

Supporting Information

Nickel-Catalyzed Reductive Coupling of 2-Pyridyl Esters with Unreactivated Alkyl Chlorides: A Universal Synthesis of Aryl-Alkyl and Dialkyl Ketones *via* Dynamic Halide Exchange

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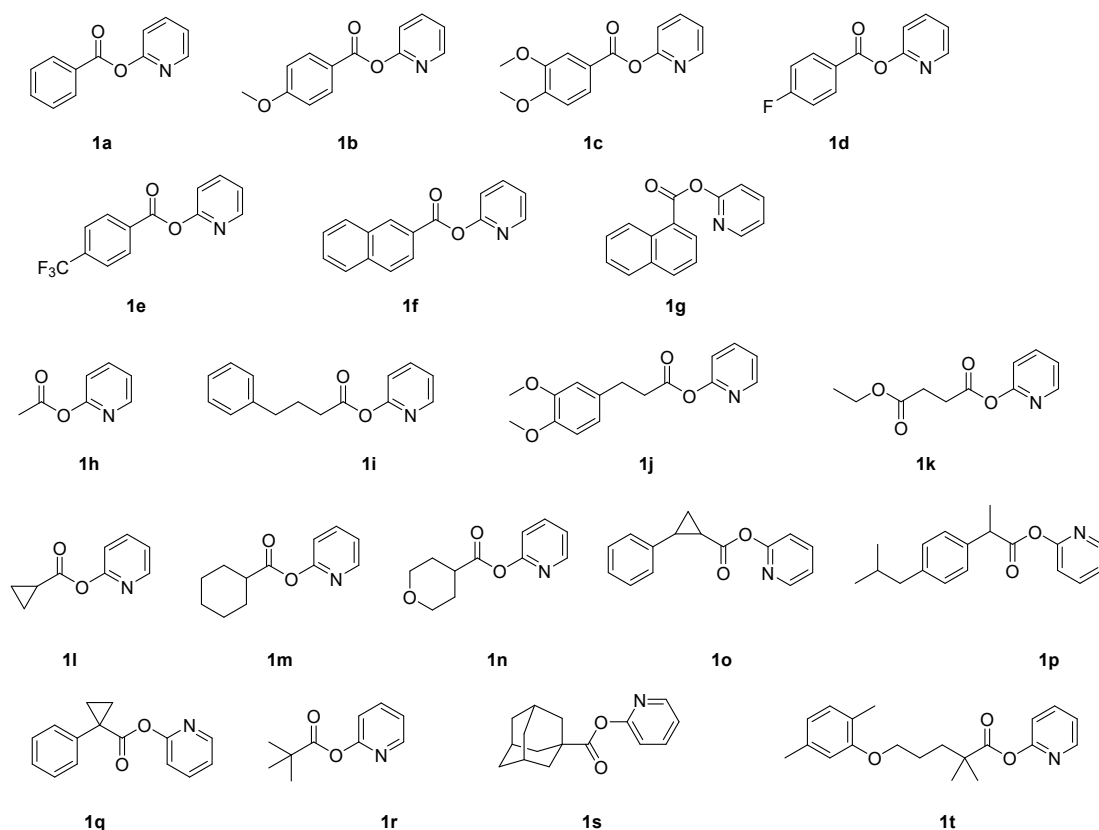
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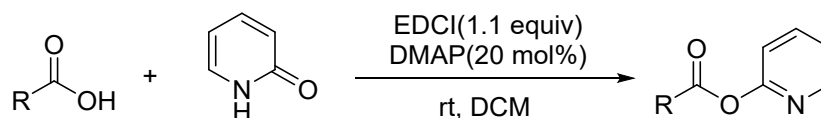
1. General Information

Unless otherwise noted, all reagents and solvents were obtained from commercial suppliers and used without further purification. Reactions involving air or moisture-sensitive reagents or intermediates were performed under an inert atmosphere of nitrogen or argon in oven-dried glassware and a glove box. The products were purified by flash column chromatography on silica gel (300-400 meshes). GC and GC-MS monitored the reactions, GC-MS results were recorded on GC-MS 7890B/7000D, and All GC analyses were performed on GC 2014C. *n*-tridecane was used as an internal standard to calculate GC yields. ^1H , ^{13}C , and ^{19}F NMR spectra were recorded on Bruker ADVANCE III 500, and chemical shifts in parts per million (ppm) were reported. ^1H NMR spectra were recorded on a 500 MHz spectrometer at ambient temperature. Data were reported as follows: (1) chemical shift in parts per million (δ , ppm) from CDCl_3 (7.26 ppm); (2) multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br for broad.); (3) coupling constants (Hz). ^{13}C NMR spectra were recorded on a 125 MHz spectrometer at ambient temperature. Chemical shifts were reported in ppm from CDCl_3 (77.00 ppm), and ^{19}F NMR spectra were recorded on a 470 MHz spectrometer at ambient temperature. The melting points (MP) were determined using the SGW® X-4 melting point apparatus. High-resolution mass spectra (HRMS) were obtained on a Waters XEVO G2-XS QTOF mass spectrometer with ESI resource.

2. Preparation of 2-Pyridyl Esters:

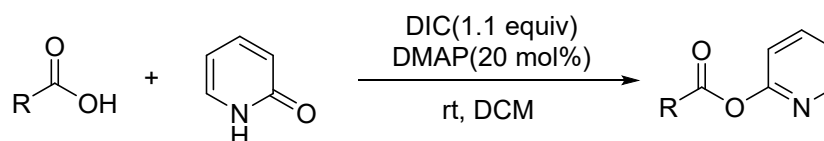


General procedure A for substituted 2-pyridyl esters synthesis (GP-A) ^[1]

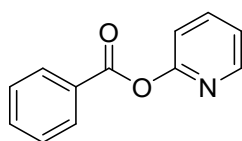


In a round-bottomed flask, carboxylic acid (1.0 equiv) was combined with pyridine-2-ol (1.0 equiv), DMAP (0.20 equiv), EDCI (1.1 equiv), and DCM (0.30 M). The reaction progress was monitored by GC/TLC. Once the starting material was completely consumed, the reaction was quenched with saturated aqueous NaHCO_3 and extracted three times with DCM. The combined organic layers were washed with brine, dried over Na_2SO_4 , and filtered. After concentration in vacuo, the residue was purified by flash column chromatography to obtain the corresponding 2-pyridyl ester.

General procedure B for substituted 2-pyridyl esters synthesis (GP-B) ^[1]



Carboxylic acid (1.0 equiv) was added to a round-bottomed flask along with pyridine-2-ol (1.0 equiv), DMAP (0.20 equiv), DIC (1.1 equiv), and DCM (0.30 M). The progress of the reaction was monitored by GC/TLC. Upon complete consumption of the starting material, the mixture was filtered through Celite and rinsed with additional DCM. The filtrate was then concentrated in vacuo, and the resulting residue was purified by flash column chromatography to obtain the corresponding 2-pyridyl ester.

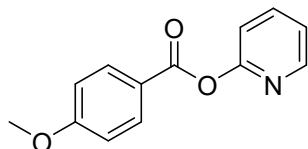


Pyridin-2-yl benzoate ^[1]

Following **GP-A**, the title compound **1a** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.47 (d, *J* = 4.8 Hz, 1H), 8.26 – 8.20 (m, 2H), 7.87 – 7.81 (m, 1H), 7.67 – 7.62 (m, 1H), 7.54 – 7.49 (m, 2H), 7.30 – 7.25 (m, 1H), 7.22 (d, *J* = 8.1 Hz, 1H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 164.8, 158.3, 148.7, 139.6, 133.9, 130.4, 129.1, 128.6, 122.1, 116.7.

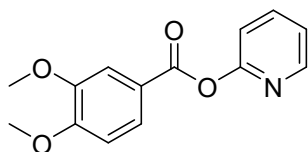


Pyridin-2-yl 4-methoxybenzoate ^[1]

Following **GP-A**, the title compound **1b** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.47 – 8.43 (m, 1H), 8.21 – 8.16 (m, 2H), 7.85 – 7.79 (m, 1H), 7.28 – 7.22 (m, 1H), 7.20 (d, *J* = 8.1 Hz, 1H), 7.01 – 6.95 (m, 2H), 3.88 (s, 3H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 164.5, 164.1, 158.4, 148.6, 139.5, 132.6, 122.0, 121.4, 116.8, 113.9, 55.5.

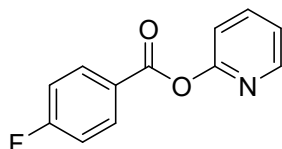


Pyridin-2-yl 3,4-dimethoxybenzoate ^[1]

Following **GP-A**, the title compound **1c** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.49 – 8.44 (m, 1H), 7.90 (dd, J = 8.5, 2.0 Hz, 1H), 7.87 – 7.81 (m, 1H), 7.70 (d, J = 2.0 Hz, 1H), 7.29 – 7.26 (m, 1H), 7.22 (d, J = 8.1 Hz, 1H), 6.96 (d, J = 8.4 Hz, 1H), 3.97 (d, J = 9.0 Hz, 6H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 164.6, 158.4, 153.8, 148.8, 148.7, 139.5, 124.8, 122.0, 121.5, 116.8, 112.5, 110.4, 56.1.

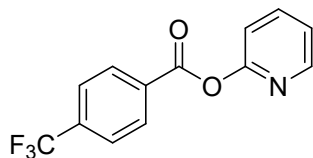


Pyridin-2-yl 4-fluorobenzoate ^[1]

Following **GP-A**, the title compound **1d** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.41 – 8.36 (m, 1H), 8.21 – 8.14 (m, 2H), 7.81 – 7.73 (m, 1H), 7.23 – 7.20 (m, 1H), 7.15 – 7.07 (m, 3H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 166.3 (d, J = 255.6 Hz), 163.9, 158.1, 148.7, 139.6, 133.0 (d, J = 9.6 Hz), 125.4 (d, J = 3.1 Hz), , 122.2, 116.6, 115.8 (d, J = 22.1 Hz).

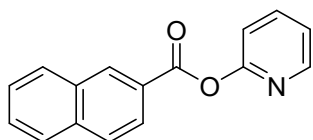


Pyridin-2-yl 4-(trifluoromethyl)benzoate ^[1]

Following **GP-A**, the title compound **1e** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.41 – 8.35 (m, 1H), 8.26 (t, J = 7.0 Hz, 2H), 7.81 – 7.73 (m, 1H), 7.69 (t, J = 7.0 Hz, 2H), 7.25 – 7.19 (m, 1H), 7.16 – 7.11 (m, 1H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 163.6, 157.9, 148.8, 139.7, 135.2 (q, J = 32.9 Hz), 132.4, 130.7, 125.7 (q, J = 3.8 Hz), 122.5 (q, J = 271.2 Hz), 122.5, 116.5..



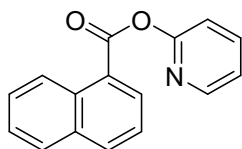
Pyridin-2-yl 2-naphthoate ^[1]

Following **GP-A**, the title compound **1f** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.84 (s, 1H), 8.51 – 8.47 (m, 1H), 8.22 (dd, J =

8.6, 1.8 Hz, 1H), 8.00 (d, J = 8.1 Hz, 1H), 7.94 (dd, J = 15.6, 8.4 Hz, 2H), 7.87 (td, J = 7.8, 2.0 Hz, 1H), 7.66 – 7.61 (m, 1H), 7.61 – 7.56 (m, 1H), 7.32 – 7.27 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 165.0, 158.3, 148.8, 139.6, 136.0, 132.5, 132.4, 129.6, 128.8, 128.5, 127.9, 126.9, 126.3, 125.5, 122.2, 116.7.

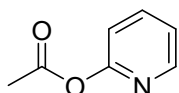


Pyridin-2-yl 1-naphthoate ^[1]

Following **GP-A**, the title compound **1g** was isolated as a white solid.

^1H NMR (500 MHz, Chloroform- d) δ 9.07 (d, J = 8.7 Hz, 1H), 8.56 (d, J = 7.2 Hz, 1H), 8.51 (dd, J = 4.9, 2.0 Hz, 1H), 8.12 (d, J = 8.2 Hz, 1H), 7.93 (d, J = 8.1 Hz, 1H), 7.88 (td, J = 7.7, 2.0 Hz, 1H), 7.68 – 7.62 (m, 1H), 7.57 (td, J = 7.5, 3.6 Hz, 2H), 7.32 – 7.26 (m, 2H).

^{13}C NMR (125 MHz, Chloroform- d) δ 165.3, 158.4, 148.8, 139.6, 134.7, 133.9, 131.8, 131.8, 128.7, 128.3, 126.5, 125.8, 125.2, 124.5, 122.1, 116.9.

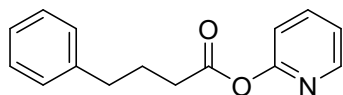


Pyridin-2-yl acetate ^[1]

Following **GP-A**, the title compound **1h** was isolated as a clear oil.

^1H NMR (500 MHz, Chloroform- d) δ 8.43 – 8.40 (m, 1H), 7.82 – 7.77 (m, 1H), 7.25 – 7.21 (m, 1H), 7.09 (d, J = 8.1 Hz, 1H), 2.35 (s, 3H).

^{13}C NMR (125 MHz, Chloroform- d) δ 169.1, 157.8, 148.6, 139.6, 122.1, 116.5, 21.2.



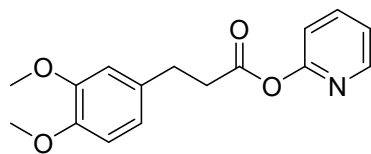
Pyridin-2-yl 4-phenylbutanoate ^[1]

Following **GP-A**, the title compound **1i** was isolated as a white solid.

^1H NMR (500 MHz, Chloroform- d) δ 8.40 (dd, J = 4.8, 2.0 Hz, 1H), 7.78 (td, J = 7.8, 2.0 Hz, 1H), 7.30 (t, J = 7.6 Hz, 2H), 7.21 (dd, J = 8.6, 3.2 Hz, 4H), 7.05 (d, J = 8.1 Hz, 1H), 2.75 (t, J = 7.6 Hz, 2H), 2.63 (t, J = 7.4 Hz, 2H), 2.10 (p, J = 7.5 Hz, 2H).

^{13}C NMR (125 MHz, Chloroform- d) δ 171.6, 158.0, 148.6, 141.2, 139.5, 128.6, 128.5,

126.1, 122.0, 116.4, 35.0, 33.7, 26.3.

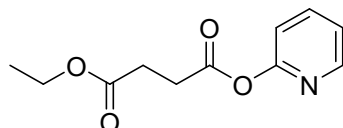


Pyridin-2-yl 3-(3,4-dimethoxyphenyl)propanoate ^[1]

Following **GP-A**, the title compound **1j** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.31 (dd, $J = 5.0, 2.0$ Hz, 1H), 7.68 (td, $J = 7.7, 2.1$ Hz, 1H), 7.17 – 7.09 (m, 1H), 6.92 (d, $J = 8.1$ Hz, 1H), 6.76 – 6.70 (m, 3H), 3.78 (d, $J = 7.8$ Hz, 6H), 2.96 (t, $J = 7.7$ Hz, 2H), 2.84 (t, $J = 7.5$ Hz, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 171.0, 157.9, 149.0, 148.6, 147.7, 139.5, 132.7, 122.1, 120.2, 116.4, 111.8, 111.4, 55.9, 55.8, 36.4, 30.4.

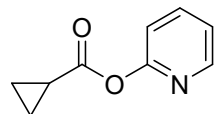


Ethyl pyridin-2-yl succinate ^[1]

Following **GP-B**, the title compound **1k** was isolated as a clear oil.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.40 (d, $J = 4.7$ Hz, 1H), 7.82 – 7.75 (m, 1H), 7.25 – 7.19 (m, 1H), 7.10 (d, $J = 8.1$ Hz, 1H), 4.18 (q, $J = 7.1$ Hz, 2H), 2.94 (t, $J = 6.8$ Hz, 2H), 2.75 (t, $J = 6.8$ Hz, 2H), 1.30 – 1.25 (m, 3H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 172.0, 170.6, 157.8, 148.6, 139.5, 122.1, 116.4, 60.8, 29.4, 29.0, 14.2.

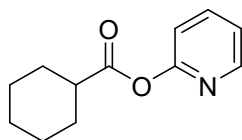


Pyridin-2-yl cyclopropanecarboxylate ^[1]

Following **GP-A**, the title compound **1l** was isolated as a clear oil.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.40 (d, $J = 4.7$ Hz, 1H), 7.80 – 7.74 (m, 1H), 7.23 – 7.18 (m, 1H), 7.09 (d, $J = 8.1$ Hz, 1H), 1.92 – 1.84 (m, 1H), 1.24 – 1.18 (m, 2H), 1.07 – 1.01 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 173.1, 158.0, 148.6, 139.4, 121.9, 116.5, 13.1, 9.6.

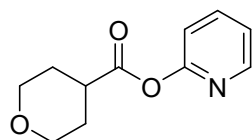


Pyridin-2-yl cyclohexanecarboxylate ^[1]

Following **GP-B**, the title compound **1m** was isolated as a yellow oil.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.40 (d, J = 4.0 Hz, 1H), 7.24 – 7.17 (m, 1H), 7.05 (d, J = 8.1 Hz, 1H), 2.65 – 2.55 (m, 1H), 2.10 (d, J = 10.8 Hz, 2H), 1.87 – 1.79 (m, 2H), 1.73 – 1.55 (m, 3H), 1.42 – 1.24 (m, 3H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 174.1, 158.2, 148.6, 139.4, 121.9, 116.4, 43.2, 28.8, 25.7, 25.3.

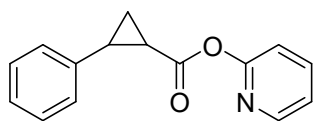


Pyridin-2-yl tetrahydro-2H-pyran-4-carboxylate ^[1]

Following **GP-B**, the title compound **1n** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.42 (d, J = 4.7 Hz, 1H), 7.83 – 7.76 (m, 1H), 7.24 (dd, J = 7.2, 5.0 Hz, 1H), 7.07 (d, J = 8.1 Hz, 1H), 4.04 (dt, J = 11.6, 3.5 Hz, 3H), 3.58 – 3.43 (m, 2H), 2.91 – 2.81 (m, 1H), 2.08 – 2.00 (m, 2H), 2.00 – 1.93 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 172.6, 158.0, 148.6, 139.5, 122.1, 116.3, 67.0, 40.2, 28.5.

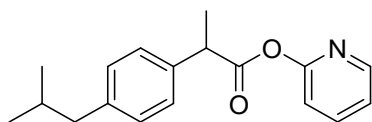


Pyridin-2-yl 2-phenylcyclopropane-1-carboxylate ^[1]

Following **GP-A**, the title compound **1o** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.43 – 8.39 (m, 1H), 7.81 – 7.77 (m, 1H), 7.25 – 7.21 (m, 2H), 7.17 – 7.14 (m, 2H), 7.11 (d, J = 8.1 Hz, 1H), 2.78 – 2.71 (m, 1H), 2.21 – 2.14 (m, 1H), 1.80 (dt, J = 9.7, 5.0 Hz, 1H), 1.54 – 1.48 (m, 1H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 171.6, 157.9, 148.6, 139.5, 139.4, 128.6, 126.8, 126.3, 122.1, 116.5, 27.4, 24.2, 18.0.

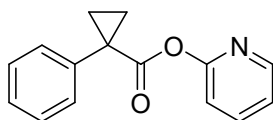


Pyridin-2-yl 2-(4-isobutylphenyl)propanoate ^[1]

Following **GP-A**, the title compound **1p** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.38 (dd, J = 4.9, 1.5 Hz, 1H), 7.75 – 7.69 (m, 1H), 7.32 (d, J = 8.1 Hz, 2H), 7.20 – 7.16 (m, 1H), 7.14 (d, J = 8.0 Hz, 2H), 6.95 (d, J = 8.1 Hz, 1H), 3.99 (q, J = 7.1 Hz, 1H), 2.46 (d, J = 7.2 Hz, 2H), 1.90 – 1.81 (m, 1H), 1.62 (d, J = 7.2 Hz, 3H), 0.91 (d, J = 6.6 Hz, 6H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 172.8, 158.1, 148.5, 140.9, 139.4, 136.9, 129.6, 127.3, 122.0, 116.3, 45.3, 45.1, 30.2, 22.4, 18.5.

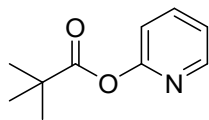


Pyridin-2-yl 1-phenylcyclopropane-1-carboxylate ^[1]

Following **GP-A**, the title compound **1q** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.37 (dd, J = 4.9, 1.5 Hz, 1H), 7.73 (td, J = 8.2, 1.9 Hz, 1H), 7.49 (dt, J = 8.2, 1.8 Hz, 2H), 7.37 – 7.32 (m, 2H), 7.30 – 7.26 (m, 1H), 7.18 (dd, J = 6.6, 5.1 Hz, 1H), 7.00 (d, J = 8.1 Hz, 1H), 1.85 (q, J = 4.1 Hz, 2H), 1.39 (q, J = 4.1 Hz, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 173.1, 158.2, 148.5, 139.4, 138.7, 130.7, 128.3, 127.5, 121.9, 116.5, 29.2, 17.6.

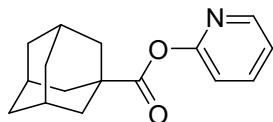


Pyridin-2-yl pivalate ^[1]

Following **GP-A**, the title compound **1k** was isolated as a clear oil.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.41 (d, J = 4.2 Hz, 1H), 7.81 – 7.71 (m, 1H), 7.23 – 7.16 (m, 1H), 7.04 (d, J = 8.1 Hz, 1H), 1.39 (s, 9H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 176.77, 158.43, 148.62, 139.40, 121.86, 116.41, 39.18, 27.05.

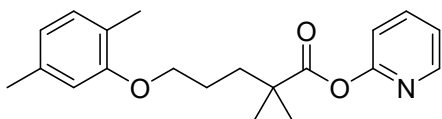


Pyridin-2-yl adamantane-1-carboxylate ^[1]

Following **GP-A**, the title compound **1s** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.41 (dd, J = 4.9, 2.0 Hz, 1H), 7.77 (td, J = 7.7, 2.0 Hz, 1H), 7.23 – 7.18 (m, 1H), 7.03 (d, J = 8.1 Hz, 1H), 2.09 (s, 9H), 1.77 (s, 6H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 175.8, 158.5, 148.6, 139.3, 121.8, 116.5, 41.1, 38.6, 36.4, 27.9.



Pyridin-2-yl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate ^[1]

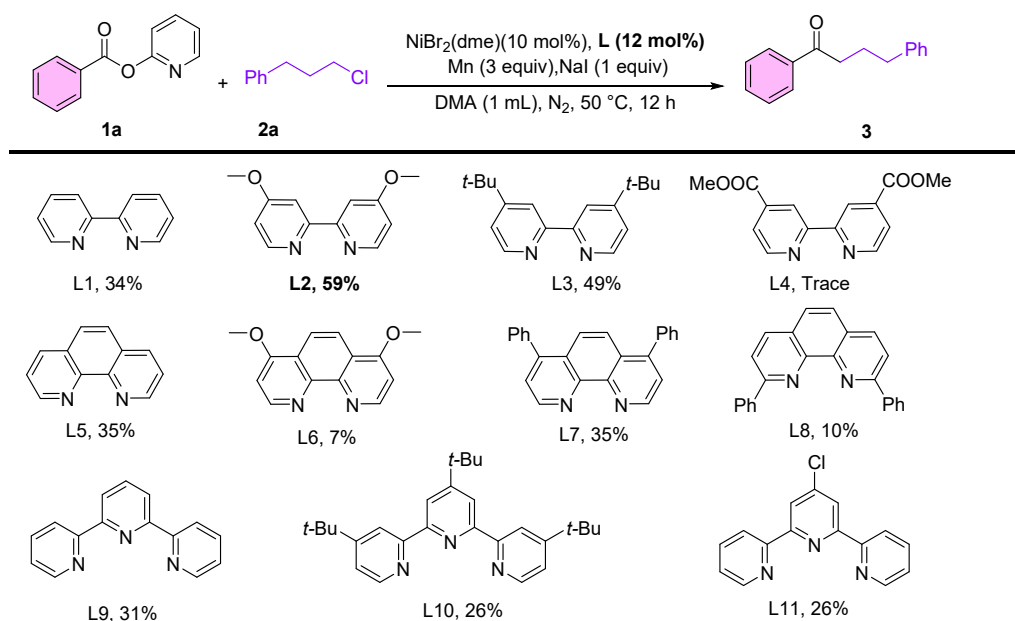
Following **GP-A**, the title compound **1t** was isolated as a white solid.

¹H NMR (500 MHz, Chloroform-*d*) δ 8.34 (dd, J = 4.9, 1.4 Hz, 1H), 7.74 – 7.66 (m, 1H), 7.16 – 7.09 (m, 1H), 6.92 (d, J = 7.4 Hz, 2H), 6.59 (d, J = 7.4 Hz, 1H), 6.55 (s, 1H), 3.92 (s, 2H), 2.23 (s, 3H), 2.10 (s, 3H), 1.84 (s, 3H), 1.33 (s, 6H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 176.1, 158.4, 156.9, 148.7, 139.4, 136.5, 130.32, 123.6, 121.9, 120.7, 116.4, 112.0, 67.8, 42.5, 37.1, 25.2, 25.1, 21.4, 15.8.

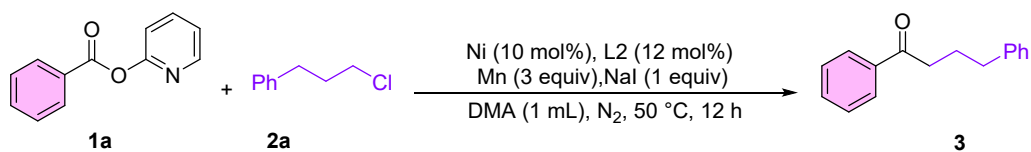
3. Optimization of the Reaction Conditions

Table S1. Optimization of the Ligands ^a



Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), NiBr₂(dme) (10 mol%), ligand (12 mol%), NaI (1.0 equiv), and Mn (3.0 equiv) in DMA (1 mL) at 50 °C for 12 h. ^a Yields were detected by GC yields vs *n*-tridecane internal standard. N.D. = not detected.

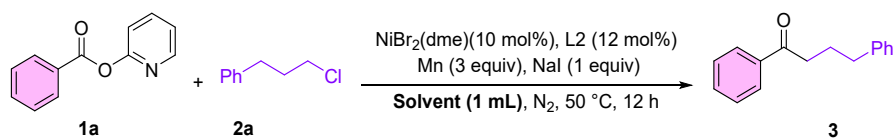
Table S2. Optimization of the Catalysts



Entry	Catalyst	3^a
1	Ni(COD) ₂	11
2	NiF ₂	N.D.
3	NiCl ₂	34
4	NiBr ₂	30
5	NiCl ₂ ·dme	18
6	NiCl ₂ ·dppf	Trace
7	NiCl ₂ ·dppe	11
8	NiCl ₂ ·(PCy ₃) ₂	Trace
9	(1,3-dppp) NiCl ₂	12
10	Ni (OAc) ₂ ·4H ₂ O	21
11	NiClO ₄ ·6H ₂ O	9
12	NiCl ₂ ·6H ₂ O	24
13	NiBr ₂ ·3H ₂ O	35
14	Ni (OTf) ₂	Trace
15	Ni(acac) ₂	29
16	NiBr₂·dme	59

Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), [Ni] (10 mol%), L2 (12 mol%), NaI (1.0 equiv), and Mn (3.0 equiv) in DMA (1.0 mL) at 50 °C for 12 h. ^a Yields were detected by GC yields vs *n*-tridecane internal standard. N.D. = not detected.

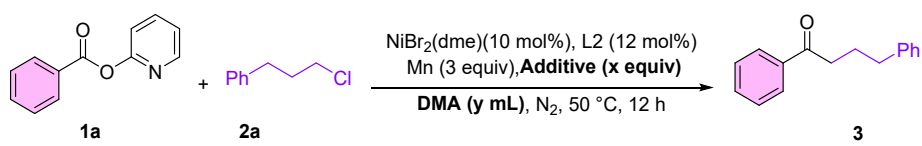
Table S3. Optimization of the Solvents



Entry	Solvent	3 ^a
1	DMA	59
2	DMF	19
3	DMSO	20
4	NMP	9
5	CH_3CN	5
6	1,4-dioxane	N.D.
7	THF	N.D.

Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), $\text{NiBr}_2(\text{dme})$ (10 mol%), L2 (12 mol%), NaI (1.0 equiv), and Mn (3.0 equiv) in solvent (1 mL) at 50 °C for 12 h.^a Yields were detected by GC yields vs *n*-tridecane internal standard. N.D. = not detected.

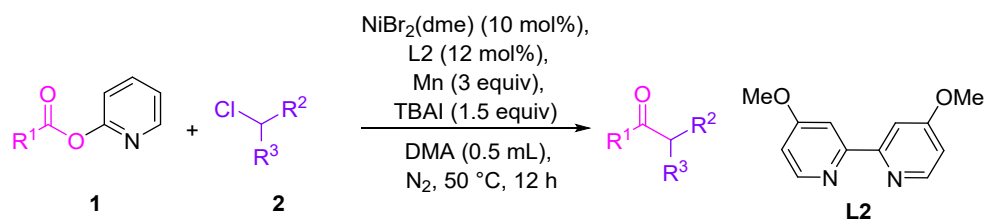
Table S4. Optimization of the Additives

			
Entry	Additive (x equiv)	DMA (y mL)	3 ^a
1	NaI (1.0 equiv)	1.0	58
2	KI (1.0 equiv)	1.0	56
3	LiI (1.0 equiv)	1.0	47
4	TBAI (1.0 equiv)	1.0	59
5	TMAI (1.0 equiv)	1.0	11
6	TBAI (1.5 equiv)	1.0	60
7	TBAI (1.5 equiv)	0.5	83
8	TBAI (1.0 equiv)	0.5	67
9	TBAI (1.0 equiv)	2.0	33

Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), NiBr₂(dme) (10 mol%), L2 (12 mol%), additive (x equiv), and Mn (3.0 equiv) in DMA (y mL) at 50 °C for 12 h. ^a

Yields were detected by GC yields vs *n*-tridecane internal standard. N.D. = not detected.

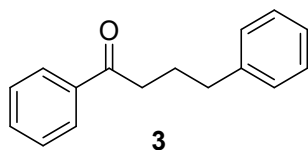
4. Nickel-Catalyzed Reductive Coupling of 2-Pyridyl Ester with Alkyl Chlorides



To a Schlenk tube (10 mL) equipped with a stir bar was added $\text{NiBr}_2(\text{dme})$ (6.2 mg, 0.02 mmol, 10 mol%), **L2** (5.2 mg, 0.024 mmol, 12 mol%) under an argon atmosphere. Next, 0.5 mL of dry DMA was added. To this solution, TBAI (110.8 mg, 0.3 mmol, 1.5 equiv), Mn powder (33.0 mg, 0.6 mmol, 3.0 equiv), 2-pyridyl esters **1** (0.2 mmol), alkyl chloride **2** (0.3 mmol, 1.5 equiv.) was added successively under nitrogen atmosphere. The reaction mixture was stirred at 50 °C for 12 hours. The resulting black solution was cooled to room temperature and then passed through a plug of silica gel (200–300 mesh). The silica gel was subsequently rinsed with 5 mL of ethyl acetate to ensure complete transfer of the product. The combined filtrates were concentrated using a rotary evaporator to remove the solvent and volatile materials. The resulting residue was then purified by silica gel chromatography to afford the desired products.

5. Analytical Data of Products

1,4-Diphenylbutan-1-one (3) ^[2]



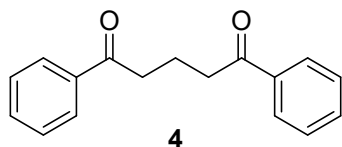
The title compound **3** was isolated as a white solid (32.3 mg, 72% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.94 – 7.90 (m, 2H), 7.57 – 7.52 (m, 1H), 7.44 (t, J = 7.5 Hz, 2H), 7.31 – 7.26 (m, 2H), 7.23 – 7.17 (m, 3H), 2.98 (t, J = 7.5 Hz, 2H), 2.72 (t, J = 7.6 Hz, 2H), 2.09 (p, J = 7.4 Hz, 2H).

¹³C NMR (125MHz, Chloroform-*d*) δ 200.2, 141.7, 137.0, 133.0, 128.6, 128.5, 128.4, 128.0, 126.0, 37.7, 35.2, 25.7.

HRMS (ESI-TOF) m/z : $[M + K]^+$ Calcd for C₁₆H₁₆O K 263.0838; Found 263.0835.

1,5-Diphenylpentane-1,5-dione (4) ^[3]



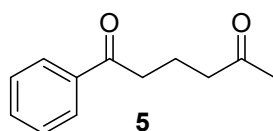
The title compound **4** was isolated as a white solid (37.8 mg, 75% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.98 (d, J = 7.7 Hz, 4H), 7.55 (t, J = 7.4 Hz, 2H), 7.46 (t, J = 7.6 Hz, 4H), 3.12 (t, J = 7.0 Hz, 4H), 2.20 (p, J = 7.0 Hz, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 199.9, 136.9, 133.1, 128.6, 128.1, 37.6, 18.7.

HRMS (ESI-TOF) m/z : $[M + Na]^+$ Calcd for C₁₇H₁₆O₂ Na 275.1048; Found 275.1057.

1-Phenylhexane-1,5-dione (5) ^[4]



The title compound **5** was isolated as a white solid (20.9 mg, 55% yield).

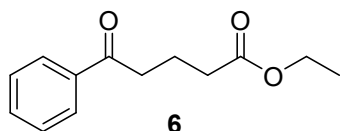
¹H NMR (500 MHz, Chloroform-*d*) δ 7.96 (d, J = 7.5 Hz, 2H), 7.56 (t, J = 7.3 Hz, 1H), 7.46 (t, J = 7.5 Hz, 2H), 3.02 (t, J = 7.0 Hz, 2H), 2.57 (t, J = 7.0 Hz, 2H), 2.15 (s, 3H),

2.02 (p, $J = 7.1$ Hz, 2H).

^{13}C NMR (125 MHz, Chloroform- d) δ 208.5, 199.8, 136.8, 133.1, 128.6, 128.1, 42.6, 37.4, 30.0, 18.2.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{12}\text{H}_{14}\text{O}_2$ H 191.1072; Found 191.1080.

Ethyl 5-oxo-5-phenylpentanoate (6) ^[5]



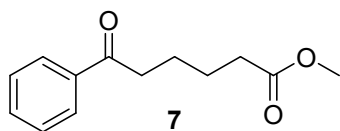
The title compound **6** was isolated as a clear oil (26.4 mg, 60% yield).

^1H NMR (500 MHz, Chloroform- d) δ 7.98 – 7.94 (m, 2H), 7.56 (t, $J = 7.3$ Hz, 1H), 7.46 (t, $J = 7.8$ Hz, 2H), 4.14 (q, $J = 7.3$ Hz, 2H), 3.06 (t, $J = 7.3$ Hz, 2H), 2.43 (t, $J = 7.3$ Hz, 2H), 2.08 (p, $J = 7.1$ Hz, 2H), 1.26 (t, $J = 7.0$ Hz, 3H).

^{13}C NMR (125 MHz, Chloroform- d) δ 199.5, 173.3, 136.9, 133.1, 128.6, 128.0, 60.4, 37.5, 33.4, 19.4, 14.2.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_3$ H 221.1177; Found 221.1177.

Methyl 6-oxo-6-phenylhexanoate (7) ^[6]



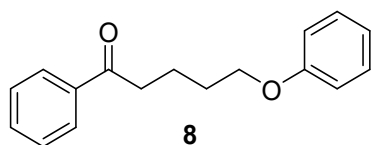
The title compound **7** was isolated as a clear oil (30.2 mg, 68% yield).

^1H NMR (500 MHz, Chloroform- d) δ 7.95 (d, $J = 7.0$ Hz, 2H), 7.56 (t, $J = 7.5$ Hz, 1H), 7.46 (t, $J = 7.8$ Hz, 2H), 3.67 (s, 3H), 3.00 (t, $J = 7.0$ Hz, 2H), 2.38 (t, $J = 7.0$ Hz, 2H), 1.81 – 1.71 (m, 4H).

^{13}C NMR (125 MHz, Chloroform- d) δ 199.8, 173.9, 137.0, 133.0, 128.6, 128.0, 51.5, 38.1, 33.9, 24.6, 23.7.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_3$ H 221.1177; Found 221.1184.

5-Phenoxy-1-phenylpentan-1-one (8) ^[7]



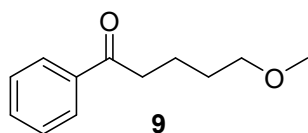
The title compound **8** was isolated as a white solid (46.3 mg, 91% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 7.5 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.30 – 7.24 (m, 2H), 6.93 (t, *J* = 7.3 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 2H), 4.01 (t, *J* = 6.0 Hz, 2H), 3.06 (t, *J* = 7.0 Hz, 2H), 1.98 – 1.86 (m, 4H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 200.0, 159.0, 137.0, 133.0, 129.4, 128.6, 128.1, 120.6, 114.5, 67.5, 38.1, 28.9, 21.0.

HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₇H₁₈O₂ H 255.1385; Found 255.1389.

5-Methoxy-1-phenylpentan-1-one (**9**)



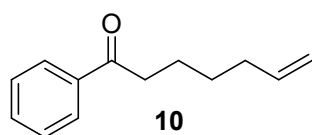
The title compound **9** was isolated as a clear oil (18.1 mg, 47% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.98 – 7.93 (m, 2H), 7.58 – 7.51 (m, 1H), 7.49 – 7.42 (m, 2H), 3.42 (t, *J* = 6.5 Hz, 2H), 3.33 (s, 3H), 3.00 (t, *J* = 7.5 Hz, 2H), 1.82 (p, *J* = 7.5 Hz, 2H), 1.72 – 1.62 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 200.2, 137.1, 132.9, 128.6, 128.0, 72.5, 58.6, 38.3, 29.2, 21.0.

HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₃H₁₆O₃ H 193.1228; Found 193.1237.

1-Phenylhept-6-en-1-one (**10**) ^[2]



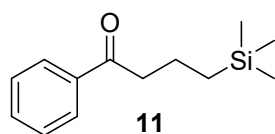
The title compound **10** was isolated as a yellow oil (12.8 mg, 34% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.91 – 7.87 (m, 2H), 7.51 – 7.46 (m, 1H), 7.42 – 7.37 (m, 2H), 5.80 – 5.70 (m, 1H), 4.98 – 4.92 (m, 1H), 4.91 – 4.86 (m, 1H), 2.94 – 2.88 (m, 2H), 2.08 – 2.01 (m, 2H), 1.73 – 1.65 (m, 2H), 1.46 – 1.38 (m, 2H).

^{13}C NMR (125 MHz, Chloroform-*d*) δ 200.4, 138.6, 137.0, 133.0, 128.6, 128.1, 114.7, 38.4, 33.6, 28.6, 23.8.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}$ 189.1279; Found 189.1276.

1-Phenyl-4-(trimethylsilyl)butan-1-one (11) ^[8]



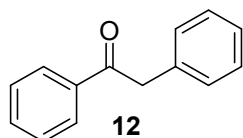
The title compound **11** was isolated as a yellow oil (14.1 mg, 32% yield).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.95 (d, $J = 7.5$ Hz, 2H), 7.54 (t, $J = 7.3$ Hz, 1H), 7.45 (t, $J = 7.8$ Hz, 2H), 2.98 (t, $J = 7.3$ Hz, 2H), 1.80 – 1.71 (m, 2H), 0.61-0.54 (m, 2H), -0.001 (s, 9H).

^{13}C NMR (125 MHz, Chloroform-*d*) δ 202.4, 138.9, 134.6, 130.3, 129.8, 44.1, 20.9, 18.4, 0.001.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{20}\text{OSi}$ 221.1361; Found 221.1364.

1,2-Diphenylethan-1-one (12) ^[9]



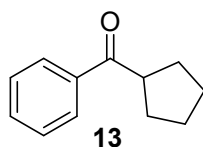
The title compound **12** was isolated as a white solid (18.0 mg, 46% yield).

^1H NMR (500 MHz, Chloroform-*d*) δ 8.01 (d, $J = 7.5$ Hz, 2H), 7.55 (t, $J = 7.3$ Hz, 1H), 7.45 (t, $J = 7.5$ Hz, 2H), 7.32 (t, $J = 7.3$ Hz, 2H), 7.29 – 7.24 (m, 3H), 4.28 (s, 2H).

^{13}C NMR (125 MHz, Chloroform-*d*) δ 197.7, 136.6, 134.6, 133.2, 129.5, 128.7, 128.7, 128.6, 126.9, 45.5.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{14}\text{H}_{12}\text{O}$ 197.0966; Found 197.0974.

Cyclopentyl(phenyl)methanone (13) ^[2]



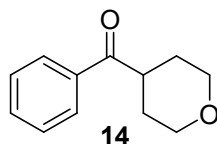
The title compound **13** was isolated as a white solid (16.7 mg, 48% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 8.01 – 7.95 (m, 2H), 7.58 – 7.51 (m, 1H), 7.46 (t, J = 7.5 Hz, 2H), 3.72 (p, J = 7.9 Hz, 1H), 1.97 – 1.88 (m, 4H), 1.78 – 1.62 (m, 4H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 202.8, 137.0, 132.7, 128.50, 128.46, 46.4, 30.0, 26.3.

HRMS (ESI-TOF) m/z : $[M + K]^+$ Calcd for C₁₂H₁₄O K 213.0682; Found 213.0675.

Phenyl(tetrahydro-2H-pyran-4-yl)methanone (14) ^[10]



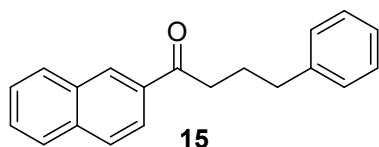
The title compound **14** was isolated as a white solid (6.5 mg, 17% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.97 – 7.93 (m, 2H), 7.60 – 7.55 (m, 1H), 7.48 (t, J = 7.7 Hz, 2H), 4.10 – 4.03 (m, 2H), 3.57 (td, J = 11.6, 2.2 Hz, 2H), 3.54 – 3.47 (m, 1H), 1.94 – 1.84 (m, 2H), 1.83 – 1.76 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 201.9, 135.8, 133.1, 128.8, 128.23, 67.3, 42.6, 29.1.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₁₄H₁₂O₂ H 191.1072; Found 191.1073.

1-(Naphthalen-2-yl)-4-phenylbutan-1-one (15) ^[11]



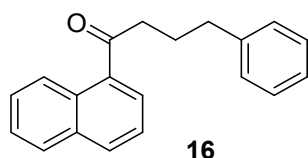
The title compound **15** was isolated as a white solid (41.2 mg, 75% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 8.40 (s, 1H), 8.00 (d, J = 8.5 Hz, 1H), 7.93 (d, J = 8.5 Hz, 1H), 7.90 – 7.84 (m, 2H), 7.59 (t, J = 7.5 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.30 (t, J = 7.5 Hz, 2H), 7.22 (dd, J = 15.7, 7.6 Hz, 3H), 3.11 (t, J = 7.3 Hz, 2H), 2.77 (t, J = 7.5 Hz, 2H), 2.15 (p, J = 7.4 Hz, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 200.1, 141.7, 135.6, 134.4, 132.6, 129.6, 129.6, 128.6, 128.4, 128.4, 128.4, 127.8, 126.7, 126.0, 123.9, 37.7, 35.2, 25.9.

HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₂₀H₁₈O Na 297.1256; Found 297.1261.

1-(Naphthalen-1-yl)-4-phenylbutan-1-one (16) ^[11]



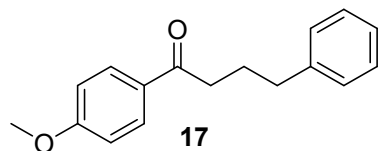
The title compound **16** was isolated as a white solid (27.4 mg, 50% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.97 – 7.93 (m, 1H), 7.86 – 7.82 (m, 1H), 7.70 (d, *J* = 8.1 Hz, 1H), 7.50 – 7.43 (m, 2H), 7.38 (t, *J* = 7.6 Hz, 1H), 7.34 – 7.26 (m, 3H), 7.24 – 7.16 (m, 3H), 3.10 (t, *J* = 7.8 Hz, 2H), 2.75 (t, *J* = 7.7 Hz, 2H), 2.13 – 2.06 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 142.2, 138.4, 133.9, 131.9, 128.8, 128.5, 128.4, 126.6, 126.0, 125.8, 125.7, 125.5, 125.4, 123.8, 35.9, 32.6, 32.3.

HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₀H₁₈O H 275.1436; Found 275.1443.

1-(4-Methoxyphenyl)-4-phenylbutan-1-one (17) ^[2]



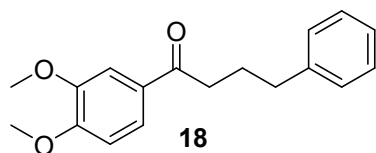
The title compound **17** was isolated as a white solid (43.8 mg, 86% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.2 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.20 (d, *J* = 6.9 Hz, 3H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H), 2.92 (t, *J* = 7.3 Hz, 2H), 2.71 (t, *J* = 7.6 Hz, 2H), 2.11 – 2.03 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 198.7, 163.4, 141.8, 130.3, 130.2, 128.5, 128.4, 125.9, 113.7, 55.5, 37.4, 35.3, 26.0.

HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₇H₁₈O₂ H 255.1385; Found 255.1394.

