Electronic Supplementary Material (ESI) for ChemComm.

Zirconocene-catalyzed sequential ethylcarboxylation of alkenes using ethylmagnesium chloride and carbon dioxide†

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Supporting Information

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1. General Comments

All the reactions were carried out using pre-dried screw capped tube with a Teflon-lined septum under N_2 atmosphere. Ethylmagnesium chloride was obtained from Alfa-aesar. All of the solvents were fresh distilled. Column chromatography was performed using silica gel (particle size 10-40 μm, Ocean Chemical Factory of Yantai, China). ^{1}H NMR and ^{13}C NMR spectra were recorded using JEOL AL-300MHz or AL-400MHz spectrometer at ambient temperature with $CDCl_3$ and d^6-DMSO as the solvent. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of $CDCl_3$ (7.26) and d^6-DMSO (2.50), to the carbon resonance of $CDCl_3$ (77.16). Coupling constants (*J*) were given in Hertz (Hz). The term m, t, d, s referred to multiplet, triplet, doublet, and singlet. Mass spectra were obtained on a Bruker Esquire ion trap mass spectrometer in positive mode. The reaction progress was monitored by GC-MS if applicable.

2. Experimental Section

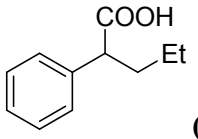
General procedure for the synthesis of product 2

To a solution of Zirconocene dichloride (0.1 mmol) in THF (2 mL) was added EtMgCl (2.7 M THF solution, 3 mmol) at -78 ^{o}C. The solution was stirred at room temperature for 1 h, followed by the addition of styrene **1** (1 mmol) via syringe at -78 ^{o}C. The mixture was stirred for 1 h at -78 ^{o}C, after which, it was warmed to room temperature and was stirred for 24 h. Then the reaction mixture was bubbled with CO_2 for 30 minutes at room temperature. After completion, then H_2O (2 mL) was added to quench the reaction and the mixture was acidified till pH = 3~4 with HCl (2 M). Then the mixture was extracted with ethyl acetate (5 mL x 3), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo, followed by purification on silica gel (petroleum ether/ethyl acetate = 5/1) to give the corresponding product **2**.

General procedure for the synthesis of product 3

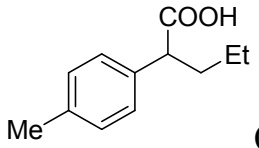
To a solution of Zirconocene dichloride (0.1 mmol) in THF (2 mL) was added EtMgCl (2.7 M THF solution, 3 mmol) at -78 ^{o}C. The solution was stirred at room temperature for 1 h, followed by the addition of styrene **1** (1 mmol) via syringe at -78 ^{o}C. The mixture was stirred for 1 h at -78 ^{o}C, after which, it was warmed to room temperature and was stirred for 24 h. Then the reaction mixture was bubbled with CO_2 for 30 minutes at room temperature. After that, CuCl (3 mmol) was added and stirred at room temperature for 1 h, followed by the addition of allyl bromide (4 mmol). The mixture was stirred for further 3 h at rt. Then H_2O (2 mL) was added to quench the reaction and the mixture was extracted with ethyl acetate (5 mL x 3), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo, followed by purification on silica gel (petroleum ether/ethyl acetate = 50/1) to give the corresponding product **3**.

3. NMR Data of Product



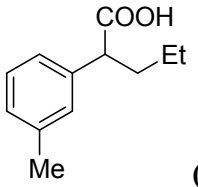
(2a)

2-Phenylpentanoic acid (2a): Colorless oil, 124.6 mg, Yield: 70%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.30 (s, 1H), 7.22-7.34 (m, 5H), 3.50 (t, *J* = 8 Hz, 1H), 1.88-1.97 (m, 1H), 1.59-1.65 (m, 1 H), 1.15-1.27 (m, 2 H), 0.86 (t, *J* = 8 Hz, 3H); ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 14.2, 20.8, 35.7, 51.1, 127.4, 128.3, 128.9, 140.4, 175.4; IR (neat) ν_{max} cm^{-1} 3027, 2957, 2930, 2872, 1713, 1601, 1495, 1454, 1200, 1178; GC-MS m/z: 178. HRMS (ESI^{-}) calcd for $C_{11}H_{13}O_2^-$ 177.0916; found: 177.0911.



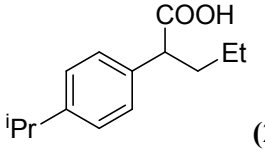
(2b)

2-(*p*-Tolyl)pentanoic acid (2b): Colorless oil, 144.0 mg, Yield: 75%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.22 (s, 1H), 7.16 (d, *J* = 8 Hz, 2H), 7.11 (d, *J* = 8 Hz, 2H), 3.44 (t, *J* = 8 Hz, 1H), 2.26 (s, 3H), 1.85-1.92 (m, 1H), 1.54-1.61 (m, 1H), 1.17-1.23 (m, 2H), 0.85 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 14.2, 20.7, 21.1, 35.7, 50.7, 128.2, 129.5, 136.4, 137.3, 175.5 ppm; IR (neat) ν_{max} cm^{-1} 3020, 2955, 2922, 2870, 1703, 1512, 1447; GC-MS m/z: 192. HRMS (ESI^{-}) calcd for $C_{12}H_{15}O_2^-$ 191.1072; found: 191.1075.



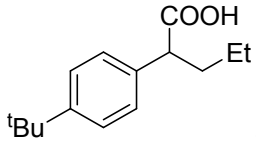
(2c)

2-(*m*-Tolyl)pentanoic acid (2c): Colorless oil, 115.2 mg, Yield: 60%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 11.47 (s, 1H), 7.06-7.22 (m, 4H), 3.51 (t, *J* = 8 Hz, 1H), 2.33 (m, 3H), 1.99-2.08 (m, 1H), 1.70-1.79 (m, 1H), 1.25-1.33 (m, 2H), 0.90 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 101 MHz): 13.9, 20.8, 21.5, 35.2, 51.4, 125.2, 128.3, 128.6, 128.9, 138.4, 138.6, 180.7 ppm; GC-MS m/z: 192. HRMS (ESI^{-}) calcd for $C_{12}H_{15}O_2^-$ 191.1072; found: 191.1070.



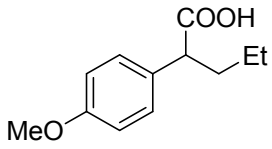
(2d)

2-(4-Isopropylphenyl)pentanoic acid (2d): Colorless oil, 173.8 mg, Yield: 79%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz) δ 12.19 (s, 1H), 7.12-7.17 (m, 4H), 3.42 (t, *J* = 8 Hz, 1H), 2.77-2.84 (m, 1H), 1.83-1.92 (m, 1H), 1.52-1.61 (m, 1H), 1.11-1.23 (m, 8H), 0.82 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 14.2, 20.8, 24.4, 33.6, 35.7, 50.8, 126.8, 128.2, 137.7, 147.4, 175.5 ppm; GC-MS m/z: 220. HRMS (ESI$^-$) calcd for $C_{14}H_{19}O_2^-$ 219.1385; found: 219.1381.



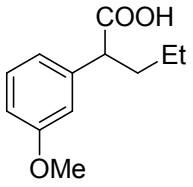
(2e)

2-(4-(Tert-butyl)phenyl)pentanoic acid (2e): Colorless oil, 196.6 mg, Yield: 84%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.23 (s, 1H), 7.33 (d, *J* = 8 Hz, 2H), 7.21 (d, *J* = 8 Hz, 2H), 3.46 (t, *J* = 8 Hz, 1H), 1.87-1.96 (m, 1H), 1.56-1.65 (m, 1H), 1.26, (s, 9H), 1.17-1.24 (m, 2H), 0.86 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 14.2, 20.8, 31.7, 34.6, 35.7, 50.7, 125.7, 127.9, 137.3, 149.6, 175.5 ppm; GC-MS m/z: 234. HRMS (ESI$^-$) calcd for $C_{15}H_{21}O_2^-$ 233.1542; found: 233.1539.



(2f)

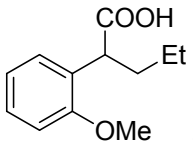
2-(4-Methoxyphenyl)pentanoic acid (2f): White solid, m.p. 67-69 ^{o}C, 187.2 mg, Yield: 90%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.20 (s, 1H), 7.20 (d, *J* = 8 Hz, 2H), 6.87 (d, *J* = 8 Hz, 2H), 3.72, (s, 3H), 3.43 (t, *J* = 8 Hz, 1H), 1.84-1.93 (m, 1H), 1.53-1.62 (m, 1H), 1.17-1.25 (m, 2H), 0.85 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 14.2, 20.7, 35.7, 50.2, 55.5, 114.3, 129.3, 132.3, 175.7 ppm; IR (neat) ν_{max} cm^{-1} 3031, 2958, 2933, 2873, 1703, 1610, 1584, 1512, 1464, 1249, 1178; GC-MS m/z: 208. HRMS (ESI$^-$) calcd for $C_{12}H_{15}O_2^-$ 207.1021; found: 207.1026.



(2g)

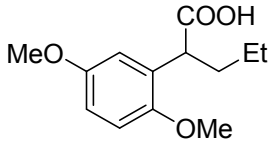
2-(3-Methoxyphenyl)pentanoic acid (2g): Colorless oil, 133.1 mg, Yield: 64%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.29 (s, 1H), 7.23 (t, *J* = 8 Hz, 1H), 6.80-6.87 (m, 3H), 3.73, (s, 3H), 3.47 (t, *J* = 8 Hz, 1H), 1.85-1.95 (m, 1H), 1.56-1.64 (m, 1H), 1.16-1.27 (m, 2H), 0.86 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 14.2, 20.8, 35.7, 51.1, 55.5, 112.6,

114.2, 120.5, 130.0, 141.9, 159.8, 175.3 ppm; IR (neat) ν_{max} cm^{-1} 3025, 2957, 2933, 2872, 1713, 1599, 1584, 1488, 1258, 1193; GC-MS m/z: 208. HRMS (ESI^-) calcd for $C_{12}H_{15}O_2^-$ 207.1021; found: 207.1024.



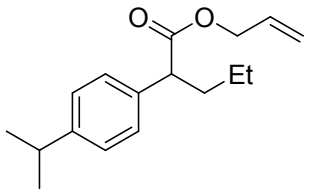
(2h)

2-(2-Methoxyphenyl)pentanoic acid (2h): Colorless oil, 112.3 mg, Yield: 54%; ^1H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.11 (s, 1H), 7.19-7.23 (m, 2H), 6.97 (d, *J* = 8 Hz, 1H), 6.91 (t, *J* = 8 Hz, 1H), 3.86 (t, *J* = 8 Hz, 1H), 3.76, (s, 3H), 1.83-1.92 (m, 1H), 1.53-1.62 (m, 1H), 1.13-1.26 (m, 2H), 0.84 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 14.3, 20.9, 34.7, 43.6, 56.0, 111.6, 120.9, 128.4, 128.4, 128.7, 157.1, 175.6 ppm; IR (neat) ν_{max} cm^{-1} 3035, 2957, 2934, 2872, 2837, 1709, 1599, 1493, 1463, 1243, 1186; GC-MS m/z: 208. HRMS (ESI^-) calcd for $C_{12}H_{15}O_2^-$ 207.1021; found: 207.1018.



(2i)

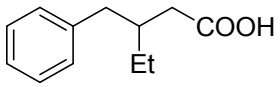
2-(2,5-Dimethoxyphenyl)pentanoic acid (2i): Colorless oil, 140.4 mg, Yield: 59%; ^1H NMR ($CDCl_3$, 400 MHz): δ 6.87 (d, *J* = 4 Hz, 1H), 6.74-6.82 (m, 2H), 4.01 (t, *J* = 8 Hz, 1H), 3.77, (s, 3H), 3.75, (s, 3H), 1.95-2.05 (m, 1H), 1.69-1.75 (m, 1H), 1.24-1.34 (m, 2H), 0.90 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 101 MHz): 14.0, 20.8, 34.2, 43.9, 55.8, 56.4, 112.1, 112.5, 114.9, 128.7, 151.4, 153.8, 180.3 ppm; IR (neat) ν_{max} cm^{-1} 3020, 2957, 2933, 2872, 2835, 1702, 1591, 1498, 1464, 1226, 1178; GC-MS m/z: 238. HRMS (ESI^-) calcd for $C_{13}H_{17}O_4^-$ 237.1127; found: 237.1122.



(3d)

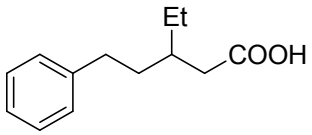
Allyl 2-(4-isopropylphenyl)pentanoate (3d): Colorless oil, 174.3 mg, Yield: 67%; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.15-7.24 (m, 4H), 5.82-5.90 (m, 1H), 5.16-5.24 (m, 2H), 4.50-4.61 (m, 2H), 3.56 (t, *J* = 8 Hz, 2H), 2.84-2.90 (m, 1H), 2.00-2.08 (m, 2H), 1.72-1.75 (m, 2H), 1.23 (d, *J* = 8 Hz, 6H), 0.91 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 101 MHz): 13.9, 20.9, 24.1, 33.8, 35.8, 51.2, 65.2, 118.0, 126.7, 127.9, 132.3, 136.6, 147.8, 174.1 ppm; IR (neat) ν_{max} cm^{-}

[1] 2959, 2927, 2872, 1735, 1511, 1461, 1156; GC-MS m/z: 260 HRMS (ESI Mode) calcd for $C_{17}H_{24}O_2$+H^+ 261.1855; found: 261.1859.



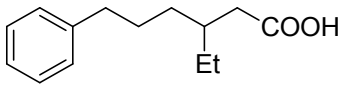
(2k)

3-Benzylpentanoic acid (2k): Colorless oil, 126.7 mg, Yield: 66%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.07 (s, 1H), 7.29 (t, *J* = 8 Hz, 2H), 7.18 (t, *J* = 8 Hz, 3H), 2.50-2.60 (m, 2H), 2.06-2.18 (m, 2H), 1.95-2.01 (m, 1H), 1.24-1.32 (m, 1H), 0.86 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 11.2, 25.9, 38.1, 38.5, 126.4, 128.7, 129.6, 140.9, 174.6 ppm; GC-MS m/z: 192. HRMS (ESI$^-$) calcd for $C_{12}H_{15}O_2^-$ 191.1072; found: 191.1069.



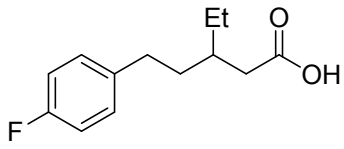
(2l)

3-Ethyl-5-phenylpentanoic acid (2l): Colorless oil, 152.4 mg, Yield: 74%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 12.01 (s, 1H), 7.13 -7.28 (m, 5H), 2.56 (t, *J* = 8 Hz, 2H), 2.15-2.25 (m, 2H), 1.70-1.77 (m, 2H), 1.50-1.59 (m, 2H), 1.30-1.41 (m, 2H), 0.84 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 11.1, 26.1, 32.8, 35.4, 36.0, 38.7, 126.1, 128.7, 128.8, 142.9, 174.7 ppm; IR (neat) ν_{max} cm^{-1} 3026, 2961, 2928, 2875, 2859, 1706, 1603, 1496, 1454, 1251, 1192; GC-MS m/z: 206. HRMS (ESI$^-$) calcd for $C_{13}H_{17}O_2^-$ 205.1229; found: 205.1226.



