Supporting Information

Chromium-Catalyzed Activation of Acyl C–O Bonds with Magnesium for Amidation of Esters with Nitroarenes

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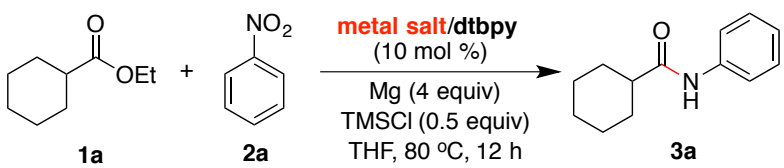
1. Materials and Methods

General. All reactions dealing with air- or moisture-sensitive compounds were carried out in a flame-dried, sealed Schlenk reaction tube under an atmosphere of nitrogen. Analytical thin-layer chromatography was performed on glass plates coated with 0.25 mm 230–400 mesh silica gel containing a fluorescent indicator (Merck). Flash silica gel column chromatography was performed on silica gel 60N (spherical and neutral, 140–325 mesh) as described by Still.[1] NMR spectra were measured on a Bruker AV-400 spectrometer and reported in parts per million. ^{1}H NMR spectra were recorded at 400 MHz in $CDCl_3$ were referenced internally to tetramethylsilane as a standard, and ^{13}C NMR spectra were recorded at 100 MHz and referenced to the solvent resonance. Analytical gas chromatography (GC) was carried out on a Thermo Trace 1300 gas chromatograph, equipped with a flame ionization detector. Mass spectra (GC-MS) were taken at Thermo Trace 1300 gas chromatograph mass spectrometer. High resolution mass spectra (HRMS) were recorded on the Exactive Mass Spectrometer (Thermo Scientific, USA) equipped with ESI ionization source. Melting points were determined with a Hanon MP-300. Electron paramagnetic resonance (EPR) spectrum was recorded on an instrument of Bruker A300-9.5/12.

Materials. Unless otherwise noted, materials were purchased from Tokyo Chemical Industry Co., Aldrich Inc., Alfa Aesar, Adamas, and other commercial suppliers and used as received. Solvents were dried over sodium (for THF and ether) by refluxing for overnight and freshly distilled prior to use. $CrCl_2$ (99.99%), $CrCl_3$ (99.99%), $CoCl_2$ (99.9%), $FeCl_2$ (98%) and Mg (99.99 %) were purchased from Aldrich Inc. and used as received. $Cr(acac)_3$ (97%) and $NiCl_2$ were purchased from Alfa Aesar and used as received.

2. Optimizing Reaction Parameters

Table S1. **Investigation of the Effect of First-Row Transition Metal Salts on the Amidation of Ester**[a]



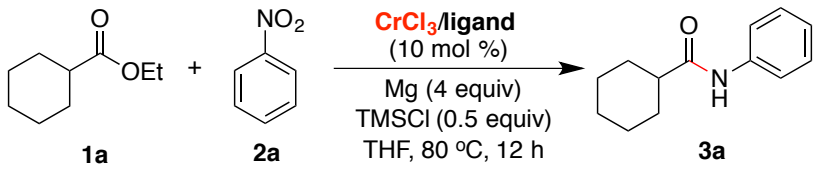
1a **2a** **3a**

Entry	Metal salt	Yield (**3a**)

1	-	19%
2	$CrCl_2$	59%
3	$CrCl_3$	60%
4	$Cr(acac)_3$	0
5	$Cr(CO)_6$	0
6	$Cr(OAc)_3$	0
7	$FeCl_2$	0
8	$FeCl_3$	0
9	$CoCl_2$	0
10	$NiCl_2$	0

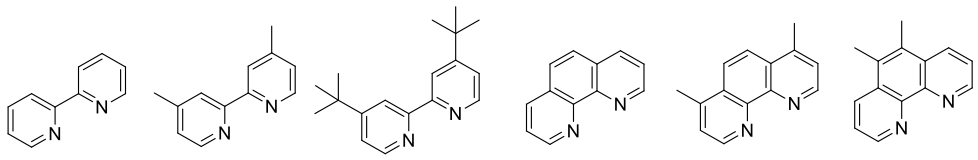
[a]Conditions: **1a** (0.8 mmol), **2a** (0.4 mmol), metal salt (0.04 mmol), Mg (1.6 mmol), TMSCl (0.2 mmol), dtbpy (0.04 mmol), THF (1 mL), 80°C, 12 h. Isolated yields were given.

Table S2. **Studying the Effect of Ligand on the Amidation of Ester**[a]



1a **2a** **3a**

Entry	**Ligand**	Yield (**3a**)
1	–	46%
2	**L1**	49%
3	**L2**	56%
4	**L3**	60%
5	**L4**	51%
6	**L5**	51%
7	**L6**	56%

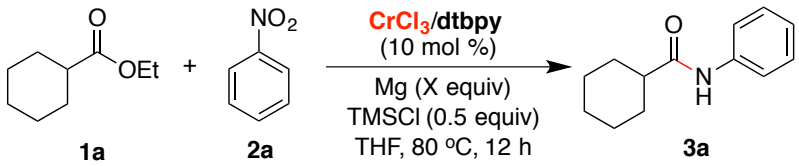


L1 **L2** **L3** **L4** **L5** **L6**

[a]Conditions: **1a** (0.8 mmol), **2a** (0.4 mmol), $CrCl_3$ (0.04 mmol), Mg (1.6 mmol), TMSCl (0.2 mmol),

ligand (0.04 mmol), THF (1 mL), 80°C, 12 h. Isolated yields were given.

Table S3. **Studying the Effect of the Amount of Mg on the Cr-Catalyzed Amidation of 1a**[a]

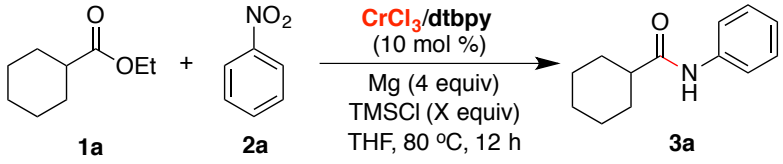


1a **2a** **3a**

Entry	Mg (X equiv)	Yield (**3a**)
1	–	0
2	1	0
3	2	0
4	3	trace
5	4	60%
6	5	61%
7	Zn (4 equiv)	0
8	Mn (4 equiv)	0

[a]Conditions: **1a** (0.8 mmol), **2a** (0.4 mmol), metal salt (0.04 mmol), Mg (X mmol), TMSCl (0.2 mmol), dtbpy (0.04 mmol), THF (1 mL), 80°C, 12 h. Isolated yields were given.

Table S4. **Studying the Effect of the Amount of TMSCl on the Cr-Catalyzed Amidation of 1a**[a]

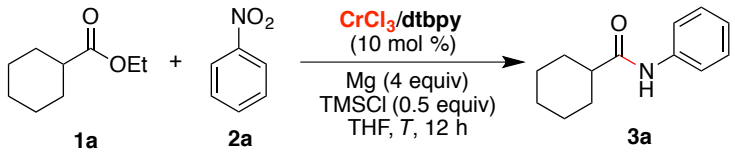


1a **2a** **3a**

Entry	TMSCl (X equiv)	Yield (**3a**)
1	0	0
2	0.5	60%
3	1	75%
4	1.5	75%
5	2	76%

[a]Conditions: **1a** (0.8 mmol), **2a** (0.4 mmol), $CrCl_3$ (0.04 mmol), Mg (1.6 mmol), TMSCl (X mmol), dtbpy (0.04 mmol), THF (1 mL), 80°C, 12 h. Isolated yields were given.

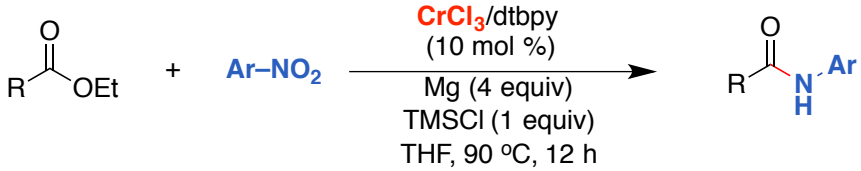
Table S5. **Studying the Effect of Temperature on the Cr-Catalyzed Amidation of 1a**[a]



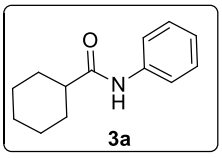
Entry	T/°C	Yield (**3a**)
1	60	0
2	70	<10%
3	80	75%
4	90	82%
5	100	83%

[a]Conditions: **1a** (0.8 mmol), **2a** (0.4 mmol), $CrCl_3$ (0.04 mmol), Mg (1.6 mmol), TMSCl (0.4 mmol), dtbpy (0.04 mmol), THF (1 mL), 12 h. Isolated yields were given.

3. General Procedure for Cr-Catalyzed Reductive Amidation of Esters with Nitroarenes

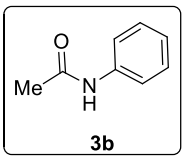


A dried Schlenk tube was charged with ester (0.8 mmol), nitroarene (0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), and TMSCl (50 μL, 0.4 mmol) followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, and the reaction mixture was stirred at 90 °C for 12 h. After quenched by HCl aqueous (2 mL, 1 mol/L), the crude product was extracted with ethyl acetate (3 X 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product.



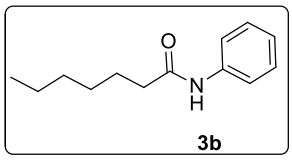
The general procedure was applied to ethyl cyclohexanecarboxylate (125 mg, 0.8 mmol), nitrobenzene

(49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (67 mg, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.61 (br s, 1H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.28 (dd, *J* = 12.9, 4.8 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 2.28–2.20 (m, 1H), 1.93 (d, *J* = 12.9 Hz, 2H), 1.82–1.79 (m, 2H), 1.68 (d, *J* = 8.2 Hz, 1H), 1.58–1.48 (m, 2H), 1.32–1.20 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.8, 138.3, 129.0, 124.1, 112.0, 46.5, 29.7, 25.7. HRMS (ESI^+): Calcd for $C_{13}H_{18}ON$ $[M+H]^+$ 204.1388, Found: 204.1387.



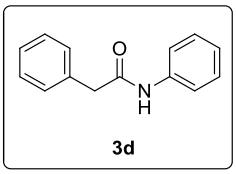
3b

The general procedure was applied to ethyl acetate (70 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/4) to afford the title compound as a white solid (36 mg, 66% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.58–7.46 (m, 3H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 2.16 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.7, 138.0, 129.1, 124.4, 120.1, 24.7. Calcd for C_8H_9ONNa $[M+Na]^+$ 158.0582, Found: 158.0582.



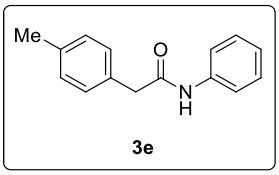
3b

The general procedure was applied to ethyl heptanoate (126 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (58 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.65 (br s, 1H), 7.52 (d, *J* = 7.9 Hz, 2H), 7.29 (t, *J* = 7.9 Hz, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 2.34 (t, *J* = 7.6 Hz, 2H), 1.74–1.61 (m, 2H), 1.35–1.25 (m, 6H), 0.90–0.86 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 172.0, 138.1, 129.0, 124.3, 120.1, 37.9, 31.7, 29.1, 25.8, 22.6, 14.1. HRMS (ESI^+): Calcd for $C_{13}H_{19}ONNa[M+Na]^+$ 228.1364, Found: 228.1366.



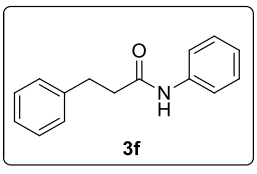
3d

The general procedure was applied to *ethyl 2-phenylacetate* (131 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (68 mg, 81% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.42–7.37 (m, 4H), 7.34–7.25 (m, 6H), 7.08 (t, *J* = 7.4 Hz, 1H), 3.72 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.4, 137.7, 134.5, 129.6, 129.3, 129.0, 127.8, 124.6, 120.0, 44.9. HRMS (ESI^+): Calcd for $C_{14}H_{14}ON$ $[M+H]^+$ 212.1075, Found: 212.1074.



3e

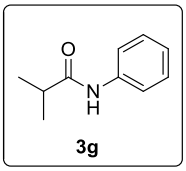
The general procedure was applied to *ethyl 2-(p-tolyl)acetate* (142 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (60 mg, 67% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.43 (d, *J* = 7.8 Hz, 2H), 7.32 (br s, 1H), 7.27 (t, *J* = 7.9 Hz, 2H), 7.24–7.17 (m, 4H), 7.08 (t, *J* = 7.4 Hz, 1H), 3.68 (s, 2H), 2.37 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.6, 137.8, 137.5, 131.5, 130.0, 129.52, 129.0, 124.5, 119.9, 44.5, 21.2. HRMS (ESI^+): Calcd for $C_{15}H_{15}ONNa$ $[M+Na]^+$ 248.1051, Found: 248.1051.



3f

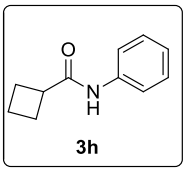
The general procedure was applied to *ethyl 3-phenylpropanoate* (142 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (66 mg, 73% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.48 (br s, 1H), 7.45 (d, *J* = 7.9 Hz, 2H), 7.31–7.26 (m,

4H), 7.22 (t, *J* = 6.5 Hz, 3H), 7.09 (t, *J* = 7.4 Hz, 1H), 3.03 (t, *J* = 7.7 Hz, 2H), 2.65 (t, *J* = 7.7 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.8, 140.7, 137.9, 129.0, 128.7, 128.5, 126.5, 124.4, 120.2, 39.4, 31.7. HRMS (ESI^+): Calcd for $C_{15}H_{15}ONNa$ $[M+Na]^+$ 248.1051, Found: 248.1048.



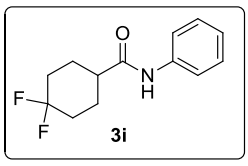
3g

The general procedure was applied to *ethyl isobutyrate* (93 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (47 mg, 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.59 (br s, 1H), 7.54 (d, *J* = 7.9 Hz, 2H), 7.29 (t, *J* = 7.8 Hz, 2H), 7.08 (t, *J* = 7.3 Hz, 1H), 2.52 (dt, *J* = 13.7, 6.8 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 175.7, 138.2, 129.0, 124.2, 120.1, 36.7, 19.7. HRMS (ESI^+): Calcd for $C_{10}H_{13}ONNa[M+Na]^+$ 186.0895, Found: 186.0900.



3h

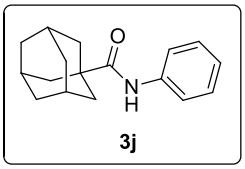
The general procedure was applied to *ethyl cyclobutanecarboxylate* (102 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (55 mg, 78% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.54 (d, *J* = 7.9 Hz, 2H), 7.44 (br s, 1H), 7.29 (t, *J* = 7.9 Hz, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 3.17 (p, *J* = 8.5 Hz, 1H), 2.45–2.29 (m, 2H), 2.25–2.14 (m, 2H), 2.03 –1.85 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 173.6, 138.2, 129.0, 124.2, 119.9, 40.9, 25.4, 18.2. HRMS (ESI^+): Calcd for $C_{11}H_{13}ONNa[M+Na]^+$ 198.0895, Found: 198.0894.



3i

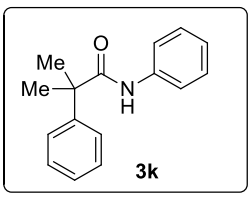
The general procedure was applied to *ethyl 4,4-difluorocyclohexanecarboxylate* (154 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg,

0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (60 mg, 63% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.50 (d, *J* = 7.9 Hz, 2H), 7.32 (t, *J* = 7.8 Hz, 3H), 7.12 (t, *J* = 7.3 Hz, 1H), 2.33 (t, *J* = 10.4 Hz, 1H), 2.22 (d, *J* = 11.7 Hz, 2H), 2.06–1.89 (m, 4H), 1.85–1.71 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 172.4, 137.8, 129.2, 124.7, 120.0, 43.9, 32.91 (t, $J_{C\text{-}F}$ = 250 Hz), 26.13, 26.03; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -92.75, -93.38, -100.48, -101.10. HRMS (ESI^+): Calcd for $C_{13}H_{15}F_2ONNa$ $[M+Na]^+$ 262.1019, Found: 262.1018.



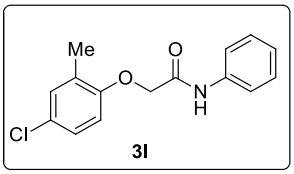
3j

The general procedure was applied to *Ethyl adamantane-1-carboxylate* (166 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (71 mg, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.54 (dd, *J* = 8.5, 1.0 Hz, 2H), 7.34 (br s, 1H), 7.32–7.28 (m, 2H), 7.11–7.06 (m, 1H), 2.09 (s, 3H), 1.96 (d, *J* = 2.7 Hz, 6H), 1.81–1.70 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 176.2, 138.2, 129.0, 124.2, 120.1, 41.6, 39.4, 36.5, 28.2. HRMS (ESI^+): Calcd for $C_{17}H_{21}ONNa$ $[M+Na]^+$ 278.1521, Found: 278.1524.



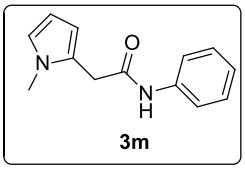
3k

The general procedure was applied to *ethyl 2-methyl-2-phenylpropanoate* (154 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (67 mg, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45–7.37 (m, 4H), 7.36–7.28 (m, 3H), 7.27–7.21 (m, 2H), 7.07–7.01 (m, 1H), 6.83 (br s, 1H), 1.66 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 175.7, 144.6, 138.0, 129.1, 128.9, 127.5, 126.5, 124.2, 119.7, 48.1, 27.1. HRMS (ESI^+): Calcd for $C_{16}H_{17}ONNa$ $[M+Na]^+$ 262.1208, Found: 262.1208.



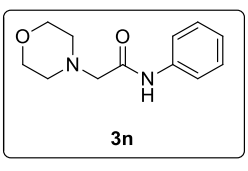
3l

The general procedure was applied to *ethyl 2-(4-chloro-2-methylphenoxy)acetate* (183 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (54 mg, 49% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.26 (br s, 1H), 7.60–7.55 (m, 2H), 7.37 (t, *J* = 7.9 Hz, 2H), 7.21–7.13 (m, 3H), 6.76 (d, *J* = 8.6 Hz, 1H), 4.58 (s, 2H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.1, 153.9, 136.8, 131.2, 129.3, 128.5, 127.2, 127.1, 125.1, 120.1, 113.1, 68.1, 16.4. HRMS (ESI^+): Calcd for $C_{15}H_{14}ONClNa$ $[M+Na]^+$ 298.0611, Found: 298.0617.



3m

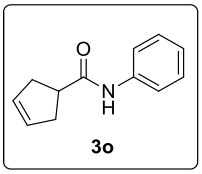
The general procedure was applied to *ethyl 2-(1-methyl-1H-pyrrol-2-yl)acetate* (133 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (46 mg, 54% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.42 (d, *J* = 7.7 Hz, 2H), 7.29 (t, *J* = 7.9 Hz, 3H), 7.09 (t, *J* = 7.4 Hz, 1H), 6.70–6.65 (m, 1H), 6.17–6.14 (m, 2H), 3.72 (s, 2H), 3.57 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.3, 137.6, 129.1, 125.5, 124.6, 123.7, 119.8, 109.8, 107.8, 36.1, 34.0. HRMS (ESI^+): Calcd for $C_{13}H_{14}ON_2Na$ $[M+Na]^+$ 237.1004, Found: 237.1002.



3n

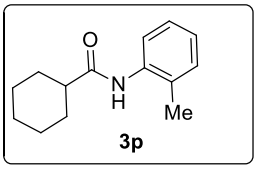
The general procedure was applied to *ethyl 2-morpholinoacetate* (138 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (49 mg, 56% yield). 1H NMR (400 MHz, $CDCl_3$) δ = 9.05 (br s, 1H), 7.57 (d, *J* = 8.1 Hz, 2H), 7.34 (t, *J* = 7.9

Hz, 2H), 7.12 (t, *J* = 7.4 Hz, 1H), 3.82–3.73 (m, 4H), 3.14 (s, 2H), 2.65–2.59 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.0, 137.6, 129.2, 124.5, 119.6, 67.2, 62.6, 53.9. HRMS (ESI^+): Calcd for $C_{12}H_{16}O_2N_2Na$ $[M+Na]^+$ 243.1109, Found: 243.1115.



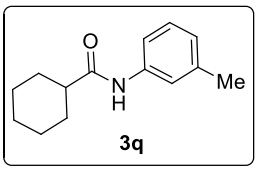
3o

The general procedure was applied to *ethyl cyclopent-3-enecarboxylate* (112 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (46 mg, 62% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.74 (br s, 1H), 7.53 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.9 Hz, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 5.70 (s, 2H), 3.16–3.08 (m, 1H), 2.78–2.62 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.7, 138.2, 129.3, 129.0, 124.2, 120.1, 44.4, 37.2. HRMS (ESI^+): Calcd for $C_{12}H_{13}ONNa$[M+Na]$^+$ 210.0895, Found: 210.0889.



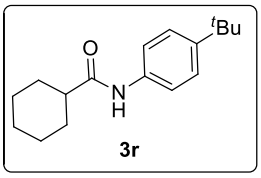
3p

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 2-nitrotoluene (55 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (54 mg, 62% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.74 (d, *J* = 7.8 Hz, 1H), 7.24–7.11 (m, 3H), 7.05 (t, *J* = 7.3 Hz, 1H), 2.32–2.25 (m, 1H), 2.22 (s, 3H), 1.96 (d, *J* = 12.2 Hz, 2H), 1.87–1.78 (m, 2H), 1.70 (d, *J* = 10.2 Hz, 1H), 1.58–1.48 (m, 2H), 1.37–1.21 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.5, 135.8, 130.4, 129.4, 126.7, 125.1, 123.5, 46.3, 29.9, 25.8, 17.8. HRMS (ESI^+): Calcd for $C_{14}H_{19}ONNa$ $[M+Na]^+$ 240.1364, Found: 240.1361.



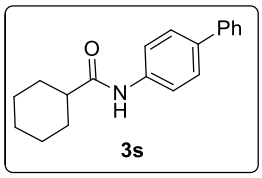
3q

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 3-nitrotoluene (55 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (73 mg, 84% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.65 (br s, 1H), 7.43 (s, 1H), 7.32 (d, *J* = 8.1 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 1H), 6.89 (d, *J* = 7.5 Hz, 1H), 2.29 (s, 3H), 2.27–2.20 (m, 1H), 1.92 (d, *J* = 13.3 Hz, 2H), 1.80 (dd, *J* = 9.3, 2.9 Hz, 2H), 1.68 (d, *J* = 7.7 Hz, 1H), 1.60–1.48 (m, 2H), 1.31–1.20 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.8, 138.8, 138.2, 128.8, 124.9, 120.7, 117.0, 46.5, 29.7, 25.8, 25.7, 21.5. HRMS (ESI^+): Calcd for $C_{14}H_{19}ONNa$ $[M+Na]^+$ 240.1364, Found: 240.1389.



3r

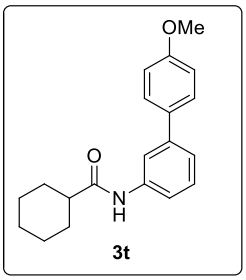
The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 1-tert-butyl-4-nitrobenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (75 mg, 72% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.47 (d, *J* = 8.6 Hz, 3H), 7.31 (d, *J* = 8.7 Hz, 2H), 2.26–2.19 (m, 1H), 1.93 (d, *J* = 13.3 Hz, 2H), 1.86–1.78 (m, 2H), 1.68 (d, *J* = 7.9 Hz, 1H), 1.60–1.48 (m, 2H), 1.30–1.24 (m, 12H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.6, 147.1, 135.7, 125.8, 119.7, 46.5, 34.4, 31.5, 29.8, 25.8. HRMS (ESI^+): Calcd for $C_{17}H_{25}ONNa$ $[M+Na]^+$ 282.1834, Found: 282.1837.



3s

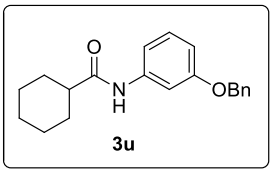
The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 4-nitrobiphenyl (80 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white

solid (75 mg, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.61 (d, *J* = 8.6 Hz, 2H), 7.58–7.51 (m, 4H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.34–7.30 (m, 2H), 2.29–2.22 (m, 1H), 1.97 (d, *J* = 13.1 Hz, 2H), 1.85 (d, *J* = 12.1 Hz, 2H), 1.73–1.70 (m, 1H), 1.61–1.52 (m, 2H), 1.35–1.25 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.6, 140.6, 137.5, 137.1, 128.9, 127.7, 127.2, 127.0, 120.2, 46.7, 29.8, 25.8. HRMS (ESI^+): Calcd for $C_{19}H_{22}ON$ $[M+H]^+$ 280.1701, Found: 280.1704.



3t

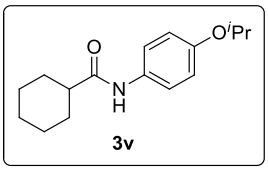
The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 4'-Methoxy-3-nitro-1,1'-biphenyl (92 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (100 mg, 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.79 (s, 1H), 7.61 (br s, 1H), 7.52–7.43 (m, 3H), 7.31 (t, *J* = 7.8 Hz, 1H), 7.26 (d, *J* = 8.7 Hz, 1H), 6.92 (d, *J* = 8.7 Hz, 2H), 3.81 (s, 3H), 2.28–2.20 (m, 1H), 1.93 (d, *J* = 13.0 Hz, 2H), 1.80 (dd, *J* = 9.5, 2.7 Hz, 2H), 1.67 (d, *J* = 8.1 Hz, 1H), 1.60–1.48 (m, 2H), 1.31–1.20 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.9, 159.3, 141.7, 138.7, 133.3, 129.3, 128.2, 122.5, 118.3, 118.2, 114.2, 55.4, 46.6, 29.8, 25.8, 25.7. HRMS (ESI^+): Calcd for $C_{20}H_{23}O_2NNa$ $[M+Na]^+$ 332.1626, Found: 332.1626.



3u

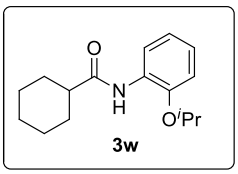
The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 1-nitro-3-phenylmethoxybenzene (92 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (87 mg, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.61 (br s, 1H), 7.49 (s, 1H), 7.42–7.37 (m, 4H), 7.35–7.30 (m, 1H), 7.18 (t, *J* = 8.1 Hz, 1H), 7.02 (d, *J* = 8.0 Hz, 1H), 6.72 (dd, *J* = 8.2, 2.1 Hz, 1H), 5.02 (s, 2H), 2.27–2.20 (m, 1H), 1.93 (d, *J* = 13.3 Hz, 2H), 1.82 (dd, *J* =

9.3, 2.9 Hz, 2H), 1.69 (d, *J* = 7.8 Hz, 1H), 1.59–1.50 (m, 2H), 1.30–1.22 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.9, 159.4, 139.6, 137.0, 129.7, 128.6, 127.98, 127.6, 112.2, 111.0, 106.4, 70.0, 46.6, 29.7, 25.7, 25.7. HRMS (ESI$^+$): Calcd for $C_{20}H_{23}O_2NNa$ $[M+Na]^+$ 332.1626, Found: 332.1618.



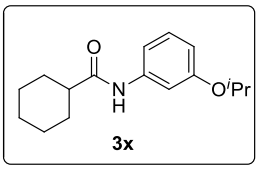
3v

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 1-nitro-4-propan-2-yloxybenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (74 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42–7.37 (m, 2H), 7.21 (br s, 1H), 6.82 (d, *J* = 8.9 Hz, 2H), 4.47 (dq, *J* = 12.1, 6.0 Hz, 1H), 2.24–2.16 (m, 1H), 1.93 (d, *J* = 13.3 Hz, 2H), 1.86–1.78 (m, 2H), 1.69 (dd, *J* = 8.2, 2.5 Hz, 1H), 1.57–1.47 (m, 2H), 1.28 (dd, *J* = 16.3, 7.6 Hz, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.4, 154.6, 131.2, 121.8, 116.5, 70.5, 46.5, 29.8, 25.8, 22.2. HRMS (ESI$^+$): Calcd for $C_{16}H_{23}O_2NNa$ $[M+Na]^+$ 284.1626, Found: 284.1623.



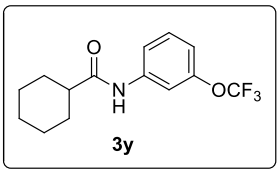
3w

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 2-Isopropoxynitrobenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (47 mg, 45% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.46–8.35 (m, 1H), 7.91 (br s, 1H), 7.00–6.96 (m, 1H), 6.94–6.90 (m, 1H), 6.87 (dd, *J* = 8.0, 1.1 Hz, 1H), 4.58 (dt, *J* = 12.1, 6.1 Hz, 1H), 2.30–2.23 (m, 1H), 1.99 (d, *J* = 13.0 Hz, 2H), 1.87–1.80 (m, 2H), 1.74–1.66 (m, 1H), 1.57–1.47 (m, 2H), 1.40–1.22 (m, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.1, 146.2, 128.9, 123.4, 121.1, 119.9, 112.8, 71.5, 46.8, 29.8, 25.9, 25.8, 22.4. HRMS (ESI$^+$): Calcd for $C_{16}H_{23}O_2NNa$ $[M+Na]^+$ 284.1626, Found: 284.1620.



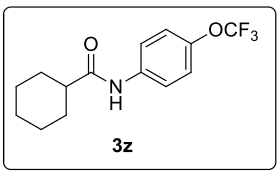
3x

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 1-isopropoxy-3-nitrobenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (66 mg, 63% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.59 (br s, 1H), 7.35 (s, 1H), 7.14 (t, *J* = 8.1 Hz, 1H), 6.96 (dd, *J* = 8.0, 0.8 Hz, 1H), 6.61 (dd, *J* = 8.2, 1.9 Hz, 1H), 4.55–4.46 (m, 1H), 2.25–2.18 (m, 1H), 1.91 (d, *J* = 13.2 Hz, 2H), 1.81–1.78 (m, 2H), 1.67 (d, *J* = 7.9 Hz, 1H), 1.56–1.47 (m, 2H), 1.26 (dd, *J* = 22.7, 7.6 Hz, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.8, 158.5, 139.6, 129.6, 112.0, 111.8, 107.4, 70.0, 46.6, 29.7, 25.8, 25.7, 22.1. HRMS (ESI^+): Calcd for $C_{16}H_{23}O_2NNa$ $[M+Na]^+$ 284.1626, Found: 284.1629.



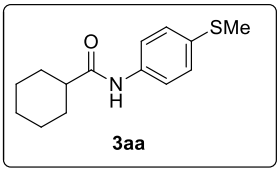
3y

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 3-(Trifluoromethoxy)nitrobenzene (83 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (91 mg, 79% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.16 (br s, 1H), 7.62 (s, 1H), 7.46–7.40 (m, 1H), 7.26 (t, *J* = 8.2 Hz, 1H), 6.93 (d, *J* = 8.2 Hz, 1H), 2.30–2.22 (m, 1H), 1.91 (d, *J* = 13.4 Hz, 2H), 1.83–1.74 (m, 2H), 1.67 (d, *J* = 5.3 Hz, 1H), 1.58–1.46 (m, 2H), 1.29–1.15 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 175.4, 149.6, 149.6, 139.8, 129.9, 118.1, 116.2, 112.99, 46.5, 29.7, 25.7, 25.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -57.82. HRMS (ESI^+): Calcd for $C_{14}H_{16}O_2F_3NNa$ $[M+Na]^+$ 310.1031, Found: 310.1035.



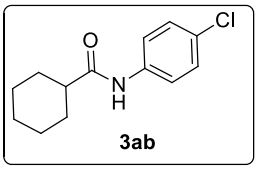
3z

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 1-Nitro-4-(trifluoromethoxy)benzene (83 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (87 mg, 76% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.71 (br s, 1H), 7.55 (t, *J* = 6.0 Hz, 2H), 7.13 (d, *J* = 8.6 Hz, 2H), 2.28–2.20 (m, 1H), 1.90 (t, *J* = 12.3 Hz, 2H), 1.85–1.75 (m, 2H), 1.72–1.62 (m, 1H), 1.56–1.47 (m, 2H), 1.32–1.17 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 175.0, 145.2, 136.9, 121.8, 121.2, 46.5, 29.7, 25.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -58.20. HRMS (ESI^+): Calcd for $C_{14}H_{16}O_2F_3NNa$ $[M+Na]^+$ 310.1031, Found: 310.1032.



3aa

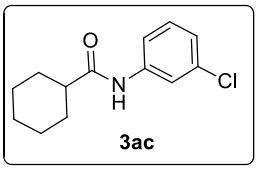
The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 4-Nitrothioanisole (68 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (72 mg, 72% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.46 (d, *J* = 8.6 Hz, 3H), 7.20 (d, *J* = 8.7 Hz, 2H), 2.44 (s, 3H), 2.25–2.18 (m, 1H), 1.92 (d, *J* = 12.3 Hz, 2H), 1.81 (dd, *J* = 9.6, 2.5 Hz, 2H), 1.68 (d, *J* = 8.0 Hz, 1H), 1.57–1.46 (m, 2H), 1.32–1.20 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.6, 135.9, 133.3, 128.1, 120.6, 46.5, 29.7, 25.7, 16.9. HRMS (ESI^+): Calcd for $C_{14}H_{19}OSNNa$ $[M+Na]^+$ 272.1085, Found: 272.1084.



3ab

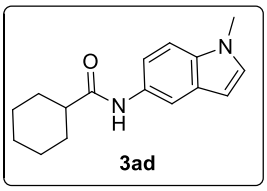
The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 4-Chloronitrobenzene (63 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (59 mg, 62% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.48 (d, *J* = 8.8 Hz, 2H), 7.35 (br s,

1H), 7.28–7.22 (m, 2H), 2.25–2.19 (m, 1H), 1.93 (d, *J* = 12.9 Hz, 2H), 1.83 (dd, *J* = 9.6, 2.5 Hz, 2H), 1.70 (d, *J* = 8.0 Hz, 1H), 1.57–1.51 (m, 2H), 1.34–1.24 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.6, 136.8, 129.1, 121.2, 120.0, 46.6, 29.7, 25.8, 25.8. HRMS (ESI^+): Calcd for $C_{13}H_{16}ONClNa$ $[M+Na]^+$ 260.0818, Found: 260.0817.



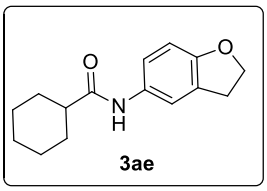
3ac

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 3-Chloronitrobenzene (63 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (62 mg, 65% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.77 (br s, 1H), 7.67 (s, 1H), 7.37 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.18 (t, *J* = 8.1 Hz, 1H), 7.04 (dd, *J* = 8.0, 1.0 Hz, 1H), 2.27–2.22 (m, 1H), 1.91 (d, *J* = 13.5 Hz, 2H), 1.81–1.78 (m, 2H), 1.71–1.63 (m, 1H), 1.58–1.45 (m, 2H), 1.31–1.18 (m, 3H); ^{13}C NMR (101 MHz, CDCl3) δ = 175.1, 139.5, 134.6, 130.0, 124.2, 120.2, 118.0, 46.5, 29.7, 25.7, 25.7. HRMS (ESI^+): Calcd for $C_{13}H_{16}ONClNa$ $[M+Na]^+$ 260.0818, Found: 260.0814.



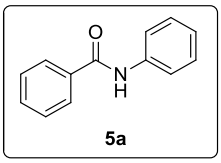
3ad

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 1-methyl-5-nitroindole (70 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/4) to afford the title compound as a white solid (68 mg, 66% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.83 (d, *J* = 1.6 Hz, 1H), 7.40 (br s, 1H), 7.30–7.25 (m, 1H), 7.20 (d, *J* = 8.7 Hz, 1H), 7.01 (d, *J* = 3.0 Hz, 1H), 6.41 (d, *J* = 2.9 Hz, 1H), 3.74 (s, 3H), 2.28–2.20 (m, 1H), 1.97 (d, *J* = 13.1 Hz, 2H), 1.87–1.79 (m, 2H), 1.73–1.67 (m, 1H), 1.62–1.52 (m, 2H), 1.36–1.23 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.5, 134.2, 130.5, 129.6, 128.6, 116.0, 112.8 109.3, 101.1, 46.5, 33.0, 29.9, 25.9. HRMS (ESI^+): Calcd for $C_{16}H_{20}ON_2Na$ $[M+Na]^+$ 279.1473, Found: 279.1478.



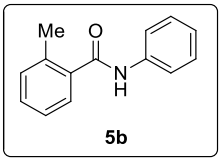
3ae

The general procedure was applied to *ethyl cyclohexanecarboxylate* (125 mg, 0.8 mmol), 5-nitro-2,3-dihydro-1-benzofuran (66 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/4) to afford the title compound as a white solid (46 mg, 47% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.52 (s, 1H), 7.32 (br s, 1H), 7.01 (dd, *J* = 8.4, 1.9 Hz, 1H), 6.67 (d, *J* = 8.5 Hz, 1H), 4.53 (t, *J* = 8.7 Hz, 2H), 3.15 (t, *J* = 8.7 Hz, 2H), 2.19 (tt, *J* = 11.7, 3.4 Hz, 1H), 1.92 (d, *J* = 13.2 Hz, 2H), 1.86–1.77 (m, 2H), 1.68 (d, *J* = 8.1 Hz, 1H), 1.57–1.46 (m, 2H), 1.26 (dd, *J* = 14.0, 8.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.5, 156.9, 131.1, 127.6, 120.4, 118.4, 109.0, 71.5, 46.4, 30.0, 29.8, 25.8. HRMS (ESI^+): Calcd for $C_{15}H_{19}O_2NNa$ $[M+Na]^+$ 268.1313, Found: 268.1309.



5a

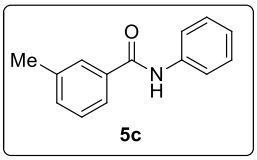
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (70 mg, 89% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.93 (br s, 1H), 7.86 (dd, *J* = 5.3, 3.3 Hz, 2H), 7.64 (d, *J* = 7.7 Hz, 2H), 7.57–7.51 (m, 1H), 7.50–7.43 (m, 2H), 7.39–7.33 (m, 2H), 7.18–7.12 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.0, 138.0, 135.1, 132.0, 129.2, 128.9, 127.2, 124.7, 120.4. HRMS (ESI^+): Calcd for $C_{13}H_{12}ON$ $[M+H]^+$ 198.0919, Found: 198.0915.



5b

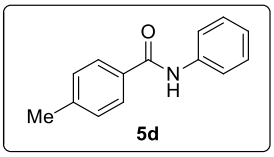
The general procedure was applied to *ethyl 2-methylbenzoate* (131 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column

chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (68 mg, 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.66 (d, *J* = 8.0 Hz, 3H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 3H), 7.30 (dd, *J* = 8.9, 3.3 Hz, 2H), 7.20 (t, *J* = 7.4 Hz, 1H), 2.53 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.3, 138.1, 136.5, 131.4, 130.4, 129.2, 126.7, 126.0, 124.7, 120.0, 19.9. HRMS (ESI^+): Calcd for $C_{14}H_{14}ON$ $[M+H]^+$ 212.1075, Found: 212.1077.



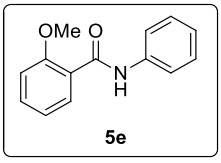
5c

The general procedure was applied to ethyl 3-methylbenzoate (131 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (57 mg, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.21 (br s, 1H), 7.64–7.59 (m, 4H), 7.34–7.25 (m, 4H), 7.14–7.08 (m, 1H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.3, 138.6, 138.1, 135.0, 132.6, 129.0, 128.6, 128.0, 124.5, 124.1, 120.5, 21.4. HRMS (ESI^+): Calcd for $C_{14}H_{14}ON$ $[M+H]^+$ 212.1075, Found: 212.1072.



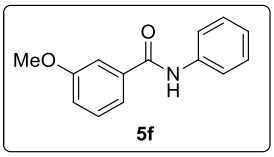
5d

The general procedure was applied to ethyl 4-methylbenzoate (131 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (57 mg, 68% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.08 (br s, 1H), 7.75 (d, *J* = 8.2 Hz, 2H), 7.68–7.61 (m, 2H), 7.34 (t, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 7.9 Hz, 2H), 7.15–7.11 (m, 1H), 2.40 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.0, 142.4, 138.2, 132.2, 129.5, 129.1, 127.2, 124.5, 120.4, 21.6. HRMS (ESI^+): Calcd for $C_{14}H_{14}ON$ $[M+H]^+$ 212.1075, Found: 212.1076.



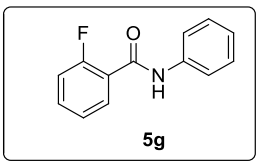
5e

The general procedure was applied to ethyl 2-methoxybenzoate (144 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (55 mg, 61% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 9.82 (br s, 1H), 8.29 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.70–7.68 (m, 2H), 7.50–7.46 (m, 1H), 7.39–7.34 (m, 2H), 7.16–7.09 (m, 2H), 7.01 (d, *J* = 8.2 Hz, 1H), 4.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 163.3, 157.2, 138.5, 133.3, 132.5, 129.0, 124.2, 121.8, 121.7, 120.5, 111.6, 56.3. HRMS (ESI^+): Calcd for $C_{14}H_{14}O_2N$ $[M+H]^+$ 228.1025, Found: 228.1016.



5f

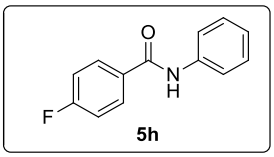
The general procedure was applied to ethyl 3-methoxybenzoate (144 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (60 mg, 66% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.16 (br s, 1H), 7.64 (d, *J* = 7.8 Hz, 2H), 7.43–7.39 (m, 1H), 7.39–7.29 (m, 4H), 7.14 (t, *J* = 7.4 Hz, 1H), 7.06–7.02 (m, 1H), 3.80 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.9, 160.0, 138.0, 136.5, 129.8, 129.1, 124.7, 120.5, 118.9, 118.1, 112.5, 55.5. HRMS (ESI^+): Calcd for $C_{14}H_{14}O_2N$ $[M+H]^+$ 228.1025, Found: 228.1024.



5g

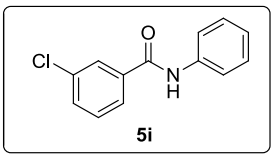
The general procedure was applied to ethyl 2-fluorobenzoate (134 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (40 mg, 46% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.47 (d, *J* = 14.6 Hz, 1H), 8.15 (dd, *J* = 11.2, 4.7 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 2H), 7.54–7.48 (m, 1H), 7.37 (t, *J* = 7.9 Hz, 2H), 7.33–7.27 (m, 1H), 7.21–7.12 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 161.4, 160.46 (d, $J_{C\text{-}F}$ =245 Hz), 137.8, 133.83 (d, $J_{C\text{-}F}$ = 10 Hz), 132.3, 129.2, 125.28 (d, $J_{C\text{-}F}$ = 3 Hz), 124.9, 121.51 (d, $J_{C\text{-}F}$ = 11 Hz), 120.6, 116.23 (d, $J_{C\text{-}F}$ = 25

Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ = -113.21. HRMS (ESI^+): Calcd for $C_{13}H_{10}OFNaN$ $[M+Na]^+$ 238.0644, Found: 238.0649.



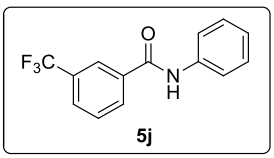
5h

The general procedure was applied to ethyl 4-fluorobenzoate (134 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (56 mg, 65% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.92–7.86 (m, 2H), 7.77 (br s, 1H), 7.62 (d, J = 7.6 Hz, 2H), 7.38 (t, J = 8.0 Hz, 2H), 7.17 (dd, J = 11.9, 5.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.04 (d, $J_{C\text{-}F}$ = 257 Hz), 164.8, 137.9, 129.55 (d, $J_{C\text{-}F}$ = 9 Hz), 129.3, 124.9, 120.4, 116. 04 (d, $J_{C\text{-}F}$ = 22 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ = -107.41. HRMS (ESI^+): Calcd for $C_{13}H_{11}OFN$ $[M+H]^+$ 216.0825, Found: 216.0818.



5i

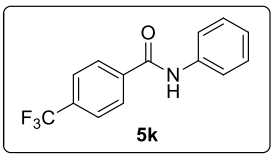
The general procedure was applied to ethyl 3-chlorobenzoate (148 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (55 mg, 60% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.98 (br s, 1H), 7.83 (t, J = 1.8 Hz, 1H), 7.74–7.69 (m, 1H), 7.62 (d, J = 7.7 Hz, 2H), 7.51–7.48 (m, 1H), 7.41–7.34 (m, 3H), 7.19–7.13 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 164.6, 137.7, 136.9, 135.1, 132.0, 130.2, 129.3, 127.5, 125.3, 125.0, 120.5. HRMS (ESI^+): Calcd for $C_{13}H_{11}OClN$ $[M+H]^+$ 232.0529, Found: 232.0506.



5j

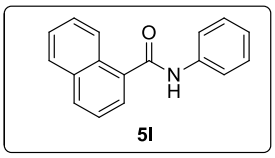
The general procedure was applied to ethyl 3-(trifluoromethyl)benzoate (174 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified

by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (74 mg, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.18 (br s, 1H), 8.10 (s, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 7.77 (d, *J* = 7.7 Hz, 1H), 7.62 (d, *J* = 7.8 Hz, 2H), 7.57 (t, *J* = 7.8 Hz, 1H), 7.35 (t, *J* = 7.9 Hz, 2H), 7.17 (t, *J* = 7.4 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 164.7, 137.6, 135.9, 131.34 (q, J_{C-F} = 32 Hz), 130.5, 129.5, 129.2, 128.49 (q, J_{C-F} = 4 Hz), 125.2, 124.21 (q, J_{C-F} = 4 Hz), 123.75 (q, J_{C-F} = 271 Hz), 120.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -62.73. HRMS (ESI^+): Calcd for $C_{14}H_{10}OF_3N$ $[M+Na]^+$ 288.0612, Found: 288.0617.



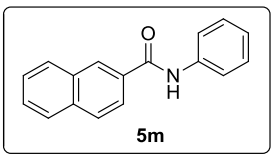
5k

The general procedure was applied to ethyl 4-(trifluoromethyl)benzoate (174 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (46 mg, 43% yield). ^{1}H NMR (400 MHz, DMSO) δ = 10.48 (br s, 1H), 8.17 (d, *J* = 8.1 Hz, 2H), 7.89 (d, *J* = 8.2 Hz, 2H), 7.81 (d, *J* = 7.6 Hz, 2H), 7.37 (dd, *J* = 10.8, 5.0 Hz, 2H), 7.13 (t, *J* = 7.4 Hz, 1H); ^{13}C NMR (101 MHz, DMSO) δ = 164.4, 138.9, 138.8, 131.45 (q, J_{C-F} = 32 Hz), 128.7, 128.6, 125.35 (q, J_{C-F} = 3 Hz), 124.0, 123.96 (q, J_{C-F} = 271 Hz), 120.5; ^{19}F NMR (376 MHz, DMSO) δ = -61.49. HRMS (ESI^+): Calcd for $C_{14}H_{10}OF_3N$ $[M+Na]^+$ 288.0612, Found: 288.0611.



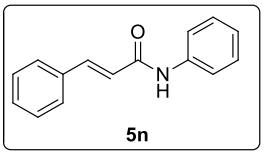
5l

The general procedure was applied to ethyl 1-naphthoate (160 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (70 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.34–8.32 (m, 1H), 7.93 (d, *J* = 8.3 Hz, 1H), 7.91–7.83 (m, 2H), 7.68 (d, *J* = 7.2 Hz, 3H), 7.57–7.51 (m, 2H), 7.45 (dd, *J* = 8.1, 7.2 Hz, 1H), 7.38 (t, *J* = 7.8 Hz, 2H), 7.18 (t, *J* = 7.4 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 167.7, 138.2, 134.5, 133.8, 131.1, 130.2, 129.3, 128.5, 127.5, 126.7, 125.4, 125.2, 124.8, 124.8, 120.1. HRMS (ESI^+): Calcd for $C_{17}H_{14}ON$ $[M+H]^+$ 248.1075, Found: 248.1067.



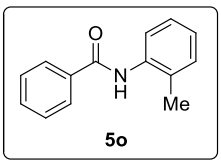
5m

The general procedure was applied to ethyl 2-naphthoate (160 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (62 mg, 63% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.37 (s, 1H), 8.04 (br s, 1H), 7.95–7.87 (m, 4H), 7.73–7.67 (m, 2H), 7.60–7.57 (m, 2H), 7.43–7.36 (m, 2H), 7.20–7.14 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.0, 138.1, 135.0, 132.7, 132.34, 129.3, 129.1, 128.9, 128.1, 127.9, 127.7, 127.1, 124.8, 123.7, 120.4. HRMS (ESI^+): Calcd for $C_{17}H_{14}ON$ $[M+H]^+$ 248.1075, Found: 248.1082.



5n

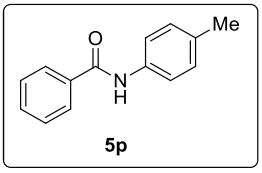
The general procedure was applied to ethyl cinnamate (153 mg, 0.8 mmol), nitrobenzene (49 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (45 mg, 50% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.79 (br s, 1H), 7.75 (d, *J* = 15.5 Hz, 1H), 7.65 (d, *J* = 7.0 Hz, 2H), 7.48 (dd, *J* = 6.5, 2.8 Hz, 2H), 7.34 (dd, *J* = 10.1, 5.3 Hz, 5H), 7.12 (t, *J* = 7.3 Hz, 1H), 6.61 (d, *J* = 15.5 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 164.3, 142.5, 138.2, 134.7, 130.1, 129.2, 129.0, 128.1, 124.6, 121.1, 120.2. HRMS (ESI^+): Calcd for $C_{15}H_{13}ONNa$ $[M+Na]^+$ 246.0895, Found: 246.0895.



5o

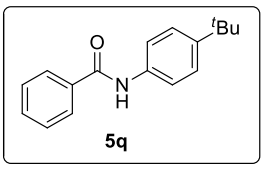
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 2-nitrotoluene (55 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (58 mg, 69% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.88–7.83 (m, 4H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 2H),

7.23 (t, J = 7.3 Hz, 2H), 7.13 (dd, J = 10.7, 4.1 Hz, 1H), 2.30 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.9, 135.8, 134.9, 131.9, 130.6, 129.9, 128.8, 127.2, 126.8, 125.5, 123.6, 17.9. HRMS (ESI^+): Calcd for $C_{14}H_{14}ON$ $[M+H]^+$ 212.1075, Found: 212.1072.



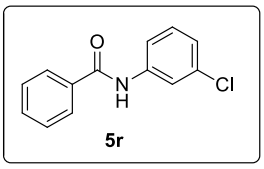
5p

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 4-nitrotoluene (55 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (62 mg, 73% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 (br s, 1H), 7.85 (d, J = 7.3 Hz, 2H), 7.52 (dd, J = 7.8, 3.4 Hz, 3H), 7.44 (t, J = 7.4 Hz, 2H), 7.15 (d, J = 8.2 Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.9, 135.5, 135.2, 134.3, 131.8, 129.7, 128.8, 127.2, 120.5, 21.0. HRMS (ESI^+): Calcd for $C_{14}H_{14}ON$ $[M+H]^+$ 212.1075, Found: 212.1069.



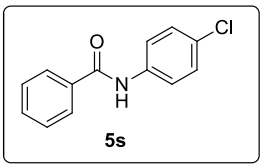
5q

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 1-tert-butyl-4-nitrobenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (61 mg, 60% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.12 (br s, 1H), 7.87–7.82 (m, 2H), 7.58 (d, J = 8.6 Hz, 2H), 7.54–7.48 (m, 1H), 7.42 (t, J = 7.5 Hz, 2H), 7.38–7.34 (m, 2H), 1.33 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.0, 147.6, 135.5, 135.2, 131.7, 128.7, 127.2, 125.9, 120.3, 34.5, 31.5. HRMS (ESI^+): Calcd for $C_{17}H_{20}ON$ $[M+H]^+$ 254.1545, Found: 254.1563.



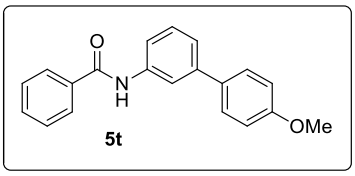
5r

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 3-Chloronitrobenzene (63 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (77 mg, 83% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.66 (d, *J* = 36.5 Hz, 1H), 7.82–7.73 (m, 3H), 7.51–7.43 (m, 2H), 7.38–7.30 (m, 2H), 7.21–7.15 (m, 1H), 7.07 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.6, 139.2, 134.5, 134.4, 132.0, 129.9, 128.7, 127.2, 124.6, 120.8, 118.7. HRMS (ESI^+): Calcd for $C_{13}H_{11}OClN$ $[M+H]^+$ 232.0529, Found: 232.0530.



5s

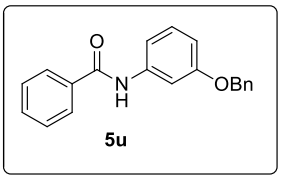
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 4-Chloronitrobenzene (63 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (72 mg, 78% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.12 (d, *J* = 8.1 Hz, 2H), 7.86 (d, *J* = 7.9 Hz, 1H), 7.61 (dd, *J* = 12.1, 4.8 Hz, 2H), 7.55 (d, *J* = 6.9 Hz, 1H), 7.52–7.45 (m, 3H), 7.33 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 171.9, 133. 9, 132.2, 130.3, 129.3, 129.0, 128.6, 127.2, 121.6. HRMS (ESI^+): Calcd for $C_{13}H_{11}OClN$ $[M+H]^+$ 232.0529, Found: 232.0522.



5t

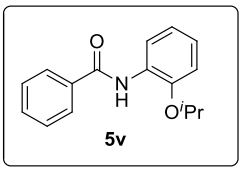
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 4'-Methoxy-3-nitro-1,1'-biphenyl (92 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/4) to afford the title compound as a white solid (99 mg, 82% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.17 (br s, 1H), 7.90–7.84 (m, 3H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.54–7.50 (m, 3H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.38–7.32 (m, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.1, 159.4, 141.8,

138.5, 135.0, 133.2, 131.9, 129.5, 128.8, 128.3, 127.2, 123.0, 118.7, 114.3, 55.4. HRMS (ESI^+): Calcd for $C_{20}H_{17}O_2NNa$ $[M+Na]^+$ 326.1157, Found: 326.1158.



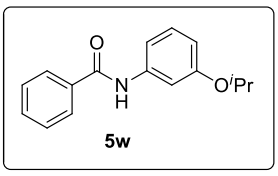
5u

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 1-nitro-3-phenylmethoxybenzene (92 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (93 mg, 77% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.28 (br s, 1H), 7.82–7.76 (m, 2H), 7.52 (d, *J* = 1.9 Hz, 1H), 7.48–7.40 (m, 1H), 7.39–7.26 (m, 7H), 7.21–7.11(m, 2H), 6.74–6.71 (m, 1H), 4.98 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.2, 159.4, 139.3, 136.9, 134.9, 131.8, 129.8, 128.7, 128.6, 128.0, 127.6, 127.2, 112.9, 111.4, 107.0, 70.0. HRMS (ESI^+): Calcd for $C_{20}H_{17}O_2NNa$ $[M+Na]^+$ 326.1157, Found: 326.1155.



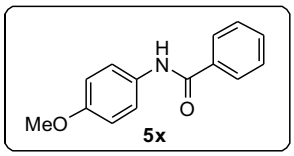
5v

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 2-Isopropoxynitrobenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (46 mg, 45% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.69 (br s, 1H), 8.57 (dd, *J* = 7.9, 1.8 Hz, 1H), 7.93–7.87 (m, 2H), 7.59–7.48 (m, 3H), 7.09–6.98 (m, 2H), 6.93 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.65 (dt, *J* = 12.1, 6.1 Hz, 1H), 1.41 (d, *J* = 6.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.1, 146.5, 135.5, 131.8, 128.9, 128.9, 127.1, 123.9, 121.3, 119.9, 112.7, 71.6, 22.4. HRMS (ESI^+): Calcd for $C_{16}H_{17}O_2NNa$ $[M+Na]^+$ 278.1157, Found: 278.1163.



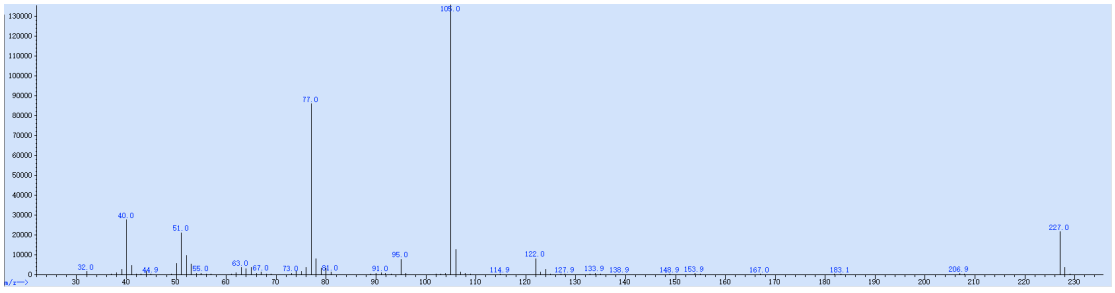
5w

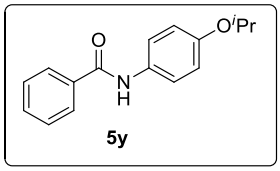
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 1-isopropoxy-3-nitrobenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (68 mg, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.13 (br s, 1H), 7.87–7.80 (m, 2H), 7.54–7.47 (m, 1H), 7.46–7.38 (m, 3H), 7.21 (t, *J* = 8.1 Hz, 1H), 7.10 (dd, *J* = 8.0, 1.0 Hz, 1H), 6.68 (dd, *J* = 8.2, 1.8 Hz, 1H), 4.54 (dt, *J* = 12.1, 6.1 Hz, 1H), 1.32 (d, *J* = 6.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.0, 158.6, 139.3, 135.1, 131.8, 129.8, 128.8, 127.1, 112.5, 112.4, 107.9, 70.1, 22.1. HRMS (ESI$^+$): Calcd for $C_{16}H_{17}O_2NNa$ $[M+Na]^+$ 278.1157, Found: 278.1165.



5x

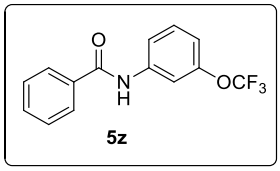
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 1-methoxy-4-nitrobenzene (61 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a yellow solid (65 mg, 72% yield). ^{1}H NMR (400 MHz, DMSO) δ 10.16 (s, 1H), 7.95 (d, *J* = 7.1 Hz, 2H), 7.69 (d, *J* = 8.9 Hz, 2H), 7.62 – 7.46 (m, 3H), 6.93 (d, *J* = 9.0 Hz, 2H), 3.73 (d, *J* = 11.3 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 165.5, 155.9, 135.4, 132.6, 131.7, 128.7, 128.0, 122.4, 114.1, 55.5. GC-MS (EI) calcd for $C_{14}H_{13}NO_2$ (M) 227.1, found 227.0.





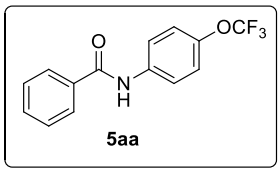
5y

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 1-nitro-4-propan-2-yloxybenzene (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (73 mg, 72% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 7.93 (br s, 1H), 7.84 (d, *J* = 7.3 Hz, 2H), 7.55–7.48 (m, 3H), 7.44 (t, *J* = 7.4 Hz, 2H), 6.90– 6.84 (m, 2H), 4.51 (dt, *J* = 12.1, 6.1 Hz, 1H), 1.33 (d, *J* = 6.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.9, 155.0, 135.2, 131.7, 131.0, 128.8, 127.1, 122.3, 116.5, 70.5, 22.2. HRMS (ESI^+): Calcd for $C_{16}H_{17}O_2NNa$ $[M+Na]^+$ 278.1157, Found: 278.1165.



5z

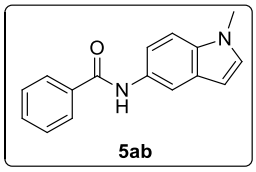
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 3-(Trifluoromethoxy)nitrobenzene (83 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (87 mg, 77% yield). ^1H NMR (400 MHz, $CDCl_3$) δ = 8.38 (br s, 1H), 7.86–7.78 (m, 2H), 7.69 (s, 1H), 7.56–7.47 (m, 2H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 8.2 Hz, 1H), 7.01–6.96 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.4, 149.7, 149.6, 139.5, 134.5, 132.2, 130.1, 128.8, 127.2, 118.5, 116.7, 113.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -57.73. HRMS (ESI^+): Calcd for $C_{14}H_{10}O_2NF_3Na$ $[M+Na]^+$ 304.0561, Found: 304.0564.



5aa

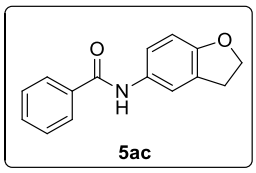
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 4-(Trifluoromethoxy)nitrobenzene (83 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^oC for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a white solid (65 mg, 58% yield). ^1H NMR (400 MHz, DMSO) δ = 10.45 (br s, 1H),

7.99–7.94 (m, 2H), 7.93–7.88 (m, 2H), 7.61 (ddd, *J* = 6.3, 3.7, 1.3 Hz, 1H), 7.57–7.51 (m, 2H), 7.37 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (101 MHz, DMSO) δ = 165.7, 143.9, 138.4, 134.7, 131.8, 128.5, 127.7, 121.7, 121.5, 54.9; ^{19}F NMR (376 MHz, DMSO) δ = -57.05. HRMS (ESI$^+$): Calcd for $C_{14}H_{10}O_2NF_3Na$ $[M+Na]^+$ 304.0561, Found: 304.0565.



