Supporting Information

Copper-catalyzed radical ring-opening halogenation with HX

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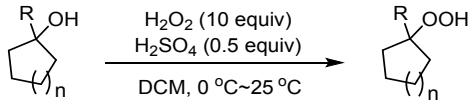
1. General Information

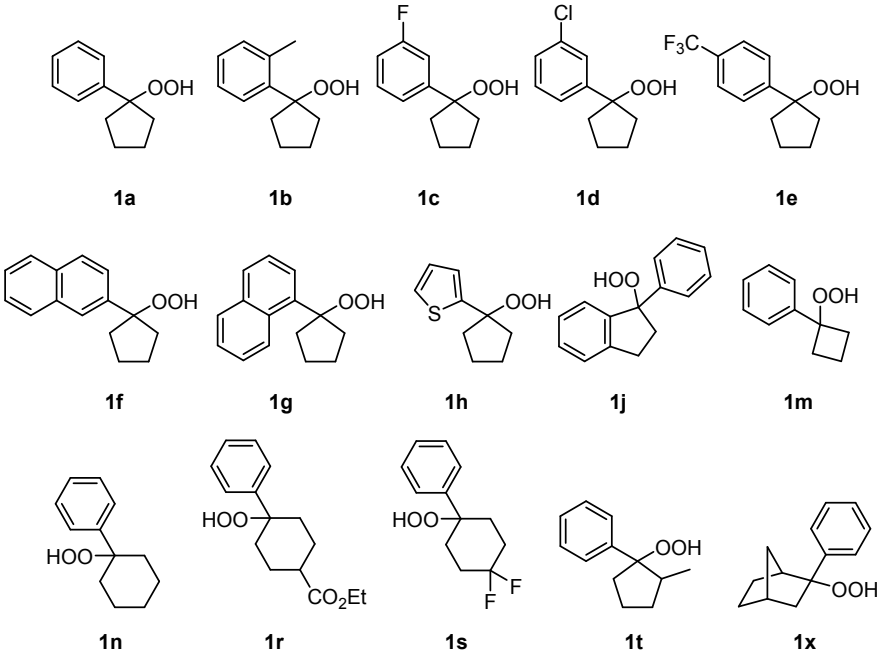
Unless otherwise noted, reagents and solvents were obtained from commercial suppliers and were used without further purification. The concentration of HX (aq) are HF (49%), HCl (36%), HBr (40%) and HI (55%-58%). All catalytic reactions were carried out under nitrogen in Schlenk-tube. Analytical TLC: aluminum backed plates pre-coated (0.25 mm) with Merck Silica Gel 60F-254. Column chromatography purifications were carried out using silica gel. Melting points were measured using open glass capillaries in a SGW® X-4A apparatus. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz spectrometer at ambient temperature. Coupling constants are reported in Hz with multiplicities denoted as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Infrared spectra were recorded on a Bruker V 70 and only major peaks were reported in cm^{-1}. HRMS were obtained on a WATERS I-Class VION IMS Q-Tof with an ESI source.

2. Starting Materials

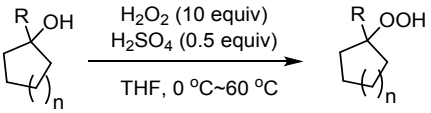
2.1 General Procedure A for the Synthesis of Hydroperoxides 1a-1h, 1j, 1m-1n, 1r-1t, 1x [1]



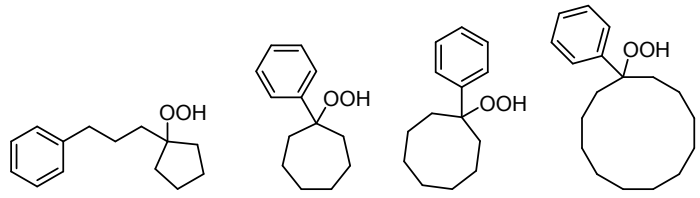
In a 25 mL reaction tube was added a solution of H_2O_2 (1.7 mL, 30 mmol, 30% wt in H_2O), and conc. H_2SO_4 (0.1 mL, 1.5 mmol), then added a solution of alcohol (3 mmol) in DCM (0.5 mL) at 0 °C. The reaction mixture was stirred vigorously for 12 h at room temperature. The aqueous layer was extracted with DCM (3 × 10 mL). The combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated to afford residue, which was purified by column chromatography on silica gel to give the target products **1a-1h**, **1j**, **1m-1n**, **1r-1t**, **1x**.



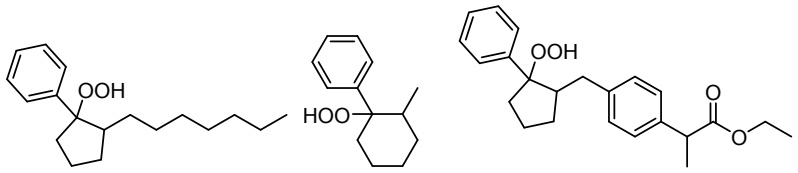
2.2 General Procedure B for the Synthesis of Hydroperoxides 1i, 1o-1q, 1u-1w [1]



In a 25 mL reaction tube was added a solution of H_2O_2 (1.7 mL, 30 mmol, 30% wt in H_2O), and conc. H_2SO_4 (0.1 mL, 1.5 mmol), then added a solution of alcohol (3 mmol) in THF (0.5 mL) at 0 °C. Then the reaction mixture was stirred vigorously for 12 h at 60 °C. The aqueous layer was extracted with DCM (3 × 10 mL). The combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated to afford residue, which was purified by column chromatography on silica gel to give the target products **1i**, **1o-1q**, **1u-1w**.

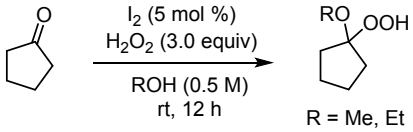


1i **1o** **1p** **1q**

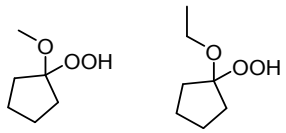


1u **1v** **1w**

2.3 General Procedure C for the Synthesis of Hydroperoxides 1k and 1l [2]



In a 25 mL reaction tube was added a solution of ketone (10 mmol) in methanol or ethanol (0.5 M), then added H_2O_2 (1.7 mL, 30 mmol, 30% wt in H_2O) and I_2 (0.5 mol, 5 mol %). The reaction mixture was stirred vigorously for 12 h at room temperature. Concentrated the reaction solution, and then the aqueous layer was extracted with DCM (3 × 10 mL). The combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated to afford residue, which was purified by column chromatography on silica gel to give the target products **1k** and **1l.**



1k **1l**

2.4 Safety notes of Hydroperoxides

All cycloalkyl hydroperoxides heated and concentrated by rotovap at below 30 °C and stored under –20 ºC. We have never experienced a safety problem with these materials.

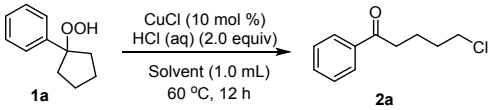
3. Optimization of Reaction Conditions

3.1 General Procedure for Halogenation of Cyclopentyl Hydroperoxide 1a

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added catalyst and " X " source (0.4 mmol, 2.0 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclopentyl hydroperoxide **1a** (0.2 mmol, 1.0 equiv) in solvent (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred. After the reaction completed, the reaction mixture was diluted with EtOAc (5.0 mL) and H_2O (5.0 mL). The organic layer was washed with saturated brine (3 × 5 mL) and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 60:1) to give the corresponding target products.

3.2 Optimization of Chlorination of Cyclopentyl Hydroperoxide 1a

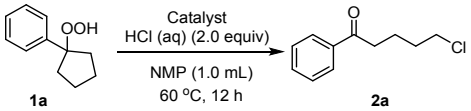
Screening of Solvent



Entry	Solvent	Yield[a] (%)
1	**NMP**	**85**
2	DMSO	65
3	DMF	81
4	MeOH	60
5	MeCN	79
6	THF	75
7	DCM	17
8	toluene	30
9	H_2O	16

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), CuCl (10 mol %), HCl (aq) (0.4 mmol, 2.0 equiv), and solvent (1.0 mL) at 60 °C for 12 h under N_2. Isolated yields.

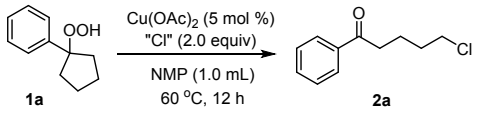
Screening of Catalyst



Entry	Catalyst (mol %)	Yield[a] (%)
1	CuCl (10)	85
2	CuBr (10)	87
3	**$Cu(OAc)_2$ (10)**	**92**
4	CuTC (10)	80
5	$Cu(CH_3CN)_4PF_6$ (10)	85
6	$Fe(OTf)_2$ (10)	88
7	$Fe(OAc)_2$ (10)	66
8	$CoCl_2$ (10)	trace
9	$NiCl_2$ (10)	trace
10	$PdCl_2$ (10)	trace
11	-	trace

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), catalyst (10 mol %), HCl (aq) (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at 60 °C for 12 h under N_2. Isolated yields.

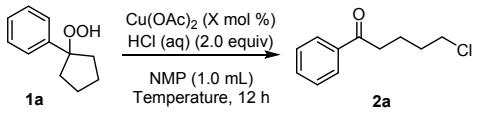
Screening of Chloride Source



Entry	Chloride Source	Yield[a] (%)
1	$MgCl_2$	88
2	NaCl	trace
3	NH_4Cl	15
4	**HCl (aq)**	**92**

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), $Cu(OAc)_2$ (10 mol %), chloride source (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at 60 °C for 12 h under N_2. Isolated yields.

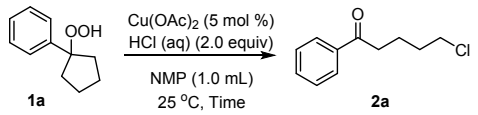
Screening of Temperature and Amount of Catalyst



Entry	Catalyst (mol %)	Temperature (°C)	Yield[a] (%)
1	$Cu(OAc)_2$ (10)	60	92
2	$Cu(OAc)_2$ (10)	40	89
3	$Cu(OAc)_2$ (10)	25	89
4	**$Cu(OAc)_2$ (5)**	**25**	**90**(95)[b]
5	$Cu(OAc)_2$ (2.5)	25	83

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), $Cu(OAc)_2$ (X mol %), HCl (aq) (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at X °C for 12 h under N_2. Isolated yields. [b]The cyclopentyl silyl peroxide was used.

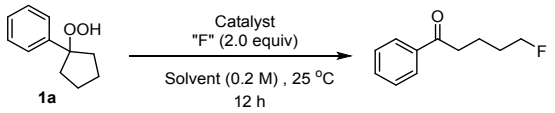
Screening of Time



Entry	Time (h)	Yield[a] (%)
1	12	92
1	**2**	**90**
2	1	88
3	0.5	67

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), $Cu(OAc)_2$ (5 mol %), HCl (aq) (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at 25 °C for X h under N_2. Isolated yields.

3.3 Optimization of Fluorination of Cyclopentyl Hydroperoxide 1a



Entry	Catalyst (mol %)	Fluorinate Source	Additive	Solvent	Yield[a] (%)
1	$Cu(OAc)_2$ (5)	HF (aq)	-	NMP	n.r. (**85**)[b]
2	CuF_2 (100)	-	-	NMP	n.r.
3	$Cu(OAc)_2$ (5)	HF (aq)	18-crown-6	DMF	n.r.
4	$Cu(OAc)_2$ (5)	KF	18-crown-6	DMF	n.r.
5	$Cu(OAc)_2$ (5)	KHF_2	18-crown-6	DMF	n.r.
6	$Cu(OAc)_2$ (5)	TBAF	18-crown-6	DMF	n.r.
7	$AgNO_3$ (10)	SelectF	-	DMF	n.r.

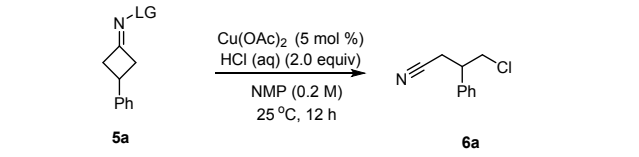
[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), catalyst, fluorinate source (0.4 mmol, 2.0 equiv), and solvent (1.0 mL) at 25 °C for 12 h under N_2. Isolated yields. [b]85% of **1a** was recovered.

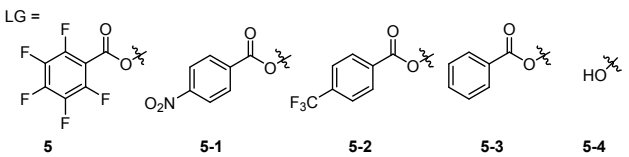
3.4 General Procedure for Halogenation of Cycloketone Oxime Ester 5a

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added catalyst and " X " source (0.4 mmol, 2.0 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cycloketone oxime ester **5a** (0.2 mmol, 1.0 equiv) in solvent (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 ^{o}C for 12 h. After the reaction completed, the reaction mixture was diluted with EtOAc (5.0 mL) and H_2O (5.0 mL). The organic layer was washed with saturated brine (3 × 5 mL) and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 15:1) to give the target products **6a-8a**.

3.5 Optimization of Chlorination of Cycloketone Oxime Ester 5a

Screening of Leaving Group

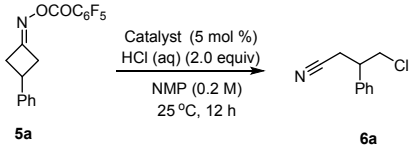




Entry	Leaving Group	Yield[a] (%)
1	**5**	**80**
2	**5-1**	n.r.
3	**5-2**	n.r.
4	**5-3**	n.r.
5	**5-4**	n.r.

[a]Reaction conditions: **5** (0.2 mmol, 1.0 equiv), $Cu(OAc)_2$ (5 mol %), HCl (aq) (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at 25 °C for 12 h under N_2. Isolated yields.

Screening of Catalyst

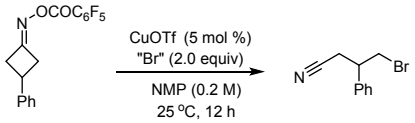


Entry	Catalyst	Yield[a] (%)
1	$Cu(OAc)_2$	80
2	**CuOTf**	**90**

[a]Reaction conditions: **5a** (0.2 mmol, 1.0 equiv), catalyst (5 mol %), HCl (aq) (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at 25 °C for 12 h under N_2. Isolated yields.

3.6 Optimization of Bromination or Iodination of Cycloketone Oxime Ester 5a

Screening of Bromide Source

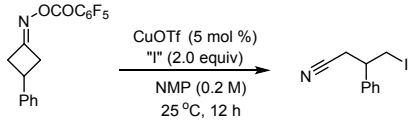


5a **7a**

Entry	Bromide Source	Yield[a] (%)
1	HBr (aq)	n.r.(53)[b]
2	NaBr	trace
3	KBr	trace
4	TBAB	trace
5	**$MgBr_2 \cdot 6H_2O$**	**82**

[a]Reaction conditions: **5a** (0.2 mmol, 1.0 equiv), CuOTf (5 mol %), bromide source (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at 25 °C for 12 h under N_2. Isolated yields. [b]Yield of 3-phenylcyclobutan-1-one.

Screening of Iodide Source



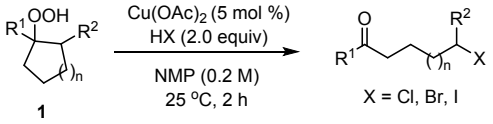
5a **8a**

Entry	Iodide Source	Yield[a] (%)
1	HI (aq)	n.r.(32)[b]
2	NaI	trace
3	KI	trace
4	TBAI	trace
5	**ZnI_2**	**79**

[a]Reaction conditions: **5a** (0.2 mmol, 1.0 equiv), CuOTf (5 mol %), iodide source (0.4 mmol, 2.0 equiv), and NMP (1.0 mL) at 25 °C for 12 h under N_2. Isolated yields. [b]Yield of 3-phenylcyclobutan-1-one.

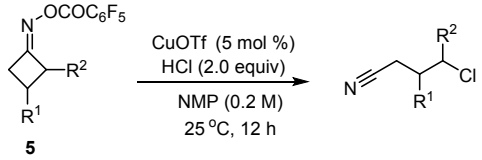
4. Representative Procedure for Schemes 2-4

4.1 Representative Procedure for Halogenation of Cycloalkyl Hydroperoxides 1



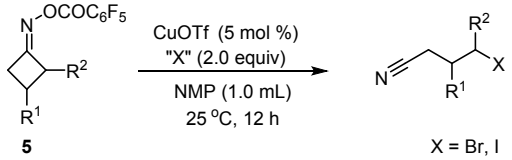
A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added $Cu(OAc)_2$ (0.01mmol, 5 mol %), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cycloalkyl hydroperoxide **1** (0.2 mmol, 1.0 equiv), and HCl (aq) or HBr (aq) or HI (aq) (0.4 mmol, 2.0 equiv) in NMP (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 °C for 2 h. After the reaction completed, the reaction mixture was diluted with EtOAc (5.0 mL) and H_2O (5.0 mL). The organic layer was washed with saturated brine (3 × 5 mL) and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 60:1) to give the target products **2**, **3** and **4** in yields as listed in **Scheme 2** and **Scheme 3**.

4.2 Representative Procedure for Chlorination of Cycloketone Oxime Esters 5



A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added CuOTf (0.01mmol, 5 mol %), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cycloketone oxime esters **5** (0.2 mmol, 1.0 equiv), and HCl (aq) (0.4 mmol, 2.0 equiv) in NMP (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 °C for 12 h. After the reaction completed, the reaction mixture was diluted with EtOAc (5.0 mL) and H_2O (5.0 mL). The organic layer was washed with saturated brine (3 × 5 mL) and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1) to give the target products **6** in yields as listed in **Scheme 4.**

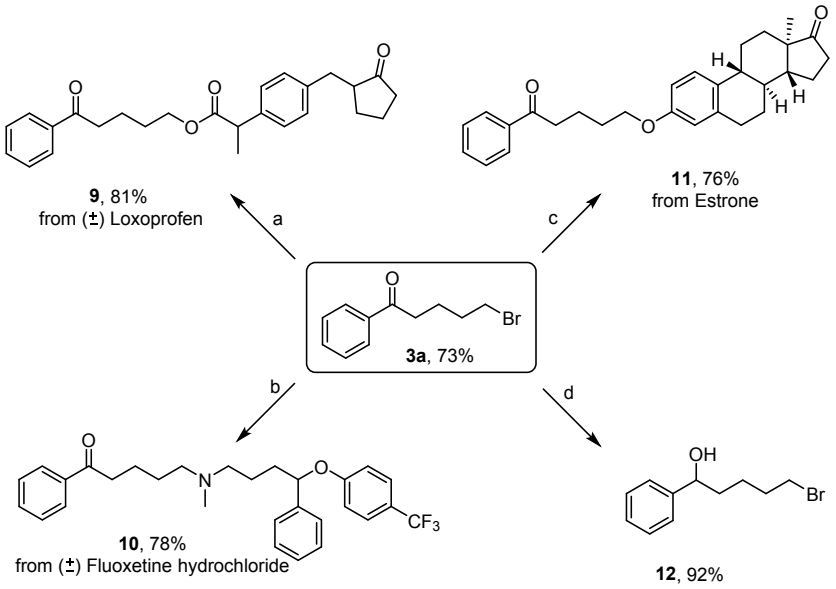
4.3 Representative Procedure for Bromination or Iodinaton of Cycloketone Oxime Esters 5



A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added CuOTf (0.01mmol, 5 mol %) and $MgBr_2 \cdot 6H_2O$ or ZnI_2 (0.4 mmol, 2.0 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cycloketone oxime esters **5** (0.2 mmol, 1.0 equiv) in NMP (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 °C for 12 h. After the reaction completed, the reaction mixture was diluted with EtOAc (5.0 mL) and H_2O (5.0 mL). The organic layer was washed with saturated brine (3

× 5 mL) and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1) to give the target products **7** and **8** in yields as listed in **Scheme 4**.

5. Procedures for Derivatization of 3a



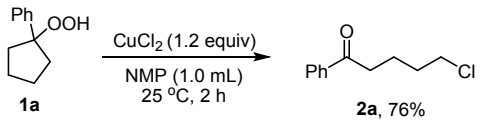
Reaction conditions: (a): A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added **3a** (0.2 mmol, 1.0 equiv), K_2CO_3 (0.6 mmol, 3.0 equiv) and Loxoprofen (0.3 mmol, 1.5 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, DMF (1 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 90 °C for 12 h. After the reaction completed, the reaction mixture was diluted with EtOAc (5.0 mL) and H_2O (5.0 mL). The organic layer was washed with saturated brine (3 × 5 mL) and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 7:1) to give the target product **9** in 81% yield.

(b) and (c): A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added **3a** (0.2 mmol, 1.0 equiv), K_2CO_3 (0.6 mmol, 3.0 equiv) and Fluoxetine hydrochloride or Estrone (0.3 mmol, 1.5 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, Acetone (1 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 70 °C for 12 h. After the reaction completed, the reaction mixture filtrated and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1) to give the target product **10** in 78% yield or **11** in 76% yield.

(d): A 10 mL oven-dried reaction tube equipped with a magnetic stirrer was added a solution of **3a** (0.2 mmol, 1.0 equiv) in MeOH (4 mL), then was added $NaBH_4$ (1.0 mmol, 5.0 equiv) slowly at 0 °C. The reaction mixture was stirred until **3a** completely converted. After that 4 mL (1 N HCl) and 20 mL H_2O was added, the water layer was extracted with DCM (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1) to give the target product **12** in 92% yield.

6. Investigation of the Reaction Mechanism

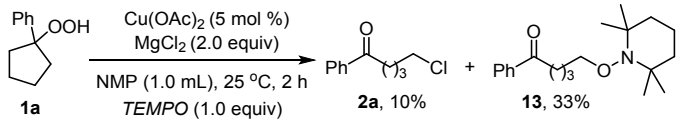
6.1 Source of Halogen Experiment



1a **2a**, 76%

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added $CuCl_2$ (0.24 mmol, 1.2 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclopentyl hydroperoxides **1a** (0.2 mmol, 1.0 equiv) in NMP (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 °C for 2 h.

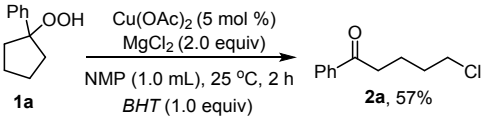
6.2 Radical Trapping Experiment



1a **2a**, 10% **13**, 33%

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added $Cu(OAc)_2$ (0.01mmol, 5 mol %), $MgCl_2$ (0.4 mmol, 2.0 equiv) and TEMPO (0.2 mmol, 1.0 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclopentyl hydroperoxides **1a** (0.2 mmol, 1.0 equiv) in NMP (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 °C for 2 h. The yield of **2a** to 10%, along with TEMPO adduct **13** isolated in 33% yield. These results indicate that a radical intermediate might be involved in this transformation.

6.3 Radical Inhibiting Experiment



1a **2a**, 57%

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added $Cu(OAc)_2$ (0.01mmol, 5 mol %), $MgCl_2$ (0.4 mmol, 2.0 equiv) and BHT (0.2 mmol, 1.0 equiv), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclopentyl hydroperoxides **1a** (0.2 mmol, 1.0 equiv) in solvent (1.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 °C for 2 h. The yield of **2a** was reduced to 57% yield. This result indicates that the reaction might proceed via a radical pathway.

6.4 Iodine clock reaction

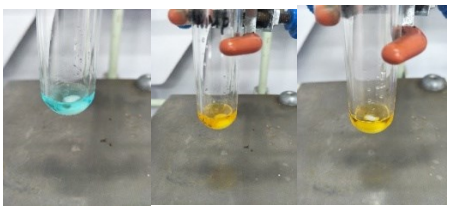


Figure 1. Left: $Cu(OAc)_2$ + NMP; Mid: $Cu(OAc)_2$ + NMP + HCl; Right: $CuCl_2$ + NMP

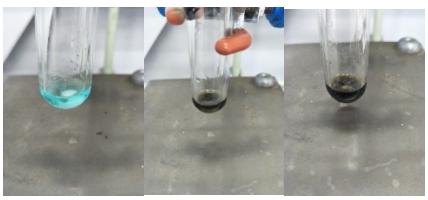
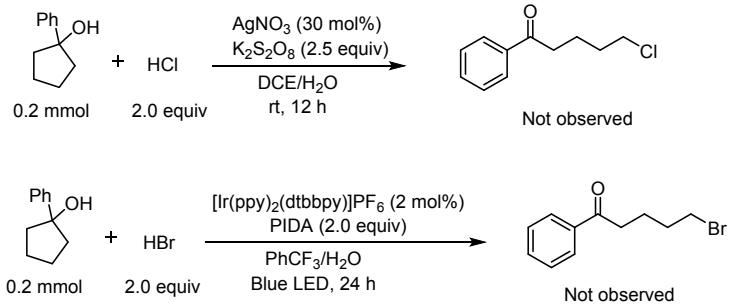


Figure 2. Left: $Cu(OAc)_2$ + NMP; Mid: $Cu(OAc)_2$ + NMP + HBr; Right: $CuBr_2$ + NMP

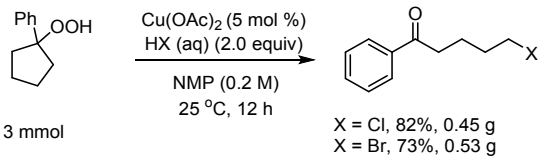
In **Figure 1**. It can be observed that when $Cu(OAc)_2$ and NMP was mixed together, the reaction mixture is light blue (Left). When HCl(aq.) was added, the color was quickly changed to yellow (Mid), which is same as the mixture of $CuCl_2$ and NMP. These results indicate that the anion exchange occurred. In **Figure 2**.The similar phenomenon were observed for HBr(aq.).

These results confirmed to a certain extent that anion exchange of $Cu(OAc)_2$ with HX (aq). We believe that the exact initiating catalyst was Cu(I)X species, which is afforded through disproportionation.

6.5 Ring-opening halogenation of cycloalkyl alcohols with HX (aq)

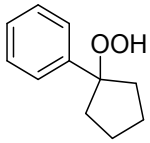


7.1 Large-Scale Synthesis

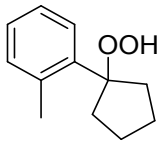


A 100 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added $Cu(OAc)_2$ (0.15 mmol, 5 mol %), then the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclopentyl hydroperoxide **1a** (3 mmol, 1.0 equiv), and HCl (aq) or HBr (aq) (6 mmol, 2.0 equiv) in NMP (15.0 mL) was added by syringe under nitrogen atmosphere. The tube was then sealed and mixture was stirred at 25 °C for 12 h. After the reaction completed, the reaction mixture was diluted with EtOAc (20 mL) and H_2O (20 mL). The organic layer was washed with saturated brine (3 × 20 mL) and the water layer was extracted with EtOAc (3 × 20 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 60:1) to give the target product **2a** in 82% yield or **3a** in 78% yield.

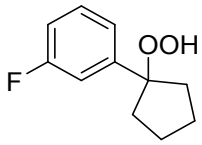
8. Characterization of Starting Materials 1



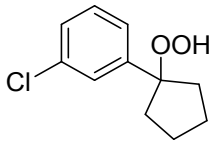
(1-Hydroperoxycyclopentyl)benzene (1a) Colorless oil (380 mg, 71%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.01 (m, 6H), 2.20 – 2.19(m, 2H), 1.86 – 1.81 (m, 4H), 1.67 – 1.66 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 127.3, 126.5, 125.5, 94.4, 34.6, 22.7. Spectral data matched literature values.[3]



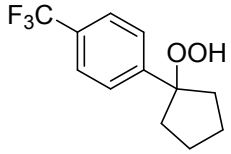
1-(1-Hydroperoxycyclopentyl)-2-methylbenzene (1b) White solid (420 mg, 73%). Melting point (°C): 31-32. R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 7.6 Hz, 1H), 7.23 – 7.14 (m, 3H), 7.06 (s, 1H), 2.53 (s, 3H), 2.47 – 2.38 (m, 2H), 2.11 – 2.00 (m, 2H), 1.87 – 1.84 (m, 2H), 1.73 – 1.69 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6, 137.5, 132.4, 128.2, 127.9, 125.4, 96.4, 35.4, 23.9, 21.5. IR (neat): υ_{max} (cm^{-1}) 3257, 2692, 1683, 1298, 767. HRMS (ESI) calcd for $C_{12}H_{16}O_2Na$ $[M+Na]^+$ 215.1043, found 215.1041.



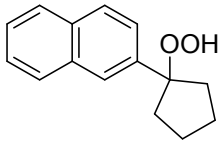
1-Fluoro-3-(1-hydroperoxycyclopentyl)benzene (1c) Colorless oil (400 mg, 68%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (s, 1H), 7.32 (t, J = 6.0 Hz, 1H), 7.25 (d, J = 8.0 Hz, 1H), 7.19 – 7.16 (m, 1H), 7.03 – 6.95 (m, 1H), 2.36 – 2.22 (m, 2H), 1.98 – 1.85 (m, 4H), 1.83 – 1.73 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0 (d, J = 244.2 Hz), 145.7 (d, J = 6.6 Hz), 129.9 (d, J = 8.1 Hz), 121.9 (d, J = 2.8 Hz), 114.4 (d, J = 21.0 Hz), 113.6 (d J = 21.8 Hz), 95.1 (d, J = 1.7 Hz), 35.9, 23.8. IR (neat): υ_{max} (cm^{-1}) 3257, 2751, 1701, 1394, 796. HRMS (ESI) calcd for $C_{11}H_{13}FO_2Na$ $[M+Na]^+$ 219.0792, found 219.0793.



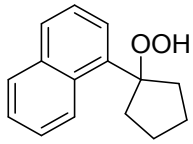
1-Chloro-3-(1-hydroperoxycyclopentyl)benzene (1d) Colorless oil (413 mg, 65%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (s, 1H), 7.28 – 7.18 (m, 4H), 2.22 – 2.12(m, 2H), 1.89 – 1.79 (m, 4H), 1.75 – 1.66 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.1, 134.4, 129.7, 127.7, 126.8, 124.6, 95.1, 35.8, 23.8. IR (neat): υ_{max} (cm^{-1}) 3395, 2963, 1681, 1474, 785. HRMS (ESI) calcd for $C_{11}H_{13}O_2ClNa$ $[M+Na]^+$ 235.0496, found 235.0500.



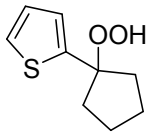
1-(1-Hydroperoxycyclopentyl)-4-(trifluoromethyl)benzene (1e) Colorless oil (501 mg, 68%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.56 (m, 4H), 7.43 (s, 1H), 2.34 – 2.28 (m, 2H), 2.00 – 1.89 (m, 4H), 1.84 – 1.79 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 129.7 (q, J = 32.2 Hz), 126.7, 125.3 (q, J = 3.7 Hz), 124.1 (q, J = 270.3 Hz), 95.1, 36.0, 23.9. IR (neat): υ_{max} (cm^{-1}) 3431, 2897, 1651, 1399, 776. HRMS (ESI) calcd for $C_{12}H_{13}F_3O_2Na$ $[M+Na]^+$ 269.0760, found 269.0760.



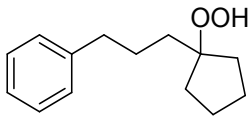
2-(1-Hydroperoxycyclopentyl)naphthalene (1f) White solid (445 mg, 65%). Melting point (°C): 55-57. R_f = 0.3 (petroleum ether/ethyl acetate = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.90 – 7.82 (m, 3H), 7.61 (dd, J = 8.4, 1.6 Hz, 1H), 7.55 – 7.45 (m, 2H), 7.37 (s, 1H), 2.48 – 2.35 (m, 2H), 2.12 – 2.07(m, 2H), 2.01 – 1.93 (m, 2H), 1.87 – 1.82 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.1, 133.1, 132.8, 128.3, 128.2, 127.6, 126.3, 126.1, 125.3, 124.8, 95.7, 35.8, 24.0. IR (neat): υ_{max} (cm^{-1}) 3389, 2733, 1701, 1414, 750, 732. HRMS (ESI) calcd for $C_{15}H_{16}O_2Na$ $[M+Na]^+$ 251.1043, found 251.1045.



1-(1-Hydroperoxycyclopentyl)naphthalene (1g) White solid (424 mg, 62%). Melting point (°C): 55-56. R_f = 0.3 (petroleum ether/ethyl acetate = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 – 8.57 (m, 1H), 7.92 – 7.86 (m, 1H), 7.82 (d, J = 8.4 Hz, 1H), 7.61 (d, J = 6.4 Hz, 1H), 7.54 – 7.47 (m, 2H), 7.43 (t, J = 8.0 Hz, 1H), 7.18 (s, 1H), 2.62 – 2.58 (m, 2H), 2.31 – 2.18 (m, 2H), 2.03 – 1.90 (m, 2H), 1.84 – 1.70 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.7, 134.7, 131.6, 129.2, 129.0, 126.2, 126.0, 125.7, 125.6, 124.8, 96.7, 36.5, 24.1. IR (neat): υ_{max} (cm^{-1}) 3379, 2698, 1678, 1387, 763. HRMS (ESI) calcd for $C_{15}H_{16}O_2Na$ $[M+Na]^+$ 251.1043, found 251.1061.

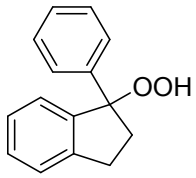


2-(1-Hydroperoxycyclopentyl)thiophene (1h) Colorless oil (331 mg, 65%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.18 (d, J = 5.1 Hz, 1H), 6.96 (d, J = 3.6 Hz, 1H), 6.89 (t, J = 4.8 Hz, 1H), 2.31 – 2.20 (m, 2H), 1.99 – 1.88 (m, 2H), 1.85 – 1.76 (m, 2H), 1.73 – 1.68 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 125.7, 124.1, 123.9, 92.0, 36.0, 22.8. IR (neat): υ_{max} (cm^{-1}) 3128, 2697, 1414, 750. HRMS (ESI) calcd for $C_9H_{12}O_2SNa$ $[M+Na]^+$ 207.0450, found 207.0452.

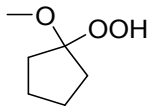


(3-(1-Hydroperoxycyclopentyl)propyl)benzene (1i) Colorless oil (363 mg, 55%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (t, J = 7.2 Hz, 2H), 7.14 – 7.09 (m, 4H), 2.58 – 2.55 (m, 2H), 1.82 – 1.71 (m, 2H), 1.67 – 1.59 (m, 6H), 1.52 – 1.45 (m, 2H), 1.42 – 1.34 (m,

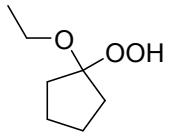
2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.5, 128.4, 128.3, 125.8, 94.7, 36.2, 35.7, 34.9, 26.2, 24.5. IR (neat): υ_{max} (cm^{-1}) 3410, 2902, 1703, 1284, 749. HRMS (ESI) calcd for $C_{14}H_{20}O_2Na$ $[M+Na]^+$ 243.1356, found 243.1351.



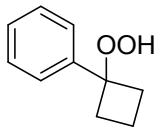
1-Hydroperoxy-1-phenyl-2,3-dihydro-1H-indene (1j) White solid (352 mg, 52%). Melting point (°C): 61-62. R_f = 0.3 (petroleum ether/ethyl acetate = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.37 – 7.34 (m, 4H), 7.31 – 7.26 (m, 2H), 7.26 – 7.22 (m, 1H), 3.19 – 3.12 (m, 1H), 2.96 – 2.89 (m, 1H), 2.64 – 2.57 (m, 1H), 2.44 – 2.31 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.2, 142.5, 142.4, 129.2, 128.4, 127.5, 126.8, 126.42, 126.36, 125.2, 97.5, 40.1, 30.2. IR (neat): υ_{max} (cm^{-1}) 3396, 2922, 1446, 757, 700. HRMS (ESI) calcd for $C_{15}H_{14}O_2Na$ $[M+Na]^+$ 249.0886, found 249.0884.



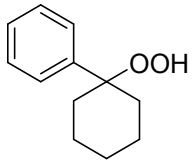
1-Hydroperoxy-1-methoxycyclopentane (1k) Colorless oil (257 mg, 65%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (br, 1H), 3.32 (s, 3H), 2.01 – 1.90 (m, 2H), 1.81 – 1.71 (m, 2H), 1.71 – 1.64 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 117.6, 50.5, 33.4, 23.8. IR (neat): υ_{max} (cm^{-1}) 3420, 2957, 1711, 1172, 751. HRMS (ESI) calcd for $C_6H_{13}O_3N$ $[M+H]^+$ 133.0859, found 133.0858.



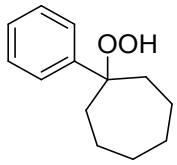
1-Ethoxy-1-hydroperoxycyclopentane (1l) Colorless oil (272 mg, 62%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (br, 1H), 3.58 (q, *J* = 7.2 Hz, 2H), 2.00 – 1.92 (m, 2H), 1.76 – 1.70 (m, 2H), 1.69 – 1.62 (m, 4H), 1.21 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 117.3, 58.7, 33.7, 23.7, 15.5. IR (neat): υ_{max} (cm^{-1}) 3741, 2960, 1732, 1261, 751. HRMS (ESI) calcd for $C_7H_{14}O_3Na$ $[M+Na]^+$ 169.0835, found 169.0841.



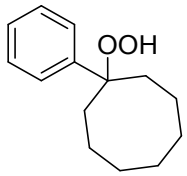
(1-Hydroperoxycyclobutyl)benzene (1m) Colorless oil (344 mg, 70%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.38 (m, 5H), 7.35 – 7.31 (m, 1H), 2.50 (t, *J* = 7.6, 4H), 2.20 – 2.08 (m, 1H), 1.88 – 1.75 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0, 128.4, 127.9, 126.3, 88.1, 31.1, 14.0. IR (neat): υ_{max} (cm^{-1}) 3396, 3030, 1684, 1449, 1276, 760. HRMS (ESI) calcd for $C_{10}H_{12}O_2Na$ $[M+Na]^+$ 187.0730, found 187.0735.



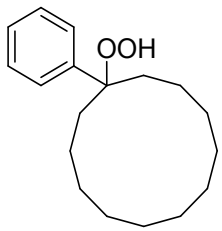
(1-Hydroperoxycyclohexyl)benzene (1n) Colorless oil (351 mg, 61%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 7.2, 0.4 Hz, 2H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.13 (s, 1H), 2.18 – 2.15 (m, 2H), 1.86 – 1.72 (m, 5H), 1.62 – 1.59 (m, 2H), 1.38 – 1.26 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.8, 128.6, 127.5, 125.7, 84.5, 34.1, 25.6, 22.0. IR (neat): υ_{max} (cm^{-1}) 3189, 2821, 1678, 754. HRMS (ESI) calcd for $C_{12}H_{16}O_2Na$ $[M+Na]^+$ 215.1043, found 215.1046,



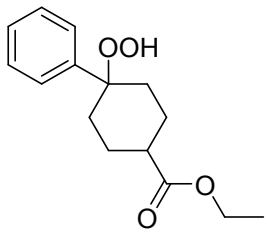
1-Hydroperoxy-1-phenylcycloheptane (1o) Colorless oil (340 mg, 55%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (dd, *J* = 8.0, 1.0 Hz, 2H), 7.36 (t, *J* = 8.4 Hz, 2H), 7.27 (t, *J* = 8.0 Hz, 1H), 7.23 (s, 1H), 2.18 – 1.99 (m, 4H), 1.84 – 1.74 (m, 2H), 1.70 – 1.63 (m, 2H), 1.60 – 1.52 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.9, 128.6, 127.3, 125.6, 89.2, 37.7, 30.1, 22.7. IR (neat): υ_{max} (cm^{-1}) 3409, 2922, 1445, 1009, 753, 699. HRMS (ESI) calcd for $C_{13}H_{18}O_2Na$ $[M+Na]^+$ 229.1199, found 229.1204.



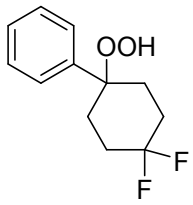
1-Hydroperoxy-1-phenylcyclooctane (1p) Colorless oil (343 mg, 52%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.48(m, 2 Hz), 7.41 – 7.34 (m, 2H), 7.33 – 7.28 (m, 1H), 7.13 (s, 1H), 2.25 – 2.19 (m, 1H), 2.05 – 1.95 (m, 3H), 1.74 – 1.68 (m, 5H), 1.56 – 1.52 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5, 127.6, 126.4, 125.1, 88.1, 30.5, 27.4, 24.1, 20.8. IR (neat): υ_{max} (cm^{-1}) 3362, 2920, 1683, 1445, 756, 699. HRMS (ESI) calcd for $C_{14}H_{20}O_2Na$ $[M+Na]^+$ 243.1356, found 243.1355.



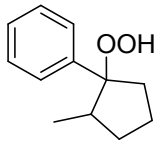
1-Hydroperoxy-1-phenylcyclododecane (1q) White solid (410 mg, 49%). Melting point (°C): 101-102. R_f = 0.3 (petroleum ether/ethyl acetate = 30:1).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (dd, *J* = 8.2, 1.0 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.24 – 7.18 (m, 1H), 7.07 (s, 1H), 2.03 – 1.88 (m, 2H), 1.63 – 1.57 (m, 2H), 1.30 (s, 16H), 1.11 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.2, 128.5, 127.3, 125.8, 88.6, 30.3, 26.3, 26.2, 22.3, 22.0, 19.4. IR (neat): υ_{max} (cm^{-1}) 3332, 2897, 1632, 1396, 765, 700, 685. HRMS (ESI) calcd for $C_{18}H_{28}O_2Na$ $[M+Na]^+$ 299.1982, found 299.1983.



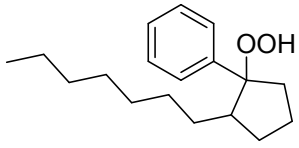
Ethyl 4-hydroperoxy-4-phenylcyclohexane-1-carboxylate (1r) Colorless oil (317 mg, 40%). R_f = 0.3 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.08 (s, 1H), 4.13 (q, *J* = 7.0 Hz, 2H), 2.67 – 2.59 (m, 1H), 2.23 – 2.13 (m, 2H), 2.09 – 1.98 (m, 3H), 1.91 – 1.79 (m, 2H), 1.25 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.2, 142.8, 128.7, 127.8, 126.2, 84.2, 60.3, 39.7, 31.2, 23.7, 14.3. IR (neat): υ_{max} (cm^{-1}) 3467, 3028, 1709, 1194, 760. HRMS (ESI) calcd for $C_{15}H_{20}O_4Na$ $[M+Na]^+$ 287.1254, found 287.1257.



(4,4-Difluoro-1-hydroperoxycyclohexyl)benzene (1s) White solid (410 mg, 60%). Melting point(°C): 55-56. R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.28 – 7.25 (m, 1H), 7.19 (s, 1H), 2.29 – 2.16 (m, 3H), 2.13 – 1.93 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 127.9, 127.0, 124.3,122.0 (dd, *J* = 236.7, 236.8 Hz), 81.8 (d, *J* = 1.6 Hz), 29.4 (d, *J* = 9.6 Hz), 28.6 (dd, *J* = 24.0, 24.1 Hz). IR (neat): υ_{max} (cm^{-1}) 3129, 2720, 1643, 1410, 790. HRMS (ESI) calcd for $C_{12}H_{14}F_2O_2Na$ $[M+Na]^+$ 251.0854, found 251.0863.

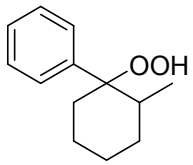


(1-Hydroperoxy-2-methylcyclopentyl)benzene (1t) Colorless oil (375 mg, 65%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.35 (m, 4H), 7.34 – 7.27 (m, 1H), 7.22 (s, 1H), 2.48 – 2.42 (m, 1H), 2.30 – 2.09 (m, 3H), 1.98 – 1.81 (m, 2H), 1.40 – 1.33 (m, 1H), 0.55 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.5, 128.4, 127.6, 127.2, 98.7, 41.5, 32.3, 30.0, 21.2, 18.7. IR (neat): υ_{max} (cm^{-1}) 3398, 2965, 1603, 1456, 788. HRMS (ESI) calcd for $C_{12}H_{16}O_2Na$ $[M+Na]^+$ 215.1043, found 215.1045.

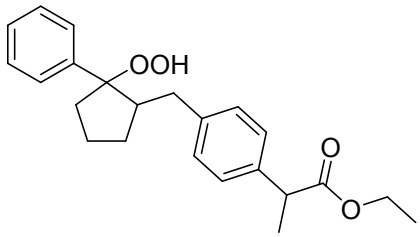


(2-Heptyl-1-hydroperoxycyclopentyl)benzene (1u) Colorless oil (447 mg, 54%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.35 (m, 4H), 7.34 – 7.28 (m, 1H), 7.21 (s, 1H), 2.50 – 2.41 (m, 1H), 2.23 – 2.17 (m, 1H), 2.11 – 2.01 (m, 1H), 2.00 – 1.78 (m, 3H), 1.47 – 1.39 (m, 1H), 1.28 – 1.19 (m, 4H), 1.17 – 1.01 (m, 7H), 0.84 (t, *J* = 7.1 Hz, 3H), 0.68 – 0.58 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 128.5, 127.6, 127.2, 98.6, 47.1, 32.3, 31.9, 31.2, 29.7, 29.5, 29.3, 27.9, 22.7, 21.5, 14.2. IR (neat): υ_{max} (cm^{-1}) 3396, 2955, 1719, 1449, 760, 700. HRMS (ESI) calcd for

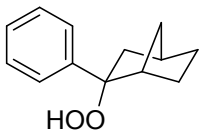
$C_{18}H_{28}O_2Na$ $[M+Na]^+$ 299.1982, found 299.1979.



(1-Hydroperoxy-2-methylcyclohexyl)benzene (1v) Colorless oil (321 mg, 52%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.37 (m, 4H), 7.3 – 7.27 (m, 1H), 6.92 (s, 1H), 2.31 – 2.27 (m, 1H), 2.10 – 2.0 (m, 3H), 1.84 – 1.77 (m, 1H), 1.73 – 1.68 (m, 1H), 1.54 – 1.63 (m, 2H), 1.39 – 1.34 (m, 1H), 0.65 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.1, 128.7, 127.6, 126.1, 87.8, 37.1, 28.4, 25.1, 21.5, 19.7, 15.9. IR (neat): υ_{max} (cm^{-1}) 3424, 2936, 1691, 1446, 755. HRMS (ESI) calcd for $C_{13}H_{18}O_2Na$ $[M+Na]^+$ 229.1199, found 229.1205.

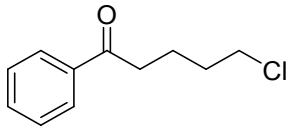


Ethyl 2-(4-((2-hydroperoxy-2-phenylcyclopentyl)methyl)phenyl)propanoate (1w) Colorless oil (662 mg, 60%). R_f = 0.3 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.47(m, 2H), 7.43 – 7.39 (m, 2H), 7.34 – 7.30 (m, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.96 (d, *J* = 8.0 Hz, 2H), 4.16 – 4.02 (m, 2H), 3.64 (q, *J* = 7.0 Hz, 1H), 2.57 – 2.48 (m, 1H), 2.43 – 2.27 (m, 3H), 1.95 – 1.84 (m, 3H), 1.82 – 1.76 (m, 1H), 1.45 (d, *J* = 7.2 Hz, 4H), 1.19 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.8, 140.4, 139.4, 138.2, 129.0, 128.7, 127.9, 127.3, 127.2, 98.1, 60.7, 48.6, 45.2, 38.0, 31.3, 29.2, 21.3, 18.6, 14.2. IR (neat): υ_{max} (cm^{-1}) 3422, 3057, 2341, 1730, 1175, 762. HRMS (ESI) calcd for $C_{23}H_{28}O_4Na$ $[M+Na]^+$ 391.1880, found 391.1882.

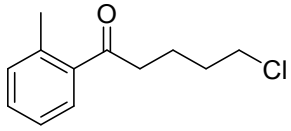


(1S,2R,4R)-2-Hydroperoxy-2-phenylbicyclo[2.2.1]heptane (1x) Colorless oil (422 mg, 69%) R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.6 Hz, 2H), 7.40 – 7.35 (m, 2H), 7.32 – 7.29 (m, 1H), 6.98 (br, 1H), 2.73 (s, 1H), 2.42 (s, 1H), 2.10 – 2.01 (m, 2H), 1.91 (d, *J* = 14 Hz, 1H), 1.55 – 1.43 (m, 2H), 1.31 (d, *J* = 9.6 Hz, 1H), 1.17 – 1.00 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.2, 128.6, 128.2, 127.8, 94.8, 44.3, 40.0, 36.9, 36.6, 29.0, 23.8. IR (neat): υ_{max} (cm^{-1}) 3449, 2958, 1449, 1324, 758, 700. HRMS (ESI) calcd for $C_{13}H_{16}O_2Na$ $[M+Na]^+$ 227.1043, found 227.1046.

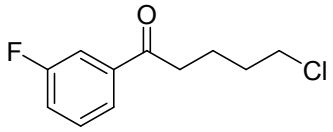
9. Characterization of Products 2-4



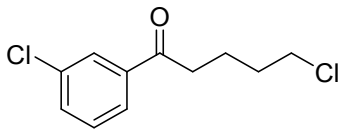
5-Chloro-1-phenylpentan-1-one (2a) White solid (35.4 mg, 90%). Melting point (°C): 63-64. R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.58 (t, *J* = 6.2 Hz, 2H), 3.02 (t, *J* = 6.8 Hz, 2H), 1.98 – 1.82 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 136.8, 133.1, 128.6, 128.0, 44.8, 37.6, 32.1, 21.5. Spectral data matched literature values.[4]



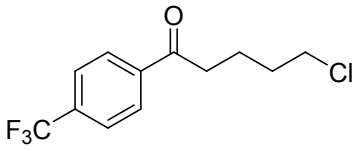
5-Chloro-1-(o-tolyl)pentan-1-one (2b) Light yellow oil (35.1 mg, 84%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.26 (t, *J* = 7.6 Hz, 2H), 3.57 (t, *J* = 6.2 Hz, 2H), 2.94 (t, *J* = 6.8 Hz, 2H), 2.49 (s, 3H), 1.87 – 1.45 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.8, 137.0, 136.9, 131.0, 130.2, 127.3, 124.7, 43.7, 39.5, 31.0, 20.6, 20.3. IR (neat): υ_{max} (cm^{-1}) 2928, 1682, 1128, 752, 648. HRMS (ESI) calcd for $C_{12}H_{16}ClO$ $[M+H]^+$ 211.0884, found 211.0876.



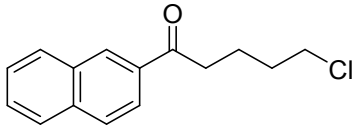
5-Chloro-1-(3-fluorophenyl)pentan-1-one (2c) Colorless oil (36.7 mg, 86%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.6 Hz, 1H), 7.64 (d, *J* = 9.2 Hz, 1H), 7.48 – 7.43 (m, 1H), 7.29 – 7.25 (m, 1H), 3.59 (t, *J* = 6.4 Hz, 2H), 3.00 (t, *J* = 6.4 Hz, 2H), 2.00 – 1.81 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3 (d, *J* = 2.0 Hz), 162.9 (d, *J* = 246.4 Hz), 138.9 (d, *J* = 6.0 Hz), 130.3 (d, *J* = 7.5 Hz), 123.8 (d, *J* = 2.9 Hz), 120.1 (d, *J* = 21.3 Hz), 114.7 (d, *J* = 22.0 Hz), 44.7, 37.7, 31.9, 21.4. IR (neat): υ_{max} (cm^{-1}) 2957, 1689, 1250, 751, 682. HRMS (ESI) calcd for $C_{11}H_{13}ClFO$ $[M+H]^+$ 215.0634, found 215.0629.



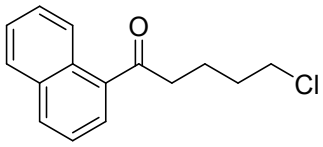
5-Chloro-1-(3-chlorophenyl)pentan-1-one (2d) Colorless oil (40.4 mg, 88%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.41 (t, *J* = 8.0 Hz, 1H), 3.58 (t, *J* = 6.4z, 2H), 2.99 (t, *J* = 6.8 Hz, 2H), 1.96 – 1.80 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 138.4, 135.0, 133.0, 130.0, 128.2, 126.1, 44.7, 37.7, 31.9, 21.3. IR (neat): υ_{max} (cm^{-1}) 2956, 1688, 1213, 750, 650. HRMS (ESI) calcd for $C_{11}H_{13}Cl_2O$ $[M+H]^+$ 231.0038, found 231.0030.



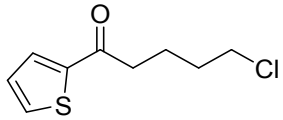
5-Chloro-1-(4-(trifluoromethyl)phenyl)pentan-1-one (2e) Colorless oil (45.4 mg, 86%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.0Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 3.59 (t, *J* = 6.0 Hz, 2H), 3.04 (t, *J* = 6.8 Hz, 2H), 1.99 – 1.83 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.5, 138.4, 133.4 (q, *J* = 32.4 Hz), 126.6, 124.7 (q, *J* = 3.7 Hz), 122.5 (q, *J* = 271.1 Hz), 43.6, 36.9, 30.9, 20.3. IR (neat): υ_{max} (cm^{-1}) 2957, 1692, 1324, 1128, 750, 602. HRMS (ESI) calcd for $C_{12}H_{13}ClF_3O$ $[M+H]^+$ 265.0602, found 265.0607.



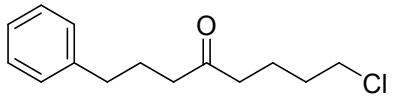
5-Chloro-1-(naphthalen-2-yl)pentan-1-one (2f) White solid (40 mg, 81%). Melting point (°C): 84-85. R_f = 0.3 (petroleum ether/ethyl acetate = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.03 (dd, *J* = 8.8, 1.6 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.89 (t, *J* = 8.0 Hz, 2H), 7.72 – 7.54 (m, 2H), 3.61 (t, *J* = 6.4 Hz, 2H), 3.15 (t, *J* = 6.4 Hz, 2H), 2.03 – 1.86 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 135.6, 134.2, 132.5, 129.7, 129.6, 128.51, 128.48, 127.8, 126.8, 123.8, 44.8, 37.6, 32.1, 21.7. IR (neat): υ_{max} (cm^{-1}) 2929, 1668, 1233, 747, 480. HRMS (ESI) calcd for $C_{15}H_{16}ClO$ $[M+H]^+$ 247.0884, found 247.0873.



