

Supporting Information
for

**Fe(III)-Catalyzed Hydroallylation of Unactivated Alkenes with
Morita-Baylis-Hillman Adducts**

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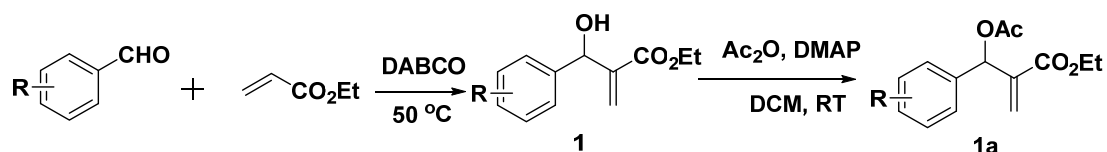
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1. General Information:

Infrared spectra were obtained on a FTIR spectrometer. ^1H NMR and ^{13}C NMR spectra were recorded on BRUKER AVANCE III 400 spectrometer. CDCl_3 were used as solvent. Chemical shifts were referenced relative to residual solvent. The following abbreviations are used to describe peak patterns where appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dq = double quartet. Coupling constants (J) are reported in Hertz (Hz). HRMS were performed on Waters GCT Premier Time of Flight Mass Spectrometer (EI) or Agilent Technologies 6224 TOF LC/MS apparatus (ESI). Melting points were measured with micro melting point apparatus.

$\text{Fe}(\text{acac})_3$, PhSiH_3 , EtOH, THF were commercially available, and the alkenes were commercially available or prepared easily. Morita-Baylis-Hillman adducts were prepared according the literature.¹

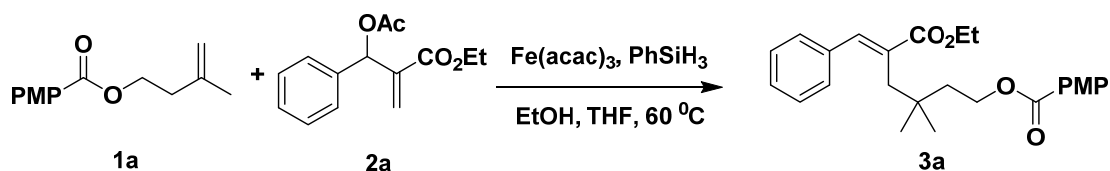
2. General Synthesis Procedure of Morita-Baylis-Hillman Adducts.



A mixture of methyl acrylate (30 mmol, 1.5 eq), aldehyde (15mmol, 1 eq) and DABCO (15mmol, 1 eq) was stirred at 50 °C for 24 hr, and then ether was added. The organic layer was separated and washed with dilute 6M HCl (30 mL), NaHCO₃ (aq) (30 mL) and water, and then dried over anhydrous Na₂SO₄. After removal of the volatile, the residue was purified by column chromatography to afford the compound **1** as oil.

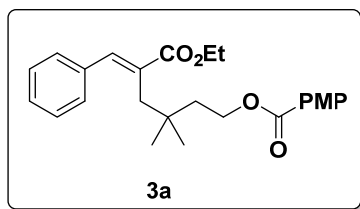
A mixture of **1** (10 mmol, 1 eq), Acetic anhydride (10 mmol, 1 eq), DMAP (1mmol, 0.1 eq) was stirred at room temperature for 1 hr. Then DCM was added. The organic layer was separated and washed with dilute aq NaHCO₃ (20 mL) and water, and then dried over anhydrous Na₂SO₄. After removal of the volatile, the residue was purified by column chromatography to afford the target product **1a**.

3. Typical Synthesis Procedure of **3a**.



A Schlenk tube containing Fe(acac)₃ (212 mg, 0.6 mmol) were evacuated and purged with Argon three times. Afterwards, alkenes **1a** (660 mg, 3 mmol), Morita-Baylis-Hillman adducts **2a** (496 mg, 2 mmol), PhSiH₃ (216 mg, 2 mmol) and EtOH (184 mg, 4 mmol), THF (10 mL) were added via syringe. The solution was kept at 60 °C until the compound **2a** was completed. Then the solution was evaporated under vacuum. The purification was performed by flash column chromatography using ethyl acetate/petroleum ether (v/v, 1:20) as eluent to give **3a** as a pale yellow liquid (656mg, 80% yield).

4. Characterization of 3, 4, 5.



(E)-5-(ethoxycarbonyl)-3,3-dimethyl-6-phenylhex-5-en-1-yl-4-methoxybenzoate

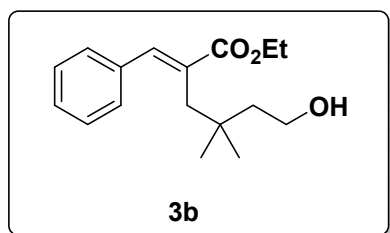
Pale yellow liquid (66 mg, 80% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, $J = 8.8$ Hz, 2H), 7.67 (s, 1H), 7.17-7.29 (m, 5H), 6.83 (d, $J = 9.2$ Hz, 2H), 4.20 (q, $J = 7.2$ Hz, 2H), 4.09 (t, $J = 7.2$ Hz, 2H), 3.78 (s, 3H), 2.68 (s, 2H), 1.50 (t, $J = 7.2$ Hz, 2H), 1.28 (t, $J = 7.2$ Hz, 3H), 0.73 (s, 6H),

^{13}C NMR (100 MHz, CDCl_3) δ 169.7, 166.4, 163.3, 140.5, 136.6, 132.4, 131.6, 128.9, 128.6, 128.0, 123.0, 113.7, 62.0, 61.1, 55.5, 40.6, 36.8, 35.4, 27.0, 14.4.

IR (Film) ν 2962, 2935, 1710, 1605, 1511, 1275, 1167, 1102, 848 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{25}\text{H}_{30}\text{O}_5$ (M^+): 410.2093; Found: 410.2095.



Ethyl (E)-2-benzylidene-6-hydroxy-4,4-dimethylhexanoate

Pale yellow liquid; (41mg, 75% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:5).

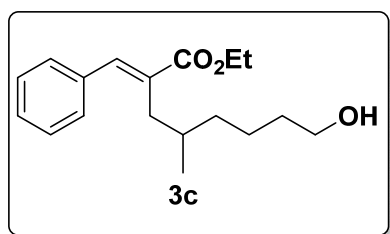
^1H NMR (400 MHz, CDCl_3) δ 7.71 (s, 1H), 7.37-7.56 (m, 5H), 4.27 (q, $J = 7.2$ Hz, 2H), 3.62 (t, $J = 7.2$ Hz, 2H), 2.69 (s, 2H), 1.41 (m, 2H), 1.35 (t, $J = 7.2$ Hz, 3H), 0.74 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 140.3, 136.8, 132.6, 129.0, 128.6, 128.0,

61.1, 59.7, 45.0, 36.7, 35.3, 27.3, 14.4.

IR (Film) ν 3412, 2959, 2933, 1710, 1466, 1367, 1245, 1133, 1078, 858 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{17}\text{H}_{24}\text{O}_3$ (M^+): 276.1725; Found: 276.1720.



Ethyl (*E*)-2-benzylidene-8-hydroxy-4-methyloctanoate

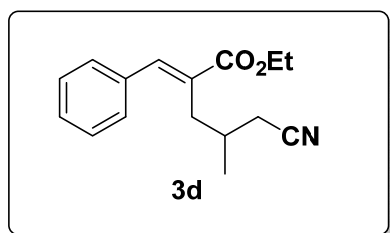
Pale yellow liquid; (44mg, 75% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:5).

^1H NMR (400 MHz, CDCl_3) δ 7.69 (s, 1H), 7.30-7.38 (m, 5H), 4.27 (q, J = 7.2 Hz, 2H), 3.59 (t, J = 6.4 Hz, 2H), 2.59 (dd, J_1 = 13.4 Hz, J_2 = 6.4 Hz, 1H), 2.42 (dd, J_1 = 13.4 Hz, J_2 = 8.2 Hz, 1H), 1.26-1.53 (m, 10H), 0.81 (d, J = 6.8 Hz, 3H),

^{13}C NMR (100 MHz, CDCl_3) δ 169.0, 139.5, 136.2, 133.5, 129.3, 128.5, 128.2, 63.1, 60.9, 36.7, 34.1, 33.1, 32.9, 23.1, 19.6, 14.4.

IR (Film) ν 3400, 2926, 2858, 1708, 1624, 1508, 1249, 1223, 1197, 1132, 829 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{18}\text{H}_{26}\text{O}_3$ (M^+): 290.1882; Found: 290.1880.



Ethyl (*E*)-2-benzylidene-5-cyano-4-methylpentanoate

Pale yellow liquid; (26mg, 51% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

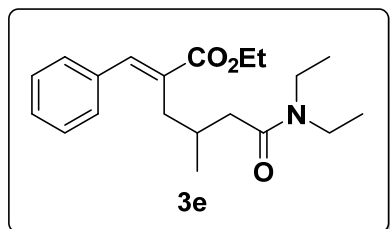
^1H NMR (400 MHz, CDCl_3) δ 7.82 (s, 1H), 7.33-7.43 (m, 5H), 4.29 (q, J = 7.2 Hz, 2H), 2.59-2.70 (m, 2H), 2.25-2.31 (m, 1H), 2.09-2.18 (m, 2H), 1.36 (t, J = 7.2 Hz, 3H), 1.02 (d, J = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 168.2, 141.4, 135.5, 131.0, 129.1, 128.84,

128.76, 118.7, 61.3, 33.1, 30.7, 19.6, 14.4.

IR (Film) ν 3385, 2964, 2926, 2244, 1705, 1629, 1447, 1368, 1260, 1130, 1107, 768 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{16}\text{H}_{19}\text{NO}_2$ (M^+): 257.1416; Found: 257.1413.



Ethyl (*E*)-2-benzylidene-6-(diethylamino)-4-methyl-6-oxohexanoate

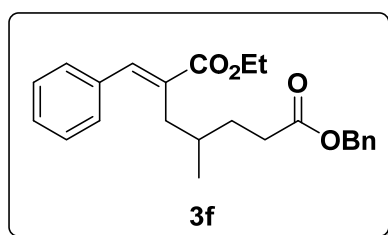
Pale yellow liquid; (52mg, 79% yield), R_f = 0.3 (EtOAc/Petroleum ether 1:10).

^1H NMR (400 MHz, CDCl_3) δ 7.71 (s, 1H), 7.30-7.38 (m, 5H), 4.26 (q, J = 7.2 Hz, 2H), 3.30-3.39 (m, 2H), 3.25 (q, J = 7.2 Hz, 2H), 2.44-2.62 (m, 2H), 2.22-2.27 (m, 2H), 1.68-1.83 (m, 1H), 1.35 (t, J = 7.2 Hz, 3H), 1.14 (t, J = 7.2 Hz, 3H), 1.08 (t, J = 7.2 Hz, 3H), 0.83 (d, J = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 172.3, 168.8, 139.8, 136.1, 133.1, 129.3, 128.5, 128.2, 60.9, 42.0, 40.1, 33.9, 33.0, 32.6, 30.9, 19.5, 14.5, 14.4, 13.2.

IR (Film) ν 3372, 2920, 2850, 1711, 1629, 1451, 1365, 1201, 1096 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{20}\text{H}_{29}\text{NO}_3$ (M^+): 331.2147; Found: 331.2149.



7-benzyl 1-ethyl (*E*)-2-benzylidene-4-methylheptanedioate

Pale yellow liquid; (58mg, 76% yield), R_f = 0.6 (EtOAc/Petroleum ether 1:20).

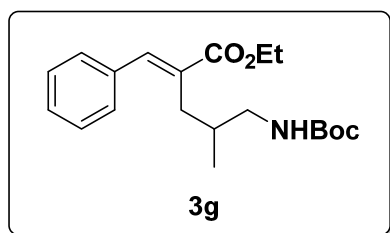
^1H NMR (400 MHz, CDCl_3) δ 7.63 (s, 1H), 7.19-7.29 (m, 10H), 4.99 (s, 2H), 4.16 (q, J = 7.2 Hz, 2H), 2.34-2.50 (m, 2H), 2.21-2.28 (m, 2H), 1.57-1.70 (m, 2H),

1.32-1.41(m, 1H), 1.24 (t, $J = 7.2$ Hz, 3H), 0.72 (d, $J = 6.8$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 173.7, 168.7, 139.9, 136.2, 136.1, 132.8, 129.2, 128.6, 128.3, 128.26, 66.2, 61.0, 33.6, 32.7, 32.1, 31.9, 19.2, 14.4.

IR (Film) ν 2958, 2926, 2874, 1736, 1454, 1379, 1229, 1163, 1108, 749 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{24}\text{H}_{28}\text{O}_4$ (M^+): 380.1988; Found: 380.1992.



Ethyl (*E*)-2-benzylidene-5-((*tert*-butoxycarbonyl) amino)-4-methylpentanoate

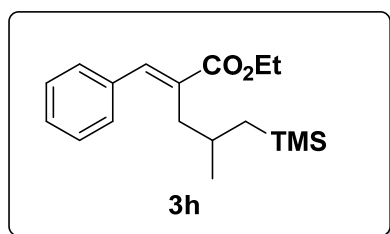
Pale yellow liquid; (32mg, 46% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:10).

^1H NMR (400 MHz, CDCl_3) δ 7.74 (s, 1H), 7.34-7.39 (m, 5H), 4.65 (brs, 1H), 4.27 (q, $J = 7.2$ Hz, 2H), 2.87-3.08 (m, 2H), 2.43-2.64 (m, 2H), 1.82-1.90 (m, 1H), 1.43 (s, 9H), 1.36 (t, $J = 7.2$ Hz, 3H), 0.84 (d, $J = 6.8$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 168.8, 156.3, 140.3, 135.9, 132.3, 129.3, 128.7, 128.5, 61.1, 45.8, 31.2, 29.8, 28.6, 17.8, 14.4.

IR (Film) ν 3383, 2975, 2928, 1708, 1511, 1453, 1356, 1251, 1170, 859 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{20}\text{H}_{29}\text{NO}_4$ (M^+): 347.2097; Found: 347.2094.



Ethyl (*E*)-2-benzylidene-4-methyl-5-(trimethylsilyl)pentanoate

Pale yellow liquid; (30mg, 50% yield), $R_f = 0.5$ (Petroleum ether).

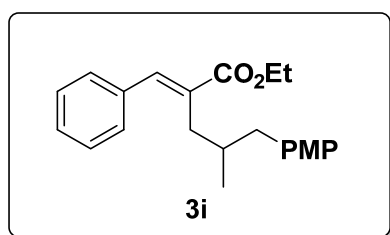
^1H NMR (400 MHz, CDCl_3) δ 7.69 (s, 1H), 7.29-7.38 (m, 5H), 4.27 (q, $J = 7.2$

Hz, 2H), 2.46-2.56 (m, 2H), 1.85-1.92 (m, 1H), 1.35 (t, $J = 7.2$ Hz, 3H), 0.84 (d, $J = 6.8$ Hz, 3H), 0.62 (dd, $J_1 = 14.4$ Hz, $J_2 = 4.4$ Hz, 1H), 0.39 (dd, $J_1 = 14.8$ Hz, $J_2 = 9.2$ Hz, 1H), -0.03 (s, 9H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.1, 139.5, 136.3, 133.6, 129.3, 128.5, 128.2, 60.9, 37.8, 29.7, 25.5, 22.7, 14.5, -0.56.

IR (Film) ν 3059, 2954, 1710, 1631, 1454, 1230, 1019, 927, 841 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{18}\text{H}_{28}\text{O}_2\text{Si}$ (M^+): 304.1859; Found: 304.1858.



Ethyl (*E*)-2-benzylidene-5-(4-methoxyphenyl)-4-methylpentanoate

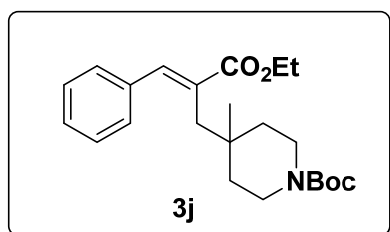
Pale yellow liquid; (39mg, 57% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.68 (s, 1H), 7.28-7.33 (m, 5H), 7.01 (d, $J = 8.4$ Hz, 2H), 6.80 (d, $J = 8.4$ Hz, 2H), 4.26 (q, $J = 7.2$ Hz, 2H), 3.79 (s, 3H), 2.30-2.64 (m, 4H), 1.93-2.04 (m, 1H), 1.34 (t, $J = 7.2$ Hz, 3H), 0.80 (d, $J = 6.8$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.1, 157.7, 139.5, 136.0, 133.3, 133.1, 130.2, 129.4, 128.3, 113.7, 60.9, 55.4, 43.0, 35.3, 33.8, 19.5, 14.4.

IR (Film) ν 2956, 2926, 1707, 1612, 1511, 1461, 1246, 1177, 1036, 769 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{22}\text{H}_{26}\text{O}_3$ (M^+): 338.1882; Found: 338.1878.



Tert-butyl (*E*)-4-(2-(ethoxycarbonyl)-3-phenylallyl)-4-methylpiperidine-1-carboxylate

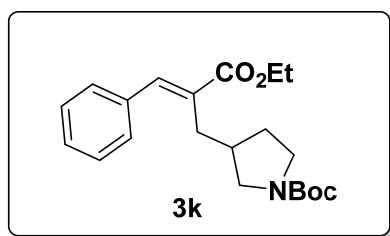
Pale yellow liquid; (70mg, 90% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:10).

^1H NMR (400 MHz, CDCl_3) δ 7.73 (s, 1H), 7.29-7.37 (m, 5H), 4.27 (q, $J = 7.2$ Hz, 2H), 3.36-3.39 (m, 2H), 2.99-3.05 (m, 2H), 2.70 (s, 2H), 1.42 (s, 9H), 1.35 (t, $J = 7.2$ Hz, 3H), 1.12-1.27 (m, 4H), 0.75 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.7, 155.0, 140.7, 136.6, 131.7, 128.9, 128.6, 128.0, 79.3, 61.1, 36.7, 36.5, 34.5, 28.6, 23.2, 14.4.

IR (Film) ν 3402, 2975, 2929, 2872, 1694, 1446, 1391, 1365, 1246, 1159, 1093, 1022, 780 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{23}\text{H}_{33}\text{O}_4\text{N}(\text{M}^+)$: 387.2410; Found: 387.2403.



***Tert*-butyl (*E*)-3-(2-(ethoxycarbonyl)-3-phenylallyl) pyrrolidine-1-carboxylate**

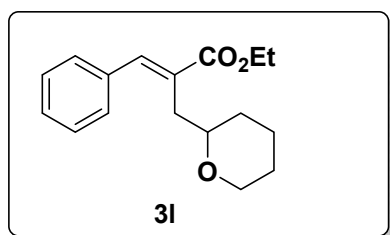
Pale yellow liquid; (38mg, 53% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:10).

^1H NMR (400 MHz, CDCl_3) δ 7.74 (s, 1H), 7.32-7.39 (m, 5H), 4.25-4.31 (m, 2H), 3.12-3.52 (m, 3H), 2.81-2.89 (m, 1H), 2.64-2.66 (m, 2H), 2.32-2.42 (m, 1H), 1.85-1.94 (m, 1H), 1.44-1.55 (m, 1H), 1.42 (s, 9H), 1.36 (td, $J_1 = 7.2$ Hz, $J_2 = 2.8$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 154.7, 140.3, 135.8, 132.2, 129.2, 129.1, 128.7, 79.1, 61.1, 51.0, 45.3, 38.9, 38.1, 31.6, 30.9, 29.8, 28.7, 14.5.

IR (Film) ν 3374, 2973, 2917, 1699, 1582, 1400, 1249, 1129, 770 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{21}\text{H}_{29}\text{NO}_4 (\text{M}^+)$: 359.2097; Found: 359.2096.



Ethyl (*E*)-3-phenyl-2-(tetrahydro-2H-pyran-2-yl) acrylate

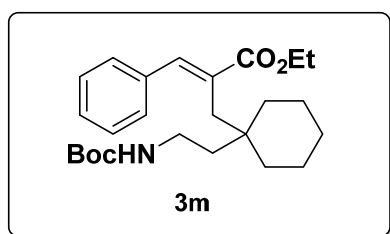
Pale yellow liquid; (41mg, 75% yield), R_f = 0.6 (EtOAc/Petroleum ether 1:50).

^1H NMR (400 MHz, CDCl_3) δ 7.75 (s, 1H), 7.54 (d, J = 7.2 Hz, 2H), 7.32-7.40 (m, 3H), 4.29 (q, J = 7.2 Hz, 2H), 3.95-3.98 (m, 1H), 3.54-3.58 (m, 1H), 3.38 (td, J_1 = 11.6 Hz, J_2 = 2.0 Hz, 1H), 2.80 (dd, J_1 = 14.0 Hz, J_2 = 8.0 Hz, 1H), 2.66 (dd, J_1 = 13.6 Hz, J_2 = 6.4 Hz, 1H), 1.44-1.64 (m, 4H), 1.35 (t, J = 7.2 Hz, 3H), 1.24-1.28 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 168.7, 140.7, 135.8, 130.4, 129.7, 128.5, 77.0, 68.8, 60.9, 34.5, 32.2, 26.2, 23.7, 14.4.

IR (Film) ν 2935, 2853, 2360, 1704, 1448, 1215, 1129, 1083, 760 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{17}\text{H}_{22}\text{O}_3$ (M^+): 274.1569; Found: 274.1566.



Ethyl (*E*)-2-((1-(2-((*tert*-butoxycarbonyl) amino) ethyl) cyclohexyl) methyl)-3-phenylacrylate

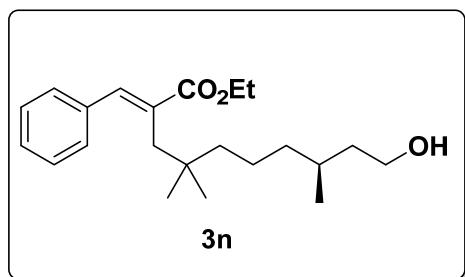
Pale yellow liquid; (56mg, 67% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.68 (s, 1H), 7.30-7.40 (m, 5H), 4.26 (q, J = 7.2 Hz, 2H), 2.68 (s, 2H), 1.42 (s, 9H), 1.02-1.37 (m, 17H).

^{13}C NMR (100 MHz, CDCl_3) δ 170.3, 140.0, 136.8, 132.7, 128.9, 128.7, 128.1, 61.1, 37.9, 36.2, 35.1, 34.6, 28.6, 26.0, 21.7, 14.4.

IR (Film) ν 3372, 2920, 2850, 1711, 1629, 1451, 1365, 1201, 1096, 889, 780 cm^{-1} .

HRMS (EI): calcd for C₂₅H₃₇NO₄ (M⁺): 415.2723; Found: 415.2721.



Ethyl (S, E)-2-benzylidene-10-hydroxy-4, 4, 8-trimethyldecanoate

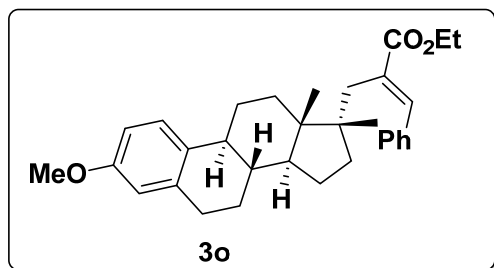
Pale yellow liquid; (35mg, 52% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:5).

¹H NMR (400 MHz, CDCl₃) δ 7.65 (s, 1H), 7.27-7.38 (m, 5H), 4.25 (q, *J* = 7.2 Hz, 2H), 3.59-3.70 (m, 2H), 2.64 (s, 2H), 1.50-1.58 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.01-1.31 (m, 7H), 0.83 (d, *J* = 6.4 Hz, 3H), 0.68 (s, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 170.1, 139.7, 136.9, 133.1, 129.0, 128.5, 127.8, 61.3, 61.0, 43.1, 40.1, 38.0, 36.8, 36.0, 29.5, 26.8, 21.4, 19.7, 14.4.

IR (Film) ν 3370, 2952, 2917, 1712, 1617, 1465, 1367, 1187, 1073, 825 cm⁻¹.

HRMS (EI): calcd for C₂₂H₃₄O₃ (M⁺): 346.2508; Found: 346.2508.



Ethyl (E)-2-(((8S, 9S, 13S, 14S)-3-methoxy-9, 13, 17-trimethyl-7, 8, 9, 11, 12, 13, 14, 15, 16, 17-decahydro-6H-cyclopenta[a]phenanthren-17-yl) methyl)-3-phenylacrylate

Pale yellow liquid; (33mg, 35% yield), R_f = 0.3 (EtOAc/Petroleum ether 1:5).

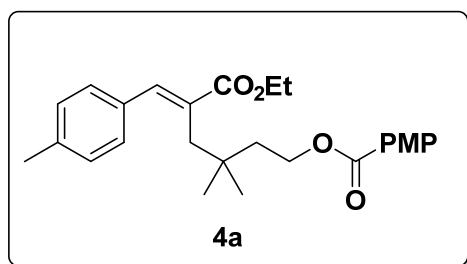
¹H NMR (400 MHz, CDCl₃) δ 7.68 (s, 1H), 7.19-7.29 (m, 6H), 7.13 (d, *J* = 8.8 Hz, 1H), 6.55 (d, *J* = 2.4 Hz, 1H), 6.23 (dd, *J*₁ = 8.8 Hz, *J*₂ = 2.8 Hz, 1H), 4.21 (q, *J* = 7.2 Hz, 2H), 3.70 (s, 3H), 2.74-2.82 (m, 3H), 2.54-2.57 (m, 1H), 2.18-1.22 (m, 1H),

2.03-2.09 (m, 1H), 1.67-1.71 (m, 1H), 1.35-1.48 (m, 5H), 1.30 (t, $J = 7.2$ Hz, 3H), 1.16-1.26 (m, 4H), 0.63 (s, 3H), 0.60 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 170.4, 157.5, 140.4, 138.3, 136.9, 134.1, 133.1, 129.1, 128.5, 127.9, 126.4, 113.9, 111.5, 61.1, 55.3, 49.7, 48.7, 46.4, 44.0, 39.7, 32.2, 31.4, 31.3, 30.0, 28.4, 26.4, 24.4, 21.7, 15.9, 14.4.

IR (Film) ν 2925, 2853, 2360, 1708, 1494, 1457, 1234, 1043, 796 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{32}\text{H}_{40}\text{O}_3$ (M^+): 472.2977; Found: 472.2977.



(E)-5-(ethoxycarbonyl)-3, 3-dimethyl-6-(p-tolyl) hex-5-en-1-yl 4-methoxybenzoate

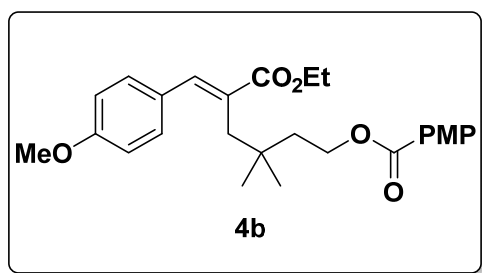
Pale yellow liquid; (50mg, 59% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.8$ Hz, 2H), 7.70 (s, 1H), 7.24 (d, $J = 8.0$ Hz, 2H), 7.12 (d, $J = 8.0$ Hz, 2H), 6.91 (d, $J = 9.2$ Hz, 2H), 4.26 (t, $J = 7.2$ Hz, 2H), 4.16 (t, $J = 7.2$ Hz, 2H), 3.85 (s, 3H), 2.76 (s, 2H), 2.30 (s, 3H), 1.58 (t, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H), 0.81 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 166.4, 163.3, 140.6, 138.0, 133.6, 131.61, 131.57, 129.3, 129.0, 123.0, 113.7, 62.0, 61.0, 55.5, 40.5, 36.8, 35.4, 27.1, 21.4, 14.4.

IR (Film) ν 2961, 2282, 1710, 1606, 1510, 1316, 1275, 1256, 1167, 1102, 1029, 848 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{26}\text{H}_{32}\text{O}_5$ (M^+): 424.2250; Found: 424.2253.



(E)-5-(ethoxycarbonyl)-6-(4-methoxyphenyl)-3,3-dimethylhex-5-en-1-yl 4-methoxybenzoate

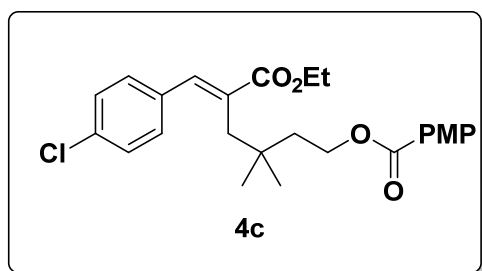
Pale yellow liquid; (60mg, 68% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 9.2$ Hz, 2H), 7.68 (s, 1H), 6.90 (d, $J = 8.8$ Hz, 2H), 6.85 (d, $J = 8.8$ Hz, 2H), 4.19-4.28 (m, 4H), 3.85 (s, 3H), 3.78 (s, 3H), 2.78 (s, 2H), 1.61 (t, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H), 0.83 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 170.0, 166.4, 163.3, 159.4, 140.2, 131.6, 130.6, 130.5, 128.9, 123.0, 114.0, 113.6, 62.0, 61.0, 55.5, 55.3, 40.6, 36.8, 35.5, 27.1, 14.4.

