Continuous Flow Hydrogenation Using an On-Demand Gas Delivery Reactor

Michael A. Mercadante, Christopher B. Kelly, Christopher Lee, and Nicholas E. Leadbeater*

Department of Chemistry, University of Connecticut, 55 N. Eagleville Road, Storrs, CT 06269, USA

nicholas.leadbeater@uconn.edu

Supporting Information

Key to abbreviated terms:

$CDCl_3$-Deuterated chloroform
DCM-Dichloromethane
EtOAc- Ethyl acetate
Hex: Hexanes
KO^tBu- Potassium *tert*-butoxide
THF – Tetrahydrofuran
TLC- Thin layer chromatography

General considerations:

General: All chemical transformations requiring inert atmospheric conditions or vacuum distillation utilized Schlenk line techniques with a 3, 4, or 5-port dual-manifold. Nitrogen was used to provide an inert atmosphere. NMR spectra (^1H, ^{13}C) were performed at 298 K on a Brüker Avance Ultra Shield 300 MHz, Brüker DRX-400 400 MHz, or Brüker Avance 500 MHz spectrometer. ^1H-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). Reactions were monitored either by an Agilent Technologies 7820A Gas Chromatograph attached to a 5975 Mass Spectrometer, ^1H-NMR or by TLC on silica gel plates. Percentage conversion of **1** to **2** was determined by integration of the terminal methyl on the propyl chain on both arenes in the ^1H-NMR spectrum of the product mixture.[a] TLC analysis was performed using hexanes/ethyl acetate 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 (60 Å porosity, 32-63 μm).

Chemicals: Deuterated NMR solvents ($CDCl_3$) were purchased from Cambridge Isotope Laboratories. $CDCl_3$ was stored over 4Å molecular sieves and K_2CO_3. Sodium sulfate, sodium carbonate, THF (reagent grade), acetone (reagent grade), anhydrous ethanol, benzene, CH_2Cl_2, diethyl ether (ACS Grade and reagent grade), allyl bromide, methylphosphonium bromide, $(PPh_3)_3RhCl$ (Wilkinson's catalyst), potassium *tert*-butoxide were purchased from Sigma-Aldrich Corporation. Hydrogen gas (ultra-high purity) was obtained from Airgas Inc. Unless otherwise specified, all alkenes were purchased from commercial sources and used without further purification.

[a] Anethole δ = 1.82 ppm; dihydroanethole δ = 0.91 ppm

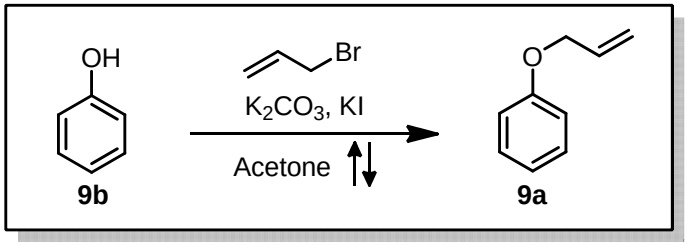
Description of the apparatus: Experiments were performed on a Vapourtec R series.[b] The system was equipped with two gas loading reactor coils. The "reagent out" port on the first reactor coil was connected to the "reagent in" port on second reactor coil using a 32 mm length of tubing. The "reagent out" port of the second reactor was linked into a T-piece which allowed a flow of acetone to mix with the reaction mixture departing the second reactor coil. The T-piece was finally equipped with a 250 psi back pressure regulator after which a length of tubing leading to a waste or collection flask.

The system was initially primed using the equipment manufacturer's suggested start-up sequence. ***CAUTION:*** *Make sure the "gas out" apertures vents into a fumehood since hydrogen is highly flammable.* After priming the unit, the reactor coils were each heated to 125 ^{o}C. Once at temperature, the system was ready for loading the reagent solution.

[b] http://www.vapourtec.co.uk

Synthesis of alkene precursors

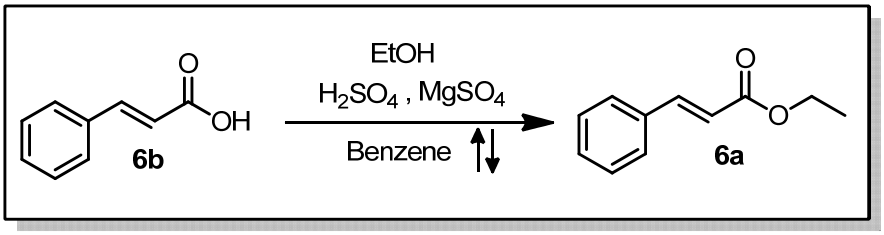
Synthesis of (allyloxy)benzene (9a)[1]



To a 500 mL round bottom flask was added a stir bar, **9b** (3.76 g, 40 mmol), K_2CO_3 (6.91 g, 150 mmol, 2.75 equiv), potassium iodide (0.664 g, 4 mmol, 0.1 equiv), allyl bromide (6.049 g, 50 mmol, 1.25 equiv), and acetone (180 mL, 0.22 M in **9b**). The flask was equipped with a reflux condenser and heated to reflux for 18 h. The flask was then allowed to cool and the solution filtered through a medium porosity sintered funnel. The solid was then washed with acetone. The acetone was removed *in vacuo* via rotary evaporation. The resulting residue was then diluted with 100 mL of diethyl ether and washed with 25 mL brine, 25 mL distilled water, and finally with 25 mL of brine. The organic layer was dried with Na_2SO_4 and the solvent removed *in vacuo* via rotary evaporation to give the crude **9a**. The crude material was then purified using flash chromatography using 95:5 (hexane:EtOAc) as an eluant to afford 6.65 g (82.6%) of **9a** as a clear colorless oil.

^{1}H NMR ($CDCl_3$, 300 MHz) δ ppm 4.57 (dt, *J*=5.32, 1.50 Hz, 2 H) 5.32 (dq, *J*=10.51, 1.47 Hz, 1 H) 5.46 (dq, *J*=17.27, 1.66 Hz, 1 H) 6.10 (ddt, *J*=17.24, 10.51, 5.32, 5.32 Hz, 1 H) 6.94 - 7.02 (m, 3 H) 7.29 - 7.36 (m, 2 H) **^{13}C NMR** ($CDCl_3$, 101 MHz) δ ppm 68.95 (CH) 115.00 (CH) 117.81 (CH_2) 121.08 (CH) 129.69 (CH) 133.64 (CH) 158.86 (C) **GC-MS** (EI) 135 ($[M+1]^+$, 10%), 134 ($[M]^+$, 100%), 133 (33%), 119 (39%), 105 (18%), 94 (38%), 91 (44%), 79 (11%), 78 (22%), 77 (24%), 66 (22%), 65 (44%), 63 (15%), 51 (18%), 41 (50%), 39 (57%), 38 (10%)

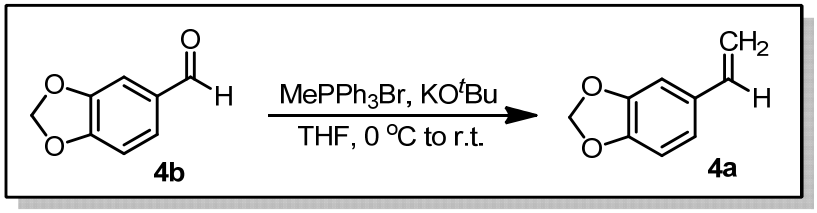
Synthesis of Ethyl Cinnamate (6a)[2]



To a 250 mL round bottom flask was added the *trans*-cinnamic acid, (37.04 g, 0.25 mol, 1 equiv.), absolute ethanol (51.75 g, 1.125 mol, 4.5 equiv.) and benzene (50 mL, 5 M in *trans*-cinnamic acid). The flask was equipped with a stirbar, a Soxhlet extractor equipped 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 (2.56 mL, 0.0475 mol, 0.19 equiv.) was added all at once via a syringe. The solution was heated to reflux. The solution was then diluted with ether (≈100 mL). The mixture was transferred to a separatory funnel and washed with 3 × 100 mL of a 10 wt% 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 pure ethyl cinnamate as a clear yellow oil (38.49 g, 87%).

^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 1.34 (t, *J*=7.09 Hz, 3 H) 4.27 (q, *J*=7.17 Hz, 2 H) 6.44 (d, *J*=16.14 Hz, 1 H) 7.31 - 7.43 (m, 3 H) 7.52 (q, *J*=3.70 Hz, 2 H) 7.69 (d, *J*=16.14 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 101 MHz) δ ppm 14.55 (CH_3) 60.70 (CH_2) 118.52 (CH) 128.26 (CH) 129.09 (CH) 130.42 (CH) 134.71 (C) 144.79 (CH) 167.18 (C) **GC-MS** (EI) 176 ($[M]^+$, 35%), 148 (17%), 147 (19%), 132 (12%), 131 (100%), 104 (16%), 103 (52%), 102 (16%), 77 (33%), 51 (11%).

Synthesis of 5-vinylbenzo[d][1,3]dioxole (4a)[3]



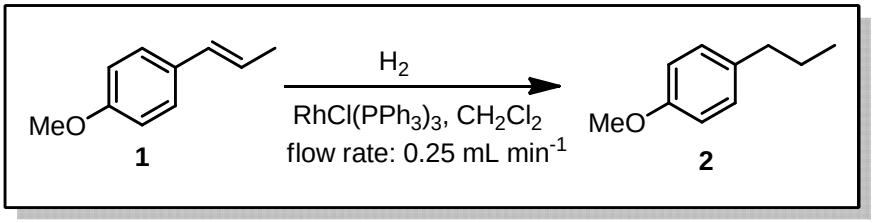
To a 500 mL three-neck round bottom flask was added methylphosphonium bromide (22.86 g, 0.064 mol, 1.28 equiv.), THF (231 mL, 0.28 M in potassium *tert*-butoxide), and stir bar. The flask was then equipped with two septa and a nitrogen gas inlet and cooled to 0 °C in a ice bath. After 5 minutes, potassium *tert*-butoxide (95% w/w, 7.70 g, 0.065 mol, 1.3 equiv) was added in 1 g portions over 10 minutes during which the solution became yellow in color. The solution

was allowed to stir for another 20 minutes at 0 °C under nitrogen. At this point, **4b** (7.506 g, 0.050 mol) was dissolved in 24 mL of THF (2.13 M) and added to the reaction flask dropwise *via* syringe, turning the solution white. The solution was then allowed to warm to room temperature over 1 h. The solution was transferred to a 1000 mL separatory funnel and diluted with ether. The solution was then washed 2 X 100 mL saturated ammonium chloride. The organic layer was then washed with 100 mL of distilled water, 100 mL of brine and dried using Na_2SO_4. The solvent was removed *in vacuo* by rotary evaporation to afford crude **4a** which was purified by vacuum distillation (b.p. 50-52 °C @ 0.20 mmHg) and obtained as a clear liquid (3.838 g, 52%)

^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 5.15 (dd, *J*=10.88, 0.61 Hz, 1 H) 5.59 (dd, *J*=17.61, 0.73 Hz, 1 H) 5.96 (s, 2 H) 6.64 (dd, *J*=17.48, 10.88 Hz, 1 H) 6.76 - 6.79 (m, 1 H) 6.83 - 6.87 (m, 1 H) 6.98 (d, *J*=1.71 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 101 MHz) δ ppm 101.29 (CH_2) 105.65 (CH) 108.43 (s, CH) 112.19 (s, CH_2) 121.25 (s, CH) 132.40 (s, C) 136.63 (s, CH) 147.62 (s, C) 148.26 (s, C) **GC-MS** (EI) 149 ($[M+1]^+$, 10%), 148 ($[M]^+$, 100%), 147 (100%), 91 (21%), 90 (10%), 89 (42%), 63 (20%).

General procedure for hydrogenation

General procedure for hydrogenation reactions (small scale)



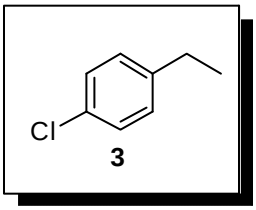
A 10 mL test tube was charged with Wilkinson's catalyst (0.0555 g, 0.060 mmol 1.2 mol %), anethole, **1**, (0.741 g, 5 mmol), and 1 mL of CH_2Cl_2. The solution was thoroughly mixed using sonication until it became a completely homogenous clear dark red solution. The flow reactor was readied using the equipment manufacturer's suggested start-up sequence followed by heating the reactor coils to 125 °C. The reaction mixture was then loaded into the reactor. Product collection was commenced immediately after this switch. After the reaction mixture had been completely loaded into the reactor, the reactor pump was set back to pumping dichloromethane. After the product had been fully discharged from the reactor coils, the resulting clear yellow solution could then be purified.

Isolation procedure

Dichloromethane and acetone were removed using rotary evaporation leaving the crude product. The crude product was dissolved in a small amount of the elution solution (9:1 Hexane:EtOAc) and loaded onto a plug of silica. The plug was rinsed thoroughly with eluting solution and the solvent was stripped *in vacuo* using a room temperature[c] water bath. This process was continued until constant weight was obtained. Pure 1-methoxy-4-propylbenzene[4], **2,** was obtained as a clear colorless oil (0.668 g, 89%). **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 0.99 (t, *J*=7.25 Hz, 3 H) 1.67 (sxt, *J*=7.60 Hz, 2 H) 2.58 (t, *J*=7.60 Hz, 2 H) 3.83 (s, 3 H) 6.88 (d, *J*=8.83 Hz, 2 H) 7.14 (d, *J*=8.20 Hz, 2H) **^{13}C NMR** ($CDCl_3$,125 MHz) δ ppm 14.02 (CH_3), 25.06 (CH_2), 37.41 (CH_2), 55.44 (CH_3), 113.88 (CH), 128.53(CH), 135.02 (C), 157.91 (C) **GC-MS** (EI) 151 ($[M+1]^+$, 5%), 150 ($[M]^+$, 44%), 122 (18%), 121 (100%), 91 (16%), 78 (16%), 77 (17%), 65 (6%).

