

Supporting Information

Copper Catalyzed Oxygen Assisted C(CNOH)-C(alkyl) Bond Cleavage: A Facile Conversion of Aryl/Aralkyl/Vinyl Ketones to Aromatic Acids

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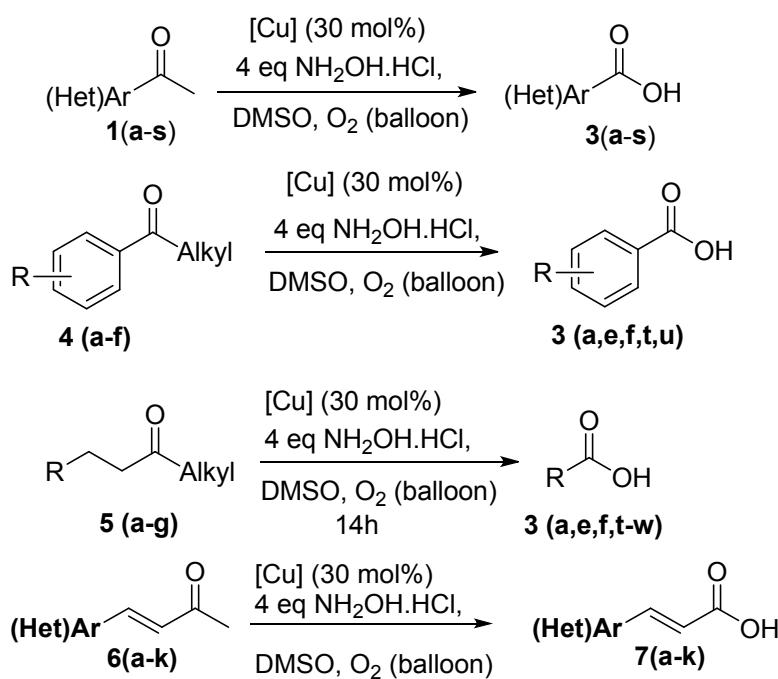
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1.0. General Information:

All reactions were carried out in oven-dried glassware. IR spectra were recorded on FT-IR spectrometer (KBr) and reported in reciprocal centimetres (cm^{-1}). ^1H NMR spectra were recorded at 300 MHz and ^{13}C NMR at 75 MHz. For ^1H NMR, tetramethylsilane (TMS) was used as internal standard ($\delta = 0$) and the values are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad singlet, dt = doublet of triplet), and the coupling constants in Hz. For ^{13}C NMR, CDCl_3 ($\delta = 77.23$) was used as internal standard and spectra were obtained with complete proton decoupling. Melting points were measured on micro melting point apparatus. The precursors, (E)-4-arylbut-3-en-2-ones and 4-arylbutan-2-ones are prepared according to reported procedures. Commercially available acetophenones, hydroxylamine hydrochloride, CuI and DMSO were used without further purification.

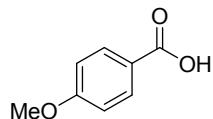
2.0. General procedure for the synthesis of Benzoic acids and Cinnamic acids:



A mixture of ketone [**1**, **4**, **5**, **6**] (1 mmol, 1 eq.), hydroxylamine hydrochloride (4 mmol, 4 eq.) and CuI (30 mol %) in dimethyl sulfoxide (10 mL) was stirred at 100 °C under oxygen atmosphere for an appropriate time. After completion of the reaction, as indicated by TLC, the mixture was diluted with water and extracted with EtOAc (3×20 mL). The extract was

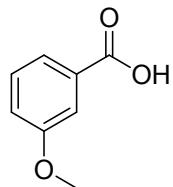
washed with brine, dried over Na_2SO_4 and evaporated, and the crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc) to obtain the product.

4-methoxybenzoic acid¹



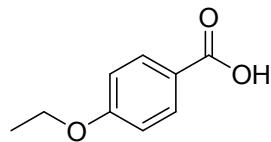
144 mg of **3a** was obtained from 150 mg (1 mmol) of **1a**. 95% yield; White solid; $R_f = 0.4$ (EtOAc/hexane = 3/7); m.p. 180-182 °C; ¹H NMR (300 MHz, CDCl_3) δ: 8.05 (d, $J = 8.92$ Hz, 2H), 6.95 (d, $J = 8.89$ Hz, 2H), 3.86 (s, 3H); ¹³C NMR (75 MHz, $\text{CDCl}_3 + \text{DMSO-d}$) δ: 167.5, 162.6, 131.1, 122.7, 112.9, 54.9; IR (KBr) v: 3094, 1685.8, 1215.4, 757.7, 669.1 cm^{-1} ; MS (EI) m/z 151 [M-1]⁺.

3-methoxybenzoic acid¹



136.5 mg of **3b** was obtained from 150 mg (1 mmol) of **1b**. 90% yield; White solid; $R_f = 0.4$ (EtOAc/hexane = 3/7); m.p. 104-106 °C; ¹H NMR (300 MHz, CDCl_3) δ: 7.75-7.72 (m, 1H), 7.64-7.63 (m, 1H), 7.39 (t, $J = 7.87$ Hz, 1H), 7.18-7.15 (m, 1H), 3.87 (s, 3H); ¹³C NMR (75 MHz, DMSO-d) δ: 167.0, 159.2, 132.1, 129.6, 121.5, 118.8, 113.8, 55.2; IR (KBr) v: 3394.3, 3020.9, 1693.2, 1411.7, 1216.1, 760.0, 670.0 cm^{-1} ; MS (EI) m/z 151 [M-1]⁺.

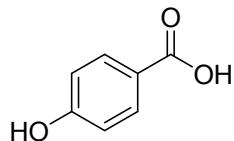
4-ethoxybenzoic acid²



154 mg of **3c** was obtained from 164 mg (1 mmol) of **1c**. 93% yield; White solid; $R_f = 0.4$ (EtOAc/hexane = 3/7); m.p. 195-197 °C; ¹H NMR (300 MHz, $\text{CDCl}_3 + \text{DMSO-d}$) δ: 7.72-7.70 (m, 2H), 6.65-6.63 (m, 2H), 3.83 (q, $J = 13.94, 6.99$ Hz, 2H), 1.17 (t, $J = 6.97$ Hz, 3H); ¹³C NMR (75 MHz, $\text{CDCl}_3 + \text{DMSO-d}$) δ: 167.2, 161.7, 130.8, 122.3, 113.1, 62.8, 13.9; IR

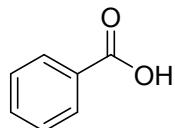
(KBr) v: 3402.7, 3021.0, 1603.9, 1300.0, 1216.0, 1169.6, 761.5, 669.8 cm⁻¹; MS (EI) *m/z* 165 [M-1]⁺.

4-hydroxybenzoic acid³



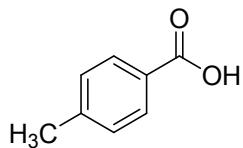
38.5 mg of **3d** was obtained from 136 mg (1 mmol) of **1d**. 28% yield; White solid; *R_f* = 0.2 (EtOAc/hexane = 9/1); m.p. 212-214 °C; ¹H NMR (300 MHz, DMSO-d) δ: 10.23 (brs, 1H), 7.78 (d, *J* = 8.67 Hz, 2H), 6.81 (d, *J* = 8.63 Hz, 2H); ¹³C NMR (75 MHz, DMSO-d) δ: 167.1, 161.5, 131.5, 121.5, 115.1; IR (KBr) v: 3399.6, 3019.4, 1654.0, 1523.2, 1215.5, 757.5, 669.1 cm⁻¹; MS (EI) *m/z* 137 [M-1]⁺.

Benzoic acid¹

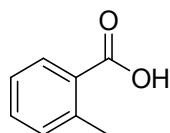


75.5 mg of **3e** was obtained from 120 mg (1 mmol) of **1e**. 62% yield; White solid; *R_f* = 0.4 (EtOAc/hexane = 3/7); m.p. 120-122 °C; ¹H NMR (300 MHz, CDCl₃) δ: 8.13 (dd, *J* = 8.11, 1.01 Hz, 2H), 7.64-7.61 (m, 1H), 7.49 (t, *J* = 7.84 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃) δ: 172.3, 134.0, 130.4, 128.7; IR (KBr) v: 3020.6, 1687.9, 1326.5, 1293.1, 934.8, 708.9 cm⁻¹; MS (EI) *m/z* 121 [M-1]⁺.

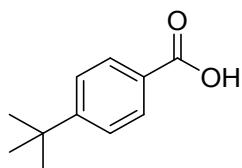
4-methylbenzoic acid⁴



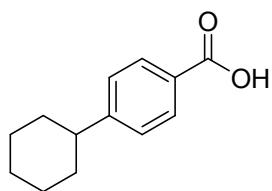
126 mg of **3f** was obtained from 134 mg (1 mmol) of **1f**. 93% yield; White solid; *R_f* = 0.5 (EtOAc/hexane = 3/7); m.p. 180-182 °C; ¹H NMR (300 MHz, CDCl₃) δ: 8.01 (d, *J* = 8.31 Hz, 2H), 7.27 (d, *J* = 7.94 Hz, 2H), 2.43 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ: 172.3, 144.8, 130.4, 129.4, 125.7, 21.9; IR (KBr) v: 3019.5, 1692.6, 1215.5, 757.8, 669.1 cm⁻¹; MS (EI) *m/z* 135 [M-1]⁺.

2-methylbenzoic acid⁵

118 mg of **3g** was obtained from 134 mg (1 mmol) of **1g**. 87% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 104-105 °C; ¹H NMR (300 MHz, CDCl₃ + DMSO-d) δ: 7.85-7.83 (m, 1H), 7.31-7.28 (m, 1H), 7.15-7.11 (m, 1H), 2.52 (s, 3H); ¹³C NMR (75 MHz, CDCl₃ + DMSO-d) δ: 170.4, 140.1, 131.7, 131.5, 130.8, 130.0, 125.5, 21.7; IR (KBr) v: 3019.5, 1692.6, 1215.5, 757.8, 669.1 cm⁻¹; MS (EI) *m/z* 135 [M-1]⁺.

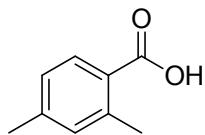
4-(*tert*-butyl) benzoic acid⁵

162 mg of **3h** was obtained from 176 mg (1 mmol) of **1h**. 91% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 167-169 °C; ¹H NMR (300 MHz, CDCl₃) δ: 8.06 (d, *J* = 8.55 Hz, 2H), 7.50 (d, *J* = 8.58 Hz, 2H), 1.36 (s, 9H); ¹³C NMR (75 MHz, CDCl₃) δ: 172.7, 157.8, 130.3, 126.8, 125.7, 35.4, 31.3; IR (KBr) v: 3408.8, 3019.7, 1633.1, 1215.9, 1069.3, 769.5, 669.2 cm⁻¹; MS (EI) *m/z* 177 [M-1]⁺.

4-cyclohexylbenzoic acid⁶

138.5 mg of **3i** was obtained from 202 mg (1 mmol) of **1i**. 68% yield; Light Gray solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 136-137 °C; ¹H NMR (300 MHz, CDCl₃) δ: 8.01 (d, *J* = 8.19 Hz, 2H), 7.28 (d, *J* = 8.17 Hz, 2H), 2.59-2.52 (m, 1H), 1.85-1.73 (m, 5H), 1.49-1.24 (m, 5H); ¹³C NMR (75 MHz, CDCl₃) δ: 171.9, 154.6, 130.5, 127.2, 127.0, 45.0, 34.3, 26.9, 26.2; IR (KBr) v: 3407.1, 3019.2, 1654.1, 1215.9, 768.8, 668.5 cm⁻¹.

2, 4-dimethylbenzoic acid⁷



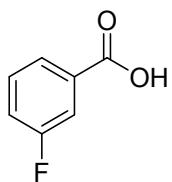
135 mg of **3j** was obtained from 148 mg (1 mmol) of **1j**. 90% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 120-122 °C; ^1H NMR (300 MHz, CDCl_3) δ: 7.98-7.97 (m, 1H), 7.09-7.08 (m, 2H), 2.63 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ: 173.2, 143.8, 141.7, 132.9, 132.0, 126.8, 125.6, 22.3, 21.6; IR (KBr) v: 3020.5, 1692.6, 1216.5, 757.5, 669.1 cm^{-1} ; MS (EI) m/z 149 [M-1]⁺.

3-bromobenzoic acid⁸



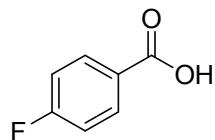
155 mg of **3k** was obtained from 197 mg (1 mmol) of **1k**. 78% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 154-156 °C; ^1H NMR (300 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$) δ: 7.89-7.88 (m, 1H), 7.71-7.68 (m, 1H), 7.41-7.38 (m, 1H), 7.08-7.04 (m, 1H); ^{13}C NMR (75 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$) δ: 166.3, 134.9, 132.7, 132.1, 129.5, 127.8, 121.6; IR (KBr) v: 3400.7, 3019.7, 1692.5, 1308.8, 1216.5, 760.3, 669.4 cm^{-1} ; MS (EI) m/z 198 [M-1]⁺.

3-fluorobenzoic acid⁴



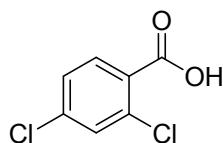
91 mg of **3l** was obtained from 138 mg (1 mmol) of **1l**. 65% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 120-122 °C; ^1H NMR (300 MHz, CDCl_3) δ: 10.42 (brs, 1H), 7.94-7.91 (m, 1H), 7.82-7.78 (m, 1H), 7.49-7.44 (m, 1H), 7.35-7.30 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ: 171.4, 164.0, 161.5, 131.7, 131.6, 130.48, 130.41, 126.24, 126.22, 121.3, 121.1, 117.4, 117.2; IR (KBr) v: 3397.2, 3021.3, 1694.1, 1413.7, 1216.0, 759.7 cm^{-1} ; MS (EI) m/z 139 [M-1]⁺.

4-fluorobenzoic acid⁴



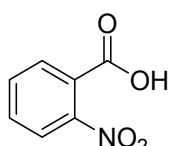
99.5 mg of **3m** was obtained from 138 mg (1 mmol) of **1m**. 71% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 182-184 °C; ^1H NMR (300 MHz, CDCl_3) δ: 8.16-8.13 (m, 2H), 7.17-7.14 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ: 171.3, 168.2, 164.9, 133.1, 133.0, 125.7, 116.1, 115.8; IR (KBr) v: 3408.3, 3019.5, 2400.1, 1601.6, 1420.2, 1215.4, 1069.0, 758.0, 669.0 cm^{-1} ; MS (EI) m/z 139 [M-] $^+$.

2, 4-dichlorobenzoic acid⁹



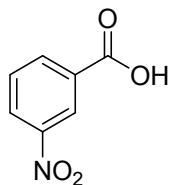
144 mg of **3n** was obtained from 188 mg (1 mmol) of **1n**. 60% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 155-157 °C; ^1H NMR (300 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$) δ: 9.05 (brs, 1H), 7.81-7.78 (m, 1H), 7.389-7.381 (m, 1H), 7.25-7.20 (m, 1H); ^{13}C NMR (75 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$) δ: 166.1, 137.3, 134.3, 132.3, 130.3, 128.8, 126.5; IR (KBr) v: 3399.4, 1698.7, 1588.1, 1384.2, 1216.3, 1051.9, 771.0, 668.5 cm^{-1} ; MS (EI) m/z 189 [M-1] $^+$.

2-nitrobenzoic acid¹⁰



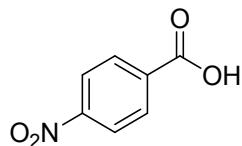
113.5 mg of **3o** was obtained from 165 mg (1 mmol) of **1o**. 68% yield; Light yellow solid; $R_f = 0.45$ (EtOAc/hexane = 3/7); m.p. 145-147 °C; ^1H NMR (300 MHz, DMSO-d₆) δ: 13.85 (brs, 1H), 7.98-7.96 (m, 1H), 7.87-7.85 (m, 1H), 7.81-7.74 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d₆) δ: 165.9, 148.4, 133.1, 132.4, 129.8, 127.2, 123.7; IR (KBr) v: 3021.2, 1676.4, 1382.1, 1216.1, 765.5 cm^{-1} ; MS (EI) m/z 166 [M-1] $^+$.

3-nitrobenzoic acid⁵



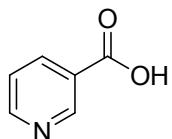
117 mg of **3p** was obtained from 165 mg (1 mmol) of **1p**. 70% yield; Lightyellow solid; R_f = 0.45 (EtOAc/hexane = 3/7); m.p. 138-140 °C; ^1H NMR (300 MHz, DMSO-d) δ : 8.59 (brs, 1H), 8.46-8.43 (m, 1H), 8.34-8.29 (m, 1H), 7.79 (t, J = 7.89 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d) δ : 165.5, 147.8, 135.3, 130.5, 127.3, 123.6; IR (KBr) v: 3021.7, 1705.9, 1352.1, 1288.4, 1215.7, 759.9, 669.5 cm^{-1} ; MS (EI) m/z 166 [M-1] $^+$.

4-nitrobenzoic acid⁴



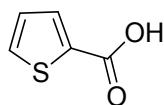
120 mg of **3q** was obtained from 165 mg (1 mmol) of **1q**. 72% yield; Light yellow solid; R_f = 0.45 (EtOAc/hexane = 3/7); m.p. 232-234 °C; ^1H NMR (300 MHz, DMSO-d) δ : 8.72 (d, J = 8.80 Hz, 2H), 8.57 (d, J = 8.80 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d) δ : 165.8, 150.0, 136.3, 130.6, 123.7; IR (KBr) v: 3019.6, 1654.5, 1384.6, 1215.5, 1069.8, 757.3, 669.1 cm^{-1} ; MS (EI) m/z 166 [M-1] $^+$.

Nicotinic acid¹¹



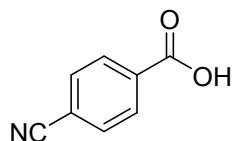
83.5 mg of **3r** was obtained from 121 mg (1 mmol) of **1r**. 68% yield; White solid; R_f = 0.3 (EtOAc/hexane = 1/1); m.p. 230-234 °C; ^1H NMR (300 MHz, DMSO-d) δ : 13.41 (brs, 1H), 9.08-9.07 (m, 1H), 8.785 (dd, J = 4.80, 1.56 Hz, 1H), 8.265 (dt, J = 7.93, 1.97 Hz, 1H), 7.56-7.52 (m, 1H); ^{13}C NMR (75 MHz, DMSO-d) δ : 166.2, 153.2, 150.2, 136.9, 126.5, 123.7; IR (KBr) v: 3401.3, 3019.3, 2400.0, 1654.1, 1215.4, 757.2, 669.0 cm^{-1} ; MS (EI) m/z 122 [M-1] $^+$.

Thiophene-2-carboxylic acid¹



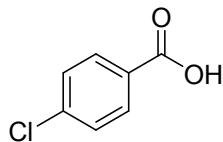
115 mg of **3s** was obtained from 126 mg (1 mmol) of **1s**. 90% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 122-125 °C; ^1H NMR (300 MHz, CDCl_3) δ : 7.87 (dd, $J = 3.75, 1.23$ Hz, 1H), 7.63 (dd, $J = 4.95, 1.22$ Hz, 1H), 7.13-7.12 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ : 167.7, 135.2, 134.2, 133.0, 128.2; IR (KBr) v: 3019.6, 1654.5, 1384.6, 1215.5, 1069.8, 757.3, 669.1 cm^{-1} ; MS (EI) m/z 128 [M^+].

4-cyanobenzoic acid¹²



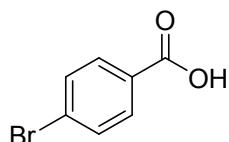
70 mg of **3t** was obtained from 148 mg (1 mmol) of **1t**. 48% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 215-127 °C; ^1H NMR (300 MHz, DMSO-d) δ : 8.09-8.07 (m, 2H), 7.99-7.96 (m, 1H); ^{13}C NMR (75 MHz, DMSO-d) δ : 166.0, 134.8, 132.6, 129.9, 118.1, 115.0; IR (KBr) v: 3401.1, 1711.1, 1423.3, 1215.8, 769.6, 670.0 cm^{-1} ; MS (EI) m/z 148 [M^+].

4-chlorobenzoic acid¹



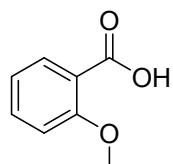
112 mg of **3u** was obtained from 154 mg (1 mmol) of **4d**. 72% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 235-237 °C; ^1H NMR (300 MHz, CDCl_3) δ : 7.75-7.72 (m, 2H), 7.42-7.39 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ : 168.4, 138.5, 131.9, 129.1, 129.0; IR (KBr) v: 3400.5, 3019.5, 1647.0, 1215.4, 757.9, 669.1 cm^{-1} ; MS (EI) m/z 155 [$\text{M}-1$] $^+$.

4-bromobenzoic acid¹



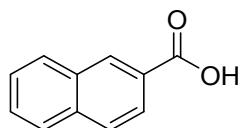
159 mg of **3v** was obtained from 197 mg (1 mmol) of **4e**. 80% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 248-250 °C; ^1H NMR (300 MHz, DMSO-d) δ: 13.17 (brs, 1H), 7.87-7.85 (m, 2H), 7.71-7.69 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d) δ: 166.5, 131.6, 131.2, 130.0, 126.8; IR (KBr) v: 3395.8, 3021.1, 1676.2, 1425.1, 1296.8, 1215.6, 761.7 cm⁻¹; MS (EI) *m/z* 198 [M-1]⁺.

2-methoxybenzoic acid¹



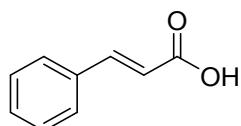
136.5 mg of **3w** was obtained from 150 mg (1 mmol) of **5b**. 90% yield; White solid; $R_f = 0.4$ (EtOAc/hexane = 3/7); m.p. 100-102 °C; ^1H NMR (300 MHz, CDCl₃) δ: 7.74-7.72 (m, 1H), 7.64-7.63 (m, 1H), 7.39 (t, *J* = 7.83 Hz, 1H), 7.18-7.15 (m, 1H), 3.87 (s, 3H); ^{13}C NMR (75 MHz, CDCl₃) δ: 172.5, 159.8, 130.8, 129.7, 122.9, 120.7, 114.6, 55.6; IR (KBr) v: 3020.6, 1692.9, 1287.9, 1216.5, 759.7, 670.0 cm⁻¹; MS (EI) *m/z* 151 [M-1]⁺.

2-naphthoic acid⁵



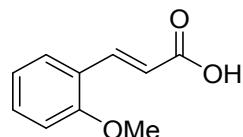
85.5 mg of **3x** was obtained from 198 mg (1 mmol) of **5f**. 49.7% yield; White solid; $R_f = 0.3$ (EtOAc/hexane = 3/7); m.p. 183-185 °C; ^1H NMR (300 MHz, CDCl₃) δ: 8.72 (s, 1H), 8.12 (dd, *J* = 8.64, 1.60 Hz, 1H), 8.00 (d, *J* = 8.09 Hz, 1H), 7.92 (t, *J* = 8.64 Hz, 2H), 7.65-7.62 (m, 1H), 7.59-7.56 (m, 1H); ^{13}C NMR (75 MHz, CDCl₃) δ: 171.5, 136.1, 132.6, 132.3, 129.7, 128.8, 128.5, 128.0, 127.0, 125.6; IR (KBr) v: 3066.3, 1686.2, 1216.4, 769.6, 669.6 cm⁻¹; MS (EI) *m/z* 171 [M-1]⁺.

cinnamic acid¹



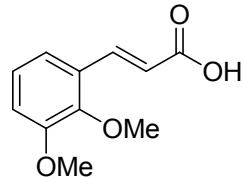
7a was obtained from 146 mg (1 mmol) of **6a**. 58% yield; White solid; $R_f = 0.3$ (EtOAc/hexane = 3/7); m.p. 129-130 °C; ^1H NMR (300 MHz, DMSO-d) δ: 7.70-7.66 (m, 2H), 7.59 (d, $J = 16.06$ Hz, 1H), 7.42 - 7.40 (m, 3H), 6.53 (d, $J = 15.93$ Hz, 1H); ^{13}C NMR (75 MHz, CDCl₃) δ: 172.7, 147.3, 134.2, 130.9, 129.1, 128.5, 117.5; IR (KBr) v: 3396.7, 3022.0, 1684.6, 1216.4, 765.3, 672.7 cm⁻¹; MS (EI) *m/z* 147 [M-1]⁺.

(E)-3-(2-methoxyphenyl) acrylic acid¹³



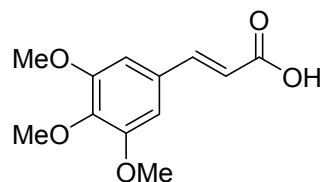
85 mg of **7b** was obtained from 176 mg (1 mmol) of **6b**. 48% yield; White solid; $R_f = 0.5$ (EtOAc/hexane = 3/7); m.p. 178-180 °C; ^1H NMR (300 MHz, CDCl₃) δ: 8.11 (d, $J = 16.10$ Hz, 1H), 7.55-7.53 (m, 1H), 7.41-7.35 (m, 1H), 7.01-6.92 (m, 2H), 6.56 (d, $J = 16.18$ Hz, 1H), 3.91 (s, 3H); ^{13}C NMR (75 MHz, CDCl₃) δ: 167.9, 157.4, 138.7, 131.0, 127.9, 120.2, 118.8, 110.8, 55.07; IR (KBr) v: 3388.0, 1620.9, 1400.9, 1249.3, 1218.6, 1069.0, 771.5 cm⁻¹; MS (EI) *m/z* 177 [M-1]⁺.

(E)-3-(2, 3-dimethoxyphenyl) acrylic acid¹⁴



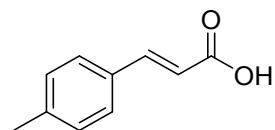
106 mg of **7c** was obtained from 206 mg (1 mmol) of **6c**. 51% yield; White solid; $R_f = 0.3$ (EtOAc/hexane = 3/7); m.p. 178-180 °C; ^1H NMR (300 MHz, CDCl₃) δ: 8.11 (d, $J = 15.88$ Hz, 1H), 7.20-7.18 (dd, $J = 7.91, 1.19$ Hz, 1H), 7.08 (t, $J = 8.09$ Hz, 1H), 6.98-6.96 (dd, $J = 8.11, 1.24$ Hz, 1H), 6.51 (d, $J = 16.10$ Hz, 1H), 3.89 (s, 3H), 3.88 (s, 3H); ^{13}C NMR (75 MHz, CDCl₃) δ: 172.2, 153.3, 148.9, 141.9, 128.4, 124.4, 118.7, 114.6, 61.6, 56.1; IR (KBr) v: 3400.0, 1626.3, 1410.6, 1253.3, 1216.9, 1069.9, 776.5 cm⁻¹; MS (EI) *m/z* 207 [M-1]⁺.

(E)-3-(3, 4, 5-trimethoxyphenyl) acrylic acid¹⁵



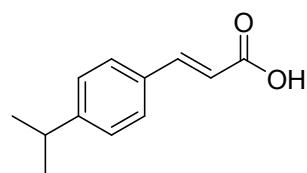
154.5 mg of **7d** was obtained from 236 mg (1 mmol) of **6d**. 65% yield; Light yellow solid; R_f = 0.3 (EtOAc/hexane = 3/7); m.p. 168-170 °C; ^1H NMR (300 MHz, CDCl_3) δ: 7.71 (d, J = 15.70 Hz, 1H), 6.79 (s, 2H), 6.37 (d, J = 15.74 Hz, 1H), 3.91 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ: 172.4, 153.6, 147.2, 140.7, 129.6, 116.6, 105.7, 61.1, 56.3; IR (KBr) v: 3401.5, 3019.3, 1631.3, 1283.0, 1216.2, 1068.9, 770.4, 669.1 cm^{-1} ; MS (EI) m/z 239 [M $^+$].

(E)-3-(p-tolyl) acrylic acid¹⁶

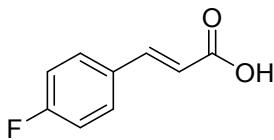


98.5 mg of **7e** was obtained from 160 mg (1 mmol) of **6e**. 61% yield; White solid; R_f = 0.3 (EtOAc/hexane = 3/7); m.p. 192-194 °C; ^1H NMR (300 MHz, CDCl_3) δ: 7.77 (d, J = 15.94 Hz, 1H), 7.45 (d, J = 7.97 Hz, 2H), 7.21 (d, J = 7.97 Hz, 2H), 6.41 (d, J = 16.00 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ: 172.6, 147.3, 141.5, 131.5, 129.9, 128.6, 116.3, 21.7; IR (KBr) v: 3387.5, 2920.1, 1628.7, 1286.6, 1217.6, 771.6, 670.0 cm^{-1} ; MS (EI) m/z 161 [M-1] $^+$.

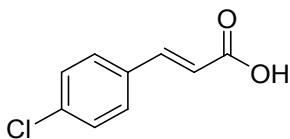
(E)-3-(4-isopropylphenyl) acrylic acid¹⁷



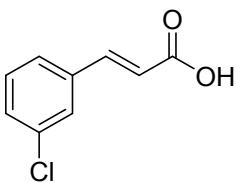
115.5 mg of **7f** was obtained from 188 mg (1 mmol) of **6f**. 61% yield; White solid; R_f = 0.3 (EtOAc/hexane = 3/7); m.p. 162-164 °C; ^1H NMR (300 MHz, CDCl_3) δ: 7.78 (d, J = 16.00 Hz, 1H), 7.49 (d, J = 8.09 Hz, 2H), 7.30 (d, J = 8.01 Hz, 2H), 6.42 (d, J = 16.05 Hz, 1H), 2.98-2.89 (m, 1H), 1.28 (s, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ: 172.4, 152.4, 147.4, 131.8, 128.7, 127.3, 116.3, 34.3, 23.9; IR (KBr) v: 3390.3, 2926.2, 1628.3, 1273.6, 1216.6, 776.6, 669.6 cm^{-1} .

(E)-3-(4-fluorophenyl) acrylic acid¹⁶

89.5 mg of **7g** was obtained from 164 mg (1 mmol) of **6g**. 54% yield; White solid; $R_f = 0.3$ (EtOAc/hexane = 3/7); m.p. 200-202 °C; ^1H NMR (300 MHz, CDCl_3) δ : 7.78-7.73 (dd, $J = 15.97, 2.37$ Hz, 1H), 7.56-7.54 (m, 2H), 7.10 (t, $J = 8.54$ Hz, 2H), 6.38 (d, $J = 16.12$ Hz, 1H); ^{13}C NMR (75 MHz, $\text{CDCl}_3 + \text{DMSO-d}$) δ : 167.5, 164.7, 161.4, 142.4, 130.4, 129.6, 129.5, 118.5, 115.6, 115.3; IR (KBr) v: 3405.9, 3019.5, 1638.0, 1402.8, 1216.1, 770.7, 669.2 cm^{-1} ; MS (EI) m/z 165 [M-1]⁺.

(E)-3-(4-chlorophenyl) acrylic acid¹⁶

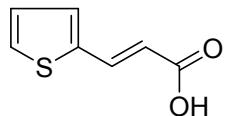
92.5 mg of **7h** was obtained from 180 mg (1 mmol) of **6h**. 51% yield; White solid; $R_f = 0.3$ (EtOAc/hexane = 3/7); m.p. 240-242 °C; ^1H NMR (300 MHz, DMSO-d) δ : 7.72 (d, $J = 8.51$ Hz, 2H), 7.58 (d, $J = 16.04$ Hz, 1H), 7.47 (d, $J = 8.48$ Hz, 2H), 6.55 (d, $J = 16.00$ Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d) δ : 167.3, 142.4, 134.6, 133.1, 129.8, 128.8, 120.0; IR (KBr) v: 3405.5, 3019.3, 1629.8, 1399.1, 1216.6, 1156.0, 1069.2, 771.1, 669.6 cm^{-1} ; MS (EI) m/z 181 [M-1]⁺.

(E)-3-(3-chlorophenyl) acrylic acid¹⁶

76.5 mg of **7i** was obtained from 180 mg (1 mmol) of **6i**. 42% yield; White solid; $R_f = 0.3$ (EtOAc/hexane = 3/7); m.p. 156-158 °C; ^1H NMR (300 MHz, CDCl_3) δ : 7.72 (d, $J = 16.03$ Hz, 1H), 7.54 (t, $J = 1.59$ Hz, 1H), 7.44-7.42 (m, 1H), 7.40-7.38 (m, 1H), 7.37-7.33 (m, 1H), 6.46 (d, $J = 15.91$ Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d) δ : 167.3, 142.3, 136.5, 133.7,

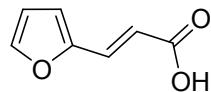
130.6, 129.8, 127.8, 126.8, 120.9; IR (KBr) v: 3389.1, 1631.3, 1402.8, 1217.8, 1068.3, 771.6, 668.9 cm⁻¹; MS (EI) *m/z* 181 [M-1]⁺.

(E)-3-(thiophen-2-yl) acrylic acid¹⁸



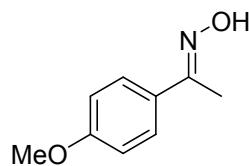
89 mg of **7j** was obtained from 152 mg (1 mmol) of **6j**. 58% yield; White solid; *R_f* = 0.3 (EtOAc/hexane = 3/7); m.p. 141-143 °C; ¹H NMR (300 MHz, DMSO-d) δ: 12.40 (brs, 1H), 7.71 (d, *J* = 15.76 Hz, 1H), 7.69 (d, *J* = 5.06 Hz, 1H), 7.50 (d, *J* = 3.45 Hz, 1H), 7.14 (dd, *J* = 4.95, 3.73 Hz, 1H), 6.17 (d, *J* = 15.68 Hz, 1H); ¹³C NMR (75 MHz, DMSO-d) δ: 167.2, 138.8, 136.7, 131.6, 129.4, 128.4, 117.4; IR (KBr) v: 3414.2, 2987.4, 2253.6, 1644.8, 1218.9, 1027.2, 756.3, 664.0 cm⁻¹; MS (EI) *m/z* 153 [M-1]⁺.

(E)-3-(furan-2-yl) acrylic acid¹⁸



84 mg of **7k** was obtained from 136 mg (1 mmol) of **6k**. 61% yield; White solid, *R_f* = 0.3 (EtOAc/hexane = 7/3); m.p. 130-132 °C; ¹H NMR (300 MHz, DMSO-d) δ: 7.83 (brs, 1H), 7.39 (d, *J* = 16.14 Hz, 1H), 6.92 (d, *J* = 3.26 Hz, 1H), 6.63-6.61 (m, 1H), 6.51 (d, *J* = 15.95 Hz, 1H); ¹³C NMR (75 MHz, DMSO-d) δ: 167.2, 150.2, 145.6, 130.7, 115.9, 115.3, 112.6; IR (KBr) v: 3412.2, 2998.2, 1637.7, 1216.0, 1053.5, 1028.4, 757.2, 667.5 cm⁻¹; MS (EI) *m/z* 137 [M-1]⁺.

3.0 Procedure for the synthesis of oxime **2a:**



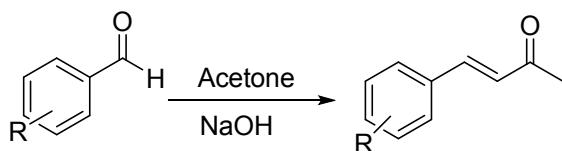
(E)-1-(4-methoxyphenyl) ethan-1-one oxime¹⁹

A mixture of acetophenone **1a** Hydroxylamine hydrochloride (2 mmol, 2 eq.) and CuI (30 mol%) in dimethyl sulfoxide (10 mL) was stirred at 50 °C under oxygen atmosphere for 2 h .

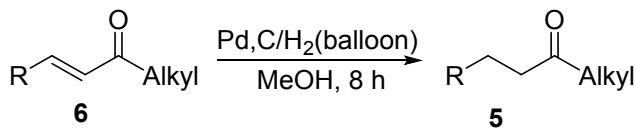
After completion of the reaction, as indicated by TLC, the mixture was diluted with water, filtered and extracted with EtOAc ($3 \times 20\text{mL}$). The extract was washed with brine, drying over Na₂SO₄ and evaporation, the crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc) to afford the product **2a**. 99% yield; White solid , $R_f = 0.6$ (EtOAc/hexane = 3/7); ¹H NMR (300 MHz, CDCl₃) δ: 8.69 (brs, 1H), 7.59-7.56 (m, 2H), 6.92-6.89 (m, 2H), 3.83 (s, 3H), 2.28 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ: 160.6, 155.7, 129.2, 127.5, 114.0, 55.5, 12.3; IR (KBr) v: 3208.1, 3077.5, 2976.8, 1621.1, 1516.2, 1298, 1025.4, 920, 752cm⁻¹; MS (EI) *m/z* 166 [M⁺].

