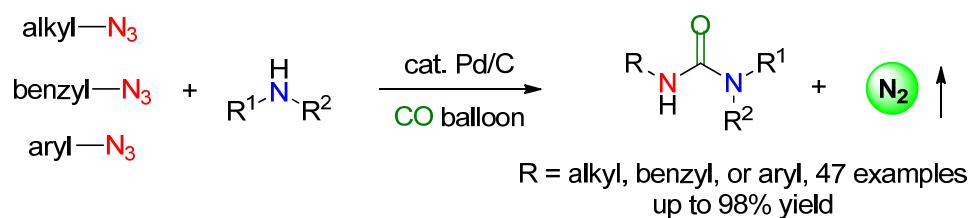


Pd/C Catalyzed Carbonylation of Azides in the Presence of Amines.

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1. Materials and methods.

Unless stated otherwise, all reactions were performed under a nitrogen atmosphere in a Schlenk reaction flask. Aromatic amines, alkyl bromides, alkyl alcohols and XPhos were obtained from Energy Chemicals. Pd/C (10 wt%, matrix activated carbon support) was obtained from Sigma-Aldrich. All solvents were distilled under a nitrogen atmosphere prior to use. 1,4-Dioxane and toluene were dried over Na with benzophenone-ketyl intermediate as indicator. MeCN and DCE were dried over CaH₂. For chromatography, 200-300 mesh silica gel (Qingdao, China) was employed. ¹H and ¹³C NMR spectra were recorded at 400 MHz and 100 MHz with Varian Mercury 400 spectrometer, or 300 MHz and 75 MHz with Bruker ARX 300 at ambient temperature. Chemical shifts are reported in ppm using tetramethylsilane as internal standard when CDCl₃ was used as solvent. IR spectra were recorded with a Thermo Electron Corporation Nicolet AVATAR 330 FT-IR spectrometer. HRMS data were obtained on a VG ZAB-HS mass spectrometer, Brucker Apex IV FTMS spectrometer. HPLC (Agilent Technologies 1200 Series) was equipped with an Eclipse Plus C18 5µm separation column (250*4.6 mm). The HRTEM method was used a JEM-2100F. This machine was operated at 200 kV, and it was equipped with a field emission gun, ultra-high-resolution pole piece, and ultrathin window JEOL detector. HRTEM images were obtained with an OSIS CANTEGA CCD camera.

2. Syntheses of azides substrates

A. Syntheses of aromatic azides substrates

To a 100 mL round bottom flask was charged with aniline (930 mg, 10 mmol) and water (15 mL). Concentrated HCl (4 mL) was added to the vigorously stirred reaction mixture in an ice-water bath. After stirring and cooling to 0 °C for 20-30 min, a freshly prepared, ice cold solution of NaNO₂ (690 mg, 10 mmol) in water (3 mL) was added dropwise to the reaction mixture while keeping the internal temperature between 0-5 °C. After addition of NaNO₂, the reaction mixture was stirred for an additional 10 min. A freshly prepared solution of sodium azide (780 mg, 12 mmol) in water (5 mL) was added drop wise to the reaction mixture via additional funnel while maintaining the internal temperature of the reaction mixture below 5 °C. Upon complete addition of the sodium azide solution, the reaction mixture was stirred for an additional 20-30 min at 0 °C, followed by stirring at rt for another 3 h. The reaction mixture was extracted with CH₂Cl₂ (2×50 mL), and combined organic layers was dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude azidobenzene was further purified by flash column chromatography over a short plug of silica gel using petroleum ether /EtOAc (15:1) as eluent to afford a yellow liquid (910 mg, 76%).

B. Syntheses of alkyl azides substrates

To a stirred solution of benzyl bromide (171 mg, 1 mmol) in 50 mL of 1:1 H₂O/Me₂CO (10 mL) was added NaN₃ (195 mg, 3 mmol). After 24 h, the reaction was extracted with EtOAc (3×20 mL), washed with brine, dried over MgSO₄, and concentrated under reduced pressure to give benzene azide as pale yellow oil. The crude product was further purified by flash column chromatography over a short plug of silica gel using petroleum ether /EtOAc (15:1) as eluent to afford a yellow liquid (106 mg, 80%).

3. Typical experimental procedure of Pd/C-catalyzed carbonylation of azides in the presence of amines.

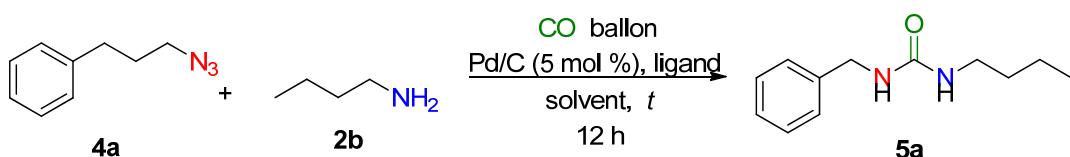
A. Typical experimental procedure of Pd/C catalyzed carbonylation of benzyl azides in the presence of amines.

The Schlenk reaction flask was degassed for two times with nitrogen, under a nitrogen atmosphere, Pd/C (21 mg, 0.02 mmol), XPhos (19 mg, 0.04 mmol) and 4-methoxyaniline (59 mg, 0.48 mmol) were added. Then the flask was sealed and evacuated to a vacuum of 15 mmHg and fitted a CO balloon. Benzyl azides (53 mg, 0.4 mmol) and PhMe (4 ml) were added *via* syringe. The mixture was stirred at 60 °C until **1a** disappeared as judged by TLC. Solution was removed in *vacuo* to leave a residue which was purified by flash silica gel chromatography using petroleum ether /EtOAc (from 3:1 to 1:1) as eluent afford pure **3a** as a white solid (94 mg, 91%).

B. Typical experimental procedure of Pd/C catalyzed carbonylation of alkyl azides in the presence of amines.

The Schlenk reaction flask was degassed for two times with nitrogen, under a nitrogen atmosphere, Pd/C (21 mg, 0.02 mmol) and XPhos (19 mg, 0.04 mmol) were added. Then the flask was sealed and evacuated to a vacuum of 15 mmHg and fitted a CO balloon. (3-azidopropyl)benzene (78 mg, 0.48 mmol), and PhMe (4 ml) were added *via* syringe. The mixture was stirred at 60 °C for 4 h, until **4a** disappeared as judged by TLC. Then n-butylamine (30 mg, 0.4 mmol) was added *via* syringe. The system was stirred at 60 °C for 8 h. Solution was removed in *vacuo* to leave a residue which was purified by flash silica gel chromatography using petroleum ether /EtOAc (from 5:1 to 1:1) to afford pure **5a** as pale yellow oil (90 mg, 96%).

Table S1. Reaction Optimization for Pd/C Catalyzed Carbonylation of Alkyl Azides in the Presence of Amines.^a



entry	ligand (mol%)	solvent	addtive	<i>t</i> (°C)	yield (%) ^b
1	XPhos (10)	dioxane	/	80	15
2	XPhos (10)	DCE	/	80	10
3	XPhos (10)	PhMe	/	80	46
4	DPPP (5)	PhMe	/	80	12
5	DPPF (5)	PhMe	/	80	11

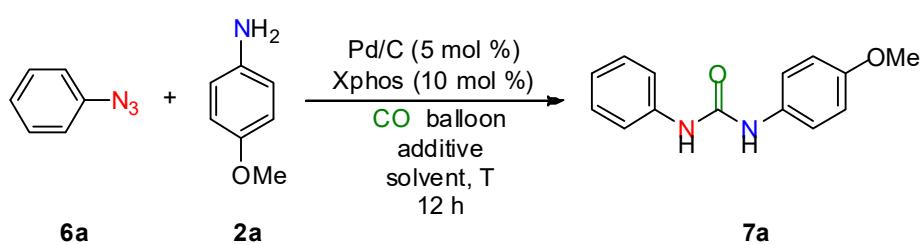
6	XPhos (10)	PhMe	Et ₃ N (1.5 eq.)	80	60
7	XPhos (10)	PhMe	PhOH (1.5 eq.)	80	50
8	XPhos (10)	PhMe	/	60	62
9	P(t-Bu) ₃ HBF ₄ (10)	PhMe	/	60	trace
10	XPhos (10)	PhMe	MgO (2 eq.)	60	64 ^c
11	XPhos (20)	PhMe	/	60	61 ^d
12	XPhos (10)	PhMe	MgO (2 eq.)	60	79 ^{c,e}
13	XPhos (10)	PhMe	/	60	96^{e,f}

^a Reaction was carried out with 1.0 equiv of **4a** and 1.2 equiv of **2b**. ^b Isolated yield. ^c **2b** was added after 12h. ^d The reaction was carried out with 10 mol% Pd/C. ^e The reaction was carried out with 1.2 equiv of **4a** and 1.0 equiv of **2b**. ^f **2b** was added after 4h.

C. Typical experimental procedure of Pd/C catalyzed carbonylation of aryl azides in the presence of amines.

The Schlenk reaction flask was degassed for two times with nitrogen, under a nitrogen atmosphere, Pd/C (21 mg, 0.02 mmol), XPhos (19 mg, 0.04 mmol) and 4-methoxyaniline (59 mg, 0.48 mmol) were added. Then the flask was sealed and evacuated to a vacuum of 15 mmHg and fitted a CO balloon. Azidobenzene (48 mg, 0.4 mmol) and water (with 5% PhMe and 5% ⁿBu₄NCl) were added *via* syringe. The mixture was stirred at room temperature until **6a** disappeared as judged by TLC. Solution was removed in *vacuo* to leave a residue which was purified by flash silica gel chromatography using petroleum ether /EtOAc (from 5:1 to 1:2) to afford pure **7a** as a white solid (94 mg, 97%).

Table S3. Reaction Optimization for Pd/C Catalyzed Carbonylation of Aryl Azides in the Presence of Amines.^a

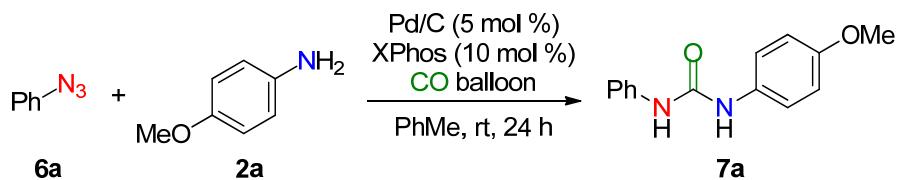


entry	Additive	Solvent	t (°C)	Yield ^b
1	/	PhMe	25	90%
2	/	H ₂ O ^b	25	65%
3	5% PTC (nBu₄NCl)	H₂O^c	25	97%

^a Reaction was carried out with 1.0 equiv of **6a** and 1.2 equiv of **2a**. ^b Isolated yield. ^c The solvent was added 5% PhMe.

4. Additional mechanism Study

a) Controlled experiment^a



entry	catalyst	yield (%)
1	A	10
2	B	22
3	C	89
4	D	90

^a Reaction conditions: **6a** (0.4 mmol), **2a** (0.48 mmol), catalyst and PhMe (4 ml) was added and reacted for 12h under CO (1 atm).

Catalyst A: Pd/C (0.02 mmol) and XPhos (0.04 mmol) were incubated in PhMe (4 mL) overnight at 30°C, then filtrated, and washed with acetone (3×5 mL), the solution was removed in *vacuum* to leave a residue as a white solid, which was confirmed mainly to be XPhos. This white solid was used as catalyst A.

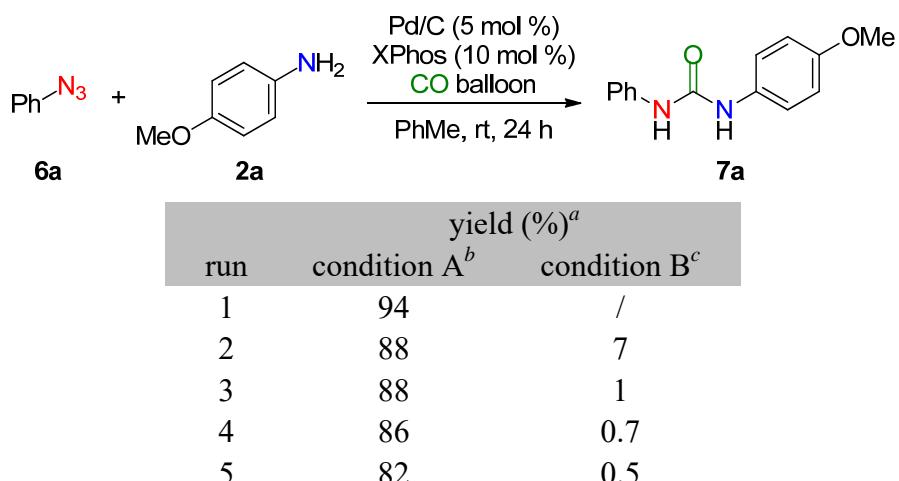
Catalyst B: Pd/C (0.02 mmol) and XPhos (0.04 mmol) were incubated in PhMe (4 mL) overnight at 30°C, then filtrated, washed with acetone (3×5 mL), the black residue solid was taken in *vacuum* to further remove the solvent. This black solid was used as catalyst B.

Catalyst C: Additional fresh XPhos (0.04 mmol) was added to catalyst B.

Catalyst D: Fresh Pd/C (0.02 mmol) and fresh XPhos (0.04 mmol) were used as catalyst D.

b) Recycling test

The recycling test of Pd/C with/without extra XPhos confirmed the consequence. Yields were obtained through HPLC.



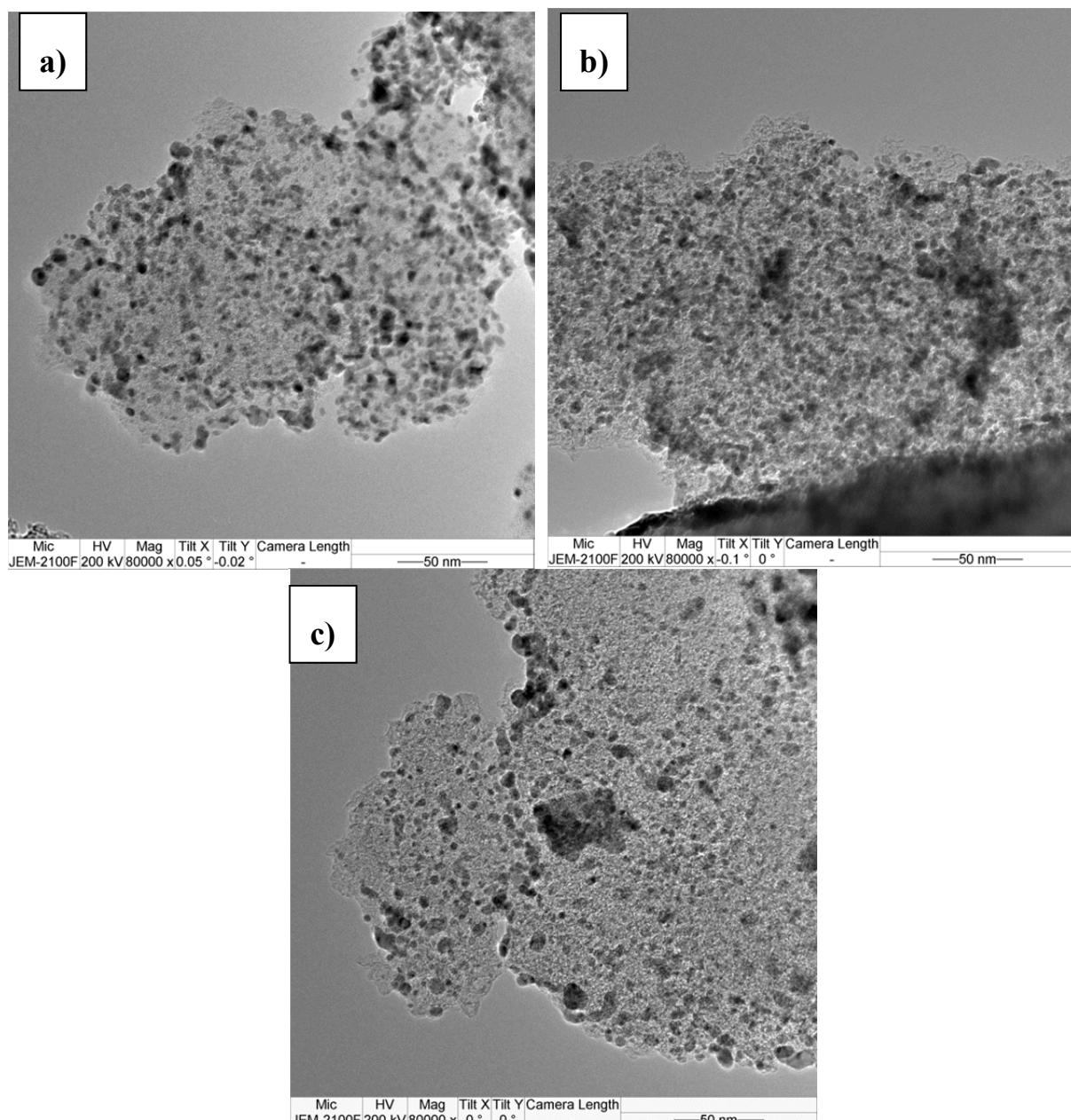
^a Yield was determined by HPLC analysis. ^b Reaction condition A: **6a** (0.4 mmol), **2a** (0.48 mmol) and PhMe (4 ml) was added and reacted for 12h under CO (1 atm). Reaction products carried out with the same batch of Pd/C with fresh XPhos (0.04 mmol). ^c Reaction condition B: **6a** (0.4 mmol), **2a** (0.48 mmol) and PhMe (4 ml) was added and

reacted for 12h under CO (1 atm). Reaction products carried out with the same batch of Pd/C. Besides, no addition XPhos was added in each run.

Controlled experiments suggested that the palladium that on the carbon was the real catalyst, even some palladium leached into the solvent. In addition, XPhos was necessary for this catalytic reaction, and it may coordinate to the palladium that on the carbon,

c) HRTEM experiment

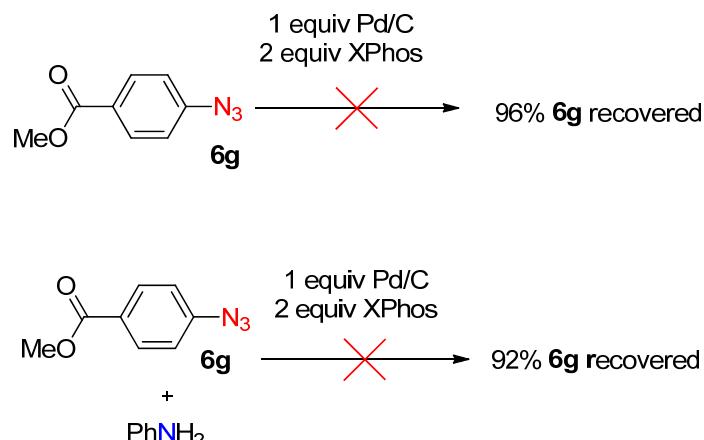
The Pd/C-XPhos systems were analyzed by the HRTEM method using a JEM-2100F. This machine was operated at 200 kV, and it was equipped with a field emission gun, ultra-high-resolution pole piece, and ultrathin window JEOL detector. HRTEM images were obtained with an OSIS CANTEGA CCD camera.



HRTEM of the Pd/C-XPhos systems. a) Fresh Pd/C. b) Pd/C incubated with XPhos in PhMe, then washed with PhMe and acetone. c) Recovered Pd/C after one time catalyzed reaction.

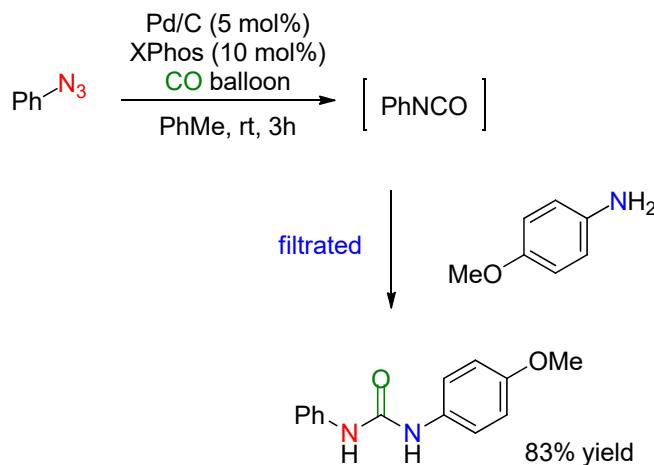
Reflected by HRTEM investigations, all the fresh Pd/C (a), filtrated Pd/C after incubated with XPhos (b) and recovered Pd/C (c), clearly exhibited higher Pd dispersion and more uniform distribution, no agglomeration of Pd crystallites was observed. This result is different with other heterogeneous catalyzed coupling reaction. (See reference: Heidenreich, R.-G.; Krauter, J. G. E.; Pietsch, J.; Köheler, K. *J. Mol. Catal. A: Chem.* **2002**, 182-183, 499.)

d) Stoichiometric reaction and sub step reaction



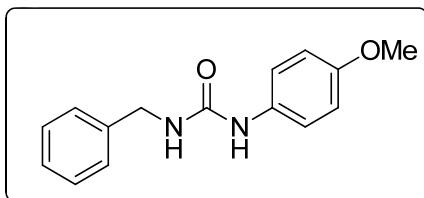
Reaction was carried out with 1.0 equiv of **1a** (0.4 mmol) and 1.2 equiv of **2a** (0.48 mmol).

Above results showed that the coordination of CO to the palladium catalyst was also critical to the decomposing of azides. When CO was removed, >90% azides could be recovered after 24h even using the stoichiometric of Pd/C.



5. Spectral data for the ureas

1-benzyl-3-(4-methoxyphenyl)urea (**3a**)¹

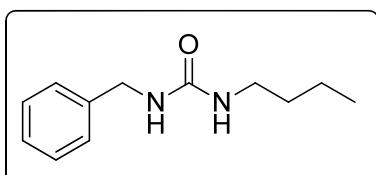


Yellow solid. (94mg, 91%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.35 (s, 1H), 7.38-7.19 (m, 7H), 6.82 (d, *J* = 8.9 Hz, 2H), 6.51 (t, *J* = 5.8 Hz, 1H), 4.29 (d, *J* = 5.8 Hz, 2H), 3.69 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 155.48, 153.97, 140.53, 133.60, 128.31, 127.12, 126.70, 119.48, 113.89, 55.14, 42.79.

1-benzyl-3-butylurea (**3b**)²

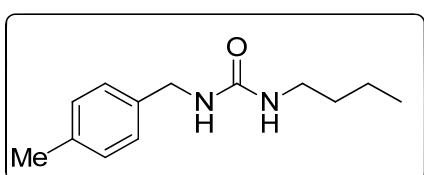


White solid. (81mg, 98%)

¹H NMR (400 MHz, CDCl₃) δ 7.30-7.24 (m, 2H), 7.24-7.17 (m, 3H), 5.38 (s, 1H), 5.04 (s, 1H), 4.25 (d, *J* = 5.8 Hz, 2H), 3.06 (d, *J* = 6.2 Hz, 2H), 1.37 (dd, *J* = 14.9, 7.2 Hz, 2H), 1.26 (dd, *J* = 14.9, 7.2 Hz, 2H), 0.86 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 158.85, 139.63, 128.61, 127.38, 127.19, 44.35, 40.25, 32.42, 20.12, 13.92.

1-butyl-3-(4-methylbenzyl)urea (**3c**)³

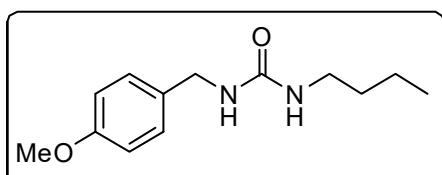


White solid. (68mg, 77%)

¹H NMR (400MHz, CDCl₃) δ 7.07-7.02 (m, 4H), 5.61 (s, 1H), 5.31 (s, 1H), 4.18-4.14 (m, 2H), 3.03-2.98 (m, 2H), 2.27(s, 3H), 1.36-1.19 (m, 4H), 0.84(t, *J* = 7.2Hz, 3H).

¹³C NMR (100MHz, CDCl₃) δ 159.09, 136.65, 136.57, 129.18, 127.31, 43.98, 40.10, 32.43, 21.12, 20.10, 13.89.

1-butyl-3-(4-methoxybenzyl)urea (**3d**)⁴

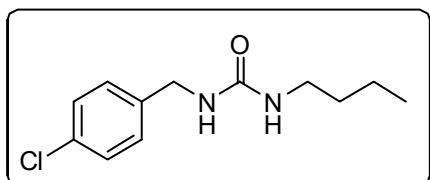


White solid. (69mg, 73%)

¹H NMR (400MHz, DMSO-d⁶) δ 7.19-7.12 (m, 2H), 6.89-6.84 (m, 2H), 6.15 (t, *J* = 6.0 Hz, 1 H), 5.83 (t, *J* = 5.6 Hz, 1H), 4.11 (d, *J* = 6.0 Hz, 2H), 3.72 (s, 3H), 3.01-2.96 (m, 2H), 1.36-1.29 (m, 2H), 1.29-1.22 (m, 2H), 0.86 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (100MHz, DMSO-d⁶) δ 158.02, 158.00, 132.90, 128.30, 113.59, 55.04, 42.35, 38.96, 32.18, 19.53, 13.73.

1-butyl-3-(4-chlorobenzyl)urea (**3e**) ⁵

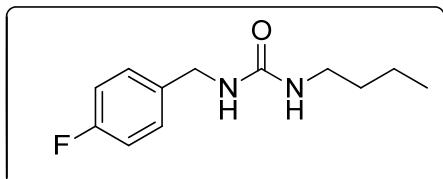


White solid. (68mg, 71%)

¹H NMR (400 MHz, DMSO-d⁶) δ 7.36 (dd, *J* = 8.3, 1.9 Hz, 2H), 7.25 (d, *J* = 8.3 Hz, 2H), 6.31 (t, *J* = 5.9 Hz, 1H), 5.94 (t, *J* = 5.6 Hz, 1H), 4.17 (d, *J* = 6.1 Hz, 2H), 3.01-2.97 (m, 2H), 1.39-1.31 (m, 2H), 1.26 (dd, *J* = 15.0, 7.2 Hz, 2H), 0.86 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 158.04, 140.26, 130.98, 128.82, 128.11, 42.21, 39.03, 32.17, 19.56, 13.75.

1-butyl-3-(4-fluorobenzyl)urea (**3f**)



White solid. (80mg, 89%)

¹H NMR (400 MHz, CDCl₃) δ 7.20-7.14 (m, 2H), 7.00- 6.89 (m, 2H), 5.21 (s, 1H), 4.87 (s, 1H), 4.25-4.21 (m, 2H), 3.08-3.05 (m, 2H), 1.42-1.32 (m, 2H), 1.31- 1.22 (m, 2H), 0.87 (t, *J* = 7.2 Hz, 3H).

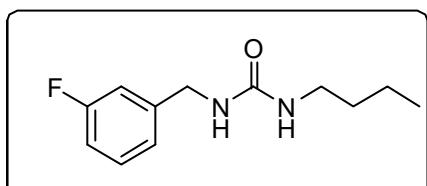
¹³C NMR (100 MHz, CDCl₃) δ 162.09 (d, *J* = 242.0 Hz), 158.64, 135.40, 129.01 (d, *J* = 8.2 Hz), 115.44 (d, *J* = 21.4 Hz), 43.67, 40.33, 32.41, 20.13, 13.89.

¹⁹F NMR (376 MHz, CDCl₃) δ -115.6.

IR (neat) ν: 2959, 2929, 1624, 1587, 1511, 1228, 1157, 825 cm⁻¹.

HRMS (ESI) calcd for C₁₂H₁₈FN₂O: 225.13977, found: 225.13973

1-butyl-3-(3-fluorobenzyl)urea (**3g**)



White solid. (91mg, >98%)

¹H NMR (400 MHz, CDCl₃) δ 7.26-7.22 (m, 1H), 7.01 (d, *J* = 7.6 Hz, 1H), 6.97-6.89 (m, 2H), 5.20 (s, 1H), 4.82 (s, 1H), 4.31 (d, *J* = 5.8 Hz, 2H), 3.15-3.10 (m, 2H), 1.48-1.37 (m, 2H), 1.34-1.25 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H).

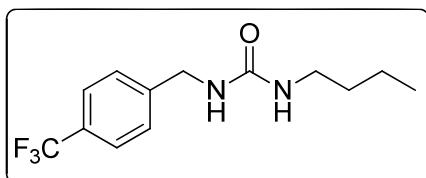
¹³C NMR (100 MHz, CDCl₃) δ 162.90 (d, *J* = 244.3 Hz), 159.21, 142.64 (d, *J* = 6.8 Hz), 129.80 (d, *J* = 8.2 Hz), 122.28 (d, *J* = 2.6 Hz), 113.71 (d, *J* = 3.0 Hz), 113.50 (d, *J* = 3.4 Hz), 43.25, 39.94, 32.29, 19.97, 13.72.

¹⁹F NMR (376 MHz, CDCl₃) δ -113.1 (dd, *J* = 15.2, 9.0 Hz).

IR (neat) v: 2957, 2934, 1626, 1585, 1256, 1065, 815 cm⁻¹.

HRMS (ESI) calcd for C₁₂H₁₈FN₂O: 225.13977, found: 225.14041.

1-butyl-3-(4-(trifluoromethyl)benzyl)urea (**3h**)



White solid. (109mg, >98%)

¹H NMR (400 MHz, CDCl₃) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.33-7.31 (m, 2H), 5.70 (s, 1H), 5.24 (s, 1H), 4.28 (d, *J* = 6.0 Hz, 2H), 3.09-3.04 (m, 2H), 1.41-1.33 (m, 2H), 1.28-1.23 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H).

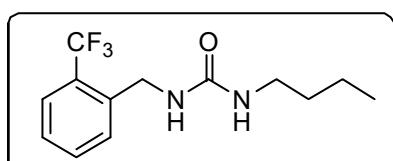
¹³C NMR (100 MHz, CDCl₃) δ 158.56, 143.89, 129.55 (q, *J* = 32.2 Hz), 127.46, 125.58 (q, *J* = 3.8 Hz), 124.21 (q, *J* = 263.8 Hz), 43.86, 40.41, 32.40, 20.12, 13.86.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.5.

IR (neat) v: 3334, 2964, 2930, 1629, 1581, 1329, 1125 cm⁻¹.

HRMS (ESI) calcd for C₁₃H₁₈F₃N₂O: 275.13657, found: 275.13731.

1-butyl-3-(2-(trifluoromethyl)benzyl)urea (**3i**)



White solid. (95mg, 87%)

¹H NMR (400 MHz, CDCl₃) δ 7.63-7.60 (m, 2H), 7.53-7.49 (m, 1H), 7.37-7.33 (m, 1H), 4.80 (s, 1H), 4.55 (d, *J* = 6.1 Hz, 2H), 4.43 (s, 1H), 3.17-3.12 (m, 2H), 1.48-1.41 (m, 2H), 1.33-1.27 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H).

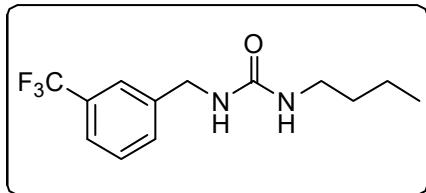
¹³C NMR (100 MHz, CDCl₃) δ 158.05, 138.14, 132.43, 132.42, 130.46, 127.44, 126.03 (q, *J* = 5.7 Hz), 124.74 (q, *J* = 288.3 Hz), 41.03, 40.54, 32.36, 20.12, 13.88.

¹⁹F NMR (376 MHz, CDCl₃) δ -59.6.

IR (neat) v: 1630, 1594, 1317, 1271, 1160, 1117, 690 cm⁻¹.

HRMS (ESI) calcd for C₁₃H₁₈F₃N₂O: 275.13657, found: 275.13714.

1-butyl-3-(3-(trifluoromethyl)benzyl)urea (3j**)**



White solid. (98mg, 90%)

¹H NMR (400 MHz, CDCl₃) δ 7.53-7.35 (m, 4H), 5.11 (s, 1H), 4.71 (s, 1H), 4.38 (d, *J* = 6.0 Hz, 2H), 3.16-3.11 (m, 2H), 1.47-1.40 (m, 2H), 1.33-1.26 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H).

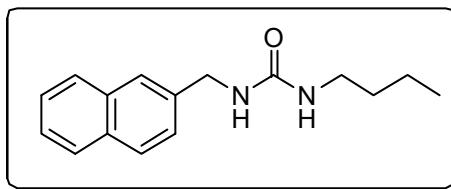
¹³C NMR (100 MHz, CDCl₃) δ 158.39, 140.80, 130.84, 130.82, 129.15, 125.40 (q, *J* = 269.8 Hz), 124.14 (q, *J* = 3.8 Hz), 123.99 (q, *J* = 3.8 Hz), 43.96, 40.45, 32.39, 20.12, 13.87.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.7.

IR (neat) ν : 1636, 1580, 1331, 1166, 1129, 703, 689 cm⁻¹.

HRMS (ESI) calcd for C₁₃H₁₈F₃N₂O: 275.13657, found: 275.13710.

1-butyl-3-(naphthalen-2-ylmethyl)urea (3k**)**



White solid. (64mg, 63%)

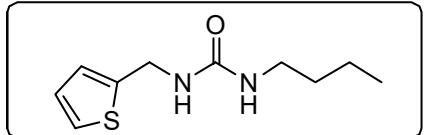
¹H NMR (400 MHz, CDCl₃) δ 7.99-7.93 (m, 1H), 7.82-7.78 (m, 1H), 7.73-7.71 (M, 1H), 7.50-7.42 (m, 2H), 7.35-7.31 (M, 2H), 5.01 (s, 1H), 4.73 (s, 1H), 4.67 (s, 2H), 3.04 (t, *J* = 7.0 Hz, 2H), 1.40-1.30 (m, 2H), 1.28-1.20 (m, 2H), 0.84 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 158.69, 134.73, 133.78, 131.35, 128.64, 128.03, 126.29, 125.78, 125.57, 125.39, 123.52, 42.24, 40.13, 32.37, 20.05, 13.85.

IR (neat) ν : 2959, 2928, 1626, 1581, 1252, 106, 775 cm⁻¹.

HRMS (ESI) calcd for C₁₆H₂₁N₂O: 257.16484, found: 257.16519.

1-butyl-3-(thiophen-2-ylmethyl)urea (3l**)**



White solid. (70mg, 85%)

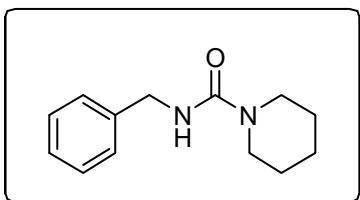
¹H NMR (400 MHz, CDCl₃) δ 7.21 (ddd, *J* = 4.9, 1.5, 0.5 Hz, 1H), 6.97-6.92 (m, 2H), 4.71 (s, 1H), 4.54 (d, *J* = 5.7 Hz, 2H), 4.39 (s, 1H), 3.19-3.14 (m, 2H), 1.50-1.43 (m, 2H), 1.37-1.30 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 157.69, 142.63, 126.97, 125.52, 125.08, 40.54, 39.64, 32.38, 20.14, 13.92.

IR (neat) ν: 3338, 2955, 1627, 1589, 689 cm⁻¹.

HRMS (ESI) calcd for C₁₀H₁₇N₂OS: 213.10561, found: 213.10571.

N-benzylpiperidine-1-carboxamide (**3m**) ⁶

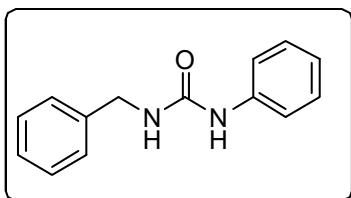


White solid. (74mg, 85%)

¹H NMR (400 MHz, CDCl₃) δ 7.35-7.23 (m, 5H), 4.77 (s, 1H), 4.42 (d, *J* = 5.5 Hz, 2H), 3.36-3.31 (m, 4H), 1.62-1.52 (m, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 157.69, 139.83, 128.67, 127.86, 127.30, 45.12, 45.07, 25.74, 24.53.

1-benzyl-3-phenylurea (**3n**) ⁷

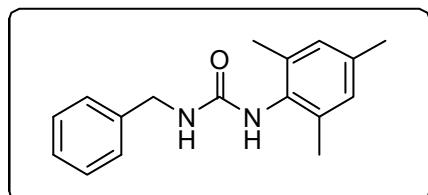


White solid. (88mg, 97%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.55 (s, 1H), 7.40 (d, *J* = 7.7 Hz, 2H), 7.36-7.28 (m, 4H), 7.26-7.20 (m, 3H), 6.91-6.87 (m, 1H), 6.61 (t, *J* = 5.9 Hz, 1H), 4.30 (d, *J* = 5.9 Hz, 2H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 155.25, 140.48, 140.38, 128.69, 128.34, 127.14, 126.75, 12.12, 117.70, 42.75.

1-benzyl-3-mesitylurea (**3o**)



White solid. (100mg, 93%)

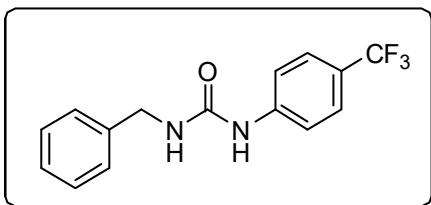
¹H NMR (400 MHz, DMSO-d⁶) δ 7.44 (s, 1H), 7.34-7.22 (m, 5H), 6.84 (s, 2H), 6.43 (s, 1H), 4.26-4.23 (m, 2H), 2.20 (s, 3H), 2.12 (s, 6H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 156.29, 141.04, 135.47, 133.32, 128.30, 128.21, 127.00, 12.86, 126.55, 42.95, 20.51, 18.20.

IR (neat) *v*: 3314, 3290, 1630, 1609, 1566, 1229, 667 cm⁻¹.

HRMS (ESI) calcd for C₁₇H₂₁N₂O: 269.16484, found: 269.16530.

benzyl-3-(4-(trifluoromethyl)phenyl)urea (3p)



White solid. (95mg, 81%)

^1H NMR (400 MHz, DMSO-d⁶) δ 9.02 (s, 1H), 7.62-7.55 (m, 4H), 7.38-7.29 (m, 4H), 7.28-7.16 (m, 1H), 6.79 (t, J = 5.9 Hz, 1H), 4.31 (d, J = 5.9 Hz, 2H).