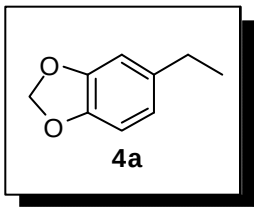
[c] Note that it was necessary to keep the bath water at room temperature in most cases due to the volatility of the alkane products

Spectral data for hydrogenation products



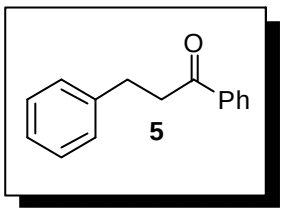
3

1-Chloro-4-ethylbenzene[5](3) (0.540, 77%) was prepared according to the representative procedure from *p*-chlorostyrene (0.693 g, 5 mmol) (**3a**) as a faint yellow oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 1.28 (t, *J*=7.57 Hz, 3 H) 2.67 (q, *J*=7.57 Hz, 2 H) 7.17 (d, *J*=8.83 Hz, 2 H) 7.30 (d, *J*=8.83 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 15.76 (CH_3), 28.52(CH_2), 128.62 (CH), 129.45 (CH), 131.54 (C), 142.86 (C) **GC-MS** (EI) 142 ($[M+2]^+$, 14%), 148 ($[M]^+$, 43%), 127 (35%), 125 (100%), 105 (48%), 103 (12%), 89 (16%), 77 (15%).



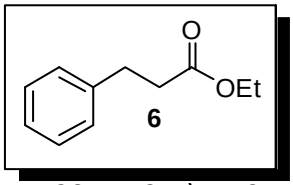
4a

5-Ethylbenzo[d][1,3]dioxole[6] (4a) (0.647 g, 86%) was prepared according to the representative procedure from 5-vinylbenzo[*d*][1,3]dioxole (0.741 g, 5 mmol) (4**a**) as a faint yellow oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 1.27 (t, *J*=7.57 Hz, 3 H) 2.63 (q, *J*=7.57 Hz, 2 H) 5.95 (s, 2 H) 6.70 (d, *J*=7.57 Hz, 1 H) 6.76 (s, 1 H) 6.79 (d, *J*=8.20 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 126 MHz) δ ppm 16.16 (CH_3), 28.87(CH_2), 100.89(CH_2), 108.28(CH), 108.61(CH), 120.59(CH), 138.38 (C), 145.66 (C), 147.76 (C) **GC-MS** (EI) 150 ($[M]^+$, 42%), 136 (9%), 135 (100%), 91 (11%), 77 (16%), 65 (7%), 63 (7%), 51 (7%).

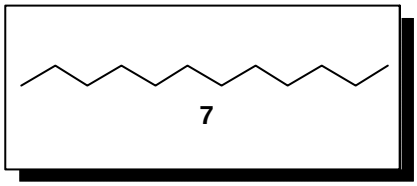


5

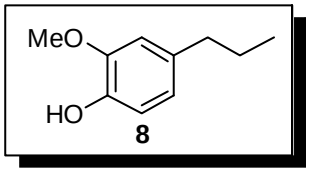
1,3-Diphenylpropan-1-one[7] (5) (1.039 g, 97%) was prepared according to the representative procedure from *trans*-chalcone (1.041 g, 5 mmol) (**5a**) as a off-white powdery solid. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 3.14 (t, *J*=7.60 Hz, 2 H) 3.36 (t, *J*=7.60 Hz, 2 H) 7.25 - 7.30 (m, 1 H) 7.31 - 7.40 (m, 4 H) 7.50 (t, *J*=7.60 Hz, 2 H) 7.61 (t, *J*=7.60 Hz, 1 H) 8.02 (d, *J*=6.94 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 30.30 (CH_2) 40.60 (CH_2) 126.32 (CH) 128.22 (CH) 128.62 (CH) 128.71 (CH) 128.78 (CH) 133.23 (CH) 137.04 (C) 141.49 (C) 199.31 (C) **GC-MS** (EI) 211 ($[M+1]^+$, 10%), 210 ($[M]^+$, 60%), 105 (100%), 91 (11%), 77 (47%), 51 (13%).



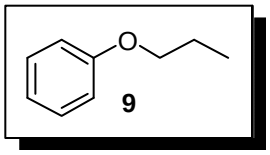
Ethyl 3-phenylpropanoate[8] (6) (0.872 g, 98%) was prepared according to the representative procedure from *trans*-ethyl cinnamate (0.881 g, 5 mmol) (**6a**) as a colorless oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm1.25 (t, *J*=7.20 Hz, 3 H) 2.64 (t, *J*=8.00 Hz, 2 H) 2.98 (t, *J*=7.88 Hz, 2 H) 4.15 (q, *J*=7.20 Hz, 2 H) 7.19 - 7.25 (m, 3 H) 7.27 - 7.34 (m, 2 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 14.26 (CH_3) 31.05 (CH_2) 35.98 (CH_2) 60.39 (CH_2) 126.29 (CH) 128.37 (CH) 128.53 (CH) 140.67 (C) 172.83 (C) **GC-MS** (EI) 178 ([M]$^+$, 42%), 133 (14%), 107 (42%), 105 (47%), 104 (100%), 103 (16%), 91 (52%), 79 (18%), 78 (14%), 77 (22%).



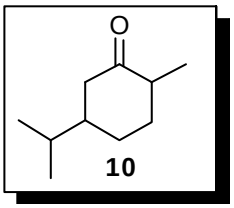
Dodecane[9] (7) (0.784 g, 92%) was prepared according to the representative procedure from 1-dodecene (0.886 g, 5 mmol) (**7a**) as a faint yellow oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 0.90 (t, *J*=6.94 Hz, 6 H) 1.21 - 1.35 (m, 20 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 14.40 (CH_3) 23.06 (CH_2) 30.05 (CH_2) 30.09 (CH_2) 32.32 (CH_2) **GC-MS** (EI) 170 ([M]$^+$, 13%), 99 (11%), 98 (11%), 85 (47%), 84 (11%), 71 (69%), 70 (17%), 69 (11%), 57 (100%), 56 (20%), 55 (26%), 43 (74%), 42 (15%), 41 (61%), 39 (15%).



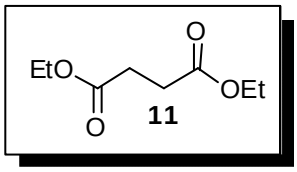
2-Methoxy-4-propylphenol[10] (8) (0.789 g, 95%) was prepared according to the representative procedure from eugenol (0.821 g, 5 mmol) (**8a**) as a yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 0.97 (t, *J*=7.34 Hz, 3 H) 1.65 (sxt, *J*=7.48 Hz, 2 H) 2.55 (t, *J*=7.60 Hz, 2 H) 3.89 (s, 3 H) 5.57 (s, 1 H, **OH**) 6.68 - 6.73 (m, 2 H) 6.86 (d, *J*=7.58 Hz, 1 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 14.01 (CH_3) 25.08 (CH_2) 37.99 (CH_2) 56.05 (CH_3) 111.29 (CH) 114.35 (CH) 121.20 (CH) 134.91 (C) 143.78 (C) 146.54 (C) **GC-MS** (EI) 166 ([M]$^+$, 31%), 138 (11%), 137 (100%), 122 (14%), 94 (10%), 77 (7%).



Propoxybenzene[11] **(9)** (0.545 g, 80%) was prepared according to the representative procedure from (allyloxy)benzene (0.671 g, 5 mmol) (**9a**) as a colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ ppm 1.09 (t, *J*=7.60 Hz, 3 H) 1.86 (sxt, *J*=7.09 Hz, 2 H) 3.96 (t, *J*=6.60 Hz, 2 H) 6.92 - 7.02 (m, 3 H) 7.33 (t, *J*=7.70 Hz, 2 H) **^{13}C NMR** ($CDCl_3$, 100 MHz) δ ppm 10.79 (CH_3) 22.88 (CH_2) 69.63 (CH_2) 114.77 (CH) 120.73 (CH) 129.66 (CH) 159.39 (C) **GC-MS** (EI) 136 ($[M]^+$, 33%), 95 (7%), 94 (100%), 77 (13%), 66 (17%), 65 (12%), 51 (7%), 39 (11%).

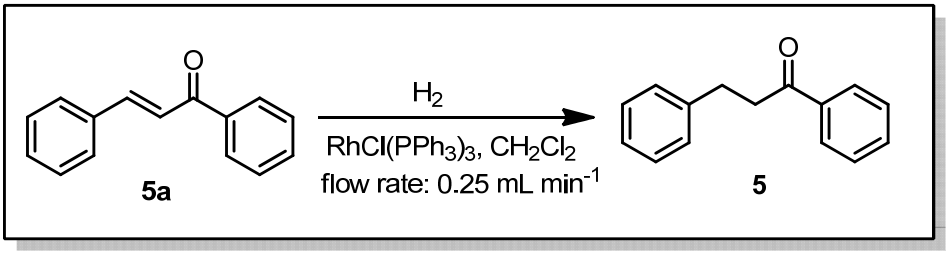


5-Isopropyl-2-methylcyclohexanone[12] **(10)** (0.732 g, 95%) was prepared according to the representative procedure from racemic dihydrocarvone (0.761 g, 5 mmol) (**10a**) as a faint yellow oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 0.83 (t, *J*=6.20 Hz, 6 H) 0.91 - 1.05 (m, 3 H) 1.23 (qd, *J*=12.60, 3.80 Hz, 1 H) 1.32 - 1.44 (m, 1 H) 1.44 - 1.70 (m, 2 H) 1.74 - 1.86 (m, 1 H) 1.92 - 2.10 (m, 2 H) 2.21 - 2.42 (m, 2 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 14.51 (CH_3) 19.49-19.76 (CH_3 both isomers) 29.06 (CH_2) 32.91 (CH) 35.26 (CH_2) 45.03 (CH) 45.52 (CH_2) 46.75 (CH) 213.65 (C) **GC-MS** (EI) 154 ($[M]^+$, 35%), 112 (11%), 111 (100%), 110 (12%), 97 (14%), 95 (20%), 83 (20%), 69 (19%), 55 (62%), 43 (11%), 42 (12%), 41 (36%), 39 (18%).



Diethyl succinate[13] **(11)** (0.855 g, 98%) was prepared according to the representative procedure from diethyl fumerate (0.861 g, 5 mmol) (**3a**) as a colorless oil. **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 1.21 (t, *J*=7.10 Hz, 6 H) 2.57 (s, 4 H) 4.10 (q, *J*=7.20 Hz, 4 H) **^{13}C NMR** ($CDCl_3$, 125 MHz) δ ppm 14.34 (CH_3) 29.36 (CH_2) 60.82 (CH_2) 172.49 (C) **GC-MS** (EI) 174 ($[M]^+$, 1%), 147 (1%), 129 (74%), 128 (19%), 102 (14%), 101 (100%), 74 (8%), 73 (17%), 56 (9%), 55 (17%).

General procedure for hydrogenation reactions (large scale)

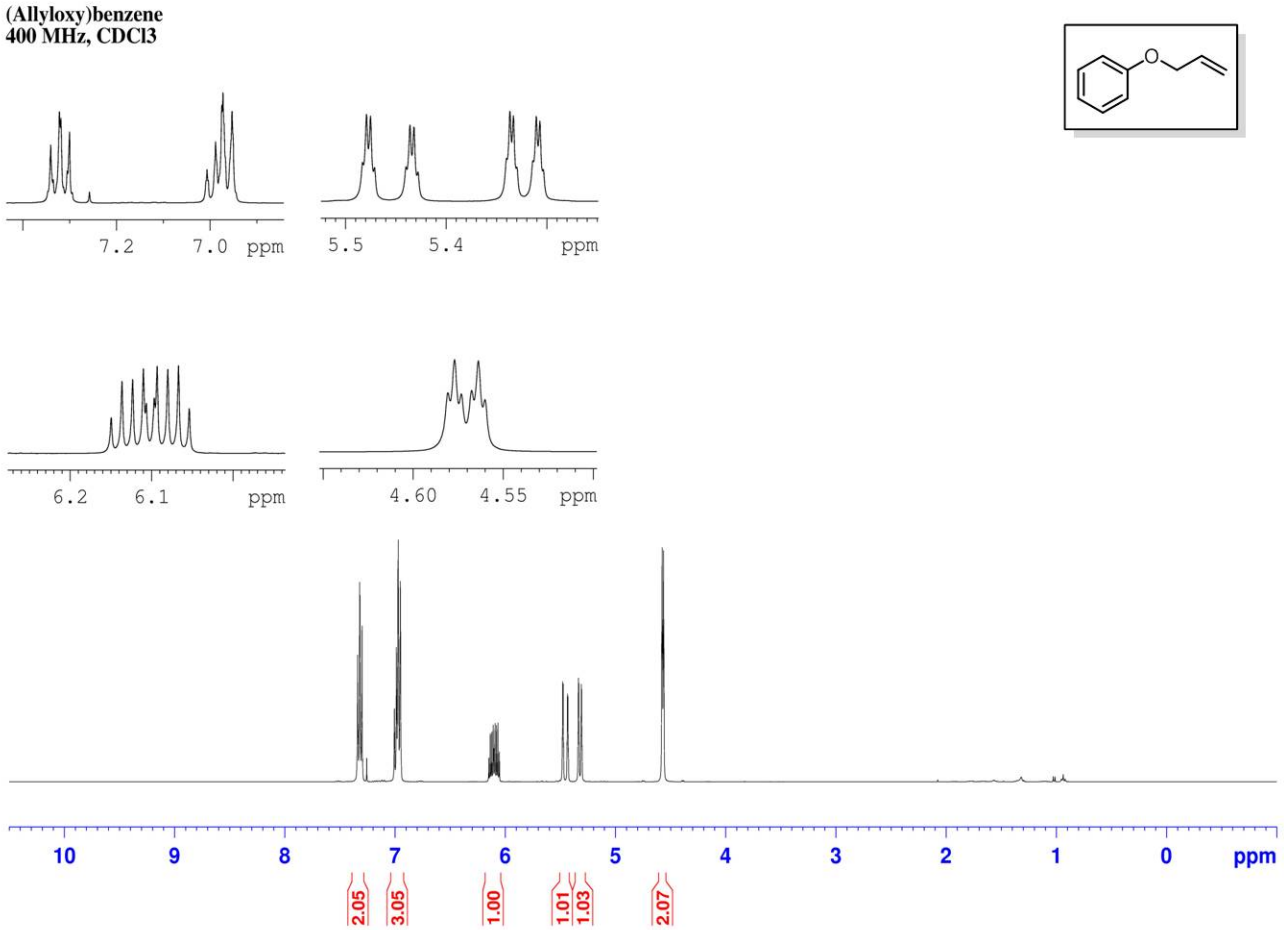


A 50 mL conical flask was charged with Wilkinson's catalyst (1.000 g, 1.08 mmol 1.2 mol %), **5a** (19.33 g, 92.8 mmol, *97% purity*), and CH_2Cl_2 (30 mL). The solution was thoroughly mixed using a stirbar until it became a completely homogenous clear dark red solution. The flow reactor was readied using the equipment manufacturer's suggested start-up sequence followed by heating the reactor coils to 125 °C. The reaction mixture was then loaded into the reactor. Product collection was commenced immediately after this switch. After the reaction mixture had been completely loaded into the reactor, the reactor pump was set back to pumping dichloromethane. After the product had been fully discharged from the reactor coils, the resulting clear yellow solution could then be purified.