1-(3,4-Dimethoxyphenyl)-4-phenylbutan-1-one (18) ^[9]



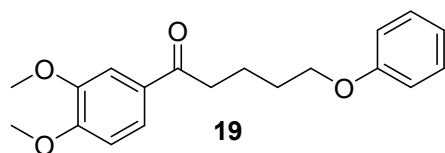
The title compound **18** was isolated as a white solid (31.3 mg, 55% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.54 – 7.49 (m, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.23 – 7.17 (m, 3H), 6.86 (d, *J* = 8.5 Hz, 1H), 3.93 (s, 3H), 3.92 (s, 3H), 2.93 (t, *J* = 7.5 Hz, 2H), 2.72 (t, *J* = 7.5 Hz, 2H), 2.08 (p, *J* = 7.4 Hz, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 198.8, 153.2, 149.0, 141.8, 130.3, 128.5, 128.4, 125.9, 122.7, 110.2, 110.0, 56.1, 56.0, 37.2, 35.3, 26.1.

HRMS (ESI-TOF) *m/z*: [M + K]⁺ Calcd for C₁₈H₂₀O₃ K 323.1049; Found 323.1049.

1-(3,4-Dimethoxyphenyl)-5-phenoxypentan-1-one (**19**)



The title compound **19** was isolated as a white solid (44.0 mg, 70% yield).

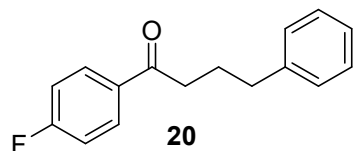
¹H NMR (500 MHz, Chloroform-*d*) δ 7.59 (dd, *J* = 2.0, 2.0 Hz, 1H), 7.53 (d, *J* = 2.0 Hz, 1H), 7.30 – 7.25 (m, 2H), 6.93 (t, *J* = 7.3 Hz, 1H), 6.91 – 6.86 (m, 3H), 4.01 (t, *J* = 6.0 Hz, 2H), 3.94 (d, *J* = 8.0 Hz, 6H), 3.02 (t, *J* = 7.0 Hz, 2H), 1.98 – 1.85 (m, 4H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 198.7, 159.0, 153.2, 149.0, 130.3, 129.4, 122.7, 120.6, 114.5, 110.1, 110.0, 67.5, 56.1, 56.0, 37.6, 28.9, 21.3.

HRMS (ESI-TOF) *m/z*: [M + K]⁺ Calcd for C₁₉H₂₂O₄ K 353.1155; Found 353.1161.

Melting point: 82–84 °C;

1-(4-Fluorophenyl)-4-phenylbutan-1-one (**20**) ^[2]



The title compound **20** was isolated as a white solid (24.2 mg, 50% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.97 – 7.92 (m, 2H), 7.32 – 7.27 (m, 2H), 7.22 – 7.18 (m, 3H), 7.14 – 7.08 (m, 2H), 2.95 (t, *J* = 7.5 Hz, 2H), 2.72 (t, *J* = 7.8 Hz, 2H),

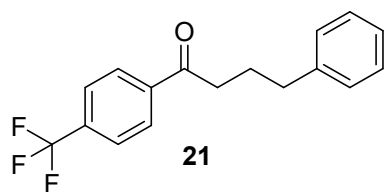
2.08 (p, $J = 7.5$ Hz, 2H).

^{13}C NMR (125 MHz, Chloroform- d) δ 198.5, 165.7 (d, $J = 254.5$ Hz), 141.6, 133.4 (d, $J = 2.9$ Hz), 130.6 (d, $J = 9.3$ Hz), 128.5, 128.4, 126.0, 115.6 (d, $J = 21.9$ Hz), 37.6, 35.2, 25.7.

^{19}F NMR (470 MHz, Chloroform- d) δ -105.57.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{15}\text{FO}$ H 243.1185; Found 243.1182.

4-Phenyl-1-(4-(trifluoromethyl)phenyl)butan-1-one (**21**) ^[2]



The title compound **21** was isolated as a white solid (25.7 mg, 44% yield).

^1H NMR (500 MHz, Chloroform- d) δ 8.00 (d, $J = 8.1$ Hz, 2H), 7.71 (d, $J = 8.2$ Hz, 2H), 7.33 – 7.27 (m, 2H), 7.23 – 7.18 (m, 3H), 2.99 (t, $J = 7.3$ Hz, 2H), 2.73 (t, $J = 7.5$ Hz, 2H), 2.10 (p, $J = 7.4$ Hz, 2H).

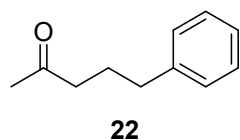
^{13}C NMR (125 MHz, Chloroform- d) δ 199.1, 141.4, 139.6, 134.3 (q, $J = 32.7$ Hz), 128.53, 128.49, 128.4, 126.1, 125.7 (q, $J = 3.7$ Hz), 123.6 (q, $J = 272.7$ Hz), 37.9, 35.1, 25.5.

^{19}F NMR (470 MHz, Chloroform- d) δ -63.07.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{17}\text{H}_{15}\text{F}_3\text{O}$ H 293.1153; Found 293.1147.

Melting point: 48 – 50 °C;

5-Phenylpentan-2-one (**22**) ^[10]



The title compound **22** was isolated as a yellow oil (7.1 mg, 22% yield).

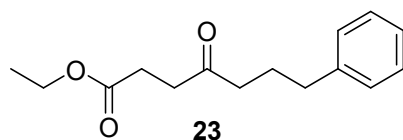
^1H NMR (500 MHz, Chloroform- d) δ 7.31 – 7.26 (m, 2H), 7.21 – 7.15 (m, 3H), 2.62 (t, $J = 7$ Hz, 2H), 2.43 (t, $J = 7.4$ Hz, 2H), 2.11 (s, 3H), 1.91 (p, $J = 7.5$ Hz, 2H).

^{13}C NMR (125 MHz, Chloroform- d) δ 208.8, 141.6, 128.5, 128.4, 126.0, 42.9, 35.1,

30.0, 25.2.

HRMS (ESI-TOF) m/z : $[M + K]^+$ Calcd for $C_{11}H_{14}O$ K 201.0682; Found 201.0679.

Ethyl 4-oxo-7-phenylheptanoate (23) ^[12]



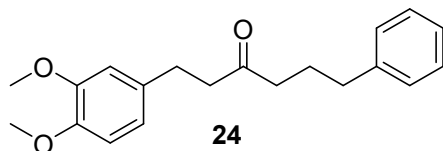
The title compound **23** was isolated as a clear oil (18.9 mg, 38% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.28 (t, J = 7.4 Hz, 2H), 7.18 (dd, J = 12.3, 7.1 Hz, 3H), 4.12 (q, J = 7.2 Hz, 2H), 2.68 (t, J = 6.5 Hz, 2H), 2.62 (t, J = 7.6 Hz, 2H), 2.56 (t, J = 6.5 Hz, 2H), 2.46 (t, J = 7.4 Hz, 2H), 1.93 (p, J = 7.5 Hz, 2H), 1.24 (t, J = 7.1 Hz, 3H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 208.7, 172.8, 141.6, 128.5, 128.4, 126.0, 60.6, 41.9, 37.1, 35.1, 28.0, 25.2, 14.2.

HRMS (ESI-TOF) m/z : $[M + K]^+$ Calcd for $C_{15}H_{20}O_3$ K 287.1049; Found 287.1057.

1-(3,4-Dimethoxyphenyl)-6-phenylhexan-3-one (24) ^[13]



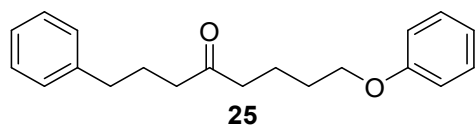
The title compound **24** was isolated as a clear oil (14.4 mg, 23% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.29 – 7.24 (m, 2H), 7.21 – 7.11 (m, 3H), 6.77 (d, J = 8.6 Hz, 1H), 6.69 (dd, J = 4.3, 2.4 Hz, 2H), 3.85 (s, 3H), 3.84 (s, 3H), 2.82 (t, J = 7.6 Hz, 2H), 2.67 (t, J = 7.5 Hz, 2H), 2.59 (t, J = 7.6 Hz, 2H), 2.38 (t, J = 7.4 Hz, 2H), 1.89 (p, J = 7.4 Hz, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 210.0, 148.9, 147.4, 141.6, 133.8, 128.5, 128.4, 126.0, 120.1, 111.8, 111.4, 56.0, 55.9, 44.6, 42.2, 35.1, 29.4, 25.2.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for $C_{20}H_{24}O_3$ H 313.1803; Found 313.1808.

8-Phenoxy-1-phenyloctan-4-one (25)



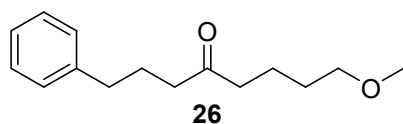
The title compound **25** was isolated as a clear oil (28.5 mg, 48% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.30 – 7.25 (m, 4H), 7.21 – 7.15 (m, 3H), 6.95 – 6.91 (m, 1H), 6.90 – 6.85 (m, 2H), 3.94 (t, *J* = 5.5 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 2.45 (t, *J* = 6.7 Hz, 2H), 2.41 (t, *J* = 7.4 Hz, 2H), 1.91 (p, *J* = 8.0, 7.6 Hz, 2H), 1.80 – 1.71 (m, 4H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 210.6, 159.0, 141.6, 129.5, 128.5, 128.4, 126.0, 120.6, 114.5, 67.4, 42.4, 42.0, 35.1, 28.8, 25.2, 20.5.

HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₀H₂₄O₂ H 297.1854; Found 297.1863.

8-Methoxy-1-phenyloctan-4-one (**26**)



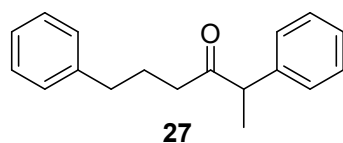
The title compound **26** was isolated as a clear oil (23.9 mg, 51% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.27 (t, *J* = 7.5 Hz, 2H), 7.20 – 7.14 (m, 3H), 3.35 (t, *J* = 6.3 Hz, 2H), 3.30 (s, 3H), 2.61 (t, *J* = 7.6 Hz, 2H), 2.40 (td, *J* = 7.3, 2.5 Hz, 4H), 1.90 (p, *J* = 7.5 Hz, 2H), 1.67 – 1.51 (m, 4H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 210.7, 141.7, 128.5, 128.4, 125.9, 72.5, 58.5, 42.5, 41.9, 35.1, 29.1, 25.2, 20.5.

HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₅H₂₂O₂ H 235.1698; Found 235.1696.

2,6-Diphenylhexan-3-one (**27**) ^[14]



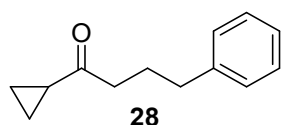
The title compound **27** was isolated as a clear oil (12.1 mg, 24% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.32 (t, J = 7.4 Hz, 2H), 7.28 – 7.17 (m, 5H), 7.15 (t, J = 7.3 Hz, 1H), 7.04 (d, J = 7.0 Hz, 2H), 3.72 (q, J = 6.9 Hz, 1H), 2.56 – 2.41 (m, 2H), 2.41 – 2.30 (m, 2H), 1.82 (p, J = 7.3 Hz, 2H), 1.38 (d, J = 7.0 Hz, 3H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 210.6, 141.7, 140.6, 128.9, 128.4, 128.3, 127.9, 127.1, 125.8, 53.0, 40.2, 34.9, 25.3, 17.4.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₁₈H₂₀O H 253.1592; Found 253.1600.

1-Cyclopropyl-4-phenylbutan-1-one (28) ^[10]



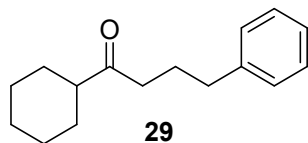
The title compound **28** was isolated as a clear oil (22.6 mg, 60% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.28 (dd, J = 8.5, 6.6 Hz, 2H), 7.21 – 7.16 (m, 3H), 2.63 (t, J = 7.6 Hz, 2H), 2.56 (t, J = 7.4 Hz, 2H), 1.95 (q, J = 7.5 Hz, 2H), 1.92 – 1.87 (m, 1H), 1.03 – 0.98 (m, 2H), 0.87 – 0.81 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 210.8, 141.7, 128.5, 128.4, 125.9, 42.7, 35.2, 25.5, 20.4, 10.6.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₁₃H₁₆O H 189.1279; Found 189.1275.

1-Cyclohexyl-4-phenylbutan-1-one (29) ^[10]



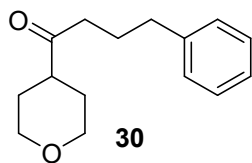
The title compound **29** was isolated as a yellow oil (25.6 mg, 55% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.28 (t, J = 7.6 Hz, 2H), 7.22 – 7.15 (m, 3H), 2.63 (t, J = 7.6 Hz, 2H), 2.56 (t, J = 7.4 Hz, 2H), 1.99 – 1.90 (m, 2H), 1.93 – 1.85 (m, 2H), 1.03 – 0.97 (m, 2H), 0.88 – 0.80 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 214.0, 141.8, 128.5, 128.4, 125.9, 50.9, 39.8, 35.2, 28.5, 25.9, 25.7, 25.1.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₁₆H₂₂O H 231.1749; Found 231.1751.

4-Phenyl-1-(tetrahydro-2H-pyran-4-yl)butan-1-one (**30**) ^[10]



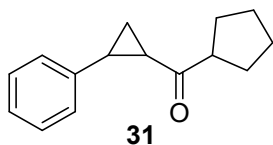
The title compound **30** was isolated as a white solid (28.8 mg, 62% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.28 (t, J = 7.5 Hz, 2H), 7.21 – 7.14 (m, 3H), 3.98 (dt, J = 11.5, 3.6 Hz, 2H), 3.39 (td, J = 11.2, 3.3 Hz, 2H), 2.62 (t, J = 7.5 Hz, 2H), 2.54 – 2.47 (m, 1H), 2.45 (t, J = 7.2 Hz, 2H), 1.92 (p, J = 7.4 Hz, 2H), 1.71 – 1.64 (m, 4H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 211.7, 141.6, 128.5, 128.4, 126.0, 67.3, 47.6, 39.3, 35.1, 28.2, 24.9.

HRMS (ESI-TOF) m/z : $[M + K]^+$ Calcd for C₁₅H₂₀O₂ K 277.1100; Found 277.1104.

Cyclopentyl(2-phenylcyclopropyl)methanone (**31**)



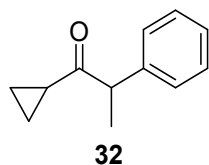
The title compound **31** was isolated as a yellow oil (15.8 mg, 37% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.31 – 7.26 (m, 2H), 7.23 – 7.17 (m, 1H), 7.12 – 7.07 (m, 2H), 3.04 (p, J = 8.0 Hz, 1H), 2.51 – 2.45 (m, 1H), 2.23 – 2.17 (m, 1H), 1.90 – 1.80 (m, 4H), 1.71 – 1.62 (m, 3H), 1.62 – 1.54 (m, 2H), 1.38 – 1.33 (m, 1H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 211.2, 140.6, 128.5, 126.4, 126.1, 52.4, 31.5, 28.9, 28.6, 26.1, 26.0, 18.7.

HRMS (ESI-TOF) m/z : $[M + K]^+$ Calcd for C₁₅H₁₈O K 253.0995; Found 253.0993.

1-Cyclopropyl-2-phenylpropan-1-one (**32**) ^[15]



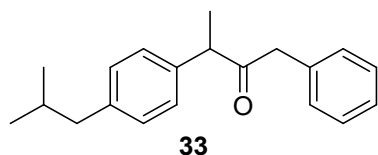
The title compound **32** was isolated as a yellow oil (17.4 mg, 50% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.34 (t, J = 7.5 Hz, 2H), 7.28 – 7.21 (m, 3H), 3.90 (q, J = 7.0 Hz, 1H), 1.85 (td, J = 7.9, 3.9 Hz, 1H), 1.41 (d, J = 7.0 Hz, 3H), 1.04 – 0.92 (m, 2H), 0.84 – 0.66 (m, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 210.9, 140.9, 128.9, 128.1, 127.0, 53.8, 19.7, 17.6, 11.3, 11.3.

HRMS (ESI-TOF) m/z : $[M + Na]^+$ Calcd for C₁₂H₁₄O Na 197.0943; Found 197.0949.

3-(4-Isobutylphenyl)-1-phenylbutan-2-one (33)



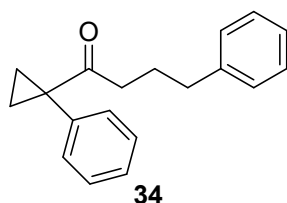
The title compound **33** was isolated as a clear oil (21.9 mg, 39% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.30 – 7.18 (m, 3H), 7.13 – 7.07 (m, 4H), 7.05 – 7.00 (m, 2H), 3.82 (q, J = 6.9 Hz, 1H), 3.62 (d, J = 3.6 Hz, 2H), 2.46 (d, J = 7.2 Hz, 2H), 1.92 – 1.80 (m, 1H), 1.35 (d, J = 6.9 Hz, 3H), 0.91 (d, J = 6.7 Hz, 6H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 208.3, 140.8, 137.5, 134.5, 129.7, 129.5, 128.5, 127.8, 126.8, 51.9, 47.9, 45.1, 30.2, 22.4, 17.6.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for C₂₀H₂₄O H 281.1905; Found 281.1908.

4-Phenyl-1-(1-phenylcyclopropyl)butan-1-one (34) ^[10]



The title compound **34** was isolated as a clear oil (40.0 mg, 76% yield).

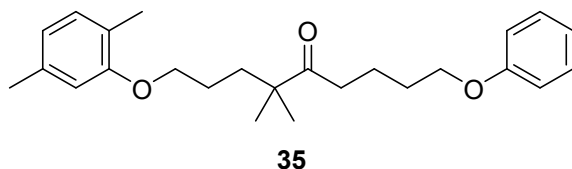
¹H NMR (500 MHz, Chloroform-*d*) δ 7.33 (d, J = 4.3 Hz, 4H), 7.31 – 7.27 (m, 1H), 7.25 – 7.21 (m, 2H), 7.15 (dd, J = 8.1, 6.4 Hz, 1H), 7.07 (d, J = 7.2 Hz, 2H), 2.50 – 2.44 (m, 2H), 2.30 (t, J = 7.1 Hz, 2H), 1.77 (p, J = 7.1 Hz, 2H), 1.58 (q, J = 3.4 Hz, 3H), 1.14 (q, J = 3.6 Hz, 2H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 210.4, 141.9, 140.9, 130.9, 128.6, 128.4, 128.3,

127.4, 125.8, 40.8, 37.4, 35.0, 25.5, 18.6.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for $C_{19}H_{20}O$ 265.1592; Found 265.1592.

1-(2,5-Dimethylphenoxy)-4,4-dimethyl-9-phenoxynonan-5-one (35) ^[2]



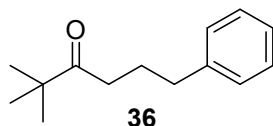
The title compound **35** was isolated as a yellow oil (48.2 mg, 63% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.21 – 7.16 (m, 2H), 6.92 (d, $J = 7.5$ Hz, 1H), 6.87 – 6.83 (m, 1H), 6.82 – 6.78 (m, 2H), 6.58 (d, $J = 7.5$ Hz, 1H), 6.52 (s, 1H), 3.88 (t, $J = 5.9$ Hz, 2H), 3.82 (t, $J = 5.9$ Hz, 2H), 2.50 (t, $J = 6.8$ Hz, 2H), 2.23 (s, 3H), 2.10 (s, 3H), 1.73 – 1.62 (m, 6H), 1.62 – 1.54 (m, 2H), 1.09 (s, 6H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 215.3, 159.0, 156.9, 136.5, 130.4, 129.5, 123.5, 120.8, 120.6, 114.5, 111.9, 67.8, 67.6, 47.3, 36.4, 36.3, 28.9, 25.0, 24.5, 21.5, 20.5, 15.9.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for $C_{25}H_{34}O_3$ 383.2586; Found 383.2581.

2,2-Dimethyl-6-phenylhexan-3-one (36) ^[10]

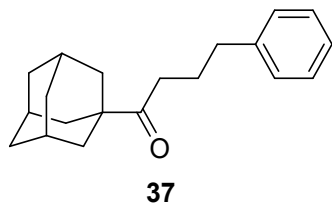


The title compound **36** was isolated as a clear oil (25.0 mg, 61% yield).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.28 (t, $J = 7.6$ Hz, 2H), 7.18 (t, $J = 6.8$ Hz, 3H), 2.61 (t, 2H), 2.50 (t, $J = 7.2$ Hz, 2H), 1.89 (p, $J = 7.3$ Hz, 2H), 1.12 (s, 9H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 215.8, 141.9, 128.5, 128.3, 125.9, 44.1, 35.6, 35.2, 26.4, 25.4.

HRMS (ESI-TOF) m/z : $[M + H]^+$ Calcd for $C_{14}H_{20}O$ 205.1592; Found 205.1593.



1-((3r,5r,7r)-Adamantan-1-yl)-4-phenylbutan-1-one (37) ^[10]

The title compound **37** was isolated as a clear oil (24.3 mg, 43% yield).

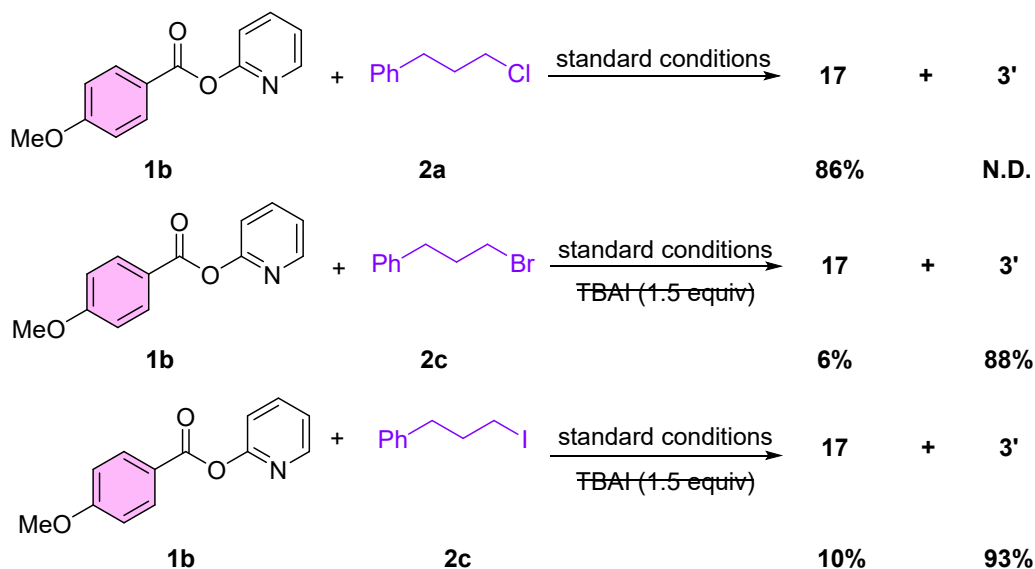
¹H NMR (500 MHz, Chloroform-*d*) δ 7.30 – 7.25 (m, 2H), 7.21 – 7.14 (m, 3H), 2.59 (t, J = 7.7 Hz, 2H), 2.45 (t, J = 7.2 Hz, 2H), 2.05 – 2.00 (m, 3H), 1.87 (p, J = 7.2 Hz, 2H), 1.81 – 1.64 (m, 12H).

¹³C NMR (125 MHz, Chloroform-*d*) δ 215.4, 142.0, 128.5, 128.3, 125.8, 46.3, 38.3, 36.6, 35.2, 35.2, 28.0, 25.2.

HRMS (ESI-TOF) m/z : $[M + K]^+$ Calcd for C₂₀H₂₆O K 321.1621; Found 321.1624.

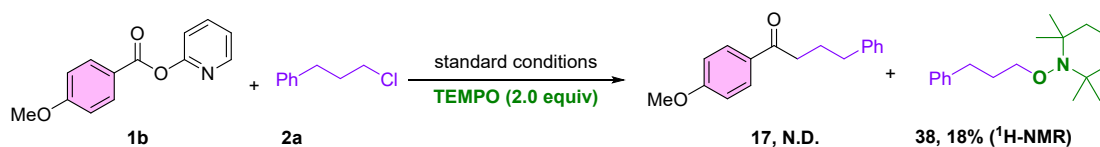
6. Control Experiments.

A)



A dry test tube equipped with a stirring bar was charged with $\text{NiBr}_2(\text{dme})$ (6.2 mg, 0.02 mmol, 10 mol %), **L2** (5.2 mg, 0.024 mmol, 12 mol%) under an argon atmosphere. Next, 0.5 mL of dry DMA was added. To this solution, TBAI (110.8 mg, 0.3 mmol, 1.5 equiv), Mn powder (33.0 mg, 0.6 mmol, 3.0 equiv), pyridin-2-yl 4-methoxybenzoate (**1b**) (45.8 mg, 0.2 mmol, 1.0 equiv) and (3-chloropropyl)benzene (**2a**), alkyl bromide (**2b**) or alkyl iodide (**2c**) (0.3 mmol, 1.5 equiv) was added successively under nitrogen atmosphere. The reaction mixture was stirred at 50 °C for 12 hours. The reaction was subjected to GC analysis.

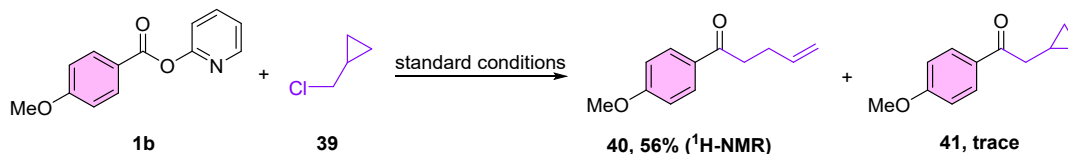
B)



A dry test tube equipped with a stirring bar was charged with $\text{NiBr}_2(\text{dme})$ (6.2 mg, 0.02 mmol, 10 mol %), **L2** (5.2 mg, 0.024 mmol, 12 mol%) under an argon atmosphere. Next, 0.5 mL of dry DMA was added. To this solution, TBAI (110.8 mg, 0.3 mmol, 1.5 equiv), Mn powder (33.0 mg, 0.6 mmol, 3.0 equiv), TEMPO (62.5 mg, 0.4 mmol, 2.0 equiv), pyridin-2-yl 4-methoxybenzoate (**1b**) (45.8 mg, 0.2 mmol, 1.0 equiv) and (3-chloropropyl)benzene (**2a**) (46.2 mg, 0.3 mmol, 1.5 equiv) was added successively

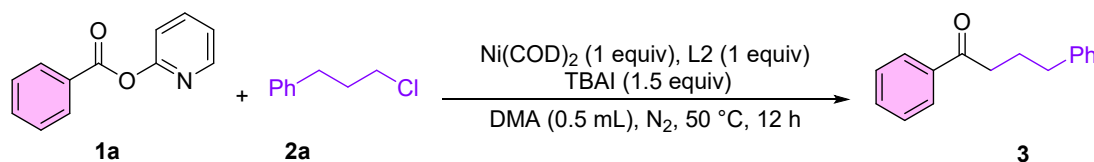
under nitrogen atmosphere. The reaction mixture was stirred at 50 °C for 12 hours. After removing the solvent, the residue was analyzed by ^1H NMR spectroscopy to calculate the yield using dibromomethane as an internal standard.

C)



A dry test tube equipped with a stirring bar was charged with $\text{NiBr}_2(\text{dme})$ (6.2 mg, 0.02 mmol, 10 mol %), **L2** (5.2 mg, 0.024 mmol, 12 mol%) under an argon atmosphere. Next, 0.5 mL of dry DMA was added. To this solution, TBAI (110.8 mg, 0.3 mmol, 1.5 equiv), Mn powder (33.0 mg, 0.6 mmol, 3.0 equiv), pyridin-2-yl 4-methoxybenzoate (**1b**) (45.8 mg, 0.2 mmol, 1.0 equiv) and (chloromethyl)cyclopropane (**39**) (27.0 mg, 0.3 mmol, 1.5 equiv) was added successively under nitrogen atmosphere. The reaction mixture was stirred at 50 °C for 24 hours. After removing the solvent, the residue was analyzed by ^1H NMR spectroscopy to calculate the yield using dibromomethane as an internal standard.

D)

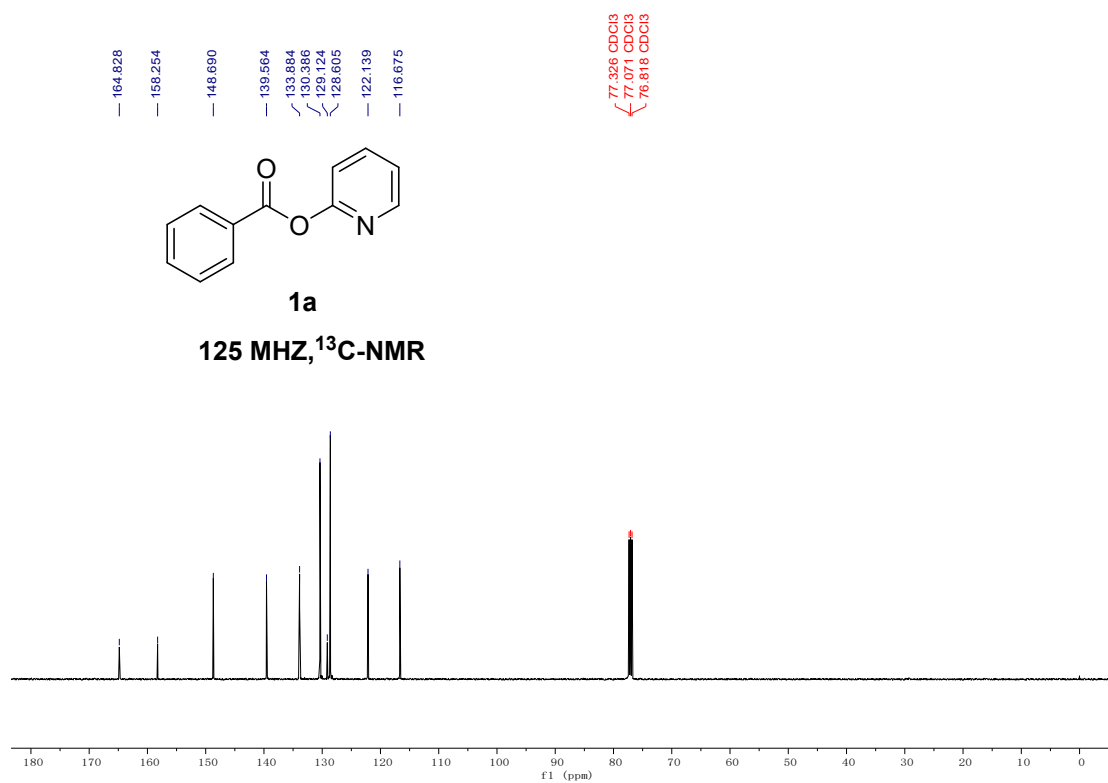
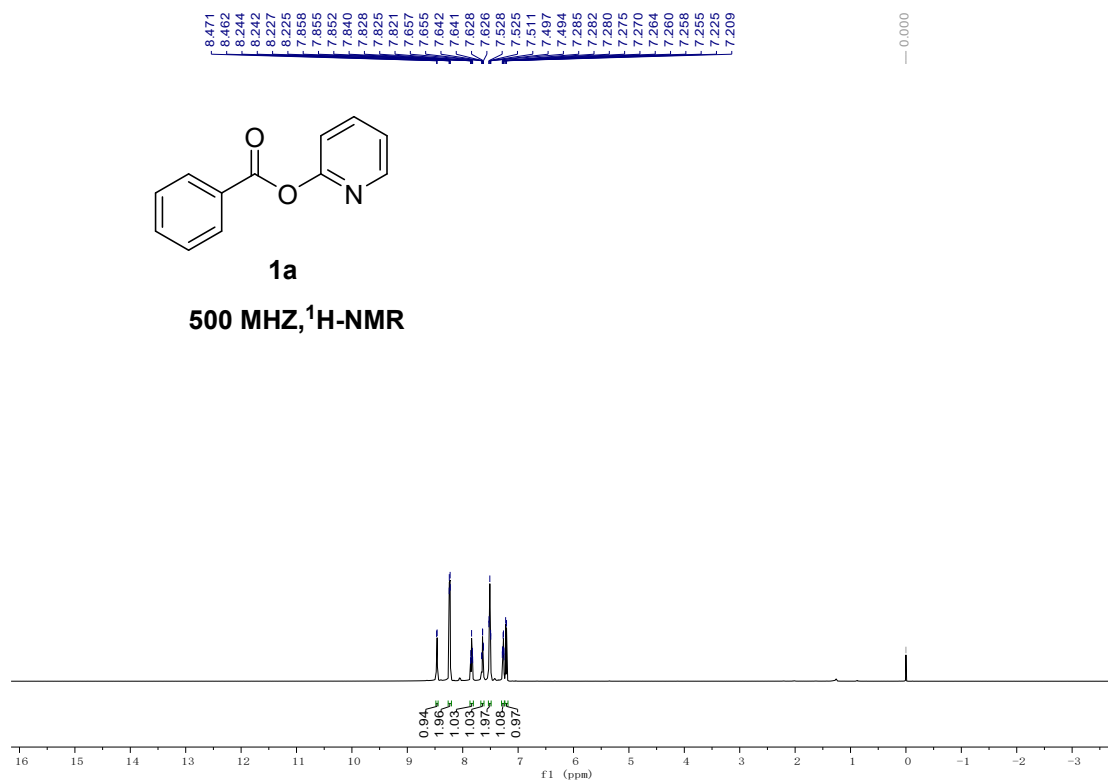


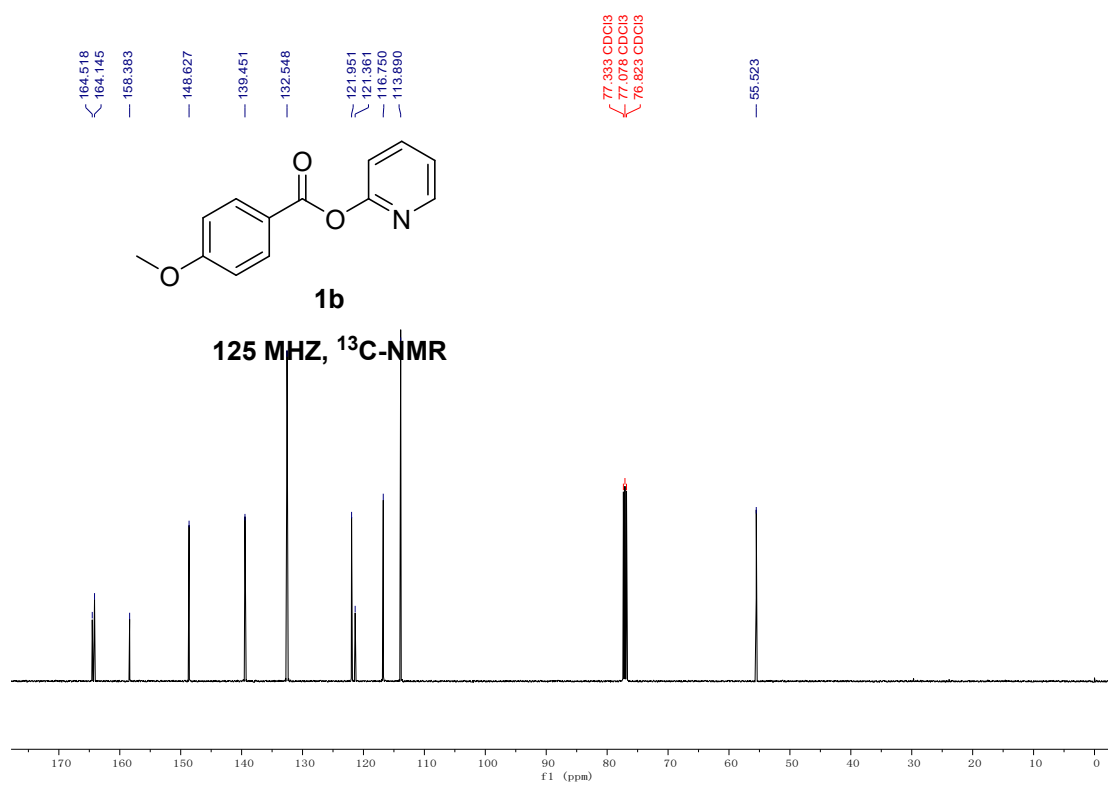
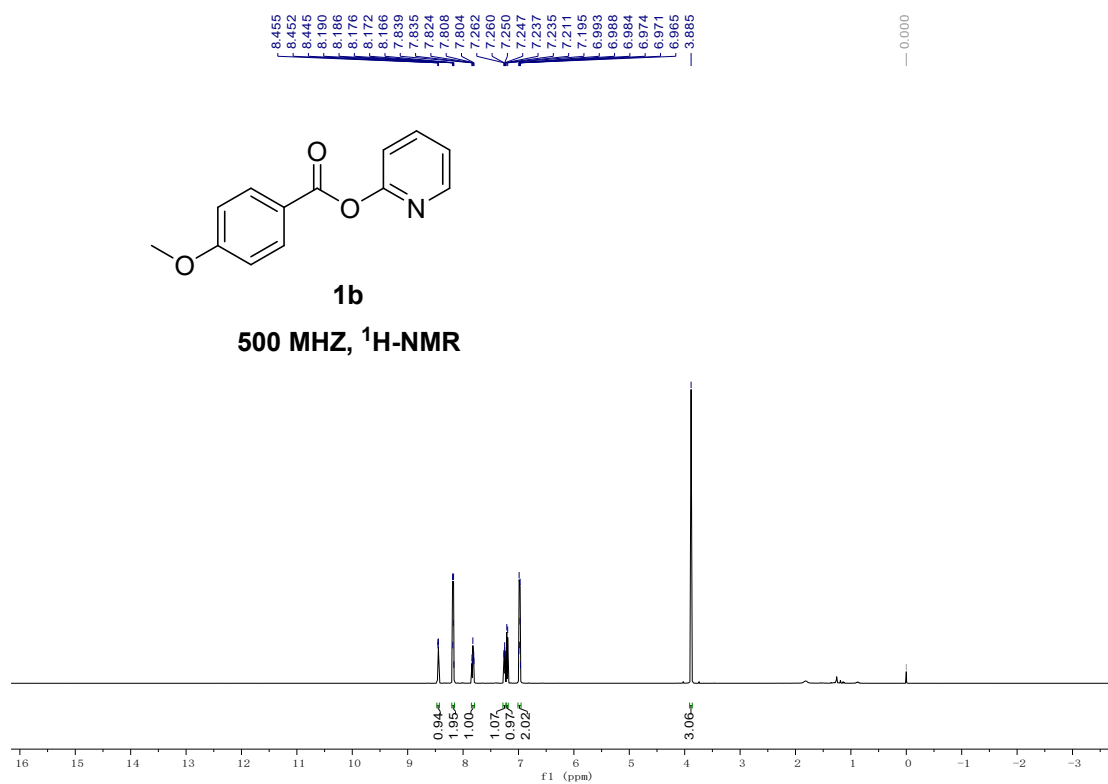
A dry test tube equipped with a stirring bar was charged with $\text{Ni}(\text{COD})_2$ (55.0 mg, 0.2 mmol, 1.0 equiv), **L2** (43.2 mg, 0.2 mmol, 1.0 equiv) under an argon atmosphere inside a glove box. Next, 0.5 mL of dry DMA was added via syringe. Subsequently, removed from the glove box, the Schlenk tube was evacuated and filled with nitrogen (three cycles). To this solution, TBAI (110.8 mg, 0.3 mmol, 1.5 equiv), cyclopropyl(phenyl)methanone (**1a**) (39.8 mg, 0.2 mmol, 1.0 equiv) and (3-chloropropyl)benzene (**2a**) (46.2 mg, 0.3 mmol, 1.5 equiv) was added successively under nitrogen atmosphere. The reaction mixture was stirred at 50 °C for 12 hours. The reaction was subjected to GC analysis.