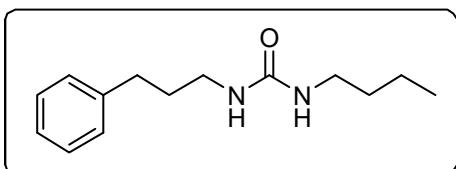
^{13}C -NMR (100 MHz, DMSO-d⁶) δ 154.87, 144.17, 140.04, 128.30, 127.14, 126.77, 125.94 (q, J = 3.9 Hz), 124.64 (q, J = 269.1 Hz), 121.04 (q, J = 31.7 Hz), 117.29, 42.76.

^{19}F NMR (376 MHz, DMSO-d⁶) δ -61.4.

IR (neat) ν : 2130, 1640, 1612, 1566, 1324, 1120 cm⁻¹.

HRMS (ESI) calcd for C₁₅H₁₄F₃N₂O: 295.10527, found: 295.10610.

1-butyl-3-(3-phenylpropyl)urea (5a)



White solid. (90mg, 96%)

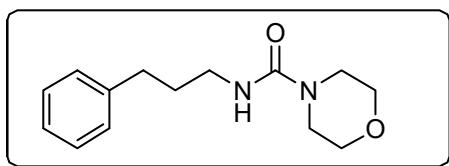
^1H NMR (400 MHz, CDCl₃) δ 7.27-7.25 (m, 2H), 7.18-7.13 (m, 3H), 5.11 (s, 1H), 5.02 (s, 1H), 3.20-3.15 (m, 2H), 3.13-3.08 (m, 2H), 2.62 (m, 2H), 1.83-1.75 (m, 2H), 1.45-1.39 (m, 2H), 1.35-1.28 (m, 2H), 0.89 (m, 3H).

^{13}C NMR (100 MHz, CDCl₃) δ 159.00, 141.78, 128.48, 128.43, 125.96, 40.22, 40.06, 33.32, 32.51, 32.08, 20.16, 13.92.

IR (neat) ν : 3333, 2930, 2861, 1630, 1573, 1259 cm⁻¹.

HRMS (ESI) calcd for C₁₄H₂₃N₂O: 235.18049, found: 235.18107.

N-(3-phenylpropyl)morpholine-4-carboxamide (5b)



White solid. (94mg, 95%)

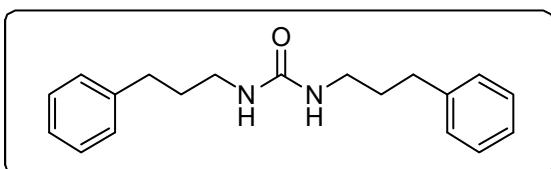
^1H NMR (400 MHz, CDCl₃) δ 7.29-7.26 (m, 2H), 7.19-7.15 (m, 3H), 4.70 (s, 1H), 3.62-3.59 (m, 4H), 3.27-3.20 (m, 6H), 2.67-2.63 (m, 2H), 1.88-1.81 (m, 2H).

^{13}C NMR (100 MHz, CDCl₃) δ 157.82, 141.81, 128.45, 128.35, 125.90, 66.45, 43.85, 40.76, 33.58, 31.56.

IR (neat) ν : 2923, 2856, 1712, 1626.1, 1538, 1262, 851 cm⁻¹.

HRMS (ESI) calcd for C₁₄H₂₁N₂O₂: 249.15975, found: 249.16045.

1,3-bis(3-phenylpropyl)urea (**5c**)



White solid. (92mg, 78%)

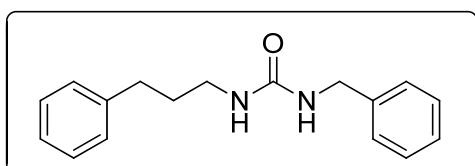
¹H NMR (400 MHz, CDCl₃) δ 7.27-7.24 (m, 5H), 7.18-7.14 (m, 5H), 4.36 (s, 2H), 3.17-3.12 (m, 4H), 2.63 (m, 4H), 1.83-1.75 (m, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 158.17, 141.53, 128.41, 128.33, 125.92, 40.11, 33.18, 31.76.

IR (neat) v: 2925, 1743, 1633, 1574, 1454, 1259 cm⁻¹.

HRMS (ESI) calcd for C₁₉H₂₅N₂O 297.19614, found: 297.19526.

1-benzyl-3-(3-phenylpropyl)urea (**5d**)



White solid. (83mg, 77%)

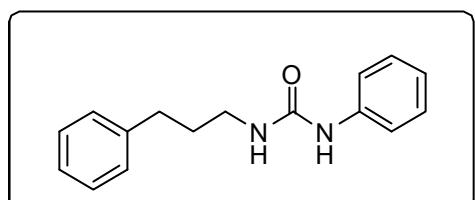
¹H NMR (300 MHz, CDCl₃) δ 7.37-7.02 (m, 10H), 5.46 (s, 1H), 5.18 (s, 1H), 4.22 (d, J = 5.7 Hz, 2H), 3.10-3.04 (m, 2H), 2.52 (t, J = 7.7 Hz, 2H), 1.78-1.58 (m, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 158.8, 141.7, 139.6, 128.6, 128.5, 128.4, 127.4, 121.2, 126.0, 44.4, 40.1, 33.2, 31.9.

IR (neat) v: 2957, 2929, 1462, 1380, 1163, 1046, 882 cm⁻¹.

HRMS (ESI) calcd for: C₁₇H₂₁N₂O, 269.16484, found : 269.16528

1-phenyl-3-(3-phenylpropyl)urea (**5e**)⁷

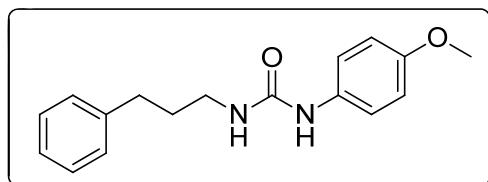


White solid. (96mg, 95%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.41 (s, 1H), 7.41-7.38 (m, 2H), 7.28-7.26 (m, 2H), 7.23-7.19 (m, 5H), 6.90-6.86 (m, 1H), 6.20 (t, J = 5.5 Hz, 1H), 3.12-3.08 (m, 2H), 2.62-2.59 (m, 2H), 1.77-1.70 (m, 2H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 155.23, 141.70, 140.57, 128.60, 128.29, 128.27, 125.72, 120.91, 177.99, 38.66, 32.50, 31.59.

1-(4-methoxyphenyl)-3-(3-phenylpropyl)urea (5f**)**



Yellow solid. (91mg, 77%)

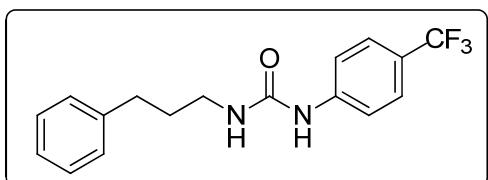
¹H NMR (400 MHz, DMSO-d⁶) δ 8.20 (s, 1H), 7.34-7.24 (m, 4H), 7.22-7.16 (m, 3H), 6.80 (d, *J* = 8.6 Hz, 2H), 6.08 (t, *J* = 5.6 Hz, 1H), 3.68 (s, 3H), 3.10-3.05 (m, 2H), 2.59 (t, *J* = 7.5 Hz, 2H), 1.78-1.64 (m, 2H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 155.49, 153.86, 141.76, 133.73, 128.33, 128.31, 125.75, 119.36, 113.87, 55.14, 38.72, 32.54, 31.69.

IR (neat) v: 1642, 1602, 1564, 1512, 1244, 832 cm⁻¹.

HRMS (ESI) calcd for C₁₇H₂₁N₂O₂: 285.15975, found: 285.16036.

1-(3-phenylpropyl)-3-(4-(trifluoromethyl)phenyl)urea (5g**)**



White solid. (69mg, 54%)

¹H NMR (300 MHz, CDCl₃) δ 8.12 (s, 1H), 7.41-7.29 (m, 4H), 7.26 – 7.09 (m, 3H), 7.02 (d, *J* = 6.9 Hz, 2H), 6.04 (s, 1H), 3.20-3.13 (m, 2H), 2.52 (t, *J* = 7.6 Hz, 2H), 1.81-1.63 (m, 2H).

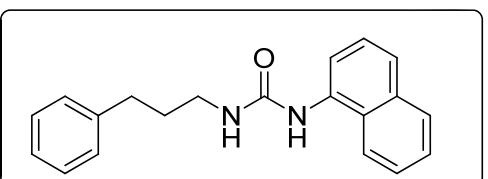
¹³C NMR (75 MHz, CDCl₃) δ 156.47, 142.31, 141.10, 128.60, 128.31, 126.38 (q, *J* = 5.0 Hz), 124.83 (q, *J* = 32.7 Hz), 124.30 (q, *J* = 269.7 Hz), 119.04, 39.93, 33.15, 31.63.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.0.

IR (neat) v: 1657, 1604, 1557, 1328, 1119, 1068, 656 cm⁻¹.

HRMS (ESI) calcd for C₁₇H₁₈F₃N₂O: 323.13657, found: 323.13569.

1-(naphthalen-2-yl)-3-(3-phenylpropyl)urea (5h**)**



White solid. (95mg, 78%)

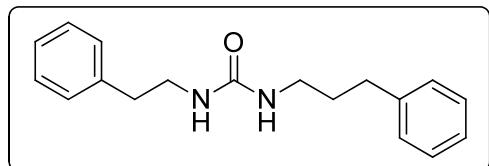
¹H NMR (400 MHz, DMSO-d⁶) δ 8.52 (s, 1H), 8.11-8.09 (m, 1H), 8.01-7.99 (m, 1H), 7.90-7.88 (m, 1H), 7.57-7.49 (m, 3H), 7.43-7.39 (m, 1H), 7.31-7.28 (m, 2H), 7.25-7.23 (m, 2H), 7.21-7.16 (m, 1H), 6.67 (t, *J* = 5.6 Hz, 1H), 3.19-3.14 (m, 2H), 2.67-2.63 (m, 2H), 1.82-1.75 (m, 2H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 155.57, 141.68, 135.20, 133.70, 128.33, 128.30, 128.29, 125.91, 125.74, 125.70, 125.46, 125.34, 121.91, 121.35, 116.32, 38.76, 32.52, 31.53.

IR (neat) ν : 2958, 2083, 1692, 1381, 1300, 1127 cm^{-1} .

HRMS (ESI) calcd for: 305.16484, found: 305.16422.

1-phenethyl-3-(3-phenylpropyl)urea (**5i**)



White solid. (92mg, 81%)

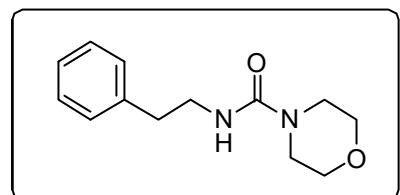
^1H NMR (400 MHz, CDCl_3) δ 7.31-7.14 (m, 10H), 4.43 (s, 2H), 3.40-3.37 (m, 2H), 3.15-3.13 (m, 2H), 2.80-2.76 (m, 2H), 2.64-2.60 (m, 2H), 1.82-1.75 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 158.27, 141.69, 139.31, 128.94, 128.71, 128.56, 128.48, 126.53, 126.07, 41.77, 40.23, 36.53, 33.32, 31.92.

IR (neat) ν : 2927, 2859, 1643, 1564, 1496, 1453, 835 cm^{-1} .

HRMS (ESI) calcd for $\text{C}_{18}\text{H}_{23}\text{N}_2\text{O}$ 283.18049, found: 283.17970.

N-phenethylmorpholine-4-carboxamide (**5j**)⁸

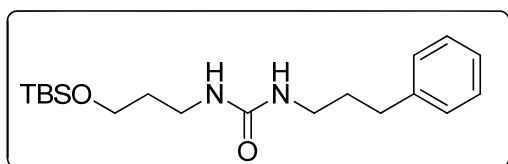


White solid. (75mg, 80%)

^1H NMR (400 MHz, CDCl_3) δ 7.33-7.29 (m, 2H), 7.25-7.19 (m, 3H), 4.46 (s, 1H), 3.66-3.64 (m, 4H), 3.53-3.48 (m, 2H), 3.29-3.26 (m, 4H), 2.85-2.81 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 157.79, 139.37, 128.94, 128.74, 126.58, 66.60, 44.04, 42.11, 36.39.

1-(3-((tert-butyldimethylsilyl)oxy)propyl)-3-(3-phenylpropyl)urea (**5k**)



White oil. (103mg, 73%)

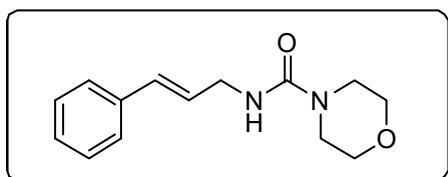
^1H NMR (400 MHz, CDCl_3) δ 7.27-7.16 (m, 5H), 4.73 (s, 1H), 4.44 (s, 1H), 3.72-3.69 (m, 2H), 3.26-3.20 (m, 2H), 2.67-2.63 (m, 2H), 1.84-1.83 (m, 2H), 1.70-1.67 (m, 4H), 0.90 (s, 9H), 0.06 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ 158.54, 141.76, 128.56, 128.50, 126.05, 61.77, 40.33, 38.77, 34.35, 32.65, 32.05, 26.08, 18.41, -5.21.

IR (neat) ν : 2928, 2856, 1631, 1575, 1255, 1097, 835 cm^{-1} .

HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{35}\text{N}_2\text{O}_2\text{Si}$: 351.24623, found: 351.24535.

N-cinnamylmorpholine-4-carboxamide (**5l**)



White oil. (51mg, 52%)

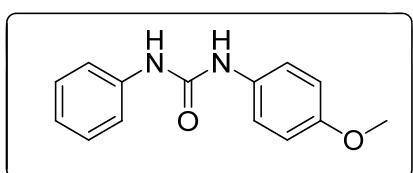
¹H NMR (400 MHz, CDCl₃) δ 7.37-7.21 (m, 5H), 6.53-6.49 (m, 1H), 6.27-6.20 (m, 1H), 4.65 (s, 1H), 4.04-4.01 (m, 2H), 3.69-3.67 (m, 4H), 3.37-3.35 (m, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 157.53, 136.58, 131.74, 128.54, 127.63, 126.69, 126.31, 66.48, 43.98, 43.07.

IR (neat) v: 2921, 2856, 1629, 1534, 1259, 1117, 966 cm⁻¹.

HRMS (ESI) calcd for C₁₄H₁₈N₂NaO₂: 269.12605, found: 269.12557.

1-(4-methoxyphenyl)-3-phenylurea (**7a**)⁹

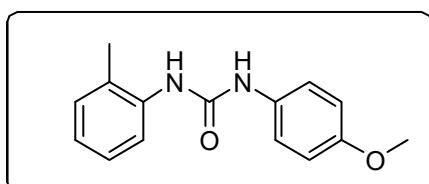


Yellow solid. (88mg, 90%)

¹H NMR (300 MHz, DMSO-d⁶) δ 8.56 (s, 1H), 8.45 (s, 1H), 7.44 (d, J = 7.8 Hz, 2H), 7.35 (d, J = 9.0 Hz, 2H), 7.29-7.23 (m, 2H), 6.97-6.92 (m, 1H), 6.90-6.81 (m, 2H), 3.71 (s, 3H).

¹³C NMR (75 MHz, DMSO-d⁶) δ 154.46, 152.70, 139.87, 132.70, 128.70, 121.56, 120.01, 118.06, 113.97, 55.15.

1-(4-methoxyphenyl)-3-(o-tolyl)urea (**7b**)¹⁰

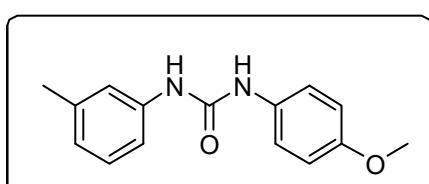


Yellow solid. (93mg, 91%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.82 (s, 1H), 7.84-7.81 (m, 1H), 7.81 (s, 1H), 7.37-7.35 (m, 2H), 7.17-7.11 (m, 2H), 6.92-6.90 (m, 1H), 6.88-6.86 (m, 2H), 3.71 (s, 3H), 2.23 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 154.32, 152.78, 137.59, 132.91, 130.12, 127.19, 126.11, 122.39, 120.78, 119.70, 114.01, 55.15, 17.89.

1-(4-methoxyphenyl)-3-(m-tolyl)urea (**7c**)¹⁰

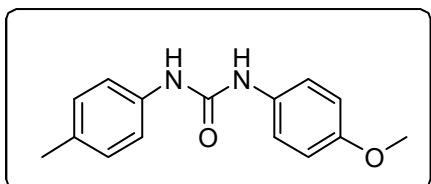


Yellow solid. (84mg, 81%)

^1H NMR (400 MHz, DMSO-d⁶) δ 8.48 (s, 1H), 8.43 (s, 1H), 7.35-7.33 (m, 2H), 7.28-7.27 (m, 1H), 7.21 (d, J = 8.0Hz, 1H), 7.15-7.11 (m, 1H), 6.87-6.85 (m, 2H), 6.77 (d, J = 4.0Hz, 1H), 3.71 (s, 3H), 2.27 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d⁶) δ 154.40, 152.67, 139.79, 137.86, 132.73, 128.56, 122.32, 119.92, 118.56, 115.23, 113.96, 55.16, 21.23.

1-(4-methoxyphenyl)-3-(p-tolyl)urea (**7d**) ¹¹

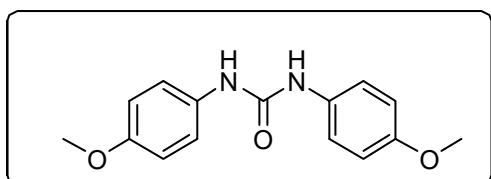


Yellow solid. (95mg, 93%)

^1H NMR (400 MHz, DMSO-d⁶) δ 8.44 (s, 1H), 8.40 (s, 1H), 7.35-7.30 (m, 4H), 7.06 (d, J = 8.0Hz, 2H), 6.85 (d, J = 8.0Hz, 2H), 3.71 (s, 3H), 2.23 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d⁶) δ 154.37, 152.77, 137.33, 132.83, 130.37, 129.14, 119.92, 118.16, 113.97, 55.16, 20.35.

1,3-bis(4-methoxyphenyl)urea (**7e**) ¹²

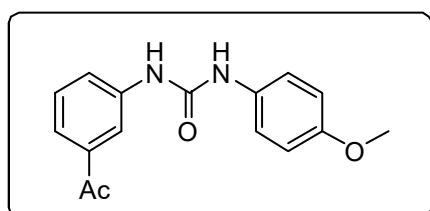


Yellow solid. (96mg, 88%)

^1H NMR (400 MHz, DMSO-d⁶) δ 8.35 (s, 2H), 7.32 (d, J = 8.0Hz, 4H), 6.84 (d, J = 8.0Hz, 4H), 3.70 (s, 6H).

^{13}C NMR (100 MHz, DMSO-d⁶) δ 154.73, 153.36, 133.34, 120.31, 114.38, 55.59.

1-(4-acetylphenyl)-3-(4-methoxyphenyl)urea (**7f**) ¹³

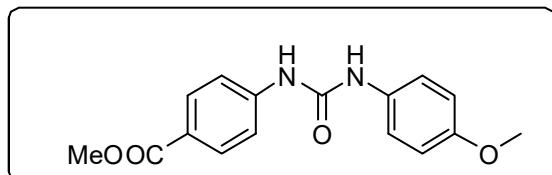


Yellow solid. (89mg, 78%)

^1H NMR (400 MHz, DMSO-d⁶) δ 8.81 (s, 1H), 8.51 (s, 1H), 8.06-8.05 (m, 1H), 7.69 – 7.64 (m, 1H), 7.56 (d, J = 7.8 Hz, 1H), 7.44-7.36 (m, 1H), 7.38-7.34 (m, 2H), 6.89-6.84 (m, 2H), 3.72 (s, 3H), 2.56 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 198.19, 155.01, 153.15, 140.77, 137.80, 132.91, 129.54, 123.09, 122.11, 120.65, 117.66, 114.40, 55.60, 27.19.

methyl 4-(3-(4-methoxyphenyl)ureido)benzoate (**7g**)



White solid. (106mg, 88%)

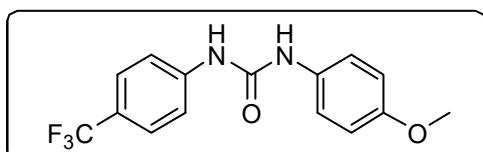
¹H NMR (400 MHz, DMSO-d⁶) δ 9.01 (s, 1H), 8.60 (s, 1H), 7.87 (d, *J* = 8.6 Hz, 2H), 7.57 (d, *J* = 8.6 Hz, 2H), 7.36 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H), 3.72 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 165.92, 154.72, 152.30, 144.59, 132.24, 130.38, 122.17, 120.26, 117.15, 114.01, 55.17, 51.75.

IR (neat) v: 1714, 1651, 1600, 1563, 1512, 1284, 832 cm⁻¹.

HRMS (ESI) calcd for C₁₆H₁₇N₂O₄: 301.11828, found: 301.11840.

1-(4-methoxyphenyl)-3-(4-(trifluoromethyl)phenyl)urea (**7h**) ¹⁴



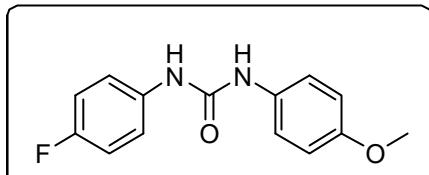
White solid. (120mg, 97%)

¹H NMR (400 MHz, DMSO-d⁶) δ 9.01 (s, 1H), 8.60 (s, 1H), 7.66-7.60 (m, 4H), 7.36 (d, *J* = 8.0Hz, 2H), 6.88 (d, *J* = 8.0Hz, 2H), 3.72 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 154.72, 152.43, 143.62, 132.25, 126.05 (q, *J* = 4.0 Hz), 124.59 (q, *J* = 270.0 Hz), 120.31, 118.14, 117.69, 114.01, 55.18.

¹⁹F NMR (376 MHz, DMSO-d⁶) δ -60.1.

1-(4-fluorophenyl)-3-(4-methoxyphenyl)urea (**7i**) ¹⁵



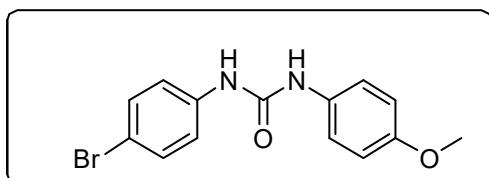
White solid. (97mg, 93%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.59 (s, 1H), 8.44 (s, 1H), 7.46-7.42 (m, 2H), 7.35-7.33 (m, 2H), 7.12-7.08 (m, 2H), 6.7-6.85 (m, 2H), 3.71 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 157.19 (q, *J* = 236.2 Hz), 154.46, 152.81, 136.22, 132.66, 120.07, 119.79 (d, *J* = 7.4 Hz), 115.21, (d, *J* = 21.2 Hz), 113.96, 55.16.

¹⁹F NMR (376 MHz, DMSO-d⁶) δ -121.9.

1-(4-bromophenyl)-3-(4-methoxyphenyl)urea (**7j**)¹⁶

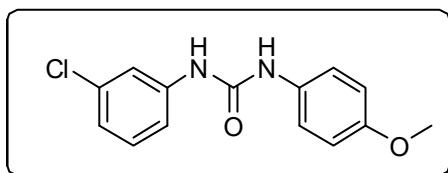


Yellow solid. (89mg, 69%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.72 (s, 1H), 8.50 (s, 1H), 7.42 (s, 4H), 7.34 (d, *J* = 8.0Hz, 2H), 6.86 (d, *J* = 8.0Hz, 2H), 3.71 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 154.56, 152.56, 139.34, 132.47, 131.45, 120.15, 119.98, 113.98, 112.91, 55.16.

1-(3-chlorophenyl)-3-(4-methoxyphenyl)urea (**7k**)¹⁰

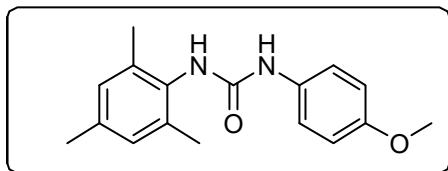


White solid. (97mg, 87%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.79 (s, 1H), 8.54 (s, 1H), 7.70-7.69 (m, 1H), 7.36-7.33 (m, 2H), 7.30-7.23 (m, 2H), 7.00-6.98 (m, 1H), 6.88-6.86 (m, 2H), 3.72 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ: 154.46, 152.54, 141.48, 133.15, 132.36, 130.35, 121.19, 120.24, 117.40, 116.49, 113.99, 55.17.

1-mesityl-3-(4-methoxyphenyl)urea (**7l**)¹⁷

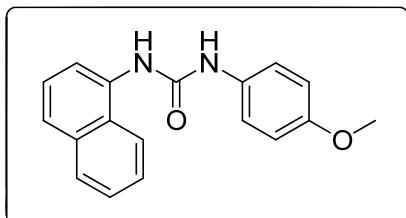


White solid. (93mg, 82%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.45 (s, 1H), 7.50 (s, 1H), 7.34-7.32 (m, 2H), 6.87 (s, 2H), 6.85-6.82 (m, 2H), 3.70 (s, 3H), 2.22 (s, 3H), 2.15 (s, 6H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 154.07, 153.53, 135.31, 134.75, 133.48, 132.86, 128.29, 119.60, 113.89, 55.1, 20.50, 18.22.

1-(4-methoxyphenyl)-3-(naphthalen-1-yl)urea (**7m**)¹⁸

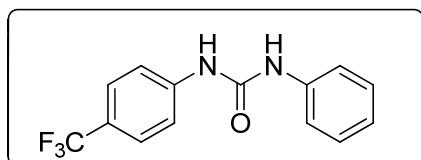


White solid. (96mg, 82%)

¹H NMR (400 MHz, DMSO-d⁶) δ 8.87(s, 1H), 8.68 (s, 1H), 8.12 (d, *J* = 7.8Hz, 1H), 8.02 (d, *J* = 7.8Hz, 1H), 7.94-7.91 (m, 1H), 7.63-7.52 (m, 3H), 7.48-7.44 (m, 1H), 7.43-7.39 (m, 2H), 6.90 (d, *J* = 9.0Hz, 2H), 3.73 (s, 3H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 154.46, 153.06, 134.50, 133.71, 132.80, 128.41, 125.88, 125.85, 125.80, 125.63, 122.66, 121.28, 119.86, 117.09, 114.06, 55.18.

phenyl-3-(4-(trifluoromethyl)phenyl)urea (**7n**) ¹⁹



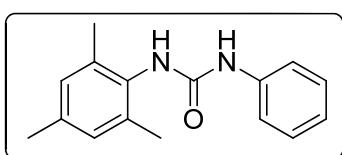
White solid. (99mg, 88%)

¹H NMR (400 MHz, DMSO-d⁶) δ 9.08 (s, 1H), 8.78 (s, 1H), 7.67-7.61 (m, 4H), 7.49-7.42 (m, 2H), 7.31-7.27 (m, 2H), 7.01-6.97 (m, 1H).

¹³C NMR (100 MHz, DMSO-d⁶) δ 152.26, 143.47, 139.28, 128.81, 126.06 (q, *J* = 3.8 Hz), 124.55 (q, *J* = 269.4 Hz), 122.20, 121.71 (q, *J* = 31.7 Hz), 118.43, 117.80.

¹⁹F NMR (376 MHz, DMSO- d⁶) δ -60.1.

1-mesityl-3-phenylurea (**7o**) ¹⁸

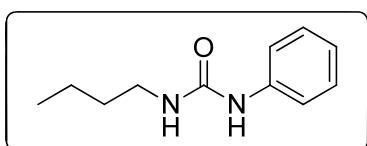


White solid. (46mg, 45%)

¹H NMR (400 MHz, DMSO-d⁶) 8.66 (s, 1H), 7.60 (s, 1H), 7.44 (d, *J* = 7.7 Hz, 2H), 7.26-7.22 (m, 2H), 6.93-6.90 (m, 1H), 6.88 (s, 2H), 2.23 (s, 3H), 2.16 (s, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 153.70, 140.78, 135.71, 135.28, 133.08, 129.09, 128.72, 121.69, 118.25, 20.91, 18.60.

1-butyl-3-phenylurea (**7p**) ²⁰

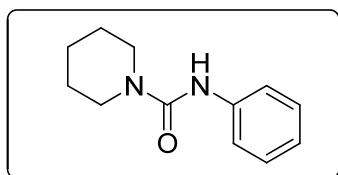


White solid. (66mg, 85%)

¹H NMR (400 MHz, CDCl₃) δ 8.35 (s, 1H), 7.38-7.35 (m, 2H), 7.23-7.16 (m, 2H), 6.89-6.85 (m, 1H), 6.08 (s, 1H), 3.09-3.04 (m, 2H), 1.45-1.36 (m, 2H), 1.35-1.27 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 155.16, 140.58, 128.58, 120.83, 171.50, 38.65, 31.87, 19.51, 13.68.

N-phenylpiperidine-1-carboxamide (**7q**)²¹

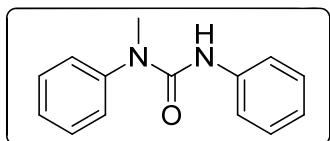


White solid. (82mg, >98%)

¹H NMR (400 MHz, CDCl₃) δ 8.40 (s, 1H), 7.44 (d, *J* = 7.8 Hz, 2H), 7.21 (t, *J* = 7.8 Hz, 2H), 6.95-6.88 (m, 1H), 3.44-3.38 (m, 4H), 1.61-1.52 (m, 2H), 1.51-1.46 (m, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 154.87, 140.77, 128.19, 121.44, 119.54, 44.66, 25.51, 24.12.

1-methyl-1,3-diphenylurea (**7r**)²²



White solid. (84mg, 92%)

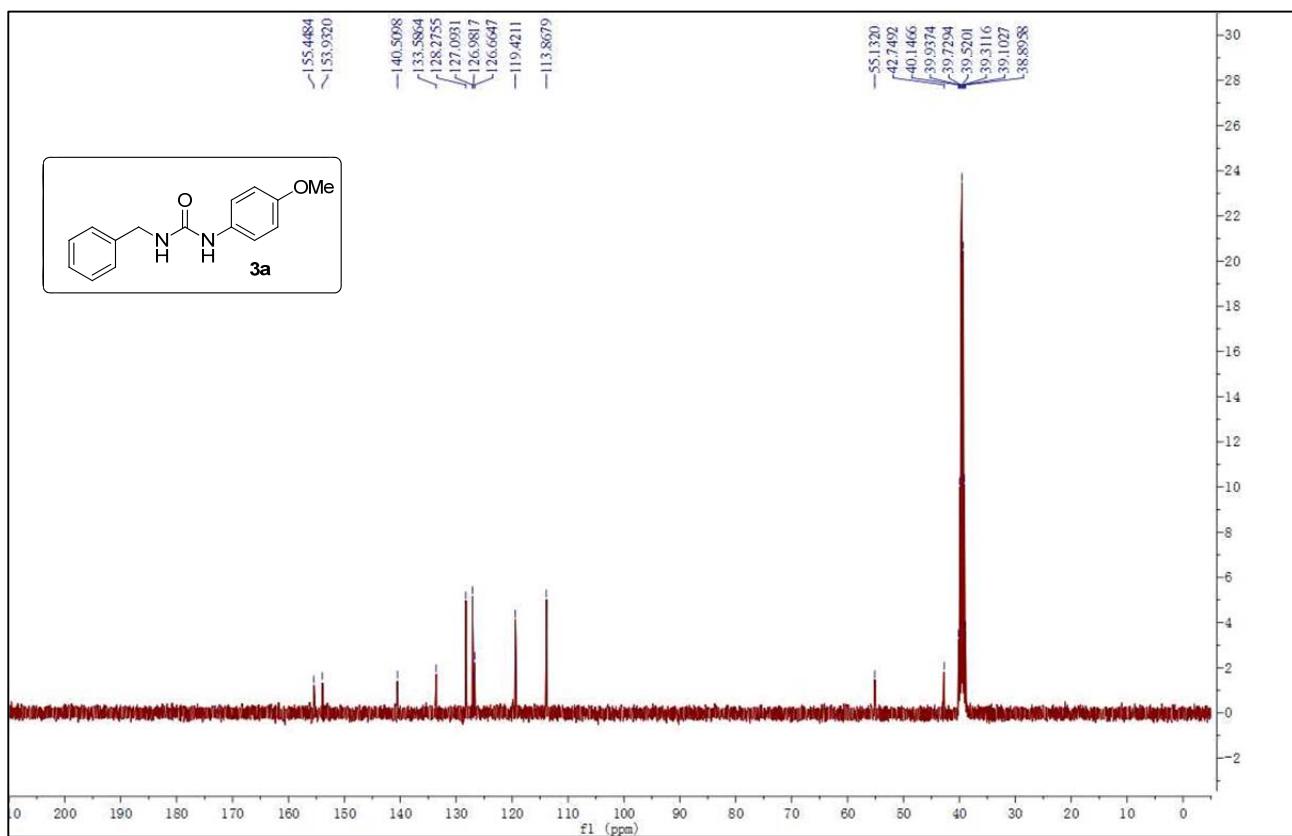
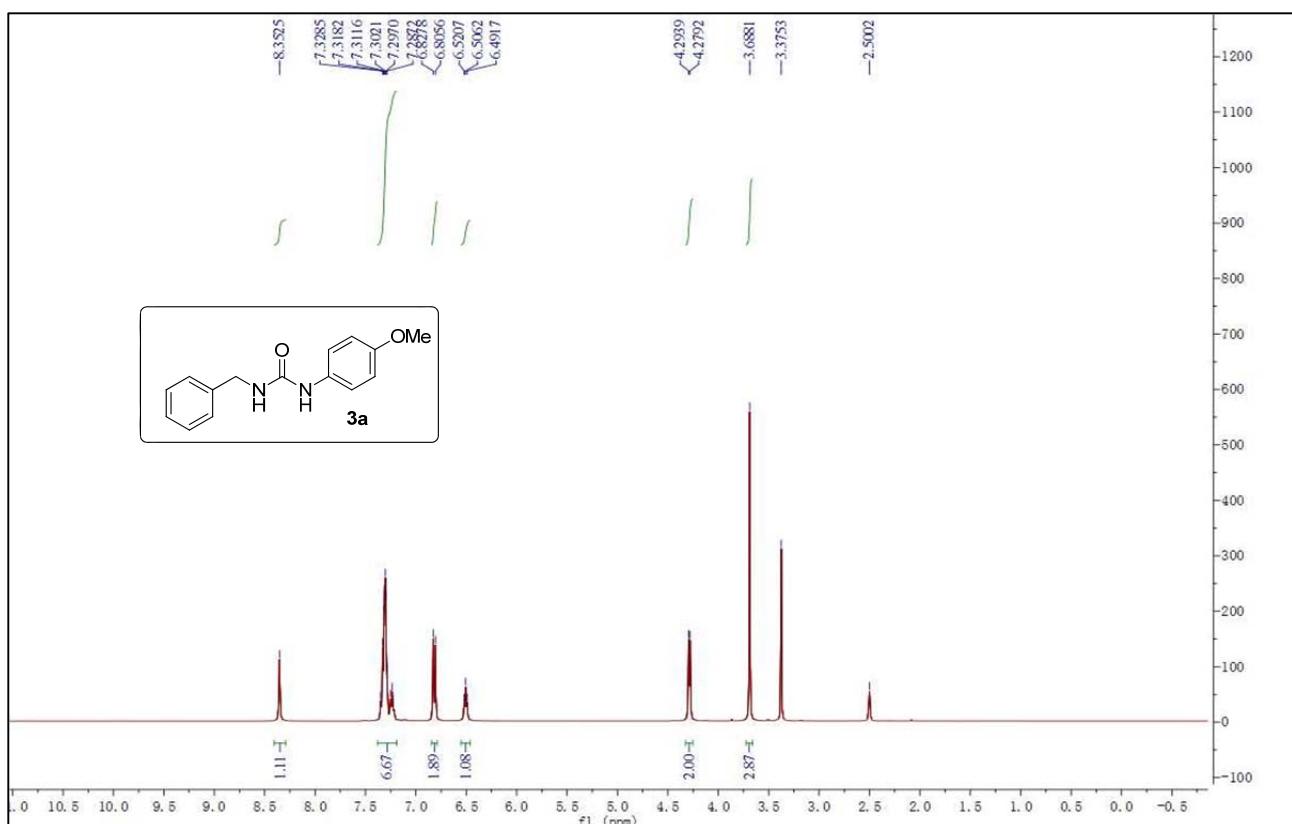
¹H NMR (400 MHz, CDCl₃) δ 8.12 (s, 1H), 7.44-7.37 (m, 4H), 7.34-7.29 (m, 2H), 7.28-7.18 (m, 3H), 6.96-6.92 (m, 1H), 3.27 (s, 3H).