5-Chloro-1-(naphthalen-1-yl)pentan-1-one (2g) Colorless oil (40.6 mg, 82%). R_f = 0.3 (petroleum ether/ethyl acetate = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (d, *J* = 8.8 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.89 – 7.84 (m, 2H), 7.64 – 7.46 (m, 3H), 3.59 (t, *J* = 6.4 Hz, 2H), 3.09 (t, *J* = 6.8 Hz, 2H), 2.01 – 1.86 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 136.0, 134.0, 132.6, 130.1, 128.5, 128.0, 127.4, 126.5, 125.7, 124.4, 44.7, 41.1, 32.1, 22.0. IR (neat): υ_{max} (cm^{-1}) 2954, 1679, 1100, 775, 648. HRMS (ESI) calcd for $C_{15}H_{16}ClO$ $[M+H]^+$ 247.0884, found 247.0878.

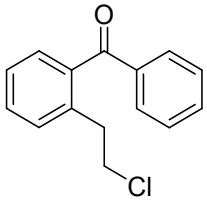


5-Chloro-1-(thiophen-2-yl)pentan-1-one (2h) Colorless oil (27.4 mg, 68%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 3.6 Hz, 1H), 7.63 (d, *J* = 4.8 Hz, 1H), 7.14 (t, *J* = 5.2 Hz, 1H), 3.58 (t, *J* = 6.4 Hz, 2H), 2.95 (t, *J* = 6.8 Hz, 2H), 1.98 – 1.82 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 144.2, 133.6, 131.8, 128.1, 44.7, 38.3, 32.0, 21.8. IR (neat): υ_{max} (cm^{-1}) 2956, 1659, 1415, 1236, 750, 648. HRMS (ESI) calcd for $C_9H_{12}ClO$ $[M+H]^+$ 203.0292 found 203.0296.

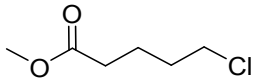


8-Chloro-1-phenyloctan-4-one (2i) Colorless oil (34.9 mg, 73%). R_f = 0.3 (petroleum ether/ethyl

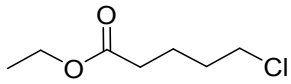
acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.18 (m, 2H), 7.13 – 7.08 (m, 3H), 3.44 (t, *J* = 6.4 Hz, 2H), 2.54 (t, *J* = 7.6 Hz, 2H), 2.33 (t, *J* = 7.2 Hz, 4H), 1.89 – 1.79 (m, 2H), 1.73 – 1.59 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.2, 140.5, 127.44, 127.36, 124.9, 43.6, 40.9, 40.7, 34.0, 30.9, 24.1, 20.0. IR (neat): υ_{max} (cm^{-1}) 2934, 1711, 1262, 750, 701. HRMS (ESI) calcd for $C_{14}H_{19}ClONa$ $[M+Na]^+$ 261.1017, found 261.1013.



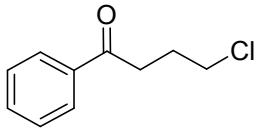
(2-(2-Chloroethyl)phenyl)(phenyl)methanone (2j) Colorless oil (24 mg, 49%). R_f = 0.3 (petroleum ether/ethyl acetate = 50:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.2 Hz, 2H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.49 – 7.45 (m, 3H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 2H), 3.74 (t, *J* = 7.2 Hz, 2H), 3.17 (t, *J* = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.1, 138.6, 137.6, 137.5, 133.4, 131.3, 130.6, 130.4, 129.3, 128.5, 126.3, 45.1, 36.6. IR (neat): υ_{max} (cm^{-1}) 2924, 1661, 1275, 751, 707. HRMS (ESI) calcd for $C_{15}H_{14}ClO$ $[M+H]^+$ 245.0728, found 245.0717.



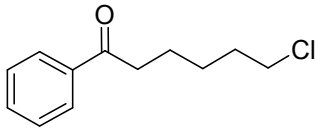
Methyl 5-chloropentanoate (2k) Colorless oil (13.0 mg, 43%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.68 (s, 3H), 3.55 (t, *J* = 6.0 Hz, 2H), 2.36 (t, *J* = 6.8 Hz, 2H), 1.86 – 1.75 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 51.6, 44.5, 33.2, 31.8, 22.2. IR (neat): υ_{max} (cm^{-1}) 2924, 1276, 751. HRMS (ESI) calcd for $C_6H_{11}ClO_2Na$ $[M+Na]^+$ 173.0340, found 173.0348.



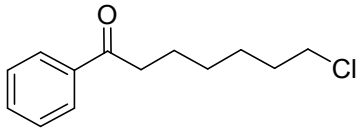
Ethyl 5-chloropentanoate (2l) Colorless oil (15.8 mg, 48%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13 (q, *J* = 7.2 Hz, 2H), 3.55 (t, *J* = 6.2 Hz, 2H), 2.34 (t, *J* = 7.0 Hz, 2H), 1.87 – 1.74 (m, 4H), 1.26 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 60.4, 44.5, 33.5, 31.9, 22.3, 14.3. IR (neat): υ_{max} (cm^{-1}) 2926, 1276, 751. HRMS (ESI) calcd for $C_7H_{14}ClO_2$ $[M+H]^+$ 165.0604, found 165.0611.



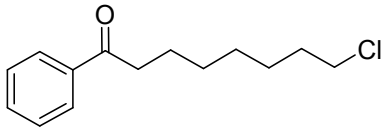
4-Chloro-1-phenylbutan-1-one (2m) Colorless oil (35 mg, 96%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 3.68 (t, *J* = 6.2 Hz, 2H), 3.18 (t, *J* = 7.0 Hz, 2H), 2.31 – 2.18 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.0, 136.7, 133.2, 128.7, 128.0, 44.7, 35.3, 26.8. Spectral data matched literature values.[4]



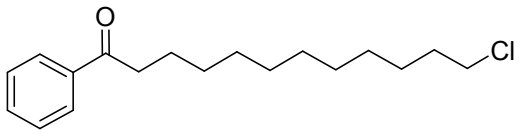
6-Chloro-1-phenylhexan-1-one (2n) Colorless oil (25.3 mg, 60%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.55 (t, *J* = 6.8 Hz, 2H), 2.99 (t, *J* = 7.4 Hz, 2H), 1.88 – 1.72 (m, 4H), 1.59 – 1.48 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 137.0, 133.0, 128.6, 128.0, 44.9, 38.3, 32.5, 26.6, 23.5. Spectral data matched literature values.[4]



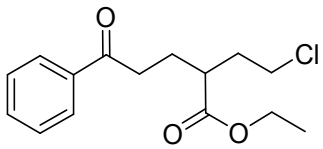
7-Chloro-1-phenylheptan-1-one (2o) White solid (31.4 mg, 70%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 34-36. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.2 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.53 (t, *J* = 6.6 Hz, 2H), 2.97 (t, *J* = 7.2 Hz, 2H), 1.81 – 1.74 (m, 4H), 1.54 – 1.35 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.3, 137.0, 128.6, 128.0, 45.1, 38.4, 32.4, 28.6, 26.7, 24.1. Spectral data matched literature values.[5]



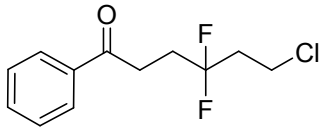
8-Chloro-1-phenyloctan-1-one (2p) Colorless oil (20 mg, 42%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.53 (t, *J* = 6.8 Hz, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 1.83 – 1.70 (m, 4H), 1.50 – 1.33 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.5, 137.0, 132.9, 128.6, 128.1, 45.1, 38.5, 32.6, 29.2, 28.8, 26.7, 24.2. Spectral data matched literature values.[6]



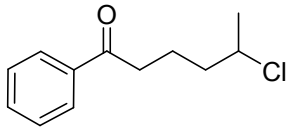
12-Chloro-1-phenyldodecan-1-one (2q) Colorless oil (46 mg, 78%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.4 Hz, 2H), 3.52 (t, *J* = 6.8 Hz, 2H), 2.96 (t, *J* = 7.4 Hz, 2H), 1.81 – 1.68 (m, 4H), 1.45 – 1.26 (m, 14H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 136.1, 131.8, 127.5, 127.0, 44.2, 37.6, 31.6, 28.45, 28.43, 28.41, 28.3, 27.8, 25.8, 23.3. IR (neat): υ_{max} (cm^{-1}) 2925, 1686, 1217, 751, 691. HRMS (ESI) calcd for $C_{18}H_{28}ClO$ $[M+H]^+$ 295.1823, found 295.1816.



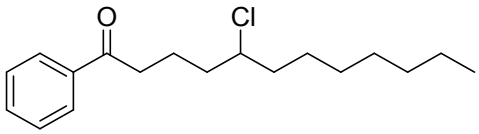
Ethyl 2-(2-chloroethyl)-5-oxo-5-phenylpentanoate (2r) Yellow oil (29.5 mg, 52%). R_f = 0.3 (petroleum ether/ethyl acetate = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.90 (m, 2H), 7.60 – 7.54 (m, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 4.17 (q, *J* = 7.0 Hz, 2H), 3.69 – 3.47 (m, 2H), 3.04 – 2.99 (m, 2H), 2.76 – 2.69 (m, 1H), 2.29 – 2.13 (m, 1H), 2.06 – 1.92 (m, 3H), 1.25 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.0, 174.8, 136.7, 133.2, 128.6, 128.0, 60.7, 42.5, 42.1, 35.9, 34.9, 26.1, 14.3. IR (neat): υ_{max} (cm^{-1}) 2961, 1727, 1156, 750, 691. HRMS (ESI) calcd for $C_{15}H_{19}ClO_3Na$ $[M+Na]^+$ 305.0915, found 305.0903.



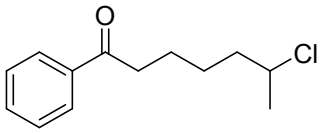
6-Chloro-4,4-difluoro-1-phenylhexan-1-one (2s) White solid (18.3 mg, 37%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 48-49. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.2 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 3.71 (t, *J* = 7.8 Hz, 2H), 3.21 (t, *J* = 7.6 Hz, 2H), 2.53 – 2.26 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.8, 135.4, 132.4, 127.7, 127.0, 122.2 (t, *J* = 240.2 Hz), 39.2 (t, *J* = 25.2 Hz), 35.8 (t, *J* = 6.0 Hz), 29.93 (t, *J* = 3.0 Hz), 29.88 (t, *J* = 24.0 Hz). IR (neat): υ_{max} (cm^{-1}) 2947, 1688, 1216, 748, 690. HRMS (ESI) calcd for $C_{12}H_{13}ClF_2ONa$ $[M+Na]^+$ 269.0515, found 269.0516.



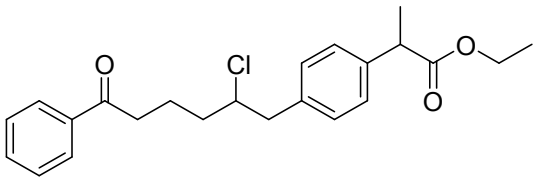
5-Chloro-1-phenylhexan-1-one (2t) Colorless oil (33.7 mg, 80%). R_f = 0.4 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 4.14 – 4.02 (m, 1H), 3.01 (td, *J* = 7.0, 1.8 Hz, 2H), 2.03 – 1.72 (m, 4H), 1.53 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.8, 136.9, 133.1, 128.6, 128.0, 58.5, 39.7, 37.8, 25.3, 21.3. IR (neat): υ_{max} (cm^{-1}) 2928, 1684, 1225, 751, 691. HRMS (ESI) calcd for $C_{12}H_{16}ClO$ $[M+H]^+$ 211.0884, found 211.0881.



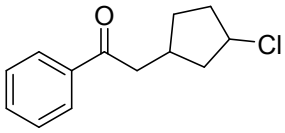
5-Chloro-1-phenyldodecan-1-one (2u) Colorless oil (40 mg, 68%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.97 – 3.91 (m, 1H), 3.03 – 2.99 (m, 2H), 2.04 – 1.94 (m, 1H), 1.89 – 1.82 (m, 2H), 1.80 – 1.68 (m, 3H), 1.58 – 1.47 (m, 1H), 1.45 – 1.36 (m, 1H), 1.31 – 1.23 (m, 8H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.8, 136.9, 133.1, 128.6, 128.0, 63.9, 38.5, 37.9, 37.8, 31.8, 29.2, 29.1, 26.5, 22.7, 21.2, 14.1. IR (neat): υ_{max} (cm^{-1}) 2926, 1686, 1225, 751, 691. HRMS (ESI) calcd for $C_{18}H_{28}ClO$ $[M+H]^+$ 295.1823, found 295.1815.



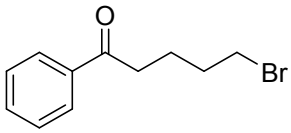
6-Chloro-1-phenylheptan-1-one (2v) Colorless oil (36.9 mg, 82%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 2H), 4.13 – 3.98 (m, 1H), 3.00 (t, *J* = 7.4 Hz, 2H), 1.87 – 1.69 (m, 4H), 1.66 – 1.54 (m, 2H), 1.51 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.1, 137.0, 133.0, 128.6, 128.0, 58.7, 40.2, 38.4, 26.4, 25.4, 23.7. IR (neat): υ_{max} (cm^{-1}) 2931, 1685, 1220, 751, 691. HRMS (ESI) calcd for $C_{13}H_{17}ClONa$ $[M+Na]^+$ 247.0860, found 247.0859.



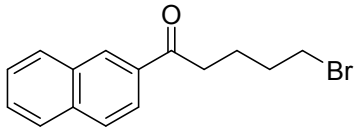
Ethyl 2-(4-(2-chloro-6-oxo-6-phenylhexyl)phenyl)propanoate (2w) Colorless oil (39.6 mg, 51%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 8.1 Hz, 2H), 4.17 – 3.94 (m, 3H), 3.62 (q, *J* = 7.1 Hz, 1H), 2.96 (d, *J* = 6.8 Hz, 2H), 2.92 (dd, *J* = 12.6, 6.6 Hz, 2H), 2.04 – 1.94 (m, 1H), 1.89 – 1.76 (m, 2H), 1.73 – 1.67 (m, 1H), 1.41 (d, *J* = 7.2 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.7, 174.6, 139.1, 136.8, 136.6, 133.1, 129.6, 128.6, 128.0, 127.5, 63.6, 60.8, 45.2, 44.5, 37.8, 37.1, 21.2, 18.6, 14.2. IR (neat): υ_{max} (cm^{-1}) 2936, 1728, 1173, 752, 692. HRMS (ESI) calcd for $C_{23}H_{27}ClO_3Na$ $[M+Na]^+$ 409.1541, found 409.1536.



(3-(Chloromethyl)cyclopentyl)(phenyl)methanone (2x) Yellow oil (29.4 mg, 66%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.90 (m, 2H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 4.48 – 4.42 (m, 0.455H), 4.40 – 4.34 (m, 0.545 H), 3.17 (d, *J* = 6.8 Hz, 1H), 3.03 (d, *J* = 7.2 Hz, 1H), 2.93 – 2.85 (m, 0.409 H), 2.63 – 2.48 (m, 1H), 2.30 – 2.15 (m, 1H), 2.10 – 1.94 (m, 2H), 1.79 – 1.71 (m, 0.543 H), 1.68 – 1.59 (m, 1H), 1.37 – 1.26 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 199.4, 137.0, 136.9, 133.1, 128.6, 128.6, 128.1, 128.0, 61.3, 60.7, 45.5, 44.4, 43.6, 43.4, 37.0, 36.6, 33.7, 33.1, 30.3, 30.2. IR (neat): υ_{max} (cm^{-1}) 2963, 1684, 1181, 752, 691. HRMS (ESI) calcd for $C_{13}H_{16}ClO$ $[M+H]^+$ 223.0884, found 223.0881.

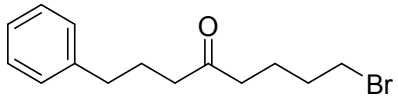


5-Bromo-1-phenylpentan-1-one (3a) Colorless oil (38.1 mg, 79%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 33-34. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.45 (t, *J* = 6.4 Hz, 2H), 3.02 (t, *J* = 6.8 Hz, 2H), 2.02 – 1.82 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 136.8, 133.1, 128.6, 128.0, 37.4, 33.4, 32.2, 22.8. Spectral data matched literature values.[7]

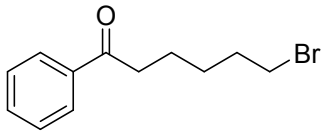


5-Bromo-1-(naphthalen-2-yl)pentan-1-one (3b) White solid (41.2 mg, 71%). R_f = 0.3 (petroleum ether/ethyl acetate = 50:1). Melting point (°C): 89-90. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.89 (t, *J* = 8.2 Hz, 2H), 7.67 – 7.51 (m, 2H), 3.49 (t, *J* = 6.2 Hz, 2H), 3.15 (t, *J* = 6.8 Hz, 2H), 2.07 – 1.92 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6,

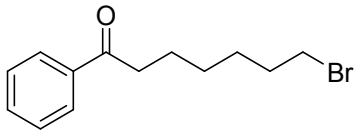
135.6, 134.2 132.5, 129.7 129.6 128.52, 128.49, 127.8, 126.8, 123.8, 37.5, 33.4 32.3 22.9. IR (neat): υ_{max} (cm^{-1}) 2994, 1679, 1275, 748, 507. HRMS (ESI) calcd for $C_{15}H_{16}BrO$ $[M+H]^+$ 291.0379, found 291.0371.



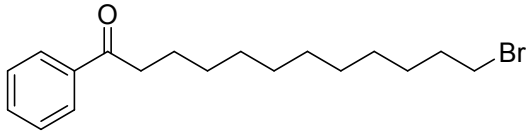
8-Bromo-1-phenyloctan-4-one (3c) Colorless oil (28.3 mg, 50%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.17 (m, 2H), 7.13 – 7.09 (m, 3H), 3.32 (t, *J* = 6.6 Hz, 2H), 2.55 (t, *J* = 7.6 Hz, 2H), 2.36 – 2.32(m, 4H), 1.88 – 1.80 (m, 2H), 1.78 – 1.73 (m, 2H), 1.67 – 1.59 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.1, 140.5, 127.44, 127.37, 124.9, 40.9, 40.6, 34.0, 32.3, 31.1, 24.1, 21.2. IR (neat): υ_{max} (cm^{-1}) 3025, 1679, 1275, 748, 700. HRMS (ESI) calcd for $C_{14}H_{19}IOBrNa$ $[M+Na]^+$ 305.0512, found 305.0504.



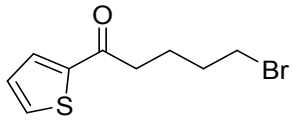
6-Bromo-1-phenylhexan-1-one (3d) White solid (27 mg, 53%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 33-34. ^1H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.43 (t, *J* = 6.8 Hz, 2H), 3.00 (t, *J* = 7.2 Hz, 2H), 1.97 – 1.87 (m, 2H), 1.82 – 1.74 (m, 2H), 1.57 – 1.50 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 137.0, 133.0, 128.6, 128.0, 38.3, 33.7, 32.6, 27.9, 23.3. Spectral data matched literature values.[8]



7-Bromo-1-phenylheptan-1-one (3e) White solid (34.4 mg, 64%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 42-43. ^1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.2 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.41 (t, *J* = 6.8 Hz, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 1.92 – 1.83 (m, 2H), 1.79 – 1.72 (m, 2H), 1.54 – 1.36 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.3, 137.0, 133.0, 128.6, 128.0, 38.4, 33.9, 32.6, 28.4, 28.0, 24.0. Spectral data matched literature values.[7]

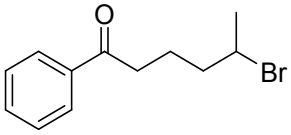


12-Bromo-1-phenyldodecan-1-one (3f) White solid (48 mg, 71%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 34-35. ^1H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 6.8 Hz, 2H), 7.59 – 7.52 (m, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 3.40 (t, *J* = 7.0 Hz, 2H), 2.96 (t, *J* = 7.4 Hz, 2H), 1.89 – 1.80 (m, 2H), 1.78 – 1.69 (m, 2H), 1.45 – 1.26 (m, 14H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.6, 137.1, 132.9, 128.6, 128.1, 38.6, 34.1, 32.8, 29.48, 29.46, 29.44, 29.42, 29.37, 28.8, 28.2, 24.4. Spectral data matched literature values.[8]

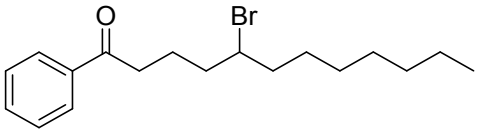


5-Bromo-1-(thiophen-2-yl)pentan-1-one (3g) Colorless oil (26.2 mg, 53%). R_f = 0.3 (petroleum

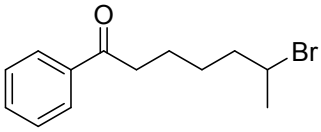
ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.64 (dd, *J* = 4.8, 1.0 Hz, 1H), 7.16 – 7.11 (m, 1H), 3.45 (t, *J* = 6.2 Hz, 2H), 2.95 (t, *J* = 6.8 Hz, 2H), 1.97 – 1.88 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 144.2, 133.6, 131.8, 128.1, 38.2, 33.3, 32.1, 23.1. Spectral data matched literature values.[9]



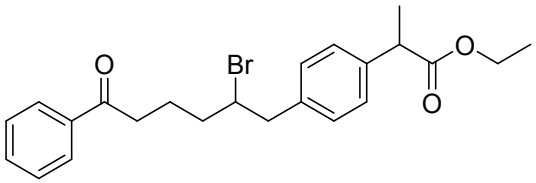
5-Bromo-1-phenylhexan-1-one (3h) Colorless oil (38.7 mg, 76%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2H), 7.61 – 7.54 (m, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 4.23 – 4.12 (m, 1H), 3.03 – 2.99 (m, 2H), 2.04 – 1.84 (m, 4H), 1.74 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.7, 136.9, 133.1, 128.6, 128.0, 51.2, 40.5, 37.6, 26.4, 22.4. Spectral data matched literature values.[8]



5-Bromo-1-phenyldodecan-1-one (3i) Colorless oil (30.5 mg, 45%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 4.10 – 4.05 (m, 1H), 3.03 – 2.99 (m, 2H), 2.06 – 1.99 (m, 1H), 1.96 – 1.86 (m, 3H), 1.87 – 1.80 (m, 2H), 1.58 – 1.48 (m, 1H), 1.45 – 1.38 (m, 1H), 1.32 – 1.24 (m, 8H), 0.88 (t, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.8, 136.9, 133.1, 128.6, 128.0, 58.3, 39.1, 38.5, 37.7, 31.8, 29.2, 29.0, 27.6, 22.7, 22.3, 14.1. IR (neat): υ_{max} (cm^{-1}) 2925, 1686, 1274, 751, 691. HRMS (ESI) calcd for $C_{18}H_{28}BrO$ $[M+H]^+$ 339.1318, found 339.1308.

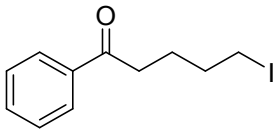


6-Bromo-1-phenylheptan-1-one (3j) Colorless oil (43 mg, 80%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 4.19 – 4.11 (m, 1H), 3.00 (t, *J* = 7.2 Hz, 2H), 1.94 – 1.75 (m, 4H), 1.72 (d, *J* = 6.4 Hz, 3H), 1.65 – 1.57 (m, 1H), 1.56 – 1.47 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.1, 136.9, 133.0, 128.6, 128.0, 51.6, 41.0, 38.4, 27.6, 26.5, 23.5. IR (neat): υ_{max} (cm^{-1}) 2930, 1684, 1211,751, 691. HRMS (ESI) calcd for $C_{13}H_{17}BrONa$ $[M+Na]^+$ 291.0355, found 291.0344.

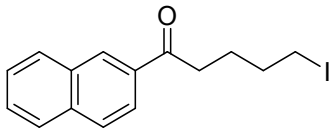


Ethyl 2-(4-(2-bromo-6-oxo-6-phenylhexyl)phenyl)propanoate (3k) Colorless oil (37 mg, 43%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 4.19 – 4.11 (m, 1H), 4.09 – 3.96 (m, 2H), 3.62 (q, *J* = 7.0 Hz, 1H), 3.13 – 3.07 (m, 2H), 2.94 – 2.89 (m, 2H), 2.04 – 1.98 (m, 1H), 1.90 – 1.78 (m, 3H), 1.41 (d, *J* = 7.2 Hz, 3H), 1.14 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR

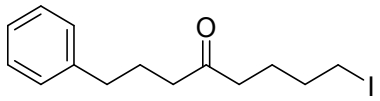
(100 MHz, $CDCl_3$) δ 199.7, 174.6, 139.2, 137.2, 136.8, 133.1, 129.5, 128.6, 128.0, 127.6, 60.8, 57.1, 45.22, 45.20, 37.7, 37.6, 22.3, 18.6, 14.2. IR (neat): υ_{max} (cm^{-1}) 2933, 1728, 1172, 752, 691. HRMS (ESI) calcd for $C_{23}H_{27}BrO_3Na$ $[M+Na]^+$ 453.1036, found 453.1027.



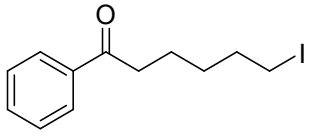
5-Iodo-1-phenylpentan-1-one (4a) Yellow solid (46.2 mg, 80%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 74-75. ^1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 2H), 3.22 (t, *J* = 6.6 Hz, 2H), 3.00 (t, *J* = 6.8 Hz, 2H), 1.94 – 1.83 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 136.8, 133.1, 128.7, 128.0, 37.2, 33.0, 25.1, 6.3. Spectral data matched literature values.[7]



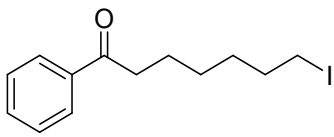
5-Iodo-1-(naphthalen-2-yl)pentan-1-one (4b) Yellow solid (49.5 mg, 73%). R_f = 0.3 (petroleum ether/ethyl acetate = 50:1). Melting point (°C): 81-82. ^1H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.03 (dd, *J* = 8.6, 1.4 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.89 (t, *J* = 7.8 Hz, 2H), 7.54 – 7.62 (m, 2H), 3.26 (t, *J* = 6.4 Hz, 2H), 3.13 (t, *J* = 6.8 Hz, 2H), 2.01 – 1.88 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.5, 135.6, 134.1, 132.5, 129.7, 129.6, 128.52, 128.50, 127.8, 126.8, 123.8, 37.3, 33.0, 25.2, 6.3. IR (neat): υ_{max} (cm^{-1}) 2947, 1670, 1210, 750, 598. HRMS (ESI) calcd for $C_{15}H_{16}IO$ $[M+H]^+$ 339.0240, found 339.0236.



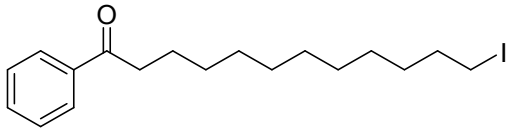
8-Iodo-1-phenyloctan-4-one (4c) Colorless oil (35.7 mg, 54%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.2 Hz, 2H), 7.21 – 7.16 (m, 3H), 3.16 (t, *J* = 6.8 Hz, 2H), 2.62 (t, *J* = 7.2 Hz, 2H), 2.43 – 2.38 (m, 4H), 1.96 – 1.87 (m, 2H), 1.85 – 1.75 (m, 2H), 1.70 – 1.62 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 209.1, 140.5, 127.44, 127.37, 124.9, 40.9, 40.4, 34.0, 31.8, 24.1, 23.5, 5.2. IR (neat): υ_{max} (cm^{-1}) 2930, 1710, 1211, 749, 700. HRMS (ESI) calcd for $C_{14}H_{19}IONa$ $[M+Na]^+$ 353.0373, found 353.0372.



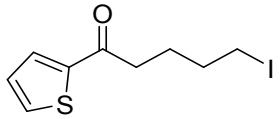
6-Iodo-1-phenylhexan-1-one (4d) Yellow oil (24.9 mg, 41%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.21 (t, *J* = 7.0 Hz, 2H), 2.99 (t, *J* = 7.4 Hz, 2H), 1.93 – 1.84 (m, 2H), 1.81 – 1.73 (m, 2H), 1.54 – 1.46 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 137.0, 133.0, 128.6, 128.0, 38.3, 33.3, 30.2, 23.1, 6.8. Spectral data matched literature values.[8]



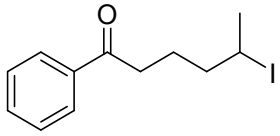
7-Iodo-1-phenylheptan-1-one (4e) Yellow solid (42.5 mg, 67%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 44-45. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.19 (t, *J* = 7.0 Hz, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 1.90 – 1.80 (m, 2H), 1.79 – 1.72 (m, 2H), 1.50 – 1.37 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.3, 136.0, 131.9, 127.6, 127.0, 37.3, 32.2, 29.3, 27.2, 23.0, 6.1. Spectral data matched literature values.[8]



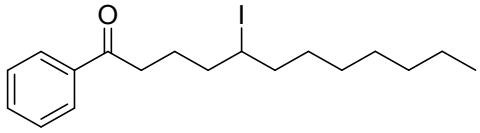
12-Iodo-1-phenyldodecan-1-one (4f) White solid (58 mg, 75%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). Melting point (°C): 39-40. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 3.18 (t, *J* = 7.0 Hz, 2H), 2.96 (t, *J* = 7.4 Hz, 2H), 1.85 – 1.79 (m, 2H), 1.77 – 1.69 (m, 2H), 1.40 – 1.27 (m, 14H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 136.1, 131.8, 127.5, 127.0, 37.6, 32.5, 29.5, 28.44, 28.42, 28.40, 28.36, 28.33, 27.5, 23.3, 6.4. Spectral data matched literature values.[8]



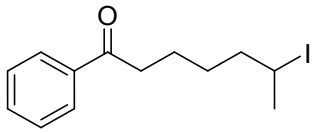
5-Iodo-1-(thiophen-2-yl)pentan-1-one (4g) Colorless oil (32.5 mg, 55%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz,) δ 7.72 (dd, *J* = 2.8, 0.8 Hz), 7.64 (dd, *J* = 3.6, 1.2 Hz), 7.13 (dd, *J* = 3.6, 1.2 Hz), 3.22 (t, *J* = 6.8 Hz), 2.94 (t, *J* = 7.2 Hz), 1.96 – 1.83 (m). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 144.2, 133.7, 131.9, 128.2, 38.1, 32.9, 25.5, 6.2. IR (neat): υ_{max} (cm^{-1}) 2934, 1660, 1262, 751, 700. HRMS (ESI) calcd for $C_9H_{11}IOSNa$ $[M+Na]^+$ 316.9468 found 316.9473.



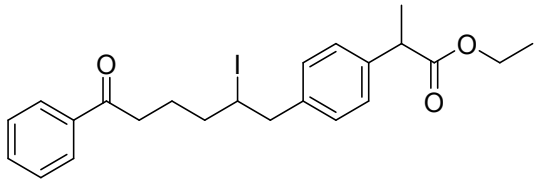
5-Iodo-1-phenylhexan-1-one (4h) Colorless oil (46 mg, 76%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.60 – 7.53 (m, 1H), 7.46 (t, *J* = 6.6 Hz, 2H), 4.29 – 4.15 (m, 1H), 3.03 – 2.98 (m, 2H), 1.94 (d, *J* = 6.8 Hz, 3H), 1.93 – 1.69 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.7, 136.8, 133.1, 128.6, 128.0, 42.2, 37.4, 29.6, 28.9, 24.4. IR (neat): υ_{max} (cm^{-1}) 2929, 1682, 1261, 751, 690. HRMS (ESI) calcd for $C_{12}H_{16}IO$ $[M+H]^+$ 303.0240, found 303.0236.



5-Iodo-1-phenyldodecan-1-one (4i) White solid (38 mg, 49%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.60 – 7.51 (m, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 4.22 – 4.09 (m, 1H), 3.03 – 2.98 (m, 2H), 2.03 – 1.77 (m, 5H), 1.75 – 1.66 (m, 1H), 1.57 – 1.47 (m, 1H), 1.40 – 1.39 (m, 1H), 1.31 – 1.25 (m, 8H), 0.89 (t, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.7, 136.8, 133.1, 128.6, 128.0, 40.6, 40.0, 39.6, 37.6, 31.8, 29.5, 29.2, 28.8, 24.2, 22.7, 14.1. IR (neat): υ_{max} (cm^{-1}) 2924, 1686, 1450, 1233, 751. HRMS (ESI) calcd for $C_{18}H_{27}IONa$ $[M+Na]^+$ 409.0999, found 409.0992.

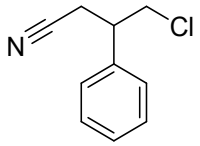


6-Iodo-1-phenylheptan-1-one (4j) Colorless oil (43 mg, 68%). R_f = 0.3 (petroleum ether/ethyl acetate = 60:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 4.28 – 4.12 (m, 1H), 3.00 (t, *J* = 7.2 Hz, 2H), 1.93 (d, *J* = 6.8 Hz, 3H), 1.90 – 1.83 (m, 1H), 1.81 – 1.72 (m, 2H), 1.70 – 1.66 (m, 1H), 1.61 – 1.55 (m, 1H), 1.53 – 1.46 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.1, 137.0, 133.0, 128.6, 128.0, 42.7, 38.4, 30.3, 29.5, 29.0, 23.3. IR (neat): υ_{max} (cm^{-1}) 2981, 1729, 1346, 761, 690. HRMS (ESI) calcd for $C_{13}H_{18}IO$ $[M+H]^+$ 317.0397, found 317.0388.

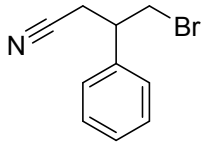


Ethyl 2-(4-(2-iodo-6-oxo-6-phenylhexyl)phenyl)propanoate (4k) Colorless oil (57.5 mg, 60%). R_f = 0.3 (petroleum ether/ethyl acetate = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.2 Hz, 2H), 7.47 (t, *J* = 6.8 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 4.25 – 4.14 (m, 1H), 4.09 – 3.95 (m, 2H), 3.59 (q, *J* = 7.0 Hz, 1H), 3.21 – 3.05 (m, 2H), 2.91 – 2.86 (m, 2H), 1.99 – 1.94 (m, 1H), 1.83 – 1.67 (m, 3H), 1.39 (d, *J* = 7.2 Hz, 3H), 1.11 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 174.6, 139.2, 138.4, 136.8, 133.1, 129.2, 128.6, 128.1, 127.6, 60.8, 47.0, 45.2, 38.9, 37.8, 37.5, 24.4, 18.6, 14.2. IR (neat): υ_{max} (cm^{-1}) 3446, 2979, 1728, 1206, 752, 657. HRMS (ESI) calcd for $C_{23}H_{27}IO_3Na$ $[M+Na]^+$ 501.0897, found 501.0897.

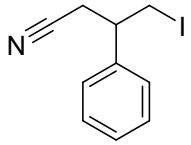
10. Characterization of Products 6-8



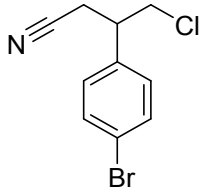
4-Chloro-3-phenylbutanenitrile (6a) Colorless oil (32.3 mg, 90%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 3H), 7.19 (d, *J* = 6.8 Hz, 2H), 3.78 – 3.68 (m, 2H), 3.331 – 3.24 (m, 1H), 2.87 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.75 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.3, 129.2, 128.4, 127.3, 117.7, 47.0, 44.2, 21.7. IR (neat): υ_{max} (cm^{-1}) 2920, 2250, 1453, 1260, 752. HRMS (ESI) calcd for $C_{10}H_{11}ClN$ $[M+H]^+$ 180.0575 found 180.0575.



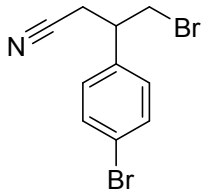
4-Bromo-3-phenylbutanenitrile (7a) Colorless oil (36.7 mg, 82%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.26 (m, 3H), 7.20 – 7.18 (m, 2H), 3.65 – 3.58 (m, 2H), 3.33 – 3.27 (m, 1H), 2.89 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.78 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 129.2, 128.5, 127.1, 117.6, 44.0, 35.3, 22.7. IR (neat): υ_{max} (cm^{-1}) 2962, 2248, 1496, 1229, 760. HRMS (ESI) calcd for $C_{10}H_{11}BrN$ $[M+H]^+$ 224.0069, found 224.0083.



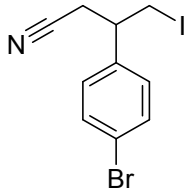
4-Iodo-3-phenylbutanenitrile (8a) Colorless oil (43 mg, 79%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.31 (m, 3H), 7.27 – 7.21 (m, 2H), 3.51 (d, *J* = 7.2 Hz, 2H), 3.31 – 3.20 (m, 1H), 2.93 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.83 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5, 129.2, 128.5, 126.9, 117.7, 44.3, 24.5, 9.0. IR (neat): υ_{max} (cm^{-1}) 2960, 2313, 1692, 1421, 1050, 699. HRMS (ESI) calcd for $C_{10}H_{11}IN$ $[M+H]^+$ 271.9931 found 271.9933.



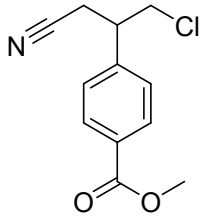
3-(4-Bromophenyl)-4-chlorobutanenitrile (6b) Colorless oil (41.2 mg, 80%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 3.82 – 3.72 (m, 2H), 3.37 – 3.28 (m, 1H), 2.92 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.80 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.2, 132.3, 129.0, 122.5, 117.4, 46.6, 43.6, 21.6. IR (neat): υ_{max} (cm^{-1}) 2958, 2249, 1592, 1489, 1010, 747. HRMS (ESI) calcd for $C_{10}H_{10}BrClN$ $[M+H]^+$ 257.9680 found 257.9689.



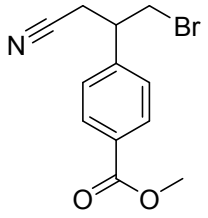
4-Bromo-3-(4-bromophenyl)butanenitrile (7b) Colorless oil (45.3 mg, 75%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 3.70 – 3.58 (m, 2H), 3.37 – 3.31 (m, 1H), 2.93 (dd, *J* = 16.8, 5.2 Hz, 1H), 2.82 (dd, *J* = 16.8, 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 132.3, 128.9, 122.5, 117.4, 43.4, 34.8, 22.6. IR (neat): υ_{max} (cm^{-1}) 2961, 2250, 1489, 1232, 1145, 823, 667. HRMS (ESI) calcd for $C_{10}H_{10}Br_2ClN$ $[M+H]^+$ 301.9175 found 301.9161.



3-(4-Bromophenyl)-4-iodobutanenitrile (8b) Colorless oil (49 mg, 70%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 3.47 (d, *J* = 7.0 Hz, 2H), 3.28 – 3.17 (m, 1H), 2.90 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.80 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 132.4, 128.7, 122.5, 117.4, 43.8, 24.4, 8.2. IR (neat): υ_{max} (cm^{-1}) 2968, 2315, 1786, 1515, 1056, 764. HRMS (ESI) calcd for $C_{10}H_{10}BrIClNNa$ $[M+H]^+$ 371.8855, found 371.8851.

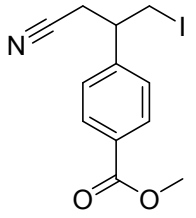


Methyl 4-(1-chloro-3-cyanopropan-2-yl)benzoate (6c) Colorless oil (42.5 mg, 90%). R_f = 0.2 (petroleum ether/ethyl acetate = 8:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 3.92 (s, 3H), 3.87 – 3.77 (m, 2H), 3.47 – 3.39 (m, 1H), 2.96 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.85 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 143.1, 130.4, 130.4, 127.4, 117.2, 52.3, 46.5, 44.0, 21.5. IR (neat): υ_{max} (cm^{-1}) 2955, 2249, 1720, 1279, 751. HRMS (ESI) calcd for $C_{12}H_{12}ClNO_2Na$ $[M+Na]^+$ 260.0449 found 260.0452.

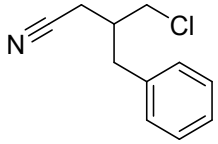


Methyl 4-(1-bromo-3-cyanopropan-2-yl)benzoate (7c) Colorless oil (44.4 mg, 79%). R_f = 0.2 (petroleum ether/ethyl acetate = 8:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 3.92 (s, 3H), 3.73 – 3.64 (m, 2H), 3.47 – 3.41 (m, 1H), 2.97 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.87 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 143.5, 130.4, 130.3, 127.2, 117.2,

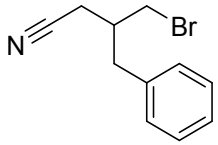
52.3, 43.8, 34.6, 22.5. IR (neat): υ_{max} (cm^{-1}) 2955, 2249, 1720, 1284, 1111, 708. HRMS (ESI) calcd for $C_{12}H_{12}BrNO_2Na$ $[M+Na]^+$ 303.9944 found 303.9949.



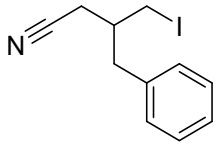
Methyl 4-(1-cyano-3-iodopropan-2-yl)benzoate (8c) Colorless oil (38.2 mg, 58%). R_f = 0.2 (petroleum ether/ethyl acetate = 8:1). ^1H (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 3.92 (s, 3H), 3.51 (d, *J* = 7.2 Hz, 2H), 3.37 – 3.27 (m, 1H), 2.92 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.84 (dd, *J* = 16.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 144.3, 130.4, 130.3, 127.0, 117.2, 52.3, 44.1, 24.3, 7.8. IR (neat): υ_{max} (cm^{-1}) 2954, 2313, 1719, 1283, 1111, 707. HRMS (ESI) calcd for $C_{12}H_{13}INO_2$ $[M+H]^+$ 329.9986 found 329.9995.



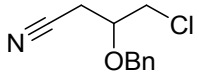
3-Benzyl-4-chlorobutanenitrile (6d) Colorless oil (33.7 mg, 87%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.2 Hz, 2H), 7.28 – 7.25 (m, 1H), 7.20 (d, *J* = 6.8 Hz, 2H), 3.66 (dd, *J* = 11.6, 4.4 Hz, 1H), 3.52 (dd, *J* = 11.6, 4.0 Hz, 1H), 2.87 – 2.75 (m, 2H), 2.55 (dd, *J* = 16.8, 6.0 Hz, 1H), 2.47 (dd, *J* = 16.8, 6.4 Hz, 1H), 2.43 – 2.33 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.4, 129.0, 128.9, 127.1, 117.7, 46.1, 39.7, 37.3, 19.6. IR (neat): υ_{max} (cm^{-1}) 2926, 2247, 1780, 1276, 750. HRMS (ESI) calcd for $C_{11}H_{13}ClN$ $[M+H]^+$ 194.0731, found 194.0733.



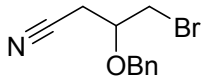
3-Benzyl-4-bromobutanenitrile (7d) Colorless oil (33.4 mg, 70%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.2 Hz, 2H), 7.29 – 7.25 (m, 1H), 7.21 (d, *J* = 7.2 Hz, 2H), 3.55 (dd, *J* = 10.8, 4.0 Hz, 1H), 3.40 (dd, *J* = 10.8, 6.4 Hz, 1H), 2.87 – 2.76 (m, 2H), 2.57 (dd, *J* = 16.8, 6.0 Hz, 1H), 2.48 (dd, *J* = 16.8, 6,4 Hz, 1H), 2.40 – 2.31 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.4, 129.0, 128.9, 127.1, 117.7, 39.4, 38.2, 35.6, 20.7. IR (neat): υ_{max} (cm^{-1}) 2926, 2247, 1780, 1447, 1254, 744. HRMS (ESI) calcd for $C_{11}H_{13}BrN$ $[M+H]^+$ 238.0023, found 238.0023.



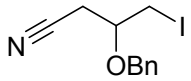
3-Benzyl-4-iodobutanenitrile (8d) Colorless oil (38.3 mg, 67%). R_f = 0.2 (petroleum ether/ethyl acetate = 15:1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.27 (m, 3H), 7.21 (d, *J* = 7.2 Hz, 2H), 3.37 (dd, *J* = 10.4, 4.4 Hz, 1H), 3.20 (dd, *J* = 10.4, 6.4 Hz, 1H), 2.80 – 2.76 (m, 2H), 2.54 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.42 (dd, *J* = 16.8, 6.4 Hz, 1H), 2.05 – 1.99 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 129.1, 129.0, 127.2, 117.7, 39.7, 39.1, 22.8, 10.6. IR (neat): υ_{max} (cm^{-1}) 2977, 2314, 1778, 1418, 1059, 700. HRMS (ESI) calcd for $C_{11}H_{13}IN$ $[M+H]^+$ 286.0087, found 286.0094.



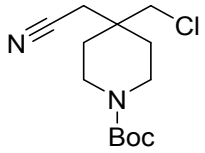
3-(Benzyloxy)-4-chlorobutanenitrile (6e) Colorless oil (27.3 mg, 65%). R_f = 0.2 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.31 (m, 5H), 4.70 (d, *J* = 11.6 Hz, 1H), 4.66 (d, *J* = 11.6 Hz, 1H), 3.94 – 3.89 (m, 1H), 3.67 (dd, *J* = 12.0, 4.4 Hz, 1H), 3.61 (dd, *J* = 11.6, 6.4 Hz, 1H), 2.79 – 2.67 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.7, 128.7, 128.4, 128.0, 116.7, 74.1, 72.5, 44.0, 21.5. IR (neat): υ_{max} (cm^{-1}) 2919, 2252, 1728, 1275, 749. HRMS (ESI) calcd for $C_{11}H_{12}ClNONa$ $[M+Na]^+$ 232.0500, found 232.0499.



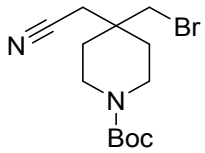
3-(Benzyloxy)-4-bromobutanenitrile (7e) Colorless oil (28 mg, 55%). R_f = 0.2 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.33 (m, 5H), 4.70 (d, *J* = 11.6 Hz, 1H), 4.64 (d, *J* = 11.6 Hz, 1H), 3.96 – 3.87 (m, 1H), 3.54 (dd, *J* = 10.8, 4.0 Hz, 1H), 3.47 (dd, *J* = 11.2, 6.8 Hz, 1H), 2.82 – 2.69 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.7, 128.8, 128.5, 128.1, 116.8, 73.7, 72.5, 32.3, 22.5. IR (neat): υ_{max} (cm^{-1}) 2919, 2252, 1720, 1417, 1067, 744. HRMS (ESI) calcd for $C_{11}H_{12}BrNONa$ $[M+Na]^+$ 275.9995, found 276.0004.



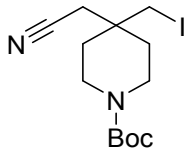
3-(Benzyloxy)-4-iodobutanenitrile (8e) Colorless oil (20 mg, 33%). R_f = 0.2 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.31 (m, 5H), 4.70 (d, *J* = 11.6 Hz, 1H). 4.60 (d, *J* = 11.2 Hz, 1H), 3.72 – 3.62 (m, 1H), 3.39 (dd, *J* = 10.8, 4.0 Hz, 1H), 3.32 (dd, *J* = 10.8, 6.4 Hz, 1H), 2.74 – 2.73 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.6, 128.7, 128.4, 128.0, 116.6, 73.6, 72.2, 23.9, 6.3. IR (neat): υ_{max} (cm^{-1}) 2975, 2314, 1695, 1514, 1056, 745. HRMS (ESI) calcd for $C_{11}H_{12}INONa$ $[M+Na]^+$ 323.9856, found 323.9860.



***tert*-Butyl 4-(chloromethyl)-4-(cyanomethyl)piperidine-1-carboxylate (6f)** Colorless oil (46.5 mg, 85%). R_f = 0.2 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.62 (s, 2H), 3.47 – 3.37 (m, 4H), 2.56 (s, 2H), 1.68 – 1.60 (m, 4H), 1.45 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.1, 116.2, 79.7, 55.9, 49.2, 36.1, 31.7, 27.9, 24.0. IR (neat): υ_{max} (cm^{-1}) 2930, 2242, 1688, 1277, 750. HRMS (ESI) calcd for $C_{13}H_{21}ClN_2O_2Na$ $[M+Na]^+$ 295.1184, found 295.1186.

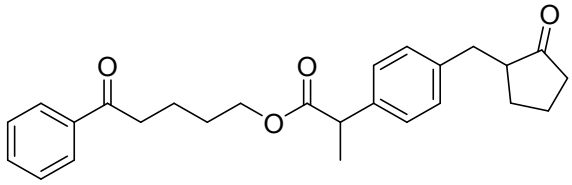


***tert*-Butyl 4-(bromomethyl)-4-(cyanomethyl)piperidine-1-carboxylate (7f)** Colorless oil (39.3 mg, 62%). R_f = 0.2 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.52 (s, 2H), 3.45 – 3.36 (m, 4H), 2.57 (s, 2H), 1.69 – 1.63 (m, 4H), 1.45 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 116.8, 80.2, 39.7, 39.1, 35.9, 33.0, 28.5, 25.6. IR (neat): υ_{max} (cm^{-1}) 2974, 2314, 1689, 1422, 1162. HRMS (ESI) calcd for $C_{13}H_{21}BrN_2O_2Na$ $[M+Na]^+$ 339.0679, found 339.0676.

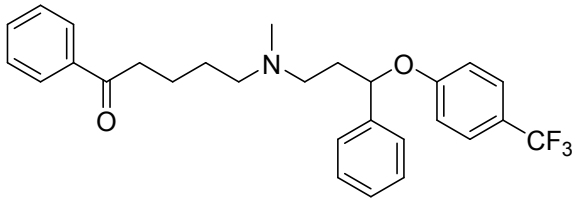


***tert*-Butyl 4-(cyanomethyl)-4-(iodomethyl)piperidine-1-carboxylate (8f)** Colorless oil (40.2 mg, 55%). R_f = 0.2 (petroleum ether/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.43 – 3.38 (m, 4H), 3.37 (s, 2H), 2.53 (s, 2H), 1.73 – 1.62 (m, 4H), 1.45 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 116.7, 80.2, 39.5, 34.8, 33.9, 28.5, 27.6, 16.1. IR (neat): υ_{max} (cm^{-1}) 2959, 2248, 1487, 1072, 815. $C_{13}H_{21}IN_2O_2Na$ $[M+Na]^+$ 387.0540, found 387.0541.

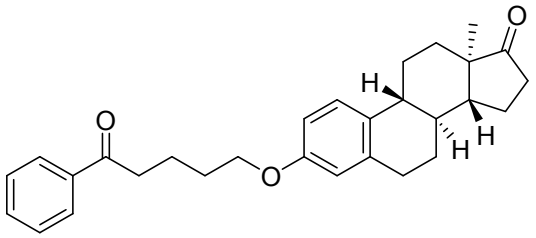
11. Characterization of Products 9-12



5-Oxo-5-phenylpentyl 2-(4-((2-oxocyclopentyl)methyl)phenyl)propanoate (9) Colorless oil (65.8 mg, 81%). R_f = 0.4 (petroleum ether/ethyl acetate = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 4.12 – 4.09 (m, 2H), 3.68 (q, *J* = 6.8 Hz, 1H), 3.10 (dd, *J* = 10, 3.6 Hz, 1H), 2.94 (t, *J* = 6.8 Hz, 2H), 2.49 – 2.44 (m, 1H), 2.36 – 2.29 (m, 2H), 2.18 – 2.02 (m, 2H), 1.99 – 1.89 (m, 1H), 1.78 – 1.67 (m, 5H), 1.57 – 1.51 (m, 1H), 1.47 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.7, 174.7, 138.8, 138.4, 136.9, 133.1, 129.1, 128.6, 128.0, 127.5, 64.4, 51.0, 45.2, 38.2, 37.8, 35.2, 29.2, 28.1, 20.54, 20.49, 18.5. IR (neat): υ_{max} (cm^{-1}) 2958, 1729, 1683, 1450, 1158, 752, 691. HRMS (ESI) calcd for $C_{26}H_{30}O_4Na$ $[M+Na]^+$ 429.2036, found 429.2033.

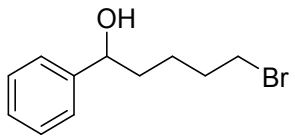


5-(Methyl(3-phenyl-3-(4-(trifluoromethyl)phenoxy)propyl)amino)-1-phenylpentan-1-one (10) Colorless oil (72.2 mg, 78%). R_f = 0.4 (petroleum ether/ethyl acetate = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 2H), 7.47 (t, *J* = 7.0 Hz, 1H), 7.41 – 7.30 (m, 4H), 7.29 – 7.15 (m, 5H), 6.82 (d, *J* = 8.4 Hz, 2H), 5.25 – 5.22 (m, 1H), 2.84 (t, *J* = 7.2 Hz, 2H), 2.57 – 2.46 (m, 1H), 2.41 – 2.26 (m, 3H), 2.16 (s, 3H), 2.11 – 2.06 (m, 1H), 1.93 – 1.88 (m, 1H), 1.68 – 1.61 (m, 2H), 1.46 – 1.41 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.2, 160.7, 141.2, 137.0, 133.0, 128.8, 128.6, 128.0, 127.8, 127.1 (q, *J* = 269.6 Hz), 126.7 (q, *J* = 3.7 Hz), 125.9, 122.6 (q, *J* = 32.5 Hz), 115.8, 78.3, 57.6, 53.6, 42.2, 38.3, 36.5, 27.0, 22.1. IR (neat): υ_{max} (cm^{-1}) 2938, 2795, 1685, 1614, 1327, 1256, 1112, 751, 700. HRMS (ESI) calcd for $C_{28}H_{30}F_3NO_2Na$ $[M+Na]^+$ 470.2301, found 470.2310.



(8S,9R,13R,14R)-13-Methyl-3-((5-oxo-5-phenylpentyl)oxy)-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (11) White solid (65 mg, 76%). R_f = 0.4 (petroleum ether/ethyl acetate = 10:1). Melting point (°C): 113-114. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 1H), 6.72 – 6.63 (m, 2H), 3.99 (t, *J* = 6.0 Hz, 2H), 3.07 (t, *J* = 7.0 Hz, 2H), 2.93 – 2.85 (m, 2H), 2.50 (dd, *J* = 18.8, 8.5 Hz, 1H), 2.40 – 2.38 (m, 1H), 2.27 – 2.25 (m, 1H), 2.17 – 2.04 (m, 2H), 1.97 – 1.86 (m, 5H), 1.68 – 1.52 (m, 4H), 1.50 – 1.38 (m, 3H), 0.91 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.1, 157.0, 137.7, 137.0, 133.0, 132.0, 128.6, 128.1, 126.3, 114.5, 112.1, 67.5, 50.4, 48.0, 44.0, 38.4, 38.1, 35.9, 31.6, 29.7, 28.9,

26.6, 25.9, 21.6, 21.0, 13.9. IR (neat): υ_{max} (cm^{-1}) 2929, 2870, 1733, 1684, 1496, 1253, 1056, 756, 693. HRMS (ESI) calcd for $C_{29}H_{34}O_3Na$ $[M+Na]^+$ 453.2400, found 453.2396.



