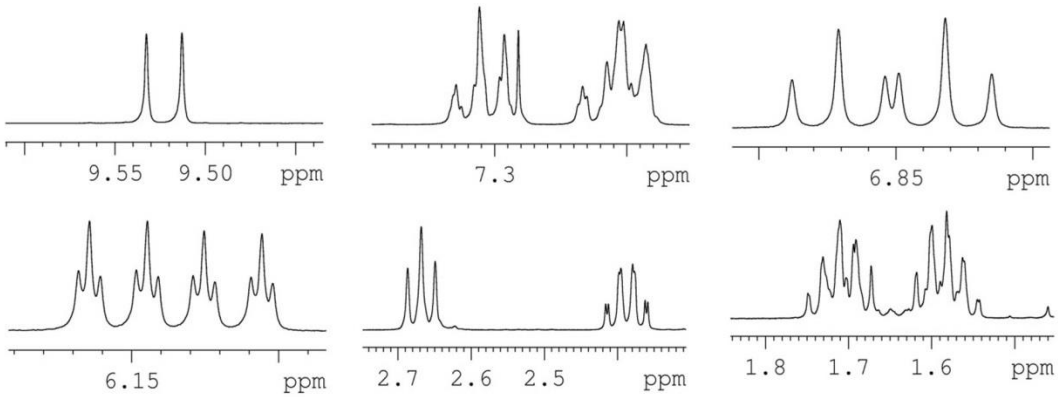
(E)−5−phenylpent−2−enal
400 MHz, CDCl3

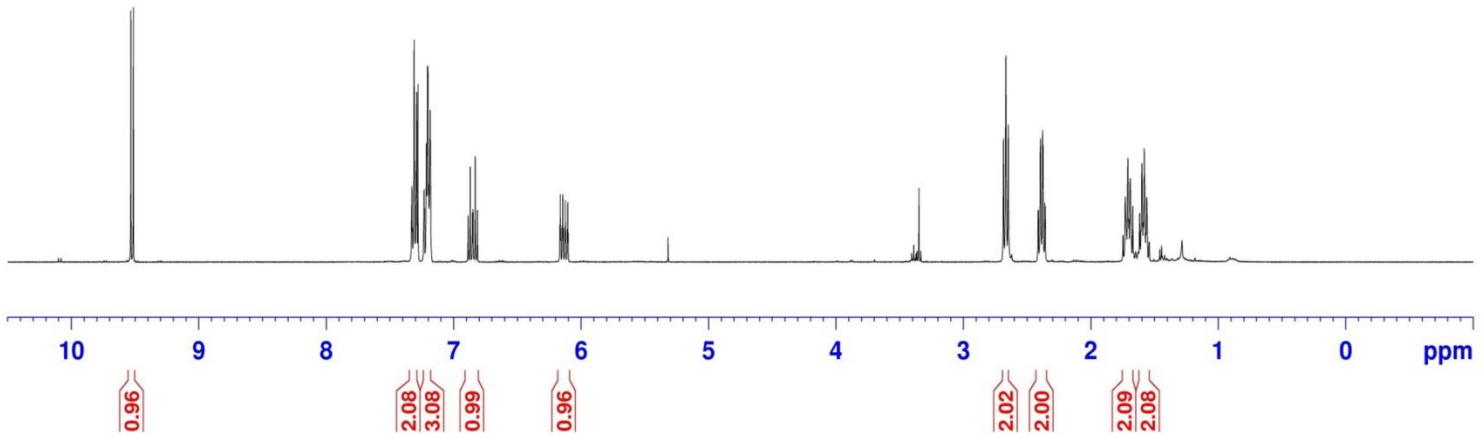


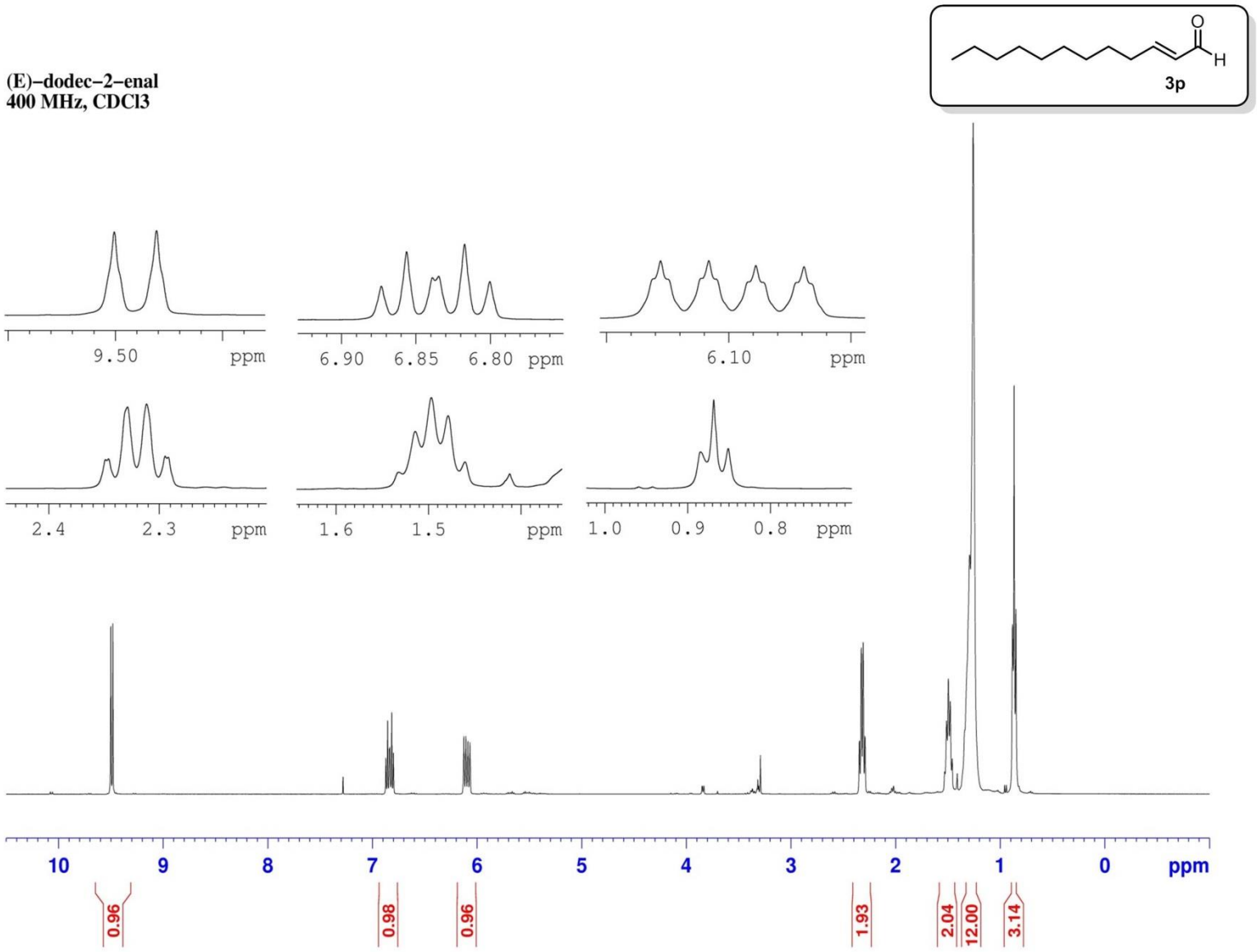


(E)−7−phenylhept−2−enal
400 MHz, CDCl3

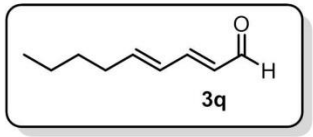


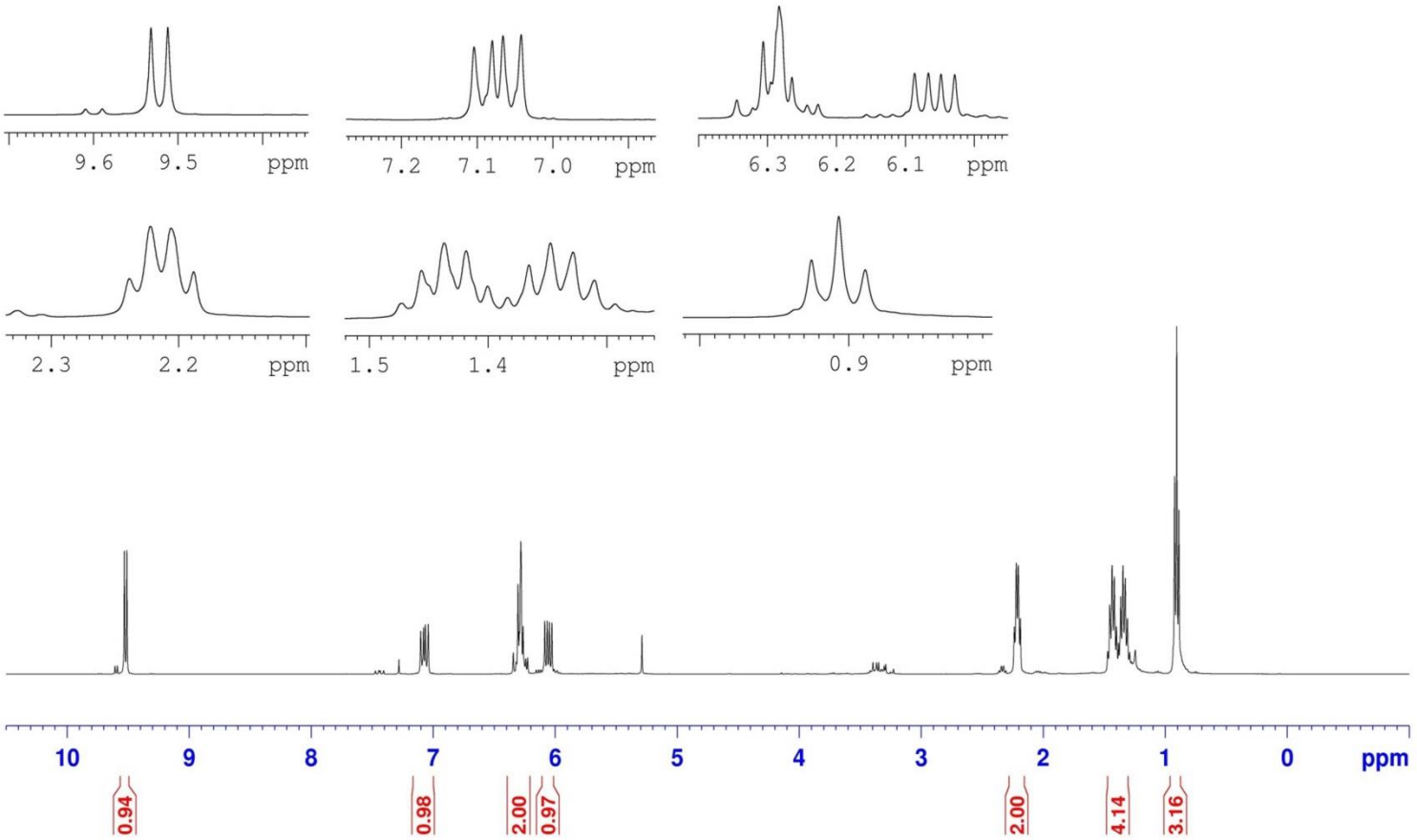




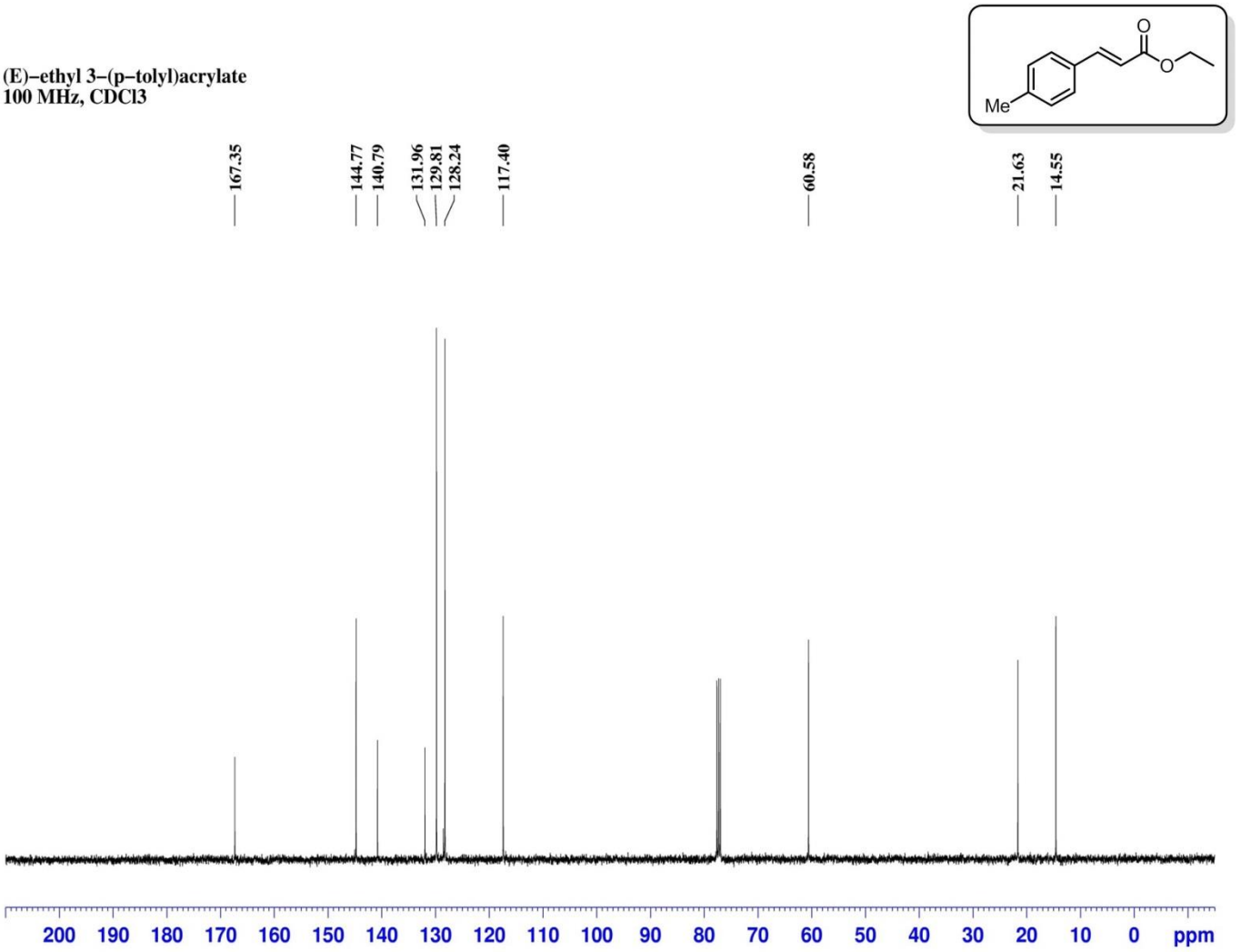

(E)−dodec−2−enal
400 MHz, CDCl3
O
H
3p
9.50
ppm
6.90
6.85
6.80
ppm
6.10
ppm
2.4
2.3
ppm
1.6
1.5
ppm
1.0
0.9
0.8
ppm
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
0.98
0.96
1.93
2.04
12.00
3.14

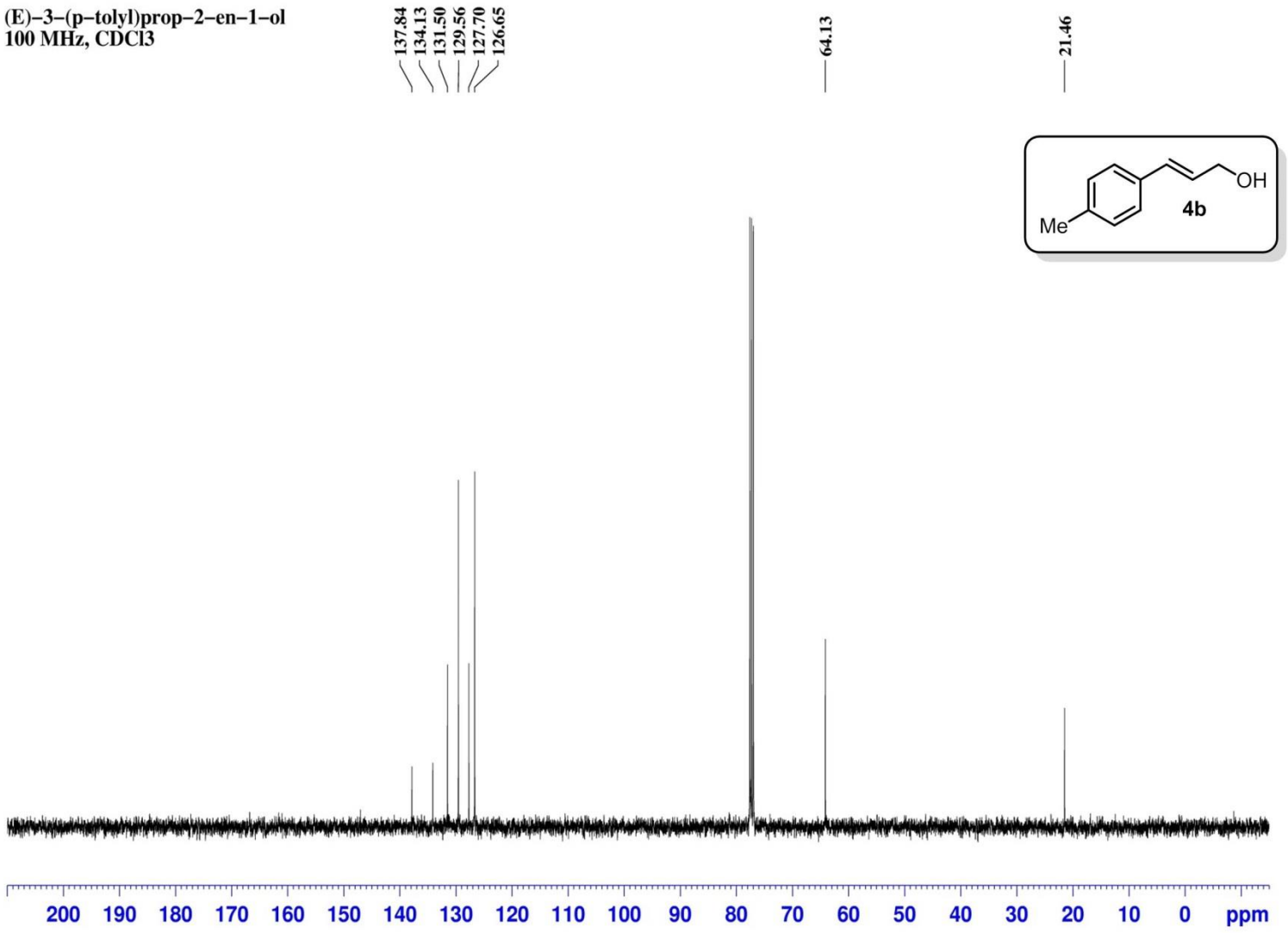
(2E,4E)−nona−2,4−dienal
400 MHz, CDCl3



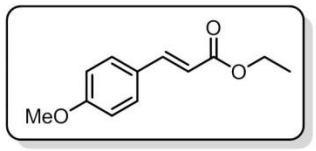


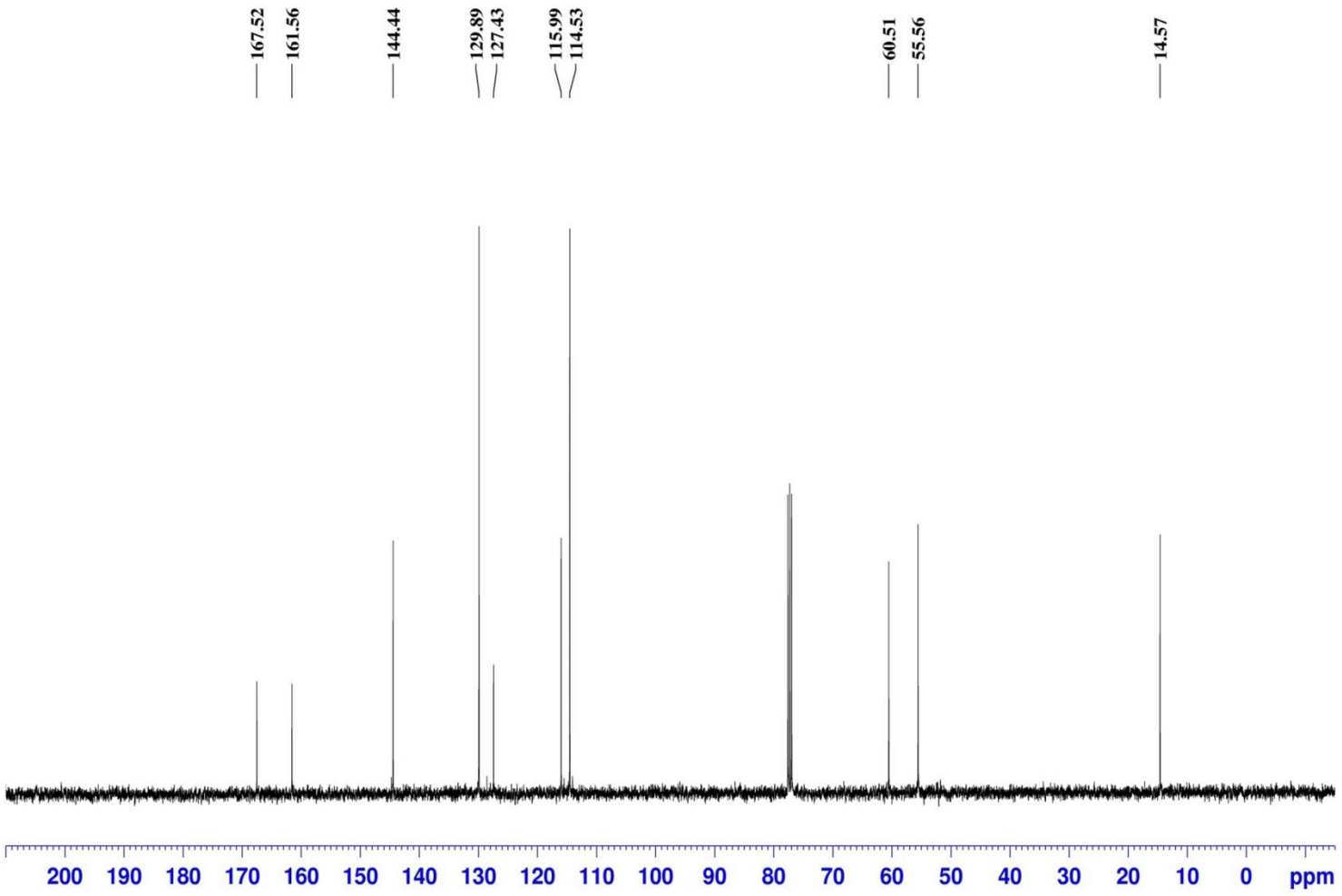
^{13}C NMR Spectra of Synthesized Compounds



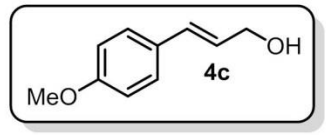

(E)−3−(p−tolyl)prop−2−en−1−ol
100 MHz, CDCl3
137.84
134.13
131.50
129.56
127.70
126.65
64.13
21.46
OH
Me
4b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

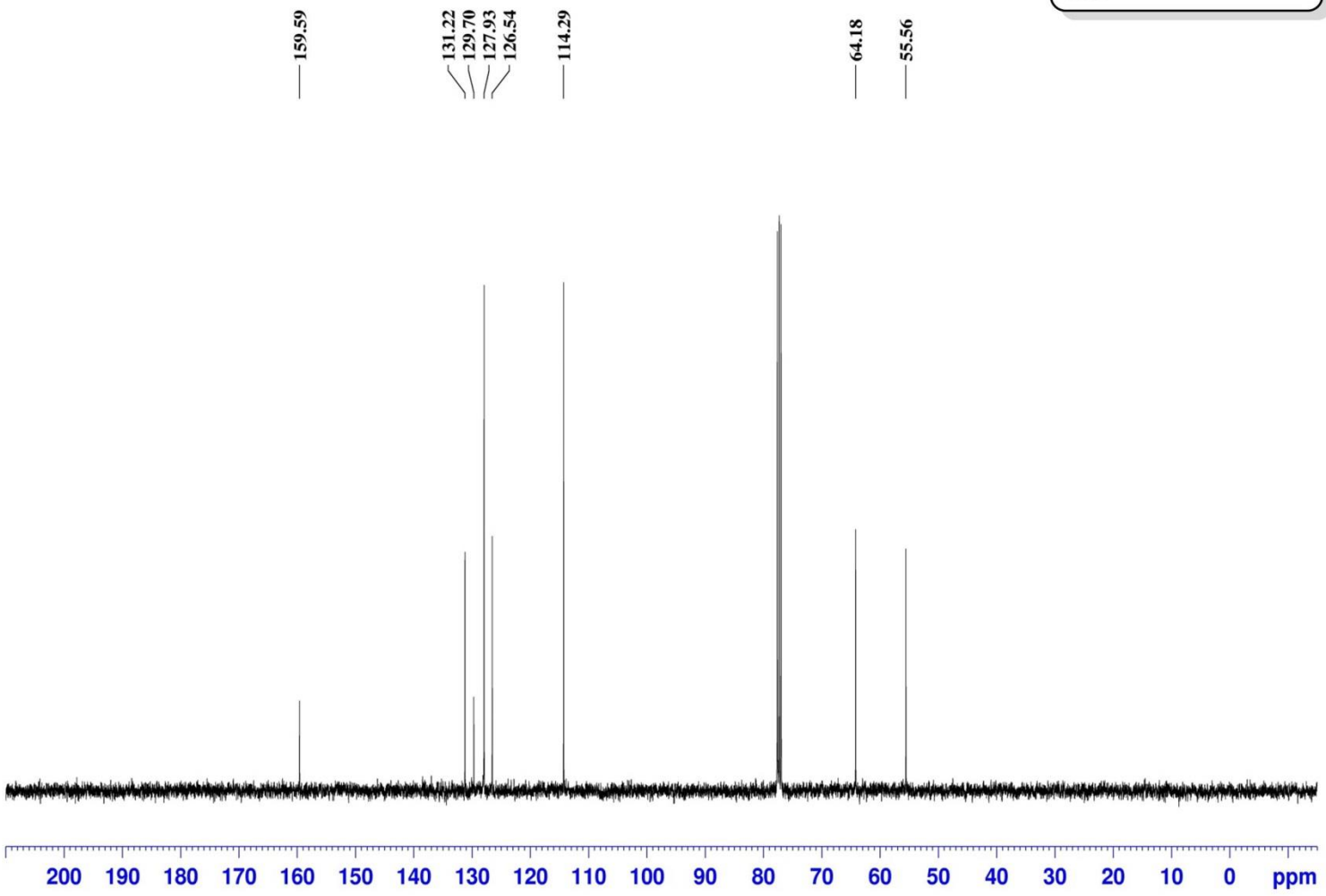
(E)–ethyl 3–(4–methoxyphenyl)acrylate
100 MHz, CDCl3



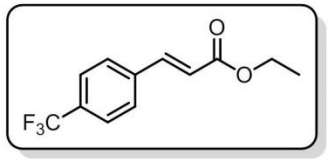


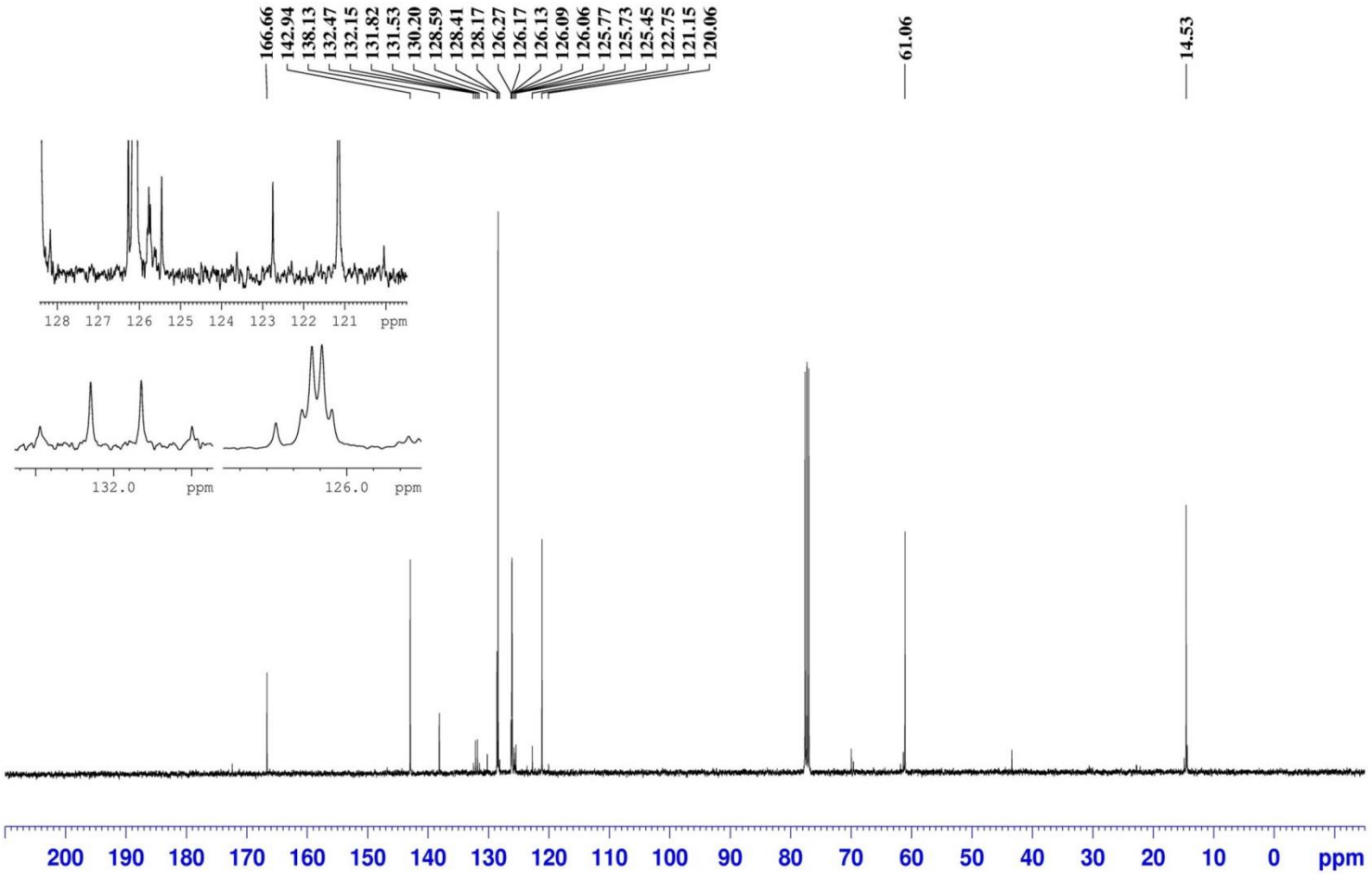
(E)−3−(4−methoxyphenyl)prop−2−en−1−ol
100 MHz, CDCl3