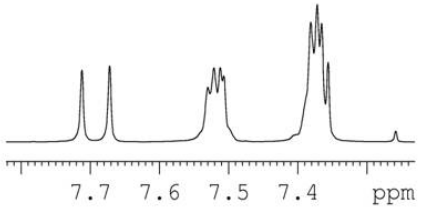
Isolation Procedure

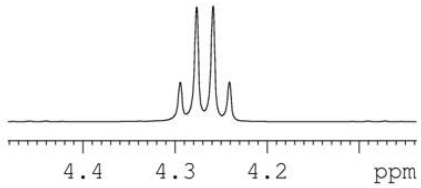
CH_2Cl_2 and acetone were removed using rotary evaporation leaving the crude product. The crude product was dissolved in a small amount of the elution solution (8:2 Hexane:EtOAc) and loaded onto a plug of silica. The plug was rinsed thoroughly with eluting solution and the solvent was stripped *in vacuo* in a 50 °C water bath. This process was repeated until constant weight was obtained. Pure **5**[7]**,** was obtained as a off-white powdery solid (18.910 g, 99%). **^{1}H NMR** ($CDCl_3$, 500 MHz) δ ppm 0.99 (t, *J*=7.25 Hz, 3 H) 1.67 (sxt, *J*=7.60 Hz, 2 H) 2.58 (t, *J*=7.60 Hz, 2 H) 3.83 (s, 3 H) 6.88 (d, *J*=8.83 Hz, 2 H) 7.14 (d, *J*=8.20 Hz, 2H) **^{13}C NMR** ($CDCl_3$,125 MHz) δ ppm 14.02 (CH_3), 25.06 (CH_2), 37.41 (CH_2), 55.44 (CH_3), 113.88 (CH), 128.53(CH), 135.02 (C), 157.91 (C) **GC-MS** (EI) 151 ($[M+1]^+$, 5%), 150 ($[M]^+$, 44%), 122 (18%), 121 (100%), 91 (16%), 78 (16%), 77 (17%), 65 (6%).

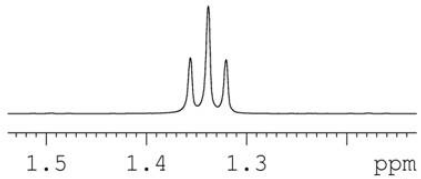
^{1}H-NMR spectra of synthesized compounds