(2m)

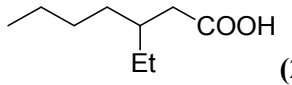
3-Ethyl-6-phenylhexanoic acid (2m): Colorless oil, 114.4 mg, Yield: 52%; ^{1}H NMR (*d*^{6}-DMSO, 400 MHz): δ 11.98 (s, 1H), 7.15-7.26 (m, 5H), 2.54 (t, *J* = 8 Hz, 2H), 2.12 (d, *J* = 8 Hz, 2H), 1.68-1.75 (m, 1H), 1.51-1.58 (m, 2H), 1.23-1.30 (m, 4H), 0.80 (t, *J* = 8 Hz, 6H) ppm; ^{13}C NMR (*d*^{6}-DMSO, 101 MHz): 11.1, 26.3, 28.6, 33.0, 35.9, 36.1, 38.9, 126.1, 128.7, 128.8, 142.7, 174.8 ppm; GC-MS m/z: 220. HRMS (ESI$^-$) calcd for $C_{14}H_{19}O_2^-$ 219.1385; found: 219.1381.



(2n)

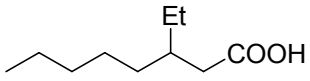
3-Ethyl-5-(4-fluorophenyl)pentanoic acid (2n): Colorless oil, 164.0 mg, Yield: 73%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.10-7.14 (m, 2H), 6.93-6.97 (m, 2H), 2.59 (t, *J* = 8 Hz, 2H), 2.34-2.36 (m, 2H), 1.84-1.90 (m, 1H), 1.59-1.66 (m, 2H), 1.37-1.50 (m, 2H), 0.91 (t, *J* = 8

Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 101MHz): 10.8, 26.2, 32.3, 35.5, 36.0, 38.4, 115.2 (d, J = 21.1 Hz), 129.7 (d, J = 7.7 Hz), 138.0, (d, J = 2.9 Hz), 161.3 (d, J = 243.3 Hz), 180.0 ppm; IR (neat) ν_{max} cm^{-1} 3030, 2962, 2927, 2878, 2862, 1703, 1601, 1509, 1458, 1220, 1157; GC-MS m/z: 224. HRMS (ESI$^-$) calcd for $C_{13}H_{16}FO_2^-$ 223.1134; found: 223.1138.



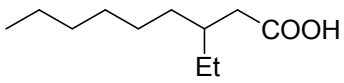
(2o)

3-Ethylheptanoic acid (2o): Colorless oil, 86.9 mg, Yield: 55%; ^{1}H NMR (d^6-DMSO, 400 MHz): δ 11.95 (s, 1H), 2.11 (d, J = 8 Hz, 2H), 1.64-1.70 (m, 1H), 1.23-1.31 (m, 8H), 0.80-0.88 (m, 6H) ppm; ^{13}C NMR (d^6-DMSO, 101 MHz): 11.2, 14.5, 22.9, 26.3, 28.7, 33.0, 36.2, 38.9, 39.4, 174.8 ppm; GC-MS m/z: 158. HRMS (ESI$^-$) calcd for $C_9H_{17}O_2^-$ 157.1229; found: 157.1225.



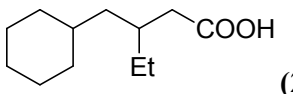
(2p)

2-Propylheptanoic acid (2p): Colorless oil, 103.2 mg, Yield: 60%; ^{1}H NMR (d^6-DMSO, 400 MHz): δ 11.96 (s, 1H), 2.11 (d, J = 4 Hz, 2H), 1.65-1.70 (m, 1H), 1.23-1.31 (m, 10H), 0.80-0.87 (m, 6H) ppm; ^{13}C NMR (d^6-DMSO, 101 MHz): 11.1, 14.4, 22.6, 26.2, 26.3, 32.1, 33.3, 36.2, 38.9, 174.8 ppm; GC-MS m/z: 172. HRMS (ESI$^-$) calcd for $C_{10}H_{19}O_2^-$ 171.1385; found: 171.1381.



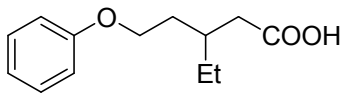
(2q)

3-Ethylnonanoic acid (2q): Colorless oil, 145.1 mg, Yield: 78%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.28 (d, J = 8 Hz, 2H), 1.78-1.83 (m, 1H), 1.27-1.42 (m, 12H), 0.88 (t, J = 8 Hz, 6H) ppm; ^{13}C NMR ($CDCl_3$, 101 MHz): 10.8, 14.2, 22.7, 26.3, 26.6, 29.6, 31.9, 33.4, 36.3, 38.7, 180.6 ppm; IR (neat) ν_{max} cm^{-1} 2959, 2925, 2857, 1705, 1190; GC-MS m/z: 186. HRMS (ESI$^-$) calcd for $C_{11}H_{21}O_2^-$ 185.1542; found: 185.1538.



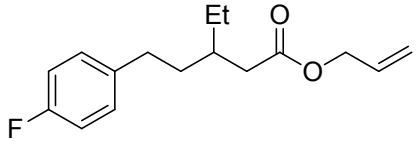
(2r)

3-(Cyclohexylmethyl)pentanoic acid (2r): Colorless oil, 104.9 mg, Yield: 53%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 11.7 (s, 1H), 2.26 (t, J = 8 Hz, 2H), 1.87-1.96 (m, 1H), 1.63-1.70 (m, 5H), 1.06-1.40 (m, 8H), 0.88 (t, J = 8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 101 MHz): 10.7, 26.4, 26.6, 26.8, 33.3, 33.6, 33.6, 34.9, 39.0, 41.6, 180.5 ppm; IR (neat) ν_{max} cm^{-1} 2959, 2920, 1704, 1197; GC-MS m/z: 198. HRMS (ESI$^-$) calcd for $C_{12}H_{21}O_2^-$ 197.1542; found: 197.1547.



(2s)

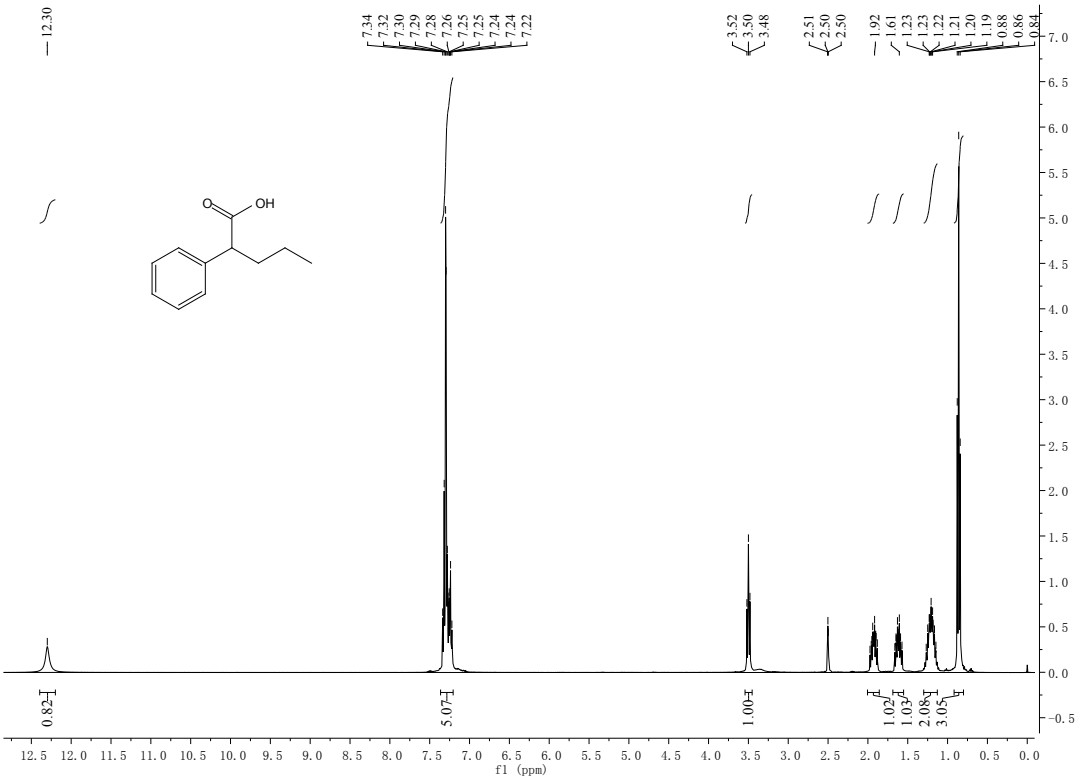
3-Ethyl-5-phenoxypentanoic acid (2s): Colorless oil, 182.0 mg, Yield: 82%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.24-7.28 (m, 2H), 6.87-6.94 (m, 3H), 4.00 (t, *J* = 8 Hz, 1H), 2.39 (d, *J* = 8 Hz, 2H), 2.04-2.10 (m, 1H), 1.77-1.90 (m, 2H), 1.41-1.50 (m, 2H), 0.93 (t, *J* = 8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 101 MHz): 10.9, 26.6, 32.8, 33.8, 38.5, 65.8, 114.5, 120.7, 129.5, 158.9, 179.9 ppm; IR (neat) ν_{max} cm^{-1} 3040, 2962, 2929, 1703, 1599, 1586, 1496, 1474, 1241, 1172; GC-MS m/z: 222. HRMS (ESI^-) calcd for $C_{13}H_{17}O_3^-$ 221.1178; found: 221.1175.



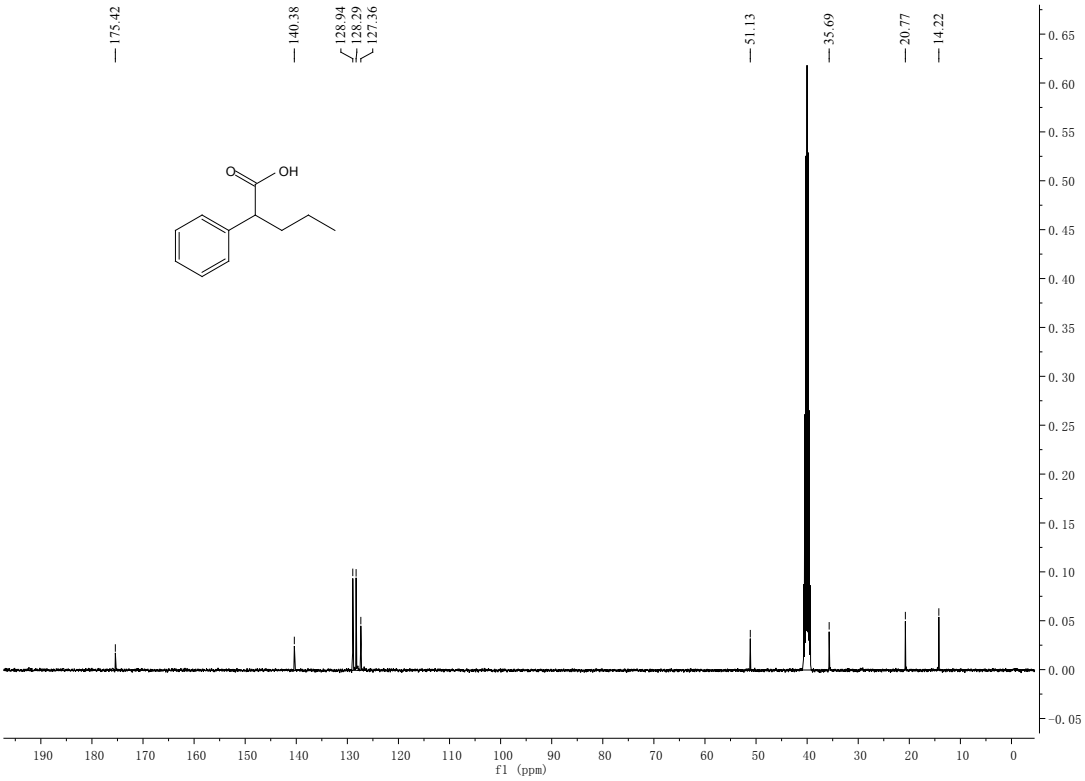
(3n)

Allyl 3-ethyl-5-(4-fluorophenyl)pentanoate (3n): Colorless oil, 163.8 mg, Yield: 62%; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.09-7.14 (m, 2H), 6.92-6.98 (m, 2H), 5.85-5.98 (m, 1H), 5.21-5.34 (m, 2H), 4.58 (d, *J* = 6 Hz, 2H), 2.58 (t, *J* = 9 Hz, 2H), 2.32-2.35 (m, 2H), 1.84-1.92 (m, 1H), 1.56-1.64 (m, 2H), 1.35-1.48 (m, 2H), 0.90 (t, *J* = 6 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 76 MHz): 10.8, 26.3, 32.3, 35.5, 36.2, 38.6, 65.1, 115.1 (d, J = 21.1 Hz), 118.3, 129.7 (d, J = 7.7 Hz), 132.4, 138.1, 161.3 (d, J = 243.2 Hz), 173.0 ppm; IR (neat) ν_{max} cm^{-1} 2962, 2930, 1733, 1601, 1509, 1458, 1220, 1156; GC-MS m/z: 264. HRMS (ESI Mode) calcd for $C_{16}H_{21}FO_2+H^+$ 265.1604; found: 265.1600.