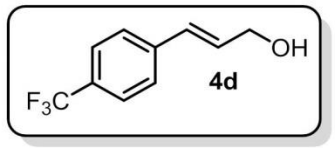


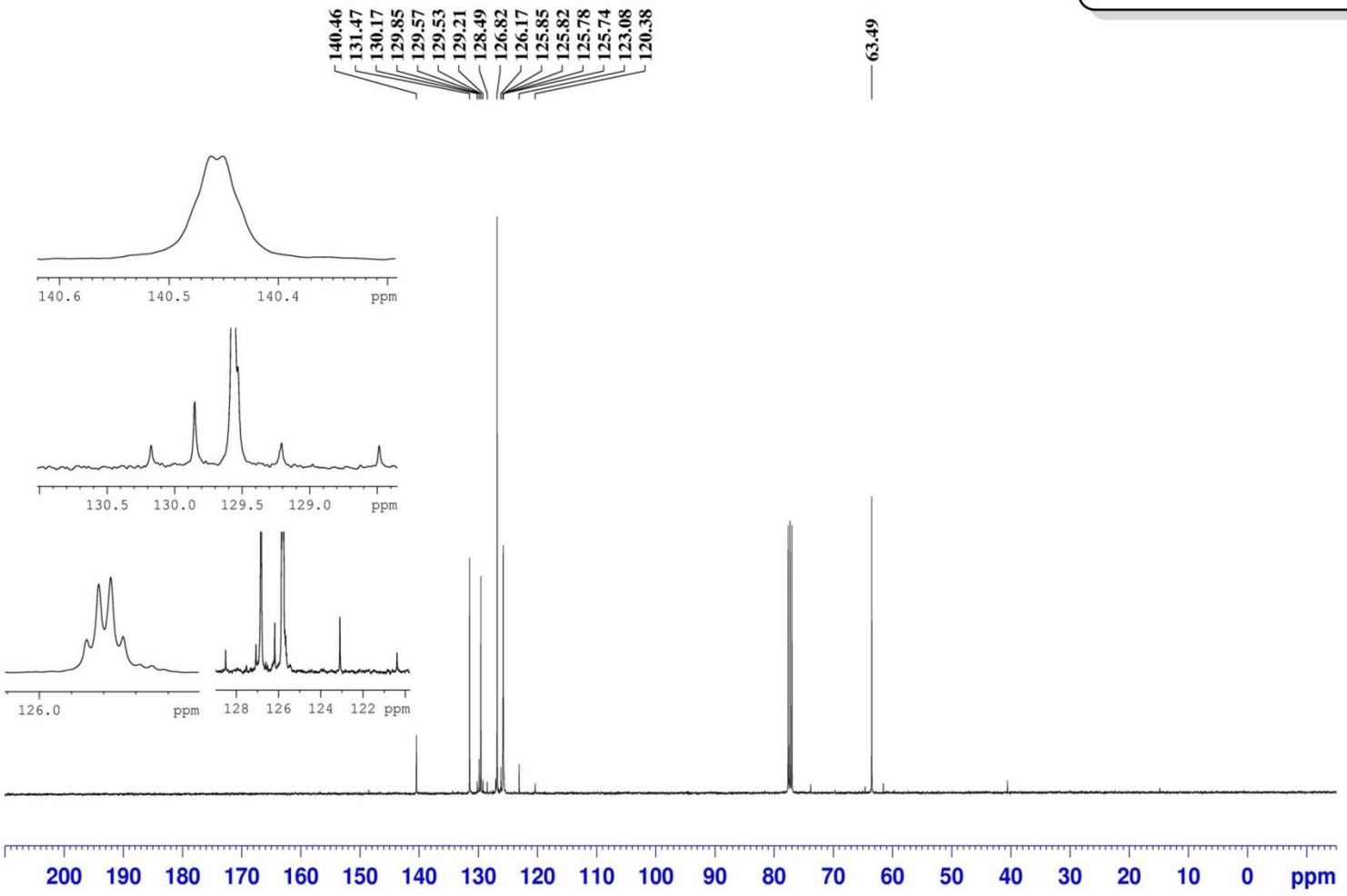
(E)−ethyl 3−(4−(trifluoromethyl)phenyl)acrylate
100 MHz, CDCl3

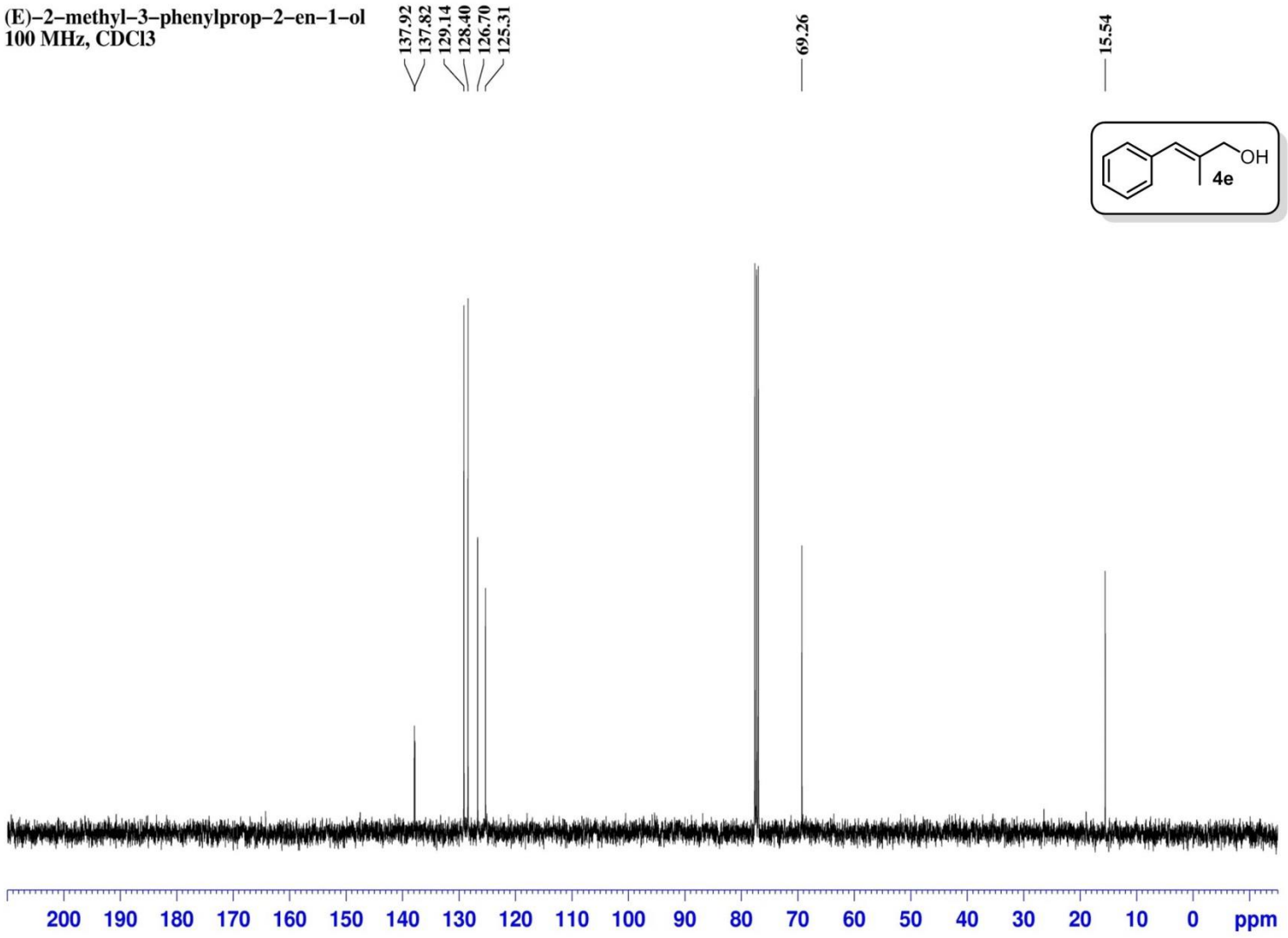




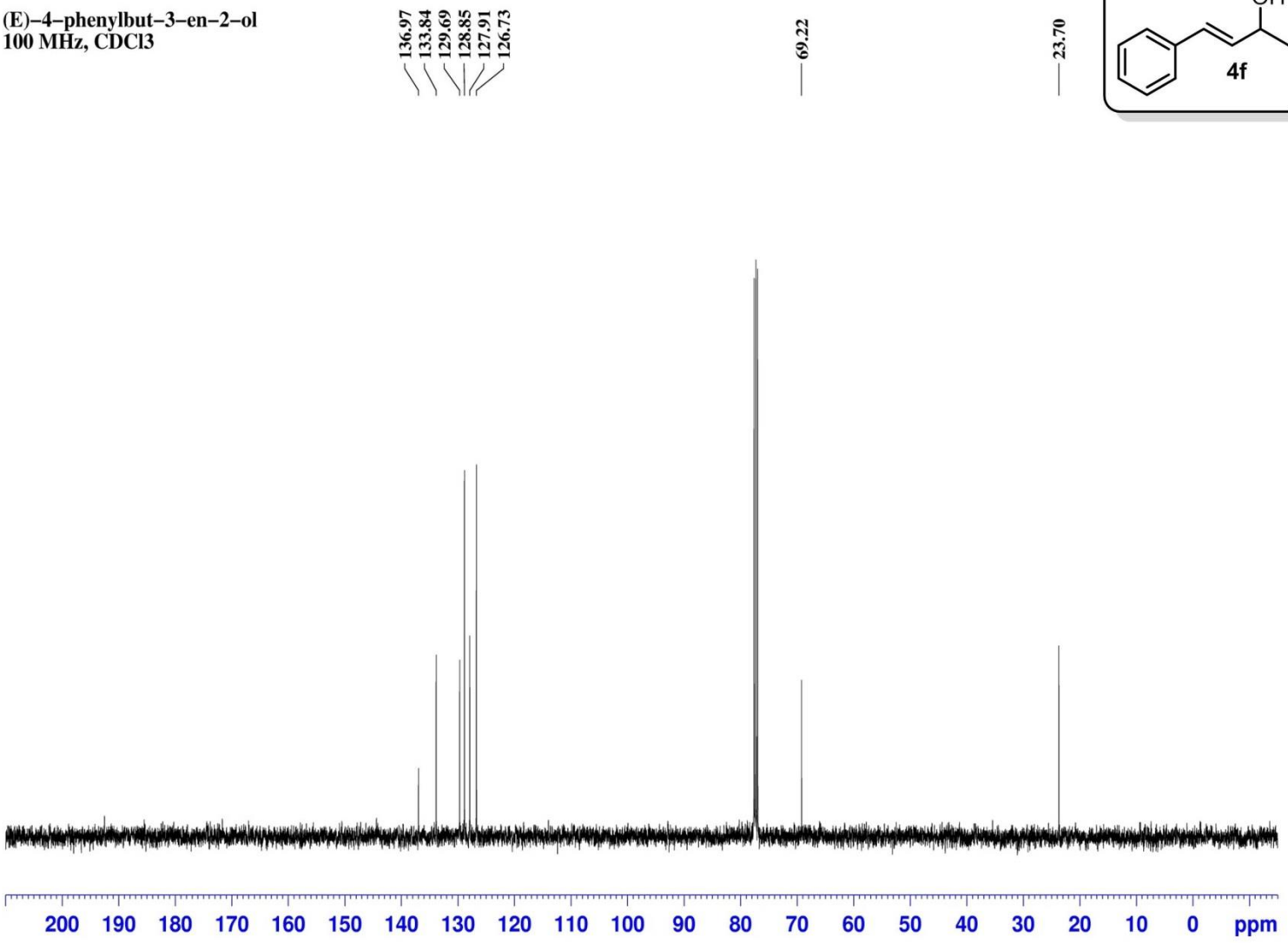
(E)−3−(4−(trifluoromethyl)phenyl)prop−2−en−1−ol
100 MHz, CDCl3



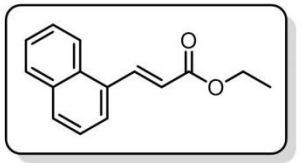


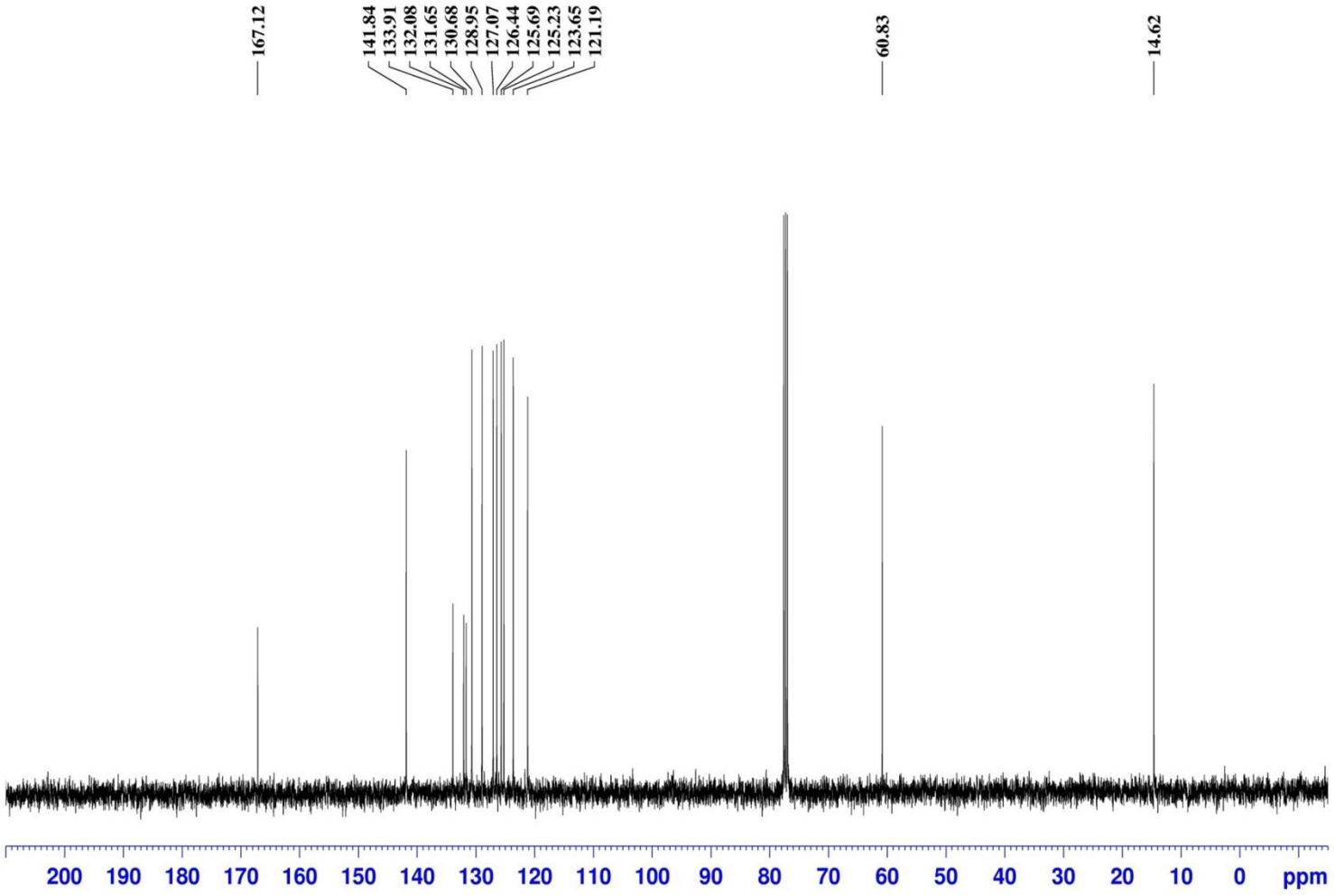

(E)–2–methyl–3–phenylprop–2–en–1–ol
100 MHz, CDCl3
137.92
137.82
129.14
128.40
126.70
125.31
69.26
15.54
OH
4e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

(E)−4−phenylbut−3−en−2−ol
100 MHz, CDCl3

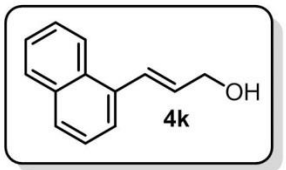


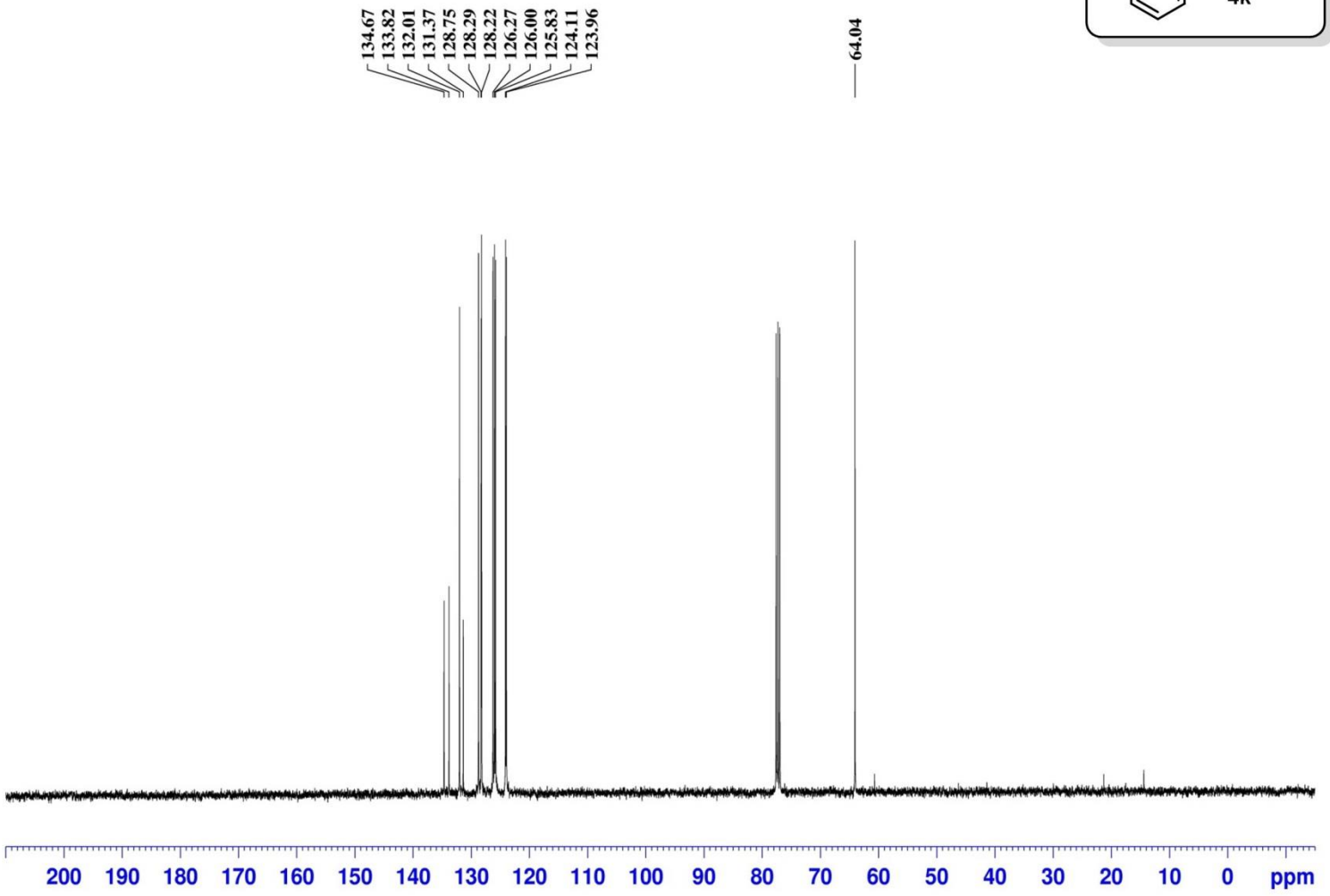
(E)–ethyl 3–(naphthalen–1–yl)acrylate
100 MHz, CDCl3



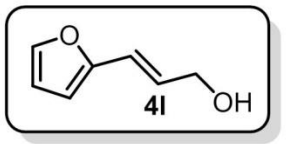


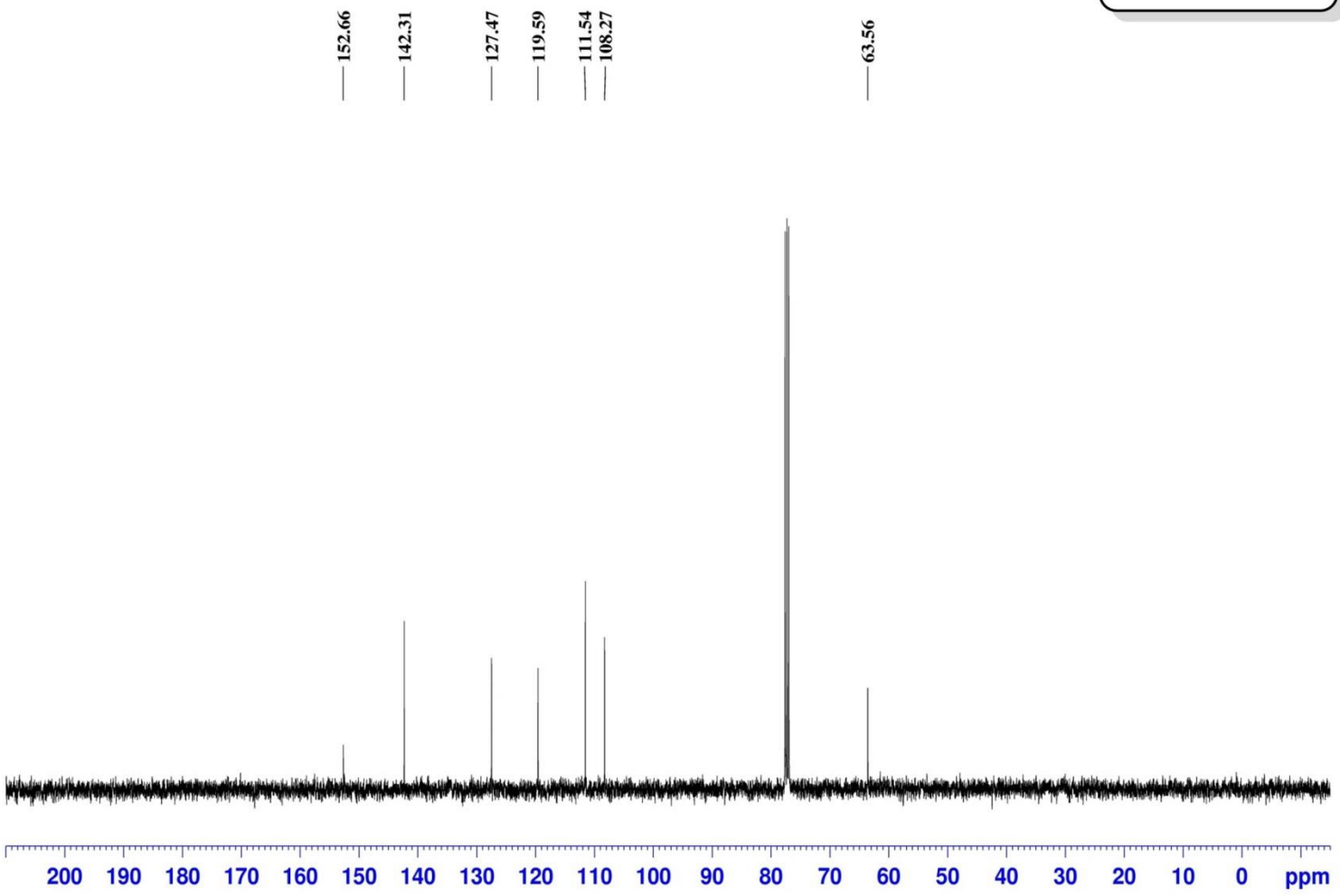
(E)−3−(naphthalen−1−yl)prop−2−en−1−ol
100 MHz, CDCl3



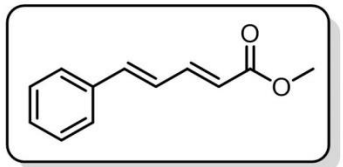


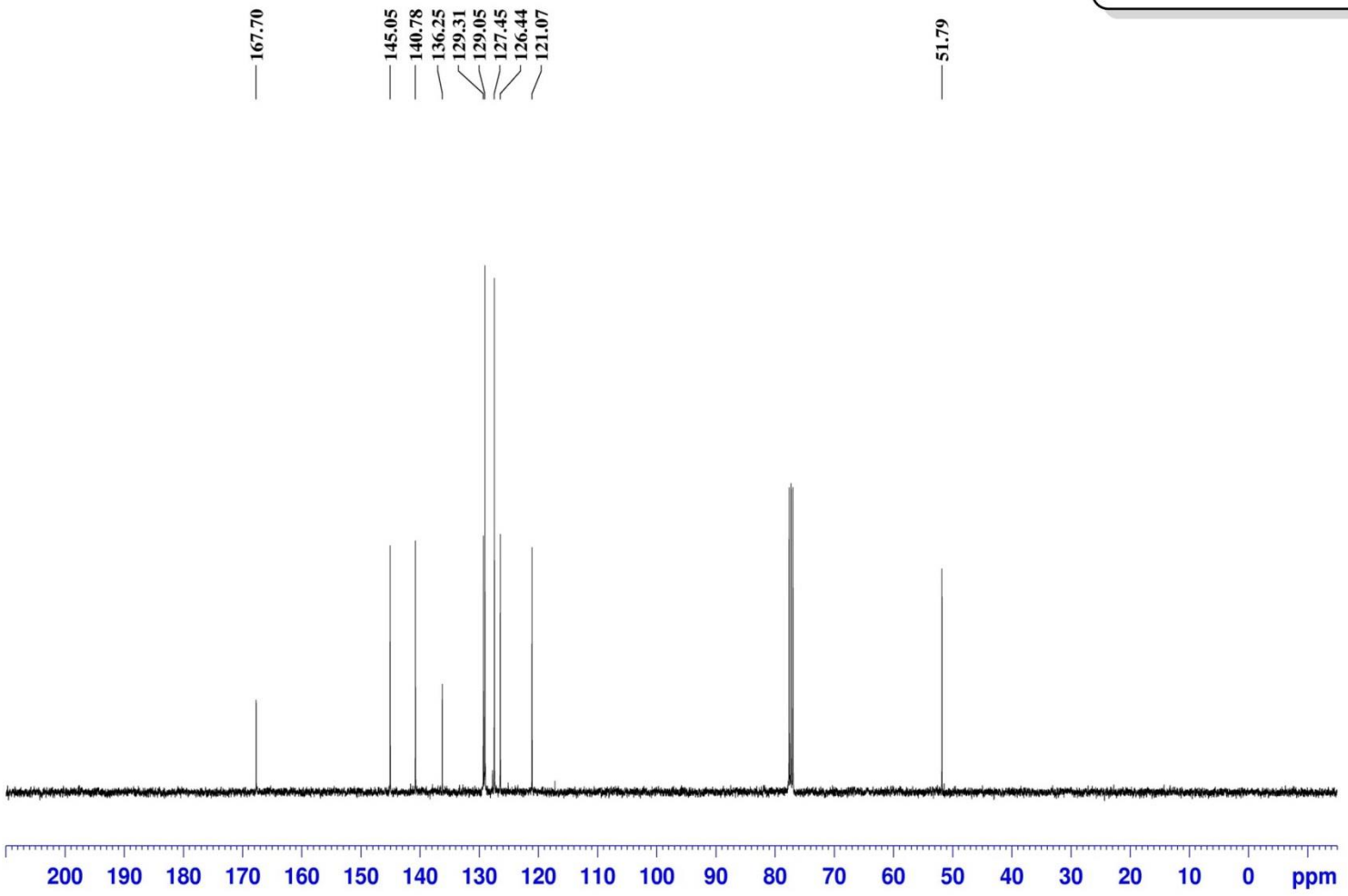
(E)−3−(furan−2−yl)prop−2−en−1−ol
100 MHz, CDCl3