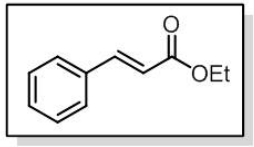


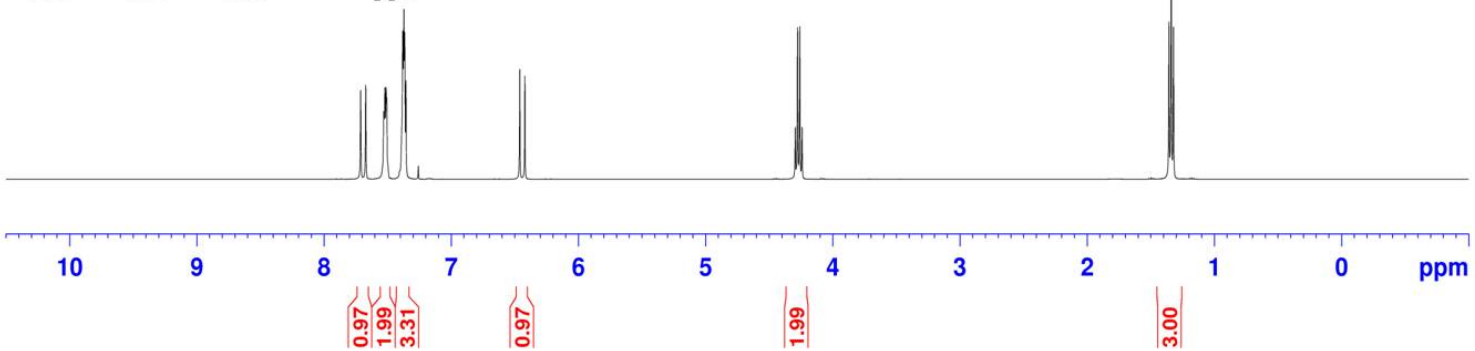
Ethyl Cinnamate
400 MHz, CDCl3



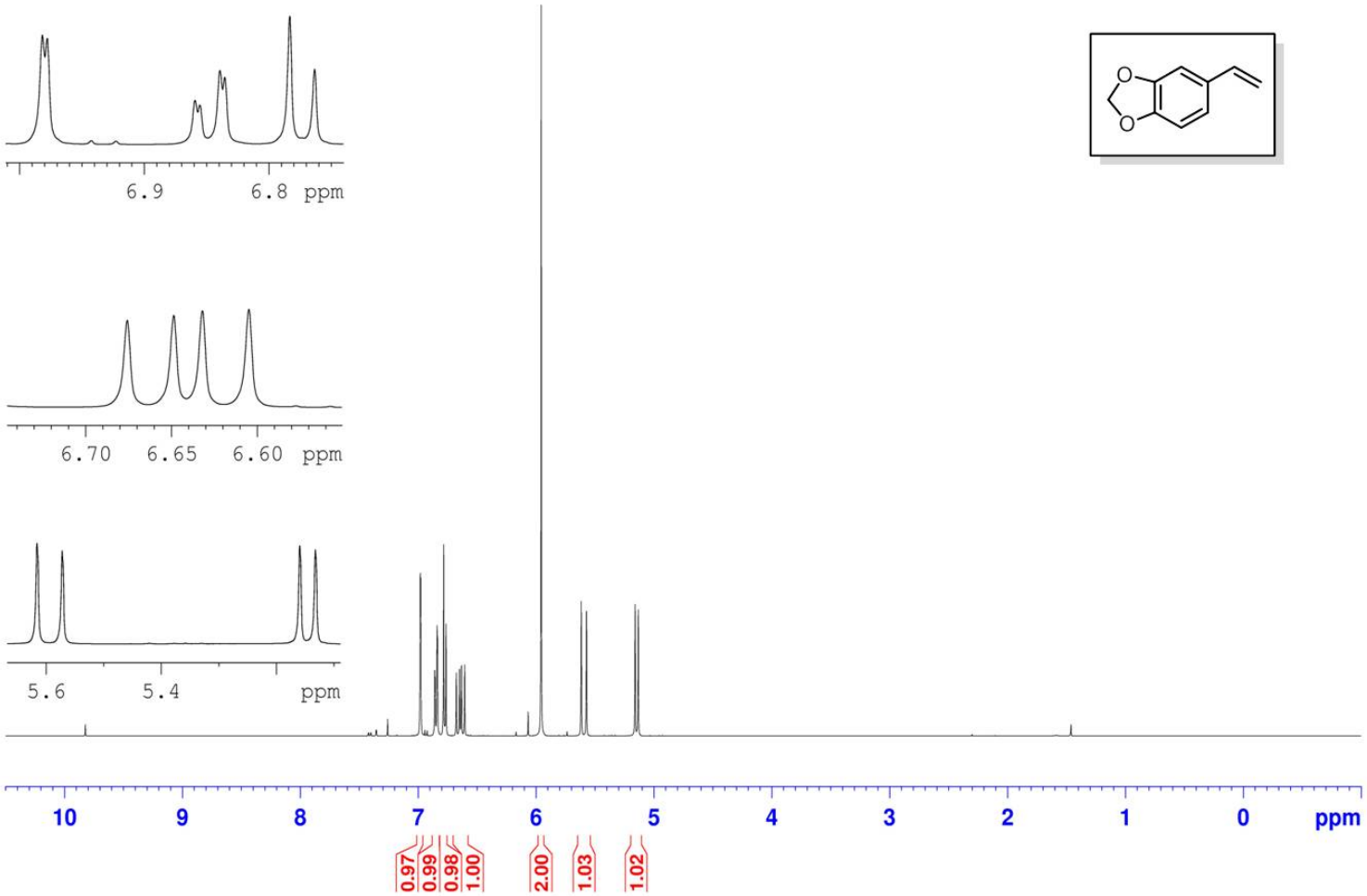




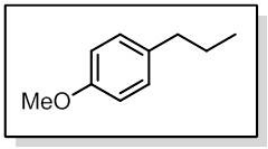


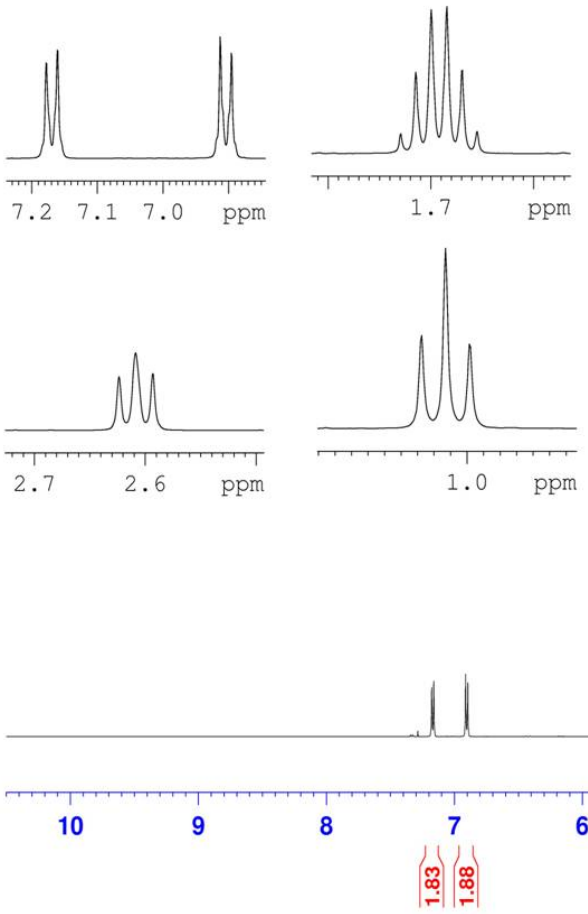


5−vinylbenzo[d][1,3]dioxole
400 MHz, CDCl3

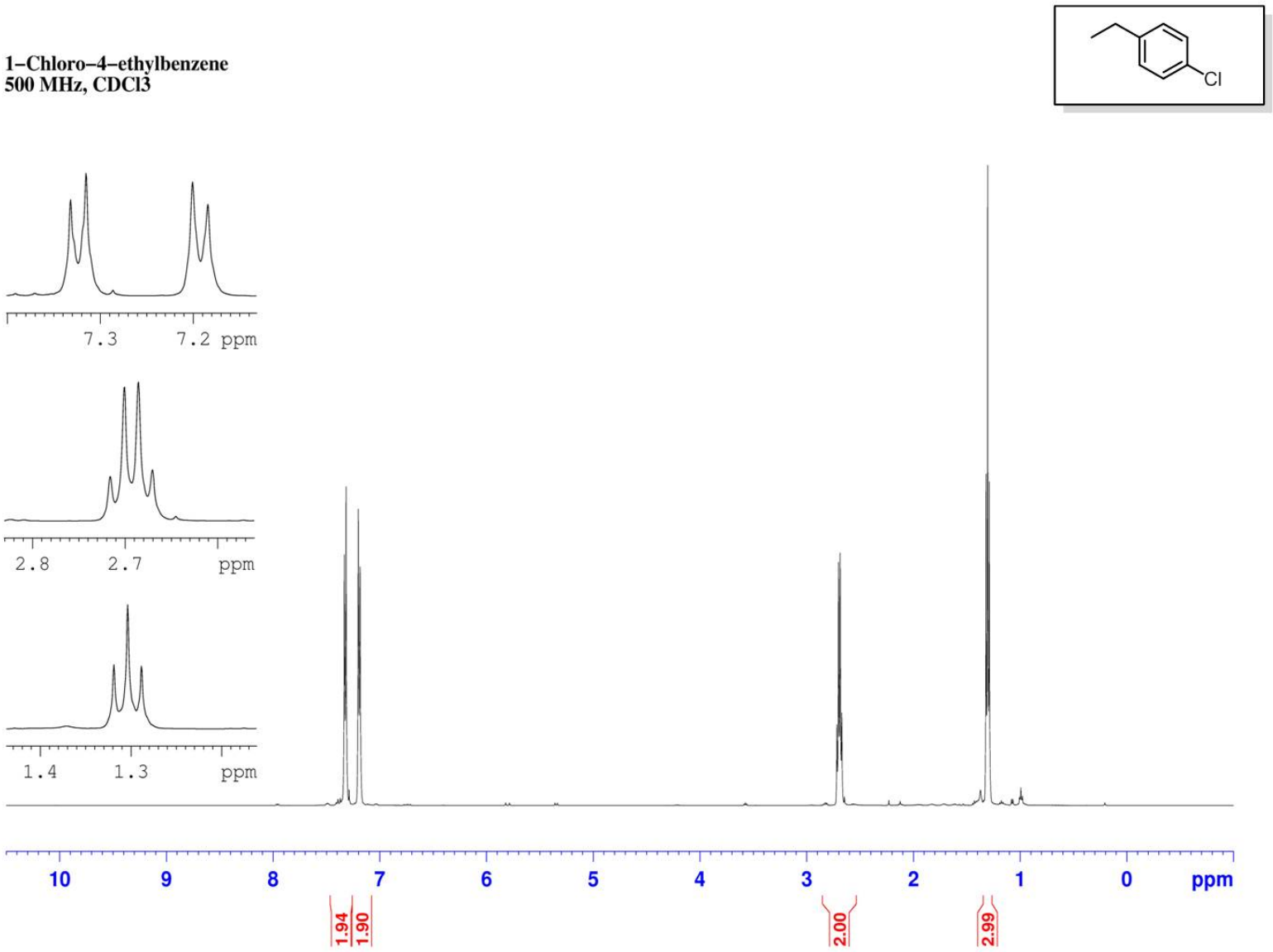


1–Methoxy–4–propylbenzene
500 MHz, CDCl3

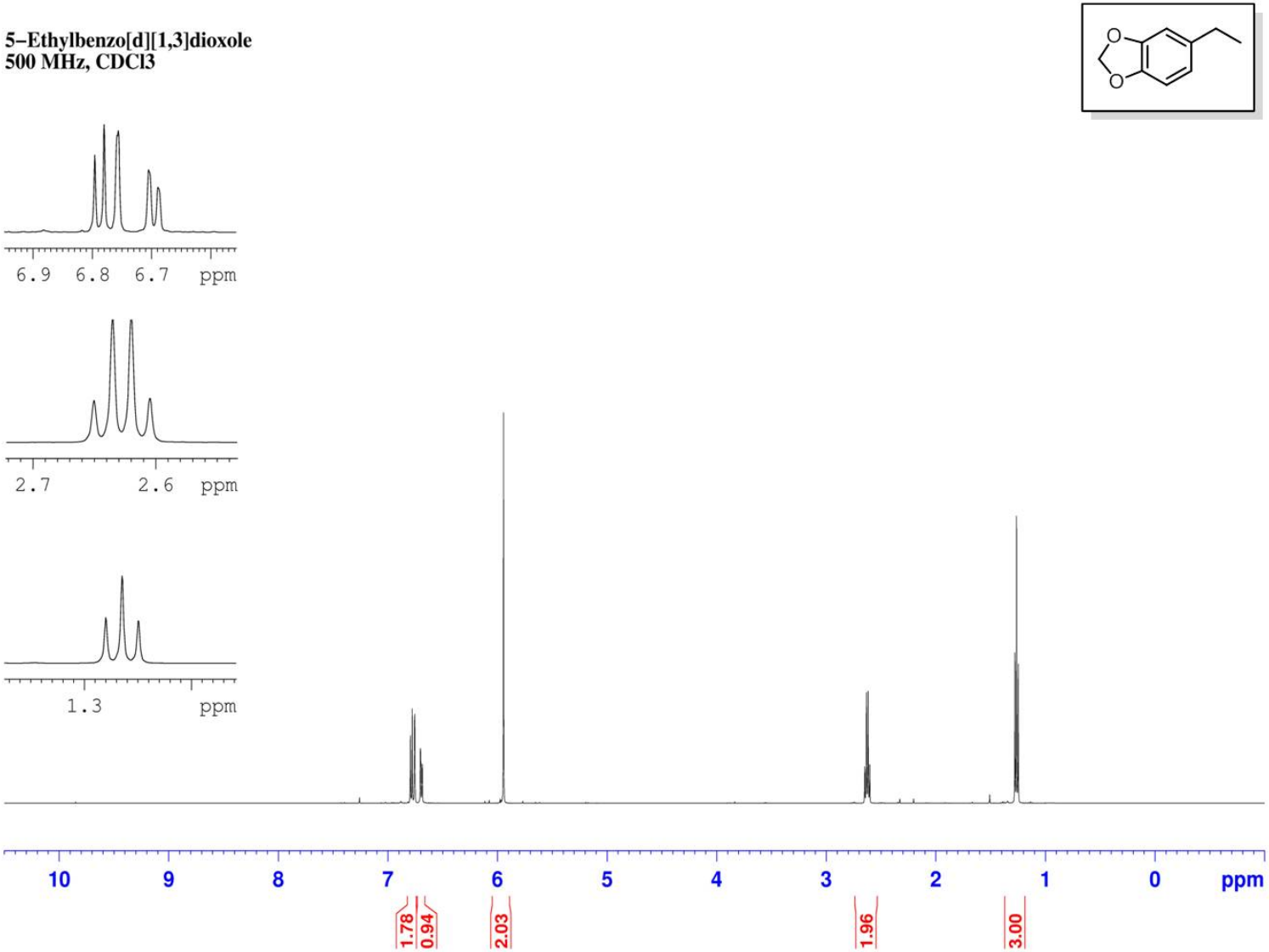




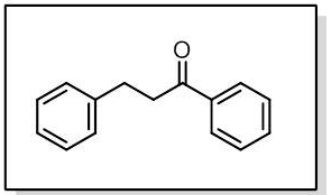
1–Chloro–4–ethylbenzene
500 MHz, CDCl3

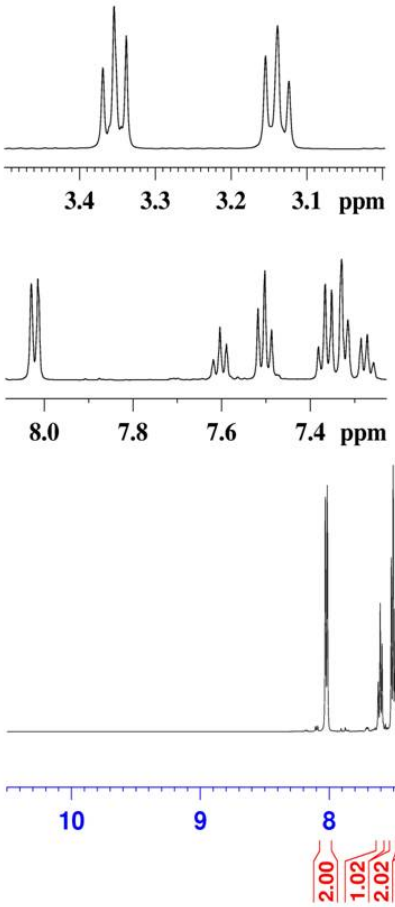


5–Ethylbenzo[d][1,3]dioxole