5.0. Synthesis of starting materials:

5.1. synthesis of (E)-4-arylbut-3-en-2-ones (6) was done following the procedure given in the ref. *Chem. Med. Chem.* 2009, 4, 963-966.²⁰

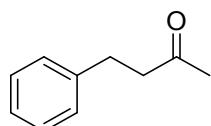


5.2. General procedure for the synthesis of 4-arylbutan-2-ones:



The substituted 4-arylbutan-2-ones were prepared by hydrogenation of the corresponding enones at room temperature at 50 psi in methanol utilizing a catalyst 5% Pd on carbon. The catalyst was filtered off and the resulting ketone was purified by column chromatography.

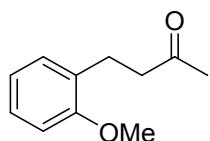
4-phenylbutan-2-one²¹



145 mg of **5a** was obtained from 146 mg (1 mmol) of **6a**. 98% yield; Colourless liquid; $R_f = 0.5$ (EtOAc/hexane = 1/9); ¹H NMR (300 MHz, CDCl₃) δ: 7.27-7.25 (m, 2H), 7.20-7.16 (m,

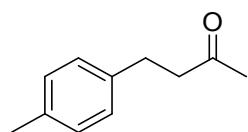
3H), 2.89 (t, $J = 7.28$ Hz, 2H), 2.75 (t, $J = 7.48$ Hz, 2H), 2.13 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ : 207.9, 141.1, 128.6, 128.4, 126.2, 45.2, 30.1, 29.9.

4-(2-methoxyphenyl) butan-2-one²²



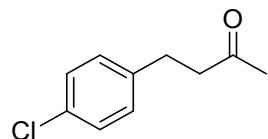
174 mg of **5b** was obtained from 176 mg (1 mmol) of **6b**. 98% yield; Colourless liquid; $R_f = 0.5$ (EtOAc/hexane = 1/9); ^1H NMR (300 MHz, CDCl_3) δ : 7.20-7.17 (m, 1H), 7.12 (dd, $J = 7.34, 1.49$ Hz, 1H), 6.88-6.83 (m, 2H), 3.81 (s, 3H), 2.88 (t, $J = 8.02$ Hz, 2H), 2.72 (t, $J = 8.06$ Hz, 2H), 2.13 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ : 208.8, 157.8, 130.1, 129.4, 127.6, 120.6, 110.3, 55.3, 43.8, 30.0, 25.1.

4-(p-tolyl) butan-2-one²³



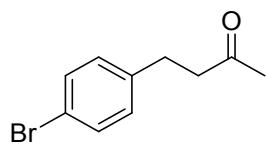
158.5 mg of **5c** was obtained from 160 mg (1 mmol) of **6e**. 98% yield; Colourless liquid, $R_f = 0.5$ (EtOAc/hexane = 1/9); ^1H NMR (300 MHz, CDCl_3) δ : 7.08-7.04 (m, 4H), 2.84 (t, $J = 7.55$ Hz, 2H), 2.71 (t, $J = 7.55$ Hz, 2H), 2.29 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ : 208.0, 138.0, 135.6, 129.2, 128.2, 45.4, 30.1, 29.4, 21.0.

4-(4-chlorophenyl) butan-2-one²³



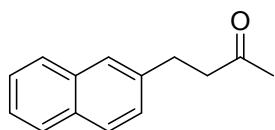
178 mg of **5d** was obtained from 180 mg (1 mmol) of **6h**. 98% yield; Colourless liquid; $R_f = 0.5$ (EtOAc/hexane = 1/9); ^1H NMR (300 MHz, CDCl_3) δ : 7.29-7.26 (m, 2H), 7.20-7.17 (m, 2H), 2.89 (t, $J = 7.74$ Hz, 2H), 2.75 (t, $J = 7.91$ Hz, 2H), 2.13 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ : 28.0, 141.1, 128.6, 128.4, 126.2, 45.2, 30.1, 29.8.

4-(4-bromophenyl) butan-2-one²³



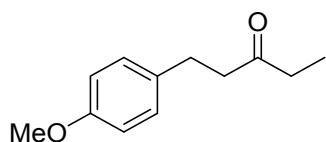
220.5 mg of **5e** was obtained from 223 mg (1 mmol) of **6l**. 98% yield; Colourless liquid; R_f = 0.5 (EtOAc/hexane = 1/9); ^1H NMR (300 MHz, CDCl_3) δ : 7.29-7.26 (m, 2H), 7.19-7.17 (m, 2H), 2.89 (t, J = 7.85 Hz, 2H), 2.76 (t, J = 7.96 Hz, 2H), 2.13 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ : 208.1, 141.1, 128.6, 128.4, 126.3, 45.3, 30.2, 29.9.

4-(naphthalen-2-yl) butan-2-one²¹



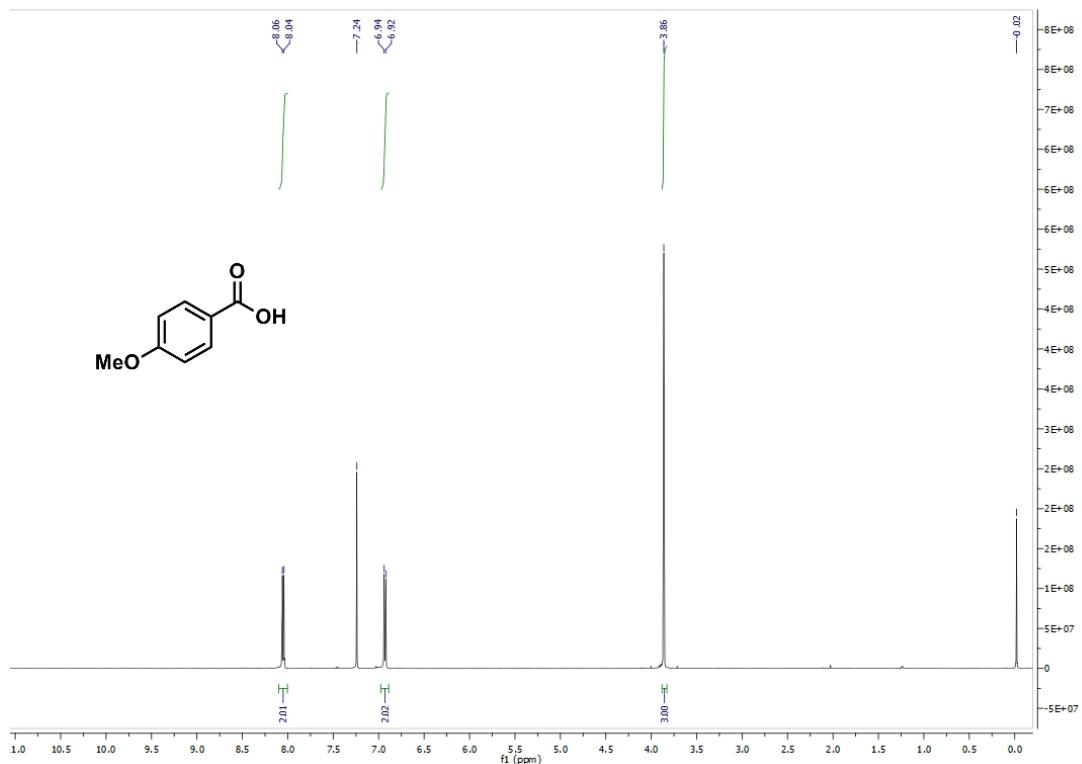
188 mg of **5f** was obtained from 196 mg (1 mmol) of **6m**. 95% yield; White solid; R_f = 0.5 (EtOAc/hexane = 1/9); ^1H NMR (300 MHz, CDCl_3) δ : 7.81-7.75 (m, 3H), 7.62 (s, 1H), 7.48-7.39 (m, 2H), 7.31 (dd, J = 8.41, 1.45 Hz, 1H), 3.06 (t, J = 7.33 Hz, 2H), 2.84 (t, J = 7.76 Hz, 2H), 2.15 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ : 208.0, 138.7, 132.2, 128.3, 127.8, 127.6, 127.2, 126.5, 126.2, 125.5, 45.2, 30.3, 30.1.

1-(4-methoxyphenyl) pentan-3-one²⁴

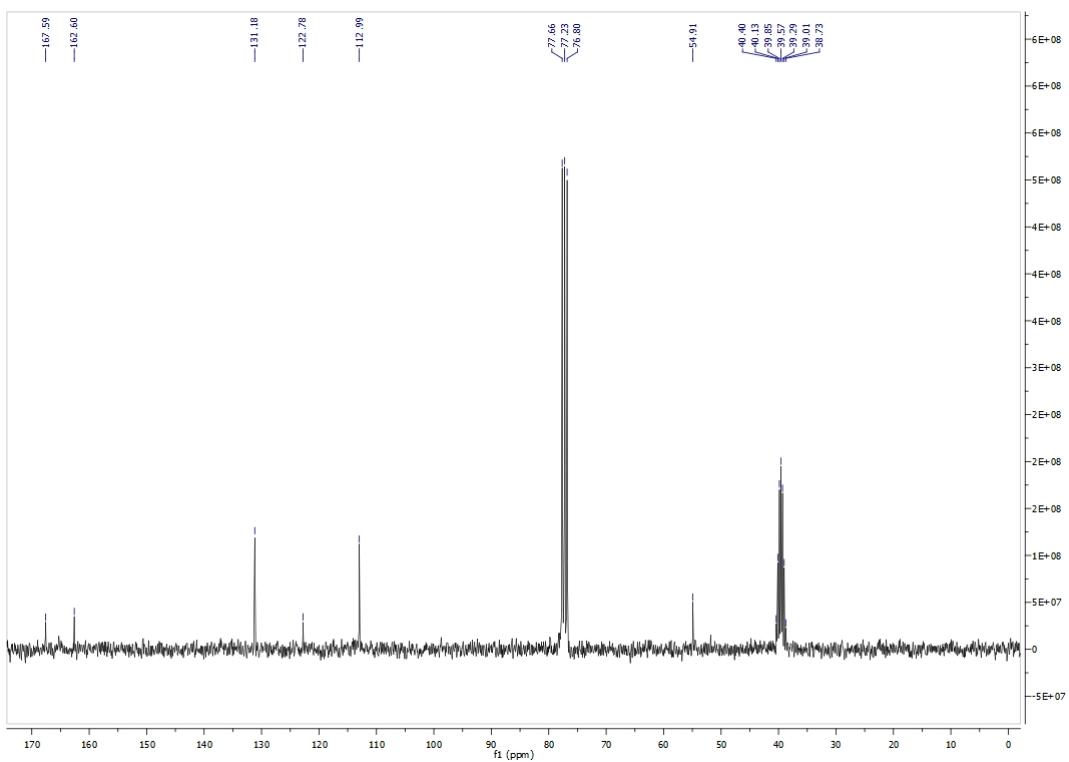


188 mg of **5g** was obtained from 190 mg (1 mmol) of **6n**. 98% yield; Colourless liquid, R_f = 0.5 (EtOAc/hexane = 1/9); ^1H NMR (300 MHz, CDCl_3) δ : 7.10 (d, J = 8.53 Hz, 2H), 6.82 (d, J = 8.53 Hz, 2H), 3.78 (s, 3H), 2.84 (t, J = 7.44 Hz, 2H), 2.70 (t, J = 7.77 Hz, 2H), 2.39 (q, J = 14.62, 7.35 Hz, 2H), 1.04 (t, J = 7.35 Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ : 211.0, 158.0, 133.3, 129.3, 114.0, 55.3, 44.3, 36.3, 29.1, 7.9.

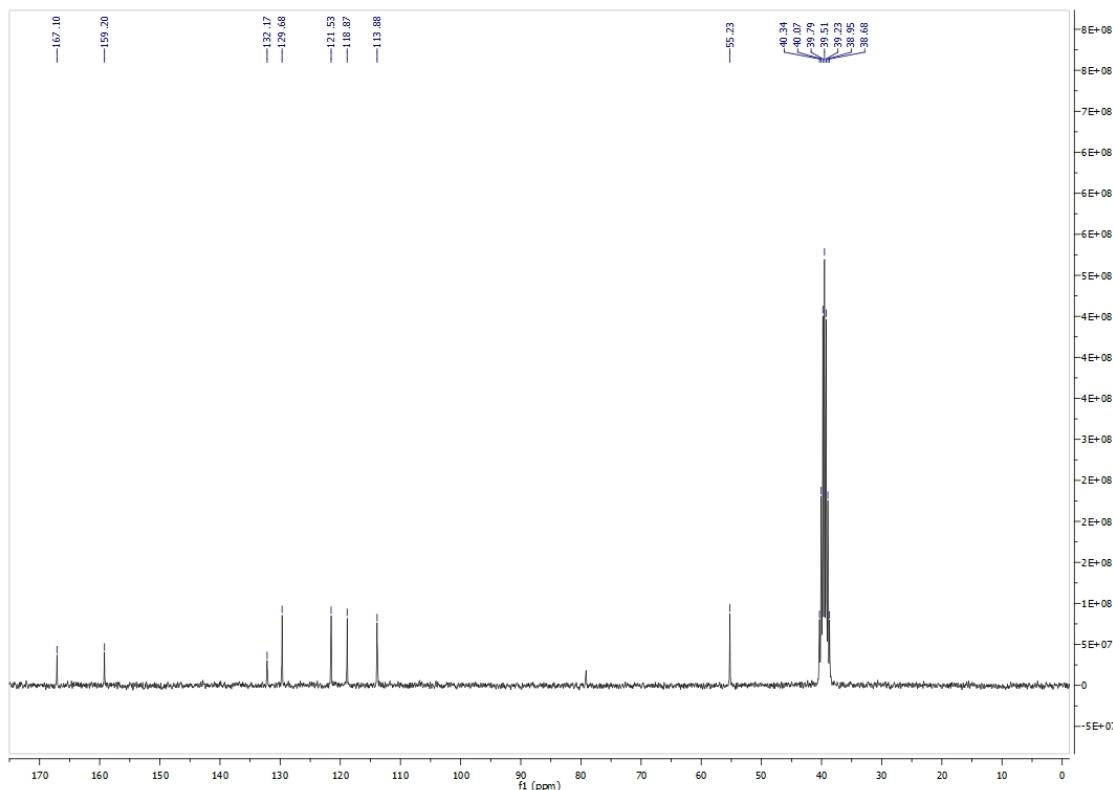
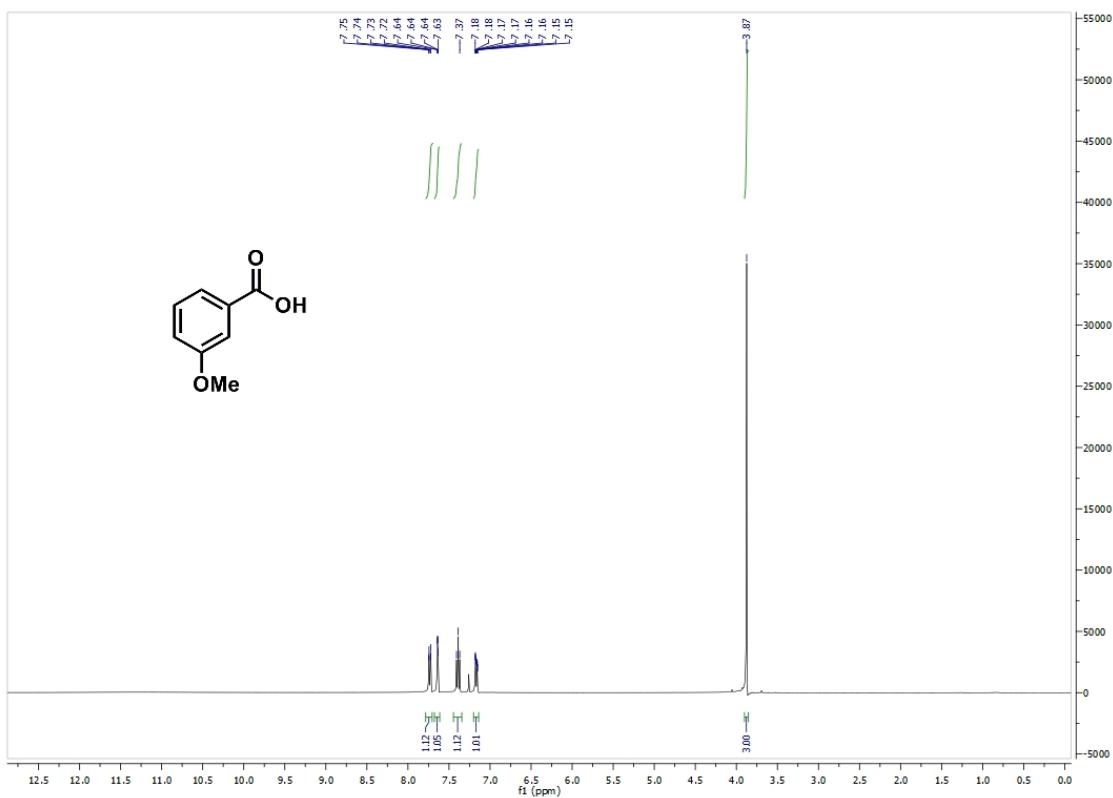
¹H and ¹³C NMR spectra of synthesised compounds

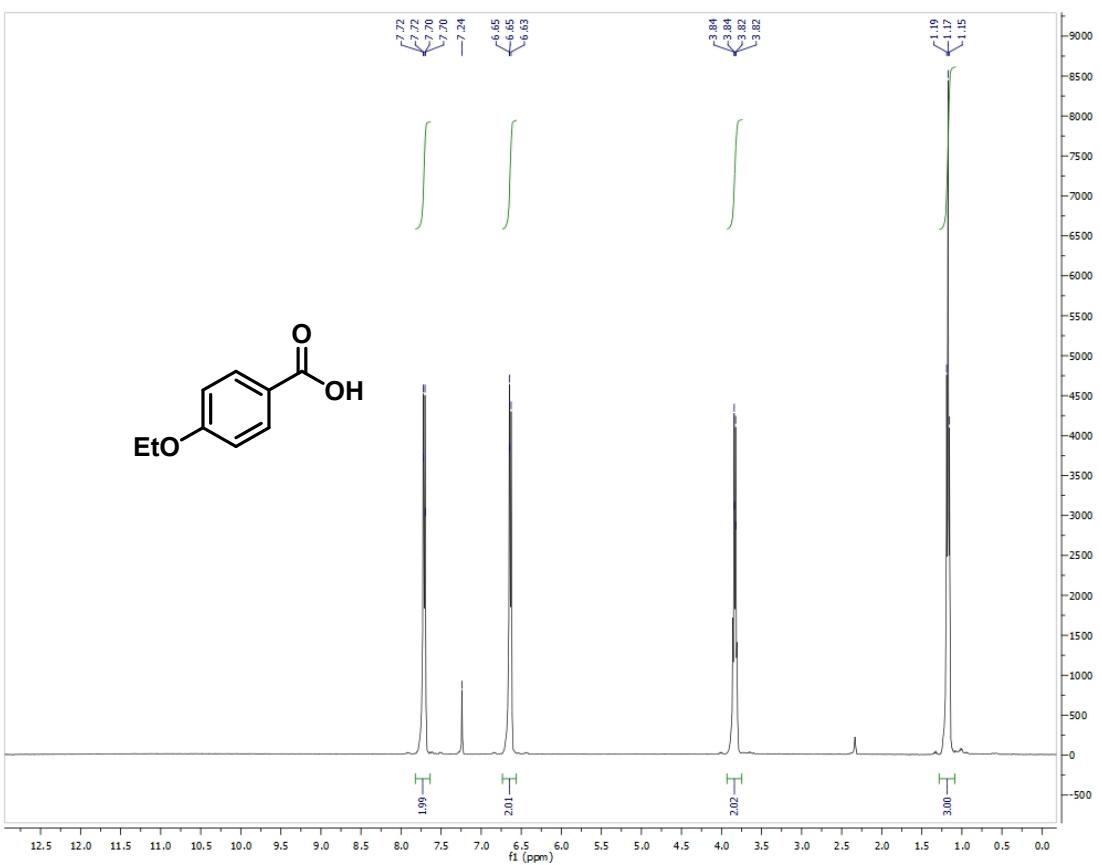
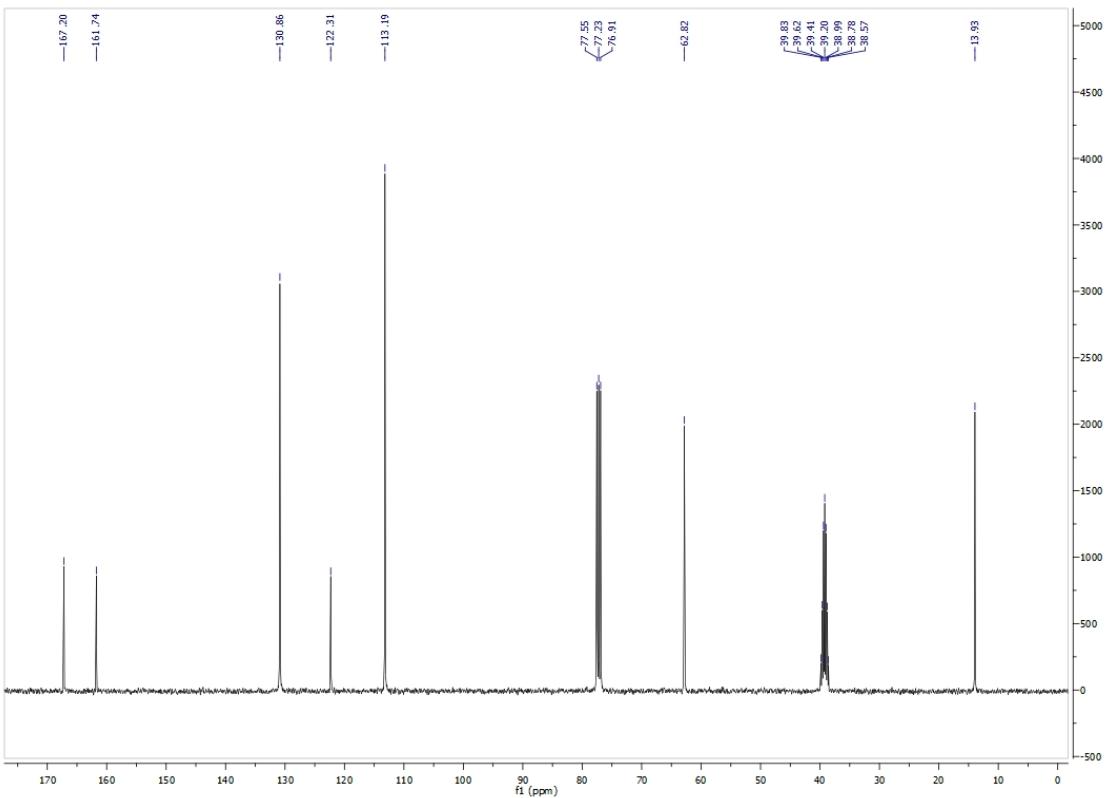


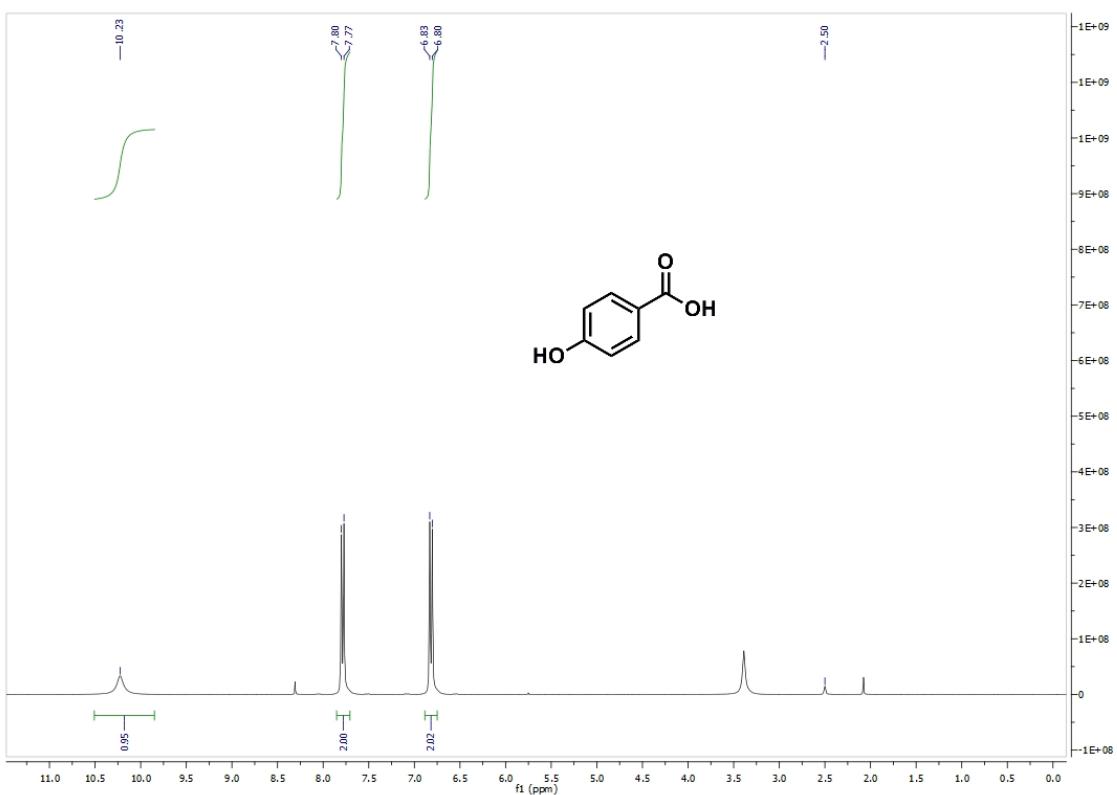
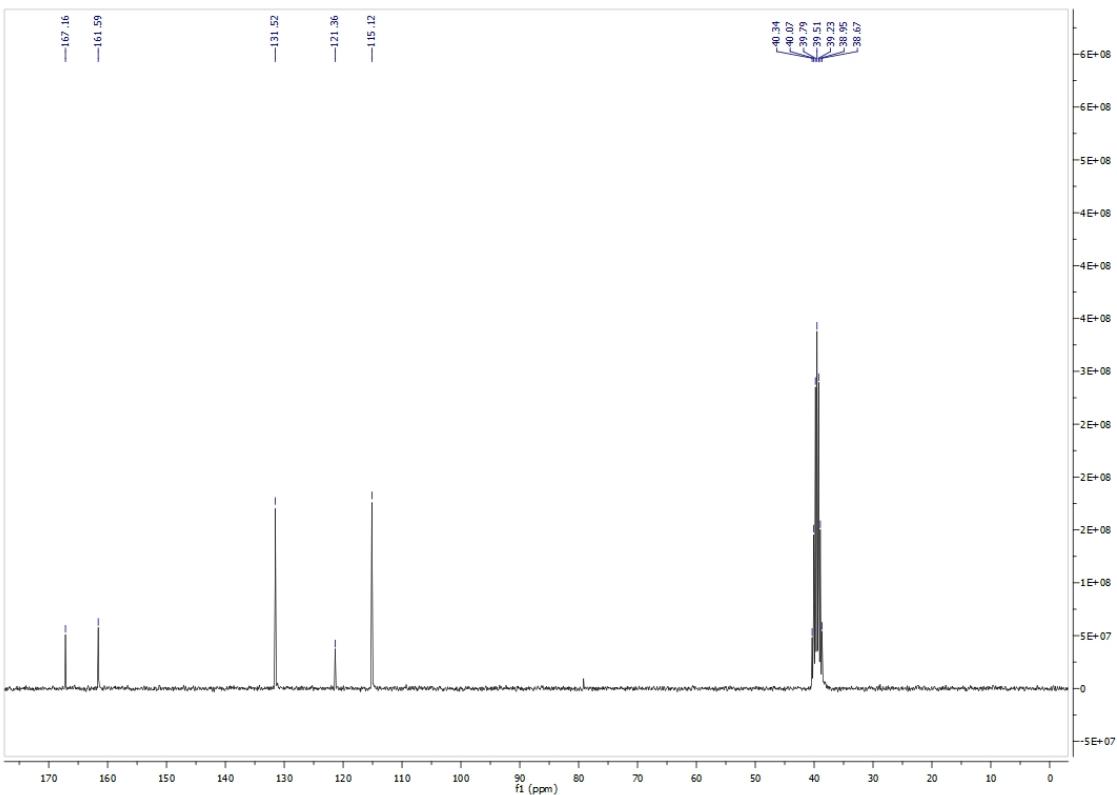
¹H-NMR spectrum of **3a** (300 MHz, CDCl₃)

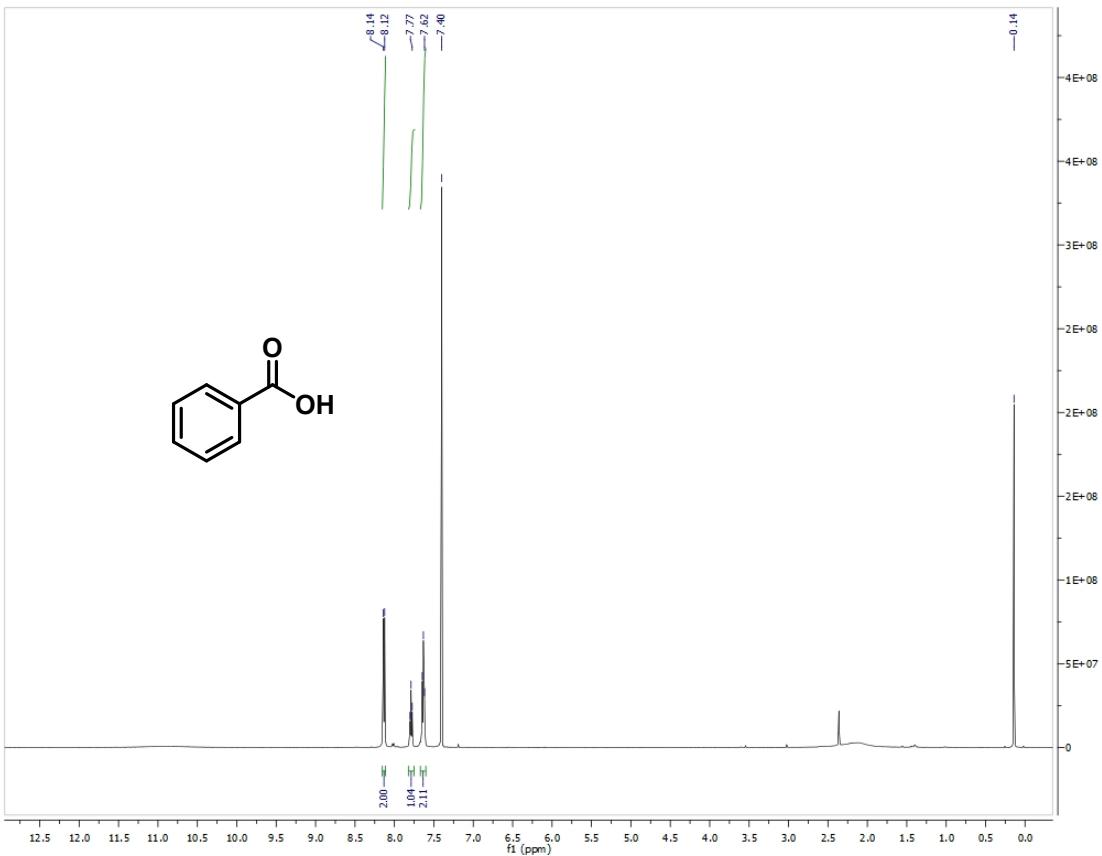
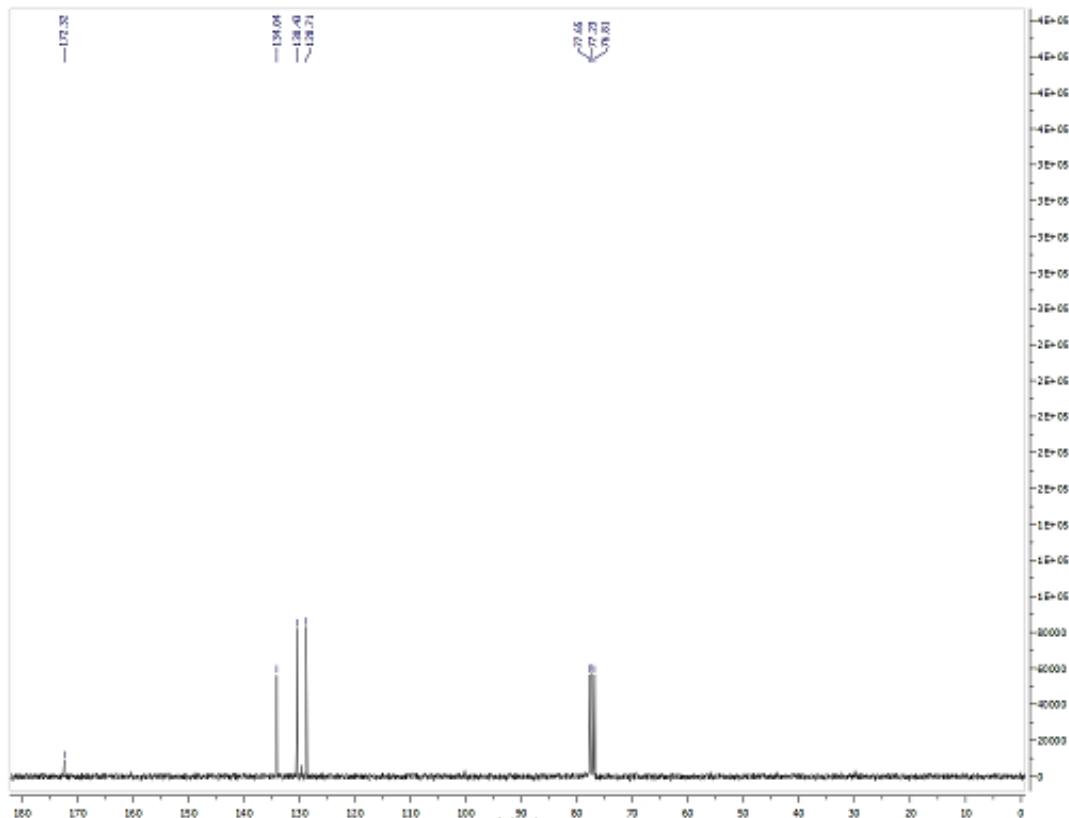


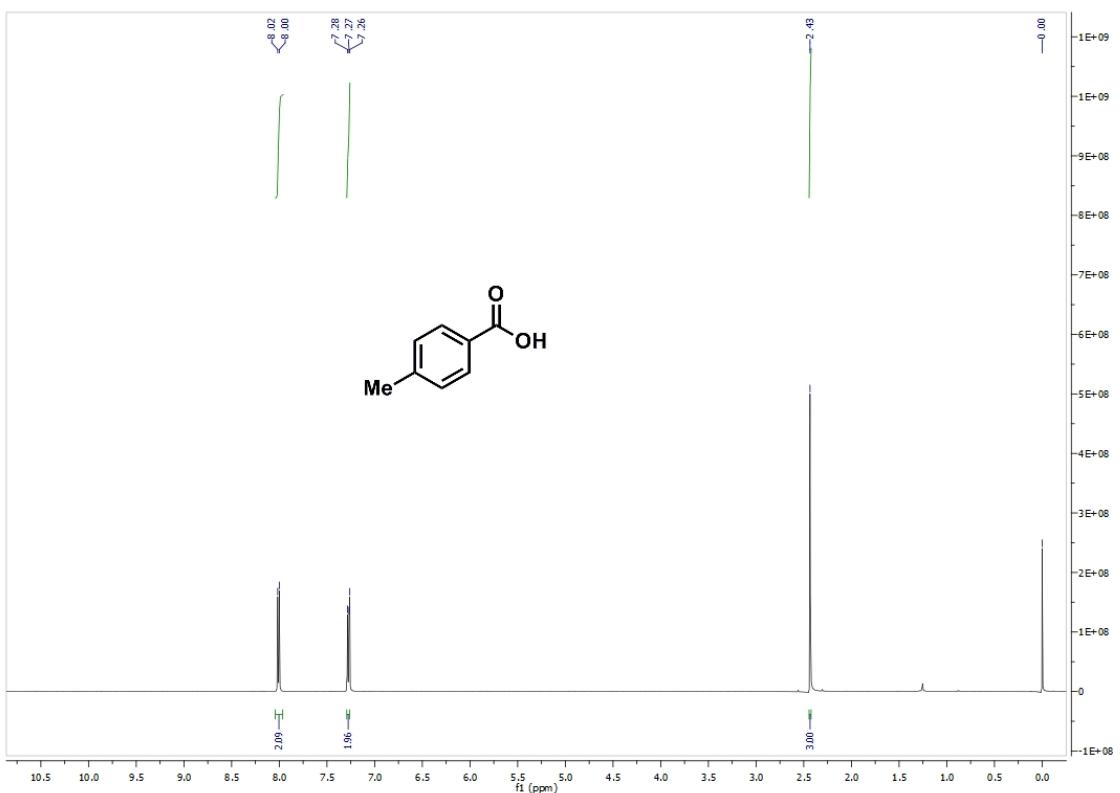
¹³C-NMR spectrum of **3a** (75 MHz, CDCl₃ + DMSO)