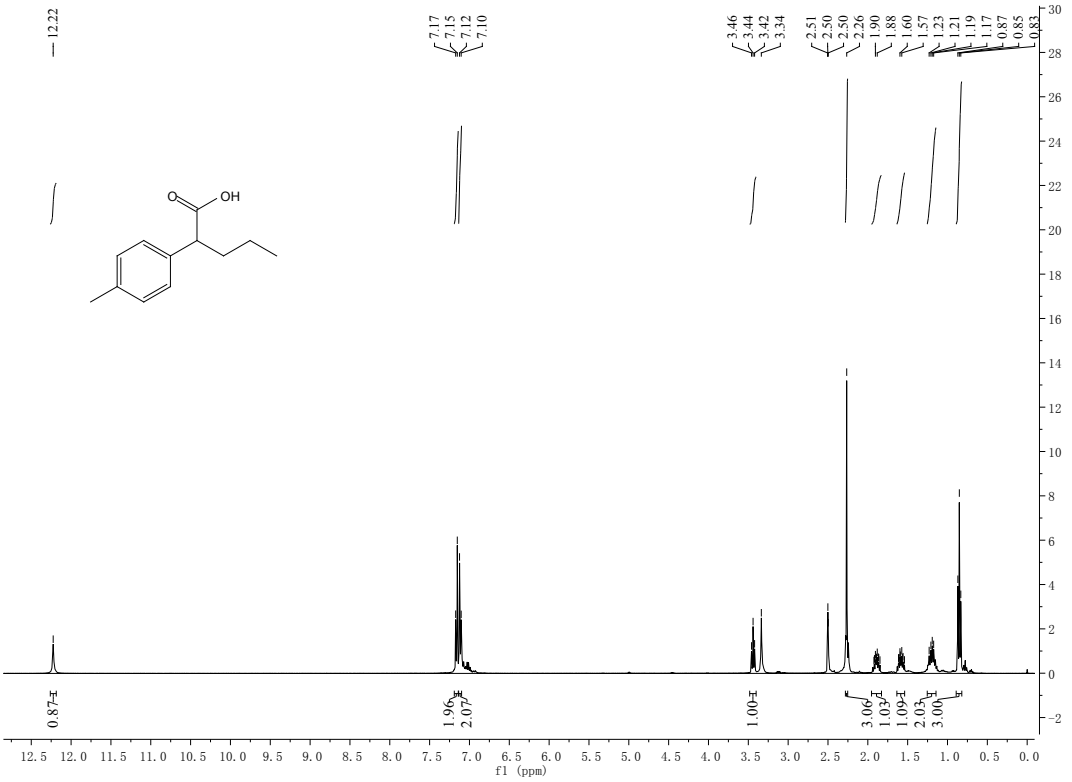
4. ^{1}H NMR and ^{13}C NMR Spectrum



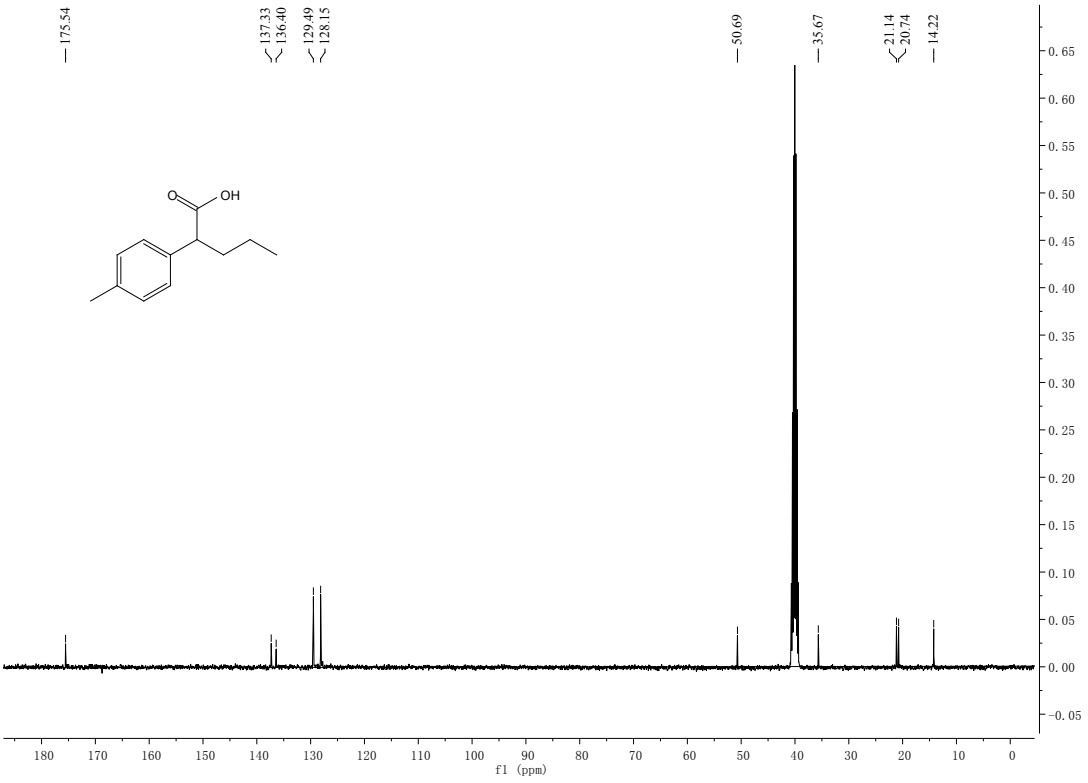
^{1}H NMR for compound **2a**



^{13}C NMR for compound **2a**

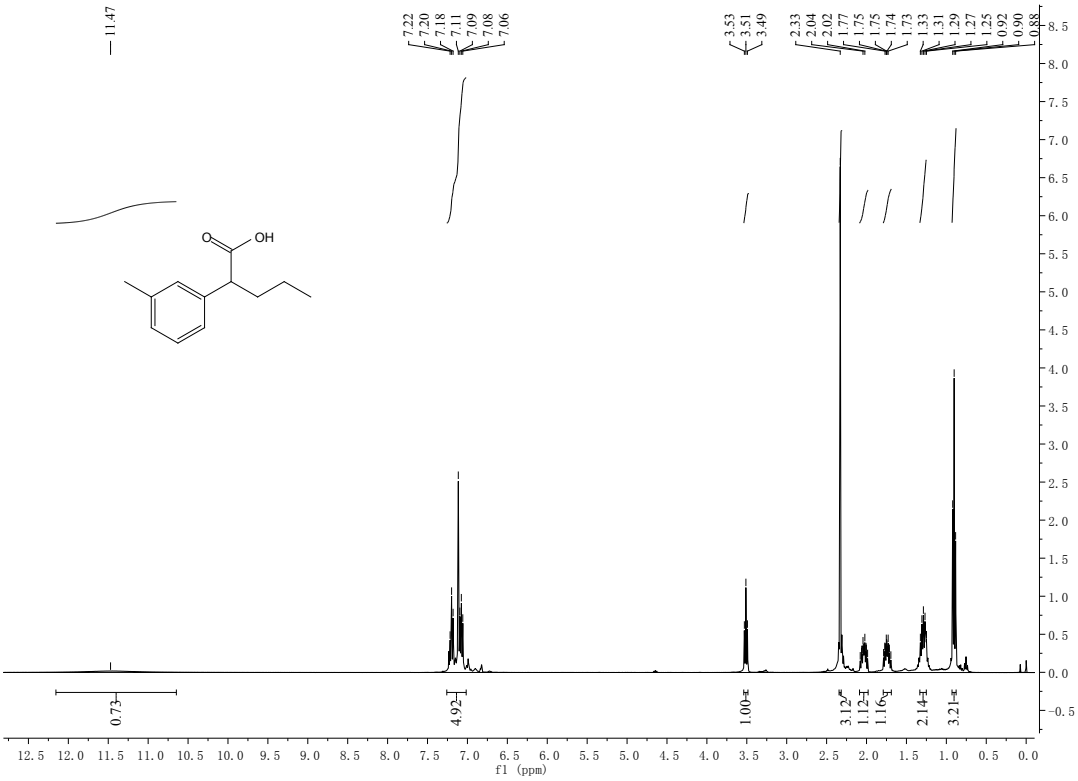


^{1}H NMR for compound **2b**

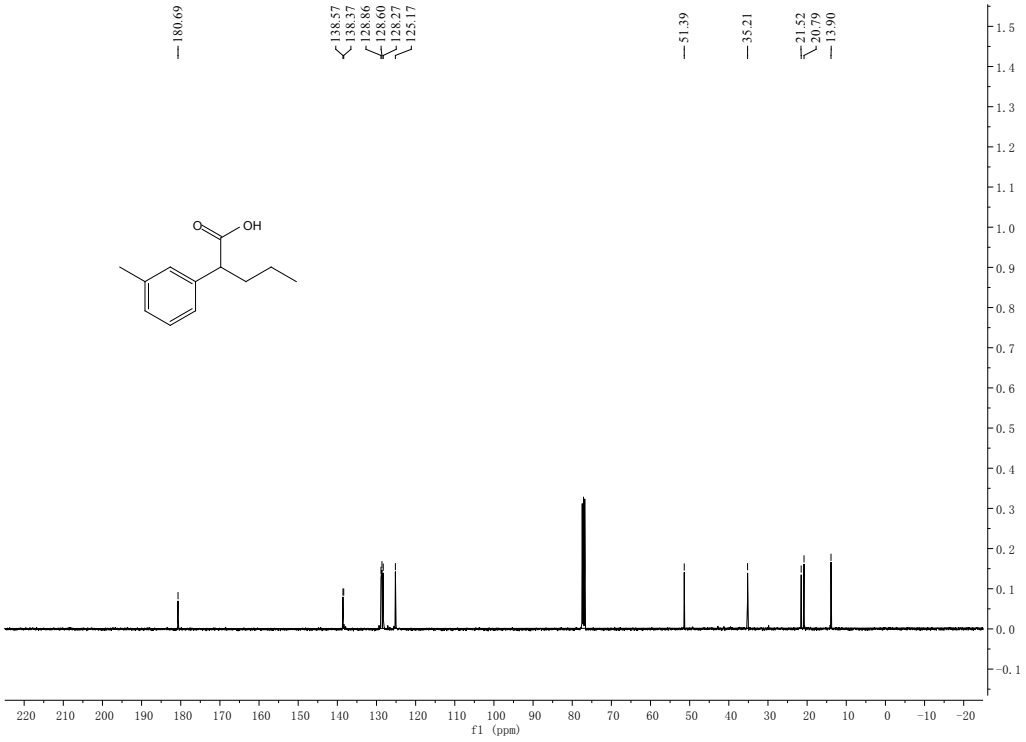


^{13}C NMR for compound **2b**

2c

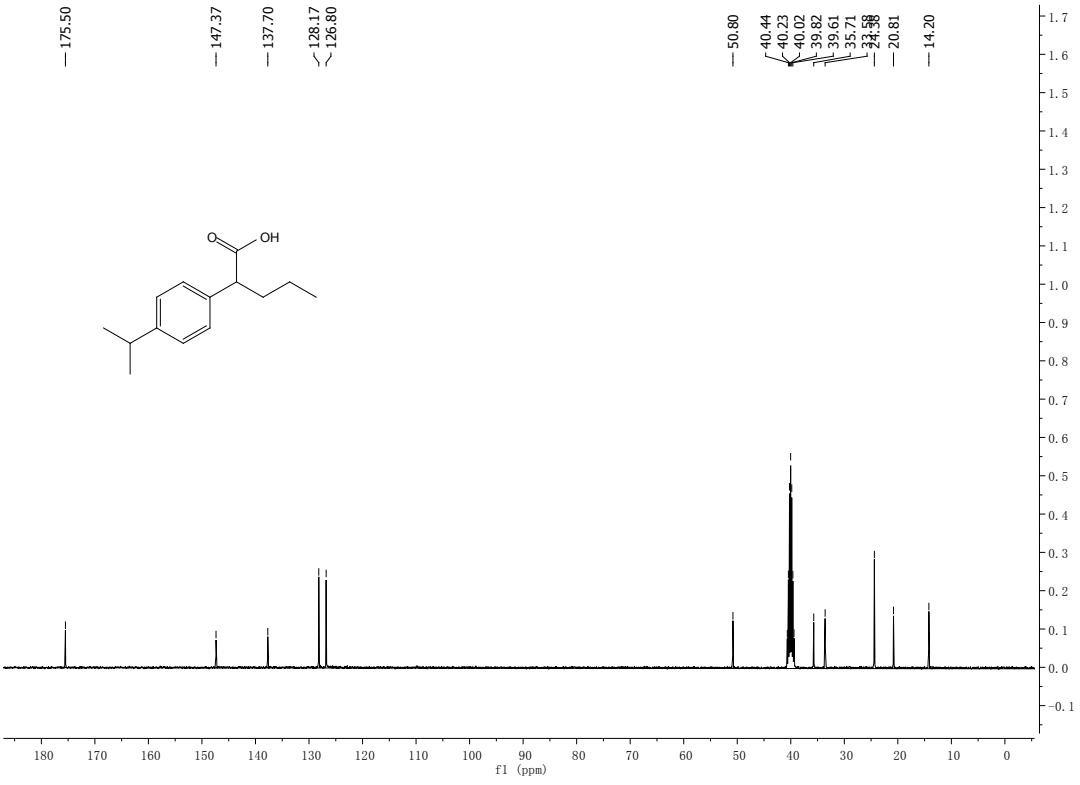


^{1}H NMR for compound **2c**

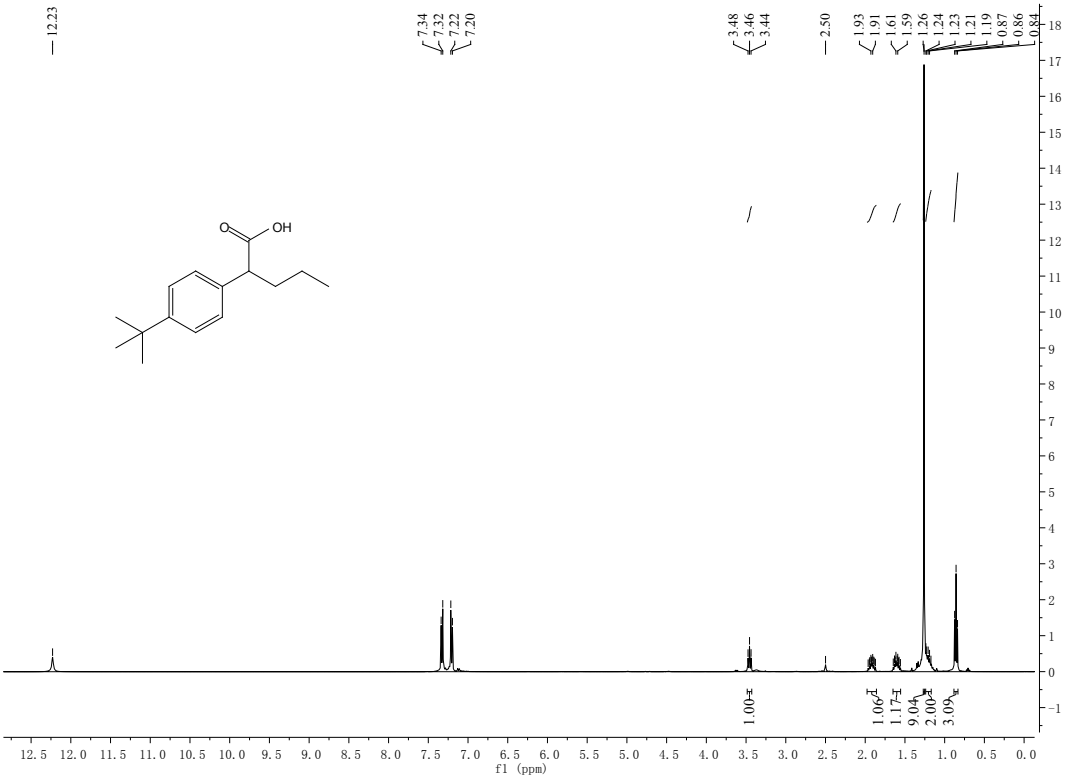