5-Bromo-1-phenylpentan-1-ol (12) Colorless oil (44.6 mg, 92%). R_f = 0.4 (petroleum ether/ethyl acetate = 10:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.17 (m, 5H), 4.60 – 4.57 (m, 1H), 3.31 (t, *J* = 6.8 Hz, 2H), 1.92 (s, 1H), 1.83 – 1.78 (m, 2H), 1.76 – 1.69 (m, 1H), 1.68 – 1.58 (m, 1H), 1.56 – 1.43 (m, 1H), 1.38 – 1.31 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.6, 128.6, 127.7, 125.9, 74.4, 38.1, 33.7, 32.7, 24.5. Spectral data matched literature values.[10]

12. Reference

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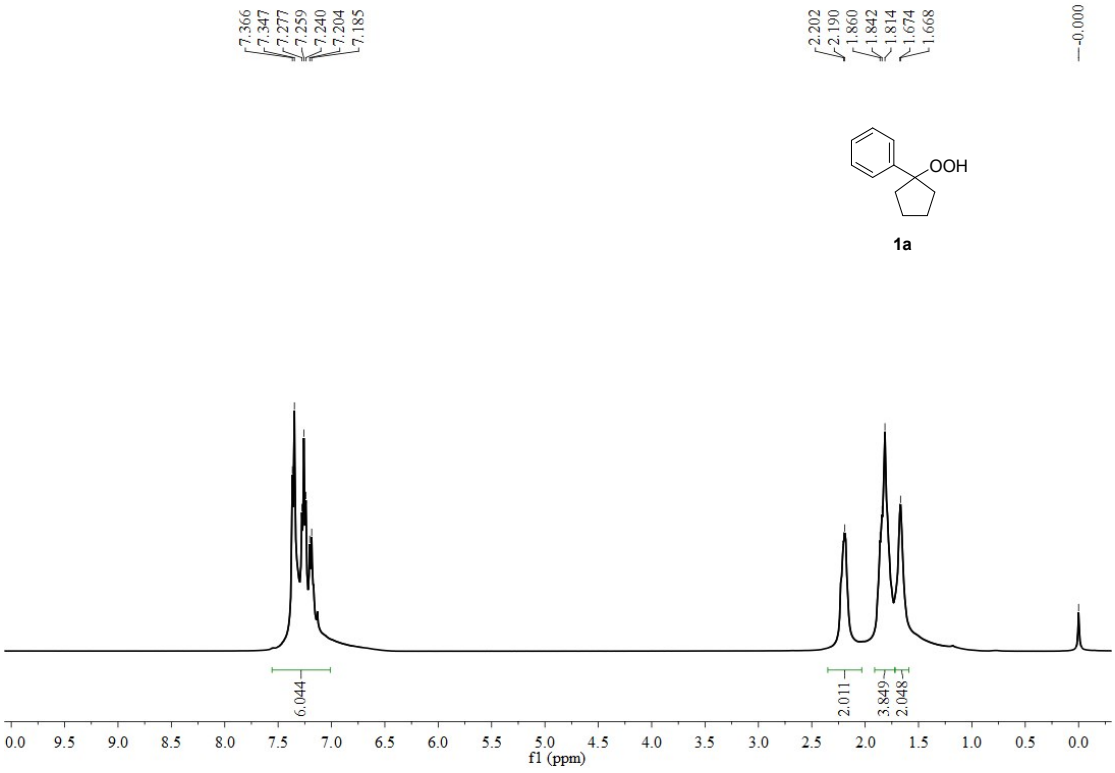
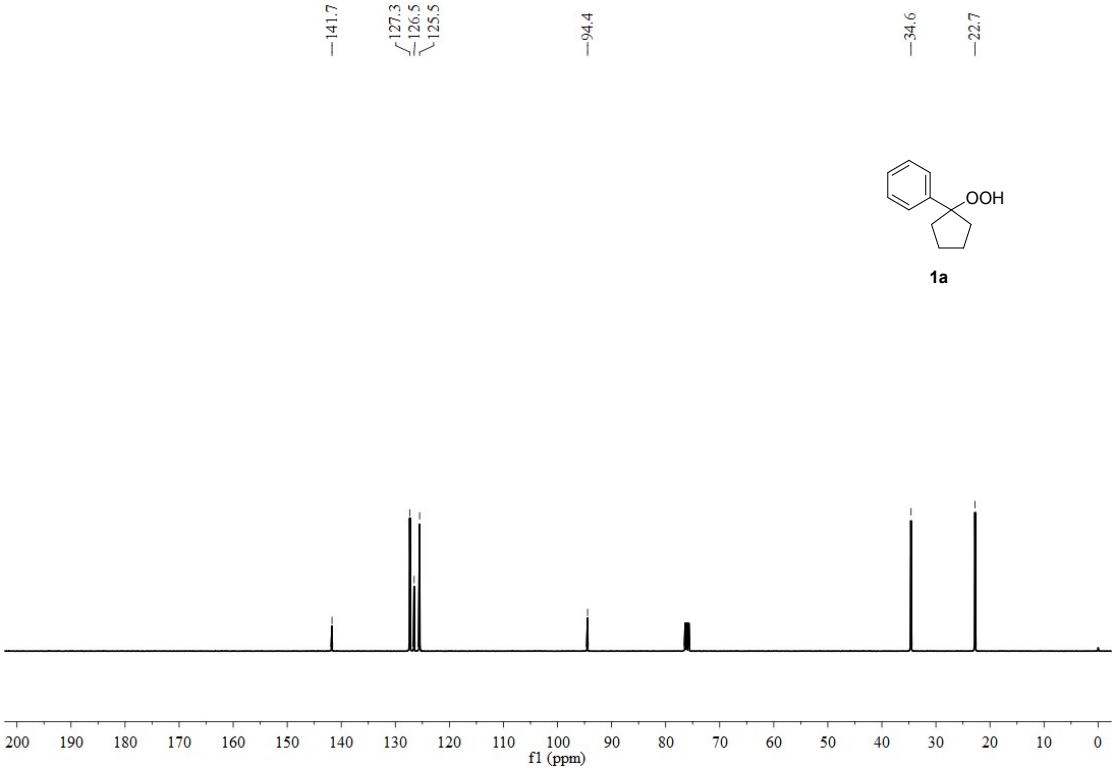
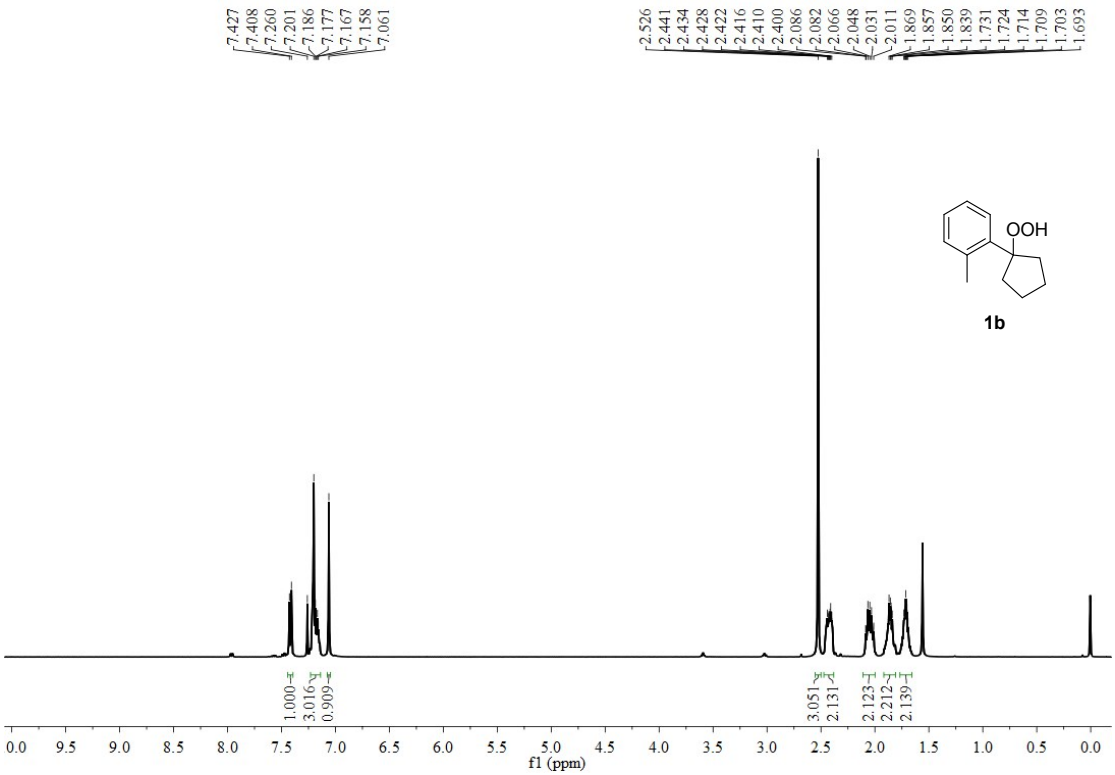
13. ^{1}H NMR and ^{13}C NMR Spectra of Starting Materials 1

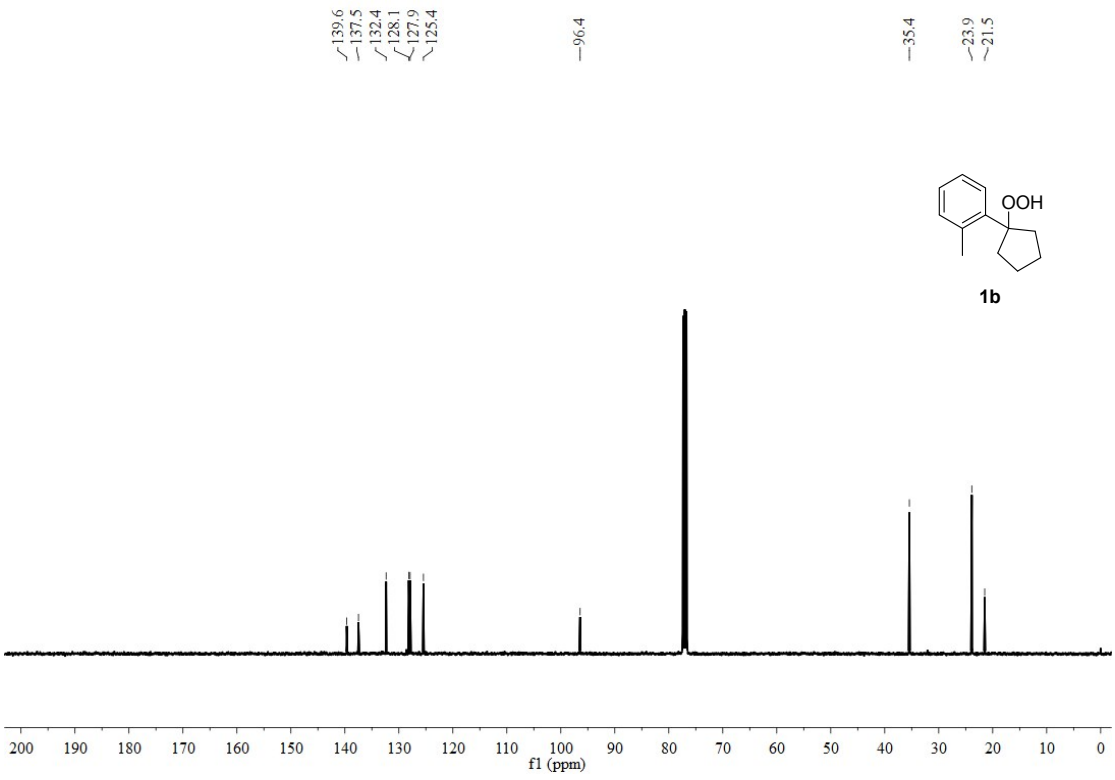
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1a**



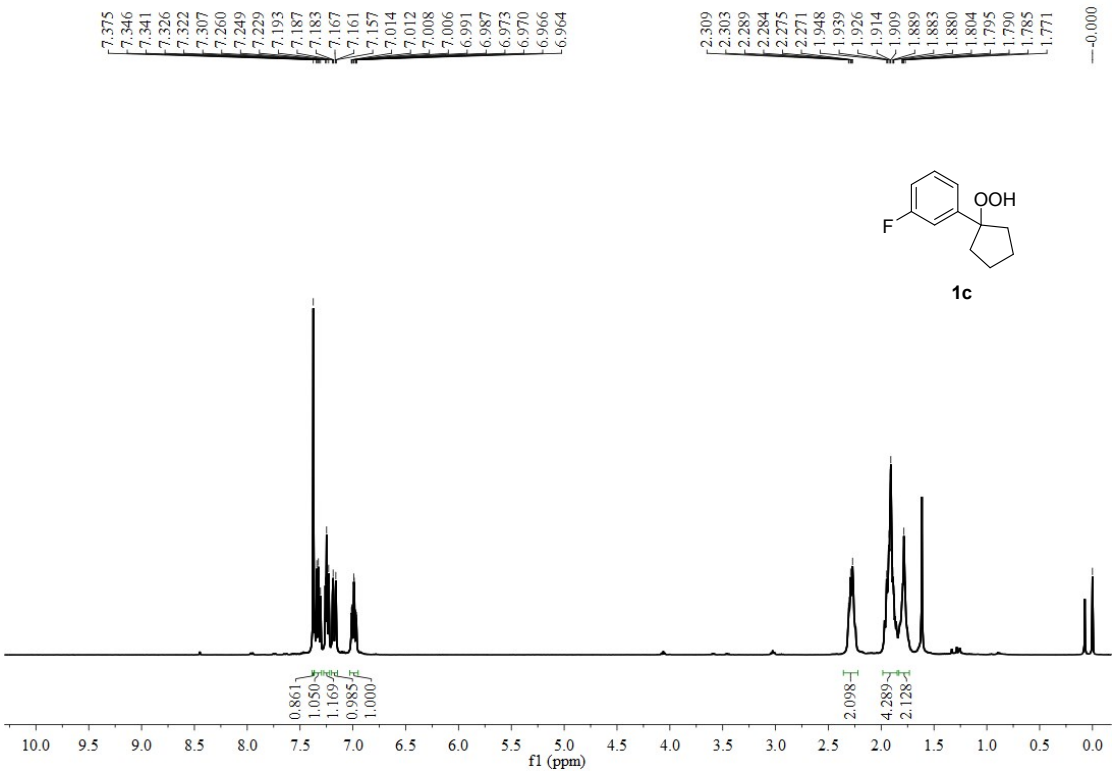


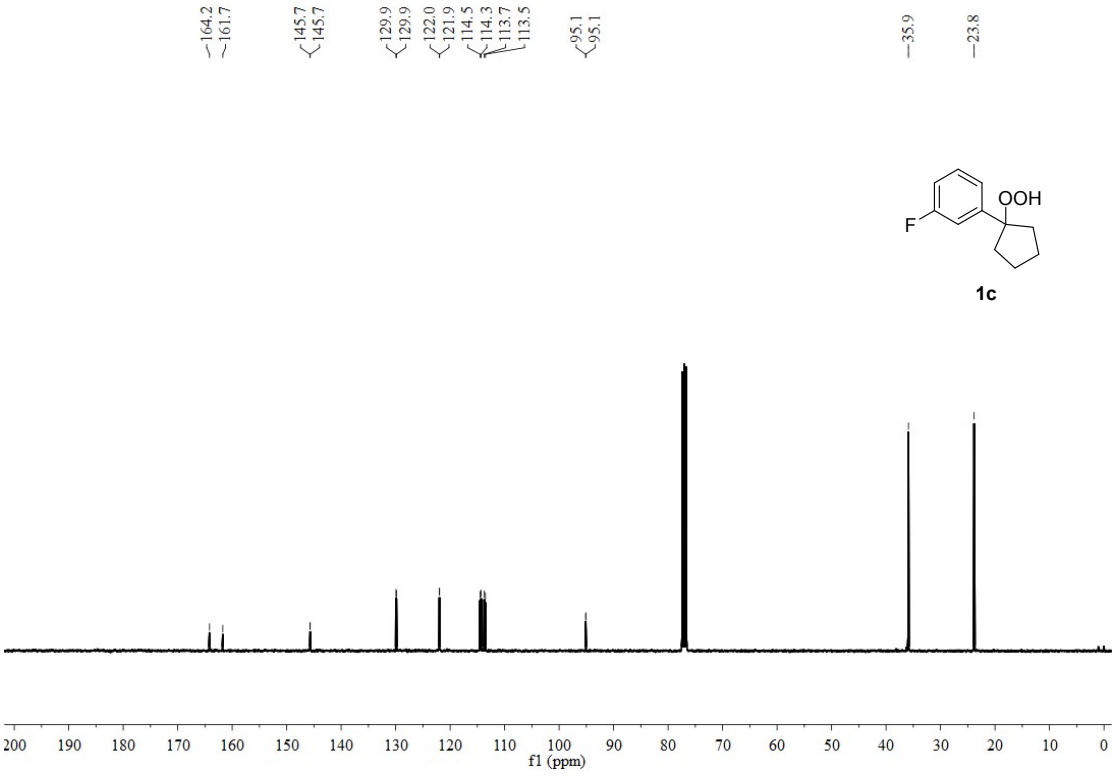
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1b**



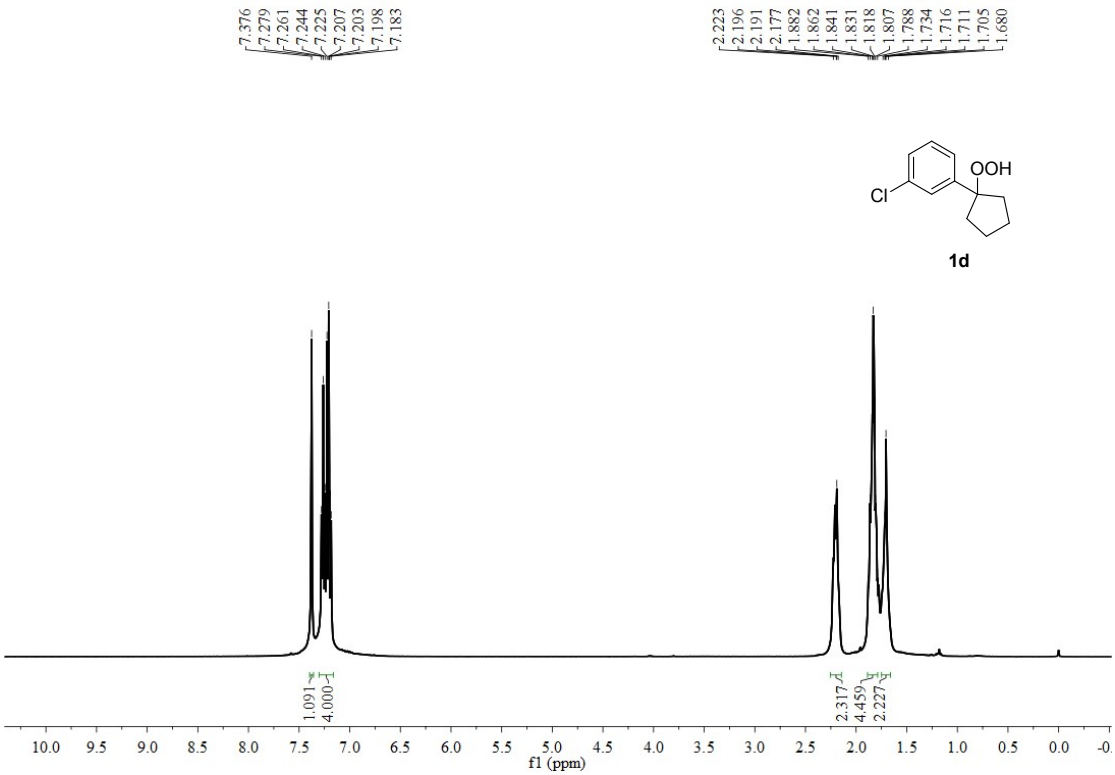


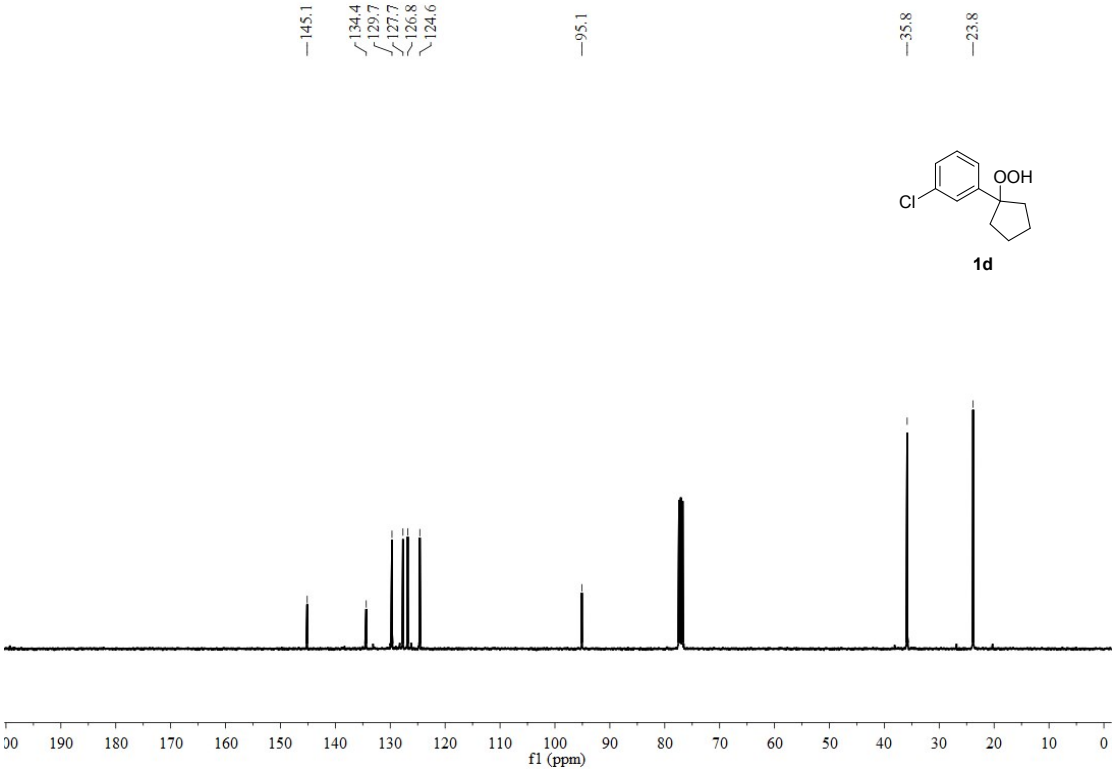
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1c**



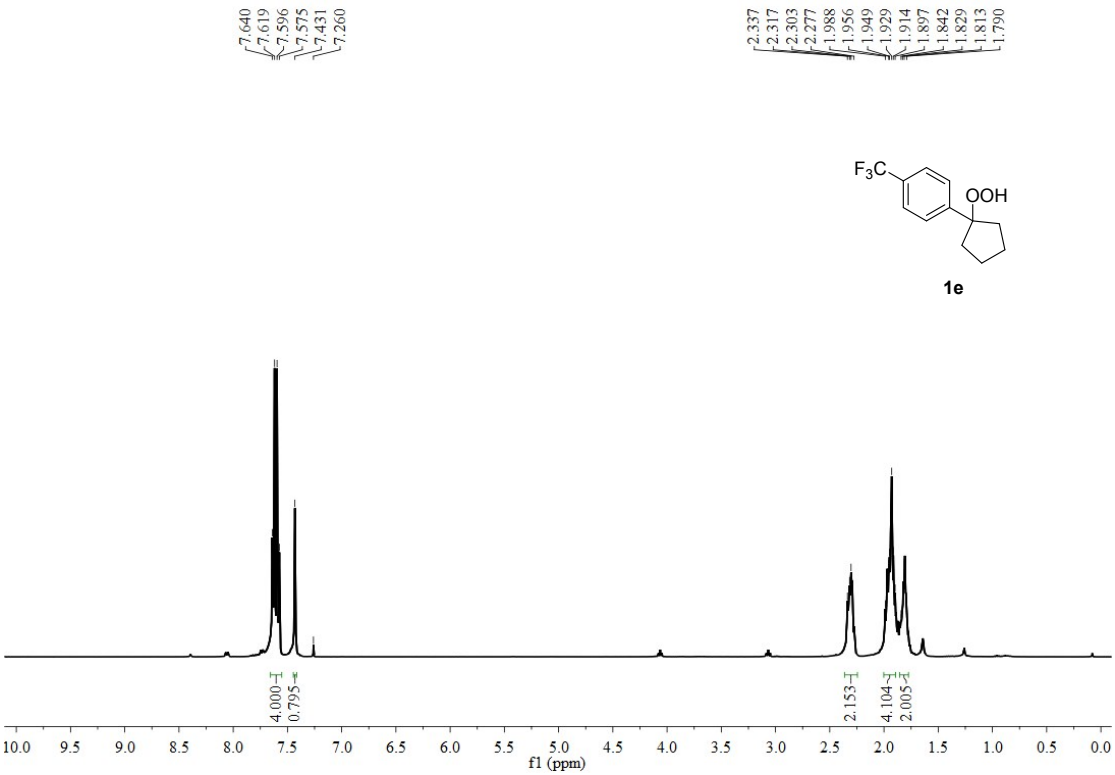


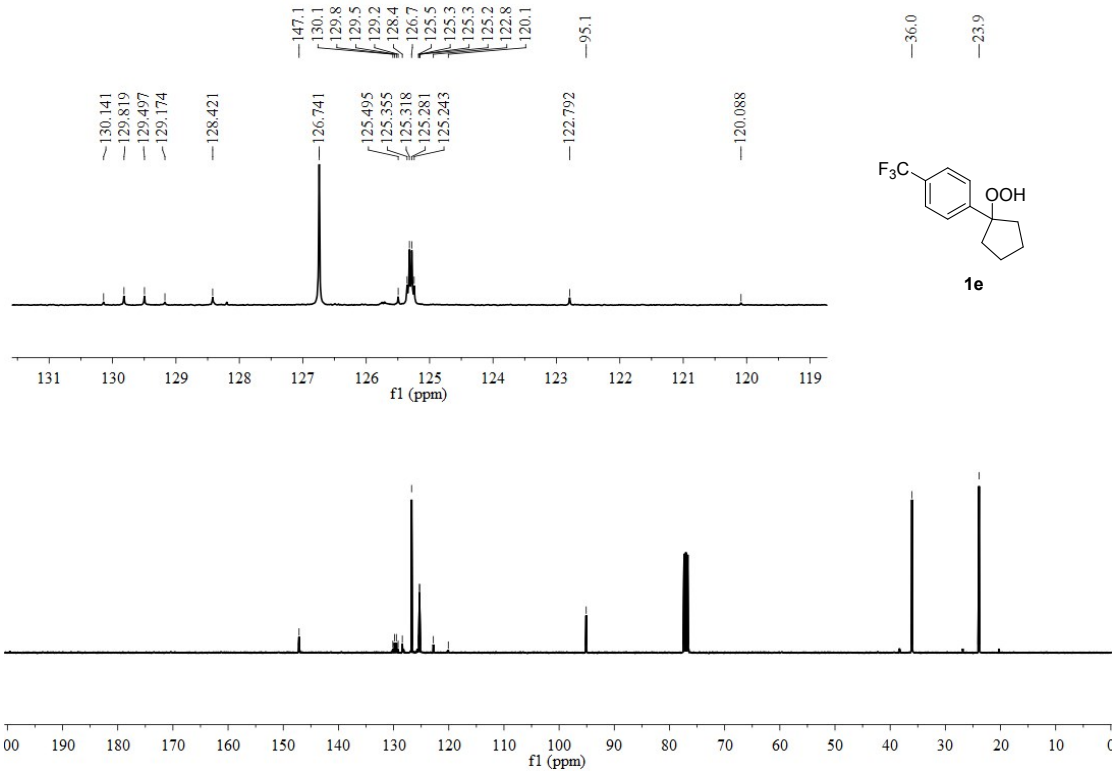
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1d**



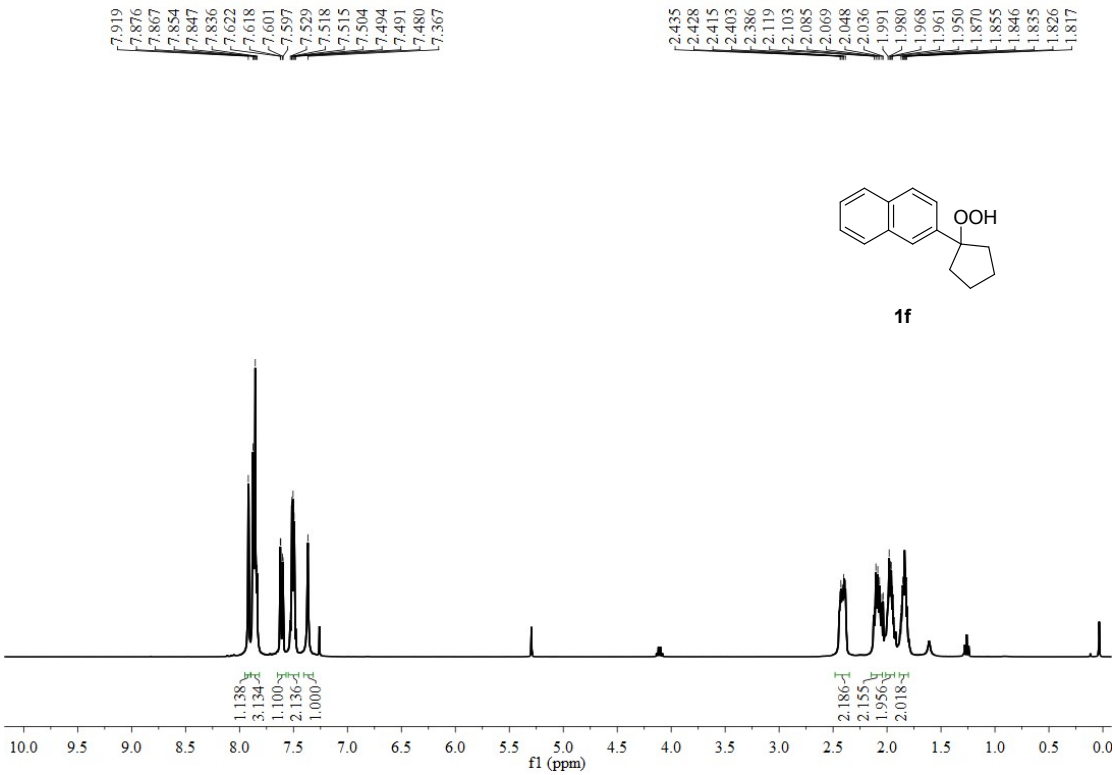


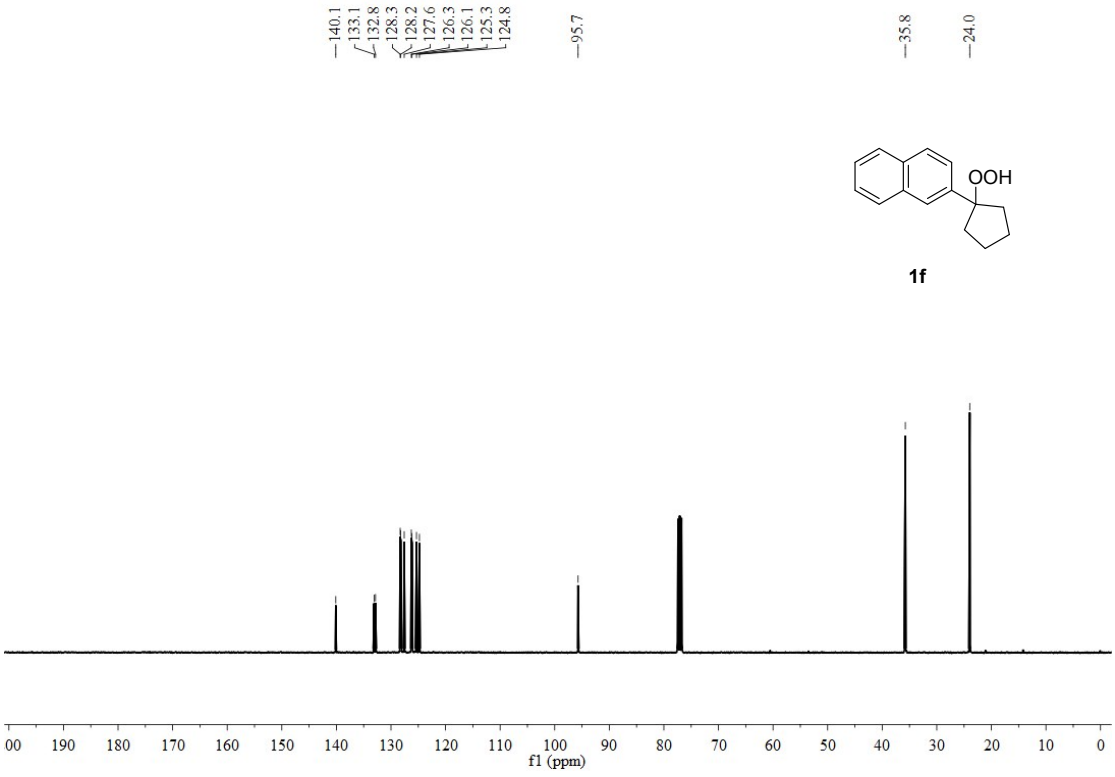
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1e**



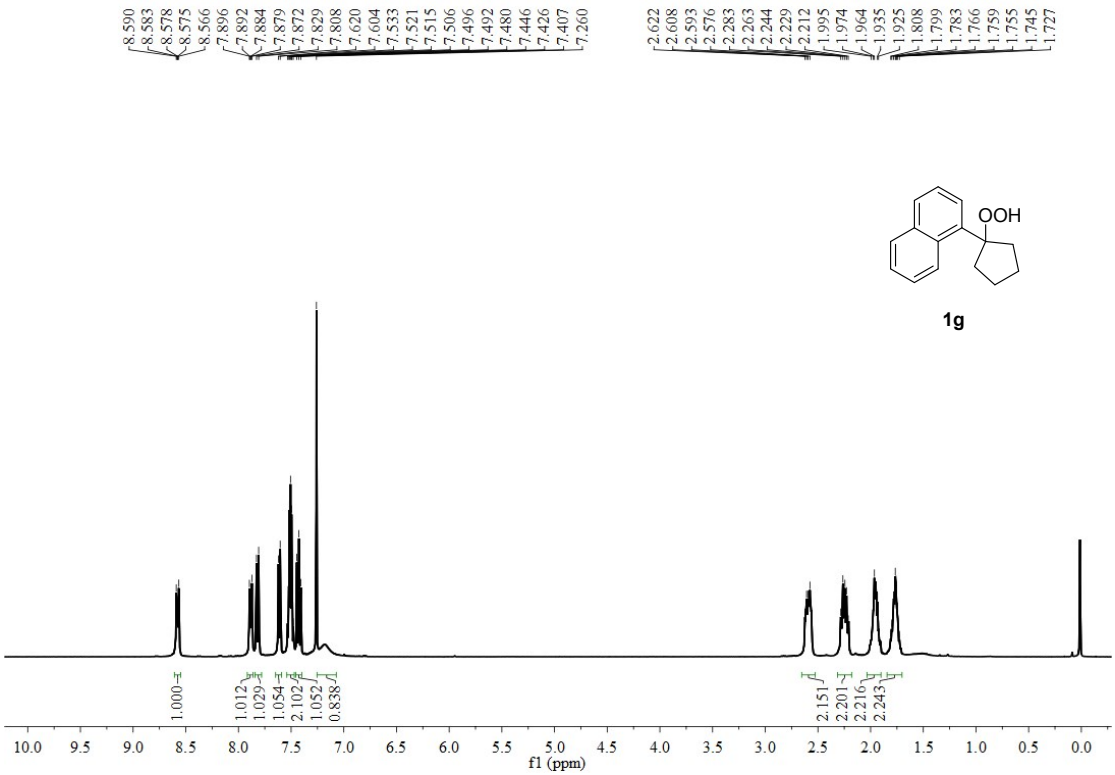


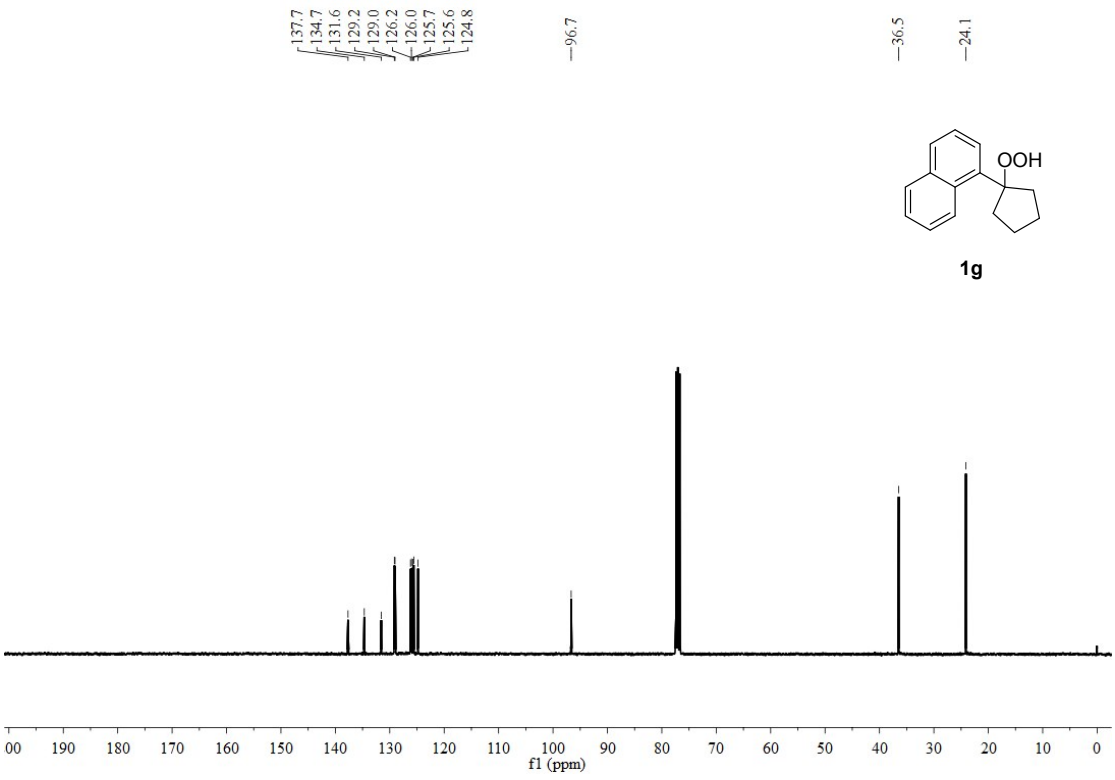
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1f**



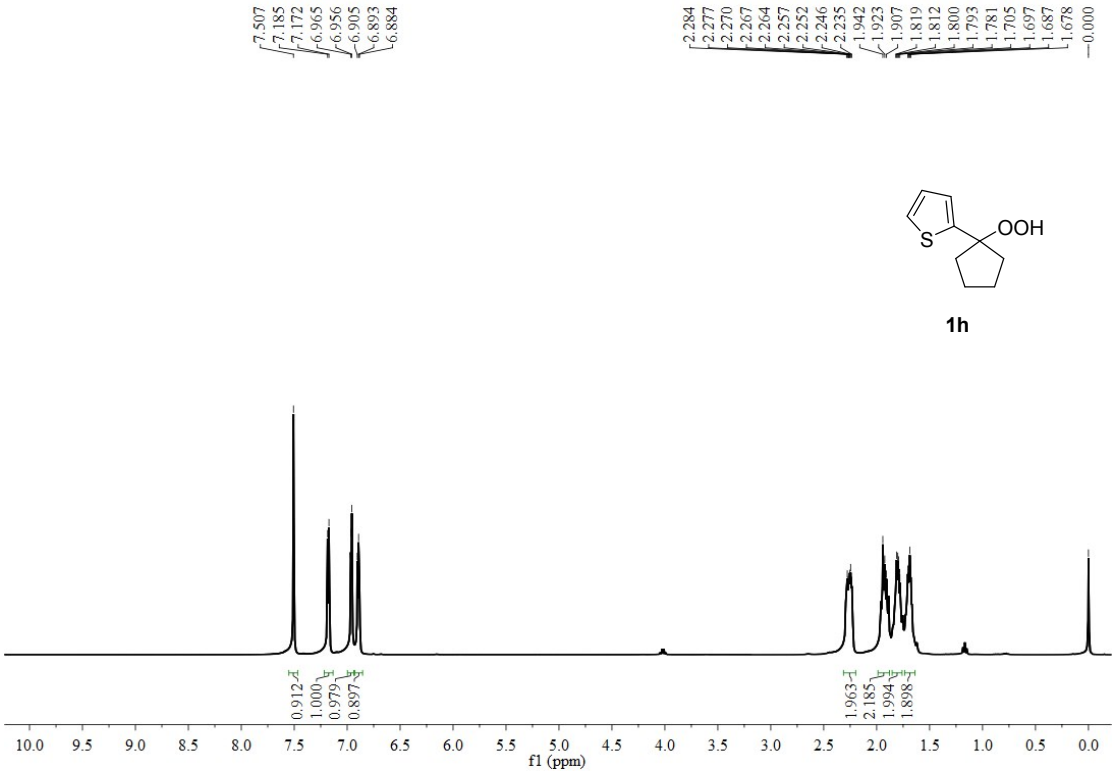


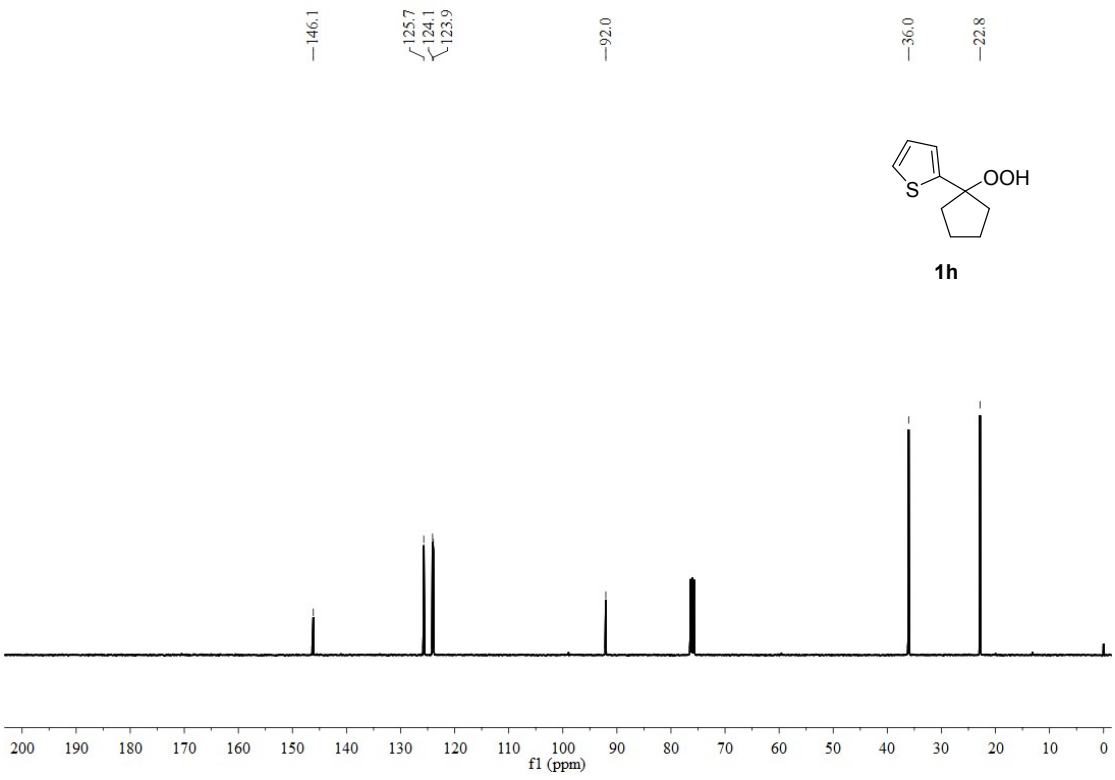
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1g**



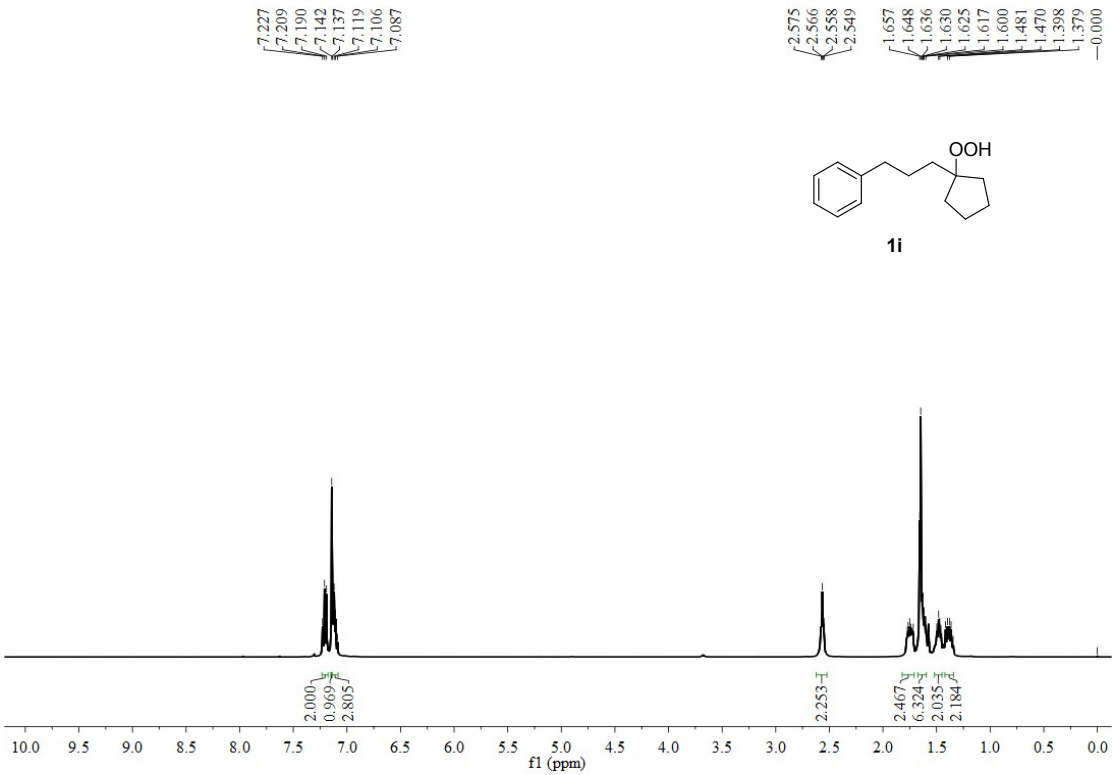


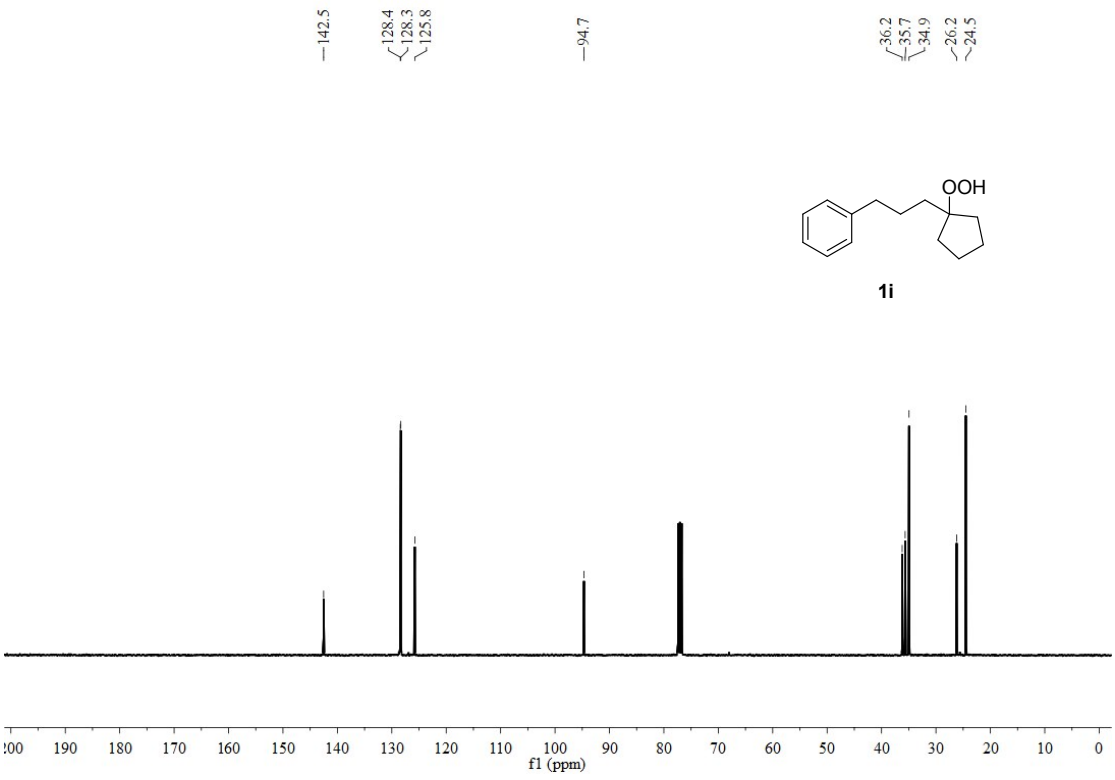
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1h**



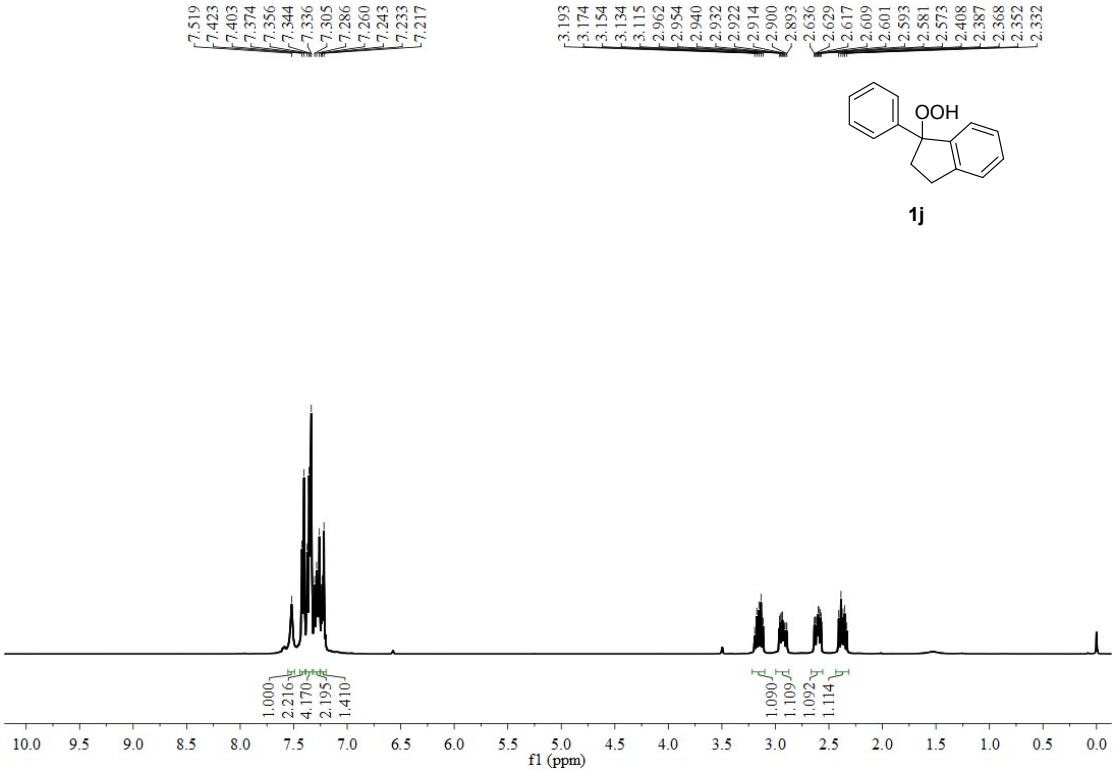


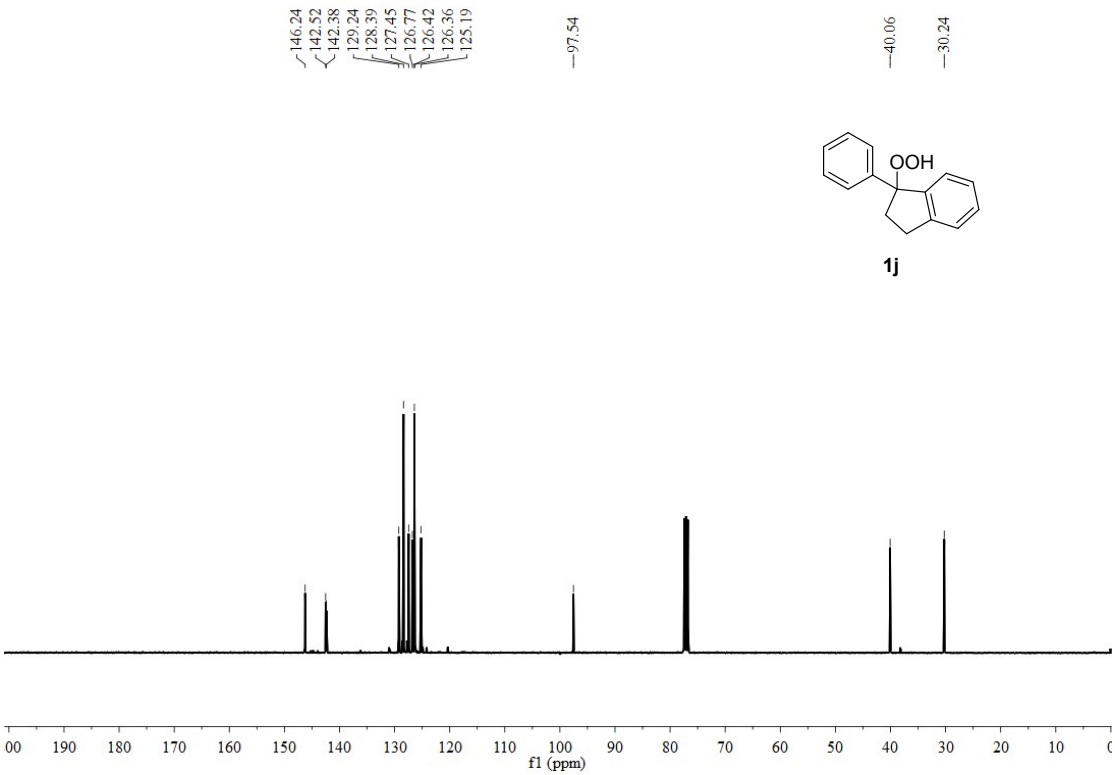
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1i**