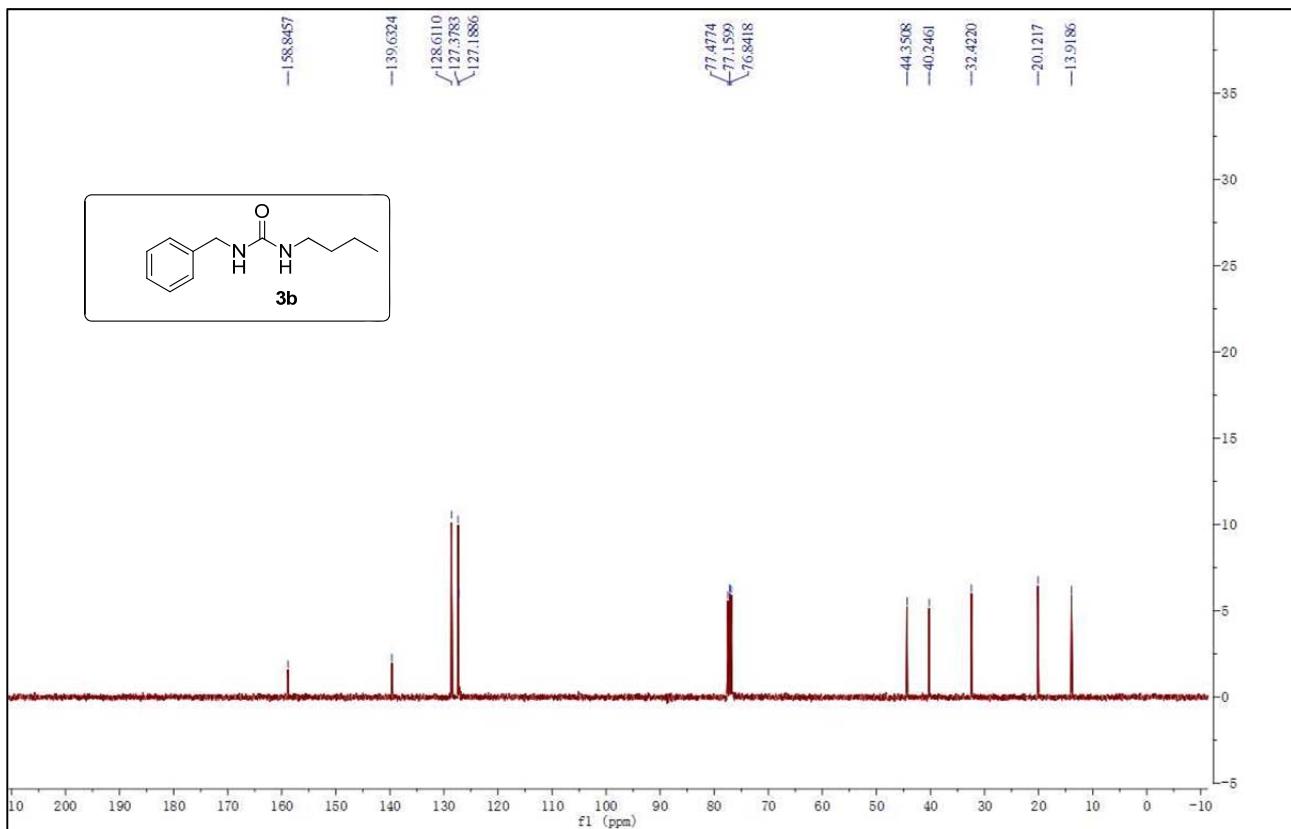
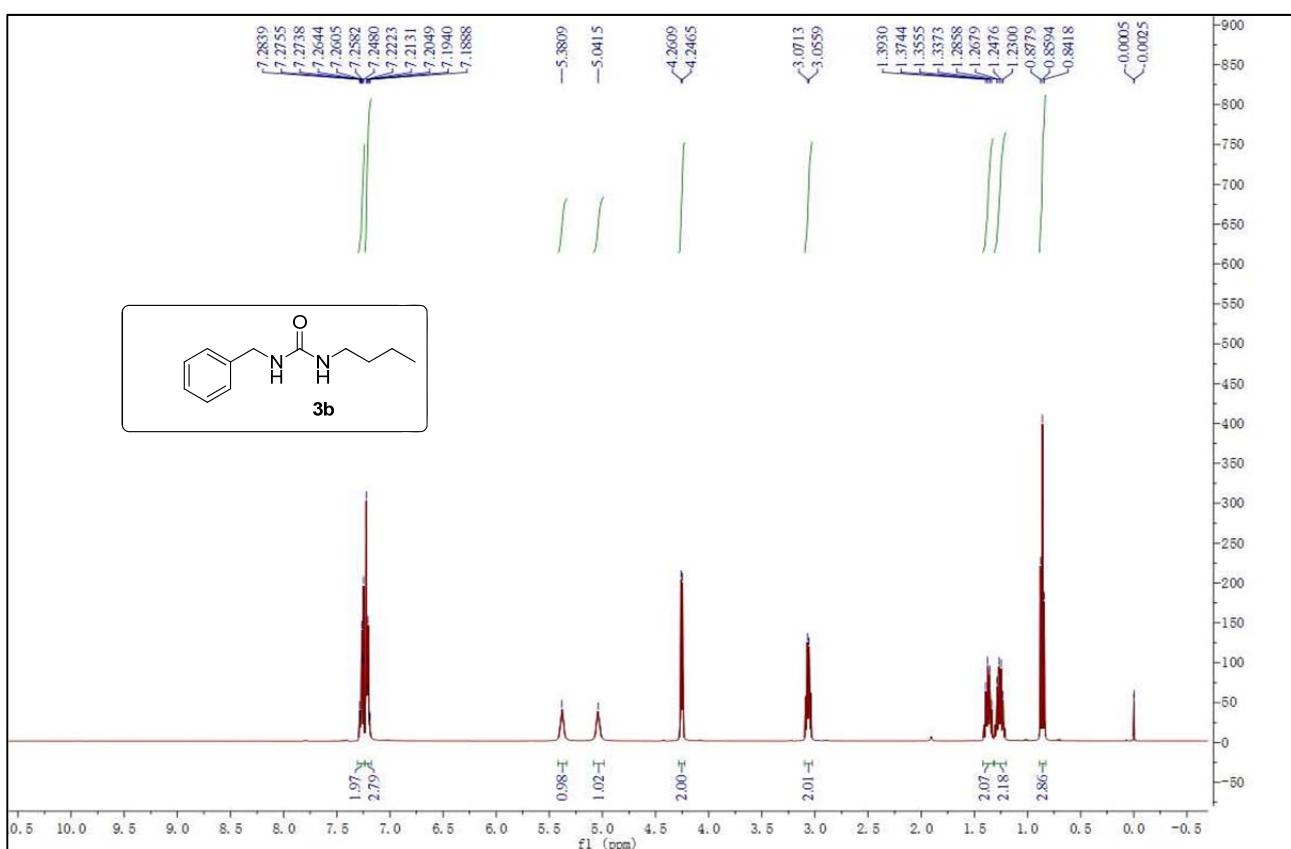
¹³C NMR (100 MHz, CDCl₃) δ 154.69, 144.08, 140.05, 129.20, 128.23, 126.18, 125.70, 121.98, 119.85, 37.52.

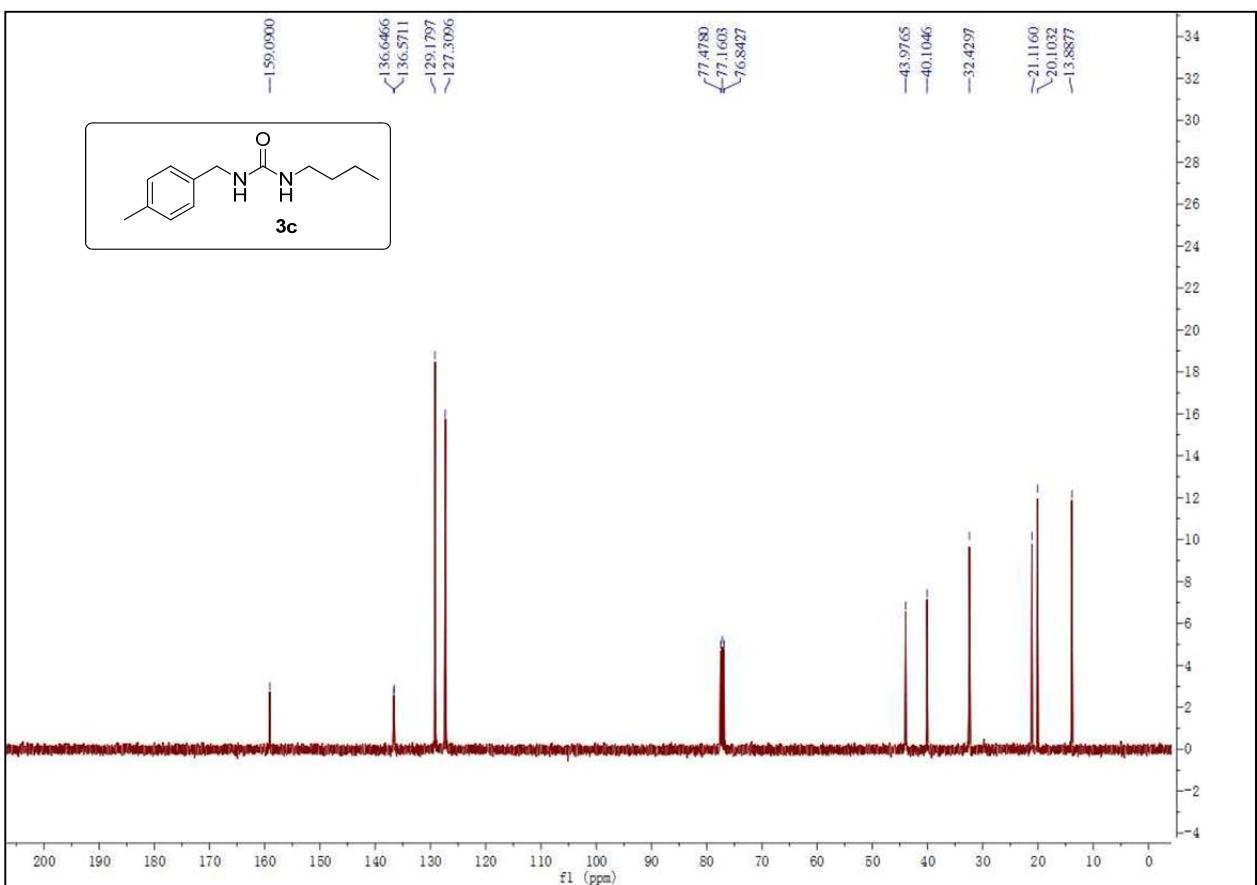
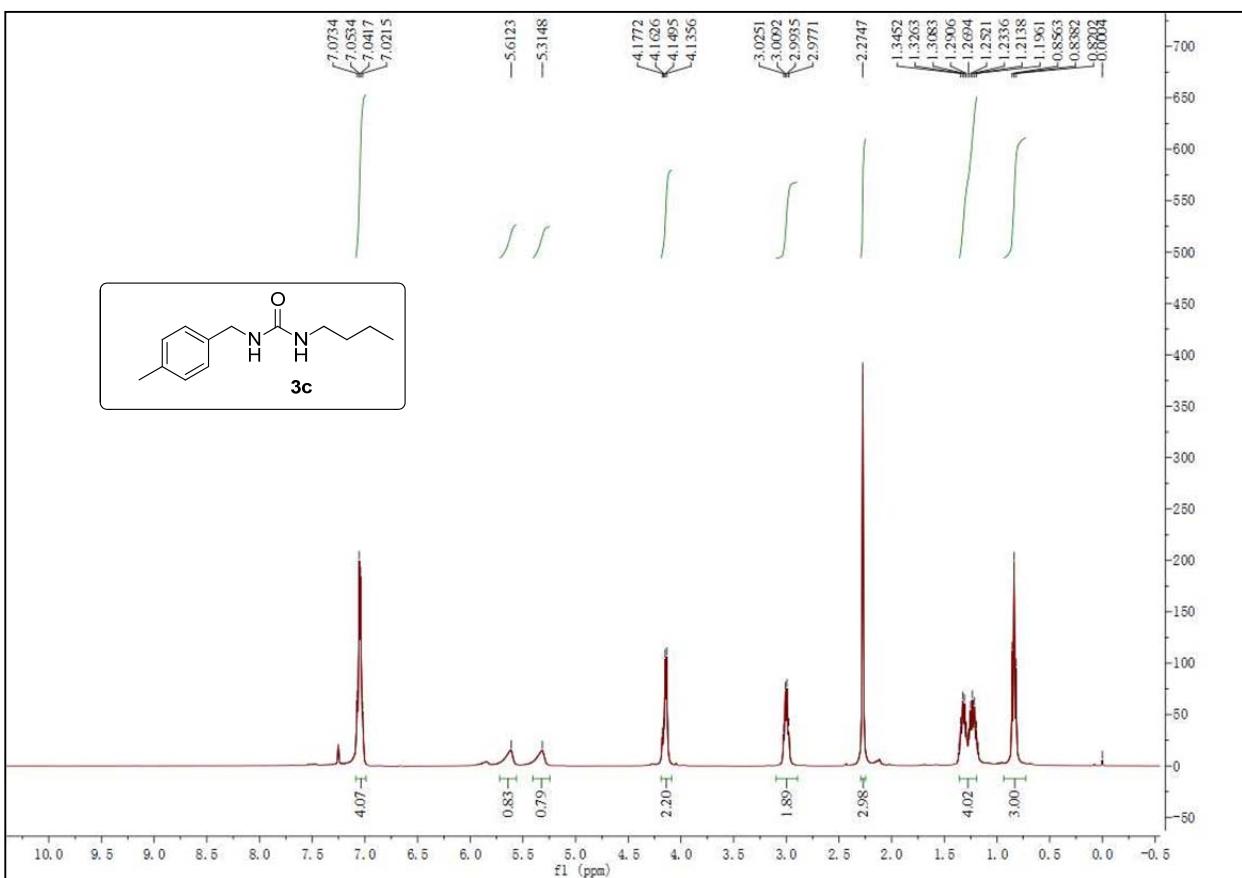
6. References:

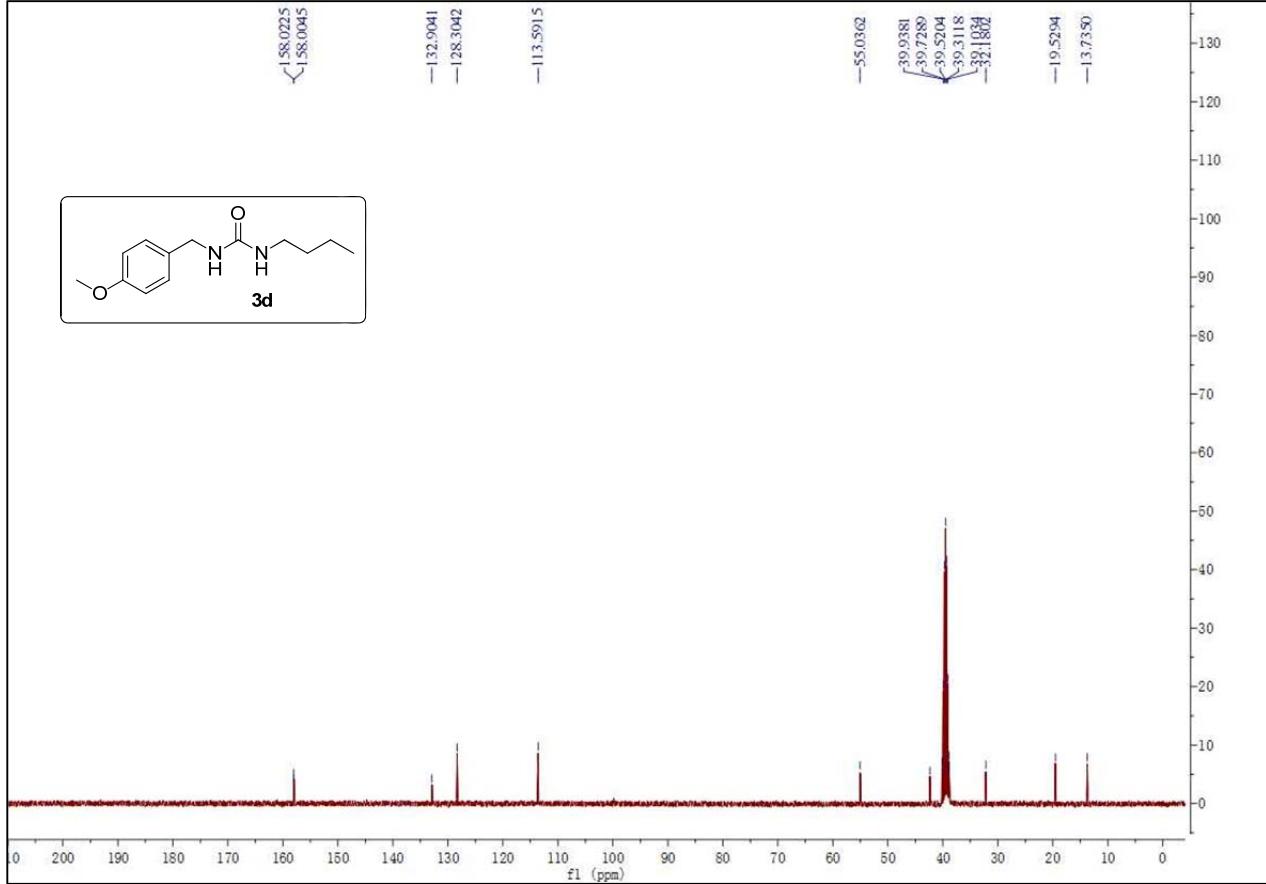
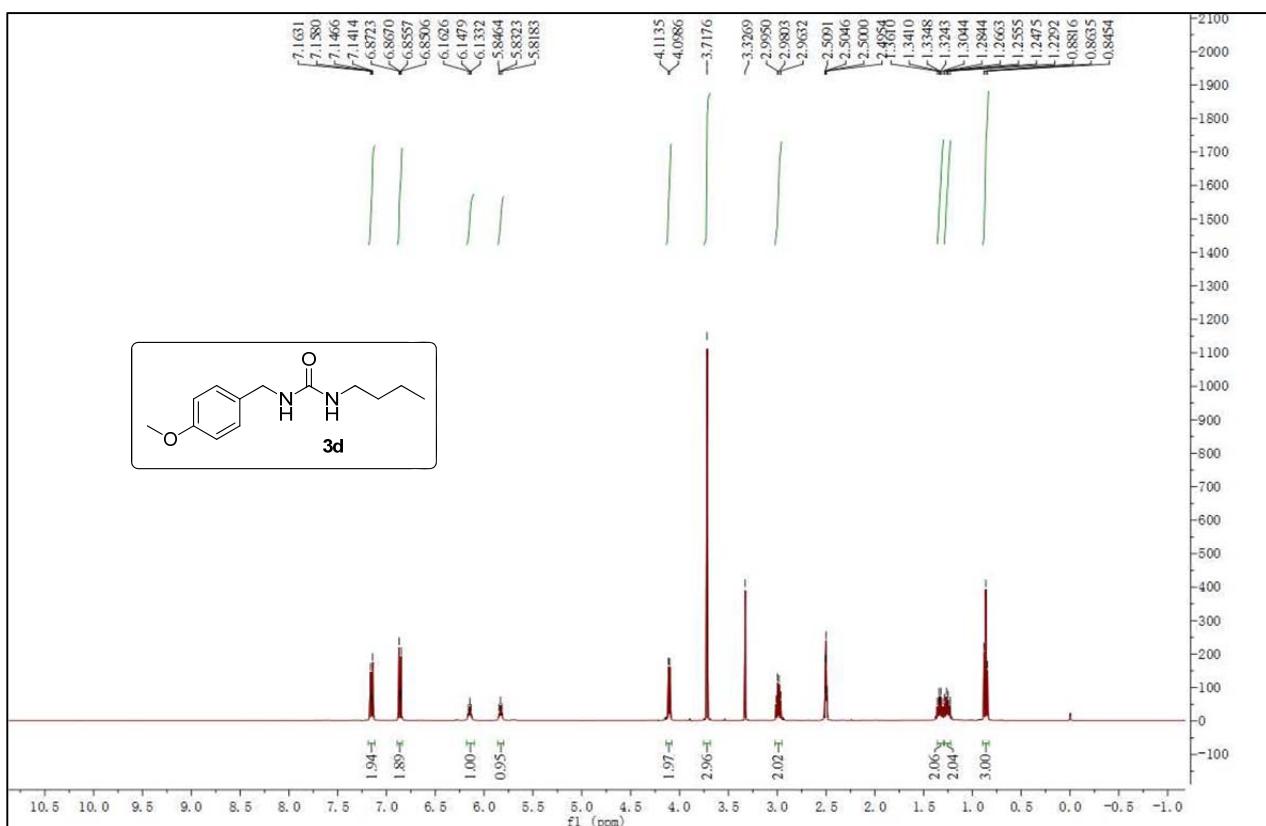
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- (22) Zhang, L.; Darko, A. K.; Johns, J. I.; McElwee-White, L. *Eur. J. Org. Chem.* **2011**, *31*, 6261.

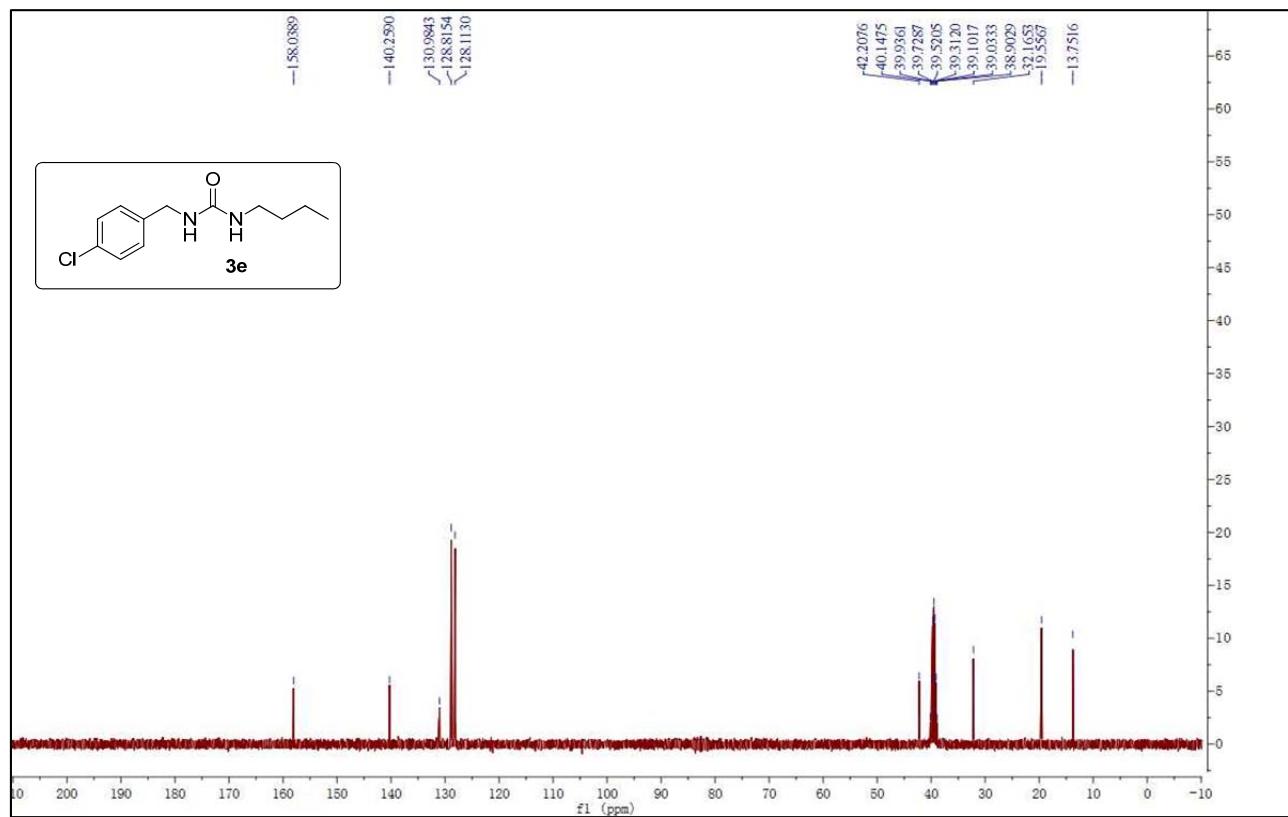
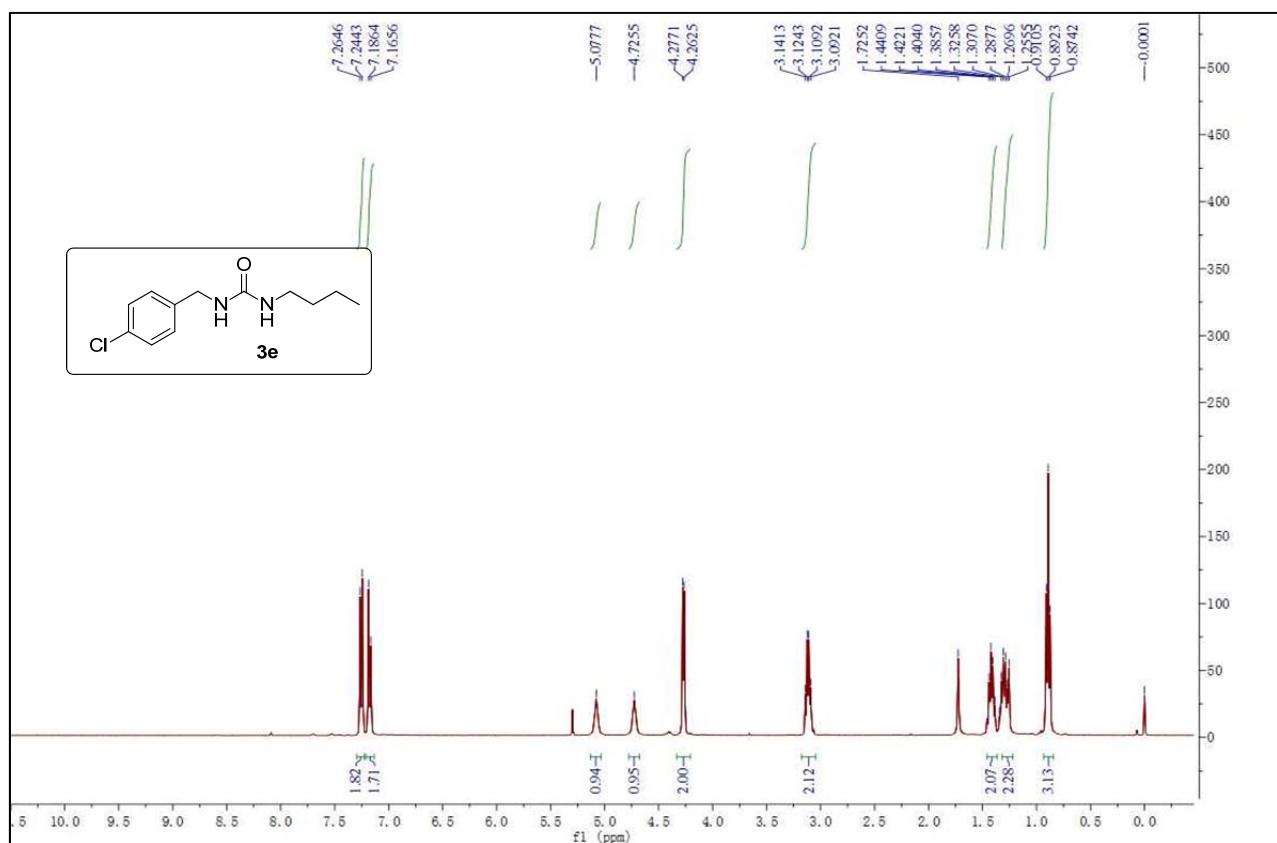
7. ^1H and ^{13}C NMR spectra

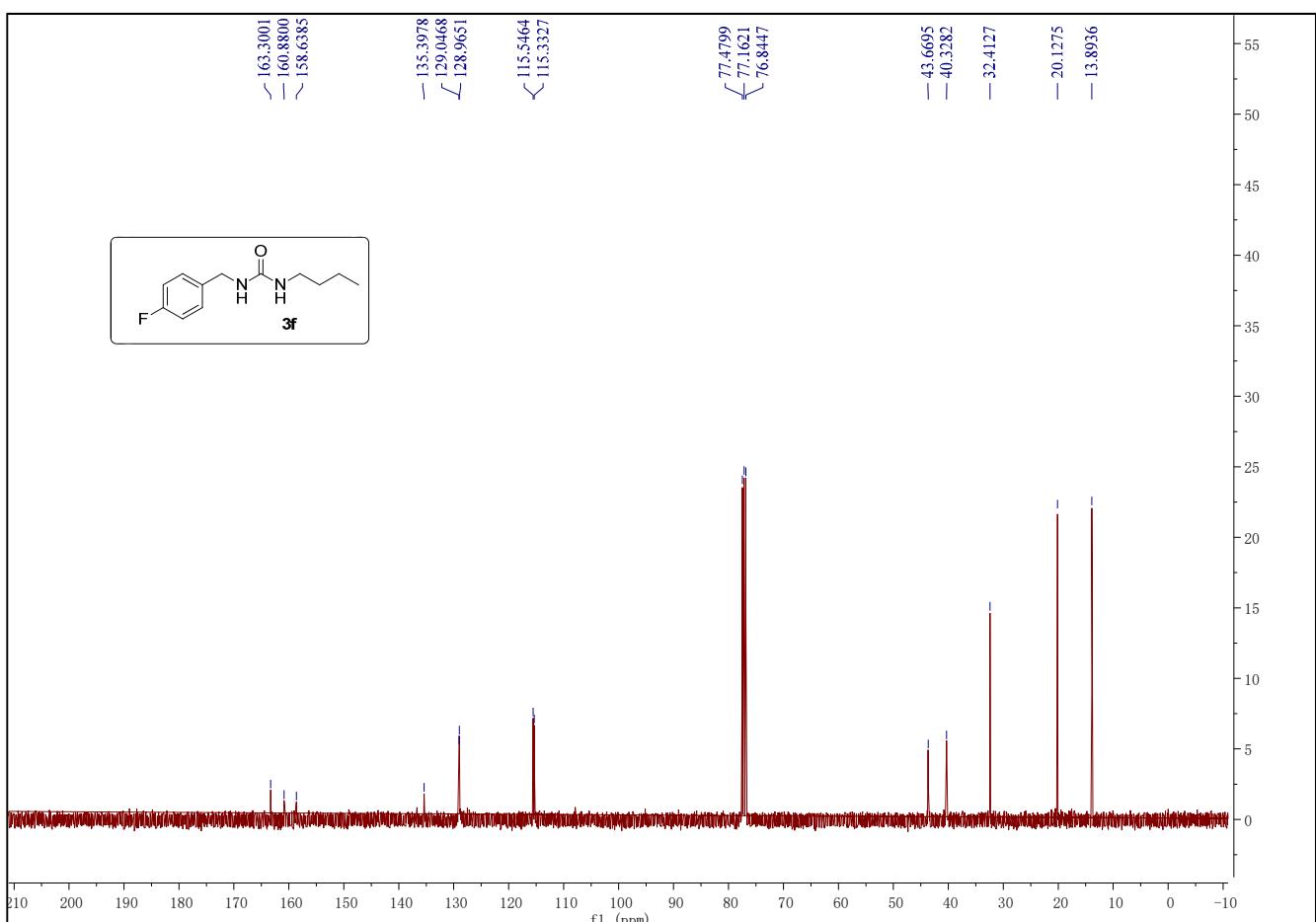
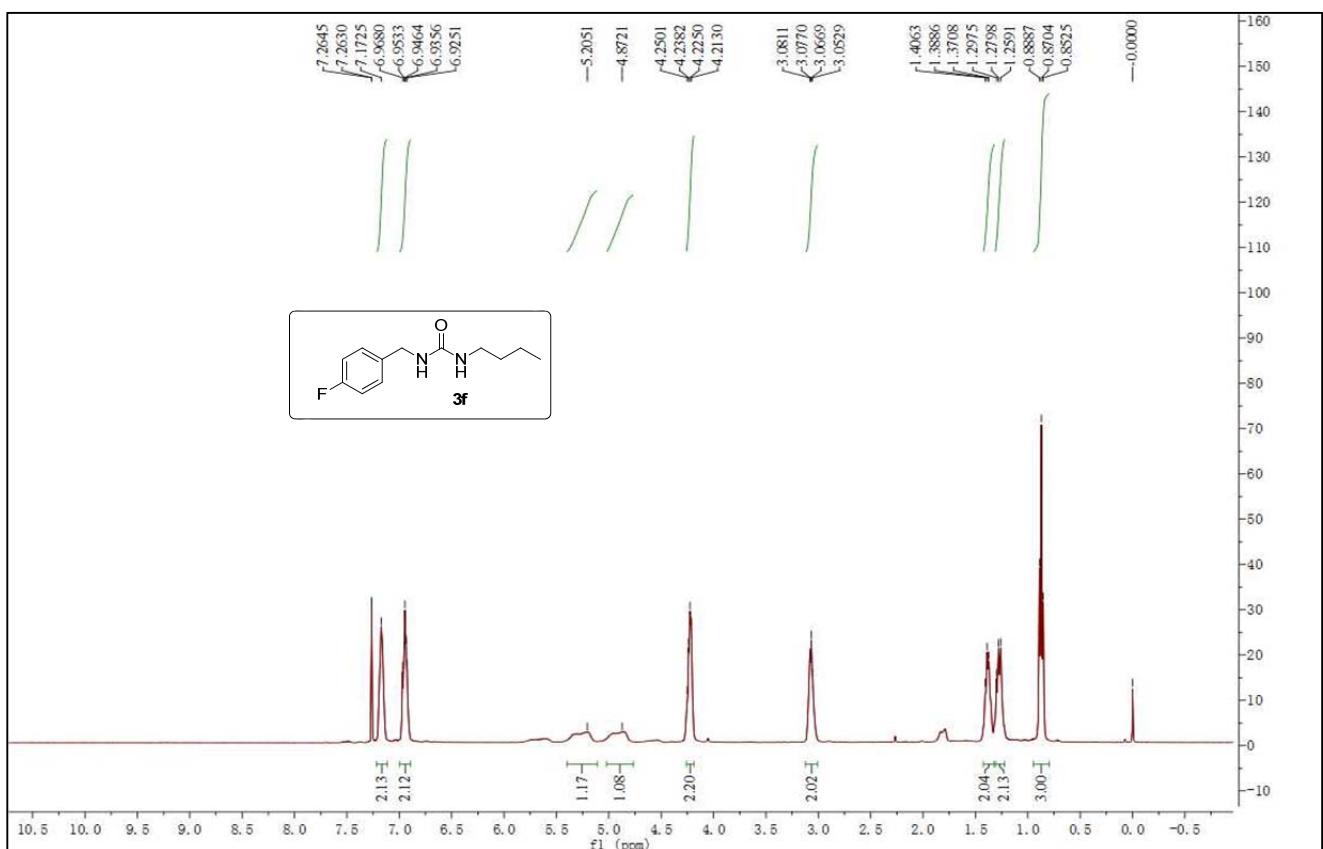


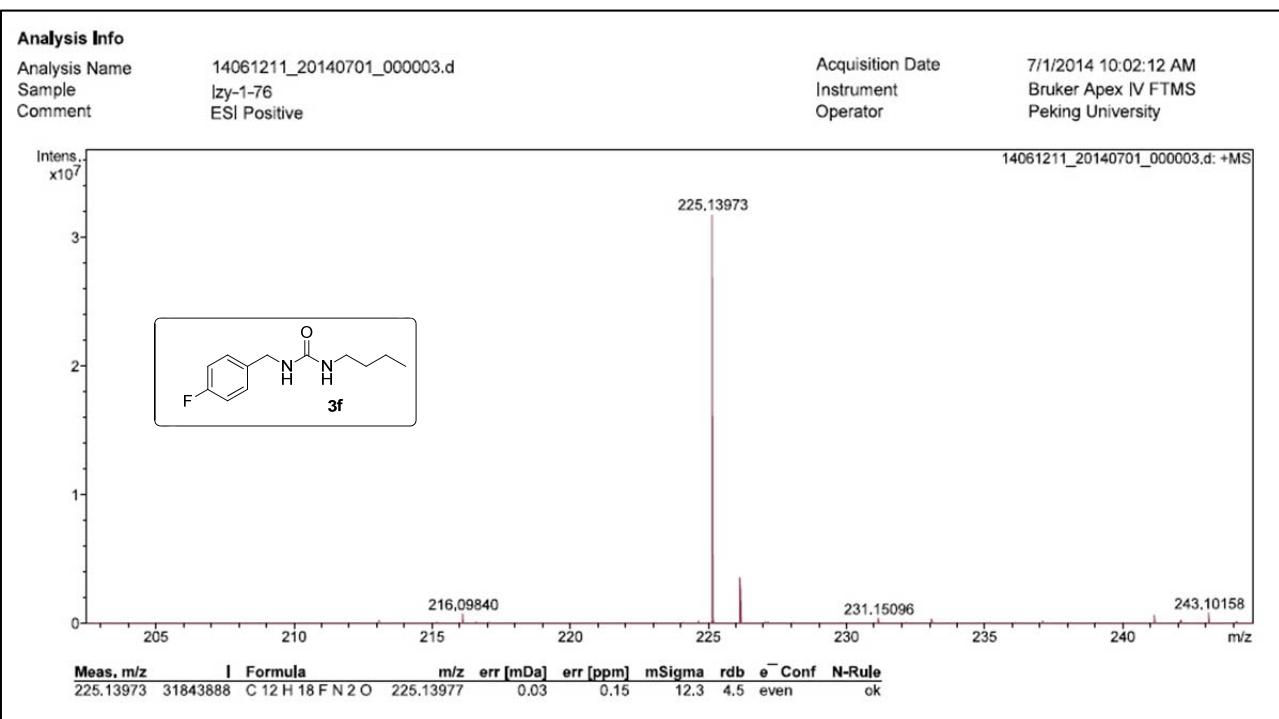
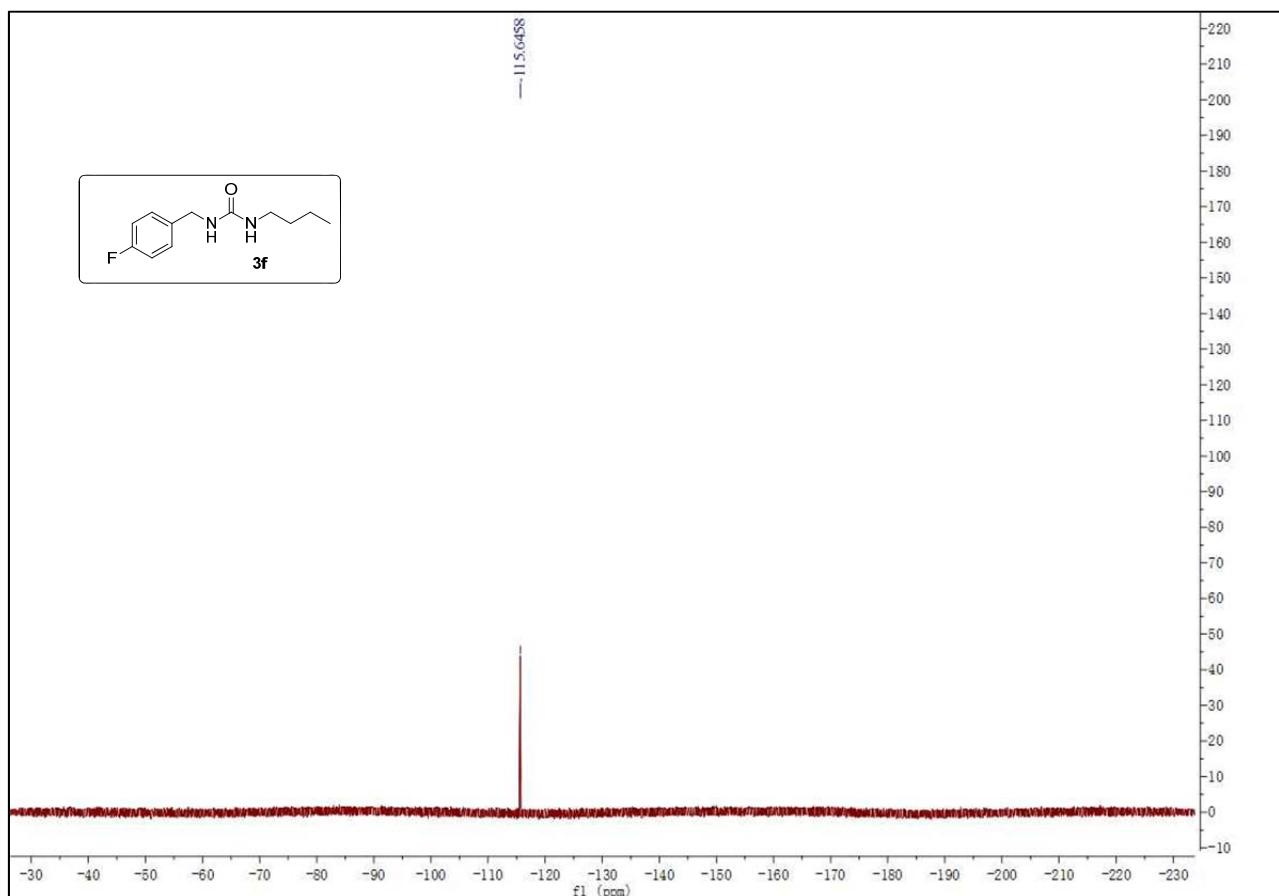