500 MHz, CDCl3

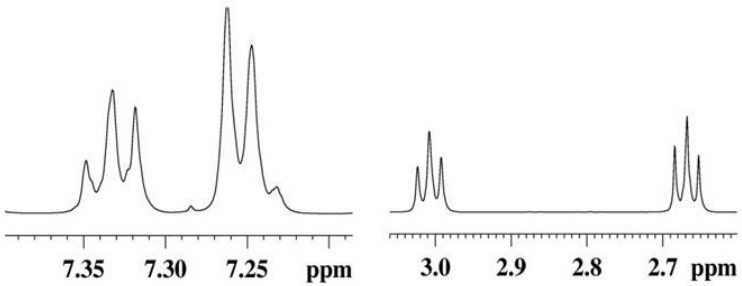


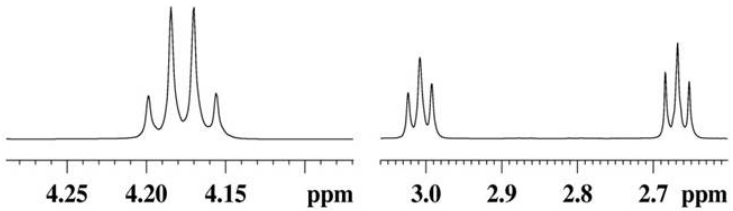
1,3–Diphenylpropan–1–one
500 MHz, CDCl3

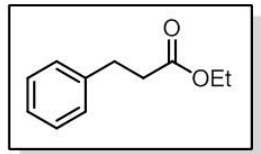


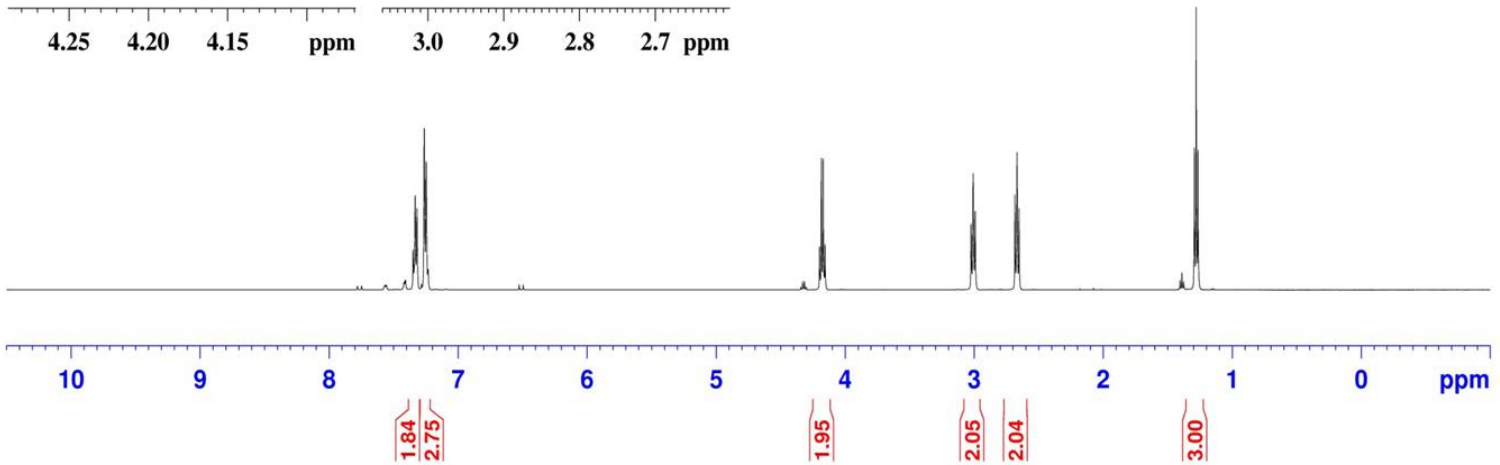


Ethyl 3–phenylpropanoate
500 MHz, CDCl3

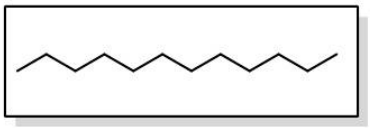


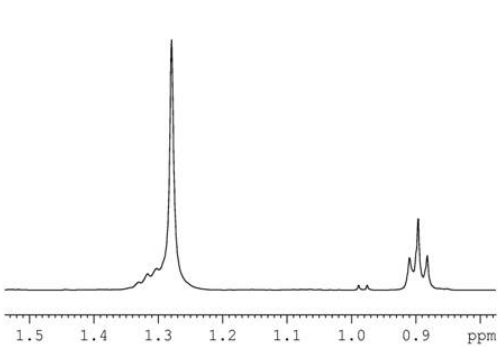




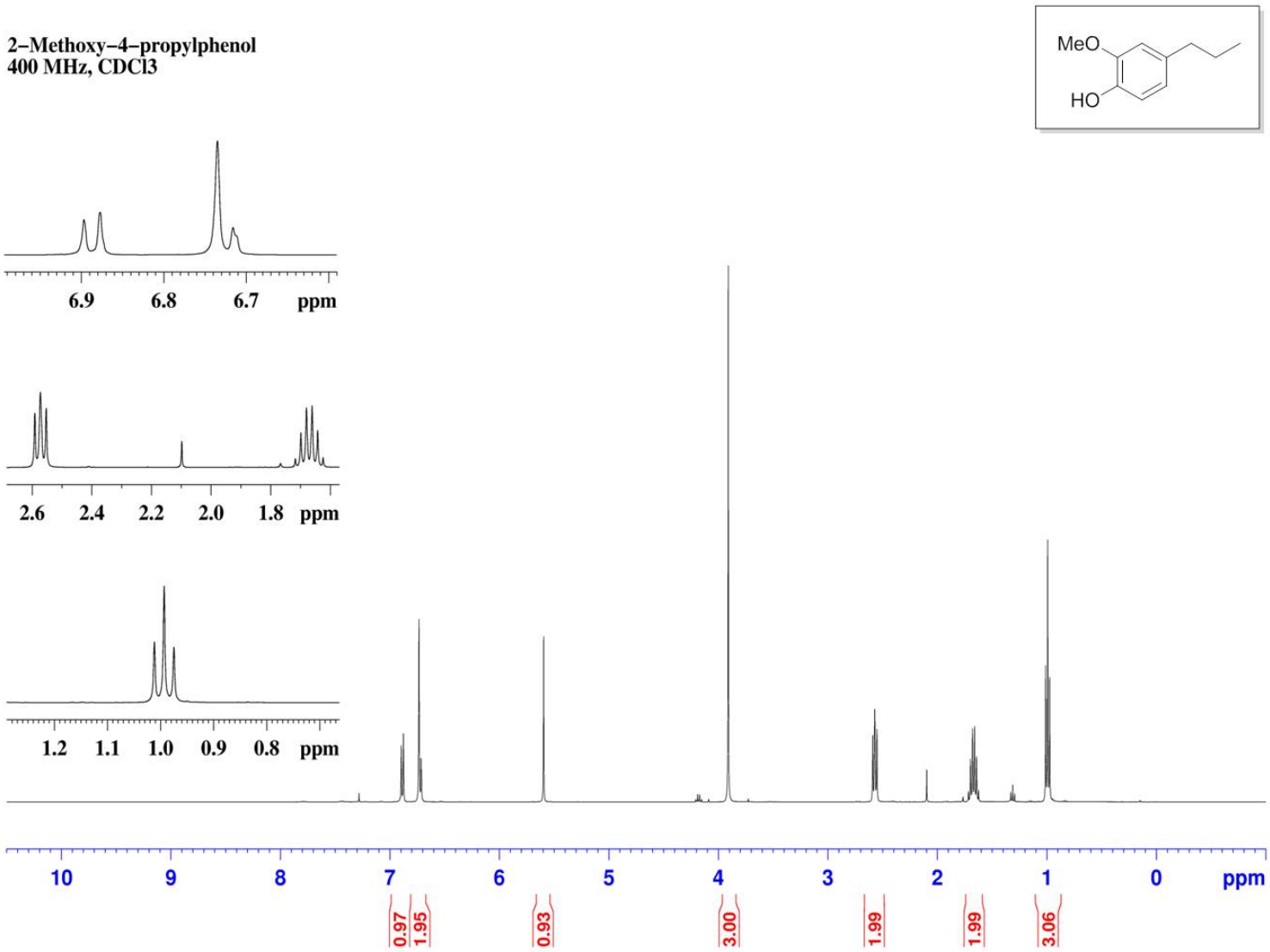


Dodecane
500 MHz, CDCl3

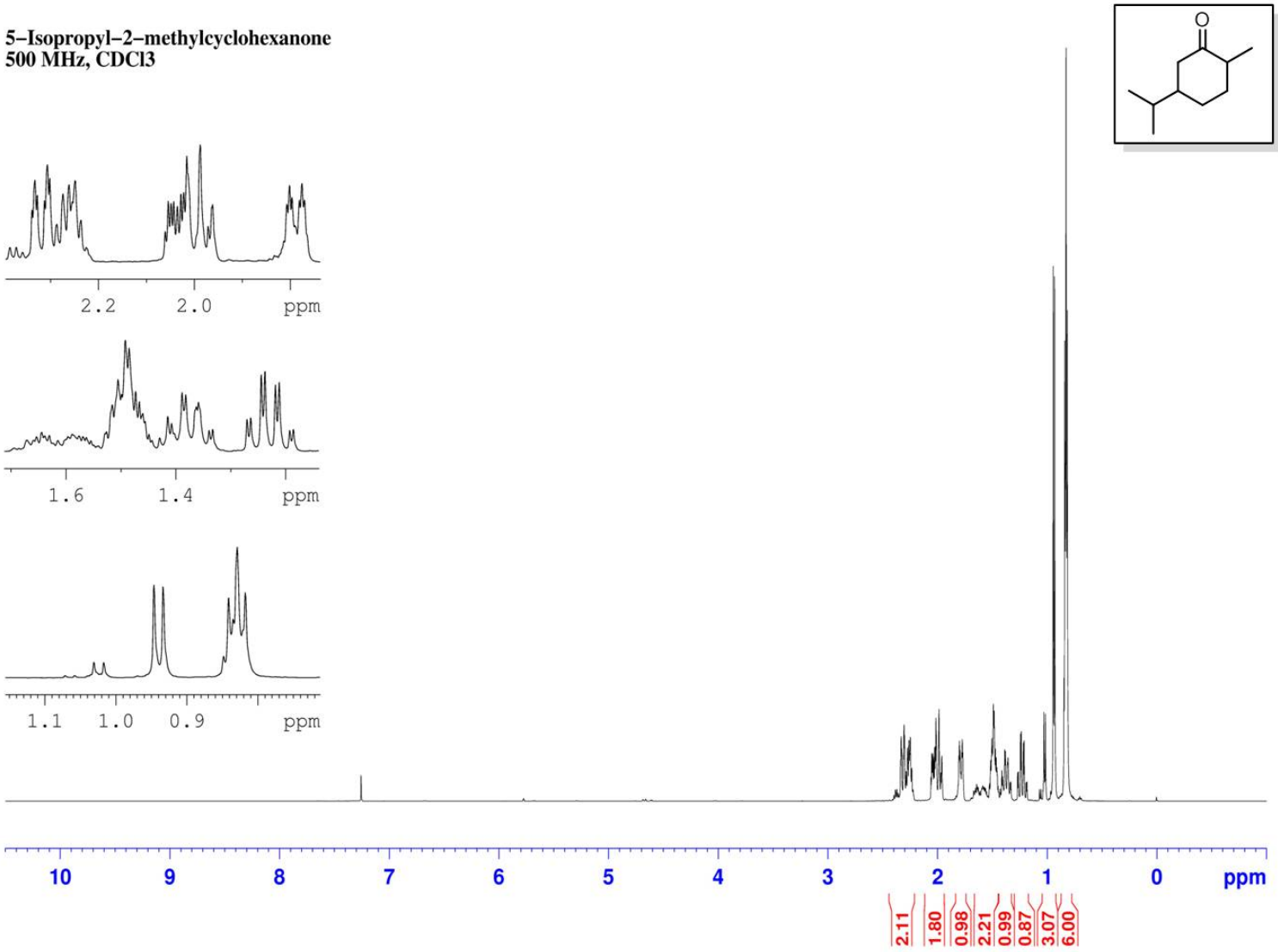




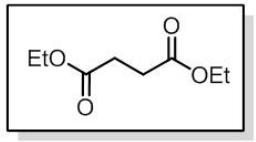
2–Methoxy–4–propylphenol
400 MHz, CDCl3

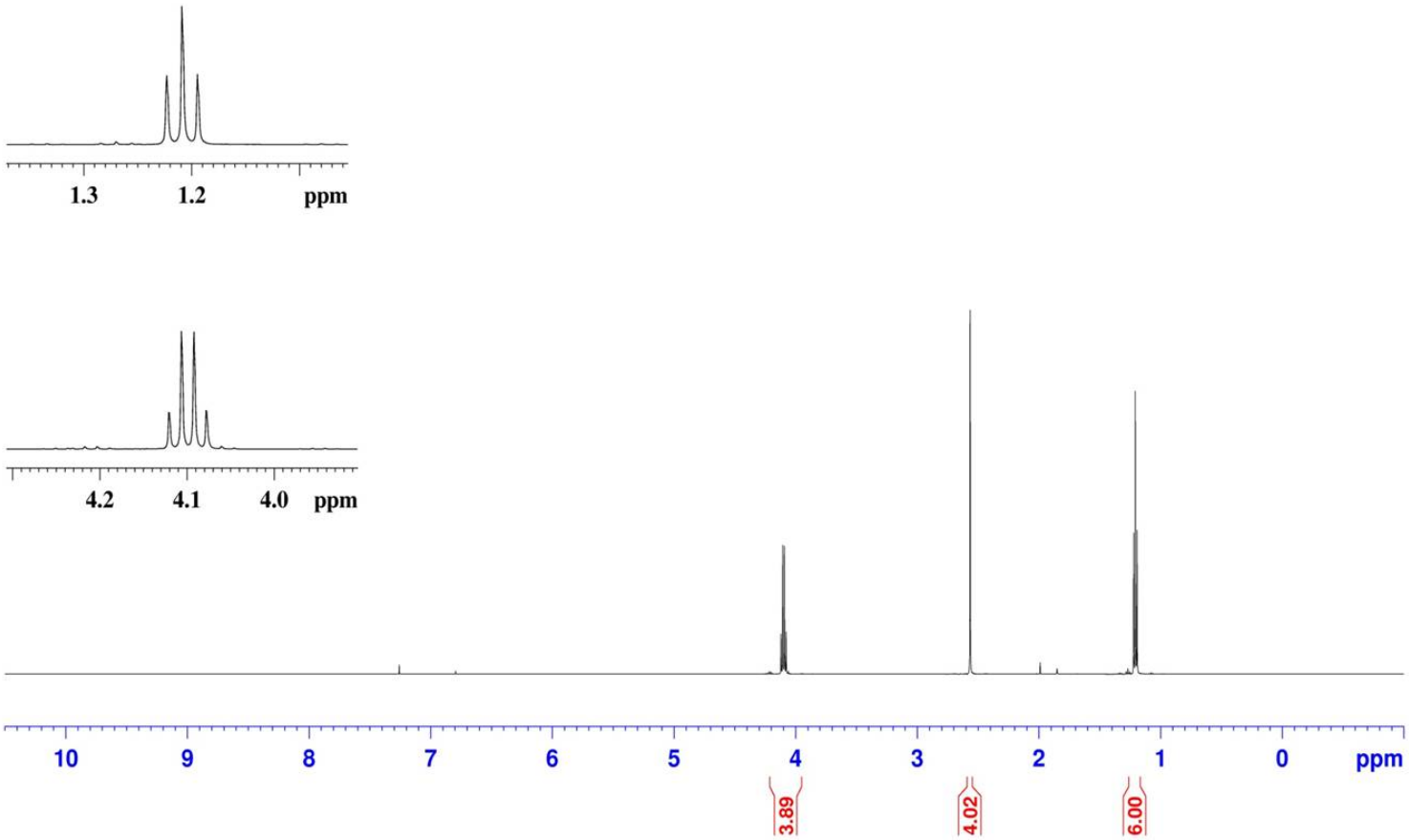