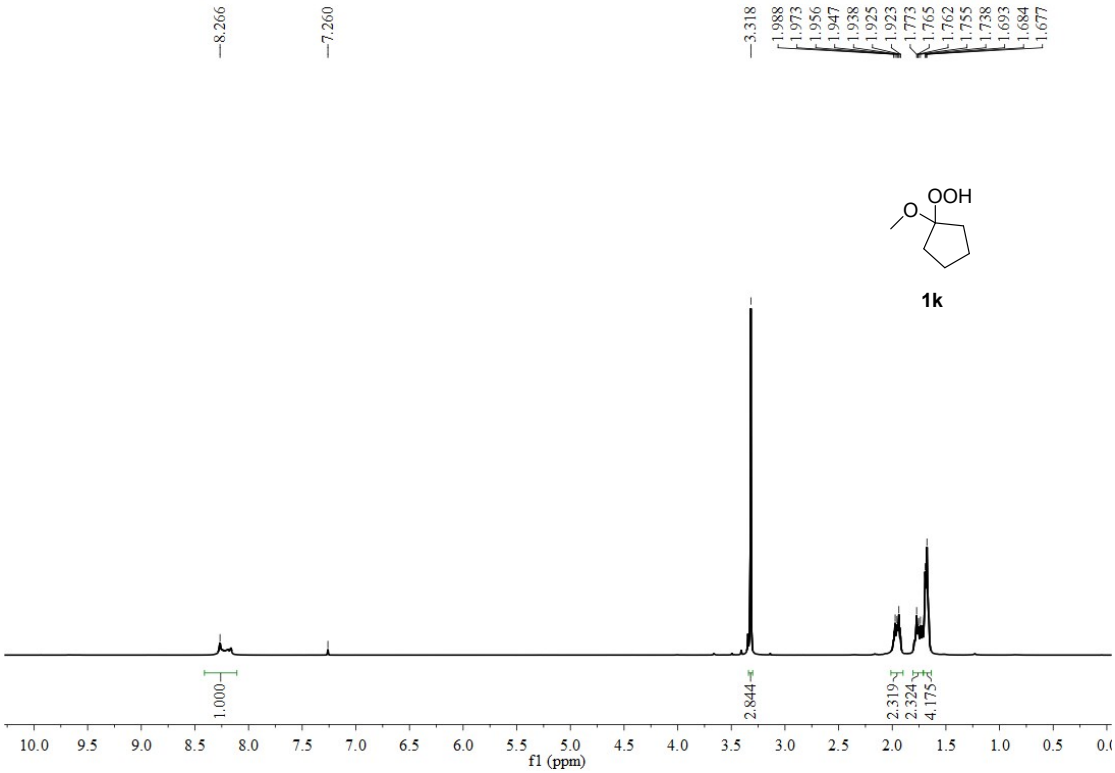


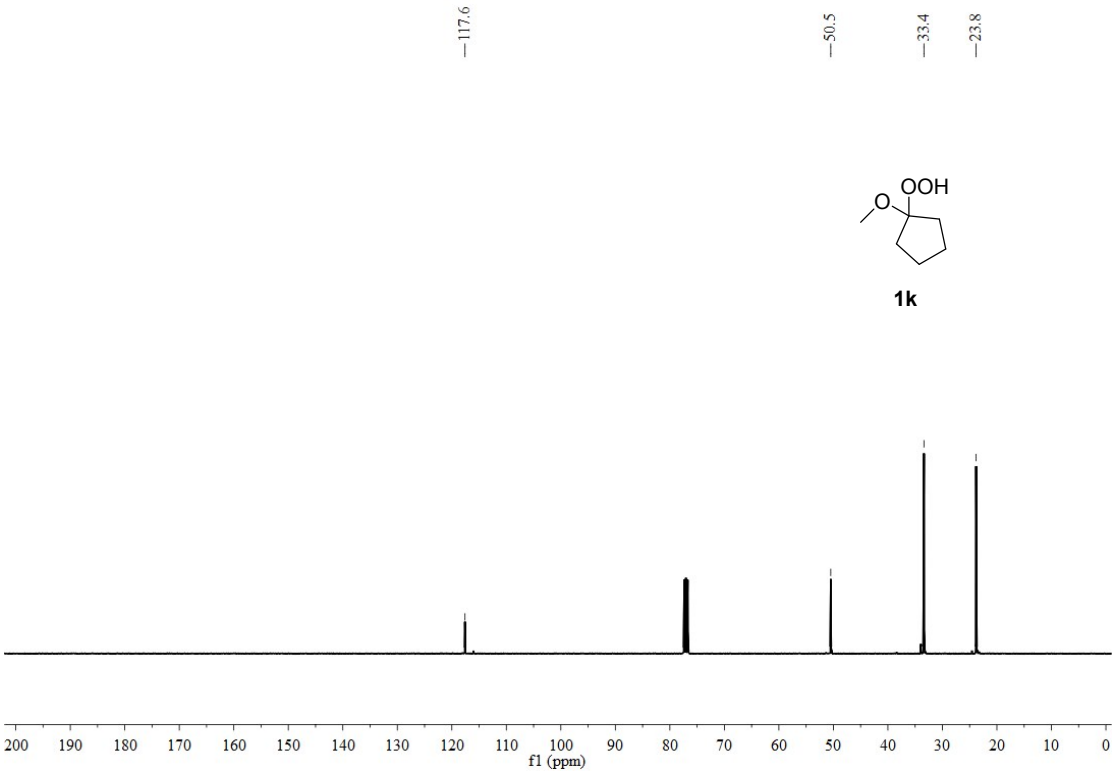
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1j**



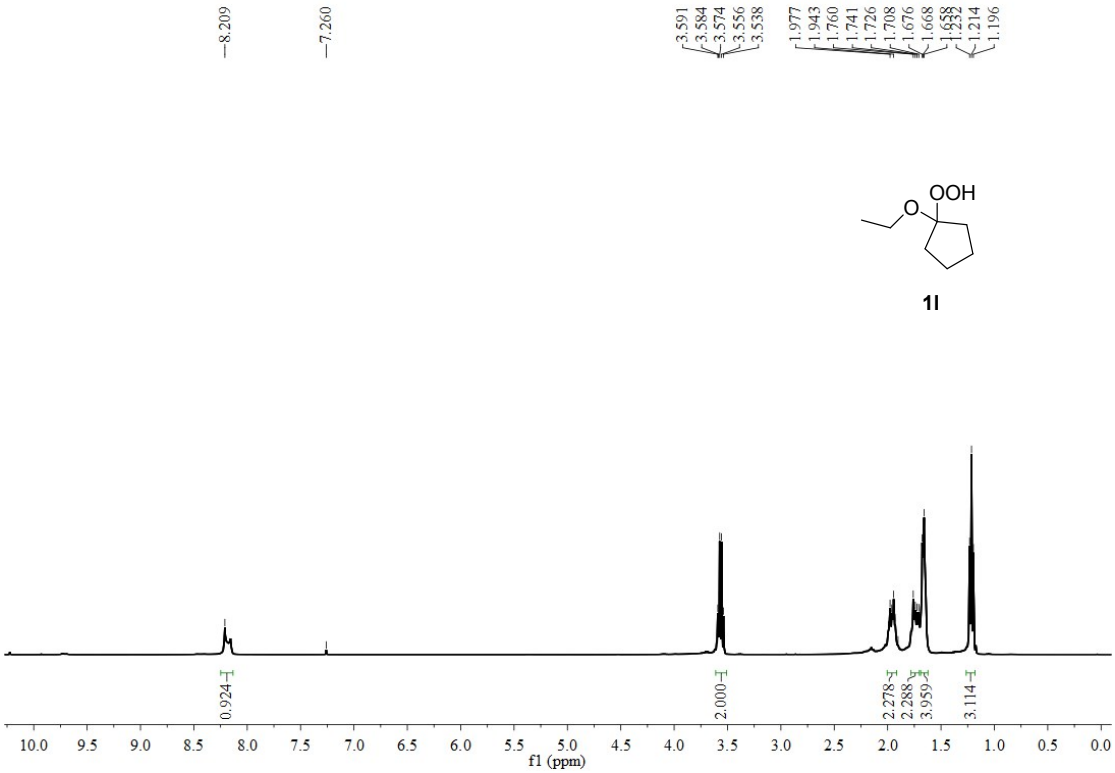


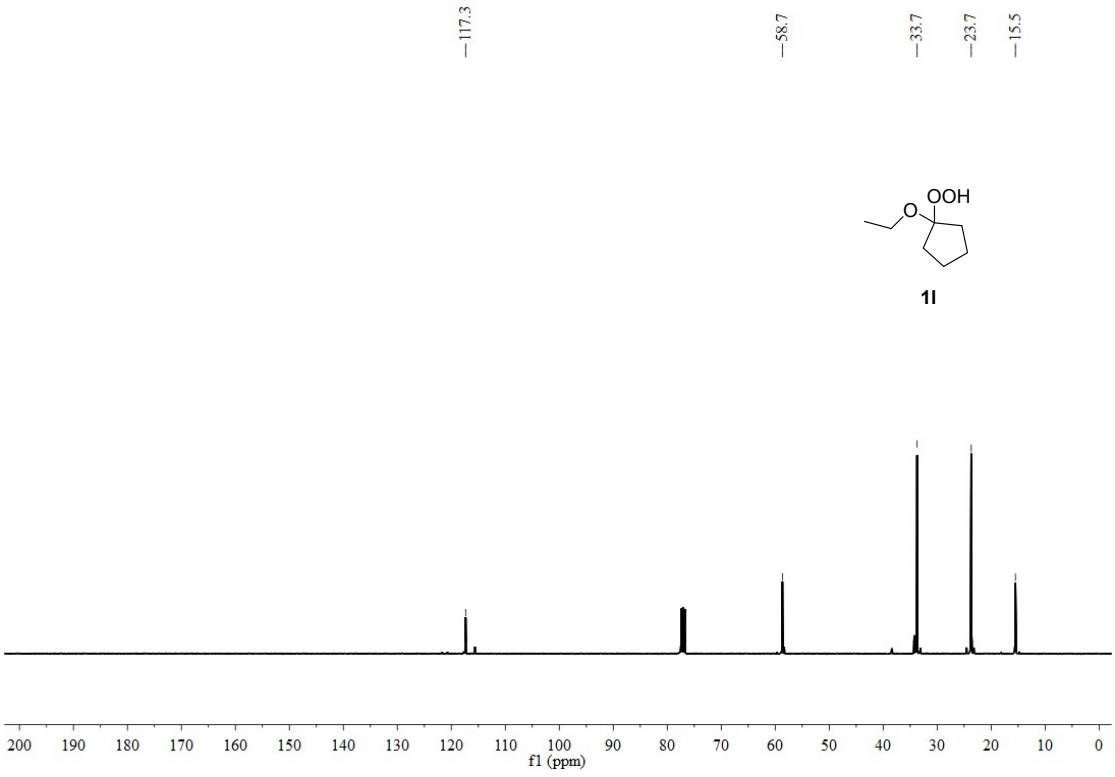
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1k**



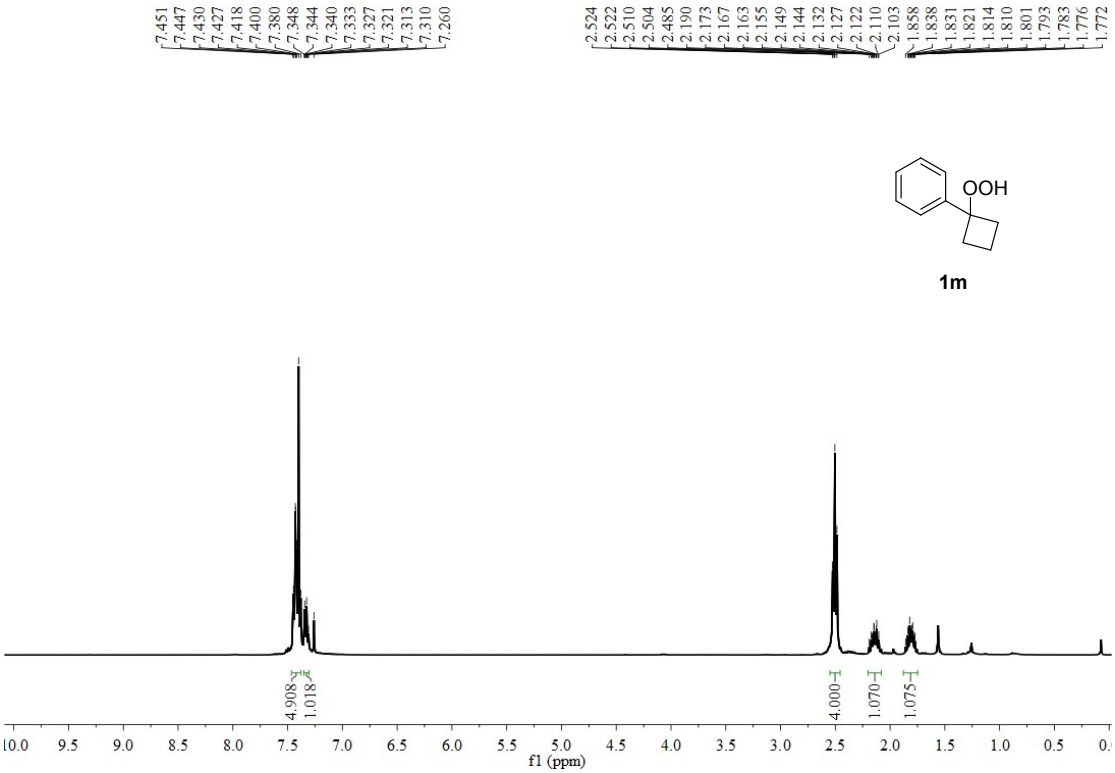


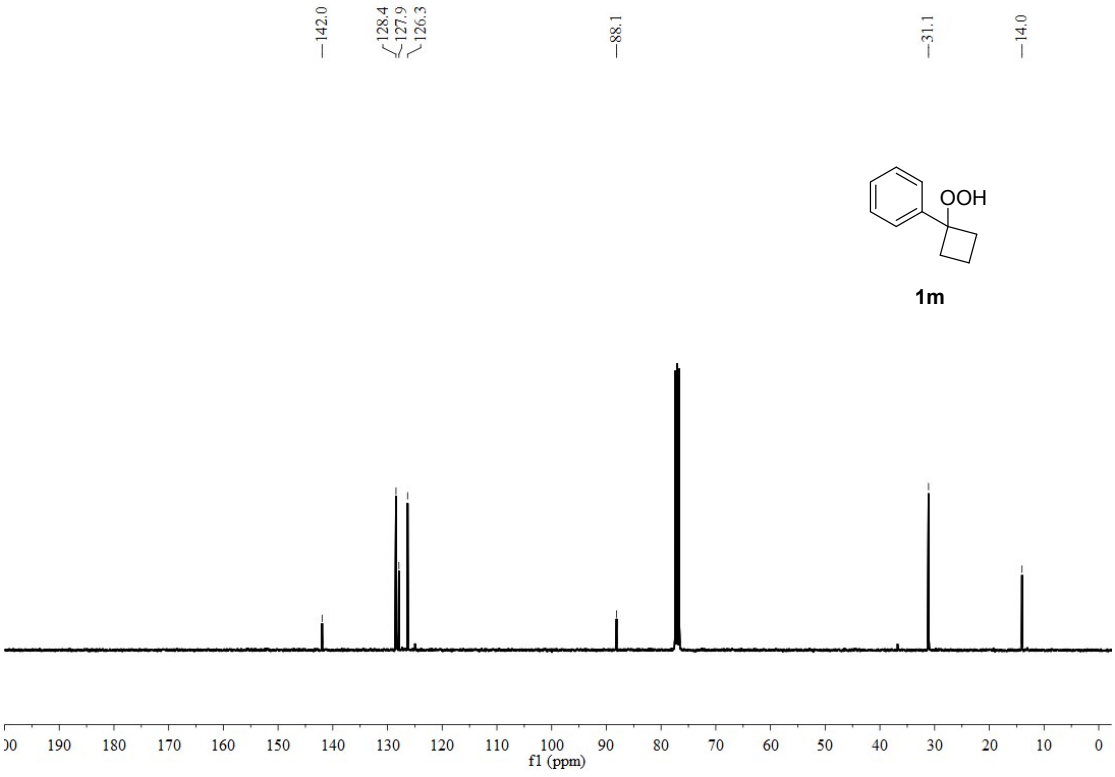
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1l**



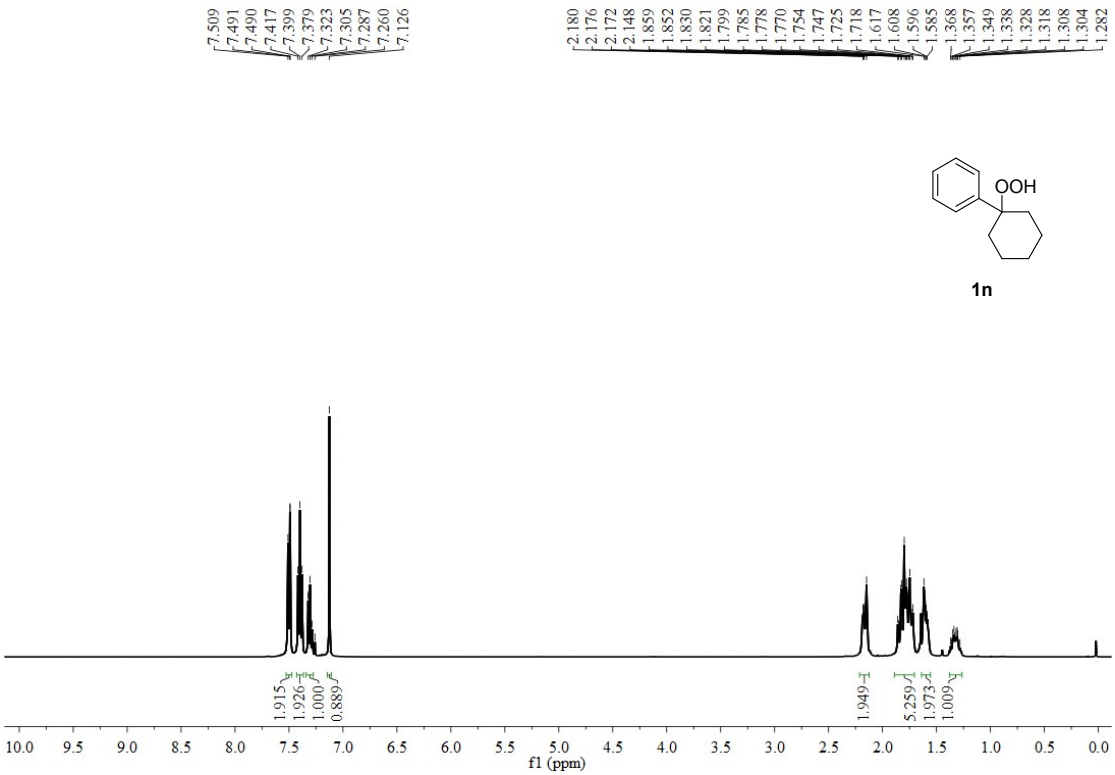


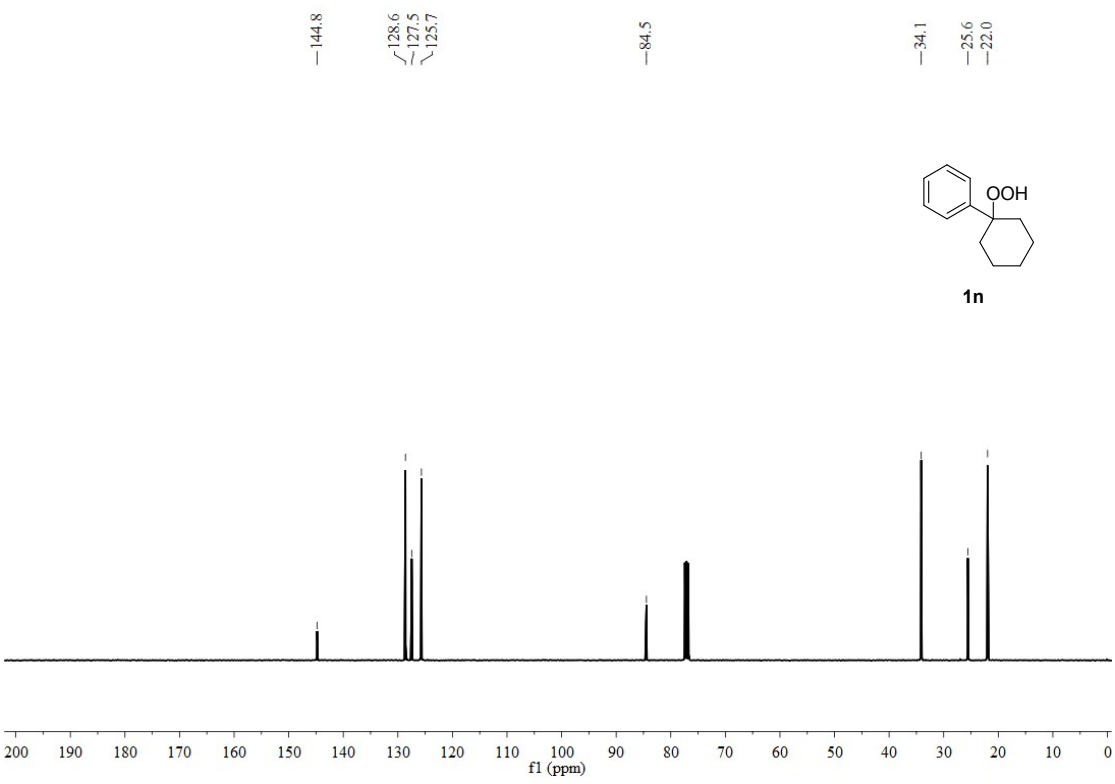
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1m**



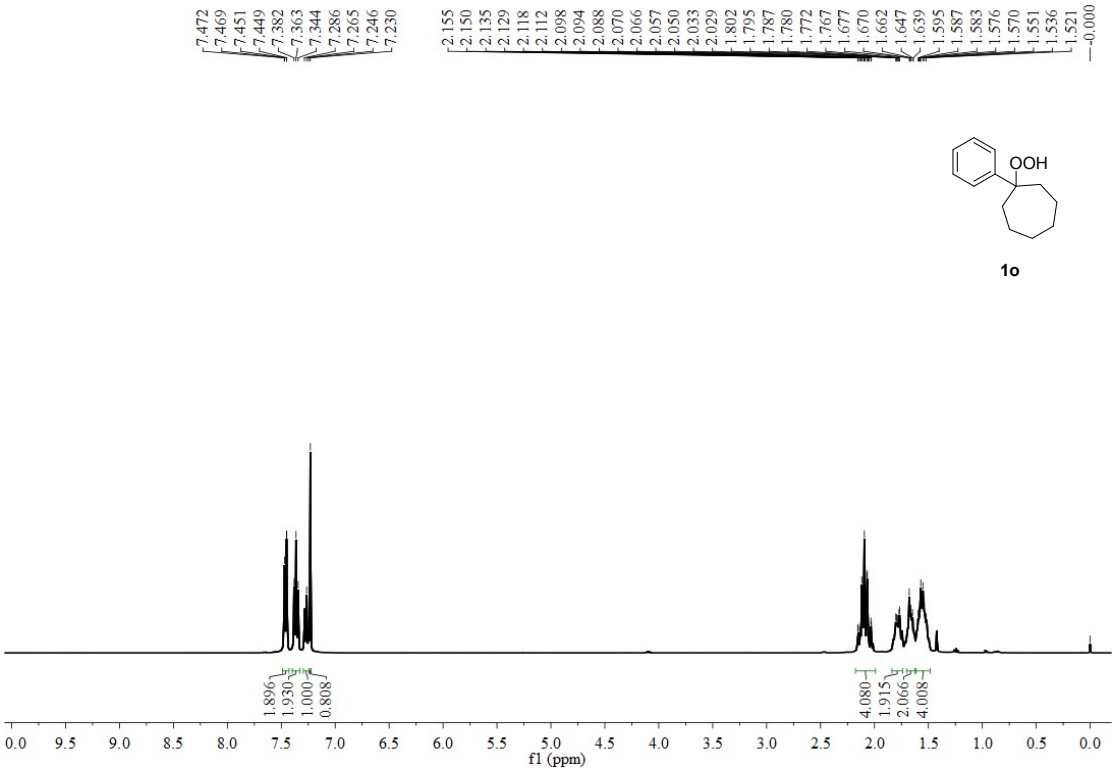


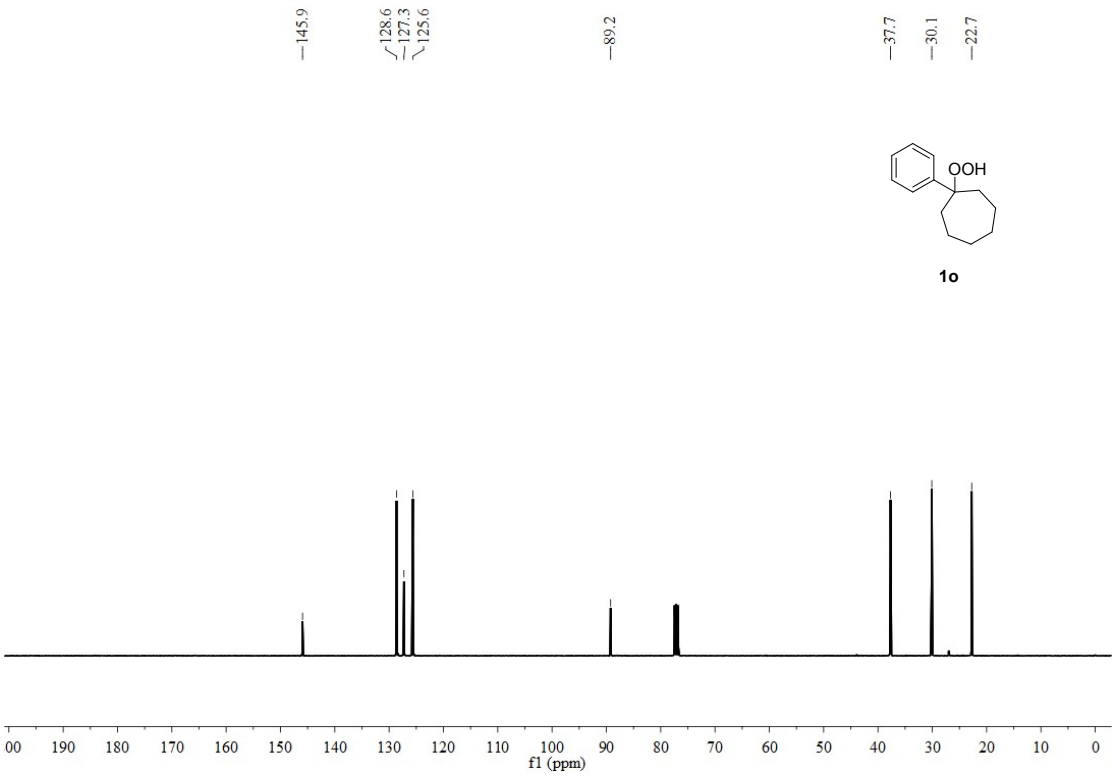
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1n**



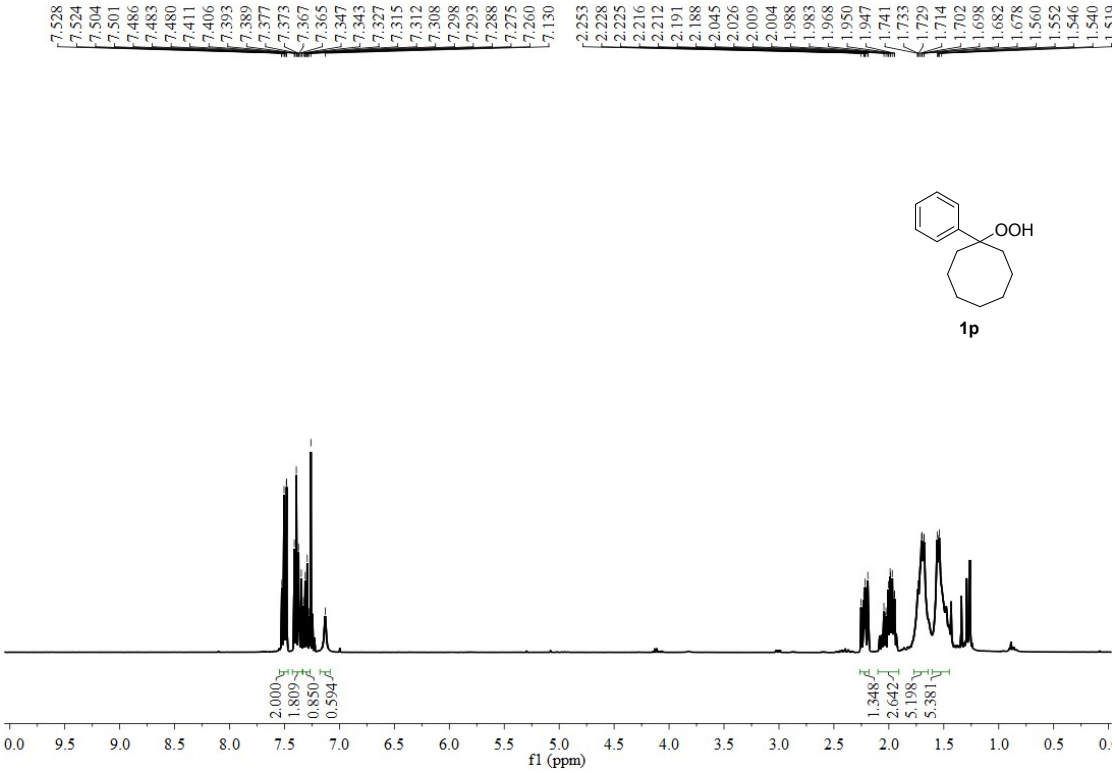


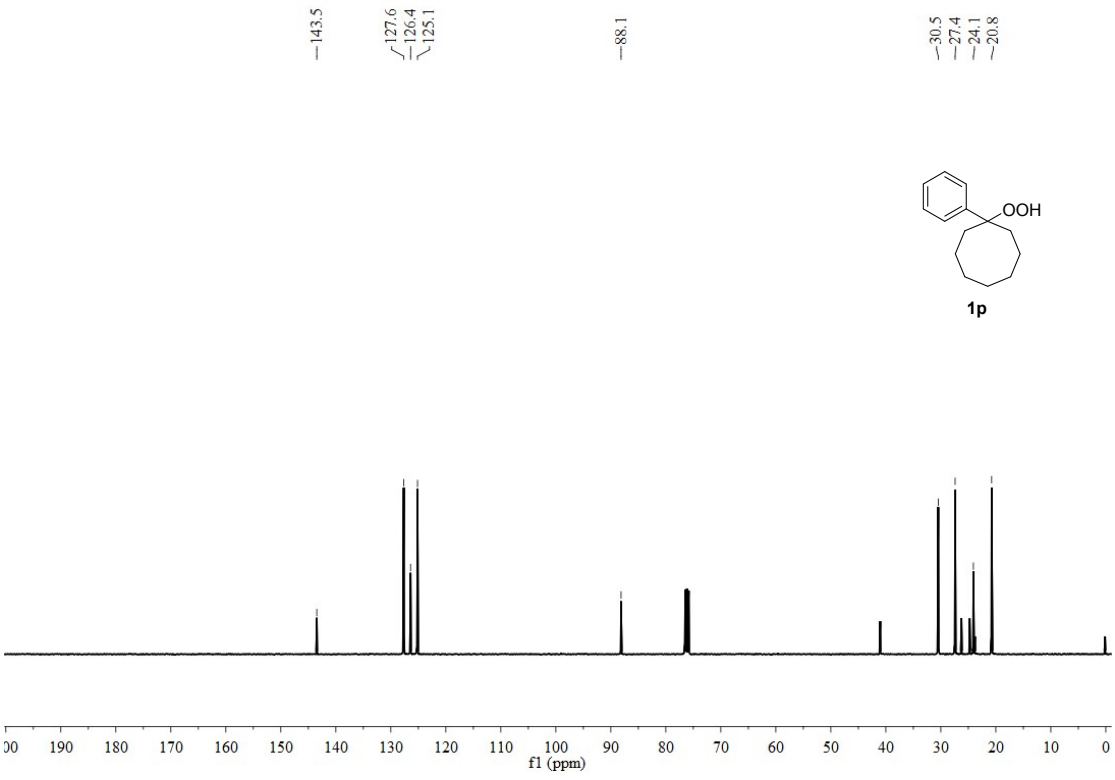
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1o**



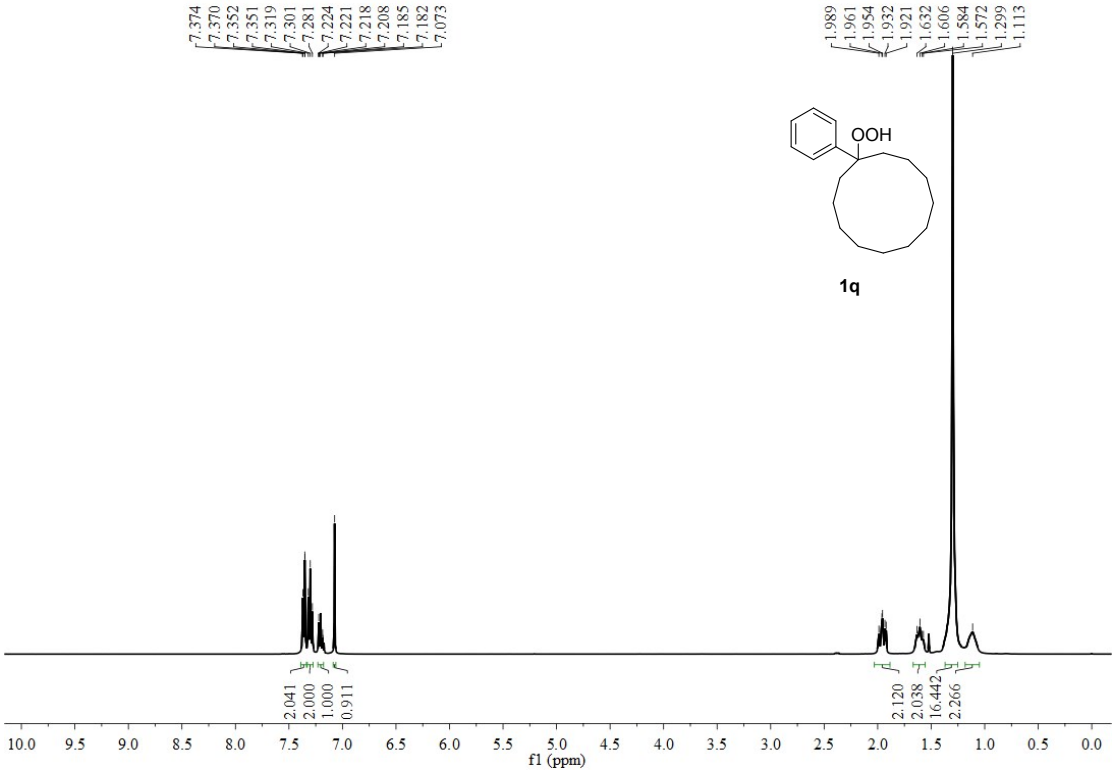


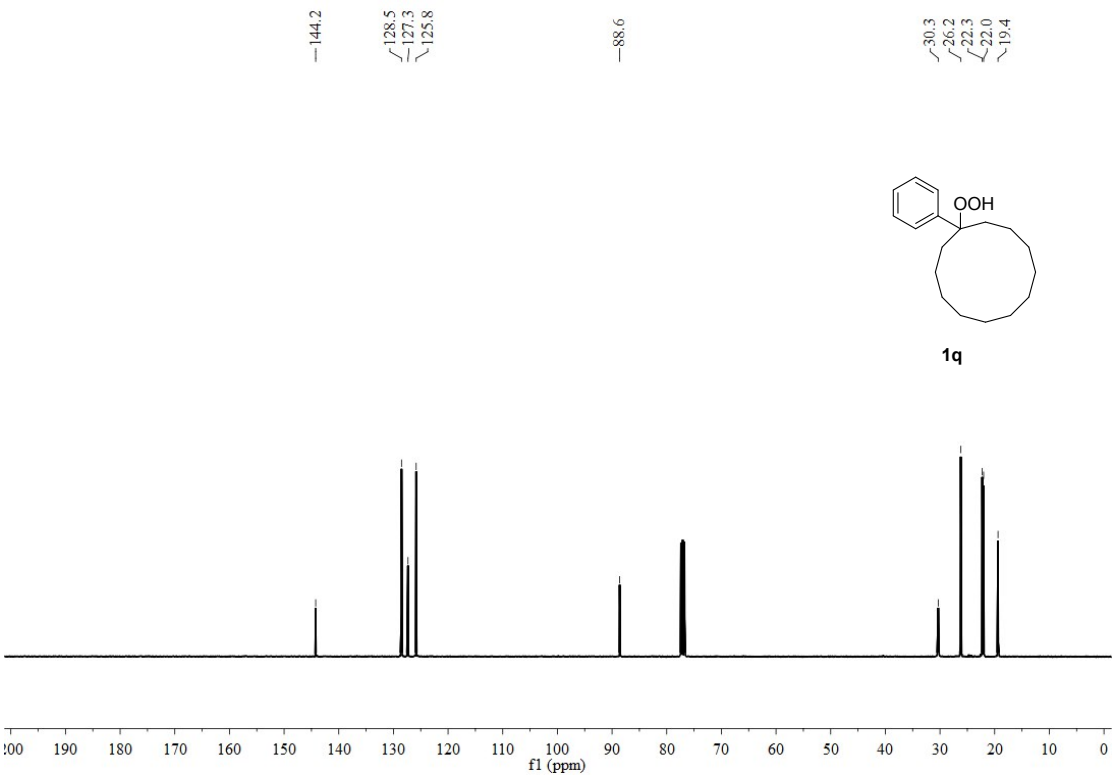
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1p**



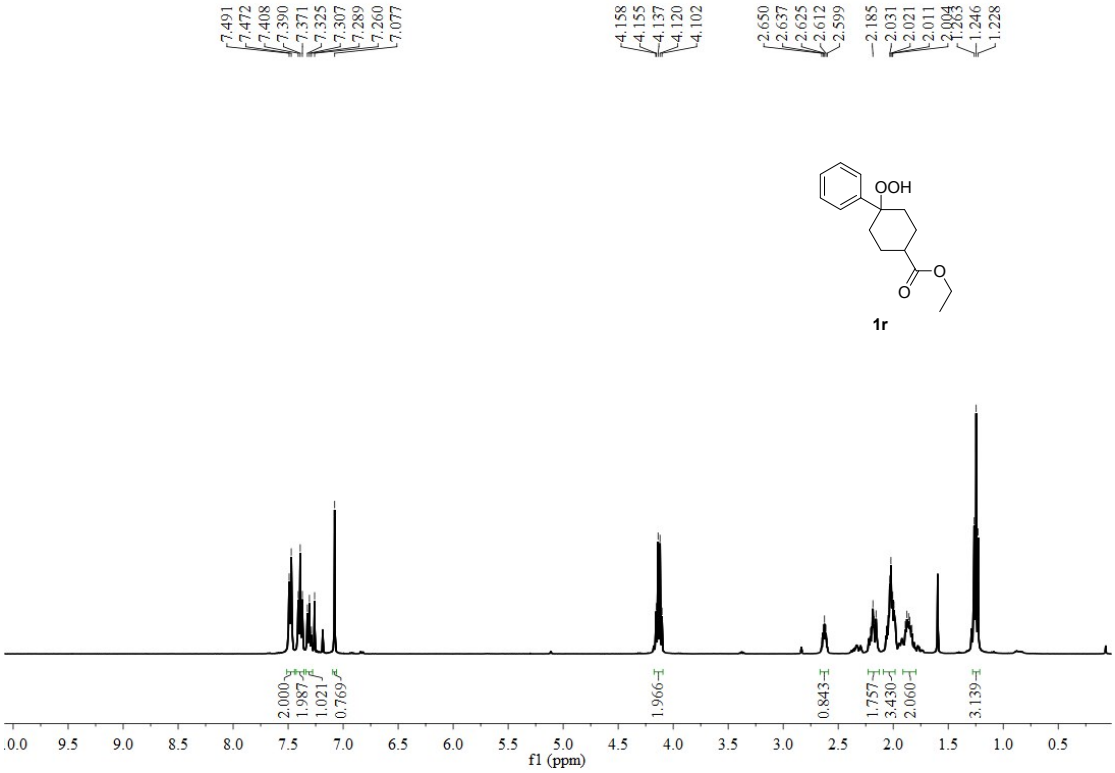


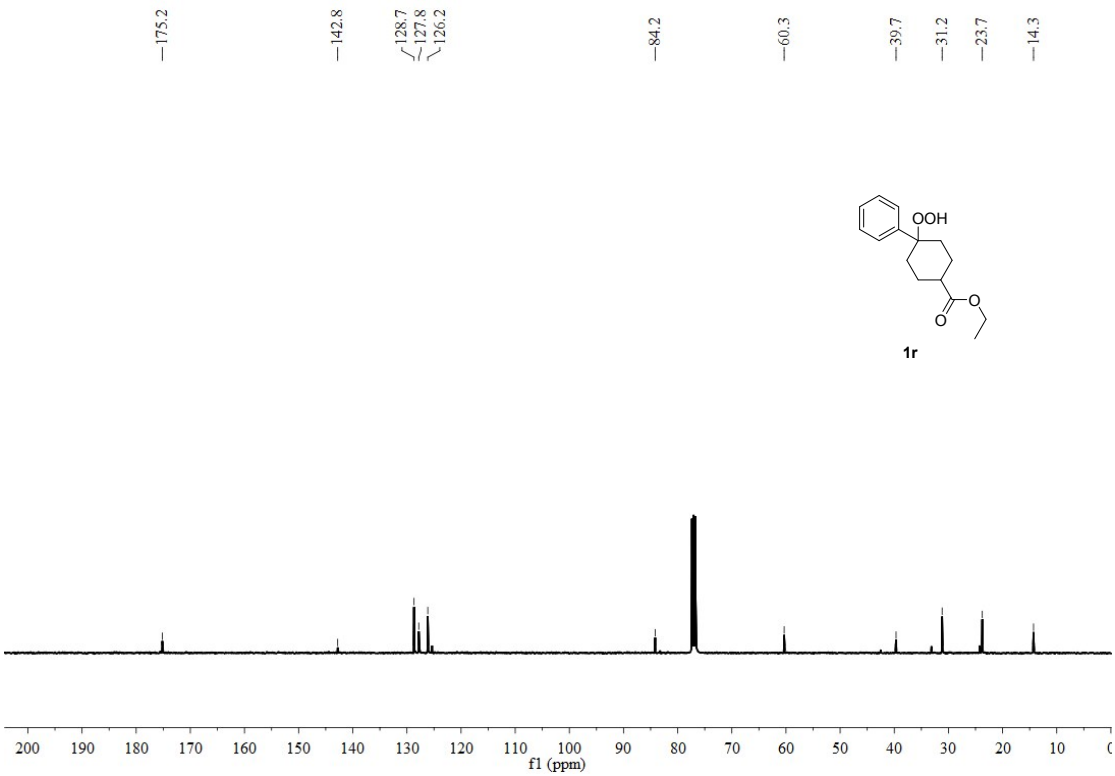
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1q**



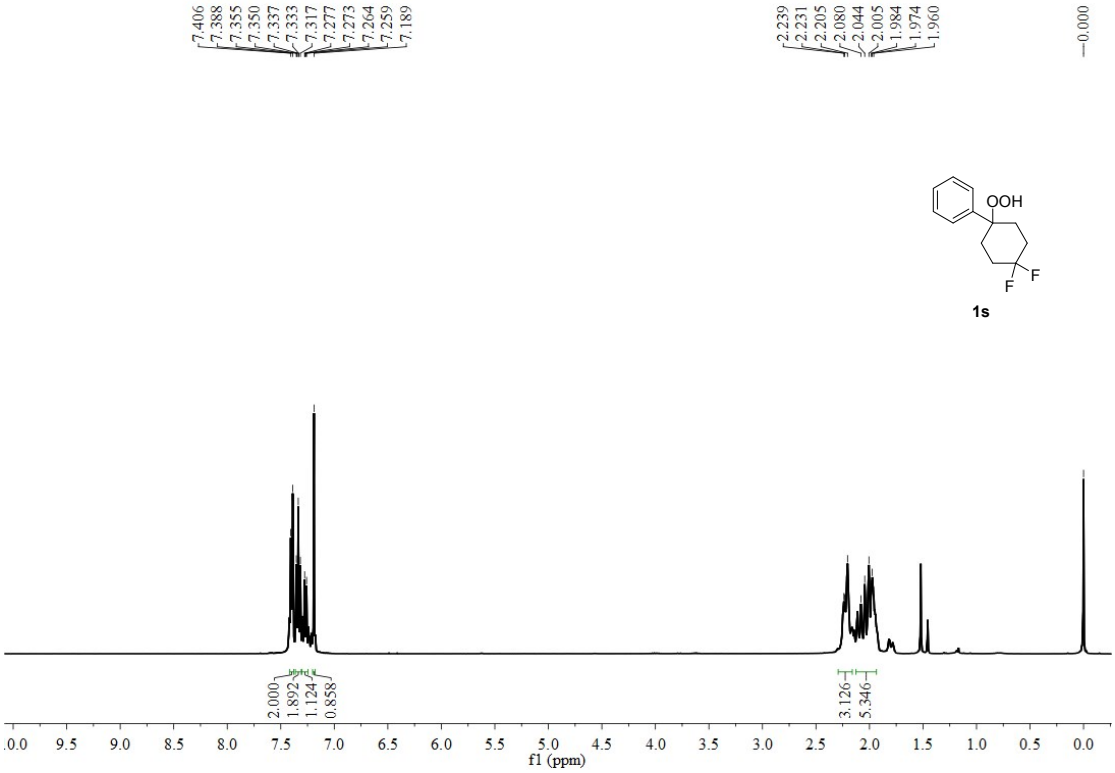


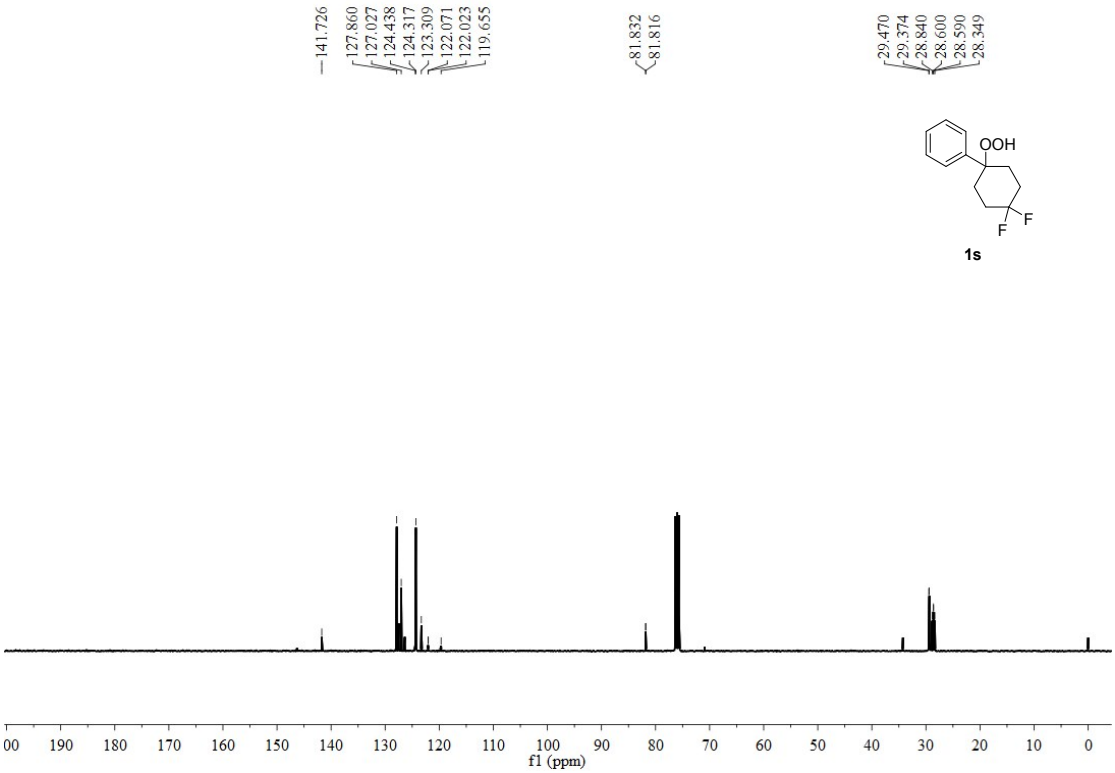
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1r**



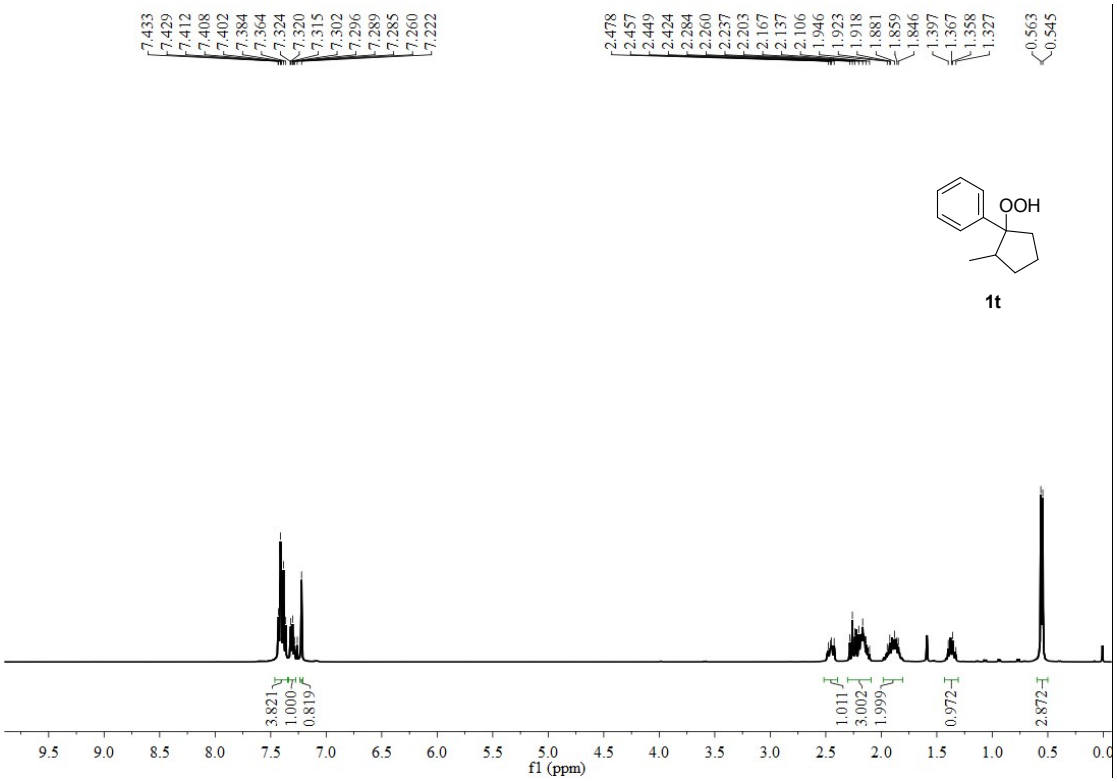


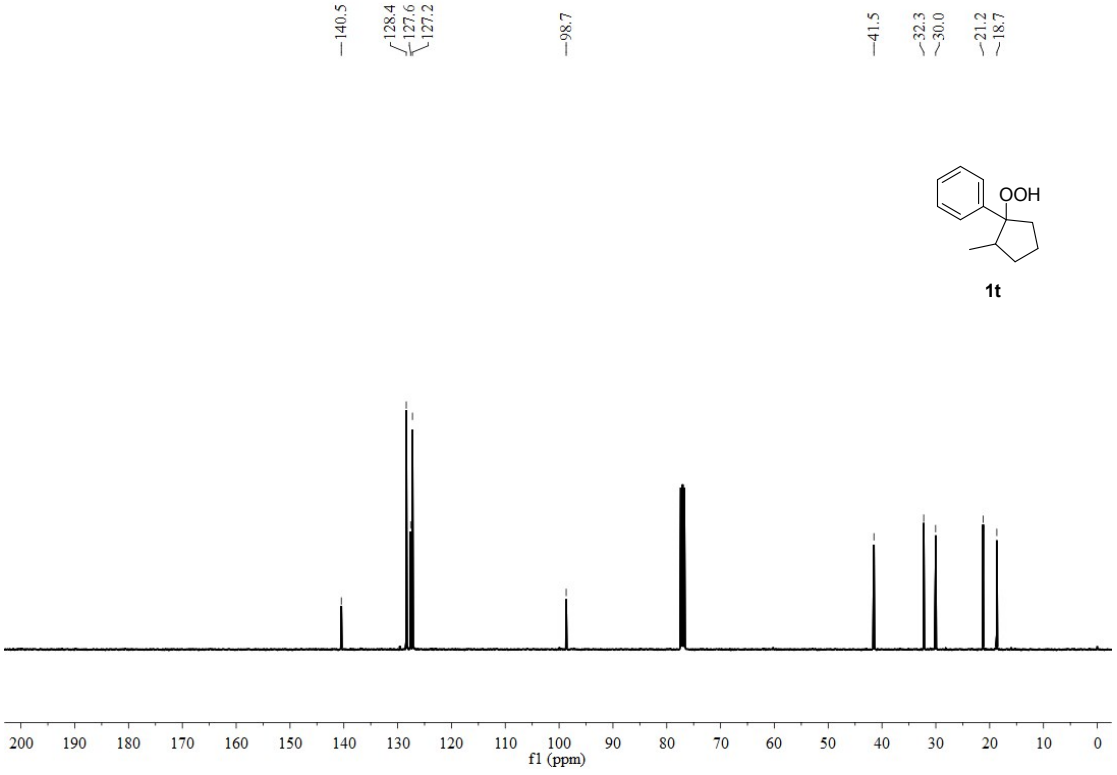
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1s**