^{13}C NMR for compound **2c**

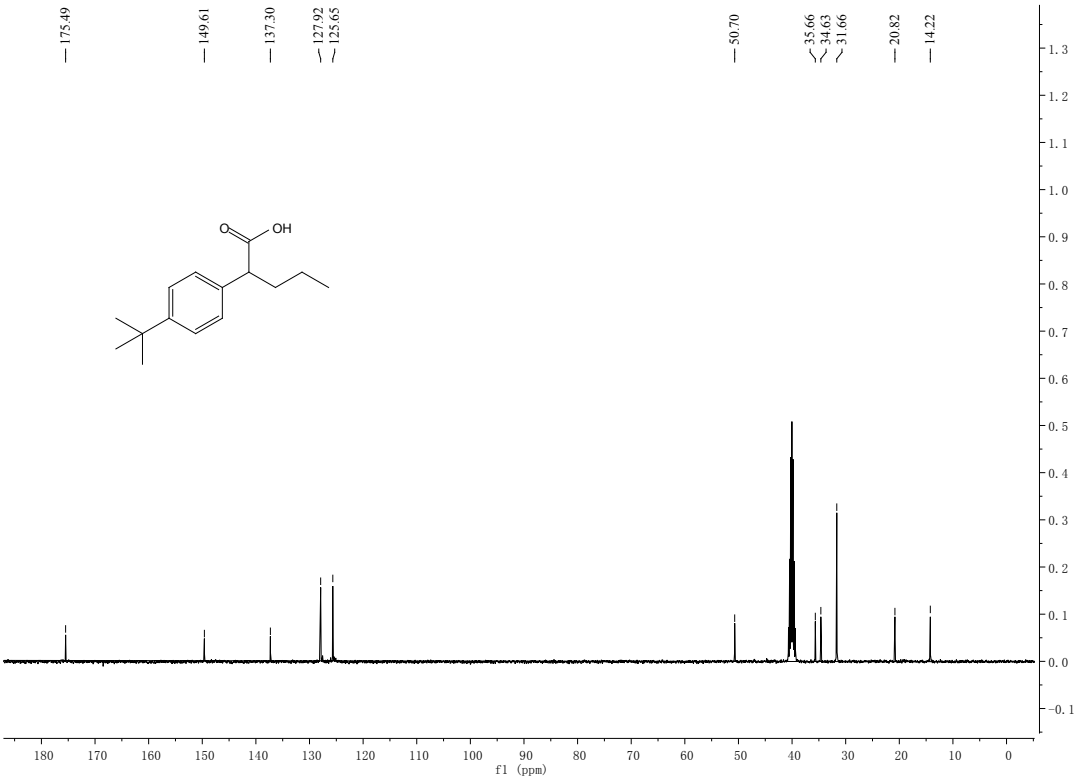
^{1}H NMR for compound **2d**



^{13}C NMR for compound **2d**

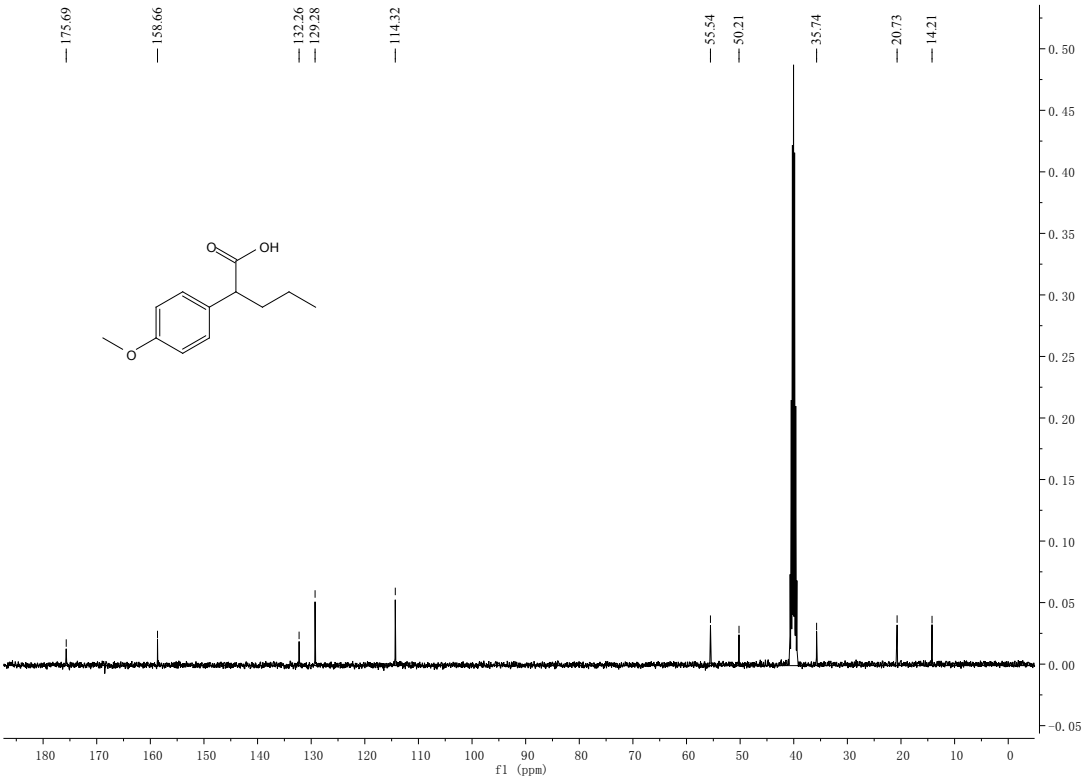


^{1}H NMR for compound **2e**

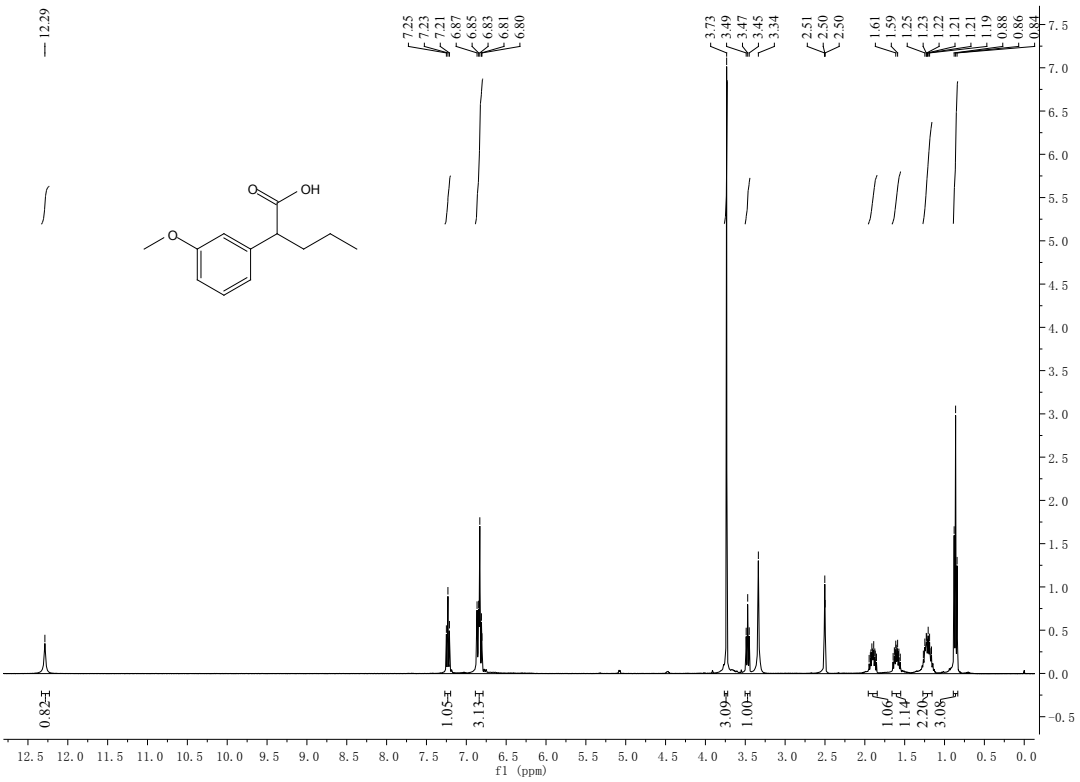


^{13}C NMR for compound **2e**

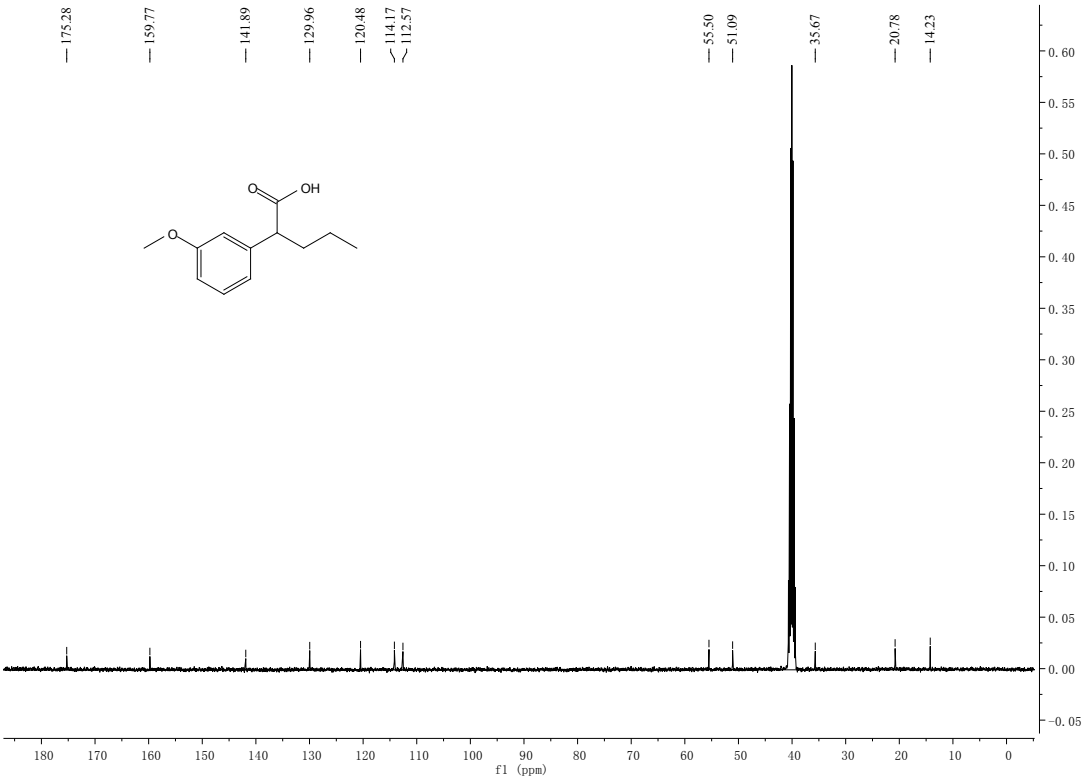
^{1}H NMR for compound **2f**



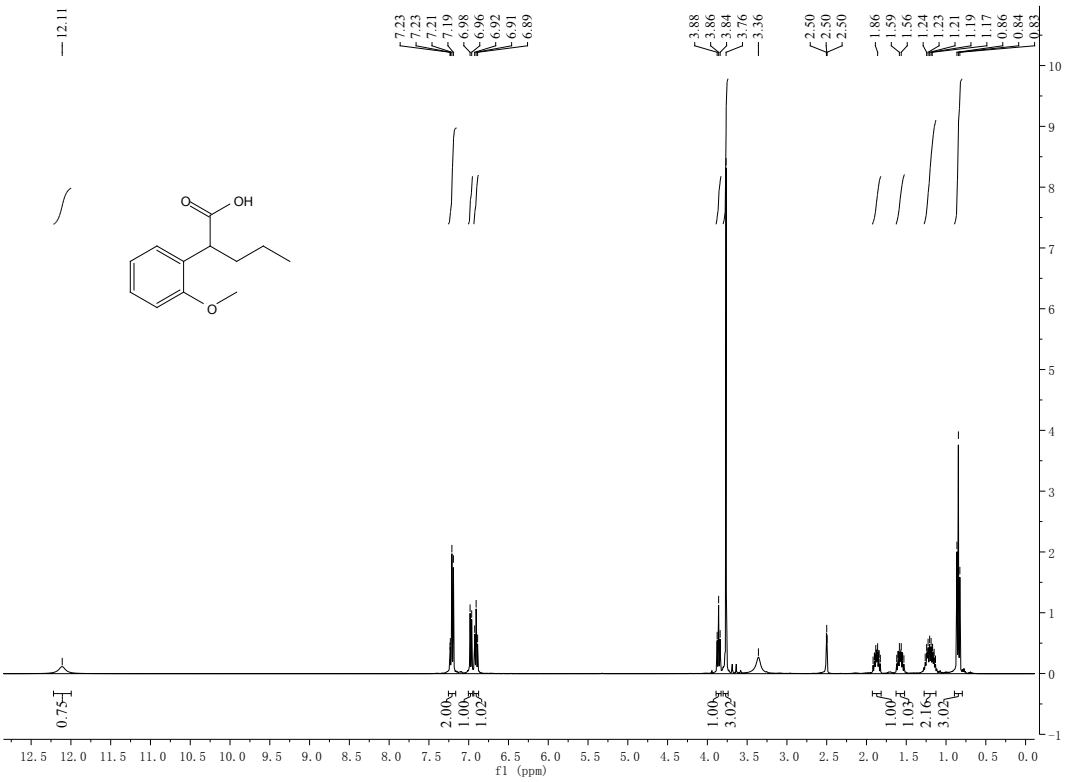
^{13}C NMR for compound **2f**



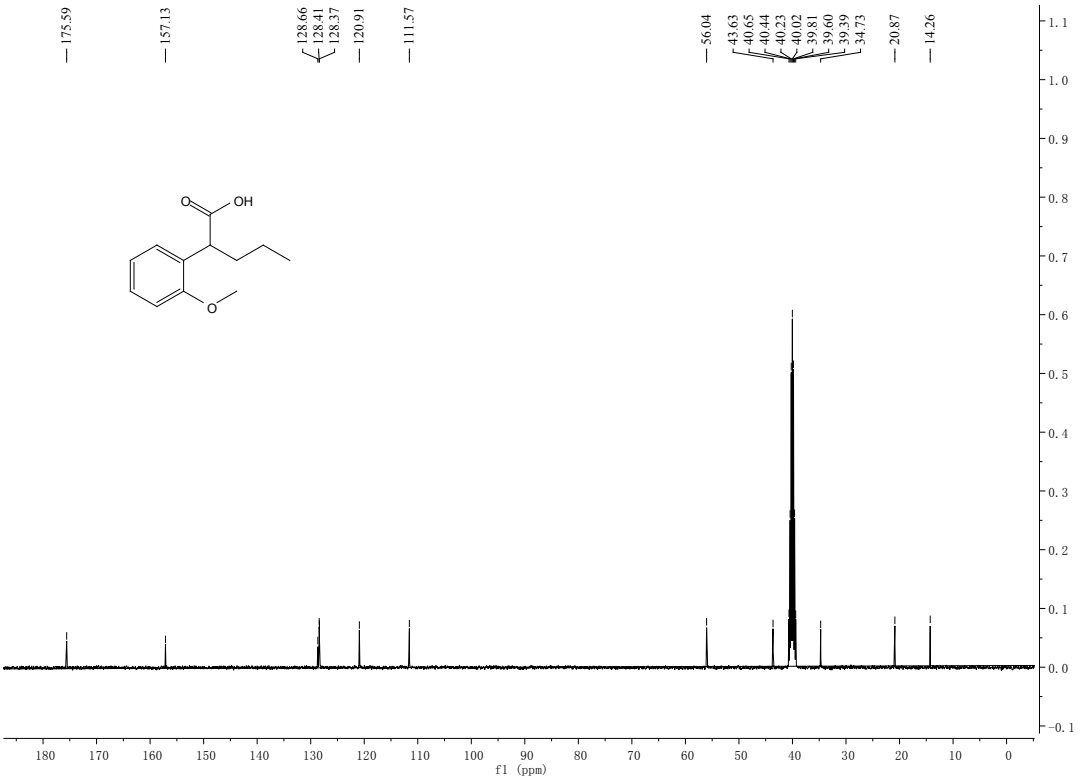