IR (Film) ν 2961, 2935, 1710, 1605, 1510, 1464, 1315, 1300, 1275, 1168, 1102, 1030, 847 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{26}\text{H}_{32}\text{O}_6$ (M^+): 440.2199; Found: 440.2193.



(E)-6-(4-chlorophenyl)-5-(ethoxycarbonyl)-3,3-dimethylhex-5-en-1-yl 4-methoxybenzoate

Pale yellow liquid; (64mg, 72% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

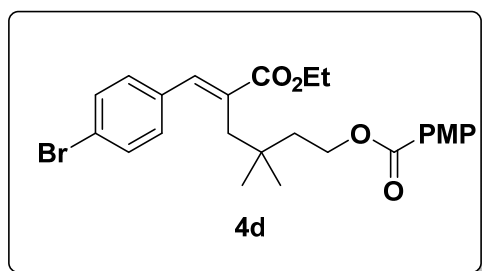
^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.8$ Hz, 2H), 7.66 (s, 1H), 7.25-7.31 (m, 4H), 6.91 (d, $J = 8.8$ Hz, 2H), 4.26 (q, $J = 7.2$ Hz, 2H), 4.20 (t, $J = 7.2$ Hz, 2H), 3.86 (s, 3H), 2.71 (s, 2H), 1.57 (t, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H), 0.80 (s,

6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.4, 166.4, 163.4, 139.1, 135.1, 133.0, 131.6, 130.2, 128.8, 122.9, 113.7, 61.9, 61.2, 55.5, 40.6, 37.0, 35.5, 27.0, 14.3.

IR (Film) ν 2962, 2935, 1711, 1606, 1511, 1275, 1167, 1101, 847 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{25}\text{H}_{29}\text{ClO}_5$ (M^+): 444.1704; Found: 444.1700.



(E)-6-(4-bromophenyl)-5-(ethoxycarbonyl)-3, 3-dimethylhex-5-en-1-yl 4-methoxybenzoate

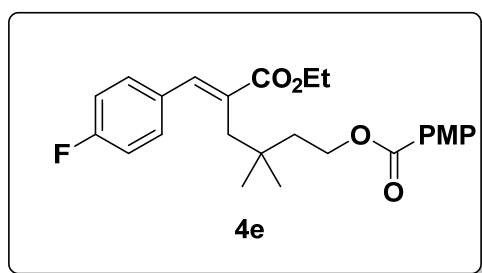
Pale yellow liquid; (73mg, 75% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, J = 8.4 Hz, 2H), 7.63 (s, 1H), 7.45 (d, J = 8.0 Hz, 2H), 7.20 (d, J = 4.4 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 4.25 (q, J = 7.2 Hz, 2H), 4.20 (t, J = 7.2 Hz, 2H), 3.86 (s, 3H), 2.71 (s, 2H), 1.57 (t, J = 7.2 Hz, 2H), 1.29-1.36 (m, 3H), 0.80 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 166.4, 163.4, 139.1, 135.6, 133.1, 131.8, 131.6, 130.5, 123.0, 122.1, 113.7, 61.9, 61.2, 55.6, 40.7, 37.1, 35.5, 31.6, 30.3, 29.8, 27.0, 14.4.

IR (Film) ν 2950, 2917, 1711, 1605, 1465, 1264, 1133, 1077, 1027, 785 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{25}\text{H}_{29}\text{BrO}_5$ (M^+): 488.1198; Found: 488.1192.



(E)-5-(ethoxycarbonyl)-6-(4-fluorophenyl)-3, 3-dimethylhex-5-en-1-yl

4-methoxybenzoate

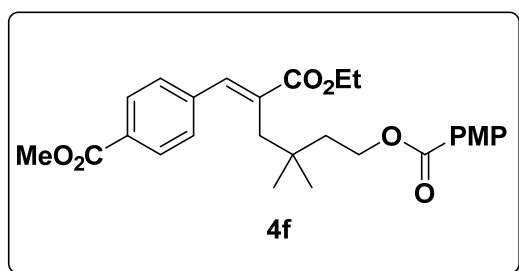
Pale yellow liquid; (60mg, 70% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.8$ Hz, 2H), 7.68 (s, 1H), 7.27-7.33 (m, 2H), 7.02 (t, $J = 8.8$ Hz, 2H), 6.91 (d, $J = 8.8$ Hz, 2H), 4.27 (q, $J = 7.2$ Hz, 2H), 4.18 (t, $J = 7.2$ Hz, 2H), 3.86 (s, 3H), 2.72 (s, 2H), 1.58 (t, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H), 0.80 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 166.4, 163.4, 139.3, 132.7, 132.5, 131.6, 130.7, 123.1, 115.8, 115.6, 113.7, 61.9, 61.1, 55.5, 40.8, 37.0, 35.5, 27.0, 14.4.

IR (Film) ν 2962, 2915, 1745, 1716, 1369, 1331, 1226, 1167, 1127, 1075, 965 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{25}\text{H}_{29}\text{FO}_5$ (M^+): 428.1999; Found: 428.1992.



(E)-5-(ethoxycarbonyl)-6-(4-(methoxycarbonyl) phenyl)-3, 3-dimethylhex-5-en-1-yl 4-methoxybenzoate

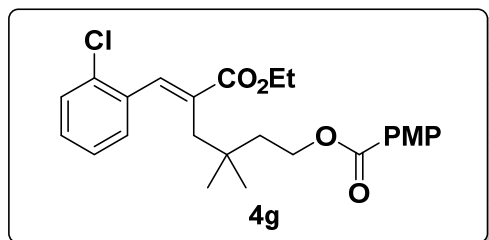
Pale yellow liquid; (77mg, 82% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:3).

^1H NMR (400 MHz, CDCl_3) δ 8.0 (d, $J = 8.0$ Hz, 2H), 7.92 (d, $J = 9.2$ Hz, 2H), 7.39 (d, $J = 8.4$ Hz, 2H), 7.23 (s, 1H), 6.90 (d, $J = 8.8$ Hz, 2H), 4.28 (q, $J = 7.2$ Hz, 2H), 4.18 (t, $J = 7.2$ Hz, 2H), 3.91 (s, 3H), 3.86 (s, 3H), 2.73 (s, 2H), 1.55 (t, $J = 7.2$ Hz, 2H), 1.35 (t, $J = 7.2$ Hz, 3H), 0.78 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.3, 166.7, 166.4, 163.4, 141.5, 139.2, 134.0, 131.6, 129.8, 129.5, 128.9, 122.9, 113.7, 61.8, 61.2, 55.5, 52.3, 40.6, 37.1, 35.5, 27.0, 14.3.

IR (Film) ν 2958, 2935, 1713, 1606, 1436, 1278, 1257, 1168, 1105, 967 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{27}\text{H}_{32}\text{O}_7$ (M^+): 468.2148; Found: 468.2142.



**(E)-6-(2-chlorophenyl)-5-(ethoxycarbonyl)-3,3-dimethylhex-5-en-1-yl
4-methoxybenzoate**

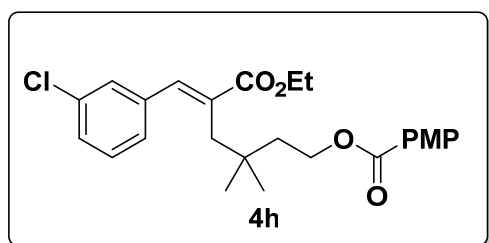
Pale yellow liquid; (43mg, 48% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, J = 8.8 Hz, 2H), 7.72 (s, 1H), 7.20-7.35 (m, 4H), 6.90 (d, J = 6.8 Hz, 2H), 4.29 (q, J = 7.2 Hz, 2H), 4.08 (t, J = 7.2 Hz, 2H), 3.86 (s, 3H), 2.62 (s, 2H), 1.53 (t, J = 7.2 Hz, 2H), 1.35 (t, J = 7.2 Hz, 3H), 0.79 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.1, 166.4, 163.3, 138.2, 135.3, 133.9, 133.6, 131.6, 130.1, 129.7, 129.3, 126.8, 123.0, 113.6, 61.9, 61.2, 55.5, 40.0, 36.9, 35.1, 26.9, 14.4.

IR (Film) ν 2961, 2917, 1712, 1605, 1467, 1276, 1167, 1102, 848 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{25}\text{H}_{29}\text{ClO}_5$ (M^+): 444.1704; Found: 444.1700.



**(E)-6-(3-chlorophenyl)-5-(ethoxycarbonyl)-3,3-dimethylhex-5-en-1-yl
4-methoxybenzoate**

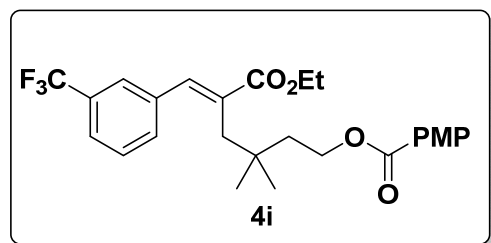
Pale yellow liquid; (78mg, 88% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.8 Hz, 2H), 7.64 (s, 1H), 7.20-7.32 (m, 4H), 6.90 (d, *J* = 8.4 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 4.19 (t, *J* = 7.2 Hz, 2H), 3.86 (s, 3H), 2.70 (s, 2H), 1.57 (t, *J* = 7.2 Hz, 2H), 1.34 (t, *J* = 7.2 Hz, 3H), 0.81 (s, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 169.4, 166.4, 163.4, 138.7, 138.5, 134.6, 133.7, 131.7, 129.9, 128.9, 128.1, 127.0, 123.0, 113.7, 61.7, 61.2, 40.7, 35.4, 27.1, 14.4.

IR (Film) ν 2916, 2848, 2358, 1709, 1511, 1370, 1257, 1166, 1226, 849 cm⁻¹.

HRMS (EI): calcd for C₂₅H₂₉ClO₅ (M⁺): 444.1704; Found: 444.1700.



(*E*)-5-(ethoxycarbonyl)-3,3-dimethyl-6-(3-(trifluoromethyl) phenyl)

hex-5-en-1-yl 4-methoxybenzoate

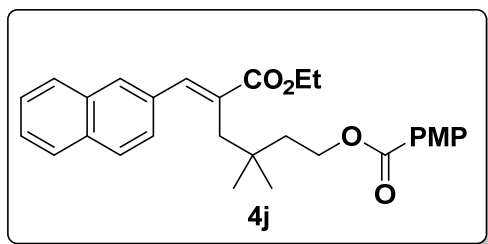
Pale yellow liquid; (88mg, 92% yield), *R*_f = 0.5 (EtOAc/Petroleum ether 1:20).

¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.8 Hz, 2H), 7.71 (s, 1H), 7.44-7.59 (m, 4H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 4.20 (t, *J* = 7.2 Hz, 2H), 3.86 (s, 3H), 2.71 (s, 2H), 1.57 (t, *J* = 7.2 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 0.80 (s, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 169.3, 166.4, 163.4, 138.5, 137.5, 134.2, 131.6, 129.2, 125.7, 124.6, 123.0, 113.7, 61.8, 61.3, 55.5, 40.7, 37.3, 35.5, 27.0, 14.3.

IR (Film) ν 2953, 2850, 1713, 1603, 1330, 1257, 1165, 1028, 898 cm⁻¹.

HRMS (EI): calcd for C₂₆H₂₉F₃O₅ (M⁺): 478.1967; Found: 478.1967.



(E)-5-(ethoxycarbonyl)-3, 3-dimethyl-6-(naphthalen-2-yl) hex-5-en-1-yl 4-methoxybenzoate

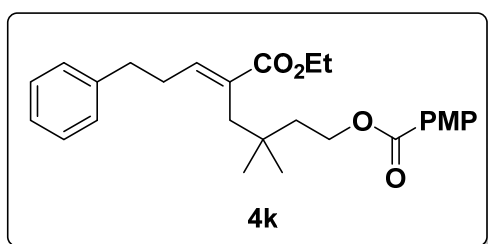
Pale yellow liquid; (78mg, 85% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.86-7.88 (m, 3H), 7.78- 7.81 (m, 4H), 7.45-7.49 (m, 3H), 6.87 (d, $J = 8.8$ Hz, 2H), 4.29 (q, $J = 7.2$ Hz, 2H), 4.17 (t, $J = 7.2$ Hz, 2H), 3.85 (s, 3H), 2.85 (s, 2H), 1.57 (t, $J = 7.2$ Hz, 2H), 1.35 (t, $J = 7.2$ Hz, 3H), 0.80 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.8, 166.4, 163.3, 140.5, 134.2, 133.3, 132.5, 131.6, 128.5, 128.2, 127.8, 126.6, 126.5, 123.0, 113.6, 62.0, 61.1, 55.5, 40.1, 37.2, 35.5, 27.0, 14.4.

IR (Film) ν 2959, 2917, 2849, 1709, 1605, 1484, 1256, 1167, 1132, 1028, 850 cm^{-1} .

HRMS (EI) : calcd for $\text{C}_{29}\text{H}_{32}\text{O}_5$ (M^+): 460.2250; Found: 460.2253.



(E)-5-(ethoxycarbonyl)-3, 3-dimethyl-8-phenyloct-5-en-1-yl 4-methoxybenzoate

Pale yellow liquid; (48mg, 55% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

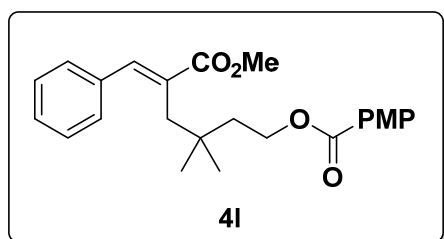
^1H NMR (400 MHz, CDCl_3) δ 0.92 (s, 6 H), 1.29 (t, $J = 7.2$ Hz, 3H), 1.71 (t, $J = 7.2$ Hz, 2H), 2.39 (s, 2H), 2.49 (dd, $J_1 = 13.6$ Hz, $J_2 = 7.6$ Hz, 2H), 2.74 (t, $J = 8.0$ Hz,

2H), 3.85 (s, 3H), 4.19 (t, $J = 7.2$ Hz, 2H), 4.36 (t, $J = 7.2$ Hz, 2H), 6.83 (t, $J = 7.2$ Hz, 1H), 6.90 (m, 2H), 7.26-7.34 (m, 5H), 7.95-7.99 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.4, 166.6, 163.4, 143.0, 141.3, 131.7, 130.9, 128.6, 128.5, 126.3, 123.0, 113.7, 62.1, 60.7, 55.5, 40.9, 38.3, 35.12, 35.08, 31.7, 26.9, 14.4.

IR (Film) ν 2960, 2953, 1711, 1605, 1511, 1275, 1257, 1167, 1102, 1030, 848 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{27}\text{H}_{34}\text{O}_5$ (M^+): 438.2406; Found: 438.2401.



(E)-5-(methoxycarbonyl)-3,3-dimethyl-6-phenylhex-5-en-1-yl 4-methoxybenzoate

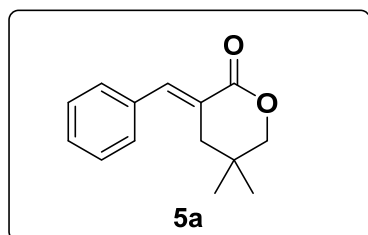
Pale yellow liquid; (65mg, 82% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.8$ Hz, 2H), 7.74 (s, 1H), 7.32-7.33 (m, 4H), 6.90 (d, $J = 8.8$ Hz, 2H), 4.16 (t, $J = 7.2$ Hz, 2H), 3.85 (s, 3H), 3.81 (s, 3H), 2.75 (s, 2H), 1.57 (t, $J = 7.2$ Hz, 3H), 0.79 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 170.2, 166.4, 163.4, 140.8, 136.6, 132.1, 131.6, 128.9, 128.6, 128.1, 123.1, 113.7, 61.9, 55.5, 52.1, 40.6, 36.9, 35.4, 27.0.

IR (Film) ν 2960, 2948, 1711, 1606, 1511, 1275, 1256, 1102, 848 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{24}\text{H}_{28}\text{O}_5$ (M^+): 396.1937; Found: 396.1933.



(E)-3-benzylidene-5,5-dimethyltetrahydro-2H-pyran-2-one

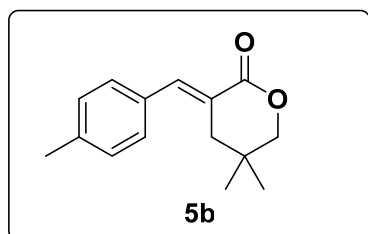
Pale yellow solid; mp: 95-97 °C; (28mg, 65% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.97 (s, 1H), 7.32-7.43 (m, 5H), 4.07 (s, 2H), 2.67 (s, 2H), 1.06 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 166.7, 142.7, 135.1, 130.3, 129.3, 128.7, 124.5, 77.7, 39.8, 30.4, 24.7.

IR (Film) ν 2964, 2917, 1694, 1618, 1283, 1238, 1183, 1107, 911 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{14}\text{H}_{16}\text{O}_2$ (M^+): 216.1150; Found: 216.1150.



(E)-5, 5-dimethyl-3-(4-methylbenzylidene) tetrahydro-2H-pyran-2-one

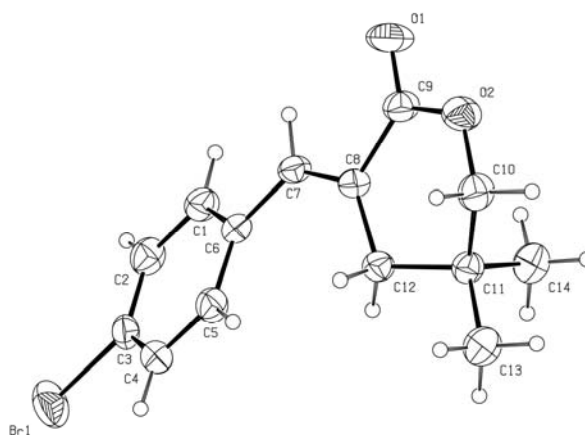
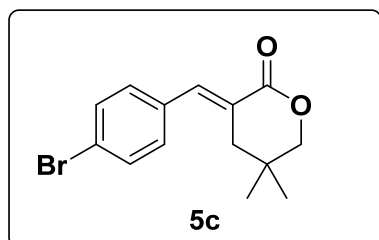
Pale yellow solid; mp: 68-71 °C; (34mg, 73% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.95 (s, 1H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.23 (d, $J = 8.0$ Hz, 2H), 4.06 (s, 2H), 2.66 (s, 2H), 2.39 (s, 3H), 1.06 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 167.0, 142.8, 139.7, 132.3, 130.5, 129.4, 123.5, 77.7, 39.9, 30.3, 24.7, 21.6.

IR (Film) ν 2962, 2915, 2358, 1713, 1608, 1234, 1170, 1106, 815 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{15}\text{H}_{18}\text{O}_2$ (M^+): 230.1307; Found: 230.1303.



(E)-3-(4-bromobenzylidene)-5, 5-dimethyltetrahydro-2H-pyran-2-one

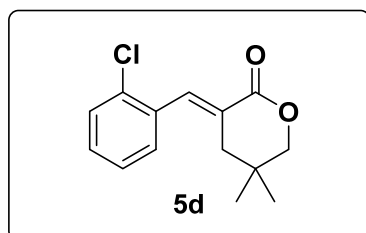
Pale yellow solid; mp: 108-110°C; (38mg, 65% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.89 (s, 1H), 7.55 (d, $J = 8.4$ Hz, 2H), 7.28 (d, $J = 8.4$ Hz, 2H), 4.07 (s, 2H), 2.62 (s, 2H), 1.06 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 141.4, 133.8, 131.9, 131.7, 125.2, 123.6, 77.7, 39.7, 30.4, 24.6, 24.2

IR (Film) ν 2959, 2919, 2357, 1710, 1617, 1379, 1234, 1173, 1010, 920 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{14}\text{H}_{15}\text{BrO}_2$ (M^+): 294.0255; Found: 294.0253.



(E)-3-(2-chlorobenzylidene)-5, 5-dimethyltetrahydro-2H-pyran-2-one

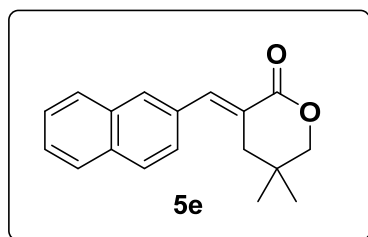
Pale yellow solid; mp: 93-95°C; (37mg, 74% yield), $R_f = 0.5$ (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 8.07 (s, 1H), 7.27-7.44 (m, 4H), 4.08 (s, 2H), 2.50 (s, 2H), 1.03 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 165.9, 139.7, 134.7, 133.5, 130.10, 130.06, 129.9, 126.9, 126.5, 78.2, 39.1, 30.5, 24.5.

IR (Film) ν 2960, 2356, 1713, 1540, 1467, 1178, 1105, 915 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{14}\text{H}_{15}\text{ClO}_2$ (M^+): 250.0761; Found: 250.0761.



(E)-5, 5-dimethyl-3-(naphthalen-2-ylmethylene) tetrahydro-2H-pyran-2-one

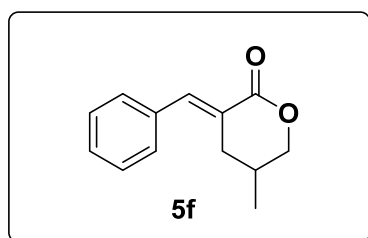
Pale yellow solid; mp:123-124°C; (32mg, 60% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 8.06 (s, 1H), 7.7-7.81 (m, 4H), 7.43-7.47 (m, 3H), 4.02 (s, 2H), 2.70 (s, 2H), 1.00 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 166.8, 142.8, 133.4, 133.1, 132.6, 130.4, 128.6, 128.3, 127.8, 127.32, 127.27, 126.8, 124.7, 77.8, 39.9, 30.4, 24.7.

IR (Film) ν 2973, 2917, 2356, 1706, 1612, 1466, 1224, 1170, 1102, 931 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{18}\text{H}_{18}\text{O}_2$ (M^+): 266.1307; Found: 266.1302.



(E)-3-benzylidene-5-methyltetrahydro-2H-pyran-2-one

Pale yellow liquid; (20mg, 50% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

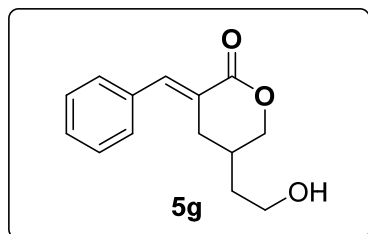
^1H NMR (400 MHz, CDCl_3) δ 7.93 (s, 1H), 7.37-7.44 (m, 5H), 4.31-4.35 (m, 1H), 4.01-4.06 (m, 1H), 2.98-3.05 (m, 1H), 2.43-2.50 (m, 1H), 2.03-2.20 (m, 1H), 1.05 (d, J = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 166.9, 142.0, 135.1, 130.3, 129.3, 128.7, 125.1,

74.0, 34.2, 28.6, 16.6.

IR (Film) ν 2970, 2953, 1694, 1612, 1245, 1187, 1049, 878 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{13}\text{H}_{14}\text{O}_2$ (M^+): 202.0994; Found: 202.0992.



(E)-3-benzylidene-5-(2-hydroxyethyl) tetrahydro-2H-pyran-2-one

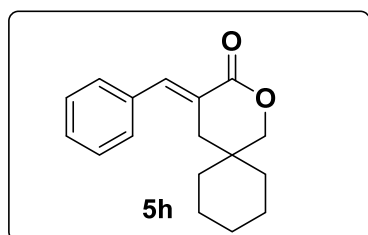
Pale yellow liquid; (29mg, 62% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:5).

^1H NMR (400 MHz, CDCl_3) δ 7.93 (s, 1H), 7.36-7.44 (m, 5H), 4.44 (d, J = 10.4, 1H), 4.14 (t, J = 9.6 Hz, 1H), 3.69-3.78 (m, 2H), 3.04-3.08 (m, 1H), 2.53-2.60 (m, 1H), 2.26 (brs, 1H), 1.29-2.13 (m, 1H), 1.58-1.69 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 167.0, 142.3, 135.0, 130.4, 129.4, 128.7, 124.8, 72.5, 60.2, 33.8, 32.3, 30.5.

IR (Film) ν 2917, 2849, 2359, 1718, 1611, 1234, 1132, 1076, 764 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{14}\text{H}_{16}\text{O}_3$ (M^+): 232.1099; Found: 232.1100 .



(E)-4-benzylidene-2-oxaspiro [5. 5] undecan-3-one

Pale yellow solid; mp: 82-83 $^{\circ}\text{C}$; (22mg, 42% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

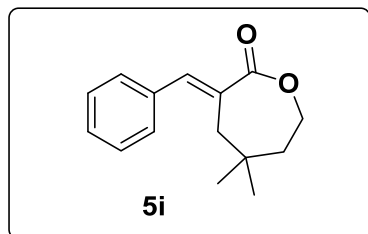
^1H NMR (400 MHz, CDCl_3) δ 7.95 (s, 1H), 7.37-7.44 (m, 5H), 4.16 (s, 2H), 2.70 (s, 2H), 1.25-1.44 (m, 10H).

^{13}C NMR (100 MHz, CDCl_3) δ 167.3, 142.6, 135.1, 130.3, 129.2, 128.7, 124.5,

75.7, 37.4, 33.1, 32.9, 30.3, 29.8, 26.2, 21.6.

IR (Film) ν 2919, 2851, 2358, 1711, 1617, 1393, 1241, 1154, 1085, 852 cm^{-1} .

HRMS (EI): calcd for $\text{C}_{17}\text{H}_{20}\text{O}_2$ (M^+): 256.1463; Found: 256.1466.



(E)-3-benzylidene-5,5-dimethyloxepan-2-one

Pale yellow solid; mp: 81-82°C; (21mg, 45% yield), R_f = 0.5 (EtOAc/Petroleum ether 1:20).

^1H NMR (400 MHz, CDCl_3) δ 7.31-7.38 (m, 6H), 4.23-4.25 (m, 2H), 2.49 (s, 2H), 1.68-1.70 (m, 2H), 0.89 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 174.7, 139.6, 135.6, 133.1, 129.1, 128.6, 128.2, 65.8, 42.5, 38.9, 33.6, 28.8.

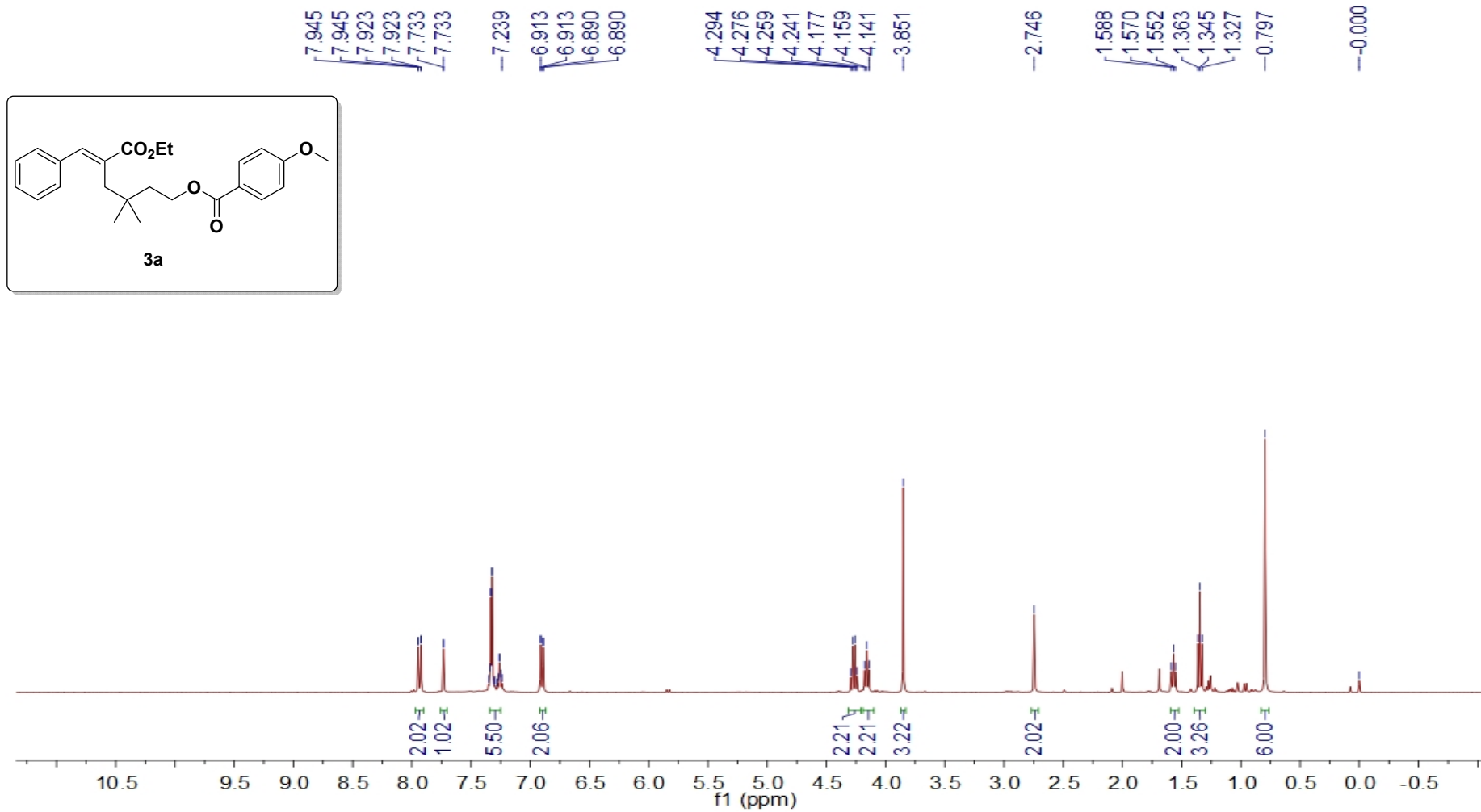
IR (Film) ν 2955, 2813, 2356, 1717, 1535, 1461, 1166, 1082, 754 cm^{-1} .

