

# Supporting Information

## Hydrogen Bond Directed Aerobic Oxidation of Amines by Photoredox Catalysis

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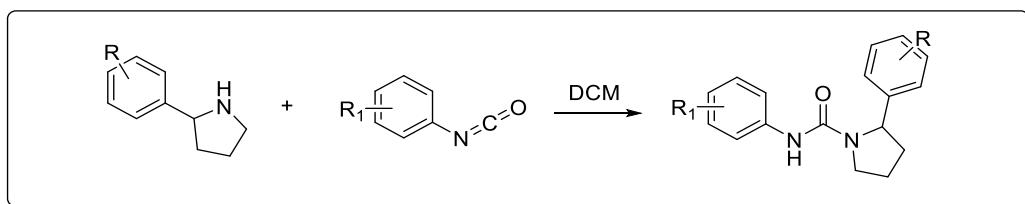
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## General information

The commercial materials and photocatalyst were purchased from Adamas-beta®, Shanghai Energy® and Tianjin Heowns®. Solvents for conjugate addition reactions were treated by the standard methods.<sup>1</sup> Reactions were powered by magnetic stirrers. Flash column chromatography was carried out on silica gel (300–400 mesh) using a forced flow of eluent. For TLC, silica gel plates were used and visualized by fluorescence quenching under UV light. All the NMR spectra were recorded on a Bruker NMR spectrometers. Chemical shifts ( $\delta$ ) for  $^1\text{H}$  NMR (400 Hz),  $^{13}\text{C}$  NMR (100 Hz) were given in ppm. Data were reported as follows: chemical shift, intergration, multiplicity (s = single, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet) and coupling constants (Hz). High resolution mass spectra (HRMS) were recorded on a Bruker Daltonics maXis UHR-TOF MS. Melting points were determined on a SGW X-4 microscope melting point apparatus and were uncorrected. X-ray crystallography analysis was performed on a Bruker X8 APEX X-ray diffraction meter. High resolution mass spectra (HRMS) were recorded on a Bruker Daltonics maXis UHR-TOF MS. Fluorescence spectra were obtained with FLS-920 Edinburgh Fluorescence Spectrometer and 1.0 cm quartz cells at the slits of 15 nm and without in situ excitation. The blue light source (465nm) was provided by WATTECS WP-TEC-1020 parallel reactor.

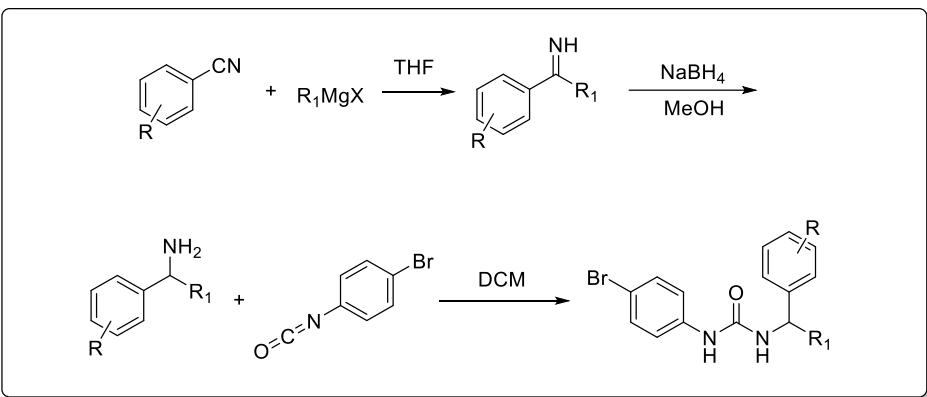
## The preparation of substrates

General procedures for the synthesis of **1 (GP1)**.



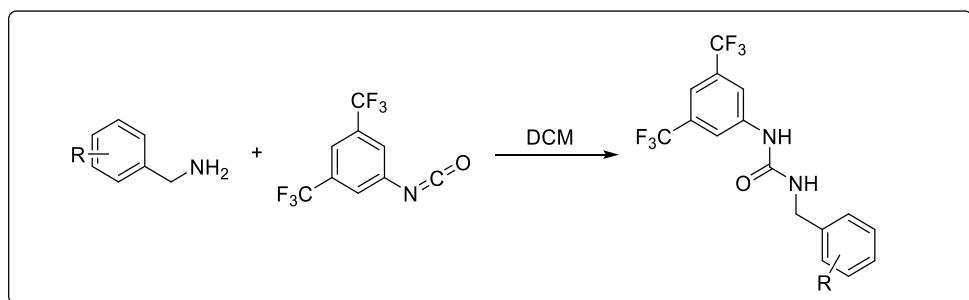
To a solution of pyrrolidine (1.1 mmol) in dichloromethane (15 mL), the isocyanate (1 mmol) was added into the mixture at 0 °C, then the mixture was stirred for 10 min at room temperature. After that, the solvent was evaporated to dryness under reduced pressure and purified via flash chromatography to obtained the corresponding compounds.

General procedures for the synthesis of **3 (Gp 2)**.



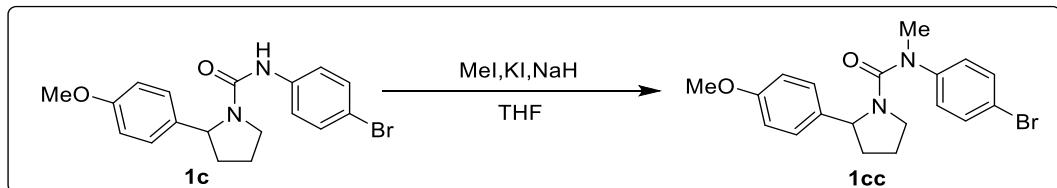
In an oven-dried round flask under Ar atmosphere, a mixture of the aryl nitrile (1 mmol) and the Grignard reagent (1.2 mmol) was stirred in dry THF (10 mL) at 70 °C for 2-8 h. The reaction mixture was cooled to room temperature and quenched by dry MeOH (10 mL) at 0 °C.<sup>2</sup> After being stirred vigorously for 10 min, the sodium borohydride was added slowly and the mixture was heated at 60 °C until the imine was disappeared determined by TLC. The volatile materials were evaporated under vacuum and purified by flash column chromatography. The amine was dissolved in DCM (15 mL) and the isocyanate was added at 0 °C, the reaction was stirred for 10 min at room temperature. The solvent was evaporated to dryness under reduced pressure and purified via flash chromatography to obtain the corresponding compounds.

General procedures for the synthesis of **5 (Gp 3)**.



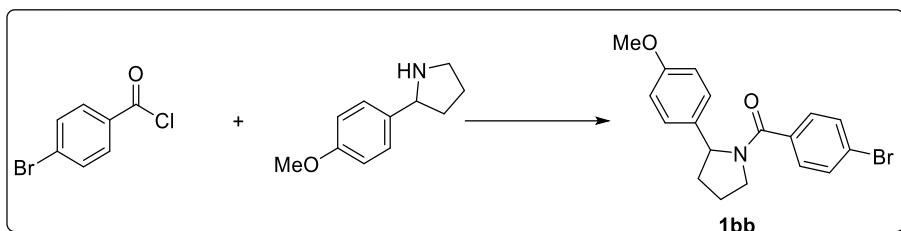
To a solution of benzylamine (1.1 mmol) in dichloromethane (15 mL), the 1-isocyanato-3,5-bis(trifluoromethyl)benzene (1 mmol) was added into the mixture at 0 °C, the reaction was stirred for 10 min at room temperature. After that, the solvent was evaporated to dryness under reduced pressure and purified via flash chromatography to obtain the compound.

The synthesis of N-(4-bromophenyl)-2-(4-methoxyphenyl)-N-methylpyrrolidine-1-carboxamide (**1cc**).



To a solution of urea (1 mmol) in tetrahydrofuran (15 mL), the sodium hydride (2.5 mmol) and potassium iodide (0.2 mmol) was added at 0 °C, then the iodomethane (1.8 mmol) and KI (0.1 mmol) was added and the reaction was stirred at room temperature until finished (determined by TLC analysis). The solvent was evaporated to dryness under reduced pressure, and purified via flash chromatography to obtain the compound. White solid; 83% yield, 322 mg; m.p. 183-185 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.45-7.43 (d, *J* = 8 Hz, 2H), 7.10-7.08 (d, *J* = 8 Hz, 2H), 6.91-6.85 (m, 4H), 4.71-4.68 (m, 1H), 3.80 (s, 3H), 3.08 (s, 3H), 3.01-2.94 (m, 1H), 2.23-2.19 (m, 1H), 1.84-1.64 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 159.47, 158.51, 145.24, 136.14, 132.31, 127.42, 126.63, 117.68, 113.76, 62.13, 55.29, 50.27, 39.00, 35.86, 25.09; HRMS (ESI) *m/z* calcd for C<sub>19</sub>H<sub>21</sub>BrN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 411.0679; found: 411.0672.

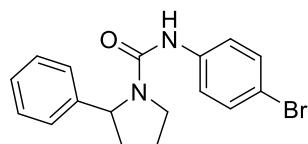
The synthesis of (2-(4-methoxyphenyl)pyrrolidin-1-yl)(phenyl)methanone (**1bb**).



To a solution of 4-bromobenzoyl chloride (1.1 mmol) in dichloromethane (15 mL), triethylamine (2.2 mmol) and pyrrolidine (1.0 mmol) were sequentially added into the mixture. The mixture was stirred at room temperature until the completion of the reaction determined by TLC analysis. The crude product was purified by flash chromatography on silica gel (eluent: PE/EtOAc from 5:1 to 2:1) affording to the corresponding compounds. White solid; 93% yield, 364 mg; m.p. 143-145 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.40-7.37 (t, *J* = 6 Hz, 2H), 7.32-7.31 (d, *J* = 4 Hz, 3H), 7.19-7.12 (m, 4H), 6.95-6.91 (d, *J* = 4 Hz, 3H), 6.08 (s, 1H), 4.88-4.85 (m, 1H), 3.83-3.71 (m, 2H), 2.46-2.39 (m, 1H), 2.01-1.89 (m, 3H); <sup>13</sup>C NMR (100 MHz, DMSO) δ 169.81, 168.78, 158.61, 158.51, 135.97, 135.84, 135.64, 135.10, 131.46, 131.08, 129.17, 128.38, 126.91, 126.67, 124.44, 123.76, 113.97, 65.89, 62.99, 60.64, 55.31, 51.04, 47.17, 35.91, 34.73, 25.29, 21.64, 15.32; HRMS (ESI) *m/z* calcd for C<sub>18</sub>H<sub>19</sub>NNaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 304.1308; found: 304.1319.

## Characterization data for the substrates.

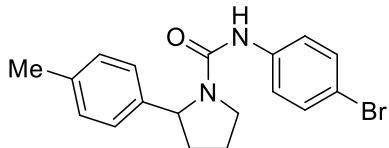
### N-(4-bromophenyl)-2-phenylpyrrolidine-1-carboxamide (**1a**)



The product was prepared according to the Gp **1**. White solid; 92% yield, 316 mg; m.p. 189-191 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.42-7.38 (t, *J* = 8 Hz, 2H), 7.34-7.25 (m, 5H), 7.03-7.01 (d, *J* = 8 Hz, 2H), 6.03 (s, 1H), 4.86-4.84 (m, 1H), 3.85-

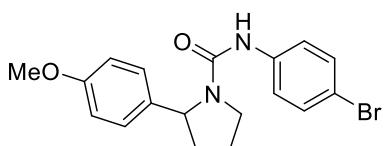
3.71 (m, 2H), 2.50-2.41 (m, 1H), 2.05-1.91 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  153.92, 142.42, 138.16, 131.59, 129.28, 128.06, 125.85, 120.74, 115.02, 61.39, 47.57, 36.95, 23.19; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 367.0416; found: 367.0417.

### N-(4-bromophenyl)-2-(p-tolyl)pyrrolidine-1-carboxamide (1b)



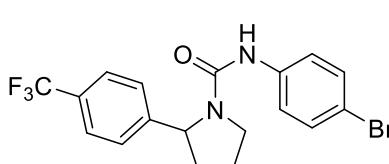
The product was prepared according to the **Gp 1**. White solid; 95% yield, 340 mg; m.p. 156-158 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28-7.24 (m, 2H), 7.19 (s, 4H), 7.04-7.02 (d,  $J = 8$  Hz, 2H), 6.10(s, 1H), 4.81-4.78 (m, 1H), 3.83-3.77 (m, 1H), 3.74-3.68 (m, 1H), 2.47-2.39 (m, 1H), 2.36 (s, 3H), 2.02-1.87 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  153.98, 139.37, 138.27, 137.84, 131.56, 129.95, 125.78, 120.72, 114.91, 61.18, 47.53, 37.06, 23.16, 21.14; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{19}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 381.0573; found: 381.0581.

### N-(4-bromophenyl)-2-(4-methoxyphenyl)pyrrolidine-1-carboxamide (1c)



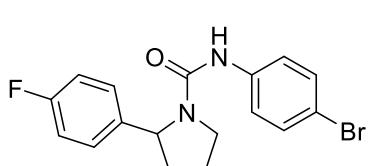
The product was prepared according to the **Gp 1**. White solid; 92% yield, 364 mg; m.p. 118-120 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.29-7.21 (m, 4H), 7.05-7.02 (d,  $J = 12$  Hz, 2H), 6.93-6.91 (d,  $J = 8$  Hz, 2H), 6.11 (1H, s), 4.80-4.77 (d,  $J = 4$  Hz, 1H), 3.81-3.68 (m, 4H), 2.46-2.38 (m, 1H), 2.04-1.86 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz, DMSO)  $\delta$  159.36, 154.01, 138.25, 134.23, 131.57, 127.05, 120.71, 114.91, 114.64, 60.88, 55.37, 47.49, 37.13, 23.14; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{19}\text{BrN}_2\text{NaO}_2^+[(\text{M}+\text{H})^+]$ : 397.0522; found: 397.0506.

### N-(4-bromophenyl)-2-(4-(trifluoromethyl)phenyl)pyrrolidine-1-carboxamide (1d)



The product was prepared according to the **Gp 1**. White solid; 96% yield, 396 mg; m.p. 173-175 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62-7.60 (d,  $J = 8$  Hz, 2H), 7.39-7.37 (d,  $J = 8$  Hz, 2H), 7.32-7.30 (d,  $J = 8$  Hz, 2H), 7.17-7.15 (d,  $J = 8$  Hz, 2H), 6.16(s, 1H), 5.06-5.03 (q,  $J = 4$  Hz, 1H), 3.77-3.67 (m, 2H), 2.48-2.39 (m, 1H), 2.03-1.96 (m, 2H), 1.94-1.87(m,1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  153.67, 147.08, 137.89, 131.70, 125.97, 125.94, 125.87, 125.38, 122.68, 121.04, 115.45, 60.88, 47.35, 35.79, 23.43;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.45; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{16}\text{BrF}_3\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 435.0290; found: 435.0267.

### N-(4-bromophenyl)-2-(4-fluorophenyl)pyrrolidine-1-carboxamide (1e)



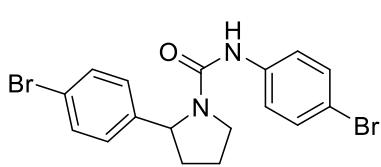
The product was prepared according to the **Gp 1**. White solid;94% yield, 340 mg; m.p. 167-169 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30-7.24 (m, 4H), 7.10-7.05 (m, 4H), 6.08(s, 1H), 4.90-4.88 (m, 1H), 3.79-3.68 (m, 2H), 2.46-2.38 (m, 1H), 2.02-1.86 (m, 3H), 2.36 (s, 3H), 2.02-1.87 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  163.46, 161.01, 153.81, 138.36, 138.04, 131.65, 127.41, 127.33, 120.85, 116.15, 115.94, 115.20, 60.66, 47.44, 36.61, 23.20;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -114.27; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{16}\text{BrFN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 385.0322; found: 385.0329.

**N-(4-bromophenyl)-2-(4-chlorophenyl)pyrrolidine-1-carboxamide (1f)**



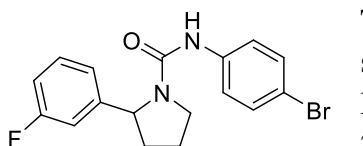
The product was prepared according to the **Gp 1**. White solid; 92% yield, 348 mg; m.p. 175-177 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35-7.28 (m, 4H), 7.22-7.20 (d, *J* = 8 Hz, 2H), 7.12-7.10 (d, *J* = 8 Hz, 2H), 6.11 (s, 1H), 4.91-4.88 (m, 1H), 3.76-3.67 (m, 2H), 2.45-2.36 (m, 1H), 2.03-1.84 (m, 3H); <sup>13</sup>C NMR (100 MHz, DMSO) δ 153.75, 141.26, 138.00, 133.47, 131.66, 129.22, 127.09, 120.93, 115.28, 60.68, 47.41, 36.32, 23.26; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrClN<sub>2</sub>O<sup>+</sup> [(M+H)<sup>+</sup>]: 379.0207; found: 379.0209.

**N,2-bis(4-bromophenyl)pyrrolidine-1-carboxamide (1g)**



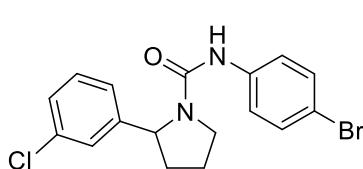
The product was prepared according to the **Gp 1**. White solid; 89% yield, 376 mg; m.p. 176-178 °C; <sup>1</sup>H NMR (400 MHz, (CD<sub>3</sub>)<sub>2</sub>CO) δ 7.69 (s, 1H), 7.48-7.45 (m, 4H), 7.34-7.31 (d, *J* = 12 Hz, 2H), 7.24-7.22 (d, *J* = 8 Hz, 2H), 5.14-5.11 (q, *J* = 4 Hz, 1H), 3.83-3.78 (m, 1H), 3.69-3.63 (m, 1H), 2.42-2.33 (m, 1H), 1.99-1.91 (m, 2H), 1.86-1.80 (m, 1H); <sup>13</sup>C NMR (100 MHz, (CD<sub>3</sub>)<sub>2</sub>CO) δ 153.52, 144.01, 139.99, 131.16, 131.11, 127.87, 120.92, 119.72, 113.49, 60.15, 46.89, 34.82, 23.30; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>Br<sub>2</sub>N<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 444.9522; found: 444.9534.

**N-(4-bromophenyl)-2-(3-fluorophenyl)pyrrolidine-1-carboxamide (1h)**



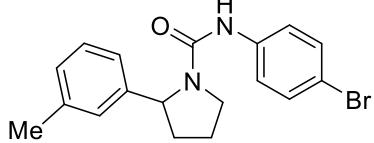
The product was prepared according to the **Gp 1**. White solid; 92% yield, 333 mg; m.p. 173-175 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.37-7.28 (m, 3H), 7.13-7.11 (d, *J* = 8 Hz, 2H), 7.08-7.06 (d, *J* = 8 Hz, 1H), 7.01-6.97 (m, 2H), 6.11 (s, 1H), 4.94-4.91 (q, *J* = 4Hz, 1H), 3.75-3.71 (m, 2H), 2.47-2.38(m, 1H), 2.02-1.88(m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.50, 162.04, 153.75, 145.59, 138.00, 131.66, 130.74, 121.32, 120.94, 115.29, 114.83, 114.62, 112.83, 112.61, 60.82, 47.41, 36.21, 23.28; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -111.67; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>BrFN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 385.0322; found: 385.0329.

**N-(4-bromophenyl)-2-(3-chlorophenyl)pyrrolidine-1-carboxamide (1i)**



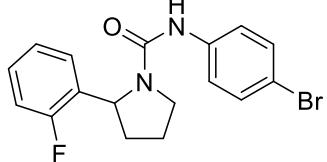
The product was prepared according to the **Gp 1**. White solid; 95% yield, 356 mg; m.p. 192-194 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.38 (s, 1H), 7.47-7.45 (d, *J* = 8 Hz, 2H), 7.38-7.36 (d, *J* = 8 Hz, 2H), 7.34-7.32 (d, *J* = 8 Hz, 1H), 7.27-7.24 (d, *J* = 12 Hz, 2H), 7.19-7.17 (d, *J* = 8 Hz, 1H), 5.08-5.05 (m, *J* = 4 Hz, 1H), 3.59-3.53 (q, *J* = 8 Hz, 1H), 3.34-3.25 (m, 1H), 1.93-1.81 (m, 2H), 1.77-1.71 (m, 1H); <sup>13</sup>C NMR (100 MHz, DMSO) δ 154.02, 147.71, 140.27, 133.37, 131.45, 130.54, 126.83, 125.87, 124.71, 121.69, 113.59, 60.25, 47.44, 34.83, 23.77; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrClN<sub>2</sub>O<sup>+</sup> [(M+H)<sup>+</sup>]: 379.0207; found: 379.0209.

**N-(4-bromophenyl)-2-(m-tolyl)pyrrolidine-1-carboxamide (1j)**



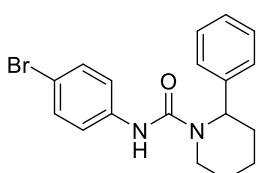
The product was prepared according to the **Gp 1**. White solid; 96% yield, 344 mg; m.p. 195-197 °C; <sup>1</sup>H NMR (400 MHz, (CD<sub>3</sub>)<sub>2</sub>CO) δ 7.82(s, 1H), 7.01-6.99 (d, *J* = 8 Hz, 2H), 6.92-6.90 (d, *J* = 8 Hz, 2H), 6.75-6.72 (t, *J* = 6 Hz, 1H), 6.57-6.52 (m, 3H), 4.62-4.60 (m, 1H), 3.33-3.28 (m, 1H), 3.13-3.07 (q, *J* = 8 Hz, 1H), 1.87-1.77 (m, 4H), 1.46-1.39 (m, 2H), 1.31-1.25 (m, 1H); <sup>13</sup>C NMR (100 MHz, (CD<sub>3</sub>)<sub>2</sub>CO) δ 153.50, 144.48, 139.95, 137.16, 130.97, 128.06, 127.07, 126.12, 122.55, 121.13, 112.97, 59.98, 46.93, 34.64, 23.14, 21.16; HRMS (ESI) *m/z* calcd for C<sub>18</sub>H<sub>19</sub>BrN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 381.0573; found: 381.0581.

#### N-(4-bromophenyl)-2-(2-fluorophenyl)pyrrolidine-1-carboxamide (1k)



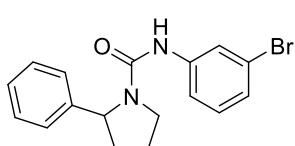
The product was prepared according to the **Gp 1**. White solid; 95% yield, 344 mg; m.p. 177-179 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ 8.41 (s, 1H), 7.48-7.46 (d, *J* = 8 Hz, 2H), 7.38-7.36 (d, *J* = 8 Hz, 2H), 7.29-7.24 (m, 1H), 7.18-7.13 (m, 3H), 5.28-5.26 (m, 1H), 3.81-3.76 (m, 1H), 3.59-3.53 (q, *J* = 8 Hz, 1H), 2.35-2.26 (m, 1H), 1.98-1.72 (m, 3H); <sup>13</sup>C NMR (100 MHz, DMSO) δ 160.92, 158.50, 153.84, 140.30, 131.42, 128.84, 128.75, 127.37, 124.57, 121.78, 115.76, 115.55, 113.60, 55.29, 47.16, 33.41, 23.74; <sup>19</sup>F NMR (376 MHz, DMSO) δ -118.62; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>BrFN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 385.0322; found: 385.0329.

#### N-(4-bromophenyl)-2-phenylpiperidine-1-carboxamide (1l)



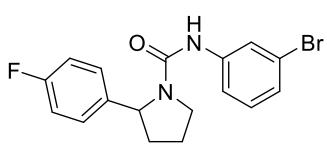
The product was prepared according to the **Gp 1**. White solid; 93% yield, 333 mg; m.p. 187-189 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ 8.72 (s, 1H), 7.50-7.48 (d, *J* = 8 Hz, 2H), 7.41-7.36 (m, 4H), 7.26-7.22 (m, 3H), 5.54 (s, 1H), 4.08-4.04 (m, 1H), 2.83-2.76 (t, *J* = 14 Hz, 1H), 2.40-2.37 (d, *J* = 12 Hz, 1H), 1.87-1.79 (m, 1H), 1.61-1.23 (m, 4H); <sup>13</sup>C NMR (100 MHz, DMSO) δ 155.67, 140.69, 140.59, 131.45, 129.07, 126.87, 126.79, 122.00, 113.56, 52.85, 40.67, 28.26, 25.71, 19.65; HRMS (ESI) *m/z* calcd for C<sub>18</sub>H<sub>19</sub>BrN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 381.0573; found: 381.0575.

#### N-(3-bromophenyl)-2-phenylpyrrolidine-1-carboxamide (1m)



The product was prepared according to the **Gp 1**. White solid; 87% yield, 300 mg; m.p. 177-179 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.44-7.38 (m, 3H), 7.34-7.29 (m, 3H), 7.07-7.00 (m, 3H), 6.10 (s, 1H), 4.86-4.85 (m, 1H), 3.83-3.71 (m, 2H), 2.48-2.40 (m, 1H), 2.02-1.91 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 153.79, 142.39, 140.38, 129.96, 129.28, 128.06, 125.81, 125.56, 122.40, 122.01, 117.61, 61.37, 47.56, 36.82, 23.19; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 367.0416; found: 367.0418.

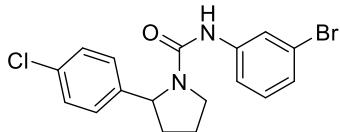
#### N-(3-bromophenyl)-2-(4-fluorophenyl)pyrrolidine-1-carboxamide (1n)



The product was prepared according to the **Gp 1**. White solid; 89% yield, 336 mg; m.p. 173-175 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.50 (s, 1H), 7.28-7.25 (m, 2H), 7.10-7.02 (m, 5H), 6.10 (s, 1H), 4.91-4.89 (m, 1H), 3.79-3.69 (m, 2H),

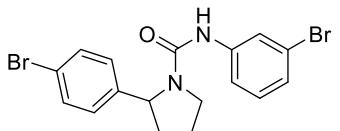
2.47-2.38 (m, 1H), 2.05-1.86 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  163.46, 161.01, 153.68, 140.23, 138.28, 130.02, 127.38, 127.30, 125.74, 122.46, 122.08, 117.66, 116.18, 115.97, 60.67, 47.44, 36.60, 23.20;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -114.37; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{16}\text{BrFN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 401.0271; found: 401.0176.

**N-(4-bromophenyl)-2-(4-chlorophenyl)pyrrolidine-1-carboxamide (1o)**



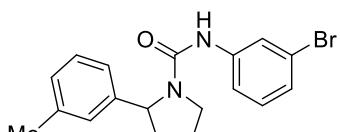
The product was prepared according to the **Gp 1**. White solid; 92% yield, 348 mg; m.p. 169-171 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (s, 1H), 7.36-7.33 (d,  $J = 12$  Hz, 2H), 7.23-7.21 (d,  $J = 8$  Hz, 2H), 7.11-7.05 (m, 3H), 6.13 (m, 1H), 4.92-4.91 (m, 1H), 3.74-3.71 (t,  $J = 6$  Hz, 2H), 2.46-2.37 (m, 1H), 2.01-1.85 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  153.65, 141.18, 140.15, 133.16, 130.04, 129.25, 127.05, 125.84, 122.48, 122.20, 117.78, 60.70, 47.42, 36.25, 23.26; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{BrClN}_2\text{O}^+[(\text{M}+\text{H})^+]$ : 379.0207; found: 379.0218.

**N-(3-bromophenyl)-2-(4-bromophenyl)pyrrolidine-1-carboxamide (1p)**



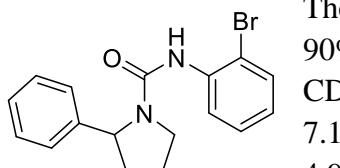
The product was prepared according to the **Gp 1**. White solid; 90% yield, 380 mg; m.p. 197-199 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53-7.48 (m, 3H), 7.17-7.15 (d,  $J = 8$  Hz, 2H), 7.11-7.03 (m, 3H), 6.13 (s, 1H), 4.91-4.88 (m, 1H), 3.73-3.70 (t,  $J = 12$  Hz, 2H), 2.46-2.37 (m, 1H), 2.01-1.84 (m, 3H);  $^{13}\text{C}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  153.61, 141.76, 140.17, 132.17, 130.04, 127.39, 125.82, 122.48, 122.17, 121.54, 117.75, 60.74, 47.41, 36.19, 23.27; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{16}\text{Br}_2\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 444.9522; found: 444.9526.

**N-(3-bromophenyl)-2-(m-tolyl)pyrrolidine-1-carboxamide (1q)**



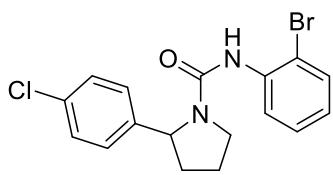
The product was prepared according to the **Gp 1**. White solid; 90% yield, 322 mg; m.p. 176-178 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.43 (s, 1H), 7.31-7.27 (d,  $J = 8$  Hz, 1H), 7.15-7.09 (m, 3H), 7.07-7.01 (m, 3H), 6.09 (s, 1H), 4.80-4.79 (m, 1H), 3.83-3.71 (m, 2H), 2.47-2.42 (m, 1H), 2.37 (s, 3H), 2.05-1.90 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  153.85, 142.32, 140.43, 139.14, 129.95, 129.17, 128.92, 126.44, 125.51, 122.91, 122.40, 121.97, 117.55, 61.39, 47.59, 36.99, 23.19, 21.58; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{19}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 381.0573; found: 381.0569.

**N-(2-bromophenyl)-2-phenylpyrrolidine-1-carboxamide (1r)**



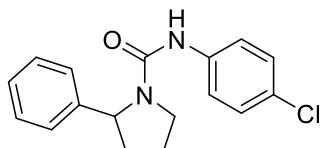
The product was prepared according to the **Gp 1**. White solid; 90% yield, 310 mg; m.p. 187-189 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.22-8.20 (d,  $J = 8$  Hz, 1H), 7.37-7.24 (m, 6H), 7.21-7.17 (t,  $J = 8$  Hz, 1H), 6.79-6.75 (t,  $J = 8$  Hz, 1H), 6.71 (s, 1H), 4.95-4.93 (m, 1H), 3.81-3.77 (m, 2H), 2.46-2.38 (m, 1H), 2.04-1.87 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  153.67, 142.35, 137.19, 131.90, 129.08, 128.11, 127.83, 125.98, 123.25, 120.83, 112.49, 61.29, 47.62, 36.75, 22.94; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 367.0416; found: 367.0420.

**N-(2-bromophenyl)-2-(4-chlorophenyl)pyrrolidine-1-carboxamide (1s)**



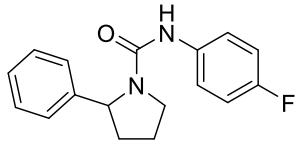
The product was prepared according to the **Gp 1**. White solid; 90% yield, 340 mg; m.p. 188-190 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.22-8.20 (d, *J* = 8 Hz, 1H), 7.40-7.38 (d, *J* = 8 Hz, 1H), 7.35-7.33 (d, *J* = 8 Hz, 2H), 7.26-7.20 (m, 3H), 6.84-6.80 (t, *J* = 8 Hz, 1H), 6.72 (s, 1H), 4.99-4.97 (m, 1H), 3.81-3.77 (t, *J* = 8 Hz, 2H), 2.49-2.40 (m, 1H), 2.05-1.86 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 153.50, 141.08, 136.91, 133.41, 131.90, 129.13, 128.22, 127.28, 123.42, 120.76, 112.55, 60.72, 47.47, 36.41, 23.05; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrClN<sub>2</sub>O<sup>+</sup> [(M+H)<sup>+</sup>]: 379.0207; found: 379.0243.

### N-(4-chlorophenyl)-2-phenylpyrrolidine-1-carboxamide (1t)



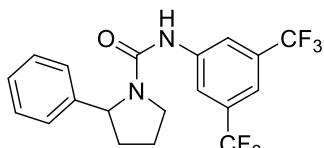
The product was prepared according to the **Gp 1**. White solid; 89% yield, 267 mg; m.p. 153-155 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41-7.37 (t, *J* = 8 Hz, 2H), 7.34-7.29 (m, 2H), 7.14-7.06 (m, 1H), 6.09 (s, 1H), 4.86-4.85 (d, *J* = 4 Hz, 1H), 3.83-3.70 (m, 2H), 2.50-2.40 (m, 1H), 2.04-1.90 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 154.00, 142.46, 137.66, 129.25, 128.66, 128.03, 127.49, 125.84, 120.46, 61.34, 47.56, 36.89, 23.19; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 323.0922; found: 323.0921.

### N-(4-fluorophenyl)-2-phenylpyrrolidine-1-carboxamide (1u)



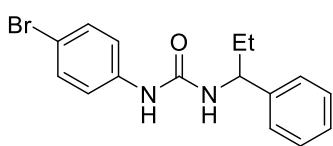
The product was prepared according to the **Gp 1**. White solid; 82% yield, 233 mg; m.p. 172-174 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.18 (s, 1H), 7.46-7.43 (t, *J* = 8 Hz, 2H), 7.32-7.29 (t, *J* = 12 Hz, 2H), 7.21-7.18 (m, 3H), 7.05-7.00 (t, *J* = 10 Hz, 2H), 5.10-5.08 (m, 1H), 3.77-3.74 (m, 1H), 3.58-3.52 (m, 1H), 2.35-2.24 (m, 1H), 1.92-1.68 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 158.88, 156.51, 154.24, 145.01, 137.23, 128.59, 126.81, 125.94, 121.58, 121.50, 115.23, 115.01, 60.41, 47.30, 35.03, 23.57; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -121.90; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>FN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 307.1217; found: 307.1227.

### N-(3,5-bis(trifluoromethyl)phenyl)-2-phenylpyrrolidine-1-carboxamide (1v)



The product was prepared according to the **Gp 1**. White solid; 90% yield, 360 mg; m.p. 165-167 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.96 (s, 1H), 8.27 (s, 2H), 7.55 (s, 1H), 7.34-7.30 (t, *J* = 8 Hz, 2H), 7.23-7.19 (m, 3H), 5.14-5.13 (m, 1H), 3.85-3.80 (m, 1H), 3.66-3.60 (m, 1H), 2.37-2.27 (m, 1H), 1.96-1.88 (m, 2H), 1.81-1.76 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 153.69, 144.52, 143.09, 131.24, 130.92, 130.59, 130.27, 128.63, 126.89, 125.86, 125.22, 122.51, 118.99, 114.24, 60.75, 47.42, 34.95, 23.55; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -61.78; HRMS (ESI) *m/z* calcd for C<sub>19</sub>H<sub>16</sub>F<sub>6</sub>N<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 425.1059; found: 425.1089.

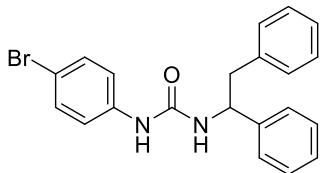
### 1-(4-bromophenyl)-3-(1-phenylpropyl)urea (3a)



The product was prepared according to the **Gp 2**. White solid; 95% yield; m.p. 171-173 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ 8.54 (s, 1H), 7.38-7.21 (m, 9H), 6.72-6.70 (d, *J* = 8 Hz, 1H), 4.62-4.57 (q, *J* = 8 Hz, 1H), 1.74-1.67 (m,

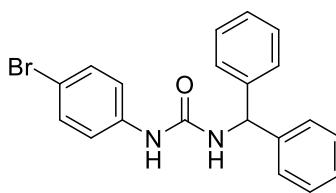
2H), 0.86-0.82 (t,  $J$  = 8 Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  154.85, 144.36, 140.26, 131.82, 128.72, 127.13, 126.75, 119.84, 112.72, 55.02, 30.05, 11.17; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{17}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 355.0416; found: 355.0443.

### **1-(4-bromophenyl)-3-(1,2-diphenylethyl)urea (3b)**



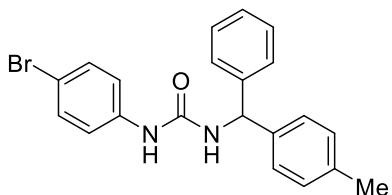
The product was prepared according to the **Gp 2**. White solid; 73% yield; m.p. 178-180 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.57 (s, 1H), 7.36-7.15 (m, 14H), 6.75-6.73 (d,  $J$  = 8 Hz, 1H), 5.01-4.95 (q,  $J$  = 8 Hz, 1H), 3.02-3.00 (d,  $J$  = 8 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  154.65, 143.99, 140.15, 138.84, 131.80, 129.68, 128.68, 128.55, 127.26, 126.90, 126.63, 119.88, 112.79, 55.13, 43.08; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{19}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 417.0573; found: 417.0579.

### **1-benzhydryl-3-(4-bromophenyl)urea (3c)**



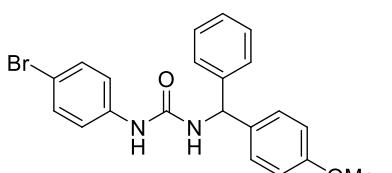
The product was prepared according to the **Gp 2**. White solid; 89% yield; m.p. 250-252 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.63 (s, 1H), 7.41-7.30 (m, 12H), 7.27-7.22 (m, 3H), 5.98-5.96 (d,  $J$  = 8 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  154.58, 143.50, 140.07, 131.89, 128.97, 127.44, 127.39, 119.94, 112.99, 57.32; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{20}\text{H}_{17}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 403.0416; found: 403.0419.

### **1-(4-bromophenyl)-3-(phenyl(p-tolyl)methyl)urea (3d)**



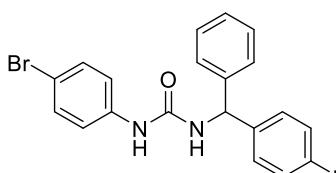
The product was prepared according to the **Gp 2**. White solid; 84% yield; m.p. 233-235 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.62 (s, 1H), 7.40-7.14 (m, 14H), 5.93-5.91 (d,  $J$  = 8 Hz, 1H), 2.26 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO)  $\delta$  154.56, 143.72, 140.53, 140.09, 136.56, 131.89, 129.49, 128.91, 127.35, 127.31, 119.89, 112.94, 57.02, 21.09; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{19}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 417.0573; found: 417.0582.

### **1-(4-bromophenyl)-3-((4-methoxyphenyl)(phenyl)methyl)urea (3e)**



The product was prepared according to the **Gp 2**. White solid; 78% yield, 320 mg; m.p. 177-179 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.61 (s, 1H), 7.45-7.44 (d,  $J$  = 4 Hz, 1H), 7.38-7.20 (m, 10H), 7.17-7.15 (d,  $J$  = 8 Hz, 1H), 6.92-6.90 (d,  $J$  = 8 Hz, 2H), 5.91-5.89 (d,  $J$  = 8 Hz, 1H), 3.72 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  158.65, 154.53, 143.88, 140.10, 135.51, 131.88, 128.89, 128.59, 127.24, 120.67, 119.88, 114.31, 112.91, 56.67, 55.53; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{19}\text{BrN}_2\text{NaO}_2^+[(\text{M}+\text{Na})^+]$ : 433.0522; found: 433.0527.

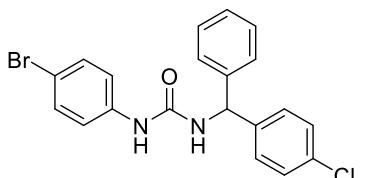
### **1-(4-bromophenyl)-3-((4-fluorophenyl)(phenyl)methyl)urea (3f)**



The product was prepared according to the **Gp 2**. White solid; 83% yield; m.p. 219-221 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.63 (s, 1H), 7.40-7.31 (m, 10H), 7.28-7.23 (m, 2H), 7.20-7.16 (t,  $J$  = 8 Hz, 2H), 6.00-5.98 (d,  $J$  = 8 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  162.79,

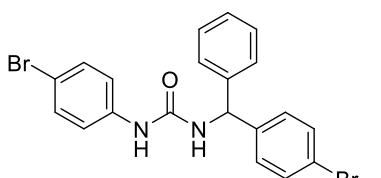
160.37, 154.56, 143.29, 140.04, 139.76, 131.89, 129.37, 129.28, 129.02, 127.54, 127.39, 119.97, 115.78, 115.57, 113.03, 56.64;  $^{19}\text{F}$  NMR (376 MHz, DMSO)  $\delta$  -61.78; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{20}\text{H}_{16}\text{BrFN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 421.0322; found: 421.0332.

**1-(4-bromophenyl)-3-((4-chlorophenyl)(phenyl)methyl)urea (3g)**



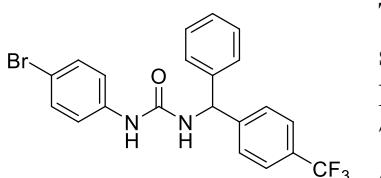
The product was prepared according to the **Gp 2**. White solid; 87% yield; m.p. 241-243 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.63 (s, 1H), 7.43-7.24 (m, 14H), 5.99-5.97 (d,  $J = 8$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  154.56, 142.94, 142.62, 140.00, 131.99, 131.89, 129.20, 129.08, 128.90, 127.66, 127.49, 119.98, 113.06, 56.73; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{20}\text{H}_{16}\text{BrClN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 437.0027; found: 437.0038.

**1-(4-bromophenyl)-3-((4-bromophenyl)(phenyl)methyl)urea (3h)**



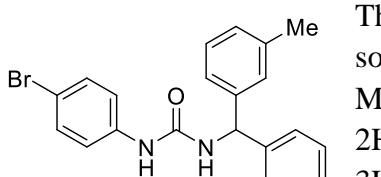
The product was prepared according to the **Gp 2**. White solid; 78% yield, 356 mg; m.p. 255-257 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.63 (s, 1H), 7.56-7.54 (d,  $J = 8$  Hz, 2H), 7.36-7.24 (m, 12H), 5.97-5.95 (d,  $J = 8$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  154.56, 143.06, 142.87, 139.99, 131.89, 131.81, 129.56, 129.08, 127.67, 127.50, 120.50, 119.98, 113.06, 56.80; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{20}\text{H}_{16}\text{Br}_2\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 480.9522; found: 480.9507.

**1-(4-bromophenyl)-3-(phenyl(4-(trifluoromethyl)phenyl)methyl)urea (3i)**



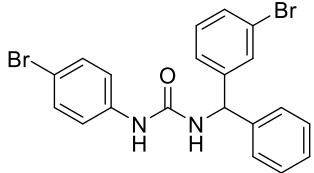
The product was prepared according to the **Gp 2**. White solid; 81% yield; m.p. 217-219°C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.67 (s, 1H), 7.74-7.72 (d,  $J = 8$  Hz, 2H), 7.57-7.55 (d,  $J = 8$  Hz, 2H), 7.41-7.27 (m, 10H), 6.07-6.05 (d,  $J = 8$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  154.60, 148.40, 142.46, 139.95, 131.90, 129.19, 128.77-120.67 (q,  $J = 270$  Hz), 128.17, 128.02, 127.85, 127.66, 125.87, 120.67, 119.99, 113.09, 57.12;  $^{19}\text{F}$  NMR (376 MHz, DMSO)  $\delta$  -60.85; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{16}\text{BrF}_3\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 471.0290; found: 471.0297.

**1-(4-bromophenyl)-3-(phenyl(m-tolyl)methyl)urea (3j)**



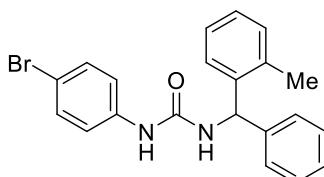
The product was prepared according to the **Gp 2**. White solid; 87% yield, 341 mg; m.p. 215-217 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.67 (s, 1H), 7.48-7.46 (d,  $J = 8$  Hz, 2H), 7.45-7.34 (m, 8H), 7.31-7.24 (m, 3H), 7.17-7.10 (m, 3H), 5.97-5.95 (m, 1H), 2.33 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  154.54, 143.61, 143.45, 140.09, 138.08, 131.89, 128.94, 128.89, 128.10, 127.93, 127.38, 127.32, 124.50, 119.89, 112.94, 57.27, 21.57; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{19}\text{BrN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 417.0573; found: 417.0558.

**1-(4-bromophenyl)-3-((3-bromophenyl)(phenyl)methyl)urea (3k)**



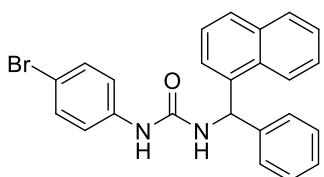
The product was prepared according to the **Gp 2**. White solid; 78% yield, 356 mg; m.p. 242-244 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.63 (s, 1H), 7.53 (s, 1H), 7.46-7.45 (d, *J* = 4 Hz, 1H), 7.41-7.27 (m, 12H), 5.99-5.97 (d, *J* = 8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 154.55, 146.48, 142.77, 139.98, 131.89, 131.20, 130.29, 129.88, 129.13, 127.74, 127.49, 126.47, 122.28, 120.00, 113.07, 56.85; HRMS (ESI) *m/z* calcd for C<sub>20</sub>H<sub>16</sub>Br<sub>2</sub>N<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 480.9522; found: 480.9515.

### 1-(4-bromophenyl)-3-(phenyl(o-tolyl)methyl)urea (3l)



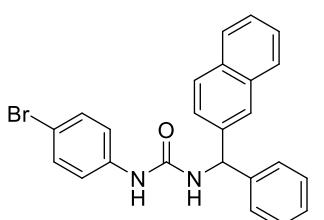
The product was prepared according to the **Gp 2**. White solid; 78% yield, 307 mg; m.p. 242-244 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.58 (s, 1H), 7.40-7.34 (m, 6H), 7.28-7.24 (m, 3H), 7.21-7.17 (m, 4H), 7.11-7.09 (d, *J* = 8 Hz, 1H), 6.14-6.12 (d, *J* = 8 Hz, 1H), 2.28 (s, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 154.46, 142.56, 141.40, 140.07, 135.80, 131.89, 130.89, 128.95, 127.68, 127.50, 127.01, 126.55, 119.88, 112.95, 54.02, 19.52; HRMS (ESI) *m/z* calcd for C<sub>21</sub>H<sub>19</sub>BrN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 417.0573; found: 417.0571.

### 1-(4-bromophenyl)-3-(naphthalen-1-yl(phenyl)methyl)urea (3m)



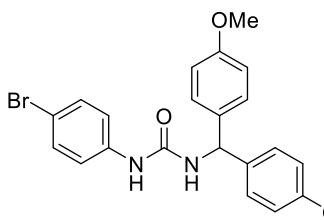
The product was prepared according to the **Gp 2**. White solid; 78% yield; m.p. 273-275 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.62 (s, 1H), 8.08-8.06 (m, 1H), 7.98-7.96 (m, 1H), 7.90-7.88 (d, *J* = 8 Hz, 1H), 7.55-7.51 (m, 3H), 7.43-7.35 (m, 9H), 7.29-7.26 (m, 2H), 6.74-6.72 (d, *J* = 8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 154.45, 142.77, 140.04, 138.84, 134.02, 131.91, 130.92, 129.17, 129.04, 128.31, 127.85, 127.66, 126.87, 126.24, 125.89, 124.98, 124.07, 119.92, 113.02, 53.98; HRMS (ESI) *m/z* calcd for C<sub>24</sub>H<sub>19</sub>BrN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 453.0573; found: 453.0563.

### 1-(4-bromophenyl)-3-(naphthalen-2-yl(phenyl)methyl)urea (3n)



The product was prepared according to the **Gp 2**. White solid; 95% yield; m.p. 216-218 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.69 (s, 1H), 7.90-7.86 (m, 4H), 7.53-7.35 (m, 12H), 7.28-7.24 (m, 1H), 6.16-6.14 (m, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 154.64, 143.30, 141.03, 140.08, 133.29, 132.53, 131.91, 129.04, 128.66, 128.20, 127.99, 127.58, 126.83, 126.40, 126.00, 125.42, 119.94, 113.00, 57.46; HRMS (ESI) *m/z* calcd for C<sub>24</sub>H<sub>19</sub>BrN<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 453.0573; found: 453.0589.

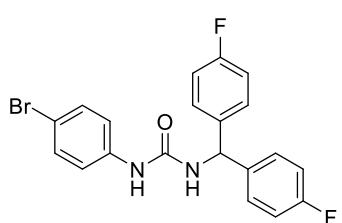
### 1-(bis(4-methoxyphenyl)methyl)-3-(4-bromophenyl)urea (3o)



The product was prepared according to the **Gp 2**. White solid; 76% yield; m.p. 201-203 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.58 (s, 1H), 7.40-7.34 (m, 4H), 7.20-7.17 (d, *J* = 12 Hz, 4H), 7.08-7.06 (d, *J* = 8 Hz, 1H), 6.91-6.89 (d, *J* = 8 Hz, 4H), 5.86-5.84 (d, *J* = 8 Hz, 1H), 3.72 (s, 6H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 158.57, 154.51, 140.14, 135.86, 131.87, 128.44, 119.86, 114.24, 112.87, 56.04, 55.53; HRMS (ESI) *m/z* calcd for C<sub>24</sub>H<sub>21</sub>BrN<sub>2</sub>O<sub>5</sub> [(M+Na)<sup>+</sup>]: 509.0673; found: 509.0663.

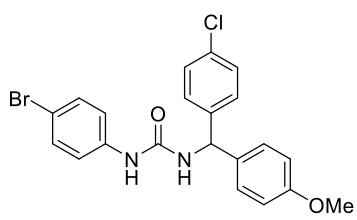
(ESI)  $m/z$  calcd for  $C_{22}H_{21}BrN_2NaO_3^+ [(M+Na)^+]$ : 463.0628; found: 463.0621.

**1-(bis(4-fluorophenyl)methyl)-3-(4-bromophenyl)urea (3p)**



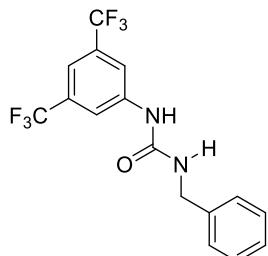
The product was prepared according to the **Gp 2**. White solid; 82% yield; m.p. 242-244 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  8.61 (s, 1H), 7.40-7.33 (m, 8H), 7.25-7.16 (m, 5H), 6.00-5.98 (d,  $J = 8$  Hz, 1H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  162.83, 160.41, 154.52, 140.00, 139.59, 139.56, 131.89, 129.39, 129.31, 119.99, 115.85, 115.64, 113.05, 55.94;  $^{19}F$  NMR (376 MHz, DMSO)  $\delta$  -115.71; HRMS (ESI)  $m/z$  calcd for  $C_{20}H_{15}BrF_2N_2NaO^+ [(M+Na)^+]$ : 439.0228; found: 439.0240.

**1-(4-bromophenyl)-3-((4-chlorophenyl)(4-methoxyphenyl)methyl)urea (3q)**



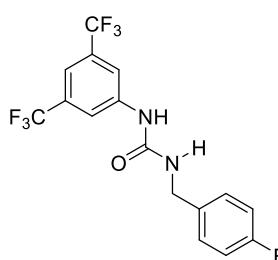
The product was prepared according to the **Gp 2**. White solid; 78% yield; m.p. 221-223 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  8.60 (s, 1H), 7.41-7.30 (m, 2H), 7.22-7.16 (m, 2H), 6.93-6.90 (d,  $J = 12$  Hz, 2H), 5.92-5.90 (d,  $J = 8$  Hz, 1H), 3.73 (s, 3H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  158.80, 154.52, 143.01, 140.03, 134.94, 131.88, 131.81, 129.06, 128.81, 128.71, 119.94, 114.42, 112.99, 56.13, 55.57; HRMS (ESI)  $m/z$  calcd for  $C_{21}H_{18}BrClN_2NaO_2^+ [(M+Na)^+]$ : 467.0132; found: 467.0151.

**1-benzyl-3-(3,5-bis(trifluoromethyl)phenyl)urea (5a)**



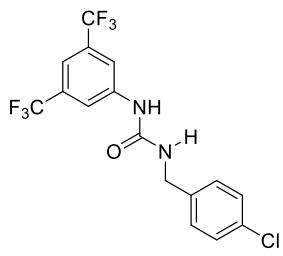
The product was prepared according to the **Gp 3**. White solid; 92% yield, 333 mg; m.p. 187-189 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  9.37 (s, 1H), 8.10 (s, 2H), 7.55(s, 1H), 7.35-7.29 (m, 4H), 7.26-7.22 (m, 1H), 7.04-7.01 (t,  $J = 6$  Hz, 1H), 4.33-4.31 (t,  $J = 8$  Hz, 1H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  155.32, 143.04, 140.43, 131.54-130.57(q,  $J = 32$  Hz), 128.74, 127.56, 127.21, 125.18, 122.47, 43.25;  $^{19}F$  NMR (376 MHz, DMSO)  $\delta$  -61.72; HRMS (ESI)  $m/z$  calcd for  $C_{16}H_{12}F_6N_2NaO^+ [(M+Na)^+]$ : 385.0746; found: 385.0740.

**1-(3,5-bis(trifluoromethyl)phenyl)-3-(4-fluorobenzyl)urea (5b)**



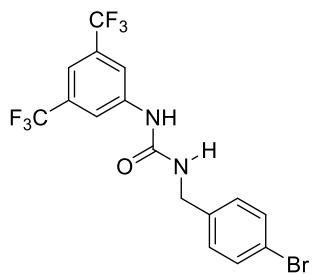
The product was prepared according to the **Gp 3**. White solid; 89% yield, 338 mg; m.p. 217-219 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  9.39 (s, 1H), 8.10 (s, 2H), 7.53(s, 1H), 7.36-7.33 (t,  $J = 6$  Hz, 2H), 7.17-7.12 (t,  $J = 10$  Hz, 2H), 7.05-7.02 (t,  $J = 6$  Hz, 1H), 4.31-4.29 (d,  $J = 8$  Hz, 2H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  162.82, 160.41, 155.30, 143.00, 136.68, 131.53-130.56 (q,  $J = 33$  Hz), 129.59, 129.51, 127.88-119.75(q,  $J = 271$  Hz), 117.77, 117.73, 115.54, 115.33, 113.92, 42.92;  $^{19}F$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.82, -116.31; HRMS (ESI)  $m/z$  calcd for  $C_{16}H_{11}F_7N_2NaO^+ [(M+Na)^+]$ : 403.0652; found: 403.0670.

**1-(3,5-bis(trifluoromethyl)phenyl)-3-(4-chlorobenzyl)urea (5c)**



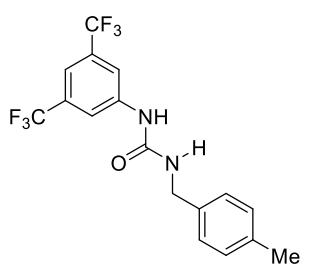
The product was prepared according to the **Gp 3**. White solid; 88% yield, 348 mg; m.p. 227-229 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.42 (s, 1H), 8.10 (s, 2H), 7.53 (s, 1H), 7.39-7.37 (d,  $J$  = 8 Hz, 2H), 7.34-7.31 (d,  $J$  = 12 Hz, 2H), 7.08-7.05 (t,  $J$  = 6 Hz, 1H), 4.31-4.30 (d,  $J$  = 4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  155.33, 142.98, 139.59, 131.74, 131.54-130.57 (q,  $J$  = 33 Hz), 129.41, 128.66, 127.87-119.75 (q,  $J$  = 271 Hz), 117.79, 113.96, 42.61;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.79; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{11}\text{F}_6\text{ClN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 419.0356; found: 419.0375.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(4-bromobenzyl)urea (5d)**



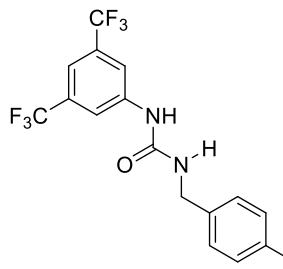
The product was prepared according to the **Gp 3**. White solid; 92% yield, 405 mg; m.p. 231-233 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.45 (s, 1H), 8.10 (s, 2H), 7.54-7.50 (m, 3H), 7.28-7.25 (d,  $J$  = 12 Hz, 2H), 7.10-7.07 (t,  $J$  = 6 Hz, 1H), 4.29-4.28 (t,  $J$  = 8 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  155.34, 142.98, 140.04, 131.59, 131.53-130.56 (q,  $J$  = 33 Hz, 1C), 129.79, 125.17, 122.46, 120.18, 117.83, 42.66;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.76; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{11}\text{BrF}_6\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 462.9851; found: 462.9779.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(4-methylbenzyl)urea (5e)**



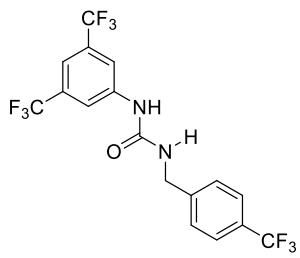
The product was prepared according to the **Gp 3**. White solid; 96% yield, 360 mg; m.p. 195-197 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.33 (s, 1H), 8.10 (s, 2H), 7.53 (s, 1H), 7.20-7.18 (t,  $J$  = 8 Hz, 2H), 7.14-7.12 (t,  $J$  = 8 Hz, 2H), 6.98-6.95 (t,  $J$  = 6 Hz, 1H), 4.28-4.26 (d,  $J$  = 8 Hz, 2H), 2.27 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  155.27, 143.04, 137.34, 136.27, 131.54-130.57 (q,  $J$  = 32 Hz, 1C), 129.28, 127.57, 125.18, 122.47, 117.75, 43.00, 21.10;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.79; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{15}\text{F}_6\text{N}_2\text{O}^+[(\text{M}+\text{H})^+]$ : 377.1083; found: 377.1099.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(4-methoxybenzyl)urea (5f)**



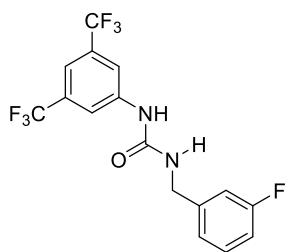
The product was prepared according to the **Gp 3**. White solid; 96% yield, 355 mg; m.p. 184-186 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.32 (s, 1H), 8.11 (s, 2H), 7.55 (s, 1H), 7.26-7.23 (d,  $J$  = 8 Hz, 2H), 6.96-6.93 (t,  $J$  = 8 Hz, 1H), 6.91-6.89 (d,  $J$  = 8 Hz, 2H), 4.26-4.25 (t,  $J$  = 4 Hz, 1H), 3.74 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  158.69, 155.23, 143.04, 132.30, 131.53-130.56 (q,  $J$  = 32 Hz, 1C), 128.97, 127.89, 125.18, 122.47, 119.76, 117.74, 114.16, 55.50, 42.72;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.77; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{15}\text{F}_6\text{N}_2\text{O}_2^+[(\text{M}+\text{H})^+]$ : 393.1032; found: 393.1045.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(4-(trifluoromethyl)benzyl)urea (5g)**



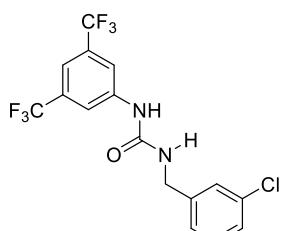
The product was prepared according to the **Gp 3**. White solid; 94% yield, 404 mg; m.p. 209-211 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.53 (s, 1H), 8.13 (s, 2H), 7.72-7.70 (d,  $J$  = 8 Hz, 2H), 7.57-7.55 (d,  $J$  = 8 Hz, 2H), 7.53 (s, 1H), 7.20-7.17 (t,  $J$  = 6 Hz, 1H), 4.44-4.42 (d,  $J$  = 8 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  155.39, 145.54, 142.94, 131.52-130.55(q,  $J$  = 32 Hz), 128.11, 127.72, 126.16, 125.66-125.55(q,  $J$  = 4 Hz), 125.16, 123.45, 122.45, 42.90;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -60.87, -61.82; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{11}\text{F}_9\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 453.0620; found: 453.0653.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(3-fluorobenzyl)urea (5h)**



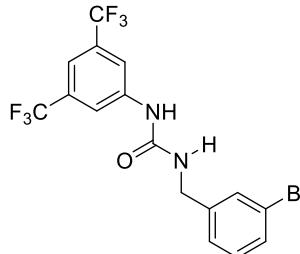
The product was prepared according to the **Gp 3**. White solid; 95% yield, 361 mg; m.p. 194-196 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.45 (s, 1H), 8.13 (s, 2H), 7.55(s, 1H), 7.42-7.36 (q,  $J$  = 8 Hz, 1H), 7.18-7.05 (m, 2H), 4.37-4.36 (d,  $J$  = 4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  163.92, 161.50, 155.34, 143.67-130.62(q,  $J$  = 7 Hz,  $J$  = 1290 Hz, 1C), 142.97, 131.54-130.57(q,  $J$  = 32 Hz, 1C), 125.17, 123.48, 122.46, 114.23, 114.01, 113.80, 42.77;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.84, -113.64; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{11}\text{F}_7\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 403.0652; found: 403.0599.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(3-chlorobenzyl)urea (5i)**



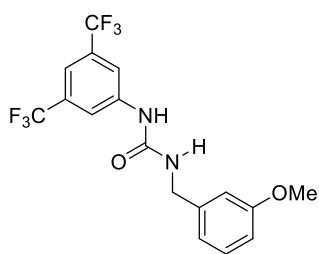
The product was prepared according to the **Gp 3**. White solid; 94% yield, 372 mg; m.p. 170-172 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.44 (s, 1H), 8.10 (s, 2H), 7.53 (s, 1H), 7.37-7.34 (m, 2H), 7.30-7.26 (m, 2H), 7.12-7.09 (t,  $J$  = 6 Hz, 1H), 4.33-4.32 (d,  $J$  = 4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  155.33, 143.22, 142.96, 133.44, 131.54-130.57(q,  $J$  = 32 Hz, 1C), 130.62, 127.30, 127.12, 126.21, 125.17, 122.46, 117.81, 42.73;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.82; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{11}\text{F}_6\text{ClN}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 419.0356; found: 419.0375.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(3-bromobenzyl)urea (5j)**



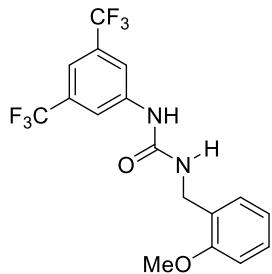
The product was prepared according to the **Gp 3**. White solid; 92% yield, 405 mg; m.p. 157-159 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  9.45 (s, 1H), 8.12 (s, 2H), 7.56-7.52 (d,  $J$  = 16 Hz, 2H), 7.47-7.44 (m, 1H), 7.35-7.29 (m, 2H), 7.13-7.10 (t,  $J$  = 6 Hz, 1H), 4.34-4.33 (d,  $J$  = 4Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  155.31, 143.49, 142.95, 131.54-130.57(q,  $J$  = 32 Hz, 1C), 130.95, 130.20, 130.04, 126.63, 125.17, 122.46, 122.08, 42.69;  $^{19}\text{F}$  NMR (376 MHz, DMSO-d6)  $\delta$  -61.79; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{11}\text{BrF}_6\text{N}_2\text{NaO}^+[(\text{M}+\text{Na})^+]$ : 462.9851; found: 462.9787.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(3-methoxybenzyl)urea (5k)**



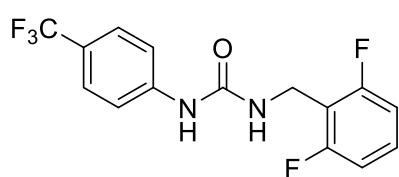
The product was prepared according to the **Gp 3**. White solid; 96% yield, 376 mg; m.p. 149-151 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.38 (s, 1H), 8.12 (s, 2H), 7.55 (s, 1H), 7.28-7.24 (t,  $J = 8$  Hz, 1H), 7.04-7.01 (t,  $J = 6$  Hz, 1H), 6.91-6.89 (m, 2H), 6.84-6.82 (d,  $J = 8$  Hz, 1H), 4.32-4.31 (d,  $J = 8$  Hz, 2H), 3.75 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  159.78, 155.29, 143.02, 142.04, 131.54-130.57 (q,  $J = 32$  Hz, 1C), 129.83, 125.17, 122.47, 119.76, 119.69, 117.78, 113.26, 112.51;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.80; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{14}\text{F}_6\text{N}_2\text{O}_2\text{Na}^+ [(\text{M}+\text{Na})^+]$ : 415.0852; found: 415.0793.

### **1-(3,5-bis(trifluoromethyl)phenyl)-3-(2-methoxybenzyl)urea (5l)**



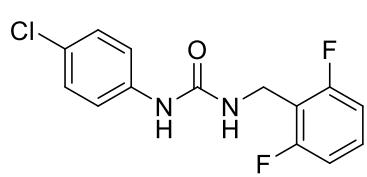
The product was prepared according to the **Gp 3**. White solid; 94% yield, 368 mg; m.p. 175-177 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  9.39 (s, 1H), 8.09 (s, 2H), 7.54 (s, 1H), 7.28-7.23 (m, 2H), 7.02-7.00 (d,  $J = 8$  Hz, 1H), 6.95-6.91 (t,  $J = 8$  Hz, 1H), 6.82-6.79 (t,  $J = 6$  Hz, 1H), 4.31-4.29 (d,  $J = 8$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  157.19, 155.23, 143.00, 131.56-130.59 (q,  $J = 32$  Hz, 1C), 128.65, 128.33, 127.88, 127.62, 125.17, 122.46, 120.61, 119.75, 117.60, 113.90, 110.92, 55.77, 38.69;  $^{19}\text{F}$  NMR (376 MHz,  $\text{DMSO-d}_6$ )  $\delta$  -61.80; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{14}\text{F}_6\text{N}_2\text{O}_2\text{Na}^+ [(\text{M}+\text{Na})^+]$ : 415.0852; found: 415.0805.

### **1-(2,6-difluorobenzyl)-3-(4-(trifluoromethyl)phenyl)urea (5m)**



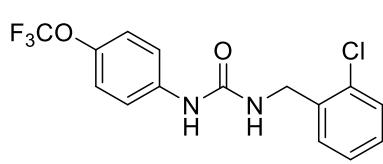
The product was prepared according to the **Gp 3**. White solid; 96% yield, 376 mg; m.p. 166-168 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  8.92 (s, 1H), 7.60-7.55 (m, 4H), 7.44-7.37 (m, 1H), 7.13-7.09 (t,  $J = 8$  Hz, 2H), 6.81-6.78 (t,  $J = 6$  Hz, 1H), 4.42-4.40 (d,  $J = 8$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  162.55, 160.18, 154.84, 144.39, 130.30, 126.43, 123.71, 122.09, 121.77, 121.45, 121.14, 117.68, 115.32, 112.14, 111.89, 31.45;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -60.03, -115.15; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{15}\text{H}_{11}\text{F}_5\text{N}_2\text{NaO}^+ [(\text{M}+\text{Na})^+]$ : 353.0684; found: 353.0693.

### **1-(4-chlorophenyl)-3-(3,5-difluorobenzyl)urea (5n)**



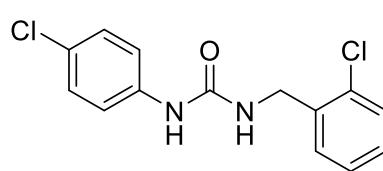
The product was prepared according to the **Gp 3**. White solid; 97% yield, 380 mg; m.p. 207-209 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  9.59 (s, 1H), 7.41-7.35 (m, 3H), 7.26-7.24 (d,  $J = 8$  Hz, 2H), 7.12-7.08 (t,  $J = 8$  Hz, 2H), 6.66-6.63 (t,  $J = 6$  Hz, 1H), 4.39-4.37 (d,  $J = 8$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  162.55, 160.10, 155.01, 139.68, 130.25, 128.93, 125.11, 119.55, 115.46, 112.13, 111.88, 31.45;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -115.16; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{14}\text{H}_{11}\text{ClF}_2\text{N}_2\text{NaO}^+ [(\text{M}+\text{Na})^+]$ : 319.0420; found: 319.0428.

### **1-(4-chlorophenyl)-3-(3,5-difluorobenzyl)urea (5o)**



The product was prepared according to the **Gp 3**. White solid; 98% yield, 384 mg; m.p. 178-180 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.94 (s, 1H), 7.53-7.51 (d, *J* = 8 Hz, 2H), 7.46-7.44 (d, *J* = 8 Hz, 1H), 7.42-7.40 (d, *J* = 8 Hz, 1H), 7.36-7.28 (m, 2H), 7.25-7.22 (d, *J* = 8 Hz, 2H), 6.77-6.74 (t, *J* = 6 Hz, 1H), 4.39-4.38 (d, *J* = 4 Hz, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 155.47, 142.56, 140.13, 137.65, 132.44, 129.56, 129.28, 129.03, 127.65, 122.09, 119.21, 41.21; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.20; HRMS (ESI) *m/z* calcd for C<sub>15</sub>H<sub>12</sub>ClF<sub>3</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 367.0432; found: 367.0422.

### 1-(2-chlorobenzyl)-3-(4-chlorophenyl)urea (5p)

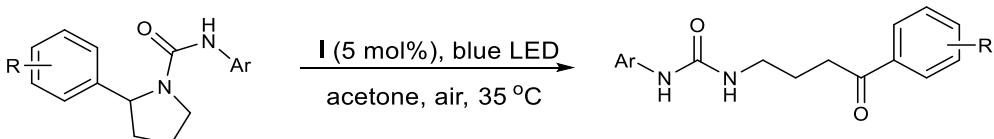


The product was prepared according to the **Gp 3**. White solid; 94% yield; m.p. 193-195 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.86 (s, 1H), 7.46-7.40 (m, 3H), 7.36-7.26 (m, 2H), 6.75-6.72 (t, *J* = 6 Hz, 1H), 4.39-4.37 (d, *J* = 8 Hz, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 155.44, 139.81, 137.66, 132.46, 129.56, 129.31, 129.03, 128.95, 127.65, 125.12, 119.63, 41.22; HRMS (ESI) *m/z* calcd for C<sub>14</sub>H<sub>12</sub>Cl<sub>2</sub>N<sub>2</sub>NaO<sup>+</sup> [(M+Na)<sup>+</sup>]: 317.0219; found: 317.0217.

## General procedures of the oxidation of amines by photoredox catalysis.

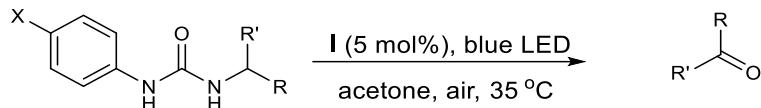


General procedures for the synthesis of **2** (**Gp 4**).



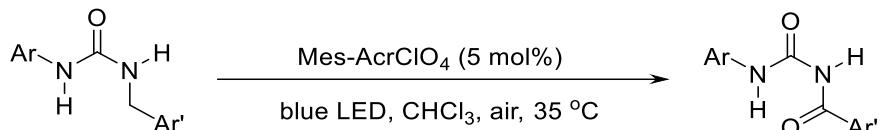
To a solution of substrate (0.05 mmol) in acetone (2 mL), the catalyst (5 mol%) was added into the mixture. The mixture was stirred at room temperature utilizing blue LED until the completion of the reaction determined by TLC analysis. The crude product was purified by flash chromatography on silica gel (eluent: PE/EtOAc from 5:1 to 2:1) affording to the compounds.

General procedures for the synthesis of **4** (Gp **5**).



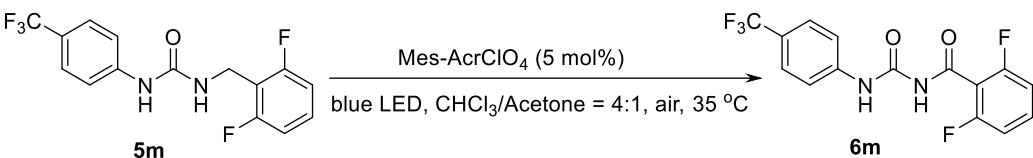
To a solution of substrate (0.05 mmol) in acetone (2 mL), the catalyst (5 mol%) was added into the mixture. Then the mixture was stirred at room temperature utilizing blue LED until the completion of the reaction determined by TLC analysis. The crude product was purified by flash chromatography on silica gel (eluent: PE/EtOAc from 10:1 to 5:1) affording to the compounds.

General procedures for the synthesis of **6** (Gp **6**).



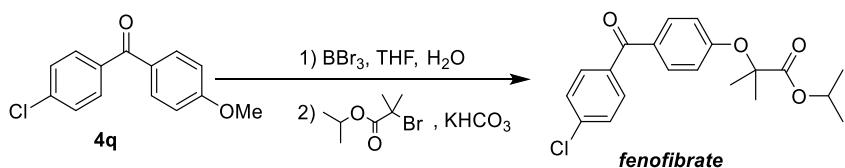
To a solution of substrate (0.05 mmol) in  $\text{CHCl}_3$  (2 mL), the catalyst (5 mol%) was added into the mixture. The mixture was stirred at room temperature utilizing blue LED until the completion of the reaction determined by TLC analysis). The crude product was purified by flash chromatography on silica gel (eluent: PE/EtOAc from 10:1 to 5:1) affording to the compounds.

## The gram scale reaction



To a solution of Mes-AcrClO<sub>4</sub> (5 mol%, 60mg) in CHCl<sub>3</sub> (40 mL) and acetone (10 mL) [Acetone was used for promoting the dissolution of **5m**], **5m** (3.1 mmol, 1.03g.) was added into the mixture which was equipped with an O<sub>2</sub> balloon. The mixture was stirred at room temperature utilizing blue LED until the completion of the reaction determined by TLC analysis. The crude product was purified by flash chromatography on silica gel (eluent: PE/EtOAc from 5:1 to 2:1) affording to the corresponding compounds.

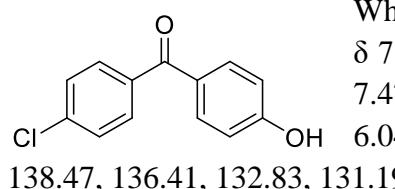
## The preparation of fenofibrate



**4q** (0.3 mmol) were weighed into an oven-dried 50 mL flask containing anhydrous THF (15 mL), BBr<sub>3</sub> (1 mmol) was added into the mixture at -78 °C. The resulting mixture was warmed to room temperature and stirred for 2 h. H<sub>2</sub>O was then added dropwise to quench the reaction at 0 °C. Then the volatile fraction was removed in vacuo. The residue was purified by flash chromatography on silica gel (eluent: PE/EtOAc from 5:1 to 1:1). The above collected solid was dissolved in

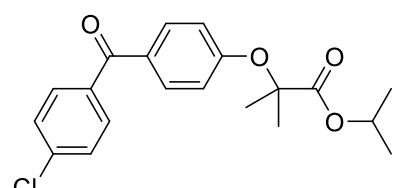
isopropylalcohol (10 mL), and then KHCO<sub>3</sub> (0.5 mmol) was slowly added into the mixture, which was stirred for 10 min at room temperature. After that, isopropyl 2-bromo-2-methylpropanoate (0.5 mmol) was added into the mixture, and the solution was heated to reflux temperature for 60 h. After the completion of the reaction, the volatile fraction was removed in vacuo. The residue was purified by flash chromatography on silica gel (eluent: PE/EtOAc from 5:1 to 2:1) affording to the fenofibrate as a white solid (82mg, 85%).<sup>5</sup>

#### (4-chlorophenyl)(4-hydroxyphenyl)methanone



White solid, 92% yield, 63 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.77-7.75 (d, *J* = 8 Hz, 2H), 7.72-7.70 (d, *J* = 8 Hz, 2H), 7.47-7.45 (d, *J* = 8 Hz, 2H), 6.93-6.91 (d, *J* = 8 Hz, 2H), 6.04 (s, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 194.63, 160.09, 138.47, 136.41, 132.83, 131.19, 129.84, 128.58, 115.32.

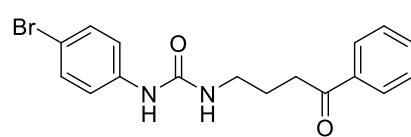
#### Fenofibrate



White solid; 85% yield, 62 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.74-7.69 (m, 4H), 7.46-7.44 (d, *J* = 8 Hz, 2H), 6.88-6.86 (d, *J* = 8 Hz, 2H), 5.12-5.05 (m, 1H), 1.66 (s, 6H), 1.21-1.20 (d, *J* = 4 Hz, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 194.23, 173.08, 159.74, 138.34, 136.43, 131.94, 131.15, 130.22, 128.53, 117.26, 79.43, 69.34, 25.37, 21.53.

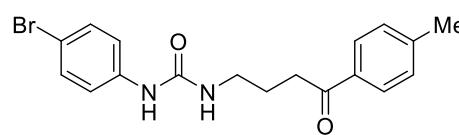
## Characterization data for the products

#### 1-(4-bromophenyl)-3-(4-oxo-4-phenylbutyl) urea (2a)



The product was prepared according to the **Gp 4**. White solid; 73% yield, 13.7 mg; m.p. 173-175 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.58 (s, 1H), 7.98-7.96 (s, 2H), 7.65-7.62 (t, *J* = 6 Hz, 1H), 7.55-7.51 (t, *J* = 8 Hz, 1H), 7.37 (s, 4H), 6.30-6.27 (t, *J* = 6 Hz, 1H), 3.18-3.13 (q, *J* = 8 Hz, 2H), 3.09-3.05 (t, *J* = 8 Hz, 2H), 1.83-1.76 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 200.11, 155.53, 140.45, 137.13, 133.54, 131.75, 129.15, 128.31, 120.01, 112.65, 39.09, 35.75, 24.89; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 383.0366; found: 383.0375.

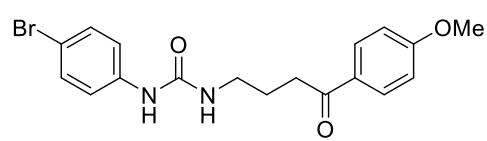
#### 1-(4-bromophenyl)-3-(4-oxo-4-(p-tolyl)butyl)urea (2b)



The product was prepared according to the **Gp 4**. White solid; 55% yield, 10.3 mg; m.p. 177-179 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.57 (s, 1H), 7.87-7.85 (d, *J* = 8 Hz, 2H), 7.38-7.36 (m, 4H), 7.33-7.31 (d, *J* = 8 Hz, 2H), 6.28-6.25 (t, *J* = 6 Hz, 1H), 3.16-3.11 (q, *J* = 8 Hz, 2H), 3.04-3.00 (t, *J* = 8 Hz, 2H), 2.37 (s, 3H), 1.80-1.73 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 199.64, 155.50, 143.86, 140.44, 134.64, 131.76, 129.69, 128.44, 119.99,

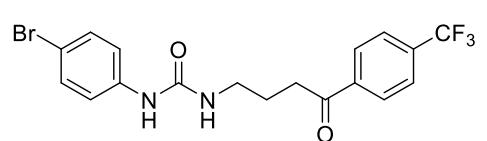
112.63, 39.11, 35.62, 24.97, 21.60; HRMS (ESI)  $m/z$  calcd for  $C_{18}H_{19}BrN_2NaO_2^+$  [(M+Na) $^+$ ]: 397.0522; found: 397.0481.

**1-(4-bromophenyl)-3-(4-methoxyphenyl)-4-oxobutylurea (2c)**



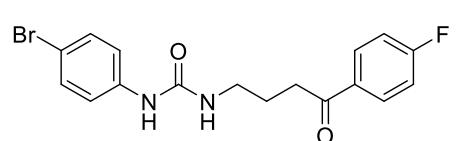
The product was prepared according to the **Gp 4**. White solid; 81% yield, 16.6 mg; m.p. 171-173 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  8.57 (s, 1H), 7.95-7.93 (d,  $J$  = 8 Hz, 2H), 7.36 (s, 4H), 7.04-7.02 (d,  $J$  = 8 Hz, 2H), 6.28-6.25 (t,  $J$  = 6 Hz, 1H), 3.83 (s, 3H), 3.16-3.11 (q,  $J$  = 8 Hz, 2H), 3.01-2.97 (t,  $J$  = 8 Hz, 2H), 1.80-1.73 (m, 2H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  198.49, 163.48, 155.51, 140.45, 131.76, 130.63, 130.05, 119.99, 114.31, 112.63, 55.97, 35.36, 25.09; HRMS (ESI)  $m/z$  calcd for  $C_{18}H_{19}BrN_2NaO_3^+$  [(M+Na) $^+$ ]: 413.0471; found: 413.0471.

**1-(4-bromophenyl)-3-(4-oxo-4-(4-(trifluoromethyl)phenyl)butyl)urea (2d)**



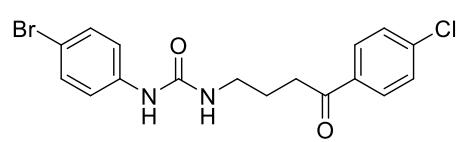
The product was prepared according to the **Gp 4**. White solid; 55% yield, 11.8 mg; m.p. 170-172 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  8.59 (s, 1H), 8.15-8.13 (d,  $J$  = 8 Hz, 2H), 7.90-7.88 (d,  $J$  = 8 Hz, 2H), 7.38-7.33 (m, 4H), 6.29-6.26 (t,  $J$  = 6 Hz, 1H), 3.18-3.11 (m, 4H), 1.84-1.77 (m, 2H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  199.57, 155.51, 140.43, 140.27, 133.32-132.37 (q,  $J$  = 33 Hz), 131.99, 131.74, 129.13, 128.32-120.20 (q,  $J$  = 270 Hz), 126.17, 120.20, 119.96, 112.63, 38.94, 36.10, 24.66;  $^{19}F$  NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -112.35; HRMS (ESI)  $m/z$  calcd for  $C_{18}H_{16}BrF_3N_2NaO_2^+$  [(M+Na) $^+$ ]: 451.0239; found: 451.0253.

**1-(4-bromophenyl)-3-(4-(4-fluorophenyl)-4-oxobutyl)urea (2e)**



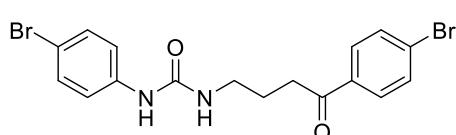
The product was prepared according to the **Gp 4**. White solid; 79% yield, 14.9 mg; m.p. 190-192 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  8.57 (s, 1H), 8.07-8.03 (s, 2H), 7.36-7.33 (m, 6H), 6.28-6.25 (t,  $J$  = 6 Hz, 1H), 3.17-3.12 (q,  $J$  = 8 Hz, 2H), 3.07-3.04 (t,  $J$  = 6 Hz, 2H), 1.82-1.75 (m, 2H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  198.71, 155.51, 140.44, 133.85, 131.75, 131.25, 119.99, 116.24, 116.02, 112.64, 39.05, 35.69, 24.85;  $^{19}F$  NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -106.35; HRMS (ESI)  $m/z$  calcd for  $C_{17}H_{16}BrFN_2NaO_2^+$  [(M+Na) $^+$ ]: 401.0271; found: 401.0256.

**1-(4-bromophenyl)-3-(4-(4-chlorophenyl)-4-oxobutyl)urea (2f)**



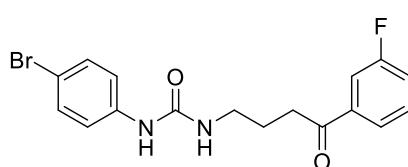
The product was prepared according to the **Gp 4**. White solid; 86% yield, 16.9 mg; m.p. 162-164 °C;  $^1H$  NMR (400 MHz, DMSO-d6)  $\delta$  8.57 (s, 1H), 7.98-7.96 (d,  $J$  = 8 Hz, 2H), 7.60-7.58 (d,  $J$  = 8 Hz, 2H), 7.36 (s, 4H), 6.27-6.25 (t,  $J$  = 4 Hz, 1H), 3.17-3.12 (q,  $J$  = 8 Hz, 2H), 3.07-3.04 (t,  $J$  = 8 Hz, 2H), 1.81-1.74 (m, 2H);  $^{13}C$  NMR (100 MHz, DMSO-d6)  $\delta$  199.13, 155.51, 140.44, 138.43, 135.79, 131.75, 130.25, 129.26, 119.99, 112.64, 39.02, 35.77, 24.80; HRMS (ESI)  $m/z$  calcd for  $C_{17}H_{16}BrClN_2NaO_2^+$  [(M+Na) $^+$ ]: 416.9976; found: 416.9962.

**1-(4-bromophenyl)-3-(4-(4-bromophenyl)-4-oxobutyl)urea (2g)**



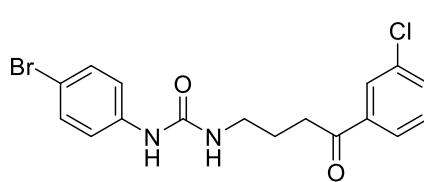
The product was prepared according to the **Gp 4**. White solid; 69% yield, 15.1 mg; m.p. 178-180 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.58 (s, 1H), 7.90-7.88 (s, 2H), 7.74-7.72 (t, *J*=6 Hz, 1H), 7.38-7.33 (m, 4H), 6.28-6.25 (t, *J*=6 Hz, 1H), 3.17-3.12 (q, *J*=8 Hz, 2H), 3.07-3.03 (t, *J*=8 Hz, 2H), 1.81-1.74 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 199.35, 155.51, 140.43, 136.11, 132.21, 131.75, 130.36, 127.60, 119.99, 112.65, 39.01, 35.74, 24.78.; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>Br<sub>2</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 460.9471; found: 460.9484.

### 1-(4-bromophenyl)-3-(4-(3-fluorophenyl)-4-oxobutyl)urea (2h)



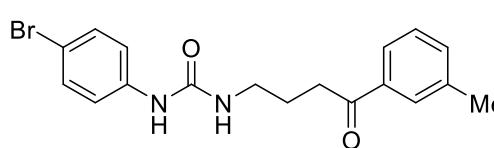
The product was prepared according to the **Gp 4**. White solid; 39% yield, 7.4 mg; m.p. 184-186 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.59 (s, 1H), 7.83-7.81 (d, *J*=8 Hz, 1H), 7.73-7.71 (d, *J*=8 Hz, 1H), 7.61-7.56 (m, 1H), 7.51-7.48 (m, 4H), 6.29-6.26 (t, *J*=6 Hz, 1H), 3.17-3.12 (q, *J*=8 Hz, 2H), 3.10-3.06 (t, *J*=8 Hz, 2H), 1.81-1.74 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 199.09, 167.42, 163.88, 161.44, 155.50, 140.44, 139.39, 131.99, 131.75, 131.34, 129.13, 124.55, 120.55, 120.34, 119.97, 114.90, 114.68, 112.63; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -112.35; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>BrFN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 401.0271; found: 401.0279.

### 1-(4-bromophenyl)-3-(4-(3-chlorophenyl)-4-oxobutyl)urea (2i)



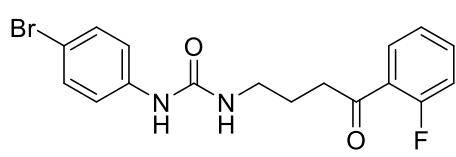
The product was prepared according to the **Gp 4**. White solid; 58% yield, 11.4 mg; m.p. 166-168 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.60 (s, 1H), 7.96-7.91 (m, 2H), 7.72-7.70 (d, *J*=8 Hz, 1H), 7.58-7.54 (t, *J*=8 Hz, 1H), 7.37-7.33 (m, 4H), 6.29-6.26 (t, *J*=6 Hz, 1H), 3.16-3.07 (m, 4H), 1.81-1.74 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 199.10, 155.50, 140.44, 138.98, 134.11, 133.24, 131.75, 131.19, 128.00, 126.99, 119.97, 112.62, 38.97, 35.90, 24.70; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>BrClN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 416.9976; found: 416.9949.

### 1-(4-bromophenyl)-3-(4-oxo-4-(m-tolyl)butyl)urea (2j)



The product was prepared according to the **Gp 4**. White solid; 50% yield, 9.4 mg; m.p. 151-153 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.58 (s, 1H), 7.77-7.75 (m, 2H), 7.46-7.39 (m, 2H), 7.38-7.34 (m, 4H), 6.29-6.27 (m, 1H), 3.17-3.12 (q, *J*=8 Hz, 2H), 3.06-3.03 (t, *J*=8 Hz, 2H), 1.81-1.74 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 200.21, 155.51, 140.45, 138.49, 137.17, 134.13, 131.76, 129.05, 128.74, 125.55, 119.98, 112.63, 39.08, 35.78, 24.90, 21.35; HRMS (ESI) *m/z* calcd for C<sub>18</sub>H<sub>19</sub>BrN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 397.0522; found: 397.0481.

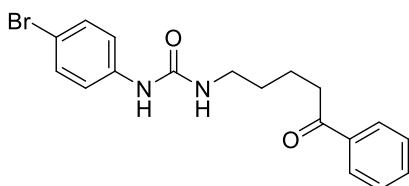
### 1-(4-bromophenyl)-3-(4-(2-fluorophenyl)-4-oxobutyl)urea (2k)



The product was prepared according to the **Gp 4**. White solid; 39% yield, 7.4 mg; m.p. 184-186 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.59 (s, 1H), 7.83-7.81 (d, *J*=8 Hz, 1H), 7.73-7.71 (d, *J*=8

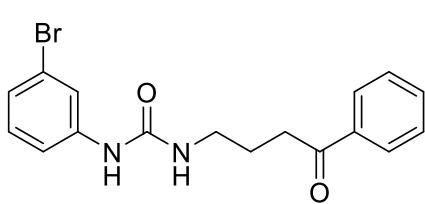
Hz, 1H), 7.61-7.56 (m, 1H), 7.52-7.47 (m, 1H), 7.39-7.35 (m, 4H), 6.29-6.26 (t,  $J$  = 6 Hz, 1H), 3.18-3.13 (q,  $J$  = 8 Hz, 2H), 3.10-3.06 (t,  $J$  = 8 Hz, 2H), 1.83-1.76 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  199.11, 163.88, 161.44, 155.52, 140.43, 139.34, 131.75, 131.32, 124.57, 120.53, 120.32, 120.00, 114.89, 114.67, 112.65, 39.01, 35.94, 24.76;  $^{19}\text{F}$  NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -112.34; HRMS (ESI)  $m/z$  calcd for C<sub>17</sub>H<sub>16</sub>BrFN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 401.0271; found: 401.0273.

### 1-(4-bromophenyl)-3-(5-(4-methoxyphenyl)-5-oxopentyl)urea (2l)



The product was prepared according to the **Gp 4**. White solid; 32% yield; m.p. 186-188 °C, 6.0 mg;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.59 (s, 1H), 7.98-7.96 (d,  $J$  = 8 Hz, 2H), 7.65-7.61 (t,  $J$  = 8 Hz, 1H), 7.54-7.50 (t,  $J$  = 8 Hz, 2H), 7.40-7.35 (m, 4H), 6.24-6.21 (t,  $J$  = 6 Hz, 1H), 3.14-3.09 (q,  $J$  = 8 Hz, 2H), 3.08-3.04 (t,  $J$  = 8 Hz, 2H), 1.67-1.60 (m, 2H), 1.52-1.45 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  200.48, 155.46, 140.48, 137.13, 133.54, 131.78, 129.17, 128.35, 119.90, 112.58, 39.23, 38.01, 29.74, 21.61; HRMS (ESI)  $m/z$  calcd for C<sub>19</sub>H<sub>21</sub>BrN<sub>2</sub>NaO<sub>3</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 397.0522; found: 397.0469.

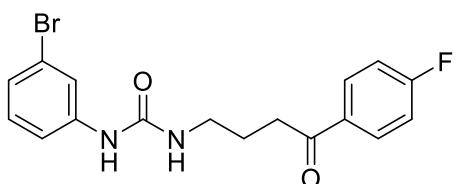
### 1-(3-bromophenyl)-3-(4-oxo-4-phenylbutyl)urea (2m)



The product was prepared according to the **Gp 4**. White solid; 45% yield, 8.1 mg; m.p. 177-179 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.65 (s, 1H), 7.98-7.96 (d,  $J$  = 8 Hz, 2H), 7.80 (s, 1H), 7.65-7.62 (m, 1H), 7.54-7.51 (t,  $J$  = 6 Hz, 1H), 7.22-7.14 (m, 1H), 7.06-7.04 (d,  $J$  = 8 Hz, 2H), 6.34-6.31 (t,  $J$  = 6

Hz, 1H), 3.18-3.13 (q,  $J$  = 8 Hz, 2H), 3.09-3.05 (t,  $J$  = 8 Hz, 2H), 1.82-1.75 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  200.08, 155.43, 142.72, 137.09, 133.56, 130.97, 129.16, 128.31, 123.85, 122.11, 120.26, 116.81, 39.07, 35.71, 24.83; HRMS (ESI)  $m/z$  calcd for C<sub>17</sub>H<sub>17</sub>BrN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 383.0366; found: 383.0273.

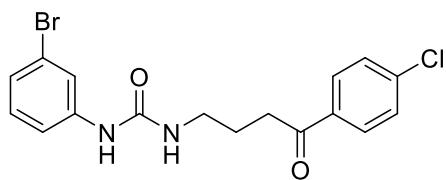
### 1-(3-bromophenyl)-3-(4-(4-fluorophenyl)-4-oxobutyl)urea (2n)



The product was prepared according to the **Gp 4**. White solid; 51% yield, 9.6 mg; m.p. 172-174 °C;  $^1\text{H}$  NMR (400 MHz, DMSO-d6)  $\delta$  8.65 (s, 1H), 8.06-8.03 (d,  $J$  = 8 Hz, 2H), 7.79 (s, 1H), 7.37-7.32 (d,  $J$  = 10 Hz, 2H), 7.22-7.14 (m, 2H), 7.06-

7.04 (m, 2H), 6.33-6.30 (t,  $J$  = 6 Hz, 1H), 3.17-3.12 (q,  $J$  = 8 Hz, 2H), 3.07-3.04 (t,  $J$  = 6 Hz, 2H), 1.82-1.75 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO-d6)  $\delta$  198.68, 166.65, 164.15, 155.43, 142.71, 133.87, 131.34, 131.25, 130.97, 123.85, 122.10, 120.25, 116.80, 116.24, 116.02, 39.03, 35.65, 24.80;  $^{19}\text{F}$  NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -106.33; HRMS (ESI)  $m/z$  calcd for C<sub>17</sub>H<sub>16</sub>BrFN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 401.0271; found: 401.0176.

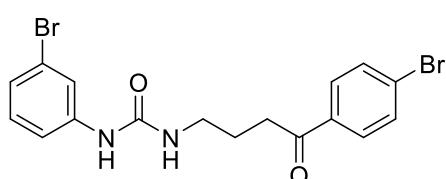
### 1-(3-bromophenyl)-3-(4-(4-chlorophenyl)-4-oxobutyl)urea (2o)



The product was prepared according to the **Gp 4**.  
White solid; 38% yield, 7.5 mg; m.p. 175-177 °C;  
<sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.65 (s, 1H),  
7.90-7.88 (d, *J* = 8 Hz, 2H), 7.79 (s, 4H), 7.74-  
7.72 (d, *J* = 8 Hz, 2H), 7.21-7.13 (m, 2H), 7.06-

7.04 (m, 2H), 6.33-6.30 (t, *J* = 6 Hz, 1H), 3.17-3.12 (q, *J* = 8 Hz, 2H), 3.07-3.03 (t, *J* = 8 Hz, 2H), 1.81-1.74 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 199.64, 155.50,  
143.86, 140.44, 134.64, 131.76, 129.69, 128.44, 119.99, 112.63, 39.11, 35.62, 24.97,  
21.60; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>BrClN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 416.9976; found:  
416.9994.

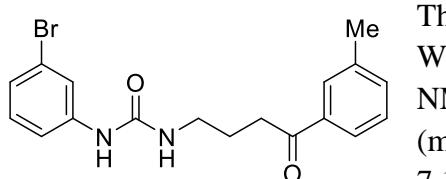
### 1-(3-bromophenyl)-3-(4-(4-bromophenyl)-4-oxobutyl)urea (2p)



The product was prepared according to the **Gp 4**.  
White solid; 47% yield, 10.8 mg; m.p. 193-195 °C;  
<sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 8.66 (s, 1H),  
7.98-7.96 (d, *J* = 8 Hz, 2H), 7.79 (m, 4H), 7.60-  
7.58 (d, *J* = 8 Hz, 2H), 7.21-7.14 (m, 2H), 7.06-

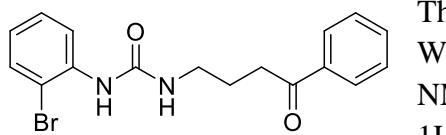
7.04 (m, 1H), 6.33-6.31 (t, *J* = 6 Hz, 1H), 3.17-3.12 (q, *J* = 8 Hz, 2H), 3.08-3.04 (t, *J* = 8 Hz, 2H), 1.81-1.74 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 199.11, 155.43,  
142.71, 138.43, 135.77, 130.97, 130.25, 129.25, 123.85, 122.10, 120.25, 116.80, 39.00,  
35.72, 24.74; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>Br<sub>2</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 460.9471;  
found: 460.9507.

### 1-(4-bromophenyl)-3-(4-oxo-4-(p-tolyl)butyl)urea (2q)



The product was prepared according to the **Gp 4**.  
White solid; 48% yield, 9.0 mg; m.p. 169-171 °C; <sup>1</sup>H  
NMR (400 MHz, DMSO-d6) δ 8.67 (s, 1H), 7.81-7.75  
(m, 3H), 7.45-7.38 (m, 2H), 7.22-7.20 (m, 1H), 7.18-  
7.14 (t, *J* = 8 Hz, 1H), 7.06-7.04 (d, *J* = 8 Hz, 1H),  
6.35-6.32 (t, *J* = 6 Hz, 1H), 3.17-3.12 (q, *J* = 8 Hz, 2H), 3.06-3.03 (t, *J* = 6 Hz, 2H),  
2.37 (s, 3H), 1.82-1.75 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 200.19, 155.43,  
142.73, 138.48, 137.16, 134.13, 130.97, 129.04, 128.75, 125.55, 123.85, 122.11, 120.24,  
116.80, 39.07, 35.76, 24.87, 21.36.; HRMS (ESI) *m/z* calcd for C<sub>18</sub>H<sub>19</sub>BrN<sub>2</sub>NaO<sub>2</sub><sup>+</sup>  
[(M+Na)<sup>+</sup>]: 397.0522; found: 397.0427.

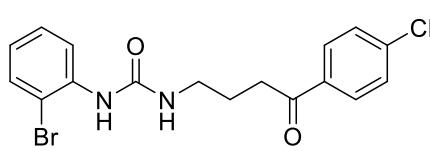
### 1-(2-bromophenyl)-3-(4-oxo-4-phenylbutyl)urea (2r)



The product was prepared according to the **Gp 4**.  
White solid; 49% yield, 8.82 mg; m.p. 183-185 °C; <sup>1</sup>H  
NMR (400 MHz, DMSO-d6) δ 8.05-8.03 (d, *J* = 8 Hz,  
1H), 7.99-7.97 (d, *J* = 8 Hz, 2H), 7.79 (s, 1H), 7.66-

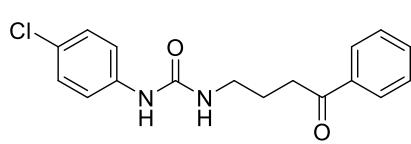
7.62 (t, *J* = 8 Hz, 1H), 7.56-7.51 (m, 3H), 7.28-7.24 (t, *J* = 8 Hz, 1H), 7.17-7.14 (t, *J* = 6 Hz, 1H), 6.90-6.86 (t, *J* = 8 Hz, 1H), 3.20-3.15 (q, *J* = 6 Hz, 2H), 3.11-3.08 (t, *J* = 6 Hz, 2H), 1.84-1.77 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 200.06, 155.24,  
138.28, 137.10, 133.58, 132.76, 129.18, 128.39, 128.32, 123.56, 121.93, 112.60, 39.06,  
35.75, 24.76; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 383.0366;  
found: 383.0273.

### 1-(2-bromophenyl)-3-(4-(4-chlorophenyl)-4-oxobutyl)urea (2s)



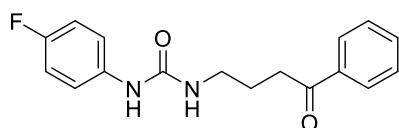
The product was prepared according to the **Gp 4**. White solid; 38% yield, 7.5 mg; m.p. 184-186 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ 8.04-8.02 (d, *J* = 8 Hz, 1H), 7.99-7.97 (d, *J* = 8 Hz, 2H), 7.78 (s, 1H), 7.61-7.58 (d, *J* = 12 Hz, 2H), 7.55-7.53 (d, *J* = 8 Hz, 1H), 7.27-7.24 (t, *J* = 6 Hz, 1H), 7.16-7.13 (t, *J* = 6 Hz, 1H), 6.90-6.86 (t, *J* = 8 Hz, 1H), 3.19-3.14 (q, *J* = 6 Hz, 2H), 3.10-3.06 (t, *J* = 8 Hz, 2H), 1.83-1.76 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d<sub>6</sub>) δ 199.09, 155.23, 138.45, 138.26, 135.77, 132.76, 130.26, 129.27, 128.38, 123.56, 121.91, 112.58, 39.00, 35.78, 24.69; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>16</sub>BrClN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 416.9976; found: 416.9984.

### 1-(4-chlorophenyl)-3-(4-oxo-4-phenylbutyl)urea (2t)



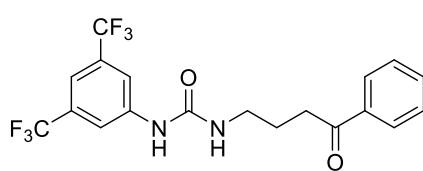
The product was prepared according to the **Gp 4**. White solid; 62% yield, 9.8 mg; m.p. 134-136 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ 8.58 (s, 1H), 7.98-7.96 (s, 2H), 7.65-7.62 (t, *J* = 6 Hz, 1H), 7.55-7.51 (t, *J* = 8 Hz, 1H), 7.42-7.40 (d, *J* = 8 Hz, 2H), 7.26-7.23 (d, *J* = 12 Hz, 2H), 6.29-6.26 (t, *J* = 6 Hz, 1H), 3.18-3.13 (q, *J* = 8 Hz, 2H), 3.09-3.05 (t, *J* = 8 Hz, 2H), 1.82-1.75 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d<sub>6</sub>) δ 200.12, 155.55, 140.03, 137.11, 133.56, 129.17, 128.88, 128.31, 124.81, 119.55, 39.08, 35.74, 24.88; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>ClN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 339.0871; found: 339.0846.

### 1-(4-bromophenyl)-3-(4-oxo-4-phenylbutyl) urea (2u)



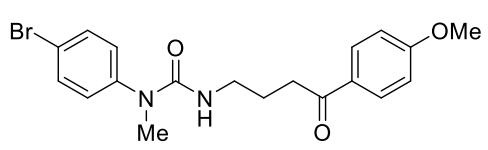
The product was prepared according to the **Gp 4**. White solid; 30% yield, 9.0 mg; m.p. 115-117 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.96-7.94 (d, *J* = 8 Hz, 2H), 7.59-7.56 (t, *J* = 6 Hz, 1H), 7.48-7.44 (t, *J* = 8 Hz, 2H), 7.31-7.27 (m, 2H), 7.00-6.96 (t, *J* = 8 Hz, 2H), 6.90-6.77 (brs, 1H), 5.09-5.07 (t, *J* = 8 Hz, 1H), 3.33-3.28 (q, *J* = 8 Hz, 2H), 3.08-3.05 (t, *J* = 8 Hz, 2H), 2.02-1.95 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 200.53, 160.47, 158.06, 156.08, 136.58, 134.62, 133.40, 128.69, 128.09, 124.87, 122.93, 115.88, 115.66, 40.07, 35.68, 24.31; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -123.50; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>FN<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 323.1166; found: 323.1178.

### 1-(3,5-bis(trifluoromethyl)phenyl)-3-(4-oxo-4-phenylbutyl)urea (2v)



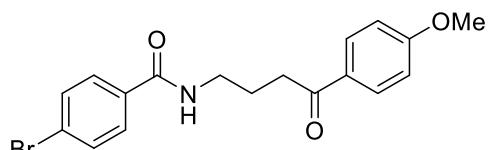
The product was prepared according to the **Gp 4**. White solid; 40% yield, 13.7 mg; m.p. 124-126 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.96-7.88 (m, 4H), 7.59-7.56 (t, *J* = 6 Hz, 1H), 7.50-7.41 (m, 4H), 5.80-5.77 (t, *J* = 8 Hz, 1H), 3.36-3.31 (q, *J* = 8 Hz, 2H), 3.13-3.09 (t, *J* = 8 Hz, 2H), 2.07-2.00 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 201.59, 155.48, 140.98, 136.29, 133.79, 132.49-131.51 (q, *J* = 33 Hz), 128.80, 128.10, 126.23-120.19 (q, *J* = 271 Hz), 118.44, 115.43, 39.92, 35.47, 24.32; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -63.09; HRMS (ESI) *m/z* calcd for C<sub>19</sub>H<sub>16</sub>F<sub>6</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 441.1008; found: 441.1054.

### 1-(4-bromophenyl)-3-(4-(4-methoxyphenyl)-4-oxobutyl)-1-methylurea (2cc)



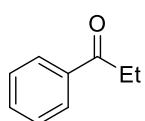
The product was prepared according to the **Gp 4**. White solid; 39% yield, 8.3 mg; m.p. 170-172 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.91-7.89 (d, *J* = 8 Hz, 2H), 7.53-7.50 (d, *J* = 12 Hz, 2H), 7.10-7.08 (d, *J* = 8 Hz, 2H), 6.95-6.92 (d, *J* = 12 Hz, 2H), 4.56-4.53 (t, *J* = 6 Hz, 1H), 3.88 (s, 3H), 3.29-3.24 (q, *J* = 8 Hz, 2H), 3.95-2.92 (t, *J* = 8 Hz, 2H), 1.91-1.84 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 198.50, 163.49, 157.03, 142.43, 133.11, 130.34, 129.72, 129.00, 120.74, 113.70, 55.49, 40.72, 37.11, 35.58, 24.25; HRMS (ESI) *m/z* calcd for C<sub>19</sub>H<sub>21</sub>BrN<sub>2</sub>NaO<sub>3</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 427.0628; found: 427.0554.

#### 4-bromo-N-(4-(4-methoxyphenyl)-4-oxobutyl)benzamide (2bb)



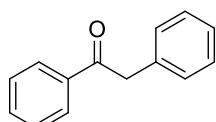
The product was prepared according to the **Gp 4**. White solid; 40% yield, 7.1 mg; m.p. 162-164 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.96-7.93 (d, *J* = 12 Hz, 2H), 7.67-7.65 (d, *J* = 12 Hz, 2H), 7.56-7.54 (d, *J* = 8 Hz, 2H), 6.94-6.92 (m, 3H), 3.87 (s, 3H), 3.55-3.51 (q, *J* = 8 Hz, 2H), 3.13-3.09 (t, *J* = 8 Hz, 2H), 2.13-2.06 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 199.34, 166.44, 163.71, 133.29, 131.68, 130.40, 129.57, 128.54, 125.93, 113.79, 55.51, 40.37, 36.24, 23.14; HRMS (ESI) *m/z* calcd for C<sub>18</sub>H<sub>19</sub>BrNO<sub>3</sub><sup>+</sup> [(M+H)<sup>+</sup>]: 376.0543; found: 376.0556.

#### Propiophenone (4a)



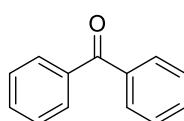
The product was prepared according to the **Gp 5**. Colorless oil; 42% yield, 3.3 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98-7.96 (d, *J* = 8 Hz, 2H), 7.58-7.54 (t, *J* = 8 Hz, 1H), 7.48-7.44 (t, *J* = 8 Hz, 2H), 3.04-2.99 (m, 2H), 1.25-1.21 (t, *J* = 8 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 200.88, 136.89, 132.91, 128.57, 127.99, 31.81, 8.26.

#### 1,2-diphenylethan-1-one (4b)



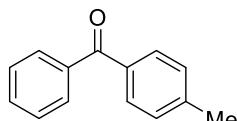
The product was prepared according to the **Gp 5**. White solid; 86% yield, 8.4 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.03-8.01 (d, *J* = 8 Hz, 2H), 7.57-7.53 (t, *J* = 8 Hz, 1H), 7.46-7.44 (m, 2H), 7.35-7.31 (t, *J* = 8 Hz, 2H), 7.28-7.25 (m, 3H), 4.29 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 197.72, 136.58, 134.53, 133.21, 129.49, 128.67, 128.65, 128.51, 126.92, 45.53.

#### benzophenone (4c)



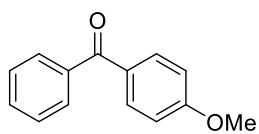
The product was prepared according to the **Gp 5**. White solid; 88% yield, 8 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.82-7.80 (d, *J* = 8 Hz, 4H), 7.61-7.57 (d, *J* = 8 Hz, 2H), 7.50-7.46 (t, *J* = 8 Hz, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 196.82, 137.58, 132.46, 130.10, 128.31.

#### phenyl(p-tolyl)methanone (4d)



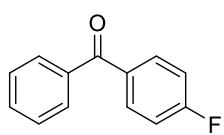
The product was prepared according to the **Gp 5**. White solid; 86% yield, 8.4 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.79-7.77 (d, *J* = 8 Hz, 2H), 7.73-7.71 (d, *J* = 8 Hz, 2H), 7.59-7.56 (t, *J* = 6 Hz, 1H), 7.49-7.45 (t, *J* = 6 Hz, 2H), 7.29-7.27 (d, *J* = 8 Hz, 2H), 2.44 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 196.57, 143.29, 137.93, 134.85, 132.21, 130.35, 129.97, 129.00, 128.24, 21.71.

**(4-methoxyphenyl)(phenyl)methanone (4e)**



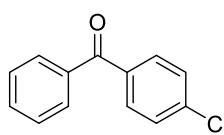
The product was prepared according to the **Gp 5**. White solid; 91% yield, 9.6 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.84-7.82 (d, *J* = 8 Hz, 2H), 7.77-7.75 (d, *J* = 8 Hz, 2H), 7.58-7.55 (t, *J* = 6 Hz, 1H), 7.49-7.45 (t, *J* = 6 Hz, 2H), 6.97-6.95 (d, *J* = 8 Hz, 2H), 3.88 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 195.62, 163.22, 138.26, 132.60, 131.94, 130.12, 129.76, 128.21, 113.56, 55.53.

**(4-fluorophenyl)(phenyl)methanone (4f)**



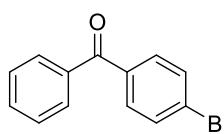
The product was prepared according to the **Gp 5**. White solid; 87% yield, 8.7 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87-7.83 (m, 2H), 7.78-7.76 (d, *J* = 8 Hz, 2H), 7.62-7.58 (t, *J* = 8 Hz, 1H), 7.51-7.47 (t, *J* = 6 Hz, 2H), 7.18-7.14 (d, *J* = 8 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 195.30, 166.65, 164.13, 137.49, 133.81, 132.74, 132.64, 132.50, 129.90, 128.38, 115.58, 115.37; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -107.08.

**(4-chlorophenyl)(phenyl)methanone (4g)**



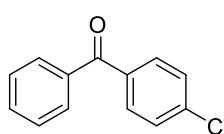
The product was prepared according to the **Gp 5**. White solid; 98% yield, 10.6 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.78-7.76 (d, *J* = 8 Hz, 2H), 7.69-7.67 (d, *J* = 8 Hz, 2H), 7.64-7.58 (m, 3H), 7.51-7.47 (t, *J* = 8 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 195.66, 137.16, 136.30, 132.71, 131.63, 131.59, 129.96, 128.43, 127.54.

**(4-bromophenyl)(phenyl)methanone (4h)**



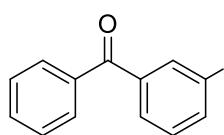
The product was prepared according to the **Gp 5**. White solid; 81% yield, 10.5 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.78-7.75 (m, 4H), 7.62-7.58 (t, *J* = 8 Hz, 1H), 7.51-7.45 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 195.52, 138.91, 137.23, 135.86, 132.67, 131.48, 129.95, 128.65, 128.42.

