Supporting Information

Organic Ligand Free Carbonylation Reactions with Unsupported Bulk Pd as Catalyst

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Experimental Procedures

1. General information and experimental section

1.1 General Information:

XRD measurements were conducted by using a STADIP automated transmission diffractometer (STOE) equipped with an incident beam curved germanium monochromator with CuKa1 radiation and current of 40 kV and 150 mA, respectively. The XRD patterns were scanned in the 2 Theta range of 10-90°. XPS were obtained using a VG ES-CALAB 210 instrument equipped with a dual Mg/Al anode X-ray source, a hemispherical capacitor analyzer, and a 5 keV Ar+ iron gun. The electron binding energy was referenced to the C1s peak at 284.8 eV. The background pressure in the chamber was less than 10-7 Pa. The peaks were fitted by Gaussian–Lorentzian curves after a Shirley background subtraction. For quantitative analysis, the peak area was divided by the element-specific Scofield factor and the transmission function of the analyzer. TEM was carried out by using a Tecnai G2 F30 S-Twin transmission electron microscope operating at 300 kV. Single-particle EDX analysis was performed by using a Tecnai G2 F30 S-Twin Field Emission TEM in STEM mode. For TEM investigations, the catalysts were dispersed in ethanol by ultrasonication and deposited on carbon-coated copper grids. The Pd dispersion determined with H_2 titration technique using a Quantachrome autosorb IQ_2 at different temperature (25, 40, 80 ^oC). Hydrogen titration program: 1. Change Gas to Helium; 2. Ramp Temp by Rate to 140 °C at 20 deg/min; 3. Action: Flow Until Setpoint; 4. Action: Flow for 30 minutes; 5. Change Gas to port: 3, Hydrogen; 6. Ramp Temp by Rate to 400 °C at 20 deg/min; 7. Action: Flow Until Setpoint; 8. Action: Flow for 60 min; 9. Change Gas to Helium; 10. Action: Flow for 45 minutes; 11. Force Cool Furnace to 25/40/80 ^oC, stabilize; 12. Change Gas to #1, nitrogen; 13. TCD Device: On (Ib current:100, zeroing time out:5); 14. Action: Flow for 15 min; 15. Perform Titration Acquisition (Manual Analysis: No); 16. TCD Device: Off; 17. end of macro; NMR spectra were measured by using a Bruker ARX 400 or ARX 100 spectrometerat 400 MHz (^1H) and 100 MHz (^{13}C). Chemical shifts were reported in parts permillion (ppm) down field from TMS with the solvent resonance as the internalstandard. Coupling constants (*J*) were reported in Hz

and refered to apparent peakmultiplications. Mass spectra were in general recorded on an Agilent 5977A MSD GC-MS.

1.2 Experimental Procedures

All solvents and chemicals were obtained commercially and used as received.

Typical procedure for carbonylation of olefins with amines and CO.

A mixture of olefins (2.0 mmol), amines (1 mmol), Pd-610 (1 mg), KI (5.0 mol%), H_3PO_4 (5.0 mol%) and dioxane (2 mL) were added a glass tube which was placed in an 100 mL autoclave. Then the autoclave was purged and charged with CO (40 bar). The reaction mixture was stirred at 130 ^oC for 24 h. After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. Subsequently, the reaction mixture was diluted with 5 mL of ethyl acetate for analysis by GC-MS. The regioselectivity were measured by ^1H NMR. The crude reaction mixture was concentrated by rot-vap and purified by column chromatography on a silica gel column to give the desired products 3a and 4a.

Typical procedure for carbonylation of aryl iodides with amines and CO.

A mixture of aryl iodides (1.0 mmol), amines (1.5 mmol), Pd-610 (1 mg), KI (5.0 mol%), K_2HPO_4 (2.0 mmol) and dioxane (2 mL) were added a glass tube which was placed in an 80 mL autoclave. Then the autoclave was purged and charged with CO (5 bar). The reaction mixture was stirred at 130 ^oC for 12 h. After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. Subsequently, the reaction mixture was diluted with 5 mL of methanol for analysis by GC-MS. The crude reaction mixture was concentrated by rot-vap and purified by column chromatography on a silica gel column to give the desired products.

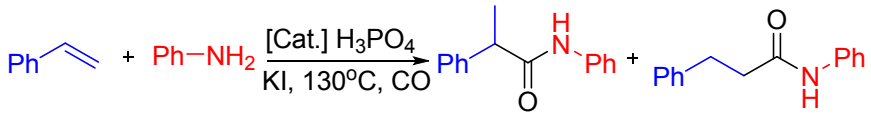
Typical procedure for oxidative carbonylation of amines.

A mixture of amines (2 mmol), Pd-610 (1 mg), KI (5.0 mol%) and dioxane (2 mL) were added a glass tube which was placed in an 80 mL autoclave. Then the autoclave was purged and charged with CO and O_2 ($CO : O_2 = 35 : 5$ bar). The reaction mixture was stirred at 130 ^oC for 24 h. After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. Subsequently, the reaction mixture was diluted with 5 mL of

methanol. The reaction mixture was concentrated by rot-vap and then heated for 0.5 h to remove aniline to give the desired products.

2. Optimization of the reaction conditions

Table S1. Screening of the catalyst for the carbonylation of styrene with aniline [a].

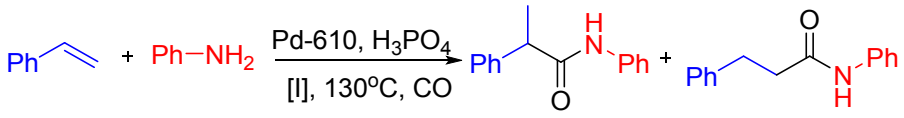


1a **2a** **3a branched** **4a linear**

Entry	Cat.	Con.[b] (%)	Sel.[b] (%)	b/l
1	Pd-148	91	98	46:54
2	**Pd-610**	**95**	**98**	**35:65**
3	Pd-1540	16	>99	60:40
4	$PdCl_2$	60	>99	44:56
5	$PdCl_2$/(2-OMePh)$_3$P	39	81	36:64
6[c]	PdI_2	58	94	65:35
7[d]	PdI_2	40	>99	59:61

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-10, Pd-148, Pd-610, Pd-1540] (1.0 mg), [$PdCl_2$] (1.77 mg), H_3PO_4 (5.0 mol%), KI (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h; [b] Determined by GC-MS; [c] 1a (2.0 mmol); [d] KI free.

Table S2. Screening of the [I] for the carbonylation of styrene with aniline [a].

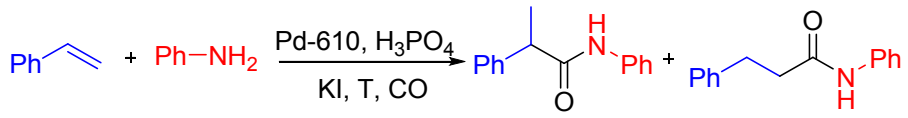


1a **2a** **3a branched** **4a linear**

Entry	[I]	Con.[b] (%)	Sel.[b] (%)	b/l
1	KI	95	98	35:65
2[c]	**KI**	**93**	**99**	**35:65**
3[d]	KI	22	>99	41:59
4[c]	NaI	69	91	45:55
5[c]	I_2	99	92	40:60
6	--	trace	--	--

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), Pd-610 (1.0 mg), H_3PO_4 (5.0 mol%), [I] (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h; [b] Determined by GC-MS; [c] 1a (2.0 mmol); [d] 1a (1.2 mmol).

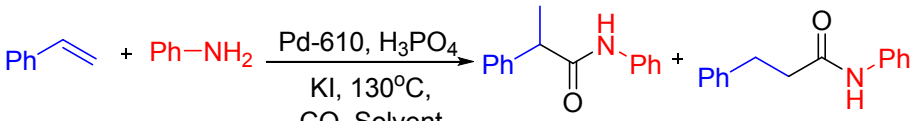
Table S3. Screening of the temperature for the carbonylation of styrene with aniline [a].



Entry	T (℃)	Con.[b] (%)	Sel.[b] (%)	b/l
1	**130**	**95**	**98**	**35:65**
2	150	97	91	36:64
3	110	12	>99	68:32

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), Pd-610 (1.0 mg), H_3PO_4 (5.0 mol%), [I] (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 12 h; [b] Determined by GC-MS.

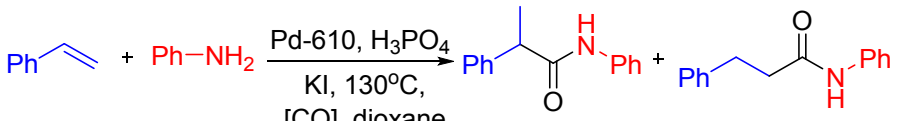
Table S4. Screening of the solvent for the carbonylation of styrene with aniline [a].



Entry	solvent	Con.[b] (%)	Sel.[b] (%)	b/l
1	**dioxane**	**95**	**98**	**35:65**
2	NMP	14	72	46:54
3	DMF	n.r.	--	--
4	THF	85	97	49:51

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), H_3PO_4 (5.0 mol%), KI (5.0 mol%), CO (40 bar), solvent (2.0 mL), 130 °C, 12 h; [b] Determined by GC-MS.

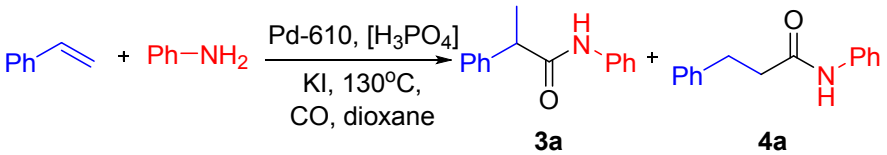
Table S5. Screening of the pressure CO for the carbonylation of styrene with aniline [a].



Entry	[CO](bar)	Con.[b] (%)	Sel.[b] (%)	b/l
1	**40**	**95**	**98**	**35:65**
2	30	62	93	47:53
3	20	60	60	32:68

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), H_3PO_4 (5.0 mol%), KI (5.0 mol%), dioxane (2.0 mL), 130 °C, 12 h; [b] Determined by GC-MS.

Table S6. Screening of the amount of acids for the carbonylation of styrene with aniline [a].



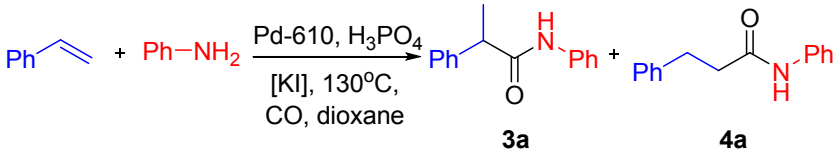
1a **2a** **3a branched** **4a linear**

Entry	[H_3PO_4]	Con.[b] (%)	Sel.[b] (%)	b/l
1	0%	trace	--	--
2	1%	trace	--	--
3	2%	trace	--	--
4	**5%**	**95**	**98**	**35:65**
5	10%	88	96	51:49

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), [KI] (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h;

[b] Determined by GC-MS.

Table S7. Screening of the amount of acids for the carbonylation of styrene with aniline [a].



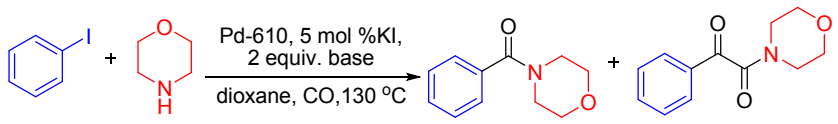
1a **2a** **3a branched** **4a linear**

Entry	[KI]	Con.[b] (%)	Sel.[b] (%)	b/l
1	1%	48	99	62:38
2	2%	82	97	55:45
3	**5%**	**95**	**98**	**35:65**
4	10%	trace	--	--

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), [H_3PO_4] (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h;

[b] Determined by GC-MS.

Table S8. Screening of the base for the carbonylation of iodobenzene with morpholine [a].

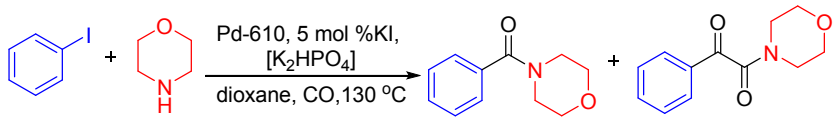


5a **6a** **7a** **8a**

Entry	base	Con.[b] (%)	Sel.[b] (%)	
			7a	**8a**
1	KOH	trace	--	--
2	K_3PO_4	99	57	43
3	KH_2PO_4	69	99	--
4	K_2HPO_4	99	99	--
5	K_2CO_3	99	42	58
6	Na_3PO_4	55	66	34
7	NaH_2PO_4	28	99	--
8	Na_2HPO_4	77	99	--
9	$NaHCO_3$	trace	--	--
10	CH_3COOK	56	70	--
11	KO*t*Bu	48	6	--
12	Et_3N	trace	--	--

[a] Reaction conditions: 5a (1.5 mmol), 6a (1.0 mmol), [Pd-610] (1.0 mg), CO (5 bar), dioxane (2.0 mL), 130 ℃, 6 h; [b] Determined by GC-MS.

Table S9. Screening of the amount of K_2HPO_4 for the carbonylation of iodobenzene with morpholine [a].

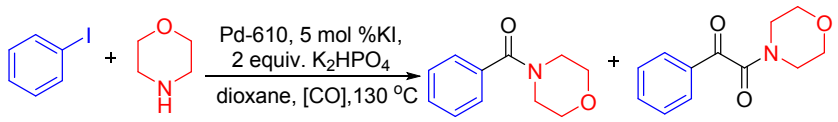


5a **6a** **7a** **8a**

Entry	K_2HPO_4(equiv.)	Con.[b] (%)	Sel.[b] (%)	
			7a	**8a**
1	2	>99	>99	--
2	1	23	>99	--
3	0.5	21	>99	--

[a] Reaction conditions: 5a (1.5 mmol), 6a (1.0 mmol), [Pd-610] (1.0 mg), CO (5 bar), dioxane (2.0 mL), 130 ℃, 6 h; [b] Determined by GC-MS.

Table S10. Screening of the pressure CO for the carbonylation of iodobenzene with morpholine [a].



5a **6a** **7a** **8a**

Entry	[CO](bar)	Con.[b] (%)	Sel.[b] (%)	
			7a	**8a**
1	5	>99	>99	--
2	3	80	>99	--
3	1	18	>99	--

[a] Reaction conditions: 5a (1.5 mmol), 6a (1.0 mmol), [Pd-610] (1.0 mg), CO (5 bar), dioxane (2.0 mL), 130 ℃, 6 h; [b] Determined by GC-MS.

Table S11. The characterization results of the catalysts.

Catalyst	Size [a]	Pd 3d (eV) [b]	XRD diffraction peaks (°) [c]	Dispersion(%)
Pd-148	148 nm	--	--	6.628
Pd-610	610 nm	335.4, 340.7	40.1, 46.7, 68.1, 82.1, 86.7	3.897
Pd-1580	1580 nm	--	--	4.372
Used Pd-610	610 nm	n. d. [d]	18.1,40.1, 46.7, 68.1, 82.1, 86.7	--

[a] Determined by SEM; [b] Determined by XPS; [c] Determined by XRD. [d] Not detectable.

3. Characterization results of the catalysts

Fig. S1 TEM of catalyst. Pd-148 (a), Pd-610 (b), Pd-1540 (c), Pd-610 used three times (d).

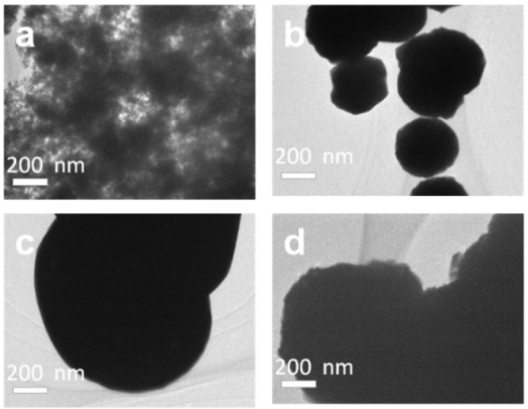


Fig. S2 XPS spectra of catalysts. (a) fresh Pd-610 and (b) Pd-610 used three times.

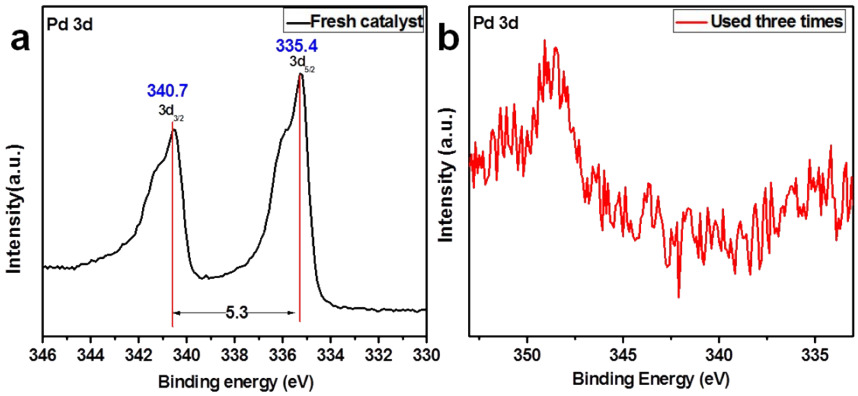
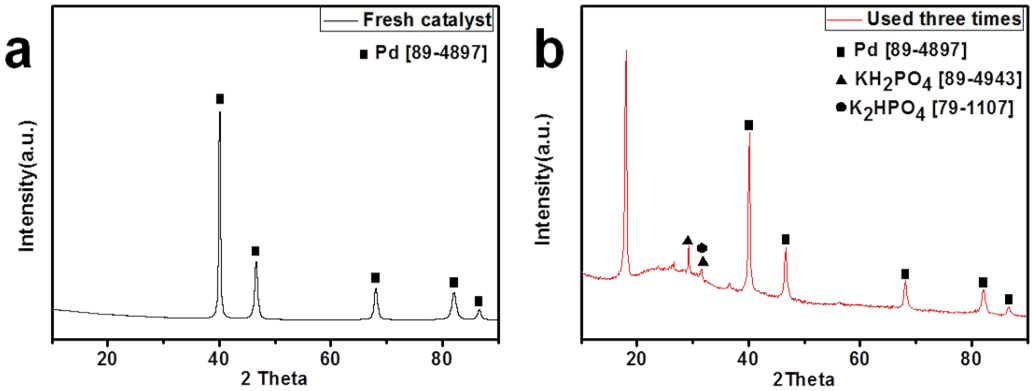
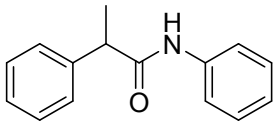


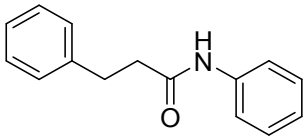
Fig. S3 XRD patterns of catalysts. (a) fresh Pd-610 and (b) Pd-610 used three times.



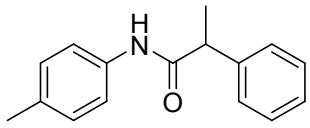
4. Characterization data for products



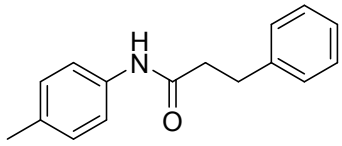
3a **N-Phenyl-2-phenylpropanamide**[1]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a light grey solid; mp: 127-129℃ (lit.[1] 134-135℃); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.43 (m, 6H), 7.25-7.33 (m, 3H), 7.04-7.08 (m, 2H), 3.72 (q, 7.2 MHz, 1H), 1.60 (d, 7.2 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 18.5, 140.9, 138.0, 129.1, 128.9, 127.7, 127.6, 124.2, 119.6, 48.1.



4a **N,3-diphenylpropanamide**[2]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 92-93℃(lit.[1] 116-117℃); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 (d,7.60 MHz, 2H), 7.21-7.31 (m, 8H), 7.08(t, 7.4 MHz, 1H), 3.04 (t, 7.6 MHz, 2H), 2.64 (t, 7.6 MHz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 140.6, 137.7, 128.9, 128.6, 128.3, 126.3, 124.3, 120.0, 39.3, 31.5.

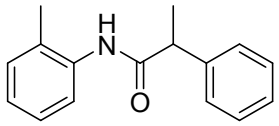


3b **2-phenyl-N-(p-tolyl)propanamide**[3]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a light yellow solid; mp: 115-117℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.38 (m, 4H), 7.29-7.31 (m, 3H), 7.05-7.08 (m, 3H), 3.70 (q, 7.2 MHz, 1H), 2.27(s, 3H), 1.58 (d, 6.8 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 141.1, 135.3, 133.8, 129.3, 129.1, 127.7, 127.5, 119.7, 48.0, 20.8, 18.6.

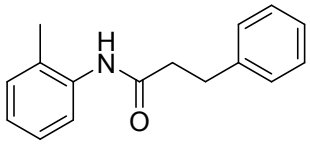


4b **3-phenyl-N-p-tolylpropanamide**[4]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 127-130℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.26-7.31 (m, 5H), 7.19-7.21 (m, 3H), 7.06-7.08 (m, 2H), 3.02 (t, 7.6 MHz, 2H), 2.61

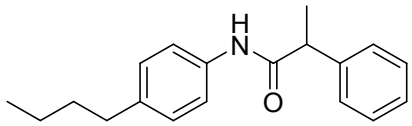
(t, 7.6 MHz, 2H), 2.29(s, 3H),; ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 140.7, 135.1, 133.9, 129.4, 128.6, 128.3, 126.3, 120.1, 39.3, 31.6, 20.8.



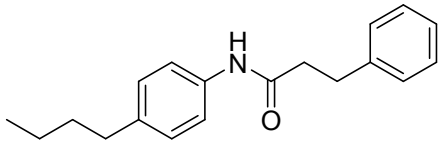
3c **2-phenyl-N-o-tolylpropanamide**[5]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20 : 1 to 10 : 1) to give the desired product which was a white solid; mp: 79-80℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88(d, 8.0 MHz, 1H), 7.31-7.43 (m, 5H), 6.98-7.19 (m, 3H), 6.86(brs, 1H), 3.79 (q, 7.2 MHz, 1H), 1.87(s, 3H), 1.65 (d, 7.2 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 140.9, 135.7, 130.3, 129.2, 128.0, 127.8, 127.7, 126.7, 124.7, 122.0, 48.1, 18.0, 17.1.



4c **3-phenyl-N-o-tolylpropanamide**[6]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1 to 6 : 1) to give the desired product which was a white solid; mp: 115-116℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.05(s, 3H), 2.69 (t, 7.4 MHz, 2H), 3.06 (t, 7.4 MHz, 2H), 6.90(brs, 1H), 7.03-7.06 (m, 1H), 7.12-7.32 (m, 8H), 7.70(d, 8.0MHz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 140.5, 135.4, 130.4, 128.6, 128.4, 126.6, 126.4, 125.2, 123.3, 39.2, 31.7, 17.5.

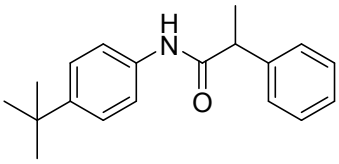


3d **N-(4-butylphenyl)-2-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1 to 30:1) to give the desired product which was a brown solid; mp: 74-75℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.39 (m, 4H), 7.07 (d, 8.4MHz, 3H), 3.70 (q, 7.1 MHz, 1H), 2.53(t, 7.6MHz, 2H), 1.59 (d, 6.8 MHz, 3H), 1.49-1.57(m, 2H), 1.25-1.35(m, 2H), 0.89(t, 7.4 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 141.0, 138.9, 135.4, 129.1, 128.7, 127.7, 127.5, 119.7, 48.0, 35.0, 33.6, 22.2, 18.5, 13.9; HRMS(m/z): [M+H] calcd. for $C_{19}H_{24}NO$, 282.1852; found, 282.1862.

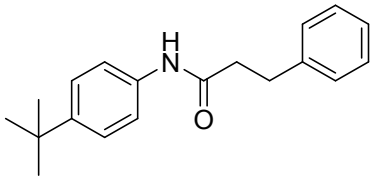


4d **N-(4-butylphenyl)-3-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography

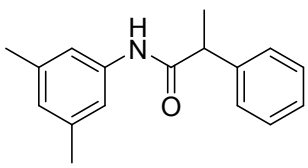
using petroleum ether/diethyl ether (30 : 1 to 10 : 1) to give the desired product which was a yellow solid; mp: 69-70℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27-7.33 (m, 4H), 7.19-7.23 (m, 3H), 7.13(brs, 1H), 7.09 (d, 8.4MHz, 2H), 3.04 (t, 7.6 MHz, 2H), 2.63 (t, 7.6 MHz, 2H), 2.55(t, 7.8MHz, 2H), 1.52-1.59(m, 2H), 1.25-1.37(m, 2H), 0.91(t, 7.4MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 140.7, 139.0, 135.3, 128.8, 128.6, 128.4, 126.3, 120.0, 39.4, 35.0, 33.6, 31.6, 22.2, 13.9; HRMS(m/z): [M+H] calcd. for $C_{19}H_{24}NO$, 282.1852; found, 282.1858.



3e **N-(4-tert-butylphenyl)-2-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (15:1) to give the desired product which was a white solid; mp: 97-98℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31-7.39 (m, 6H), 7.25-7.29 (m, 3H), 7.09 (brs, 1H), 3.71 (q, 7.2 MHz, 1H), 1.59(d, 7.2MHz, 3H), 1.27 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 147.2, 141.0, 135.2, 129.1, 127.7, 127.5, 125.6, 119.4, 48.0, 34.3, 31.3, 18.5; HRMS(m/z): [M+H] calcd. for $C_{19}H_{24}NO$, 282.1852; found, 282.1849.

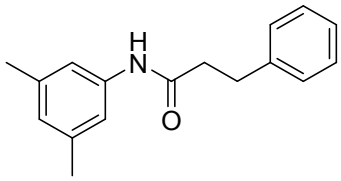


4e **N-(4-tert-butylphenyl)-3-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (15:1) to give the desired product which was a white solid; mp: 99-101℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.36 (m, 2H), 7.25-7.30 (m, 5H), 7.19-7.22 (m, 3H), 3.03 (t, 7.6 MHz, 2H), 2.63 (t, 7.6 MHz, 2H), 1.28(s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 147.2, 140.6, 135.1, 128.6, 128.4, 126.3, 125.7, 119.8, 39.3, 34.3, 31.6, 31.3; HRMS(m/z): [M+H] calcd. for $C_{19}H_{24}NO$, 282.1852; found, 282.1855.

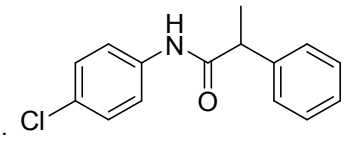


3f **N-(3,5-dimethylphenyl)-2-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50 : 1 to 20 : 1) to give the desired product which was a white solid; mp:

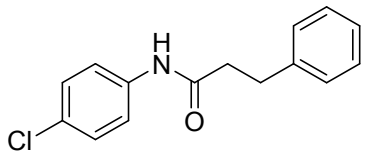
86-87℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48(brs, 1H), 7.21-7.33 (m, 5H), 7.08(s, 2H), 6.68(s, 1H), 3.69 (q, 7.1 MHz, 1H), 2.20(s, 6H), 1.55 (d, 7.2 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 141.0, 138.3, 137.7, 128.8, 127.5, 127.2, 117.6, 29.6, 21.1, 18.5; HRMS(m/z): [M+H] calcd. for $C_{17}H_{20}NO$, 254.1539; found, 254.1544.



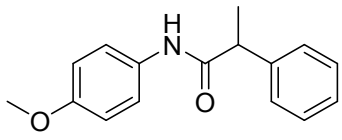
4f **N-(3,5-dimethylphenyl)-3-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20 : 1 to 10 : 1) to give the desired product which was a white solid; mp: 82-84℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.28(s, 1H), 7.08-7.20(m, 7H), 6.66(s, 1H), 2.94 (t, 7.6 MHz, 2H), 2.57 (t, 7.8 MHz, 2H), 2.15(s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 140.5, 138.1, 137.7, 128.2, 128.0, 125.9, 125.7, 118.0, 38.6, 31.4, 21.0; HRMS(m/z): [M+H] calcd. for $C_{17}H_{20}NO$, 254.1539; found, 254.1530.



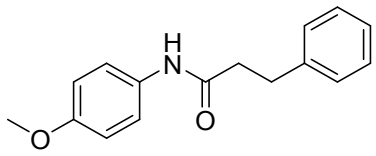
3g **Benzeneacetamide**[5]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a brown solid; mp: 114-115℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36-7.40 (m, 4H), 7.26-7.32 (m, 3H), 7.06-7.08 (m, 2H), 6.98 (brs, 1H), 3.70 (q, 7.07 MHz, 1H), 1.54 (d, 7.2 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 140.7, 136.4, 129.2, 128.8, 127.6, 120.9, 48.0, 18.5.



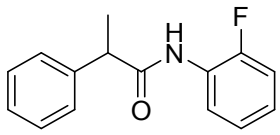
4g **N-(4-chlorophenyl)-3-phenylpropanamide**[7]:The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid. Mp=136-137℃.1H NMR (400 MHz, CDCl3) δ 7.36(d, 8.4 MHz, 2H), 7.20-7.31 (m, 7H), 7.17(brs, 1H), 3.03 (t, 7.6 MHz, 2H), 2.64 (t, 7.6 MHz, 2H); **13C NMR** (100 MHz, CDCl3) δ 170.4, 140.4, 136.2, 129.2, 128.9, 128.7, 128.3, 126.5, 121.1, 39.4, 31.5.



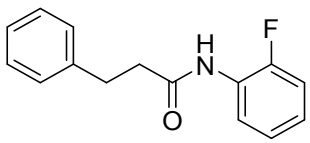
3h **N-(4-methoxyphenyl)-2-phenylpropanamide**[3]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (6:1) to give the desired product which was a light brown solid; mp: 139-140℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.40 (m, 4H), 7.28-7.33 (m, 3H), 7.04 (brs, 1H), 6.78-6.82 (m, 2H), 3.75 (s,3H), 3.70 (q, 7.1 MHz, 1H), 1.59(d, 6.8MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 156.3, 141.0, 130.9, 129.1, 127.7, 127.5, 121.6, 114.0, 55.4, 47.9, 18.6.



4h **N-(4-methoxyphenyl)-3-phenylpropanamide**[6]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (6:1) to give the desired product which was a light yellow solid; mp: 127-129℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.62 (t, 7.6 MHz, 2H), 3.03 (t, 7.6 MHz, 2H), 3.76(s, 3H), 6.79-6.82 (m, 2H), 7.19-7.22 (m, 4H), 7.27-7.35 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 156.4, 140.7, 130.8, 128.6, 128.4, 126.3, 121.9, 114.0, 55.4, 39.2, 31.6.

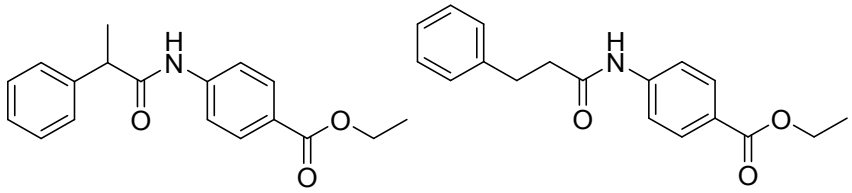


3i **N-(2-fluorophenyl)-2-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30 : 1 to 10 : 1) to give the desired product which was a white solid; mp: 85-86℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.29-8.33 (m, 1H), 7.36-7.41 (m, 4H), 7.29-7.34 (m, 2H), 7.05-7.11 (m, 1H), 6.98-7.02 (m, 2H), 3.76 (q, 7.2 MHz, 1H), 1.62 (d, 7.2 MHz, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.67; ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 152.3 (*J* = 241.6 Hz), 140.5, 129.1, 127.6, 127.6, 126.3 (*J* = 9.9 Hz), 124.4 (*J* = 3.7 Hz), 124.2 (*J* = 7.6 Hz), 121.5, 114.6 (*J* = 19.0 Hz), 48.2, 18.4; HRMS(m/z): [M+H] calcd. for $C_{15}H_{15}FNO$, 244.1132; found, 244.1129.



4i **N-(2-fluorophenyl)-3-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light yellow solid; mp: 91-94℃; **^{1}H**

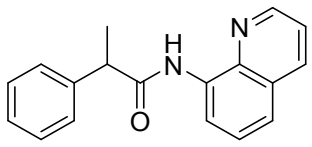
NMR (400 MHz, $CDCl_3$) δ 8.29-8.33(m, 1H), 7.20-7.34(m, 6H), 7.00-7.14(m, 3H), 3.07 (t, 7.8 MHz, 2H), 2.72 (t, 7.6MHz, 2H); **^{19}F NMR**(376 MHz, $CDCl_3$) δ -116. 79; ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 152.2 (*J* = 241.3 Hz), 140.4, 128.6, 128.3, 126.4, 126.2 (*J* = 9.9 Hz), 124.5(*J* = 3.6 Hz), 124.2(*J* = 7.6 Hz), 121.8, 114.7 (*J* = 19.1 Hz), 39.4, 31.3. HRMS(m/z): [M+H] calcd. for $C_{15}H_{15}FNO$, 244.1132; found, 244.1122.



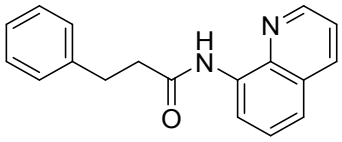
3j **ethyl 4-(2-phenylpropanamido)benzoate**[5]**:**

4j **ethyl 4-(3-phenylpropanamido)benzoate**[4]**:**

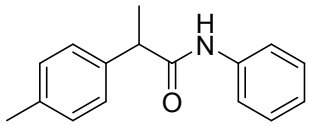
The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30 : 1 to 5 : 1) to give the desired product which were white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93-7.99 (m, 4H), 7.49-7.54 (m, 4H), 7.20-7.41 (m, 12H), 4.28-4.37 (m, 4H), 3.74 (q, 7.1 MHz, 1H), 3.05 (t, 7.6 MHz, 2H), 2.69 (t, 7.6 MHz, 2H), 1.60 (d, 7.2MHz, 3H), 1.35-1.40 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 170.7, 166.2, 166.1, 141.9, 140.5, 140.4, 130.7, 130.6, 129.2, 128.6, 128.3, 127.7, 127.6, 126.4, 125.8, 118.8, 118.7, 60.8, 48.2, 39.4, 31.3, 18.5, 14.3.



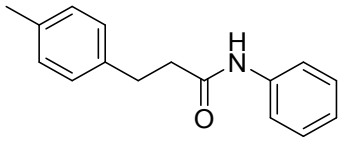
3k **2-phenyl-N-(quinolin-8-yl)propanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30 : 1 to 20 : 1) to give the desired product which was a white solid; mp: 209-210℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.91 (brs, 1H), 8.77 (dd, 1.6 MHz, 7.6 MHz, 1H), 8.69 (dd, 1.6 MHz, 4.0 MHz, 1H), 8.10 (dd, 1.6 MHz, 8.0 MHz, 1H), 7.44-7.52 (m, 4H), 7.36-7.40 (m, 3H), 7.27-7.31(m, 1H), 3.80 (q, 7.1 MHz, 1H), 1.69 (d, 7.2 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 148.0, 141.1, 138.4, 136.2, 134.5, 128.9, 127.8, 127.7, 127.3, 127.3, 121.5, 121.4, 116.3, 48.6, 18.6; HRMS(m/z): [M+H] calcd. for $C_{18}H_{17}N_2O$, 277.1335; found, 277.1342.



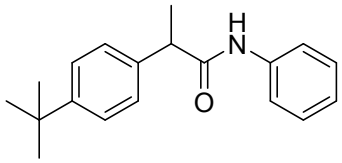
4k **3-phenyl-N-(quinolin-8-yl)propanamide**[8]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1 to 30:1) to give the desired product which was a grey solid; mp: 58-60℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.81 (brs, 1H), 8.79 (dd, 1.2 MHz, 7.2 MHz, 1H), 8.77 (dd, 1.6 MHz, 4.0 MHz, 1H), 8.15 (dd, 1.6 MHz, 8.4 MHz, 1H), 7.48-7.56 (m, 2H), 7.43-7.46 (m, 1H), 7.18-7.31 (m, 5H), 3.15 (t, 8.0 MHz, 2H), 2.89 (t, 8.0 MHz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 148.0, 140.7, 138.2, 136.4, 134.4, 128.5, 128.4, 127.9, 127.4, 126.2, 121.5, 121.4, 116.5, 39.7, 31.4.



3l **N-phenyl-2-p-tolylpropanamide**[5]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light grey brown solid; mp: 119-120℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40-7.42 (m, 2H), 7.24-7.28 (m, 4H), 7.17-7.19 (m, 2H), 7.04-7.08 (m, 2H), 3.68 (q, 7.1 MHz, 1H), 2.35(s, 3H), 1.58 (d, 6.8 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 137.8, 137.3, 129.8, 128.9, 127.6, 124.1, 119.6, 47.7, 21.0, 18.5.

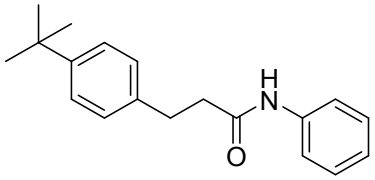


4l **N-phenyl-3-(p-tolyl)propanamide**[9]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light brown solid; mp: 114-115℃ (lit.[9] 120-121℃); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42-7.44 (m, 2H), 7.22-7.30 (m, 3H), 7.06-7.13 (m, 5H), 3.00 (t, 7.6 MHz, 2H), 2.63 (t, 7.6 MHz, 2H), 2.31(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 137.7, 137.5, 135.9, 129.3, 128.9, 128.2, 124.2, 119.9, 39.5, 31.1, 21.0.

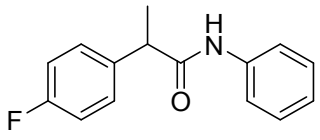


3m **2-(4-tert-butylphenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 120-

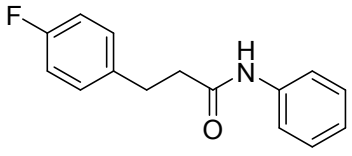
121℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.44 (m, 4H), 7.25-7.30 (m, 4H), 7.04-7.10 (m, 2H), 3.70 (q, 7.1 MHz, 1H), 1.59(d, 7.2MHz, 3H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 150.5, 137.9, 137.7, 128.9, 127.3, 126.0, 124.1, 119.7, 47.6, 34.5, 31.3, 18.5; HRMS(m/z): [M+H] calcd. for $C_{19}H_{24}NO$, 282.1852; found, 282.1855.



4m **3-(4-tert-butylphenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 110-113℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.04-7.42 (m, 2H), 7.25-7.33 (m, 4H), 7.16-7.18 (m, 3H), 7.06-7.10 (m, 1H), 3.02 (t, 7.6 MHz, 2H), 2.65 (t, 7.6 MHz, 2H), 1.31(s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 149.2, 137.7, 137.5, 128.9, 128.0, 125.5, 124.2, 119.9, 39.4, 34.4, 31.3, 31.0; HRMS(m/z): [M+H] calcd. for $C_{19}H_{24}NO$, 282.1852; found, 282.1848.

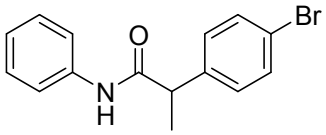


3n **2-(4-fluorophenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light grey solid; mp: 112-113℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30-7.36 (m, 2H), 7.17-7.27 (m, 4H), 7.12 (brs, 1H), 6.95-7.02 (m, 3H), 3.61 (q, 7.1 MHz, 1H), 1.49 (d, 7.2 MHz, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114. 82; ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 162.1 (*J* = 244.7 Hz), 137.7, 136.6 (*J* = 3.2 Hz), 129.2 (*J* = 8.0 Hz), 128.9, 124.4, 119.7, 115.9 (*J* = 21.2 Hz), 47.3, 18.8; HRMS(m/z): [M+H] calcd. for $C_{15}H_{15}FNO$, 244. 1132; found, 244.1143.

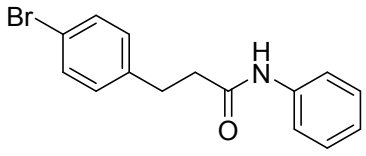


4n **3-(4-fluorophenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light yellow solid; mp: 128-129℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43-7.45 (m, 2H), 7.26-7.31 (m, 3H), 7.15-7.18 (m, 2H), 7.08-7.11 (m, 1H), 6.94-6.98 (m, 2H), 3.01 (t, 7.6 MHz, 2H), 2.62 (t, 7.4MHz, 2H); **^{19}F NMR** (376 MHz, CDCl3) δ -116. 79;^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 161.5 (*J* = 242.6 Hz), 137.6, 136.3 (*J* = 5.4 Hz), 129.8 (*J* =

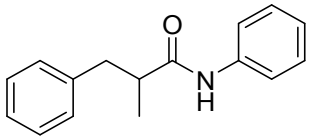
7.8 Hz), 128.9, 124.4, 119.9, 115.3 (J = 21.1 Hz), 39.4, 30.6; HRMS(m/z): [M+H] calcd. for $C_{15}H_{15}FNO$, 244.1132; found, 244.1130.



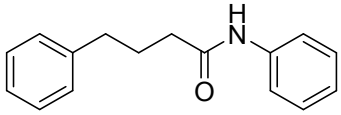
3o **2-(4-bromophenyl)-N-phenylpropanamide**[5]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 162-165℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42-7.50 (m, 4H), 7.23-7.30 (m, 4H), 7.06-7.13 (m, 2H), 3.66 (q, 6.9 MHz, 1H), 1.56 (d, 6.8 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 139.9, 137.7, 132.2, 129.4, 129.0, 124.5, 121.5, 119.8, 47.6, 18.7.



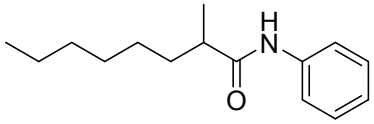
4o **3-(4-bromophenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 158-160℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.45 (m, 4H), 7.25-7.31 (m, 3H), 7.07-7.12 (m, 3H), 2.99 (t, 7.6 MHz, 2H), 2.61 (t, 7.6 MHz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 139.6, 137.6, 131.6, 130.1, 129.0, 124.4, 120.1, 119.9, 39.0, 30.8; HRMS(m/z): [M+H] calcd. for $C_{15}H_{15}BrNO$, 304.0332; found, 304.0332.



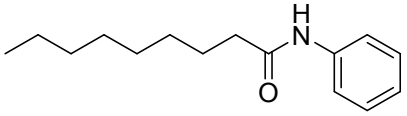
3p **3-cyano-2-methyl-N-phenylpropanamide**[3]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a white solid; mp: 125-126℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.36 (m, 2H), 7.19-7.30 (m, 7H), 7.05-7.09 (m, 1H), 6.88 (brs, 1H), 3.01-3.06 (m, 1H), 2.74-2.80 (m, 1H), 2.54-2.63 (m, 1H), 1.28 (d, 6.8 MHz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 139.6, 137.6, 128.9, 128.8, 128.5, 126.4, 124.2, 120.0, 44.7, 40.5, 17.7.



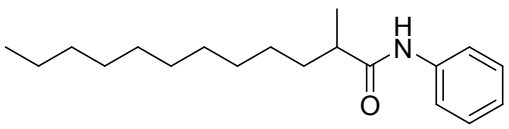
4p **N,4-diphenylbutanamide**[2]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a white solid; mp: 91-92℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48-7.50 (m, 2H), 7.25-7.32 (m, 5H), 7.17-7.21 (m, 3H), 7.07-7.11 (m, 1H), 2.69 (t, 7.4 MHz, 2H), 2.33 (t, 7.4 MHz, 2H), 2.02-2.09 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 141.3, 137.8, 128.9, 128.5, 128.4, 126.0, 124.2, 119.8, 36.7, 35.0, 26.8.



3q **2-methyl-N-phenyloctanamide**[10]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1) to give the desired product which was a white solid; mp: 61-64℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.55 (m, 2H), 7.26-7.33 (m, 3H), 7.08-7.12 (m, 1H), 2.30-2.37 (m, 1H), 1.60-1.78 (m, 2H), 1.22-1.30 (m, 11H), 0.85-0.88 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0, 138.0, 128.9, 124.1, 119.8, 42.7, 34.5, 31.7, 29.3, 27.5, 22.6, 17.9, 14.0.

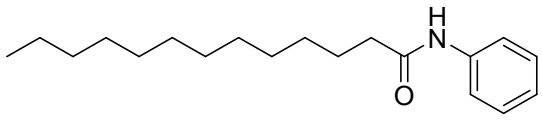


4q **N-phenylnonanamide**[2]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1) to give the desired product which was a white solid; mp: 51-52℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51-7.53 (m, 2H), 7.26-7.35 (m, 3H), 7.08-7.11 (m, 1H), 2.32-2.37 (m, 2H), 1.59-1.76 (m, 2H), 1.26-1.34 (m, 10H), 0.86-0.89 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 137.9, 128.9, 124.1, 119.8, 37.8, 31.8, 29.3, 29.3, 29.1, 25.6, 22.6, 14.1.

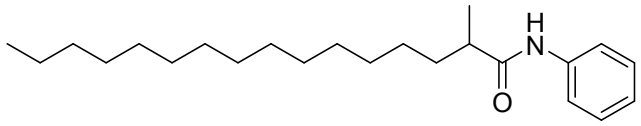


3r **2-methyl-N-phenyldodecanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (60:1 to 30:1) to give the desired product which was a light yellow solid; mp: 47-48℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.55 (m, 2H), 7.29-7.33 (m, 3H), 7.24 (brs, 1H), 7.08-7.11 (m, 1H), 2.28-2.37 (m, 2H), 1.41-1.78 (m, 4H), 1.22-1.26 (m, 16H), 0.86-0.90 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0, 138.0, 128.9, 124.1, 119.8, 34.5, 33.9, 31.9, 29.6, 29.5, 29.5, 29.3,

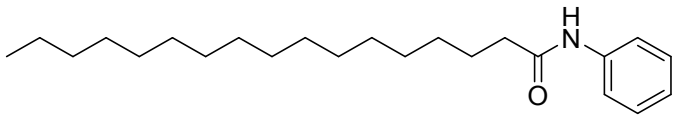
27.5, 24.7, 22.7, 17.9, 14.1; HRMS(m/z): [M+H] calcd. for $C_{19}H_{32}NO$, 290.2478; found, 290.2491.



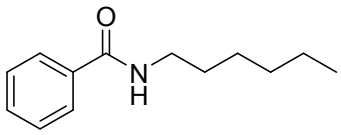
4r **N-phenyltridecanamide**[11]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30:1) to give the desired product which was a light yellow solid; mp: 70-71℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51-7.53 (m, 2H), 7.26-7.33 (m, 3H), 7.07-7.11 (m, 1H), 2.32-2.37 (m, 2H), 1.59-1.75 (m, 2H), 1.25-1.31 (m, 18H), 0.86-0.90 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 138.0, 128.9, 124.1, 119.8, 37.8, 31.9, 29.6, 29.6, 29.5, 29.4, 29.3, 29.3, 28.7, 25.6, 22.7, 14.1.



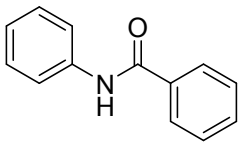
3s **2-methyl-N-phenylhexadecanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (100:1) to give the desired product which was a white solid; mp: 84-85℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.54-7.56 (m, 2H), 7.26-7.30 (m, 2H), 7.06-7.09 (m, 1H), 2.32-2.42 (m, 1H), 1.59-1.75 (m, 2H), 1.16-1.26 (m, 27H), 0.86-0.89 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1, 138.0, 128.9, 124.1, 119.8, 42.6, 34.4, 31.9, 29.7, 29.6, 29.6, 29.6, 29.5,29.3, 27.5, 22.7, 17.9, 14.1; HRMS(m/z): [M+H] calcd. for $C_{23}H_{40}NO$, 346.3104; found, 346.3105.



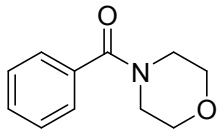
4s **N-phenylheptadecanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (100:1) to give the desired product which was a white solid; mp: 86-87℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51-7.53 (m, 2H), 7.26-7.33 (m, 3H), 7.08-7.11 (m, 1H), 2.33-2.37 (m, 2H), 1.59-1.76 (m, 2H), 1.25-1.32 (m, 26H), 0.86-0.90 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 137.9, 128.9, 124.1, 119. 8, 37.8, 33.8, 31.9, 29.7, 29.6, 29.6, 29.5, 29.4, 29.3, 29.3, 29.1, 25.6, 24.8, 22.7, 14.1; HRMS(m/z): [M+H] calcd. for $C_{23}H_{40}NO$, 346.3104; found, 346.3114.



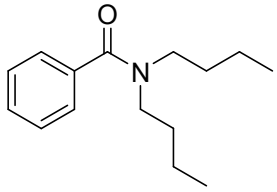
7a **N-hexylbenzamide**[12]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77-7.79 (m, 2H), 7.44-7.48 (m, 2H), 7.36-7.40 (m, 2H), 6.65(brs, 1H), 3.38-3.43 (m, 2H), 1.55-1.62 (m, 2H), 1.26-1.36 (m, 6H), 0.86-0.90 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 134.7, 131.0, 128.3, 126.8, 40.0, 31.4, 29.5, 26.6, 22.4, 13.9.



7b **N-Phenyl-benzamide**[1]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light yellow solid; mp: 163℃(lit.**1** 162-163℃); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86-7.88 (m, 3H), 7.64-7.66 (m, 2H), 7.53-7.58 (m, 1H), 7.46-7.50 (m, 2H), 7.35-7.39 (m, 2H), 7.14-7.18 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 137.9, 135.0, 131.8, 129.1, 128.8, 127.0, 124.6, 120.2.

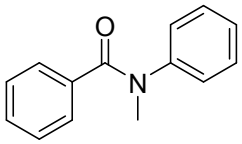


7c **Morpholino(phenyl)methanone**[13]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1 to 2:1) to give the desired product which was a white solid; mp: 68-70℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40-7.44 (m, 5H), 3.38-3.80 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 135.2, 129.8, 128.4, 127.0, 66.8, 48.1, 42.4.

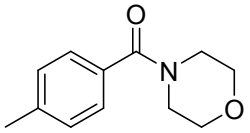


7d **N,N-Dibutylbenzamide**[14]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1 to 10:1) to give the desired product which was a light yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31-7.36

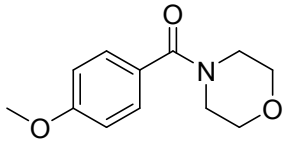
(m, 5H), 3.18-3.49 (m, 4H), 1.65 (m, 2H), 1.39-1.47 (m, 4H), 1.11-1.13 (m, 2H), 0.92-0.97 (m, 3H), 0.77-0.86 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 137.2, 128.8, 128.1, 126.2, 48.5, 44.2, 30.6, 29.4, 20.1, 19.5, 13.7, 13.4.



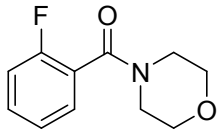
7e **N-methyl-N-phenylbenzamide**[13]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50 : 1 to 5 : 1) to give the desired product which was a light brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27-7.30 (m, 2H), 7.19-7.23 (m, 3H), 7.10-7.16 (m, 3H), 7.02-7.04 (m, 2H), 3.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 144.7, 135.8, 129.5, 129.0, 128.6, 127.6, 126.8, 126.4, 38.3.



7f **N-(4-toluoyl)morpholine**[15]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1) to give the desired product which was a white oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27-7.30 (m, 2H), 7.19-7.21 (m, 2H), 3.50-3.69 (m, 8H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 140.0, 132.2, 129.0, 127.1, 66.8, 48.2, 42.6, 21.3.

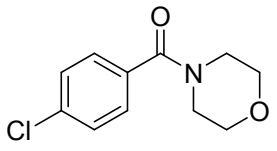


7g **N-(4-methoxybenzoyl)morpholine**[15]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1) to give the desired product which was a brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.40 (m, 2H), 6.90-6.93 (m, 2H), 3.82 (s, 3H), 3.64-3.69 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 160.7, 129.0, 127.1, 113. 6, 66.7, 55.1, 42.8.