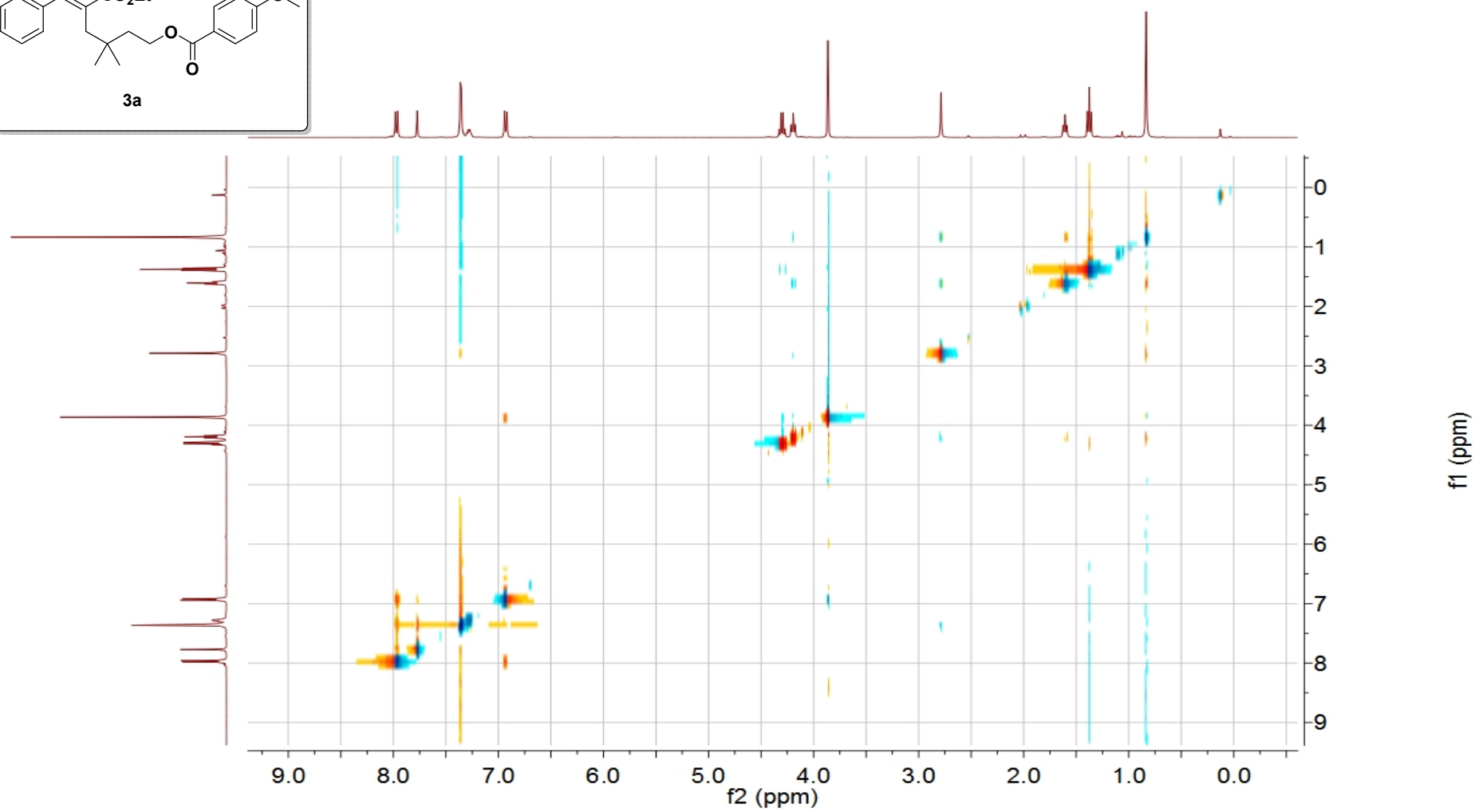
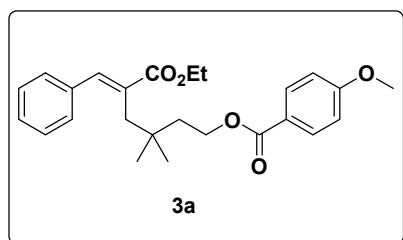
HRMS (EI): calcd for $\text{C}_{15}\text{H}_{18}\text{O}_2$ (M^+): 230.1307; Found: 230.1302.

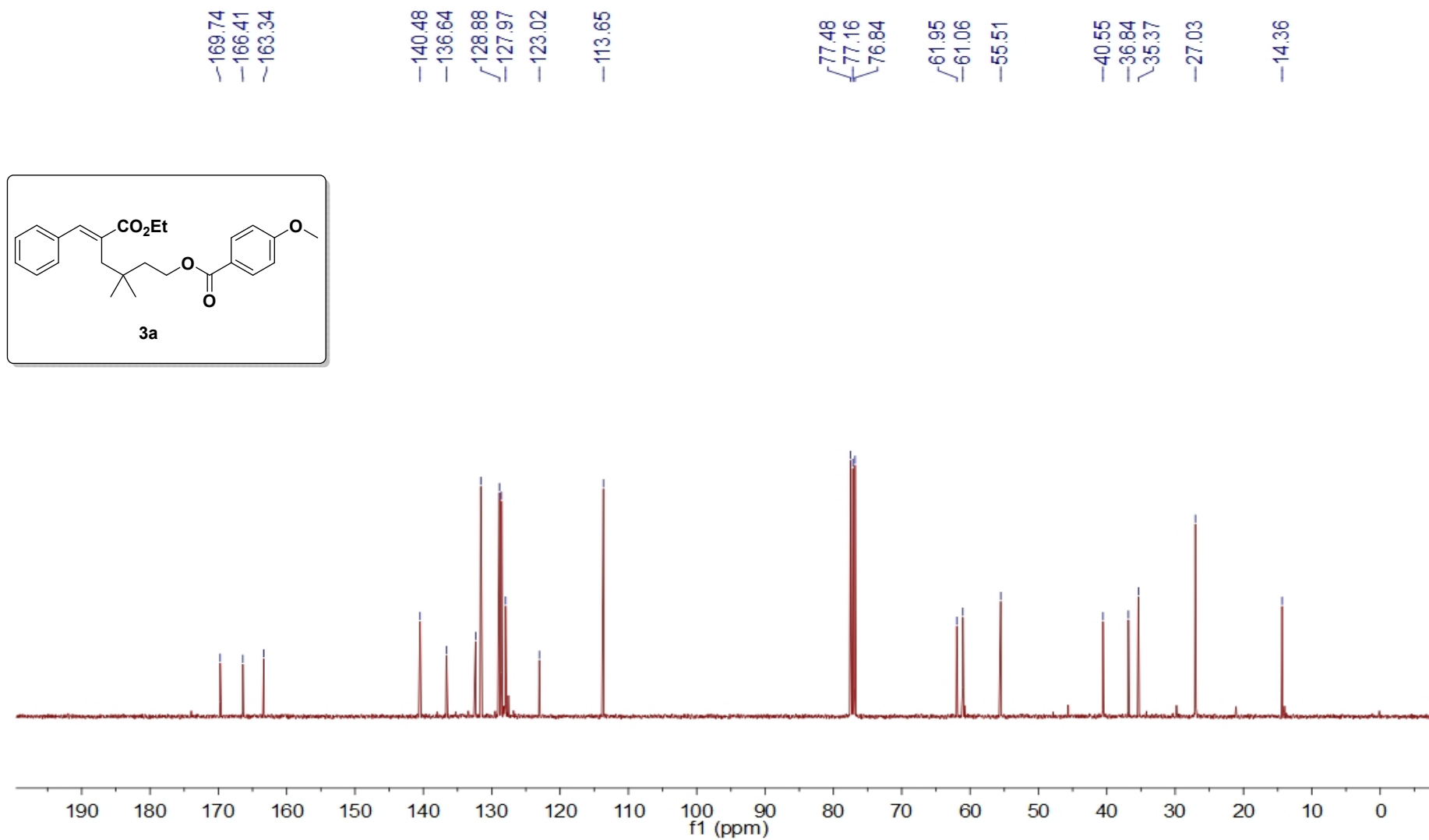
5. Reference

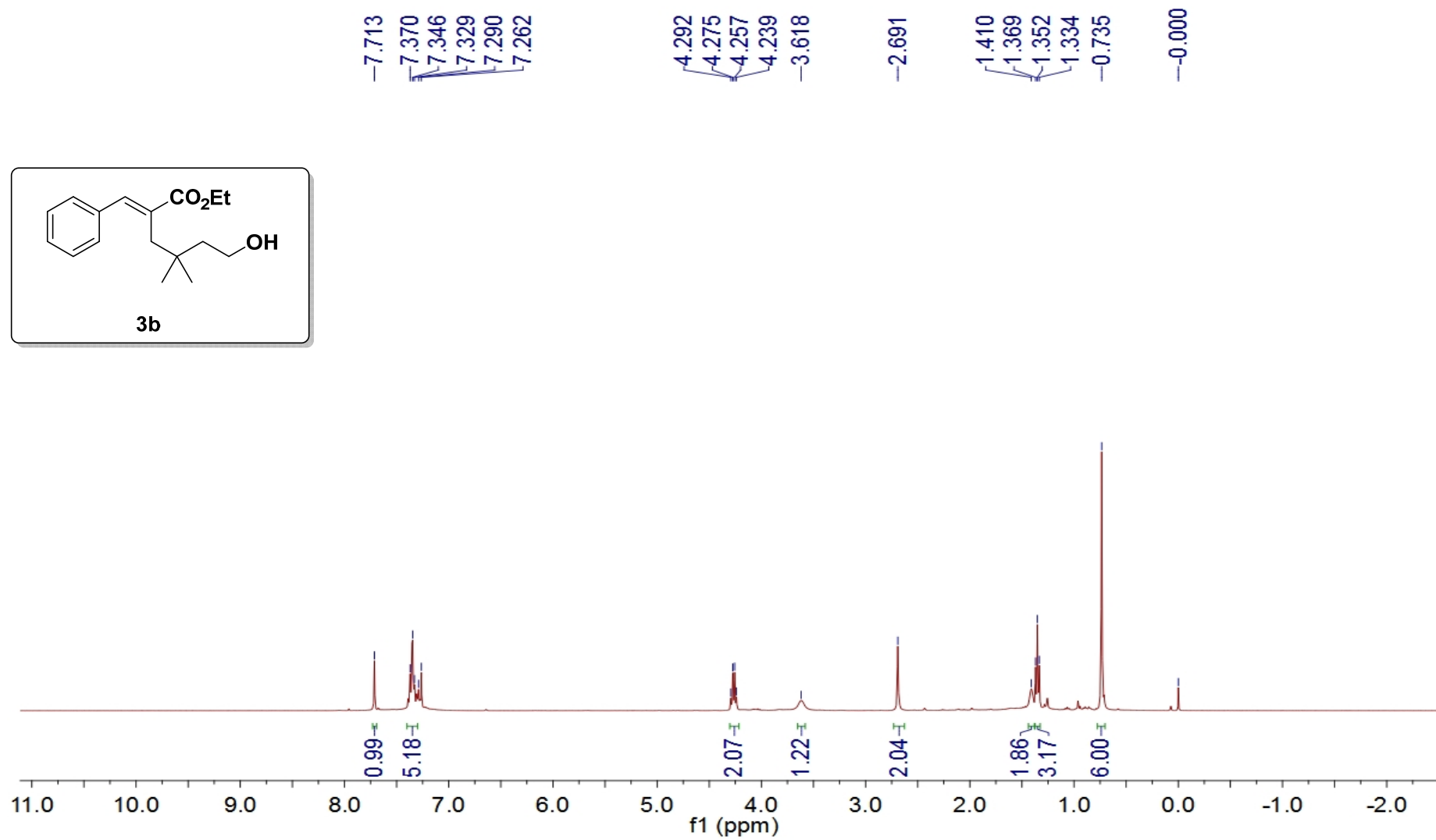
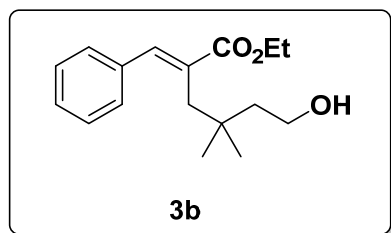
1. a) H. Batchu, S. Bhattacharyya, S. Batra, *Org. Lett.* **2012**, 14, 6330; b) J. Deng, X. Hu, J. Huang, S. Yu, D. Wang, Z. Duan, Z. Zheng, *J. Org. Chem.* **2008**, 73, 2015; c) J. Holz, B. Schäffner, O. Zayas, A. Spannenberg, A. Börner, *Advanced Synthesis & Catalysis*. **2008**, 350, 2533.

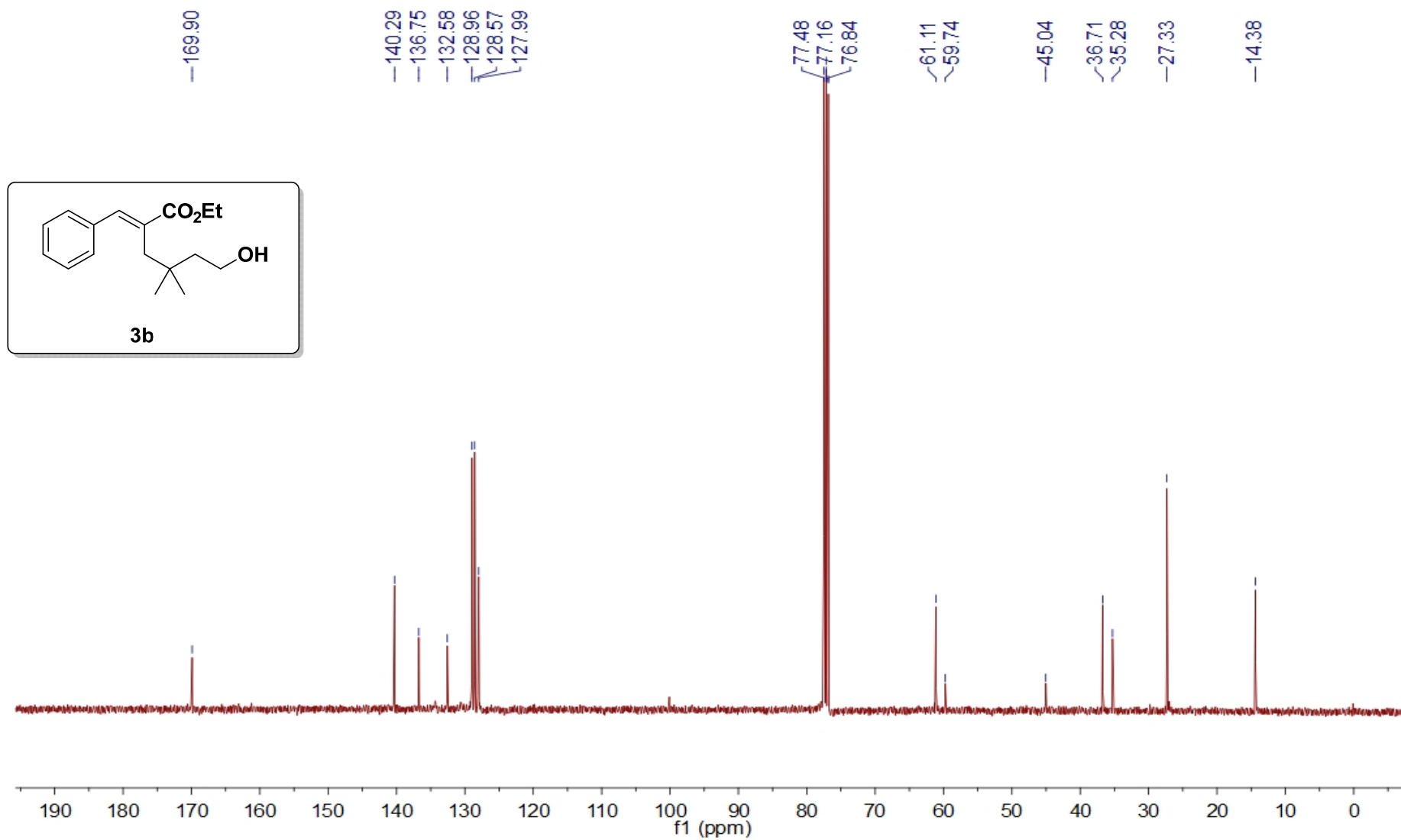
6. Copies of NMR Spectra.

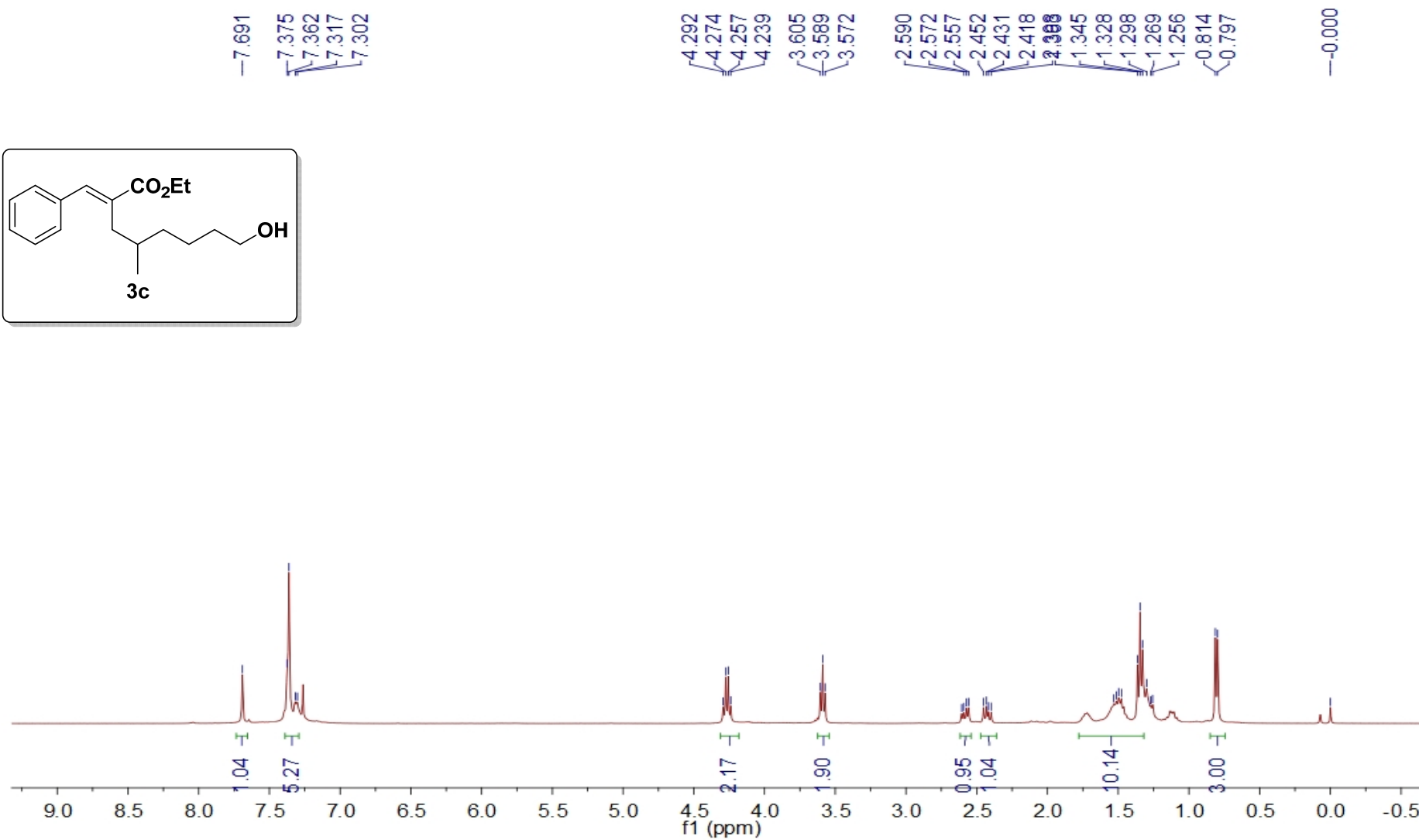
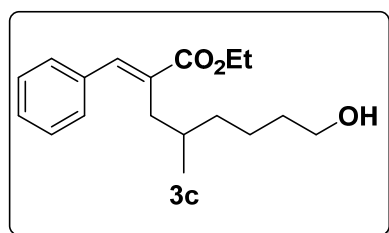


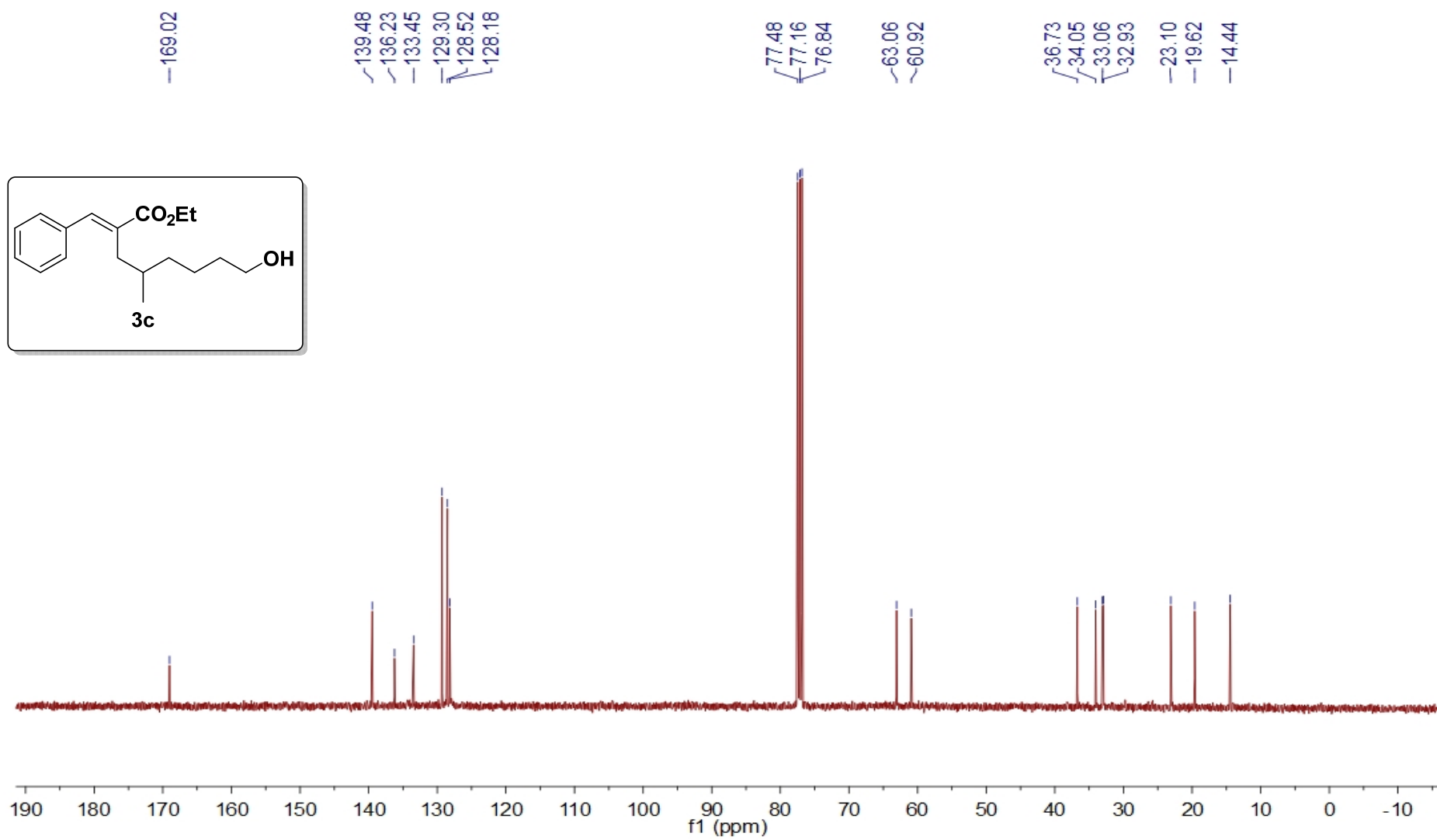


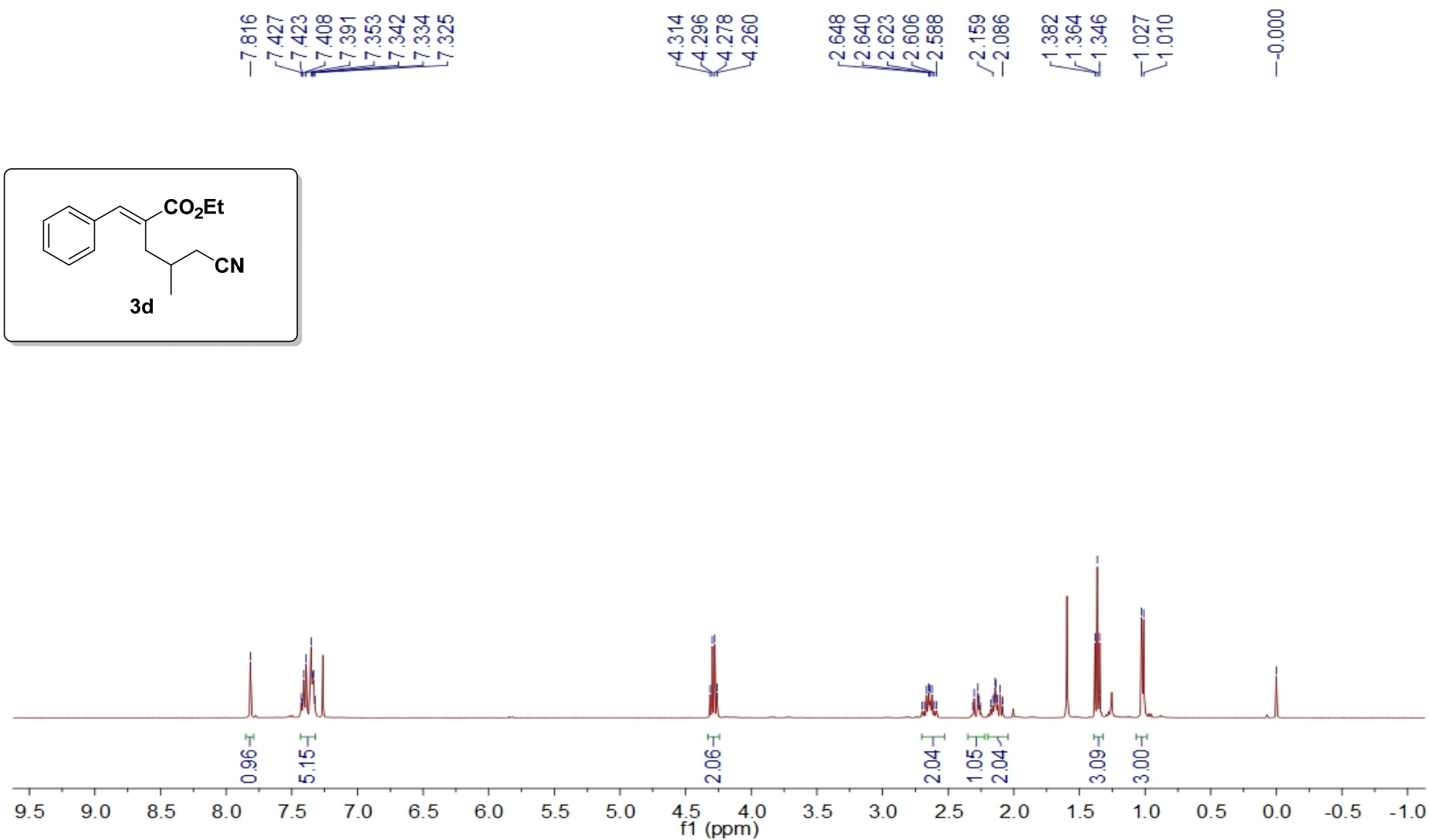
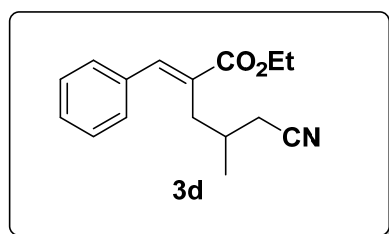