5−Isopropyl−2−methylcyclohexanone
500 MHz, CDCl3

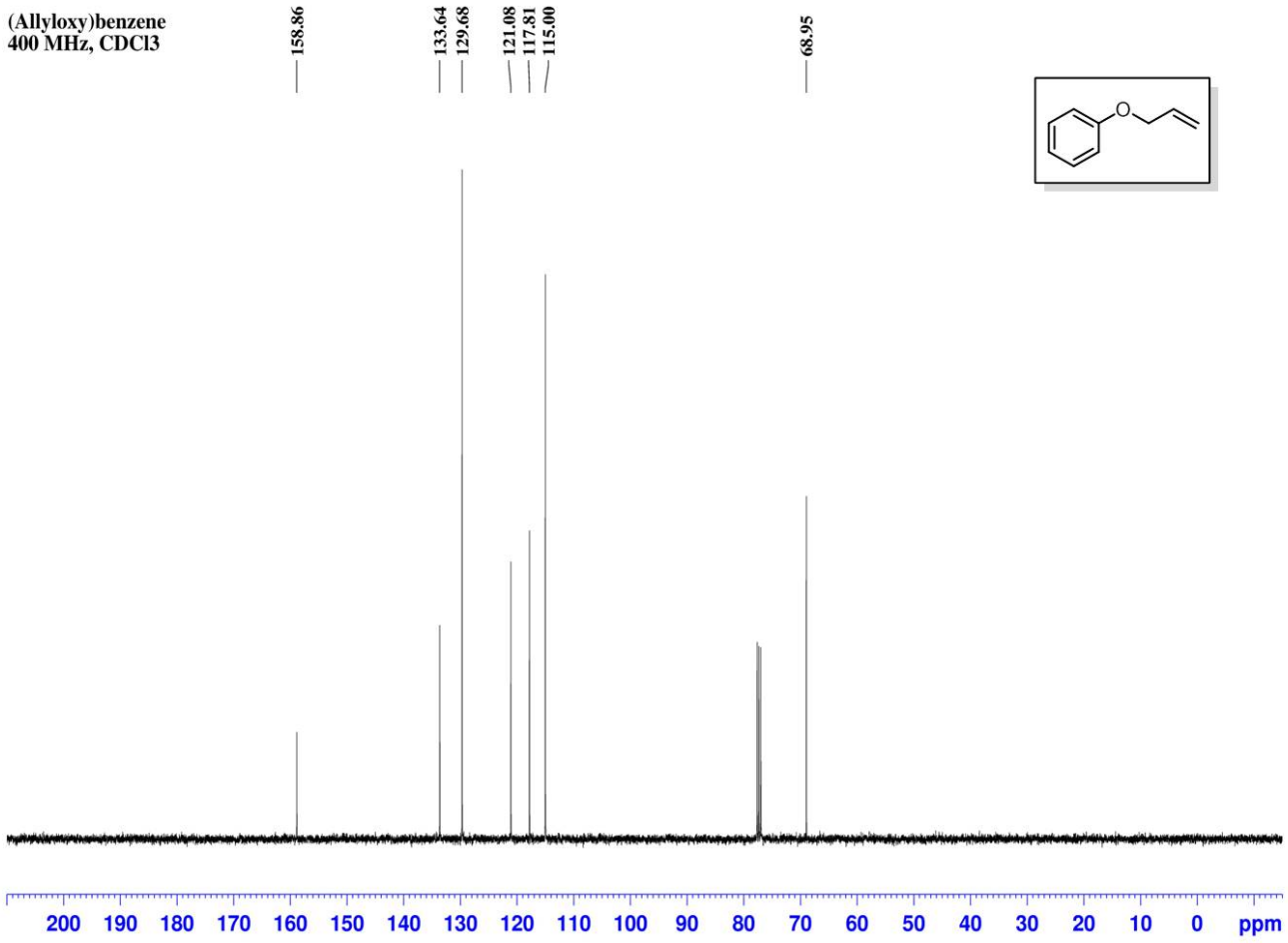


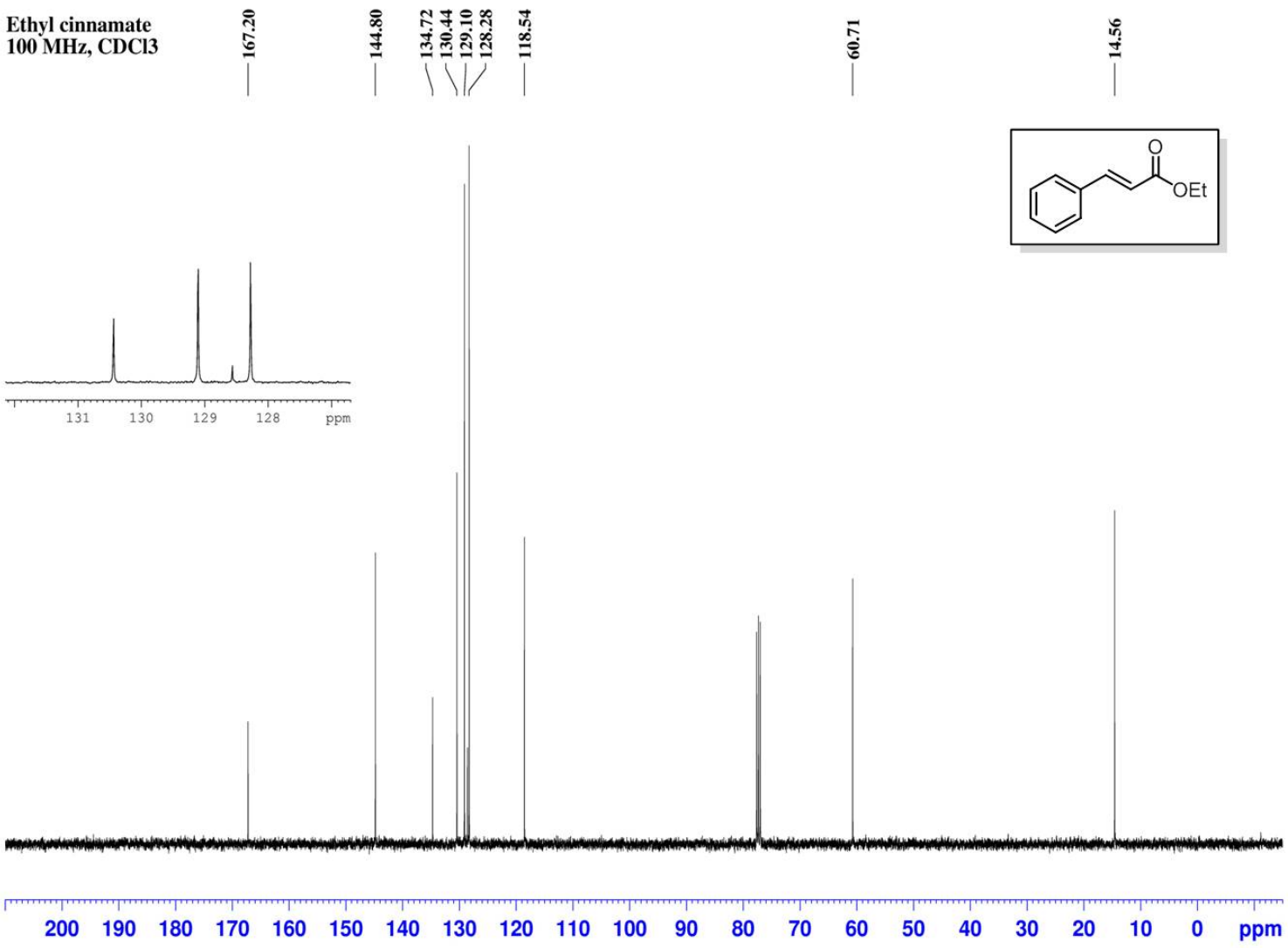
Diethyl Succinate
500 MHz, CDCl3



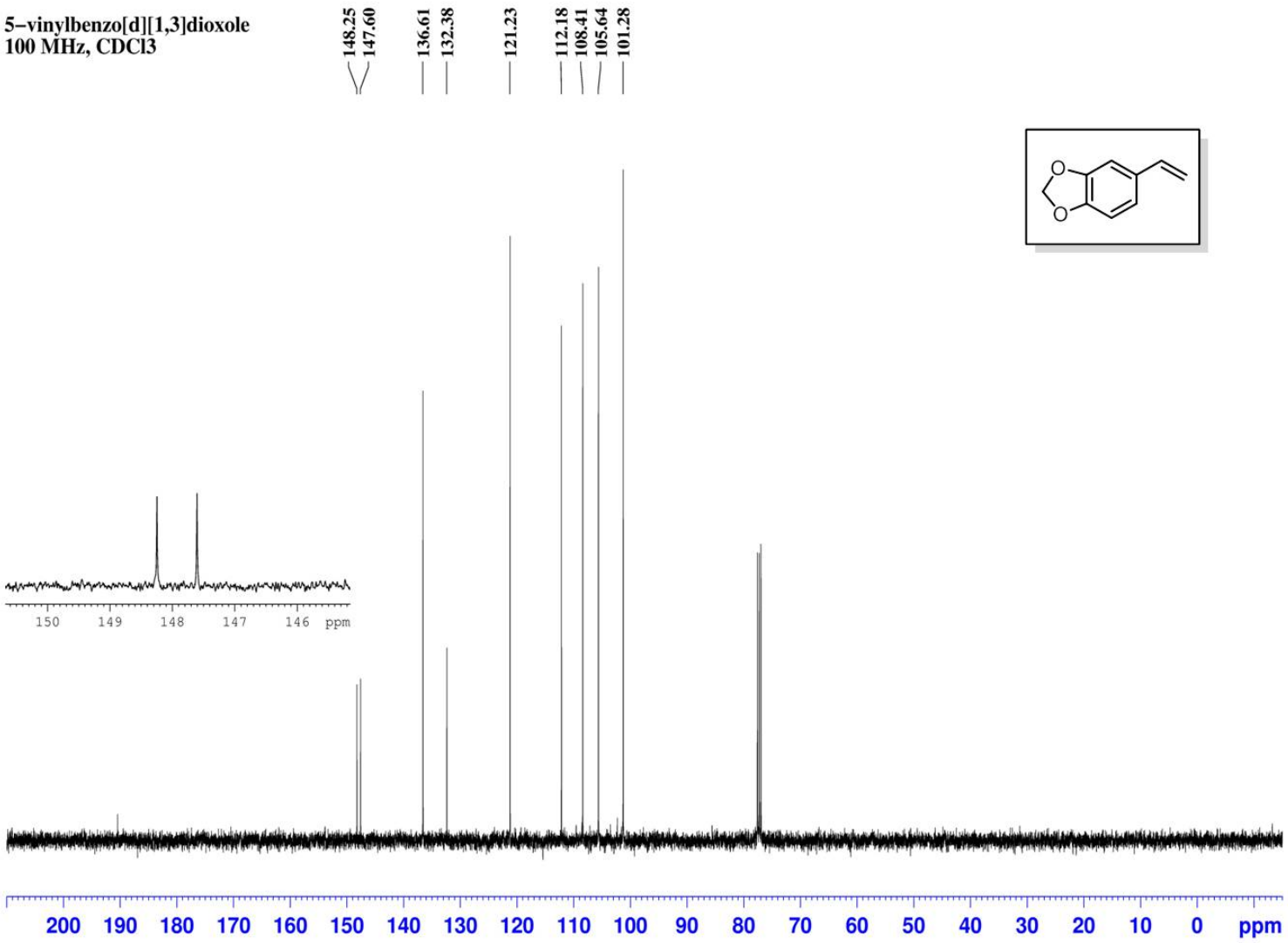


^{13}C-NMR spectra of synthesized compounds

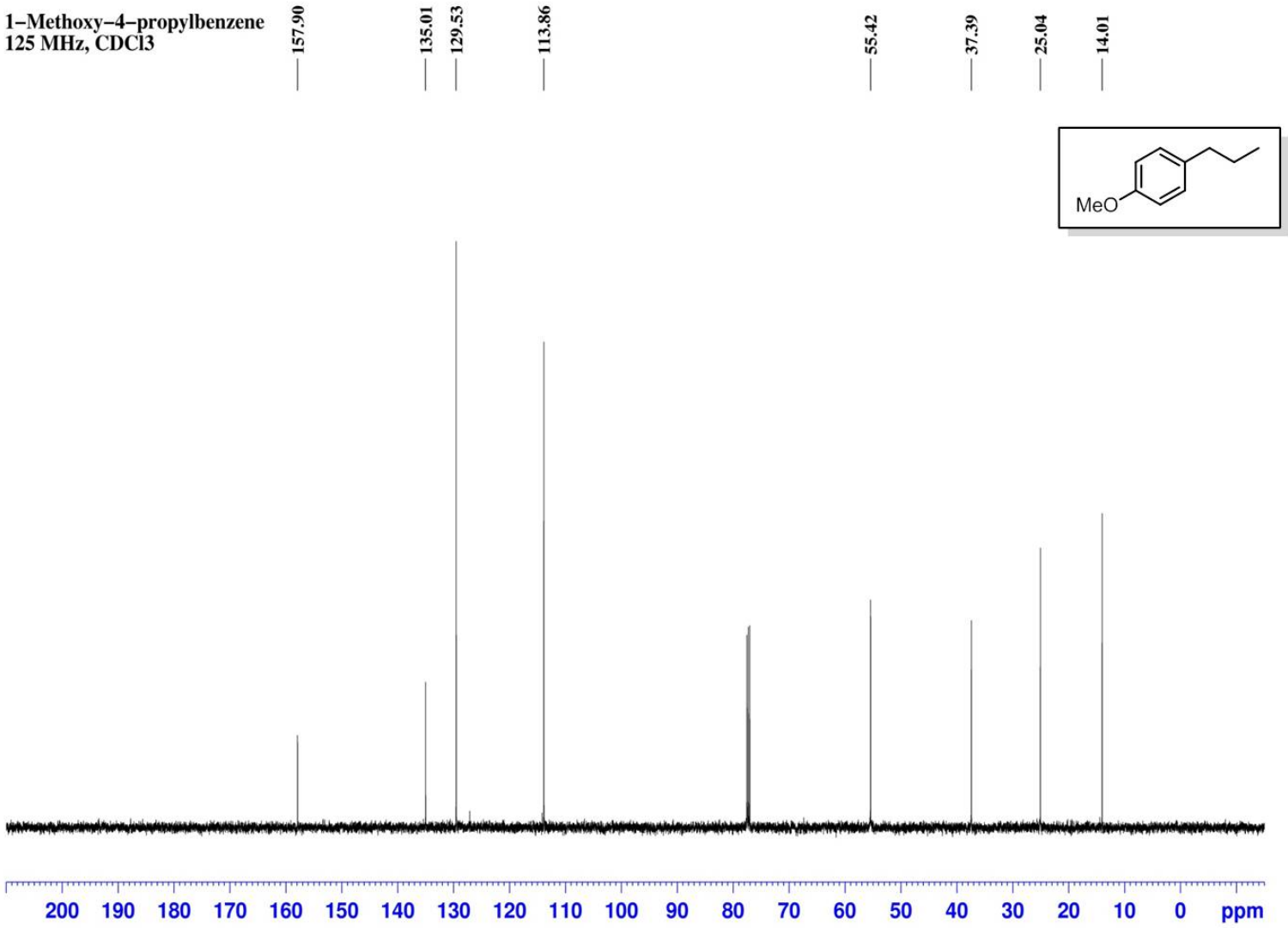



Ethyl cinnamate
100 MHz, CDCl3
167.20
144.80
134.72
130.44
129.10
128.28
118.54
60.71
14.56
O
OEt
131
130
129
128
ppm
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

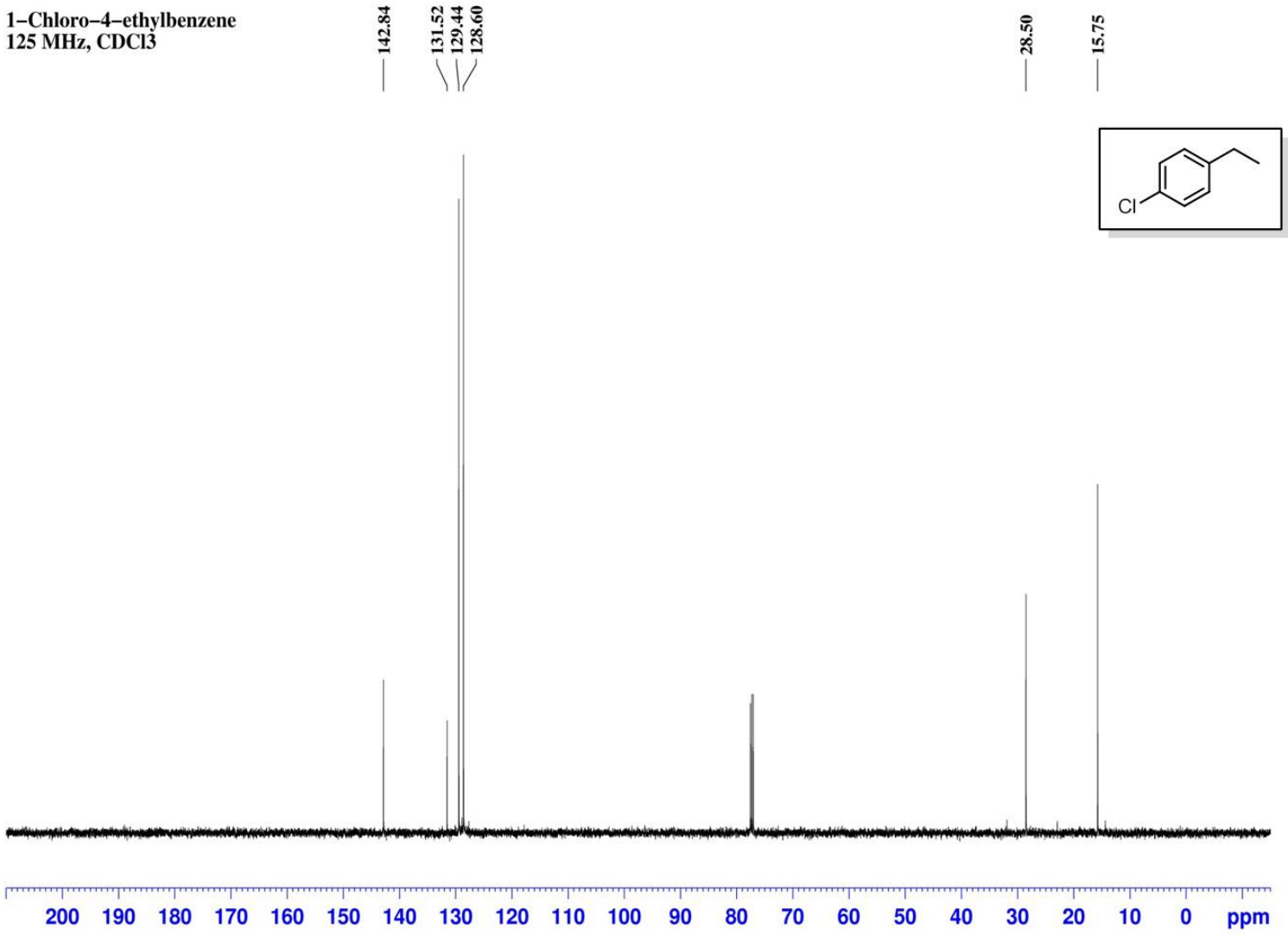
5–vinylbenzo[d][1,3]dioxole
100 MHz, CDCl3