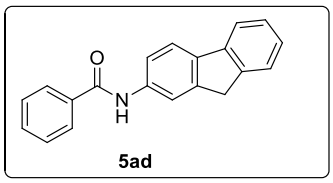
5ab

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 1-methyl-5-nitroindole (70 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/4) to afford the title compound as a white solid (45 mg, 45% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95–7.89 (m, 4H), 7.56–7.51 (m, 1H), 7.48 (t, *J* = 7.3 Hz, 2H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.28 (d, *J* = 8.7 Hz, 1H), 7.06 (d, *J* = 3.0 Hz, 1H), 6.46 (d, *J* = 3.0 Hz, 1H), 3.78 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.9, 135.6, 134.5, 131.6, 130.3, 129.9, 128.8, 128.7, 127.1, 116.2, 113.2, 109.5, 101.2, 33.1. HRMS (ESI$^+$): Calcd for $C_{16}H_{15}ON_2$ $[M+H]^+$ 251.1184, Found: 251.1183.



5ac

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 5-nitro-2,3-dihydro-1-benzofuran (66 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 ^{o}C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/4) to afford the title compound as a white solid (33 mg, 34% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.94 (br s, 1H), 7.84 (d, *J* = 7.3 Hz, 2H), 7.61 (s, 1H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.44 (t, *J* = 7.4 Hz, 2H), 7.14 (d, *J* = 7.5 Hz, 1H), 6.73 (d, *J* = 8.4 Hz, 1H), 4.56 (t, *J* = 8.7 Hz, 2H), 3.19 (t, *J* = 8.7 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.9, 157.3, 135.1, 131.7, 130.9, 128.8, 127.8, 127.1, 121.0, 118.8, 109.2, 71.6, 30.0. HRMS (ESI$^+$): Calcd for $C_{15}H_{13}O_2NNa$ $[M+Na]^+$ 262.0844, Found: 262.0846.



5ad

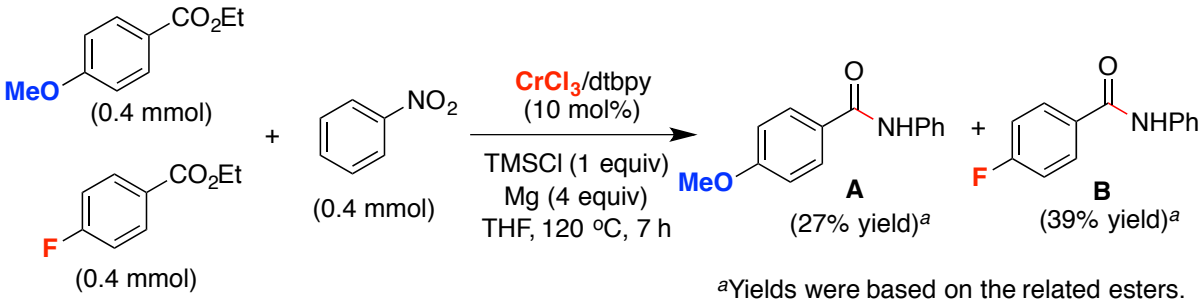
The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 2-nitrofluorene (85 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/4) to afford the title compound as a white solid (44.5 mg, 39% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.04 (s, 1H), 7.97 (br s, 1H), 7.92–7.87 (m, 2H), 7.75 (d, *J* = 8.2 Hz, 2H), 7.57–7.47 (m, 5H), 7.37 (t, *J* = 7.4 Hz, 1H), 7.29 (td, *J* = 7.4, 1.0 Hz, 1H), 3.91 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.9, 144.6, 143.4, 141.4, 138.5, 136.9, 135.2, 132.0, 129.0, 127.2, 126.9, 126.5, 125.1, 120.3, 119.7, 119.1, 117.3, 37.2. HRMS (ESI$^+$): Calcd for $C_{20}H_{15}ONNa$ $[M+Na]^+$ 308.1051, Found: 308.1056.

Gram scale amidation by reaction of ethyl benzoate with nitrobenzene:

A dried Schlenk tube was charged with ethyl benzoate (3 g, 20 mmol), nitrobenzene (1.23 g, 10 mmol), $CrCl_3$ (157 mg, 0.1 mmol), Mg (960 mg, 40 mmol), dtbpy (250 mg, 0.1 mmol), and TMSCl (2.5 mL, 10 mmol) followed by adding a freshly distilled THF (25 mL) by syringe under atmosphere of nitrogen, and the reaction mixture was stirred at 90 °C for 12 h. After quenched by HCl aqueous (20 mL, 1 M), the crude product was extracted with ethyl acetate (3 X 30 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product **5a** as a white solid (531 mg, 27% yield).

4. Competing Experiments and Application in the Synthesis of Biologically Interesting Motifs

Competing experiment between electron-rich and electron-withdrawing aryl esters

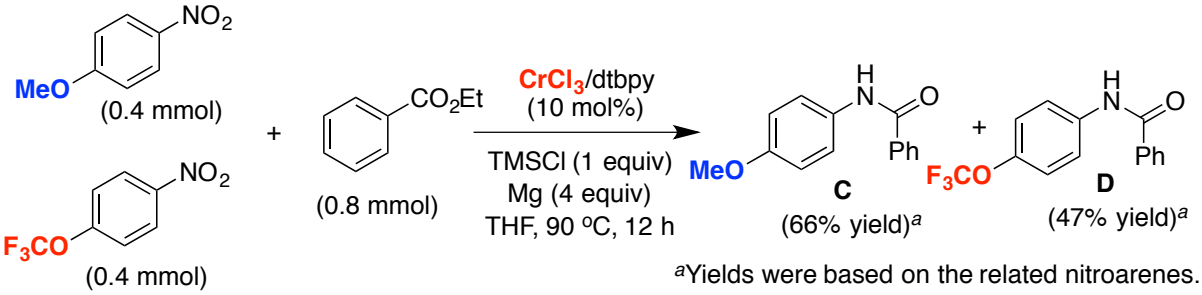


[a]Yields were based on the related esters.

The general procedure was applied to *nitrobenzene* (49 mg, 0.4 mmol), ethyl 4-fluorobenzoate

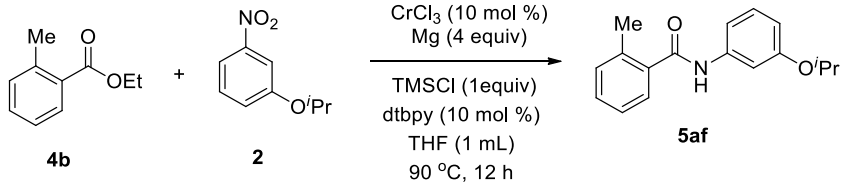
(67 mg, 0.4 mmol), ethyl 4-methoxybenzoate (72 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL, 0.4 mmol), THF (1 mL) at 120 °C for 7 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound **B** as a white solid (34 mg, 39% yield) and **A** as a yellow solid (25 mg, 27%).

Competing experiment between electron-rich and electron-withdrawing nitrobenzenes

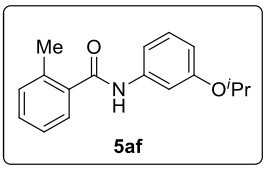


[a]Yields were based on the related nitroarenes.

The general procedure was applied to *ethyl benzoate* (120 mg, 0.8 mmol), 1-nitro-4-(trifluoromethoxy)benzene (83 mg, 0.4 mmol), 1-methoxy-4-nitrobenzene (61 mg, 0.4 mmol), $CrCl_3$ (13 mg, 0.08 mmol), Mg (77 mg, 3.2 mmol), dtbpy (21 mg, 0.08 mmol), TMSCl (100 μL, 0.8 mmol), THF (2 mL) at 90 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound **D** as a white solid (53 mg, 47% yield) and **C** as a yellow solid (60 mg, 66%).

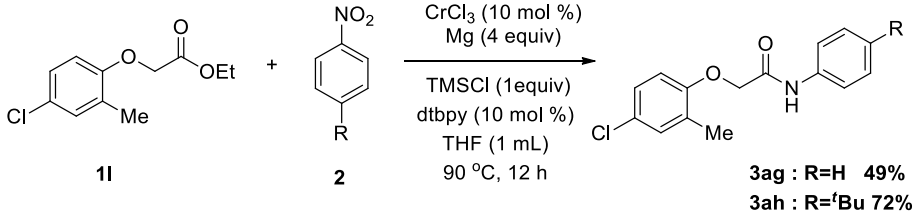


A dried Schlenk tube was charged with **4b** (131 mg, 0.8 mmol), $CrCl_3$ (6 mg, 0.04 mmol), 1-isopropoxy-3-nitrobenzene (72 mg, 0.4 mmol), TMSCl (50 μL, 0.4 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol) followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 90 °C for 12 h. After quenched by aqueous HCl (2 mL, 1 mol/L), the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product **5af** (72 mg, 67%).

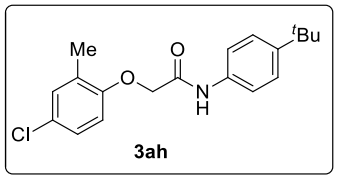


5af

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.70 (br s, 1H), 7.41–7.35 (m, 2H), 7.32 (td, *J* = 7.6, 1.1 Hz, 1H), 7.19 (dd, *J* = 14.5, 7.5 Hz, 3H), 7.05 (d, *J* = 7.7 Hz, 1H), 6.67 (dd, *J* = 8.2, 2.0 Hz, 1H), 4.55 (dt, *J* = 11.9, 5.9 Hz, 1H), 2.44 (s, 3H), 1.32 (d, *J* = 6.0 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.2, 158.6, 139.3, 136.5, 136.4, 131.3, 130.3, 129.8, 126.7, 125.9, 112.2, 112.0, 107.6, 70.05, 22.1, 20.0. HRMS (ESI$^+$): Calcd for $C_{17}H_{19}O_2NNa$ $[M+Na]^+$ 292.1313, Found: 292.1311.



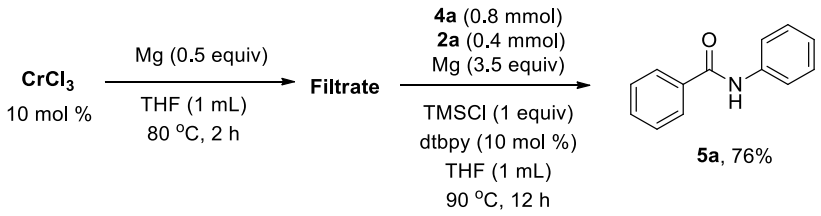
A dried Schlenk tube was charged with *ethyl 2-(4-chloro-2-methylphenoxy)acetate* (183 mg, 0.8 mmol), $CrCl_3$ (6 mg, 0.04 mmol), **2** (0.4 mmol), and TMSCl (50 μL, 0.4 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 90 °C for 12 h. After quenched by HCl (2 mL, 1 mol/L), the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product **3ah** (95 mg, 72%).



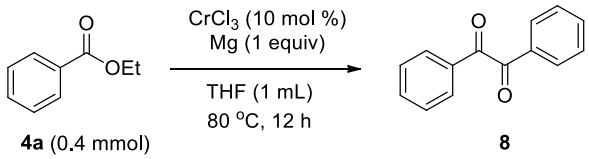
3ah

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.20 (br s, 1H), 7.48 (d, *J* = 8.6 Hz, 2H), 7.38 (d, *J* = 8.6 Hz, 2H), 7.19 (s, 1H), 7.15 (dd, *J* = 8.6, 2.3 Hz, 1H), 6.76 (d, *J* = 8.6 Hz, 1H), 4.58 (s, 2H), 2.33 (s, 3H), 1.32 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 166.0, 154.0, 148.2, 134.2, 131.1, 128.6, 127.2, 127.1, 126.1, 120.0, 113.2, 68.2, 34.6, 31.5, 16.4. HRMS (ESI$^+$): Calcd for $C_{19}H_{22}O_2NClNa$ $[M+Na]^+$ 354.1237, Found: 354.1239.

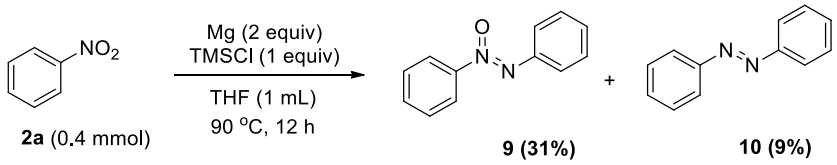
5. Mechanistic Studies



A dried Schlenk tube was charged with $CrCl_3$ (6 mg, 0.04 mmol), Mg (2 mg, 0.2 mmol), followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen. After stirring at 80 °C for 2 h. then filtrate, a mixture of **4a** (120 mg, 0.8 mmol), **2a** (49 mg, 0.4 mmol), Mg (134 mg, 1.4 mmol), dtbpy (10 mg, 0.04 mmol), TMSCl (50 μL , 0.4 mmol), THF (1 mL) in a 15 mL glass vial were added. The resulting mixture was stirred at 90 °C for 12 h. After quenched by HCl (2 mL), the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product **5a**(76%).

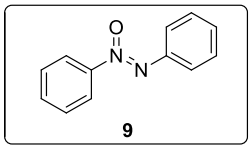


A dried Schlenk tube was charged with **4a** (60 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (10 mg, 0.4 mmol), followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 80 °C for 12 h. After quenched by HCl (2 mL, 1 mol/L), the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product **8** (13 mg, 30%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.98 (dd, *J* = 8.2, 1.0 Hz, 4H), 7.69–7.62 (m, 2H), 7.51 (t, *J* = 7.7 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 194.7, 135.0, 133.1, 130.0, 129.2.



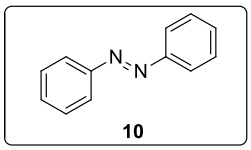
A dried Schlenk tube was charged with **2a** (49 mg, 0.4 mmol), Mg (19 mg, 0.8 mmol), TMSCl (50 μL, 0.4 mmol), followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 90 °C for 12 h. After quenched by HCl (2 mL, 1 mol/L),

the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired products **9** (31%) and **10** (9%).



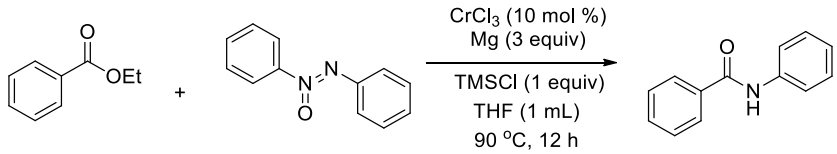
9

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.33 (d, *J* = 7.5 Hz, 2H), 8.19 (d, *J* = 8.0 Hz, 2H), 7.57–7.48 (m, 5H), 7.41 (t, *J* = 7.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 148.5, 144.1, 131.7, 129.7, 128.9, 128.8, 125.7, 122.5. HRMS (ESI^+): Calcd for $C_{12}H_{10}N_2O$ $[M]^+$ 198.0793, Found: 198.0795.



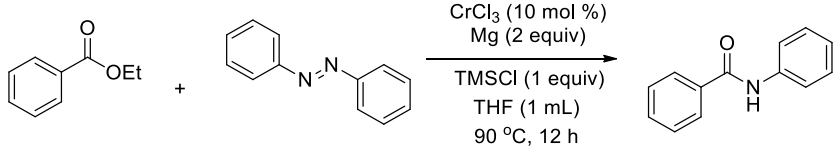
10

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.96–7.90 (m, 4H), 7.56–7.46 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 152.8, 131.1, 129.2, 123.0. HRMS (ESI) calculated for $C_{12}H_{11}N_2$ $[M+H]^+$ 183.0922; found 183.0913.



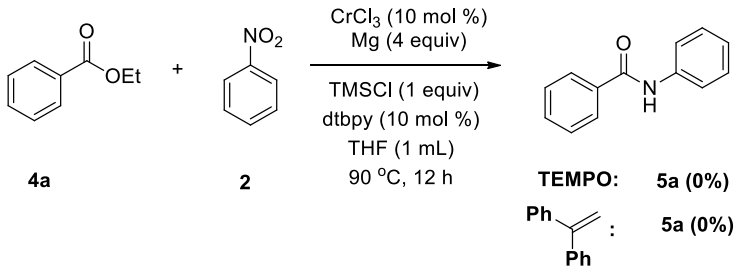
4a **9** **5a,90%**

A dried Schlenk tube was charged with **4a** (120 mg, 0.8 mmol), $CrCl_3$ (6 mg, 0.04 mmol), **9** (40 mg, 0.2 mmol),and TMSCl (50 μL, 0.4 mmol), Mg (29 mg, 1.2 mmol), dtbpy (10 mg, 0.04mmol), followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 90 °C for 12 h. After quenched by HCl (2 mL, 1 mol/L), the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product **5a**.

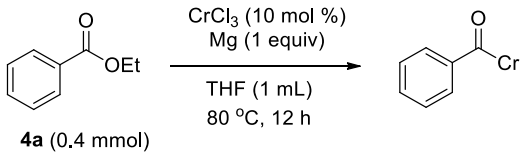


4a **10** **5a,89%**

A dried Schlenk tube was charged with **4a** (120 mg, 0.8 mmol), $CrCl_3$ (6 mg, 0.04 mmol), **10** (36 mg, 0.2 mmol), TMSCl (50 μL, 0.4 mmol), Mg (19 mg, 0.8 mmol), dtbpy (10 mg, 0.04 mmol), followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 90 ^oC for 12 h. After quenched by HCl (2 mL, 1 mol/L), the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by flash chromatography on silica gel (gradient eluent of EtOAc in petroleum ether) to give the desired product **5a**.



Two dried Schlenk tube were charged with **4a** (120 mg, 0.8 mmol), $CrCl_3$ (6 mg, 0.04 mmol), **2** (49 mg, 0.4 mmol), TMSCl (50 μL, 0.4 mmol), Mg (38 mg, 1.6 mmol), dtbpy (10 mg, 0.04 mmol), 2,2,6,6-tetramethylpiperidin-1-oxy (TEMPO) (125 mg, 0.8 mmol) or 1,1-diphenylethylene (144 mg, 0.8 mmol) followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 90 ^oC for 12 h. After quenched by HCl (2 mL, 1 mol/L), the crude product was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under vacuum. Direct detection by TLC or GC-Ms, no desired product.



A dried Schlenk tube was charged with **4a** (60 mg, 0.4 mmol), $CrCl_3$ (6 mg, 0.04 mmol), Mg (10 mg, 0.4 mmol), followed by adding a freshly distilled THF (1 mL) by syringe under atmosphere of nitrogen, the reaction mixture was stirred at 80 ^oC for 12 h. The resulting mixture was then measured by EPR studies.

Electron paramagnetic resonance (EPR) spectrum (Figure 1**)**

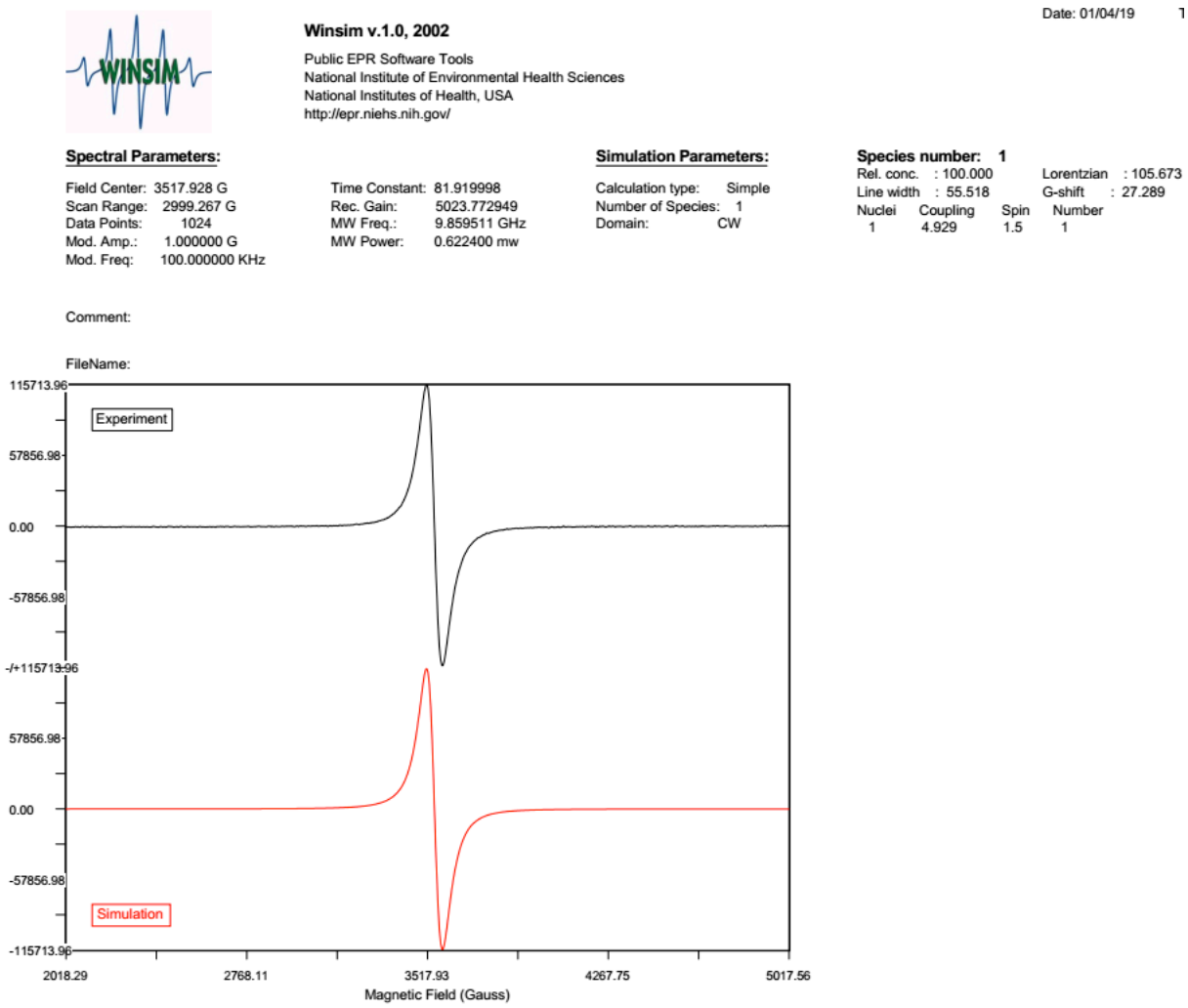
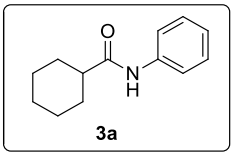
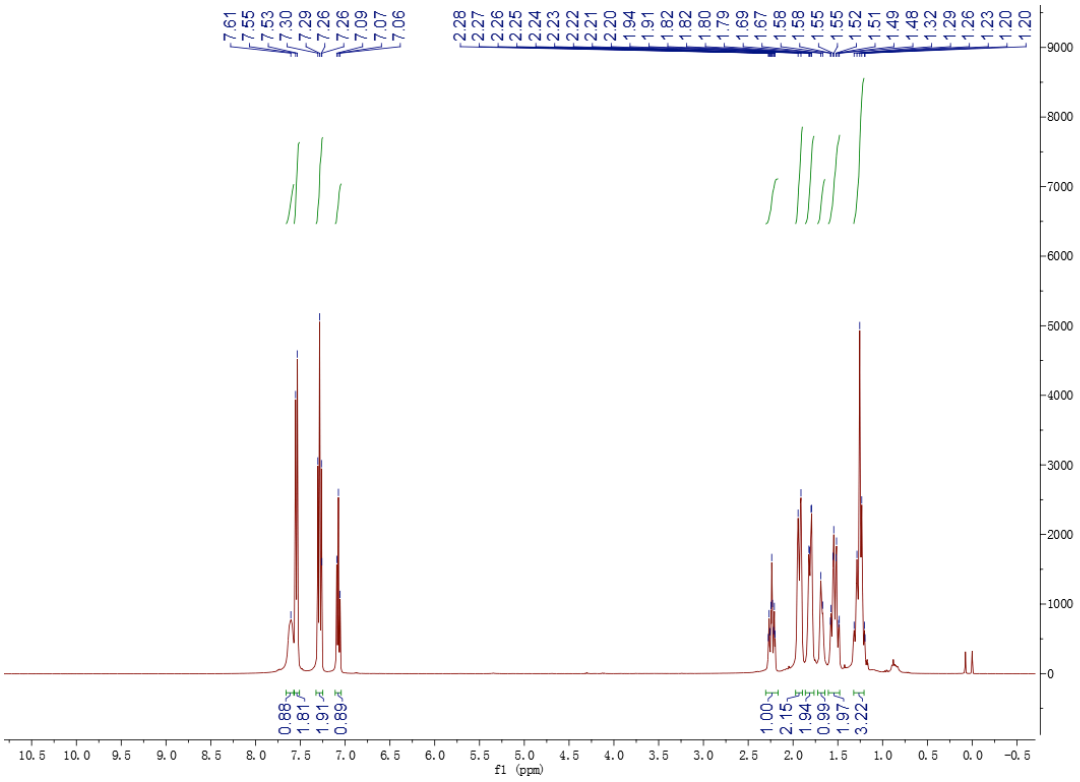


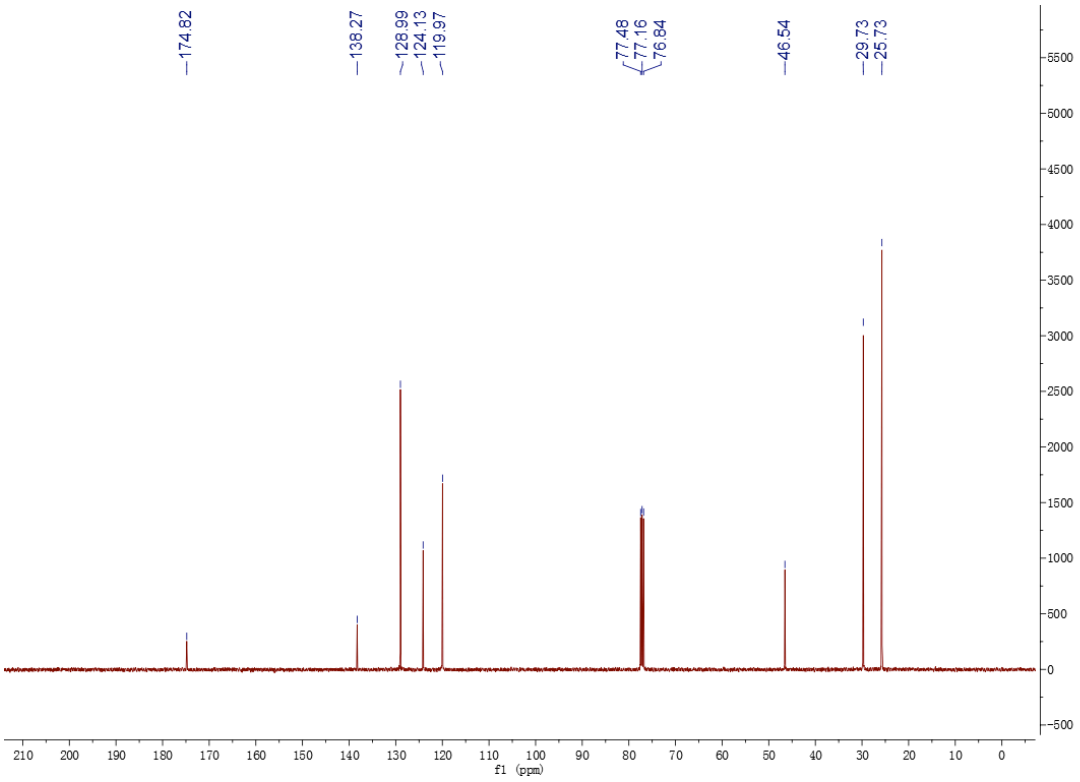
Figure 1. EPR studies of stoichiometric reactions.

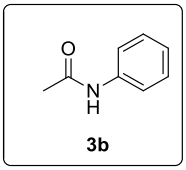
6. ^{1}H, ^{13}C and ^{19}F NMR spectra



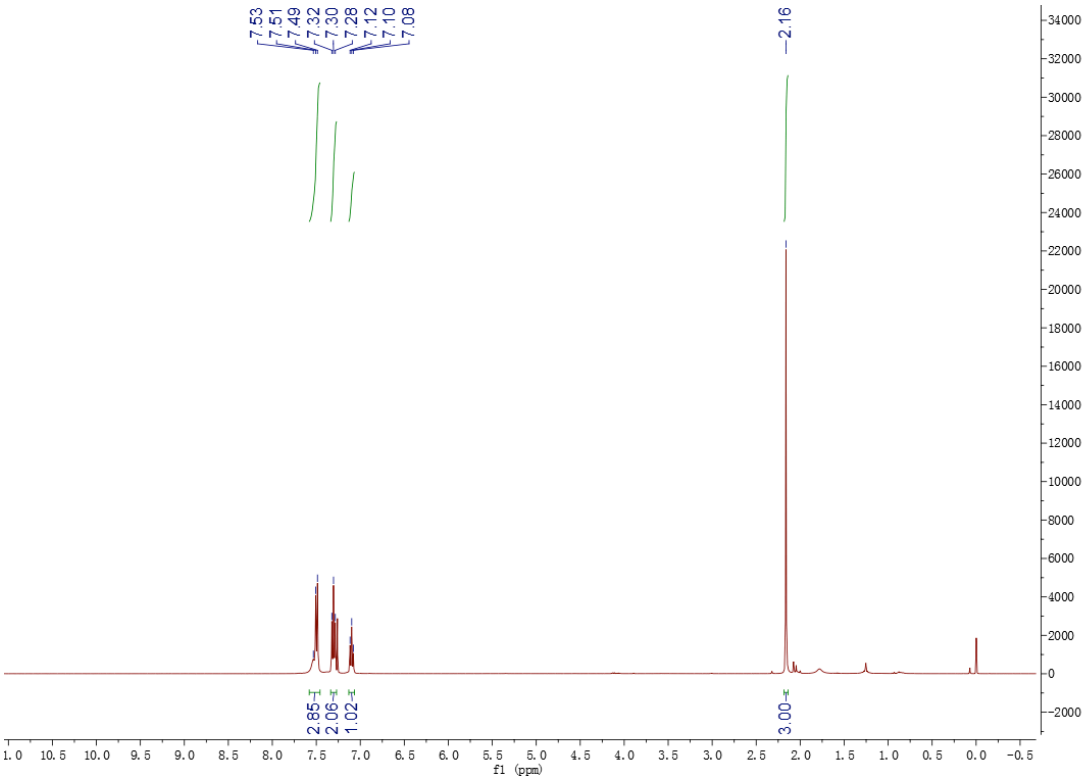
3a






O
N
H

3b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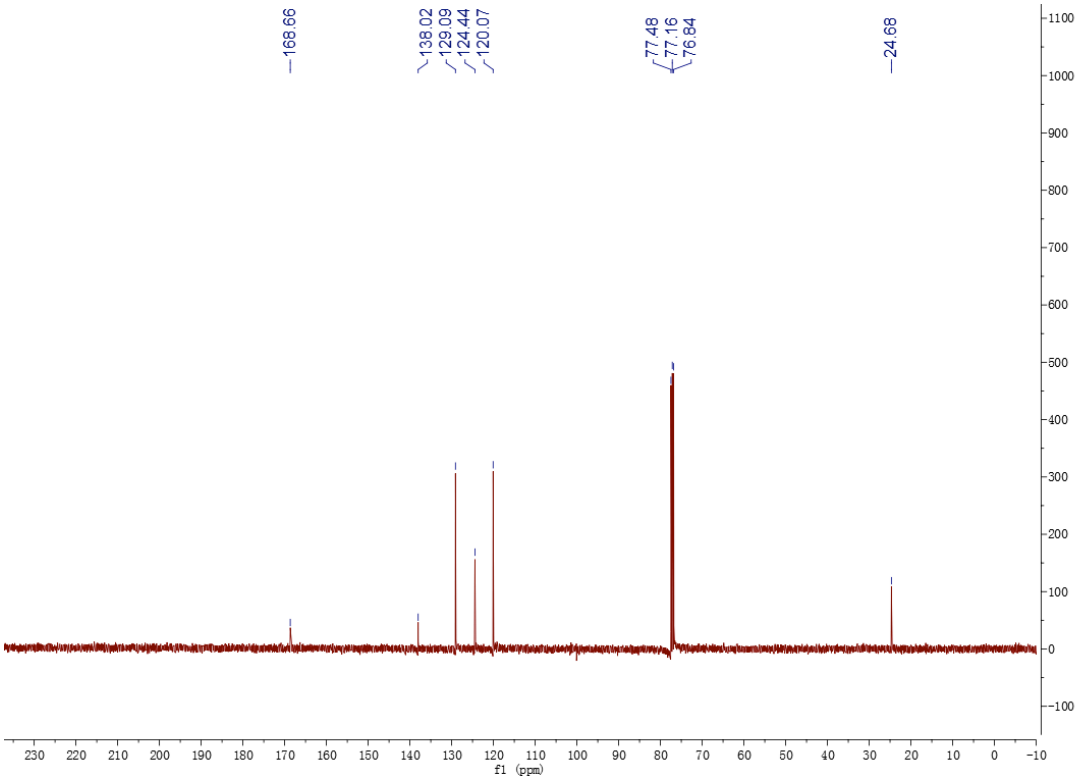
2.85
2.06
1.02
3.00
f1 (ppm)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120.07
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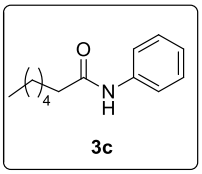
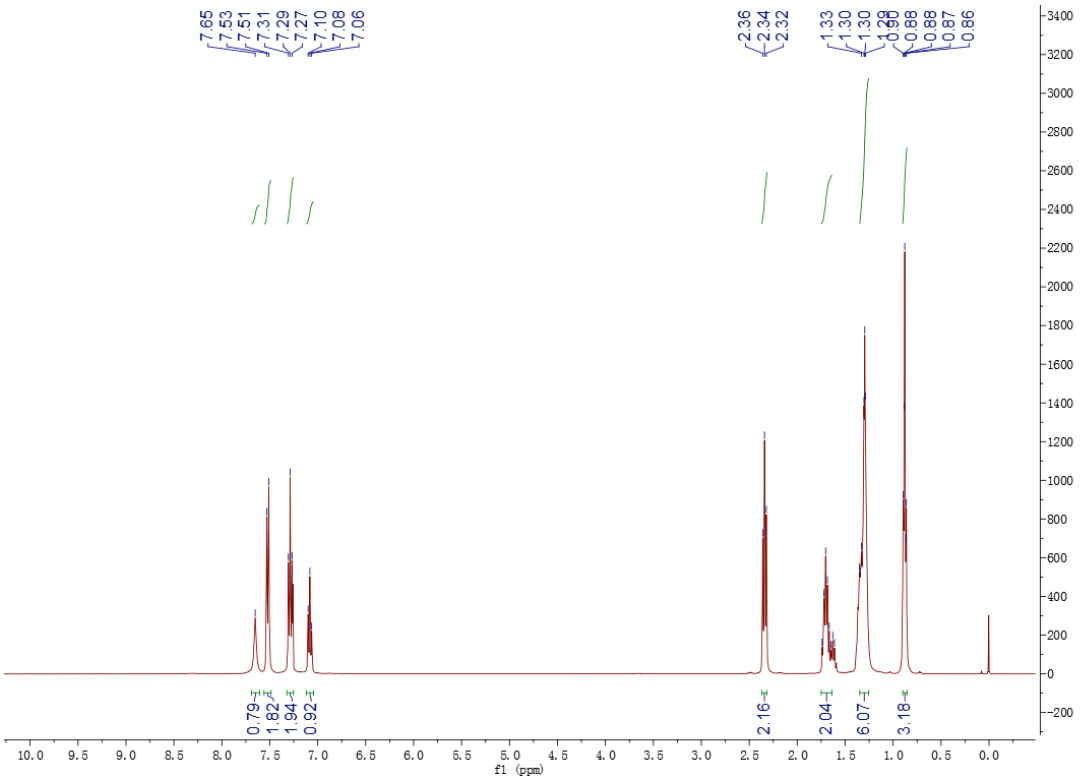
77.16
76.84
24.68
f1 (ppm)


O
N
H
4

3c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7.06
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2.34
2.32
1.33
1.30
1.30
0.90
0.88
0.88
0.87
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0.79
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1.94
0.92
2.16
2.04
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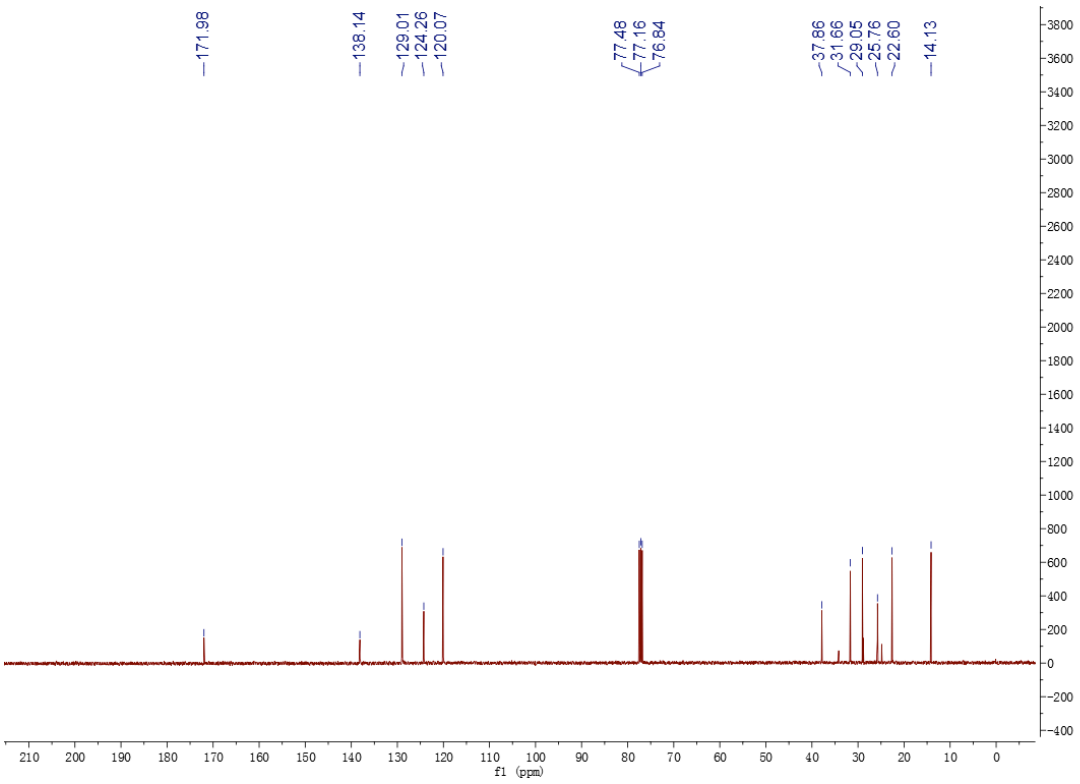
f1 (ppm)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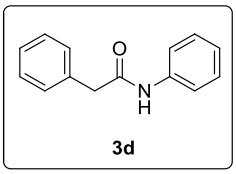
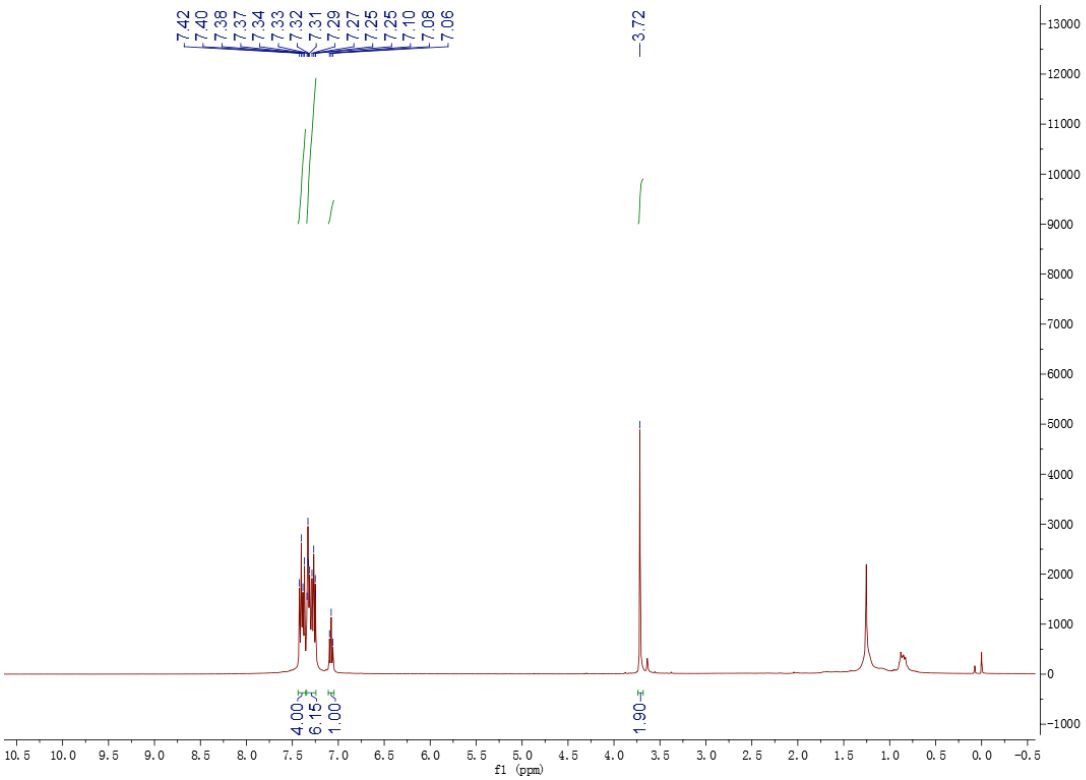
29.05
25.76
22.60
14.13
f1 (ppm)


O
N
H

3d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4.00
6.15
1.00
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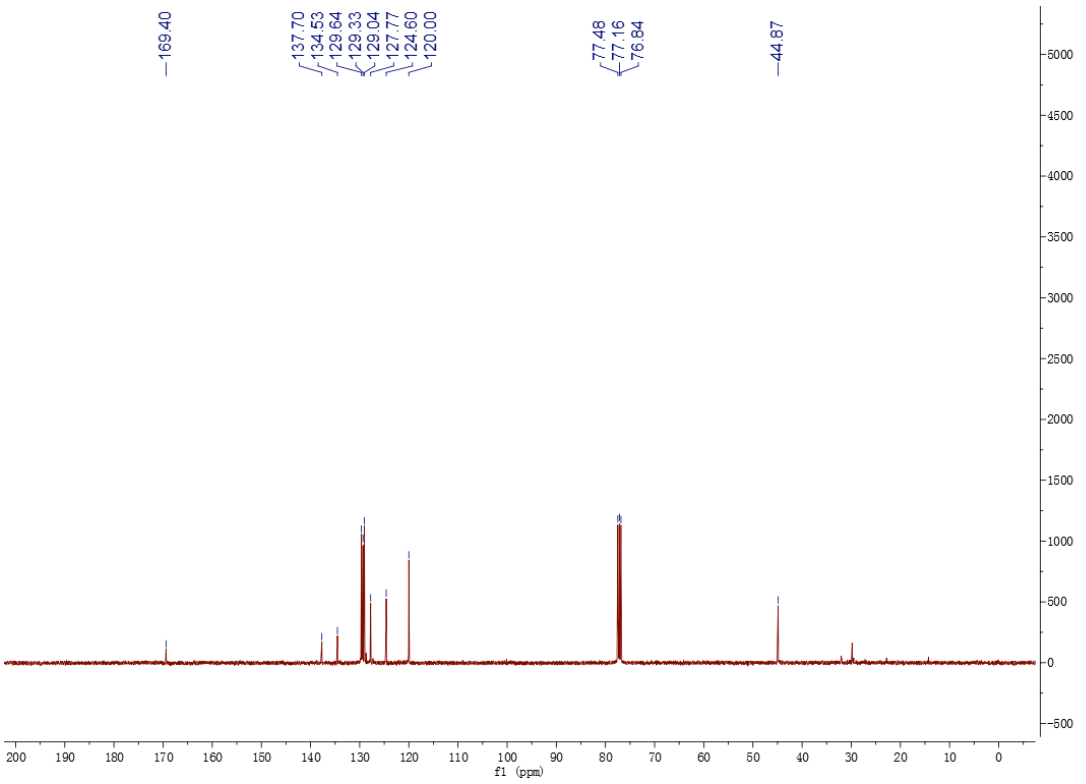
f1 (ppm)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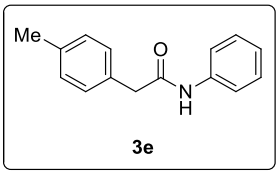
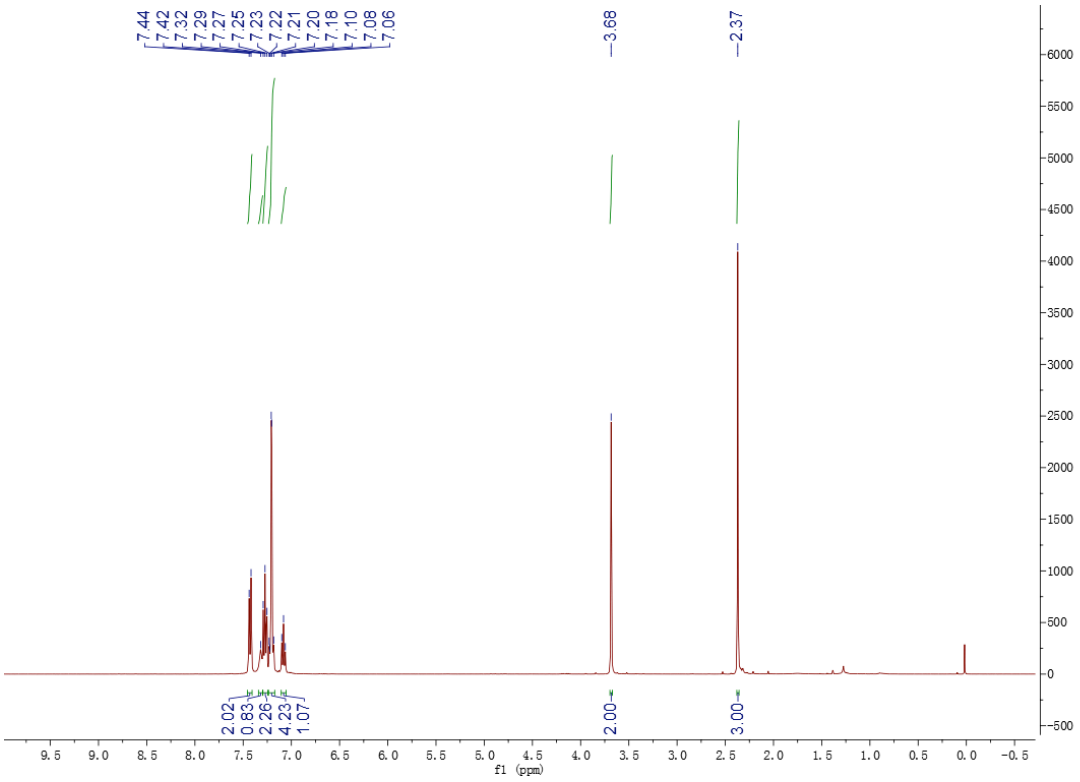
f1 (ppm)


Me
O
N
H

3e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7.22
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7.20
7.18
7.10
7.08
7.06
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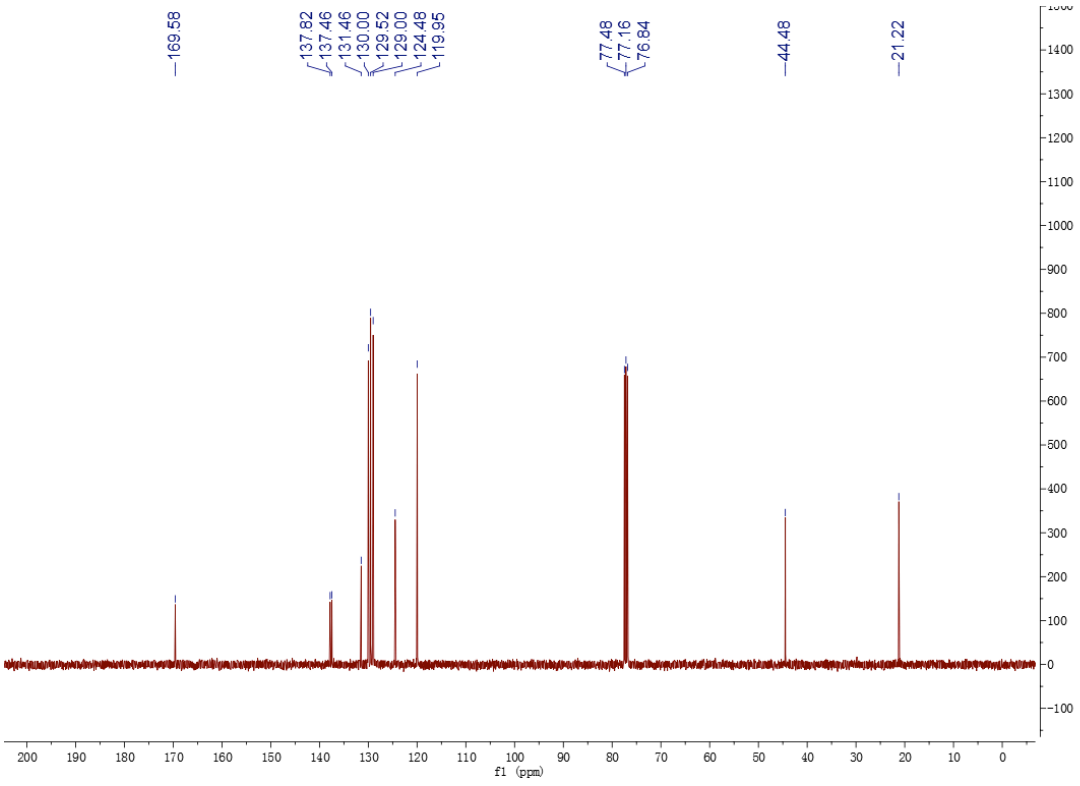
0.83
2.26
4.23
1.07
2.00
3.00
f1 (ppm)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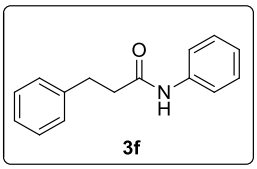
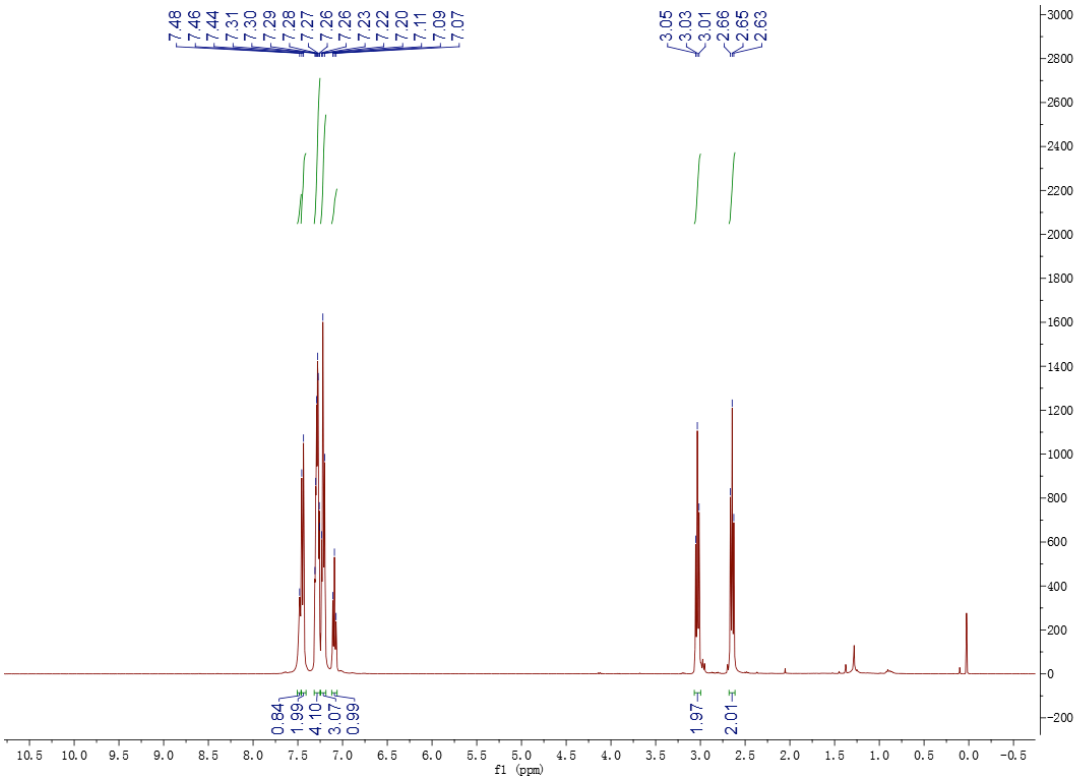
44.48
21.22
f1 (ppm)


O
N
H

3f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7.07
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3.03
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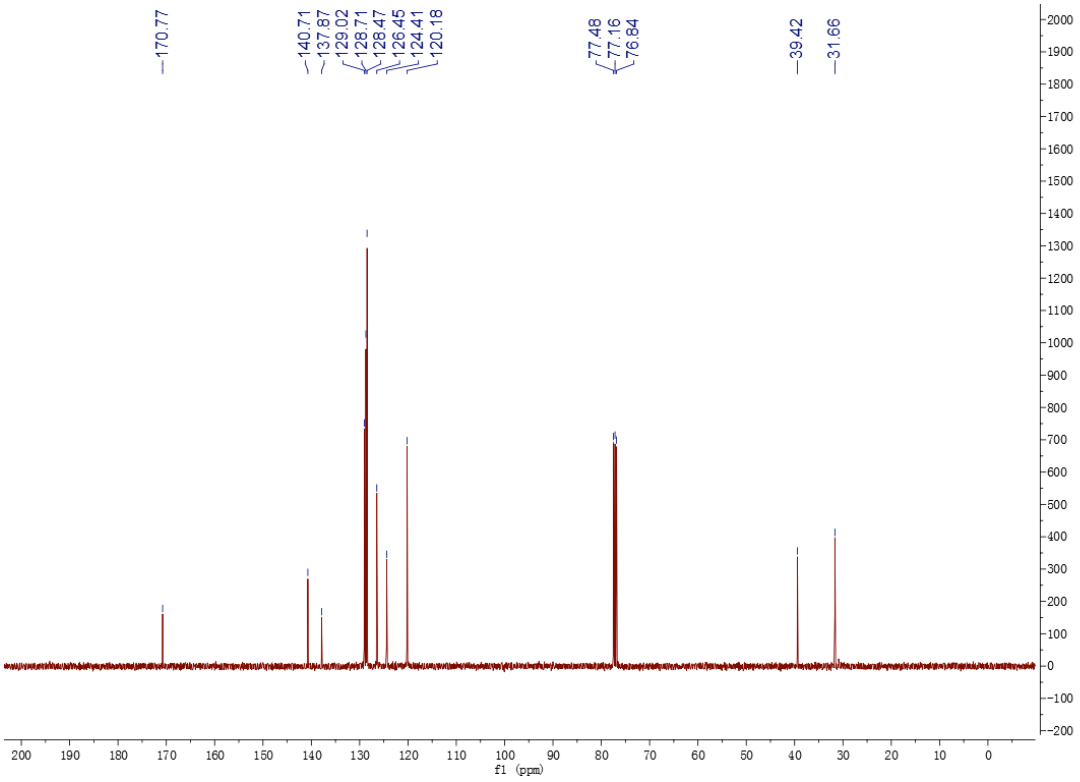
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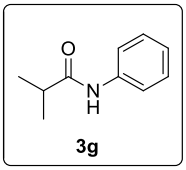
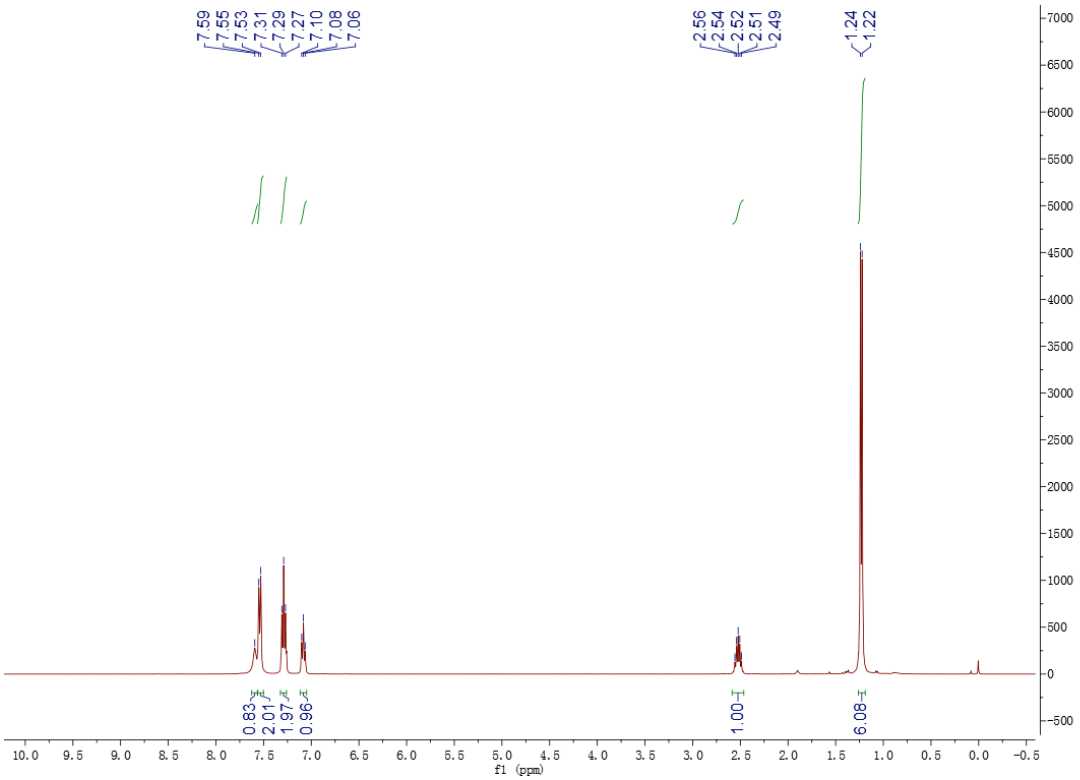