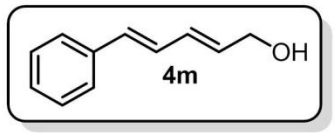


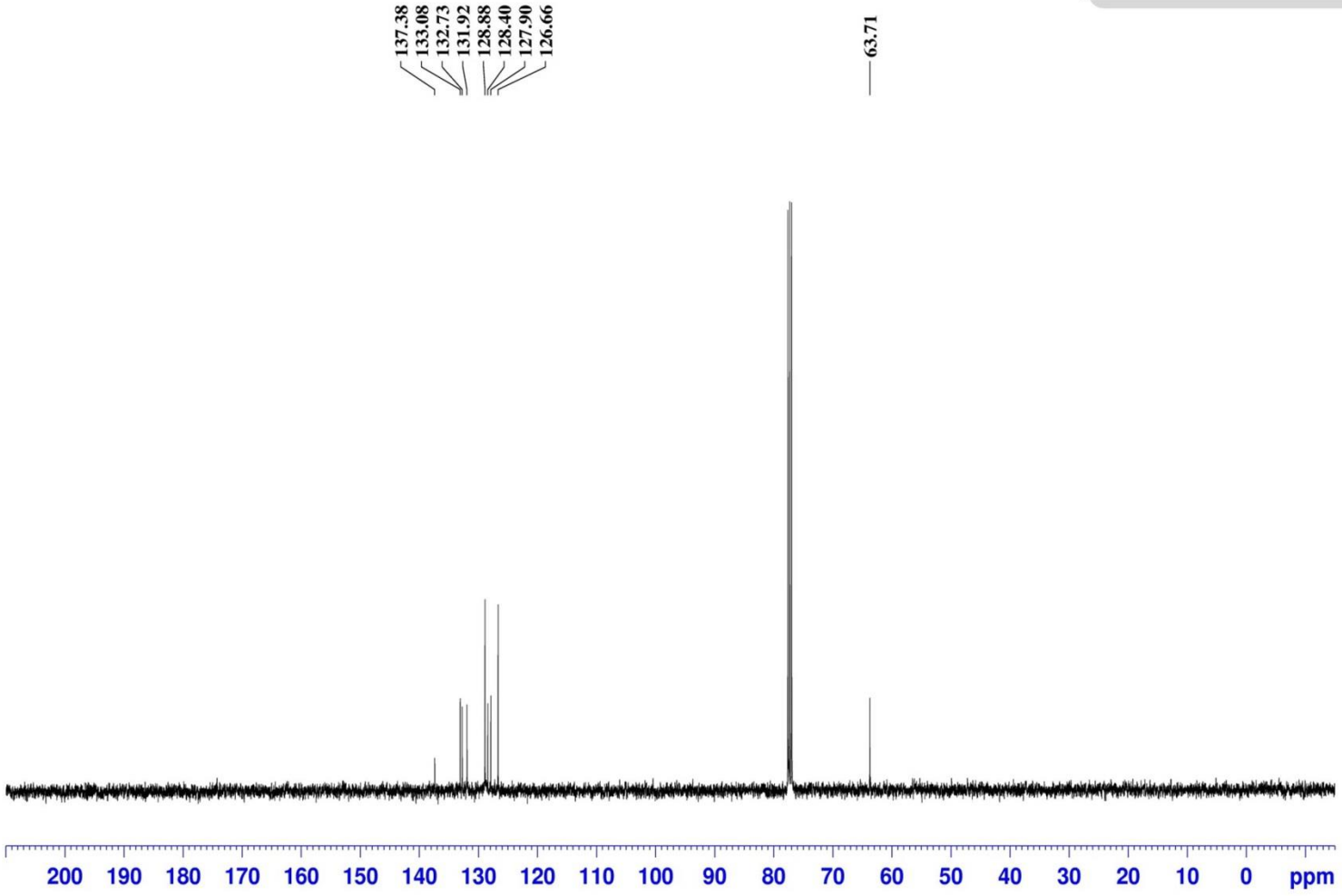
(2E,4E)−methyl 5−phenylpenta−2,4−dienoate
100 MHz, CDCl3

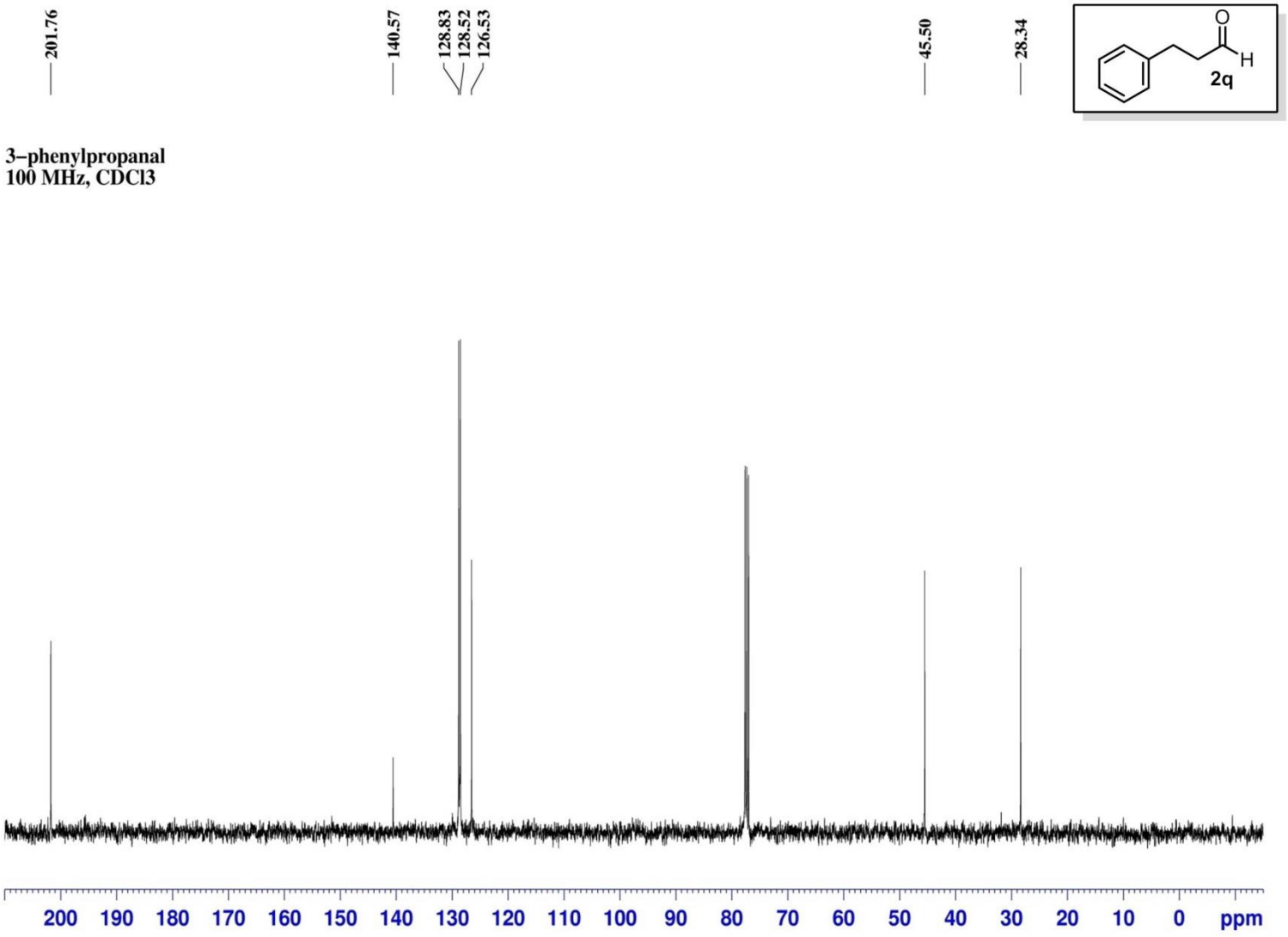




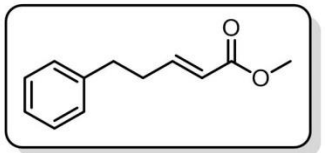
(2E,4E)−5−phenylpenta−2,4−dien−1−ol
100 MHz, CDCl3

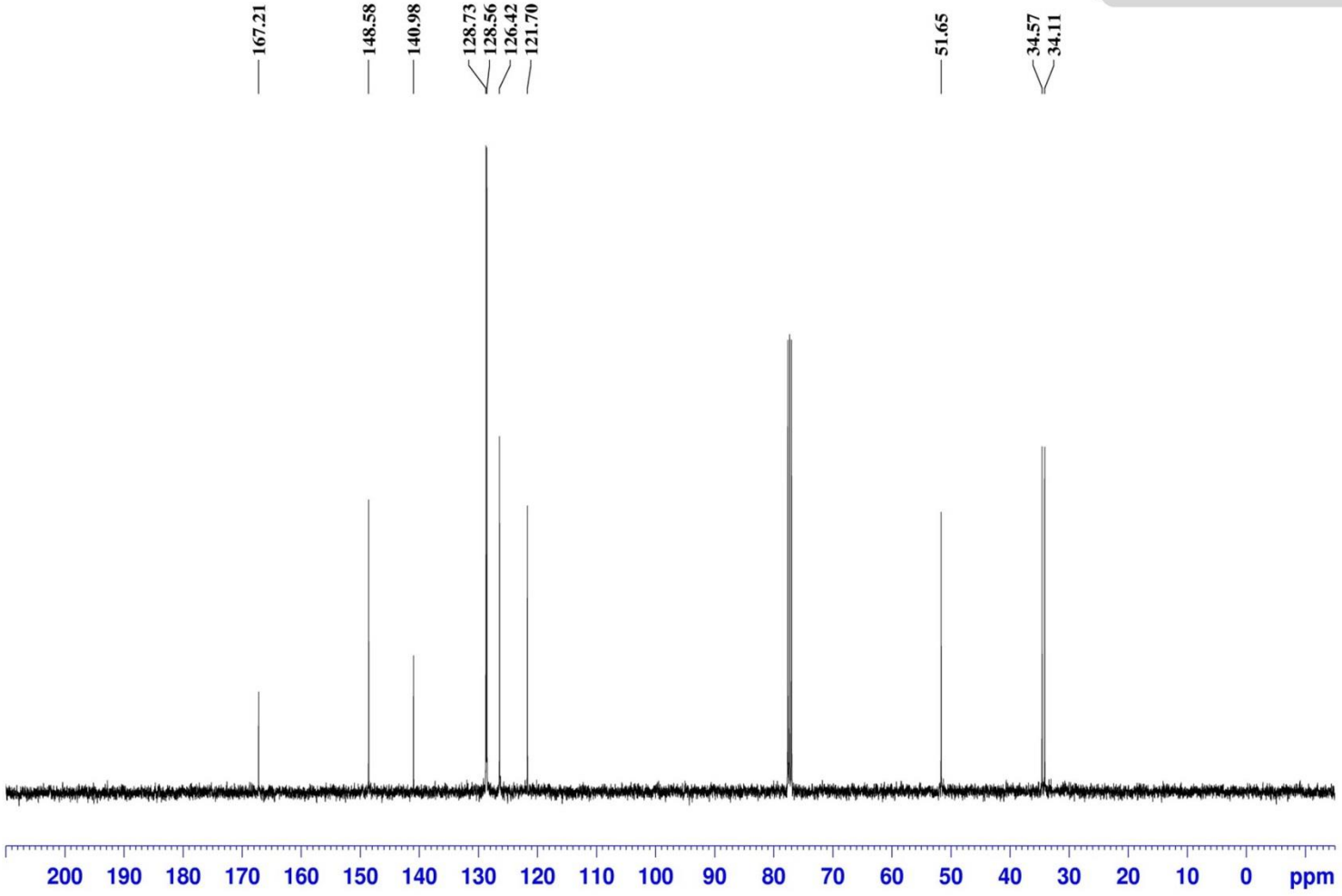




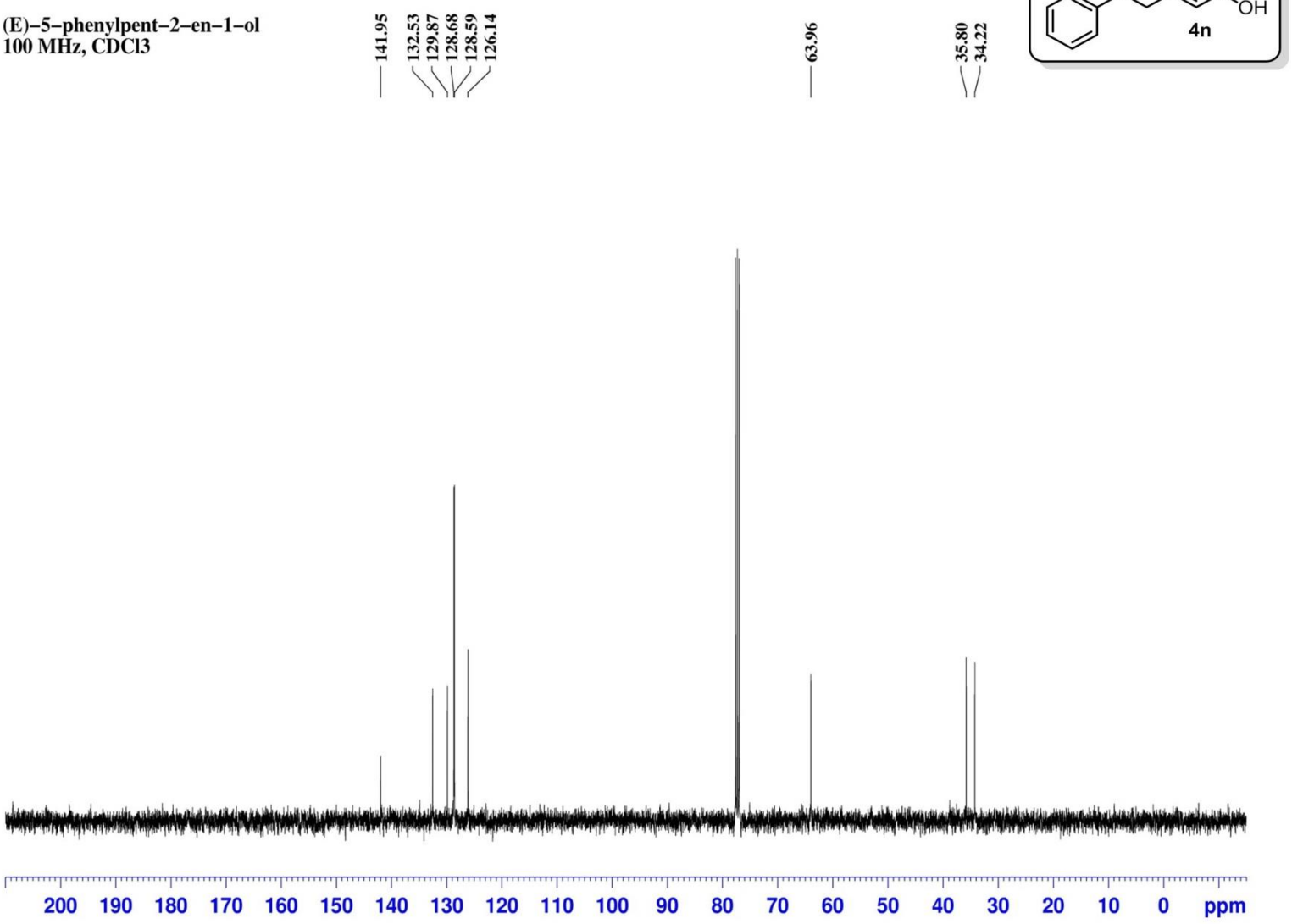

201.76
140.57
128.83
128.52
126.53
45.50
28.34
O
H
2q
3–phenylpropanal
100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

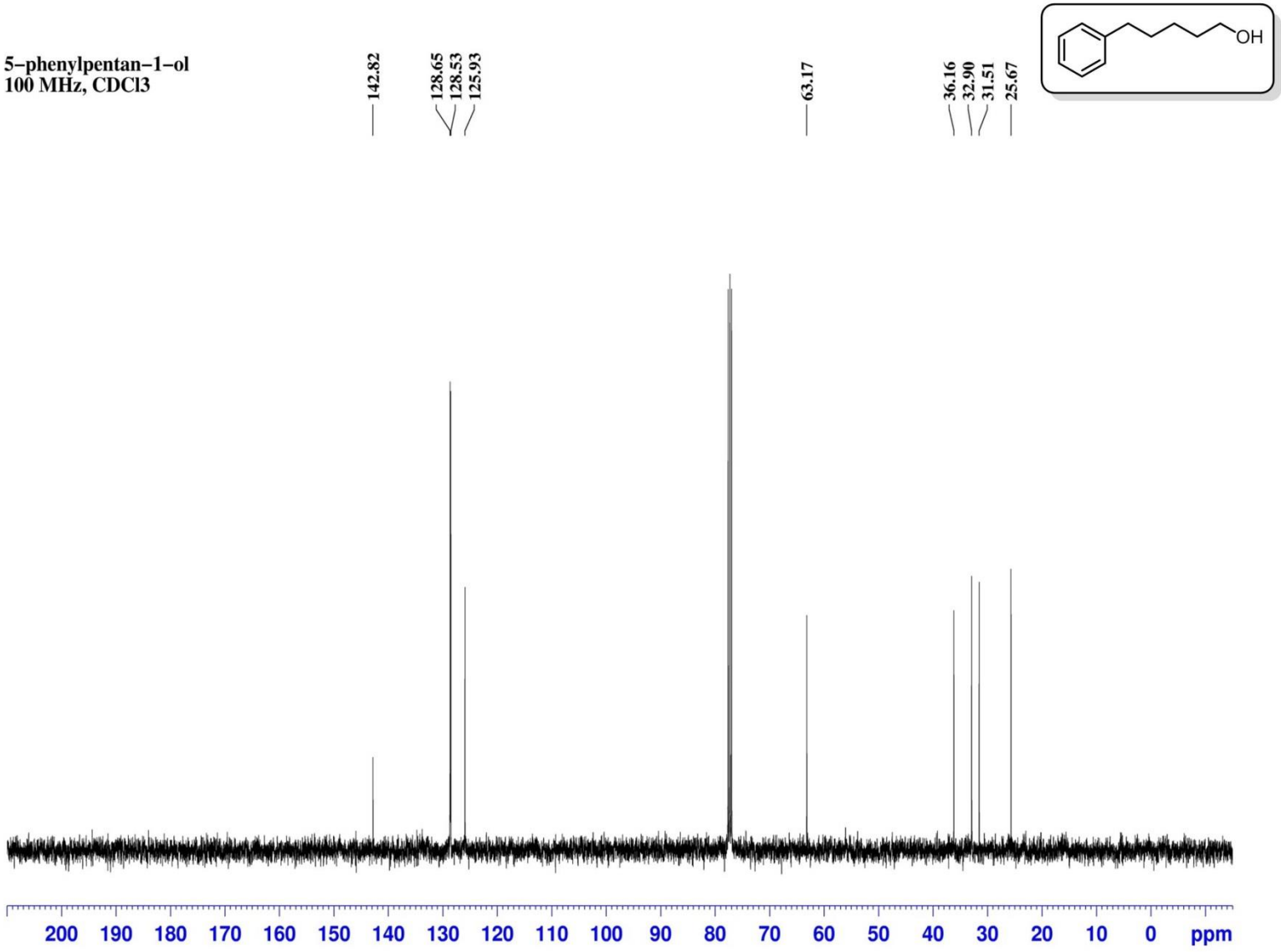
(E)−methyl 5−phenylpent−2−enoate
100 MHz, CDCl3



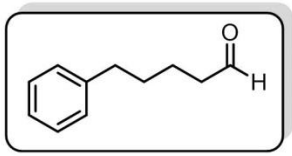


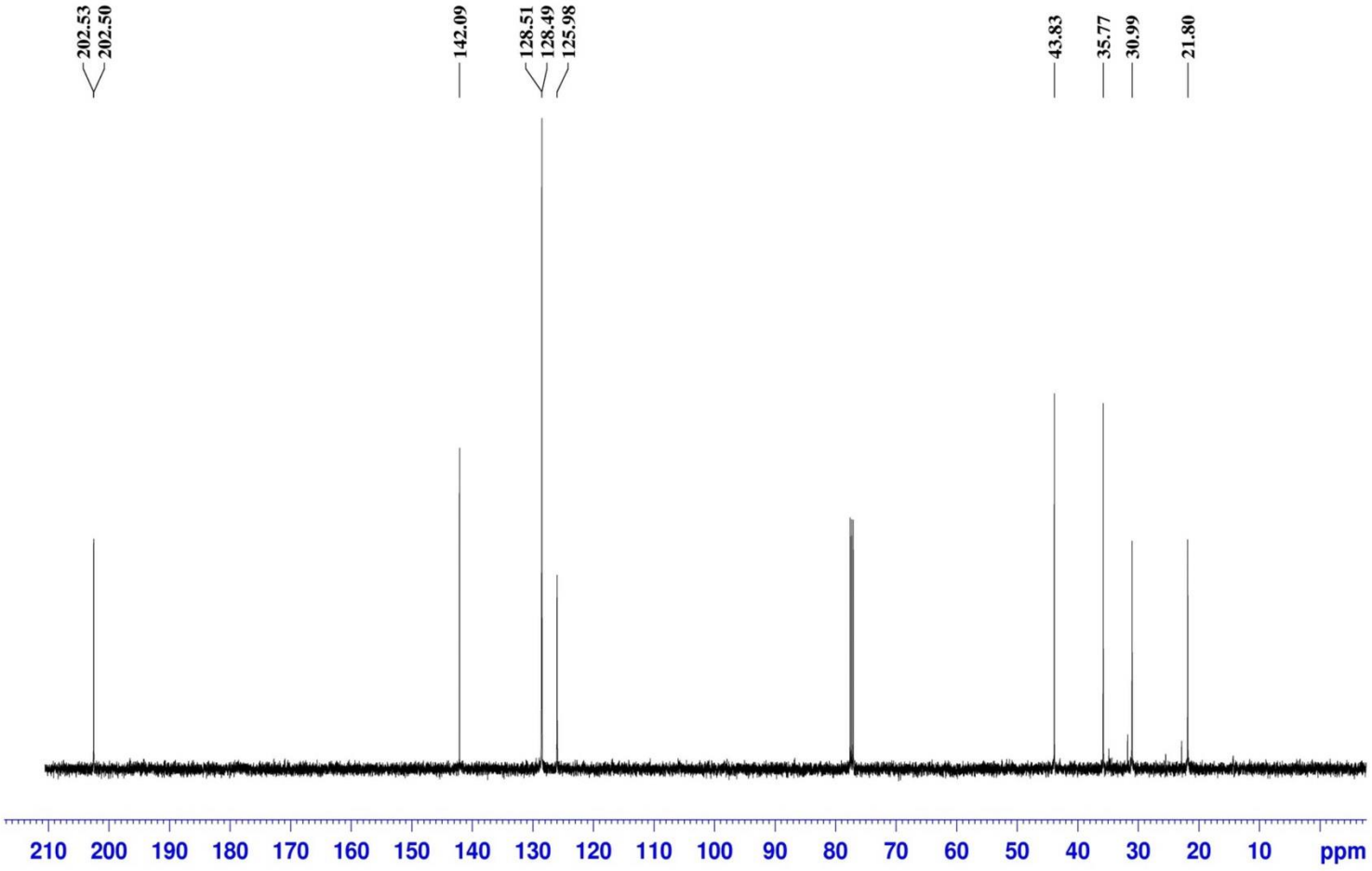
(E)–5–phenylpent–2–en–1–ol
100 MHz, CDCl3



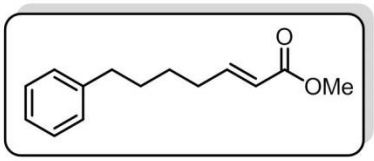

5−phenylpentan−1−ol
100 MHz, CDCl3
142.82
128.65
128.53
125.93
63.17
36.16
32.90
31.51
25.67
OH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