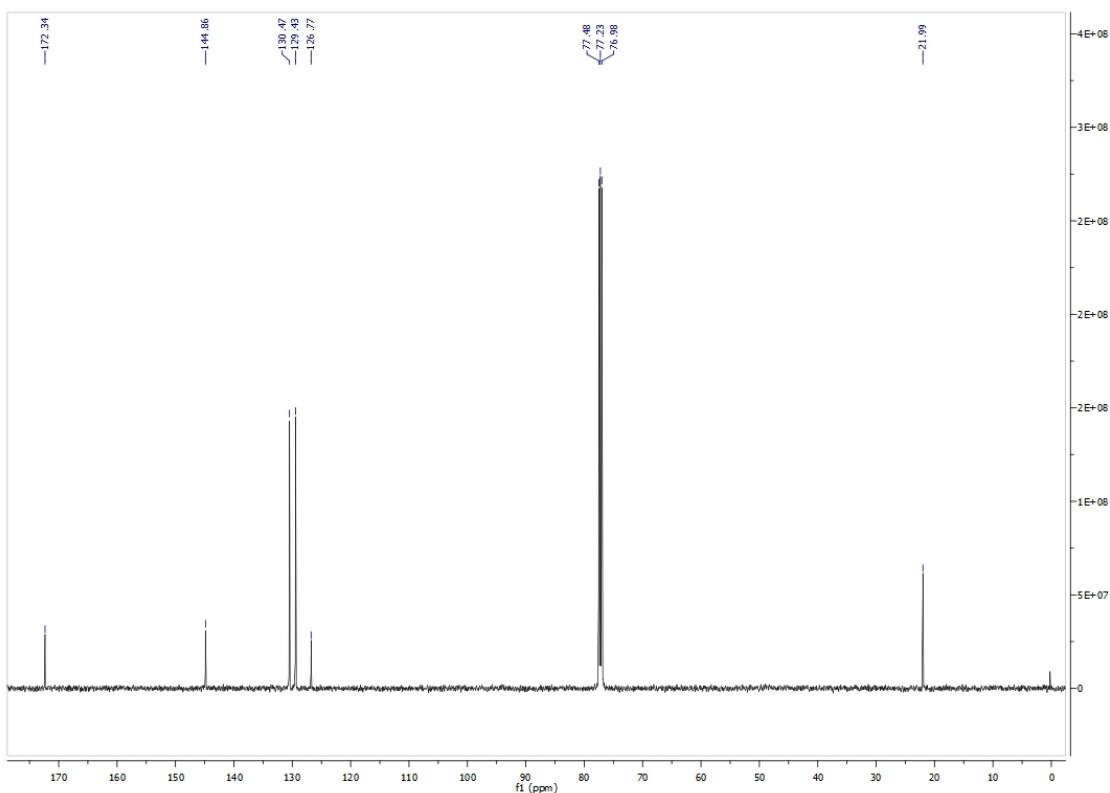
¹H-NMR spectrum of **3c** (300 MHz, CDCl₃ + DMSO)¹³C-NMR spectrum of **3c** (75 MHz, CDCl₃ + DMSO)

¹H-NMR spectrum of **3d** (300 MHz, DMSO)¹³C-NMR spectrum of **3d** (75 MHz, DMSO)

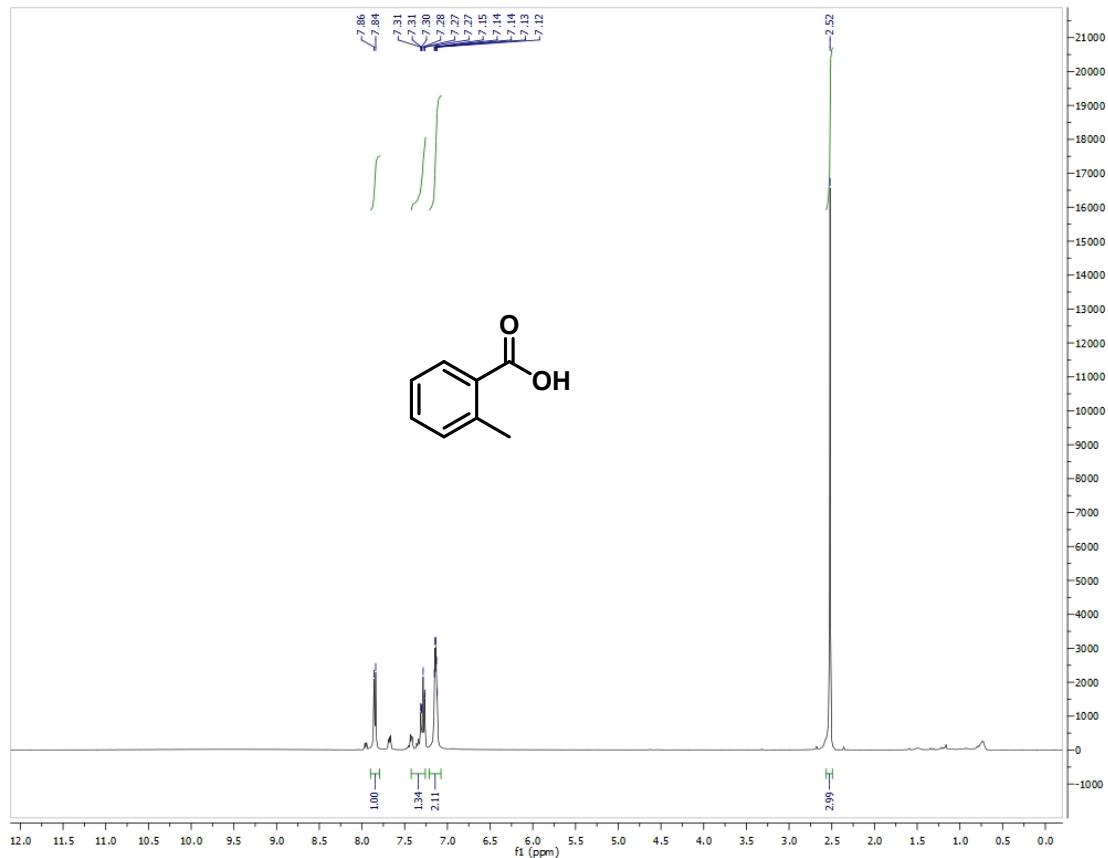
¹H-NMR spectrum of 3e (300 MHz, CDCl₃)¹³C-NMR spectrum of 3e (75 MHz, CDCl₃)



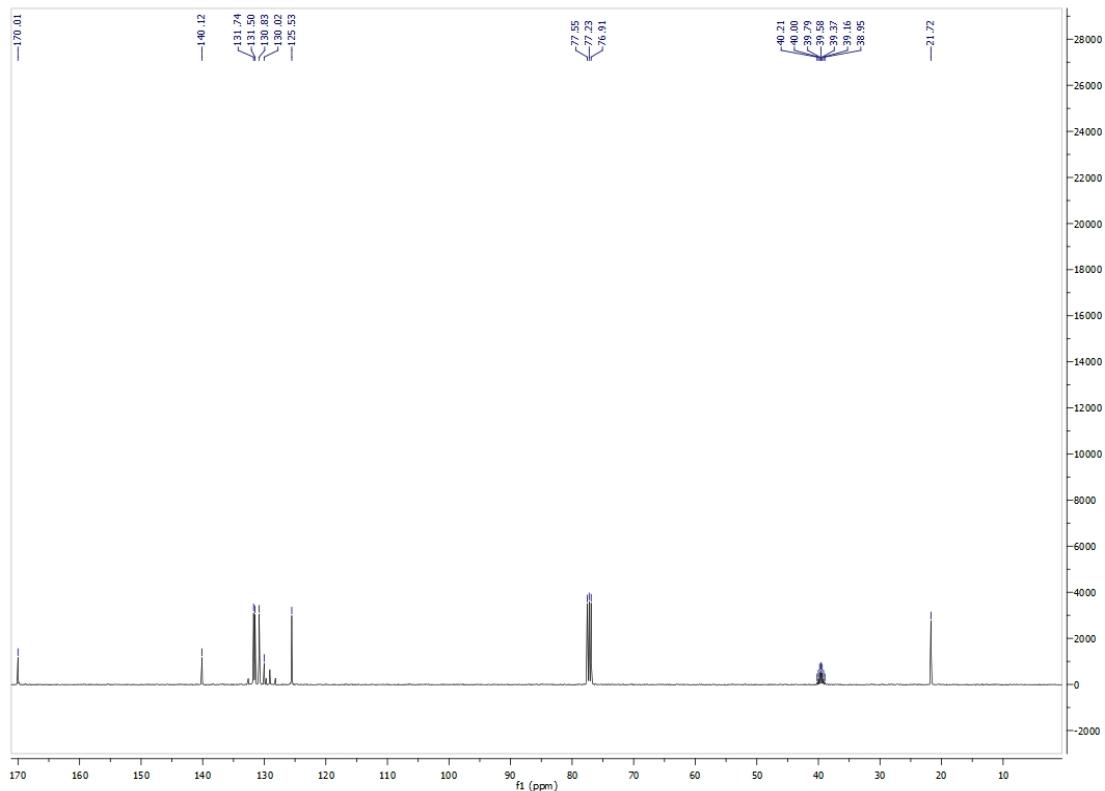
¹H-NMR spectrum of **3f** (300 MHz, CDCl₃)



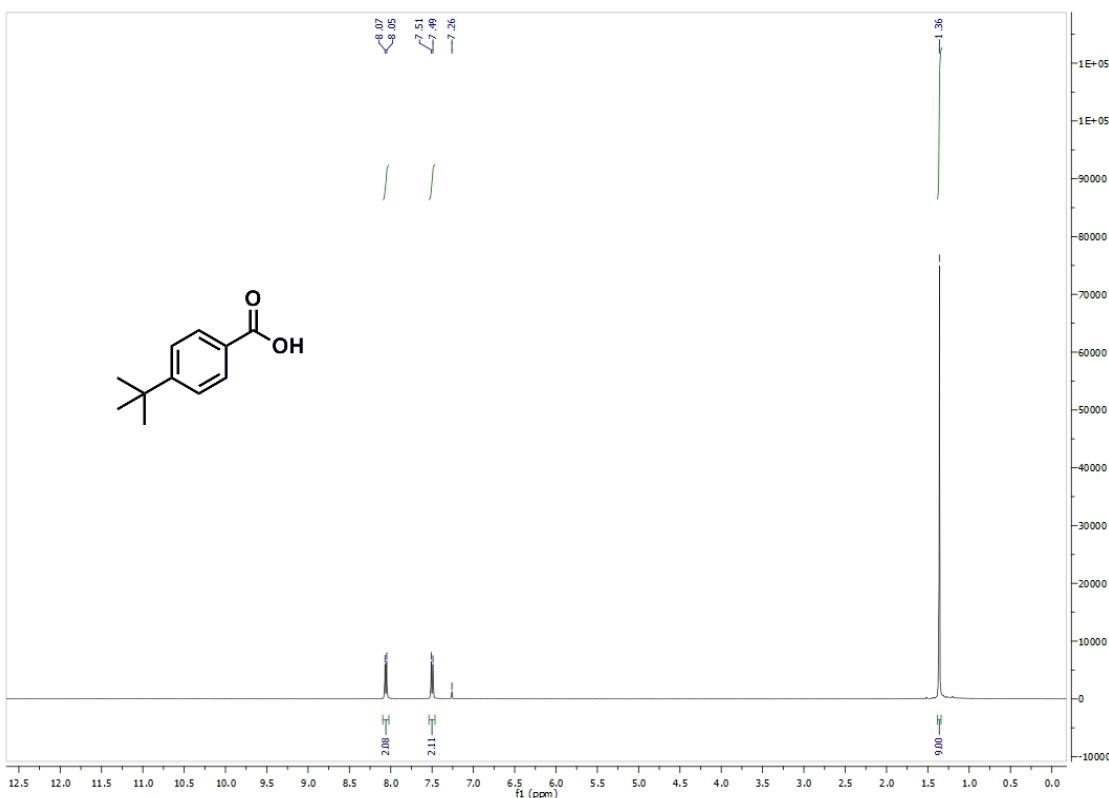
¹³C-NMR spectrum of **3f** (75 MHz, CDCl₃)



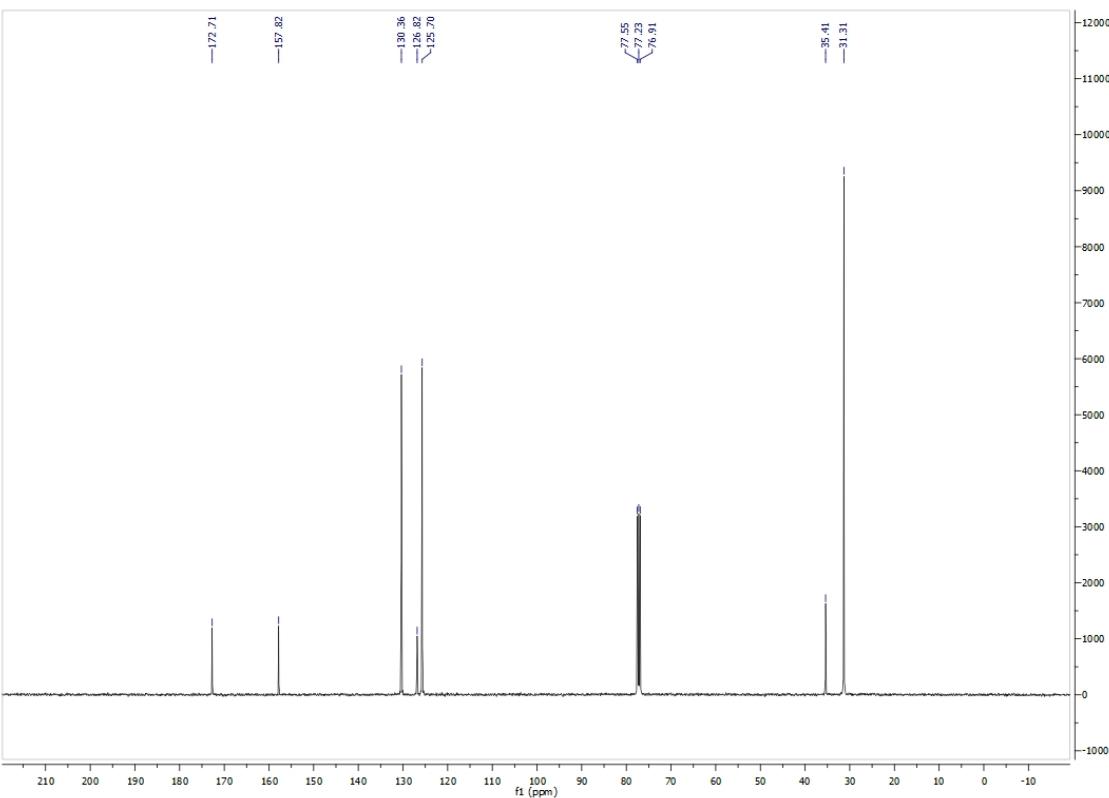
^1H -NMR spectrum of **3g** (300 MHz, CDCl_3)



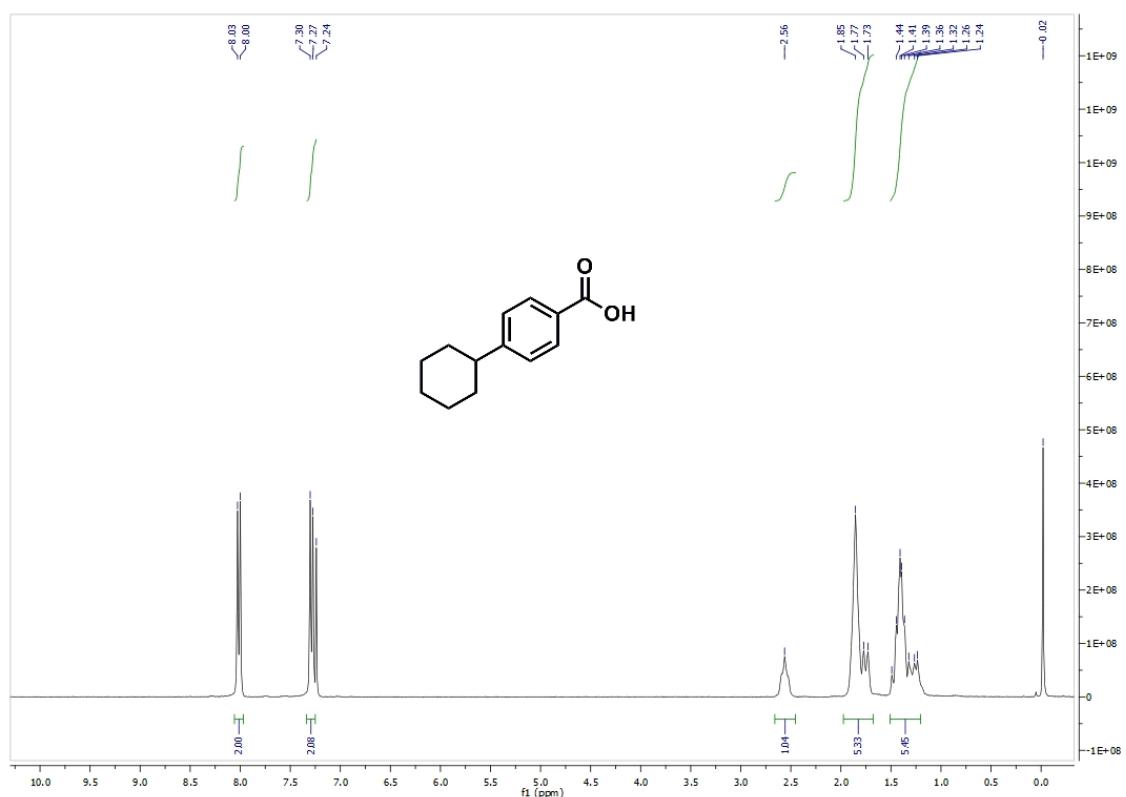
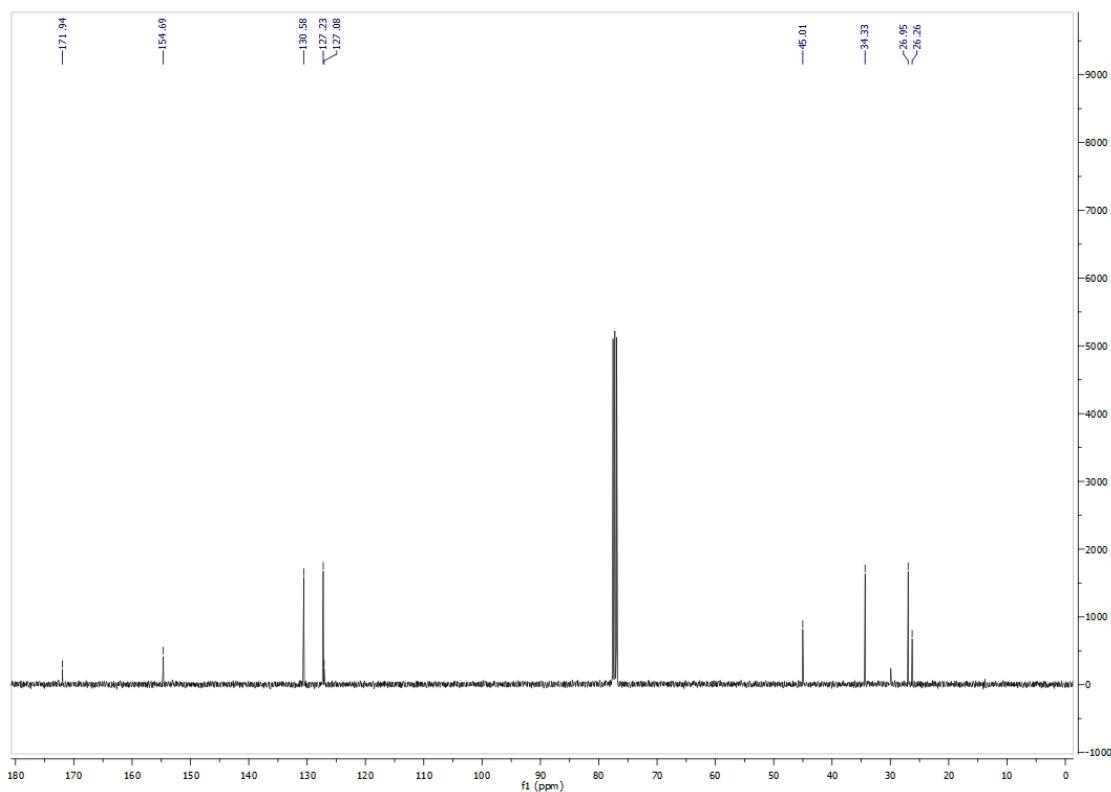
^{13}C -NMR spectrum of **3g** (75 MHz, $\text{CDCl}_3 + \text{DMSO}$)

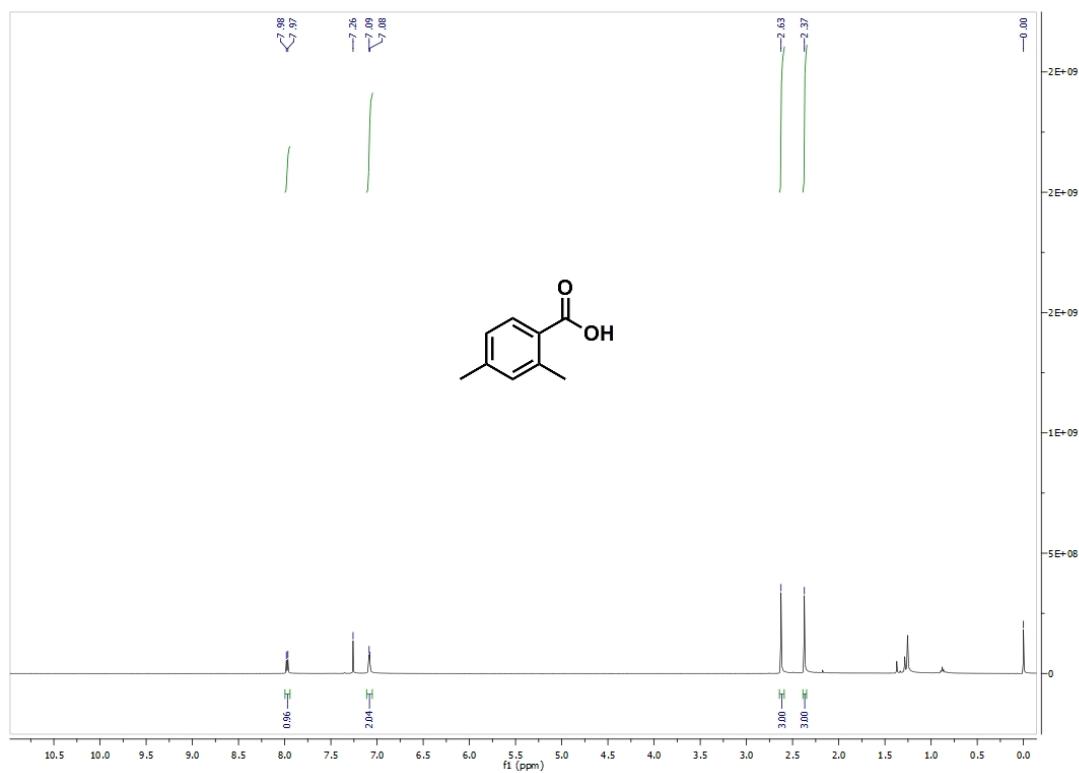


¹H-NMR spectrum of **3h** (300 MHz, CDCl₃)

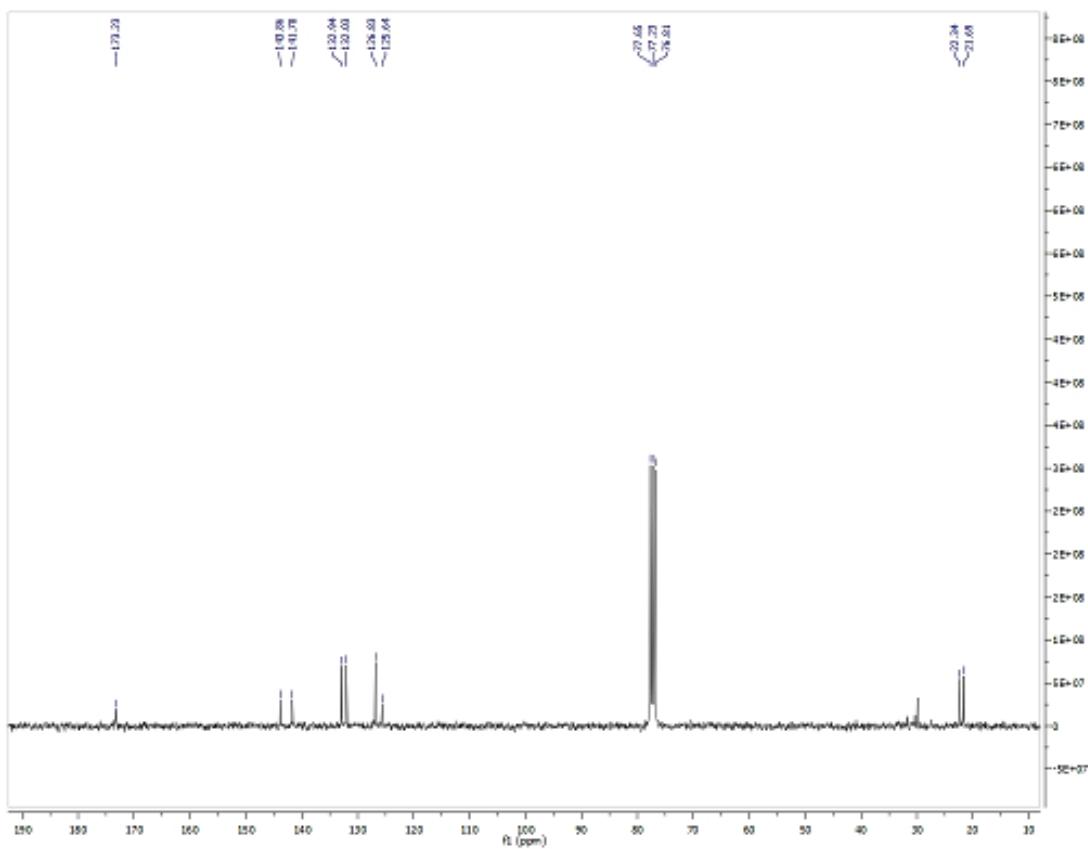


¹³C-NMR spectrum of **3h** (75 MHz, CDCl₃)

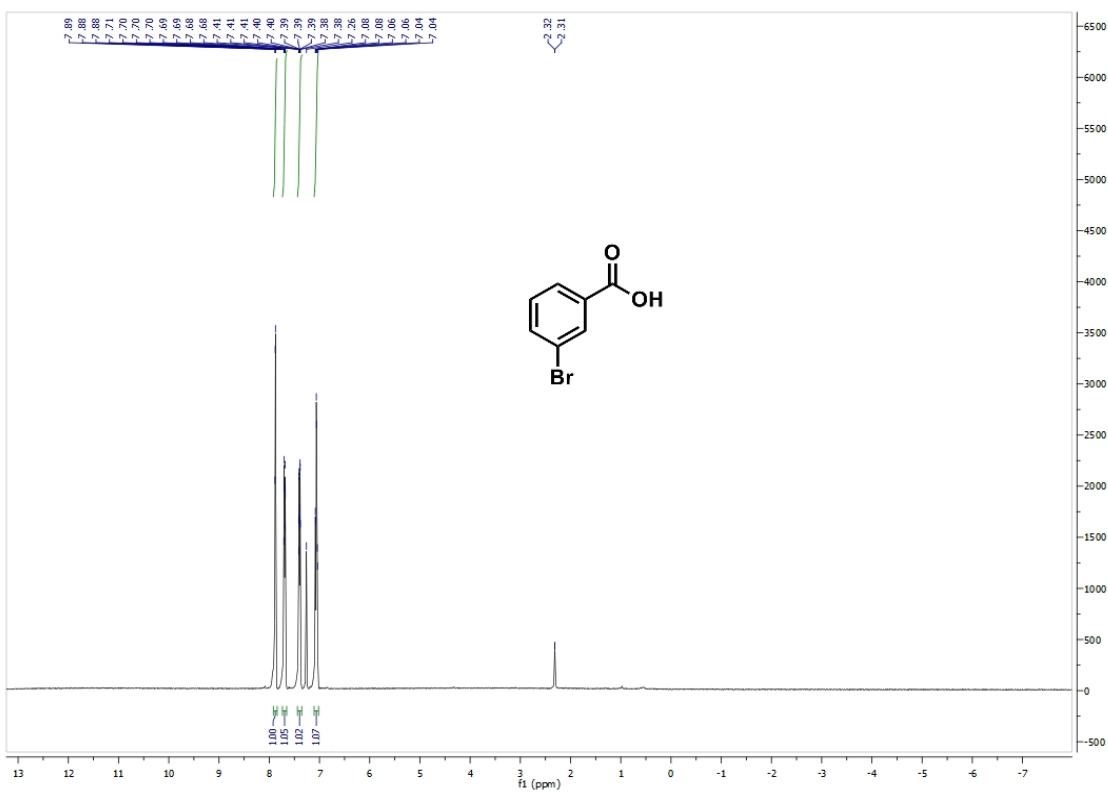
¹H-NMR spectrum of **3i** (300 MHz, CDCl₃)¹³C-NMR spectrum of **3i** (75 MHz, CDCl₃)



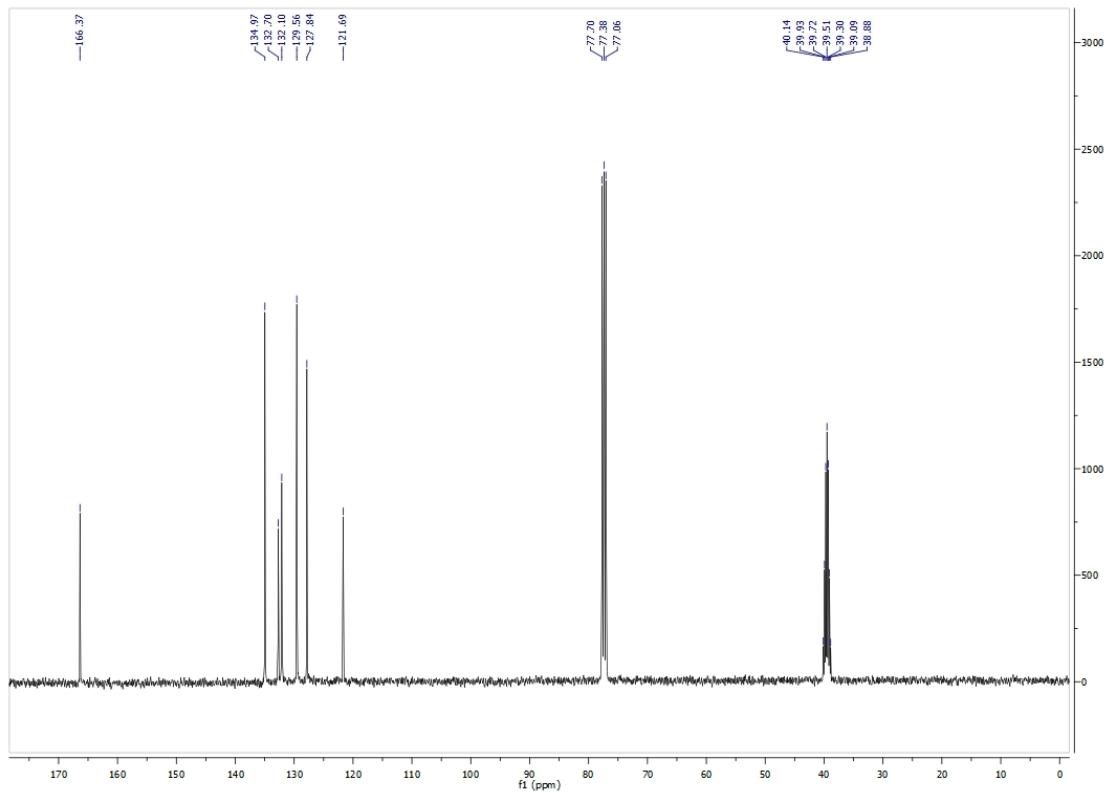
¹H-NMR spectrum of **3j** (300 MHz, CDCl₃)



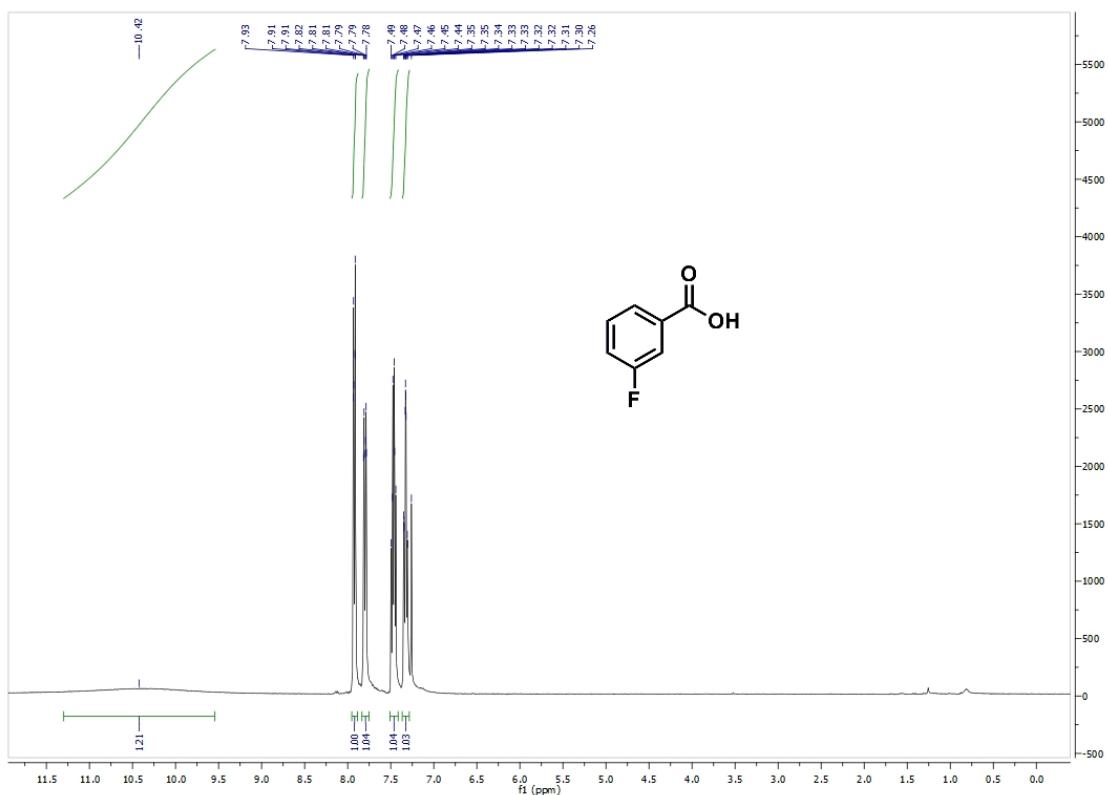
¹³C-NMR spectrum of **3j** (75 MHz, CDCl₃)