**phenyl(4-(trifluoromethyl)phenyl)methanone (4i)**



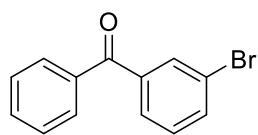
The product was prepared according to the **Gp 5**. White solid; 84% yield, 10.5 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.91-7.89 (d, *J* = 8 Hz, 2H), 7.82-7.80 (d, *J* = 8 Hz, 2H), 7.77-7.75 (t, *J* = 4 Hz, 1H), 7.65-7.62 (d, *J* = 6 Hz, 2H), 7.53-7.49 (t, *J* = 8 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 195.58, 140.71, 136.72, 133.88, 133.56, 133.12, 130.16, 130.13, 128.55, 125.38, 125.03, 122.32; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -63.16.

**phenyl(m-tolyl)methanone (4j)**



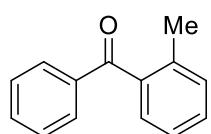
The product was prepared according to the **Gp 5**. White solid; 82% yield, 8.0 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81-7.79 (d, *J* = 8 Hz, 2H), 7.63-7.57 (m, 3H), 7.50-7.47 (t, *J* = 6 Hz, 2H), 7.42-7.37 (m, 2H), 2.43 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 197.02, 138.18, 137.75, 137.62, 133.22, 132.36, 130.48, 130.07, 128.26, 128.10, 127.39, 21.40.

**(3-bromophenyl)(phenyl)methanone (4k)**



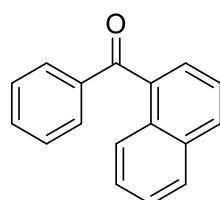
The product was prepared according to the **Gp 5**. White solid; 72% yield, 9.4 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.95-7.91 (m, 1H), 7.80-7.78 (d, *J* = 8 Hz, 2H), 7.73-7.71 (d, *J* = 8 Hz, 2H), 7.64-7.60 (t, *J* = 8 Hz, 1H), 7.52-7.48 (t, *J* = 8 Hz, 2H), 7.39-7.35 (t, *J* = 8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 195.19, 139.46, 136.90, 135.30, 132.88, 132.81, 130.05, 129.90, 128.58, 128.49, 122.59.

#### **phenyl(o-tolyl)methanone (4l)**



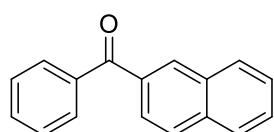
The product was prepared according to the **Gp 5**. White solid; 76% yield, 7.4 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81-7.80 (d, *J* = 4 Hz, 2H), 7.60-7.57 (t, *J* = 6 Hz, 1H), 7.48-7.44 (d, *J* = 8 Hz, 2H), 7.41-7.37 (t, *J* = 8 Hz, 1H), 7.32-7.25 (m, 3H), 2.33 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 198.68, 138.60, 137.72, 136.77, 133.16, 131.01, 130.26, 130.15, 128.54, 128.47, 125.21, 20.02.

#### **naphthalen-1-yl(phenyl)methanone (4m)**



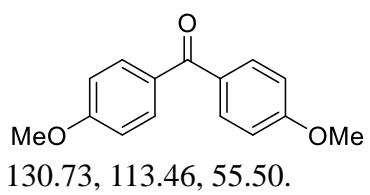
The product was prepared according to the **Gp 5**. White solid; 32% yield, 3.7 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.11-8.09 (d, *J* = 8 Hz, 1H), 8.02-8.00 (d, *J* = 8 Hz, 1H), 7.94-7.92 (d, *J* = 8 Hz, 1H), 7.88-7.86 (d, *J* = 8 Hz, 2H), 7.62-7.45 (m, 7H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 198.07, 138.30, 136.33, 133.26, 131.29, 130.95, 130.44, 128.46, 128.42, 127.81, 127.28, 126.48, 125.69, 125.03, 124.35.

#### **naphthalen-2-yl(phenyl)methanone (4n)**



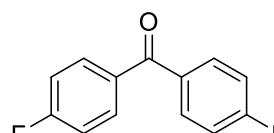
The product was prepared according to the **Gp 5**. White solid; 76% yield, 8.8 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.27 (s, 1H), 7.95-7.91 (m, 4H), 7.88-7.85 (d, *J* = 6 Hz, 2H), 7.64-7.60 (m, 2H), 7.57-7.50 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 196.82, 137.89, 135.28, 134.81, 132.64, 132.42, 132.25, 131.92, 130.13, 129.44, 128.37, 128.33, 127.85, 126.83, 125.81.

#### **bis(4-methoxyphenyl)methanone (4o)**



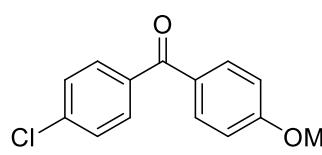
The product was prepared according to the **Gp 5**. White solid; 69% yield, 8.3 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.80-7.78 (d, *J* = 4 Hz, 4H), 6.98-6.95 (d, *J* = 12 Hz, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 194.50, 162.82, 132.26, 130.73, 113.46, 55.50.

#### **bis(4-fluorophenyl)methanone (4p)**



The product was prepared according to the **Gp 5**. White solid; 92% yield, 10 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.83-7.80 (m, 4H), 7.19-7.15 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 193.82, 166.66, 164.13, 133.71, 132.55, 132.46, 115.68, 115.46; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -105.74.

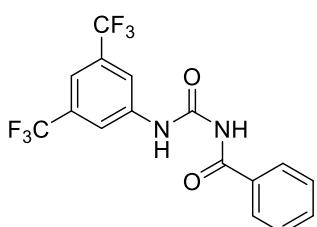
#### **(4-chlorophenyl)(4-methoxyphenyl)methanone (4q)**



The product was prepared according to the **Gp 5**. White solid; 79% yield, 9.7 mg; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81-7.79 (d, *J* = 8 Hz, 2H), 7.72-7.70 (d, *J* = 8 Hz, 2H), 7.46-7.44 (d, *J* = 8 Hz, 2H), 6.98-6.96 (d, *J* = 8 Hz, 2H),

3.89 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  194.30, 163.39, 138.28, 136.55, 132.47, 131.17, 129.79, 128.53, 113.69, 55.55.

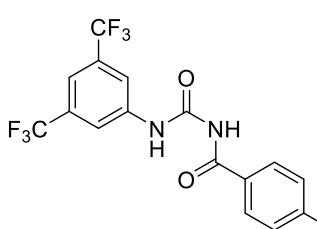
**N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)benzamide (6a)**



The product was prepared according to the **Gp 6**. White solid; 80% yield, 14.3 mg; m.p. 177-179 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  11.54 (s, 1H), 9.97 (s, 1H), 8.07-8.02 (m, 4H), 7.68-7.65 (m, 2H), 7.55-7.51 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.12, 152.21, 138.75, 133.97, 132.98-131.98 (q,  $J = 33$  Hz), 131.43, 129.03, 127.90, 127.12, 124.41, 121.70, 120.01, 117.71;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.91;

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{10}\text{F}_6\text{N}_2\text{NaO}_2^+ [(\text{M}+\text{Na})^+]$ : 399.0539; found: 399.0579.

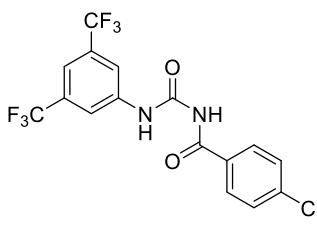
**N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-4-fluorobenzamide (6b)**



The product was prepared according to the **Gp 6**. White solid; 72% yield, 13 mg; m.p. 210-212 °C;  $^1\text{H}$  NMR (400 MHz, Acetone-d6)  $\delta$  11.36 (s, 1H), 10.22 (s, 1H), 8.38 (s, 2H), 8.24-8.20 (m, 2H), 7.76 (s, 1H), 7.37-7.33 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, Acetone-d6)  $\delta$  167.85, 167.01, 164.50, 151.25, 140.11, 132.23, 131.90, 131.57, 131.24, 131.21, 131.11, 128.66, 124.80, 122.10, 120.03, 116.62,

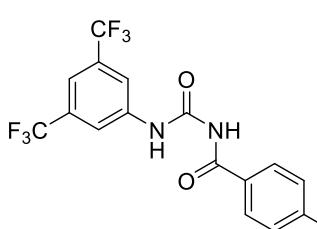
115.86, 115.64;  $^{19}\text{F}$  NMR (376 MHz, Acetone-d6)  $\delta$  -63.93, -107.08; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_9\text{F}_7\text{N}_2\text{NaO}_2^+ [(\text{M}+\text{Na})^+]$ : 417.0444; found: 417.0422.

**N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-4-bromo-N-methylbenzamide (6c)**



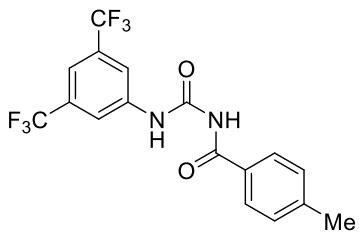
The product was prepared according to the **Gp 6**. White solid; 75% yield, 15.4 mg; m.p. 198-200 °C;  $^1\text{H}$  NMR (400 MHz, Acetone-d6)  $\delta$  11.32 (s, 1H), 10.28 (s, 1H), 8.38 (s, 2H), 8.15-8.13 (d,  $J = 8$  Hz, 2H), 7.76 (s, 1H), 7.63-7.61 (d,  $J = 8$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz, Acetone-d6)  $\delta$  167.97, 151.16, 140.08, 139.08, 132.23-131.24 (q,  $J = 33$  Hz), 130.97, 130.02, 128.93, 127.50-119.39 (q,  $J = 271$  Hz), 120.05, 116.62;  $^{19}\text{F}$  NMR (376 MHz, Acetone-d6)  $\delta$  -63.52; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_9\text{ClF}_6\text{N}_2\text{NaO}_2^+ [(\text{M}+\text{Na})^+]$ : 433.0149; found: 433.0130.

**N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-4-bromobenzamide (6d)**



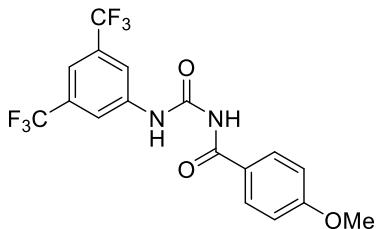
The product was prepared according to the **Gp 6**. White solid; 67% yield, 12.9 mg; m.p. 215-217 °C;  $^1\text{H}$  NMR (400 MHz, Acetone-d6)  $\delta$  11.31 (s, 1H), 10.27 (s, 2H), 8.39 (s, 2H), 8.07-8.05 (d,  $J = 8$  Hz, 2H), 7.80-7.77 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz, Acetone-d6)  $\delta$  168.13, 151.12, 140.09, 132.23-131.24 (q,  $J = 33$  Hz), 131.96, 131.42, 130.11, 127.70, 127.50-119.39 (q,  $J = 271$  Hz), 120.01, 116.62;  $^{19}\text{F}$  NMR (376 MHz, Acetone-d6)  $\delta$  -63.52; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_9\text{BrF}_6\text{N}_2\text{NaO}_2^+ [(\text{M}+\text{Na})^+]$ : 476.9644; found: 476.9622.

**N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-4-methylbenzamide (6e)**



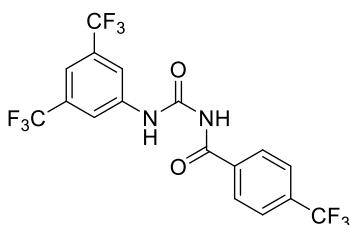
The product was prepared according to the **Gp 6**. White solid; 71% yield, 9.9 mg; m.p. 187-189 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 11.23 (s, 1H), 11.17 (s, 1H), 8.36 (s, 2H), 7.97-7.95 (d, *J* = 8 Hz, 2H), 7.80 (s, 1H), 7.38-7.36 (d, *J* = 8 Hz, 2H), 2.40 (s, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 168.78, 152.09, 144.16, 140.34, 131.71-130.73 (q, *J* = 33 Hz), 130.09, 129.65, 129.12, 128.90, 127.73-119.60 (q, *J* = 271 Hz), 125.02, 122.31, 120.61, 116.95, 21.57; <sup>19</sup>F NMR (376 MHz, DMSO-d6) δ -61.54; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>12</sub>F<sub>6</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 413.0695; found: 413.0641.

#### N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-4-methoxybenzamide (6f)



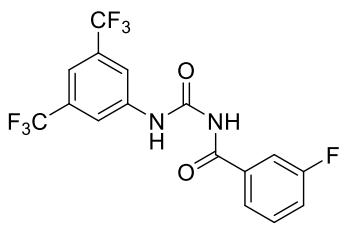
The product was prepared according to the **Gp 6**. White solid; 61% yield, 12.4 mg; m.p. 186-188 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 11.50 (s, 1H), 10.02 (s, 1H), 8.38 (s, 2H), 8.15-8.13 (d, *J* = 8 Hz, 2H), 7.75 (s, 1H), 7.11-7.09 (d, *J* = 8 Hz, 2H), 3.92 (s, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 168.13, 163.99, 151.48, 140.28, 132.21-131.22 (q, *J* = 33 Hz), 130.38, 129.09, 127.53-119.42 (q, *J* = 271 Hz), 124.82, 124.01, 122.12, 119.88, 116.43, 114.01, 55.16; <sup>19</sup>F NMR (376 MHz, DMSO-d6) δ -63.52; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>12</sub>F<sub>6</sub>N<sub>2</sub>NaO<sub>3</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 429.0644; found: 429.0629.

#### N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-4-(trifluoromethyl)benzamide (6g)



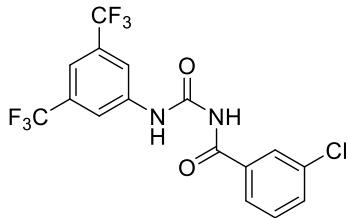
The product was prepared according to the **Gp 6**. White solid; 78% yield, 17.3 mg; m.p. 208-210 °C; <sup>1</sup>H NMR (400 MHz, Acetone-d6) δ 11.14 (s, 1H), 10.30 (s, 1H), 8.27 (s, 2H), 8.20-8.18 (d, *J* = 8 Hz, 2H), 7.82-7.80 (d, *J* = 8 Hz, 2H), 7.65 (s, 1H); <sup>13</sup>C NMR (100 MHz, Acetone-d6) δ 167.97, 151.02, 140.02, 136.09, 133.95, 133.63, 132.23-131.24 (q, *J* = 33 Hz), 129.10, 127.49-119.39 (q, *J* = 271 Hz), 125.68, 125.20, 122.50, 120.08, 116.80; <sup>19</sup>F NMR (376 MHz, Acetone-d6) δ -63.51, 63.66; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>9</sub>F<sub>9</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 467.0413; found: 467.0496.

#### N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-3-fluorobenzamide (6h)



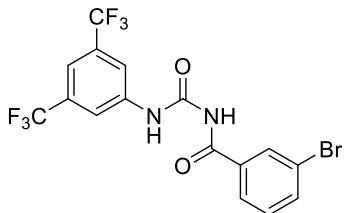
The product was prepared according to the **Gp 6**. White solid; 73% yield, 14.4 mg; m.p. 170-172 °C; <sup>1</sup>H NMR (400 MHz, Acetone-d6) δ 11.29 (s, 1H), 10.27 (s, 1H), 8.38 (s, 2H), 7.99-7.97 (d, *J* = 8 Hz, 2H), 7.88-7.85 (d, *J* = 8 Hz, 2H), 7.77 (s, 1H), 7.67-7.62 (m, 1H), 7.50-7.46 (t, *J* = 8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, Acetone-d6) δ 167.65, 163.79, 161.34, 151.07, 140.05, 134.57, 132.24-131.25 (q, *J* = 33 Hz), 130.86, 127.50-119.39 (q, *J* = 271 Hz), 124.24, 120.29, 120.07, 116.69, 115.15, 114.91; <sup>19</sup>F NMR (376 MHz, Acetone-d6) δ -63.92, -112.27; HRMS (ESI) *m/z* calcd for C<sub>16</sub>H<sub>9</sub>F<sub>7</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 417.0444; found: 417.0403.

#### N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-3-chlorobenzamide (6i)



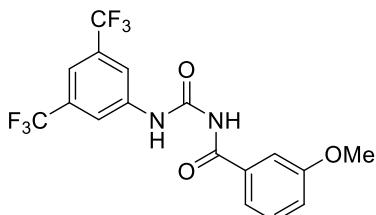
The product was prepared according to the **Gp 6**. White solid; 74% yield, 15.2 mg; m.p. 160-162 °C; <sup>1</sup>H NMR (400 MHz, Acetone-d6) δ 11.27 (s, 1H), 10.30 (s, 1H), 8.38 (s, 2H), 8.12-8.11 (t, *J* = 2 Hz, 2H), 8.08-8.06 (m, 1H), 7.77 (s, 1H), 7.74-7.71 (m, 1H), 7.64-7.60 (t, *J* = 8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, Acetone-d6) δ 167.68, 151.04, 140.04, 134.28, 134.25, 133.11, 132.24-131.25 (q, *J* = 33 Hz), 130.53, 128.11, 127.50-119.39 (q, *J* = 270 Hz), 126.72, 124.79, 122.09, 120.04, 116.70; <sup>19</sup>F NMR (376 MHz, Acetone-d6) δ -63.52; HRMS (ESI) *m/z* calcd for C<sub>16</sub>H<sub>9</sub>ClF<sub>6</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 433.0149; found: 433.0111.

#### N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-4-bromo-N-methylbenzamide (6j)



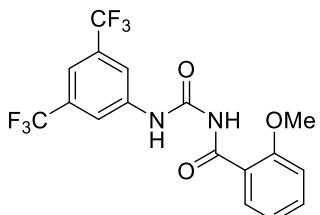
The product was prepared according to the **Gp 6**. White solid; 84% yield, 19.1 mg; m.p. 168-170 °C; <sup>1</sup>H NMR (400 MHz, Acetone-d6) δ 11.27 (s, 1H), 10.31 (s, 1H), 8.38 (s, 2H), 8.26-8.25 (t, *J* = 2 Hz, 2H), 8.12-8.10 (d, *J* = 8 Hz, 1H), 7.88-7.86 (d, *J* = 8 Hz, 1H), 7.76 (s, 1H), 7.57-7.53 (t, *J* = 8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, Acetone-d6) δ 167.59, 151.03, 140.04, 136.07, 134.43, 132.24-131.24 (q, *J* = 33 Hz), 131.02, 130.74, 127.50-119.39 (q, *J* = 270 Hz), 127.14, 122.18, 120.06, 116.66; <sup>19</sup>F NMR (376 MHz, Acetone-d6) δ -63.52; HRMS (ESI) *m/z* calcd for C<sub>16</sub>H<sub>9</sub>BrF<sub>6</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 476.9644; found: 476.9580.

#### N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-3-methoxybenzamide (6k)



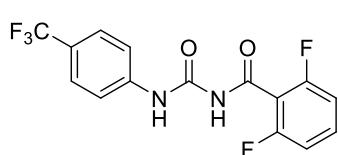
The product was prepared according to the **Gp 6**. White solid; 80% yield, 16.2 mg; m.p. 152-154 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 11.46 (s, 1H), 9.78-9.72 (m, 1H), 8.06 (s, 2H), 7.64 (s, 1H), 7.58-7.56 (d, *J* = 2 Hz, 1H), 7.50 (s, 1H), 7.44-7.40 (t, *J* = 8 Hz, 1H), 7.19-7.17 (d, *J* = 2 Hz, 1H), 3.84 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 168.83, 160.10, 151.98, 138.72, 132.76, 132.97-131.97 (q, *J* = 33 Hz), 130.08, 127.12-118.98 (q, *J* = 270 Hz), 120.00, 119.57, 117.69, 113.19; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.95; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>12</sub>F<sub>6</sub>N<sub>2</sub>NaO<sub>3</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 429.0644; found: 429.0598.

#### N-((3,5-bis(trifluoromethyl)phenyl)carbamoyl)-2-methoxybenzamide (6l)



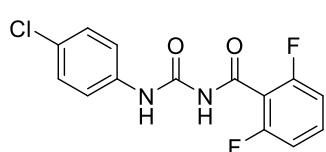
The product was prepared according to the **Gp 6**. White solid; 55% yield, 7.1 mg; m.p. 154-156 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 11.36 (s, 1H), 10.14 (s, 1H), 8.23-8.20 (d, *J* = 12 Hz, 1H), 8.10 (s, 2H), 7.64-7.59 (m, 2H), 7.19-7.15 (t, *J* = 8 Hz, 1H), 7.09-7.07 (d, *J* = 2 Hz, 1H), 4.09 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.51, 158.04, 151.05, 139.13, 135.57, 132.85-131.85 (q, *J* = 33 Hz), 132.70, 127.21-119.07 (q, *J* = 271 Hz), 121.92, 119.81, 118.74, 117.29, 111.89; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -63.01; HRMS (ESI) *m/z* calcd for C<sub>17</sub>H<sub>12</sub>F<sub>6</sub>N<sub>2</sub>NaO<sub>3</sub><sup>+</sup> [(M+Na)<sup>+</sup>]: 429.0644; found: 429.0599.

#### 2,6-difluoro-N-((4-(trifluoromethyl)phenyl)carbamoyl)benzamide (6m)



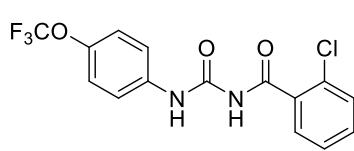
The product was prepared according to the **Gp 6**. White solid; 75% yield, 11.2 mg; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 11.54 (s, 1H), 10.42 (s, 1H), 7.82-7.80 (d, *J* = 12 Hz, 2H), 7.73-7.71 (d, *J* = 12 Hz, 2H), 7.68-7.61 (m, 1H), 7.29-7.25 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 162.57, 160.33, 157.91, 150.45, 141.56, 133.75, 130.08, 128.78, 126.59, 126.09, 124.92, 124.60, 124.29, 123.97, 120.69, 120.48, 112.73; <sup>19</sup>F NMR (376 MHz, DMSO-d6) δ -59.07, -114.19.

#### **N-((4-chlorophenyl)carbamoyl)-2,6-difluorobenzamide (6n)**



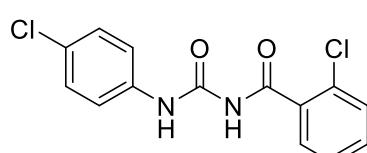
The product was prepared according to the **Gp 6**. White solid; 64% yield, 5.6 mg; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 11.46 (s, 1H), 10.23 (s, 1H), 7.67-7.60 (m, 3H), 7.42-7.40 (d, *J* = 8 Hz, 2H), 7.28-7.24 (m, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 162.52, 160.32, 157.90, 150.41, 136.80, 133.69, 129.24, 128.17, 122.22, 112.48; <sup>19</sup>F NMR (376 MHz, DMSO-d6) δ -115.10.

#### **2-chloro-N-((4-(trifluoromethoxy)phenyl)carbamoyl)benzamide (6o)**



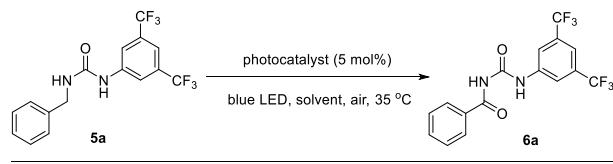
The product was prepared according to the **Gp 6**. White solid; 56% yield, 10 mg; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 11.31 (s, 1H), 10.52 (s, 1H), 7.73-7.71 (d, *J* = 8 Hz, 2H), 7.64-7.62 (d, *J* = 8 Hz, 1H), 7.59-7.55 (m, 2H), 7.49-7.45 (t, *J* = 8 Hz, 1H), 7.38-7.36 (d, *J* = 8 Hz, 2H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 169.00, 150.96, 144.47, 137.18, 135.02, 132.50, 130.23, 130.18, 129.56, 127.69, 122.27, 121.90; <sup>19</sup>F NMR (376 MHz, DMSO-d6) δ -57.13.

#### **2-chloro-N-((2-chlorophenyl)carbamoyl)benzamide (6p)**

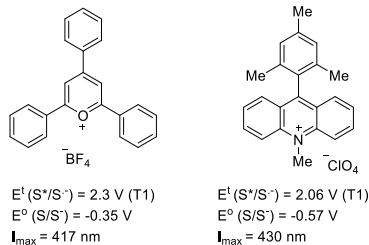


The product was prepared according to the **Gp 6**. White solid; 62% yield, 6.8 mg; <sup>1</sup>H NMR (400 MHz, DMSO-d6) δ 11.27 (s, 1H), 10.47 (s, 1H), 7.64-7.61 (m, 3H), 7.59-7.52 (m, 2H), 7.48-7.40 (m, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-d6) δ 173.74, 155.62, 141.69, 139.80, 137.23, 134.98, 134.93, 134.30, 134.05, 133.73, 132.75, 132.43, 127.64, 126.75.

**Table S1. Optimization of the reaction conditions.\***

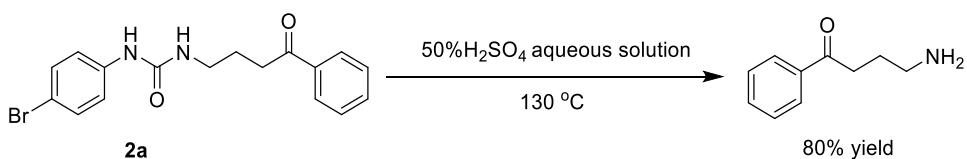
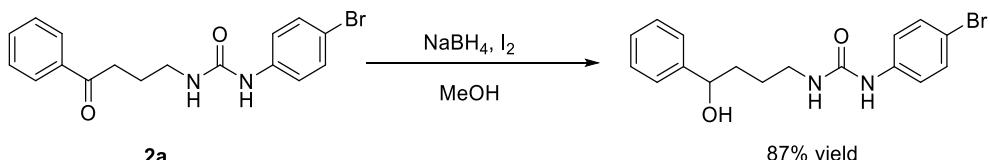


Entry	photocatalyst	solvent	yield
1	2,4,6-triphenylpyrylium tetrafluoroborate	acetone	19%
2	2,4,6-triphenylpyrylium tetrafluoroborate	CHCl <sub>3</sub>	42%
3	Mes-AcrClO <sub>4</sub>	CHCl <sub>3</sub>	80%
4	[Ru(bpy) <sub>3</sub> Cl <sub>2</sub> ]·6H <sub>2</sub> O	CHCl <sub>3</sub>	NR
5	[Ir(ppy) <sub>2</sub> (dtbbpy)]PF <sub>6</sub>	CHCl <sub>3</sub>	NR



\*The reactions were carried out with **5a** (0.05 mmol) in the presence of a catalyst (5 mol%) in solvent (1 mL). Yield of **6a** are isolated yields. NR: no reaction

### Scheme S1. Reduction and Hydrolysis Experiments of **2a**



The procedure of the reduction reaction:

To a solution of **2a** (0.2 mmol) in methanol (10 mL), iodine was added into the mixture (0.05 equiv.). After that, the mixture was cooled to 0 °C, and then the sodium borohydride was slowly added into the solution. The mixture was stirred at room temperature until the completion of the reaction determined by TLC analysis. The crude product was purified by flash chromatography on silica gel affording to the compound.

#### **1-(4-bromophenyl)-3-(4-hydroxy-4-phenylbutyl)urea**

White solid; 87% yield, 364 mg; m.p. 156-158 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.55 (s, 1H), 7.38-7.36 (m, 4H), 7.32-7.31 (m, 4H), 7.23-7.19 (m, 1H), 6.19-6.16 (t, *J* = 6 Hz, 1H), 5.20-5.19 (d, *J* = 4 Hz, 1H), 4.56-4.52 (m, 1H), 3.10-3.05 (q, *J* = 8 Hz, 1H),

1.63-1.47 (s, 3H), 1.41-1.34 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.42, 146.84, 140.48, 131.77, 128.39, 127.05, 126.21, 119.89, 112.57, 72.48, 39.46, 37.20, 26.80; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{20}\text{BrN}_2\text{O}_2^+ [(\text{M}+\text{H})^+]$ : 363.0703; found: 363.0679.

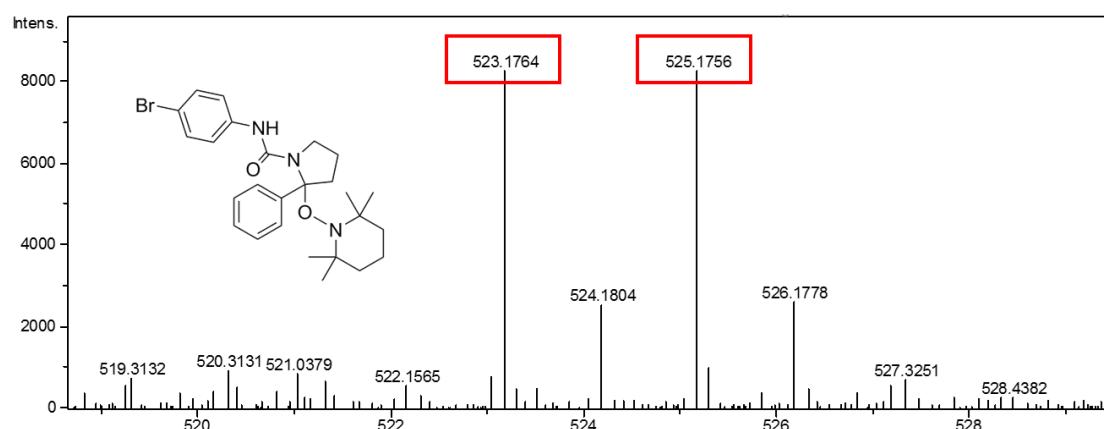
The procedure of the hydrolysis reaction:

**2a** (0.1mol) were weighed into an 50 mL flask, and then 50% aqueous sulfuric acid (10 mL) was added into the flask. The solution was heated to 140 °C stirring for 2 h.<sup>4</sup> After the completion of the reaction, the mixture was cooled to room temperature and neutralized with saturated sodium bicarbonate solution. Then the solution was extracted with ethyl acetate (20 mL, two times) and purified by flash chromatography.

#### 4-amino-1-phenylbutan-1-one

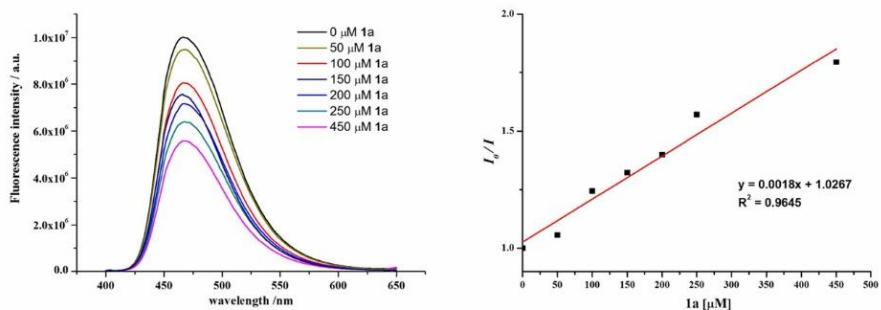
Yellow oil; 80% yield, 13.1 mg;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  7.84-7.82(d,  $J = 8$  Hz, 2H), 7.46-7.42 (m, 3H), 3.96-3.92 (t,  $J = 8$  Hz, 2H), 2.93-2.89 (t,  $J = 8$  Hz, 2H), 1.98-1.90 (m, 2H), 1.31-1.19 (brs, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  172.70, 134.86, 130.68, 128.88, 127.91, 61.36, 34.97, 22.75., 23.14; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{10}\text{H}_{14}\text{NO}^+ [(\text{M}+\text{H})^+]$ : 164.1070; found: 164.1077.

**Figure S1. Determination of the reaction intermediate.**



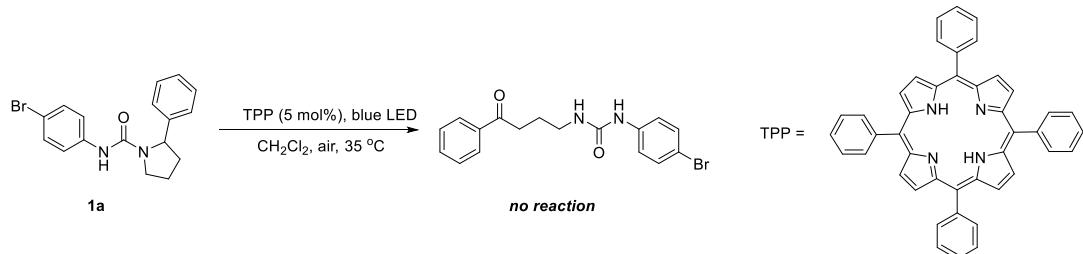
**N-(4-bromophenyl)-2-phenylpyrrolidine-1-carboxamide (**2a**, 17.3 mg, 0.05 mmol) and acetone (2.0 mL) were added to an oven-dried reaction tube with magnetic stirring bar, then the 2,2,6,6-tetramethylpiperidinooxy (TEMPO, 2equiv., 0.1mmol, 15.6mg) and 2,4,6-Triphenylpyrylium fluoroborate (1 equiv., 20 mg) was added. The tube was exposed to blue LED (420–425 nm, 10 W) irradiation at room tem with stirring for 1h under the air, after that the reaction mixture was diluted with methanol and determined by high resolution mass spectrometer.**

**Figure S2. Fluorescence quenching experiments.**



Fluorescence quenching studies were conducted using FLS-920 Edinburgh Fluorescence Spectrometer. The photocatalyst 2,4,6-Triphenylpyrylium tetrafluoroborate (5.0  $\mu$ M in acetone) and increasing concentrations of quencher **1a** (N-(4-bromophenyl)-2-phenylpyrrolidine-1-carboxamide) (50, 100, 150, 200, 250, 450  $\mu$ M in acetone) were added into a screwtop 1.0 cm quartz cuvette. Each sample was irradiated at 360 nm and the emission spectrum was recorded. Plots of intensity of emission (462 nm) vs concentration of quencher are shown according to the Stern-Volmer equation.<sup>6</sup>

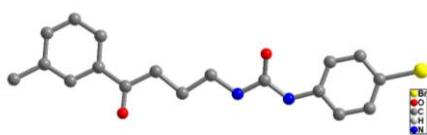
**Figure S3. Control experiments for  ${}^1\text{O}_2$  sensitization.**



The reaction was carried out with **1a** (0.05 mmol), TPP (5 mol%) in the dichloromethane (2 mL) at 35 °C under 10 W blue LED irradiation.

TPP/DCM system, which is a well-known singlet oxygen generation system, was also tested in the model reaction, but no desired product **2a** was obtained after stirring for 8 h.

## Crystal data and structure refinement for 2j (CCDC 1833871)



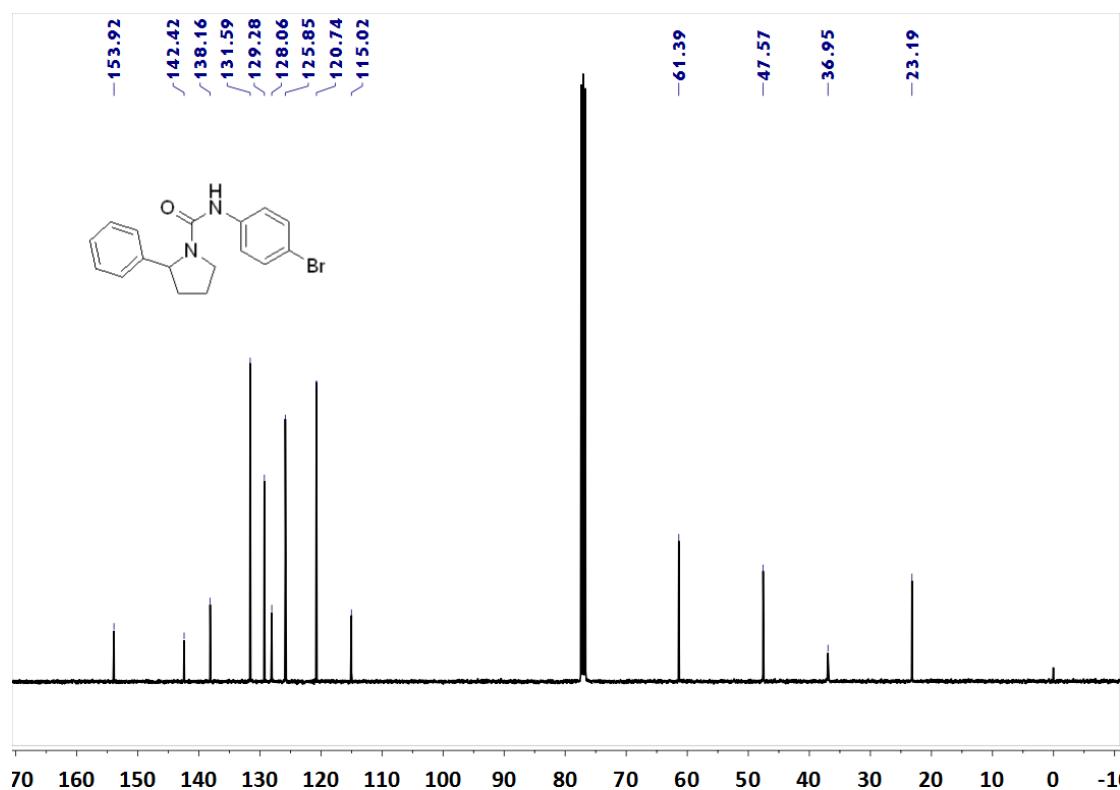
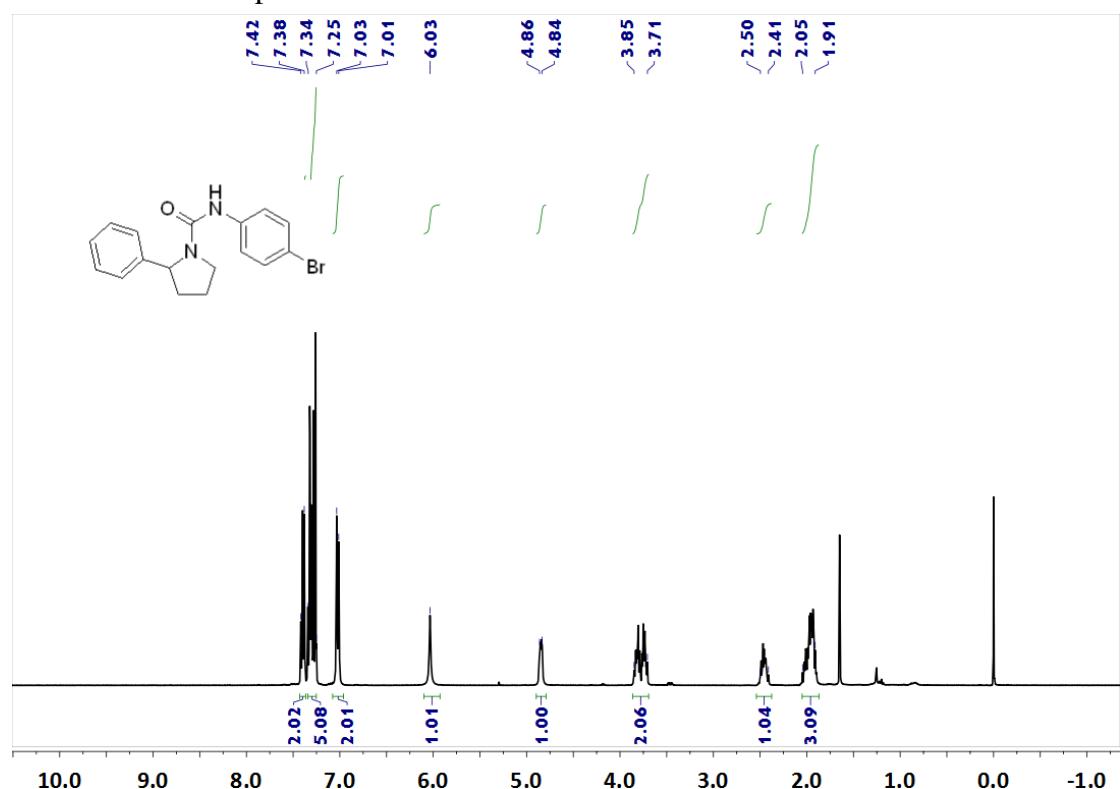
Empirical formula	C36H38Br2N4O4
Formula weight	750.52
Temperature	173(2) K
Wavelength	1.54184 Å
Crystal system, space group	Triclinic, <i>P-1</i>
Unit cell dimensions	a = 9.5585(4) Å    alpha = 84.171(4) deg. b = 11.0081(5) Å    beta = 85.069(4) deg. c = 16.9501(8) Å    gamma = 70.438(4) deg.
Volume	1669.30(14)
Z, Calculated density	2, 1.493 Mg/m <sup>3</sup>
Absorption coefficient	3.447 mm <sup>-1</sup>
F(000)	768
Theta range for data collection	4.275 to 71.198 deg.
Limiting indices	-11<= h<= 6, -13<= k<= 10, -20<= l<= 20
Reflections collected / unique	10598 / 6311 [R(int) = 0.0172]
Completeness to theta = 67.684	99.5 %
Absorption correction	multi-scan
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	6311 / 0 / 429
Goodness-of-fit on F <sup>2</sup>	1.058
Final R indices [I>2sigma(I)]	R1 = 0.0409, wR2 = 0.1141
R indices (all data)	R1 = 0.0470, wR2 = 0.1200
Largest diff. peak and hole	0.308 and -0.808 e.Å <sup>-3</sup>

## References

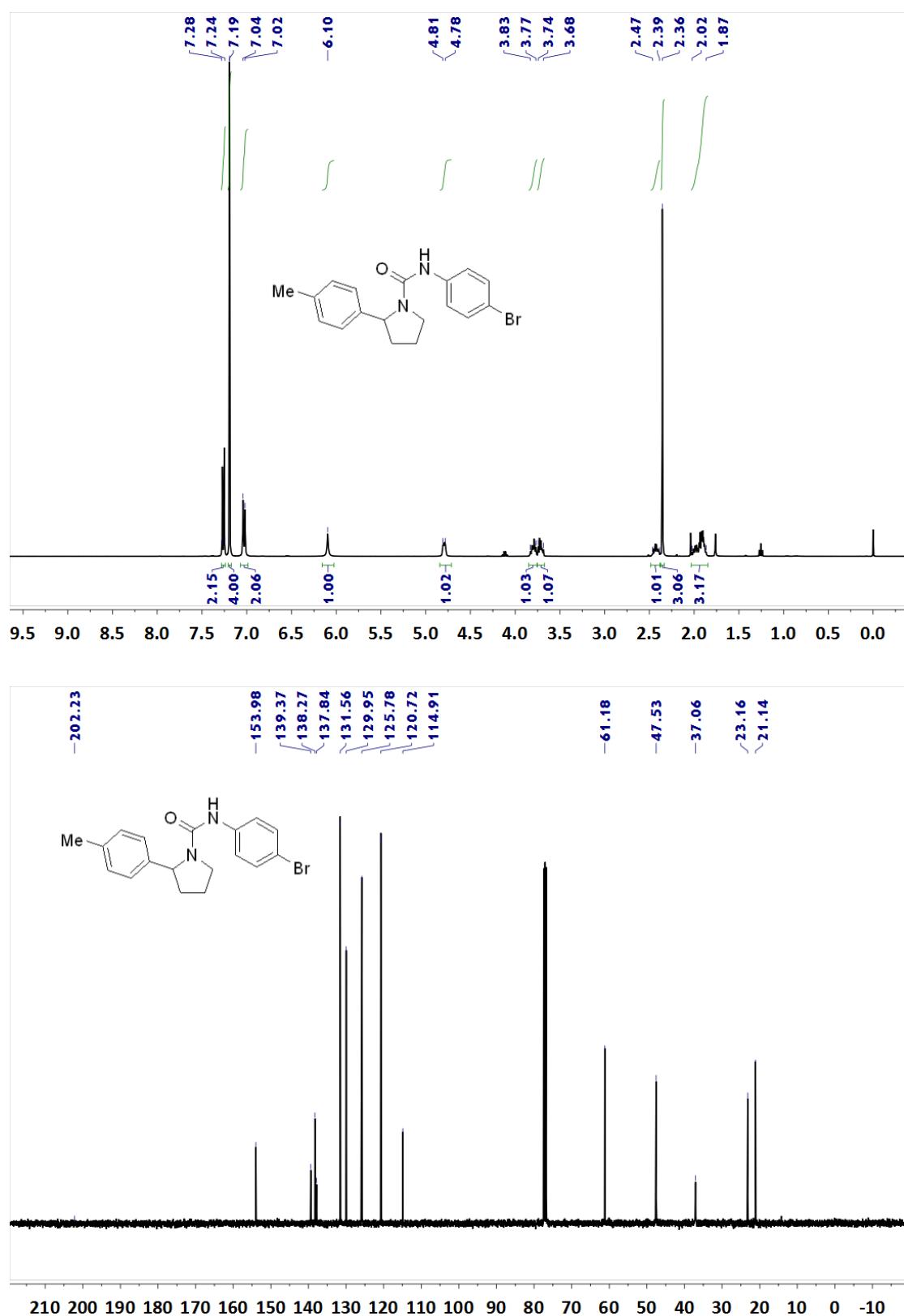
- (1) W. L. F. Armarego, C. L. L. Chai, *Purification of Laboratory Chemicals*, Elsevier, 6<sup>th</sup> Ed, **2009**.
- (2) W.-Q. Wu, P.-H. Li, S. Yang, L. Wang, *Chem. Commun.* 2017, **53**, 8482-8485.
- (3) Z.-G. Zhao, Z.-Y. Peng, Y.-L. Zhao, H. Liu, C.-N. Li, J.-F. Zhao, *J. Org. Chem.* 2017, **82**, 11848-11853.
- (4) X. Chen, C.-J. Zhu, F. Guo, X.-W. Qiu, Y.-C. Yang, S.-Z. Zhang, M.-L. He, C.-J. Jing, J. Li, B. Ma, *J. Med. Chem.* 2010, **53**, 8330–8344.
- (5) A. Sagadevan, P. V. Charpe, A. Ragupathi, K.-C. Hwang, *J. Am. Chem. Soc.* 2017, **139**, 2896–2899.
- (6) G.-Q. Xu, J.-T. Xu, Z.-T. Feng, H. Liang, Z.-Y. Wang, Y. Qin, P.-F. Xu, *Angew. Chem. Int. Ed.* 2018, **57**, 5110–5114.

## <sup>1</sup>H, <sup>13</sup>C and <sup>19</sup>F-NMR spectra

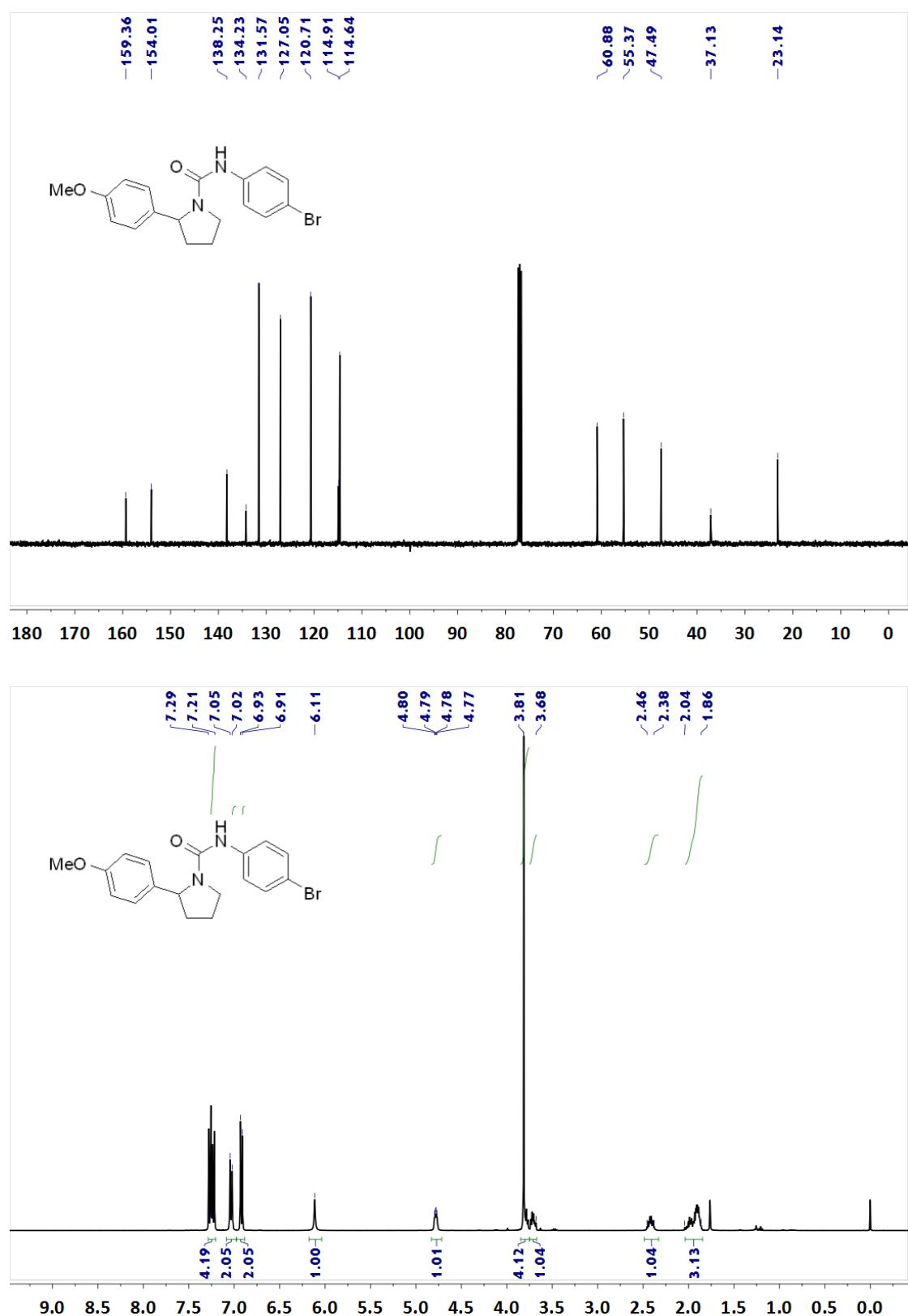
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1a**.



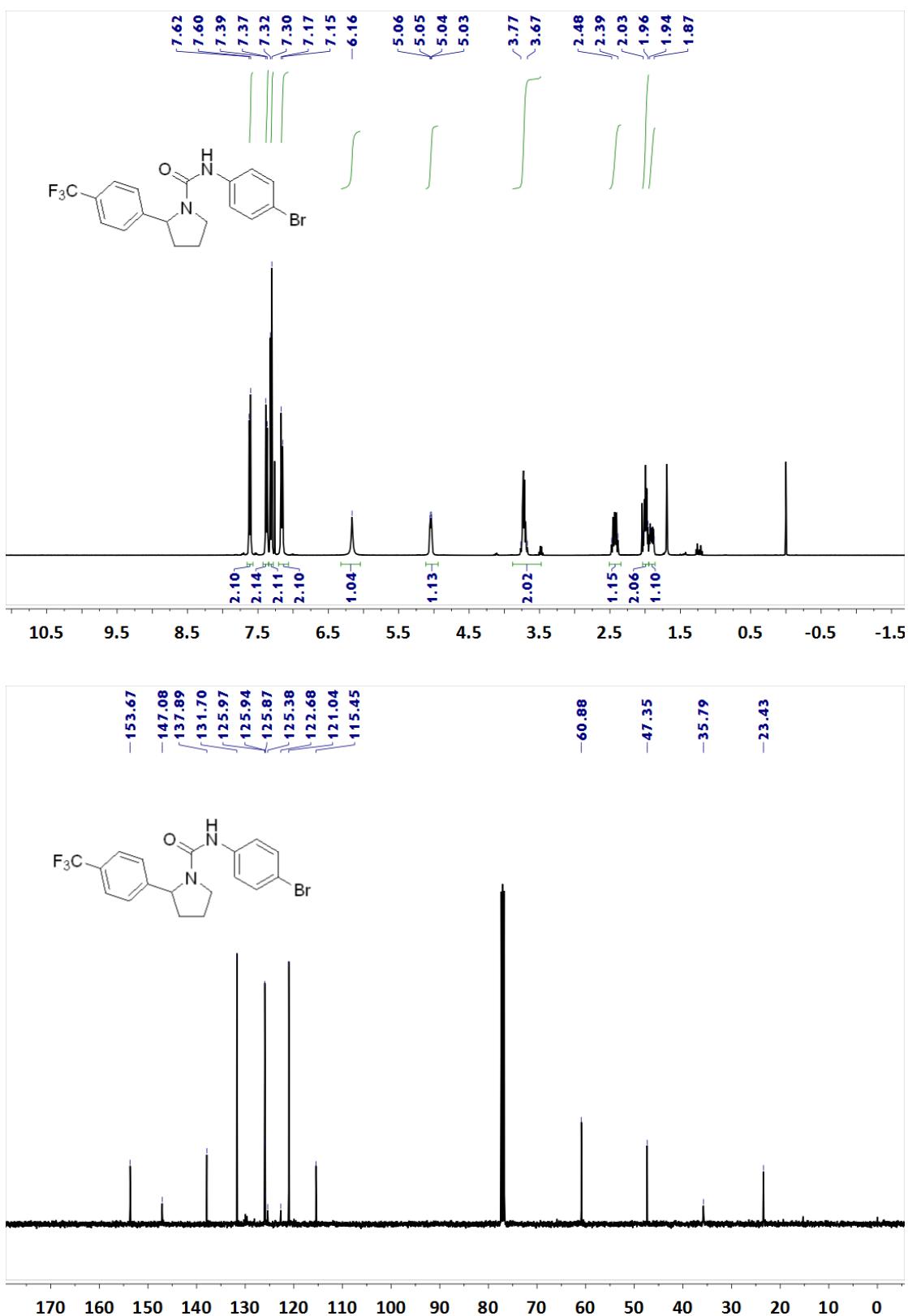
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1b**.

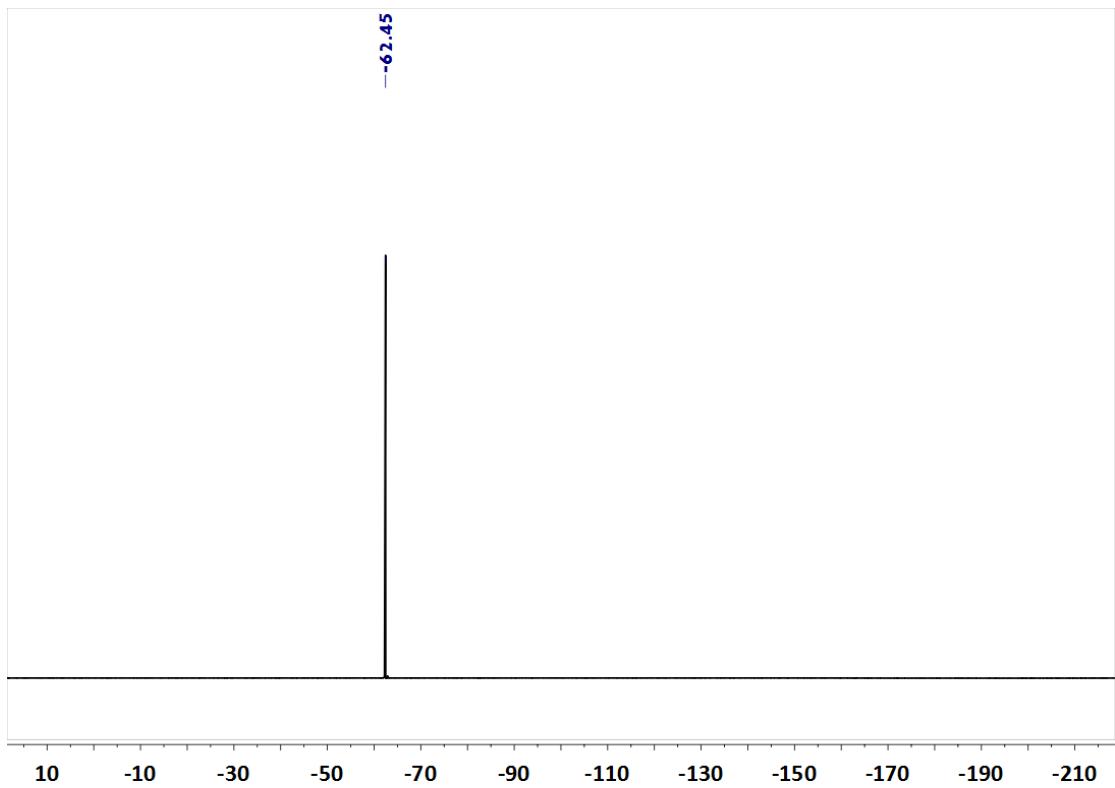


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1c**.

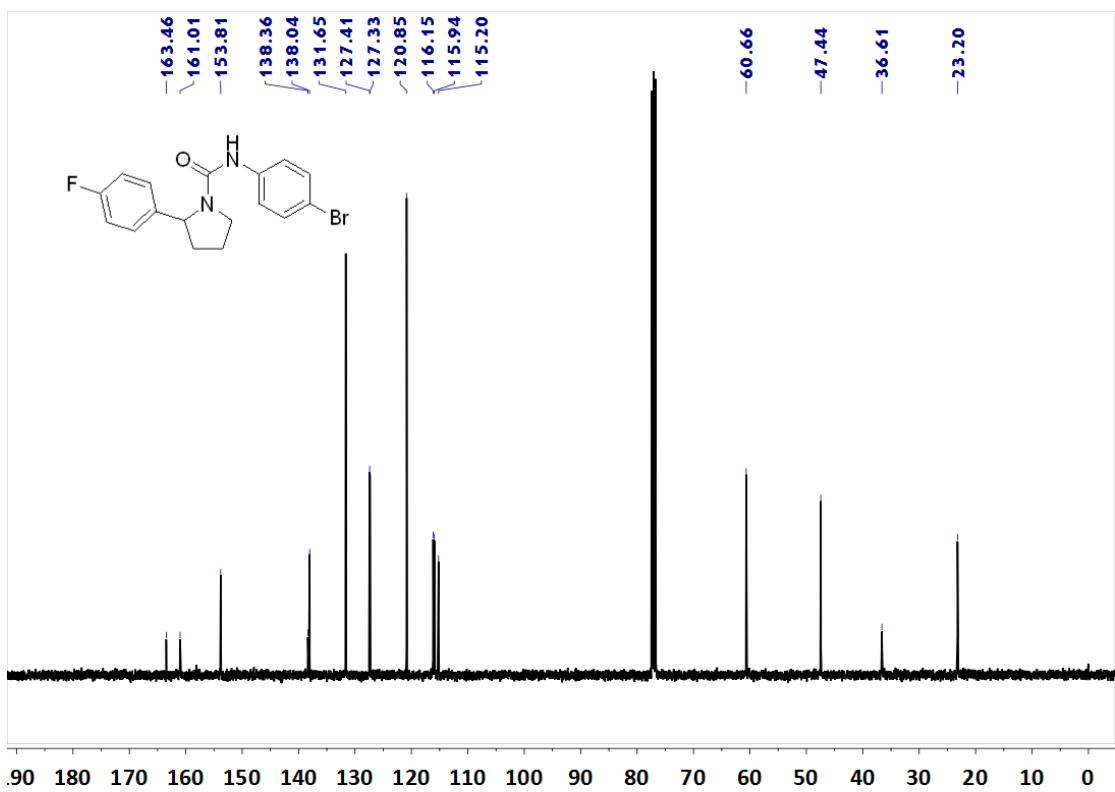


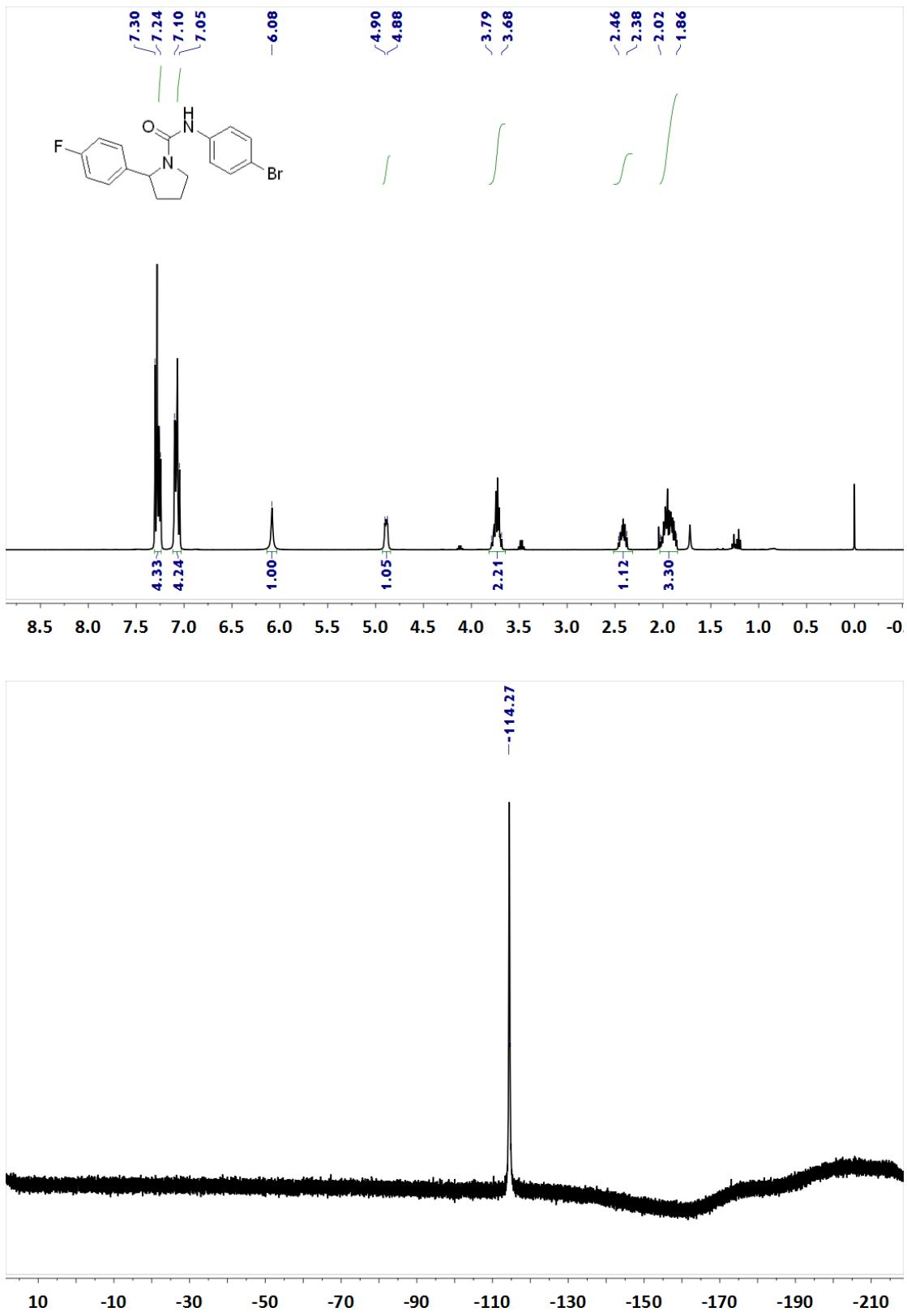
<sup>1</sup>H, <sup>13</sup>C and <sup>19</sup>F-NMR spectra of **1d**.



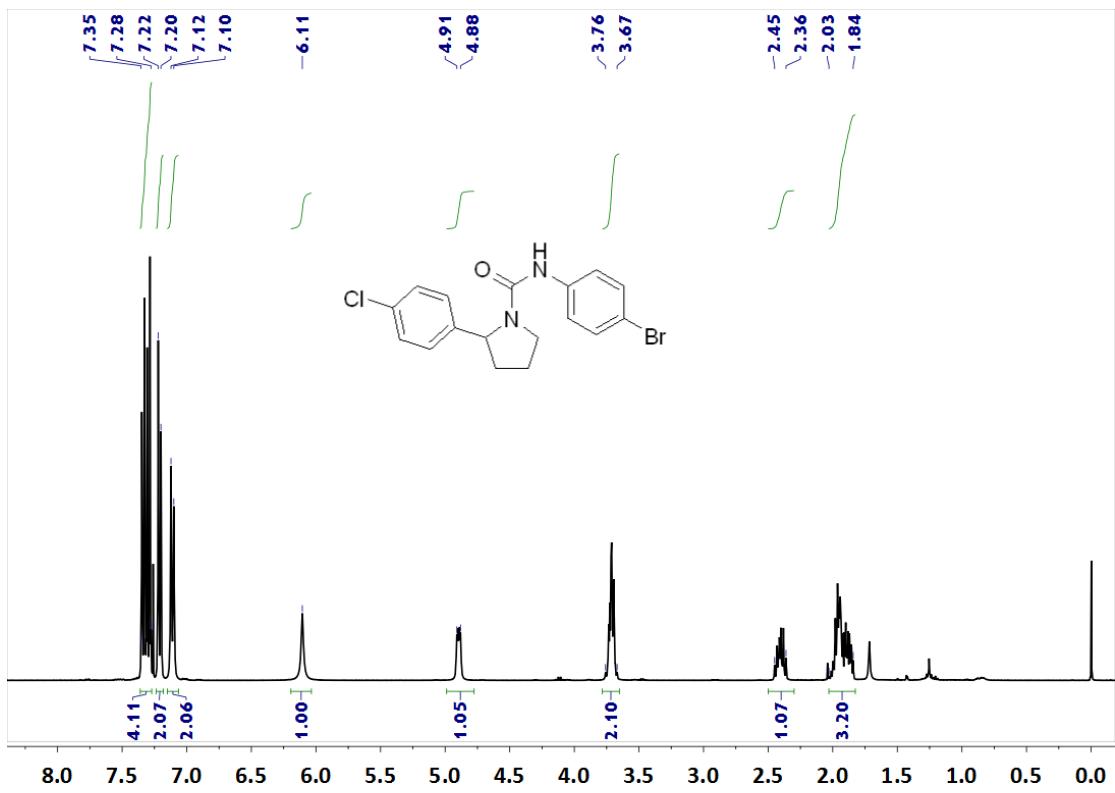
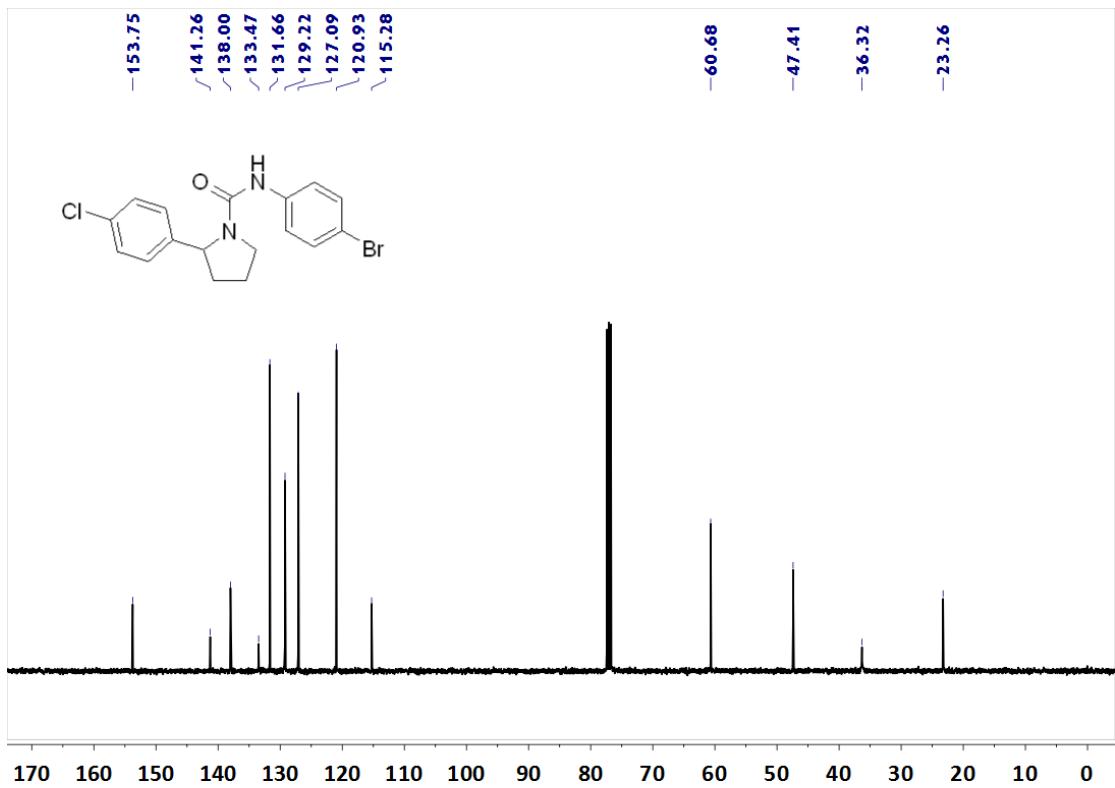


<sup>1</sup>H, <sup>13</sup>C and <sup>19</sup>F- NMR spectra of 1e.

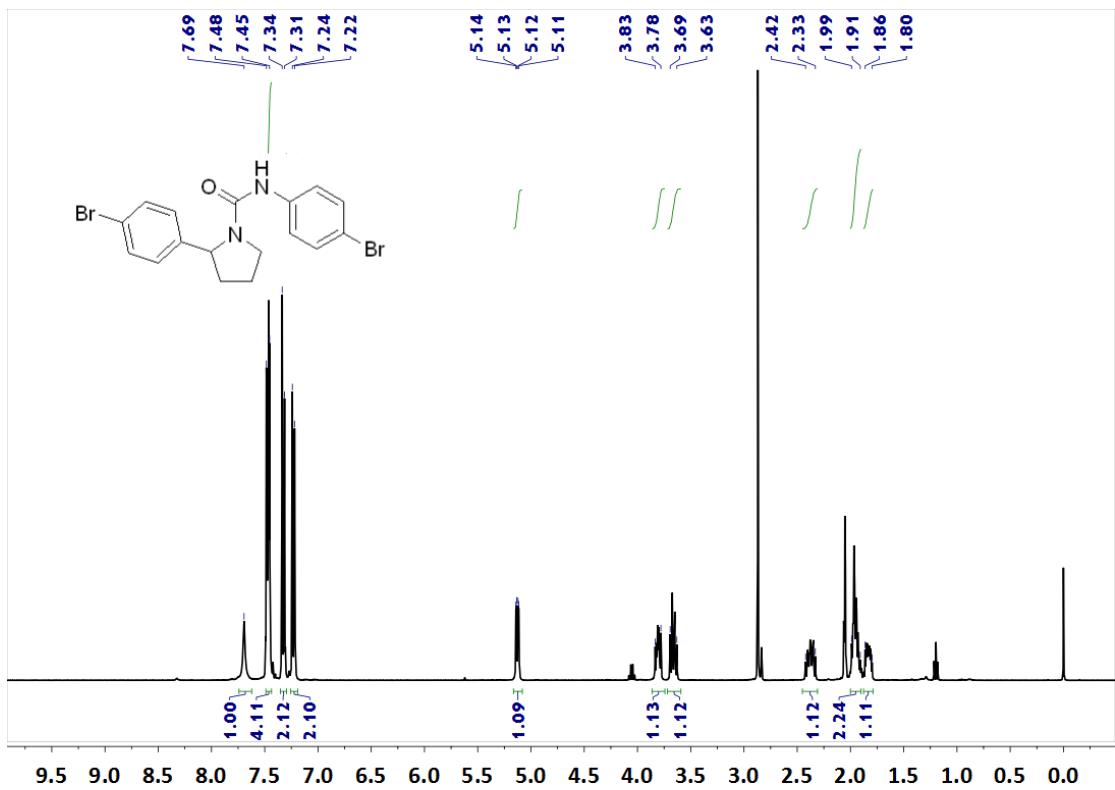
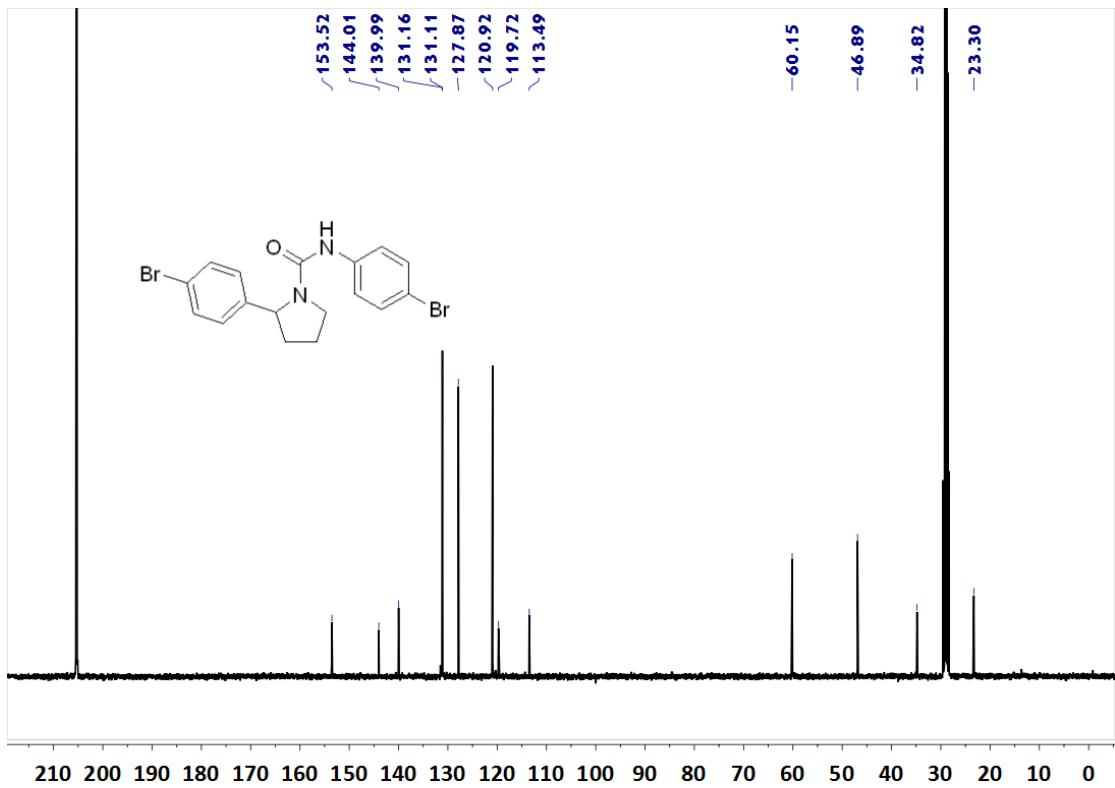




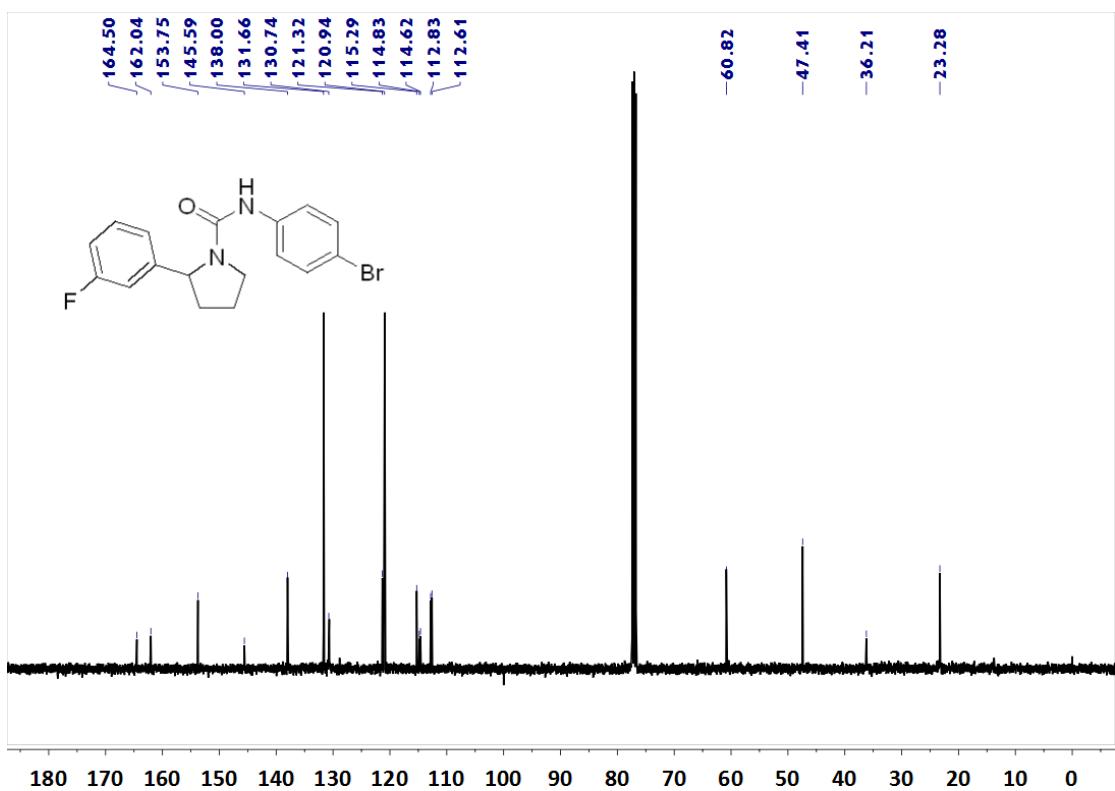
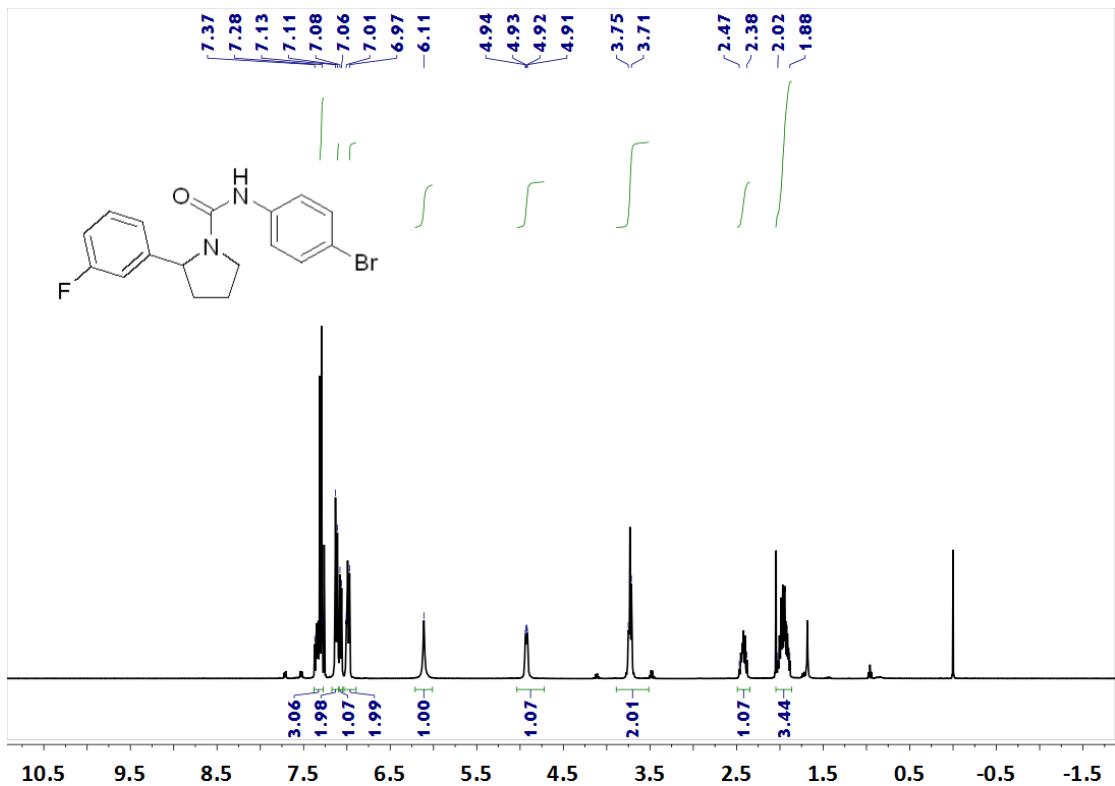
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **1f**.

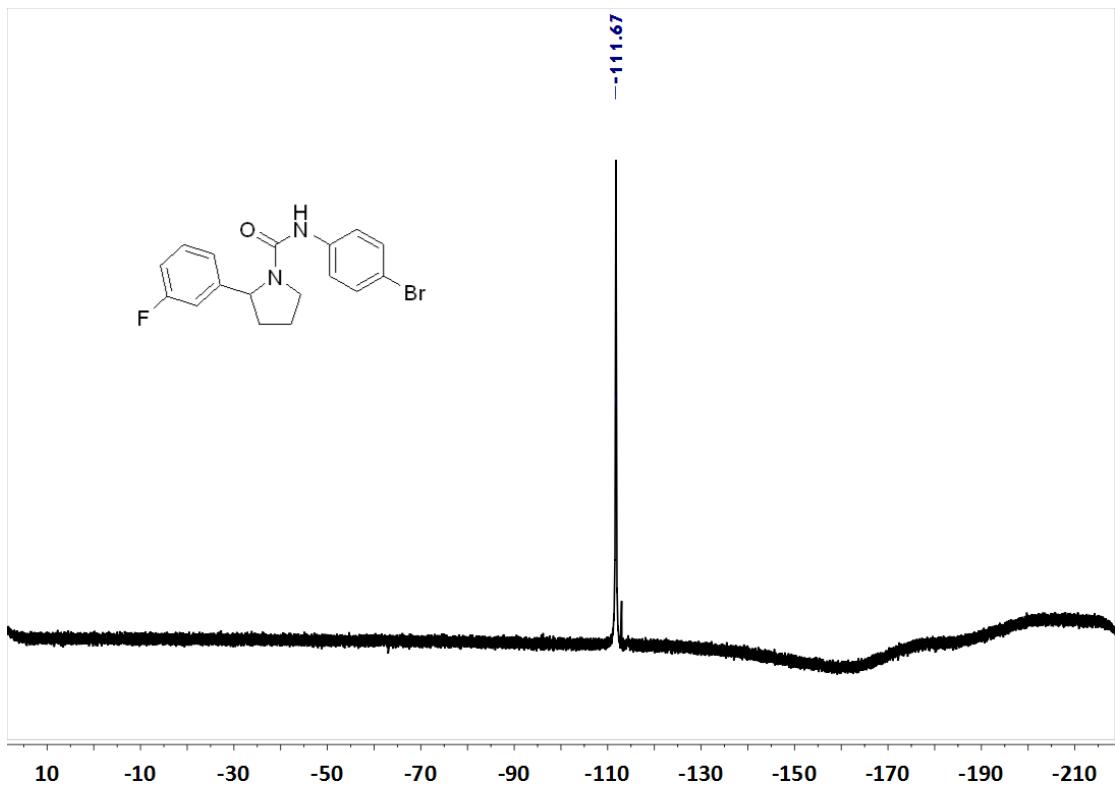


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1g**.

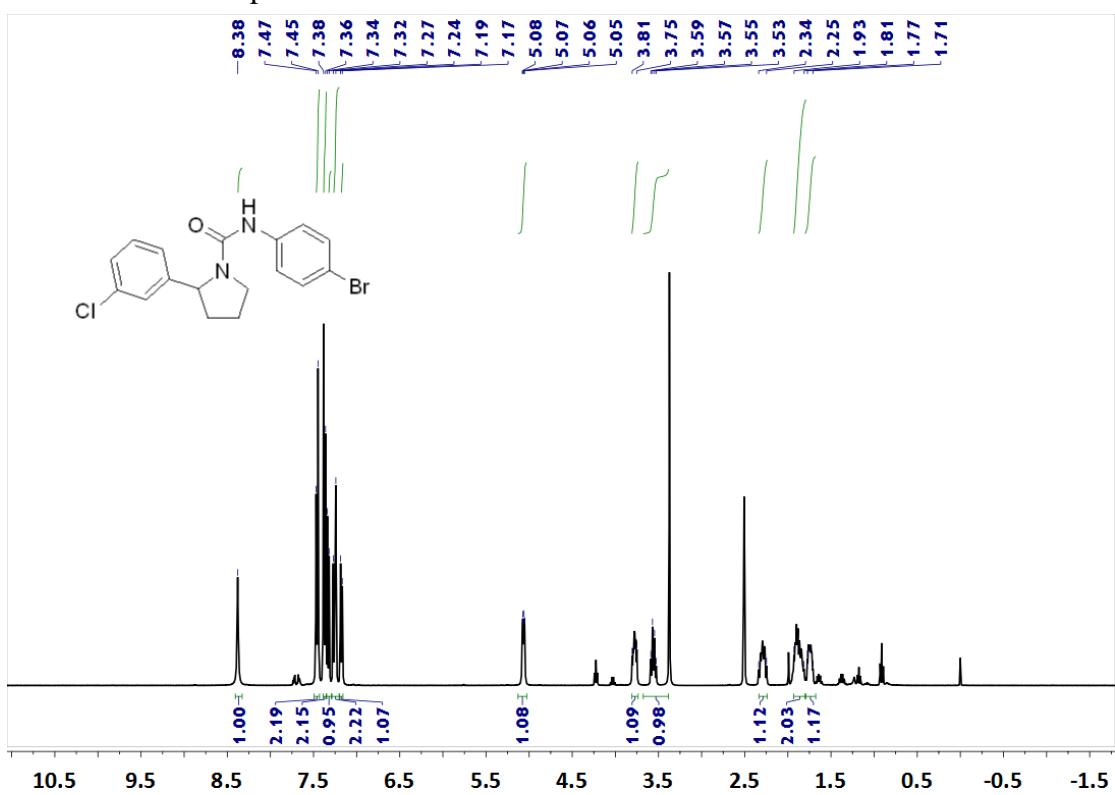


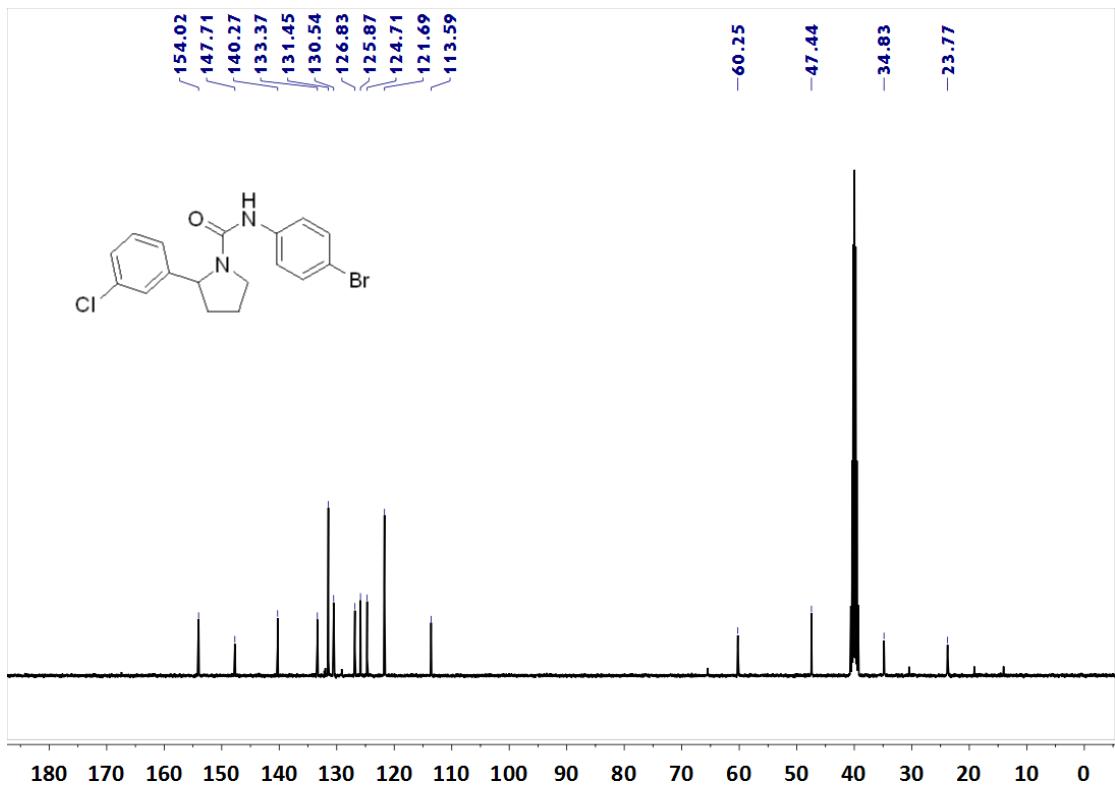
<sup>1</sup>H, <sup>13</sup>C and <sup>19</sup>F-NMR spectra of **1h**.



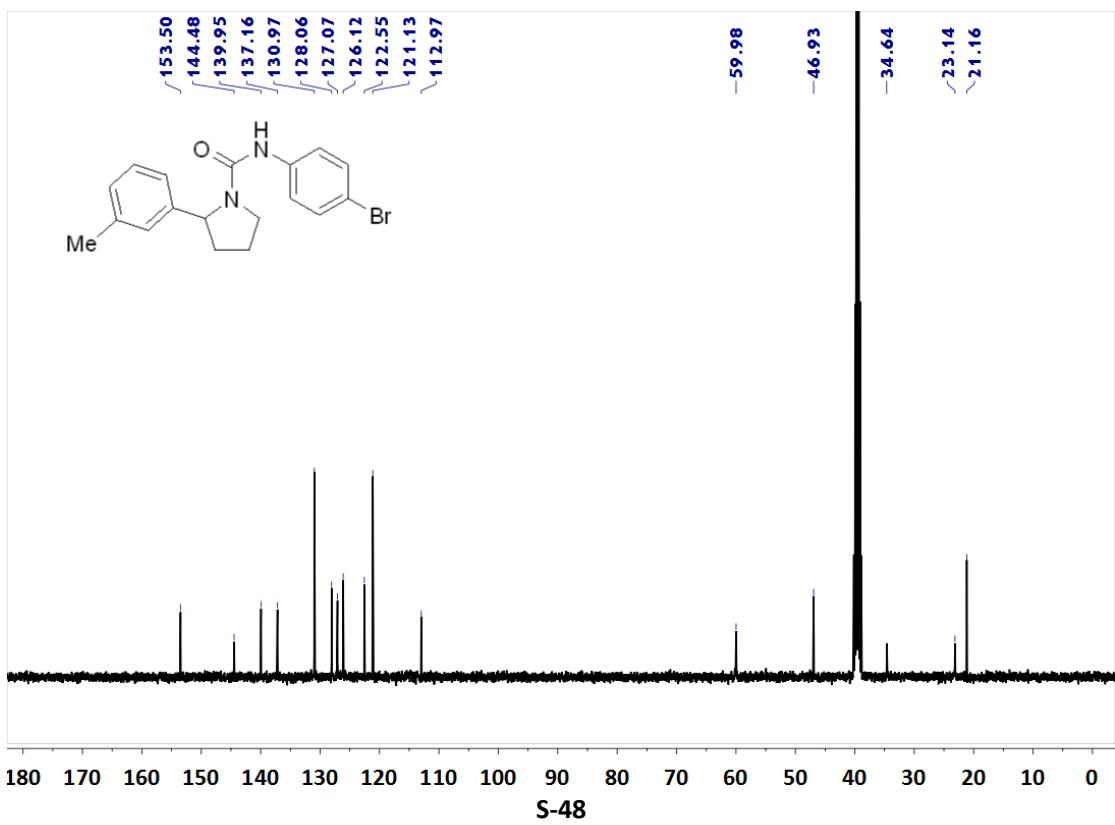


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1i**.

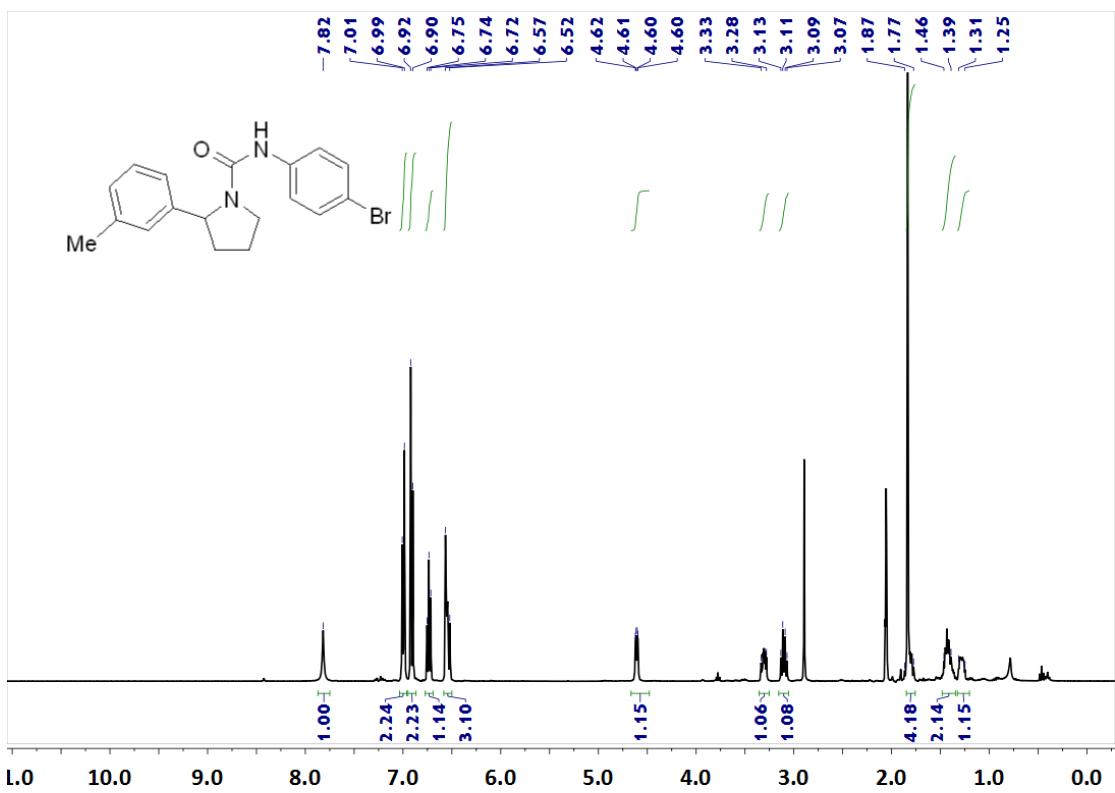




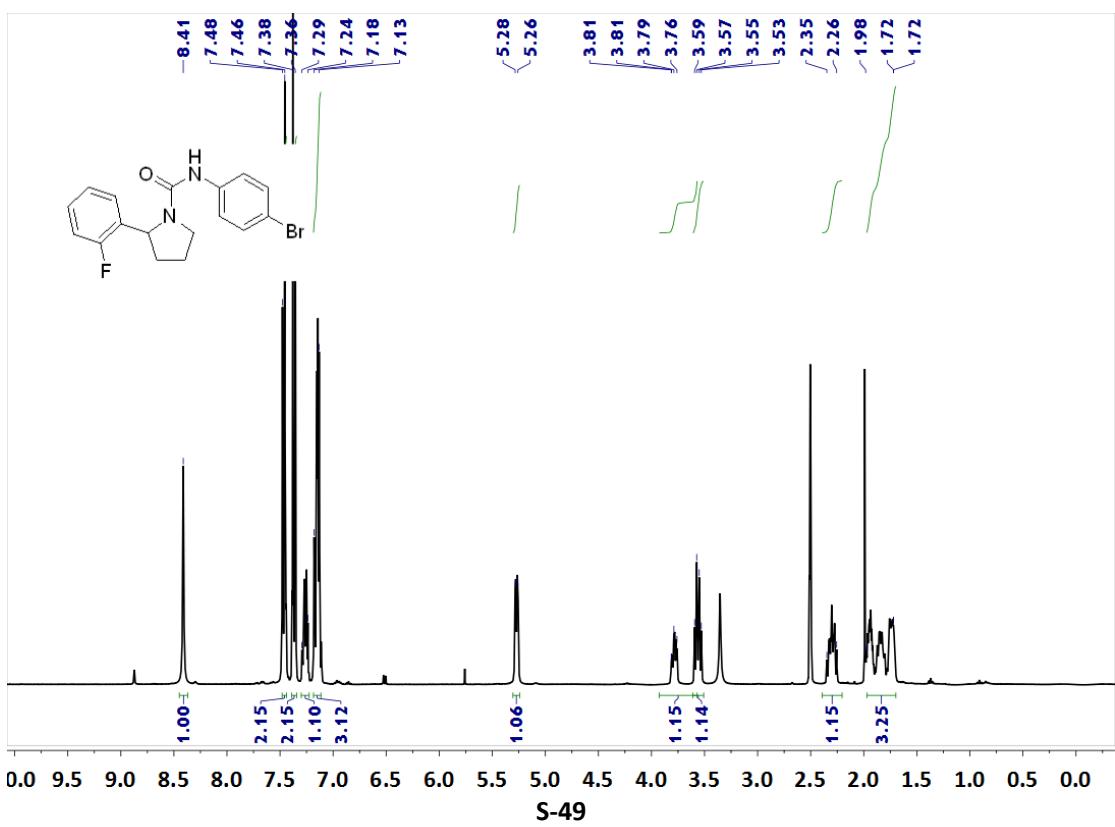
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1j**.

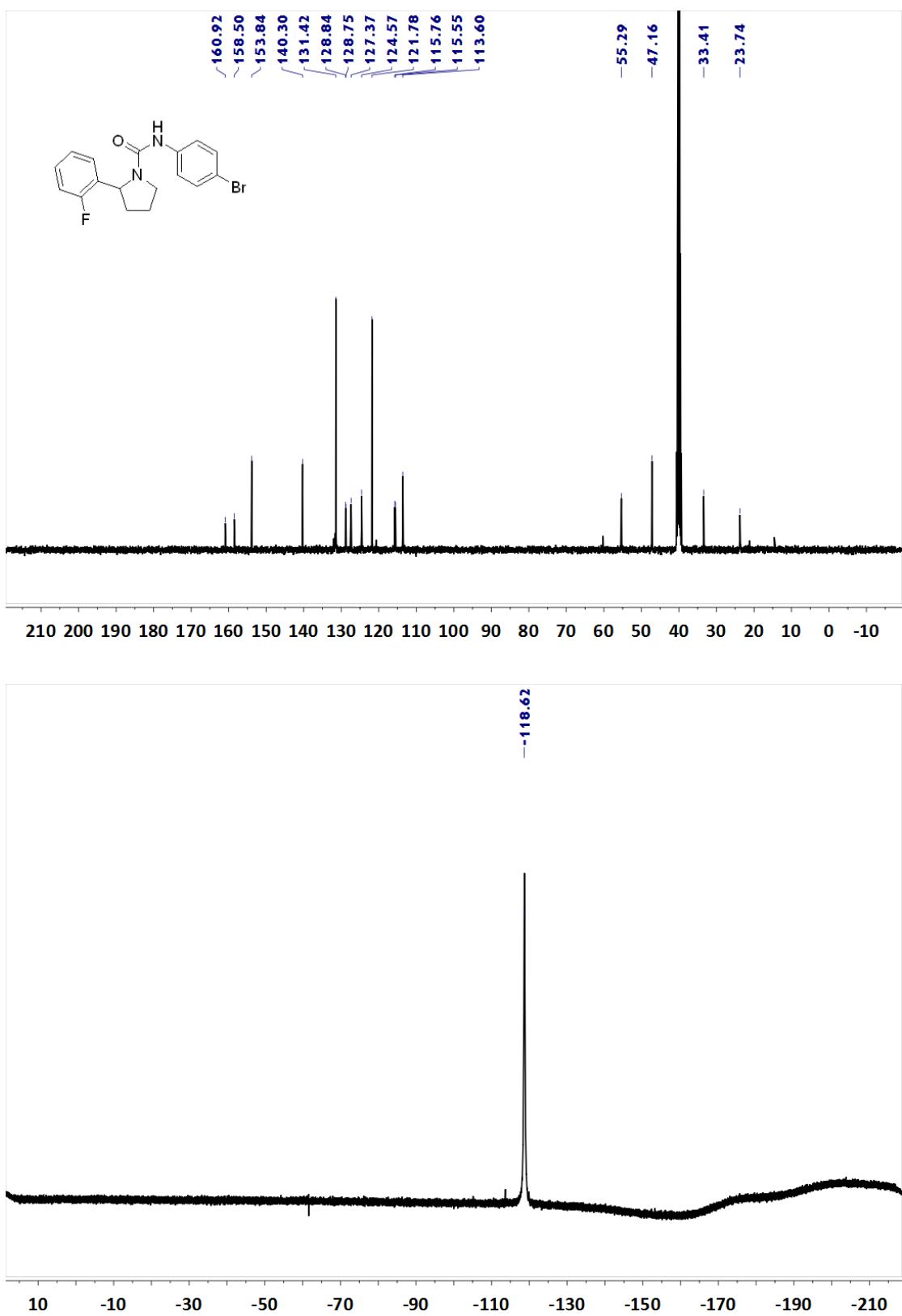


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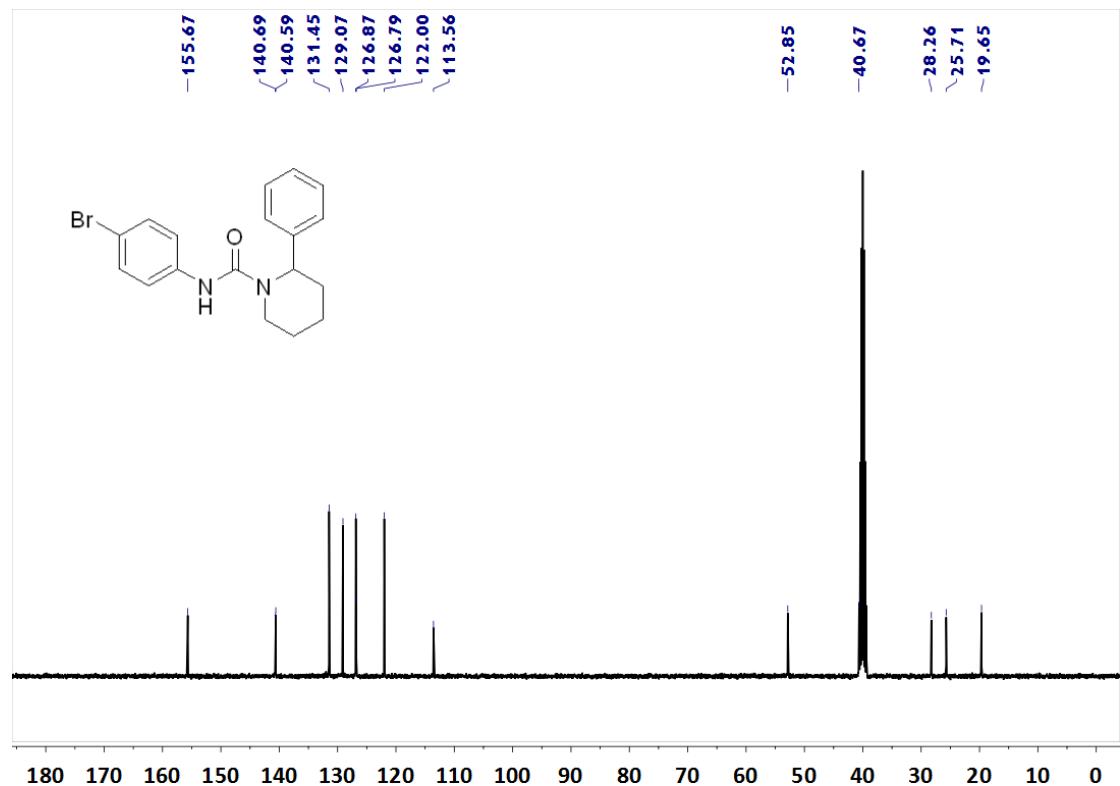
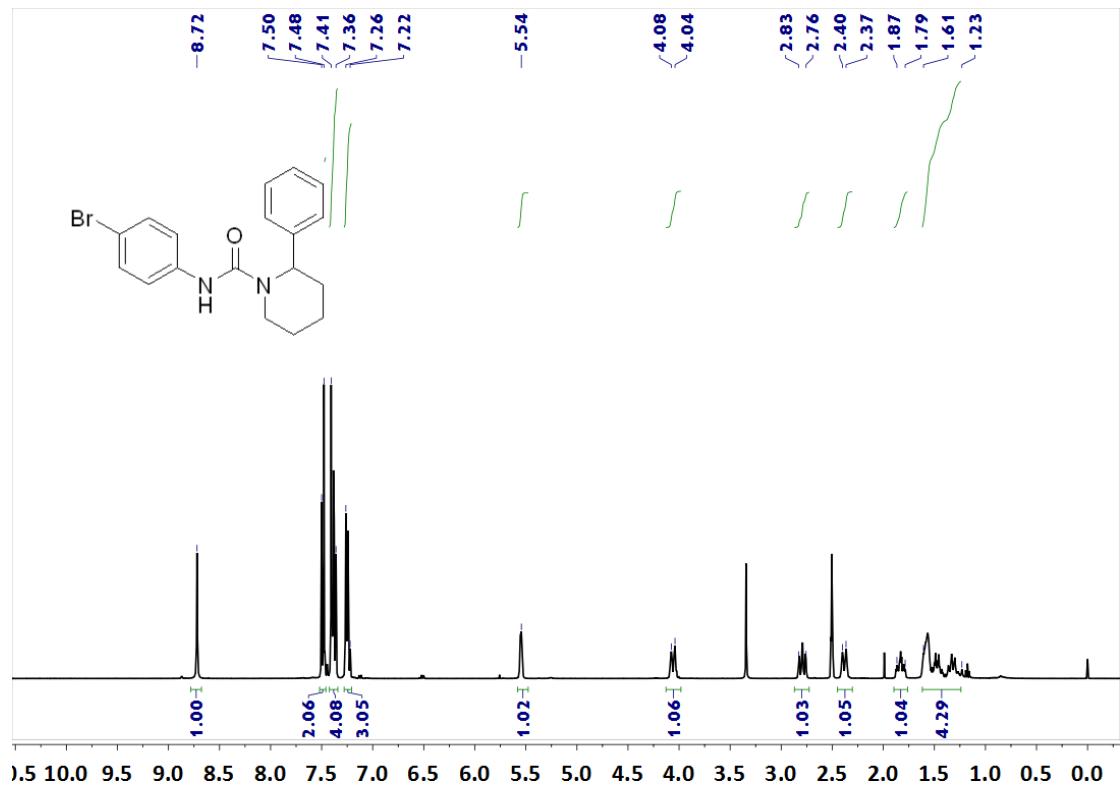


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1k**.