O
N
H

3g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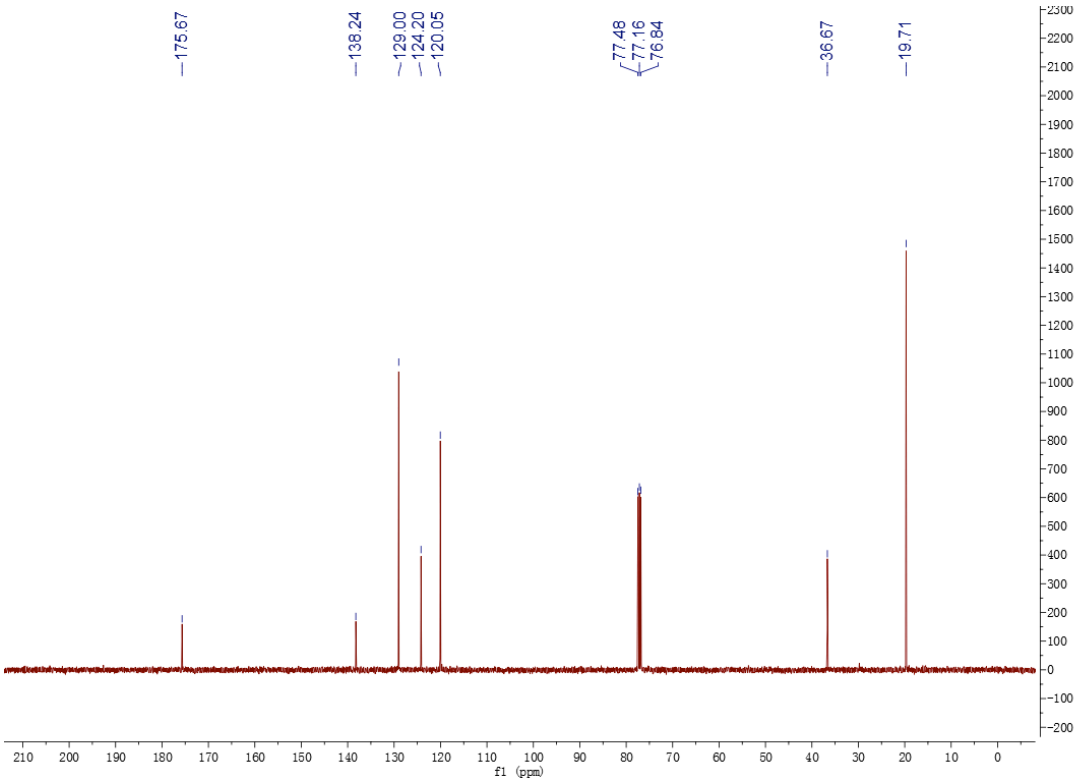
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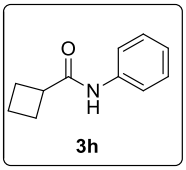
O
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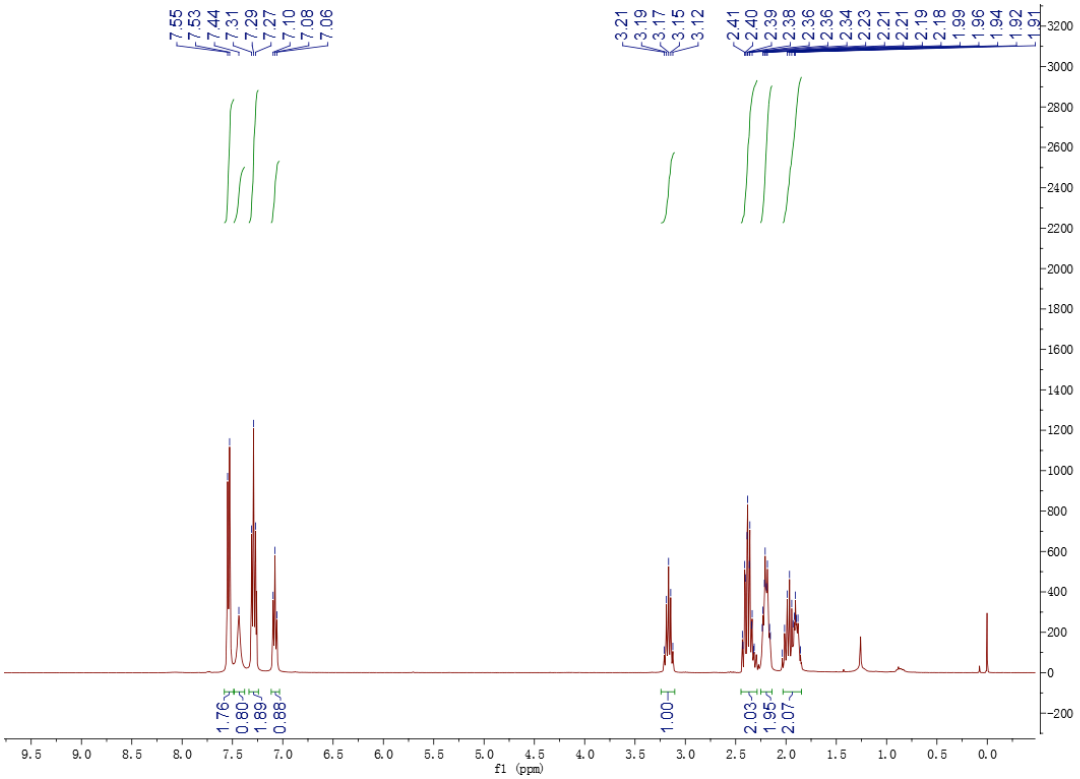
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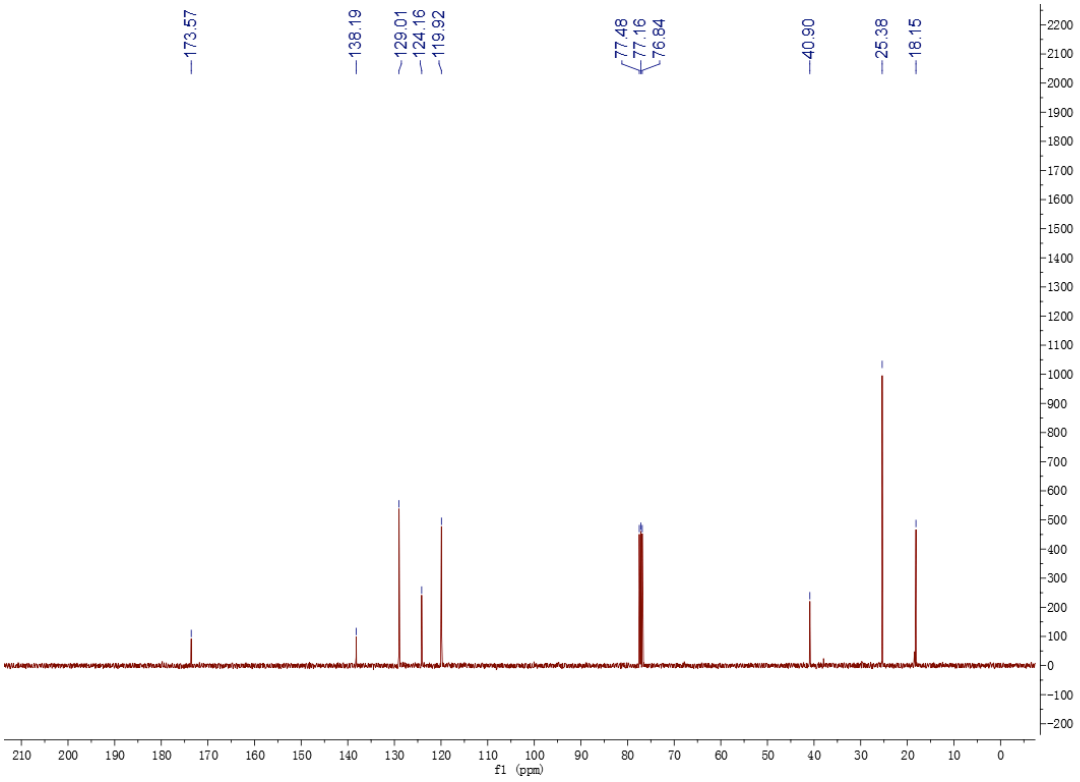
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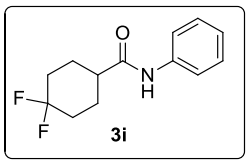
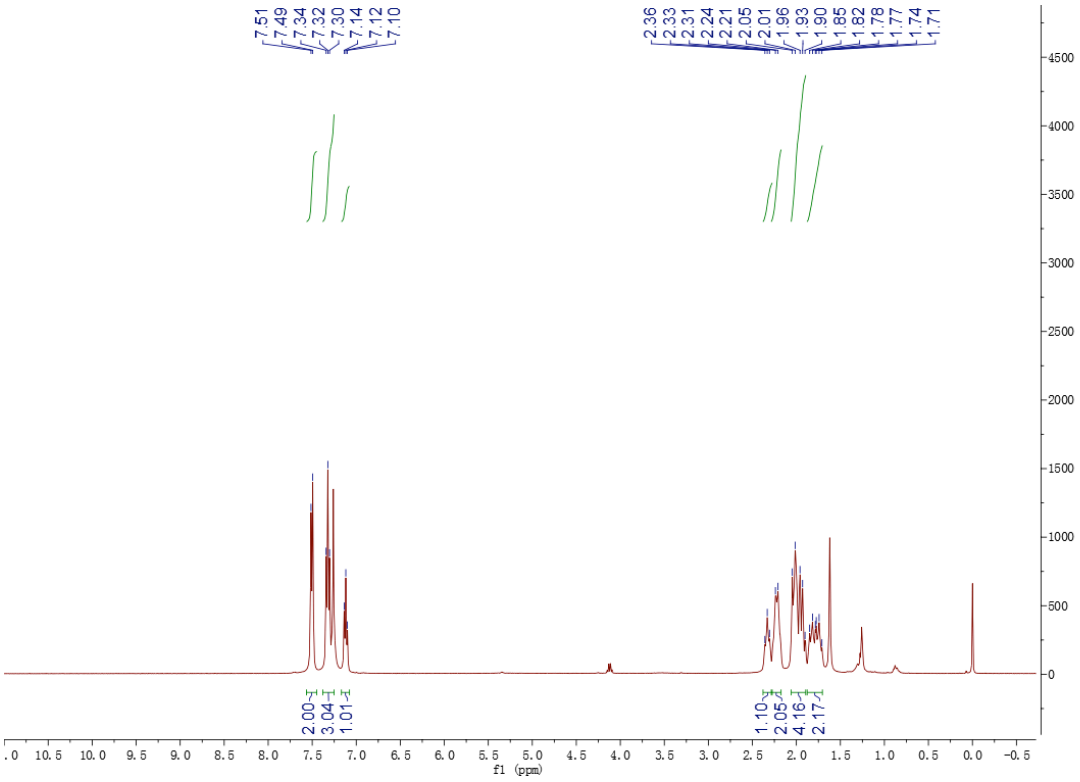

O
N
H
F
F

3i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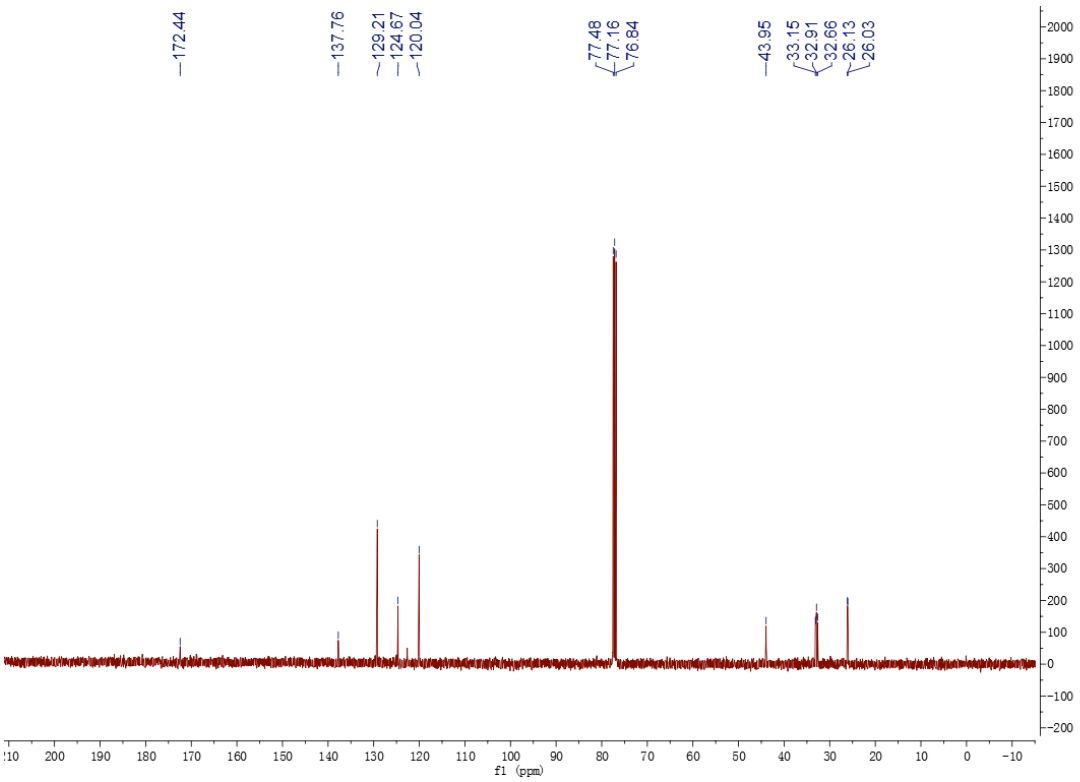
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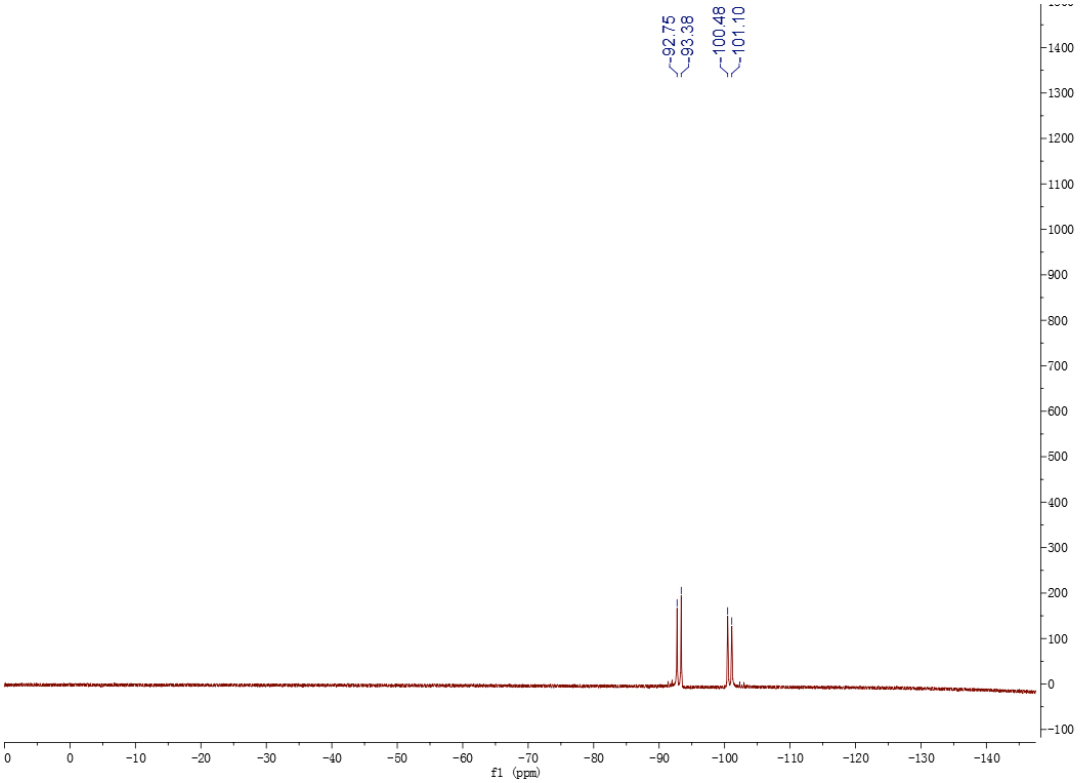
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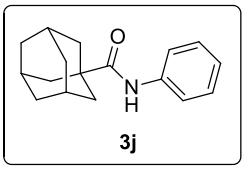
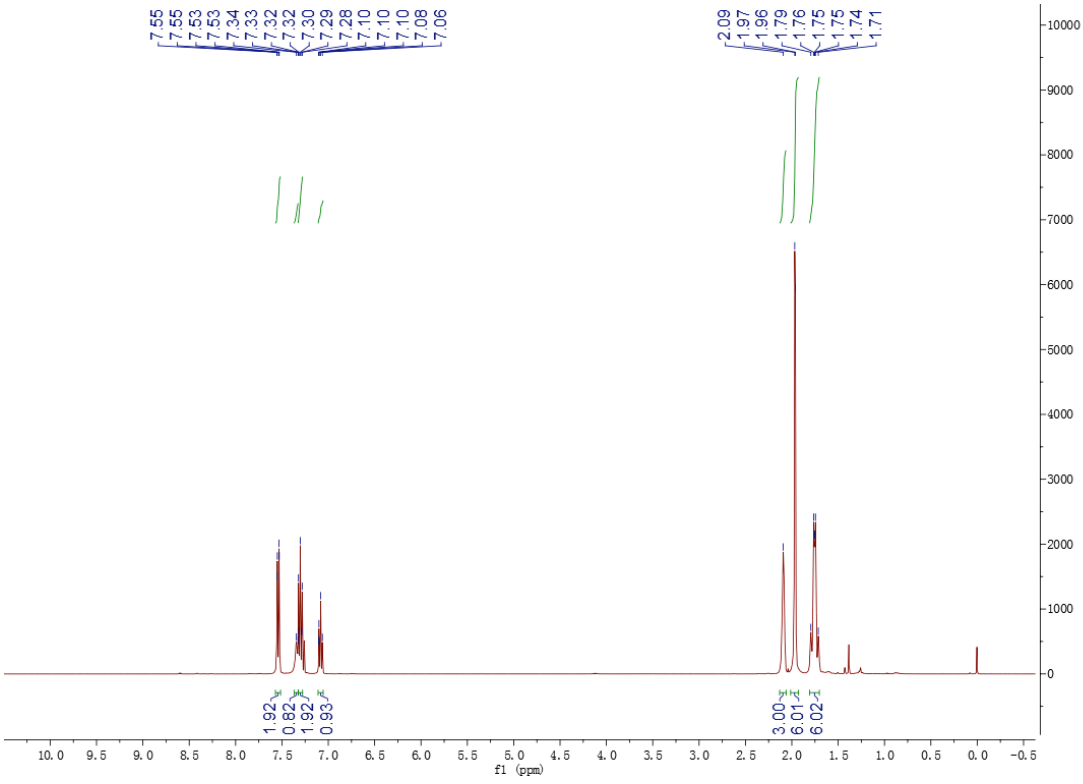

O
N
H

3j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7.08
7.06
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1.76
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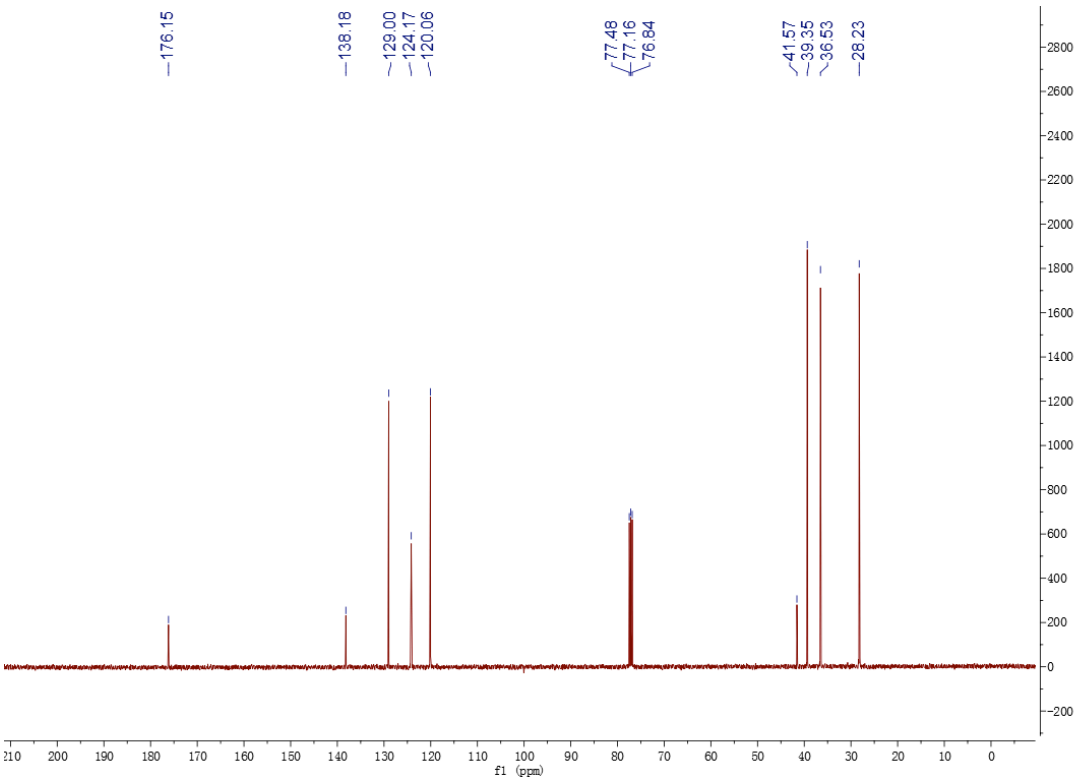
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77.48
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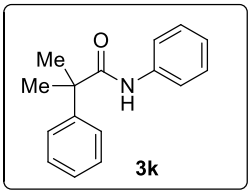
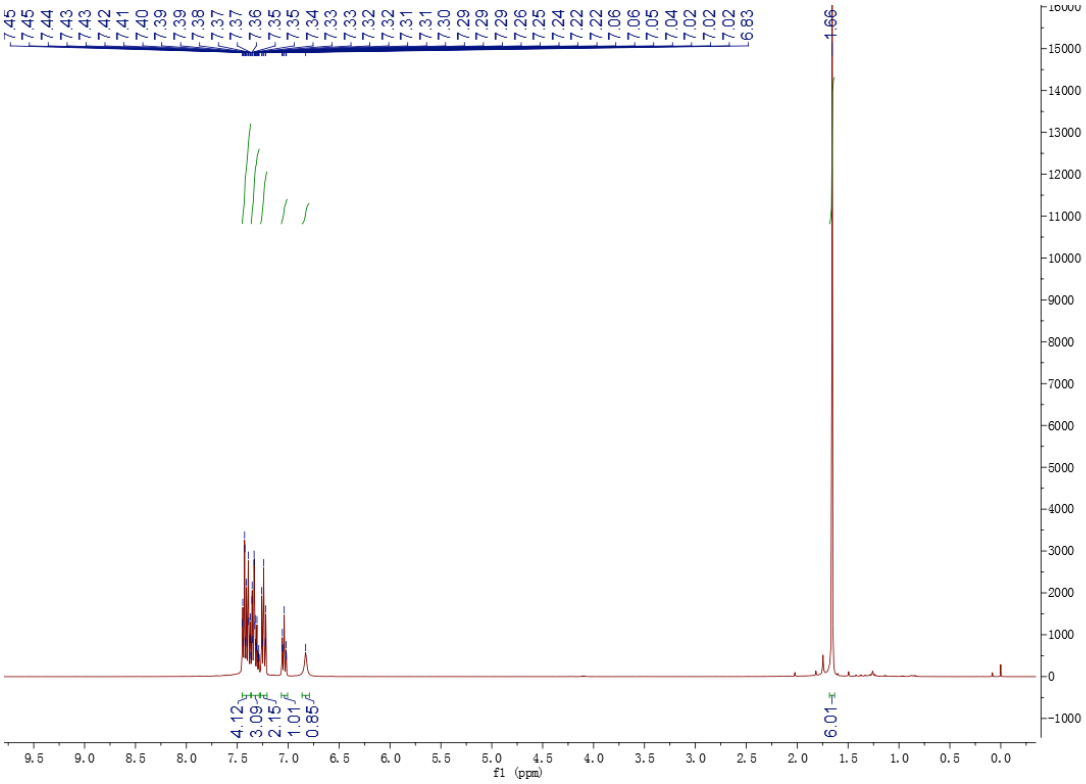
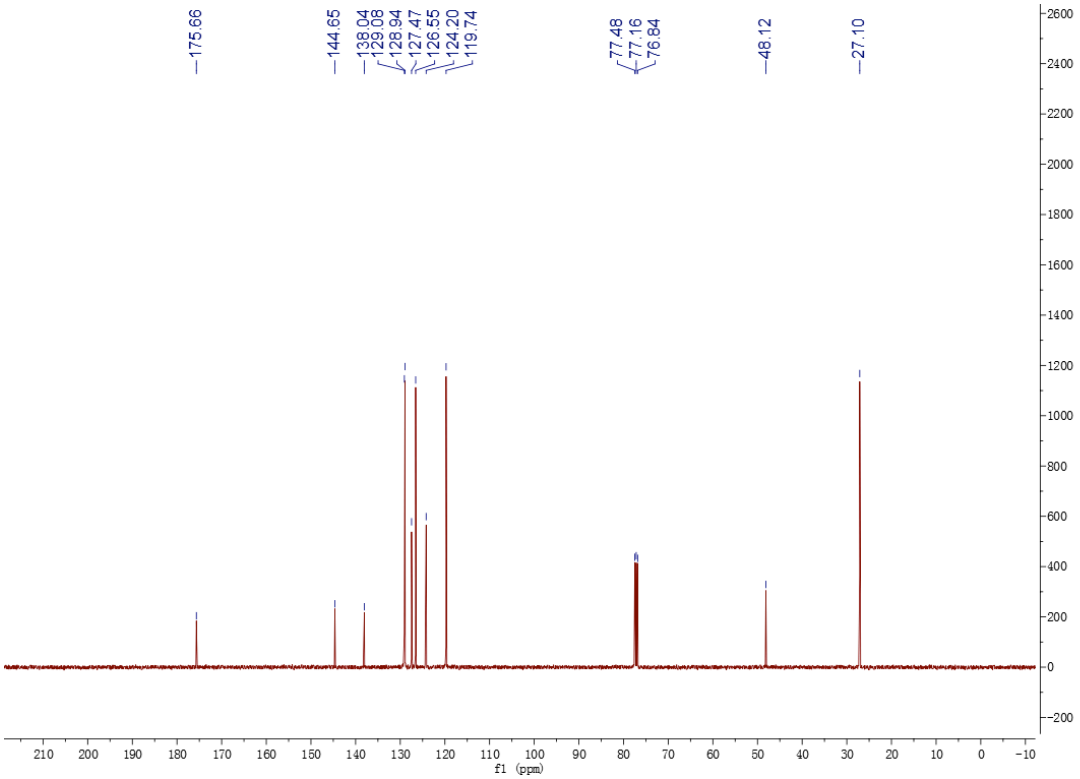
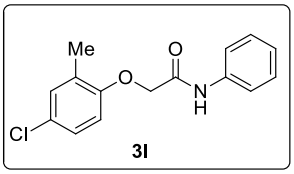
41.57
39.35
36.53
28.23
f1 (ppm)

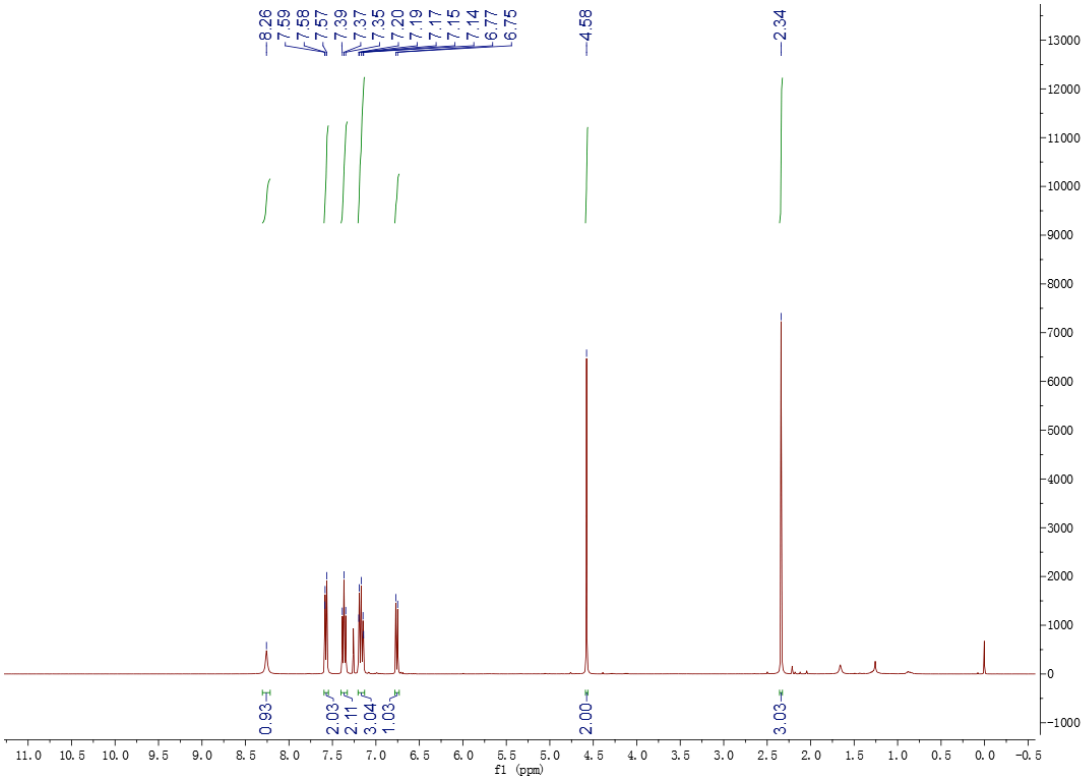

Me
Me
O
N
H
3k

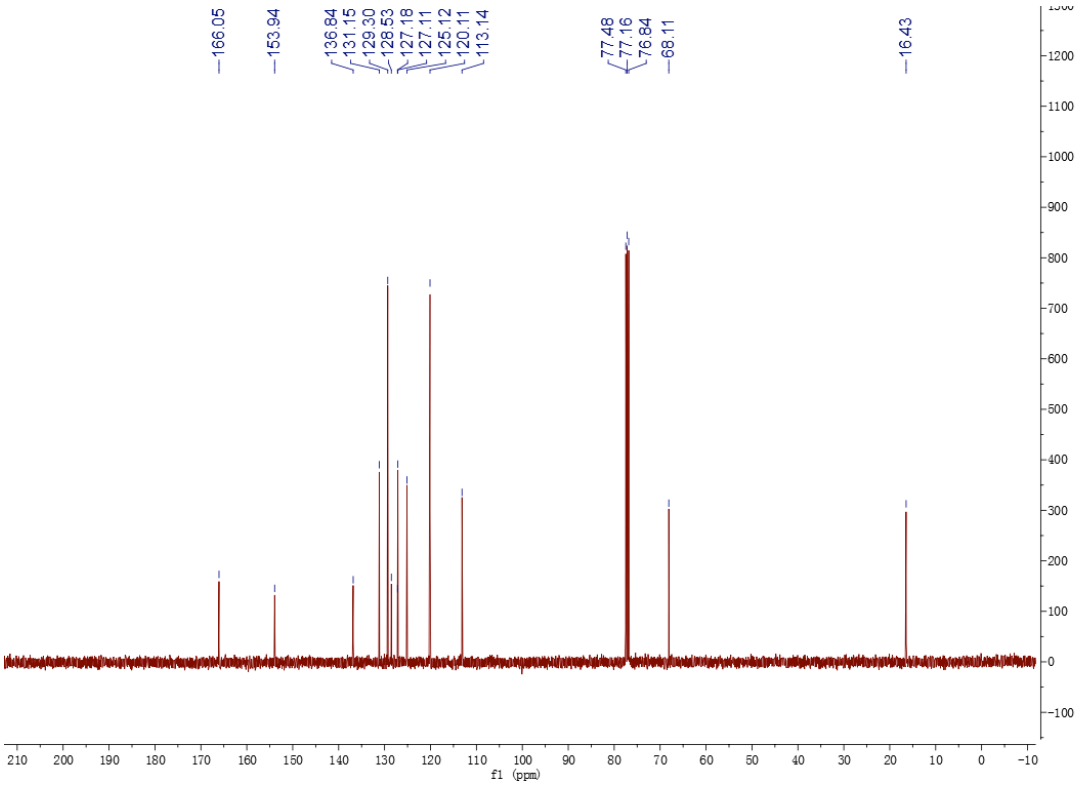

1.66
4.12
3.09
2.15
1.01
0.85
6.01
f1 (ppm)

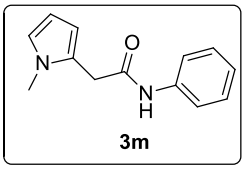

175.66
144.65
138.04
129.08
128.94
127.47
126.55
124.20
119.74
77.48
77.16
76.84
48.12
27.10
f1 (ppm)

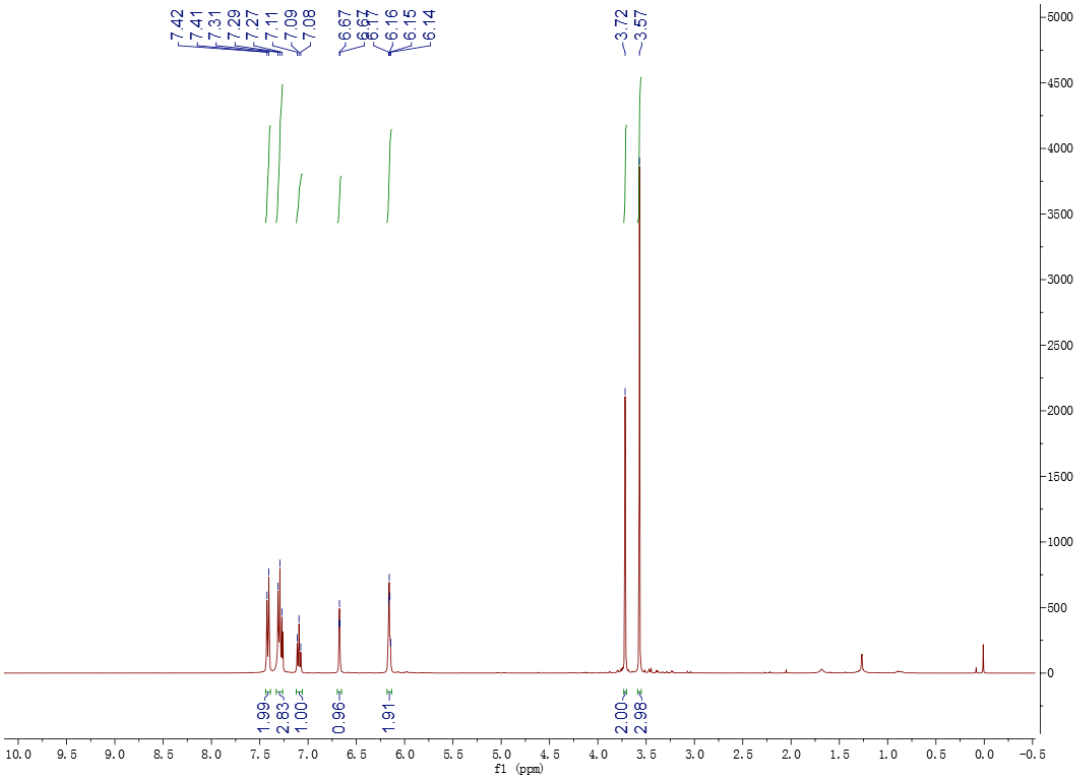

Me
O
O
N
H
Cl

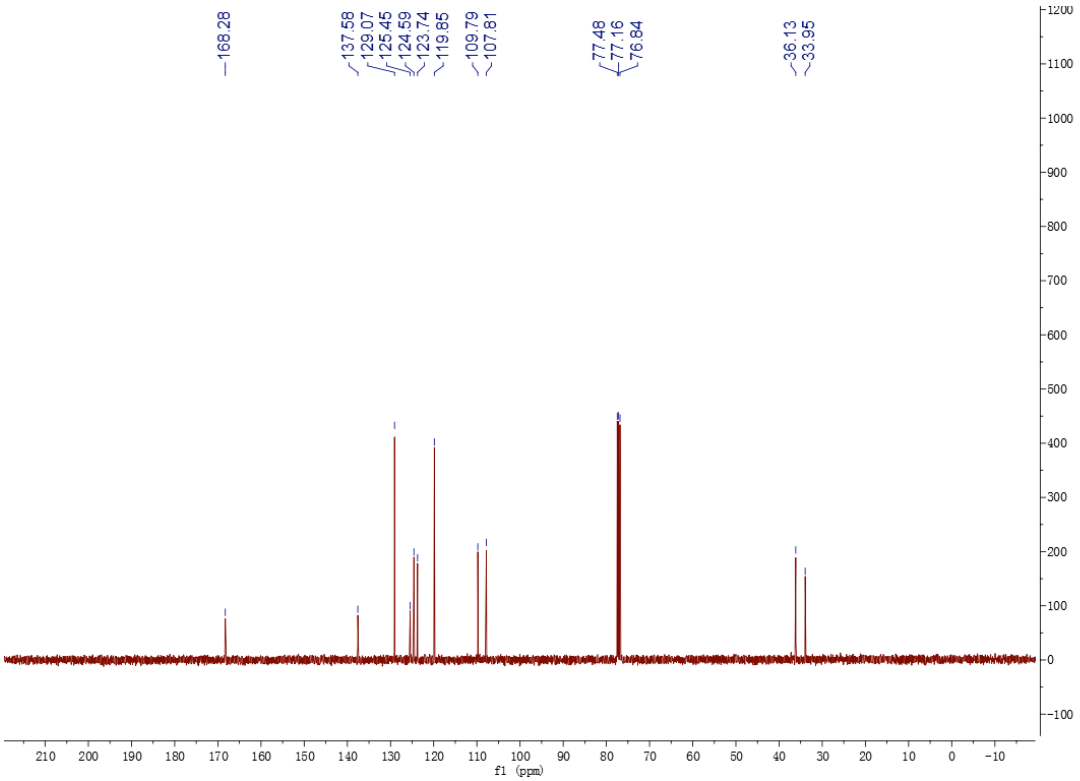
3l

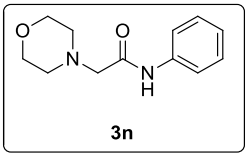

8.26
7.59
7.58
7.57
7.39
7.37
7.35
7.20
7.19
7.17
7.15
7.14
6.77
6.75
4.58
2.34
0.93
2.03
2.11
3.04
1.03
2.00
3.03
f1 (ppm)


166.05
153.94
136.84
131.15
129.30
128.53
127.18
127.11
125.12
120.11
113.14
77.48
77.16
76.84
68.11
16.43
f1 (ppm)

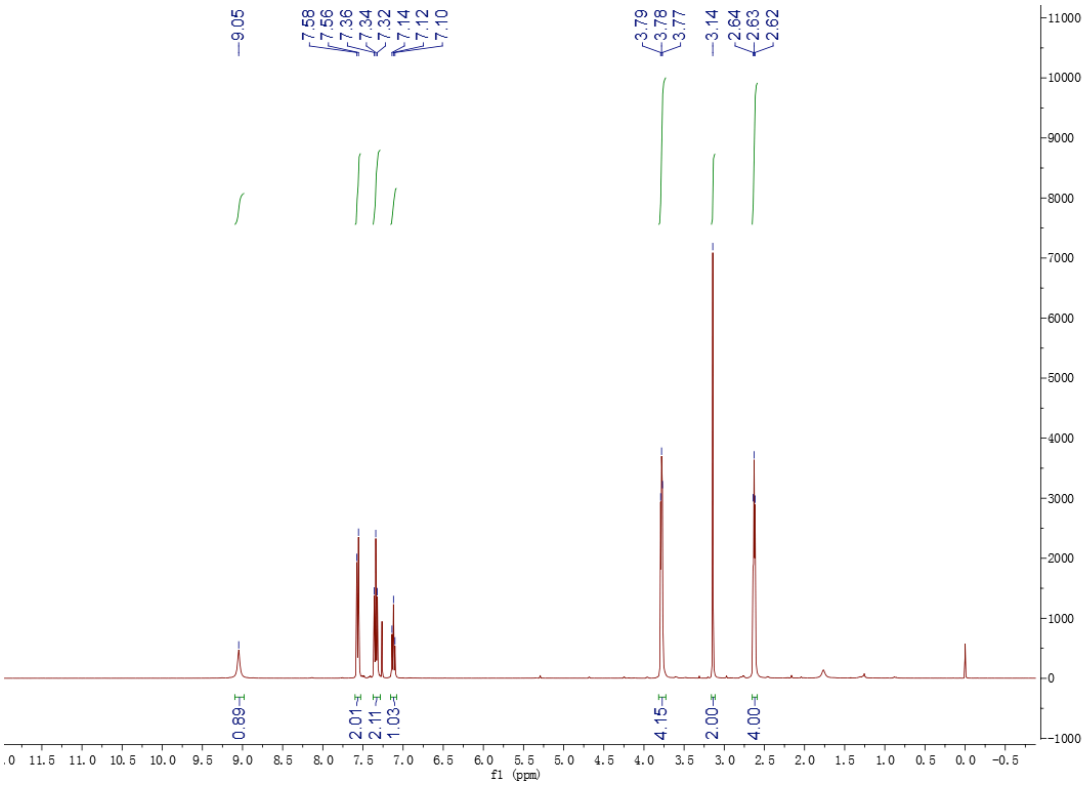

O
N
H
3m

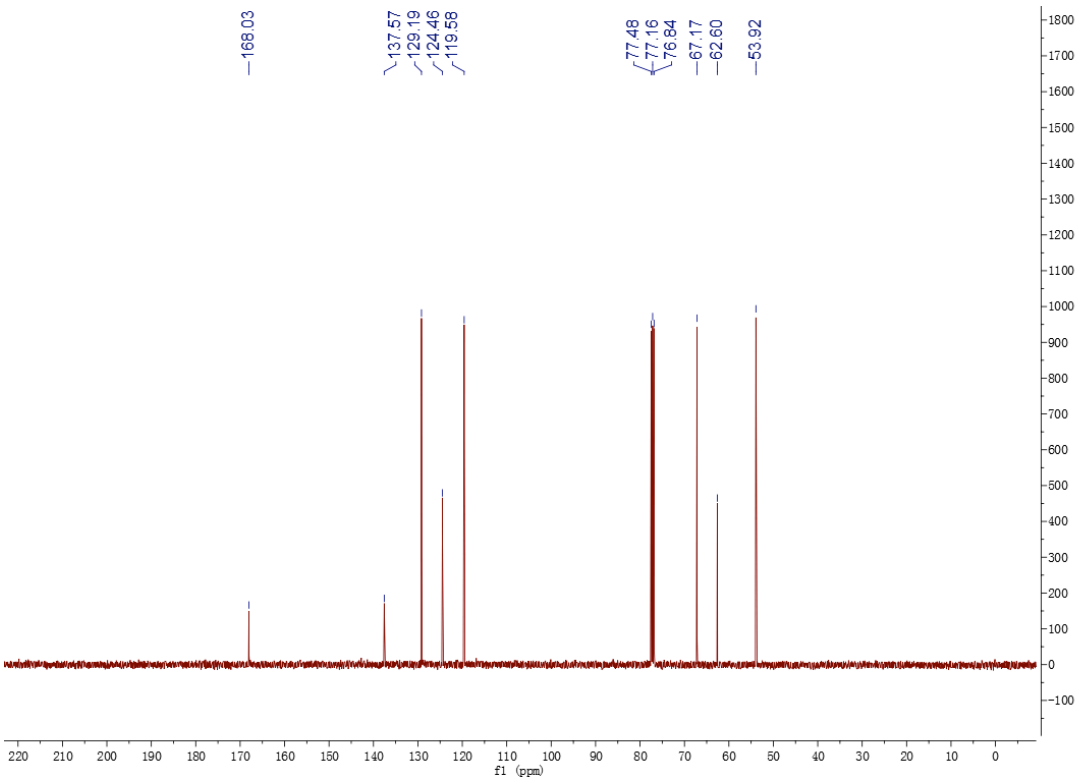

7.42
7.41
7.31
7.29
7.27
7.11
7.09
7.08
6.67
6.17
6.16
6.15
6.14
3.72
3.57
1.99
2.83
1.00
0.96
1.91
2.00
2.98
f1 (ppm)

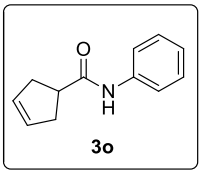

168.28
137.58
129.07
125.45
124.59
123.74
119.85
109.79
107.81
77.48
77.16
76.84
36.13
33.95
f1 (ppm)


O
N
O
N
H

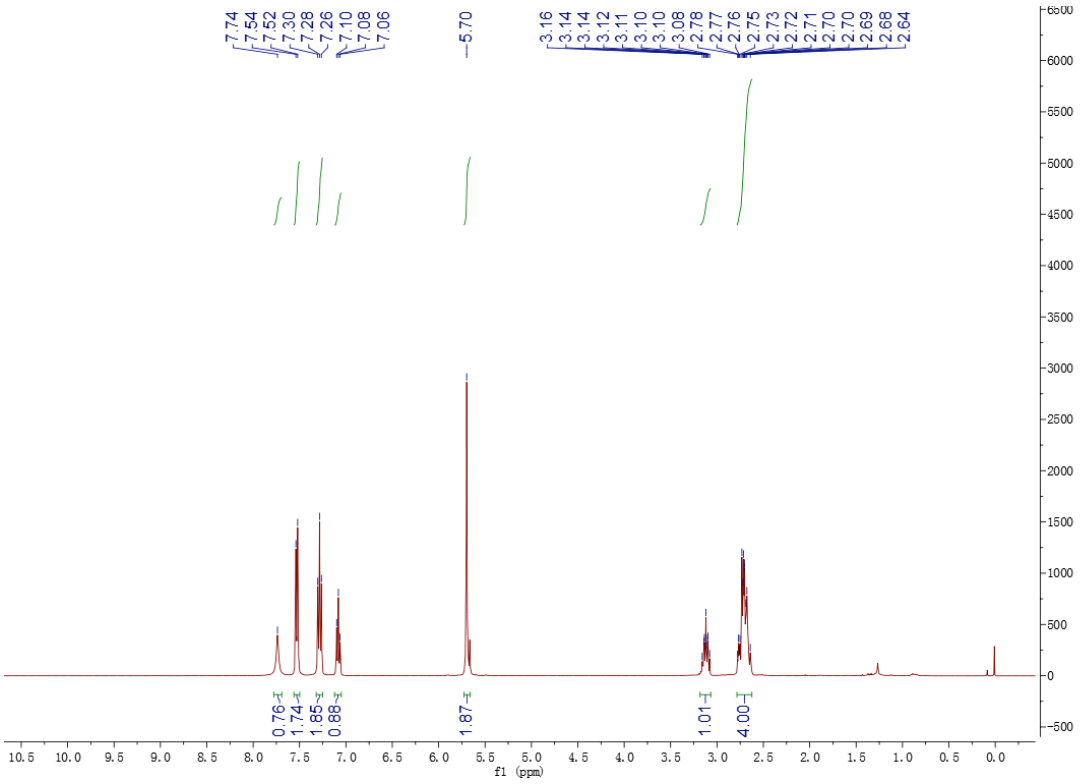
3n

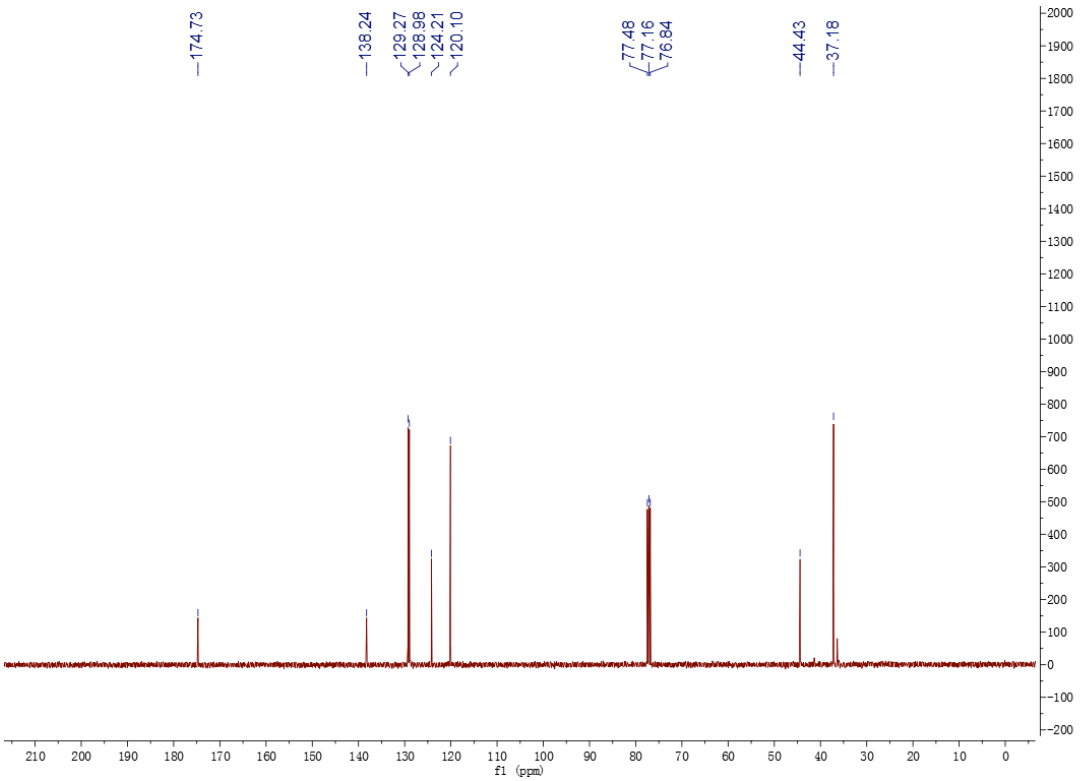

9.05
7.58
7.56
7.36
7.34
7.32
7.14
7.12
7.10
3.79
3.78
3.77
3.14
2.64
2.63
2.62
0.89
2.01
2.11
1.03
4.15
2.00
4.00
f1 (ppm)

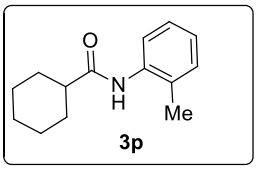

168.03
137.57
129.19
124.46
119.58
77.48
77.16
76.84
67.17
62.60
53.92
f1 (ppm)


O
N
H

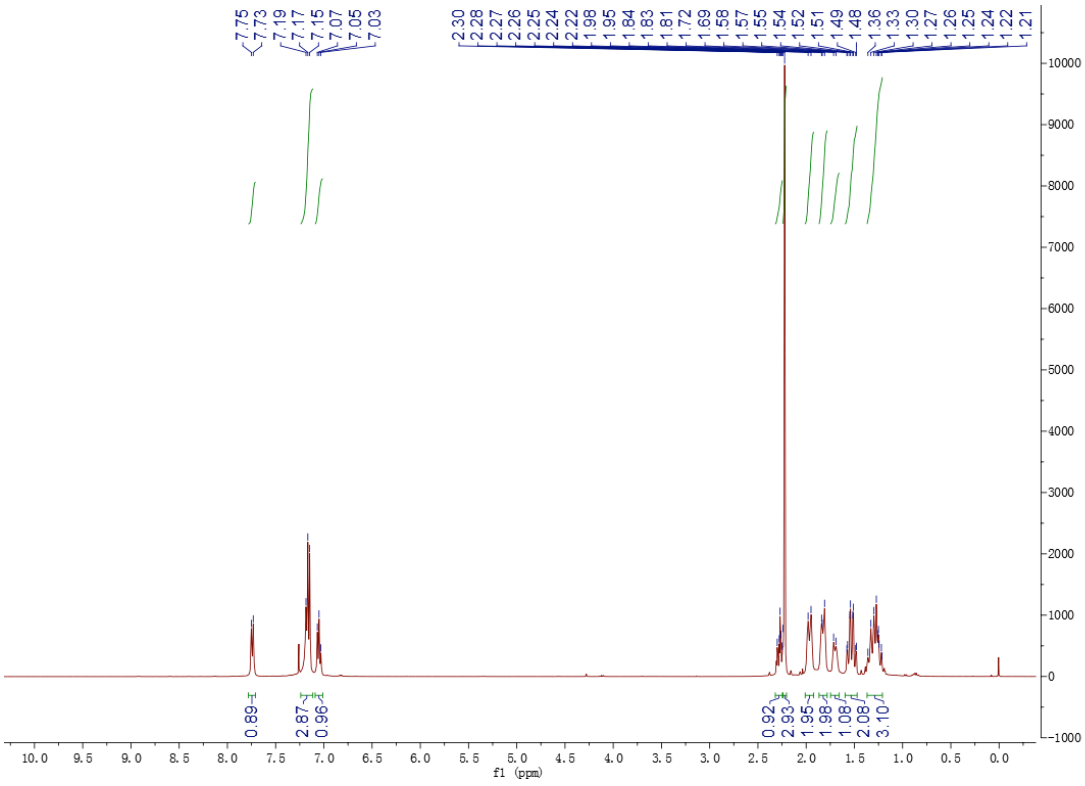
3o

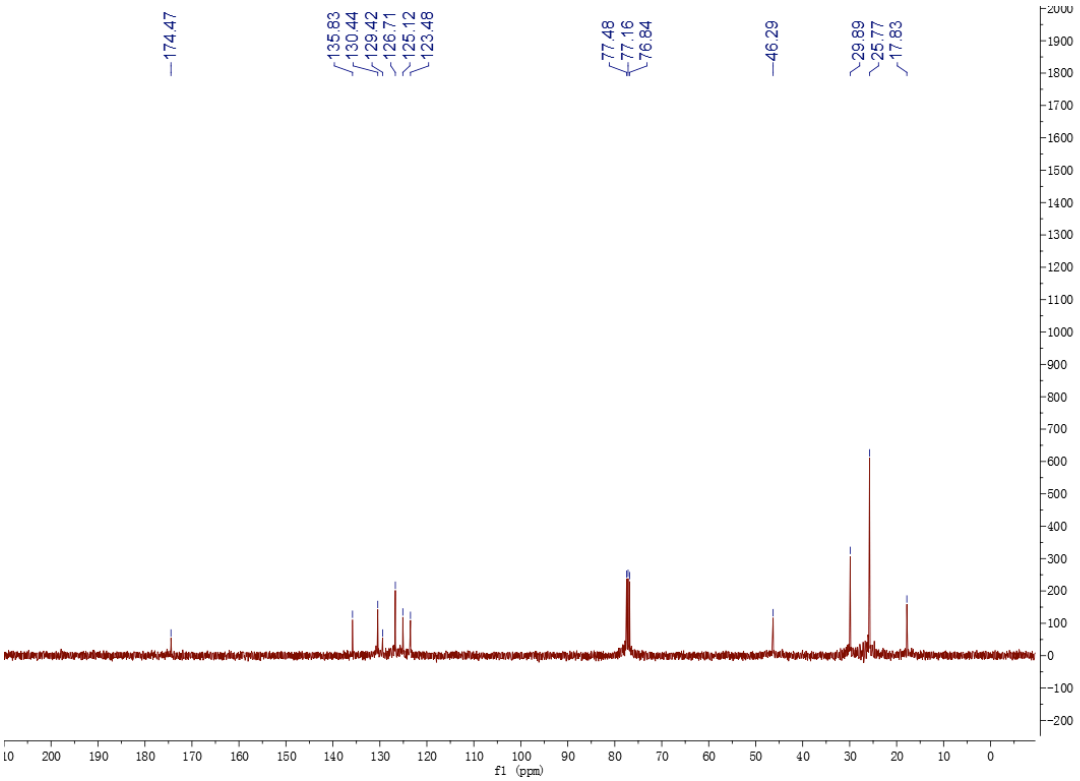

7.74
7.54
7.52
7.30
7.28
7.26
7.10
7.08
7.06
5.70
3.16
3.14
3.14
3.12
3.11
3.10
3.10
3.08
2.78
2.77
2.76
2.75
2.73
2.72
2.71
2.70
2.70
2.69
2.68
2.64
0.76
1.74
1.85
0.88
1.87
1.01
4.00
f1 (ppm)

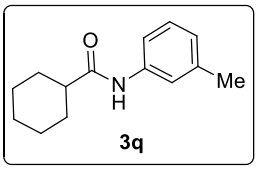

174.73
138.24
129.27
128.98
124.21
120.10
77.48
77.16
76.84
44.43
37.18
f1 (ppm)


O
N
H
Me

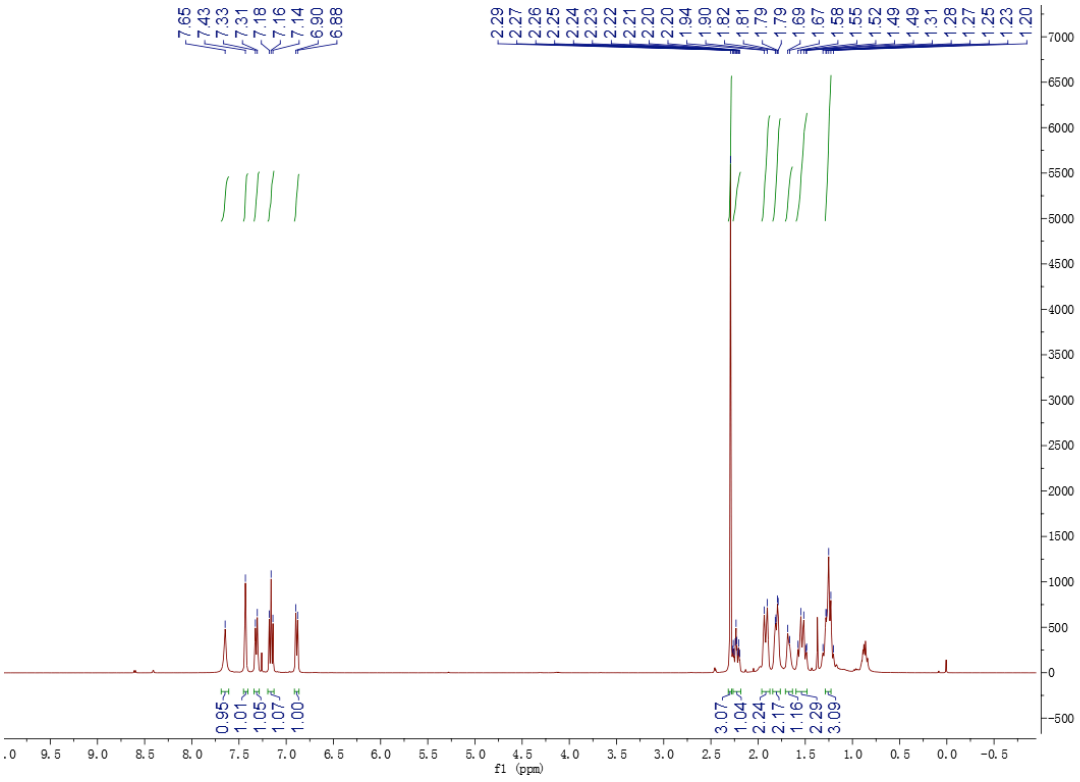
3p

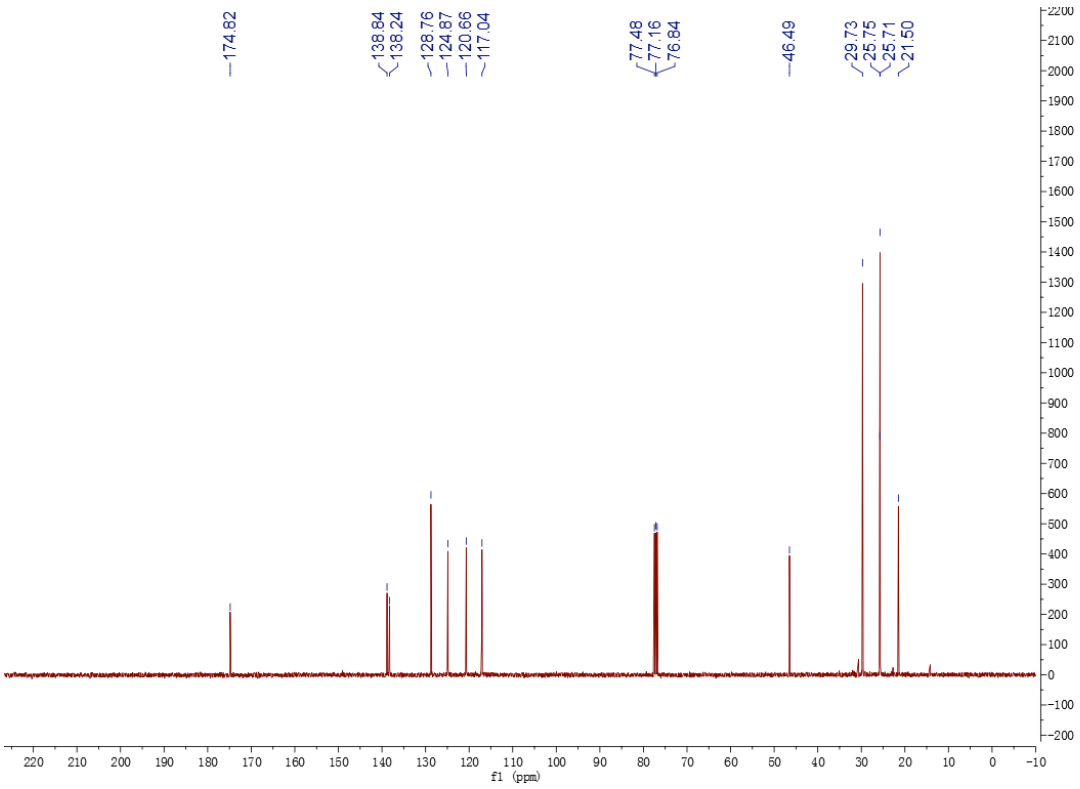

7.75
7.73
7.19
7.17
7.15
7.07
7.05
7.03
2.30
2.28
2.27
2.26
2.25
2.24
2.22
1.98
1.95
1.84
1.83
1.81
1.72
1.69
1.58
1.57
1.55
1.54
1.52
1.51
1.49
1.48
1.36
1.33
1.30
1.27
1.26
1.25
1.24
1.22
1.21
0.89
2.87
0.96
0.92
2.93
1.95
1.98
1.08
2.08
3.10
f1 (ppm)

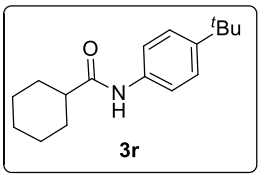

174.47
135.83
130.44
129.42
126.71
125.12
123.48
77.48
77.16
76.84
46.29
29.89
25.77
17.83
f1 (ppm)


O
N
H
Me

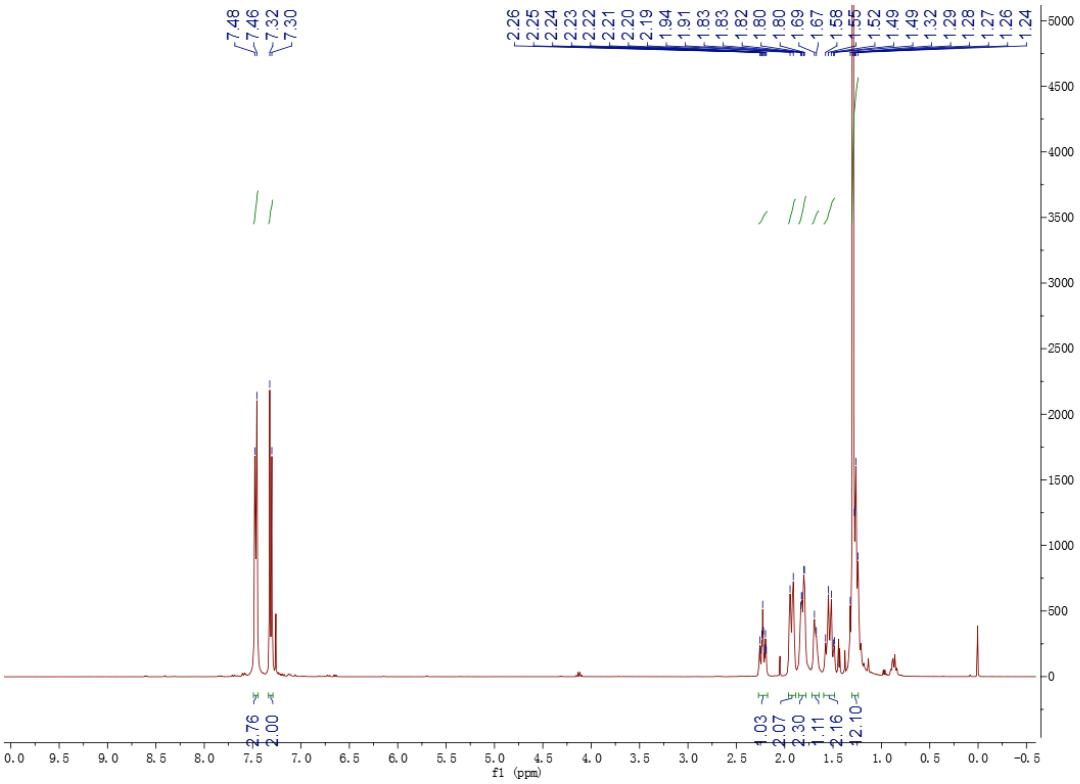
3q

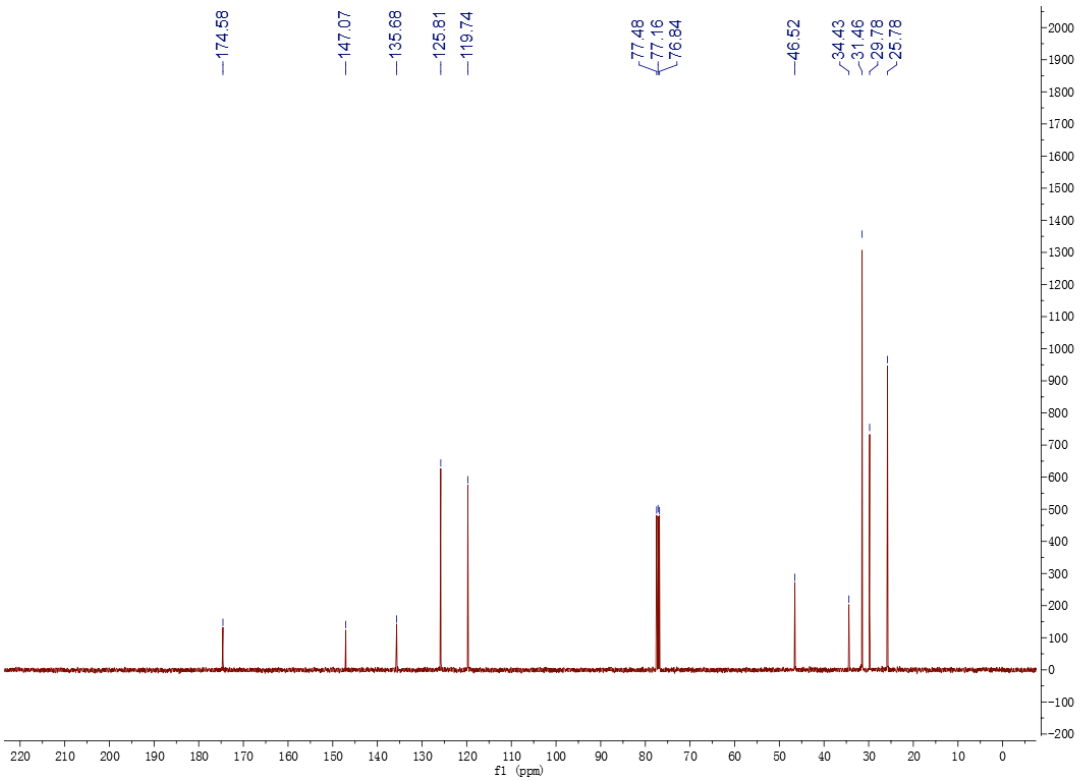

7.65
7.43
7.33
7.31
7.18
7.16
7.14
6.90
6.88
2.29
2.27
2.26
2.25
2.24
2.23
2.22
2.21
2.20
2.20
1.94
1.90
1.82
1.81
1.79
1.79
1.69
1.67
1.58
1.55
1.52
1.49
1.31
1.28
1.27
1.25
1.23
1.20
0.95
1.01
1.05
1.07
1.00
3.07
1.04
2.24
2.17
1.16
2.29
3.09
f1 (ppm)