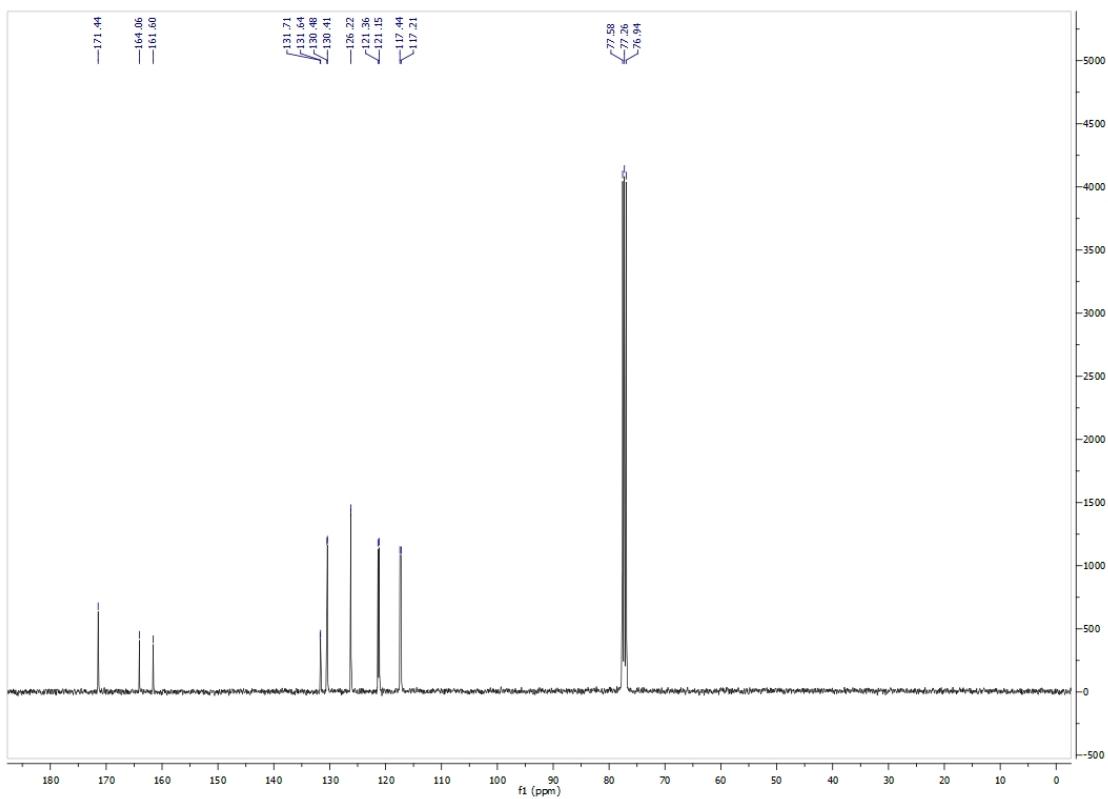
¹H-NMR spectrum of **3k** (300 MHz, DMSO)



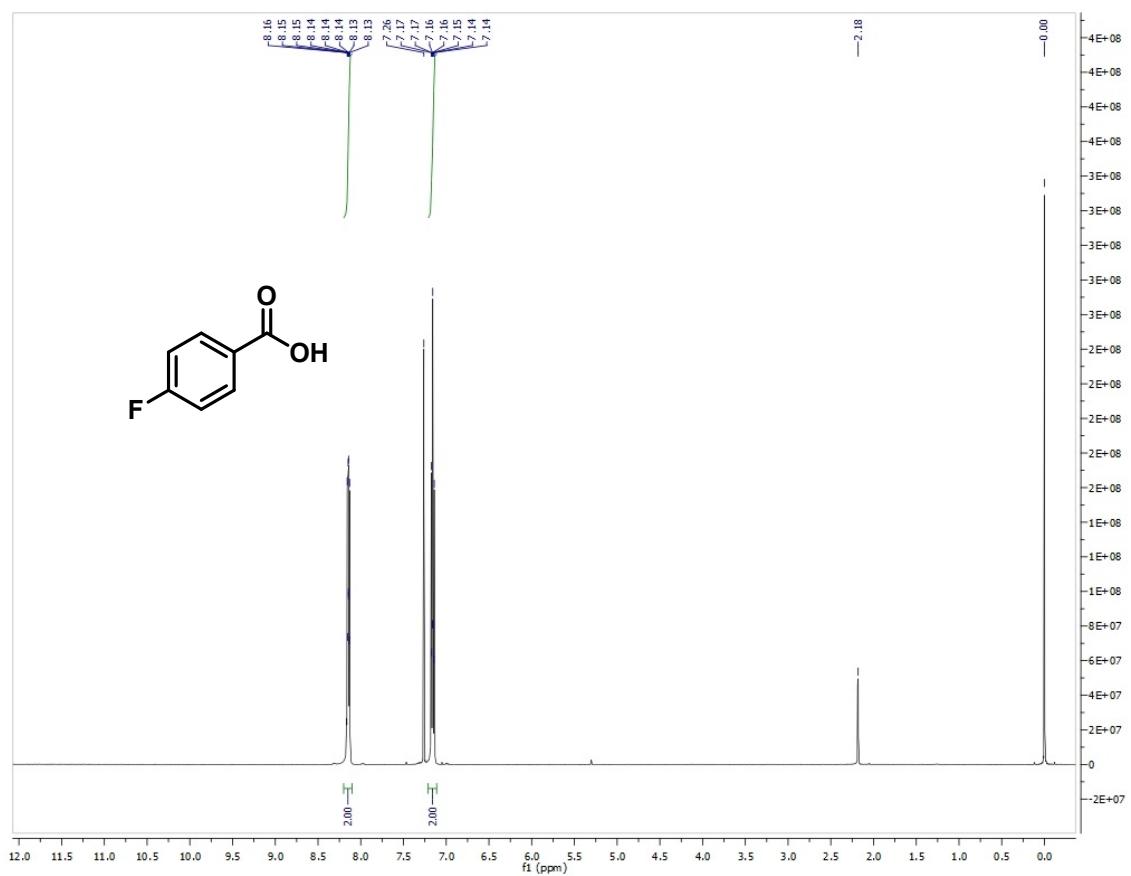
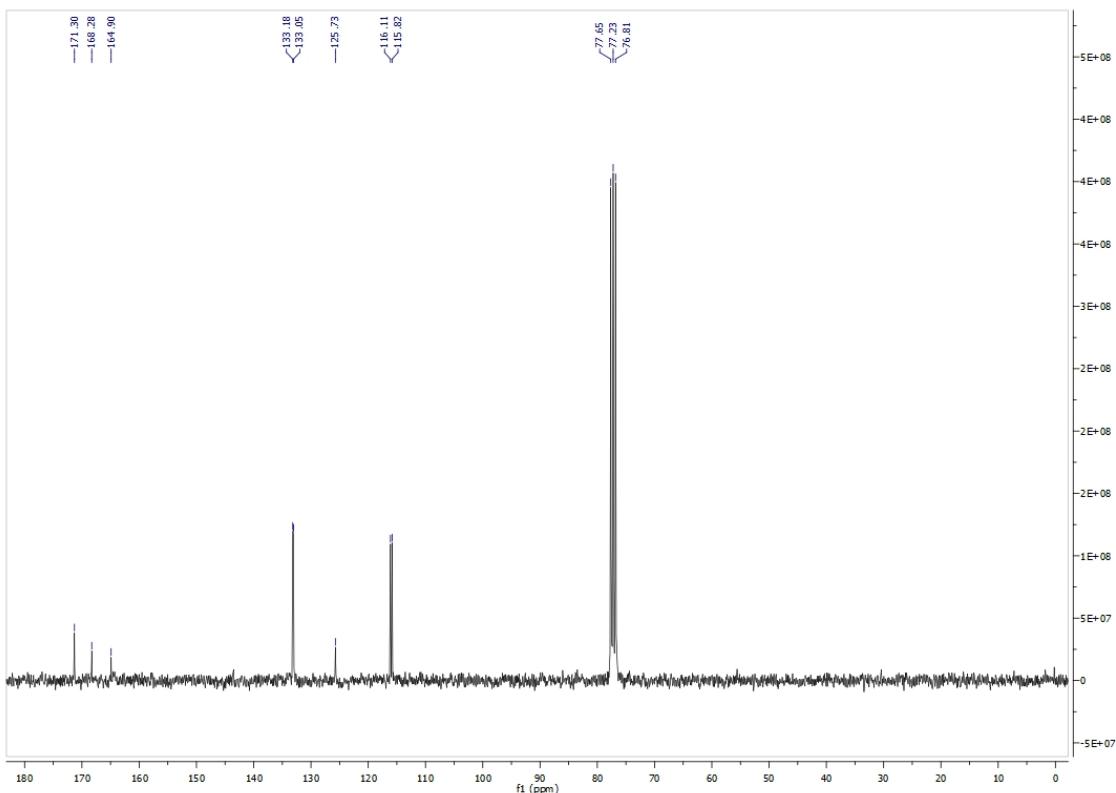
¹³C -NMR spectrum of **3k** (300 MHz, CDCl₃+ DMSO)

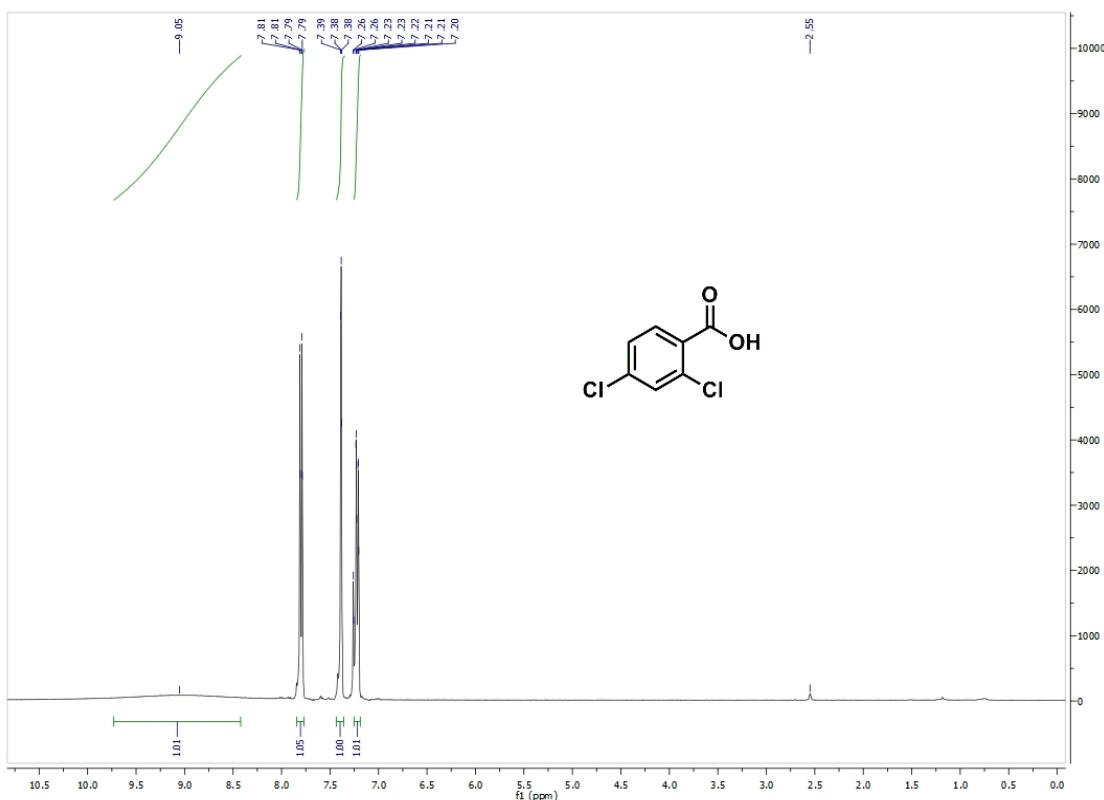


¹H-NMR spectrum of **3I** (300 MHz, CDCl₃)

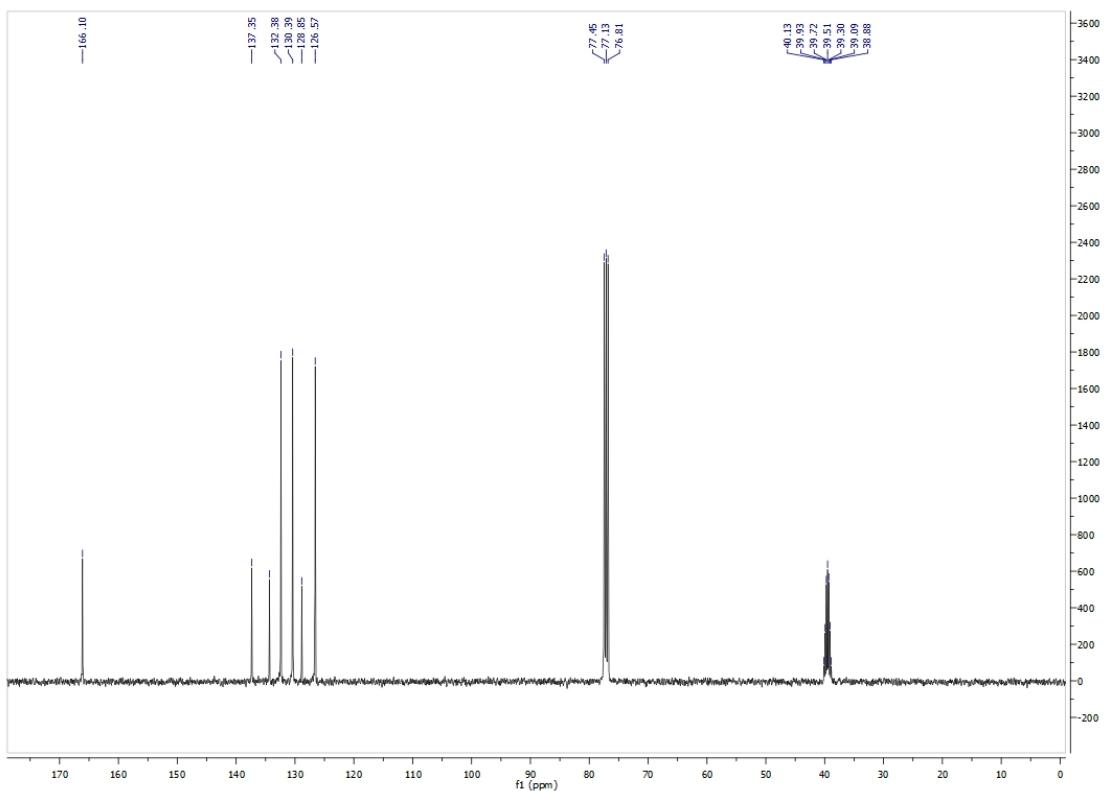


¹³C -NMR spectrum of **3I** (300 MHz, CDCl₃)

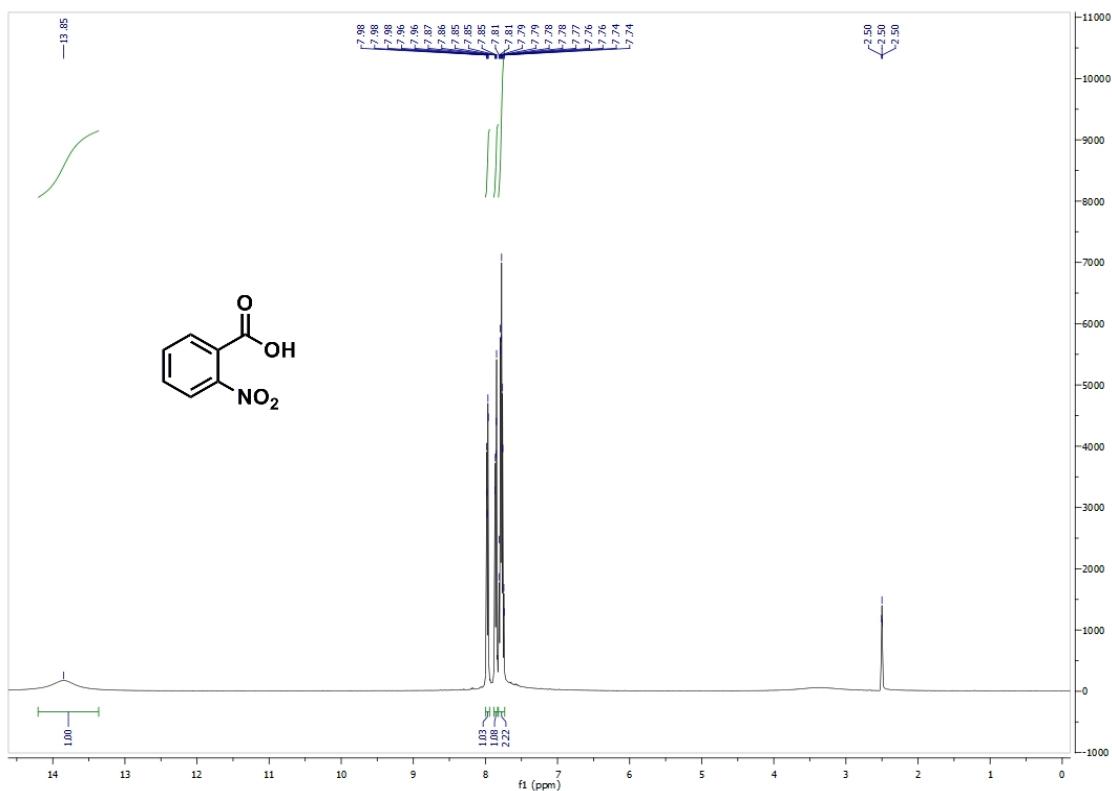
¹H-NMR spectrum of **3m** (300 MHz, CDCl₃)¹³C-NMR spectrum of **3m** (300 MHz, CDCl₃)



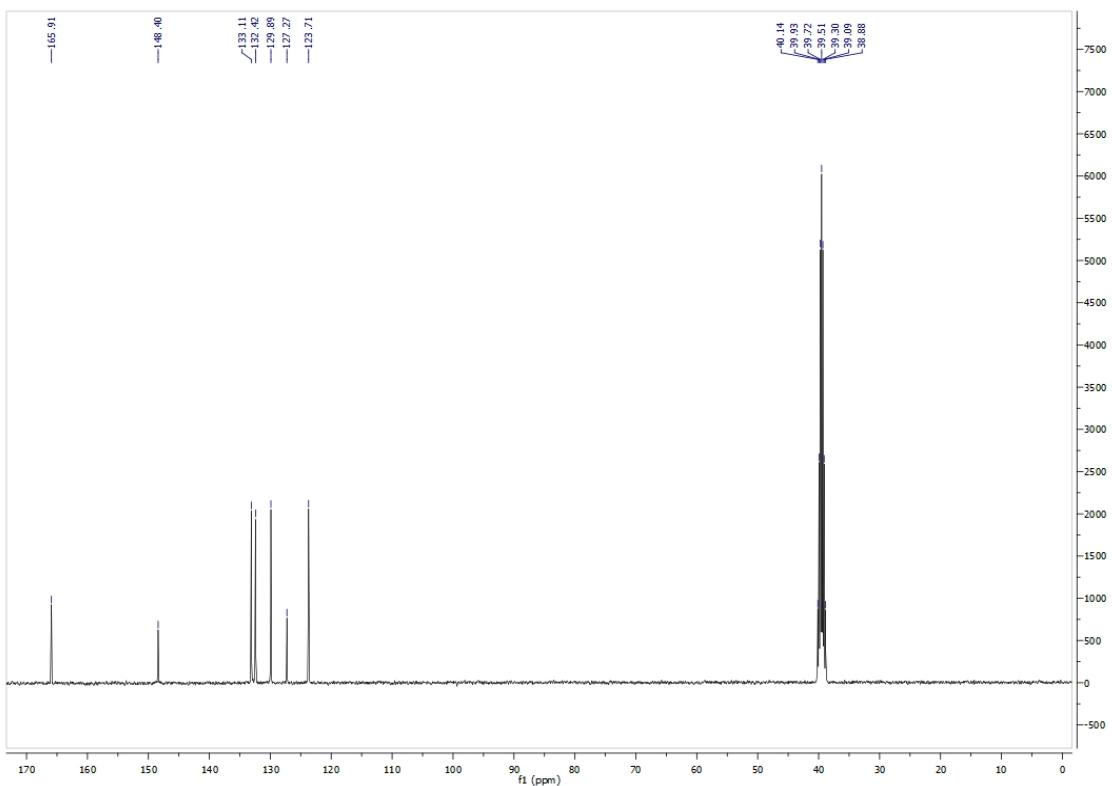
¹H-NMR spectrum of **3n** (300 MHz, DMSO)



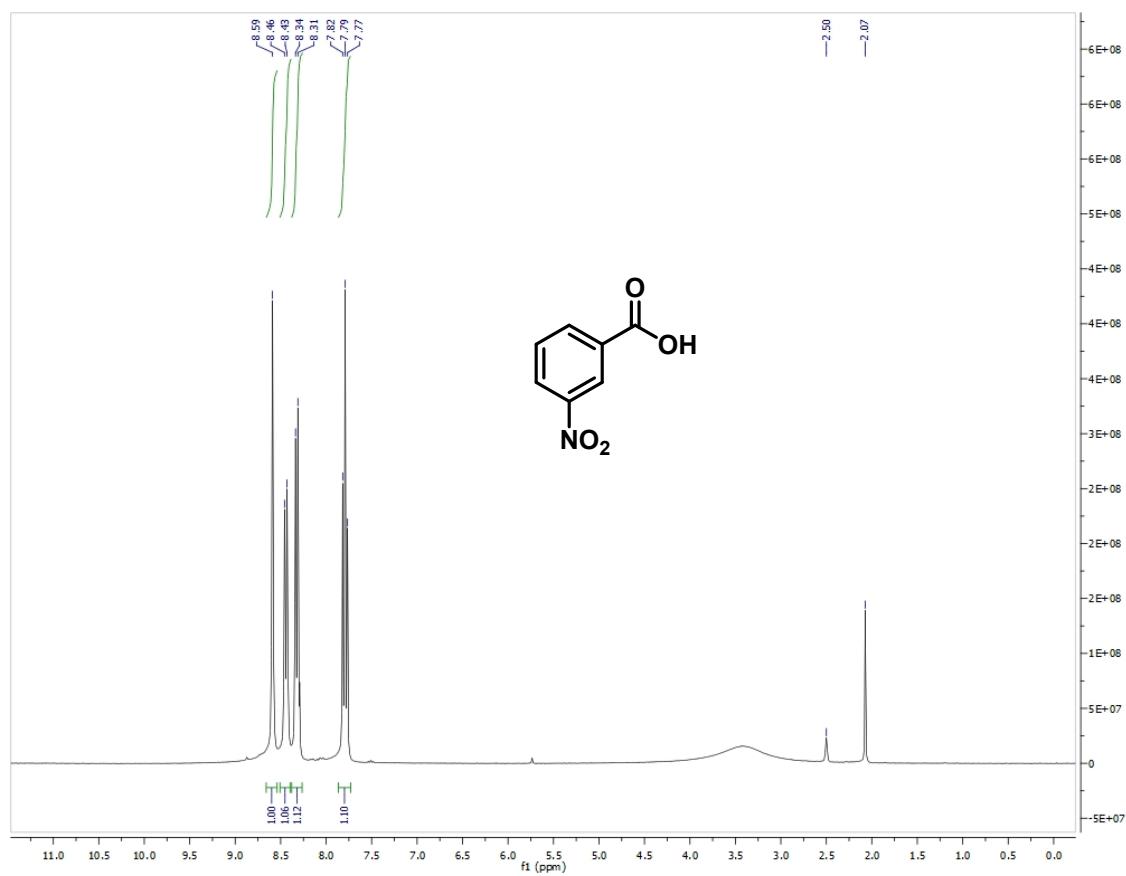
¹³C -NMR spectrum of **3n** (300 MHz, CDCl₃+ DMSO)



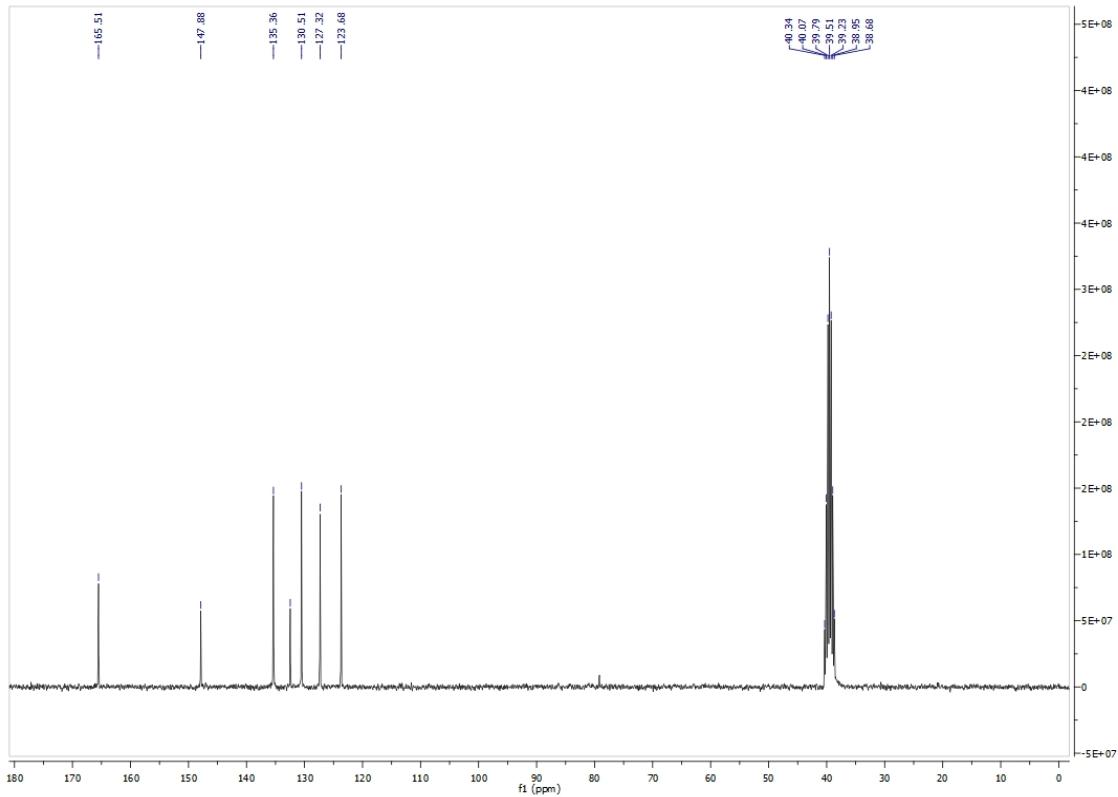
¹H-NMR spectrum of **3o** (300 MHz, DMSO)

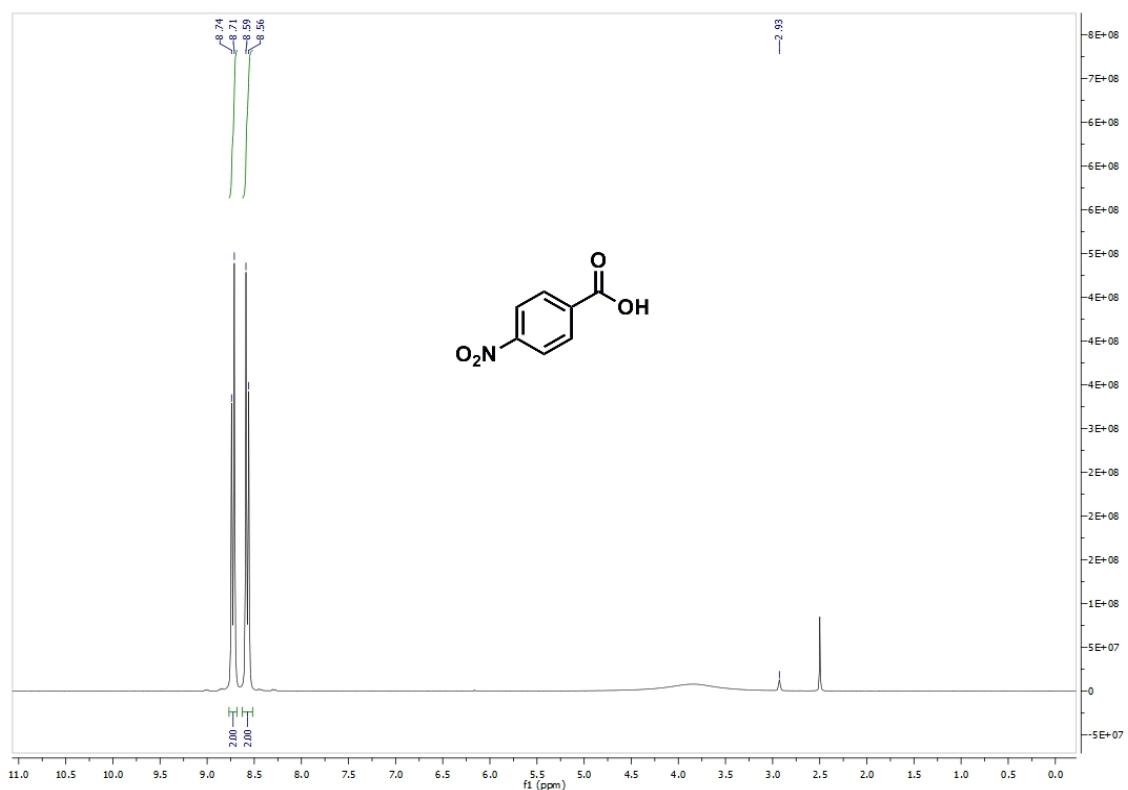


¹³C -NMR spectrum of **3o** (300 MHz, DMSO)

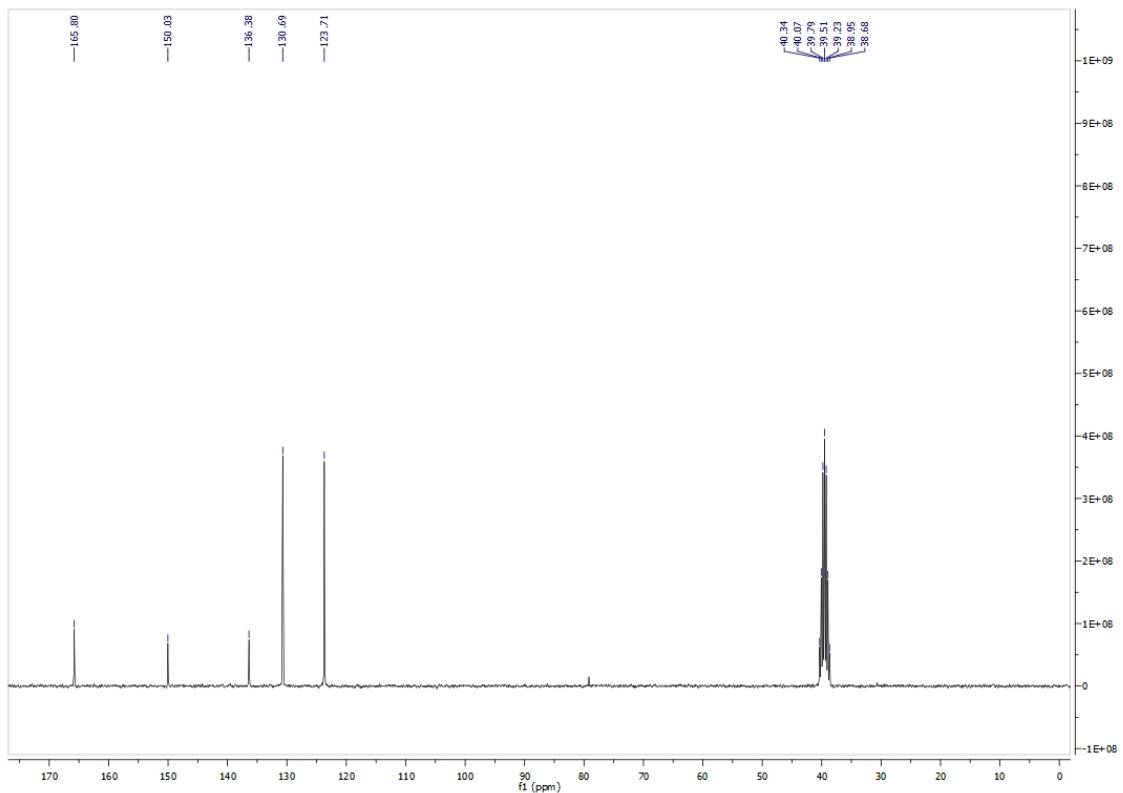


¹H-NMR spectrum of **3p** (300 MHz, DMSO)

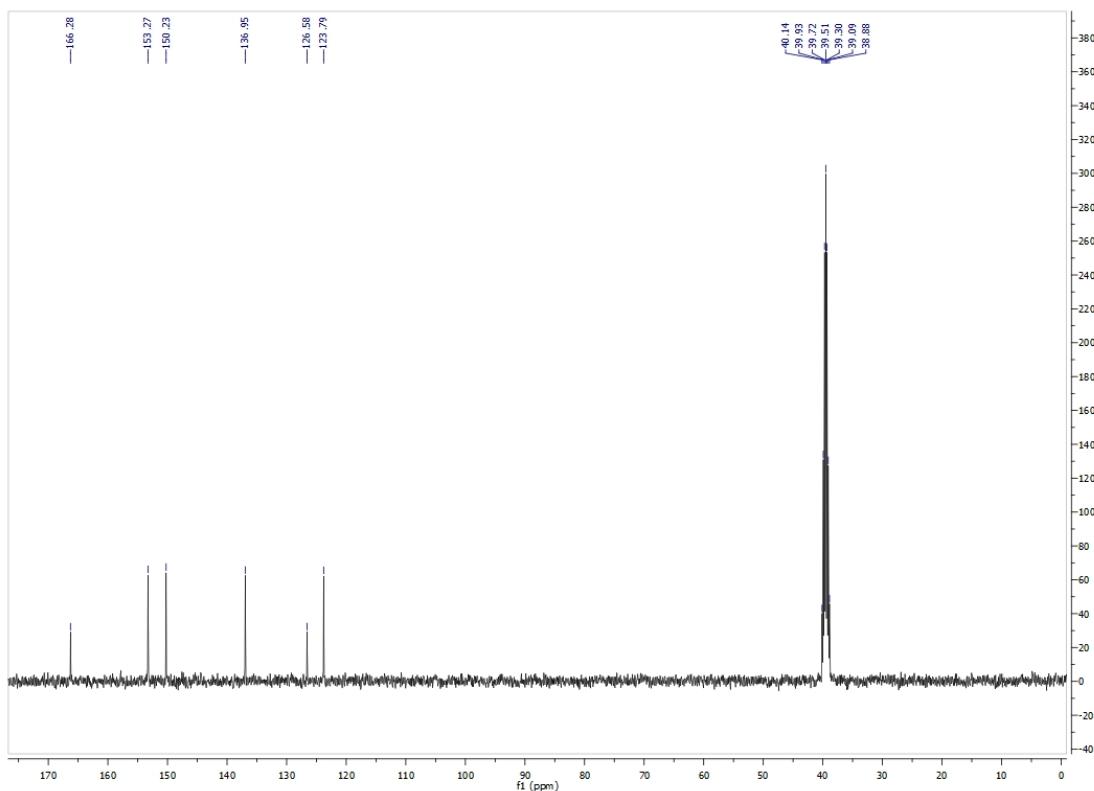
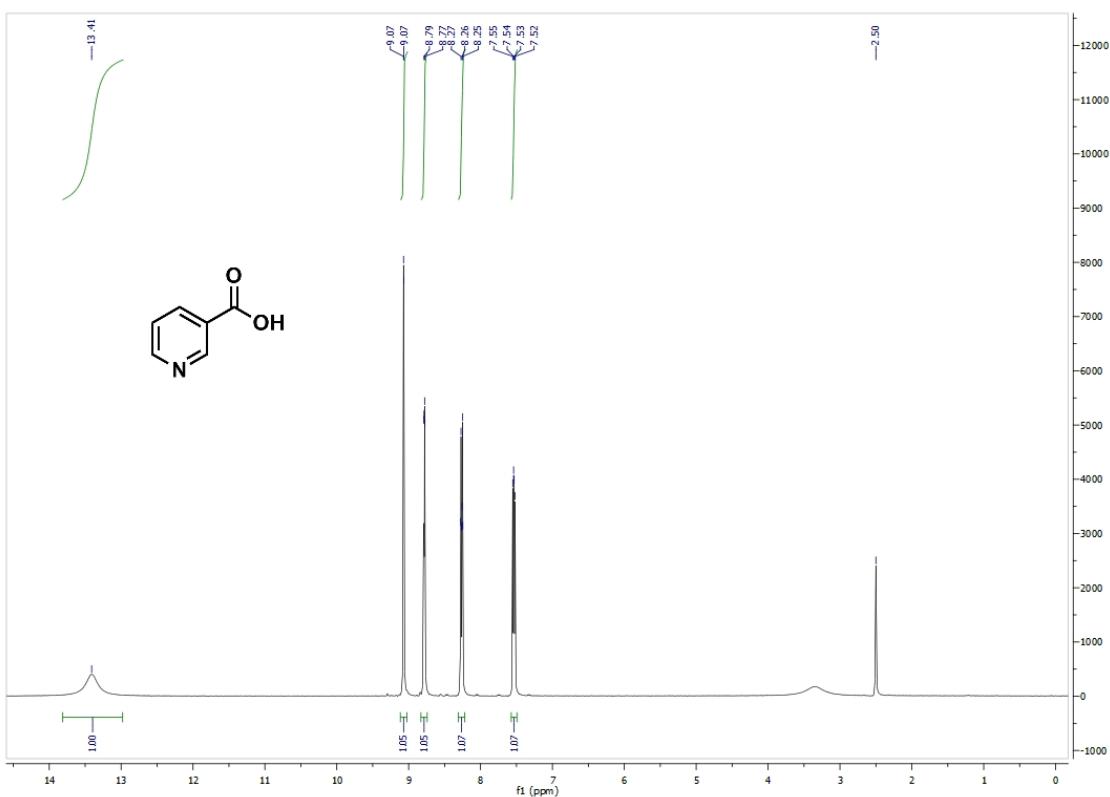