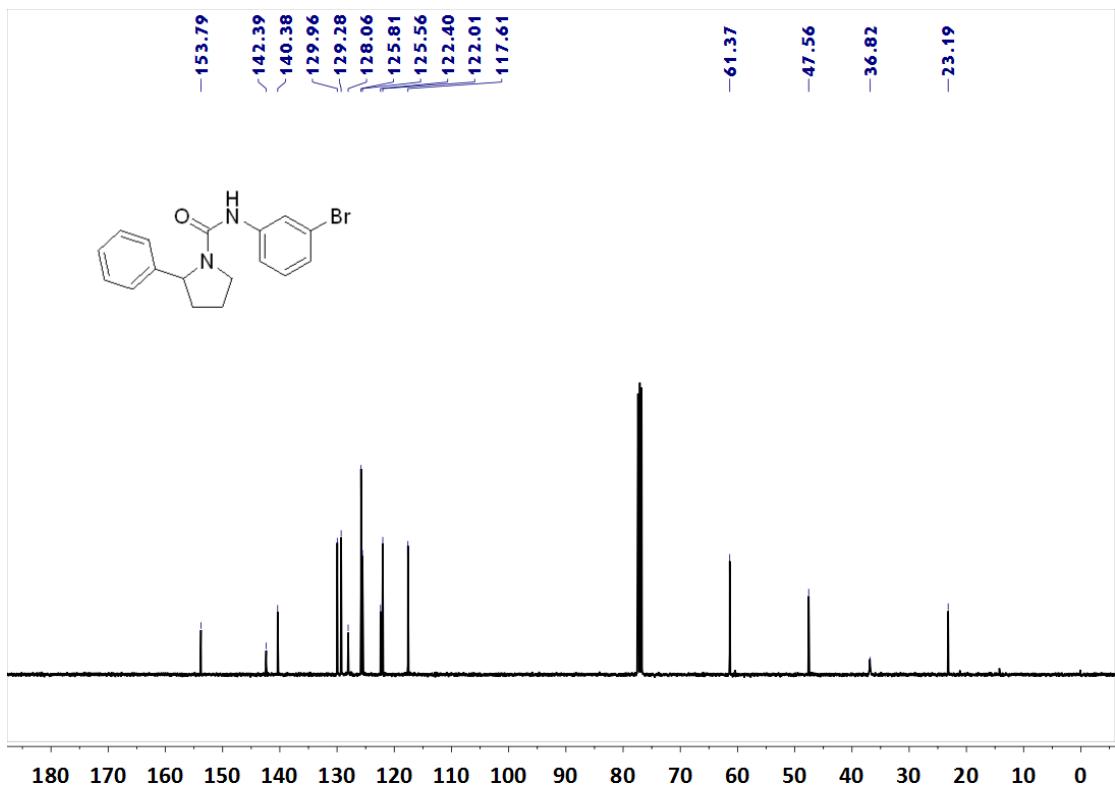
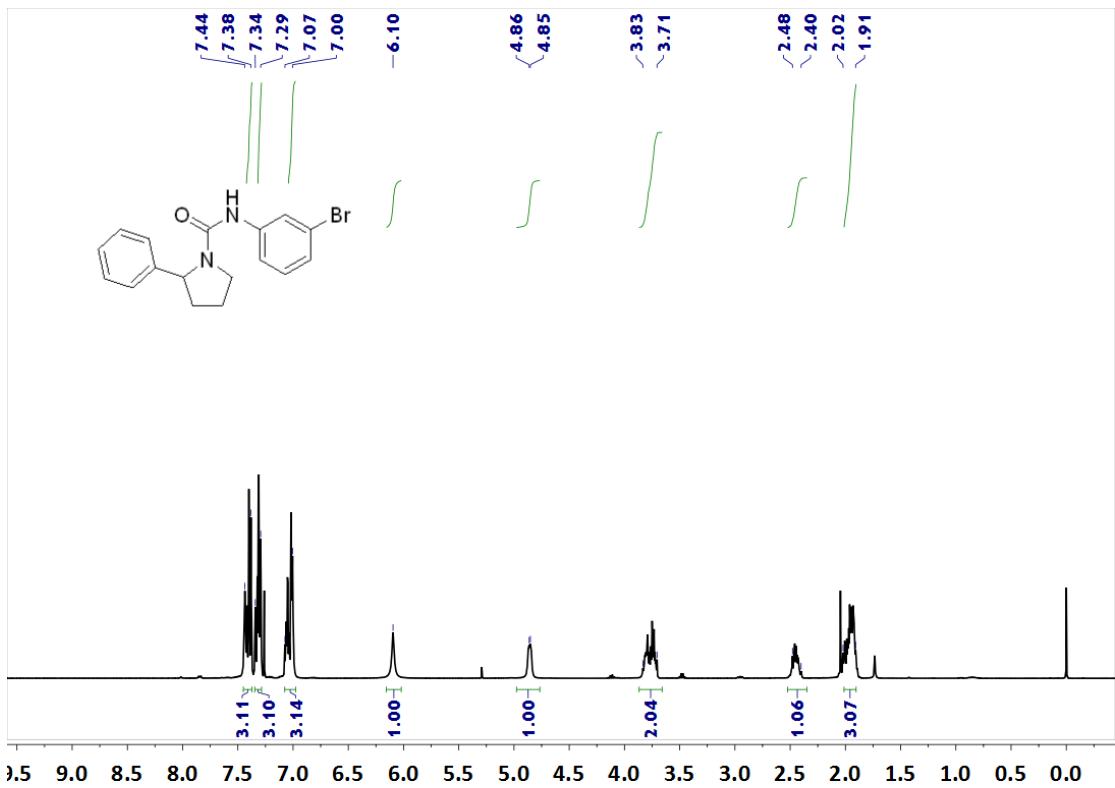




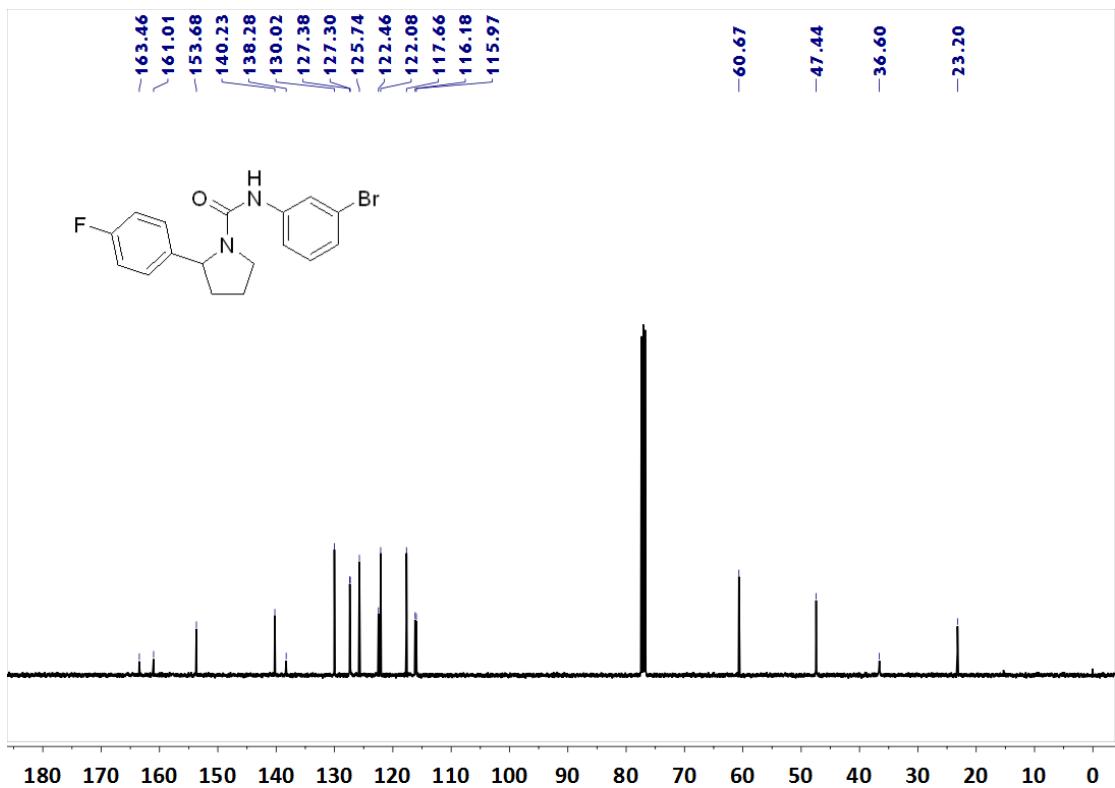
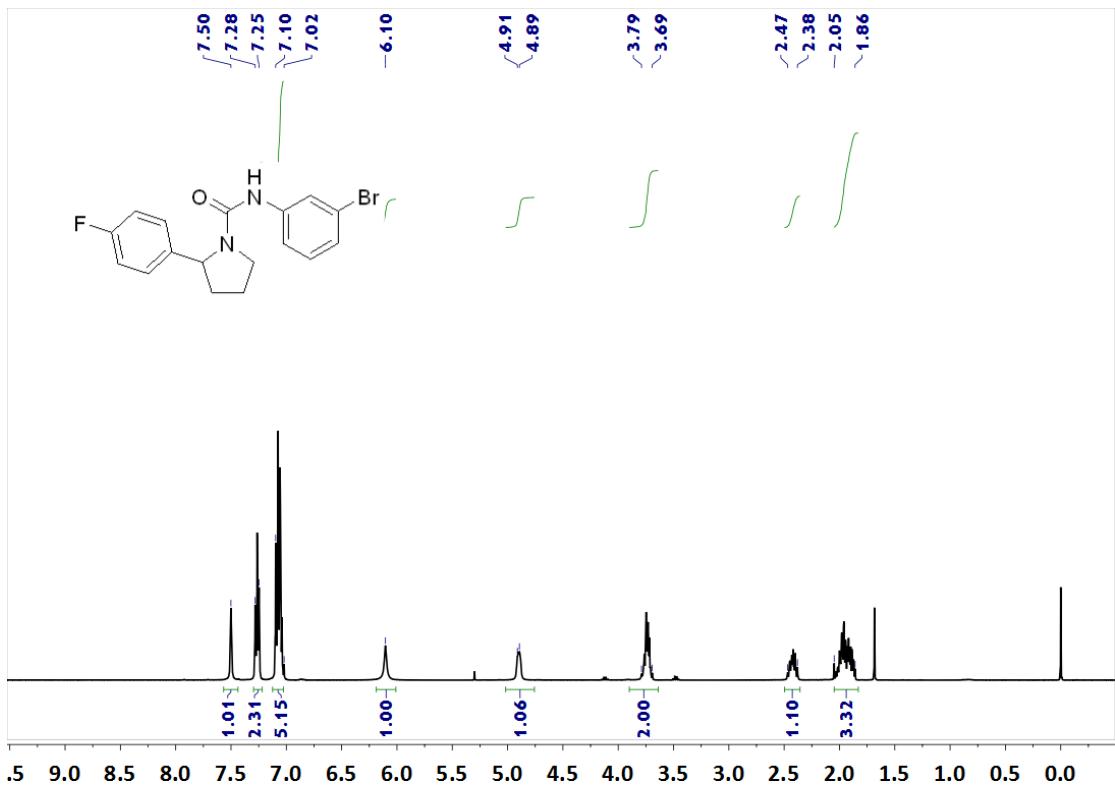
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **1l**.

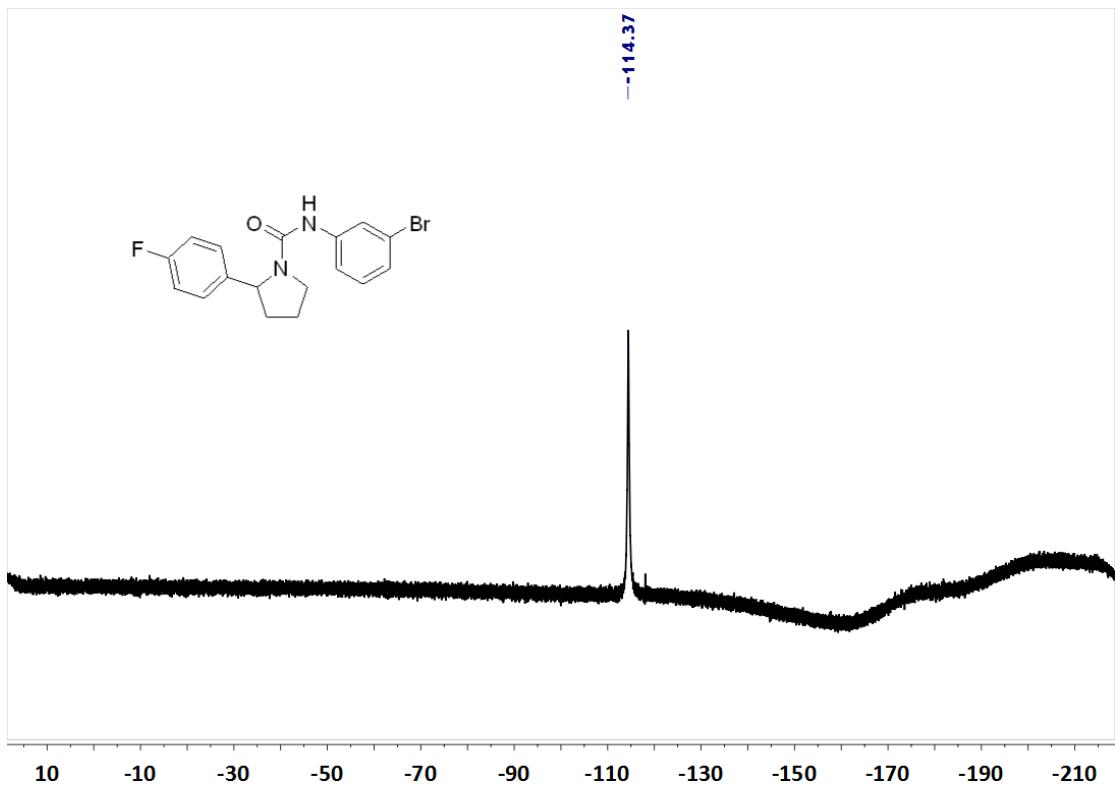


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1m**.

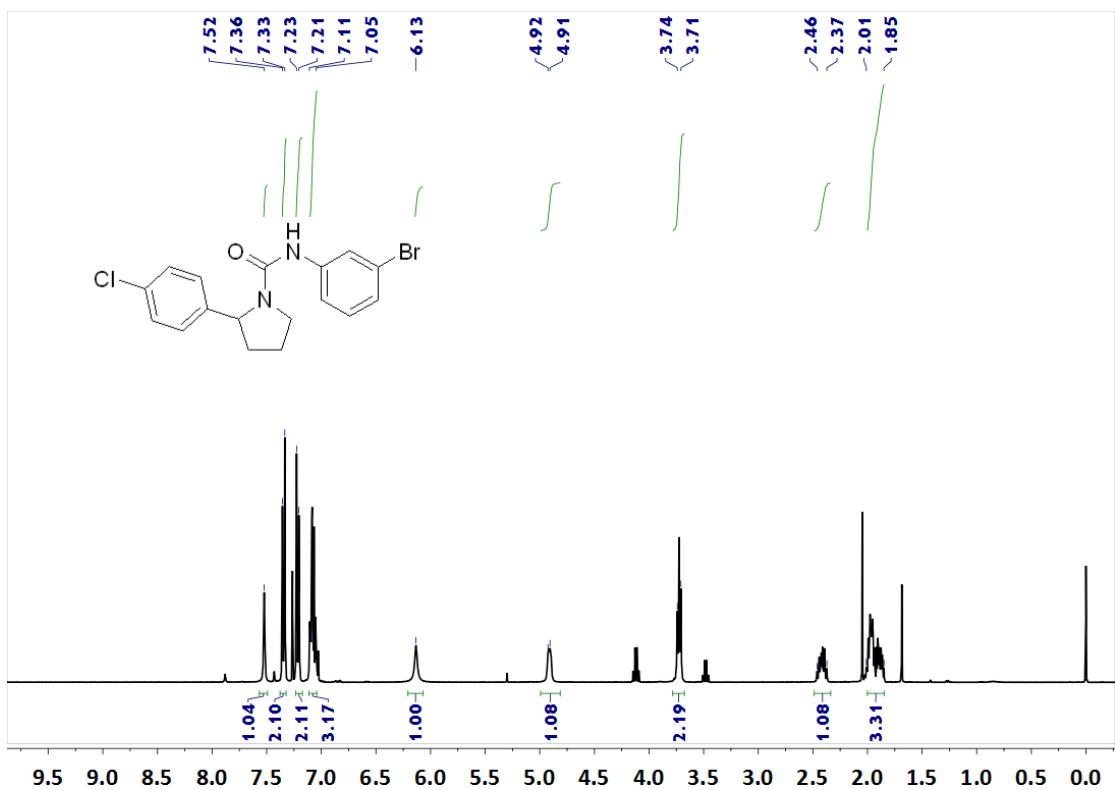


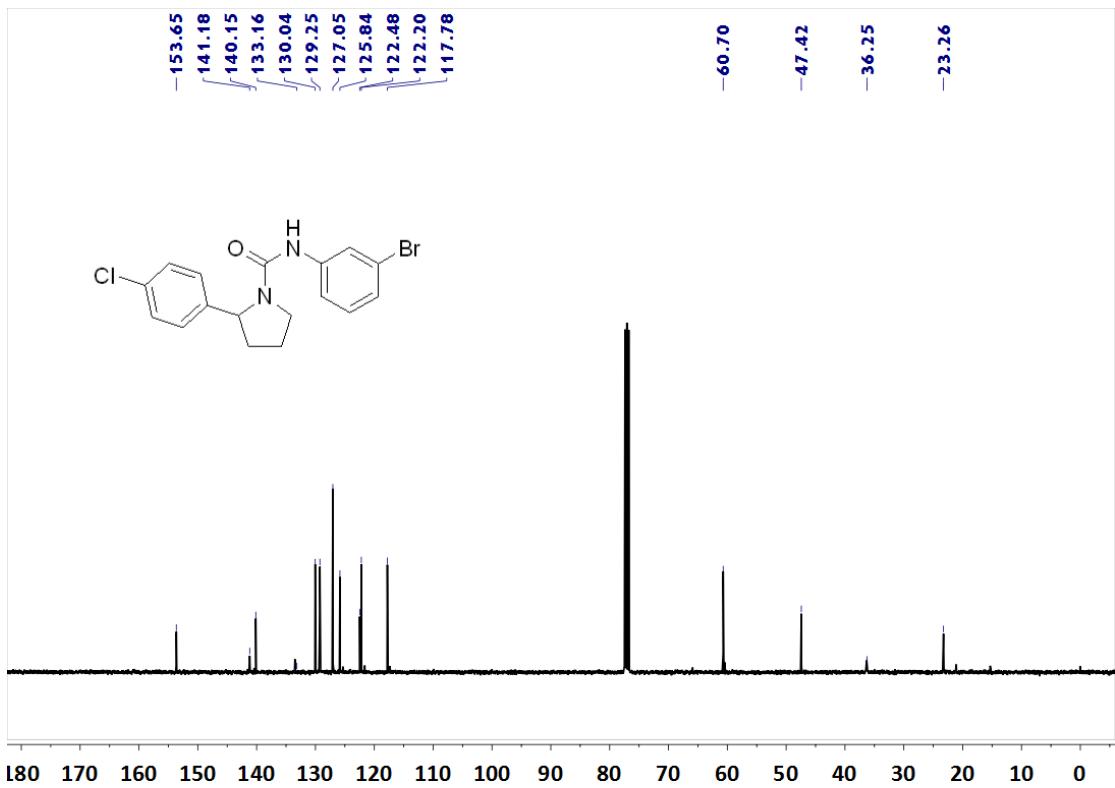
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1n**.



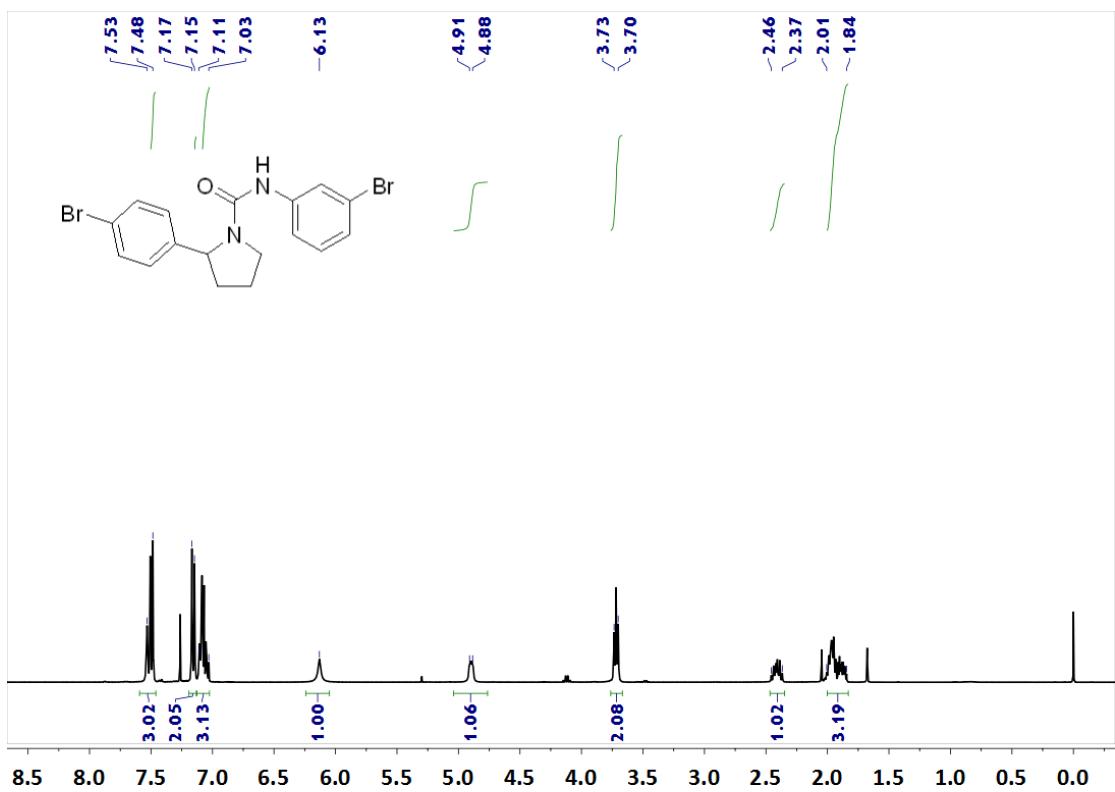


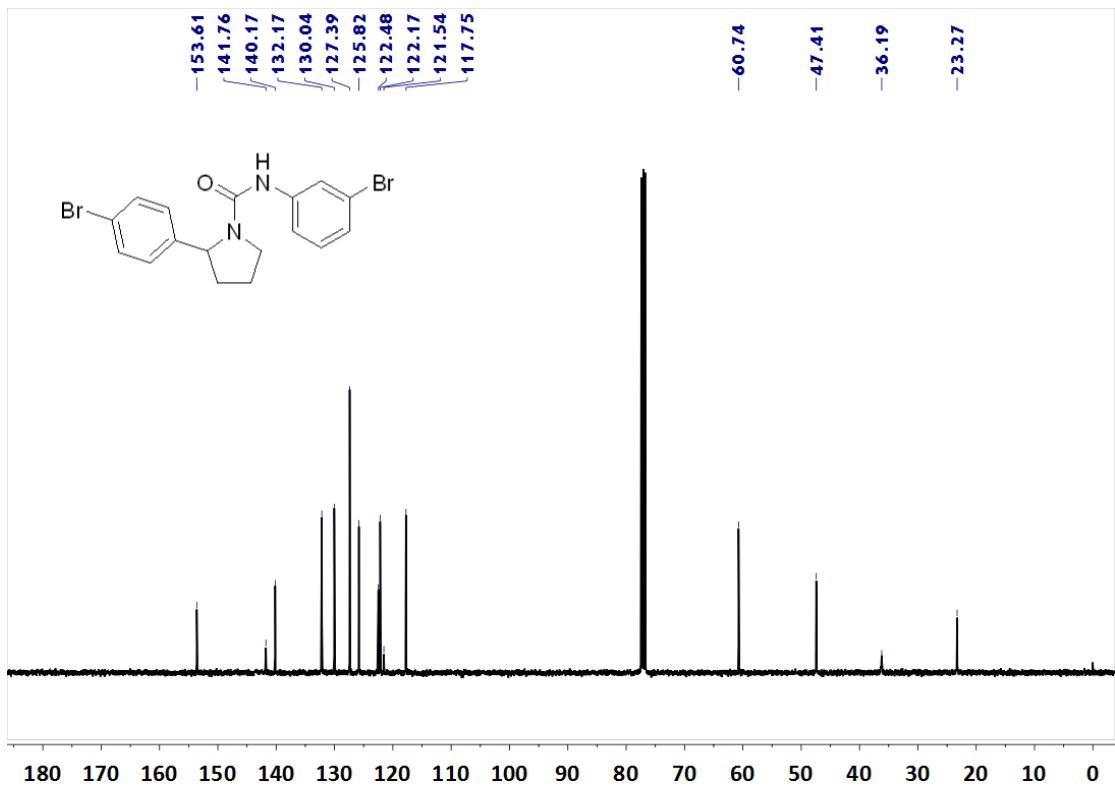
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1o**.



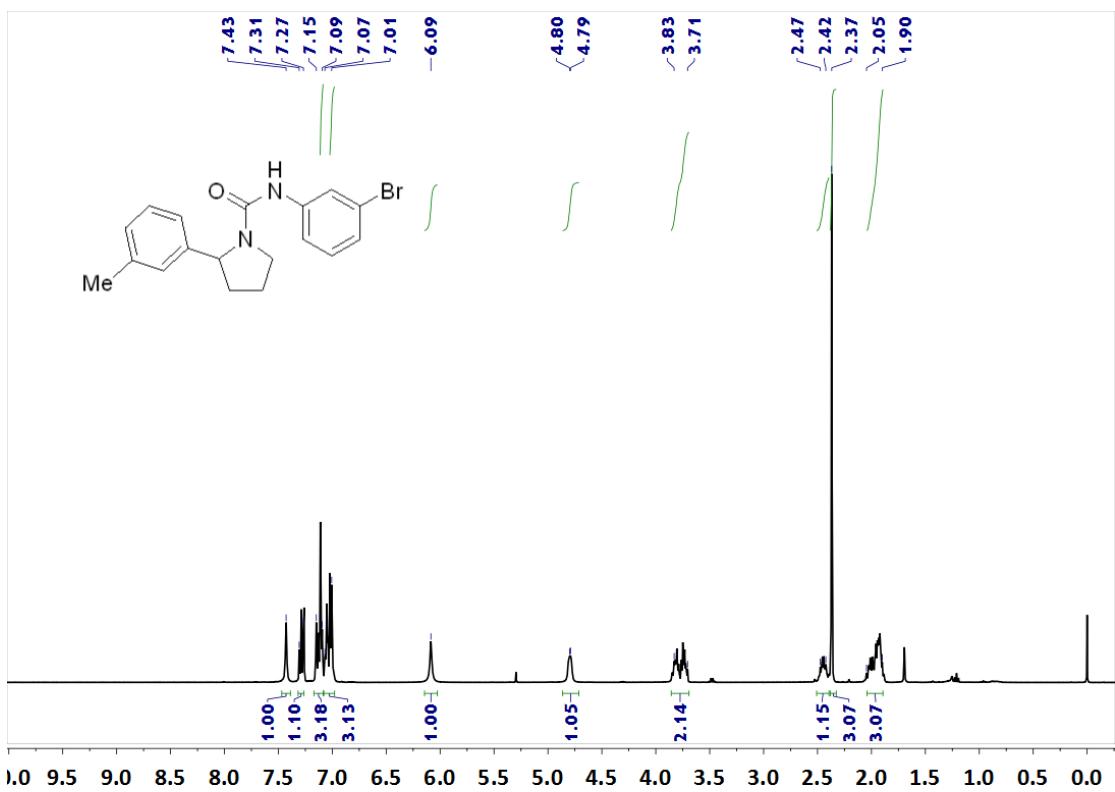


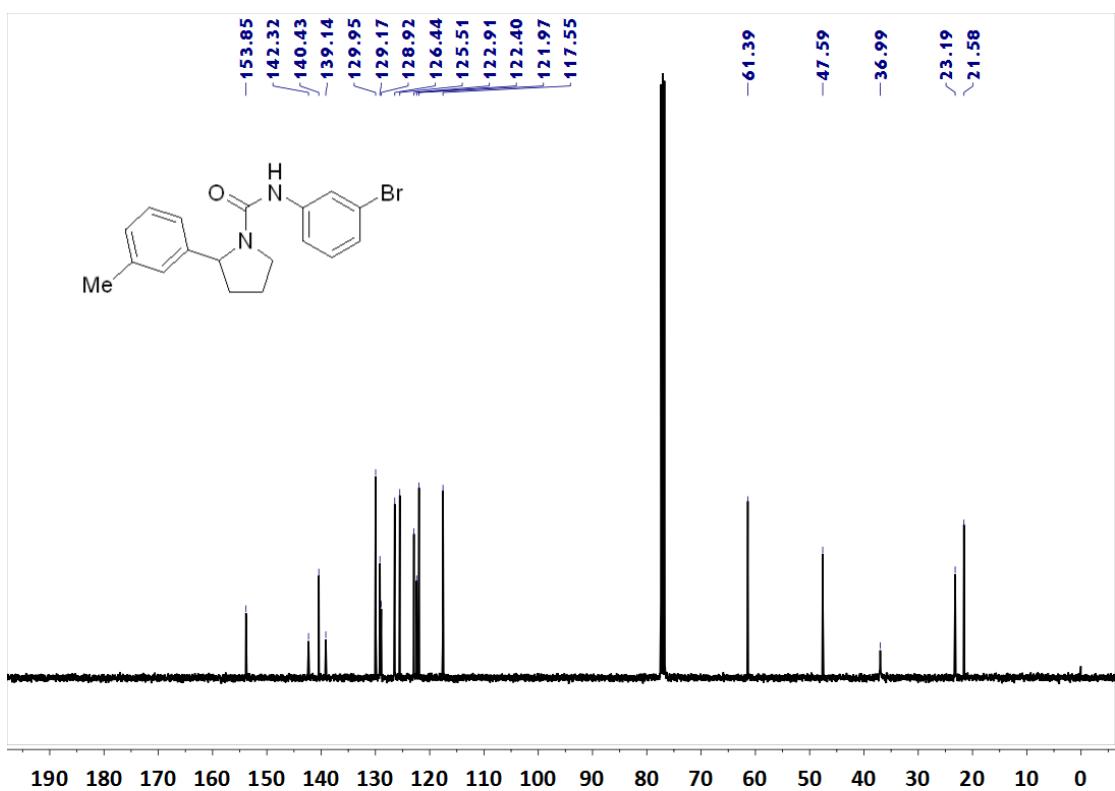
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1p**.



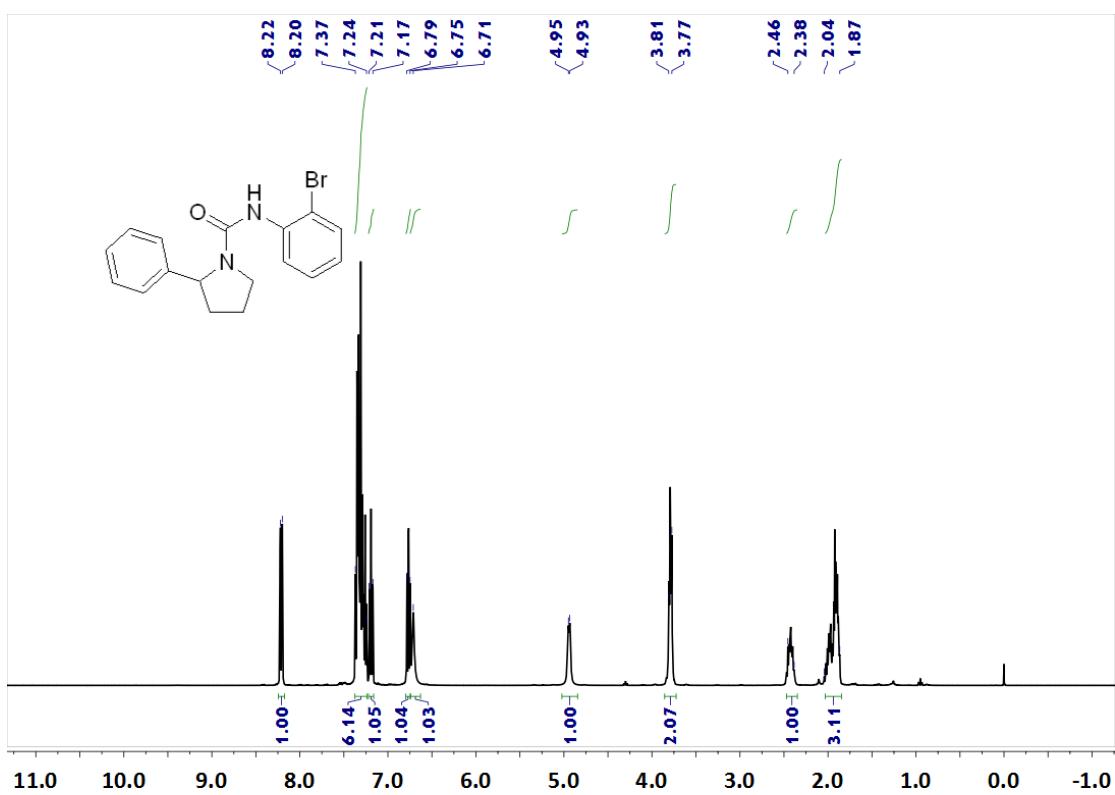


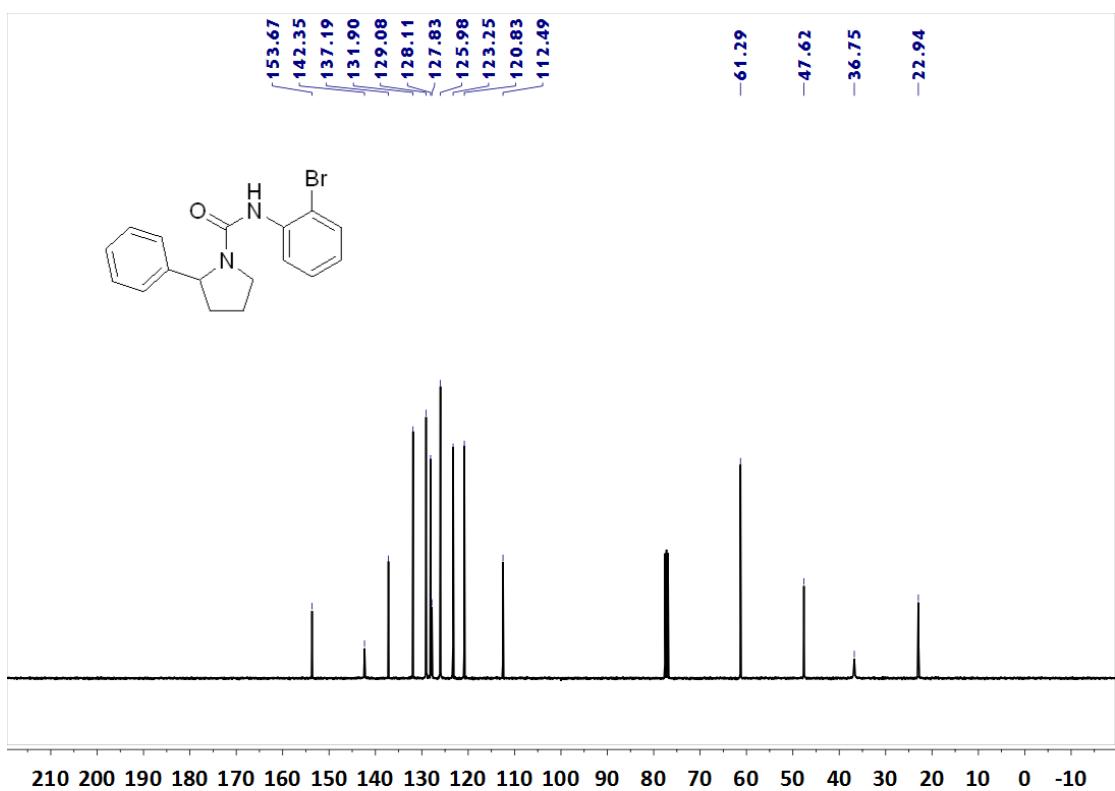
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1q**.



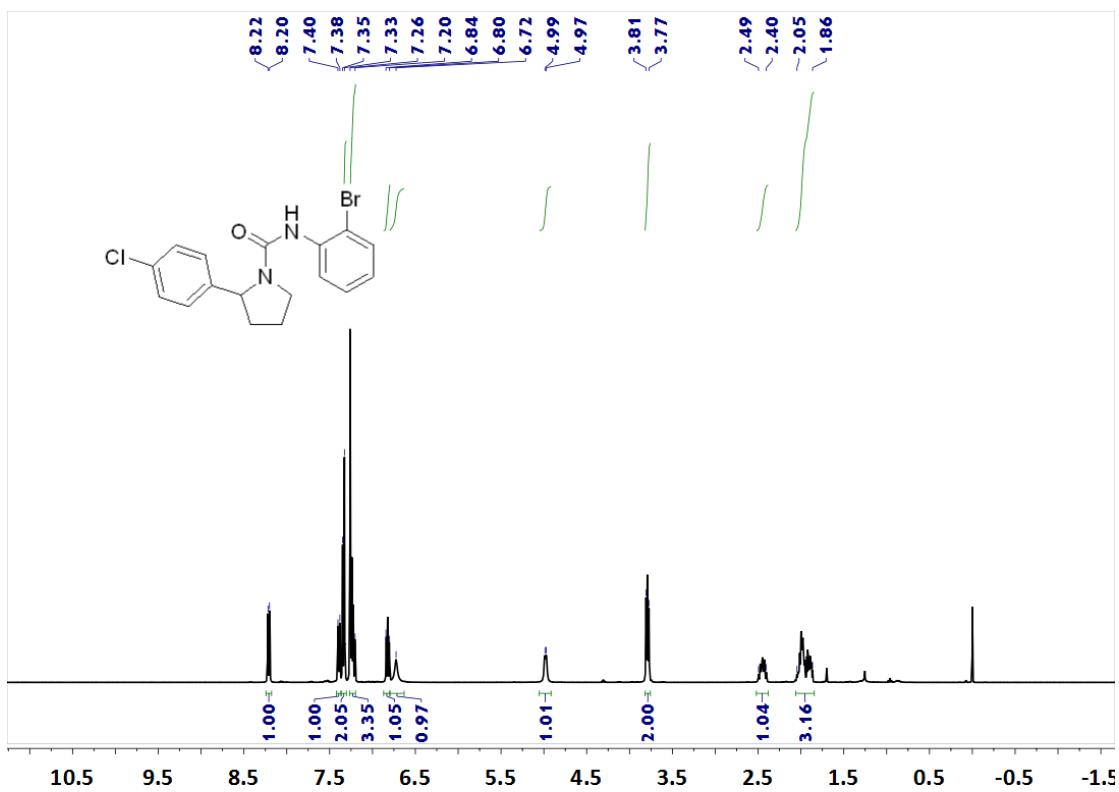


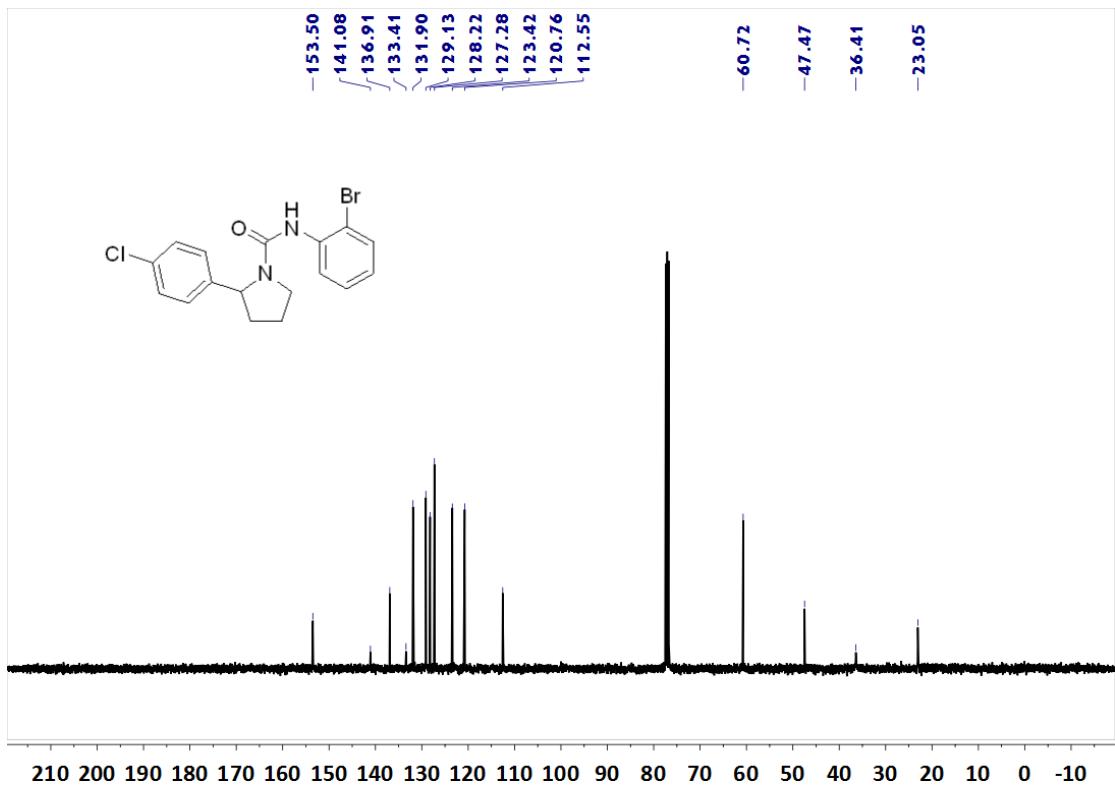
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1r**.



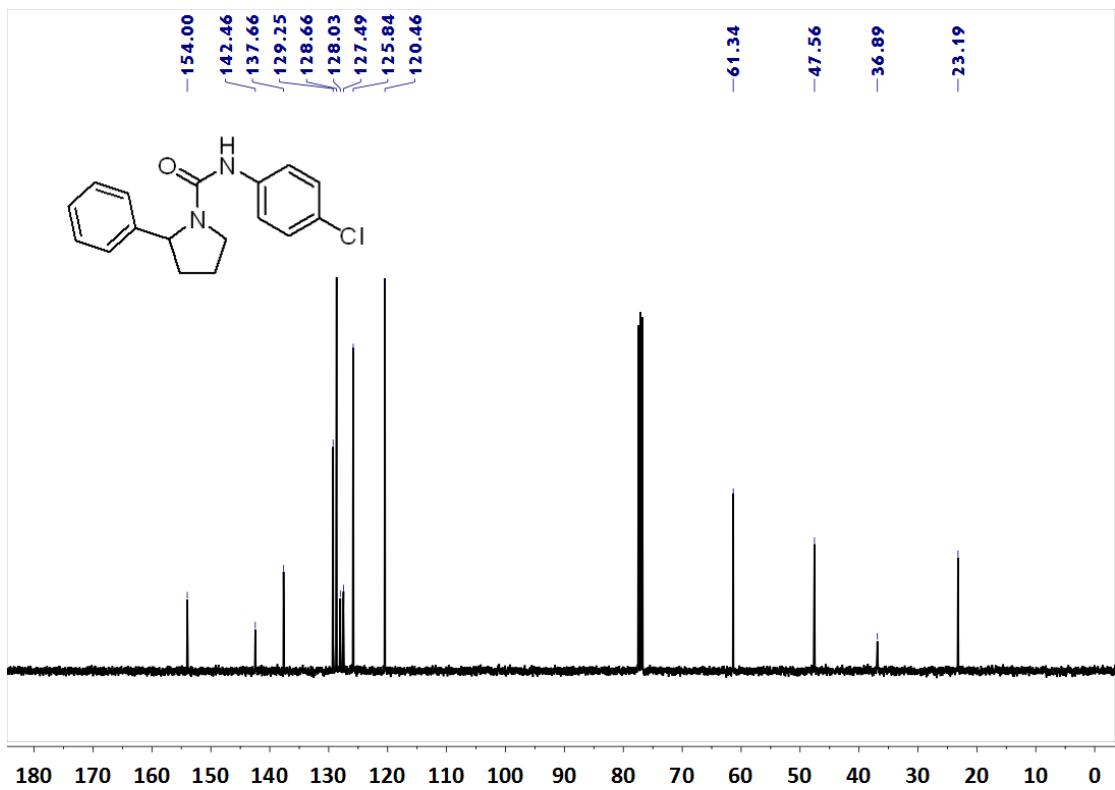


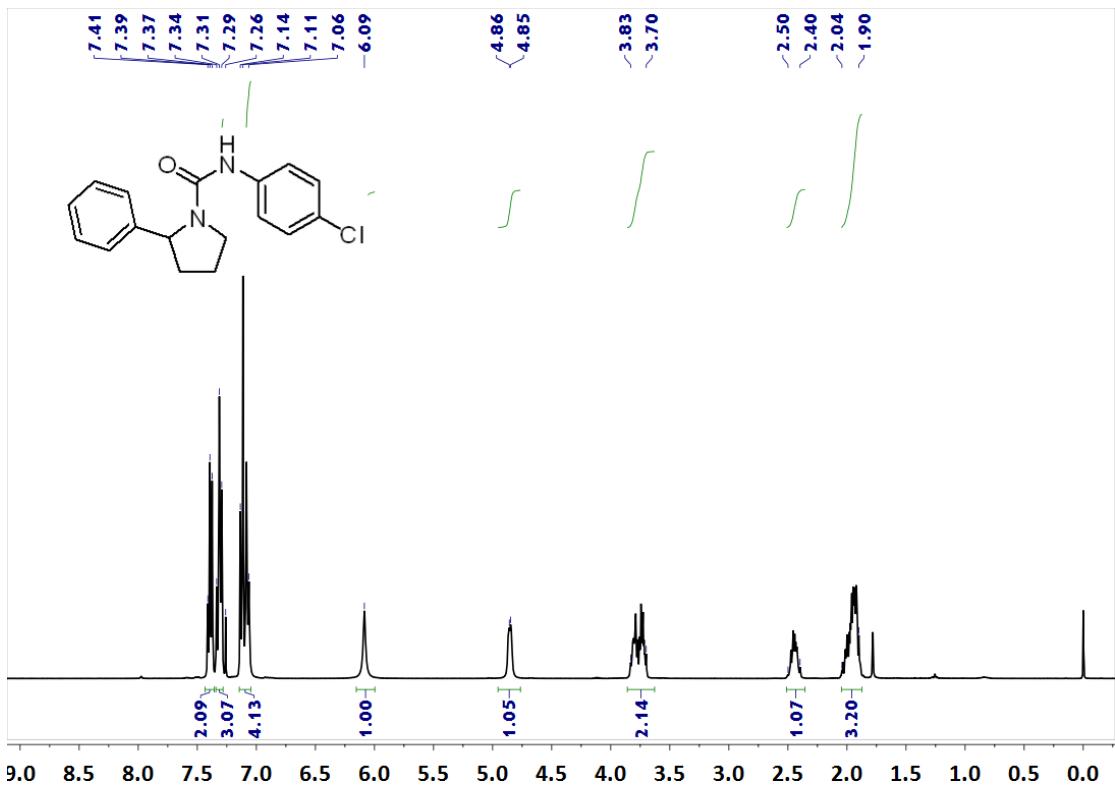
1H and 13C-NMR spectra of **1s**.



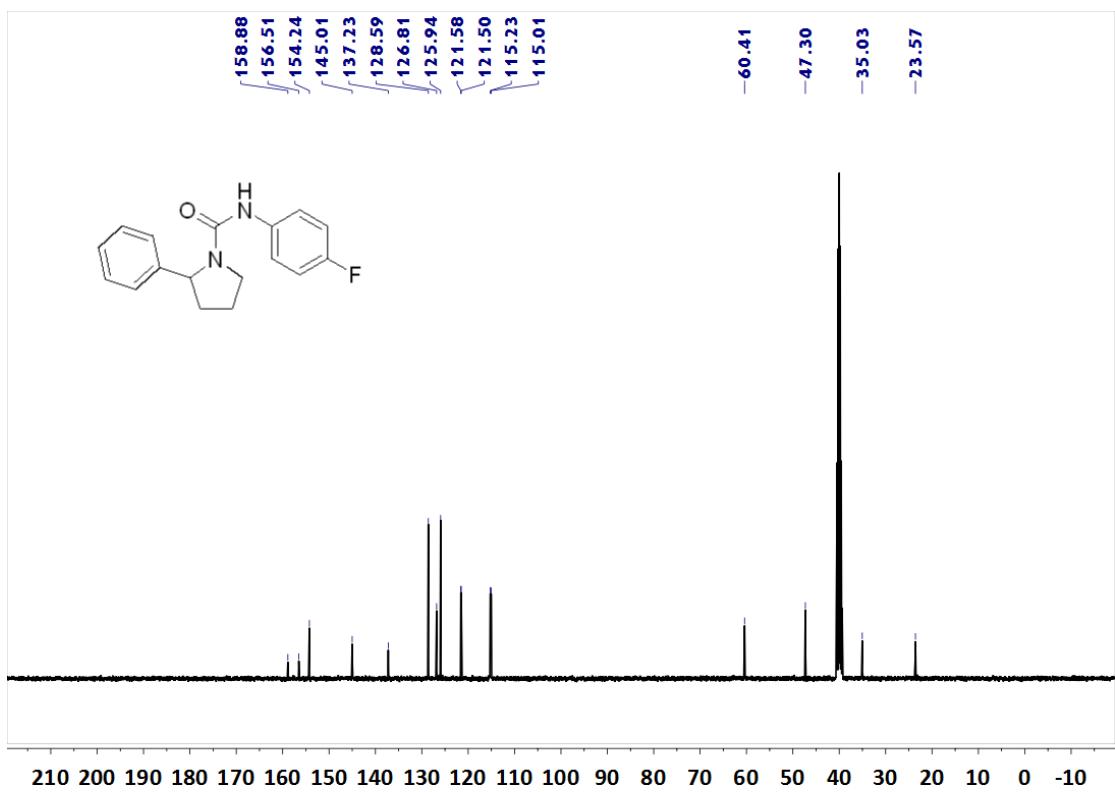


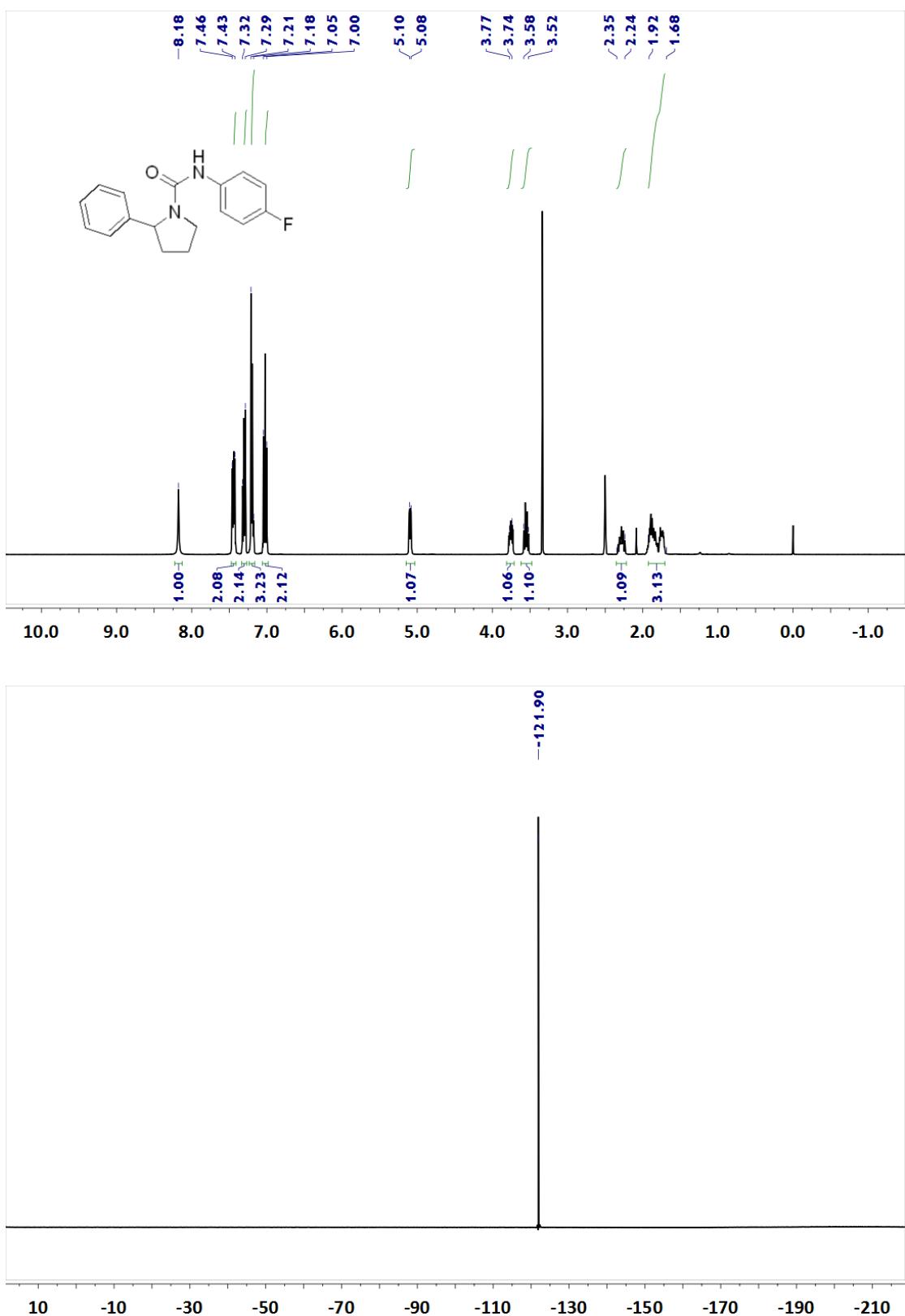
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1t**.



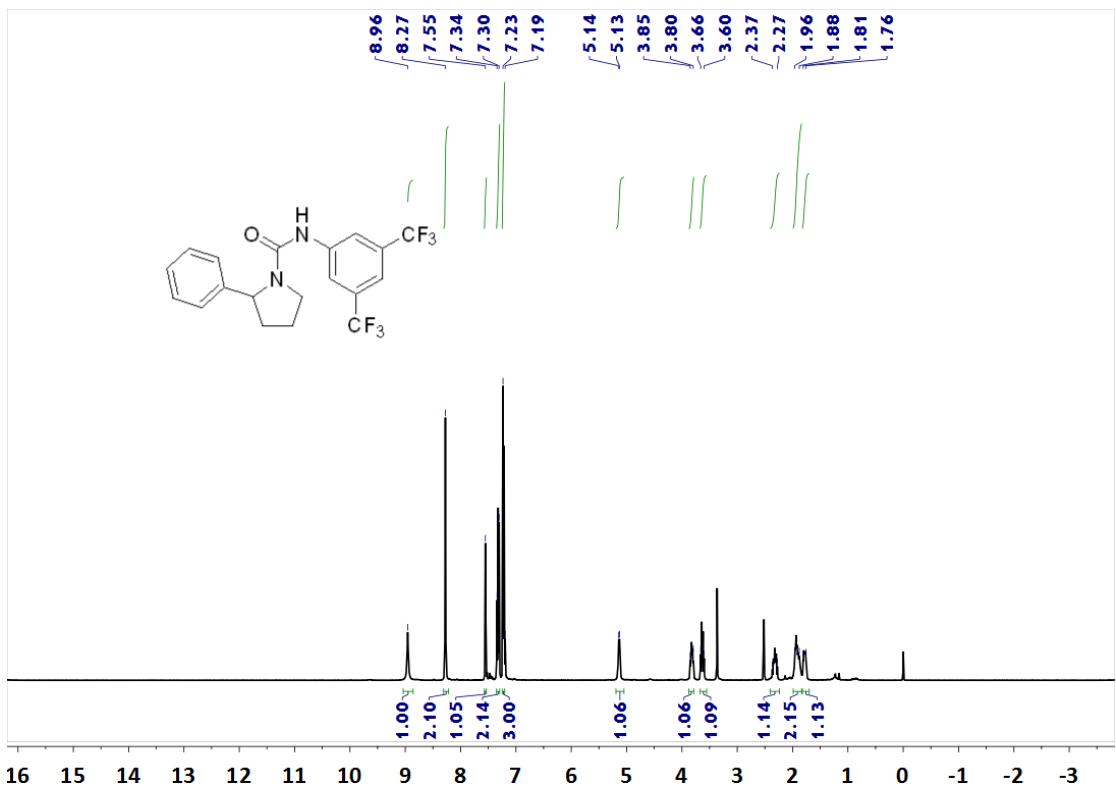
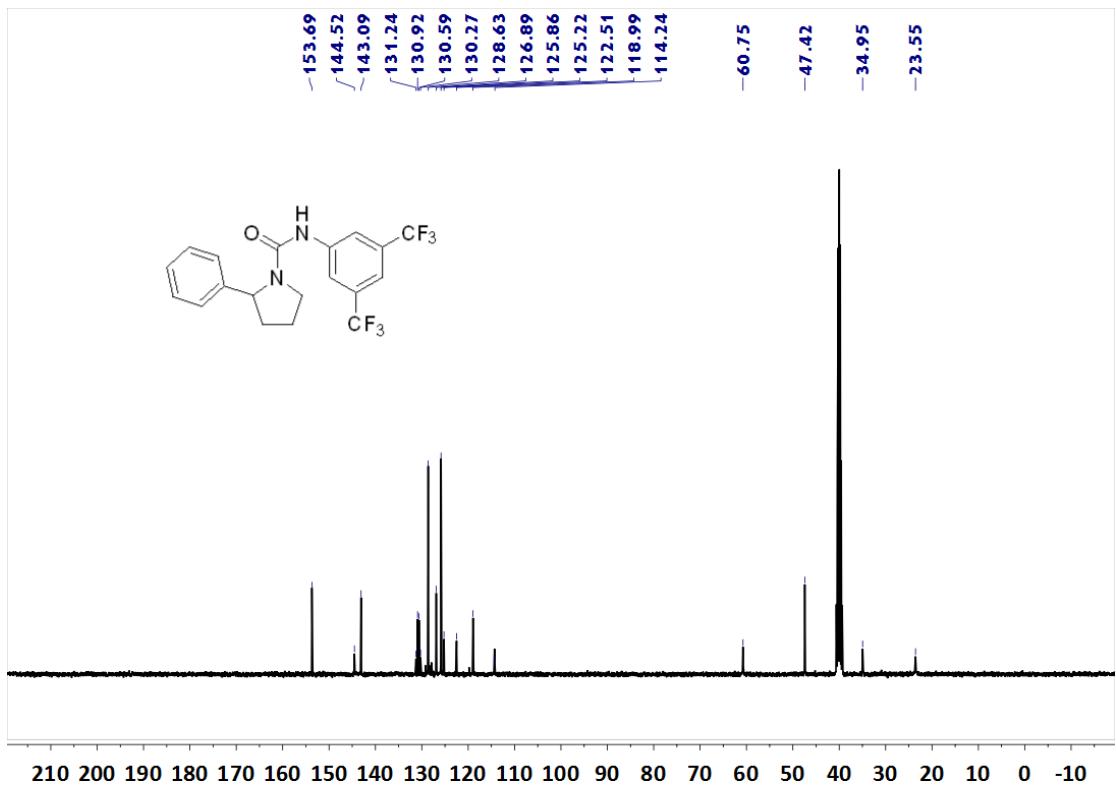


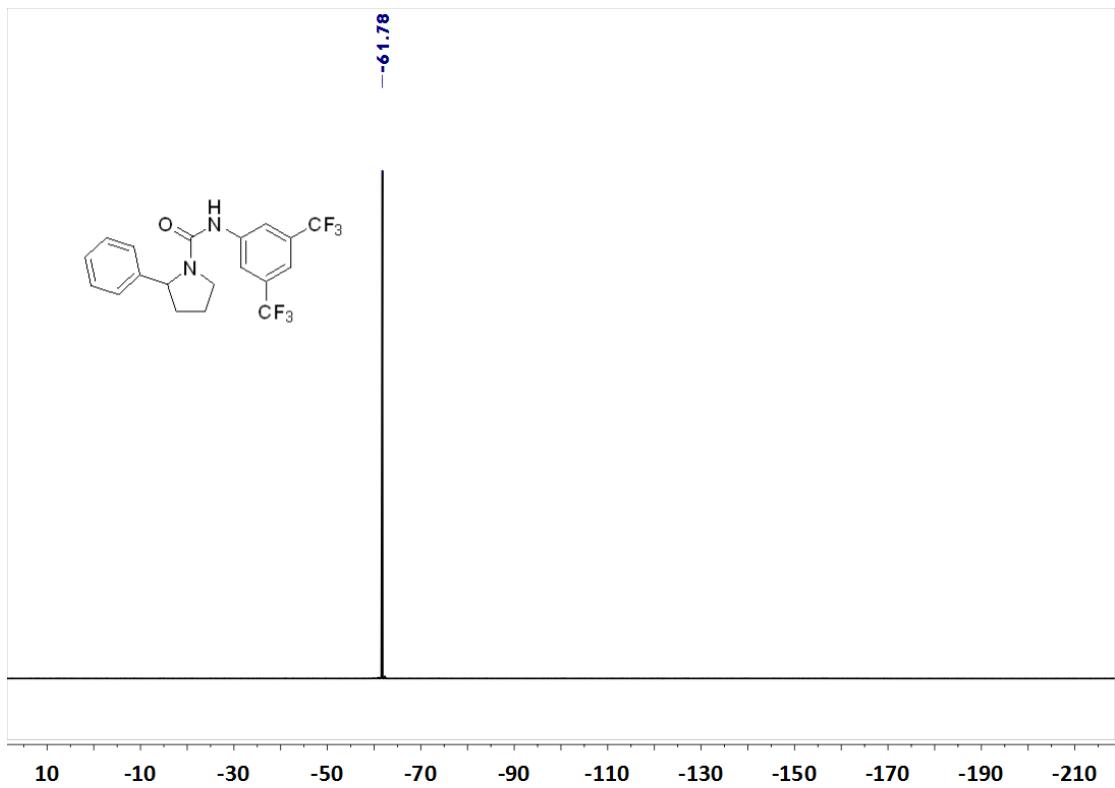
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **1u**.



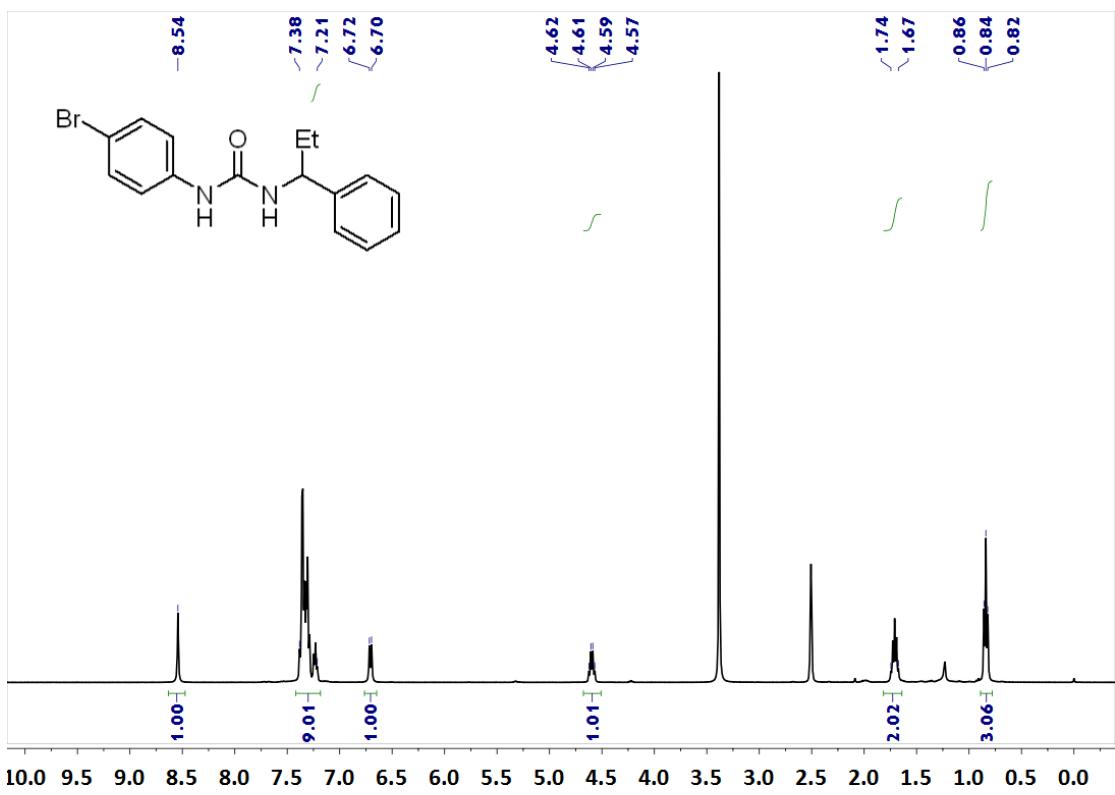


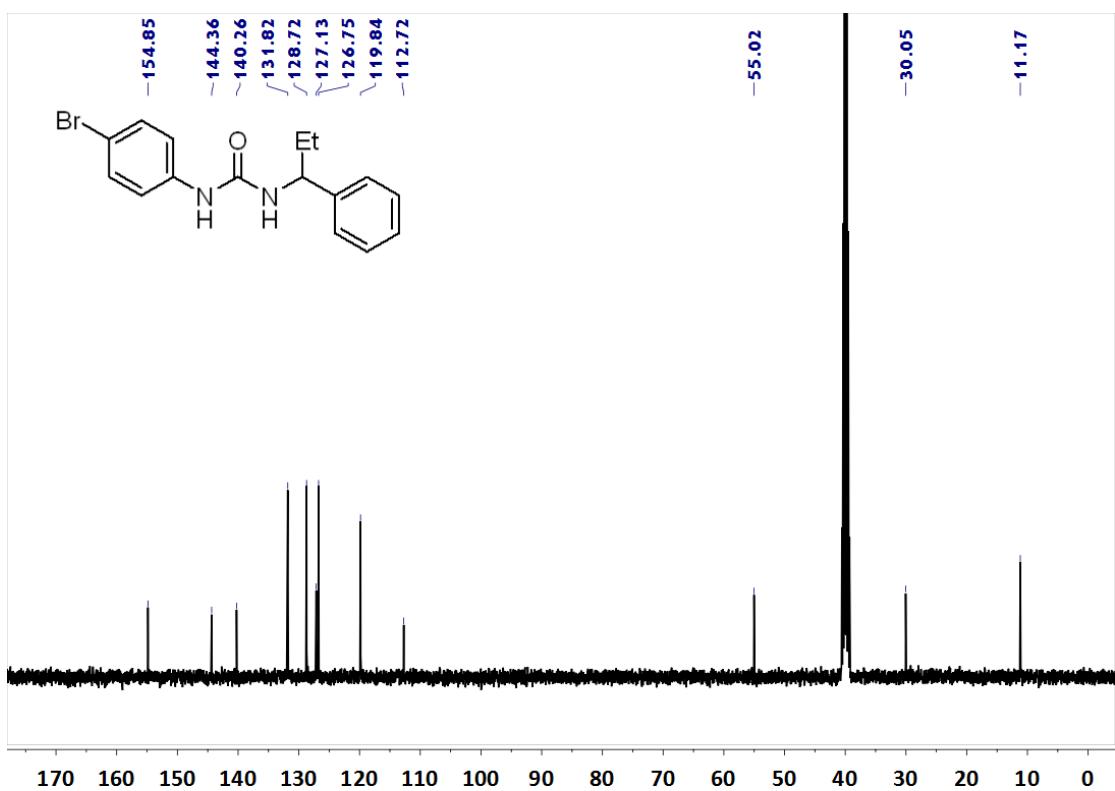
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **1v**.



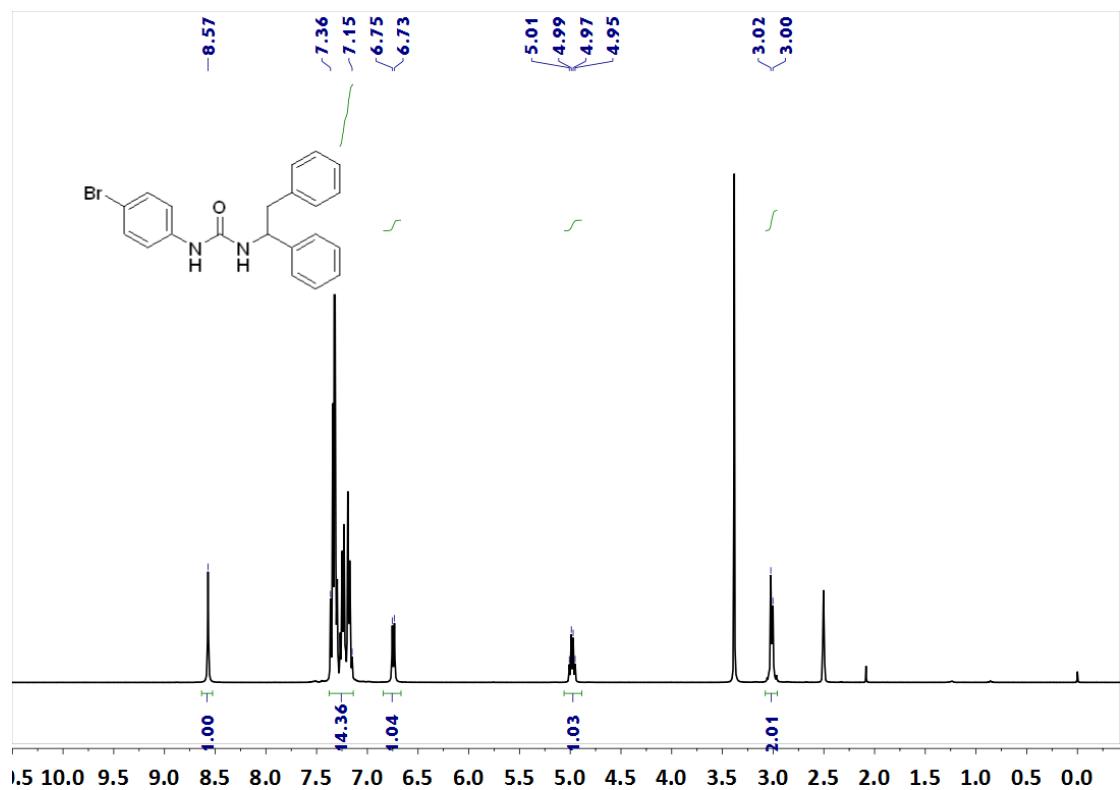


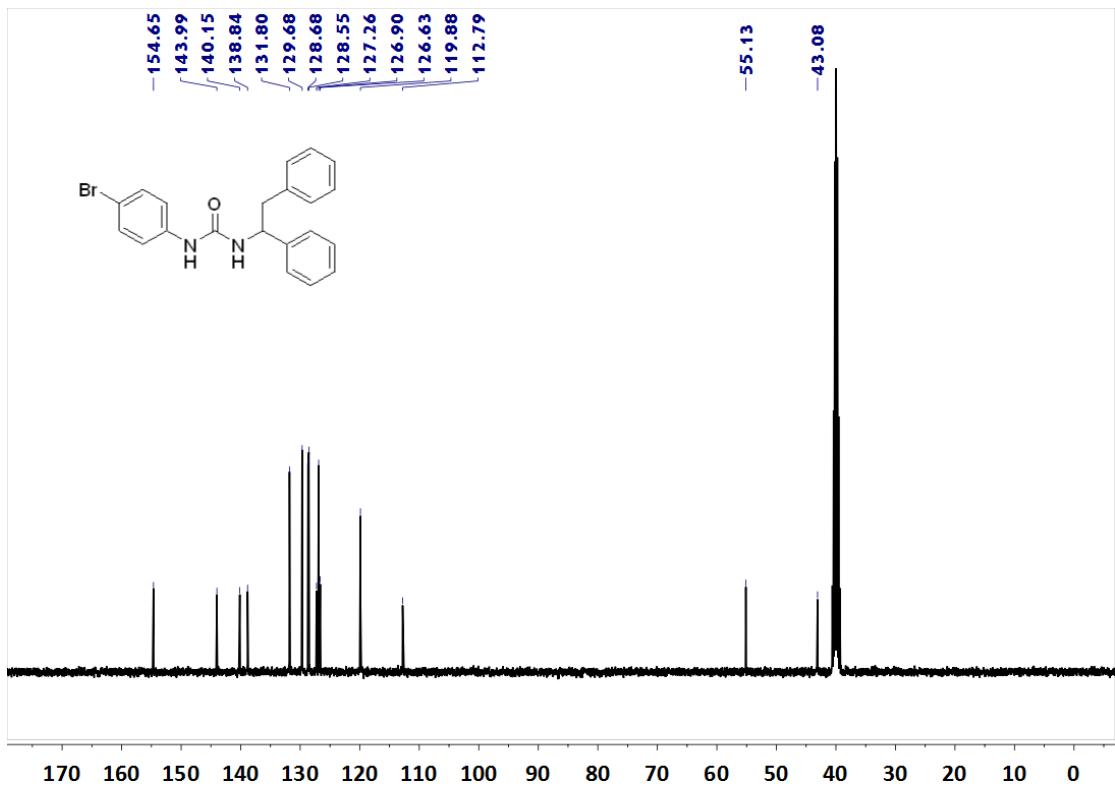
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **3a**.



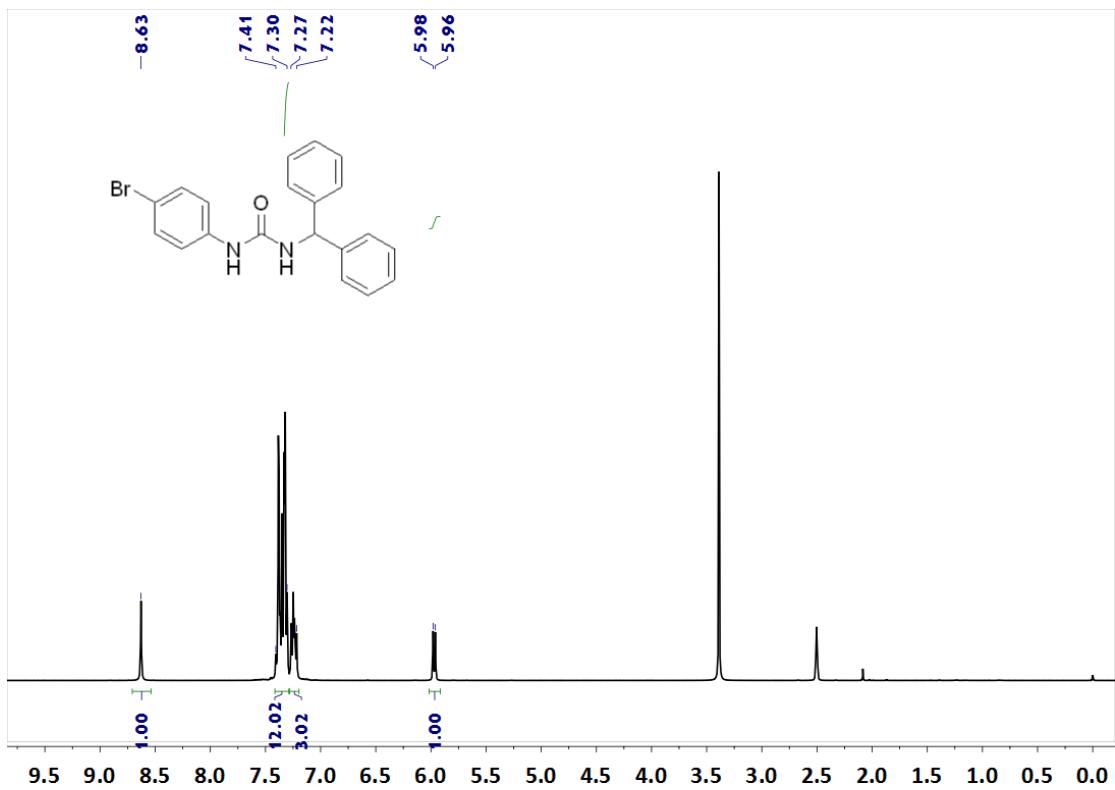


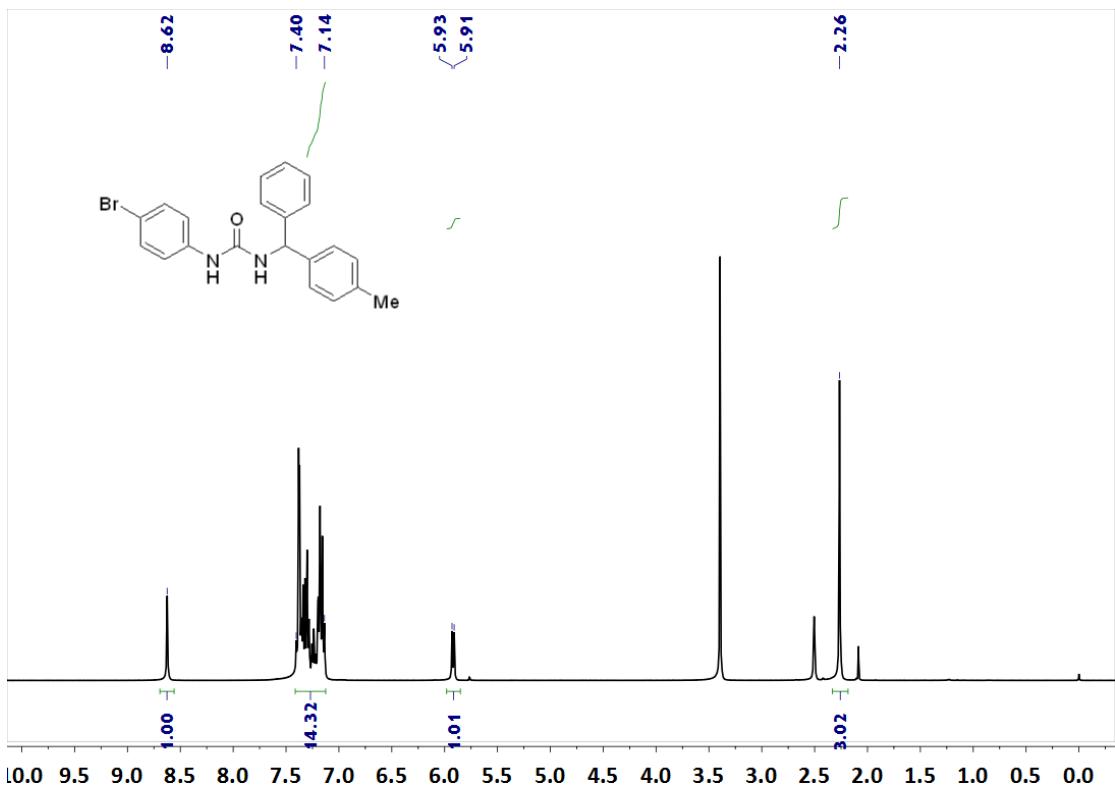
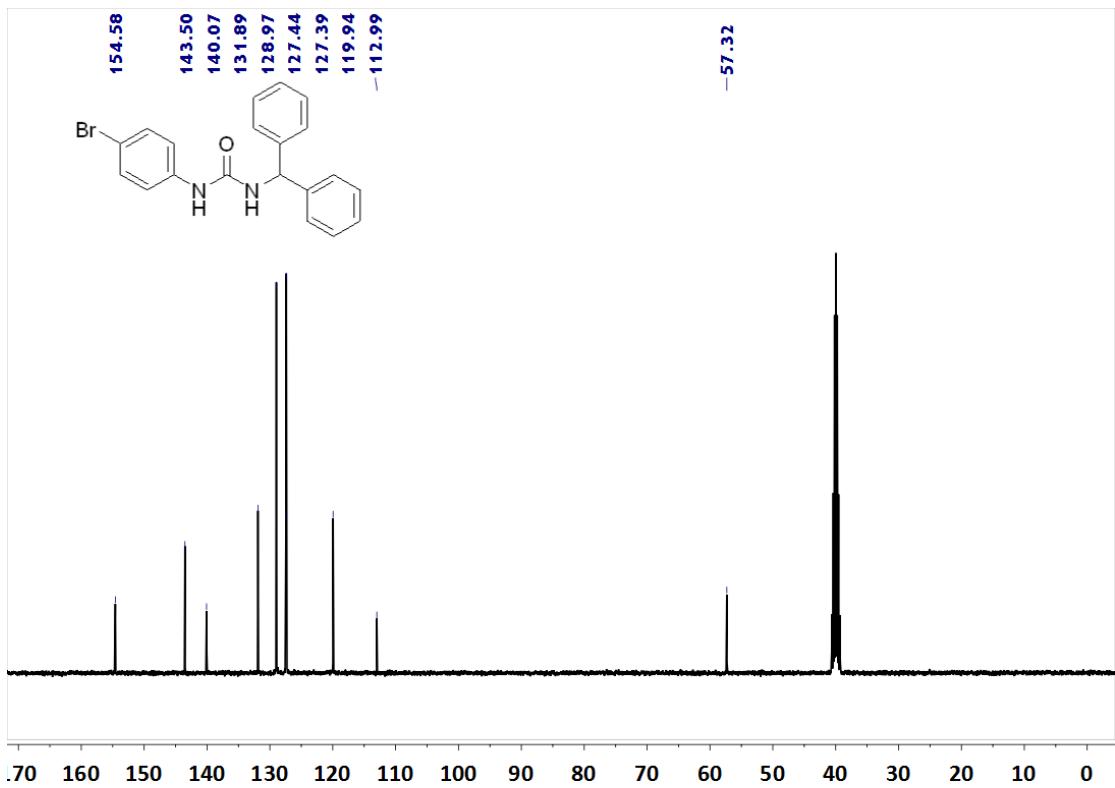
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **3b**.

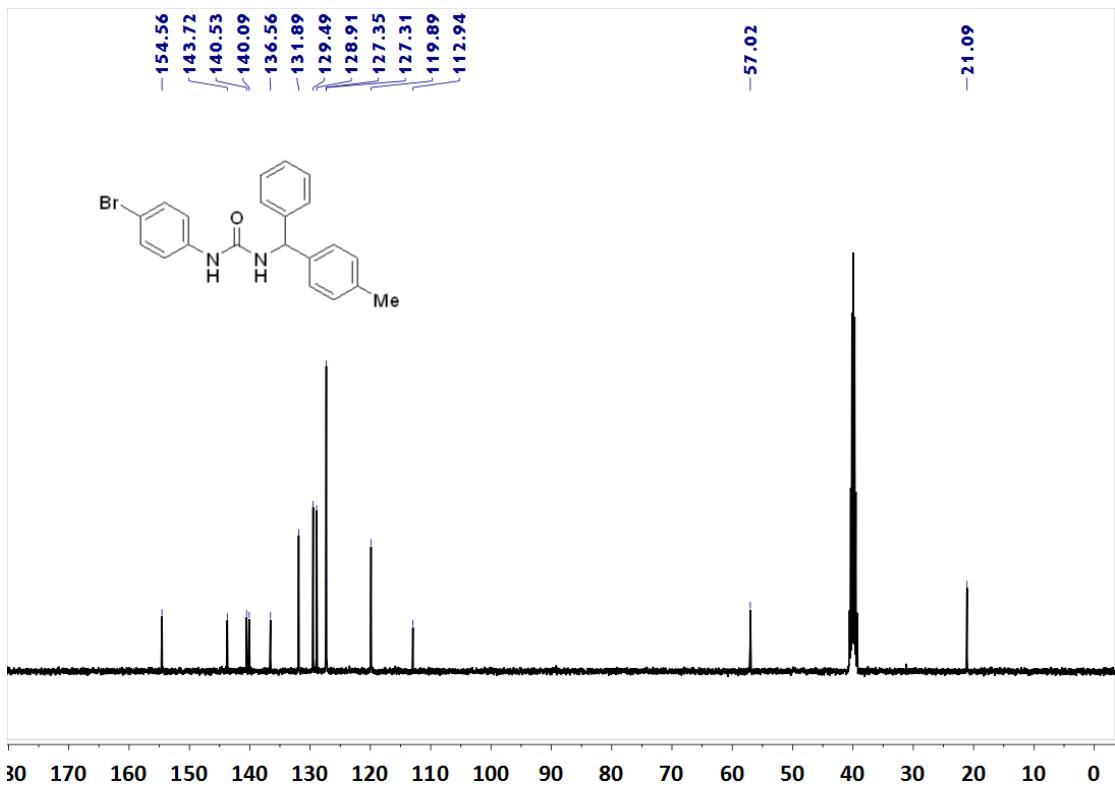




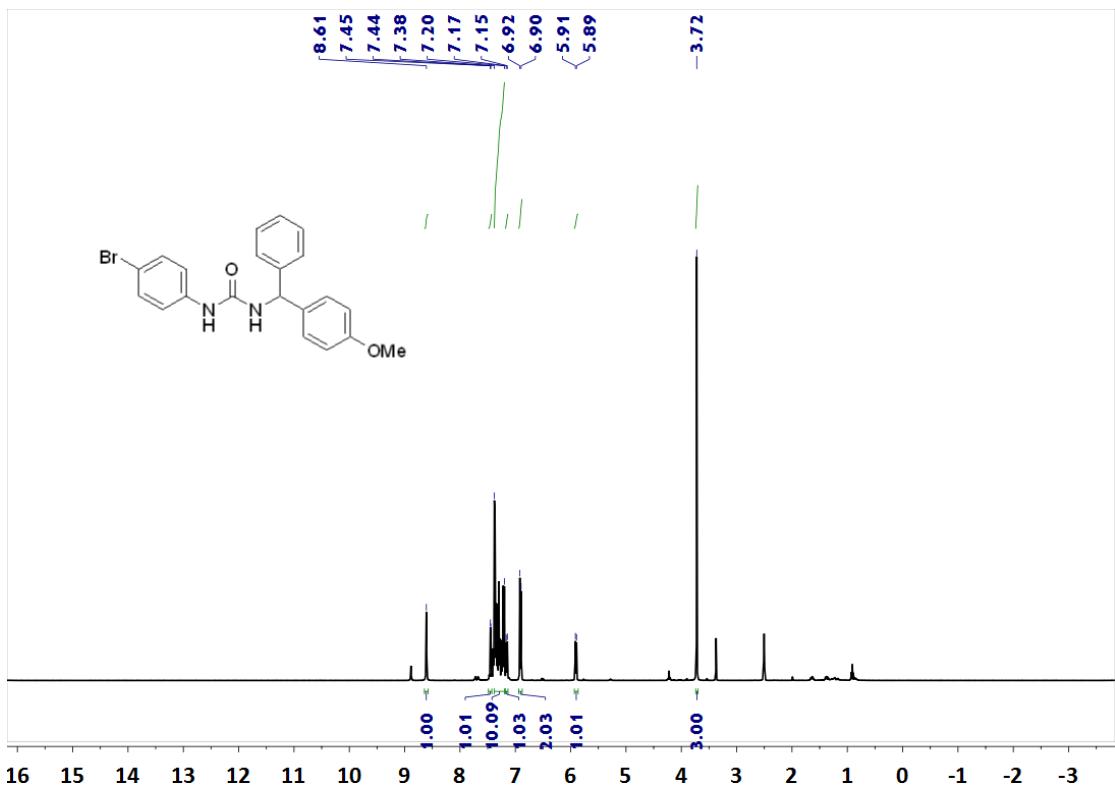
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3c.

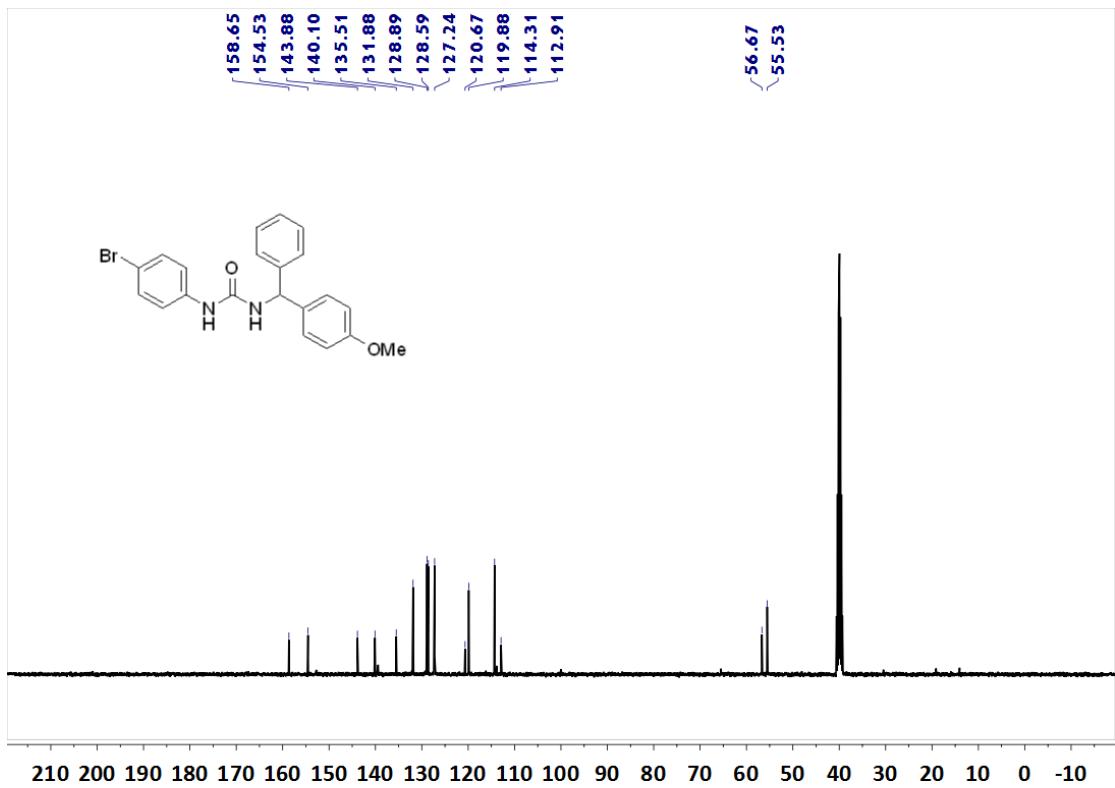




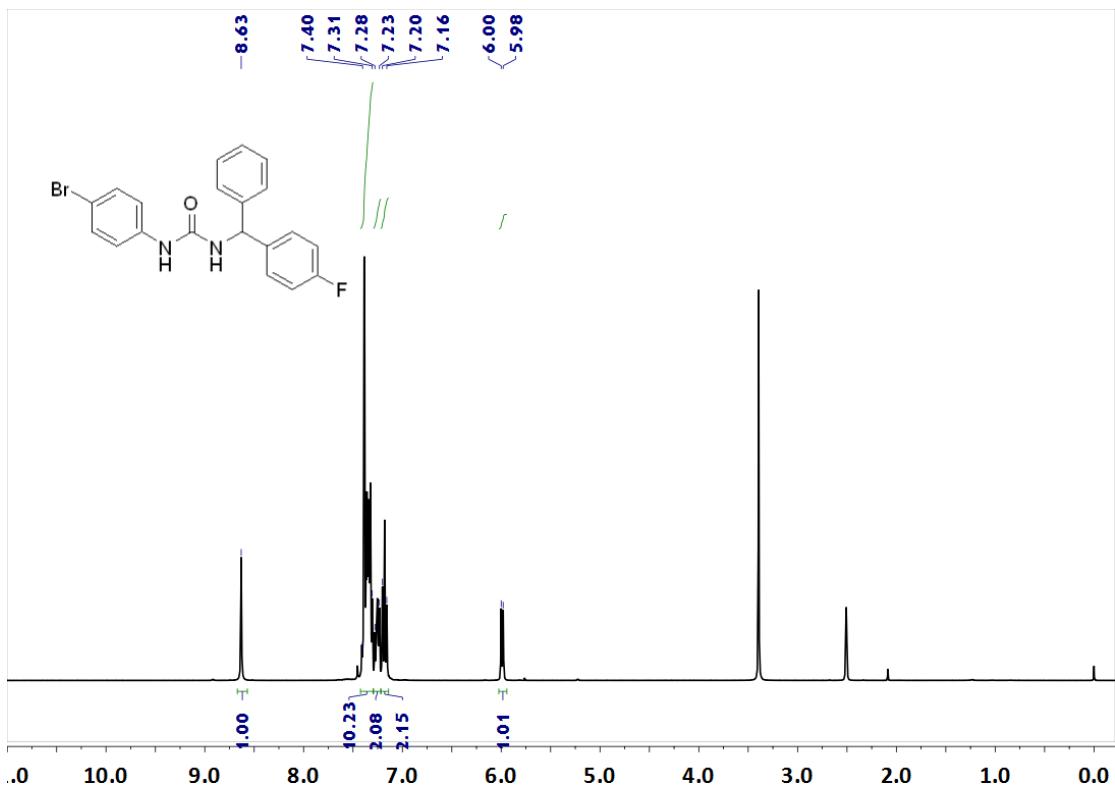


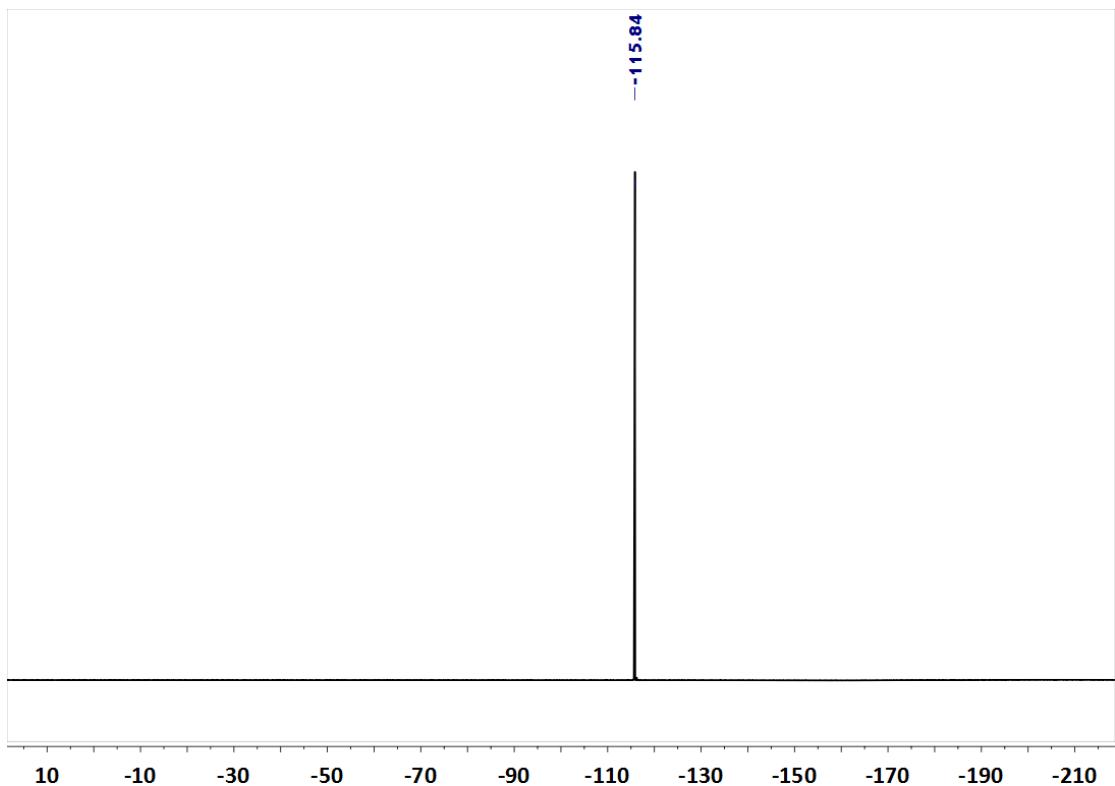
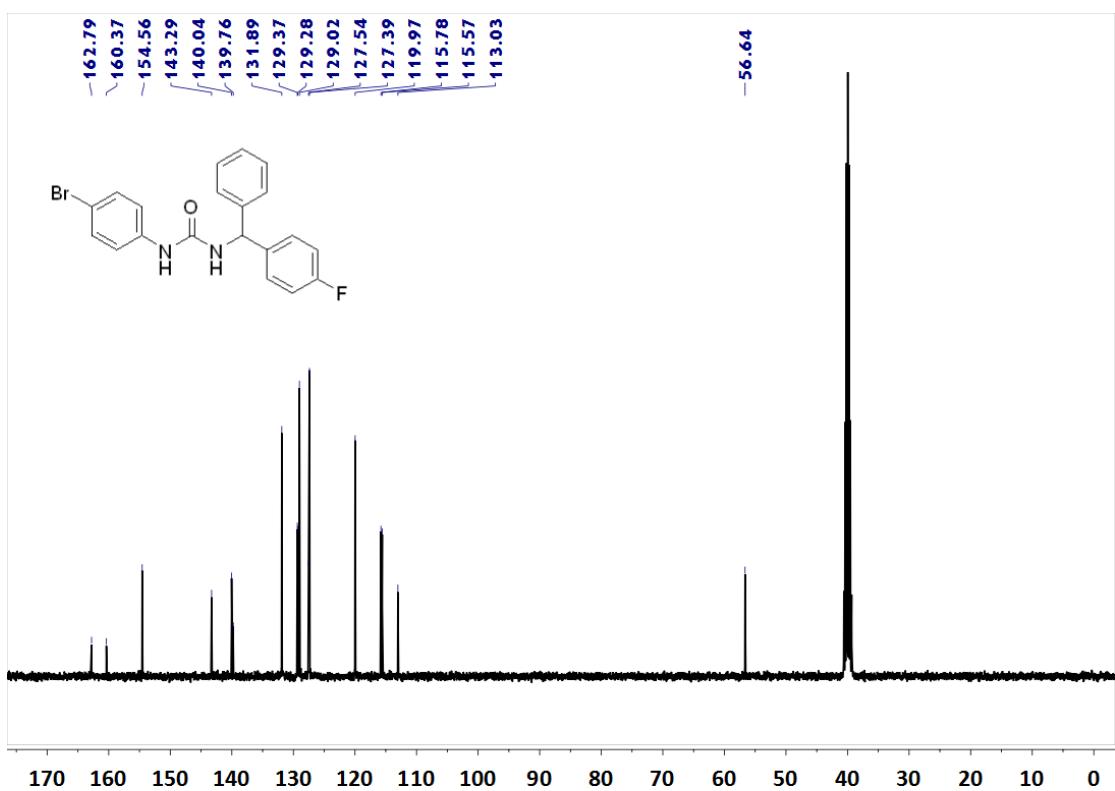
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3e.



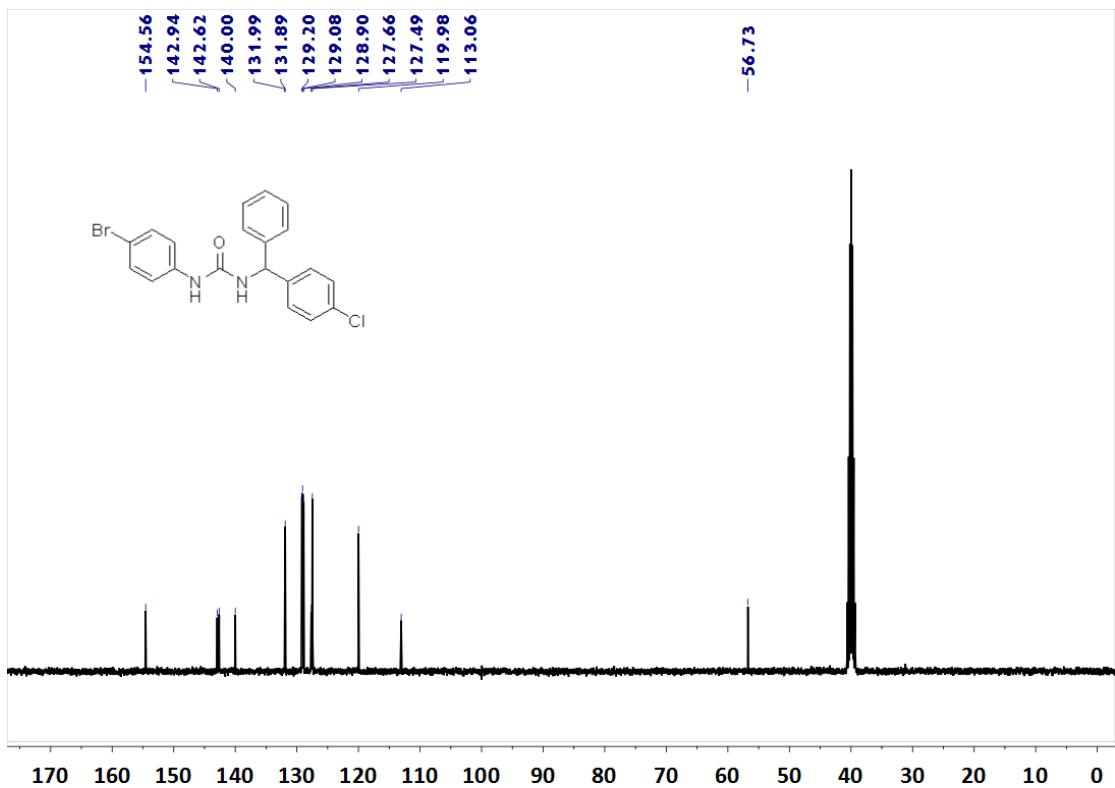
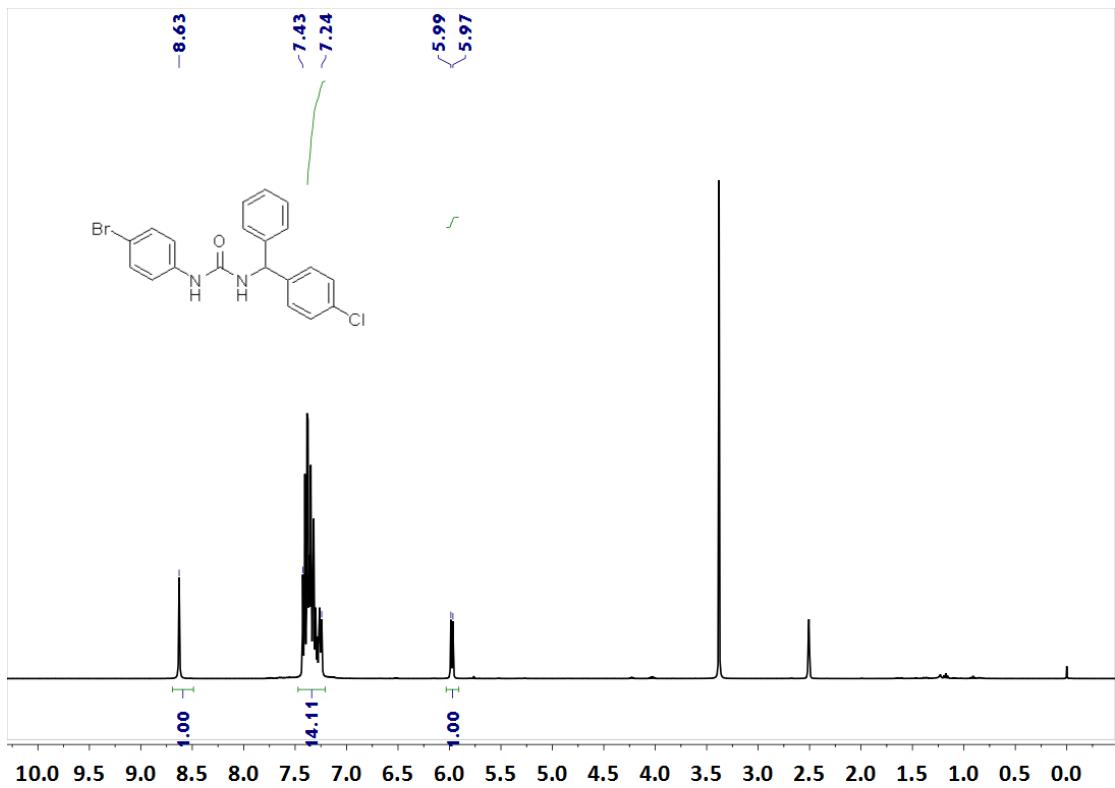


$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **3f**.

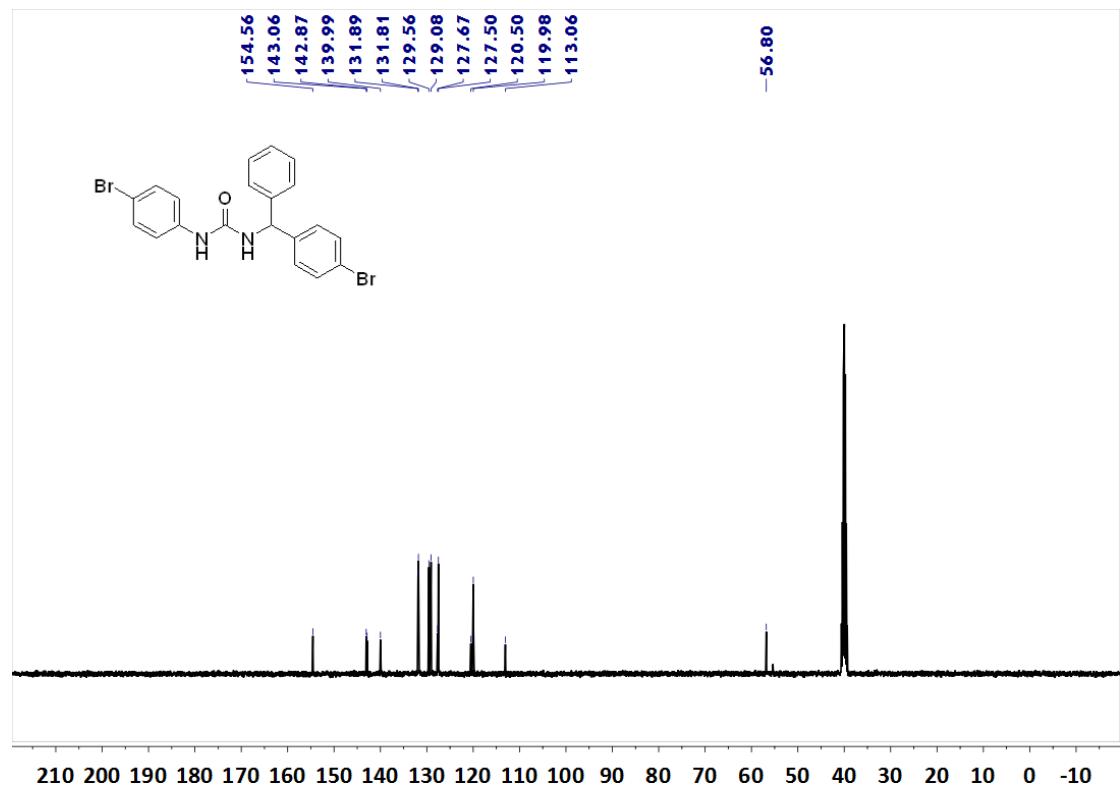
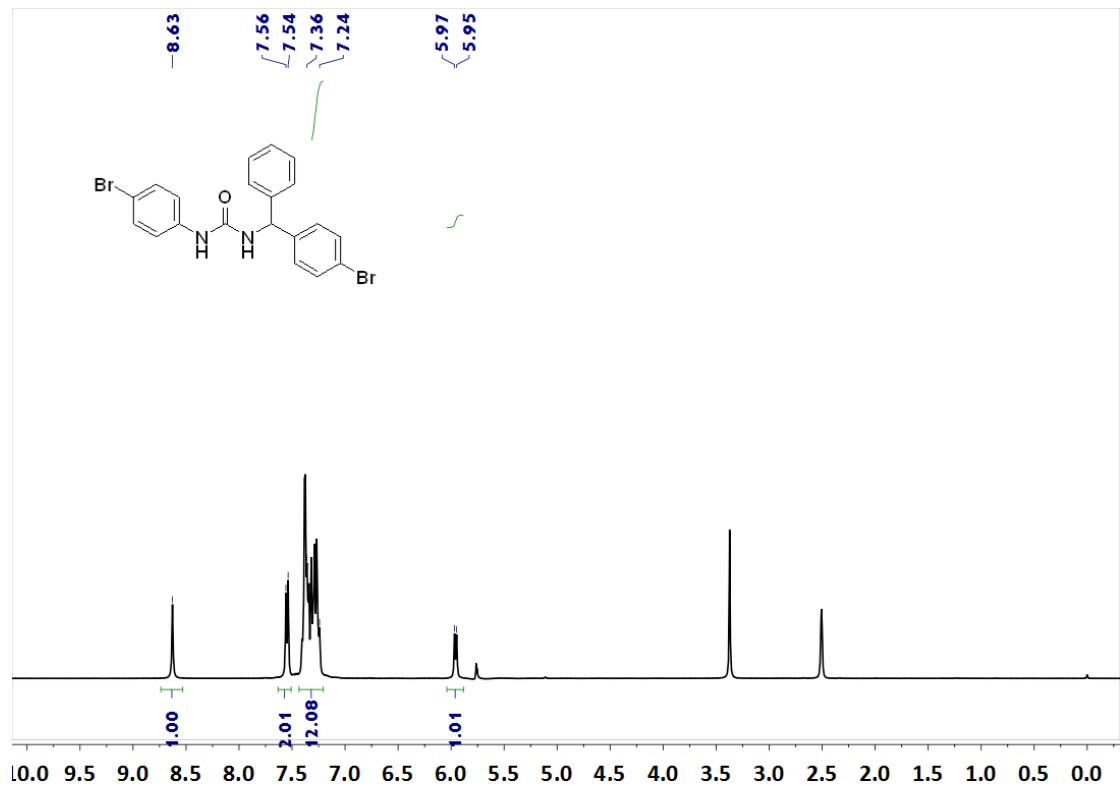




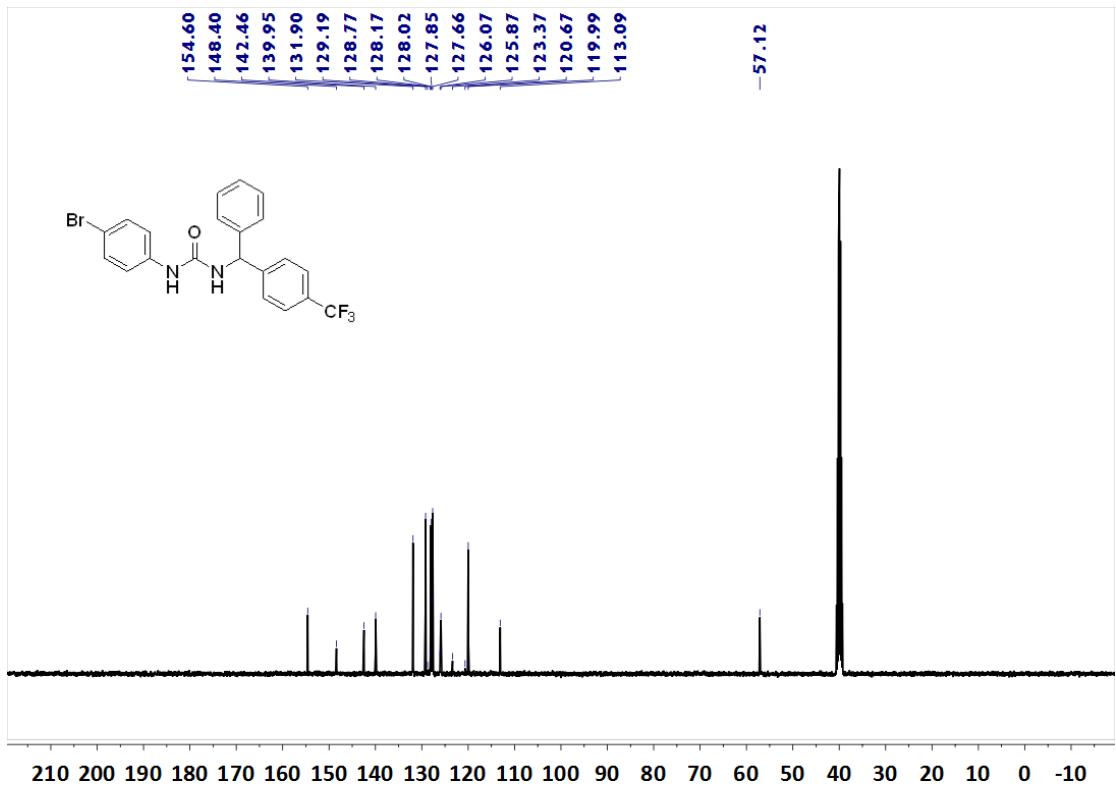
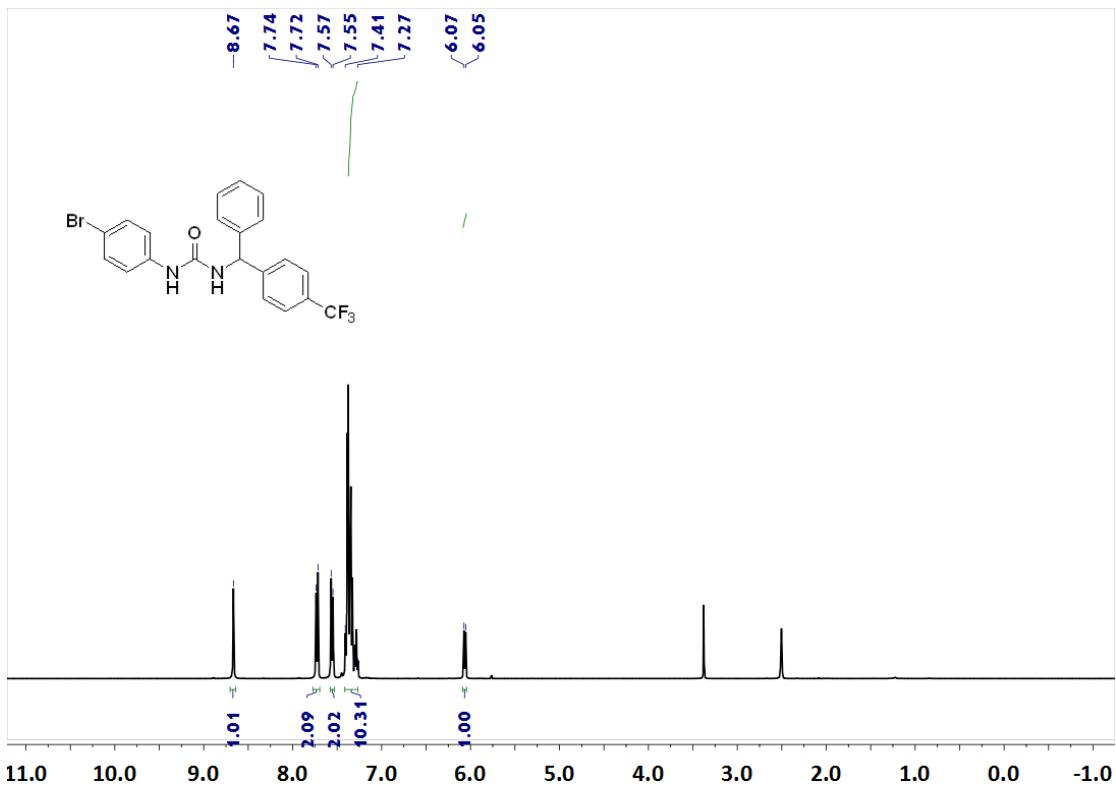
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3g.