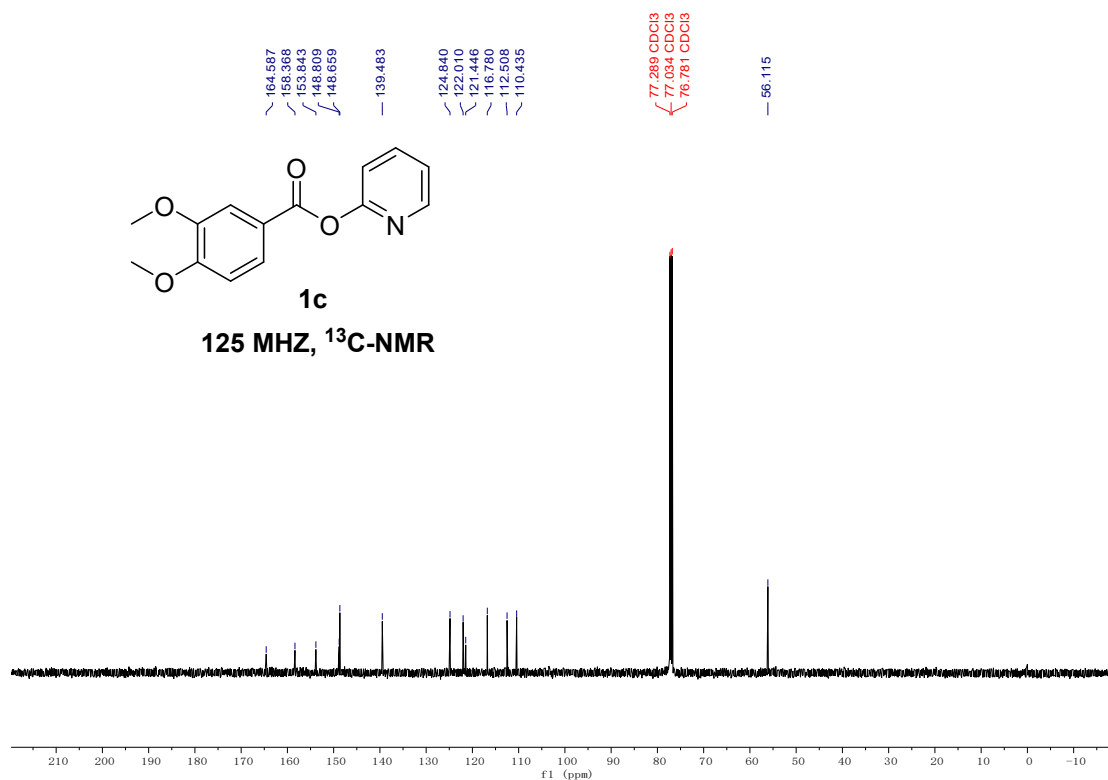
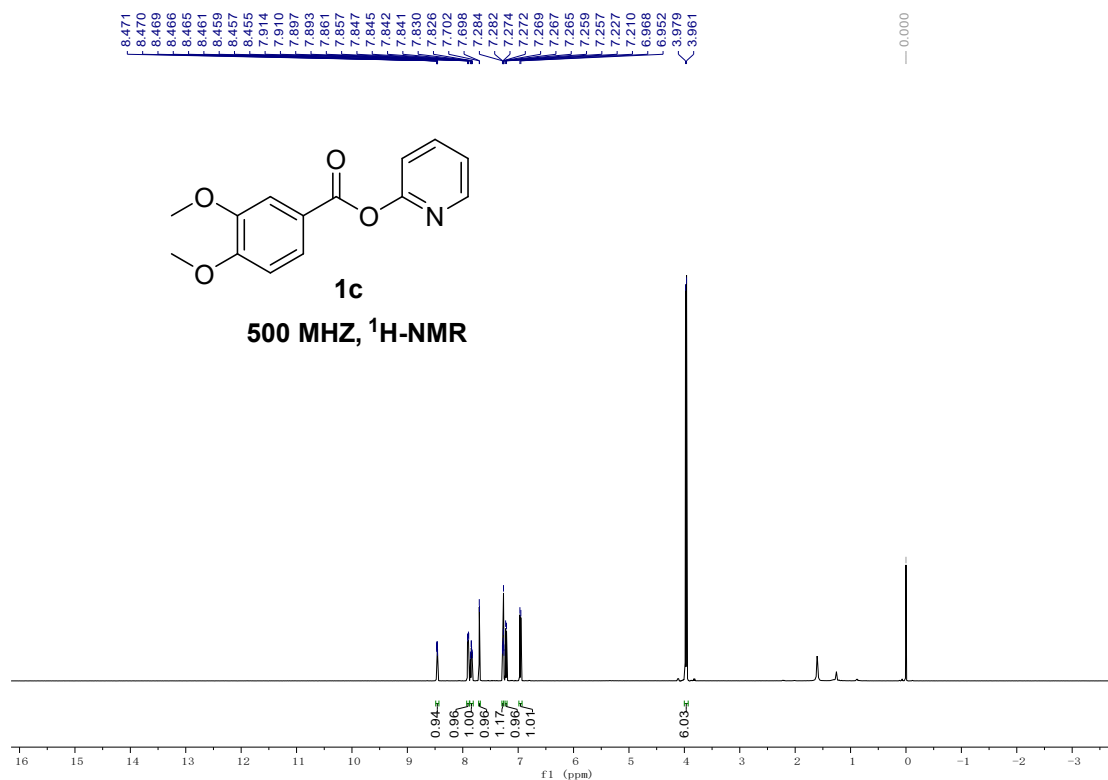
7. References

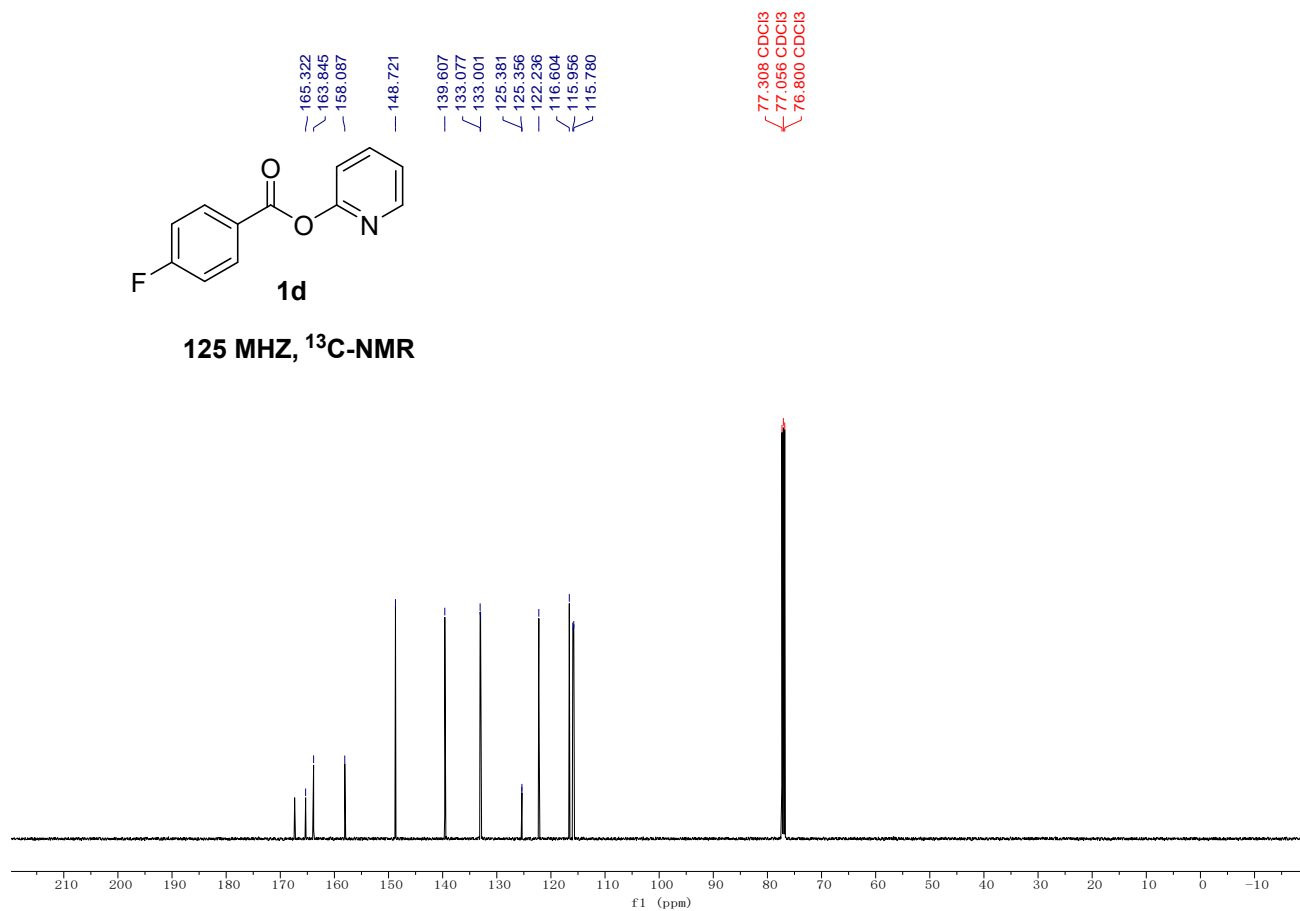
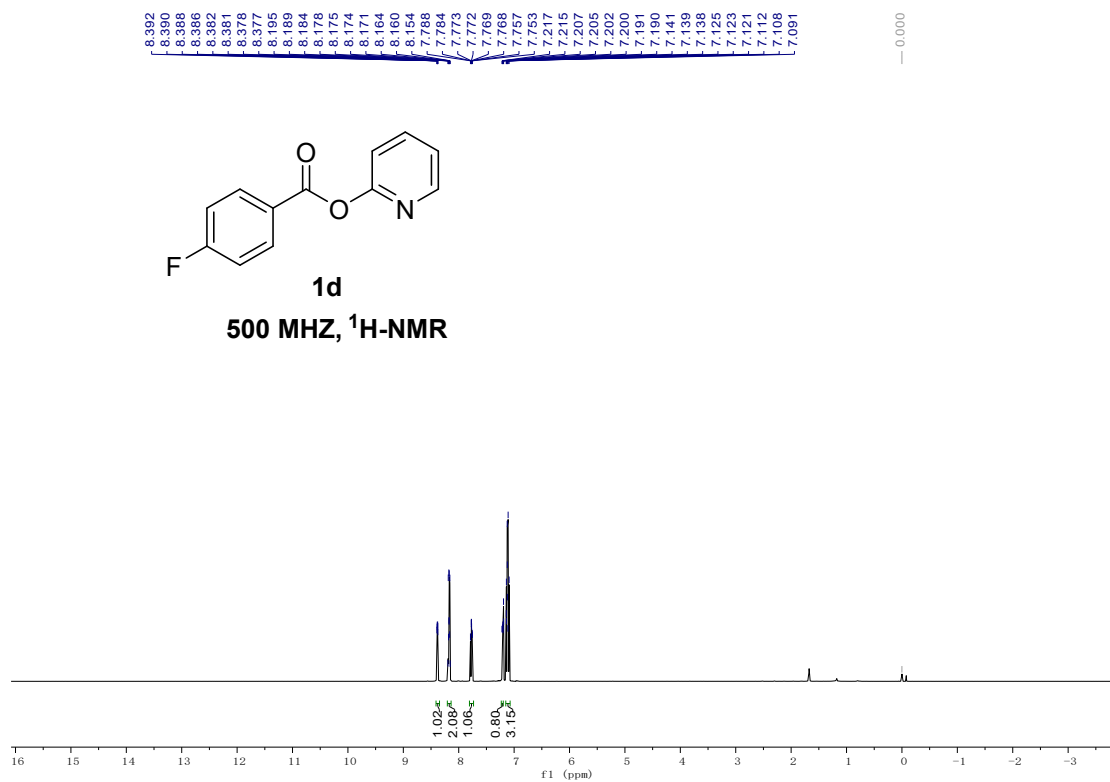
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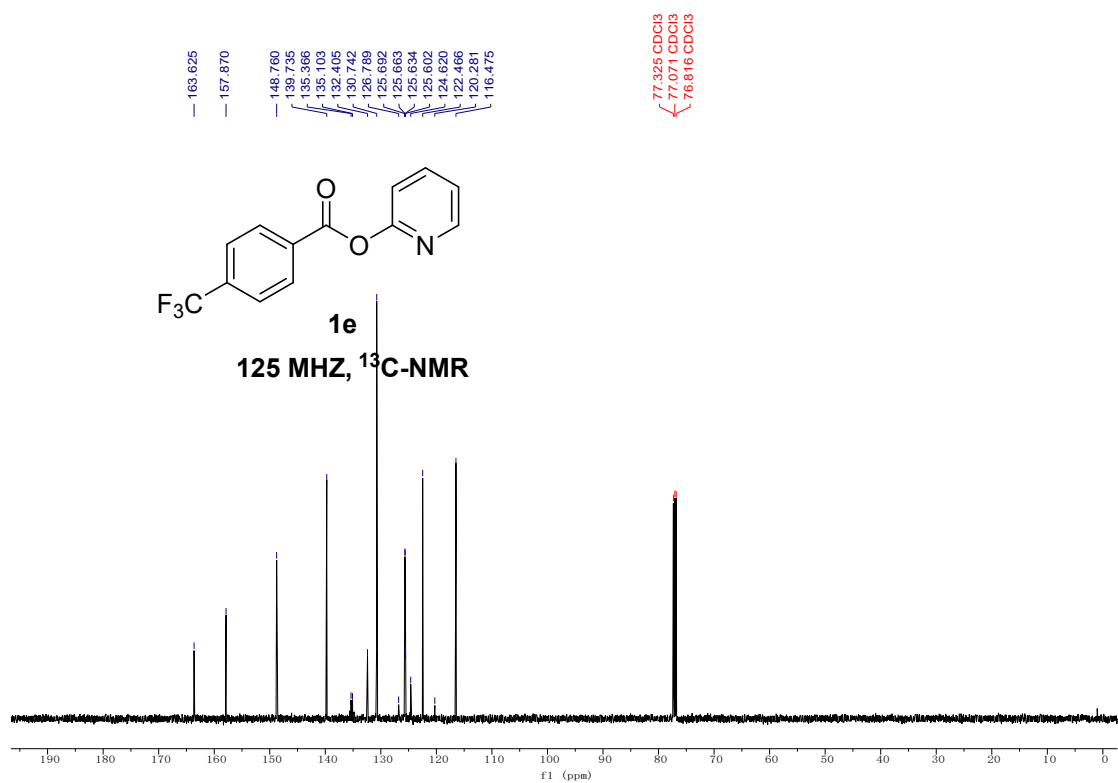
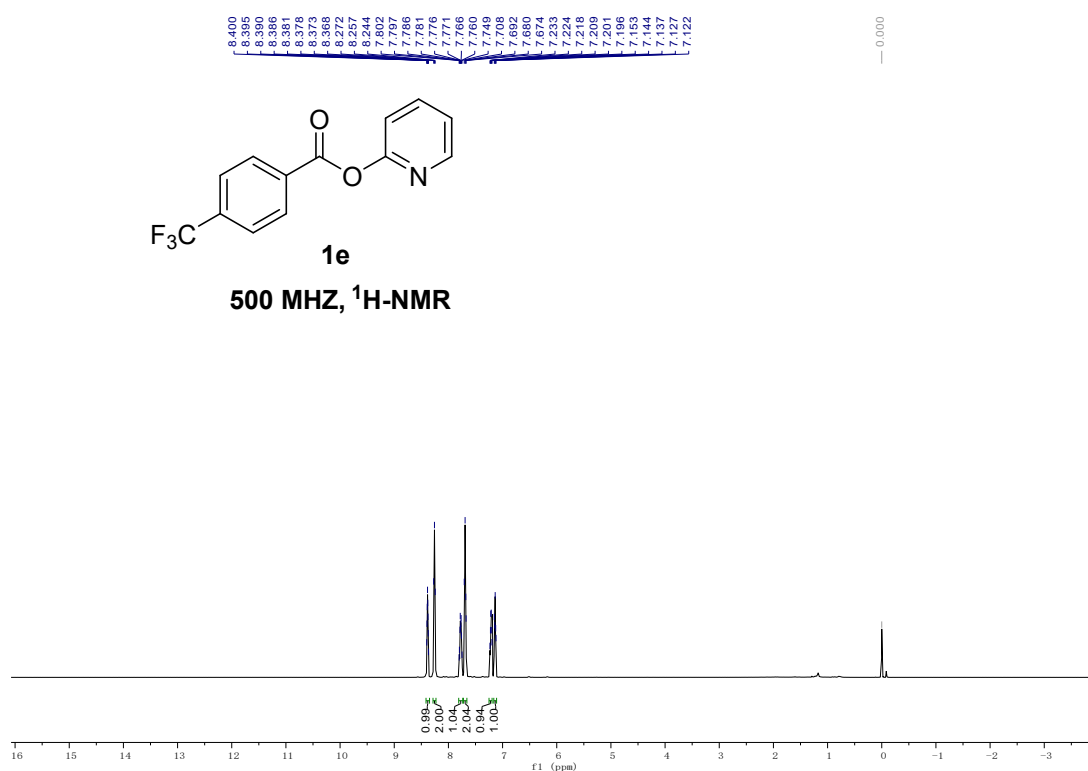
8. NMR Spectra

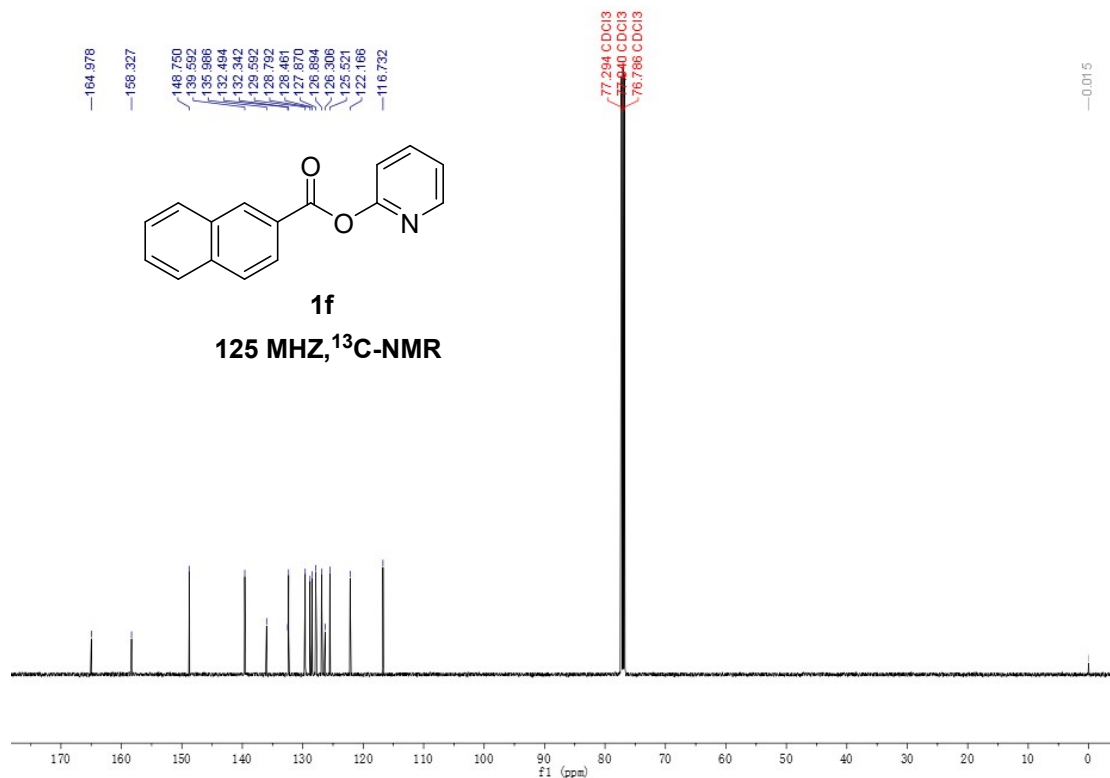
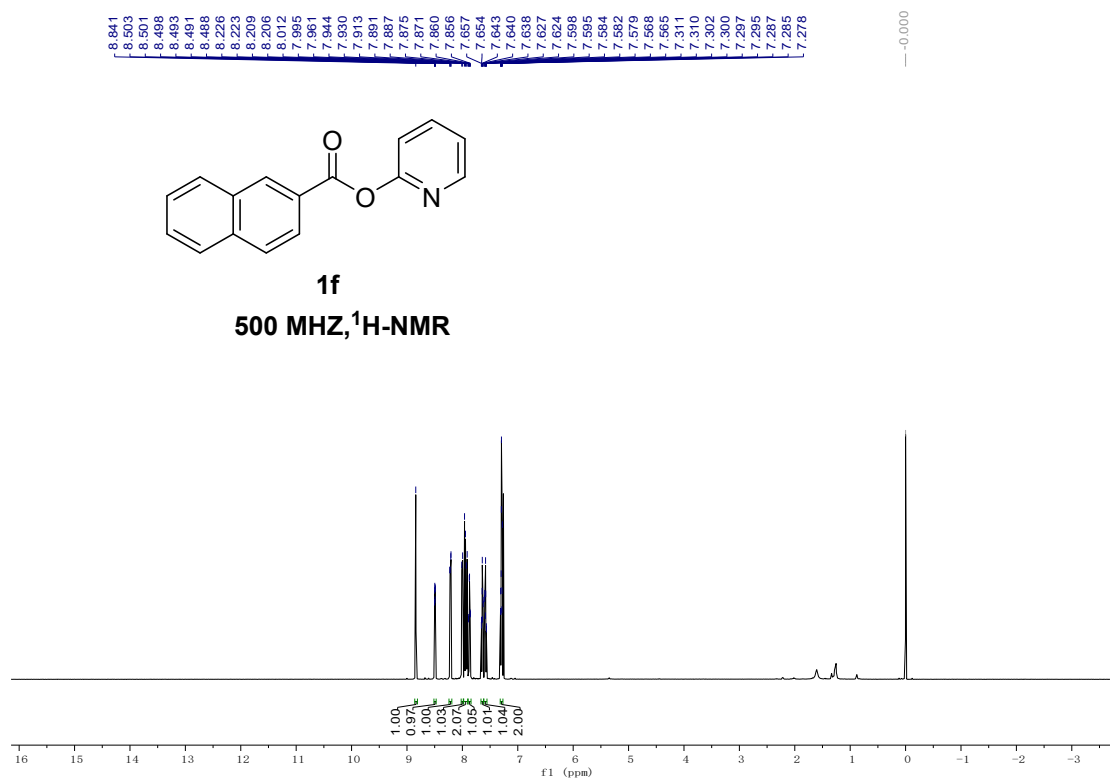


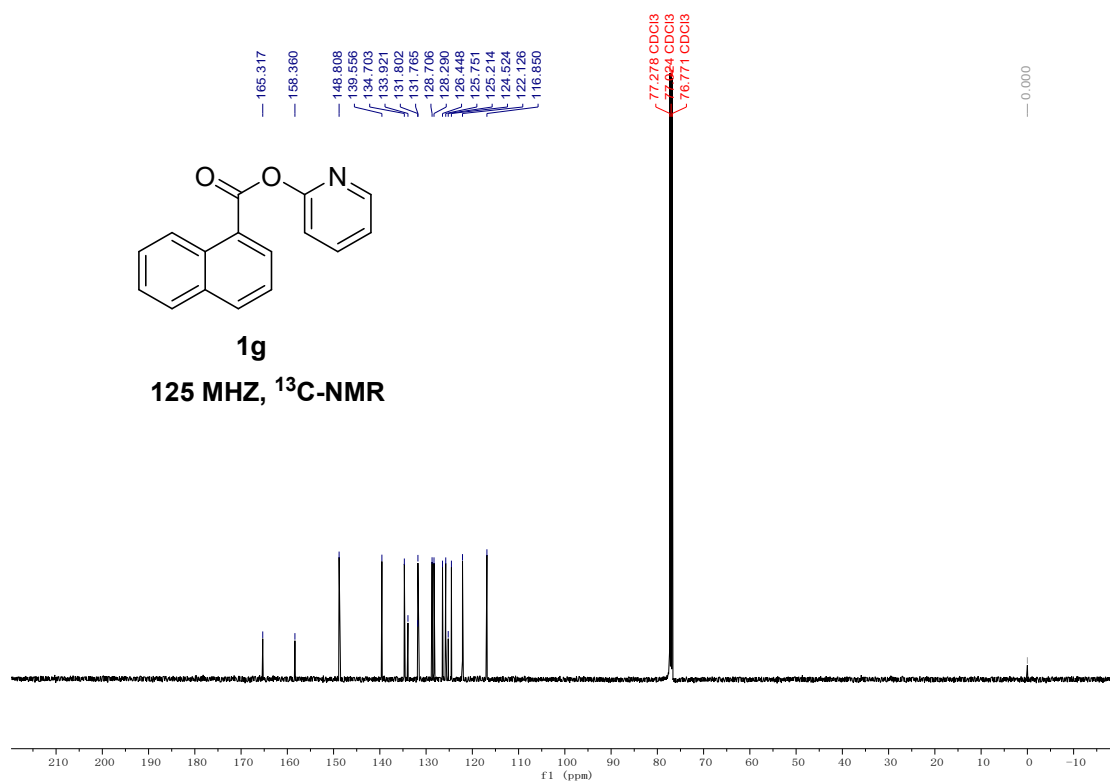
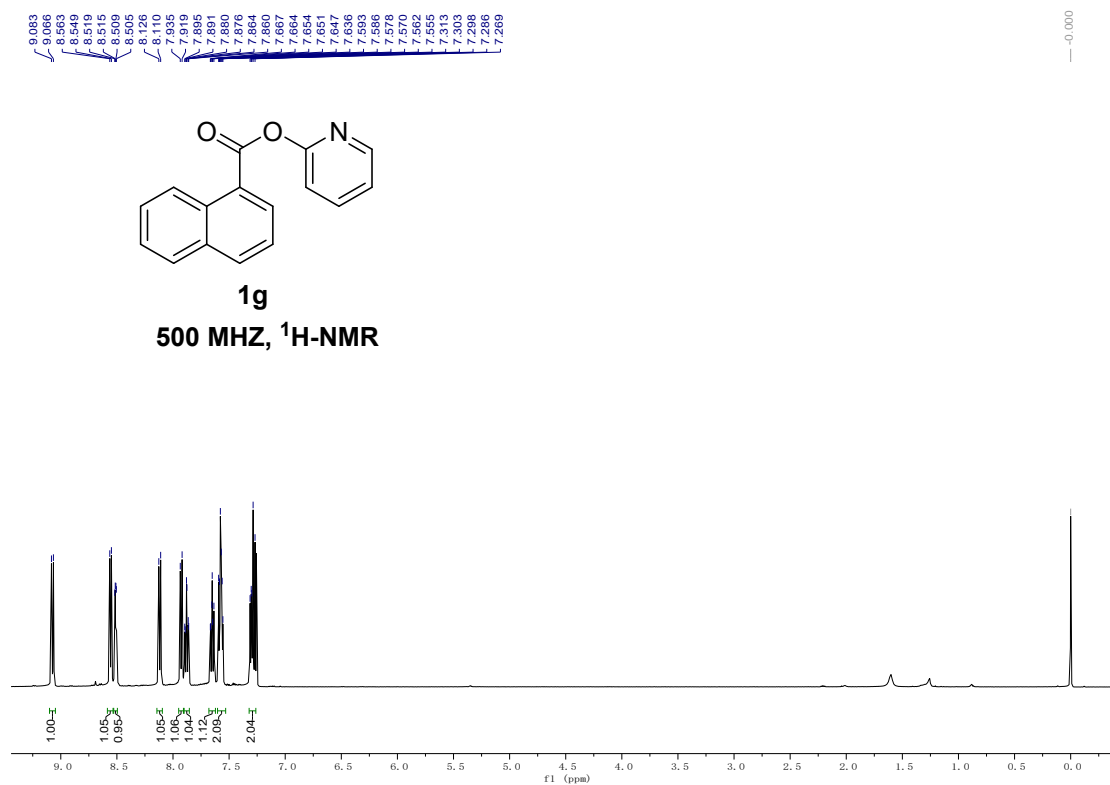


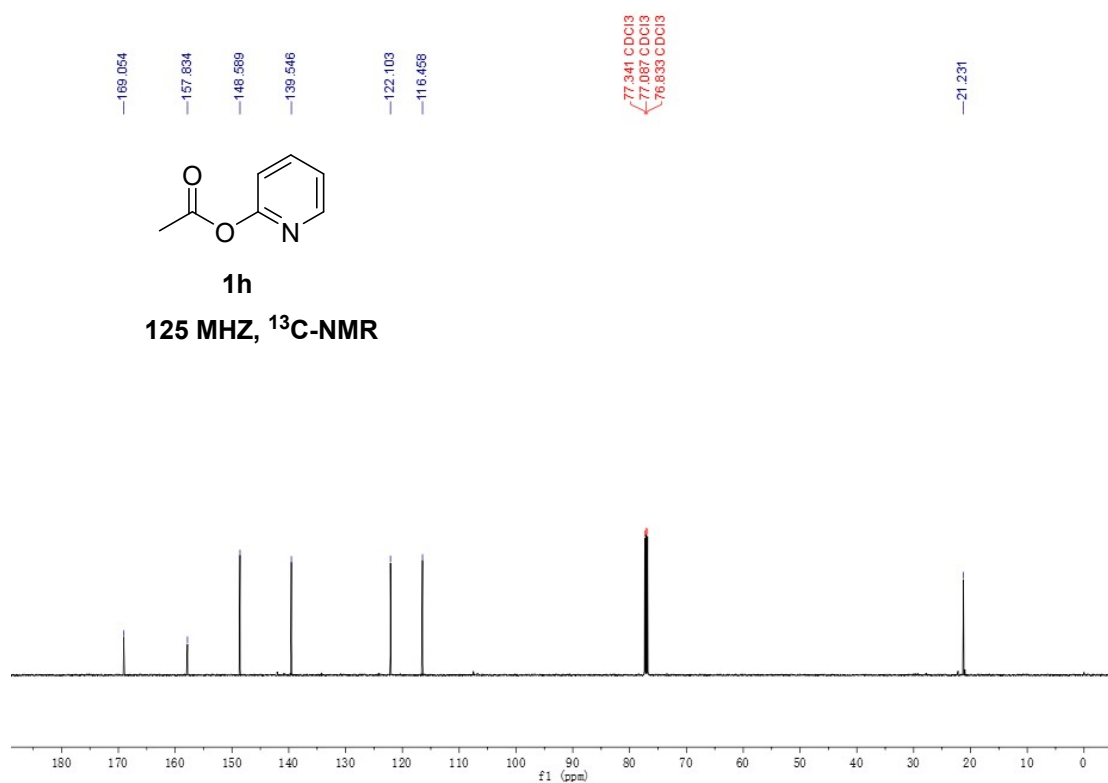
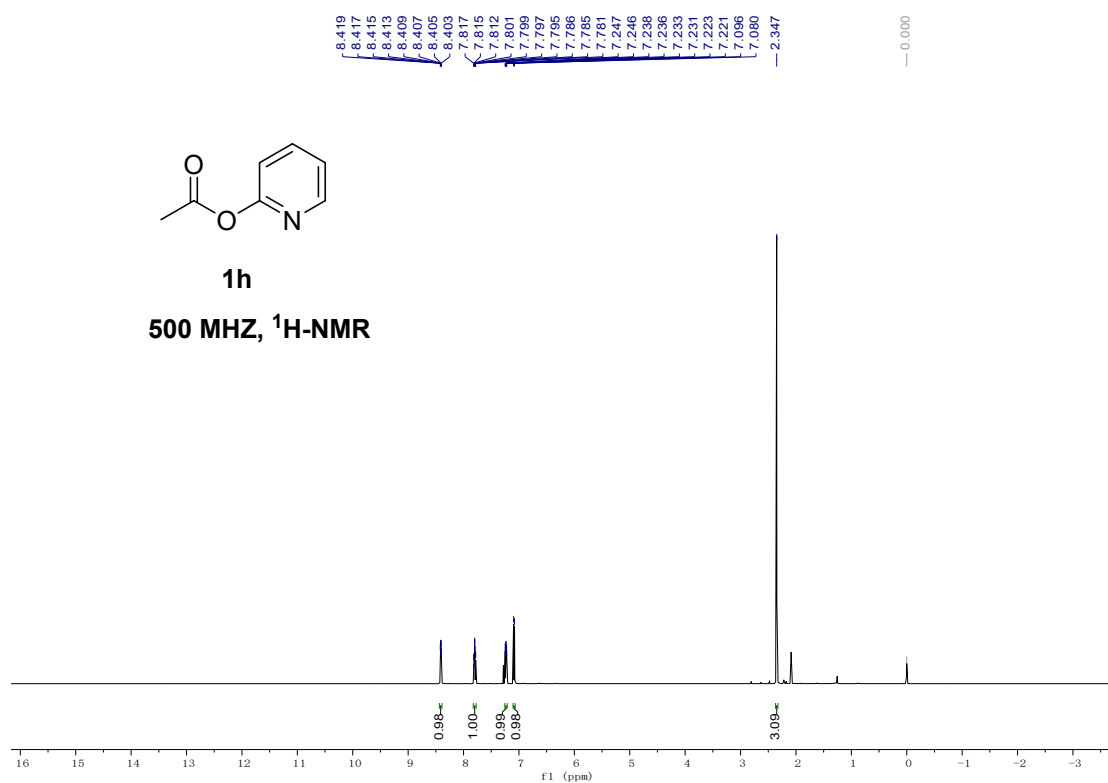










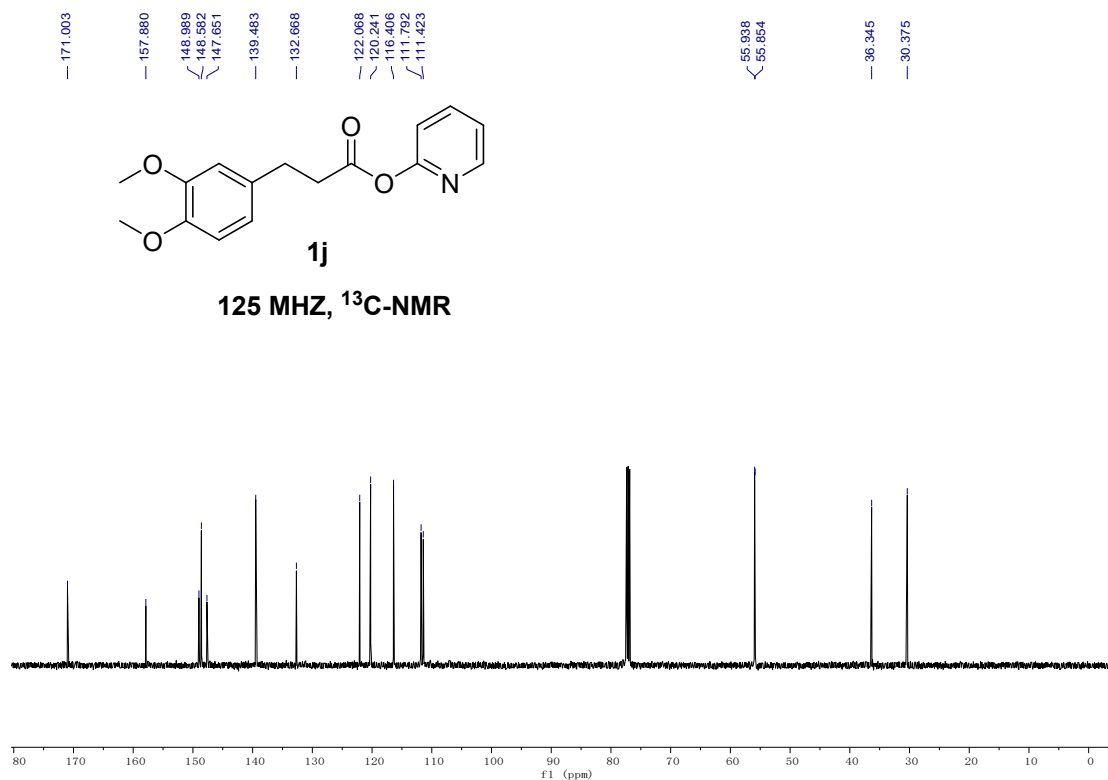
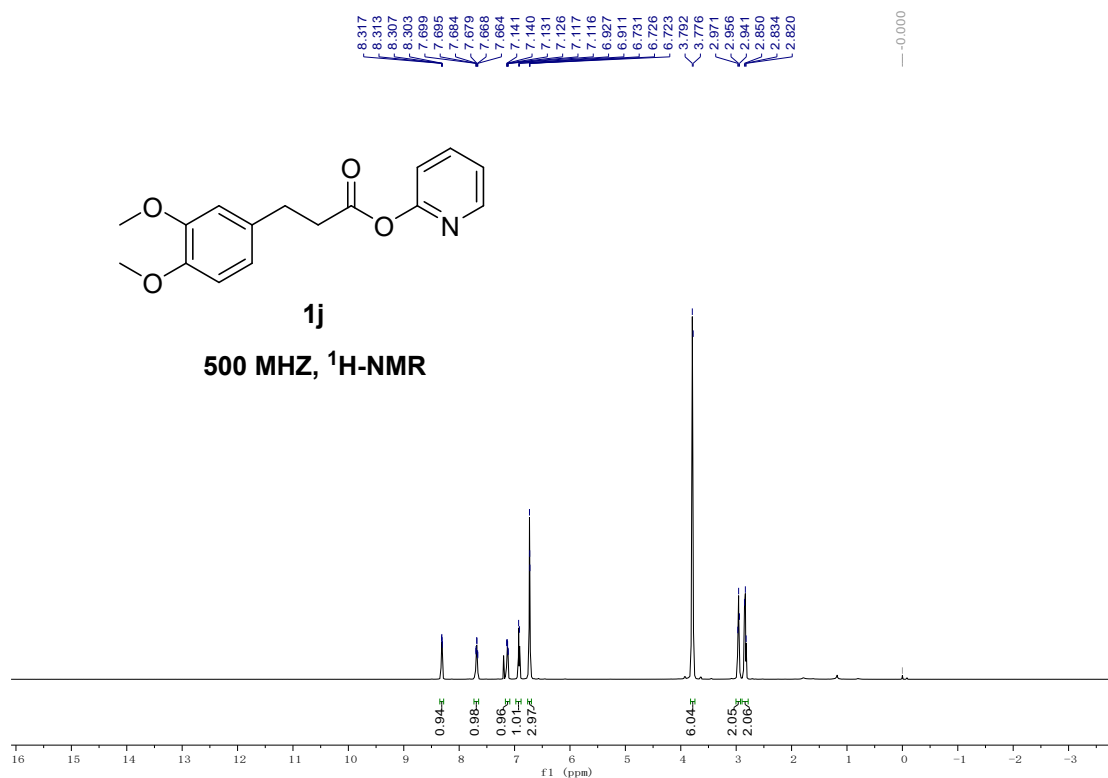


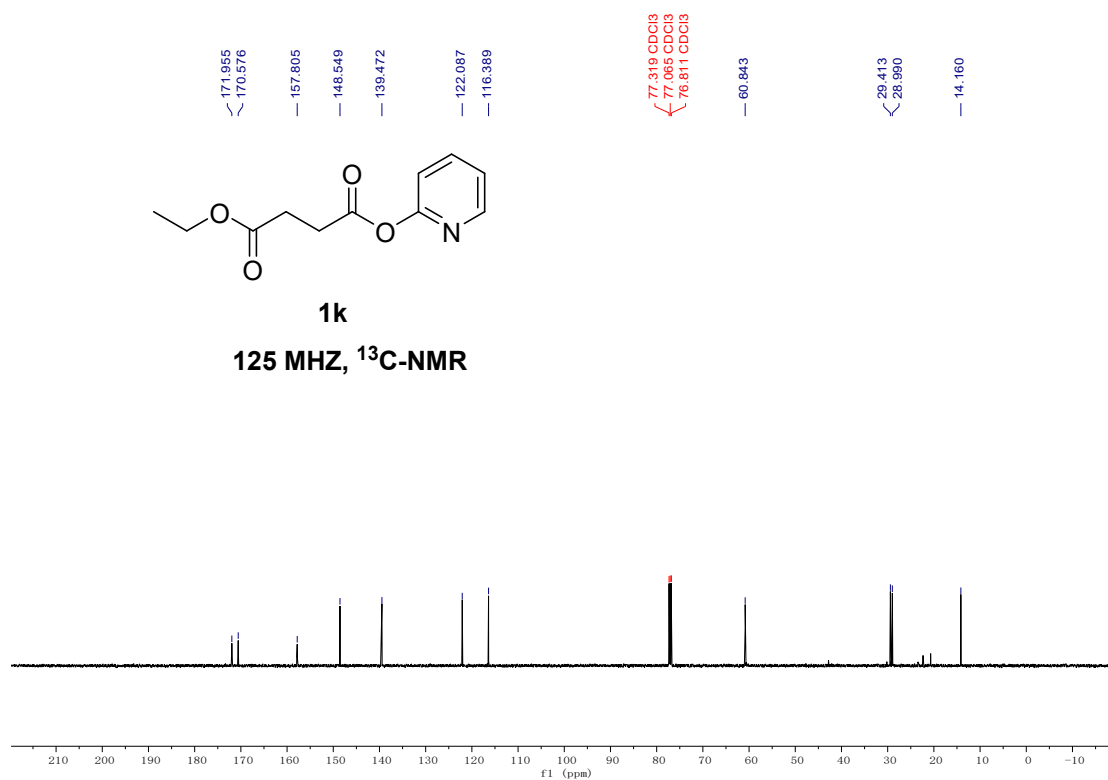
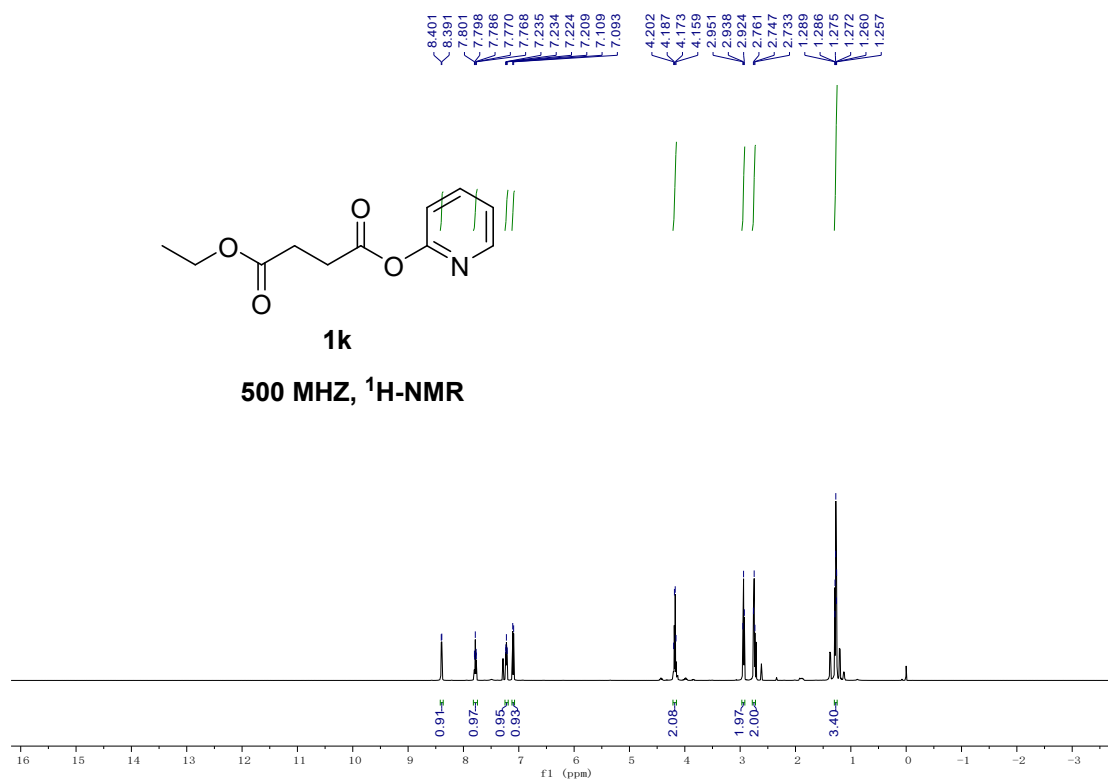


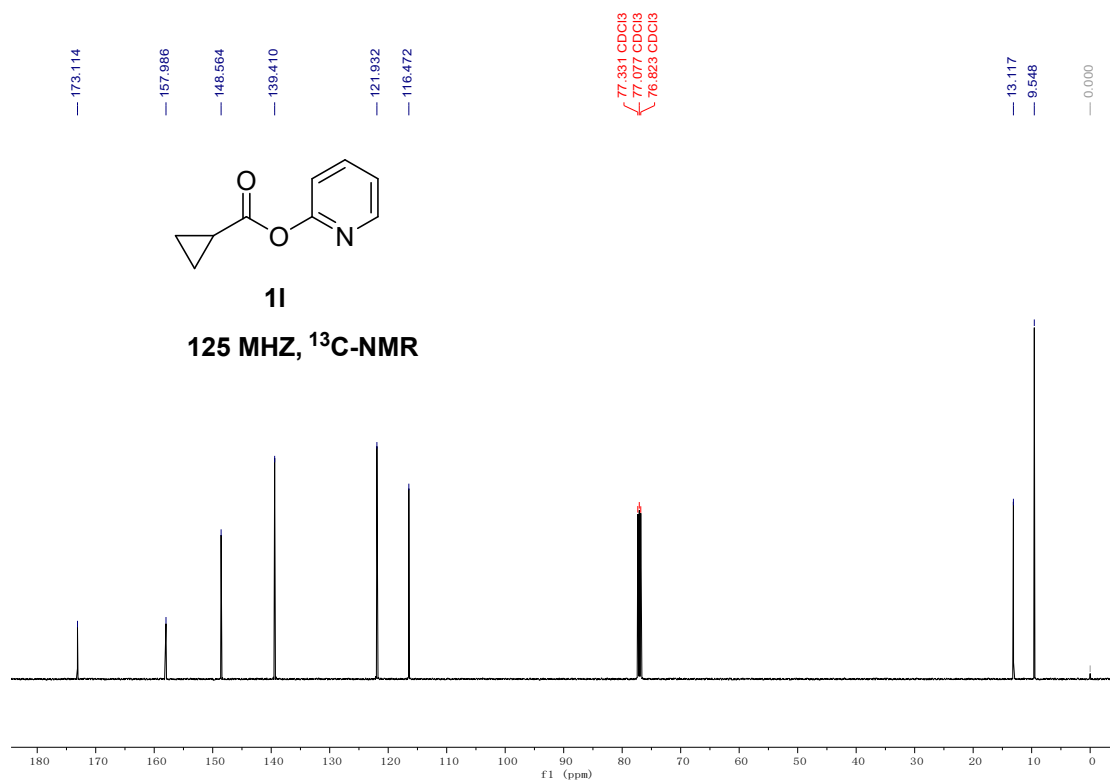
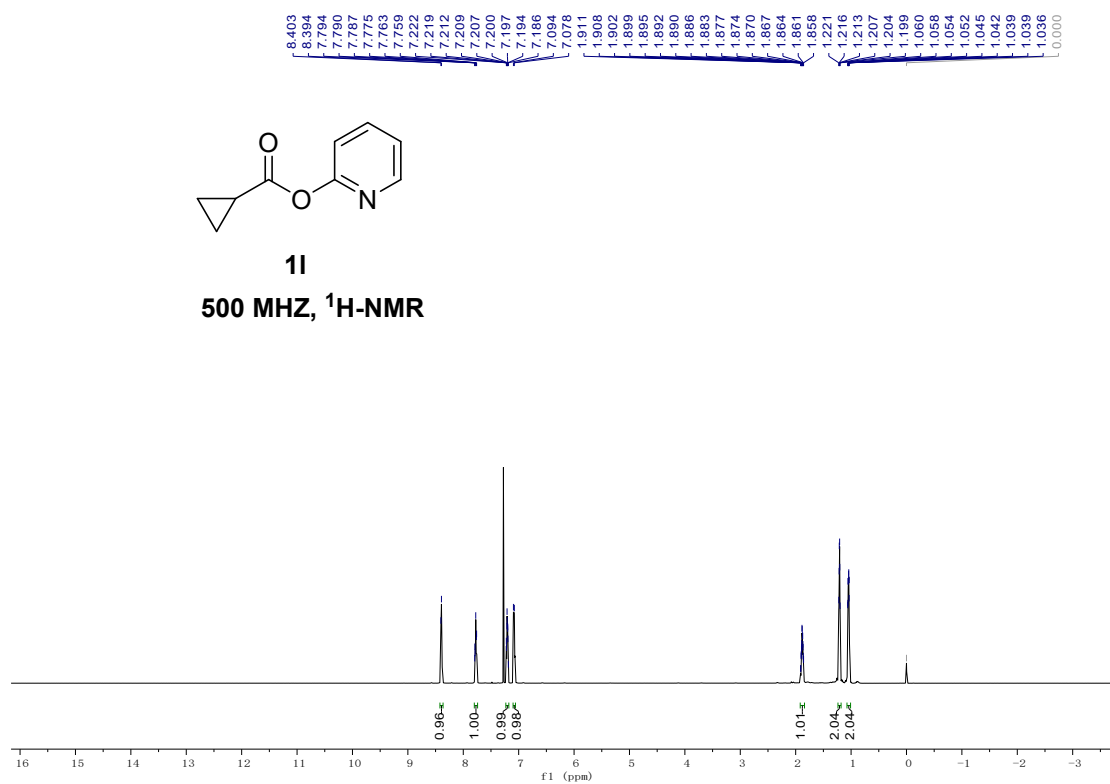
¹H NMR spectrum of compound 10 in CDCl₃. The x-axis is labeled 'f1 (ppm)' and ranges from 16 to -3. The spectrum shows several peaks with integration values indicated below them:

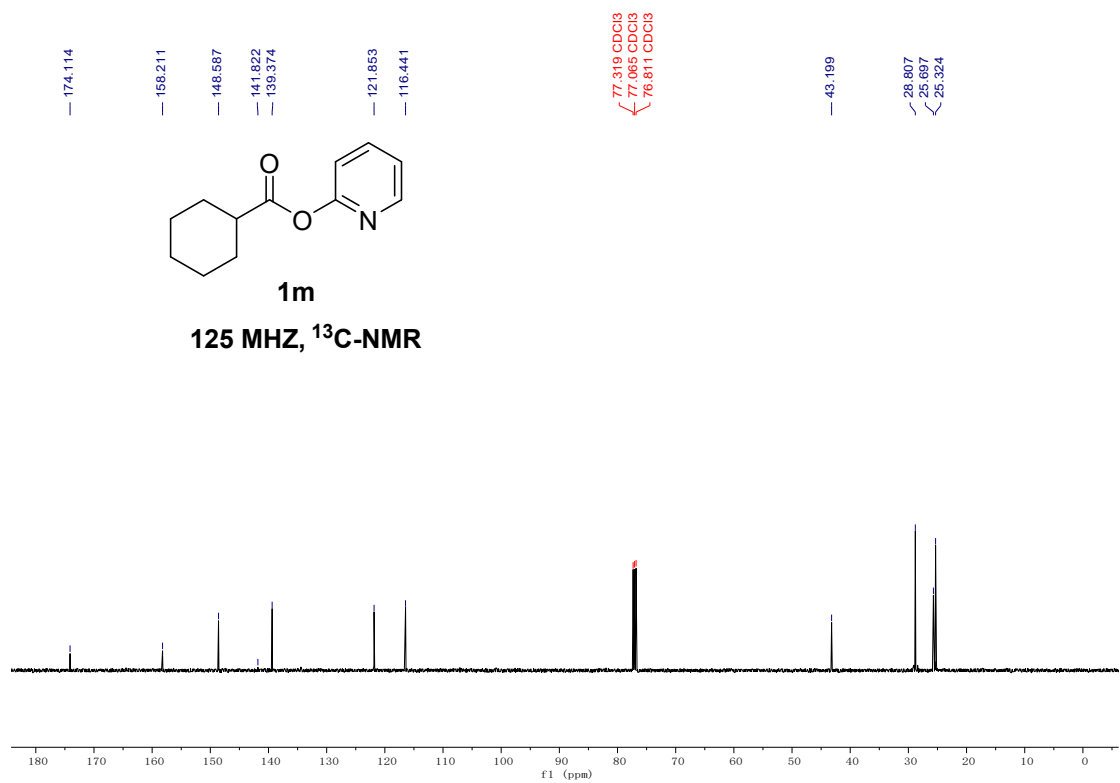
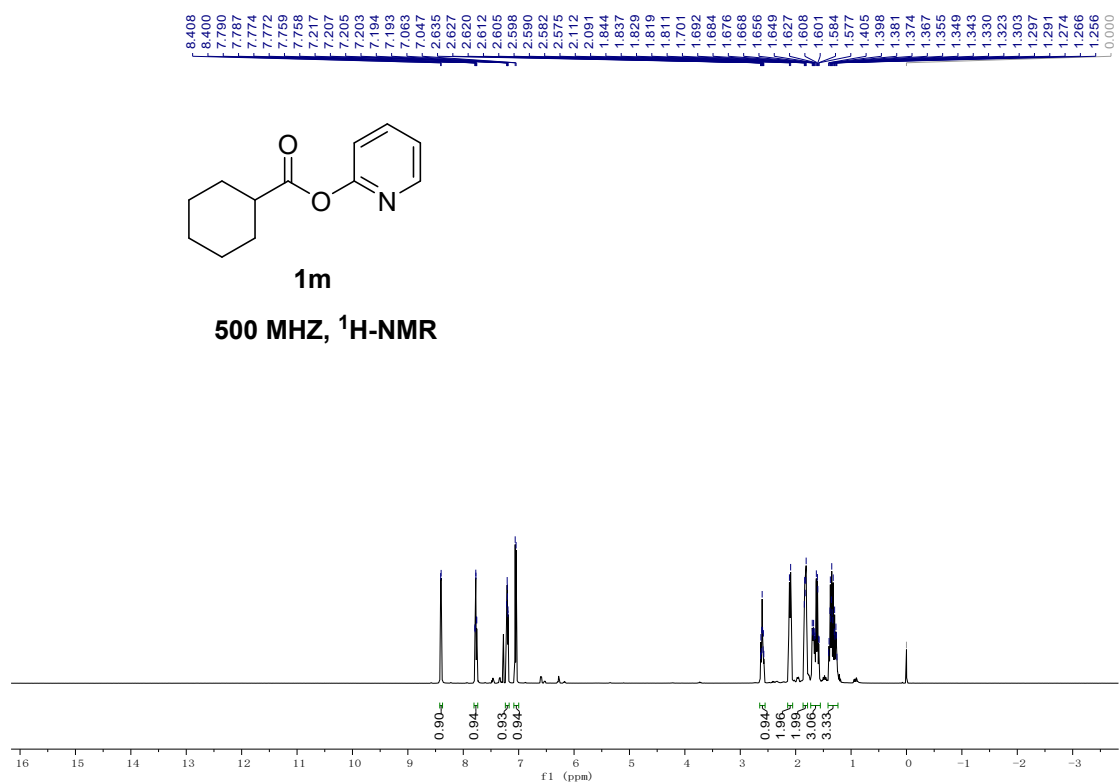
- Peak at ~8.5 ppm: 0.94H
- Peak at ~7.8 ppm: 0.95H
- Peak at ~7.6 ppm: 2.12H
- Peak at ~7.4 ppm: 3.86H
- Peak at ~7.2 ppm: 0.95H
- Peak at ~6.5 ppm: (small peak)
- Peak at ~3.0 ppm: 2.01H
- Peak at ~2.8 ppm: 1.98H
- Peak at ~2.6 ppm: 2.09H
- Peak at ~0.1 ppm: (small peak)

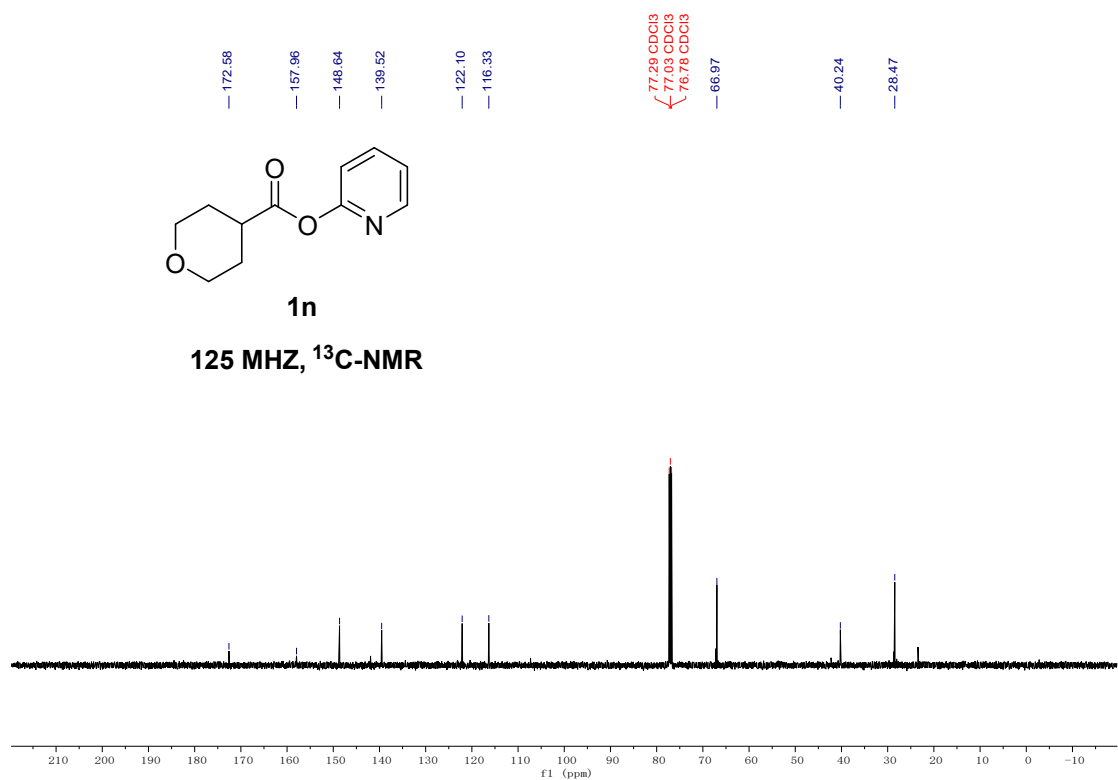
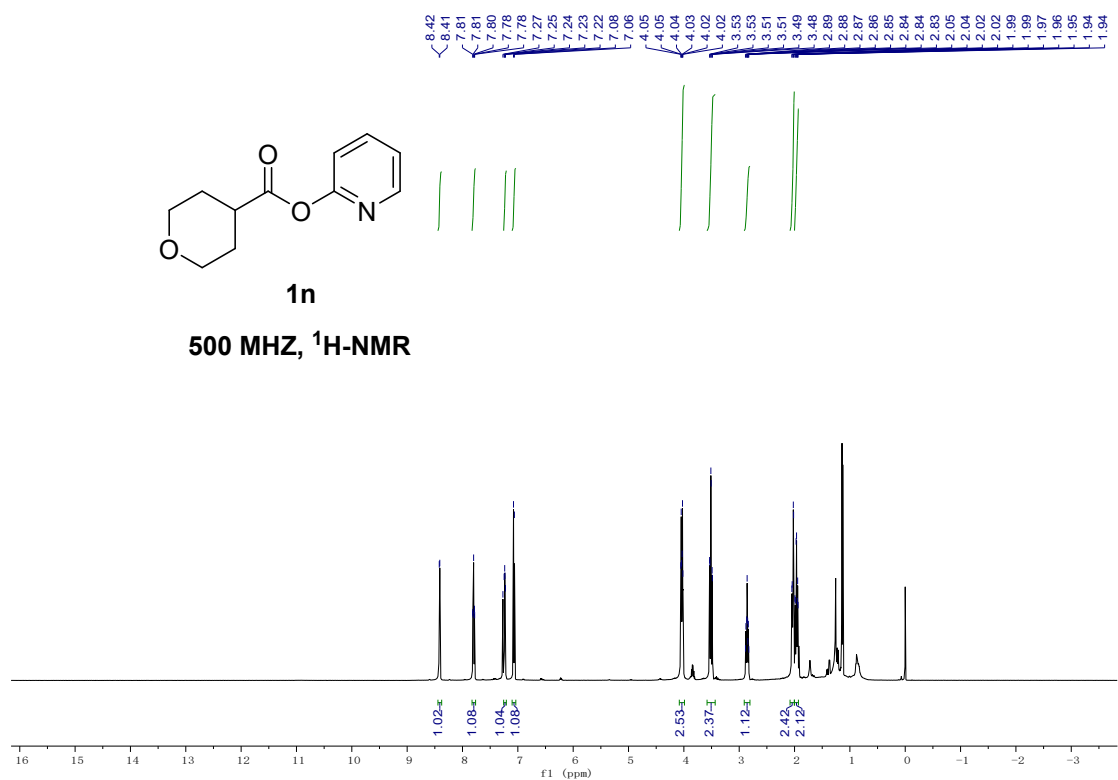


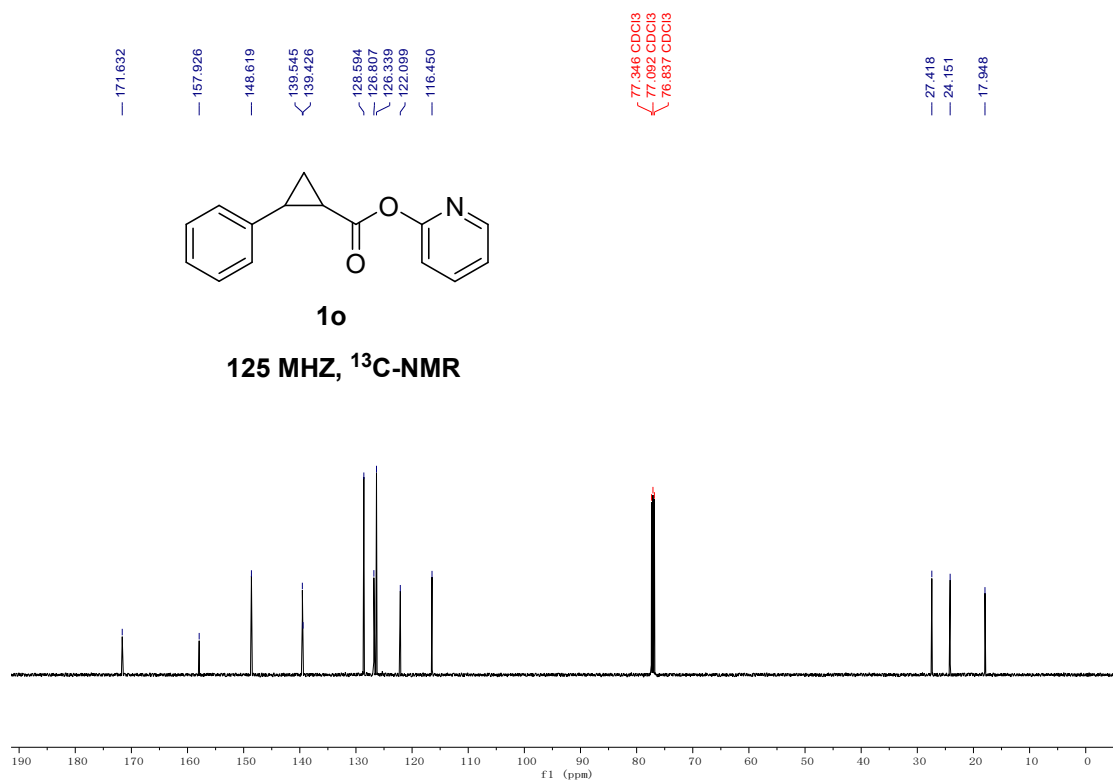
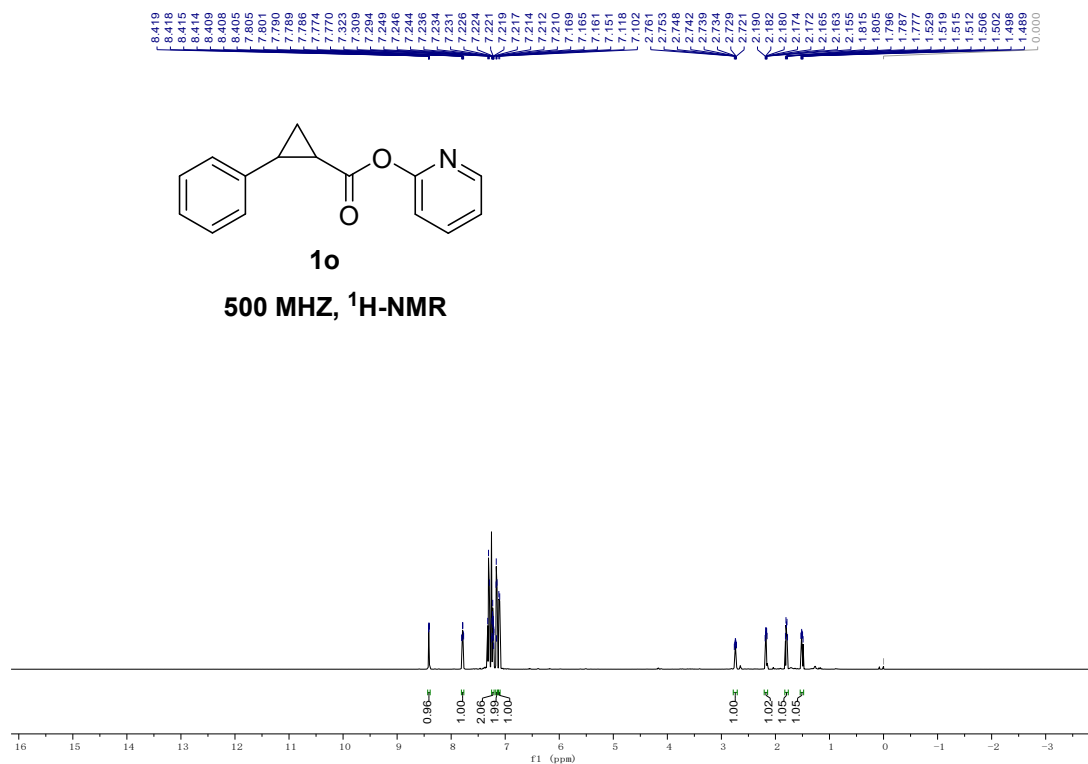


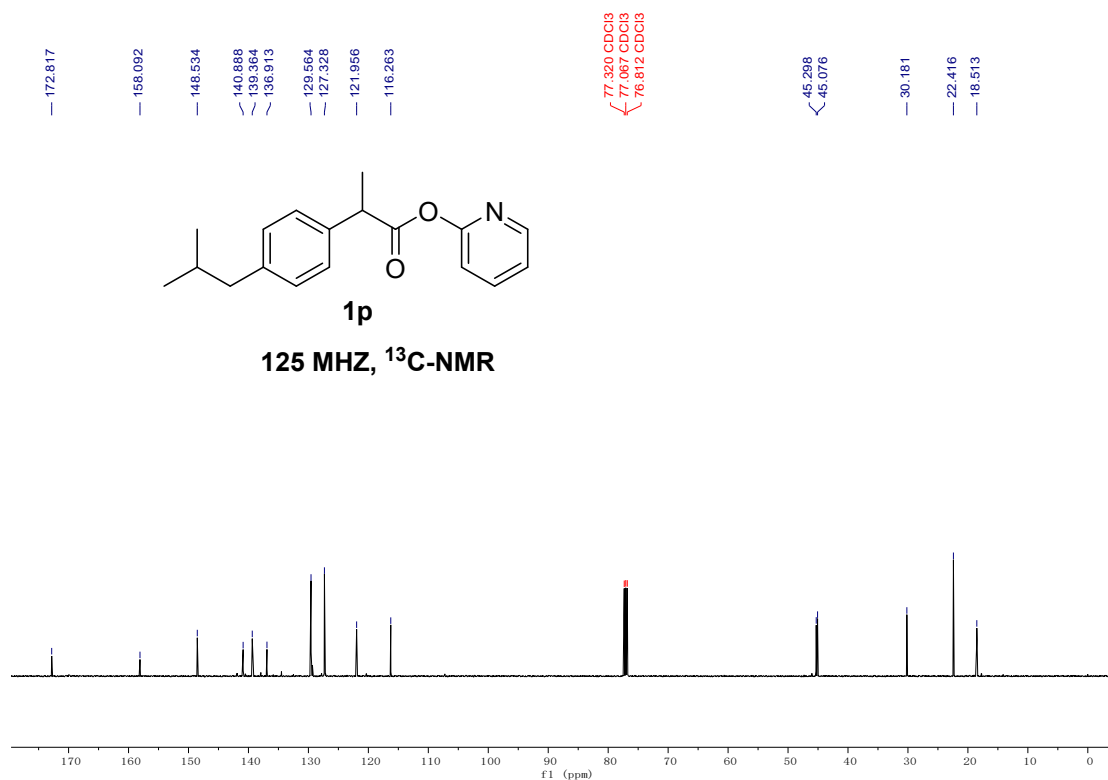
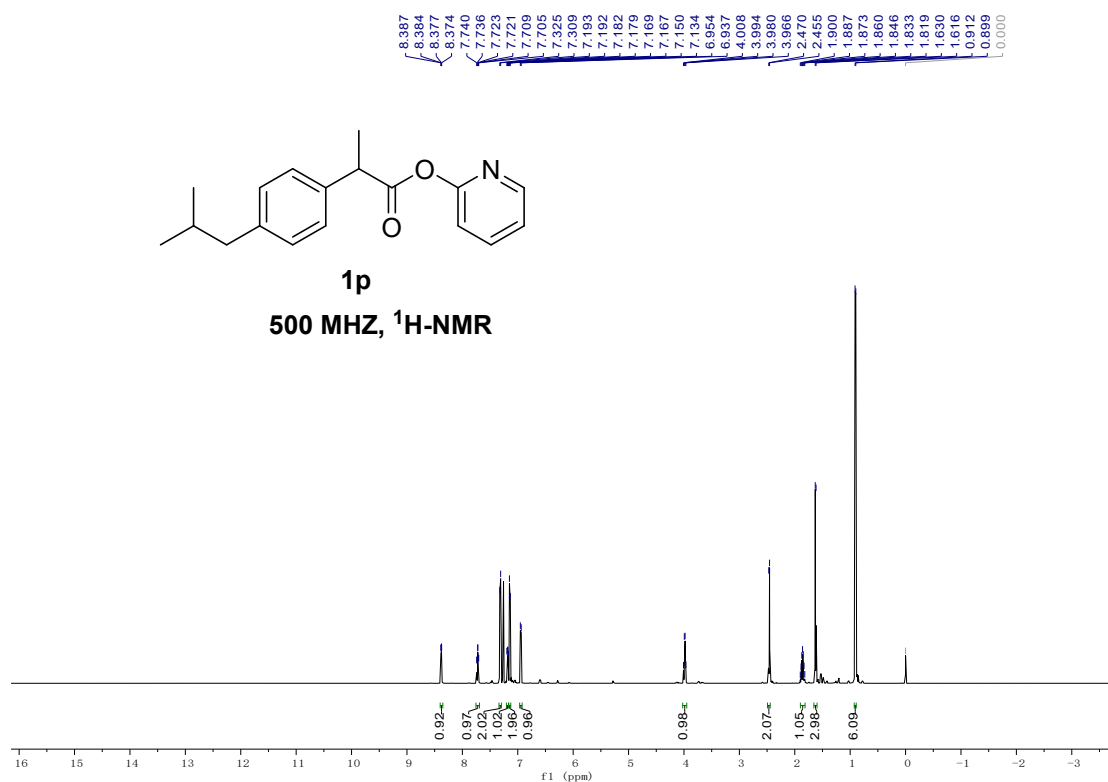


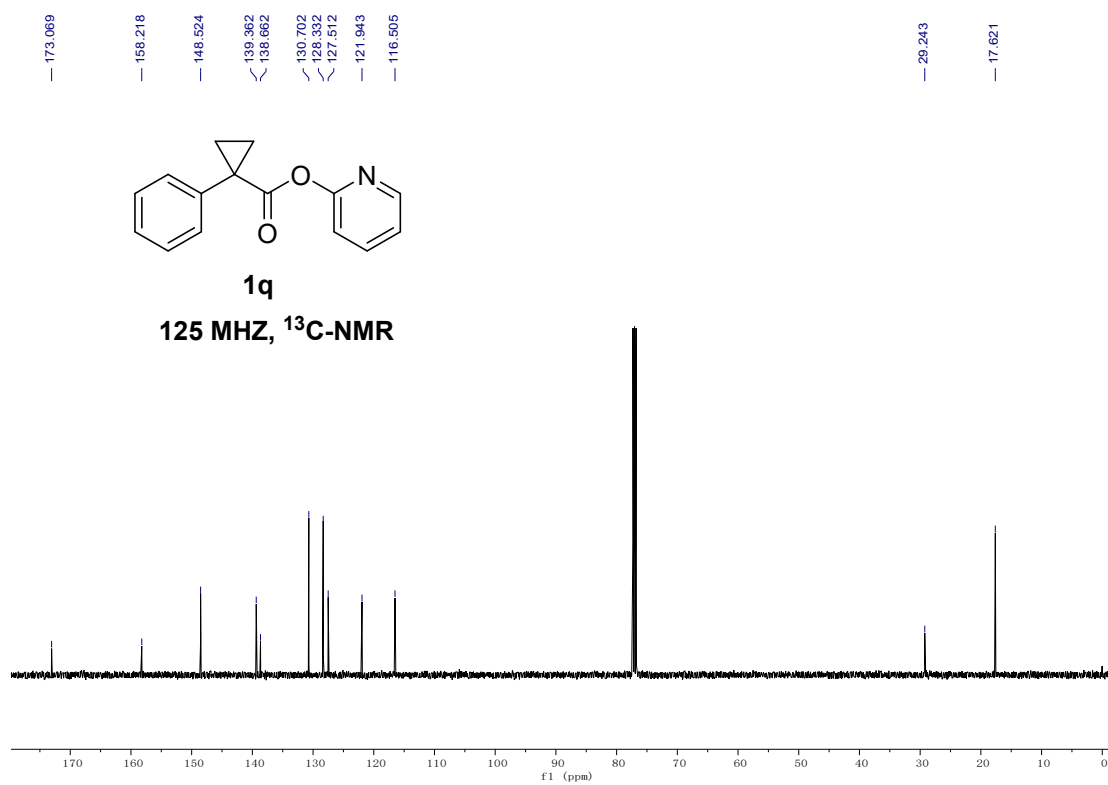
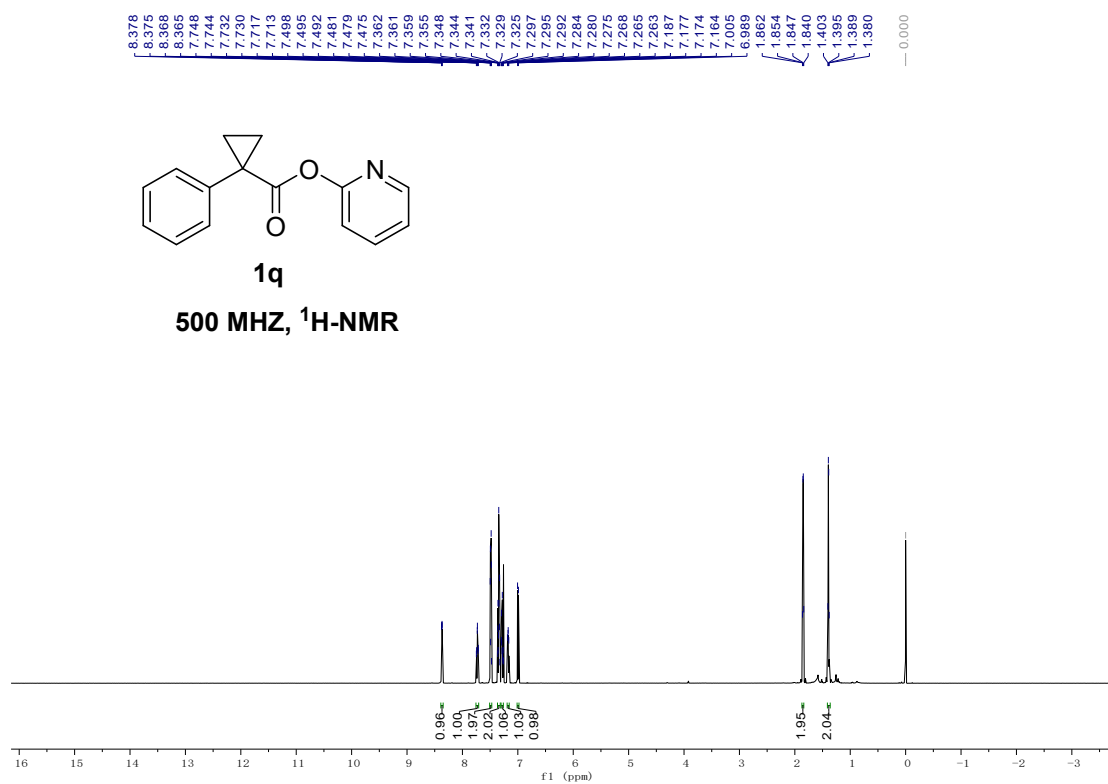


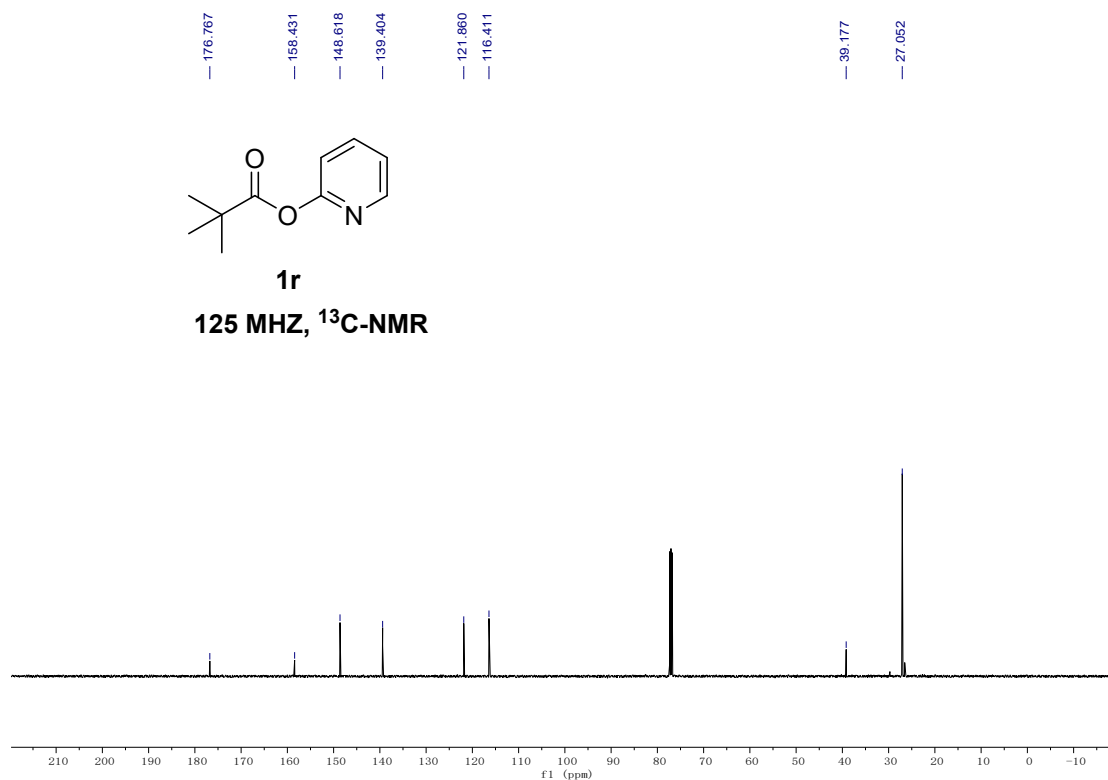
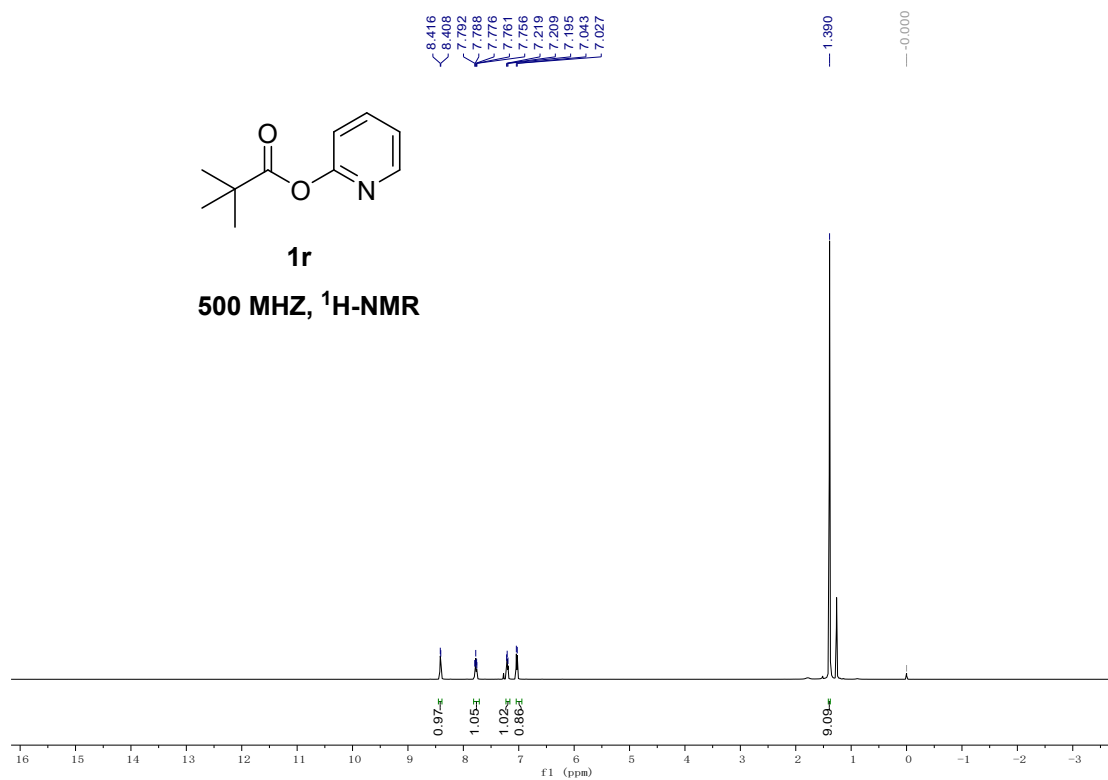


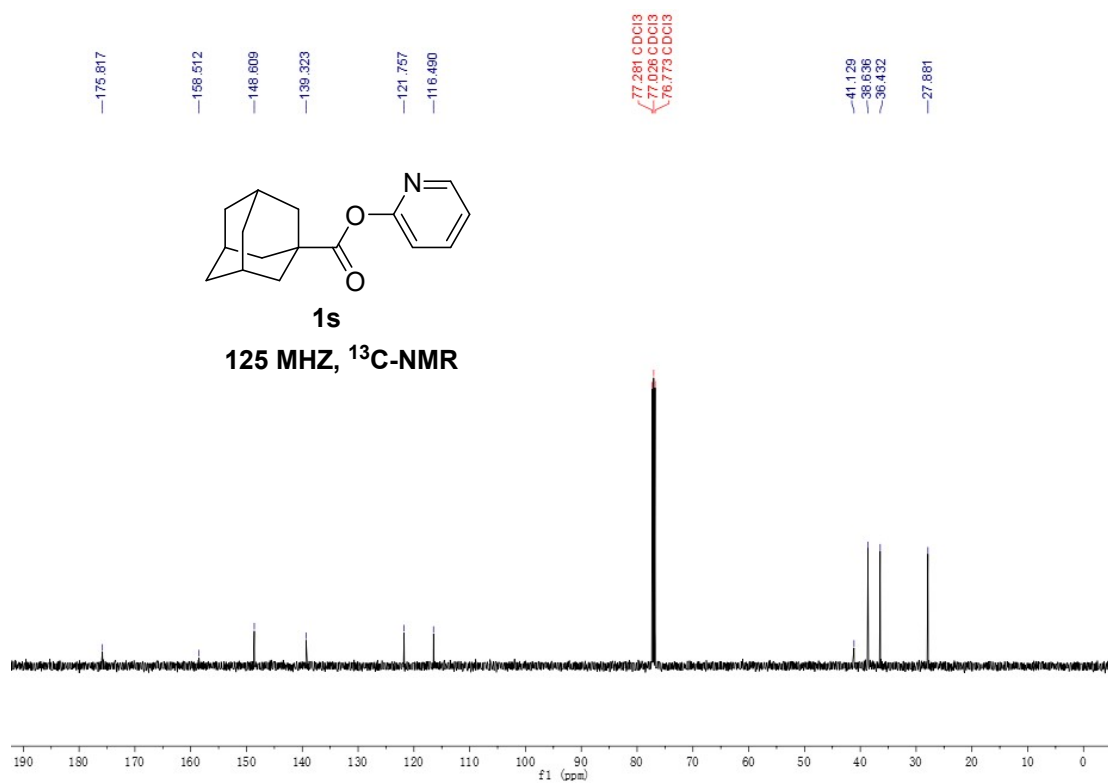
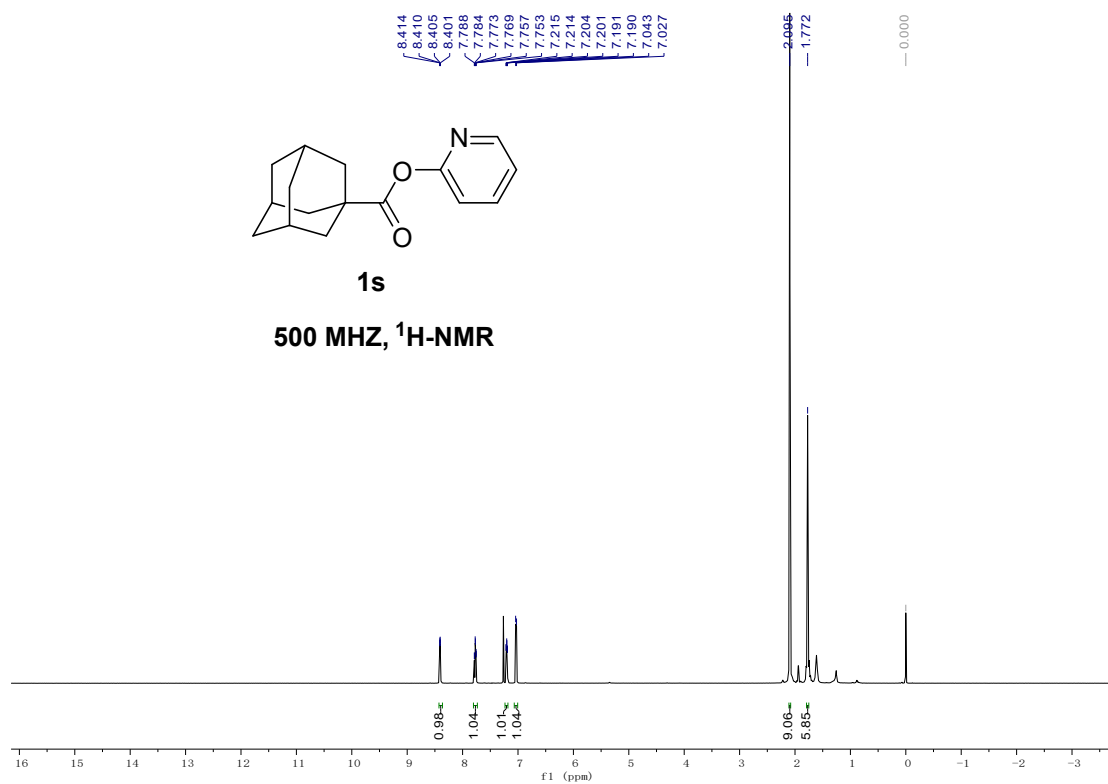


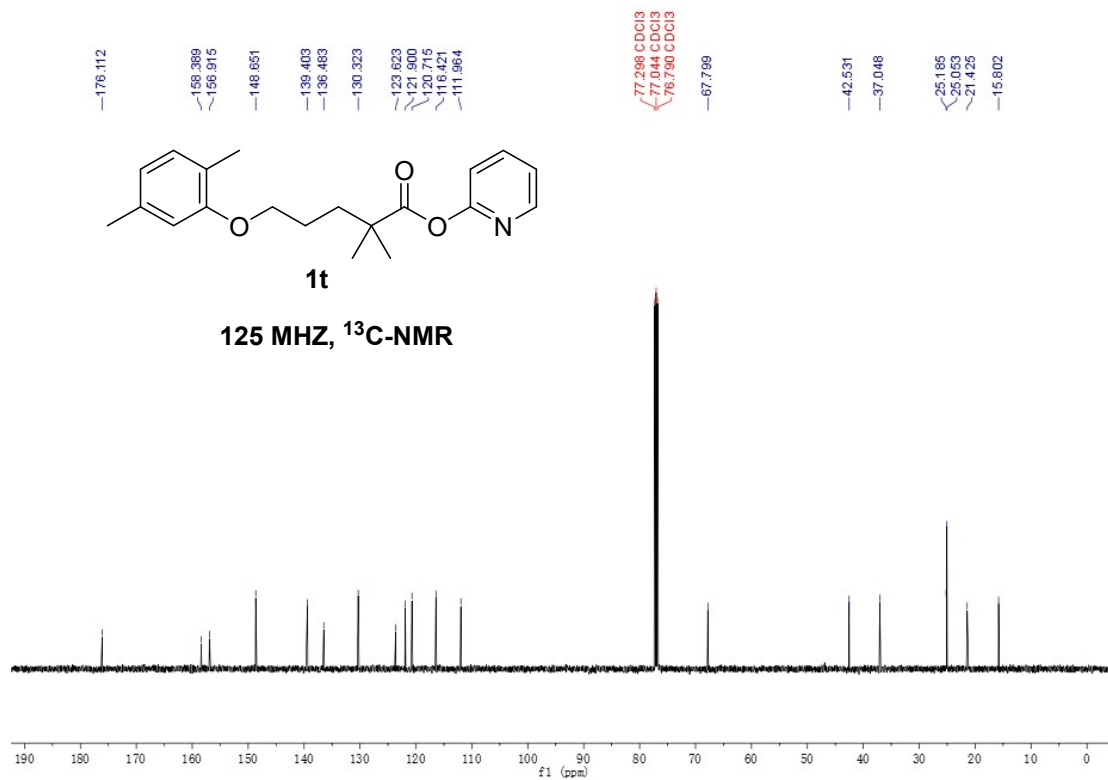
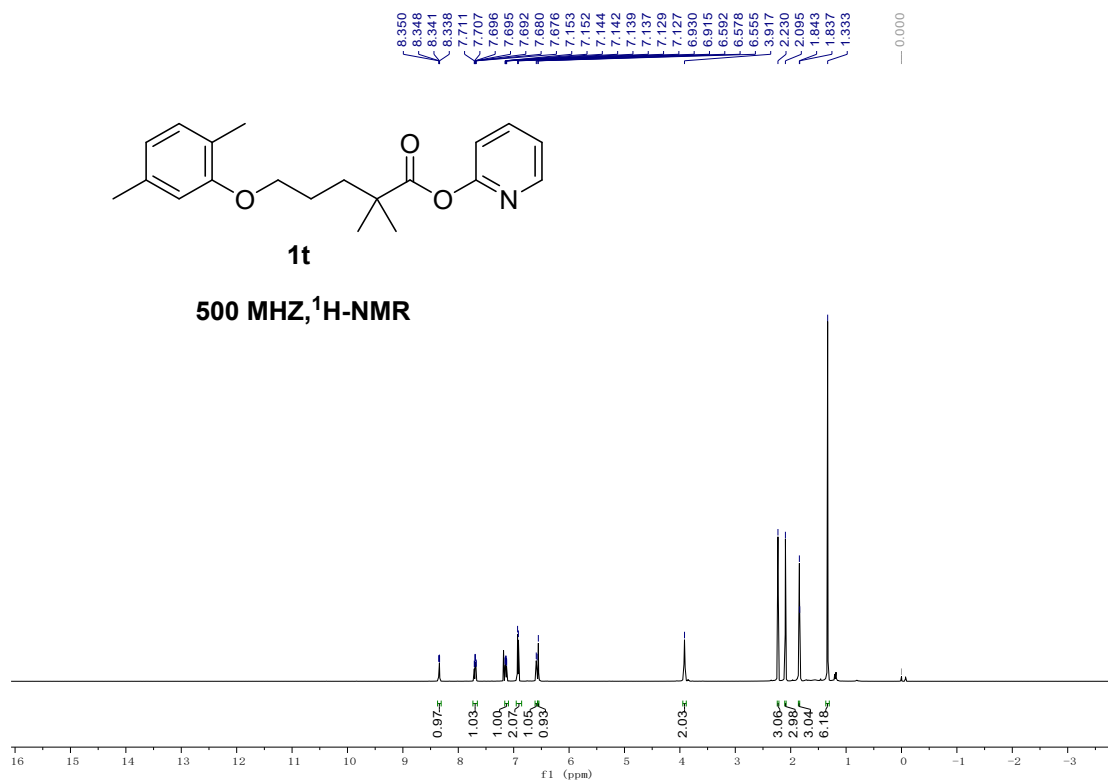


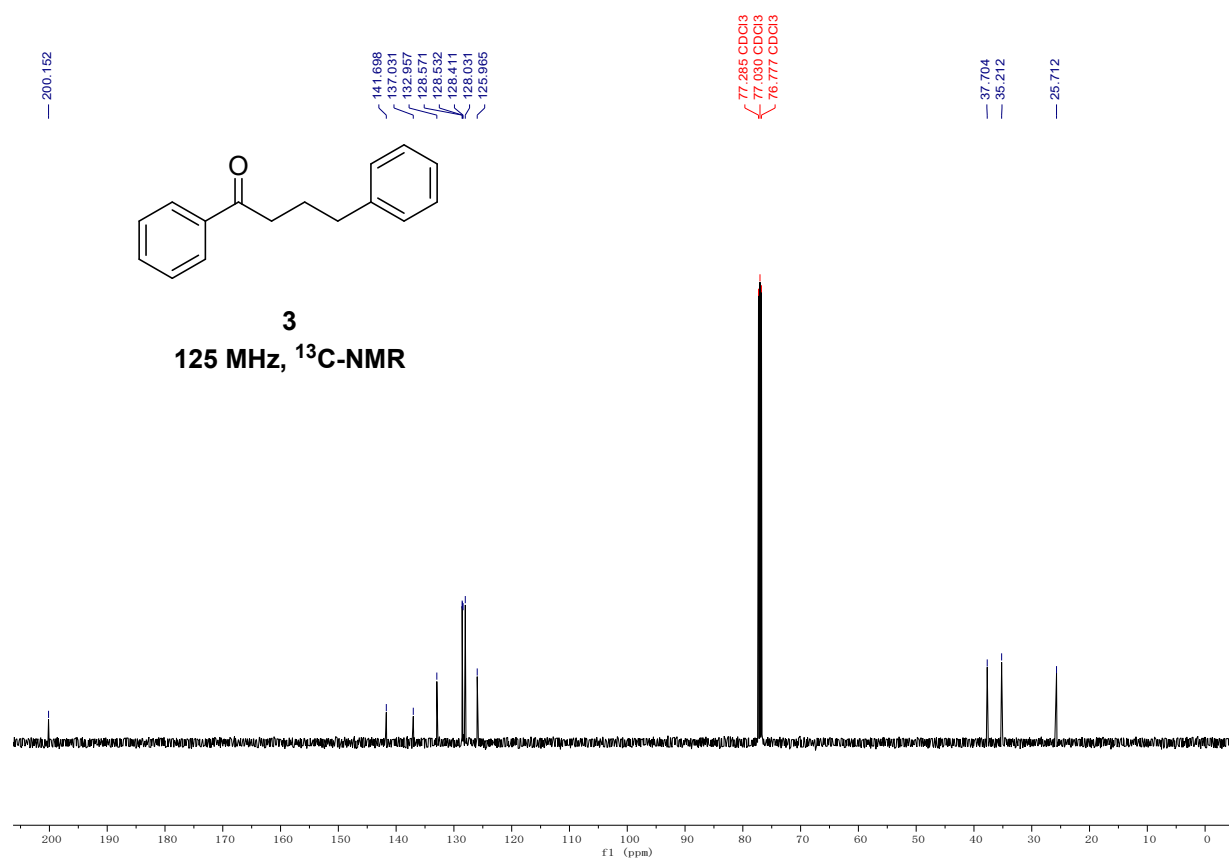
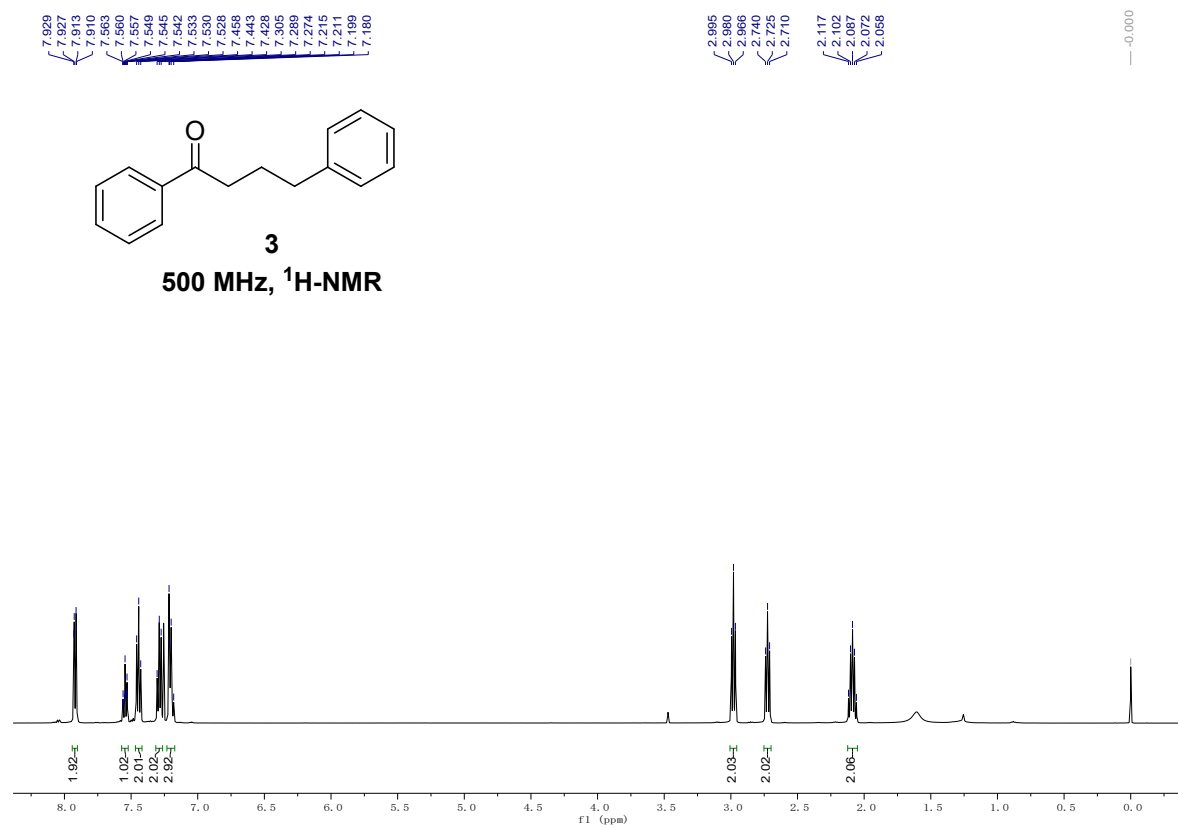