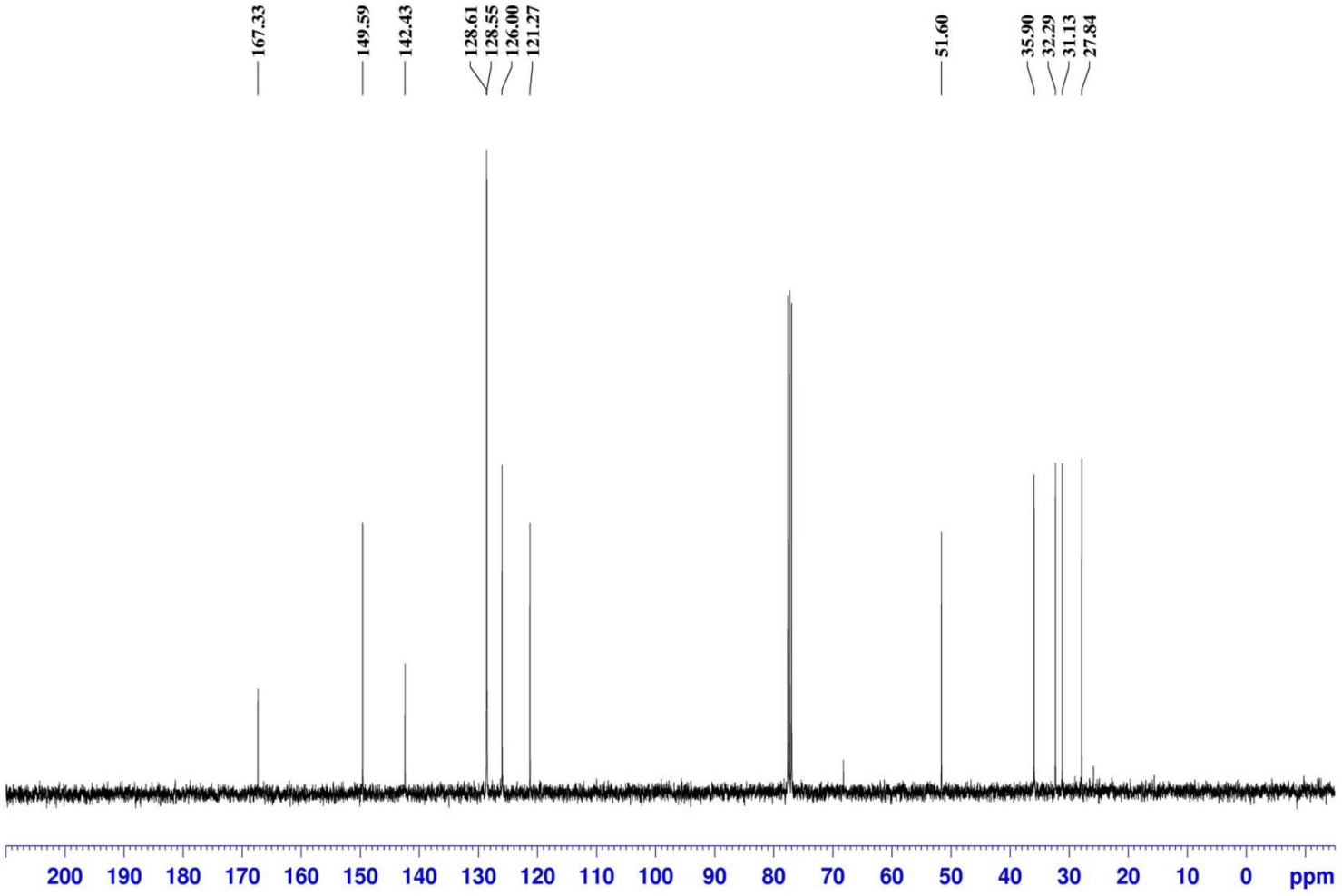
5–phenylpentanal
CDCl3, 100 MHz

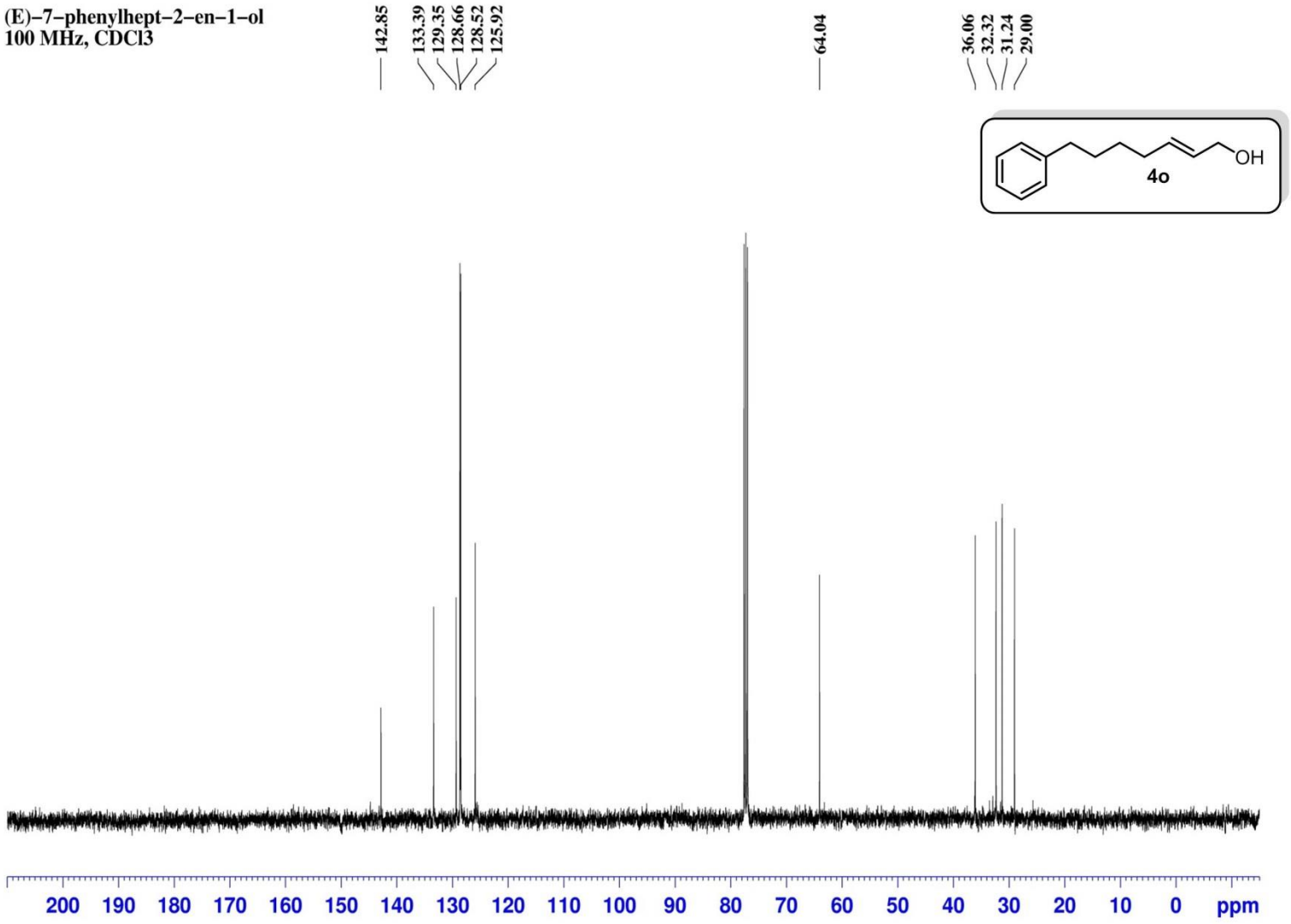


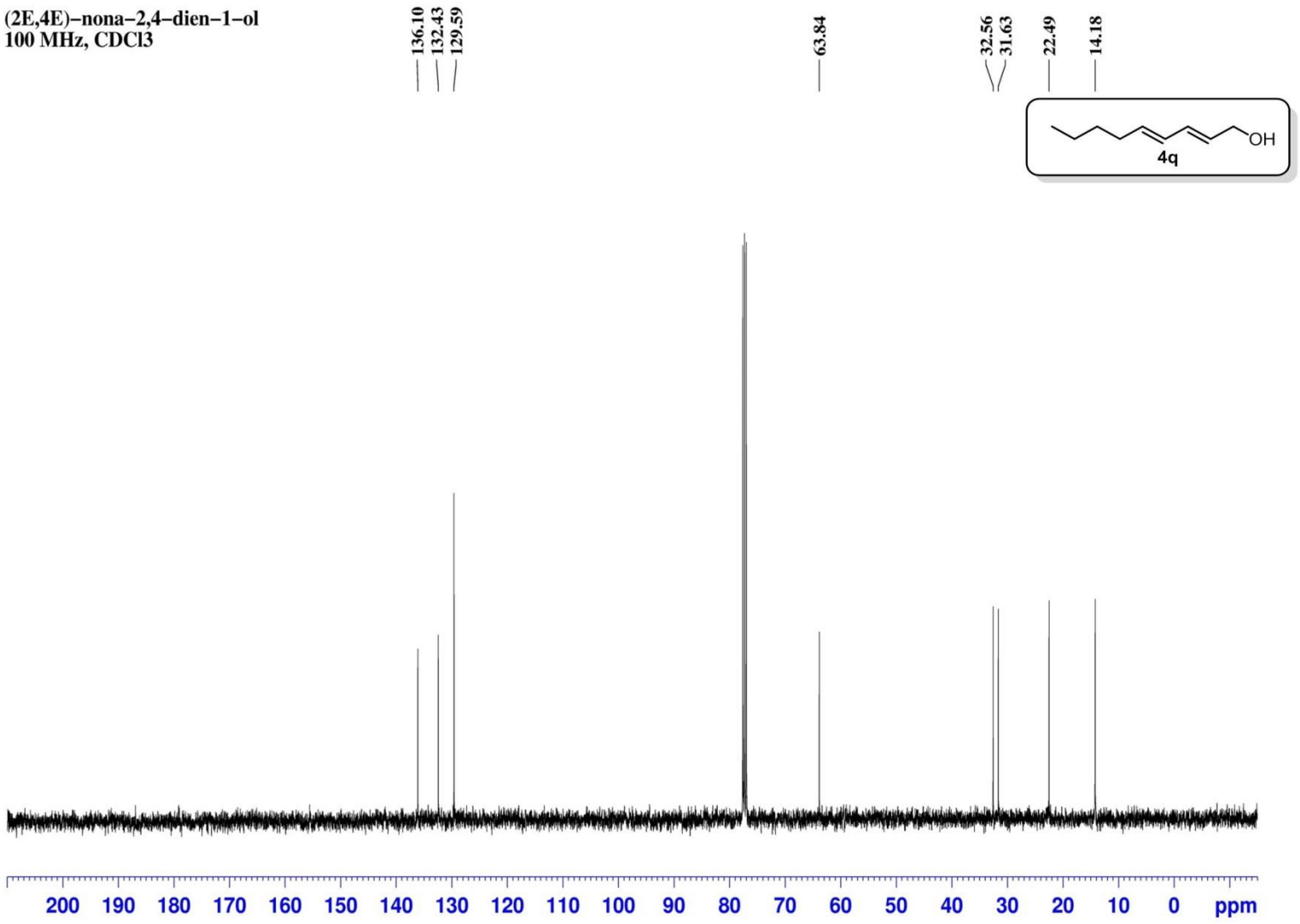


(E)−methyl 7−phenylhept−2−enoate
100 MHz, CDCl3

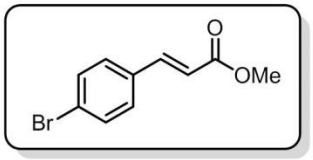


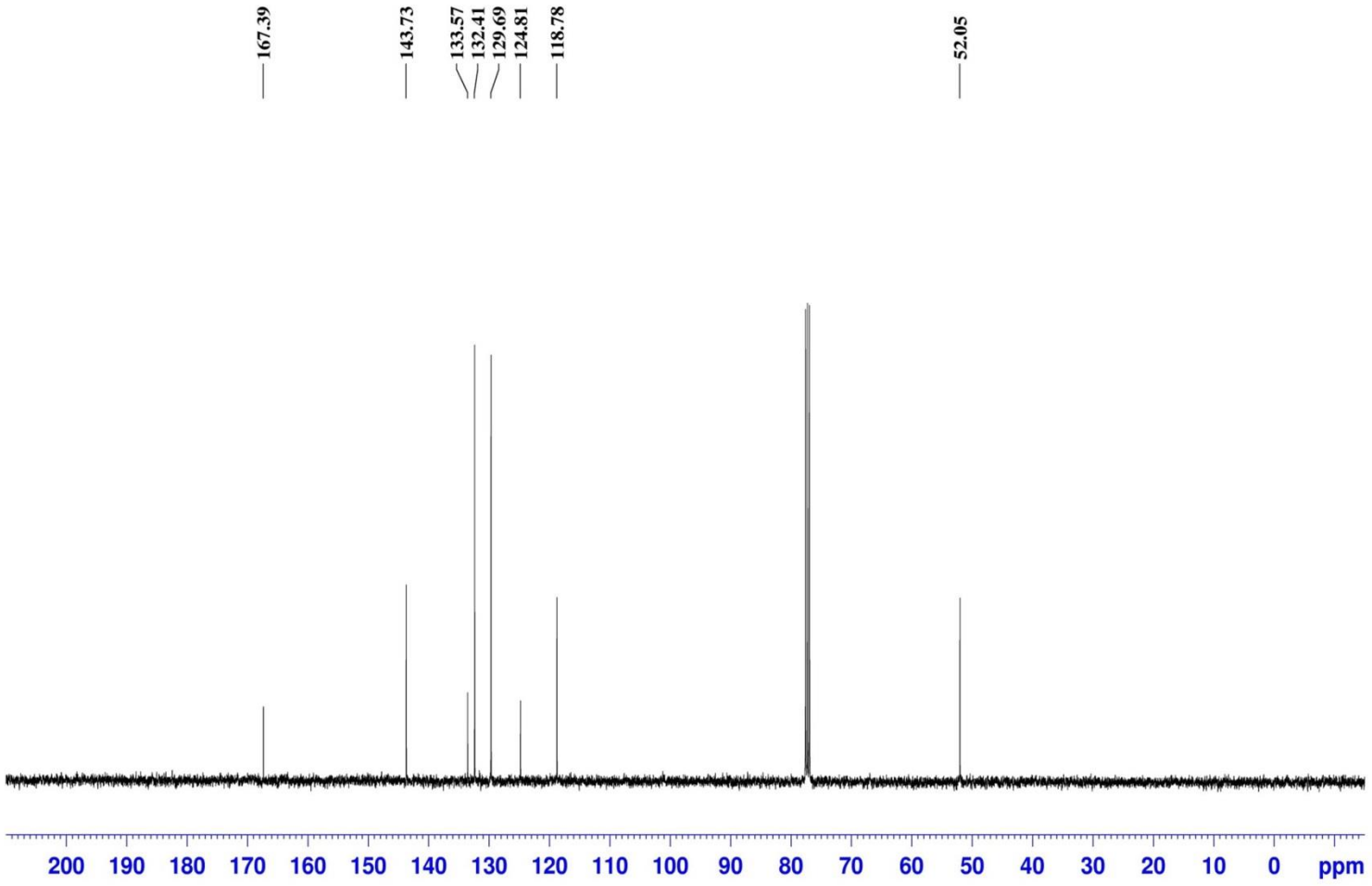



(E)–7–phenylhept–2–en–1–ol
100 MHz, CDCl3
142.85
133.39
129.35
128.66
128.52
125.92
64.04
36.06
32.32
31.24
29.00
OH
4o
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

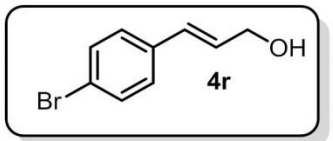

(2E,4E)–nona–2,4–dien–1–ol
100 MHz, CDCl3
136.10
132.43
129.59
63.84
32.56
31.63
22.49
14.18
OH
4q
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

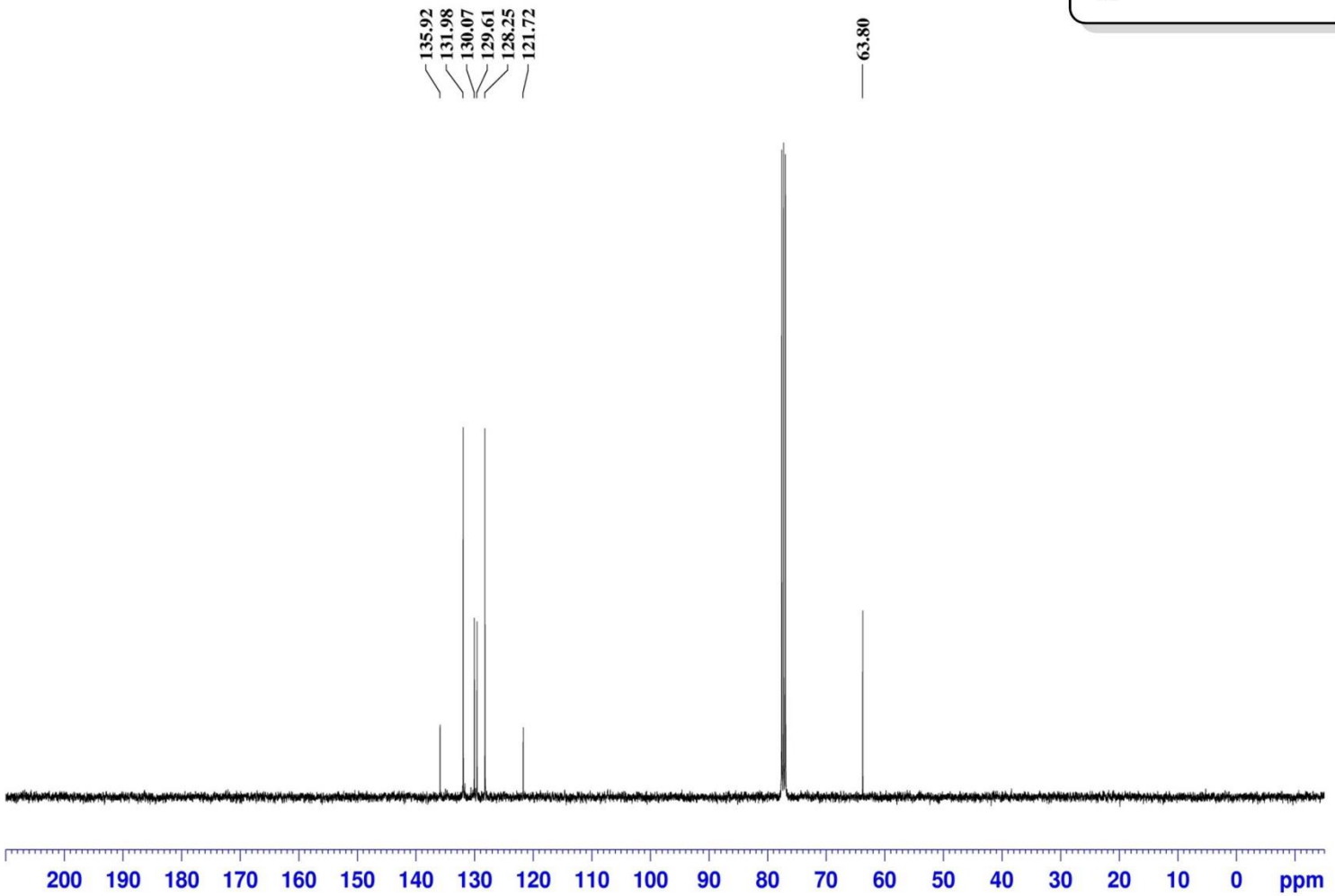
trans–methyl 4–bromocinnamate
100 MHz, CDCl3



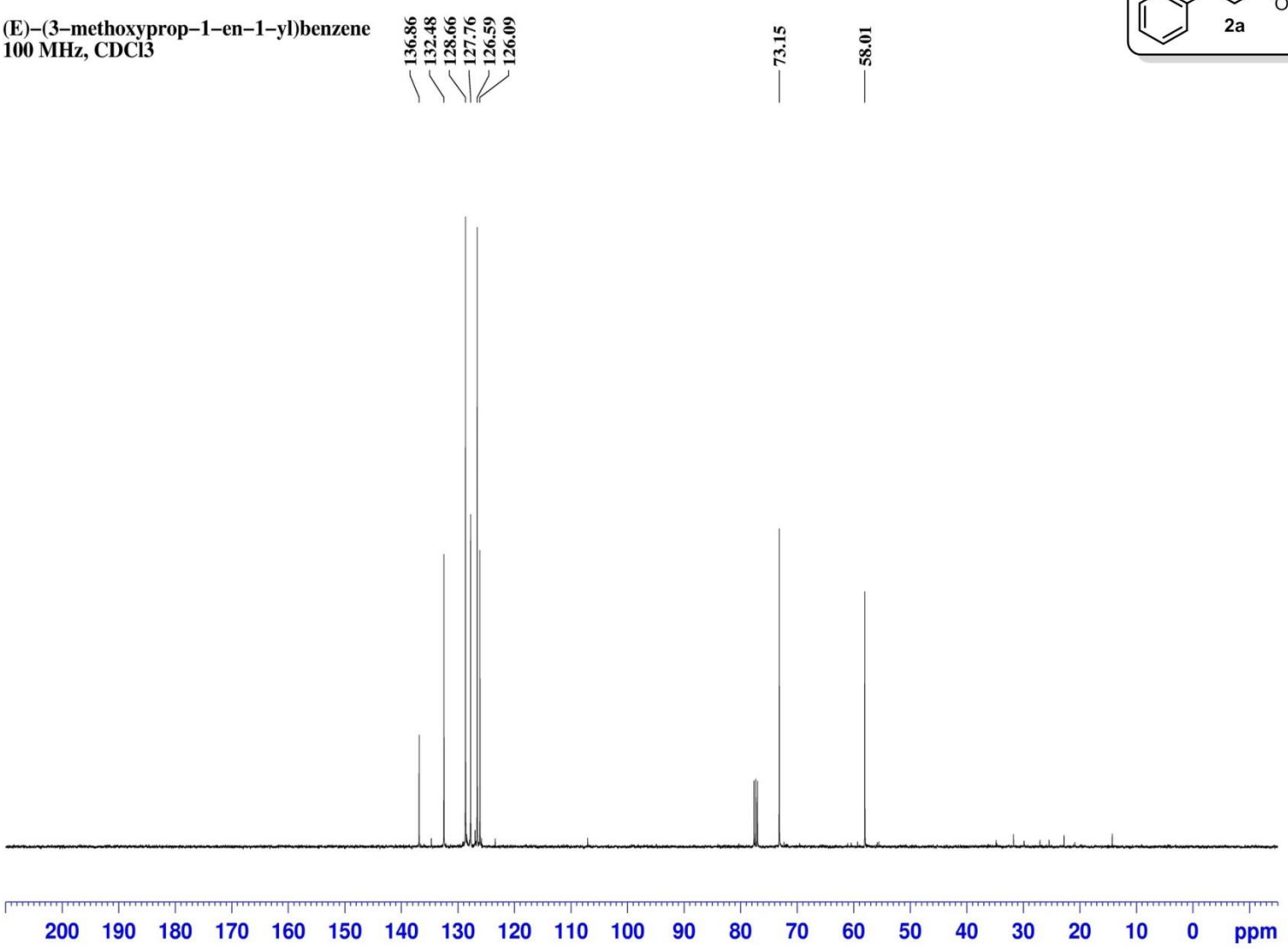


(E)−3−(4−bromophenyl)prop−2−en−1−ol
100 MHz, CDCl3

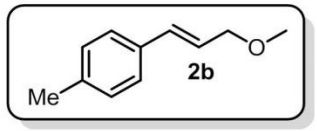


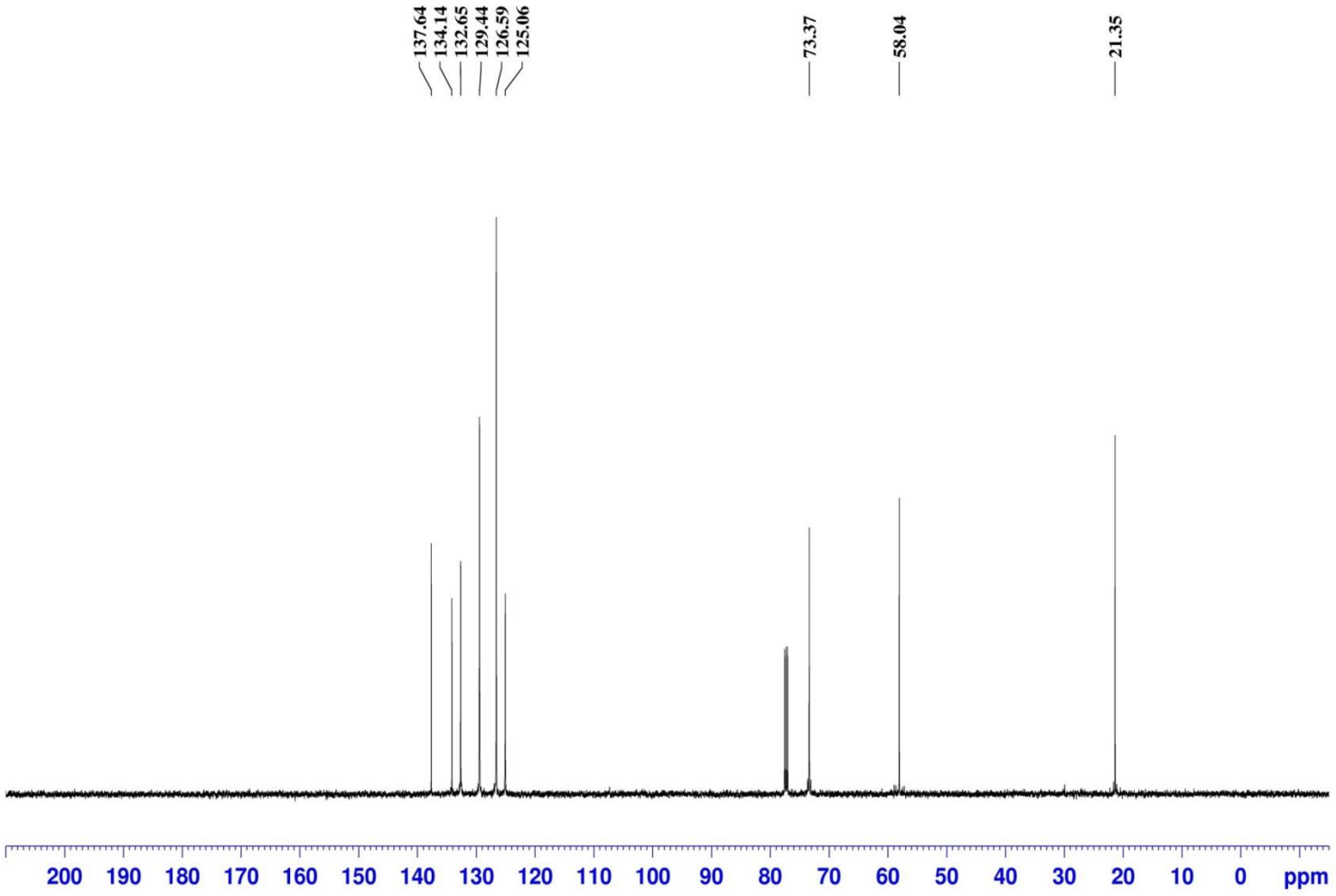


(E)−(3−methoxyprop−1−en−1−yl)benzene
100 MHz, CDCl3

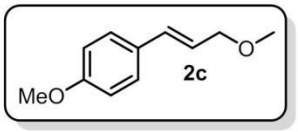


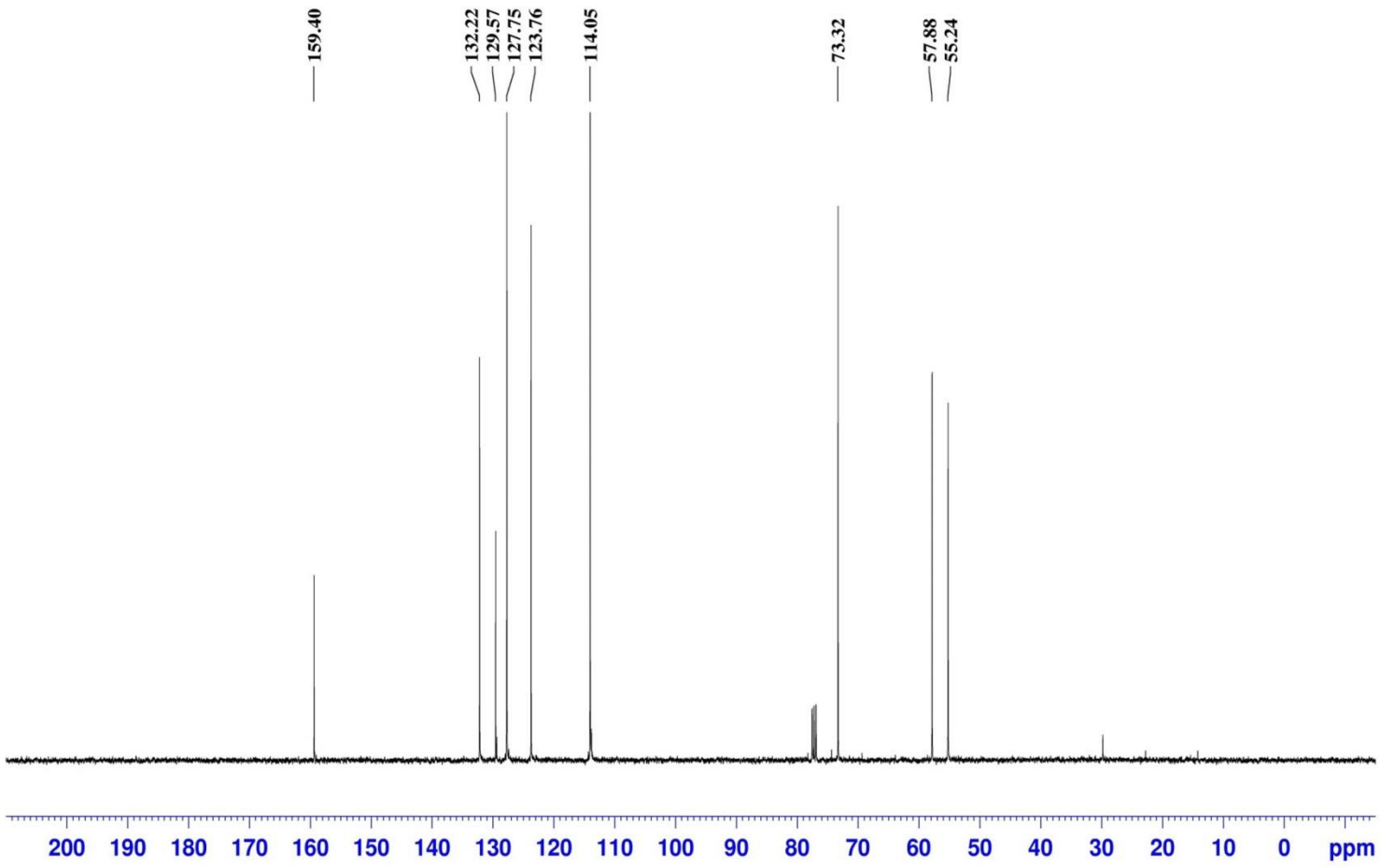
(E)−1−(3−methoxyprop−1−en−1−yl)−4−methylbenzene
125 MHz, CDCl3



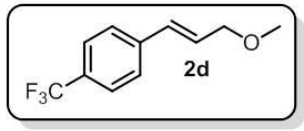


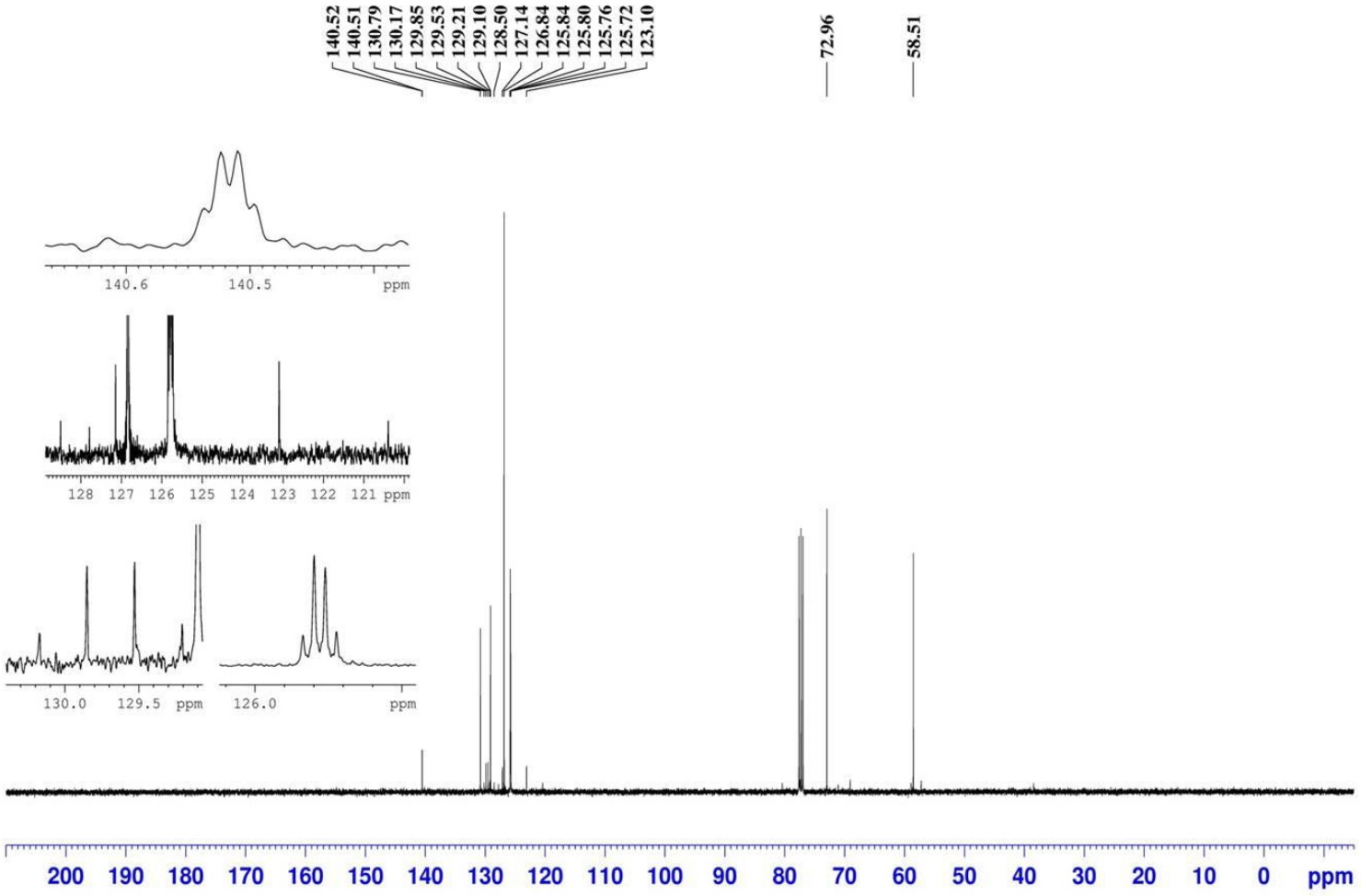
(E)−1−methoxy−4−(3−methoxyprop−1−en−1−yl)benzene
100 MHz, CDCl3



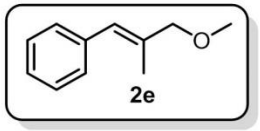


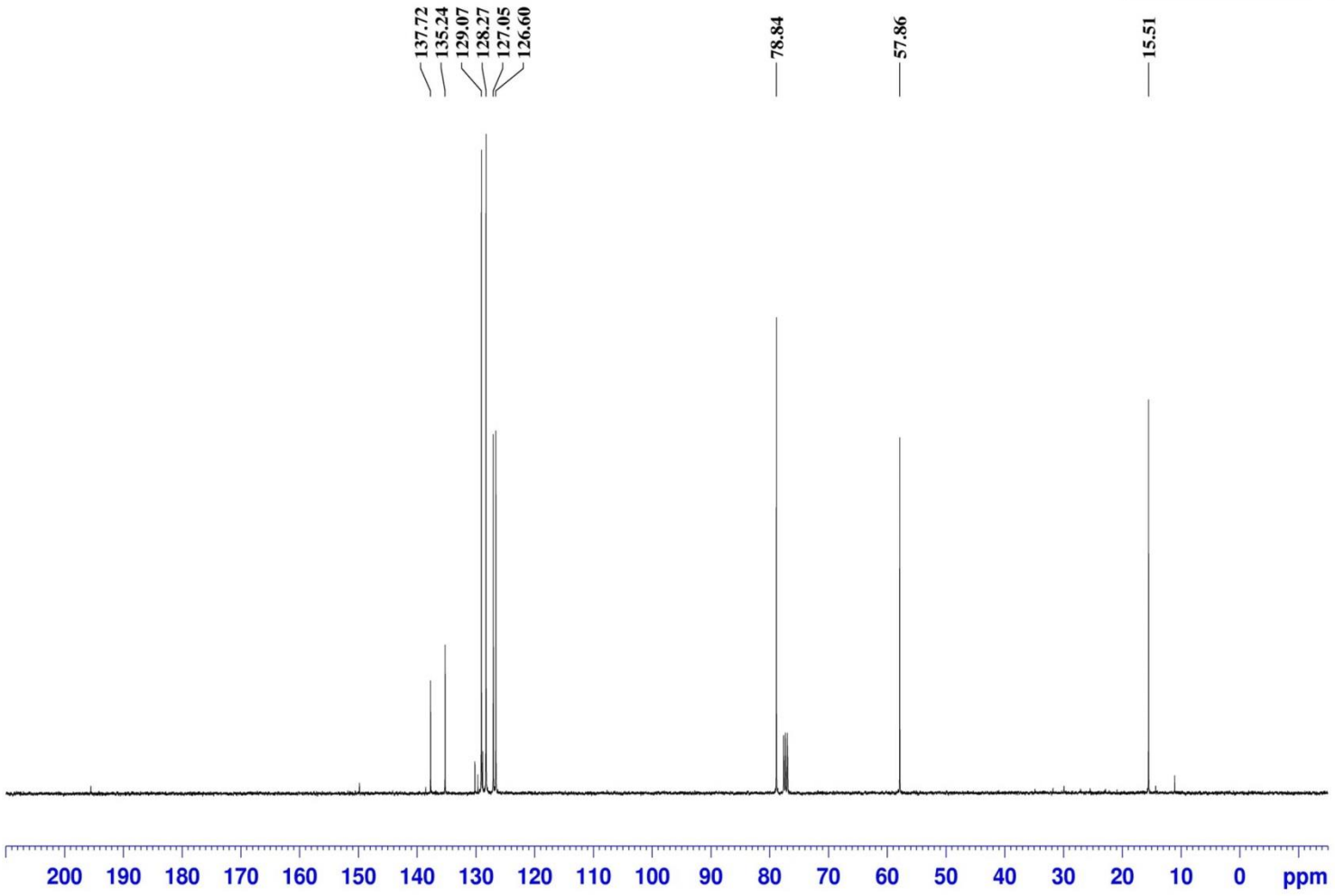
(E)−1−(3−methoxyprop−1−en−1−yl)−4−(trifluoromethyl)benzene
100 MHz, CDCl3