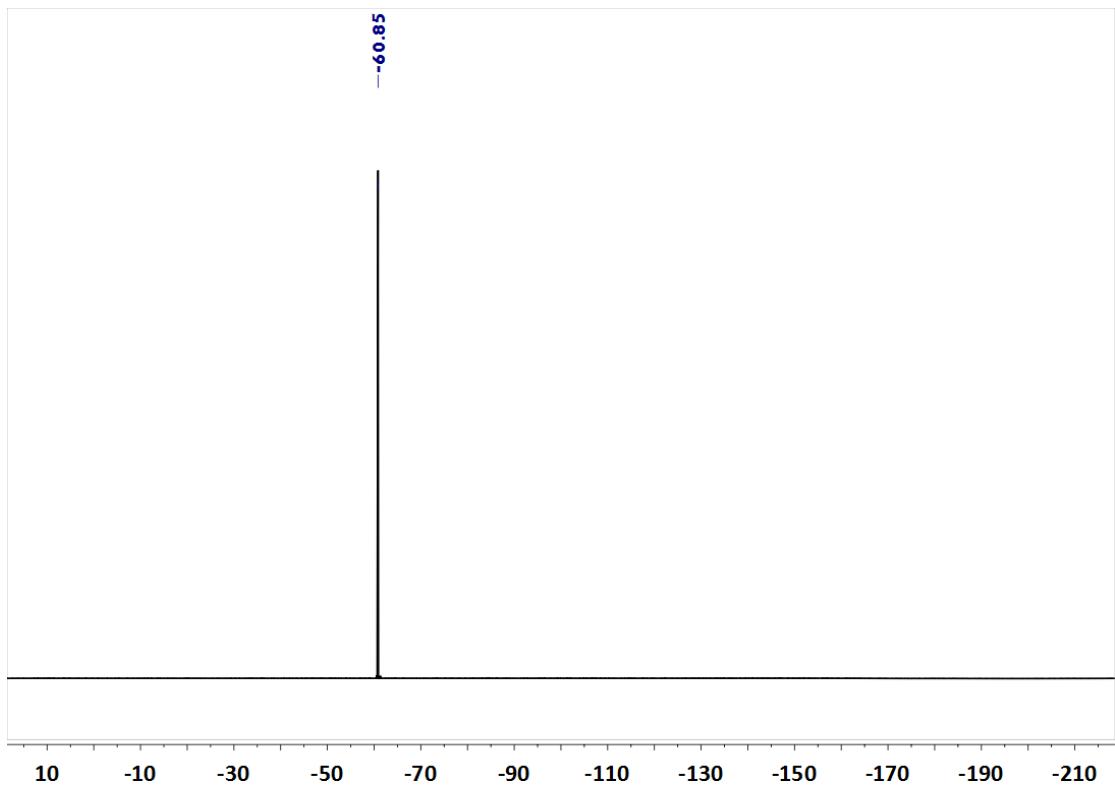


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3h.

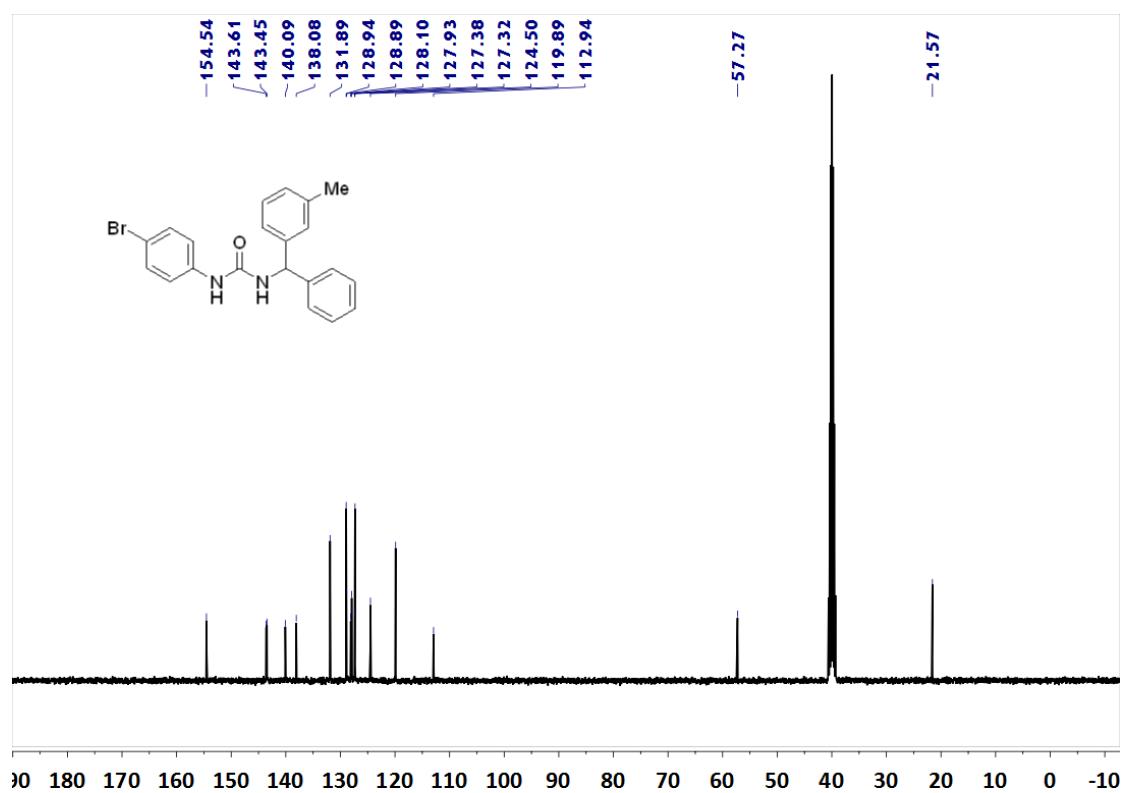


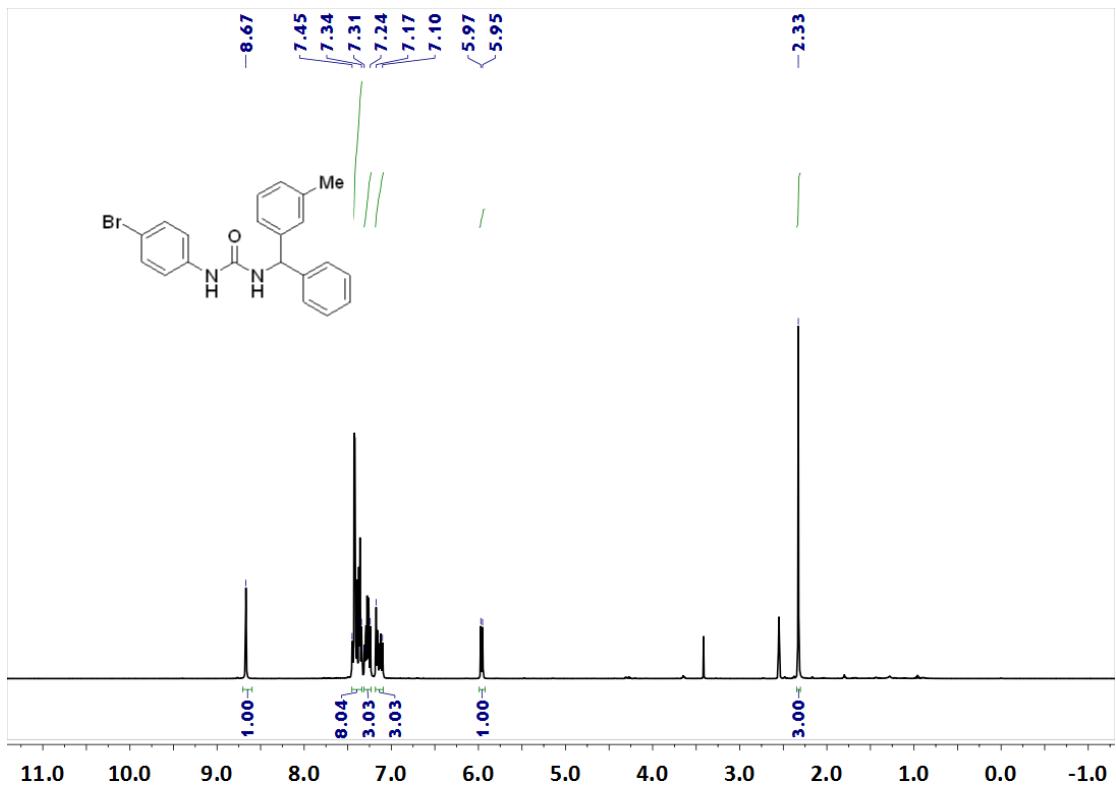
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3i.



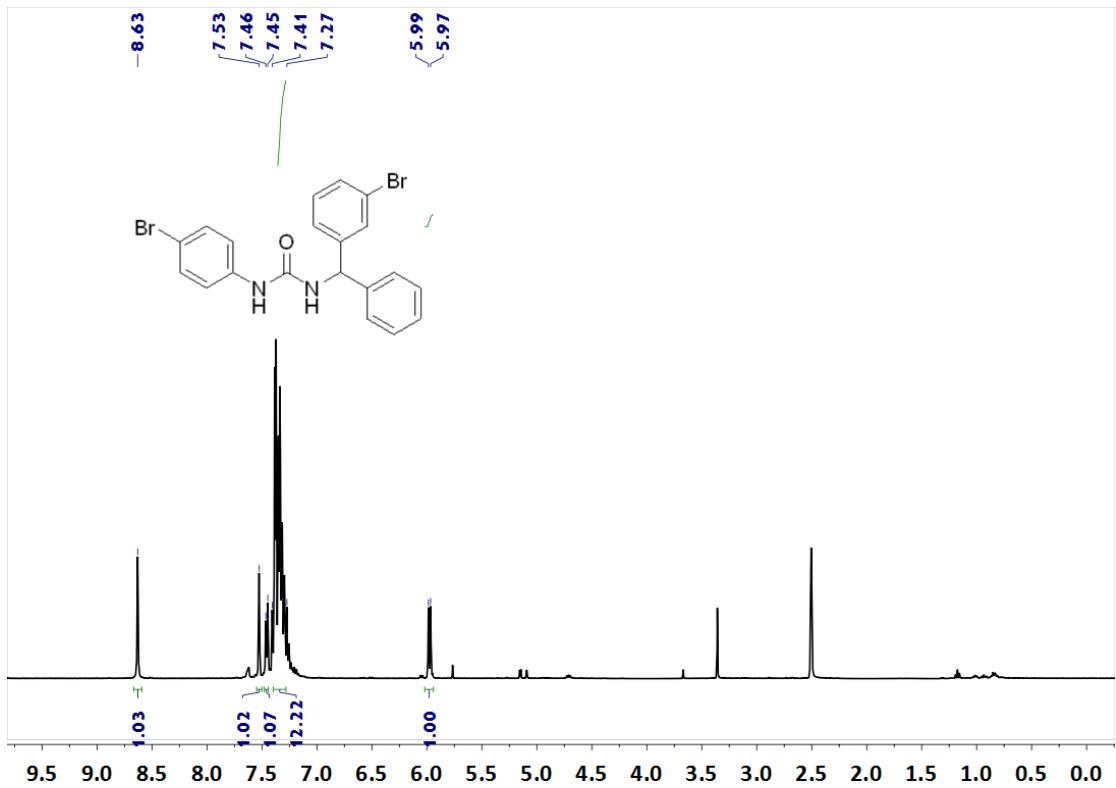


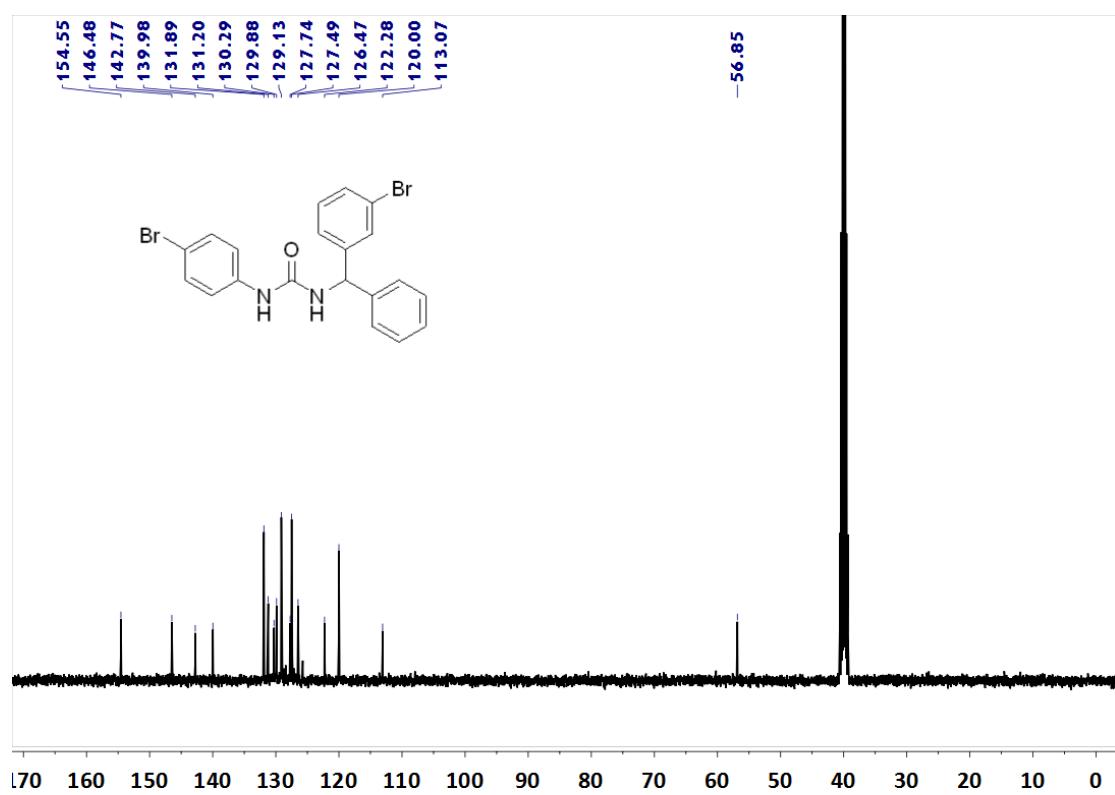
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **3j**.



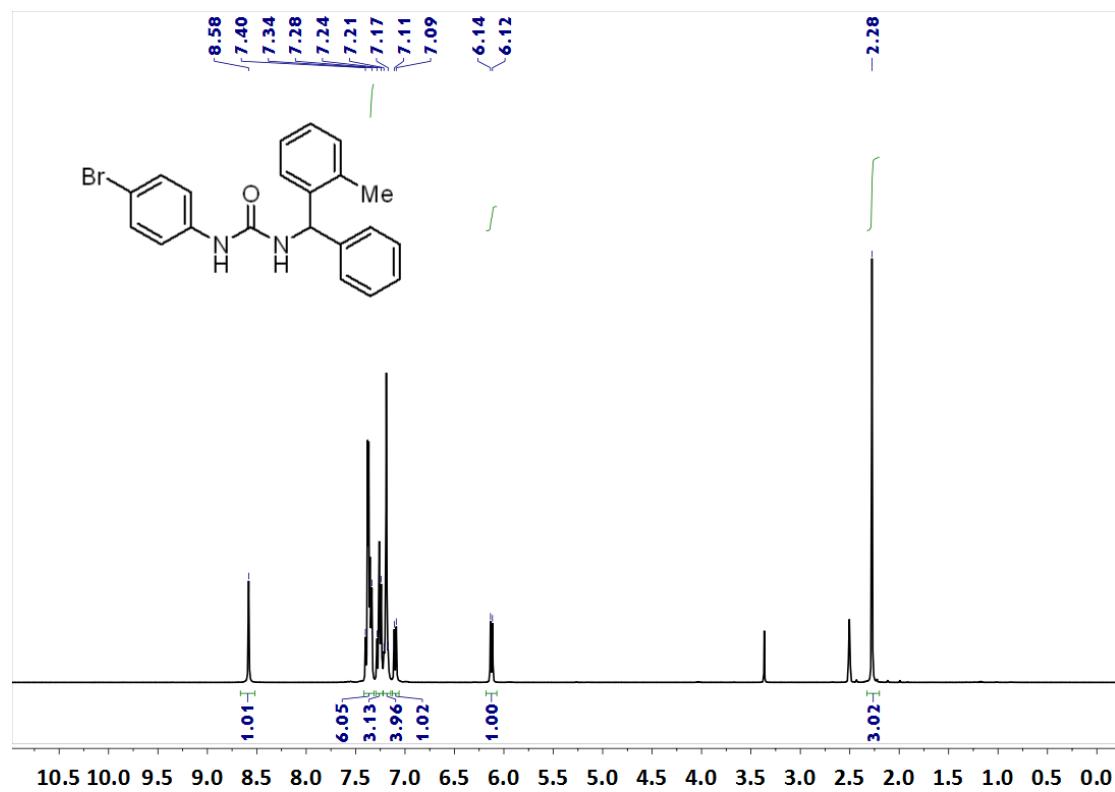


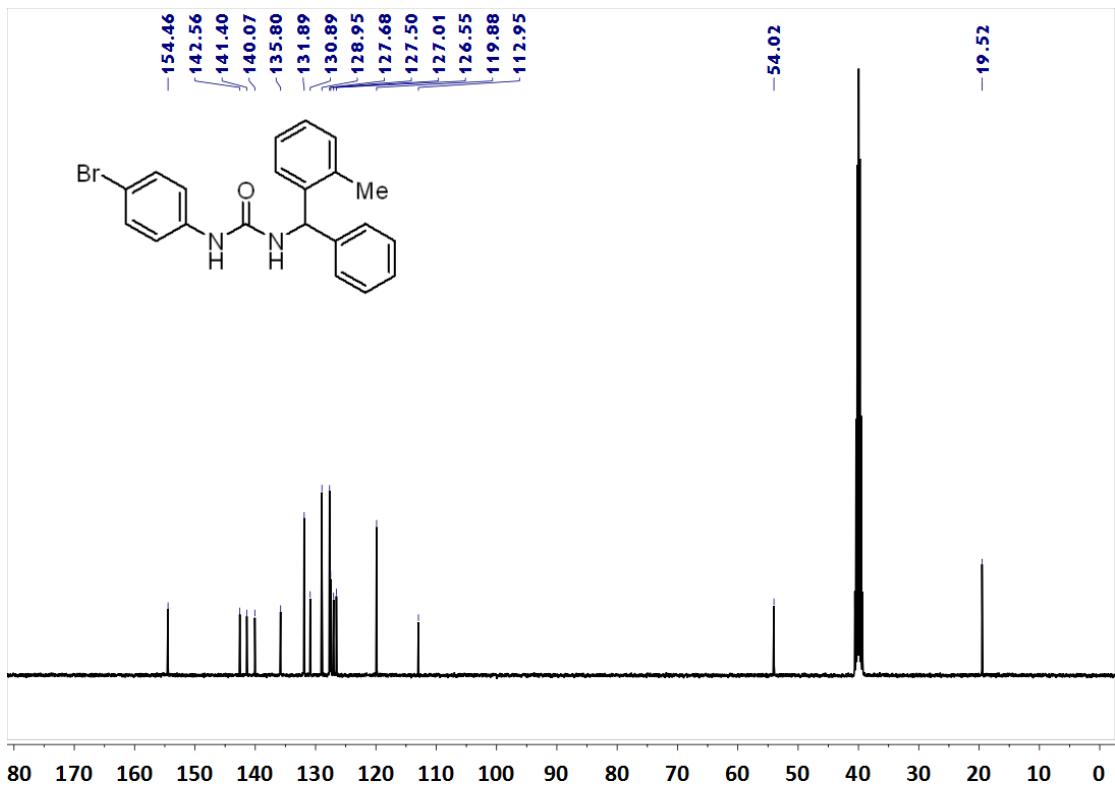
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3k.



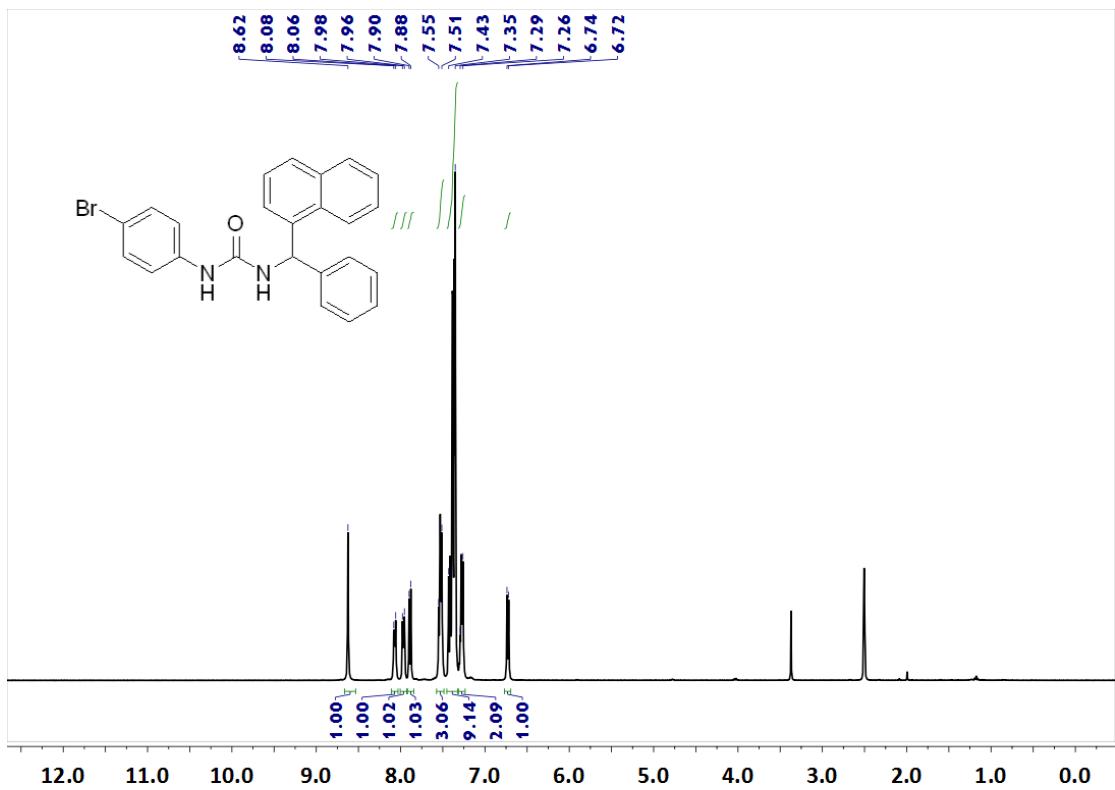


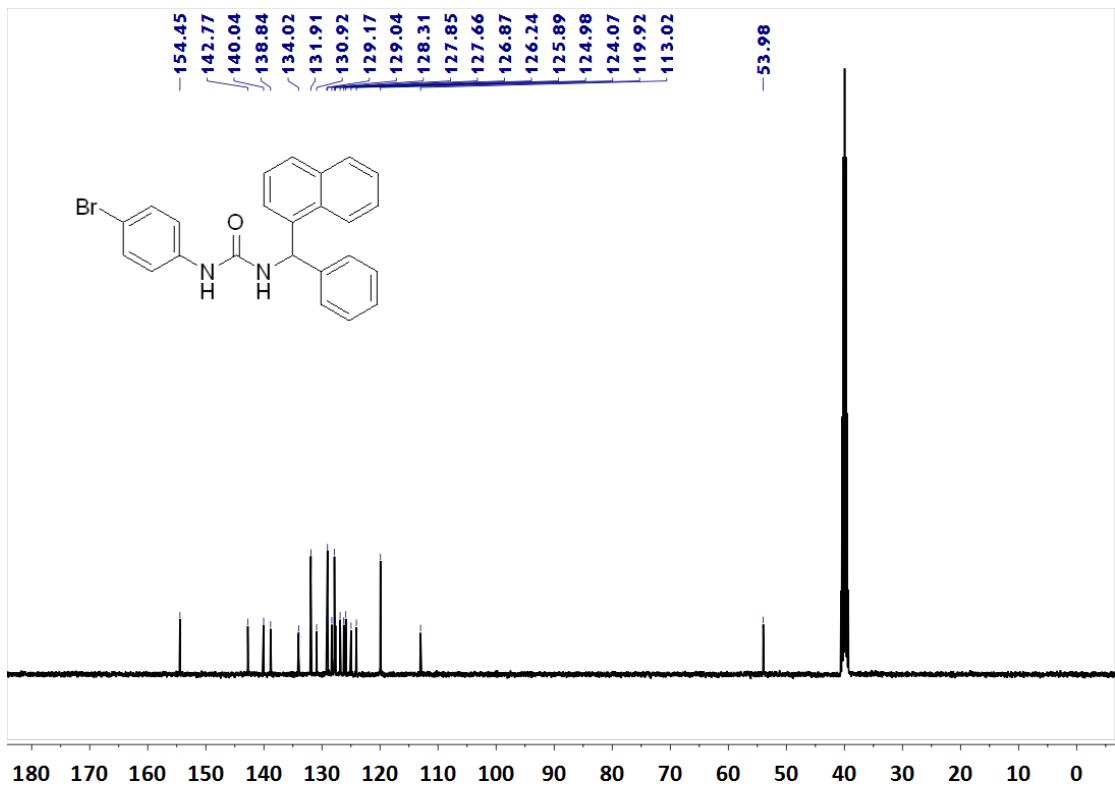
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3l.



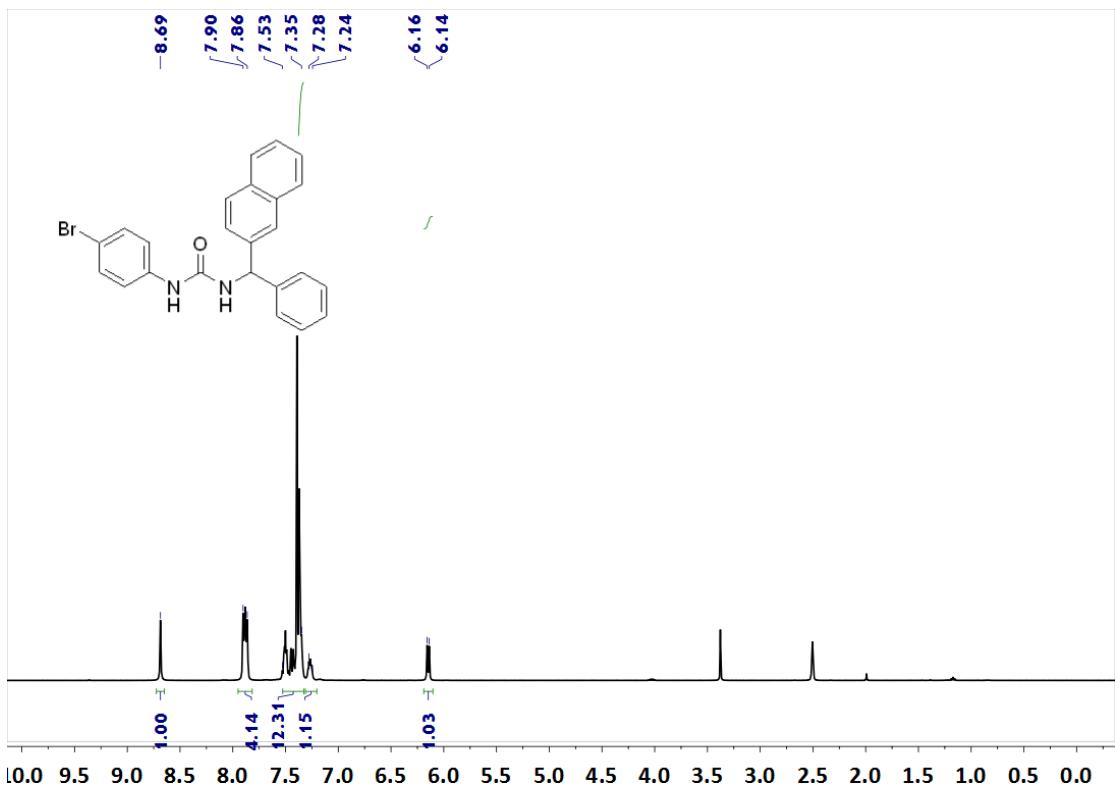


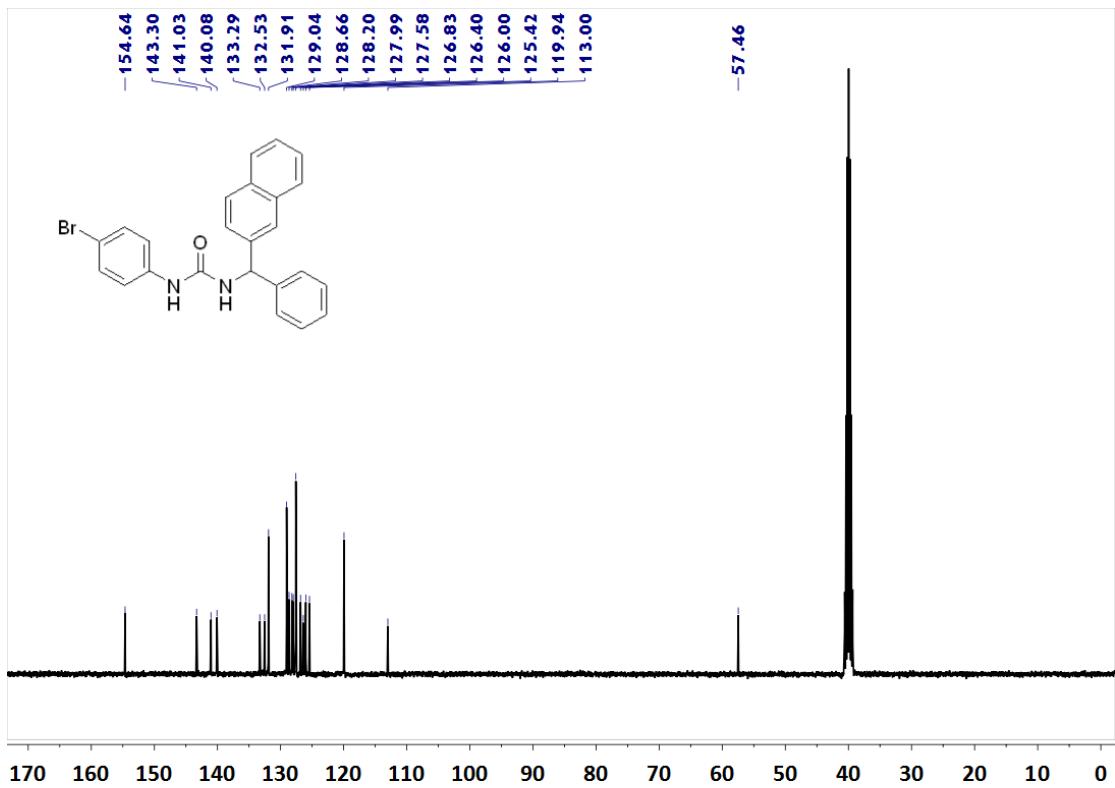
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **3m**.



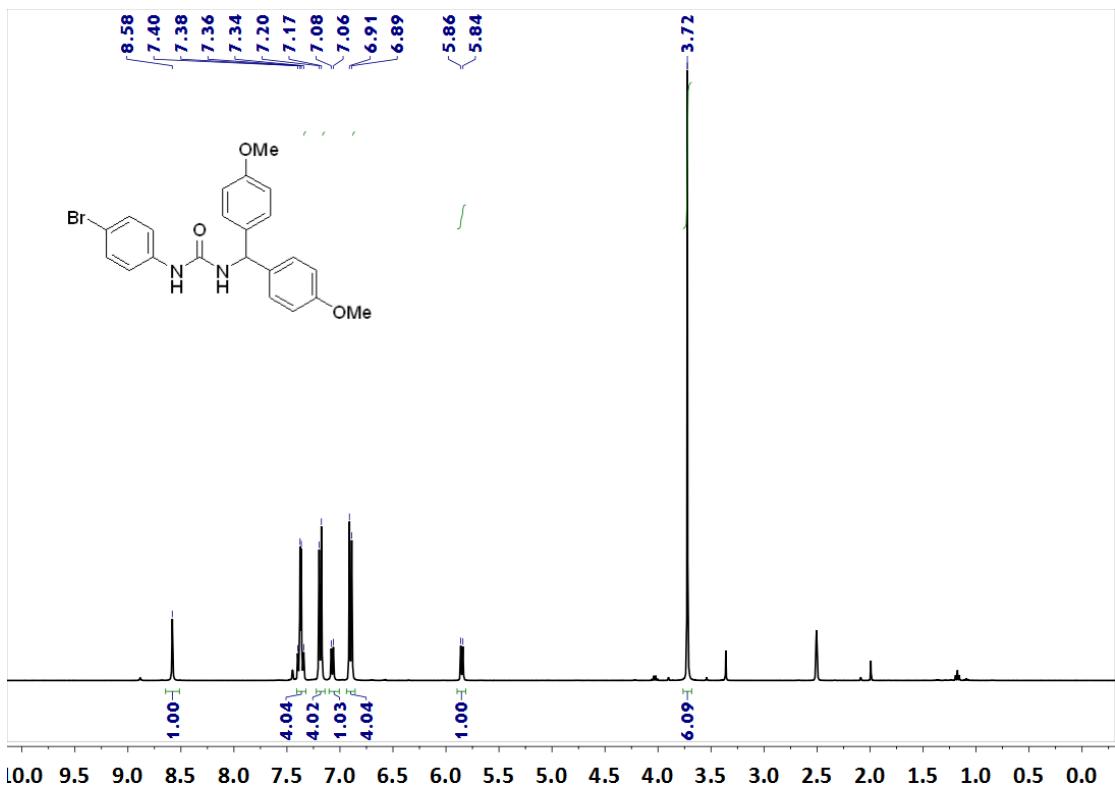


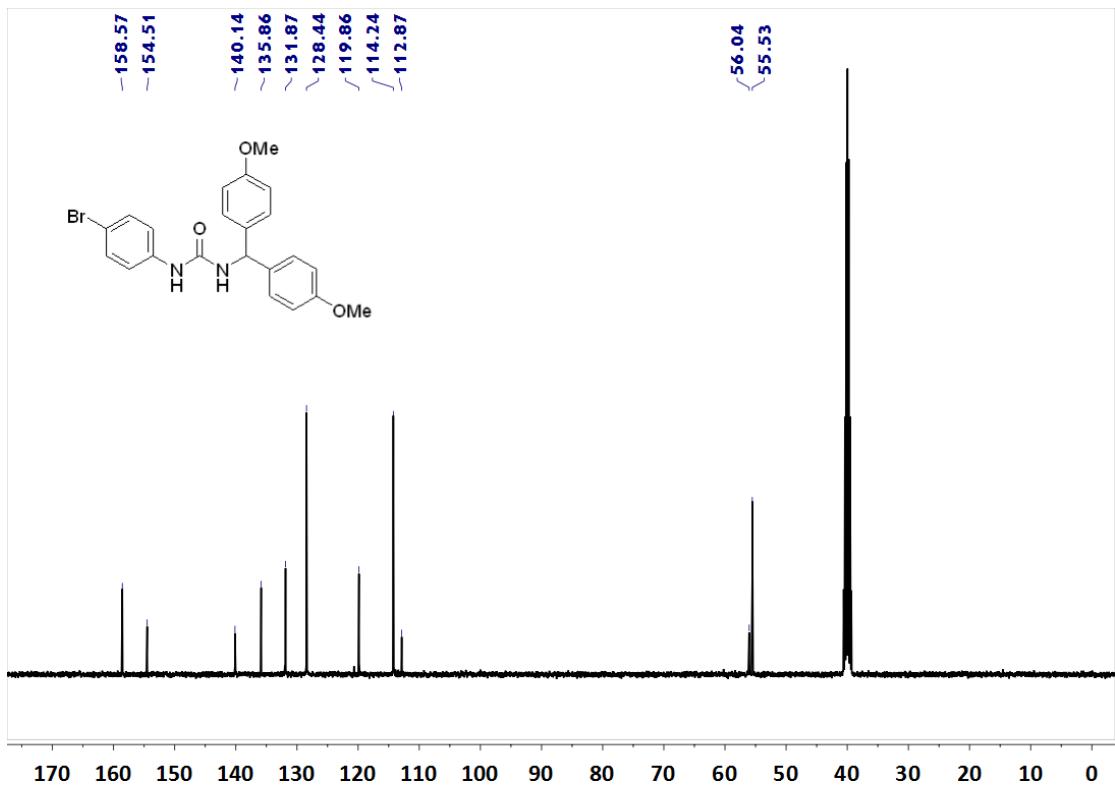
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **3n**.



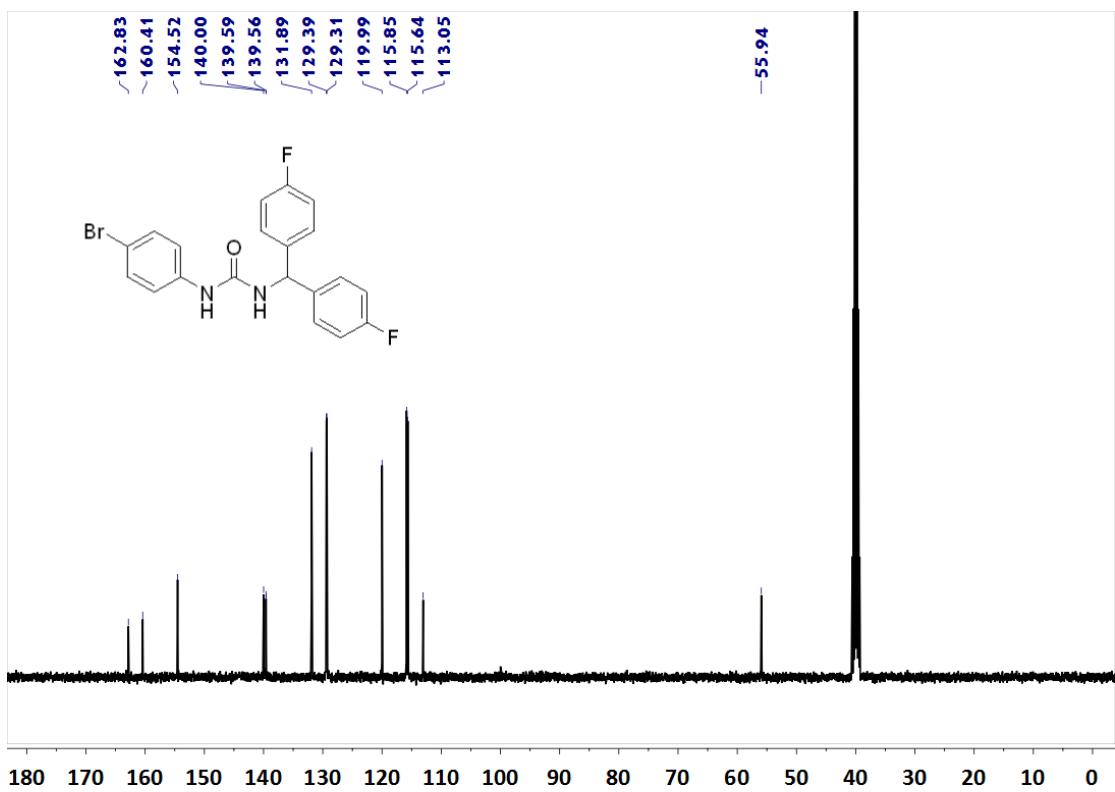


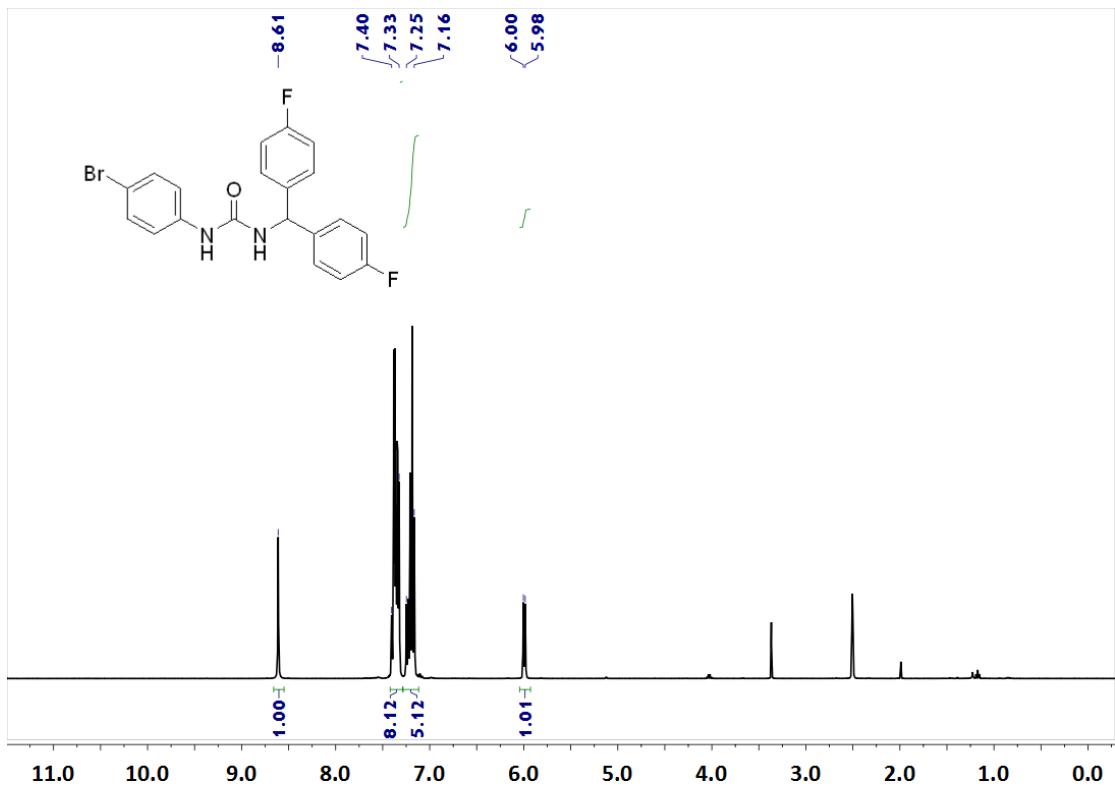
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **3o**.



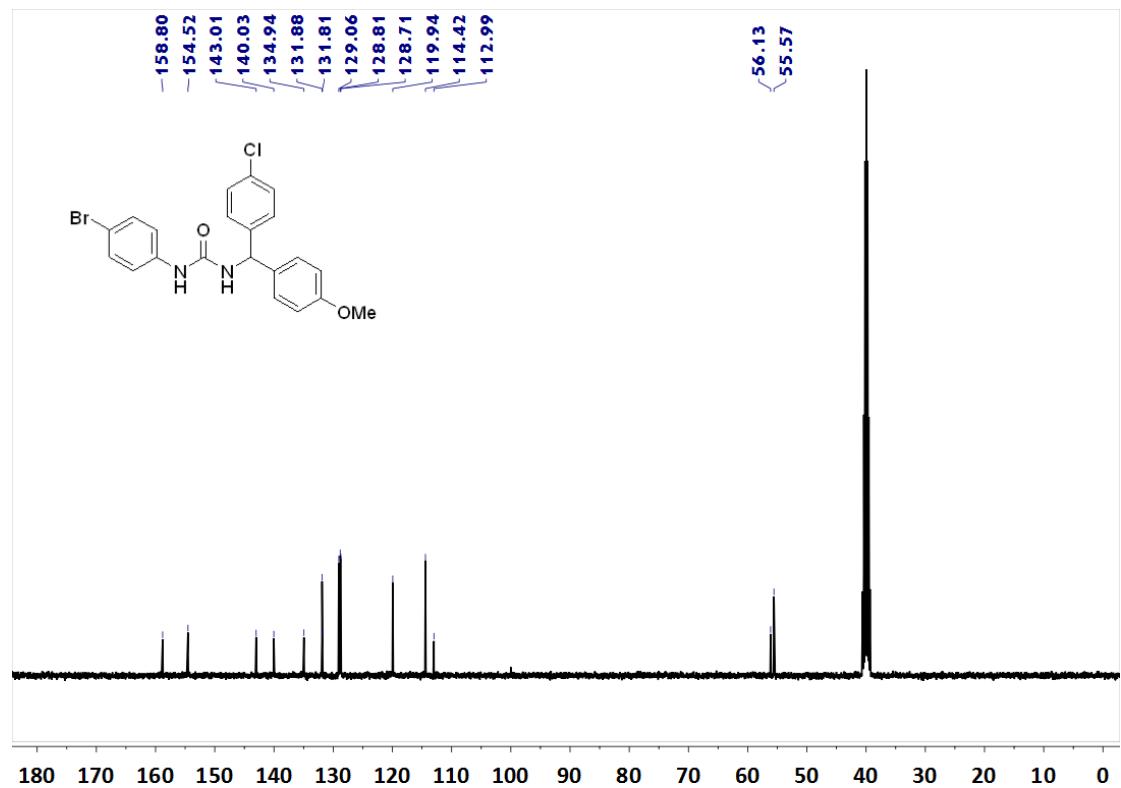
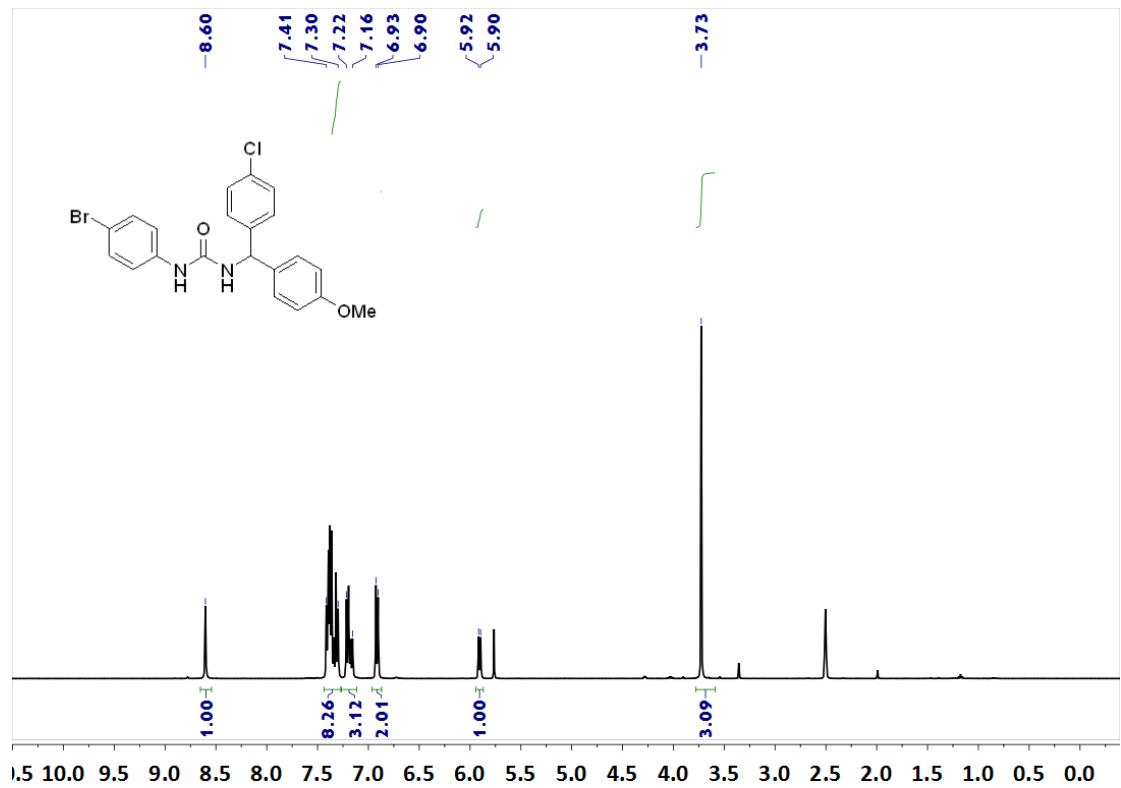


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 3p.

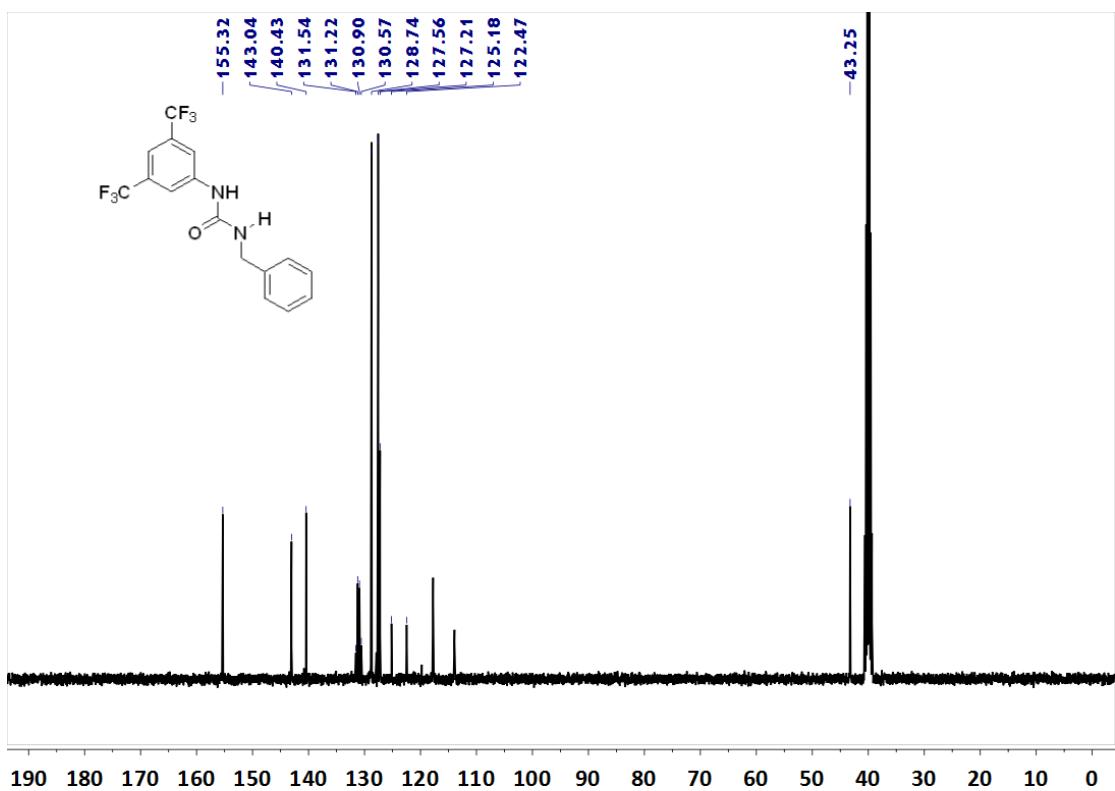
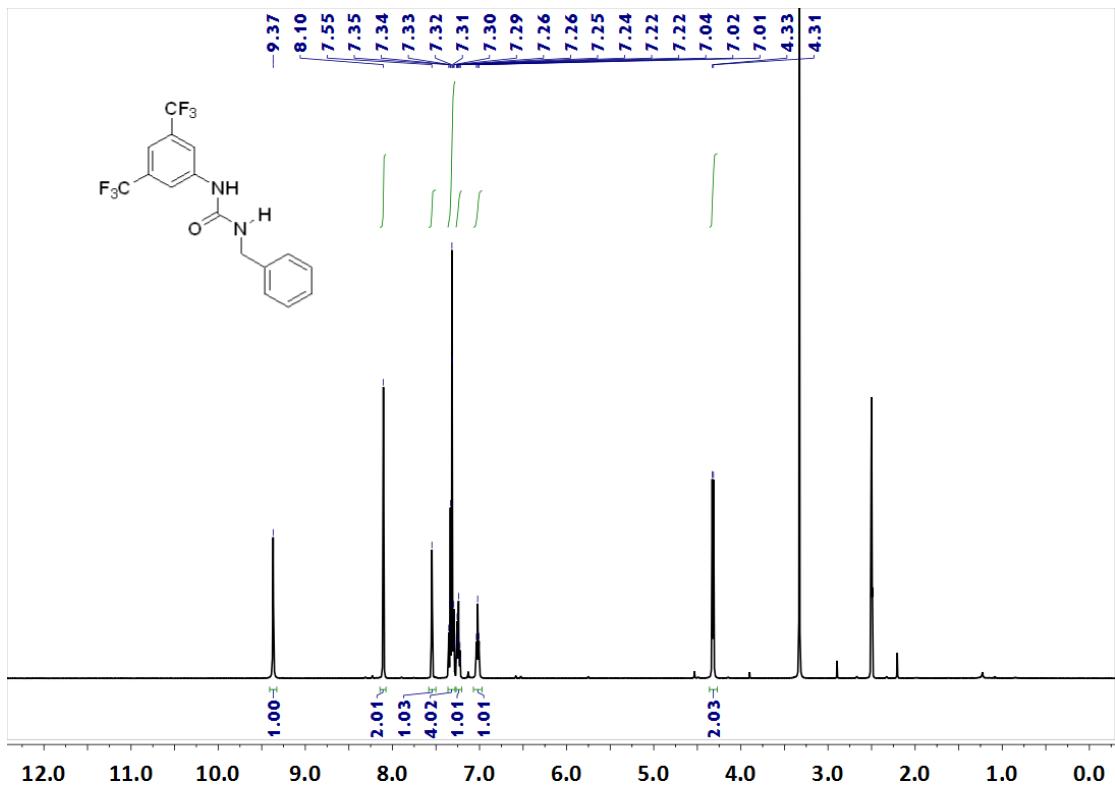


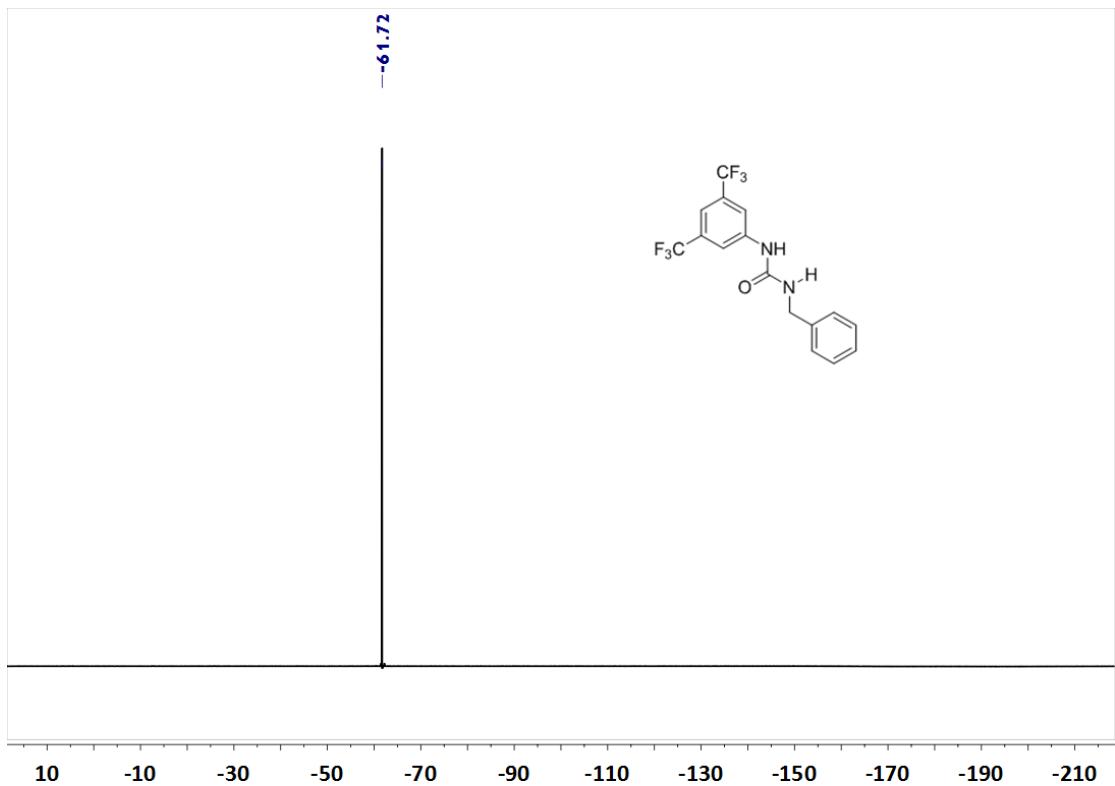


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **3q**.

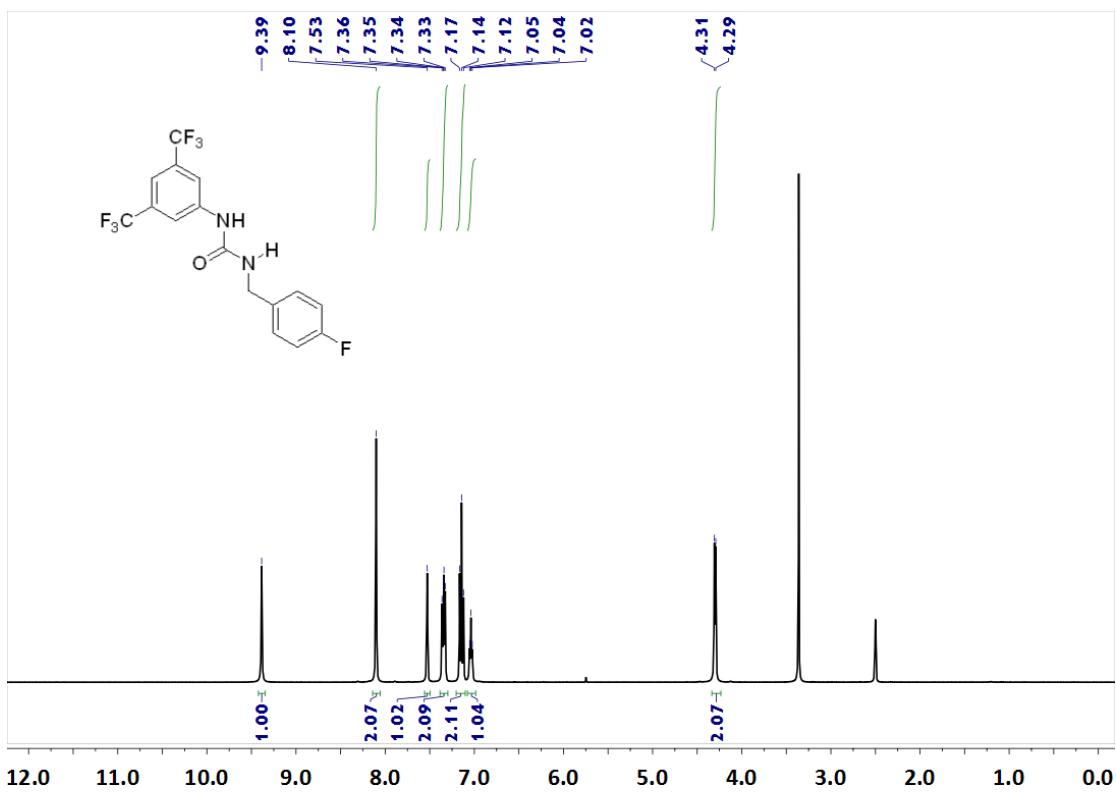


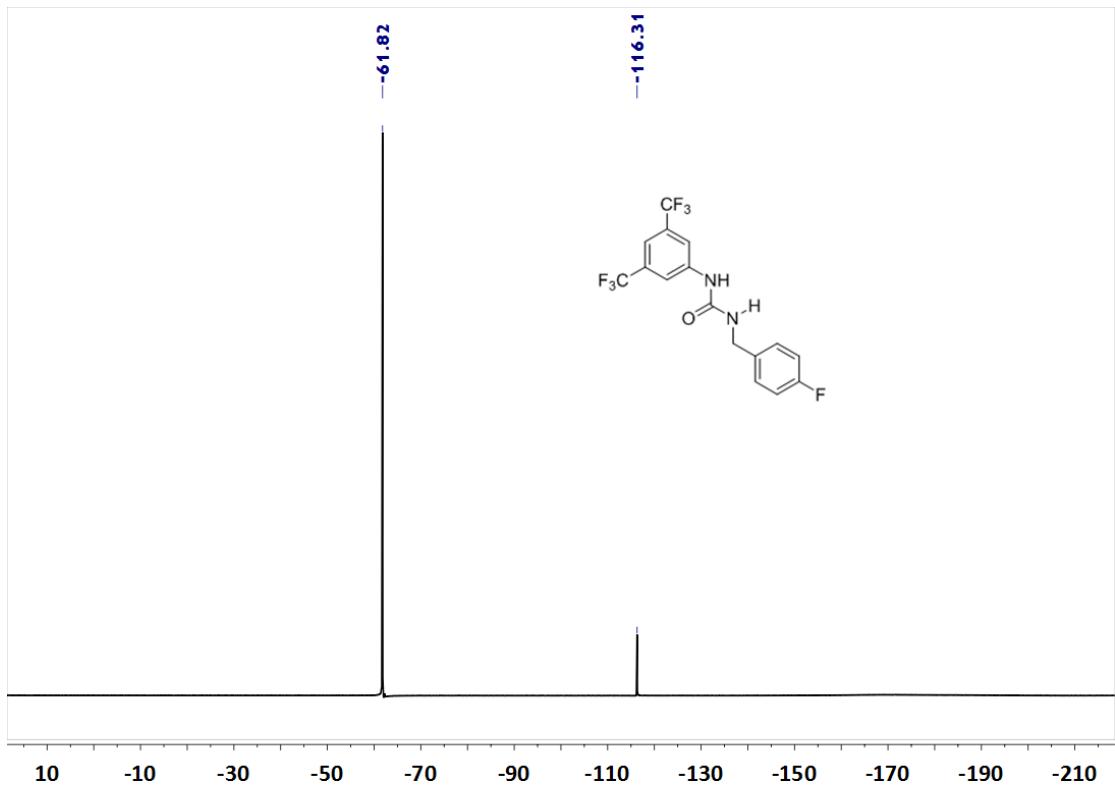
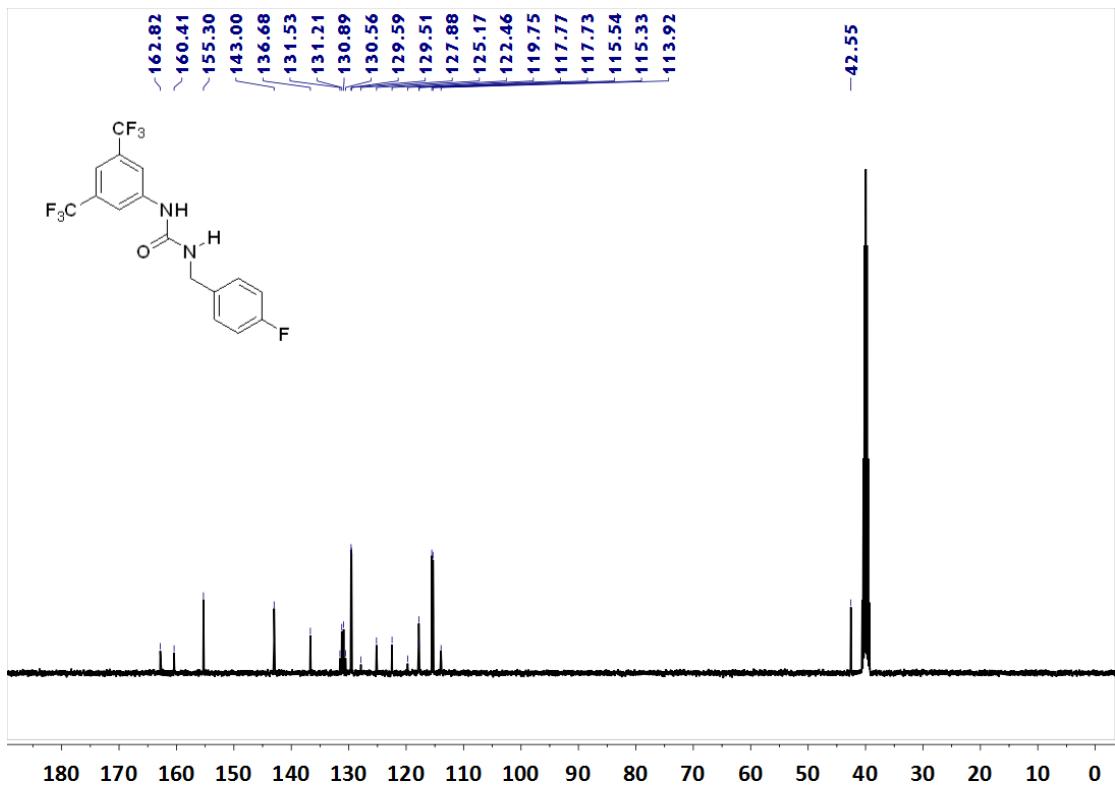
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5a**.



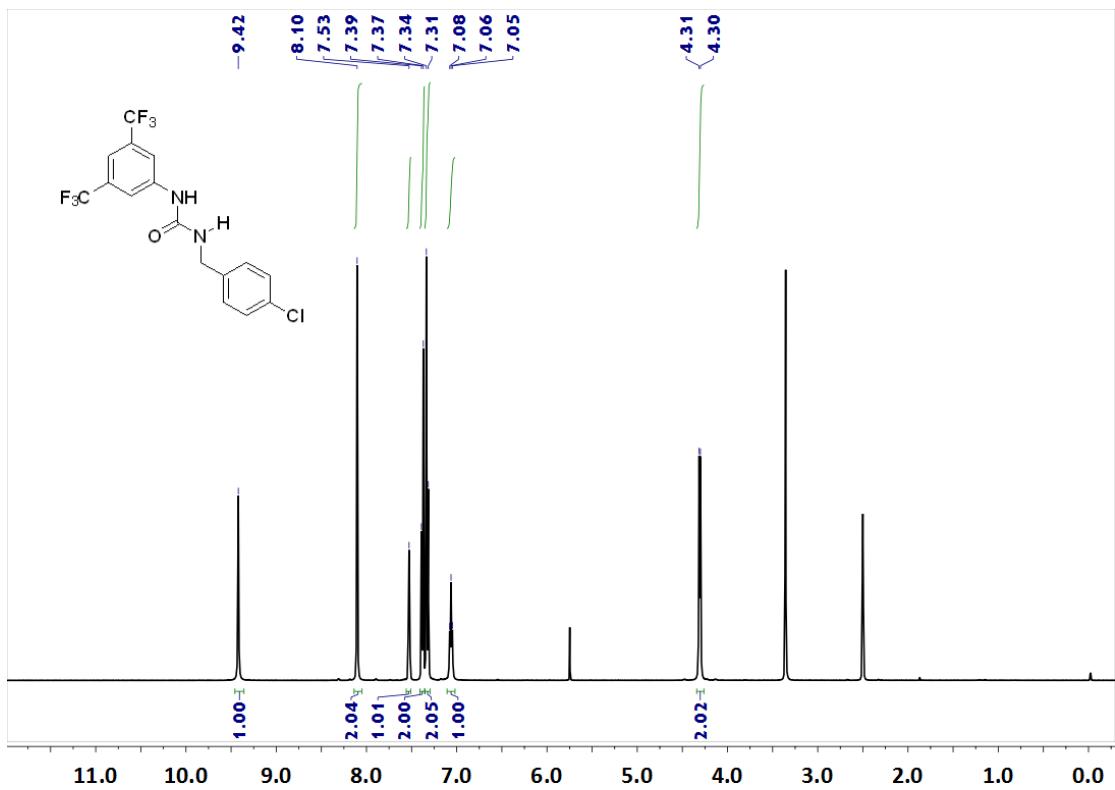
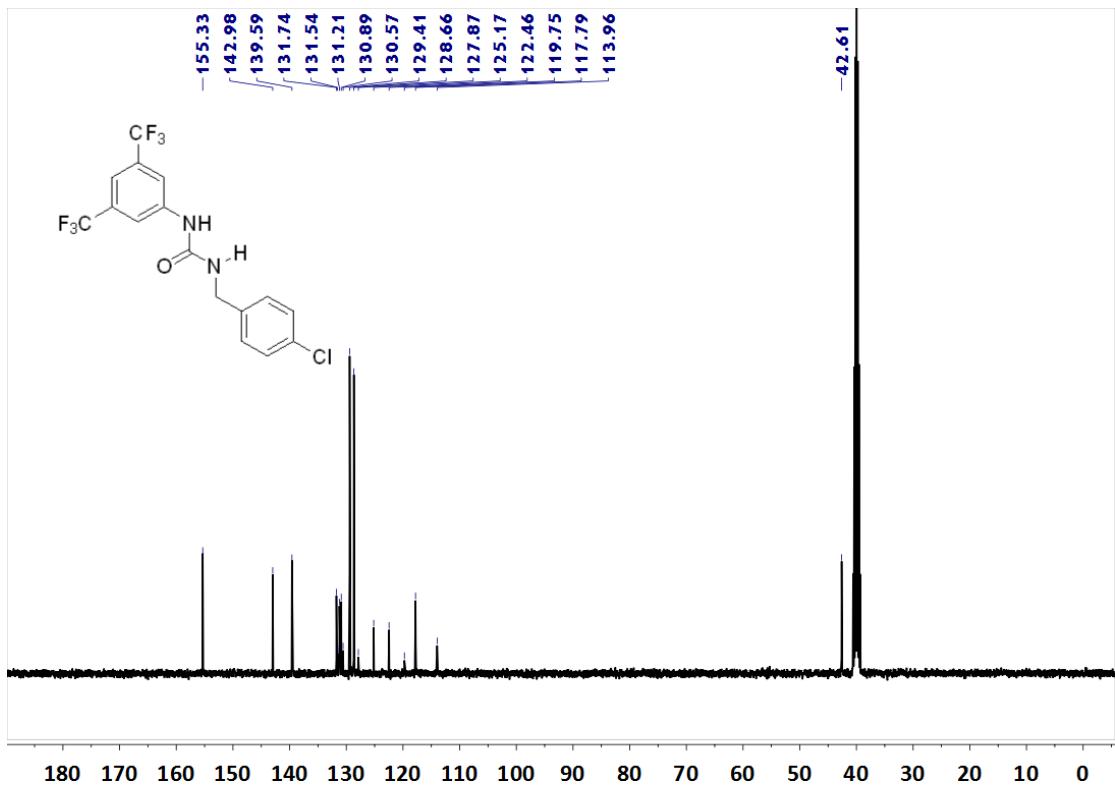


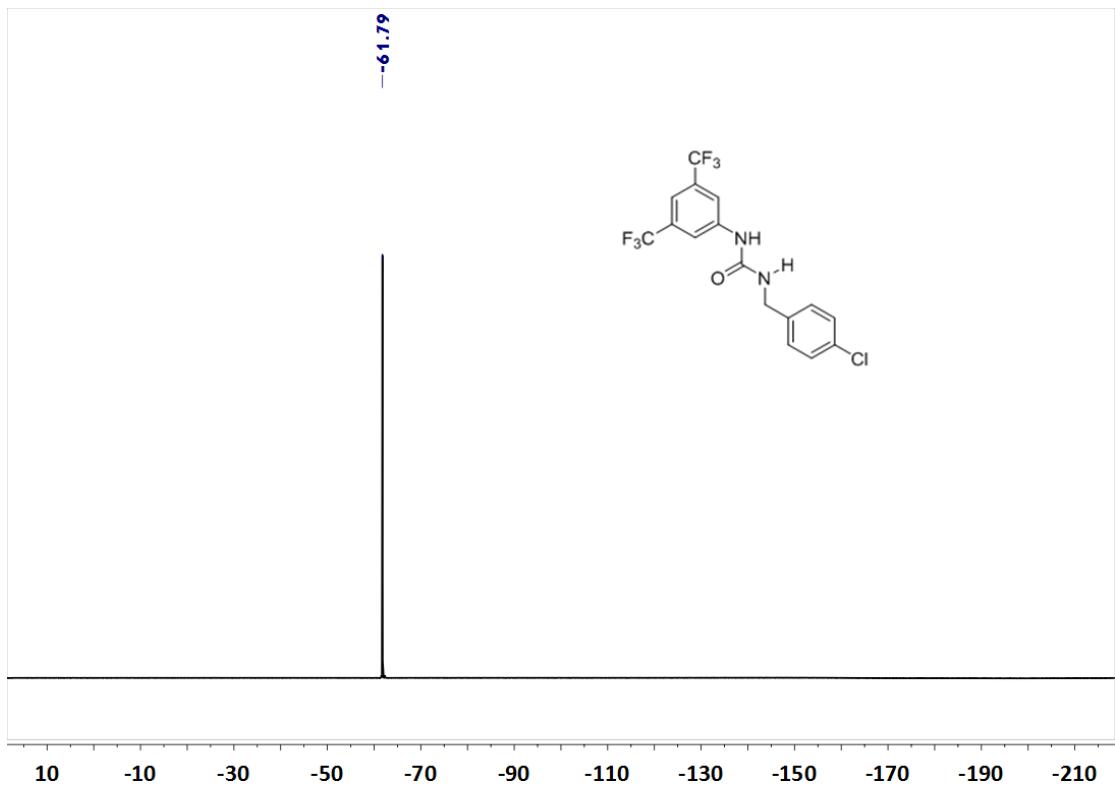
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **5b**.



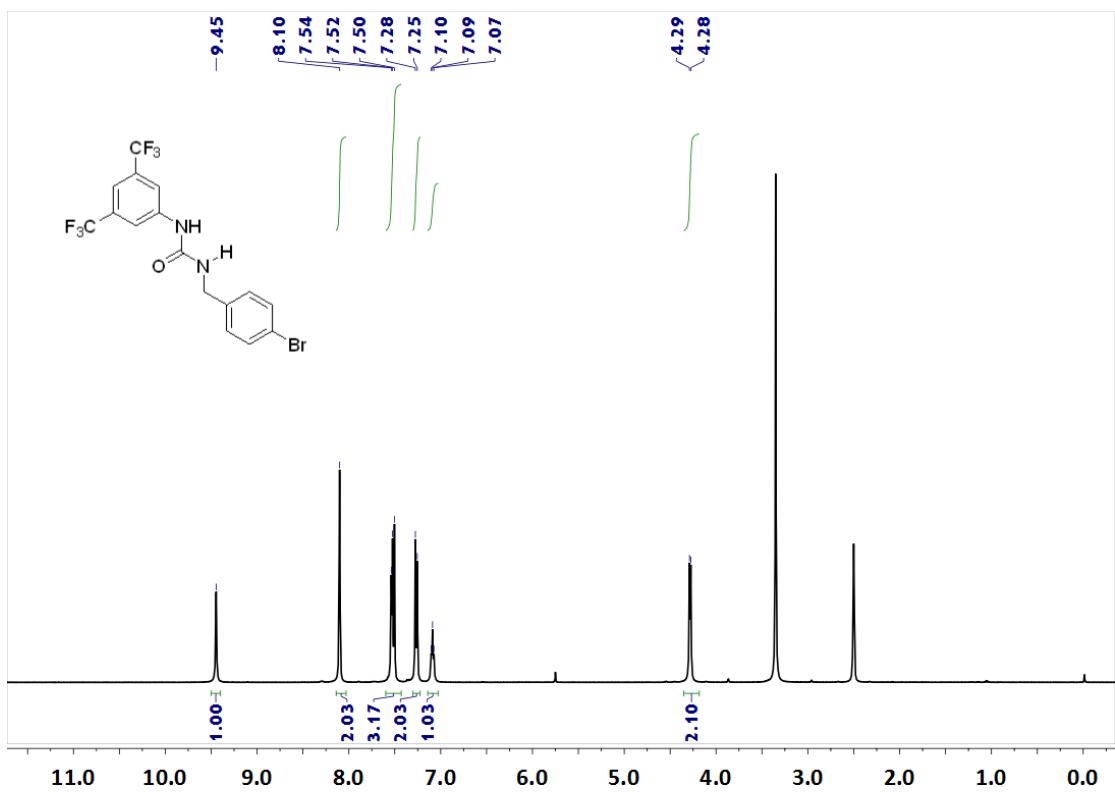


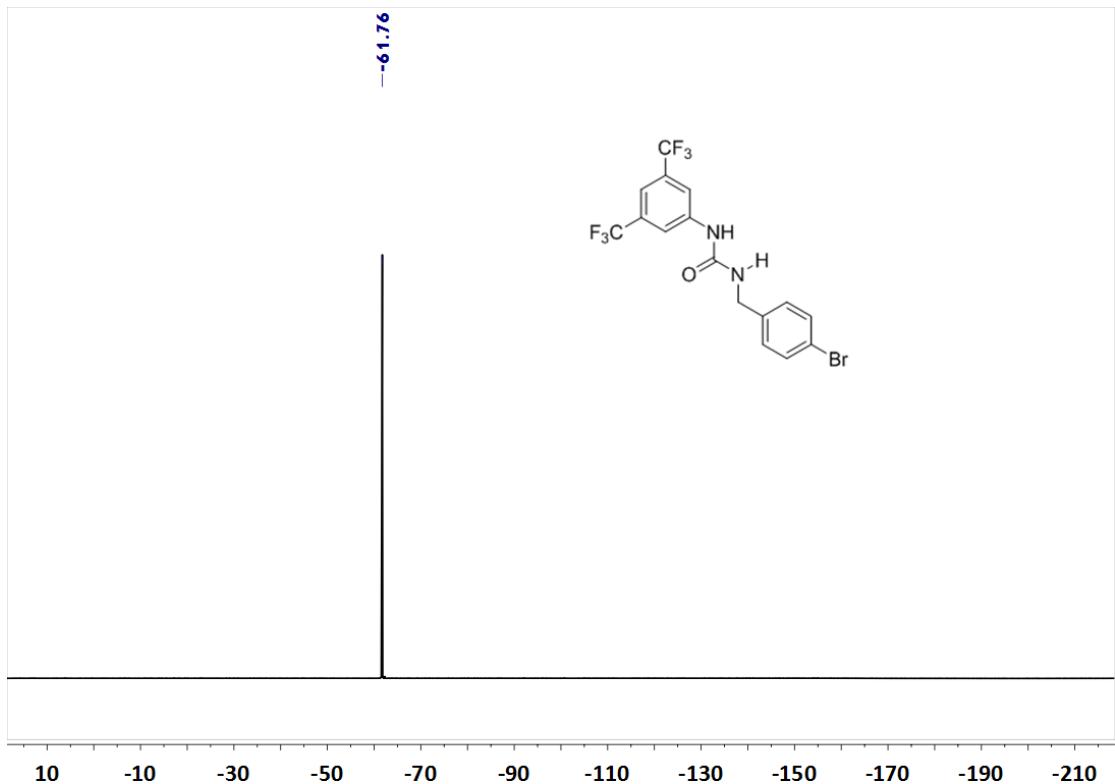
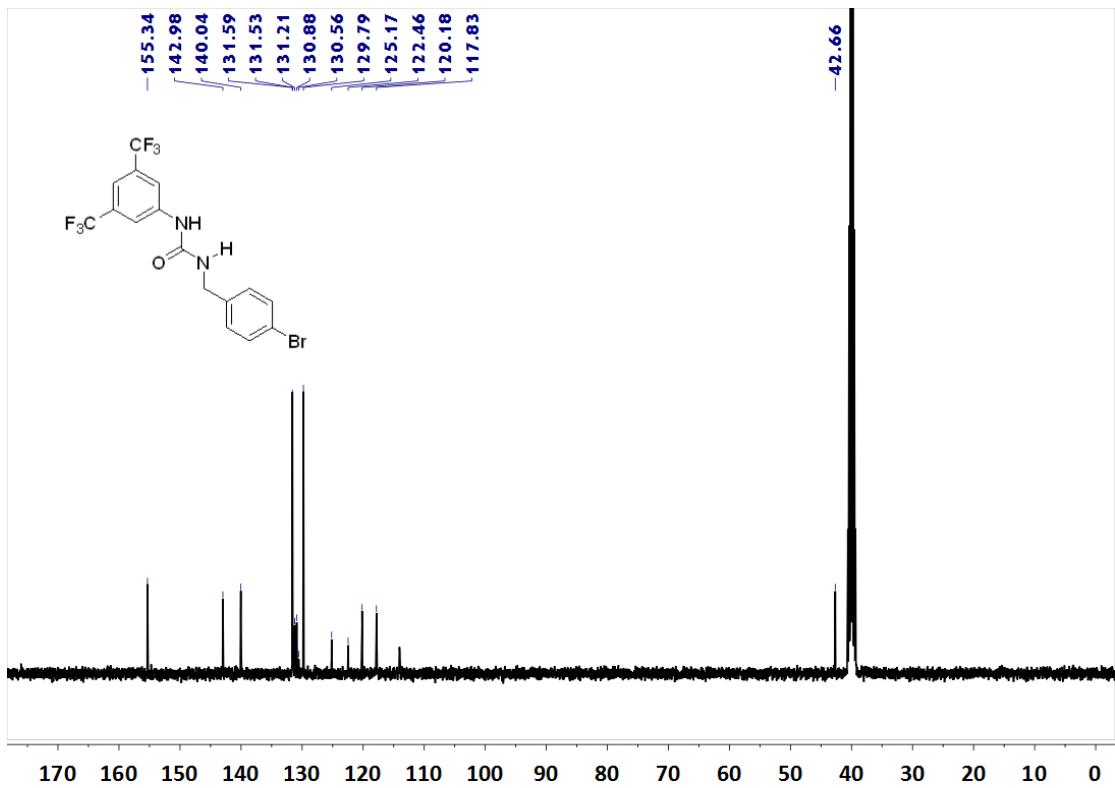
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5c**.



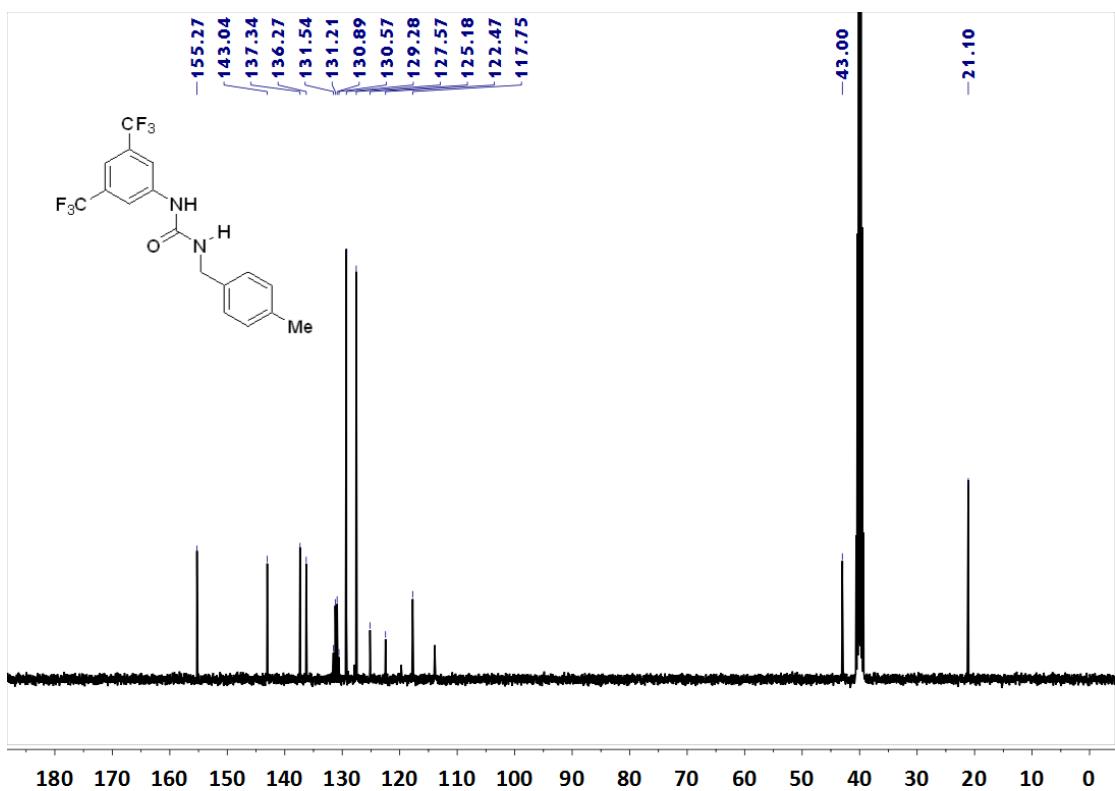
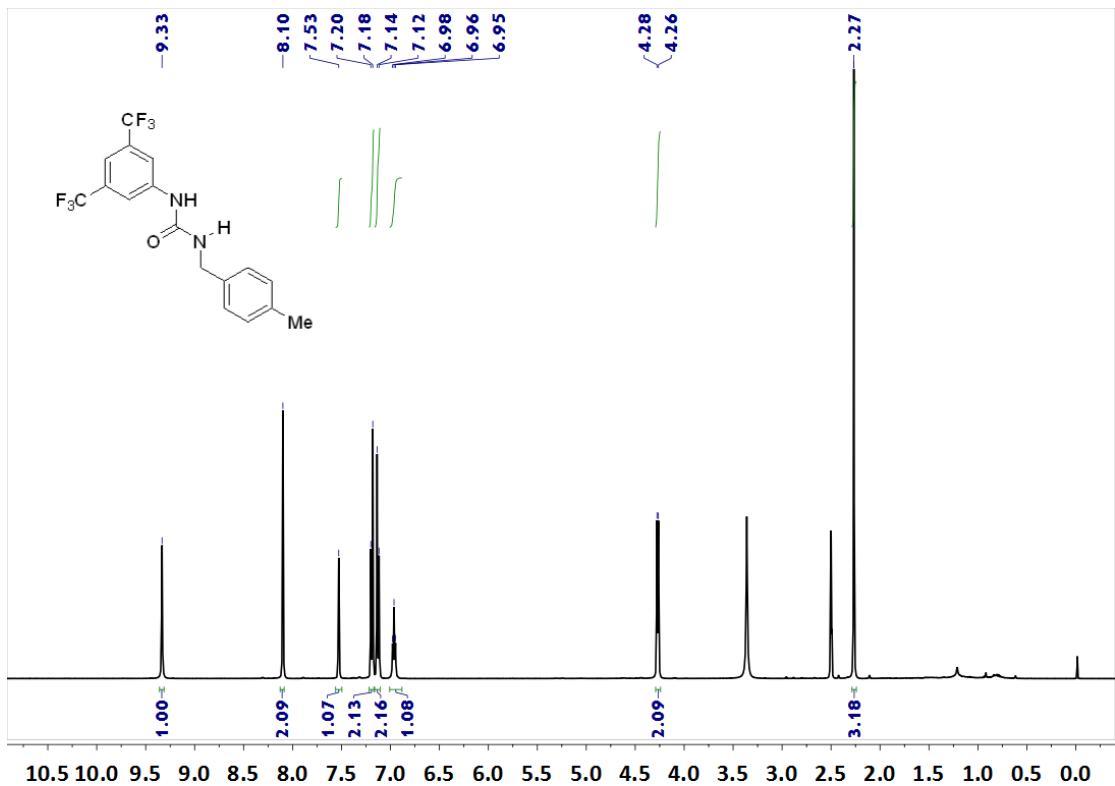


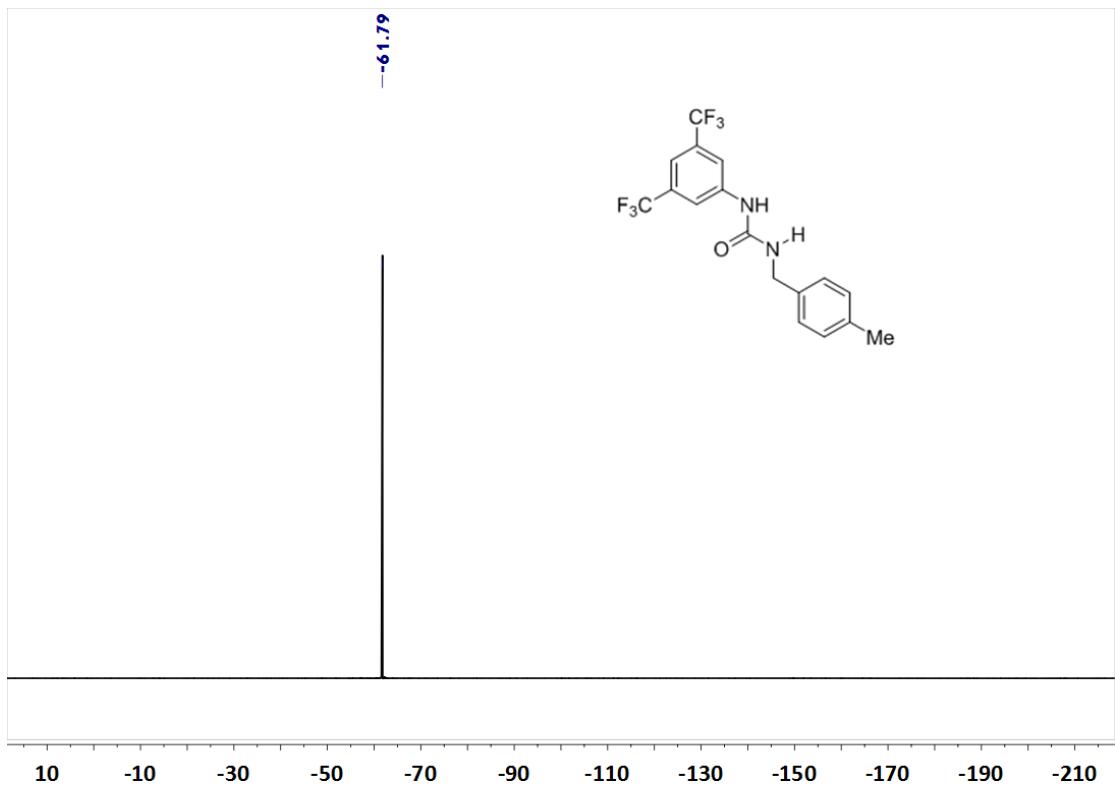
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **5d**.



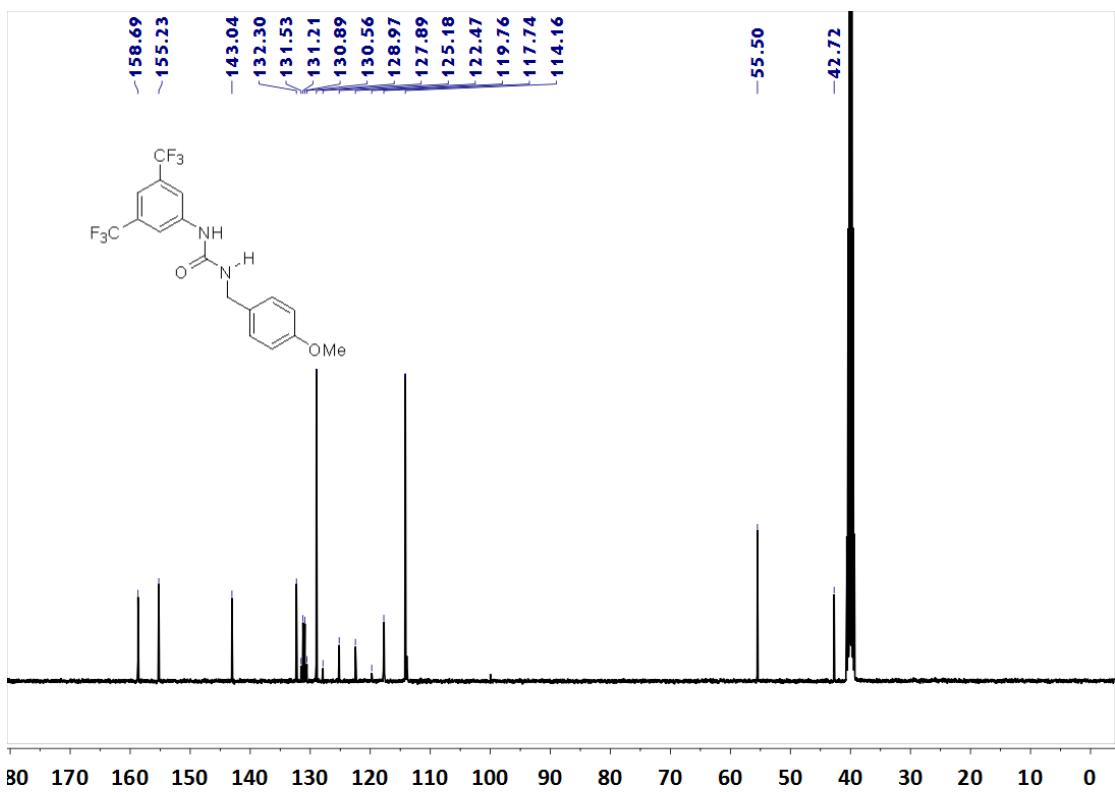


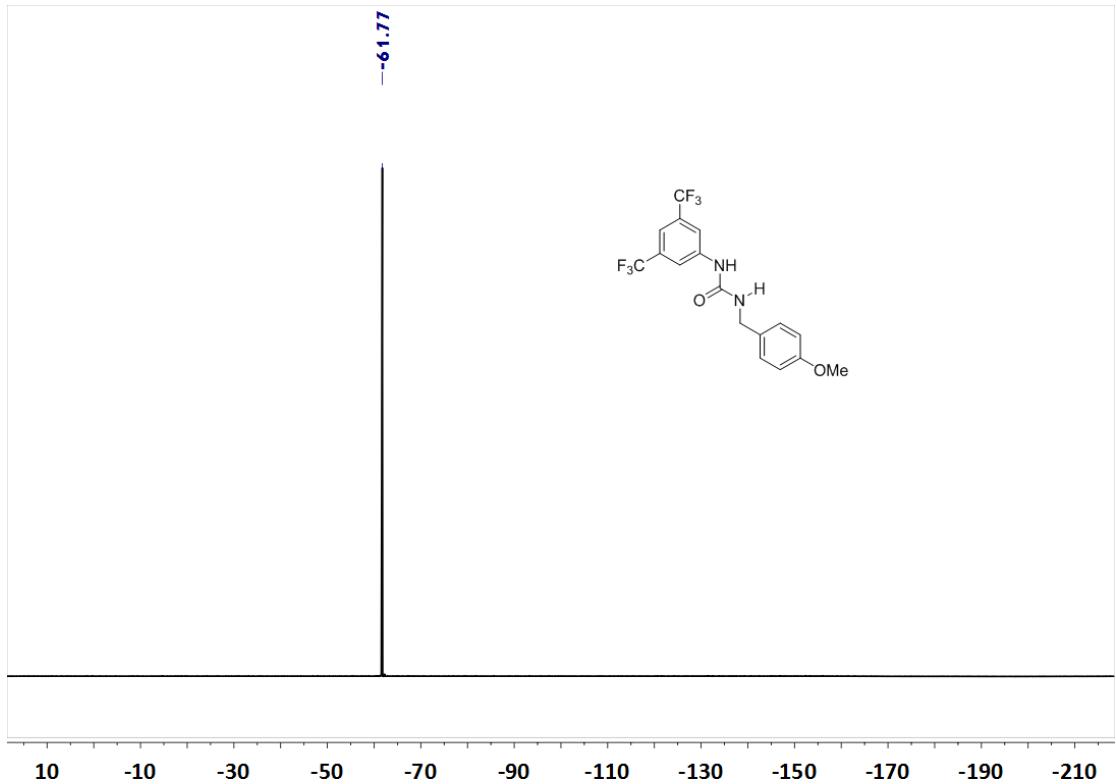
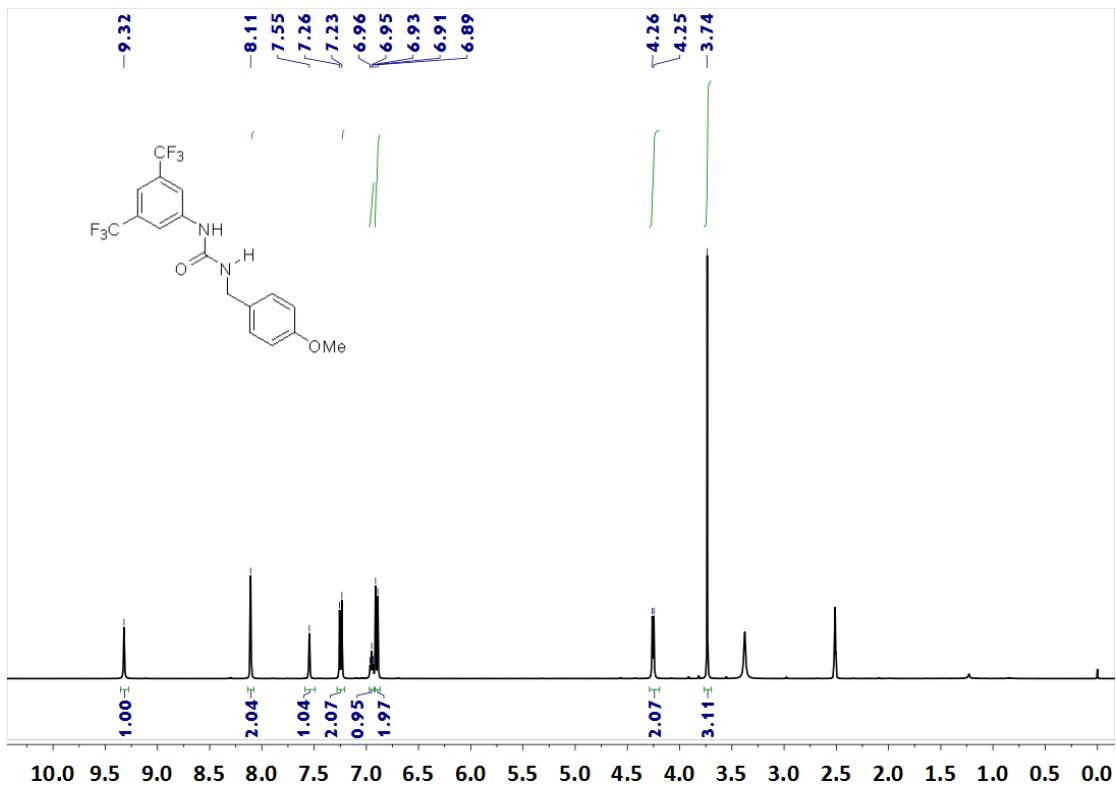
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5e**.



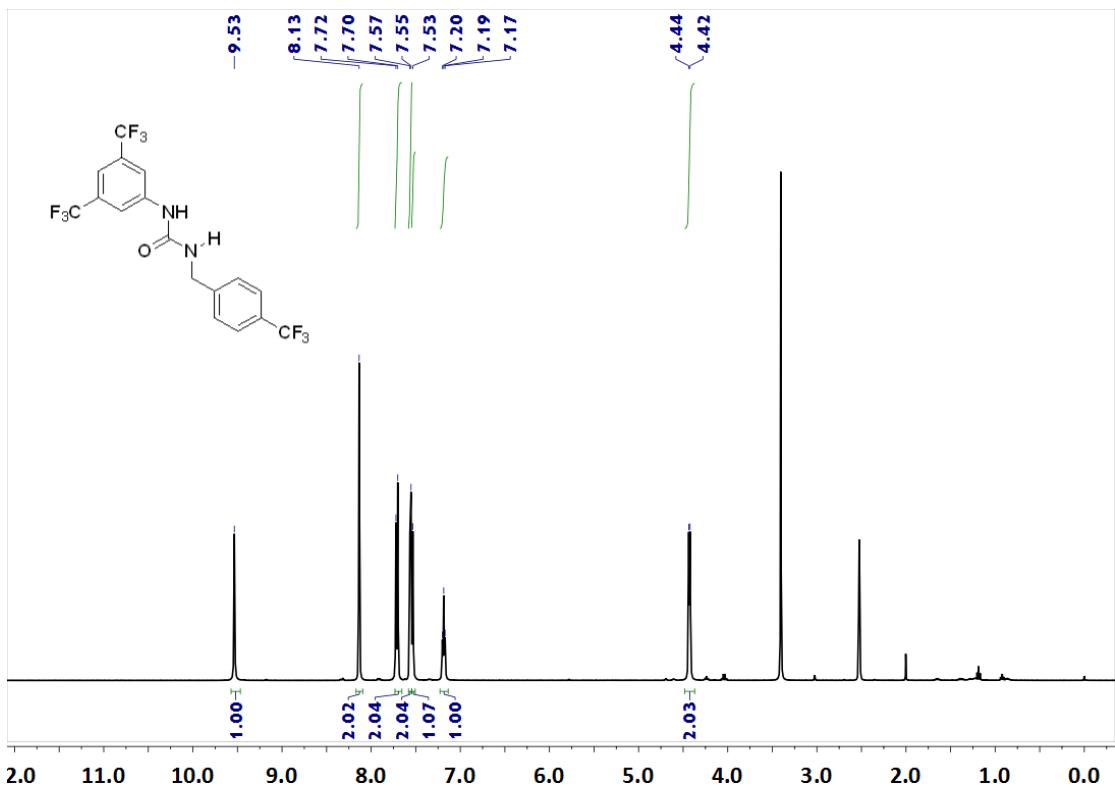
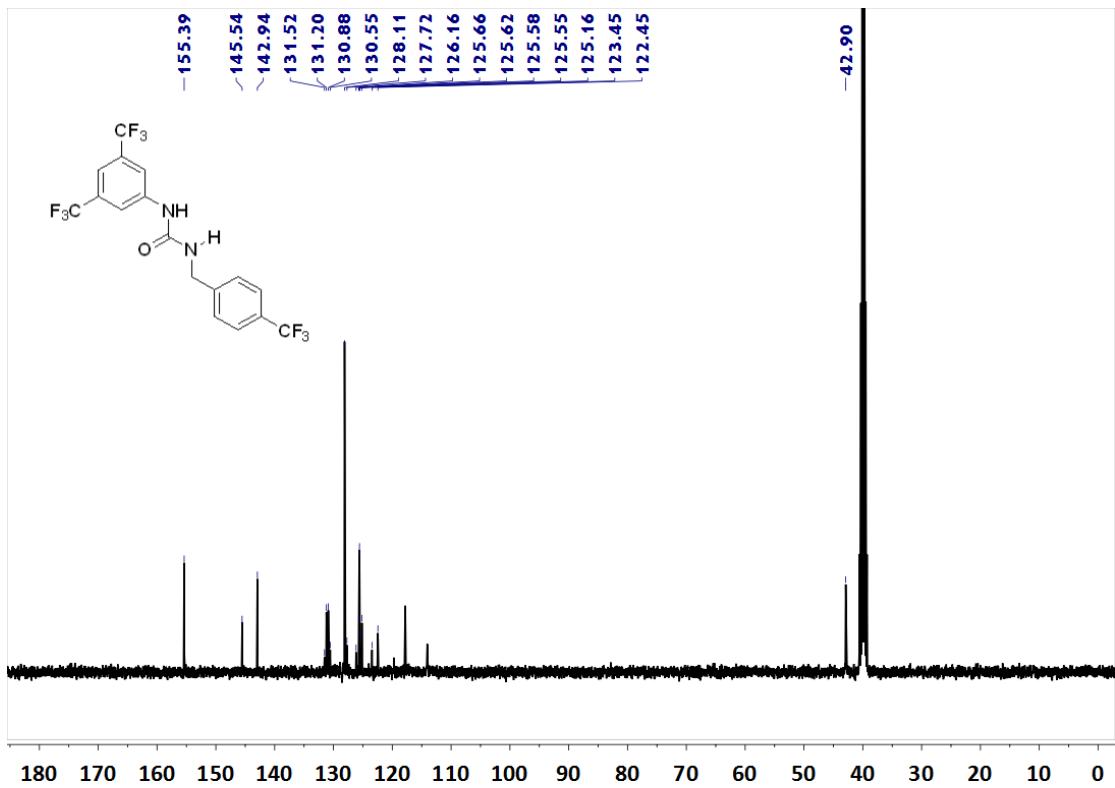


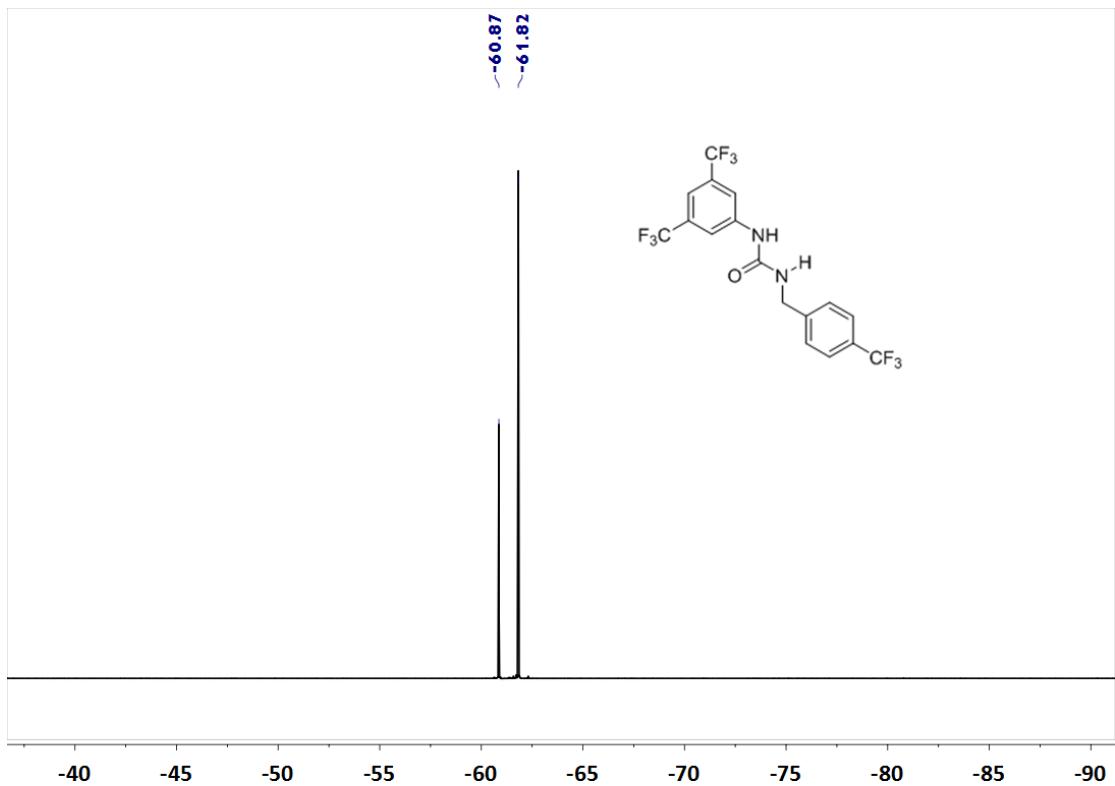
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **5f**.



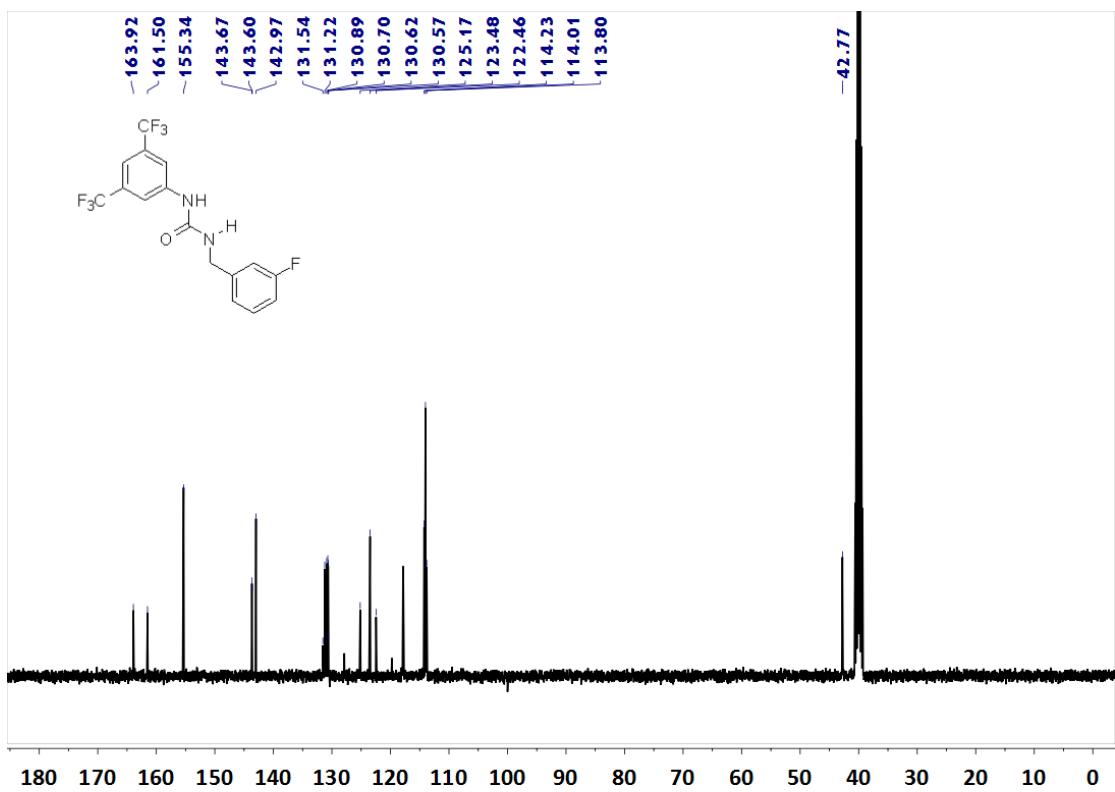


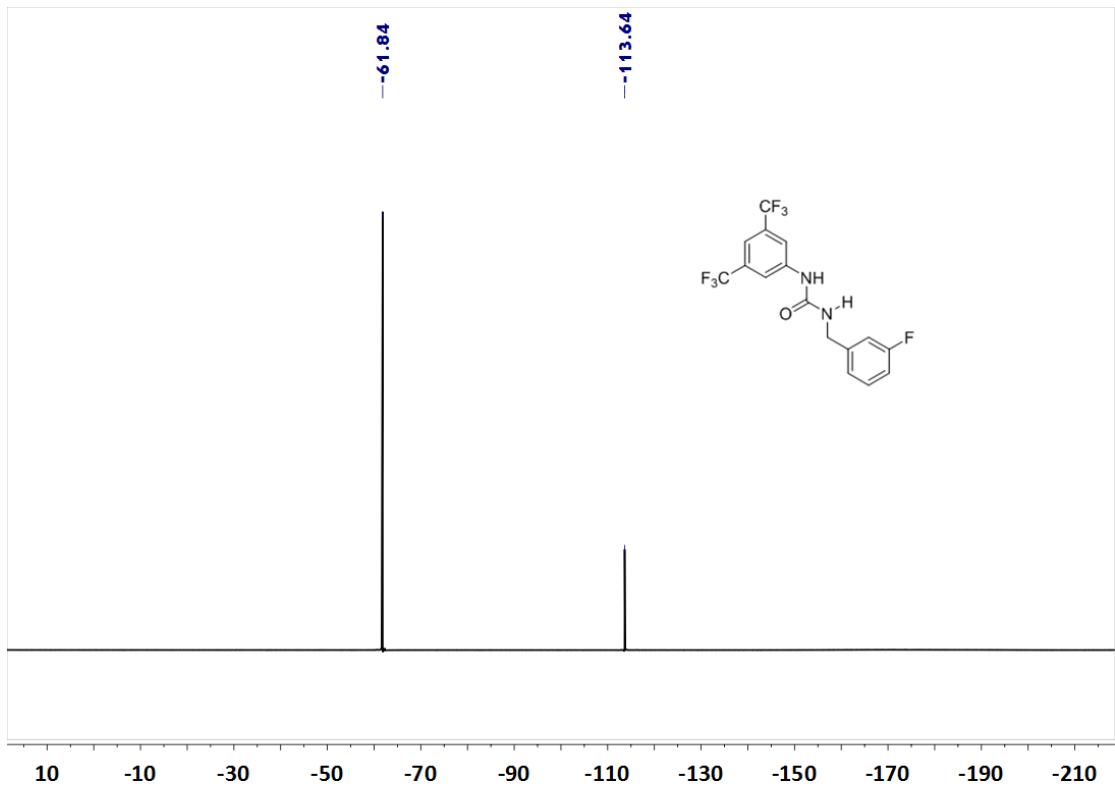
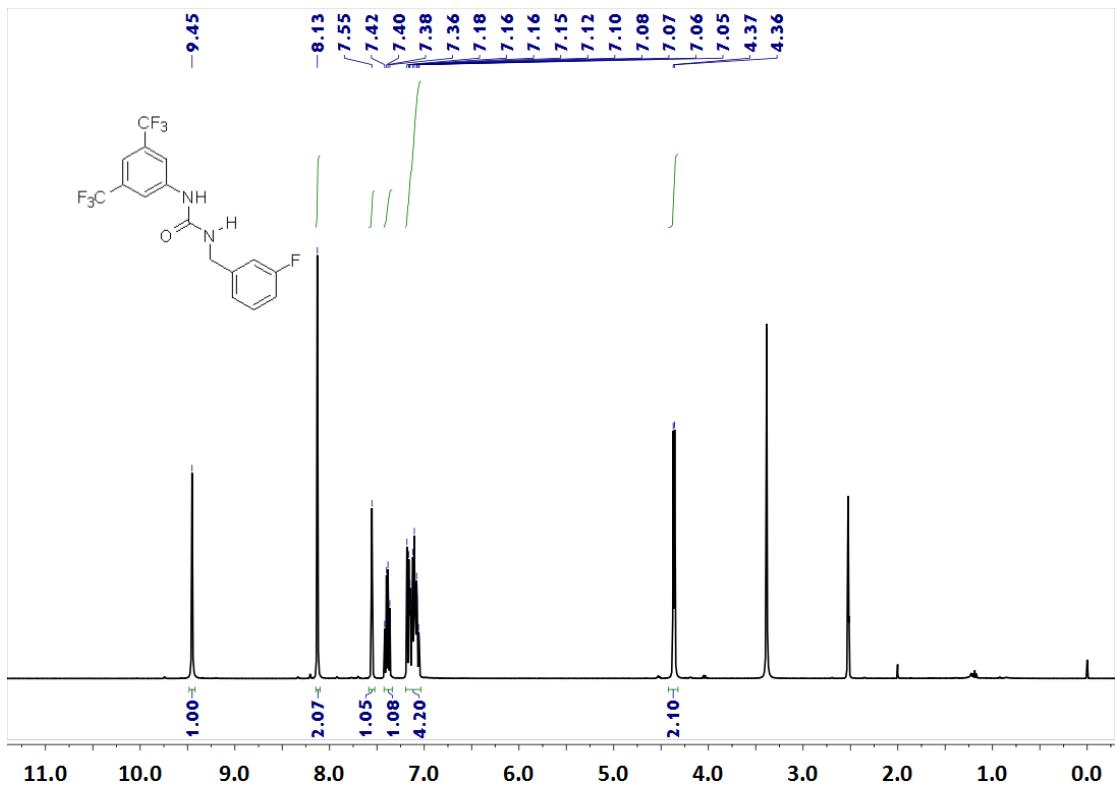
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5g**.