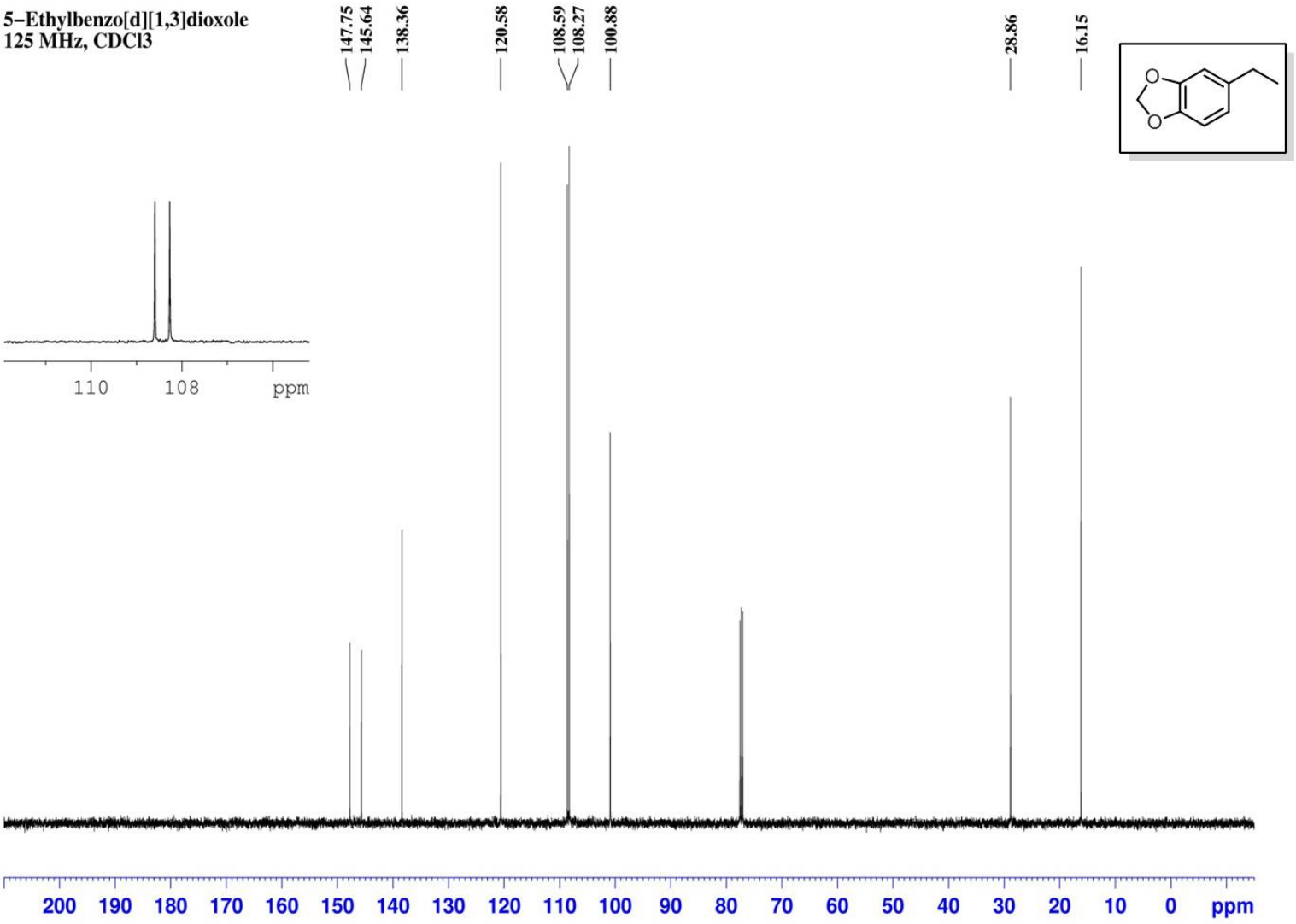
1–Methoxy–4–propylbenzene
125 MHz, CDCl3



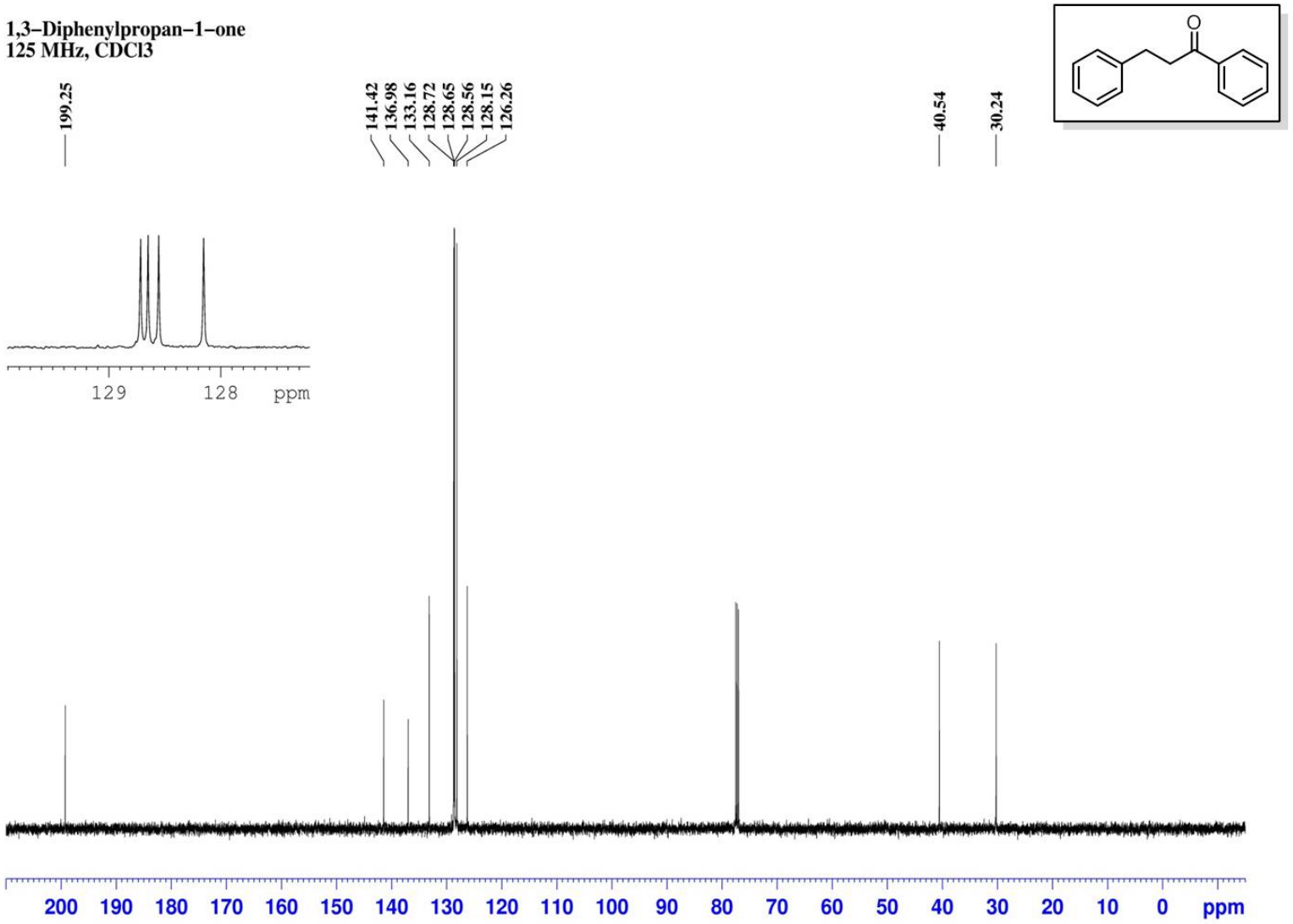
1–Chloro–4–ethylbenzene
125 MHz, CDCl3

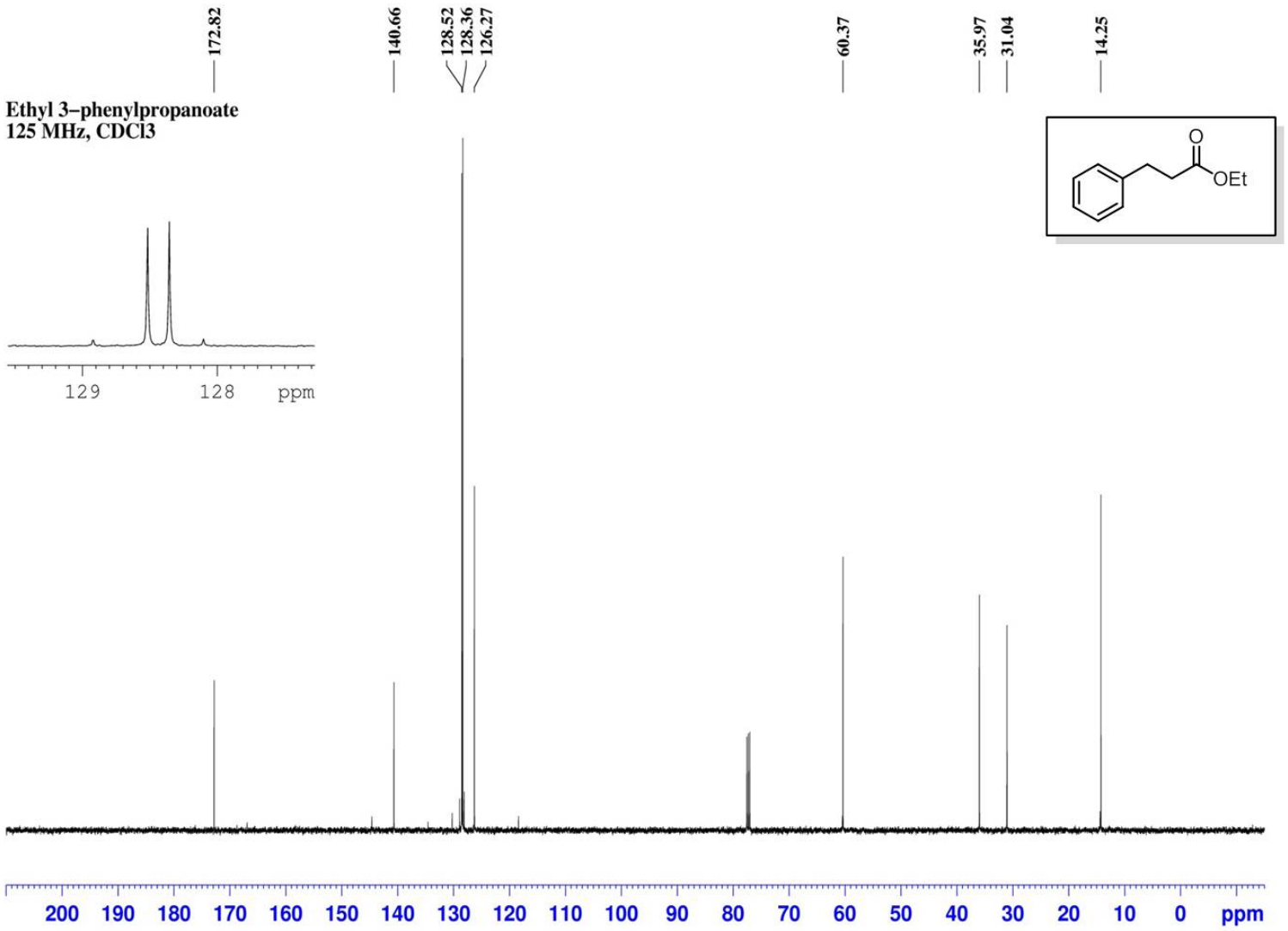


5–Ethylbenzo[d][1,3]dioxole
125 MHz, CDCl3

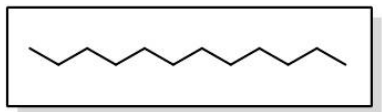


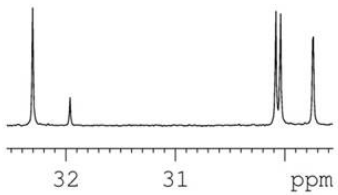
1,3–Diphenylpropan–1–one
125 MHz, CDCl3

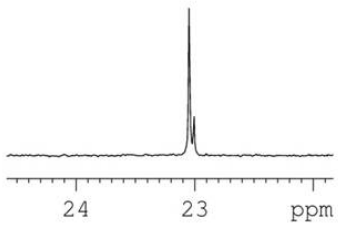



172.82
140.66
128.52
128.36
126.27
60.37
35.97
31.04
14.25
Ethyl 3–phenylpropanoate
125 MHz, CDCl3
O
OEt
129
128
ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