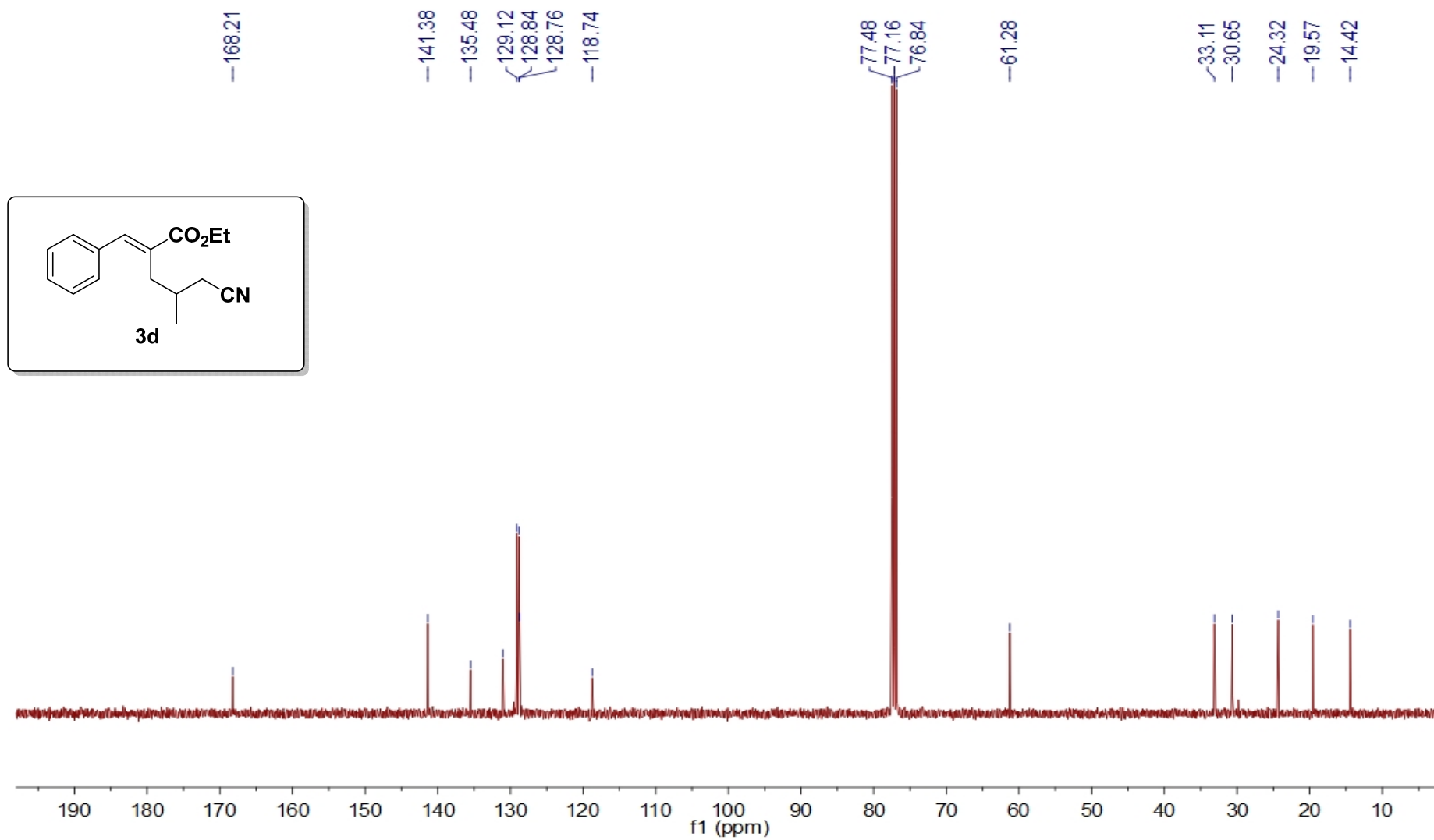


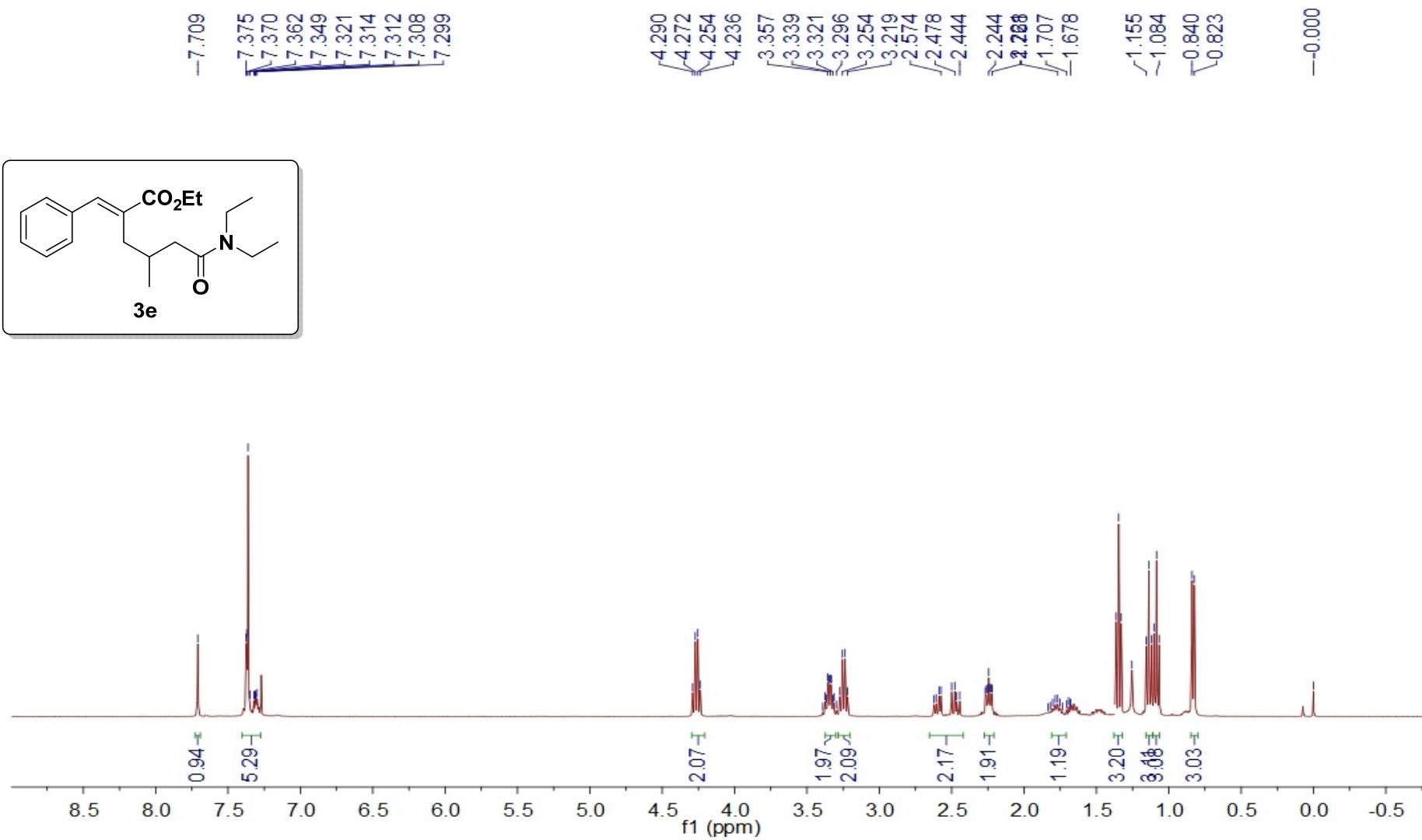
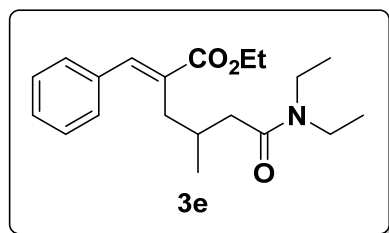


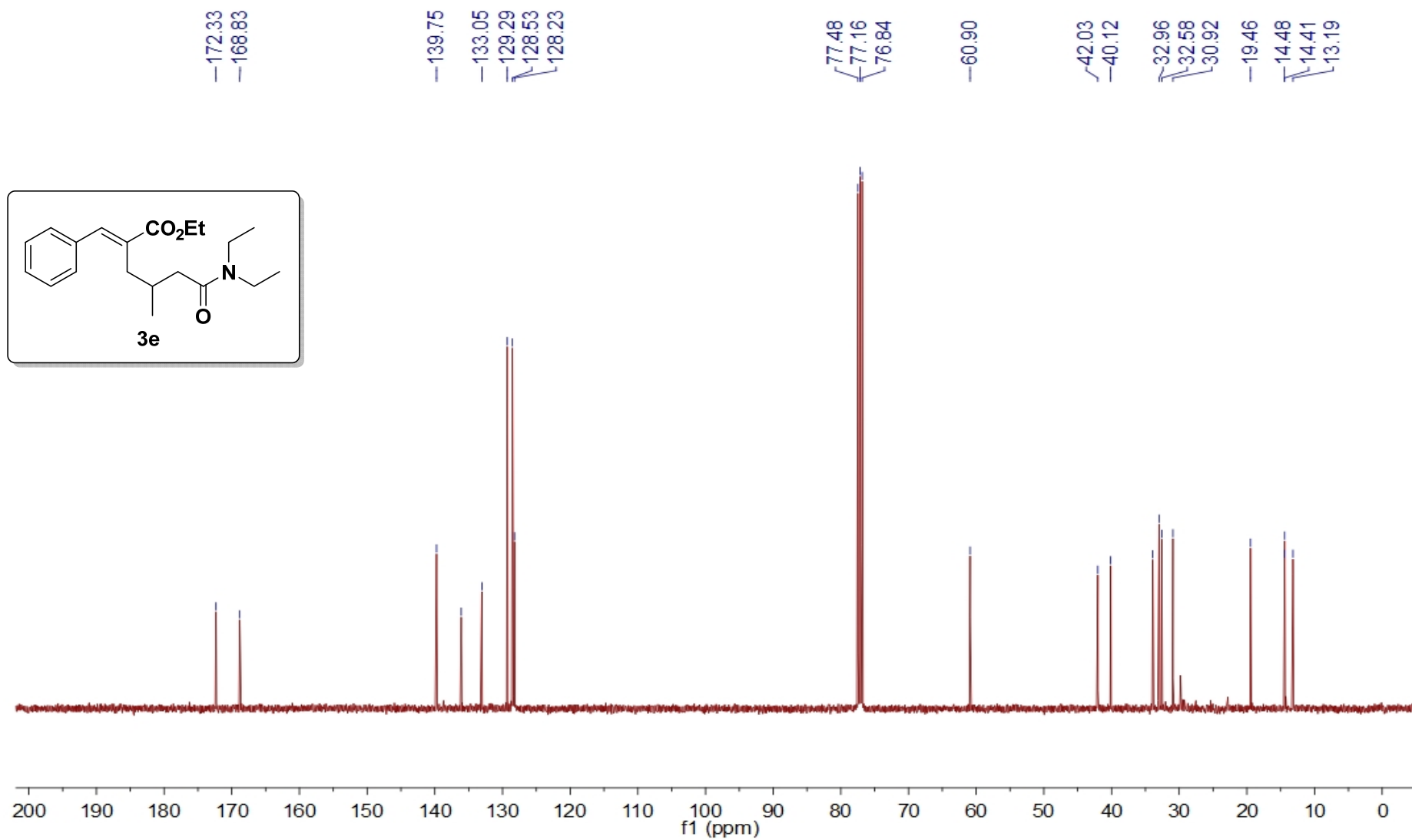


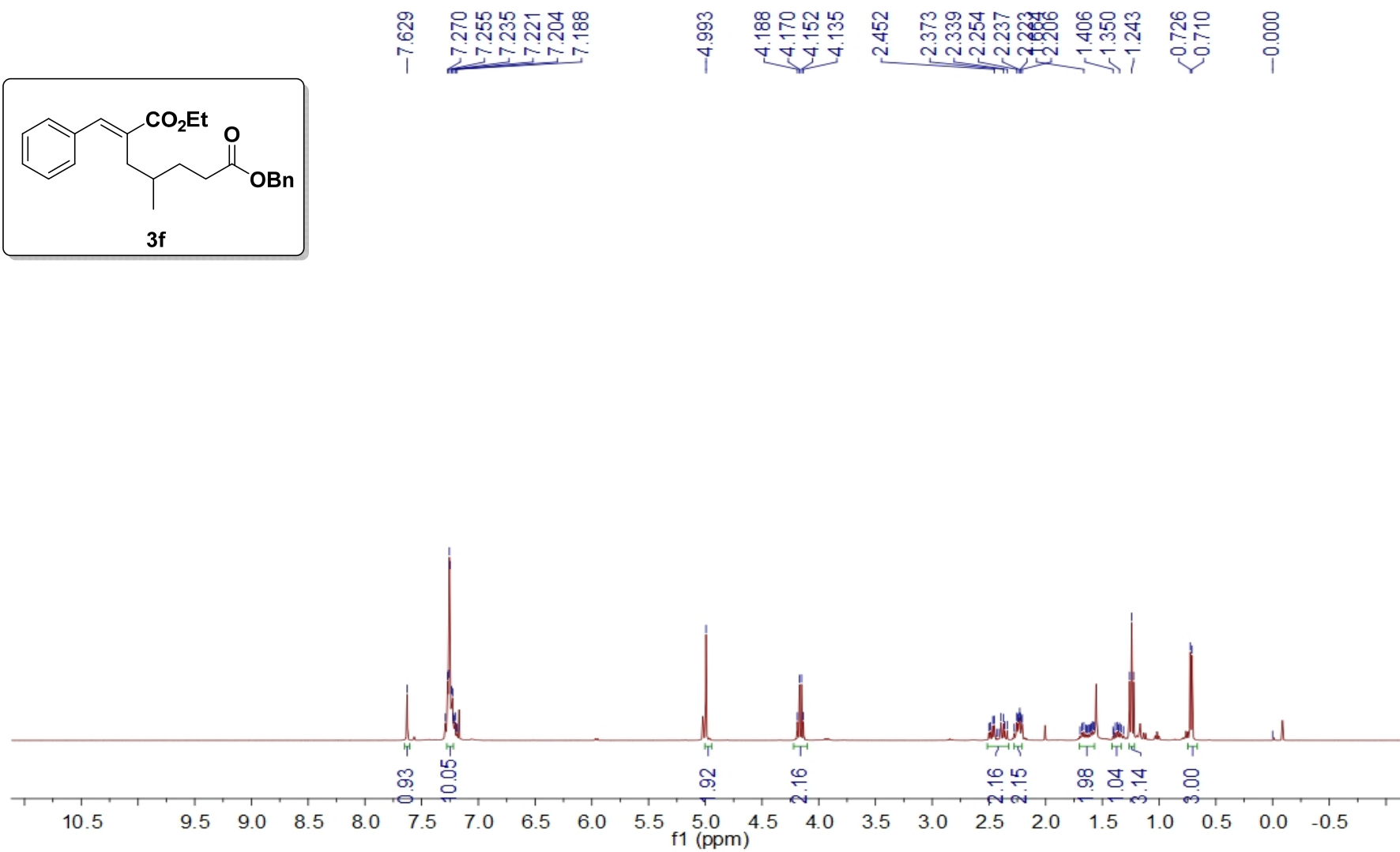
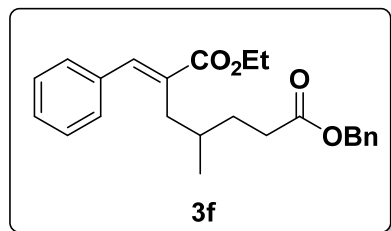


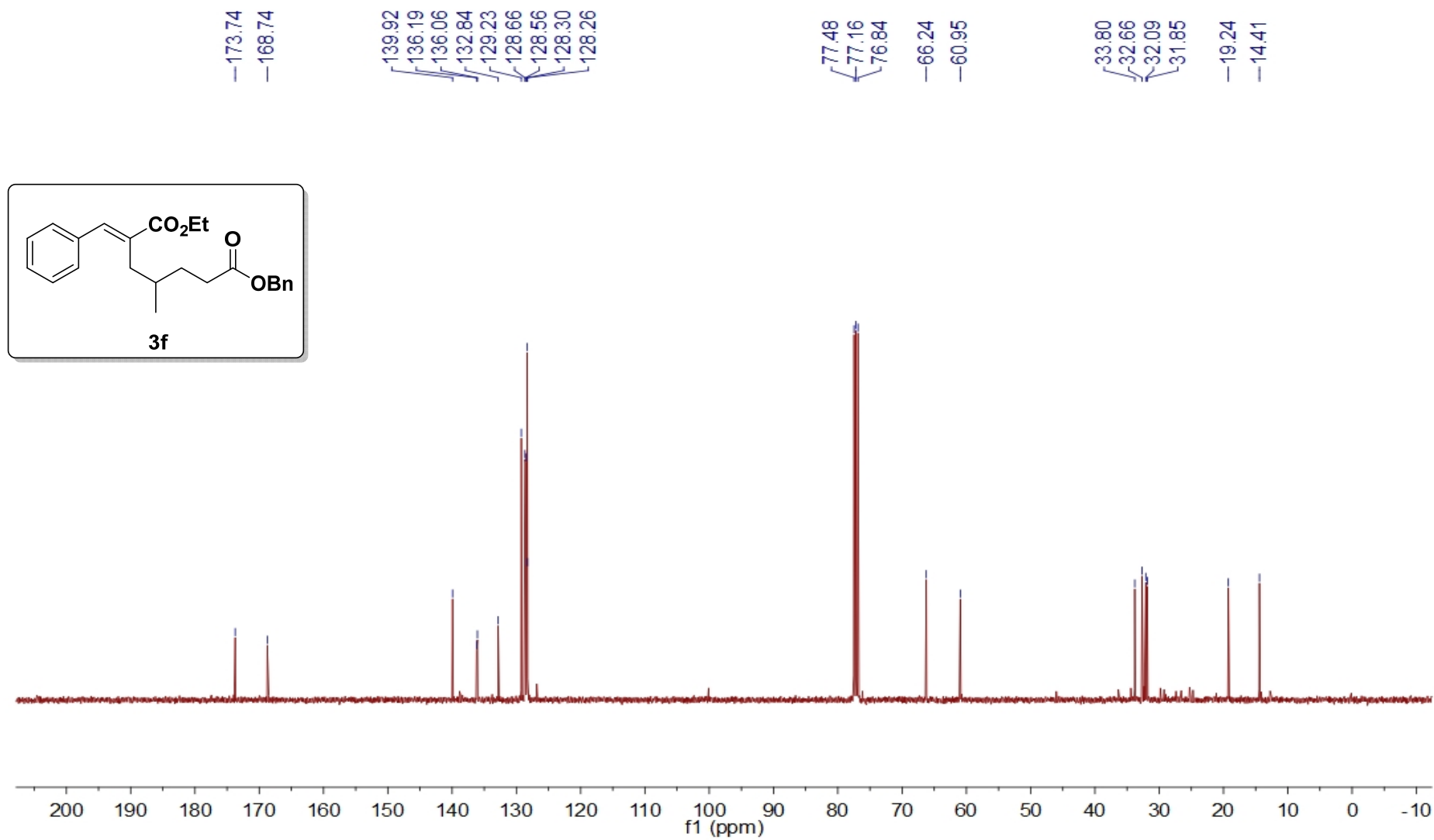


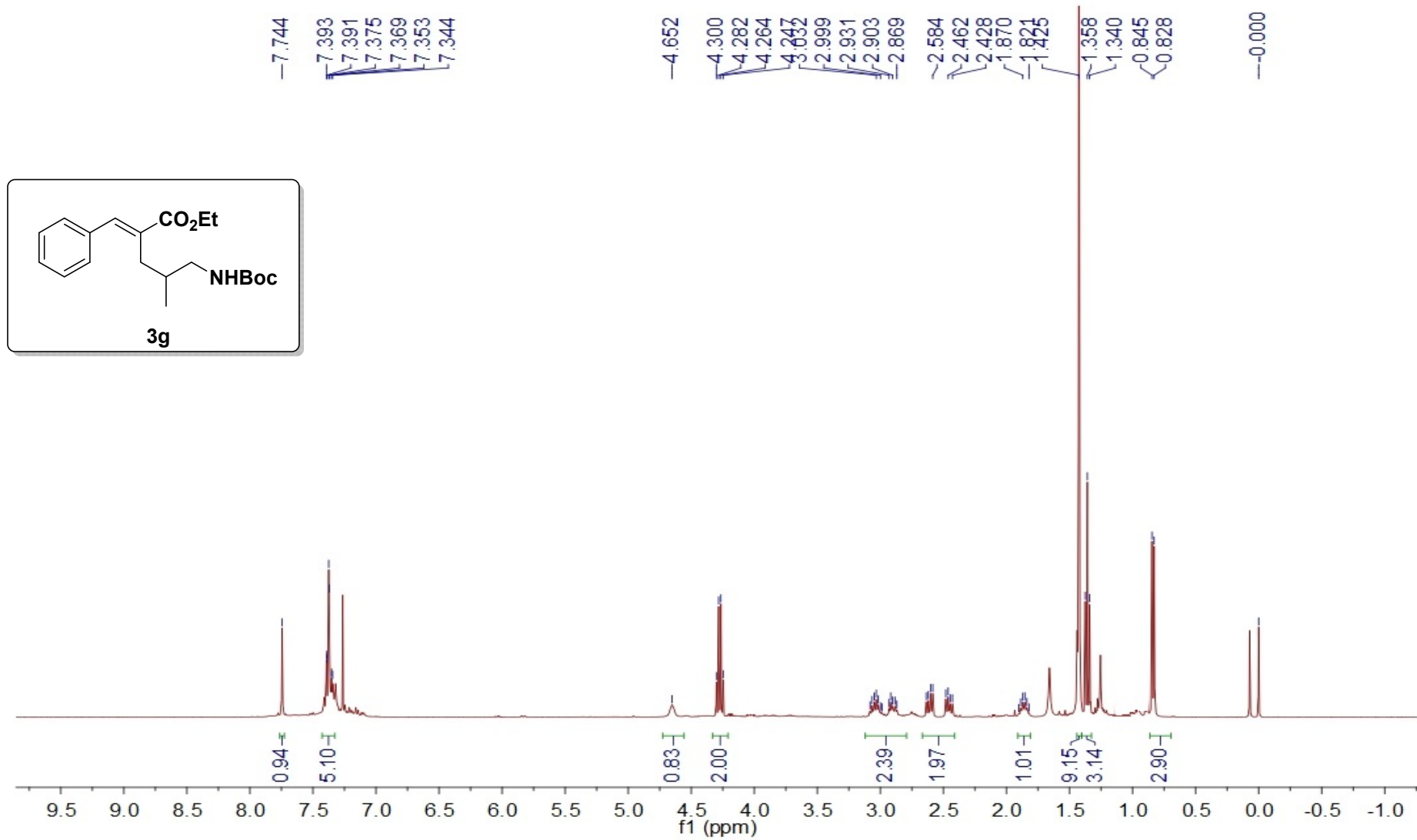


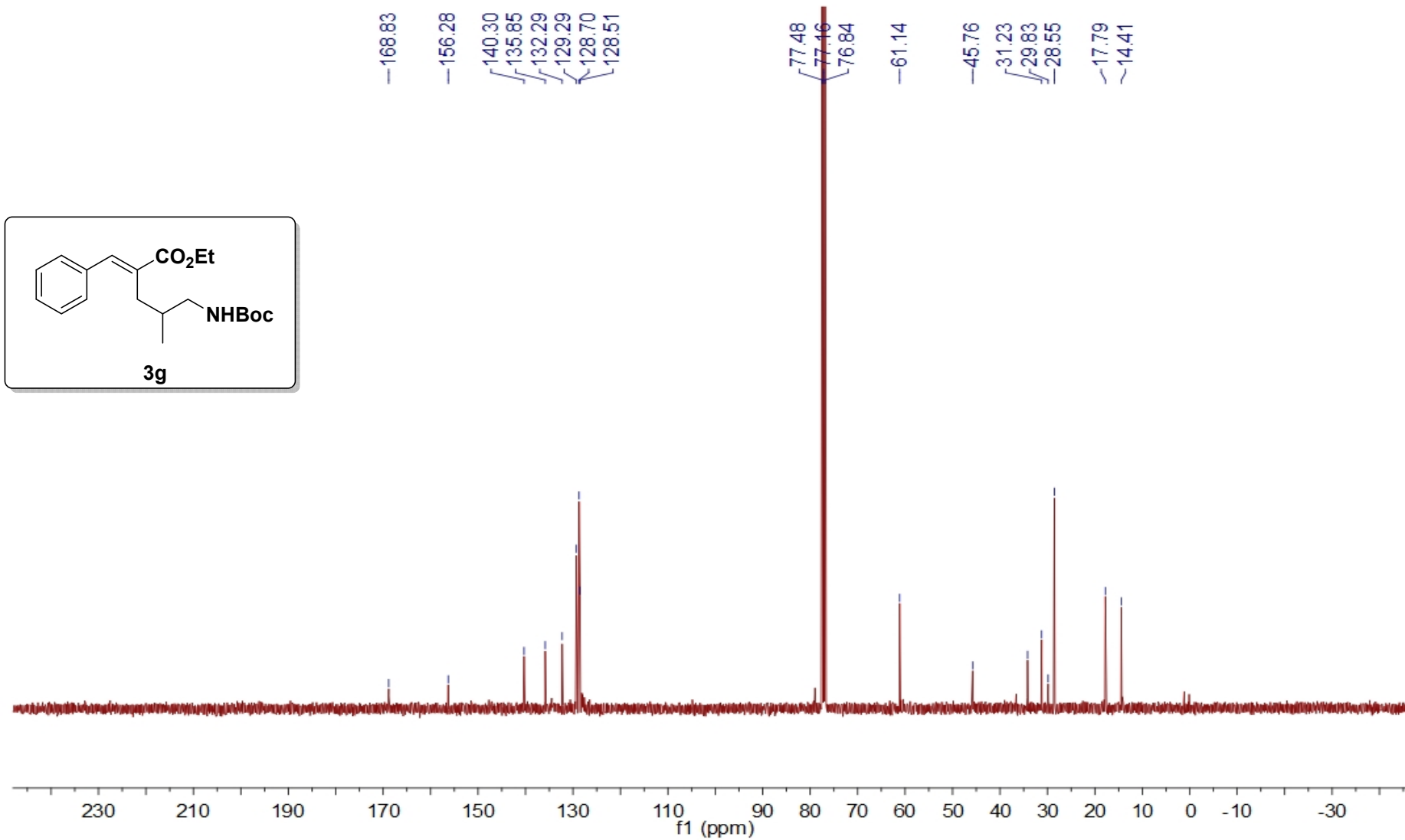
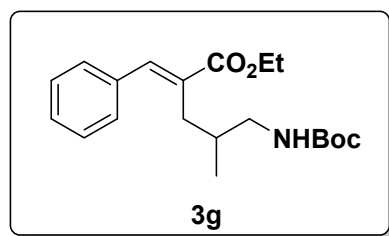