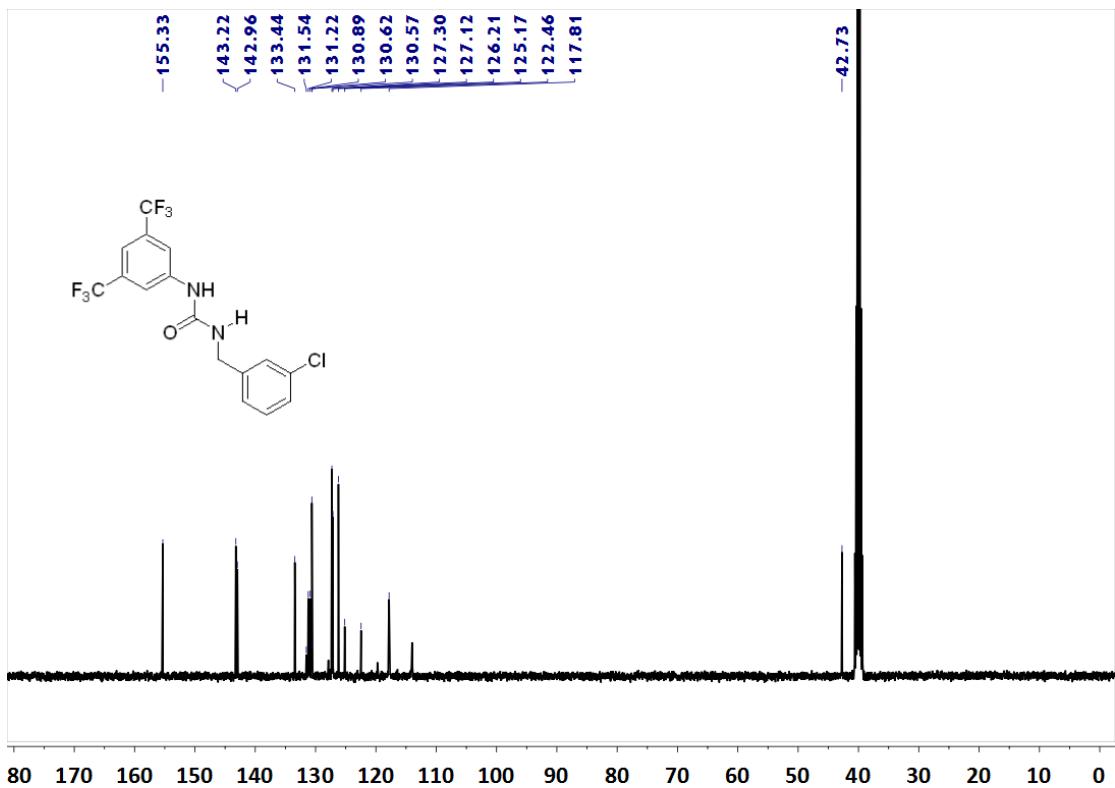
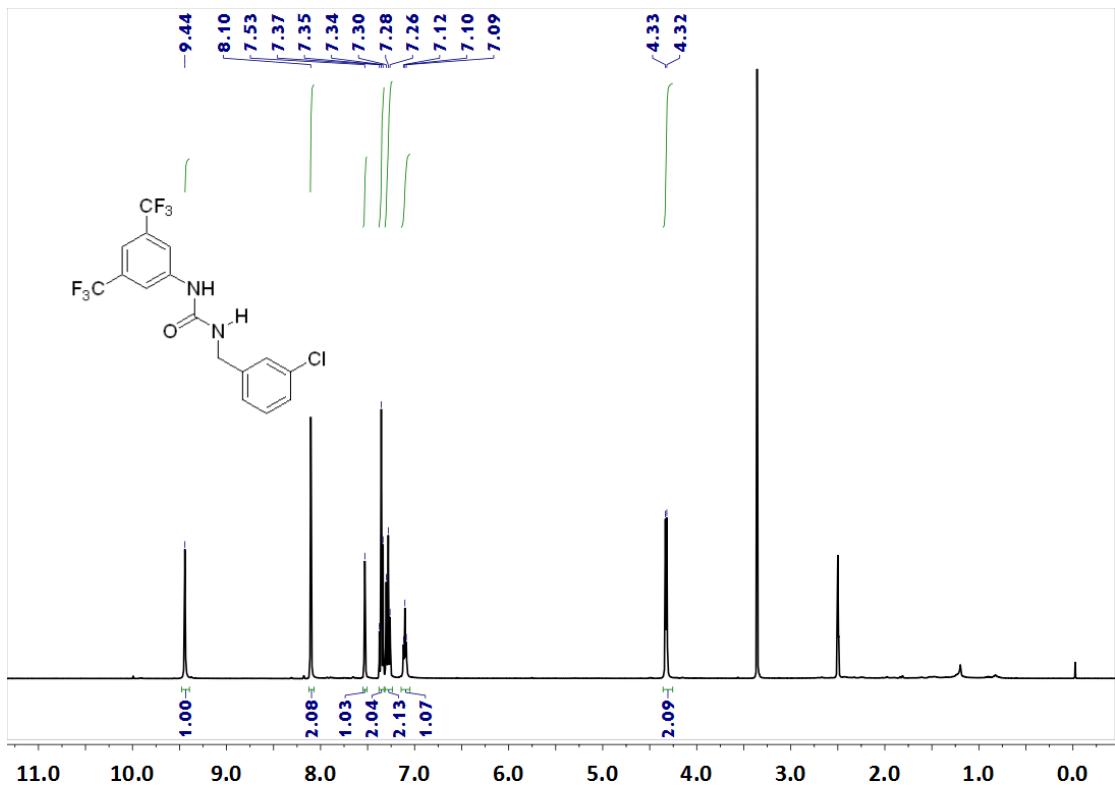


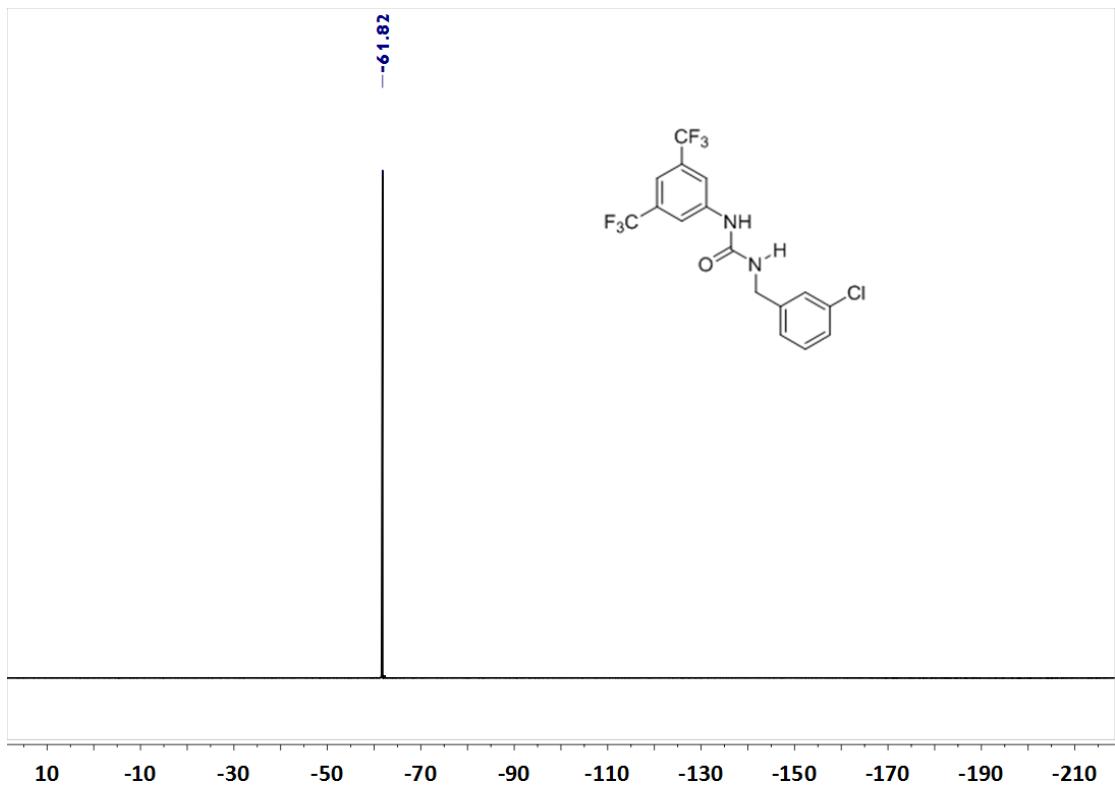
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5h**.



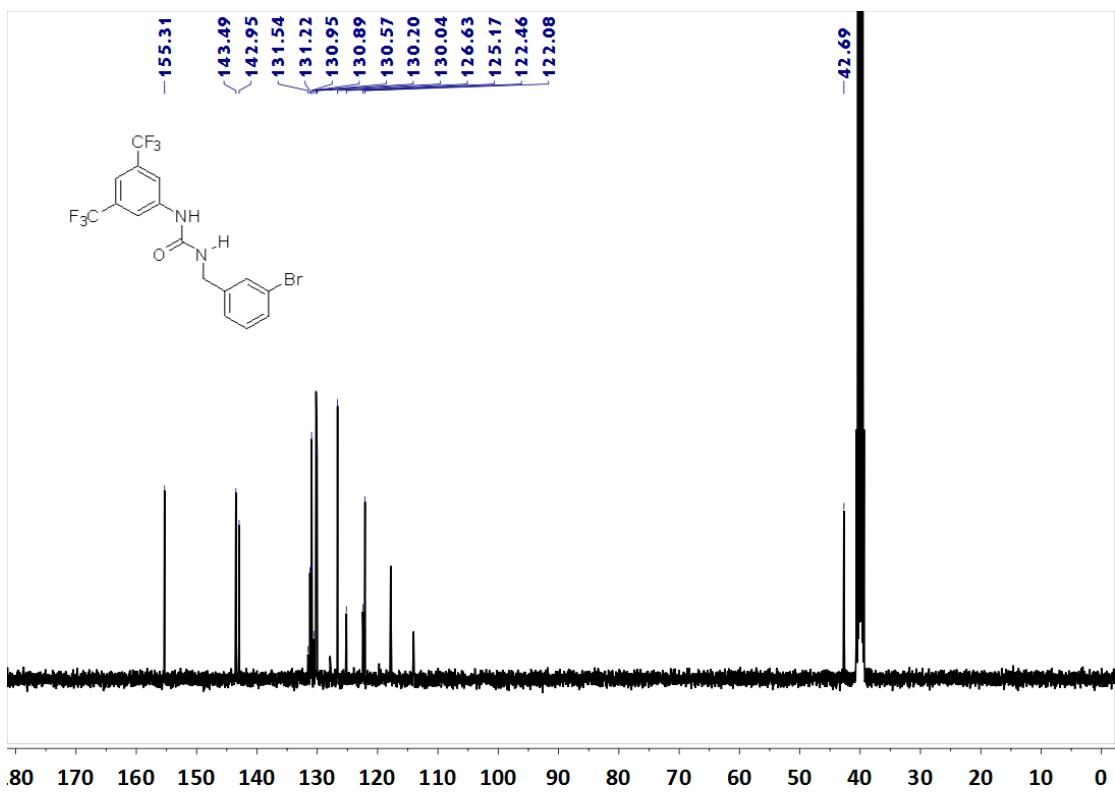


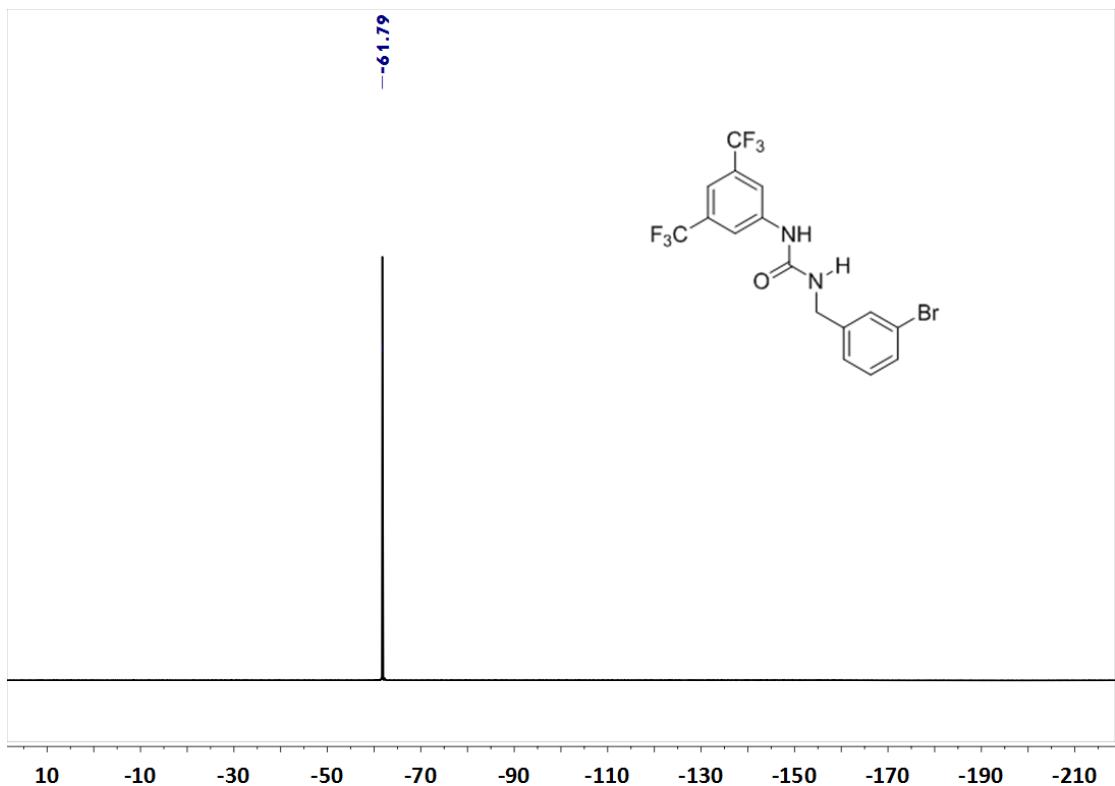
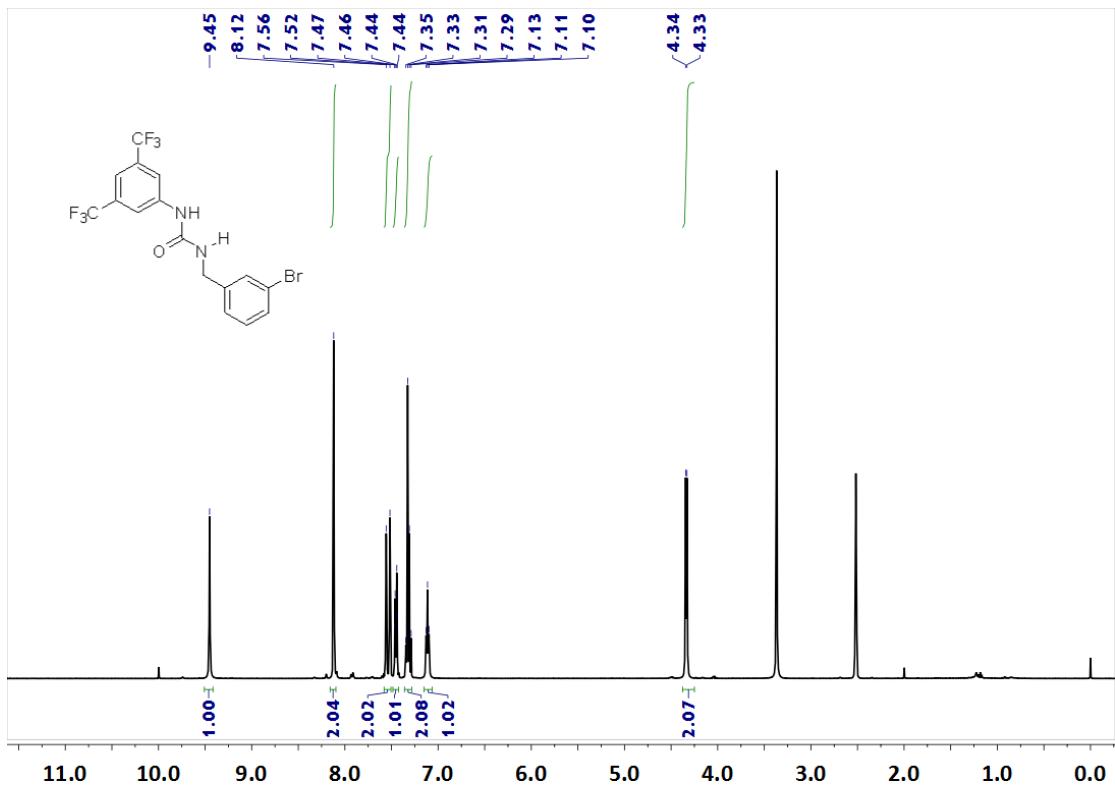
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5i**.



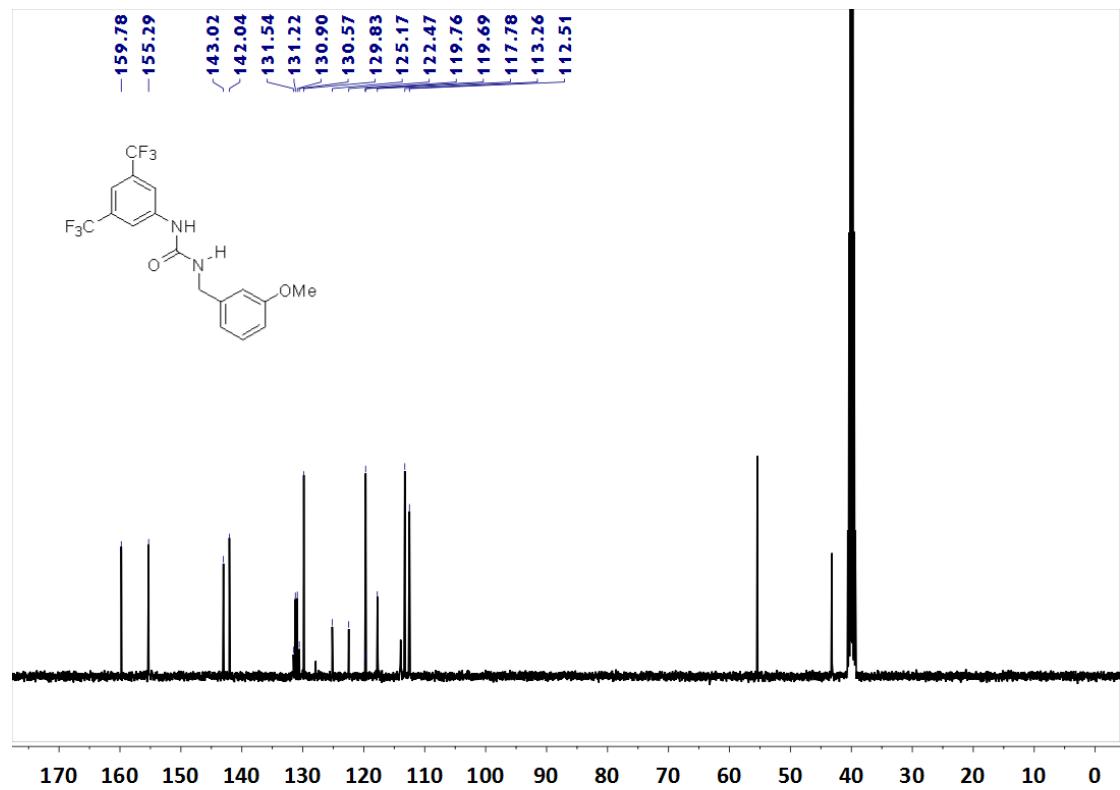
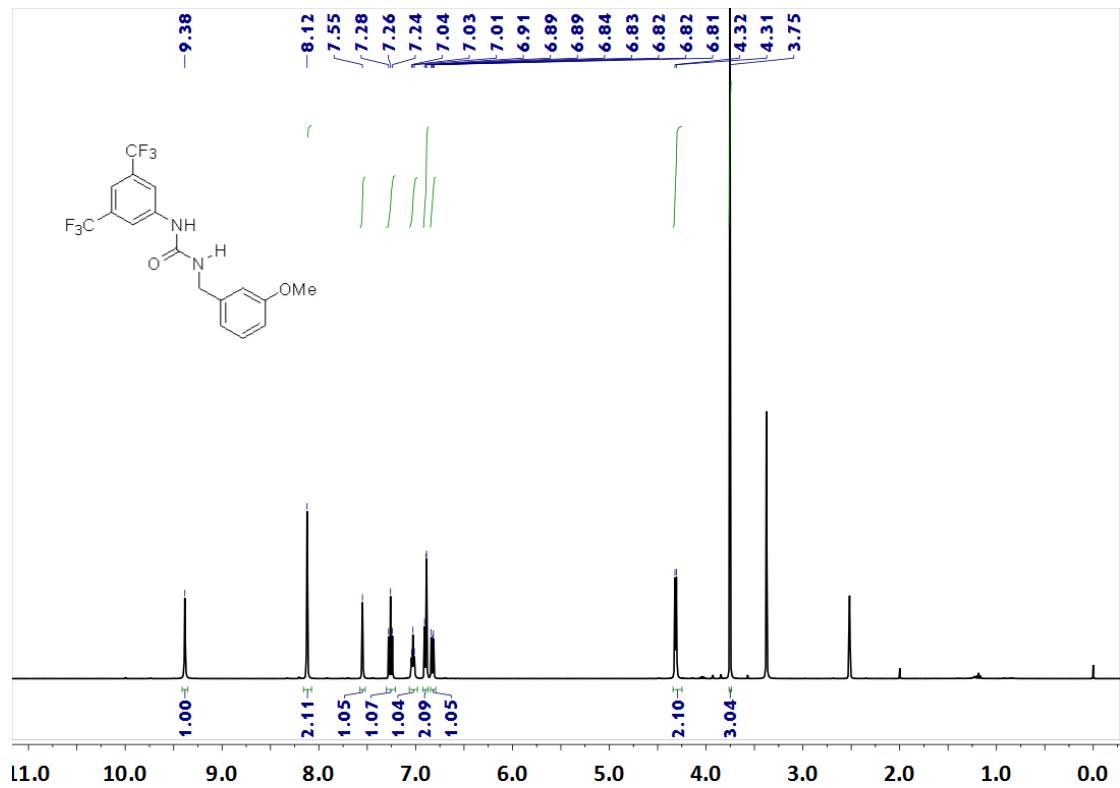


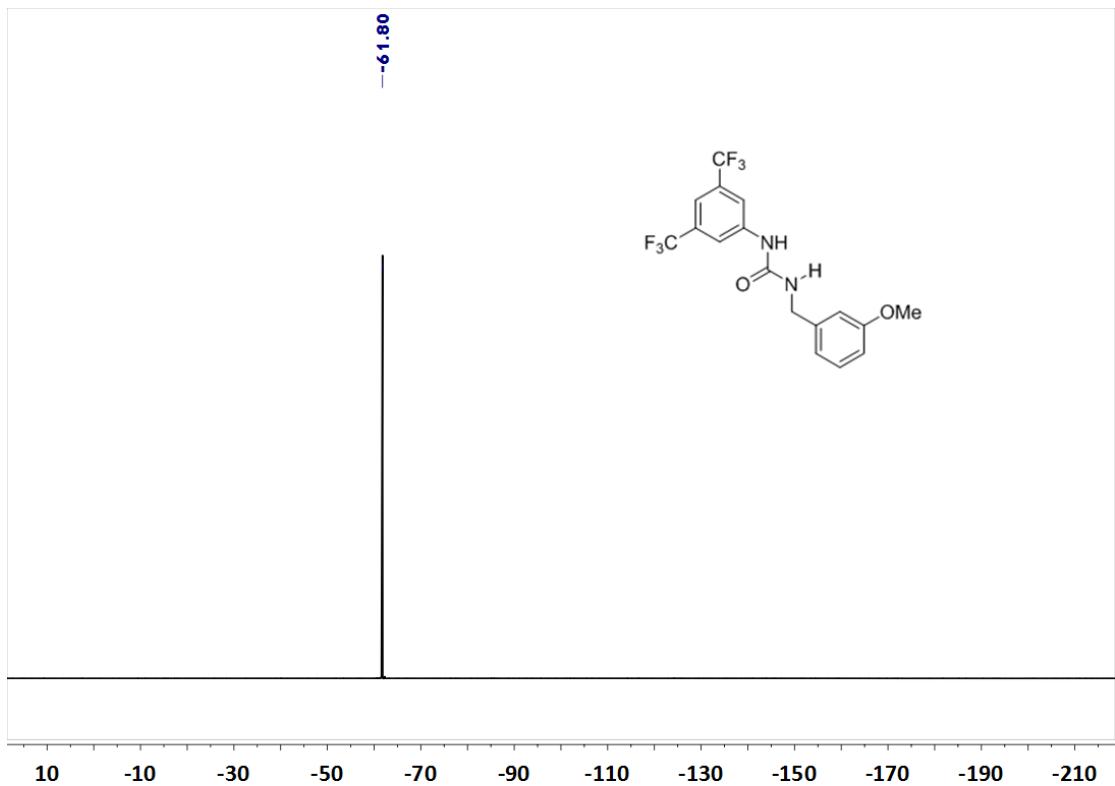
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **5j**.



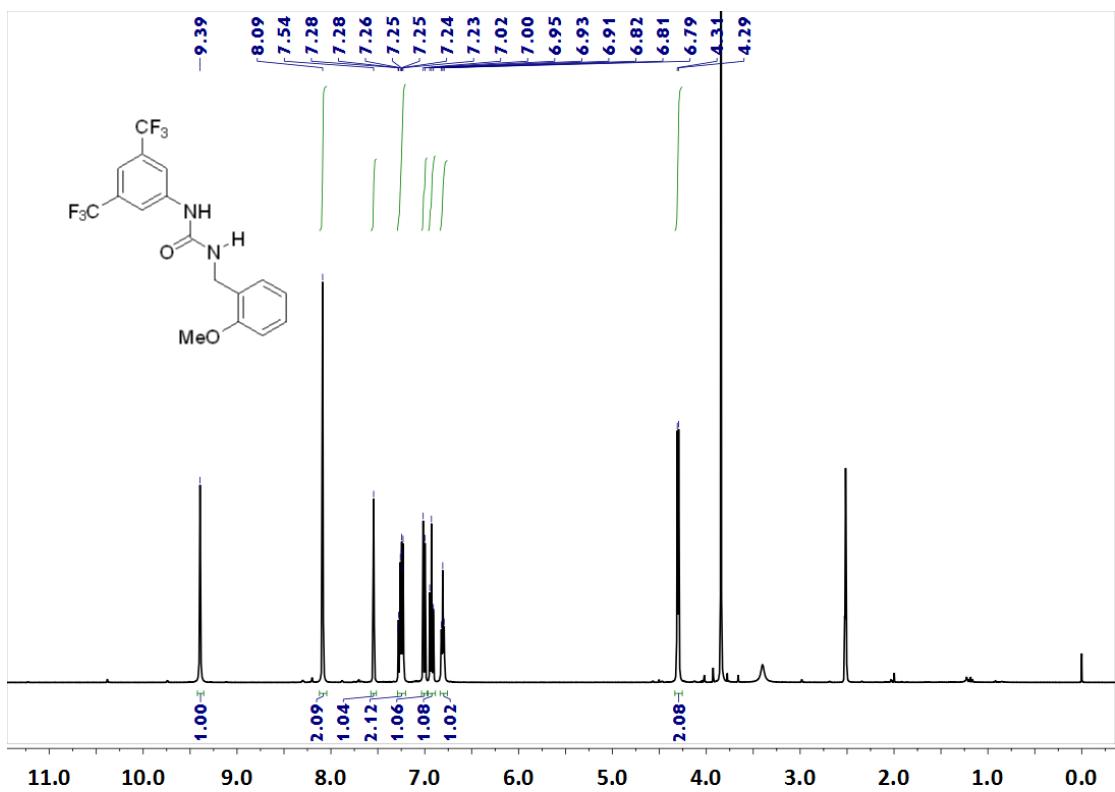


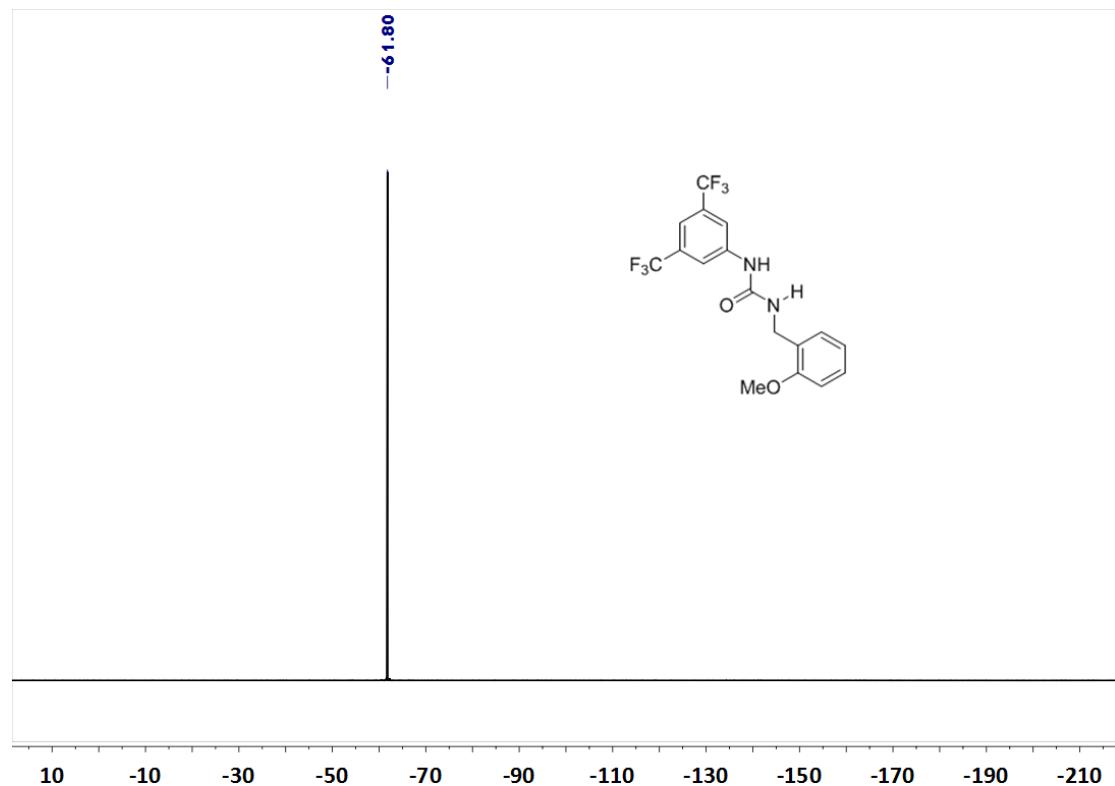
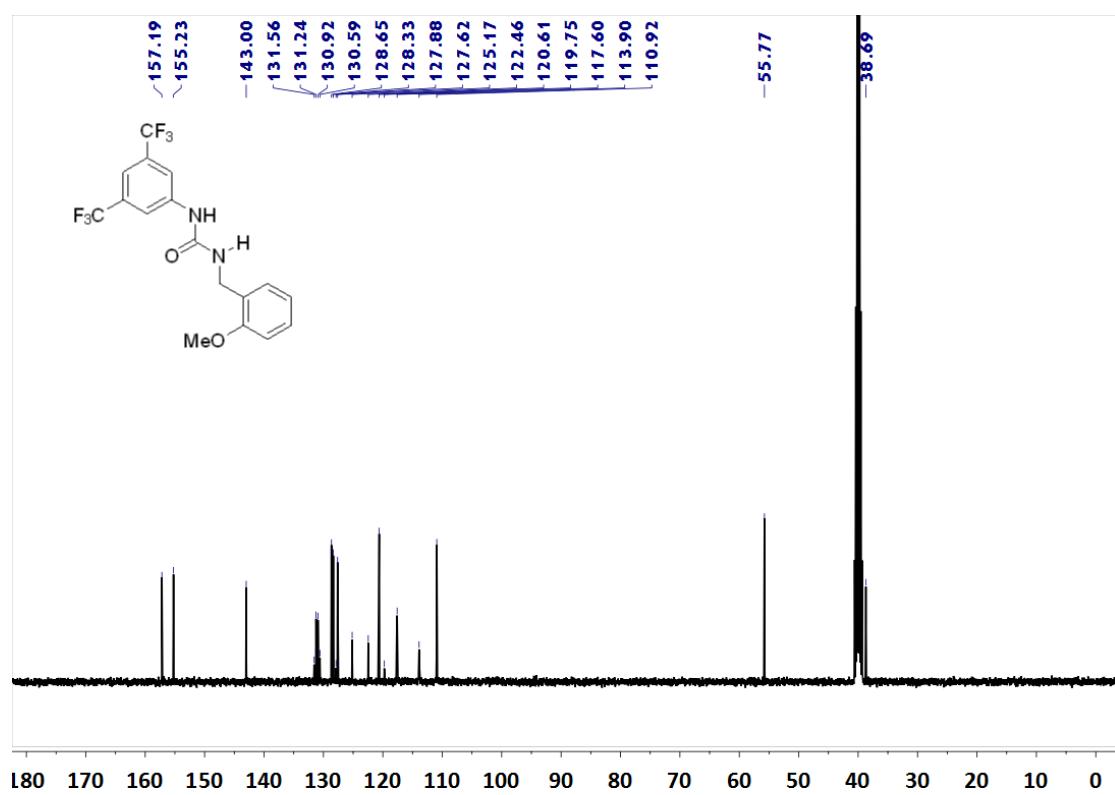
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5k**.



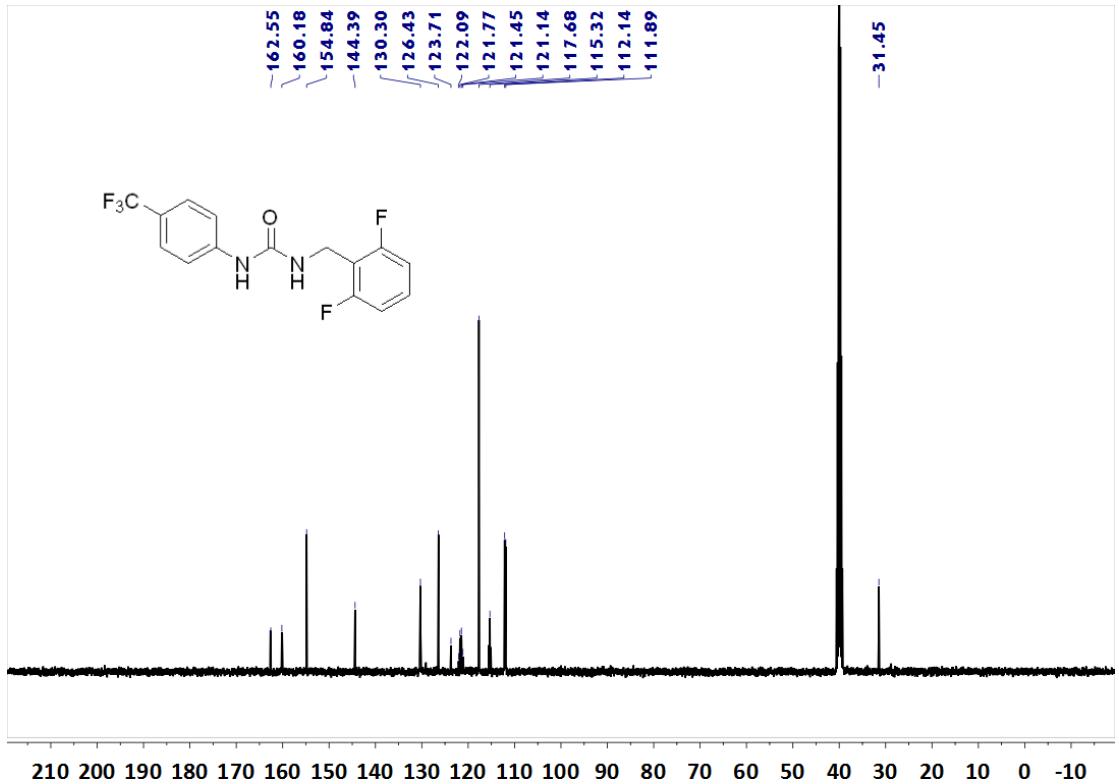
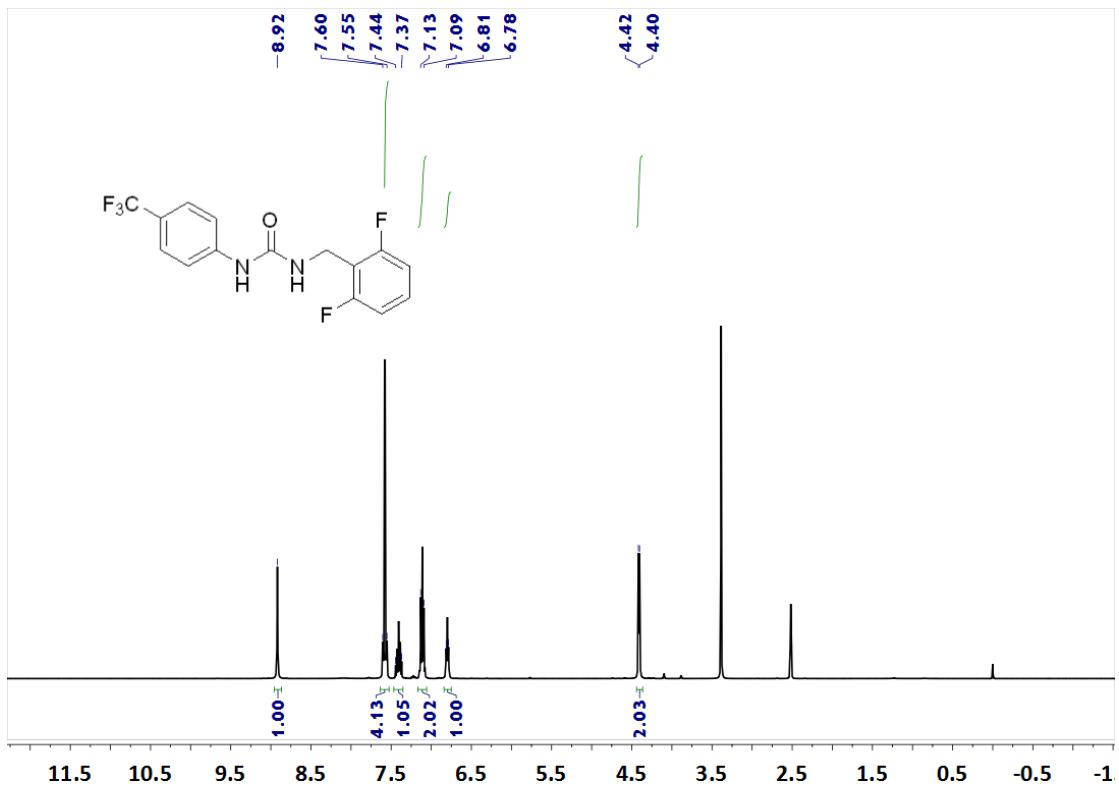


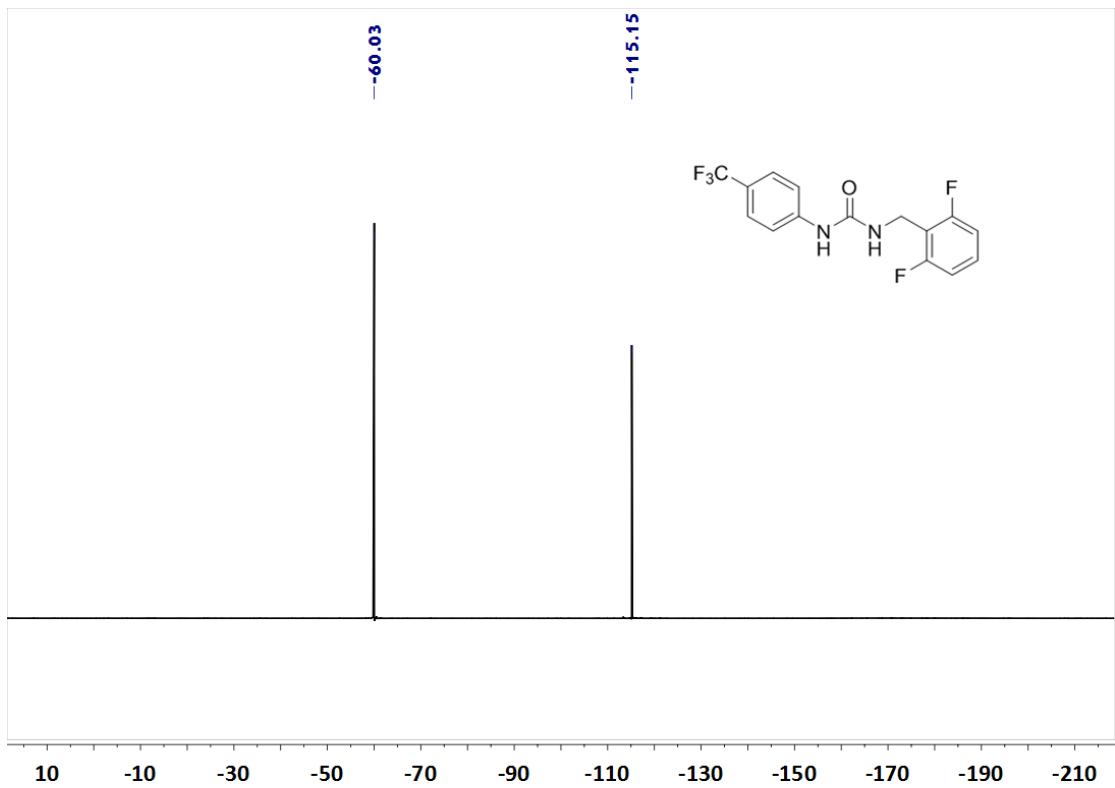
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **5l**.



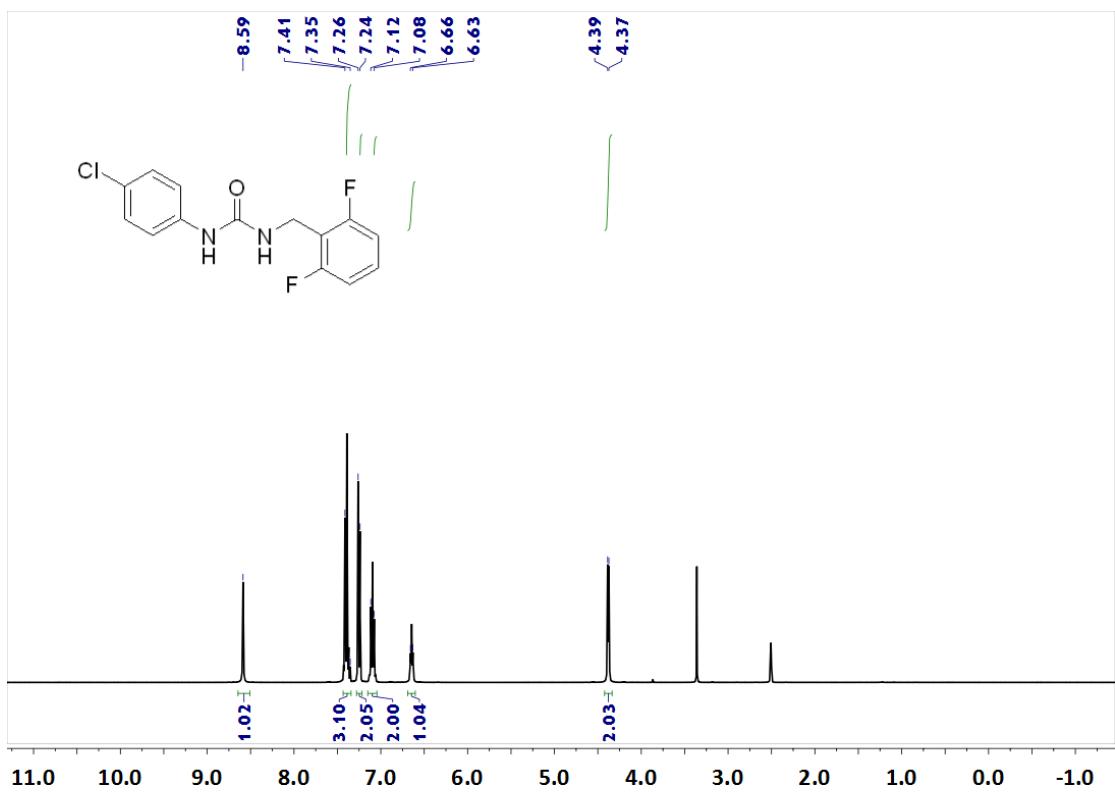


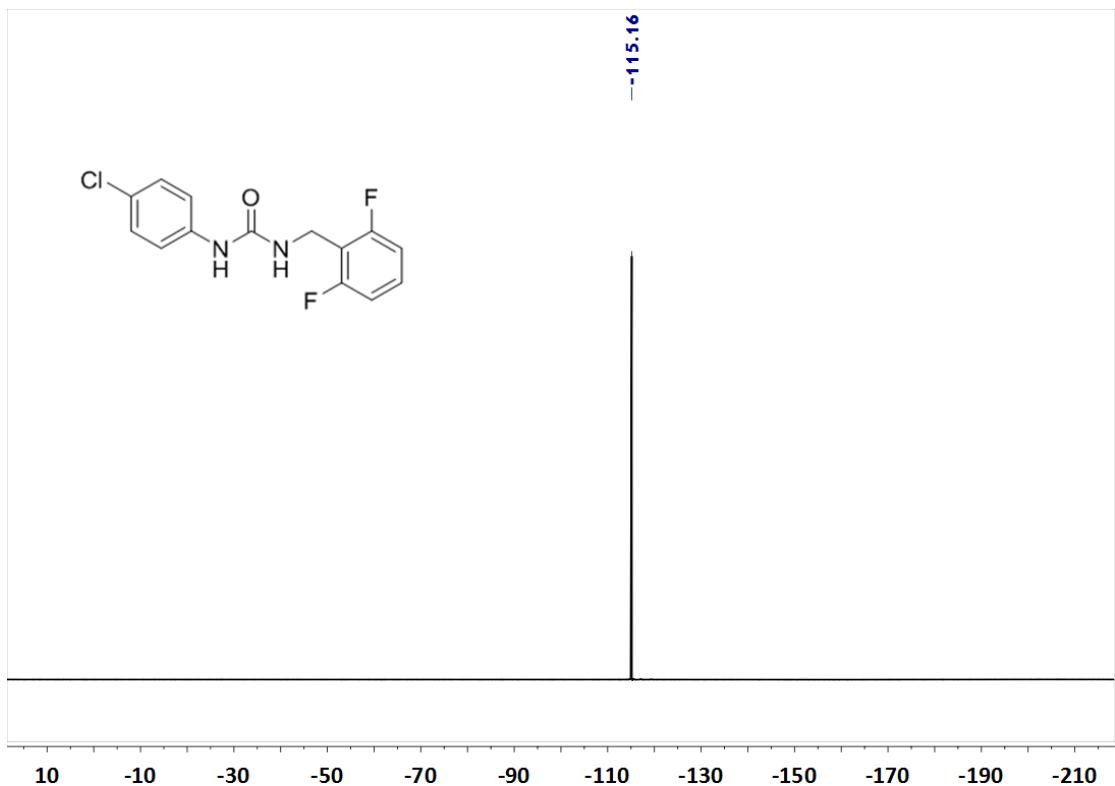
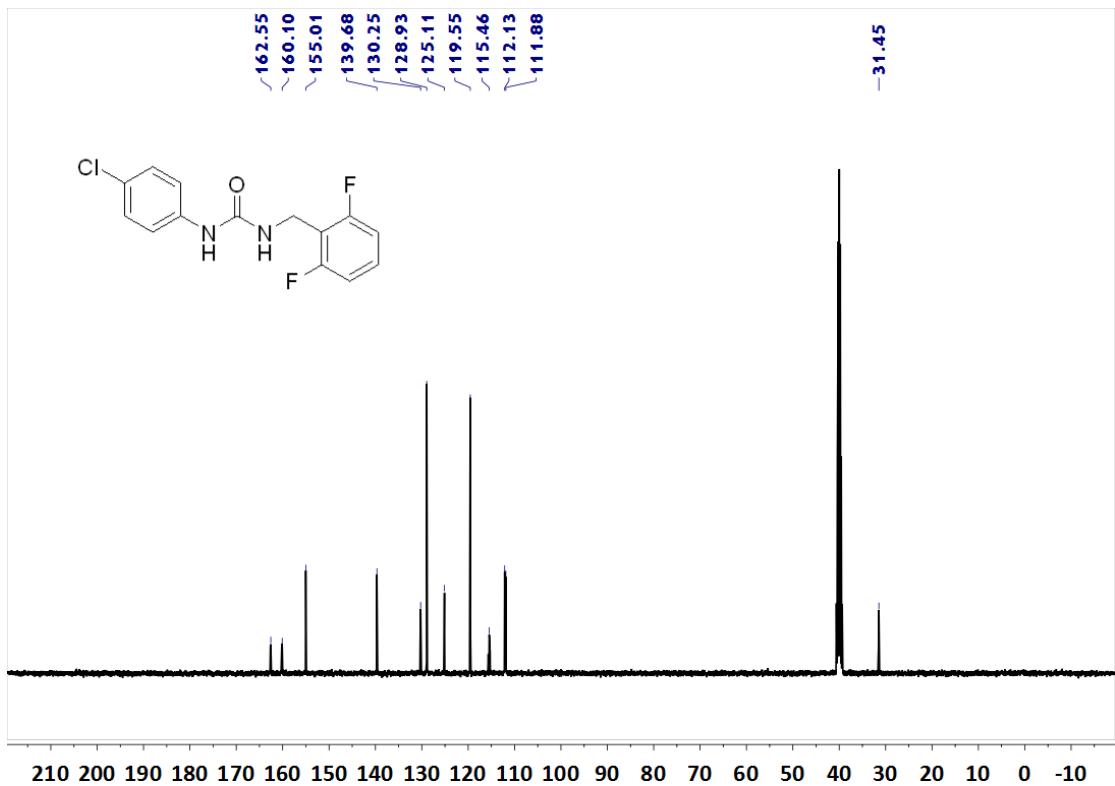
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5m**.



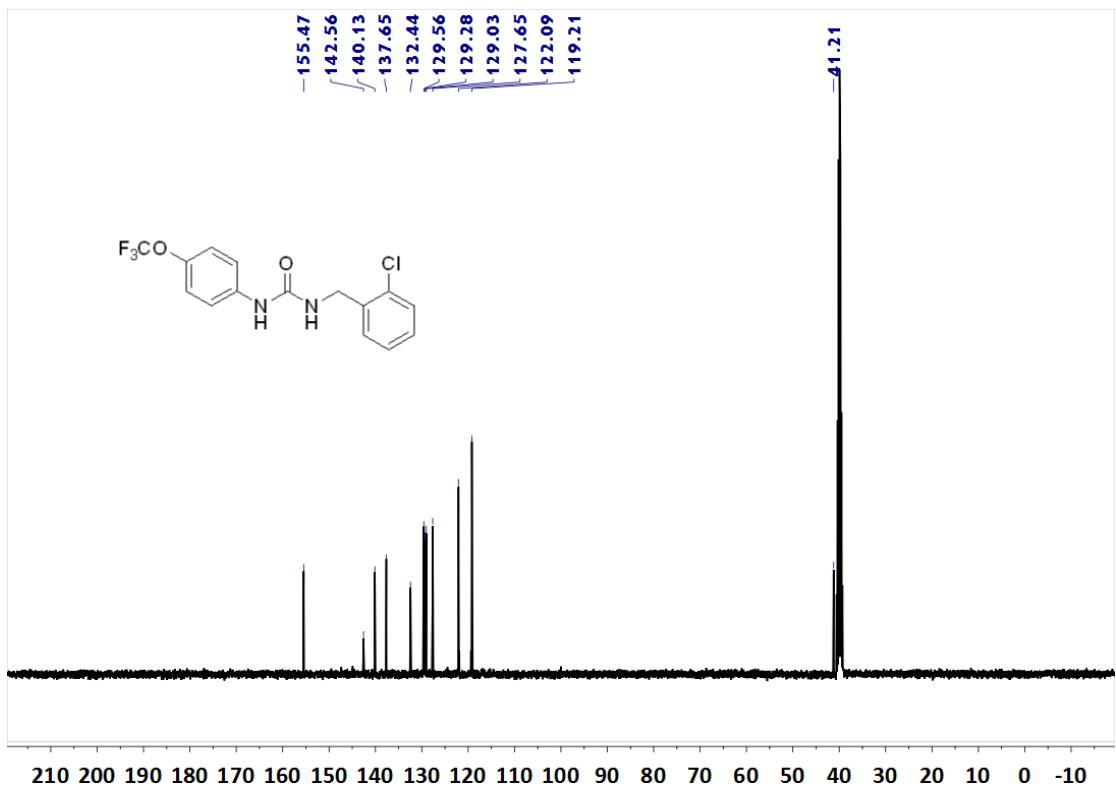
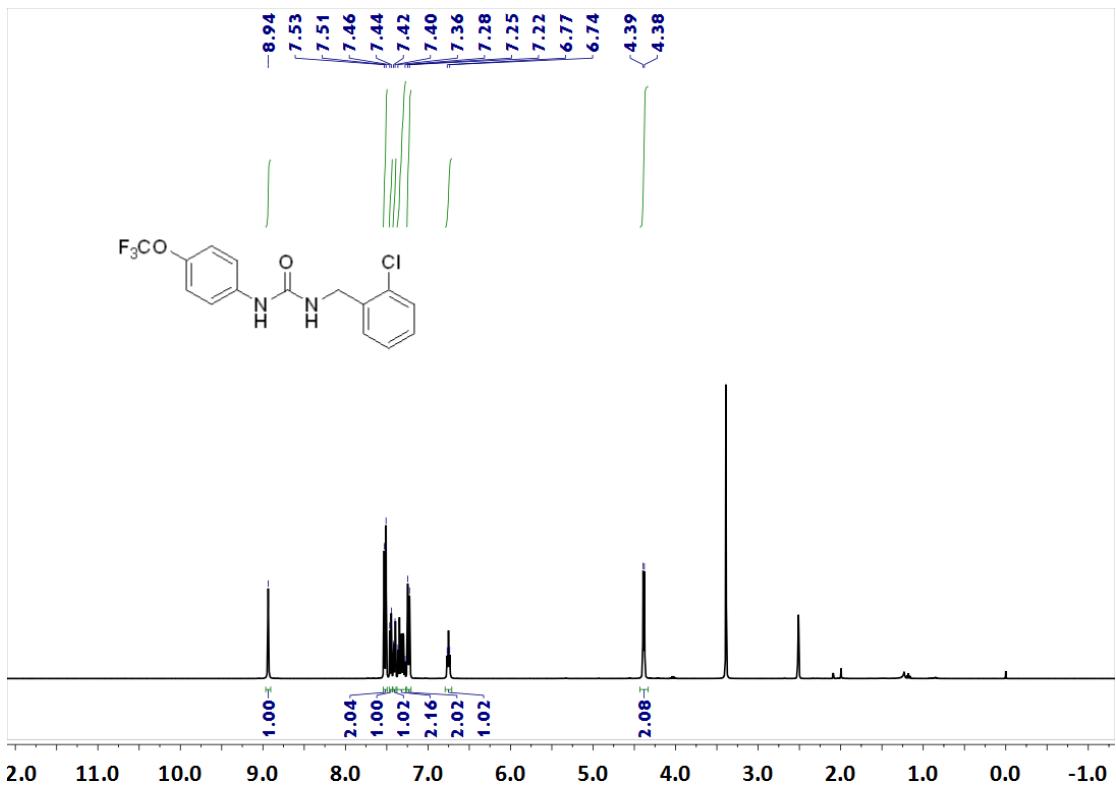


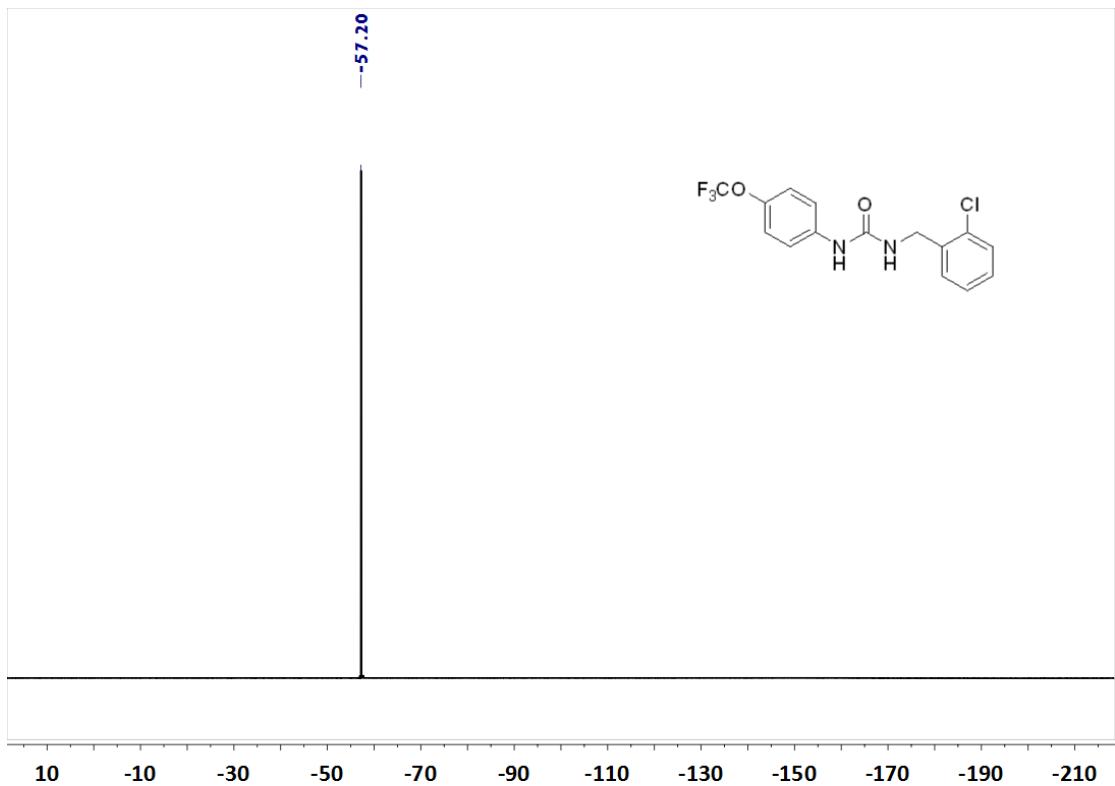
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5n**.



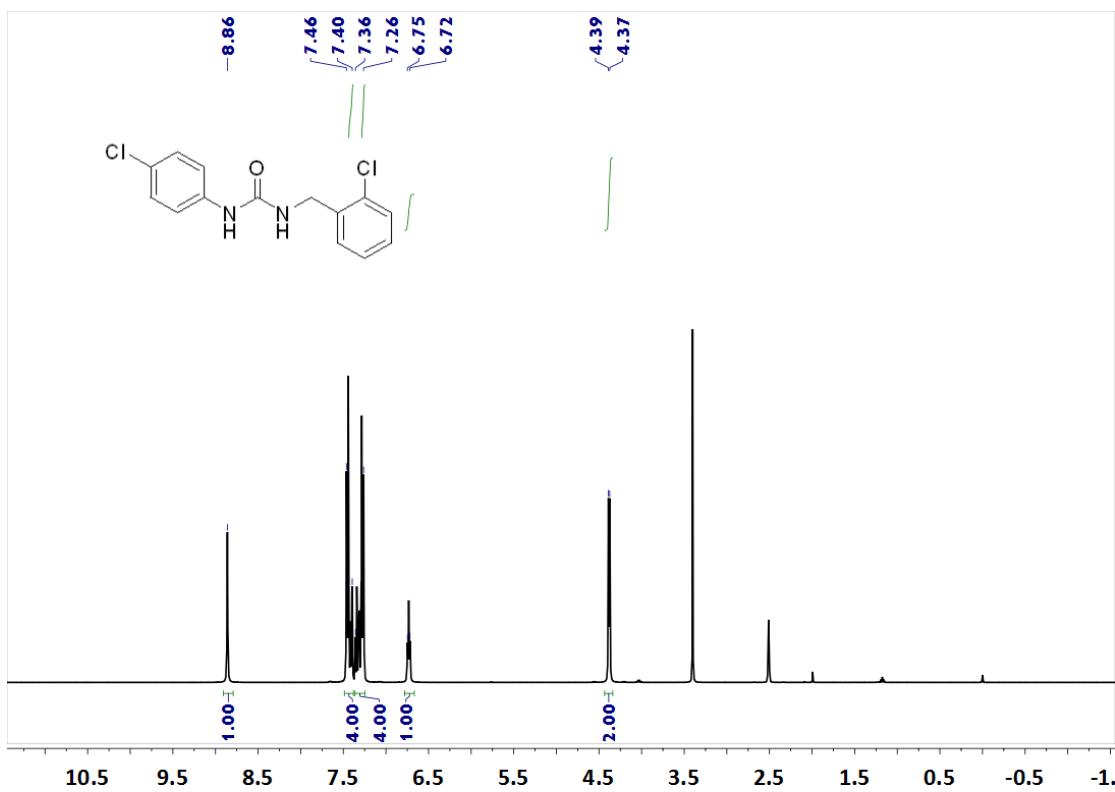


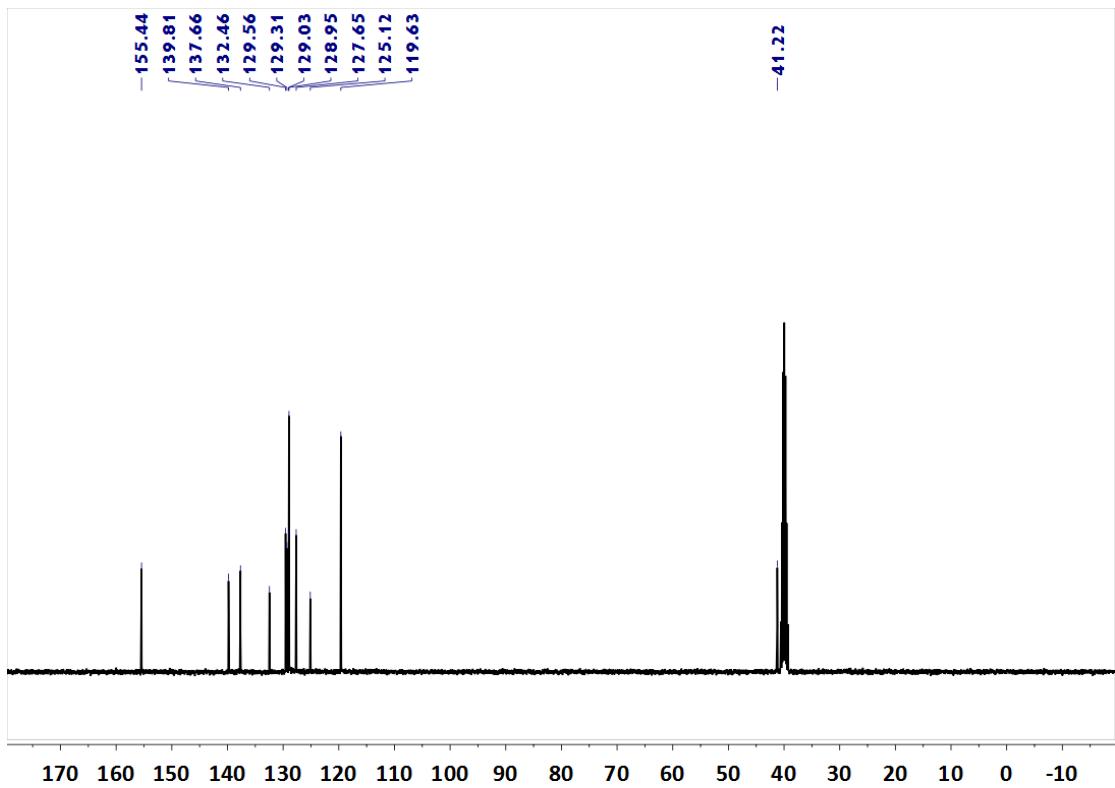
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **5o**.



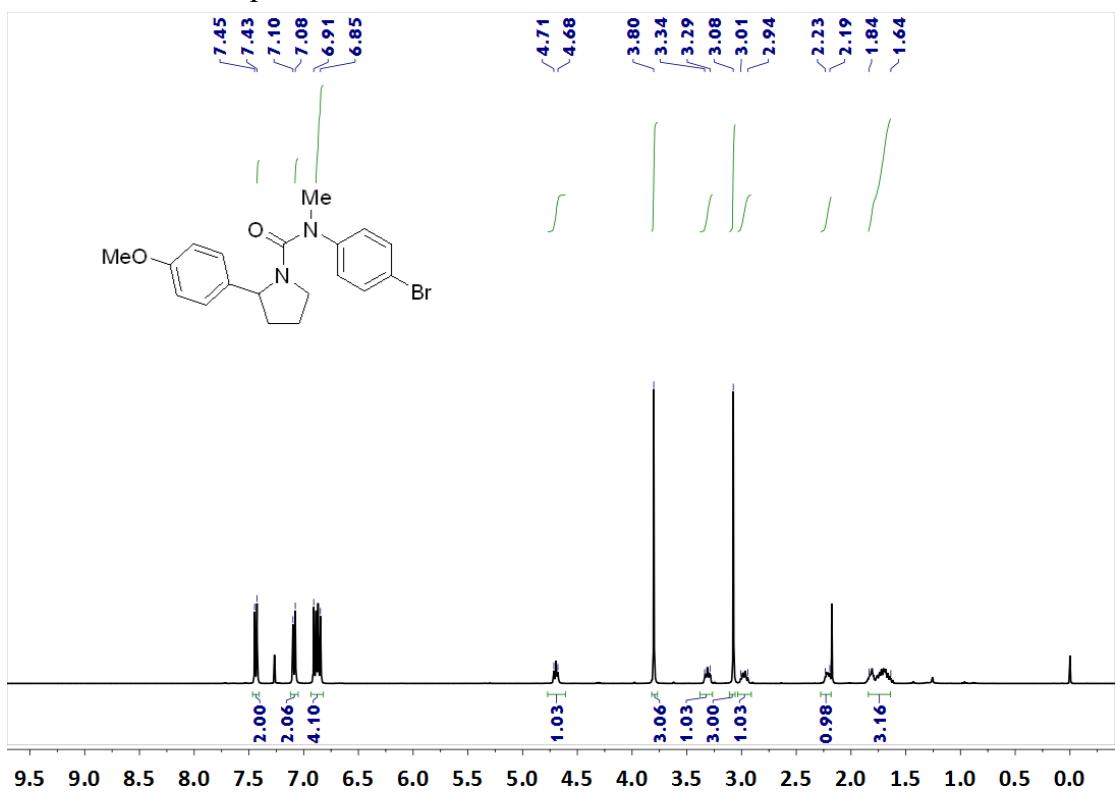


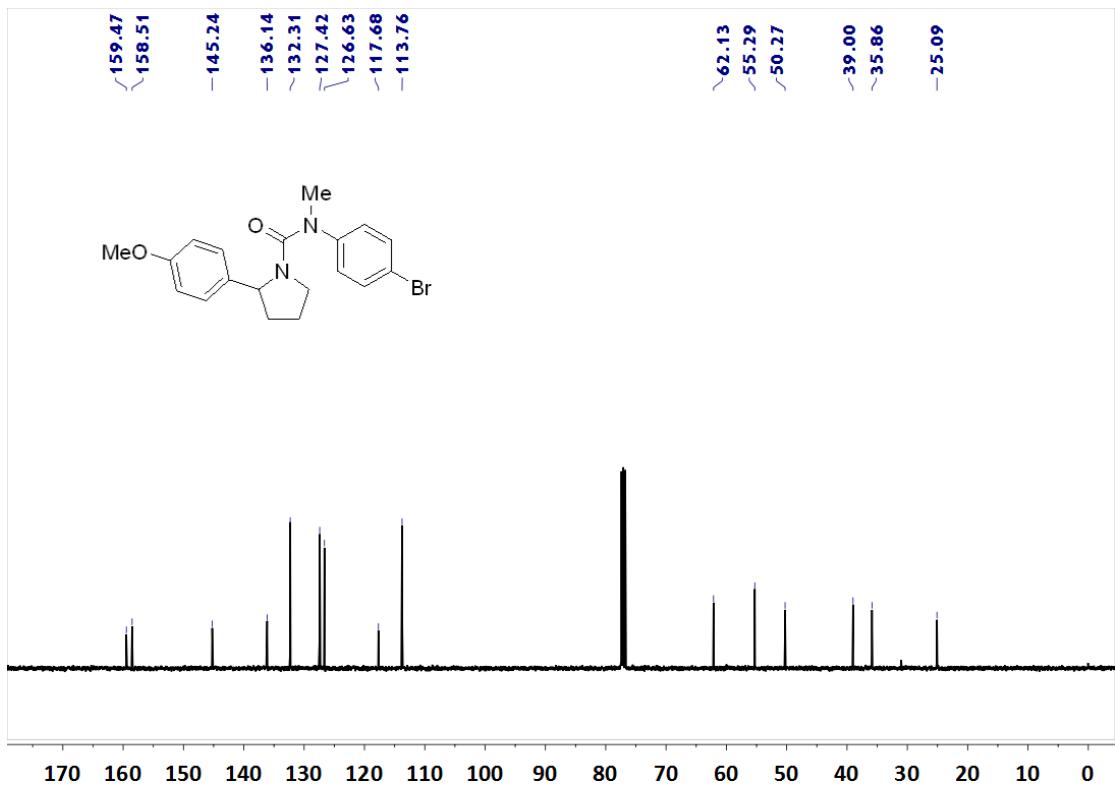
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **5p**.



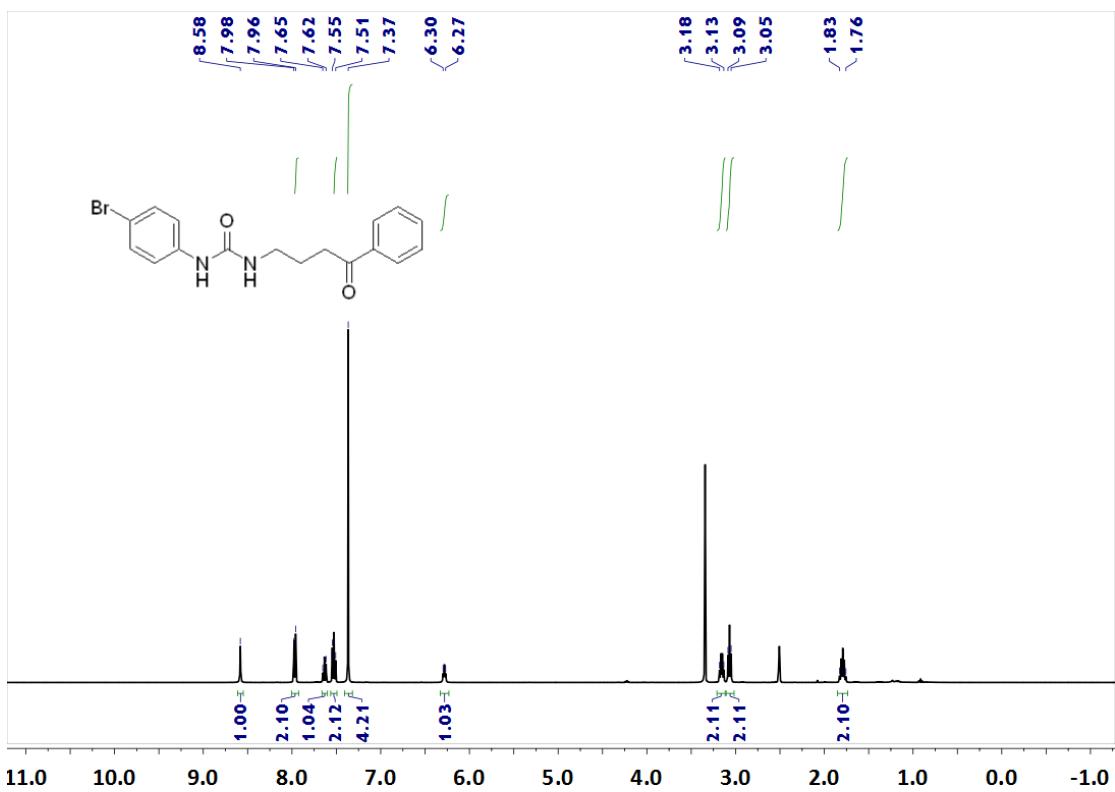


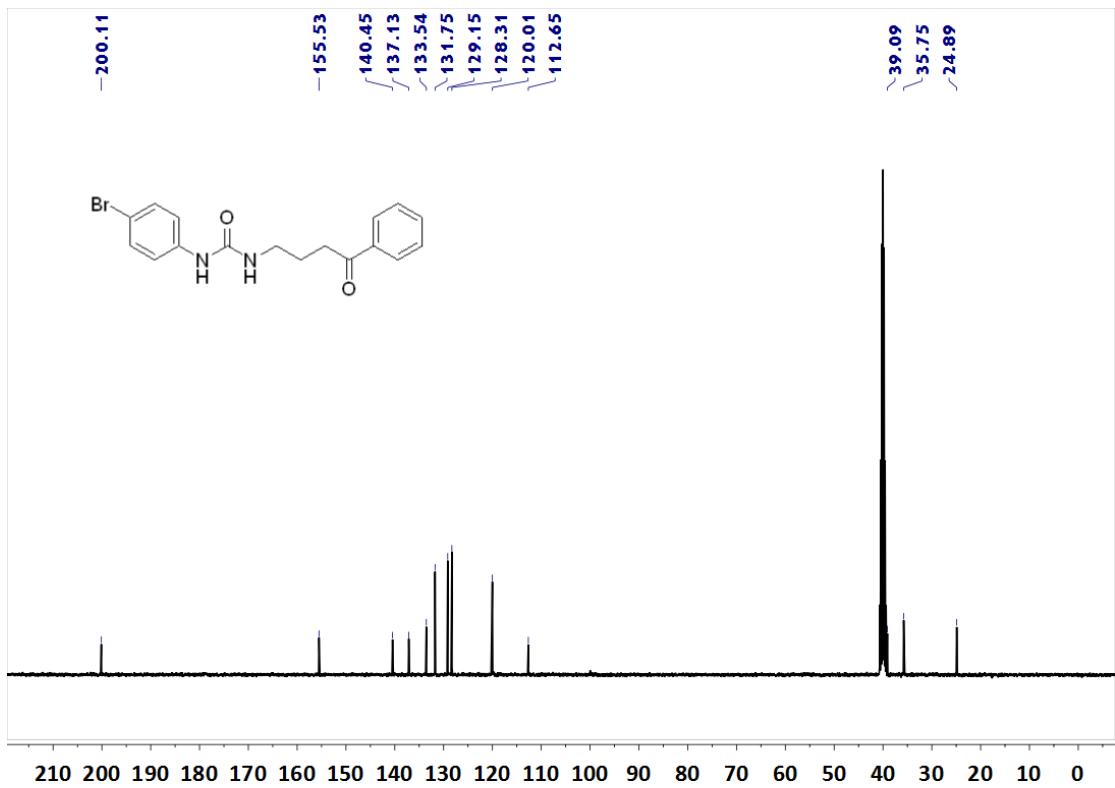
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 1cc.



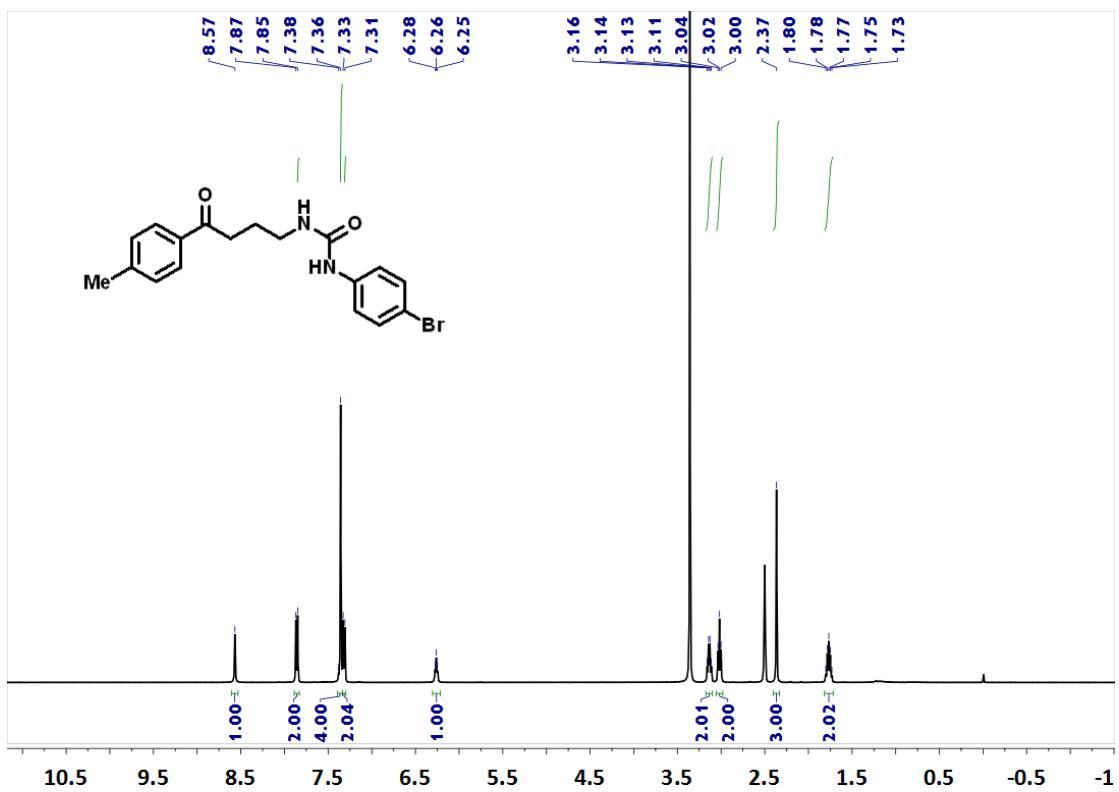


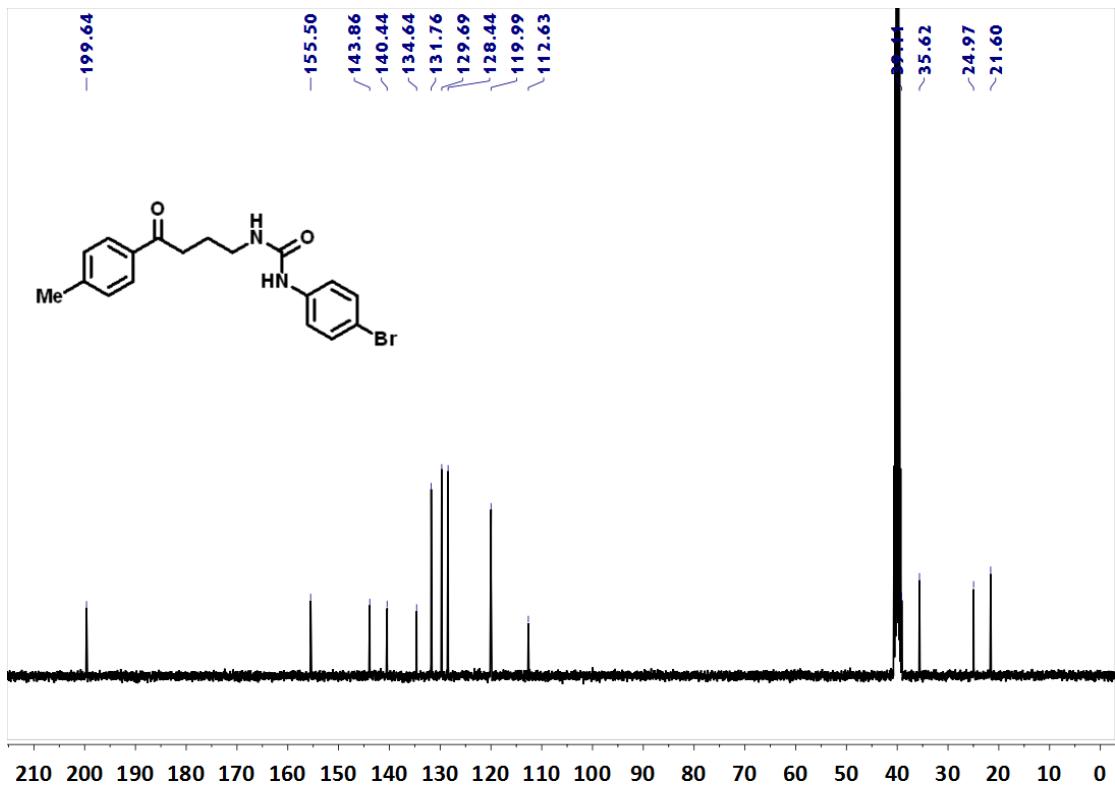
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2a**.



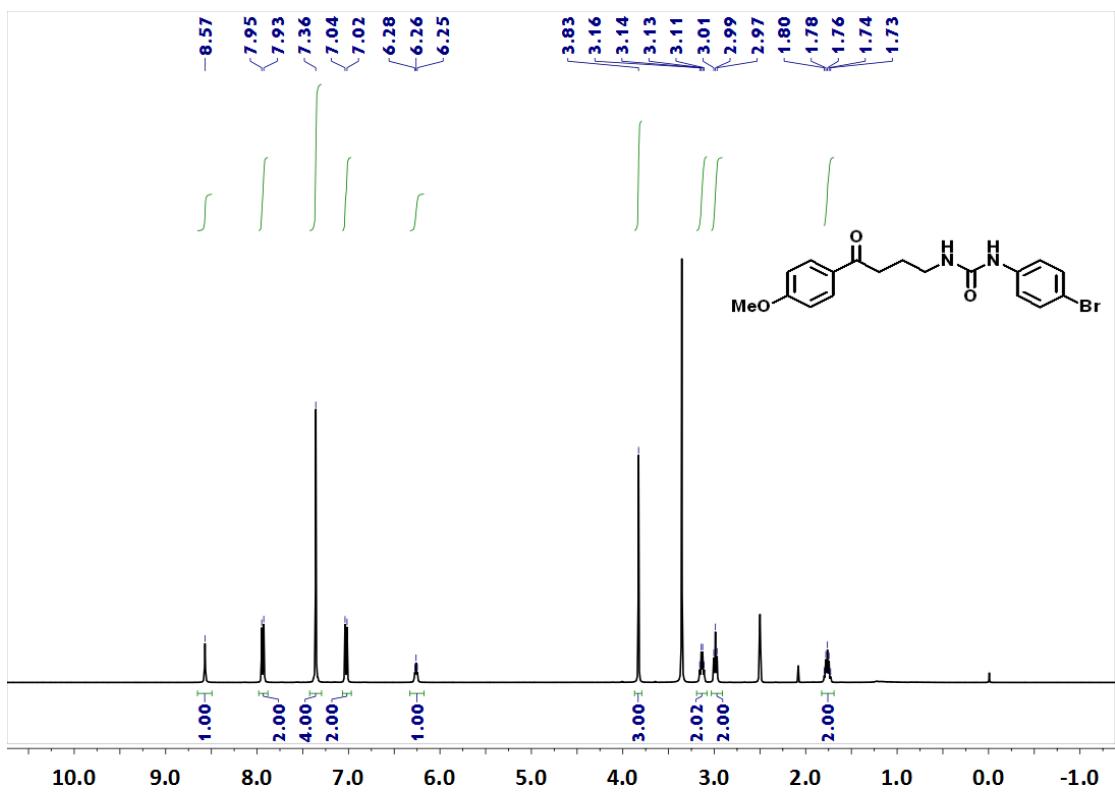


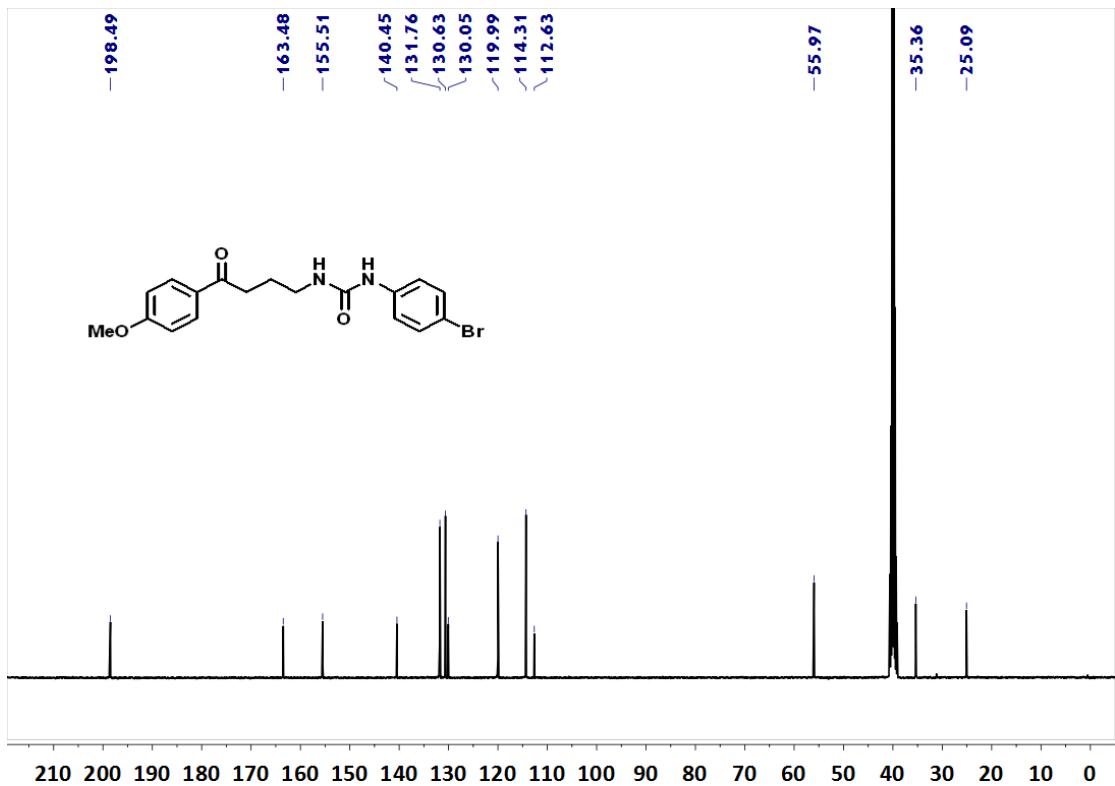
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2b**.



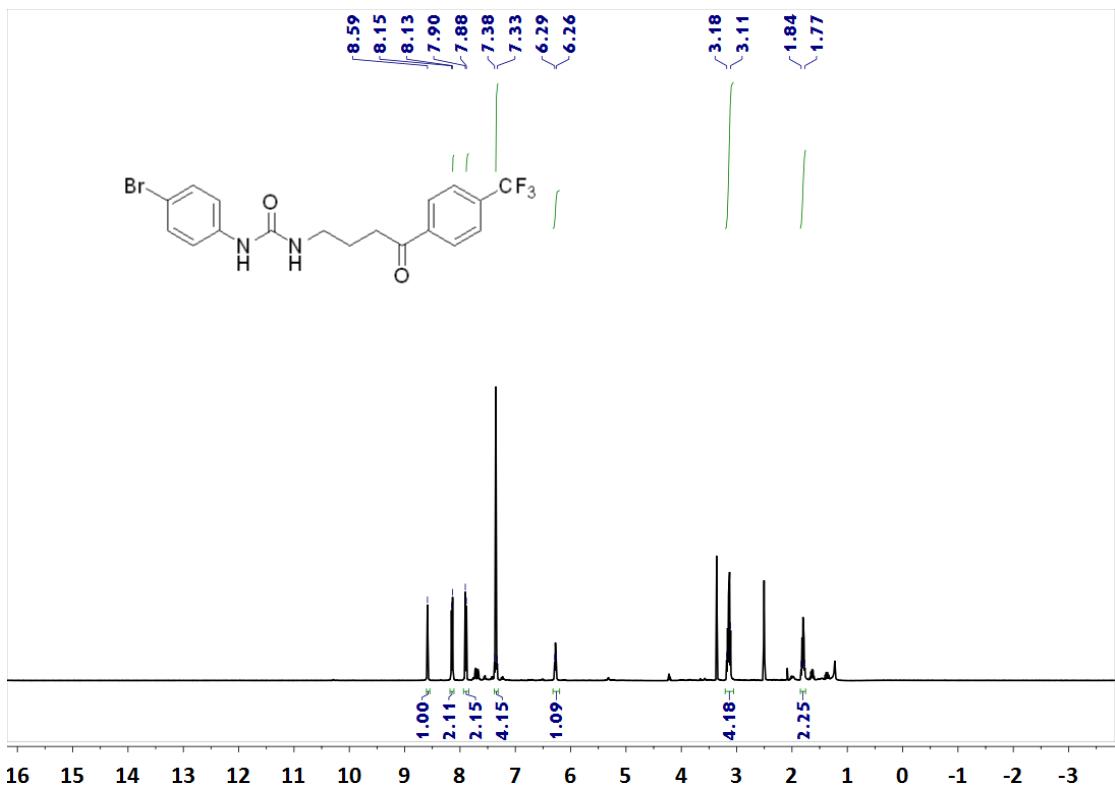


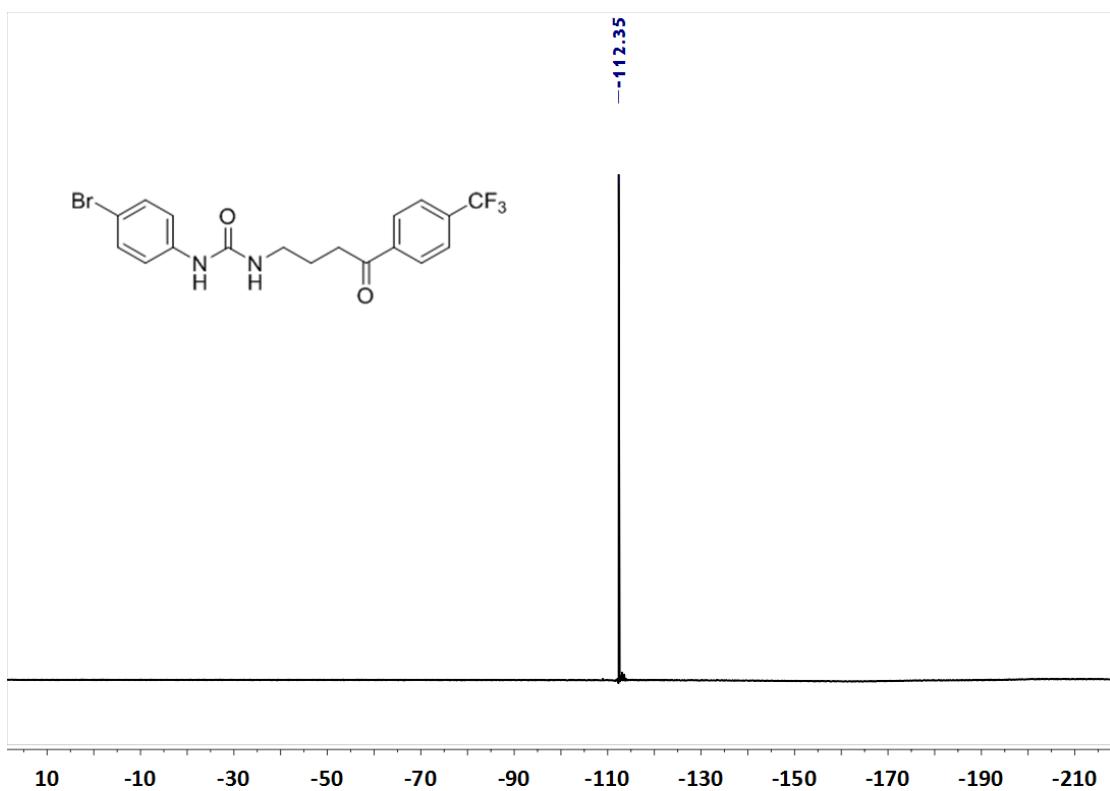
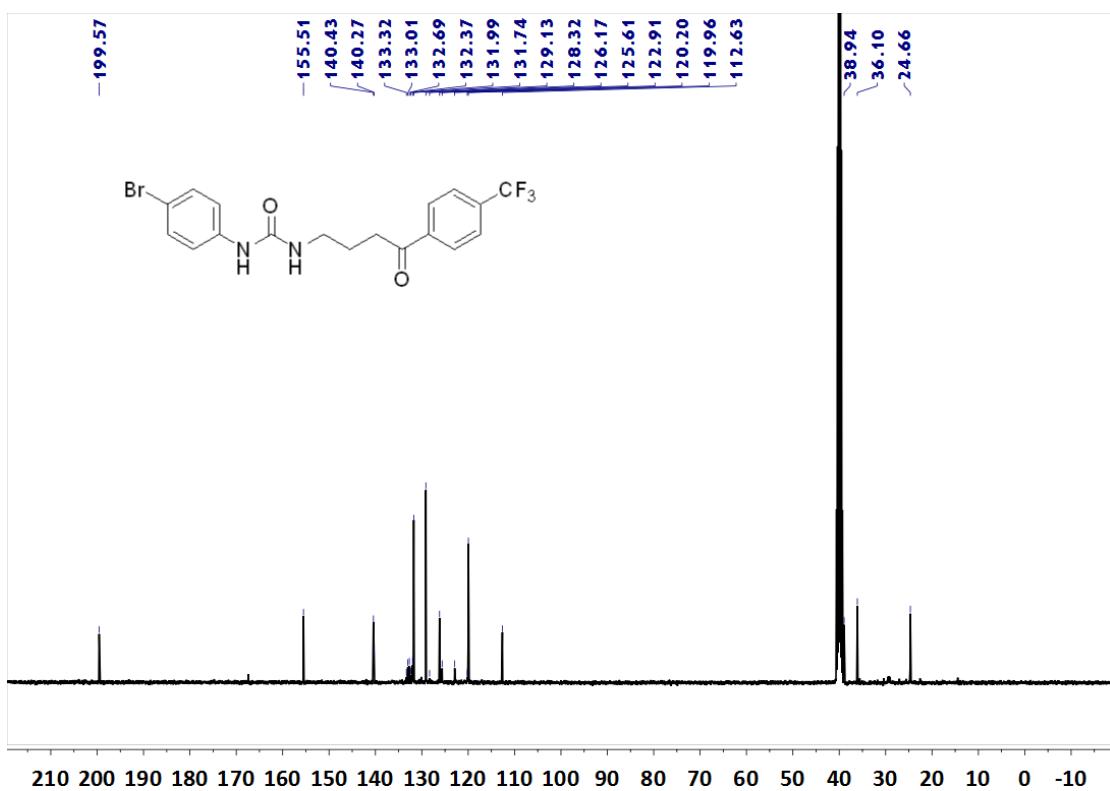
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2c**.



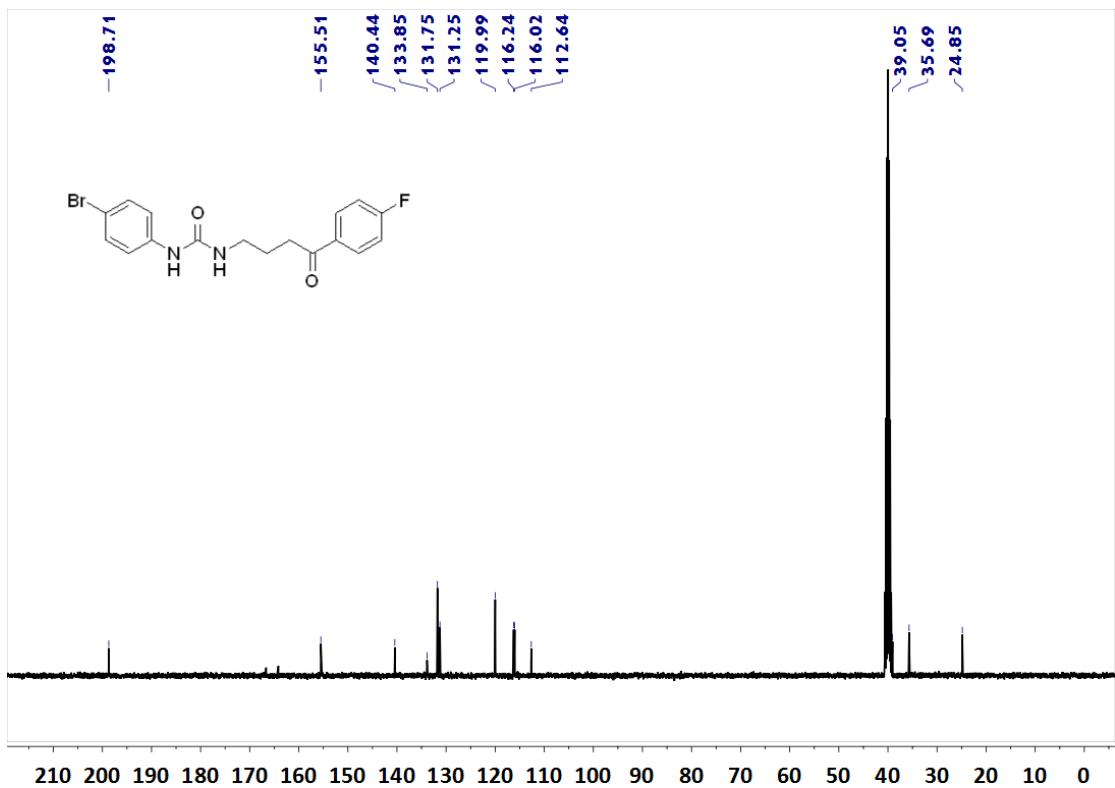
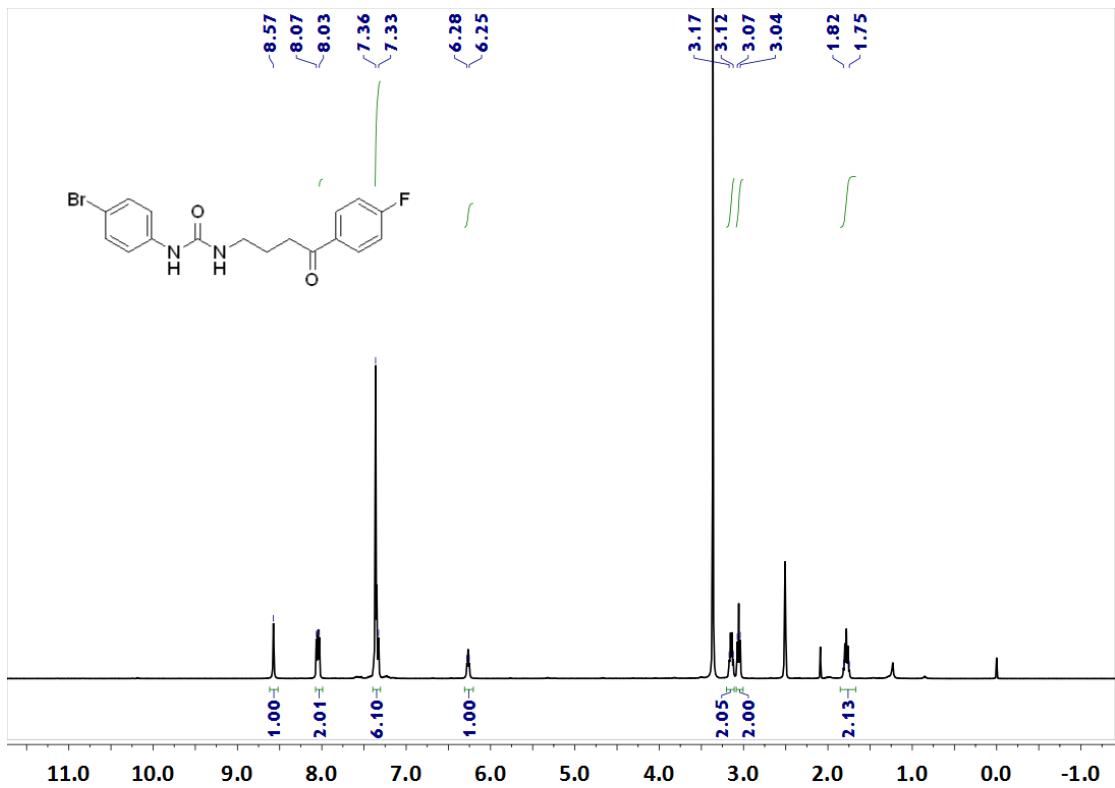


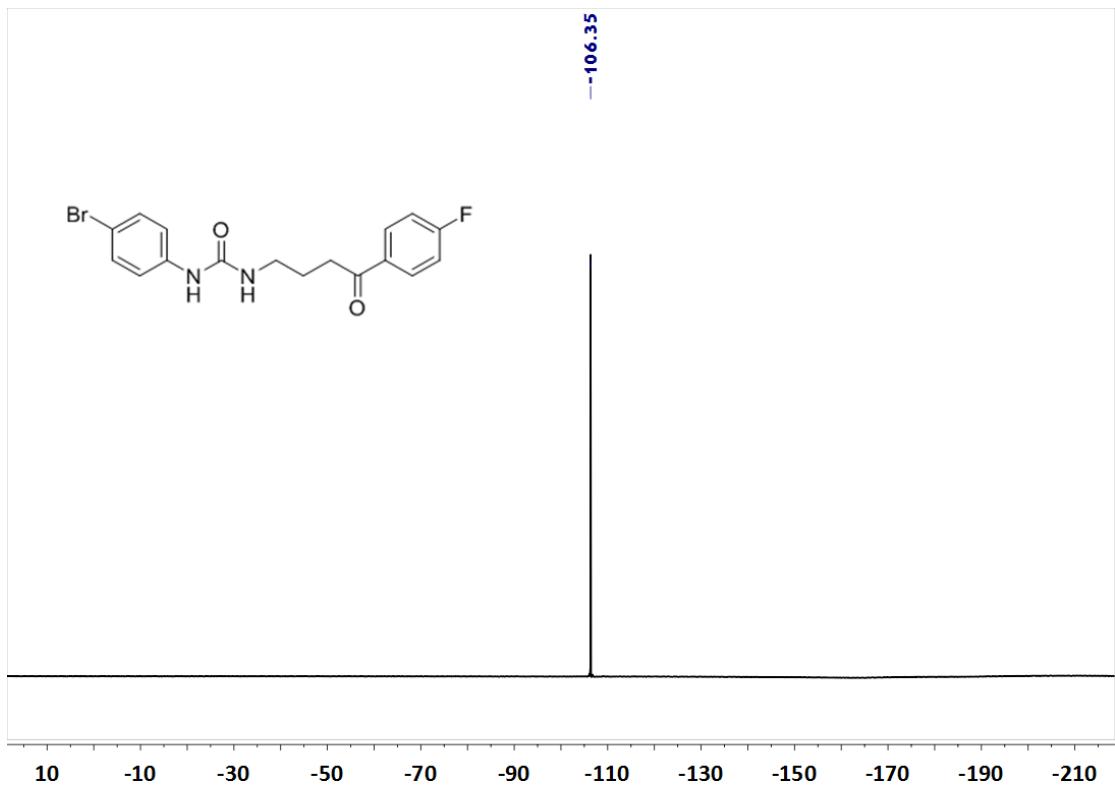
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2d**.



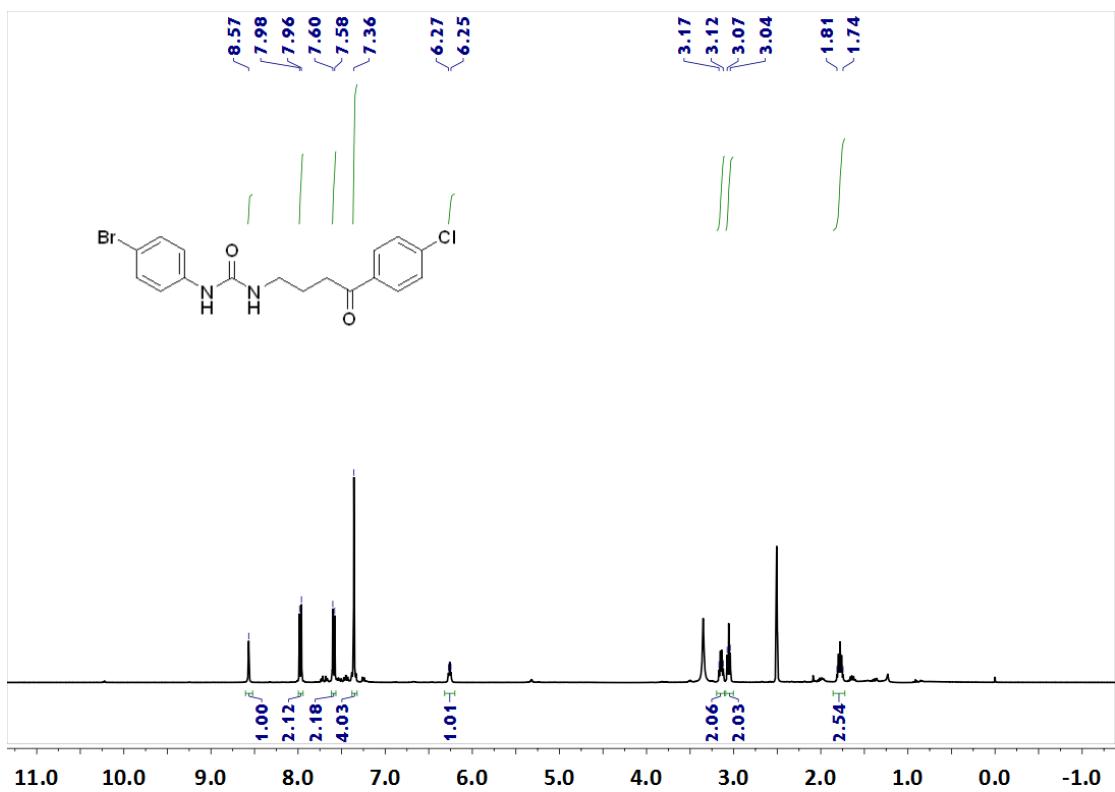


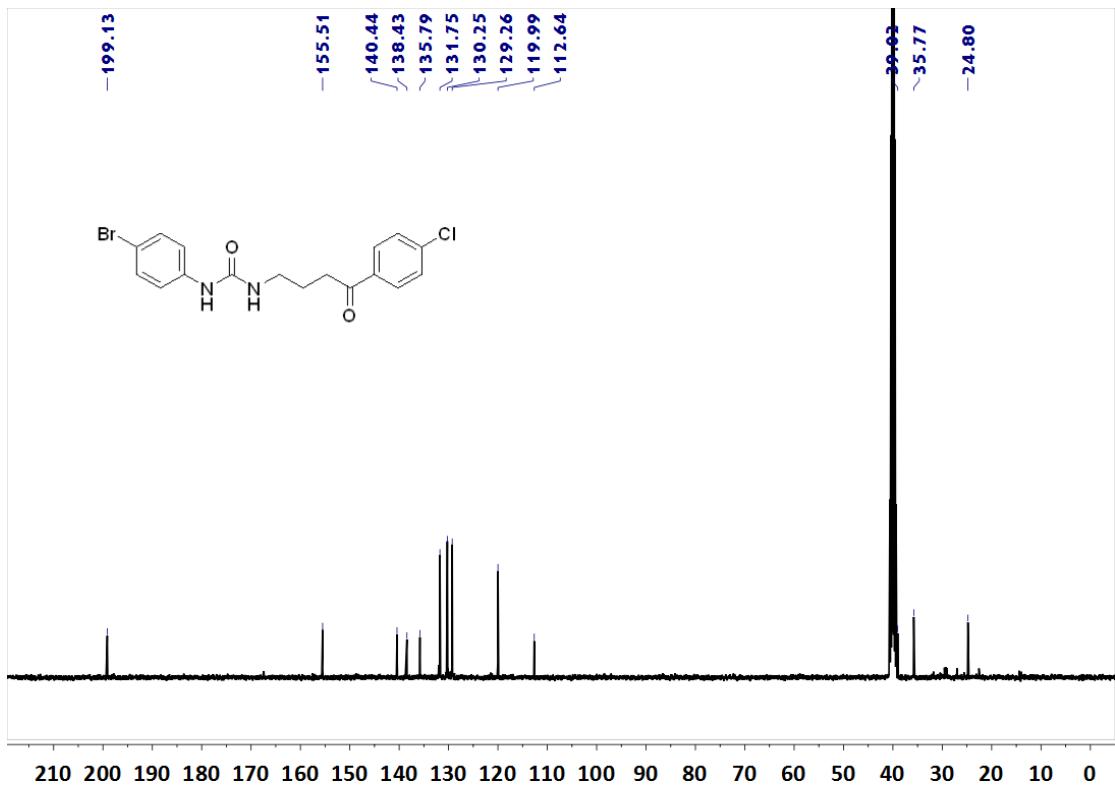
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2e**.