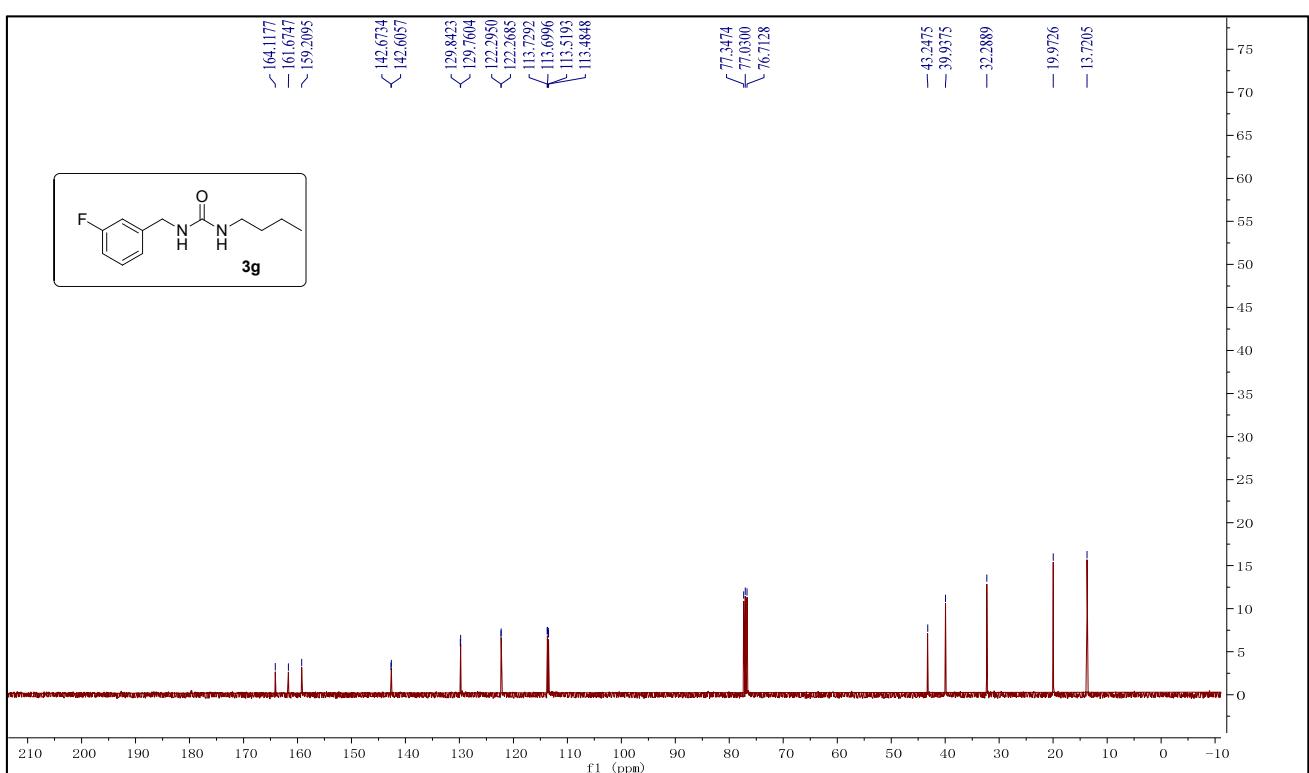
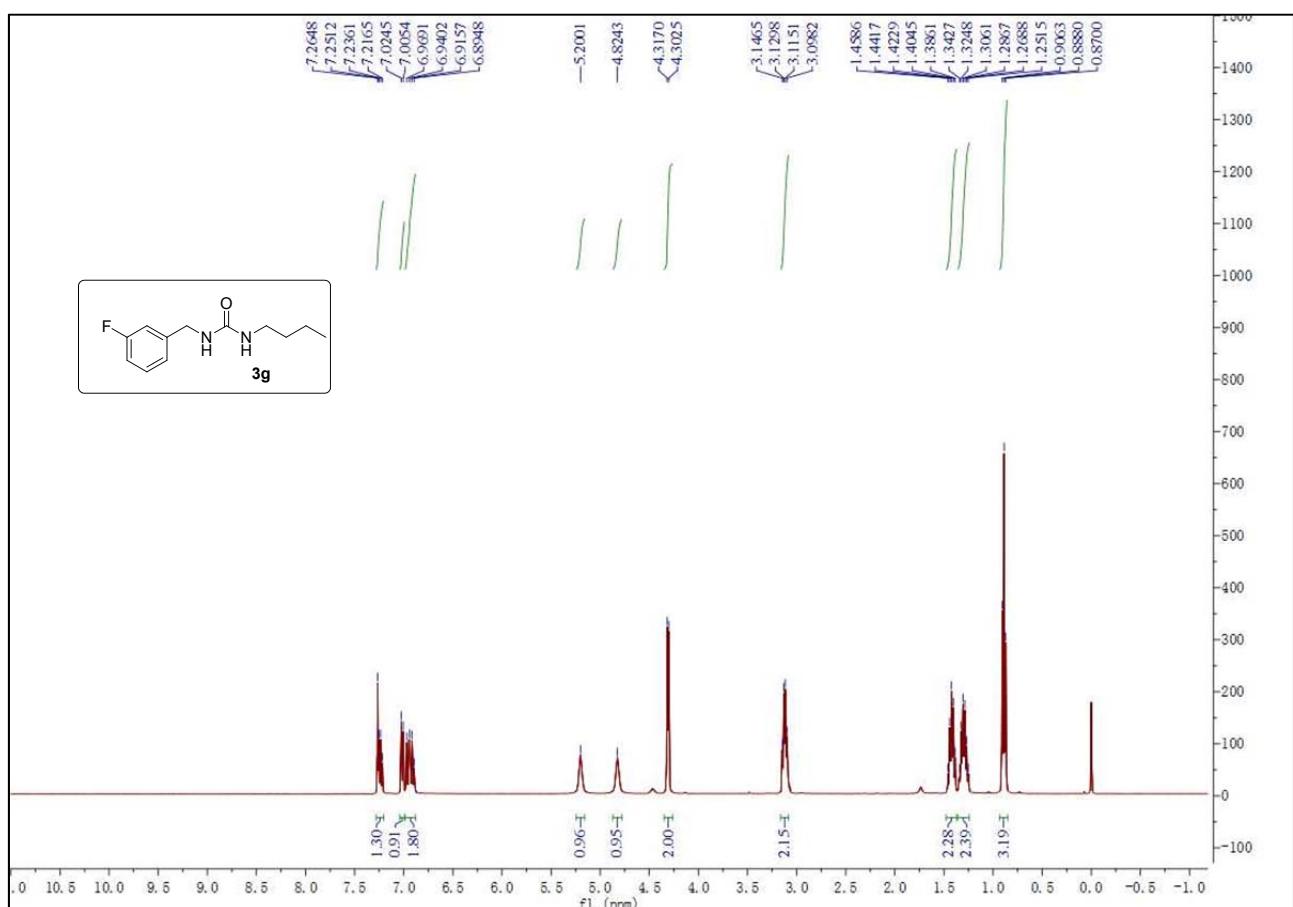


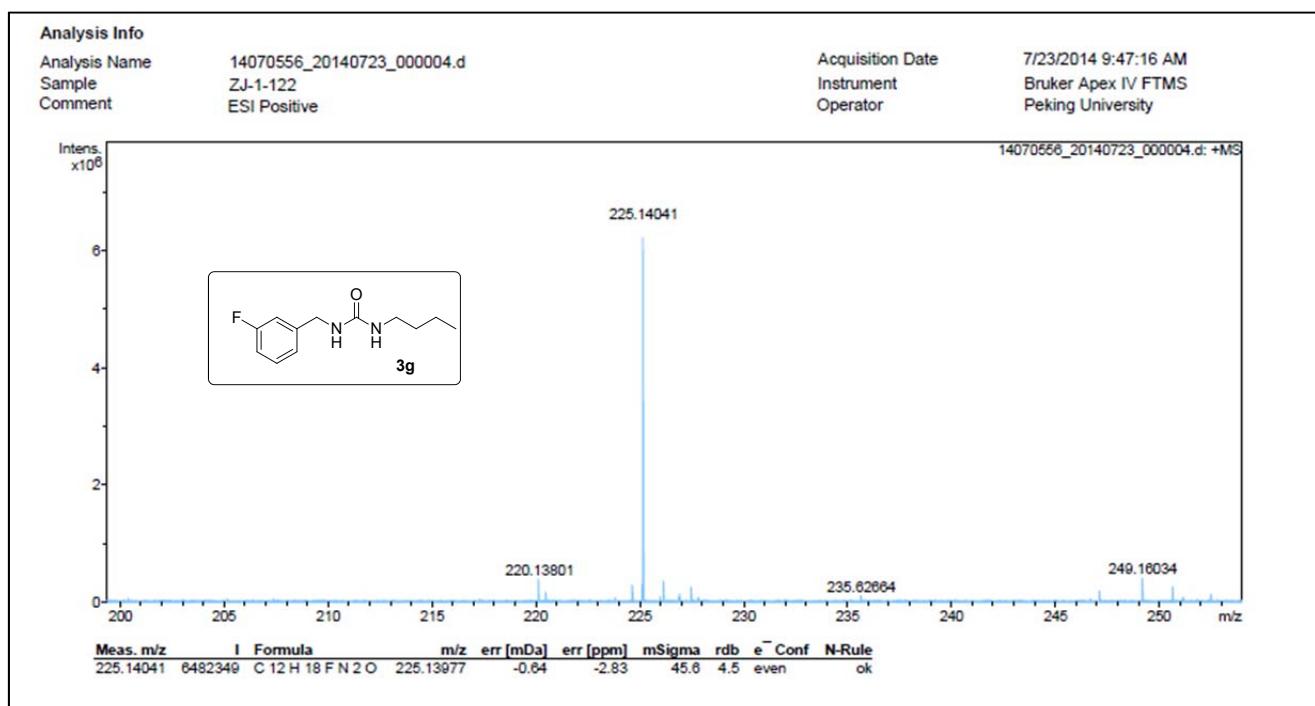
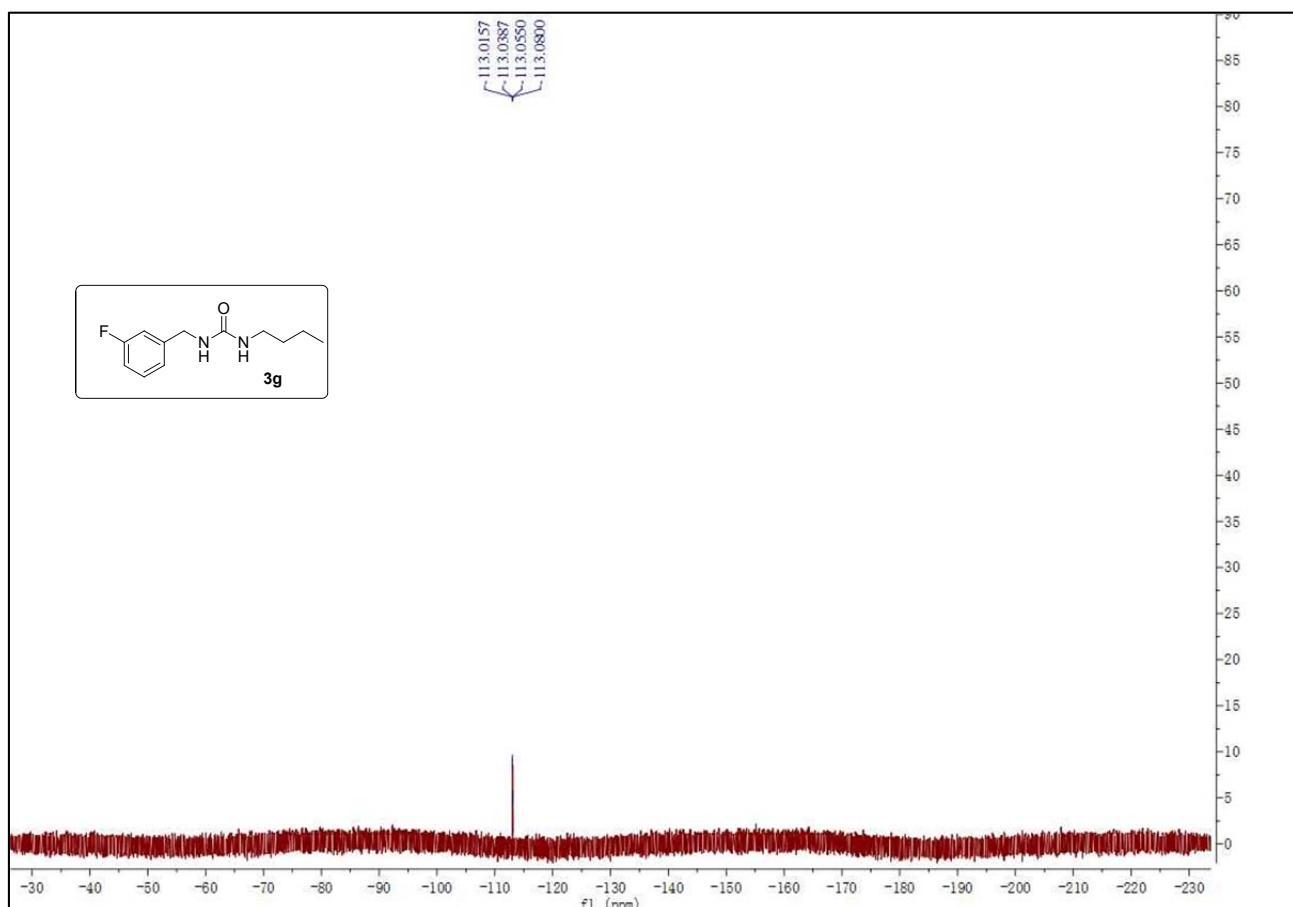


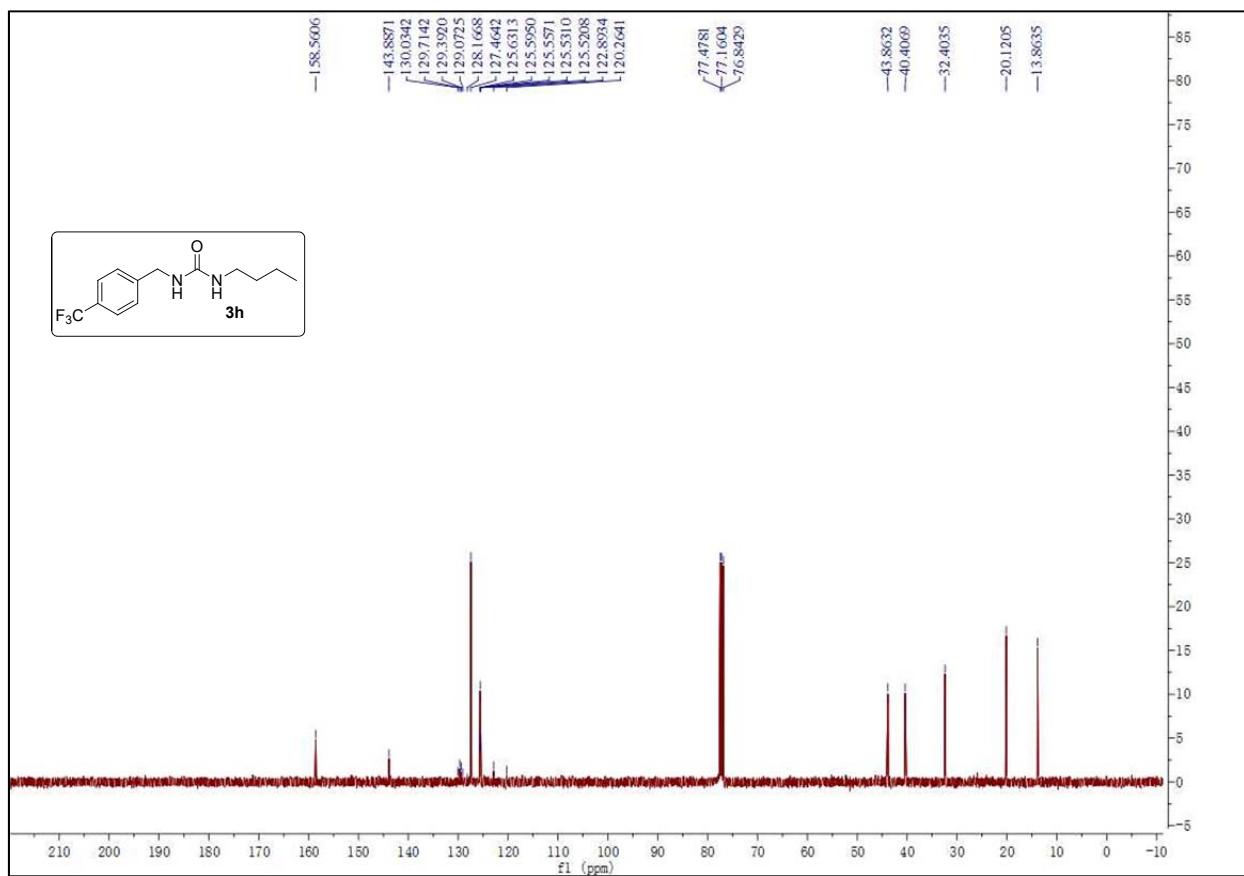
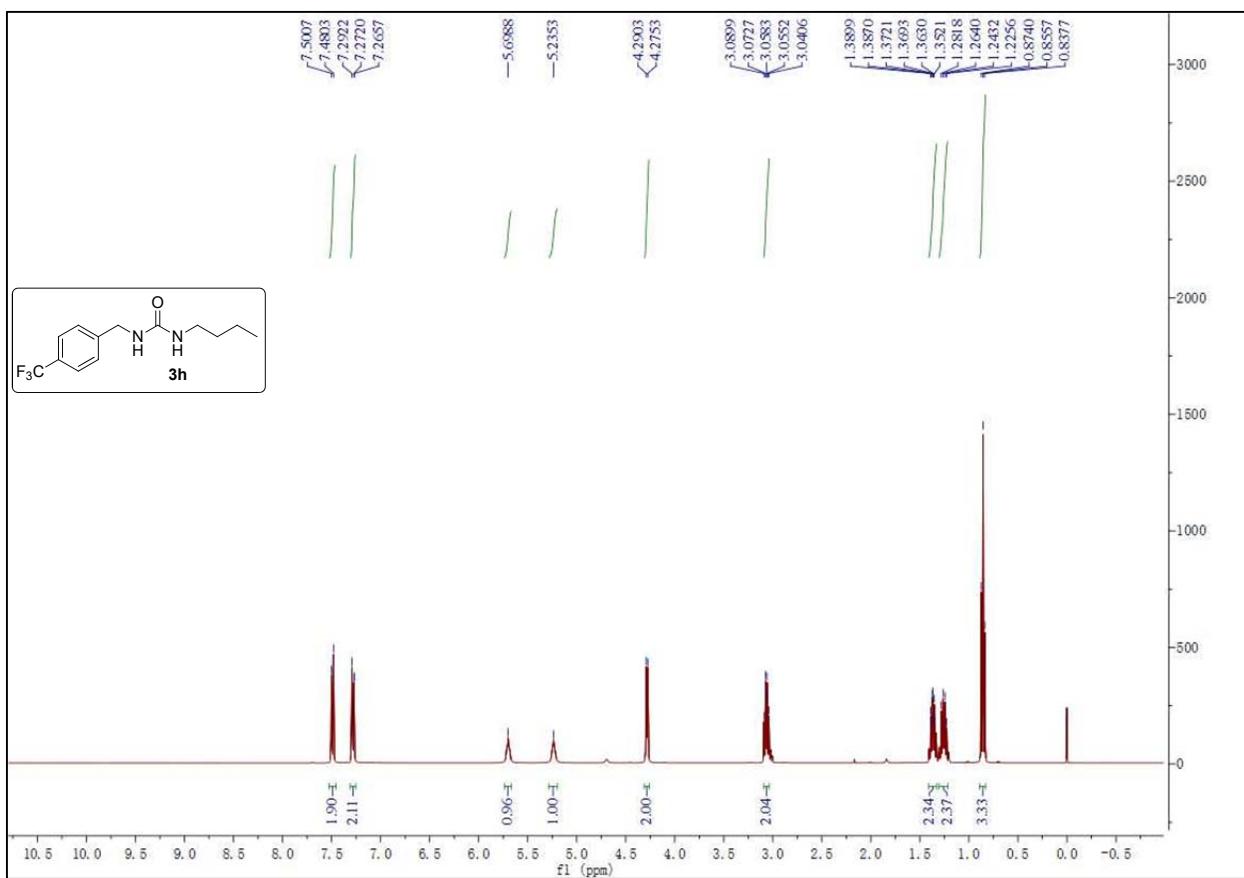


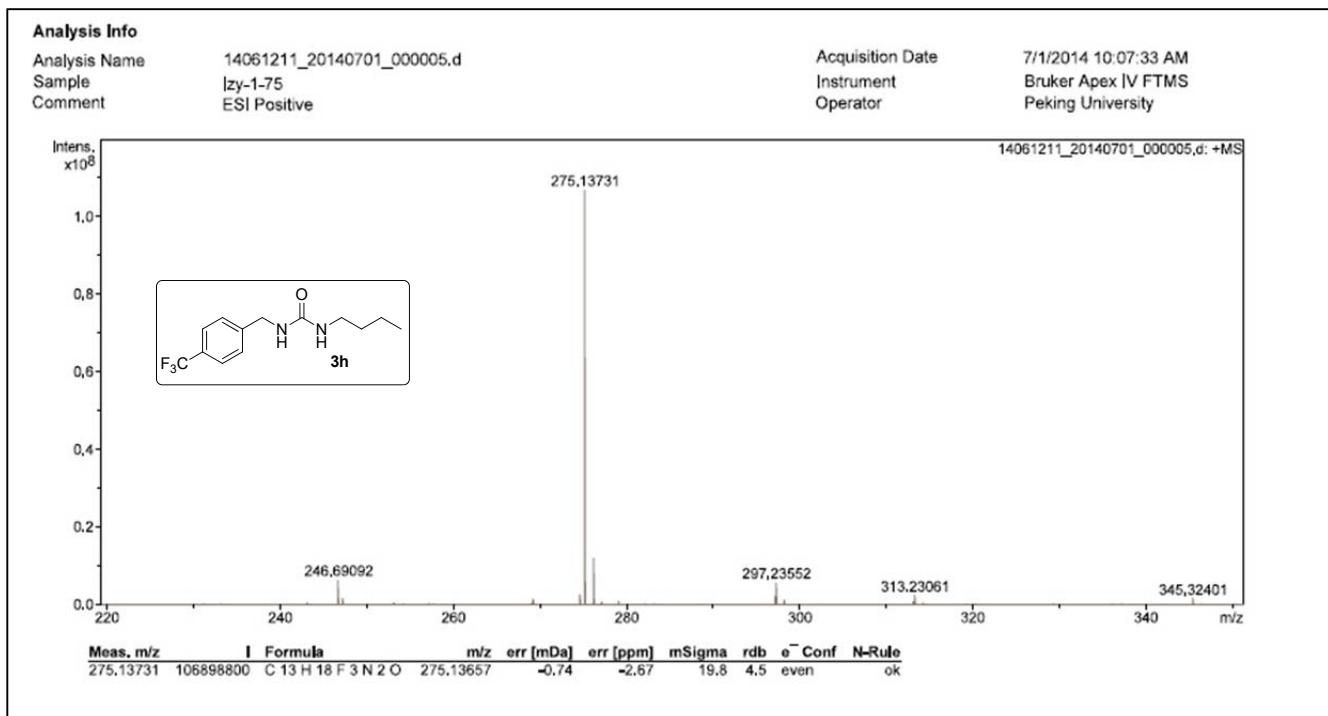
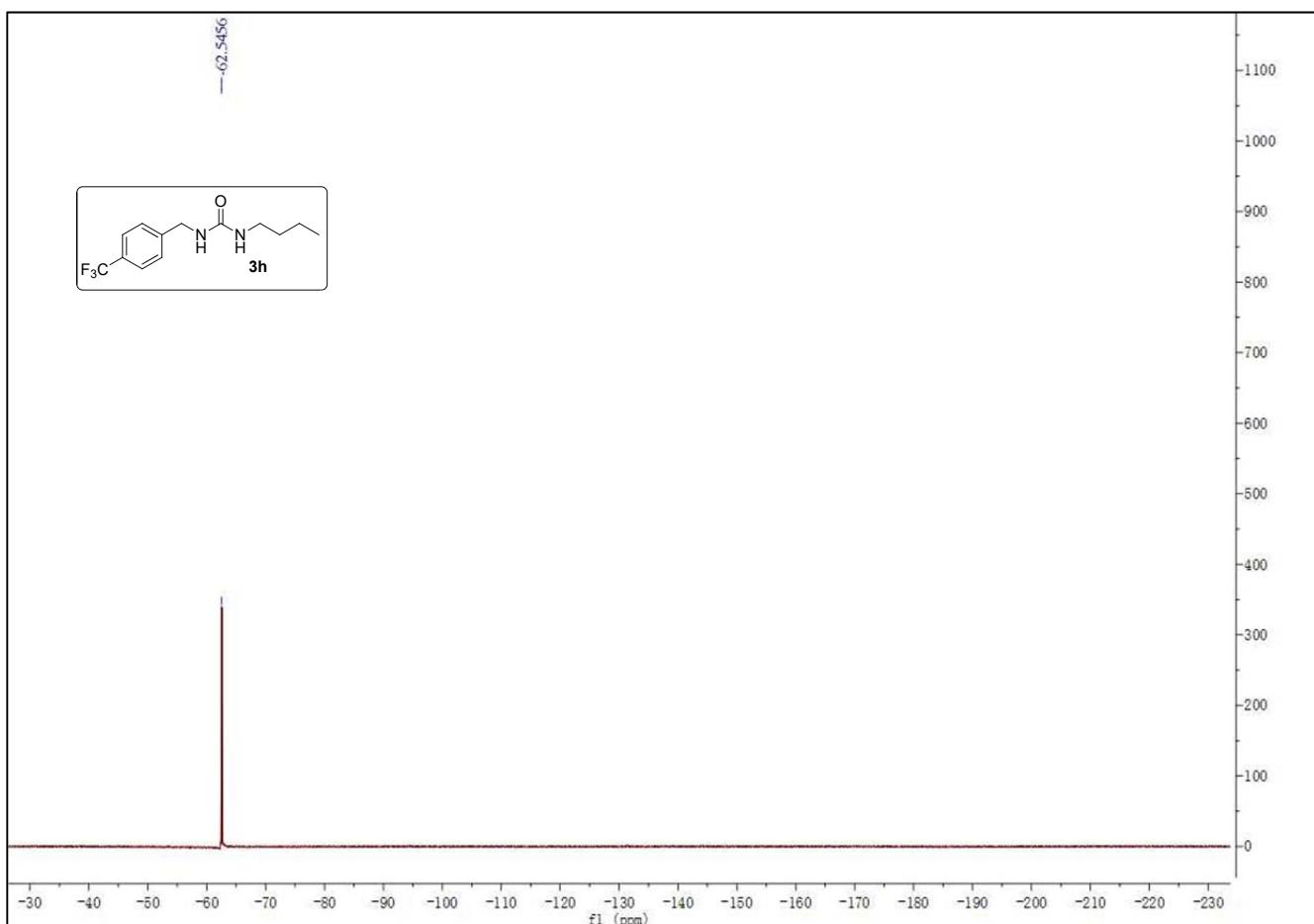


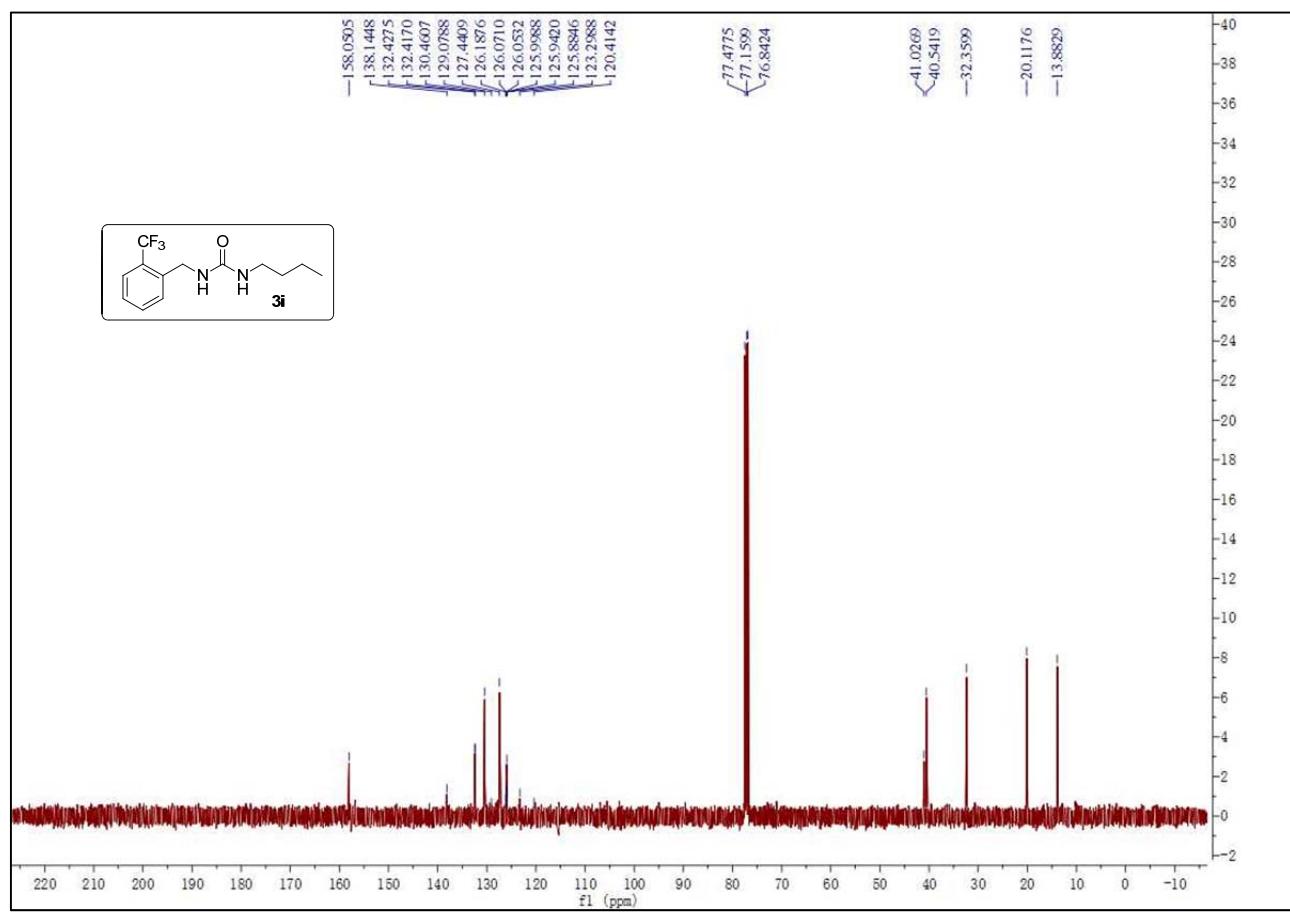
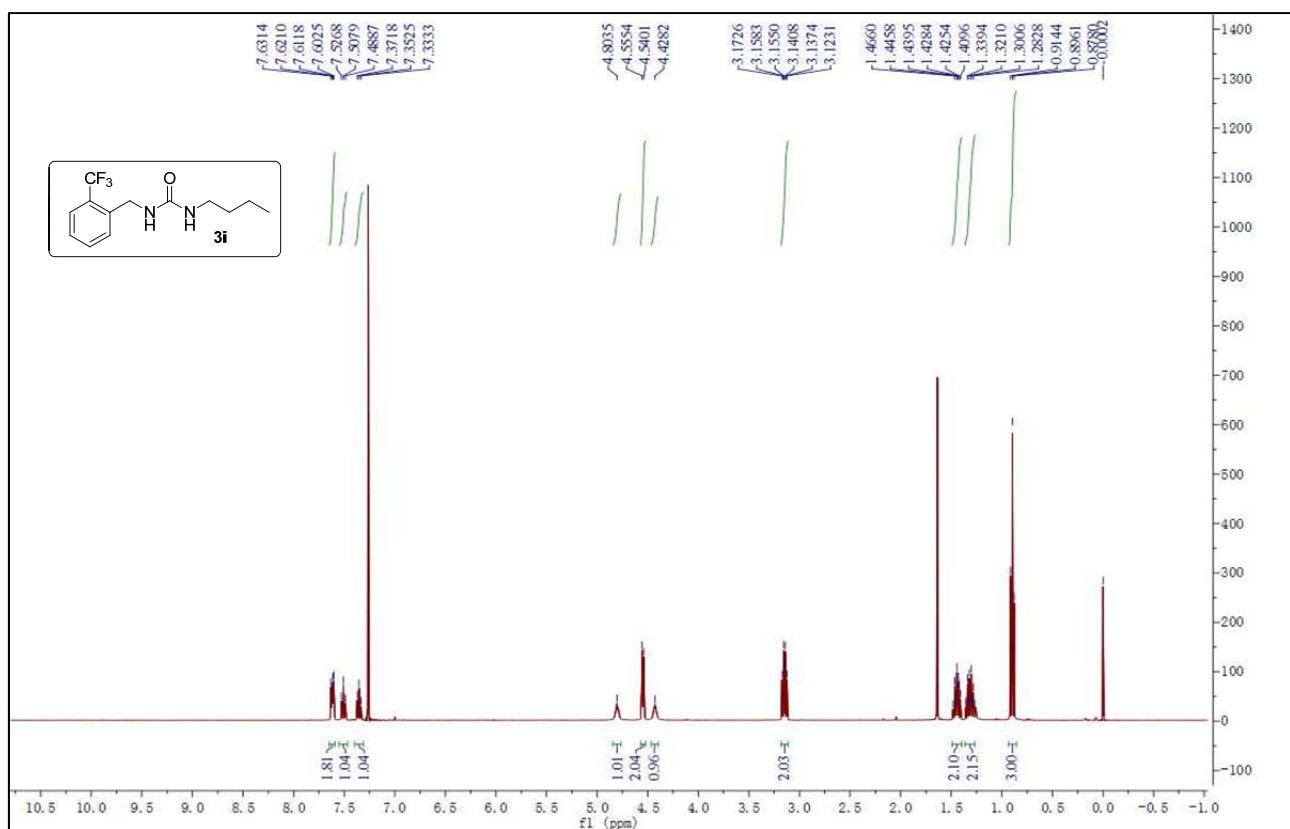