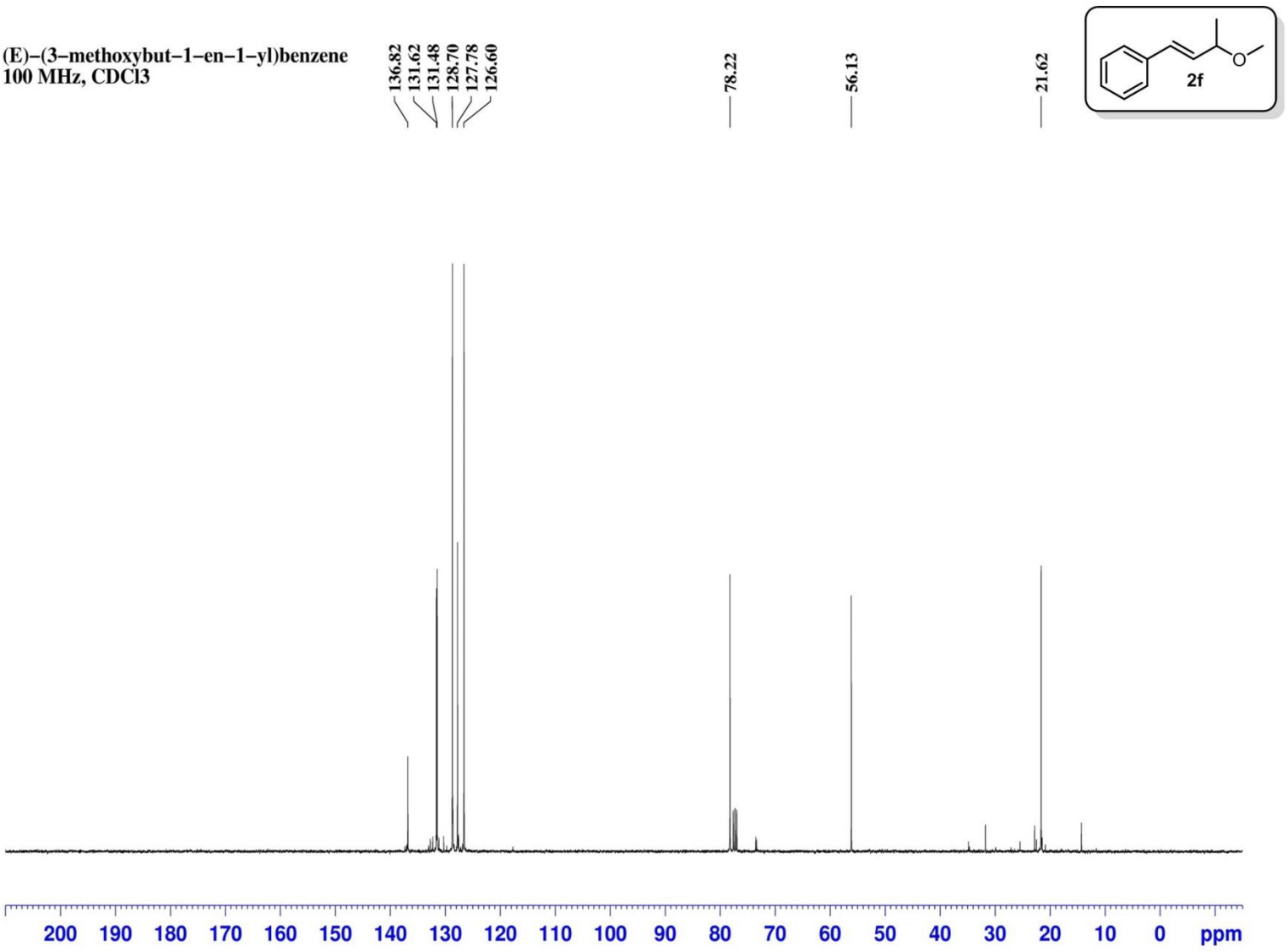


(E)–(3–methoxy–2–methylprop–1–en–1–yl)benzene
100 MHz, CDCl3

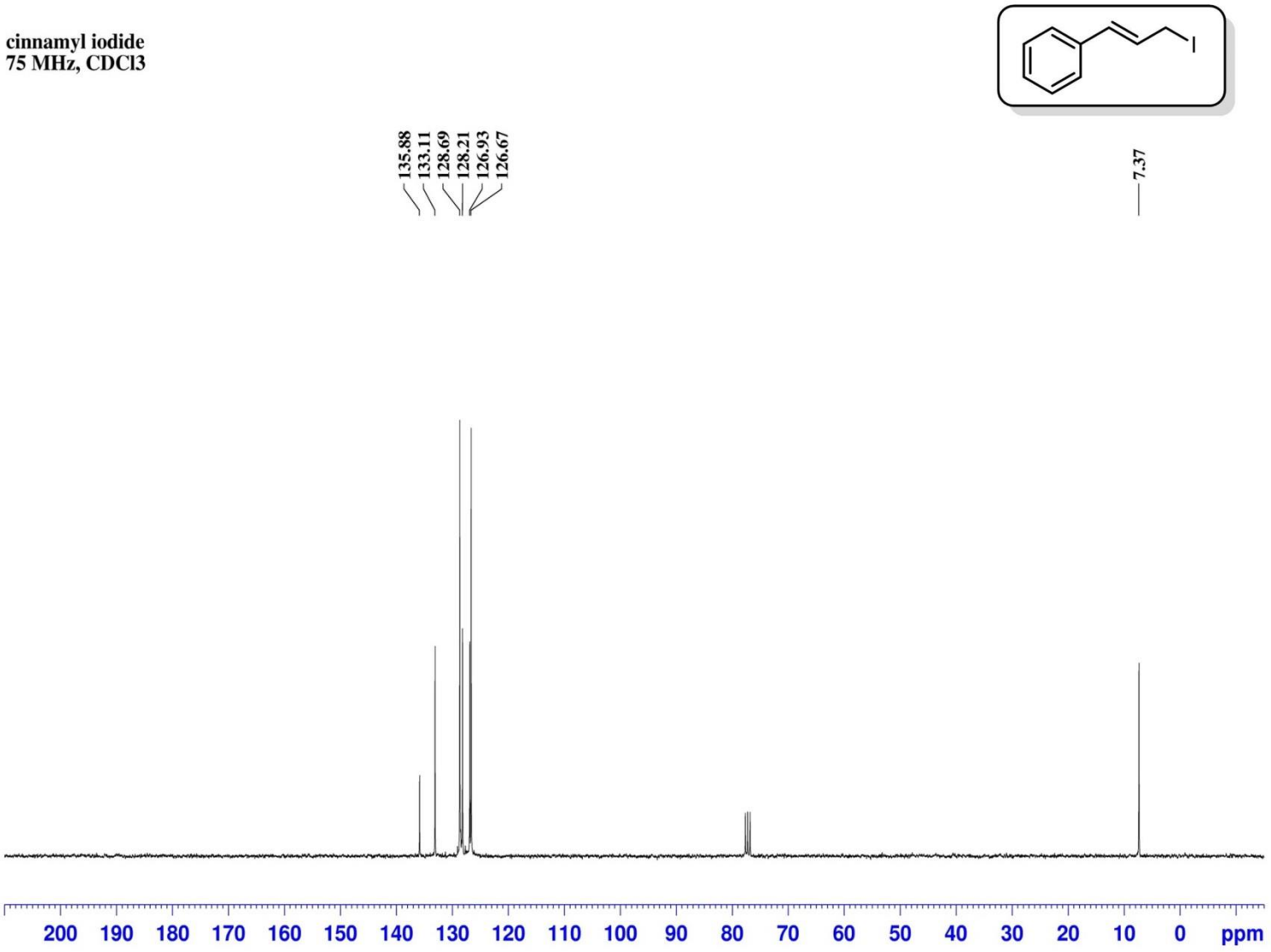


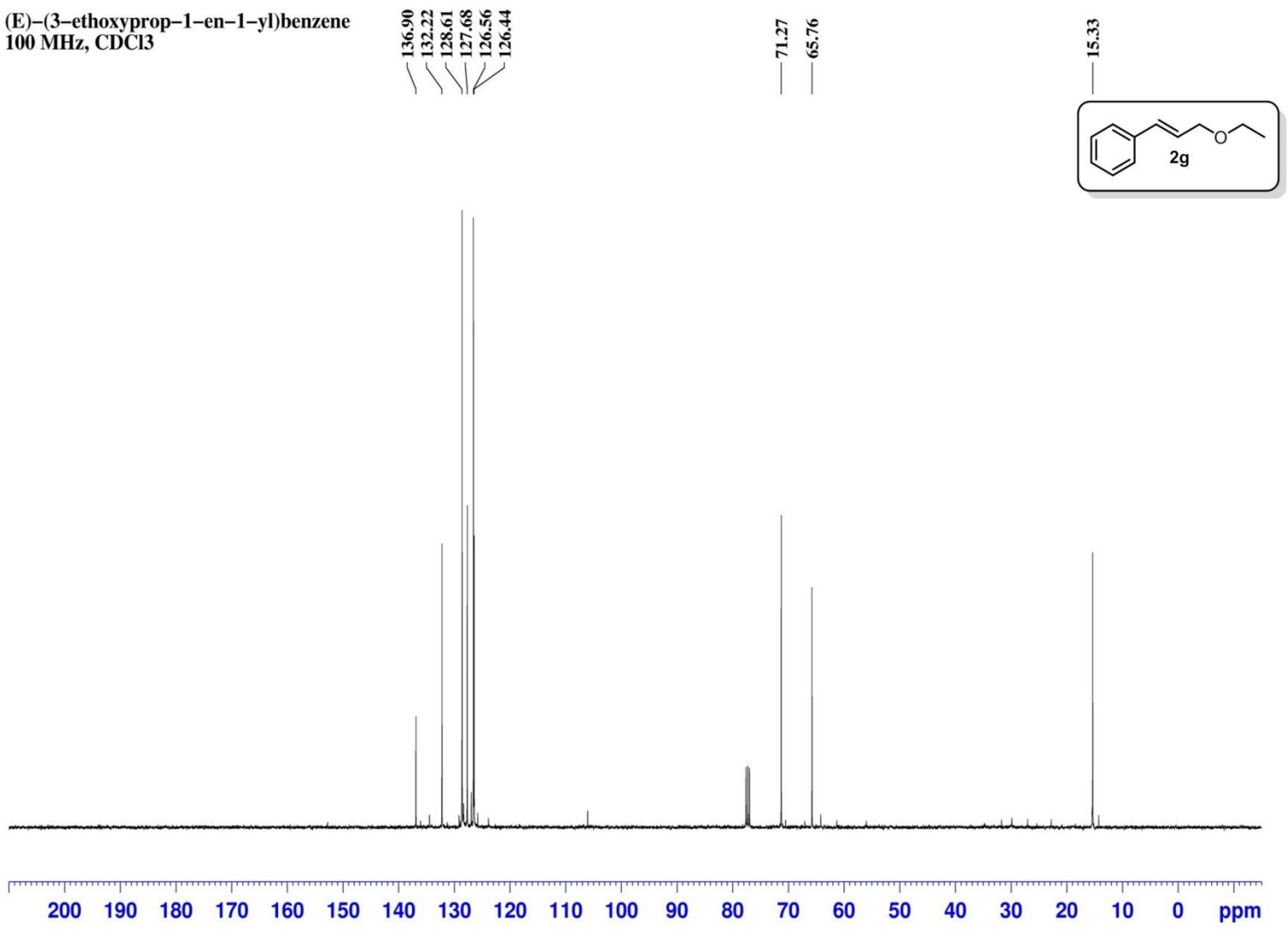


(E)−(3−methoxybut−1−en−1−yl)benzene
100 MHz, CDCl3

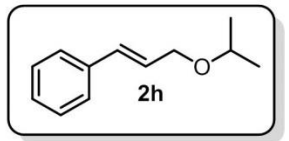


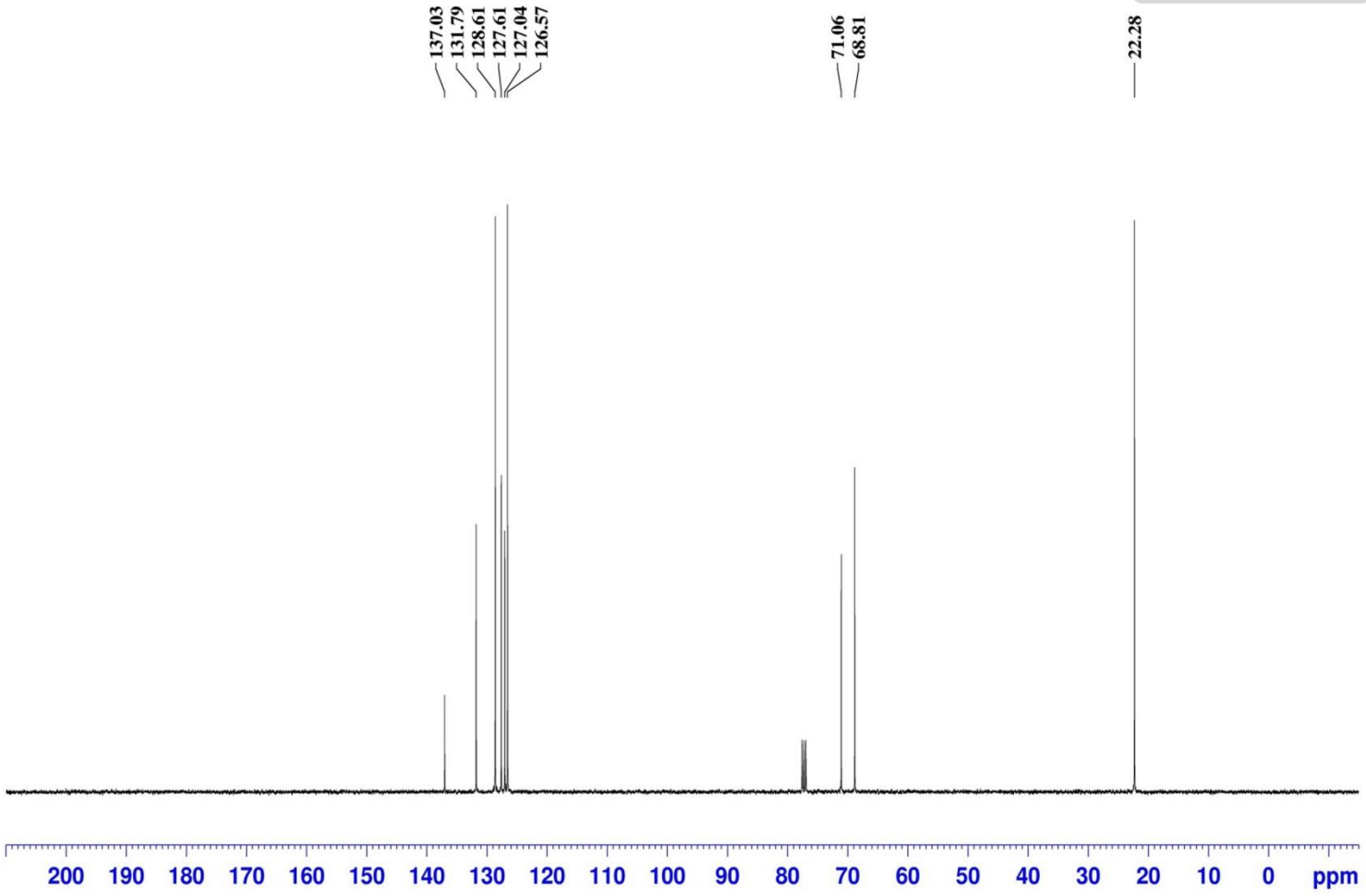
cinnamyl iodide
75 MHz, CDCl3



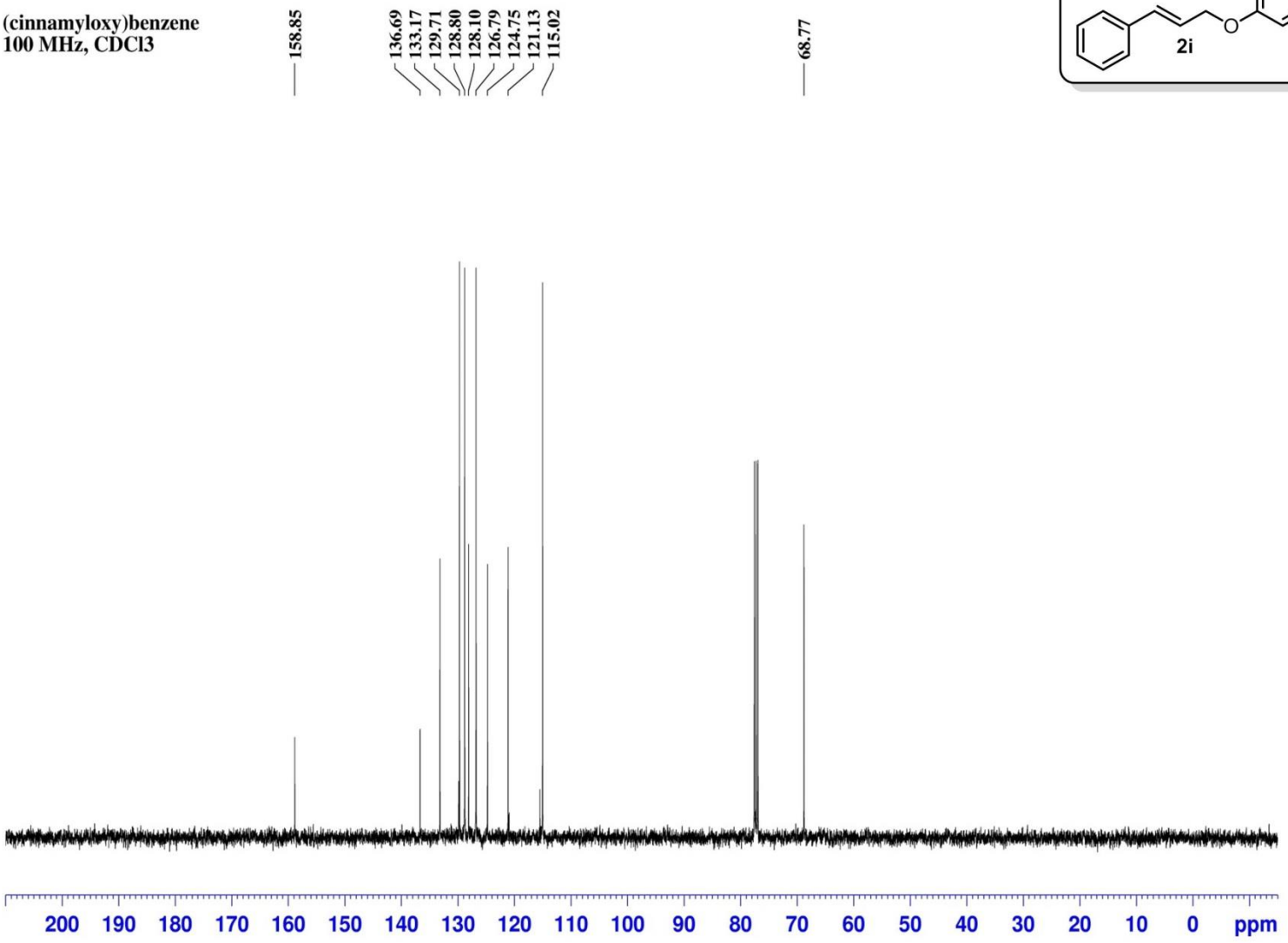

(E)−(3−ethoxyprop−1−en−1−yl)benzene
100 MHz, CDCl3
136.90
132.22
128.61
127.68
126.56
126.44
71.27
65.76
15.33
2g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

(E)−(3−isopropoxyprop−1−en−1−yl)benzene
100 MHz, CDCl3

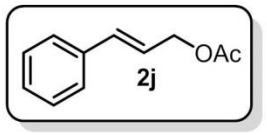


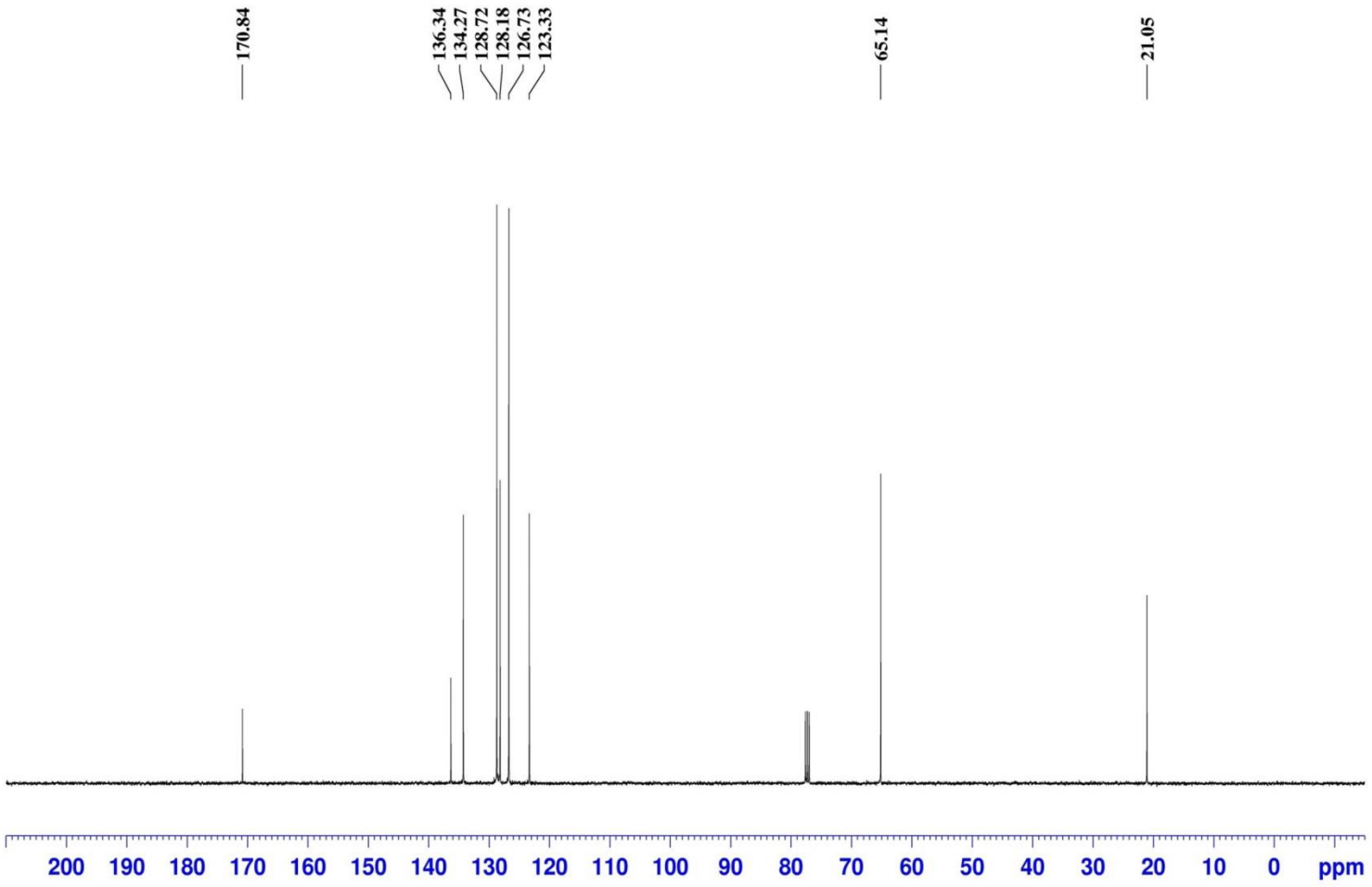


(cinnamyloxy)benzene
100 MHz, CDCl3

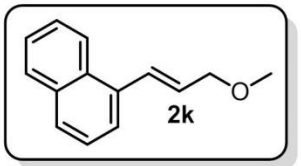


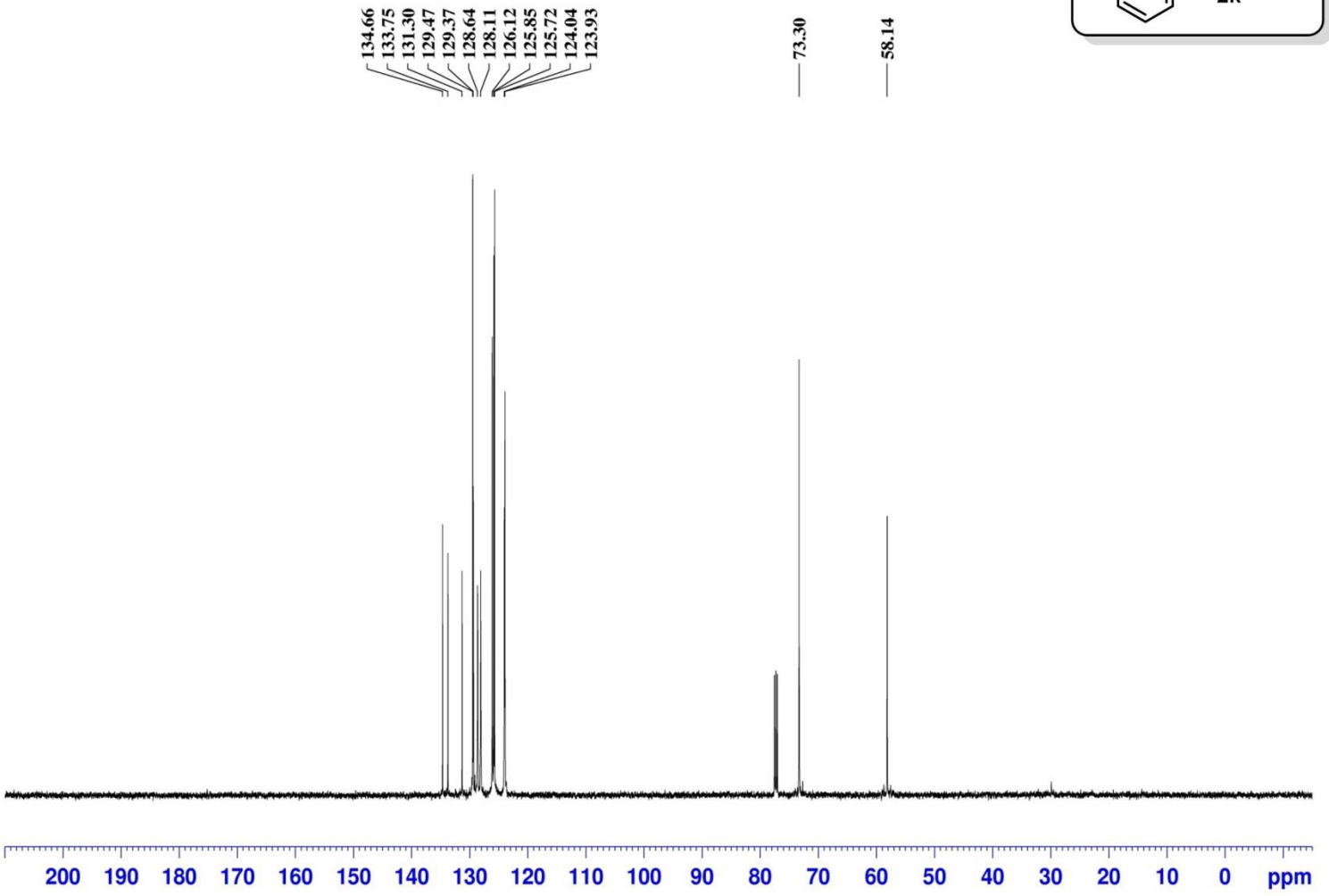
cinnamyl acetate
100 MHz, CDCl3



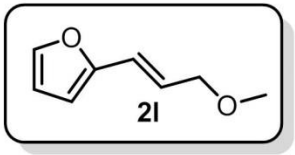


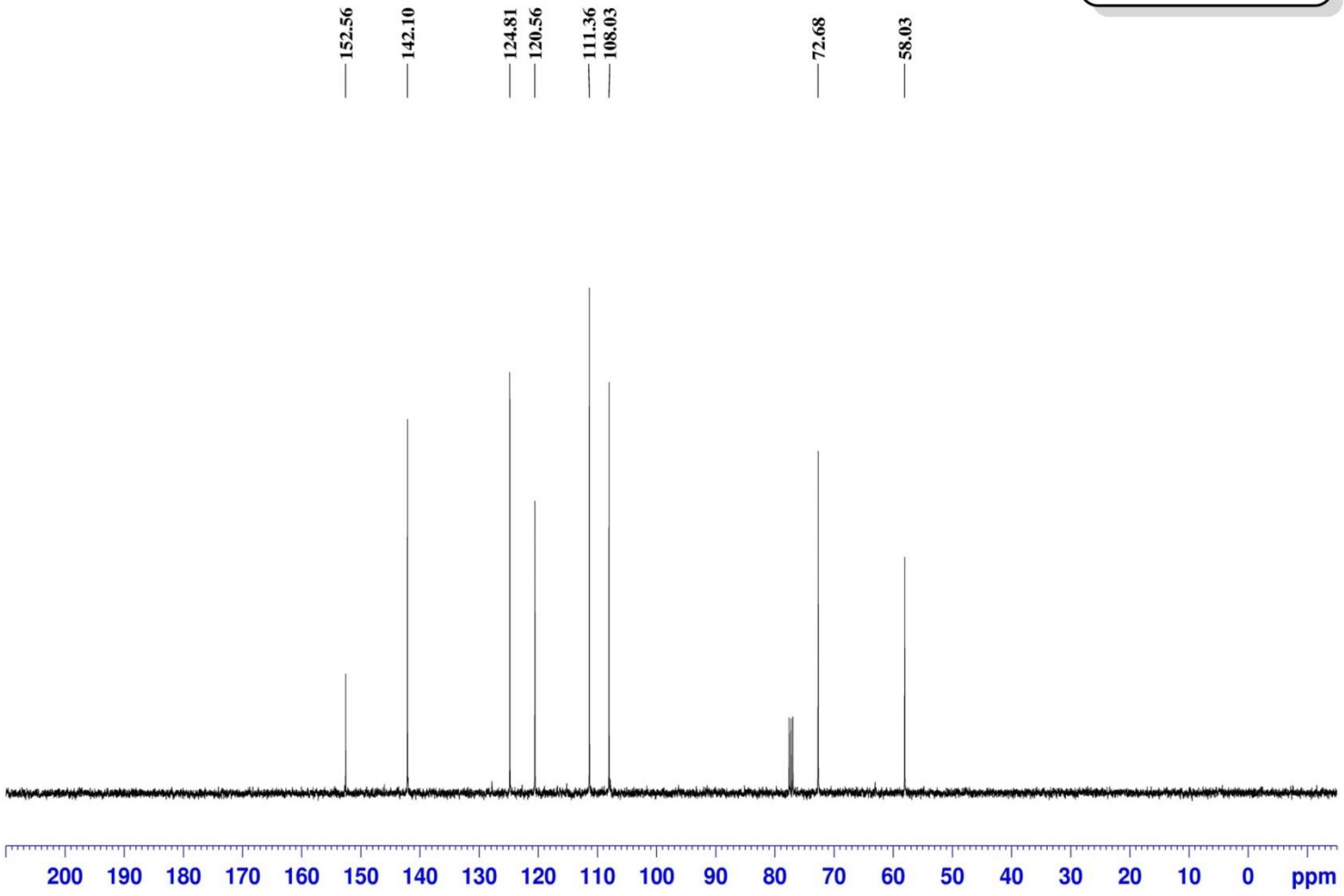
(E)−1−(3−methoxyprop−1−en−1−yl)naphthalene
125 MHz, CDCl3