174.82
138.84
138.24
128.76
124.87
120.66
117.04
77.48
77.16
76.84
46.49
29.73
25.75
25.71
21.50
f1 (ppm)


O
N
H
tBu

3r


7.48
7.46
7.32
7.30
2.26
2.25
2.24
2.23
2.22
2.21
2.20
2.19
1.94
1.91
1.83
1.83
1.82
1.80
1.80
1.69
1.67
1.58
1.55
1.52
1.49
1.49
1.32
1.29
1.28
1.27
1.26
1.24
2.76
2.00
1.03
2.07
2.30
1.11
2.16
12.10
f1 (ppm)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147.07
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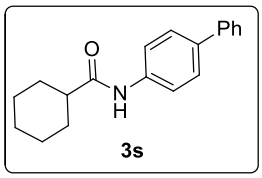
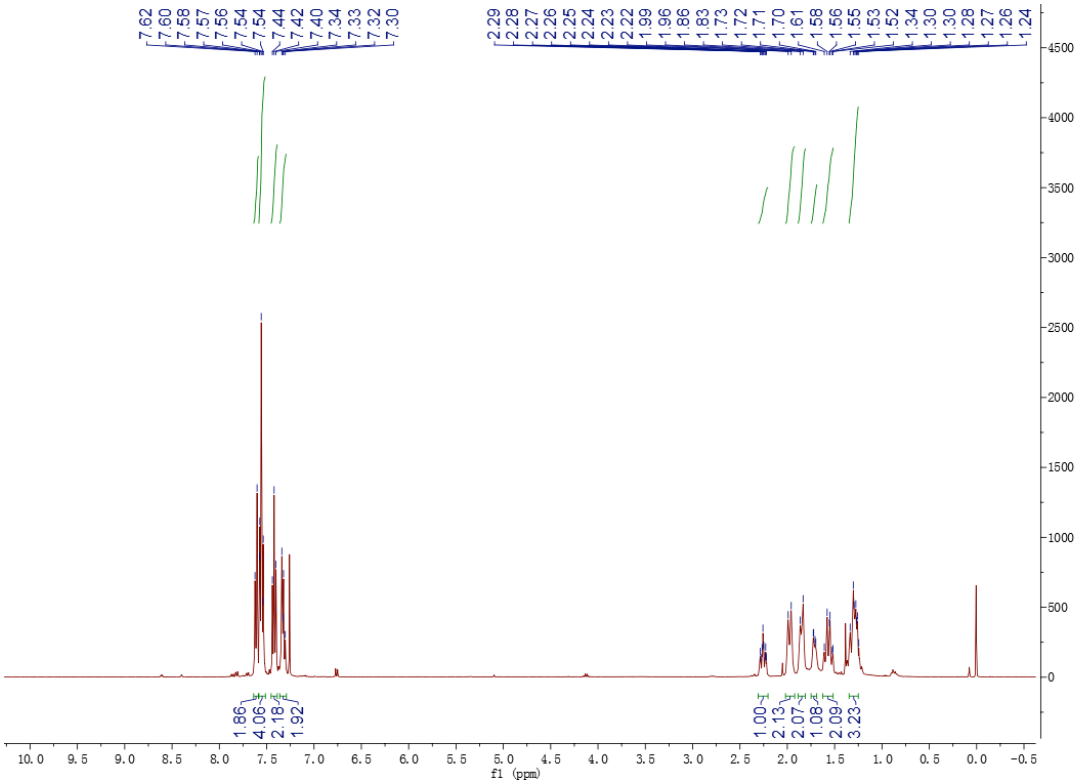
125.81
119.74
77.48
77.16
76.84
46.52
34.43
31.46
29.78
25.78
f1 (ppm)


O
N
H
Ph

3s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7.54
7.44
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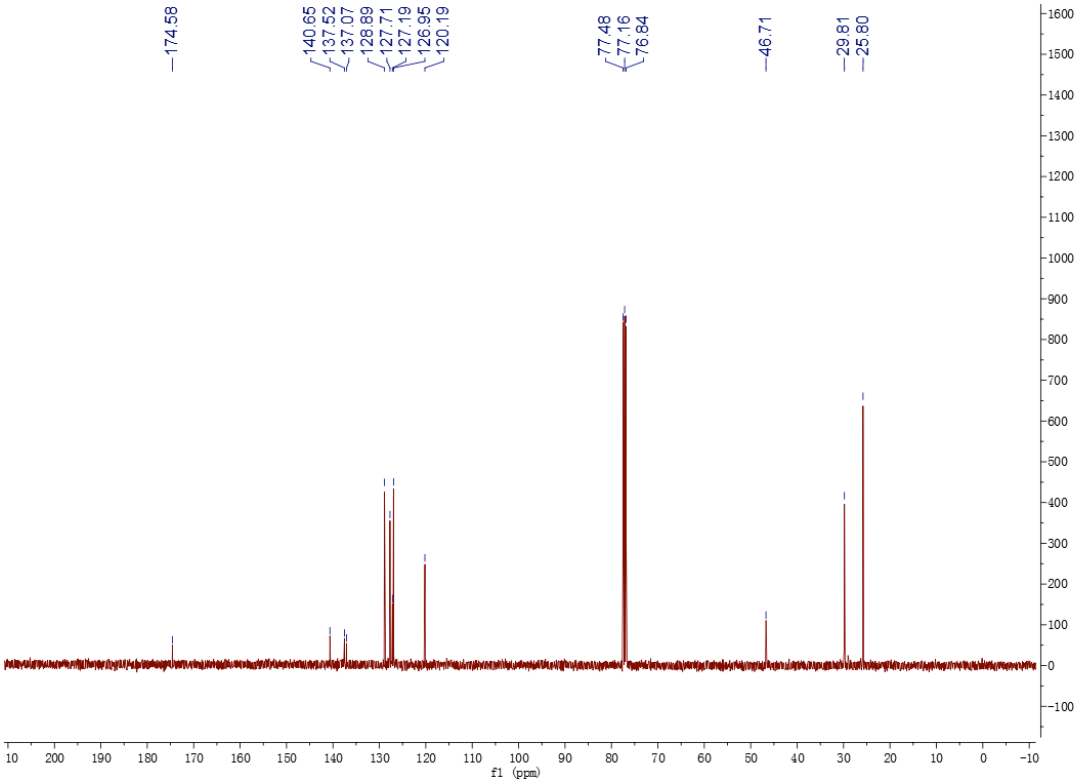
7.40
7.34
7.33
7.32
7.30
2.29
2.28
2.27
2.26
2.25
2.24
2.23
2.22
1.99
1.96
1.86
1.83
1.73
1.72
1.71
1.70
1.61
1.58
1.56
1.55
1.53
1.52
1.34
1.30
1.30
1.28
1.27
1.26
1.24
1.86
4.06
2.18
1.92
1.00
2.13
2.07
1.08
2.09
3.23
f1 (ppm)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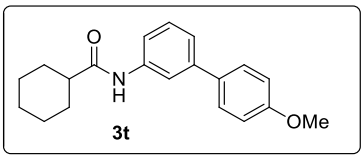
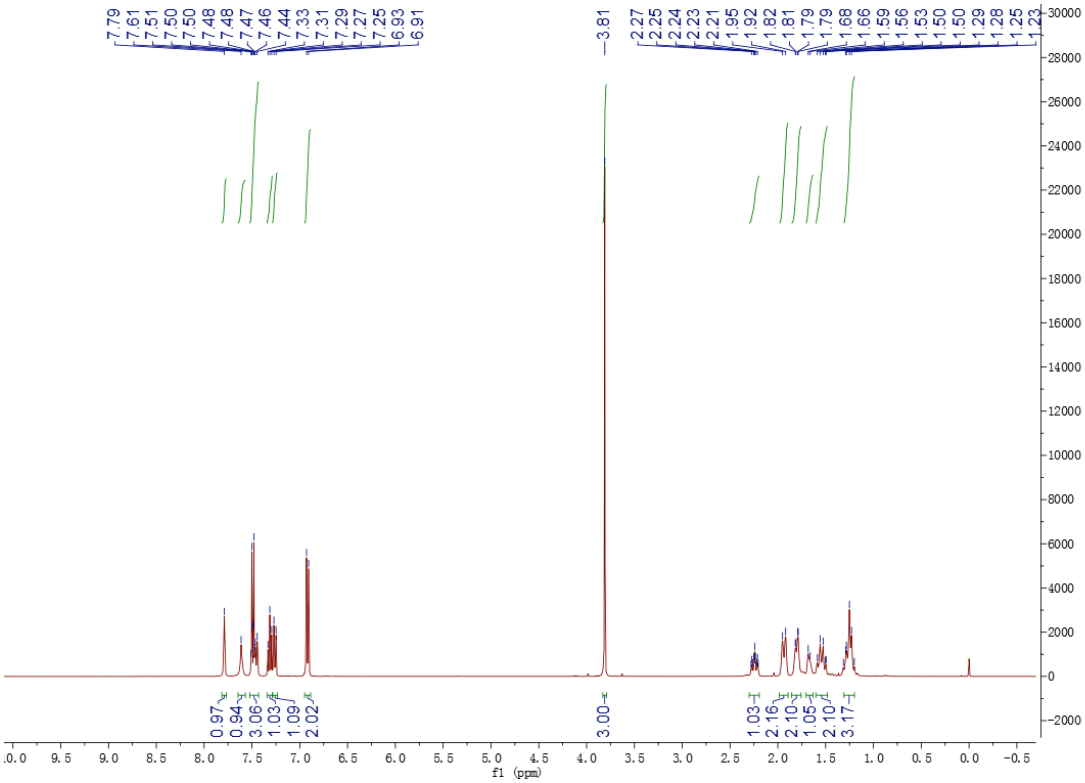
137.52
137.07
128.89
127.71
127.19
126.95
120.19
77.48
77.16
76.84
46.71
29.81
25.80
f1 (ppm)


O
N
H
OMe

3t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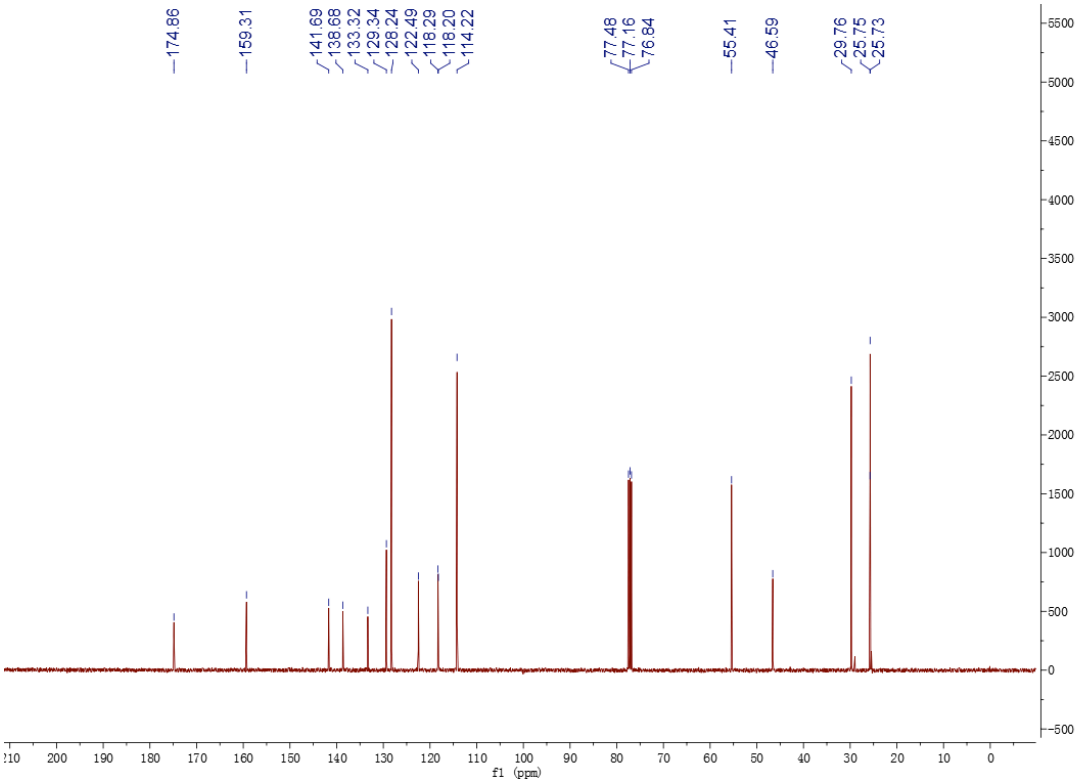
7.48
7.47
7.46
7.44
7.33
7.31
7.29
7.27
7.25
6.93
6.91
3.81
2.27
2.25
2.24
2.23
2.21
1.95
1.92
1.82
1.81
1.79
1.79
1.68
1.66
1.59
1.56
1.53
1.50
1.50
1.29
1.28
1.25
0.97
0.94
3.06
1.03
1.09
2.02
3.00
1.03
2.16
2.10
1.05
2.10
3.17
f1 (ppm)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133.32
129.34
128.24
122.49
118.29
118.20
114.22
77.48
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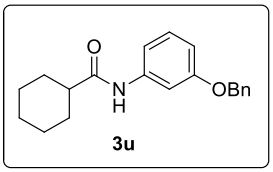
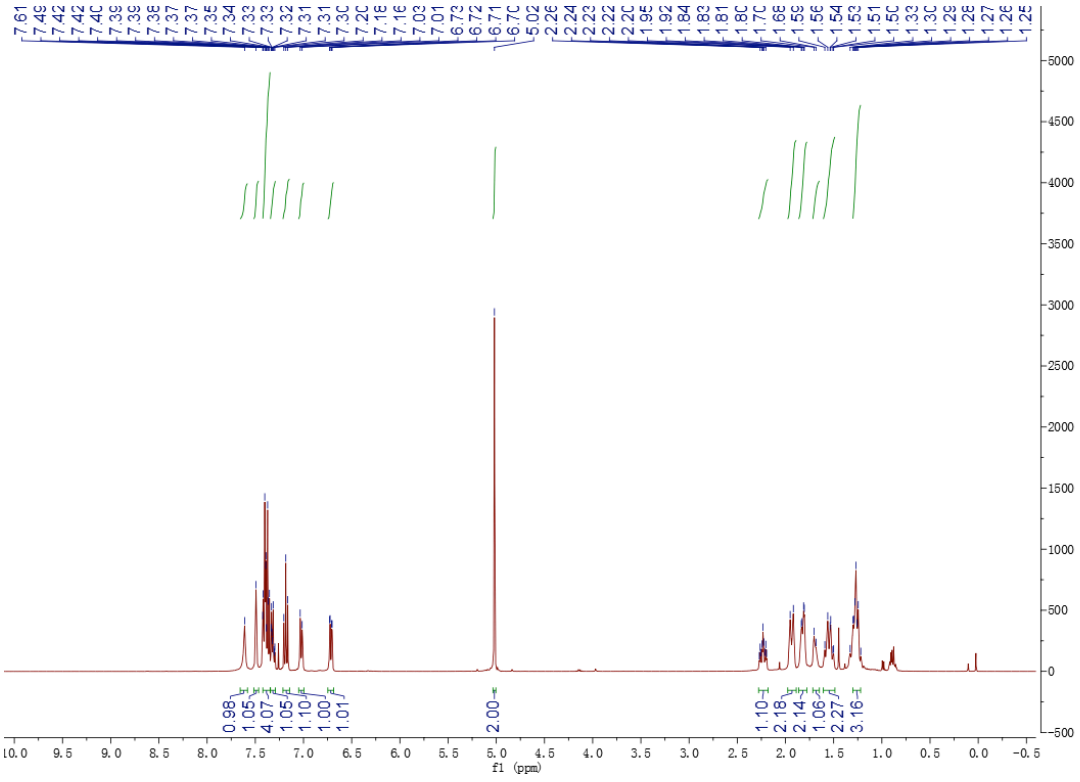
46.59
29.76
25.75
25.73
f1 (ppm)


O
N
H
OBn

3u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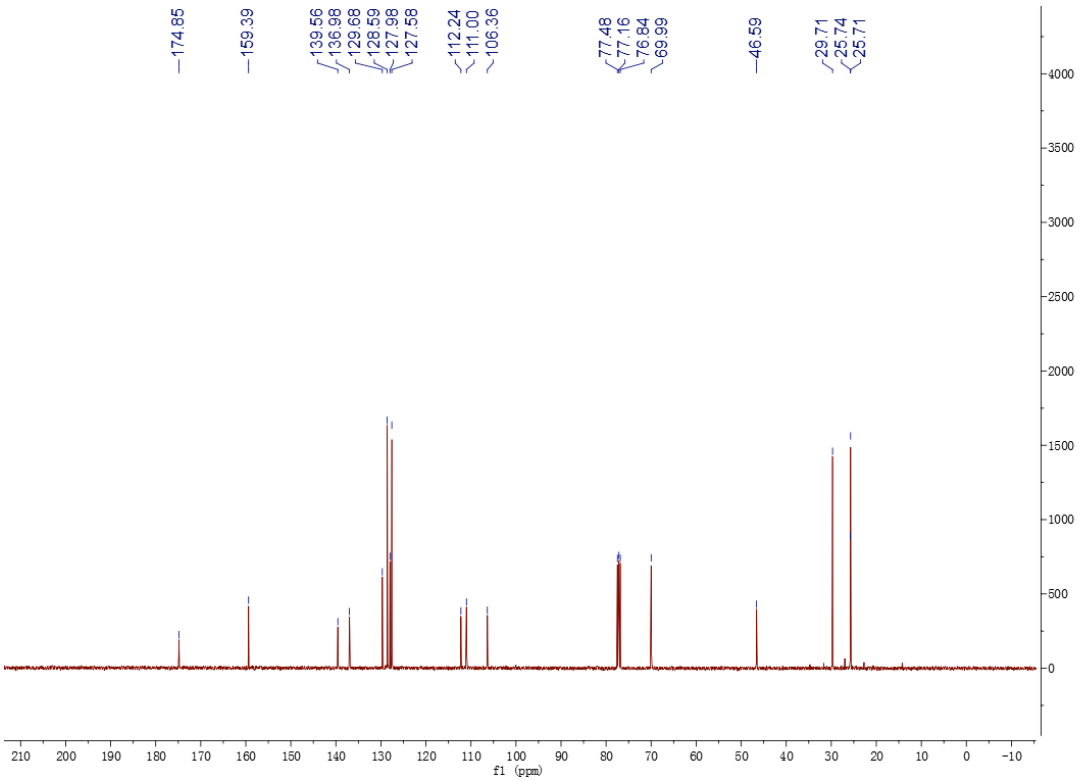
7.39
7.38
7.37
7.37
7.35
7.34
7.33
7.33
7.32
7.31
7.31
7.30
7.20
7.18
7.16
7.03
7.01
6.73
6.72
6.71
6.70
5.02
2.26
2.24
2.23
2.22
2.20
1.95
1.93
1.84
1.83
1.81
1.80
1.70
1.66
1.58
1.56
1.54
1.53
1.51
1.50
1.33
1.30
1.29
1.28
1.27
1.26
1.25
0.98
1.05
4.07
1.05
1.10
1.00
1.01
2.00
1.10
2.18
2.14
1.06
2.27
3.16
f1 (ppm)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136.98
129.68
128.59
127.98
127.58
112.24
111.00
106.36
77.48
77.16
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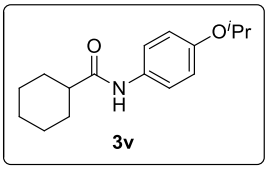
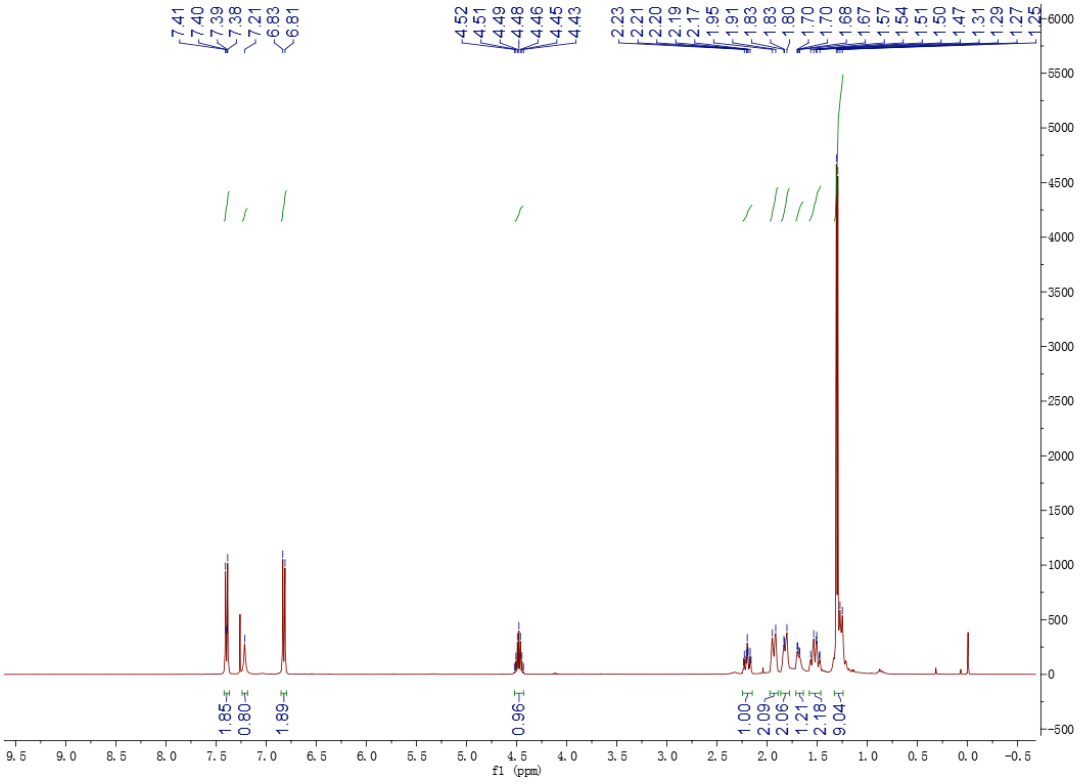
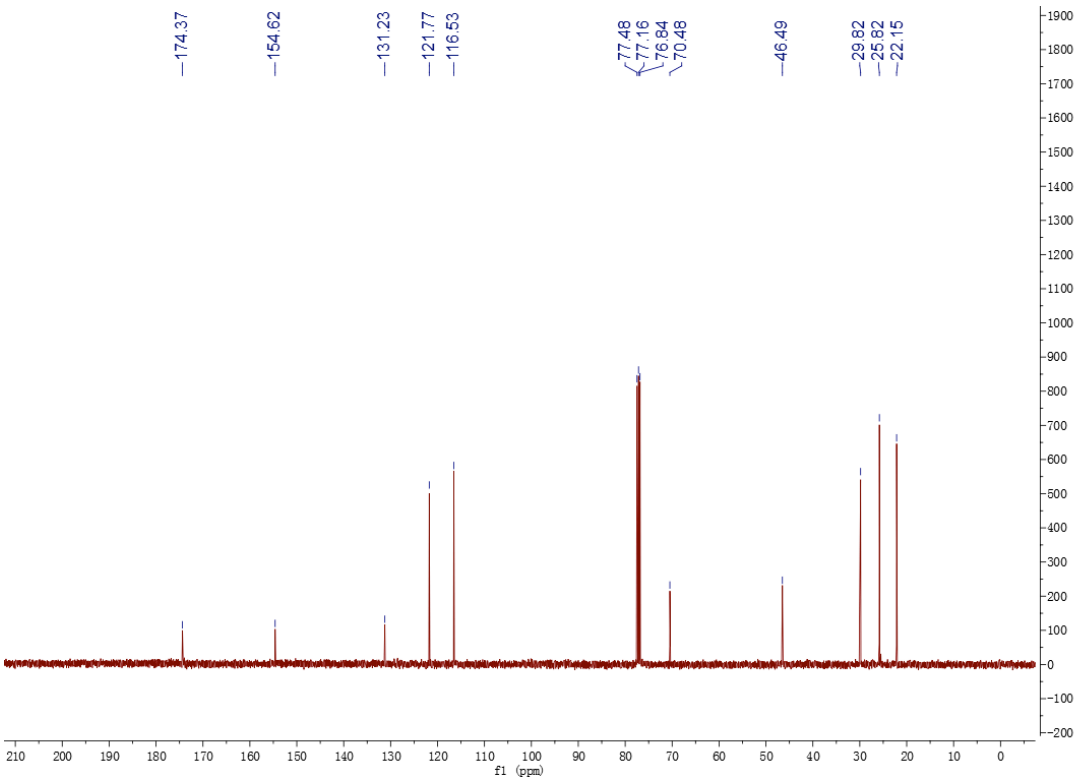
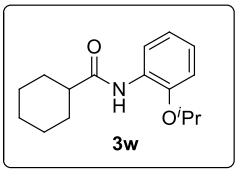
69.99
46.59
29.71
25.74
25.71
f1 (ppm)


O
N
H
O^{i}Pr

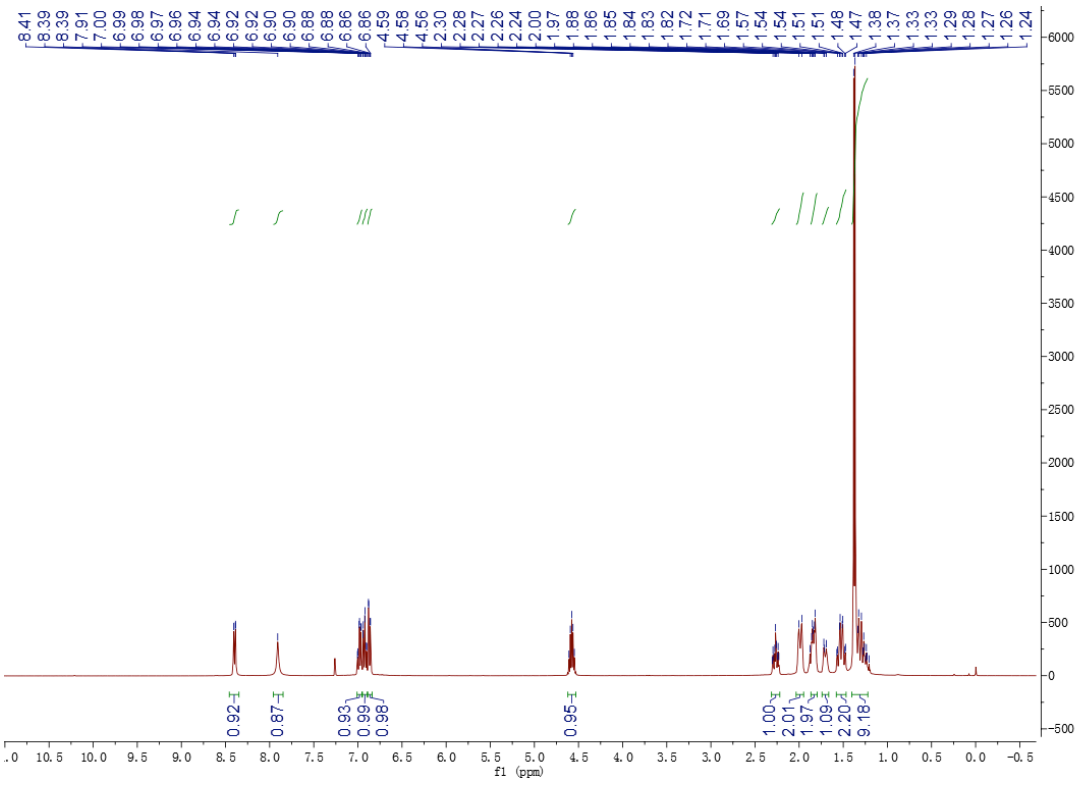
3v


7.41
7.40
7.39
7.38
7.21
6.83
6.81
4.52
4.51
4.49
4.48
4.46
4.45
4.43
2.23
2.21
2.20
2.19
2.17
1.95
1.91
1.83
1.80
1.70
1.68
1.67
1.57
1.54
1.51
1.50
1.47
1.31
1.29
1.27
1.25
1.85
0.80
1.89
0.96
1.00
2.09
2.06
1.21
2.18
9.04
f1 (ppm)


174.37
154.62
131.23
121.77
116.53
77.48
77.16
76.84
70.48
46.49
29.82
25.82
22.15
f1 (ppm)


O
N
H
O^{i}Pr

3w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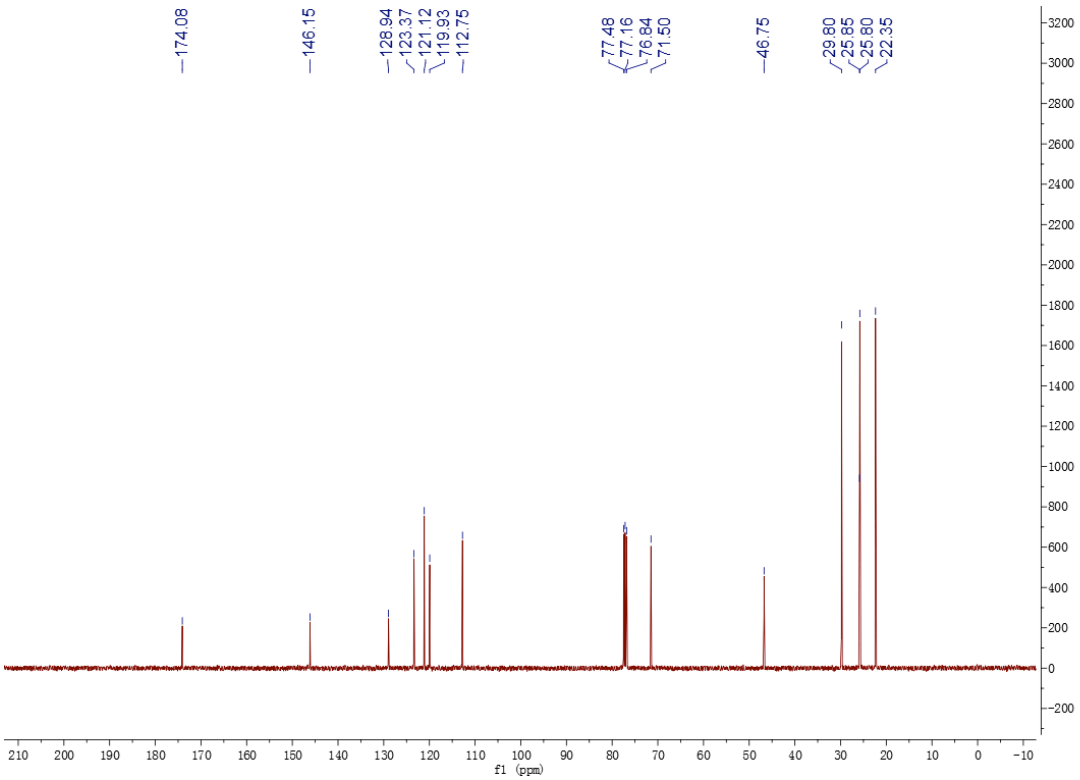
8.39
8.39
7.91
7.00
6.99
6.98
6.97
6.96
6.94
6.94
6.92
6.92
6.90
6.90
6.88
6.88
6.86
6.86
4.59
4.58
4.56
2.30
2.28
2.27
2.26
2.24
2.00
1.97
1.88
1.86
1.85
1.84
1.83
1.82
1.72
1.71
1.69
1.57
1.54
1.54
1.51
1.51
1.48
1.47
1.38
1.37
1.33
1.33
1.29
1.28
1.27
1.26
1.24
0.92
0.87
0.93
0.99
0.98
0.95
1.00
2.01
1.97
1.09
2.20
9.18
f1 (ppm)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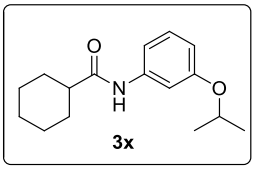
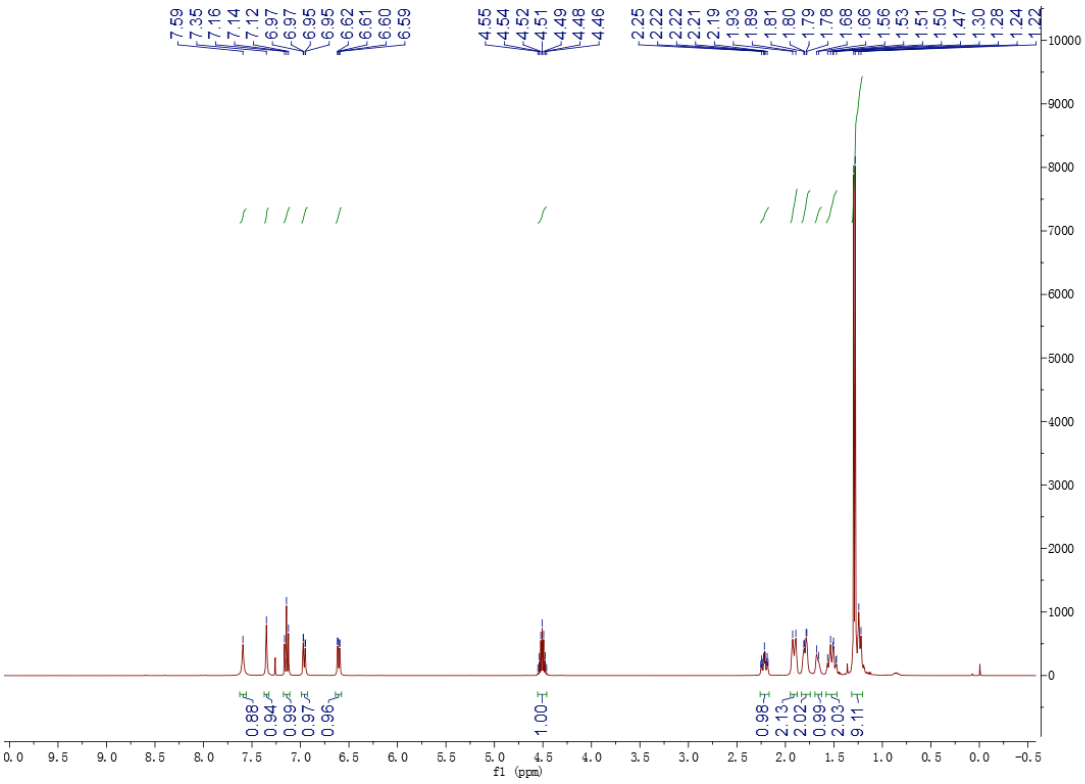
146.15
128.94
123.37
121.12
119.93
112.75
77.48
77.16
76.84
71.50
46.75
29.80
25.85
25.80
22.35
f1 (ppm)


O
N
H
O

3x


7.59
7.35
7.16
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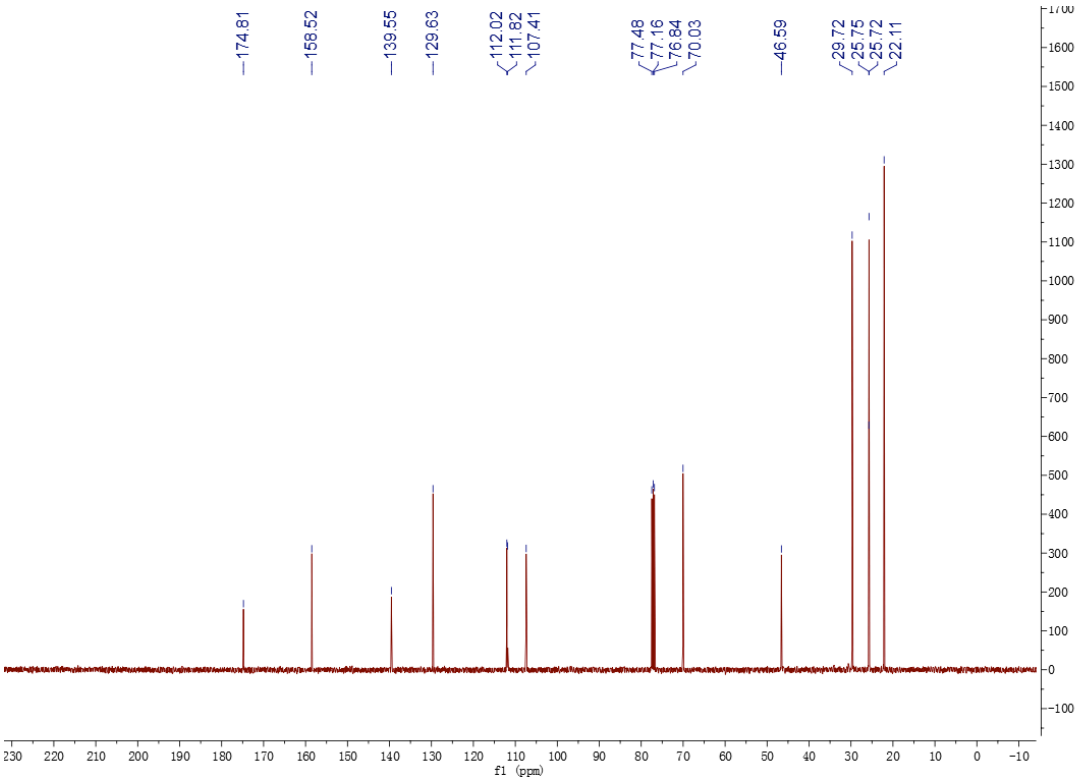
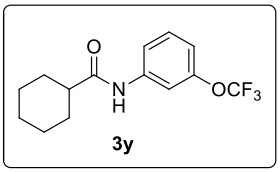
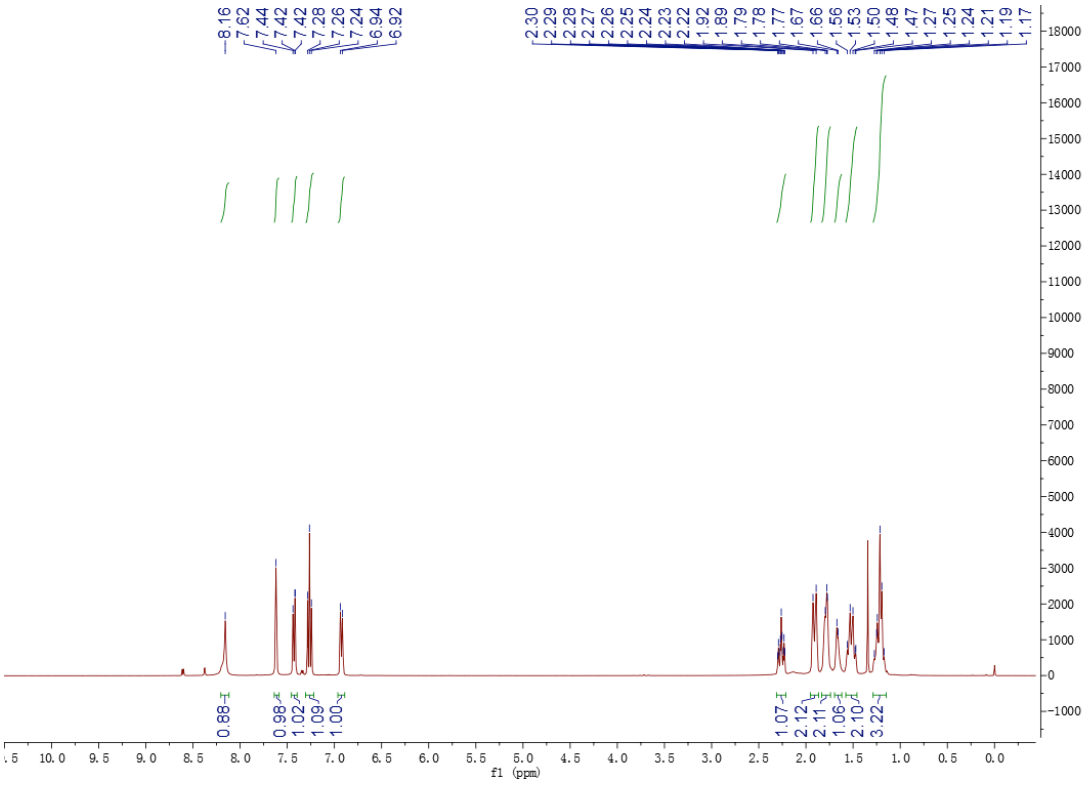
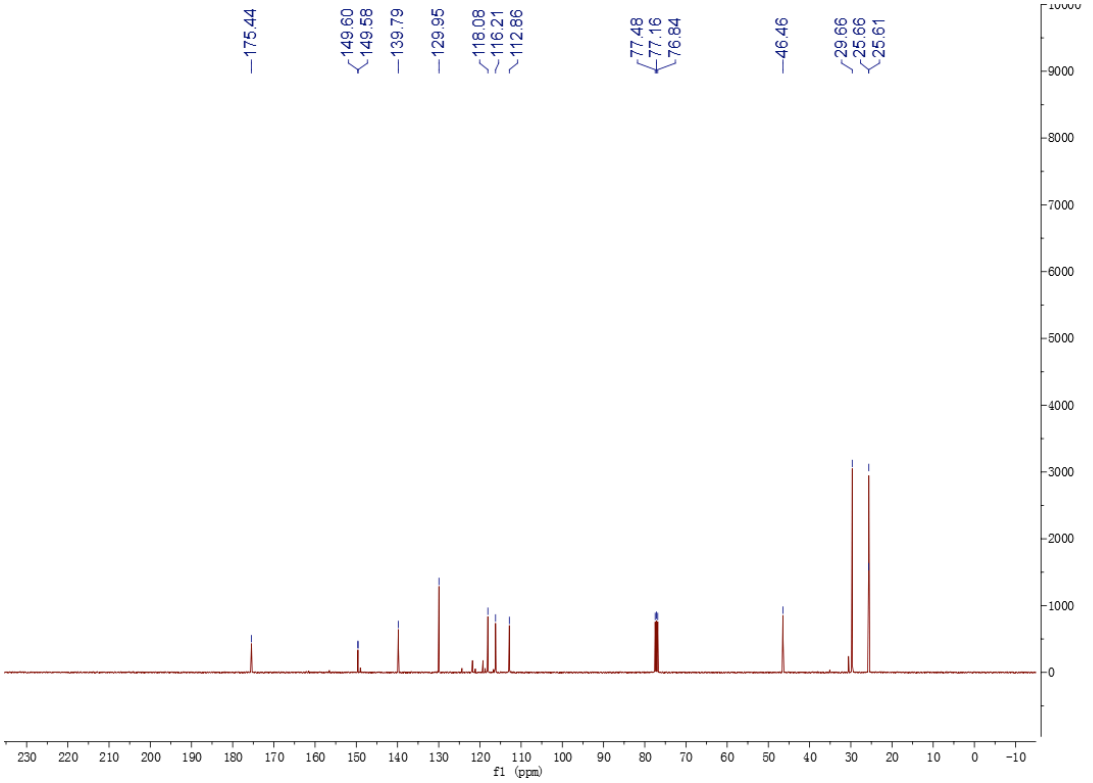
6.97
6.97
6.95
6.62
6.61
6.60
6.59
4.55
4.54
4.52
4.51
4.49
4.48
4.46
2.25
2.22
2.22
2.21
2.19
1.93
1.89
1.81
1.80
1.79
1.78
1.68
1.66
1.56
1.53
1.51
1.50
1.47
1.30
1.28
1.24
1.22
0.88
0.94
0.99
0.97
0.96
1.00
0.98
2.13
2.02
0.99
2.03
9.11
f1 (ppm)

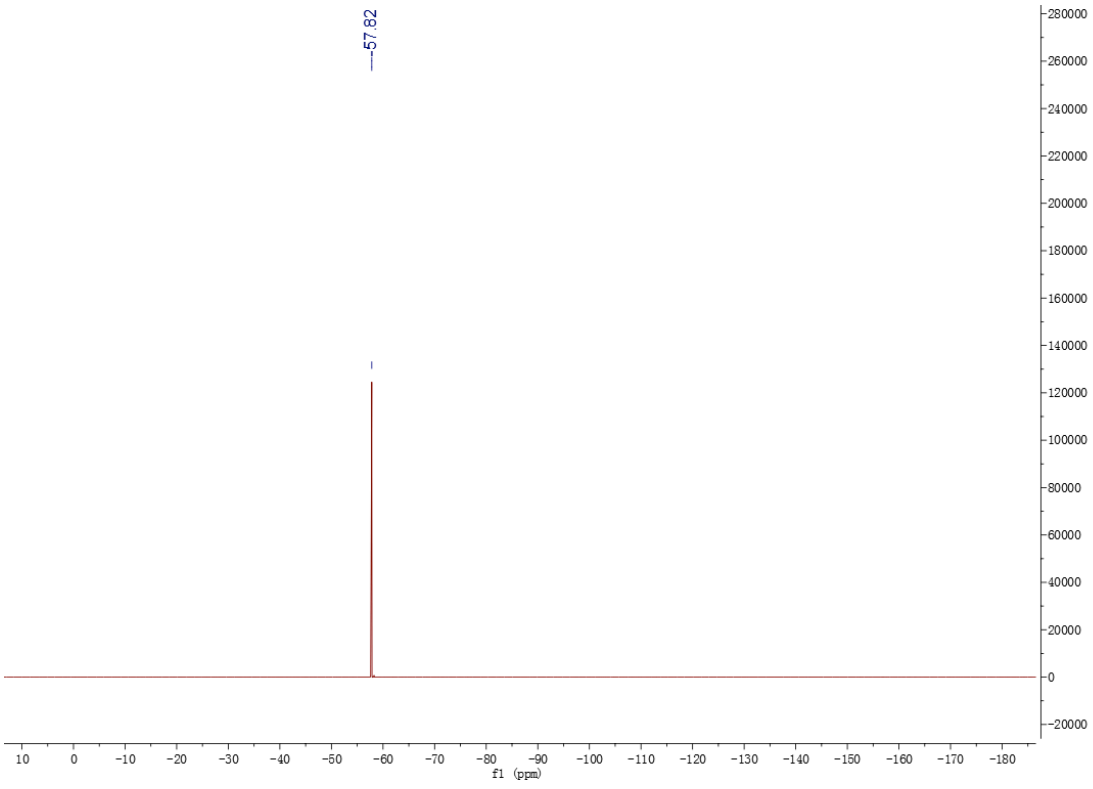

174.81
158.52
139.55
129.63
112.02
111.82
107.41
77.48
77.16
76.84
70.03
46.59
29.72
25.75
25.72
22.11
f1 (ppm)

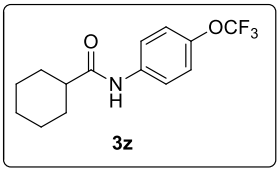

O
N
H
OCF3

3y

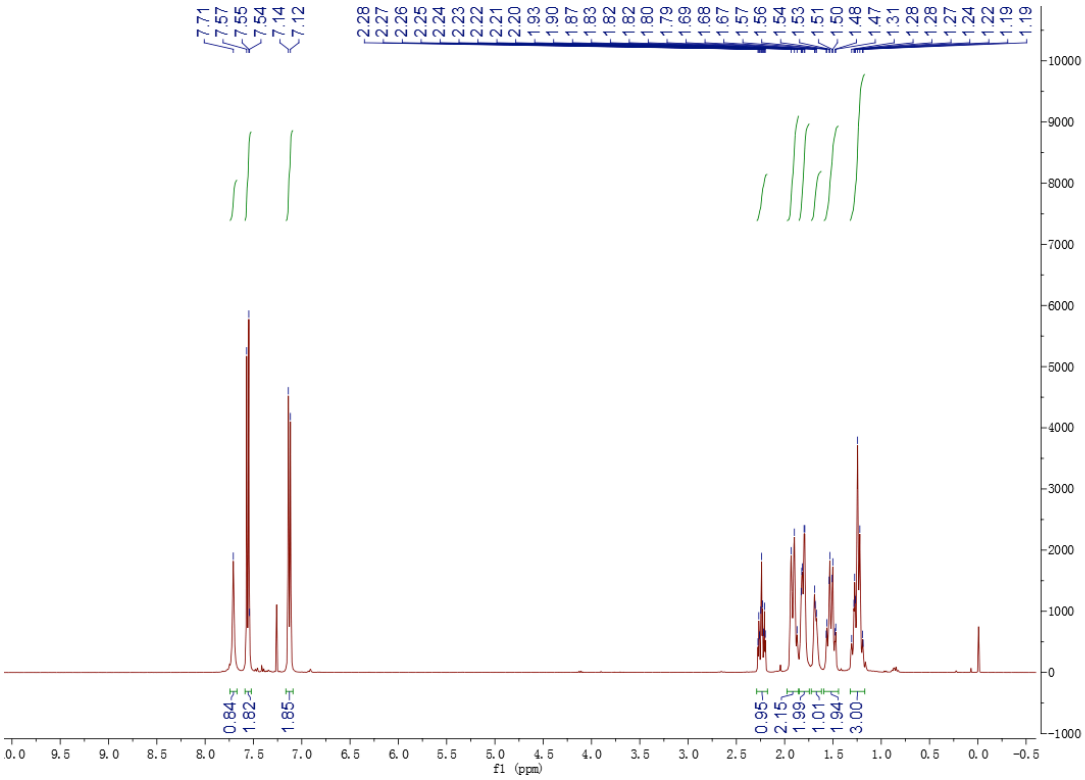

8.16
7.62
7.44
7.42
7.42
7.28
7.26
7.24
6.94
6.92
2.30
2.29
2.28
2.27
2.26
2.25
2.24
2.23
2.22
1.92
1.89
1.79
1.78
1.77
1.67
1.66
1.56
1.53
1.50
1.48
1.47
1.27
1.25
1.24
1.21
1.19
1.17
0.88
0.98
1.02
1.09
1.00
1.07
2.12
2.11
1.06
2.10
3.22
f1 (ppm)


175.44
149.60
149.58
139.79
129.95
118.08
116.21
112.86
77.48
77.16
76.84
46.46
29.66
25.66
25.61
f1 (ppm)


57.82
f1 (ppm)


O
N
H
OCF3

3z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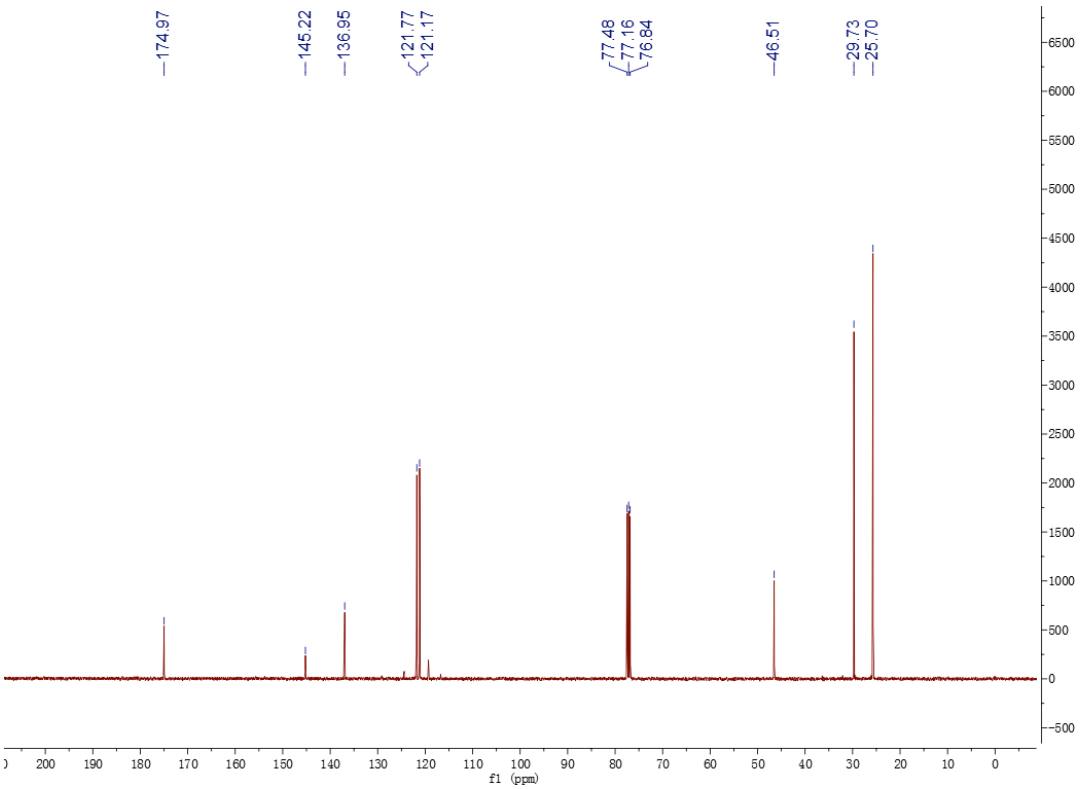
7.57
7.55
7.54
7.14
7.12
2.28
2.27
2.26
2.25
2.24
2.23
2.22
2.21
2.20
1.93
1.90
1.87
1.83
1.82
1.82
1.80
1.79
1.69
1.68
1.67
1.57
1.56
1.54
1.53
1.51
1.50
1.48
1.47
1.31
1.28
1.28
1.27
1.24
1.22
1.19
1.19
0.84
1.82
1.85
0.95
2.15
1.99
1.01
1.94
3.00
f1 (ppm)


174.97
145.22
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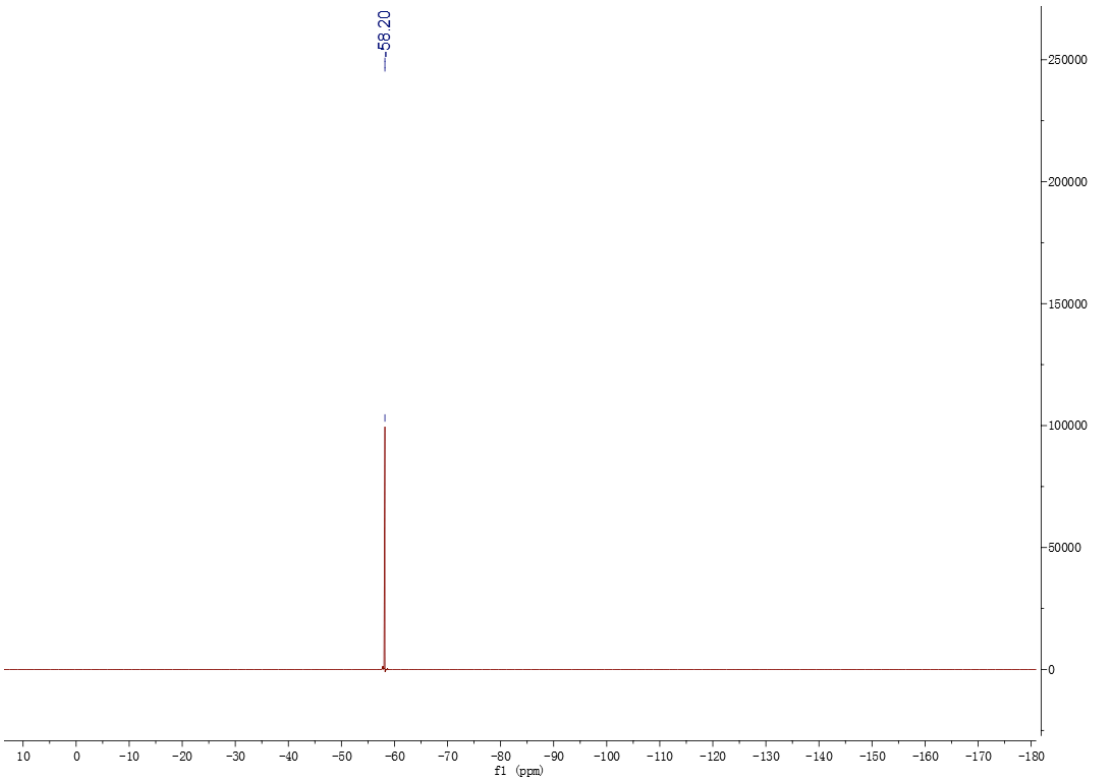
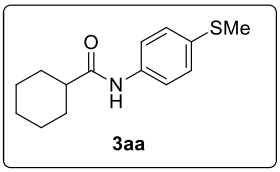
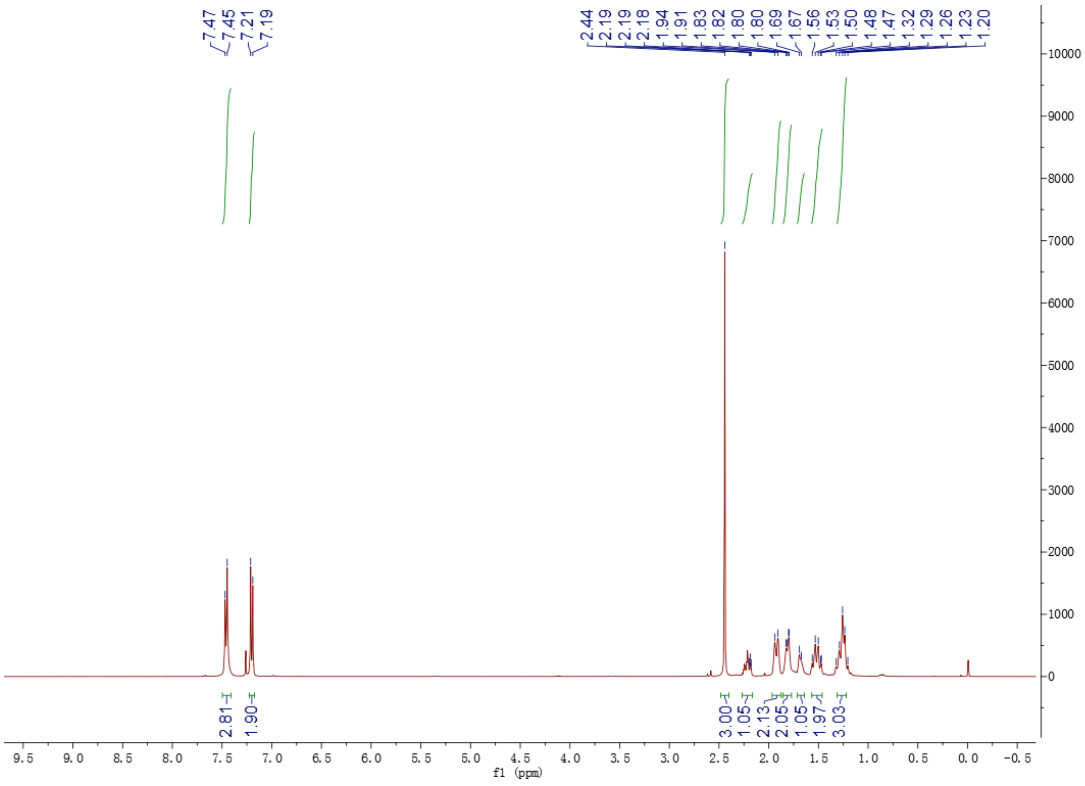
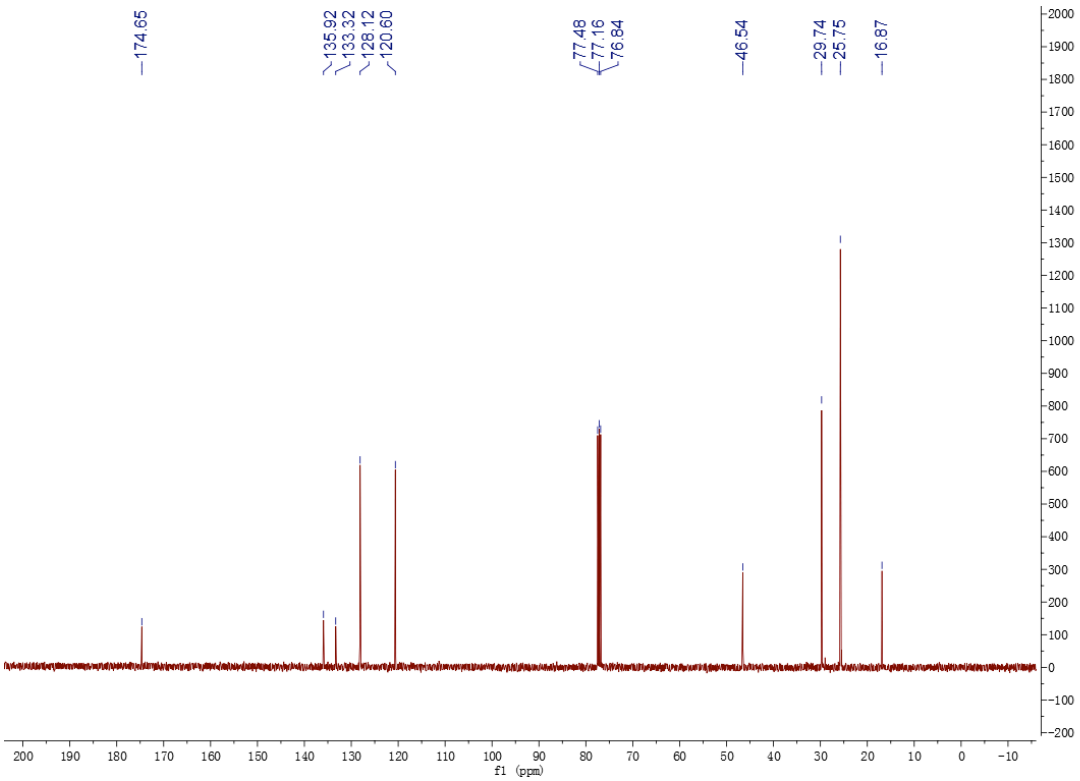
121.77
121.17
77.48
77.16
76.84
46.51
29.73
25.70
f1 (ppm)