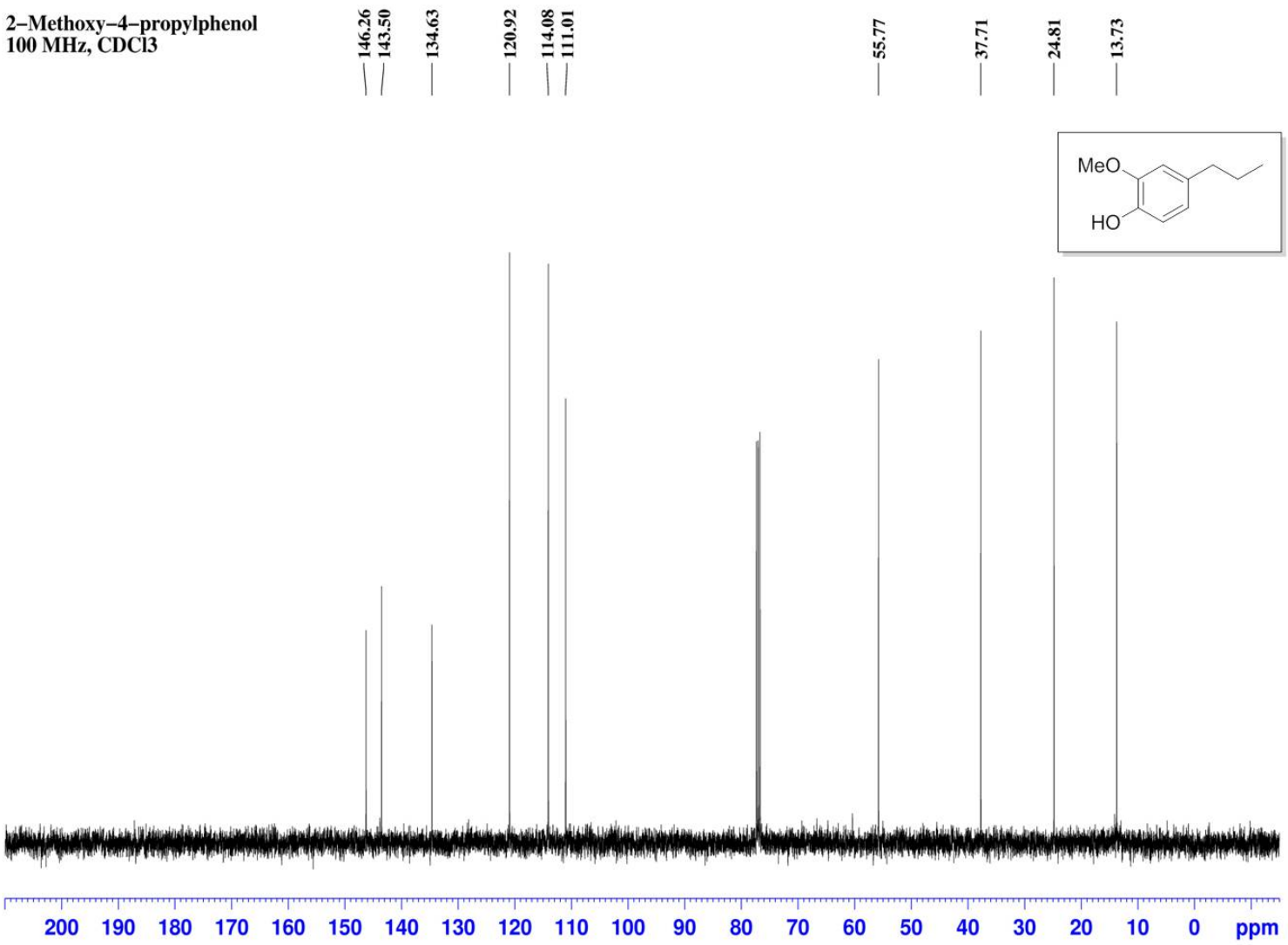
Dodecane
125 MHz, CDCl3



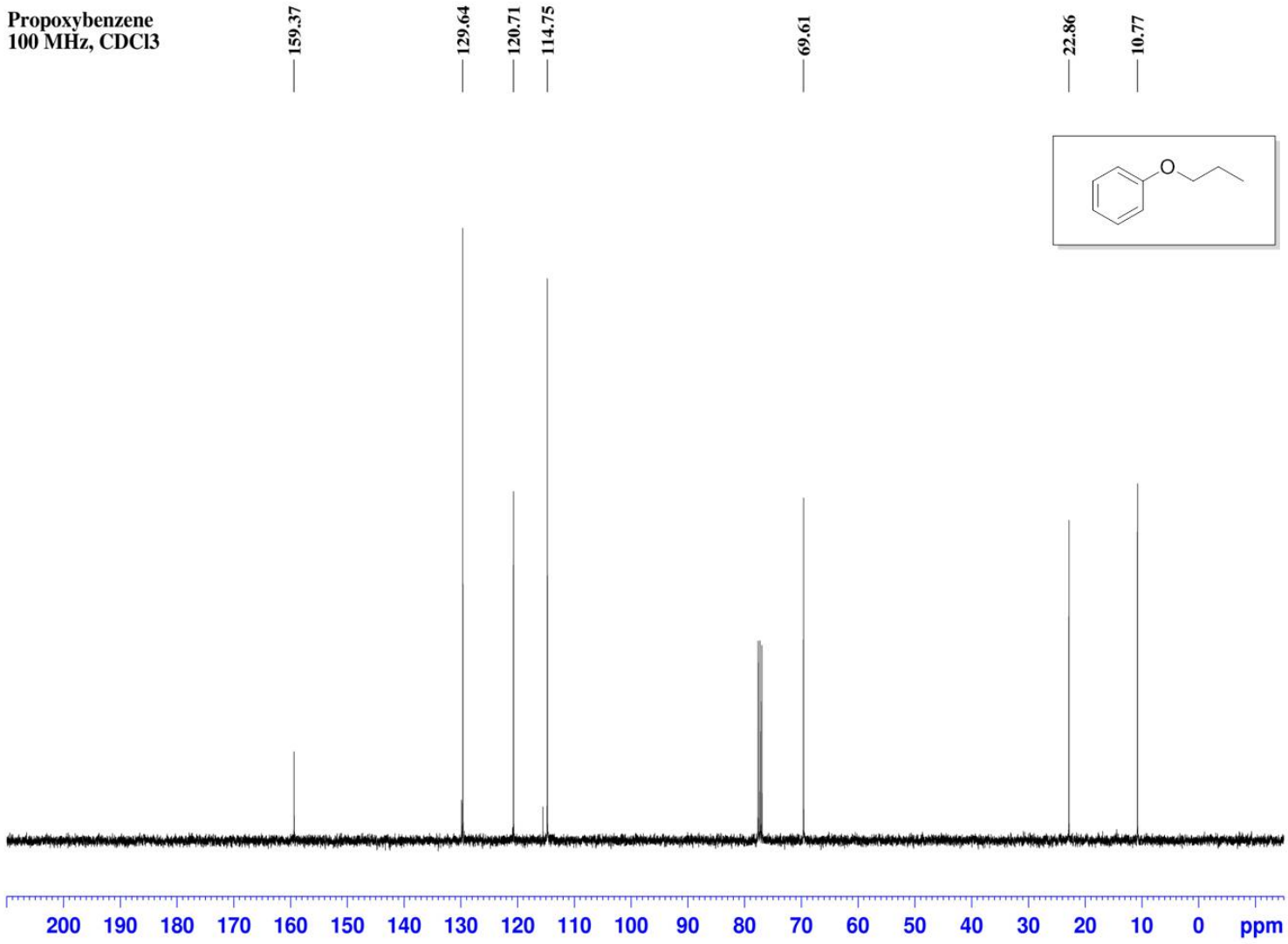




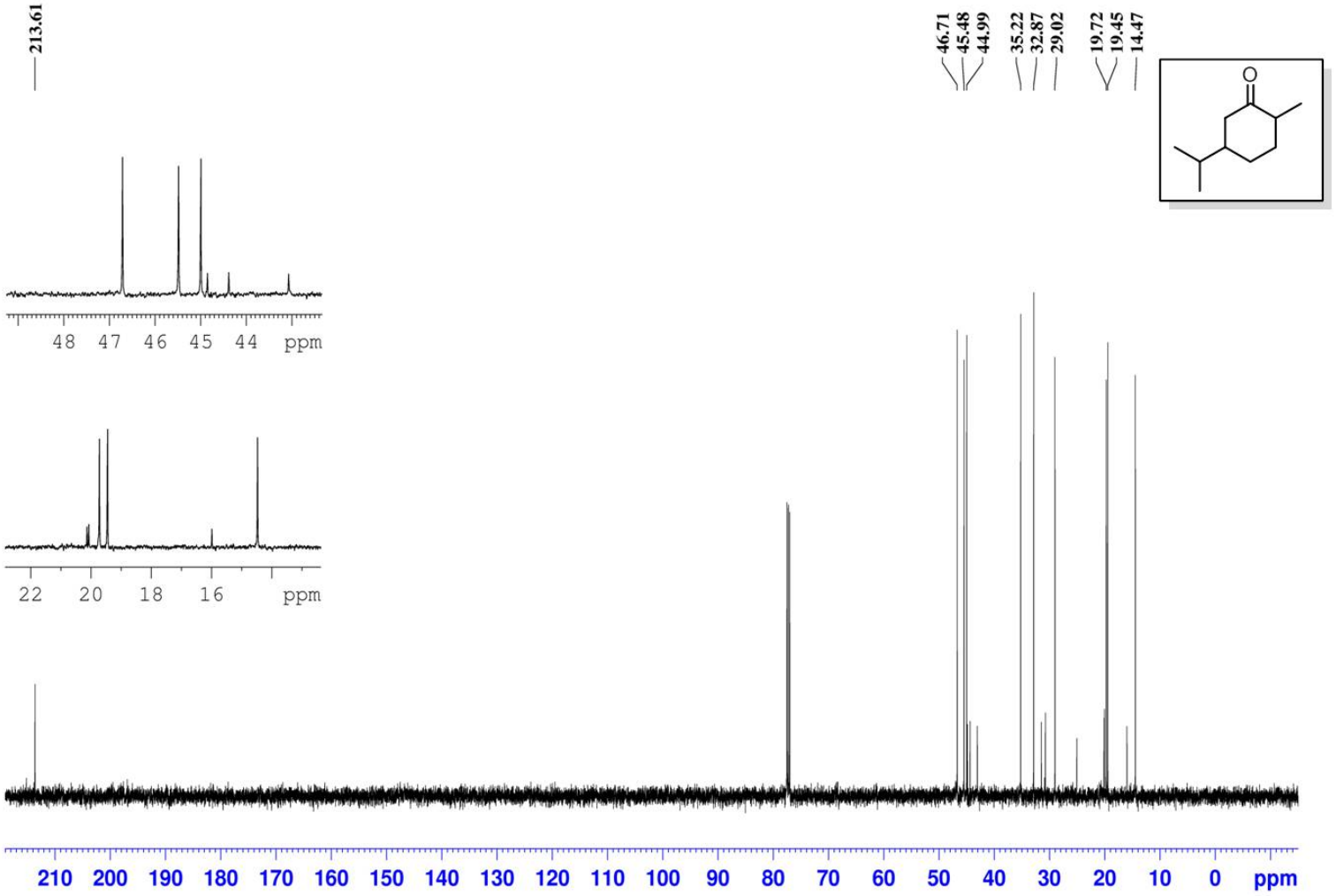
2–Methoxy–4–propylphenol
100 MHz, CDCl3



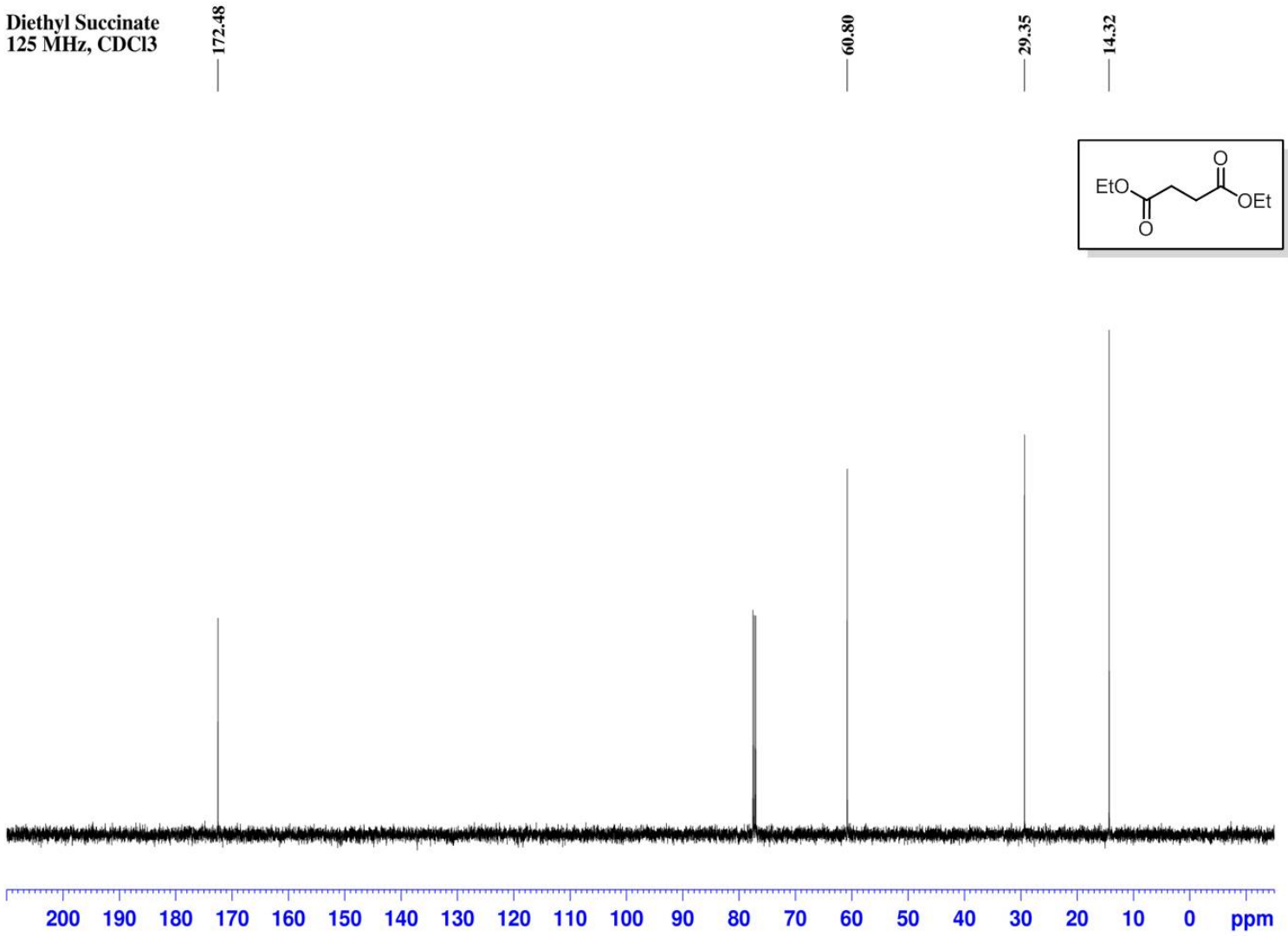
Propoxybenzene
100 MHz, CDCl3



5–Isopropyl–2–methylcyclohexanone
125 MHz, CDCl3



Diethyl Succinate
125 MHz, CDCl3



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