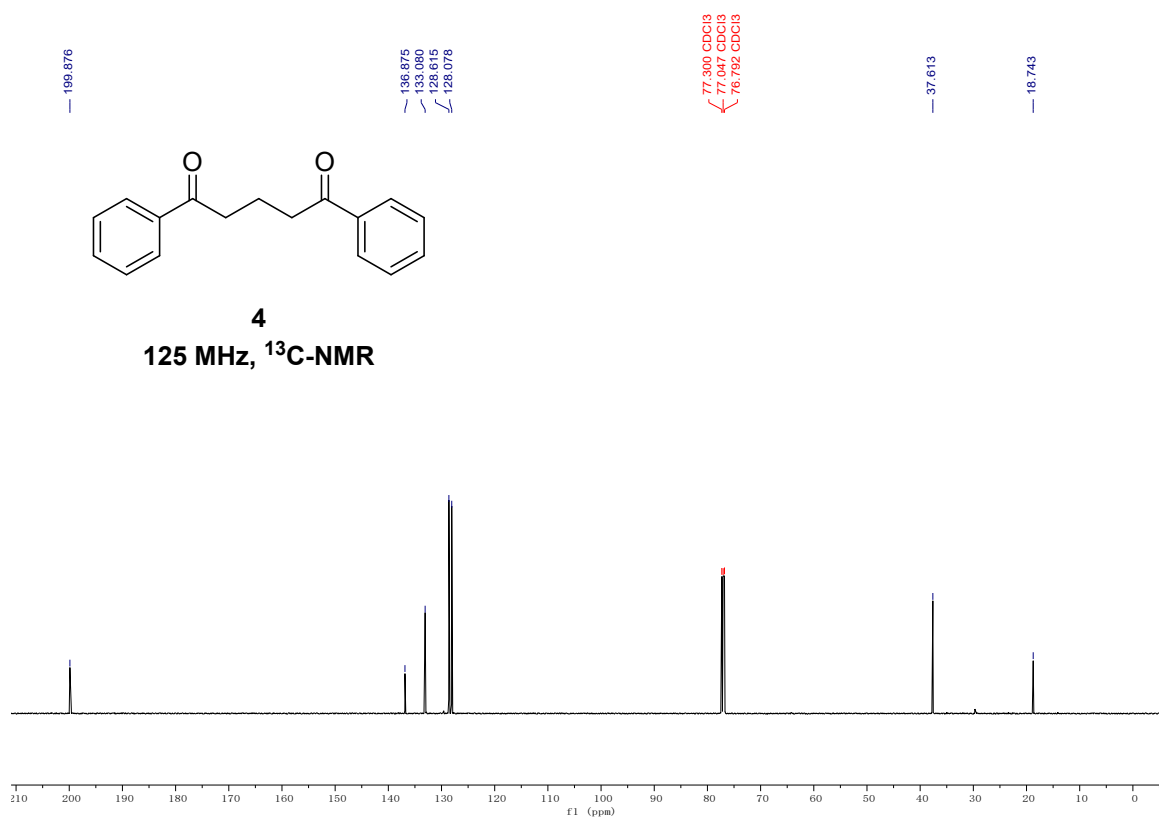
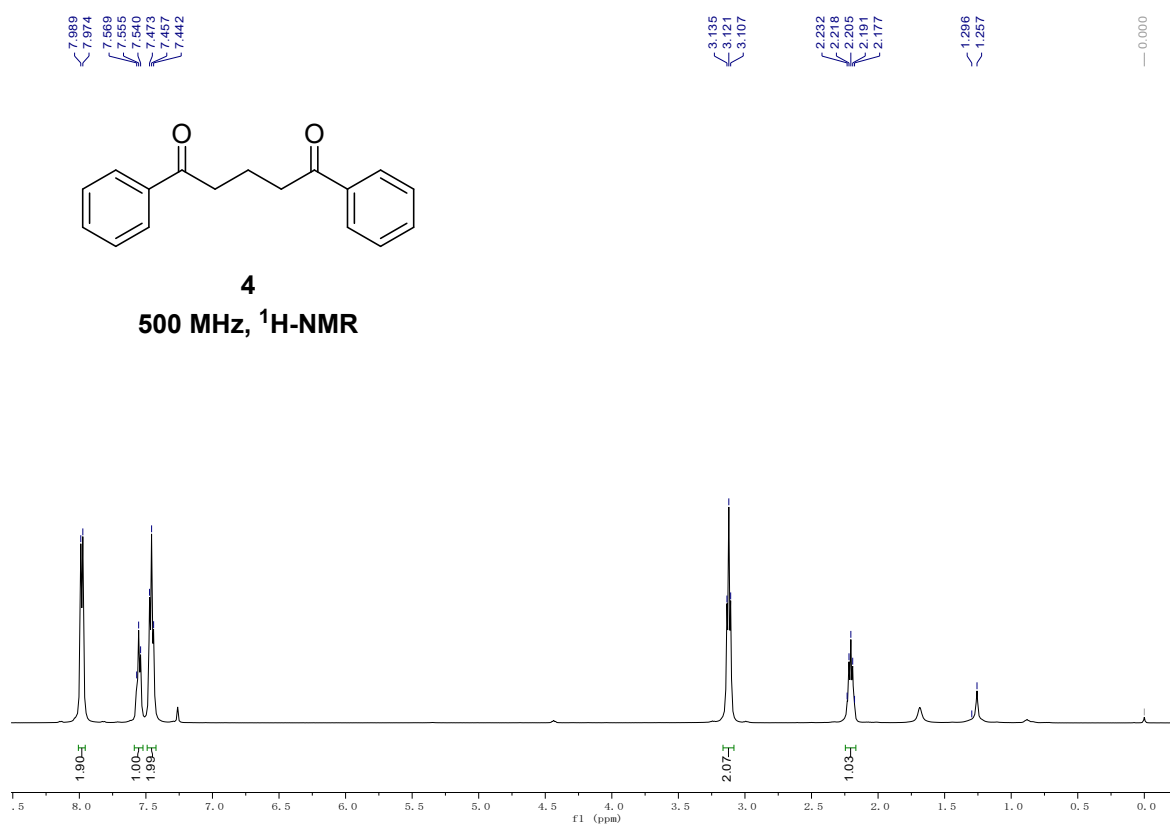


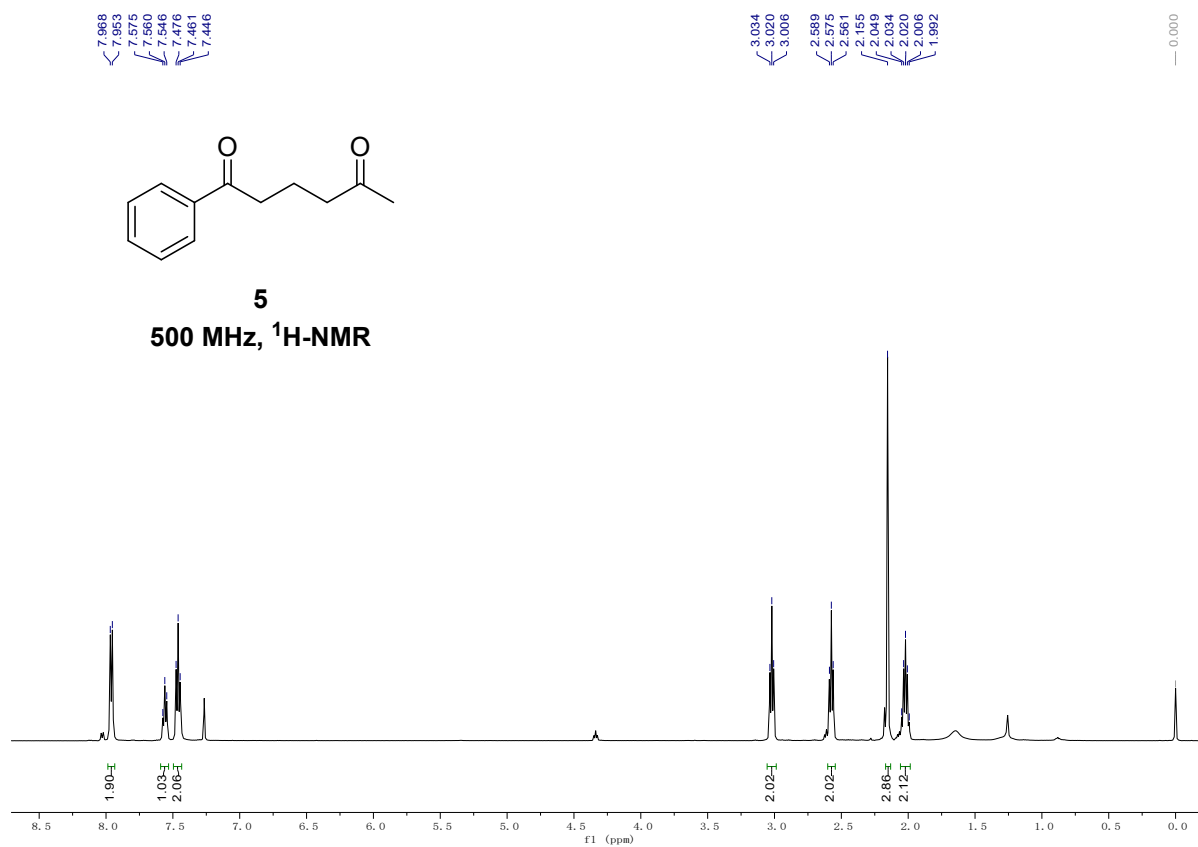


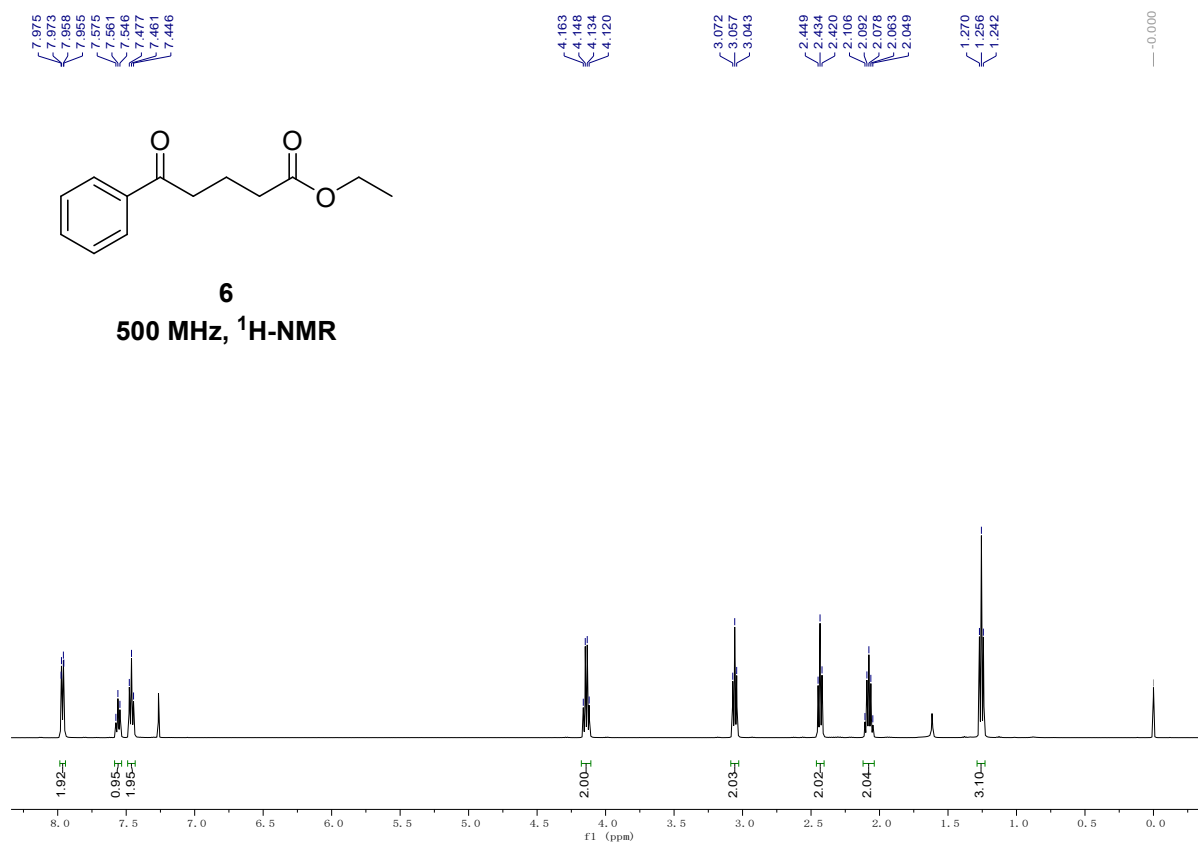
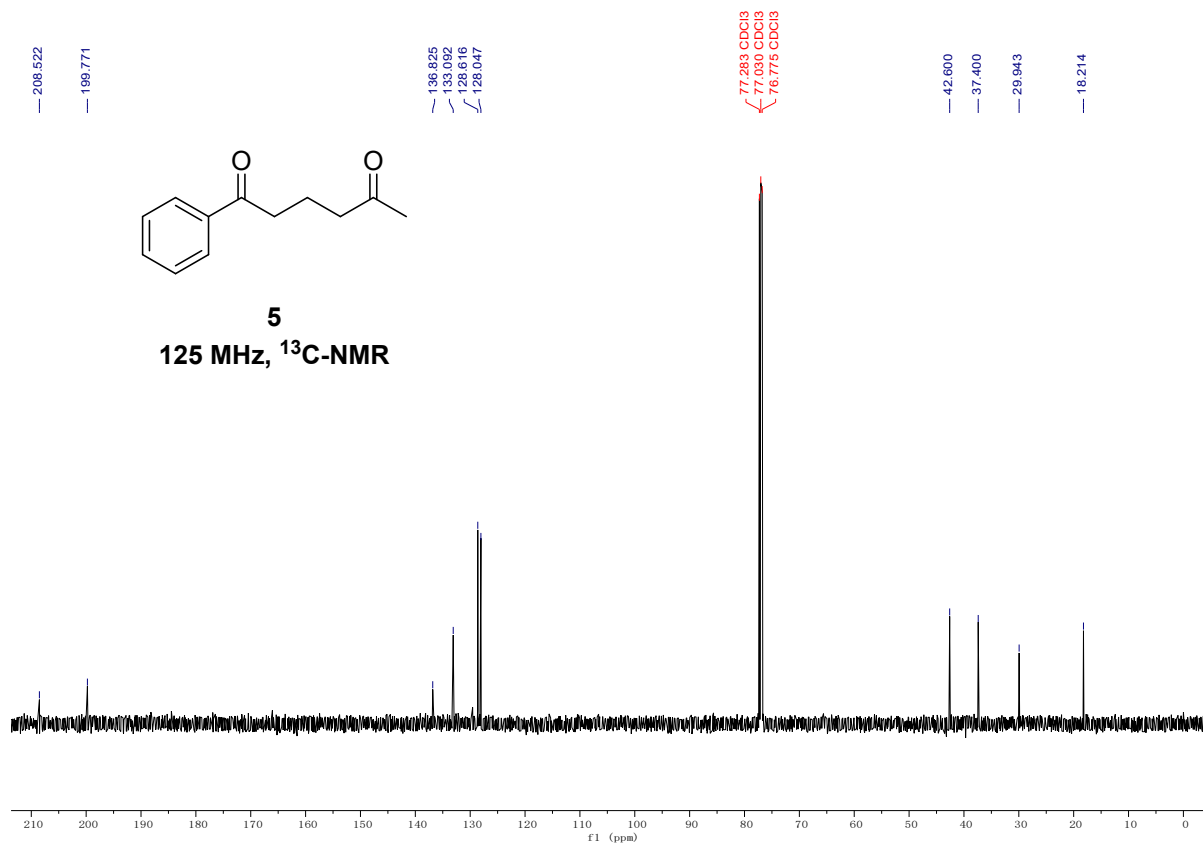


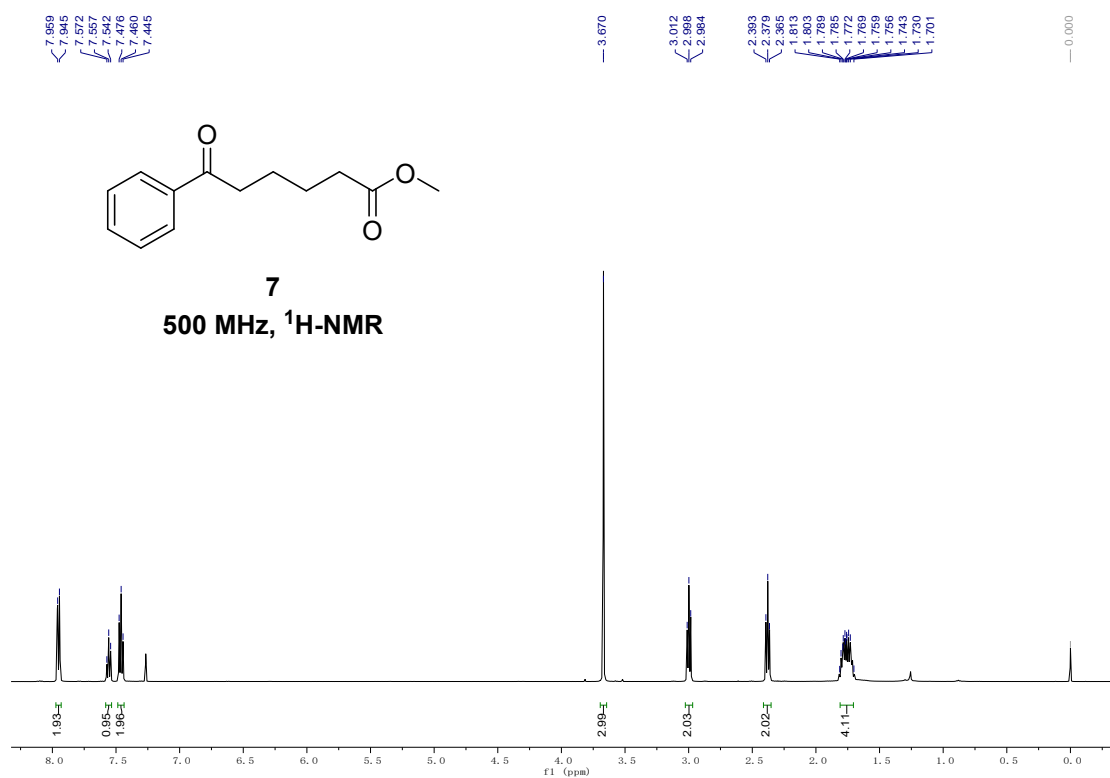
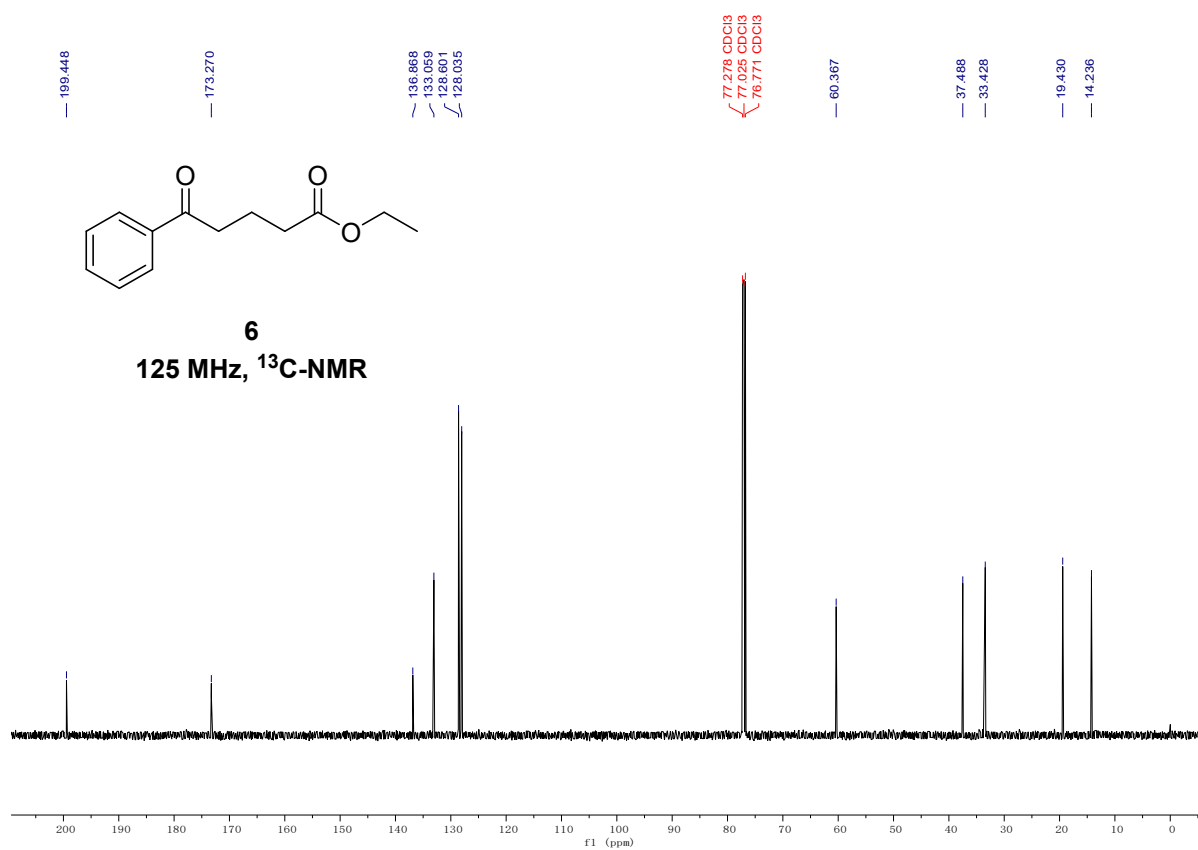


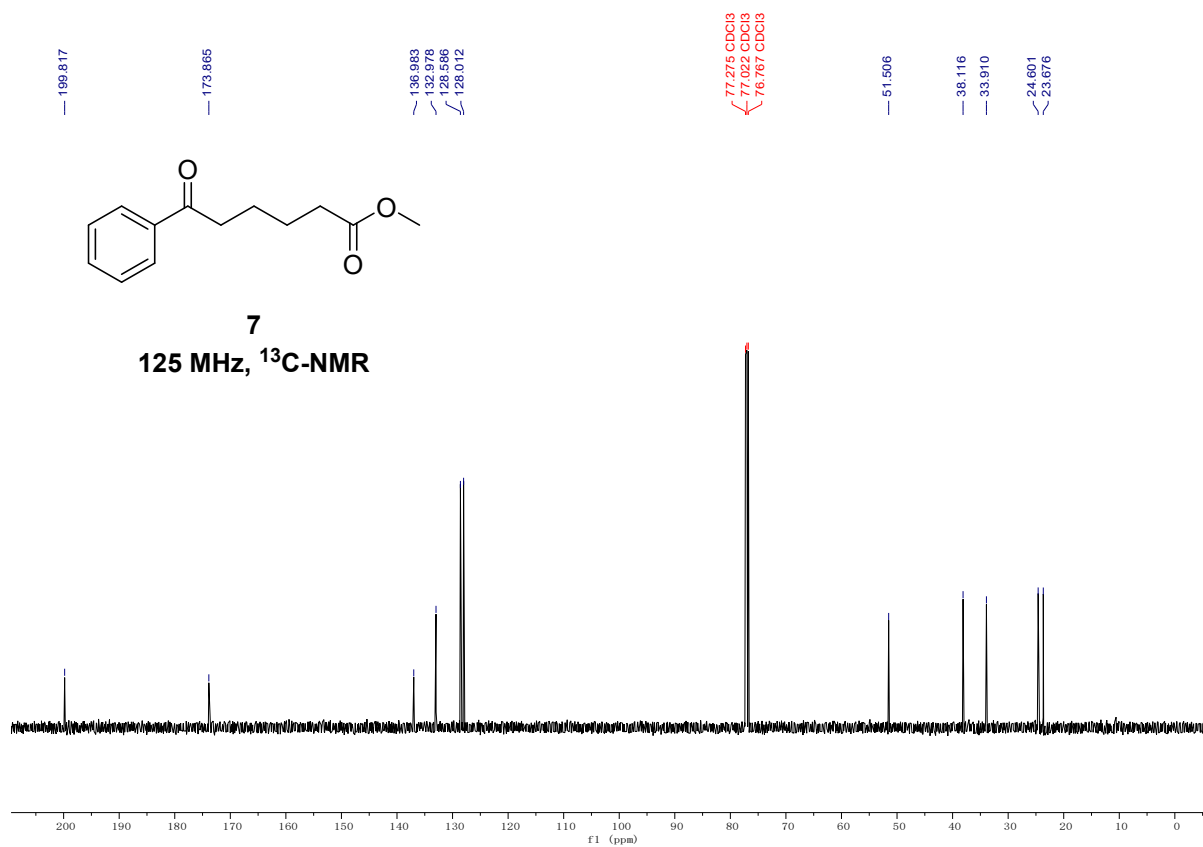


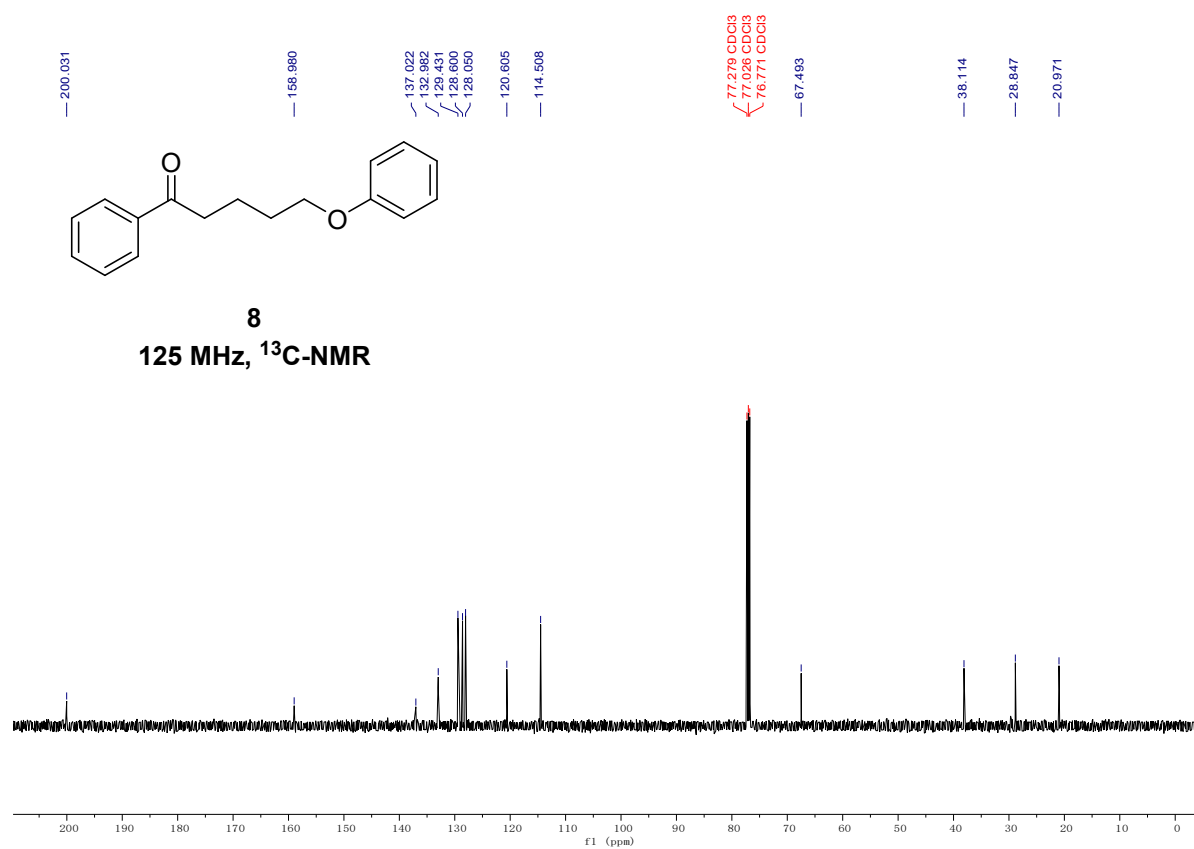
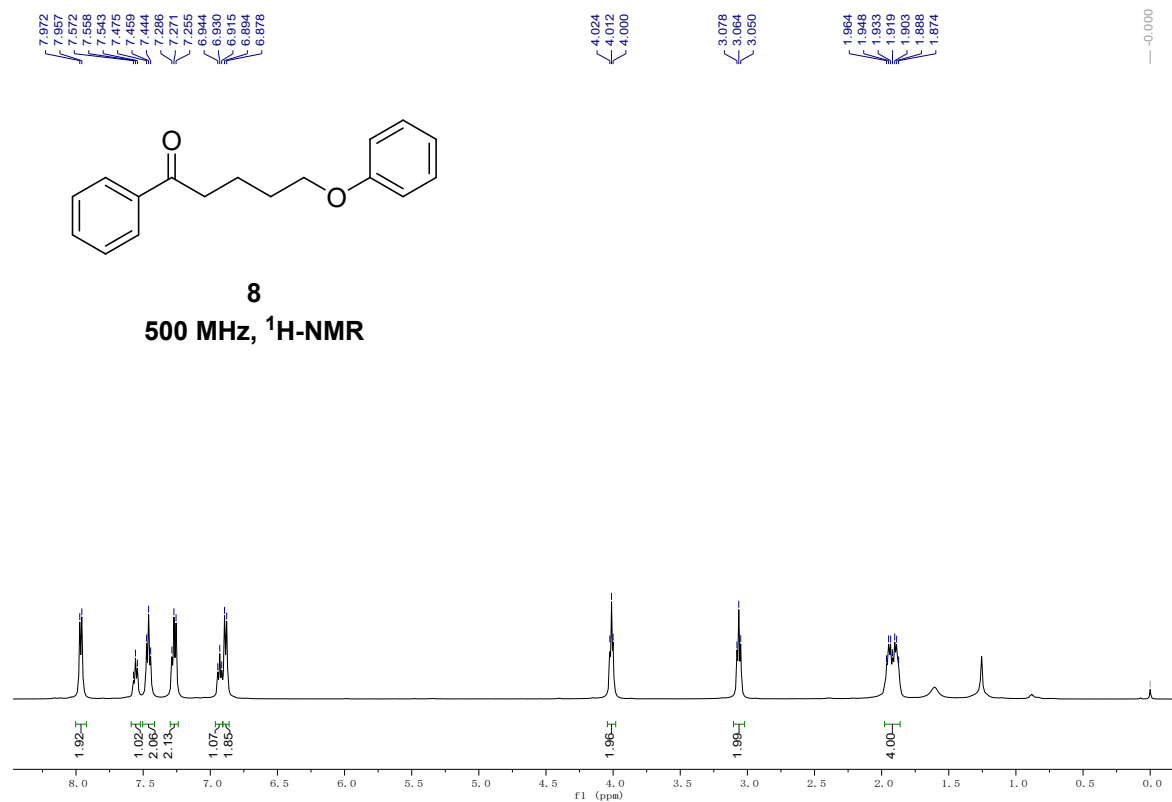


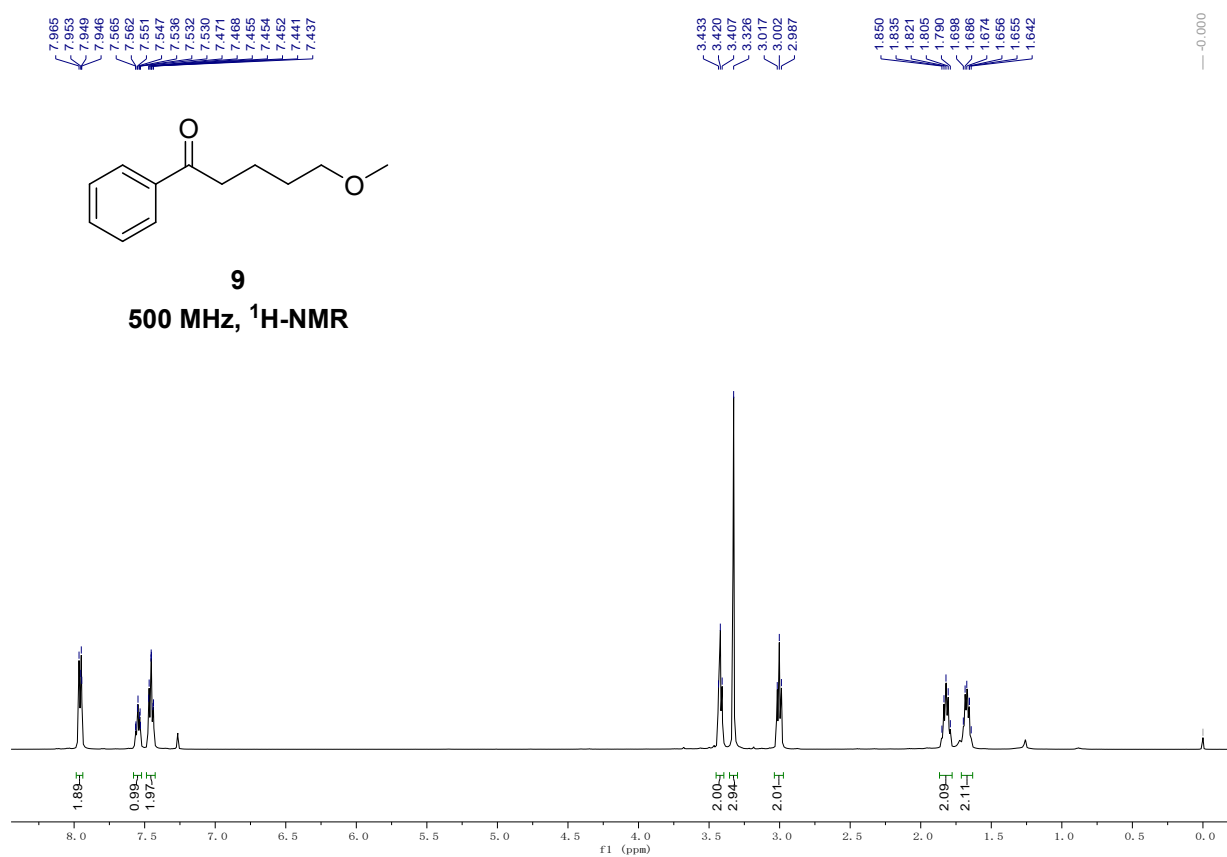


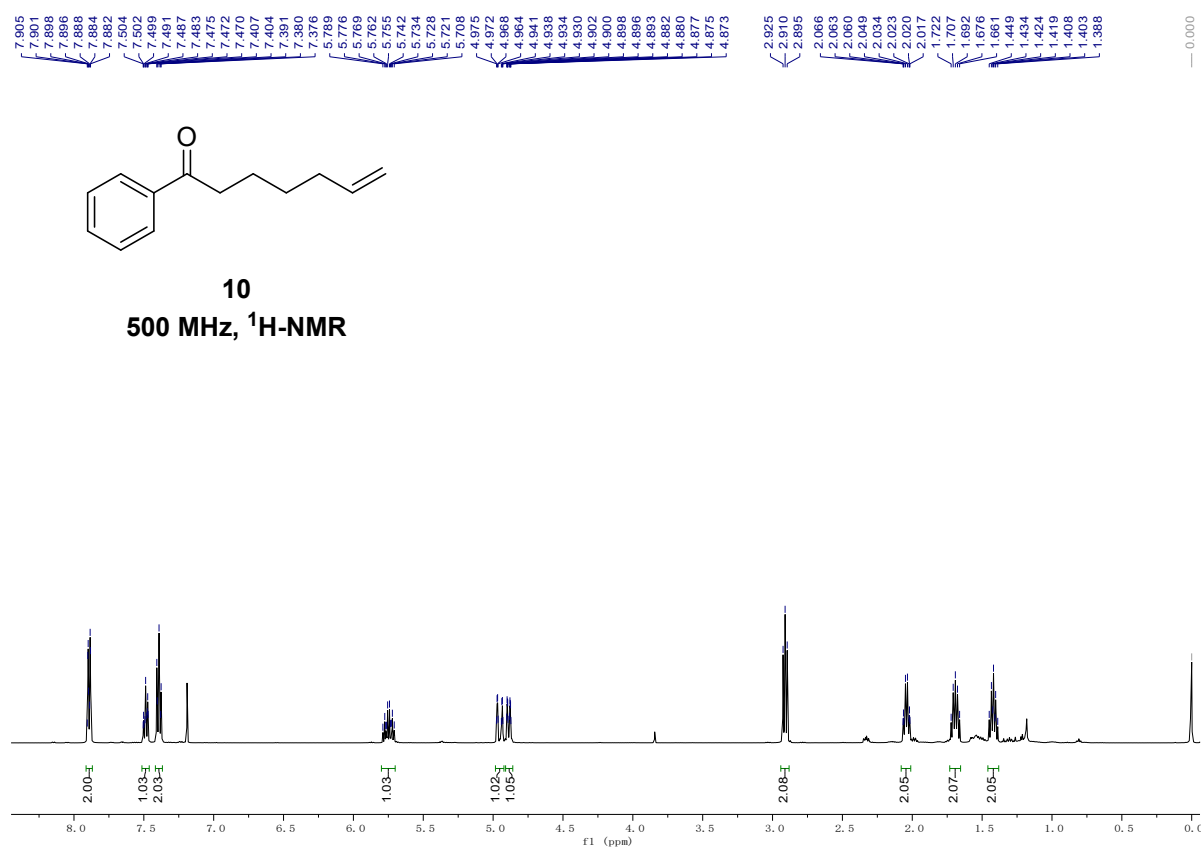
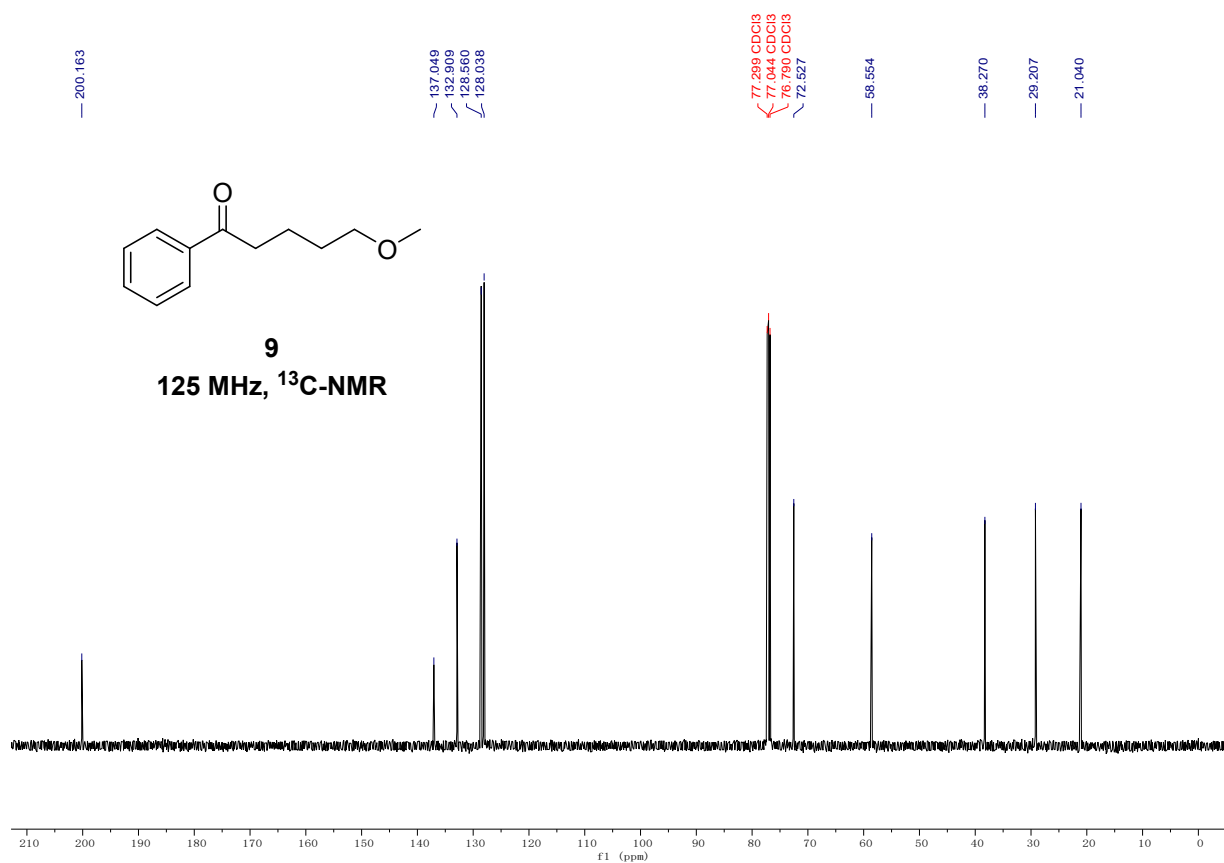