-58.20
250000
200000
150000
100000
50000
0
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180
f1 (ppm)


O
N
H
SMe

3aa


7.47
7.45
7.21
7.19
2.44
2.19
2.19
2.18
1.94
1.91
1.83
1.82
1.80
1.80
1.69
1.67
1.56
1.53
1.50
1.48
1.47
1.32
1.29
1.26
1.23
1.20
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
2.81
1.90
3.00
1.05
2.13
2.05
1.05
1.97
3.03
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)


174.65
135.92
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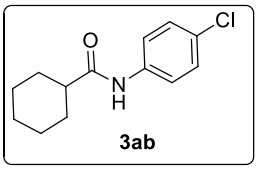
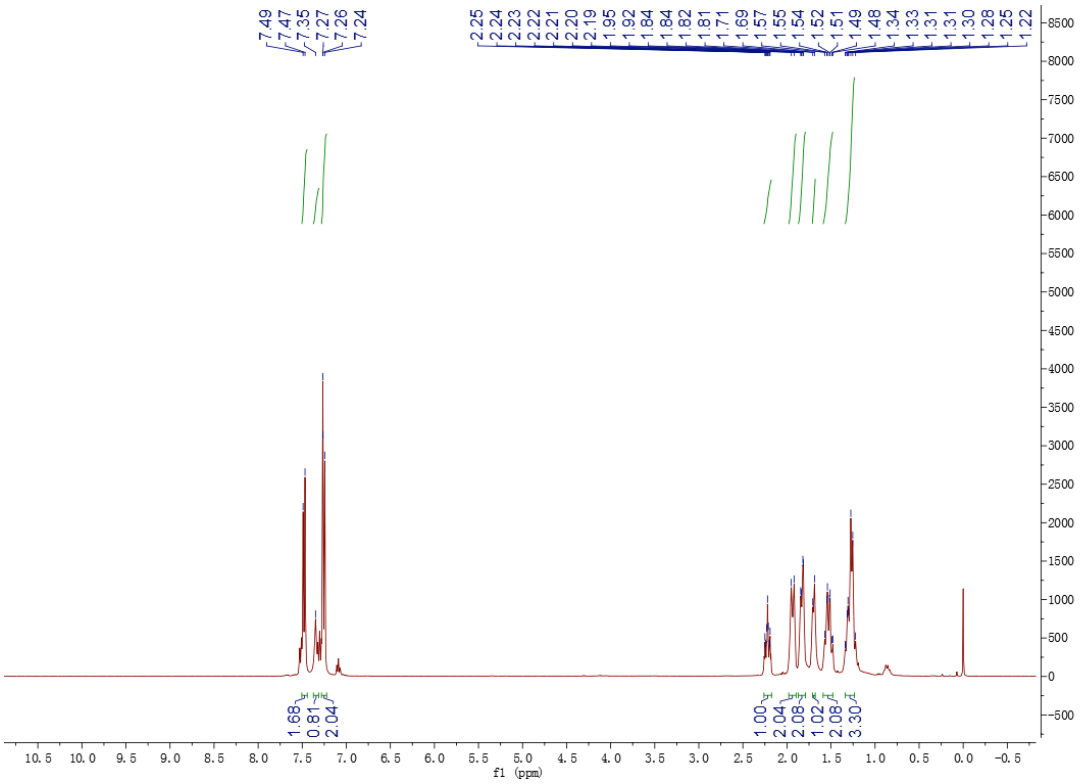
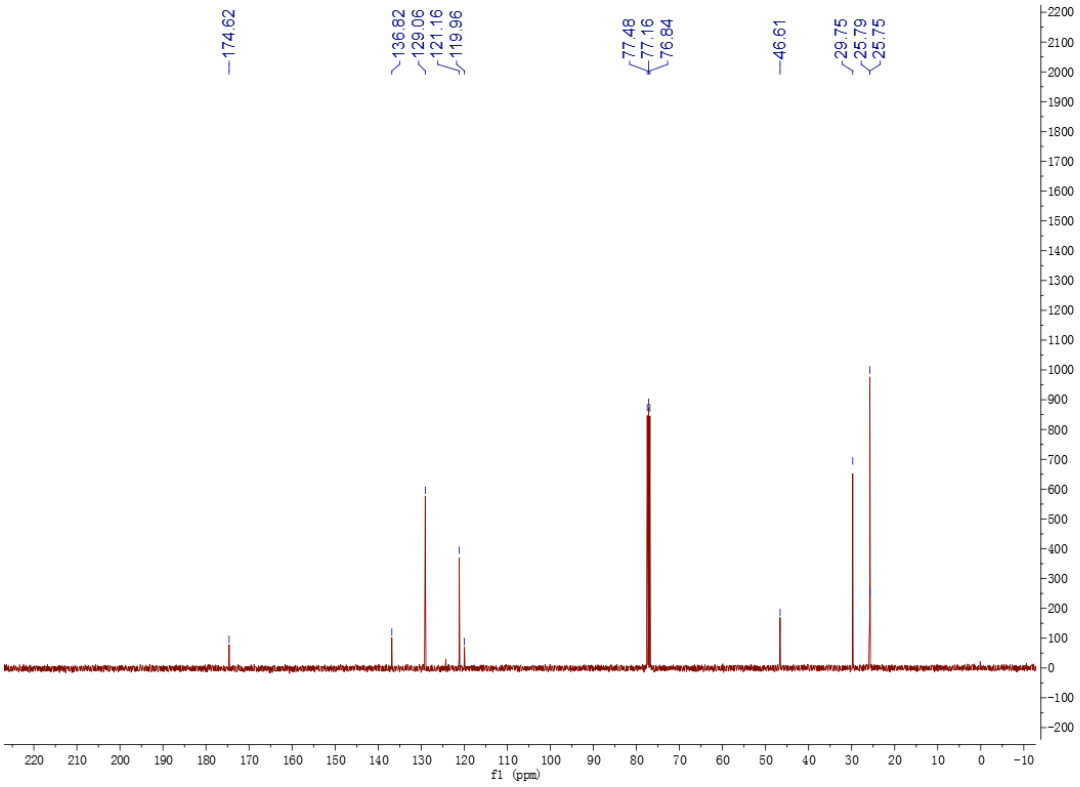
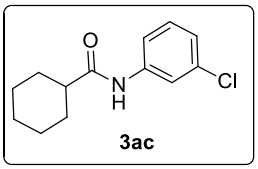
128.12
120.60
77.48
77.16
76.84
46.54
29.74
25.75
16.87
f1 (ppm)


O
N
H
Cl

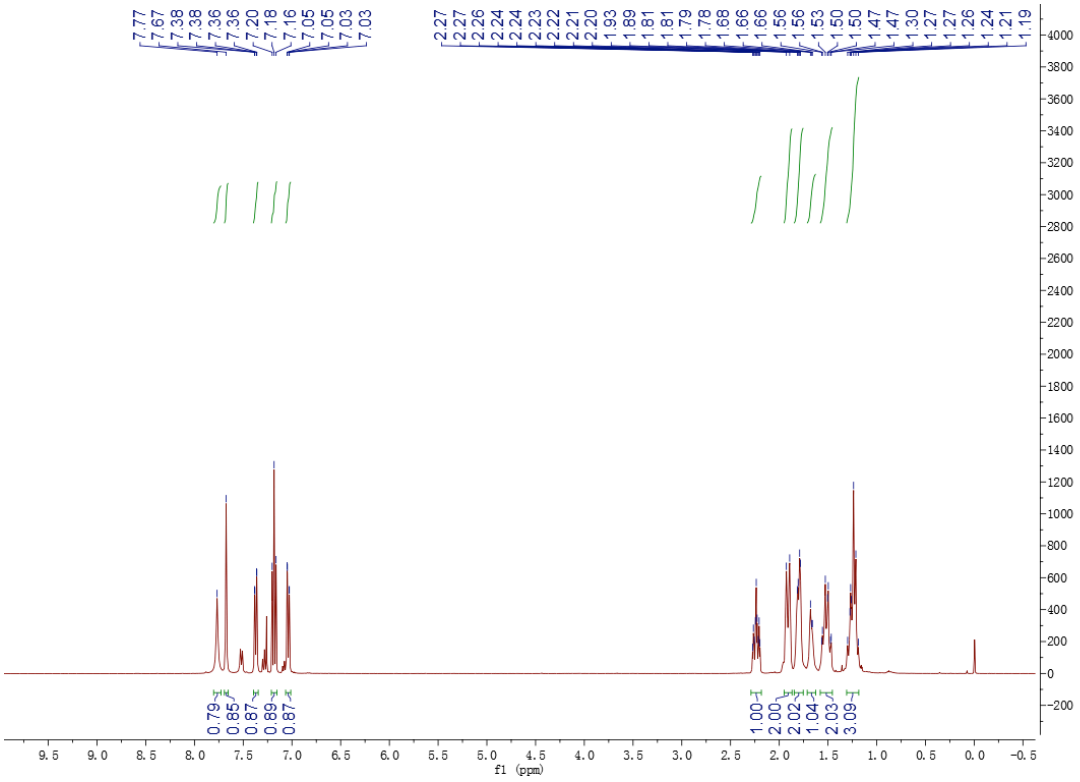
3ab


7.49
7.47
7.35
7.27
7.26
7.24
2.25
2.24
2.23
2.22
2.21
2.20
2.19
1.95
1.92
1.84
1.84
1.82
1.81
1.71
1.69
1.57
1.55
1.54
1.52
1.51
1.49
1.48
1.34
1.33
1.31
1.31
1.30
1.28
1.25
1.22
1.68
0.81
2.04
1.00
2.04
2.08
1.02
2.08
3.30
f1 (ppm)


174.62
136.82
129.06
121.16
119.96
77.48
77.16
76.84
46.61
29.75
25.79
25.75
f1 (ppm)


O
N
H
Cl

3ac


7.77
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7.36
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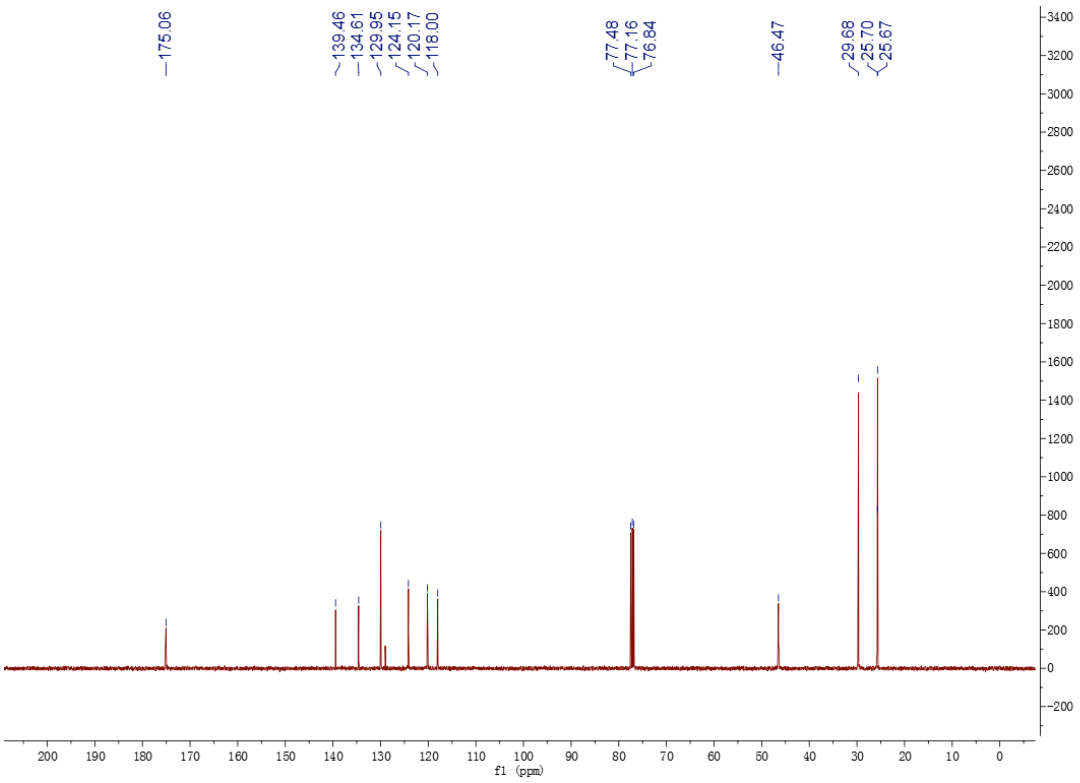
7.20
7.18
7.16
7.05
7.05
7.03
7.03
2.27
2.27
2.26
2.24
2.24
2.23
2.22
2.21
2.20
1.93
1.89
1.81
1.81
1.79
1.78
1.68
1.66
1.66
1.56
1.56
1.53
1.50
1.50
1.47
1.47
1.30
1.27
1.27
1.26
1.24
1.21
1.19
0.79
0.85
0.87
0.89
0.87
1.00
2.00
2.02
1.04
2.03
3.09
f1 (ppm)


175.06
139.46
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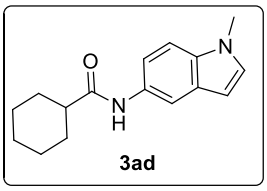
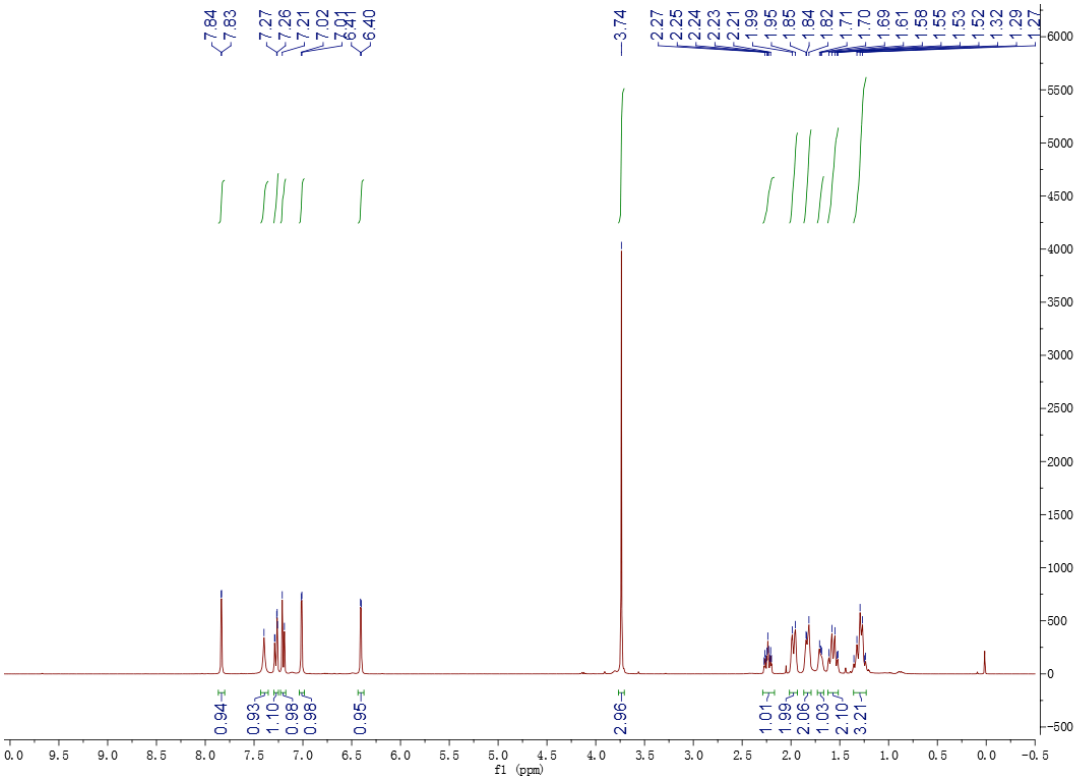
129.95
124.15
120.17
118.00
77.48
77.16
76.84
46.47
29.68
25.70
25.67
f1 (ppm)


O
N
H
N

3ad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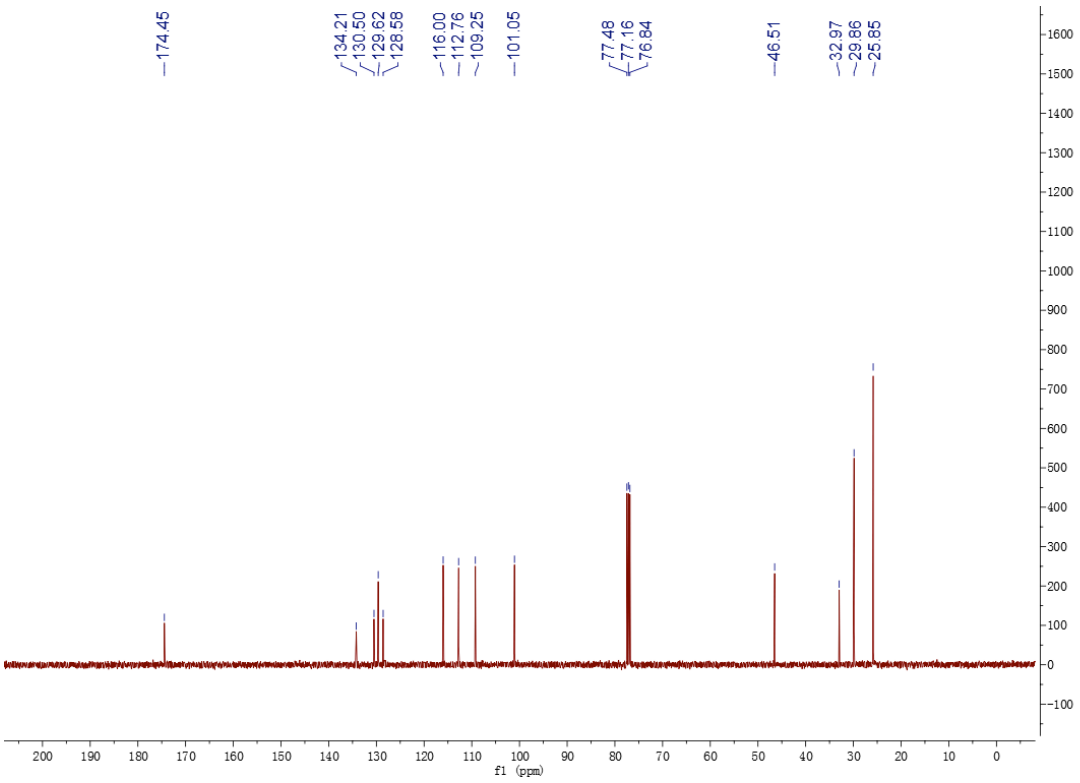
7.26
7.21
7.02
7.01
6.40
3.74
2.27
2.25
2.24
2.23
2.21
1.99
1.95
1.85
1.84
1.82
1.71
1.70
1.69
1.61
1.58
1.55
1.53
1.52
1.32
1.29
1.27
0.94
0.93
1.10
0.98
0.98
0.95
2.96
1.01
1.99
2.06
1.03
2.10
3.21
f1 (ppm)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129.62
128.58
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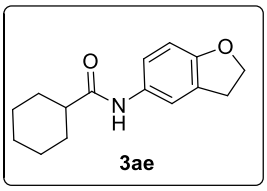
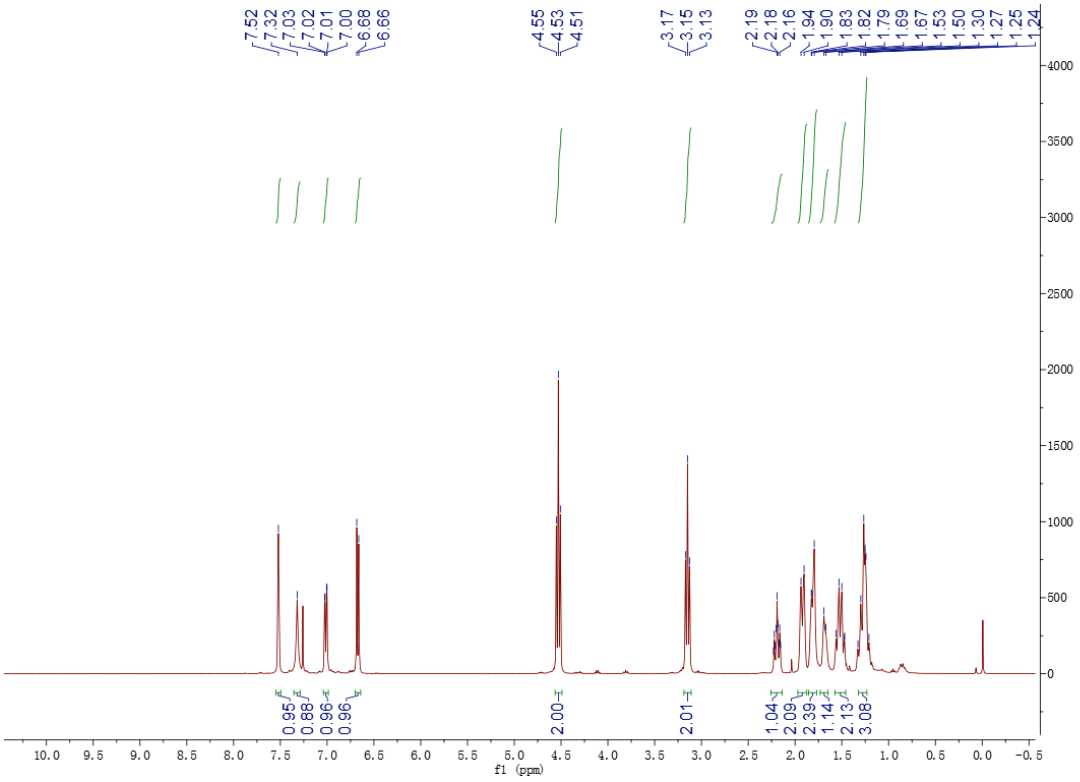
101.05
77.48
77.16
76.84
46.51
32.97
29.86
25.85
f1 (ppm)


O
N
H
O

3ae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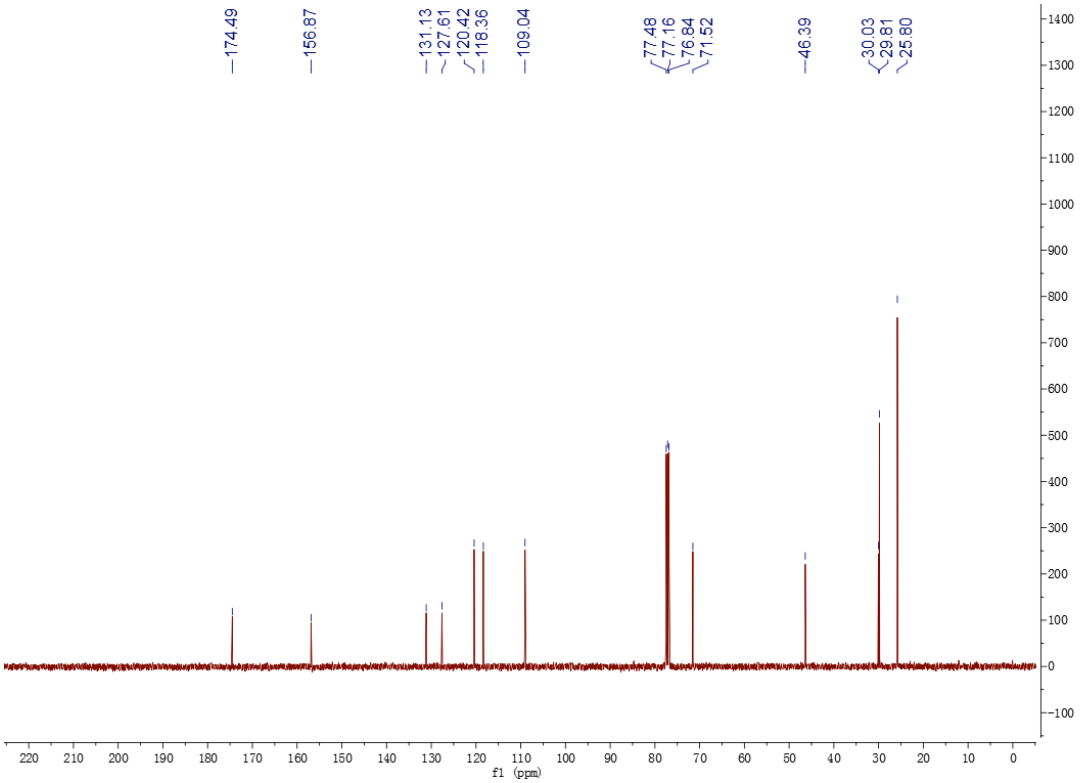
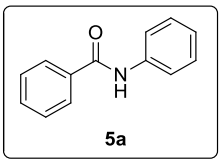
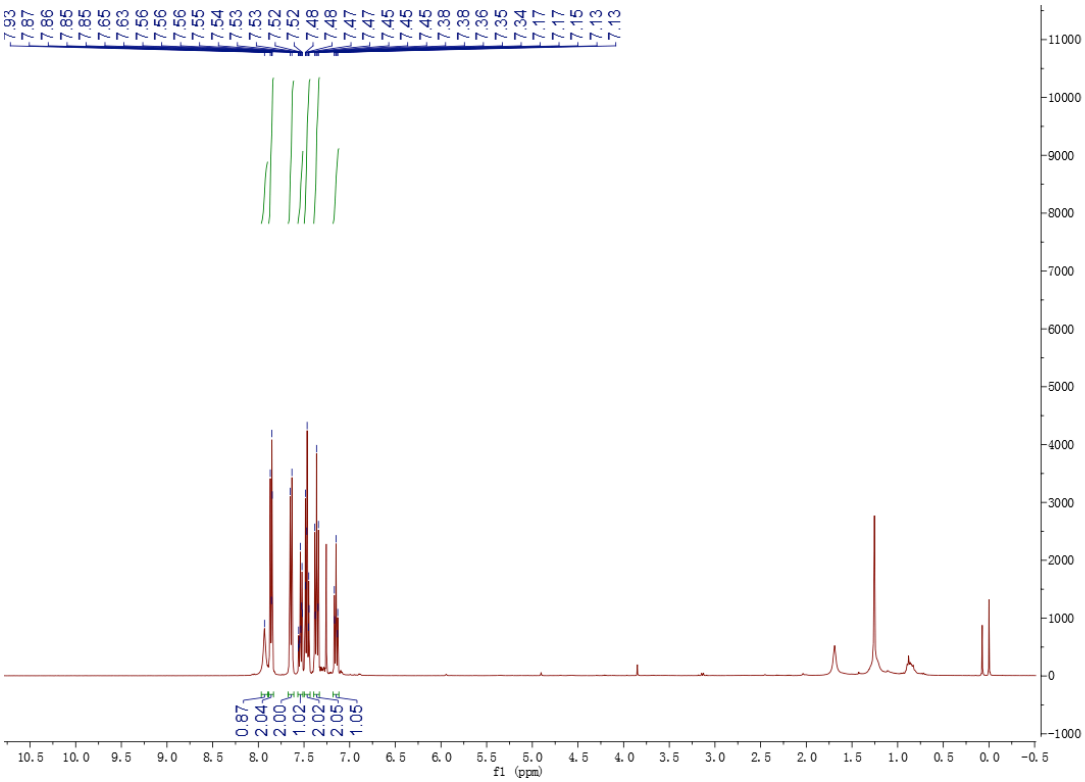
7.02
7.01
7.00
6.68
6.66
4.55
4.53
4.51
3.17
3.15
3.13
2.19
2.18
2.16
1.94
1.90
1.83
1.82
1.79
1.69
1.67
1.53
1.50
1.30
1.27
1.25
1.24
0.95
0.88
0.96
0.96
2.00
2.01
1.04
2.09
2.39
1.14
2.13
3.08
f1 (ppm)


174.49
156.87
131.13
127.61
120.42
118.36
109.04
77.48
77.16
76.84
71.52
46.39
30.03
29.81
25.80
f1 (ppm)


O
N
H

5a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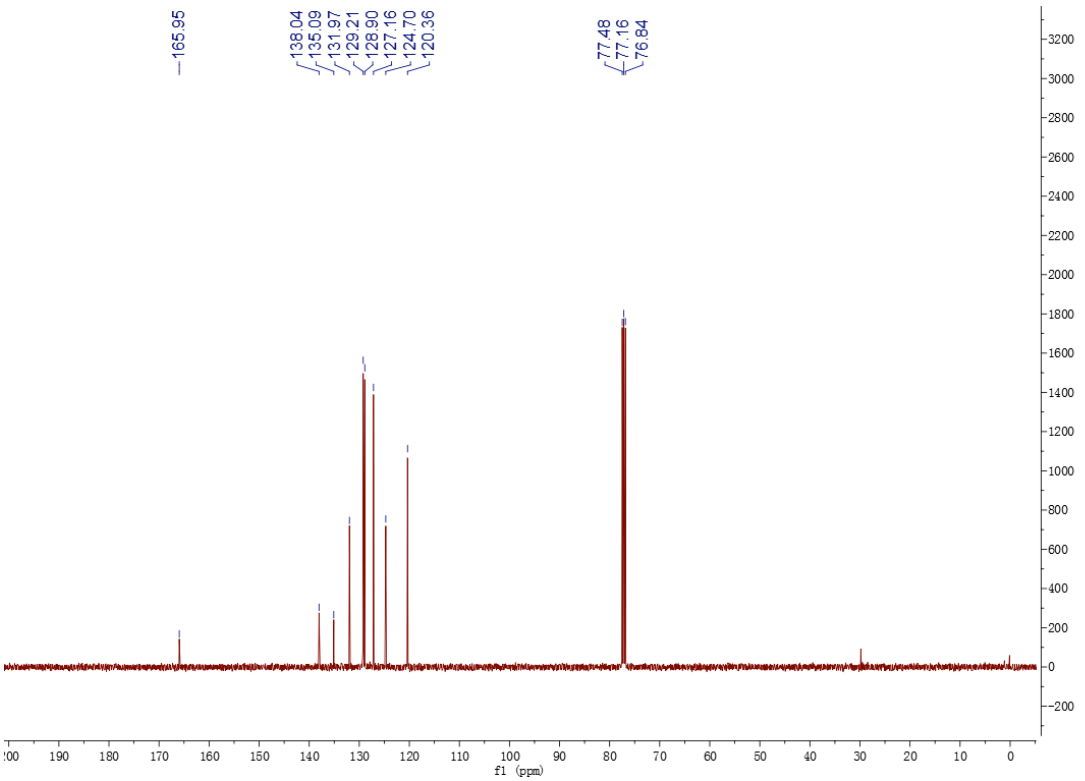
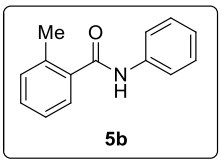
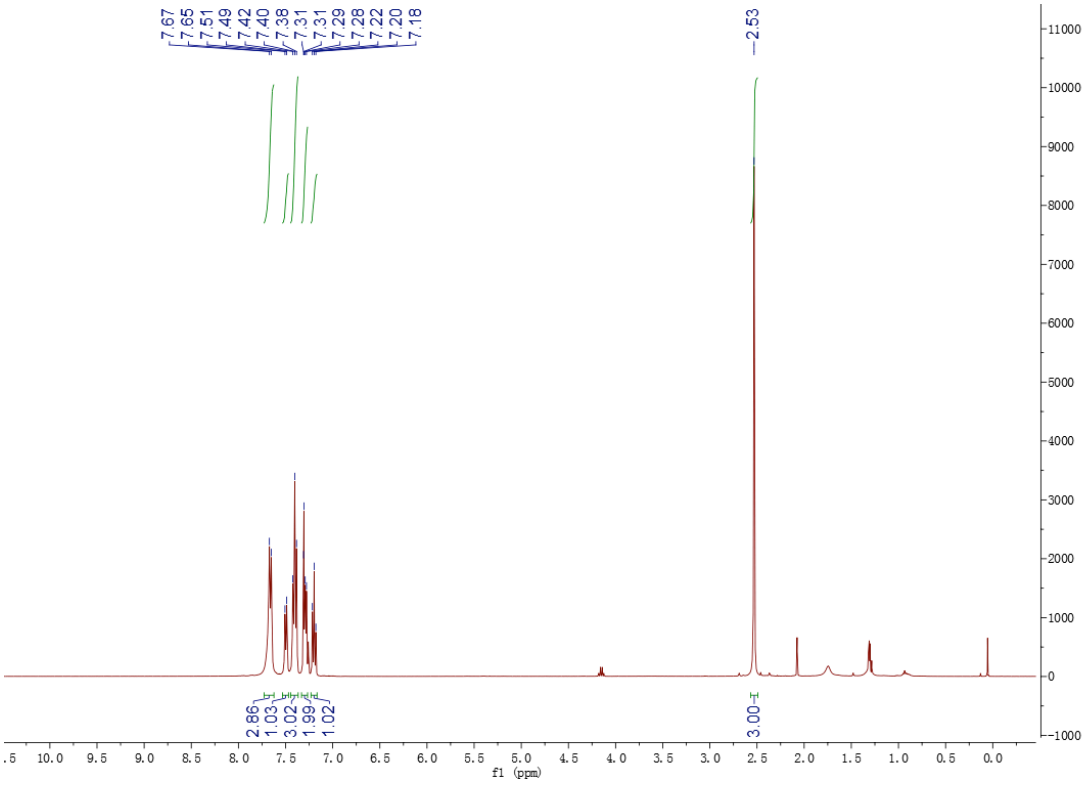
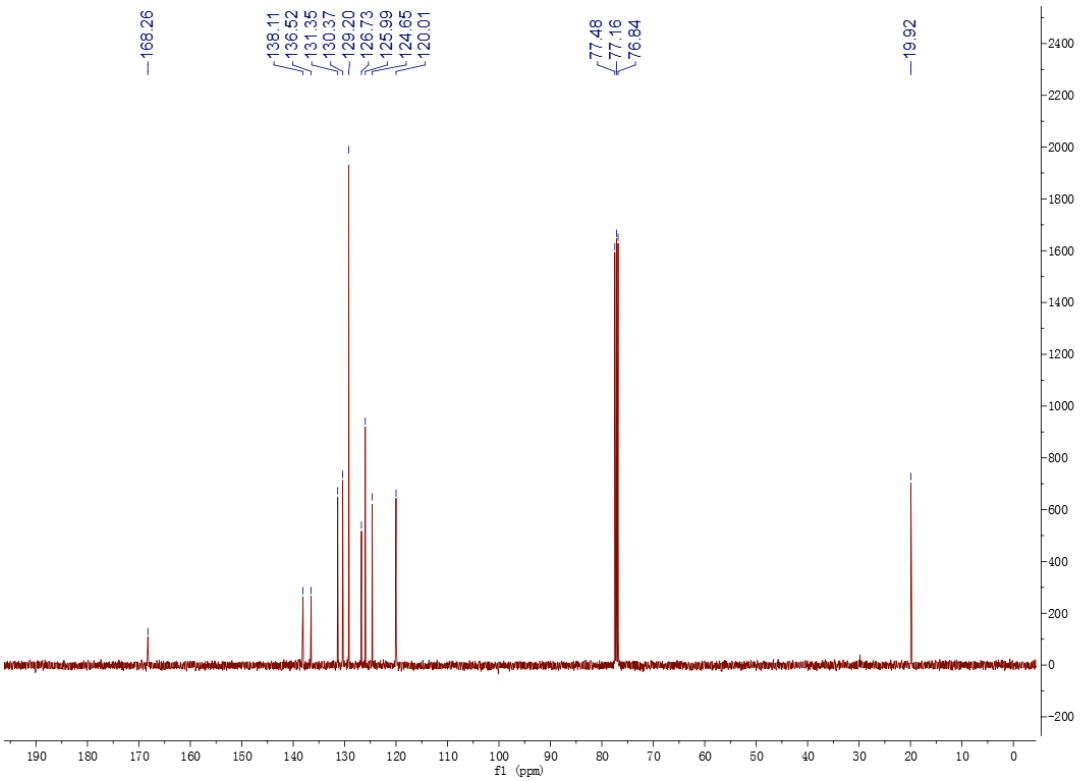
7.63
7.56
7.56
7.56
7.55
7.54
7.53
7.53
7.52
7.52
7.48
7.48
7.47
7.47
7.45
7.45
7.45
7.38
7.38
7.36
7.35
7.34
7.17
7.17
7.15
7.13
7.13
0.87
2.04
2.00
1.02
2.02
2.05
1.05
f1 (ppm)

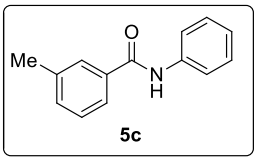

165.95
138.04
135.09
131.97
129.21
128.90
127.16
124.70
120.36
77.48
77.16
76.84
f1 (ppm)


Me
O
N
H

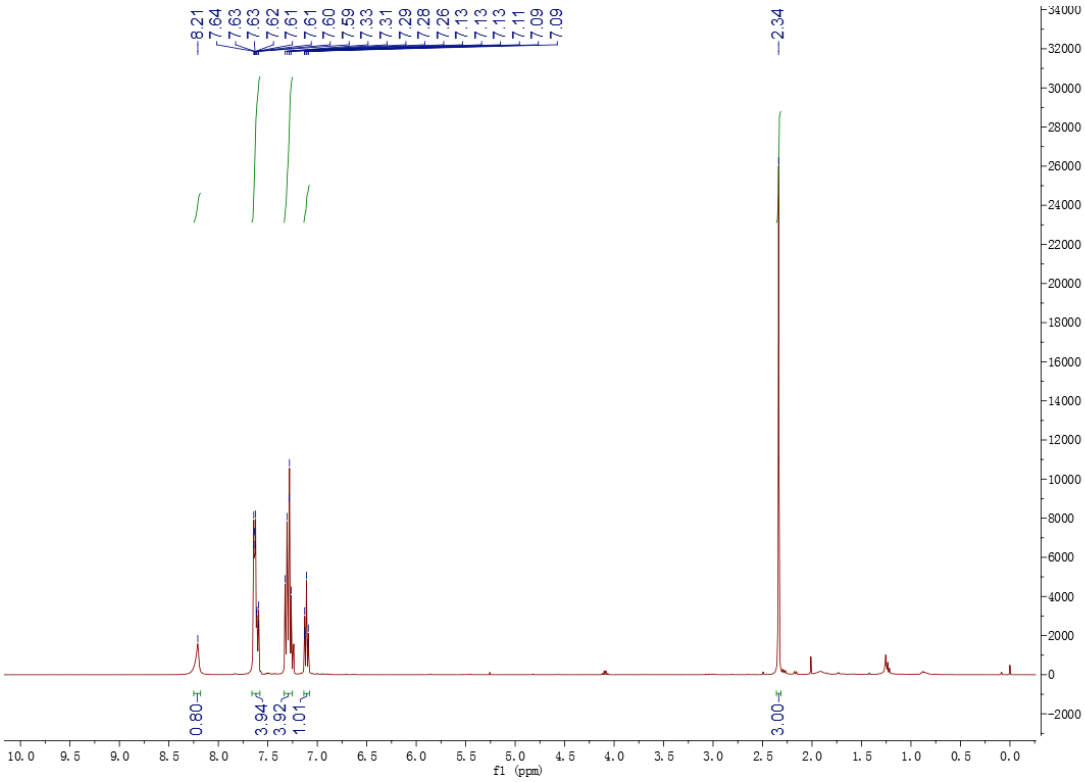
5b

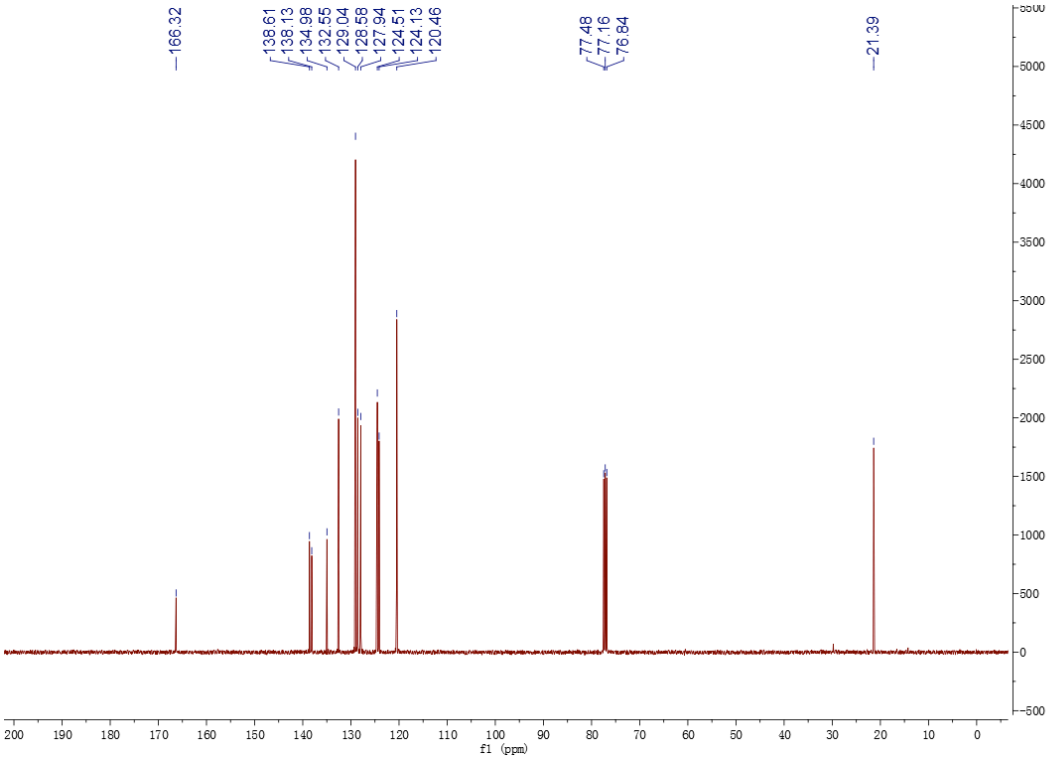

7.67
7.65
7.51
7.49
7.42
7.40
7.38
7.31
7.31
7.29
7.28
7.22
7.20
7.18
2.53
2.86
1.03
3.02
1.99
1.02
3.00
f1 (ppm)

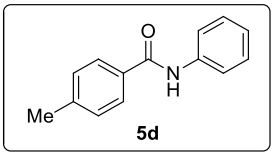

168.26
138.11
136.52
131.35
130.37
129.20
126.73
125.99
124.65
120.01
77.48
77.16
76.84
19.92
f1 (ppm)


Me
O
N
H

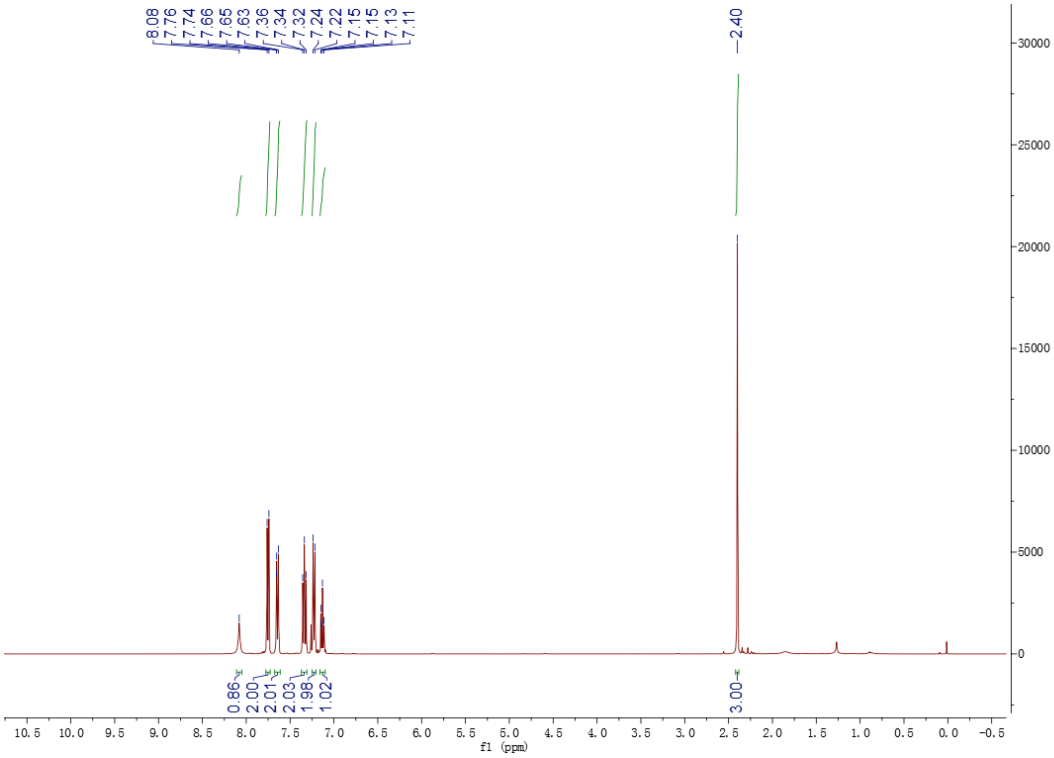
5c


8.21
7.64
7.63
7.62
7.61
7.61
7.60
7.59
7.33
7.31
7.29
7.28
7.26
7.13
7.13
7.13
7.11
7.09
7.09
2.34
0.80
3.94
3.92
1.01
3.00
f1 (ppm)


166.32
138.61
138.13
134.98
132.55
129.04
128.58
127.94
124.51
124.13
120.46
77.48
77.16
76.84
21.39
f1 (ppm)


O
N
H
Me

5d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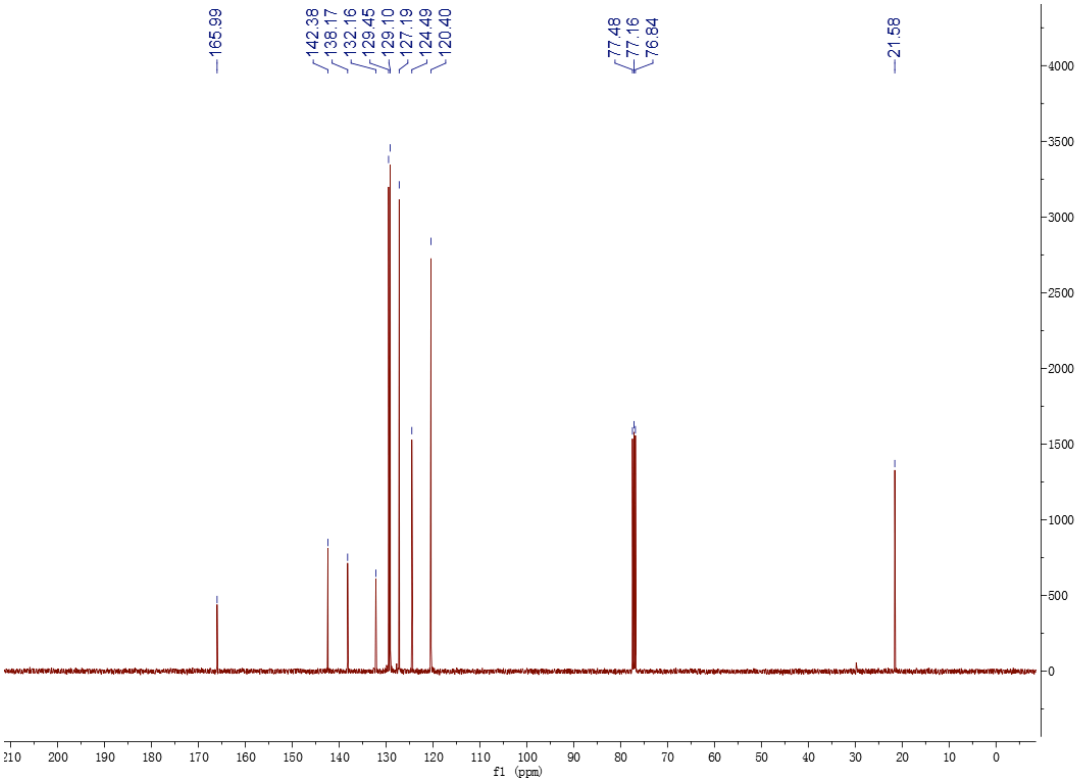
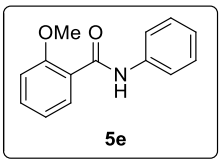
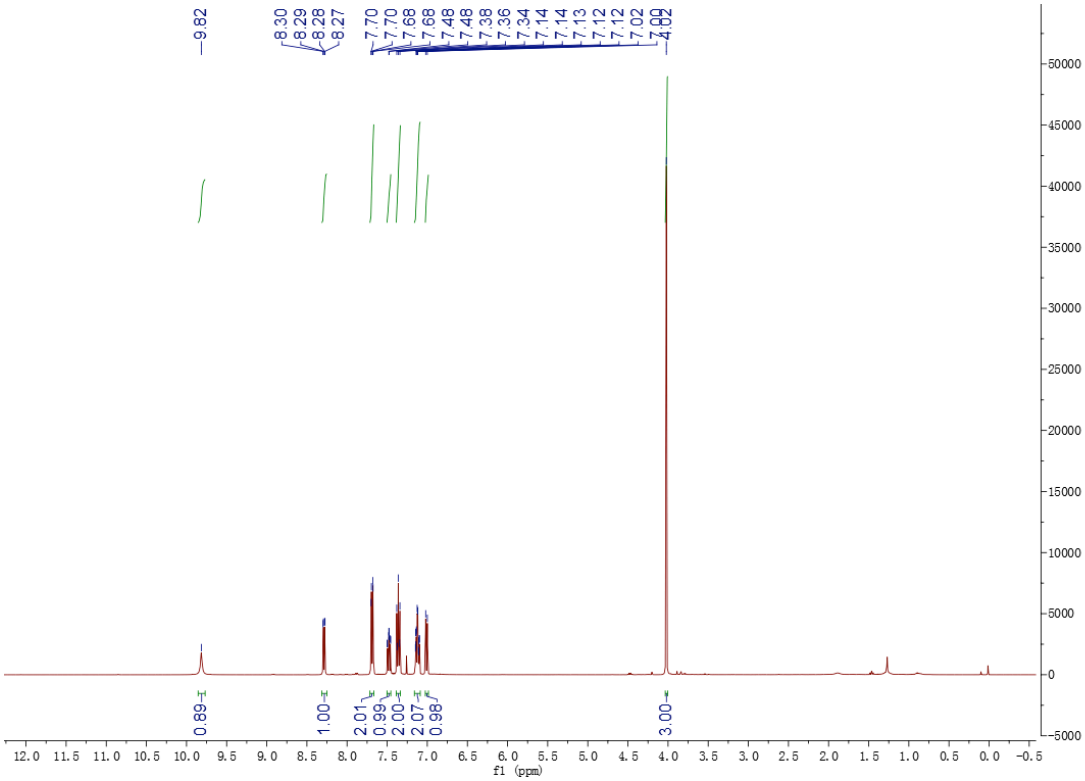
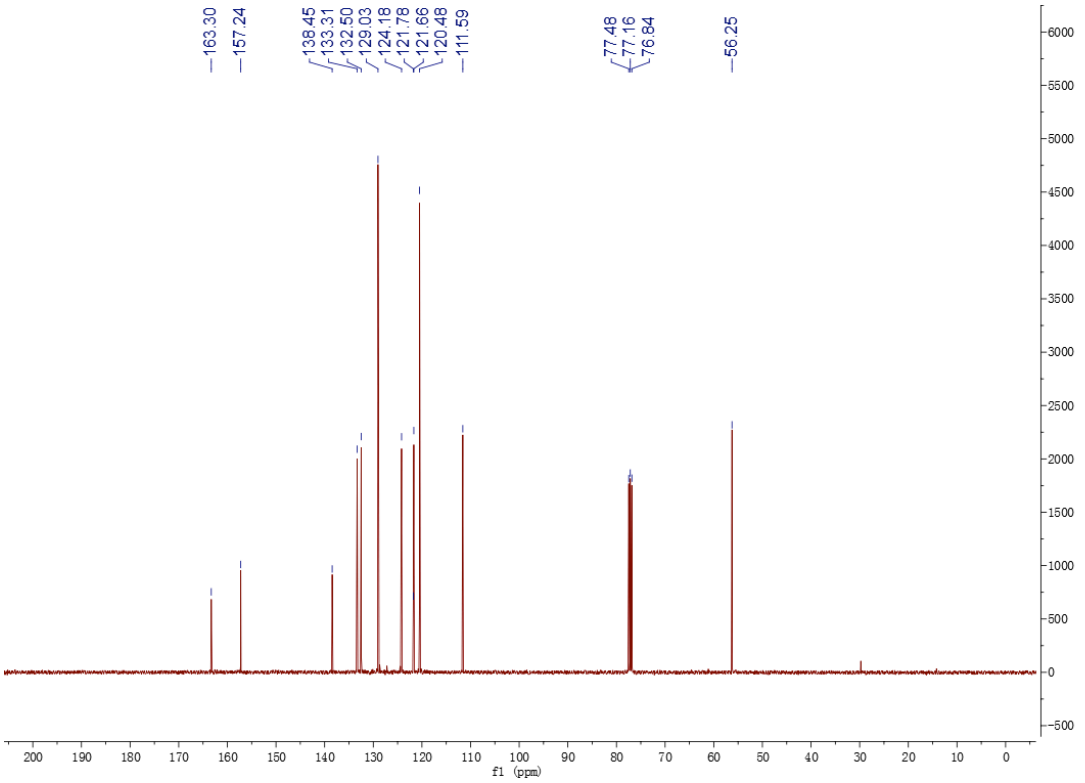
7.76
7.74
7.66
7.65
7.63
7.36
7.34
7.32
7.24
7.22
7.15
7.13
7.11
2.40
0.86
2.00
2.01
2.03
1.98
1.02
3.00
f1 (ppm)

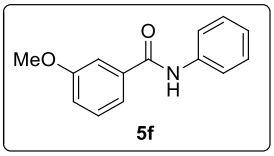

165.99
142.38
138.17
132.16
129.45
129.10
127.19
124.49
120.40
77.48
77.16
76.84
21.58
f1 (ppm)


OMe
O
N
H

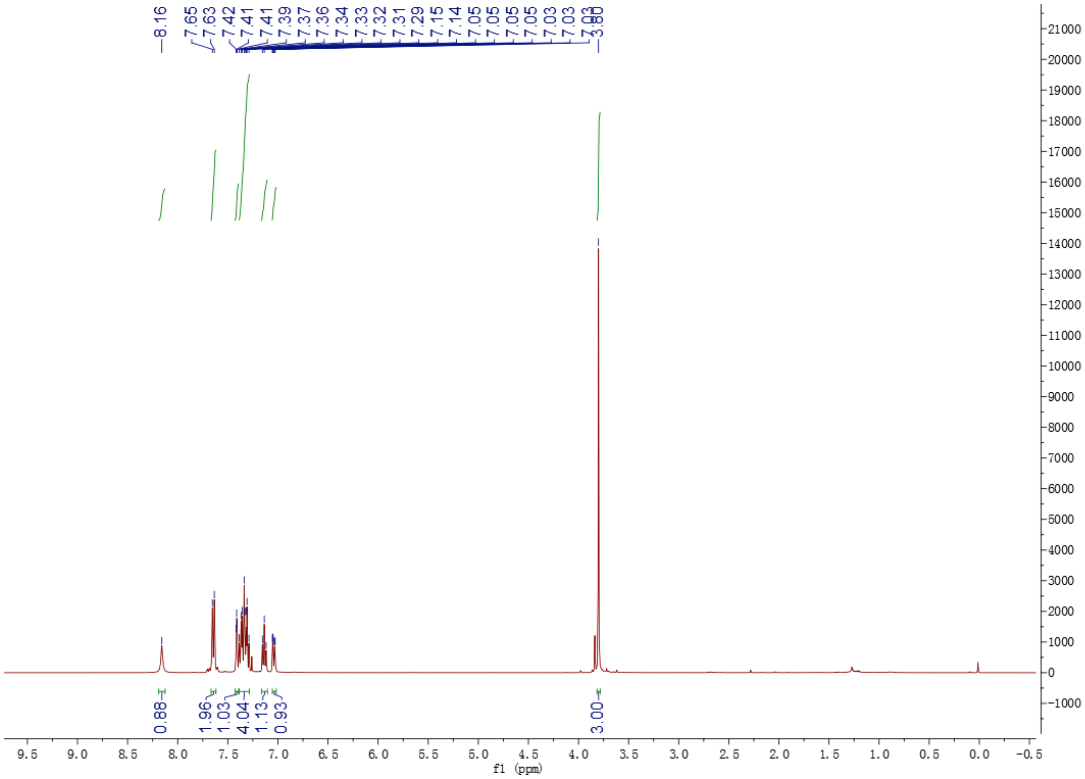
5e


9.82
8.30
8.29
8.28
8.27
7.70
7.70
7.68
7.68
7.48
7.48
7.38
7.36
7.34
7.14
7.14
7.13
7.12
7.12
7.02
7.00
4.02
0.89
1.00
2.01
0.99
2.00
2.07
0.98
3.00
f1 (ppm)


163.30
157.24
138.45
133.31
132.50
129.03
124.18
121.78
121.66
120.48
111.59
77.48
77.16
76.84
56.25
f1 (ppm)


MeO
O
N
H

5f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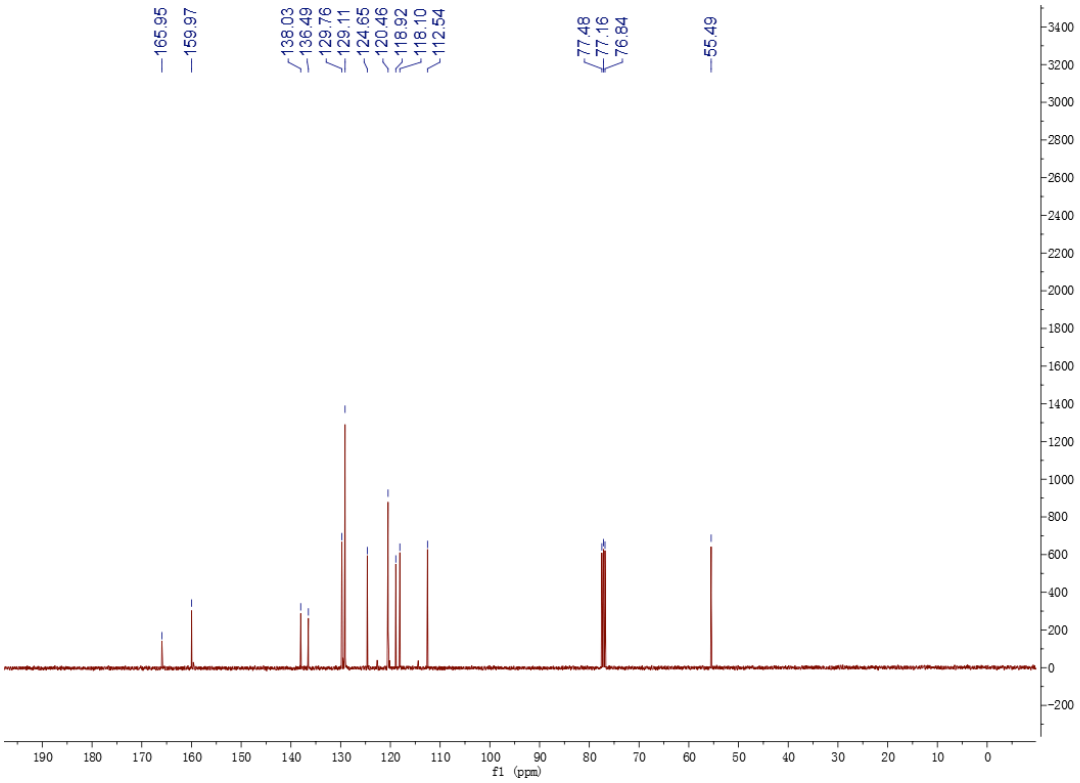
7.37
7.36
7.34
7.33
7.32
7.31
7.29
7.15
7.14
7.05
7.05
7.05
7.05
7.03
7.03
3.90
0.88
1.96
1.03
4.04
1.13
0.93
3.00
f1 (ppm)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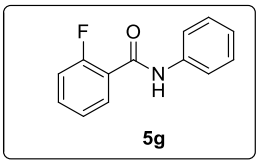
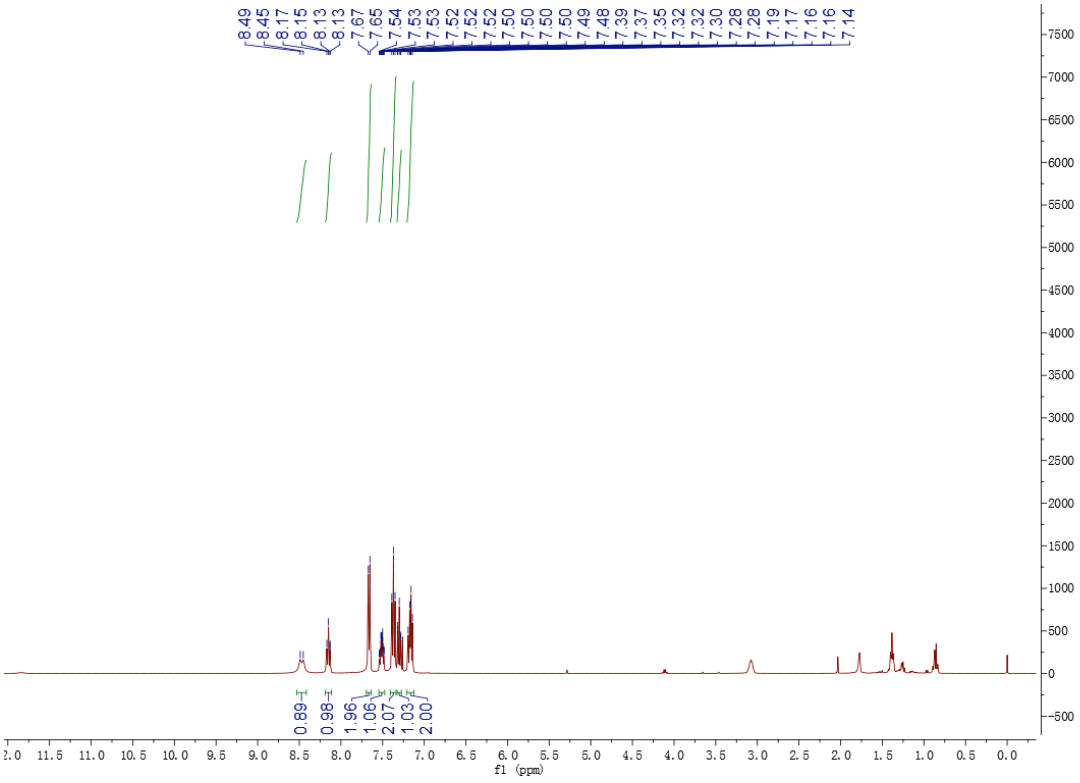
159.97
138.03
136.49
129.76
129.11
124.65
120.46
118.92
118.10
112.54
77.48
77.16
76.84
55.49
f1 (ppm)