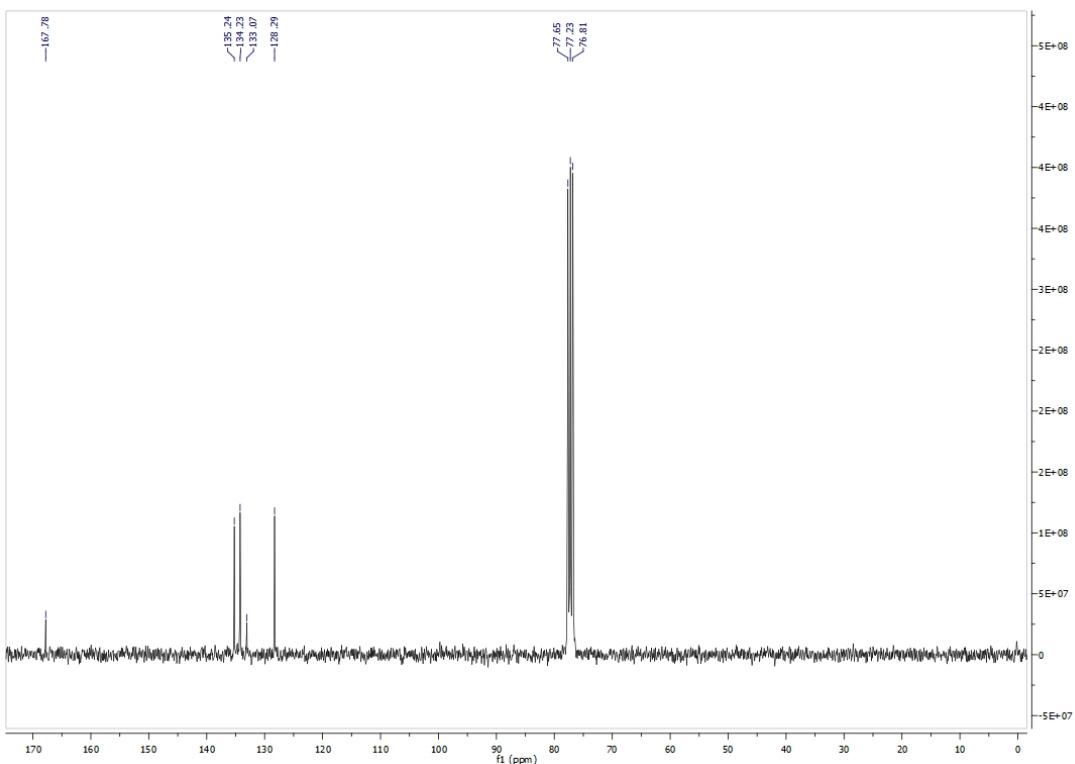
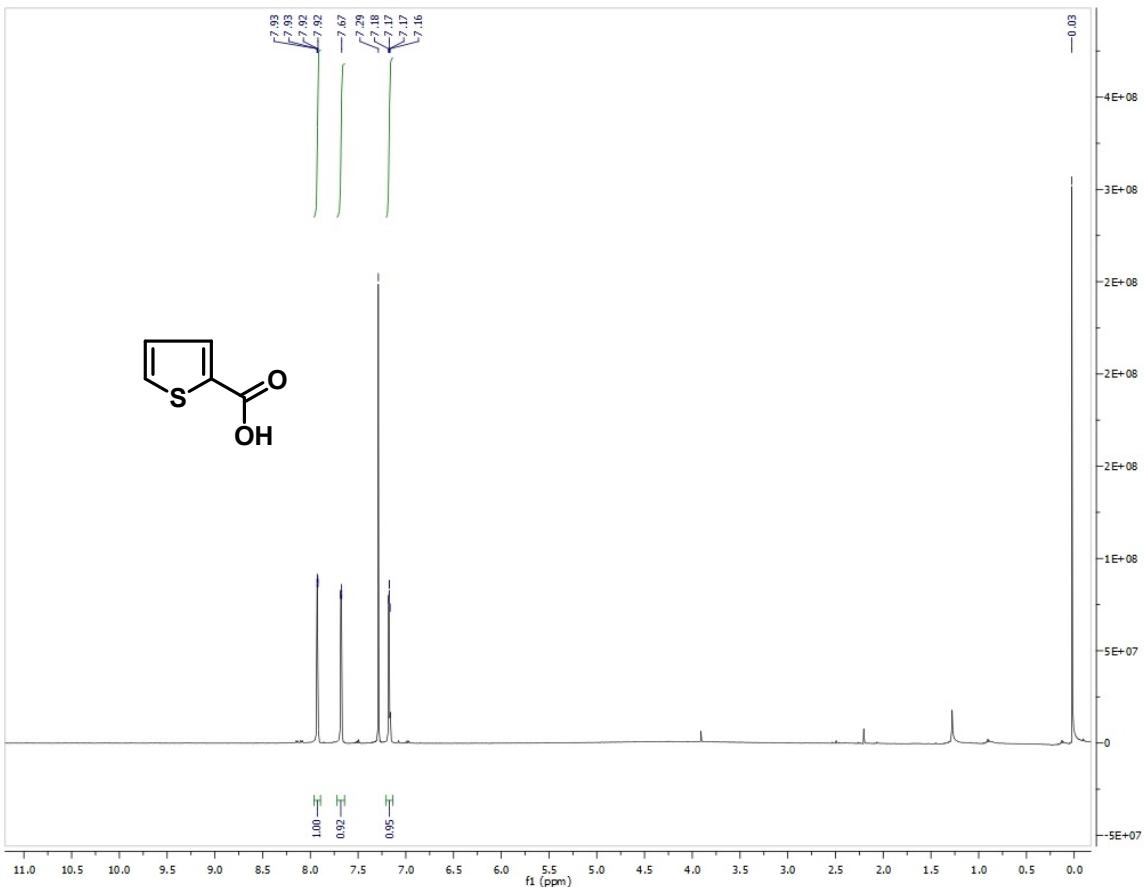


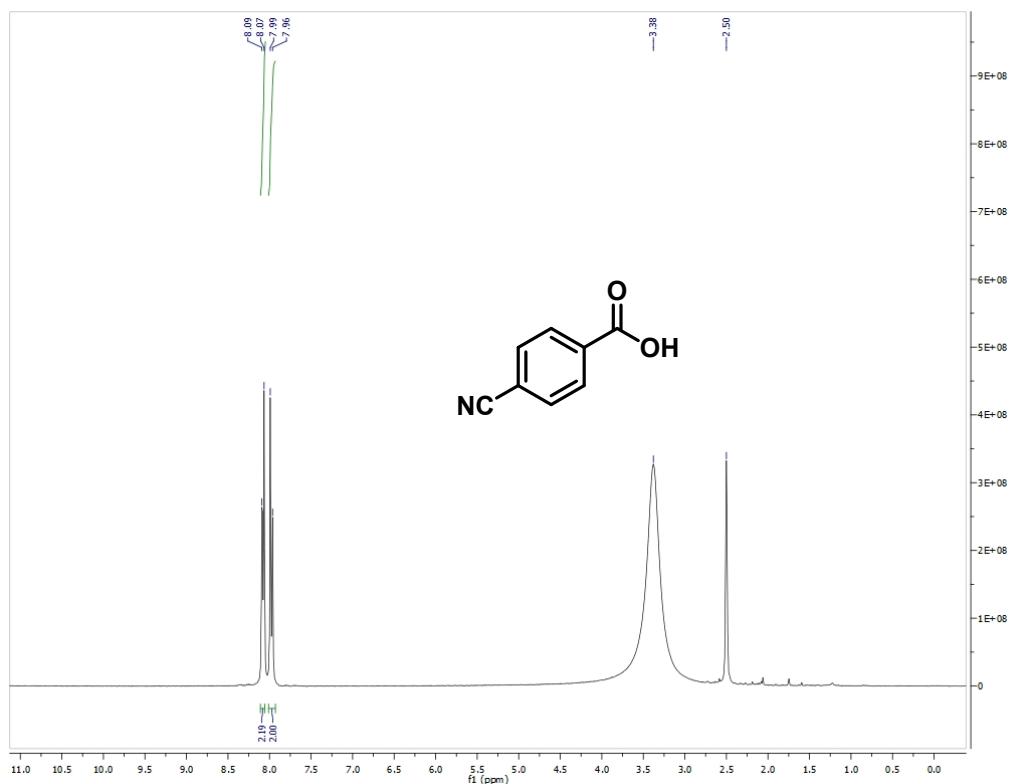
¹H-NMR spectrum of **3q** (300 MHz, DMSO)



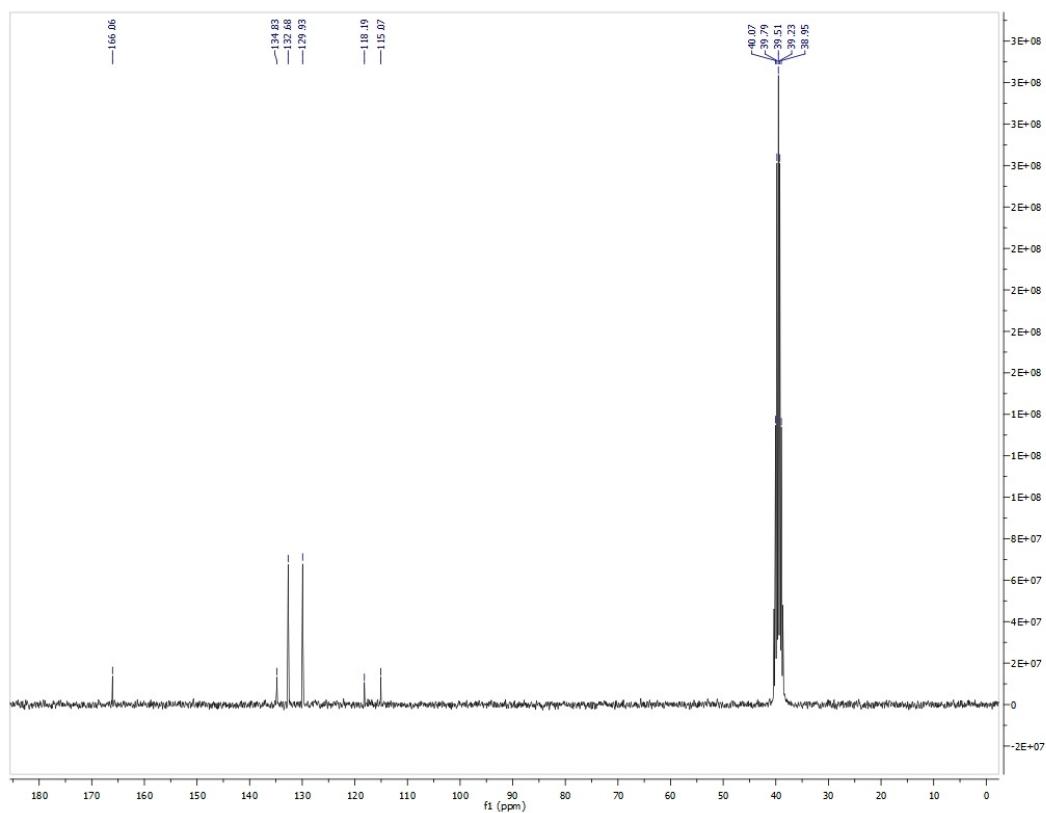
¹³C-NMR spectrum of **3q** (300 MHz, DMSO)



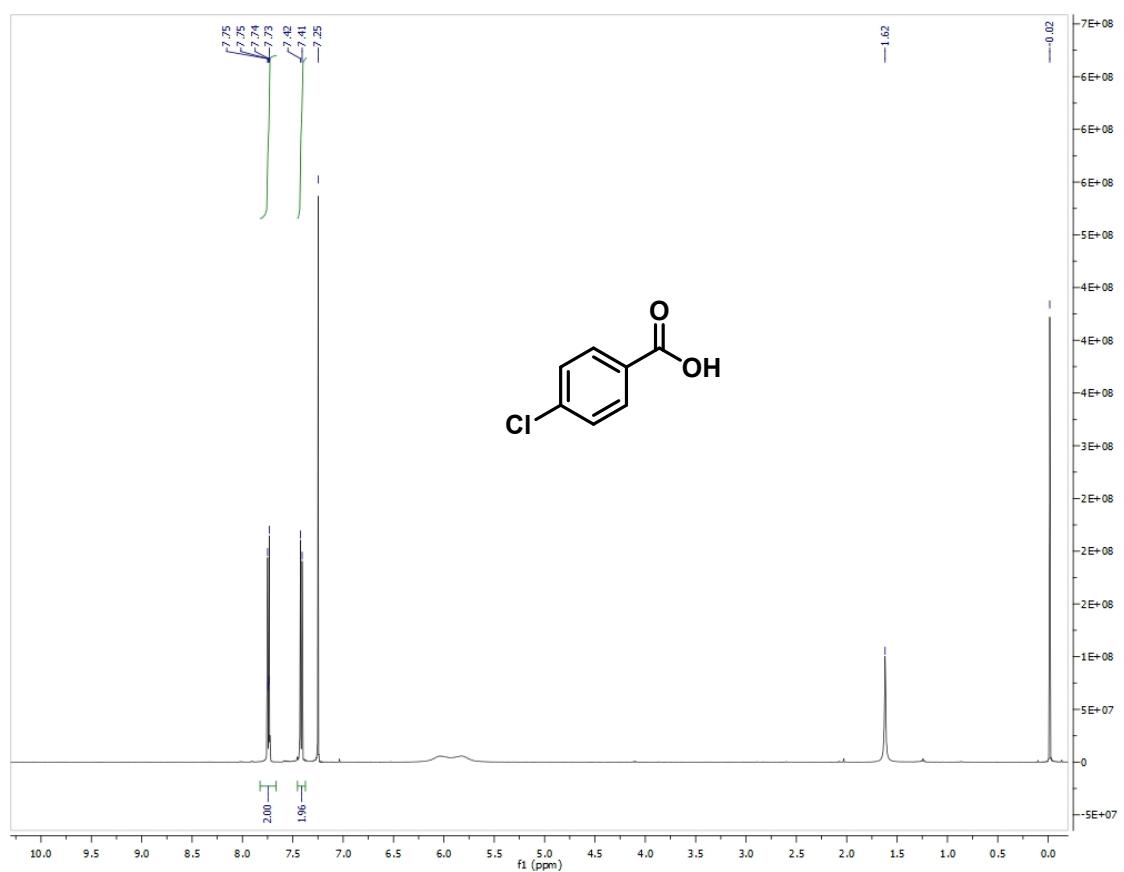




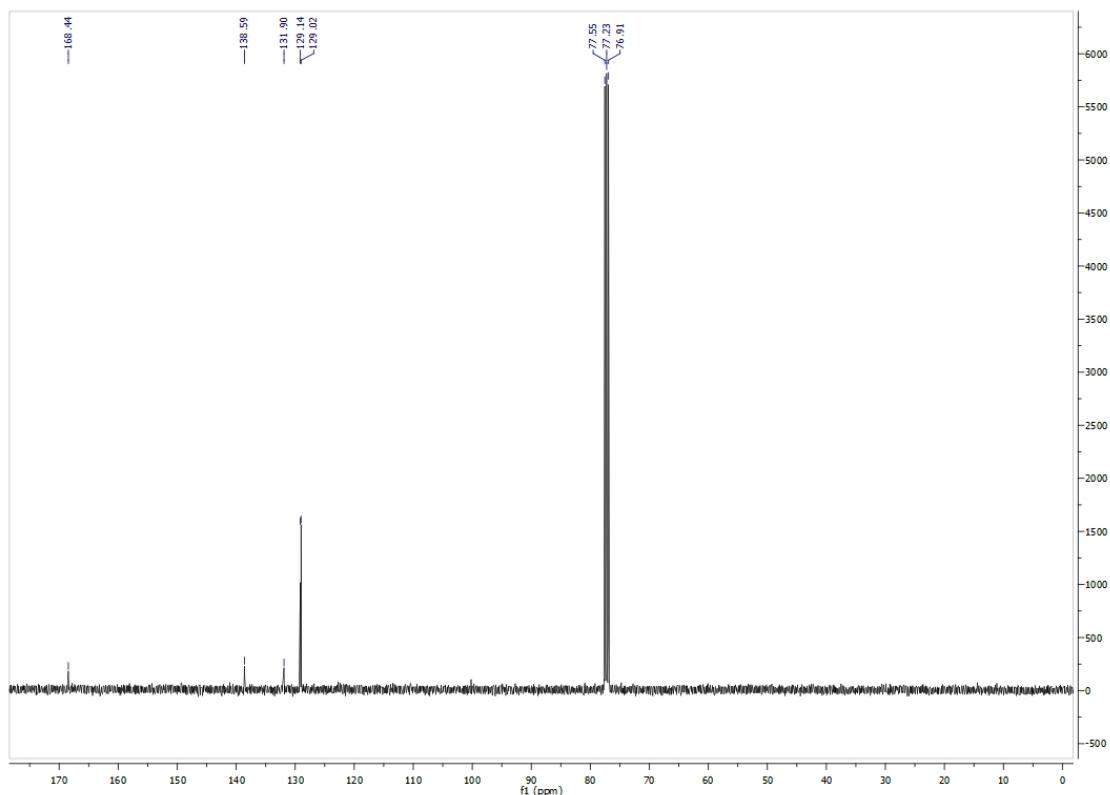
¹H-NMR spectrum of **3t** (300 MHz, DMSO)



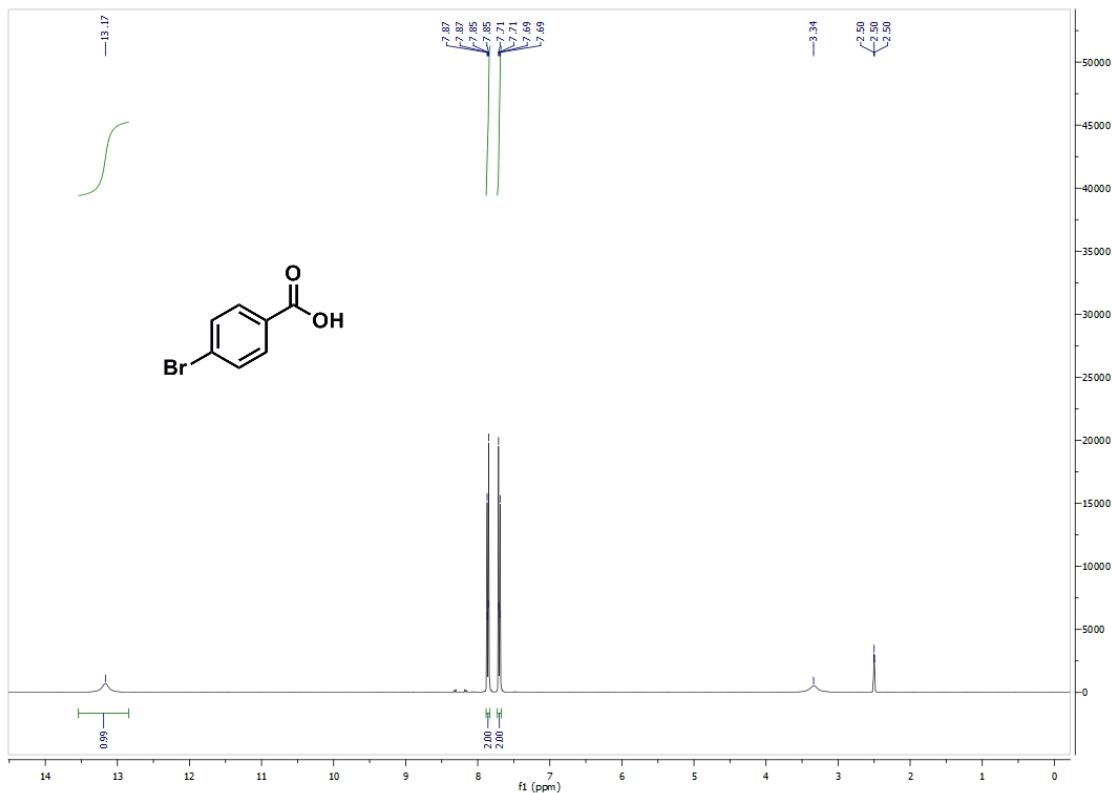
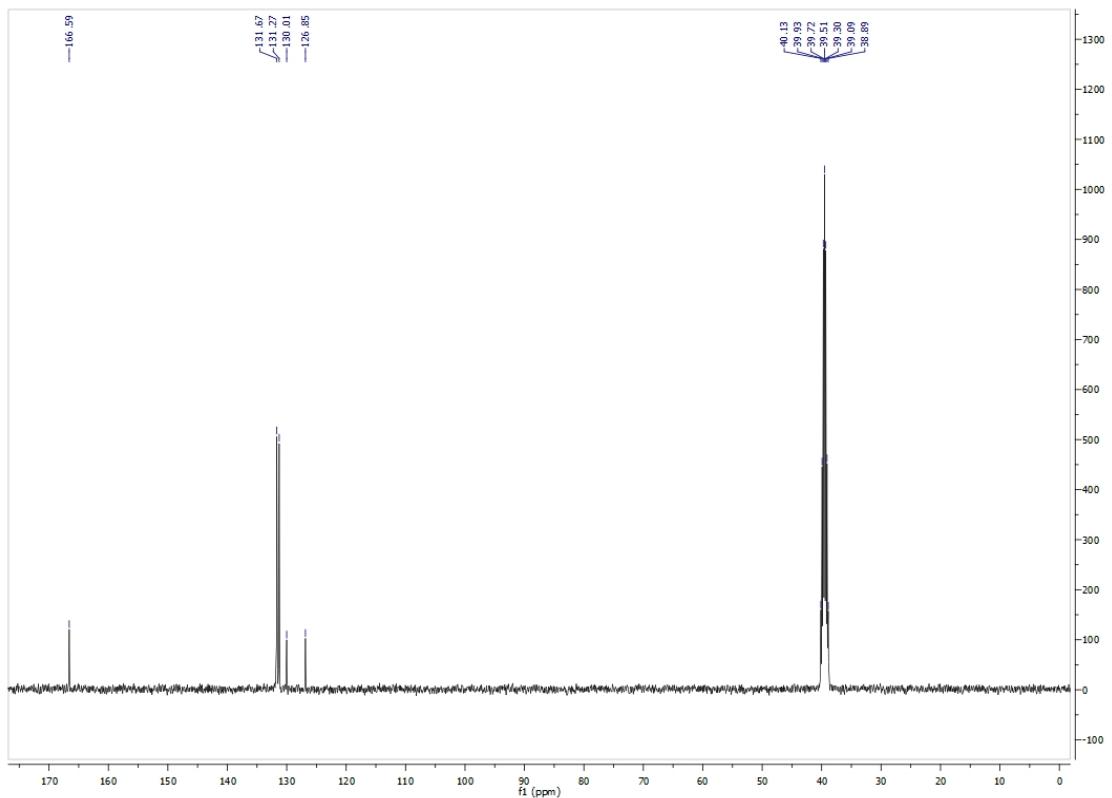
¹³C-NMR spectrum of **3t** (300 MHz, DMSO)

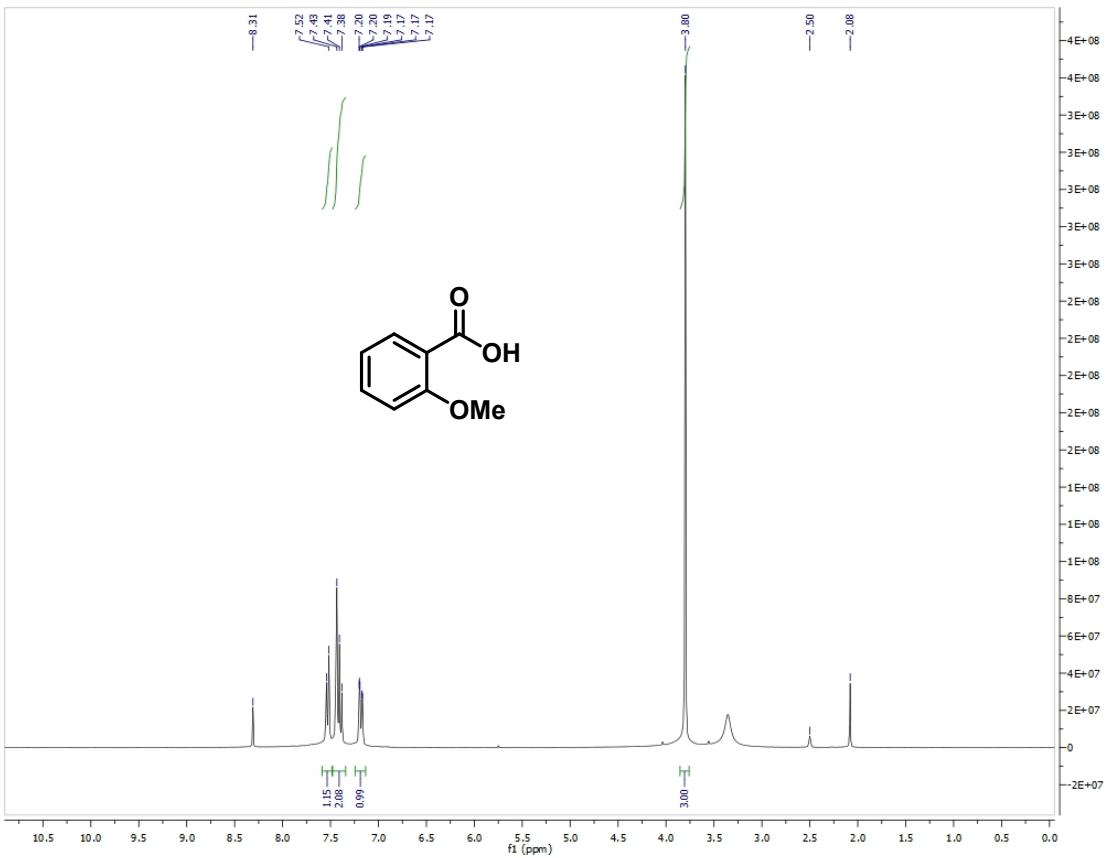
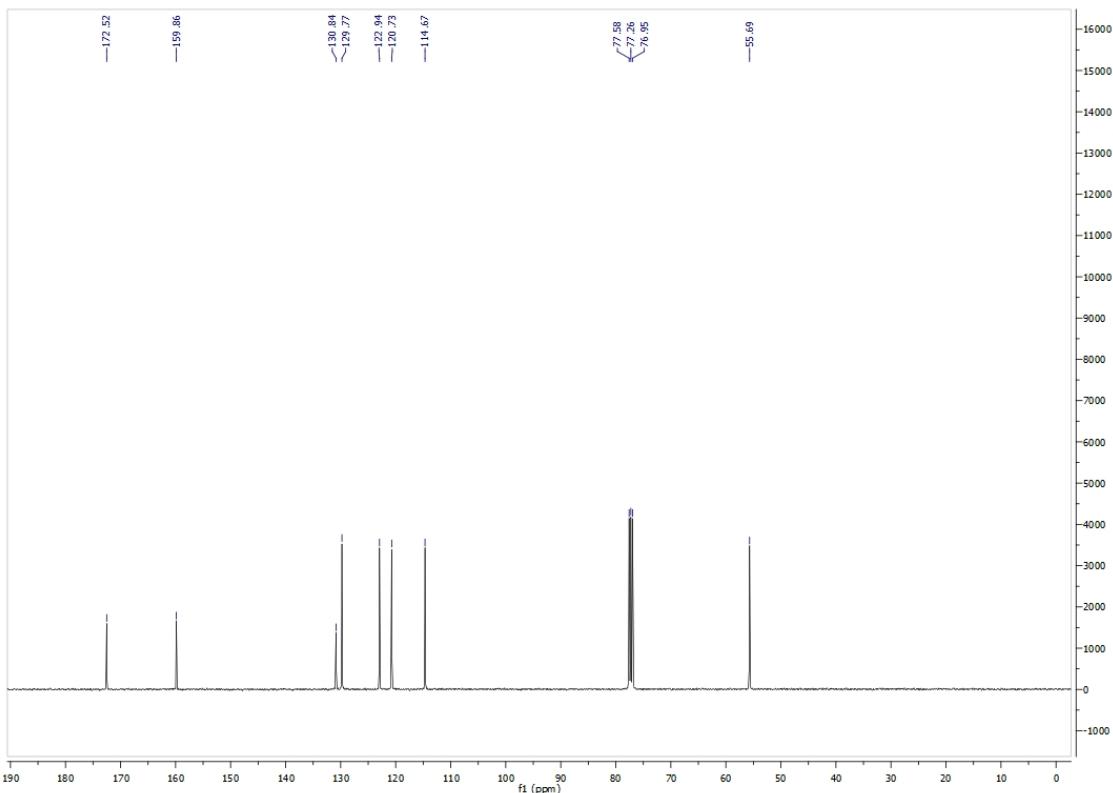


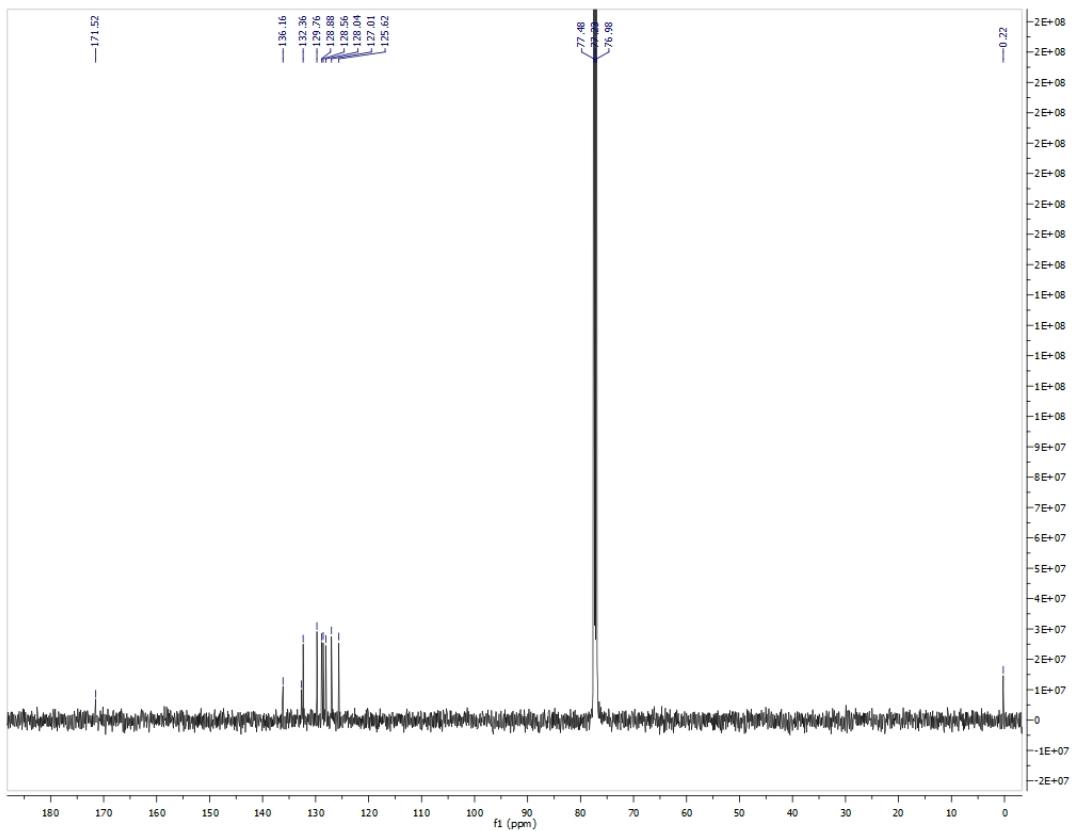
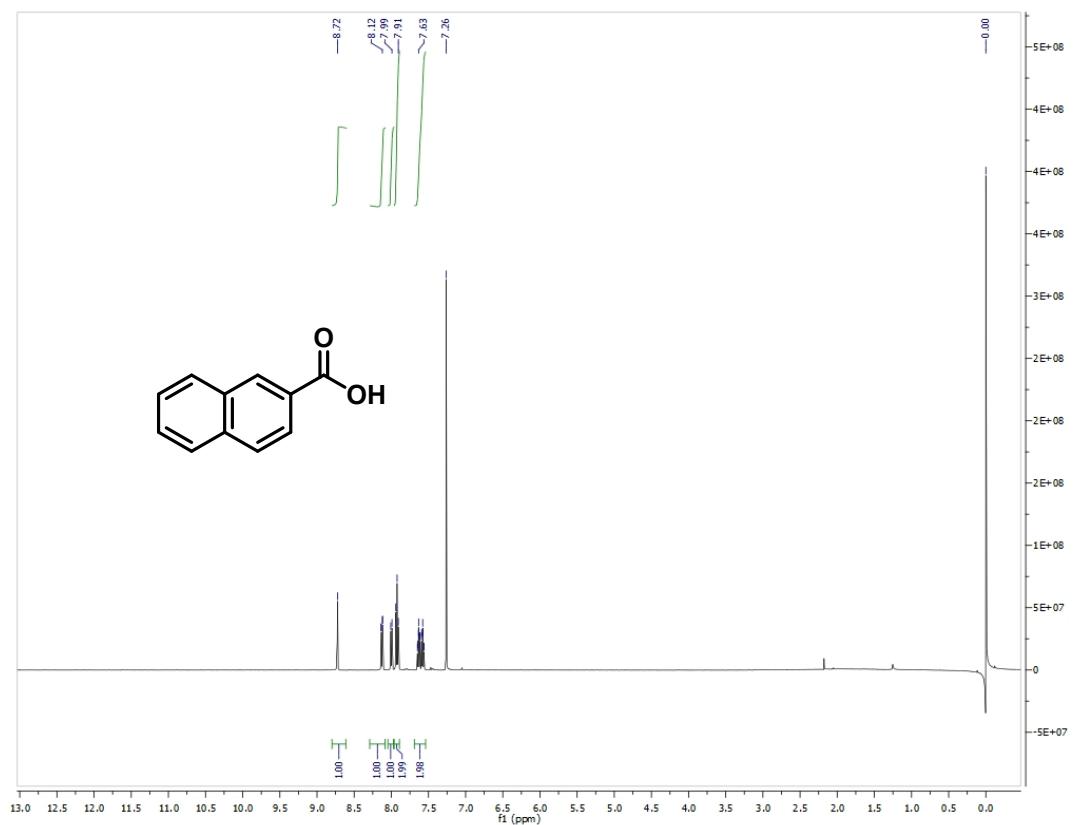
¹H-NMR spectrum of **3u** (300 MHz, CDCl₃)