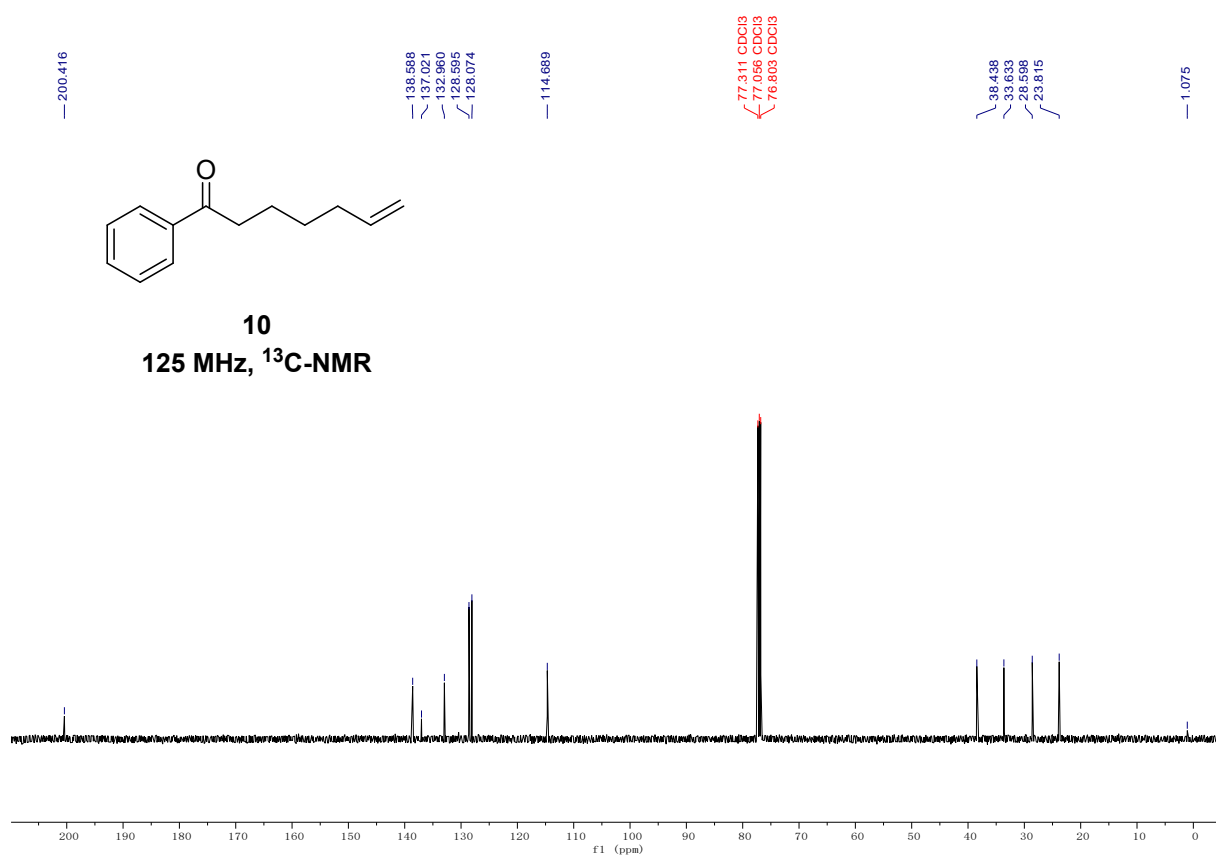


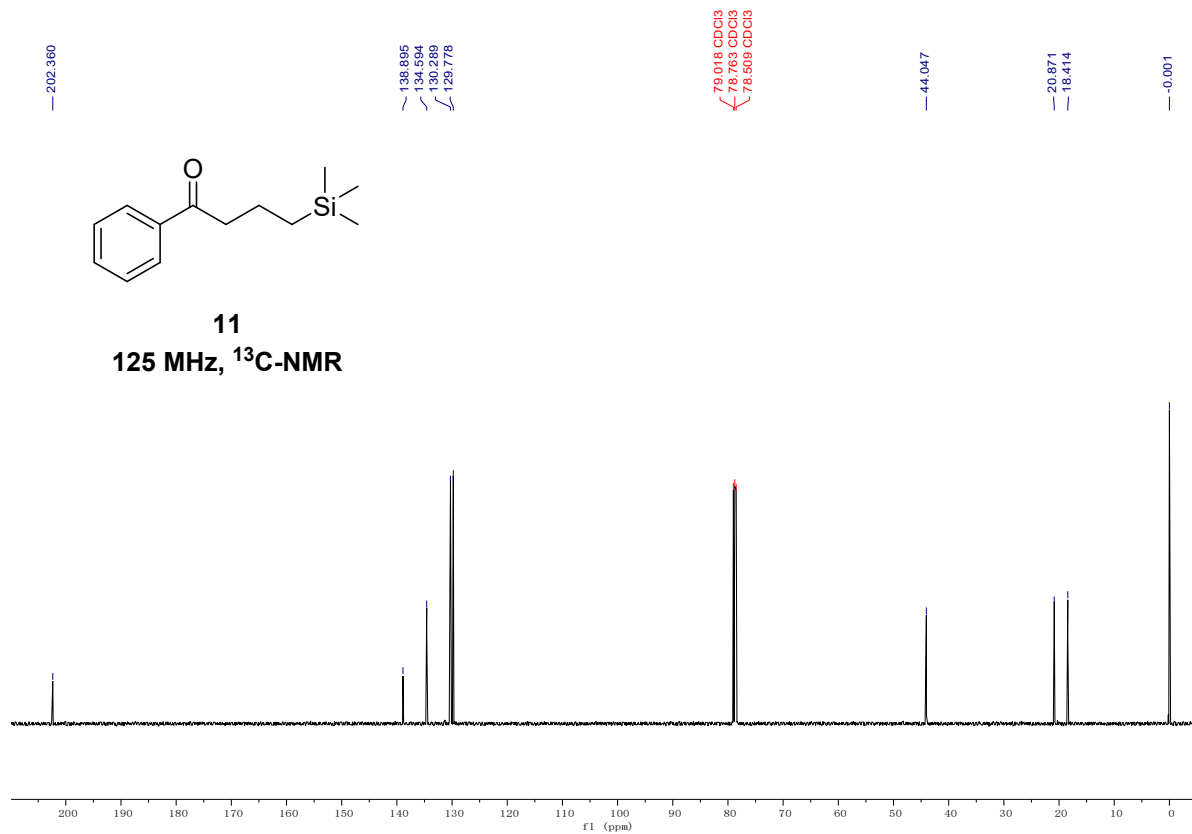
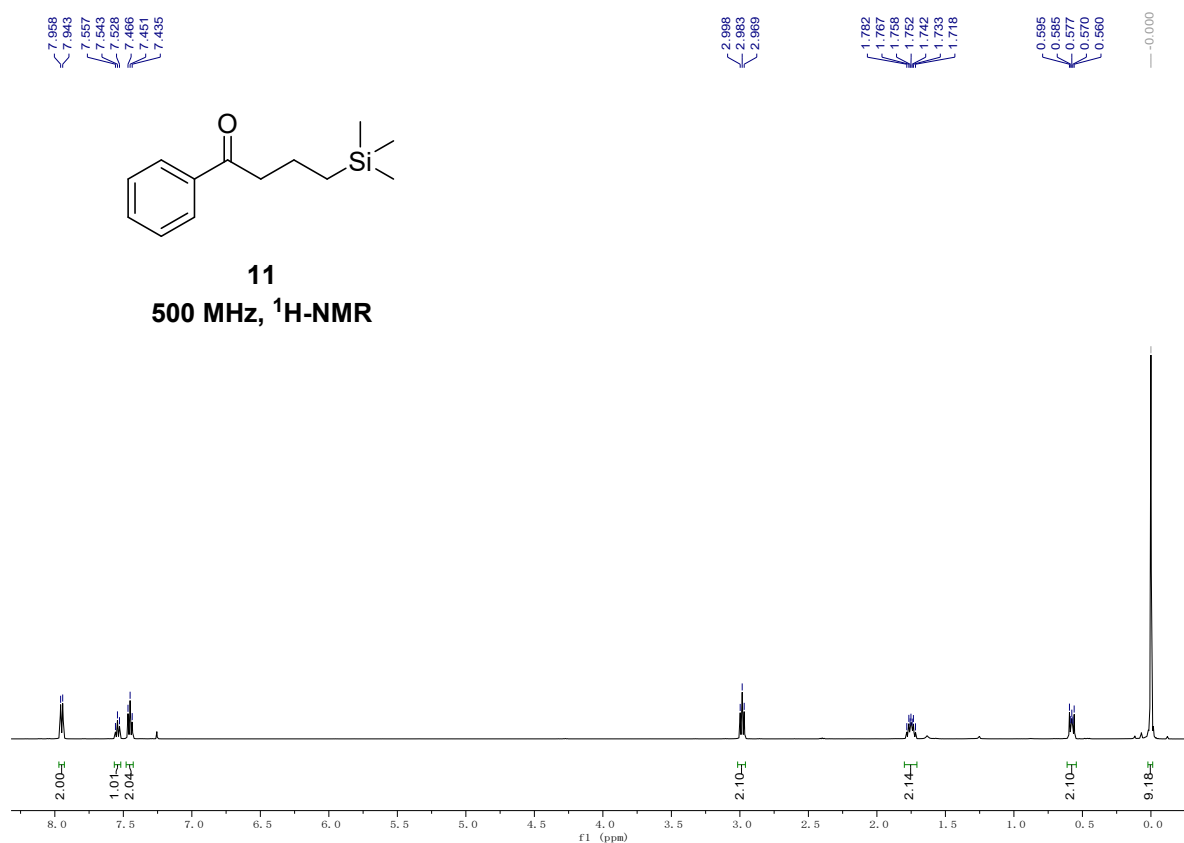


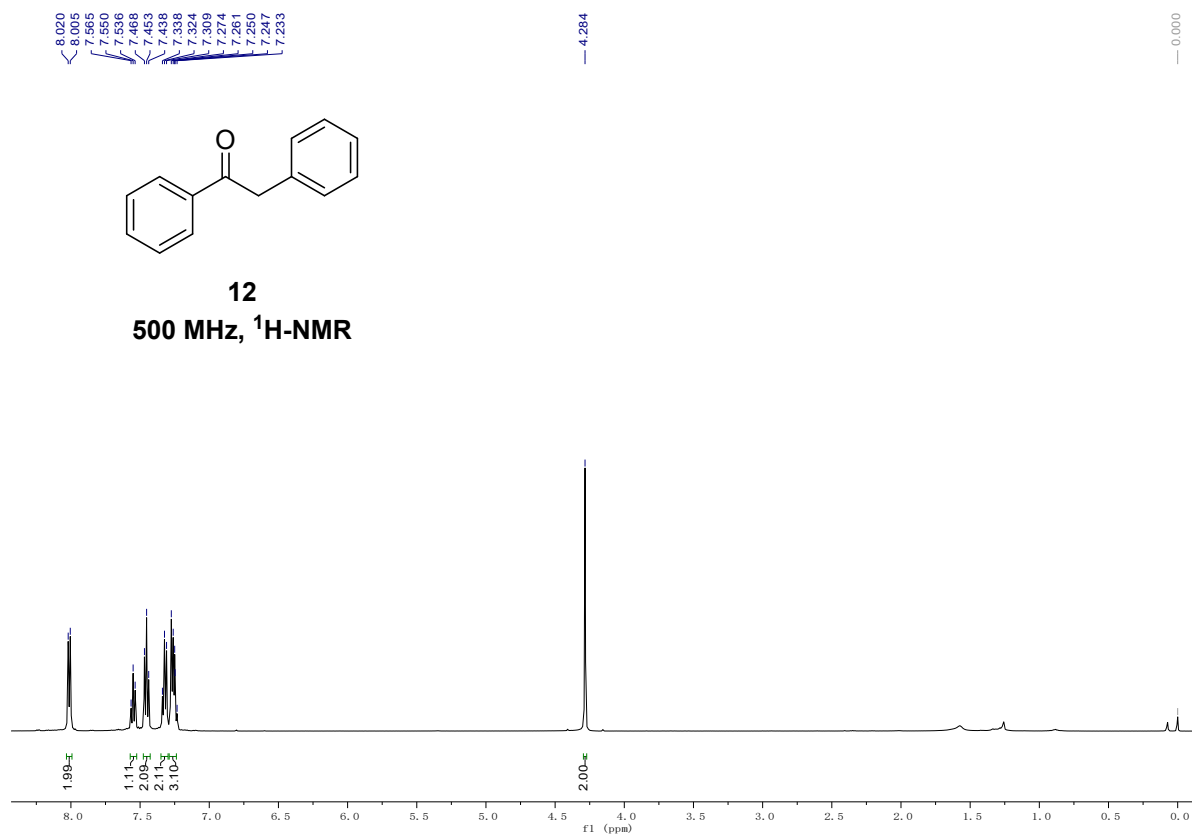


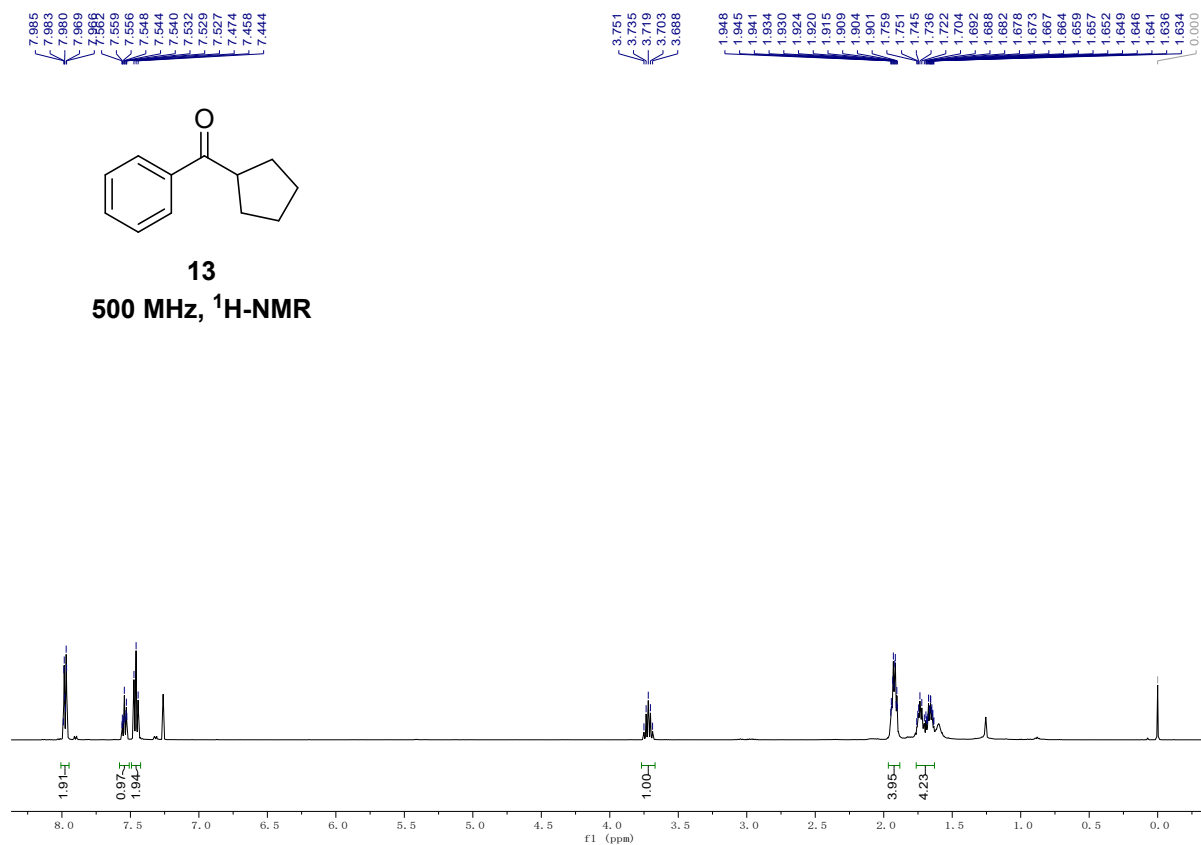
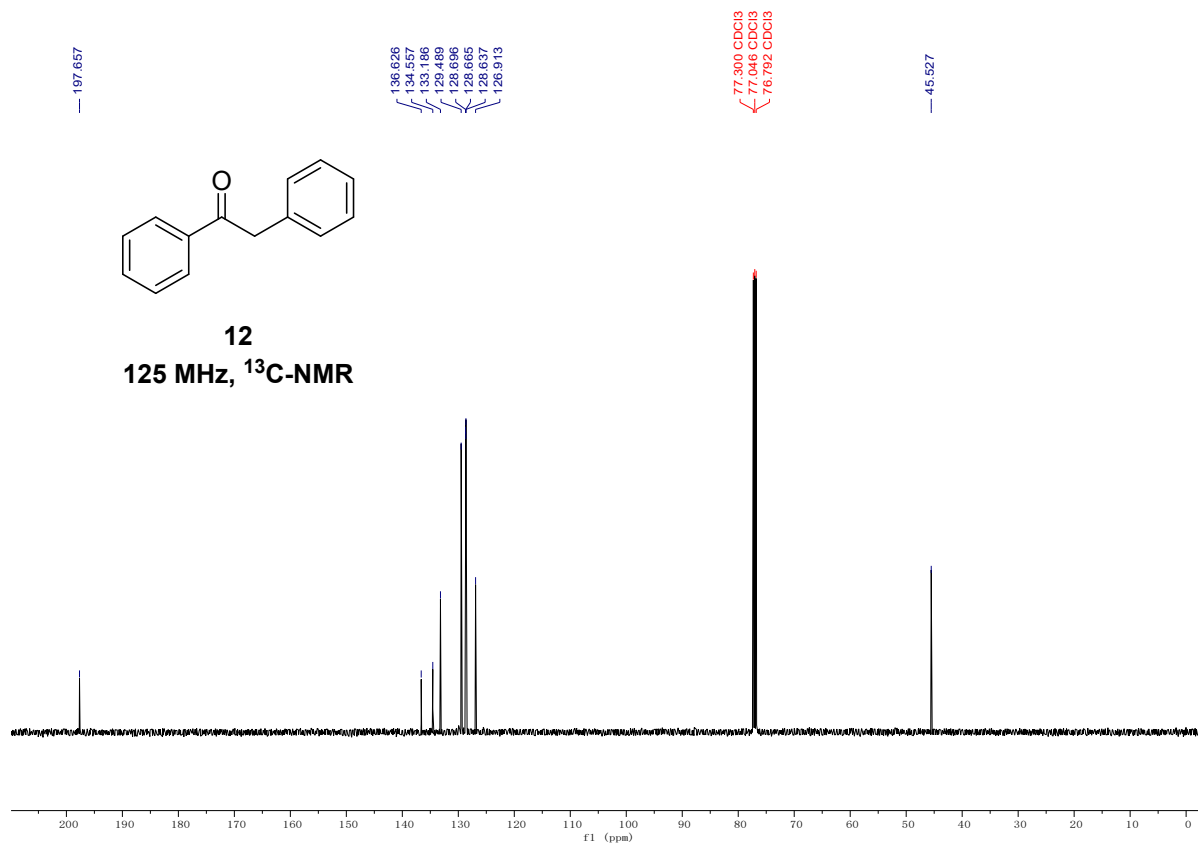


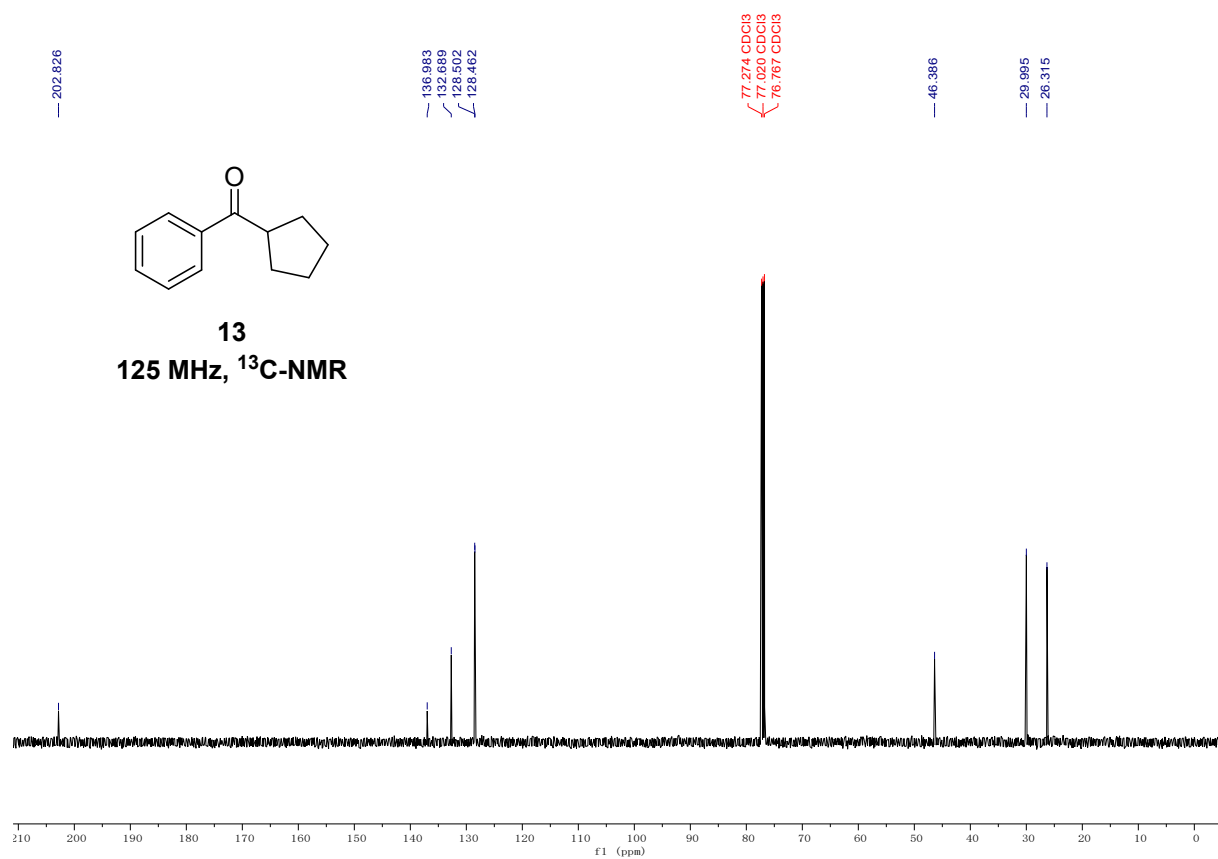




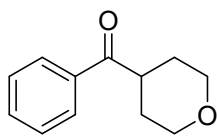






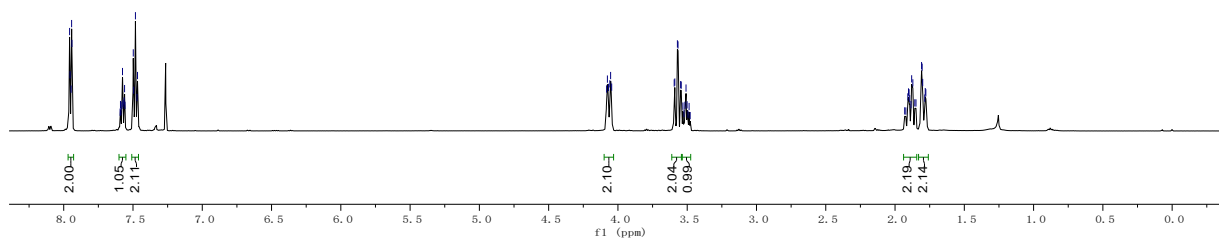


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7.467



14
500 MHz, ¹H-NMR

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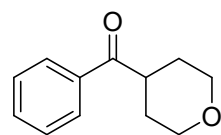
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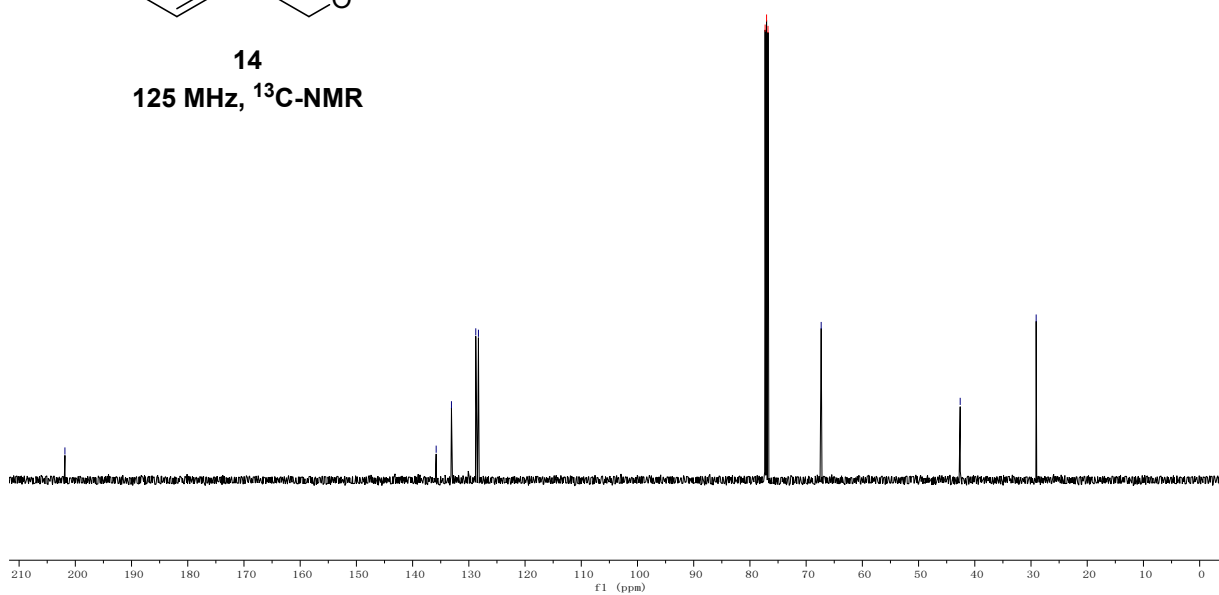
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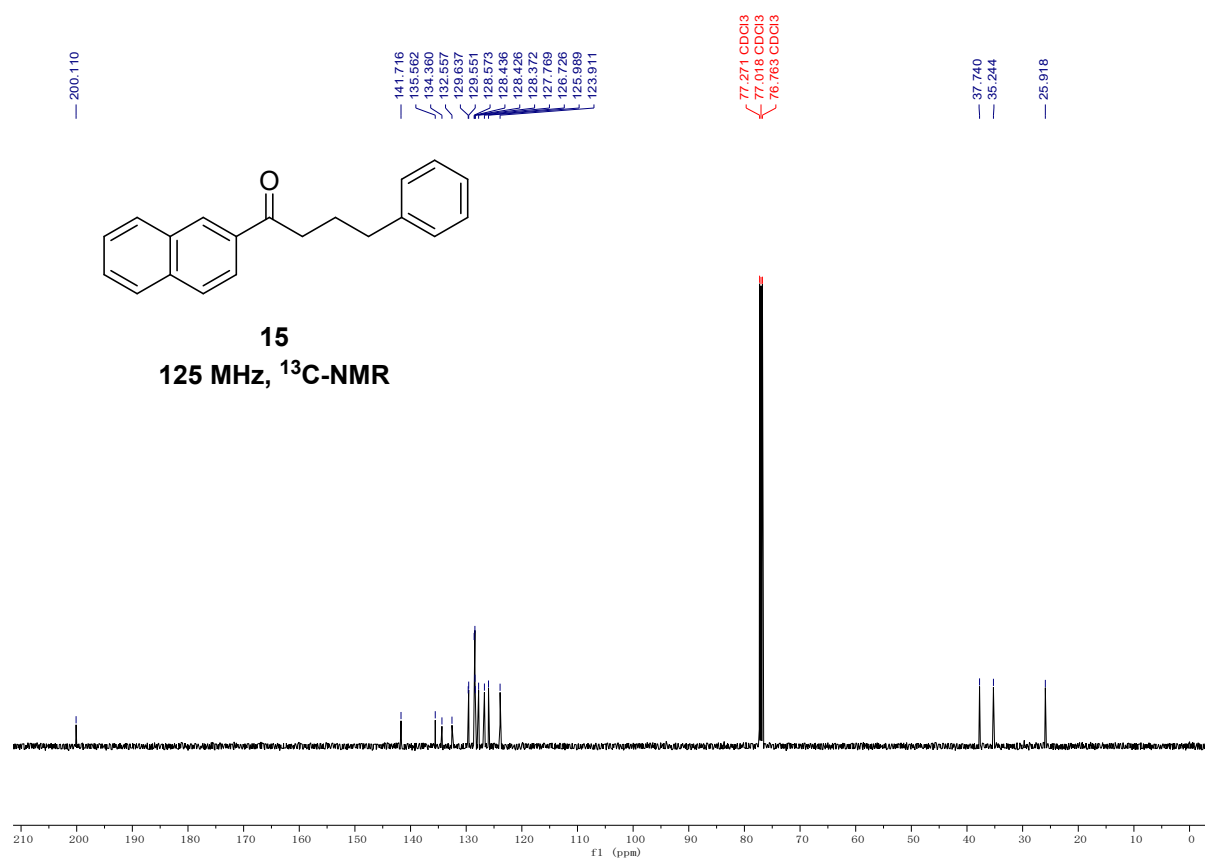
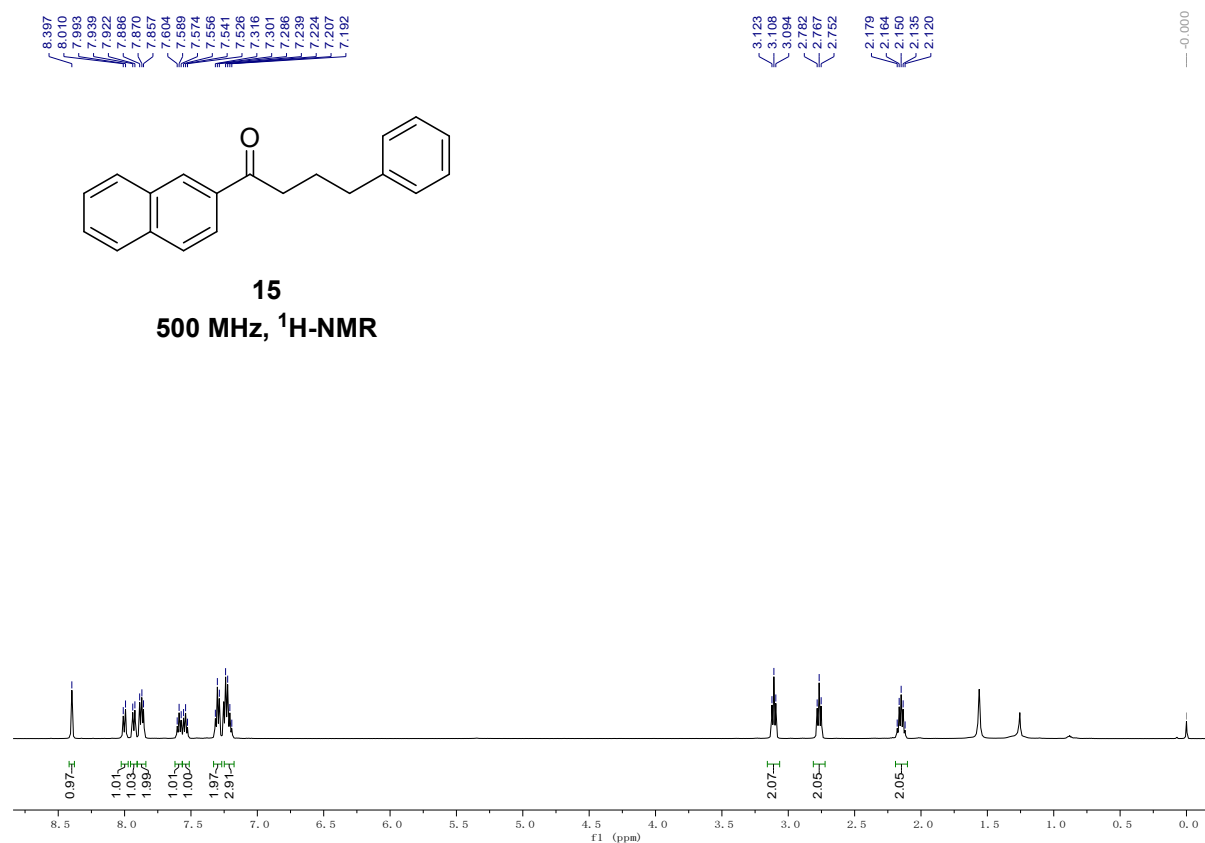
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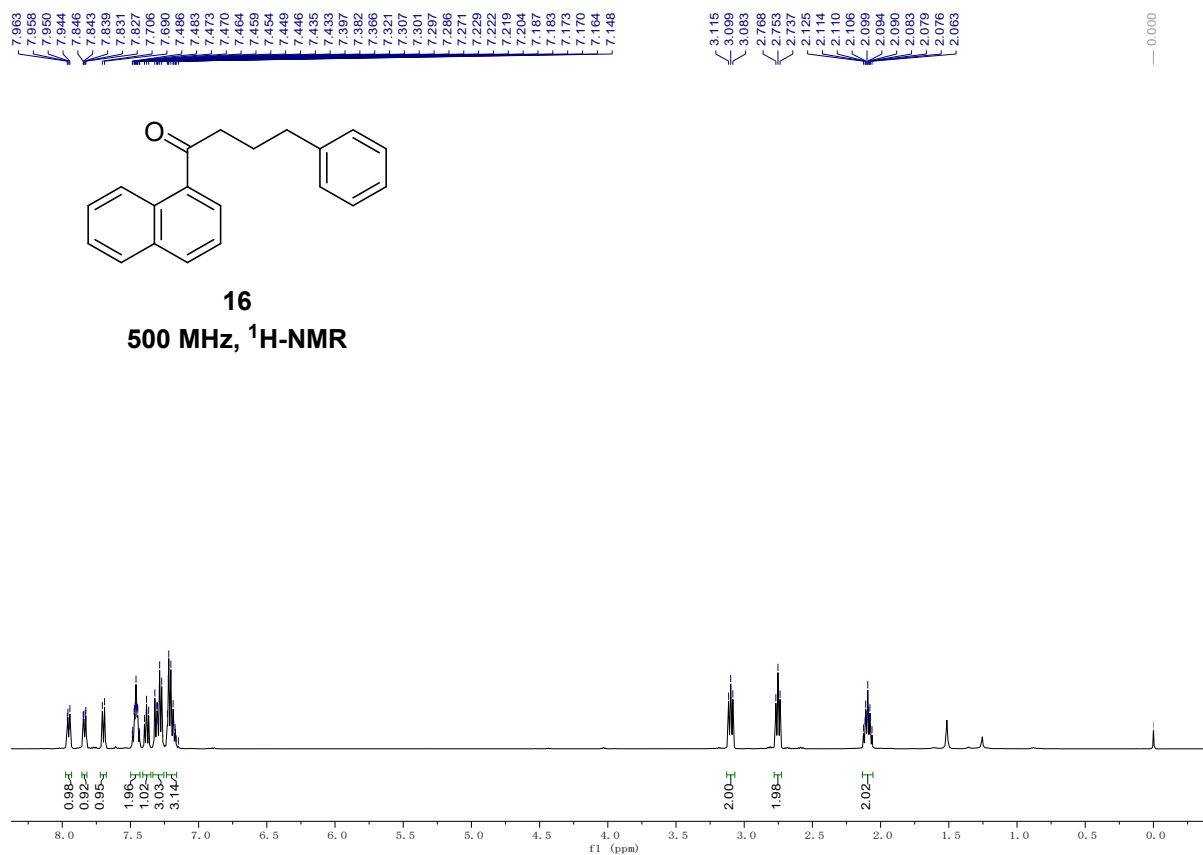
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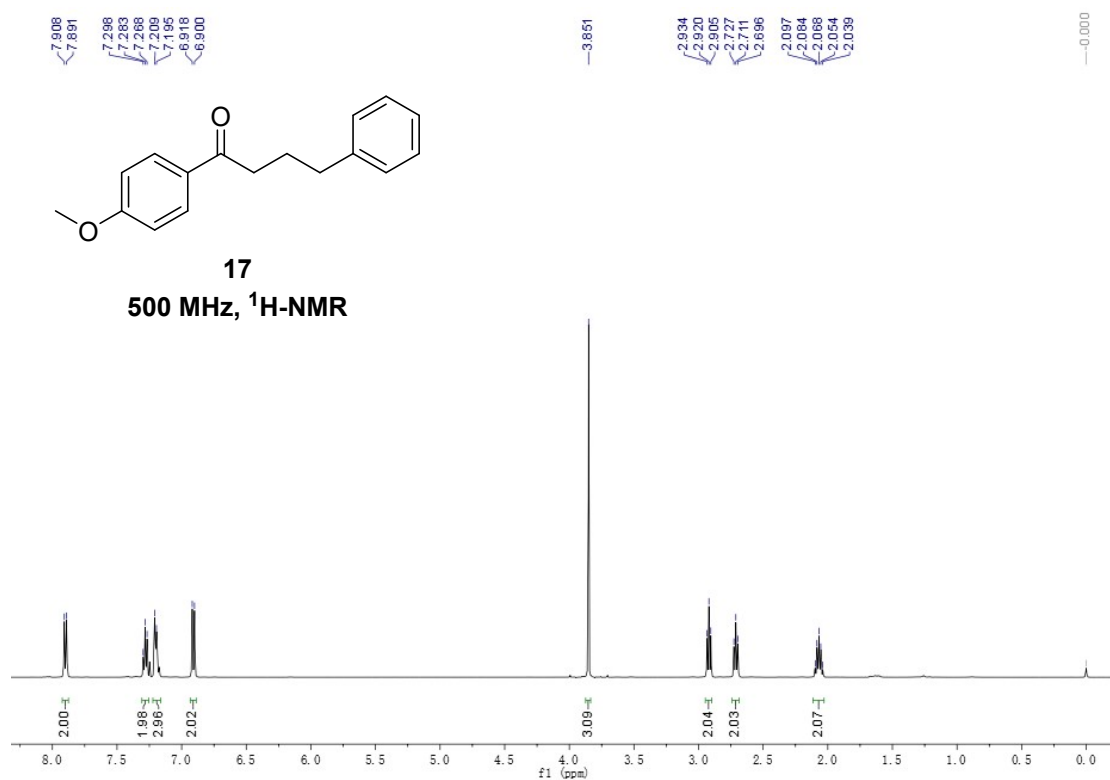
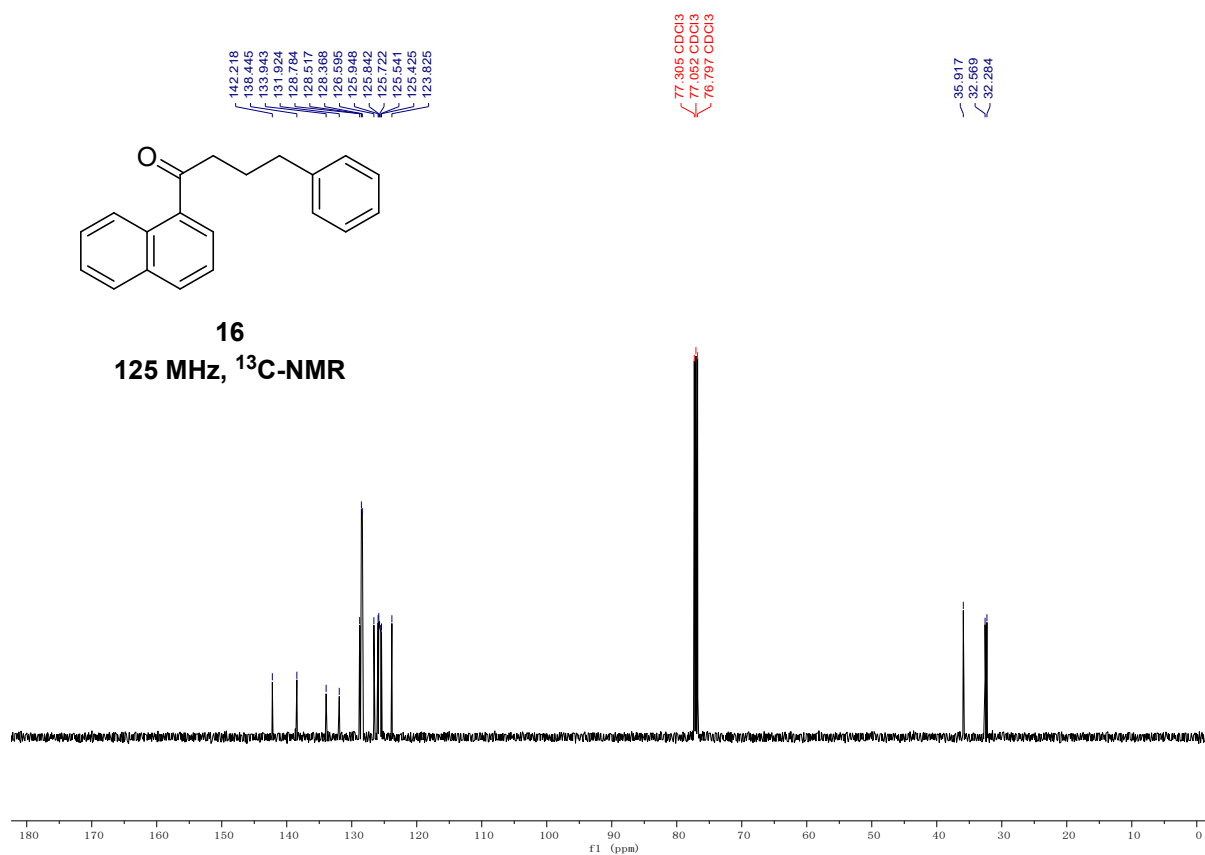


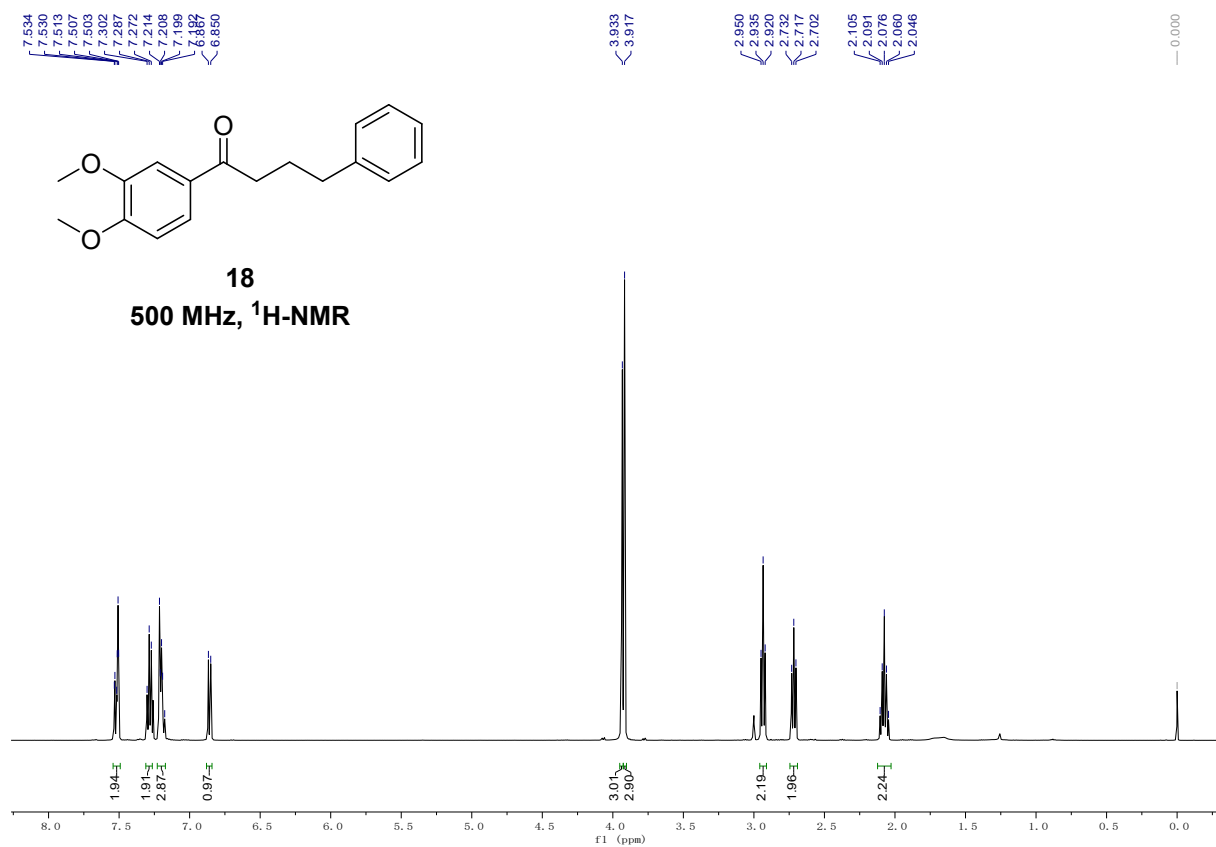
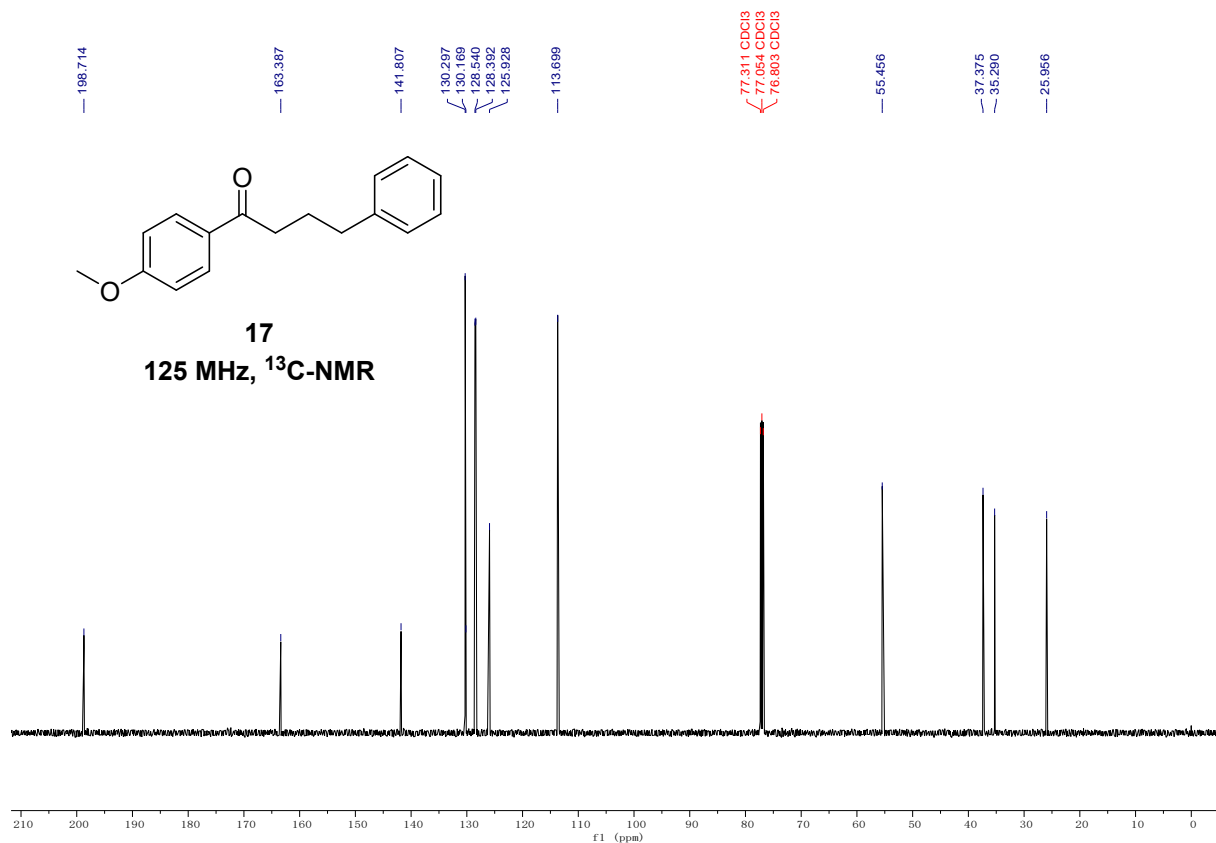
14
125 MHz, ¹³C-NMR