F
O
N
H

5g


8.49
8.45
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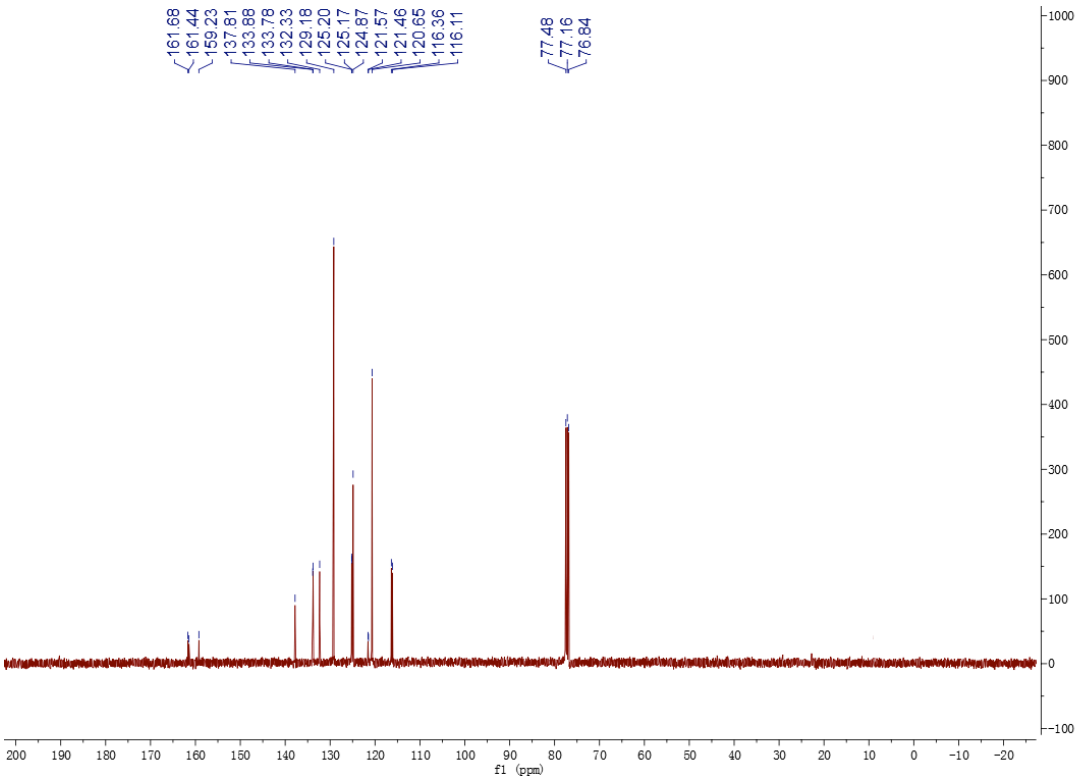
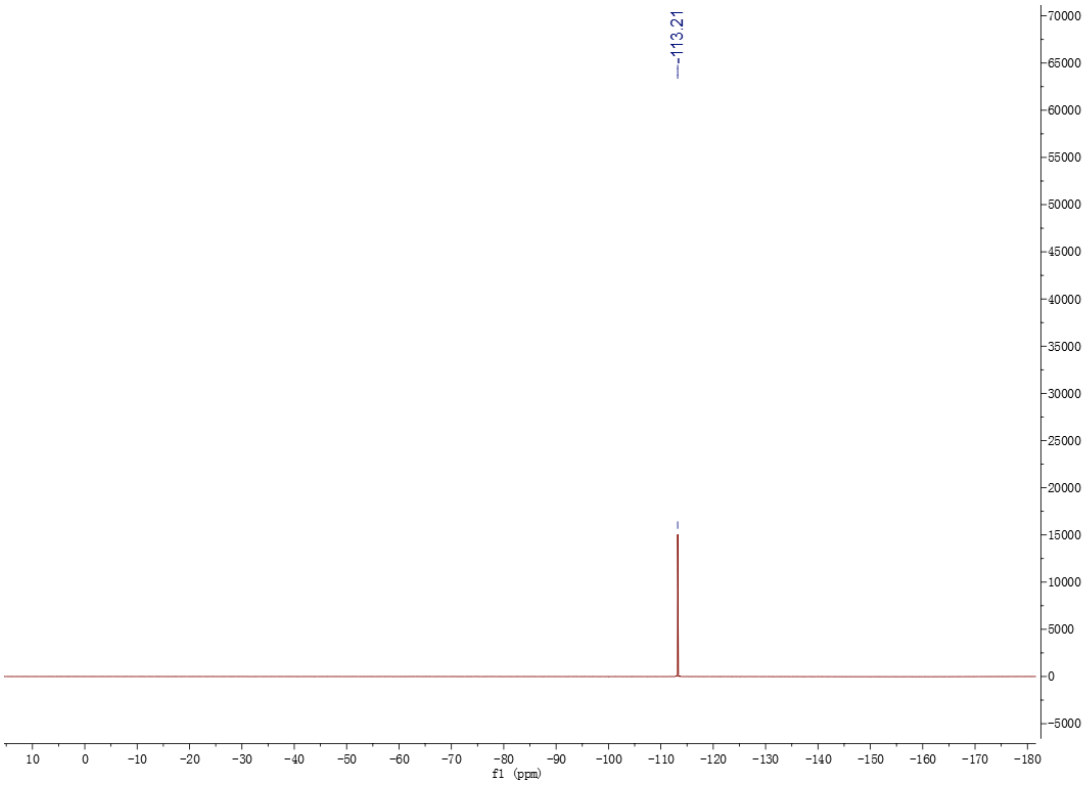
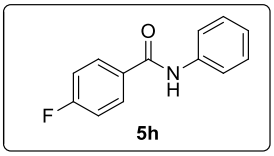
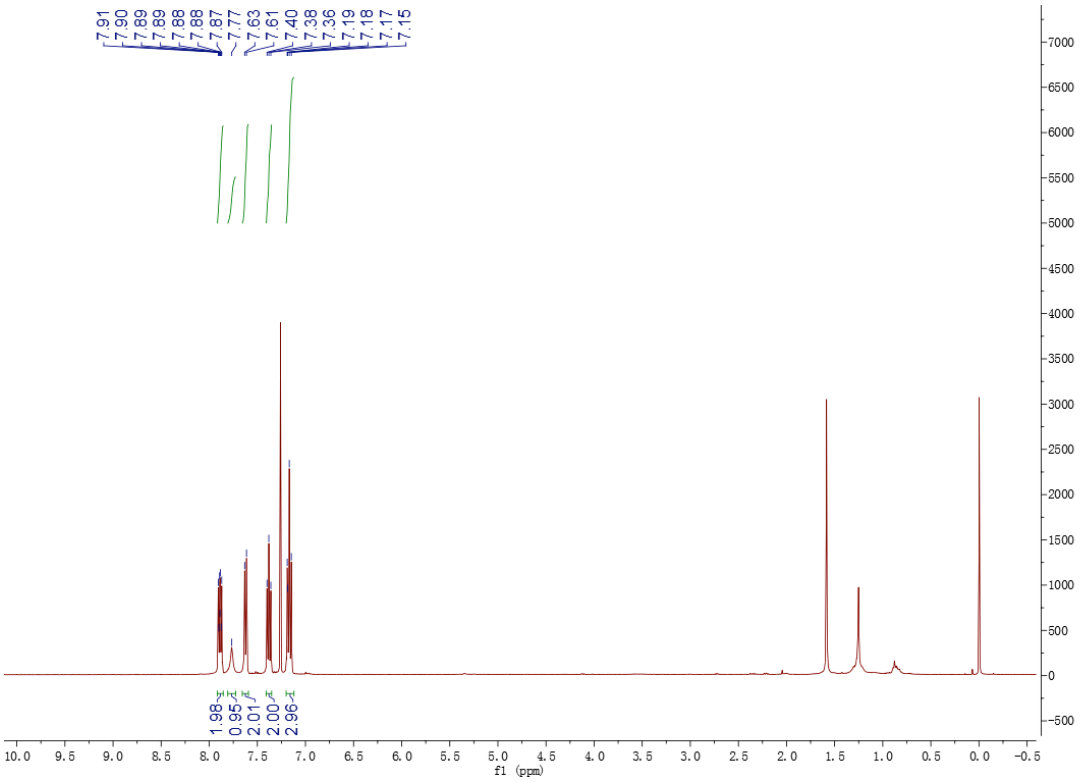
8.15
8.13
8.13
7.67
7.65
7.54
7.53
7.53
7.52
7.52
7.52
7.50
7.50
7.50
7.50
7.49
7.48
7.39
7.37
7.35
7.32
7.32
7.30
7.28
7.28
7.19
7.17
7.16
7.16
7.14
0.89
0.98
1.96
1.06
2.07
1.03
2.00
f1 (ppm)

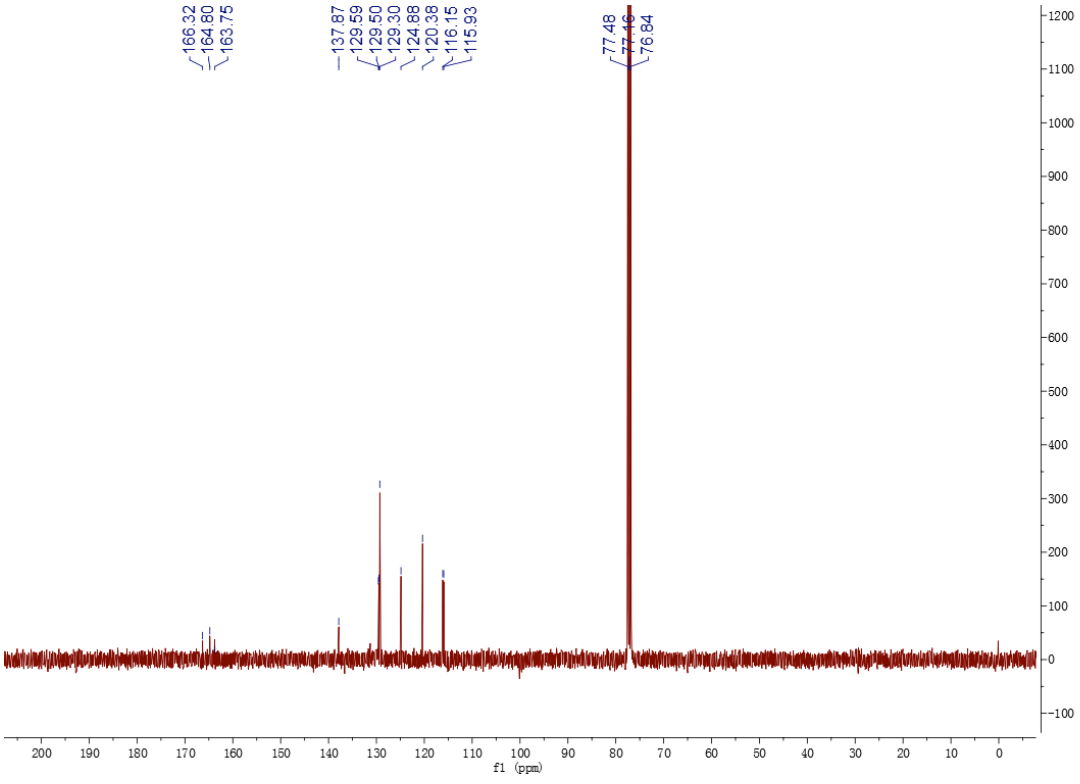

161.68
161.44
159.23
137.81
133.88
133.78
132.33
129.18
125.20
125.17
124.87
121.57
121.46
120.65
116.36
116.11
77.48
77.16
76.84
f1 (ppm)

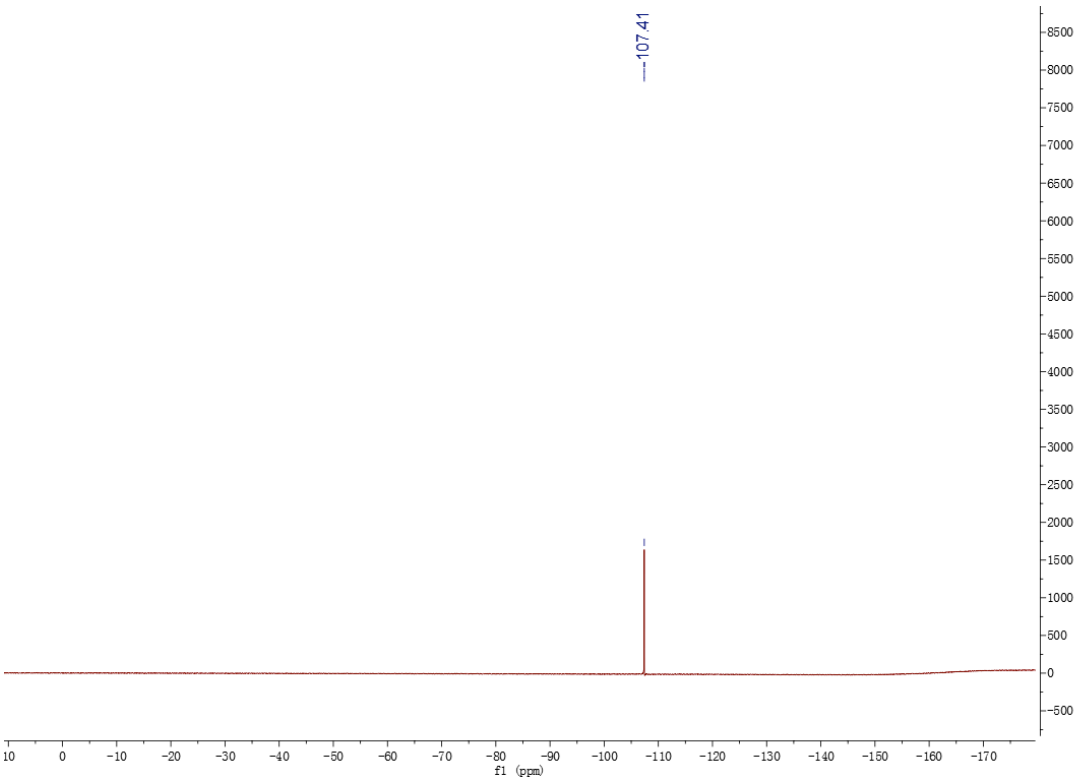

113.21
f1 (ppm)

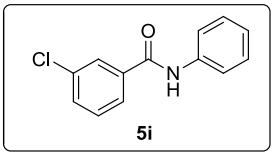

O
N
H
F

5h

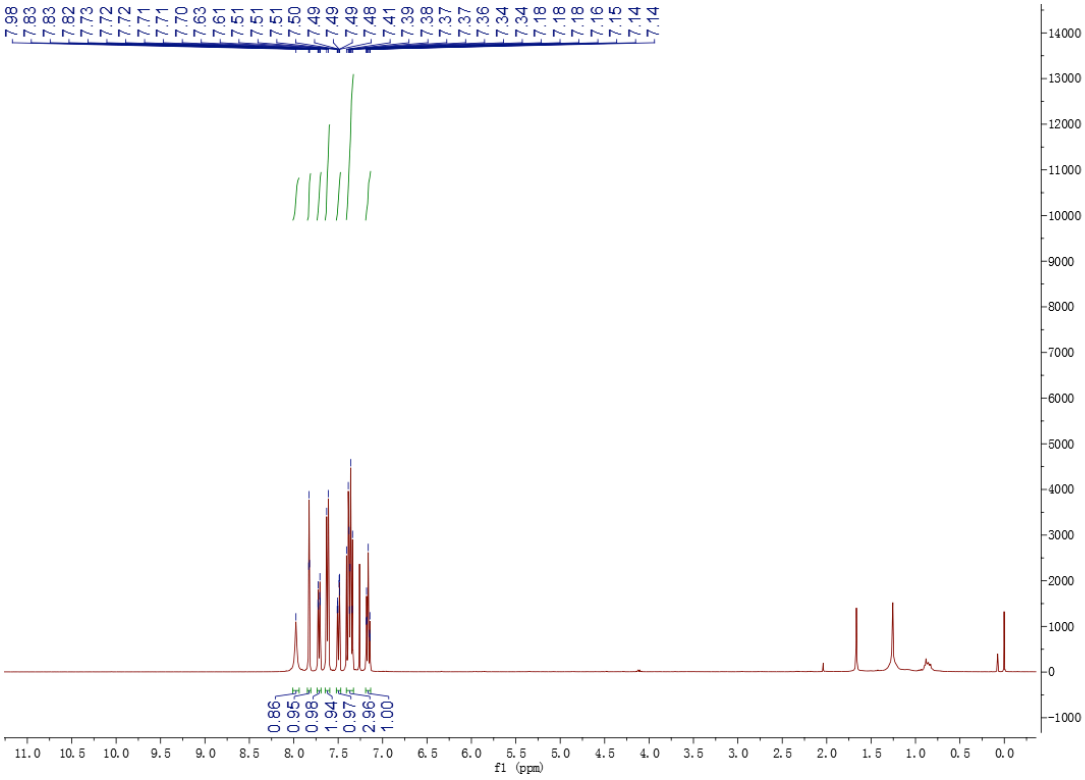

7.91
7.90
7.89
7.89
7.88
7.88
7.87
7.77
7.63
7.61
7.40
7.38
7.36
7.19
7.18
7.17
7.15
1.98
0.95
2.01
2.00
2.96
f1 (ppm)


166.32
164.80
163.75
137.87
129.59
129.50
129.30
124.88
120.38
116.15
115.93
77.48
77.16
76.84
f1 (ppm)


-107.41
f1 (ppm)


Cl
O
N
H

5i


7.98
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7.83
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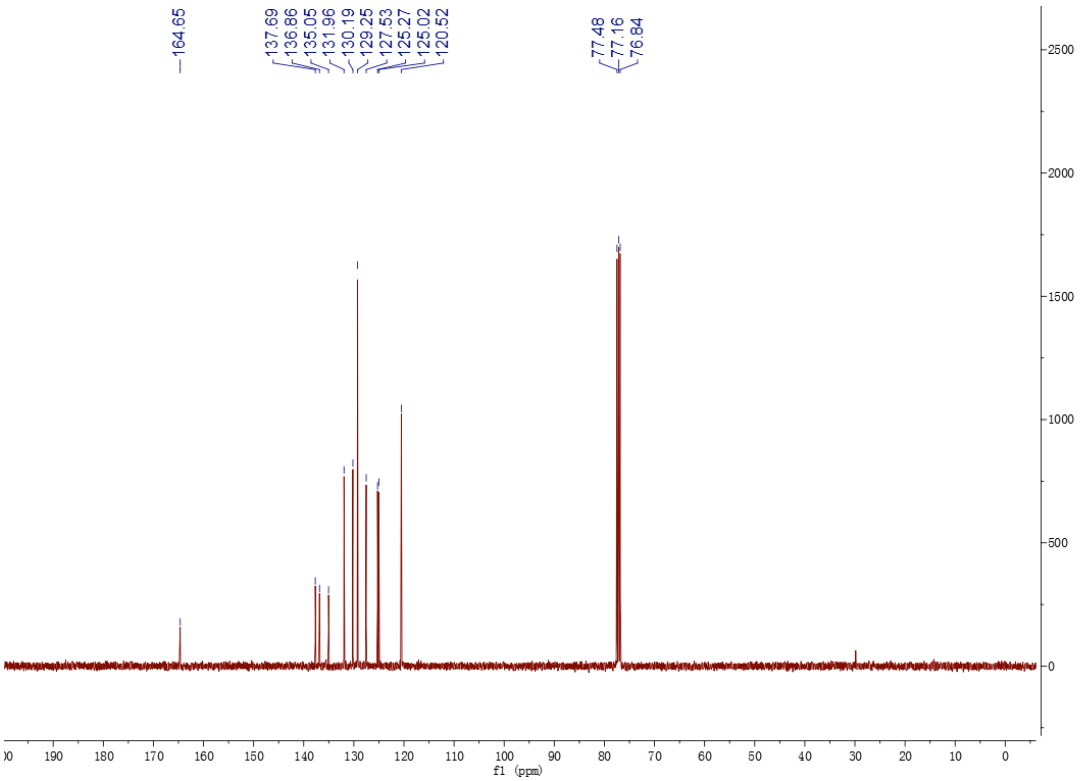
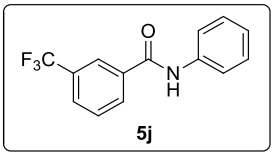
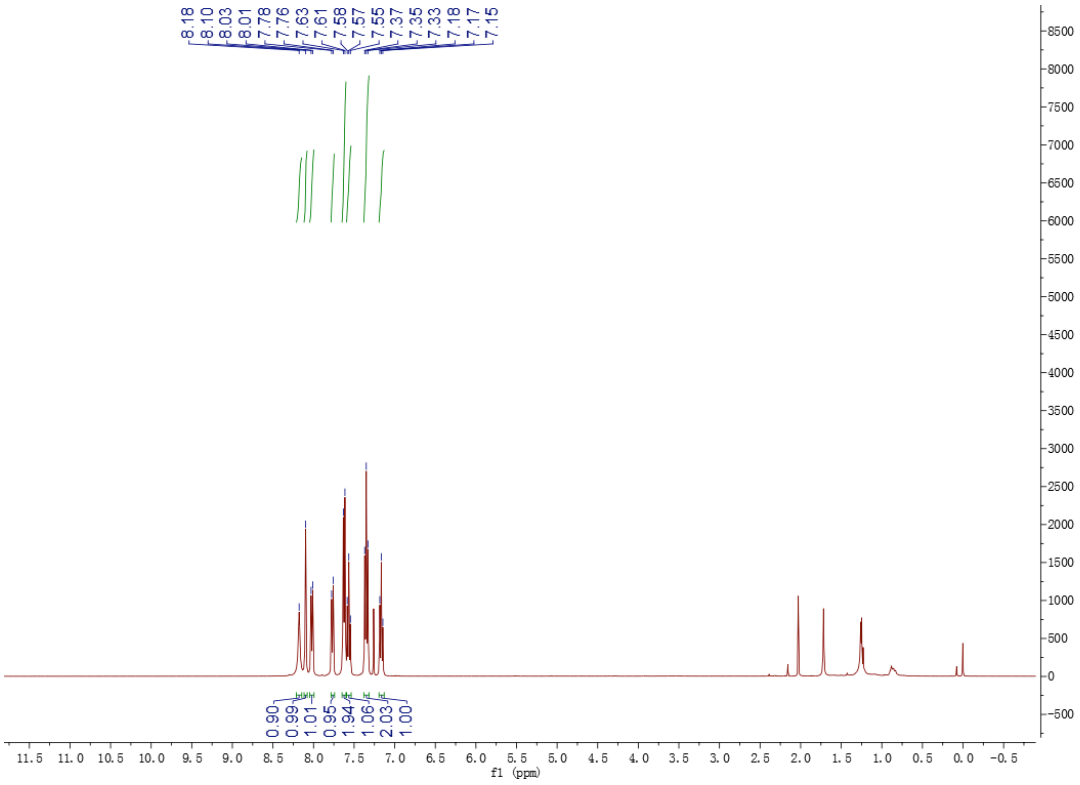
7.73
7.72
7.72
7.71
7.71
7.70
7.63
7.61
7.51
7.51
7.51
7.50
7.49
7.49
7.49
7.48
7.41
7.39
7.38
7.37
7.37
7.36
7.34
7.34
7.18
7.18
7.18
7.16
7.15
7.14
7.14
0.86
0.95
0.98
1.94
0.97
2.96
1.00
f1 (ppm)


164.65
137.69
136.86
135.05
131.96
130.19
129.25
127.53
125.27
125.02
120.52
77.48
77.16
76.84
f1 (ppm)


O
F3C
N
H

5j


8.18
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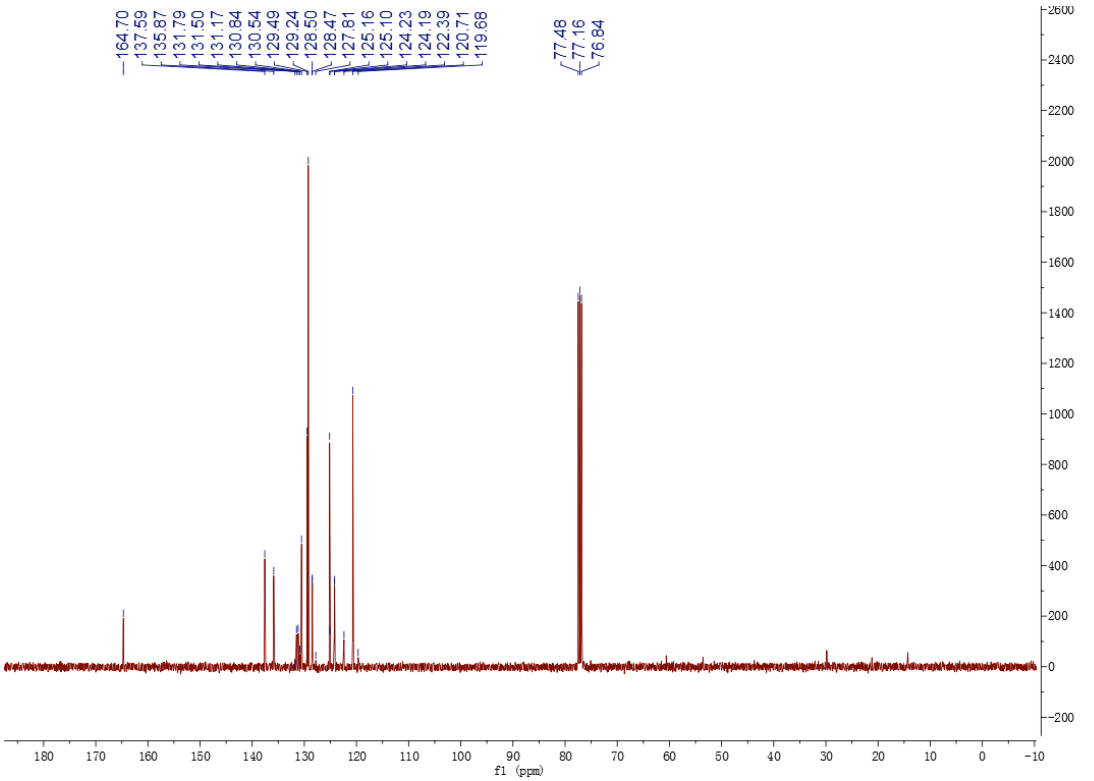
8.03
8.01
7.78
7.76
7.63
7.61
7.58
7.57
7.55
7.37
7.35
7.33
7.18
7.17
7.15
0.90
0.99
1.01
0.95
1.94
1.06
2.03
1.00
f1 (ppm)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131.17
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130.54
129.49
129.24
128.50
128.47
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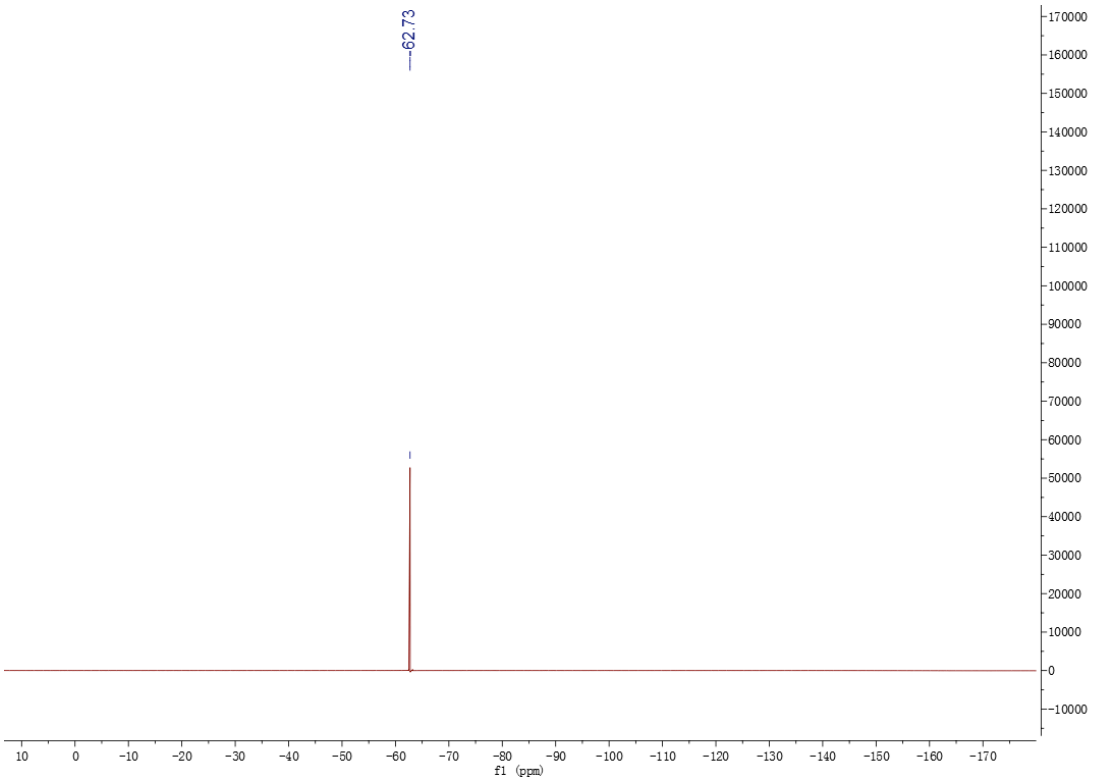
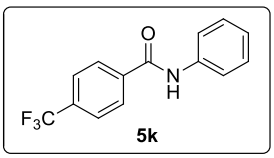
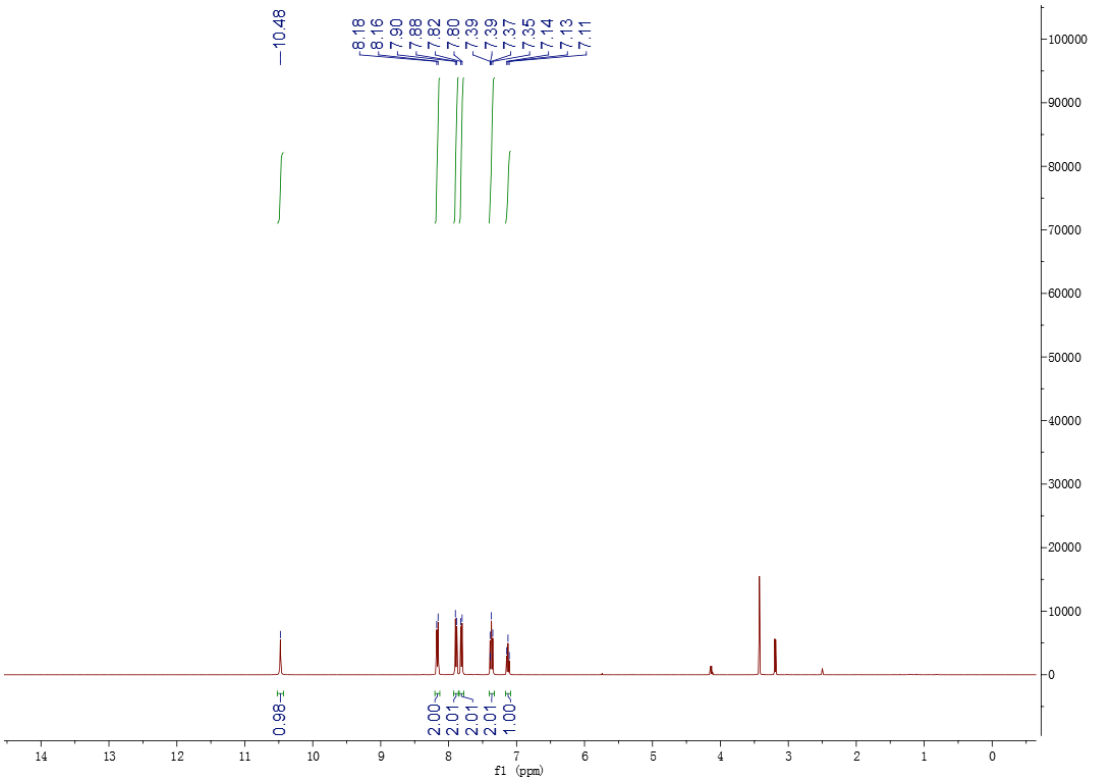
125.16
125.10
124.23
124.19
122.39
120.71
119.68
77.48
77.16
76.84
f1 (ppm)


-62.73
f1 (ppm)


O
N
H
F3C

5k


10.48
8.18
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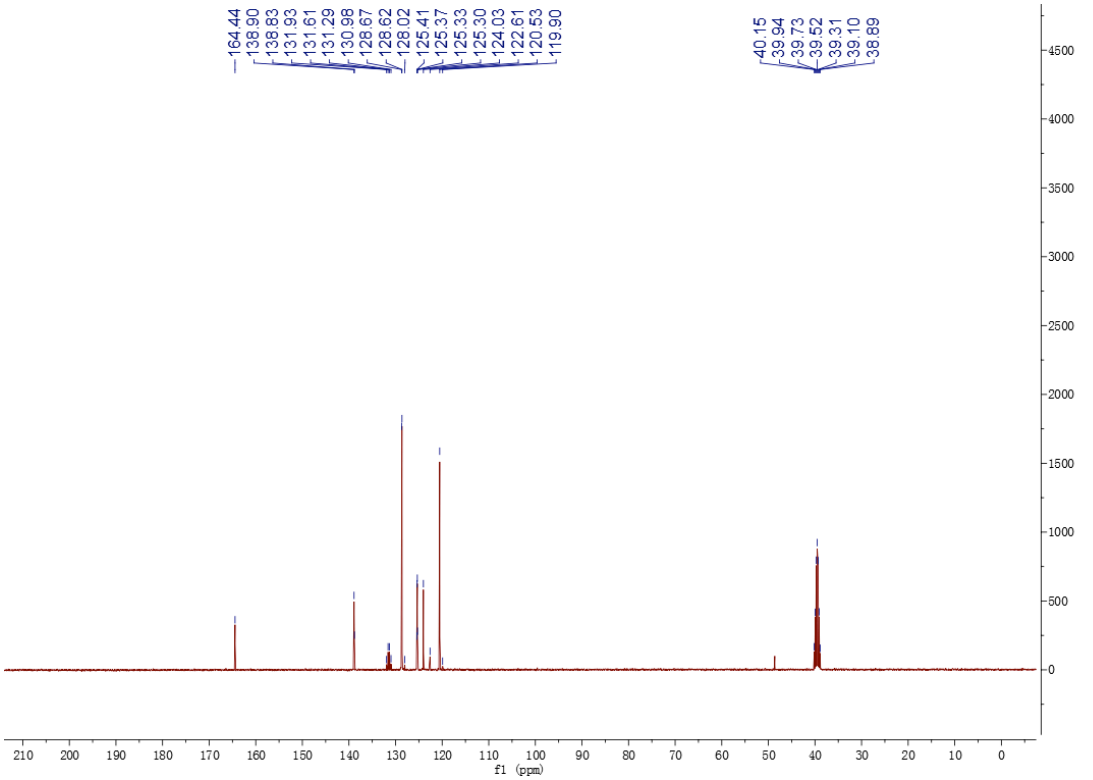
7.88
7.82
7.80
7.39
7.39
7.37
7.35
7.14
7.13
7.11
0.98
2.00
2.01
2.01
2.01
1.00
f1 (ppm)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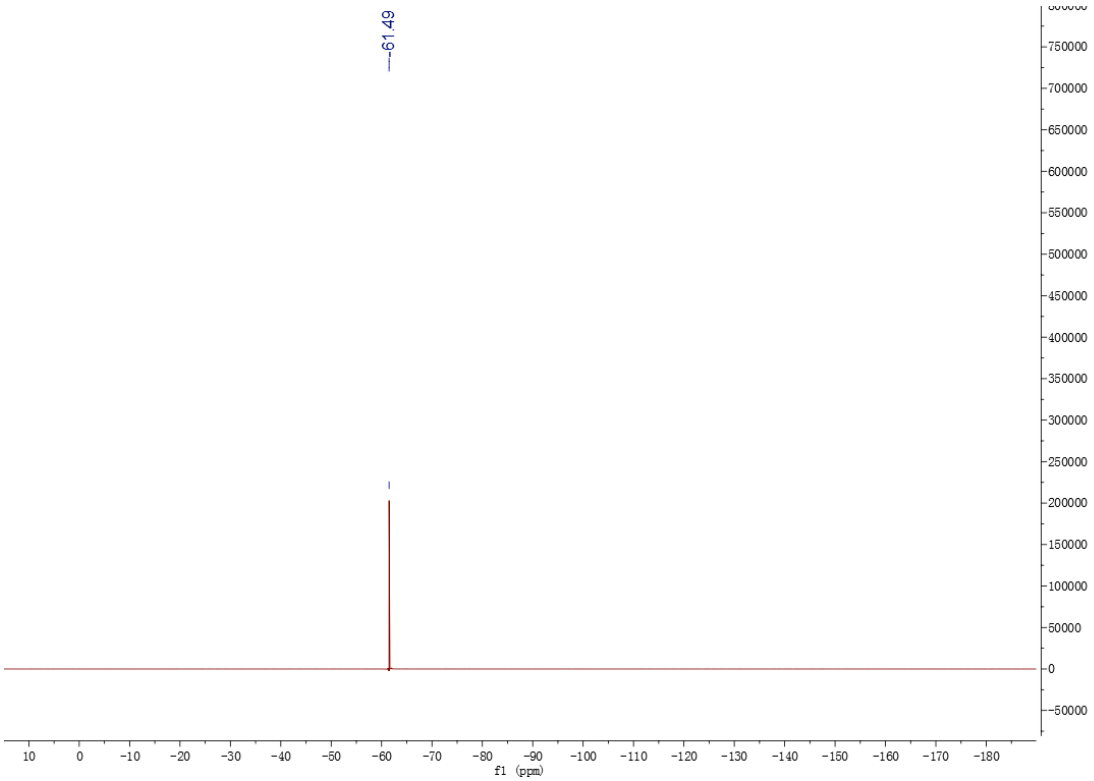
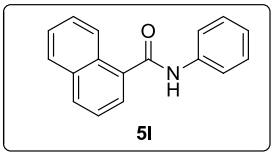
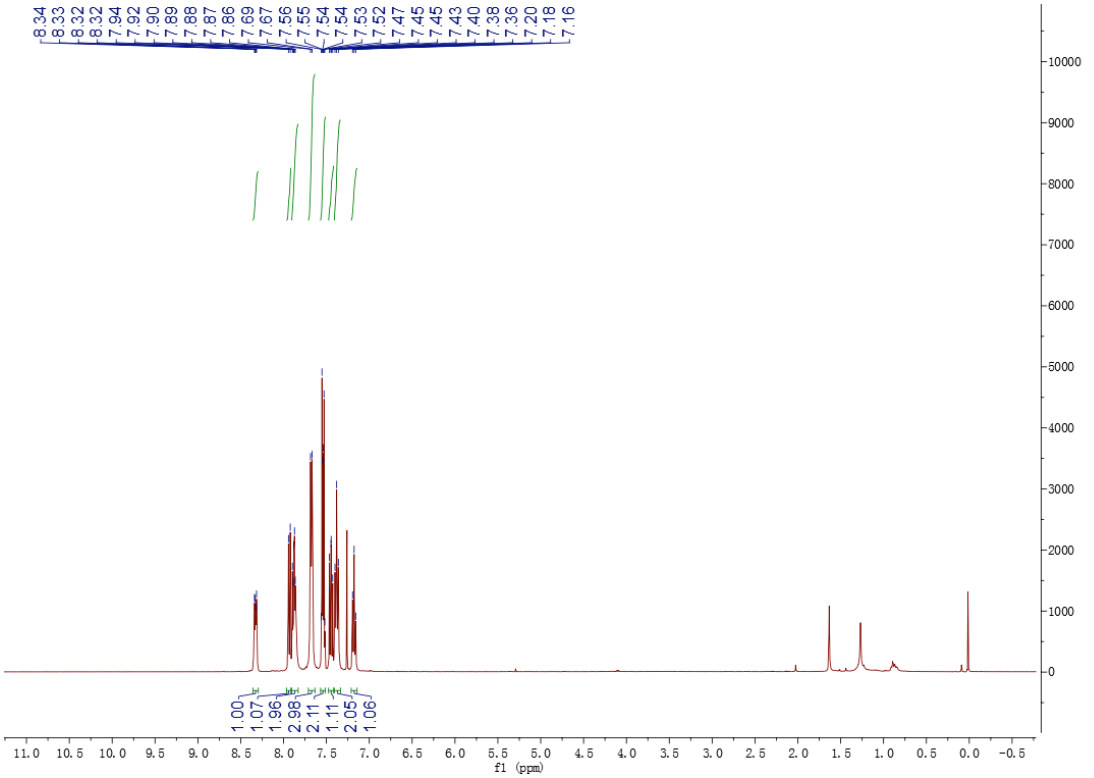
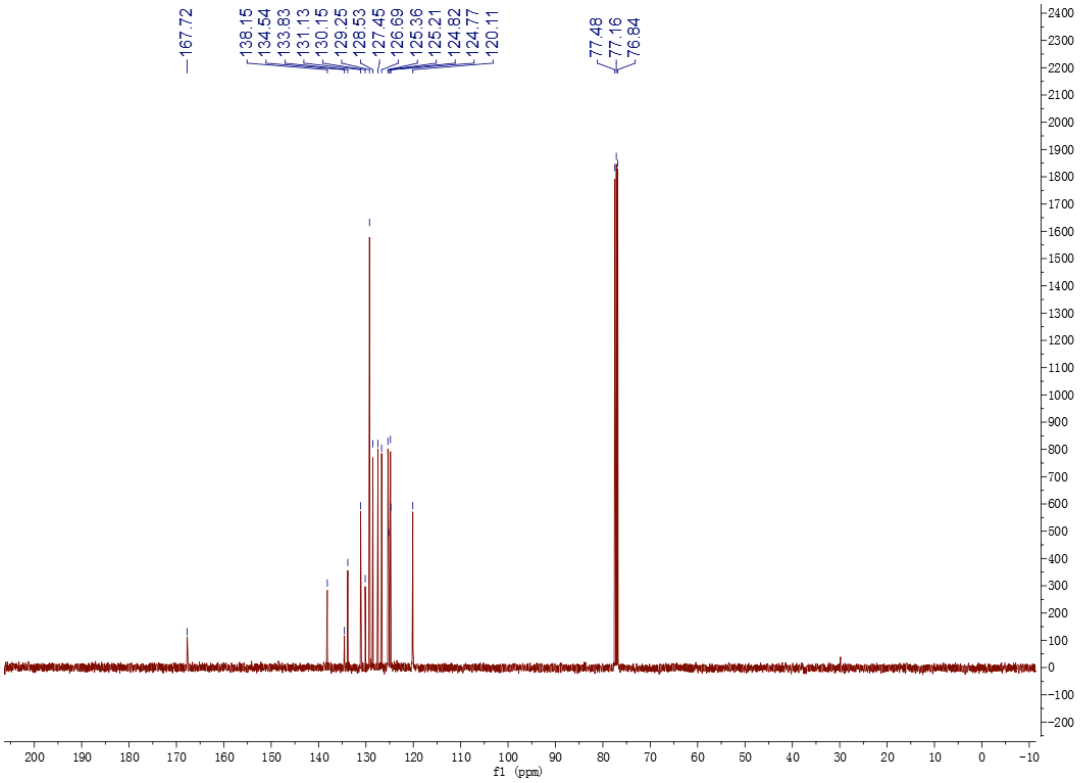
128.67
128.62
128.02
125.41
125.37
125.33
125.30
124.03
122.61
120.53
119.90
40.15
39.94
39.73
39.52
39.31
39.10
38.89
f1 (ppm)

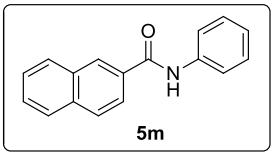

-61.49
f1 (ppm)


O
N
H

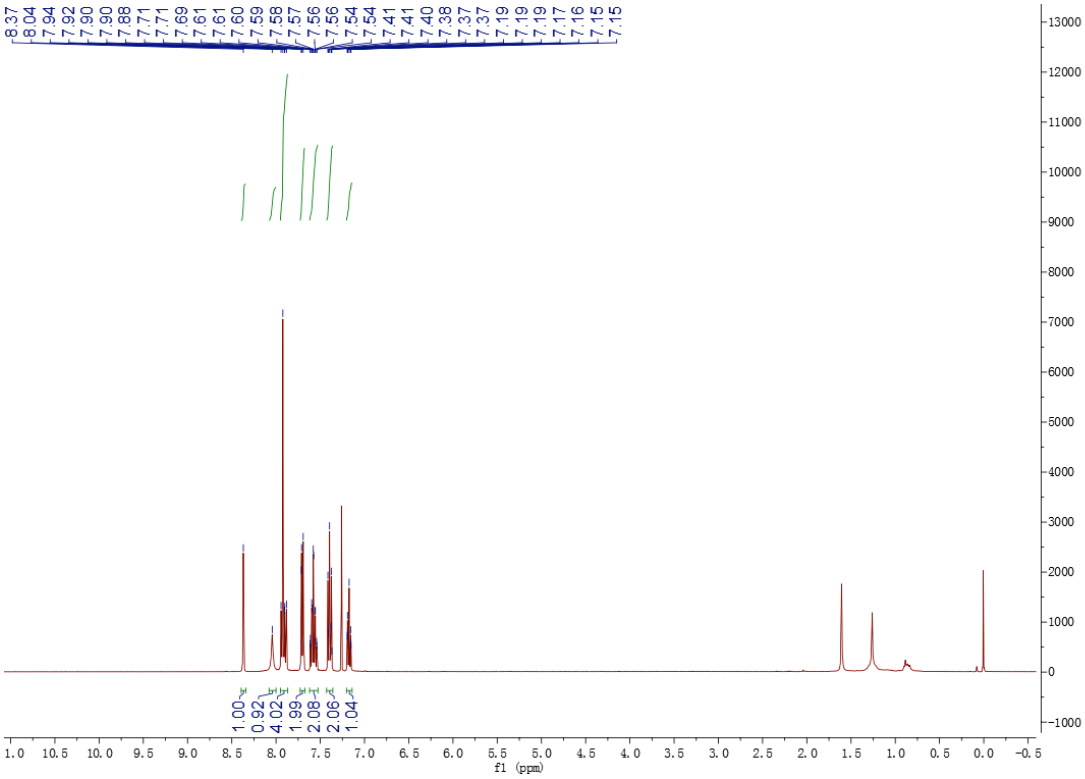
5l


8.34
8.33
8.32
8.32
7.94
7.92
7.90
7.89
7.88
7.87
7.86
7.69
7.67
7.56
7.55
7.54
7.54
7.53
7.52
7.47
7.45
7.45
7.43
7.40
7.38
7.36
7.20
7.18
7.16
1.00
1.07
1.96
2.98
2.11
1.11
2.05
1.06
f1 (ppm)


167.72
138.15
134.54
133.83
131.13
130.15
129.25
128.53
127.45
126.69
125.36
125.21
124.82
124.77
120.11
77.48
77.16
76.84
f1 (ppm)


O
N
H

5m


8.37
8.04
7.94
7.92
7.90
7.90
7.88
7.71
7.71
7.69
7.61
7.61
7.60
7.59
7.58
7.57
7.56
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7.54
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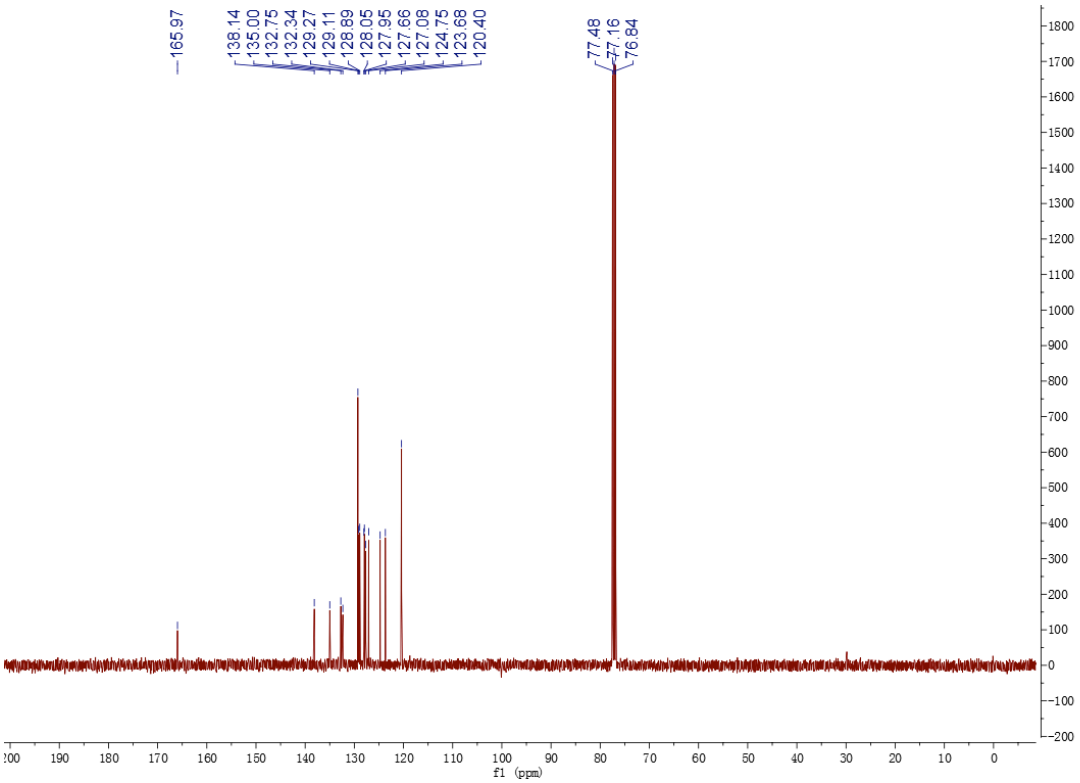
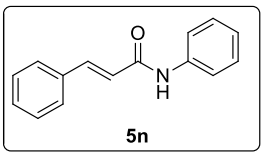
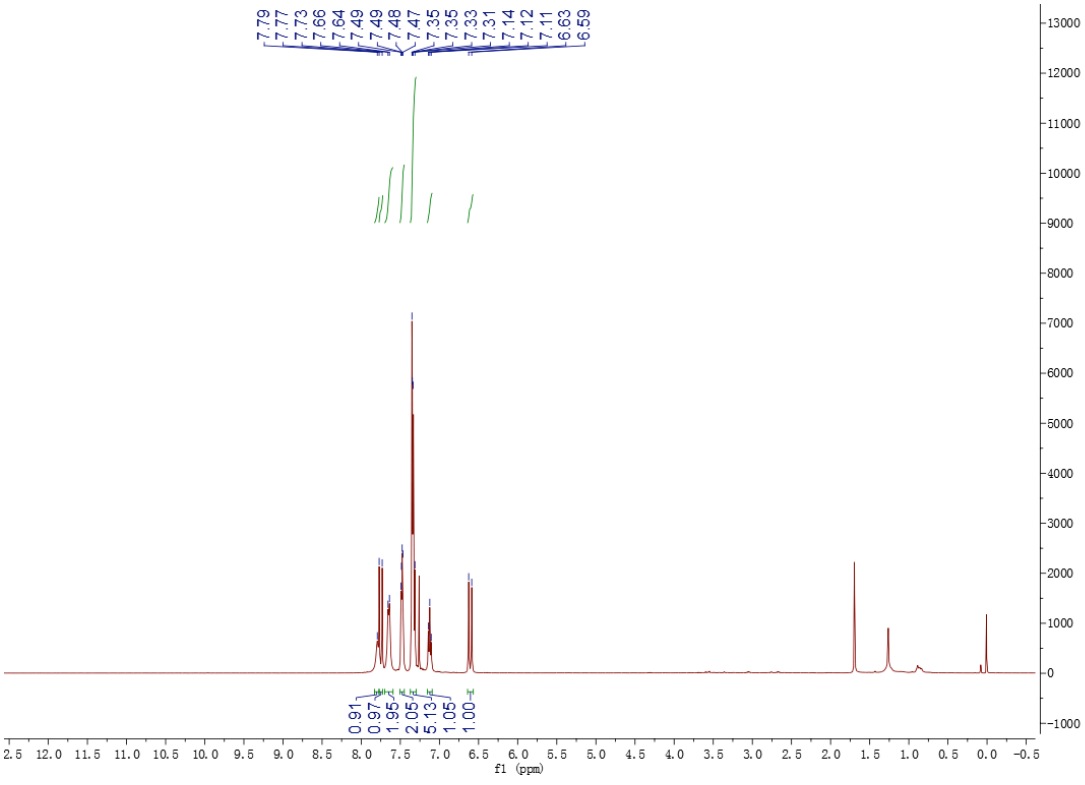
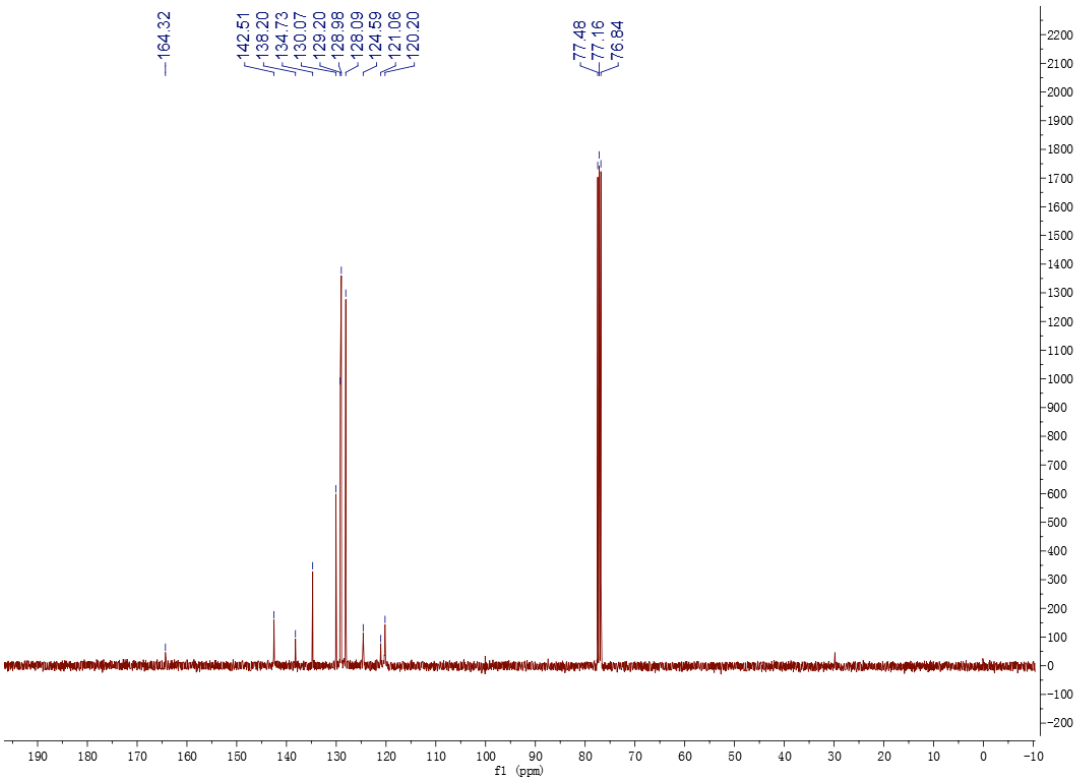
7.41
7.40
7.38
7.37
7.37
7.19
7.19
7.19
7.17
7.16
7.15
7.15
1.00
0.92
4.02
1.99
2.08
2.06
1.04
f1 (ppm)

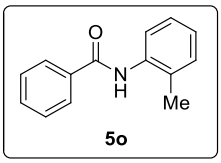

165.97
138.14
135.00
132.75
132.34
129.27
129.11
128.89
128.05
127.95
127.66
127.08
124.75
123.68
120.40
77.48
77.16
76.84
f1 (ppm)


O
N
H

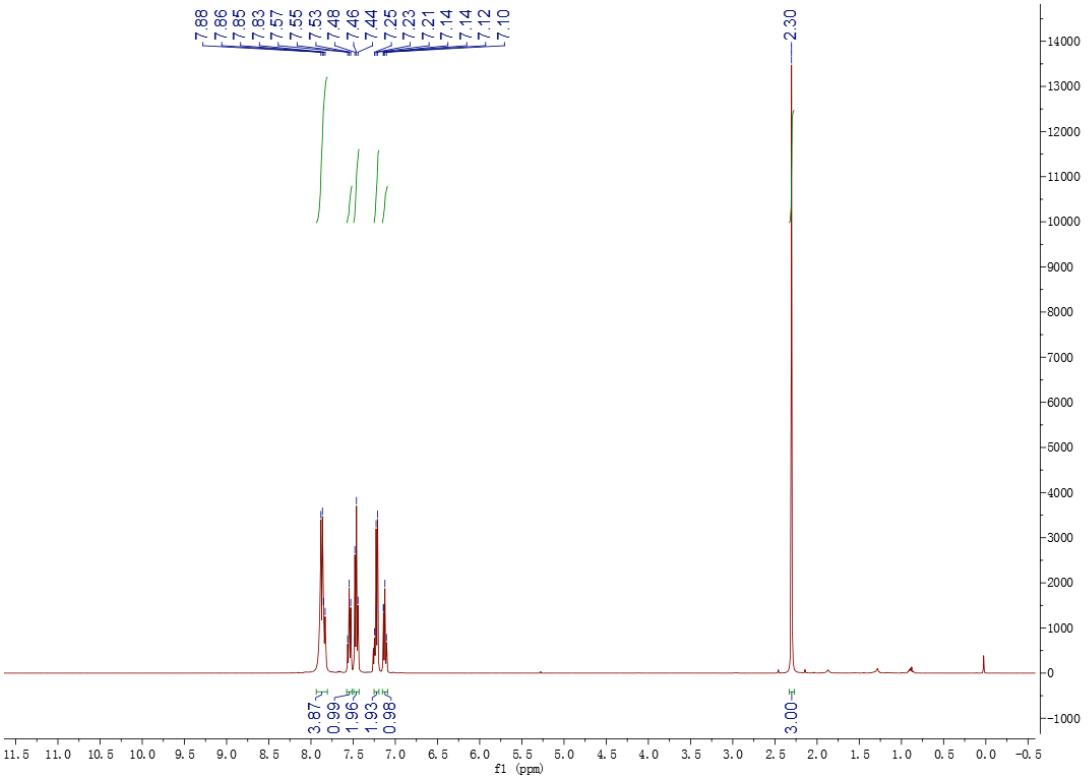
5n

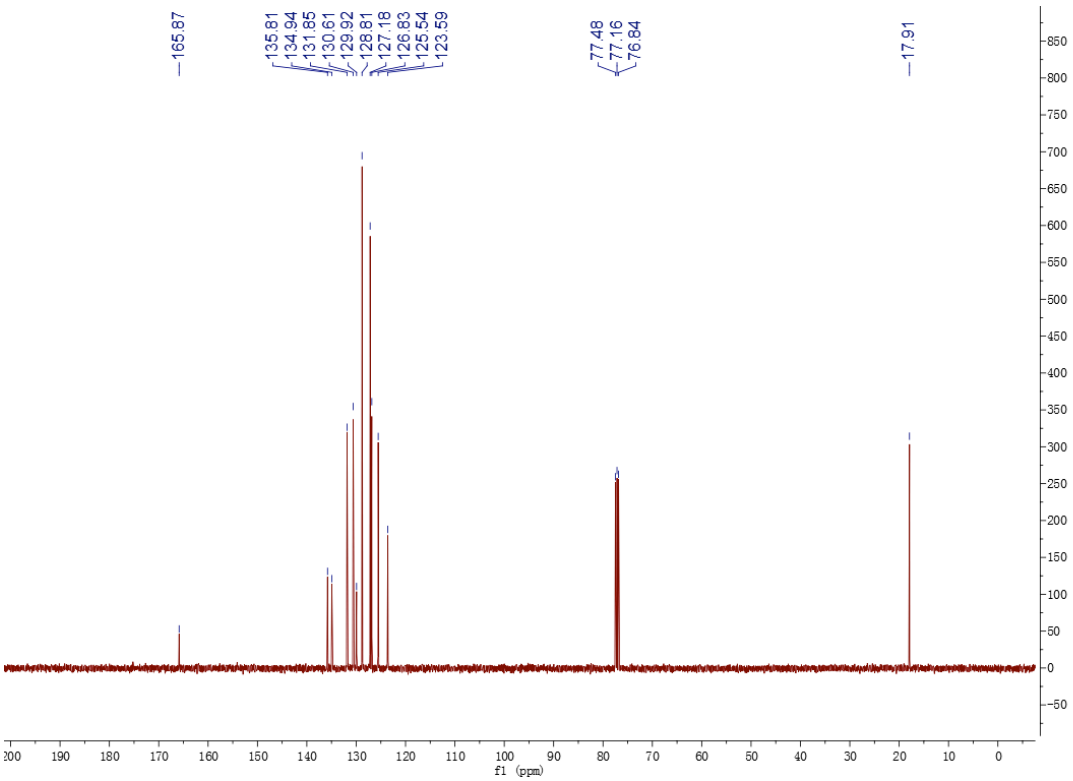

7.79
7.77
7.73
7.66
7.64
7.49
7.49
7.48
7.47
7.35
7.35
7.33
7.31
7.14
7.12
7.11
6.63
6.59
0.91
0.97
1.95
2.05
5.13
1.05
1.00
f1 (ppm)