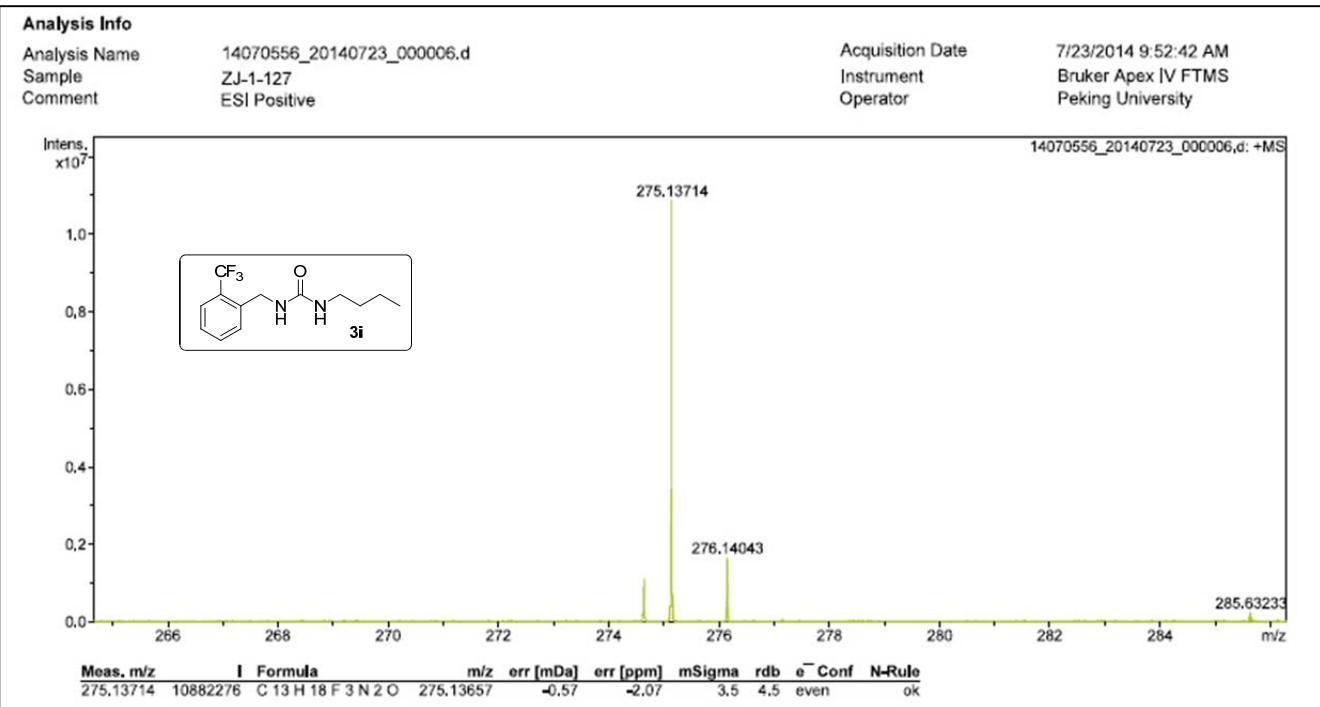
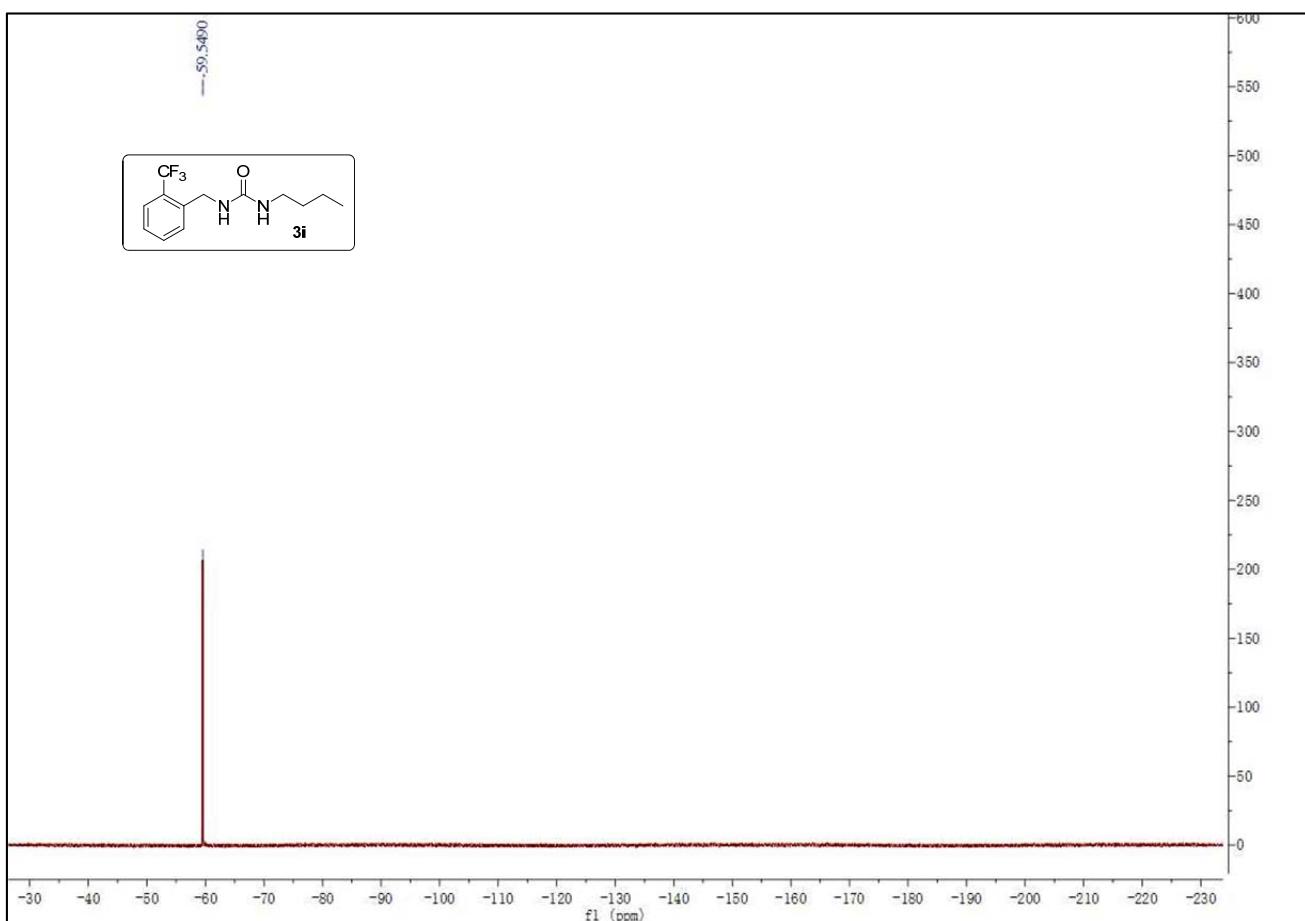


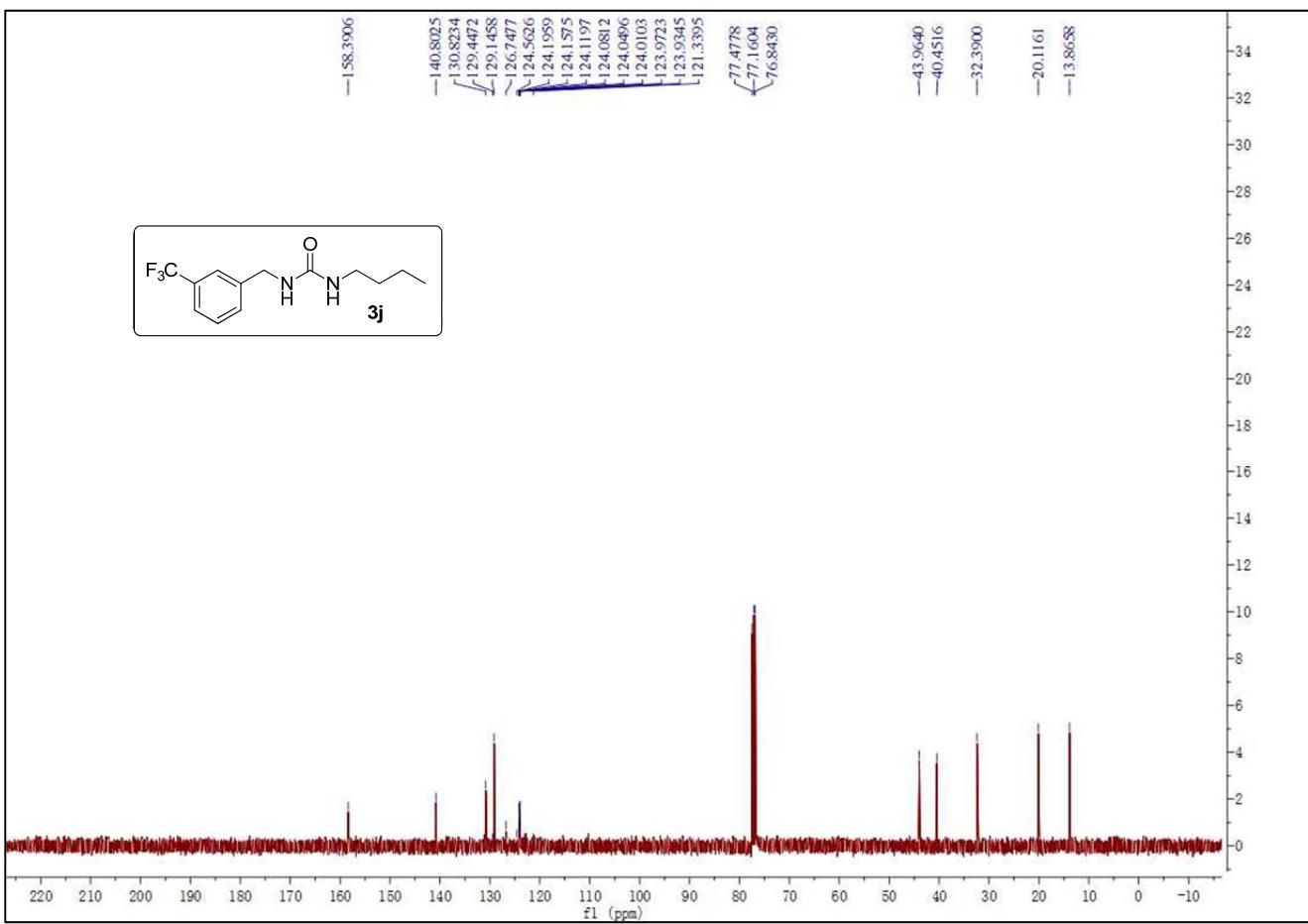
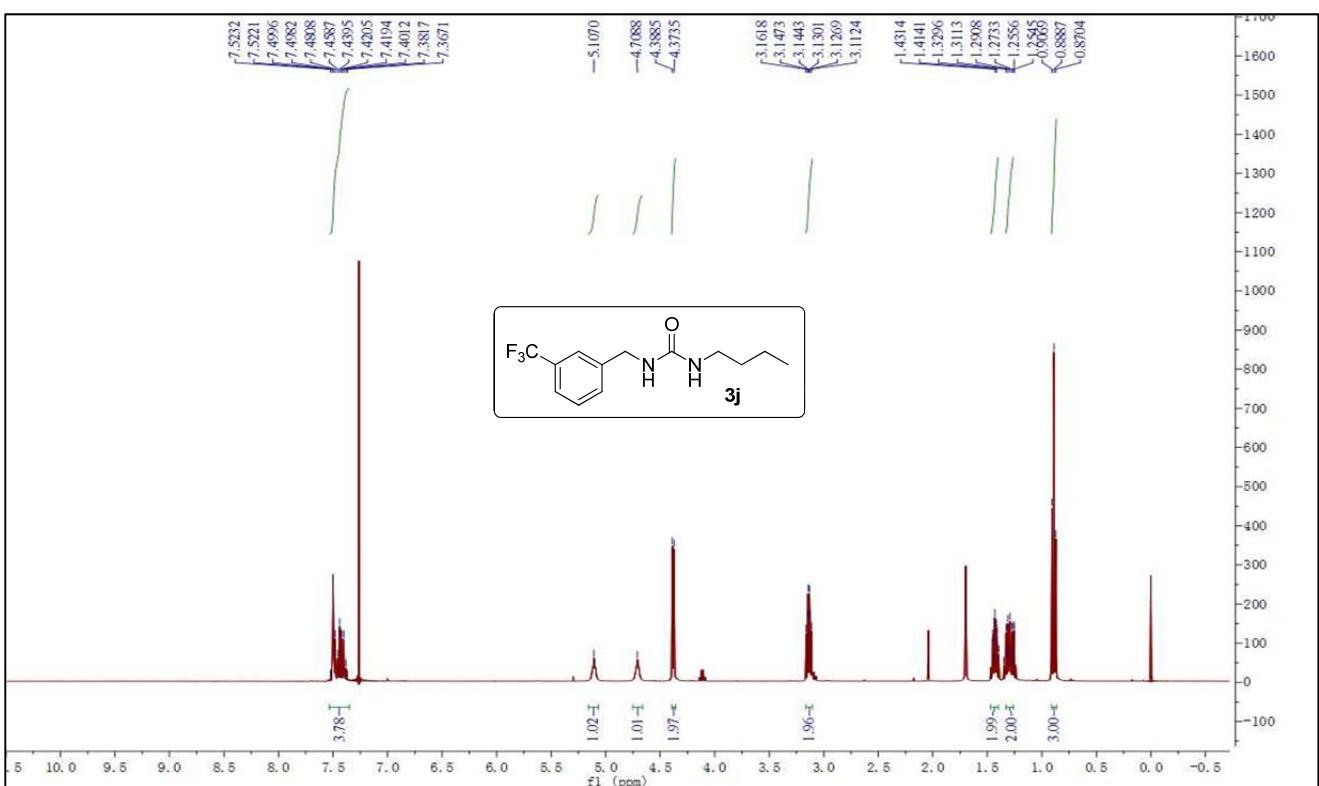


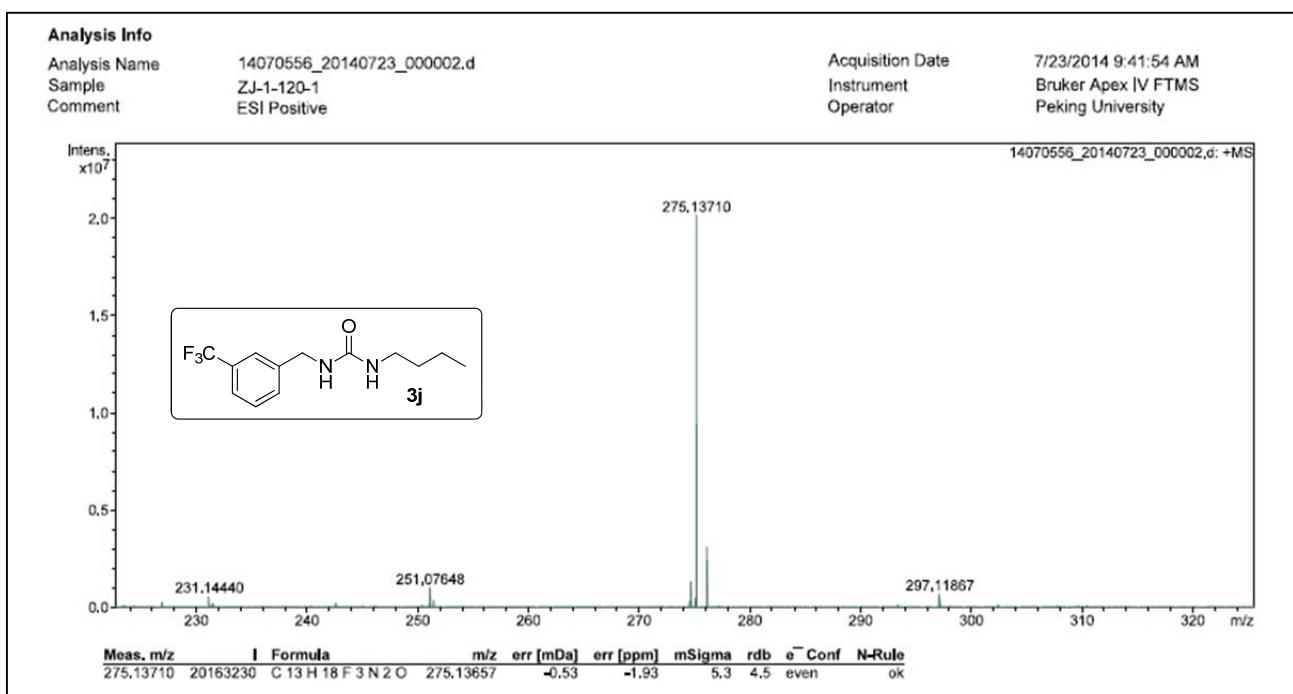
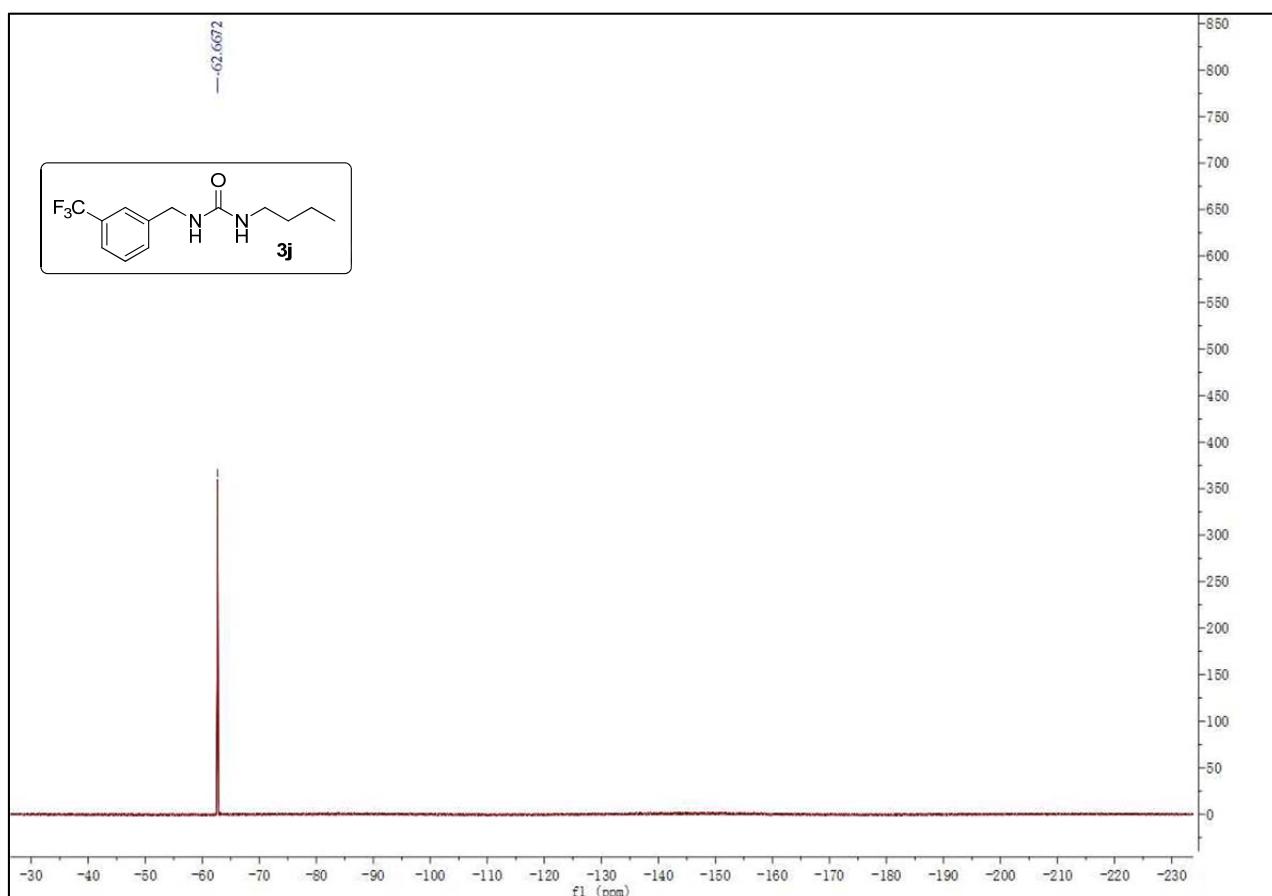


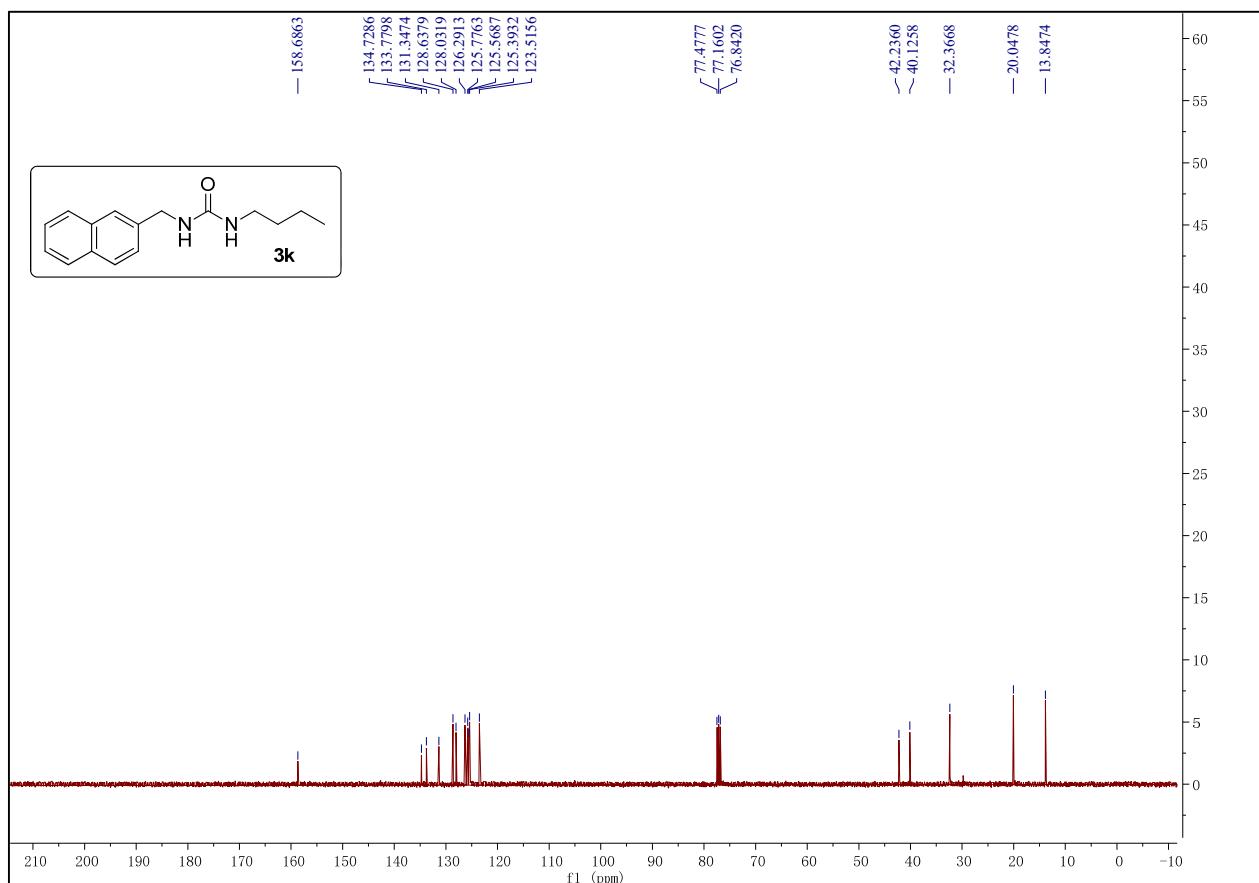
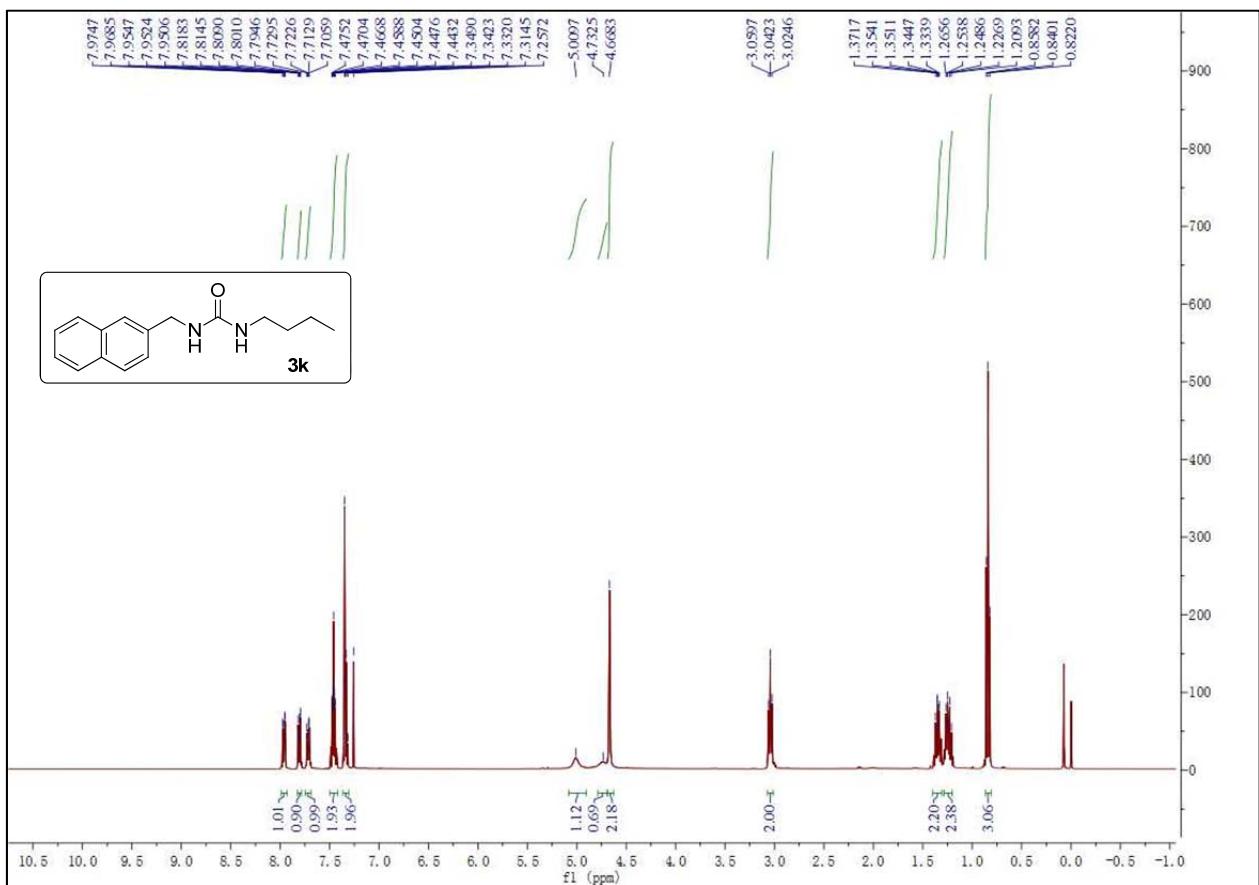


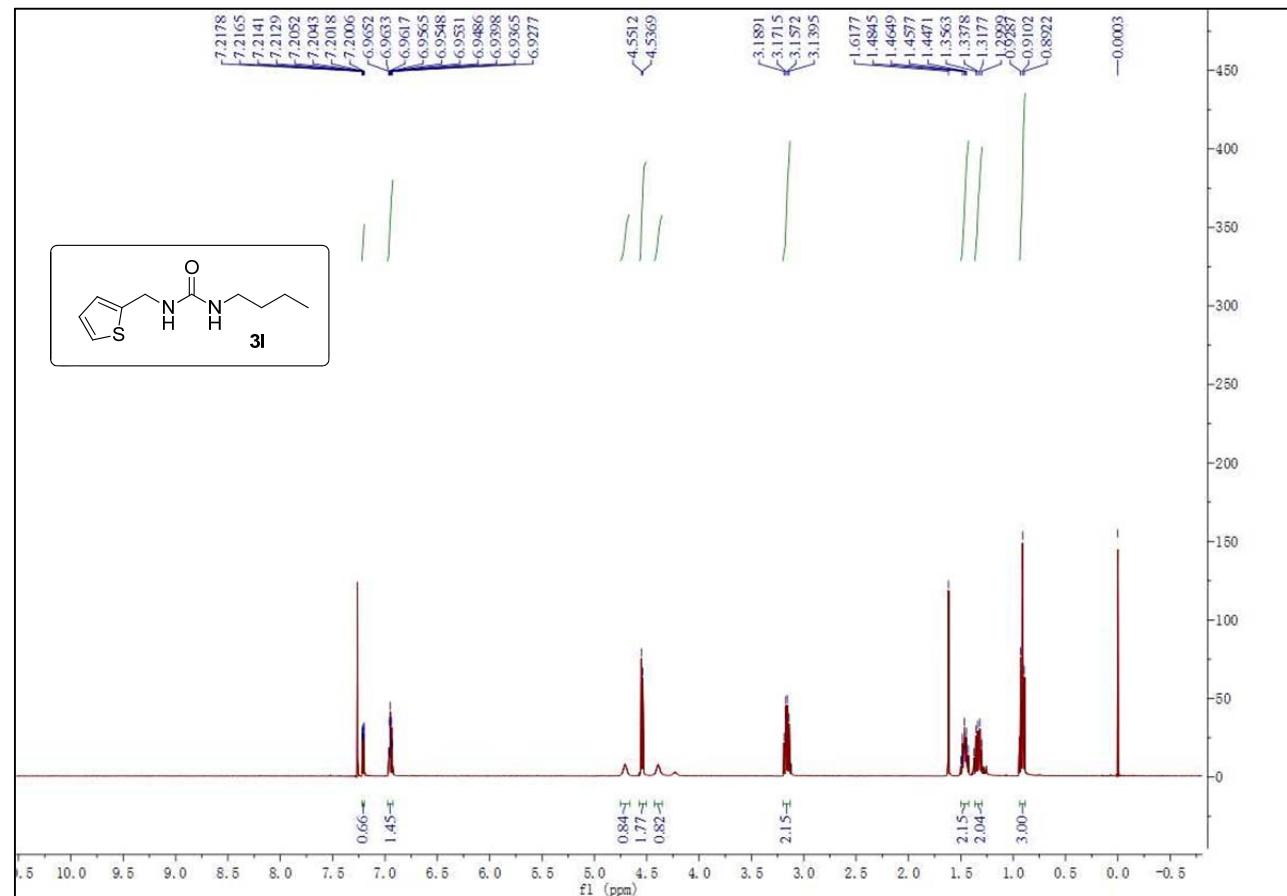
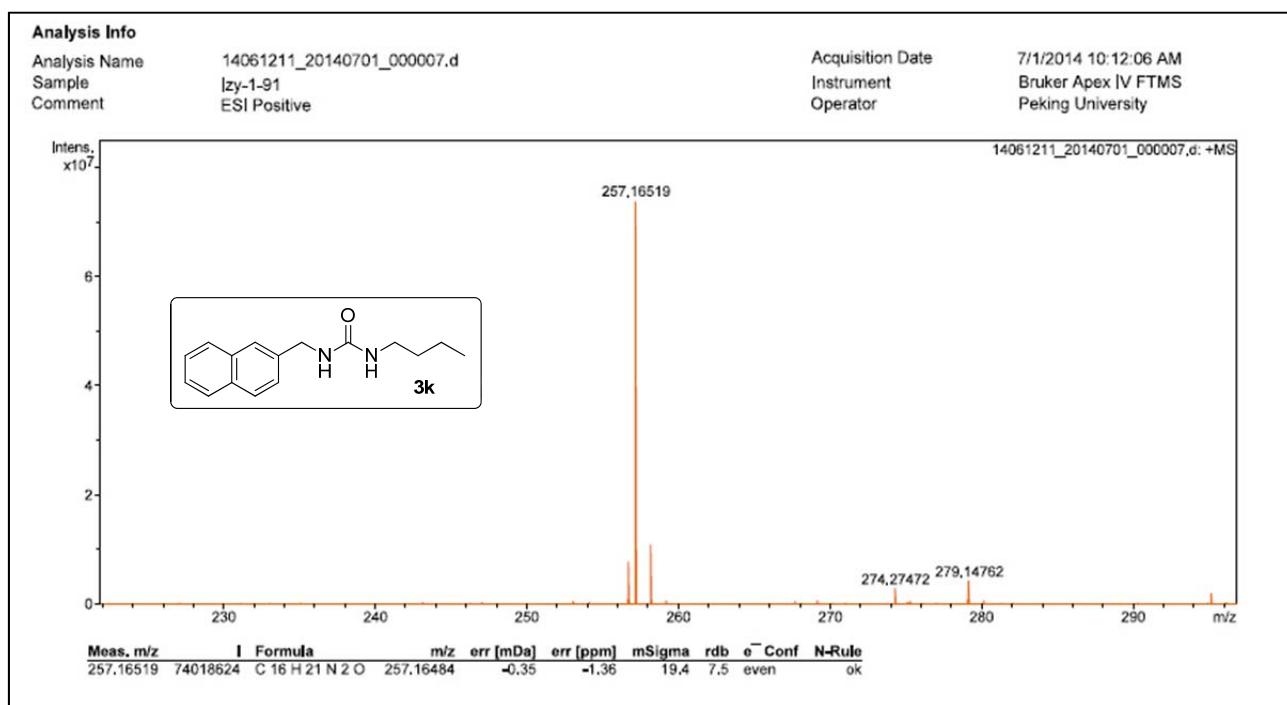


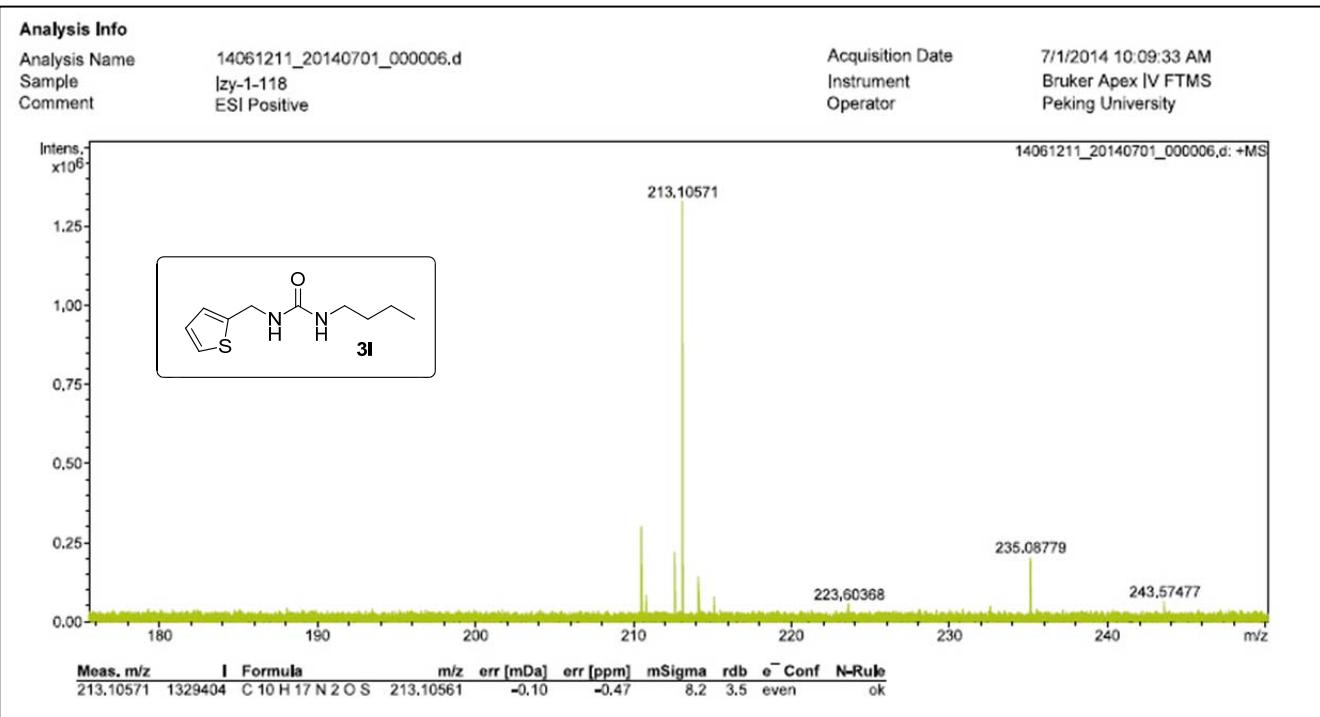
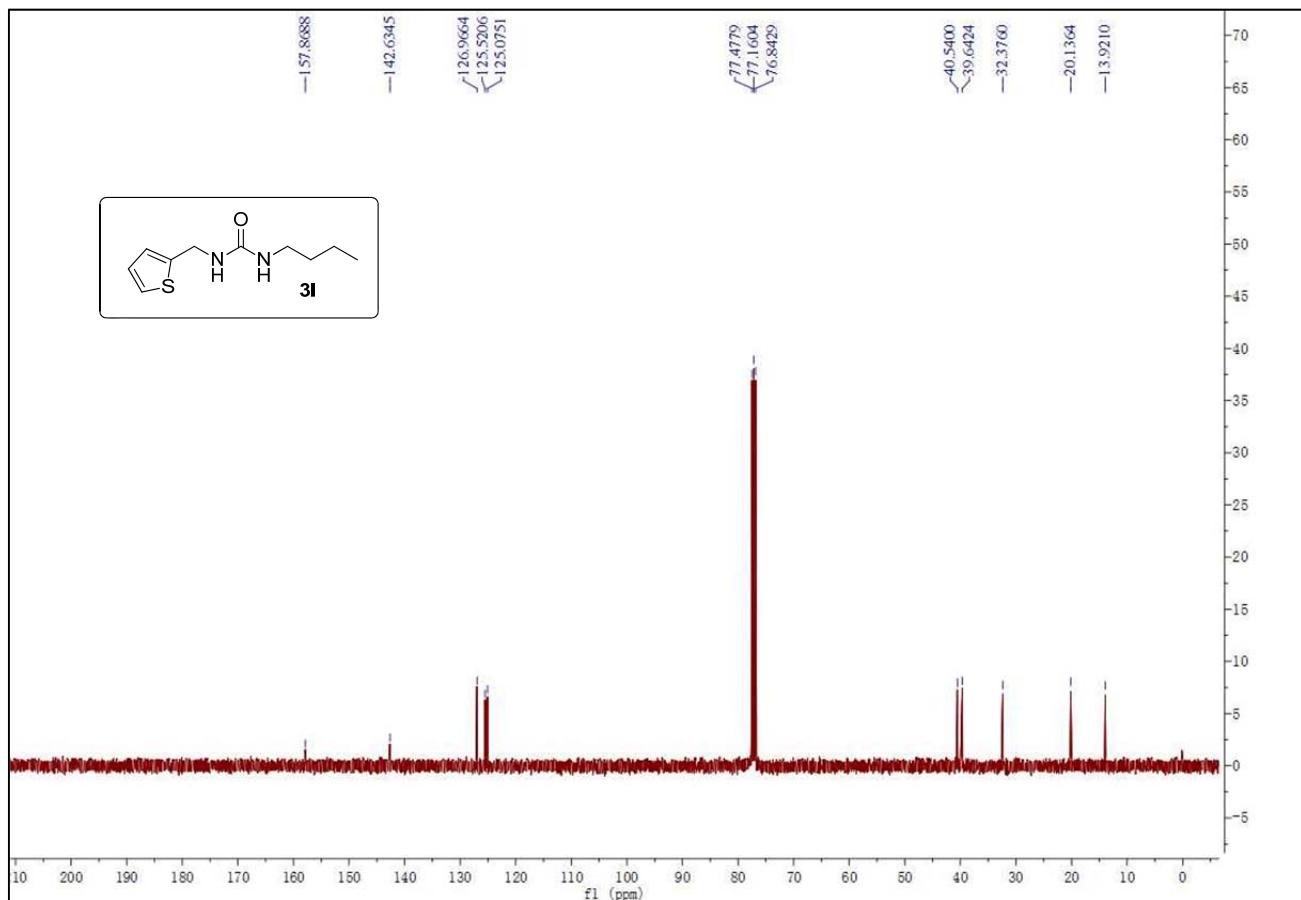