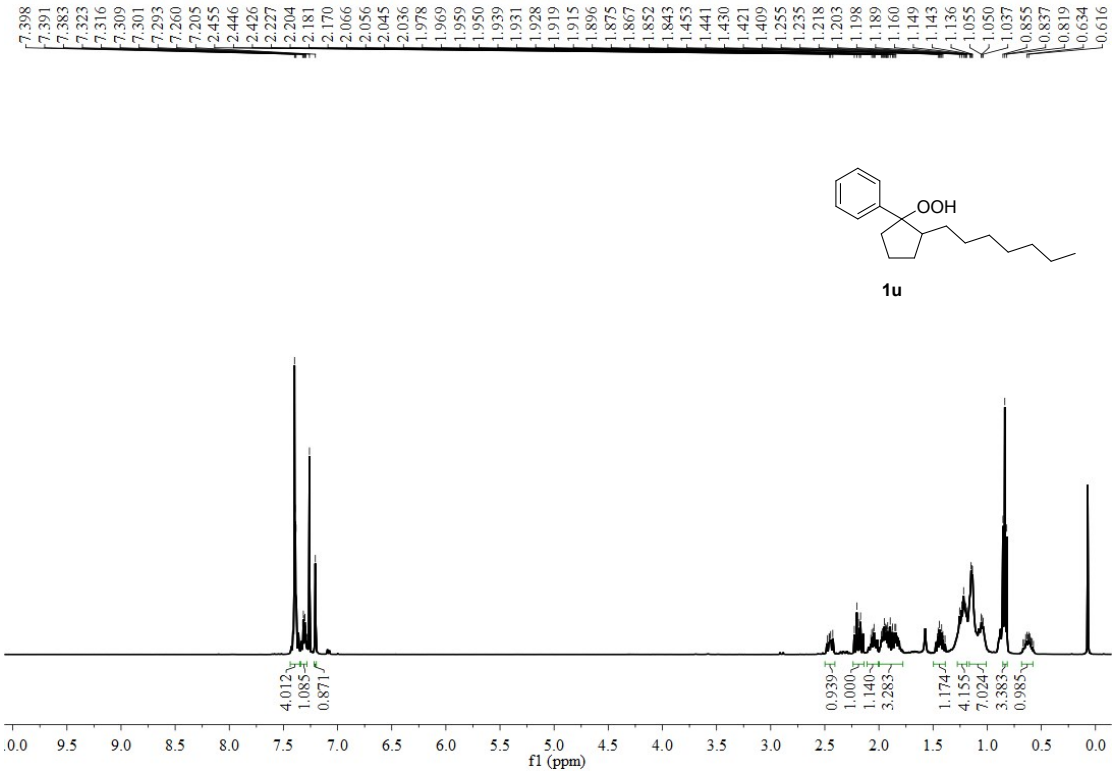


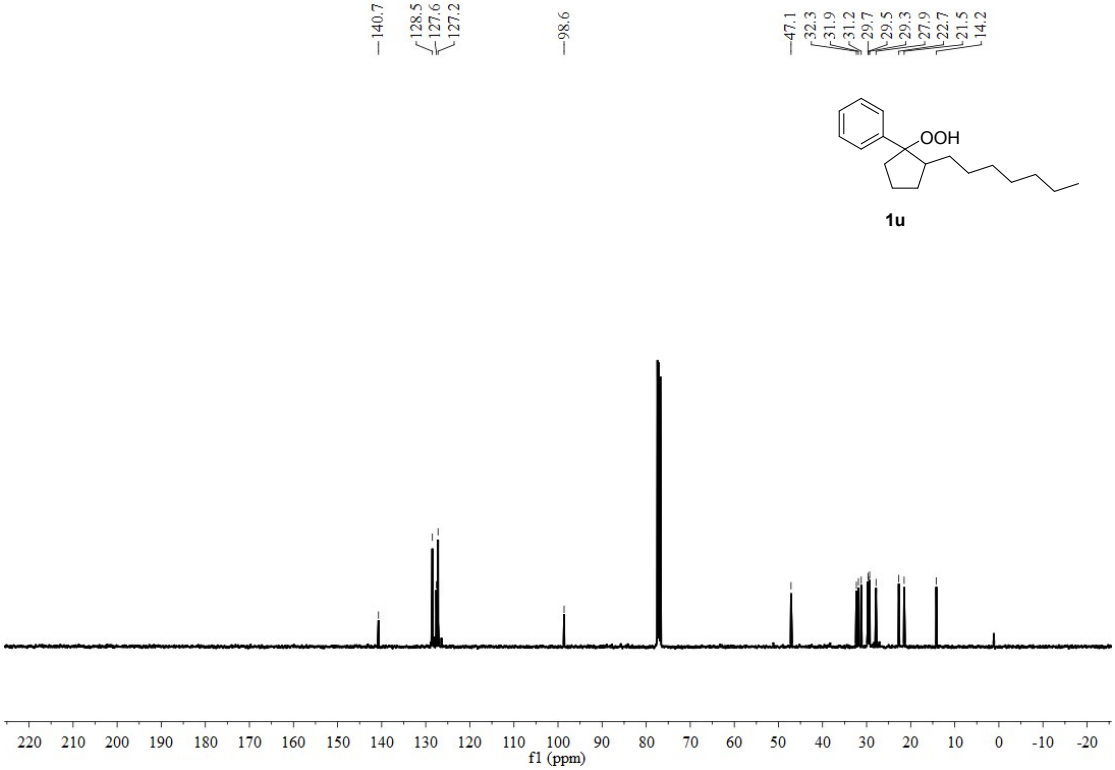
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1t**



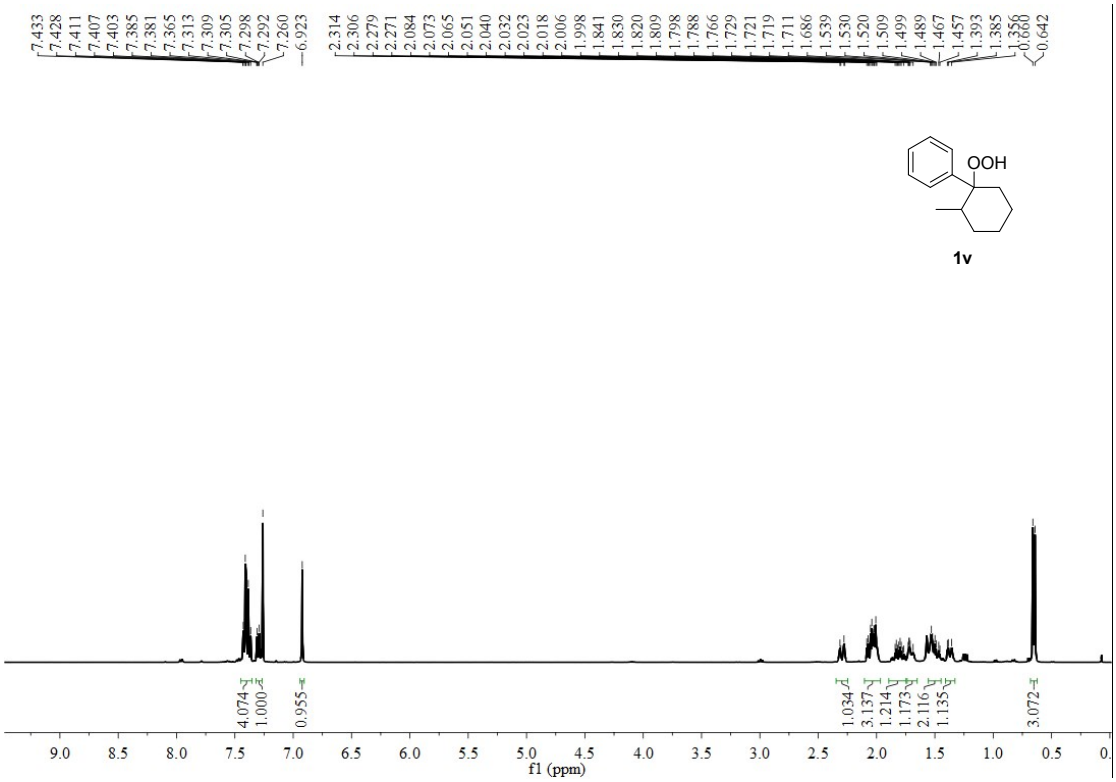


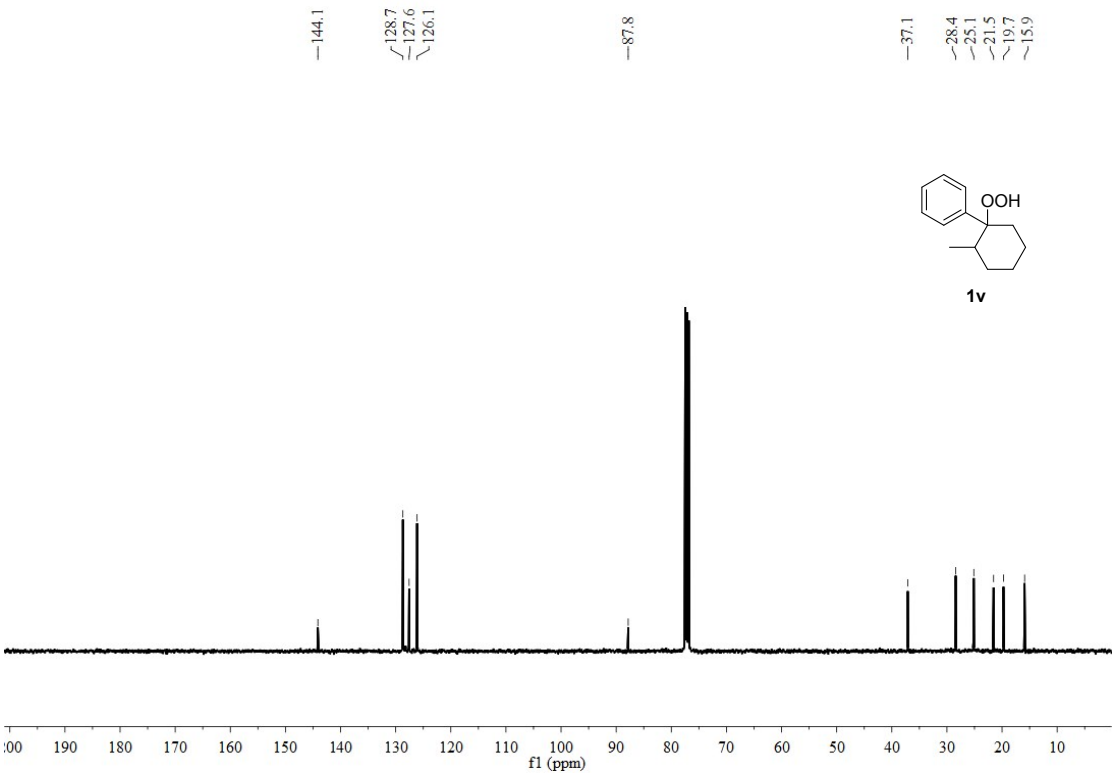
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1u**



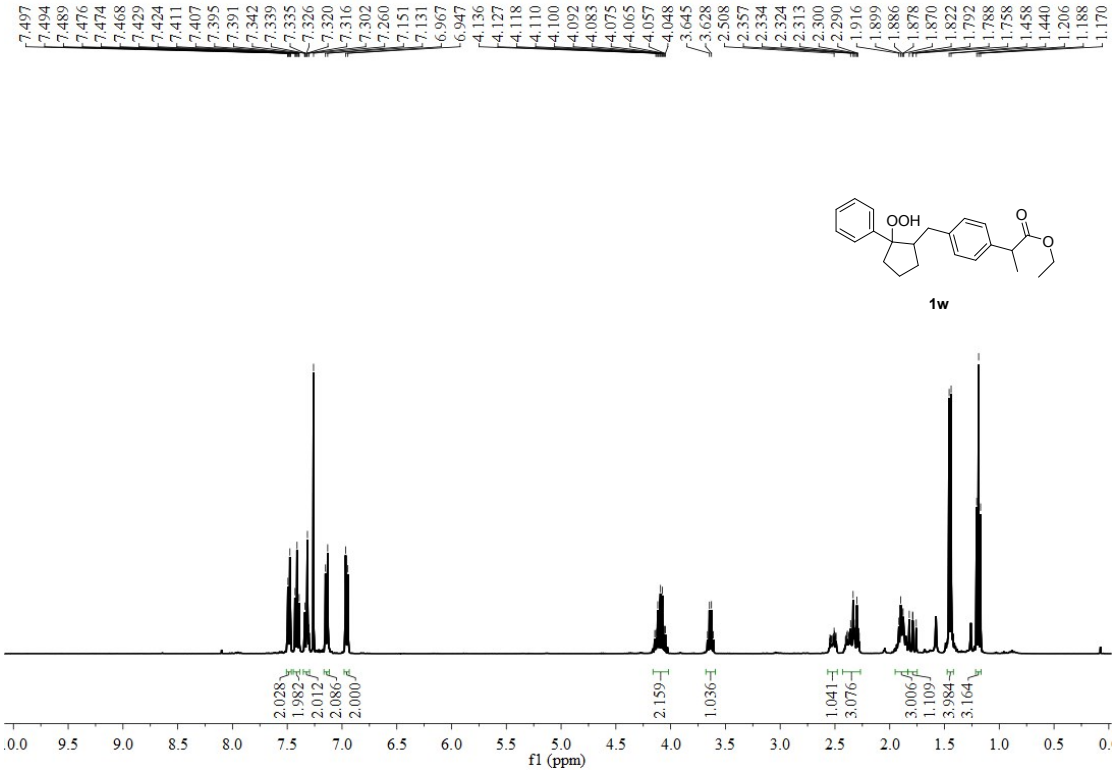


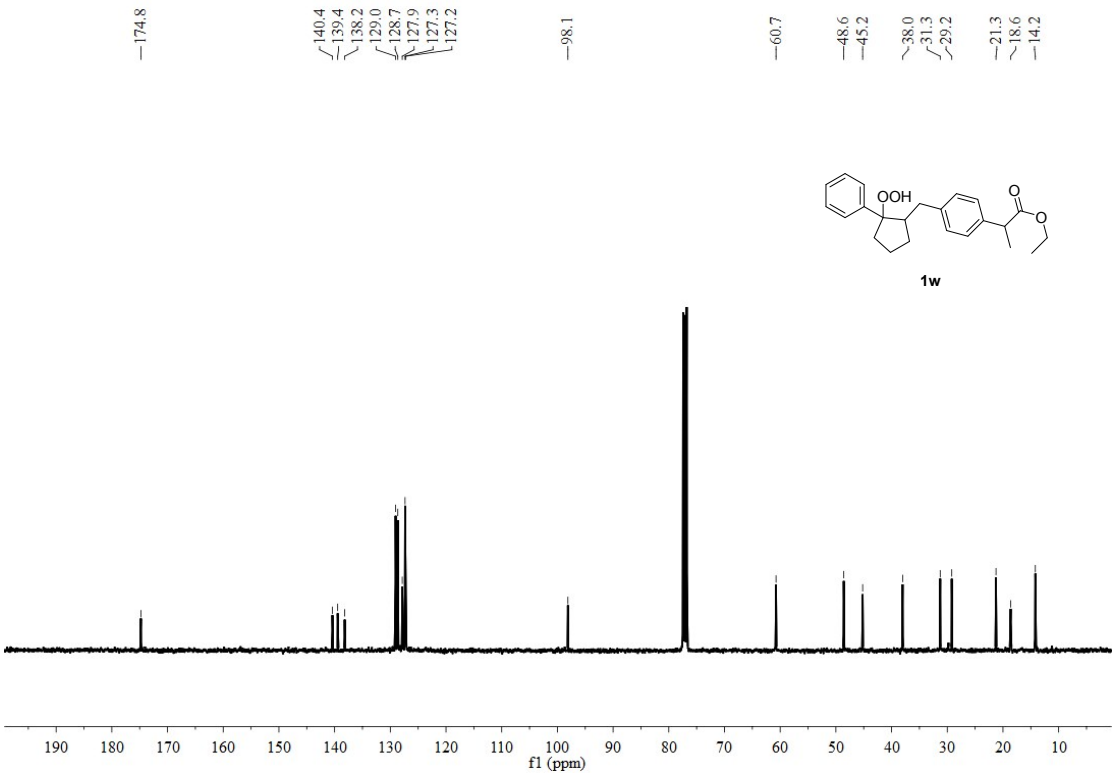
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1v**



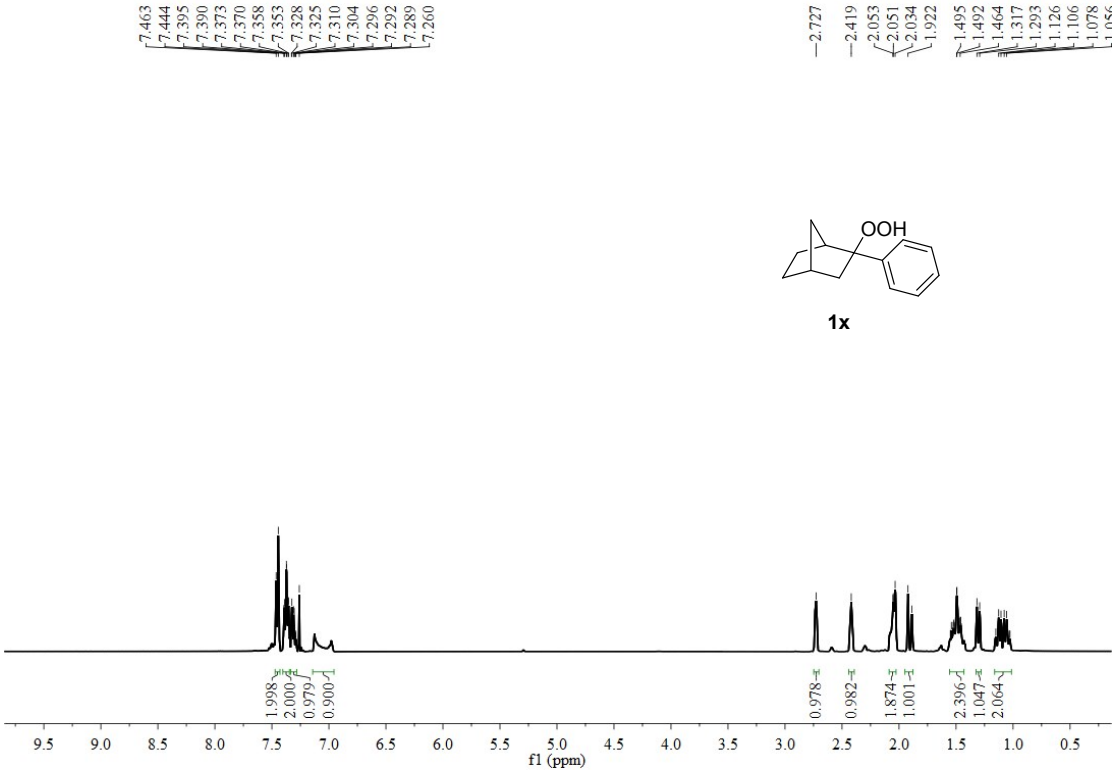


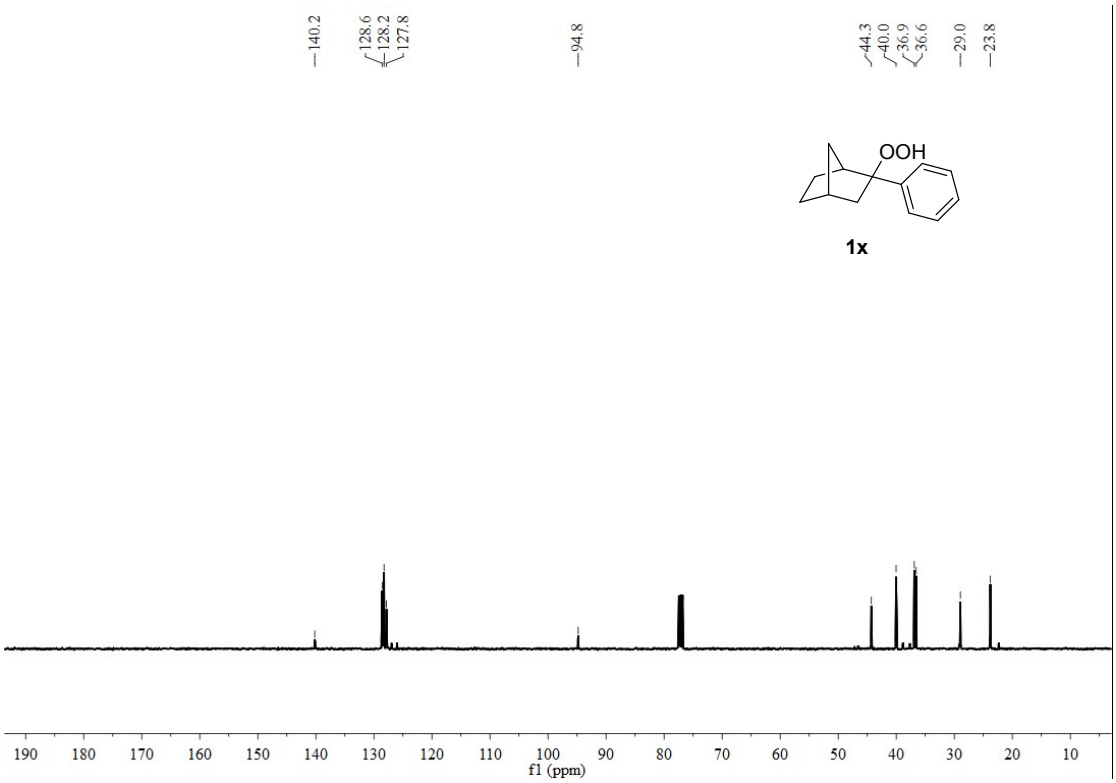
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1w**





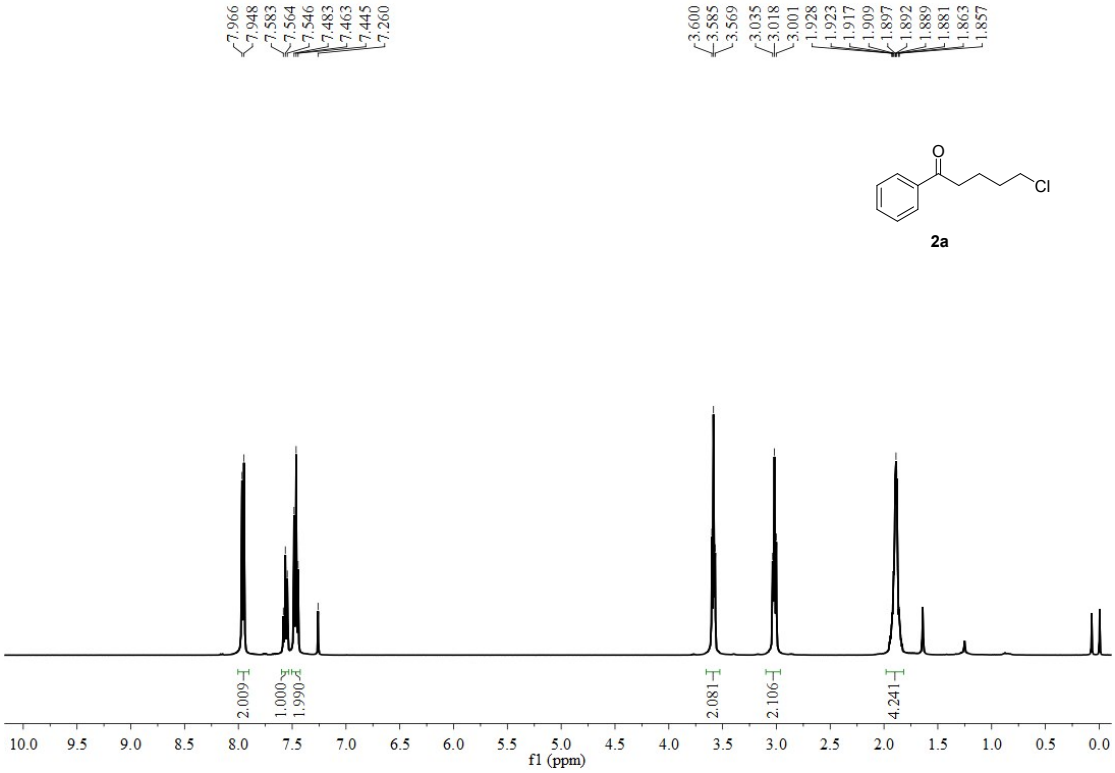
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of **1x**

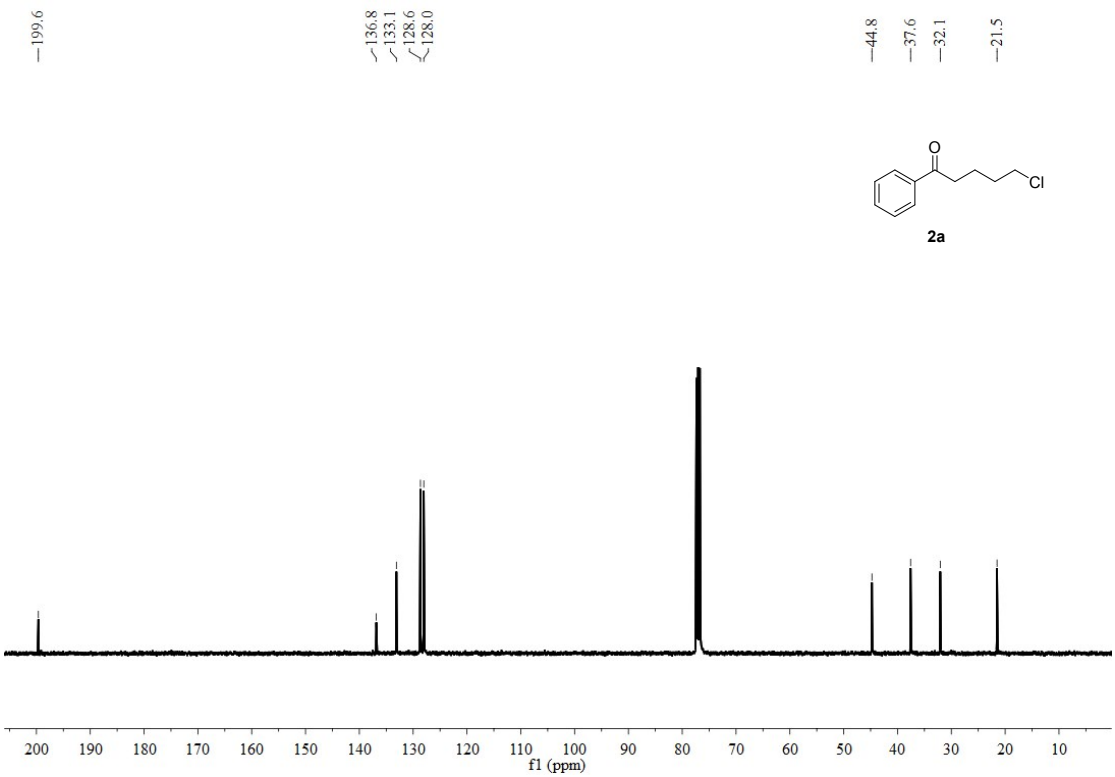




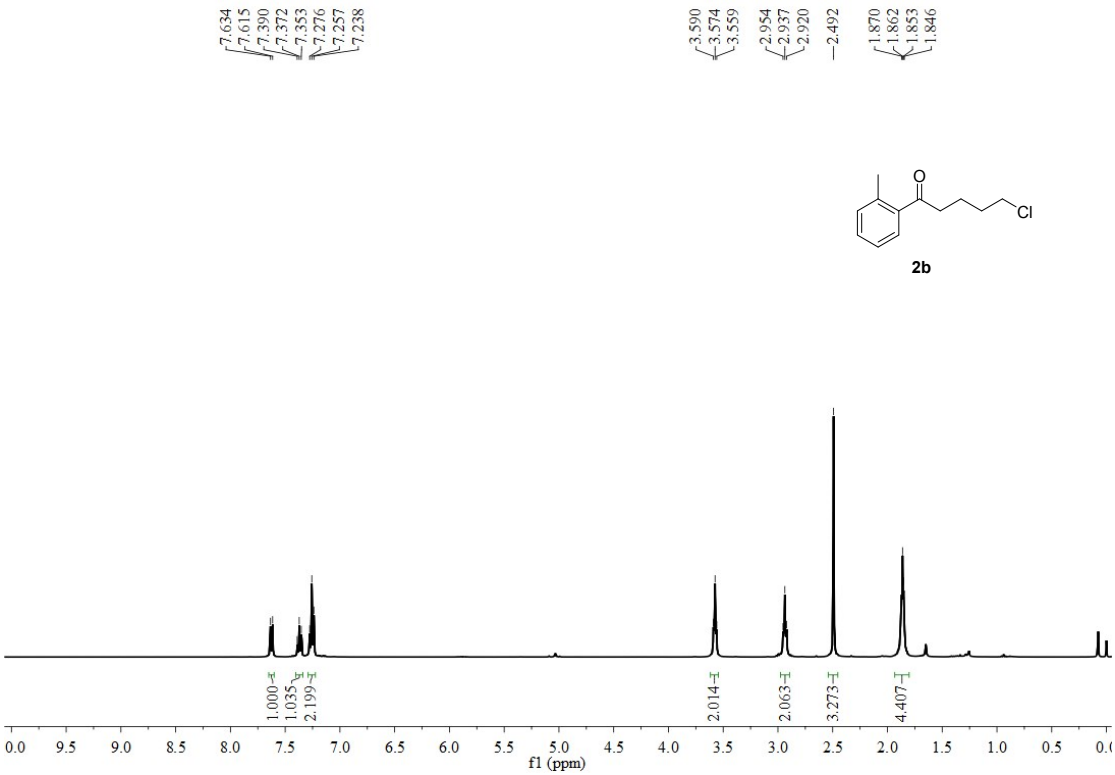
14. ^{1}H NMR and ^{13}C NMR Spectra of Products 2

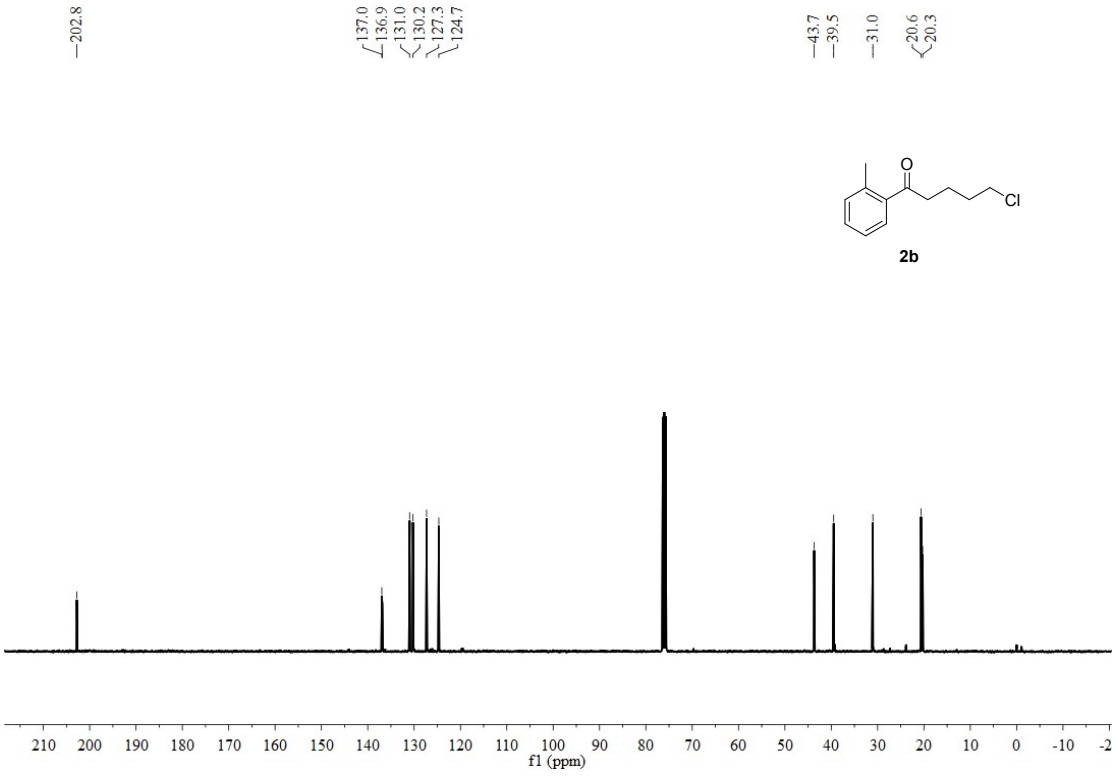
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2a**



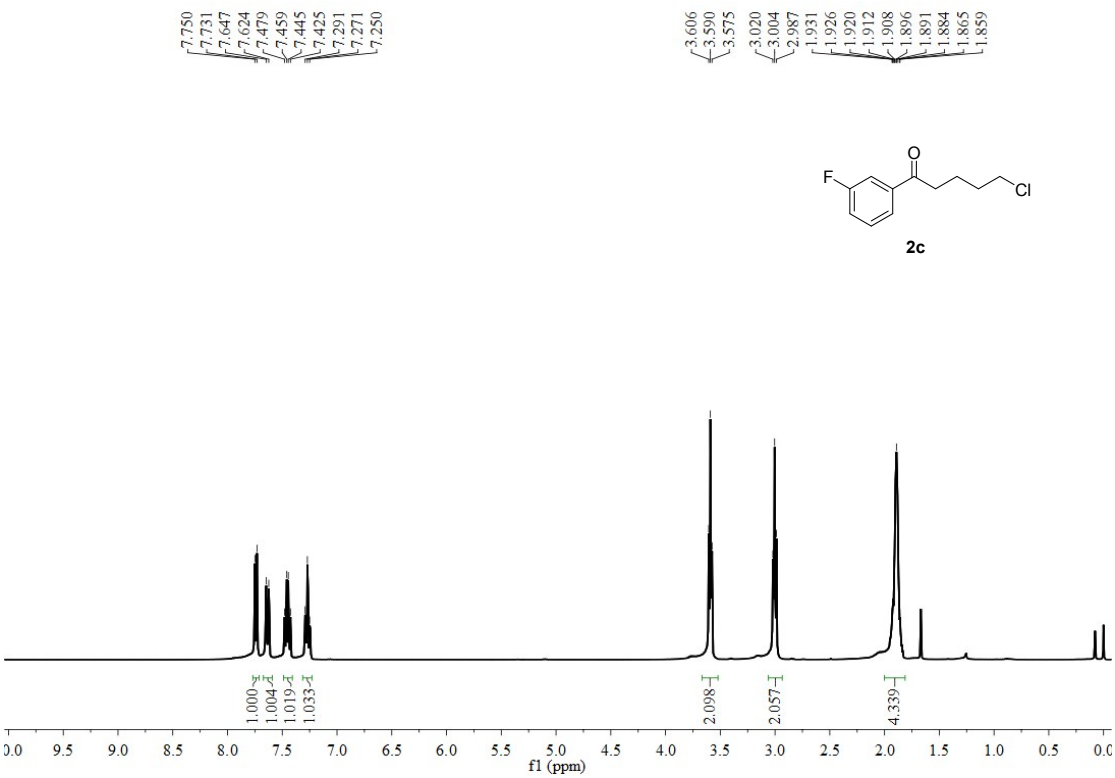


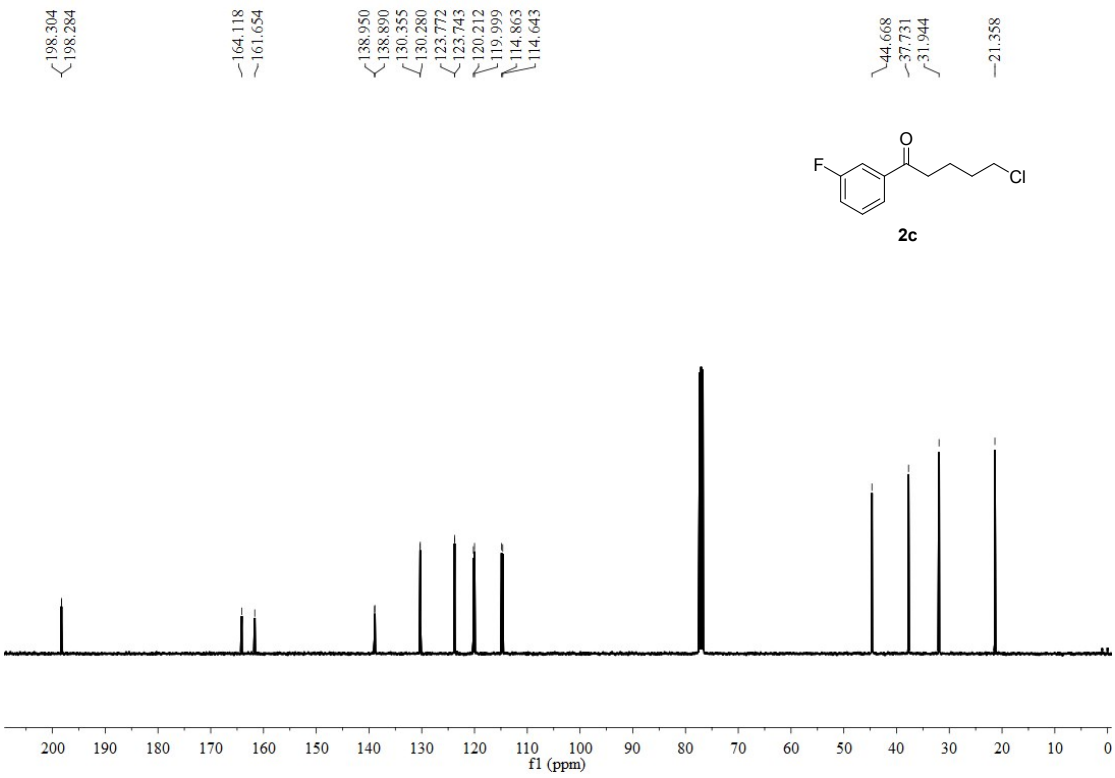
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2b**



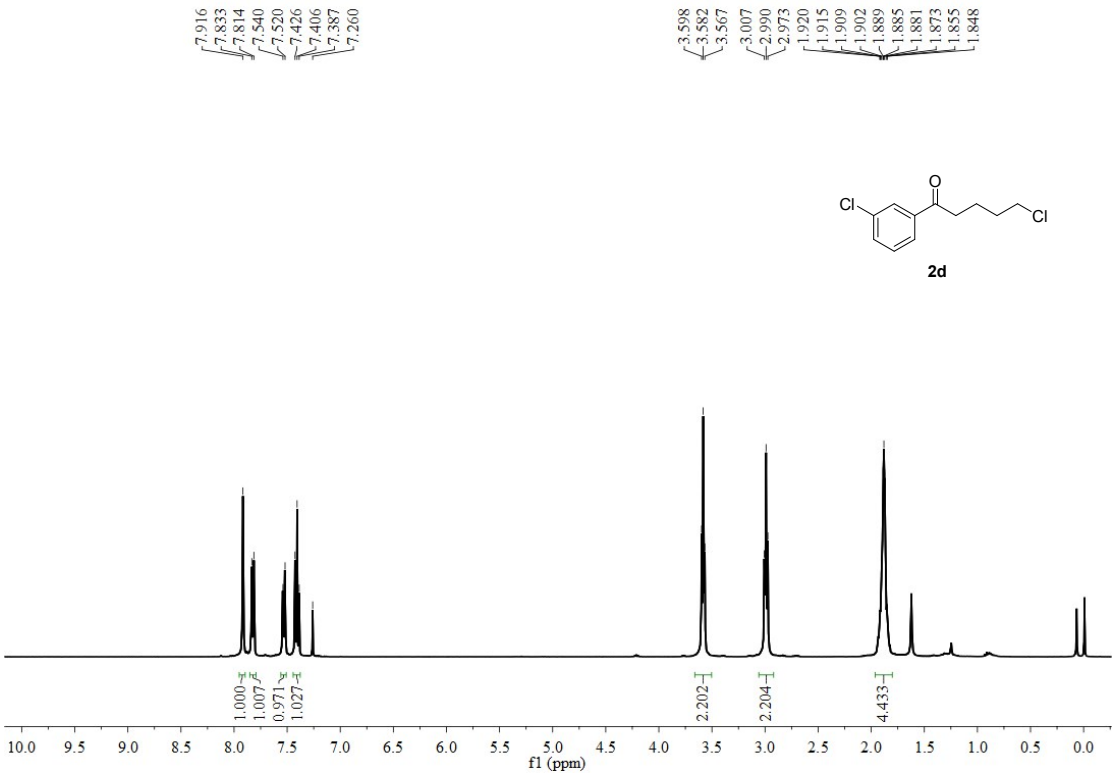


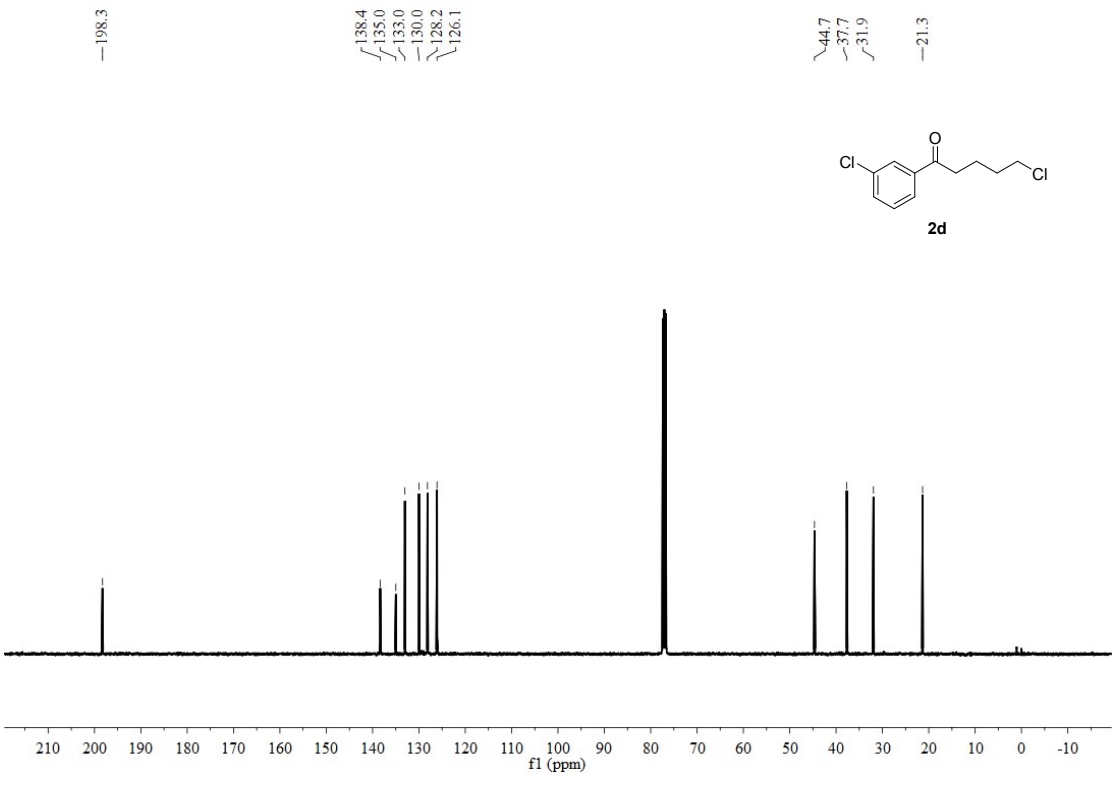
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2c**



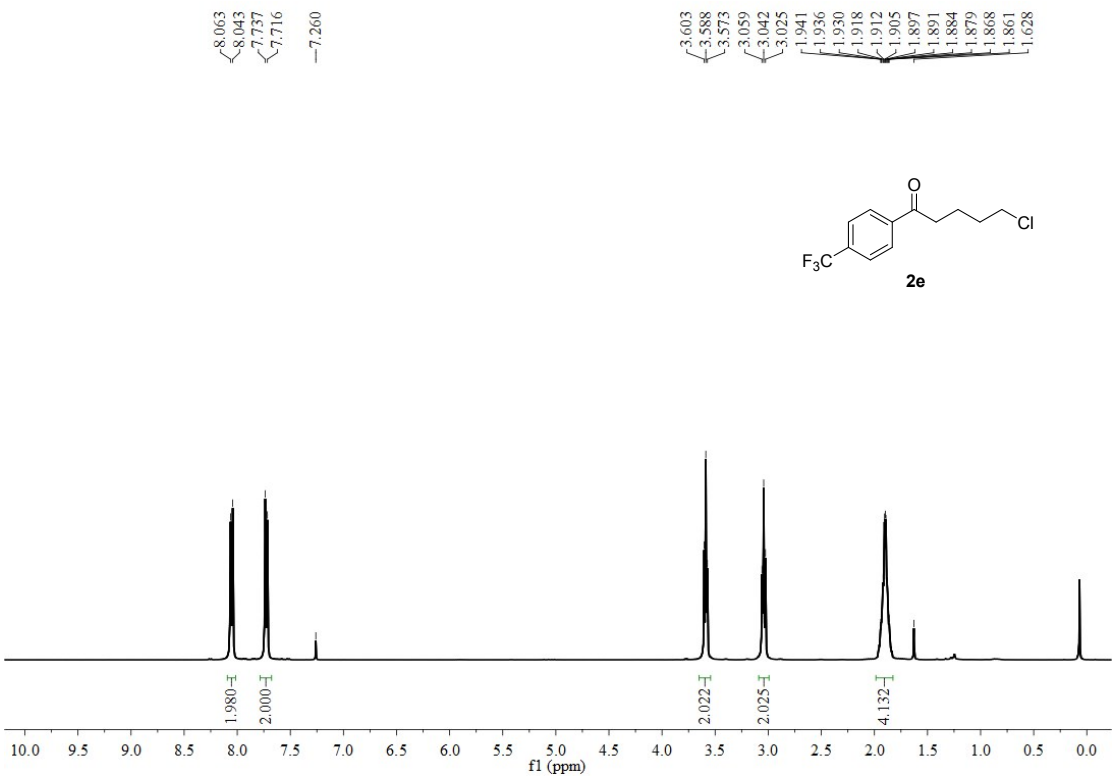


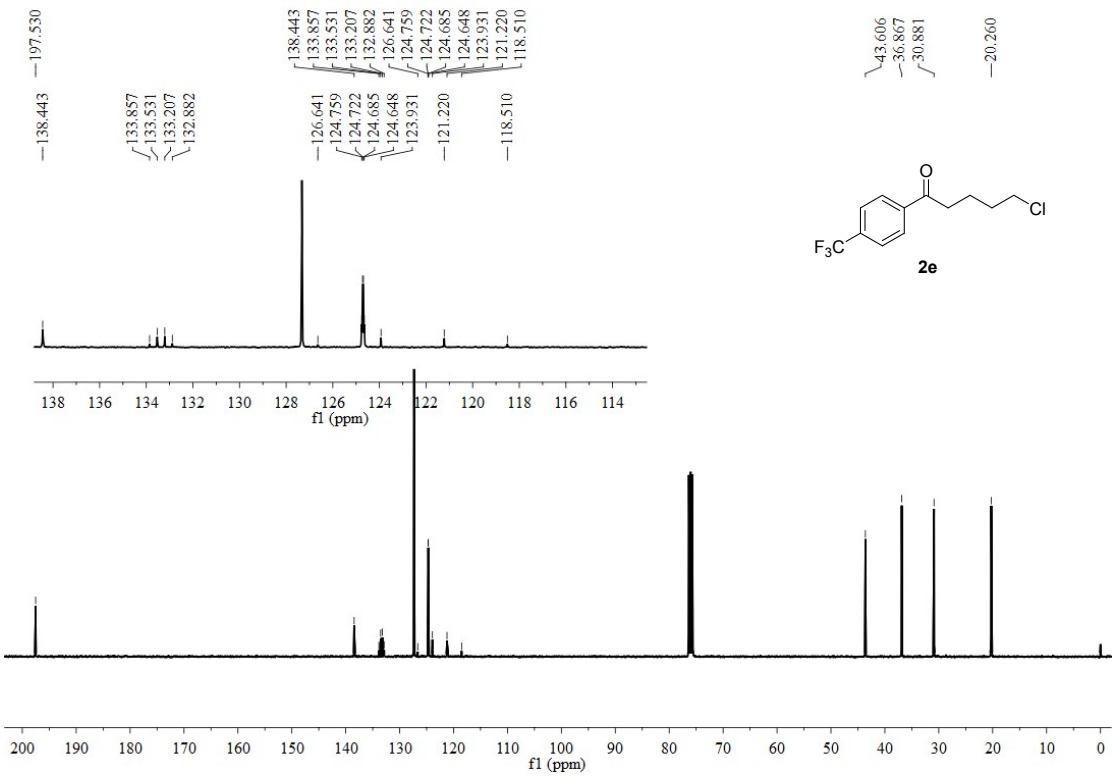
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2d**



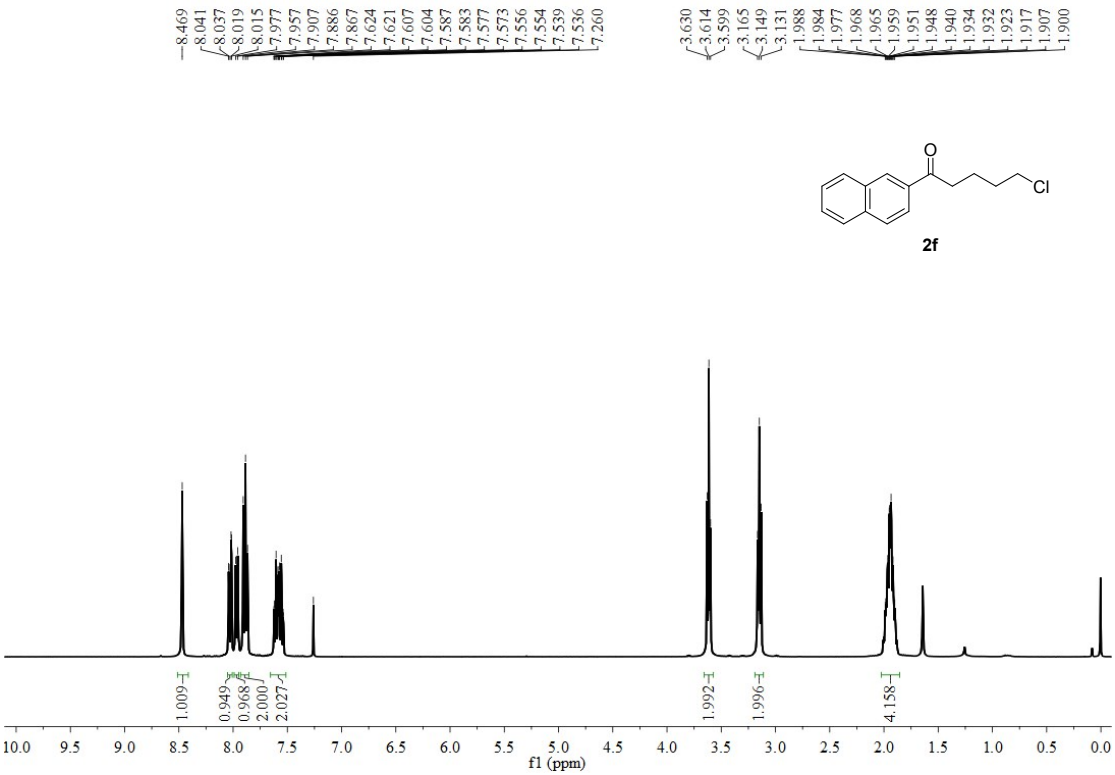


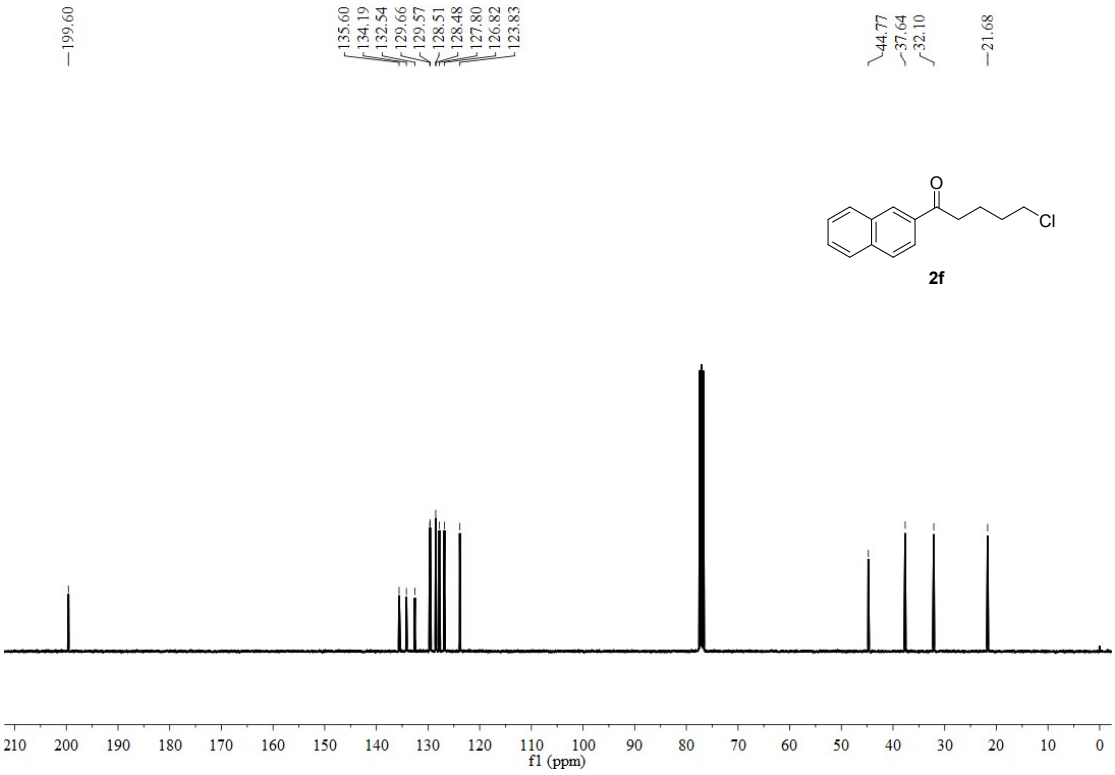
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2e**