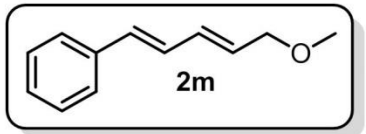


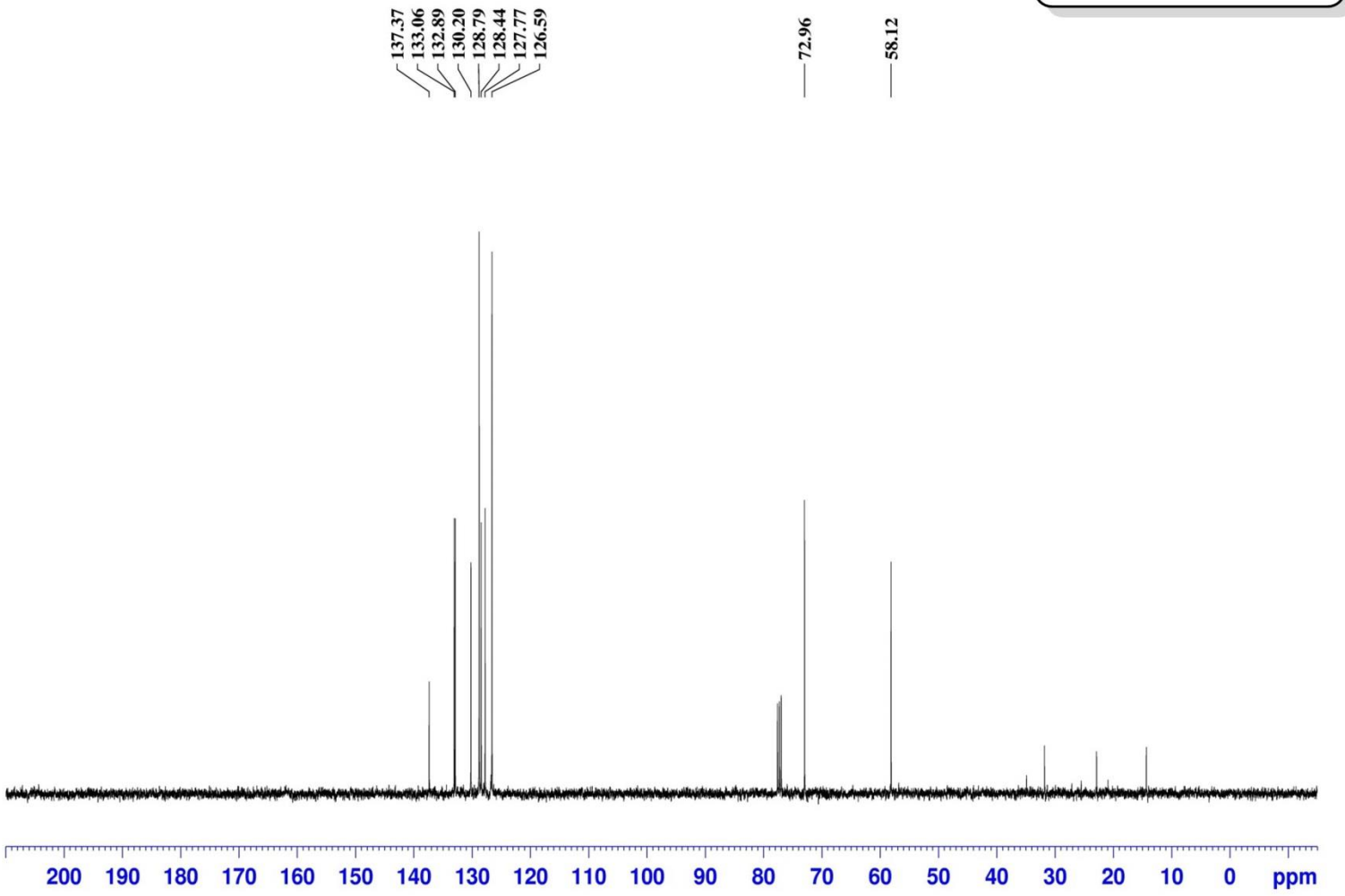
(E)−2−(3−methoxyprop−1−en−1−yl)furan
100 MHz, CDCl3



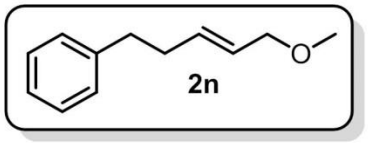


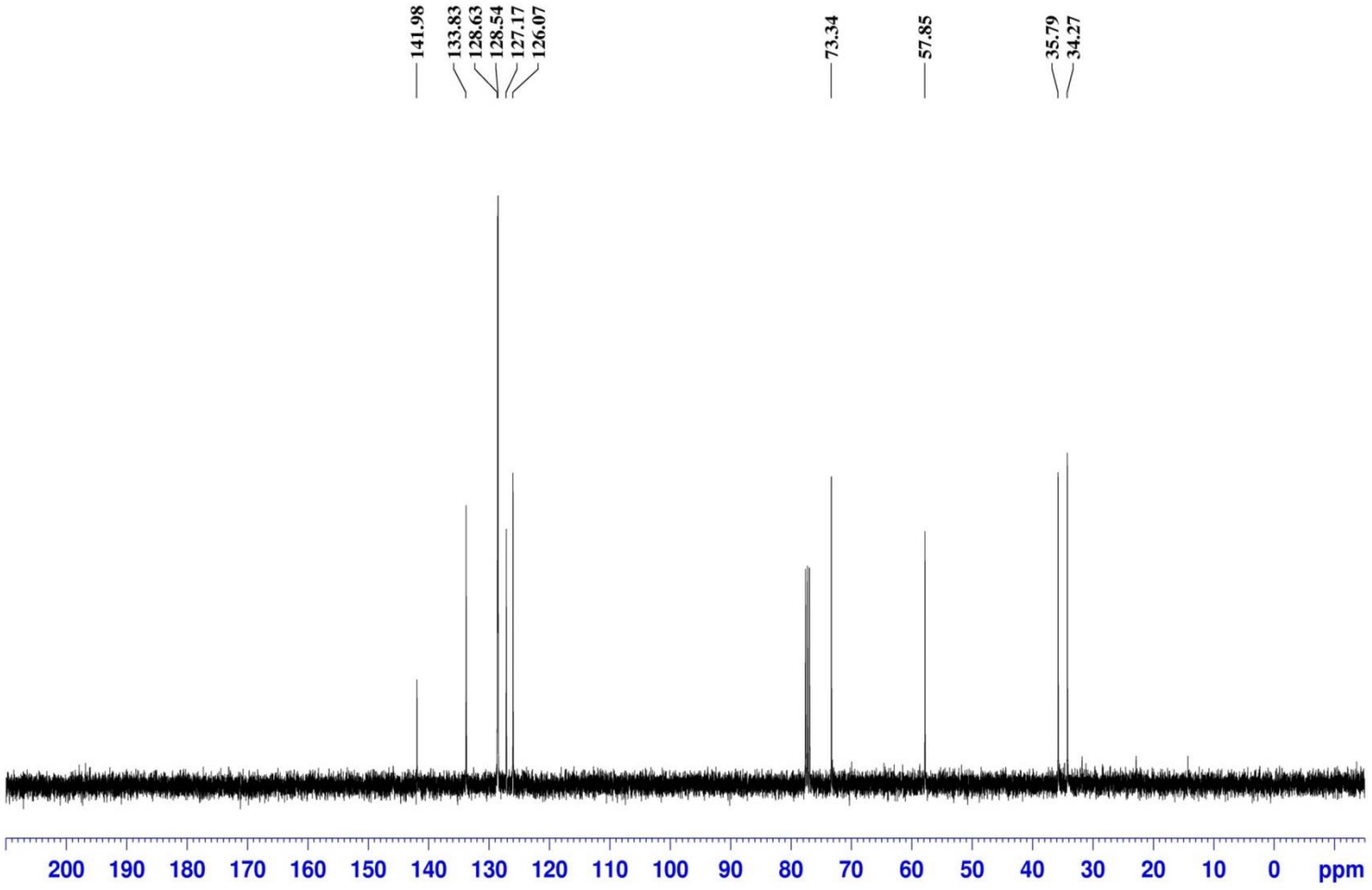
((1E,3E)−5−methoxypenta−1,3−dien−1−yl)benzene
100 MHz, CDCl3

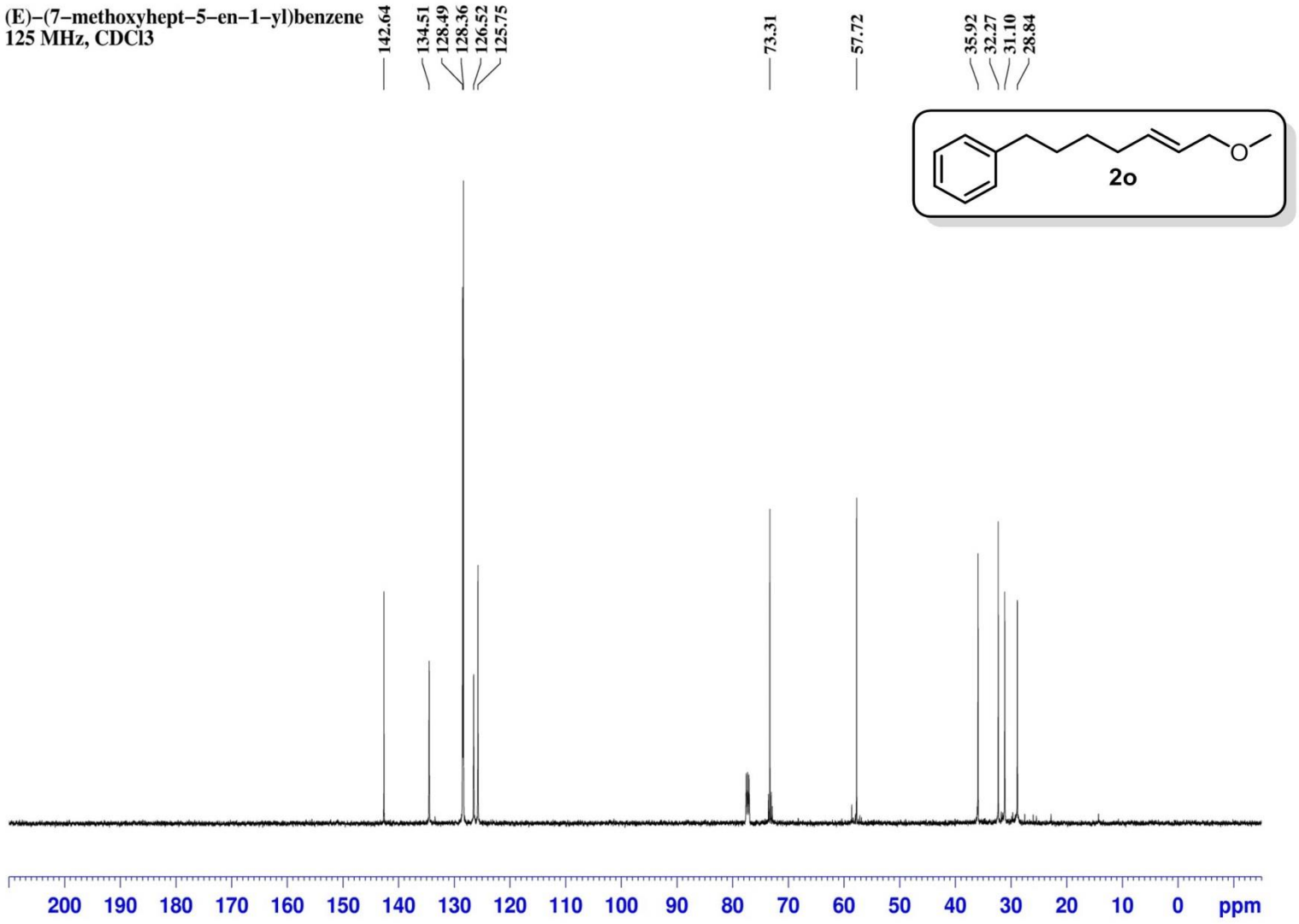


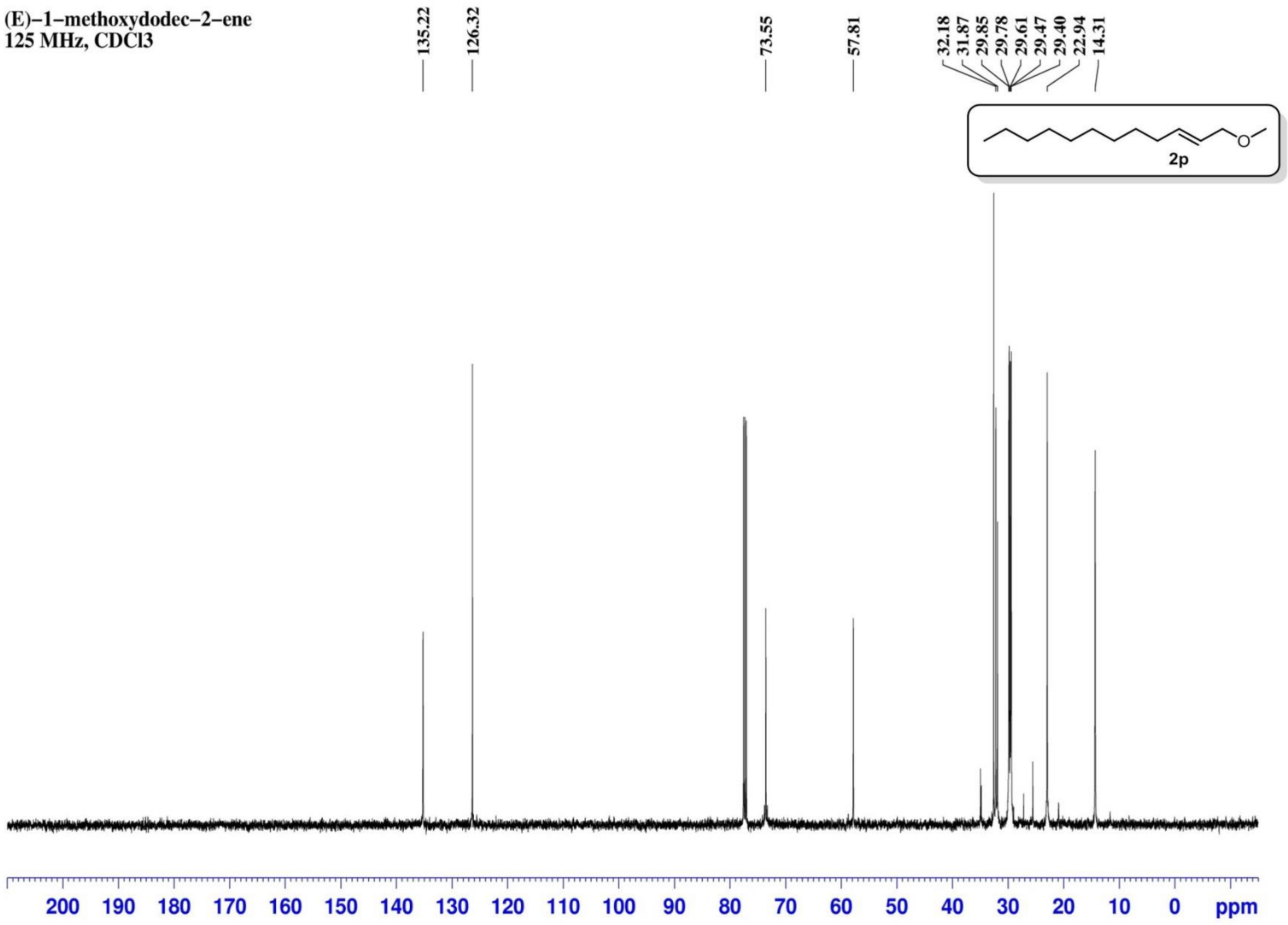


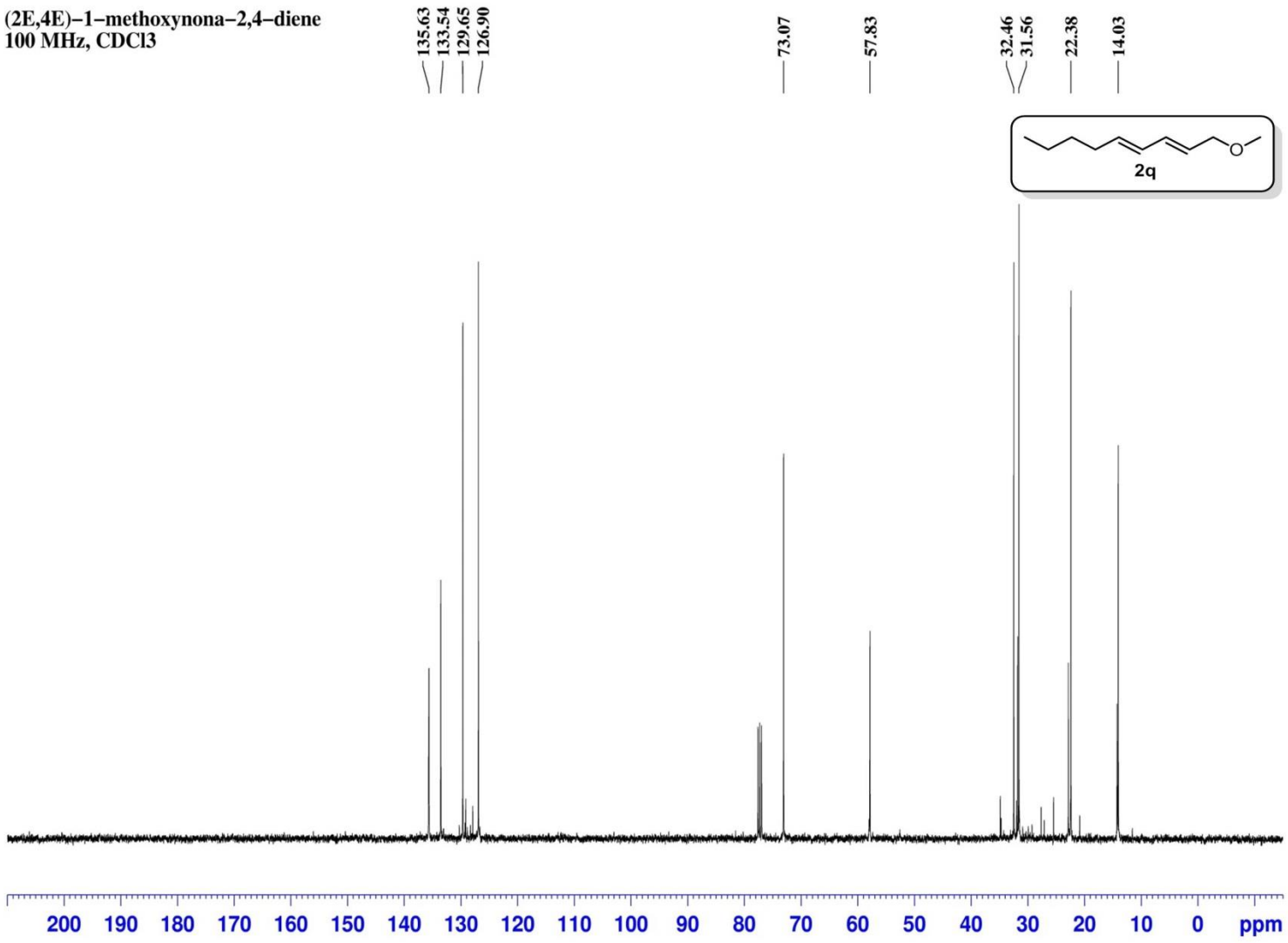
(E)−(5−methoxypent−3−en−1−yl)benzene
100 MHz. CDCl3



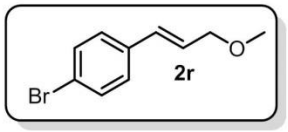


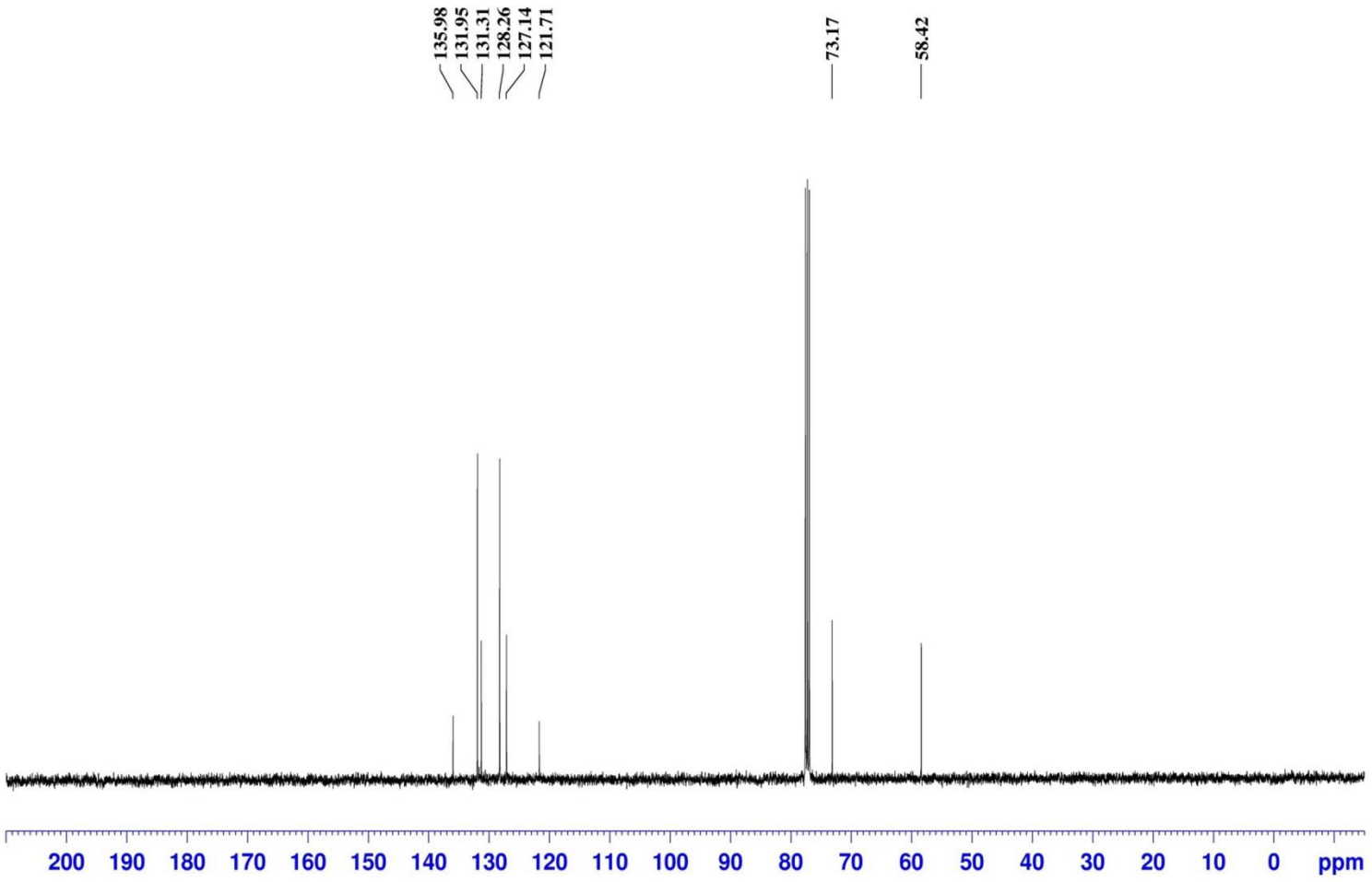

(E)−(7−methoxyhept−5−en−1−yl)benzene
125 MHz, CDCl3
142.64
134.51
128.49
128.36
126.52
125.75
73.31
57.72
35.92
32.27
31.10
28.84
2o
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


(E)−1−methoxydodec−2−ene
125 MHz, CDCl3
135.22
126.32
73.55
57.81
32.18
31.87
29.85
29.78
29.61
29.47
29.40
22.94
14.31
2p
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

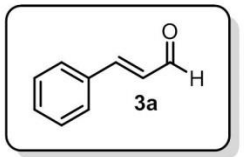

(2E,4E)−1−methoxynona−2,4−diene
100 MHz, CDCl3
135.63
133.54
129.65
126.90
73.07
57.83
32.46
31.56
22.38
14.03
2q
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

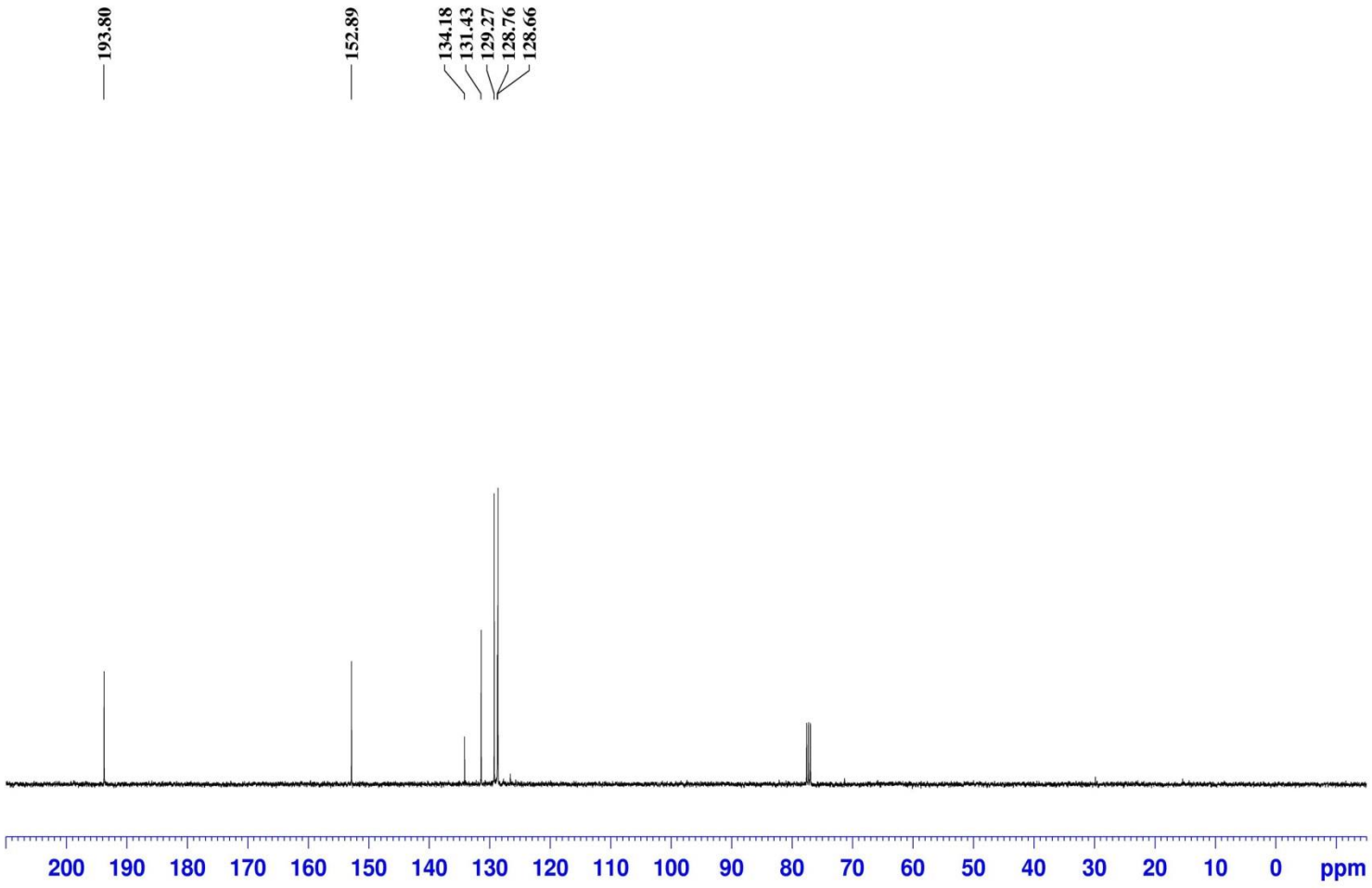
(E)−1−bromo−4−(3−methoxyprop−1−en−1−yl)benzene
100 MHz, CDCl3