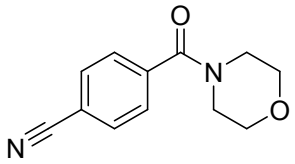


7h **4-(2-Fluorobenzoyl)-morpholine**[16]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1) to give the desired product which was a light yellow solid; mp: 73-74℃ (lit.**17** 75-

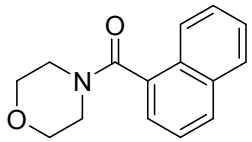
77℃); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39-7.44 (m, 2H), 7.20-7.27 (m, 1H), 7.08-7.13 (m, 1H), 3.77-3.83 (m, 4H), 3.64-3.65 (m, 2H), 3.35 (s, 2H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114. 94; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 158.0 (*J* = 246.8Hz), 131.5 (*J* = 8.0Hz), 129.2 (*J* = 3.6Hz), 124.8 (*J* = 3.4Hz), 123.6 (*J* = 17.5Hz), 115.8 (*J* = 21.4 Hz), 66.8, 66.7, 47.4, 42.4.



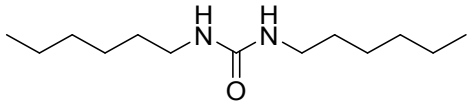
7i **N-(4-Chlorobenzoyl)piperidine**[14]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1 to 2:1) to give the desired product which was a light brown solid; mp: 66-68℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.42 (m, 4H), 3.38-3.80 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3, 136.0, 128.8, 128.6, 66.8, 48.1.



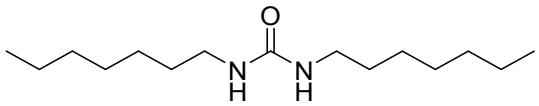
7j **4-(morpholine-4-carbonyl)benzonitrile**[15]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1 to 1 : 1) to give the desired product which was a light yellow solid; mp: 141-142℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73-7.75 (m, 2H), 7.51-7.54 (m, 2H), 3.39-3.80 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.2, 139.6, 132.5, 127.8, 117.9, 113.7, 66.7, 48.0, 42.5.



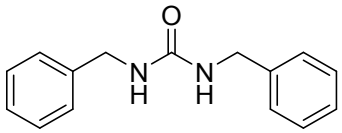
7k **morpholino(naphthalen-1-yl)methanone**[17]**:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1 to 2:1) to give the desired product which was a brown oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83-7.85 (m, 3H), 7.39-7.54 (m, 4H), 3.78-4.01 (m, 4H), 3.44-3.47 (m, 2H), 3.13-3.17 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.4, 133.6, 133.4, 129.4, 129.3, 128.4, 127.0, 126.4, 125.1, 124.5, 123.8, 67.0, 66.9, 47.5, 42.1.



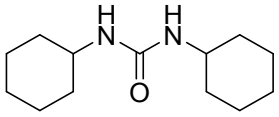
9a **1,3-dihexylurea**[18]**:** The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a brown oil; **^{1}H NMR** (400 MHz, DMSO-d6) δ 5.73 (brs, 1H), 3.50 (s, 1H), 2.92-3.11 (m, 3H), 1.24-1.44 (m, 16H), 0.84-0.86 (m, 6H); ^{13}C NMR (100 MHz, DMSO-d6) δ 158.1, 39.2, 31.1, 30.0, 26.1, 22.1, 13.9.



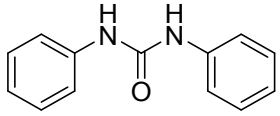
9b **1,3-diheptylurea**[18]**:** The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a yellow solid; mp: 65℃; **^{1}H NMR** (400 MHz, DMSO-d6) δ 2.95-3.11 (m, 4H), 1.24-2.02 (m, 20H), 0.86 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 40.5, 31.7, 30.3, 29.0, 26.9, 22.5, 14.0.



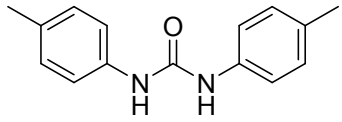
9c **1,3-Dibenzylurea**[19]**:** The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was light brown solid; mp: 209-210℃; **^{1}H NMR** (400 MHz, DMSO-d6) δ 7.24-7.31 (m, 10H), 6.44 (brs , 2H), 4.23 (s, 4H); ^{13}C NMR (100 MHz, DMSO-d6) δ 158.1, 140.9, 128.2, 127.0, 126.5, 40.0.



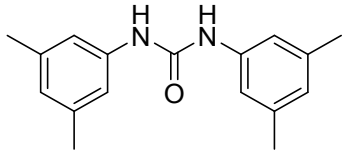
9d **1,3-dicyclohexylurea**[20]**:** The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a light grey solid; mp: 209-210℃; **^{1}HNMR** (400 MHz, $CDCl_3$) δ 3.46-3.41 (m, 2H) 1.10-1.95 (m, 20H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 49.1, 33.9, 25.6, 24.9.



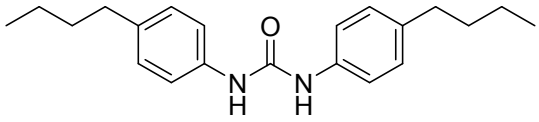
9e **1,3-diphenylurea**[3]**:** The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a white solid; mp: 227-232℃; **^{1}H NMR** (400 MHz, DMSO-d6) δ 8.68 (brs, 2H), 7.45-7.47 (m, 4H), 7.26-7.30 (m, 4H), 6.95-6.99 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d6) δ 152.5, 139.7, 128.8, 121.8, 118.1.



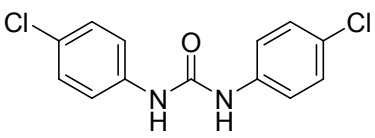
9f **1,3-Bis(4-methylphenyl)urea**[19]: The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was white solid; mp: 266-267℃ (lit.[20] 281-282℃); **^{1}H NMR** (400 MHz, DMSO-d6) δ 8.51 (brs, 2H), 7.33 (d, 7.6 MHz, 4H), 7.07 (d, 7.6 MHz, 4H), 2.24 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d6) δ 152.6, 137.2, 130.5, 129.1, 118.2, 20.3.



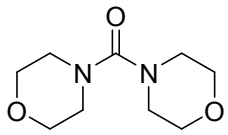
9g **1,3-bis(3,5-dimethylphenyl)urea**[21]**:** The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a dark brown solid; mp: 267℃; **^{1}H NMR** (400 MHz, DMSO-d6) δ 8.49 (brs, 2H), 7.07 (s, 4H), 6.60 (s, 2H), 2.22 (s, 12H) ; ^{13}C NMR (100 MHz, DMSO-d6) δ 152.4, 21.1, 139.6, 137.7, 123.3, 115.8.



9h **1,3-bis(4-butylphenyl)urea**[22]**:** The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a light brown solid; mp: 180-181℃ (lit.[20] 192-195℃); **^{1}H NMR** (400 MHz, DMSO-d6) δ 8.51 (brs, 2H), 7.34 (d, 8.4 MHz, 4H), 7.08 (d, 8.4 MHz, 4H), 2.49-2.52 (m, 4H), 1.45-1.55 (m, 4H), 1.25-1.34 (m, 4H), 0.89 (t, 7.2 MHz, 6H); **^{13}C NMR** (100 MHz, DMSO-d6) δ 152.6, 137.4, 135.5, 128.5, 118.2, 34.1, 33.3, 21.7, 13.8.

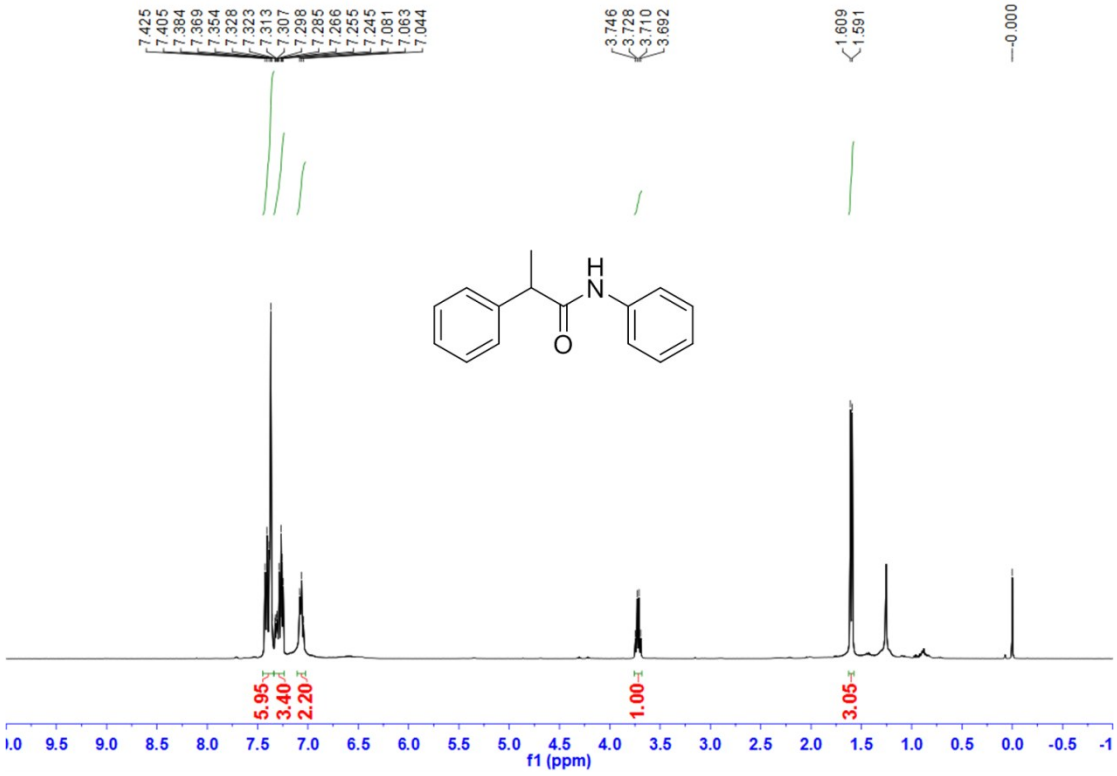


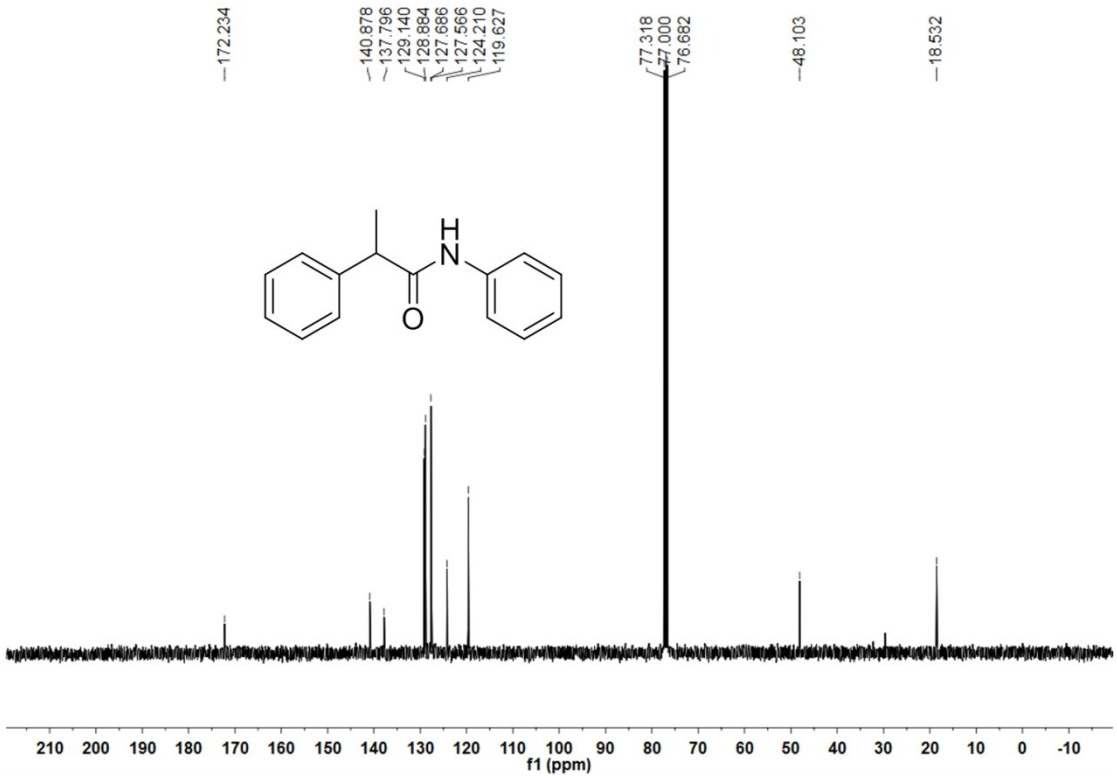
. 9i **1,3-Bis(4-chlorophenyl)urea**[19]: The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a brown solid; mp: 278-280℃ (lit.[20] 322-325℃); **^{1}H NMR** (400 MHz, DMSO-d6) δ 8.89 (brs, 2H), 7.48 (d, 8.0 MHz, 4H), 7.33 (d, 8.4 MHz, 4H); ^{13}C NMR (100 MHz, DMSO-d6) δ 152.3, 138.5, 128.6, 125.5, 119.8.

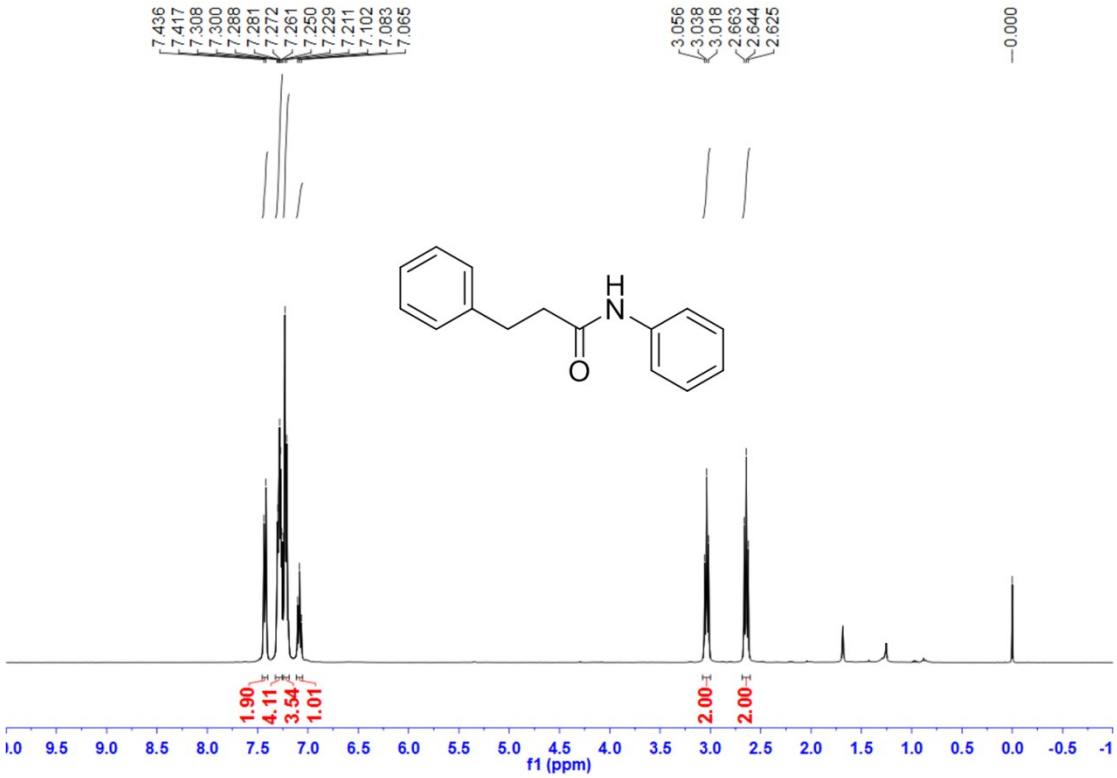


9j **4,4'-Carbonyldimorpholine**[23]: The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a brown sticky oil; **^{1}H NMR** (400 MHz, DMSO-d6) δ 3.13-3.57 (m, 16H); ^{13}C NMR (100 MHz, DMSO-d6) δ 162.6, 66.2, 65.9, 65.7, 46.8, 45.8, 40.6.

5. NMR spectra of the products



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7.083
7.065
3.056
3.038
3.018
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N
O
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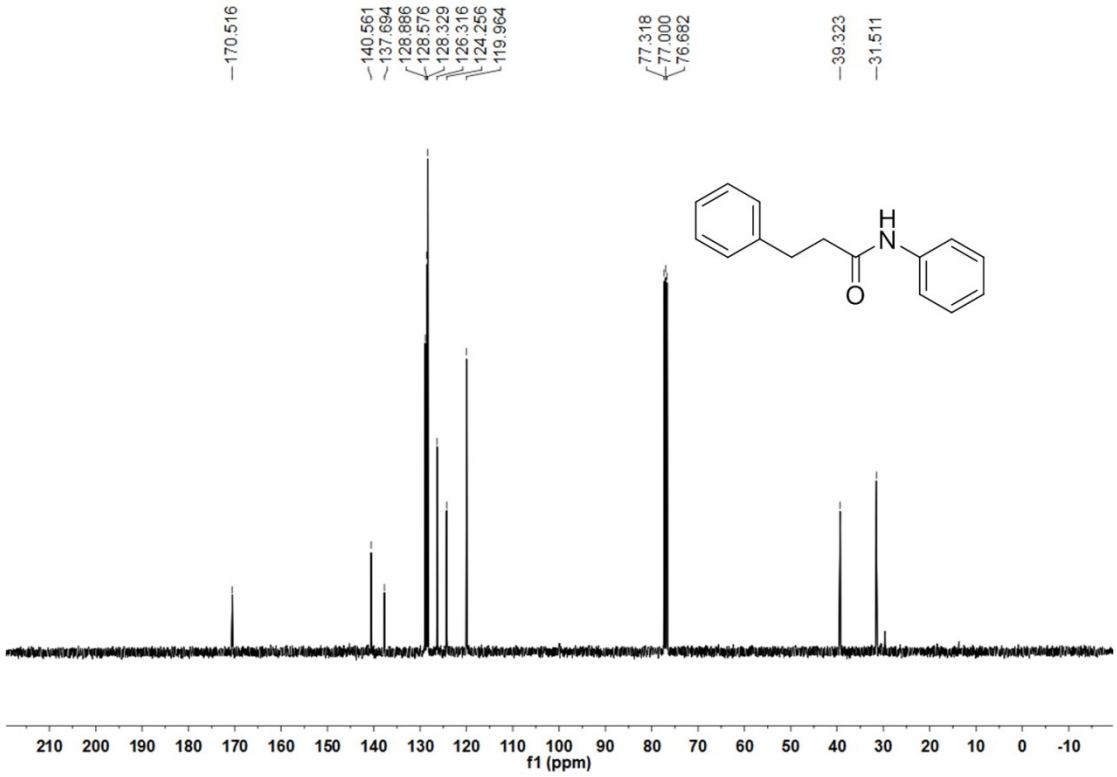
3.54
1.01
2.00
2.00
f1 (ppm)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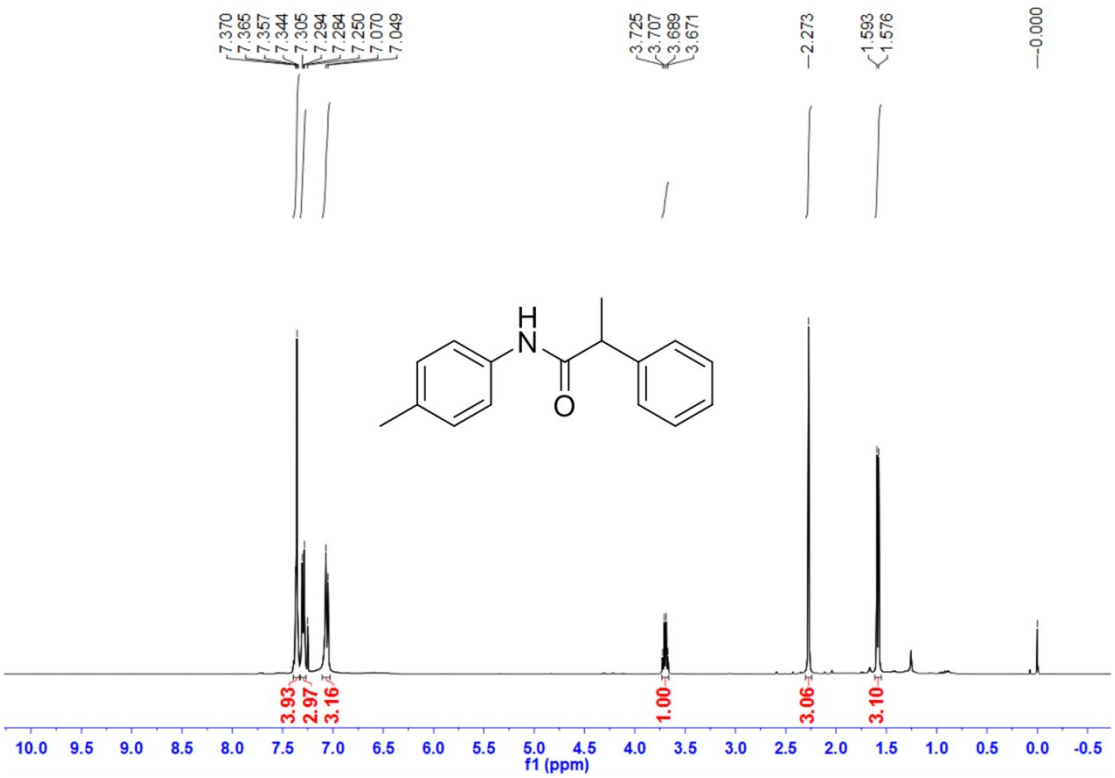
H
N
O
f1 (ppm)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7.294
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7.049
3.725
3.707
3.689
3.671
2.273
1.593
1.576
0.000
H
N
O
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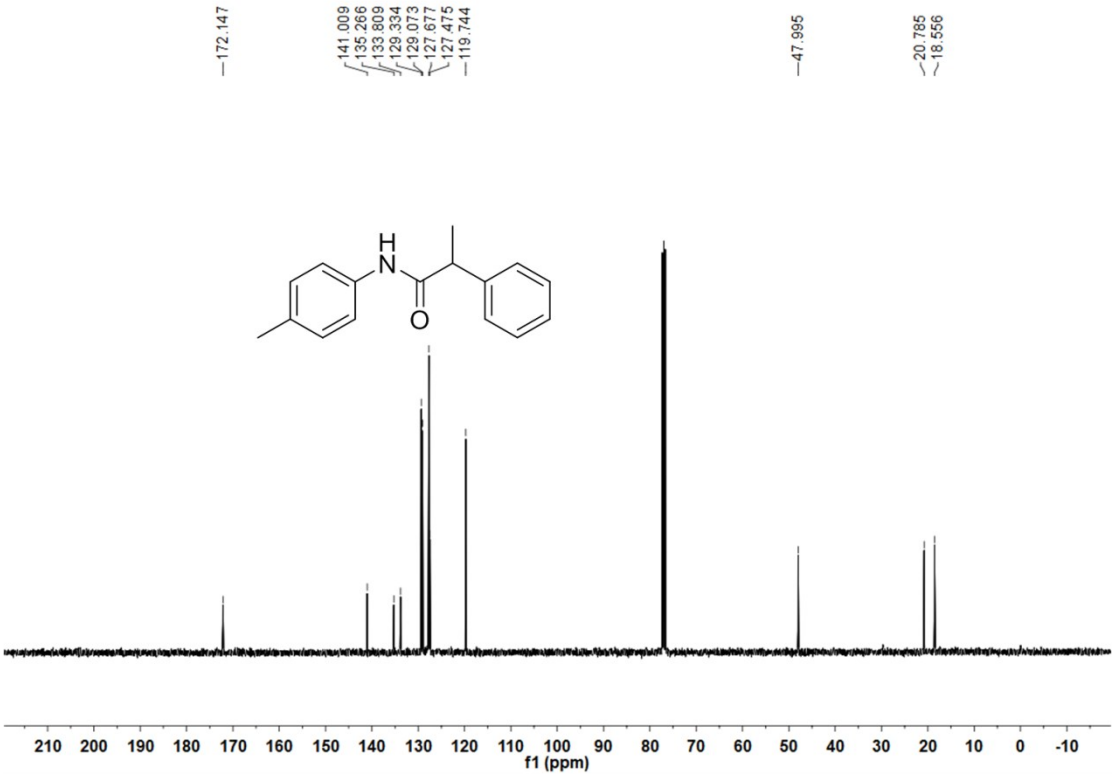
2.97
3.16
1.00
3.06
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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127.475
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47.995
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18.556
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N
O
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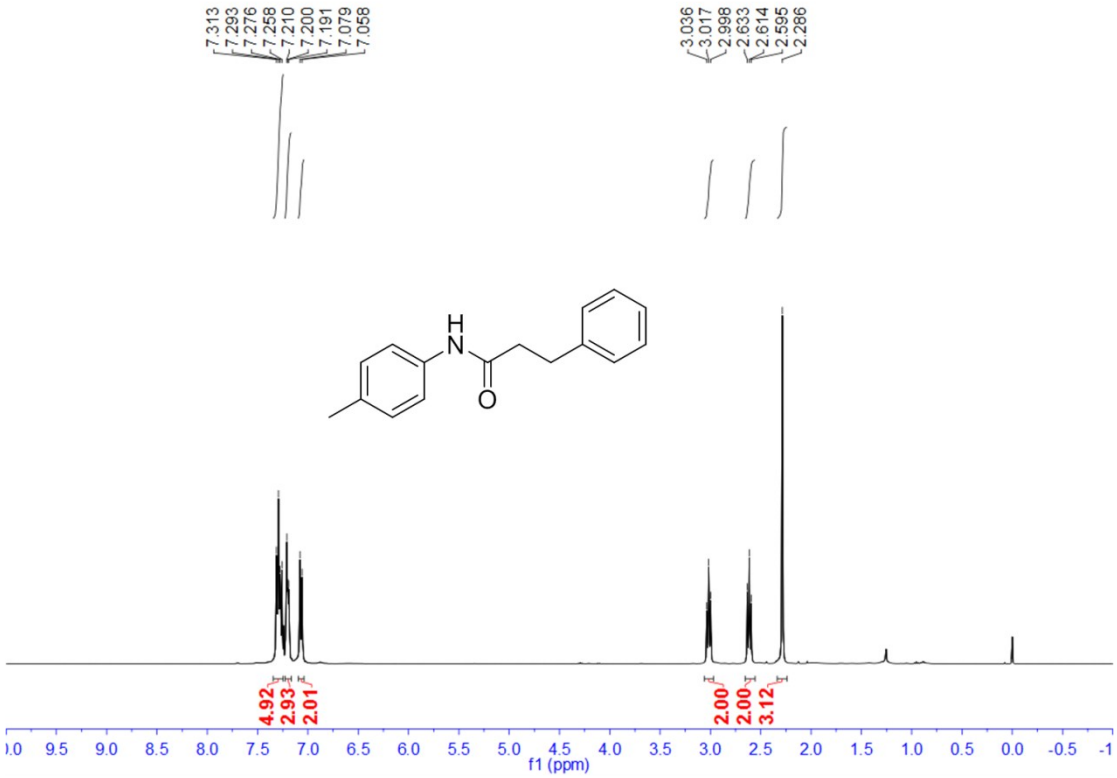
f1 (ppm)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7.079
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2.595
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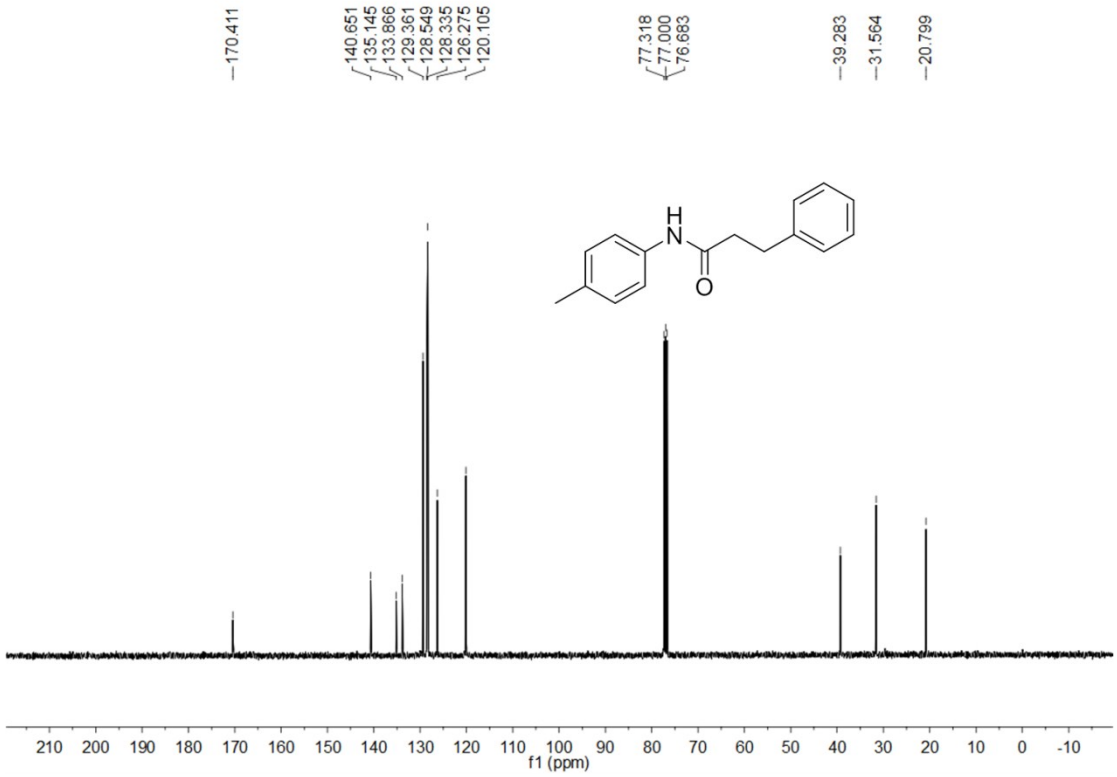
2.01
2.00
2.00
3.12
f1 (ppm)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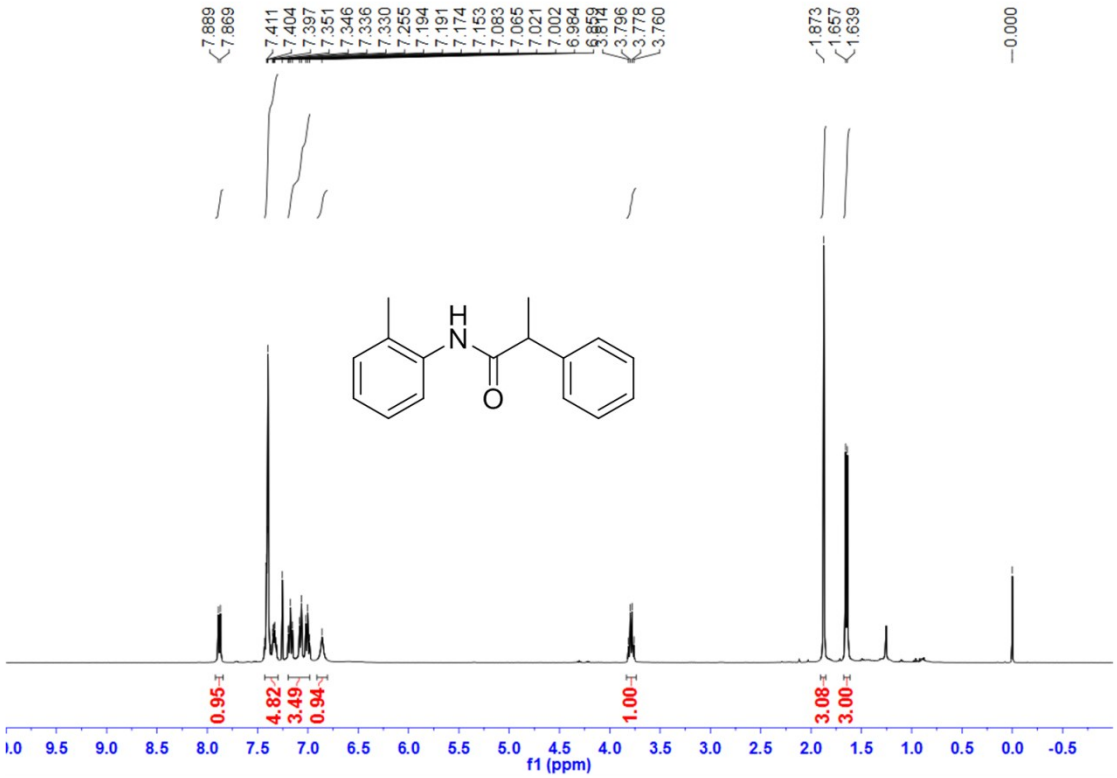
20.799
f1 (ppm)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7.153
7.083
7.065
7.021
7.002
6.984
3.796
3.778
3.760
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1.657
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4.82
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1.00
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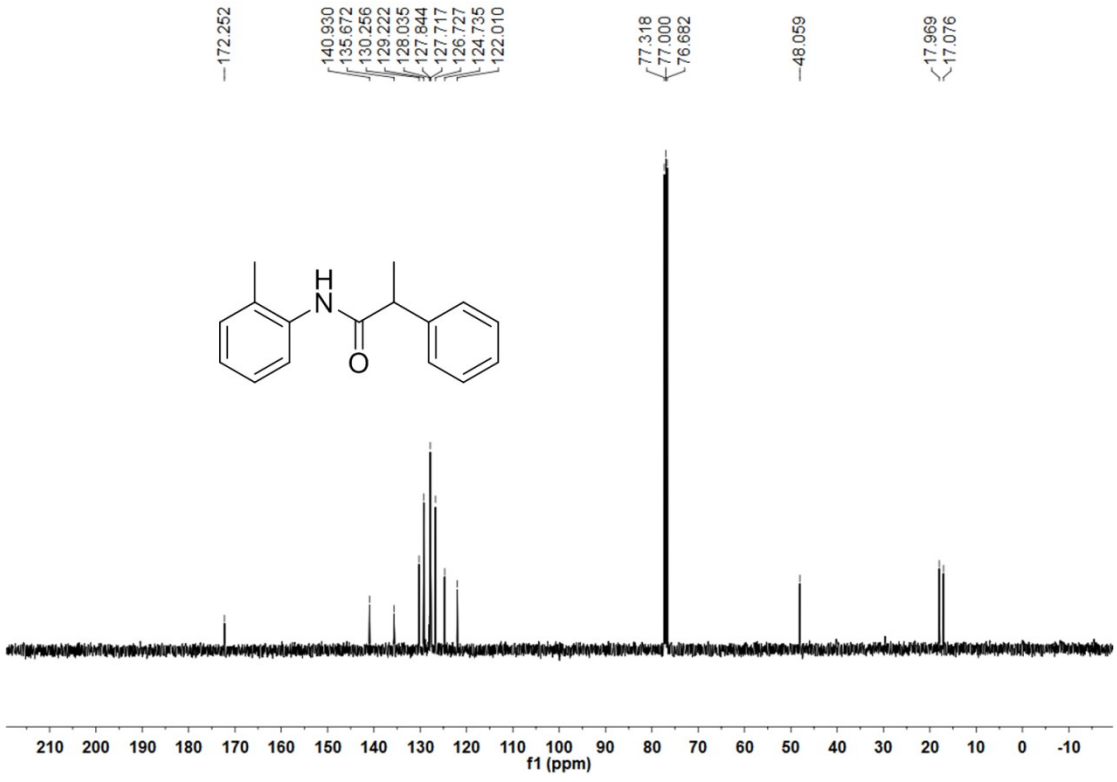
3.00
f1 (ppm)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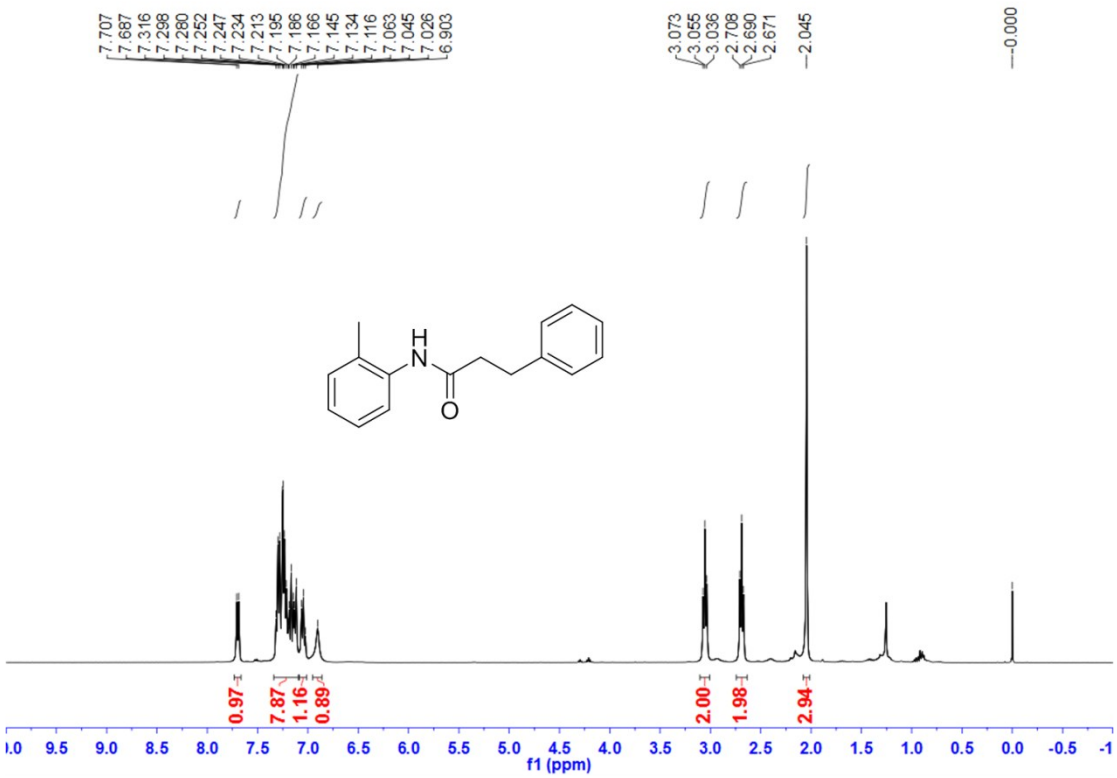
f1 (ppm)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7.234
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7.195
7.186
7.166
7.145
7.134
7.116
7.063
7.045
7.026
6.903
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3.055
3.036
2.708
2.690
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O
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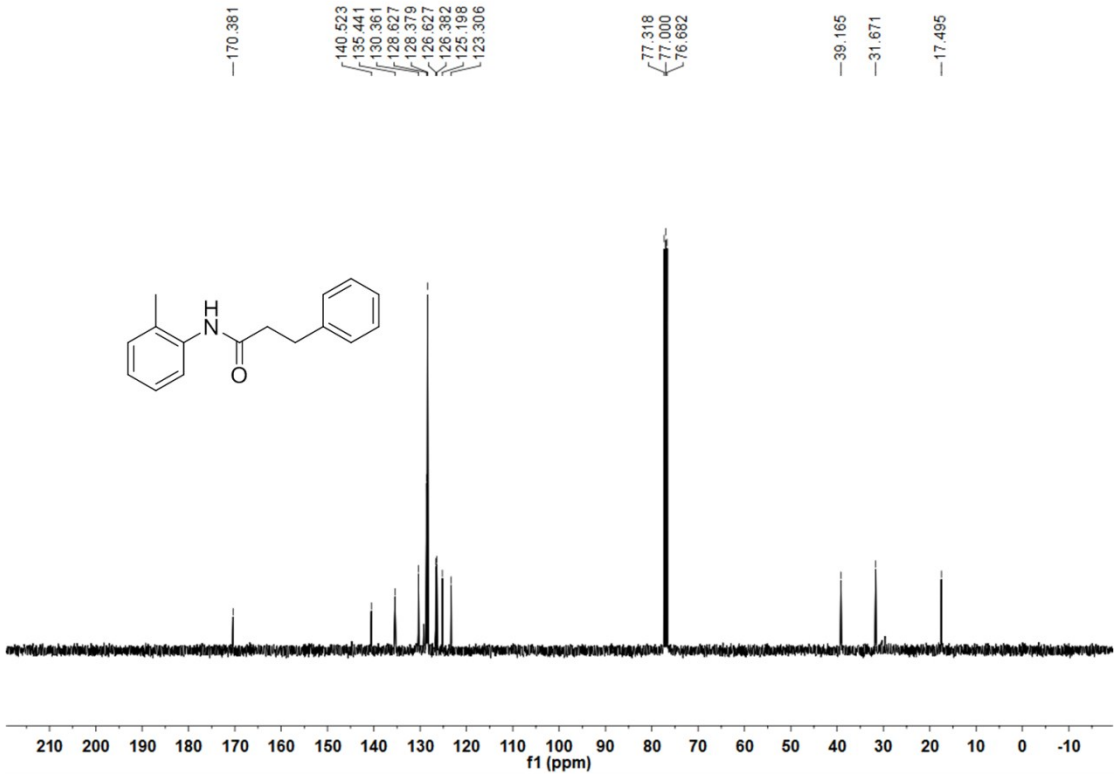
f1 (ppm)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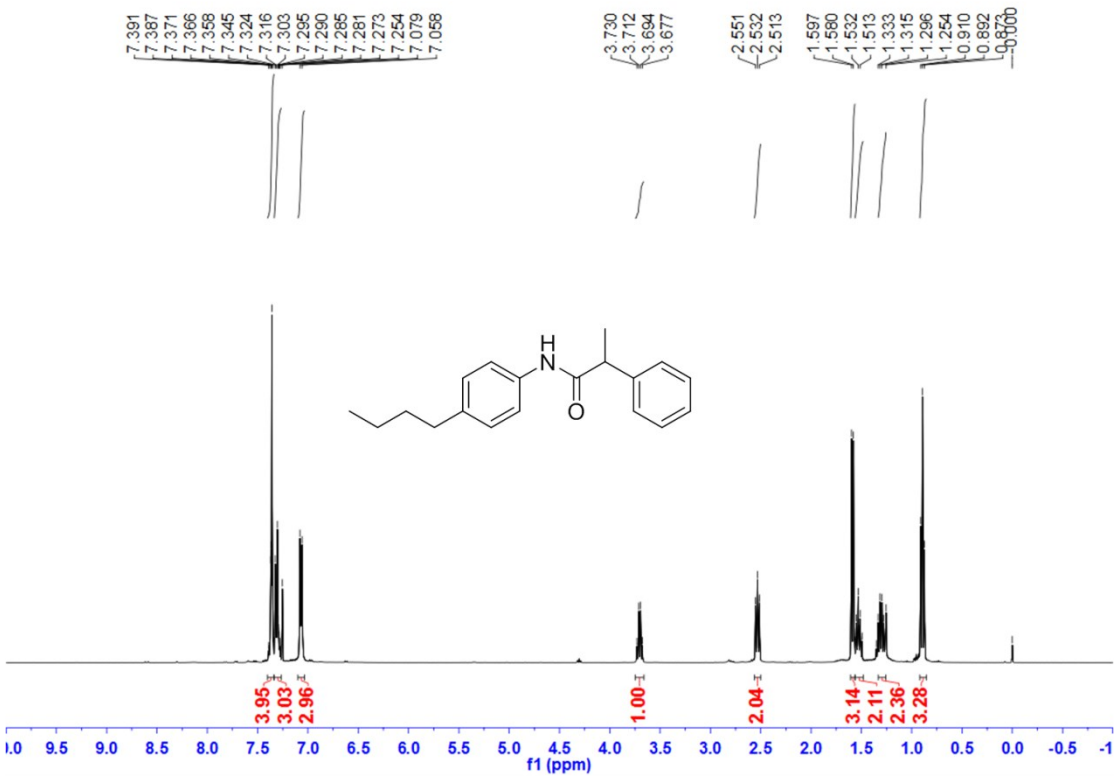
O
f1 (ppm)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7.273
7.254
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7.058
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3.712
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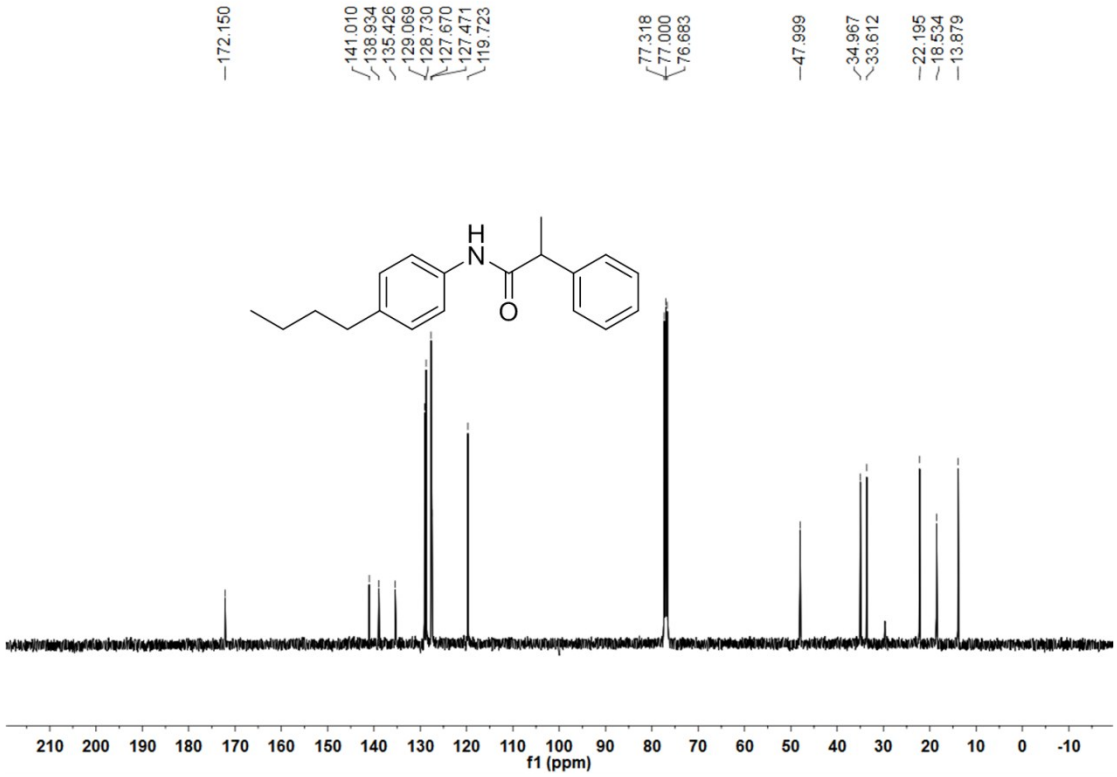
f1 (ppm)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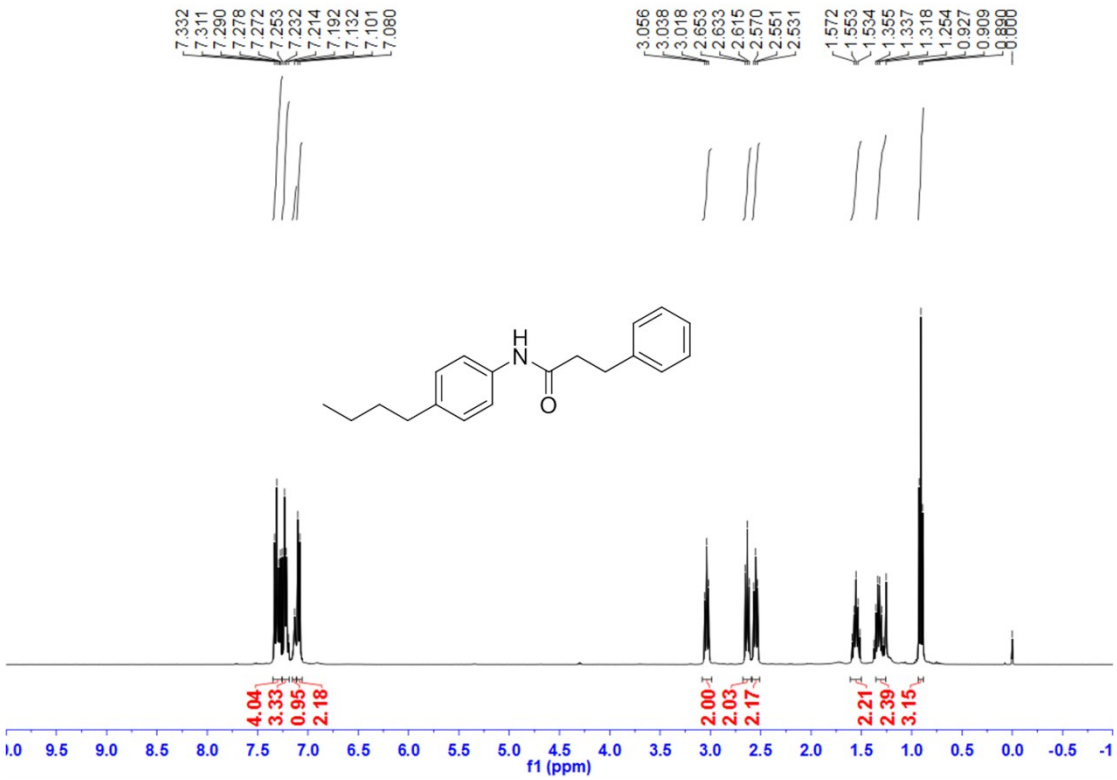
O
f1 (ppm)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2.570
2.551
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1.572
1.553
1.534
1.355
1.337
1.318
1.254
0.927
0.909
0.890
0.880
H
N
O
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0.95
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2.21
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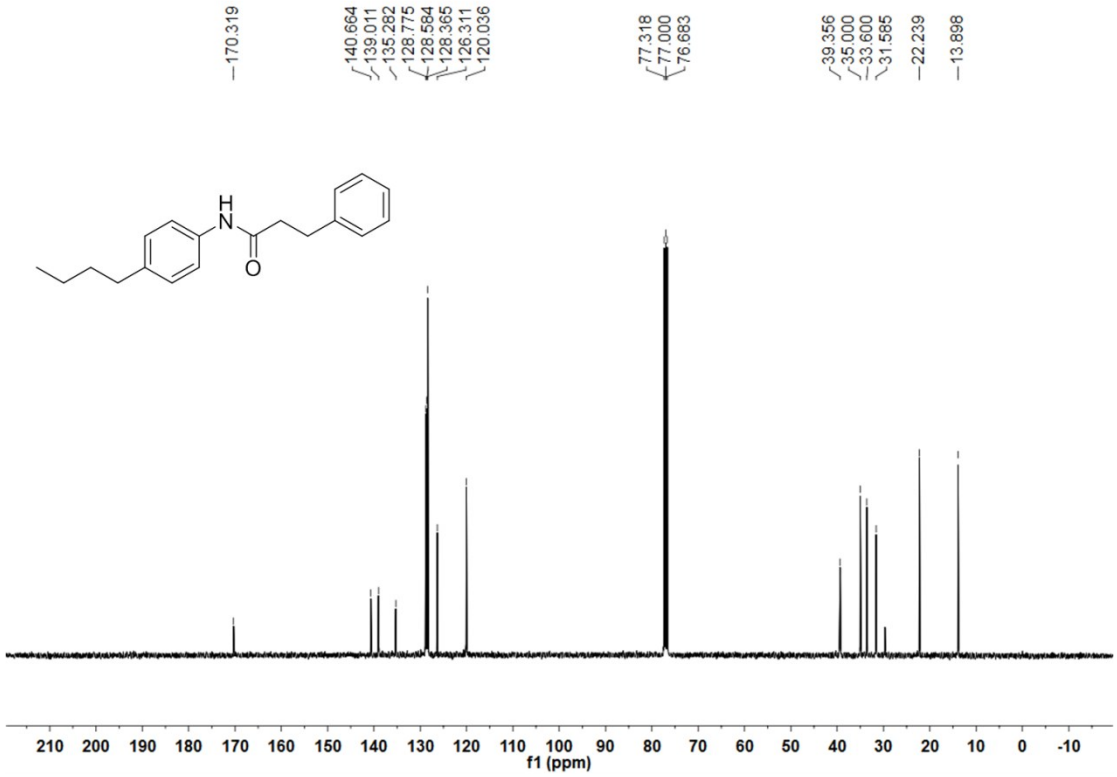
f1 (ppm)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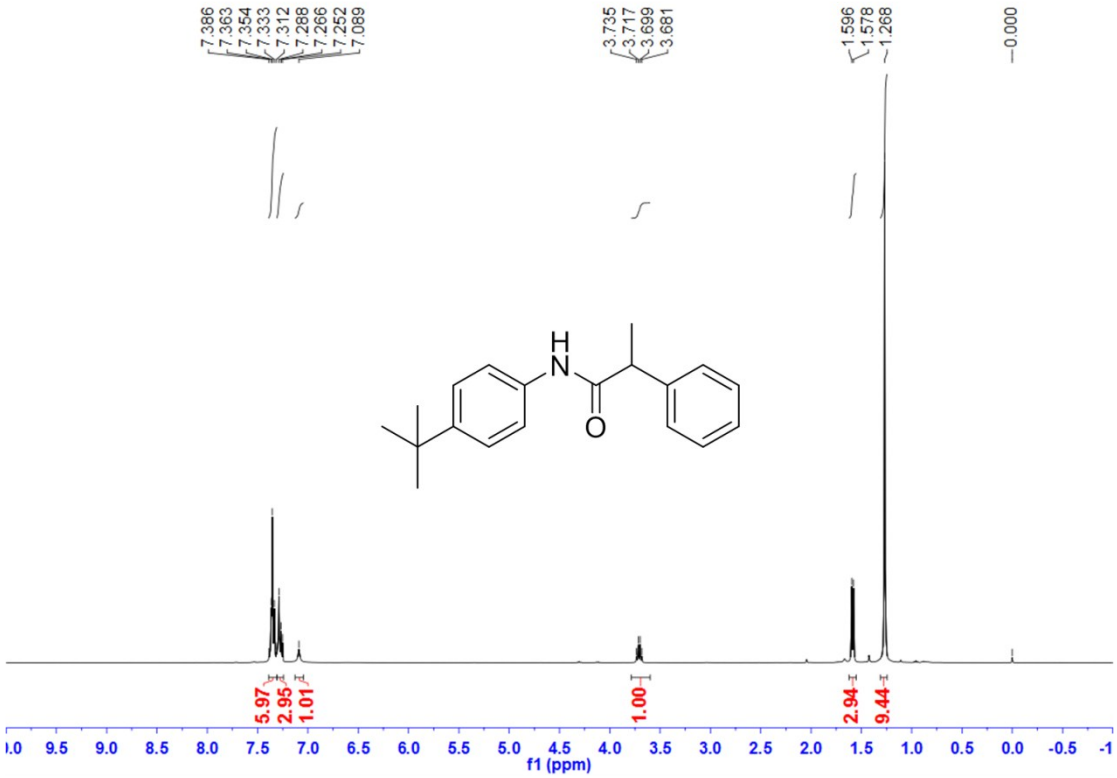
39.356
35.000
33.600
31.585
22.239
13.898
H
N
O
f1 (ppm)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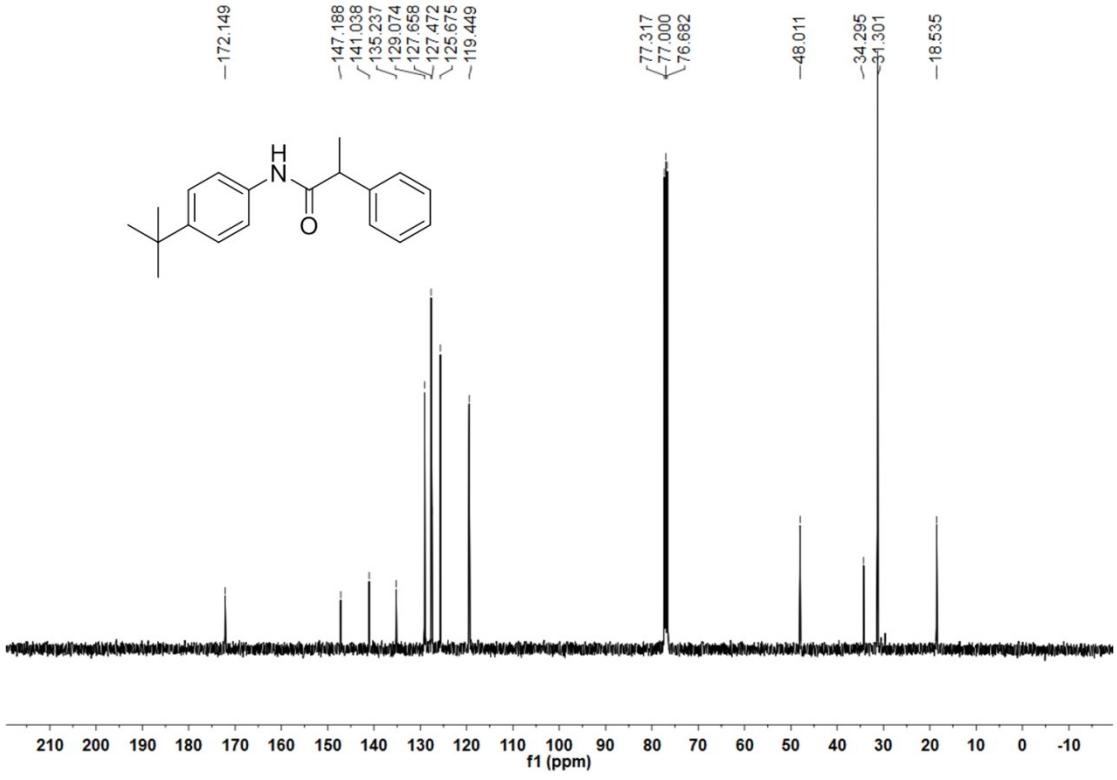
7.354
7.333
7.312
7.288
7.266
7.252
7.089
3.735
3.717
3.699
3.681
1.596
1.578
1.268
0.000
H
N
O
5.97
2.95
1.01
1.00
2.94
9.44
f1 (ppm)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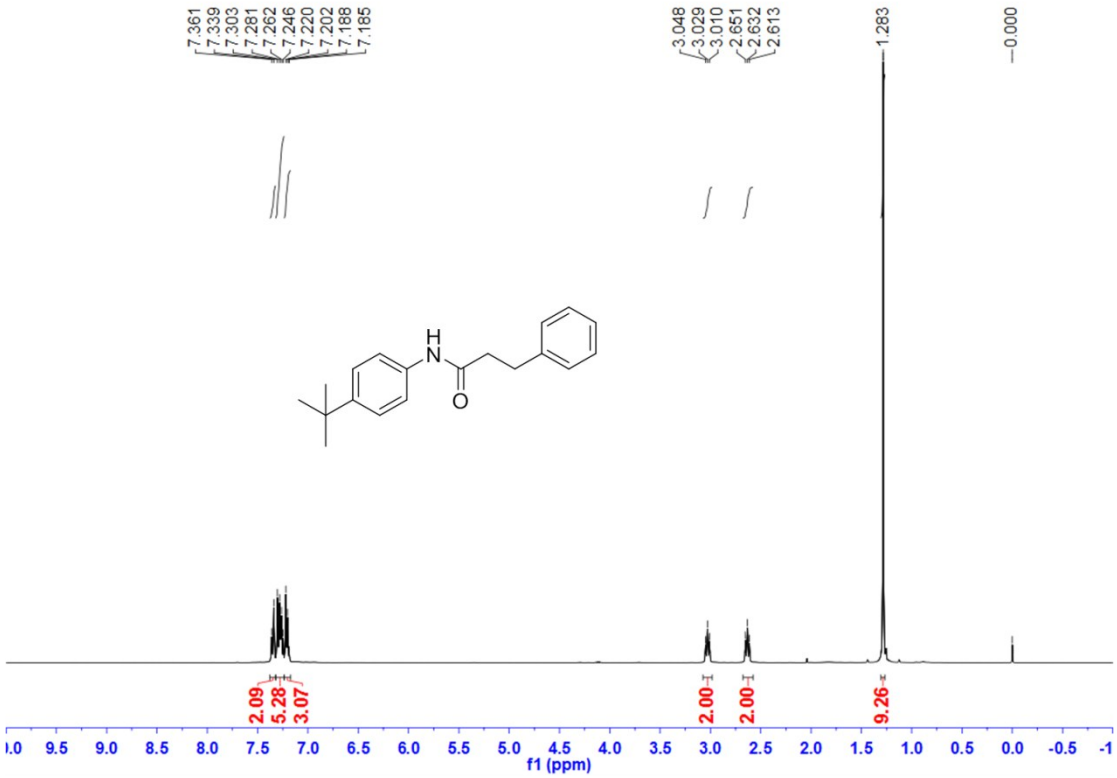
147.188
141.038
135.237
129.074
127.658
127.472
125.675
119.449
77.317
77.000
76.682
48.011
34.295
31.301
18.535
H
N
O
f1 (ppm)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7.246
7.220
7.202
7.188
7.185
3.048
3.029
3.010
2.651
2.632
2.613
1.283
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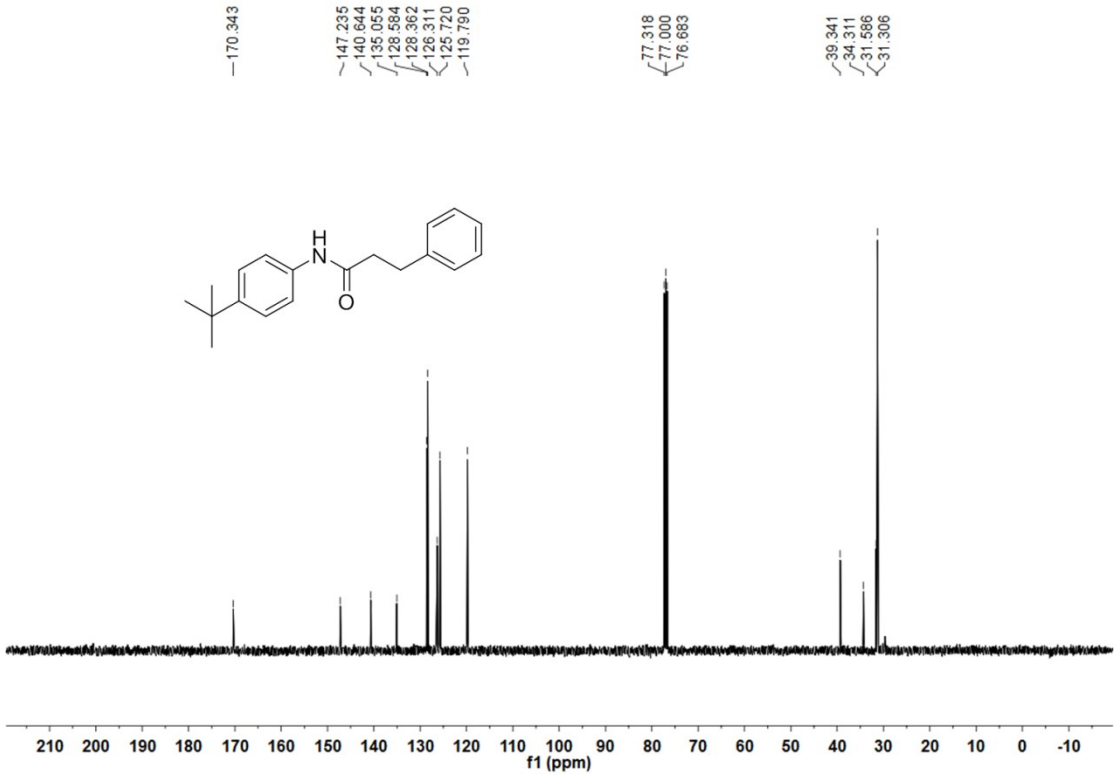
H
N
O
2.09
5.28
3.07
2.00
2.00
9.26
f1 (ppm)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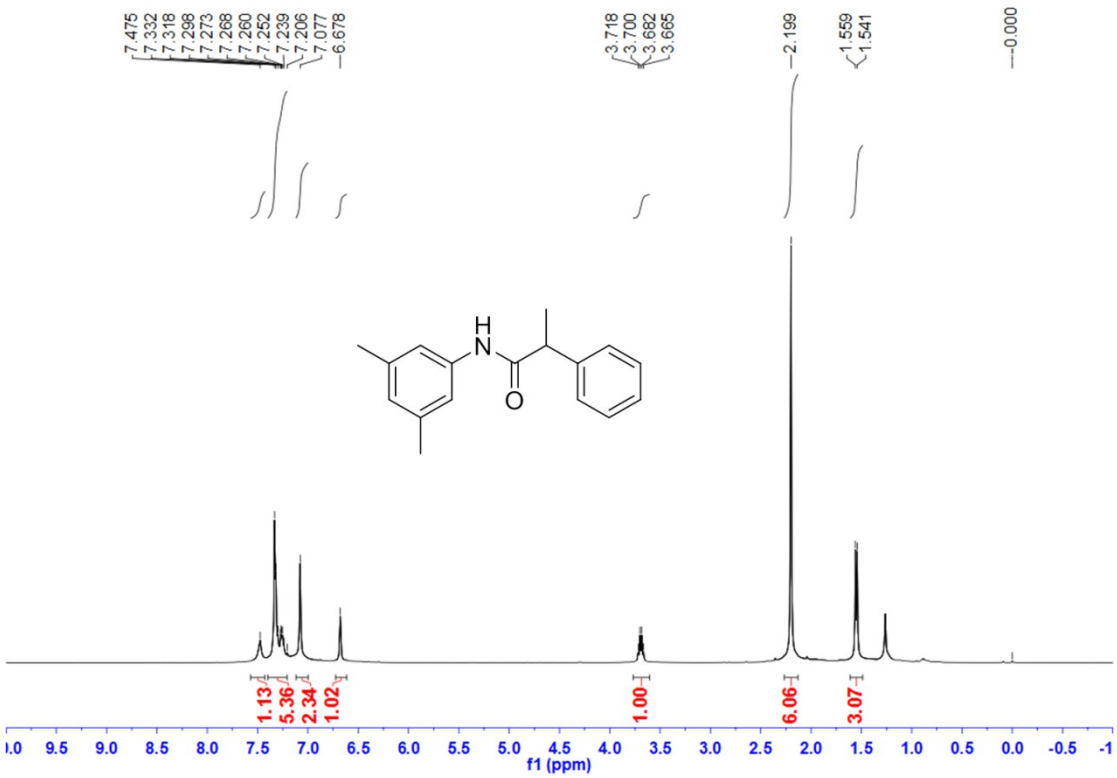
128.362
126.311
125.720
119.790
77.318
77.000
76.683
39.341
34.311
31.586
31.306
H
N
O
f1 (ppm)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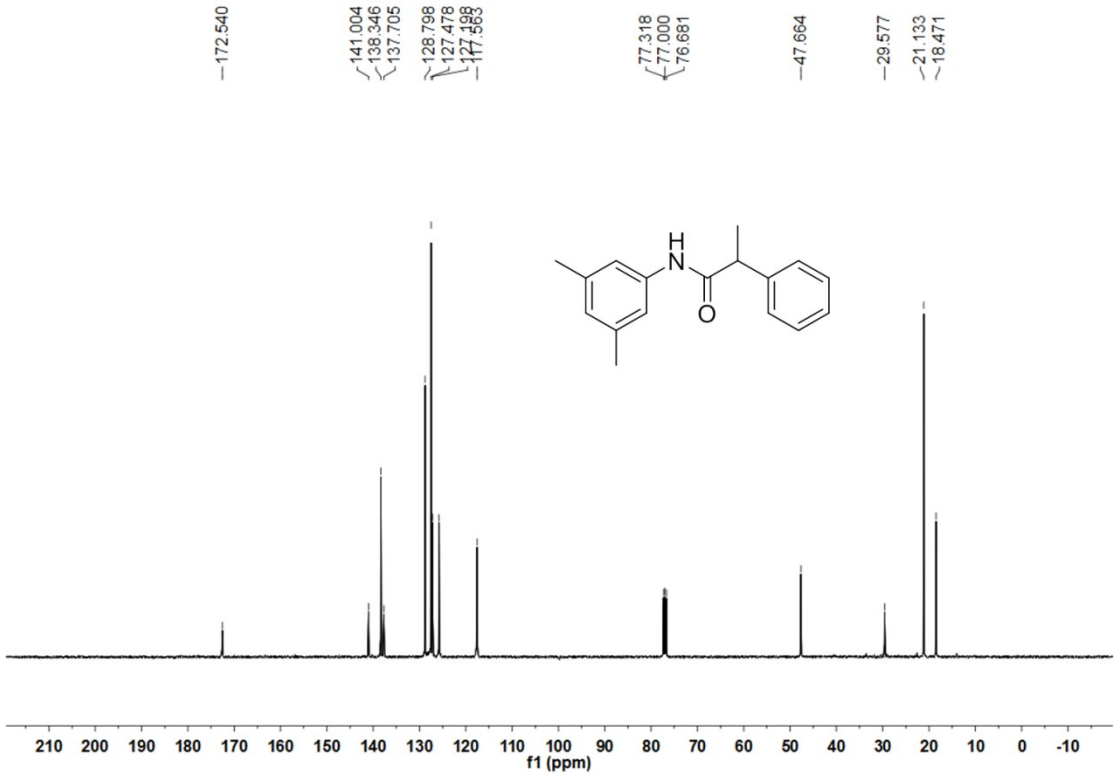
7.318
7.298
7.273
7.268
7.260
7.252
7.239
7.206
7.077
6.678
3.718
3.700
3.682
3.665
2.199
1.559
1.541
0.000
1.13
5.36
2.34
1.02
1.00
6.06
3.07
f1 (ppm)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141.004
138.346
137.705
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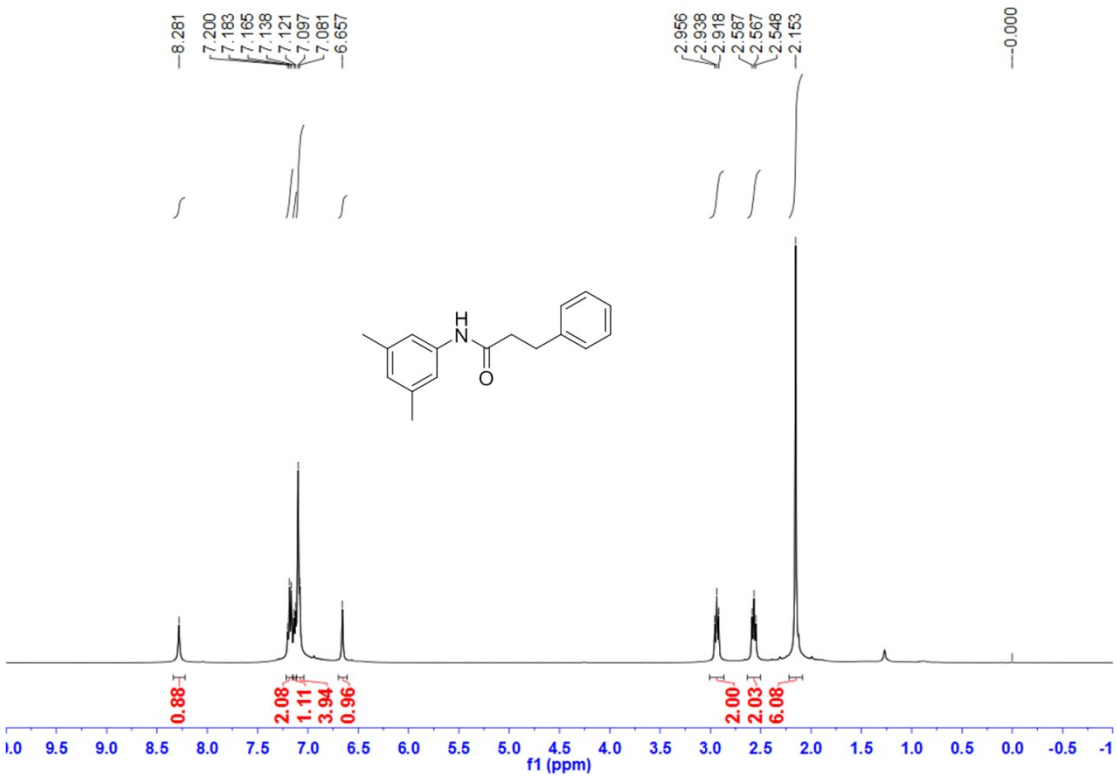
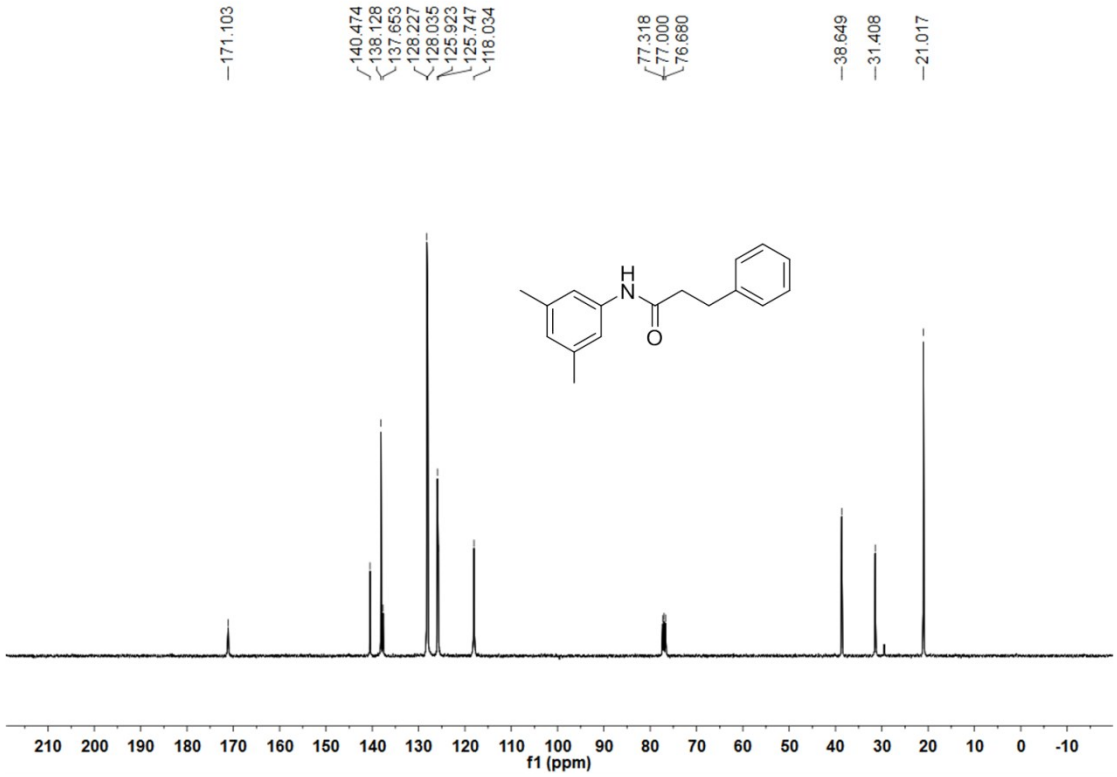
127.478
77.318
77.000
76.681
47.664
29.577
21.133
18.471
f1 (ppm)