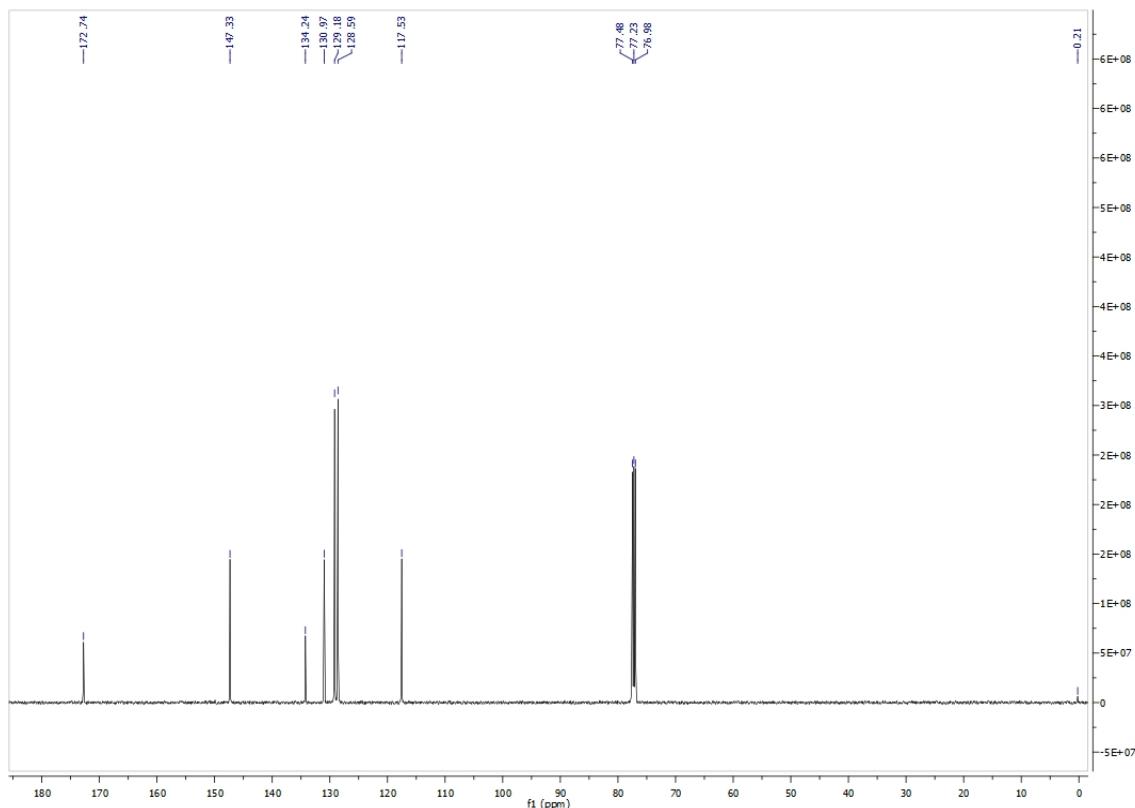
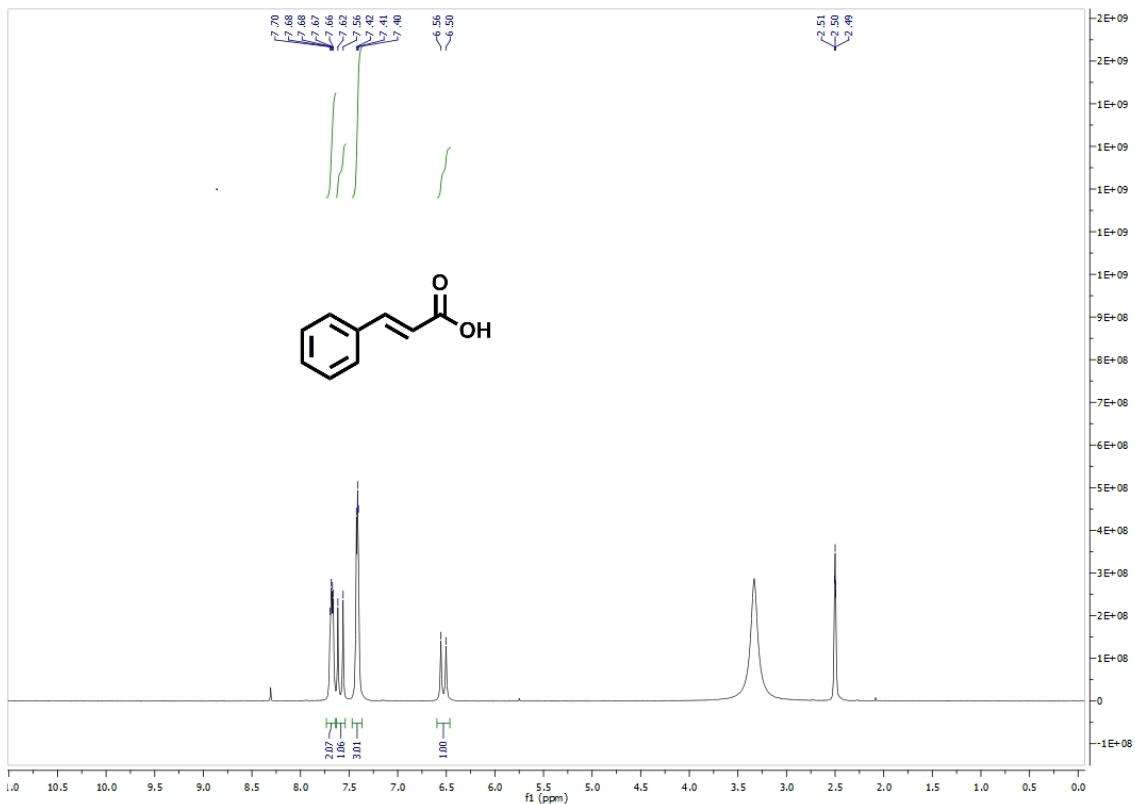


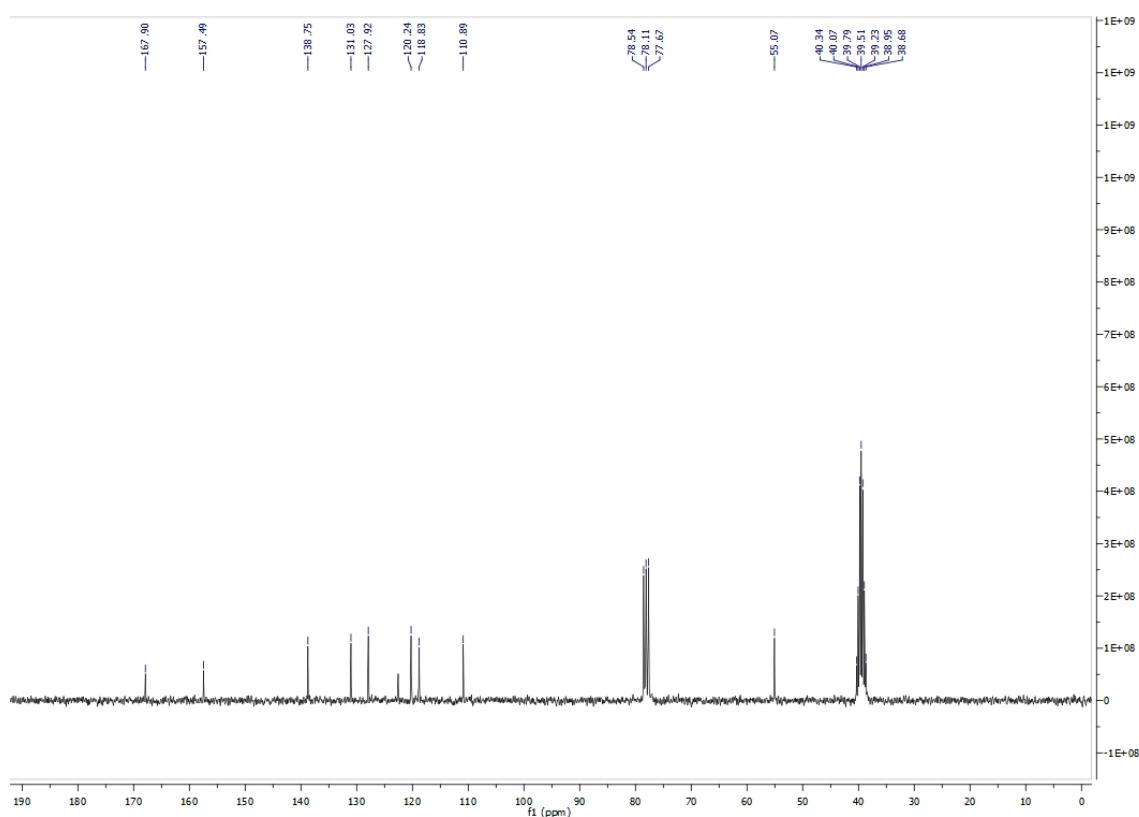
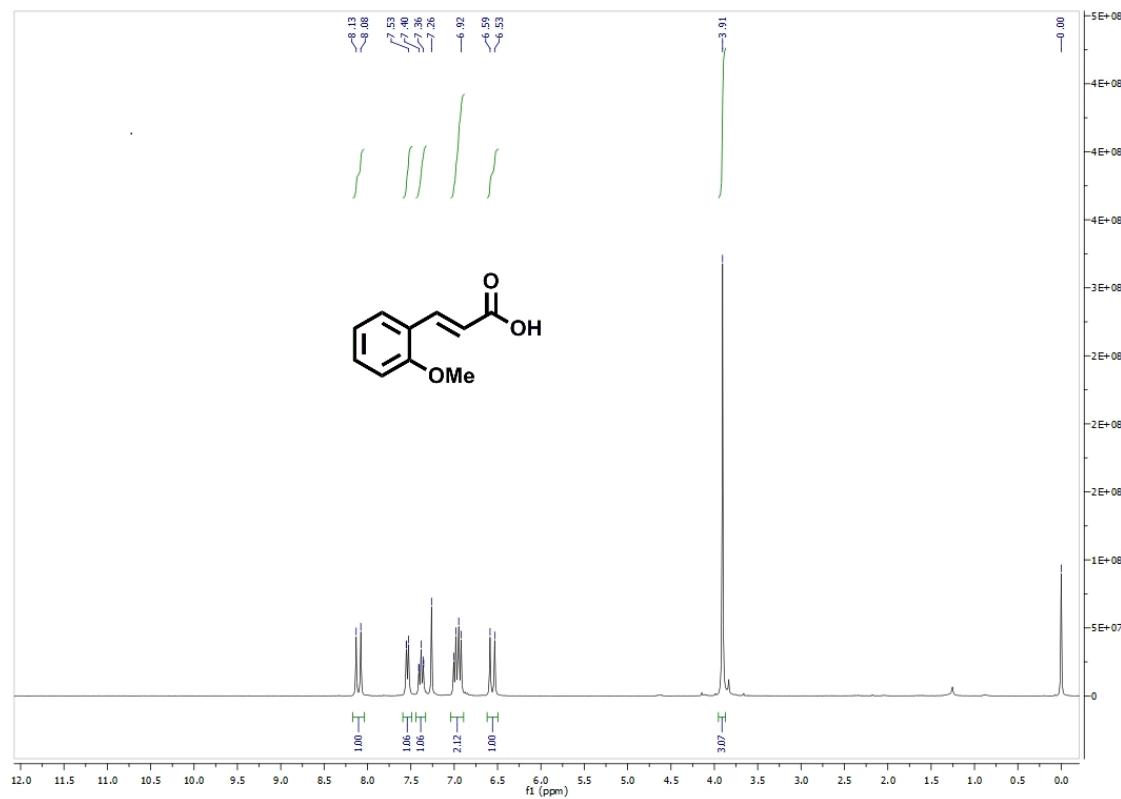
¹³C-NMR spectrum of **3u** (300 MHz, CDCl₃)

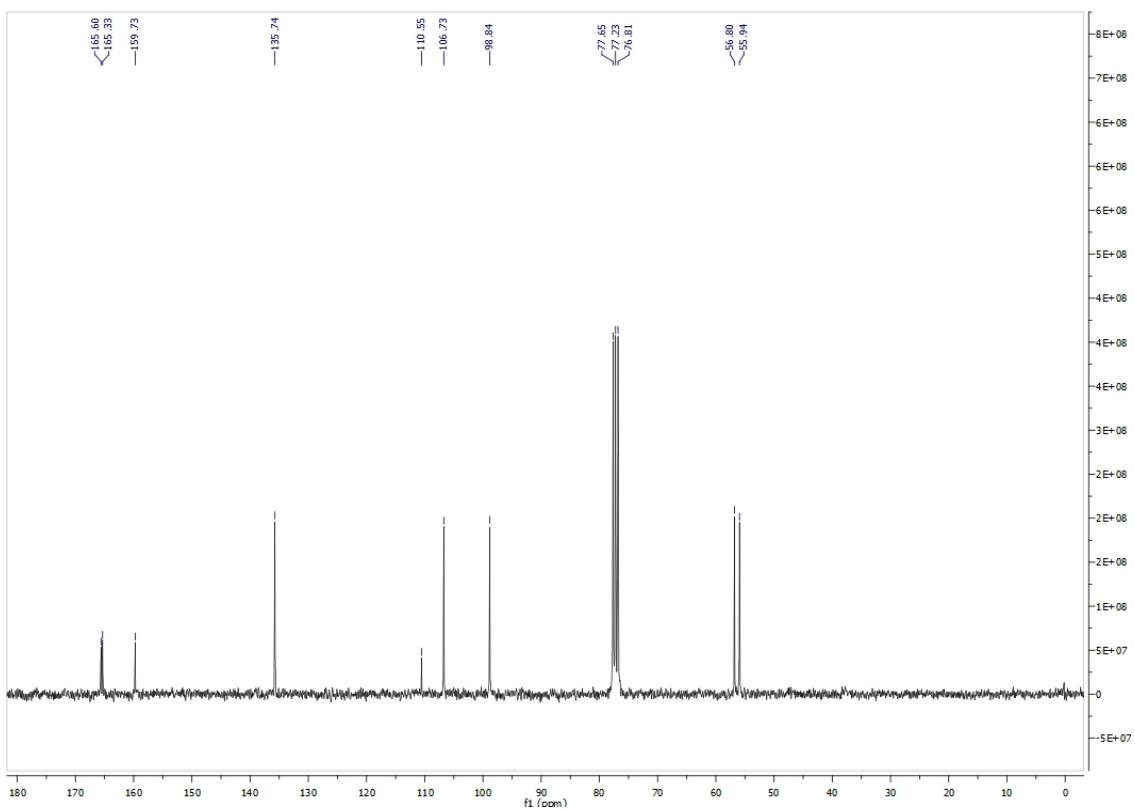
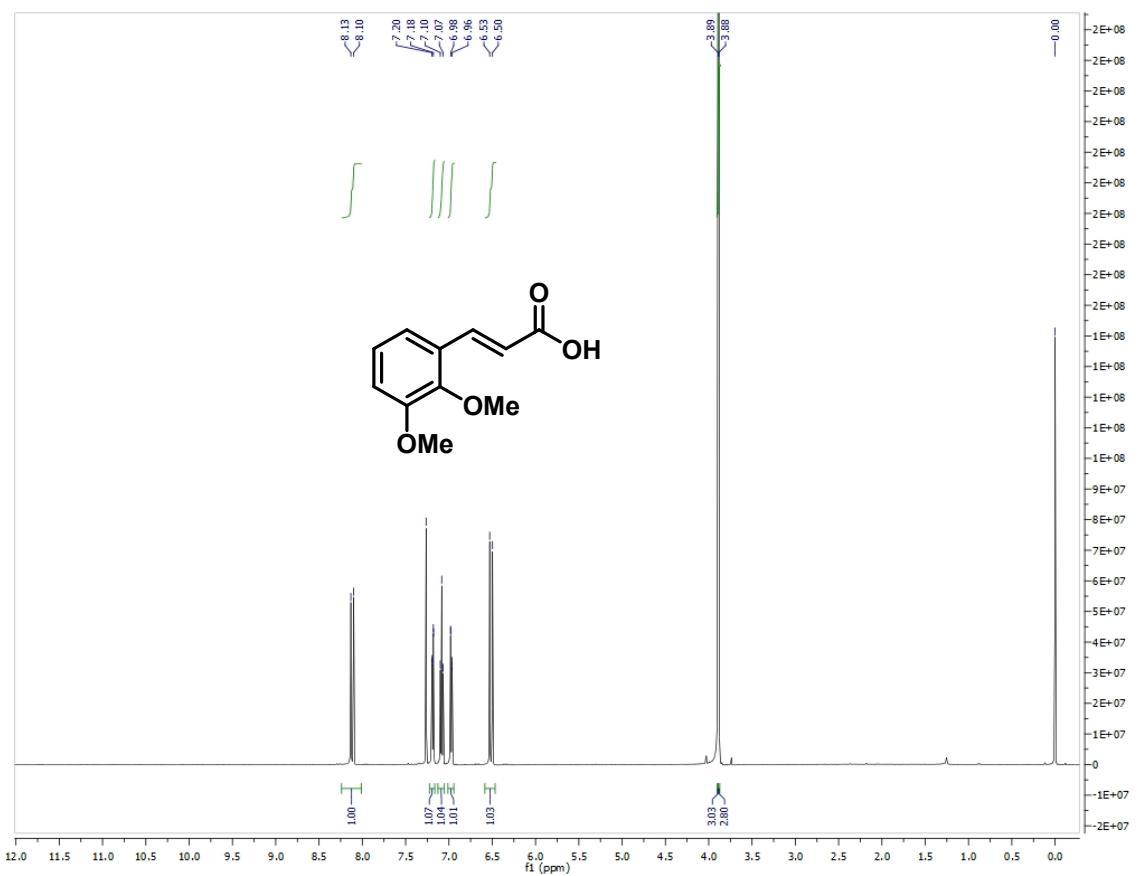
¹H-NMR spectrum of **3v** (300 MHz, DMSO)¹³C -NMR spectrum of **3v** (300 MHz, DMSO)

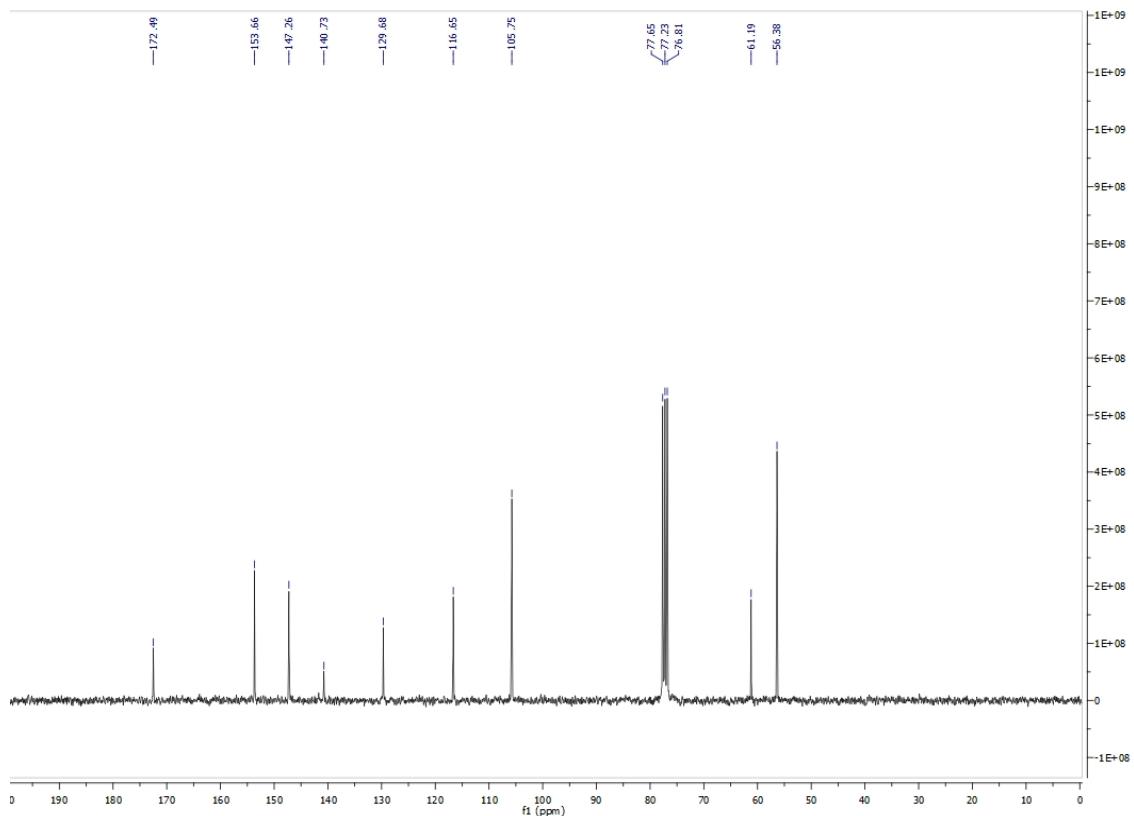
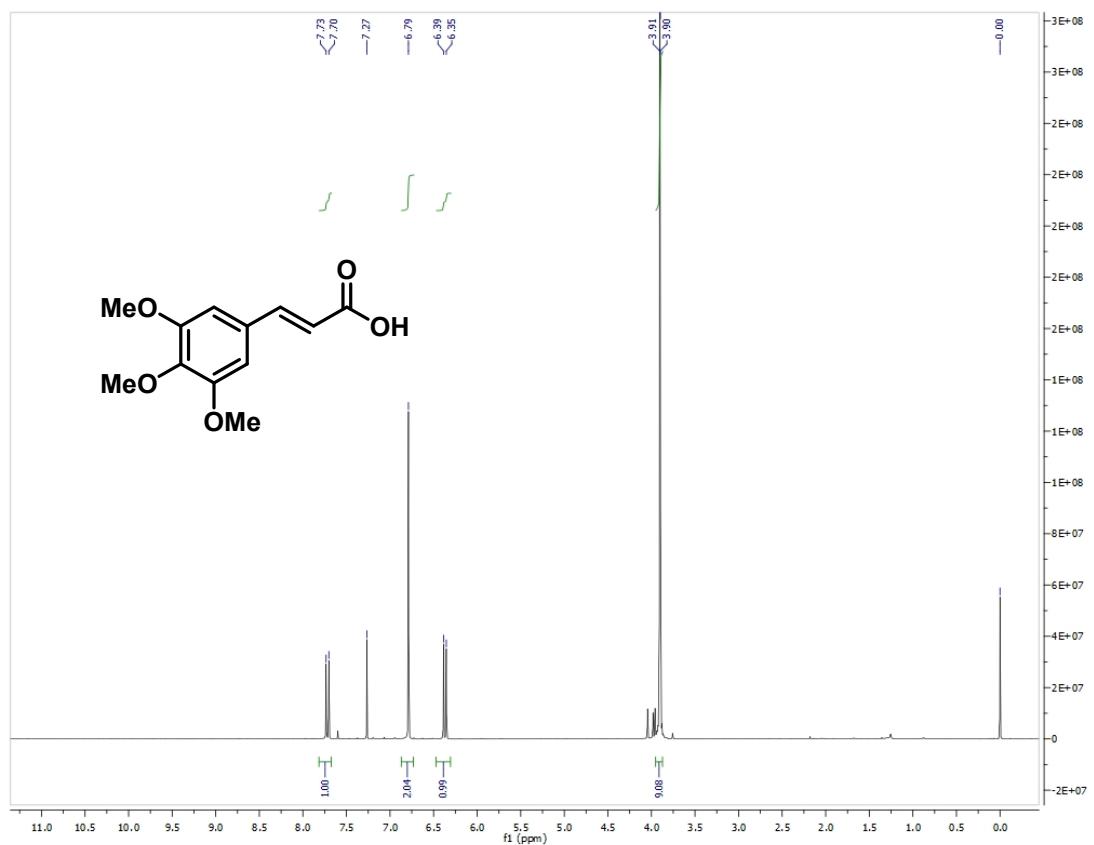
¹H-NMR spectrum of **3w** (300 MHz, CDCl₃)¹³C -NMR spectrum of **3w** (300 MHz, CDCl₃)

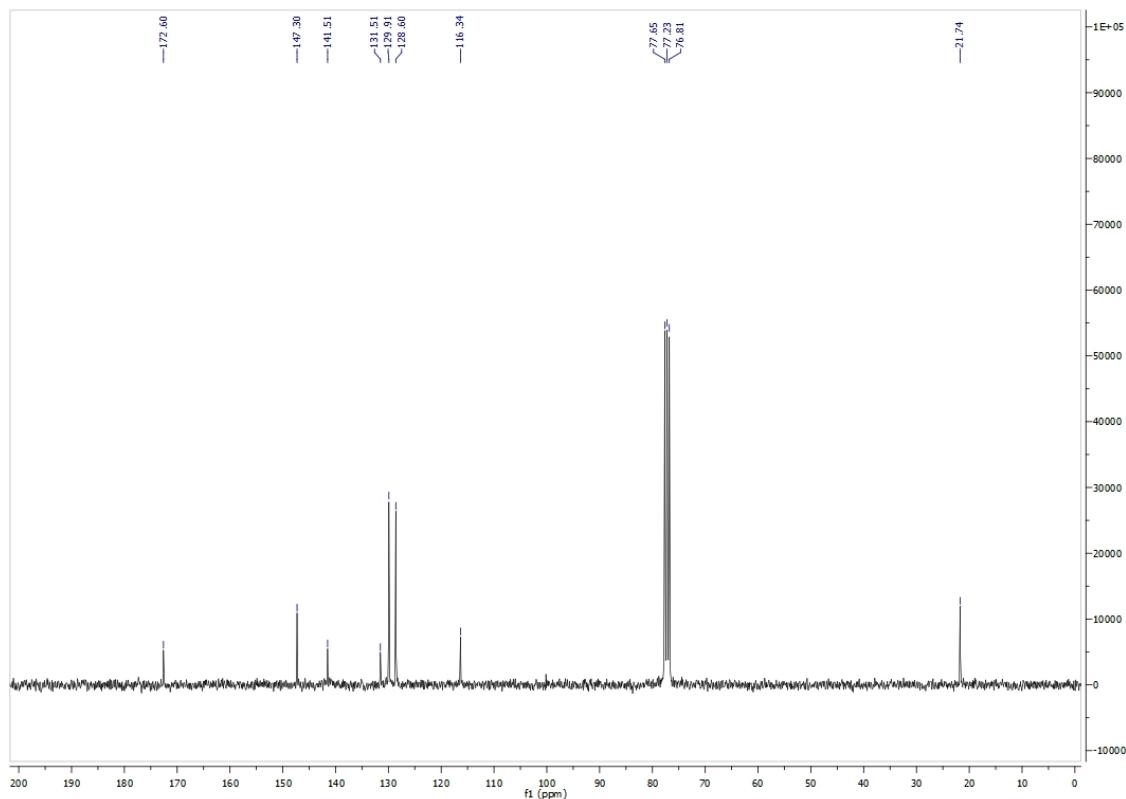
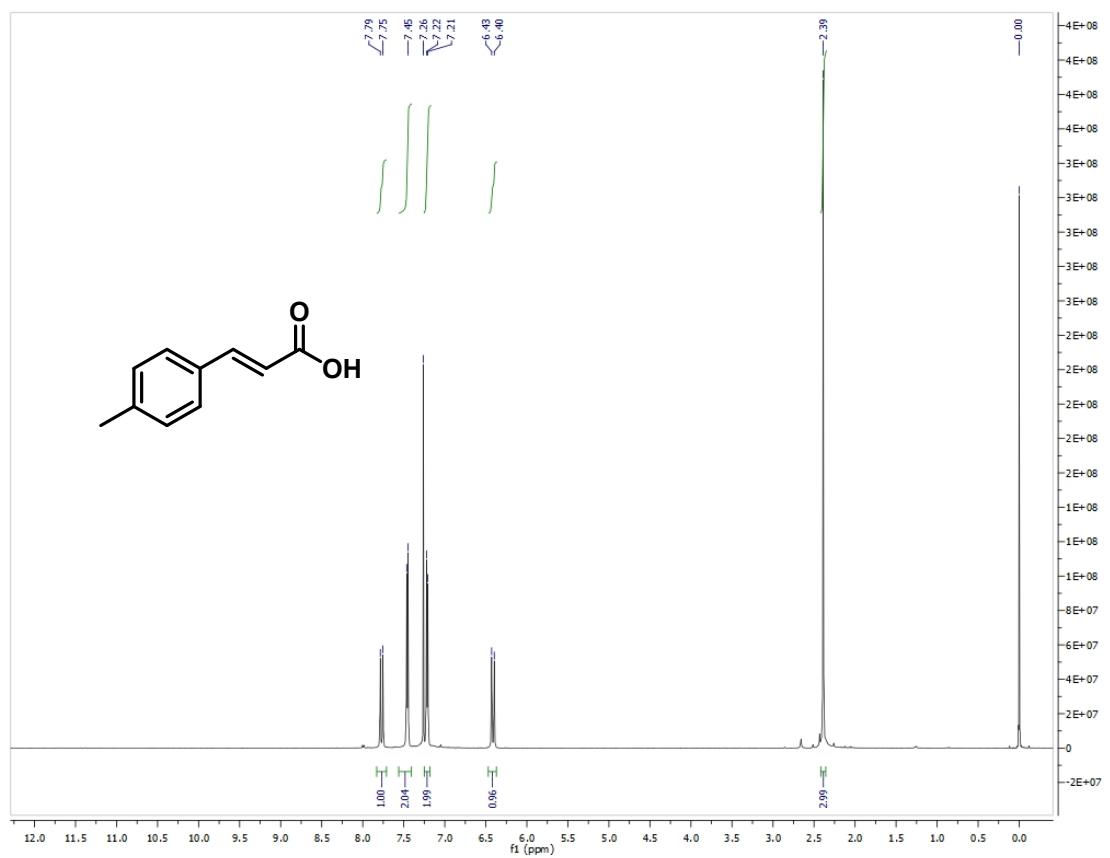


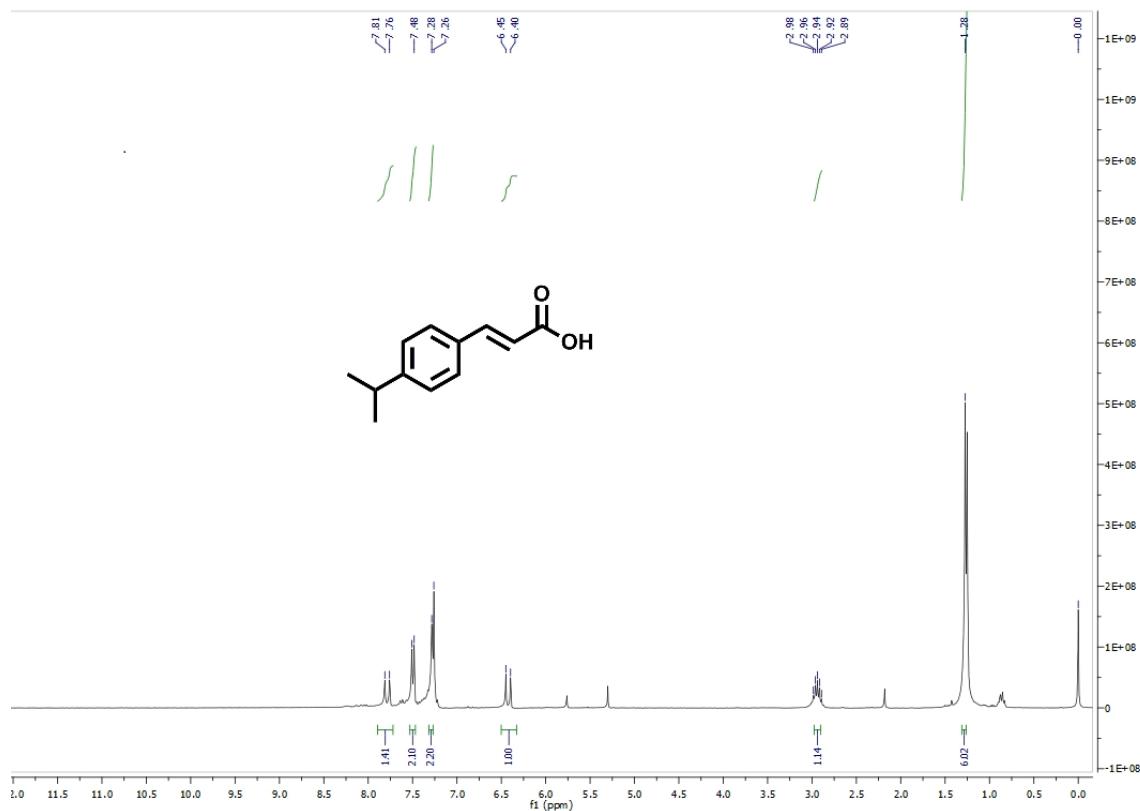
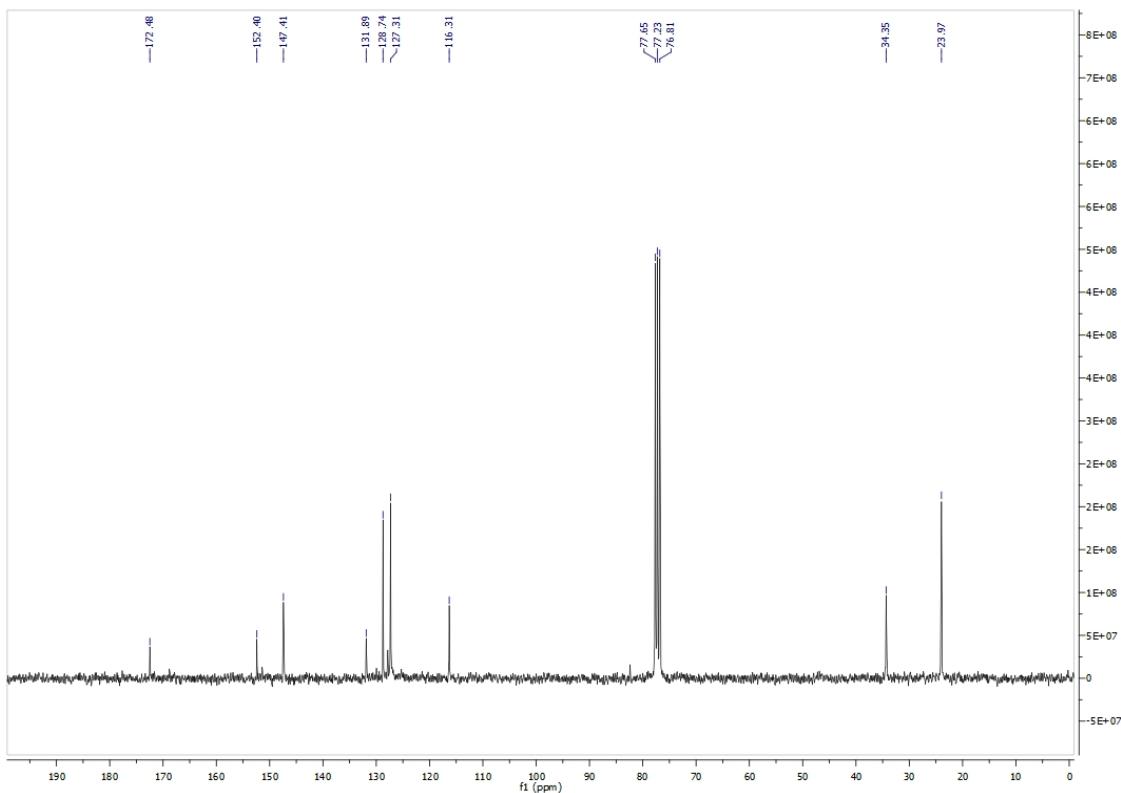