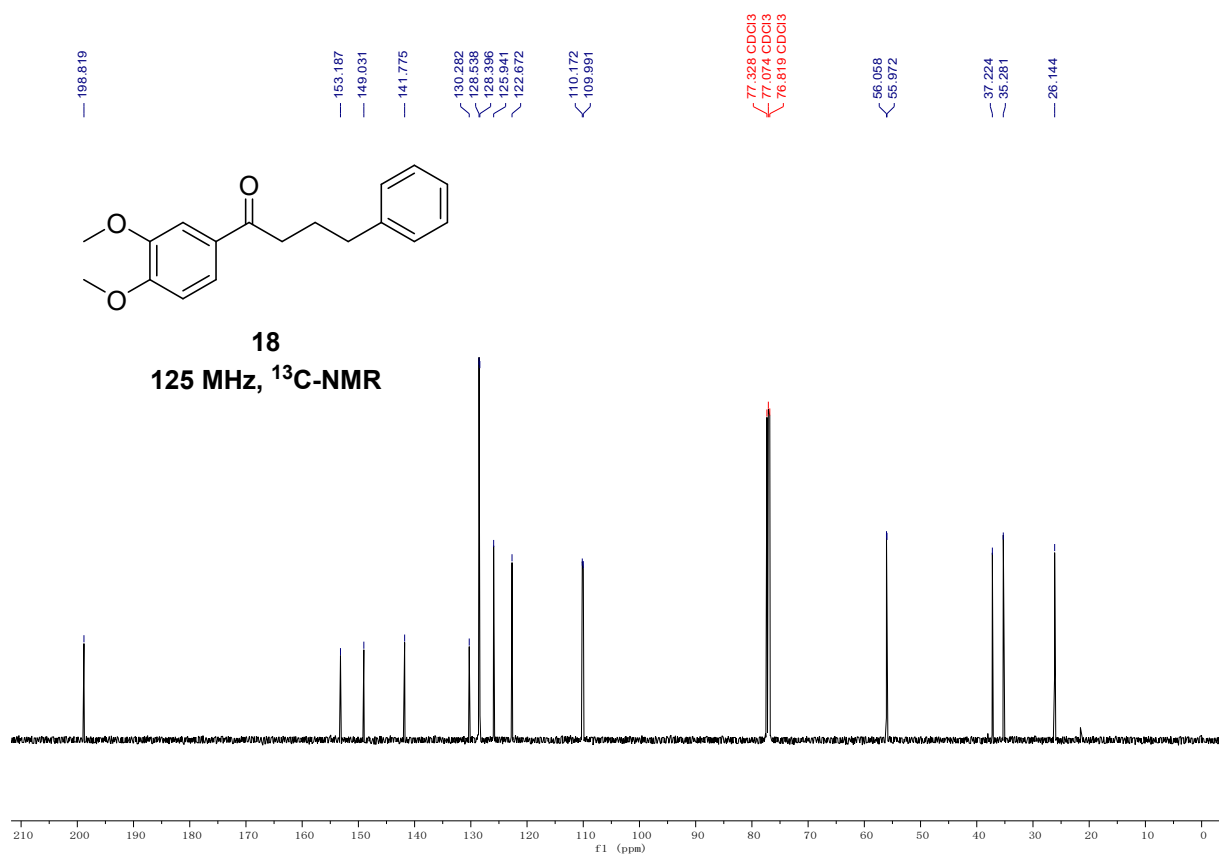


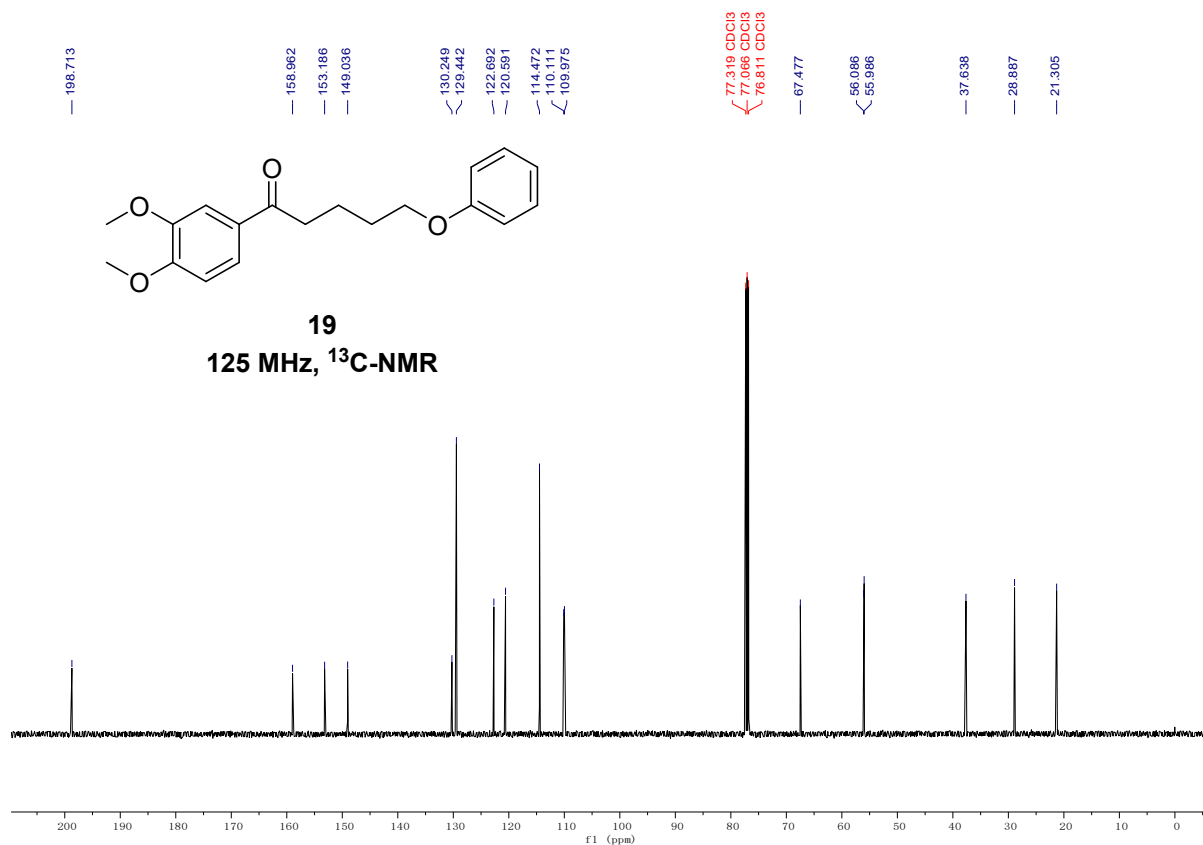
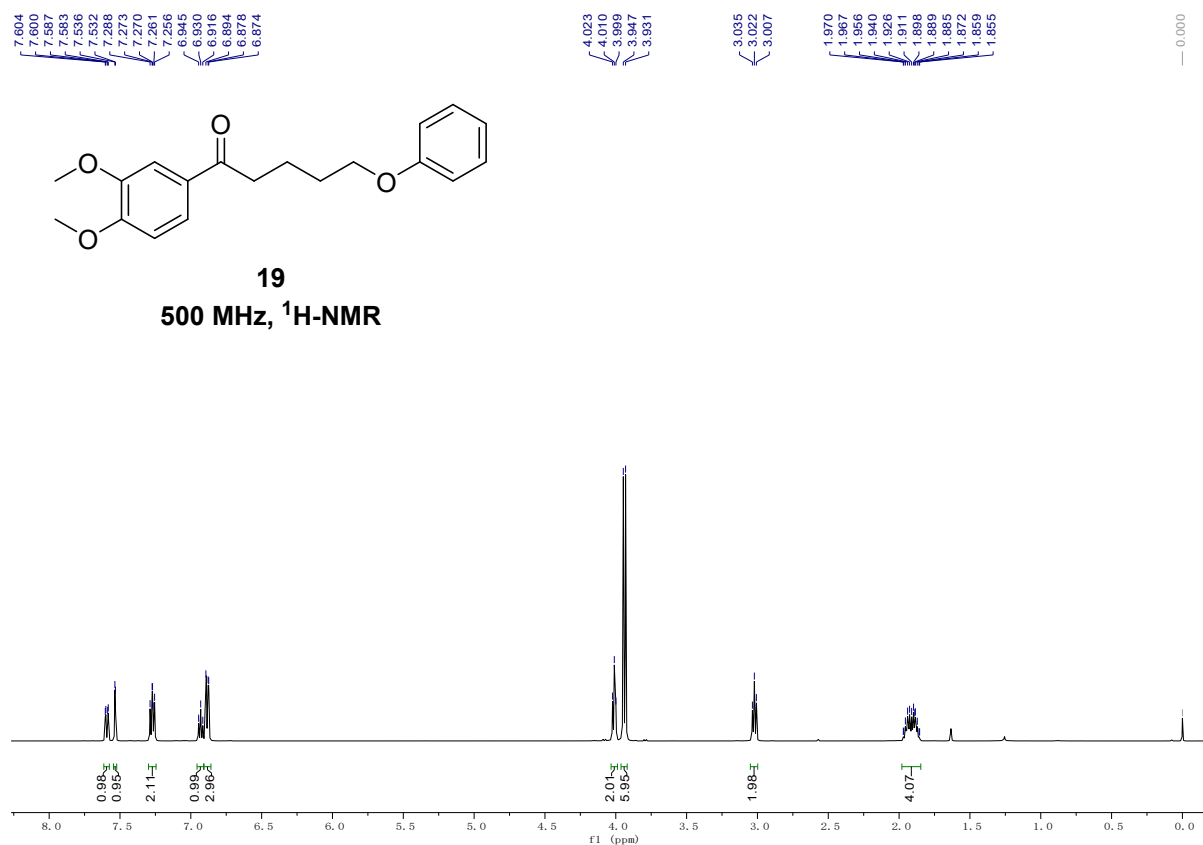


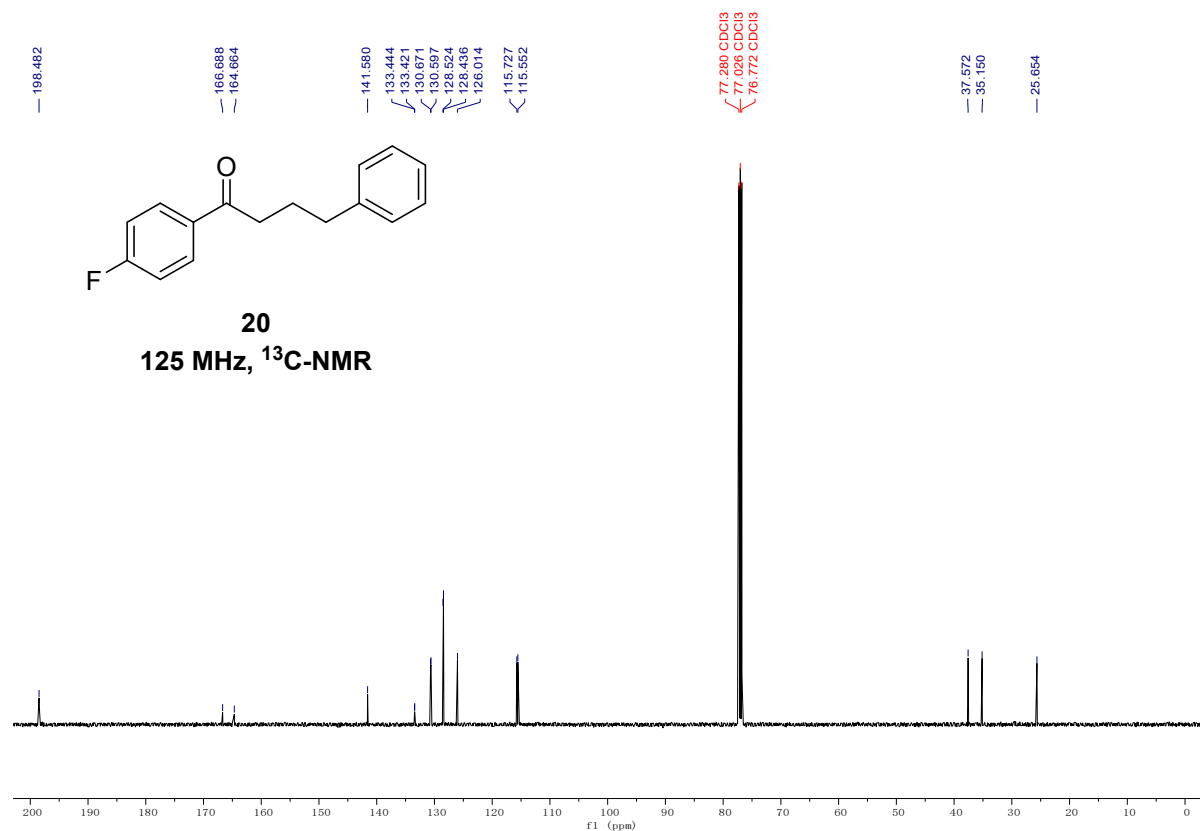
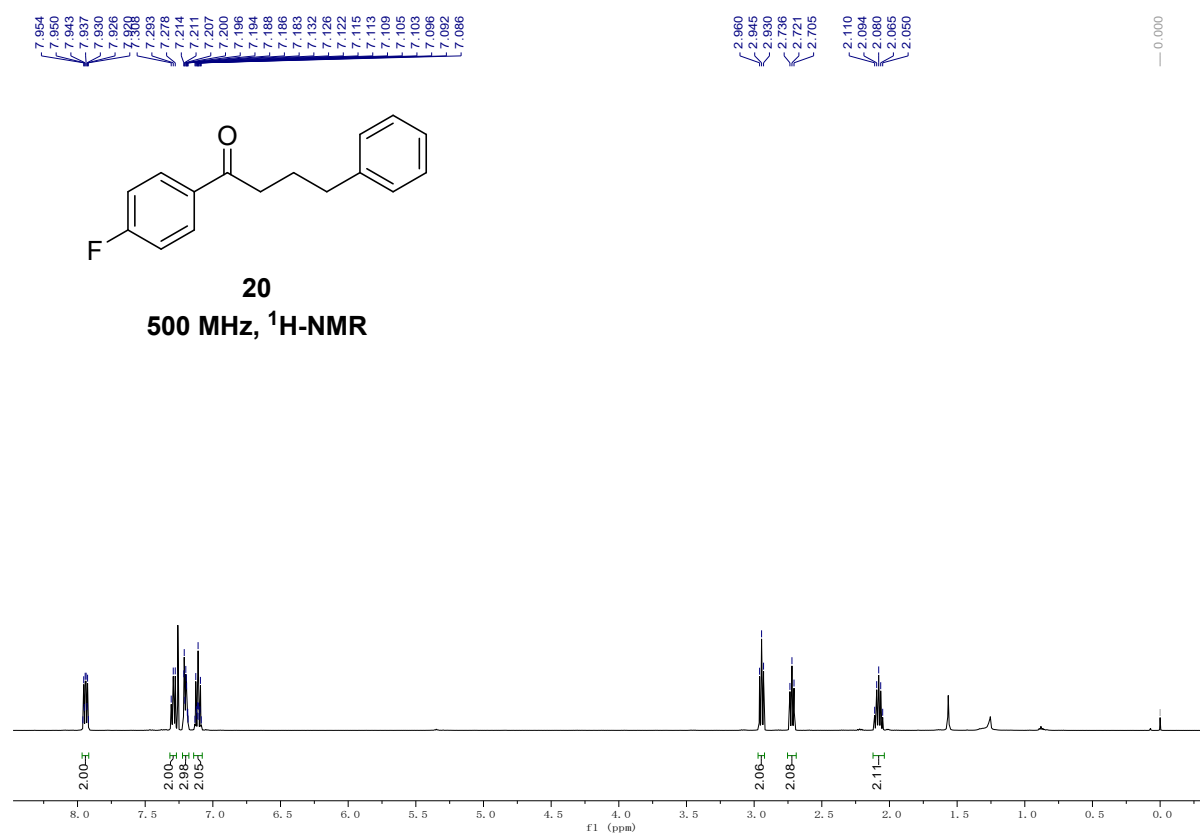


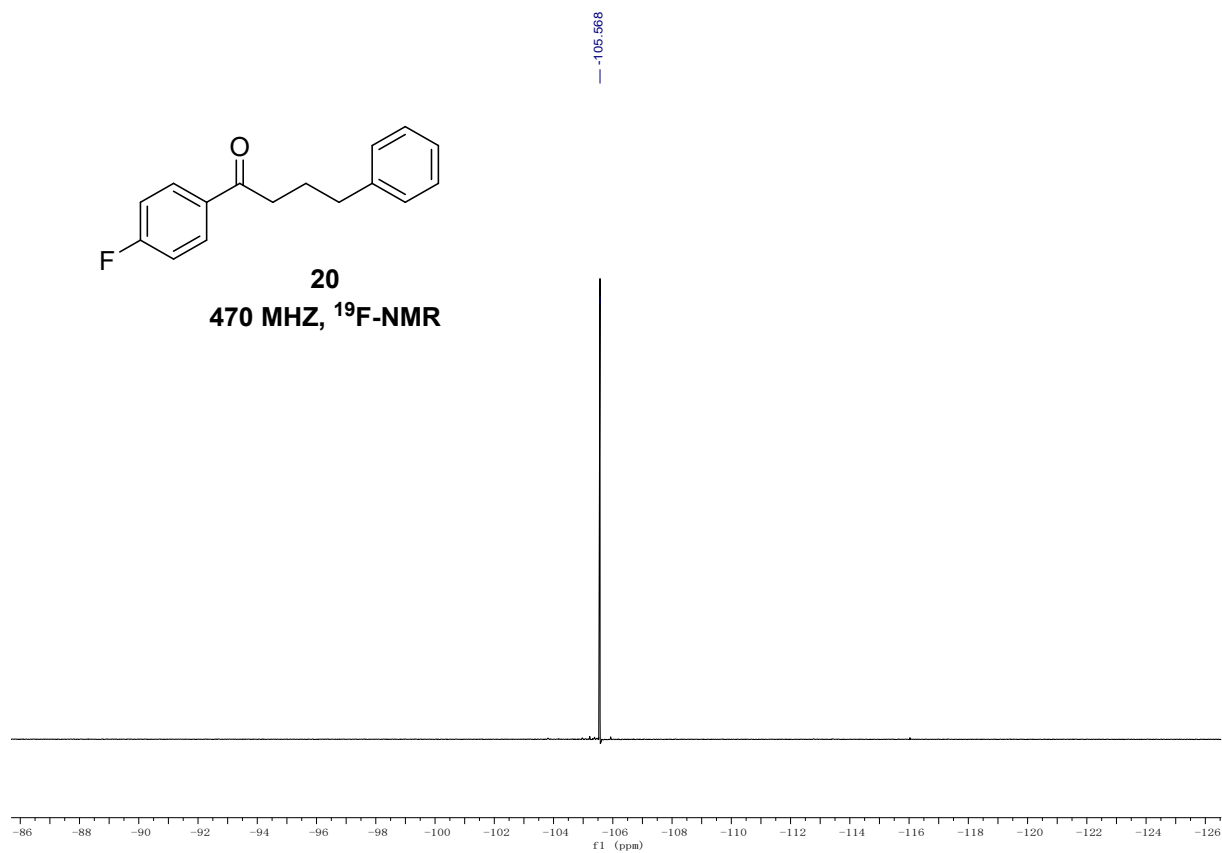


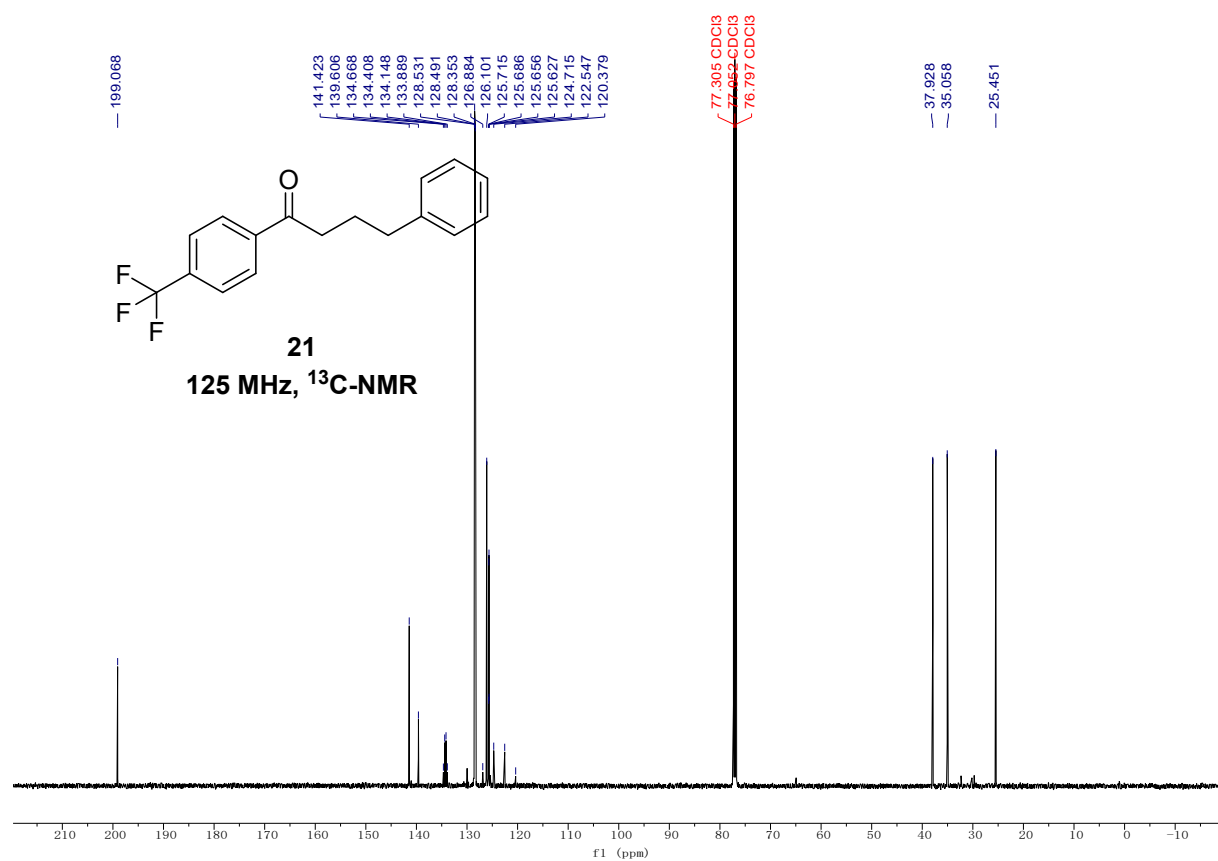
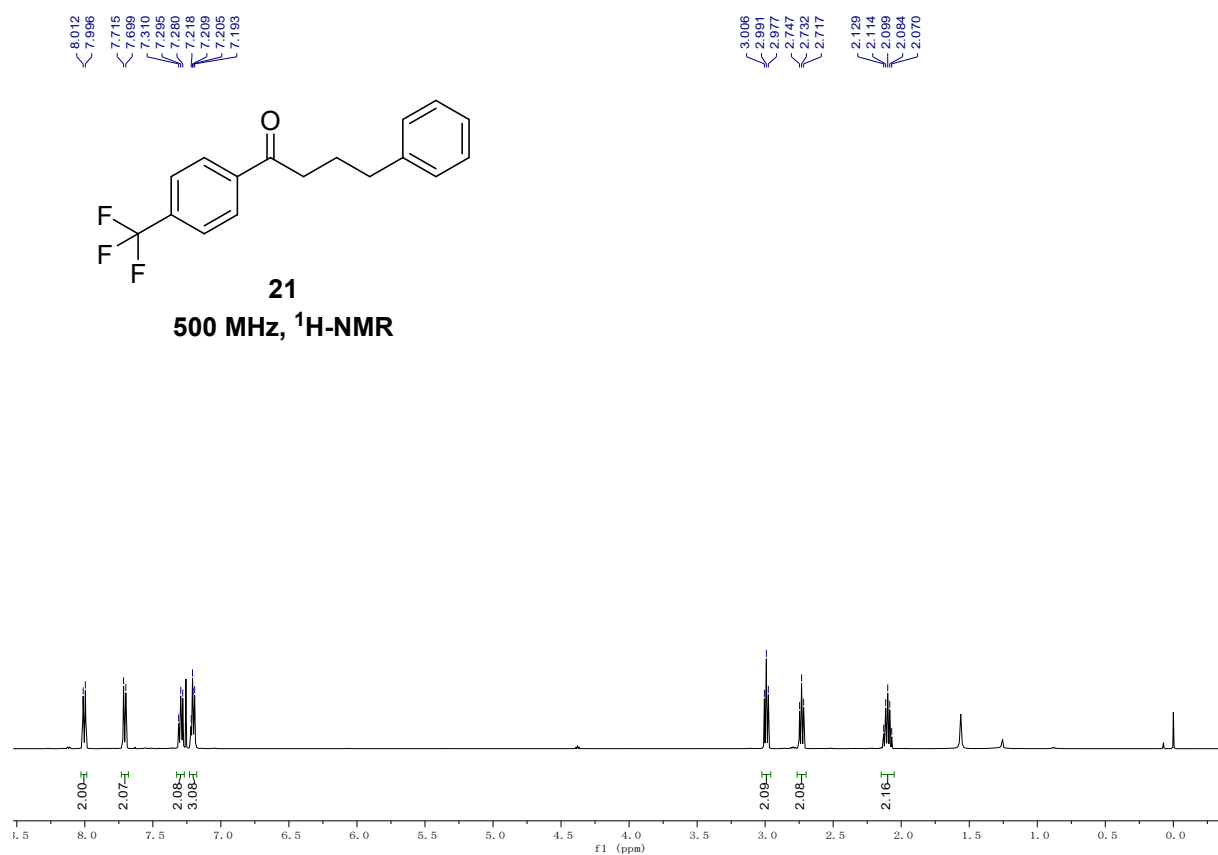


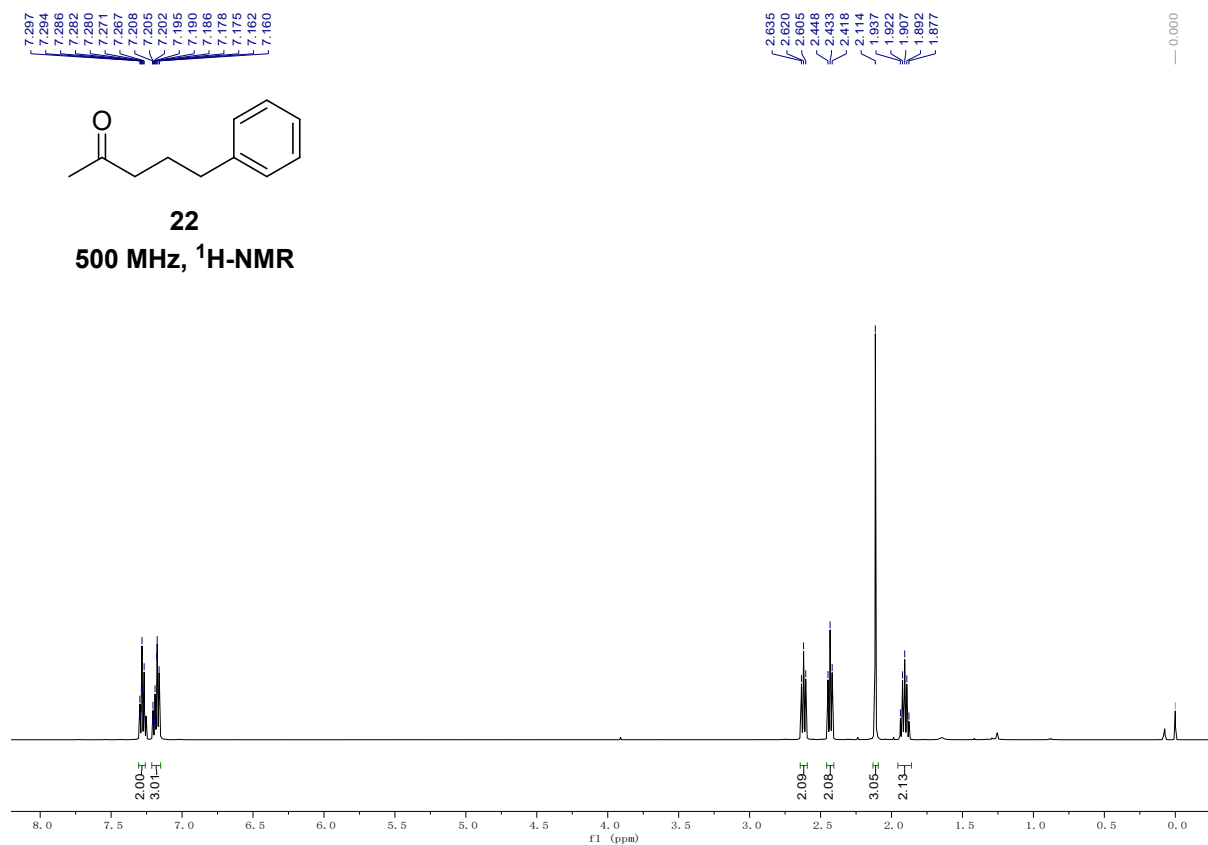
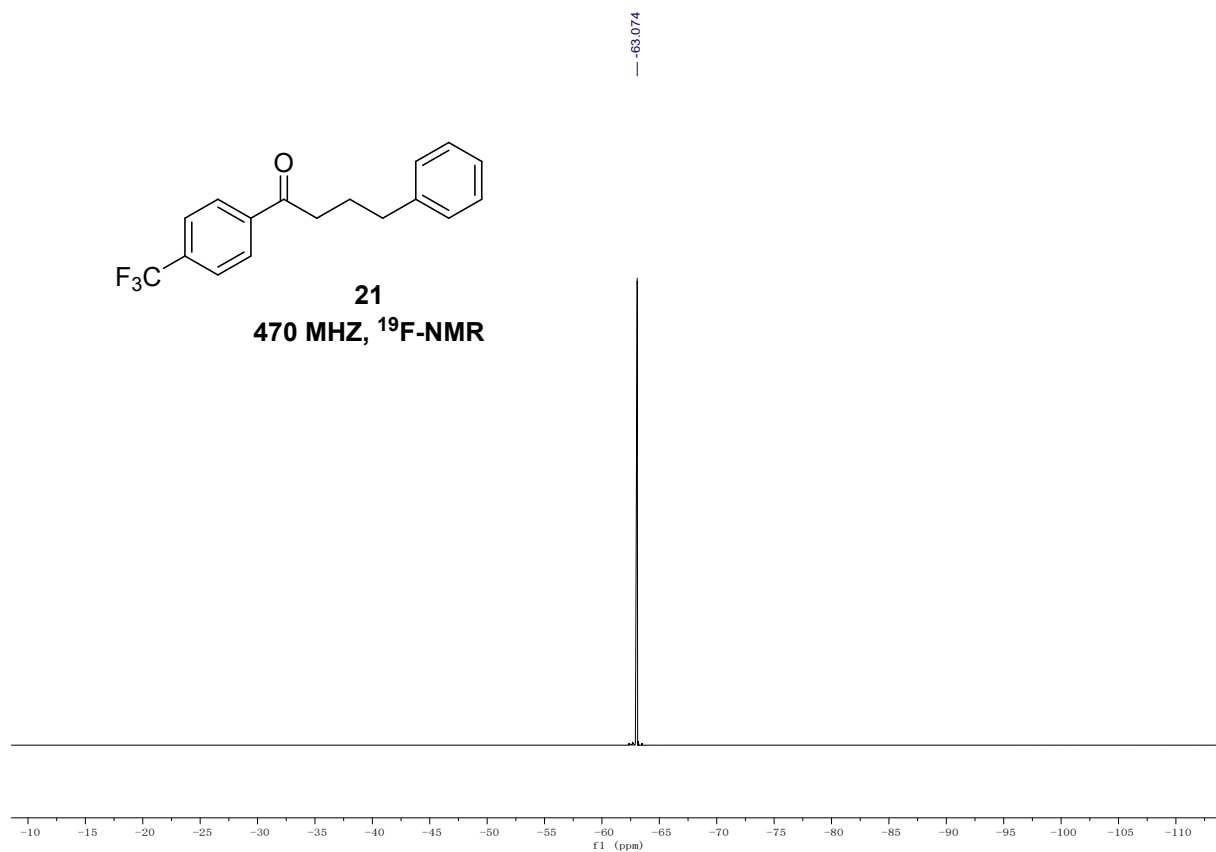


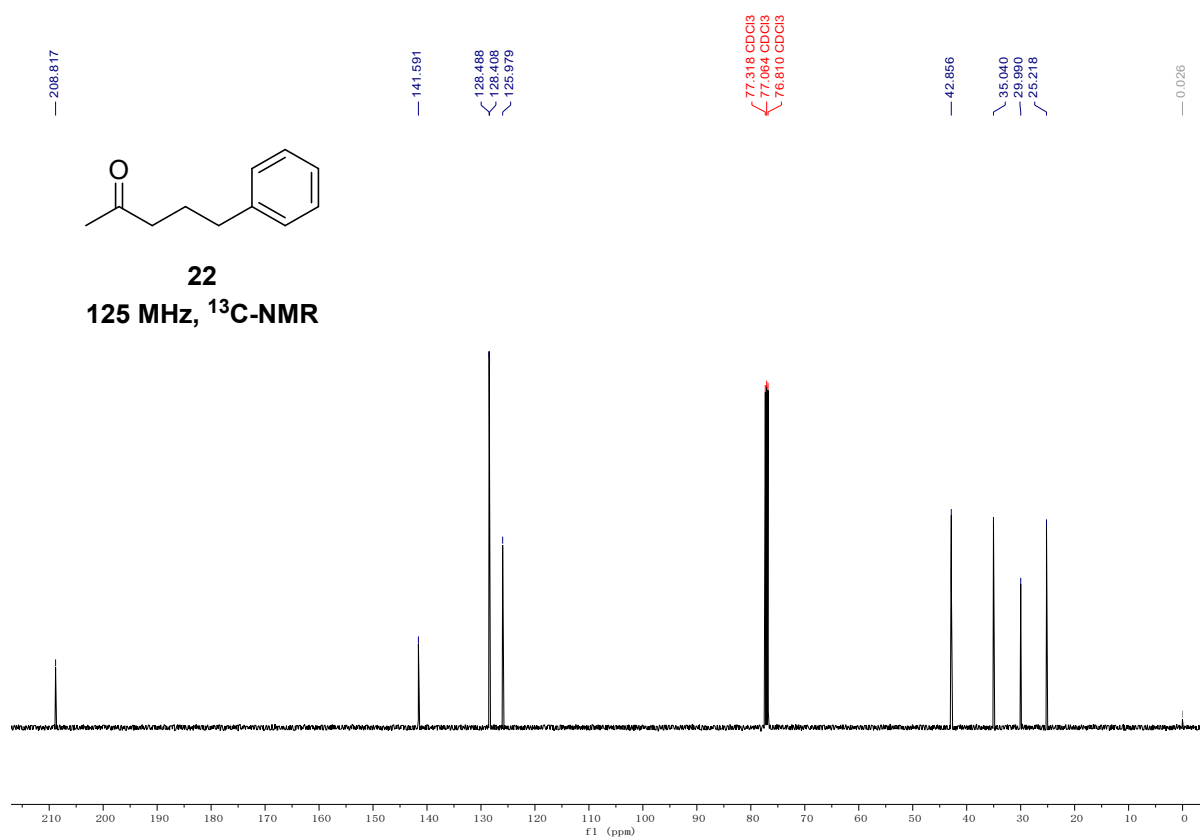


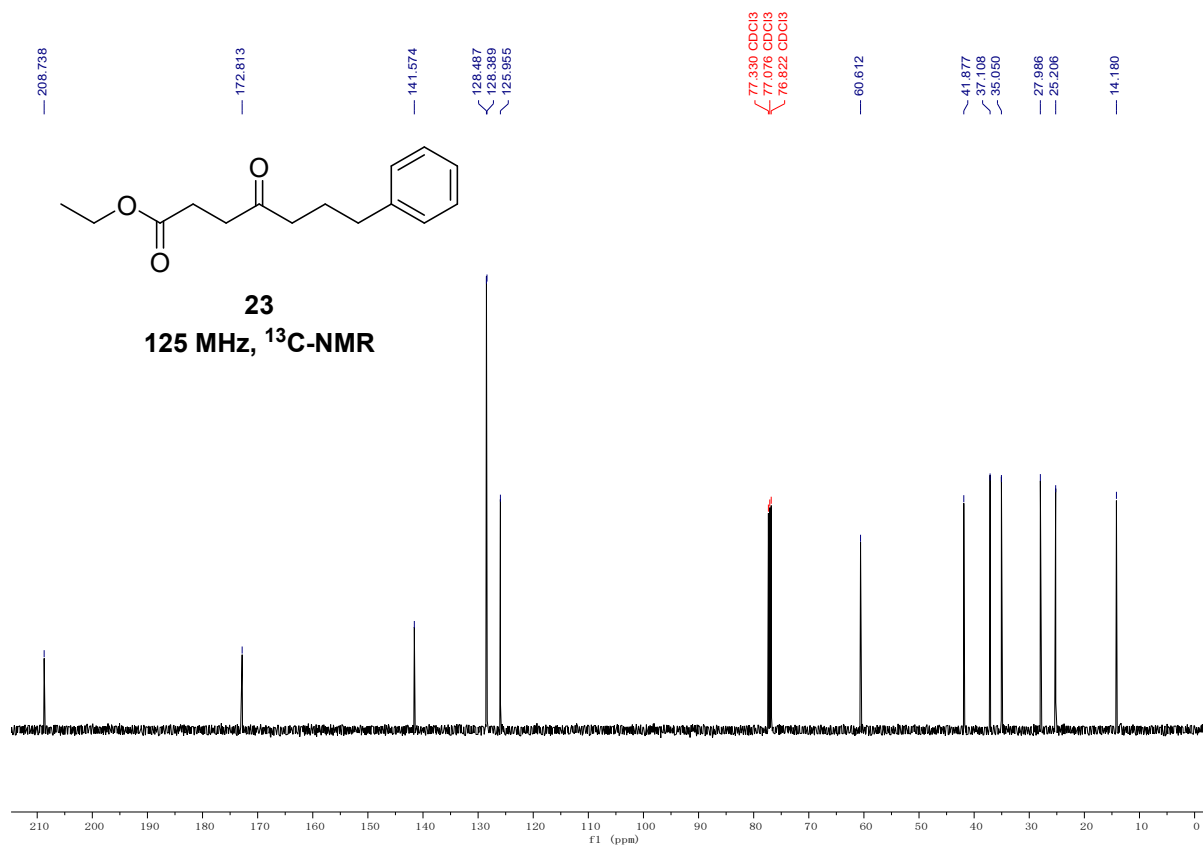
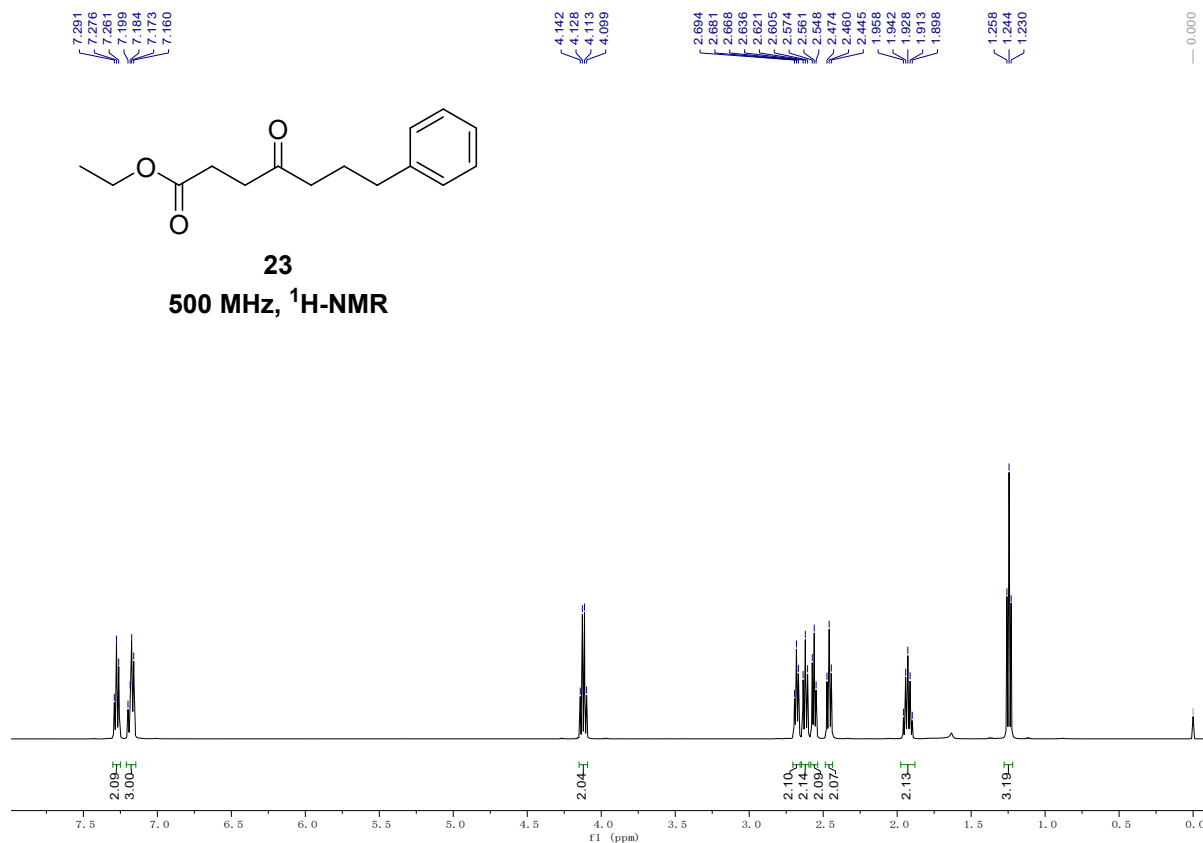


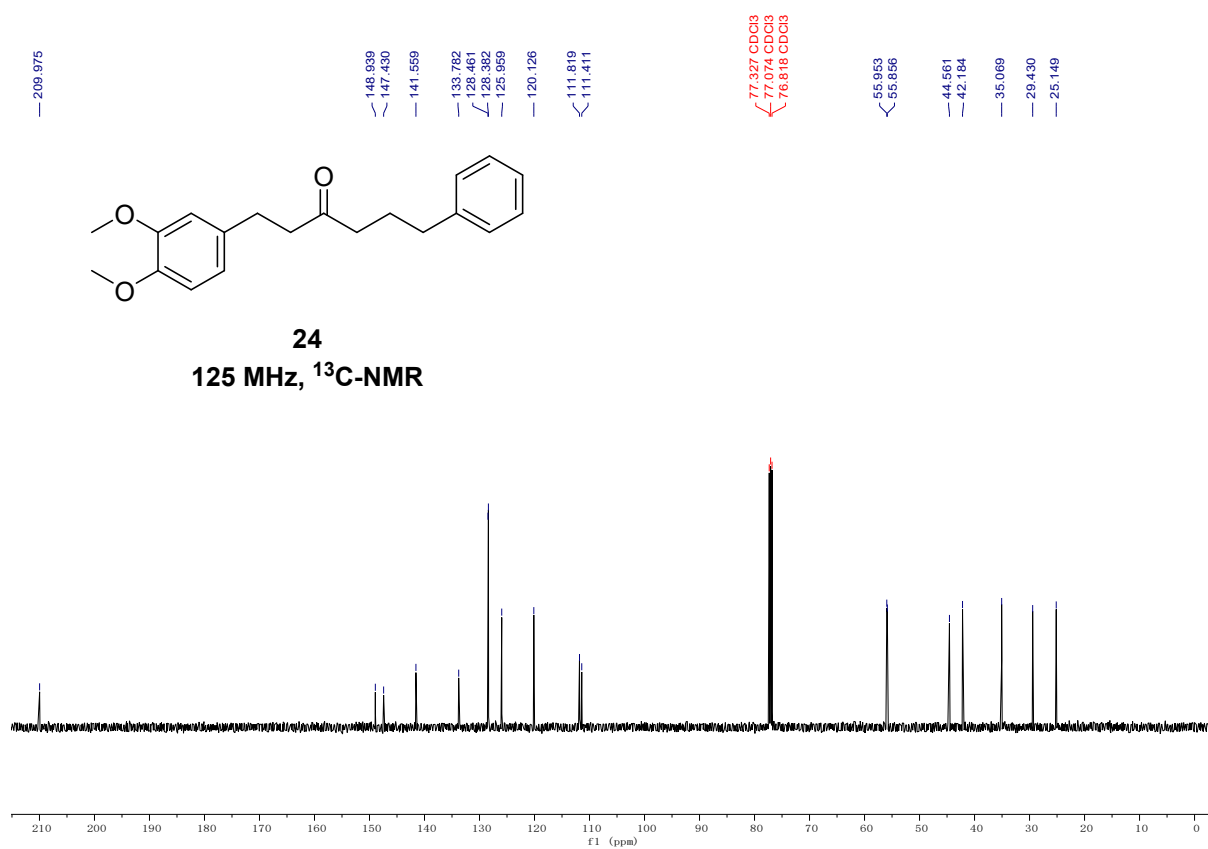
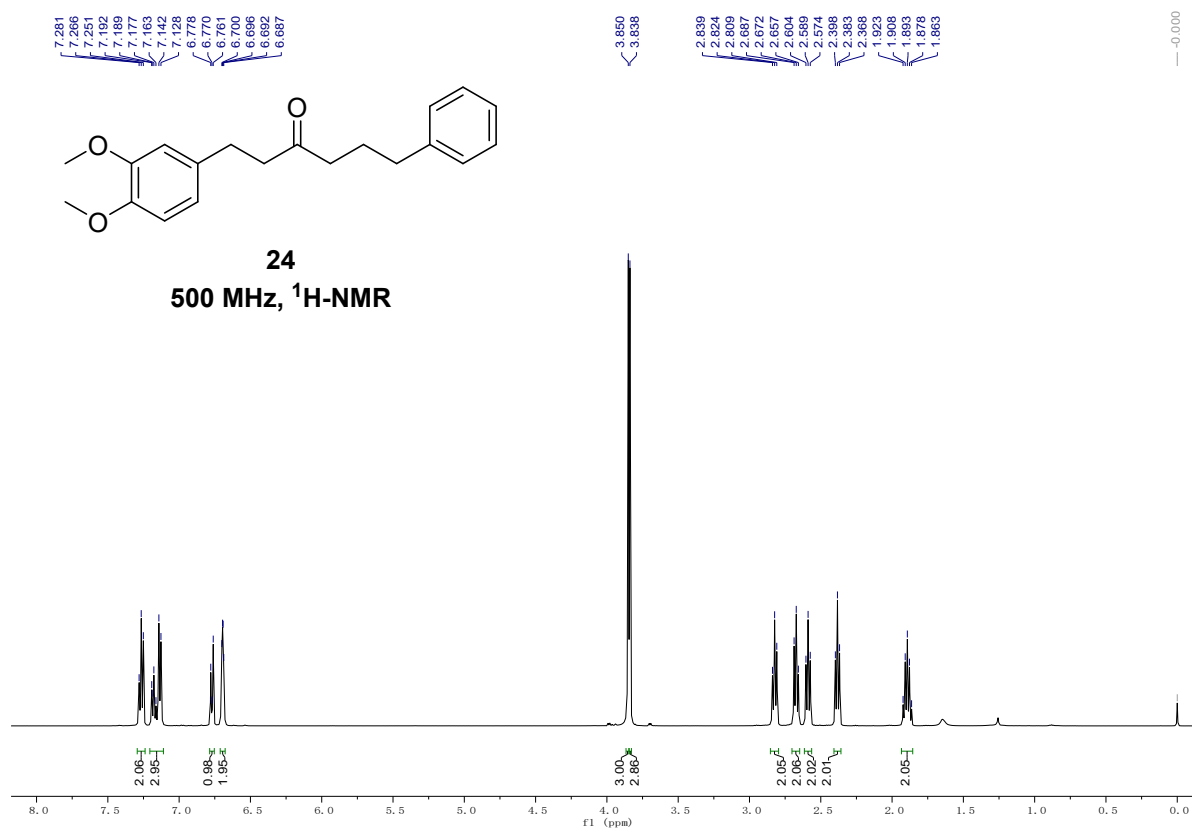