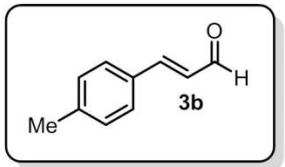


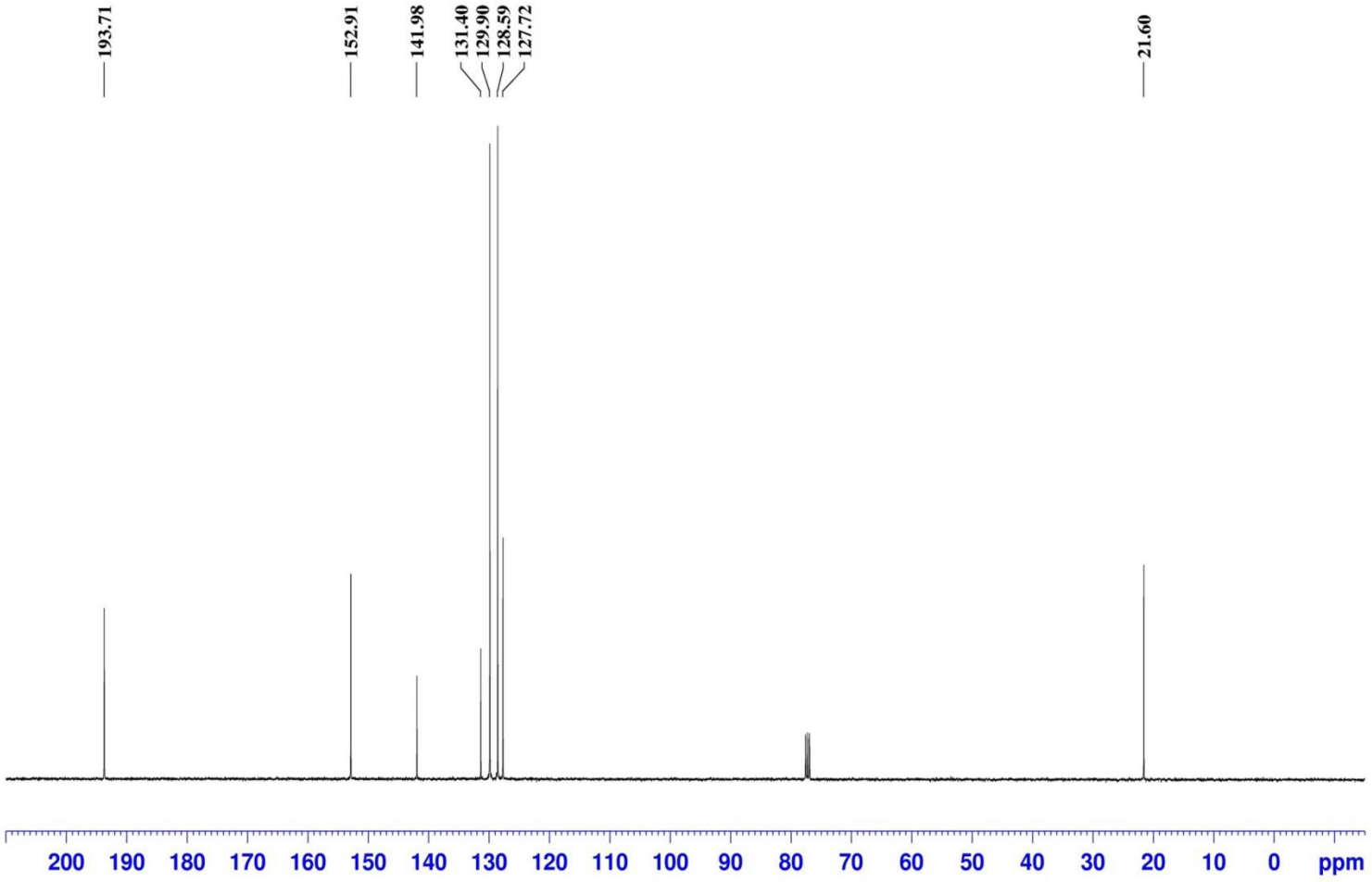
cinnamaldehyde
400 MHz, CDCl3



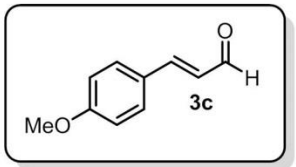


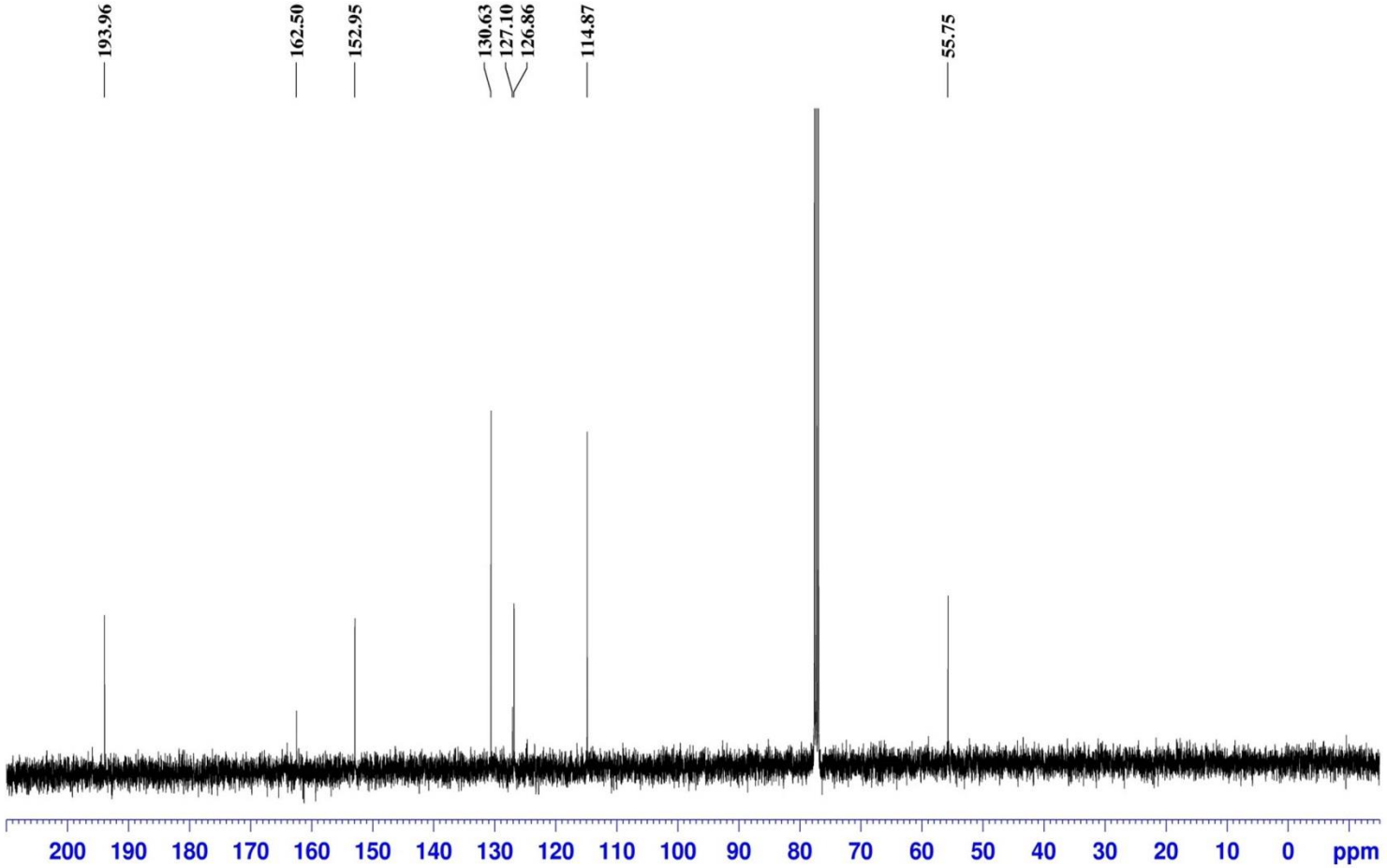
trans–4–methylcinnamaldehyde
100 MHz, CDCl3



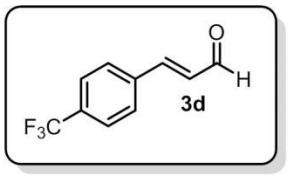


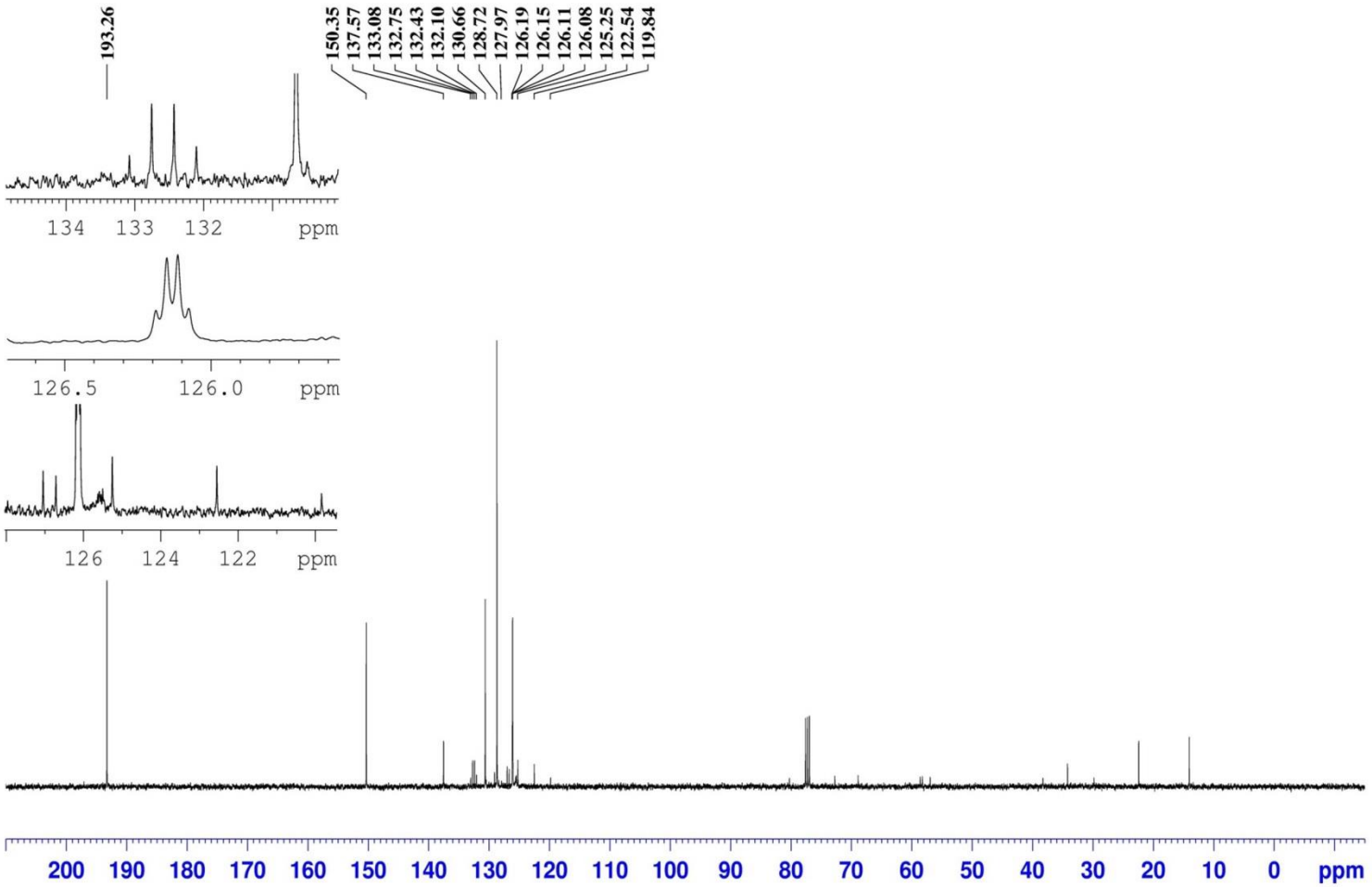
trans−4−methoxycinnamaldehyde
100 MHz, CDCl3



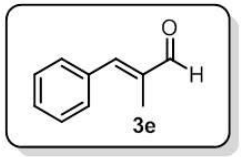


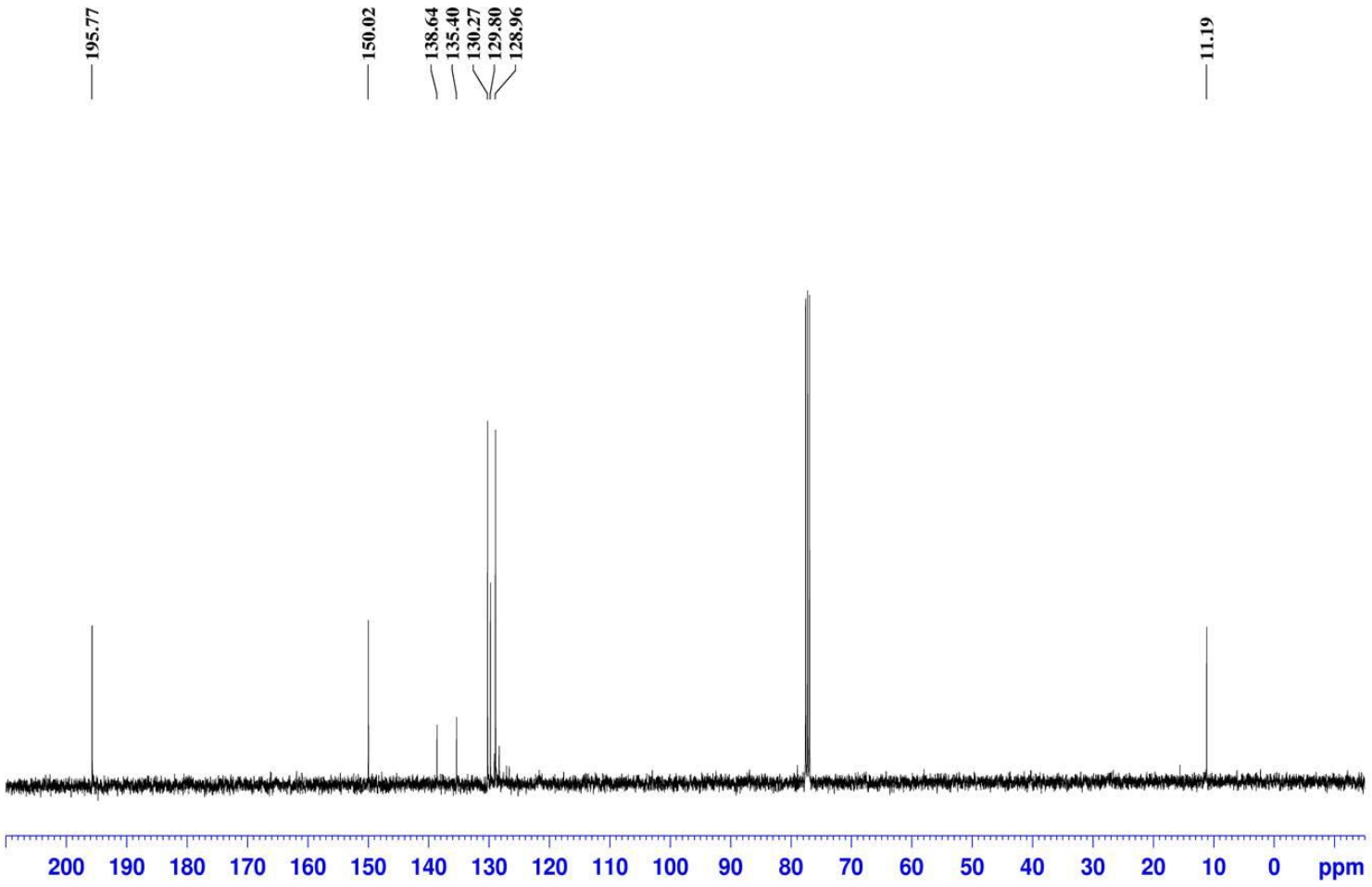
trans−4−(trifluoromethyl)cinnamaldehyde
100 MHz, CDCl3



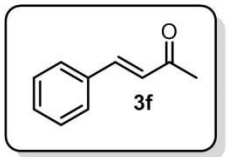


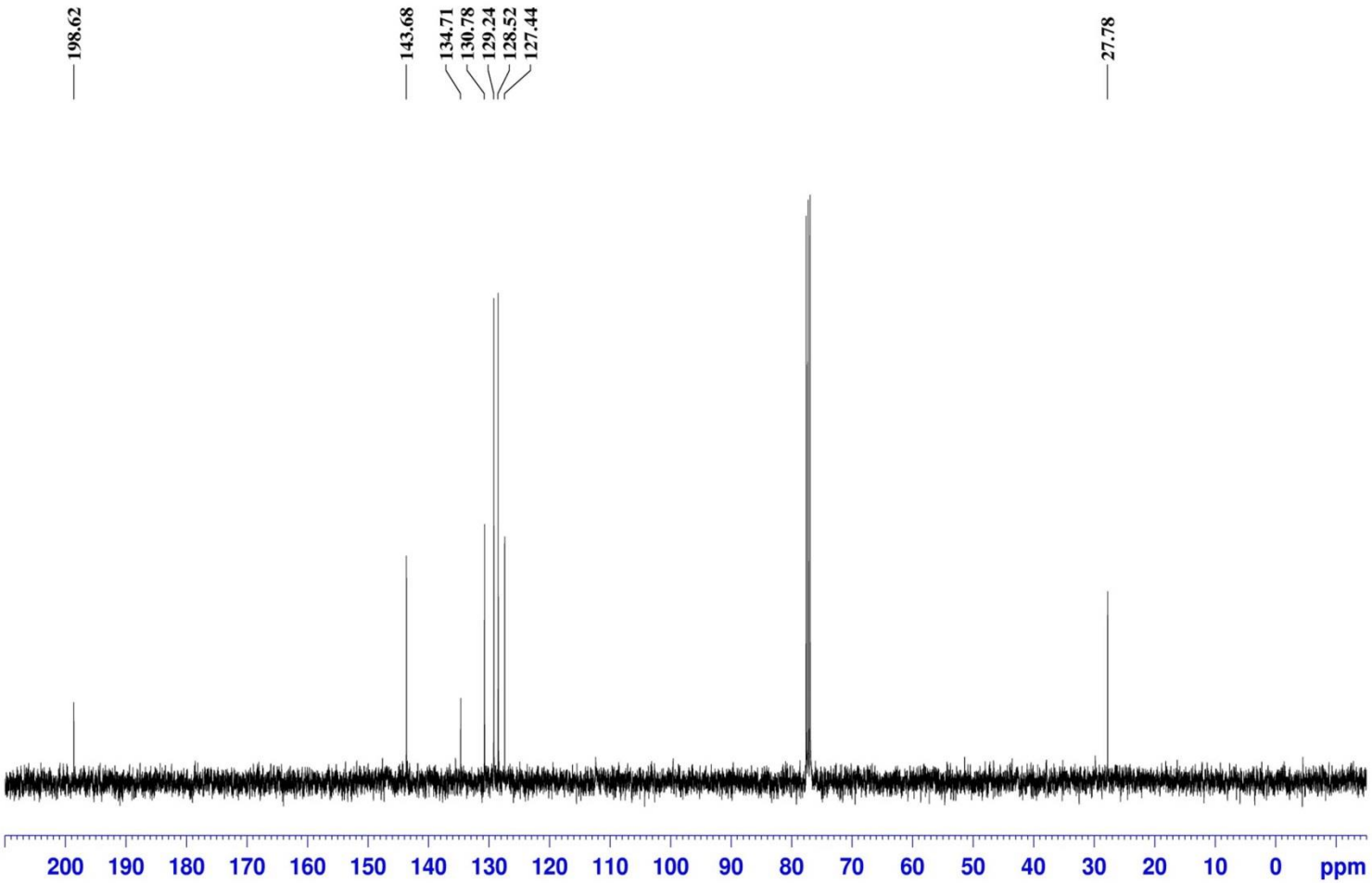
alpha–methylcinnamaldehyde
100 MHz, CDCl3



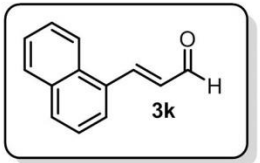


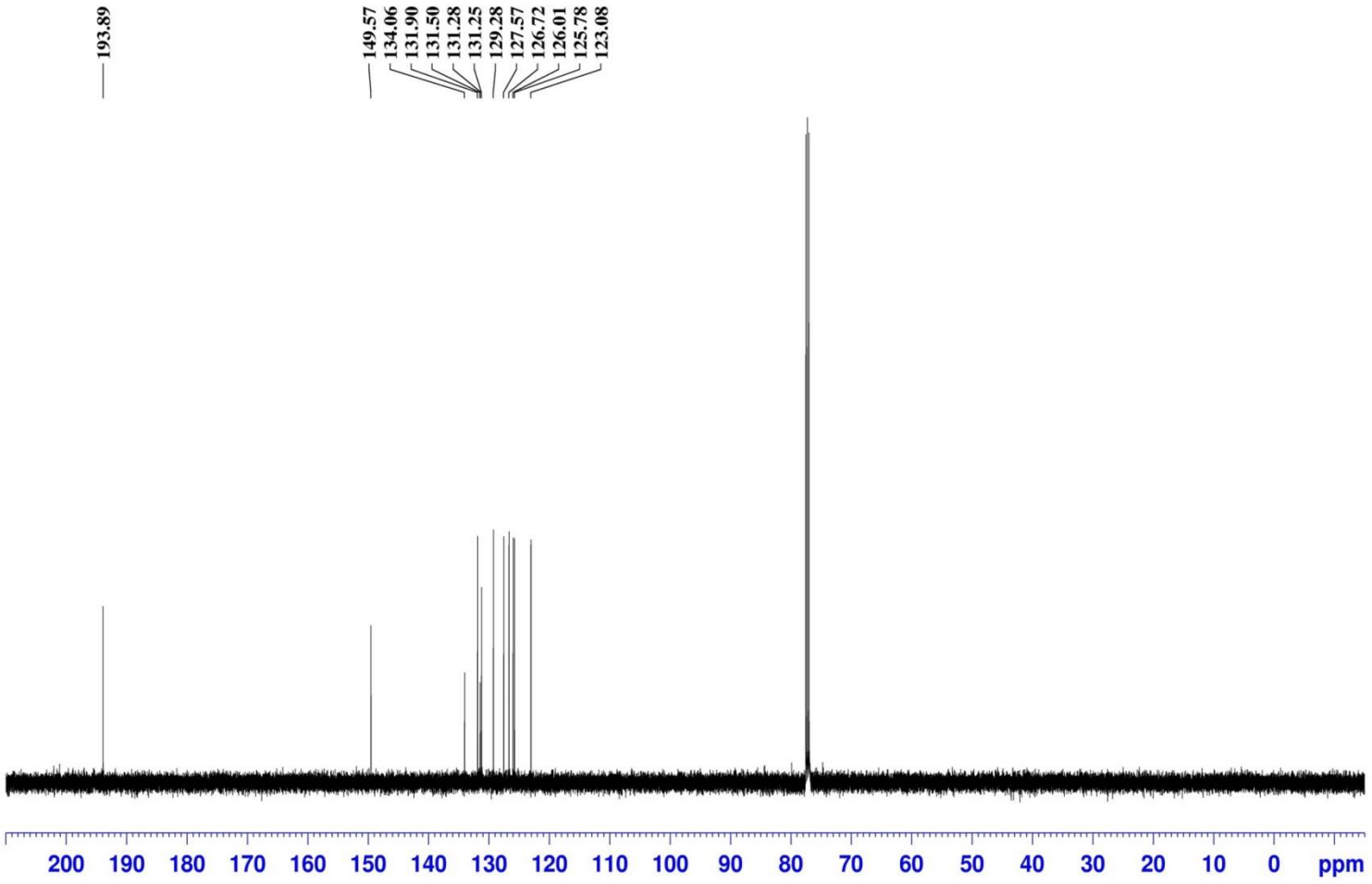
(E)−4−phenylbut−3−en−2−one
100 MHz, CDCl3



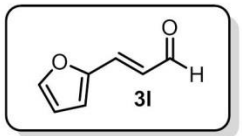


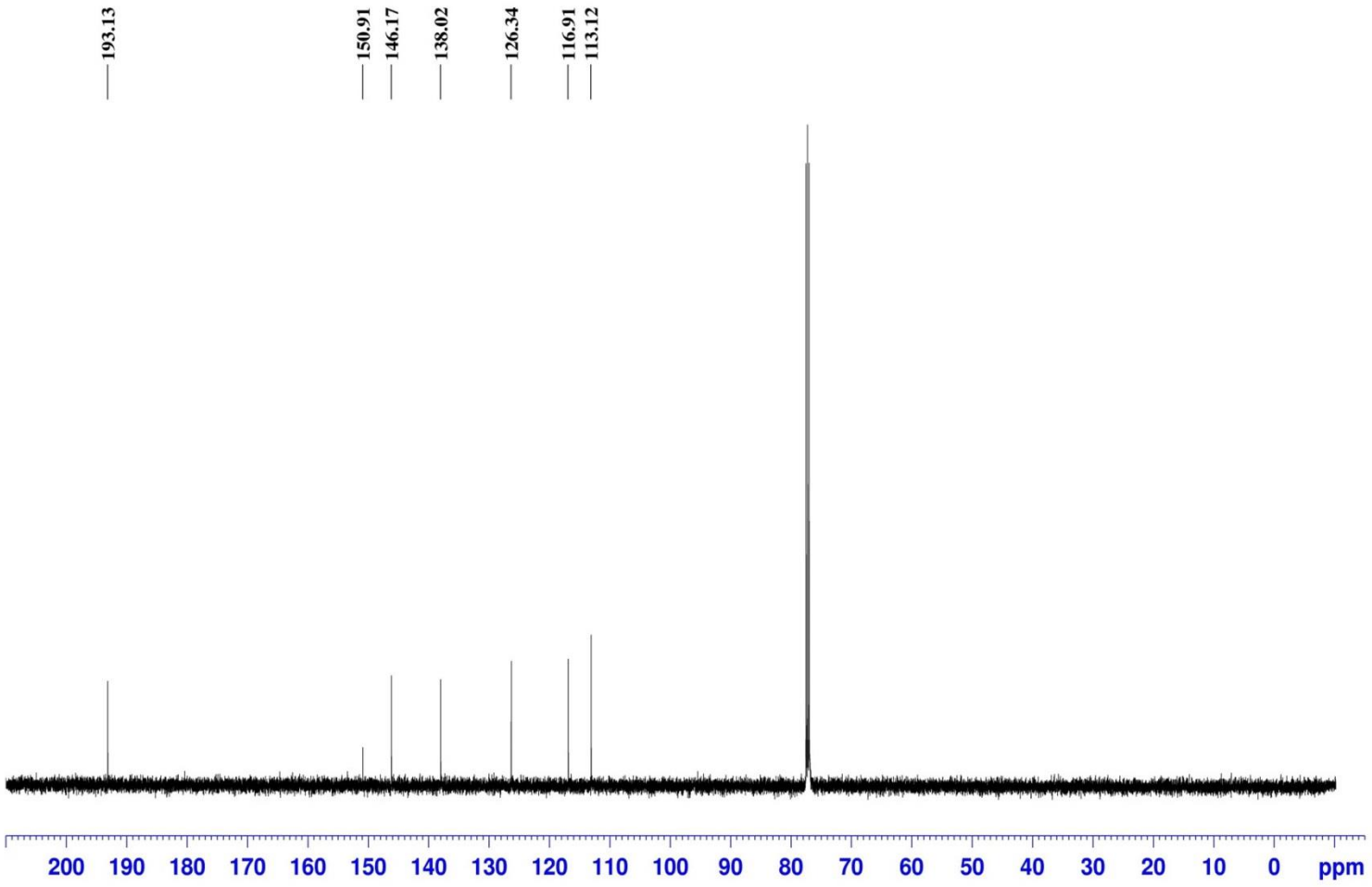
(E)−3−(naphthalen−1−yl)acrylaldehyde
125 MHz, CDCl3