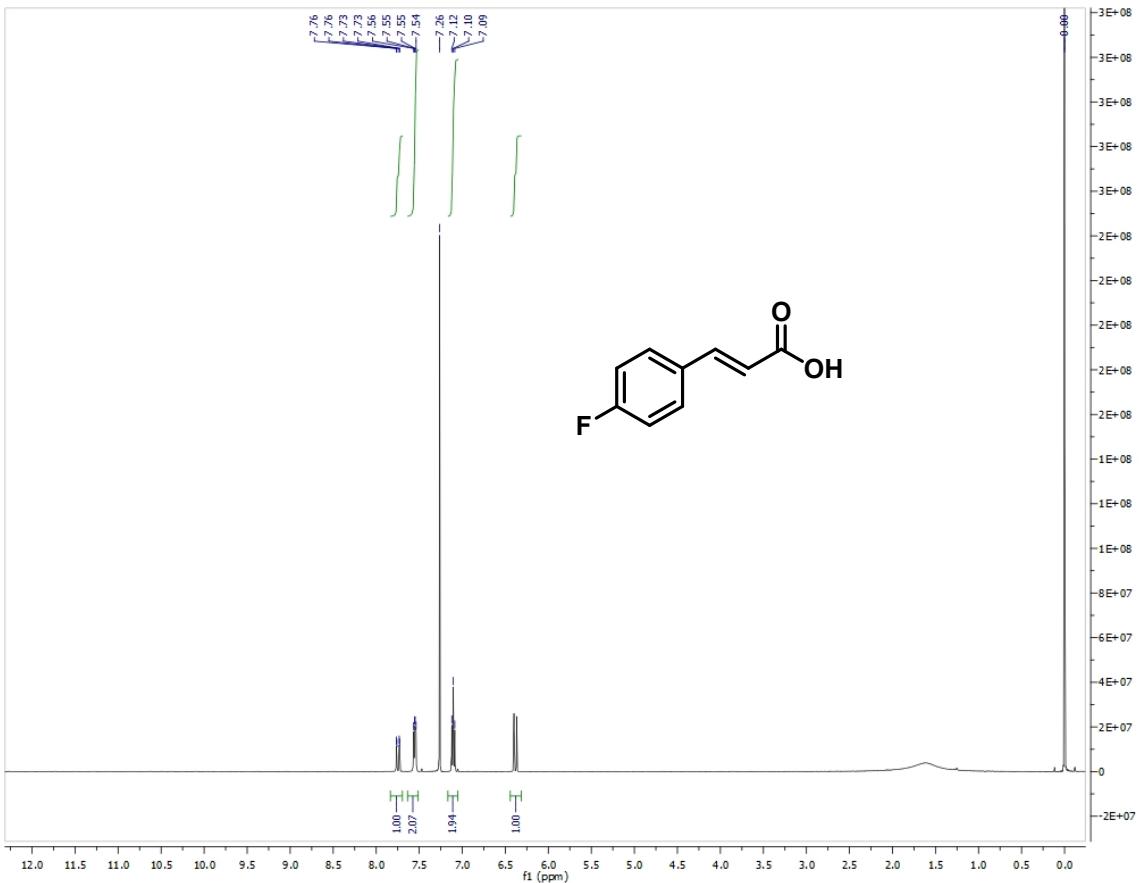




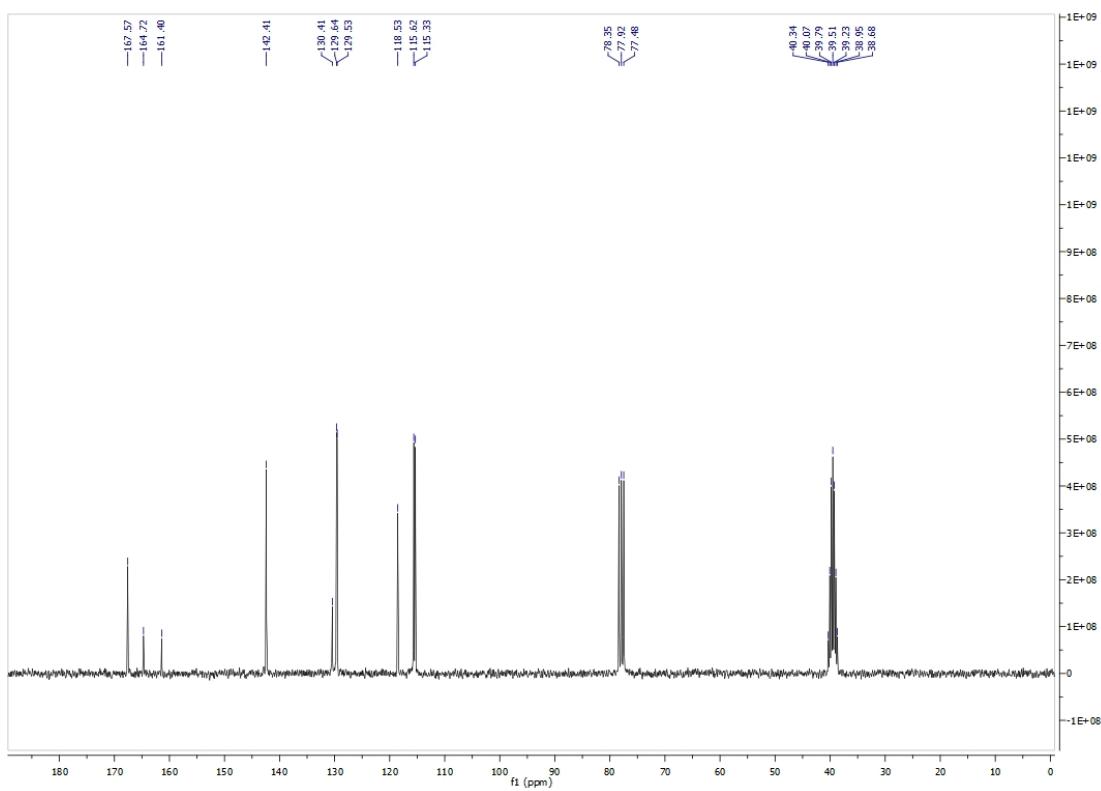




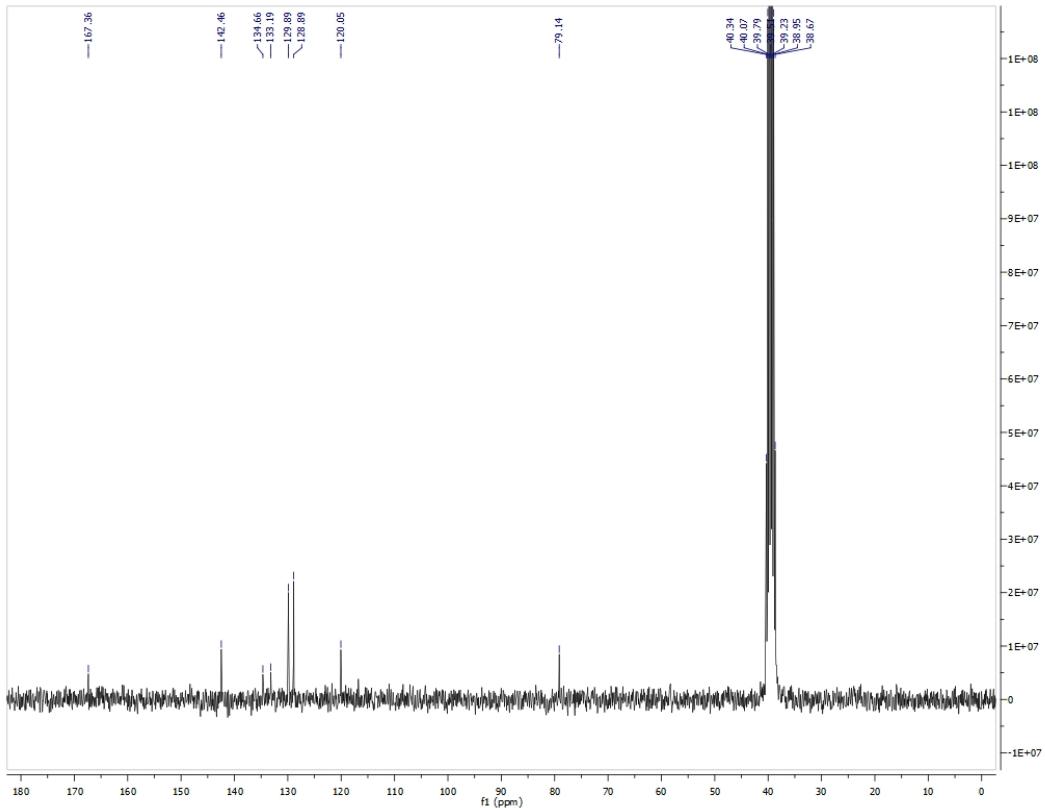
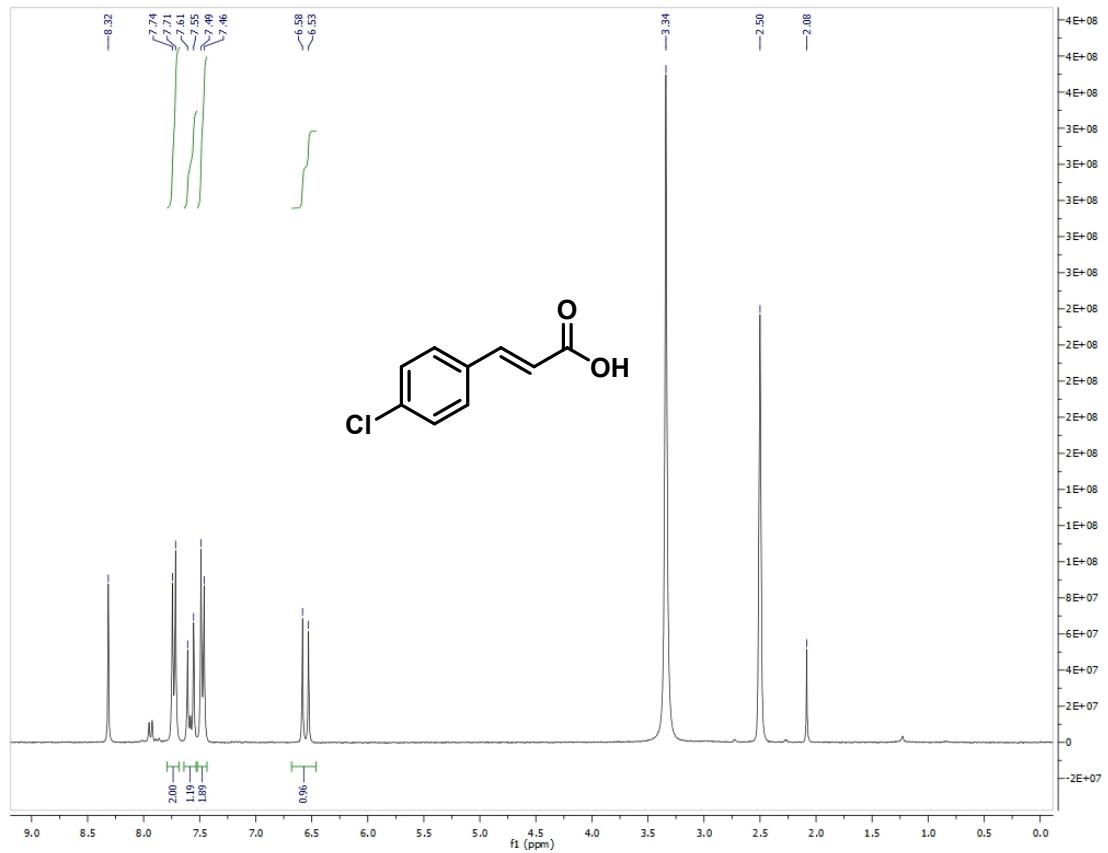
¹H-NMR spectrum of 7f (300 MHz, CDCl₃)¹³C-NMR spectrum of 7f (300 MHz, CDCl₃)

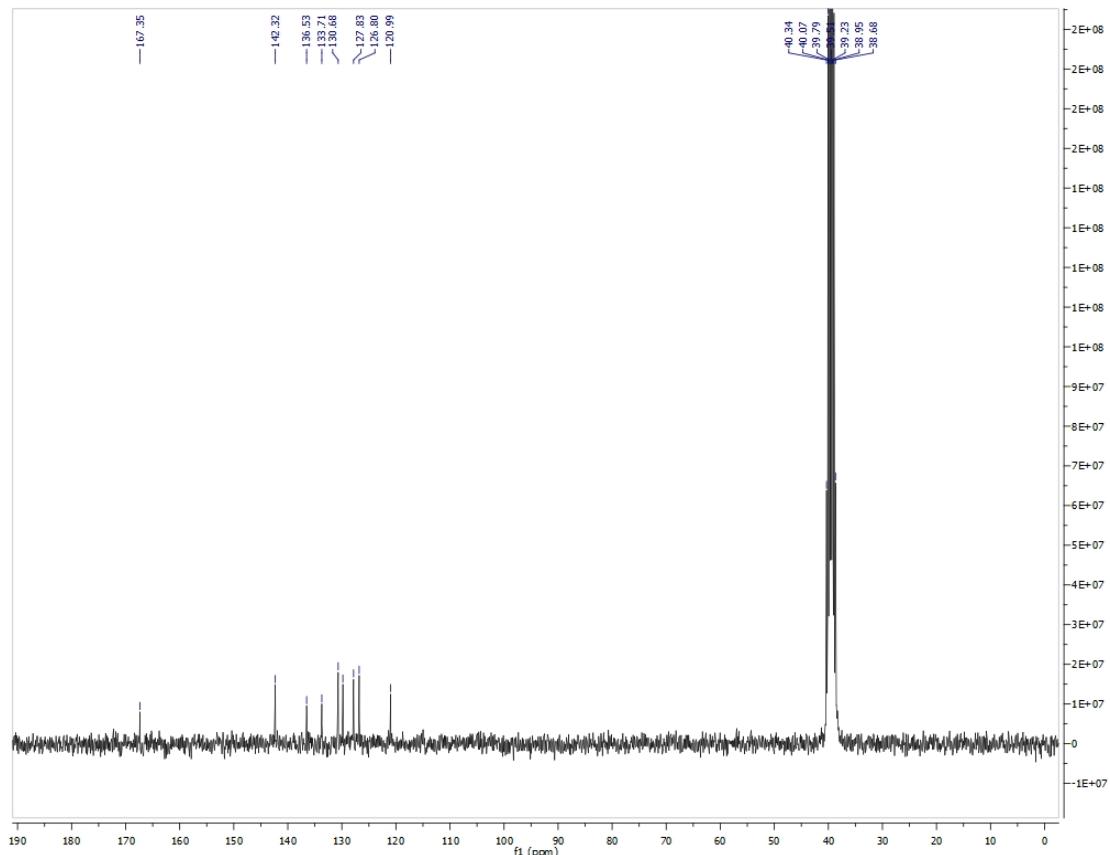


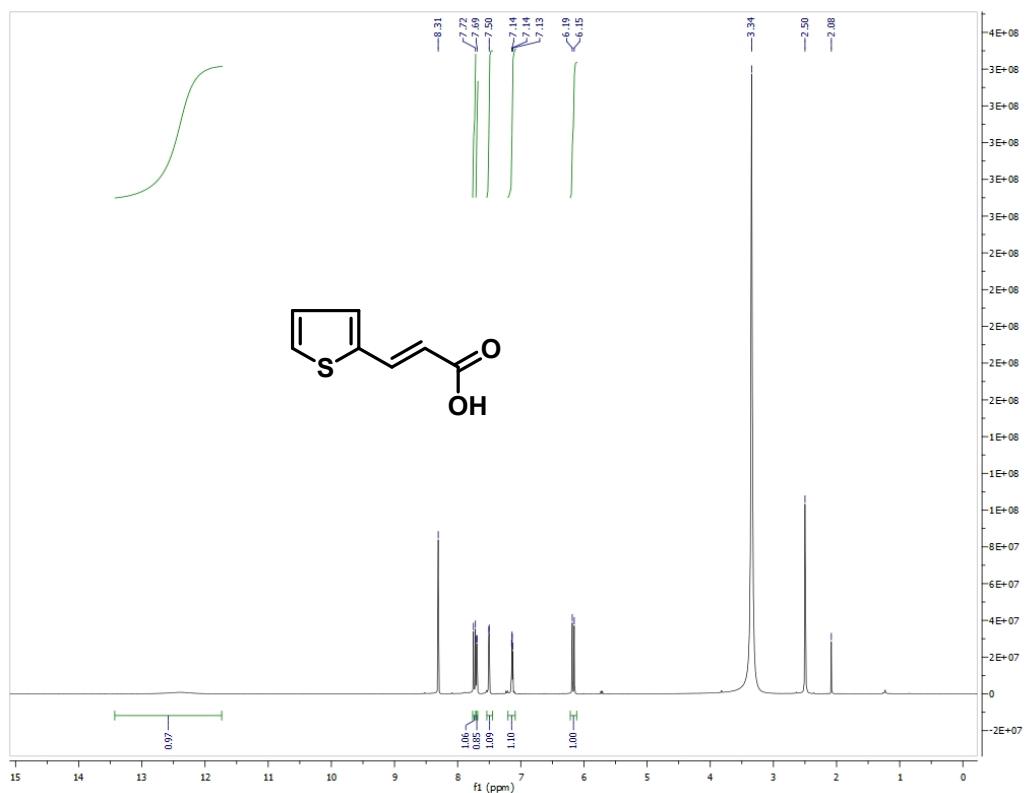
^1H -NMR spectrum of **7g** (300 MHz, CDCl_3)



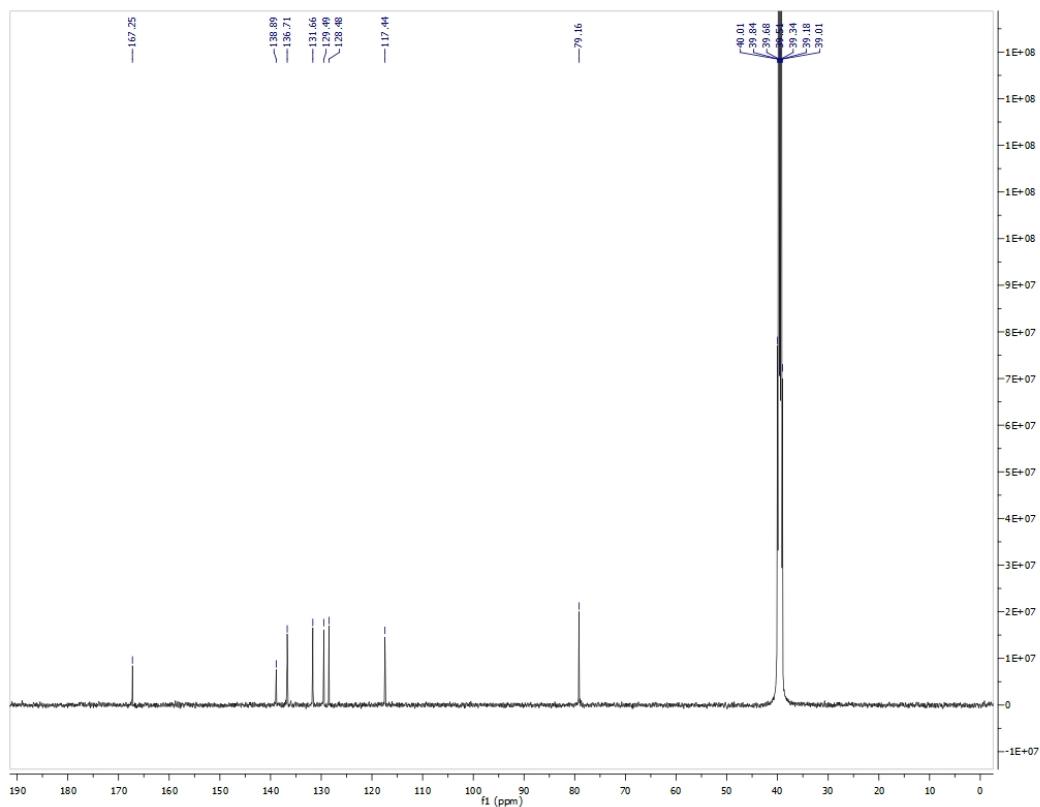
^{13}C -NMR spectrum of **7g** (300 MHz, $\text{CDCl}_3+\text{DMSO}$)



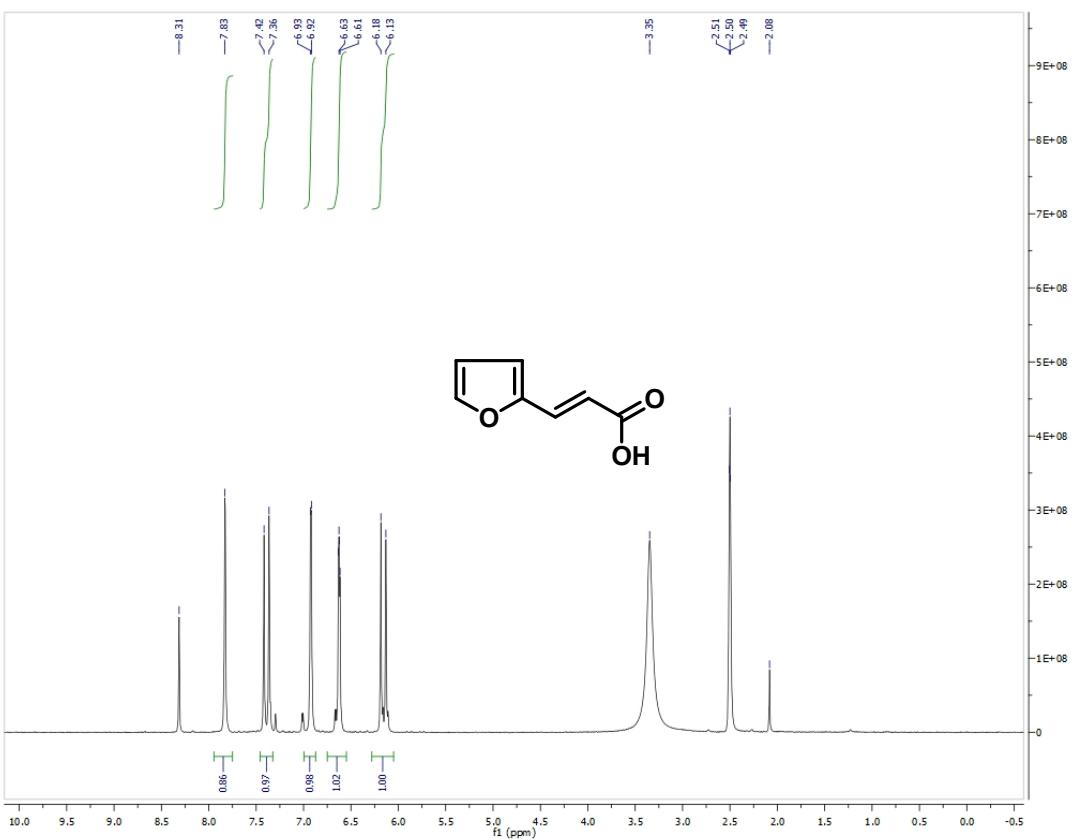
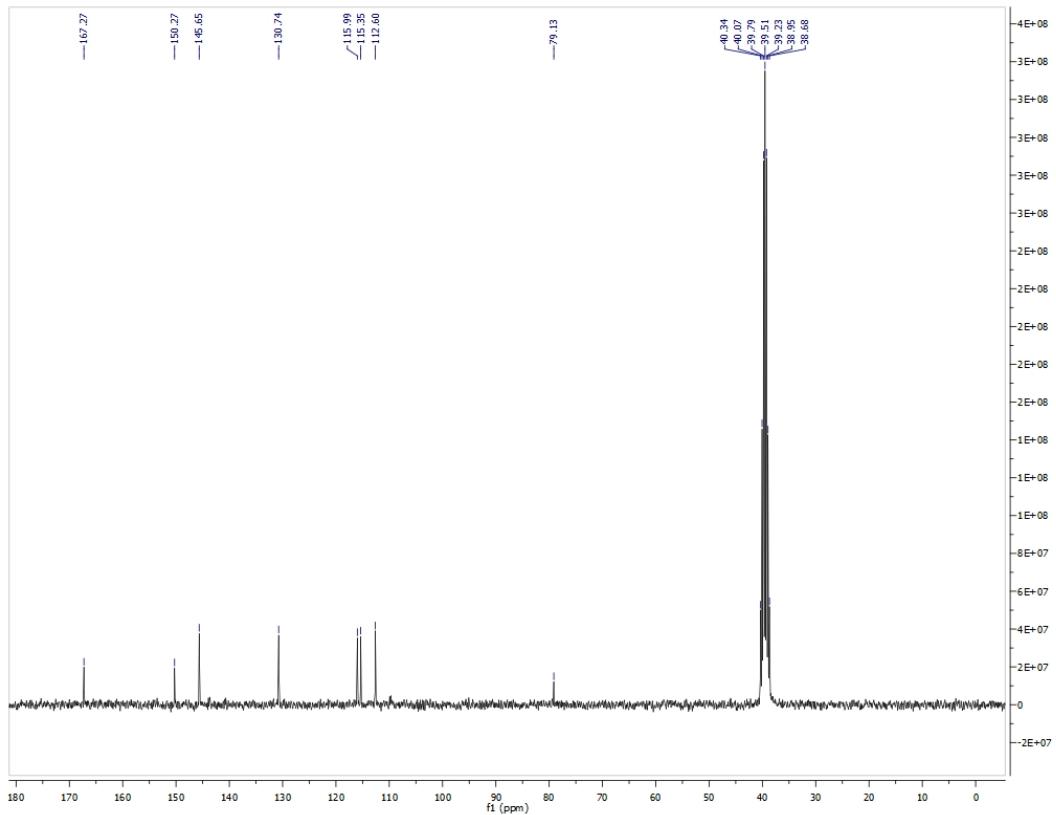
¹H-NMR spectrum of **7i** (300 MHz, CDCl₃)¹³C-NMR spectrum of **7i** (300 MHz, DMSO)

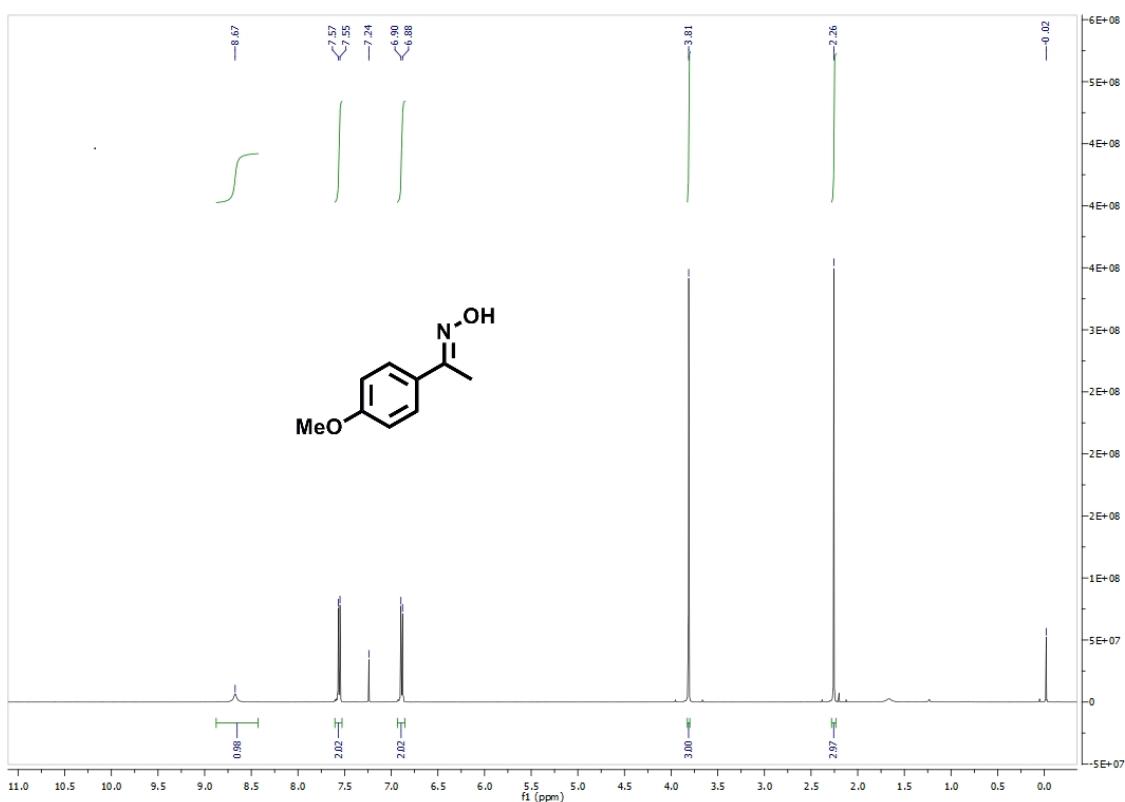
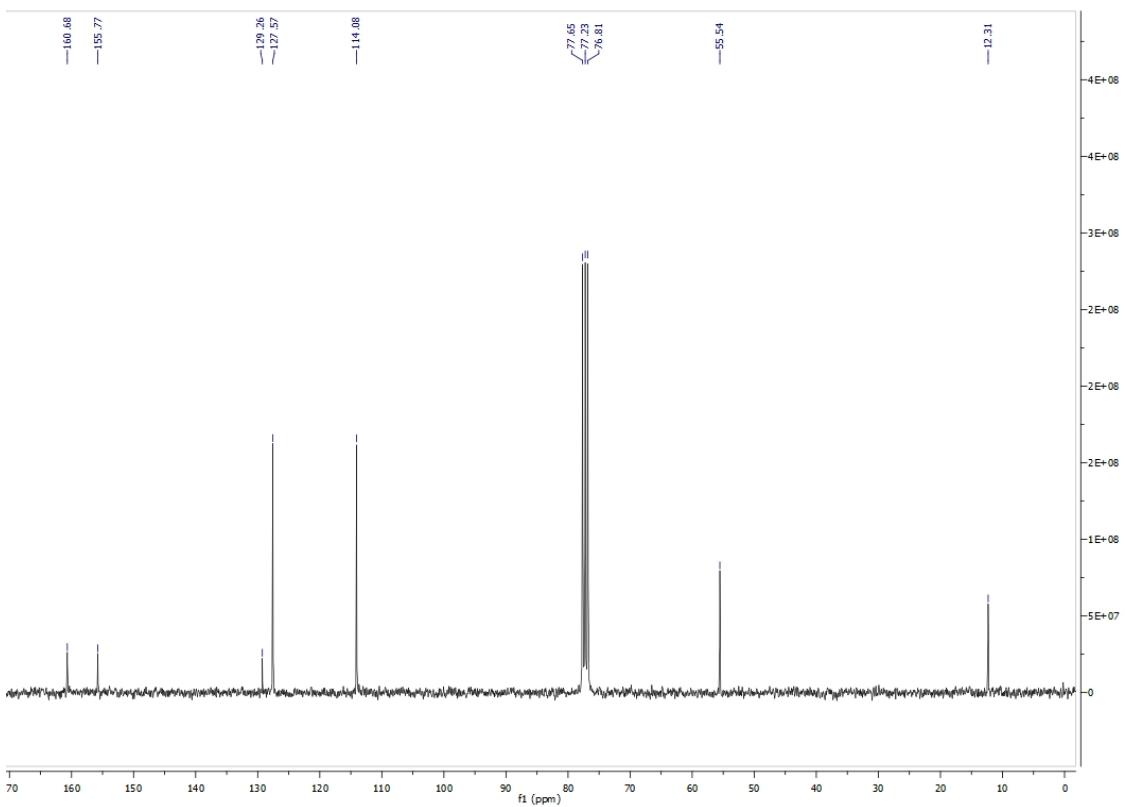


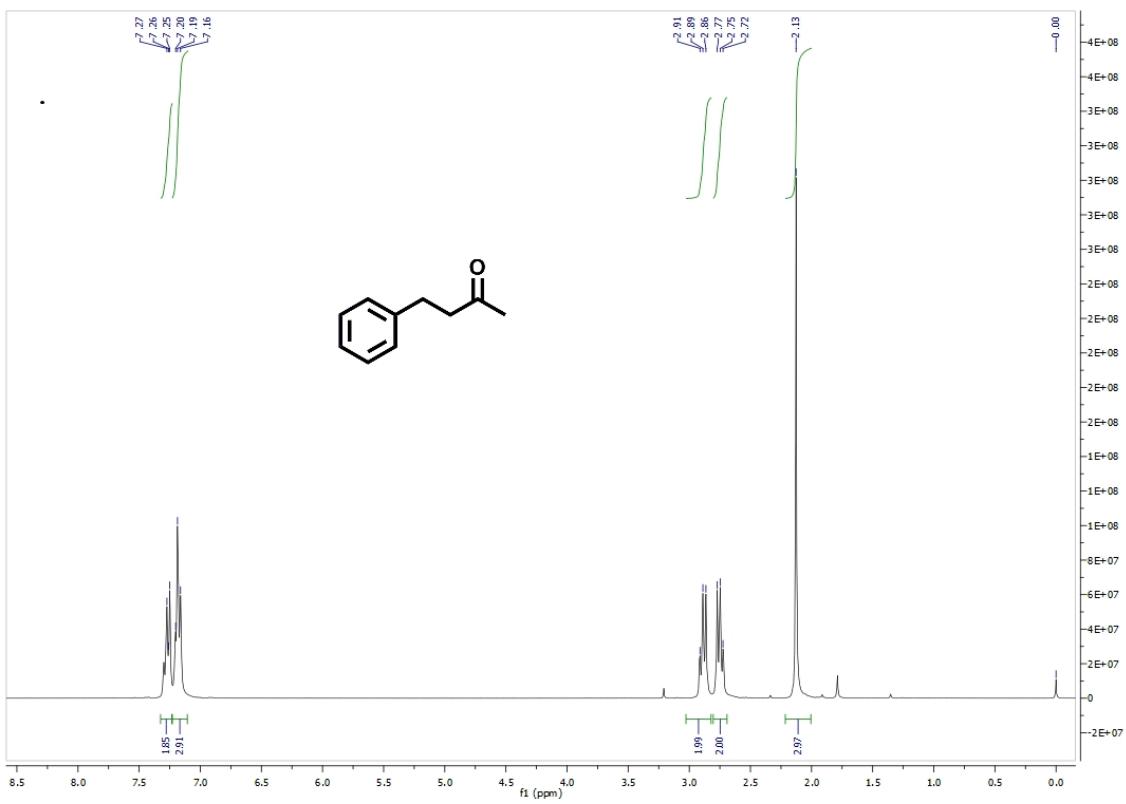
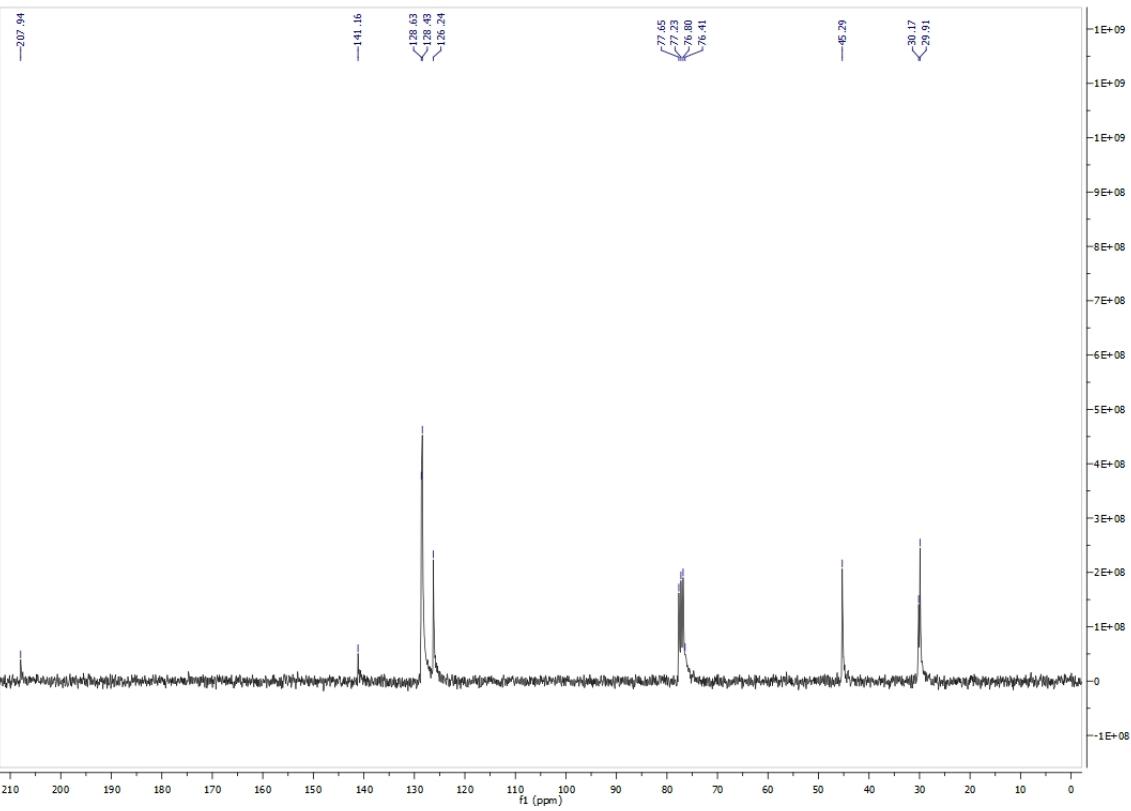
¹H-NMR spectrum of **7j** (300 MHz, DMSO)