8.281
7.200
7.183
7.165
7.138
7.121
7.097
7.081
6.657
2.956
2.938
2.918
2.587
2.567
2.548
2.153
0.000
0.88
2.08
1.11
3.94
0.96
2.00
2.03
6.08
f1 (ppm)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140.474
138.128
137.653
128.227
128.035
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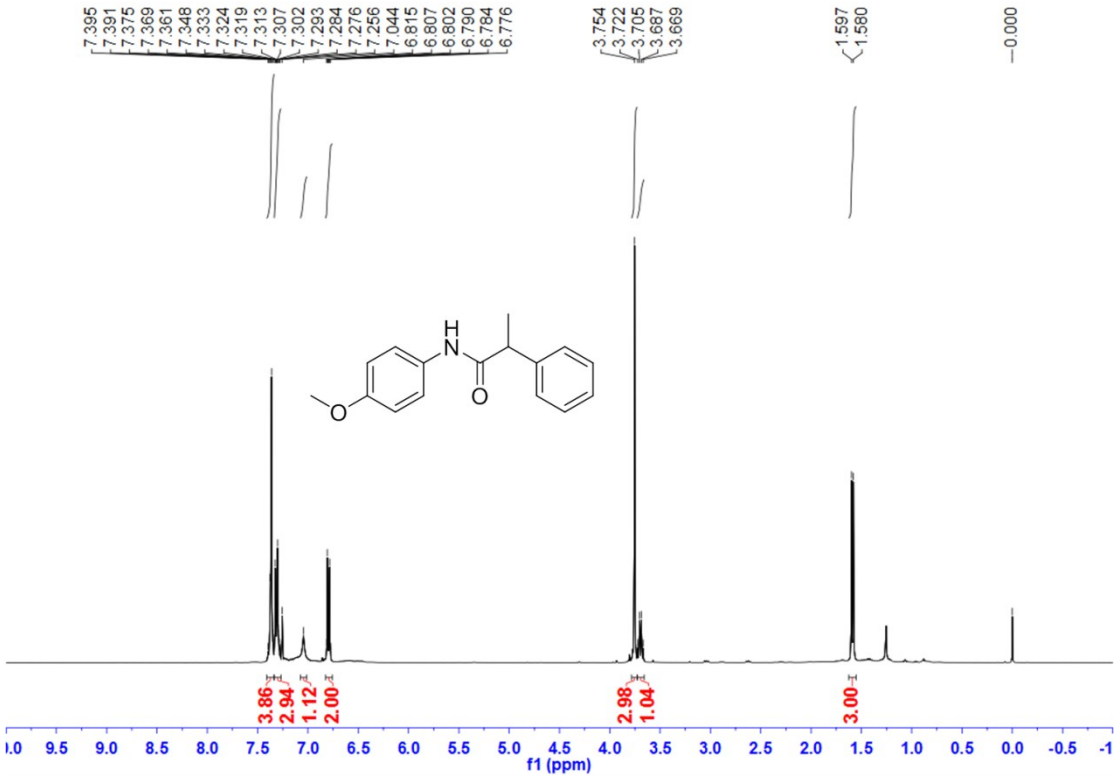
125.747
118.034
77.318
77.000
76.680
38.649
31.408
21.017
f1 (ppm)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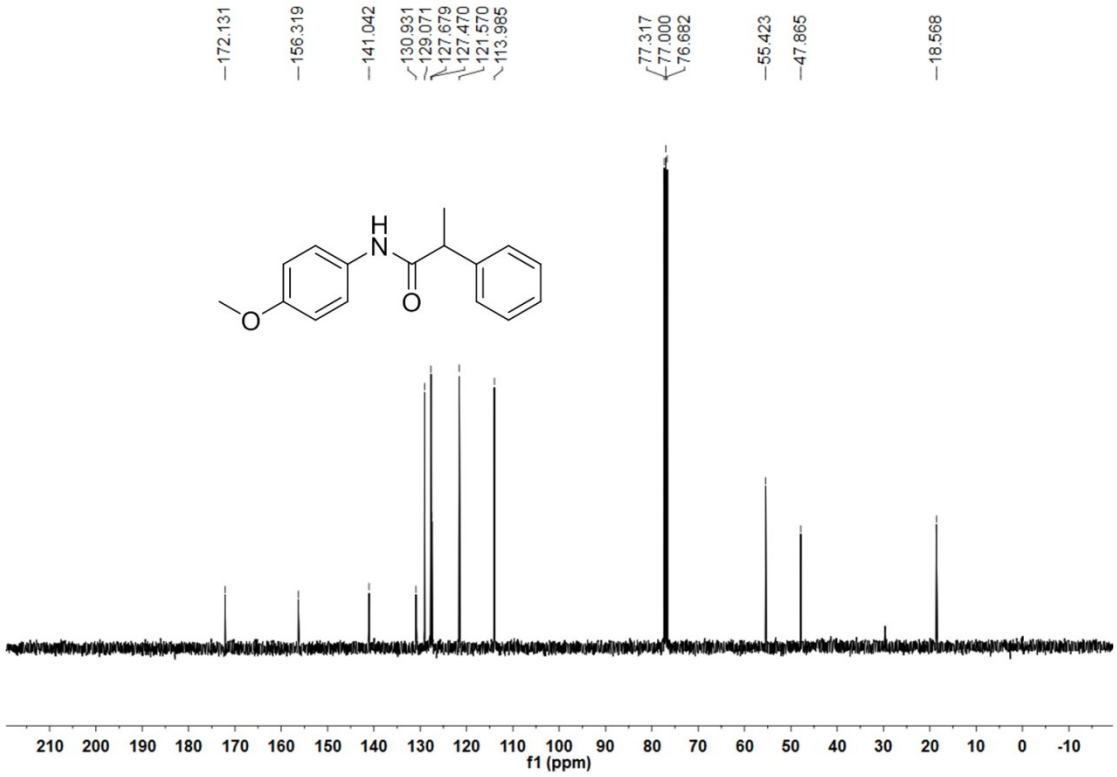
7.391
7.375
7.369
7.361
7.348
7.333
7.324
7.319
7.313
7.307
7.302
7.293
7.284
7.276
7.256
7.044
6.815
6.807
6.802
6.790
6.784
6.776
3.754
3.722
3.705
3.687
3.669
1.597
1.580
0.000
H
N
O
O
3.86
2.94
1.12
2.00
2.98
1.04
3.00
f1 (ppm)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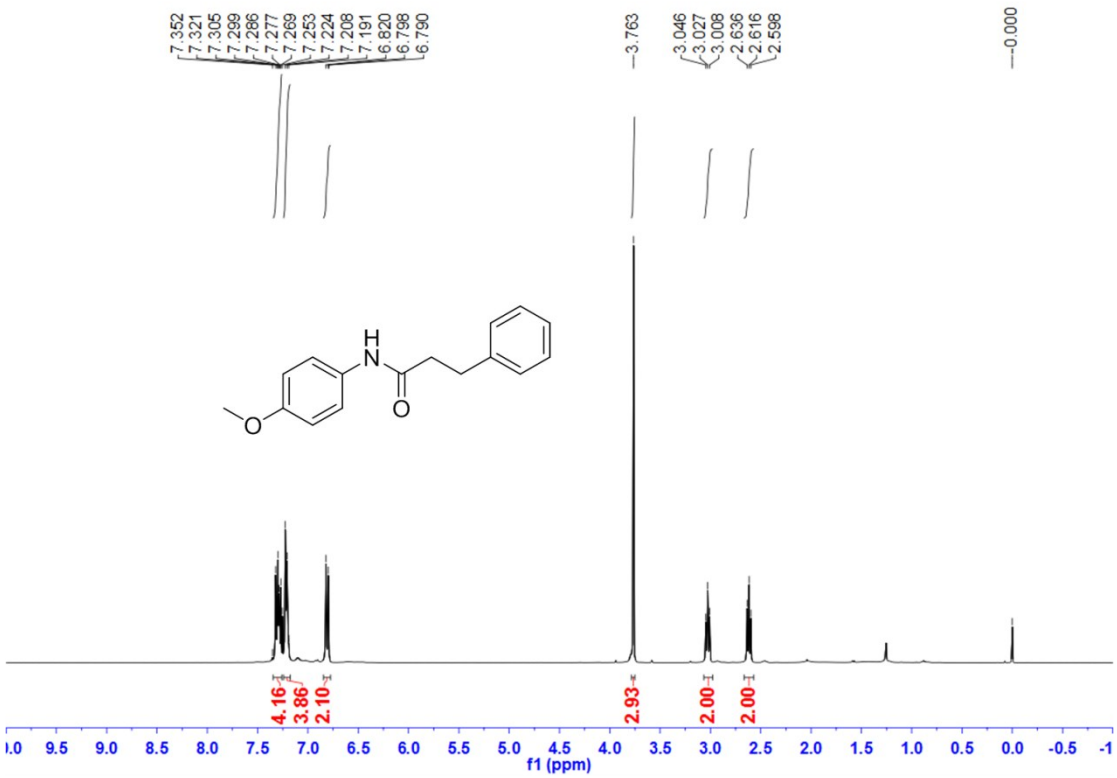
156.319
141.042
130.931
129.071
127.679
127.470
121.570
113.985
77.317
77.000
76.682
55.423
47.865
18.568
H
N
O
O
f1 (ppm)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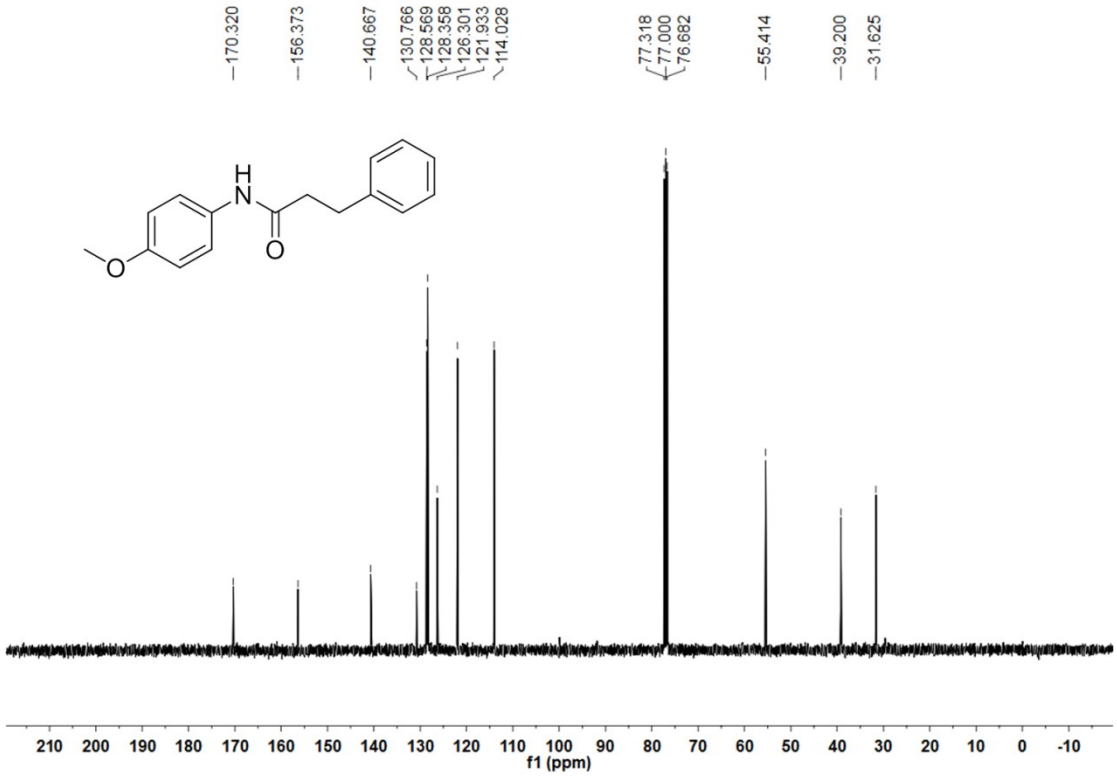
7.321
7.305
7.299
7.286
7.277
7.269
7.253
7.224
7.208
7.191
6.820
6.798
6.790
3.763
3.046
3.027
3.008
2.636
2.616
2.598
0.000
H
N
O
O
4.16
3.86
2.10
2.93
2.00
2.00
f1 (ppm)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140.667
130.766
128.569
128.358
126.301
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77.318
77.000
76.682
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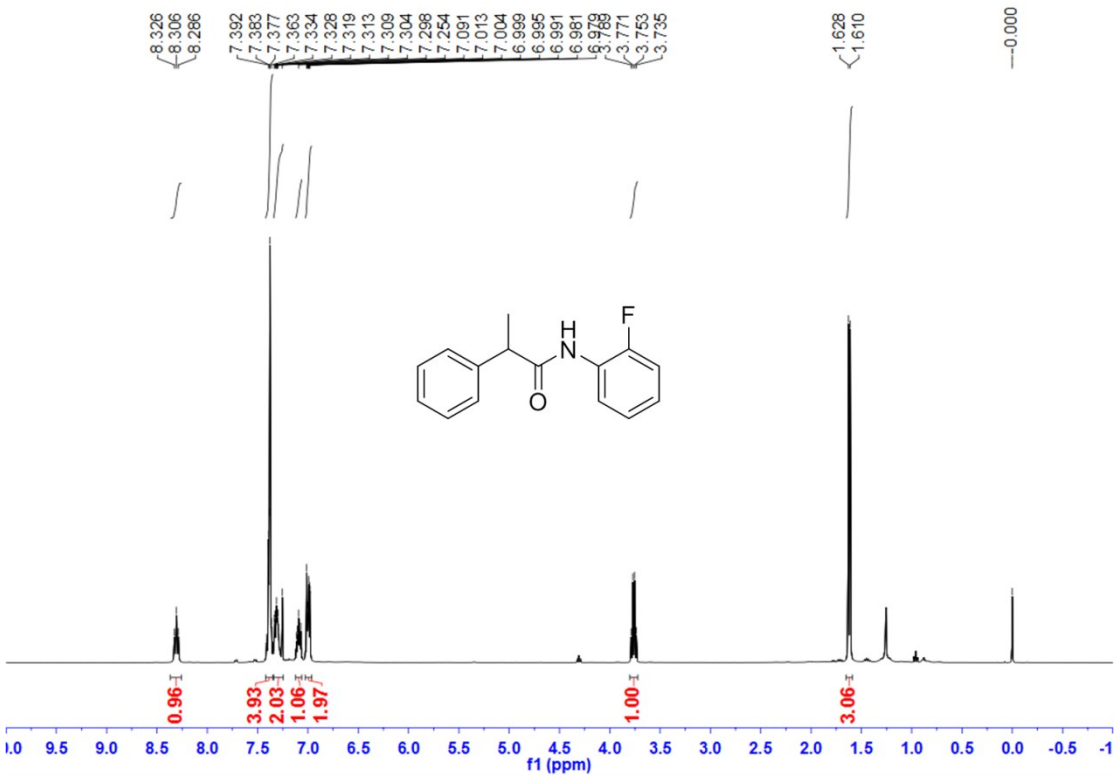
39.200
31.625
H
N
O
O
f1 (ppm)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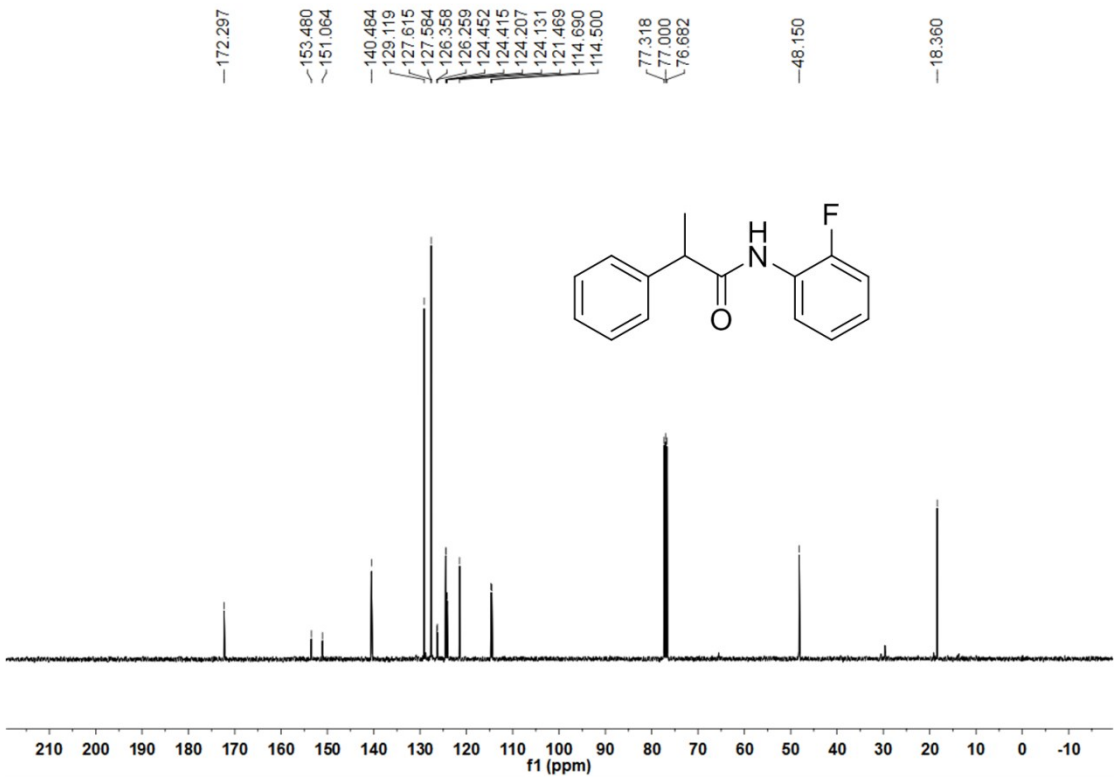
8.306
8.286
7.392
7.383
7.377
7.363
7.334
7.328
7.319
7.313
7.309
7.304
7.298
7.254
7.091
7.013
7.004
6.999
6.995
6.991
6.981
6.979
6.789
3.771
3.753
3.735
1.628
1.610
0.000
H
N
O
F
0.96
3.93
2.03
1.06
1.97
1.00
3.06
f1 (ppm)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151.064
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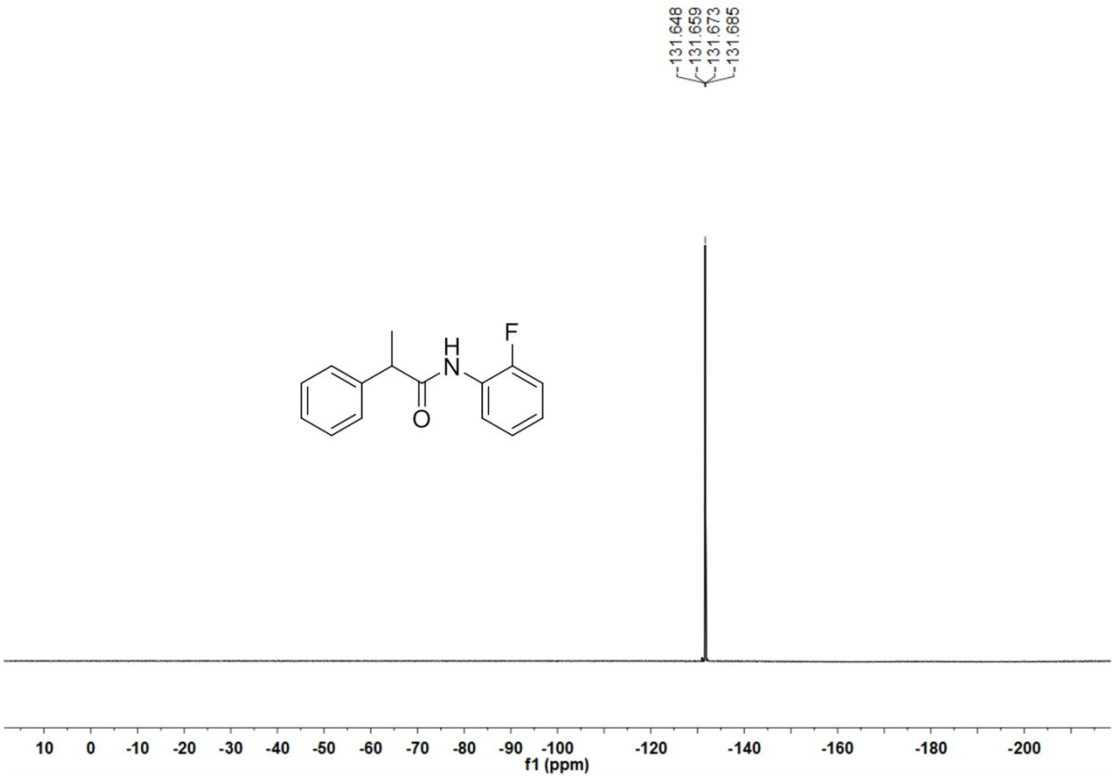
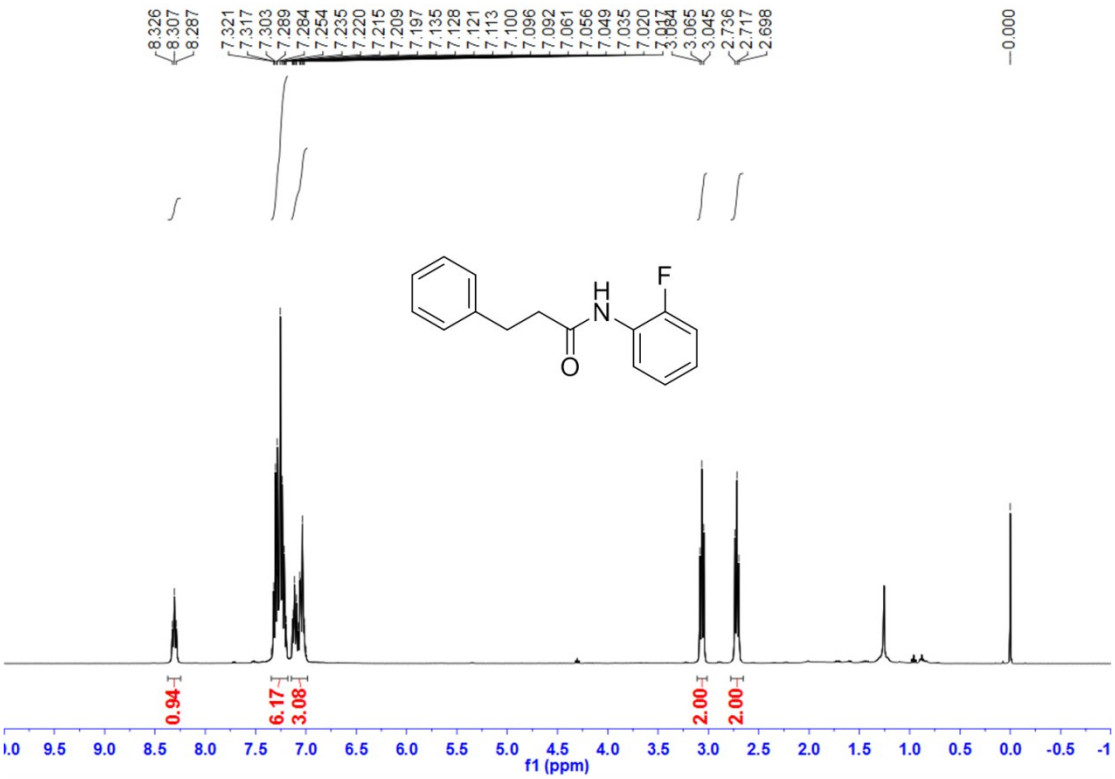
127.615
127.584
126.358
126.259
124.452
124.415
124.207
124.131
121.469
114.690
114.500
77.318
77.000
76.682
48.150
18.360
H
N
O
F
f1 (ppm)


131.648
131.659
131.673
131.685
F
H
N
O
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)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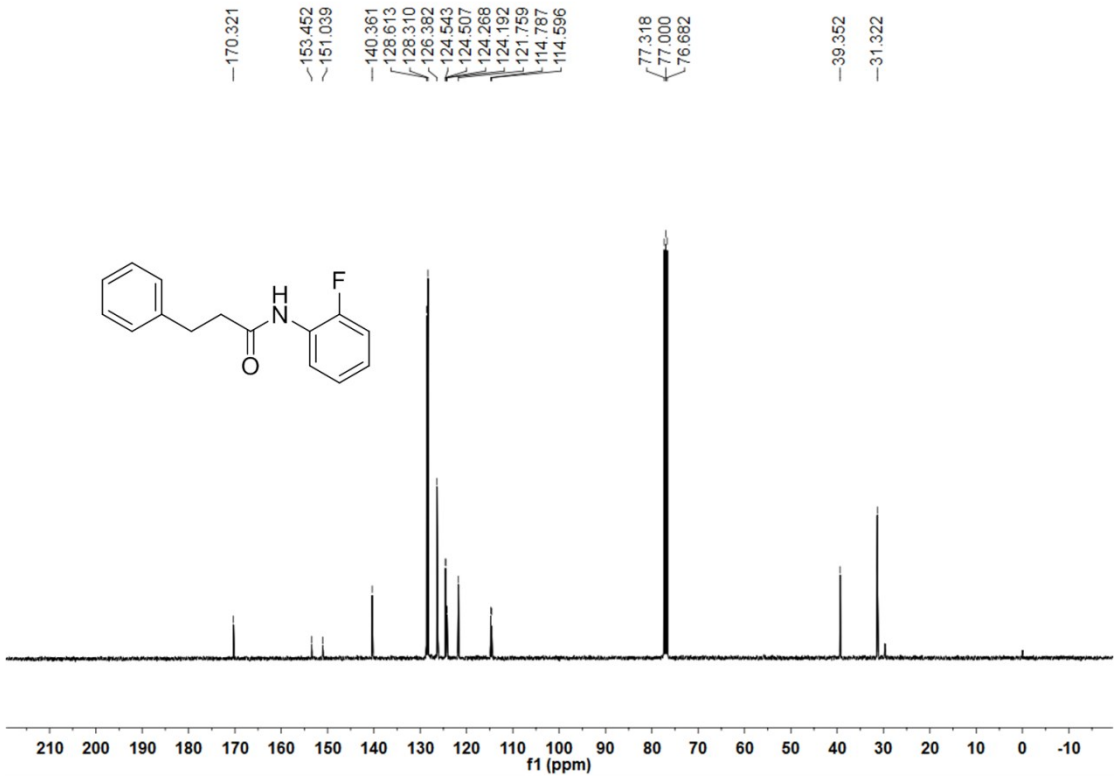
8.307
8.287
7.321
7.317
7.303
7.289
7.284
7.254
7.235
7.220
7.215
7.209
7.197
7.135
7.128
7.121
7.113
7.100
7.096
7.092
7.061
7.056
7.049
7.035
7.020
3.065
3.045
2.736
2.717
2.698
0.000
F
H
N
O
0.94
6.17
3.08
2.00
2.00
f1 (ppm)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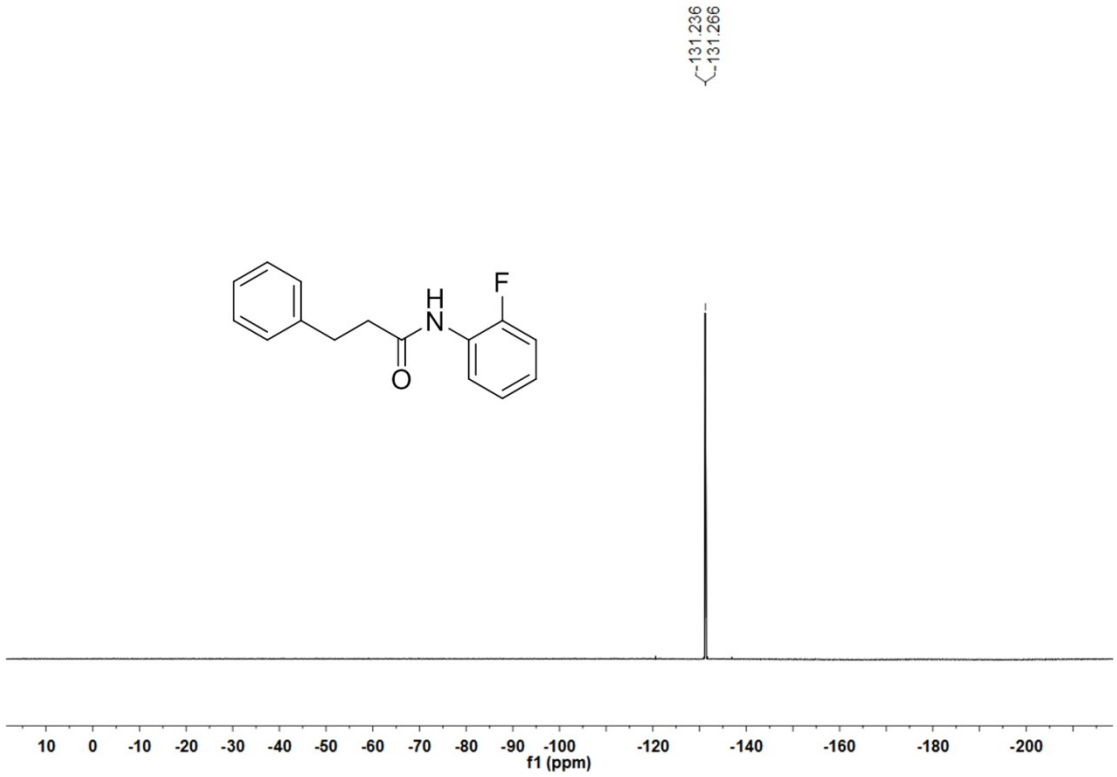
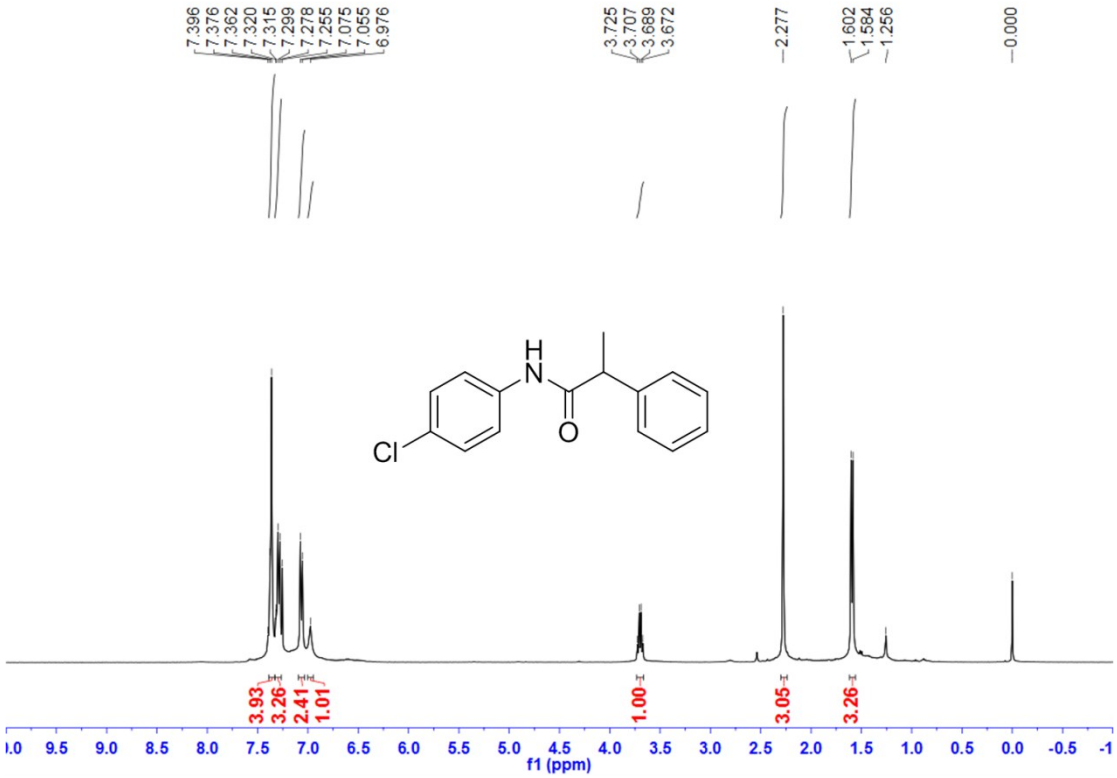
151.039
140.361
128.613
128.310
126.382
124.543
124.507
124.268
124.192
121.759
114.787
114.596
77.318
77.000
76.682
39.352
31.322
F
H
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