^{1}H NMR for compound **2g**



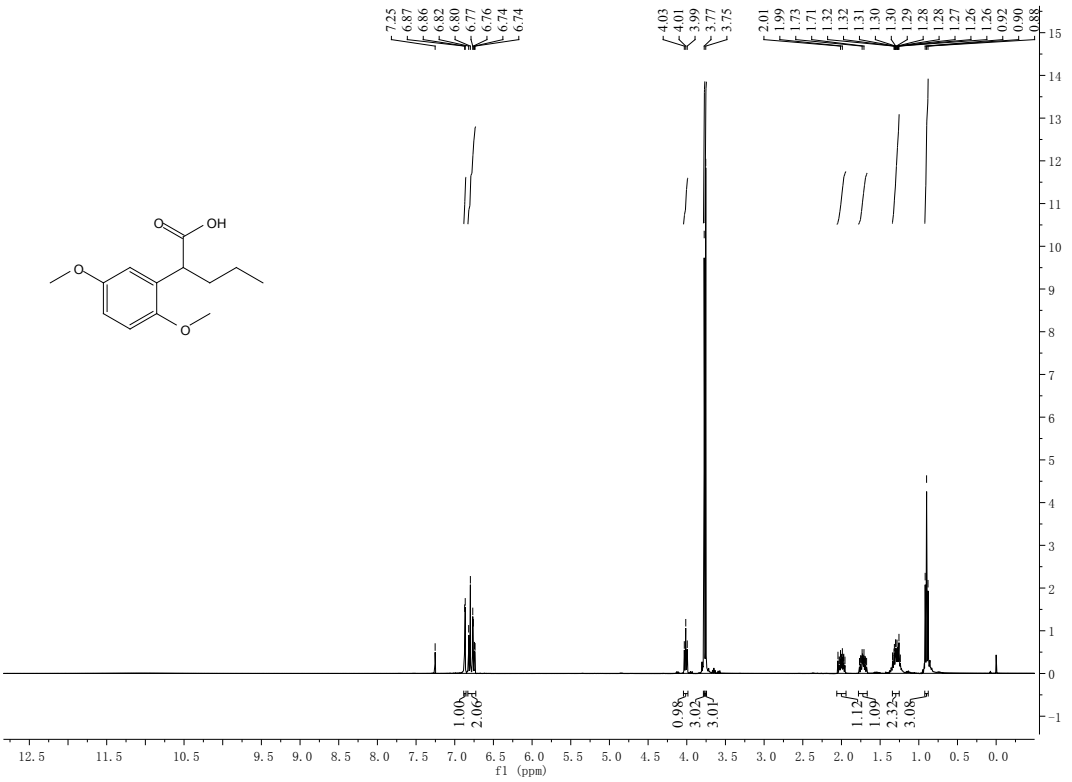
^{13}C NMR for compound **2g**



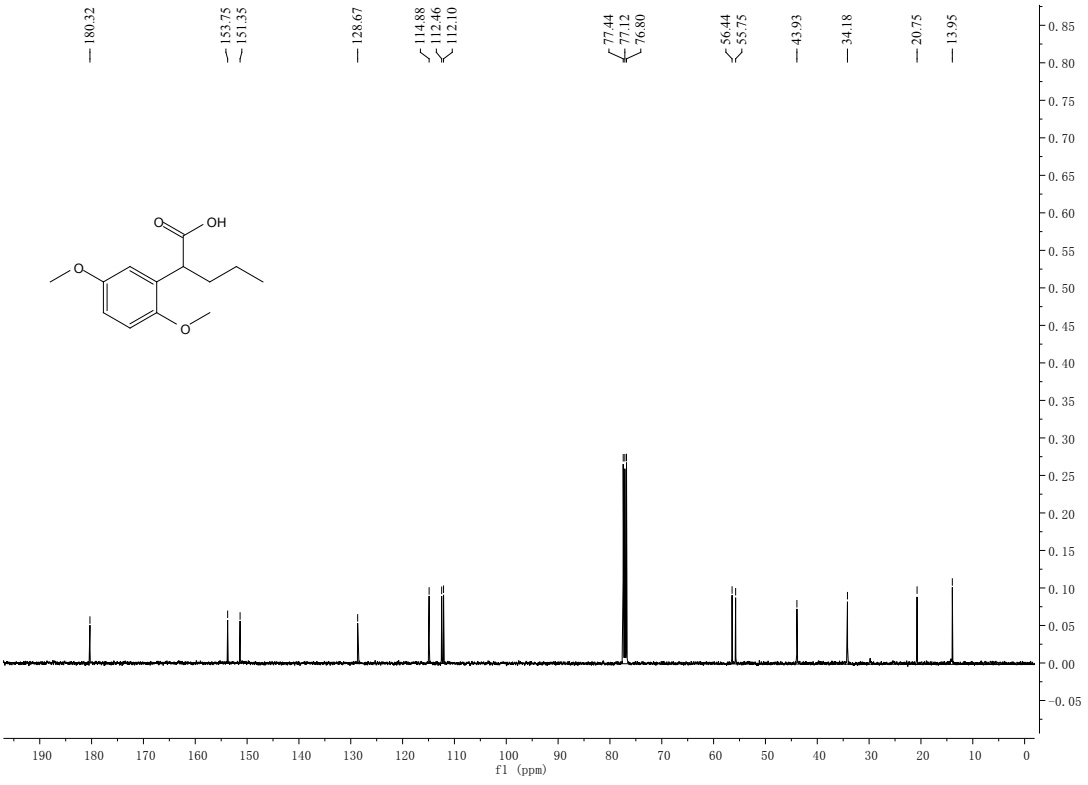
^{1}H NMR for compound **2h**



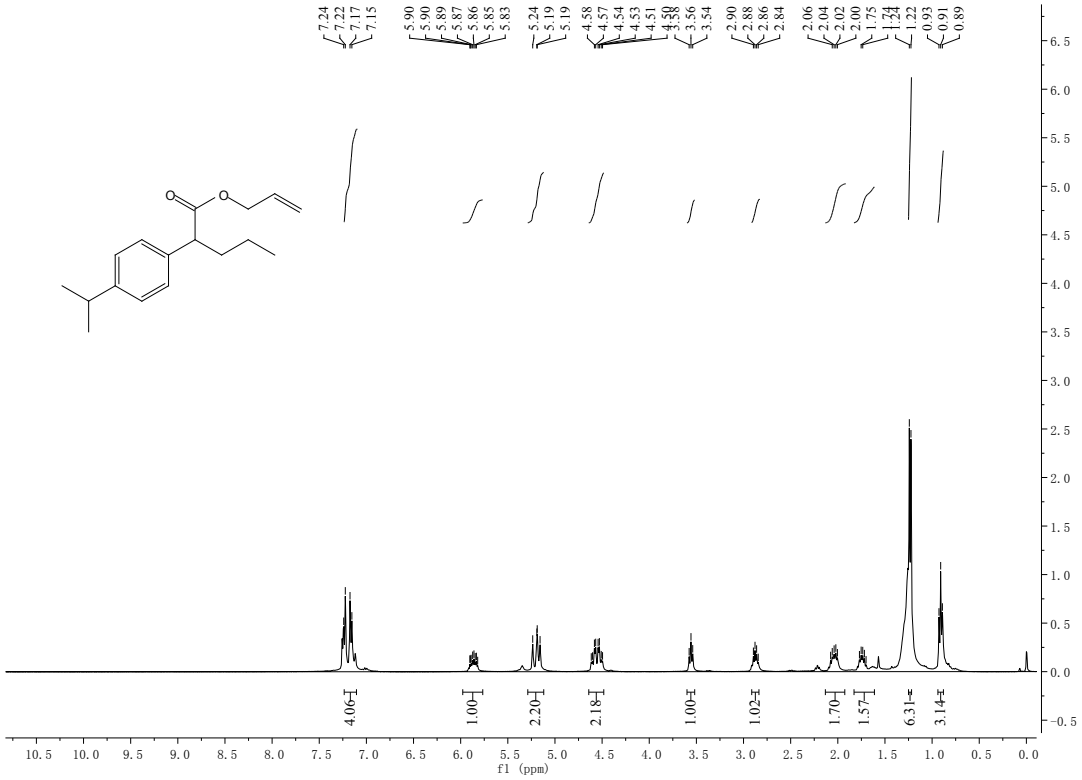
^{13}C NMR for compound **2h**



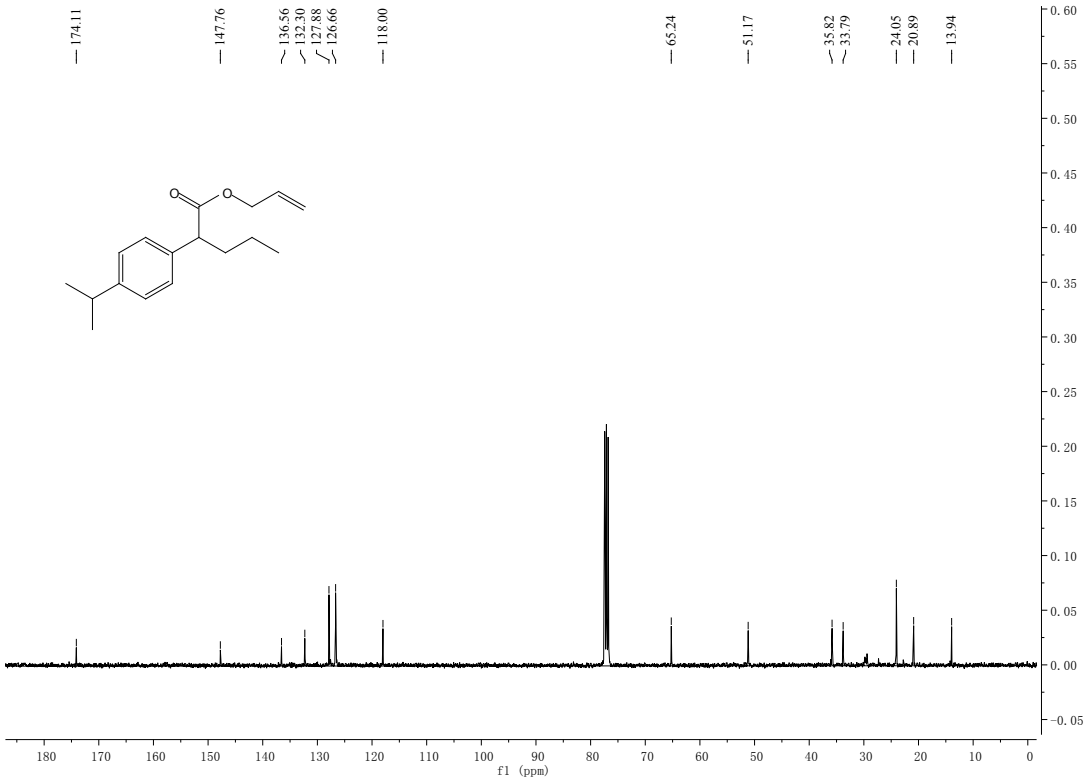
^{1}H NMR for compound **2i**



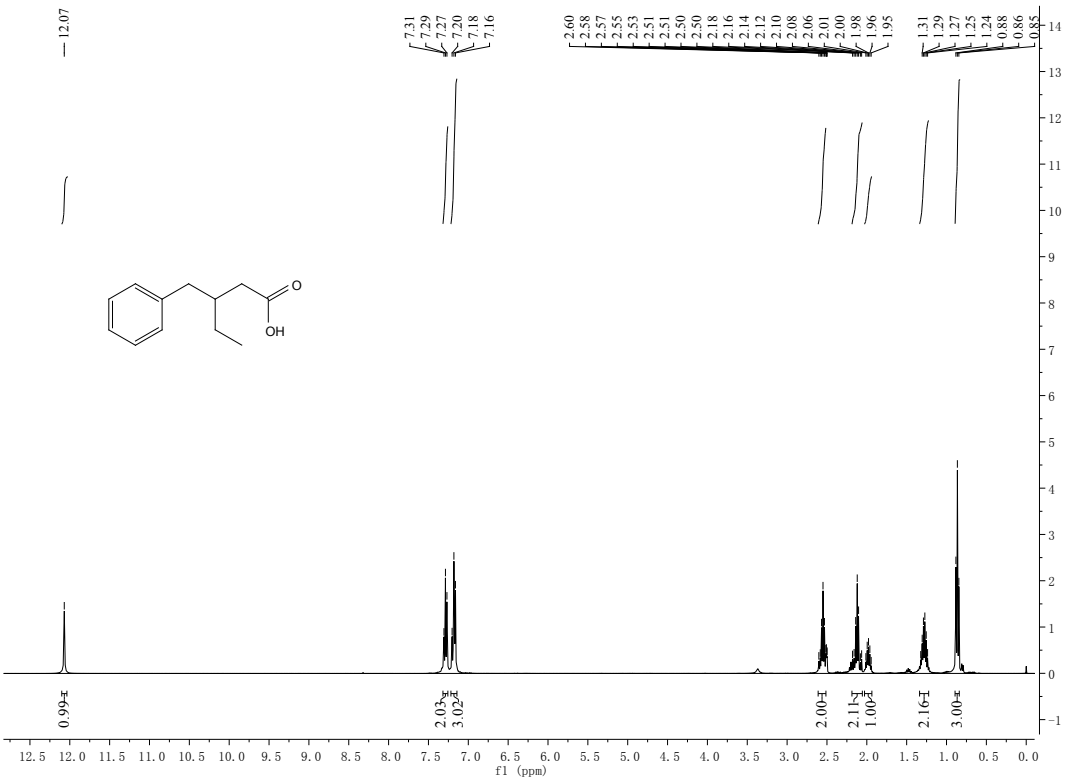
^{13}C NMR for compound **2i**



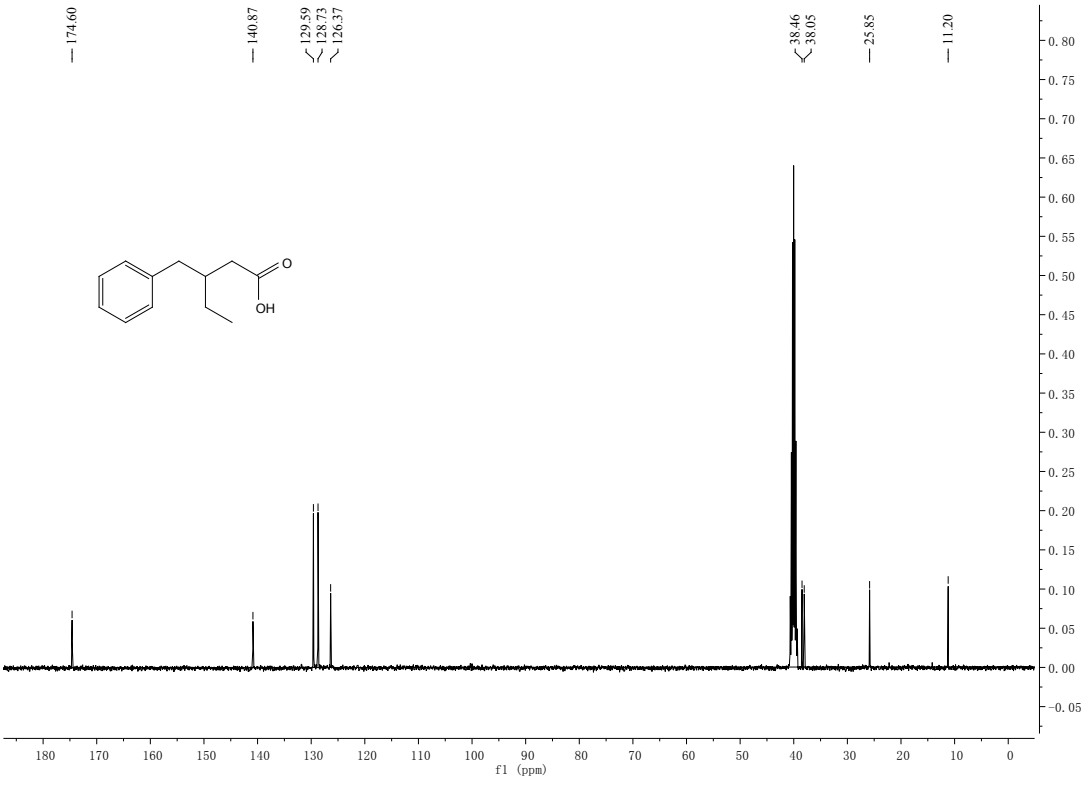
^{1}H NMR for compound **3d**