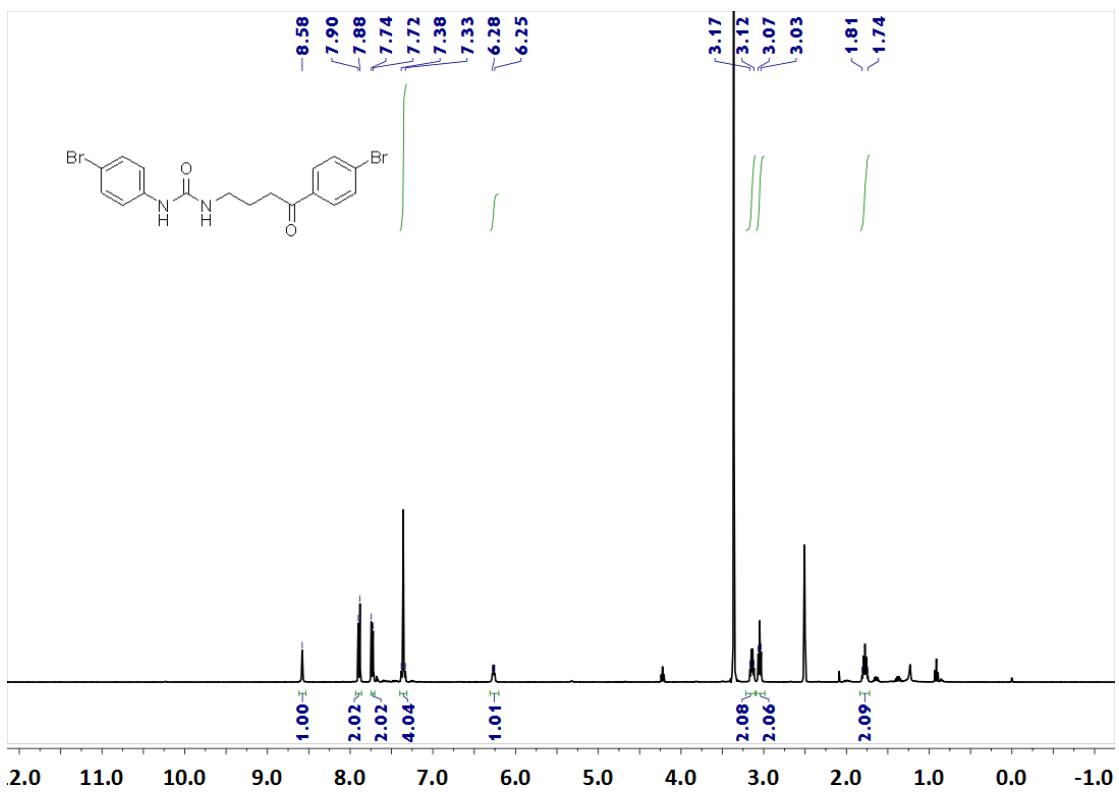


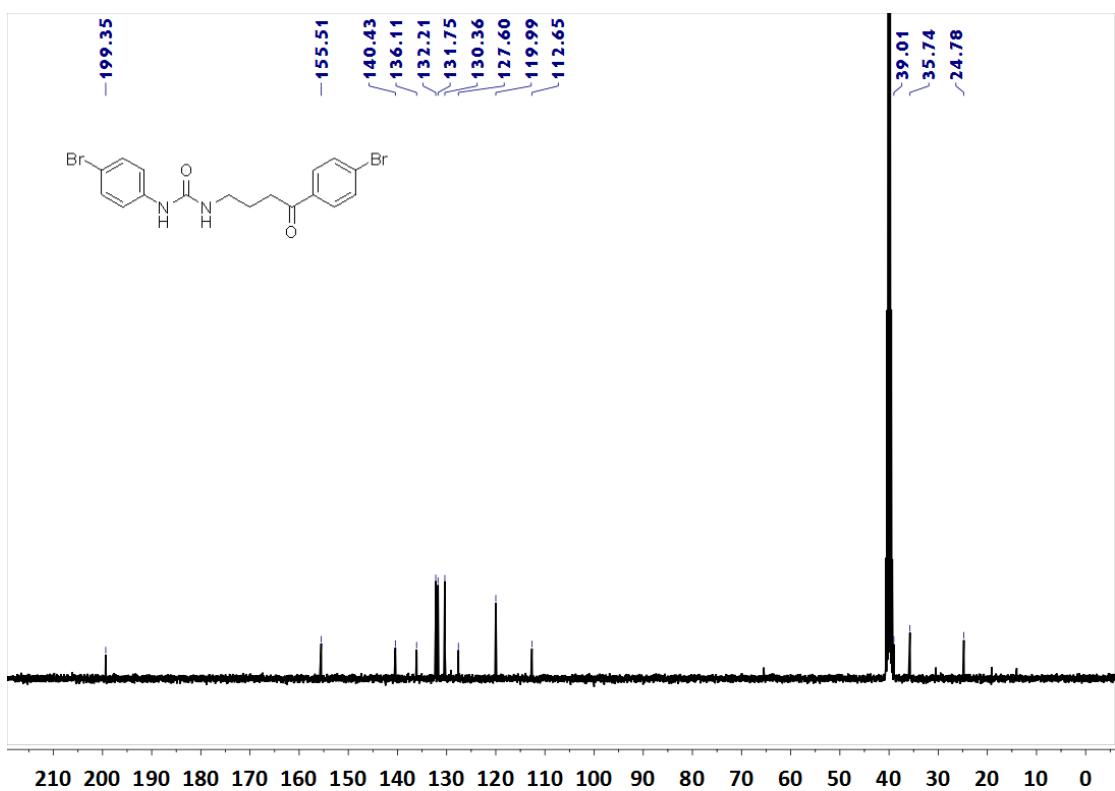
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2f**.



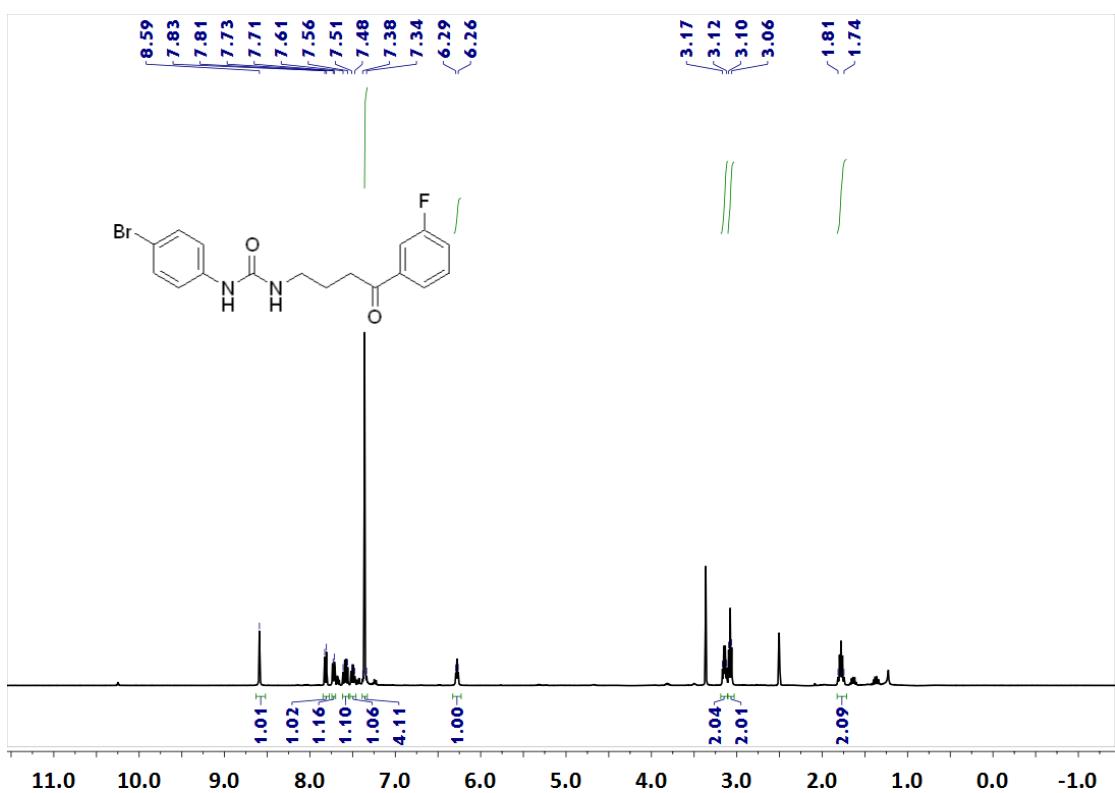


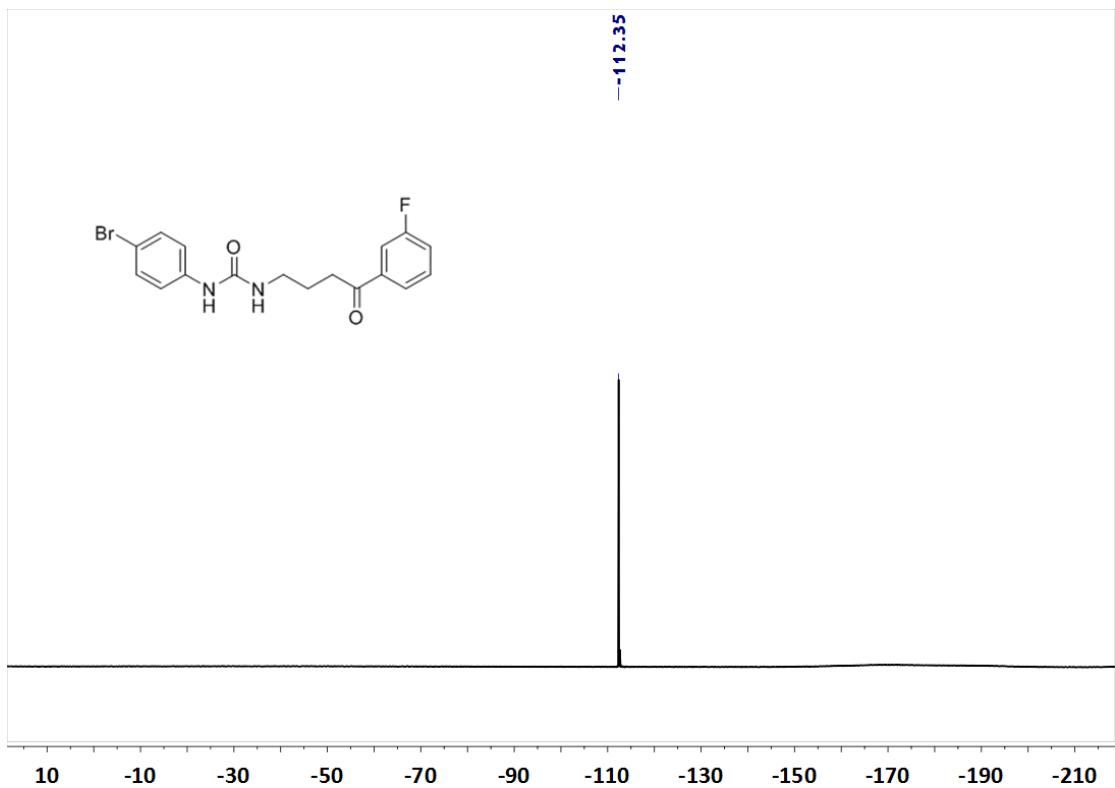
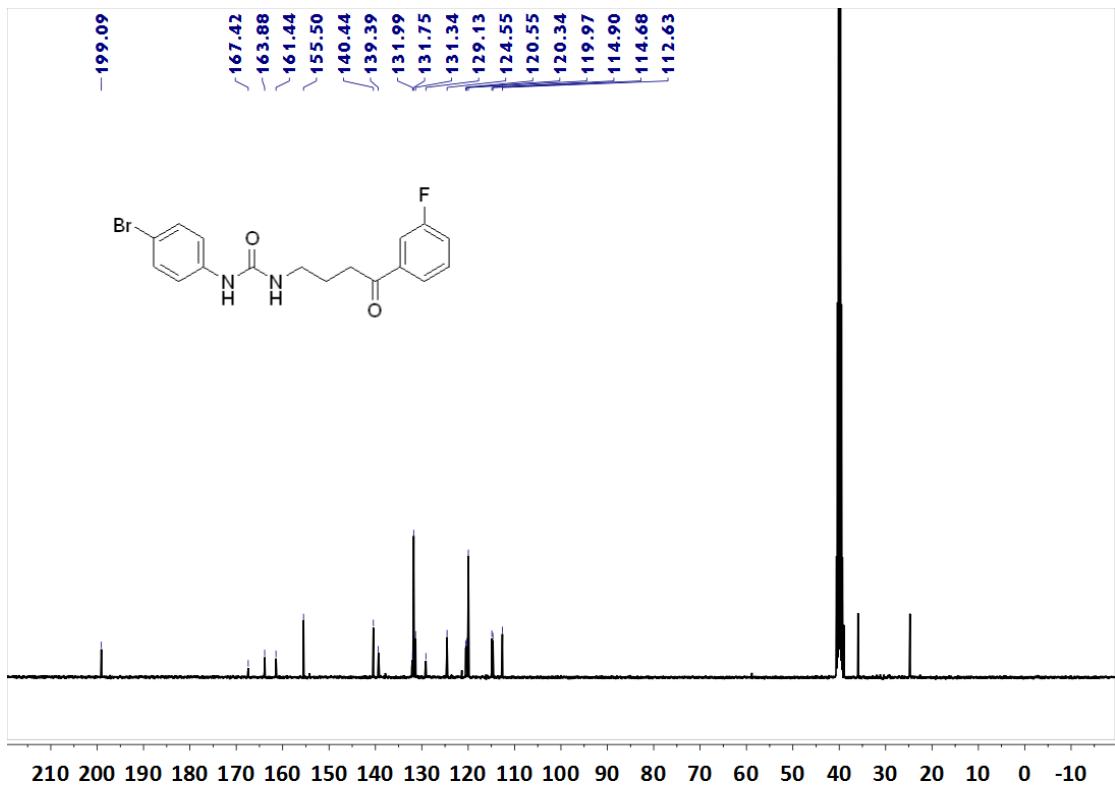
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2g**.



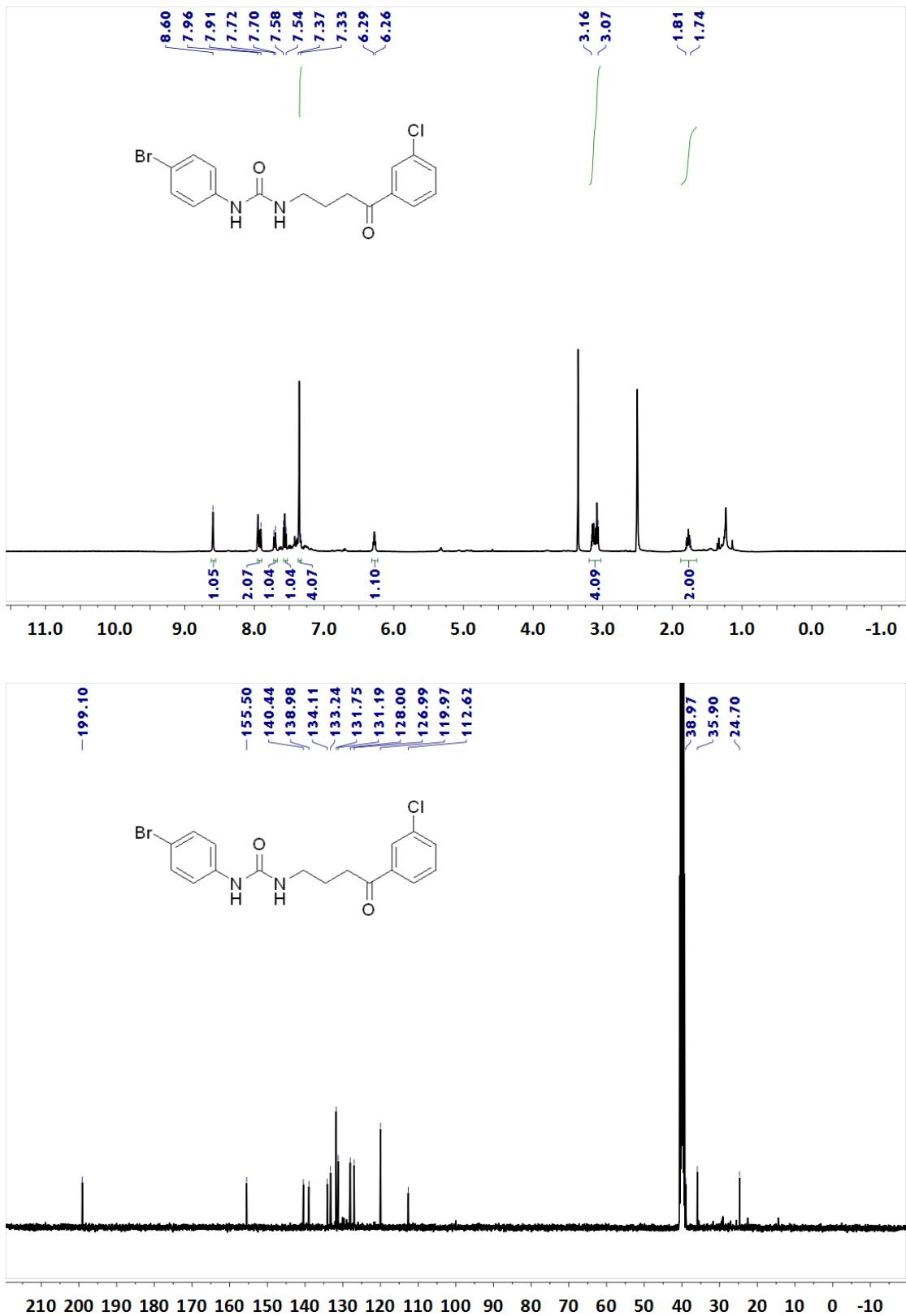


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2h**.

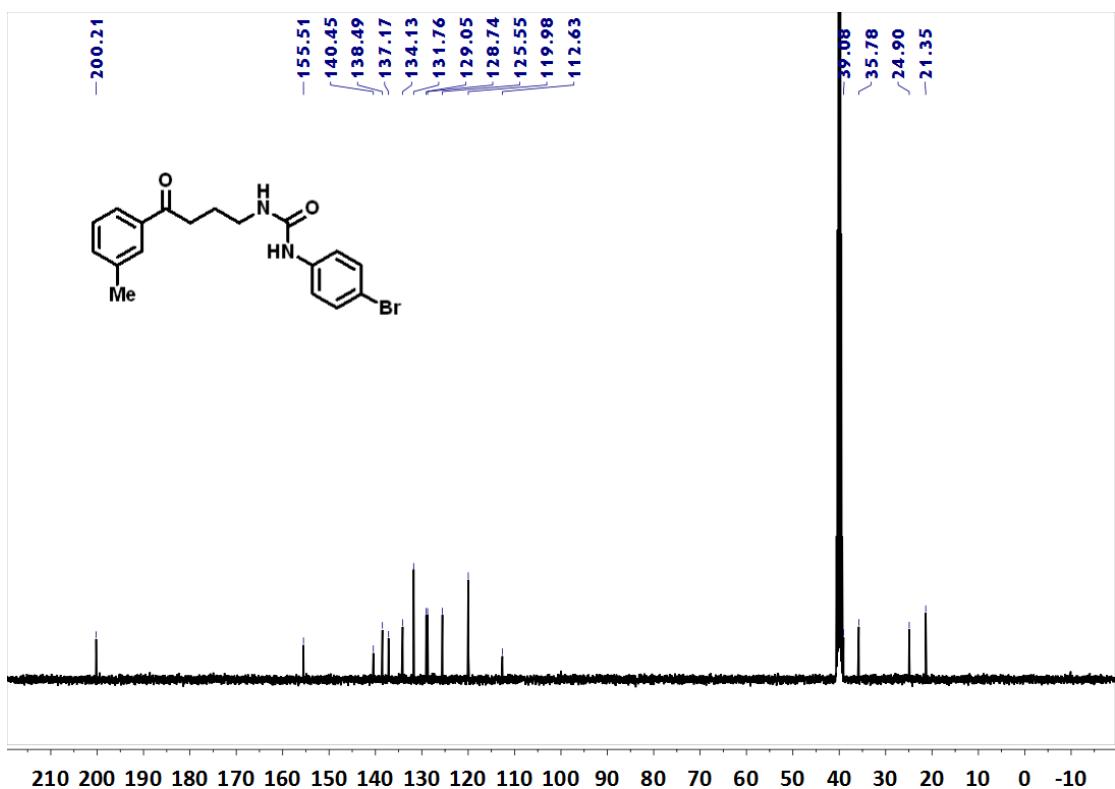
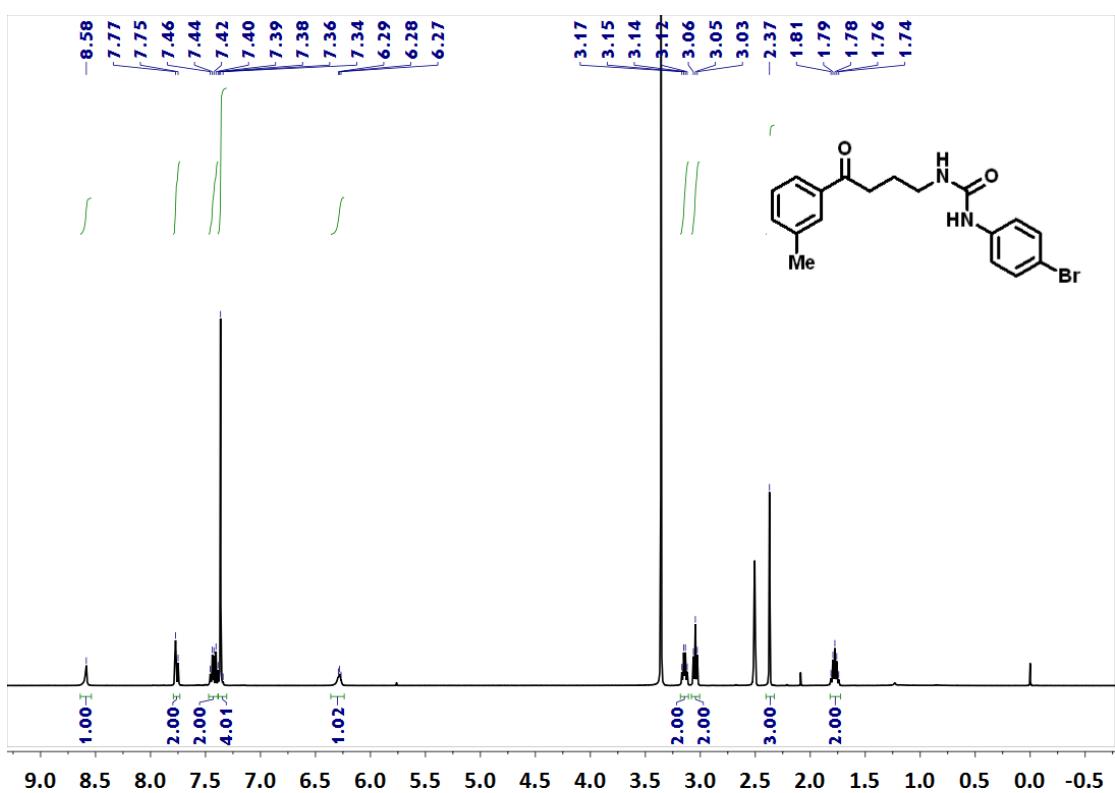




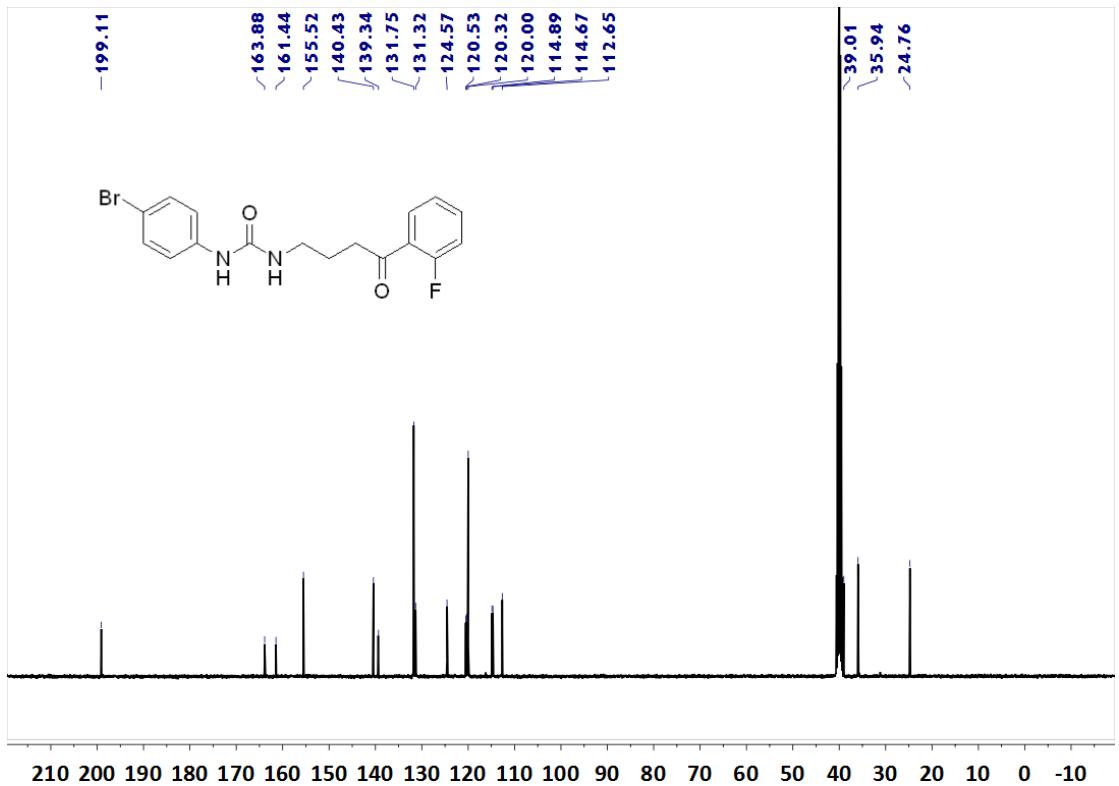
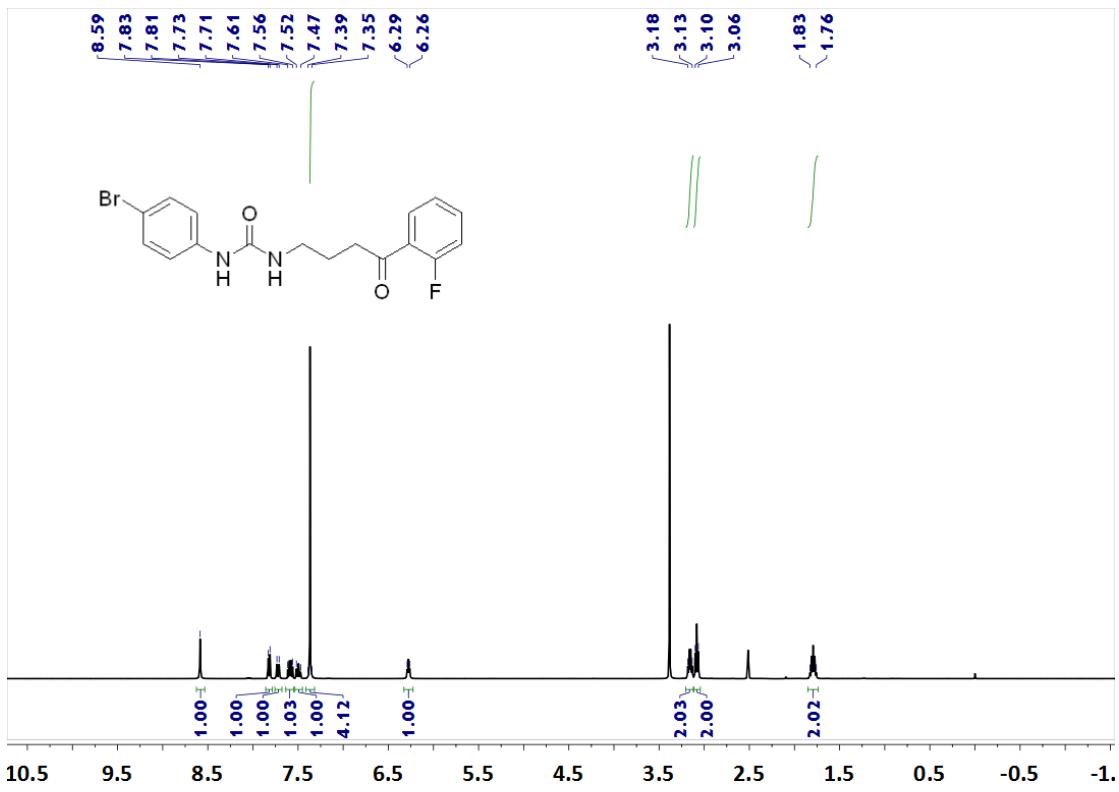
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2i**.

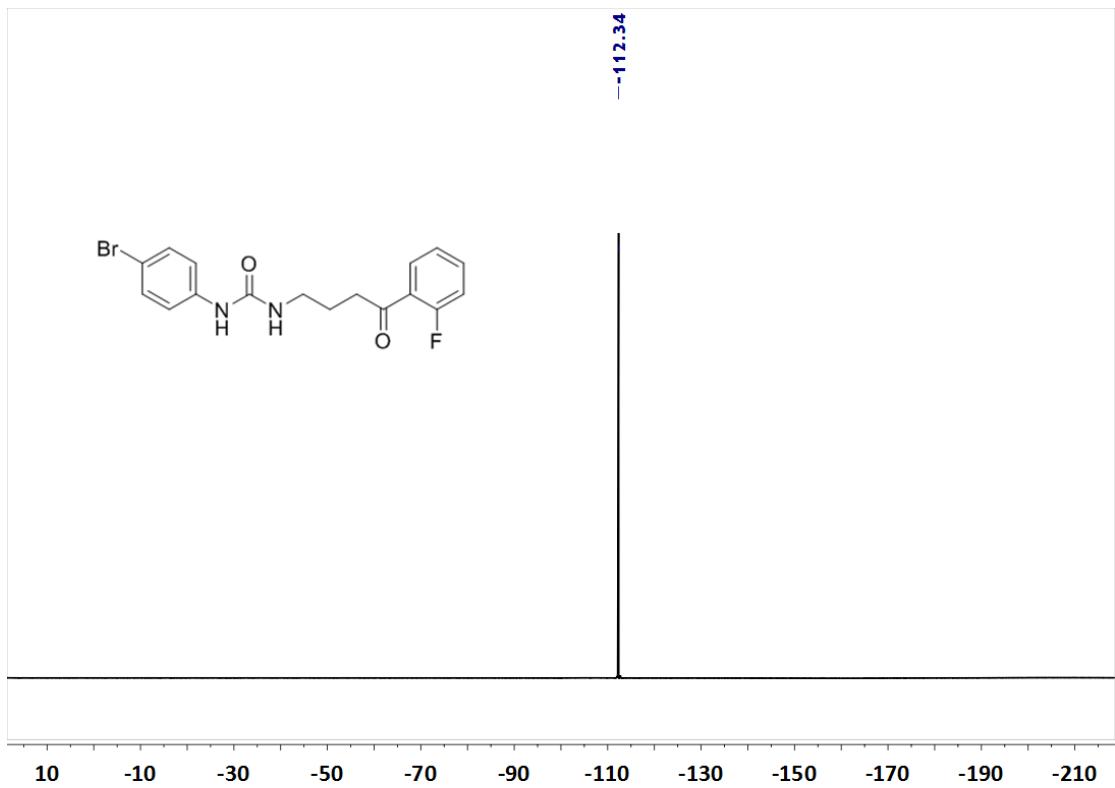


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2j**.

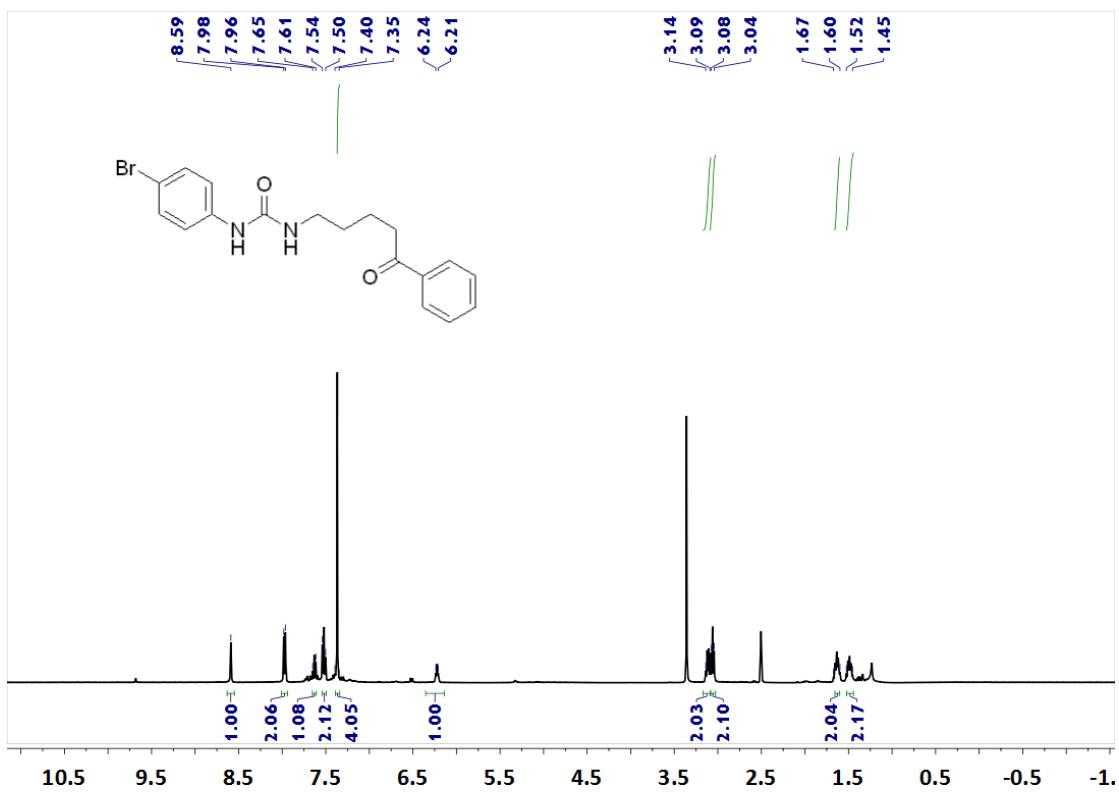


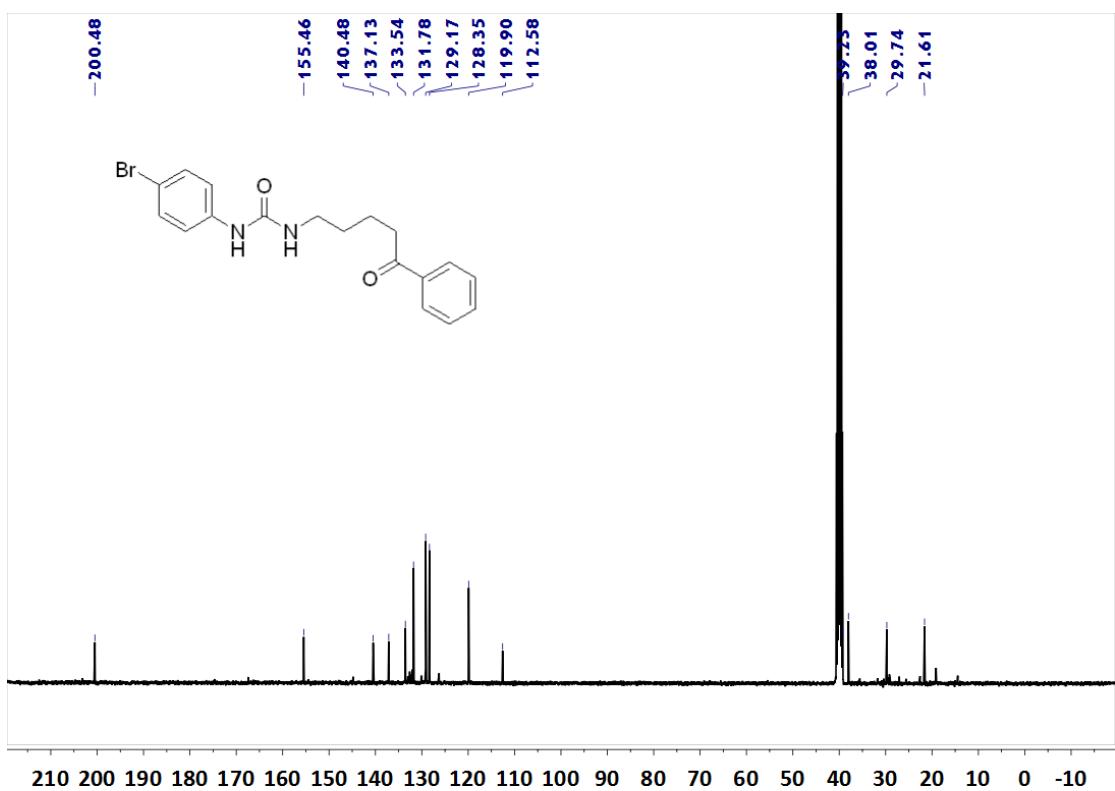
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 2k.



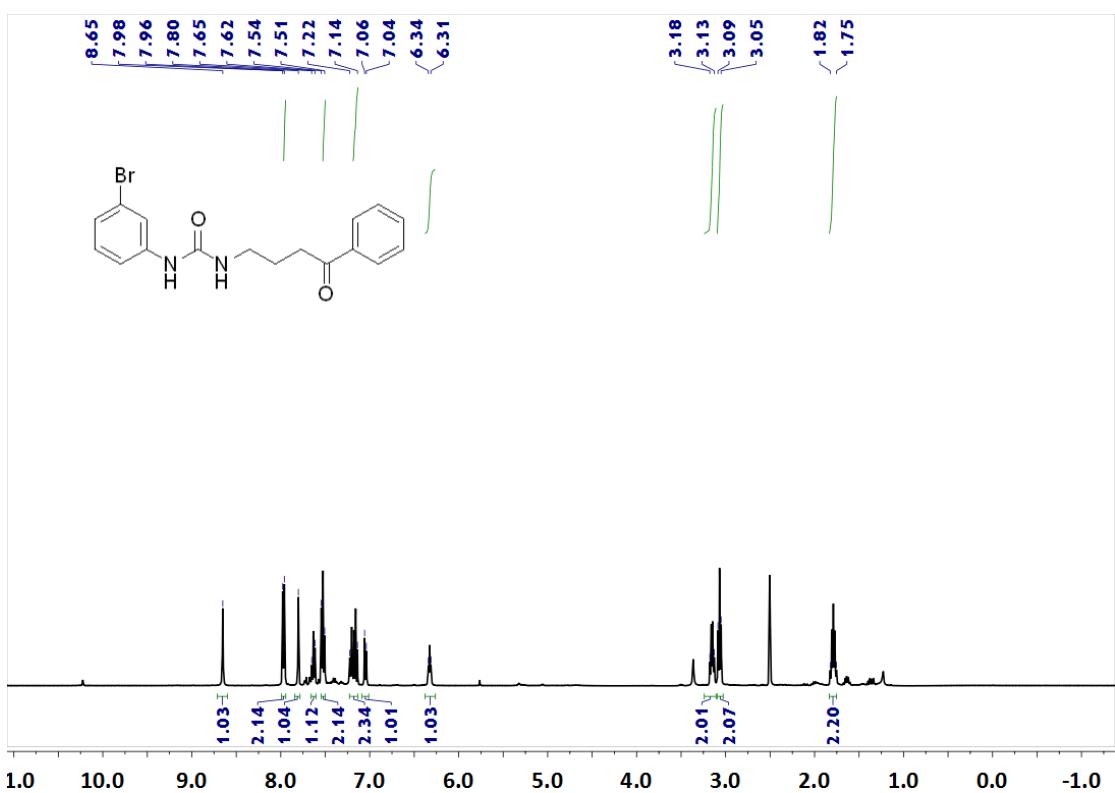


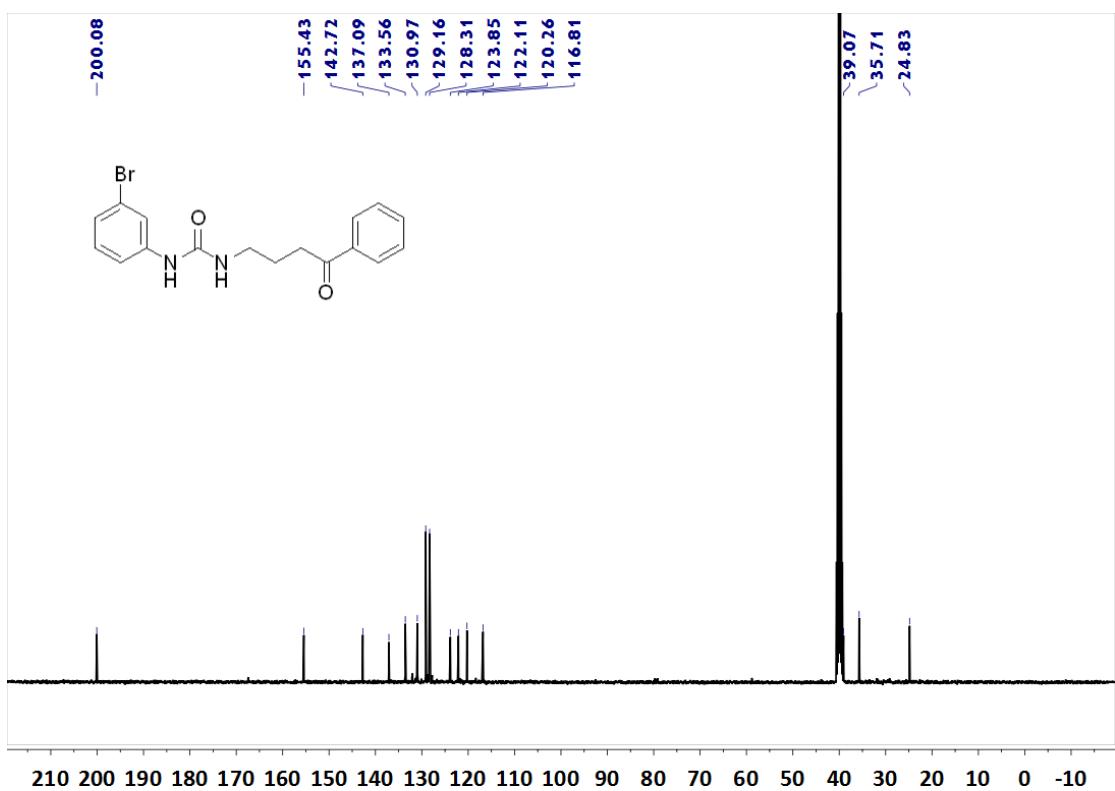
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2l**.



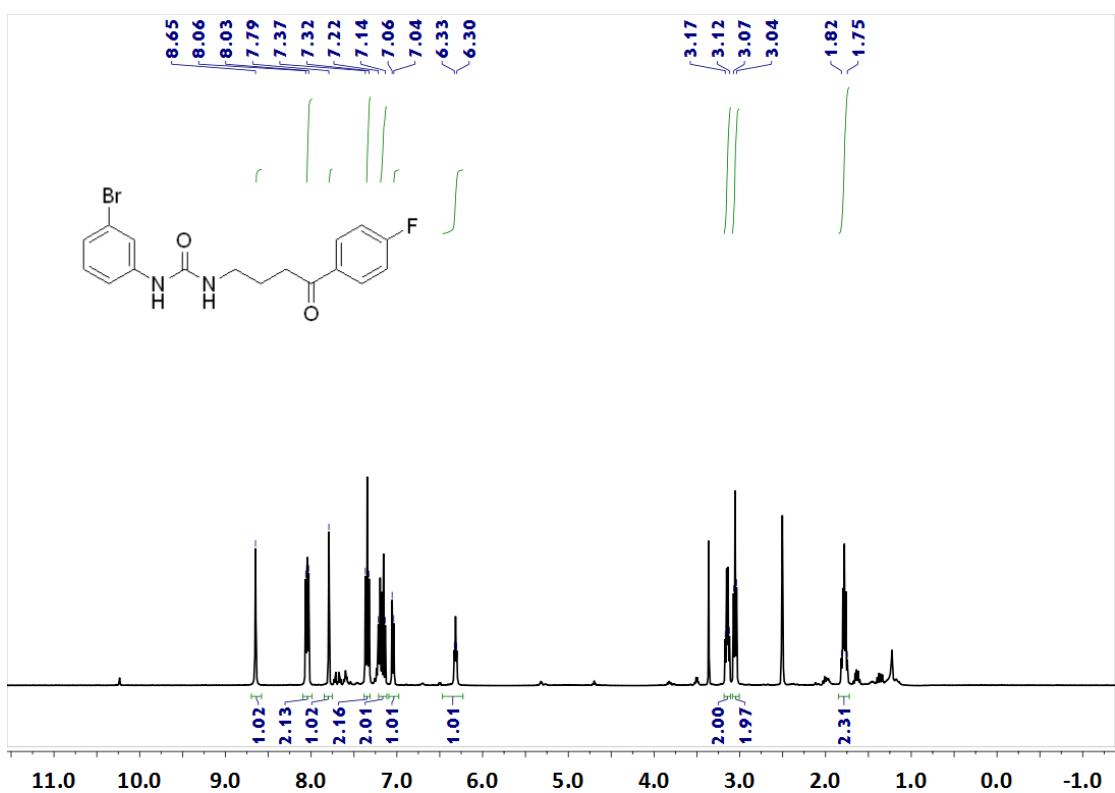


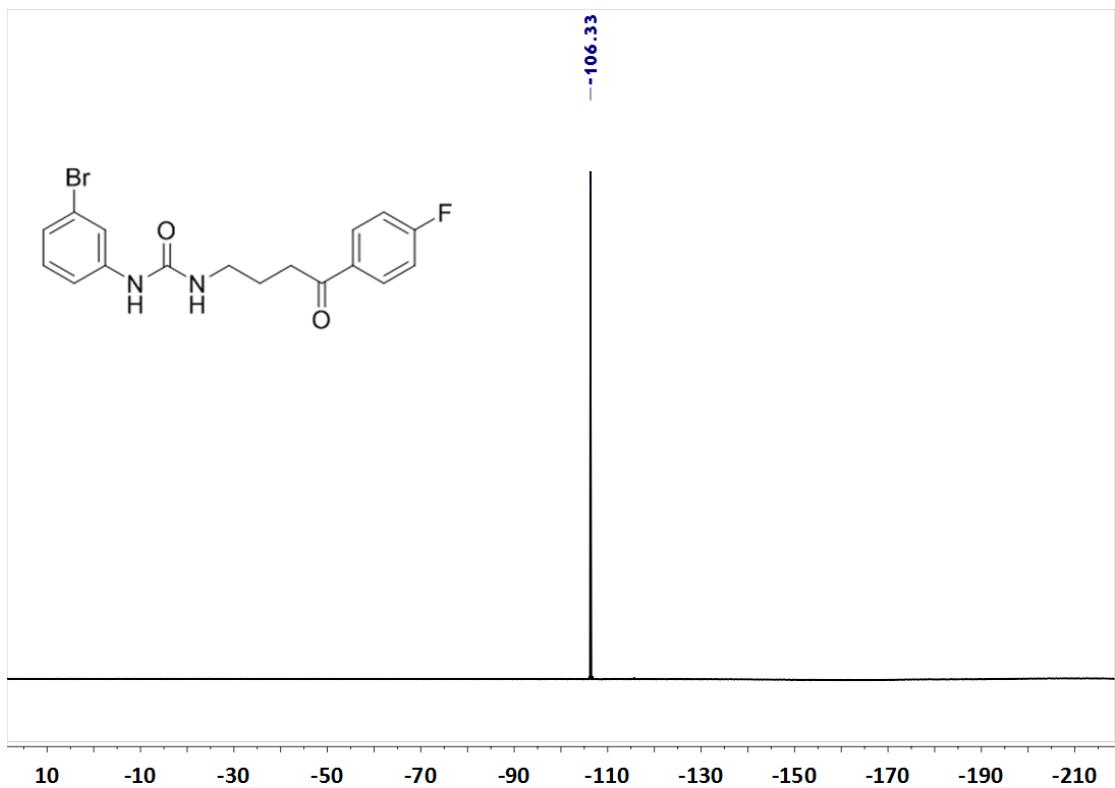
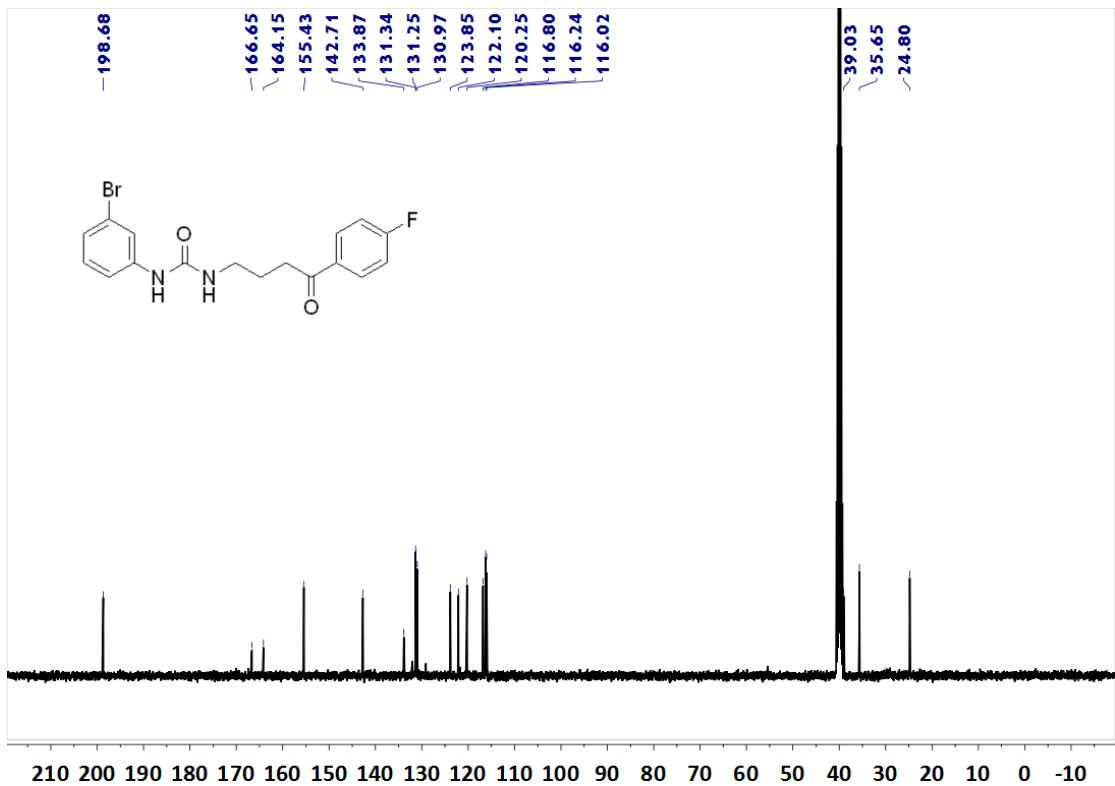
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2m**.



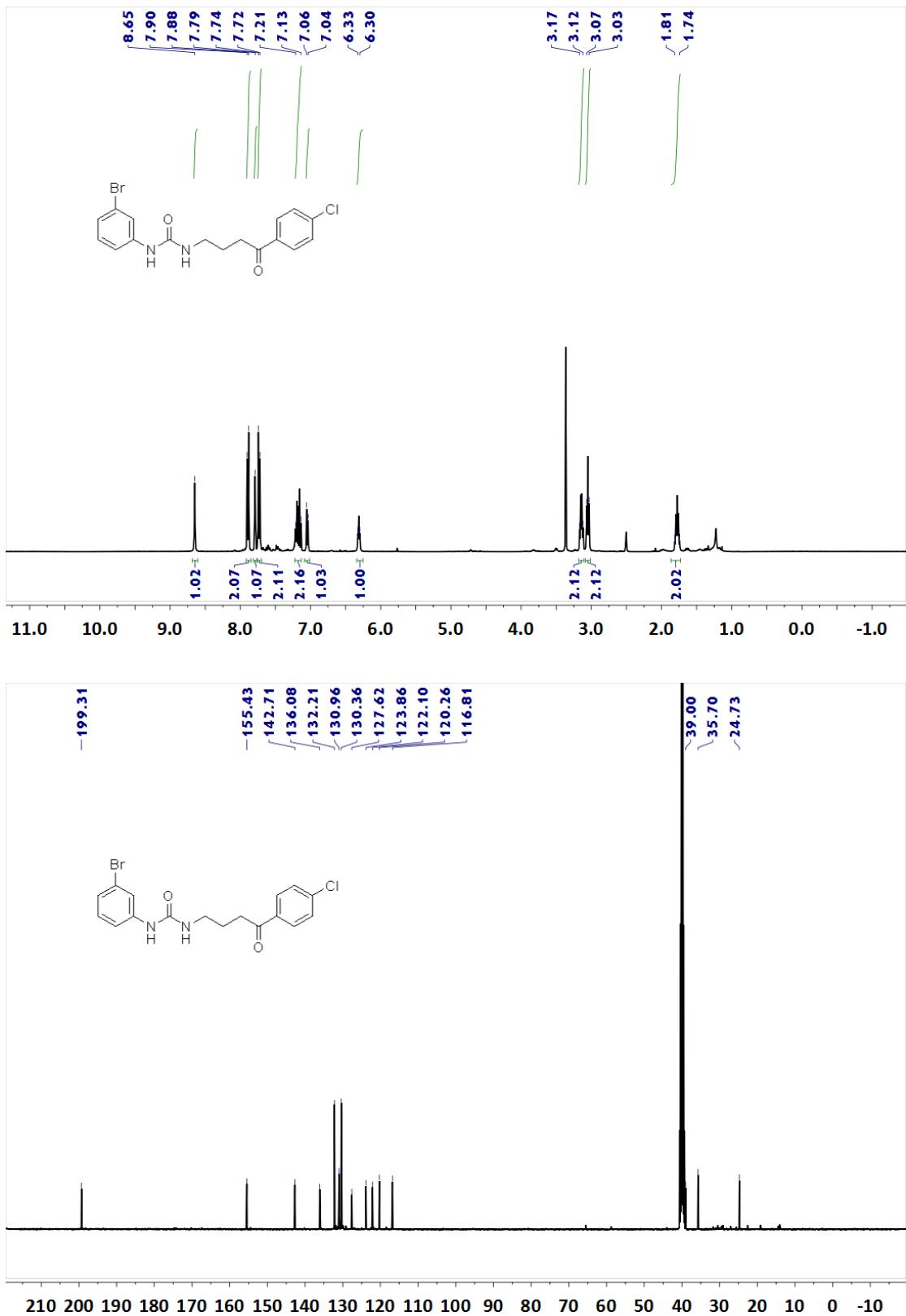


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2n**.

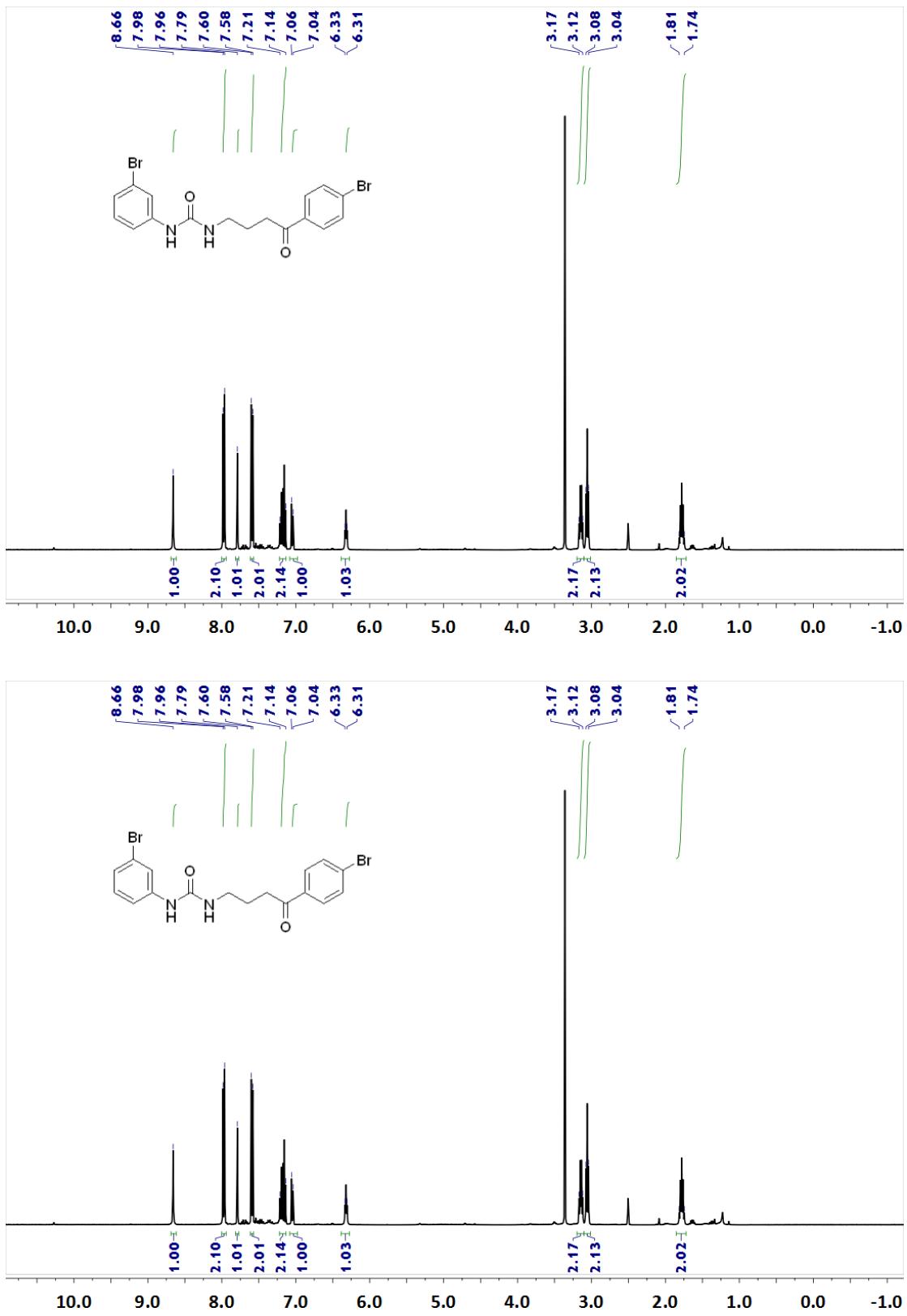




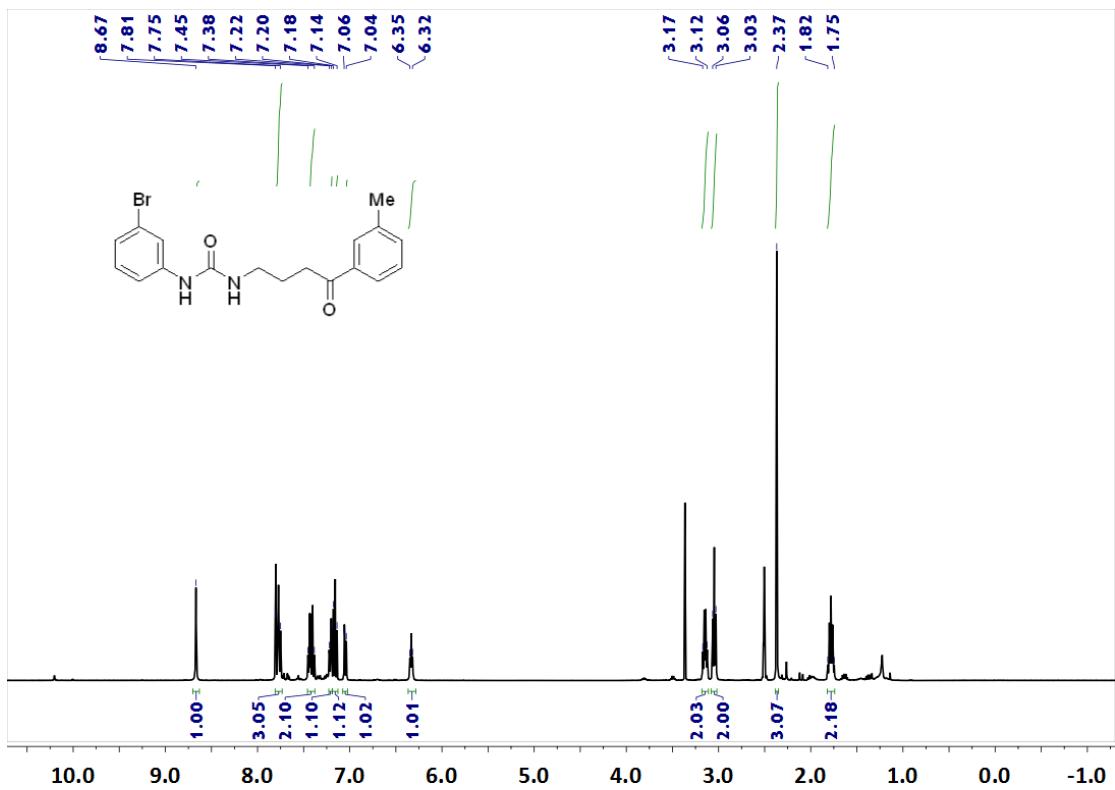
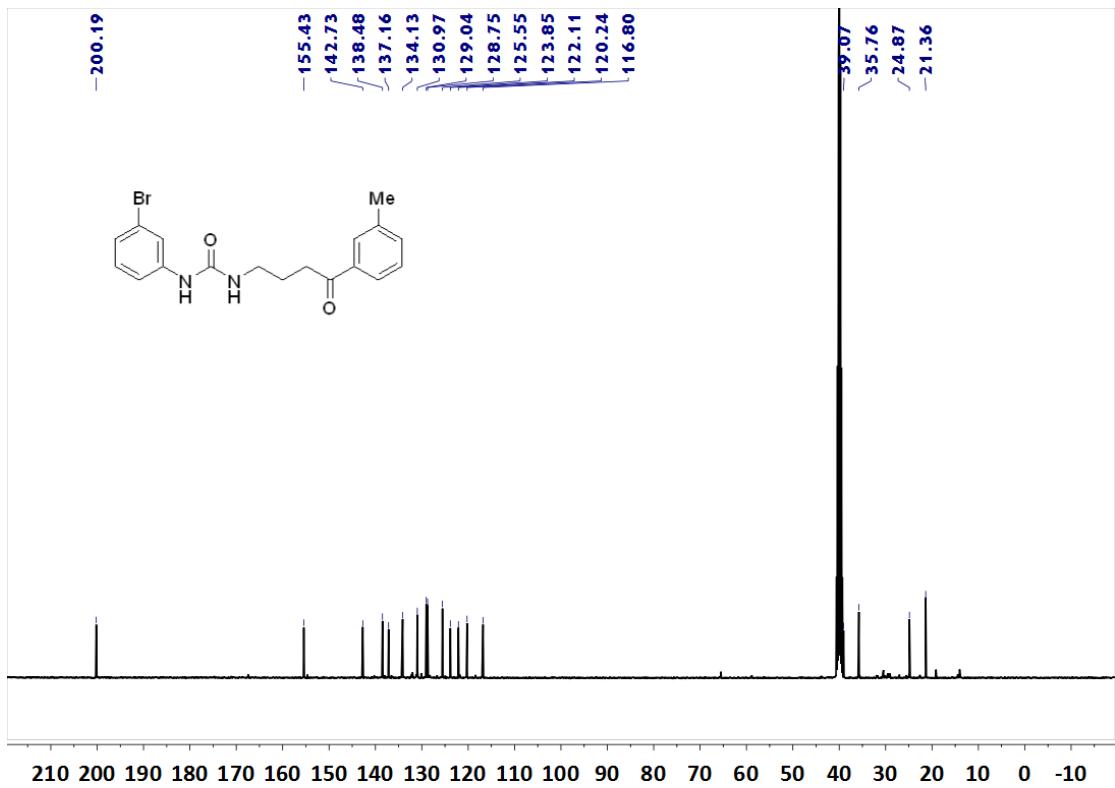
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2o**.



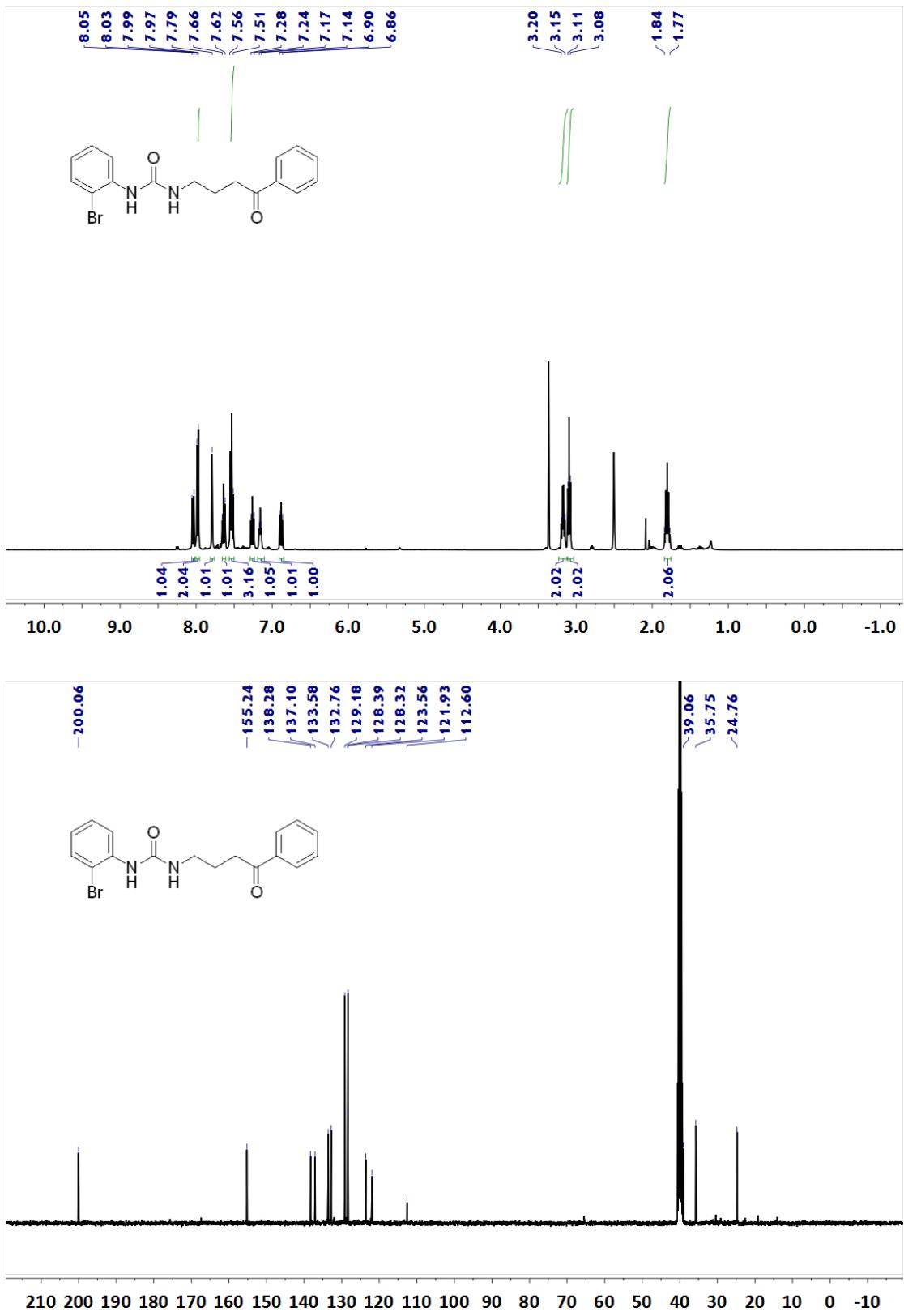
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2p**.



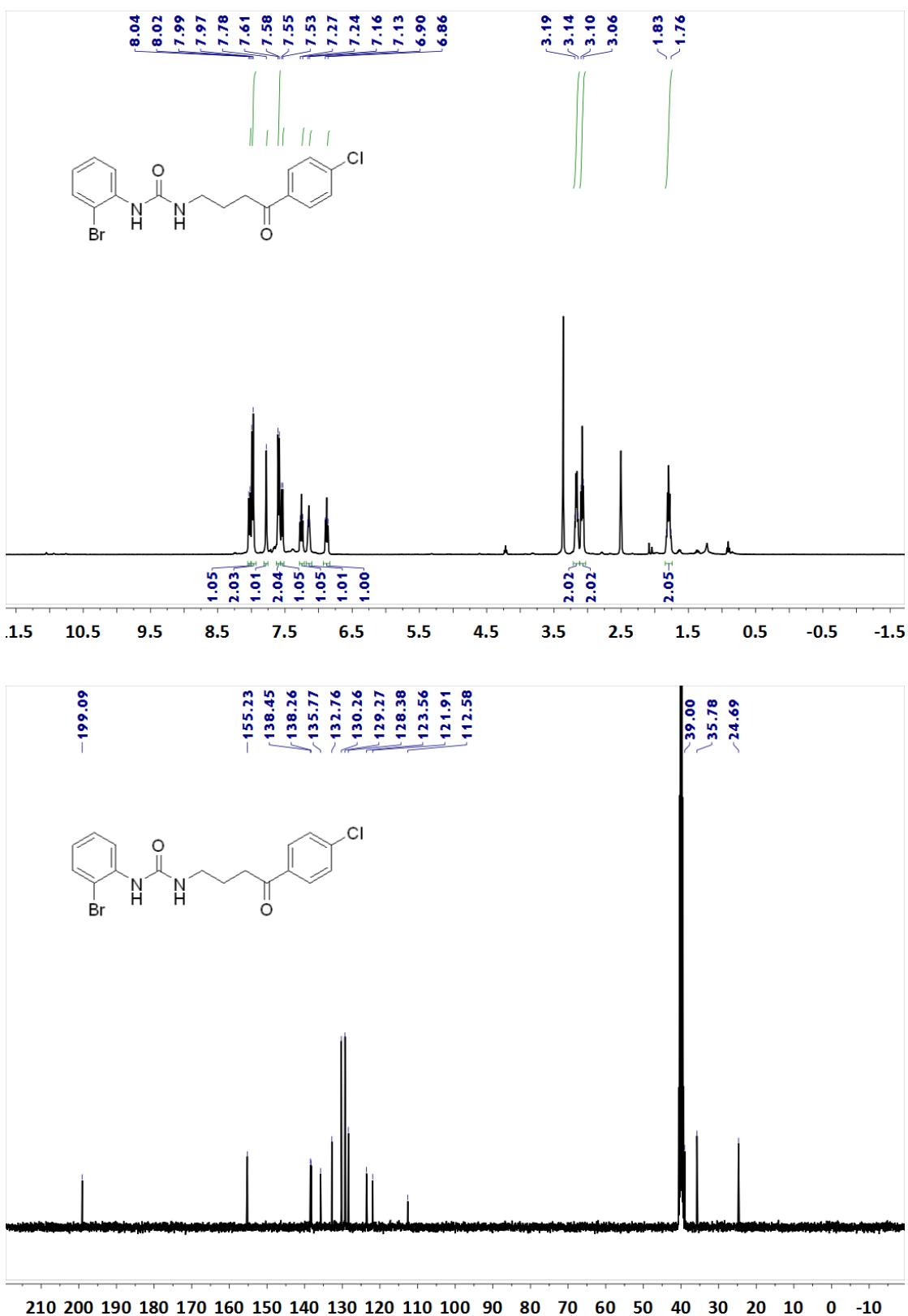
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2q**.



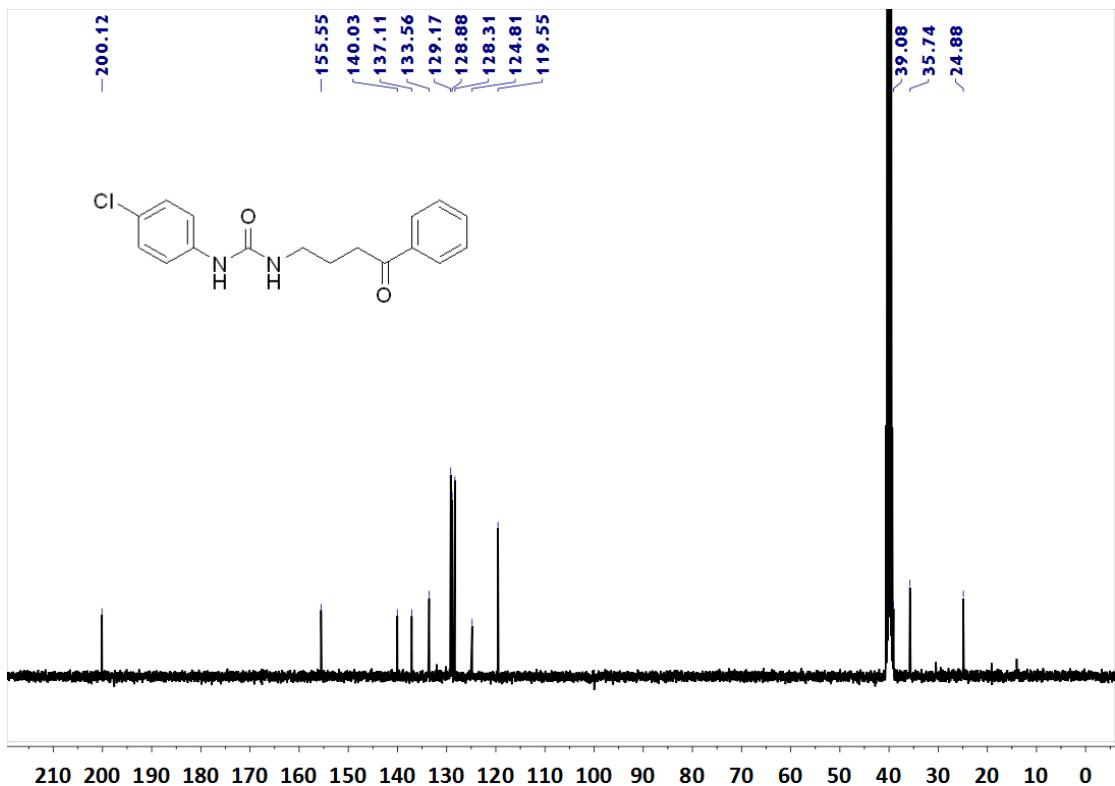
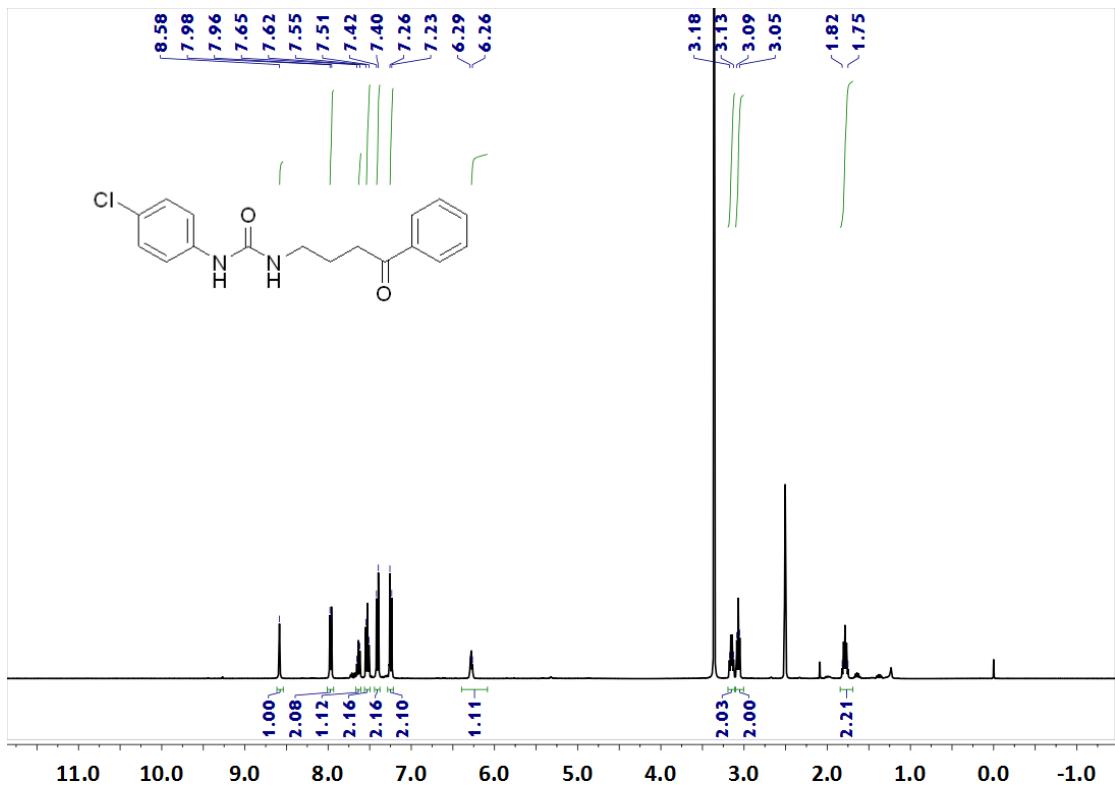
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 2r.



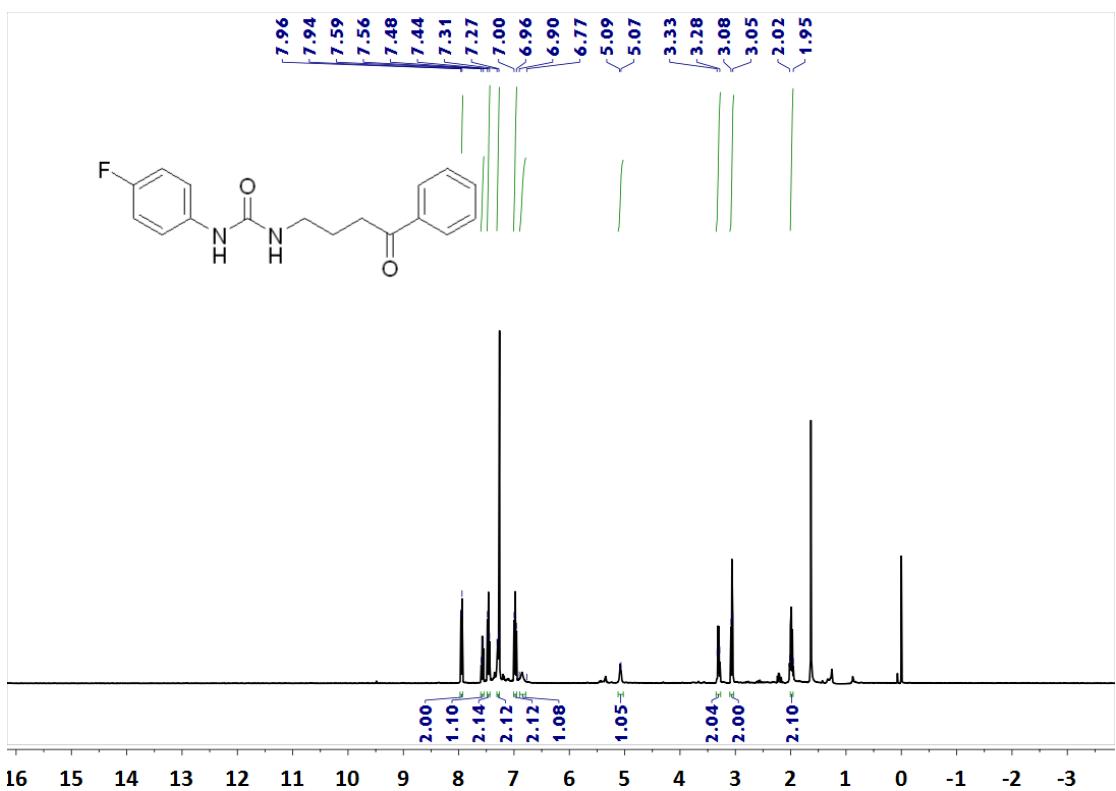
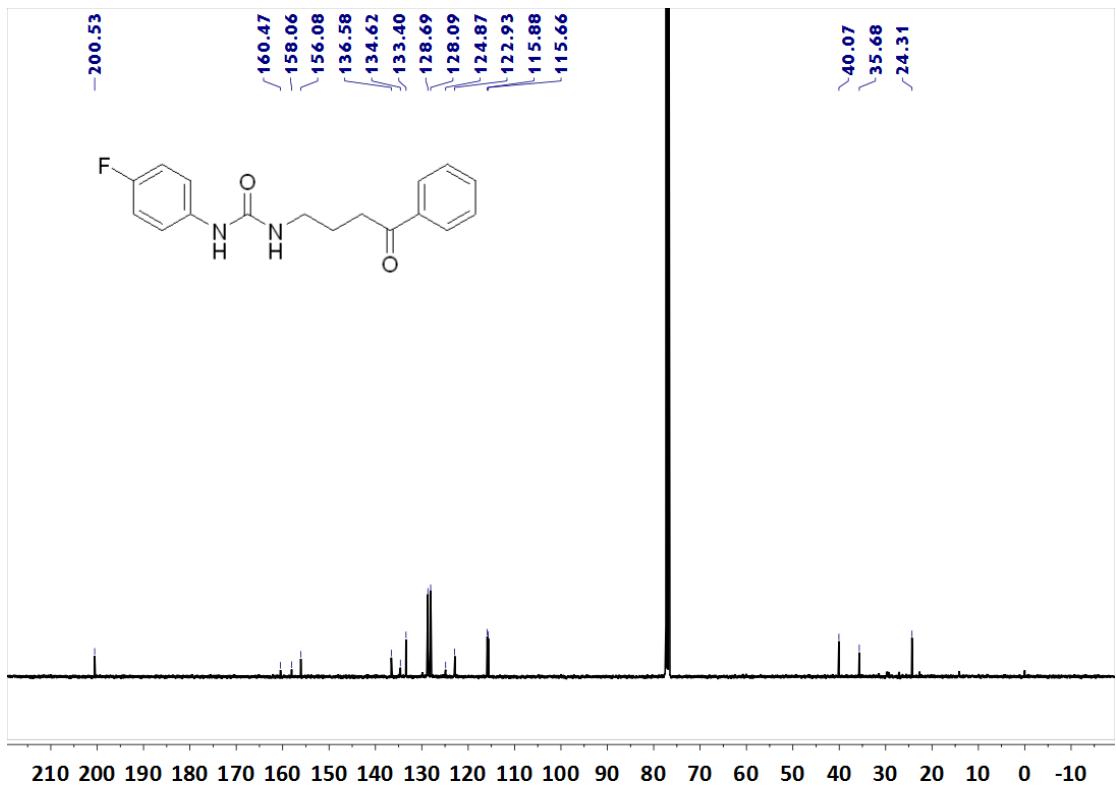
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2s**.

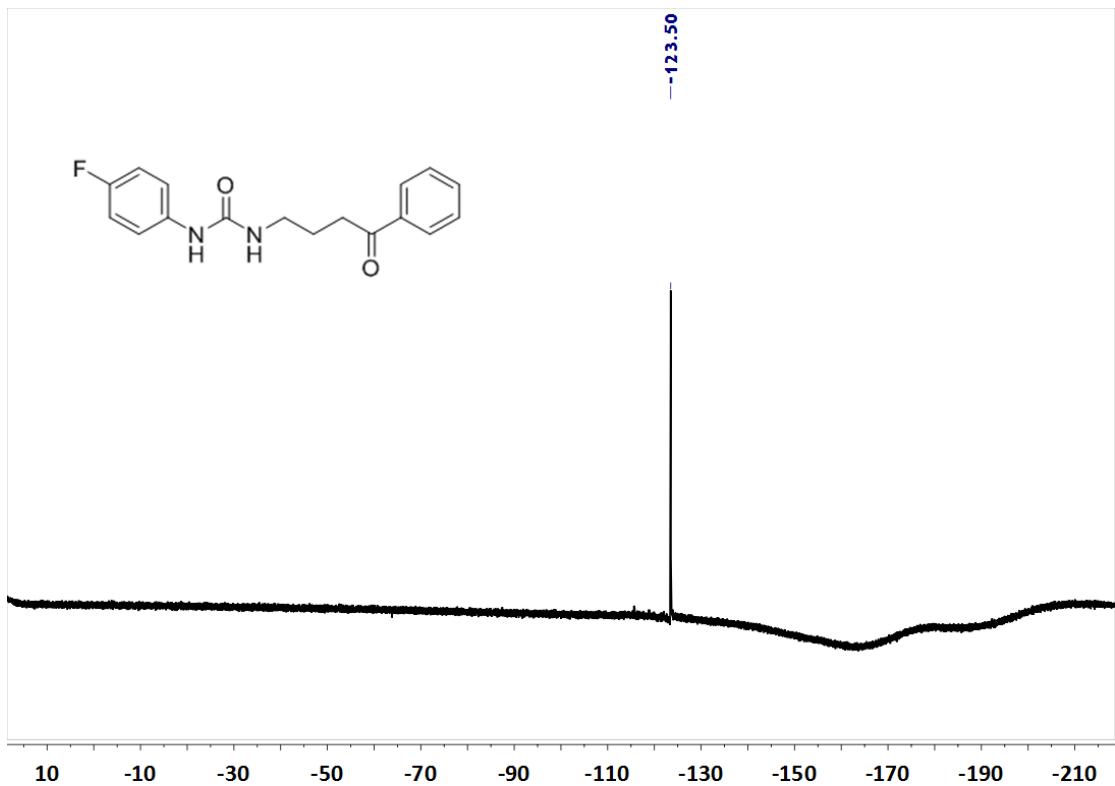


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2t**.

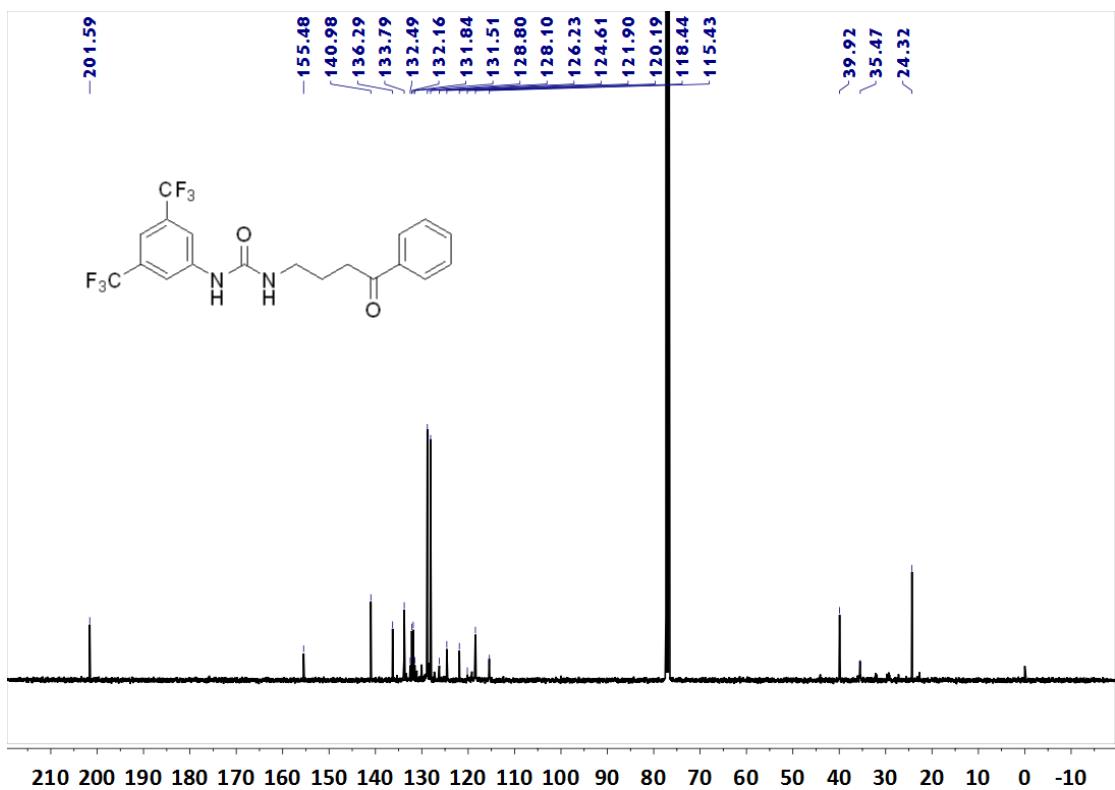


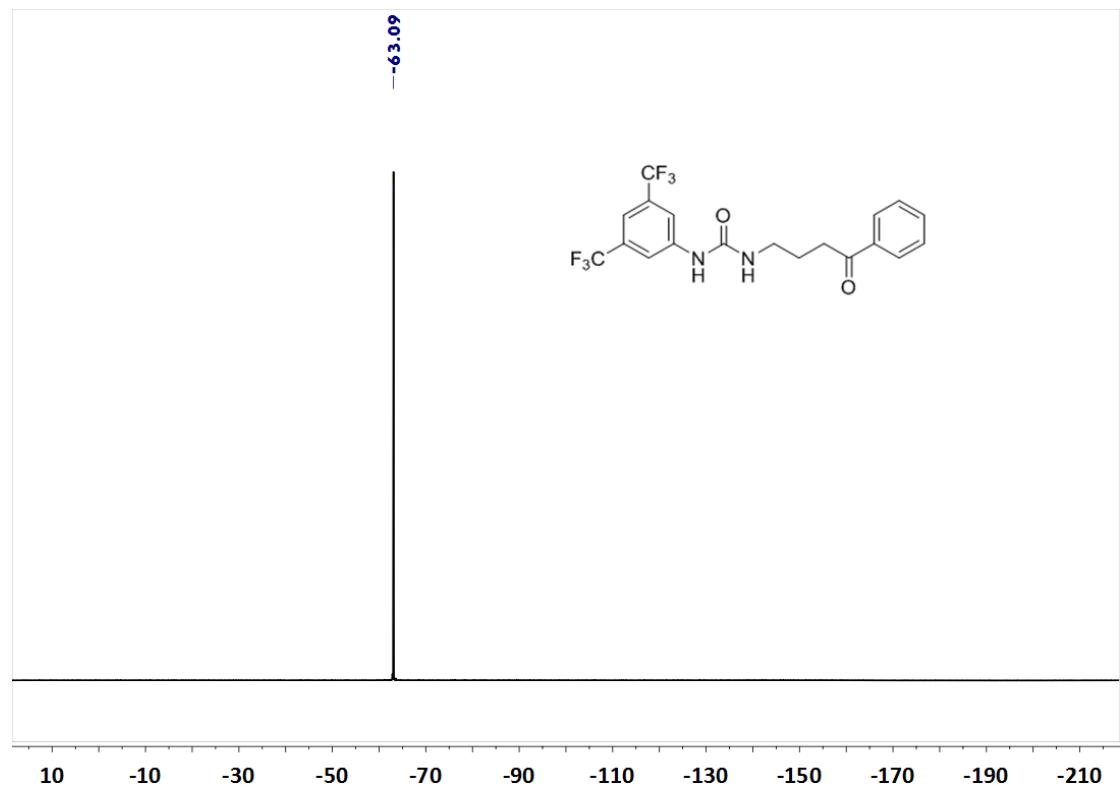
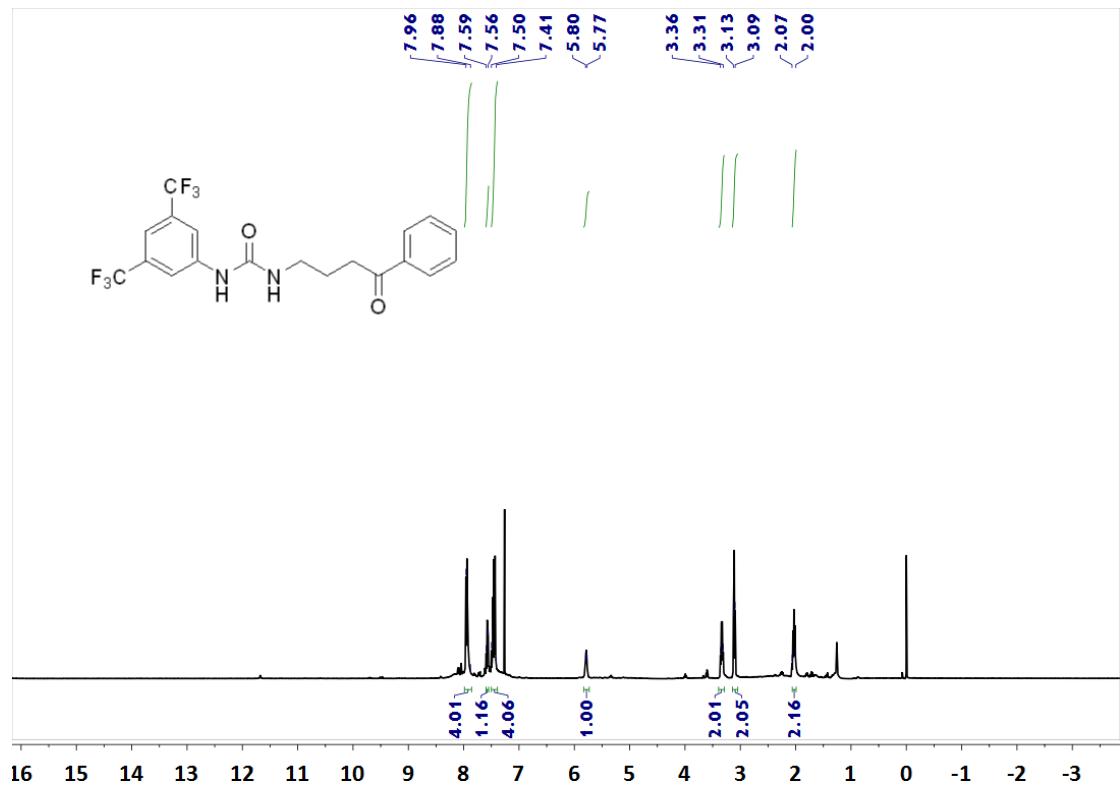
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2u**.



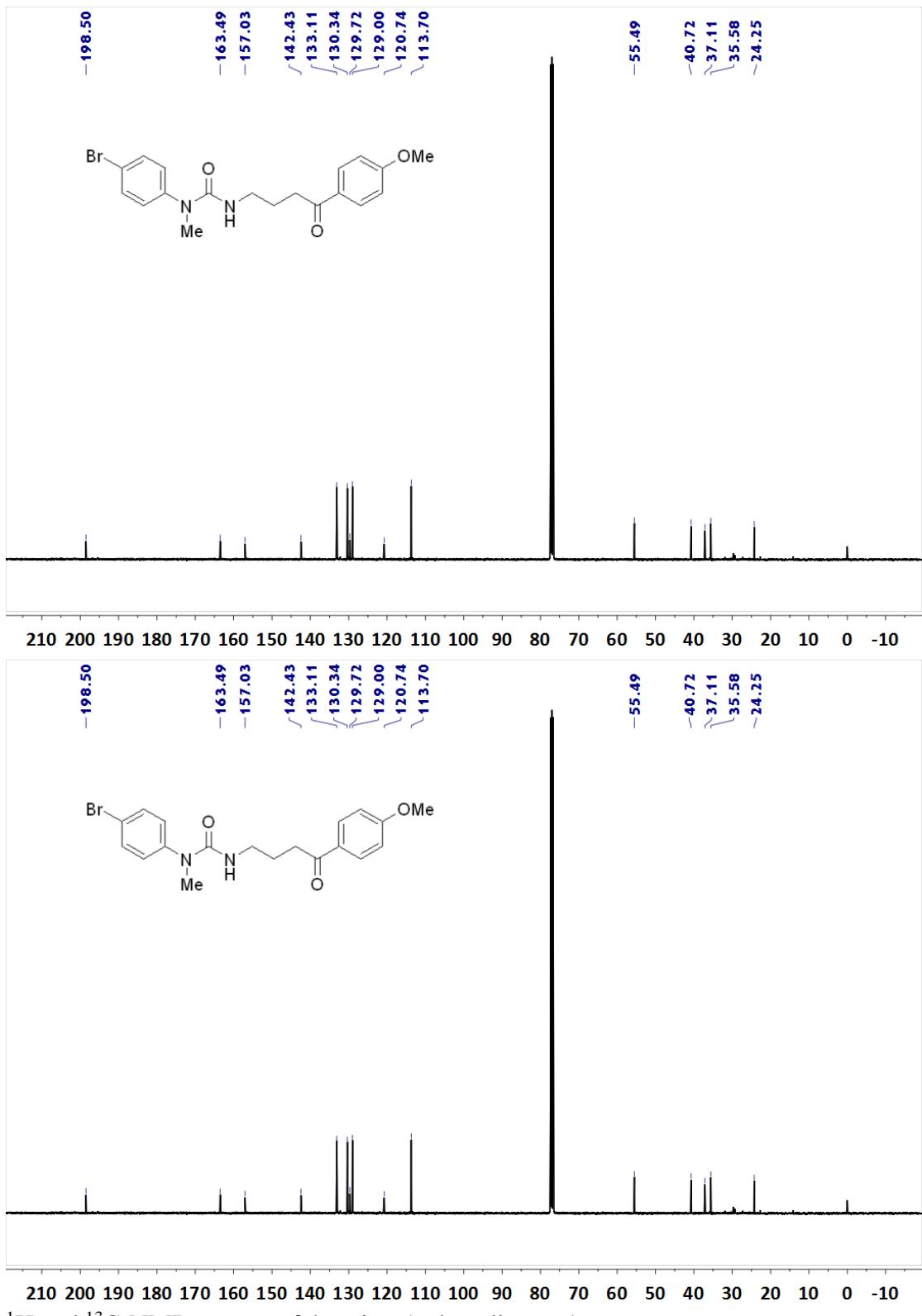


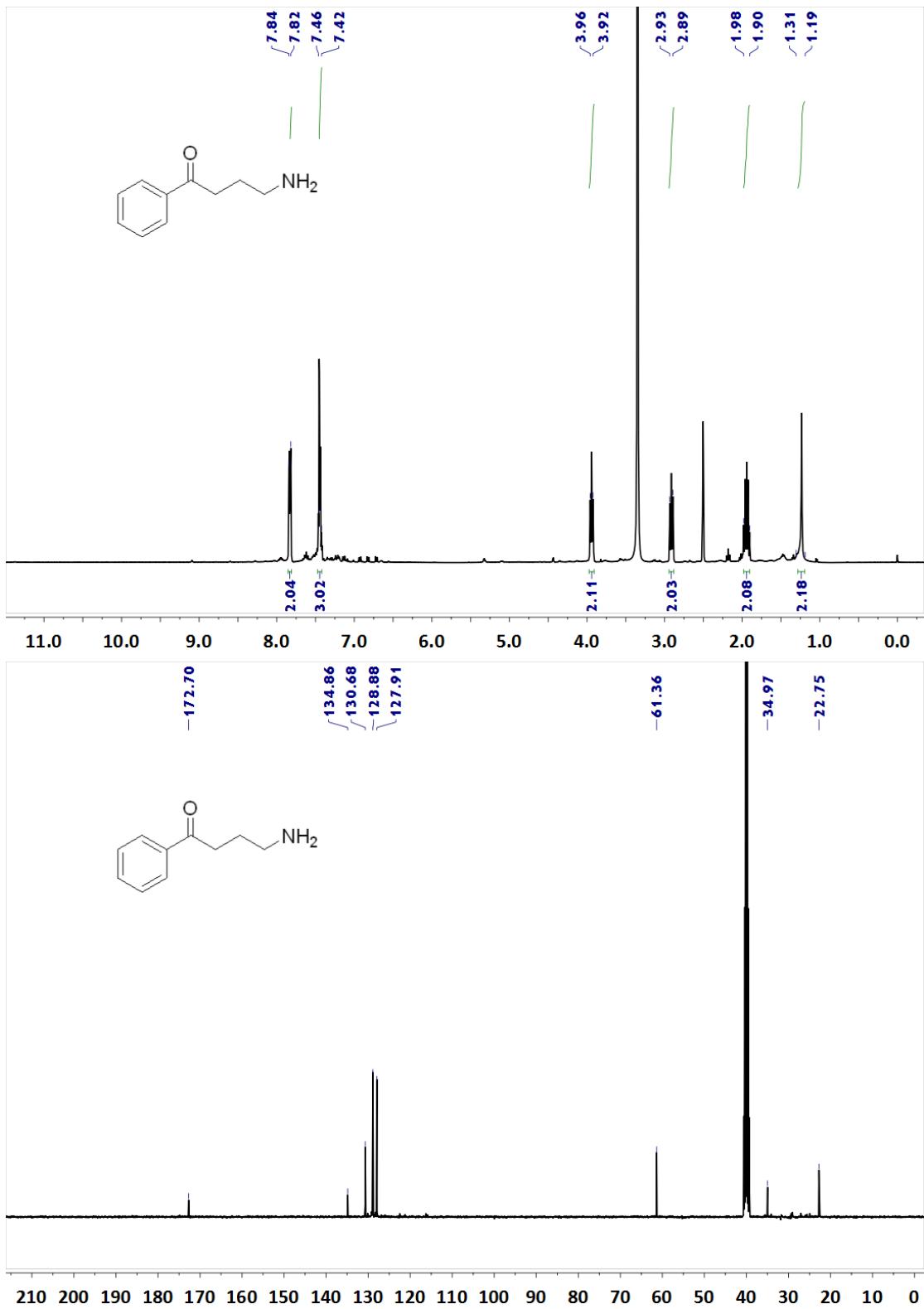
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2v**.

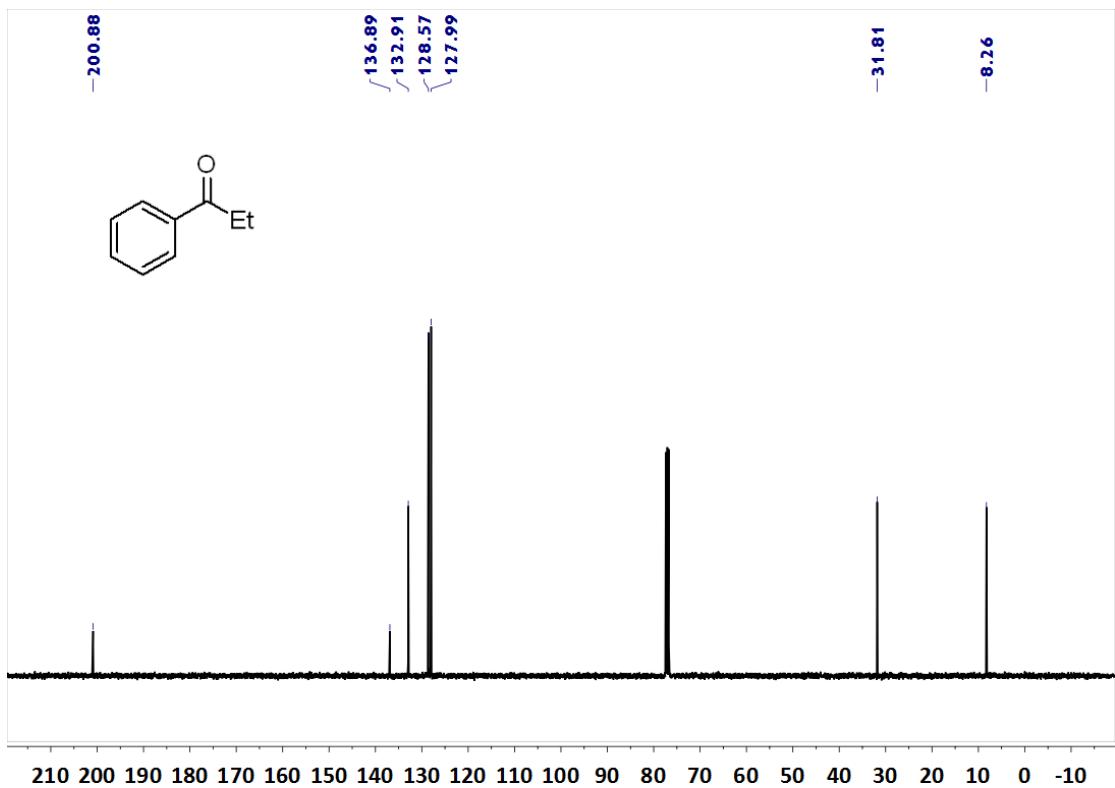
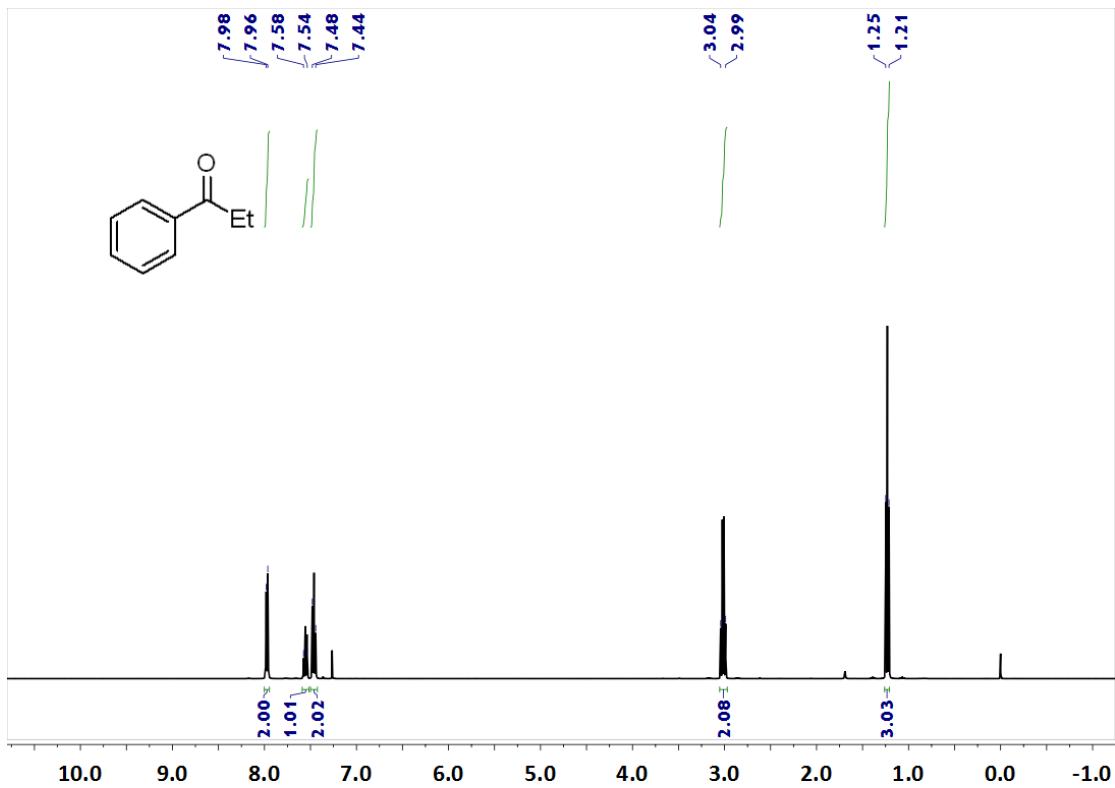




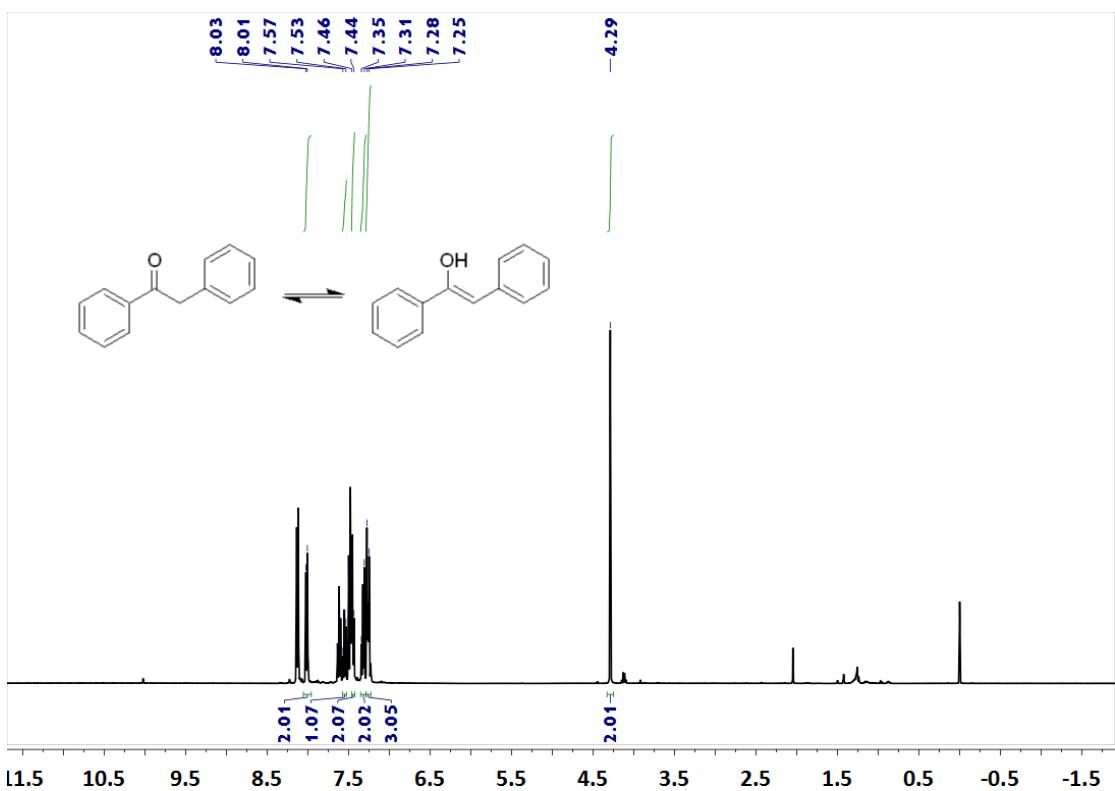
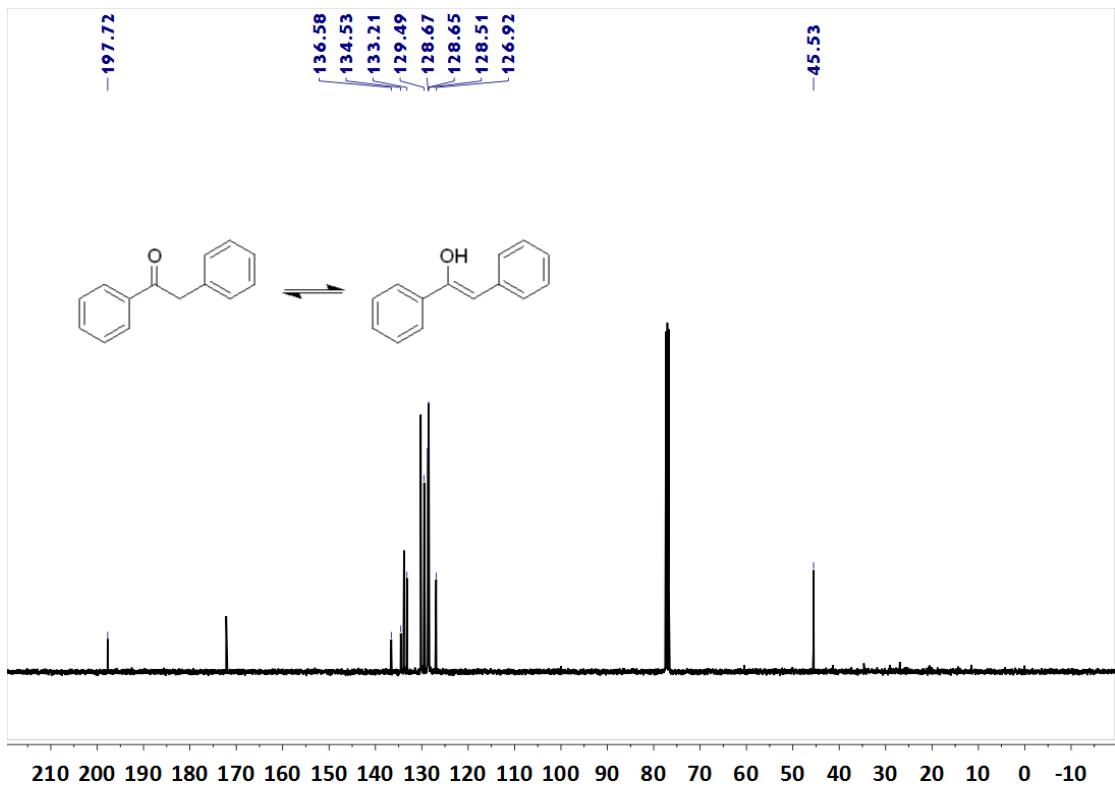
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **2cc**.