131.236
131.266
F
H
N
O
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)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7.299
7.278
7.255
7.075
7.055
6.976
3.725
3.707
3.689
3.672
2.277
1.602
1.584
1.256
0.000
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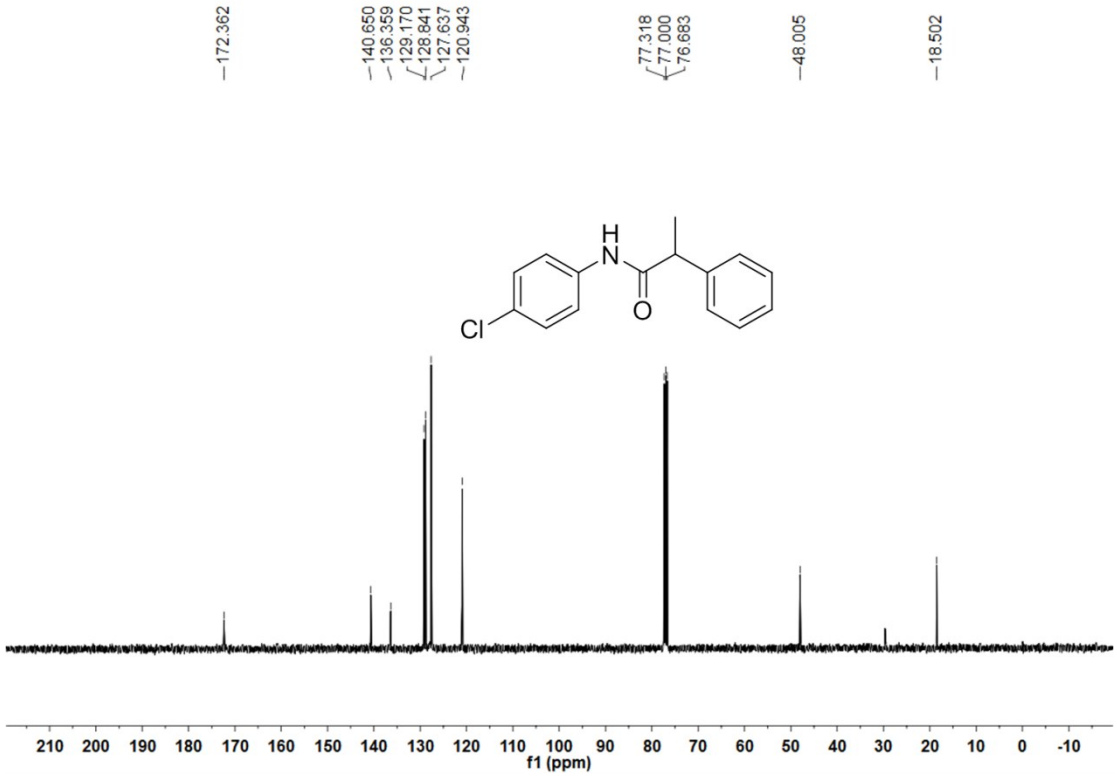
N
O
Cl
3.93
3.26
2.41
1.01
1.00
3.05
3.26
f1 (ppm)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136.359
129.170
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120.943
77.318
77.000
76.683
48.005
18.502
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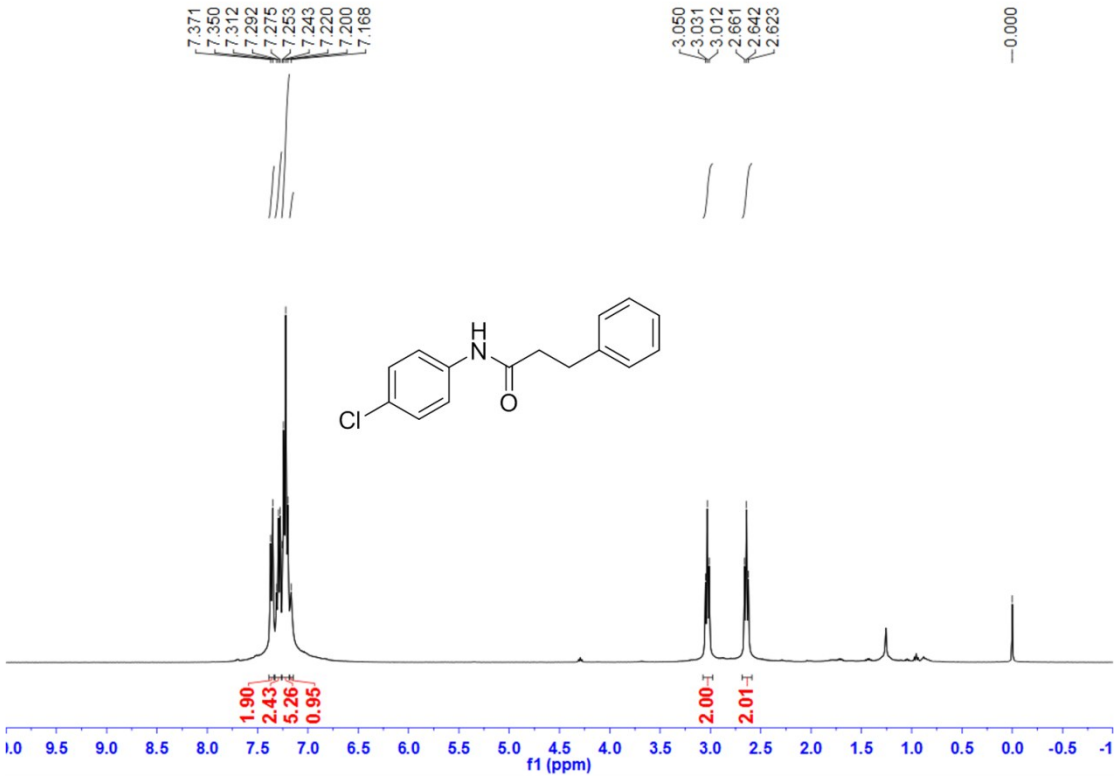
N
O
Cl
f1 (ppm)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7.220
7.200
7.168
3.050
3.031
3.012
2.661
2.642
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H
N
O
Cl
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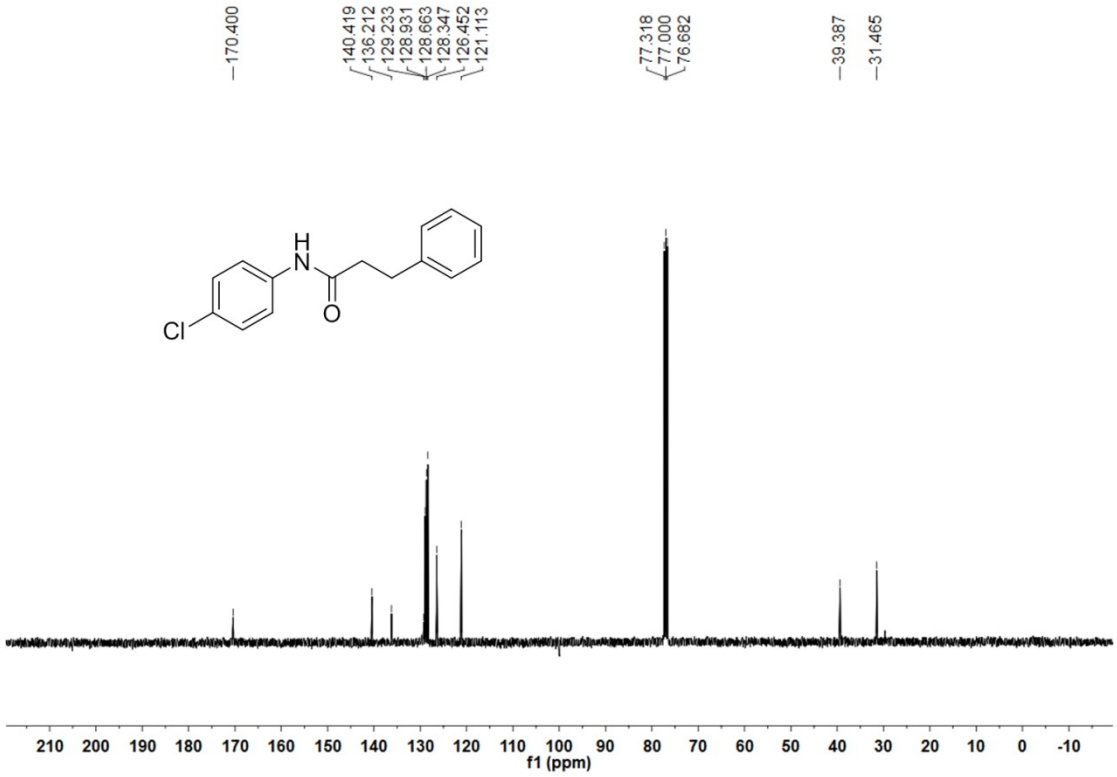
2.43
5.26
0.95
2.00
2.01
f1 (ppm)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136.212
129.233
128.931
128.663
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76.682
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31.465
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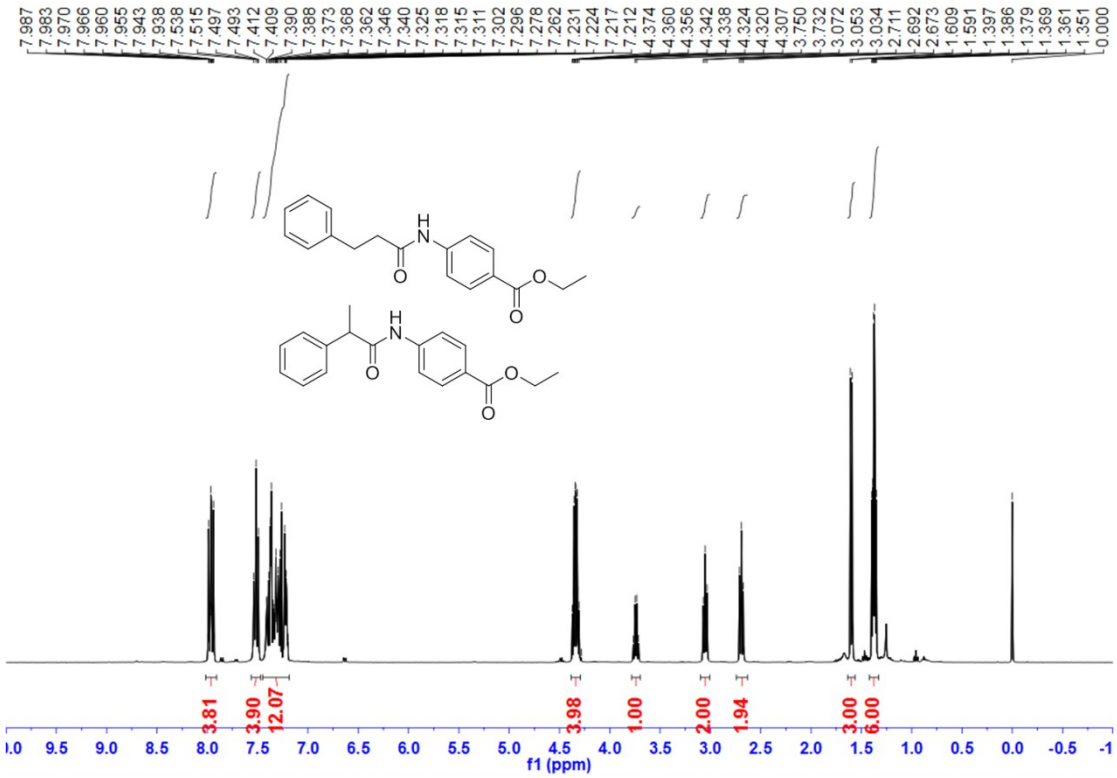
N
O
Cl
f1 (ppm)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7.938
7.538
7.515
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7.493
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7.409
7.390
7.388
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7.362
7.346
7.340
7.325
7.318
7.315
7.311
7.302
7.296
7.278
7.262
7.231
7.224
7.217
7.212
4.374
4.360
4.356
4.342
4.338
4.324
4.320
4.307
3.750
3.732
3.072
3.053
3.034
2.711
2.692
2.673
1.609
1.591
1.397
1.386
1.379
1.369
1.361
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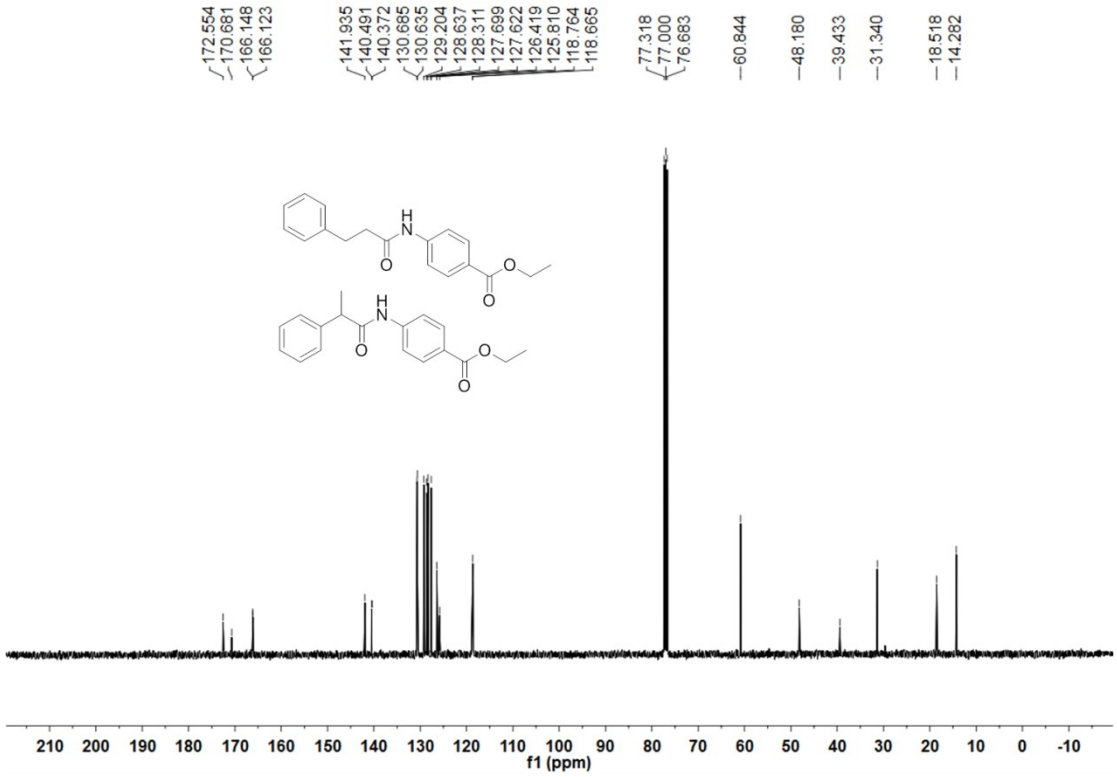
3.00
6.00
f1 (ppm)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130.685
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129.204
128.637
128.311
127.699
127.622
126.419
125.810
118.764
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77.000
76.683
60.844
48.180
39.433
31.340
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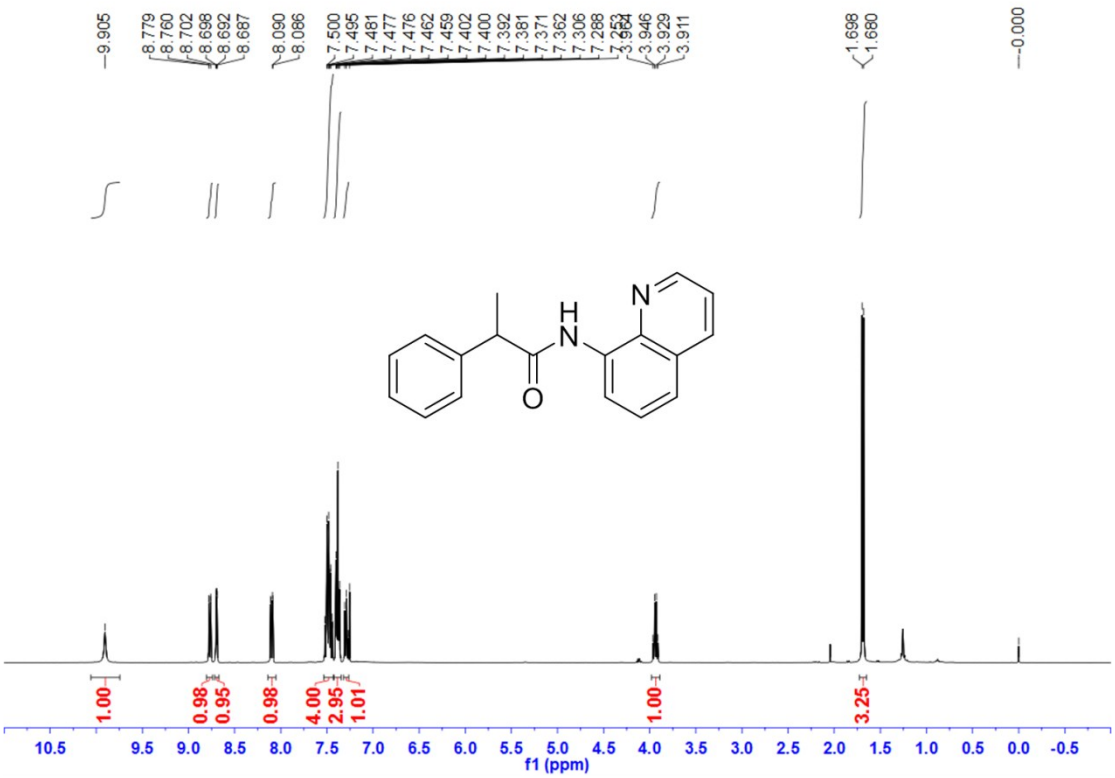
14.282
f1 (ppm)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8.760
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8.090
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7.481
7.477
7.476
7.462
7.459
7.402
7.400
7.392
7.381
7.371
7.362
7.306
7.288
3.946
3.929
3.911
1.698
1.680
0.000
H
N
O
N
1.00
0.98
0.95
0.98
4.00
2.95
1.01
1.00
3.25
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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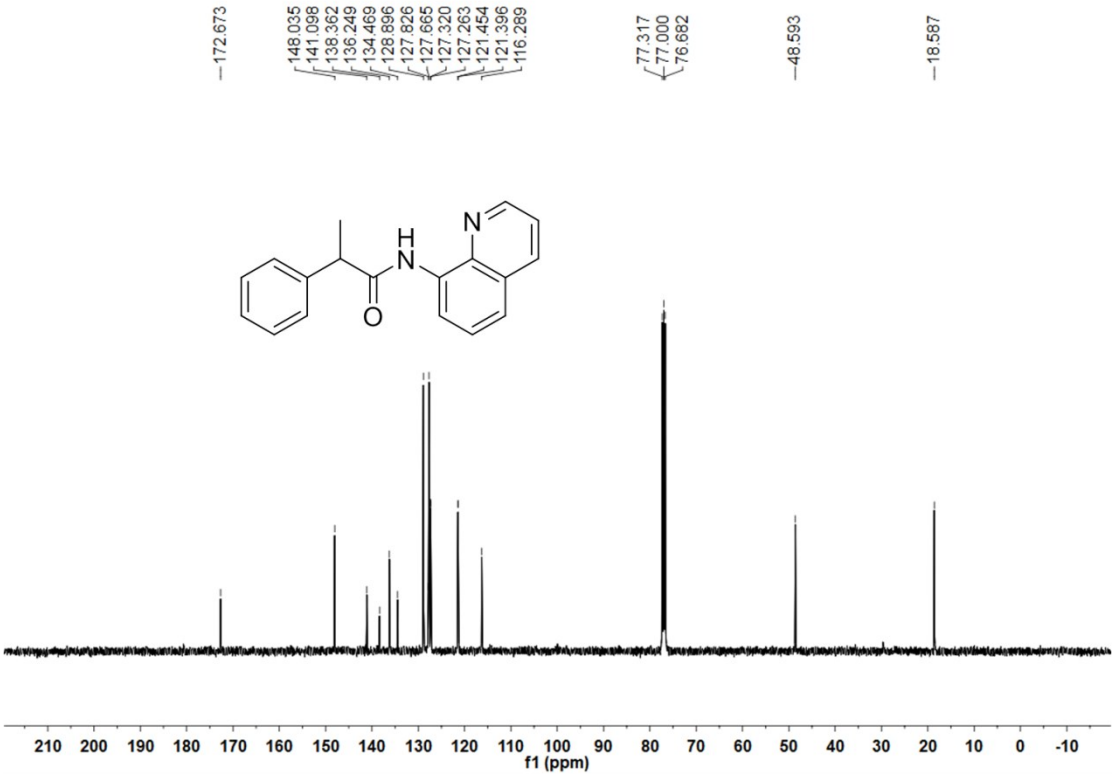
1.0
0.5
0.0
-0.5
f1 (ppm)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127.826
127.665
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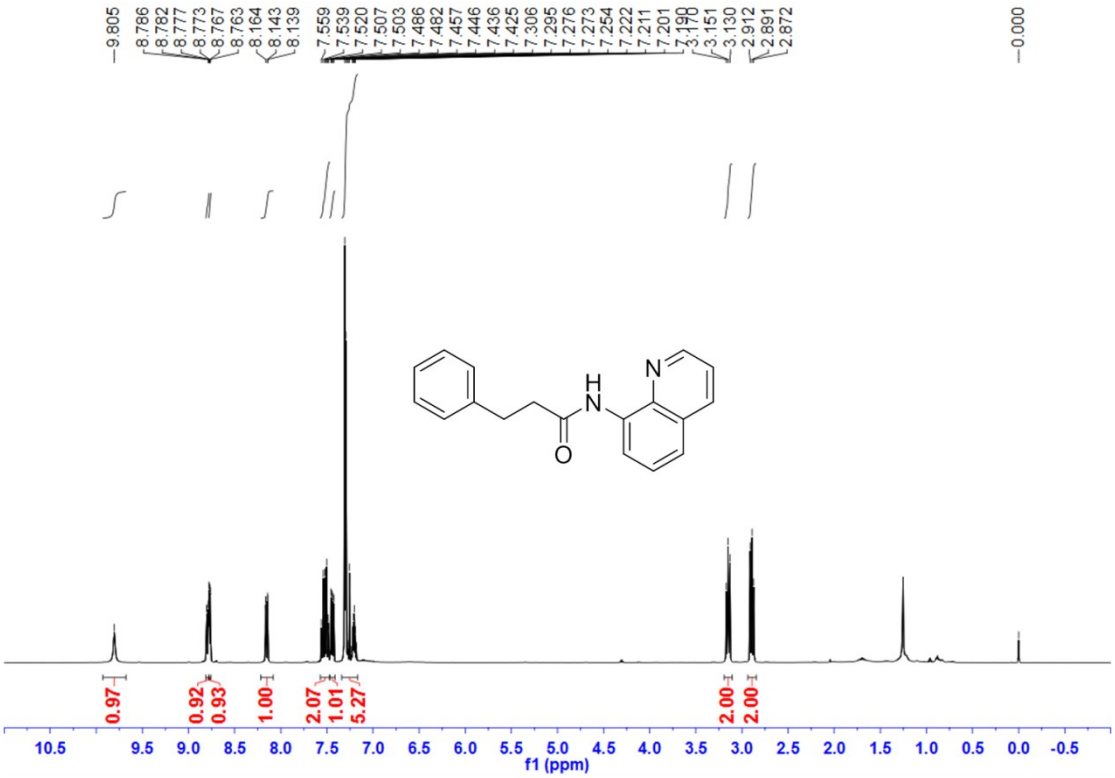
127.263
121.454
121.396
116.289
77.317
77.000
76.682
48.593
18.587
H
N
O
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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8.164
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7.520
7.507
7.503
7.486
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7.457
7.446
7.436
7.425
7.306
7.295
7.276
7.273
7.254
7.222
7.211
7.201
3.151
3.130
2.912
2.891
2.872
0.000
H
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O
N
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0.93
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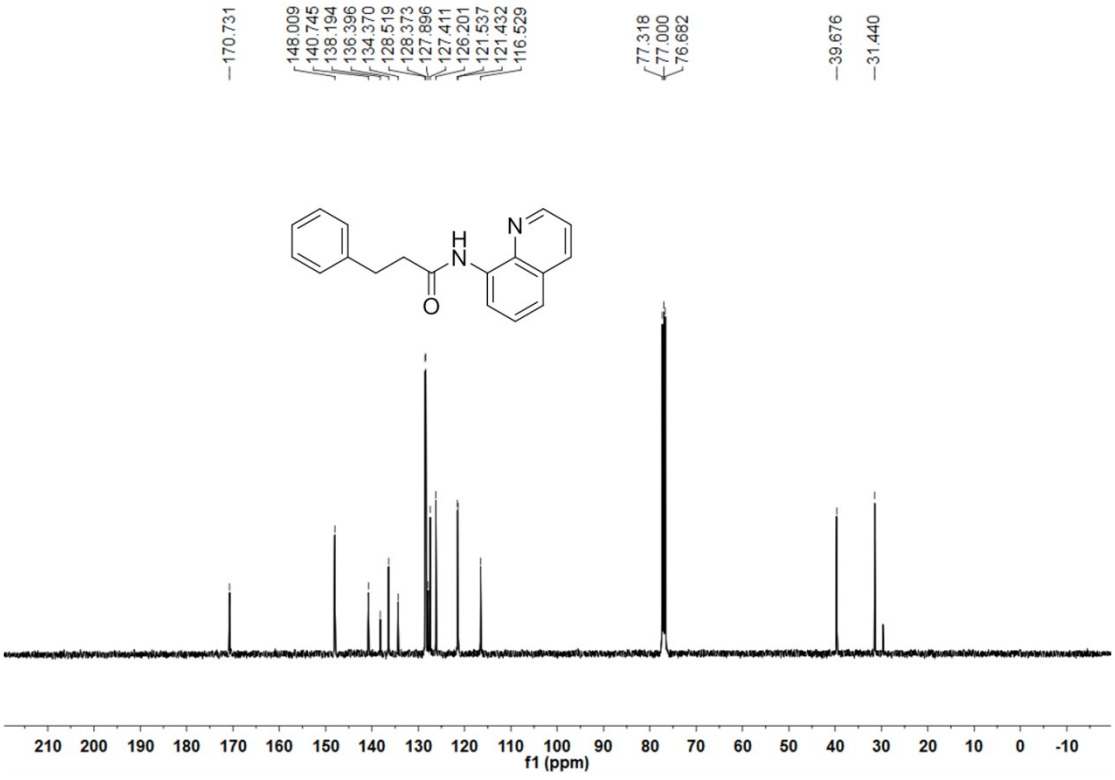
1.01
5.27
2.00
2.00
f1 (ppm)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128.373
127.896
127.411
126.201
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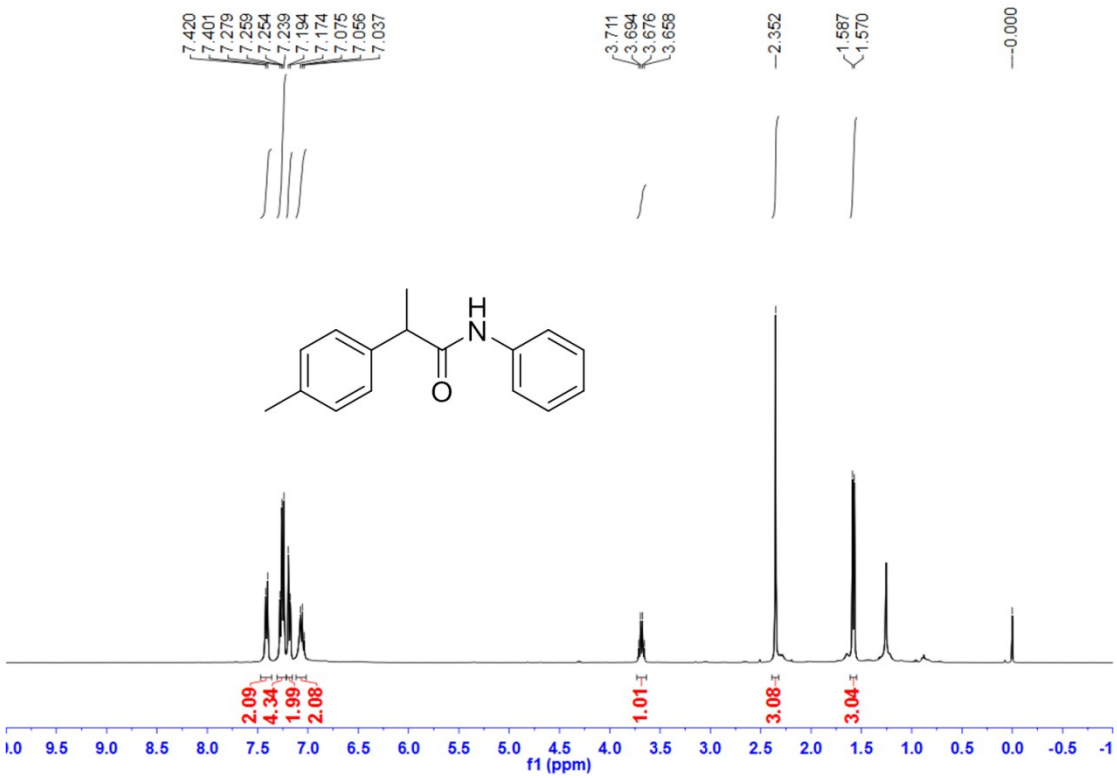
116.529
77.318
77.000
76.682
39.676
31.440
H
N
O
N
f1 (ppm)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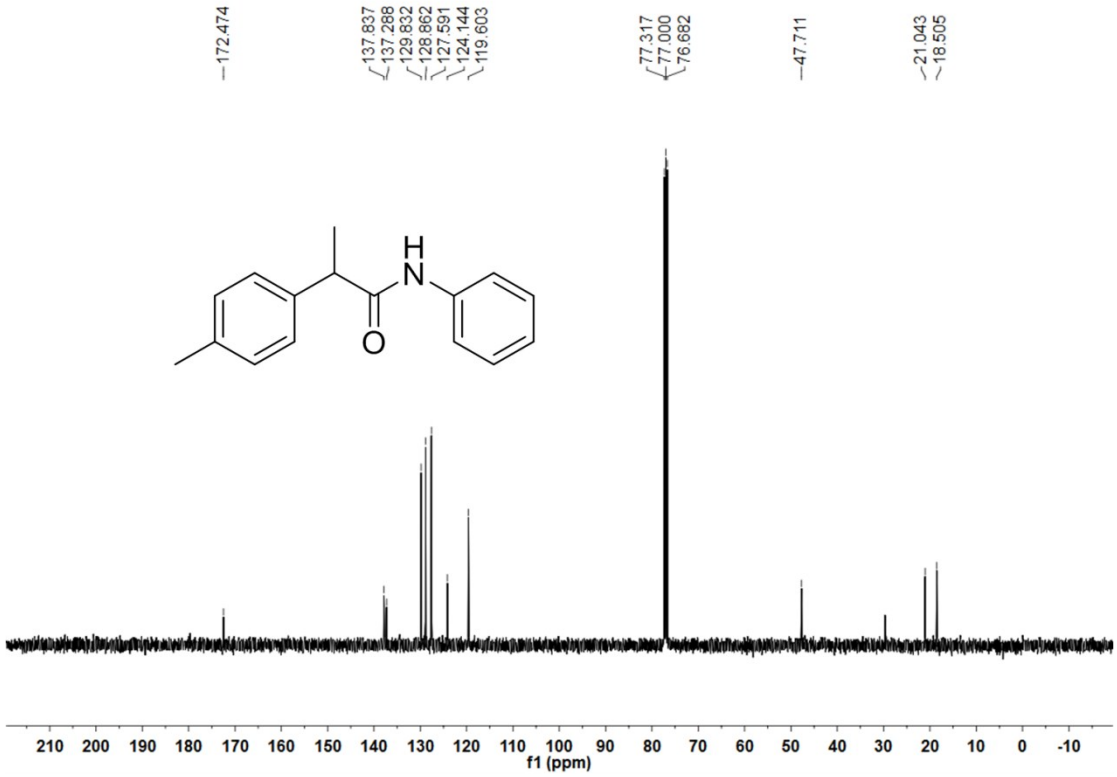
7.056
7.037
3.711
3.694
3.676
3.658
2.352
1.587
1.570
0.000
H
N
O
2.09
4.34
1.99
2.08
1.01
3.08
3.04
f1 (ppm)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129.832
128.862
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124.144
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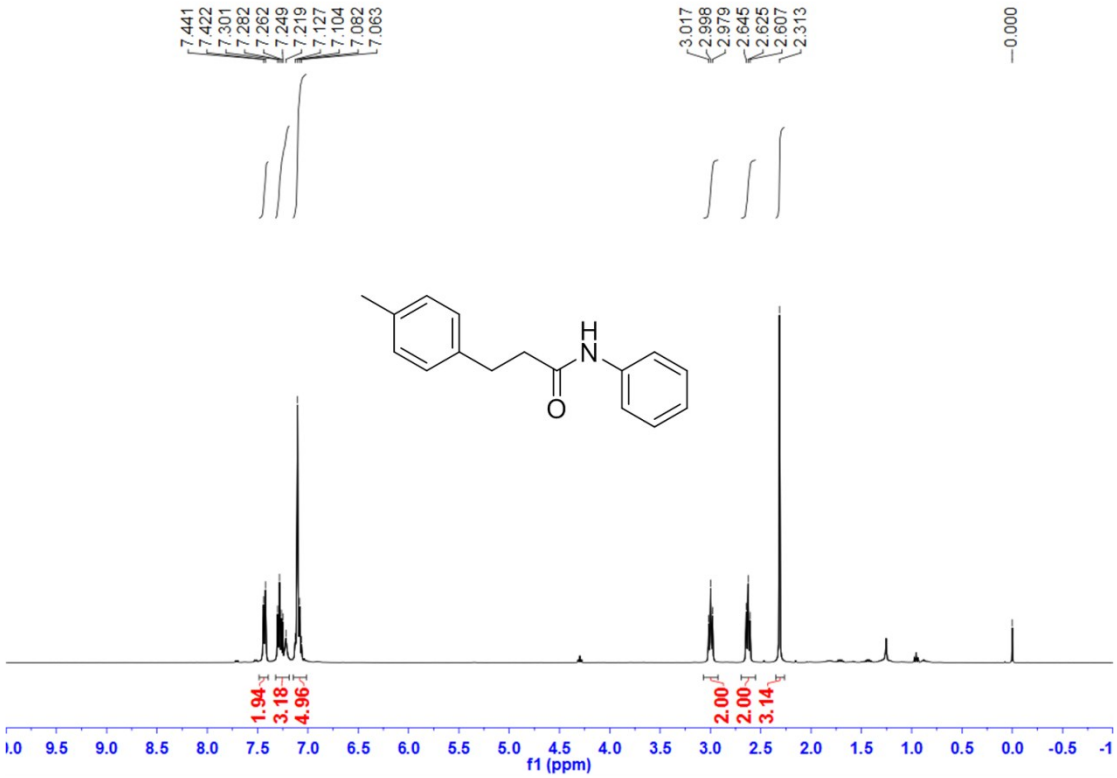
77.000
76.682
47.711
21.043
18.505
H
N
O
f1 (ppm)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7.301
7.282
7.262
7.249
7.219
7.127
7.104
7.082
7.063
3.017
2.998
2.979
2.645
2.625
2.607
2.313
0.000
H
N
O
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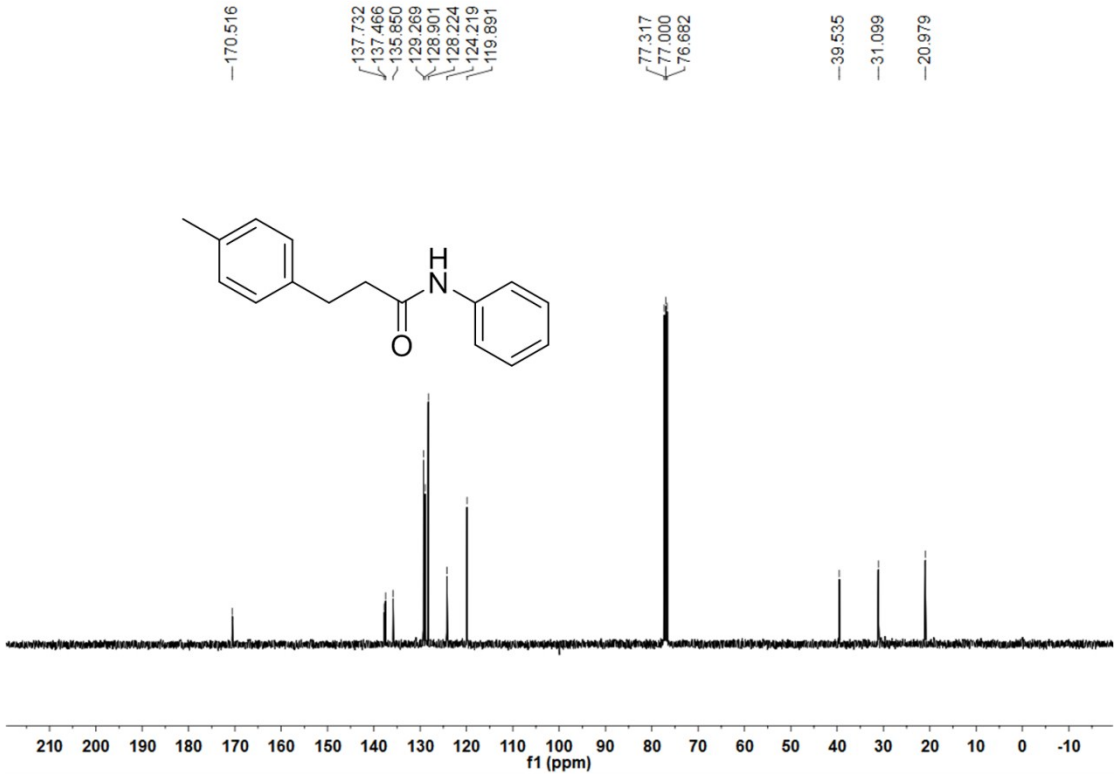
3.18
4.96
2.00
2.00
3.14
f1 (ppm)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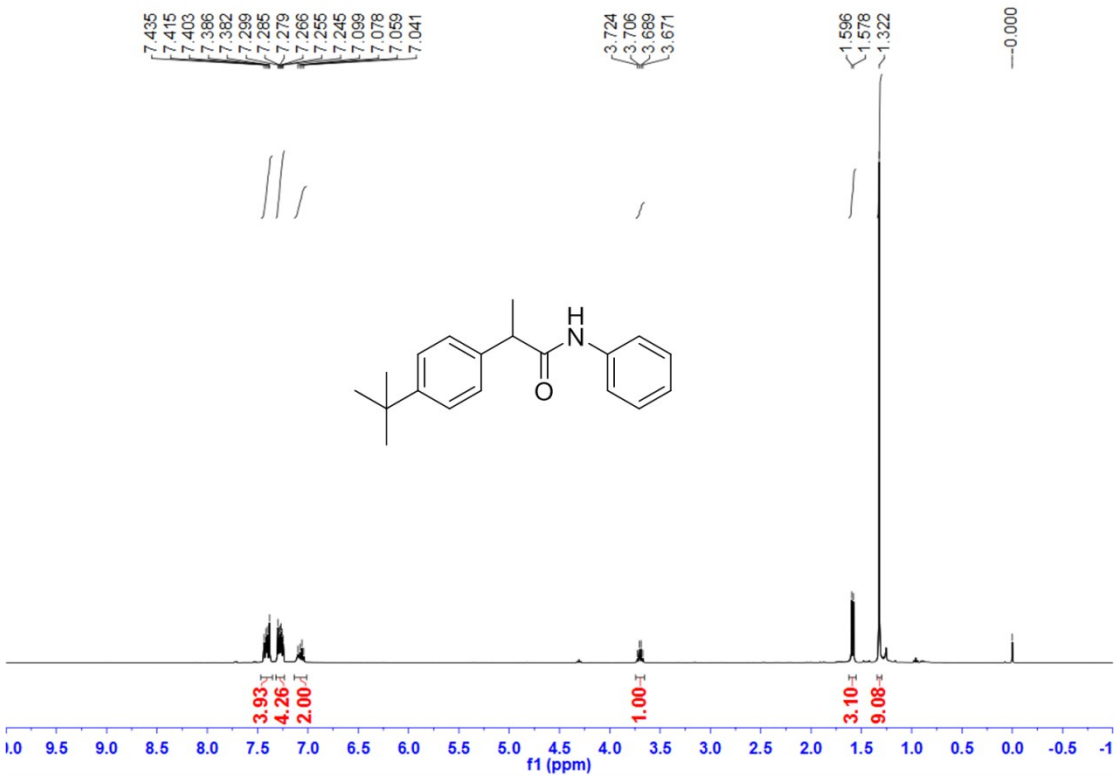
128.901
128.224
124.219
119.891
77.317
77.000
76.682
39.535
31.099
20.979
H
N
O
f1 (ppm)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7.299
7.285
7.279
7.266
7.255
7.245
7.099
7.078
7.059
7.041
3.724
3.706
3.689
3.671
1.596
1.578
1.322
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N
O
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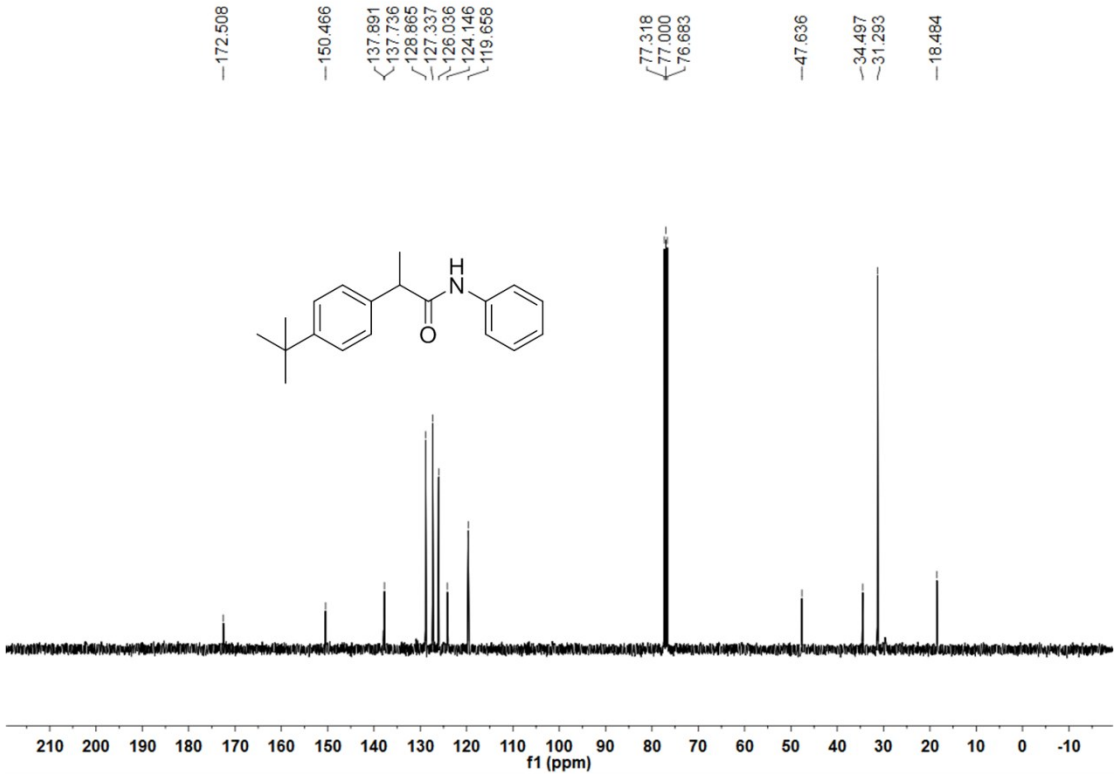
4.26
2.00
1.00
3.10
9.08
f1 (ppm)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124.146
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77.000
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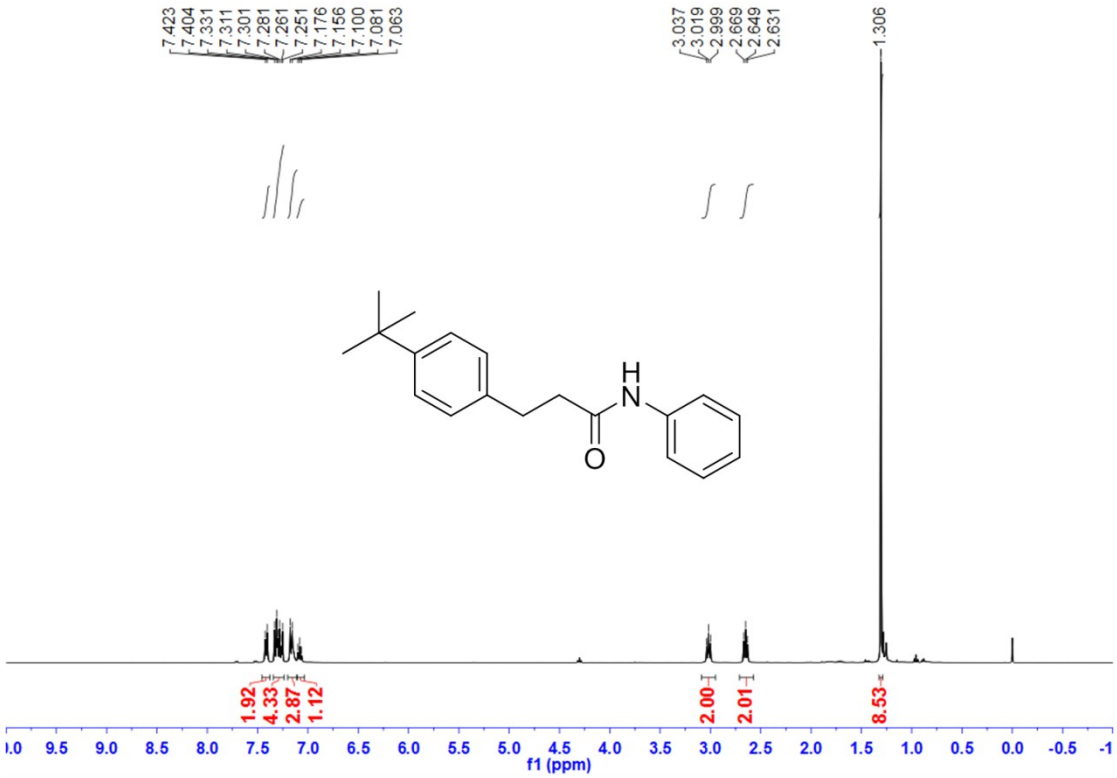
34.497
31.293
18.484
H
N
O
f1 (ppm)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7.281
7.261
7.251
7.176
7.156
7.100
7.081
7.063
3.037
3.019
2.999
2.669
2.649
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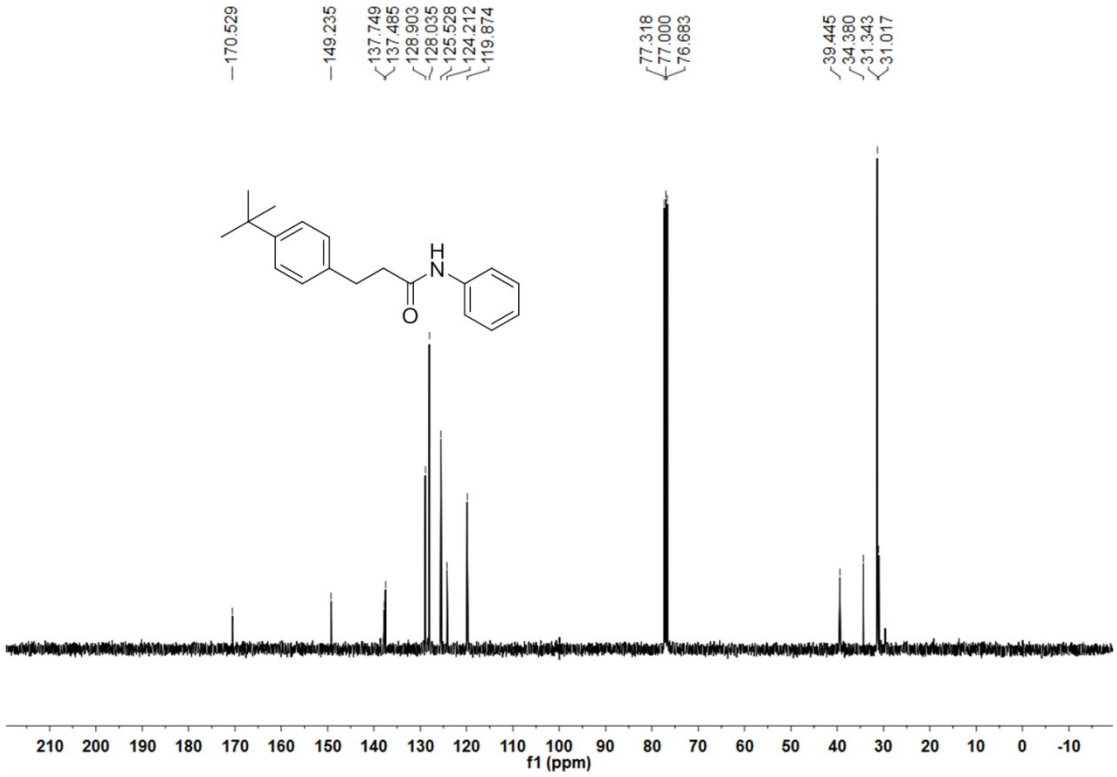
1.306
H
N
O
1.92
4.33
2.87
1.12
2.00
2.01
8.53
f1 (ppm)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31.017
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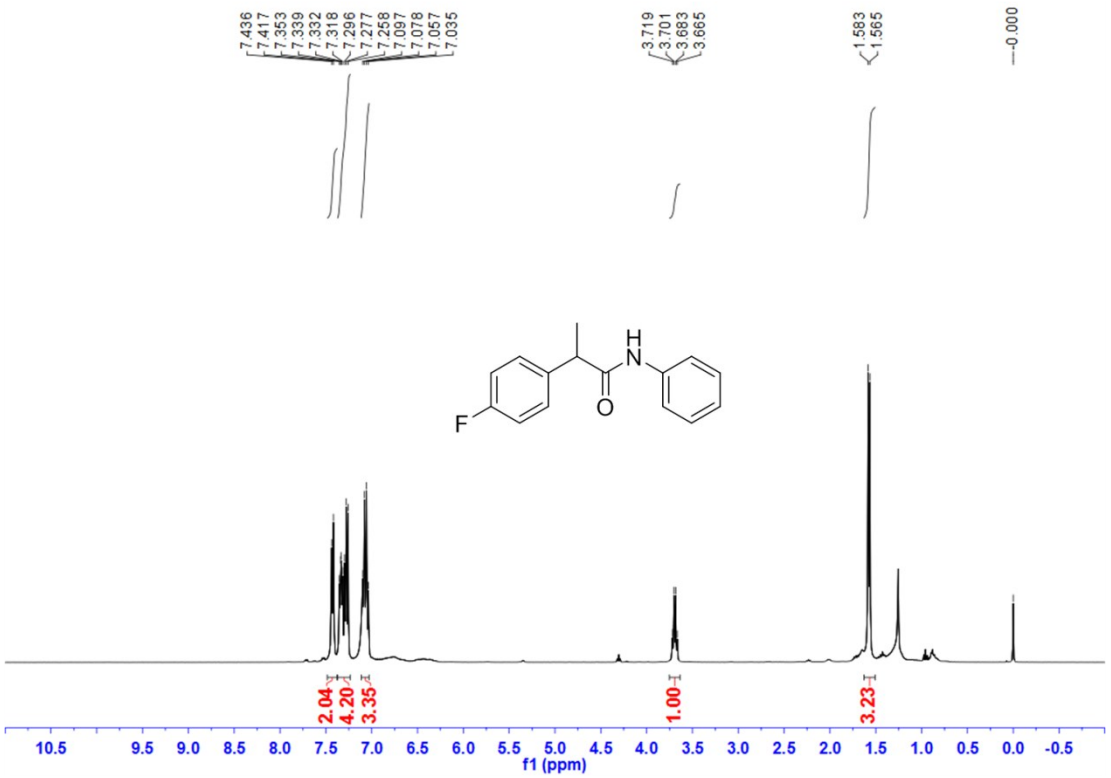
N
O
f1 (ppm)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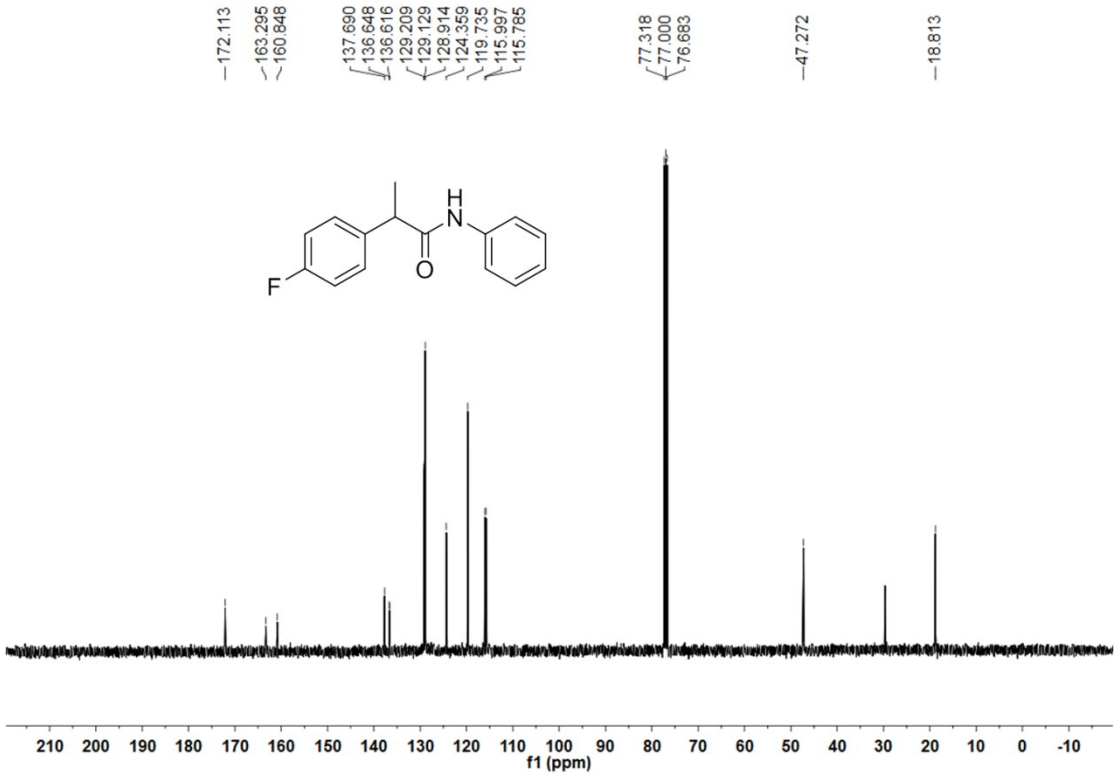
7.332
7.318
7.296
7.277
7.258
7.097
7.078
7.057
7.035
3.719
3.701
3.683
3.665
1.583
1.565
0.000
H
N
O
F
2.04
4.20
3.35
1.00
3.23
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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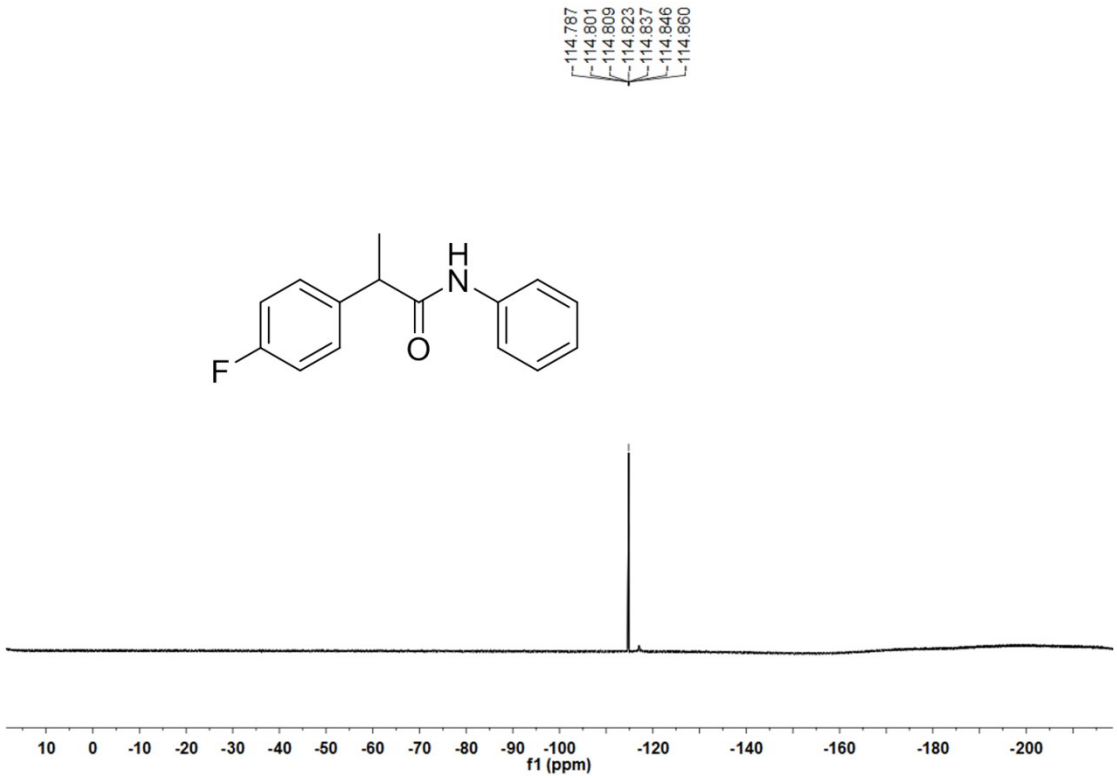
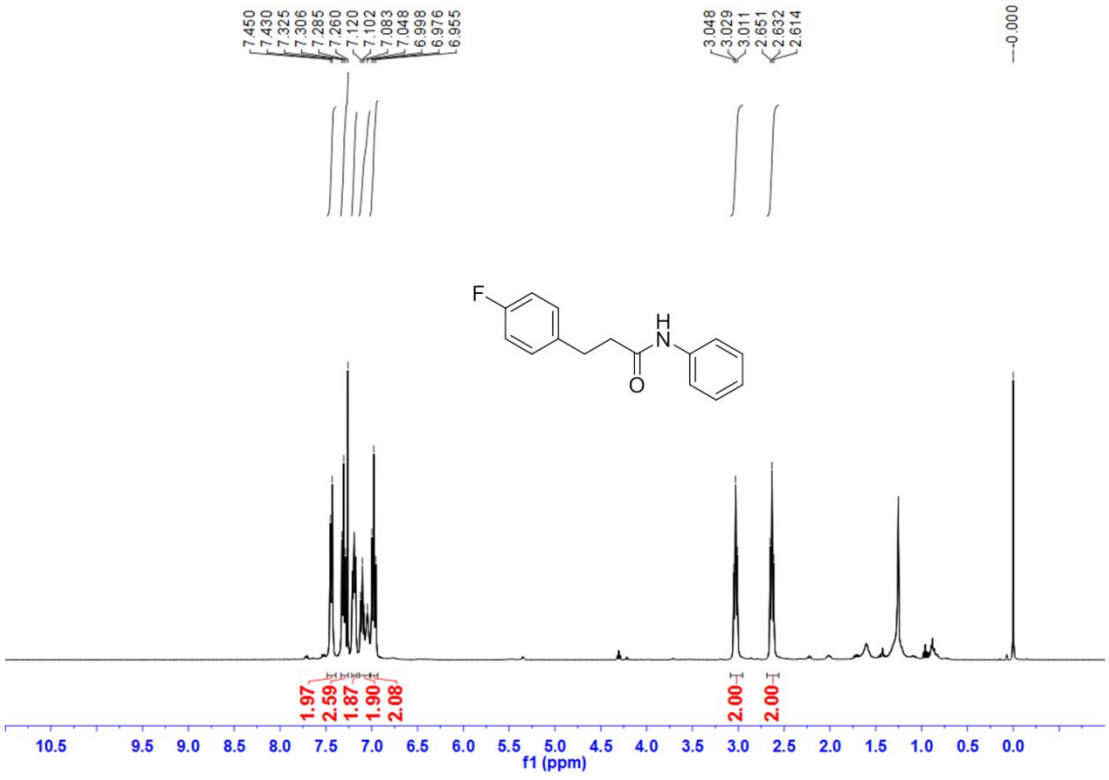
160.848
137.690
136.648
136.616
129.209
129.129
128.914
124.359
119.735
115.997
115.785
77.318
77.000
76.683
47.272
18.813
H
N
O
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