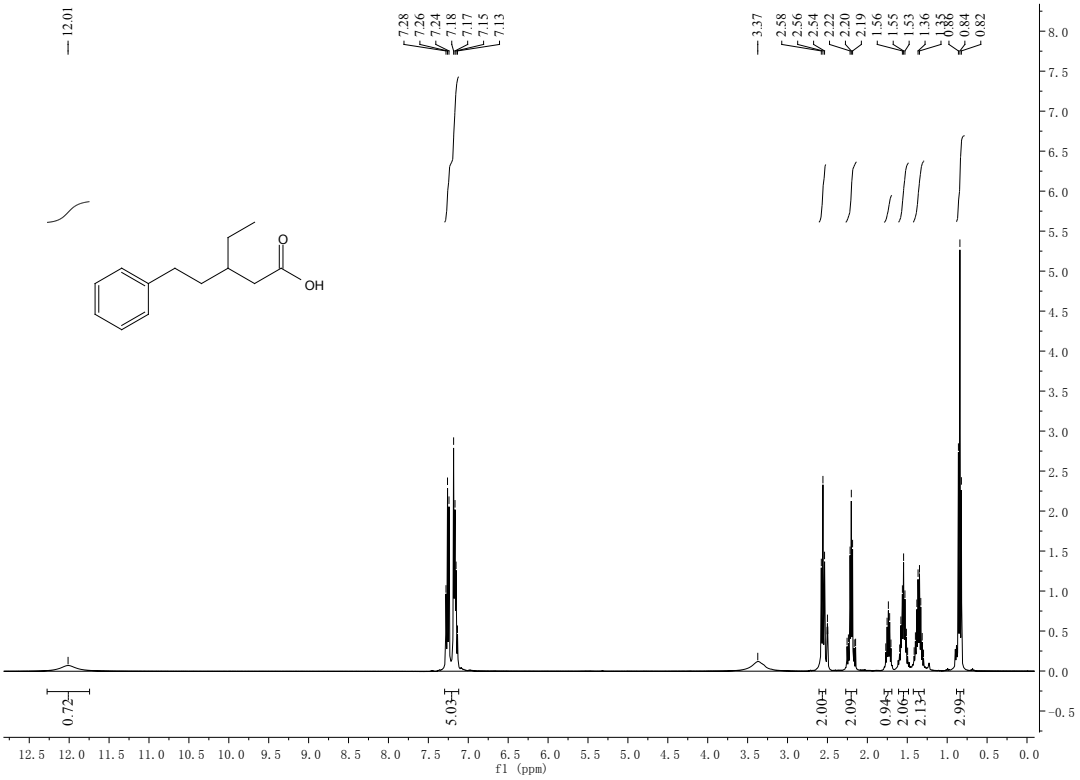
^{13}C NMR for compound **3d**



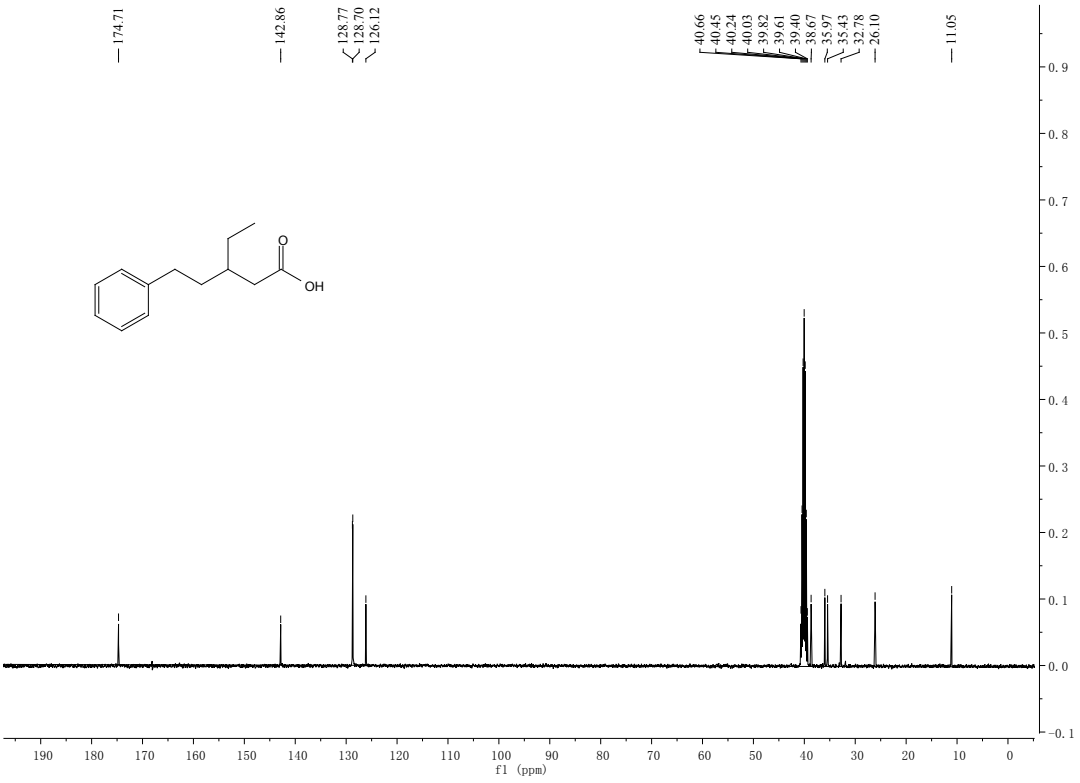
^{1}H NMR for compound **2k**



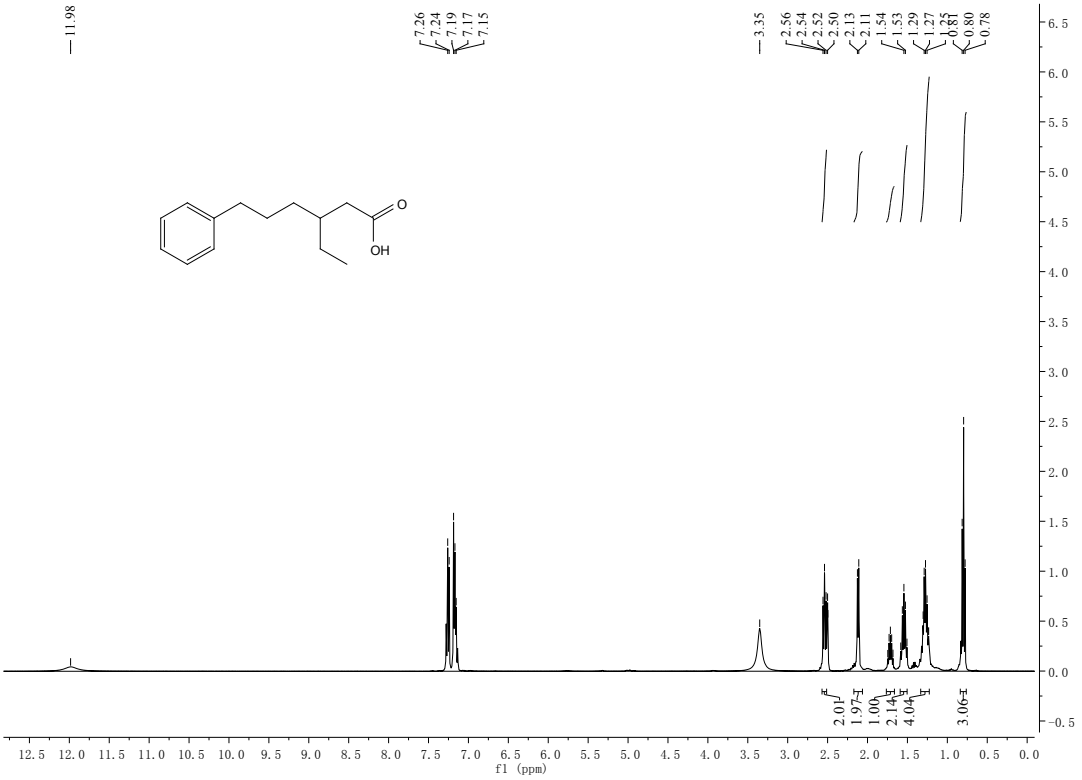
^{13}C NMR for compound **2k**



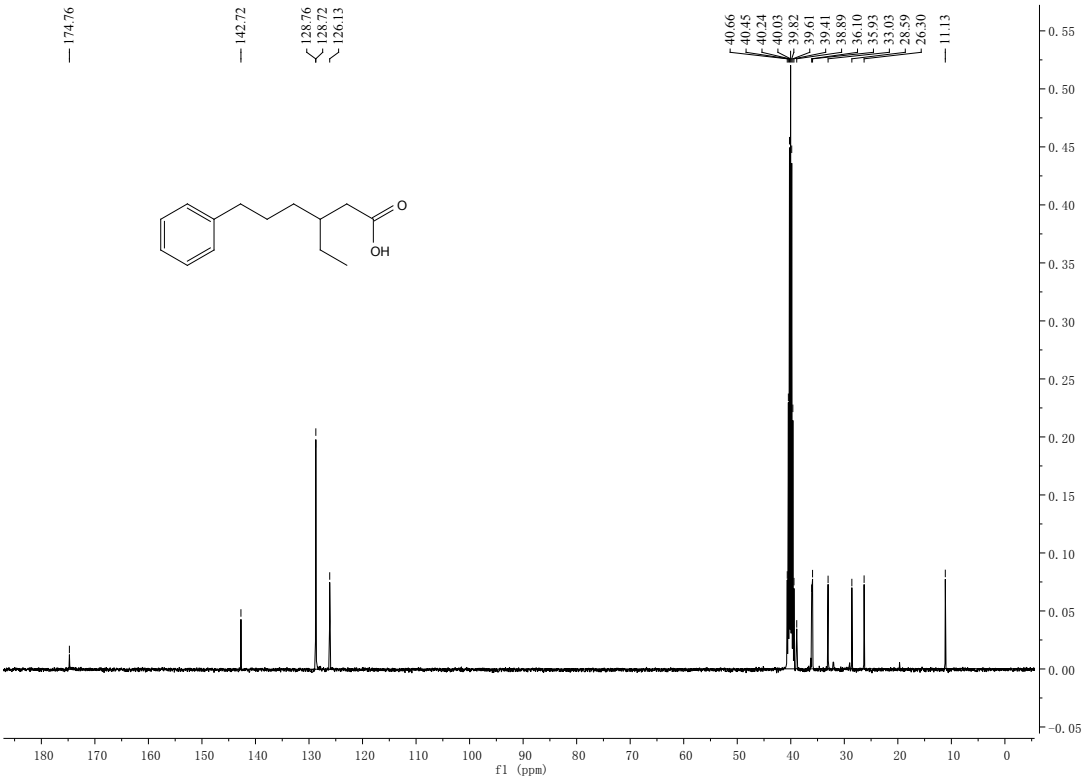
^{1}H NMR for compound **2l**



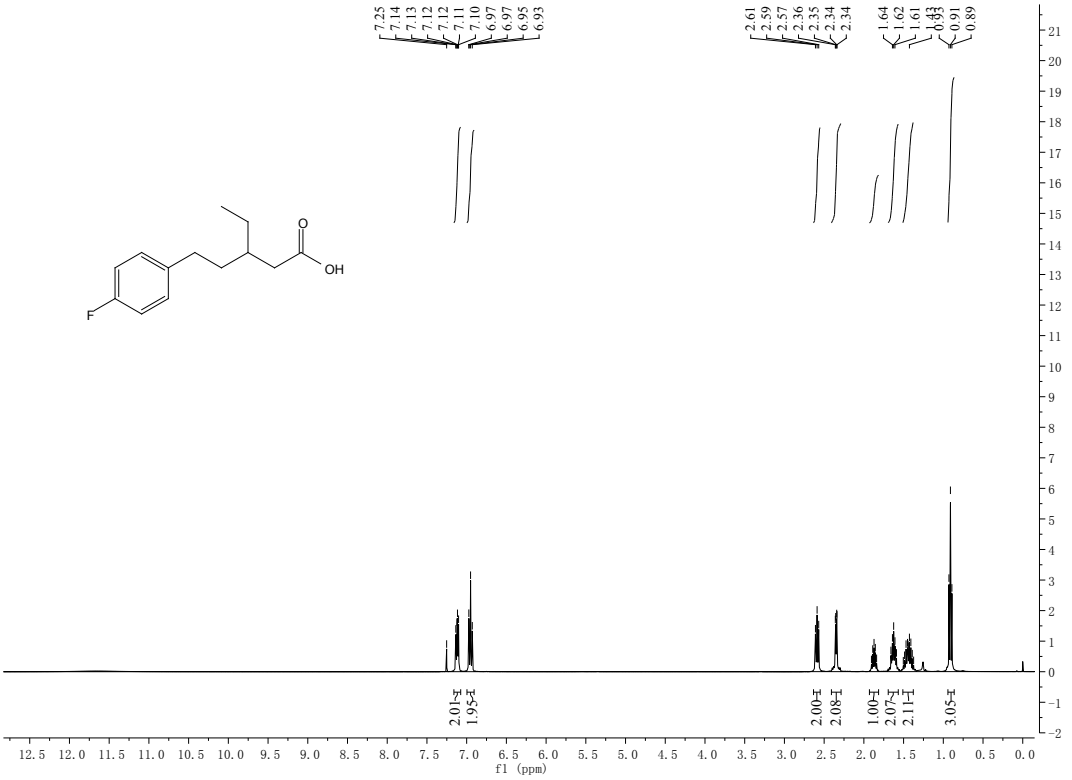
^{13}C NMR for compound **2l**



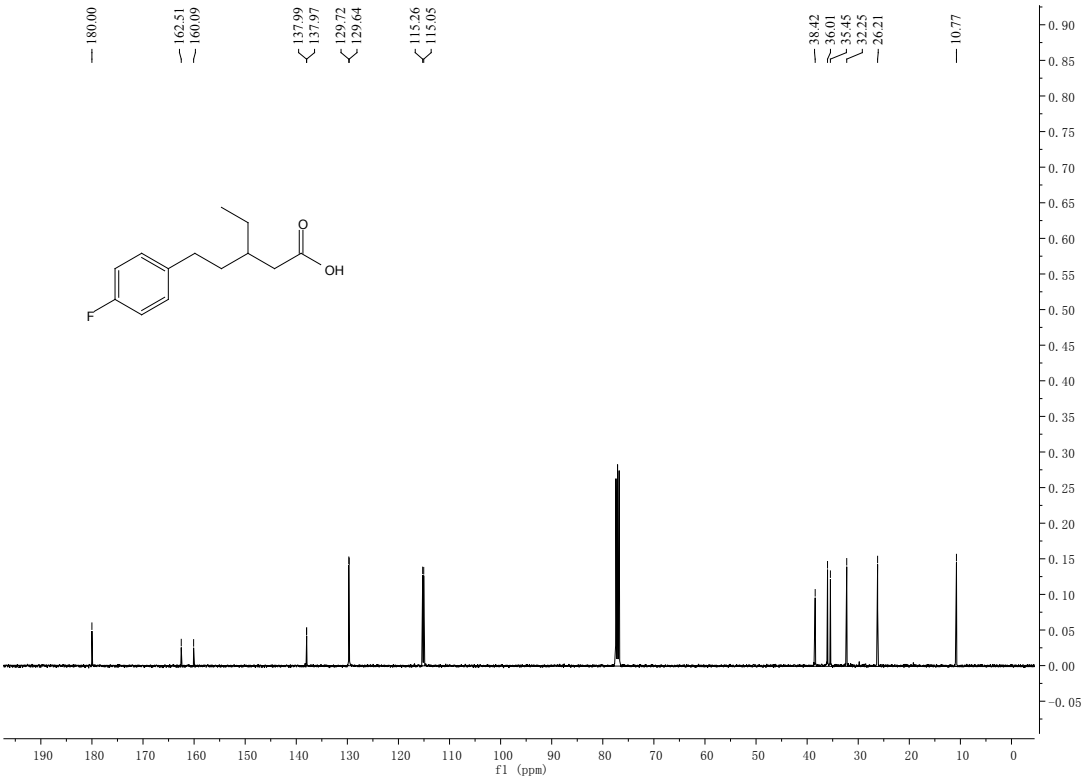
^{1}H NMR for compound **2m**