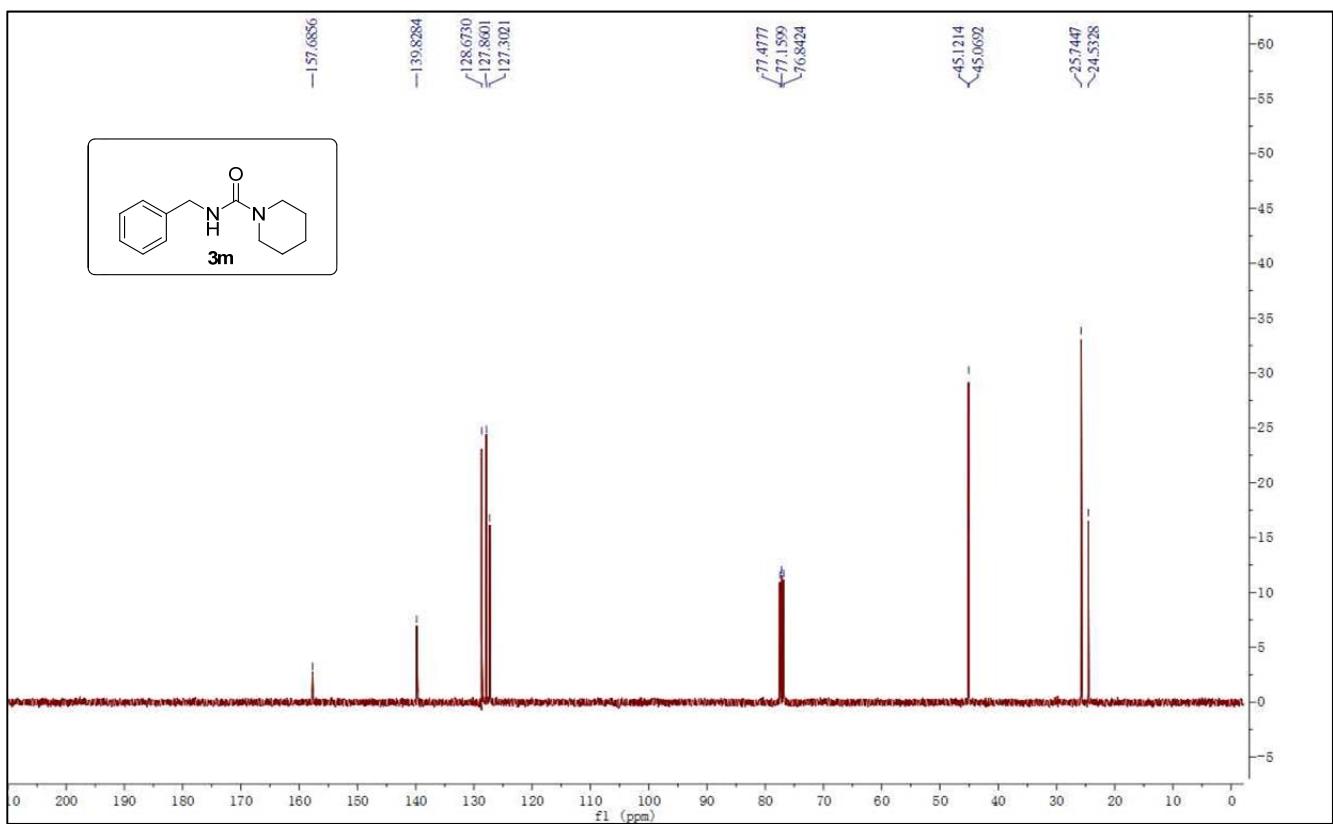
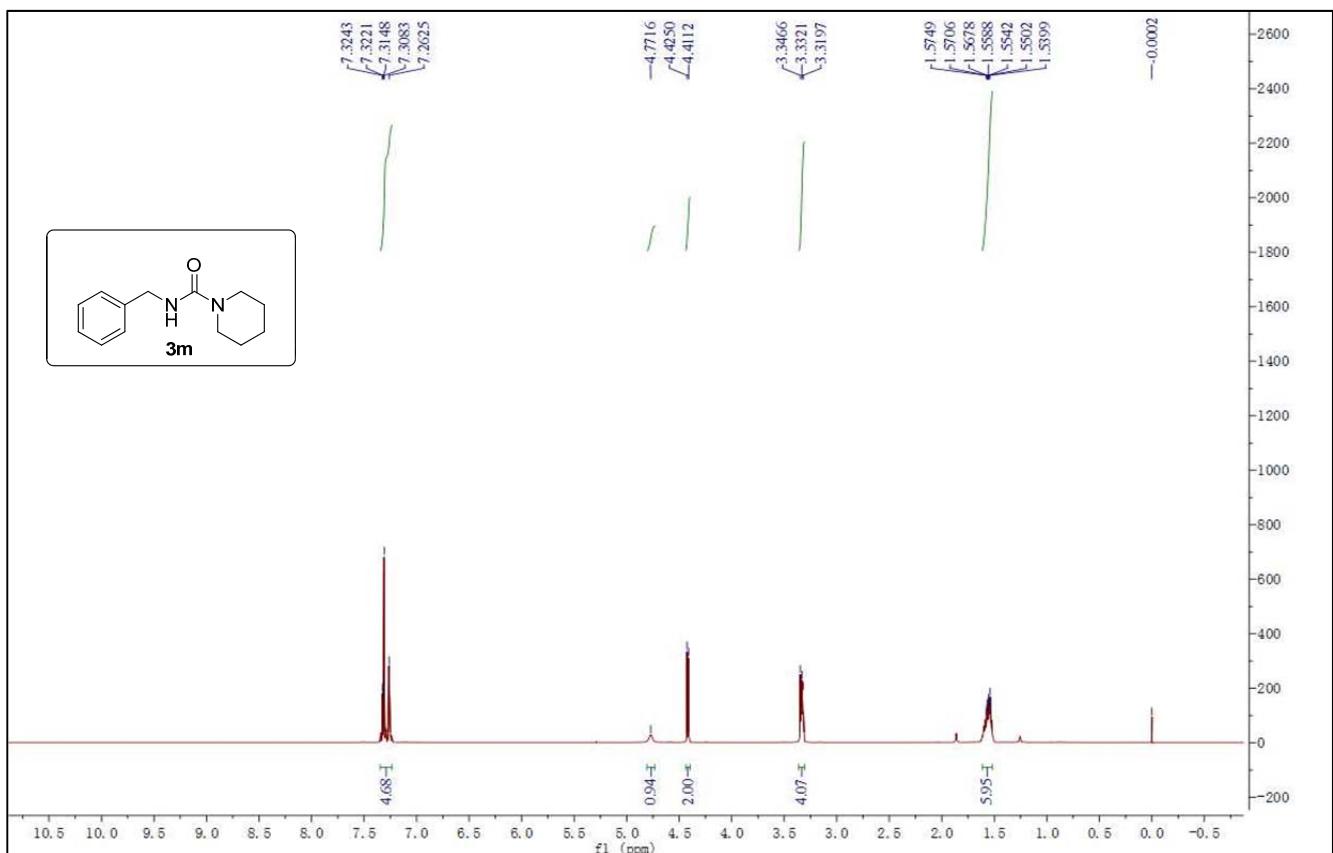


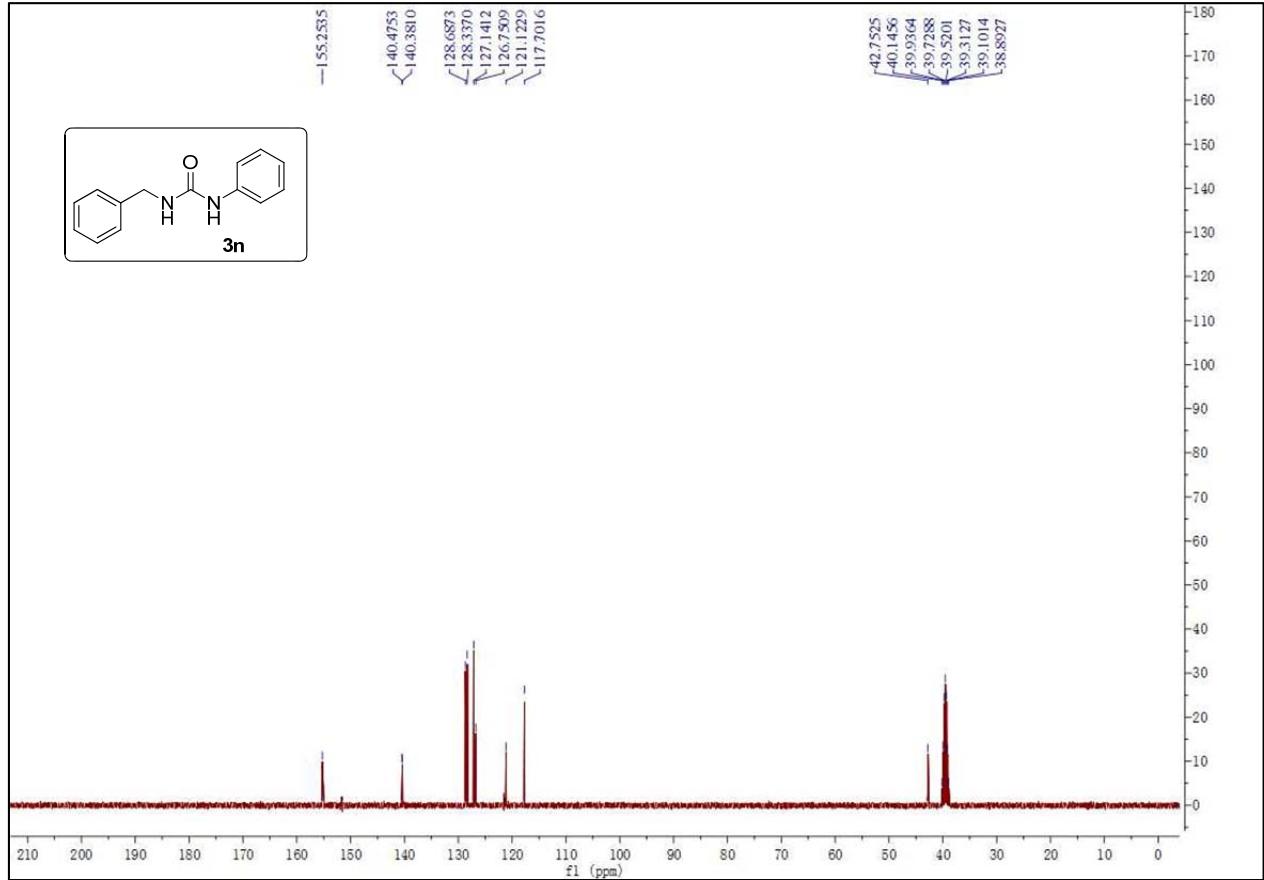
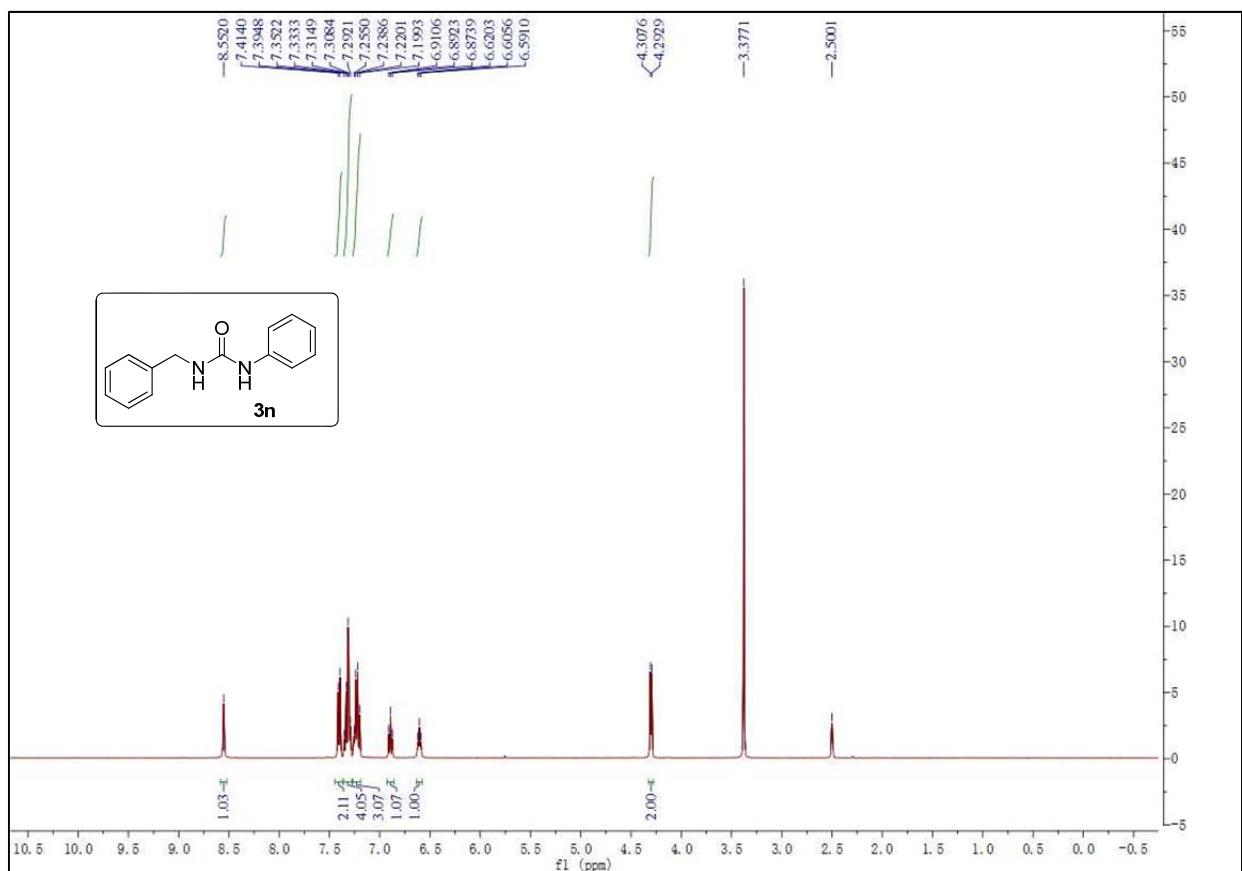


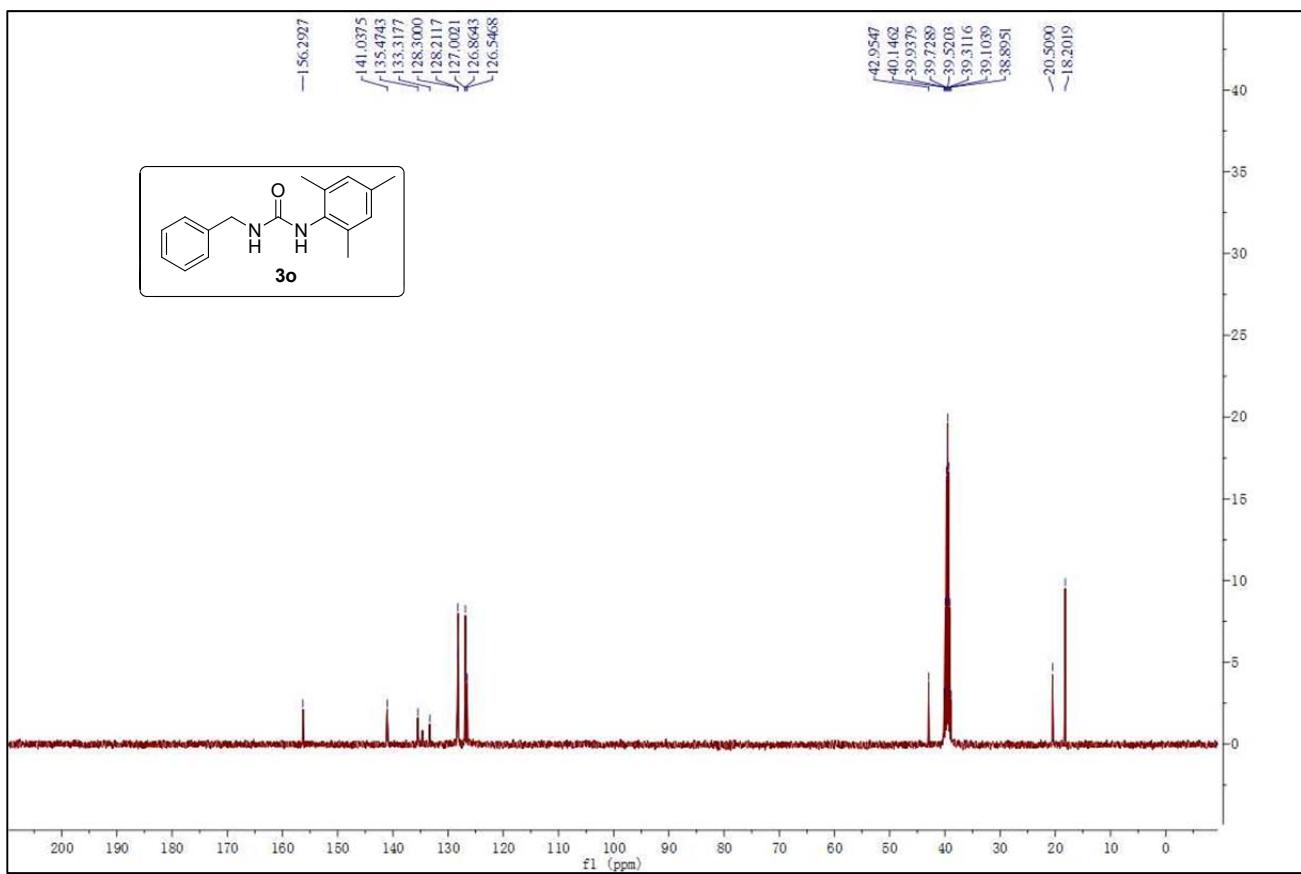
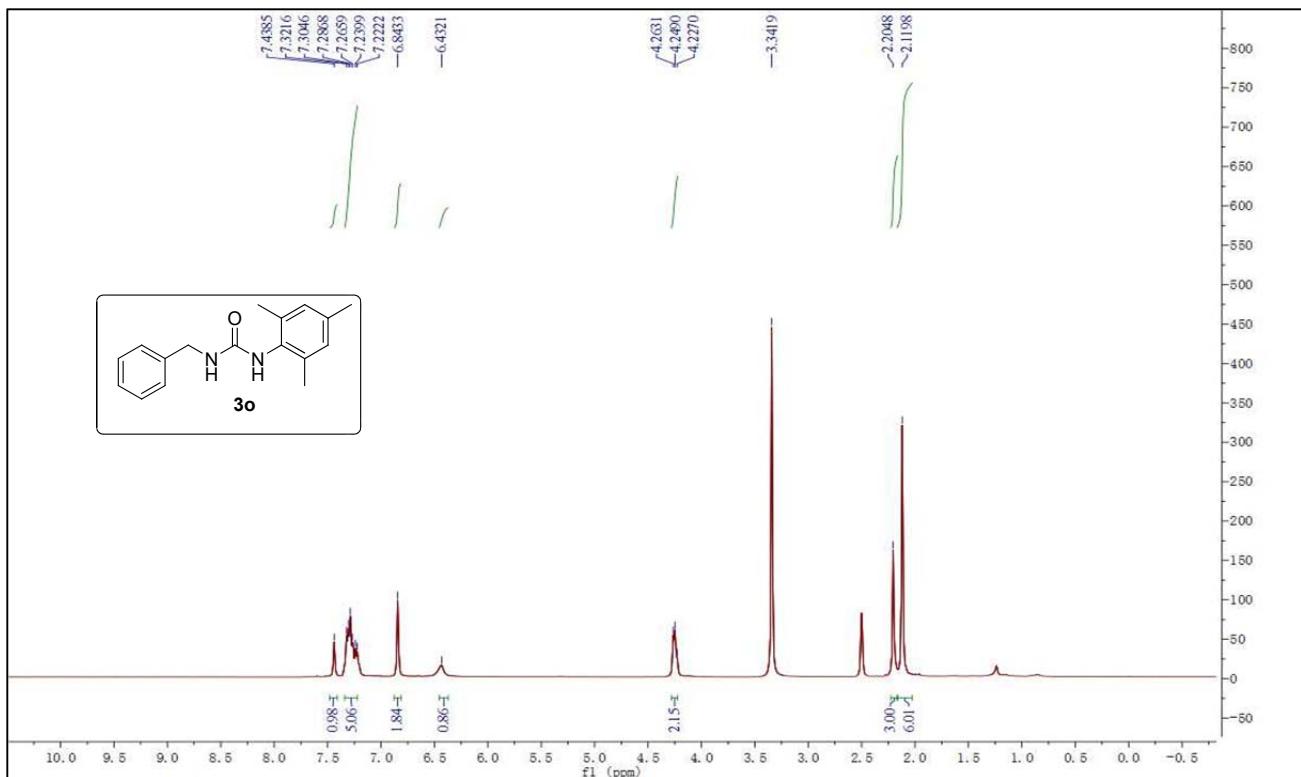


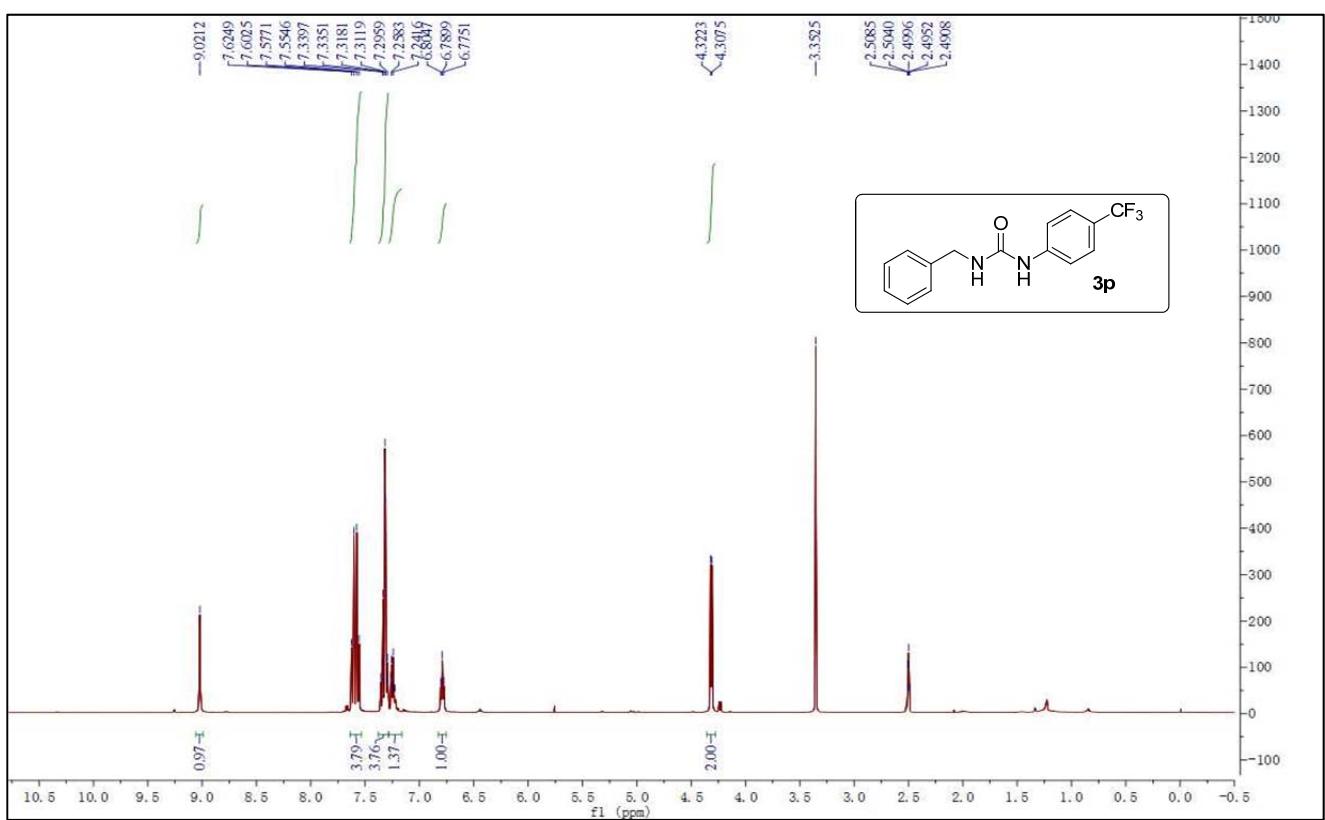
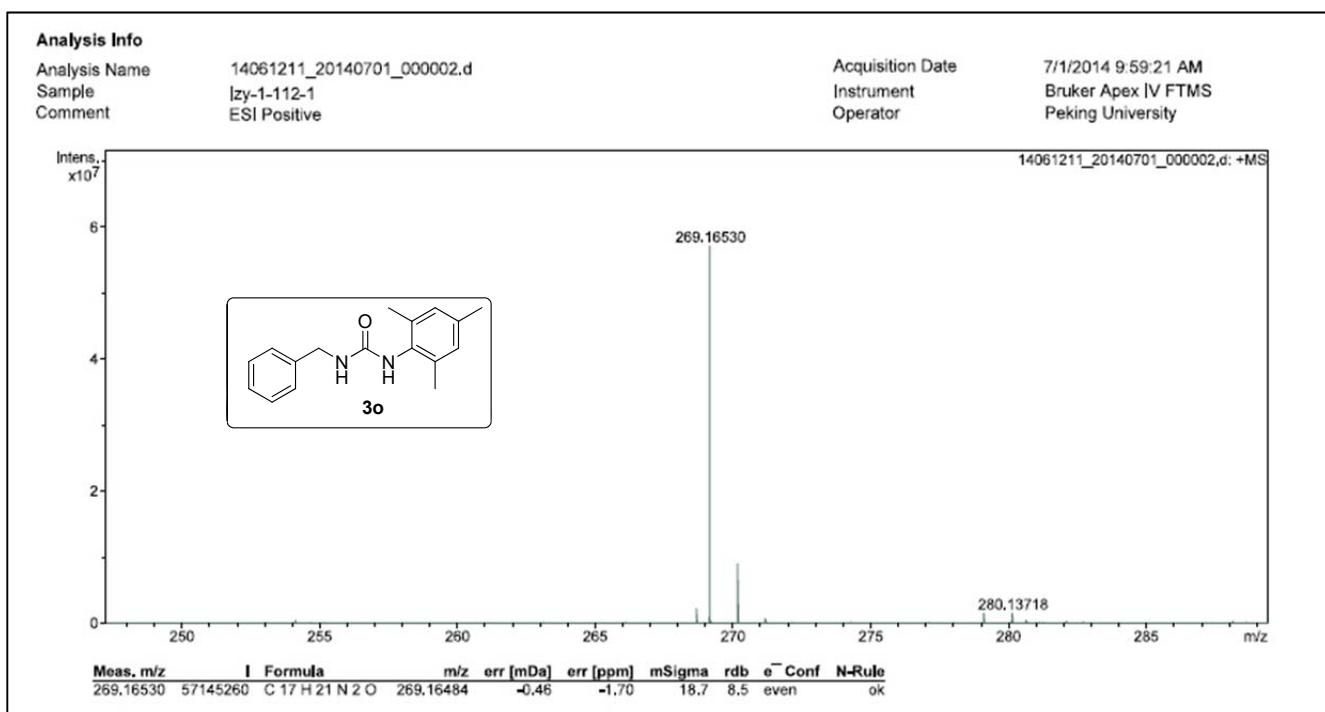


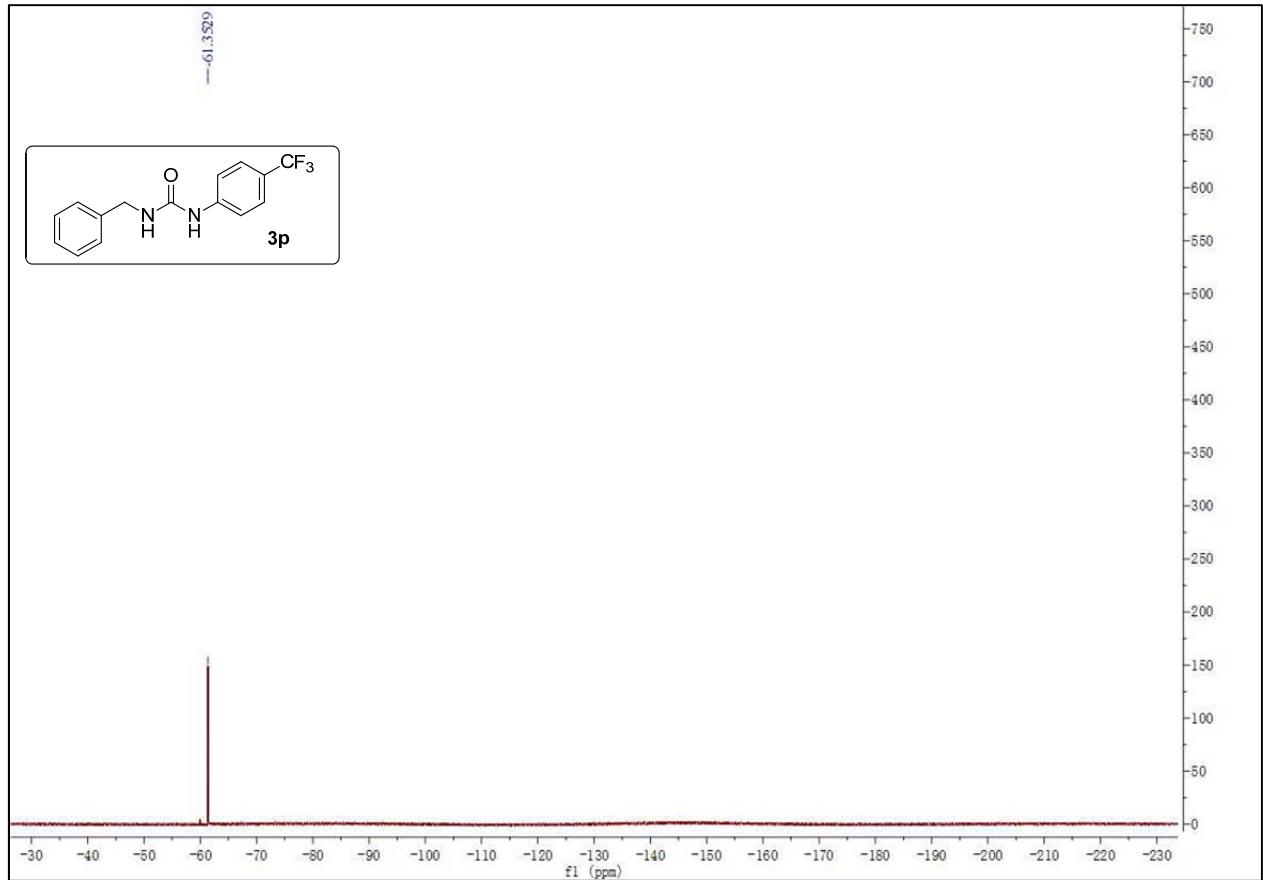
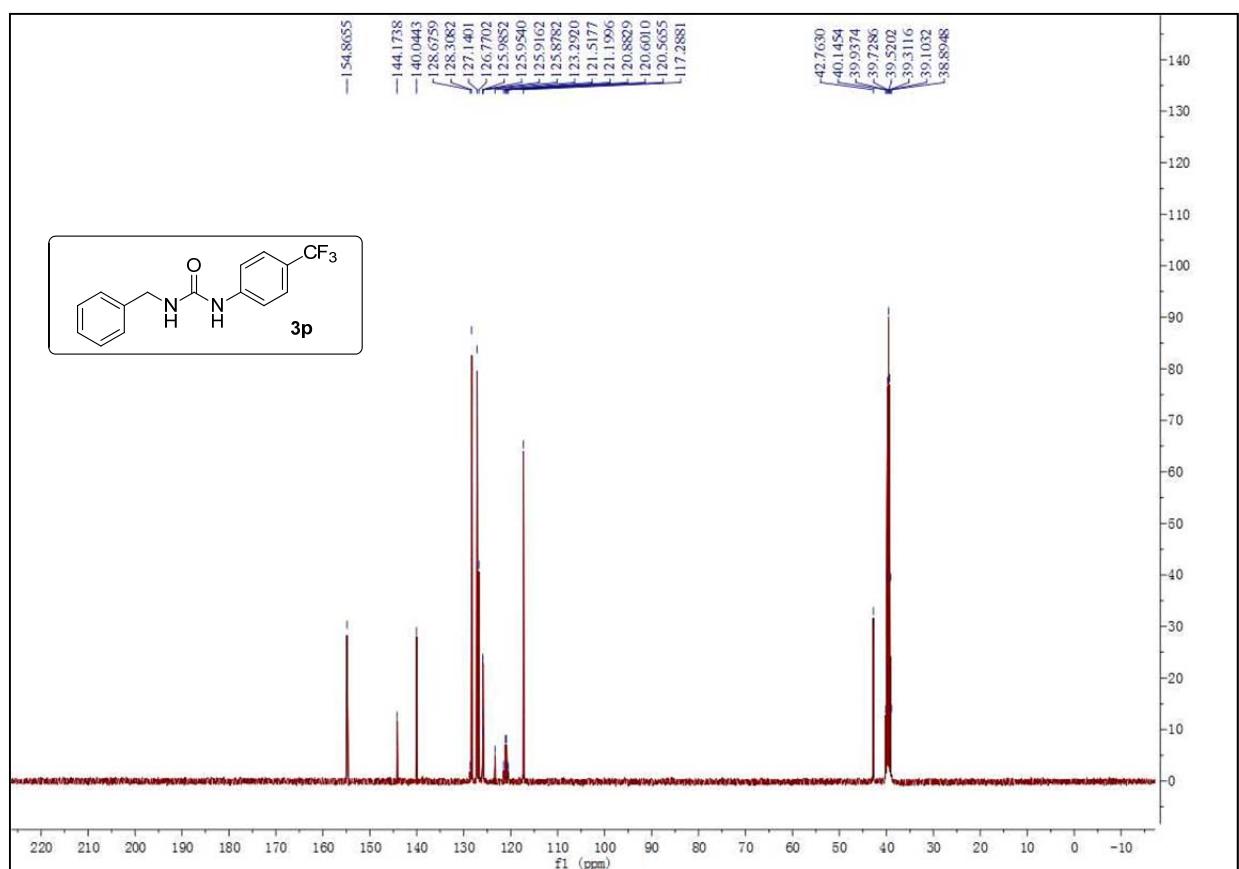


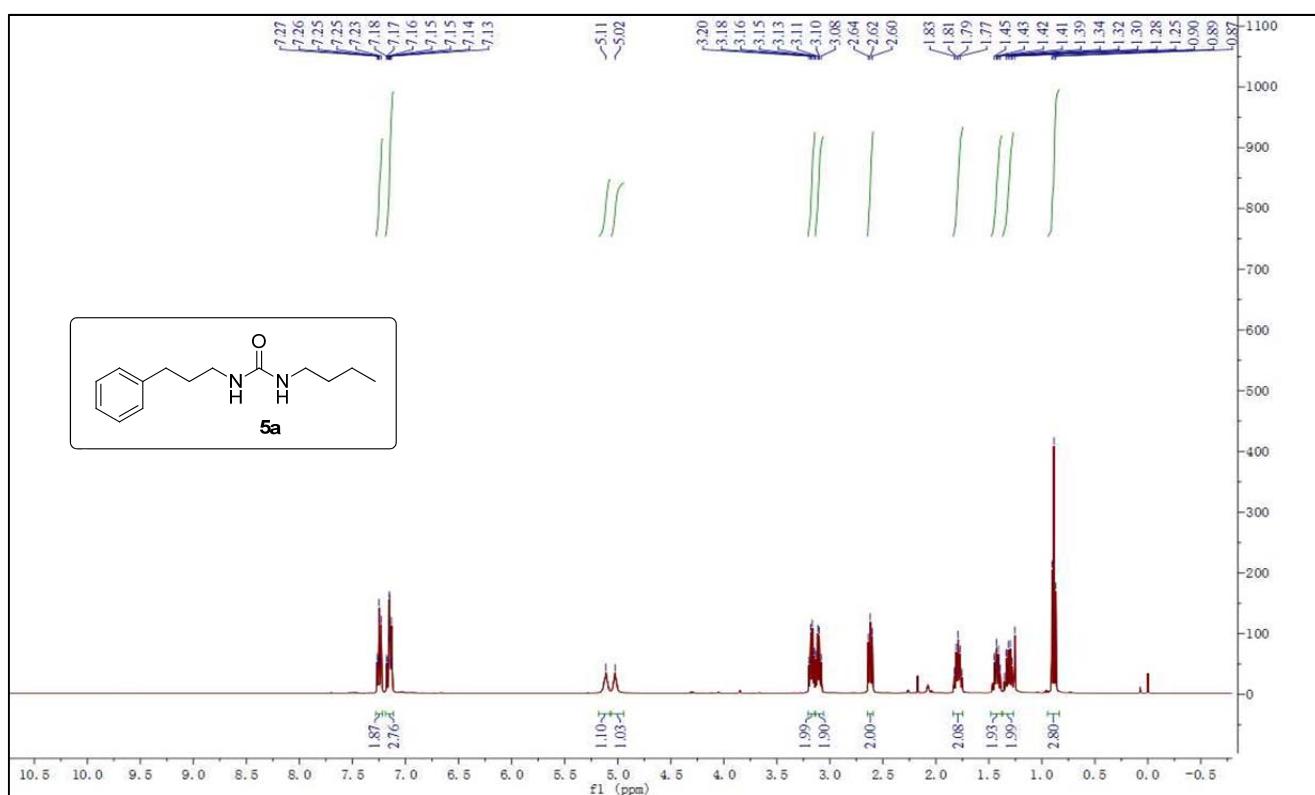
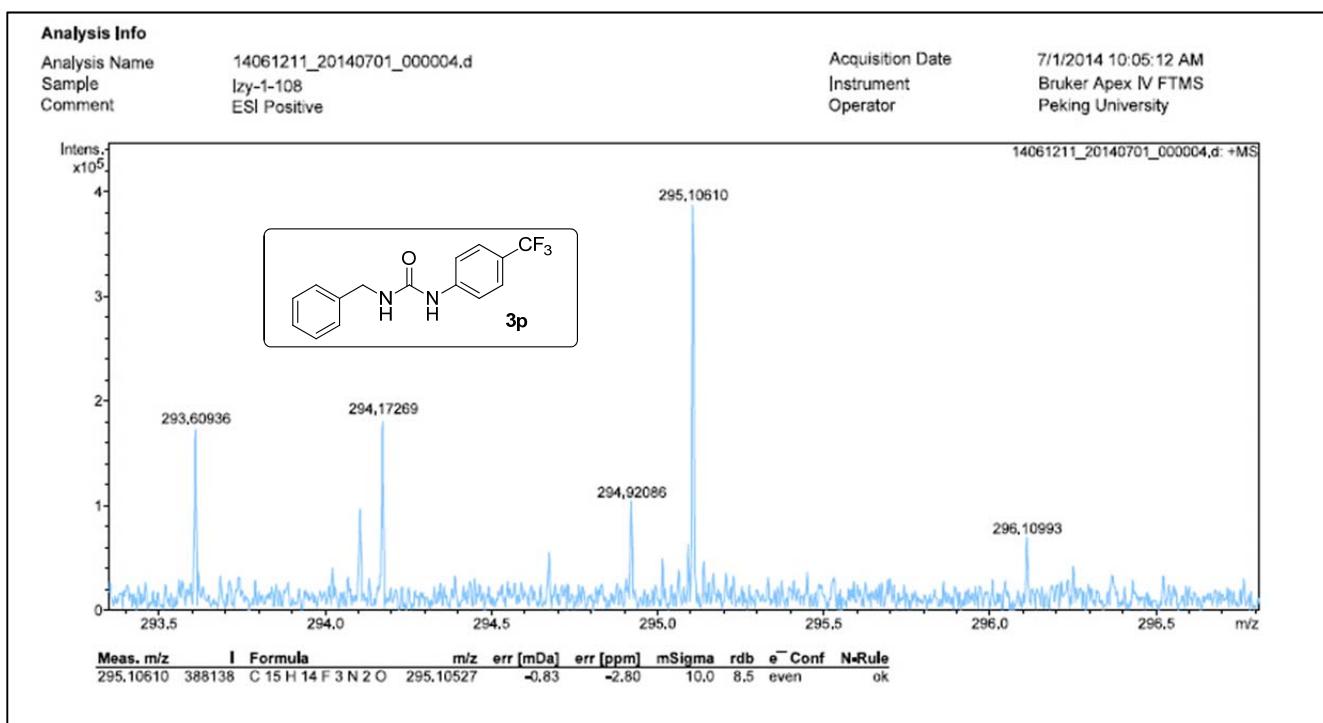