114.787
114.801
114.809
114.823
114.837
114.846
114.860
H
N
O
F
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)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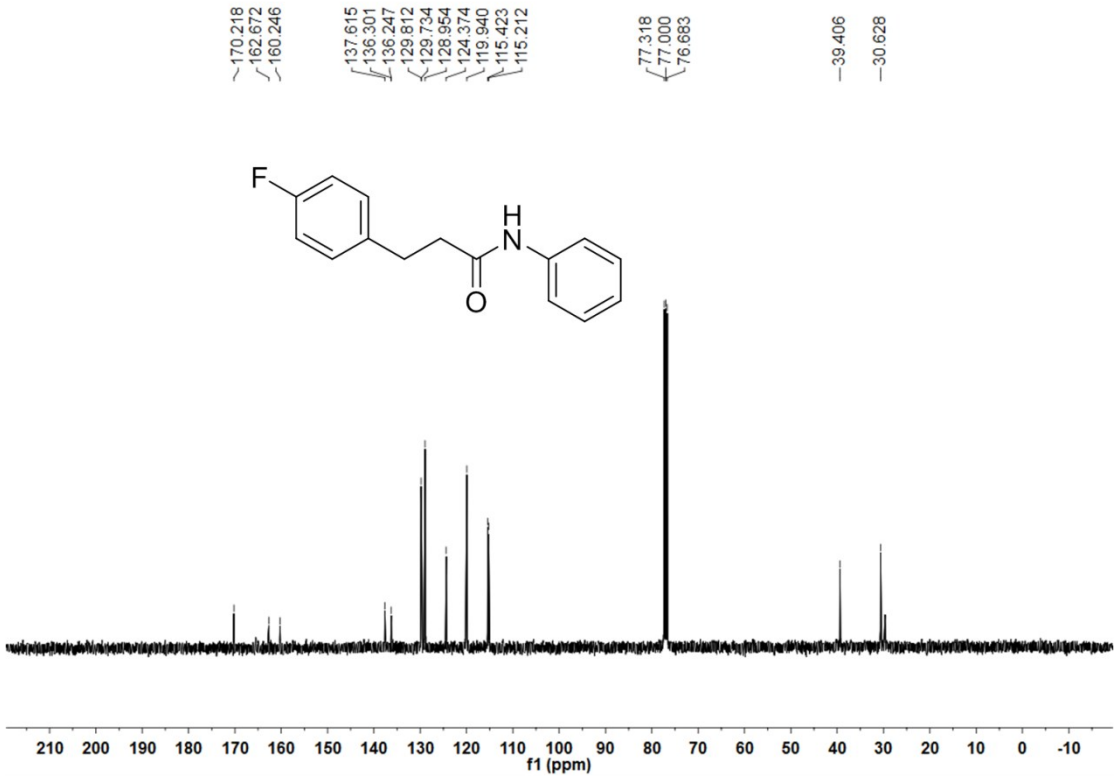
7.325
7.306
7.285
7.260
7.120
7.102
7.083
7.048
6.998
6.976
6.955
3.048
3.029
3.011
2.651
2.632
2.614
0.000
F
H
N
O
1.97
2.59
1.87
1.90
2.08
2.00
2.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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136.301
136.247
129.812
129.734
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124.374
119.940
115.423
115.212
77.318
77.000
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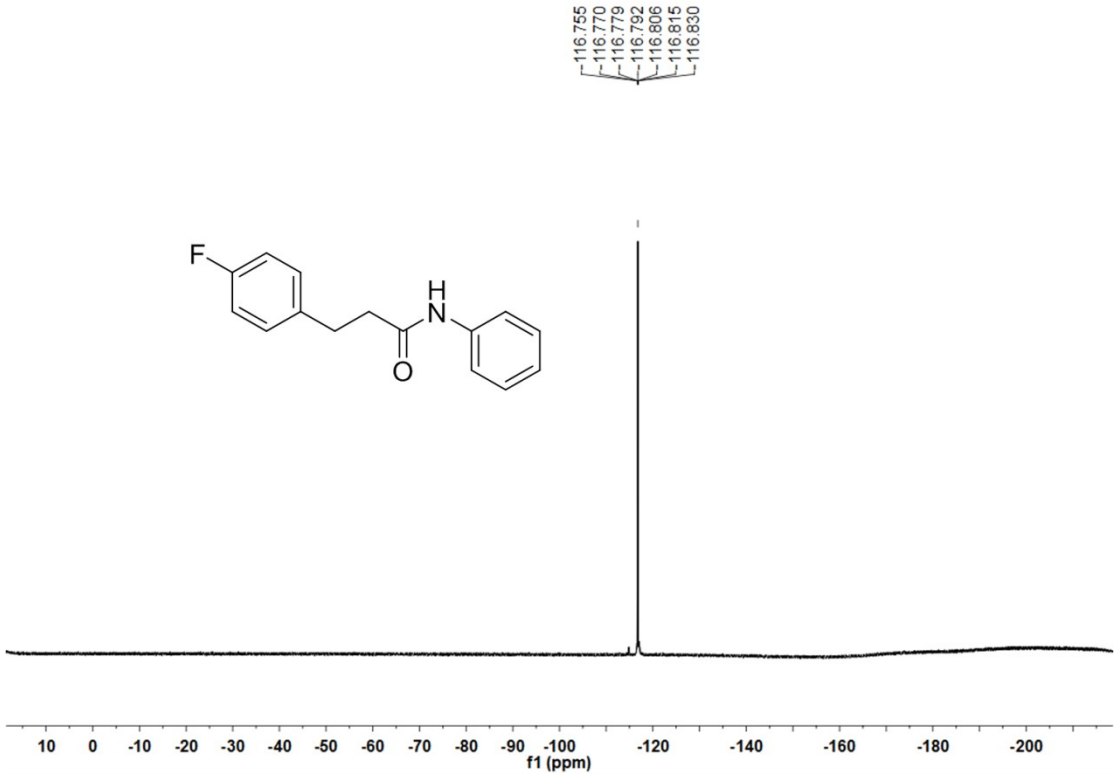
39.406
30.628
F
H
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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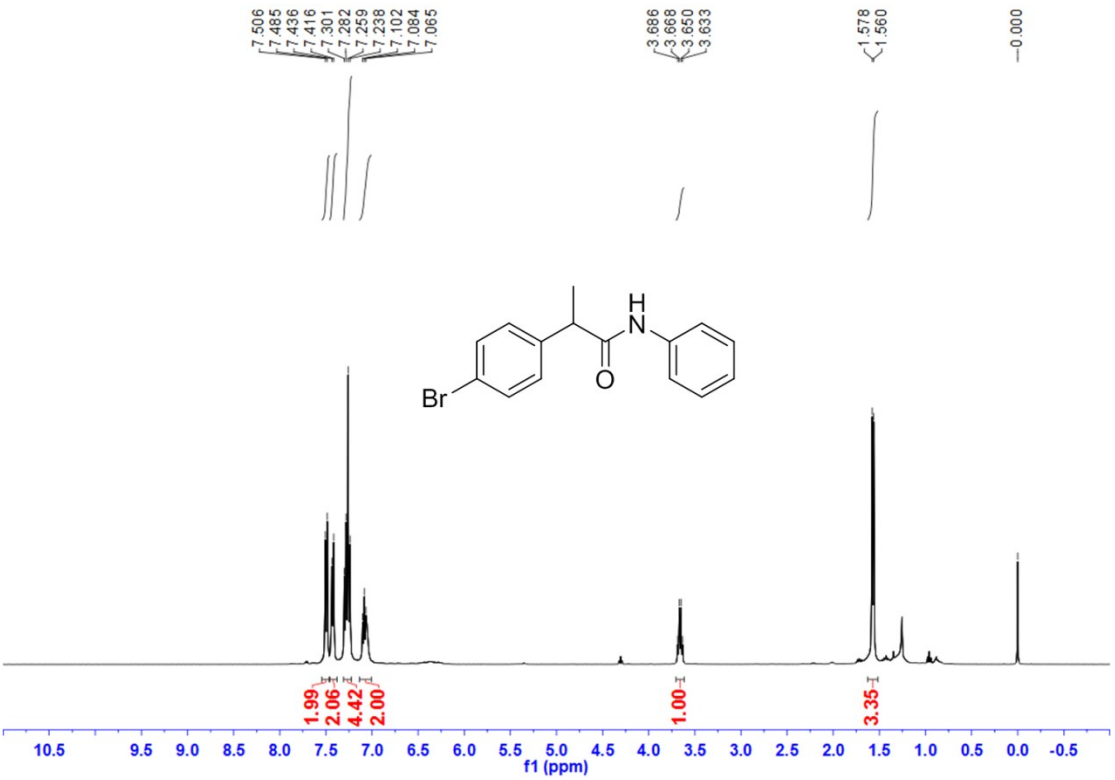
116.770
116.779
116.792
116.806
116.815
116.830
F
H
N
O
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)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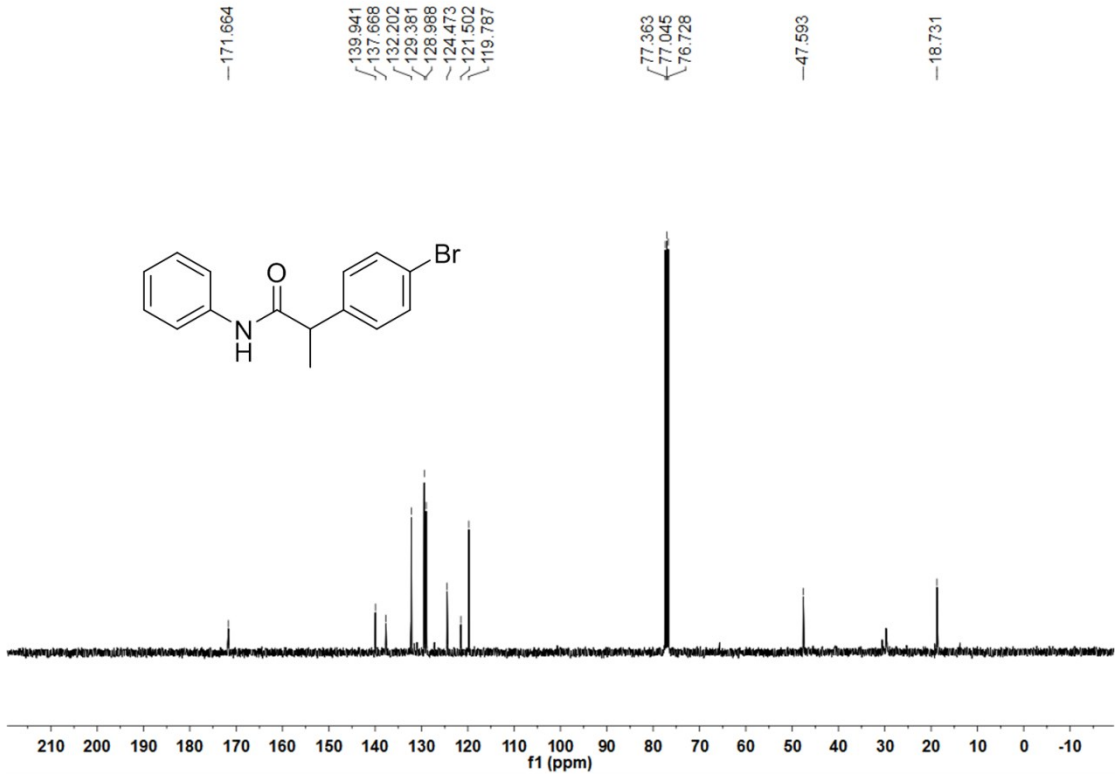
7.282
7.259
7.238
7.102
7.084
7.065
3.686
3.668
3.650
3.633
1.578
1.560
0.000
Br
H
N
O
1.99
2.06
4.42
2.00
1.00
3.35
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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137.668
132.202
129.381
128.988
124.473
121.502
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77.363
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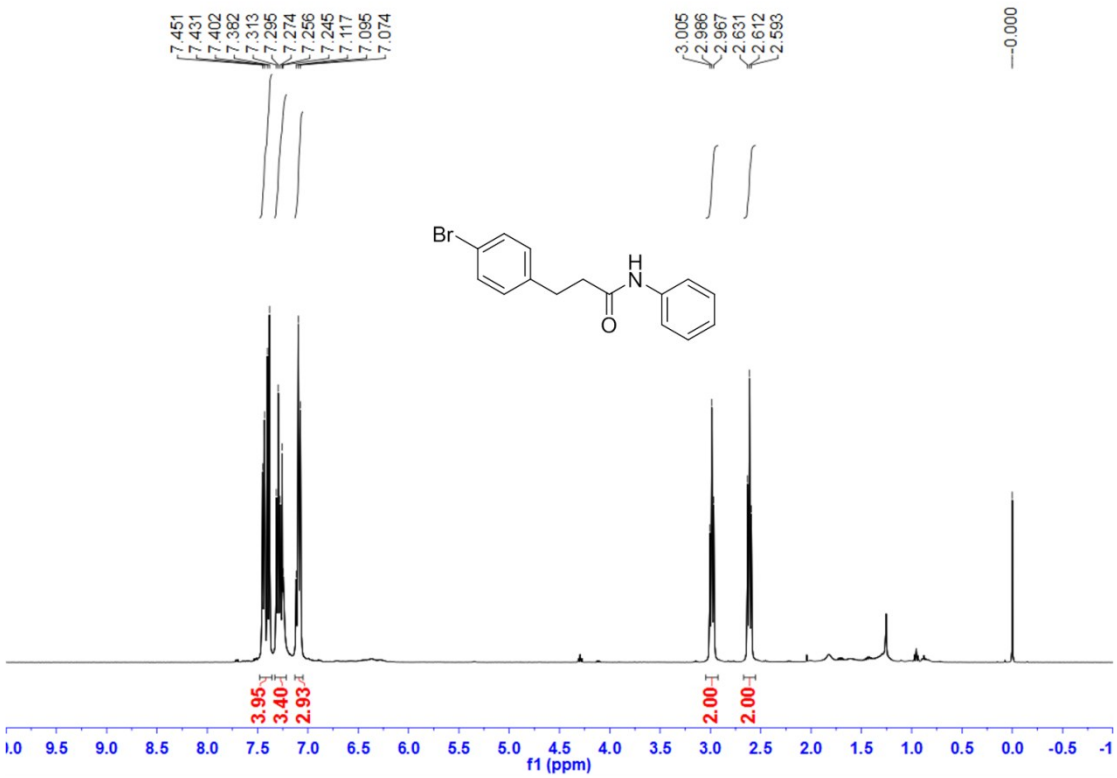
47.593
18.731
Br
O
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.295
7.274
7.256
7.245
7.117
7.095
7.074
3.005
2.986
2.967
2.631
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0.000
Br
H
N
O
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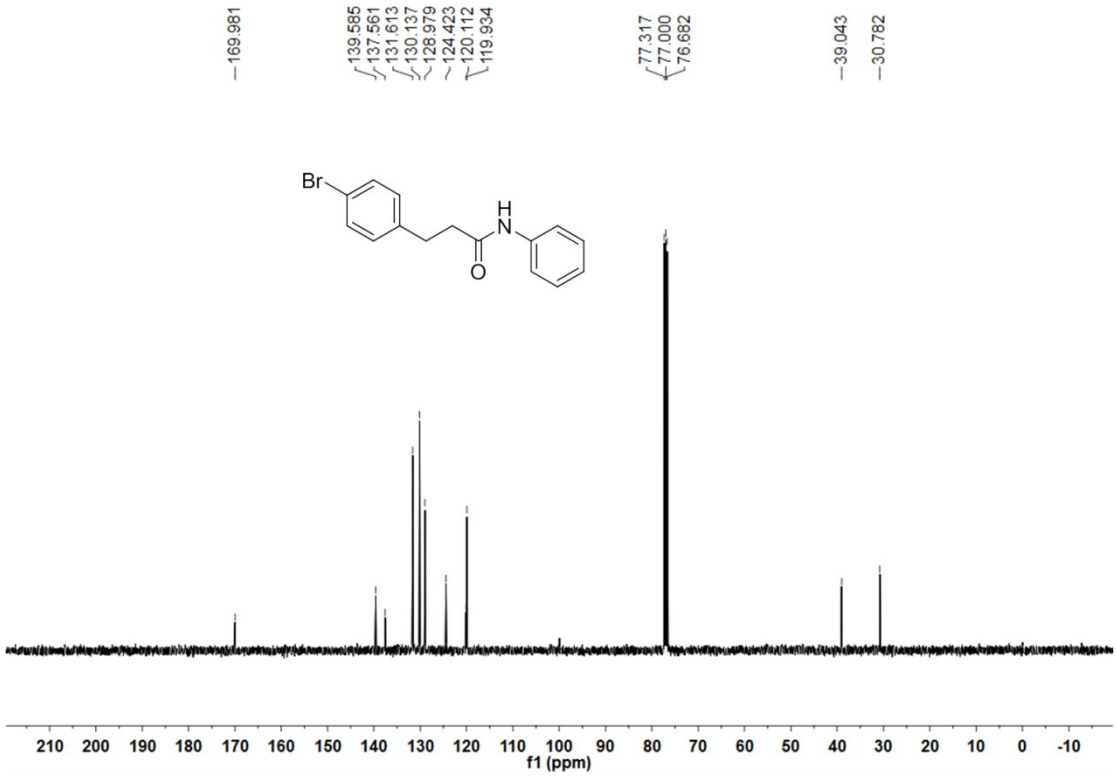
3.40
2.93
2.00
2.00
f1 (ppm)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128.979
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77.000
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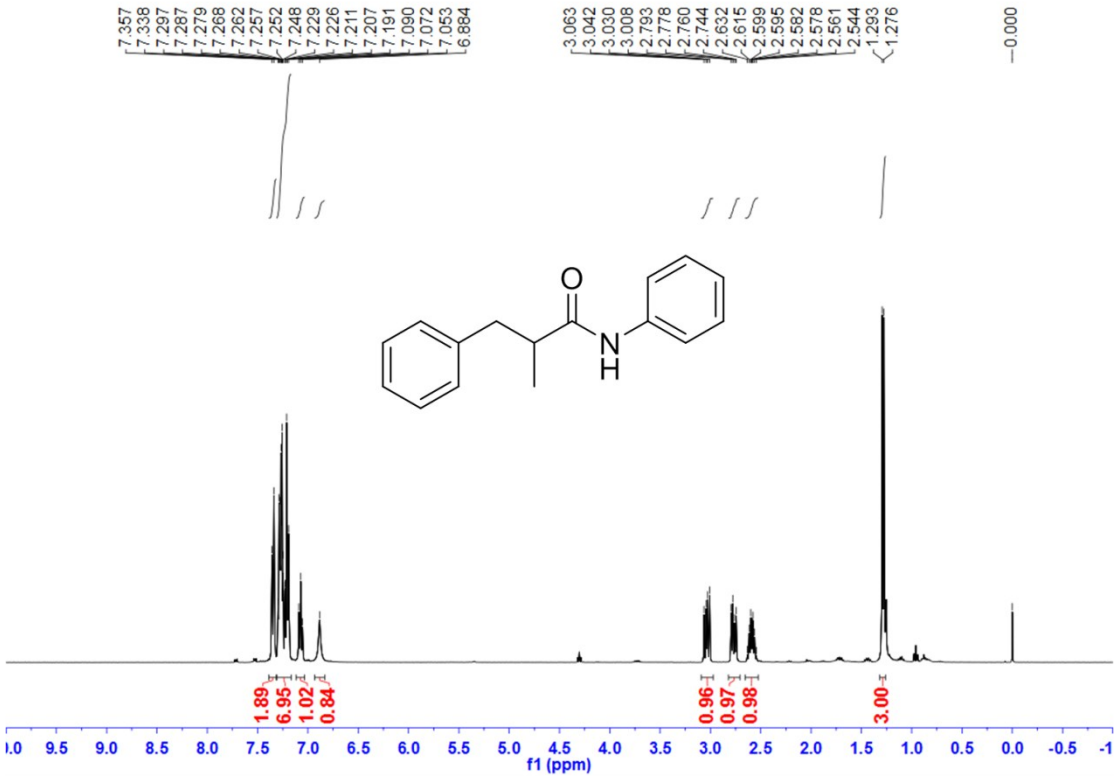
30.782
Br
H
N
O
f1 (ppm)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7.252
7.248
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7.226
7.211
7.207
7.191
7.090
7.072
7.053
6.884
3.063
3.042
3.030
3.008
2.793
2.778
2.760
2.744
2.632
2.615
2.599
2.595
2.582
2.578
2.561
2.544
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1.276
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0.97
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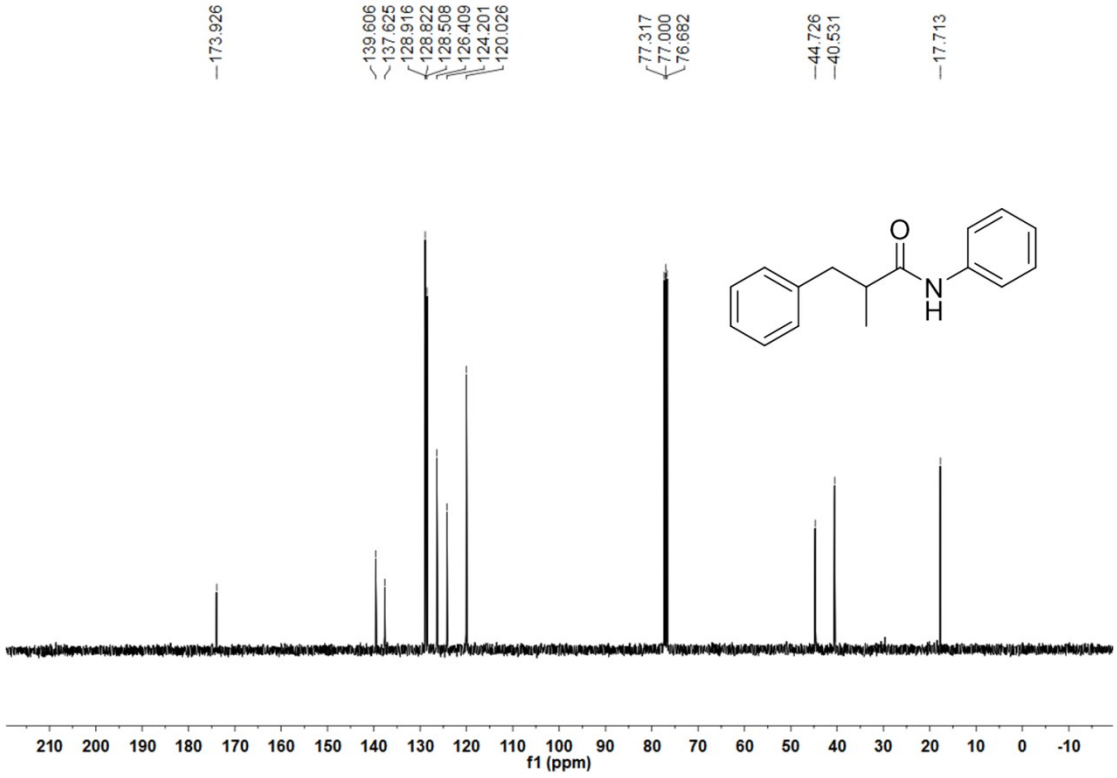
f1 (ppm)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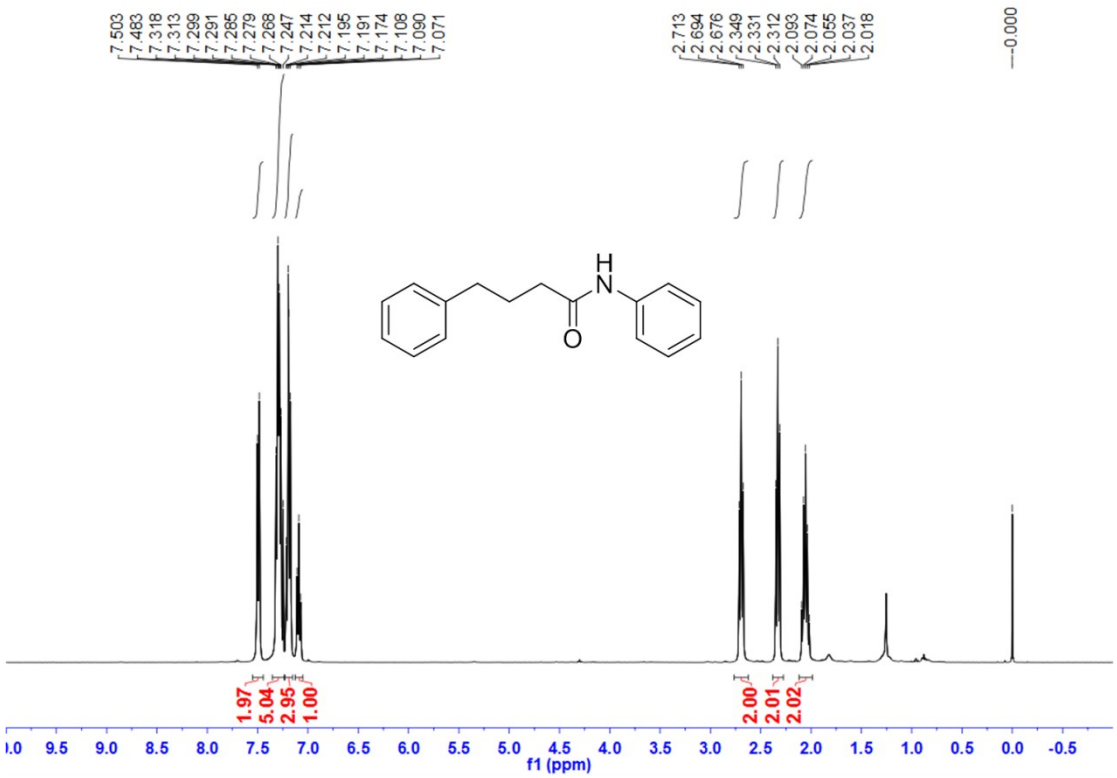
N
H
f1 (ppm)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7.212
7.195
7.191
7.174
7.108
7.090
7.071
2.713
2.694
2.676
2.349
2.331
2.312
2.093
2.074
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-0.000
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5.04
2.95
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2.00
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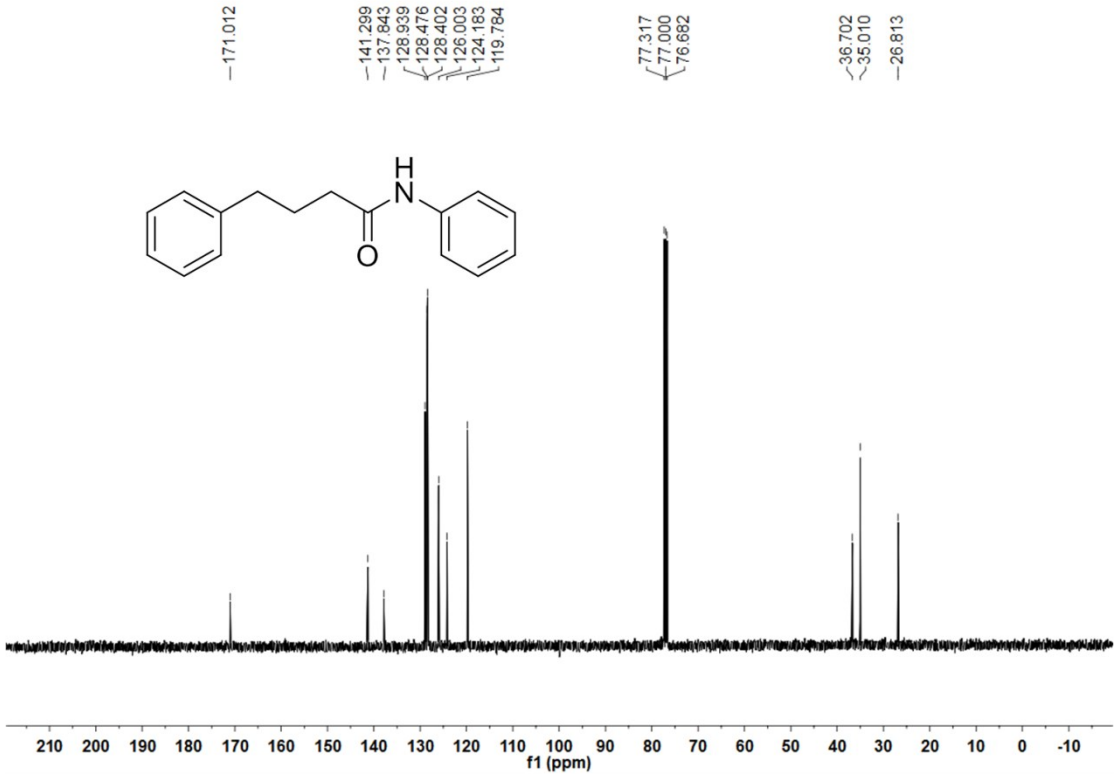
2.02
f1 (ppm)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77.000
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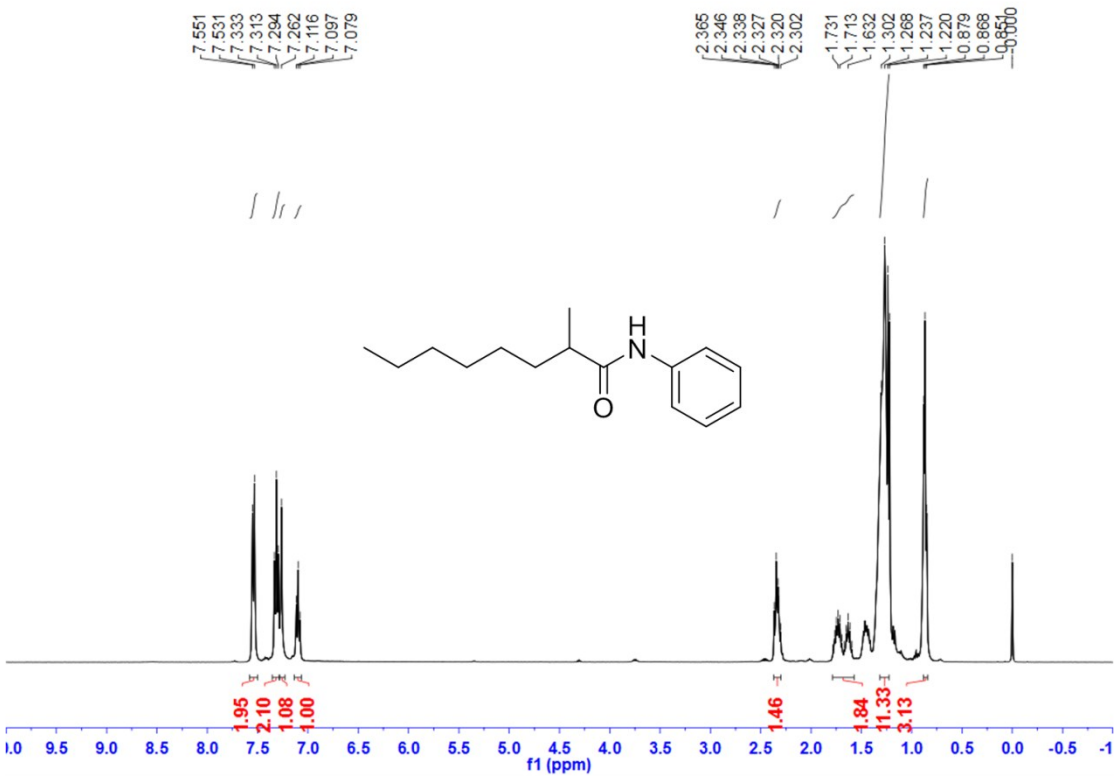
26.813
f1 (ppm)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7.097
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2.327
2.320
2.302
1.731
1.713
1.632
1.302
1.268
1.237
1.220
0.879
0.868
0.851
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1.08
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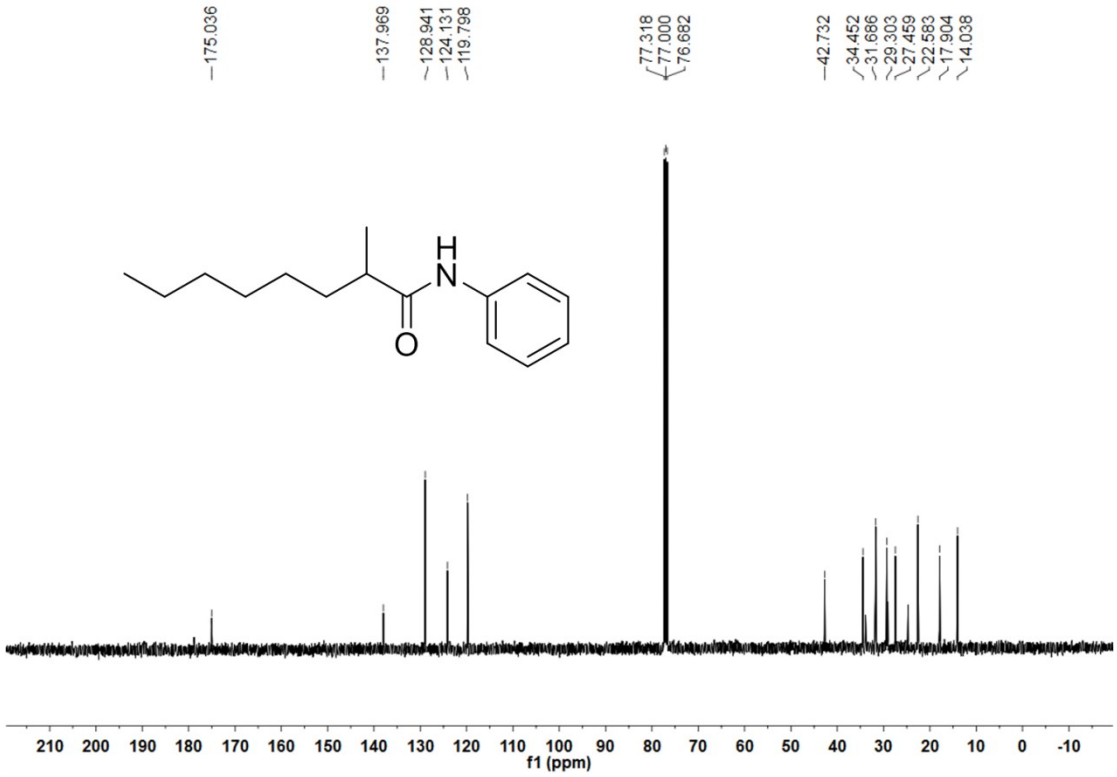
3.13
f1 (ppm)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77.000
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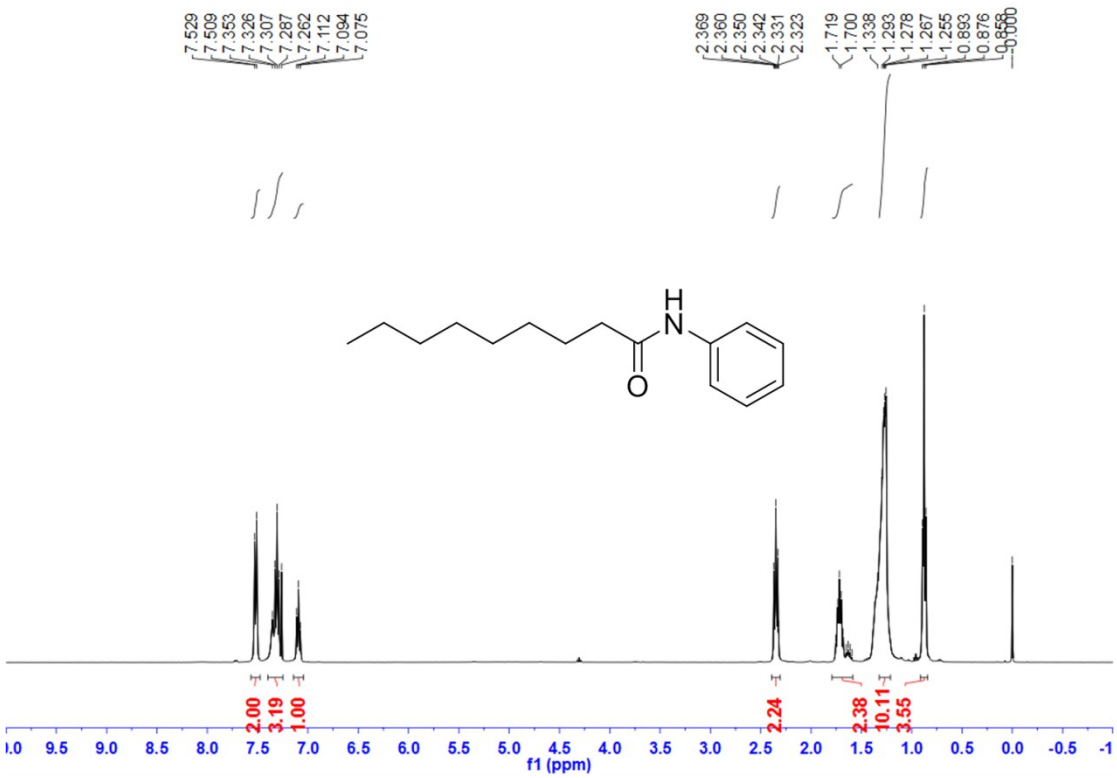
N
O
f1 (ppm)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2.331
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1.700
1.338
1.293
1.278
1.267
1.255
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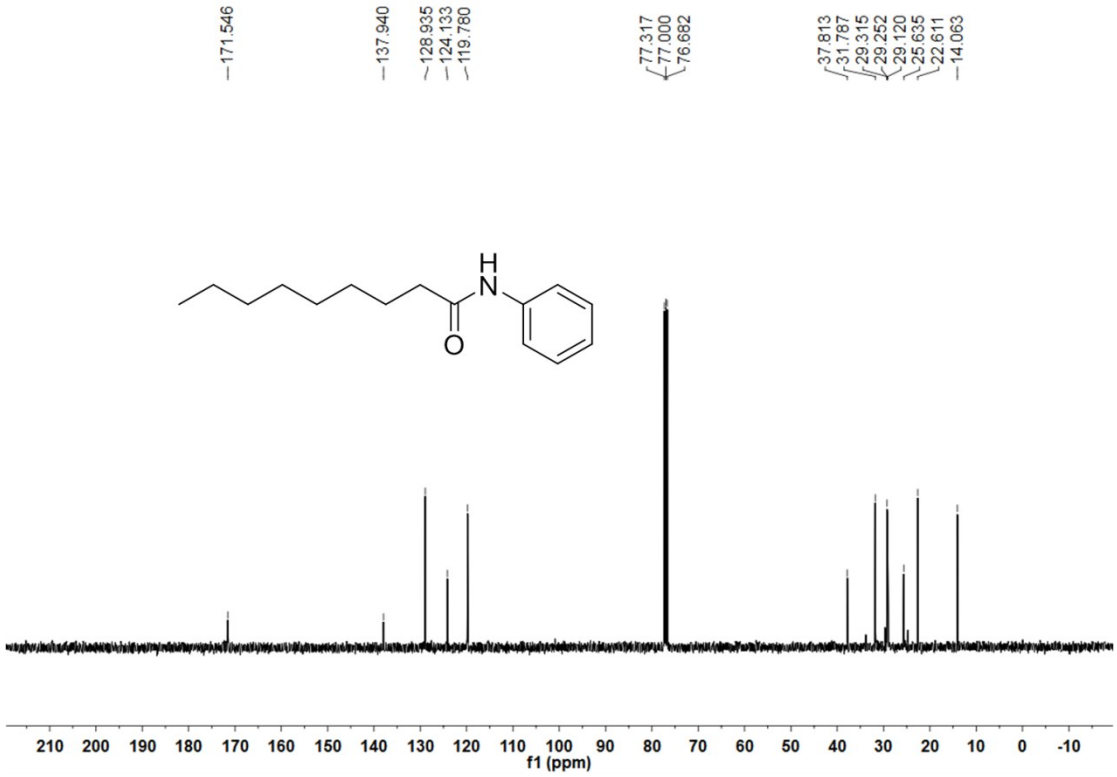
f1 (ppm)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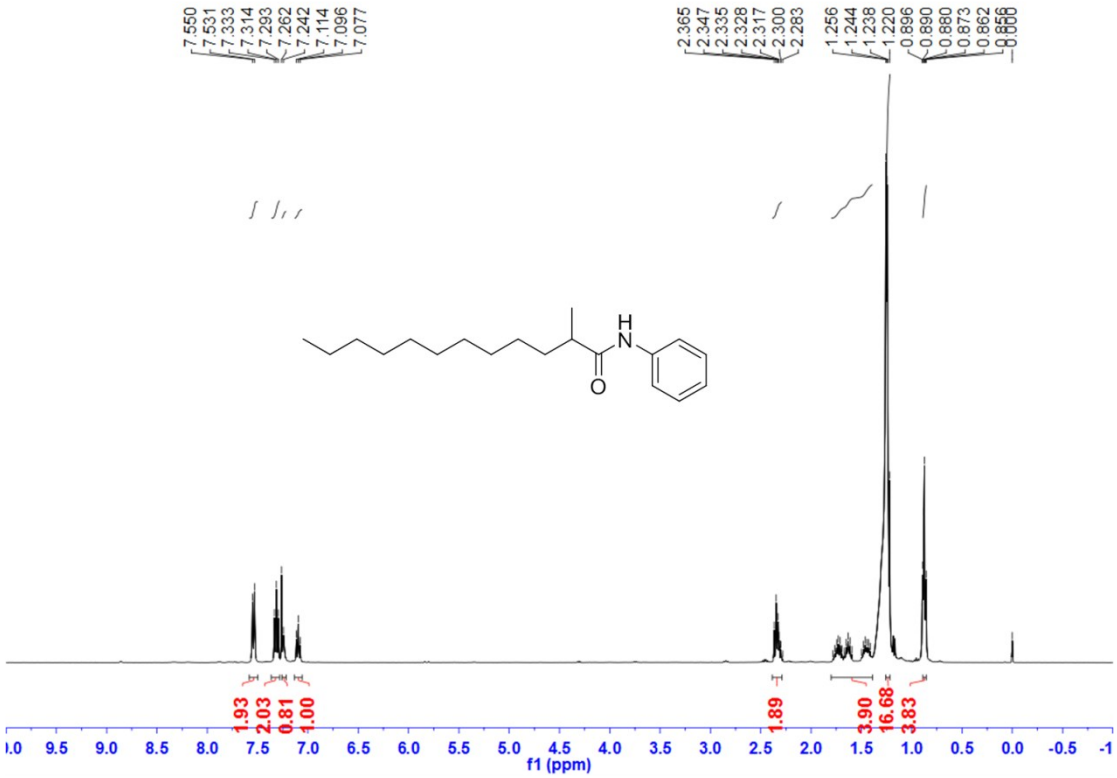
N
O
f1 (ppm)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2.328
2.317
2.300
2.283
1.256
1.244
1.238
1.220
0.896
0.890
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1.93
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1.00
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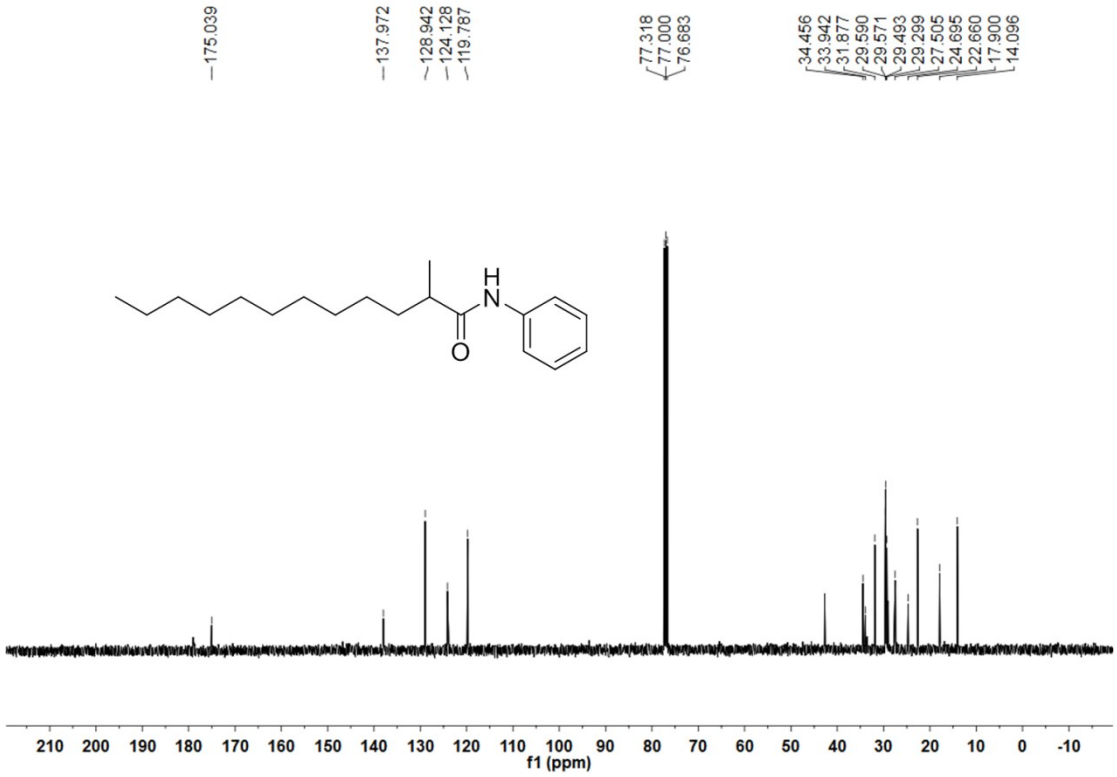
3.83
f1 (ppm)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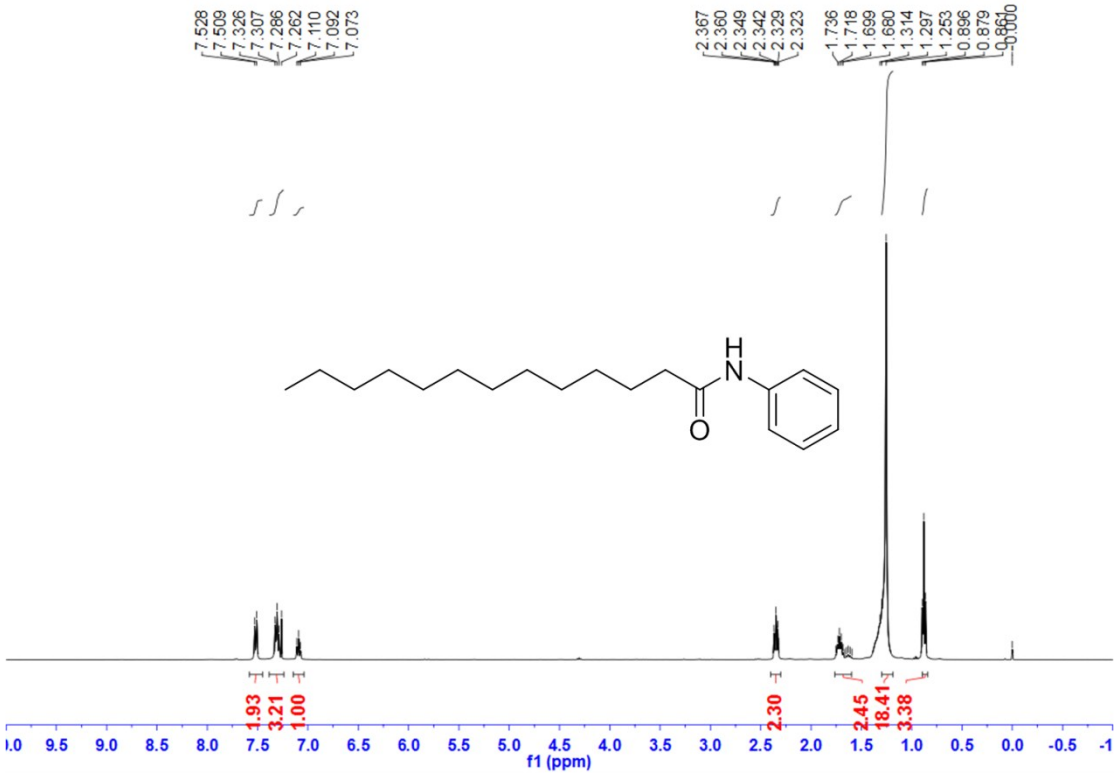
14.096
f1 (ppm)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2.329
2.323
1.736
1.718
1.699
1.680
1.314
1.297
1.253
0.896
0.879
0.861
0.000
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N
O
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1.00
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2.45
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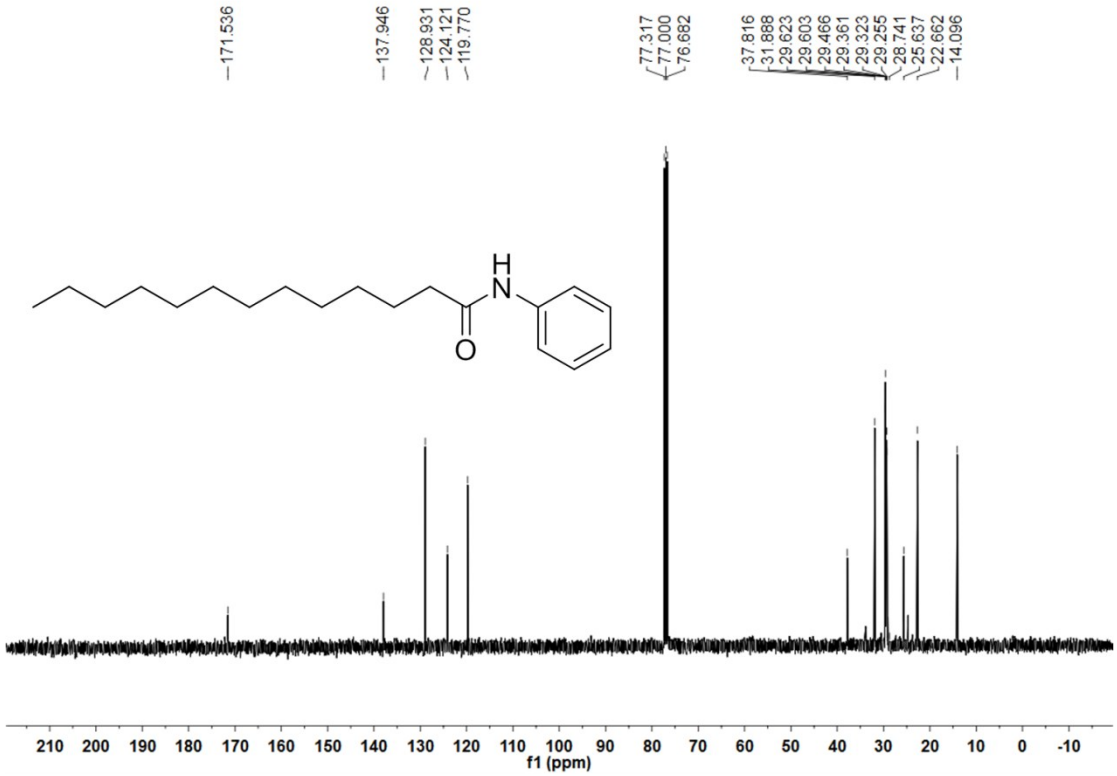
3.38
f1 (ppm)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25.637
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14.096
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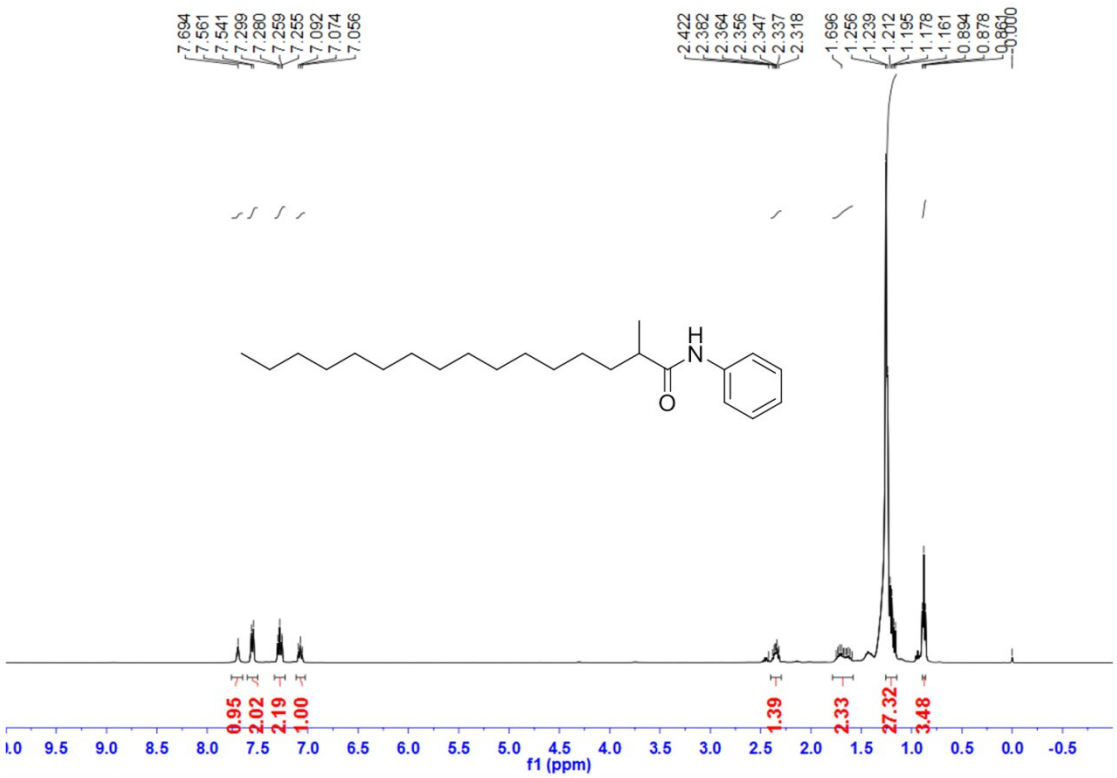
N
O
f1 (ppm)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1.161
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0.878
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H
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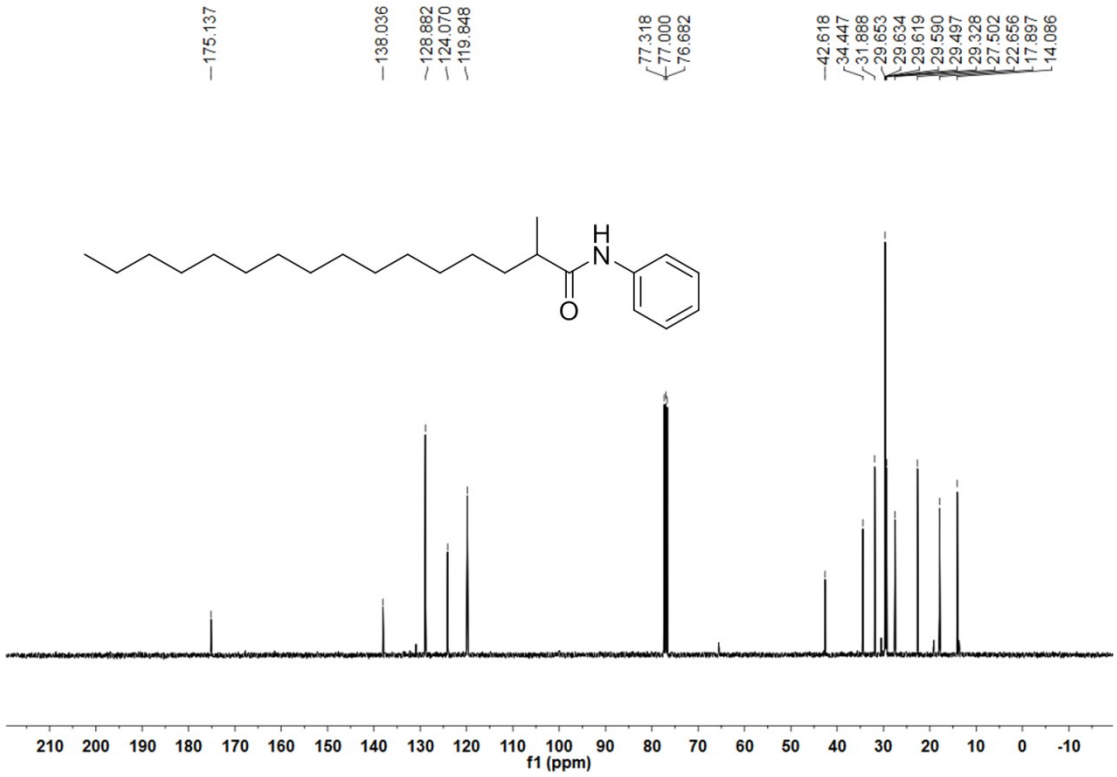
2.19
1.00
1.39
2.33
27.32
3.48
f1 (ppm)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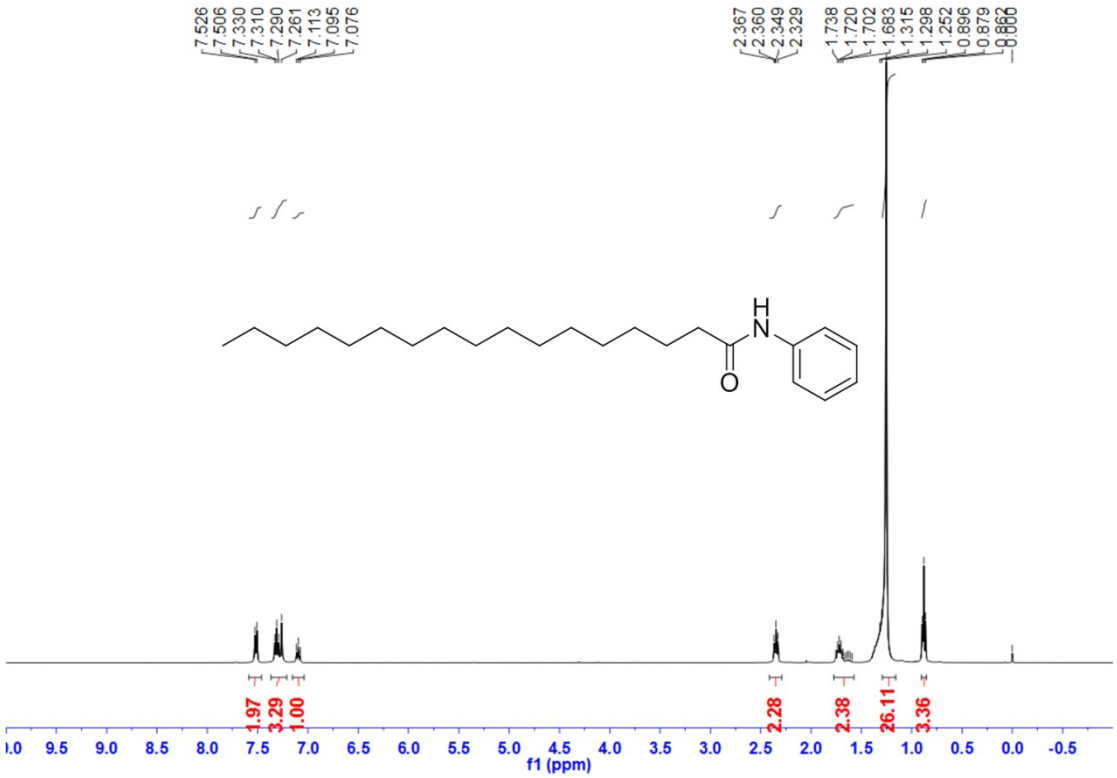
138.036
128.882
124.070
119.848
77.318
77.000
76.682
42.618
34.447
31.888
29.653
29.634
29.619
29.590
29.497
29.328
27.502
22.656
17.897
14.086
H
N
O
f1 (ppm)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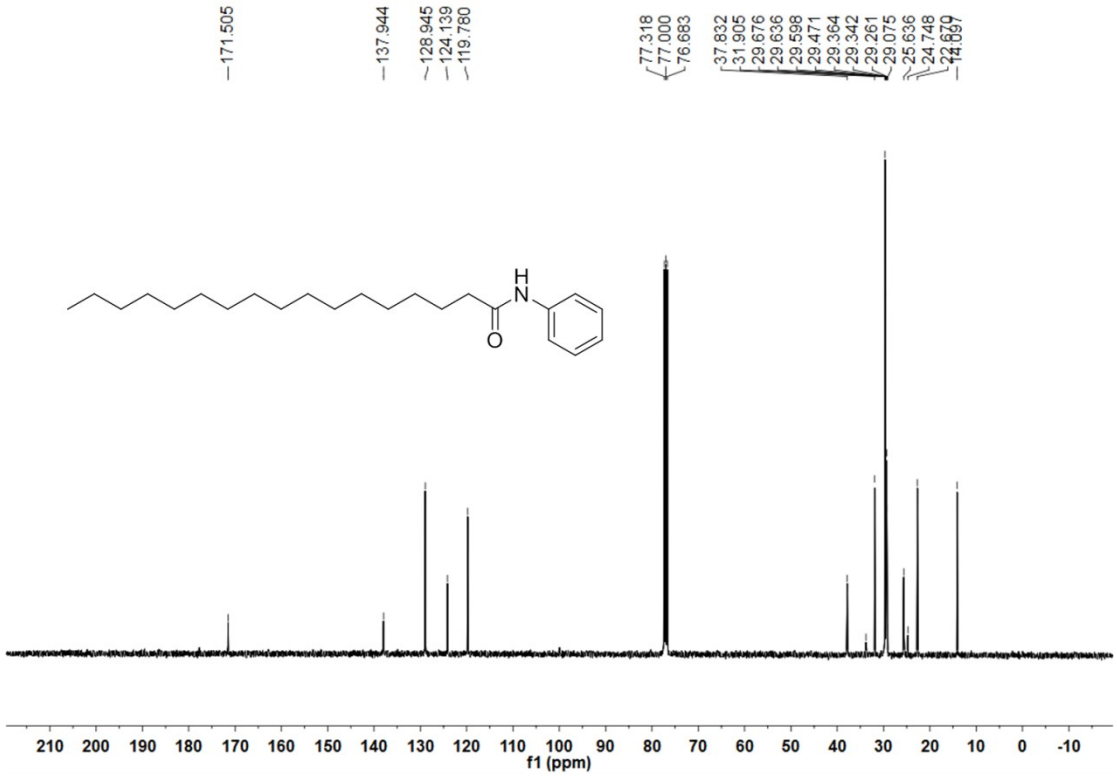
7.506
7.330
7.310
7.290
7.261
7.113
7.095
7.076
2.367
2.360
2.349
2.329
1.738
1.720
1.702
1.683
1.315
1.298
1.252
0.896
0.879
1.97
3.29
1.00
2.28
2.38
26.11
3.36
f1 (ppm)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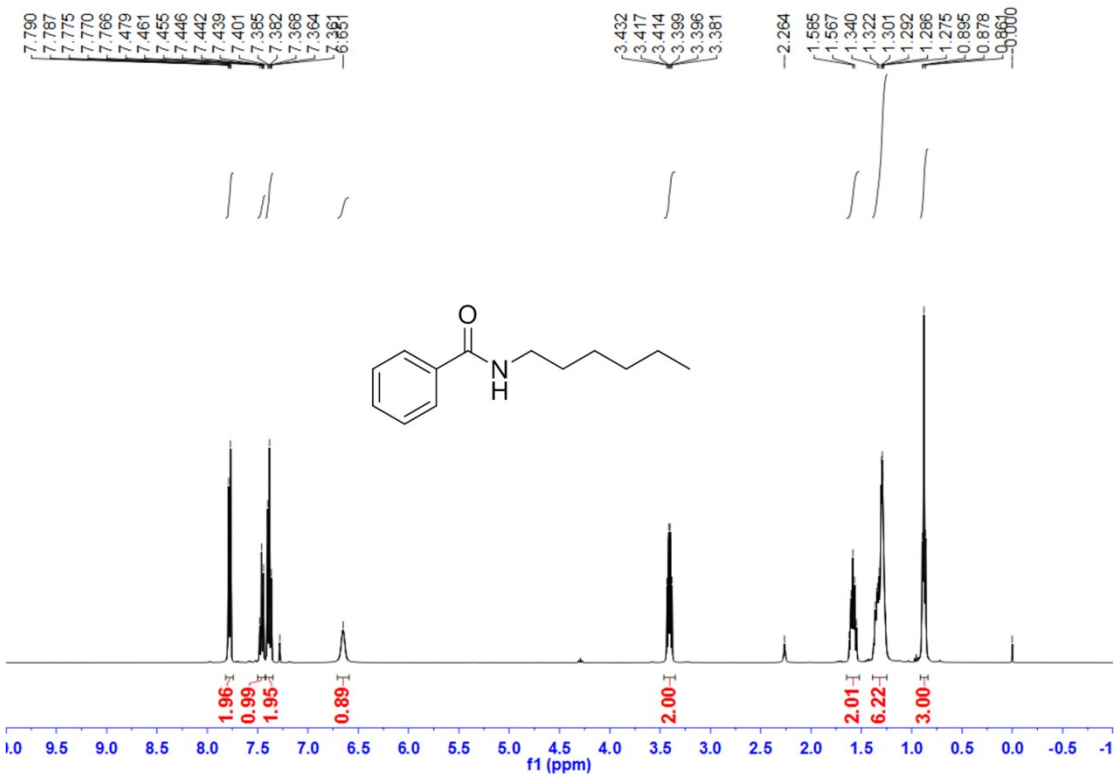
137.944
128.945
124.139
119.780
77.318
77.000
76.683
37.832
31.905
29.676
29.636
29.598
29.471
29.364
29.342
29.261
29.075
25.636
24.748
22.670
14.097
f1 (ppm)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7.787
7.775
7.770
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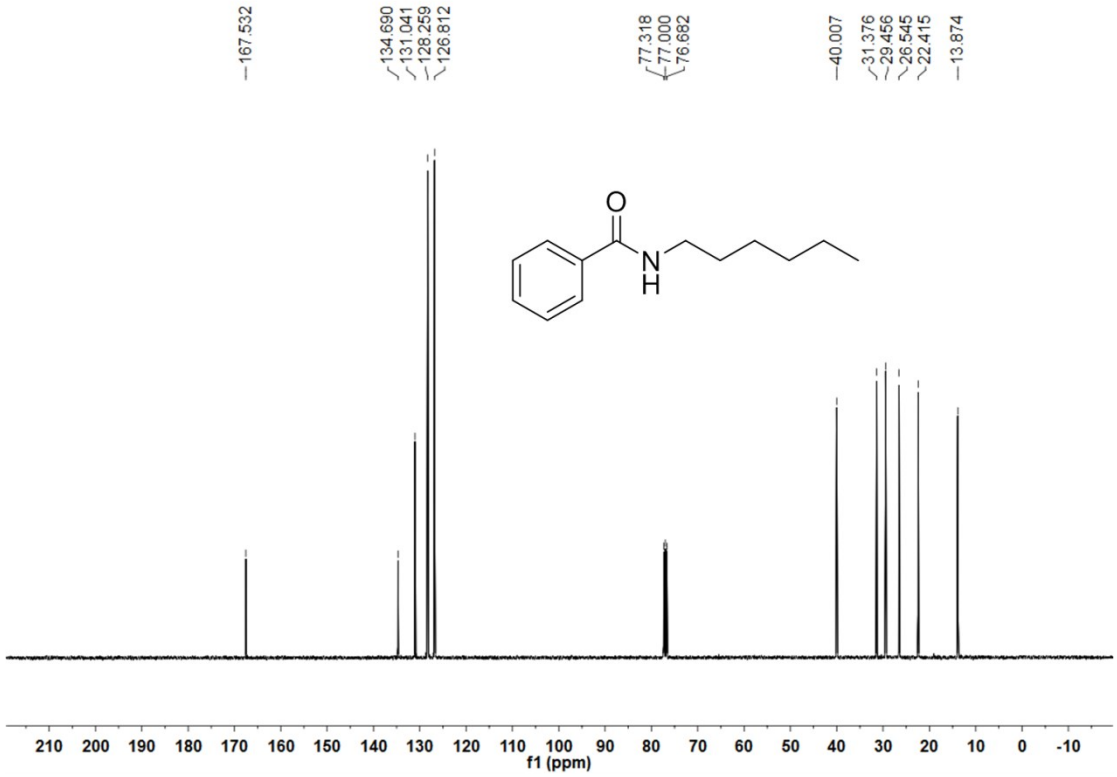
7.455
7.446
7.442
7.439
7.401
7.385
7.382
7.368
7.364
7.361
6.851
3.432
3.417
3.414
3.399
3.396
3.381
2.264
1.585
1.567
1.340
1.322
1.301
1.292
1.286
1.275
0.895
0.878
0.861
0.000
O
N
H
1.96
0.99
1.95
0.89
2.00
2.01
6.22
3.00
f1 (ppm)