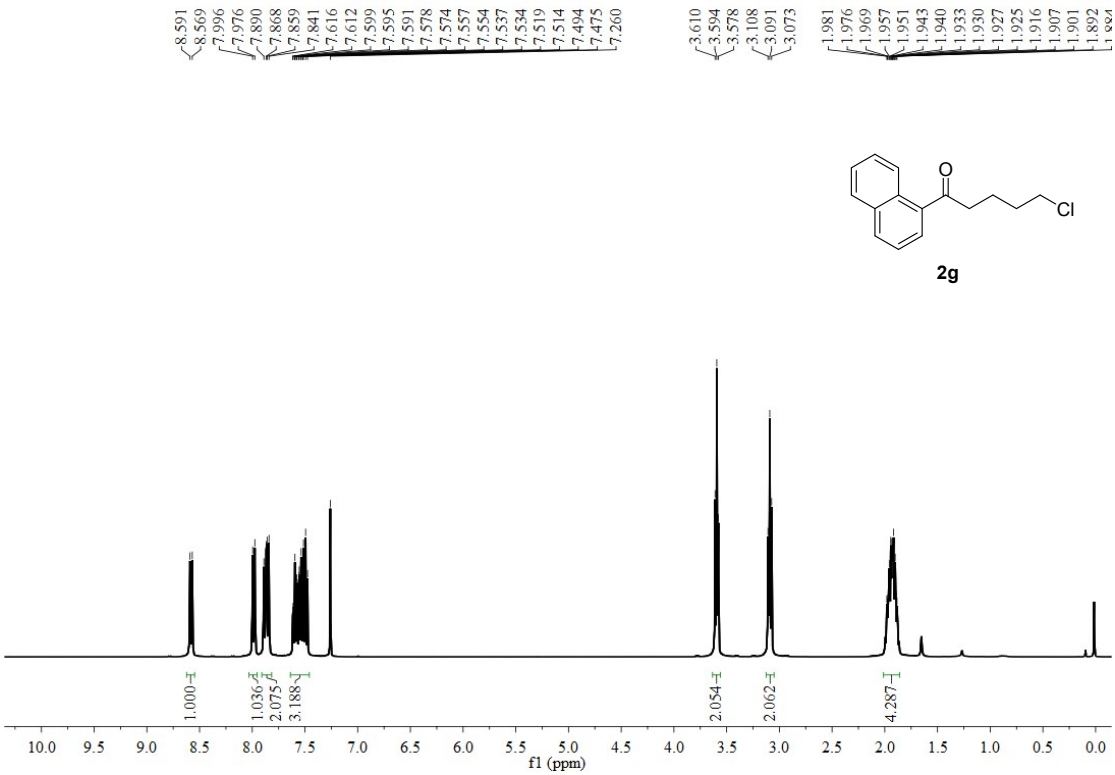


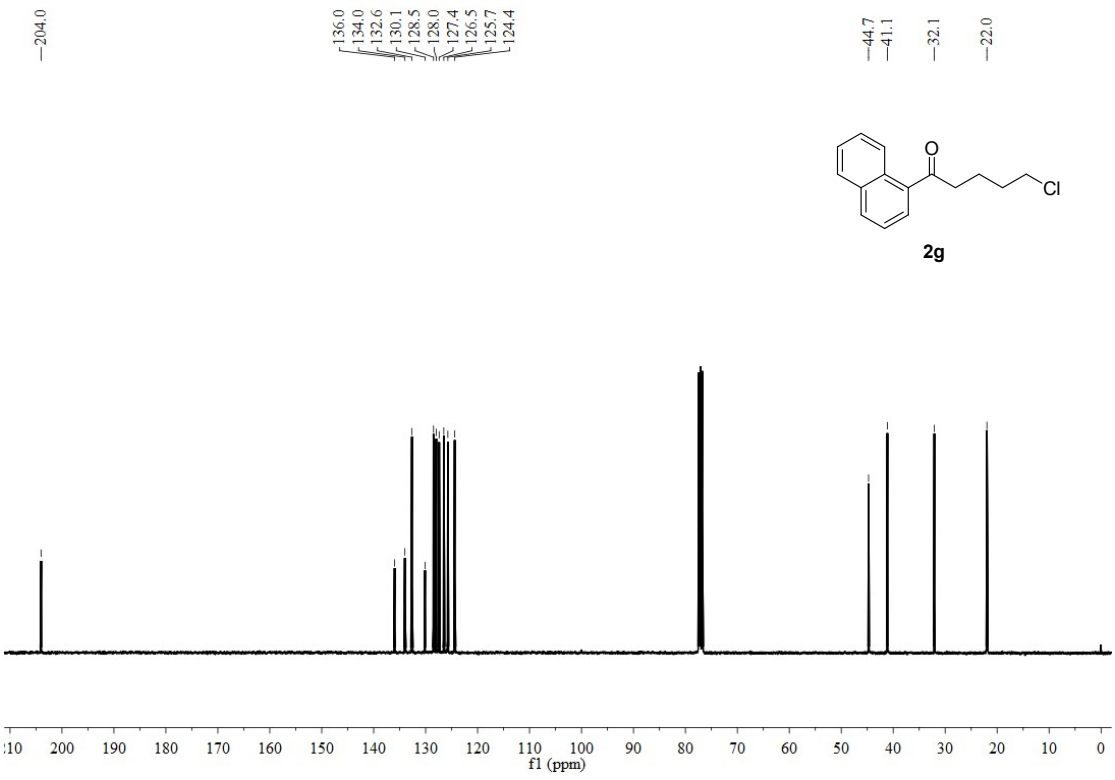
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2f**



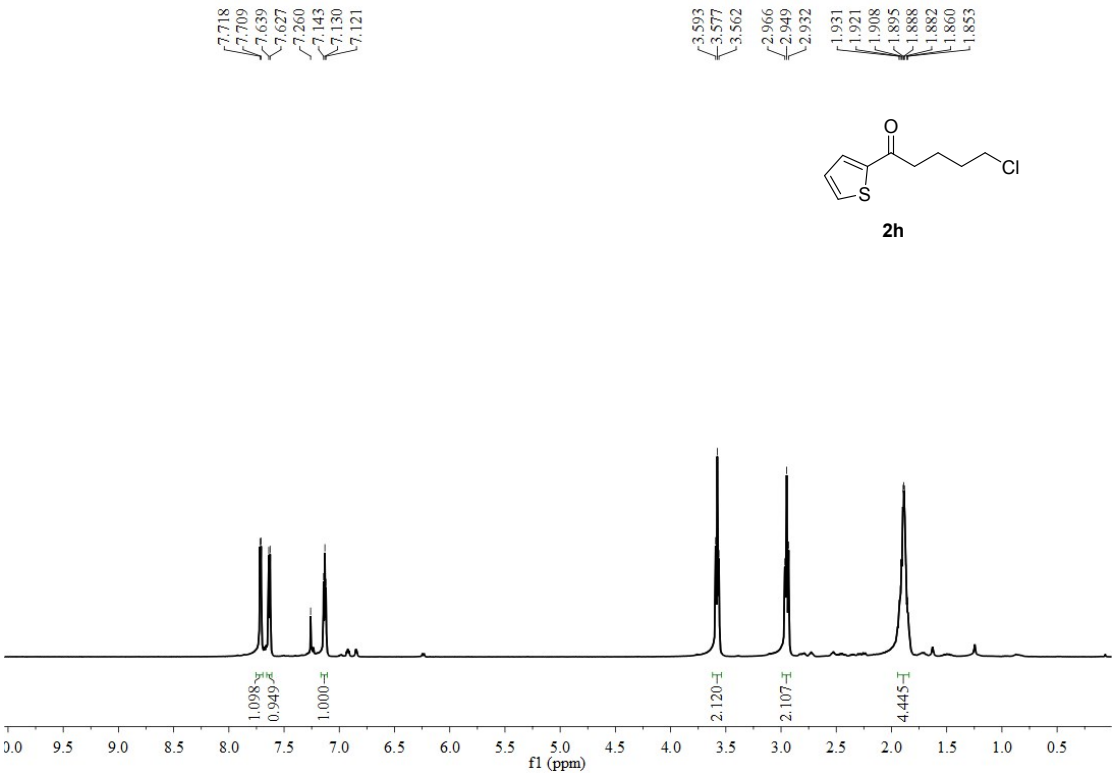


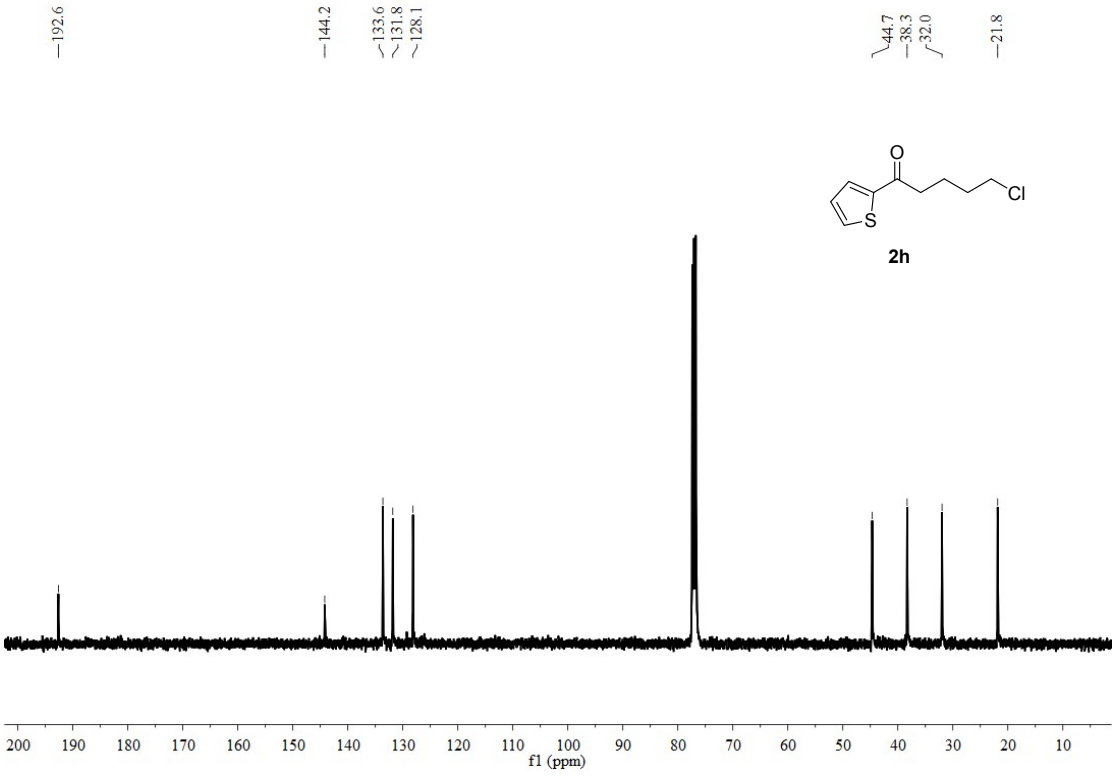
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2g**



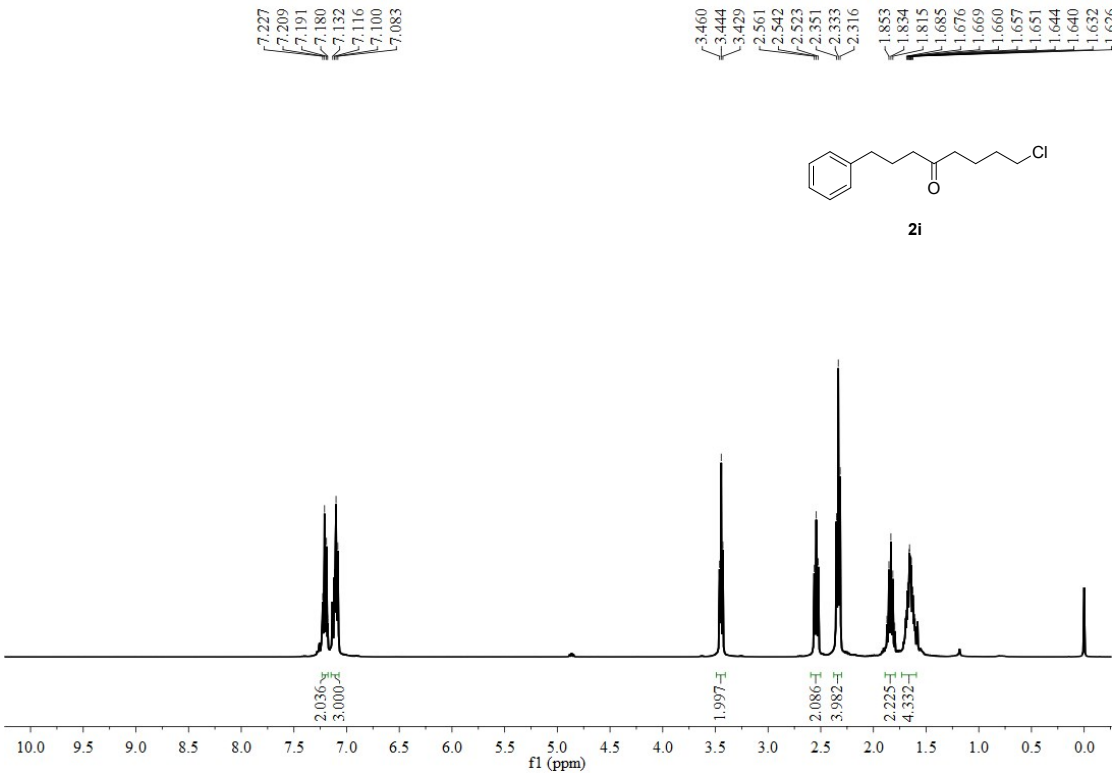


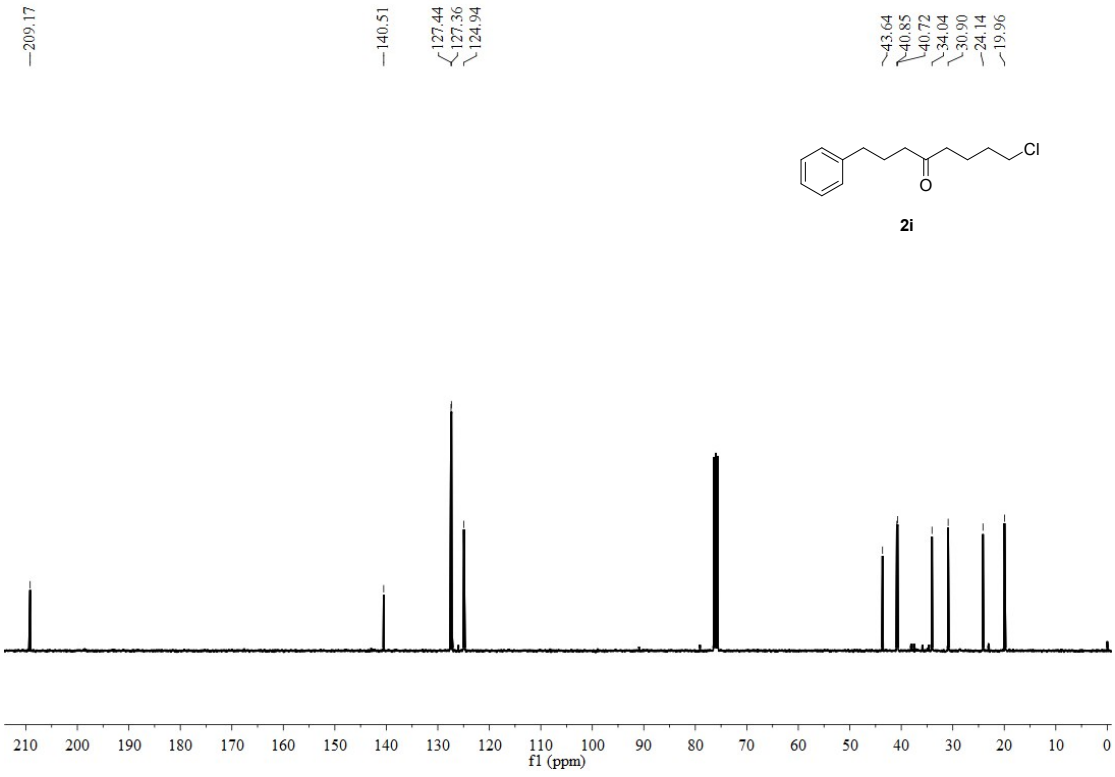
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2h**



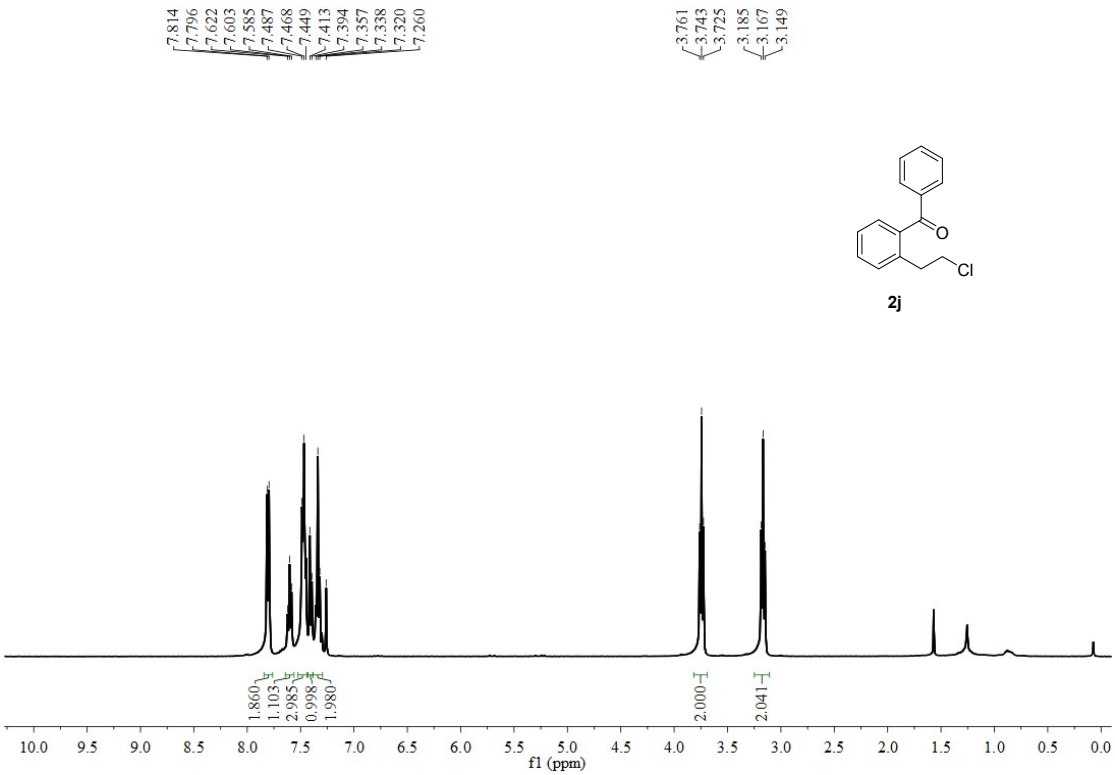


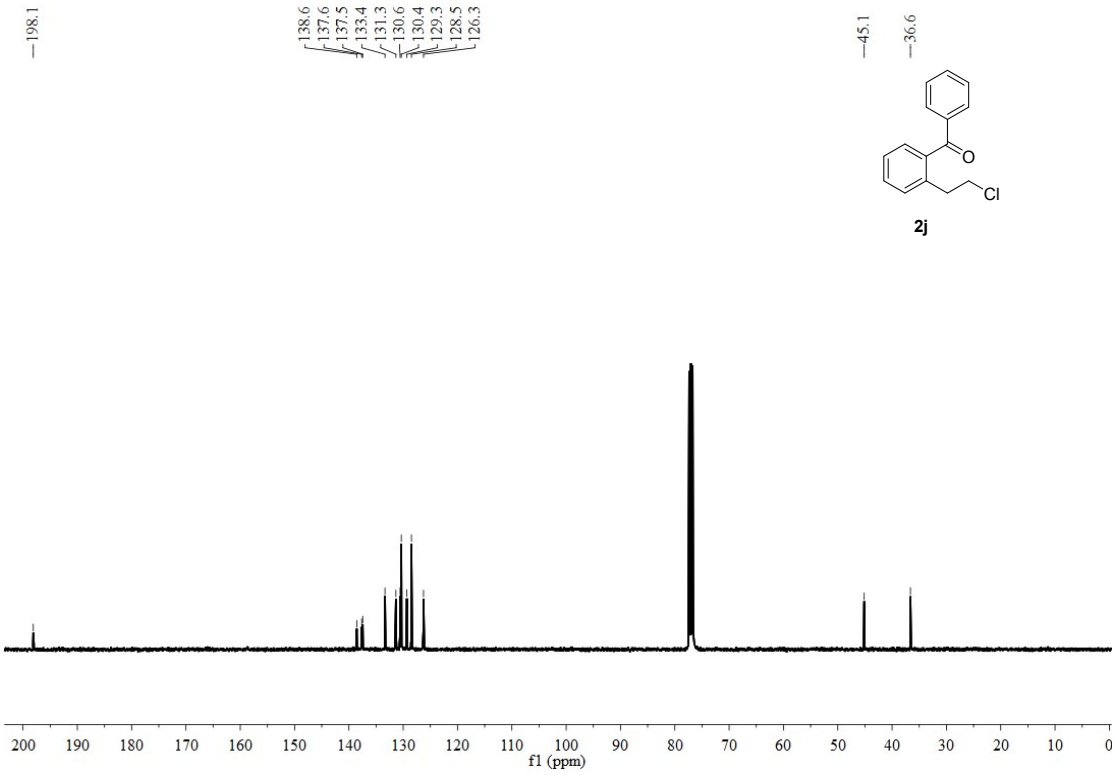
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2i**



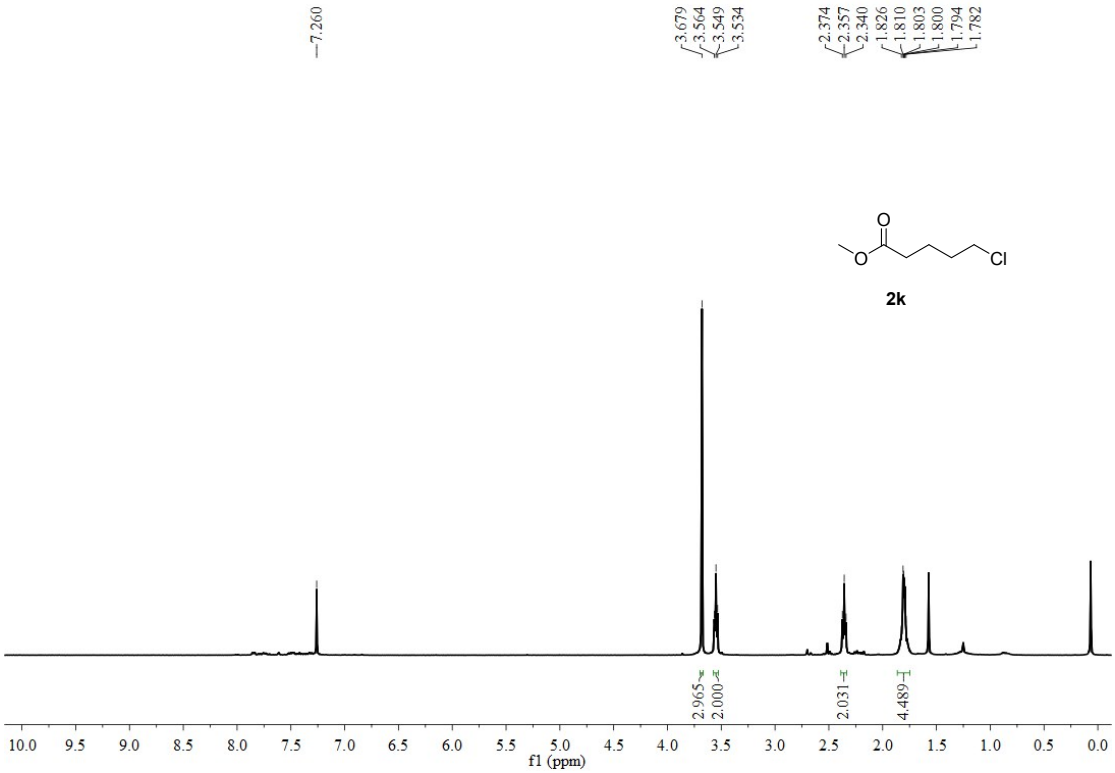


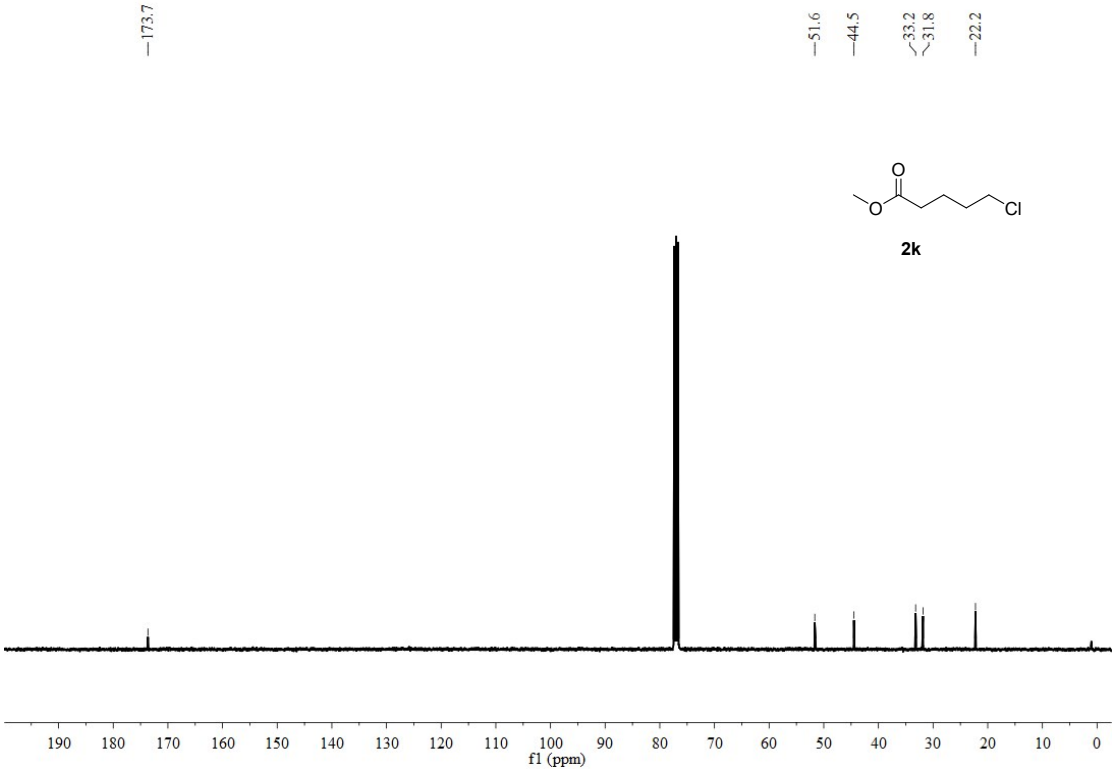
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2j**



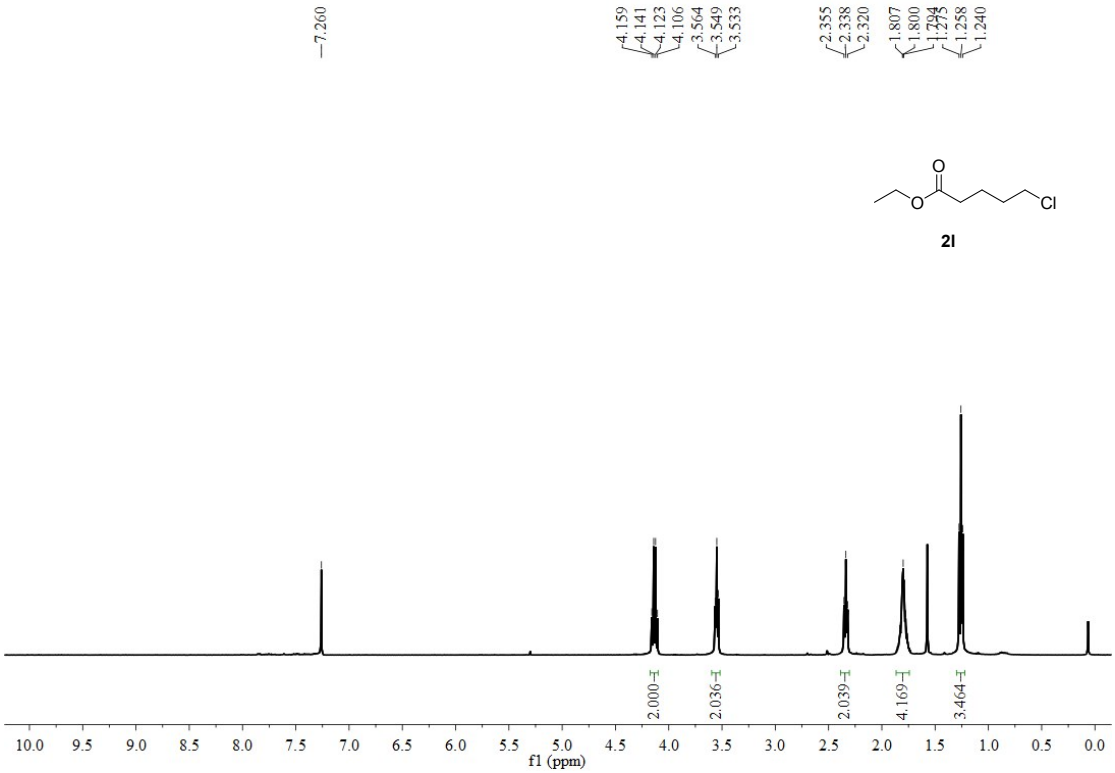


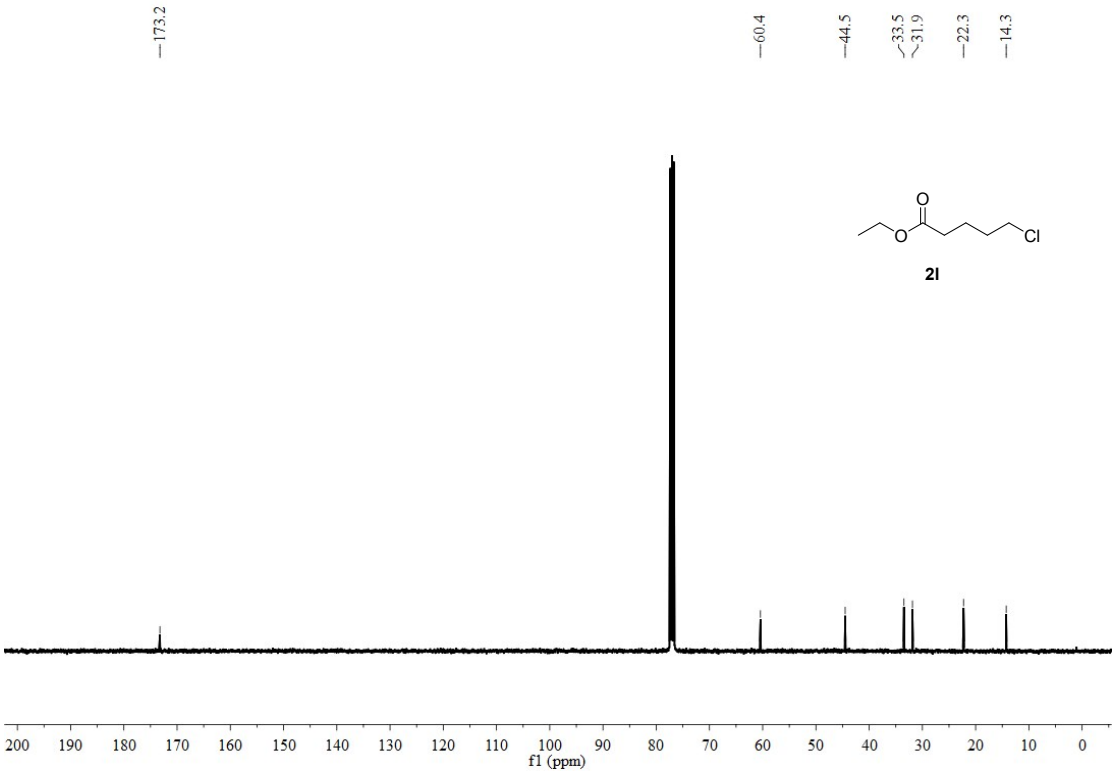
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2k**



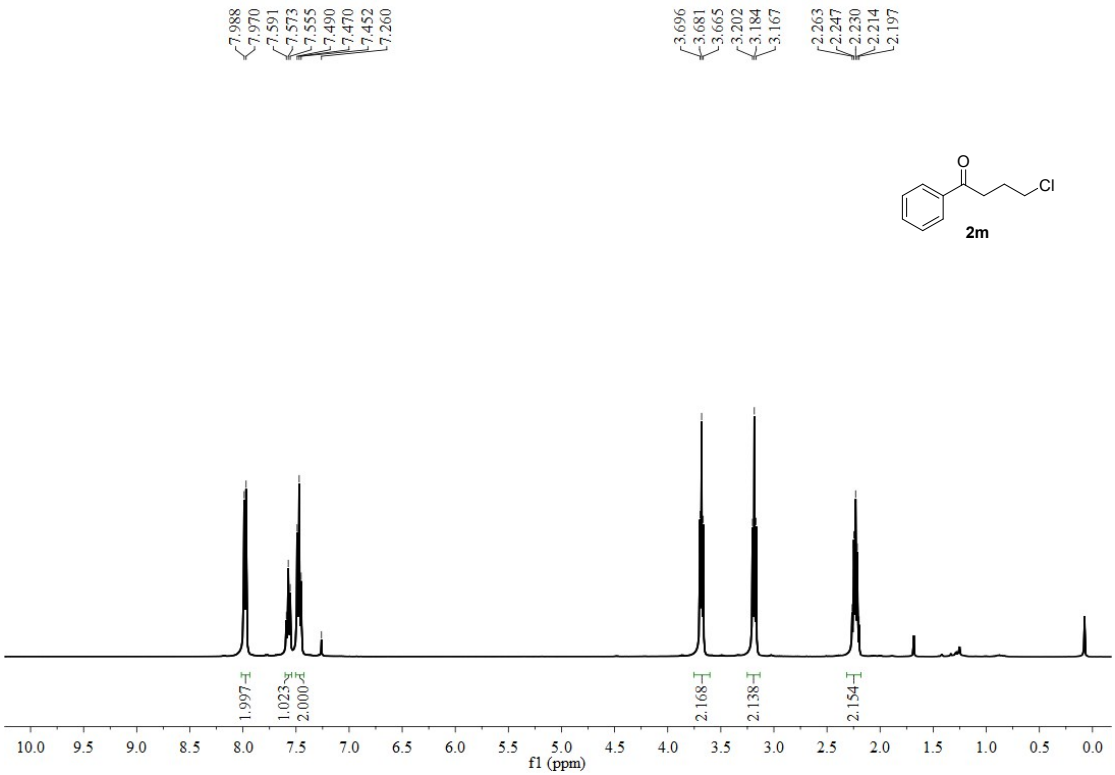


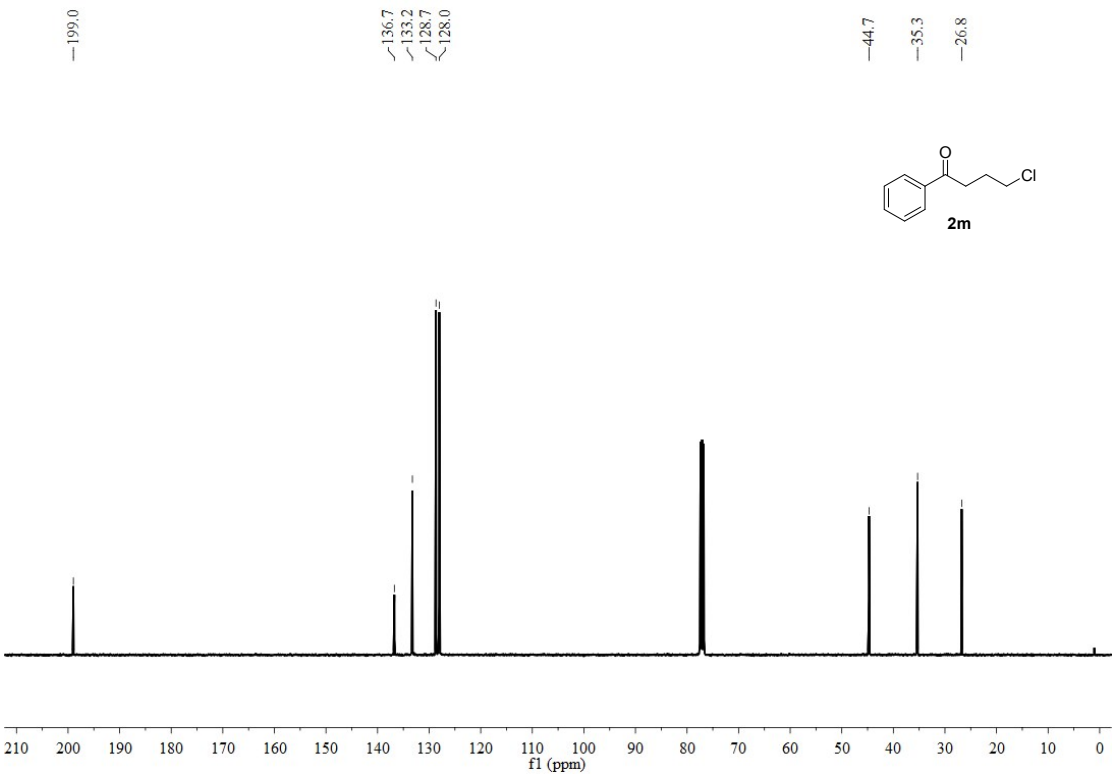
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2l**



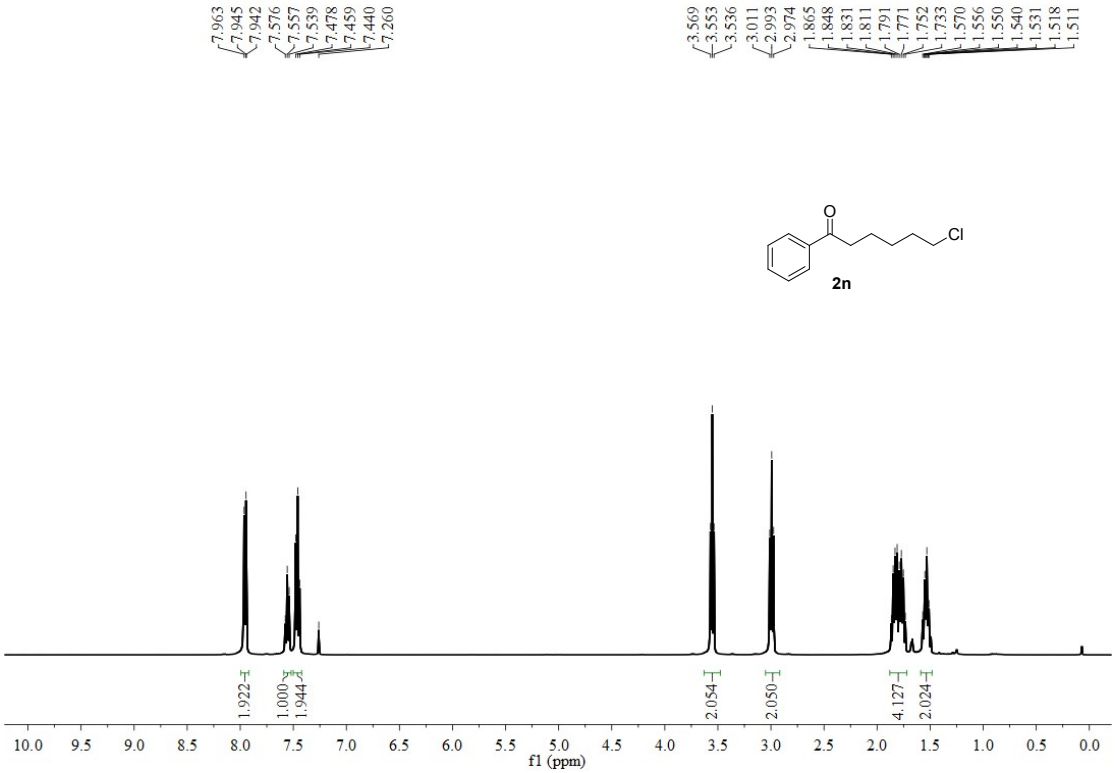


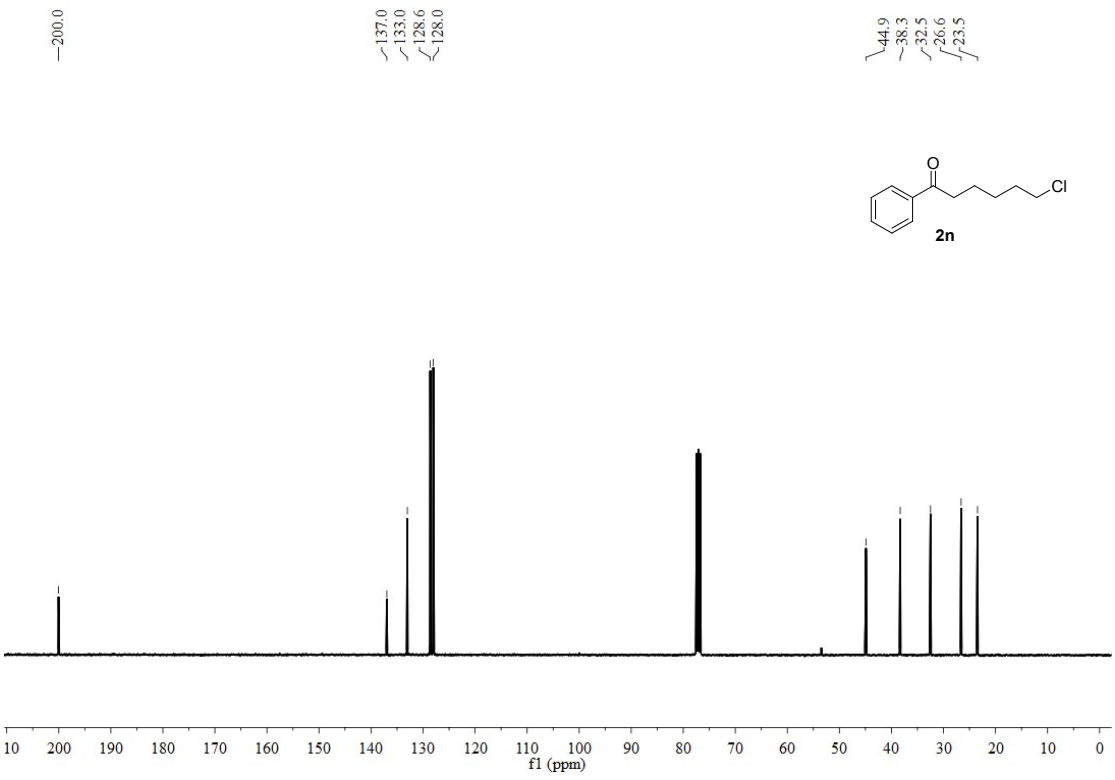
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2m**



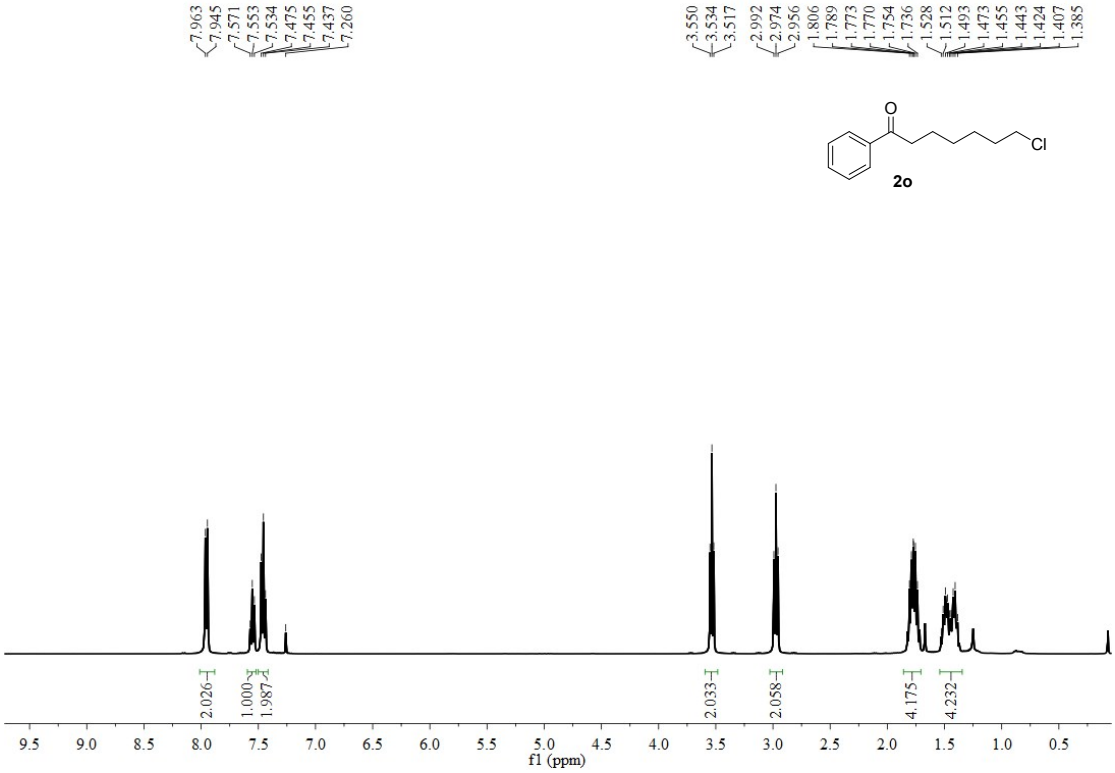


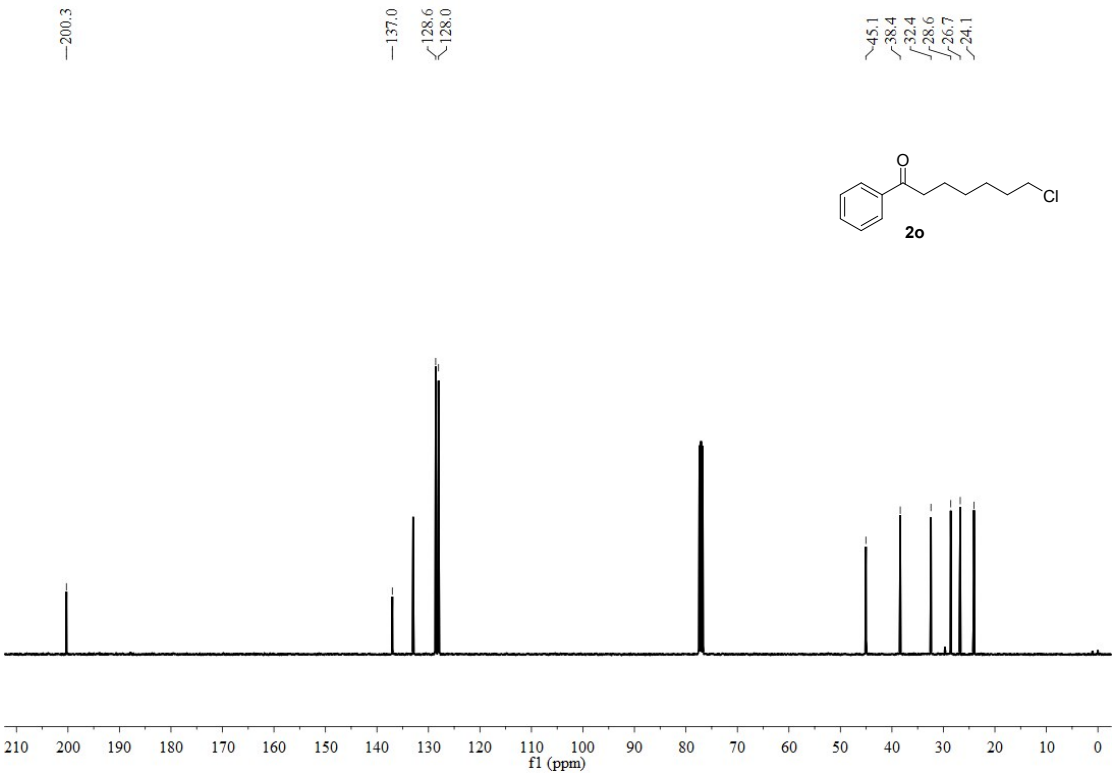
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2n**



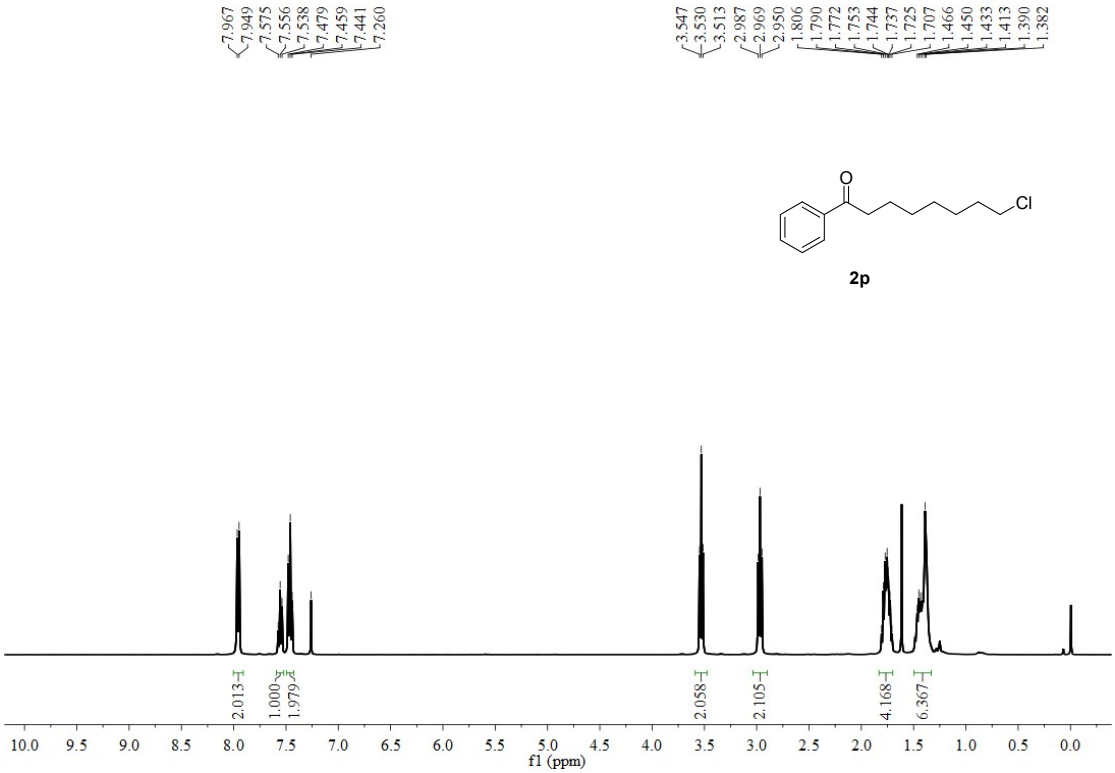


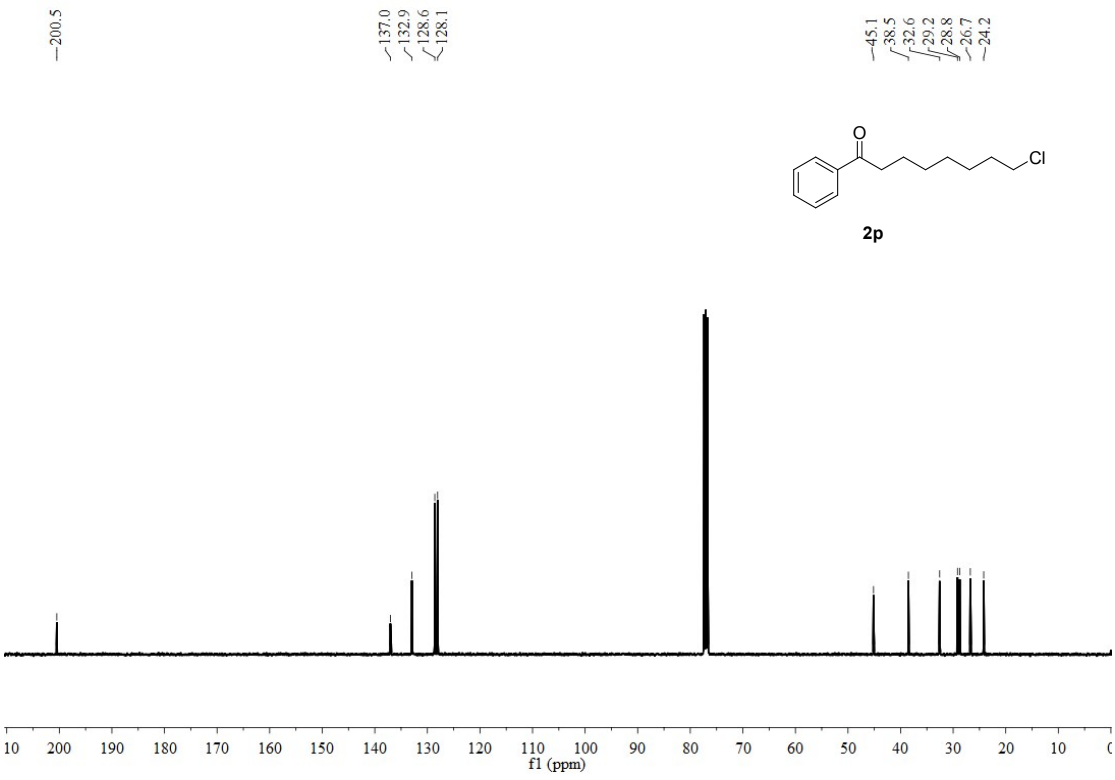
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2o**