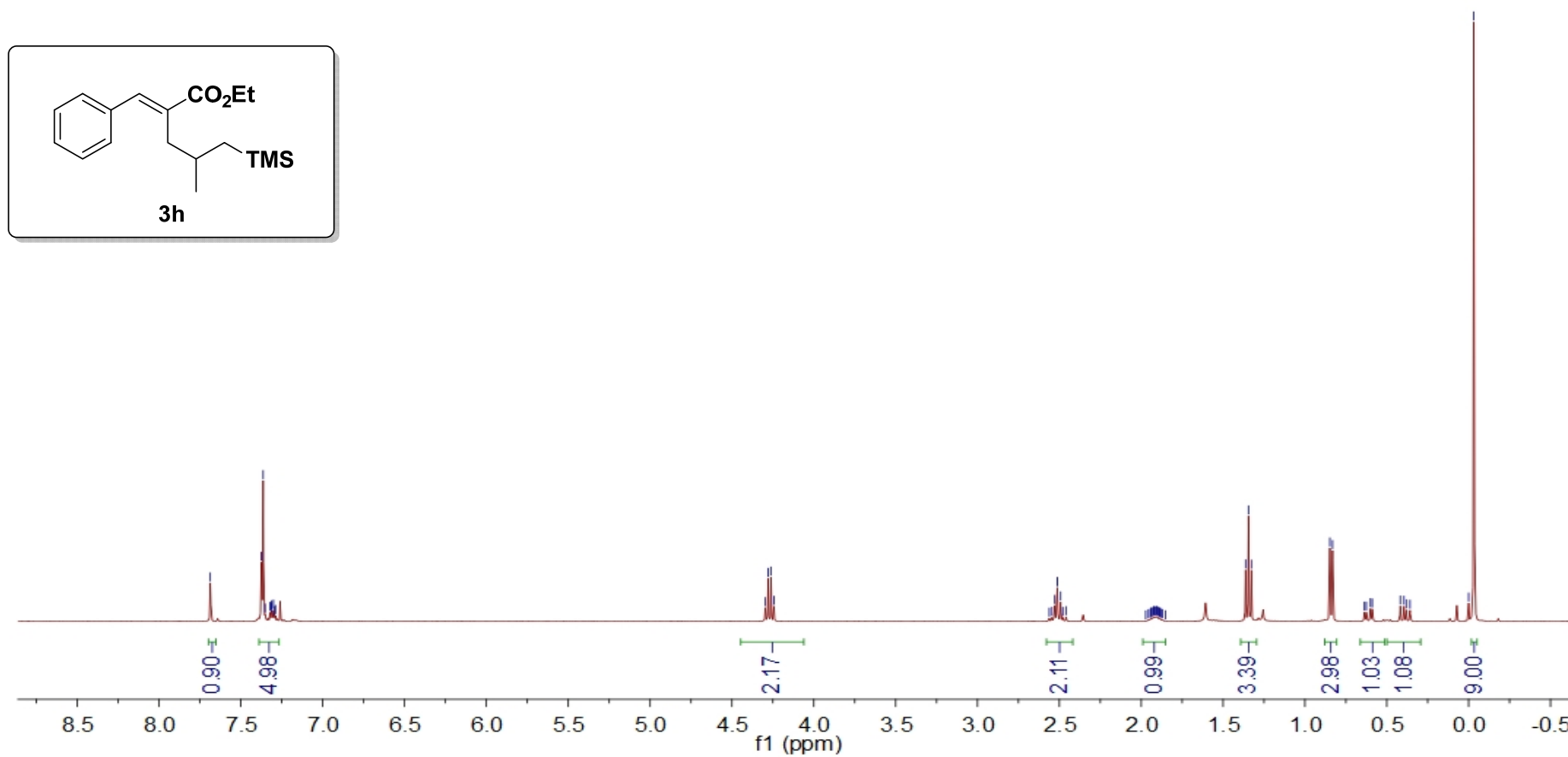


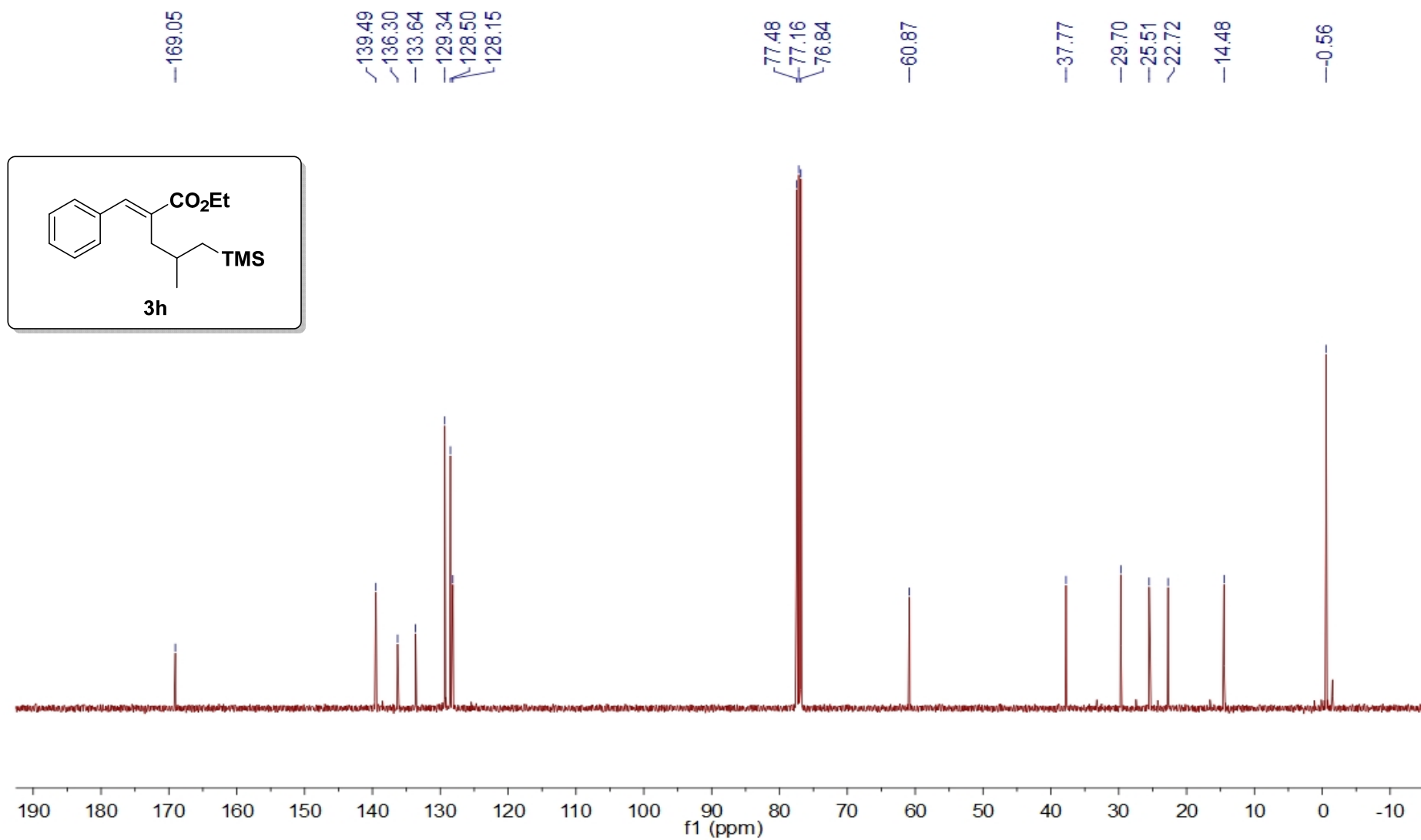


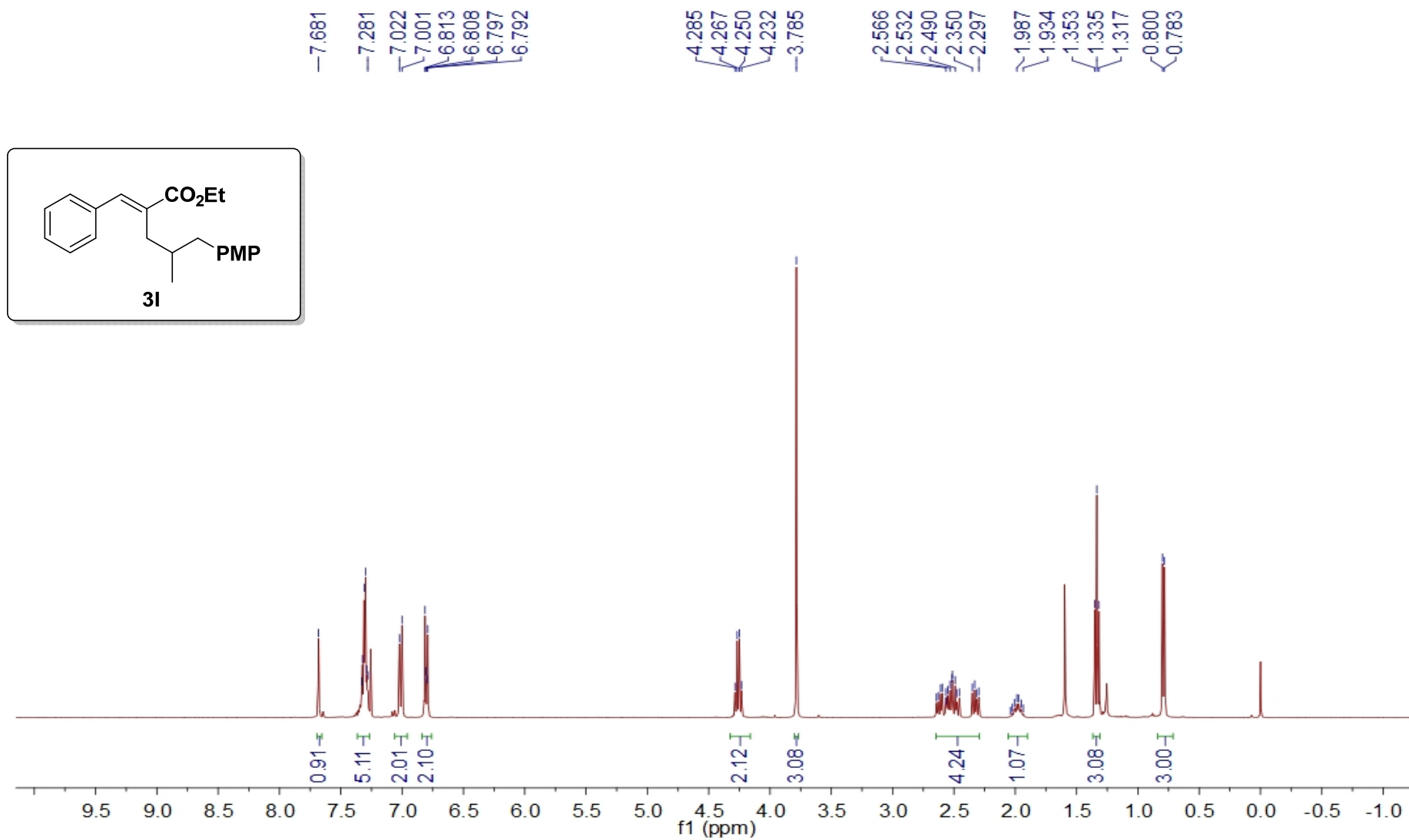


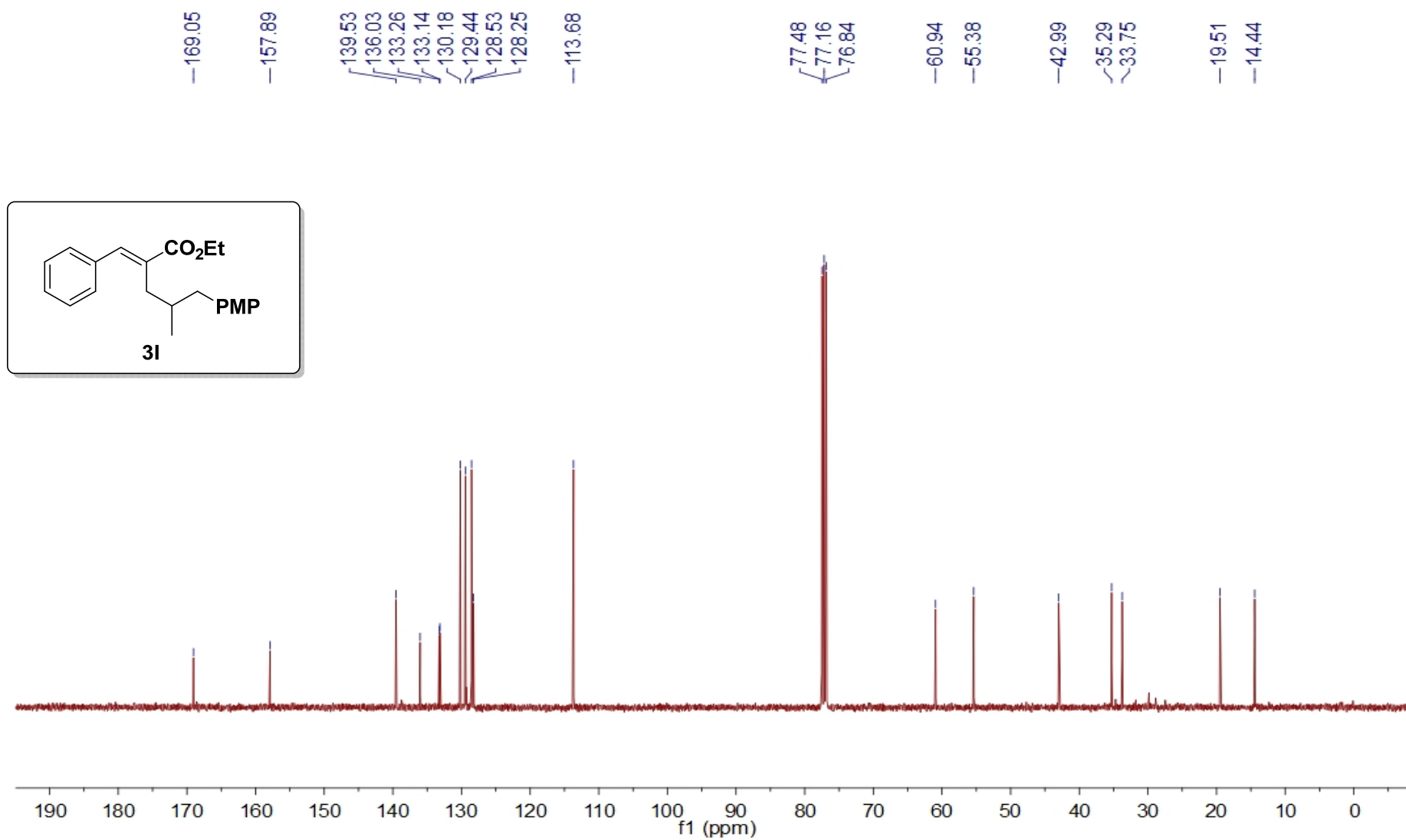


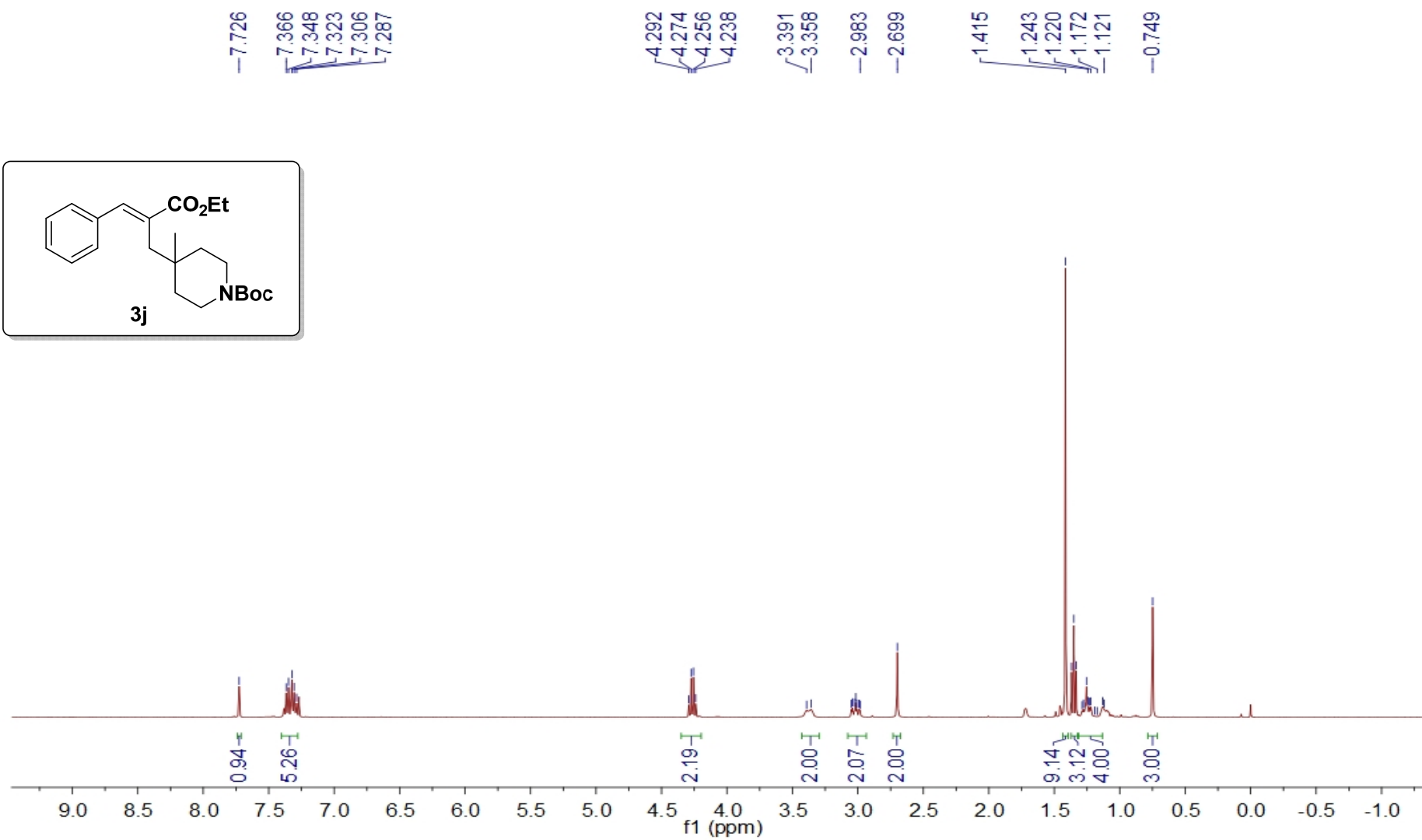
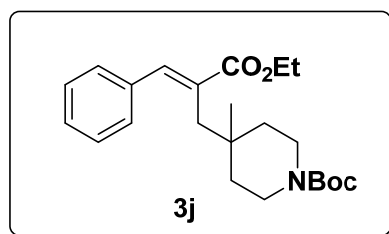


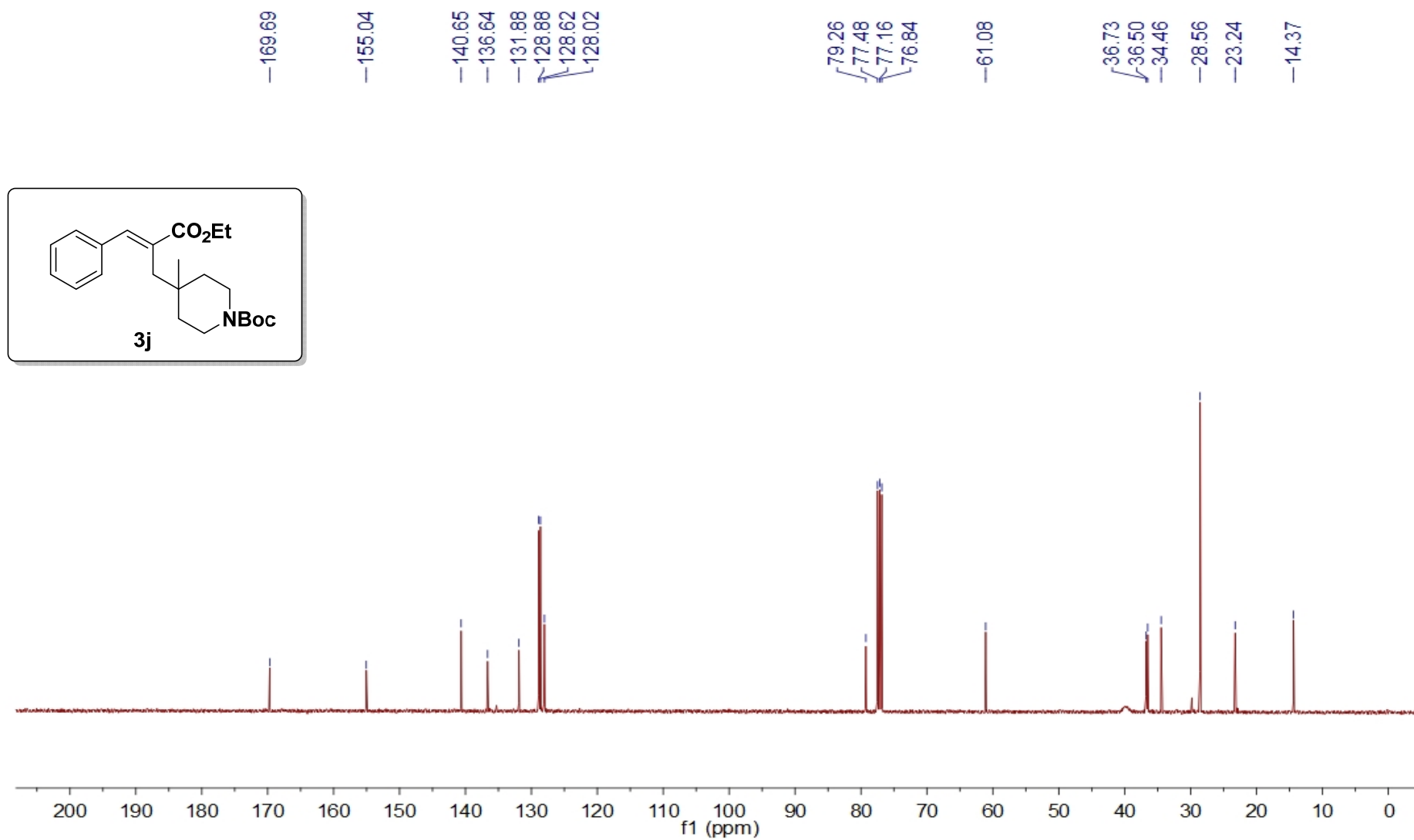


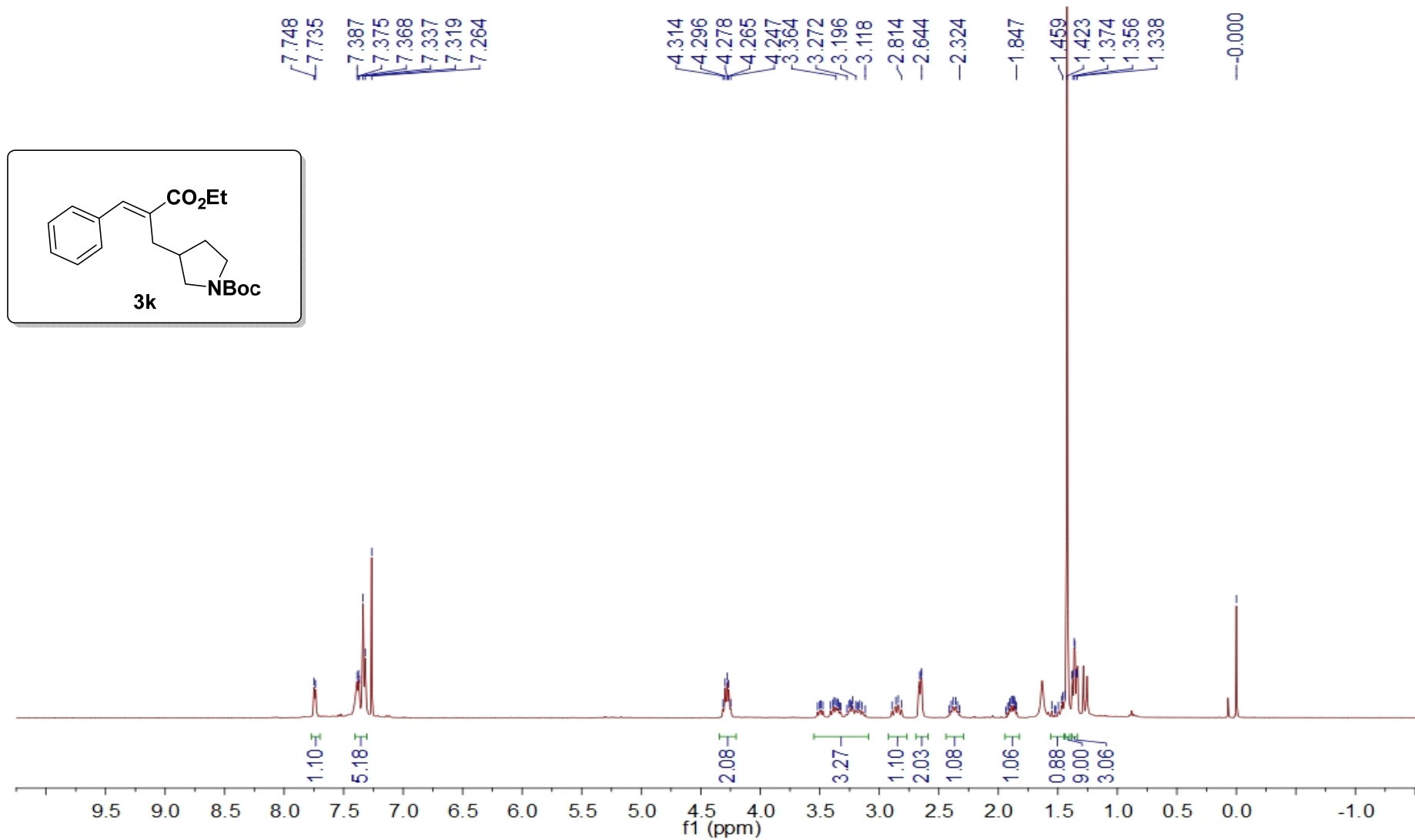


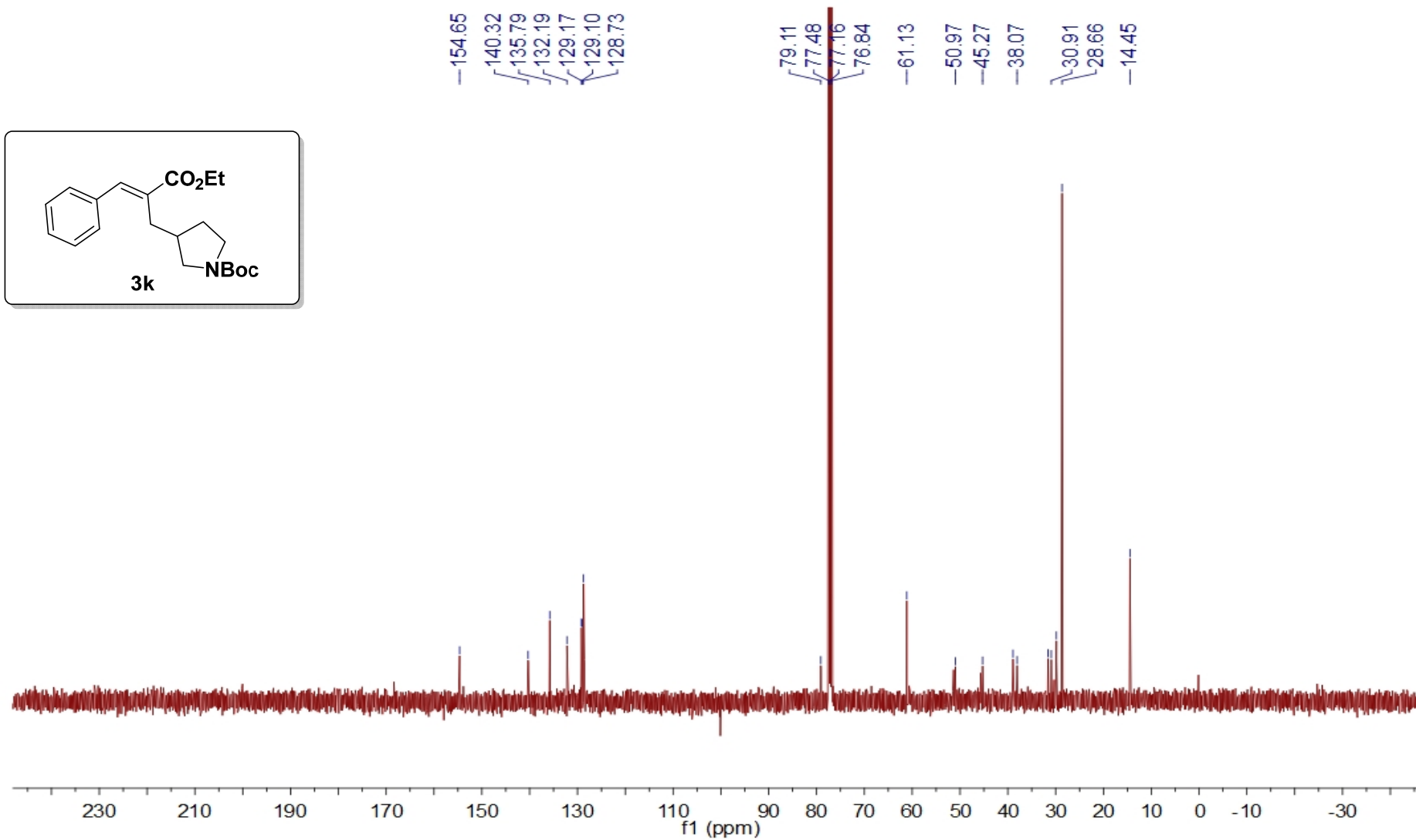
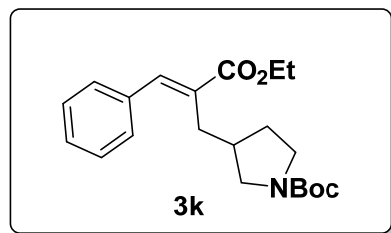