167.532
134.690
131.041
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126.812
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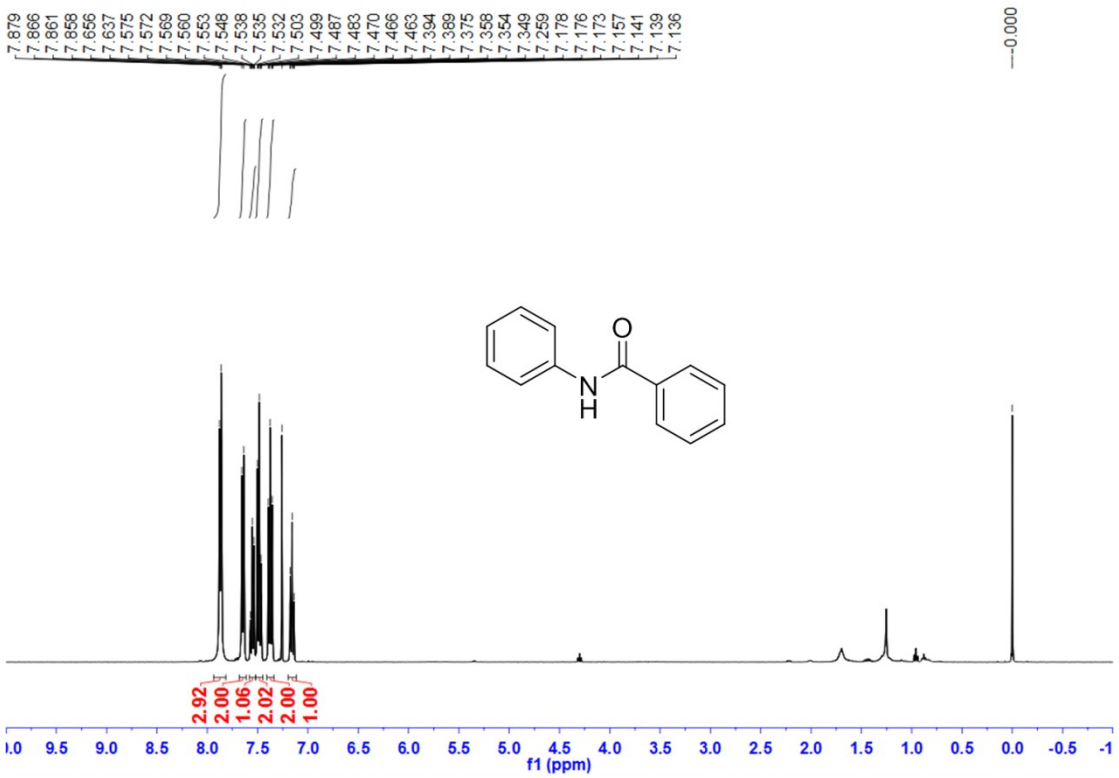
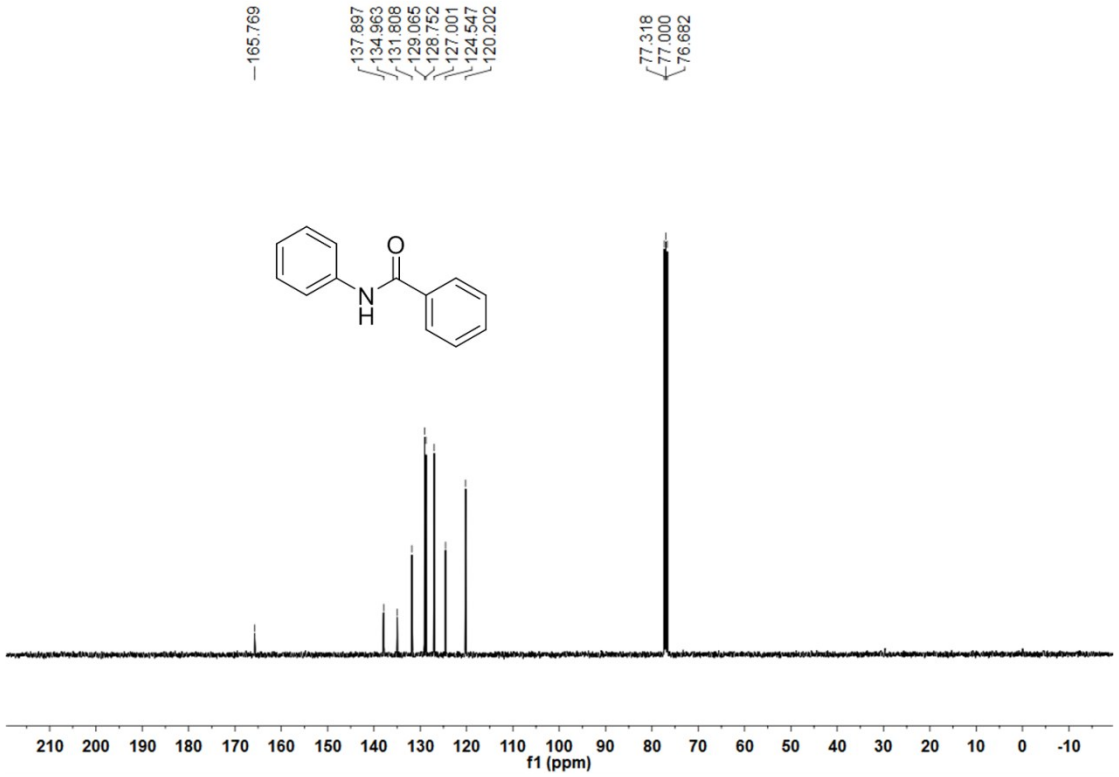
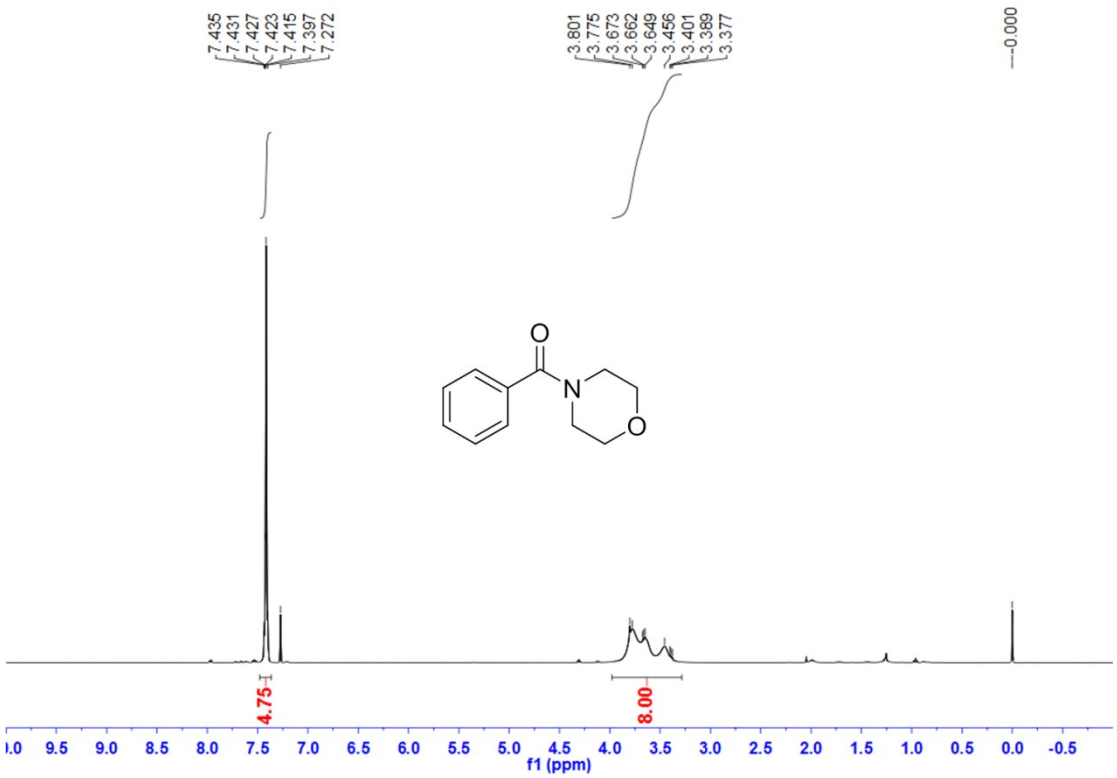
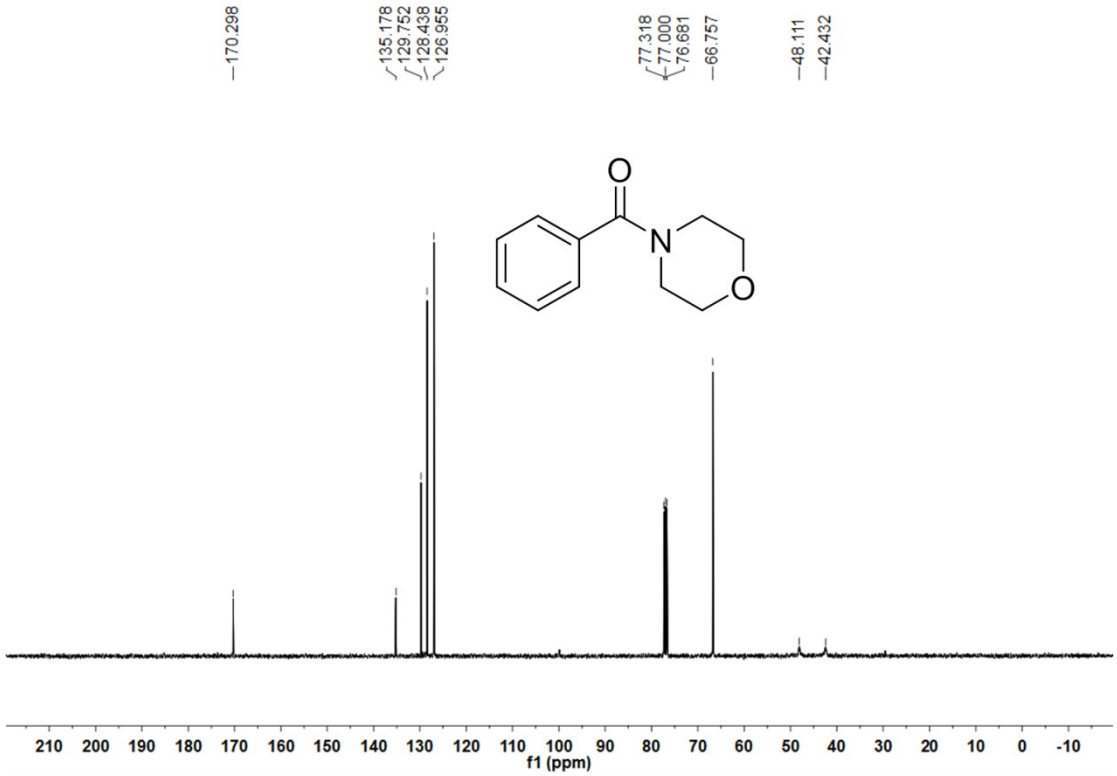
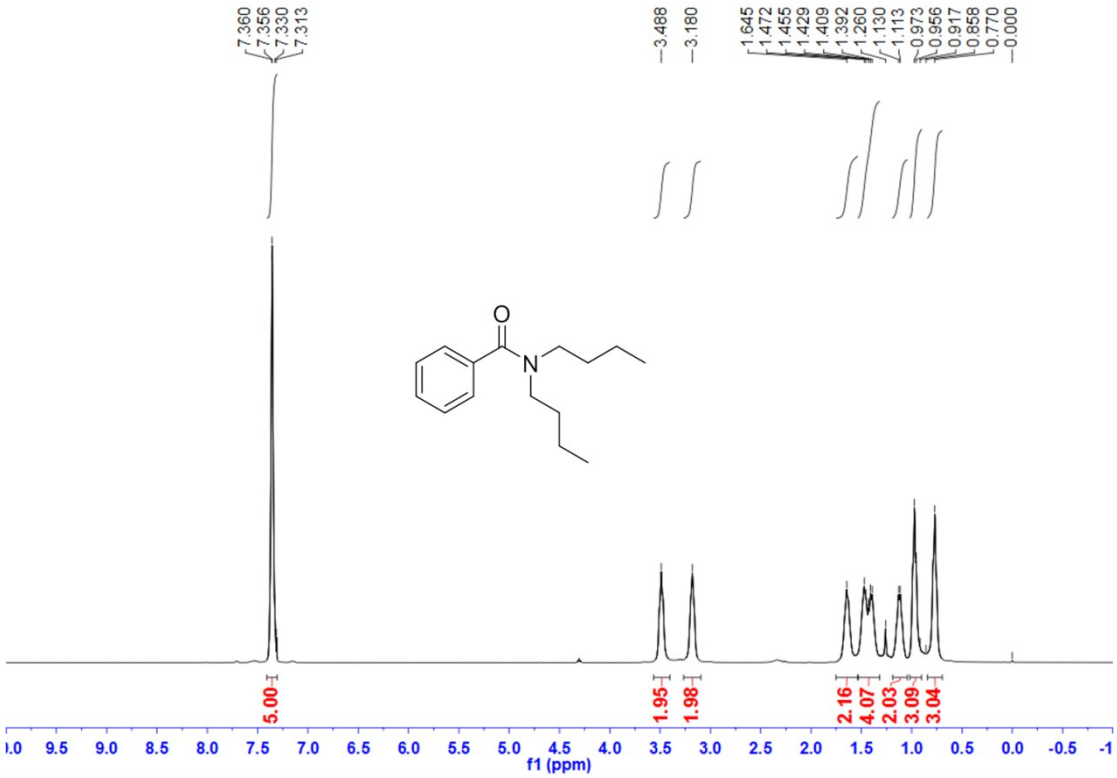
77.000
76.682
40.007
31.376
29.456
26.545
22.415
13.874
O
N
H
f1 (ppm)

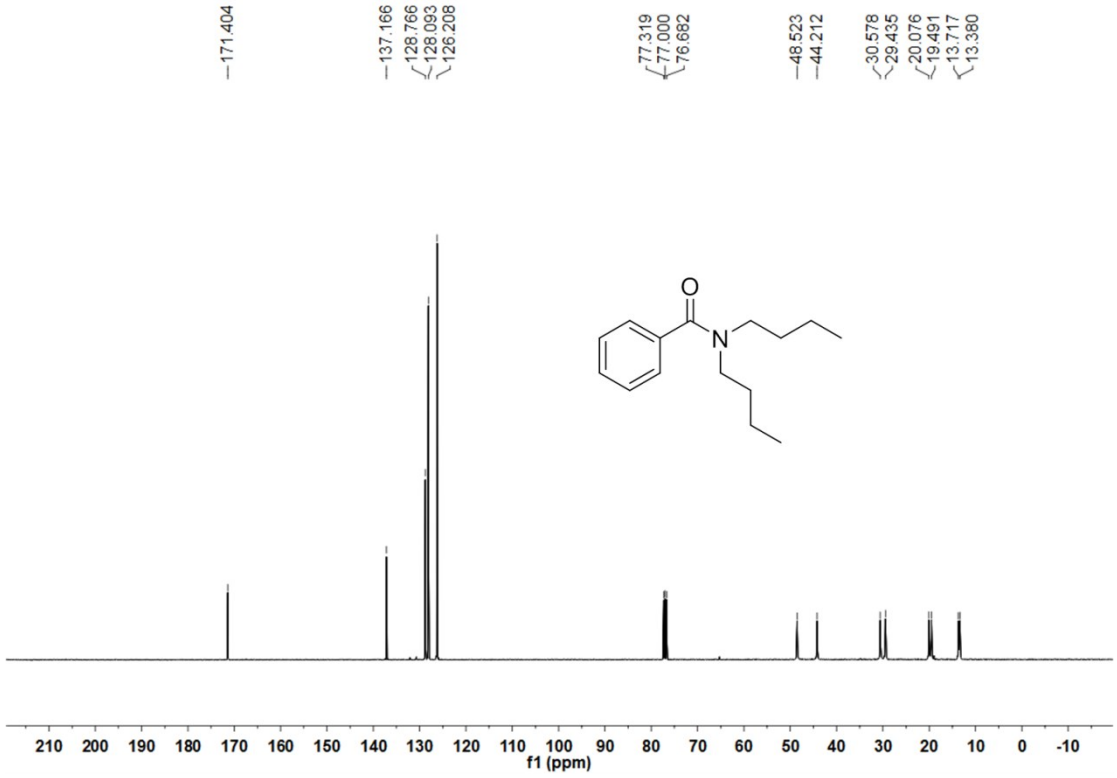

7.879
7.866
7.861
7.858
7.656
7.637
7.575
7.572
7.569
7.560
7.553
7.548
7.538
7.535
7.532
7.503
7.499
7.487
7.483
7.470
7.466
7.463
7.394
7.389
7.375
7.358
7.354
7.349
7.259
7.178
7.176
7.173
7.157
7.141
7.139
7.136
0.000
O
N
H
2.92
2.00
1.06
2.02
2.00
1.00
f1 (ppm)

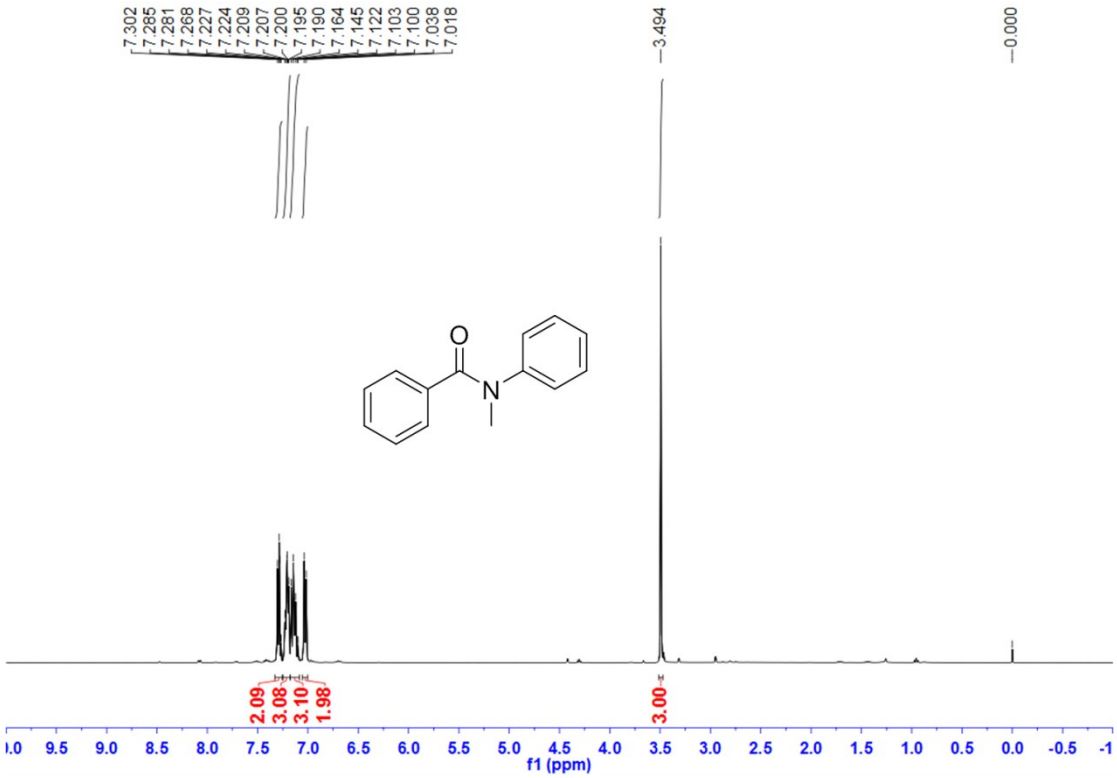

165.769
137.897
134.963
131.808
129.065
128.752
127.001
124.547
120.202
77.318
77.000
76.682
O
N
H
f1 (ppm)

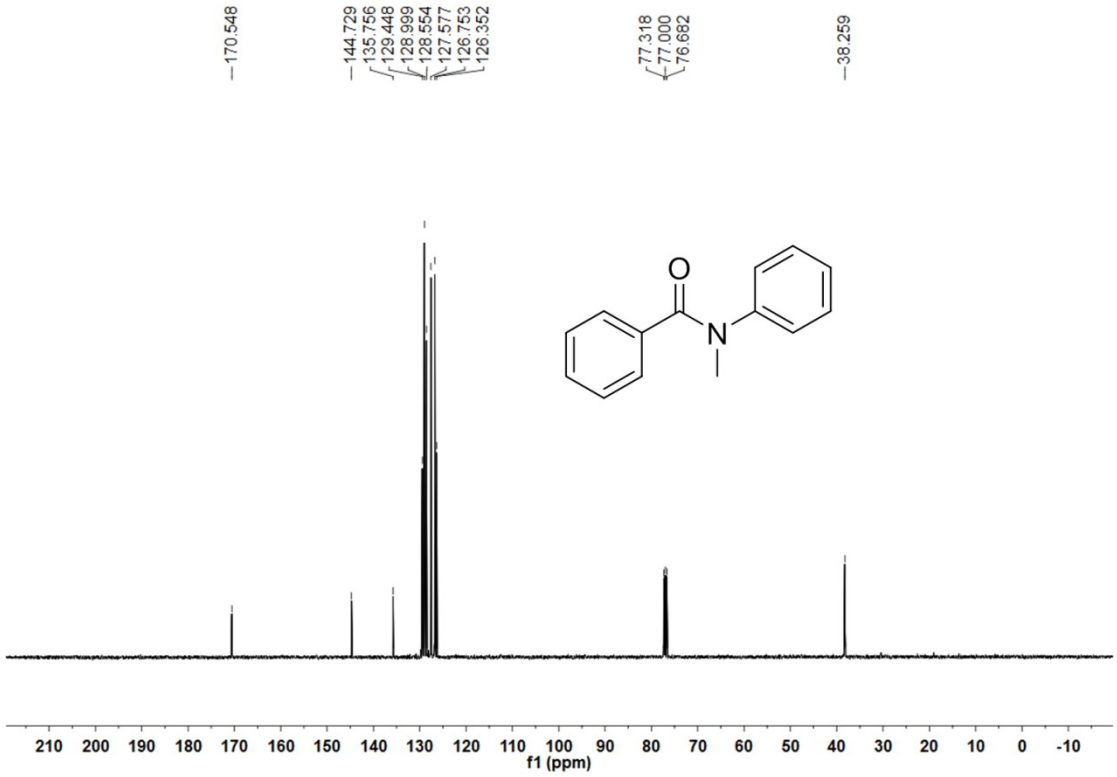

7.435
7.431
7.427
7.423
7.415
7.397
7.272
3.801
3.775
3.673
3.662
3.649
3.456
3.401
3.389
3.377
-0.000
O
N
O
4.75
8.00
f1 (ppm)