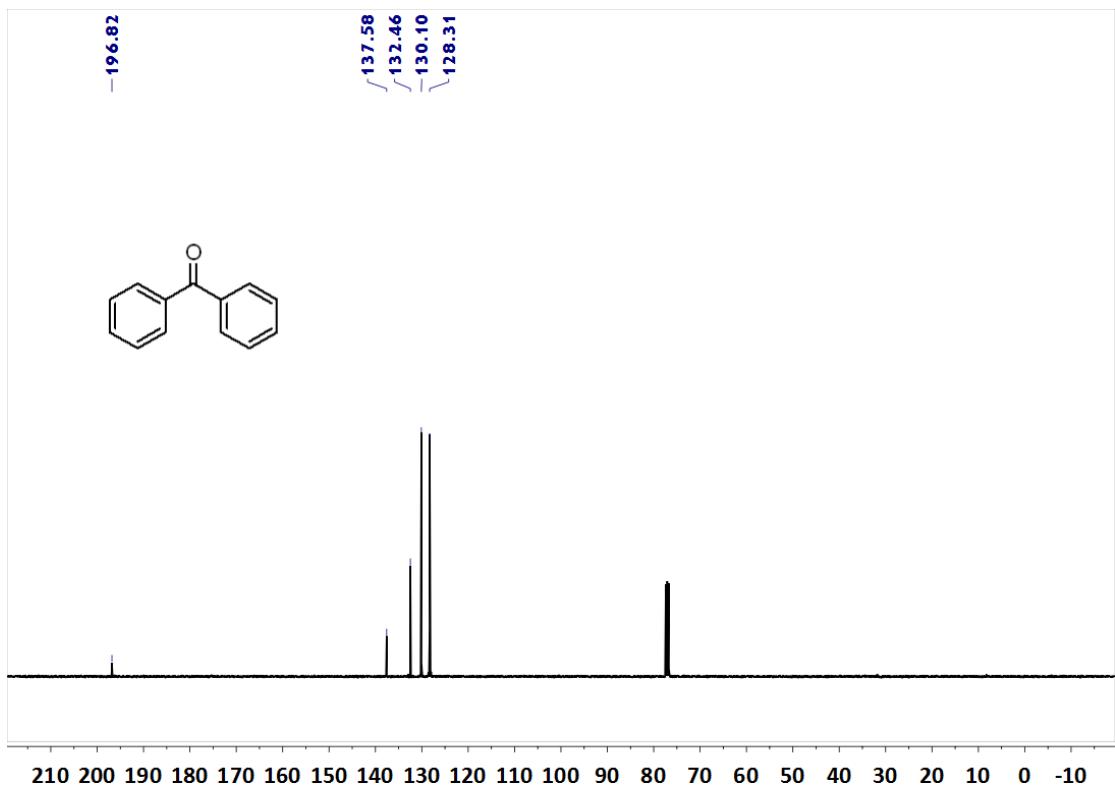
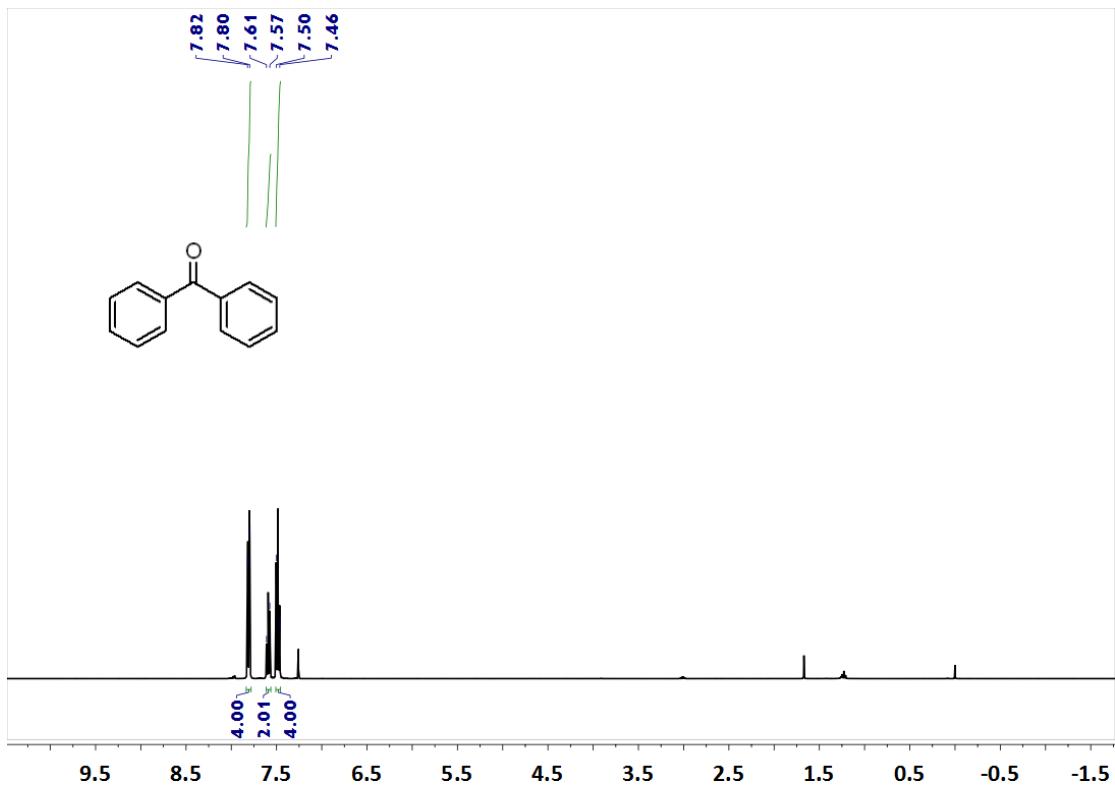




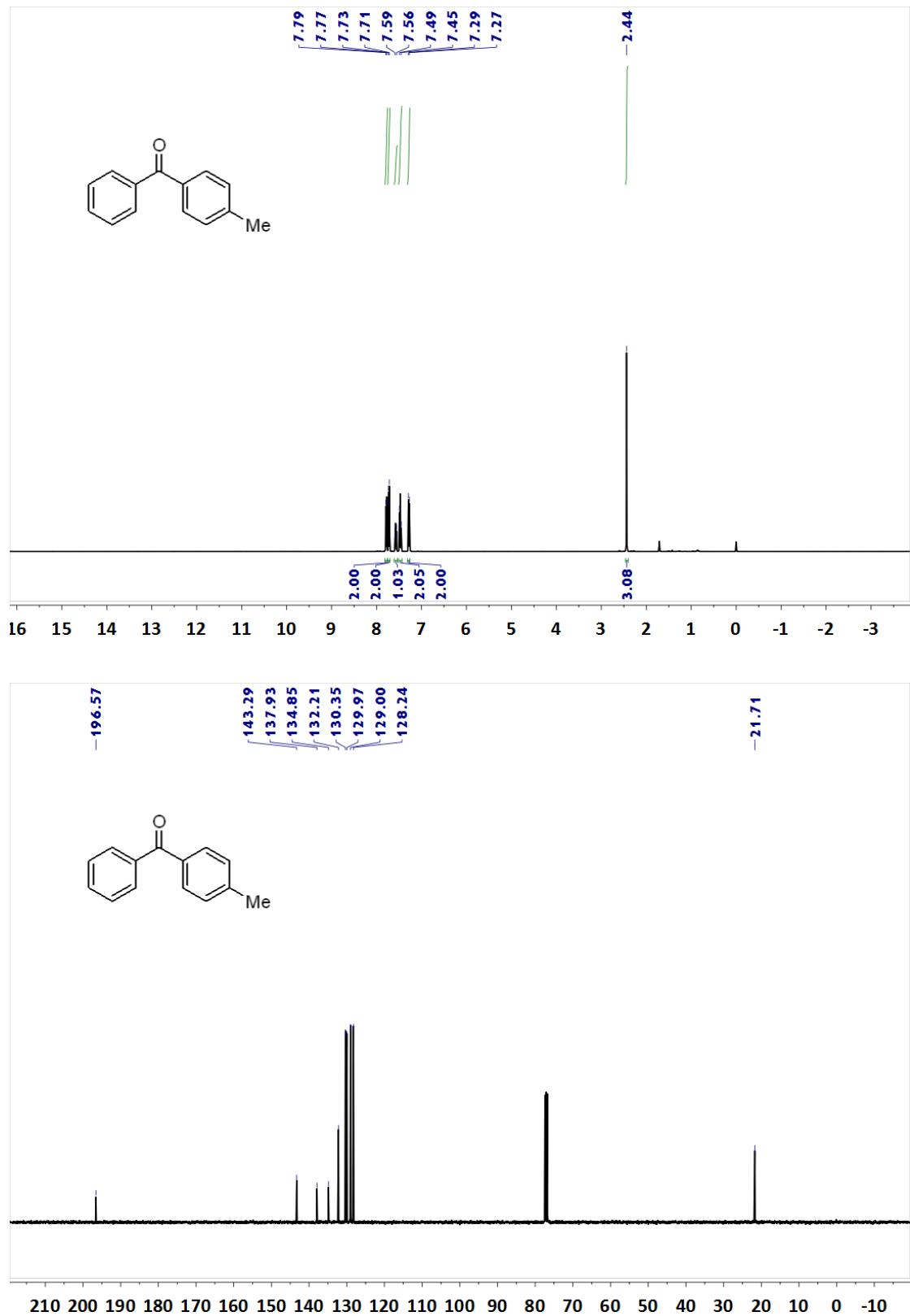
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **4b**.



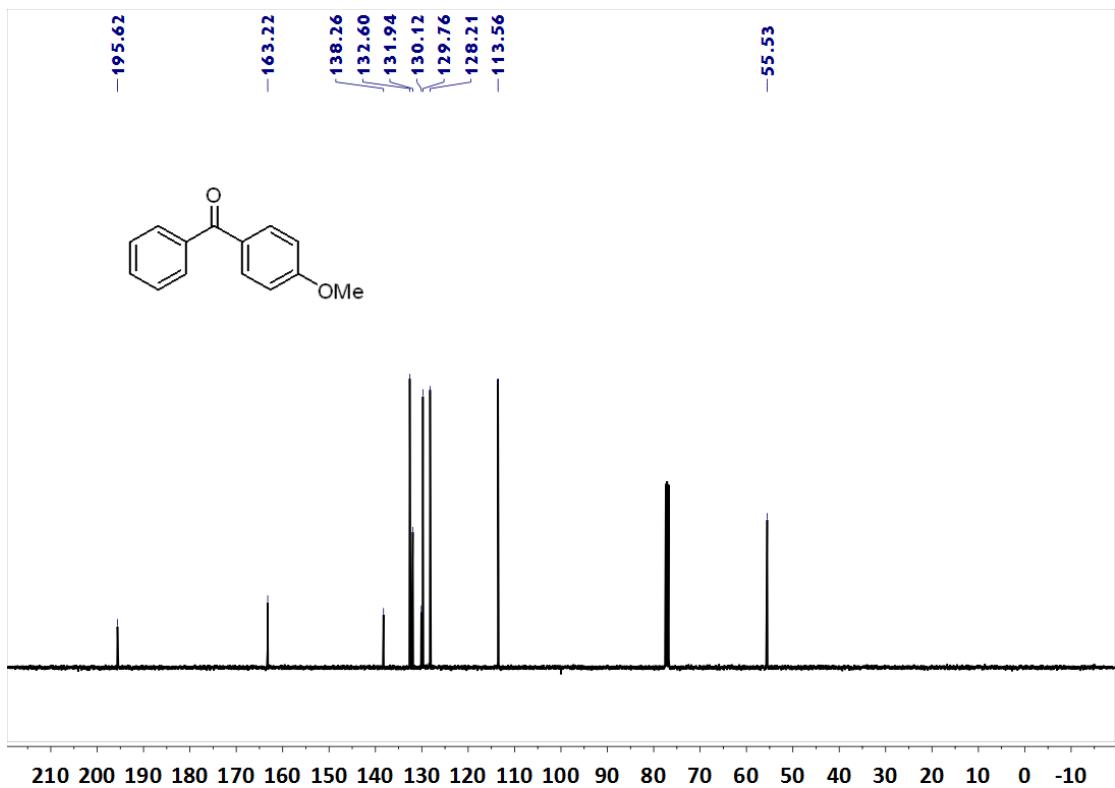
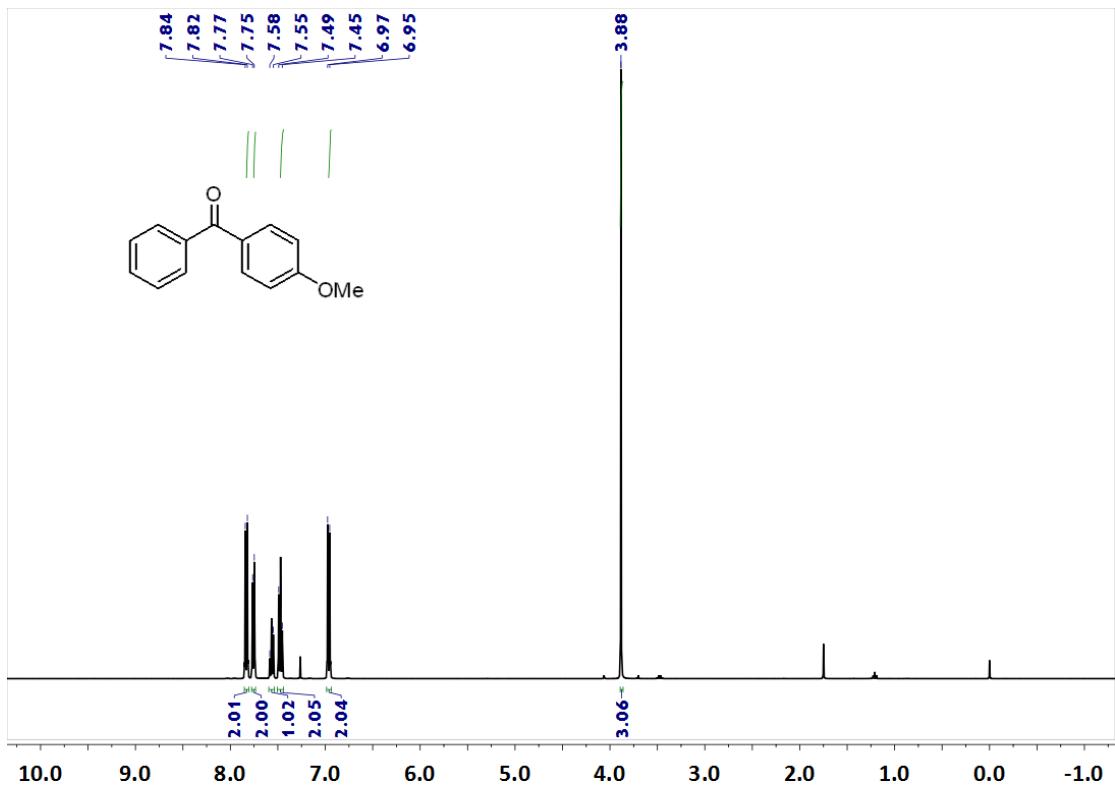
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 4c.



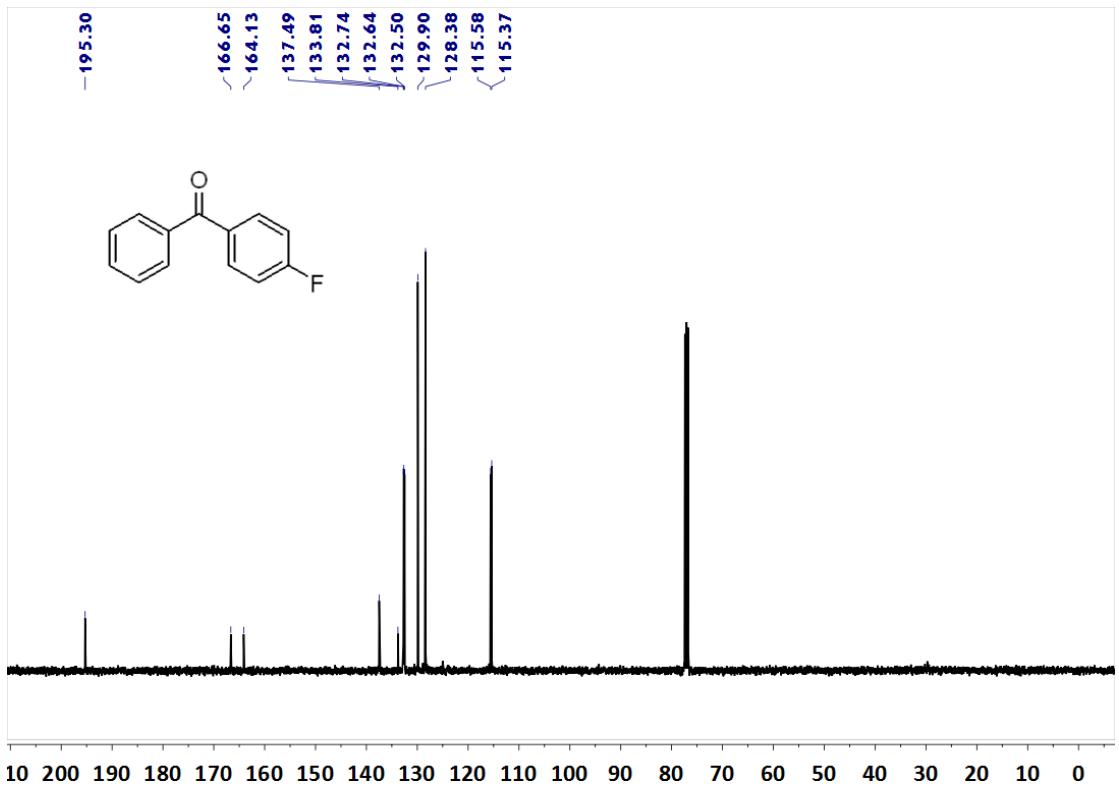
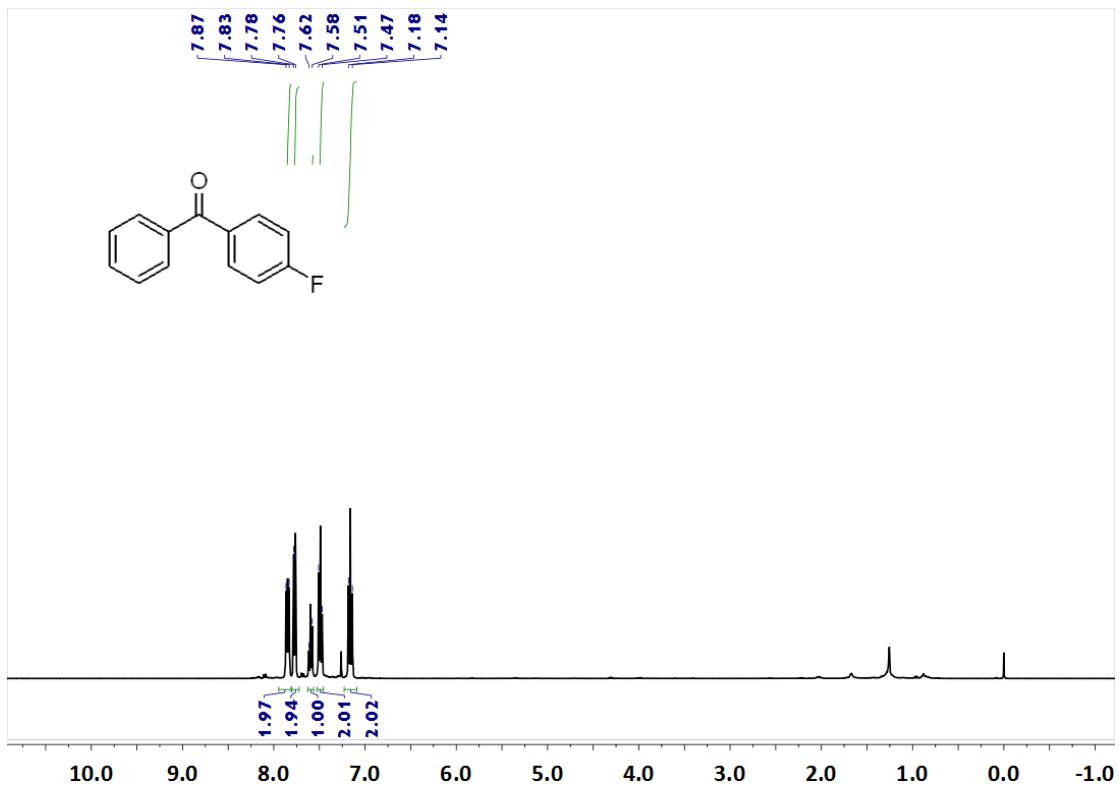
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4d**.

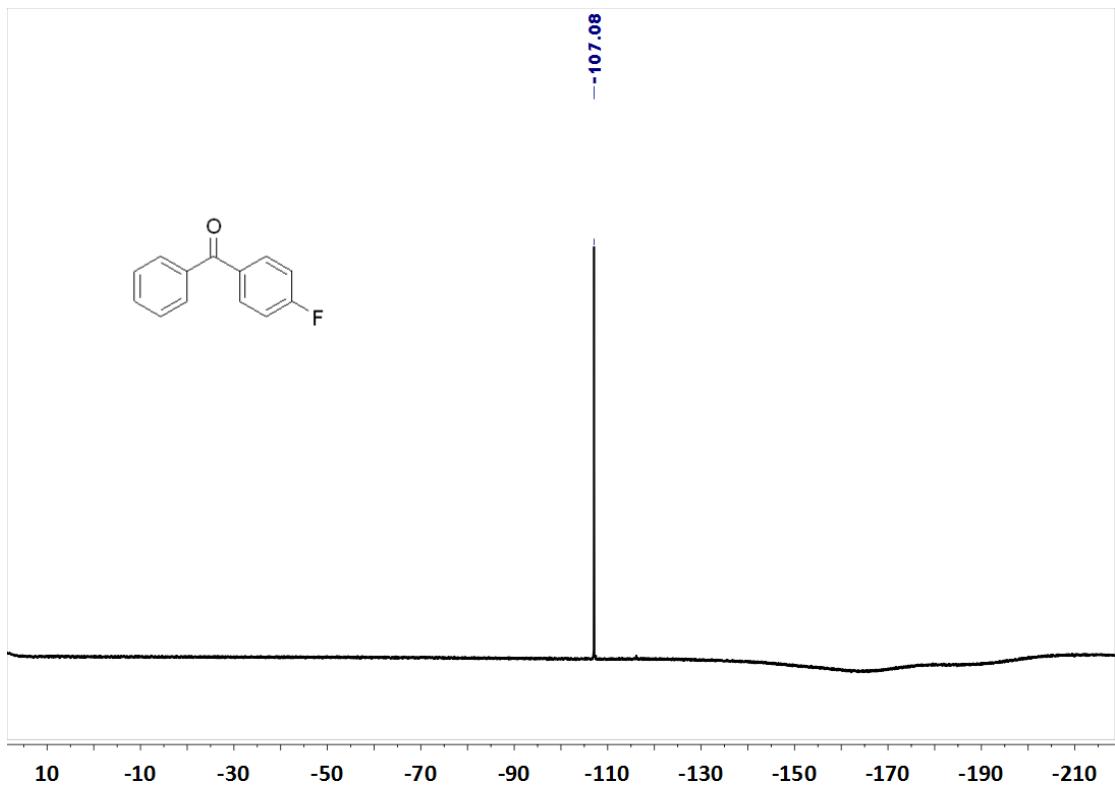


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4e**.

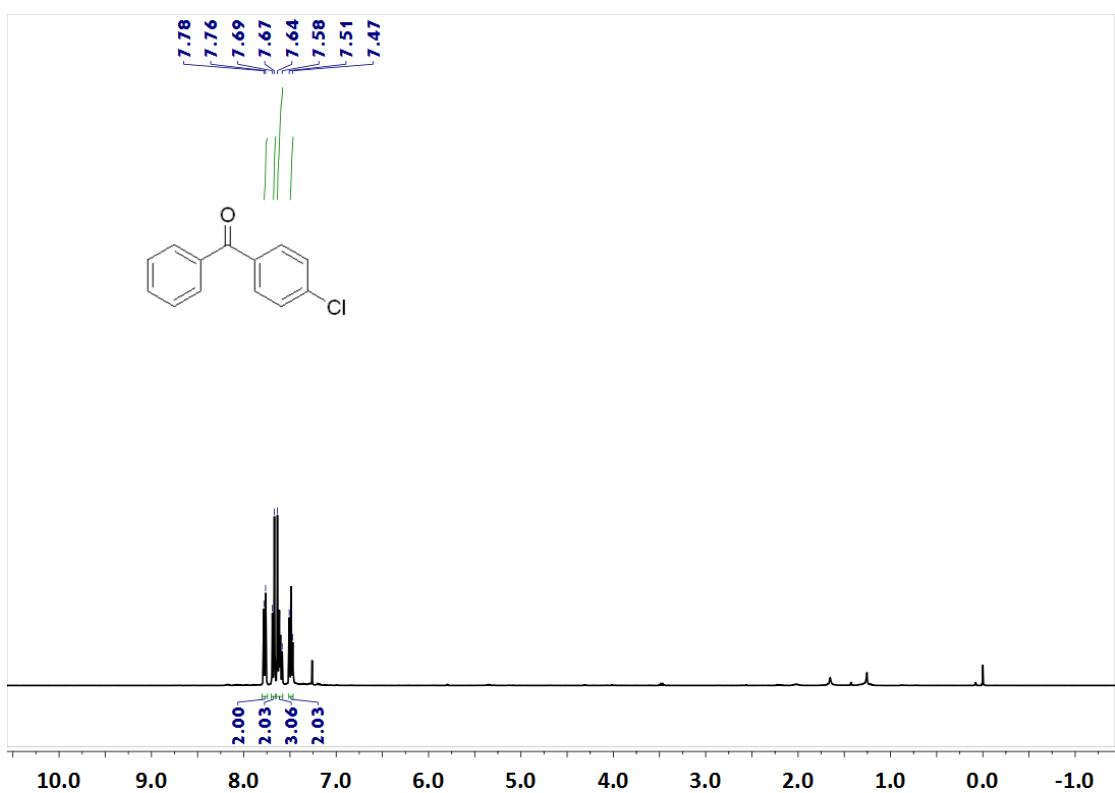


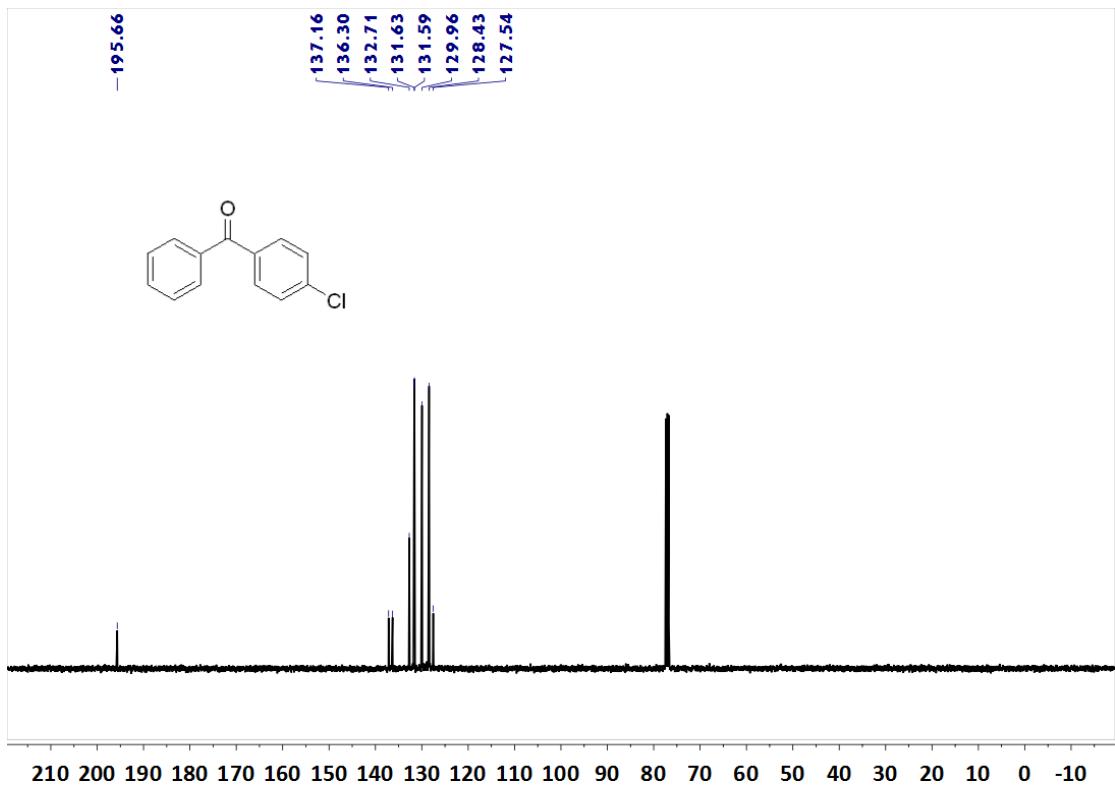
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4f**.



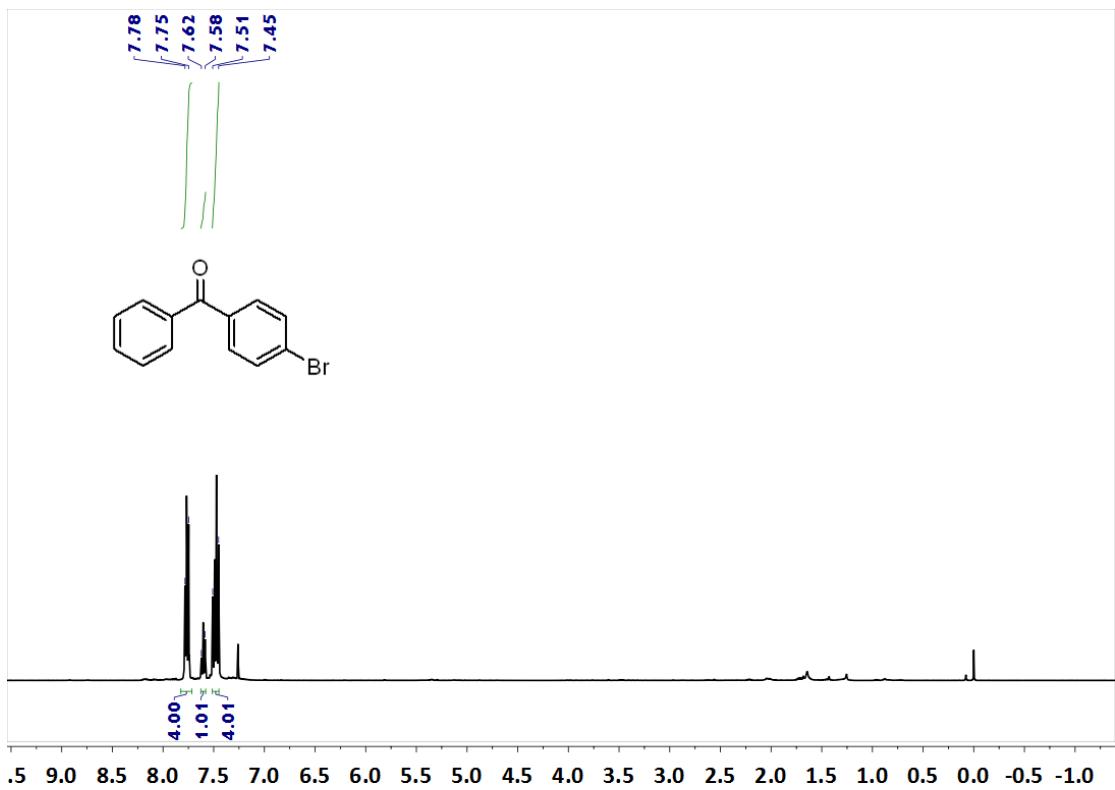


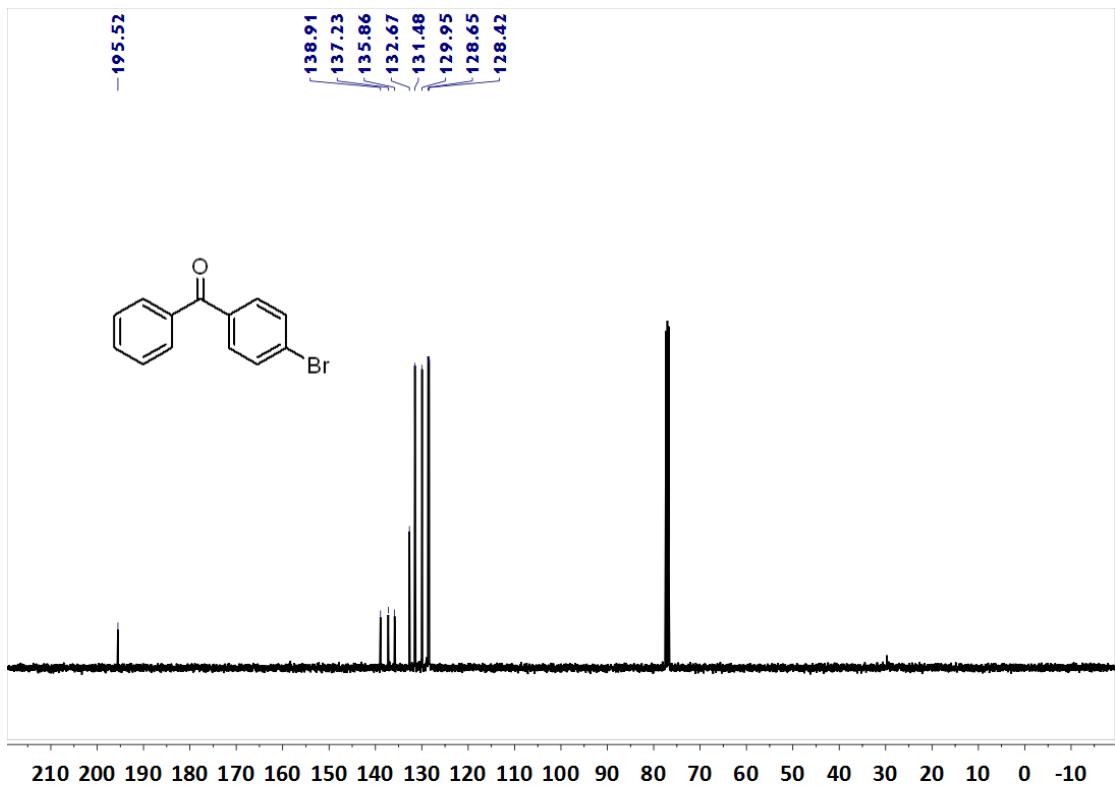
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 4g.



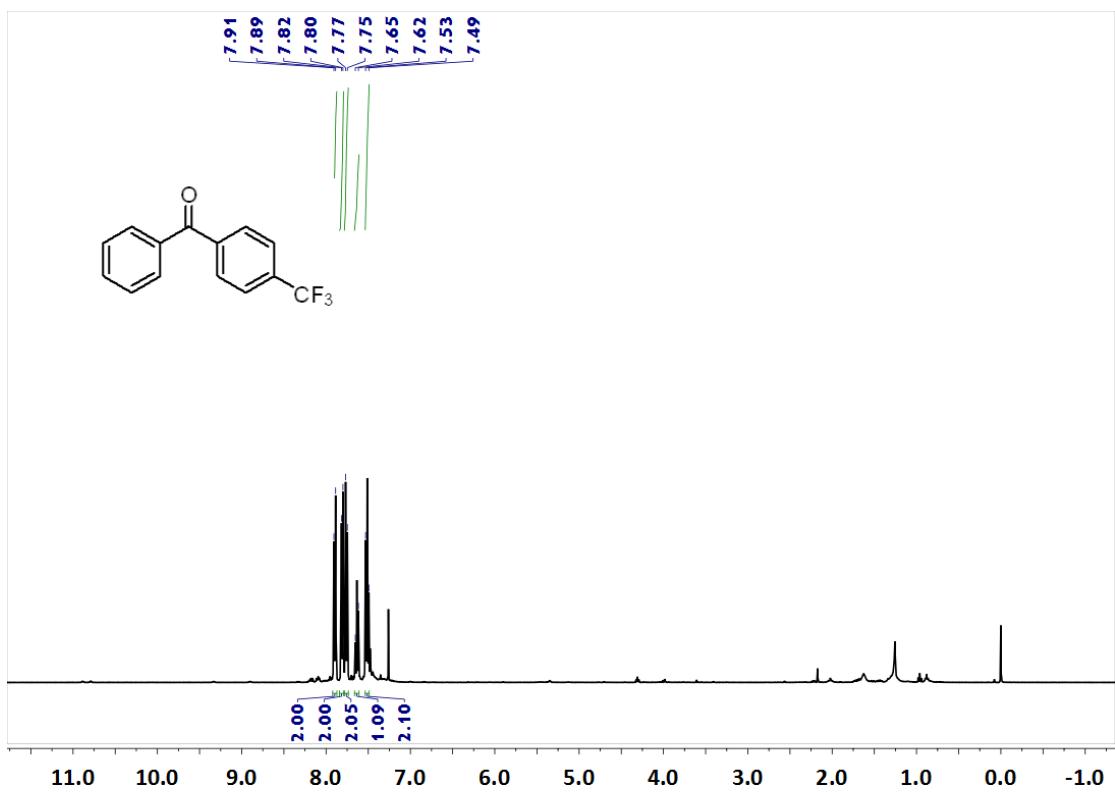


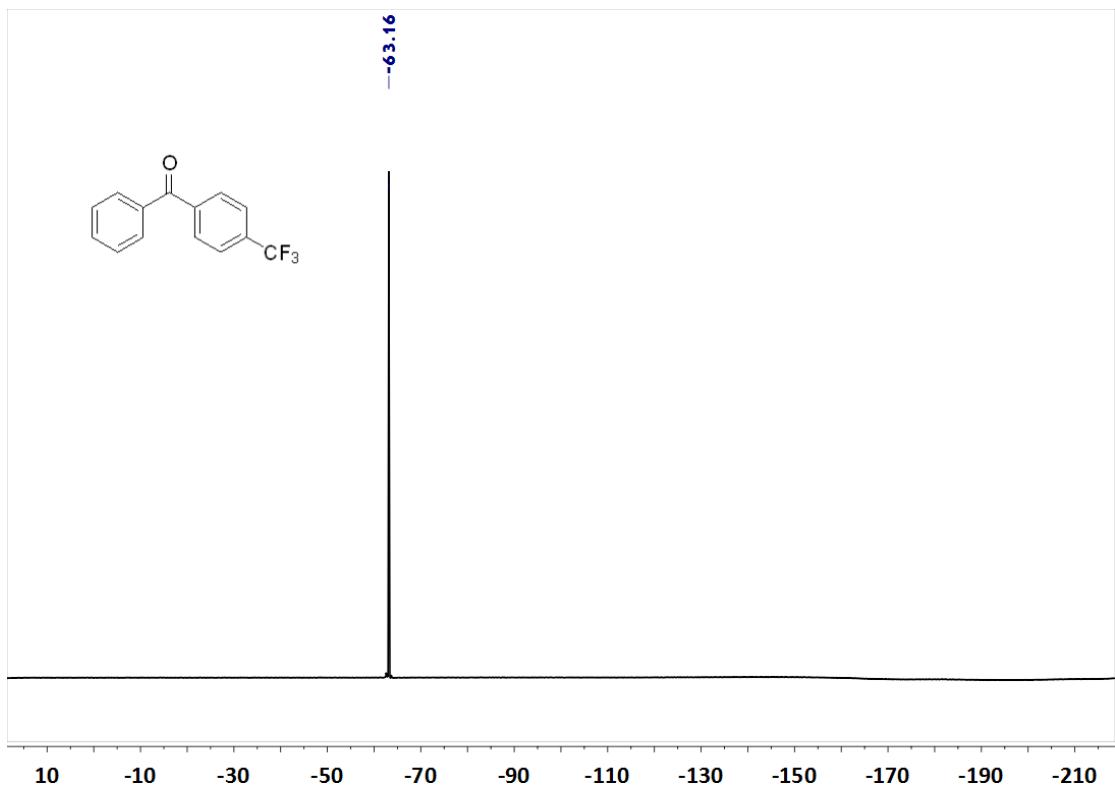
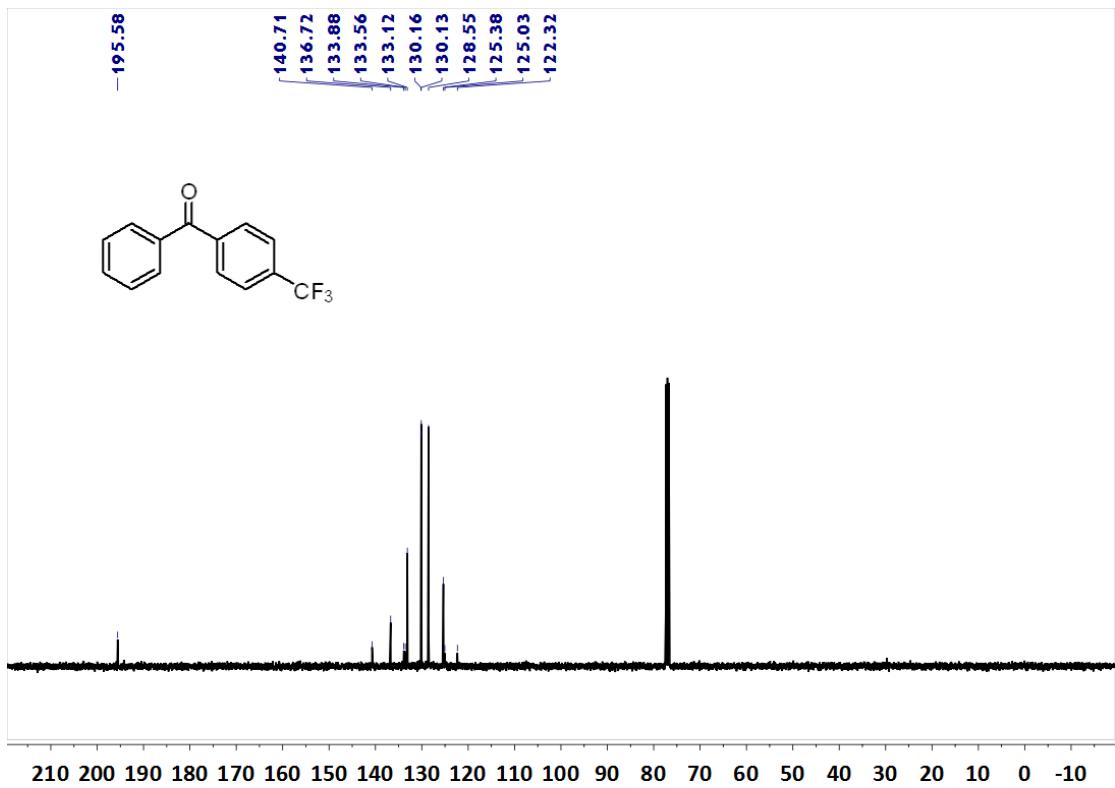
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4h**.



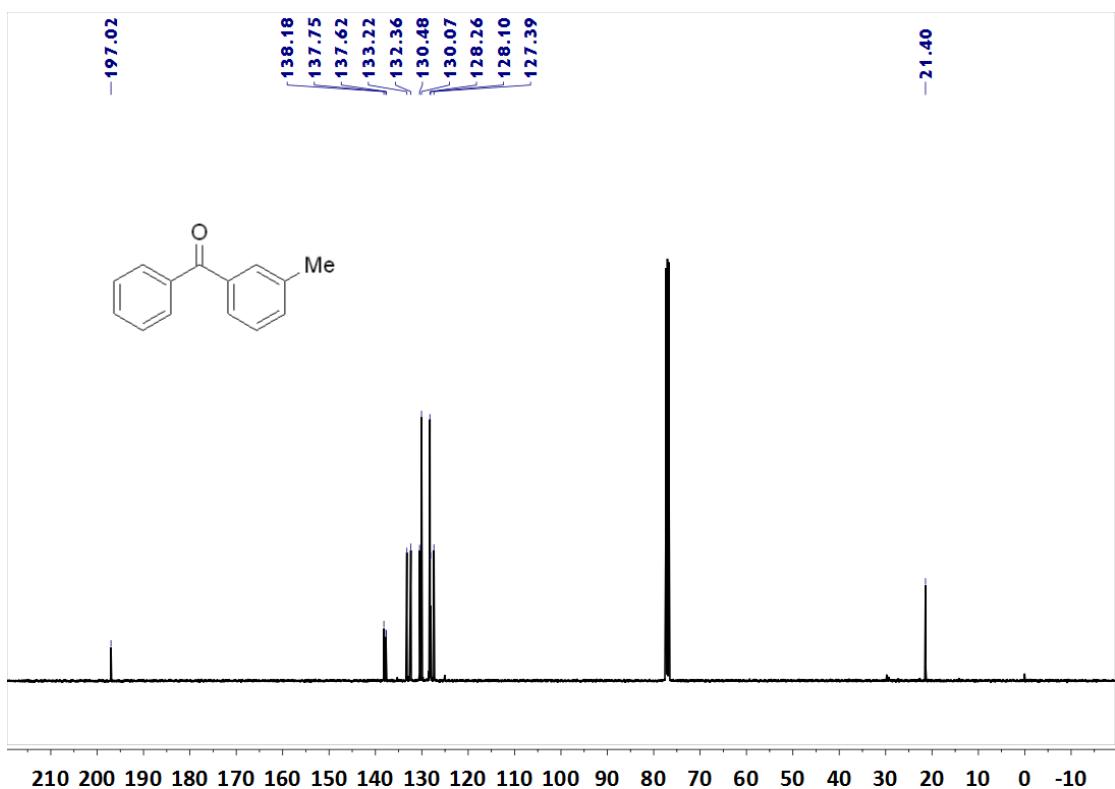
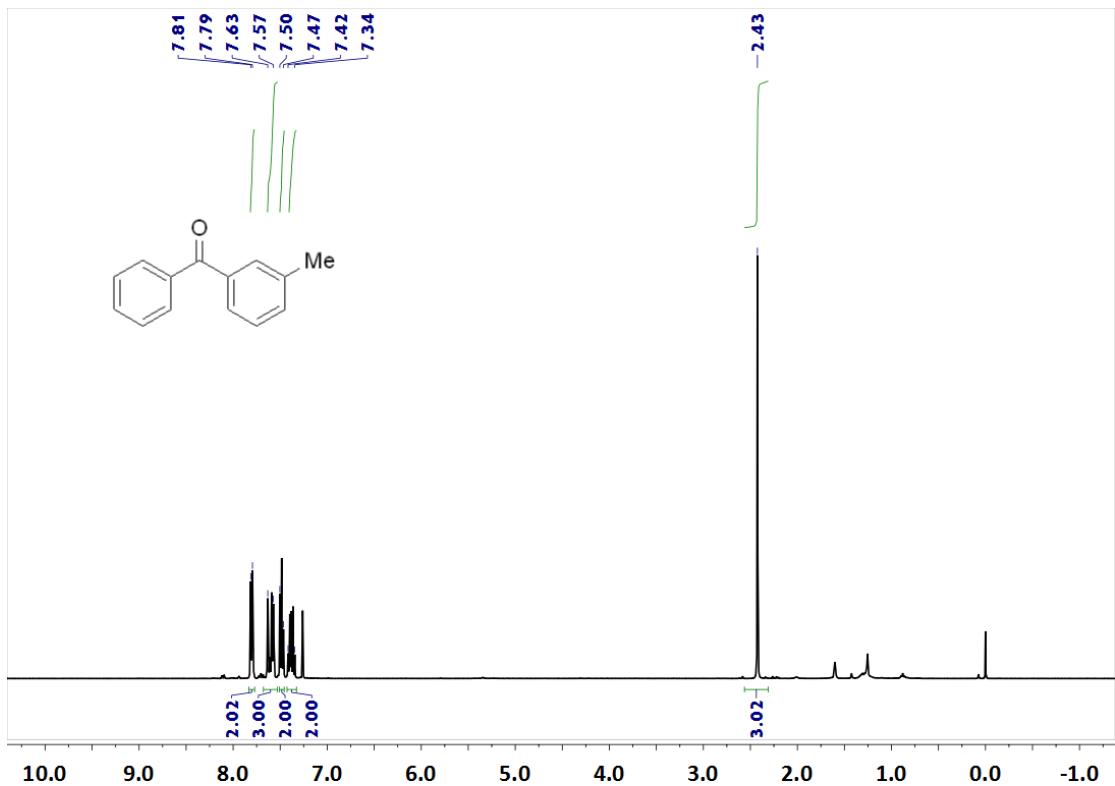


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4i**.

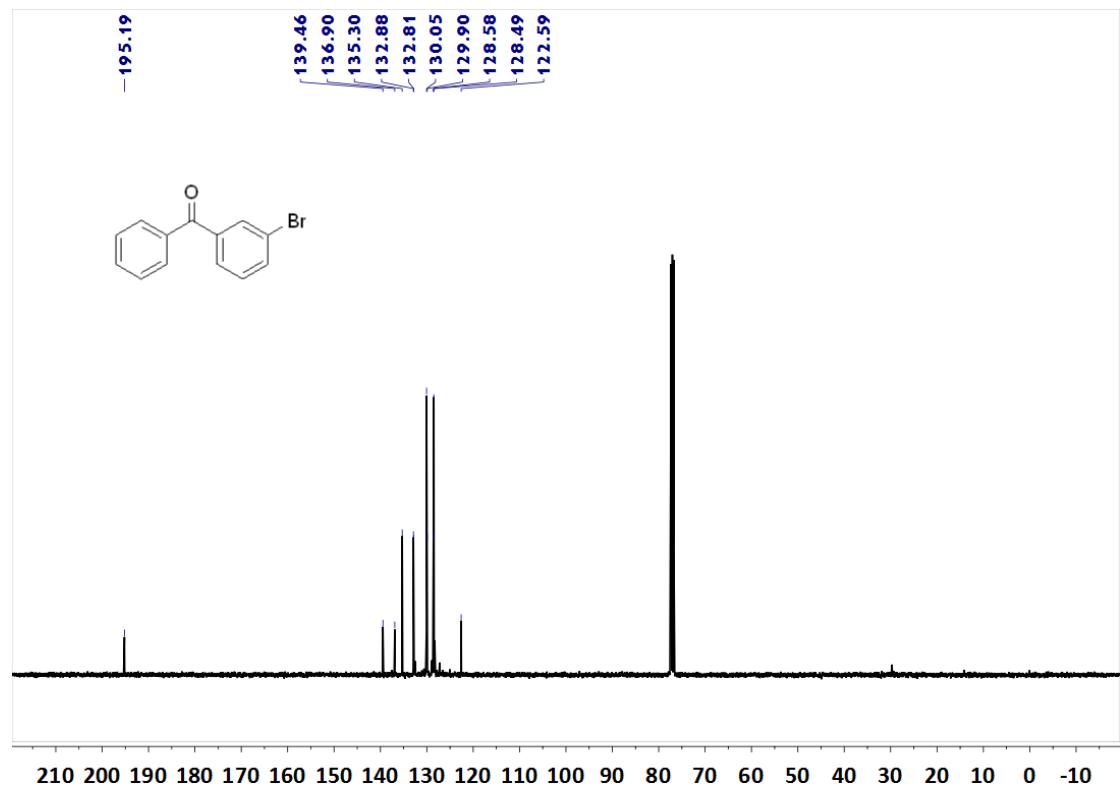
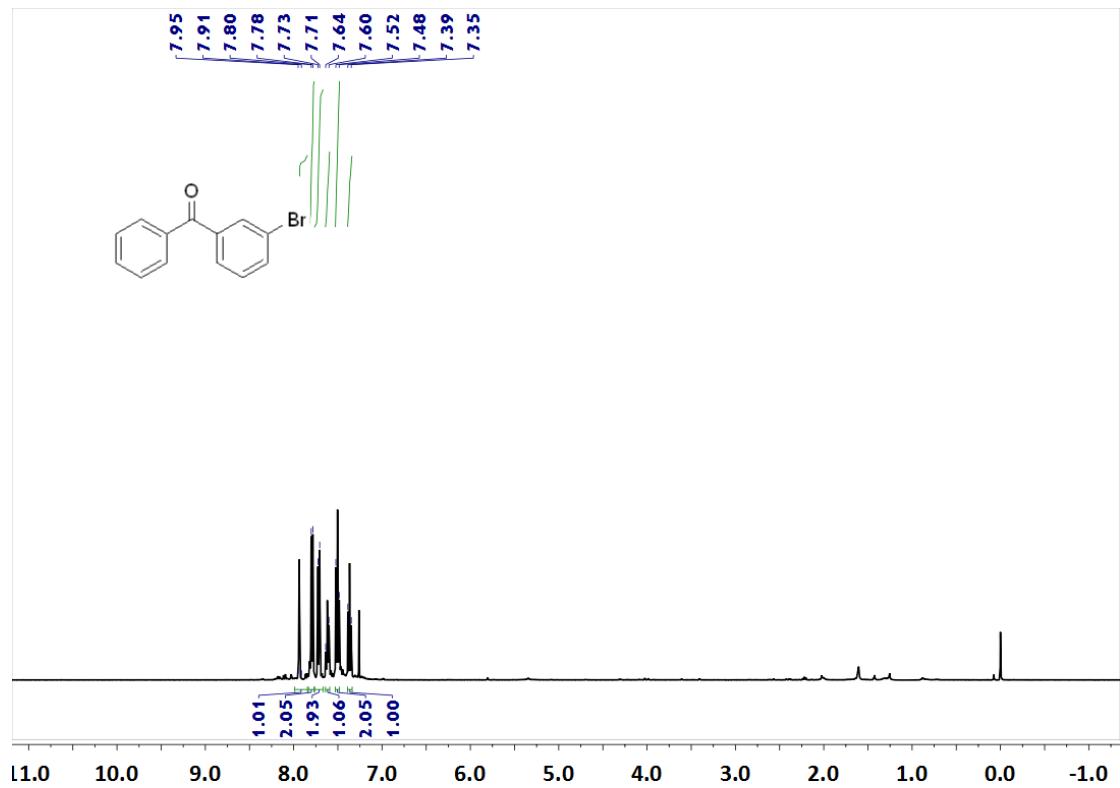




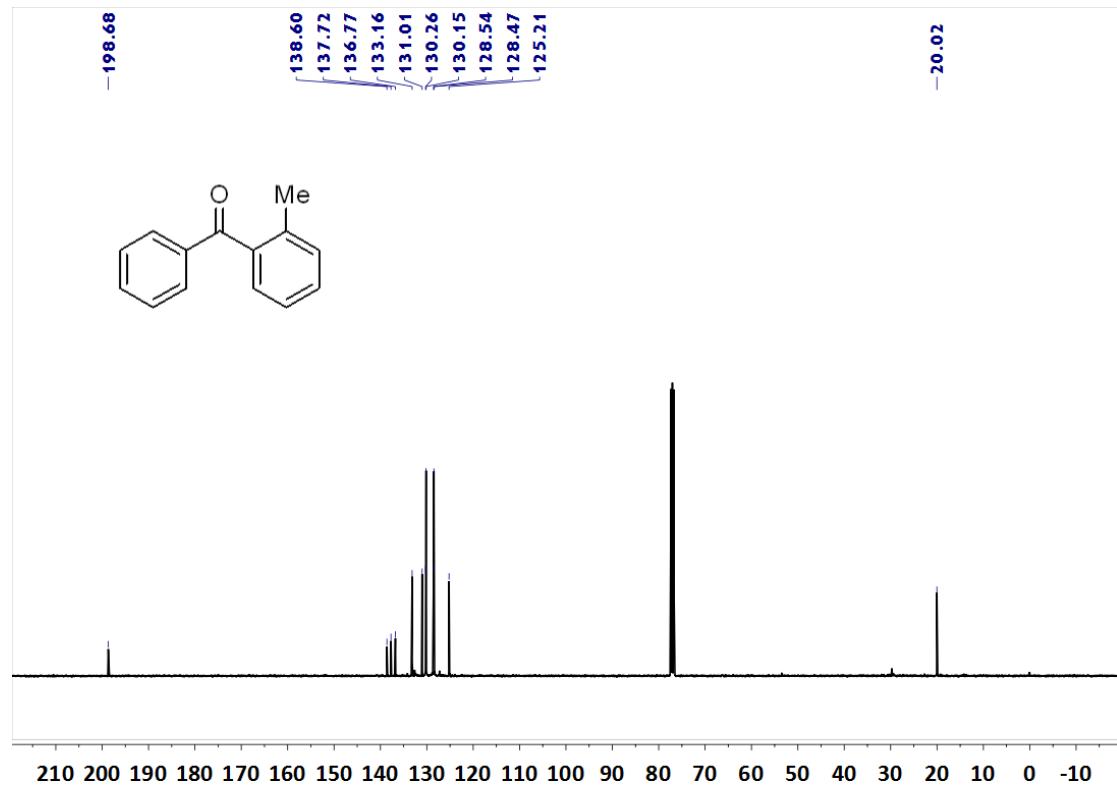
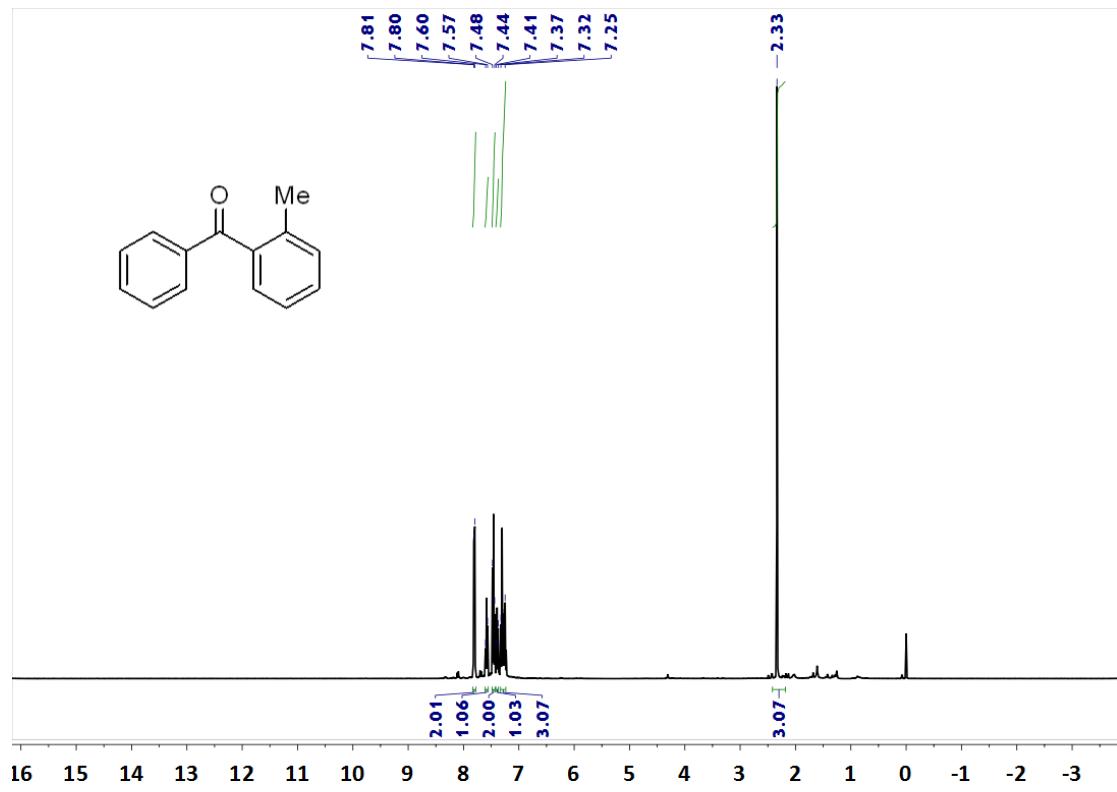
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4j**.



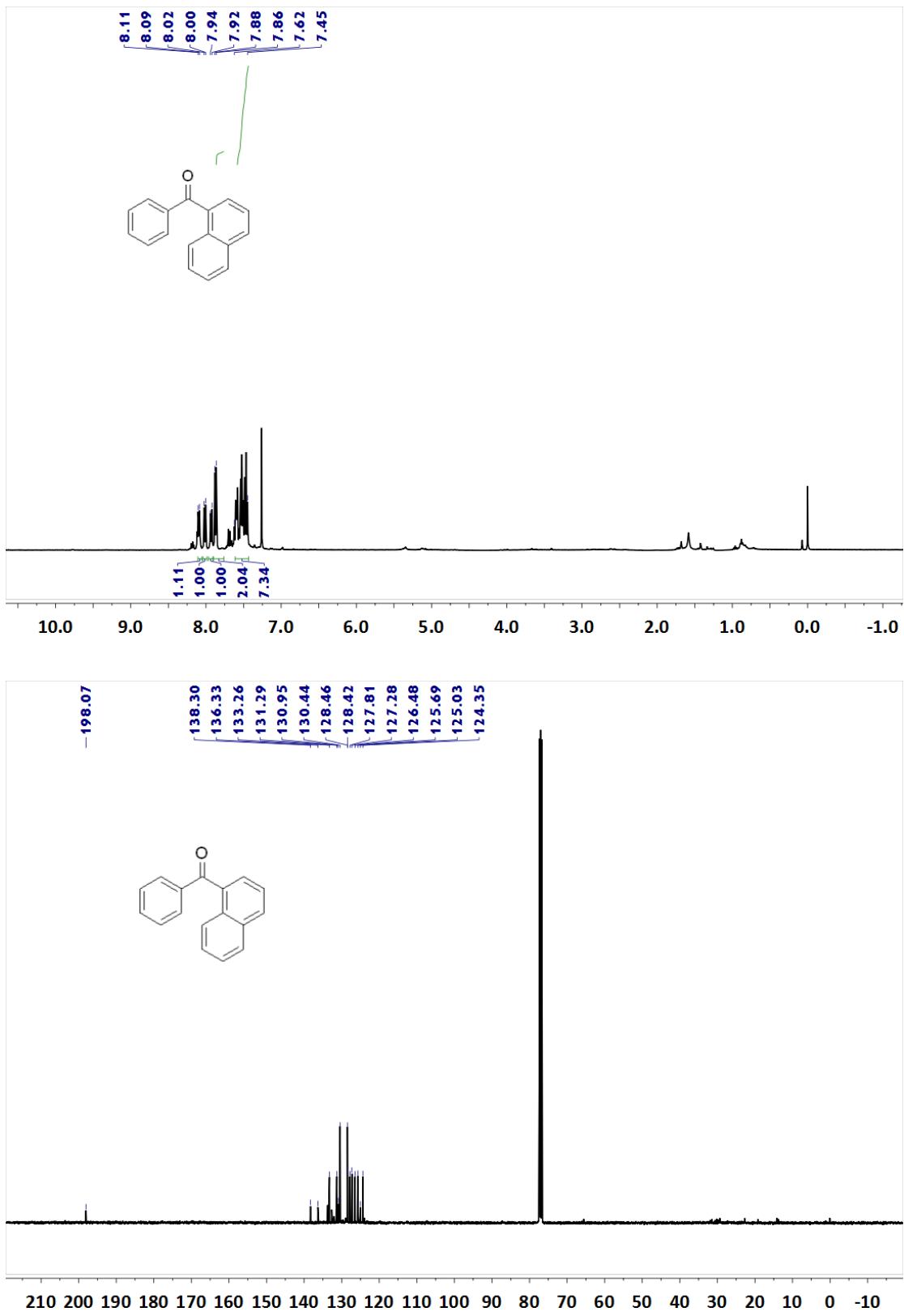
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4k**.



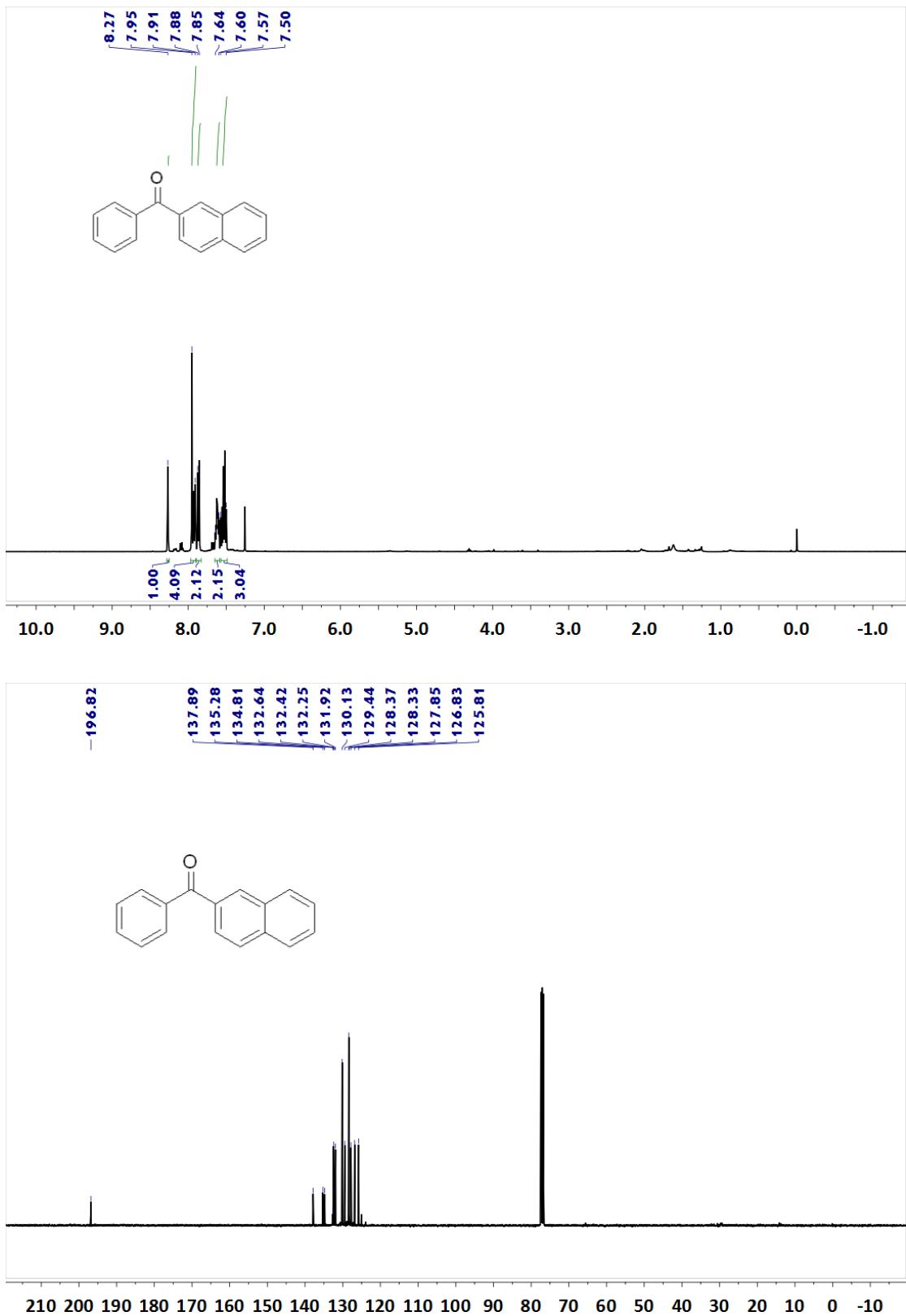
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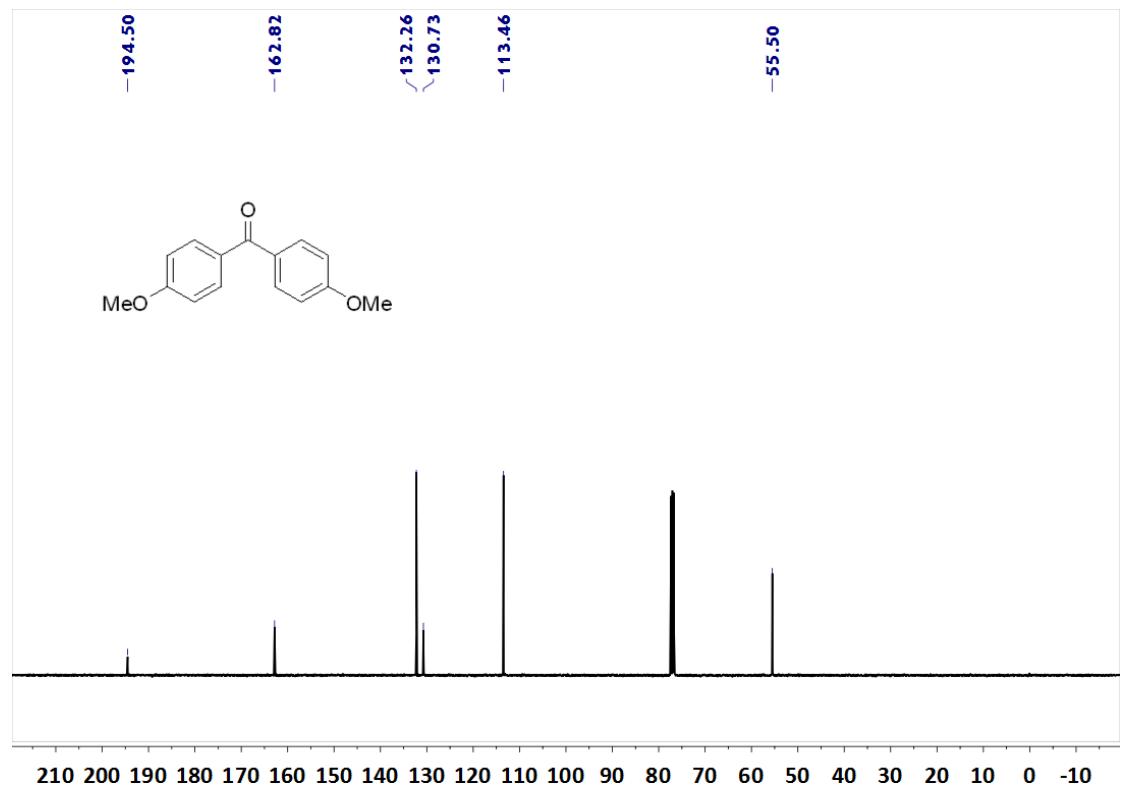
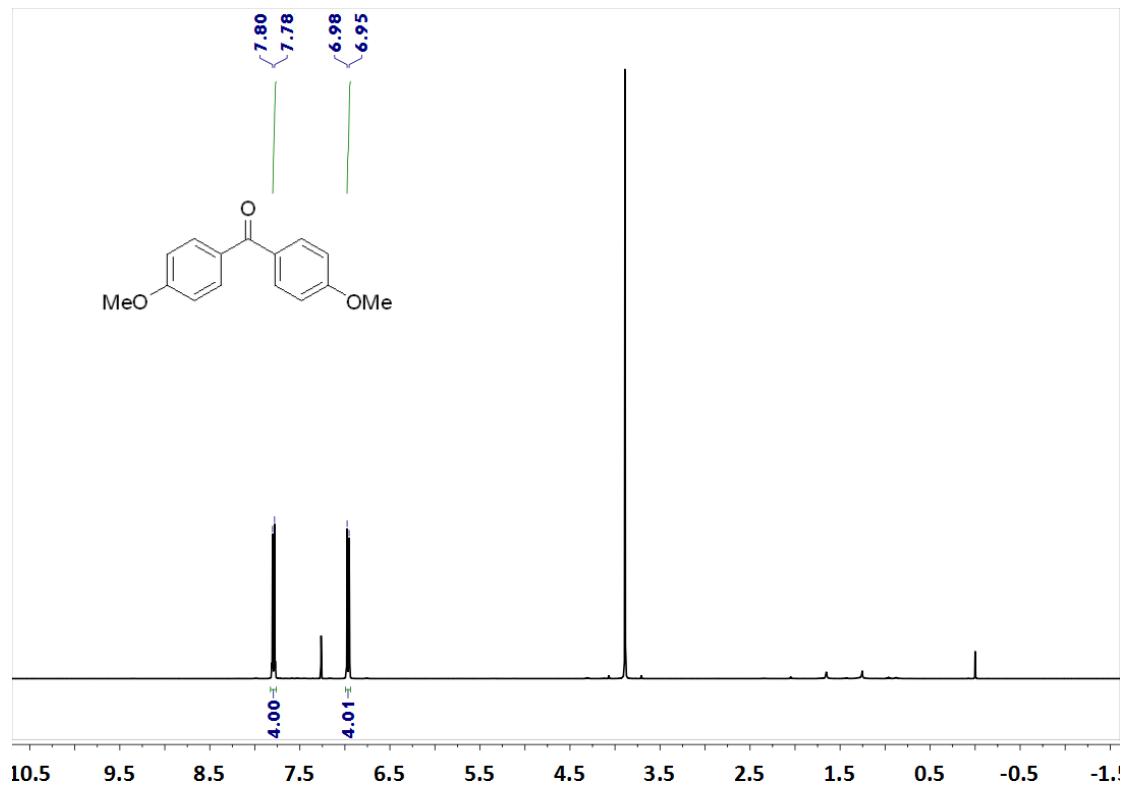


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4m**.

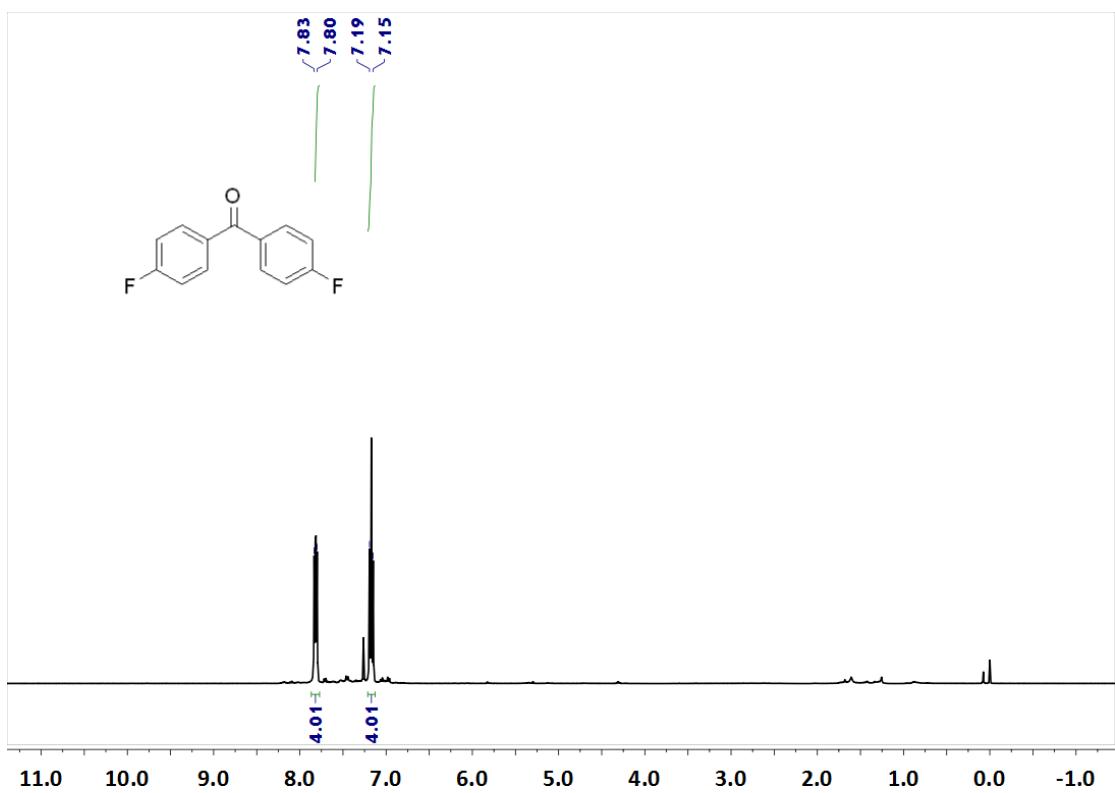
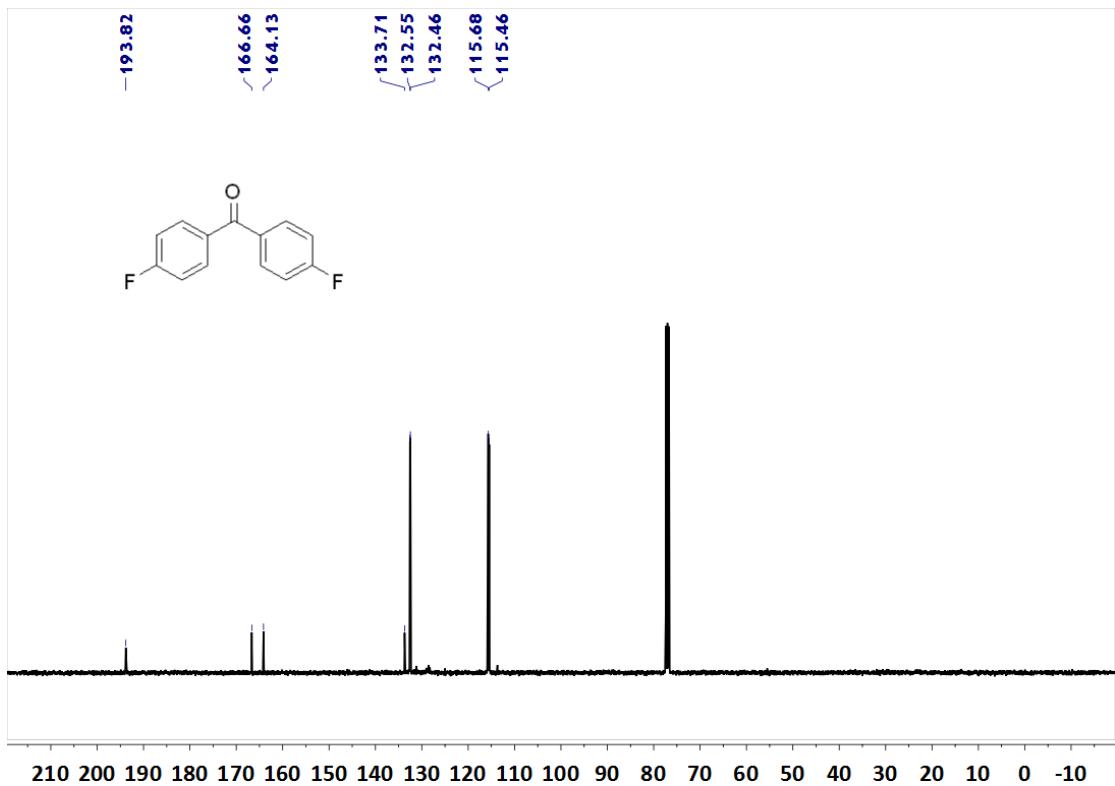


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **4n**.





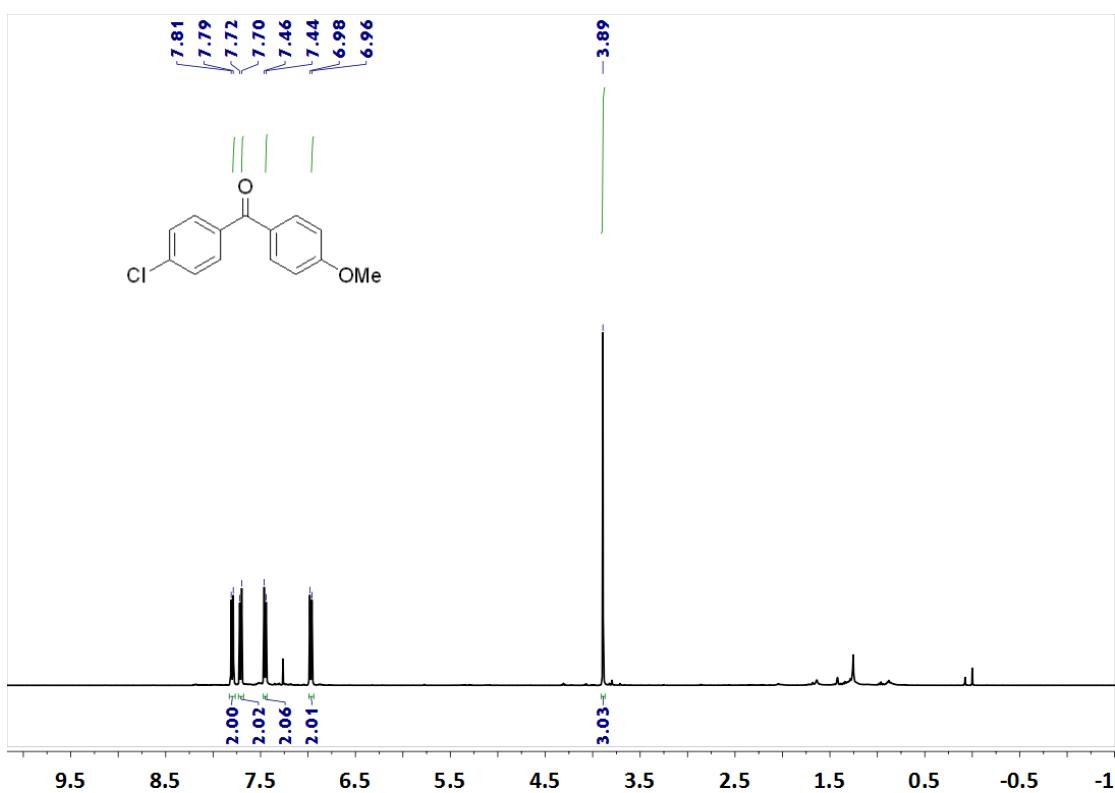
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **4p**.

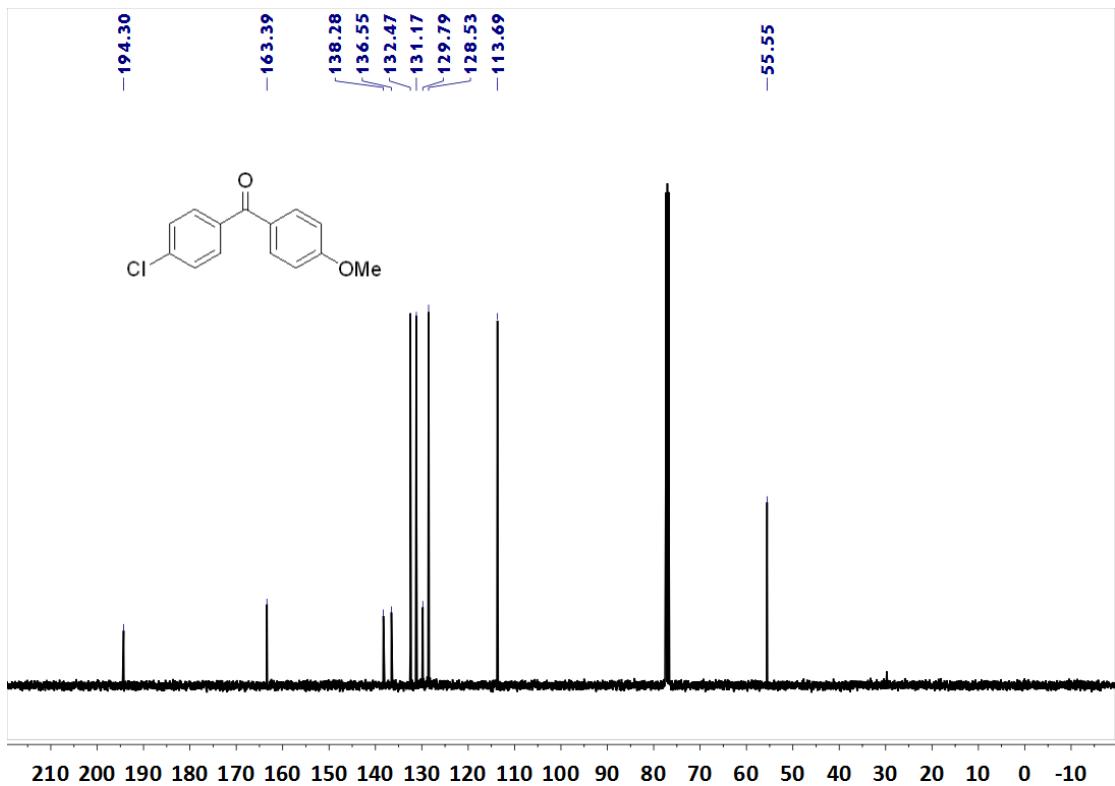


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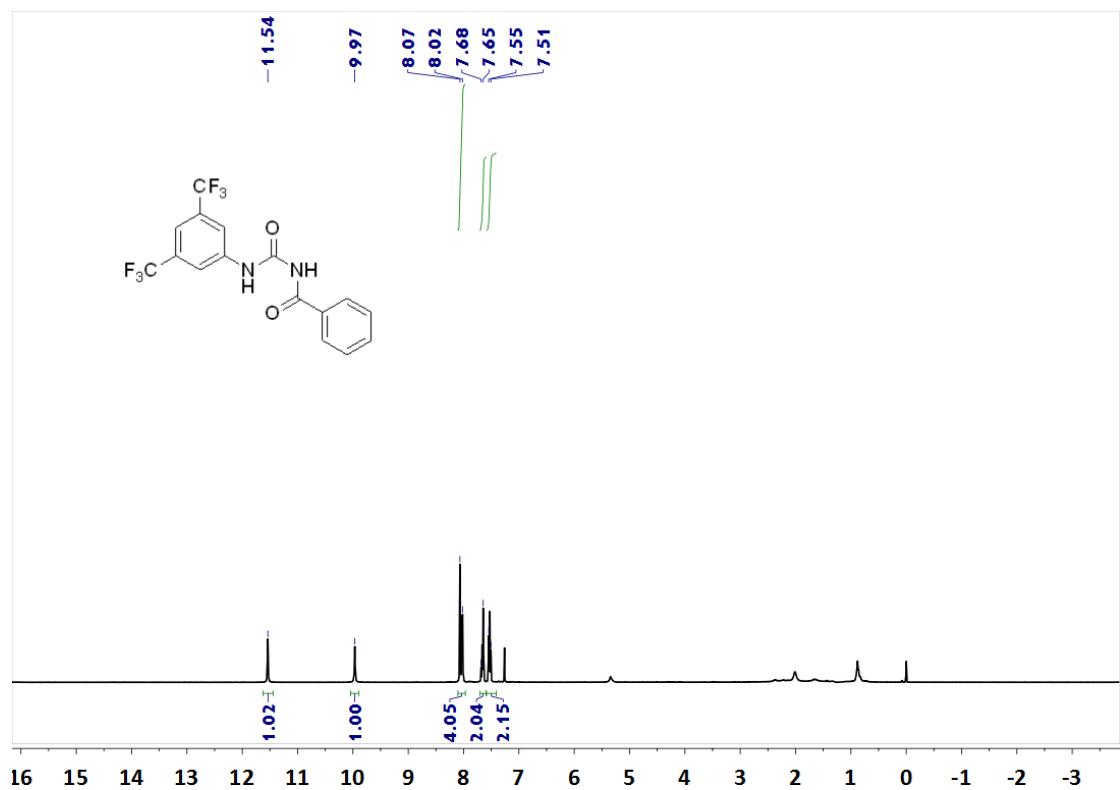


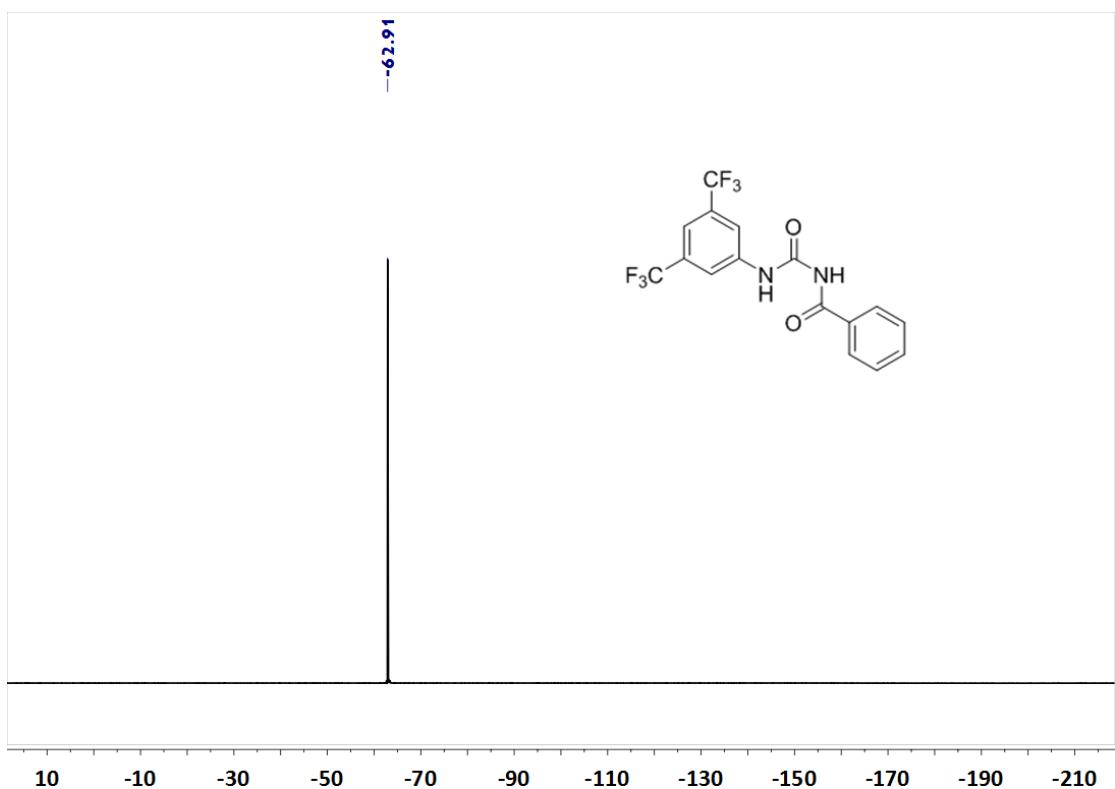
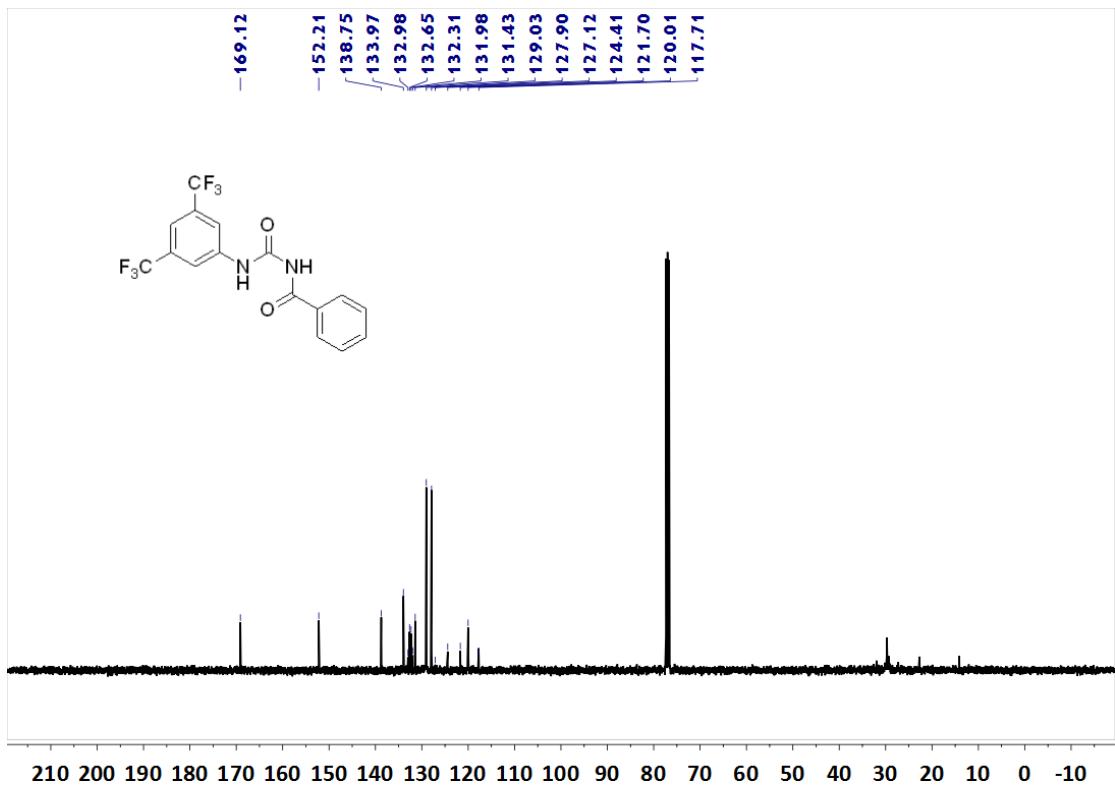
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **4q**.



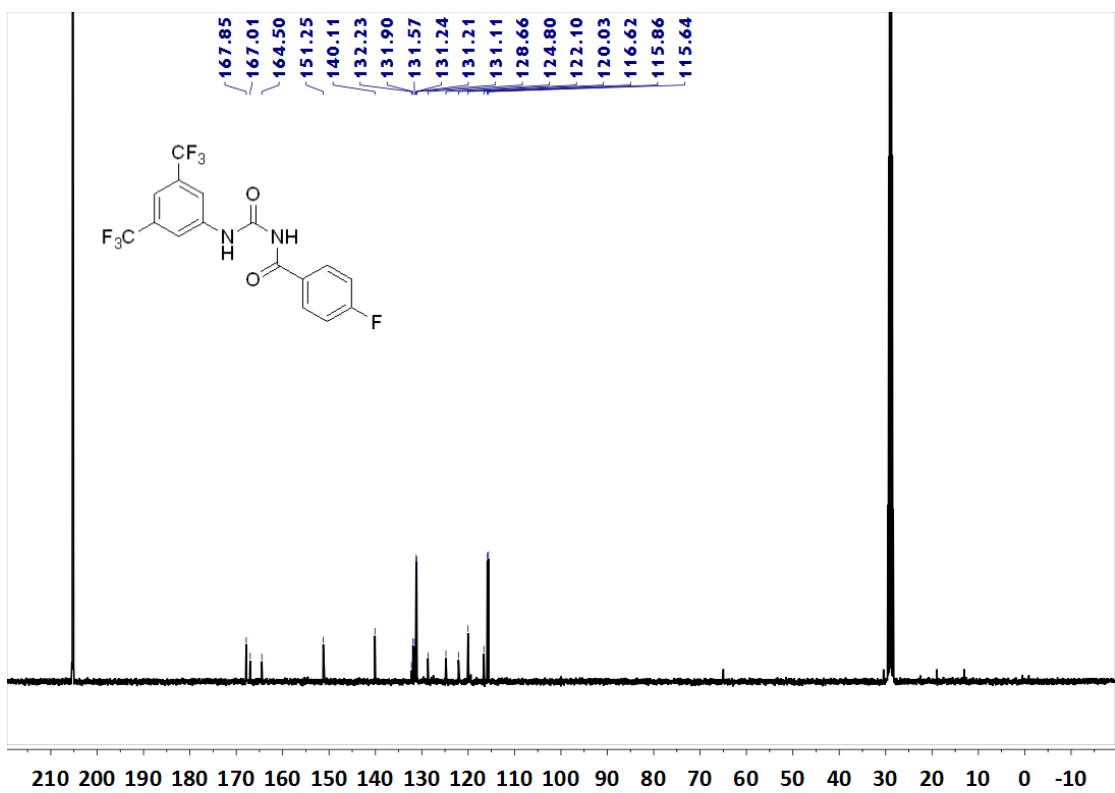
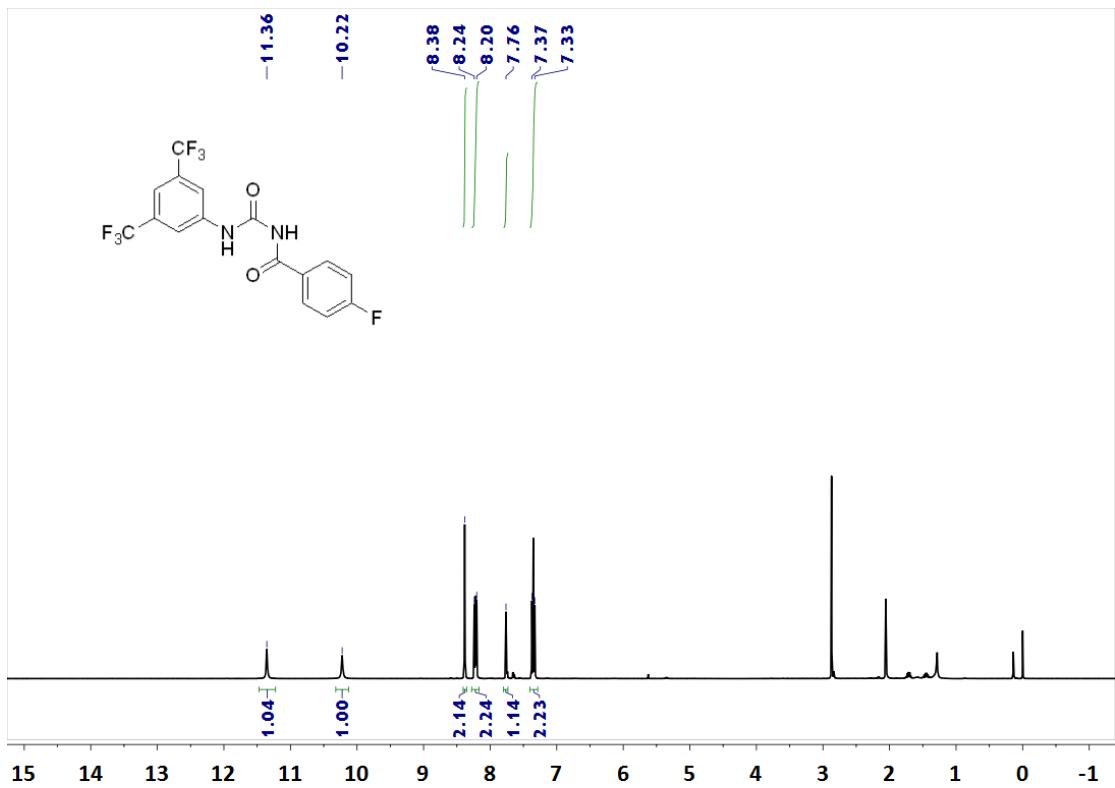


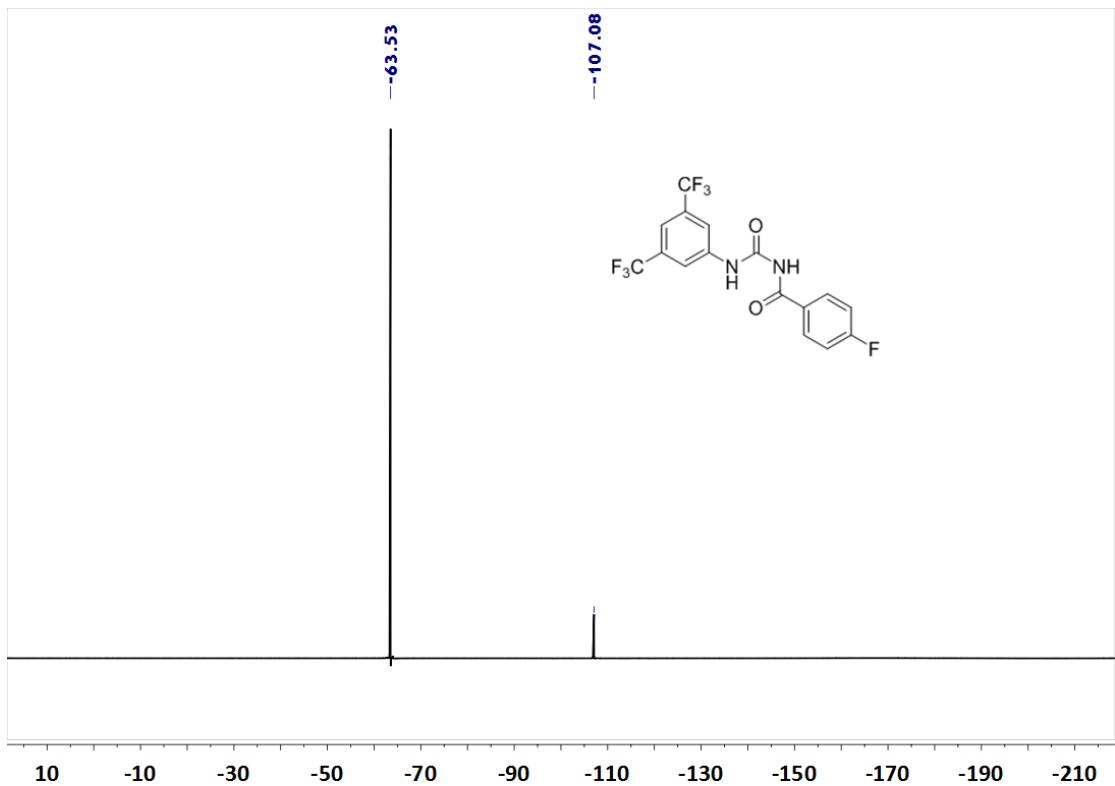
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6a**.



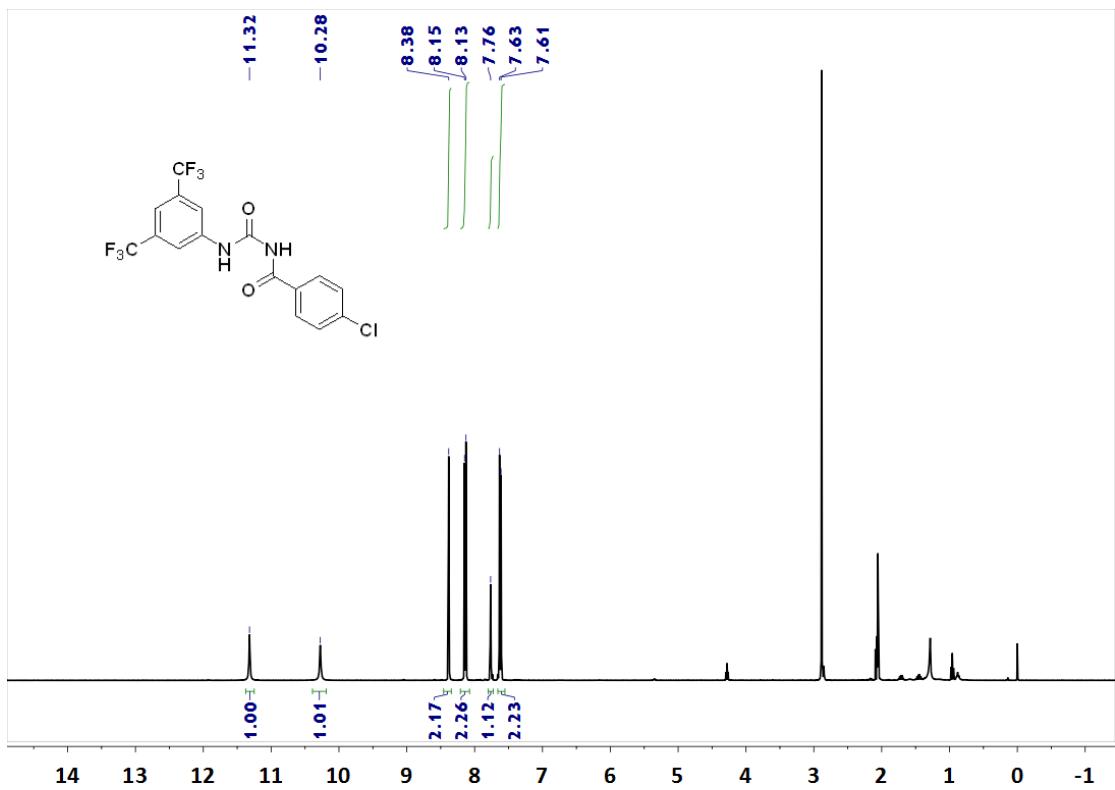


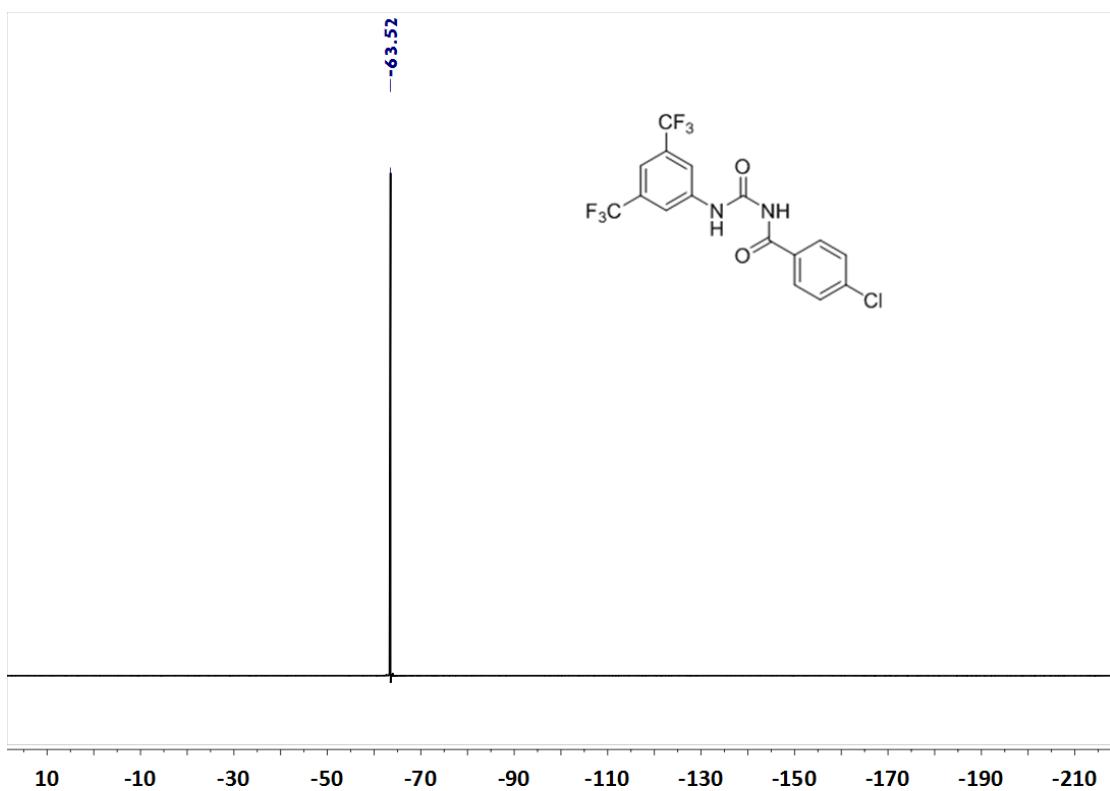
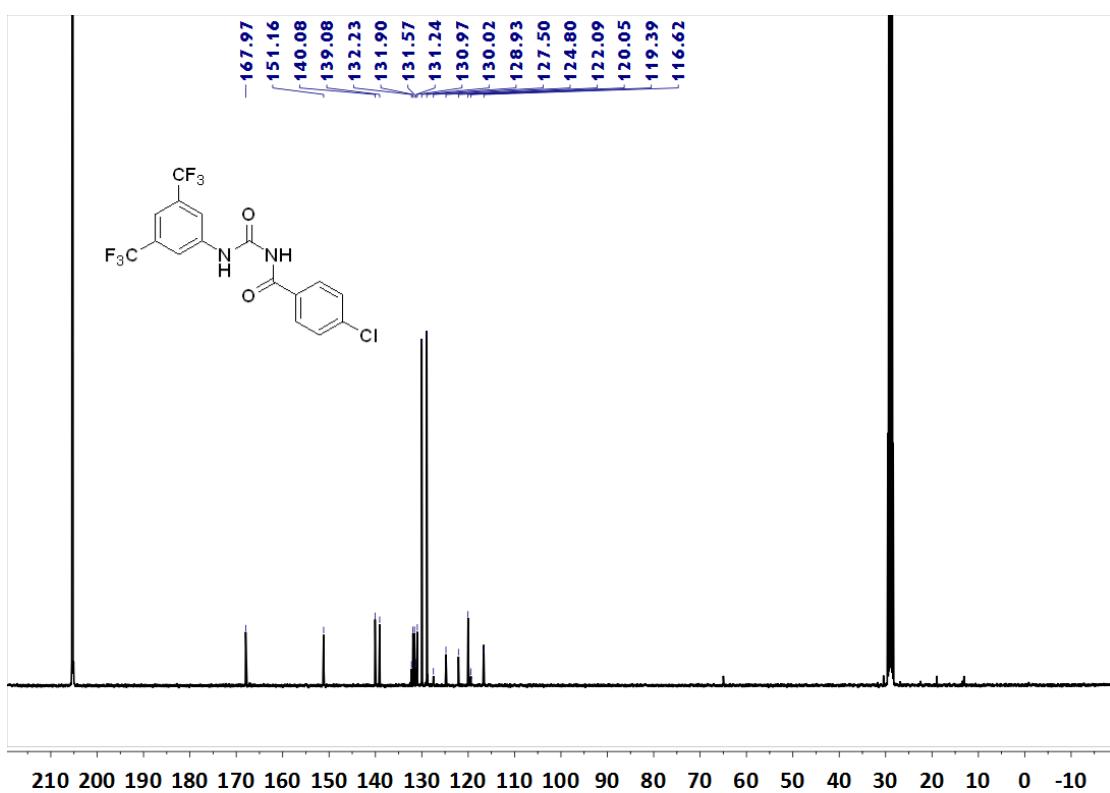
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6b**.