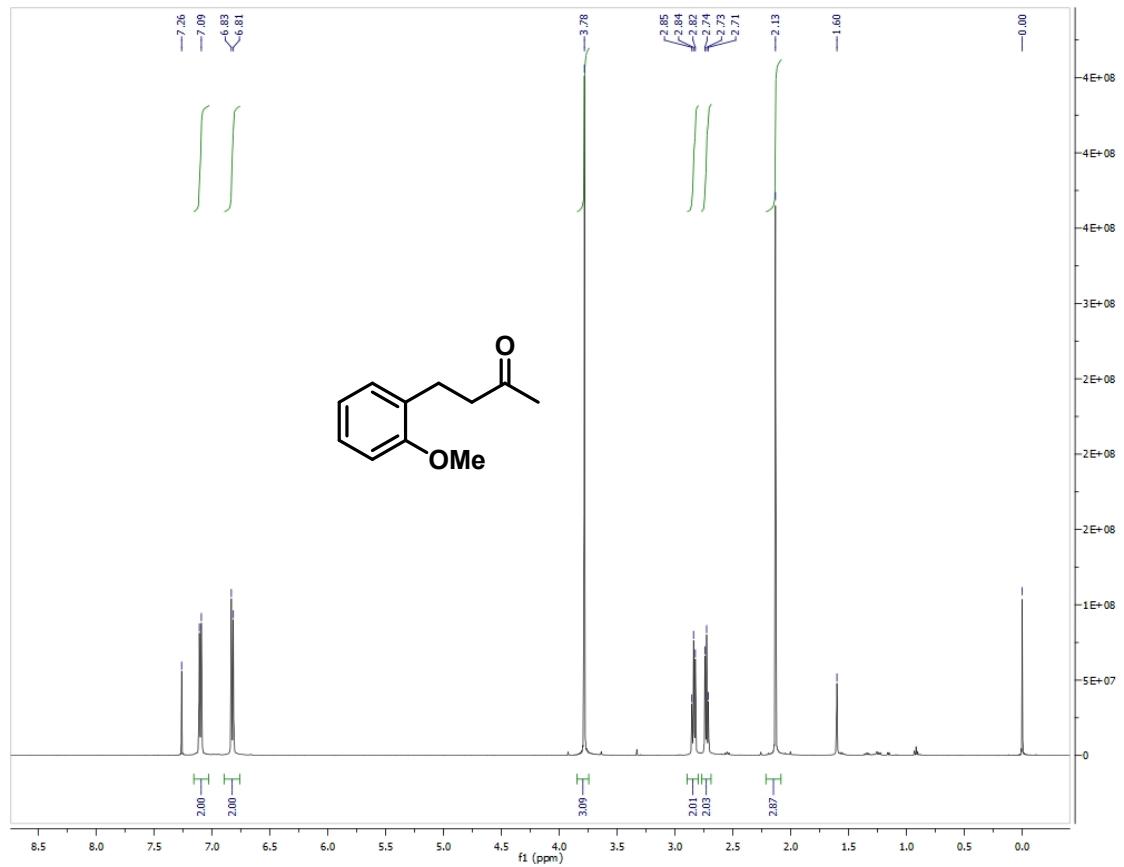
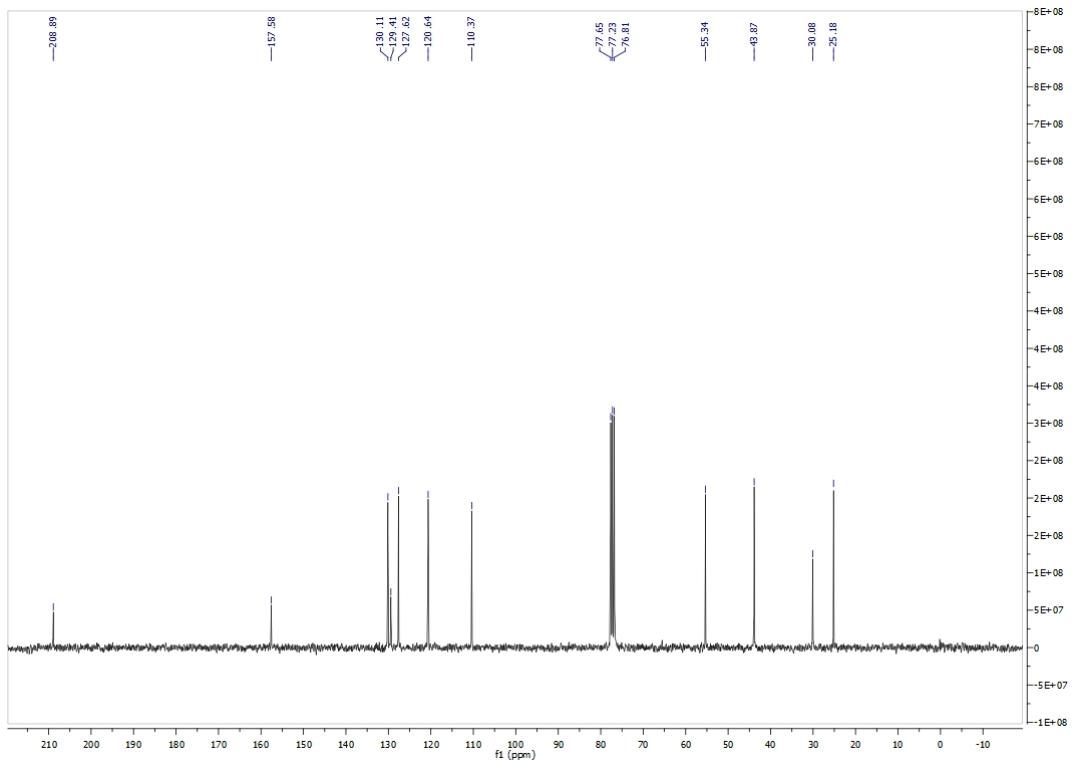


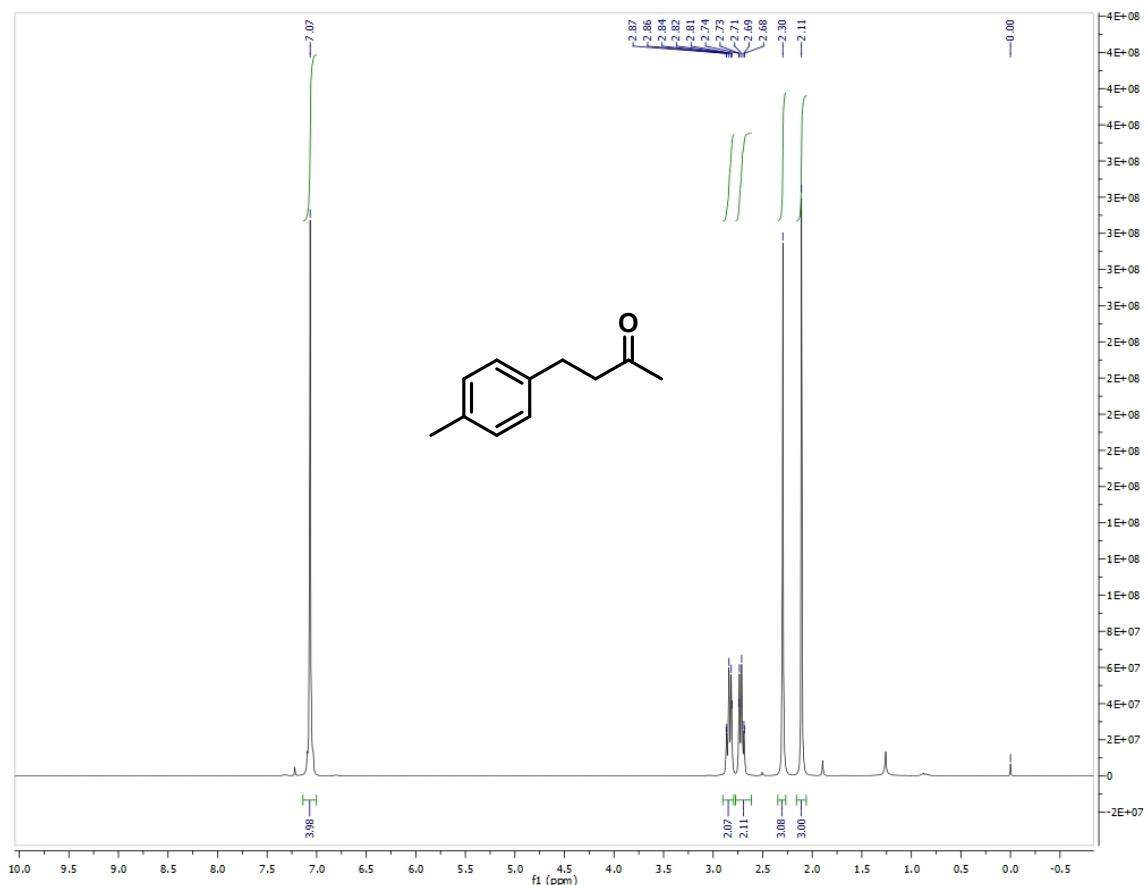
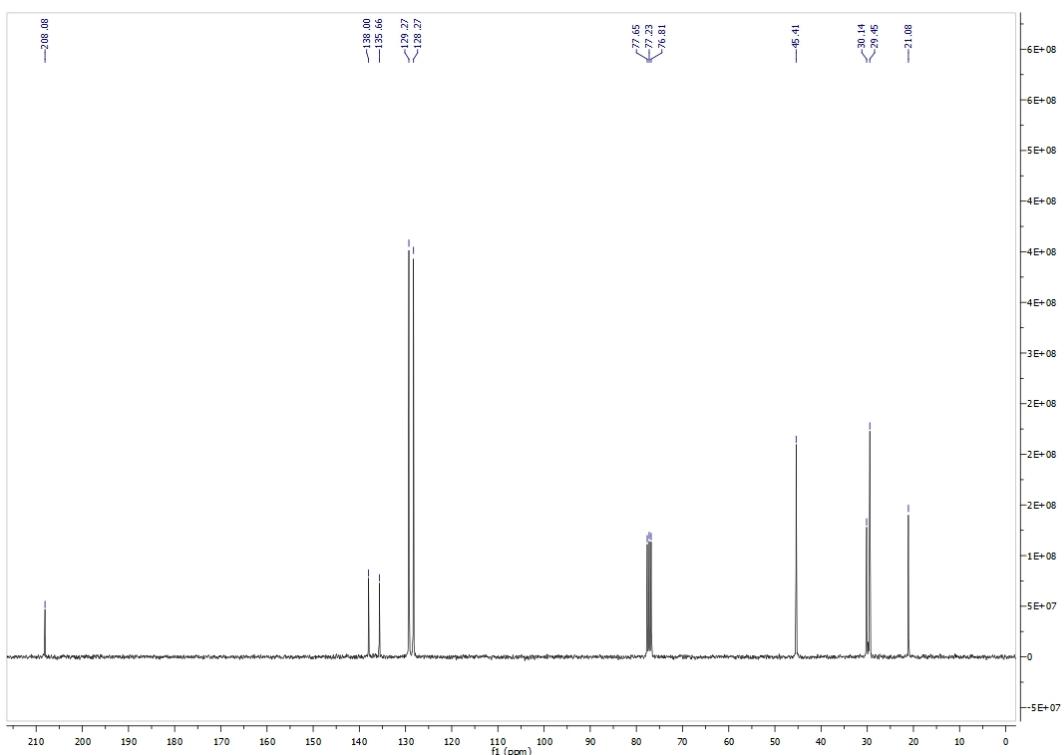
¹³C -NMR spectrum of **7j** (300 MHz, DMSO)

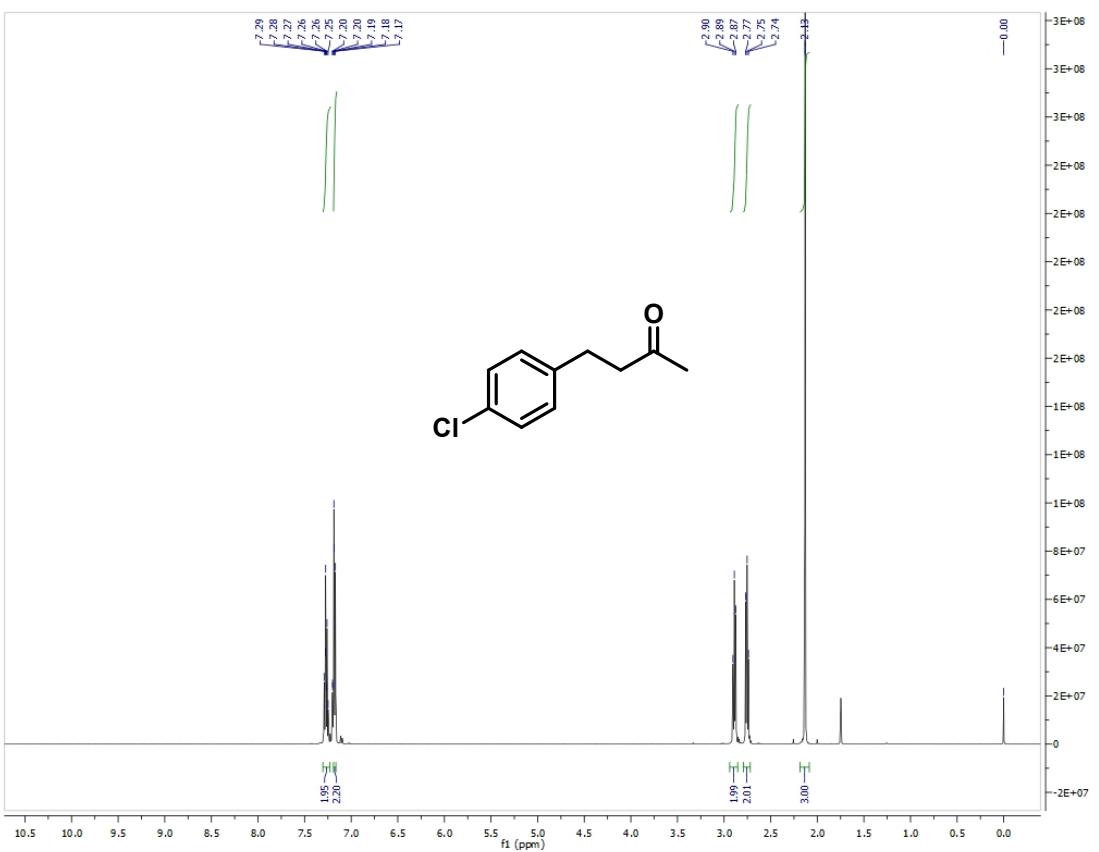
¹H-NMR spectrum of **7k** (300 MHz, DMSO)¹³C-NMR spectrum of **7k** (300 MHz, DMSO)

¹H-NMR spectrum of **2a** (300 MHz, CDCl₃)¹³C-NMR spectrum of **2a** (300 MHz, CDCl₃)

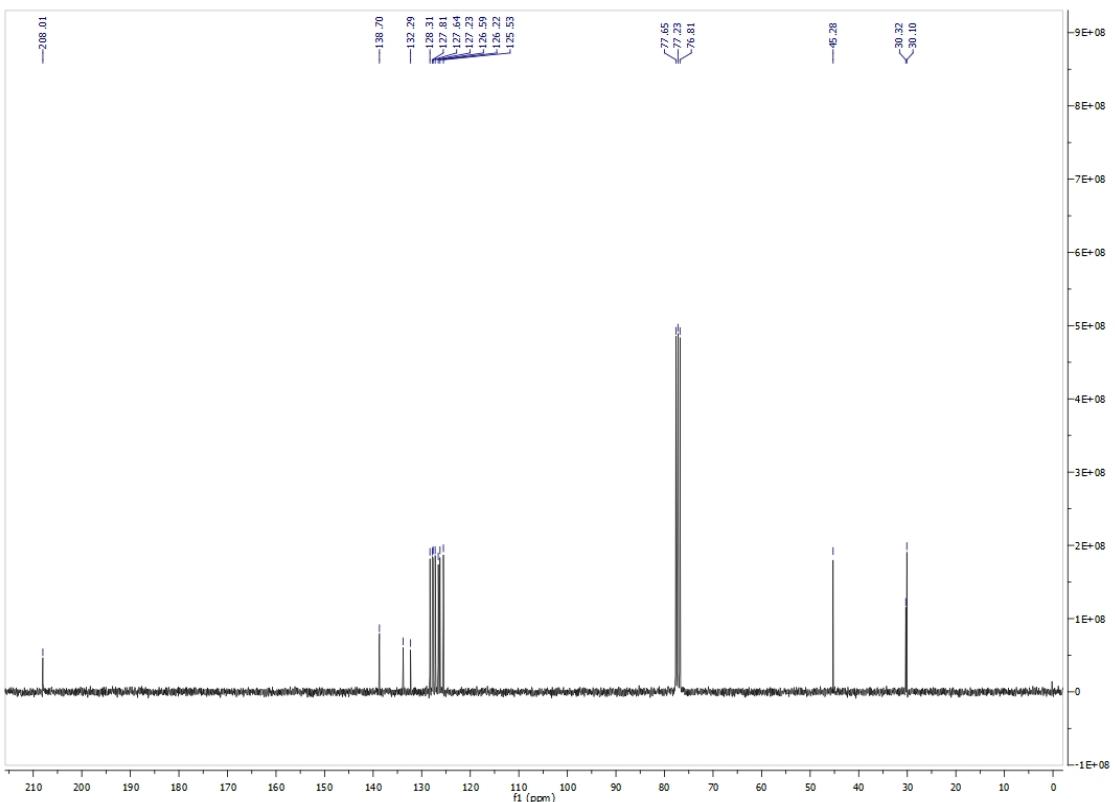
¹H-NMR spectrum of **5a** (300 MHz, CDCl₃)¹³C-NMR spectrum of **5a** (300 MHz, CDCl₃)

¹H-NMR spectrum of **5b** (300 MHz, CDCl₃)¹³C -NMR spectrum of **5b** (300 MHz, CDCl₃)

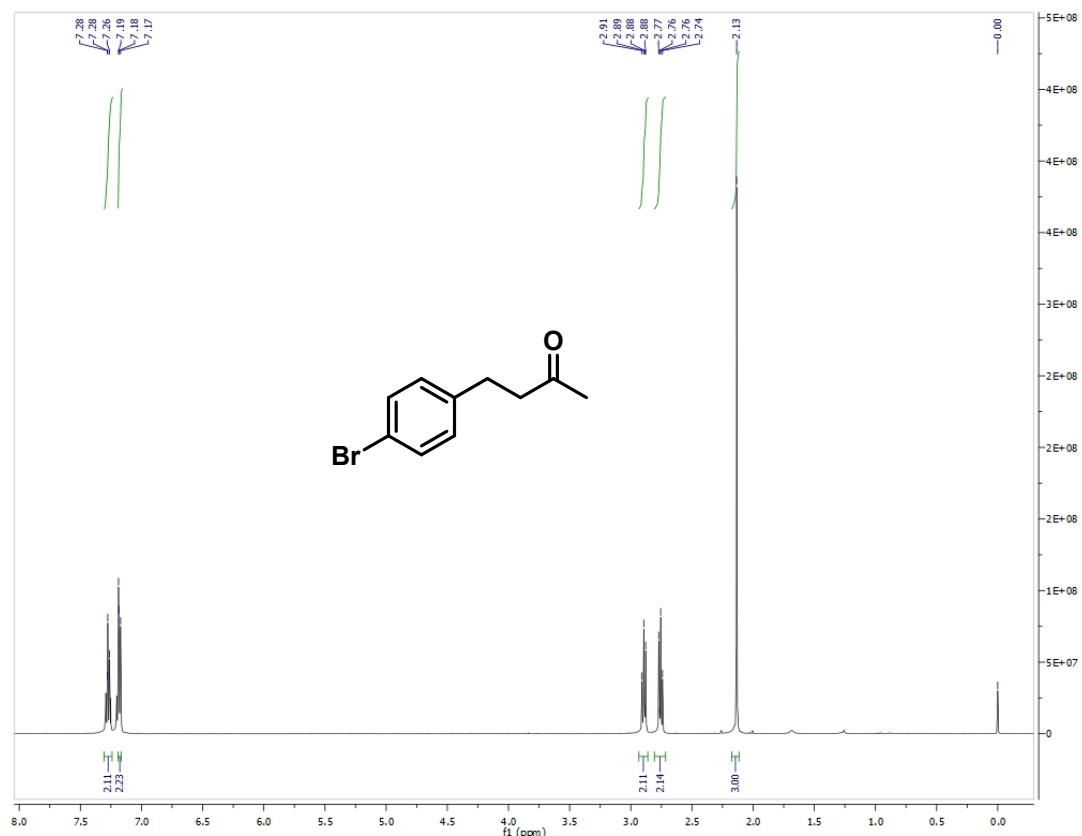
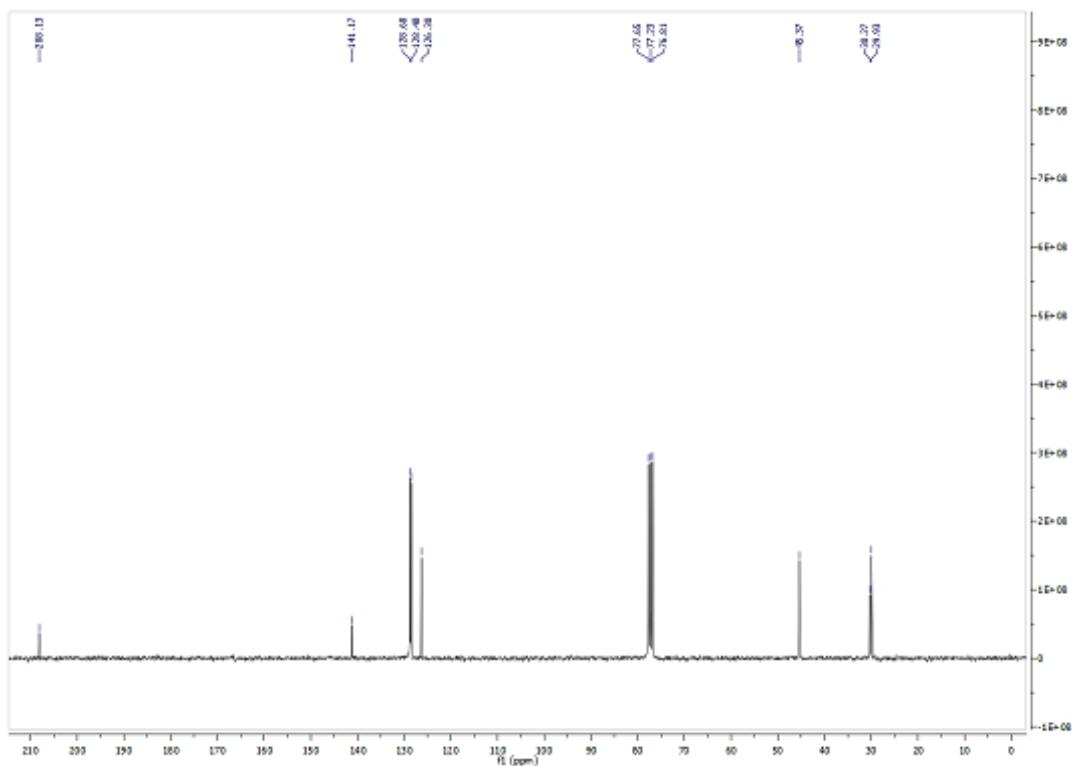
¹H-NMR spectrum of **5c** (300 MHz, CDCl₃)¹³C-NMR spectrum of **5c** (300 MHz, CDCl₃)

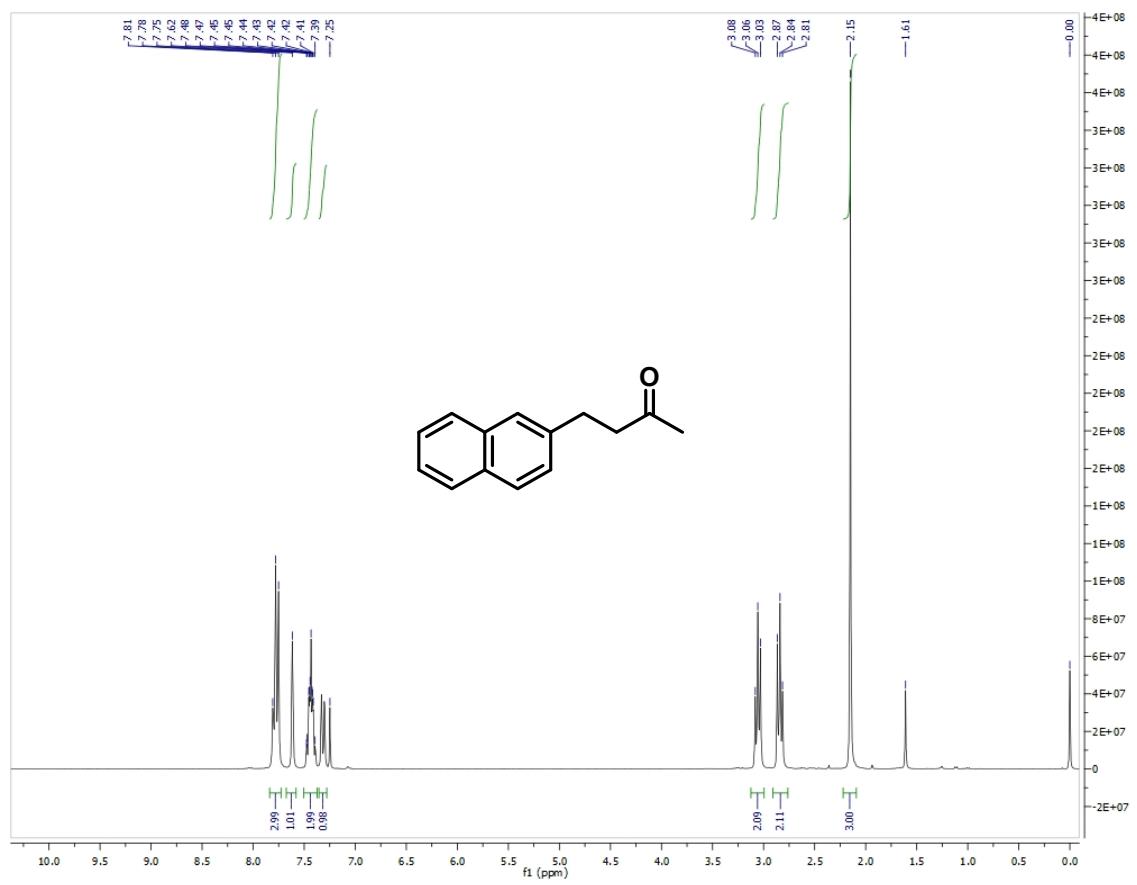
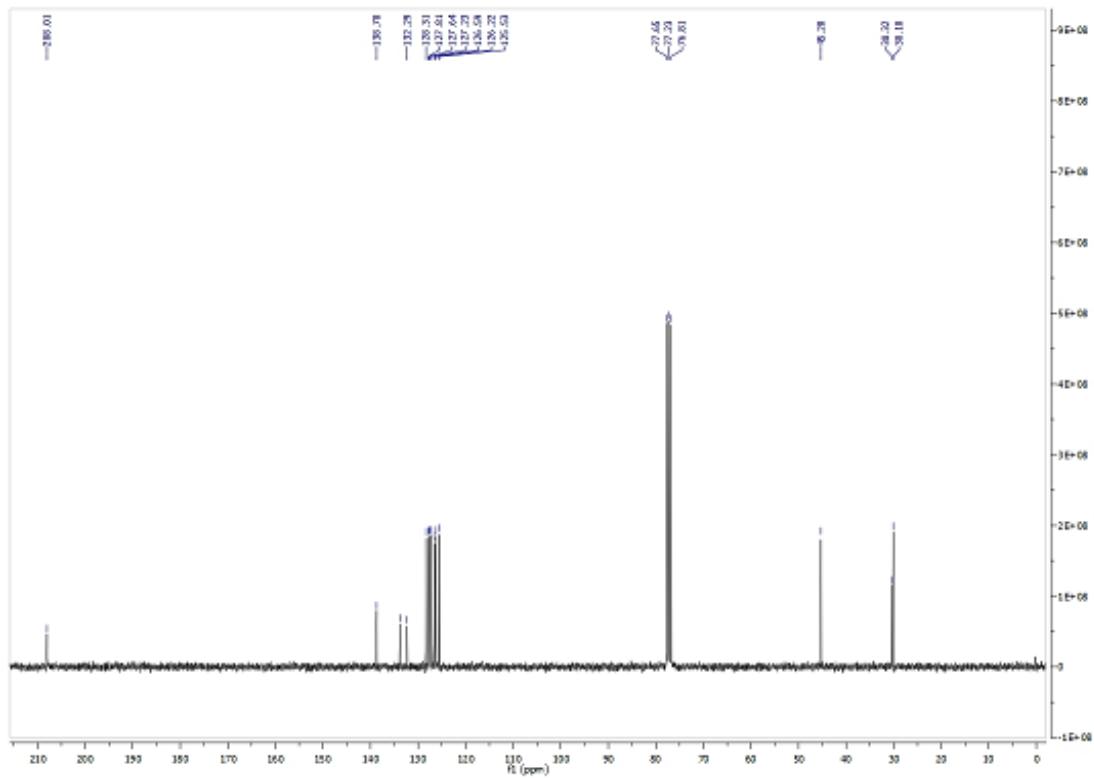


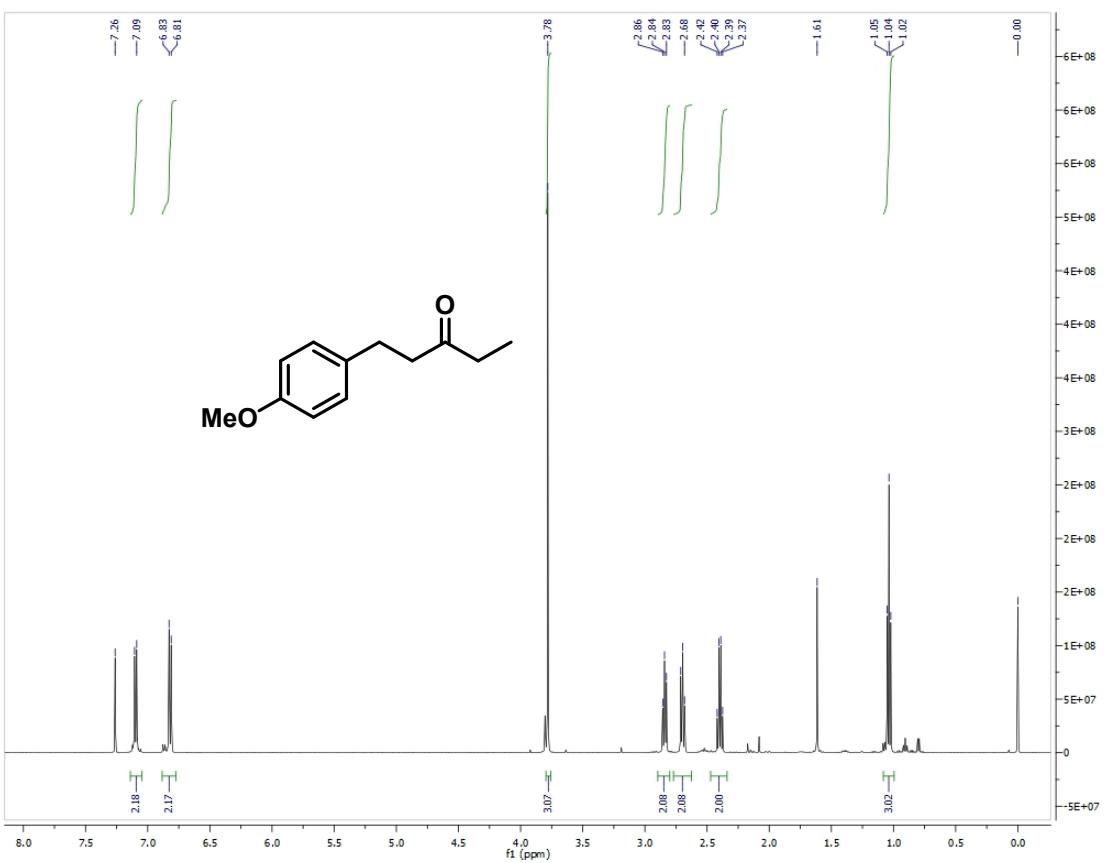
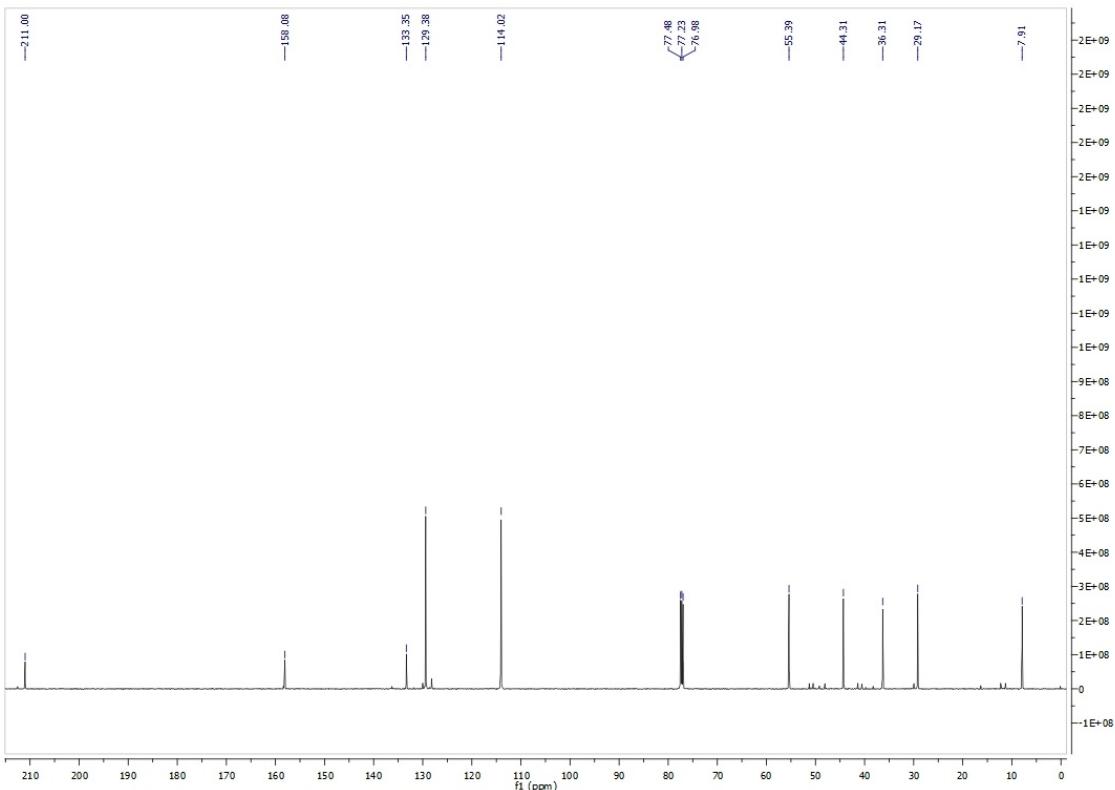
^1H -NMR spectrum of **5d** (300 MHz, CDCl_3)



^{13}C -NMR spectrum of **5d** (300 MHz, CDCl_3)

¹H-NMR spectrum of **5e** (300 MHz, CDCl₃)¹³C-NMR spectrum of **5e** (300 MHz, CDCl₃)

¹H-NMR spectrum of **5f** (300 MHz, CDCl₃)¹³C -NMR spectrum of **5f** (300 MHz, CDCl₃)

¹H-NMR spectrum of **5g** (300 MHz, CDCl₃)¹³C -NMR spectrum of **5g** (300 MHz, CDCl₃)

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