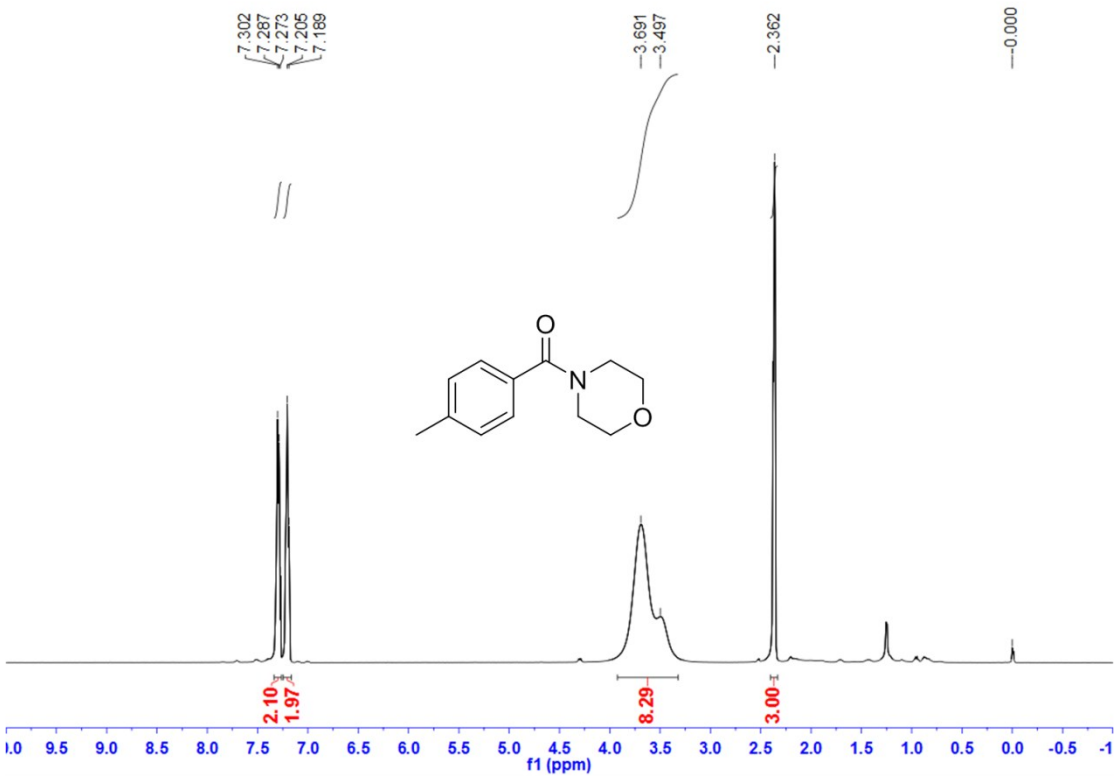

170.298
135.178
129.752
128.438
126.955
77.318
77.000
76.681
66.757
48.111
42.432
O
N
O
f1 (ppm)

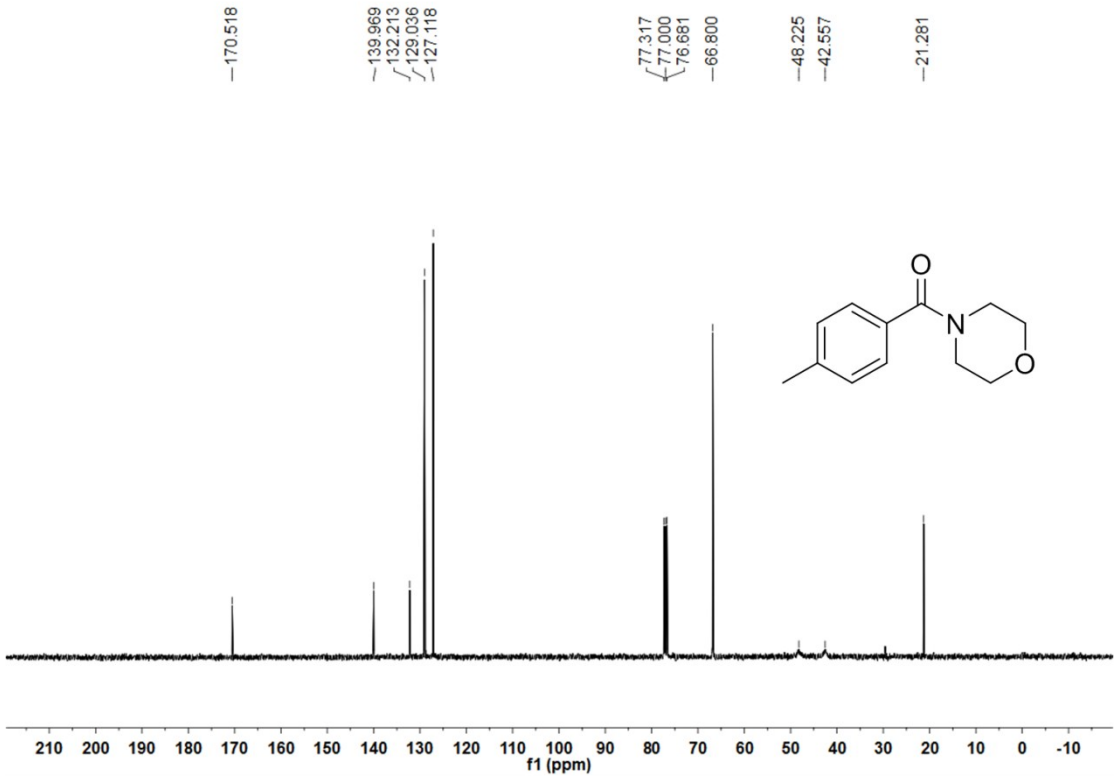

7.360
7.356
7.330
7.313
3.488
3.180
1.645
1.472
1.455
1.429
1.409
1.392
1.260
1.130
1.113
0.973
0.956
0.917
0.858
0.770
0.000
O
N
5.00
1.95
1.98
2.16
4.07
2.03
3.09
3.04
f1 (ppm)


171.404
137.166
128.766
128.093
126.208
77.319
77.000
76.682
48.523
44.212
30.578
29.435
20.076
19.491
13.717
13.380
O
N
f1 (ppm)


7.302
7.285
7.281
7.268
7.227
7.224
7.209
7.207
7.200
7.195
7.190
7.164
7.145
7.122
7.103
7.100
7.038
7.018
3.494
0.000
O
N
2.09
3.08
3.10
1.98
3.00
f1 (ppm)


170.548
144.729
135.756
129.448
128.999
128.554
127.577
126.753
126.352
77.318
77.000
76.682
38.259
O
N
f1 (ppm)


7.302
7.287
7.273
7.205
7.189
3.691
3.497
2.362
0.000
O
N
O
2.10
1.97
8.29
3.00
f1 (ppm)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139.969
132.213
129.036
127.118
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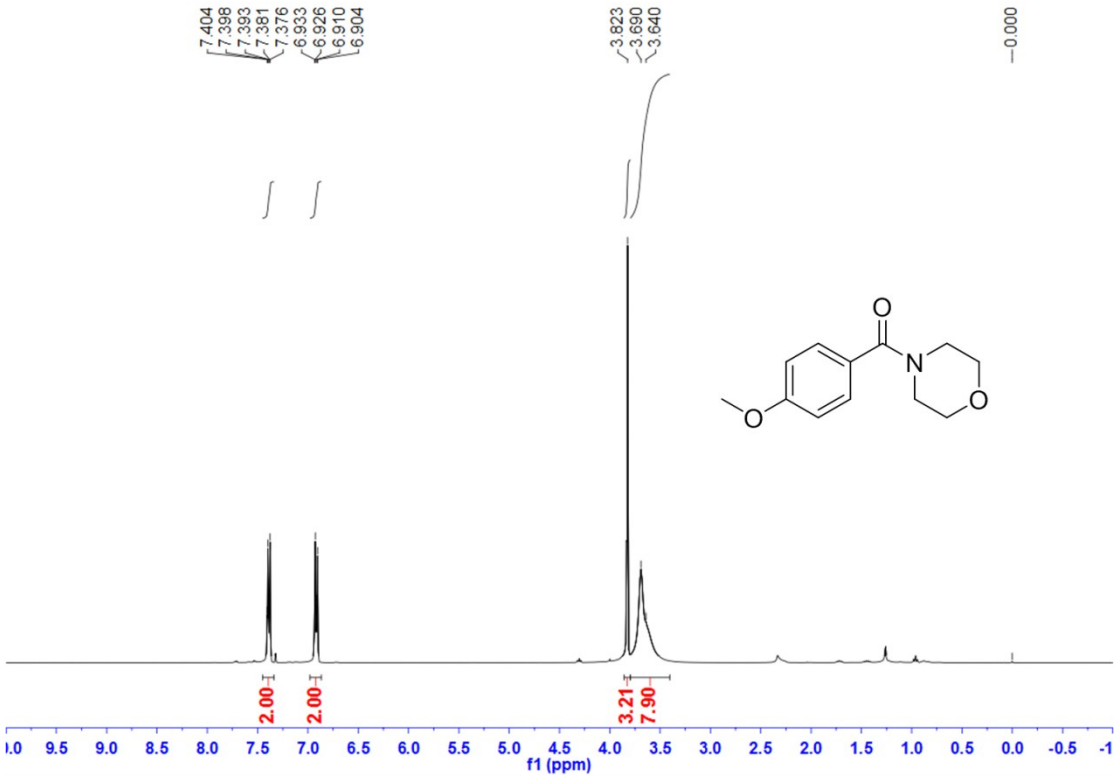
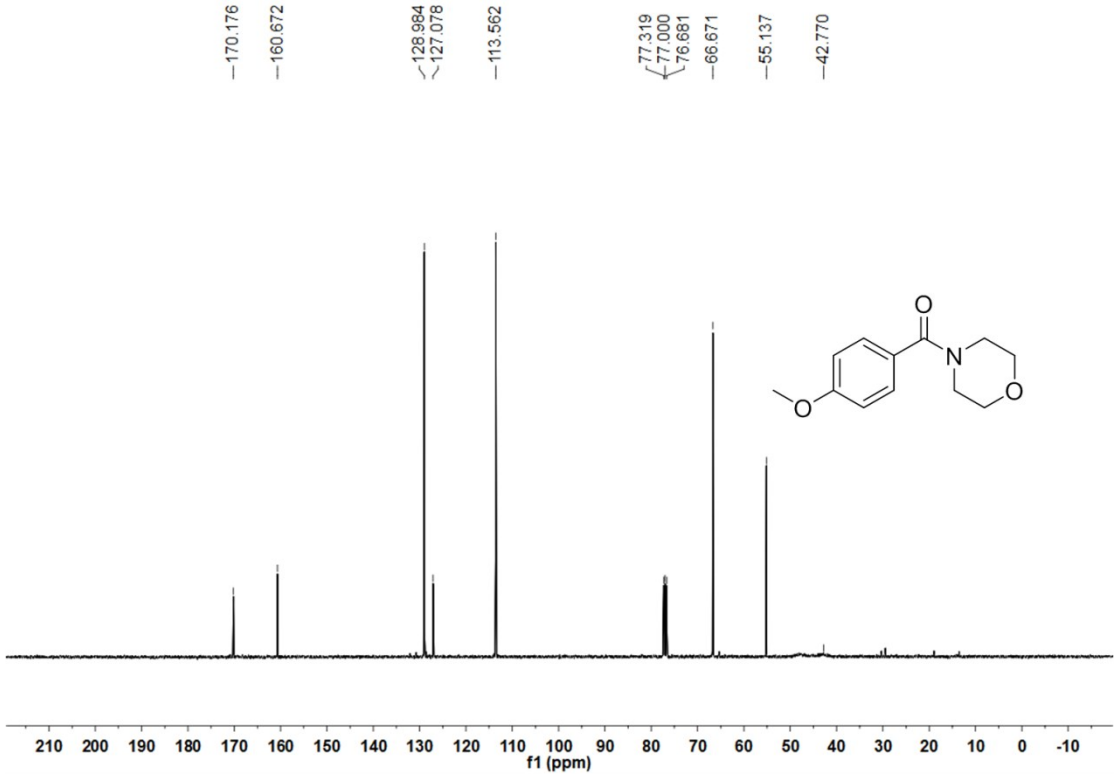
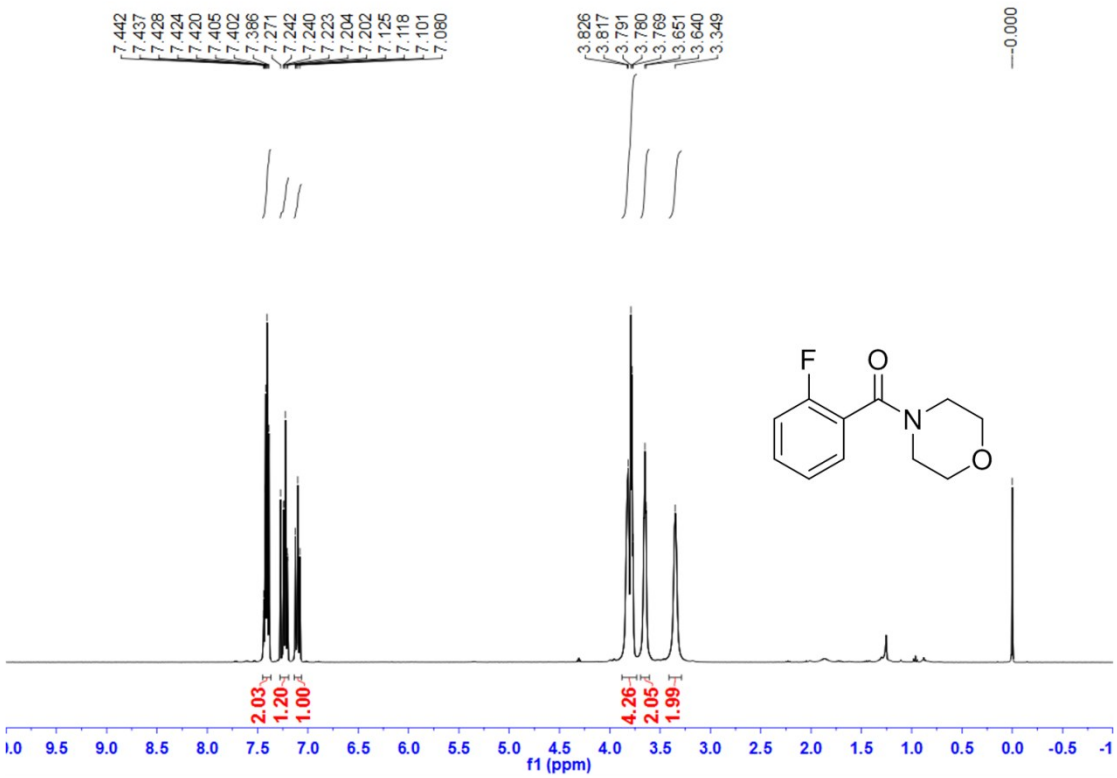
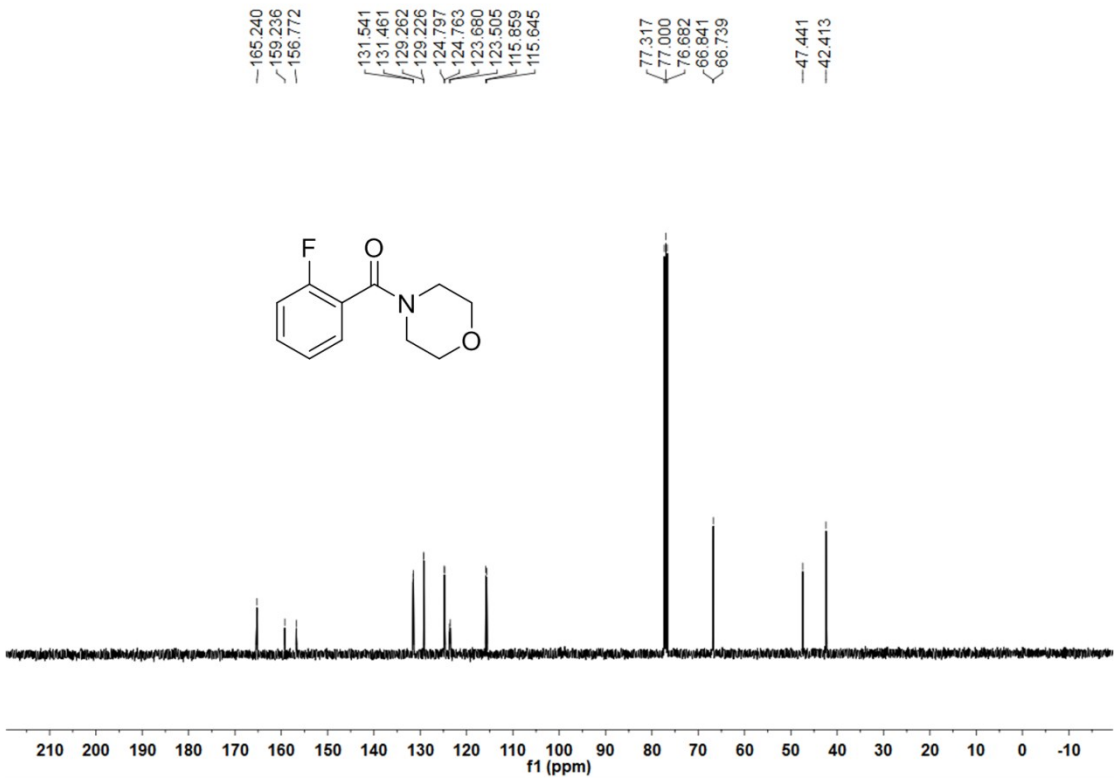
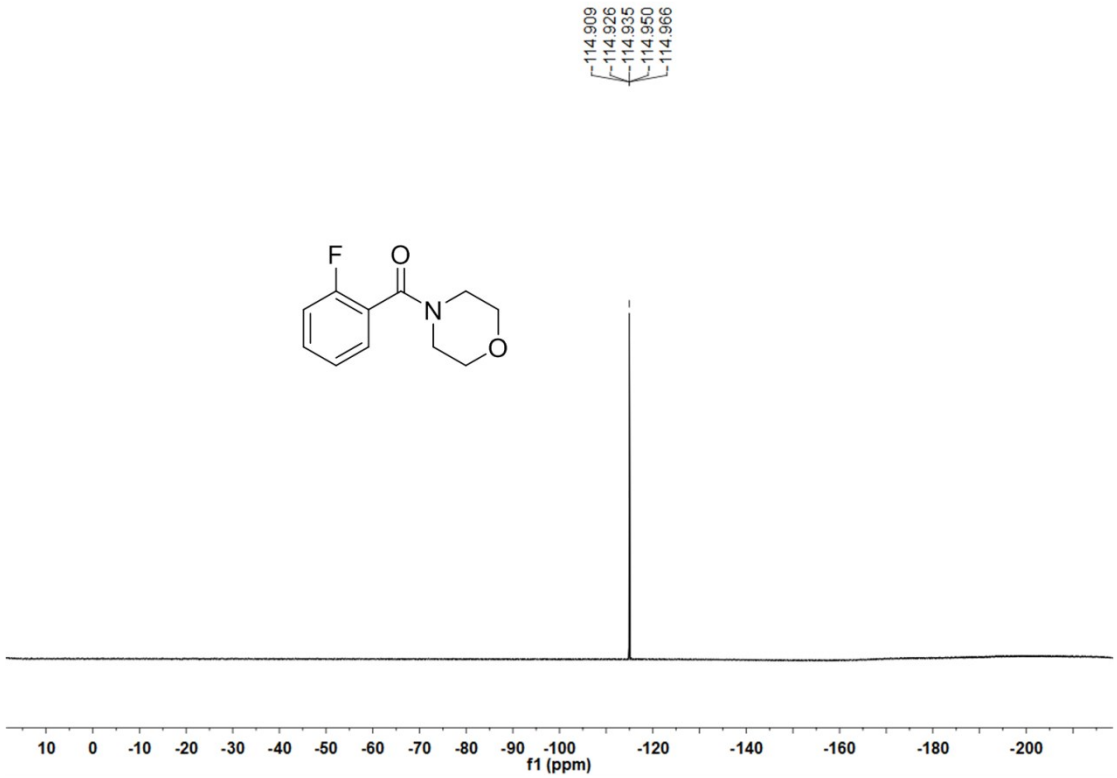
77.000
76.681
66.800
48.225
42.557
21.281
O
N
O
f1 (ppm)

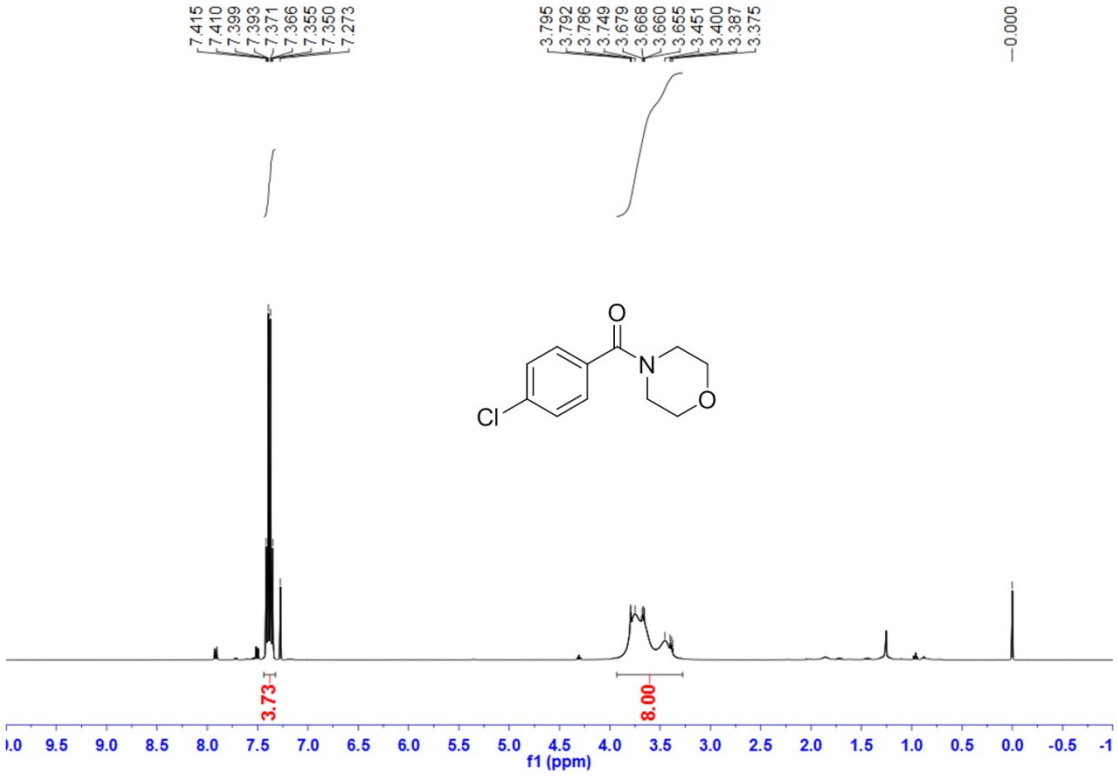

7.404
7.398
7.393
7.381
7.376
6.933
6.926
6.910
6.904
3.823
3.690
3.640
0.000
O
N
O
O
2.00
2.00
3.21
7.90
f1 (ppm)


170.176
160.672
128.984
127.078
113.562
77.319
77.000
76.681
66.671
55.137
42.770
O
N
O
O
f1 (ppm)


7.442
7.437
7.428
7.424
7.420
7.405
7.402
7.386
7.271
7.242
7.240
7.223
7.204
7.202
7.125
7.118
7.101
7.080
3.826
3.817
3.791
3.780
3.769
3.651
3.640
3.349
-0.000
F
O
N
O
2.03
1.20
1.00
4.26
2.05
1.99
f1 (ppm)


165.240
159.236
156.772
131.541
131.461
129.262
129.226
124.797
124.763
123.680
123.505
115.859
115.645
77.317
77.000
76.682
66.841
66.739
47.441
42.413
F
O
N
O
f1 (ppm)


114.909
114.926
114.935
114.950
114.966
F
O
N
O
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)


7.415
7.410
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7.393
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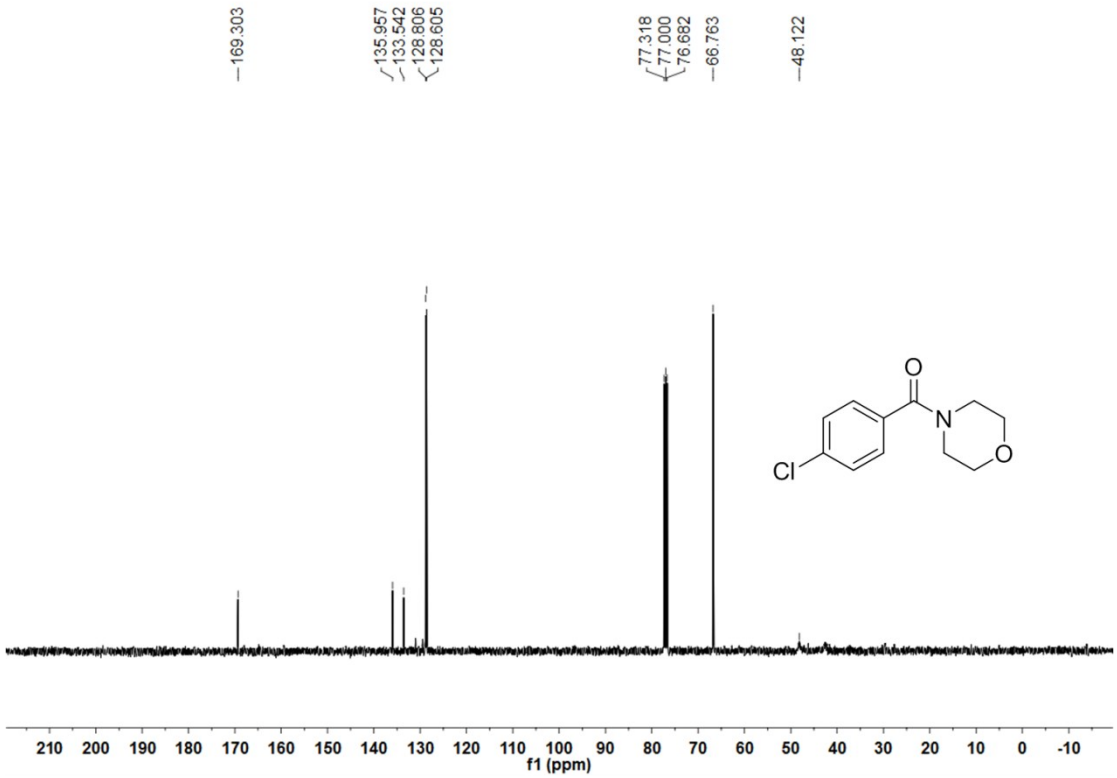
7.366
7.355
7.350
7.273
3.795
3.792
3.786
3.749
3.679
3.668
3.660
3.655
3.451
3.400
3.387
3.375
0.000
O
N
O
Cl
3.73
8.00
f1 (ppm)


169.303
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133.542
128.806
128.605
77.318
77.000
76.682
66.763
48.122
O
N
O
Cl
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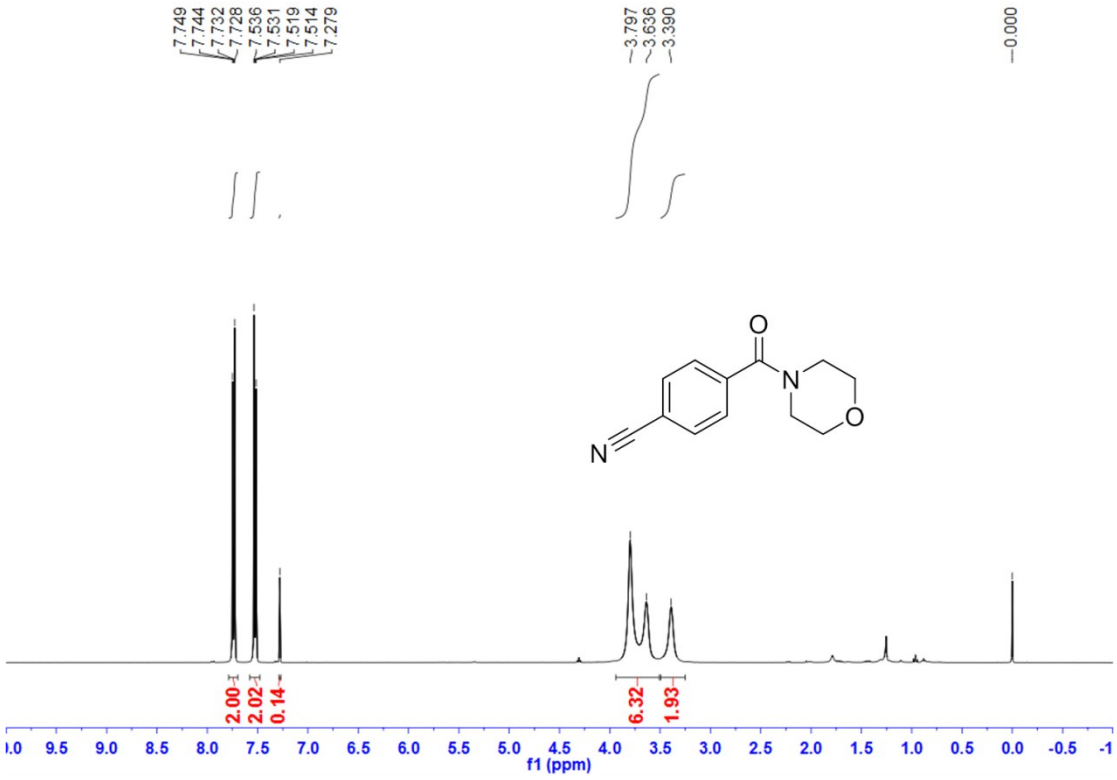
f1 (ppm)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7.744
7.732
7.728
7.536
7.531
7.519
7.514
7.279
3.797
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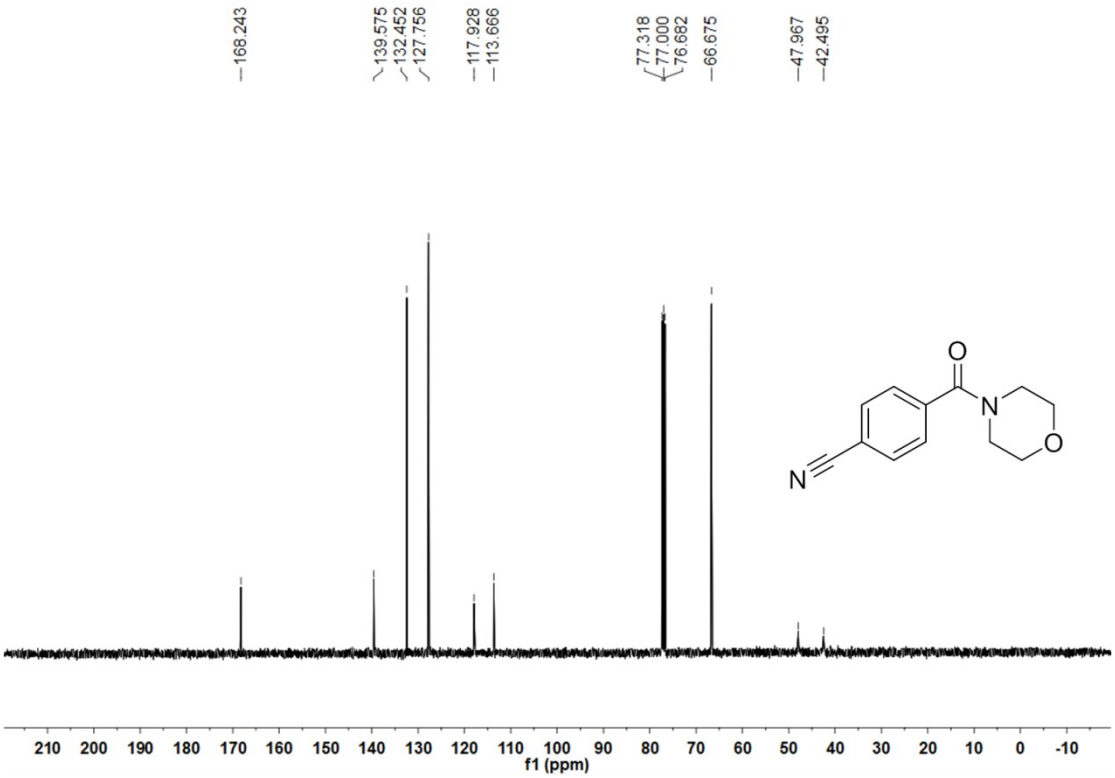
0.000
O
N
O
N
2.00
2.02
0.14
6.32
1.93
f1 (ppm)


168.243
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132.452
127.756
117.928
113.666
77.318
77.000
76.682
66.675
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42.495
O
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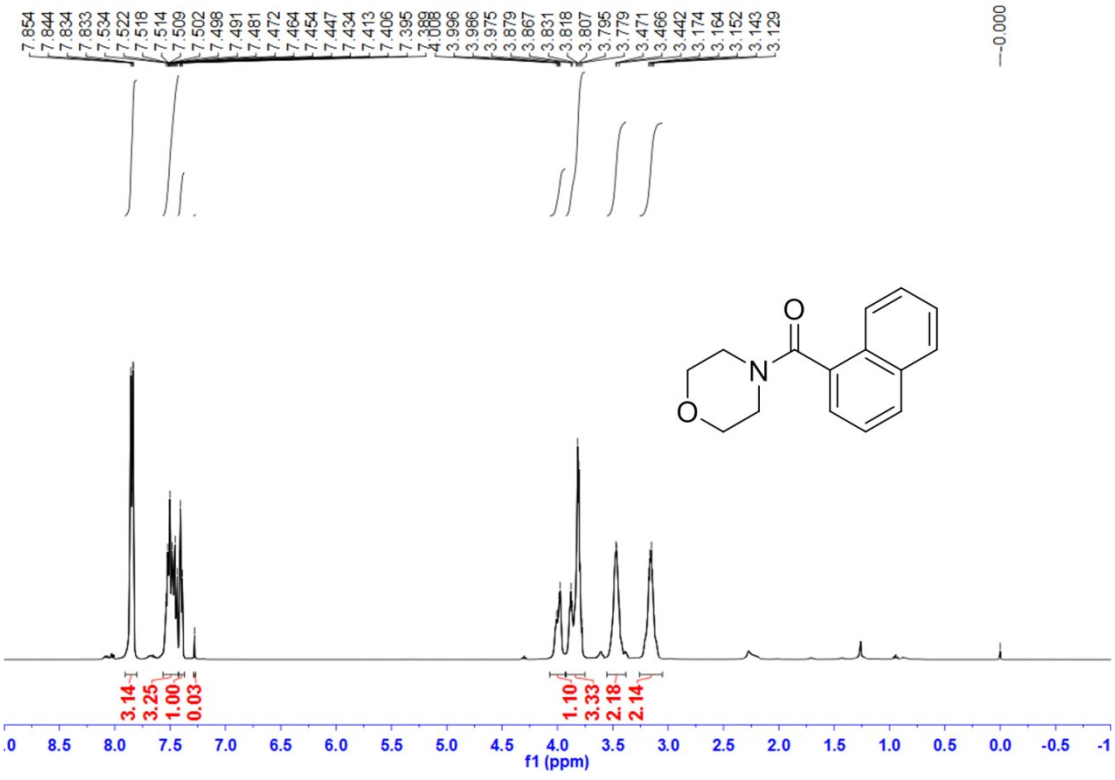
O
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.833
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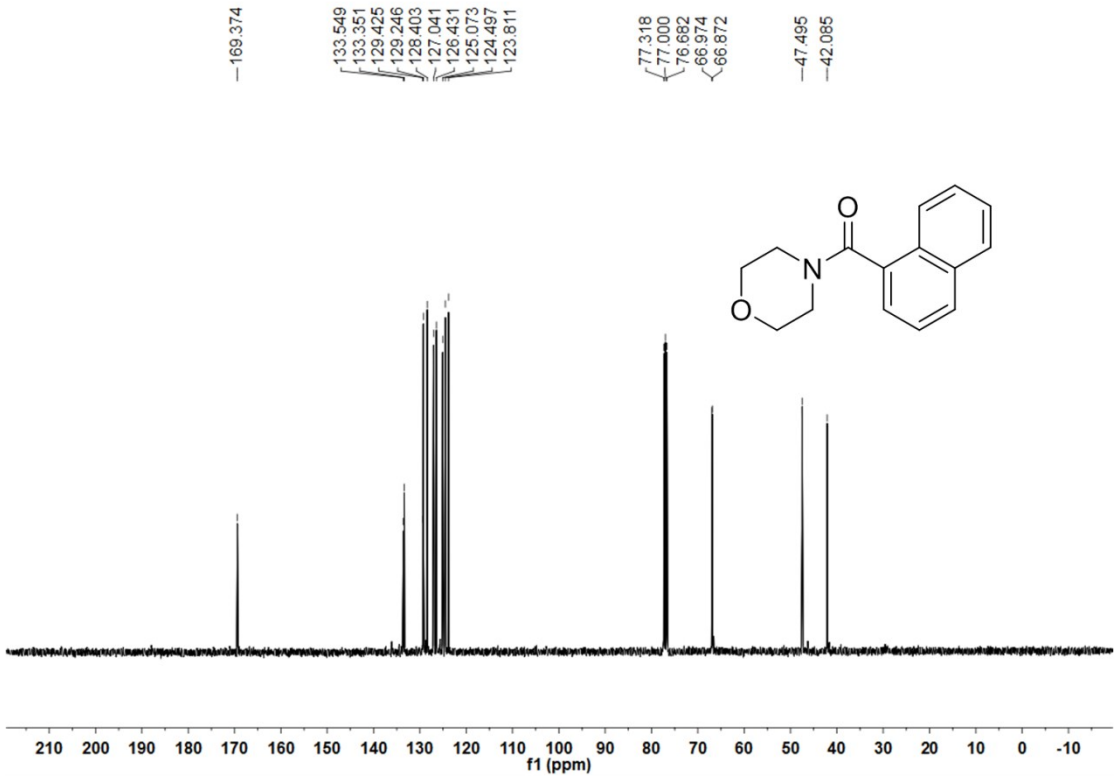
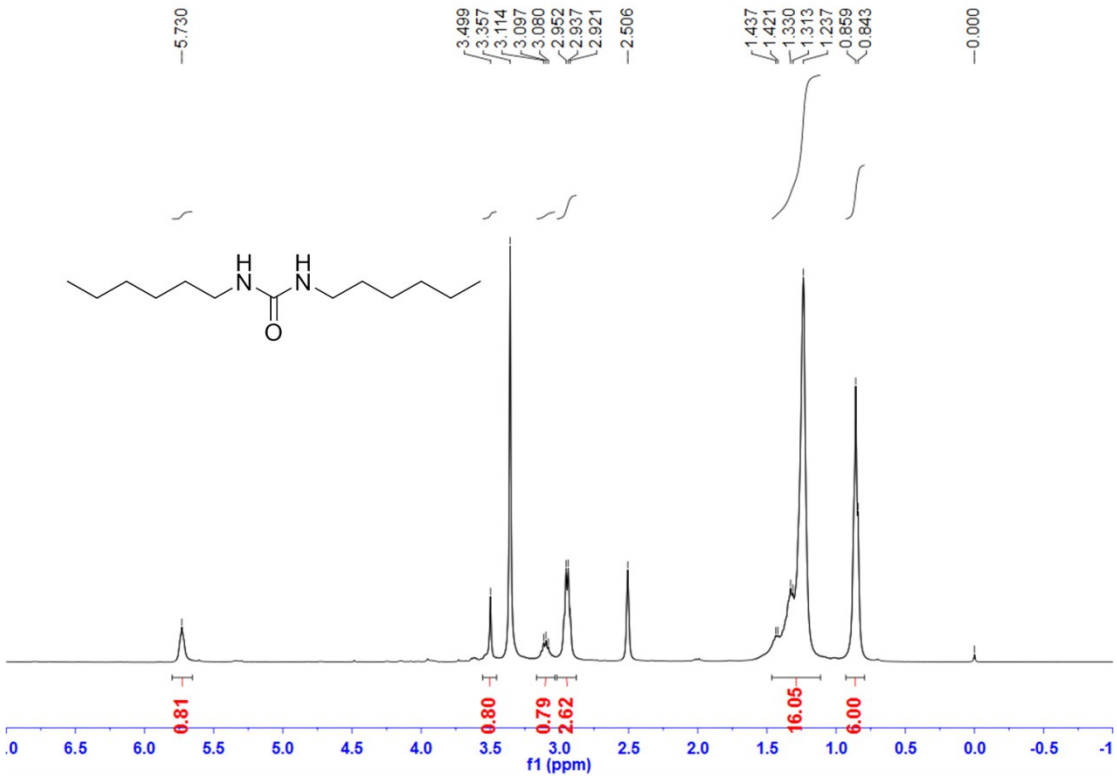
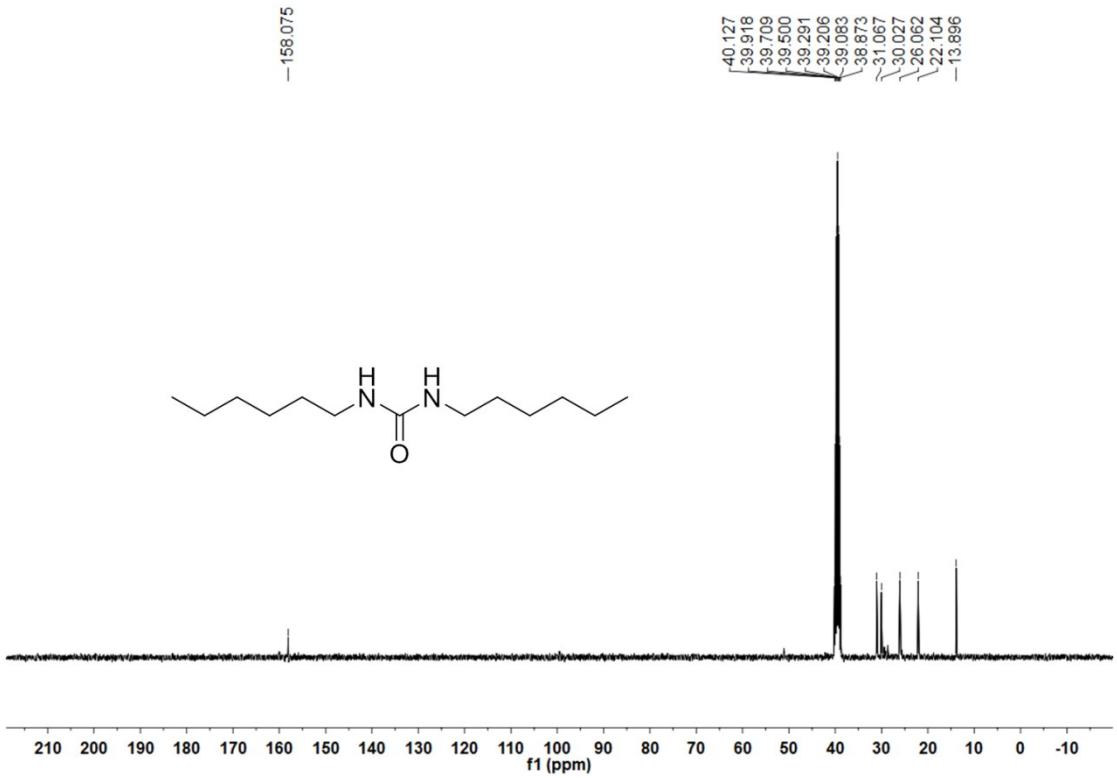
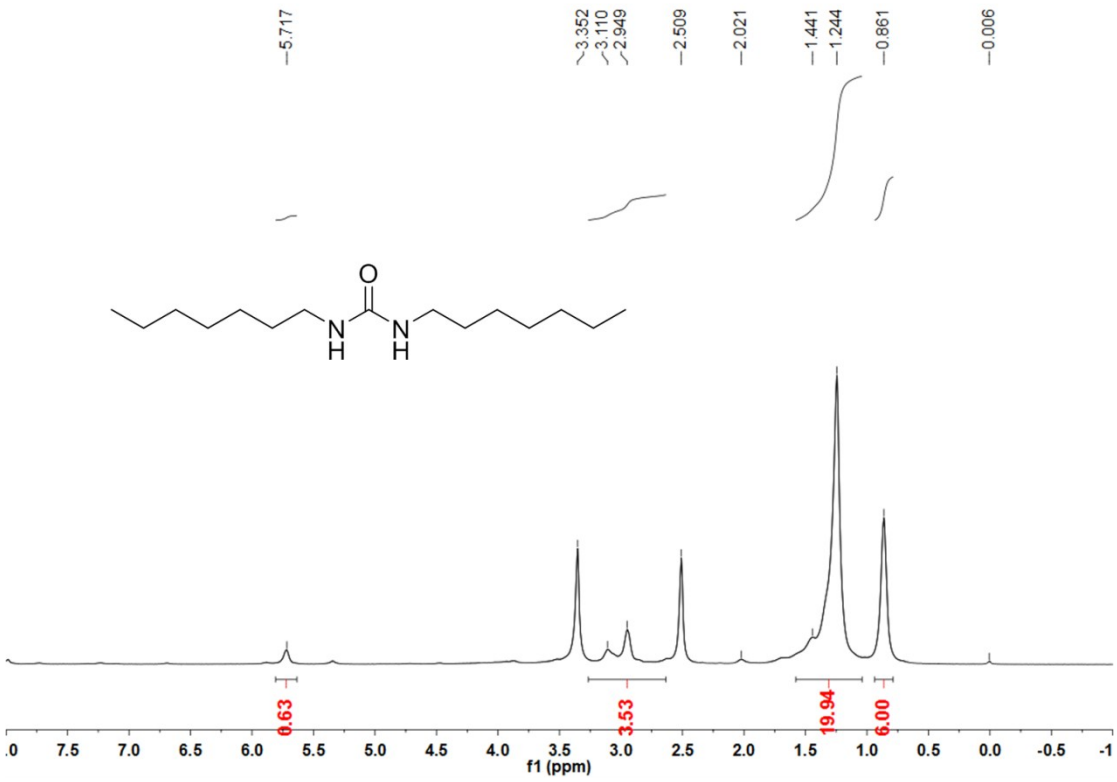
7.514
7.509
7.502
7.498
7.491
7.481
7.472
7.464
7.454
7.447
7.434
7.413
7.406
7.395
7.389
4.008
3.996
3.986
3.975
3.879
3.867
3.831
3.818
3.807
3.795
3.779
3.471
3.466
3.442
3.174
3.164
3.152
3.143
3.129
0.000
O
N
O
3.14
3.25
1.00
0.03
1.10
3.33
2.18
2.14
.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


169.374
133.549
133.351
129.425
129.246
128.403
127.041
126.431
125.073
124.497
123.811
77.318
77.000
76.682
66.974
66.872
47.495
42.085
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


5.730
3.499
3.357
3.114
3.097
3.080
2.952
2.937
2.921
2.506
1.437
1.421
1.330
1.313
1.237
0.859
0.843
0.000
H
N
H
N
O
0.81
0.80
0.79
2.62
16.05
6.00
.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


158.075
40.127
39.918
39.709
39.500
39.291
39.206
39.083
38.873
31.067
30.027
26.062
22.104
13.896
H
N
H
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