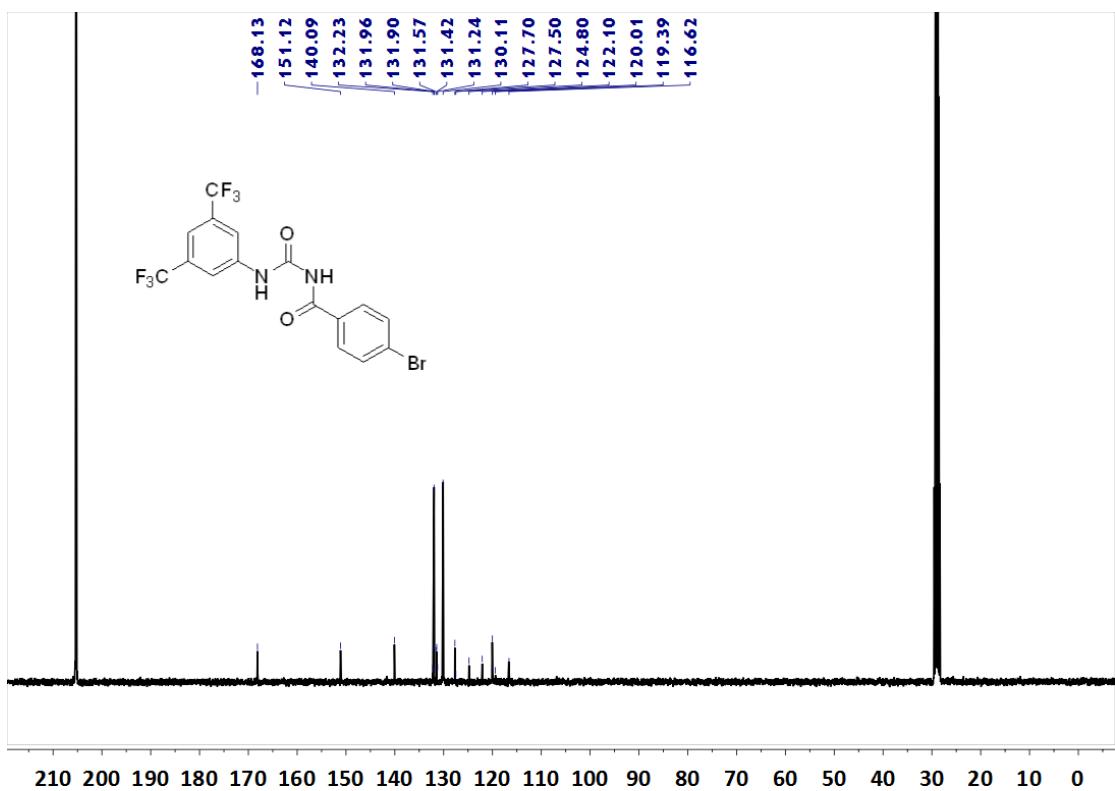
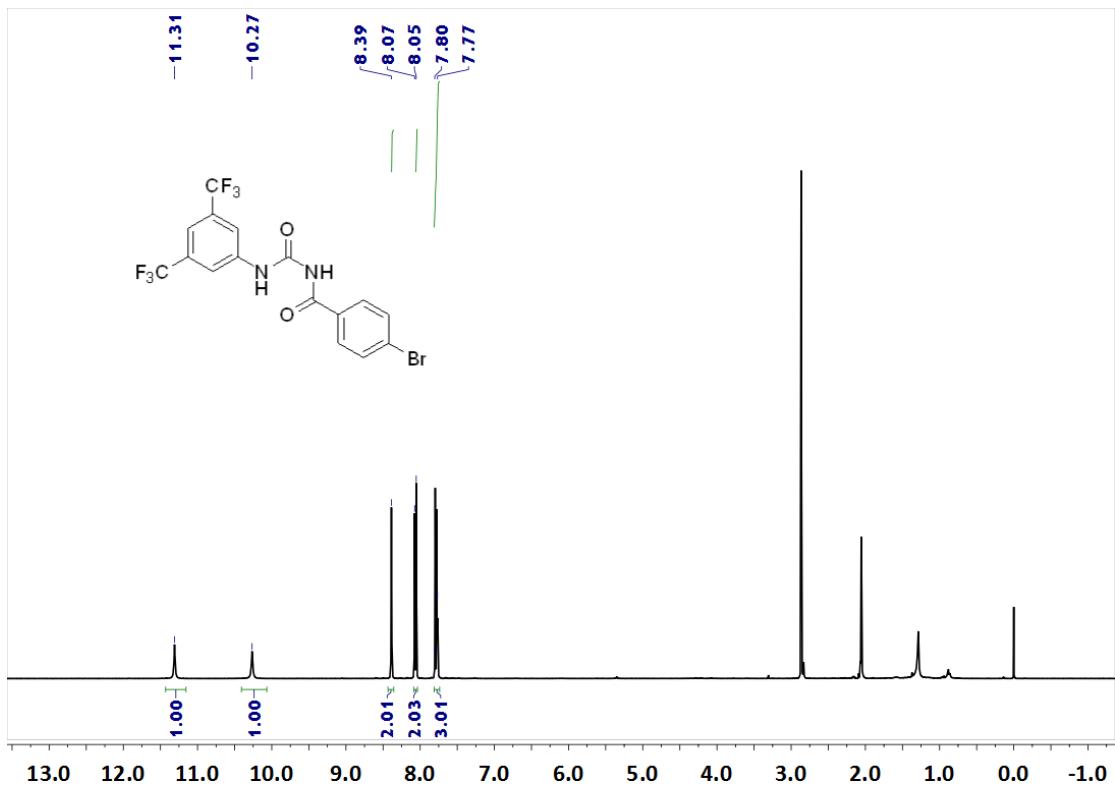


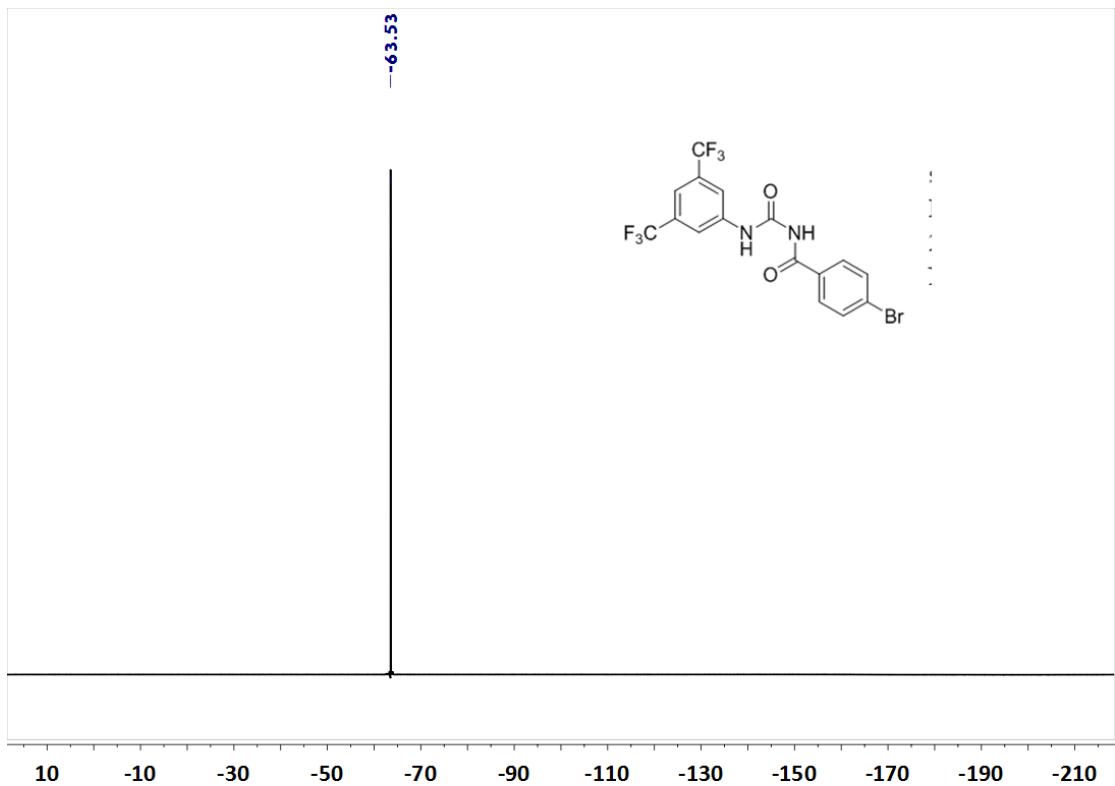
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6c**.



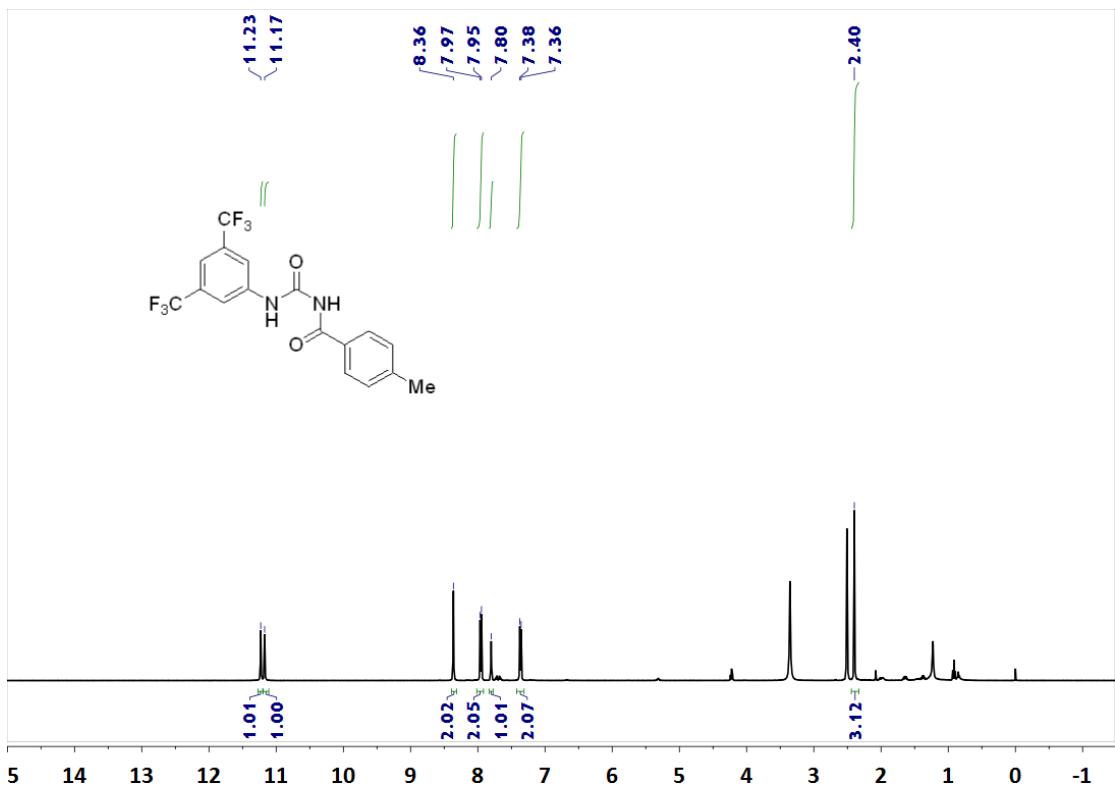


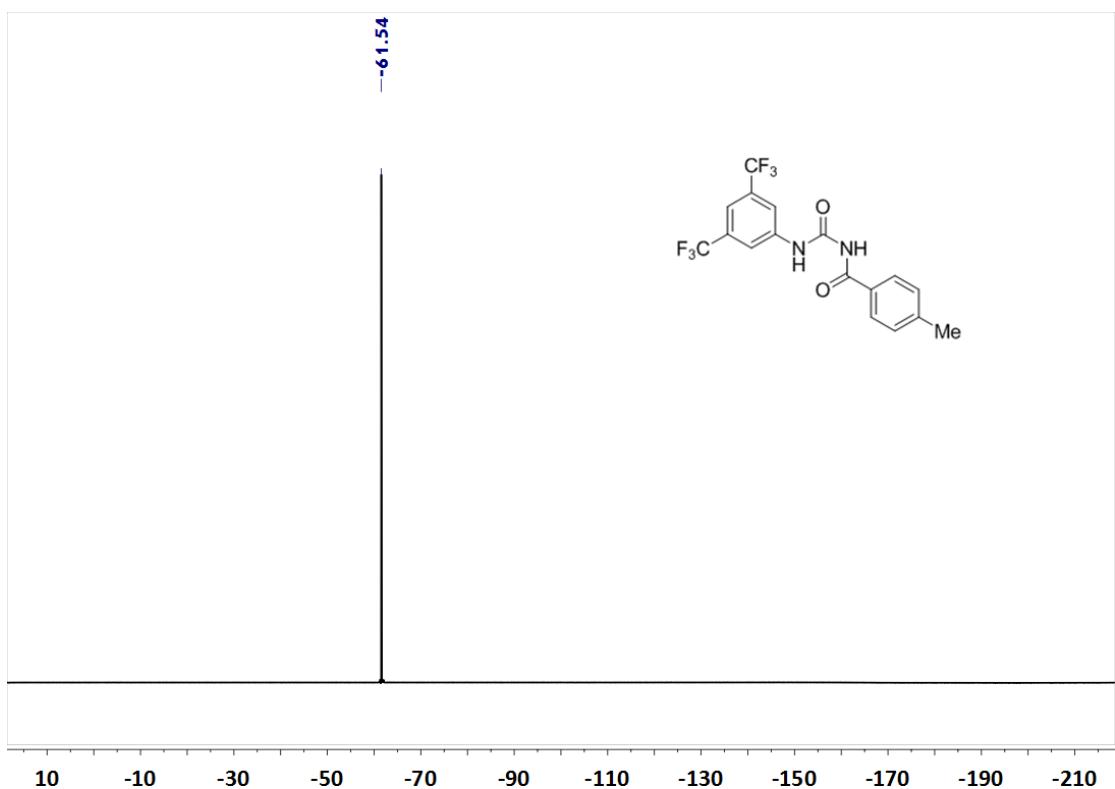
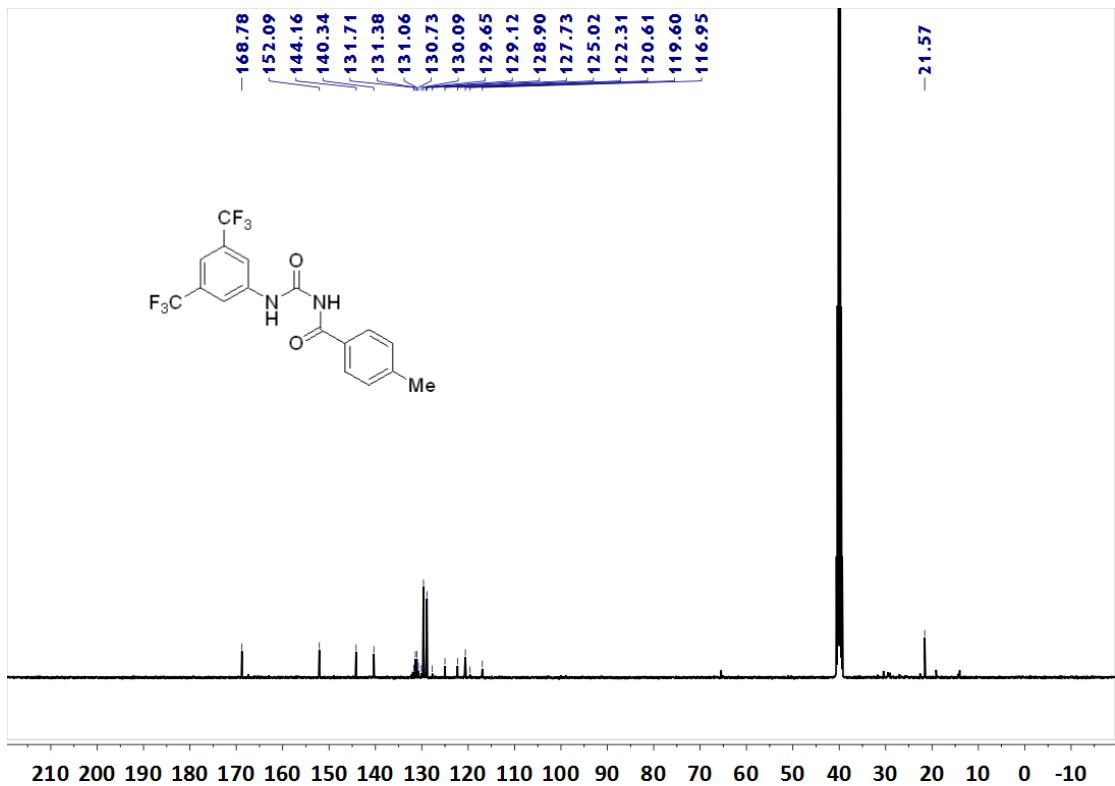
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6d**.



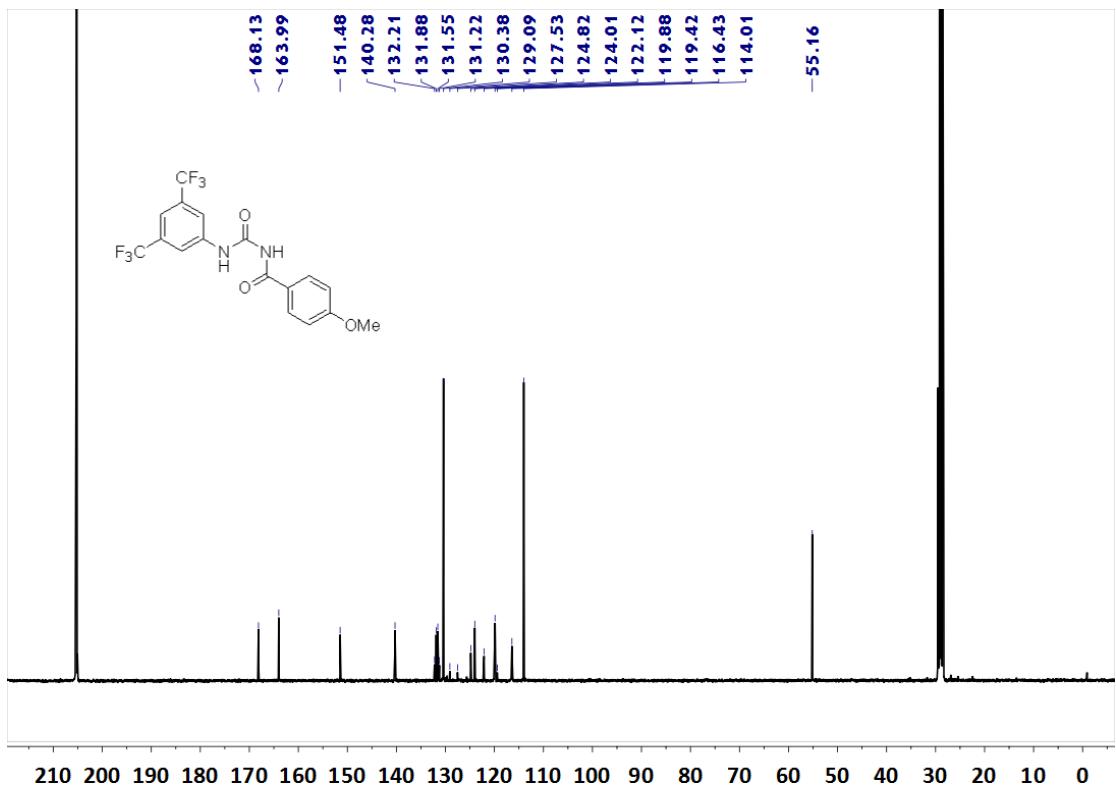
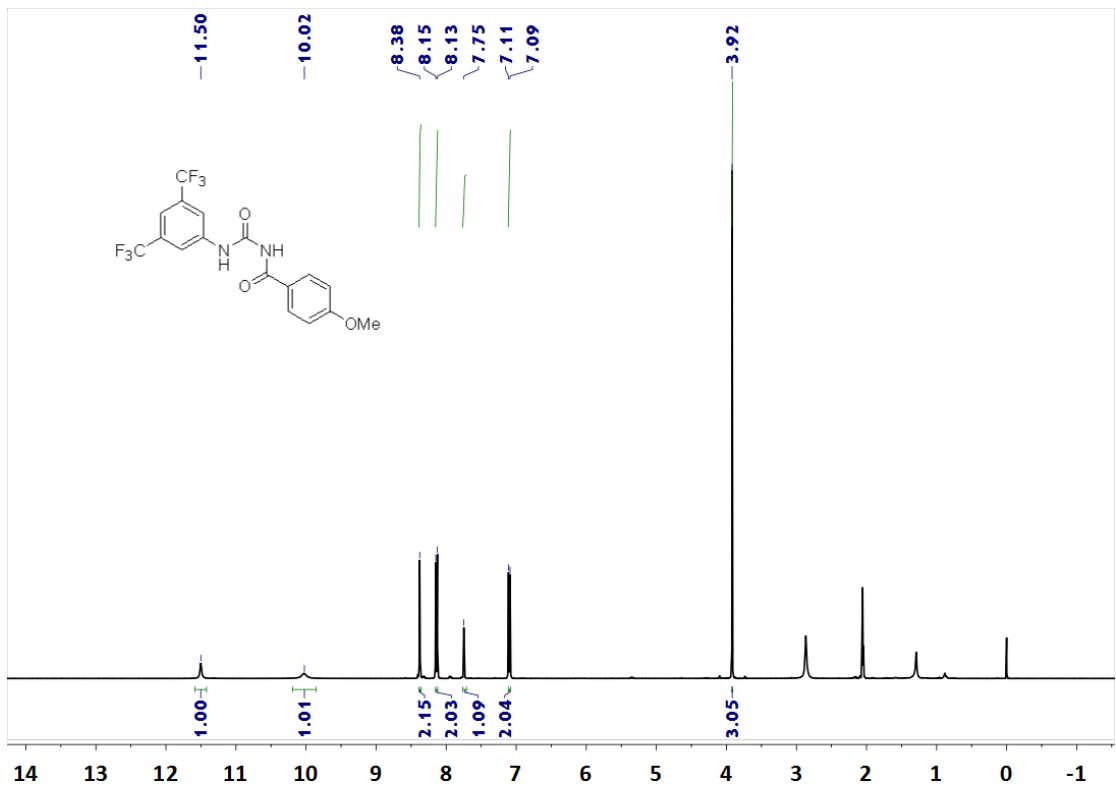


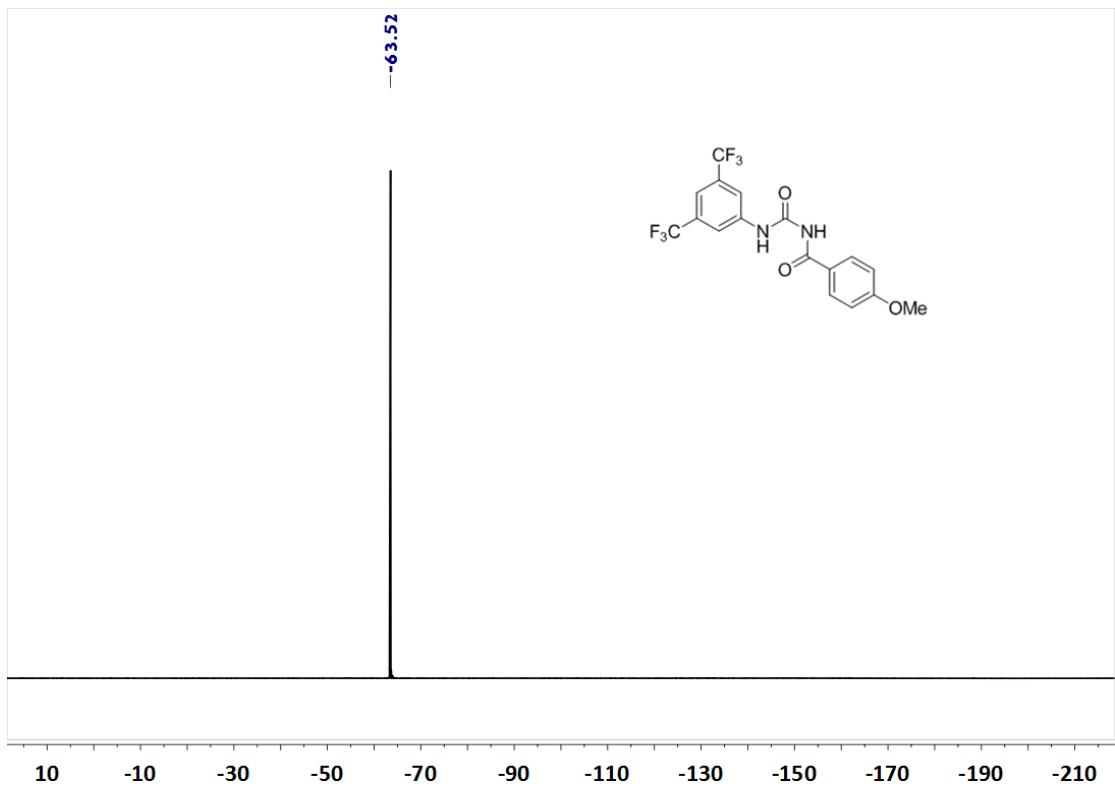
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6e**.



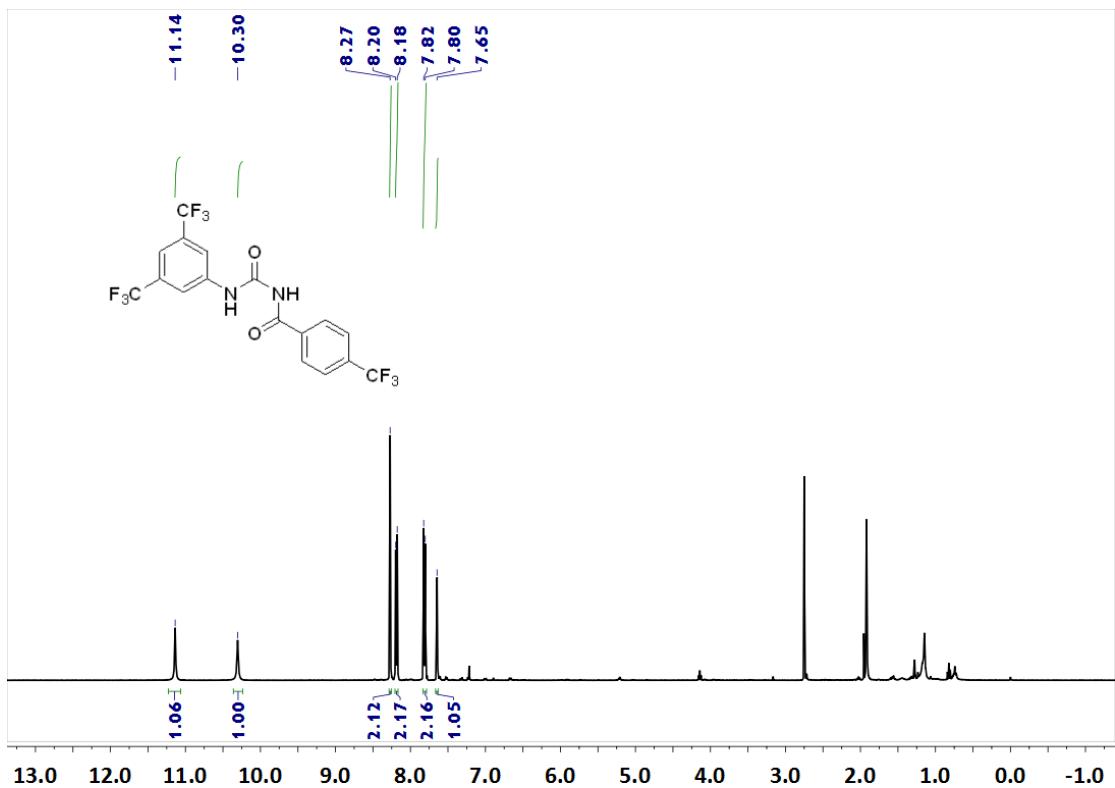


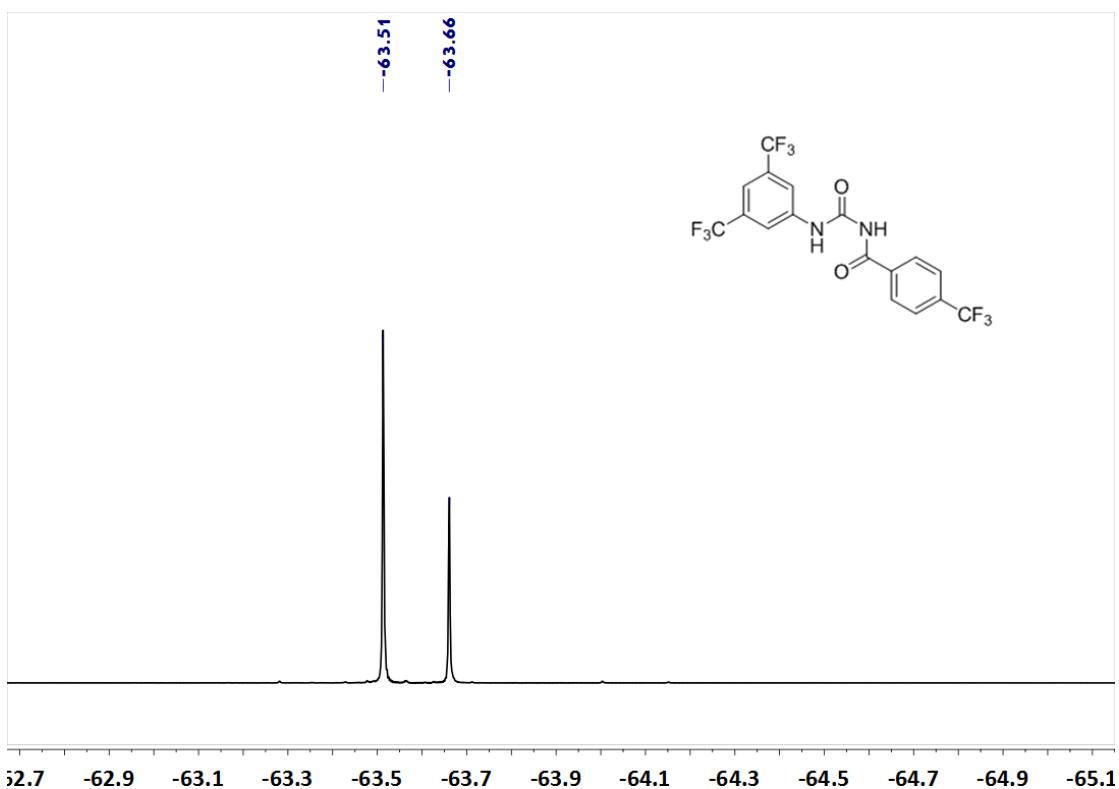
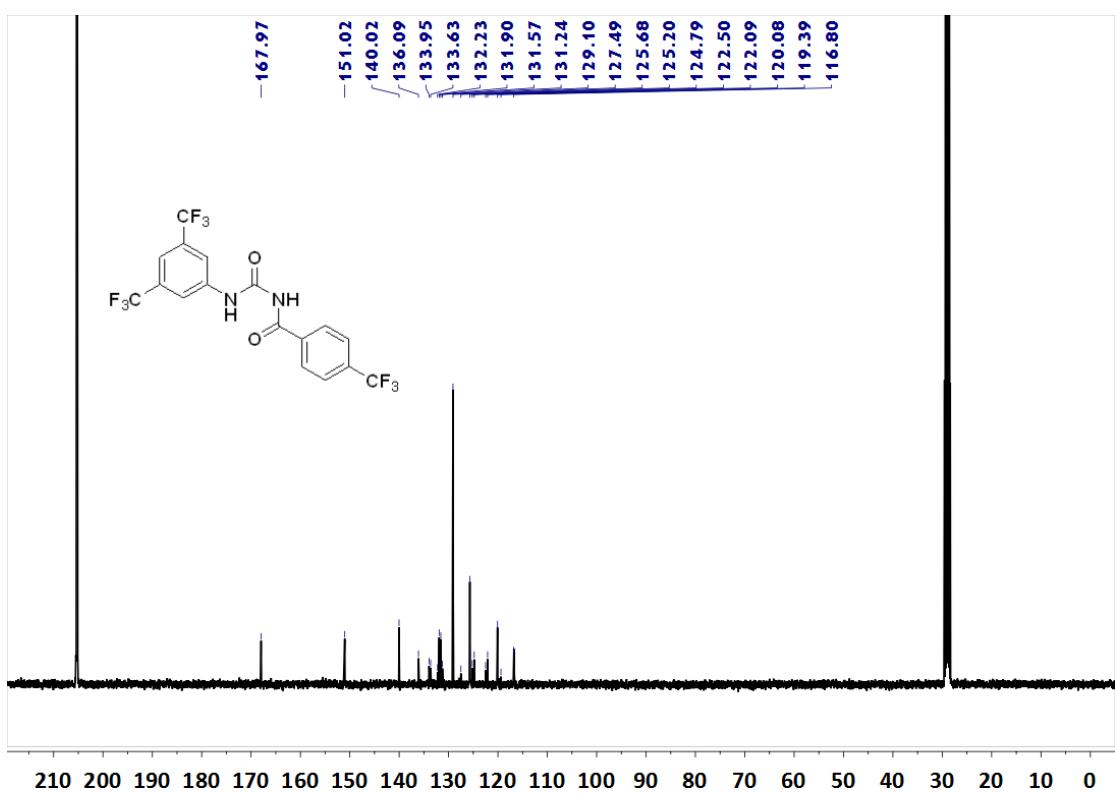
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6f**.



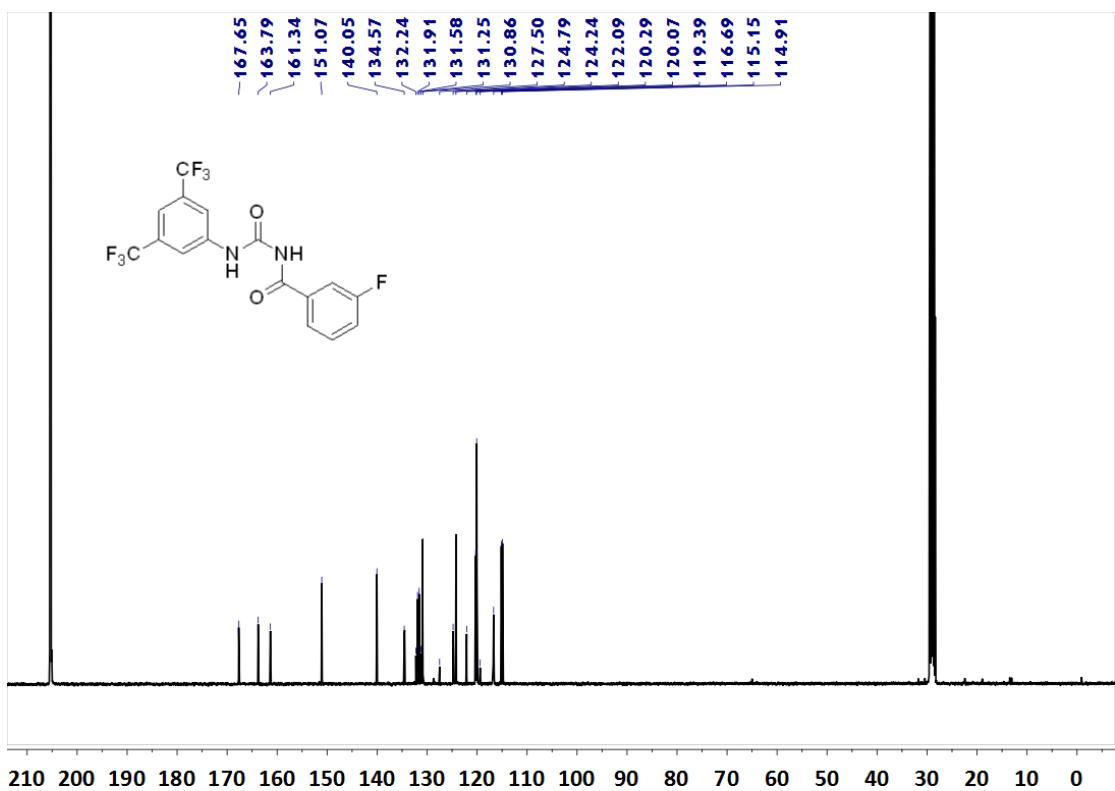
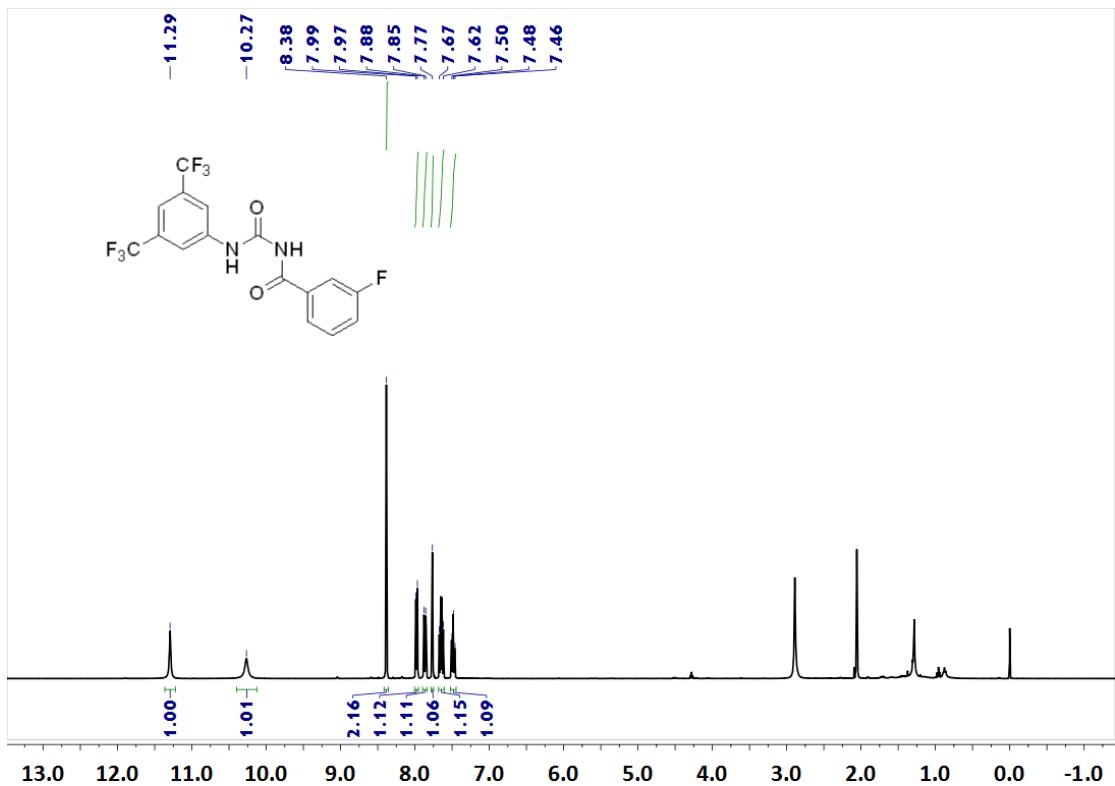


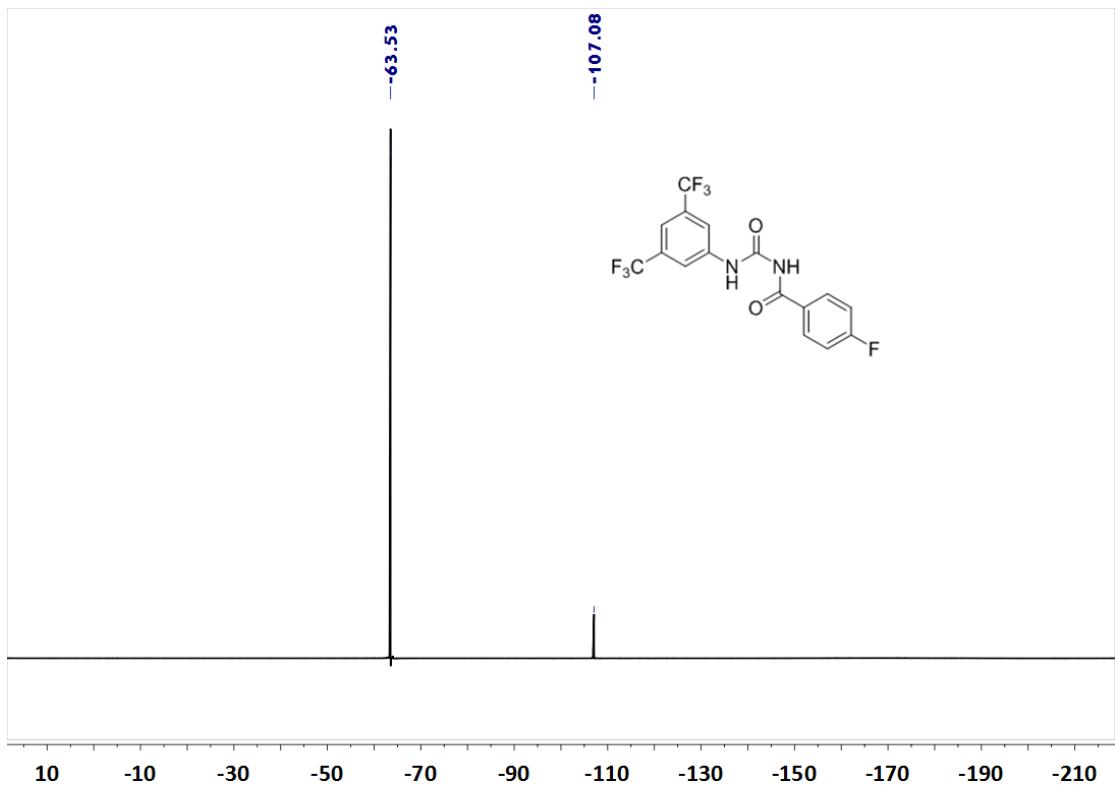
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **6g**.



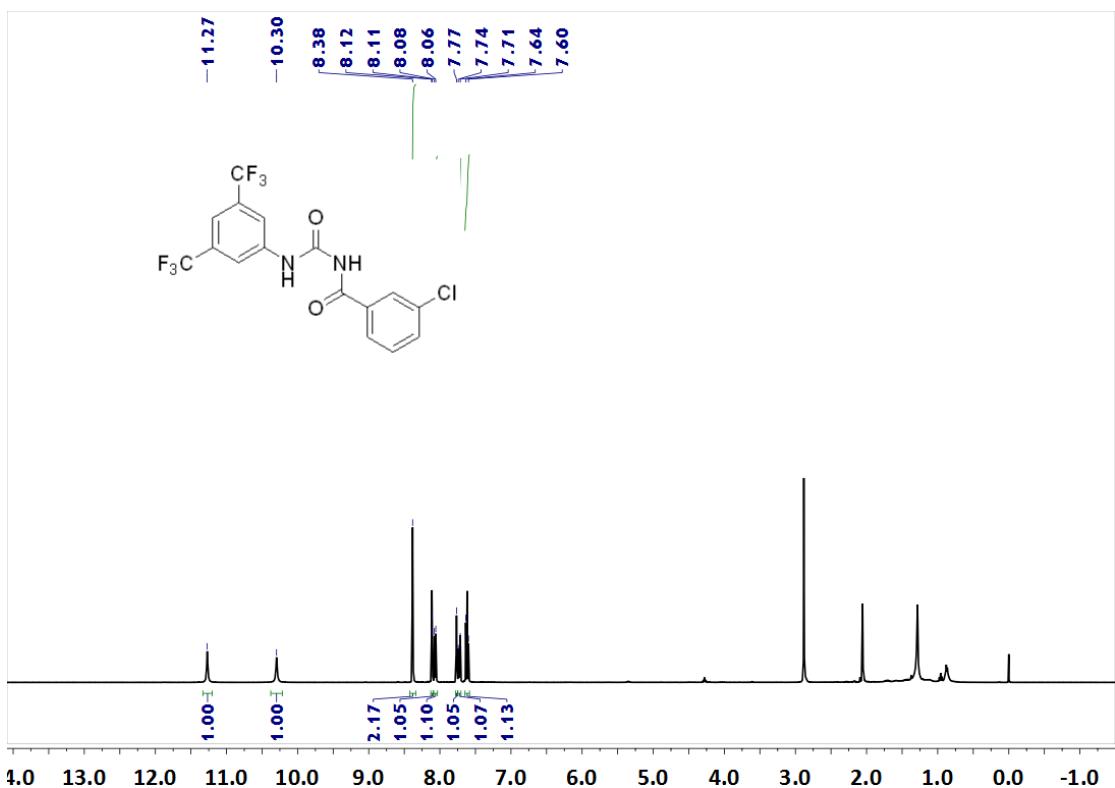


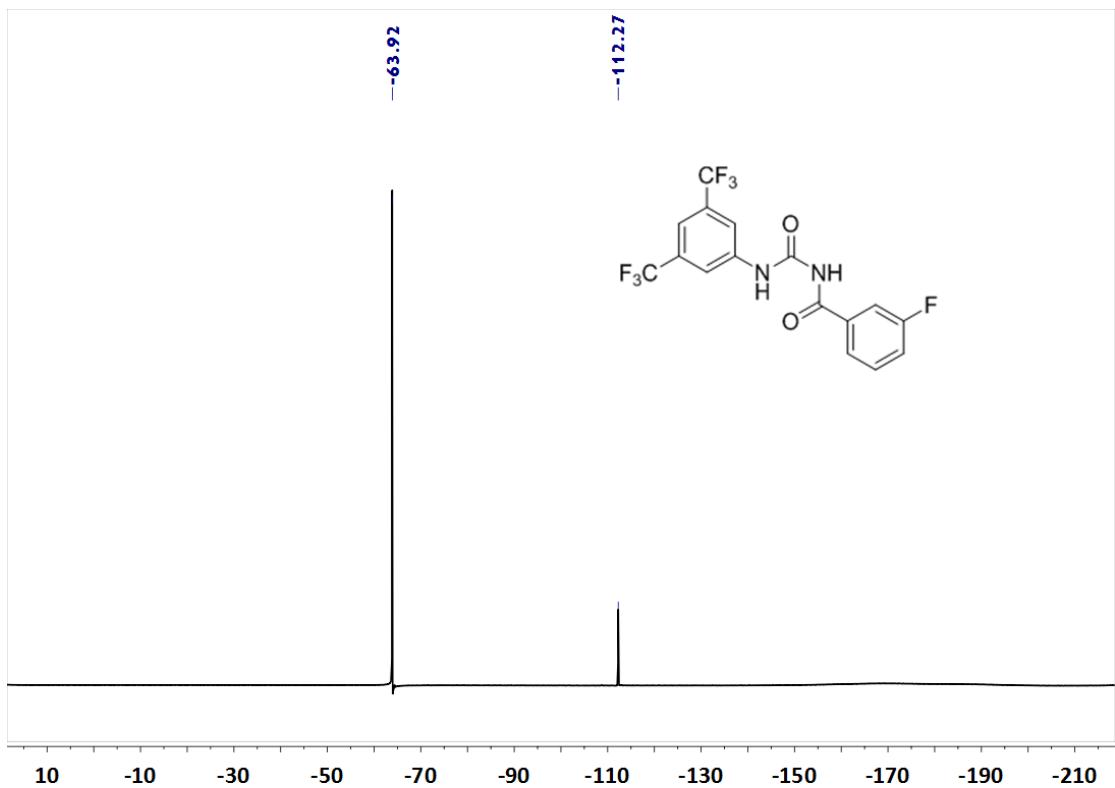
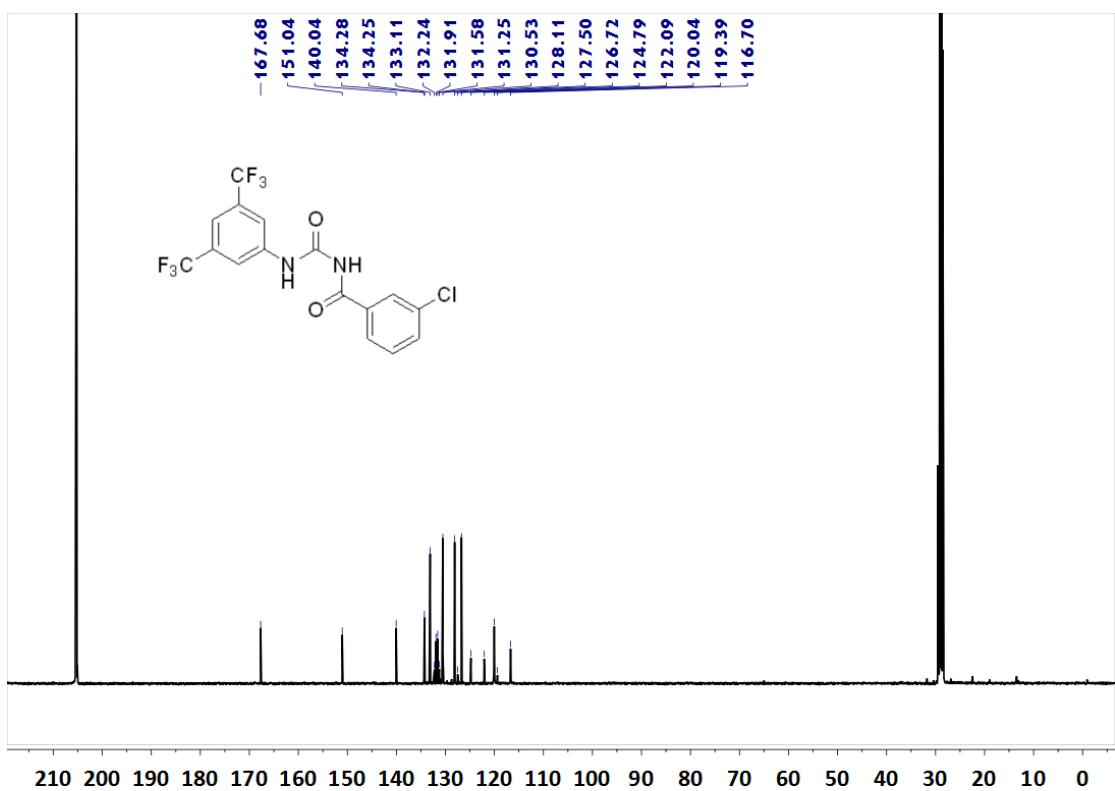
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6h**.



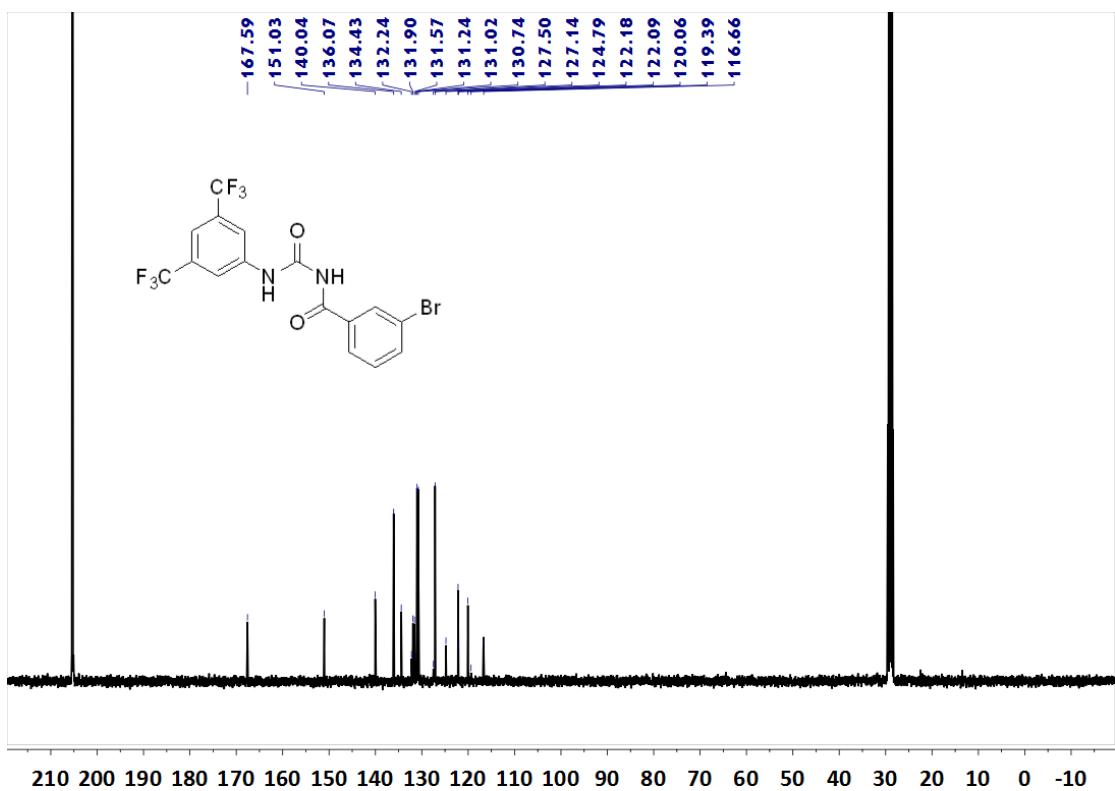
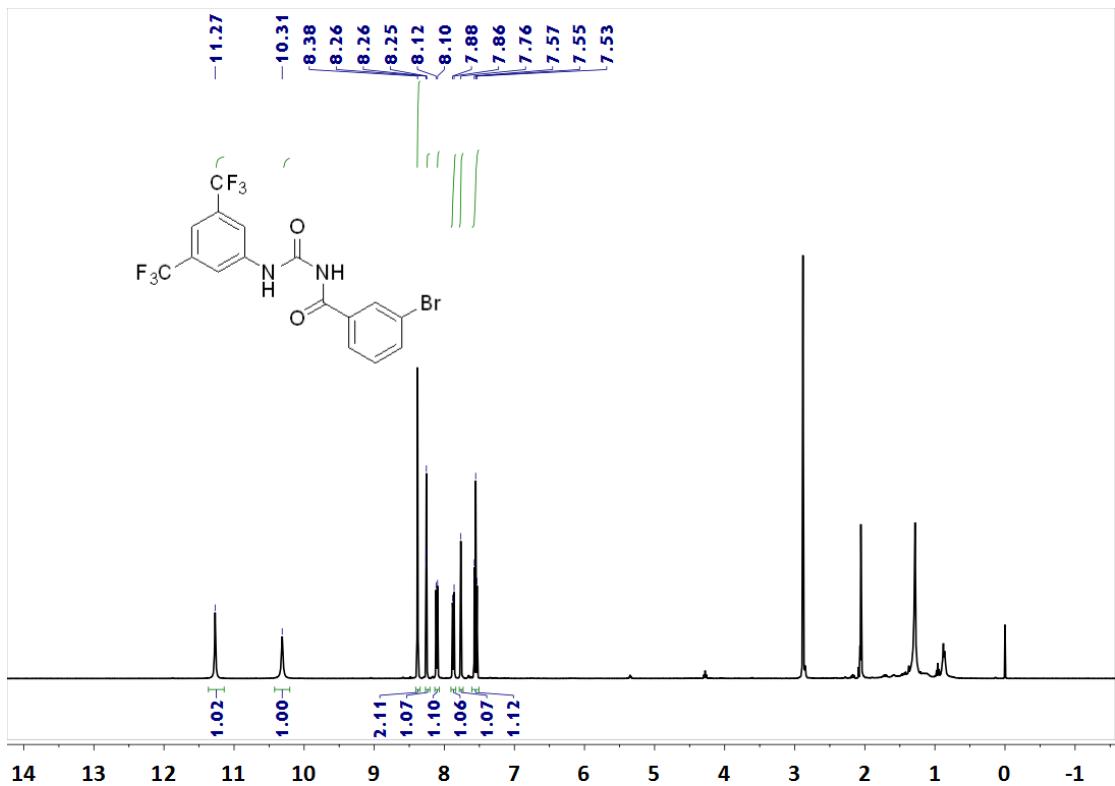


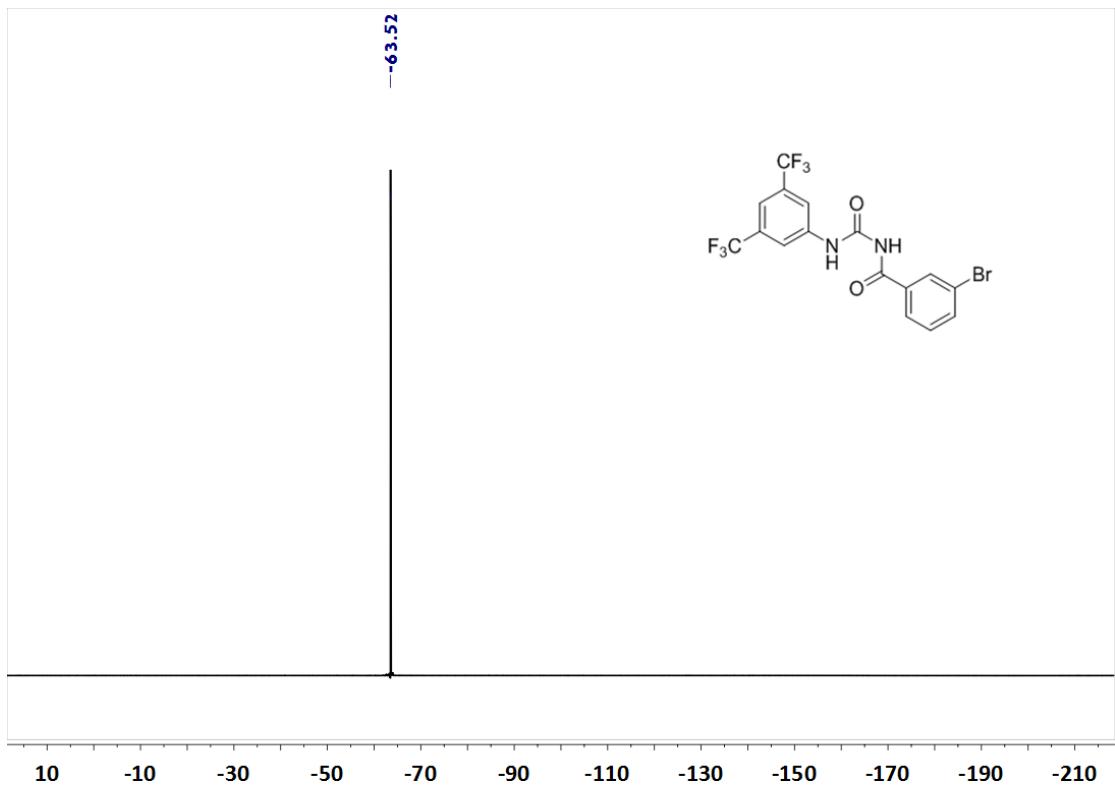
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6i**.



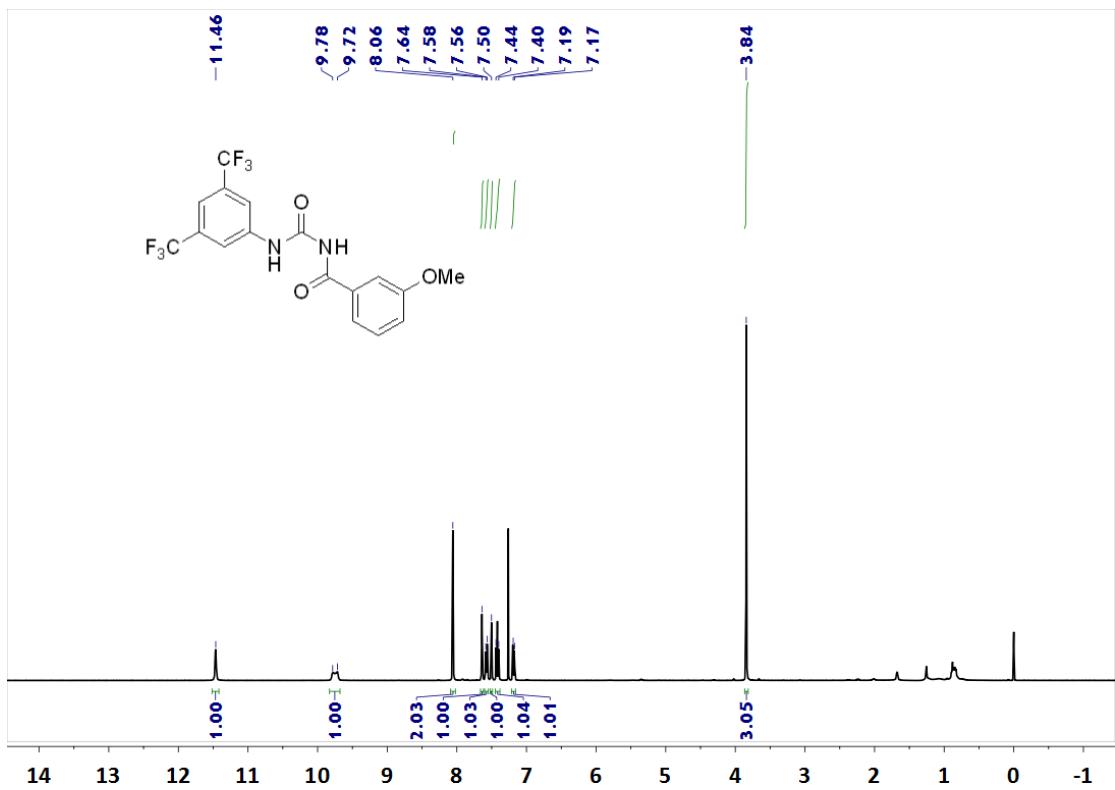


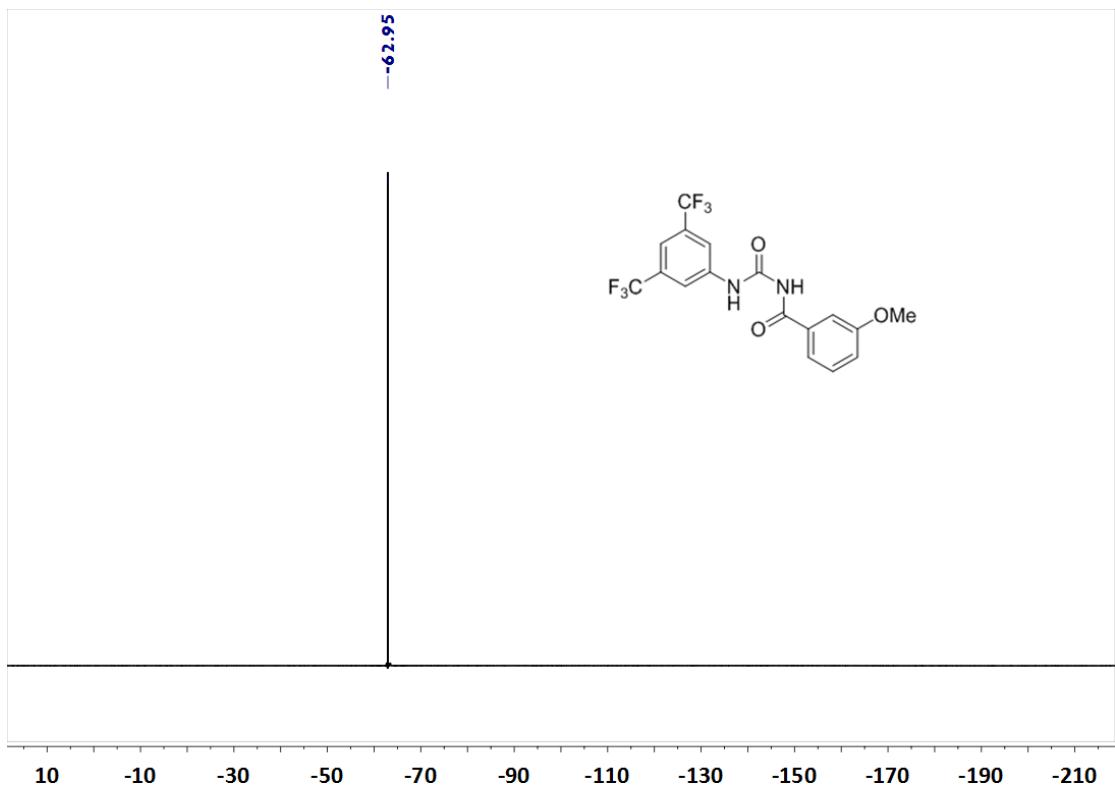
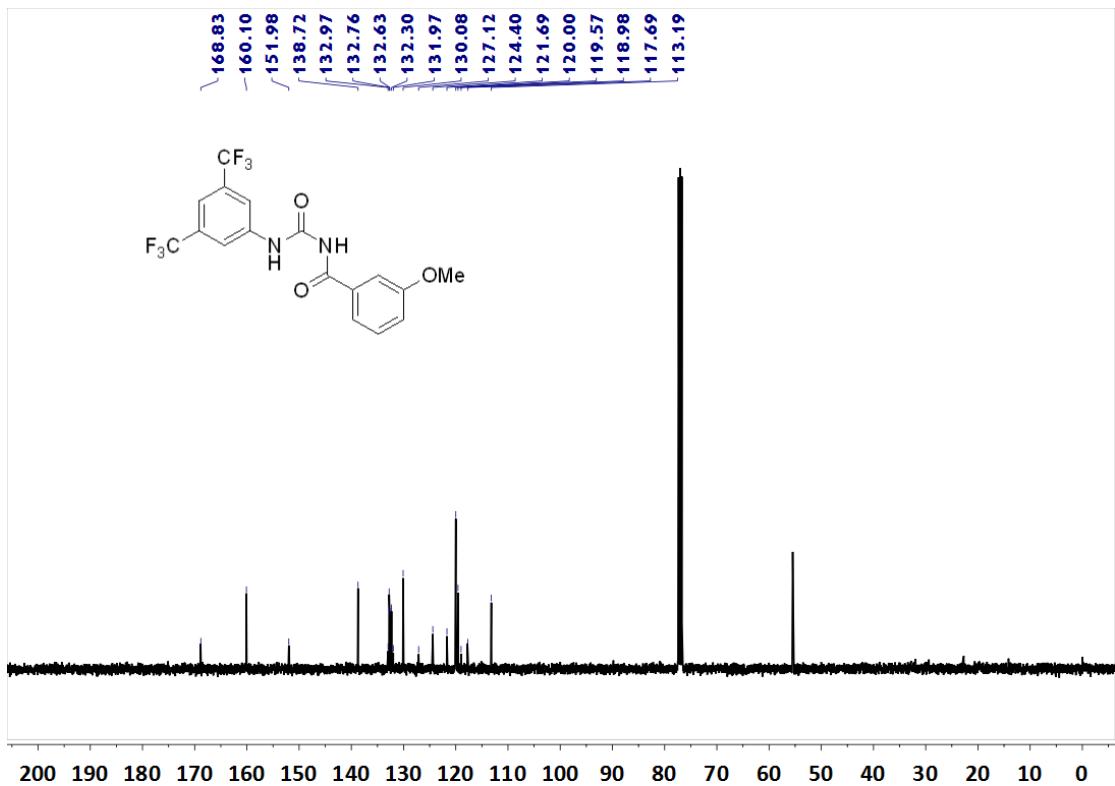
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6j**.



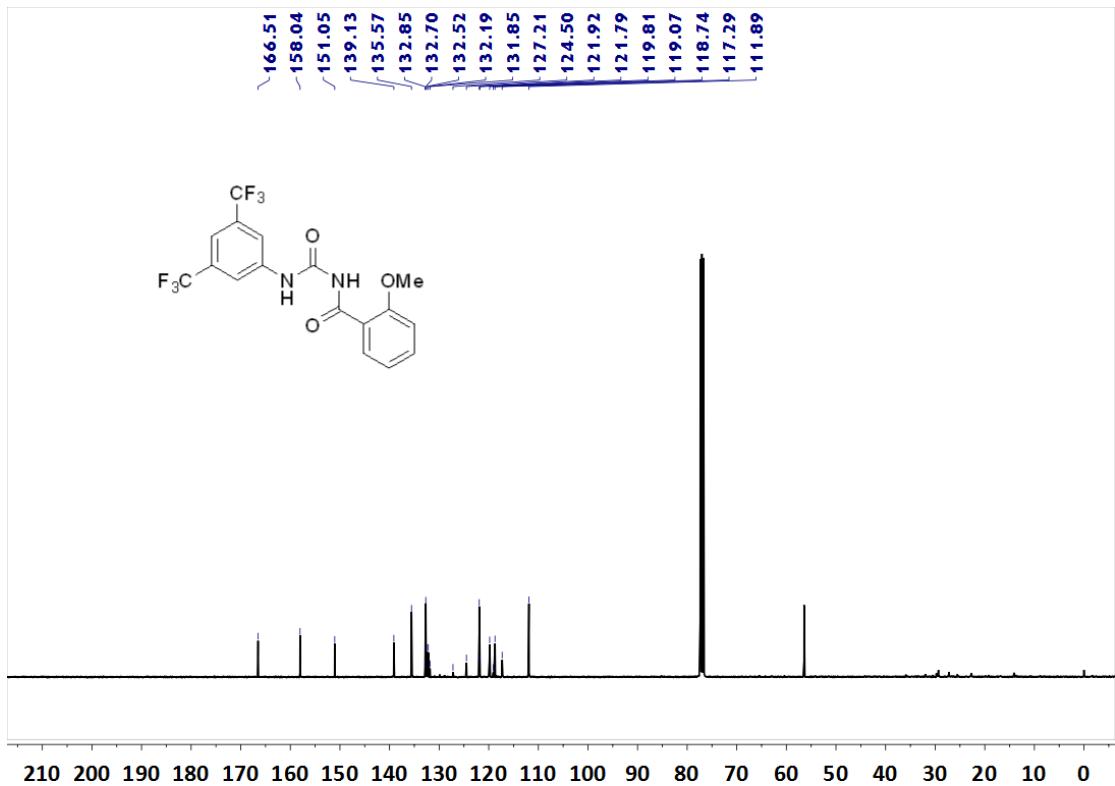
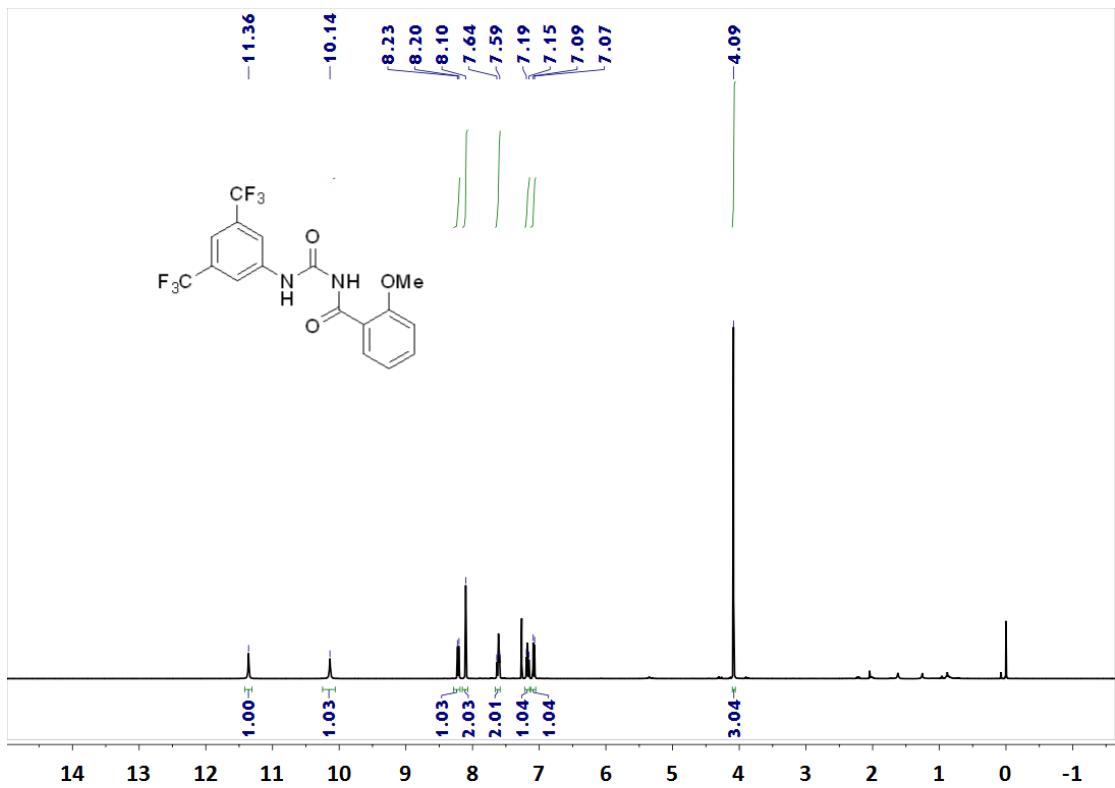


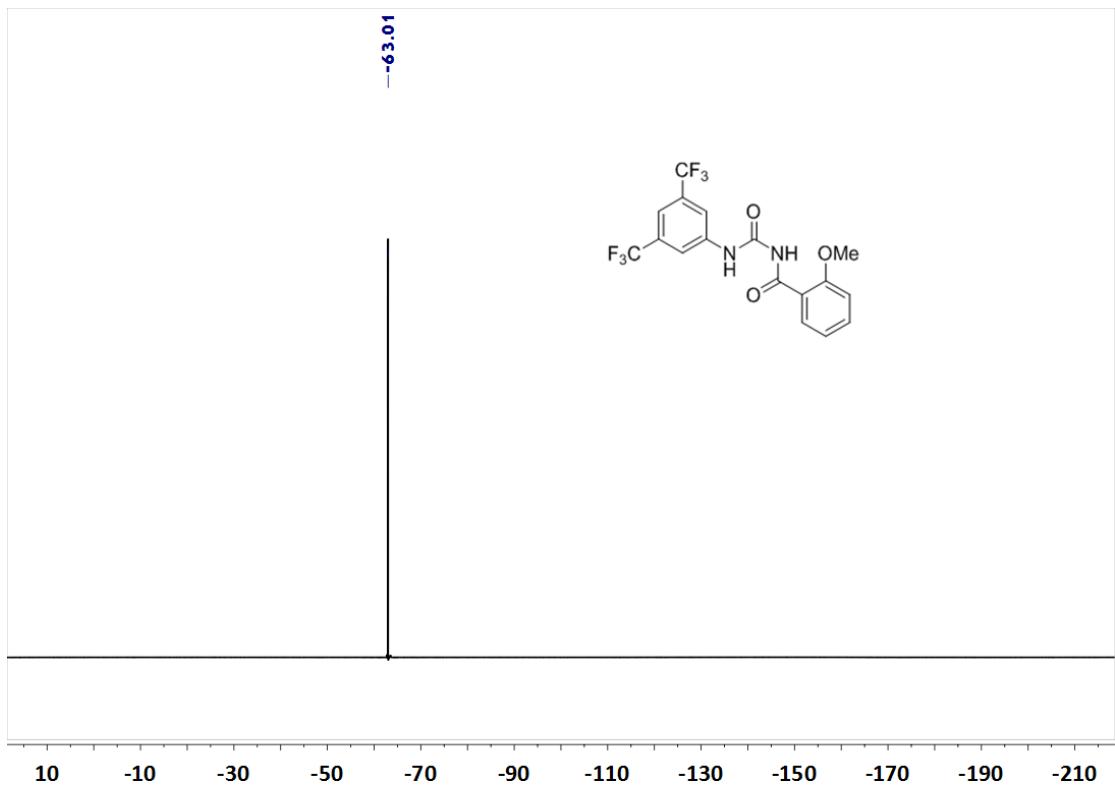
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6k**.



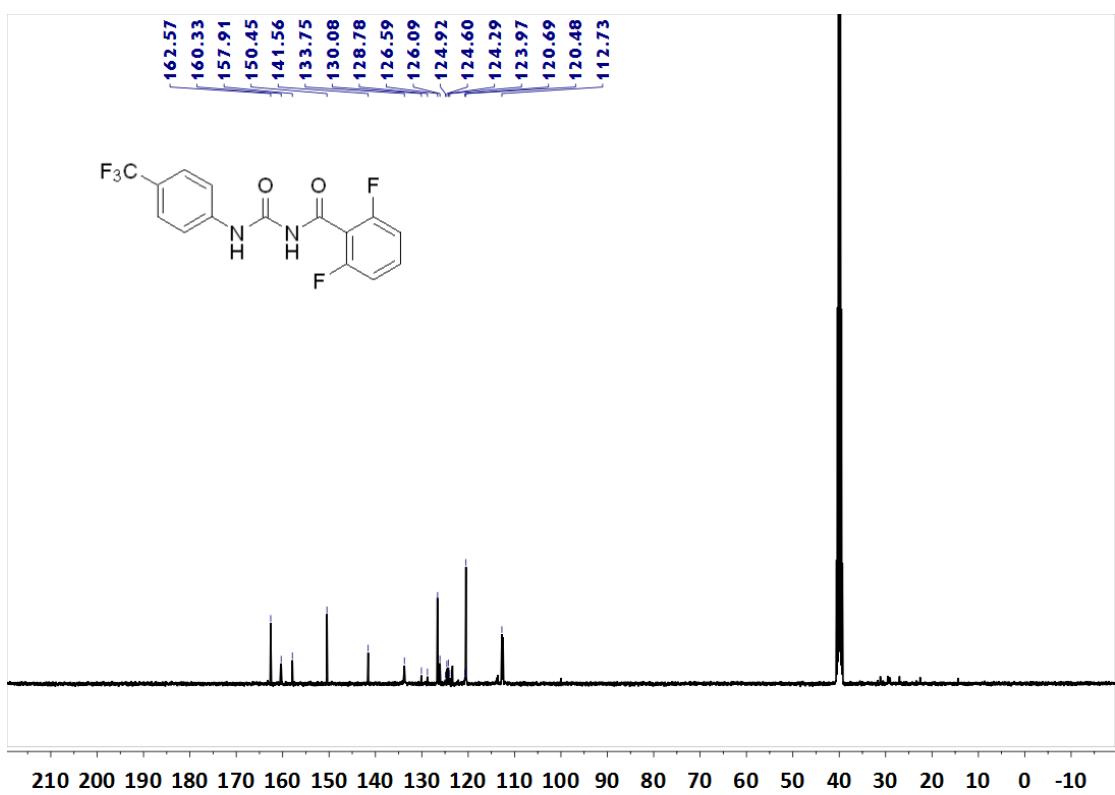


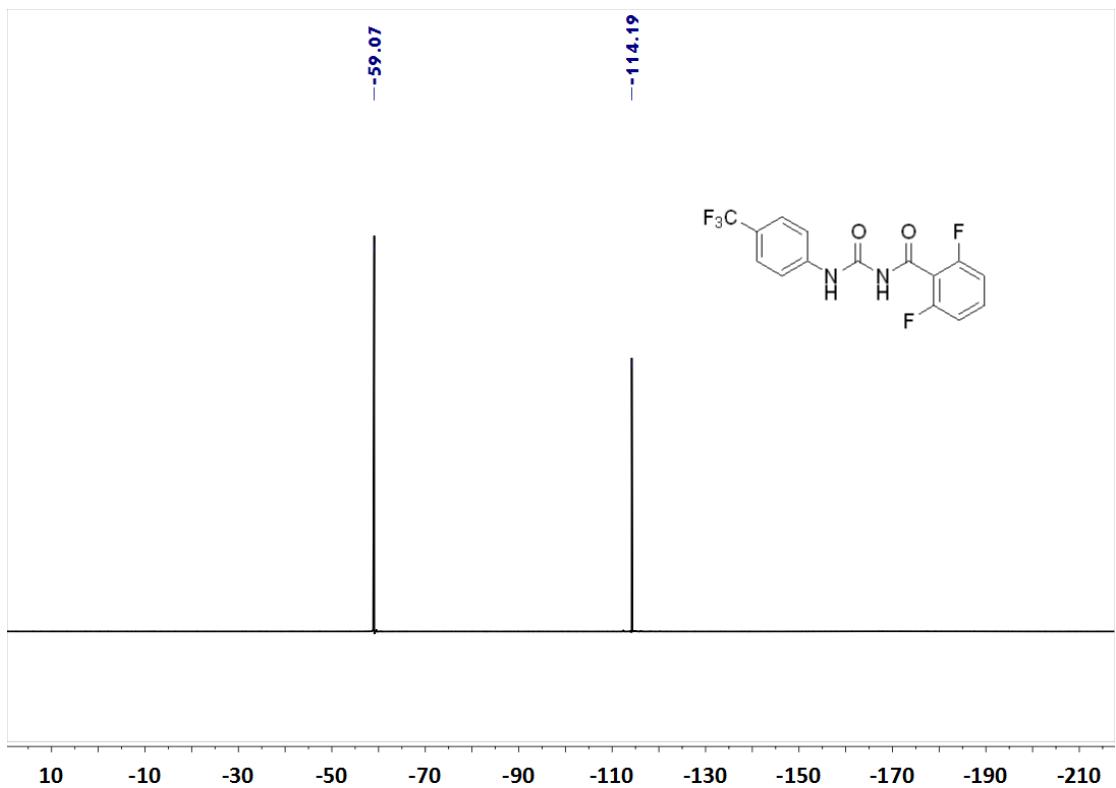
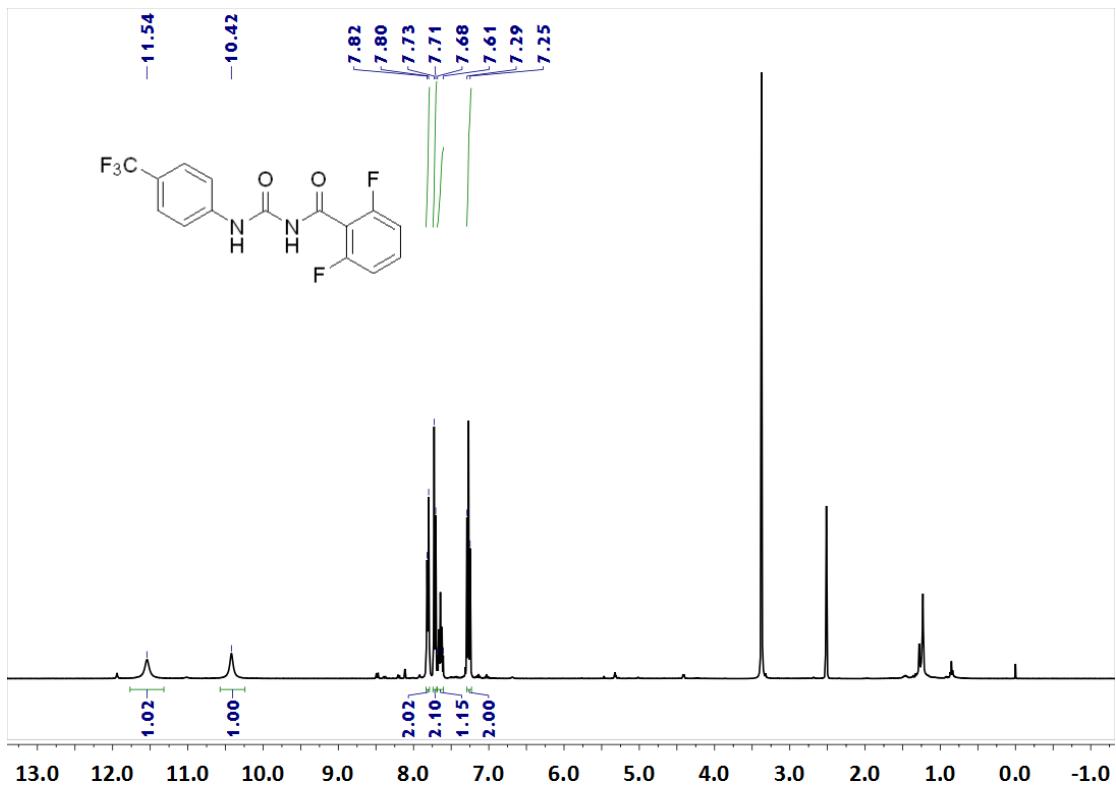
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6l**.



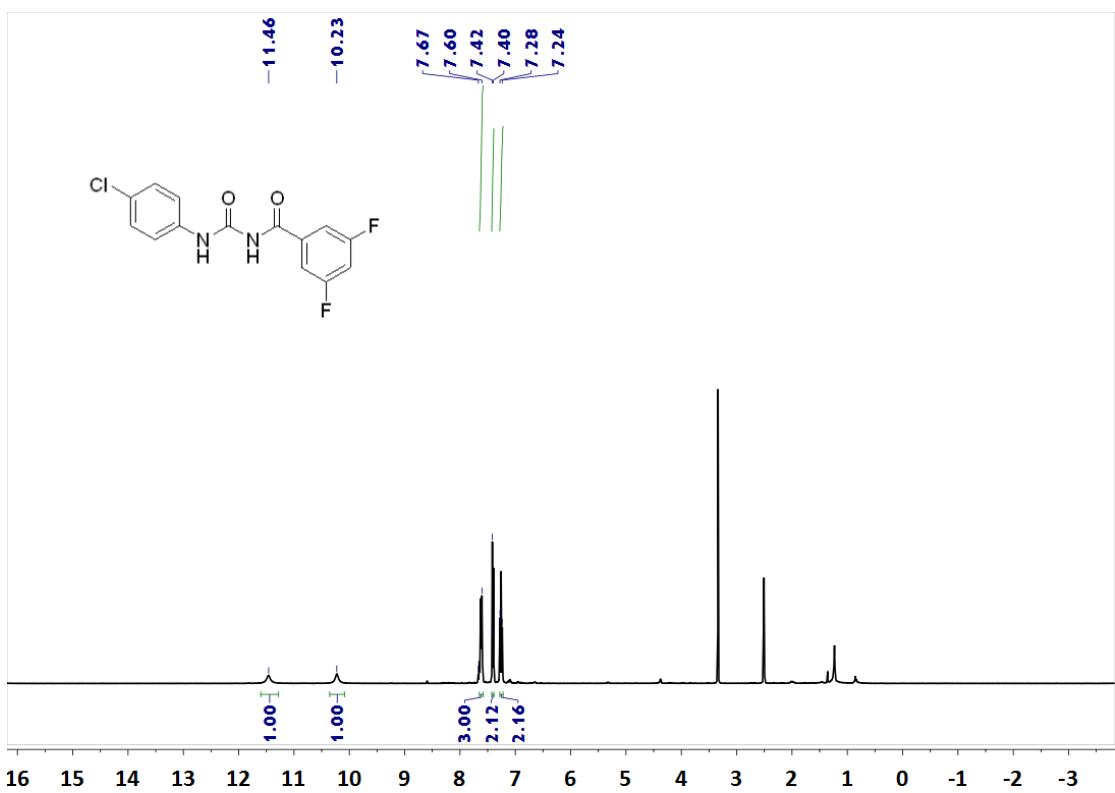
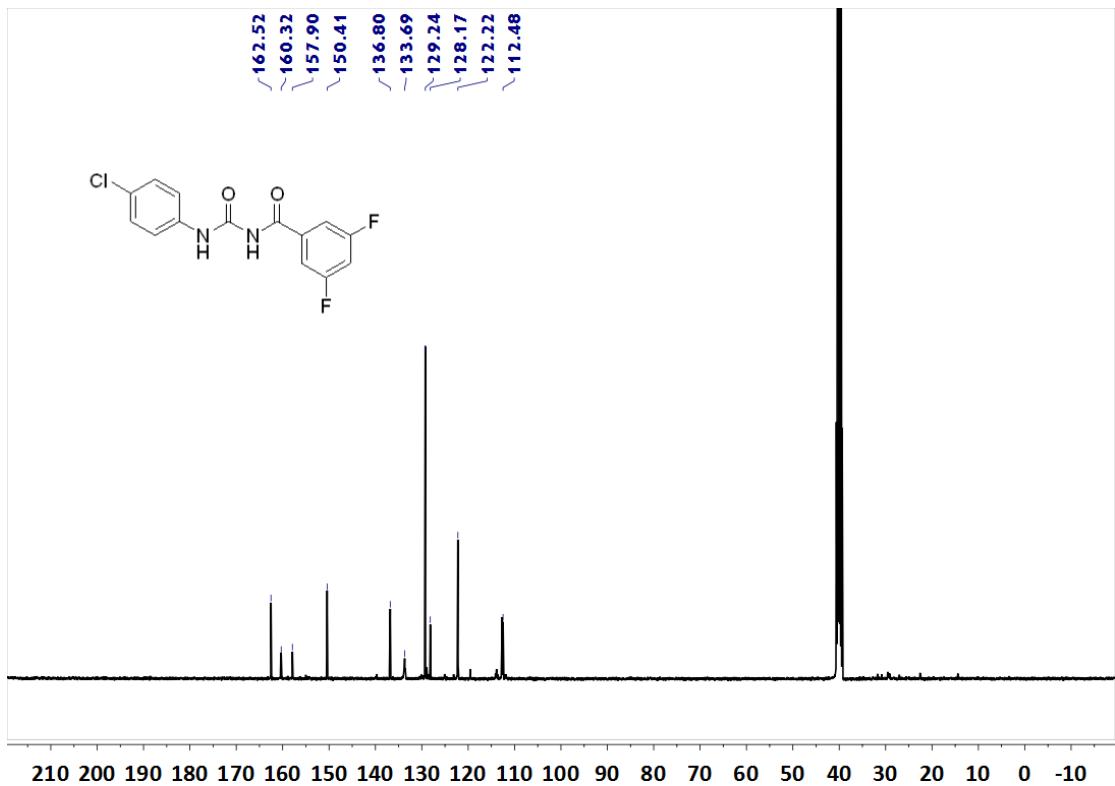


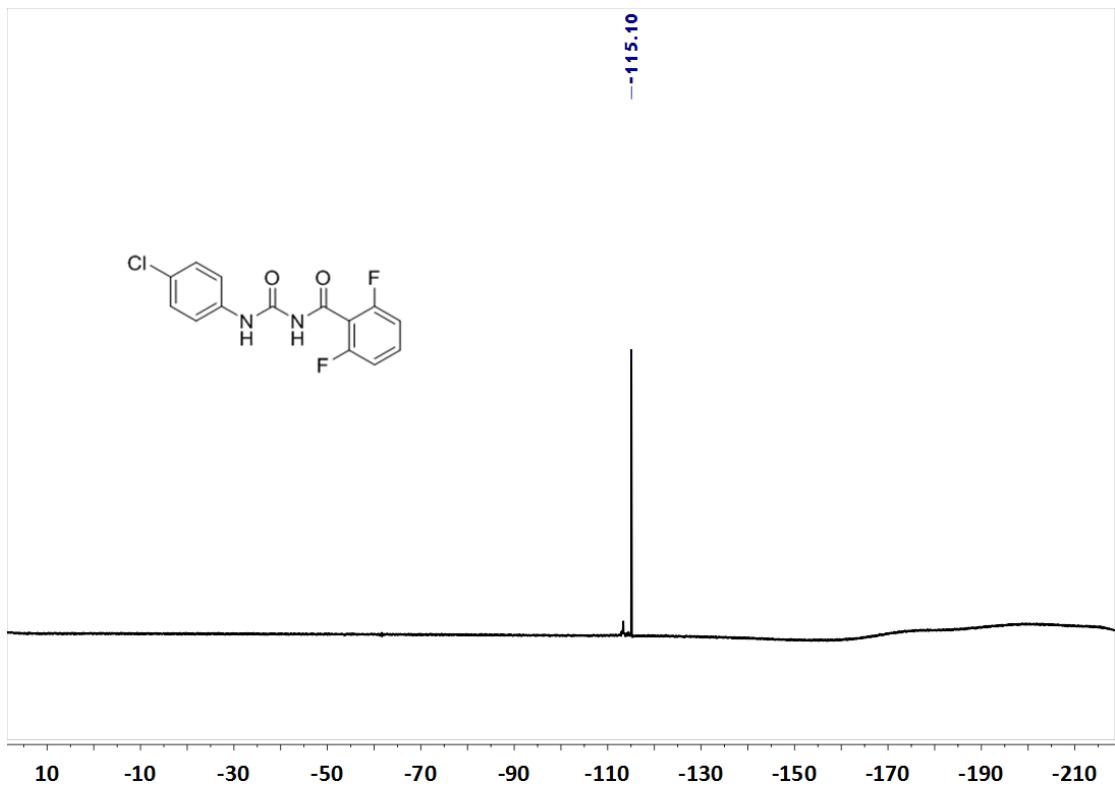
$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **6m**.



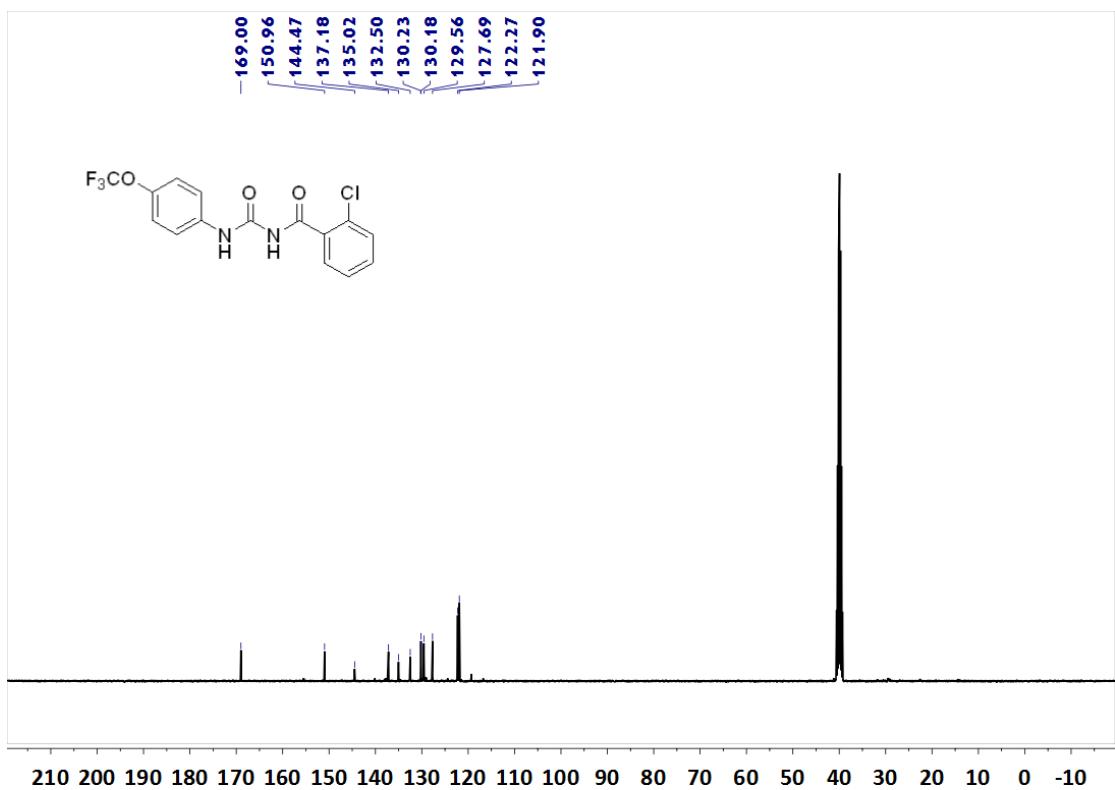


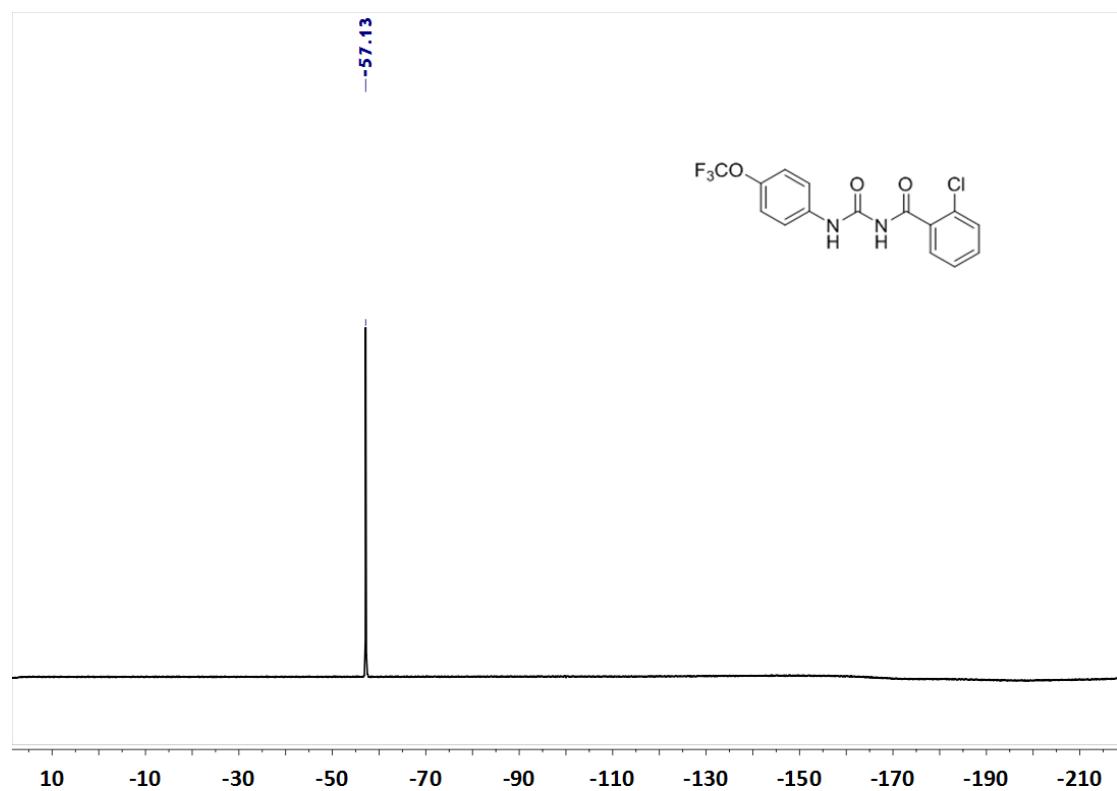
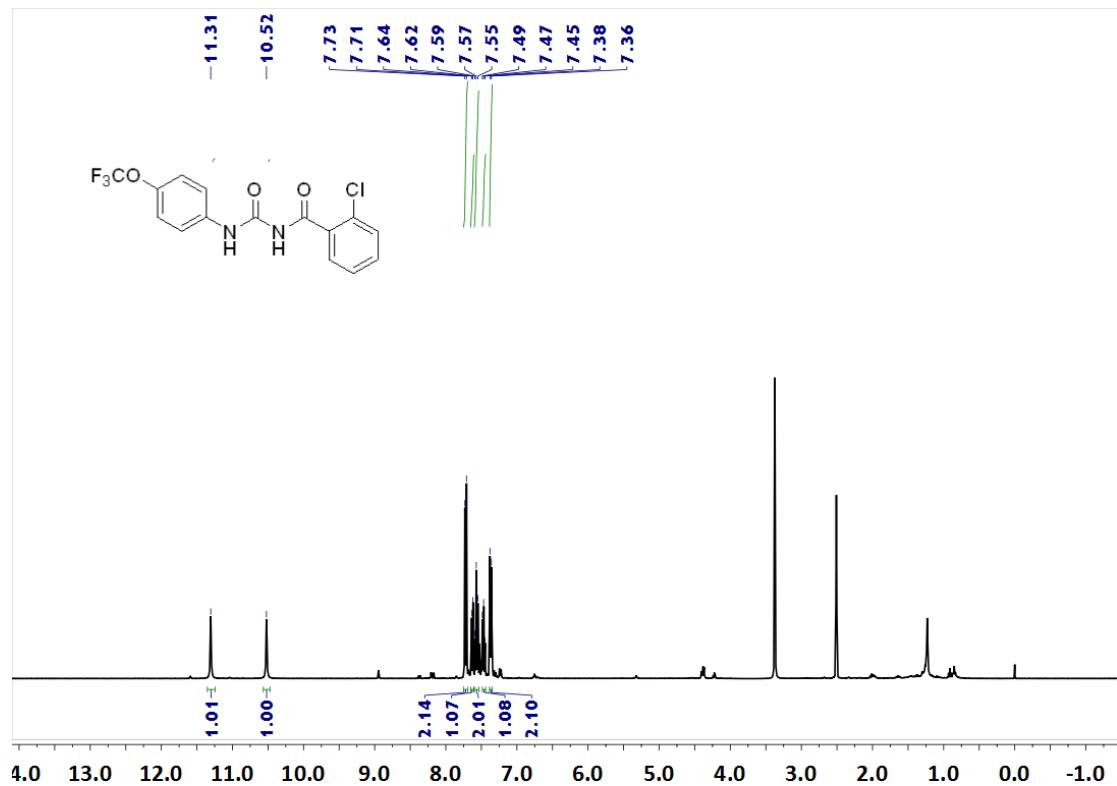
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6n**.



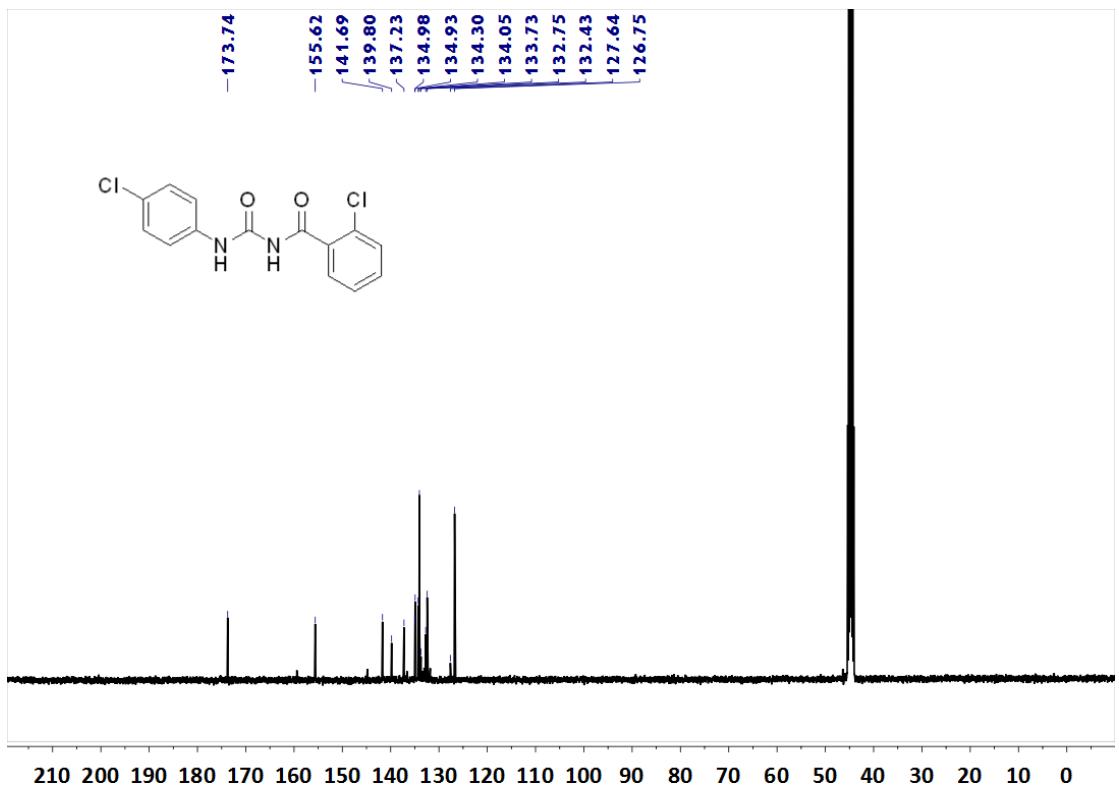
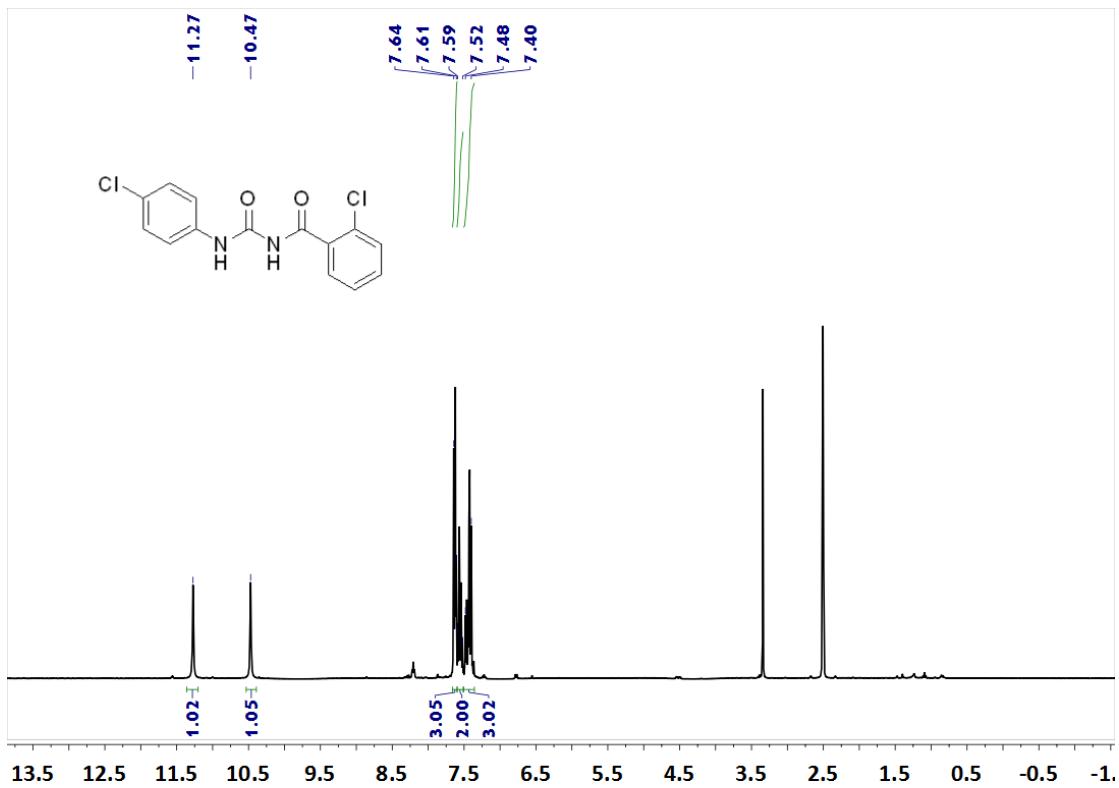


<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6o**.

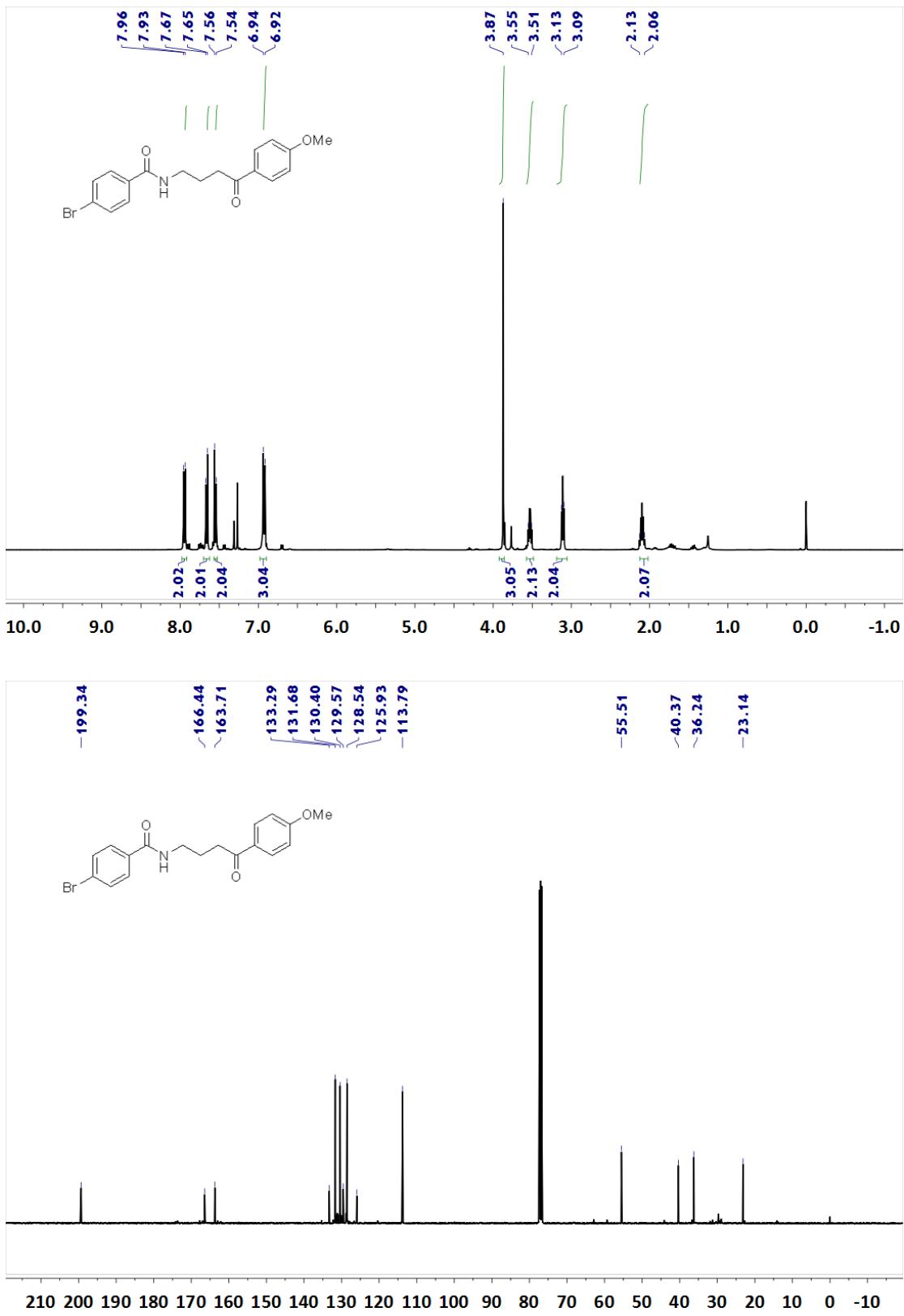




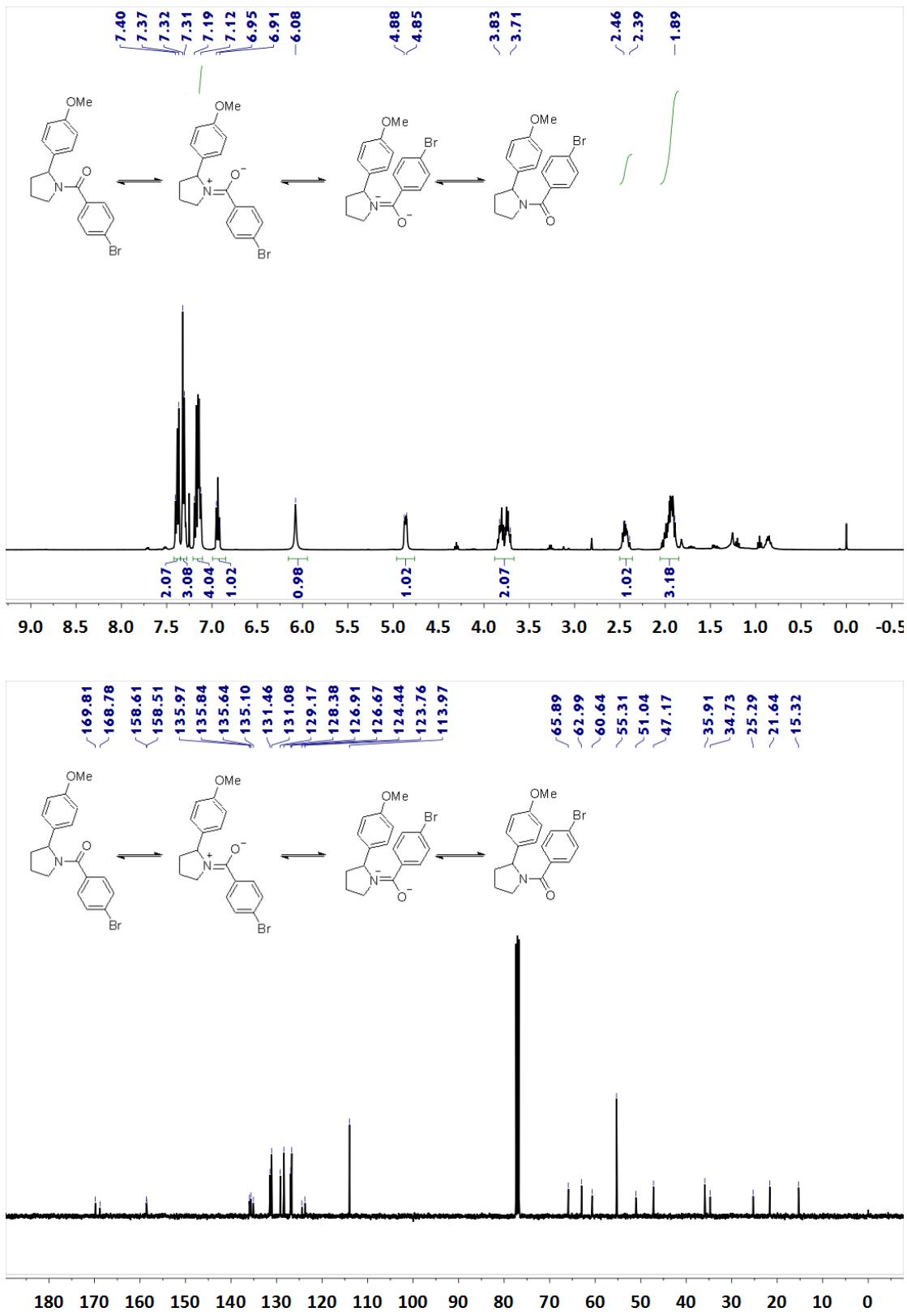
<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **6p**.



$^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of **2bb**.



<sup>1</sup>H and <sup>13</sup>C-NMR spectra of **1bb**.



<sup>1</sup>H and <sup>13</sup>C-NMR spectra of 1-(4-bromophenyl)-3-(4-hydroxy-4-phenylbutyl)urea.