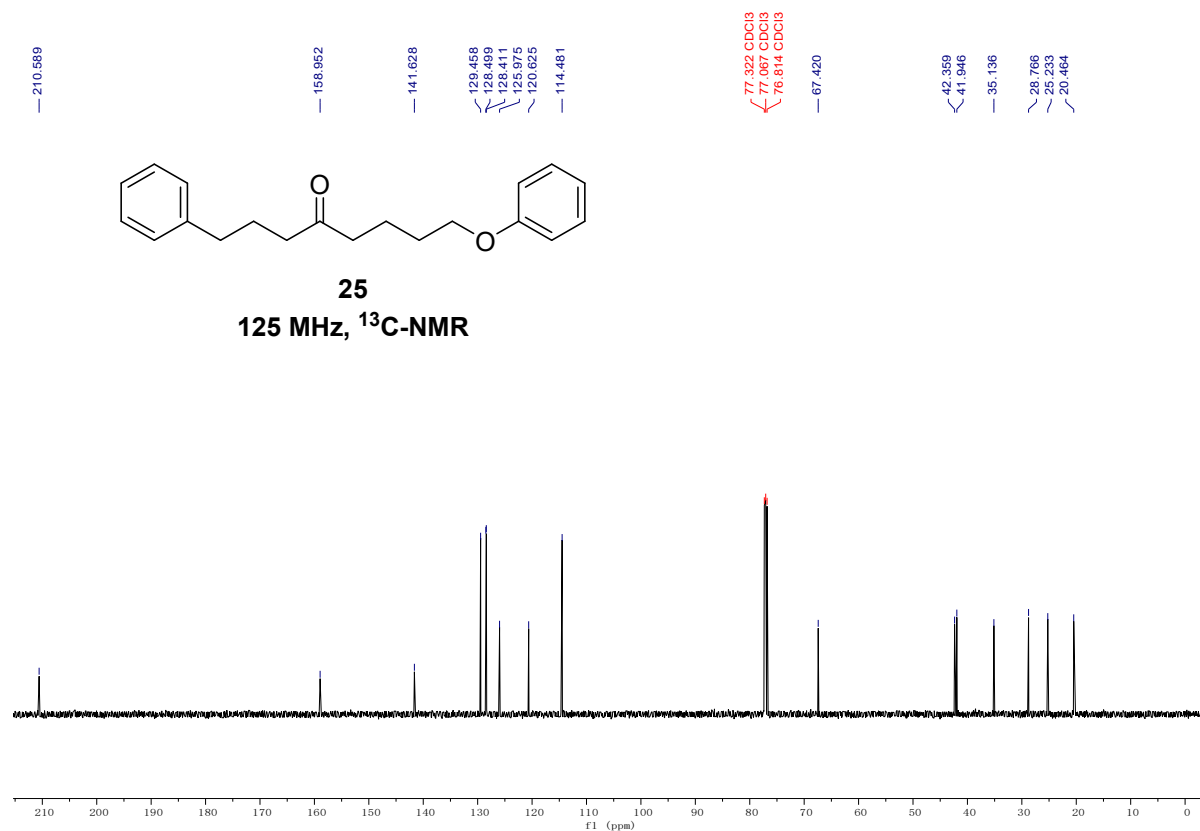
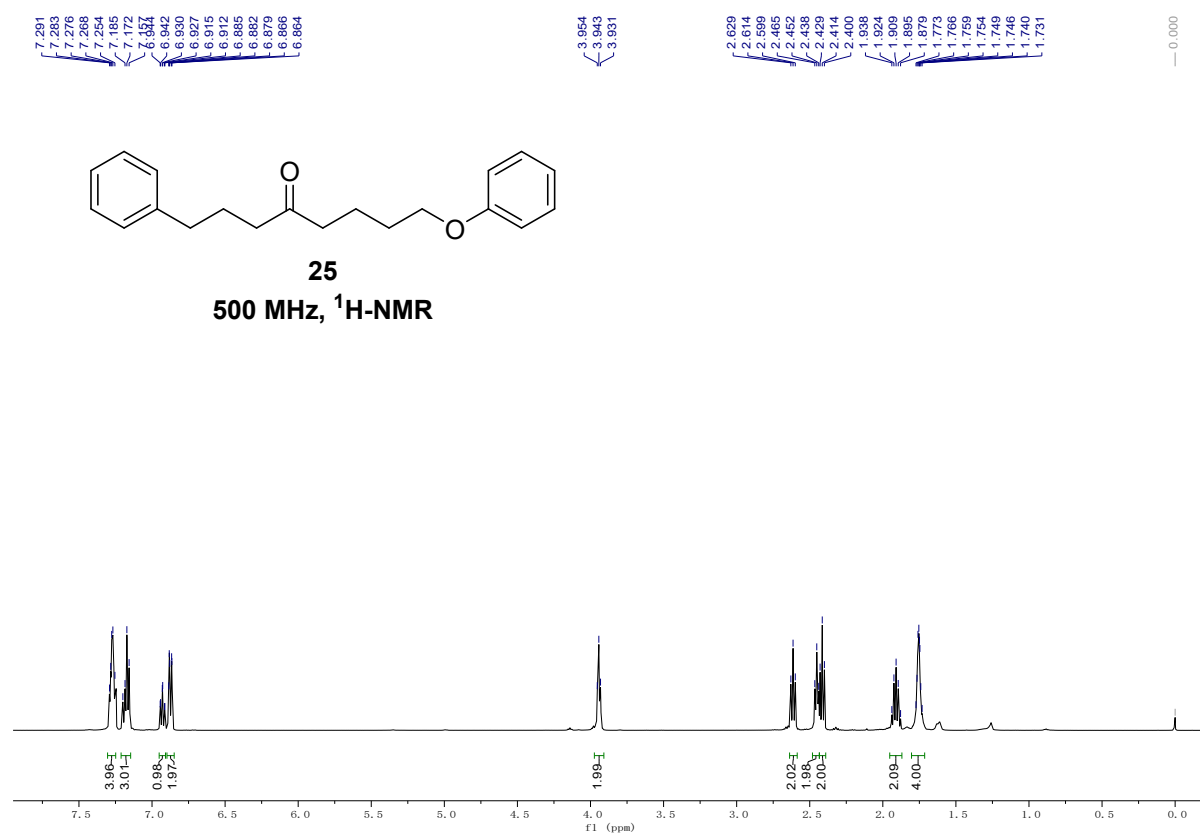


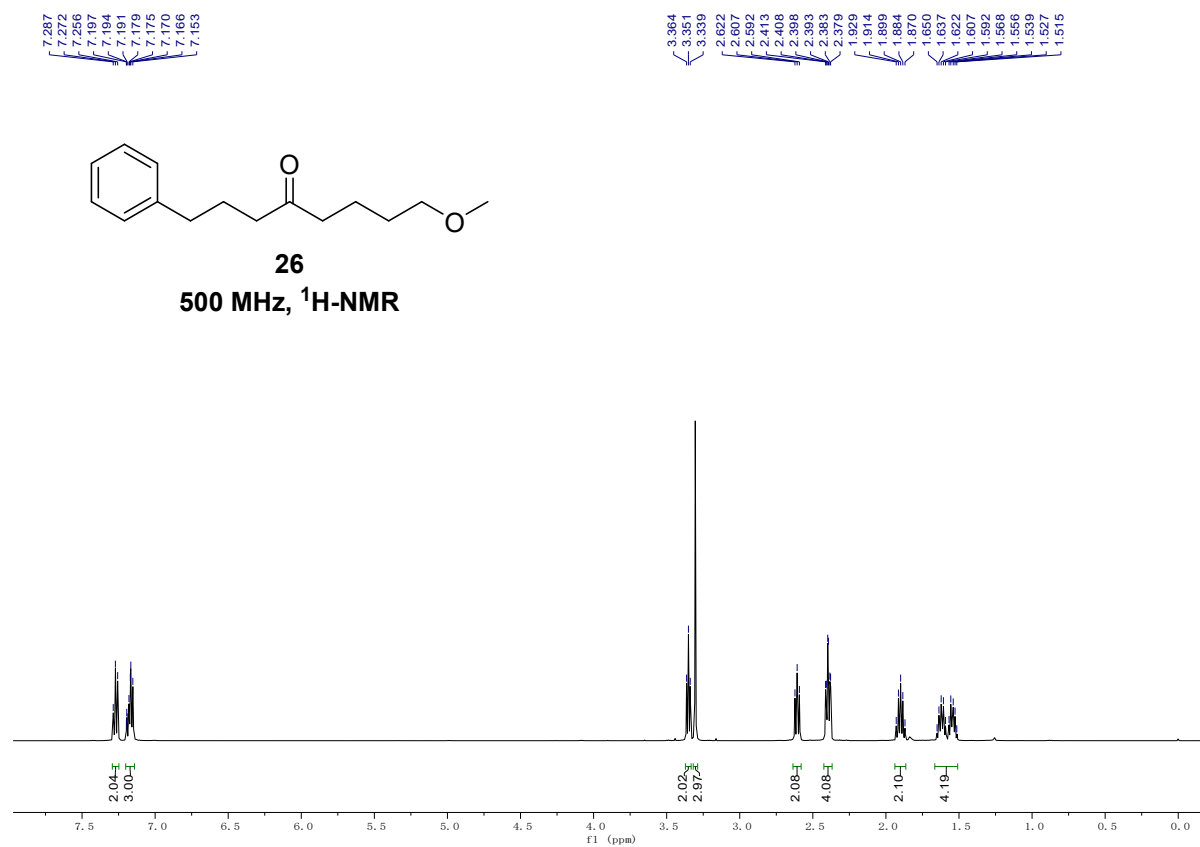


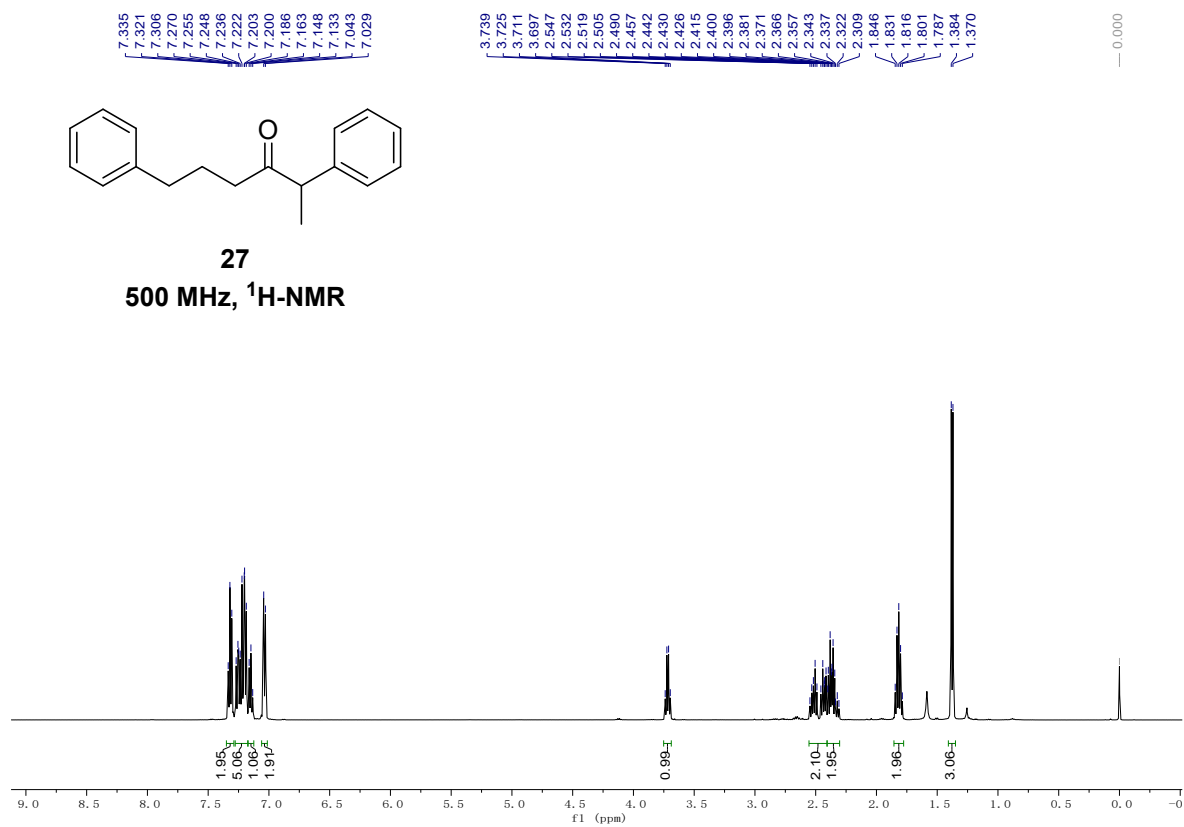
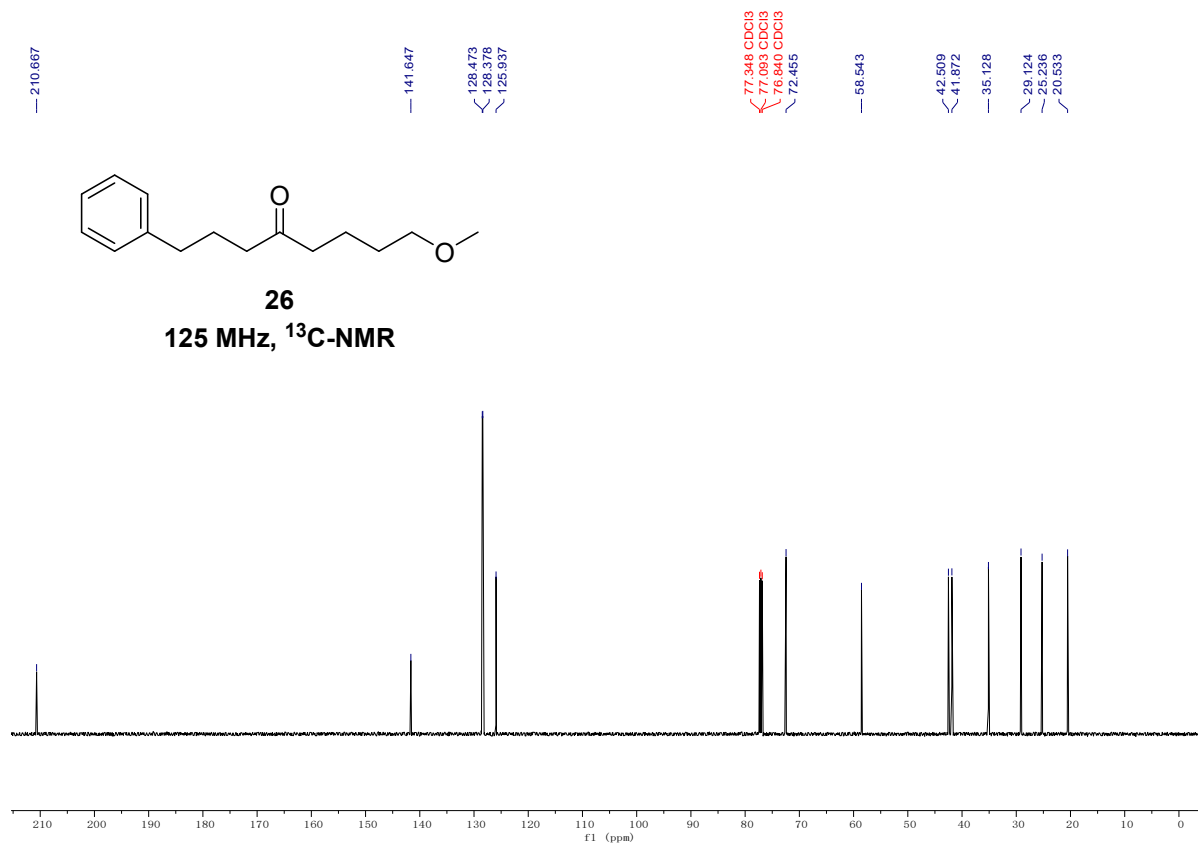


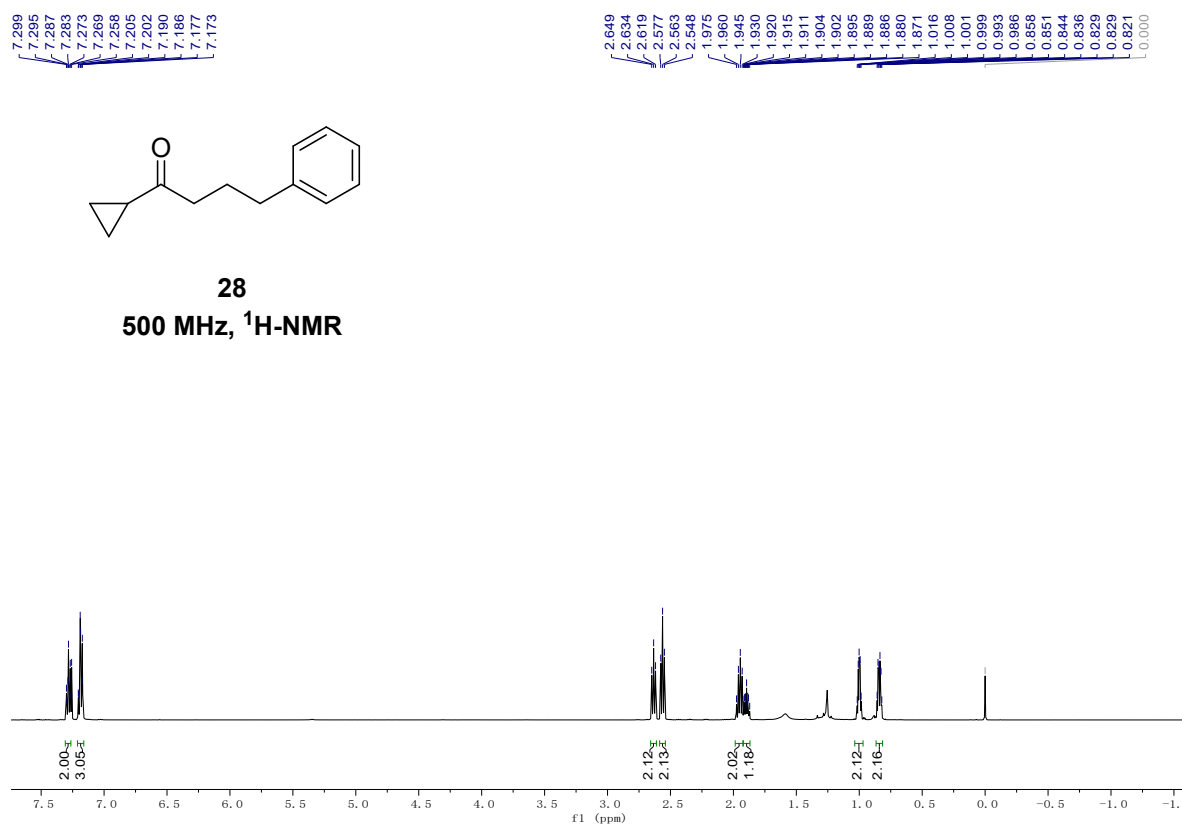
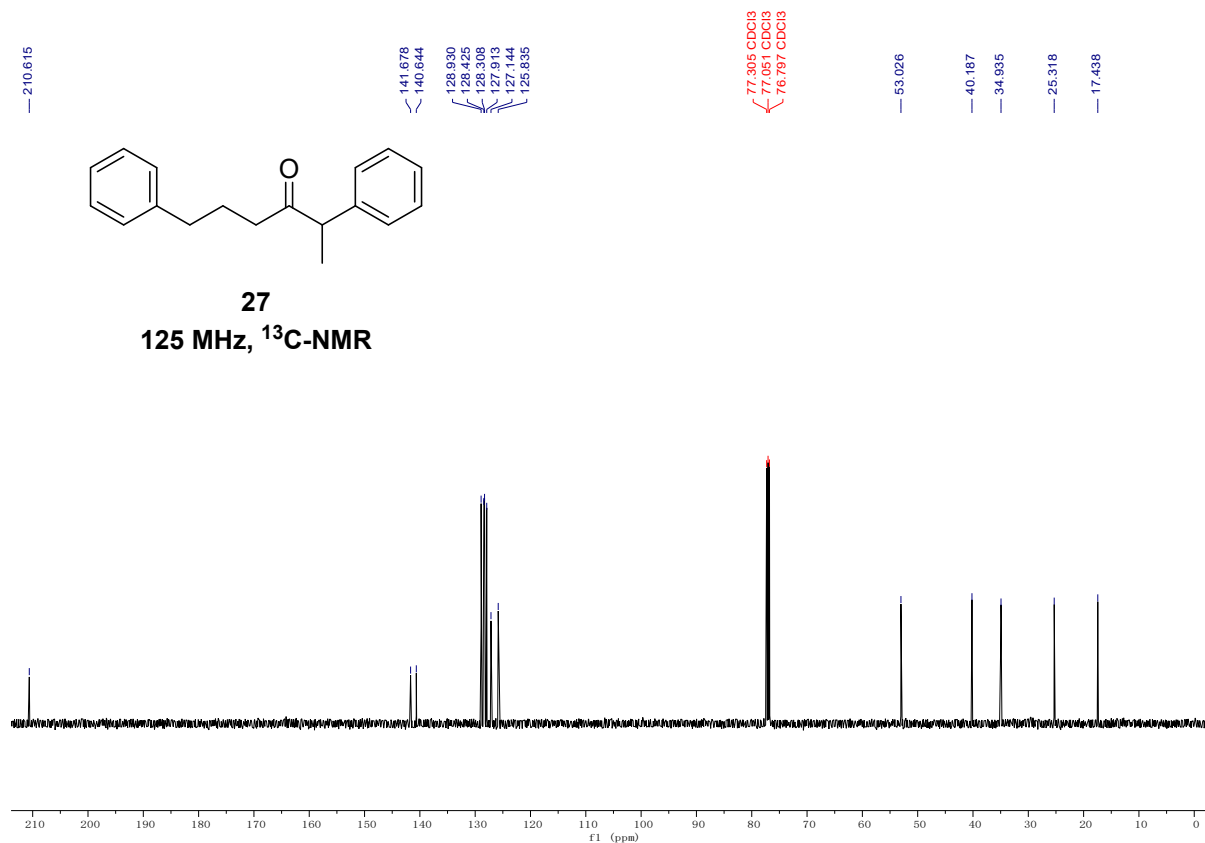


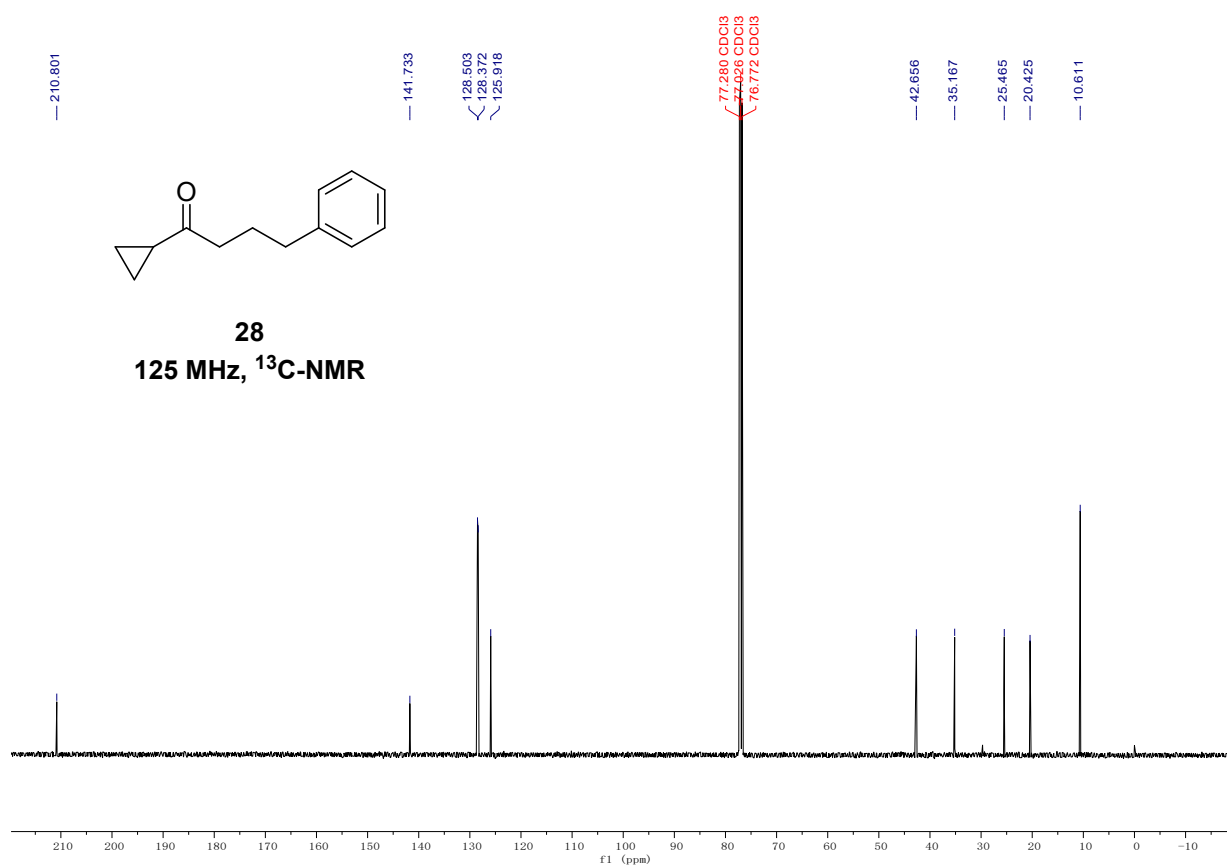


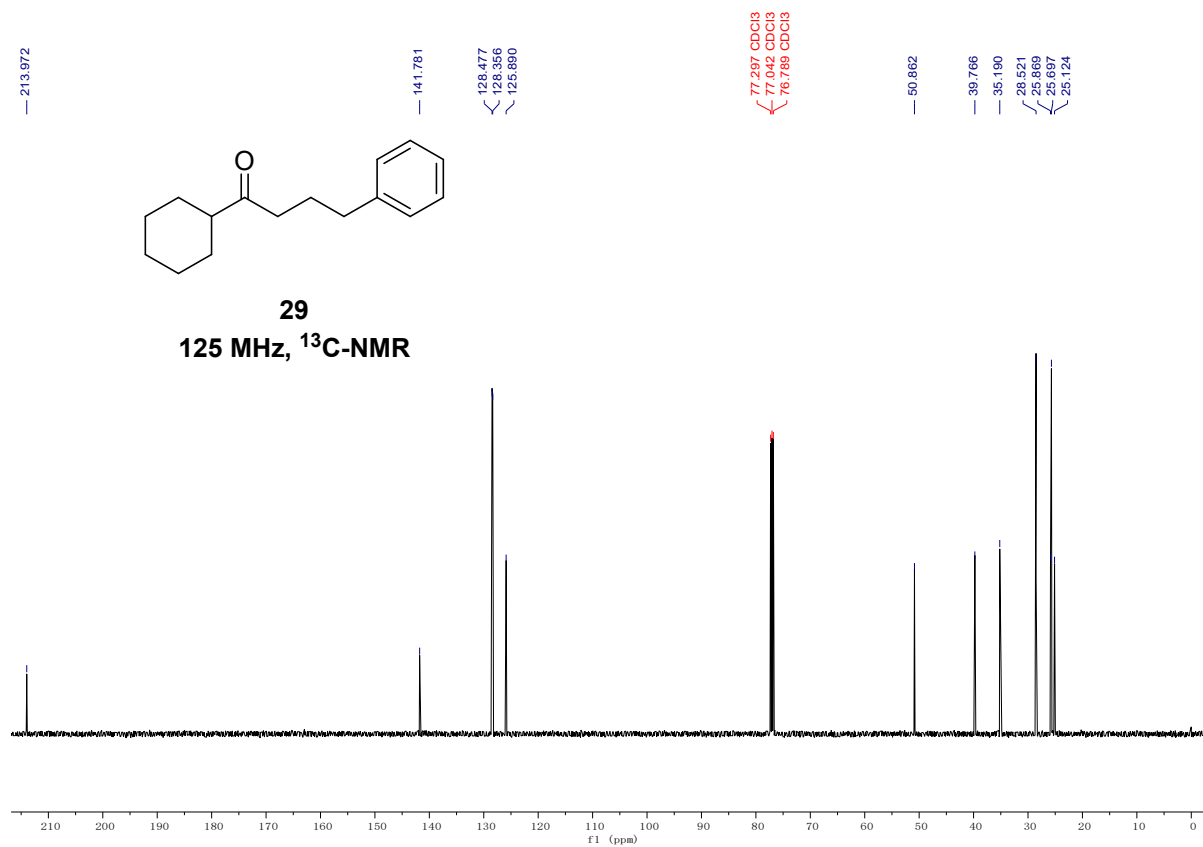
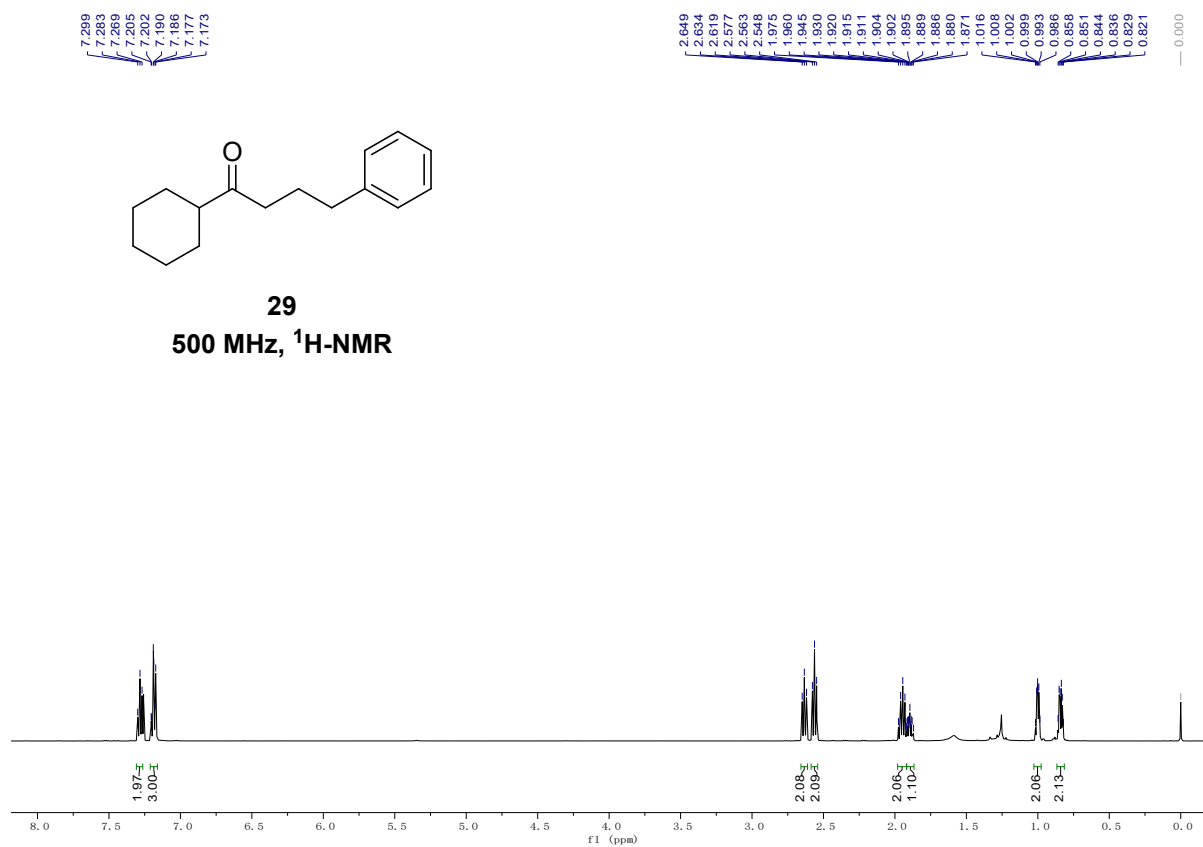


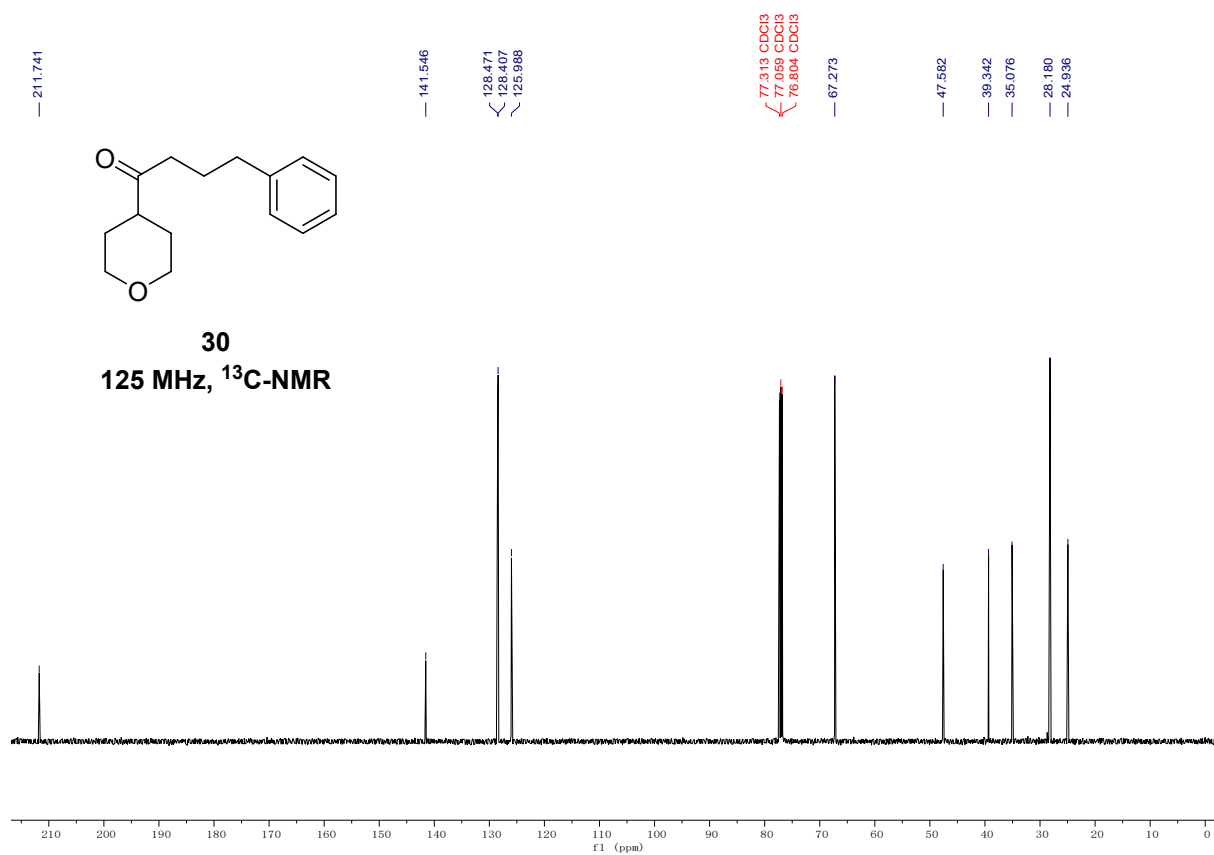
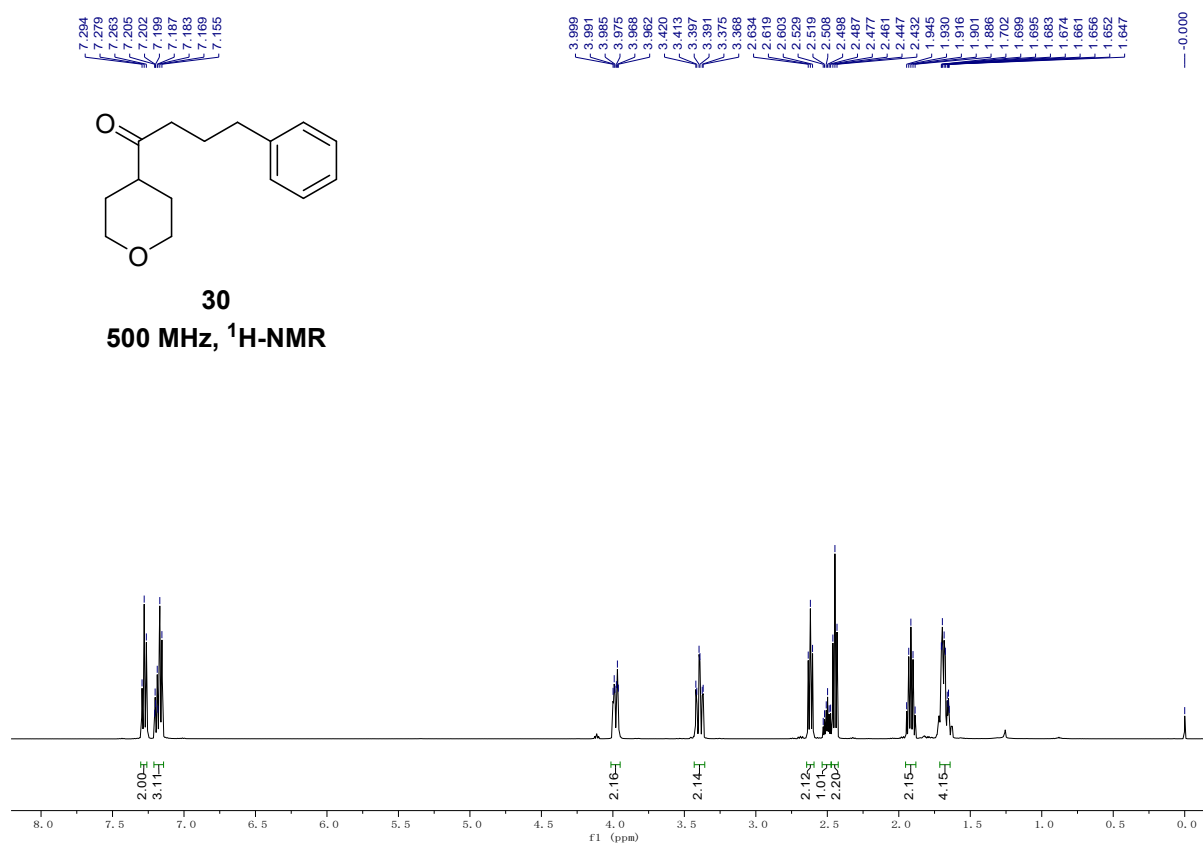


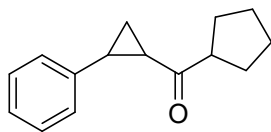




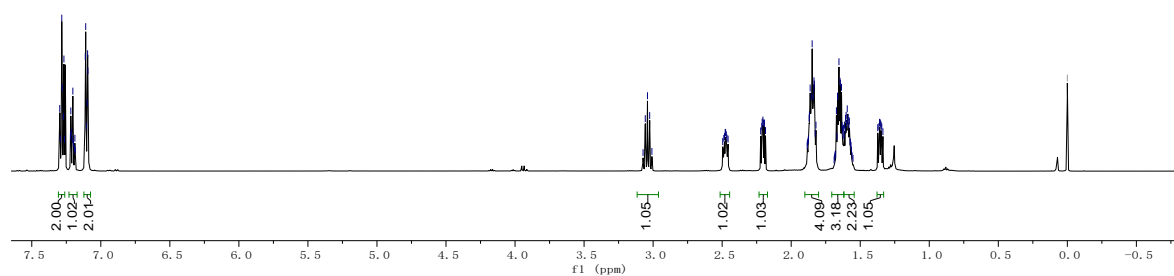








31
500 MHz, ^1H -NMR



211.159

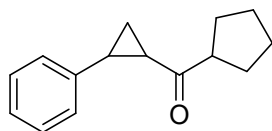
140.630

128.493
126.433
126.146

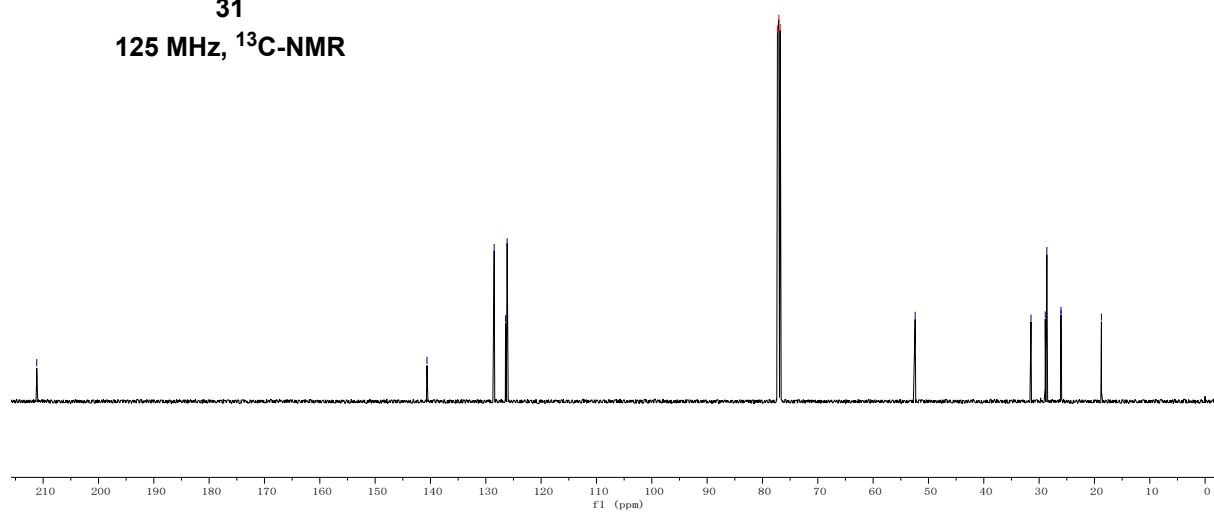
77.298 CDCl3
77.044 CDCl3
76.790 CDCl3

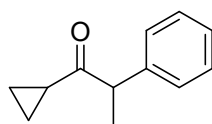
52.408

31.473
28.898
28.605
26.058
26.032
18.743

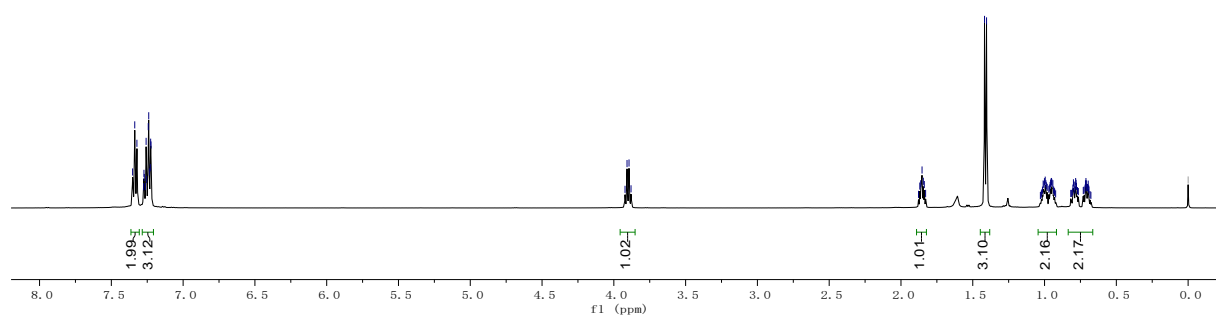


31
125 MHz, ^{13}C -NMR





32
500 MHz, ^1H -NMR



— 210.863

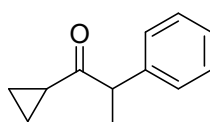
— 140.934

128.847
128.087
126.990

77.283 CDCl₃
77.030 CDCl₃
76.775 CDCl₃

— 53.746

19.719
17.598
11.332
11.260



32
125 MHz, ^{13}C -NMR