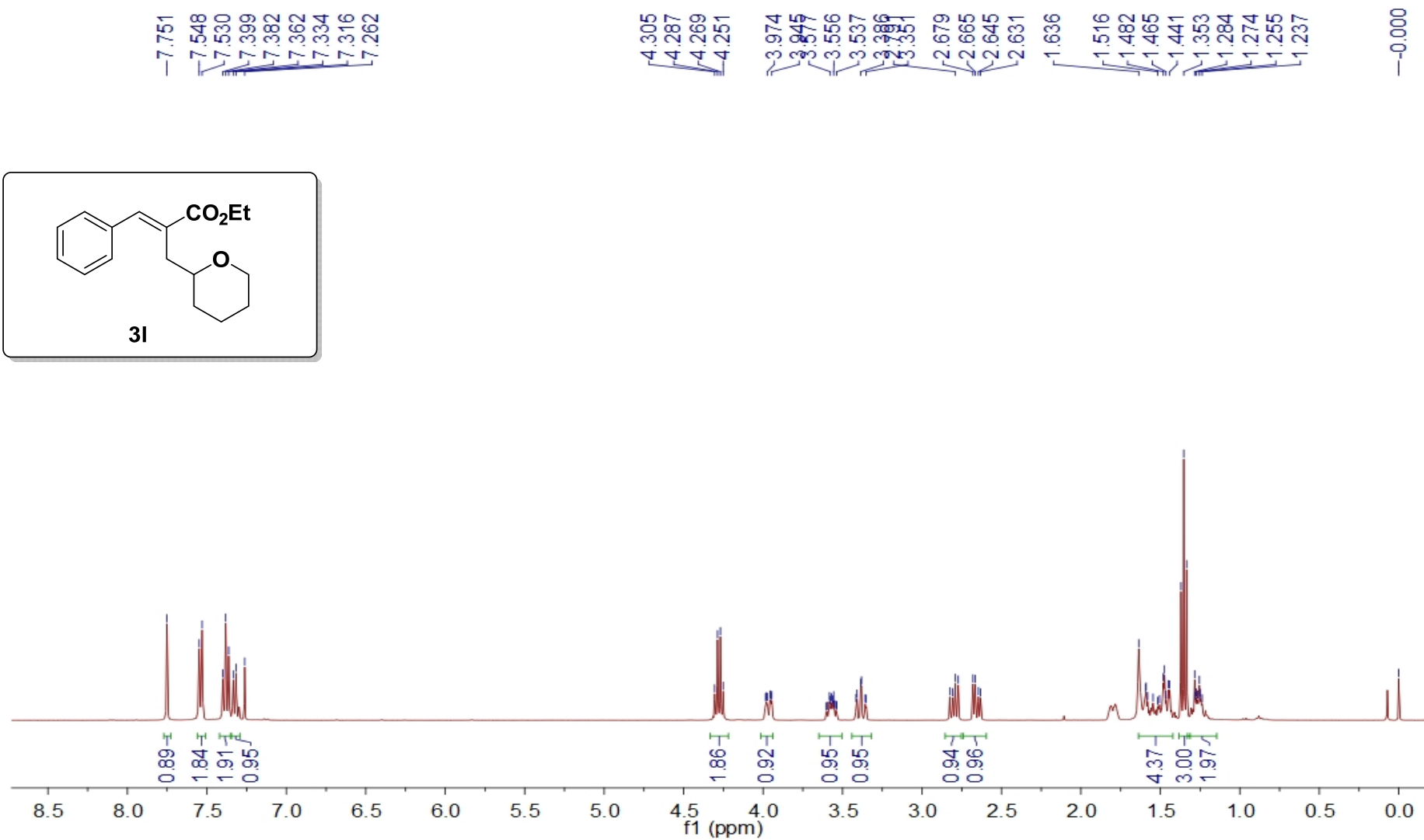
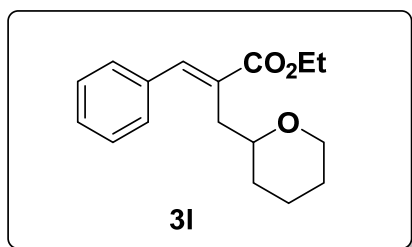


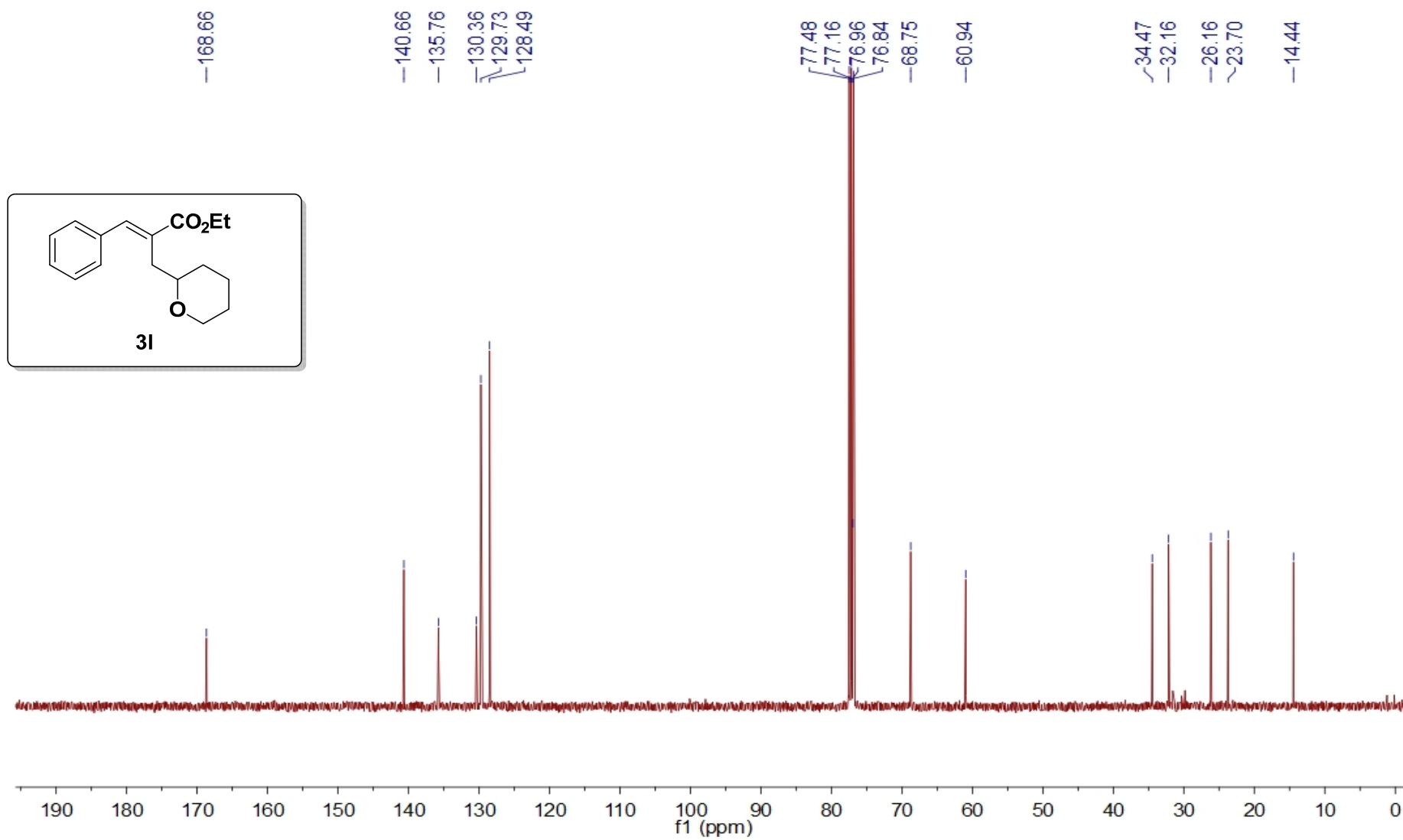


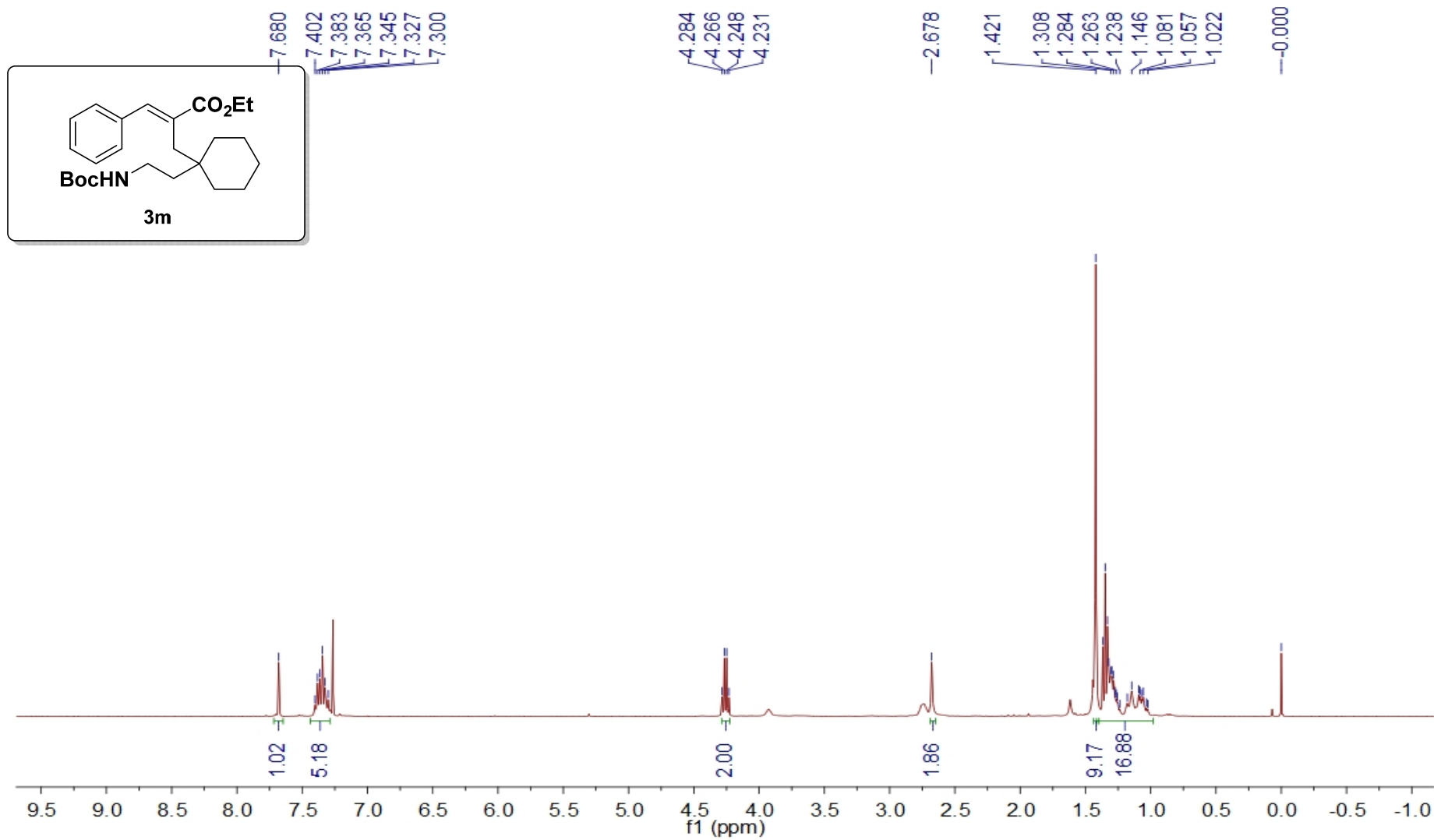


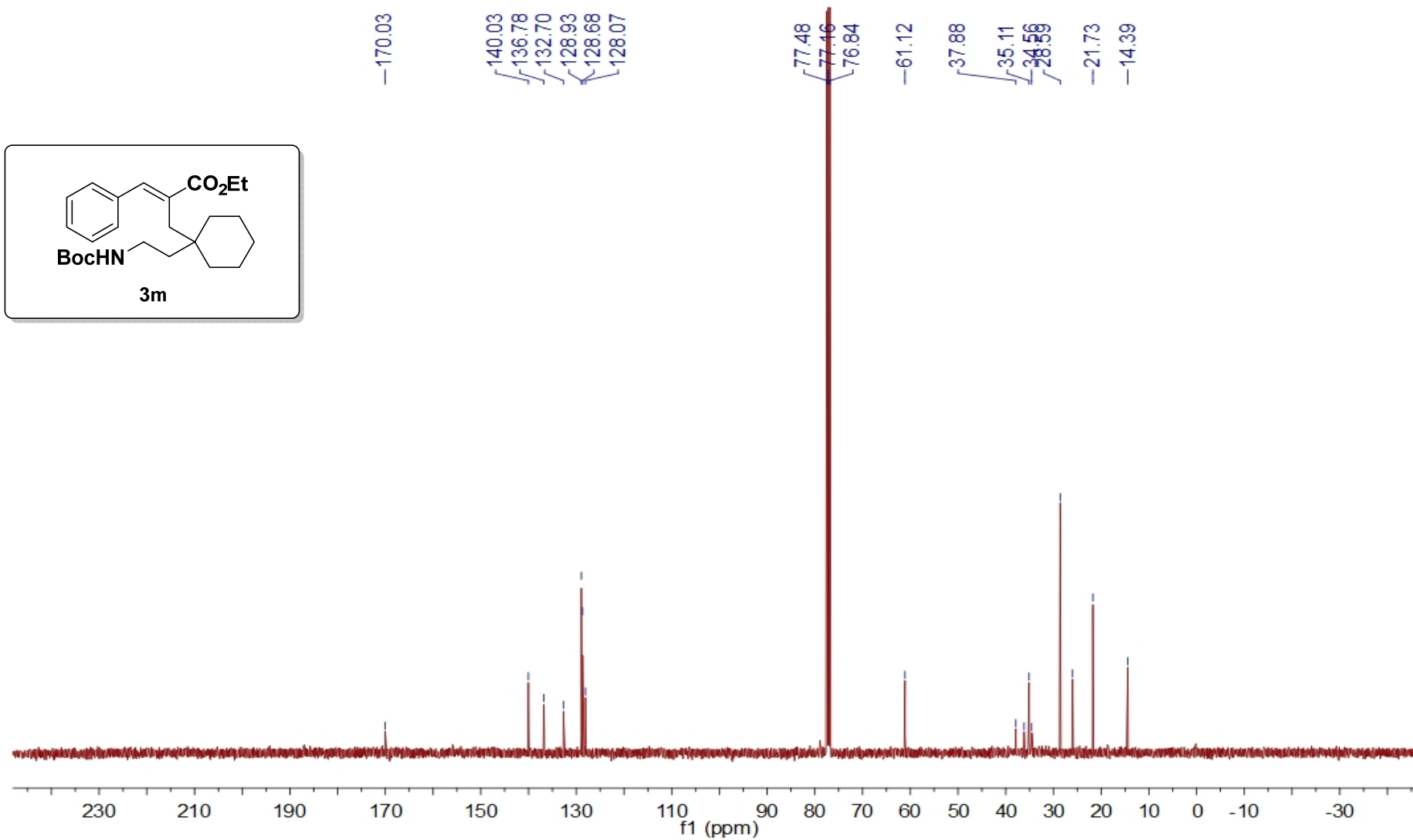
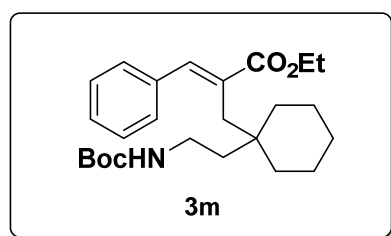


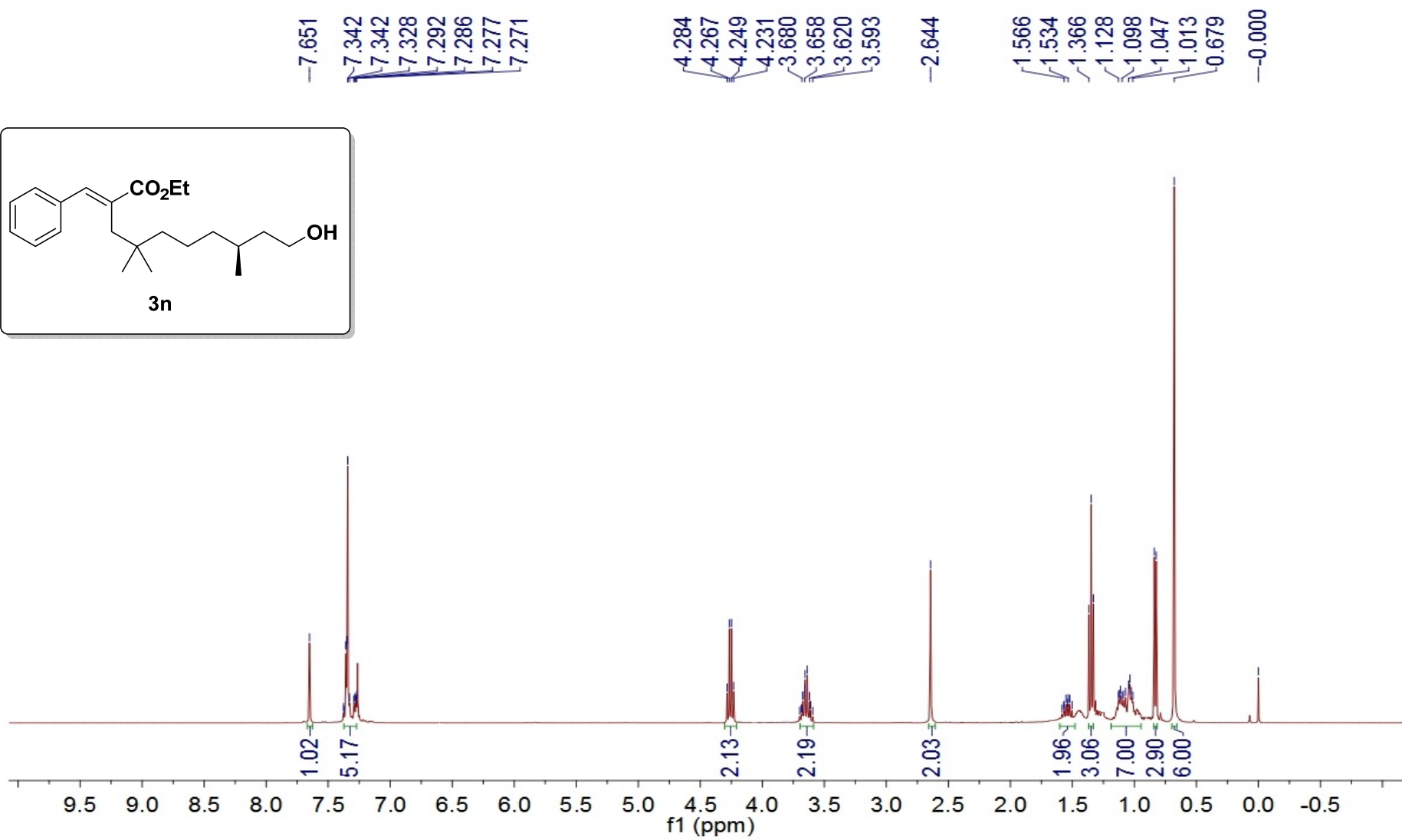
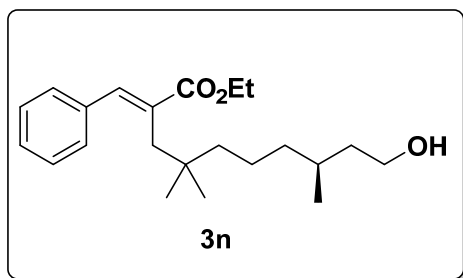


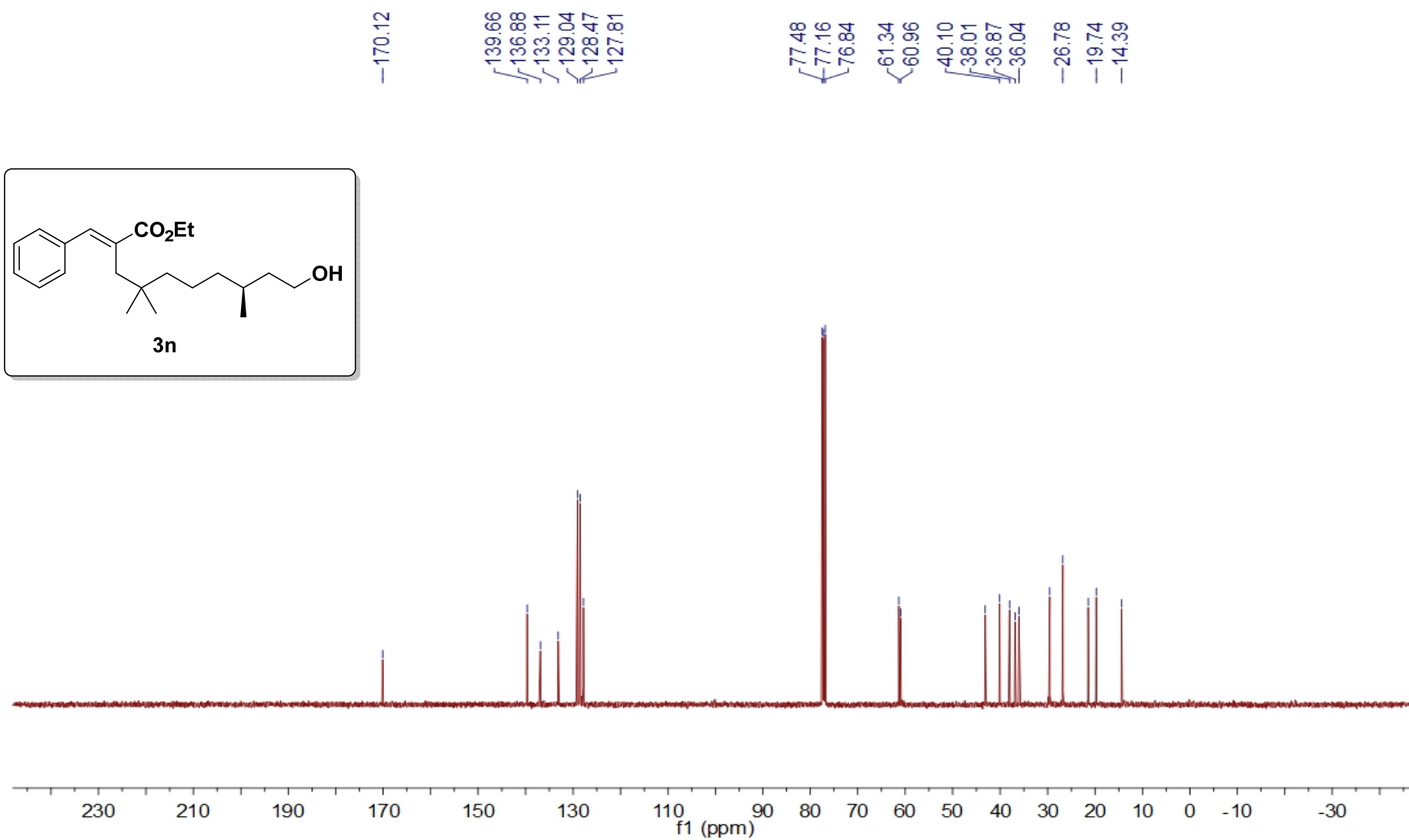
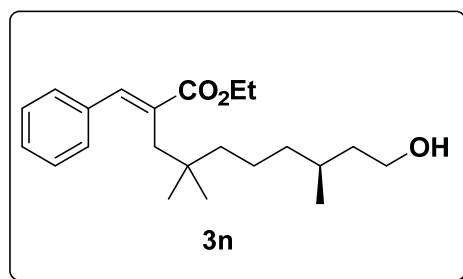