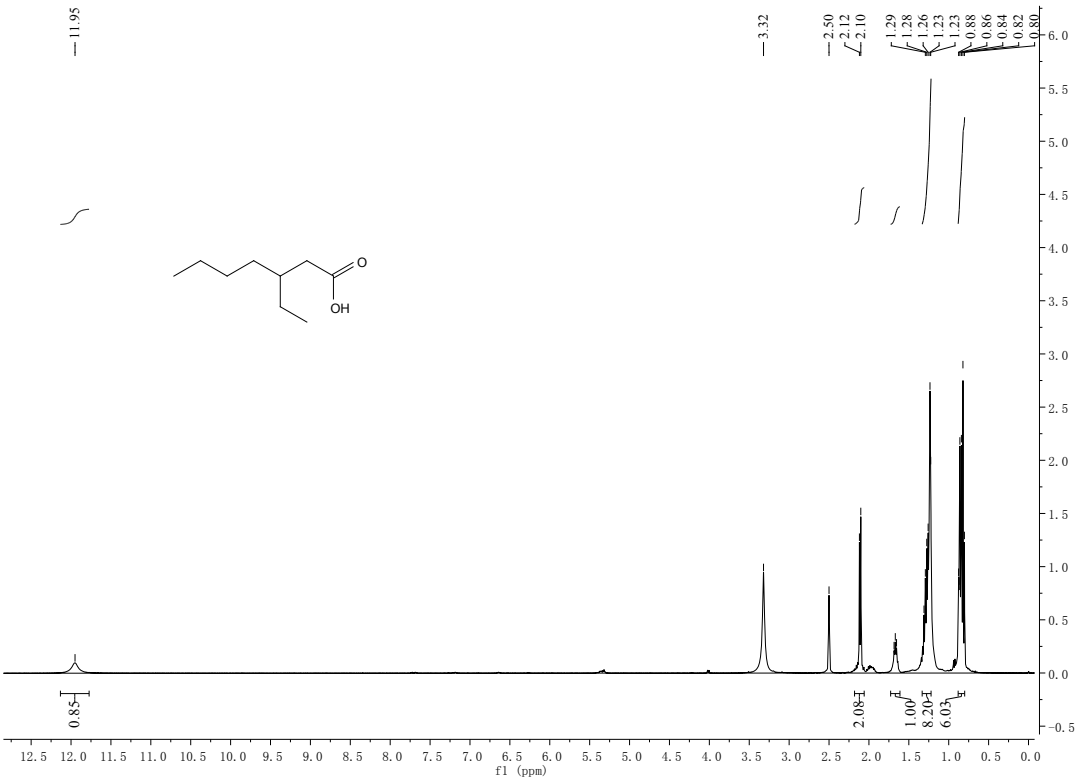
^{13}C NMR for compound **2m**



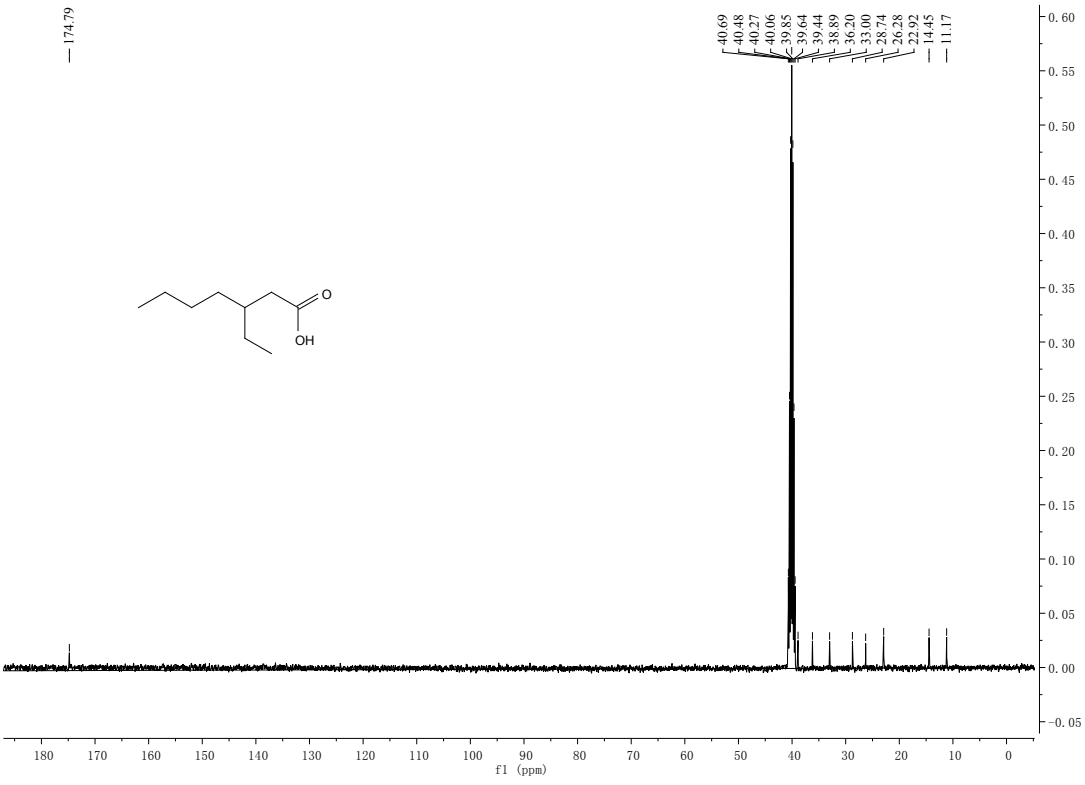
^{1}H NMR for compound **2n**



^{13}C NMR for compound **2n**

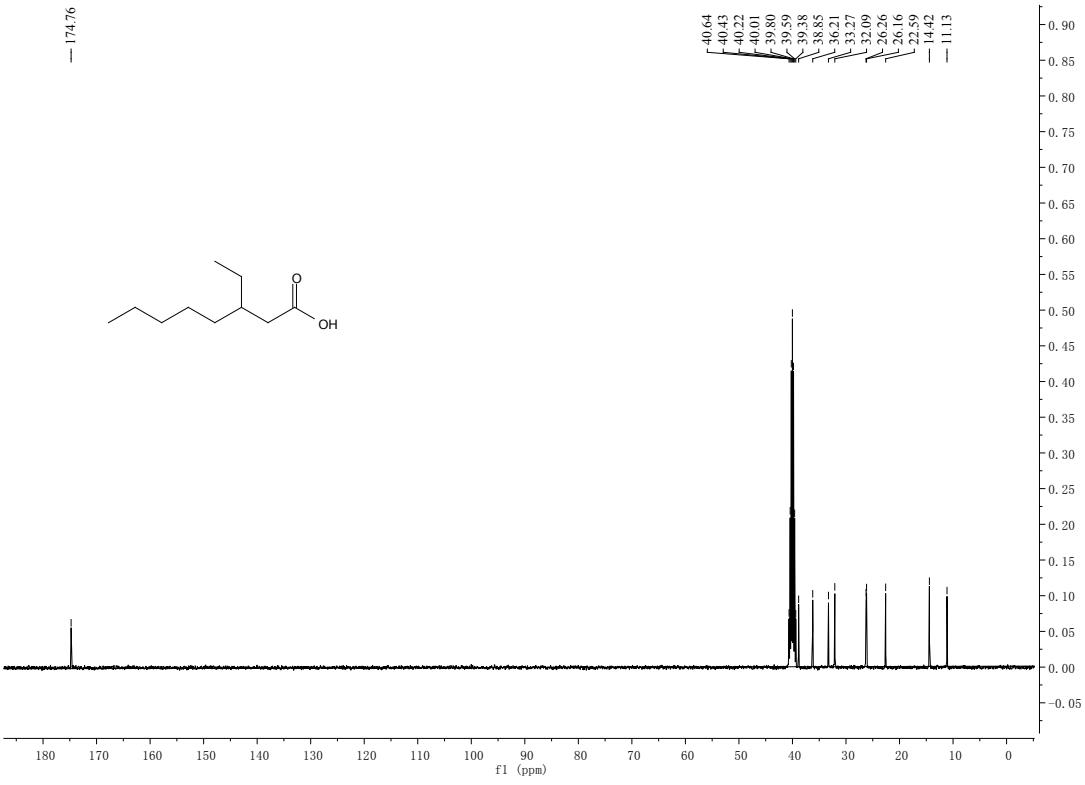


^{1}H NMR for compound **2o**

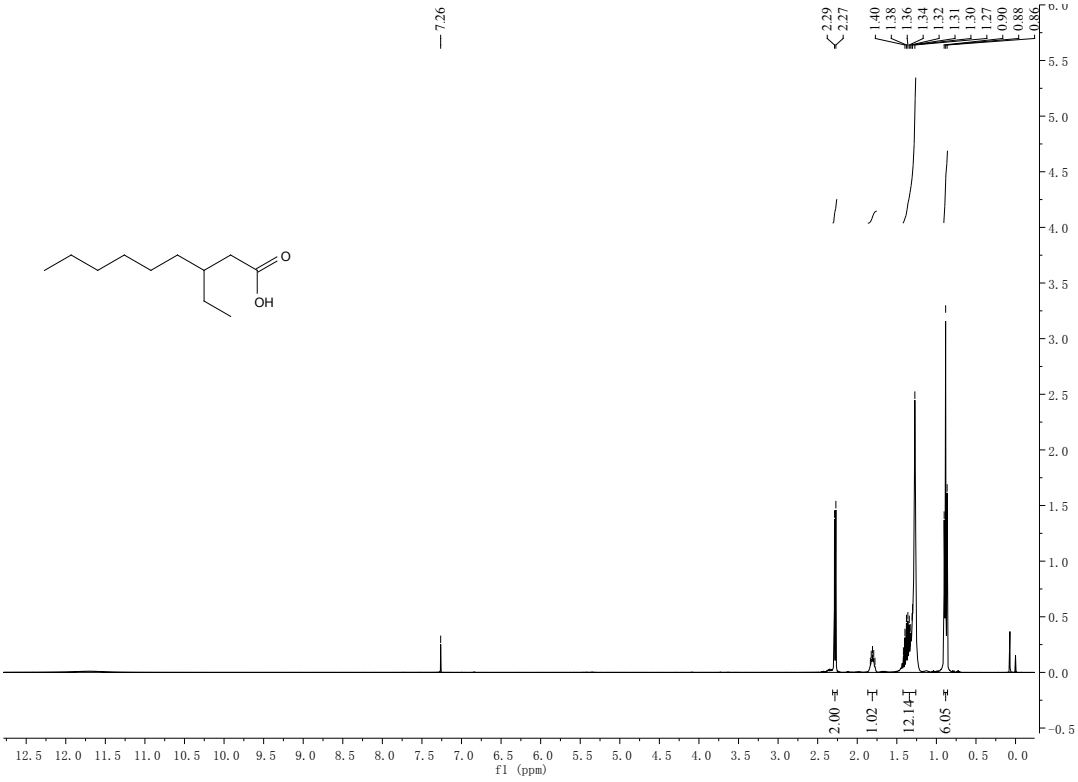


^{13}C NMR for compound **2o**

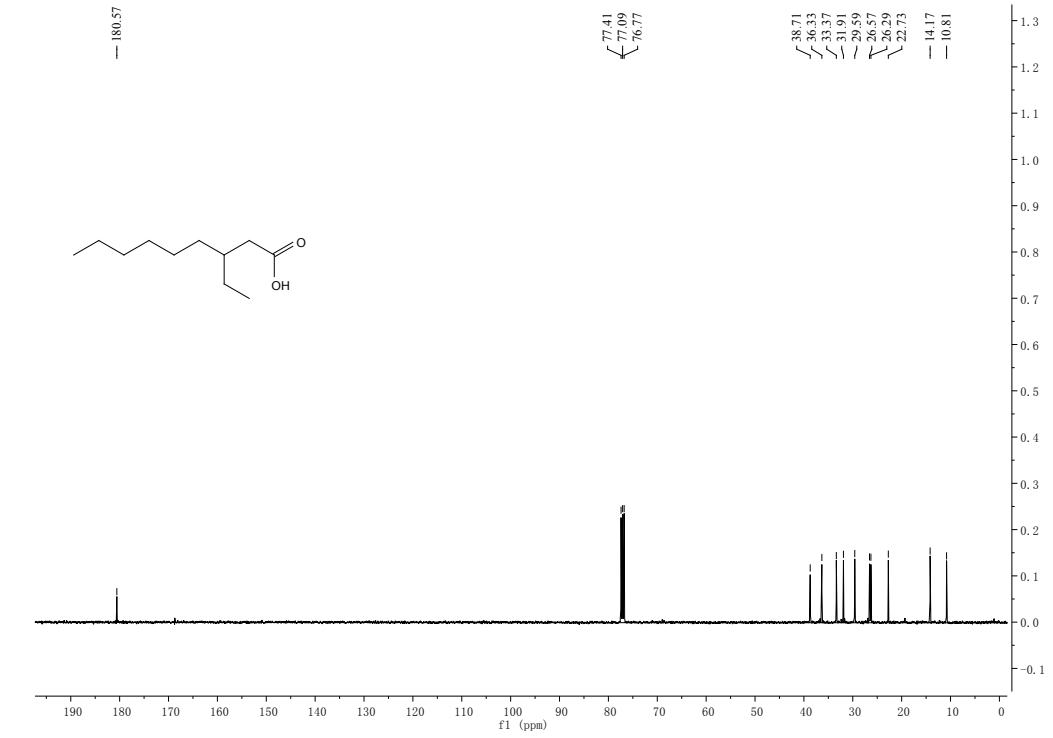
^{1}H NMR for compound **2p**



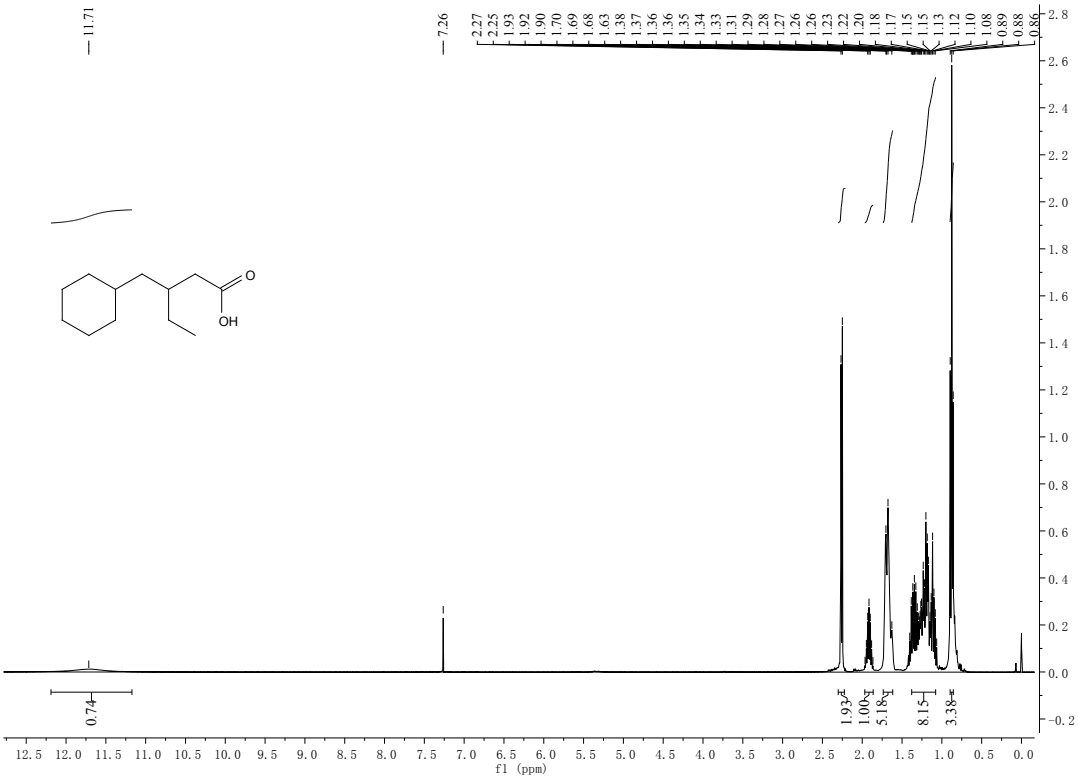
^{13}C NMR for compound **2p**