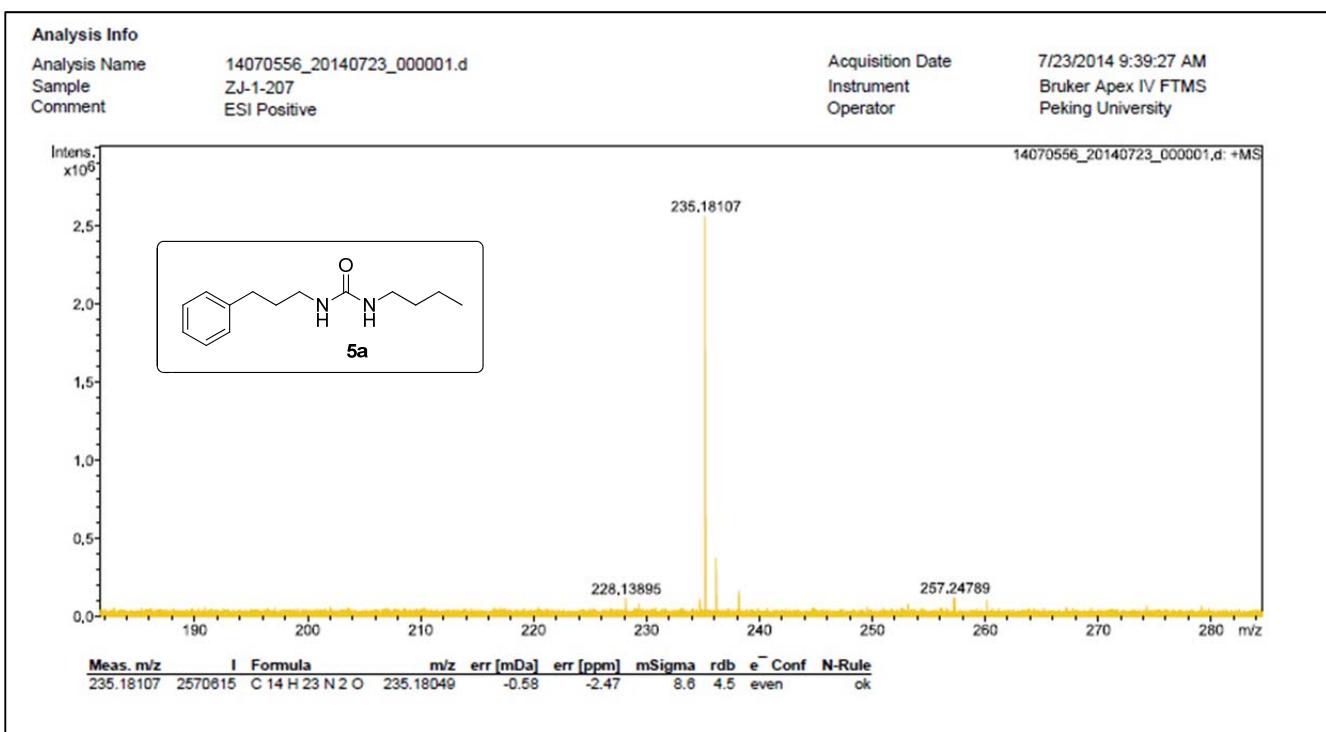
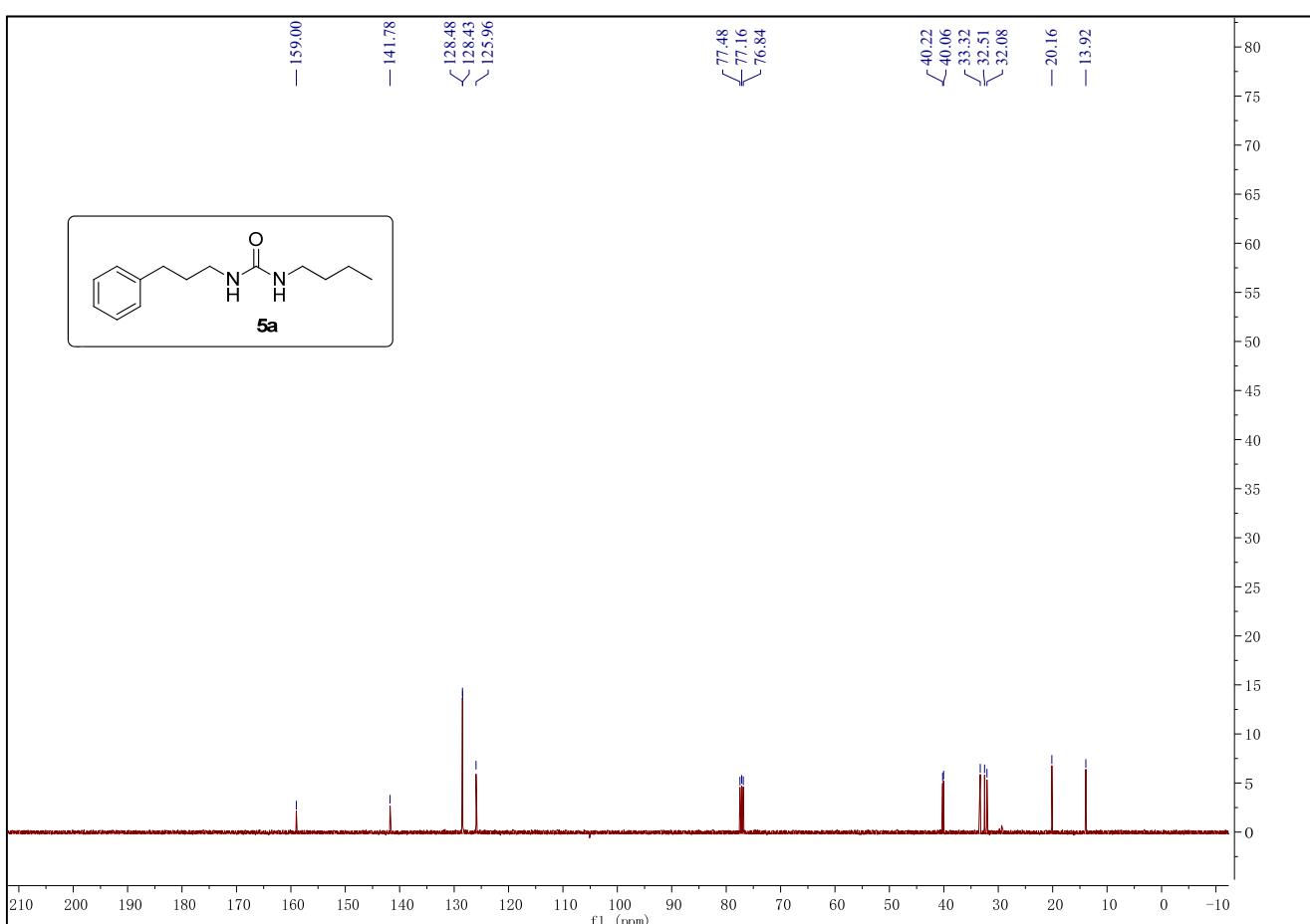


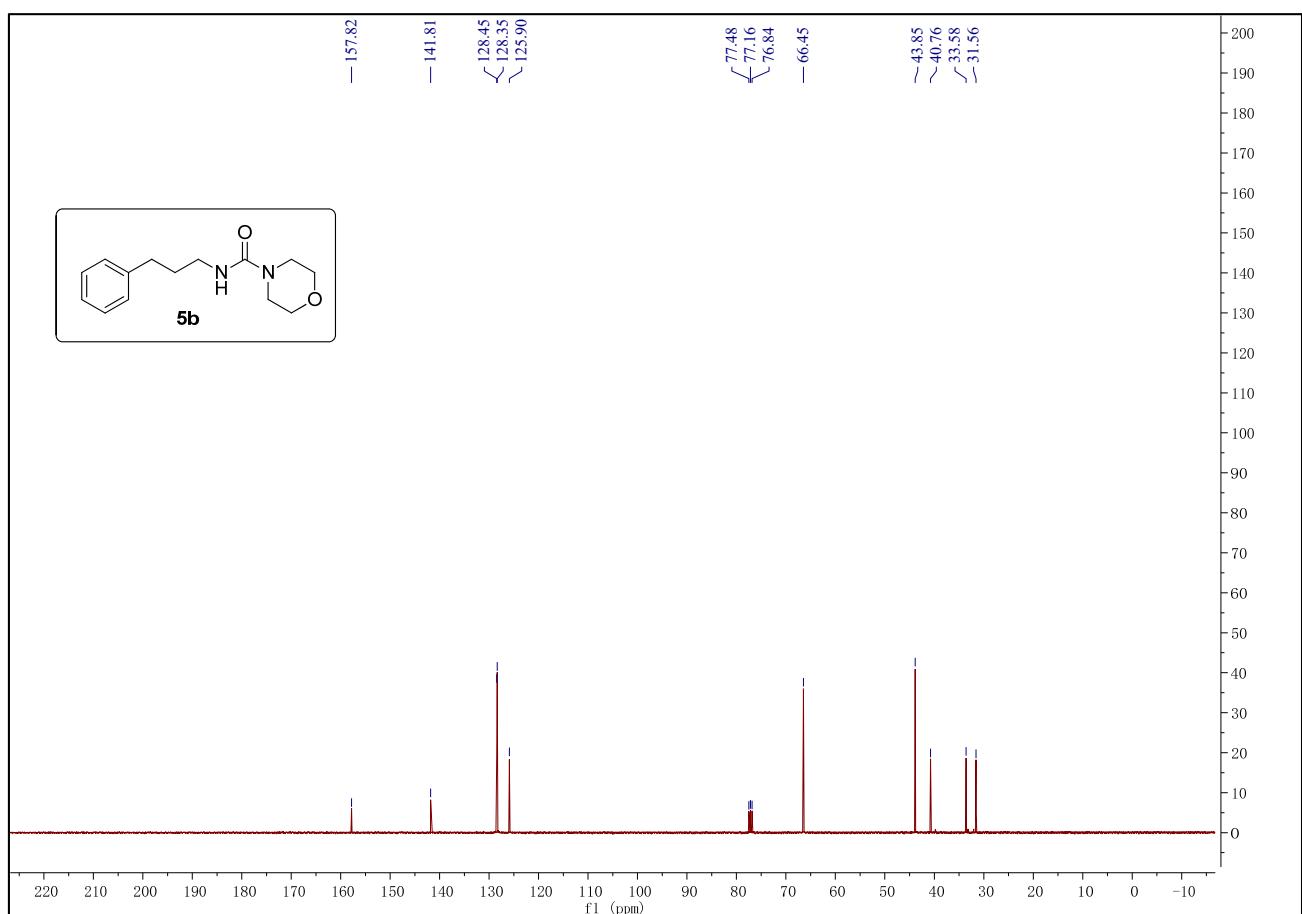
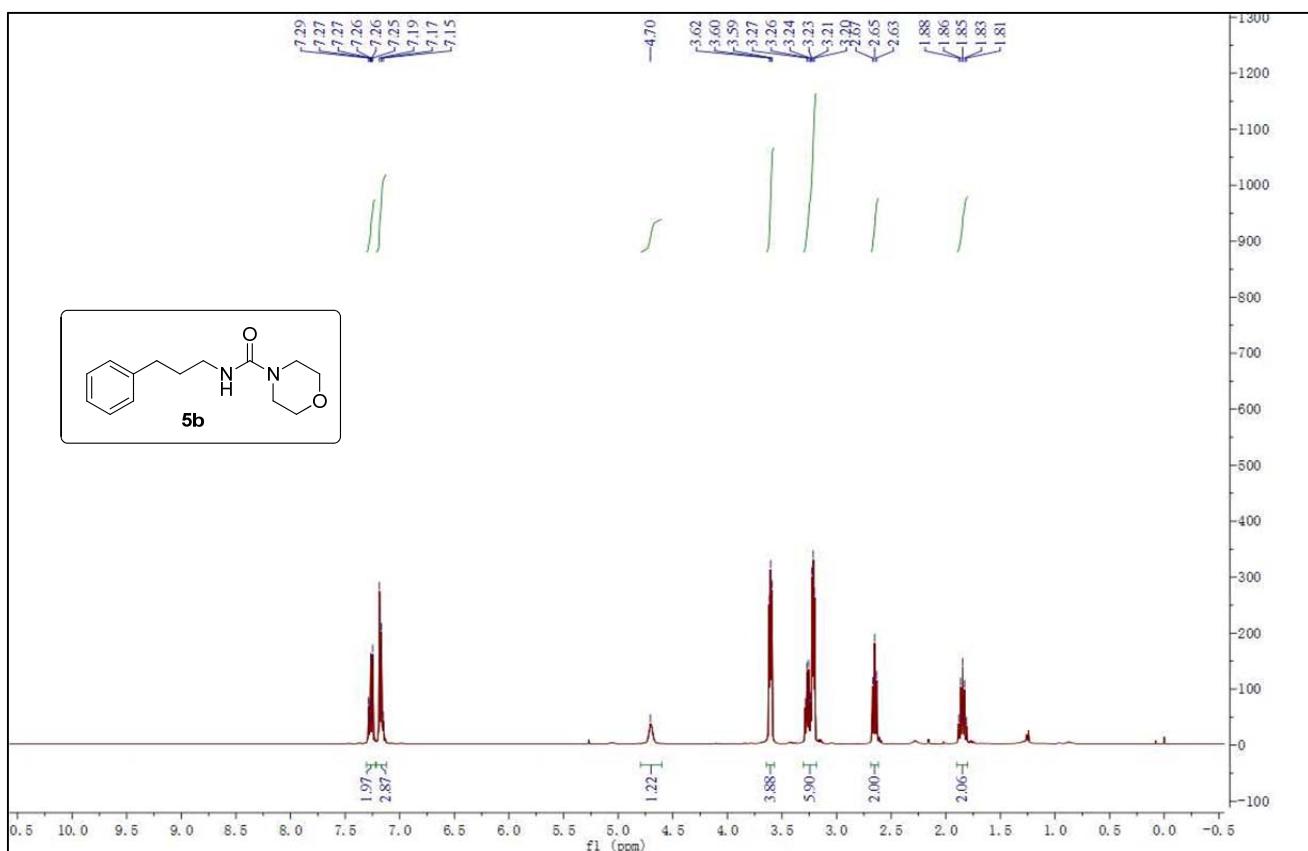


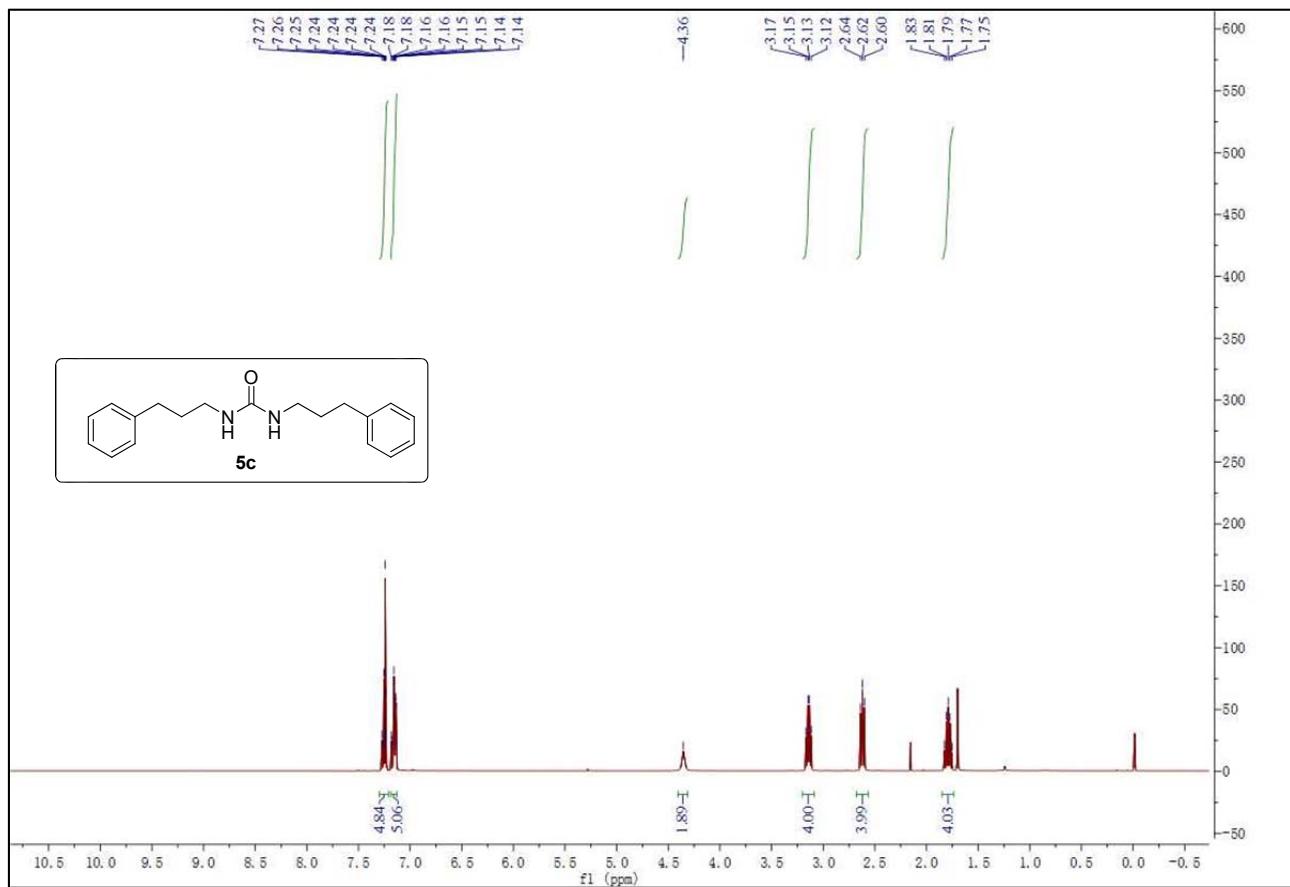
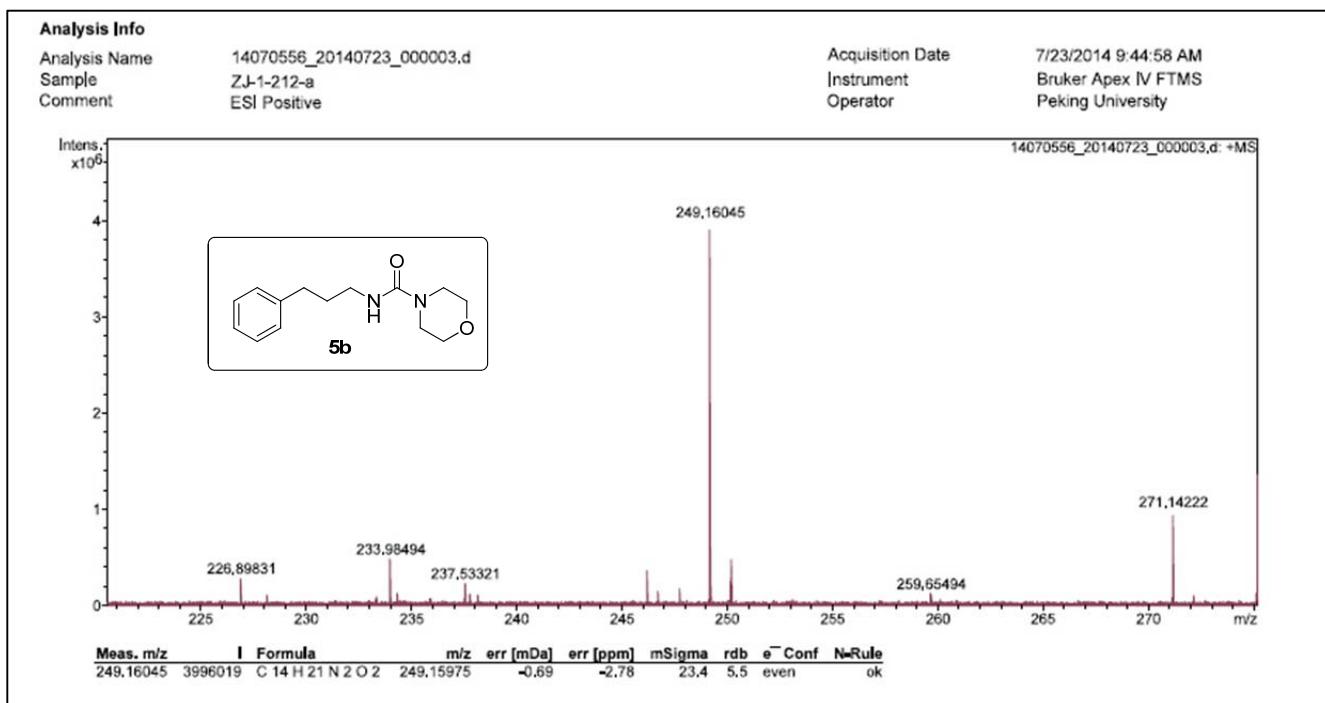


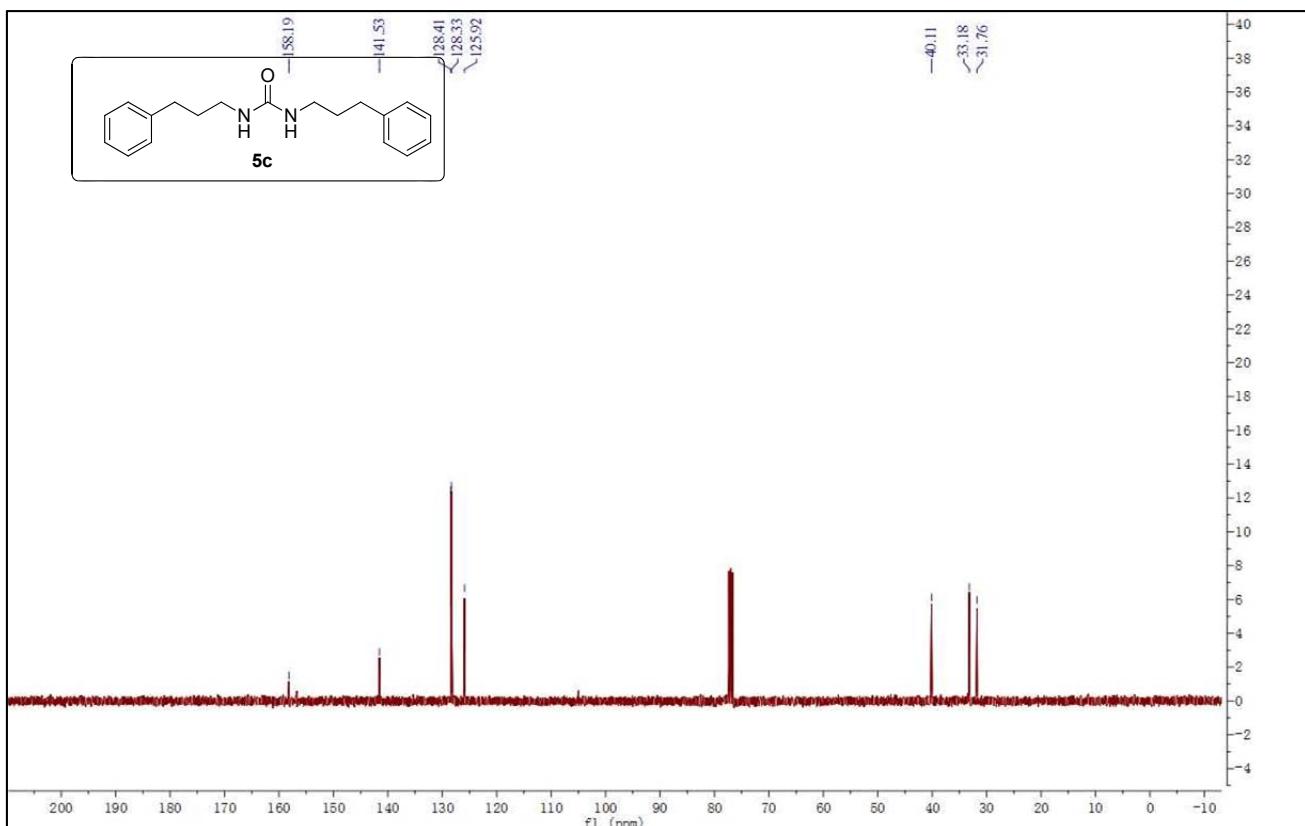








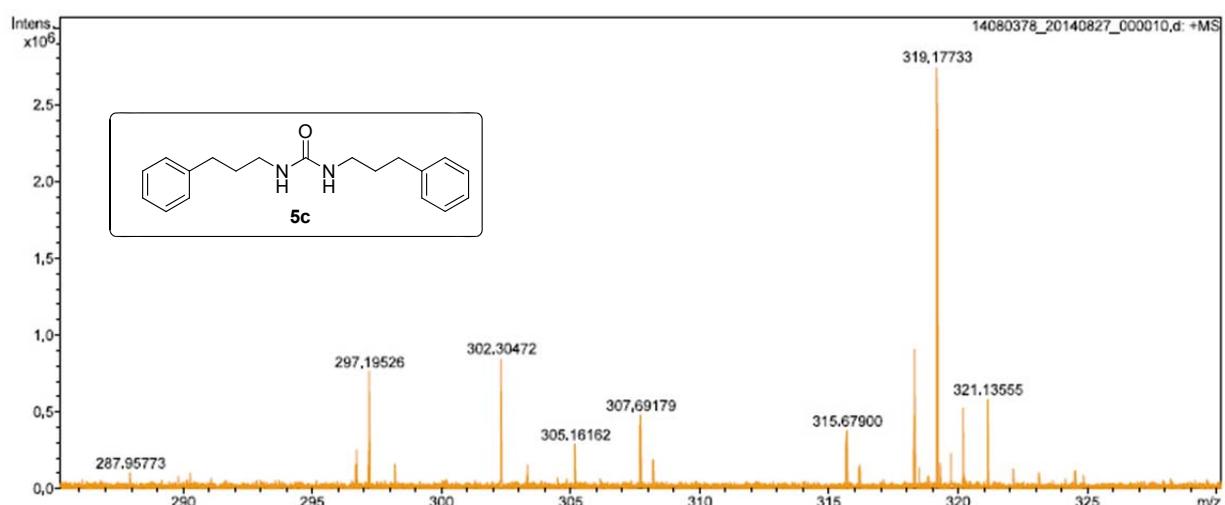


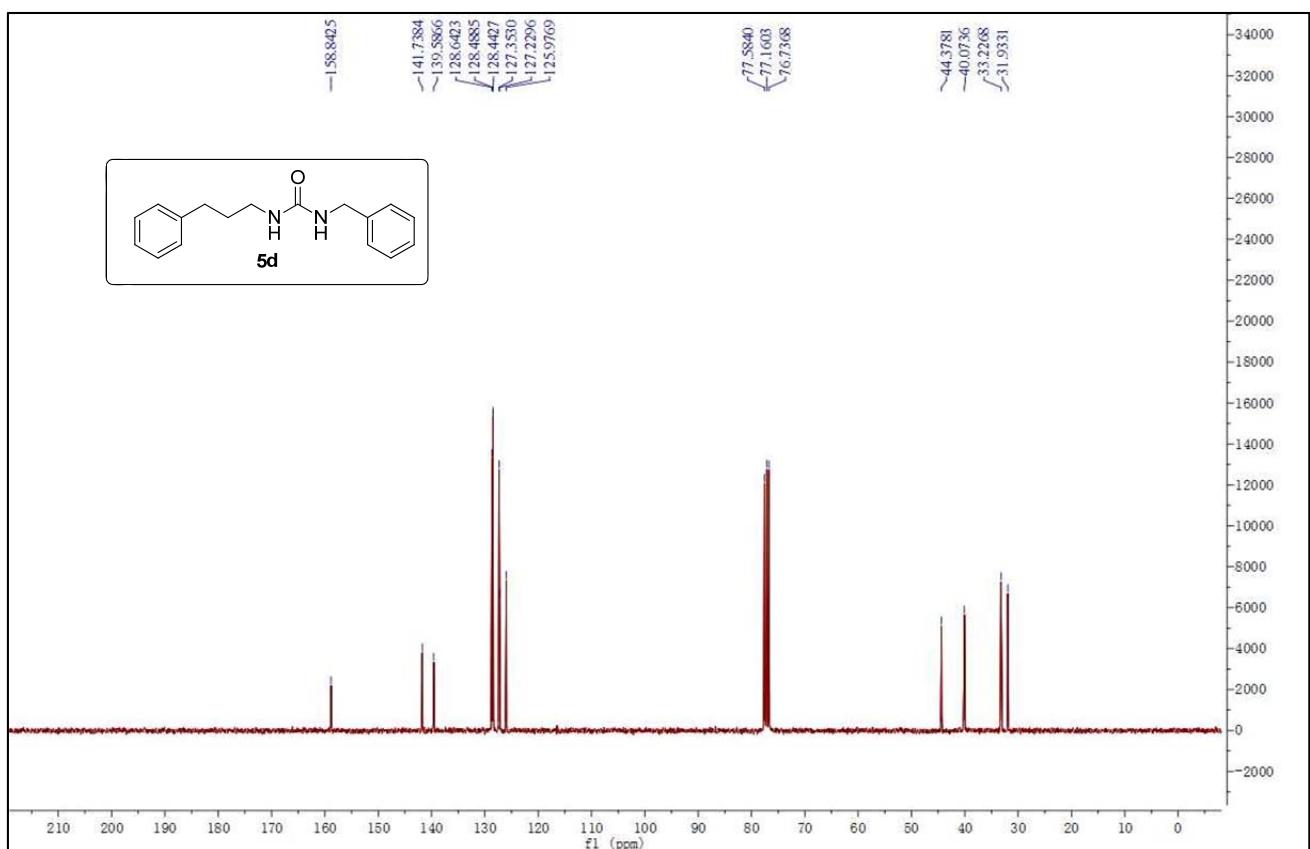
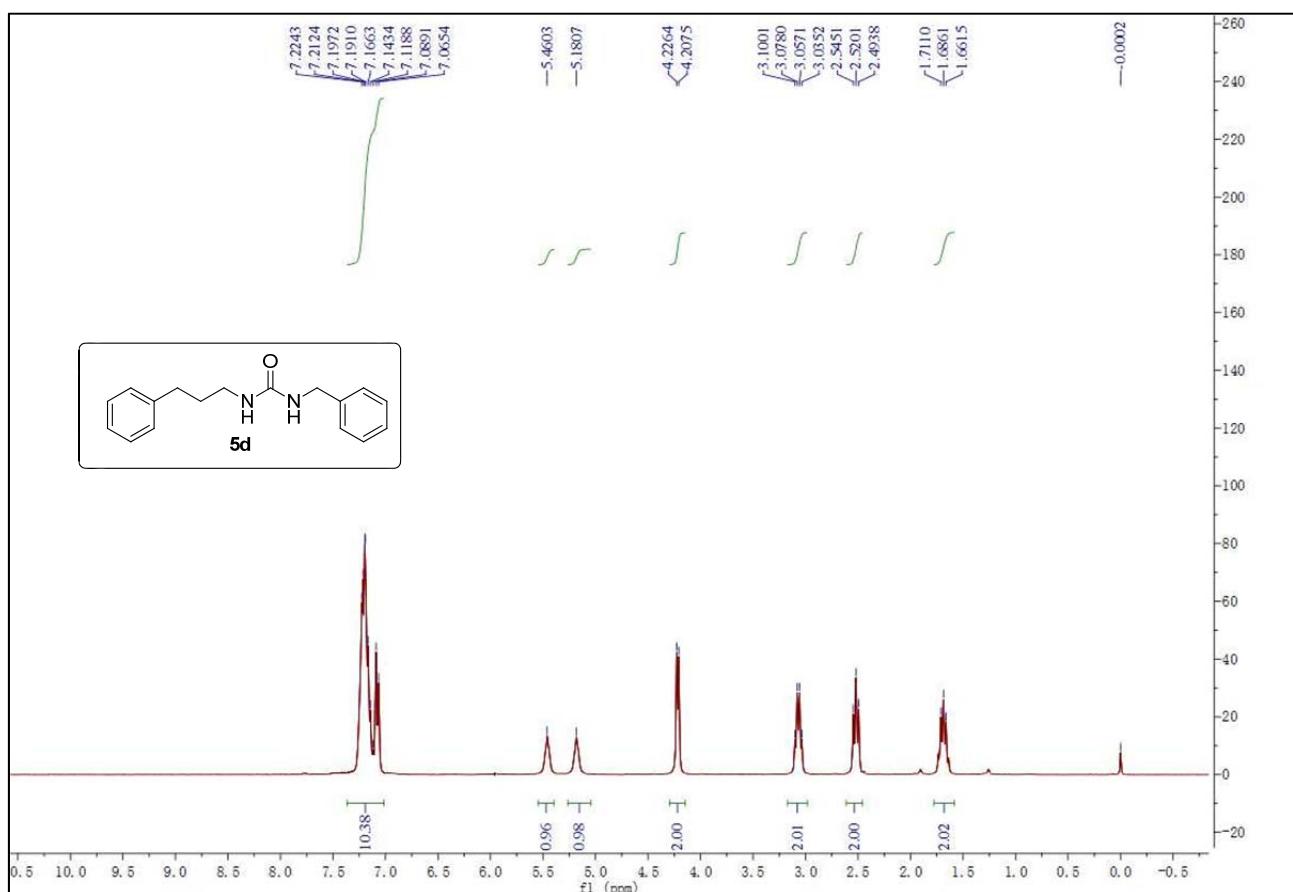