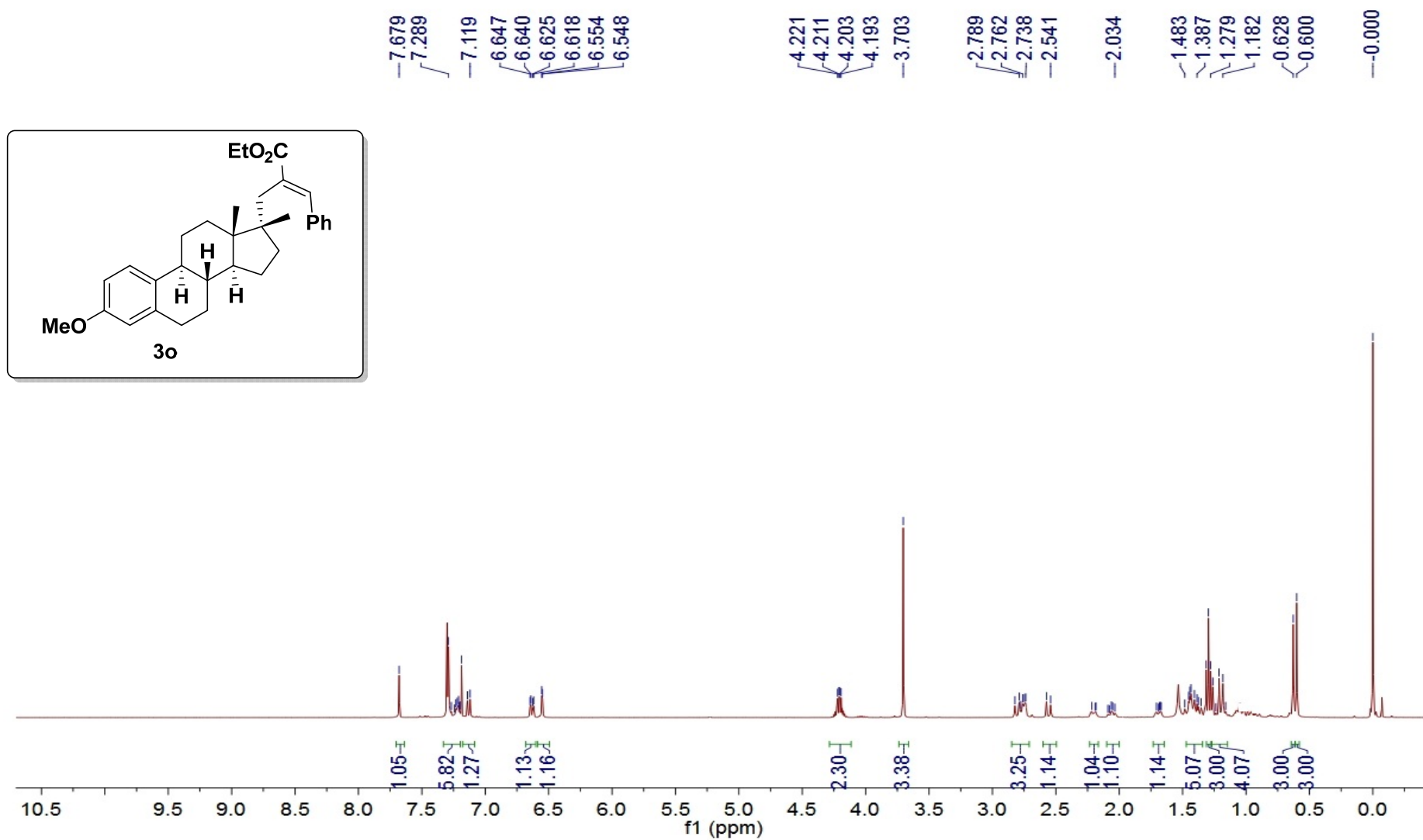


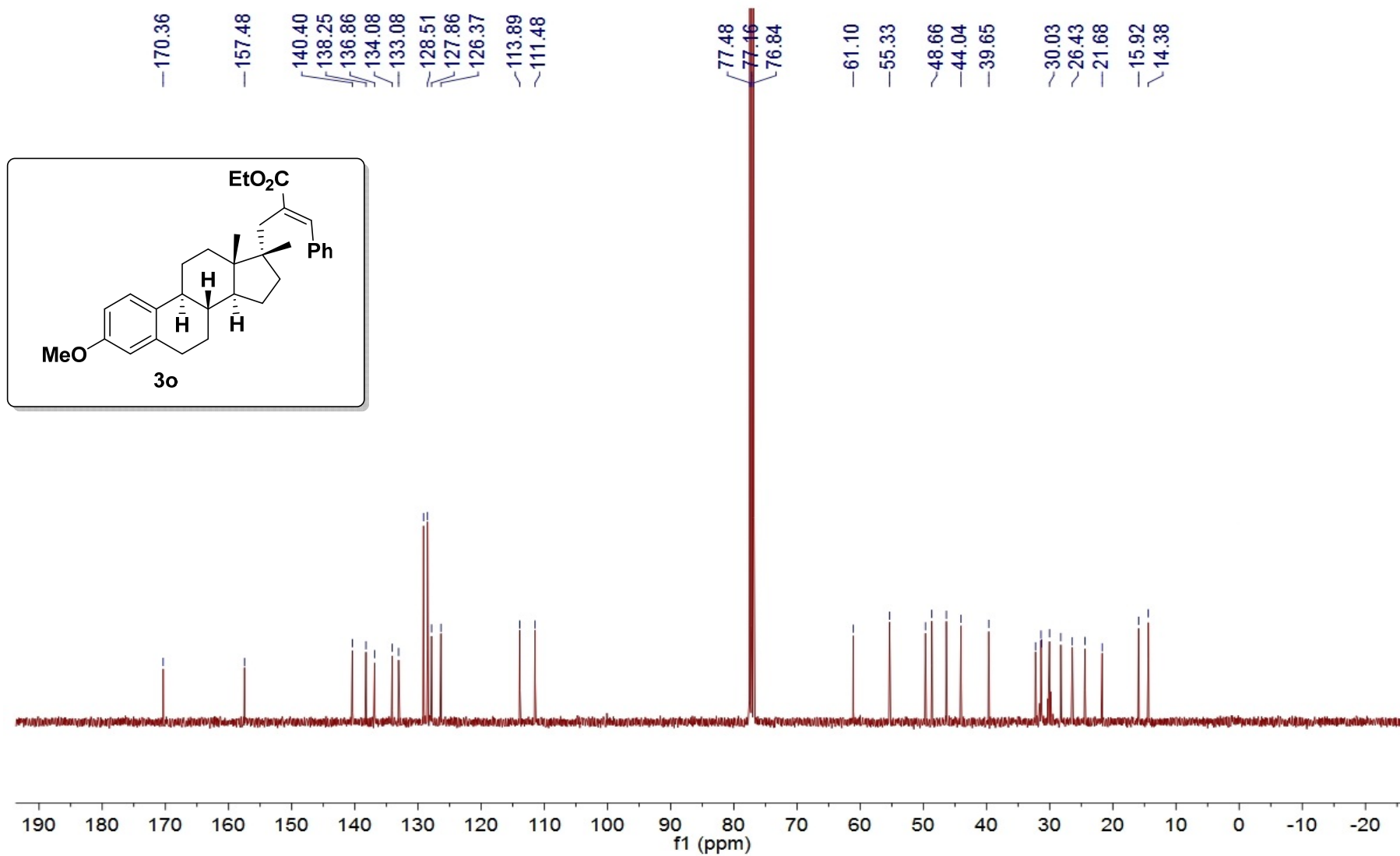


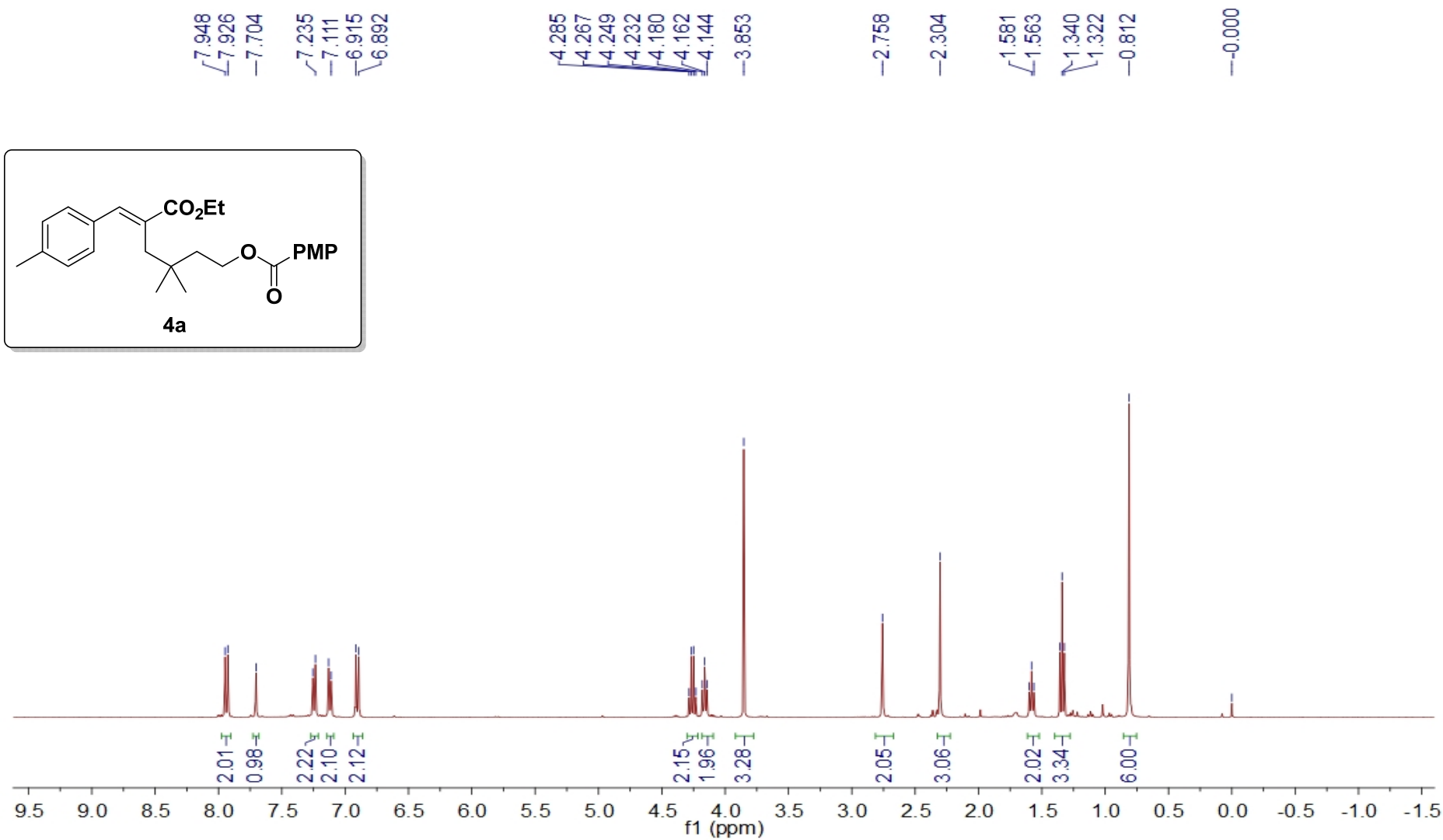
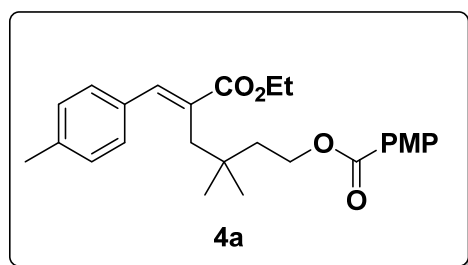


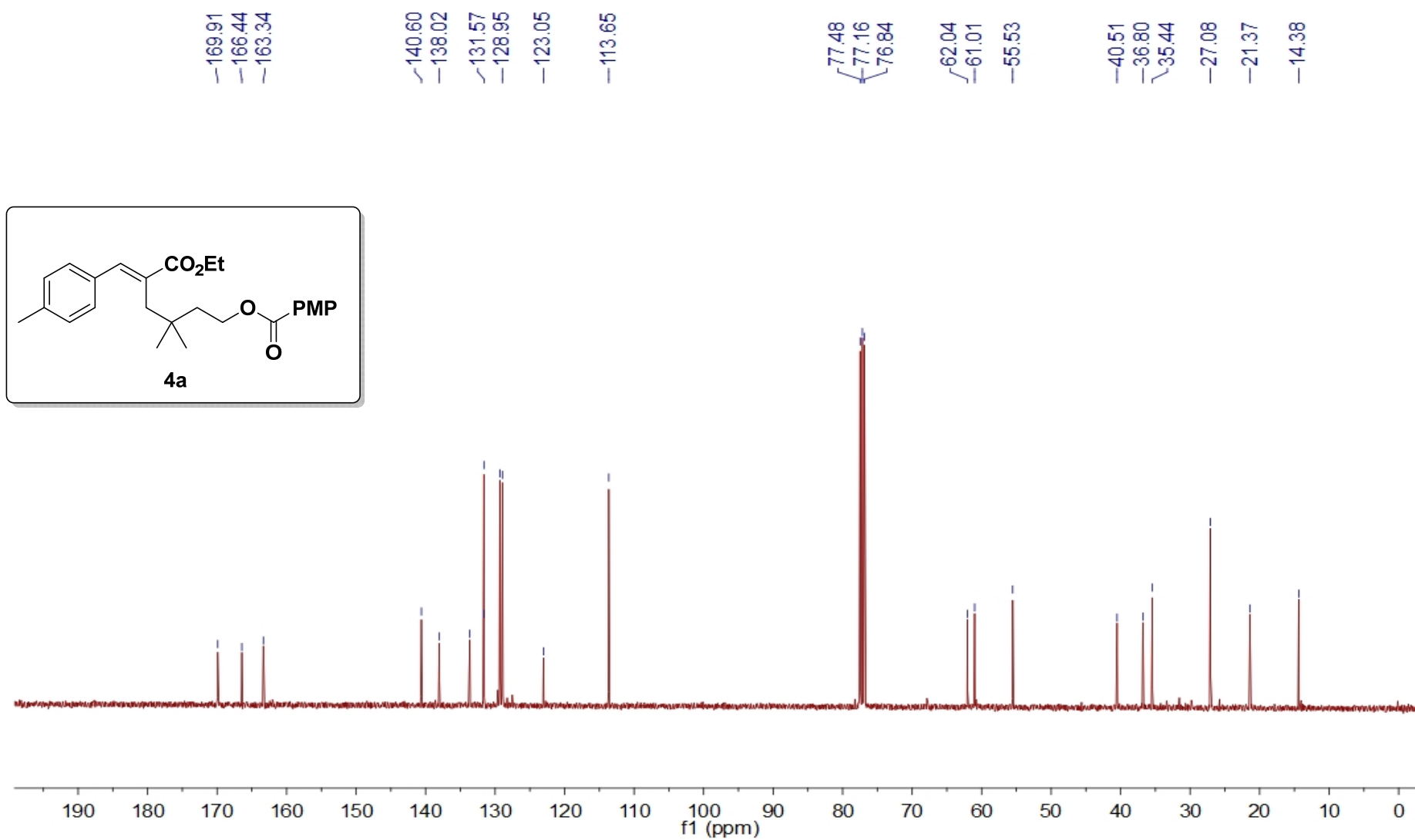


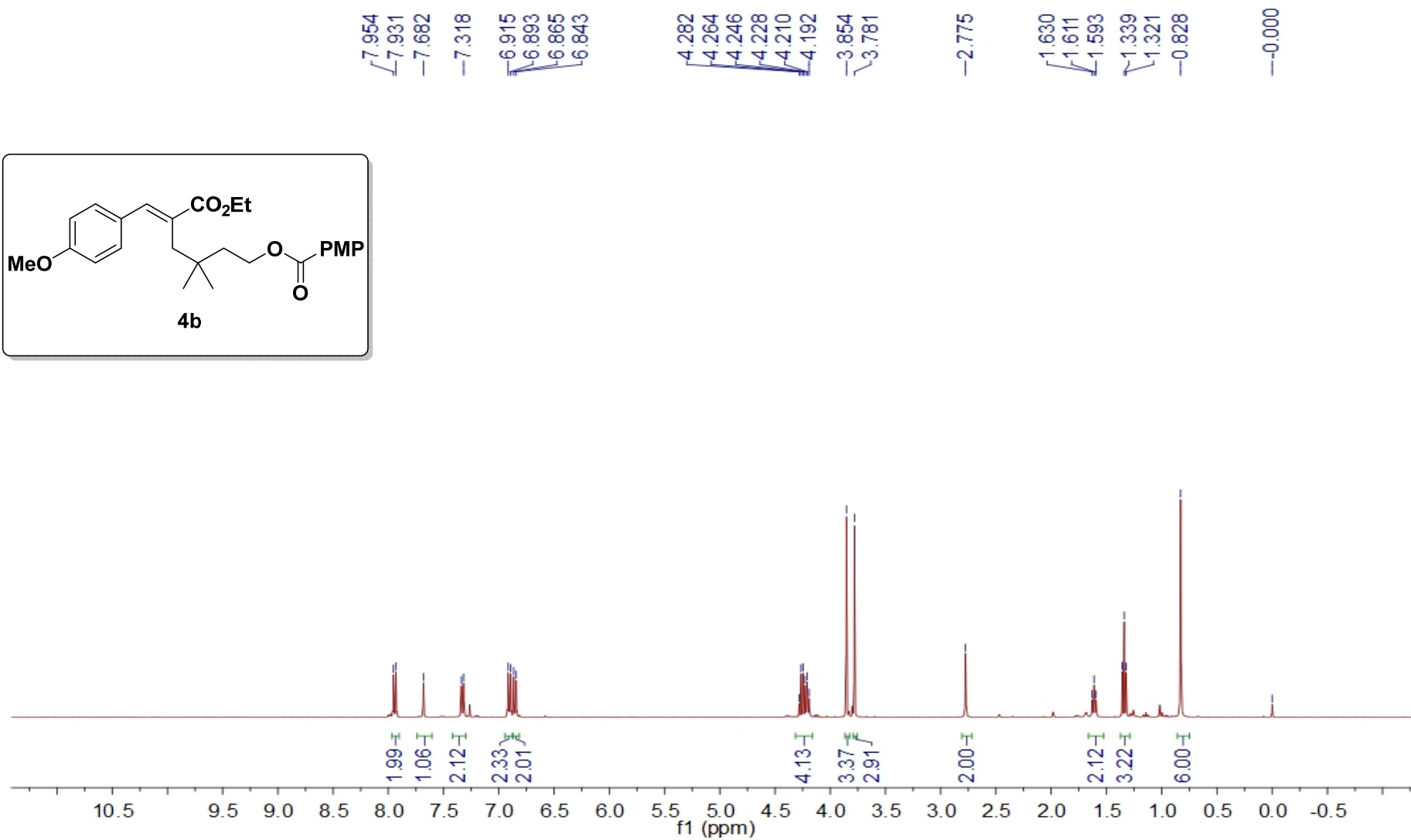
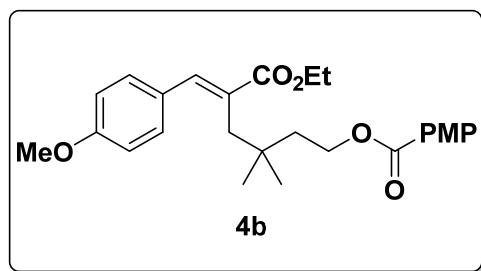


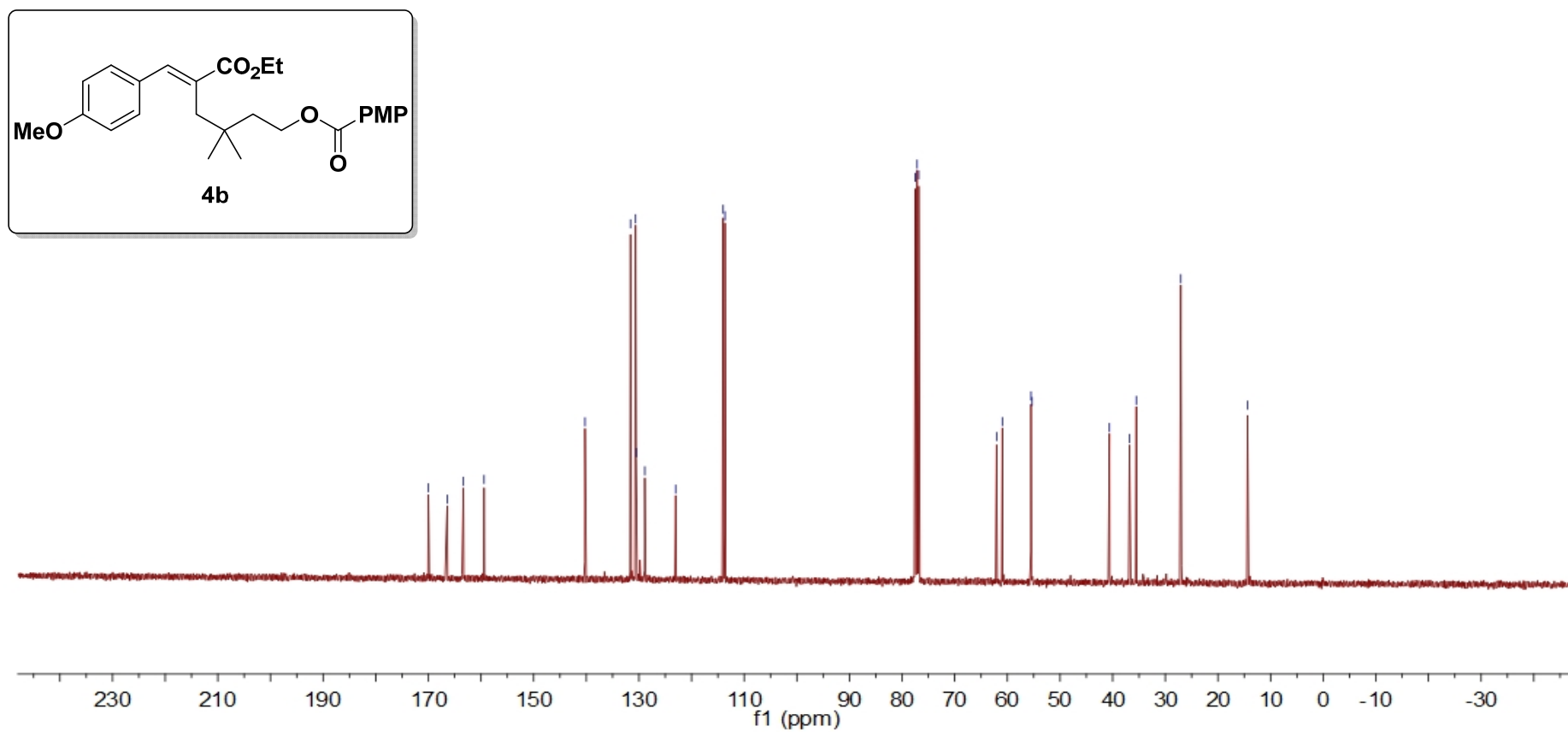


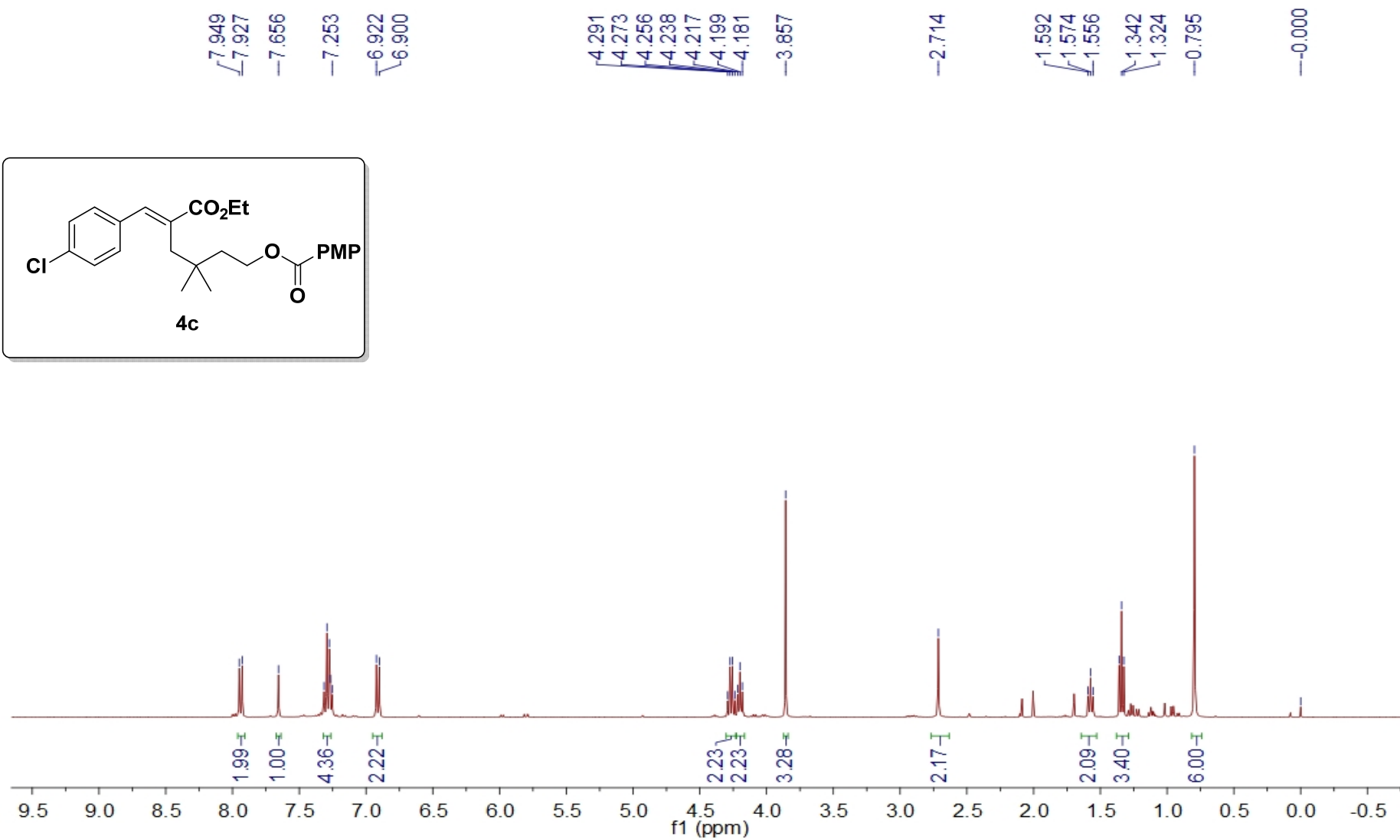
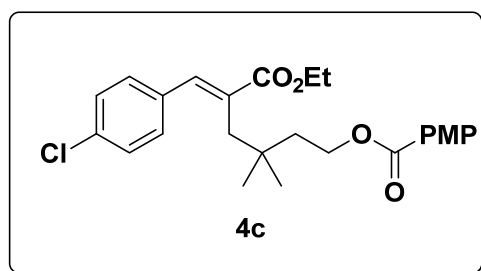


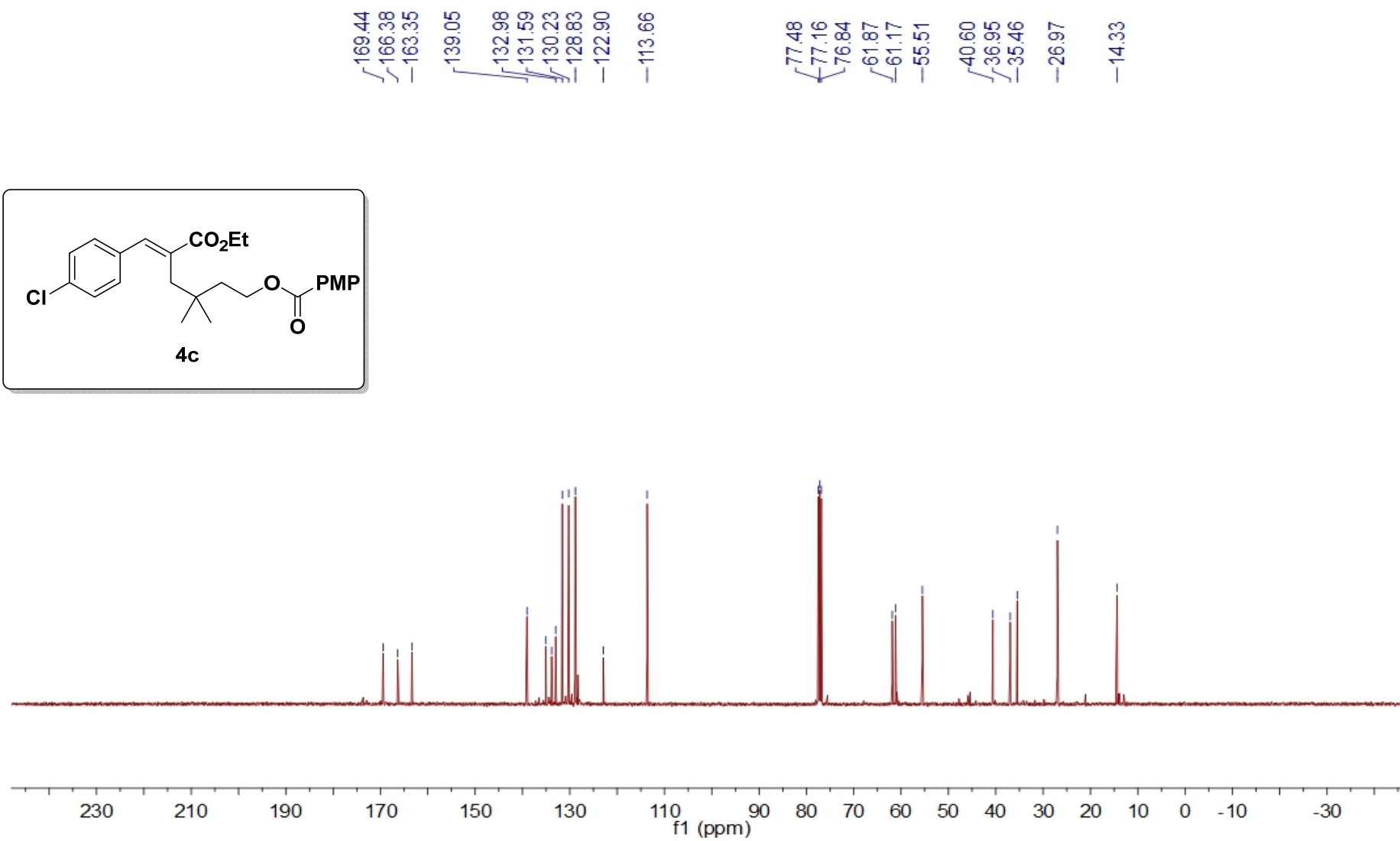
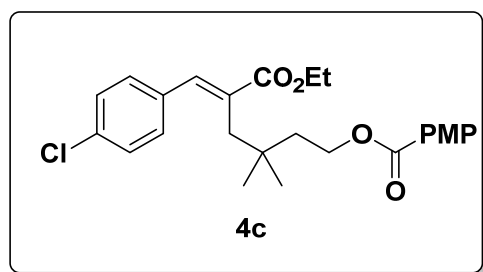