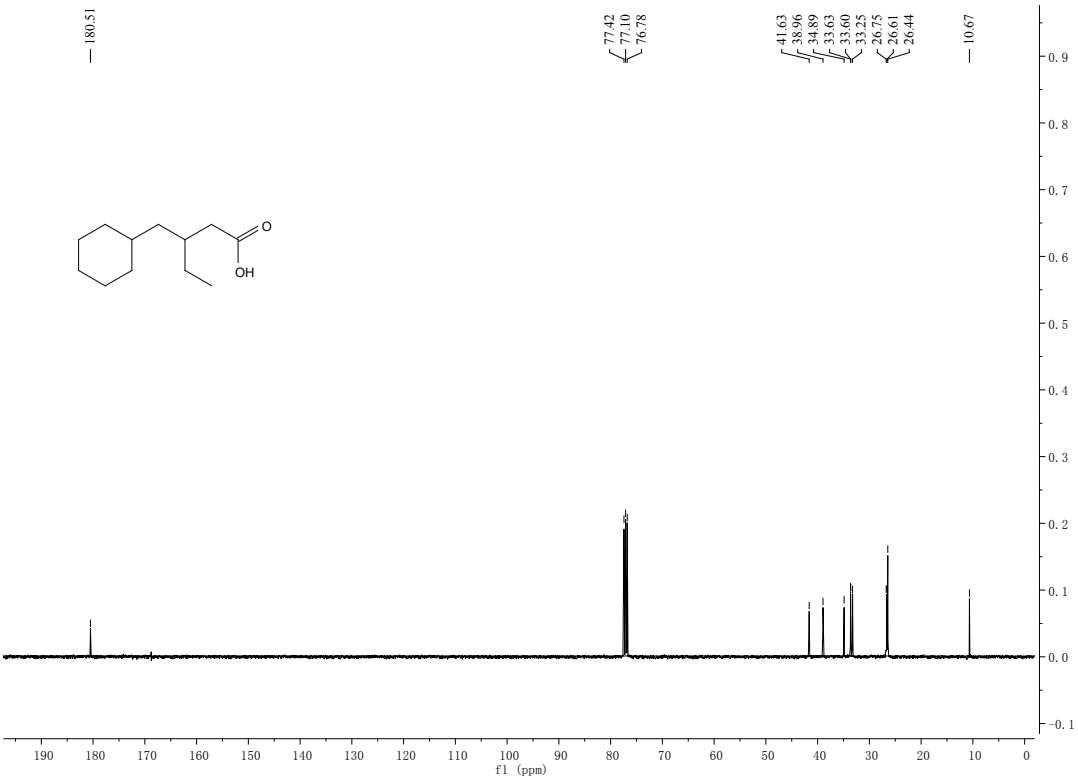
^{1}H NMR for compound **2q**



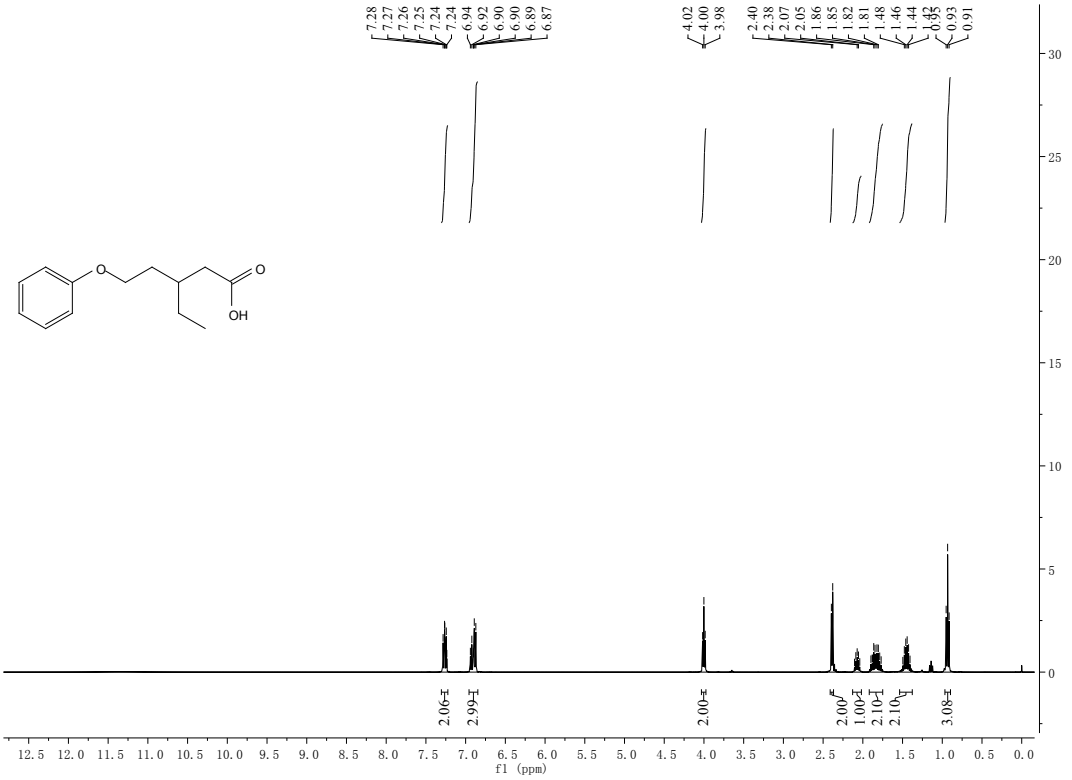
^{13}C NMR for compound **2q**



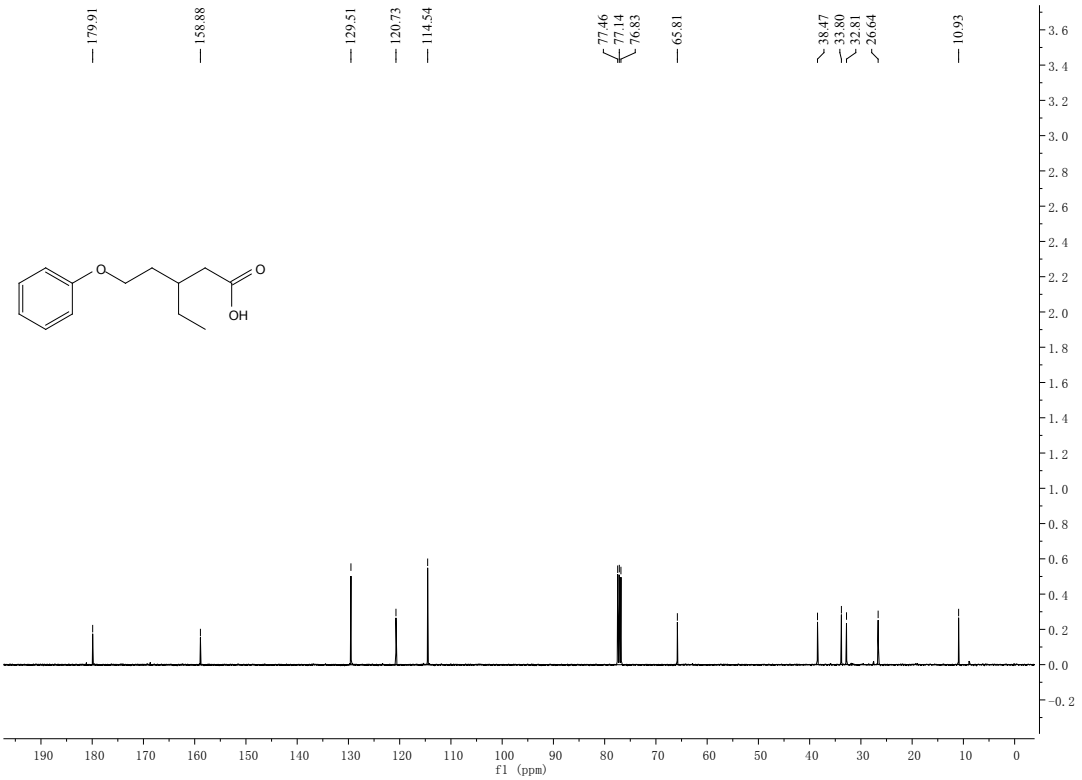
^{1}H NMR for compound **2r**



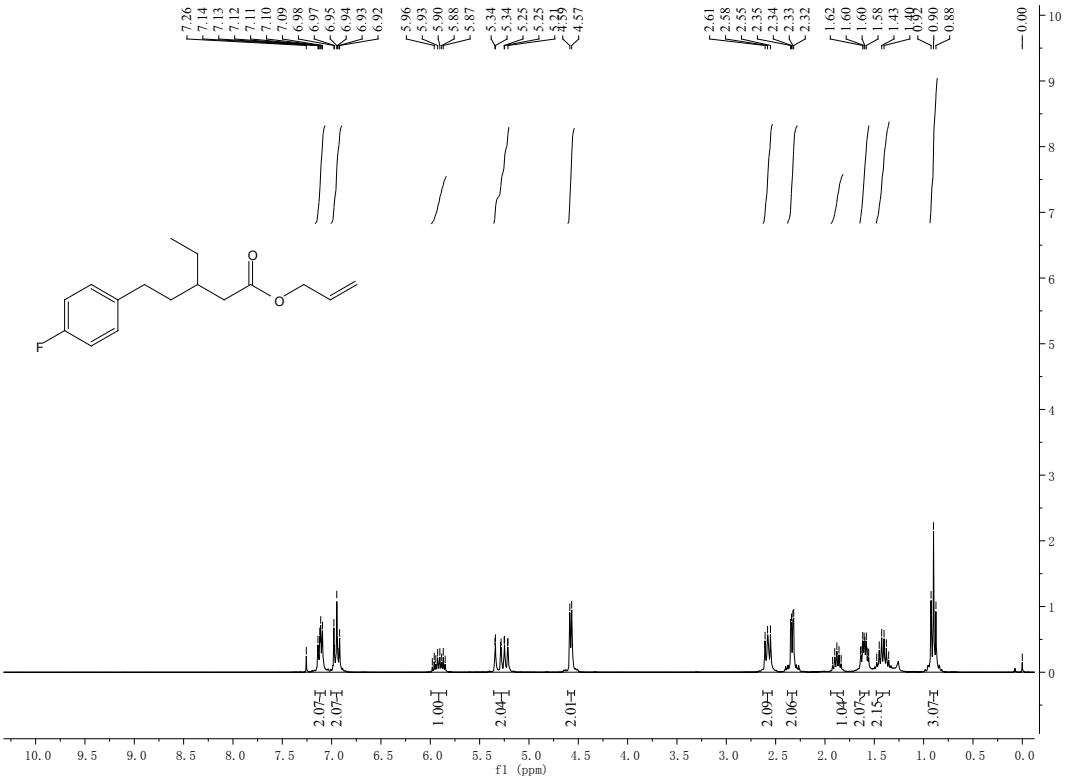
^{13}C NMR for compound **2r**



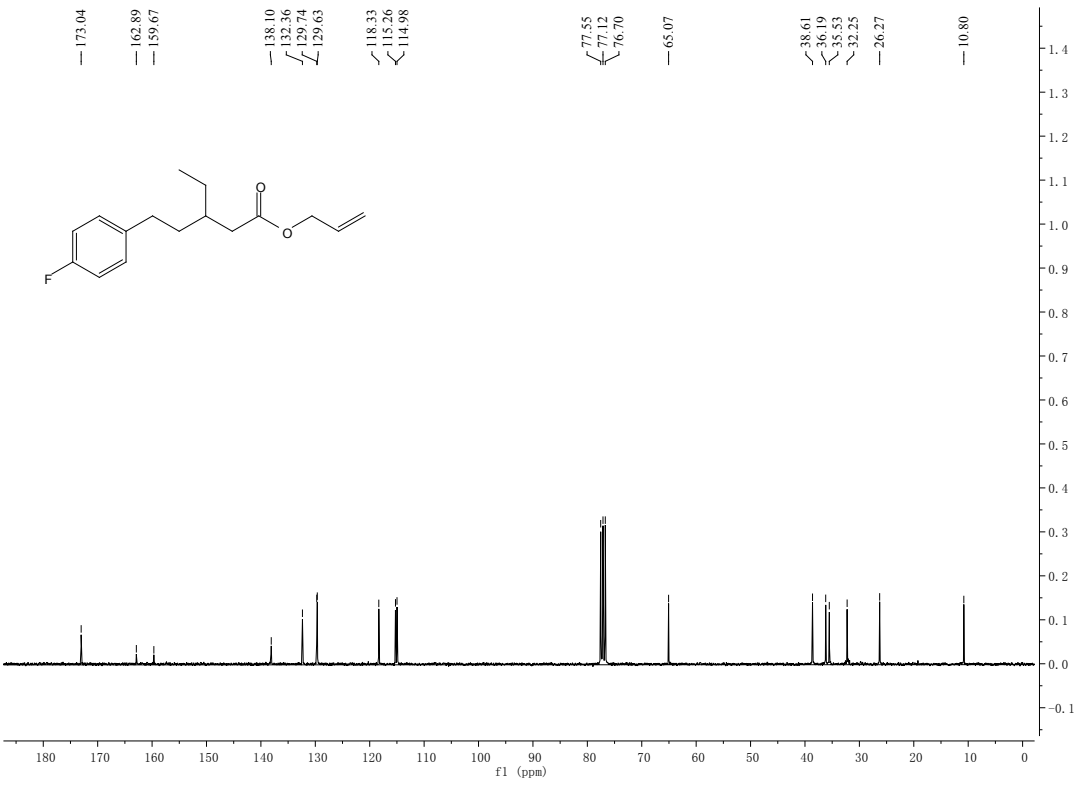
^{1}H NMR for compound **2s**



^{13}C NMR for compound **2s**



^{1}H NMR for compound **3n**



^{13}C NMR for compound **3n**