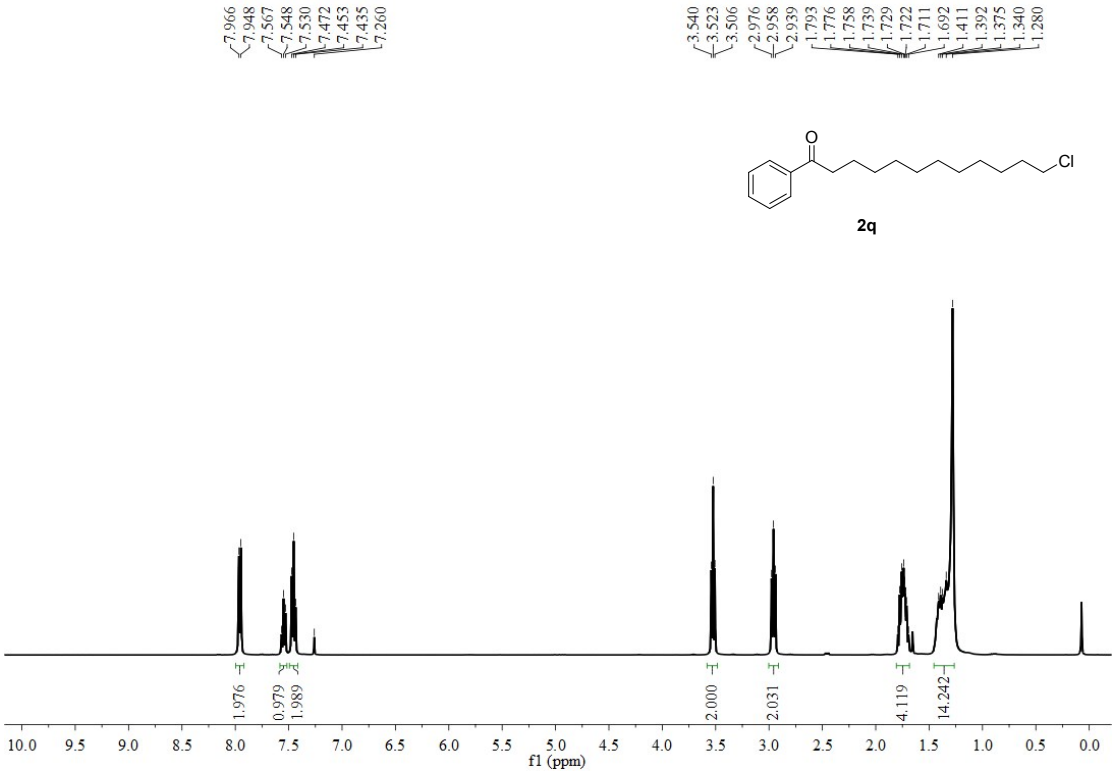


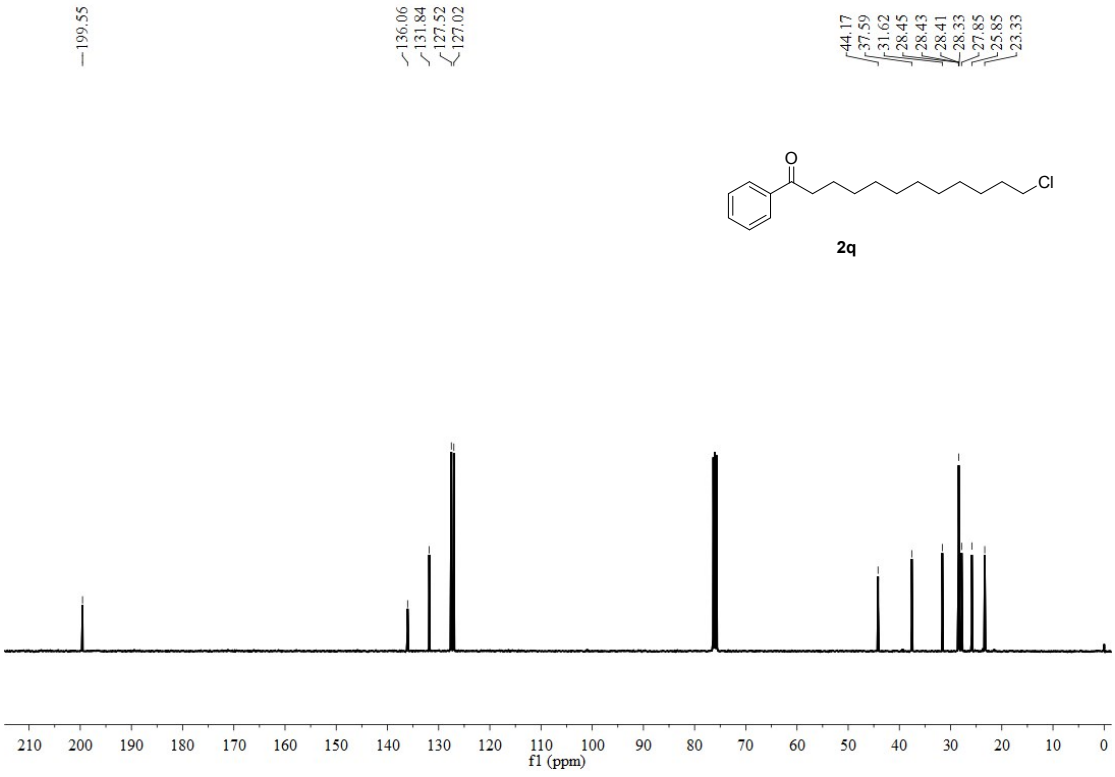
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2p**



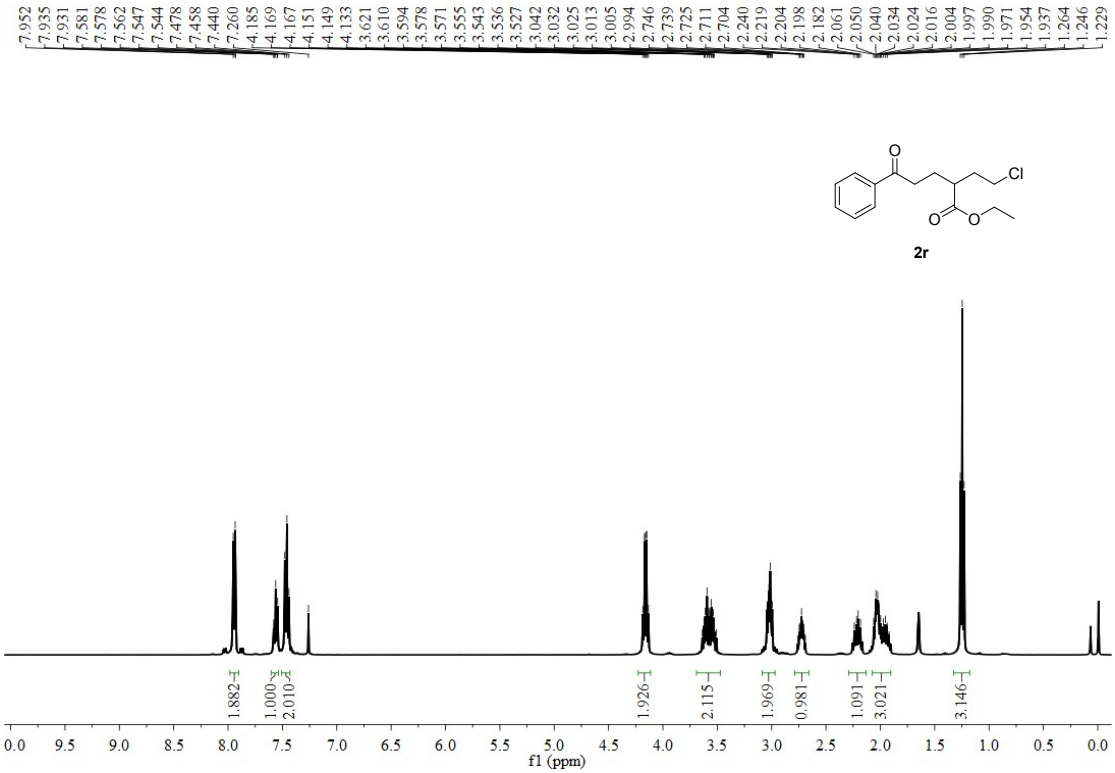


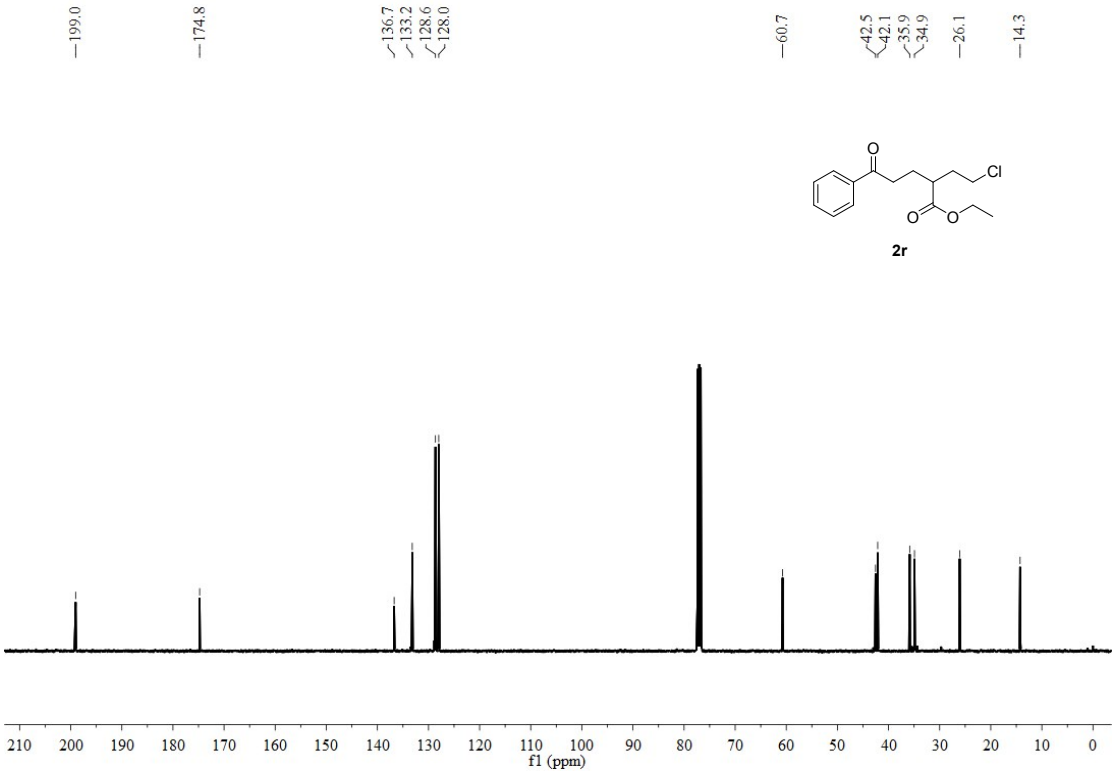
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2q**



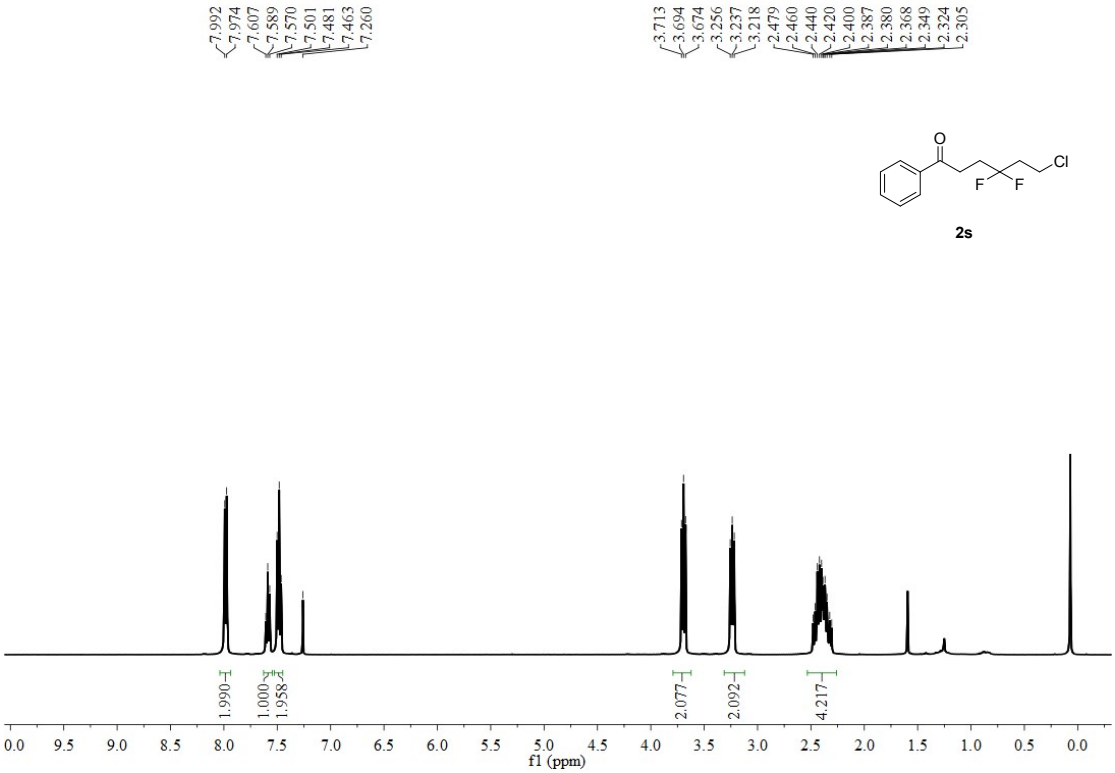


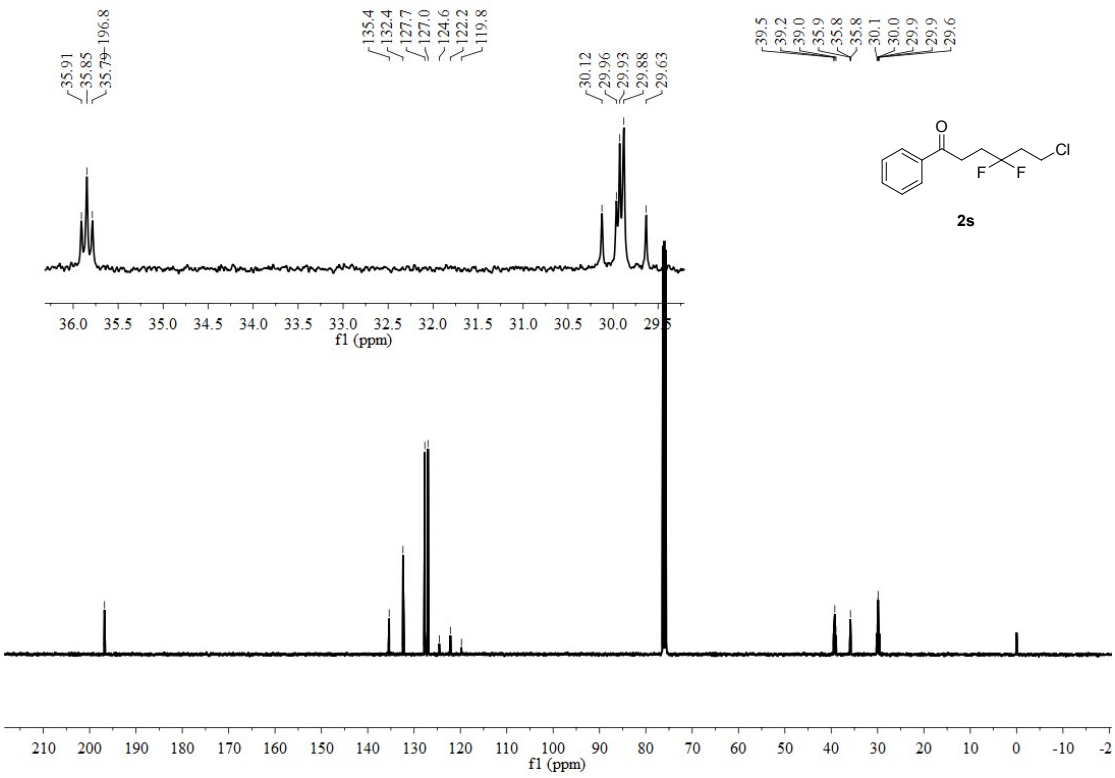
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2r**



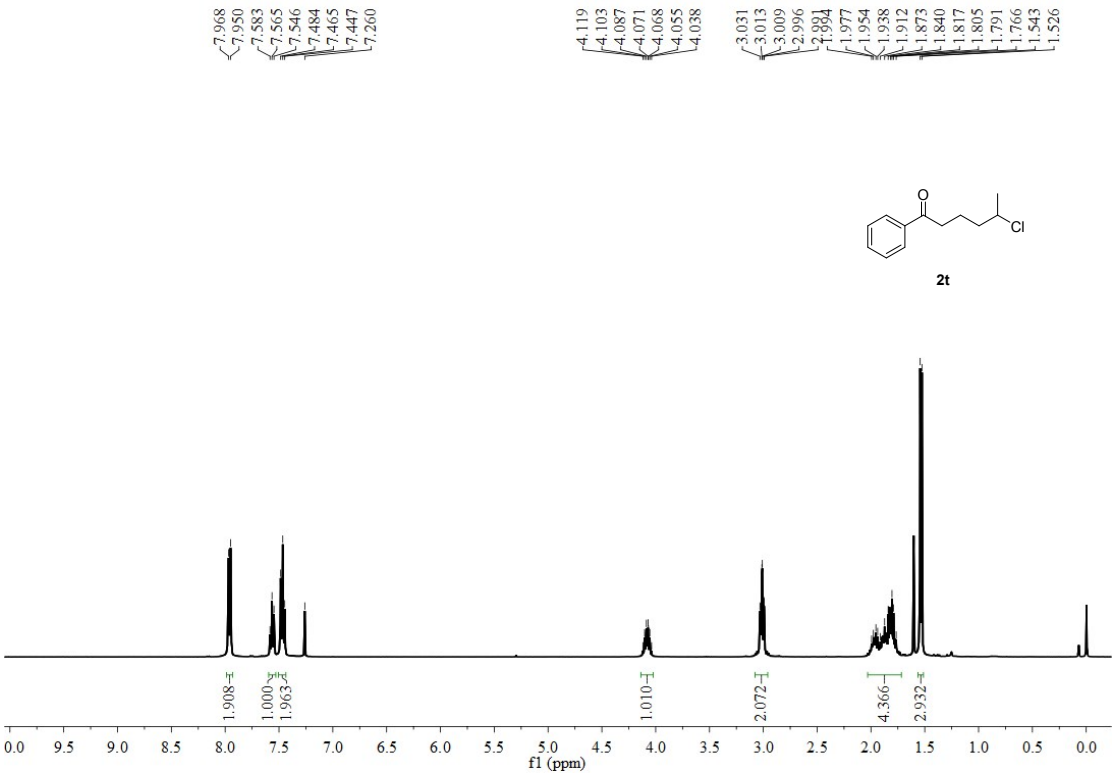


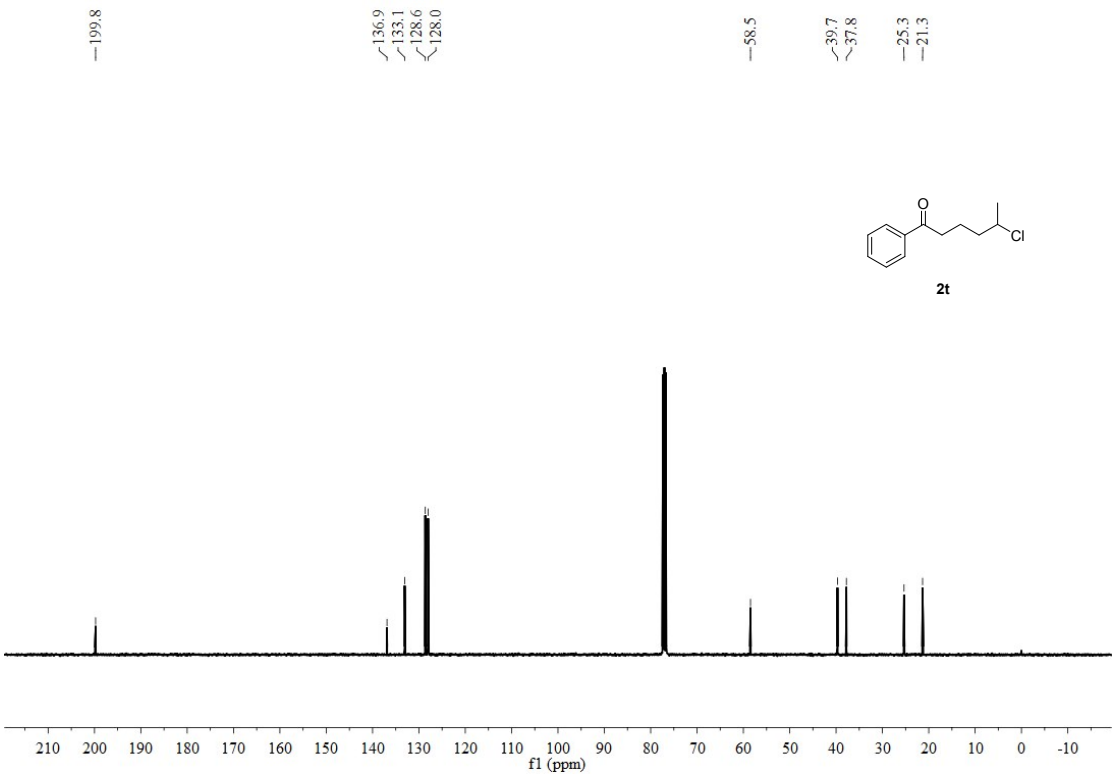
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2s**



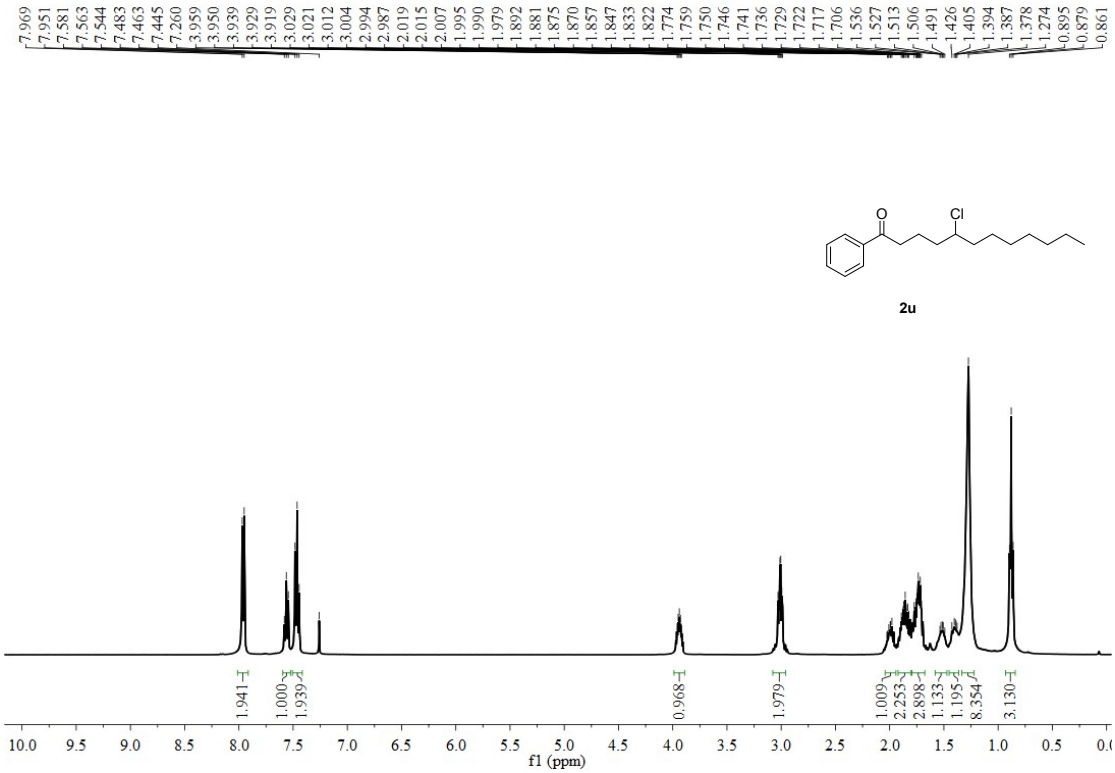


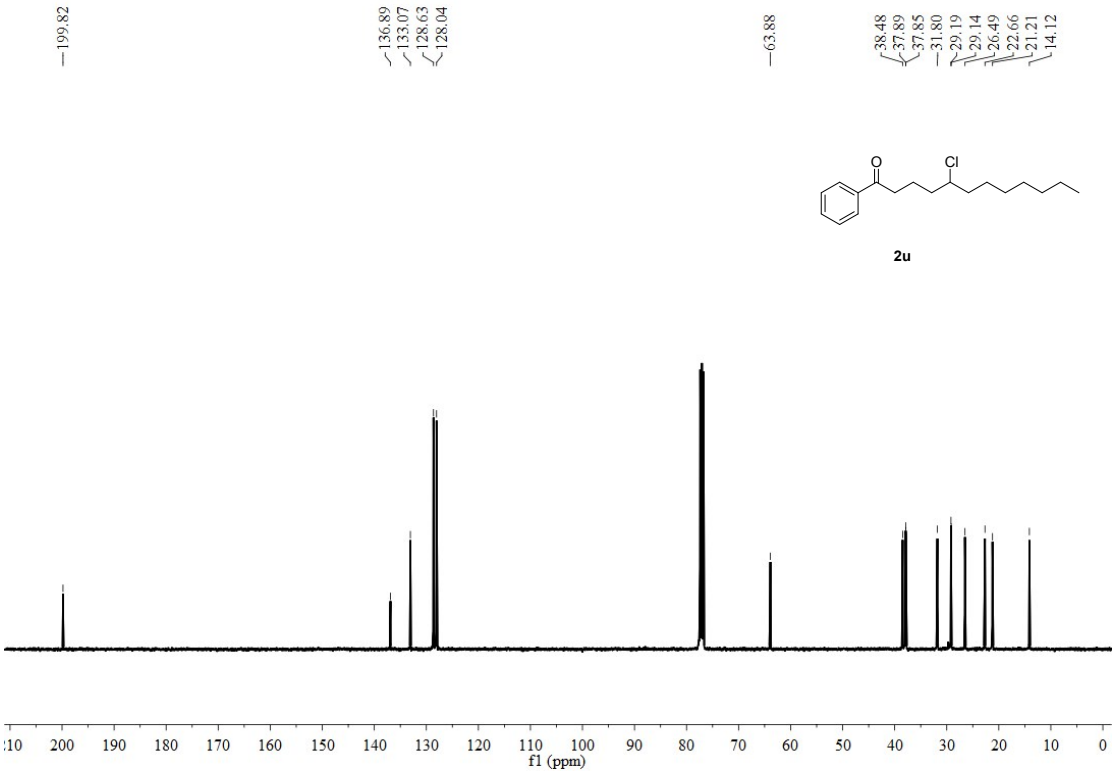
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2t**



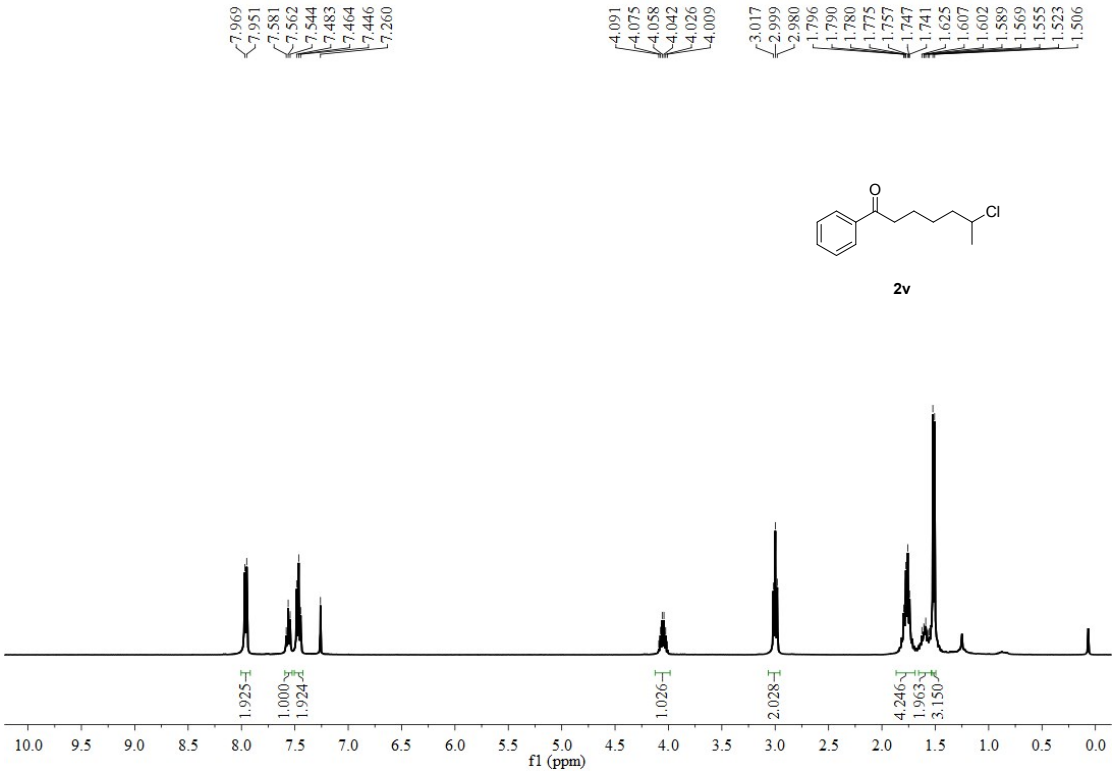


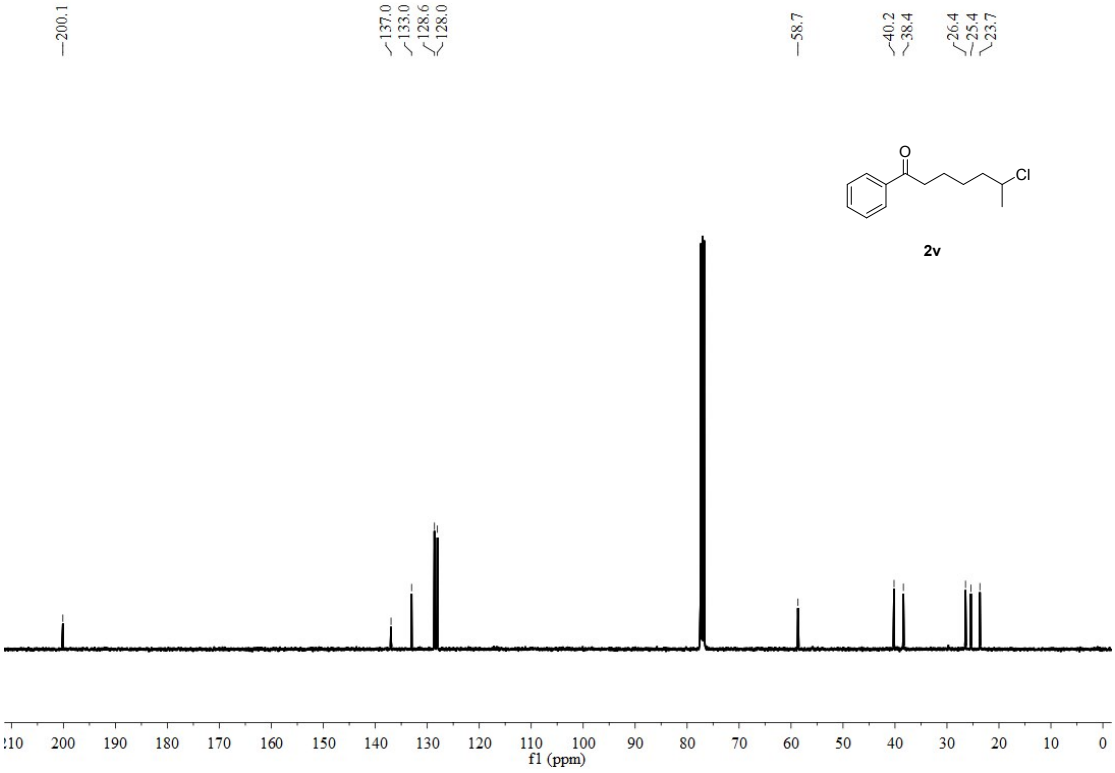
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2u**



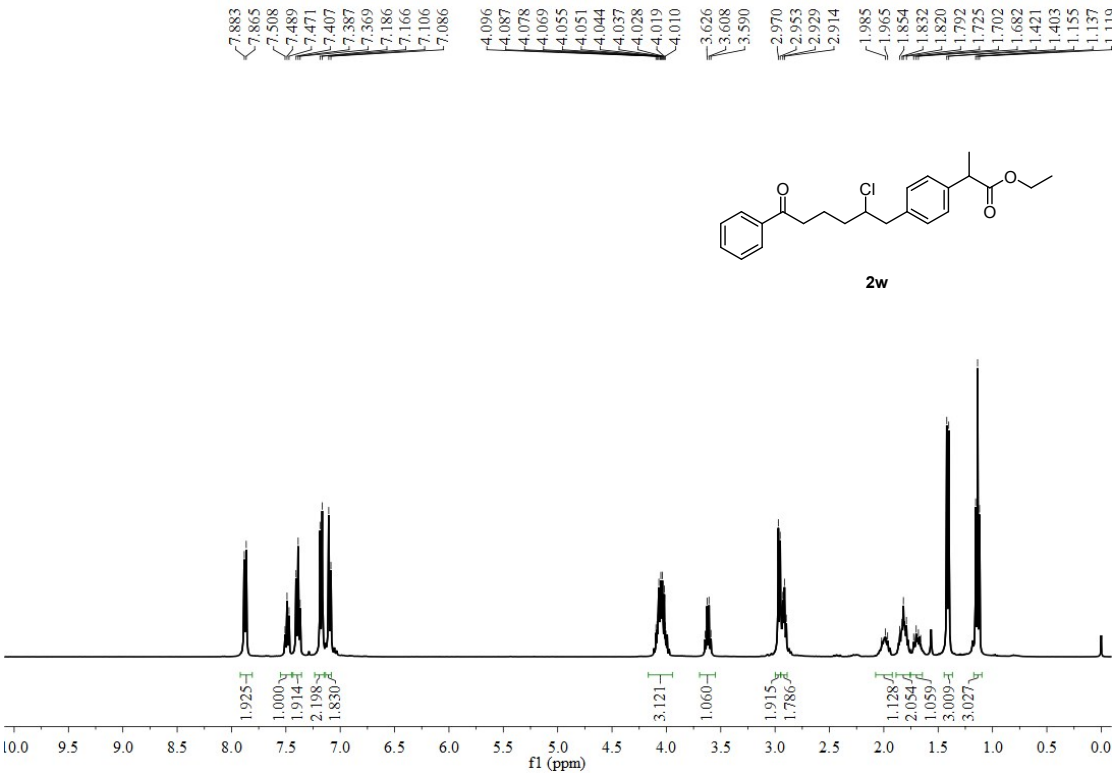


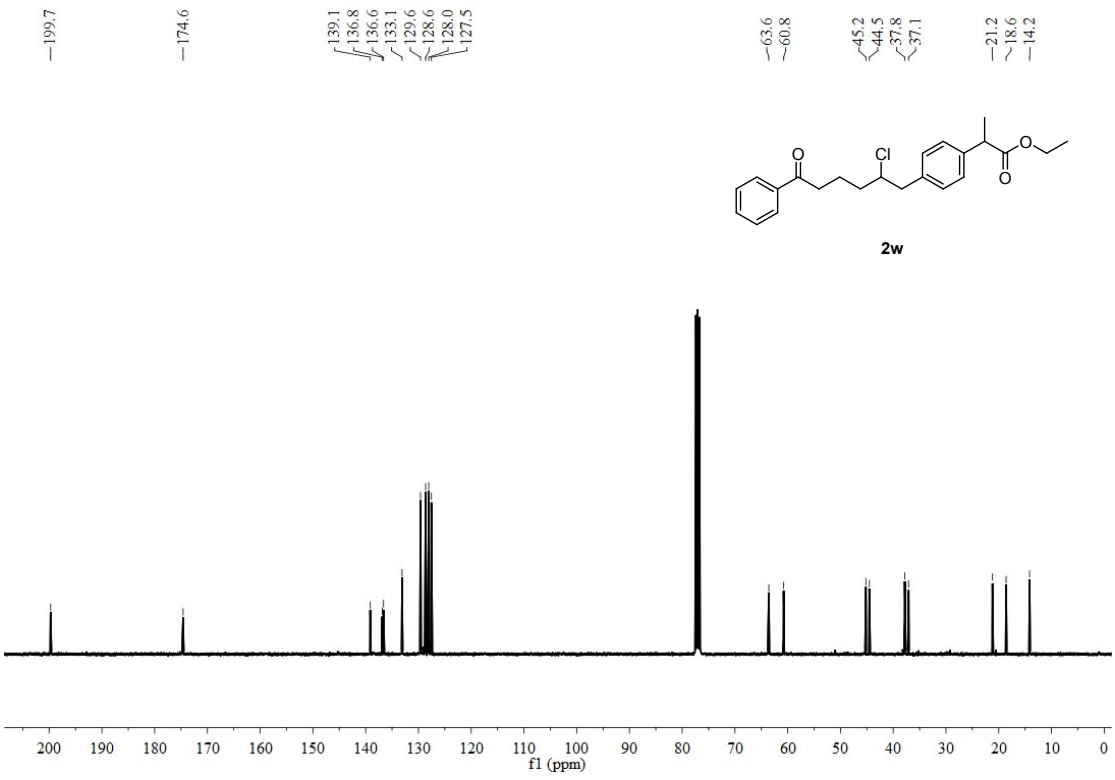
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2v**



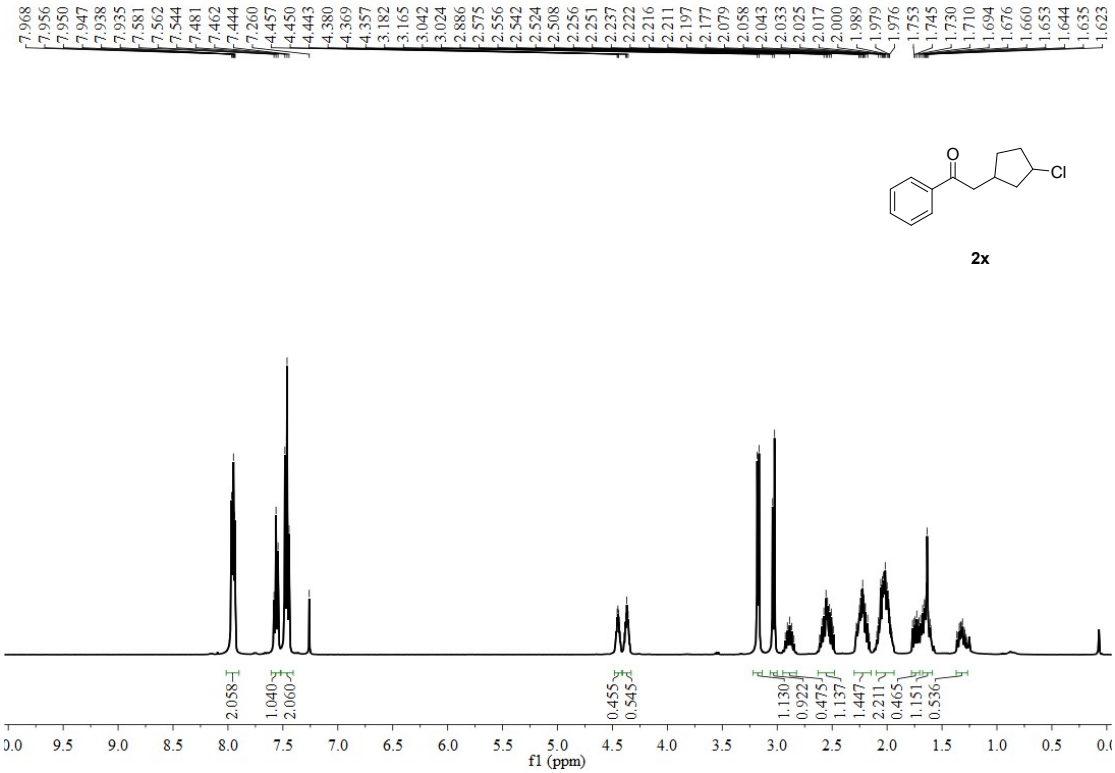


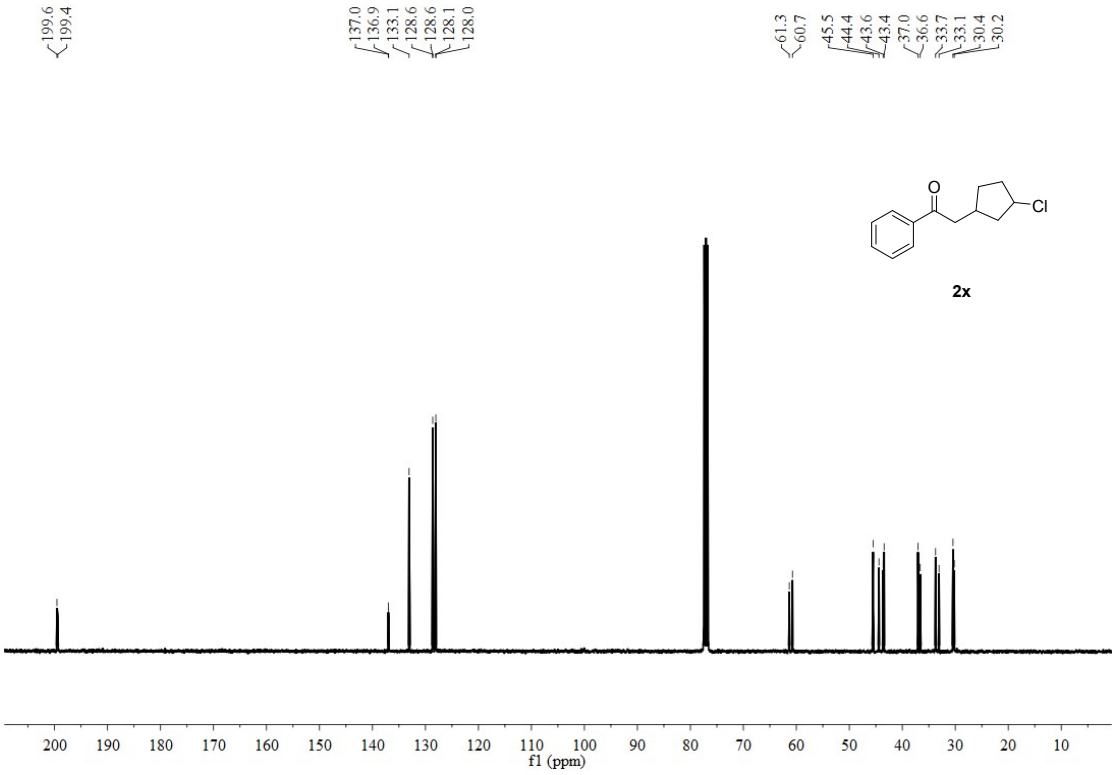
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2w**





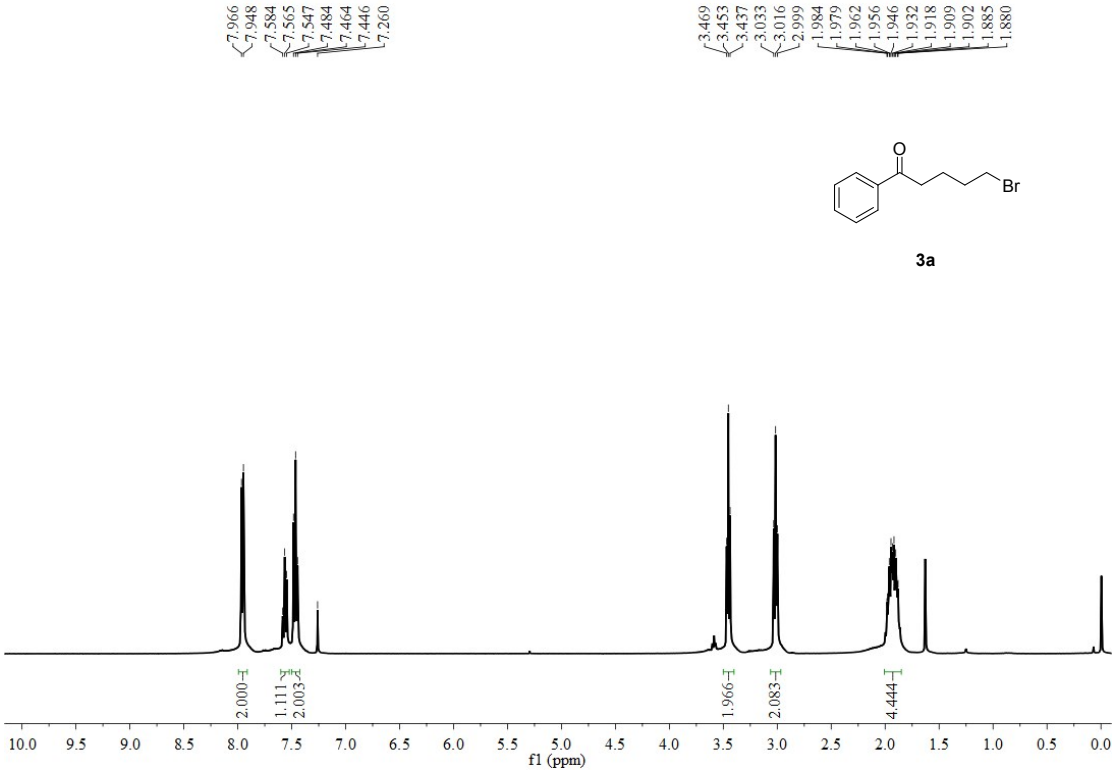
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **2x**

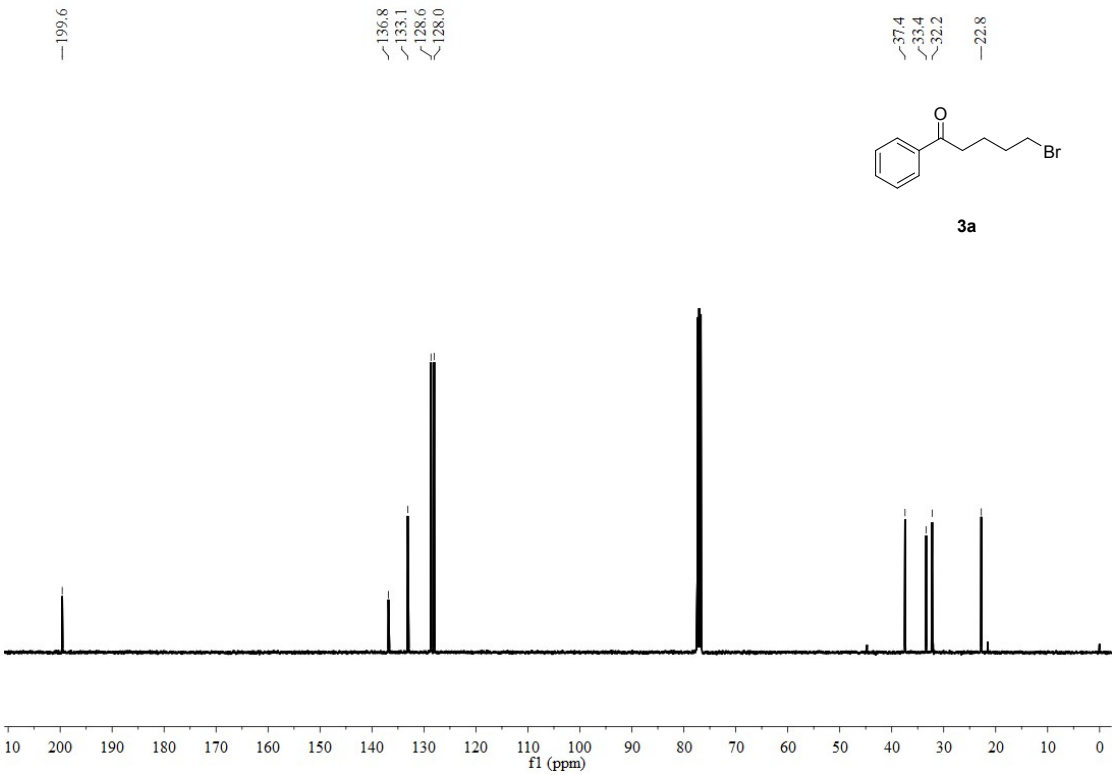




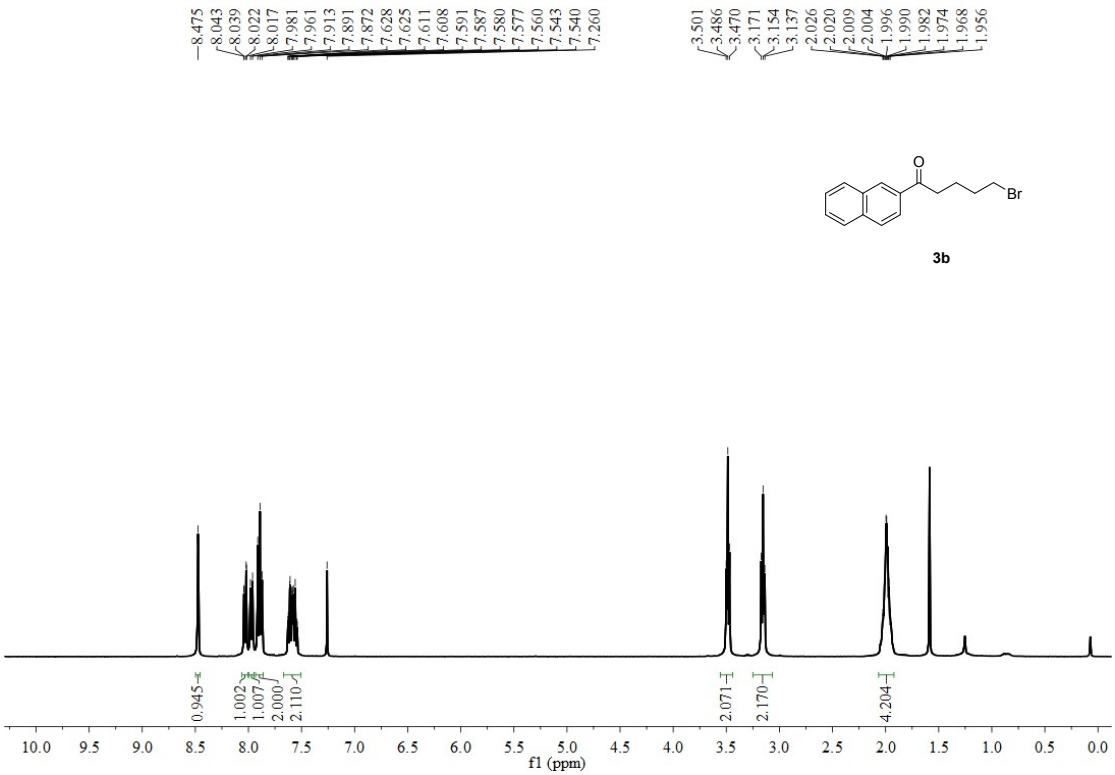
15. ^{1}H NMR and ^{13}C NMR Spectra of Products 3

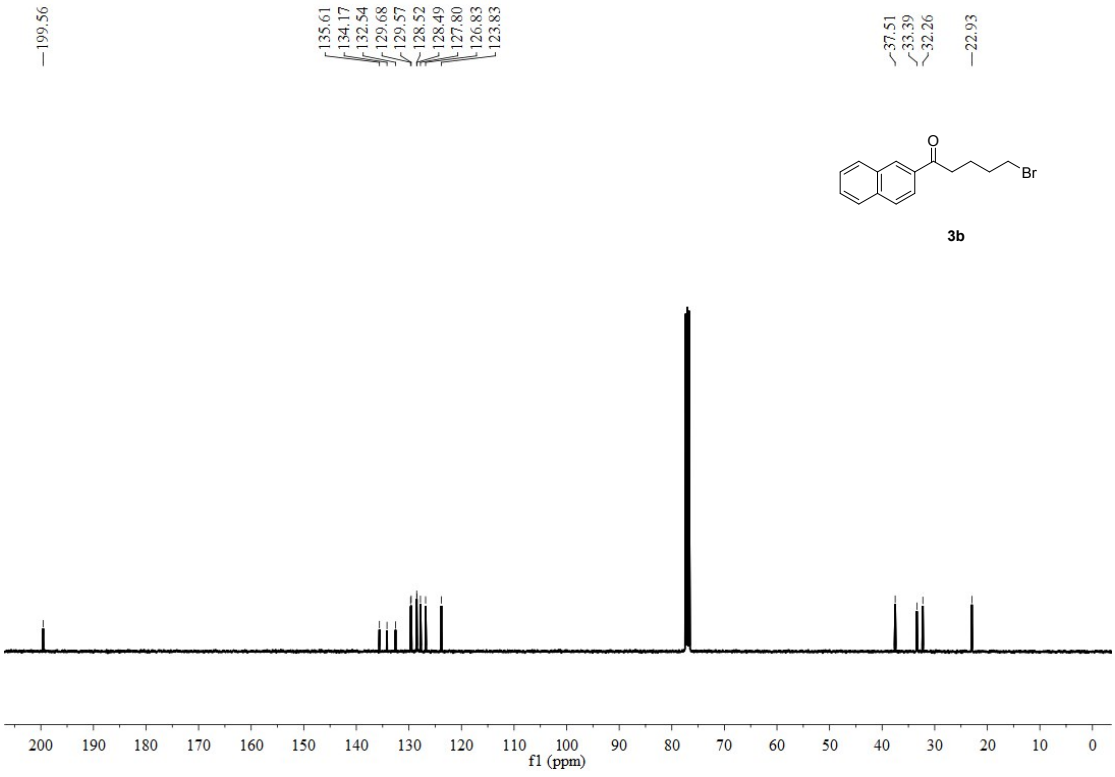
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3a**