164.32
142.51
138.20
134.73
130.07
129.20
128.98
128.09
124.59
121.06
120.20
77.48
77.16
76.84
f1 (ppm)


O
N
H
Me

5o


7.88
7.86
7.85
7.83
7.57
7.55
7.53
7.48
7.46
7.44
7.25
7.23
7.21
7.14
7.12
7.10
2.30
3.87
0.99
1.96
1.93
0.98
3.00
f1 (ppm)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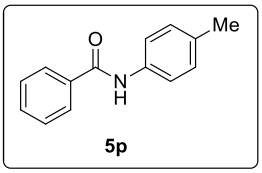
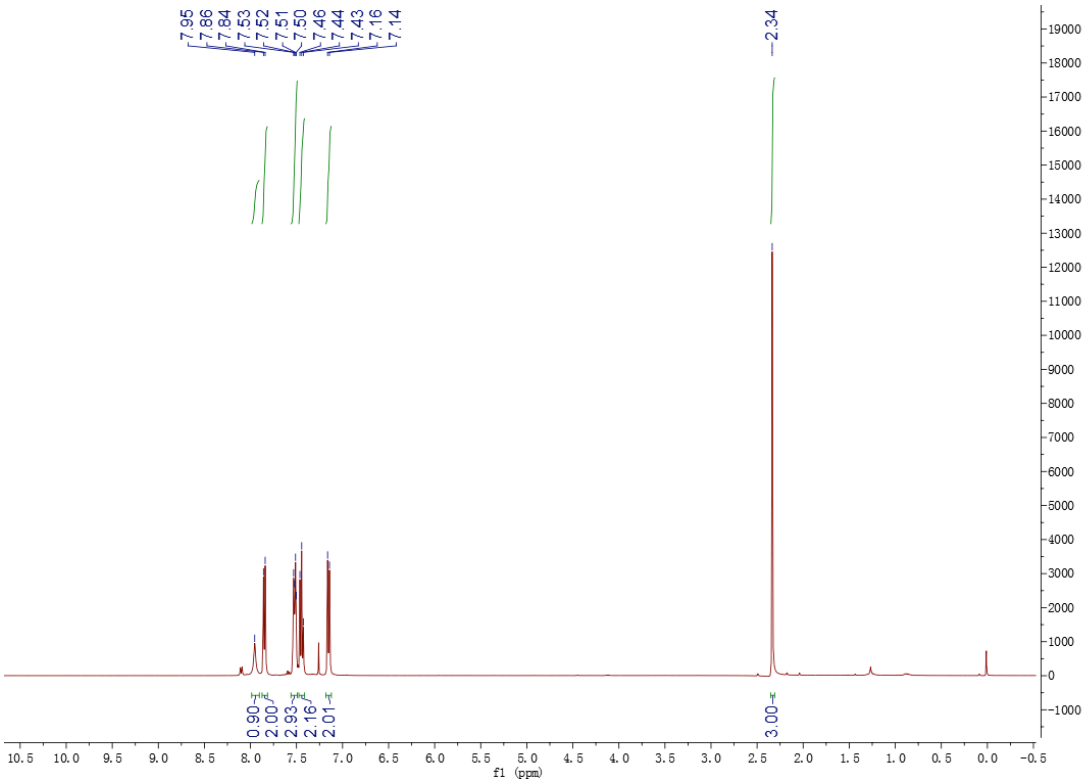
135.81
134.94
131.85
130.61
129.92
128.81
127.18
126.83
125.54
123.59
77.48
77.16
76.84
17.91
f1 (ppm)


O
N
H
Me

5p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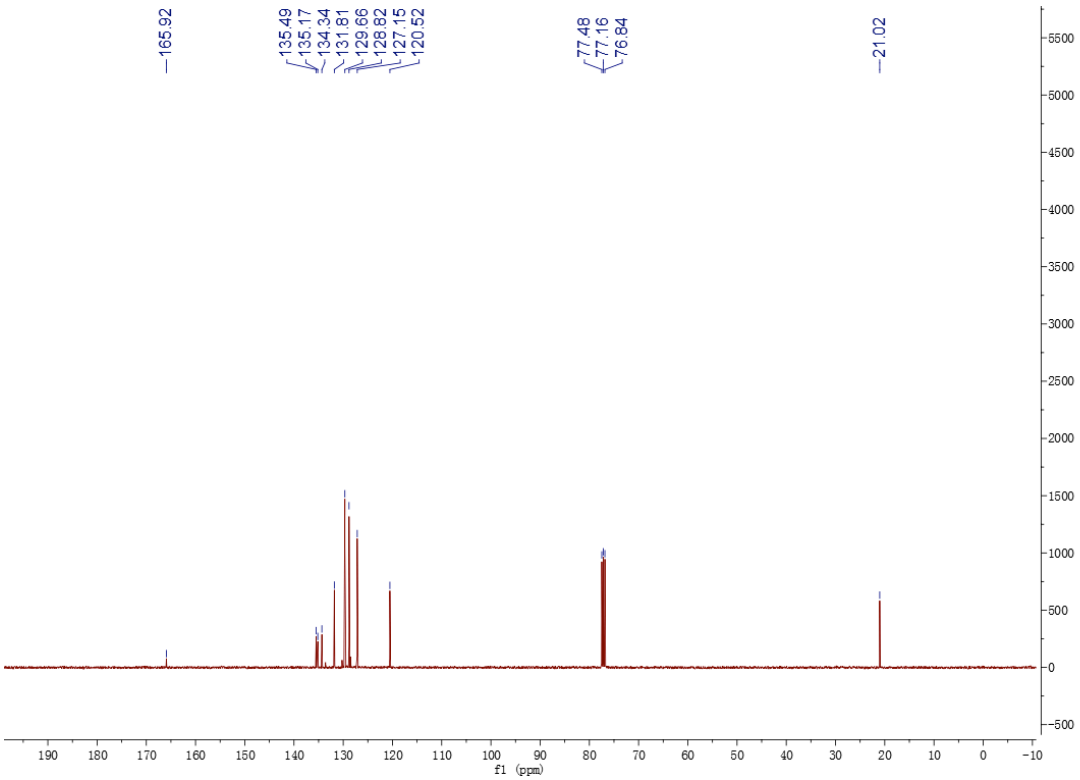
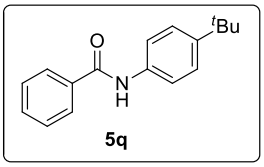
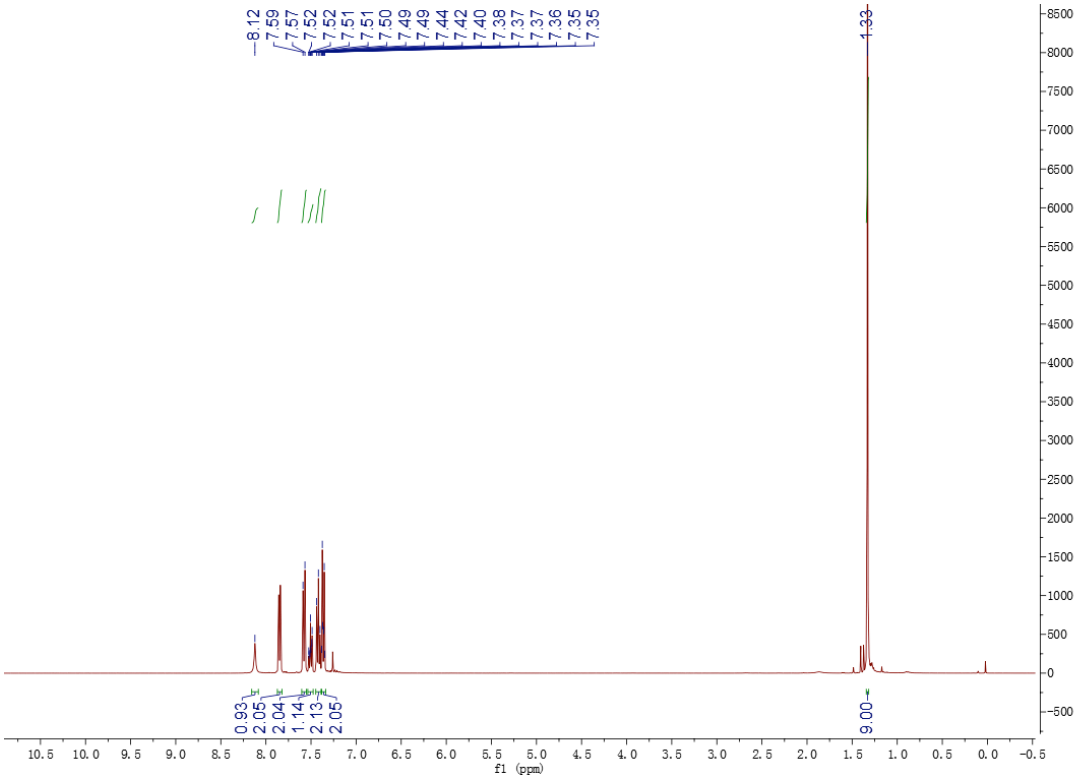
7.86
7.84
7.53
7.52
7.51
7.50
7.46
7.44
7.43
7.16
7.14
2.34
0.90
2.00
2.93
2.16
2.01
3.00
f1 (ppm)


165.92
135.49
135.17
134.34
131.81
129.66
128.82
127.15
120.52
77.48
77.16
76.84
21.02
f1 (ppm)


O
N
H
tBu
5q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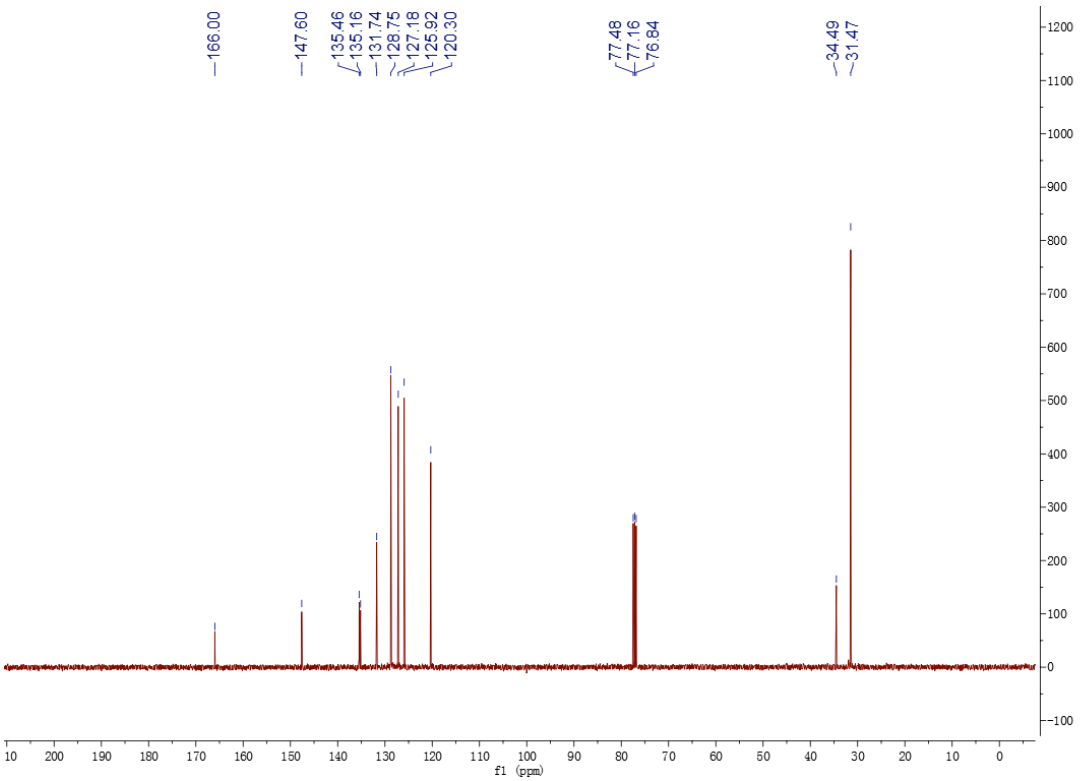
7.59
7.57
7.52
7.52
7.51
7.51
7.50
7.49
7.49
7.44
7.42
7.40
7.38
7.37
7.37
7.36
7.35
7.35
1.33
0.93
2.05
2.04
1.14
2.13
2.05
9.00
f1 (ppm)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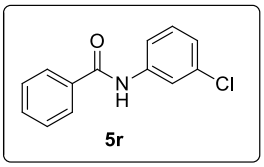
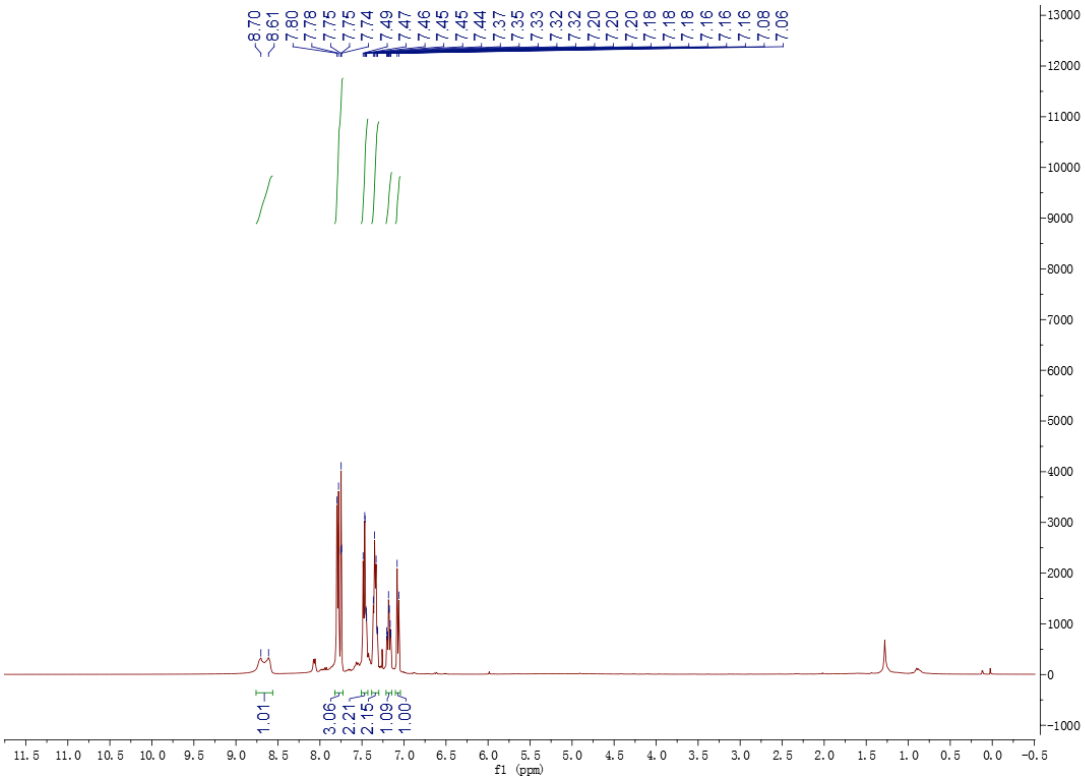
147.60
135.46
135.16
131.74
128.75
127.18
125.92
120.30
77.48
77.16
76.84
34.49
31.47
f1 (ppm)


O
N
H
Cl

5r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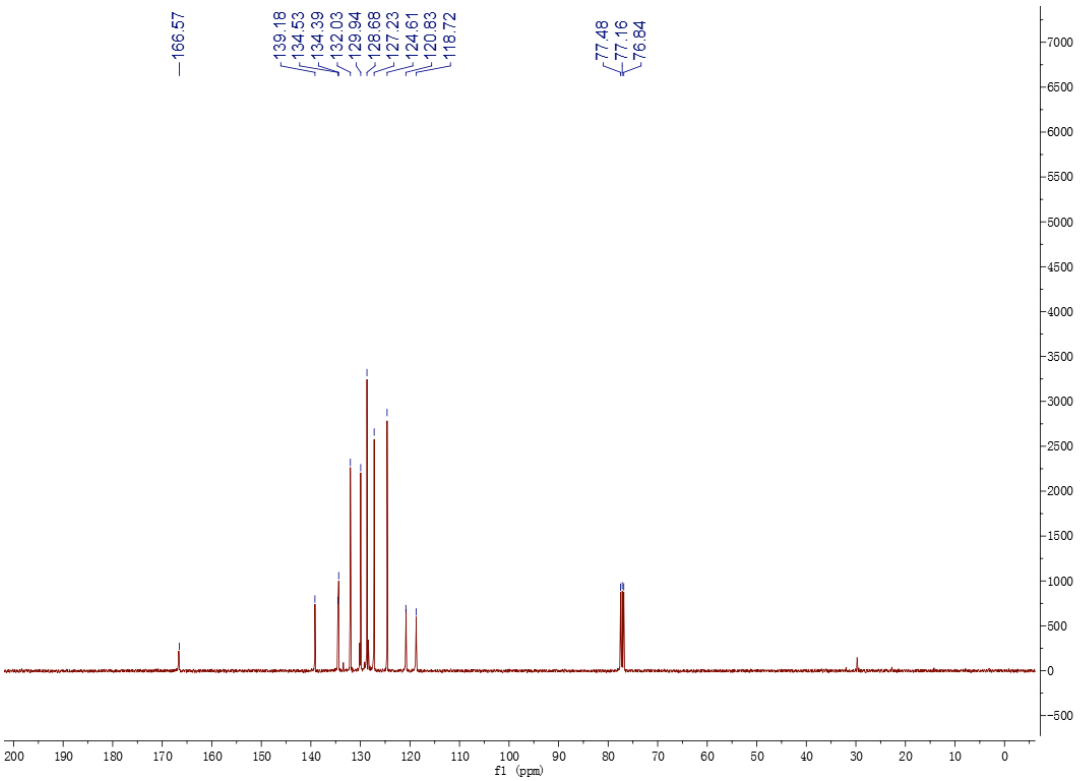
8.61
7.80
7.78
7.75
7.75
7.74
7.49
7.47
7.46
7.45
7.45
7.44
7.37
7.35
7.33
7.32
7.32
7.20
7.20
7.20
7.18
7.18
7.18
7.16
7.16
7.16
7.08
7.06
1.01
3.06
2.21
2.15
1.09
1.00
f1 (ppm)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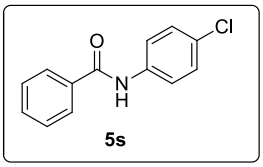
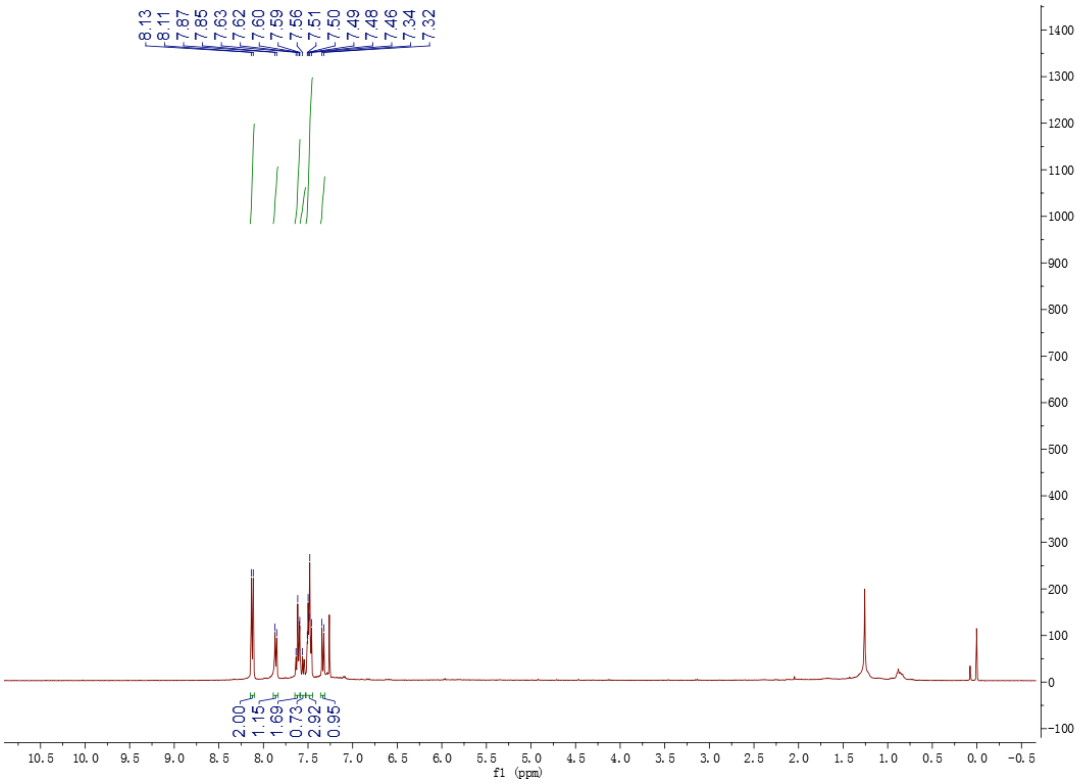
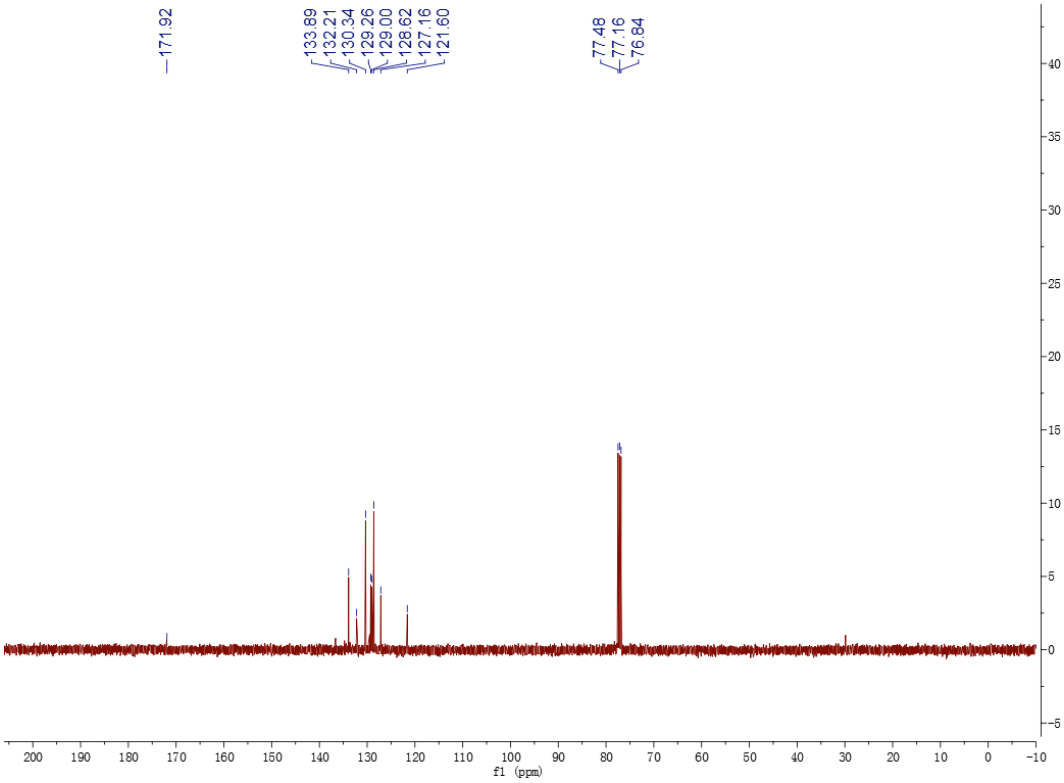
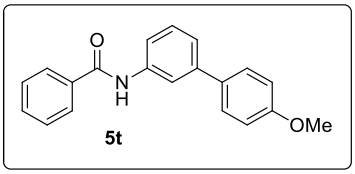
139.18
134.53
134.39
132.03
129.94
128.68
127.23
124.61
120.83
118.72
77.48
77.16
76.84
f1 (ppm)

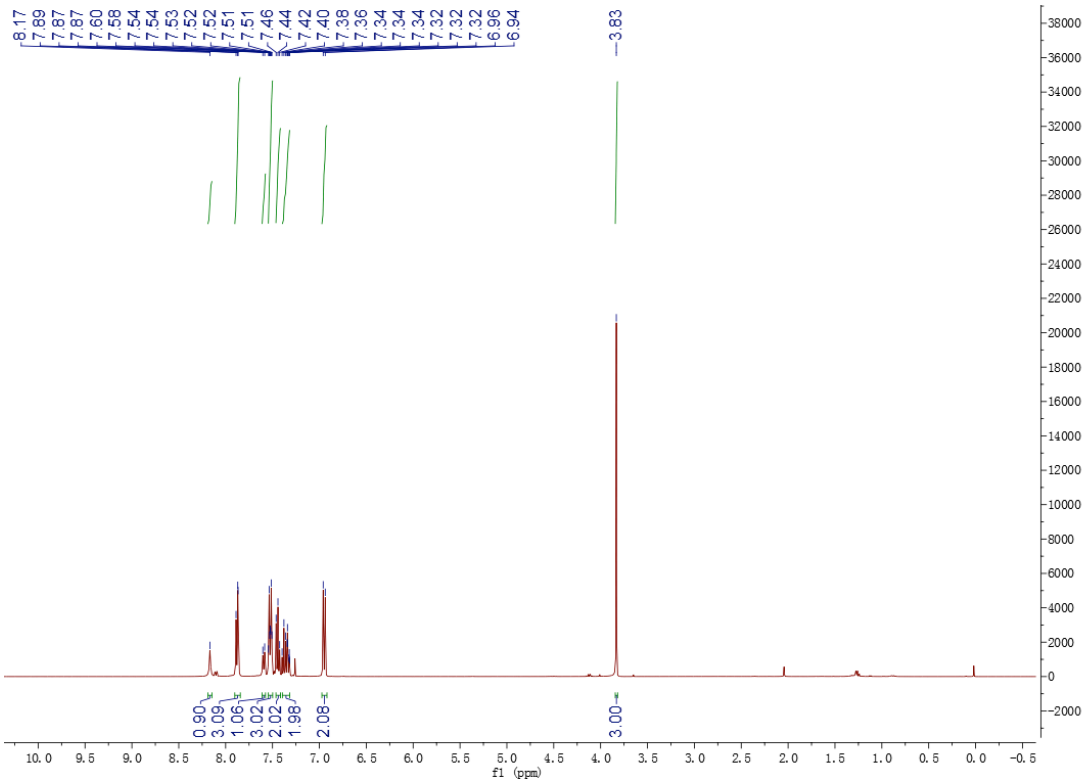

O
N
H
Cl

5s


8.13
8.11
7.87
7.85
7.63
7.62
7.60
7.59
7.56
7.51
7.50
7.49
7.48
7.46
7.34
7.32
2.00
1.15
1.69
0.73
2.92
0.95
f1 (ppm)


171.92
133.89
132.21
130.34
129.26
129.00
128.62
127.16
121.60
77.48
77.16
76.84
f1 (ppm)


O
N
H
OMe
5t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7.89
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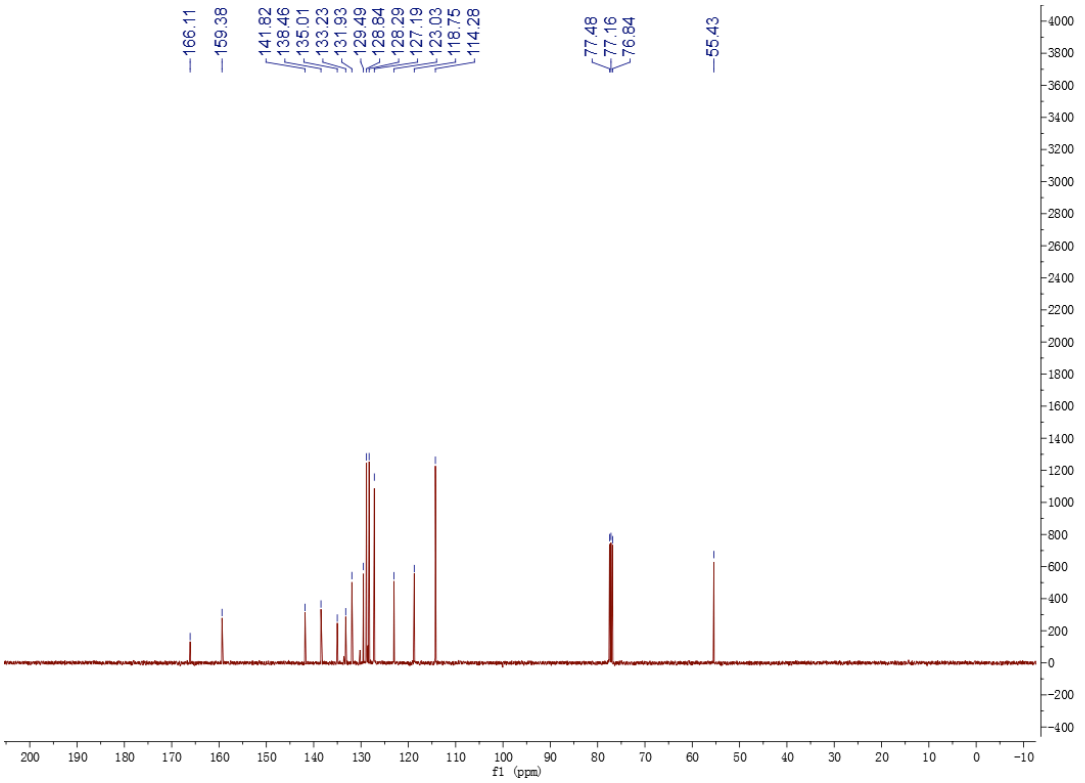
7.87
7.60
7.58
7.54
7.54
7.53
7.52
7.52
7.51
7.51
7.46
7.44
7.42
7.40
7.38
7.36
7.34
7.34
7.34
7.32
7.32
6.96
6.94
3.83
0.90
3.09
1.06
3.02
2.02
1.98
2.08
3.00
f1 (ppm)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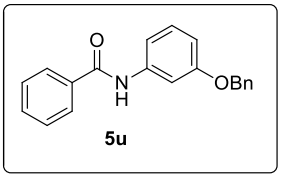
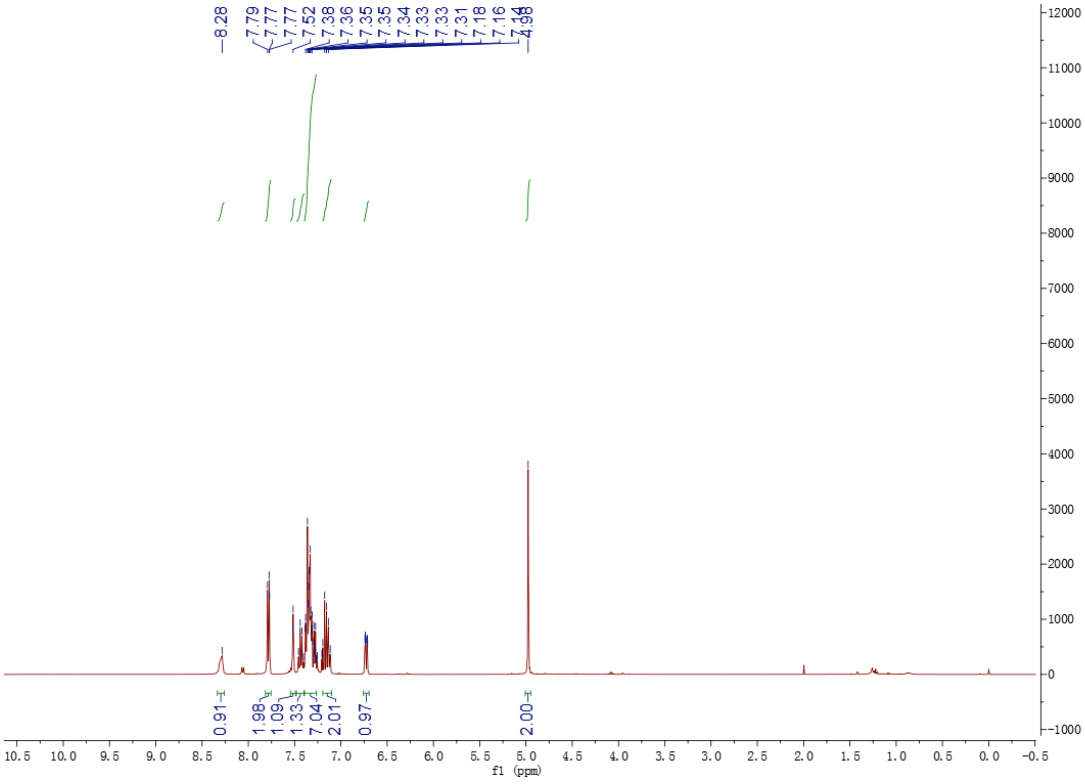
141.82
138.46
135.01
133.23
131.93
129.49
128.84
128.29
127.19
123.03
118.75
114.28
77.48
77.16
76.84
55.43
f1 (ppm)


O
N
H
OBn

5u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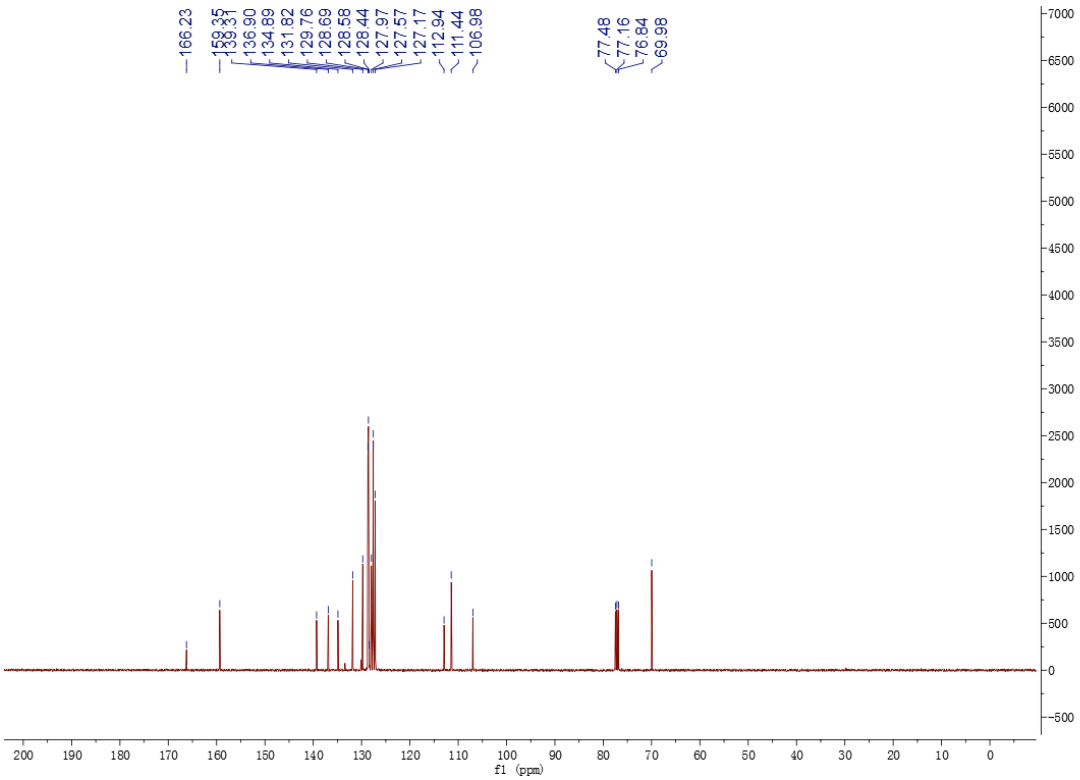
7.77
7.77
7.52
7.38
7.36
7.35
7.35
7.34
7.33
7.33
7.31
7.18
7.16
0.91
1.98
1.09
1.33
7.04
2.01
0.97
2.00
f1 (ppm)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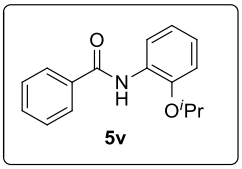
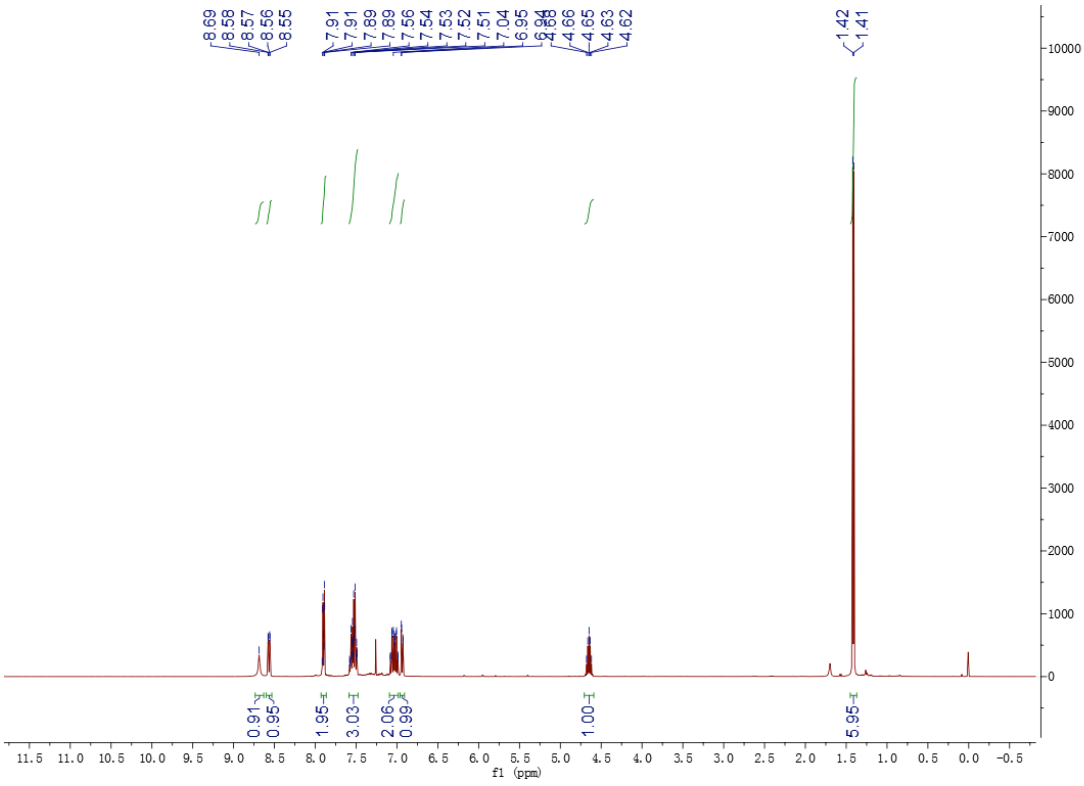
159.35
139.31
136.90
134.89
131.82
129.76
128.69
128.58
128.44
127.97
127.57
127.17
112.94
111.44
106.98
77.48
77.16
76.84
69.98
f1 (ppm)


O
N
H
O^{i}Pr

5v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8.56
8.55
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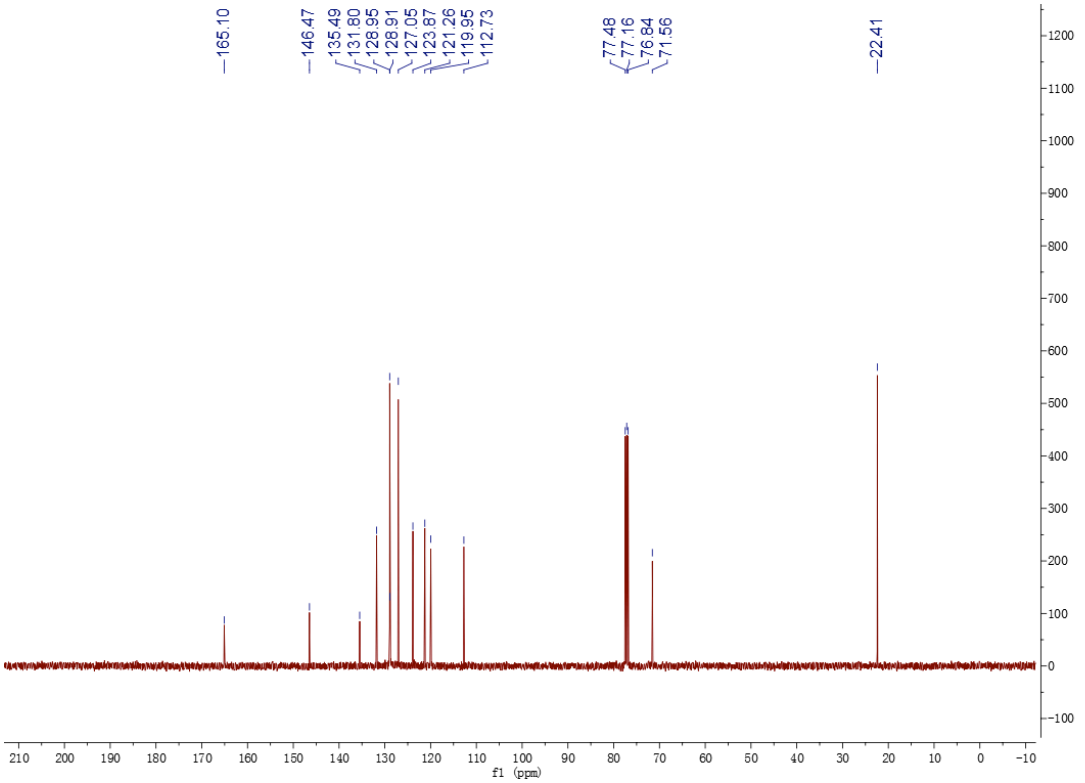
7.91
7.89
7.89
7.56
7.54
7.53
7.52
7.51
7.04
6.95
4.66
4.65
4.63
4.62
1.42
1.41
0.91
0.95
1.95
3.03
2.06
0.99
1.00
5.95
f1 (ppm)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127.05
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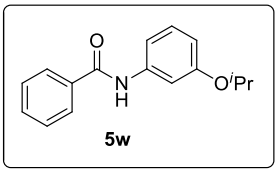
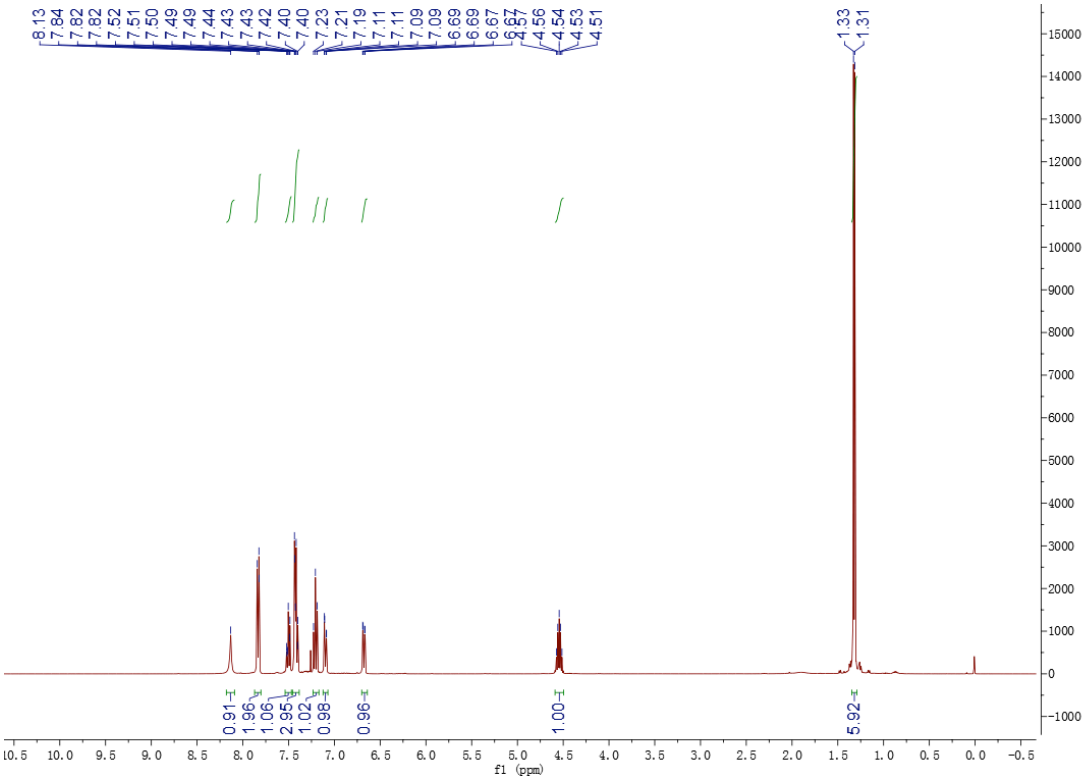
121.26
119.95
112.73
77.48
77.16
76.84
71.56
22.41
f1 (ppm)


O
N
H
O^{i}Pr

5w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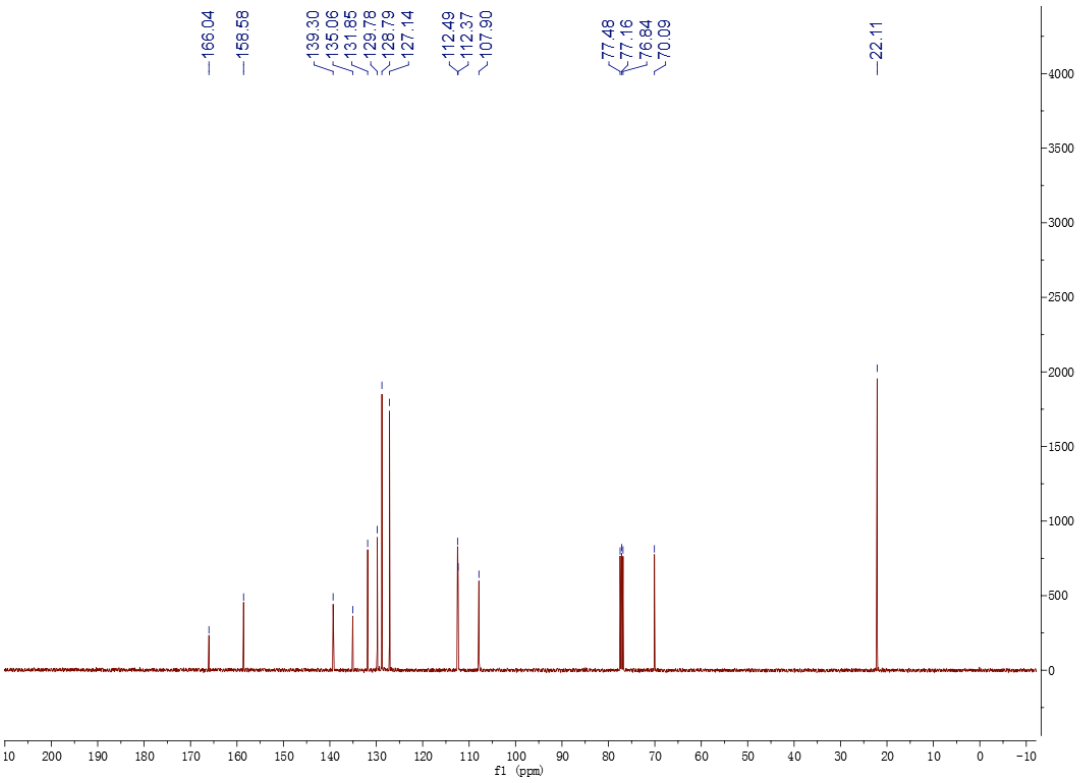
7.82
7.52
7.51
7.50
7.49
7.49
7.44
7.43
7.43
7.42
7.40
7.40
7.23
7.21
7.19
7.11
7.11
7.09
7.09
6.69
6.69
6.67
6.67
4.57
4.56
4.54
4.53
4.51
1.33
1.31
0.91
1.96
1.06
2.95
1.02
0.98
0.96
1.00
5.92
f1 (ppm)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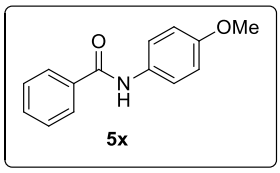
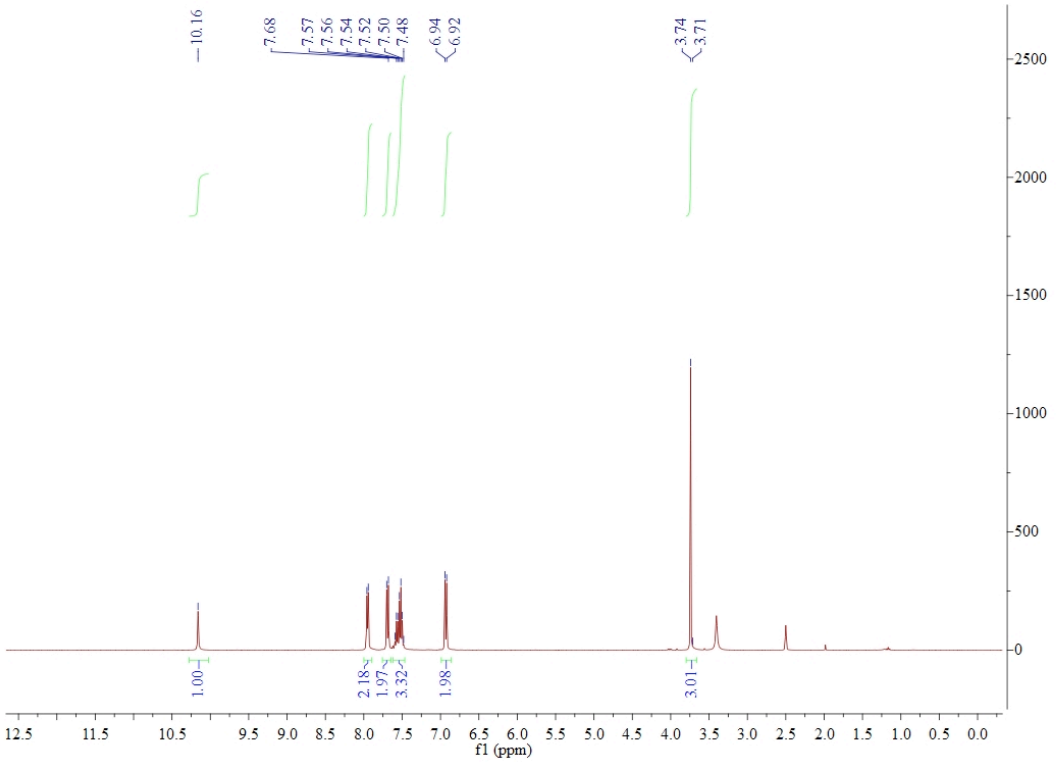
127.14
112.49
112.37
107.90
77.48
77.16
76.84
70.09
22.11
f1 (ppm)


O
N
H
OMe

5x


10.16
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7.56
7.54
7.52
7.50
7.48
6.94
6.92
3.74
3.71
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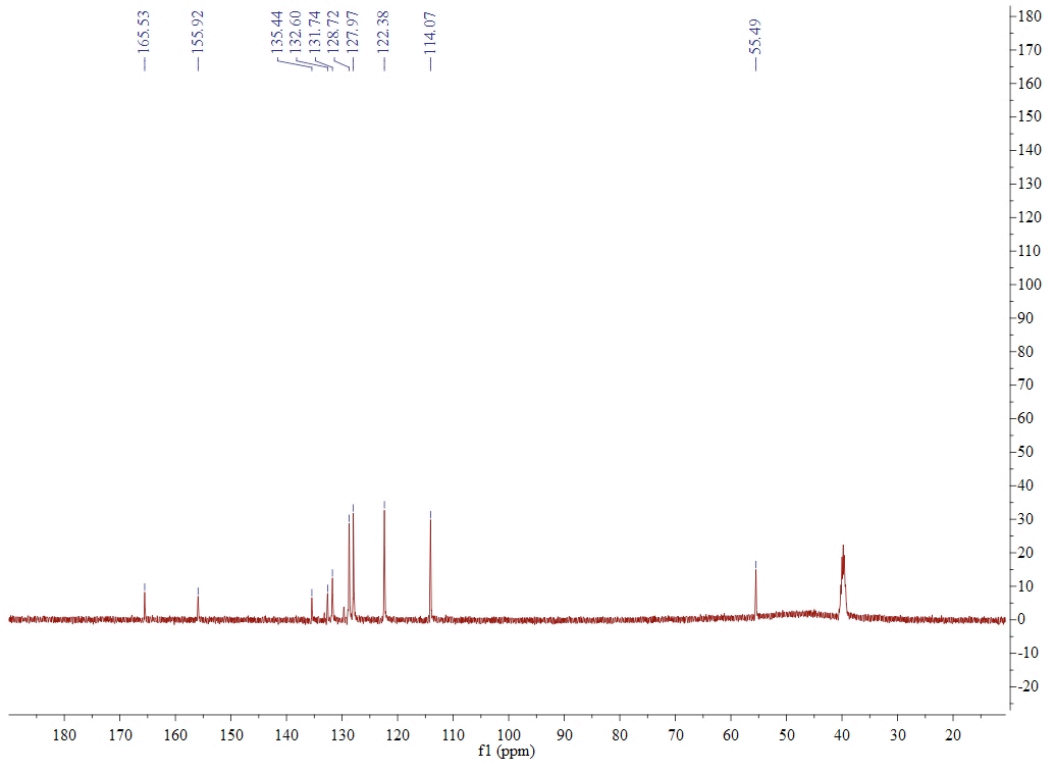
1.97
3.32
1.98
3.01
f1 (ppm)


165.53
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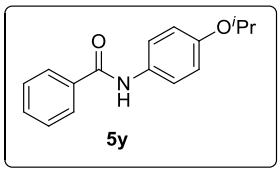
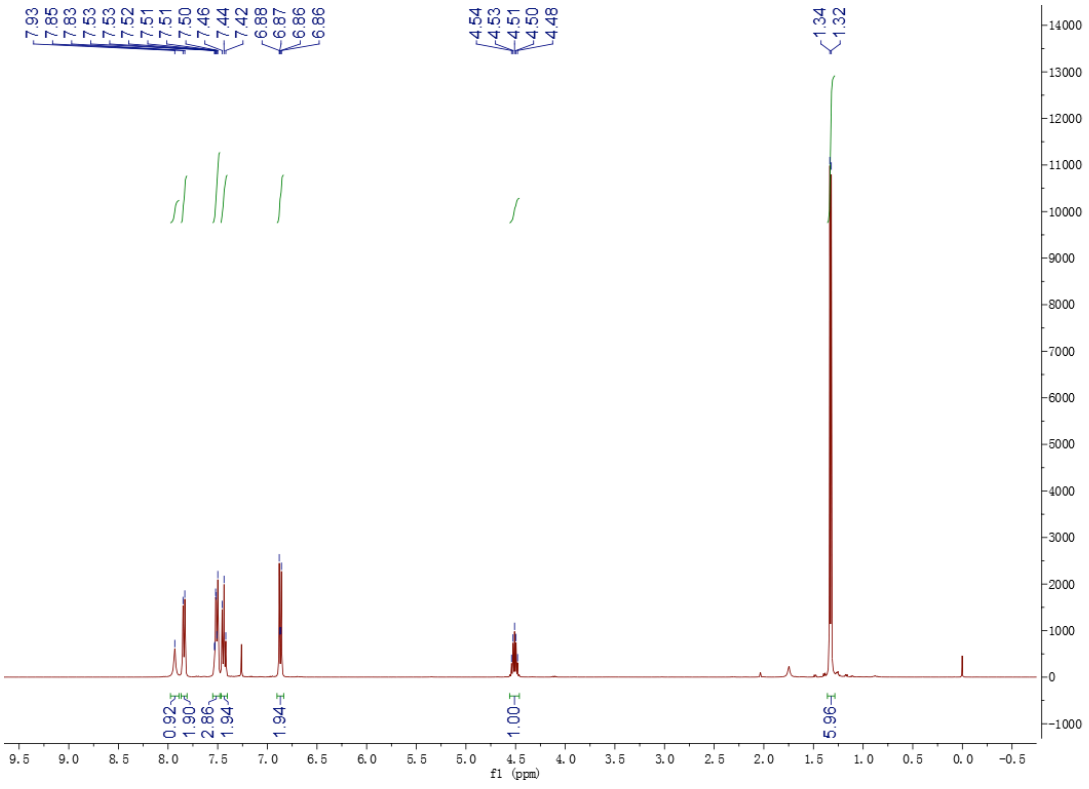
132.60
131.74
128.72
127.97
122.38
114.07
55.49
f1 (ppm)


O
N
H
O^{i}Pr

5y


7.93
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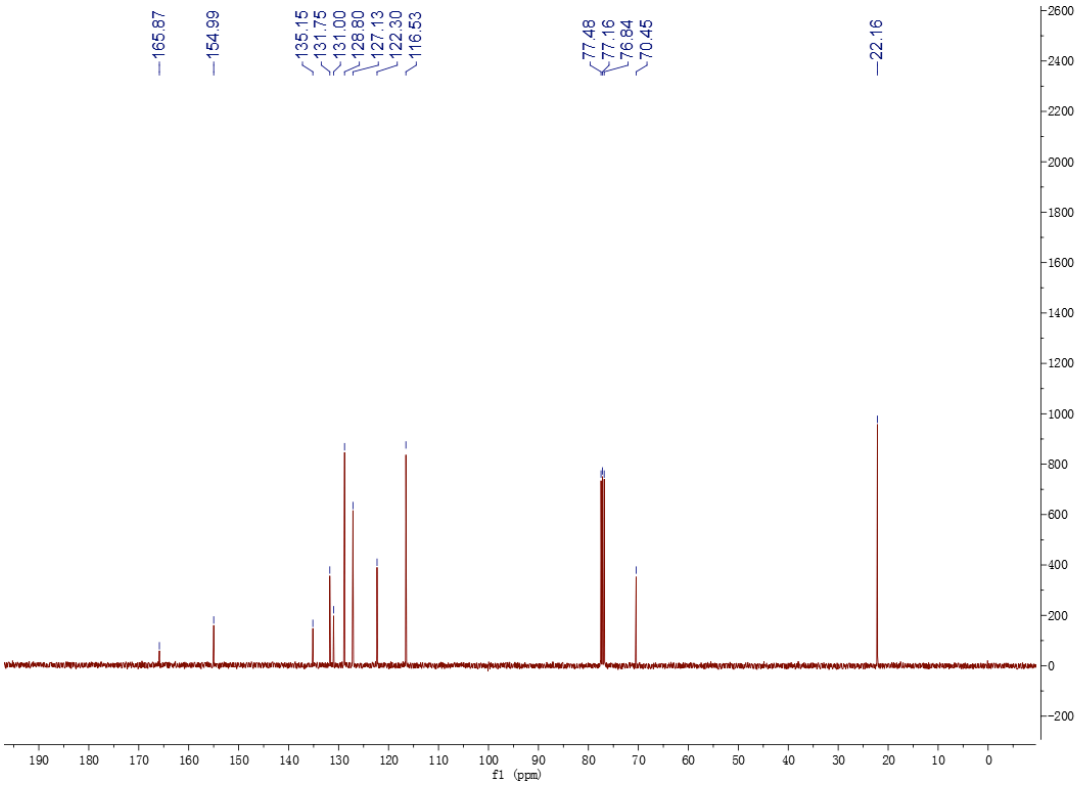
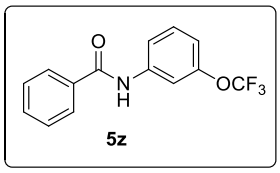
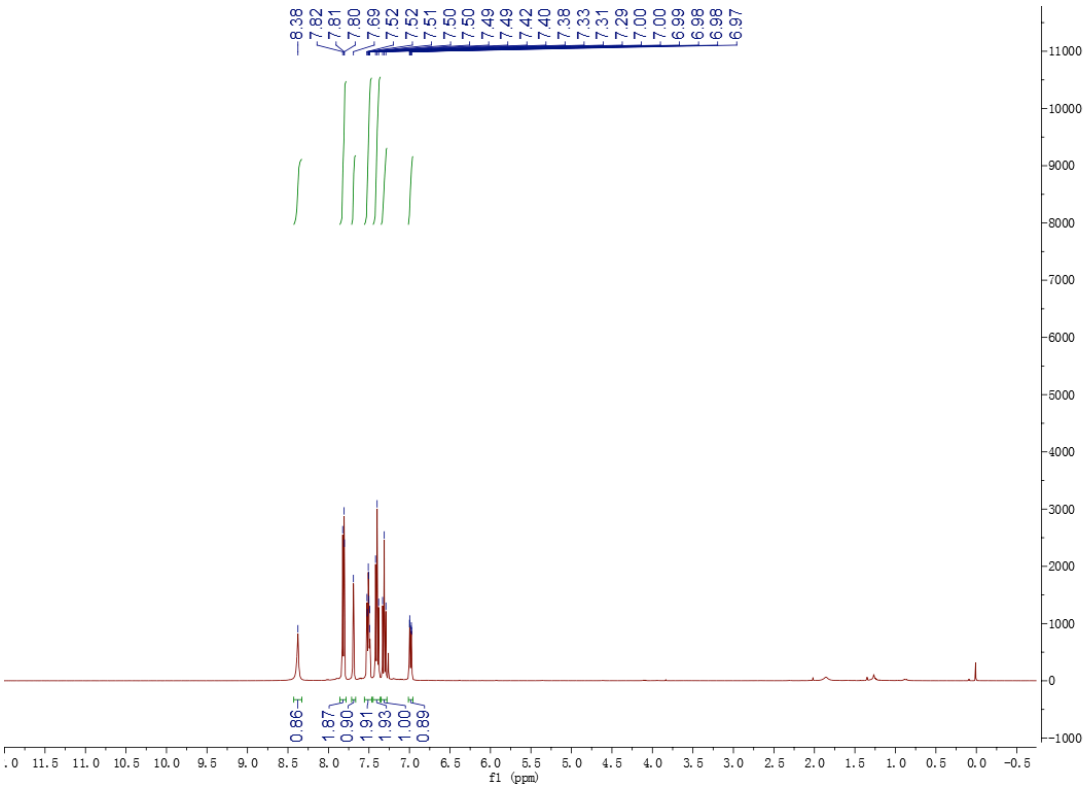
7.53
7.53
7.52
7.51
7.51
7.50
7.46
7.44
7.42
6.88
6.87
6.86
6.86
4.54
4.53
4.51
4.50
4.48
1.34
1.32
0.92
1.90
2.86
1.94
1.94
1.00
5.96
f1 (ppm)