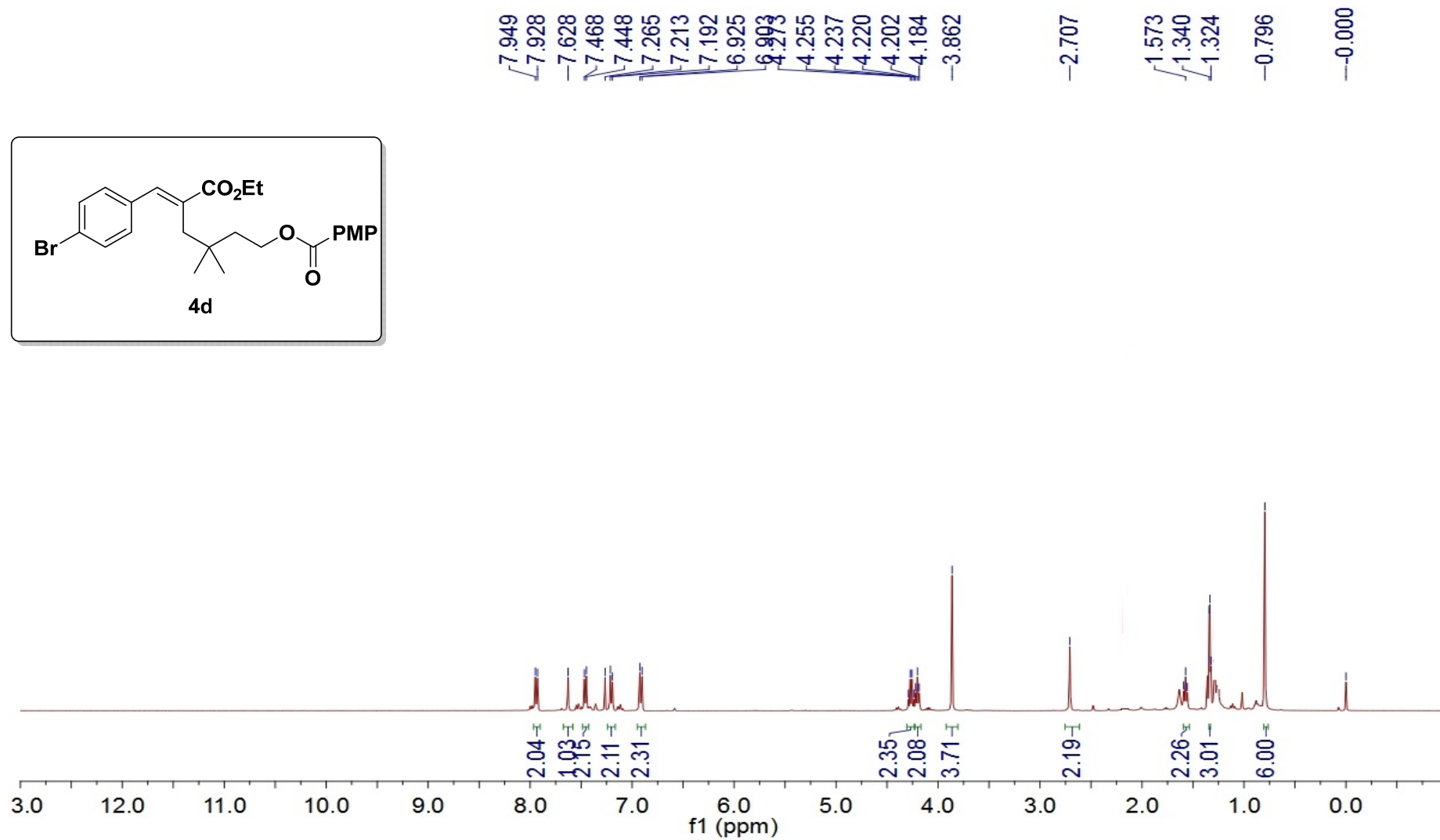
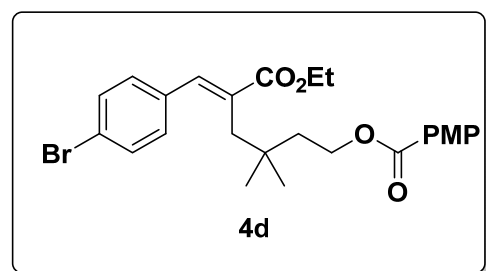


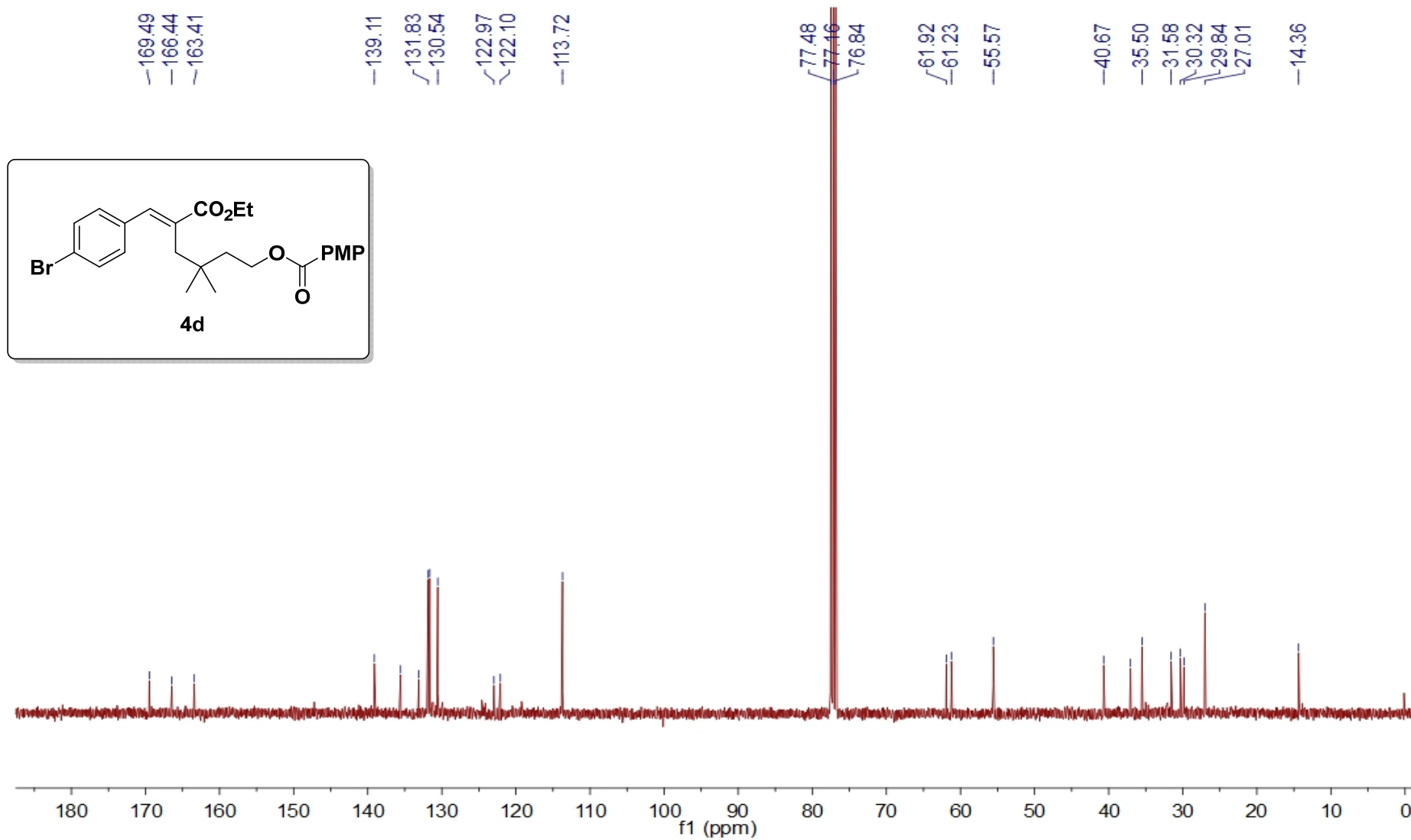


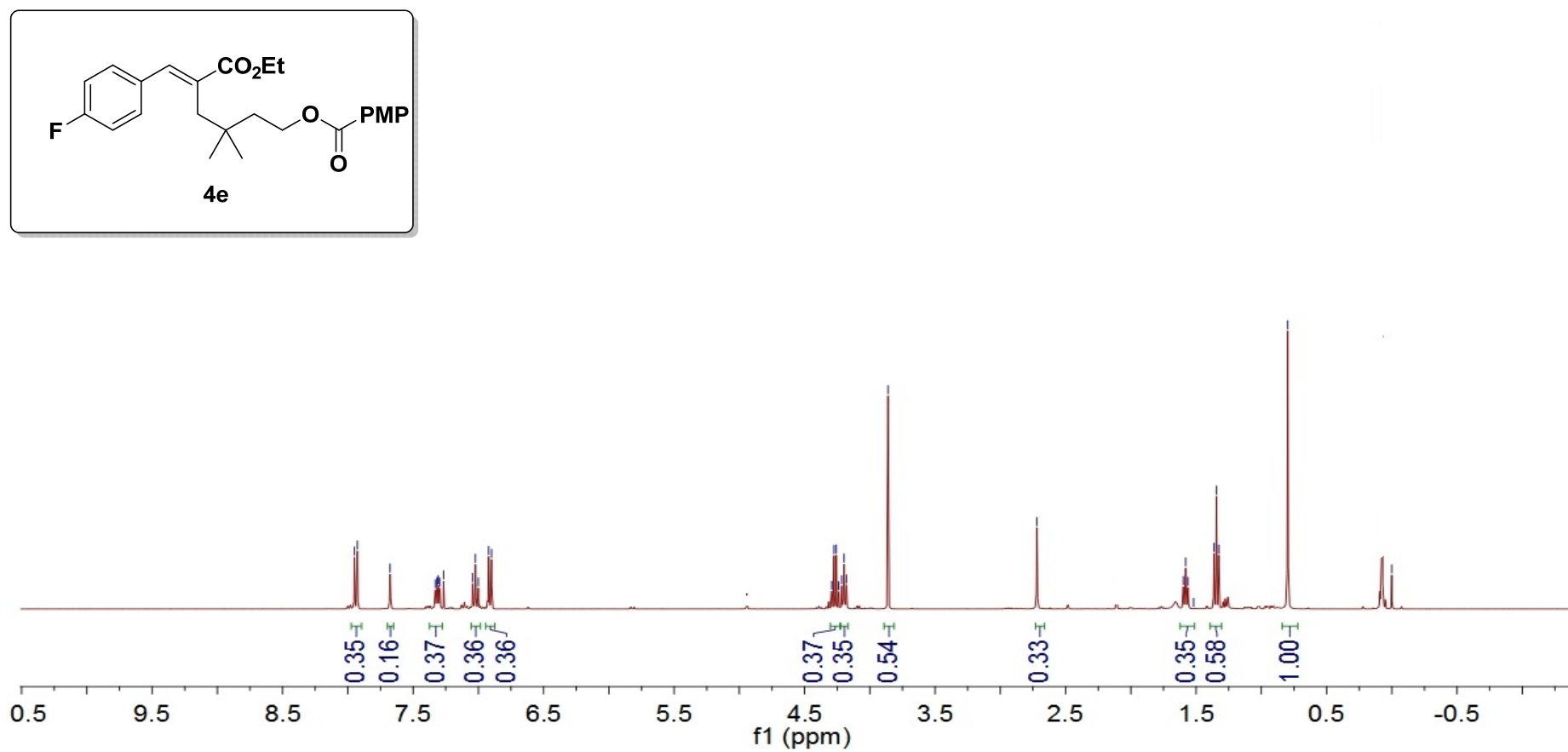


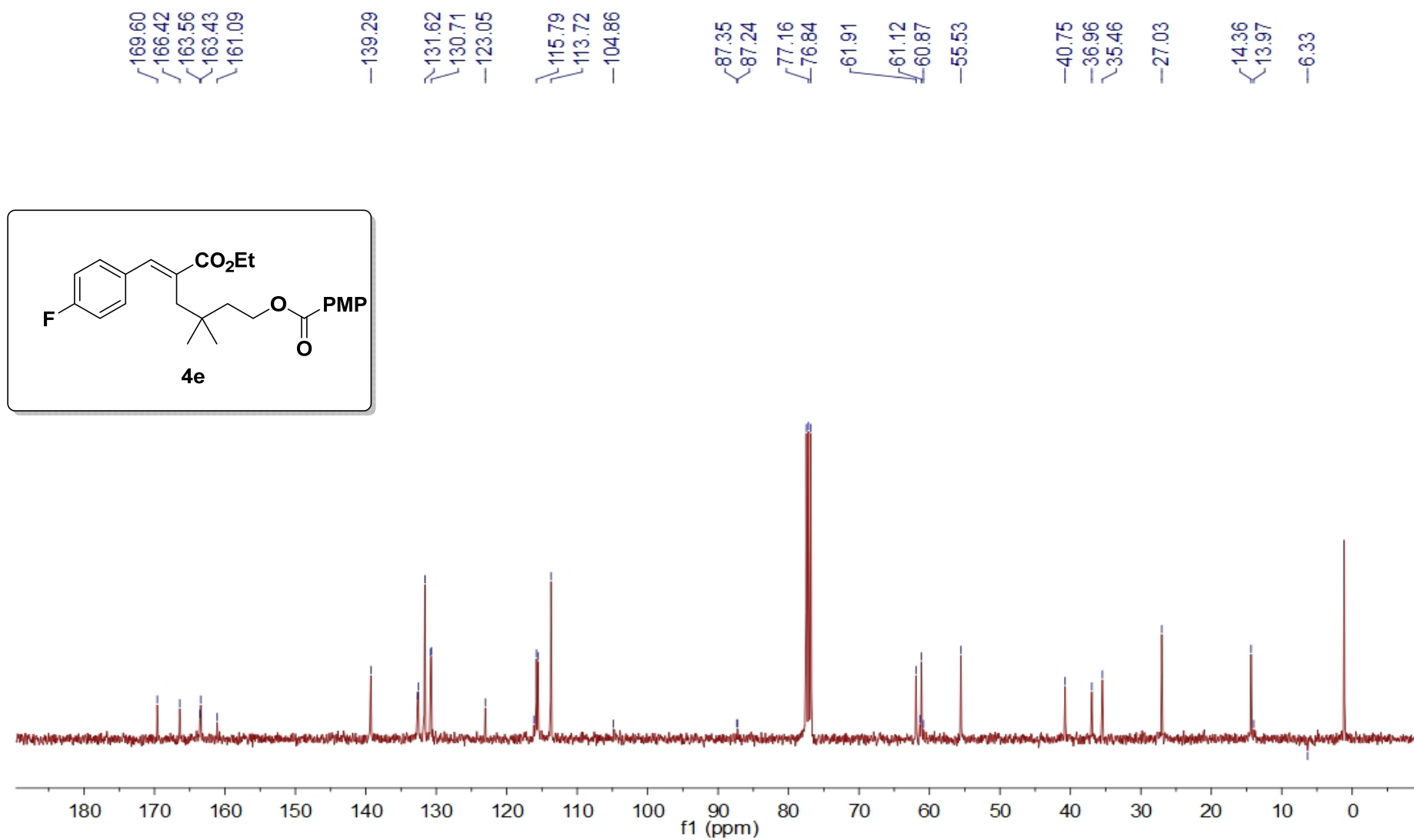


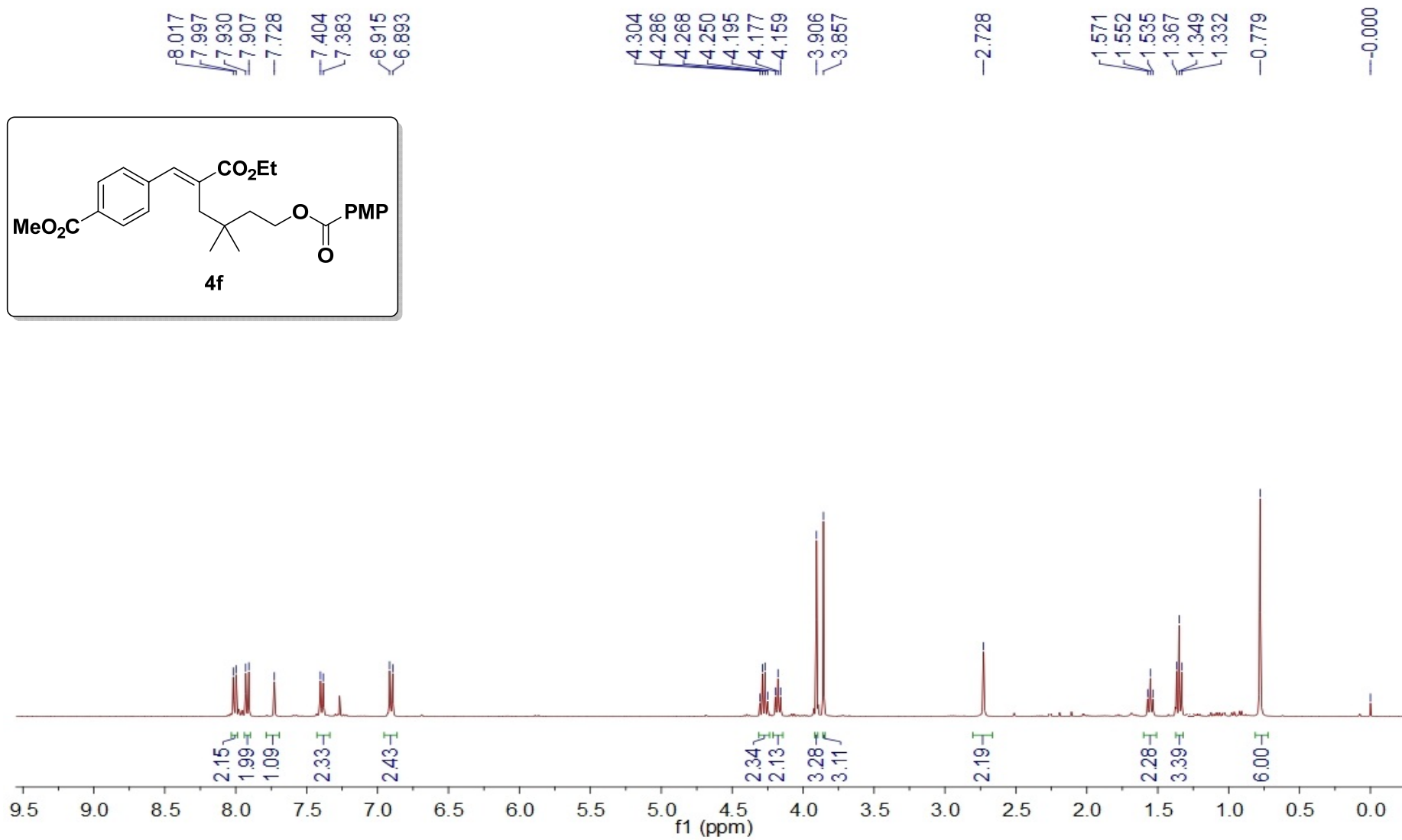


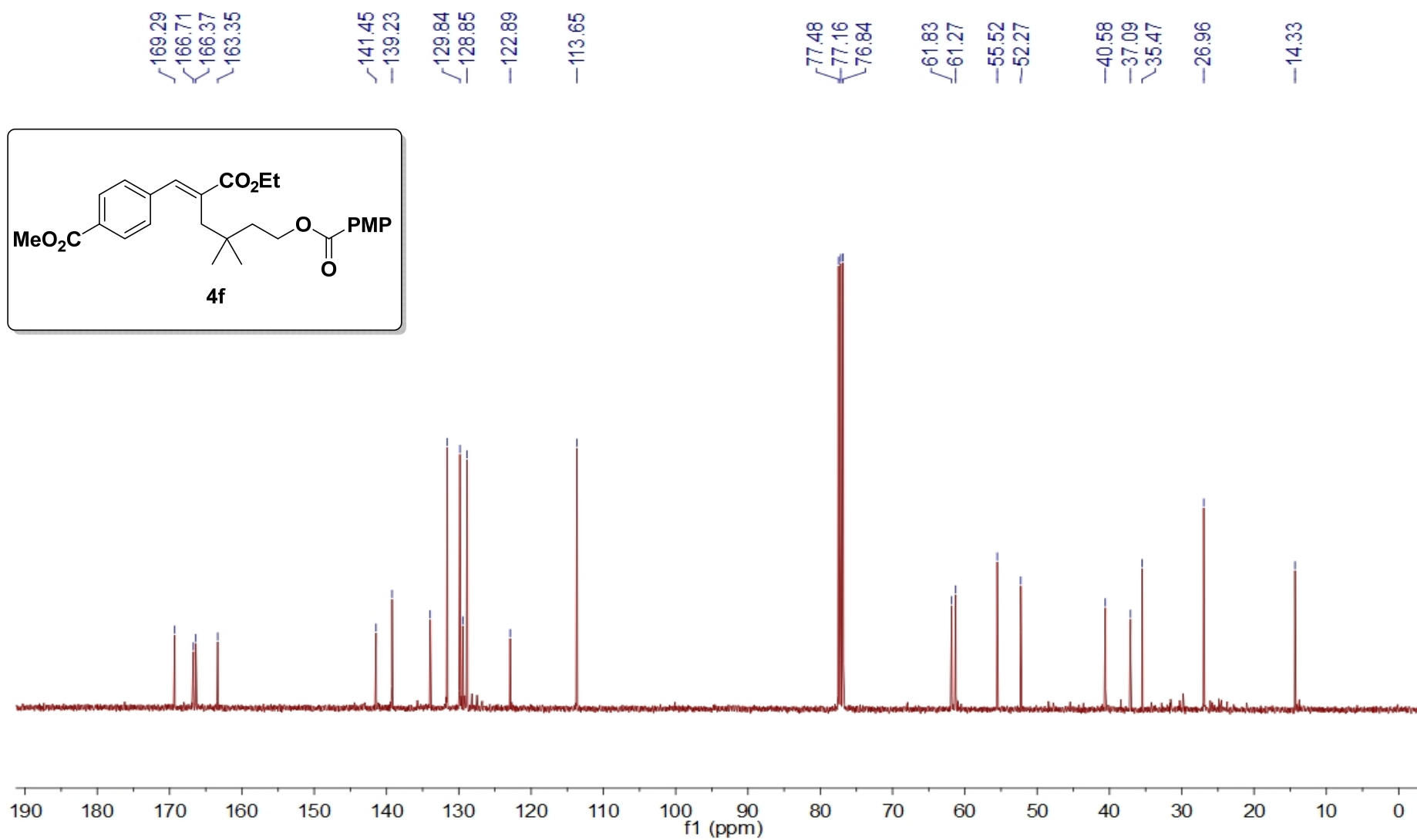


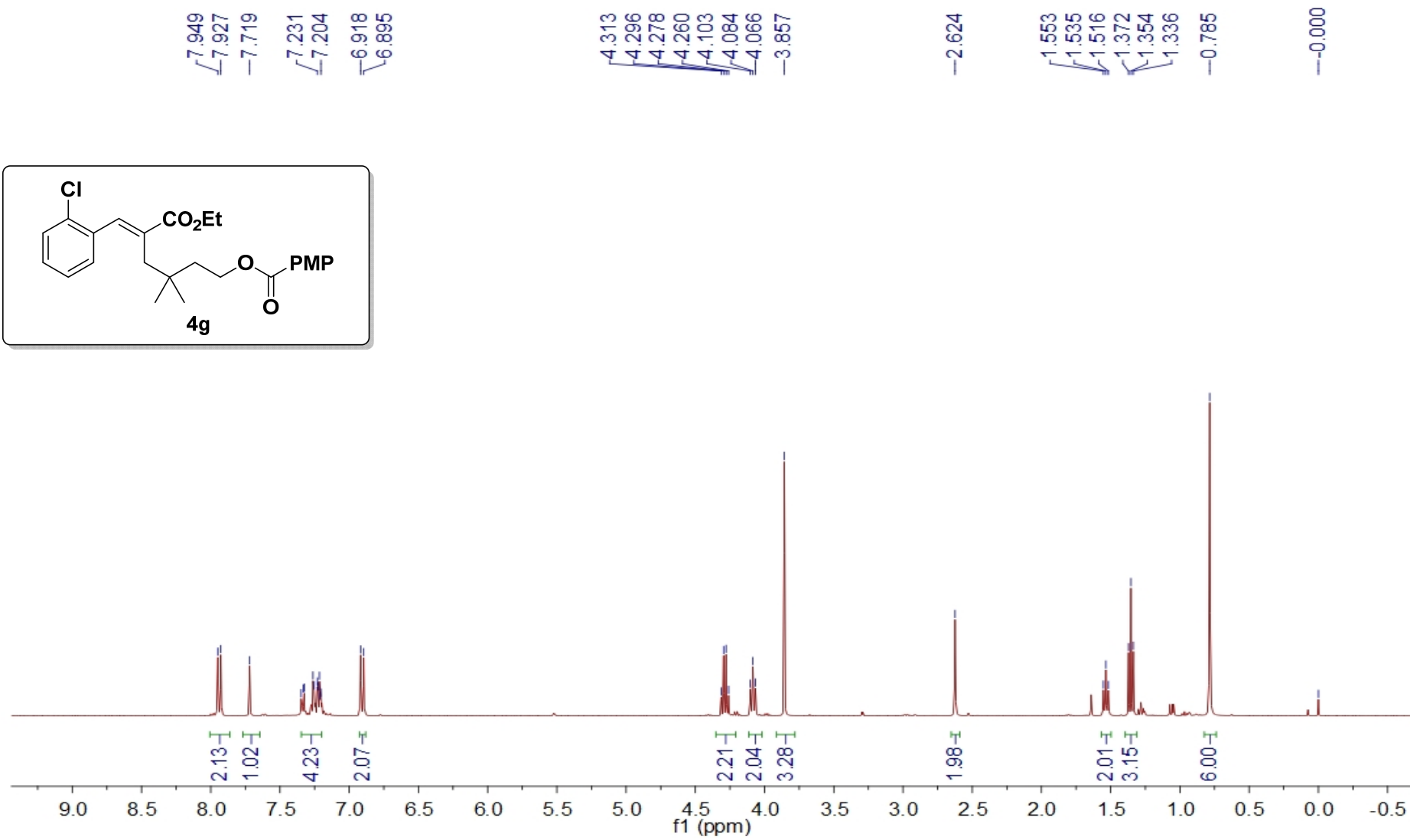
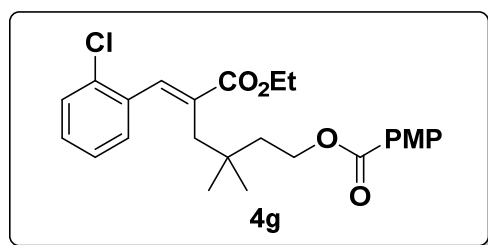


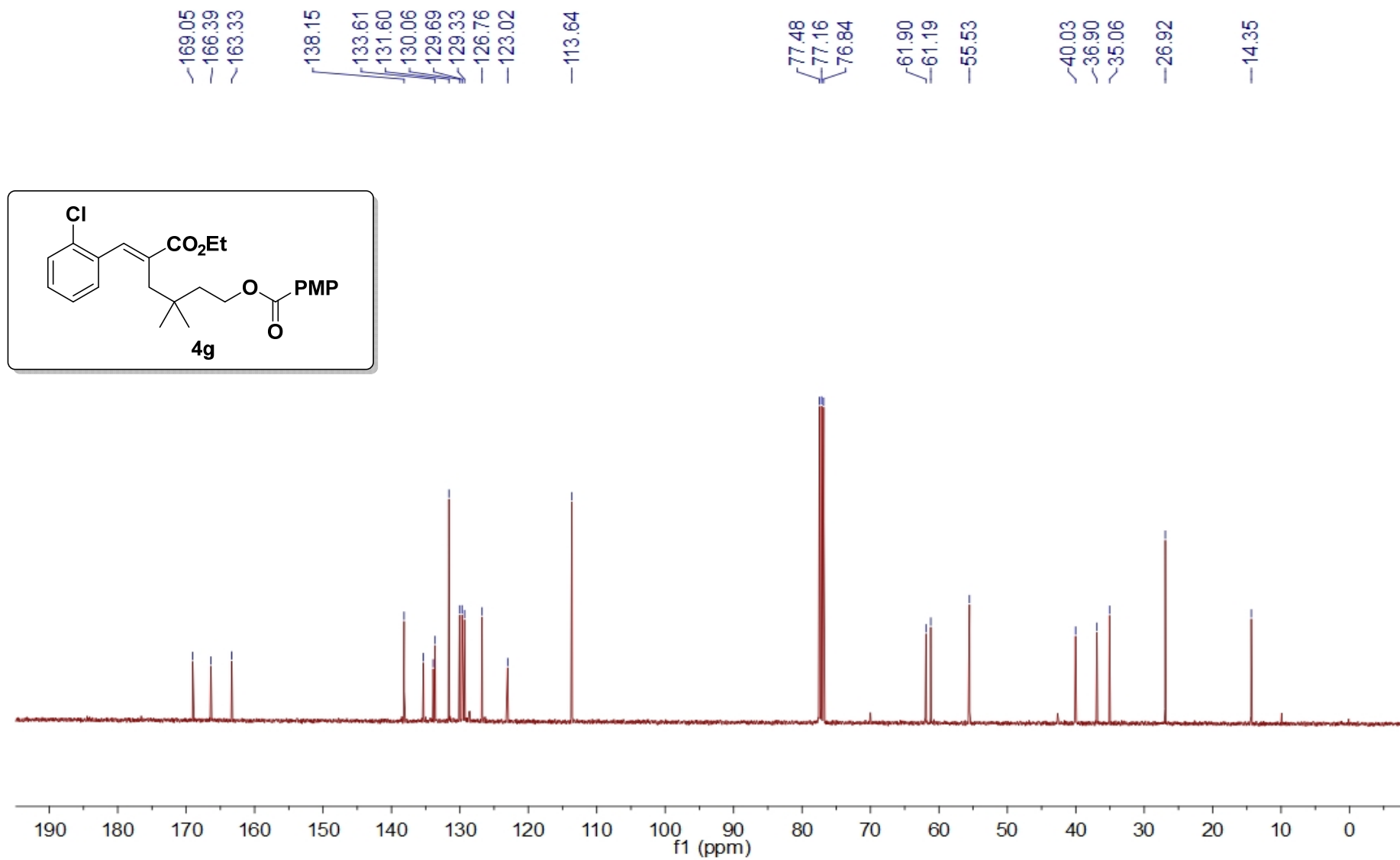












S69