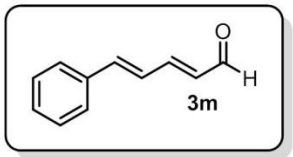


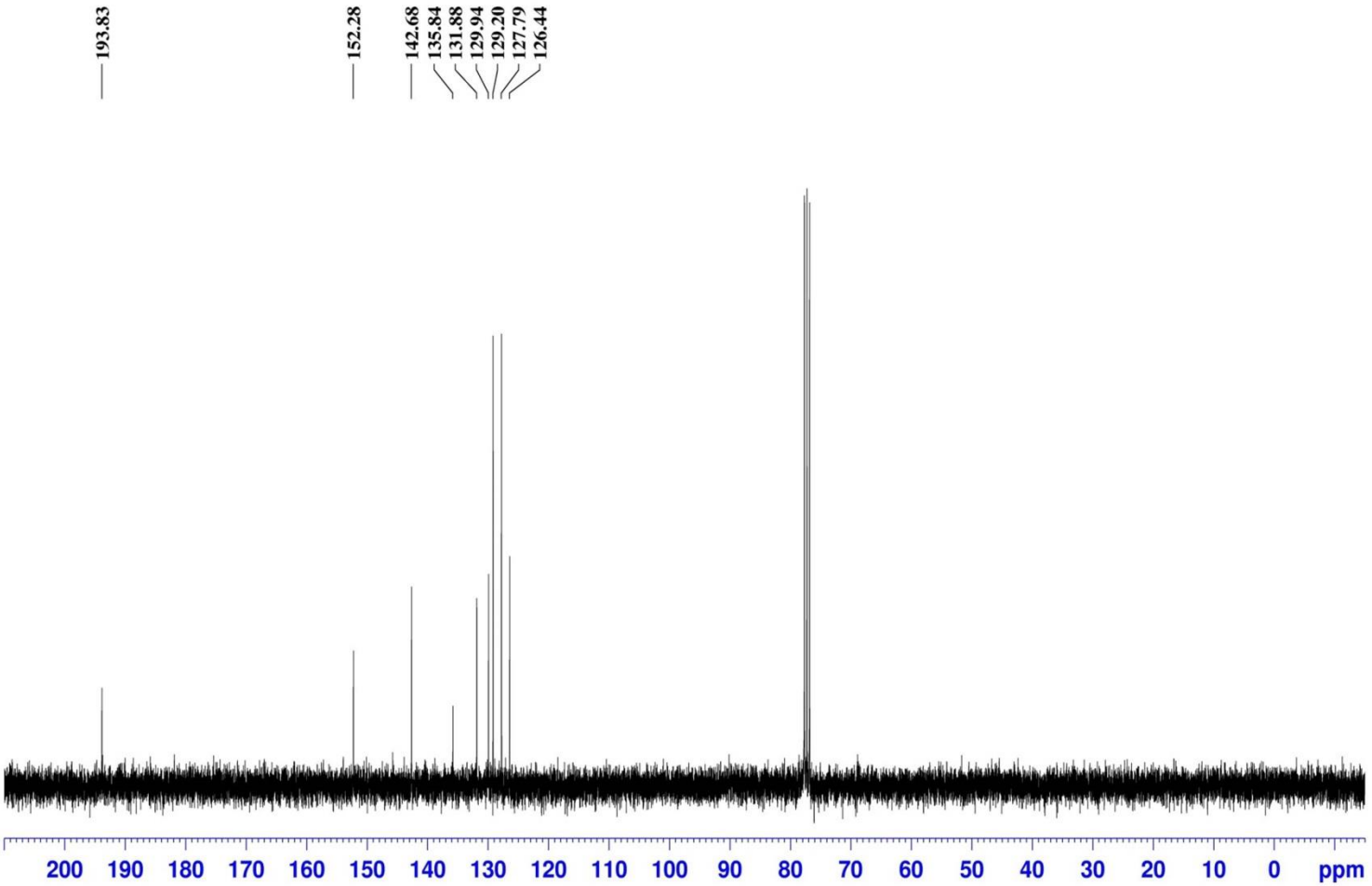
(E)−3−(furan−2−yl)acrylaldehyde
125 MHz, CDCl3



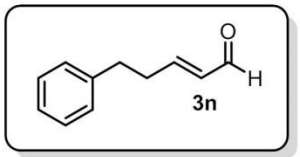


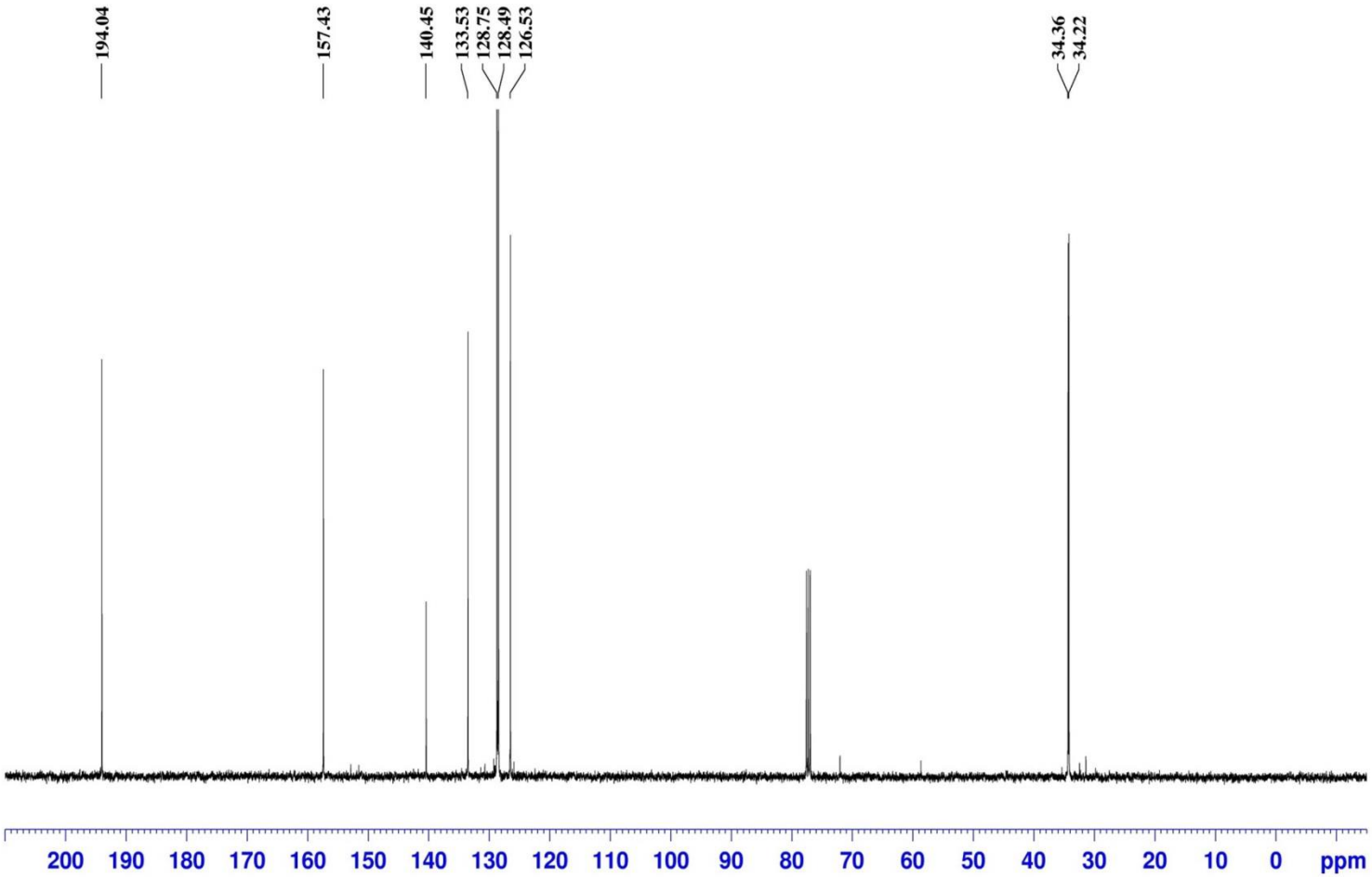
(2E,4E)−5−phenylpenta−2,4−dienal
75 MHz, CDCl3



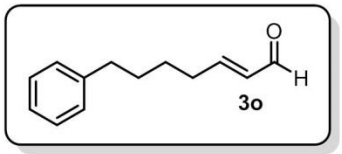


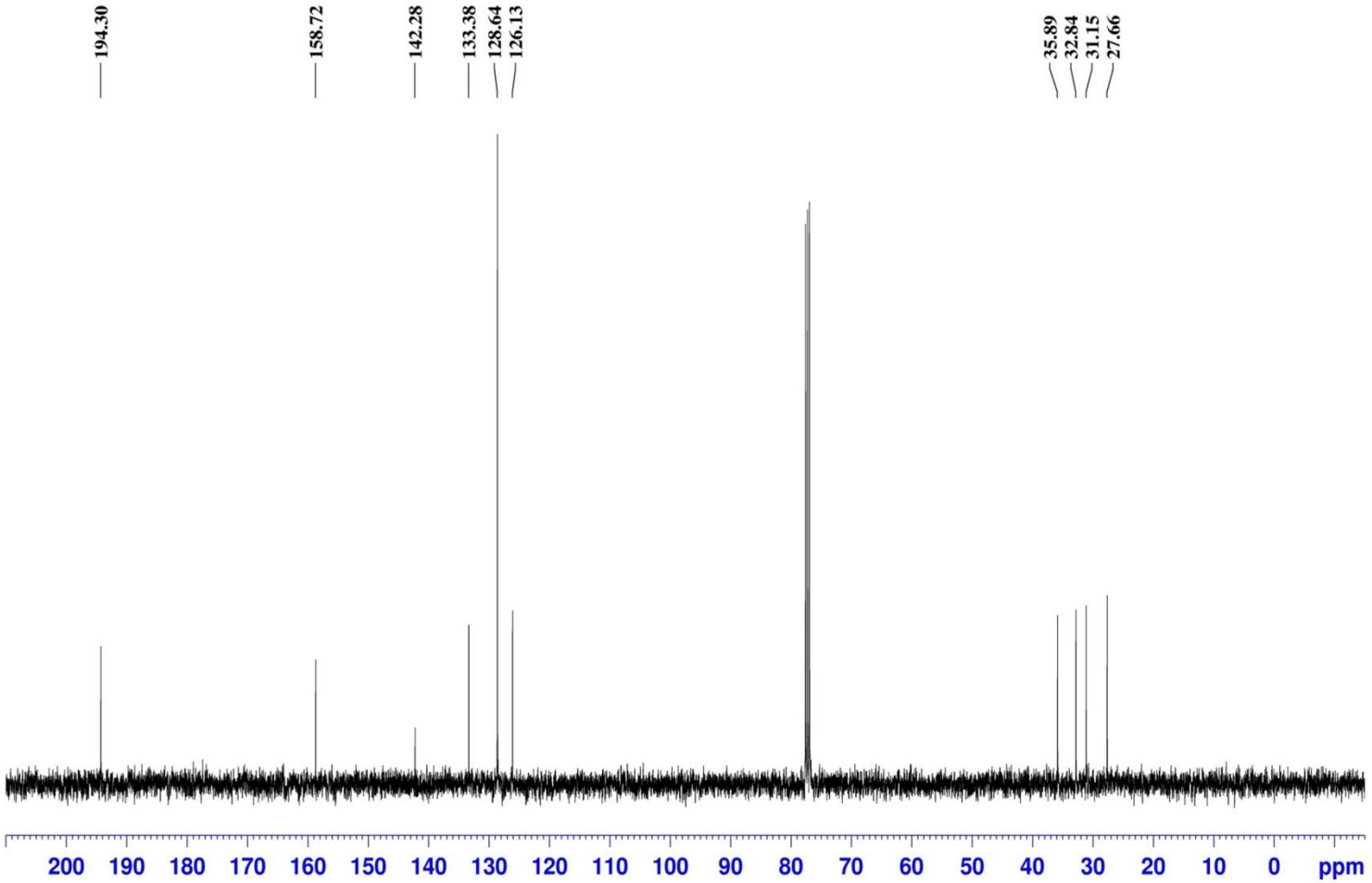
(E)−5−phenylpent−2−enal
100 MHz, CDCl3



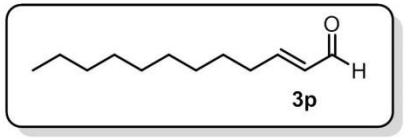


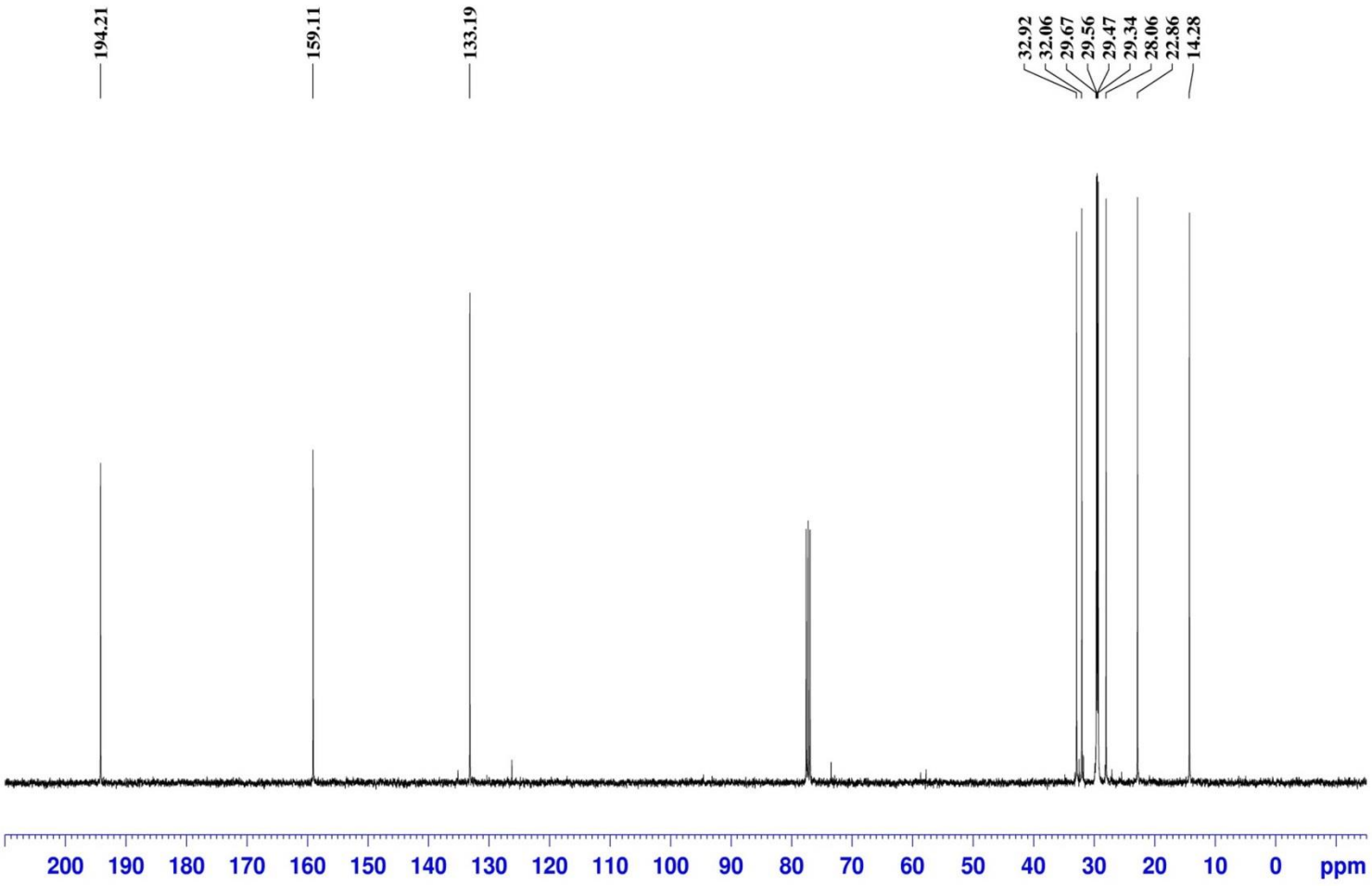
(E)−7−phenylhept−2−enal
100 MHz, CDCl3



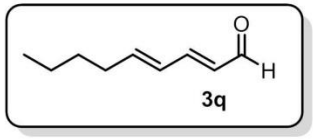


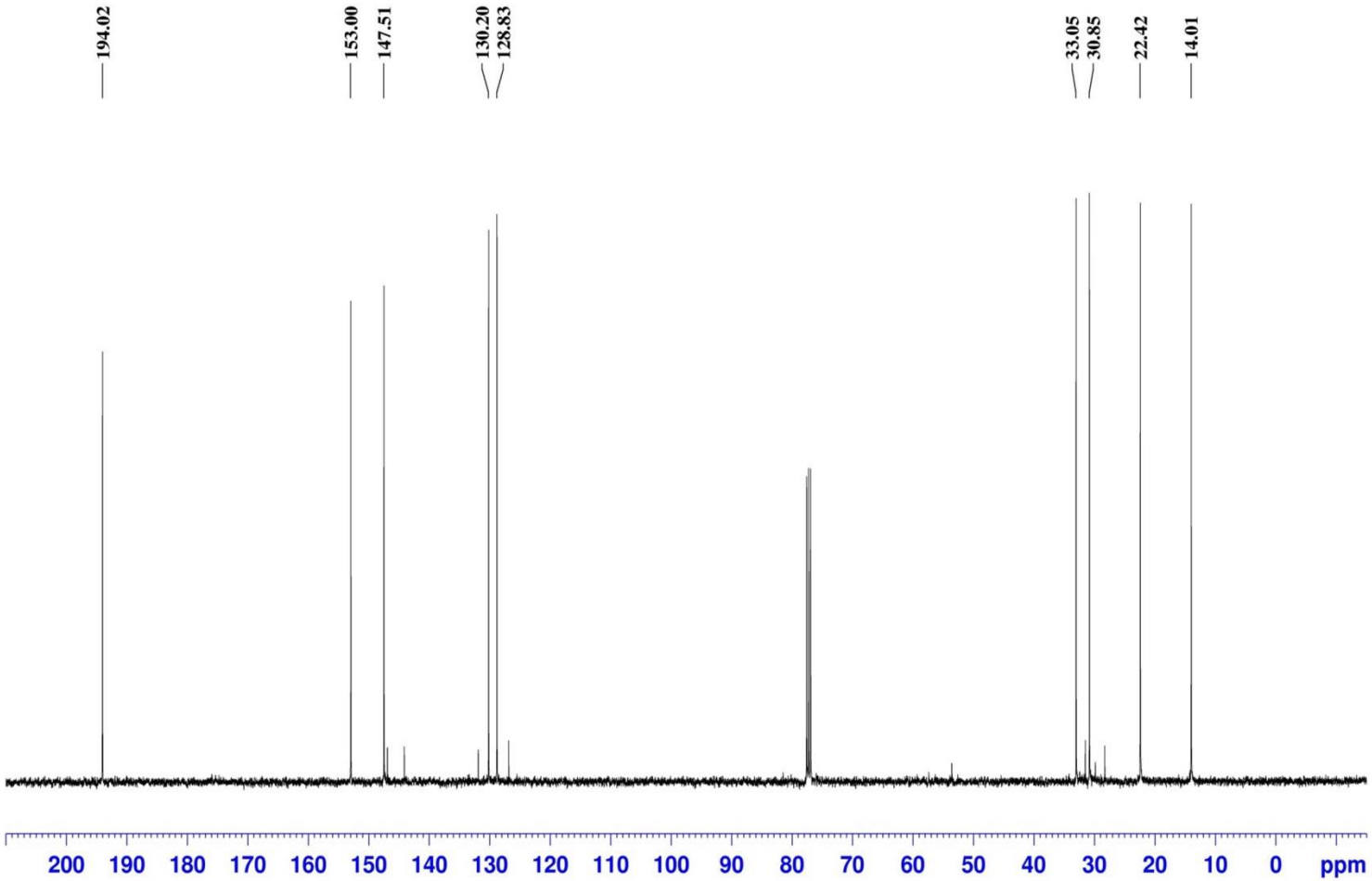
(E)−dodec−2−enal
100 MHz, CDCl3



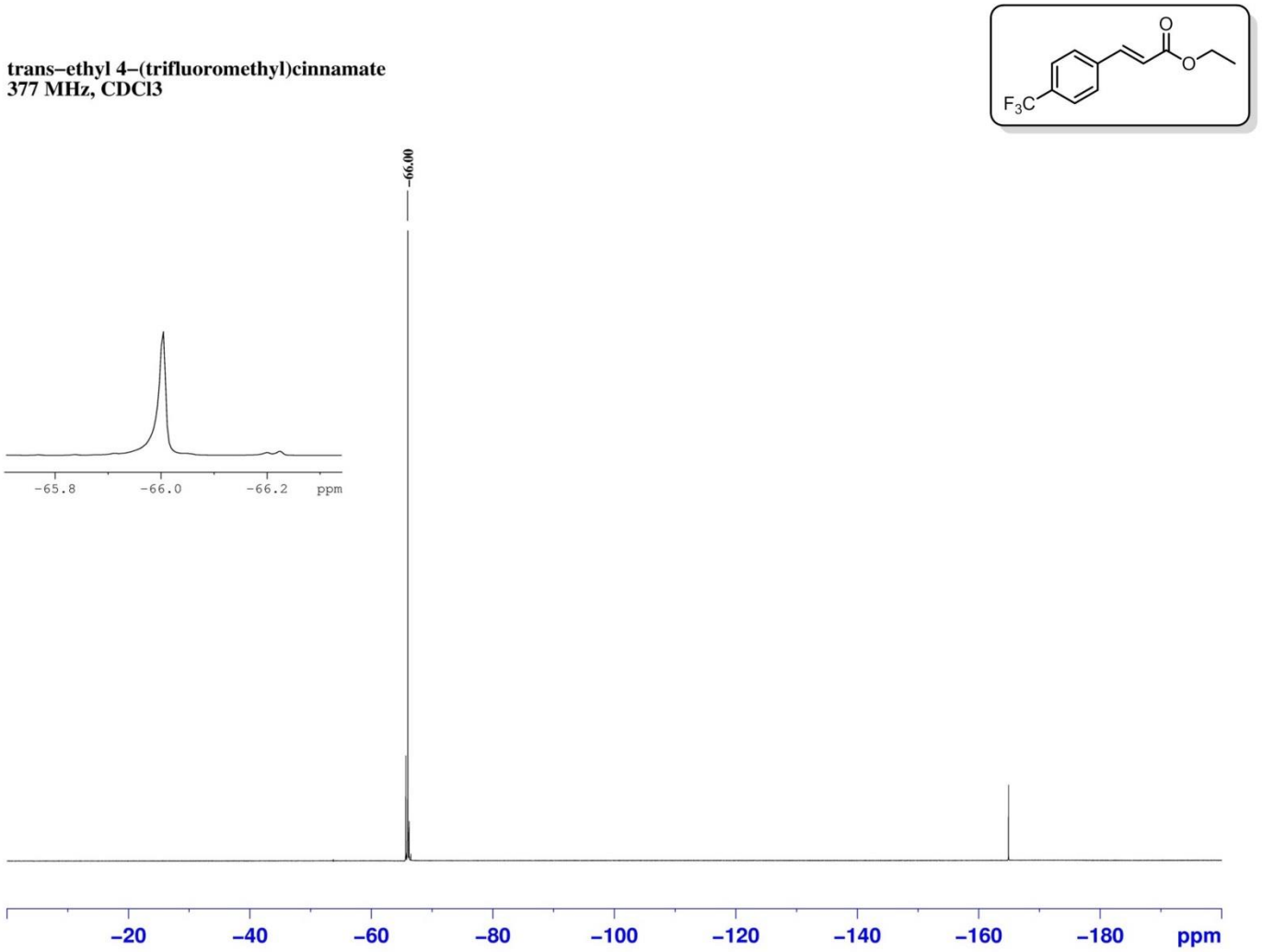


(2E,4E)−nona−2,4−dienal
100 MHz, CDCl3

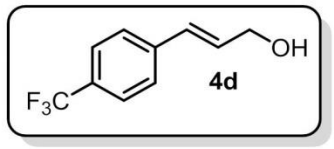


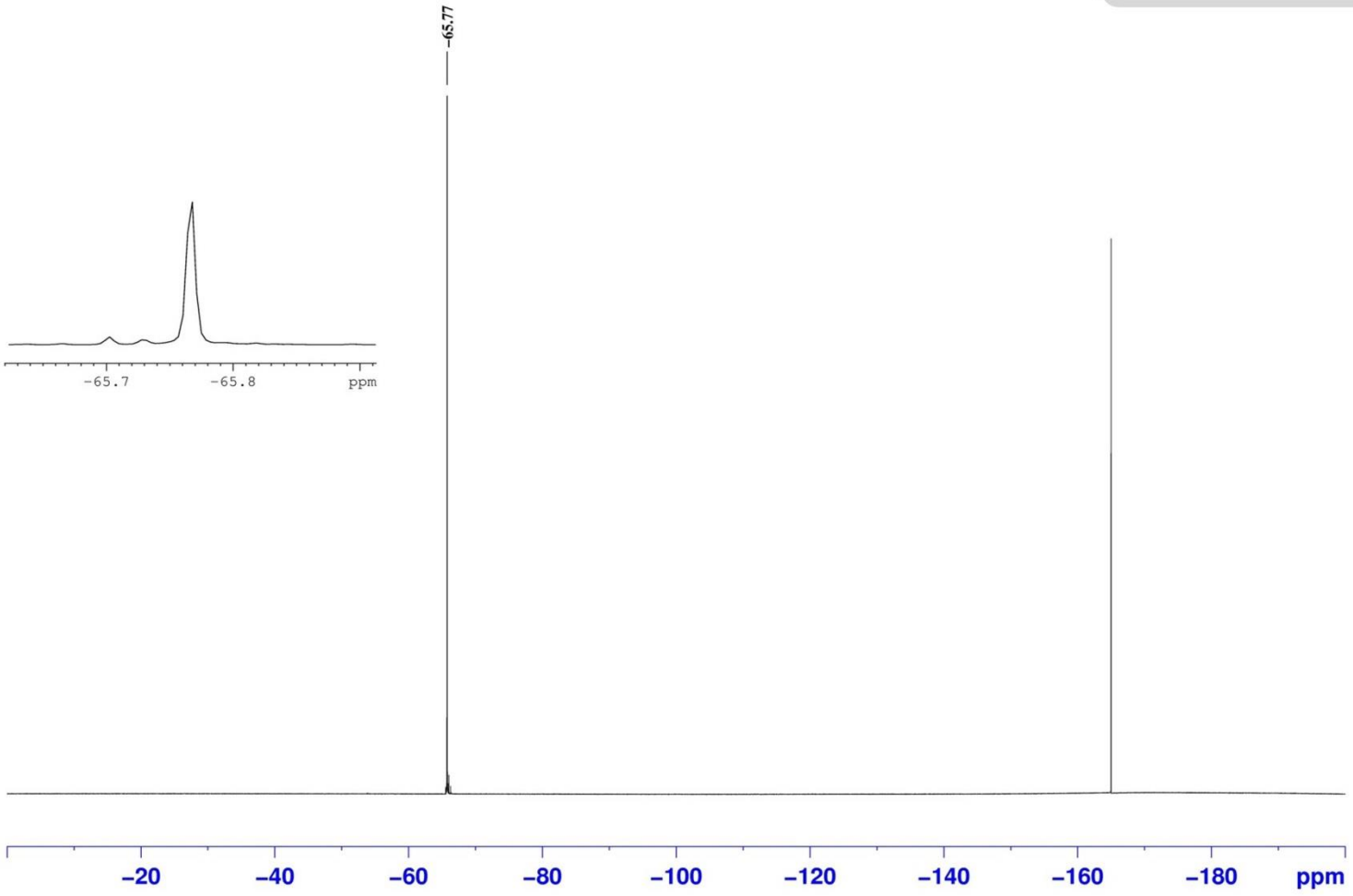


^{19}F NMR Spectra of Synthesized Compounds

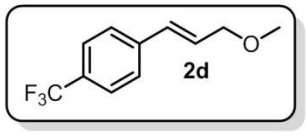


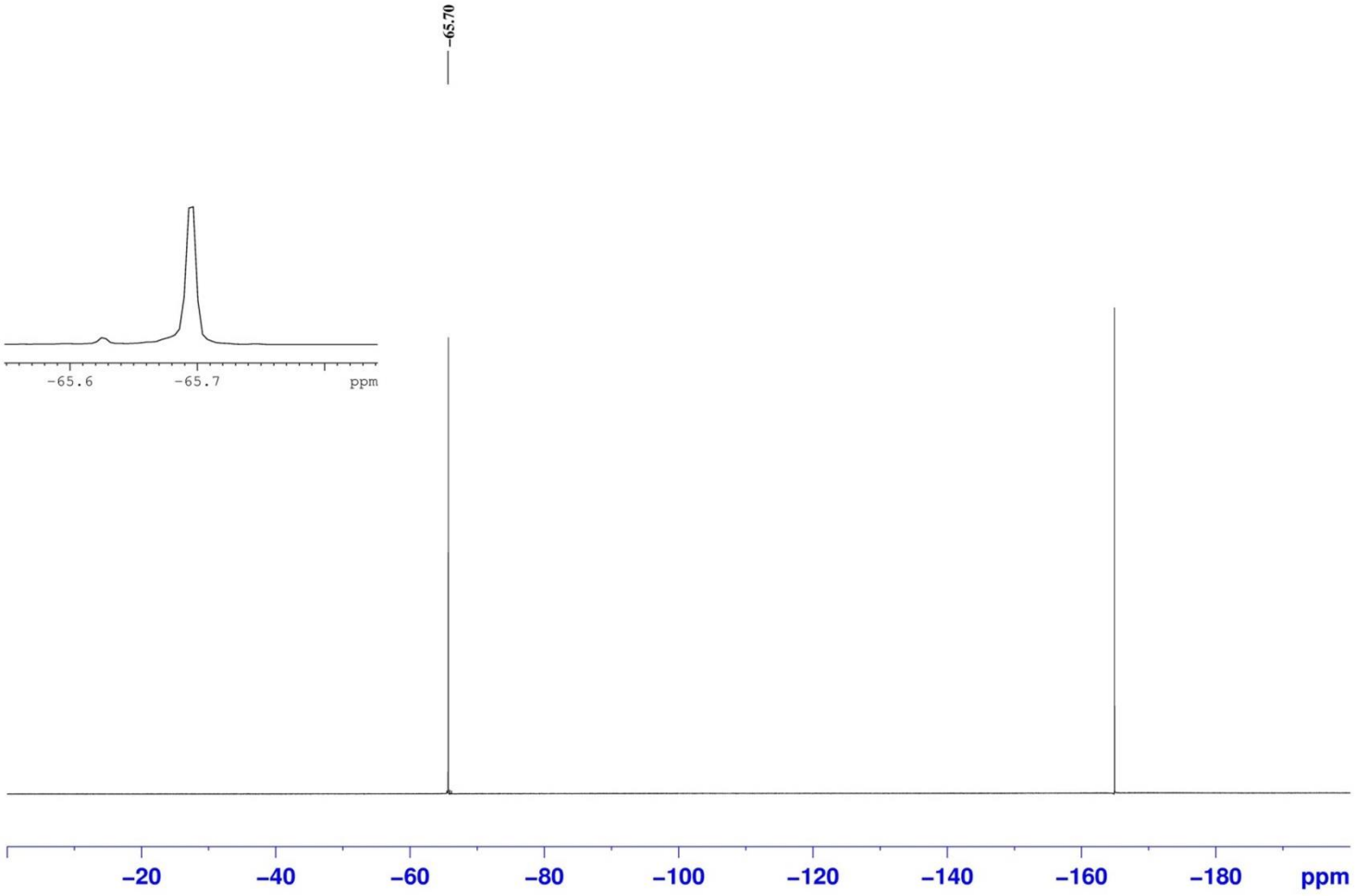
(E)−3−(4−(trifluoromethyl)phenyl)prop−2−en−1−ol
377 MHz, CDCl3





(E)−1−(3−methoxyprop−1−en−1−yl)−4−(trifluoromethyl)benzene
377 MHz, CDCl3





trans–4–(trifluoromethyl)cinnamaldehyde
377 MHz, CDCl3