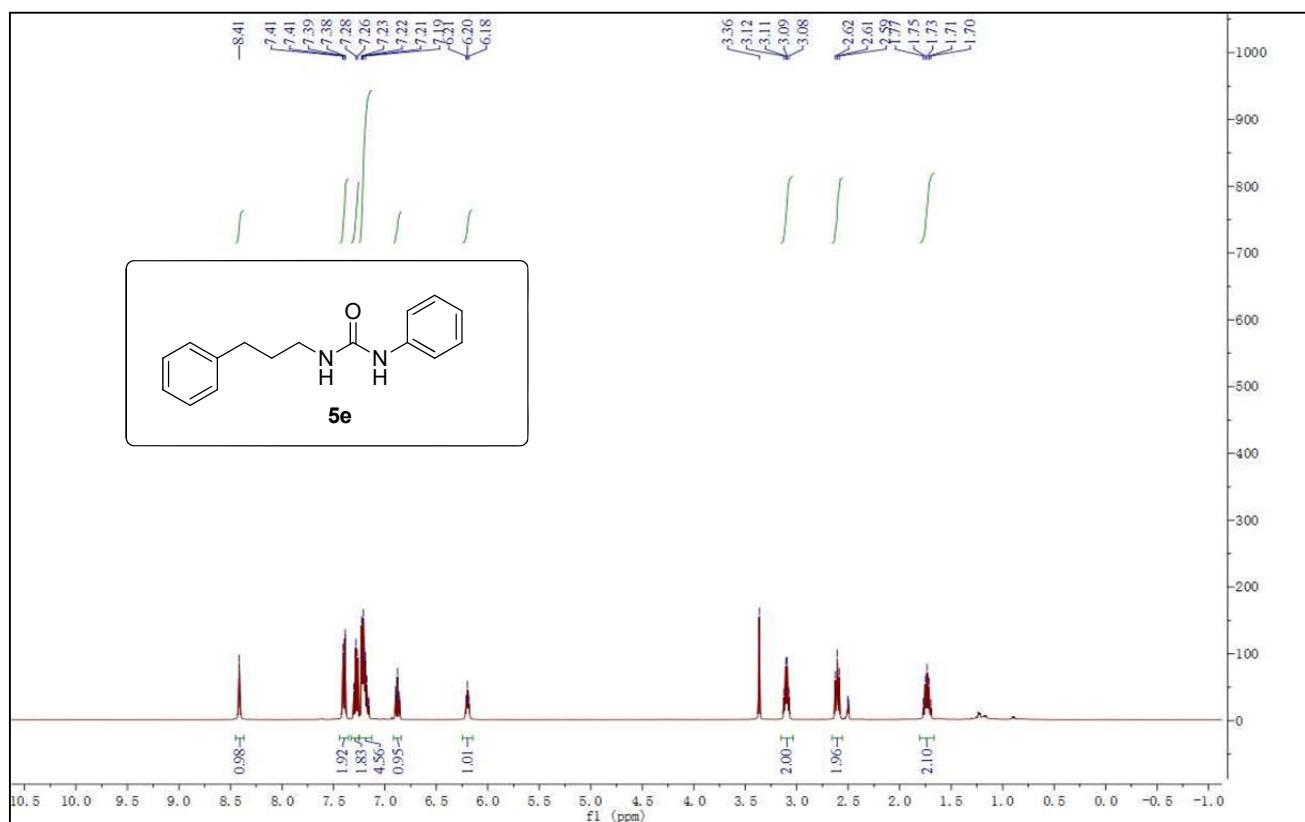
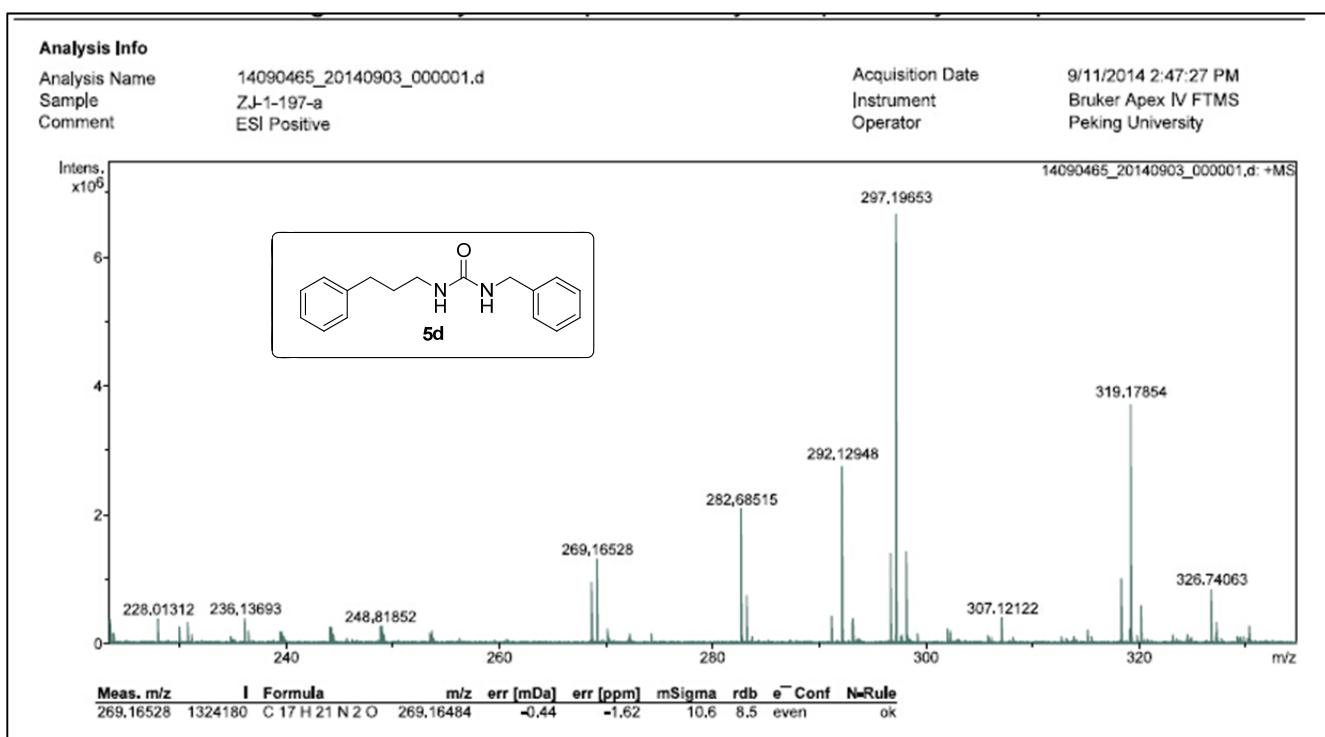
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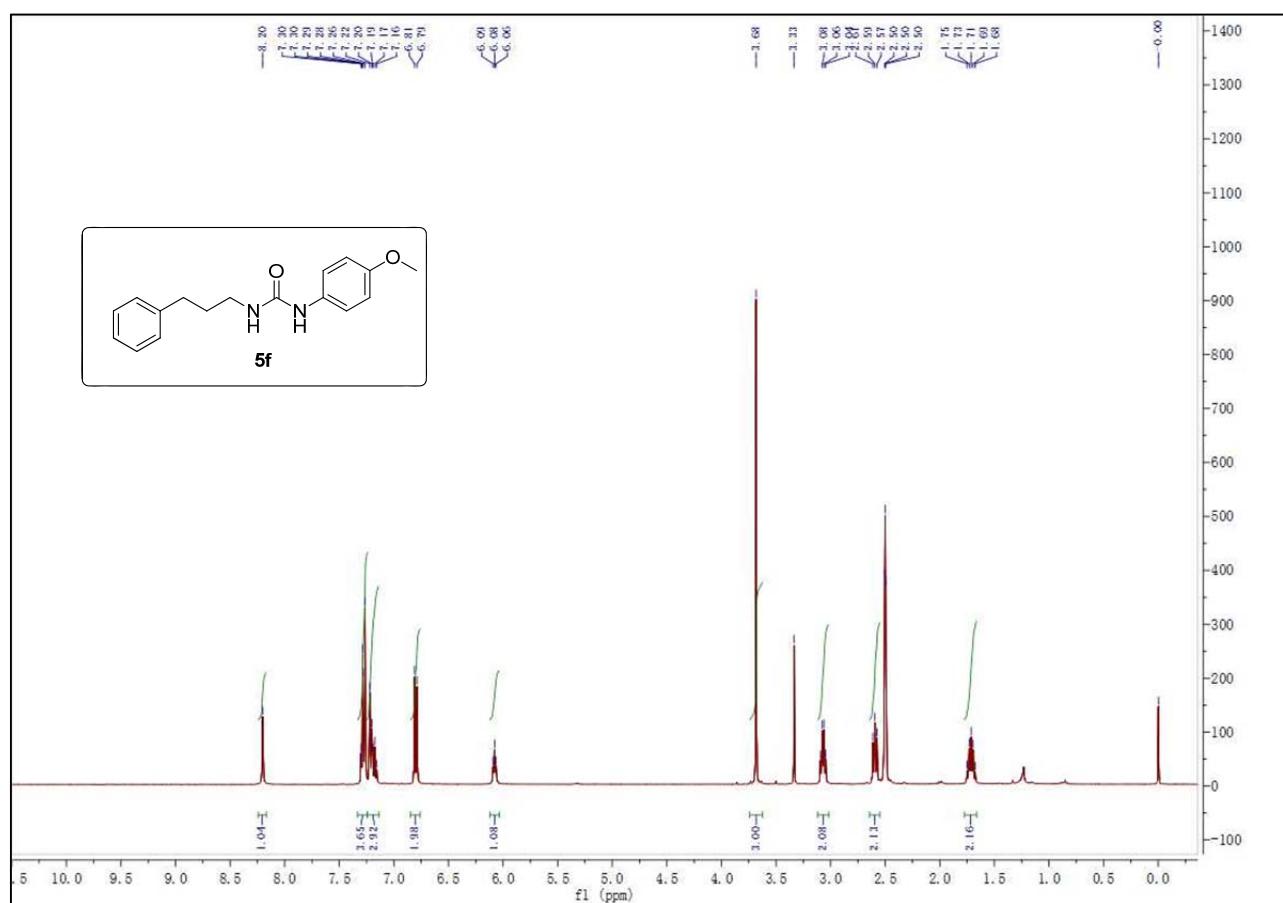
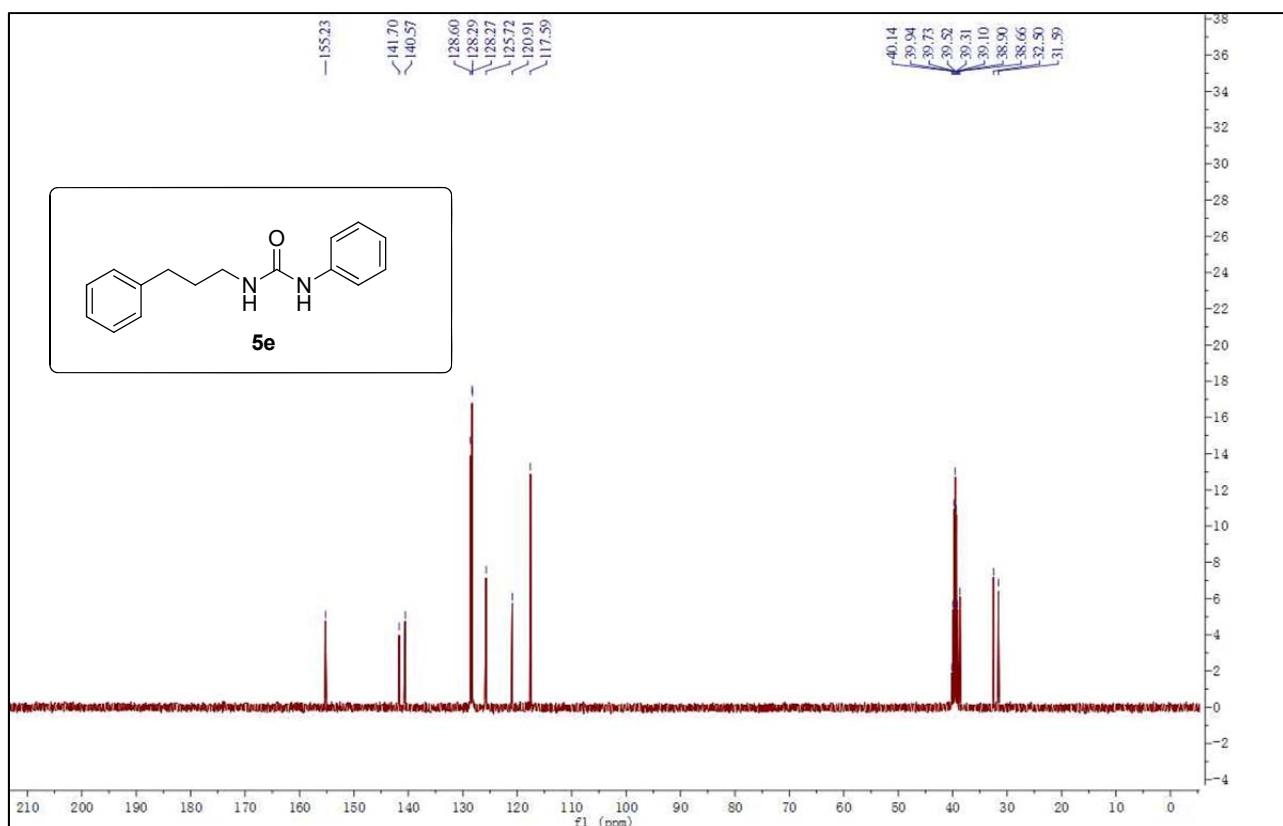
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Sample zj-2-1
Comment ESI Positive

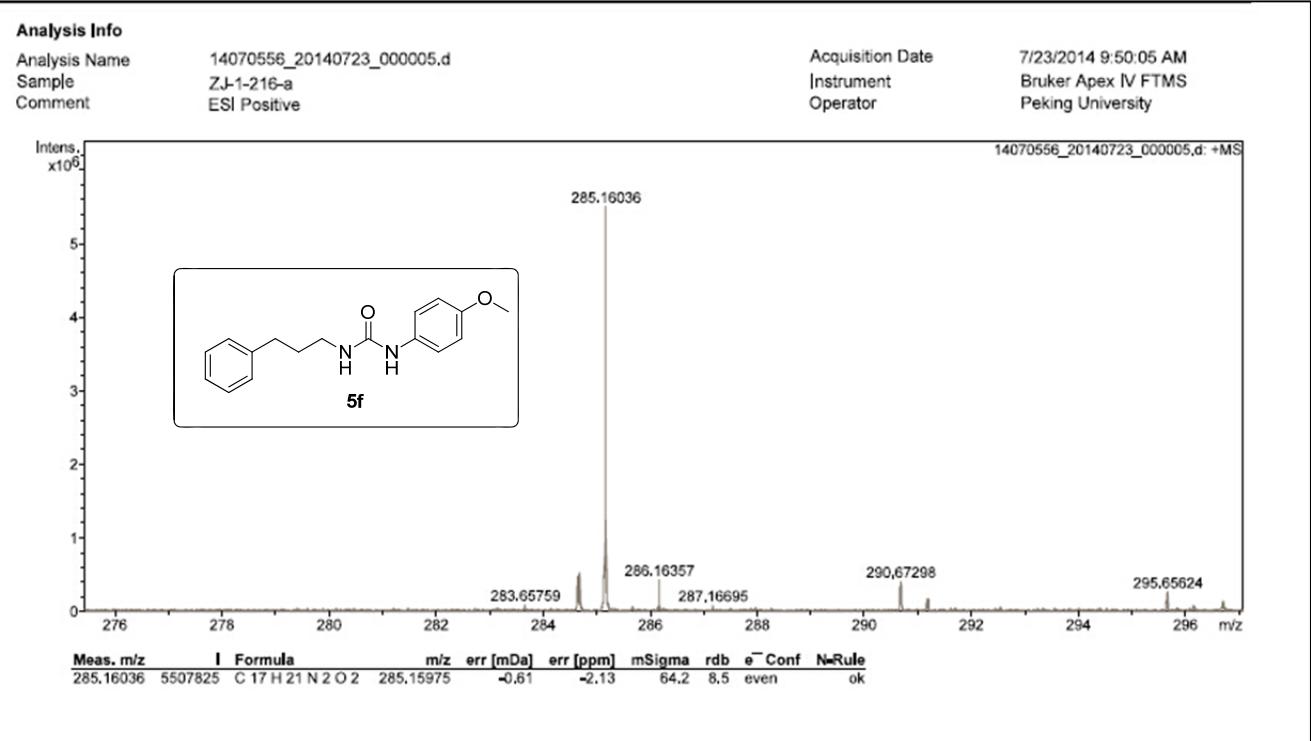
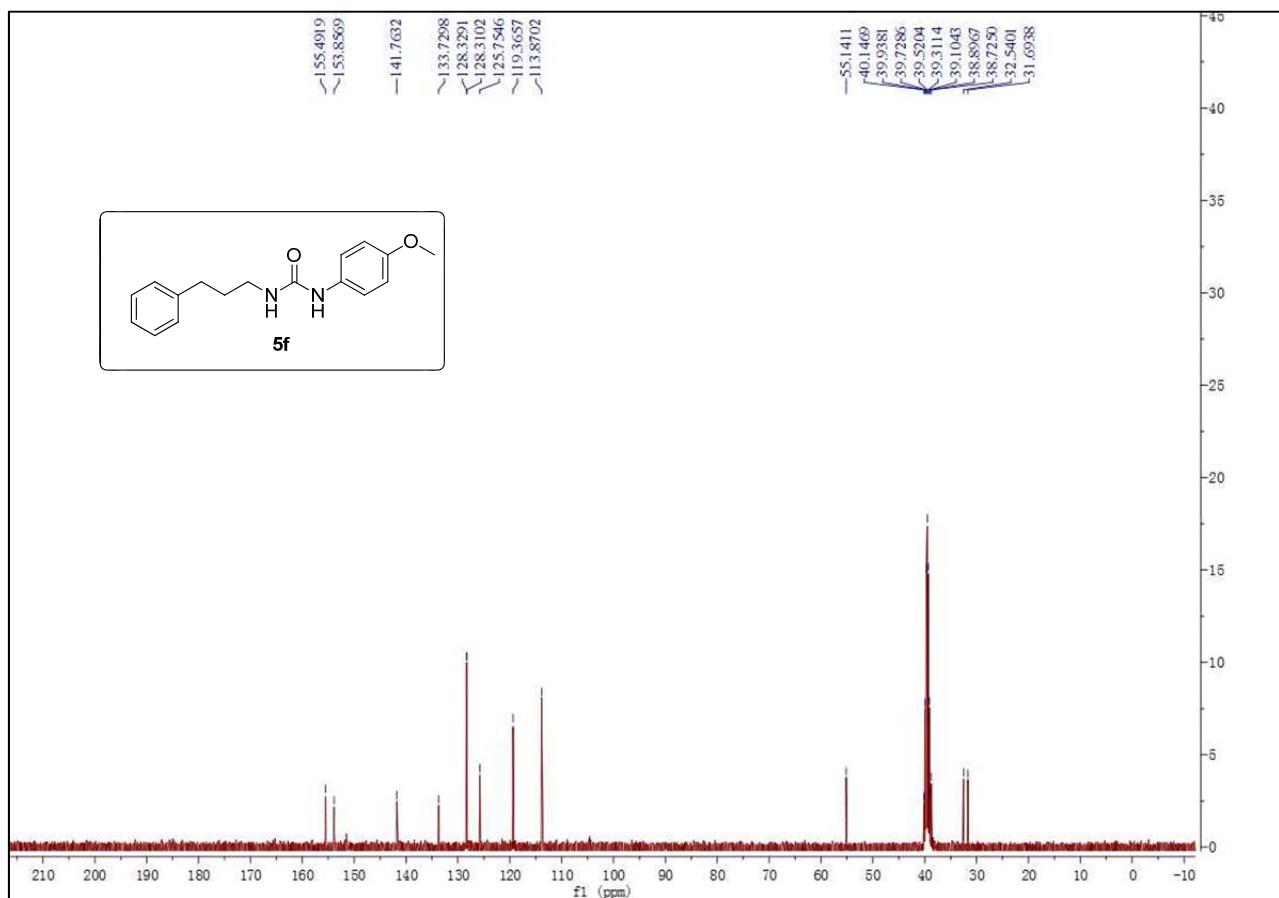
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Instrument Bruker Apex IV FTMS
Operator Peking University

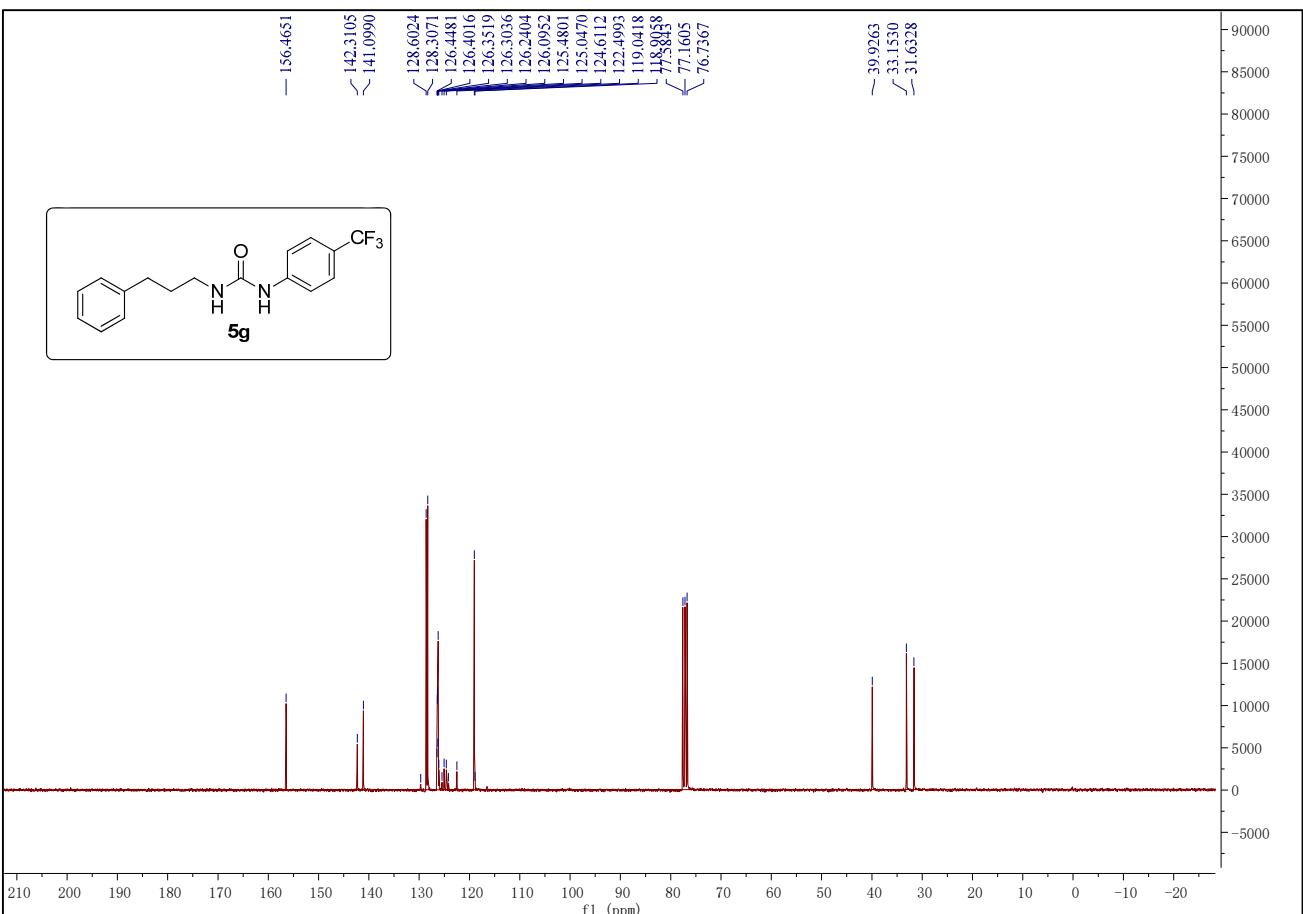
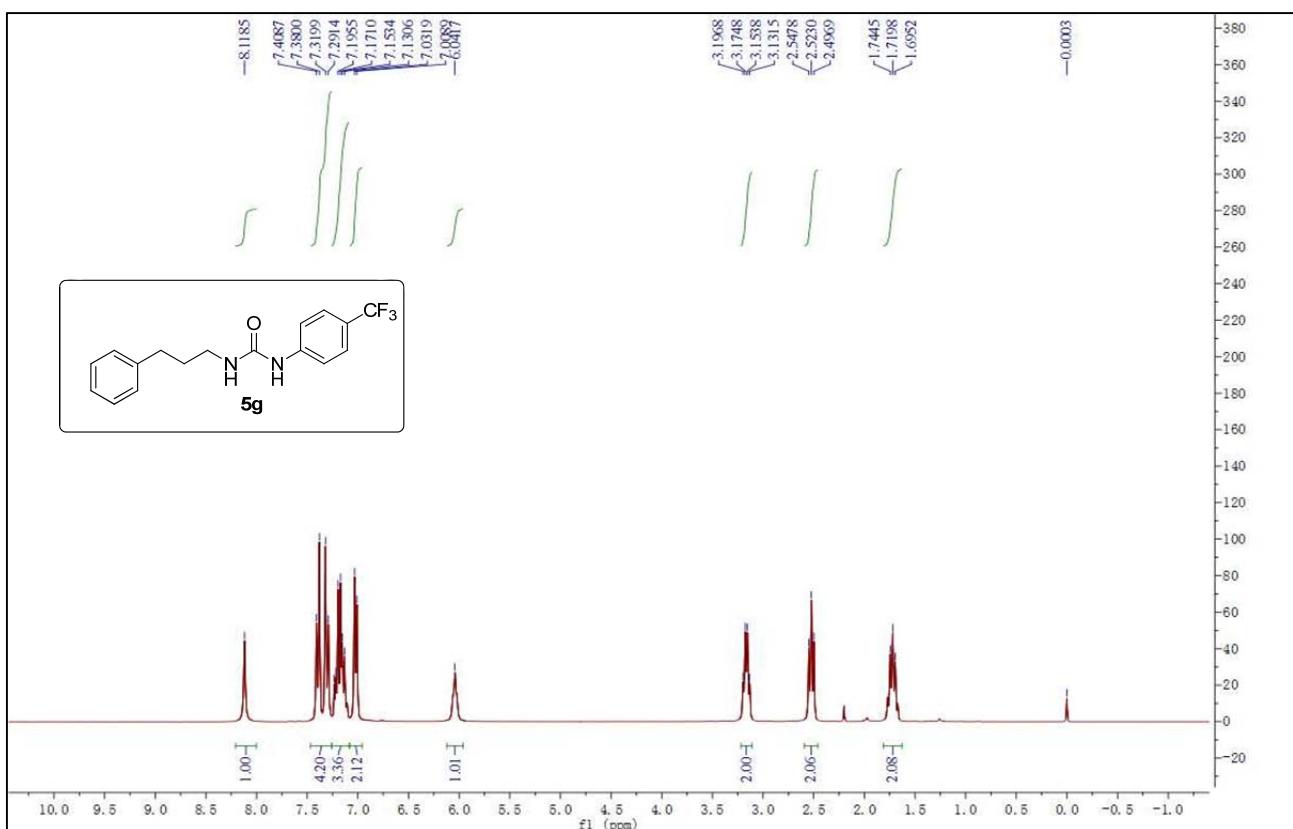


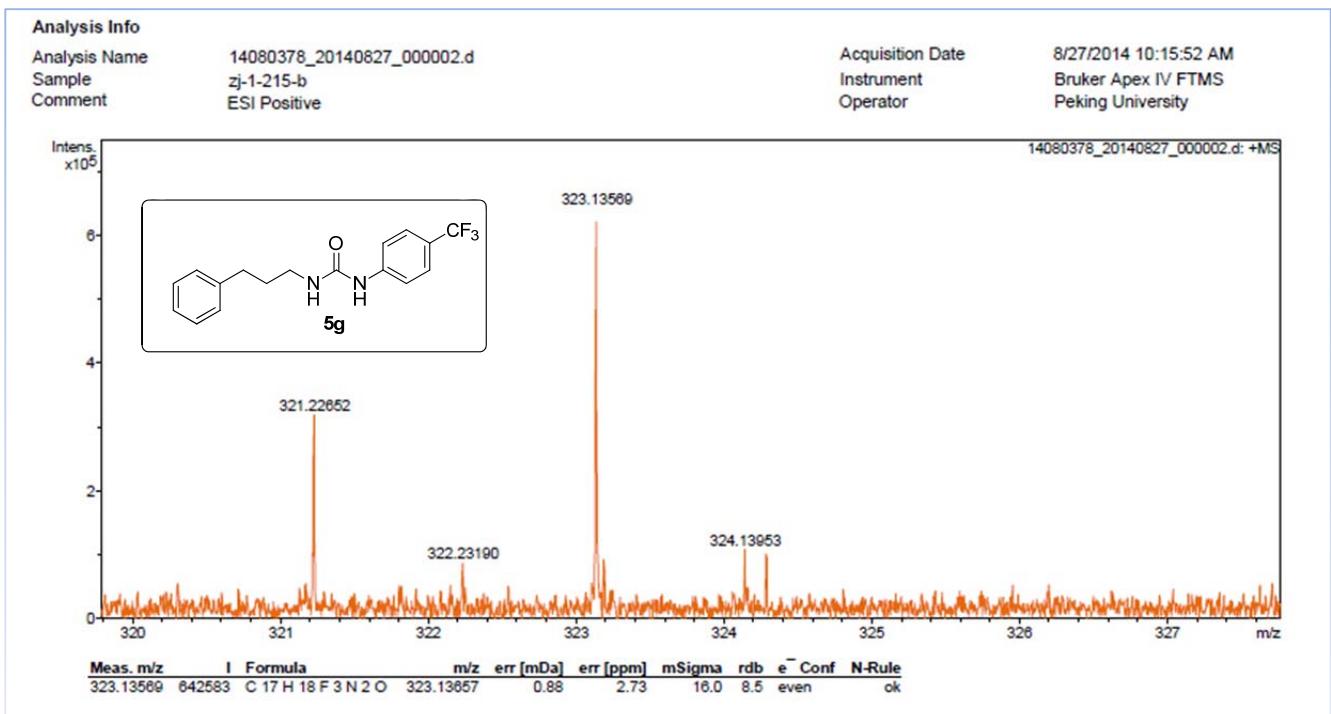
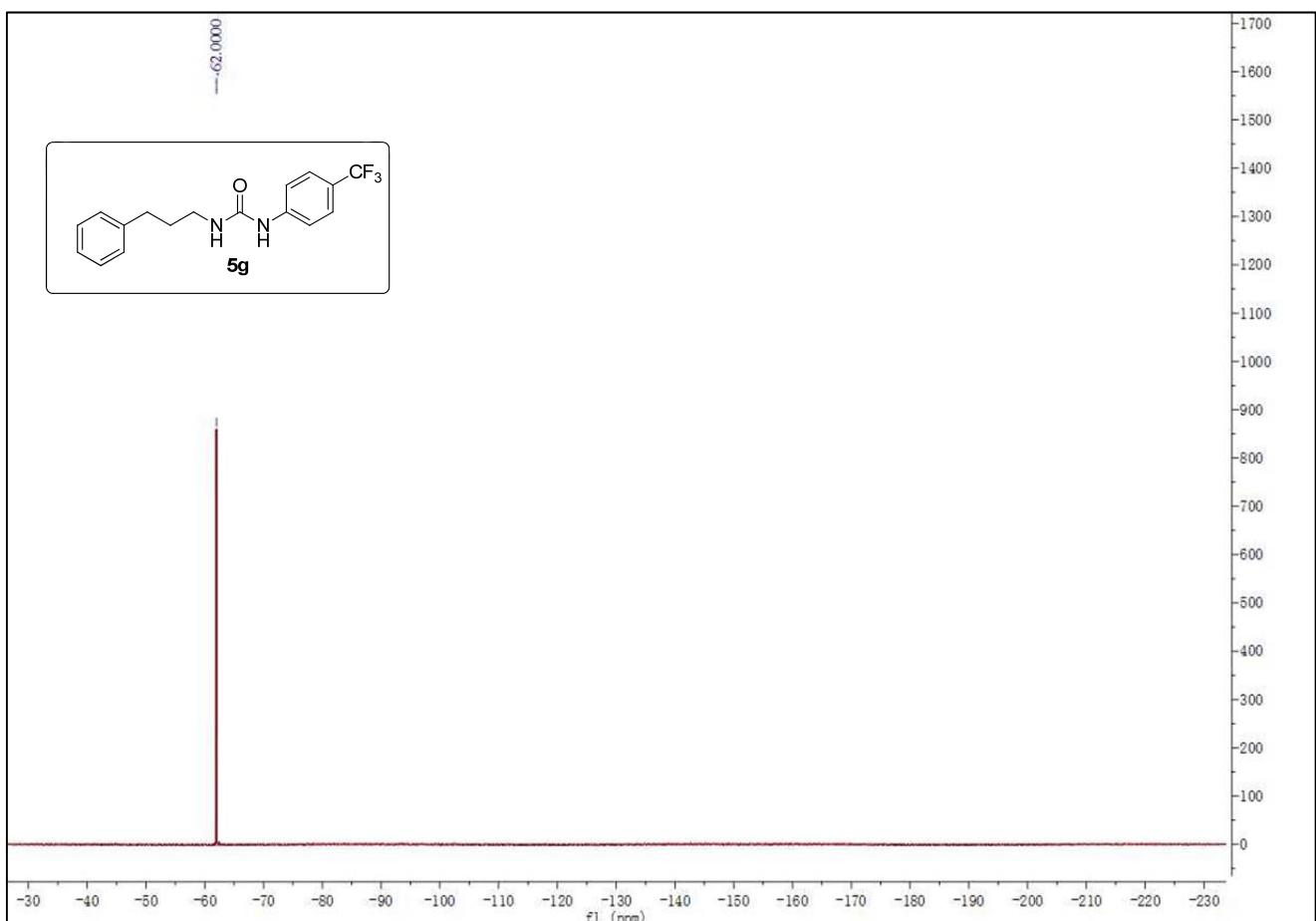


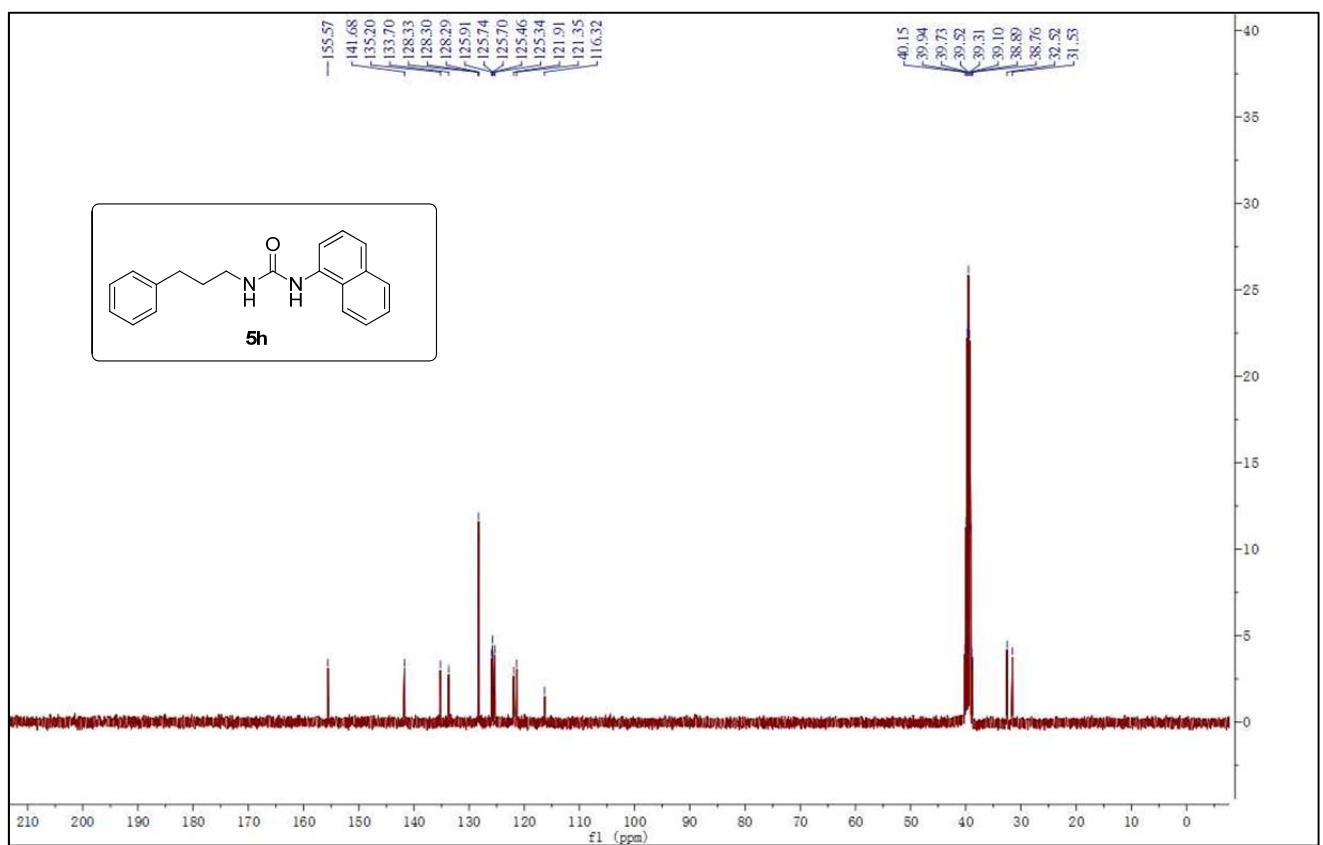
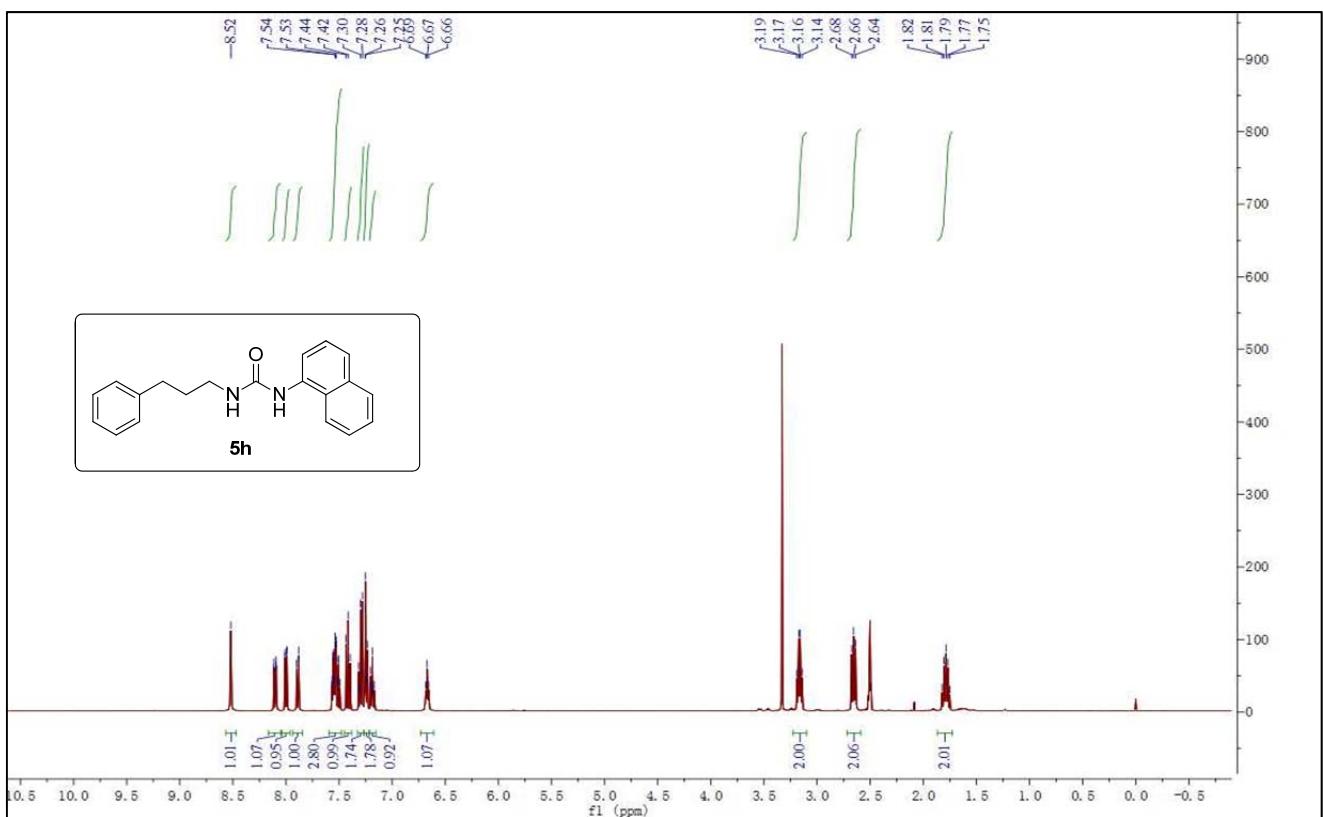












Lizongyang-C20H20N2O #64 RT: 0.29 AV: 1 NL: 3.80E8
T: FTMS + p ESI Full ms [60.00-500.00]