165.87
154.99
135.15
131.75
131.00
128.80
127.13
122.30
116.53
77.48
77.16
76.84
70.45
22.16
f1 (ppm)


O
N
H
OCF3

5z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7.82
7.81
7.80
7.69
7.52
7.52
7.51
7.50
7.50
7.49
7.49
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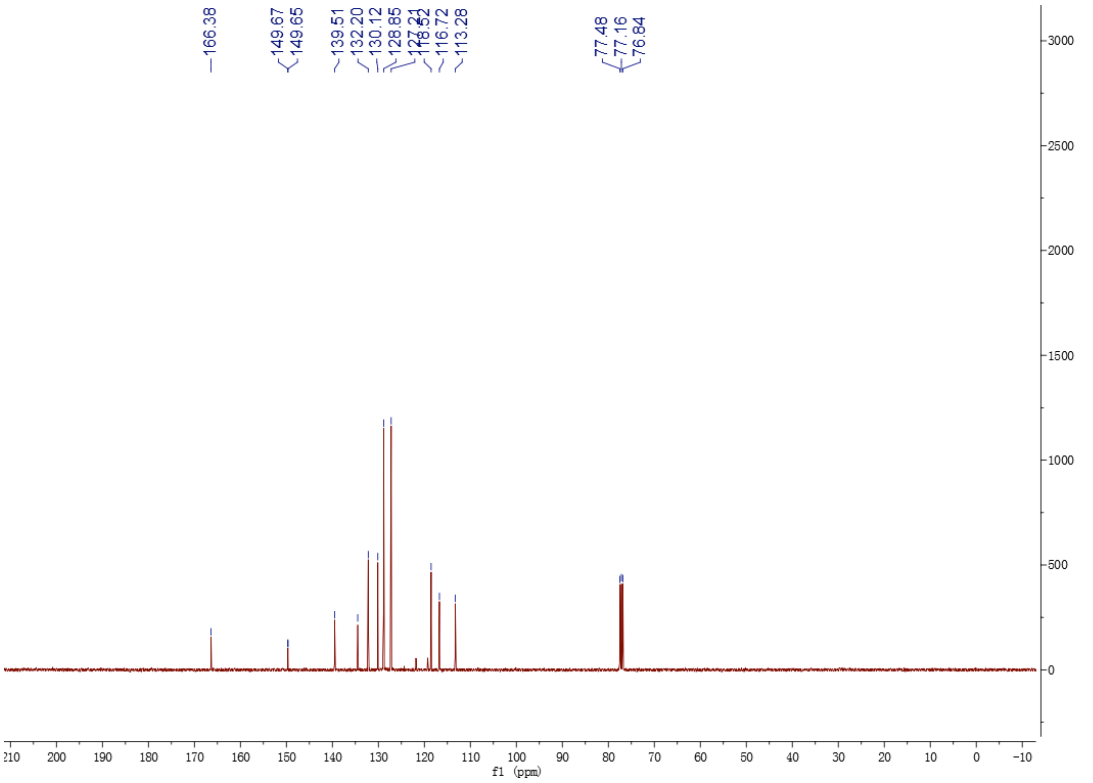
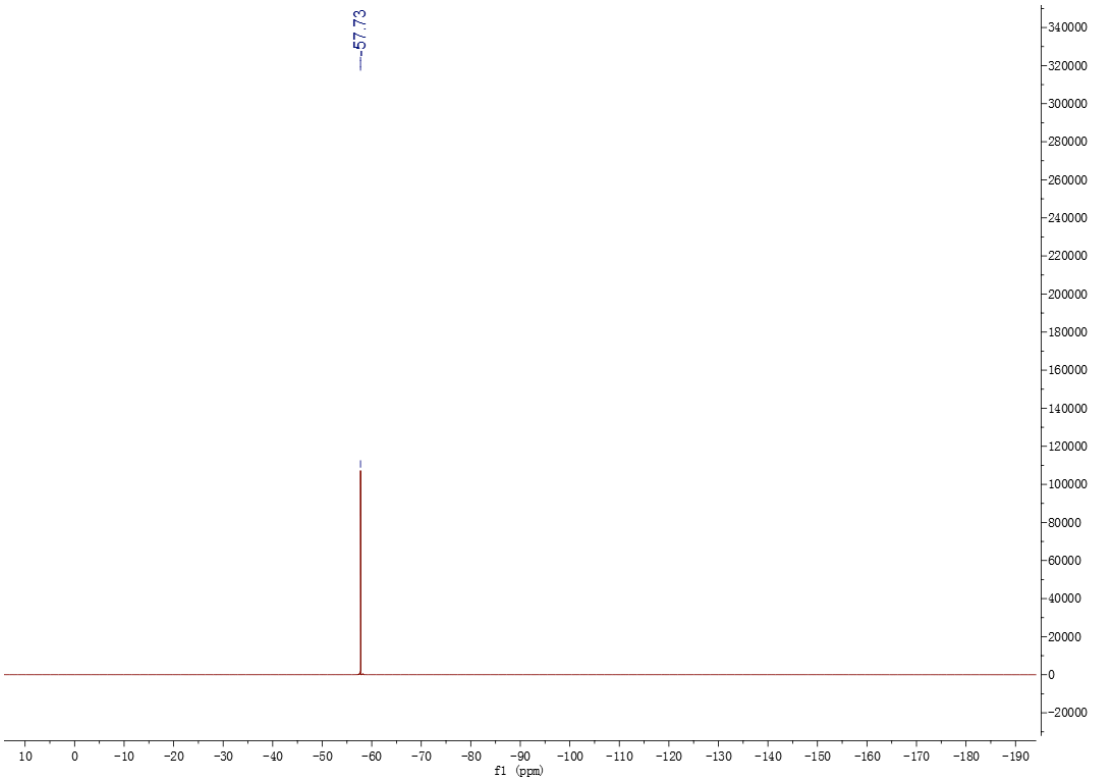
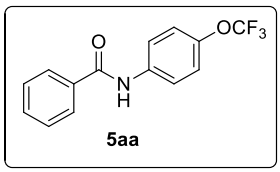
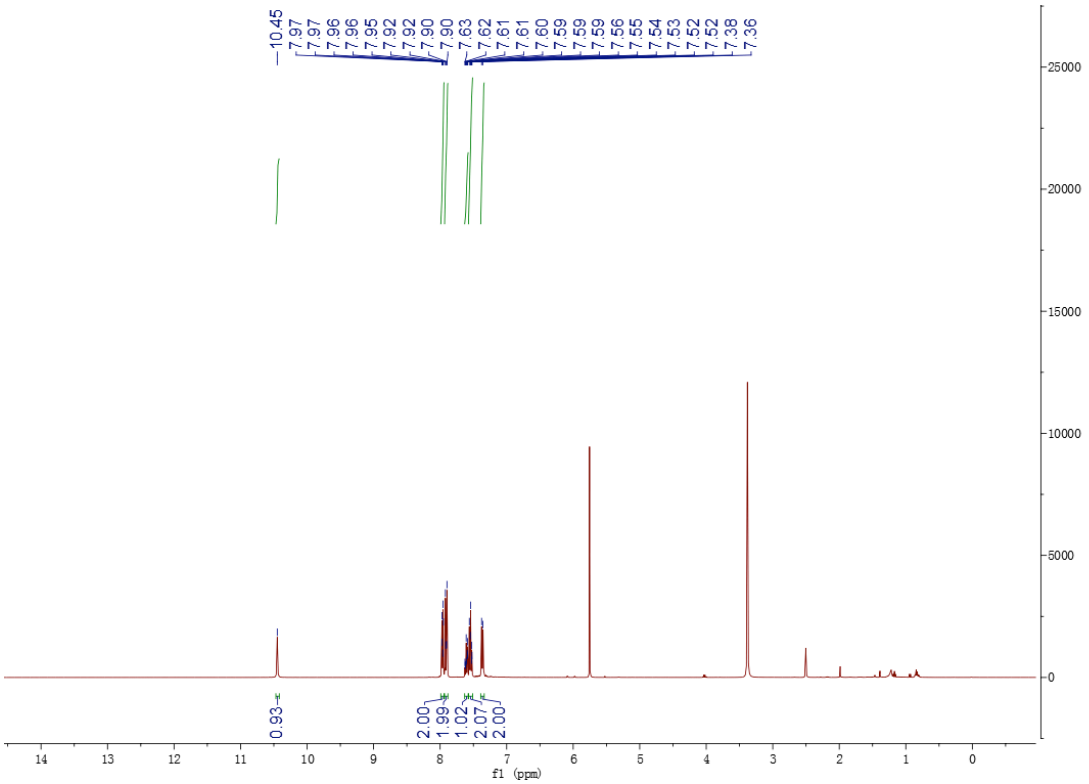
7.40
7.38
7.33
7.31
7.29
7.00
7.00
6.99
6.98
6.98
6.97
0.86
1.87
0.90
1.91
1.93
1.00
0.89
f1 (ppm)


166.38
149.67
149.65
139.51
132.20
130.12
128.85
127.21
118.52
116.72
113.28
77.48
77.16
76.84
f1 (ppm)


57.73
f1 (ppm)


O
N
H
OCF3

5aa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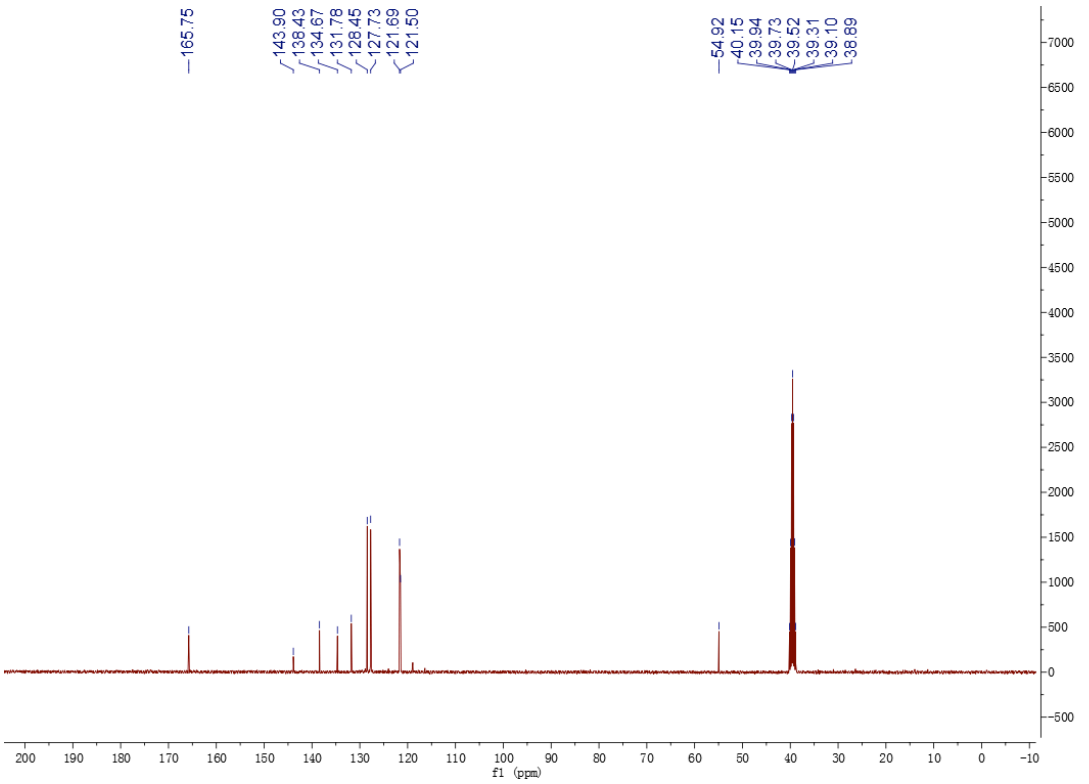
7.97
7.97
7.96
7.96
7.95
7.92
7.92
7.90
7.90
7.63
7.62
7.61
7.61
7.60
7.59
7.59
7.59
7.56
7.55
7.54
7.53
7.52
7.52
7.38
7.36
0.93
2.00
1.99
1.02
2.07
2.00
f1 (ppm)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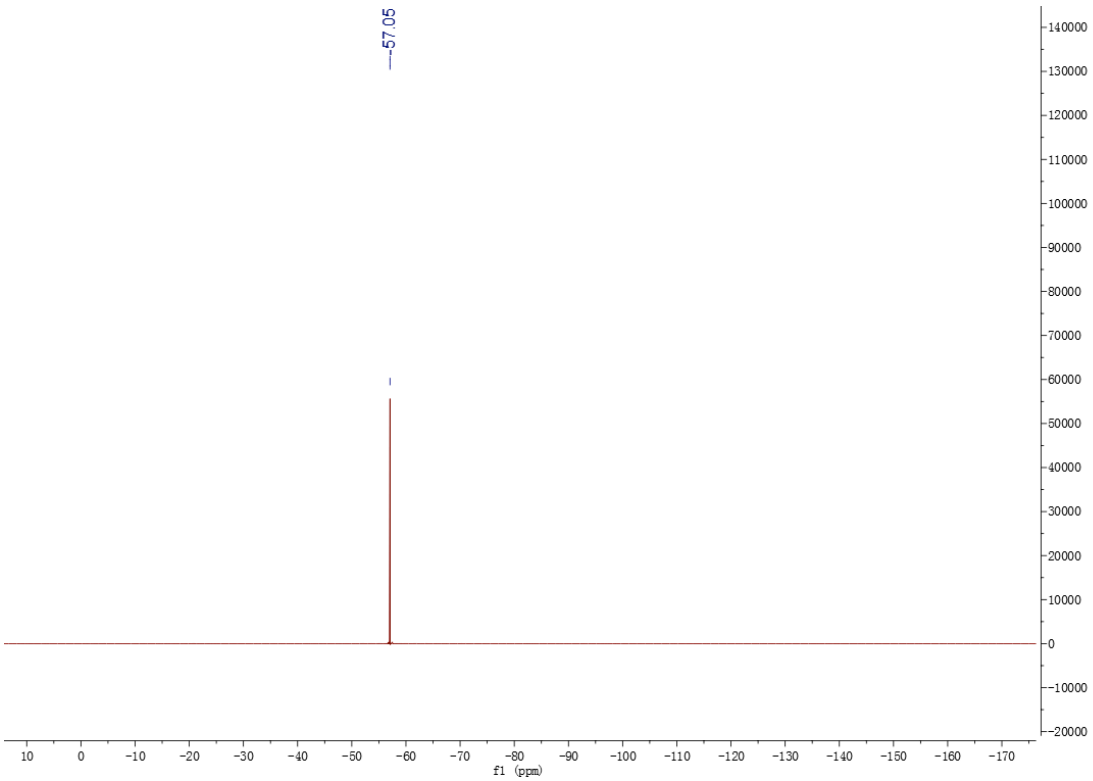
143.90
138.43
134.67
131.78
128.45
127.73
121.69
121.50
54.92
40.15
39.94
39.73
39.52
39.31
39.10
38.89
f1 (ppm)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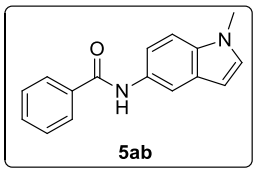
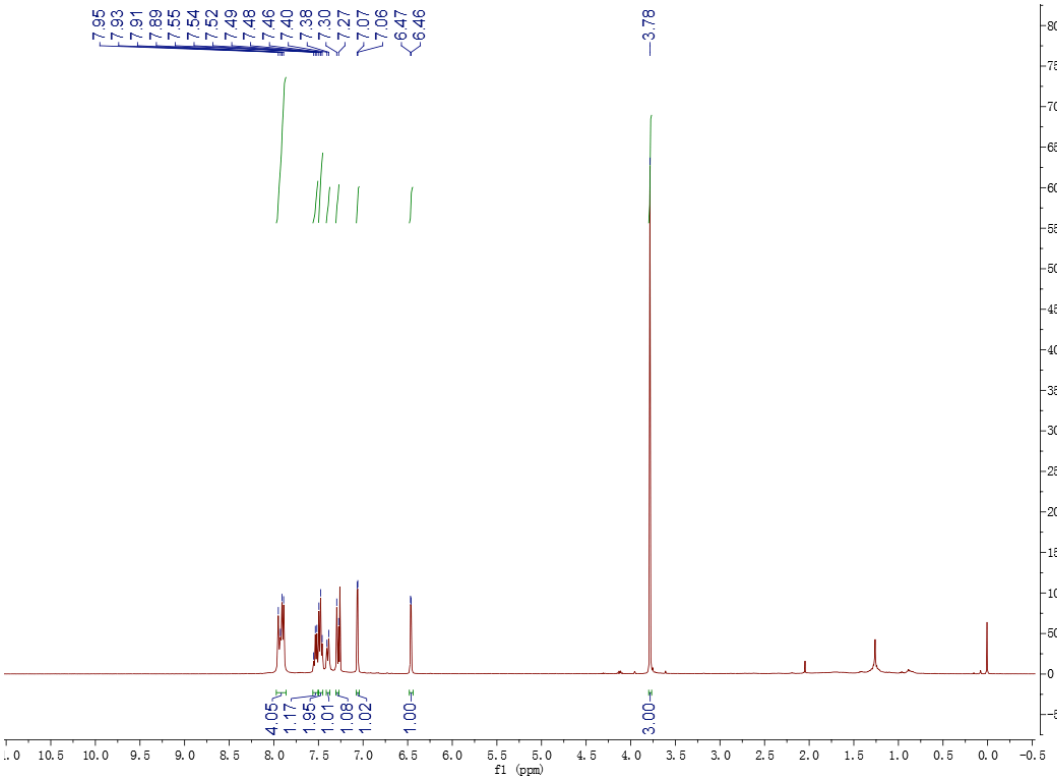
f1 (ppm)


O
N
H
N

5ab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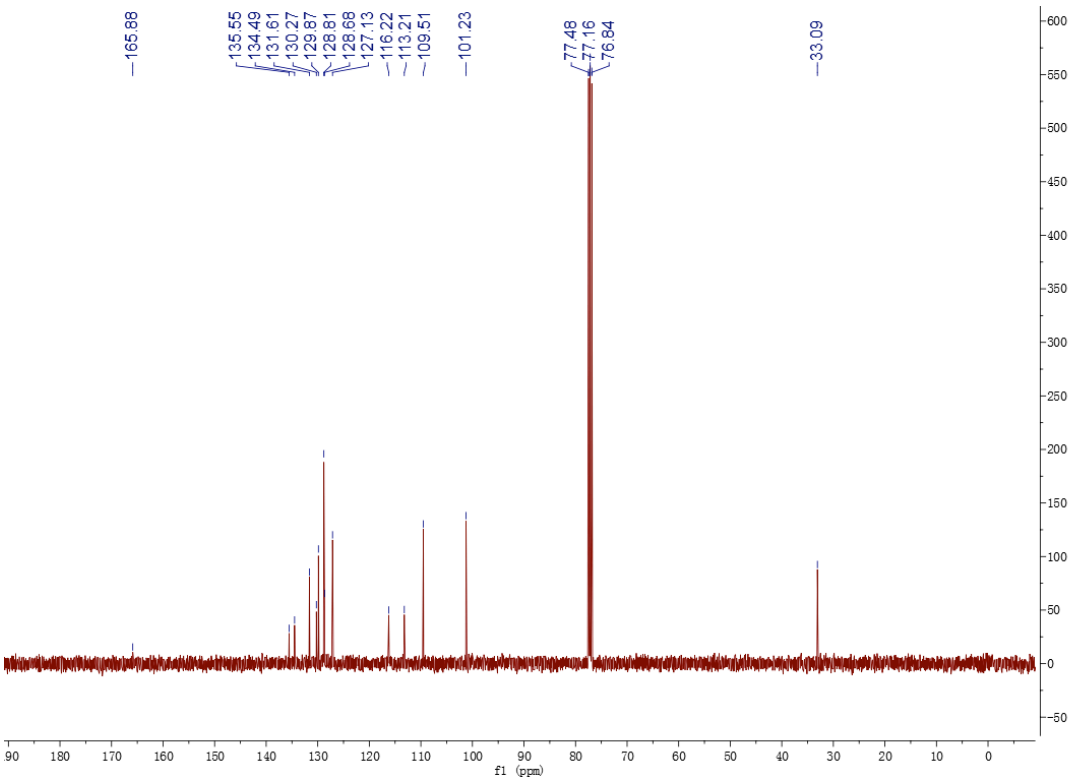
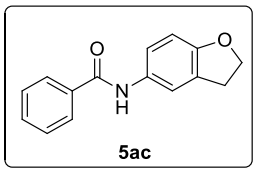
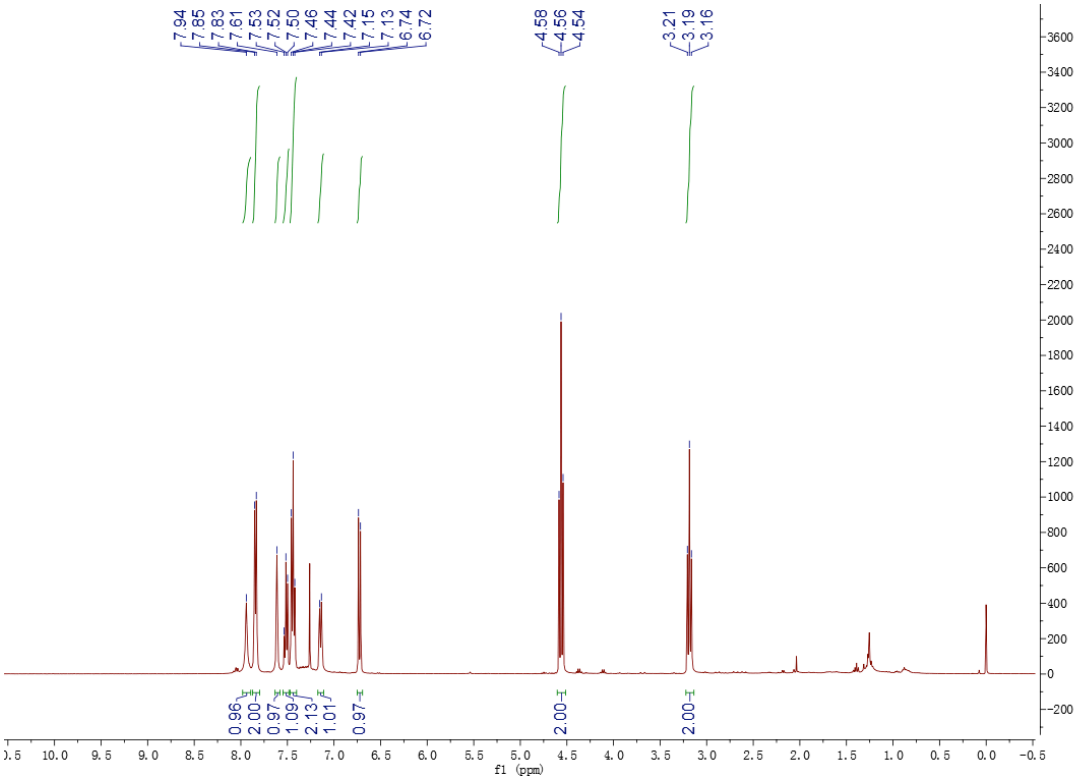
7.93
7.91
7.89
7.55
7.54
7.52
7.49
7.48
7.46
7.40
7.38
7.30
7.27
7.07
7.06
6.47
6.46
3.78
4.05
1.17
1.95
1.01
1.08
1.02
1.00
3.00
f1 (ppm)


165.88
135.55
134.49
131.61
130.27
129.87
128.81
128.68
127.13
116.22
113.21
109.51
101.23
77.48
77.16
76.84
33.09
f1 (ppm)


O
N
H
O

5ac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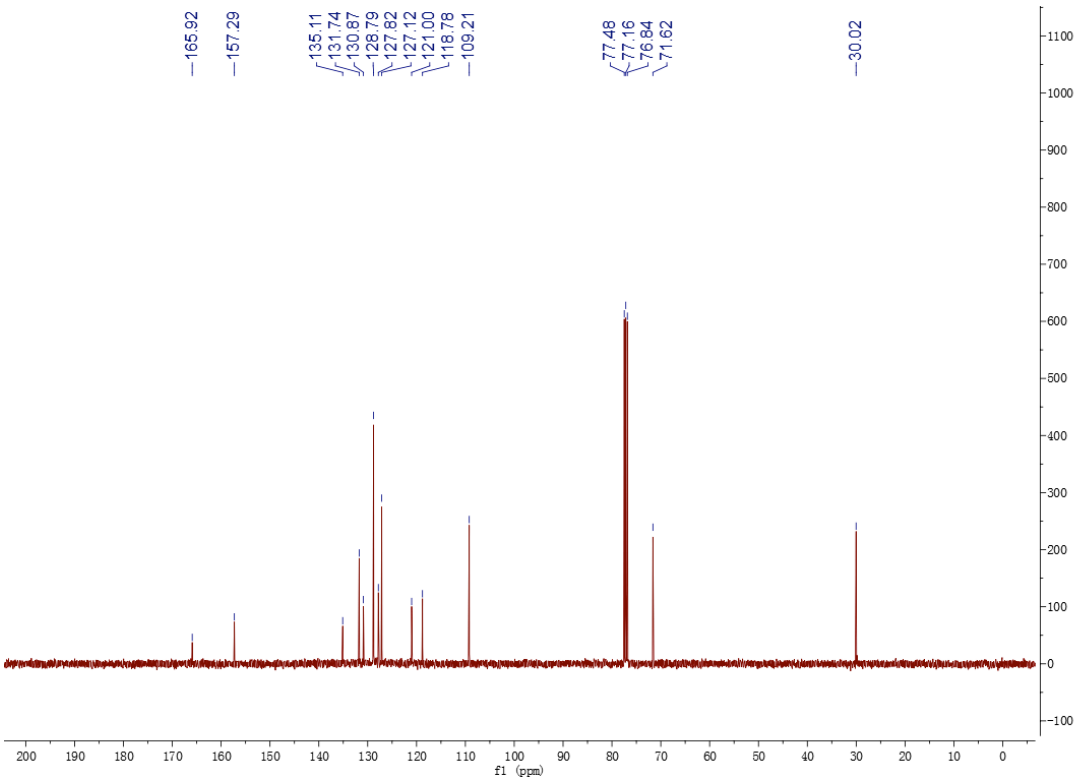
7.85
7.83
7.61
7.53
7.52
7.50
7.46
7.44
7.42
7.15
7.13
6.74
6.72
4.58
4.56
4.54
3.21
3.19
3.16
0.96
2.00
0.97
1.09
2.13
1.01
0.97
2.00
2.00
f1 (ppm)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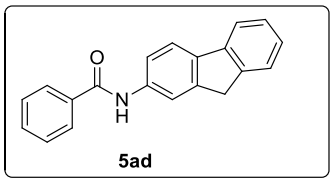
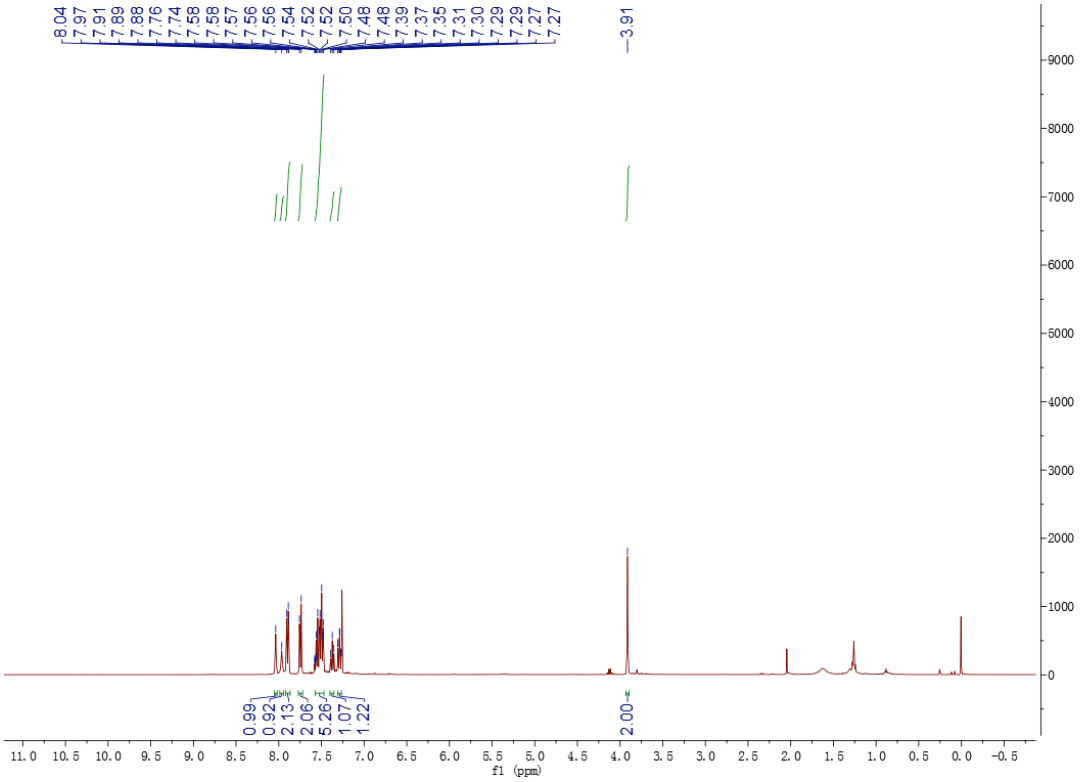
157.29
135.11
131.74
130.87
128.79
127.82
127.12
121.00
118.78
109.21
77.48
77.16
76.84
71.62
30.02
f1 (ppm)


O
N
H

5ad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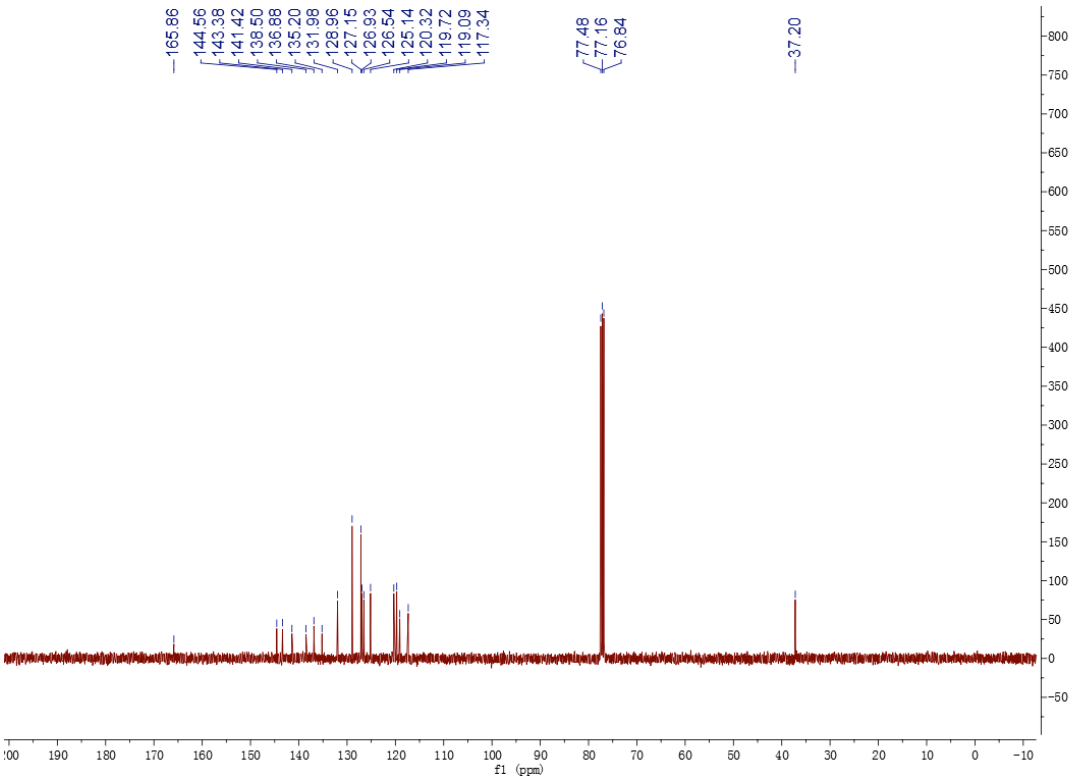
7.89
7.88
7.76
7.74
7.58
7.58
7.57
7.56
7.56
7.54
7.52
7.52
7.50
7.48
7.48
7.39
7.37
7.35
7.31
7.30
7.29
7.29
7.27
7.27
3.91
0.99
0.92
2.13
2.06
5.26
1.07
1.22
2.00
f1 (ppm)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141.42
138.50
136.88
135.20
131.98
128.96
127.15
126.93
126.54
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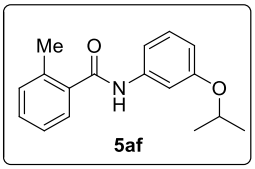
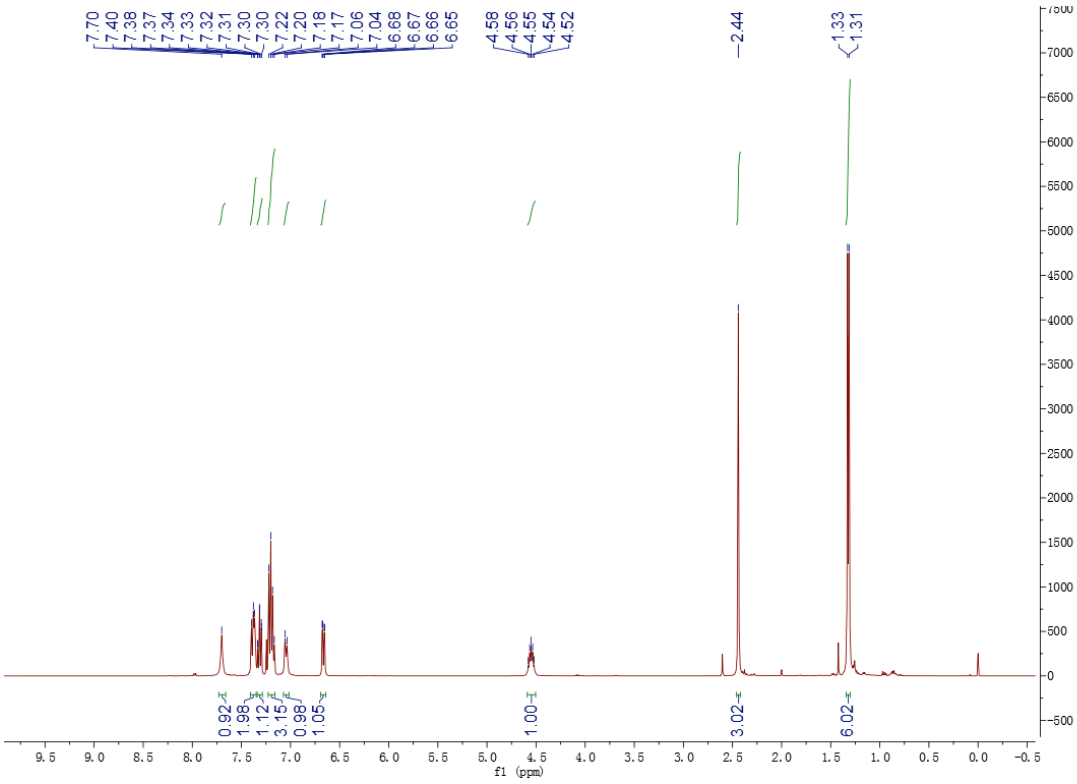
77.48
77.16
76.84
37.20
f1 (ppm)


Me
O
N
H
O

5af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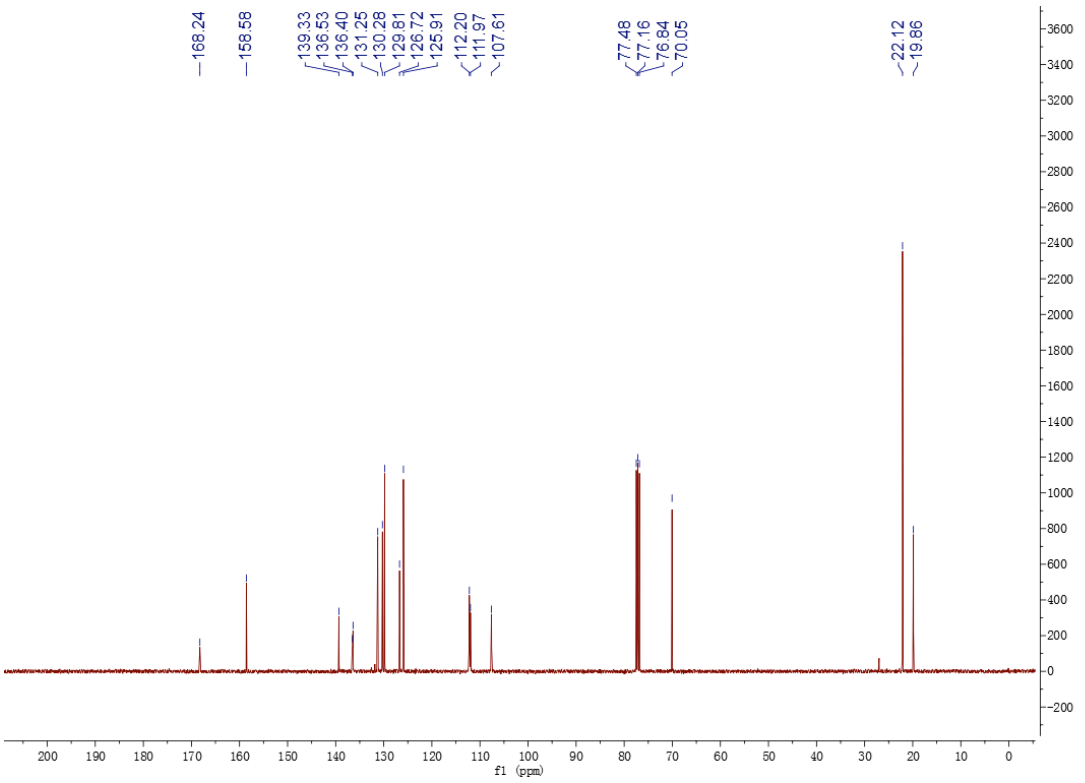
7.38
7.37
7.34
7.33
7.32
7.31
7.30
7.30
7.22
7.20
7.18
7.17
7.06
7.04
6.68
6.67
6.66
6.65
4.58
4.56
4.55
4.54
4.52
2.44
1.33
1.31
0.92
1.98
1.12
3.15
0.98
1.05
1.00
3.02
6.02
f1 (ppm)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158.58
139.33
136.53
136.40
131.25
130.28
129.81
126.72
125.91
112.20
111.97
107.61
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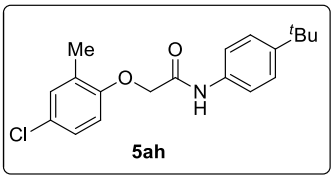
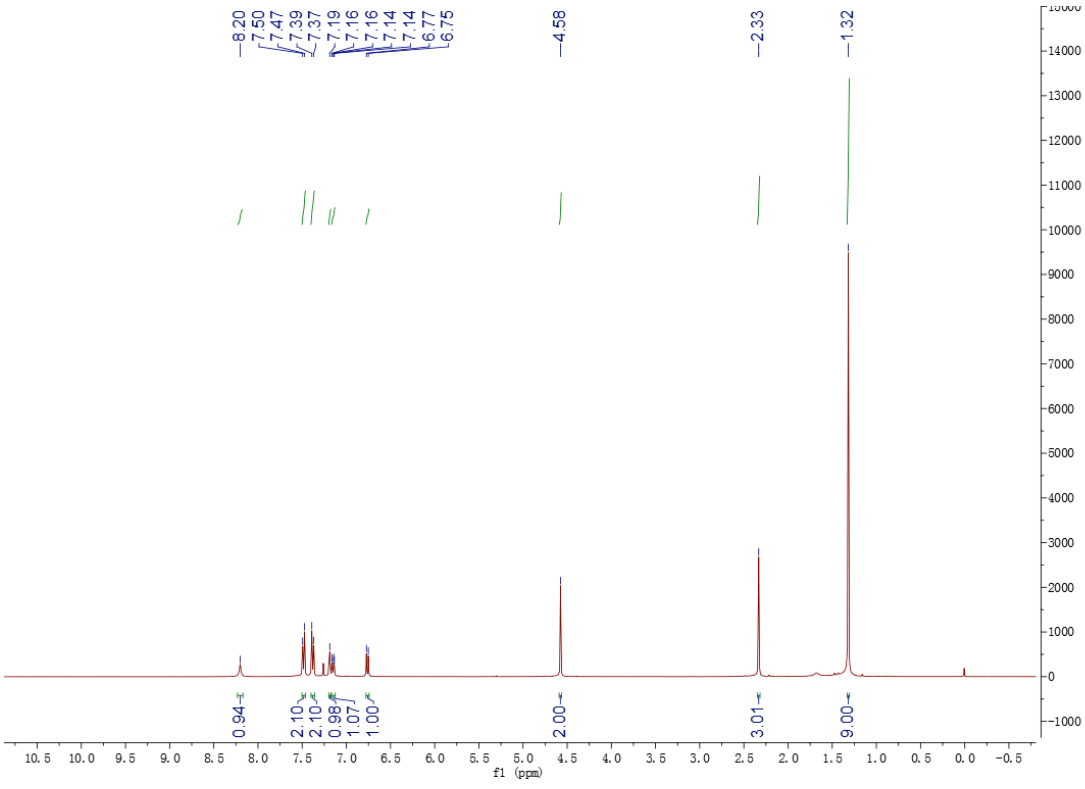
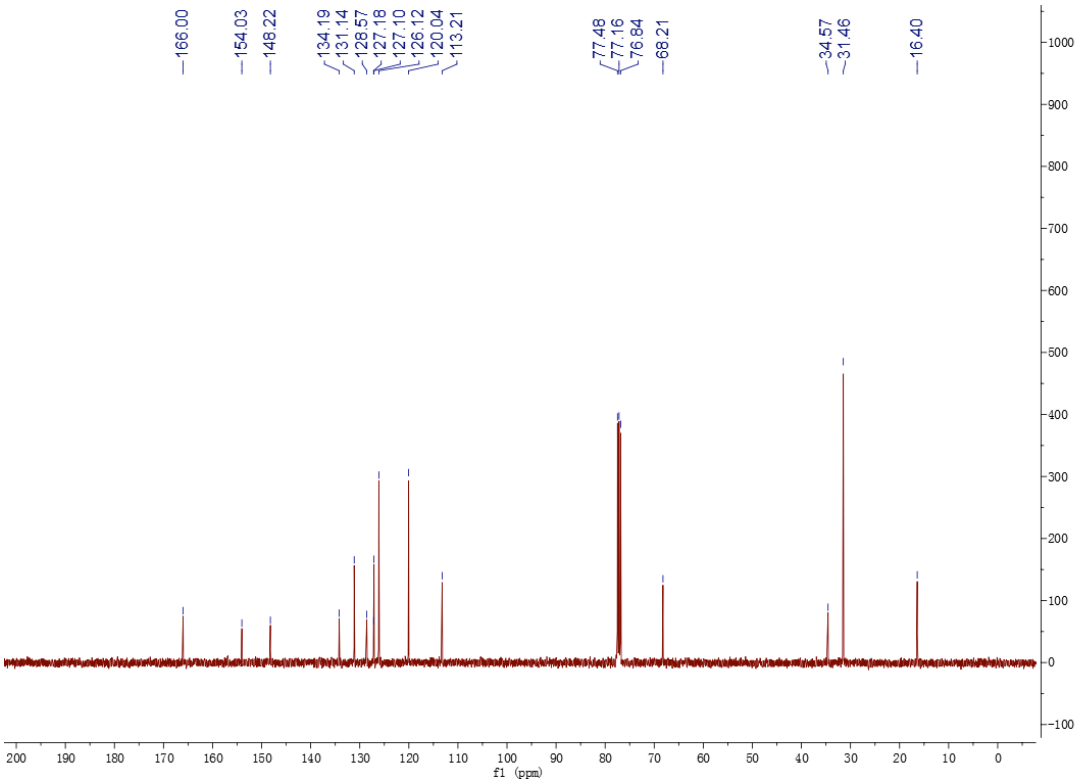
77.16
76.84
70.05
22.12
19.86
f1 (ppm)


Me
O
O
N
H
tBu
Cl

5ah


8.20
7.50
7.47
7.39
7.37
7.19
7.16
7.16
7.14
7.14
6.77
6.75
4.58
2.33
1.32
0.94
2.10
2.10
0.98
1.07
1.00
2.00
3.01
9.00
f1 (ppm)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154.03
148.22
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131.14
128.57
127.18
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120.04
113.21
77.48
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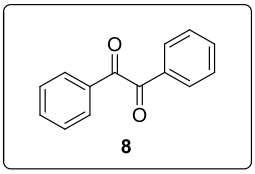
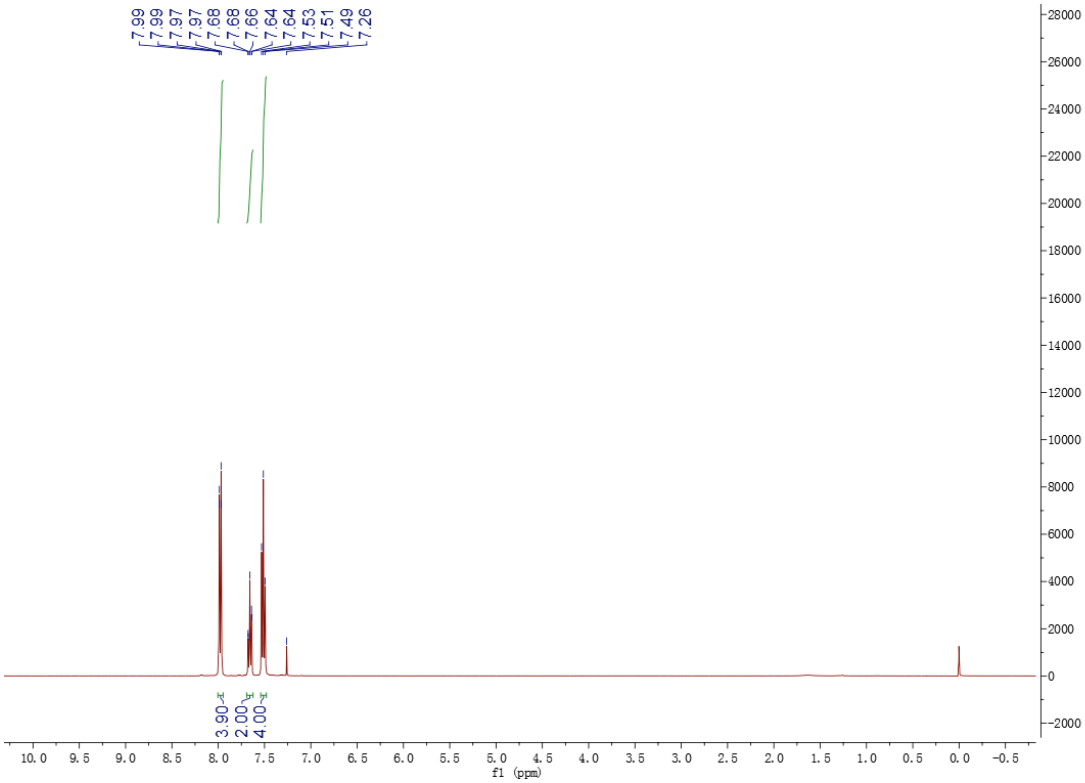
68.21
34.57
31.46
16.40
f1 (ppm)


O
O

8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7.97
7.97
7.68
7.66
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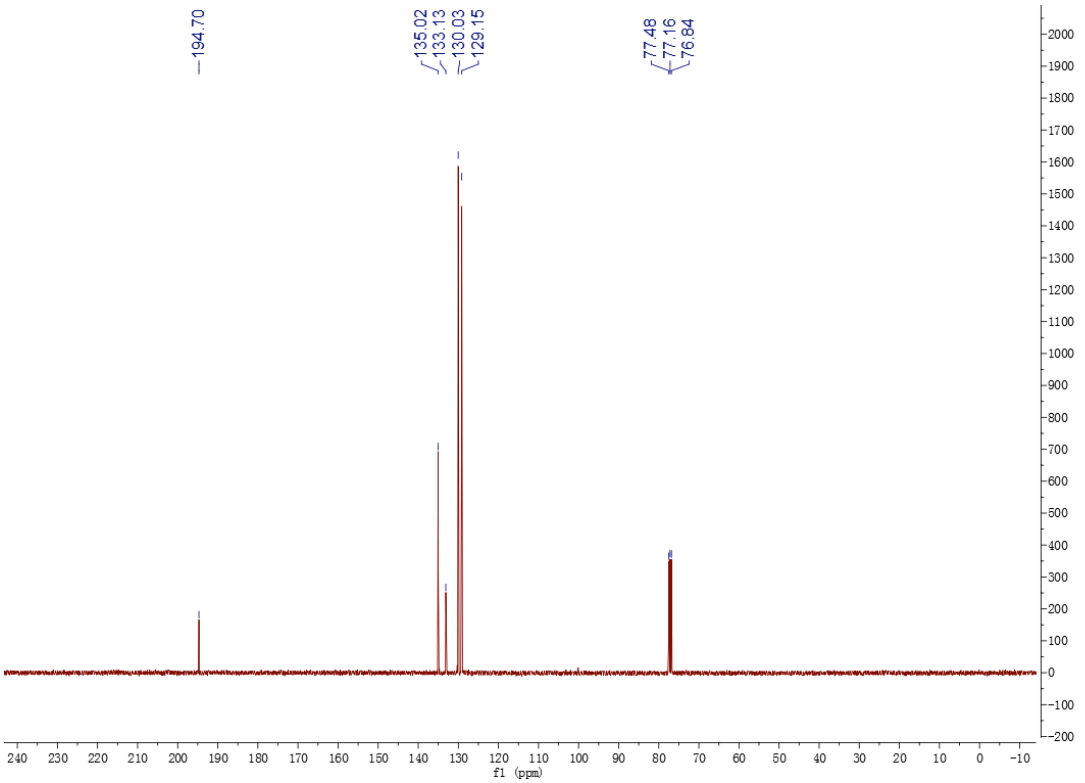
7.64
7.53
7.51
7.49
7.26
3.90
2.00
4.00
f1 (ppm)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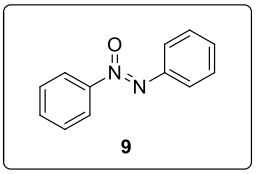
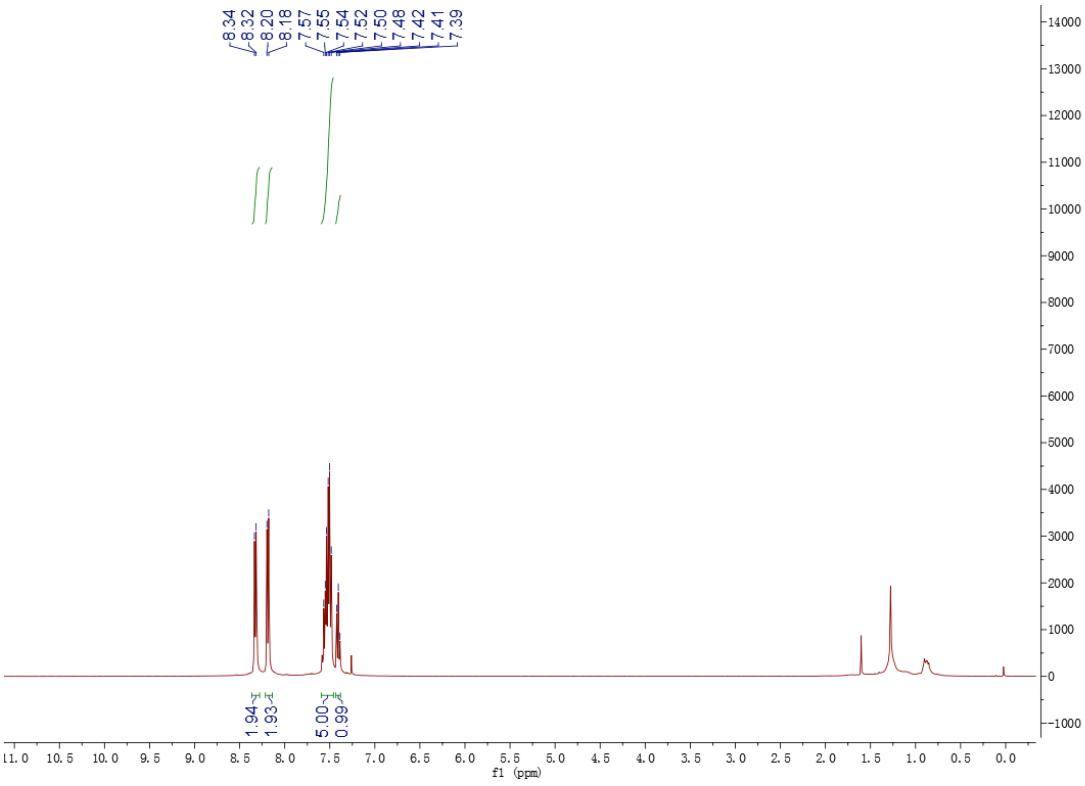
129.15
77.48
77.16
76.84
f1 (ppm)


O
N
N

9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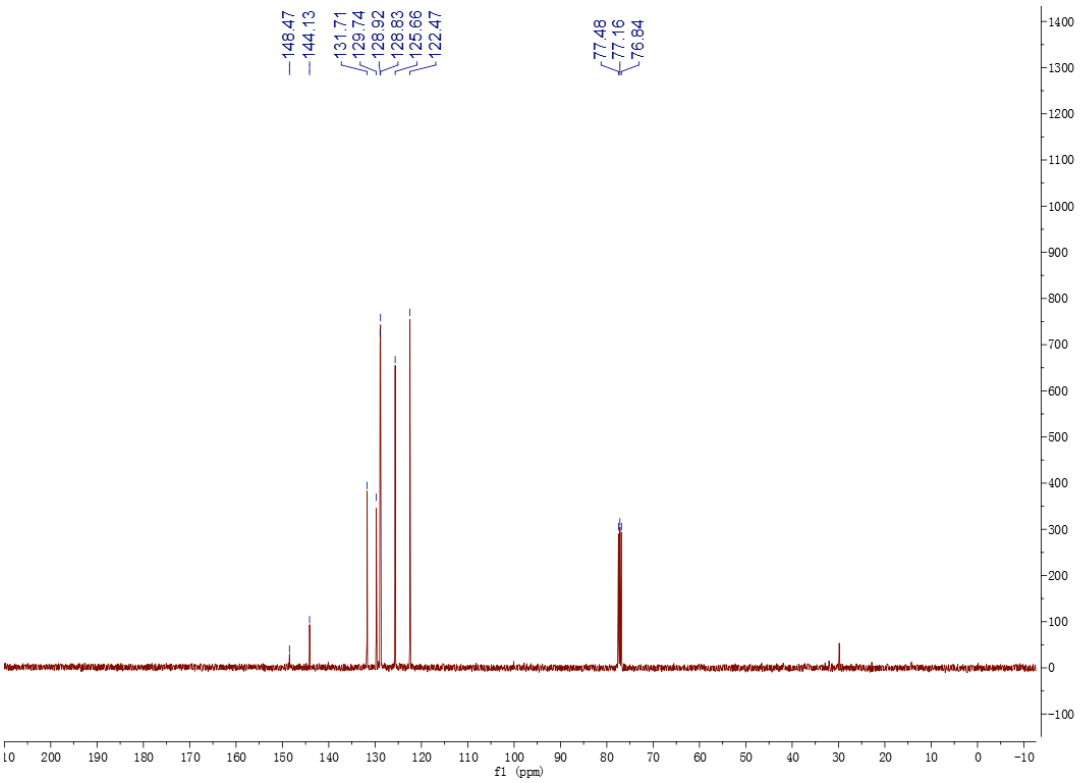
8.32
8.20
8.18
7.57
7.55
7.54
7.52
7.50
7.48
7.42
7.41
7.39
1.94
1.93
5.00
0.99
f1 (ppm)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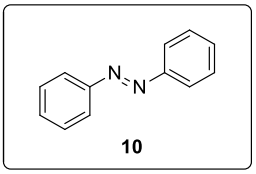
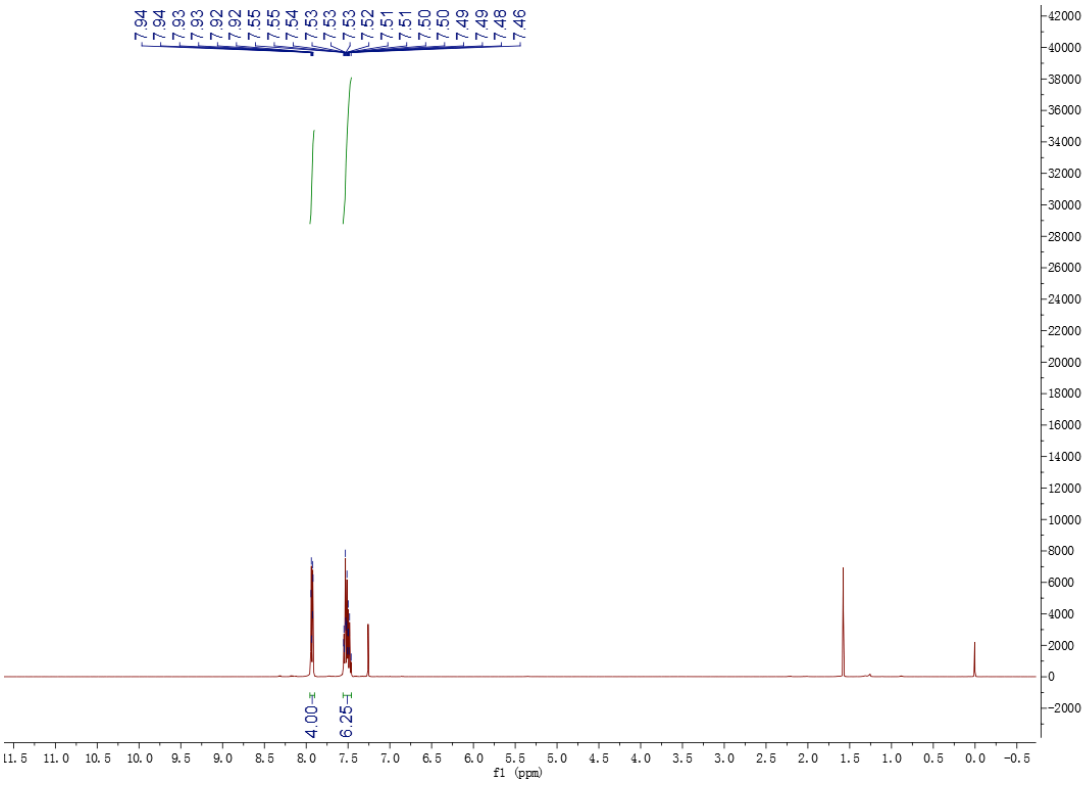
128.83
125.66
122.47
77.48
77.16
76.84
f1 (ppm)



10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7.94
7.93
7.93
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7.92
7.55
7.55
7.54
7.54
7.53
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7.46
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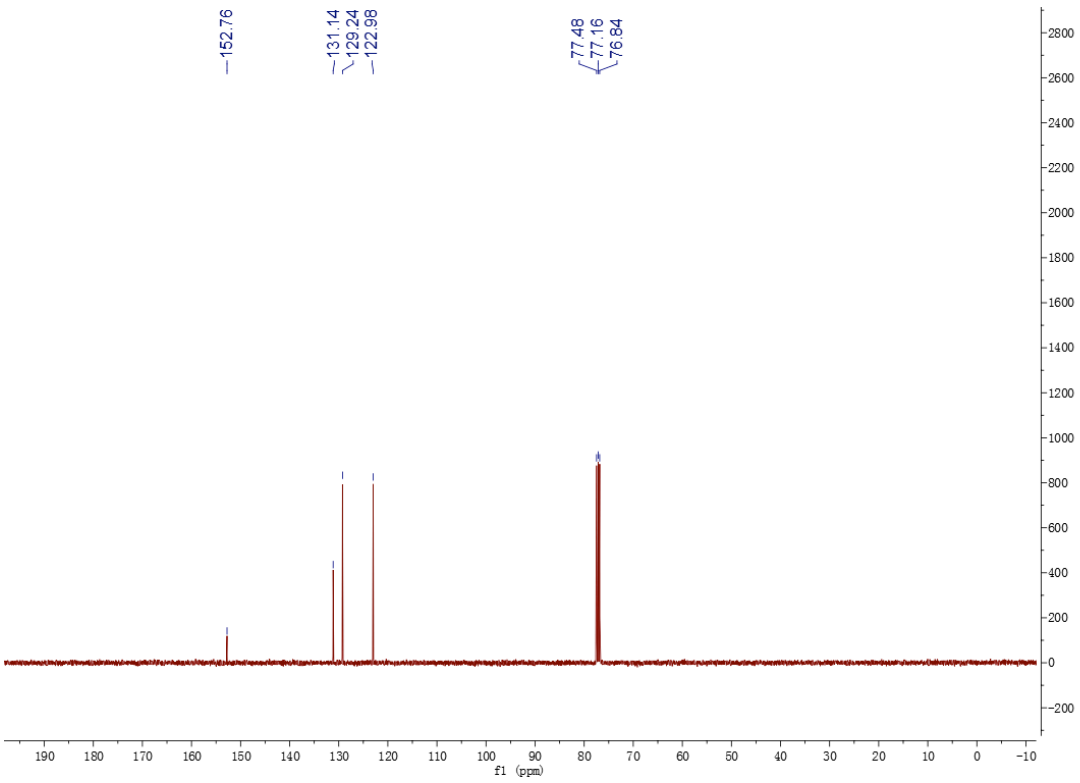
6.25
f1 (ppm)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122.98
77.48
77.16
76.84
f1 (ppm)