5.717
3.352
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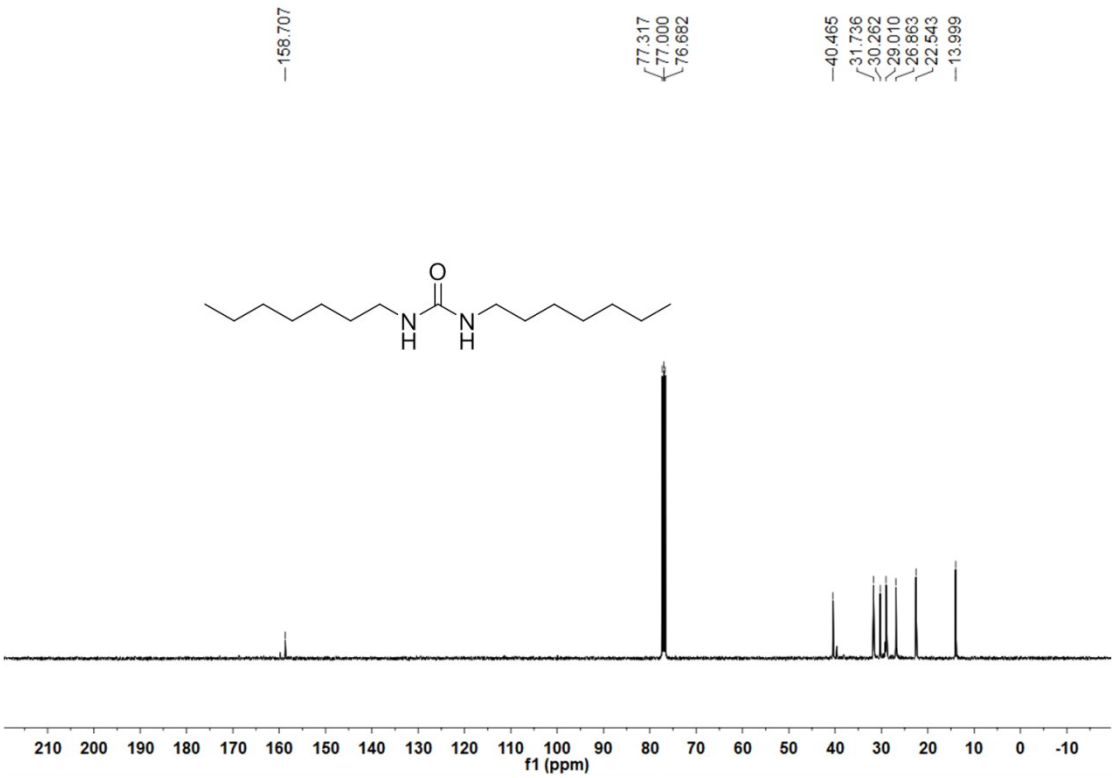
2.509
2.021
1.441
1.244
0.861
0.006
O
N
H
N
H
0.63
3.53
19.94
6.00
.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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31.736
30.262
29.010
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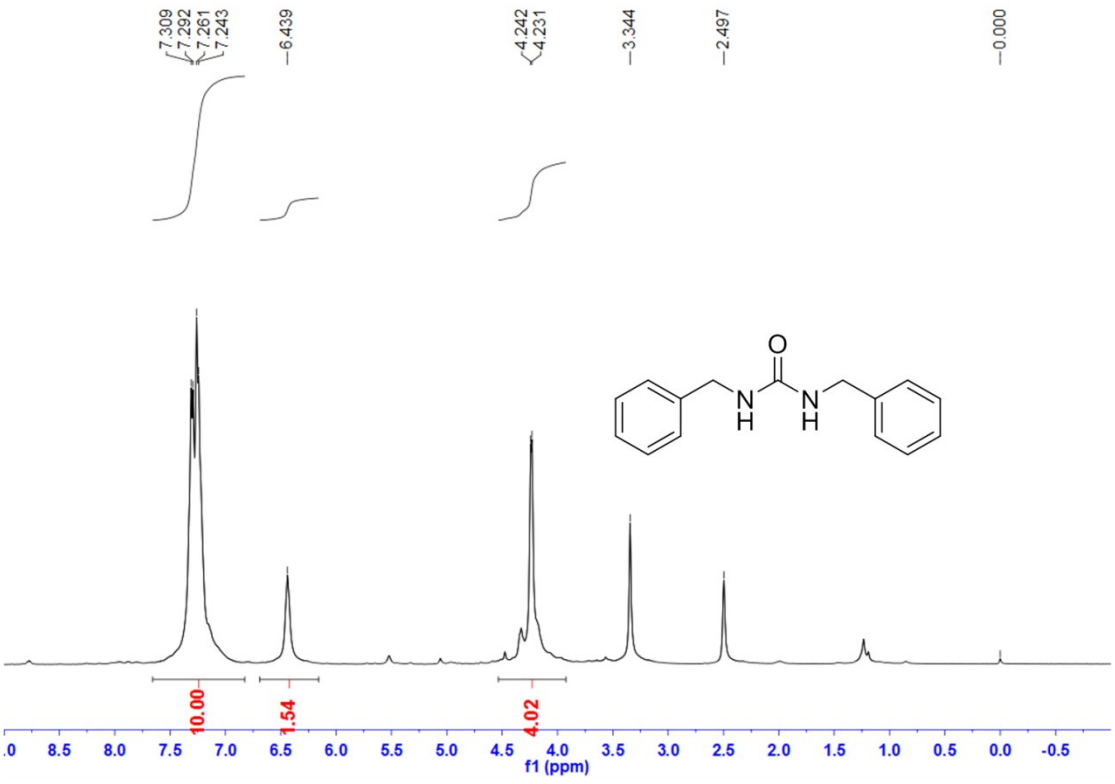
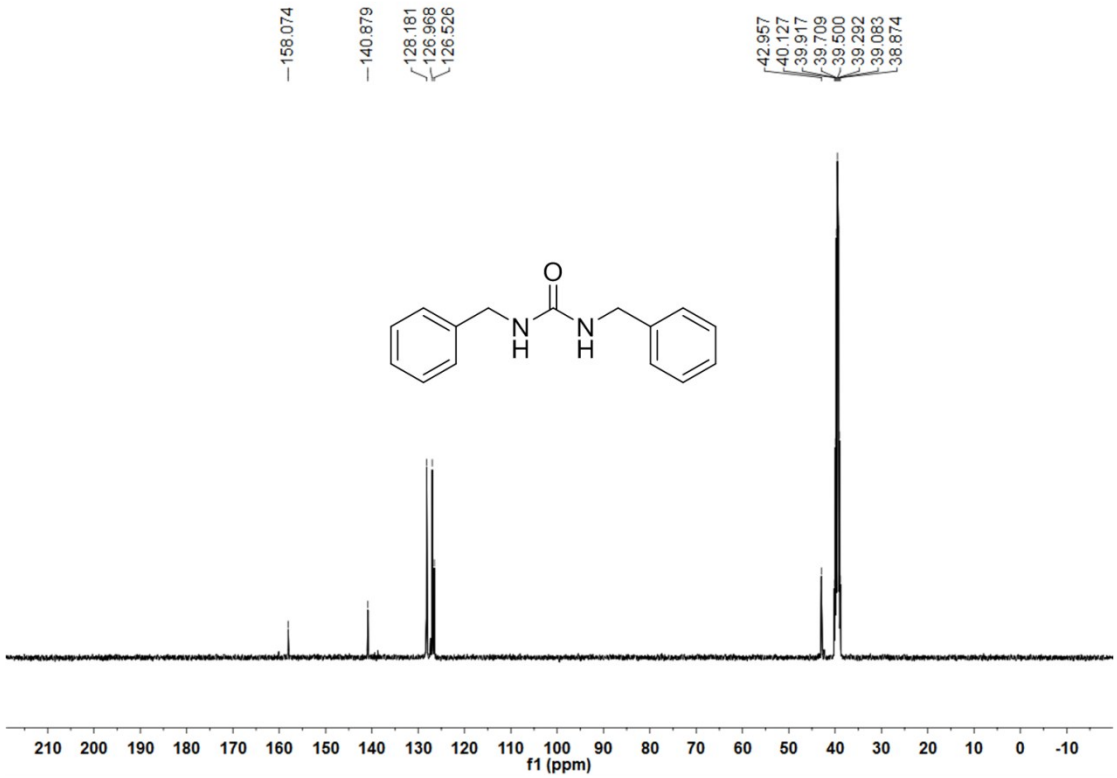
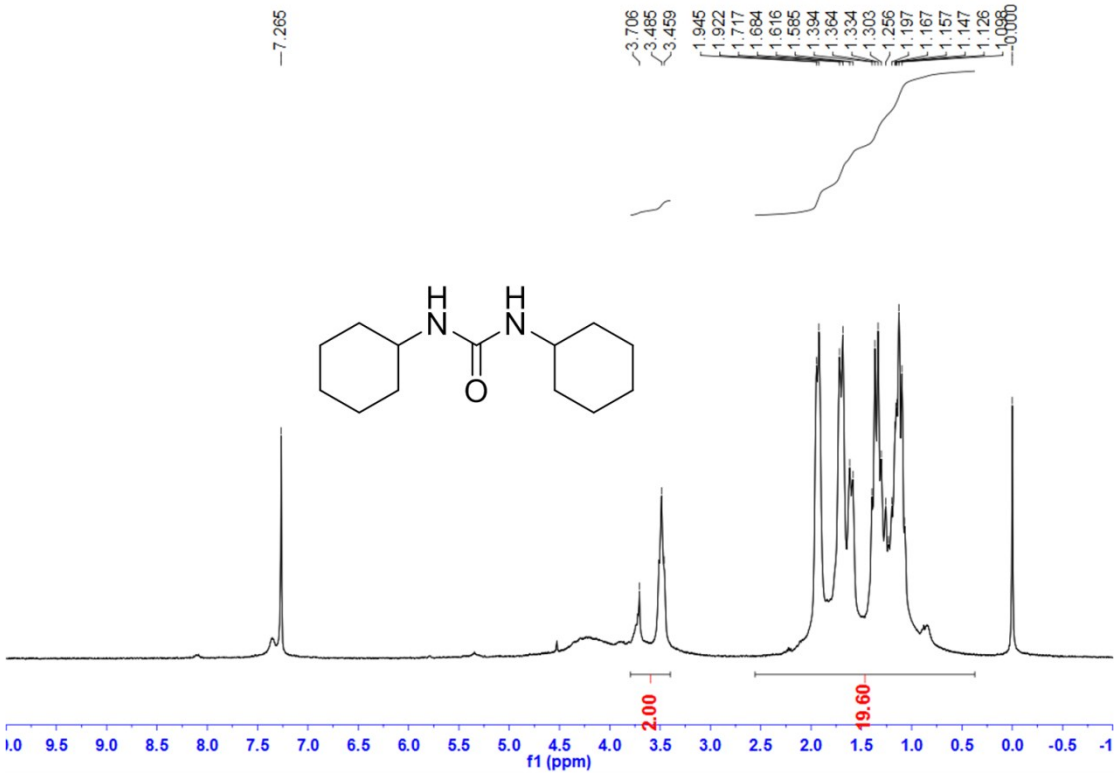
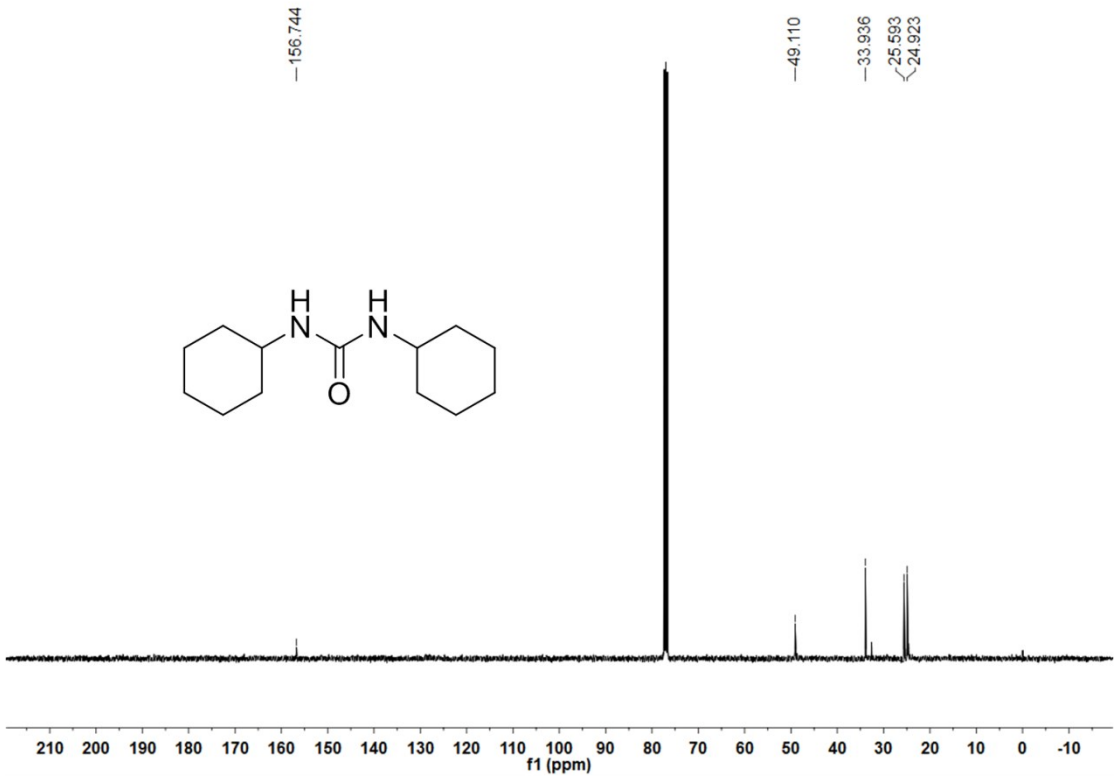
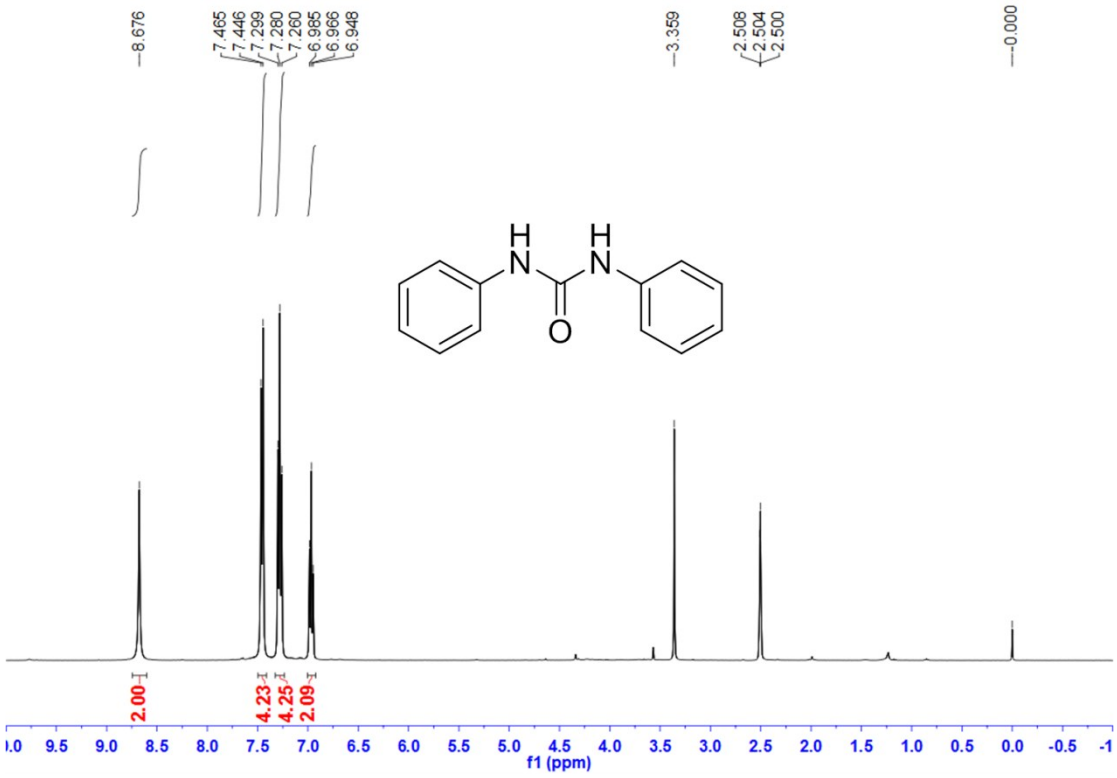
13.999
O
N
H
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

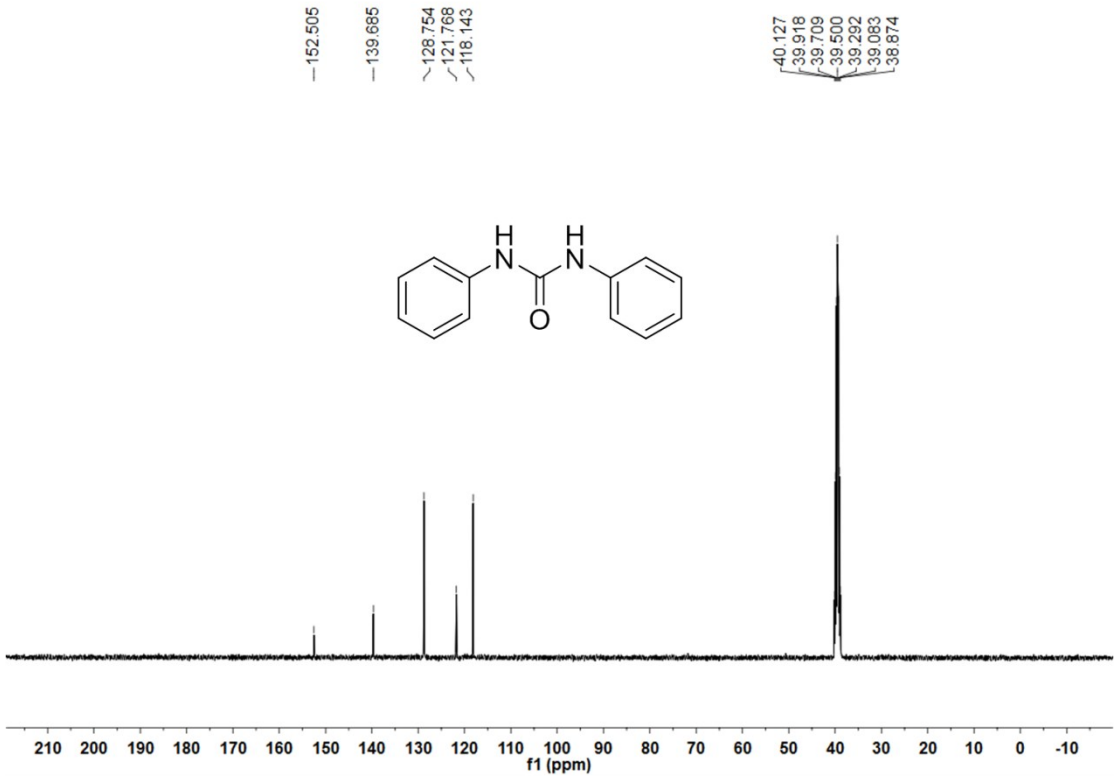

7.309
7.292
7.261
7.243
6.439
4.242
4.231
3.344
2.497
0.000
O
N
H
N
H
10.00
1.54
4.02
.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

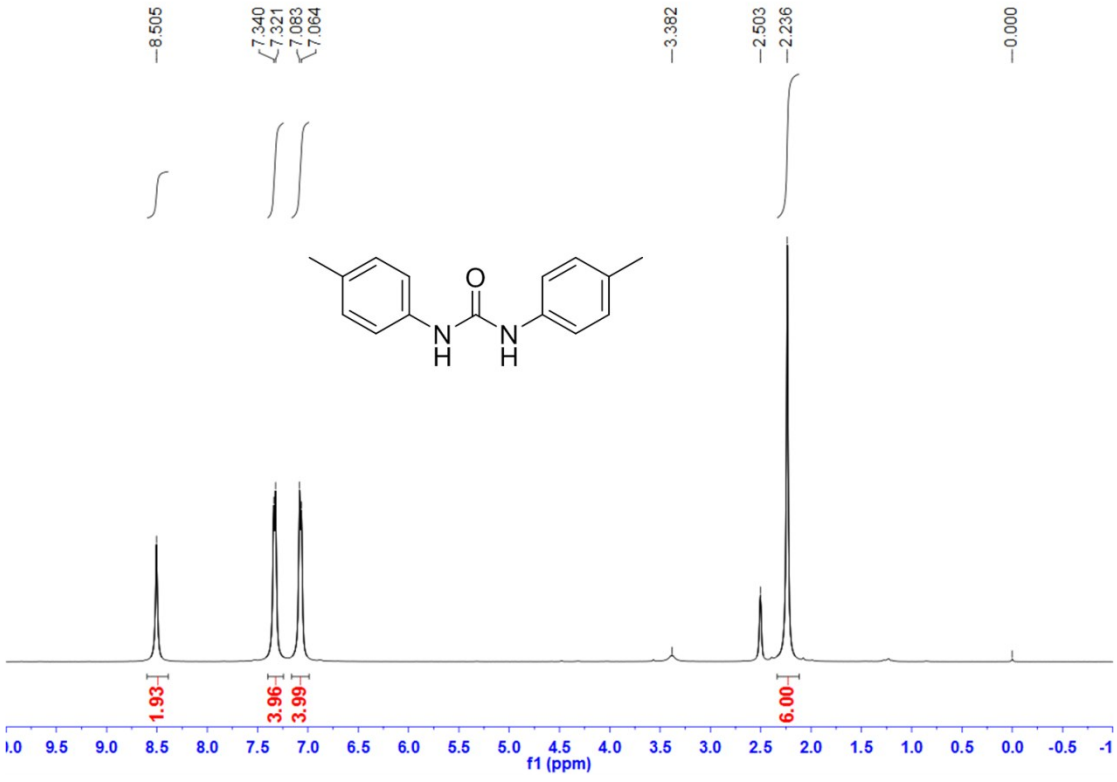

158.074
140.879
128.181
126.968
126.526
42.957
40.127
39.917
39.709
39.500
39.292
39.083
38.874
O
N
H
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

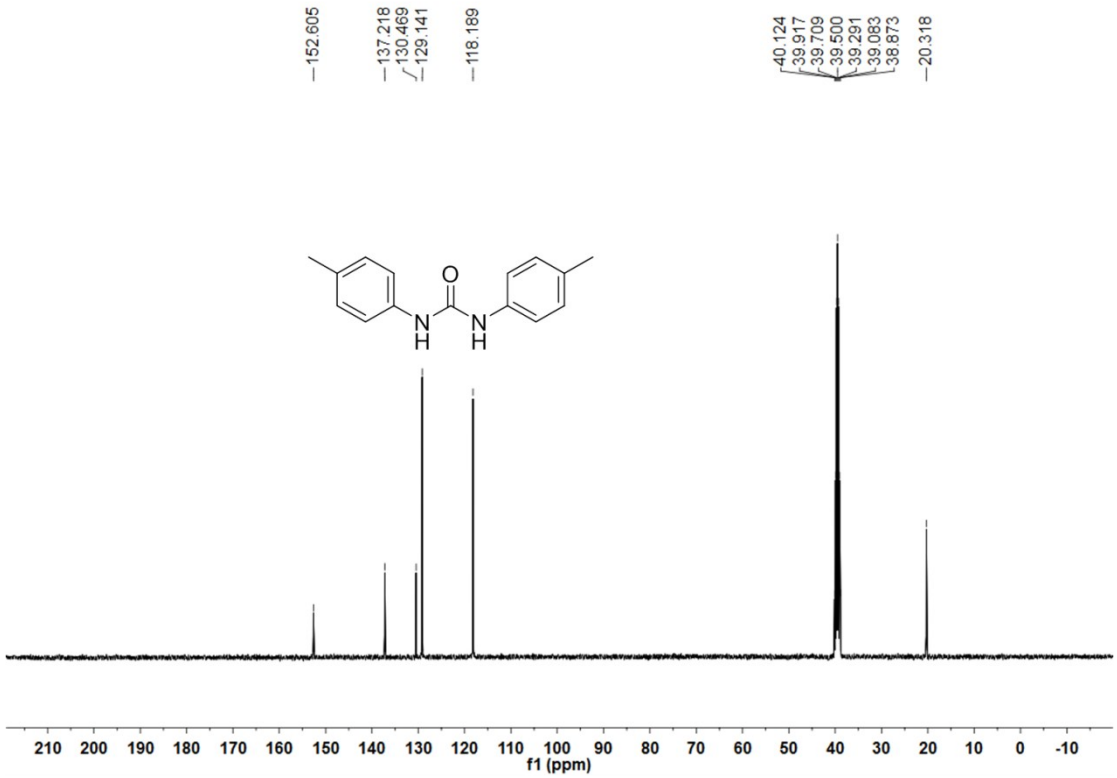

7.265
3.706
3.485
3.459
1.945
1.922
1.717
1.684
1.616
1.585
1.394
1.364
1.334
1.303
1.256
1.197
1.167
1.157
1.147
1.126
1.098
0.000
H
N
H
N
O
2.00
19.60
f1 (ppm)

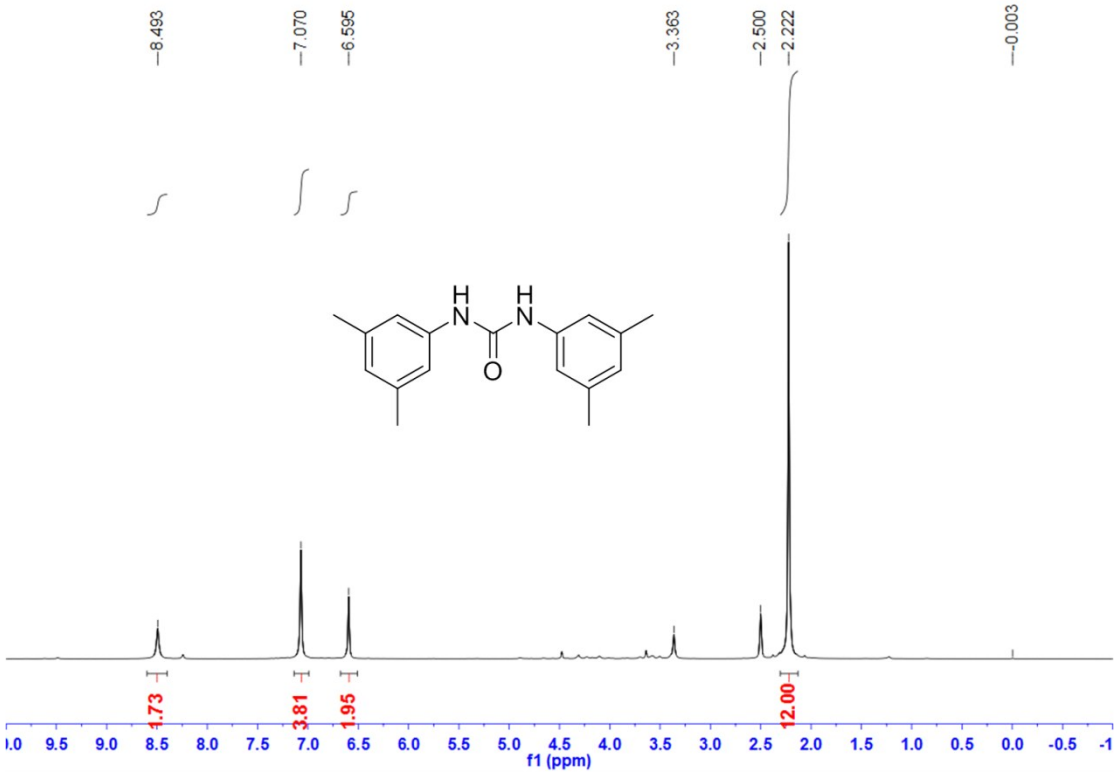

156.744
49.110
33.936
25.593
24.923
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

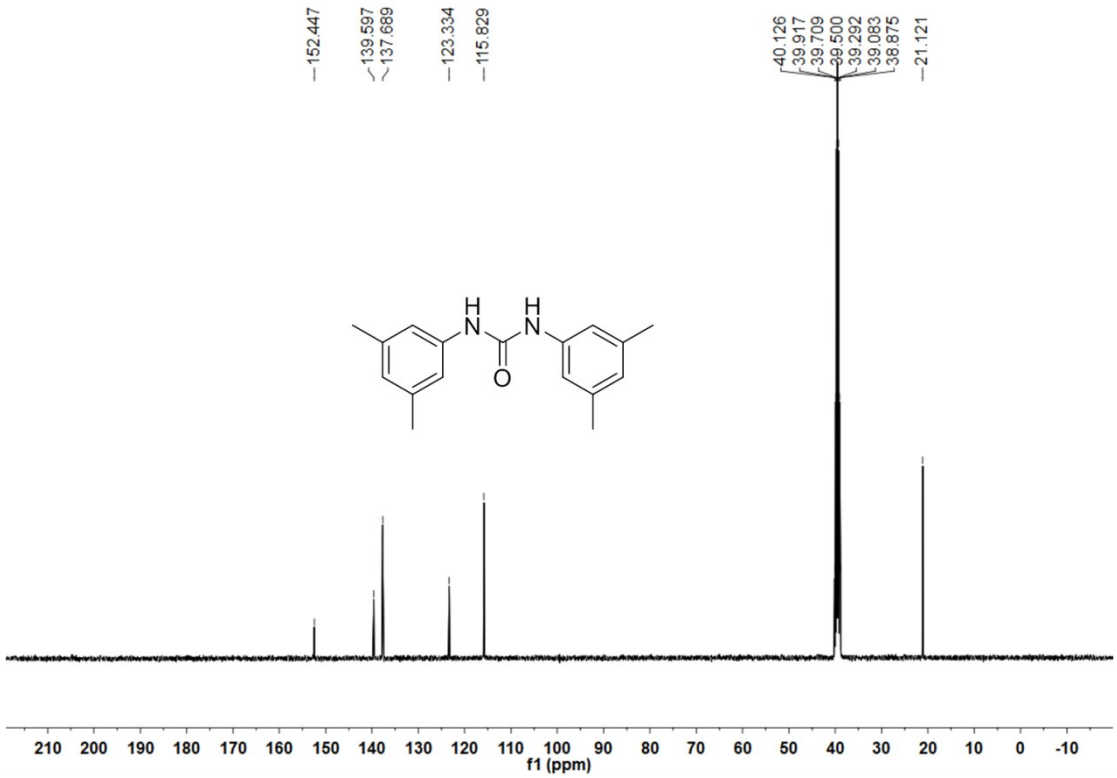

8.676
7.465
7.446
7.299
7.280
7.260
6.985
6.966
6.948
3.359
2.508
2.504
2.500
0.000
2.00
4.23
4.25
2.09
f1 (ppm)

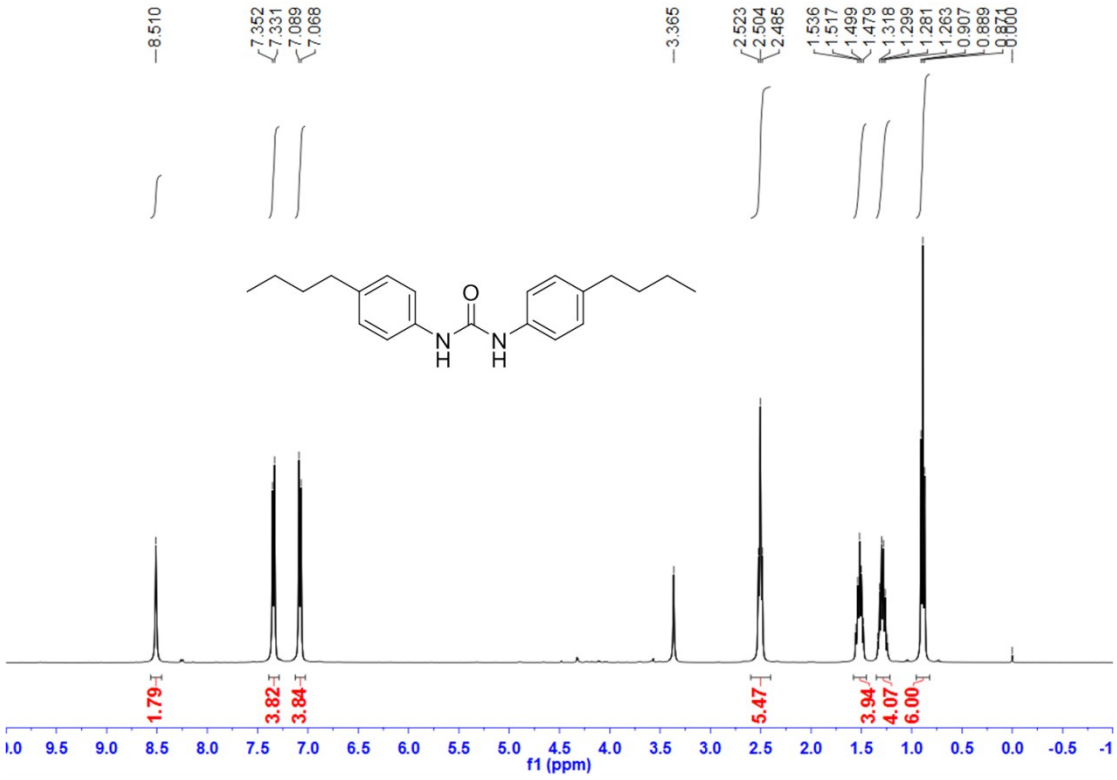

152.505
139.685
128.754
121.768
118.143
40.127
39.918
39.709
39.500
39.292
39.083
38.874
H
N
H
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

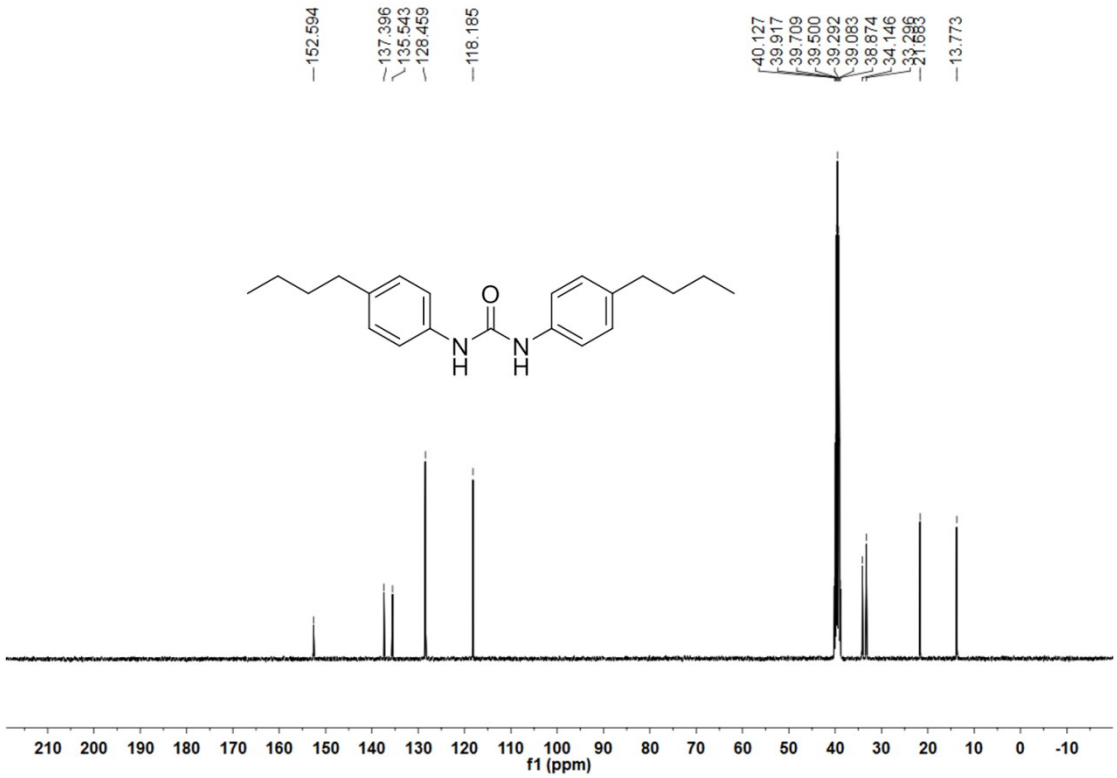

8.505
7.340
7.321
7.083
7.064
3.382
2.503
2.236
0.000
O
N
H
N
H
1.93
3.96
3.99
6.00
f1 (ppm)

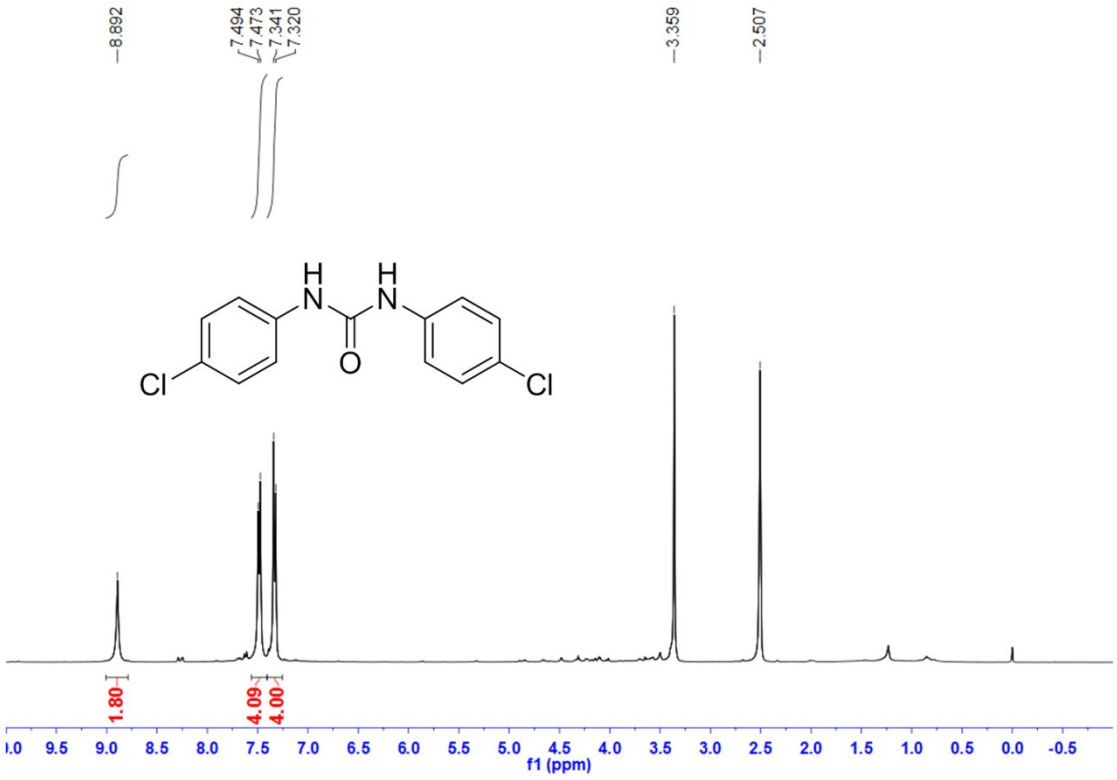

152.605
137.218
130.469
129.141
118.189
40.124
39.917
39.709
39.500
39.291
39.083
38.873
20.318
O
N
H
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

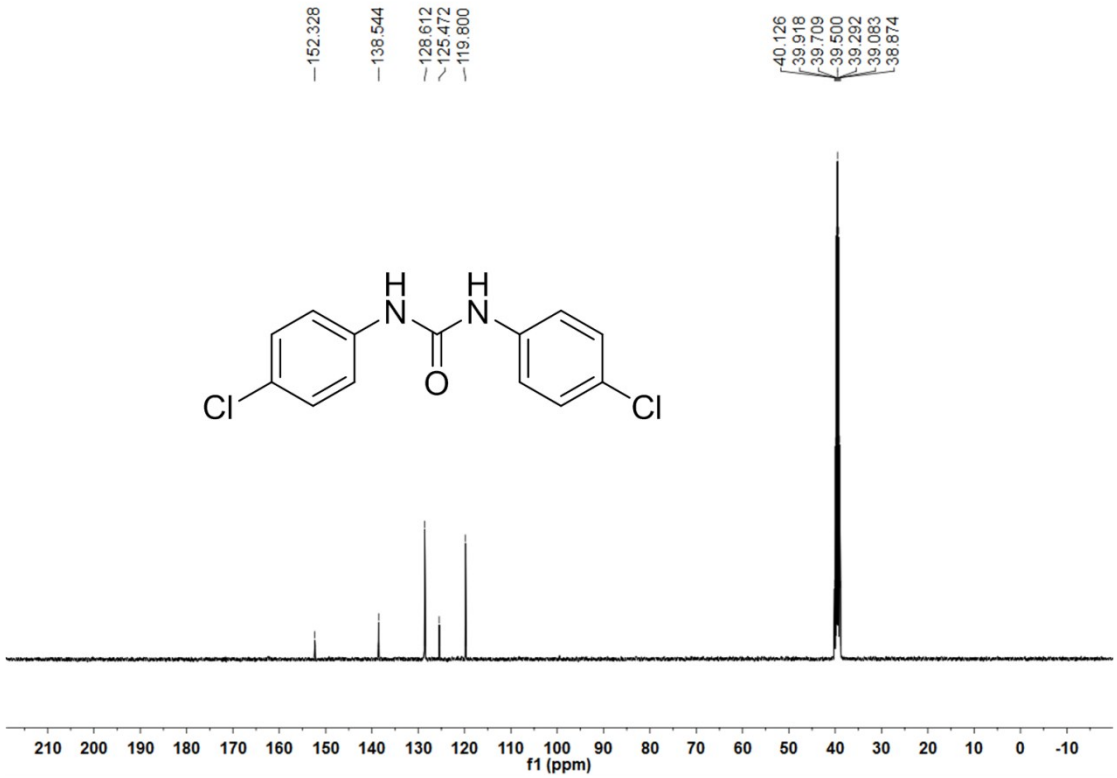

8.493
7.070
6.595
3.363
2.500
2.222
0.003
H
N
H
N
O
1.73
3.81
1.95
12.00
1.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

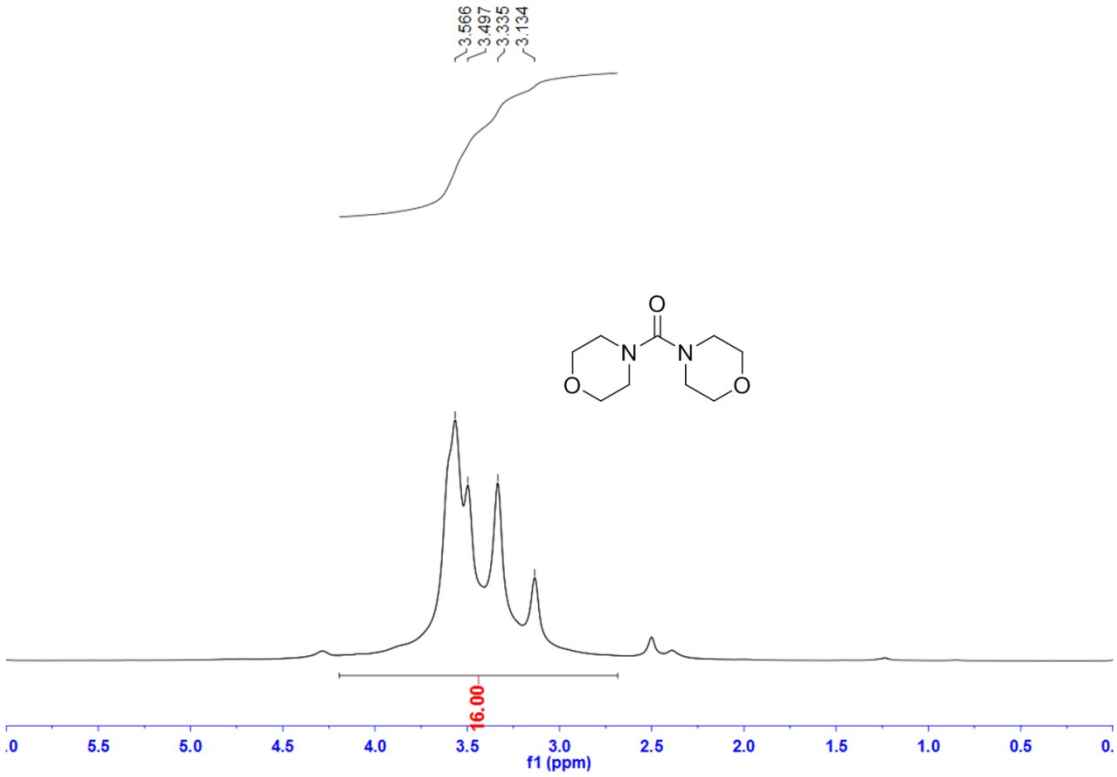

152.447
139.597
137.689
123.334
115.829
40.126
39.917
39.709
39.500
39.292
39.083
38.875
21.121
H
N
H
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

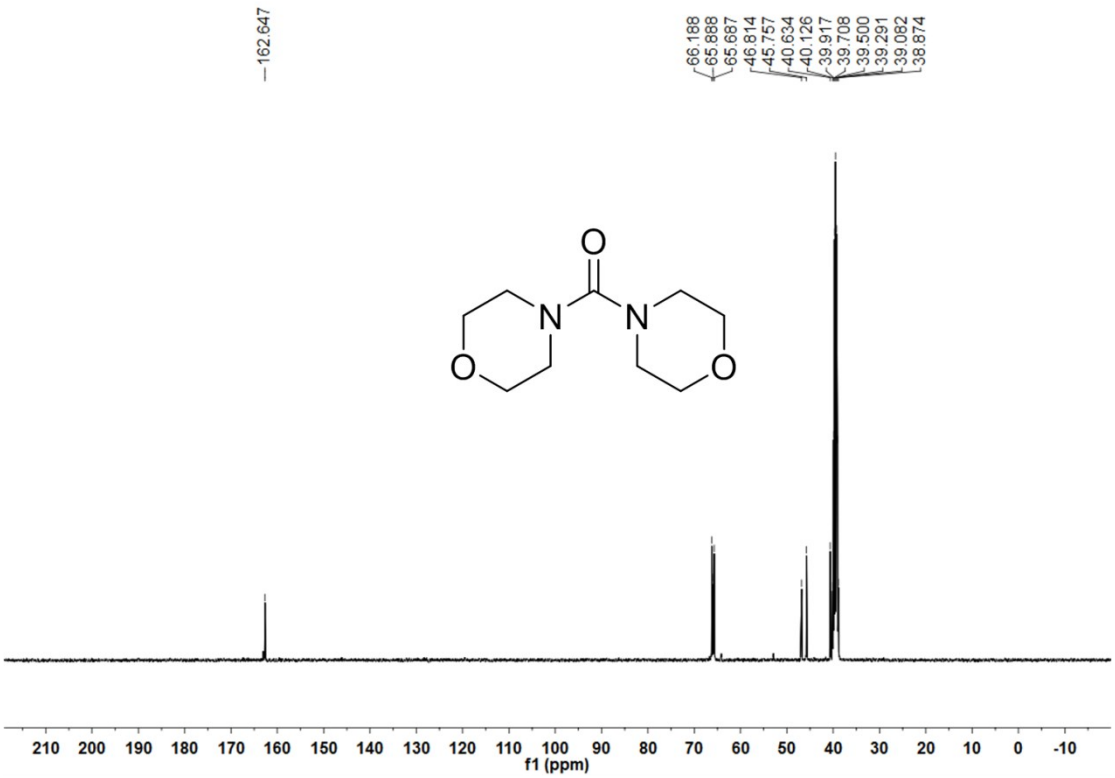

8.510
7.352
7.331
7.089
7.068
3.365
2.523
2.504
2.485
1.536
1.517
1.499
1.479
1.318
1.299
1.281
1.263
0.907
0.889
0.871
0.853
O
N
H
N
H
1.79
3.82
3.84
5.47
3.94
4.07
6.00
1.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

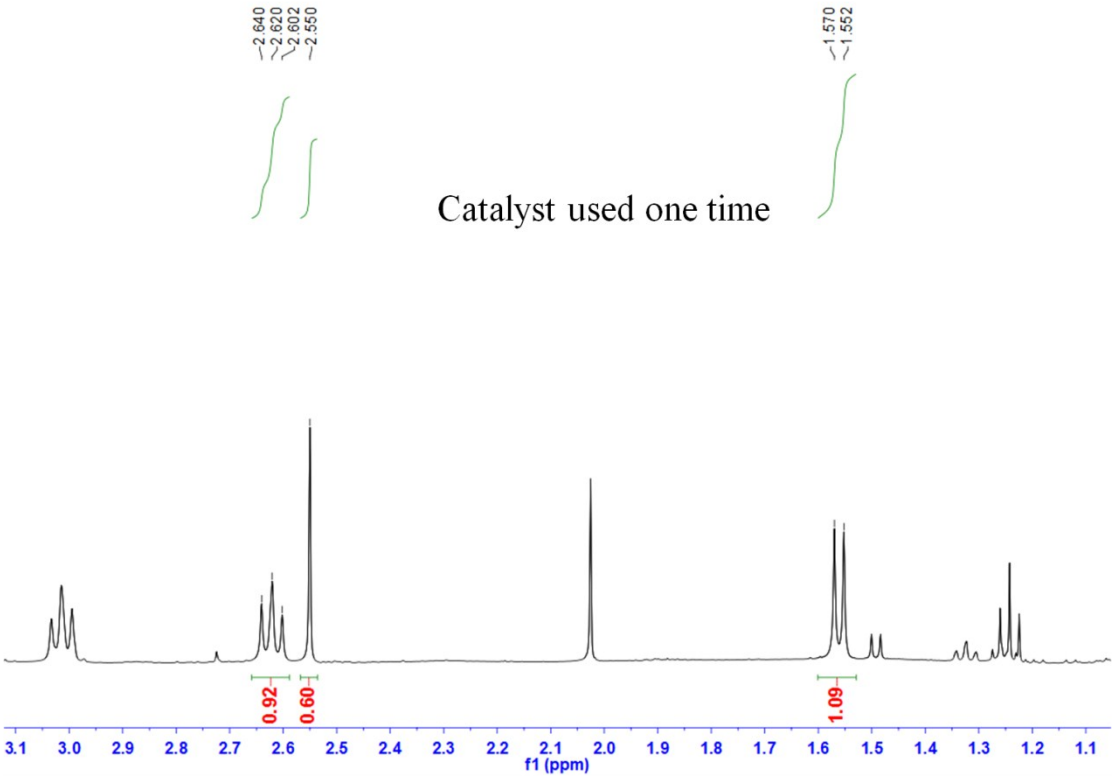

152.594
137.396
135.543
128.459
118.185
40.127
39.917
39.709
39.500
39.292
39.083
38.874
34.146
33.296
21.683
13.773
O
N
H
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

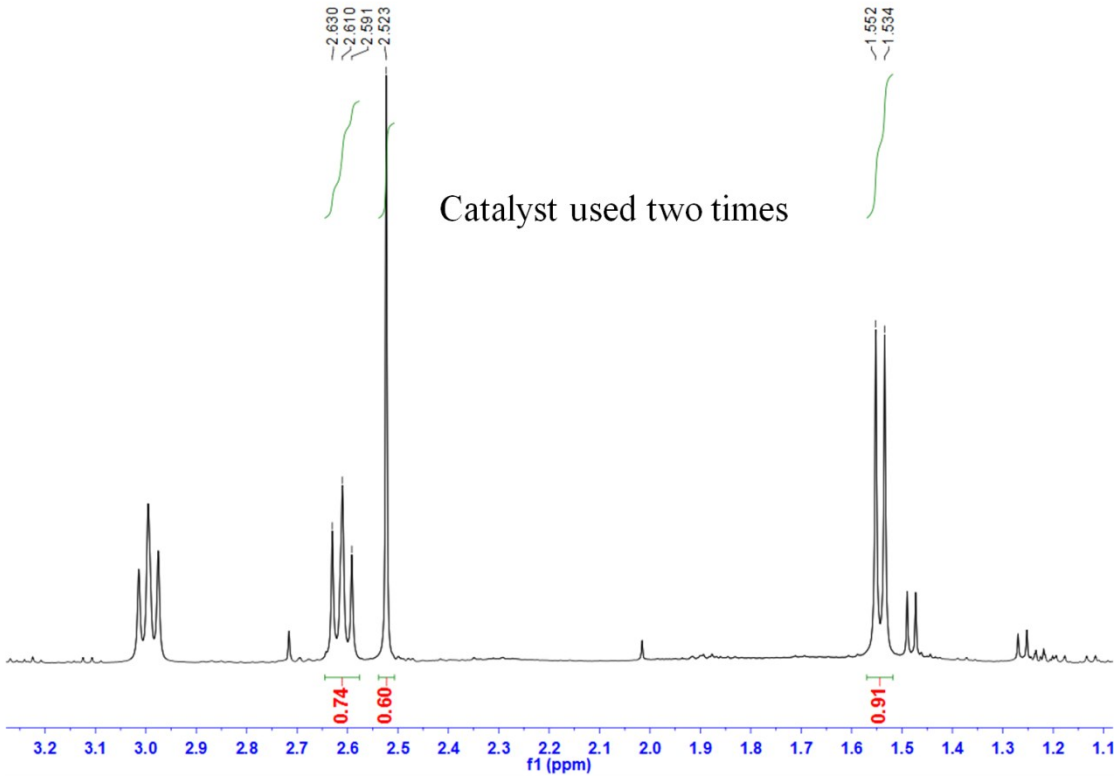

8.892
7.494
7.473
7.341
7.320
3.359
2.507
H
N
H
N
O
Cl
Cl
1.80
4.09
4.00
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

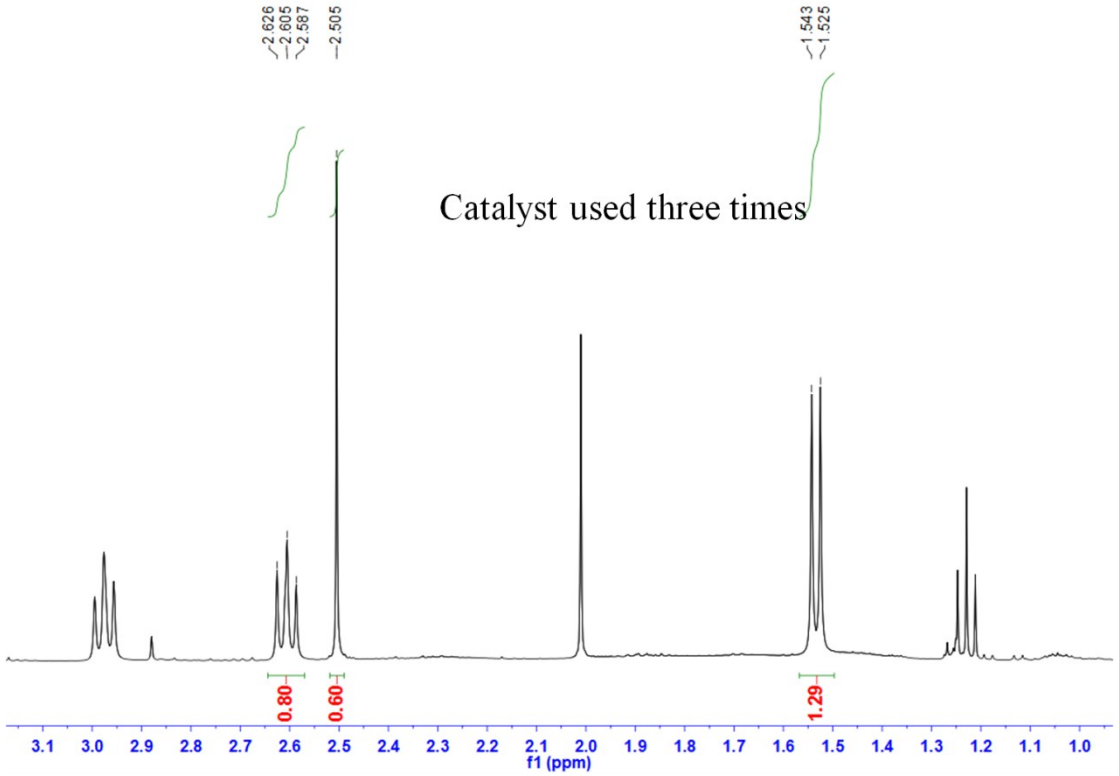

152.328
138.544
128.612
125.472
119.800
40.126
39.918
39.709
39.500
39.292
39.083
38.874
H
N
H
N
O
Cl
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


3.566
3.497
3.335
3.134
O
N
N
O
O
16.00
.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)


162.647
66.188
65.888
65.687
46.814
45.757
40.634
40.126
39.917
39.708
39.500
39.291
39.082
38.874
O
N
N
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


2.640
2.620
2.602
2.550
1.570
1.552
Catalyst used one time
0.92
0.60
1.09
3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1
f1 (ppm)


2.630
2.610
2.591
2.523
1.552
1.534
Catalyst used two times
0.74
0.60
0.91
3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1
f1 (ppm)


2.626
2.605
2.587
2.505
1.543
1.525
Catalyst used three times
0.80
0.60
1.29
3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0
f1 (ppm)

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7. Author contributions

S. L. developed the reactions and expanded the scope. H. W. conceived and prepared the manuscript. X. D. tested the Pd dispersion of bulk Pd catalyst and discussed the results. F.S. conceived and supervised the project as well as revised the manuscript.