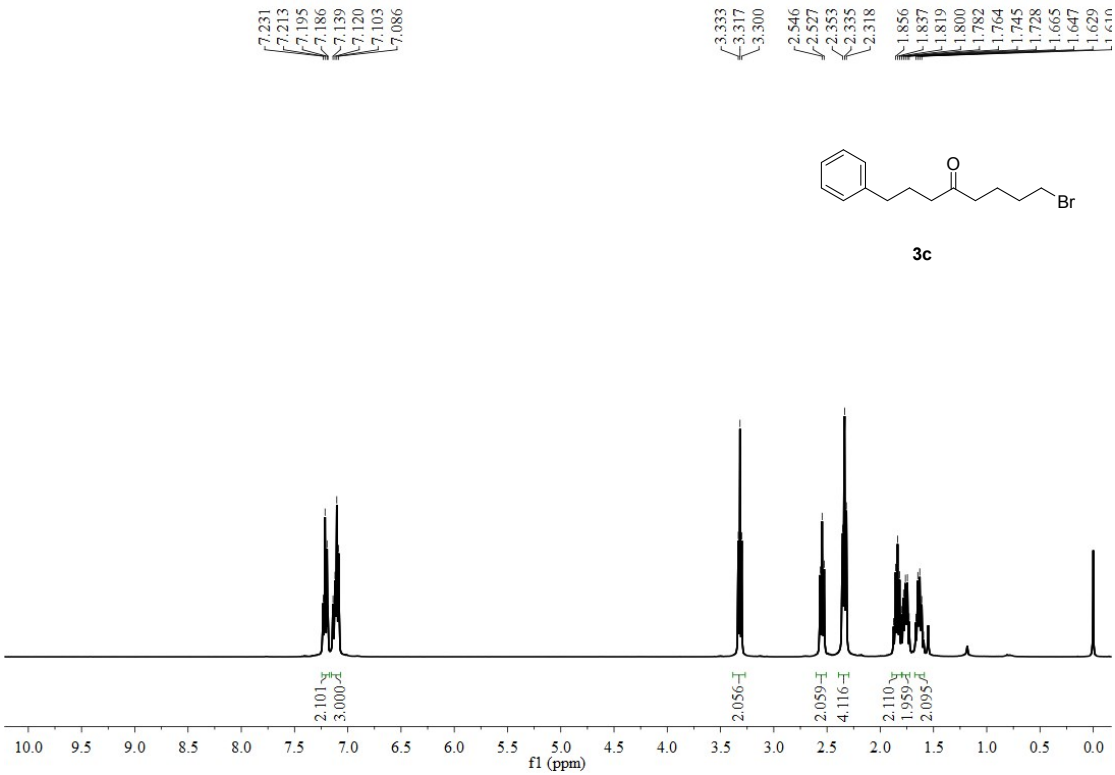


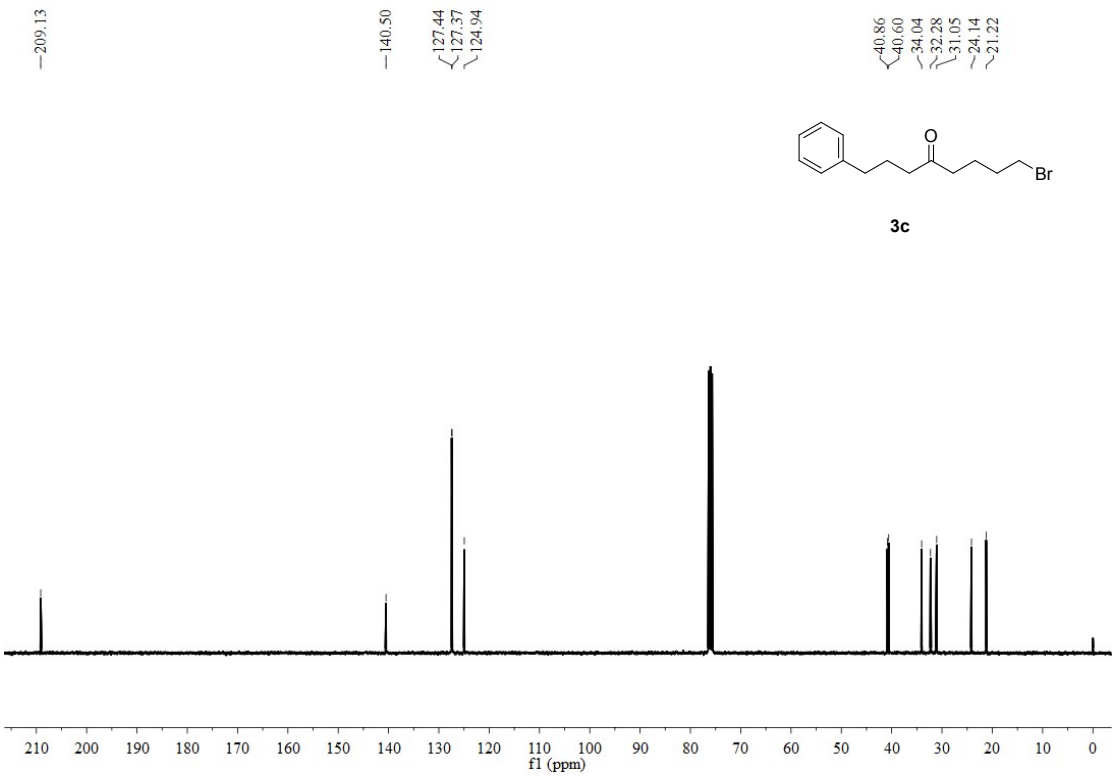
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3b**



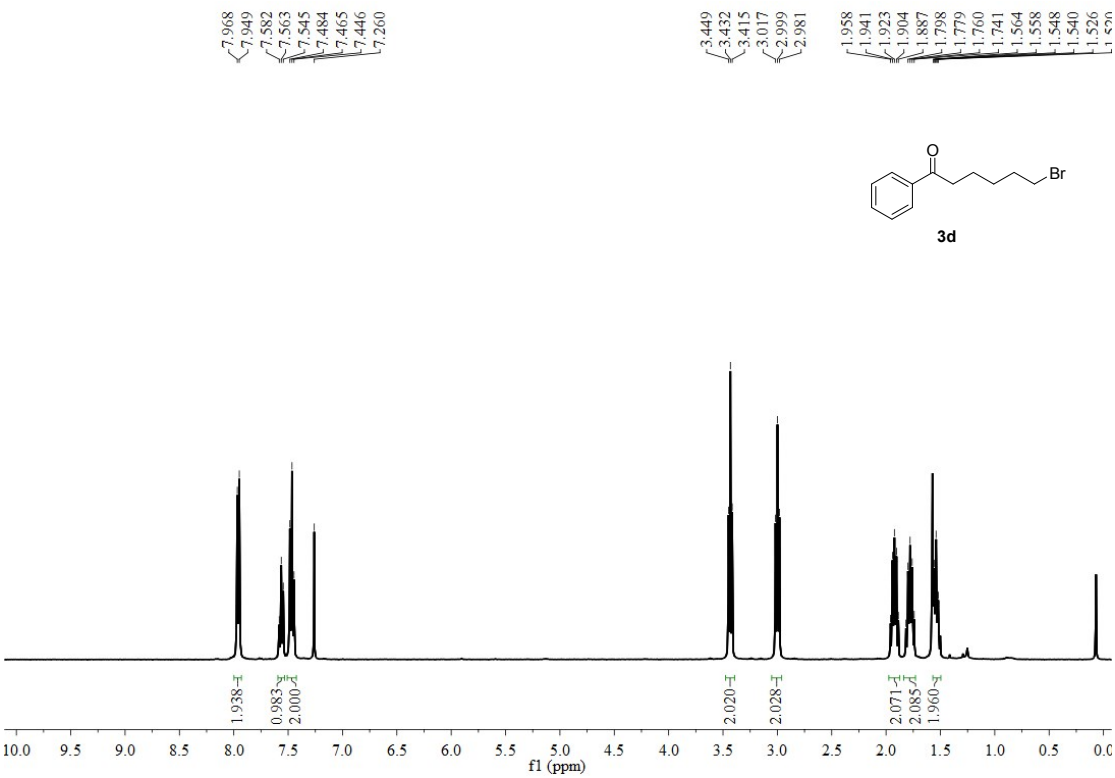


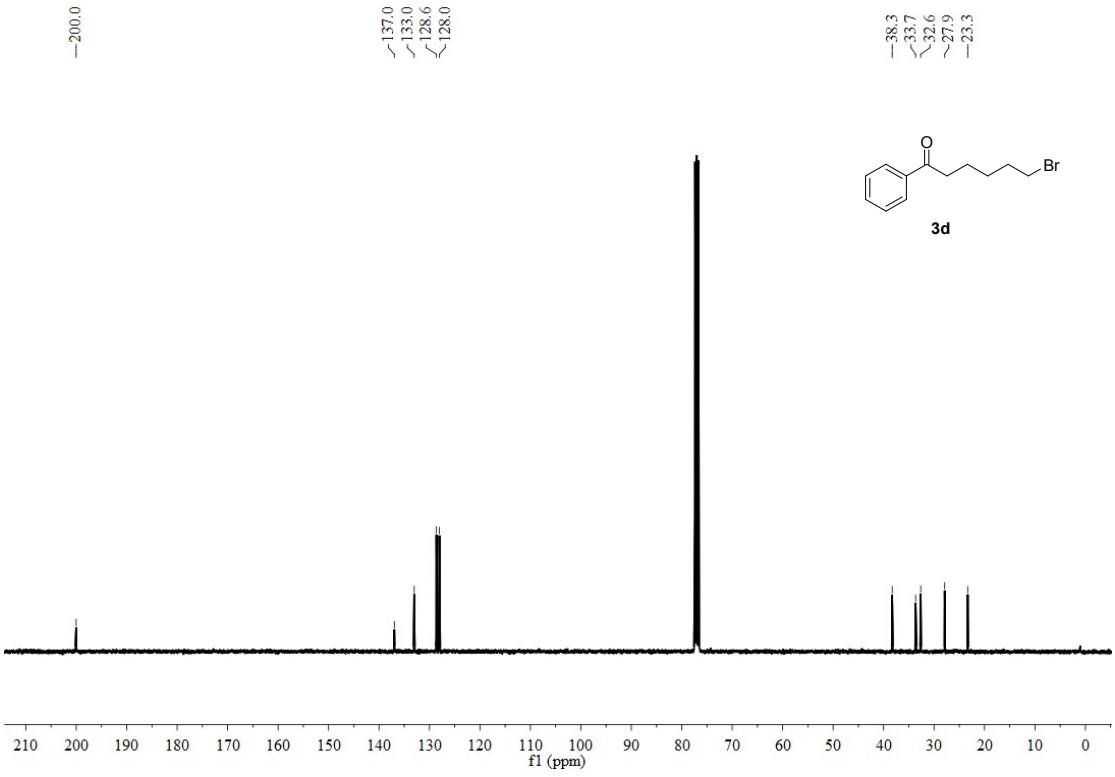
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3c**



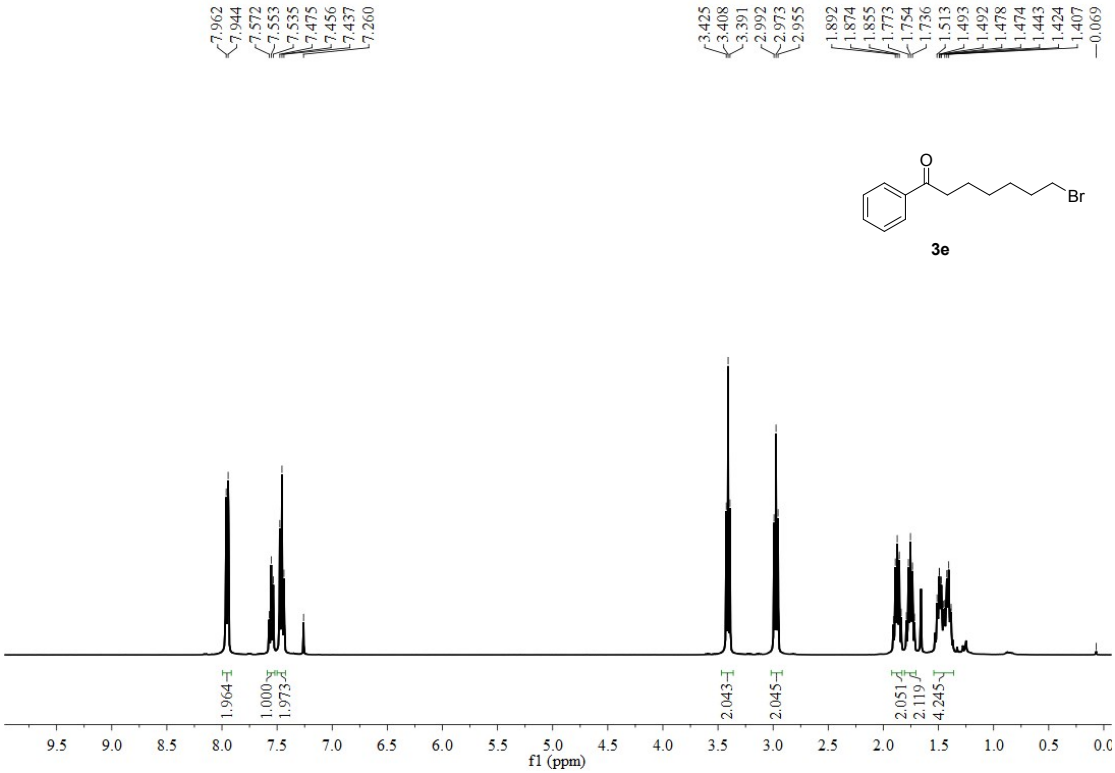


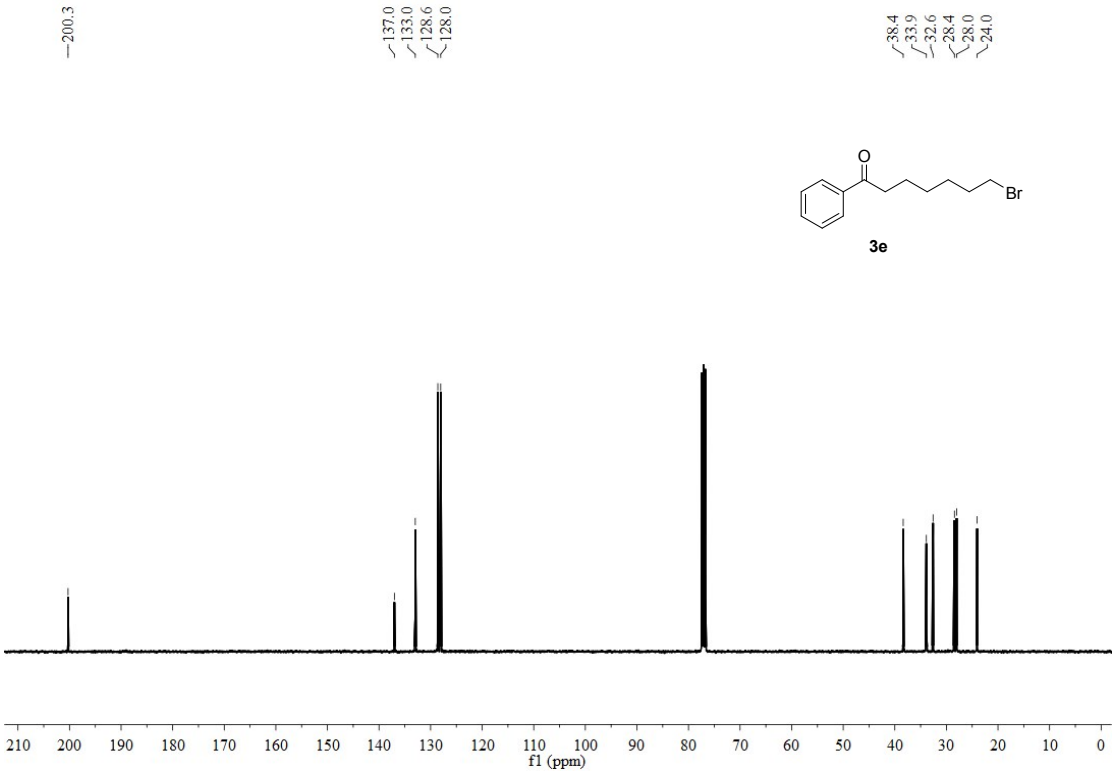
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3d**



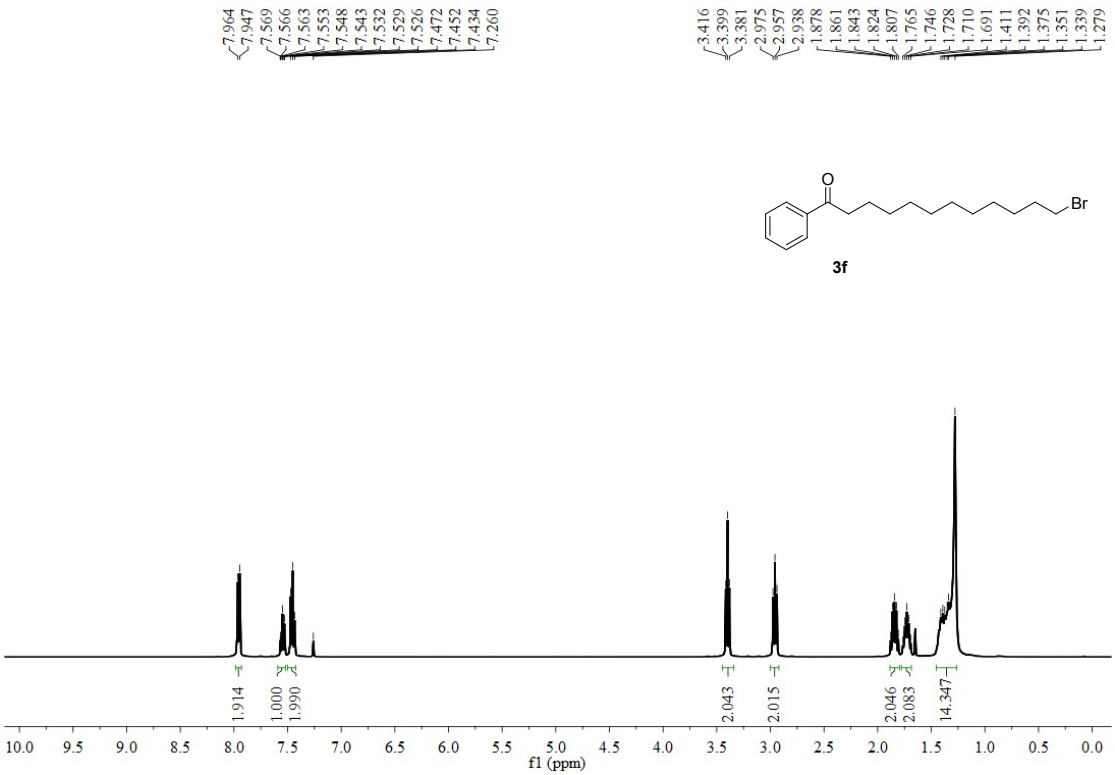


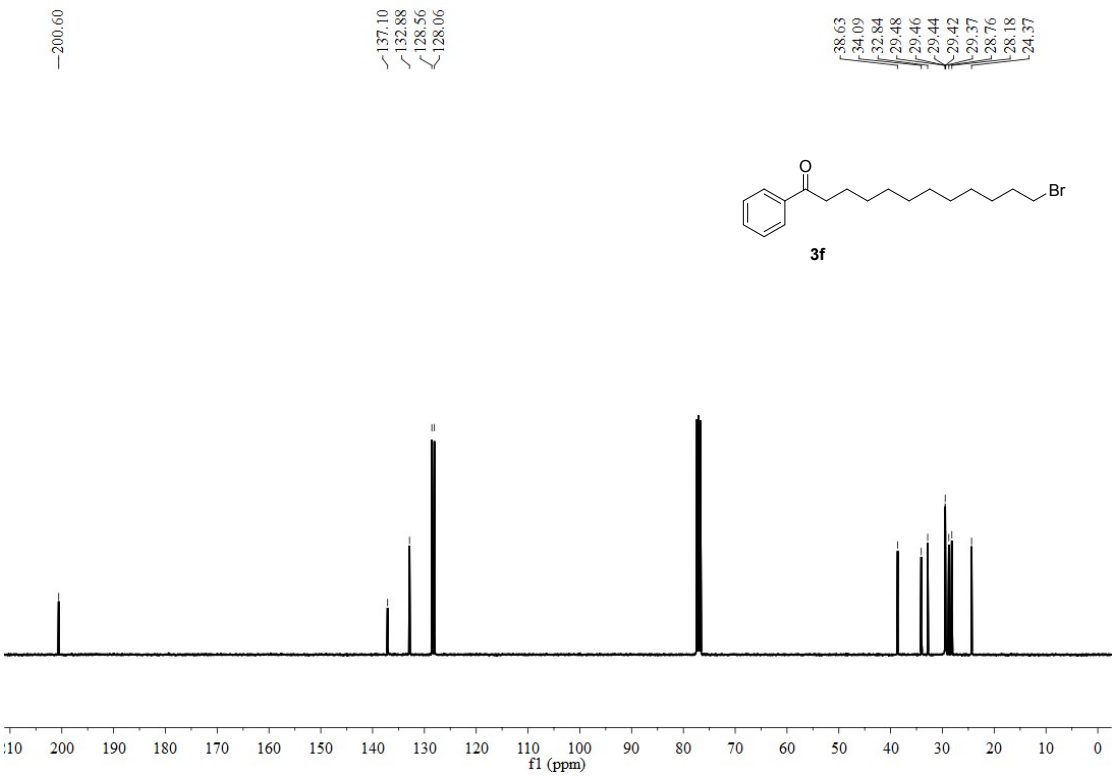
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3e**



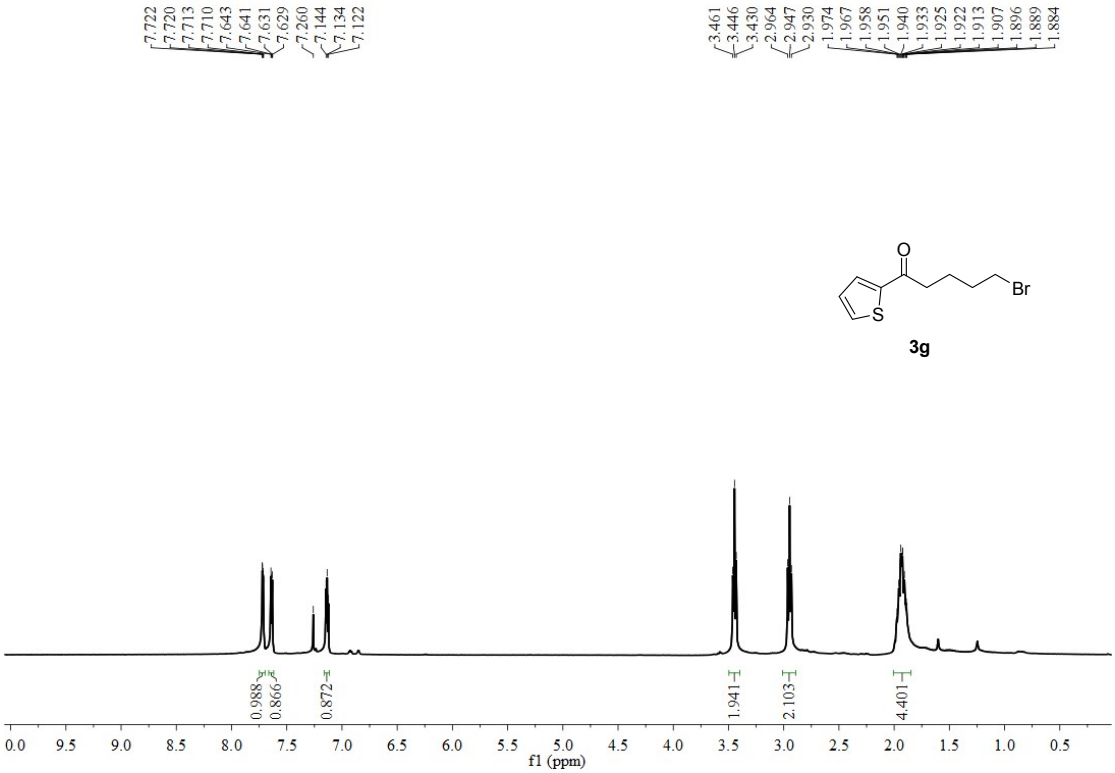


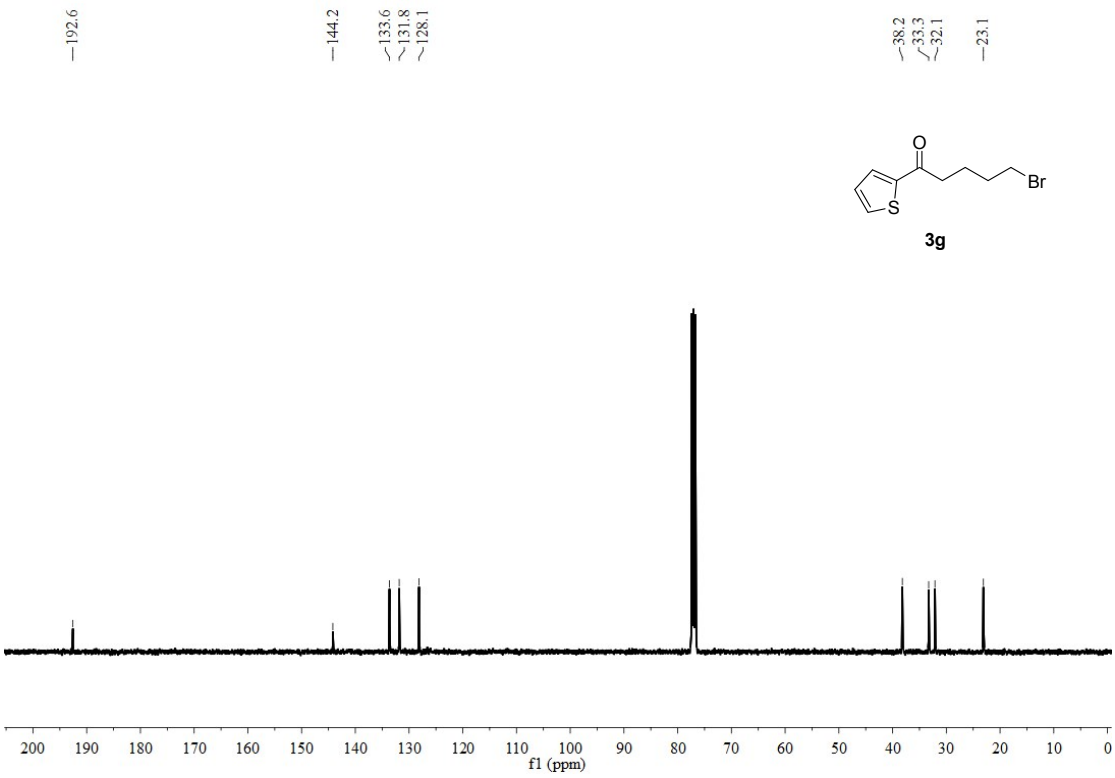
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3f**



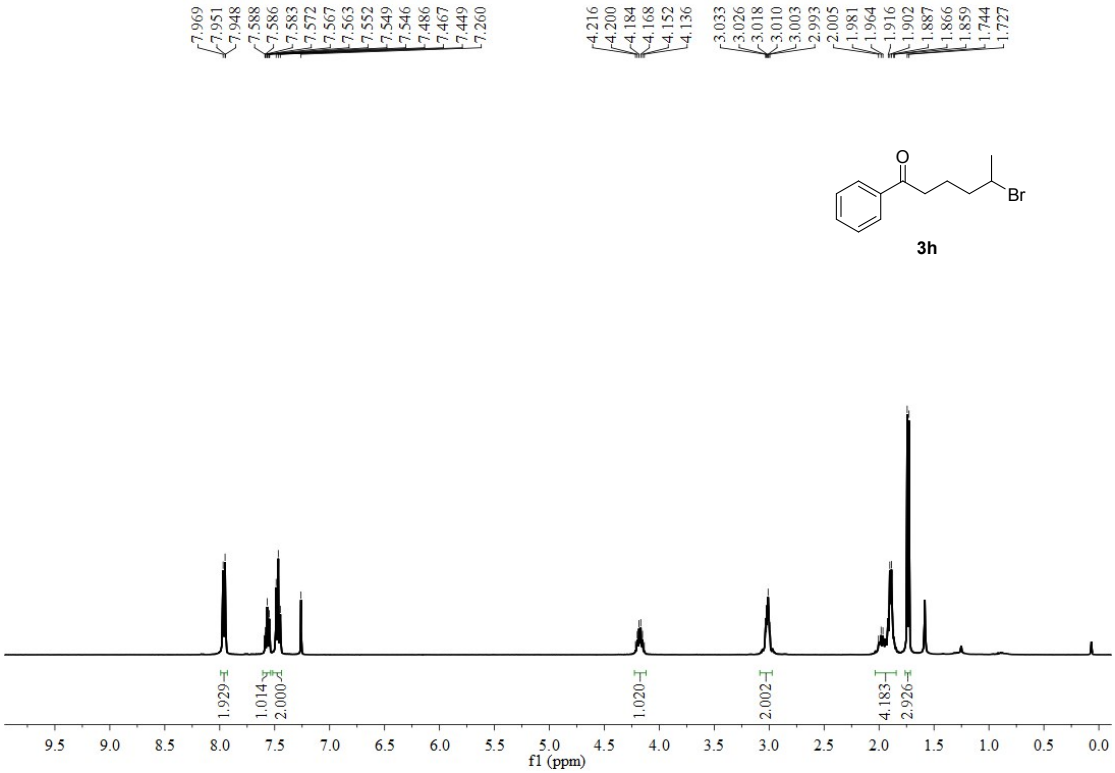


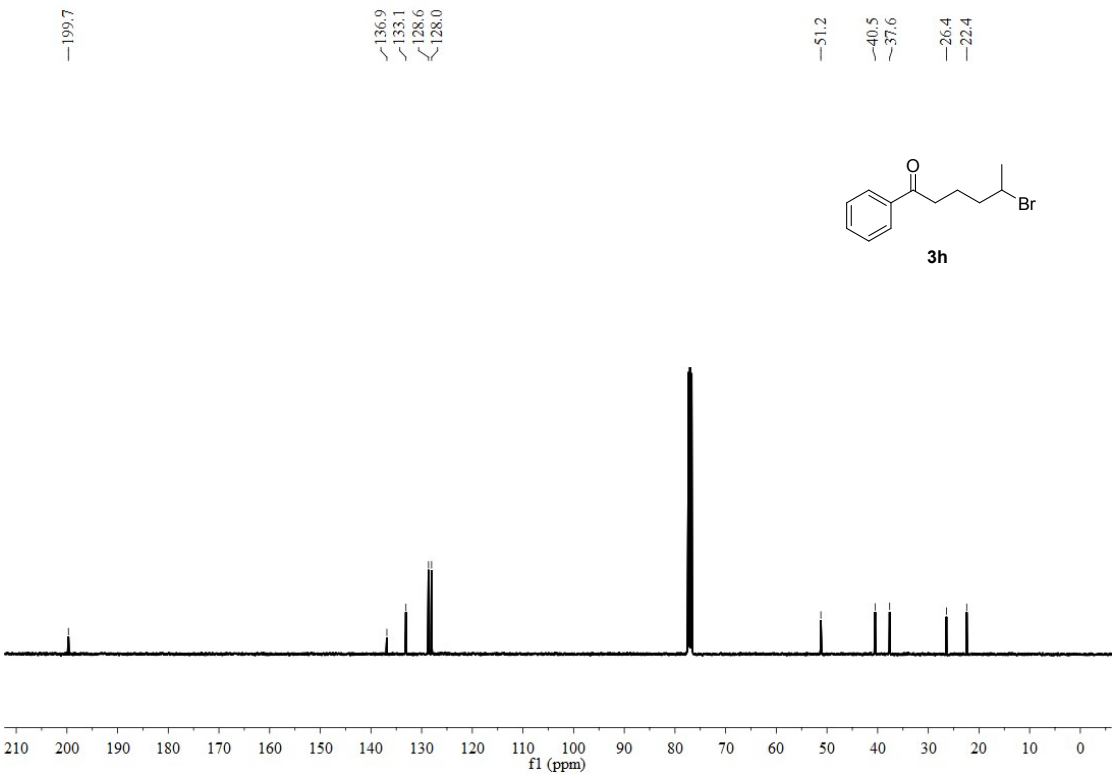
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3g**



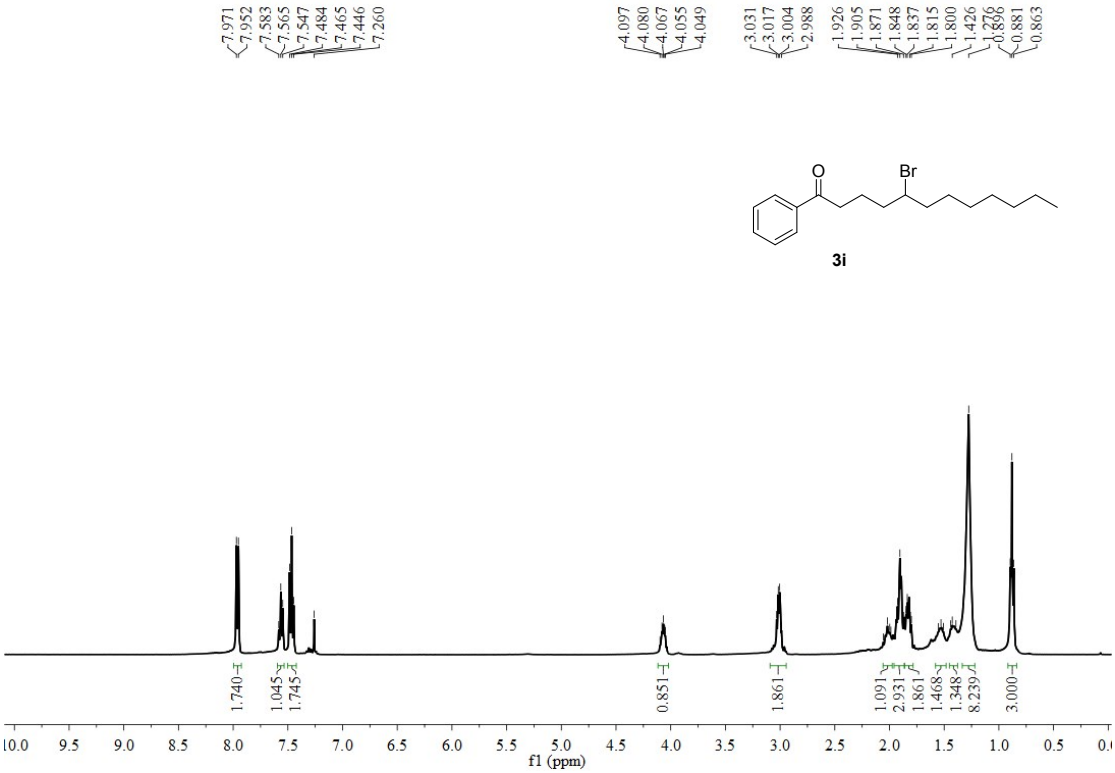


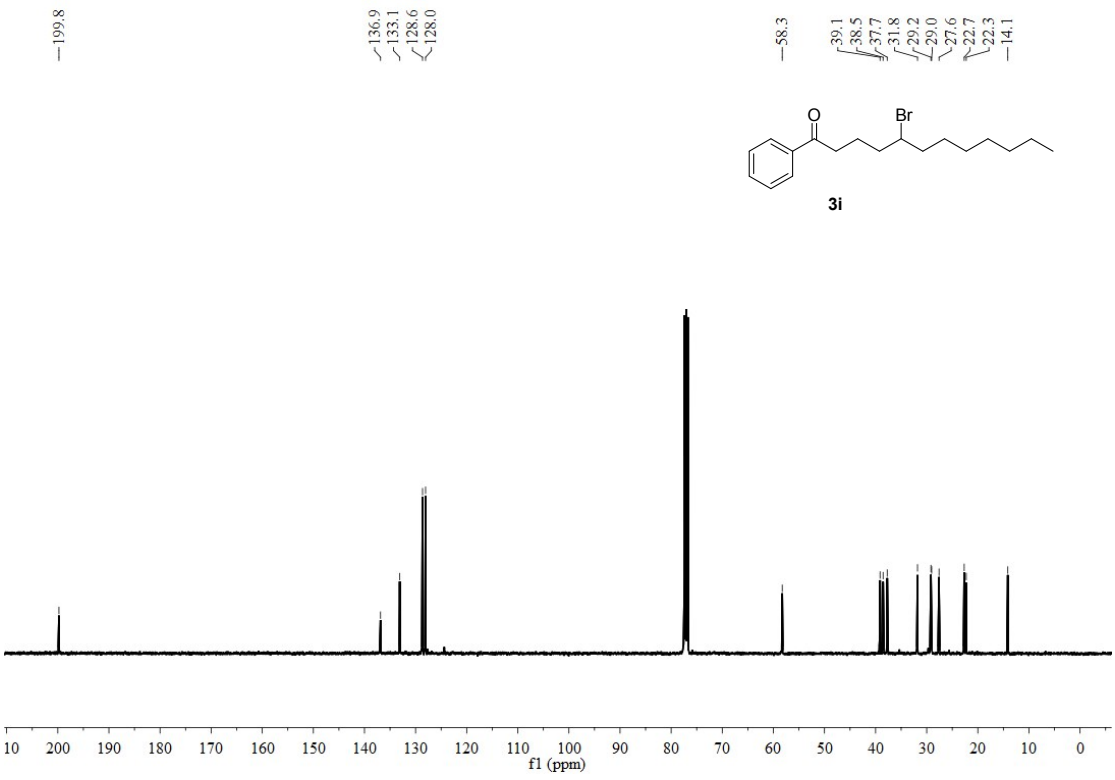
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3h**



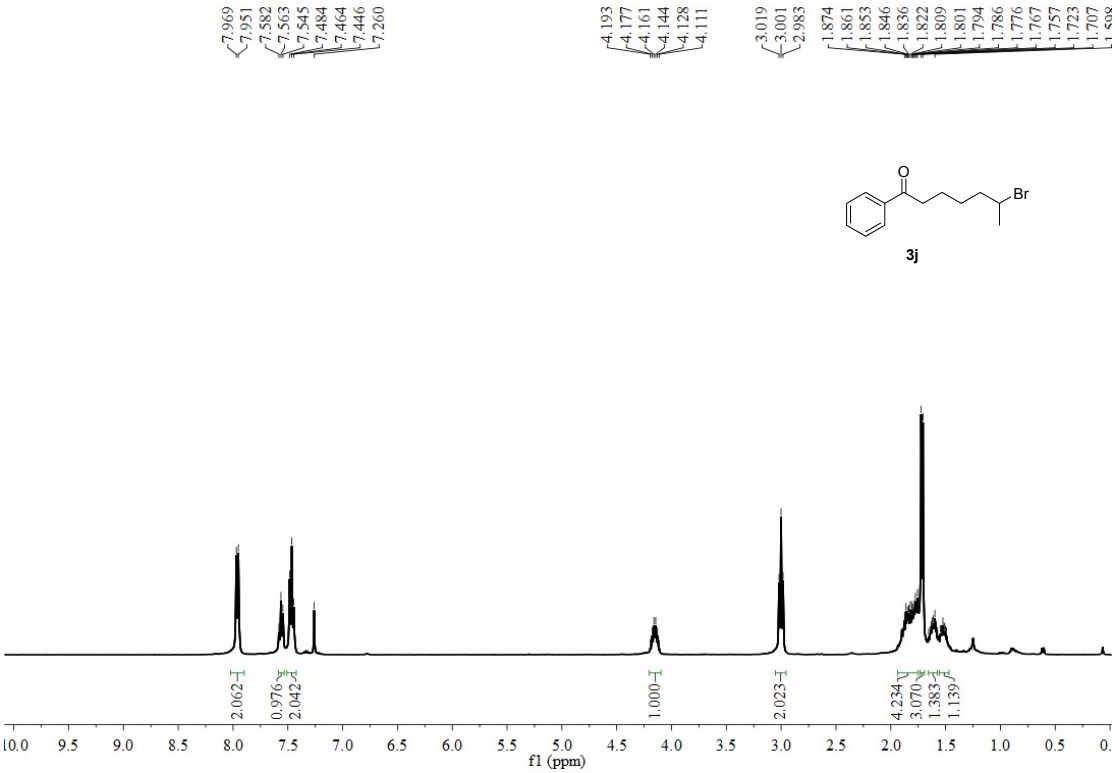


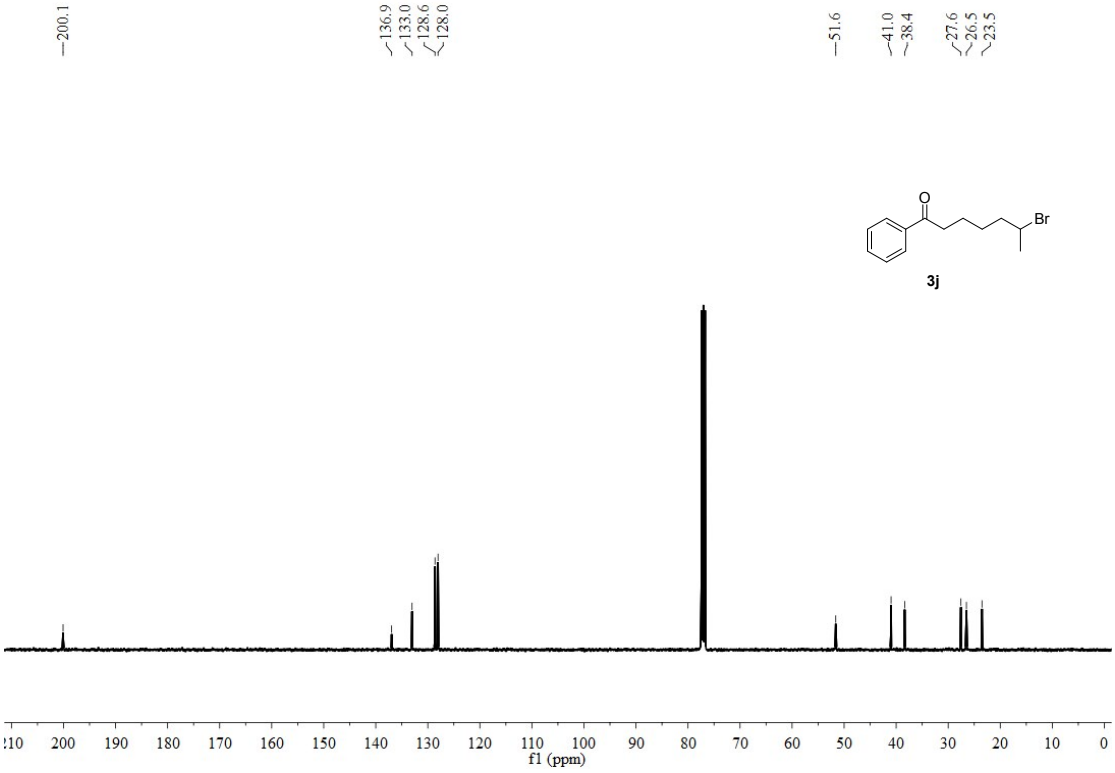
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3i**



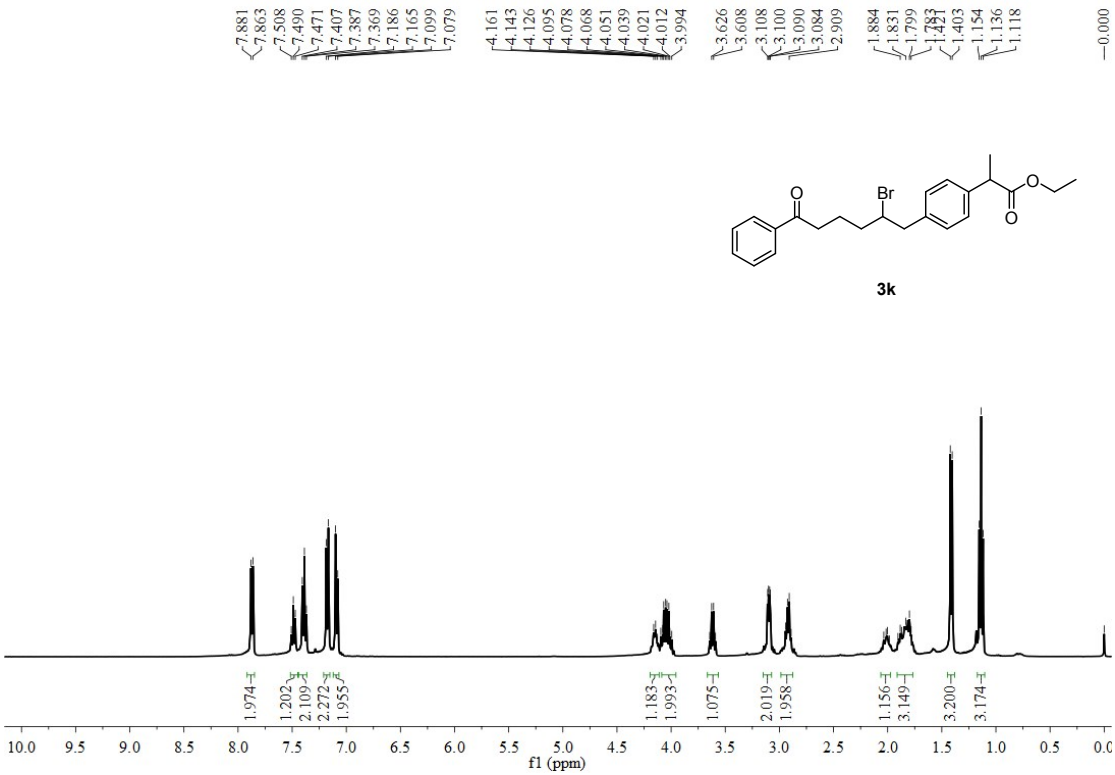


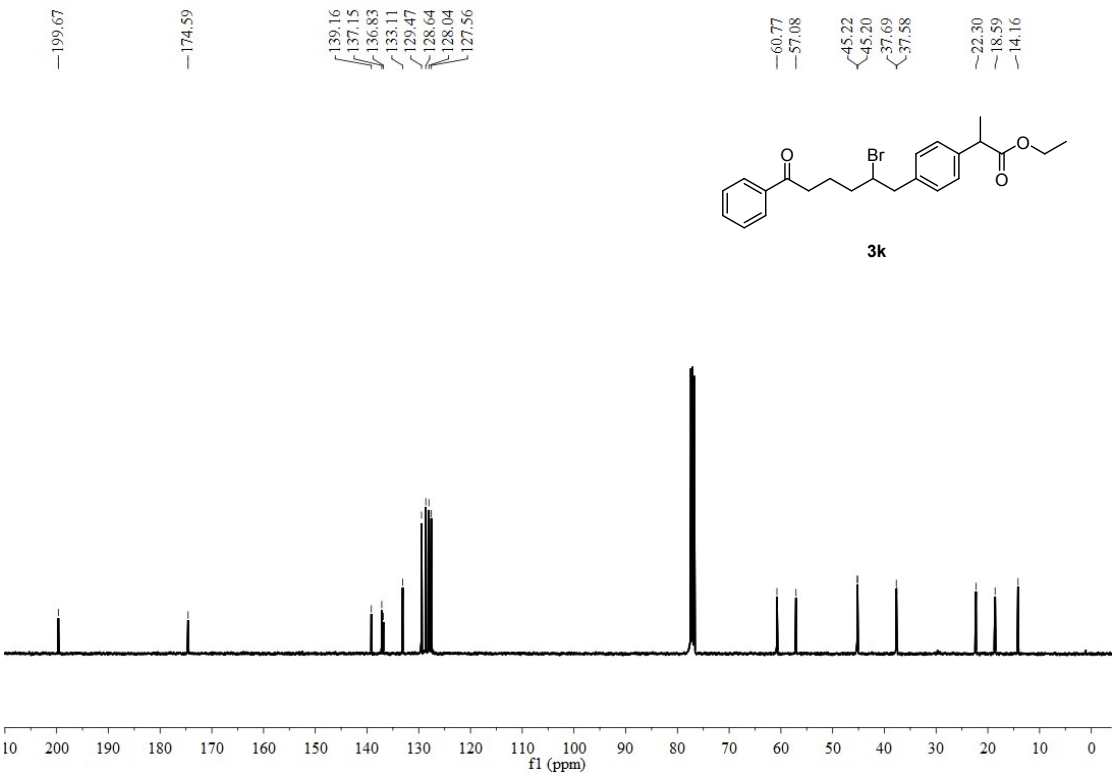
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3j**





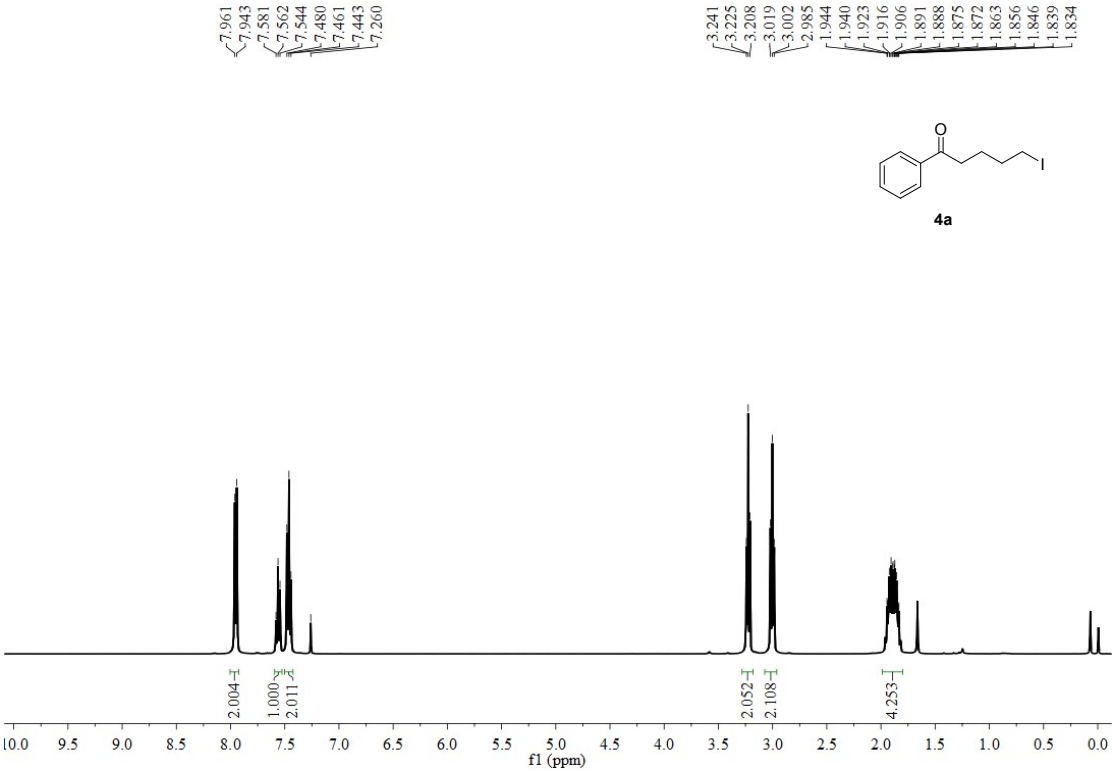
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **3k**

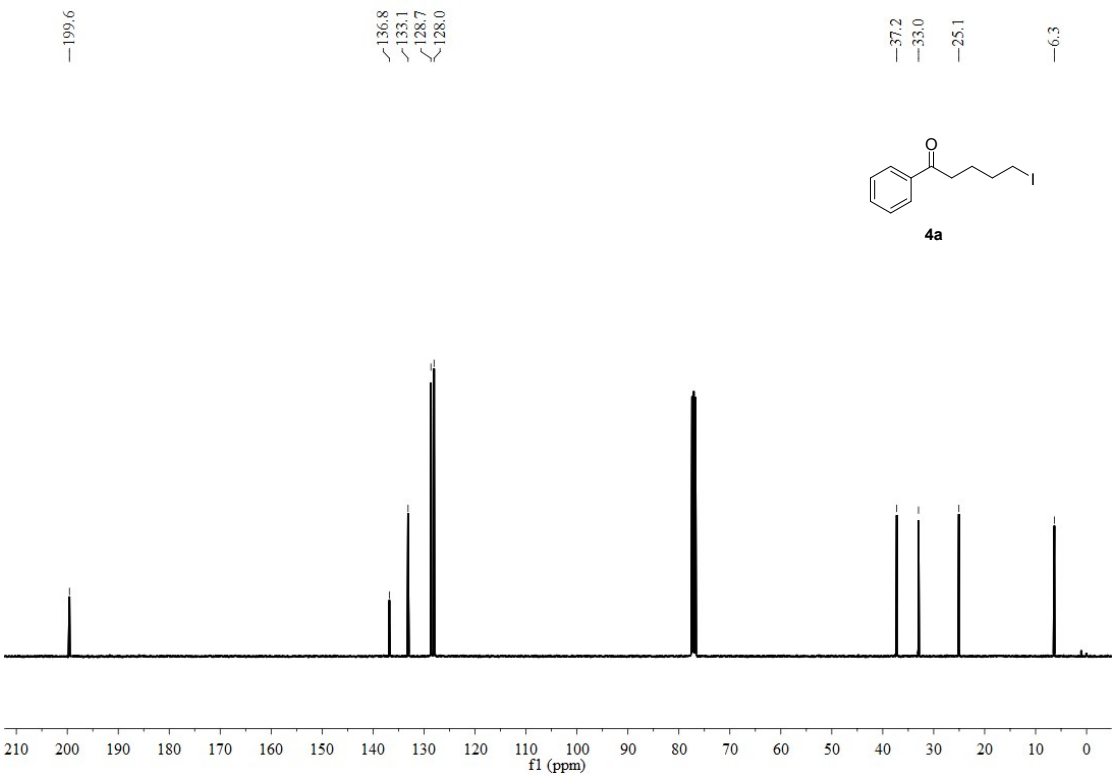




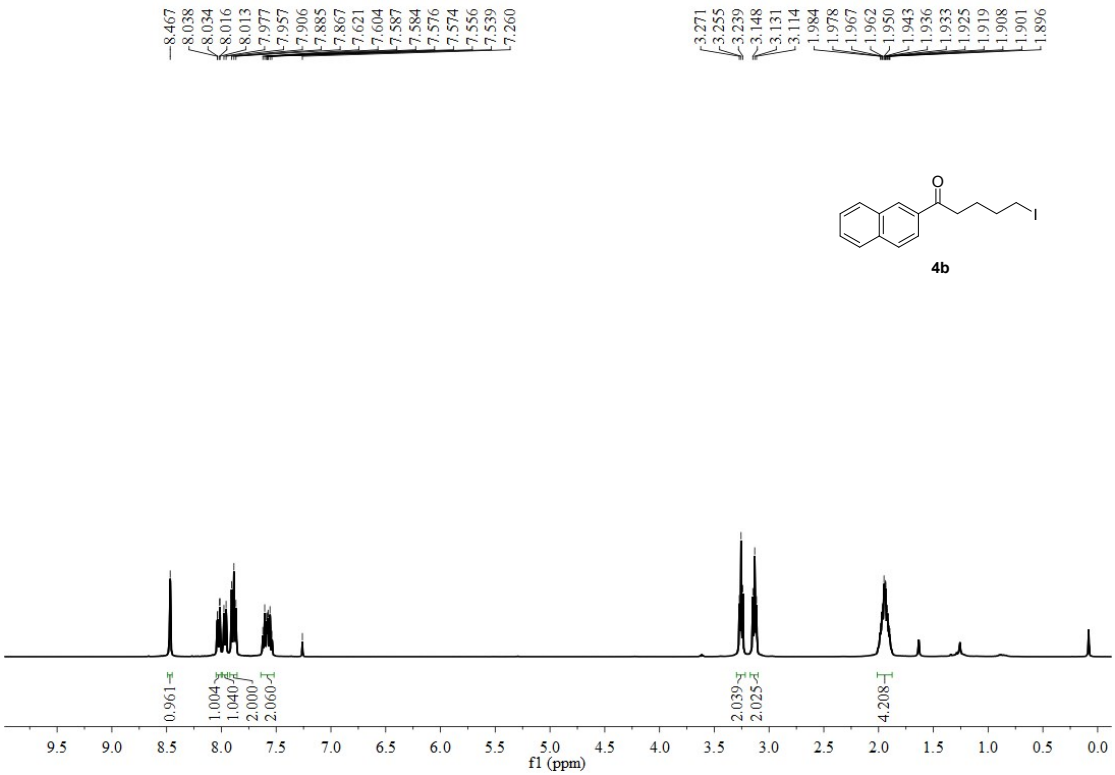
16. ^{1}H NMR and ^{13}C NMR Spectra of Products 4

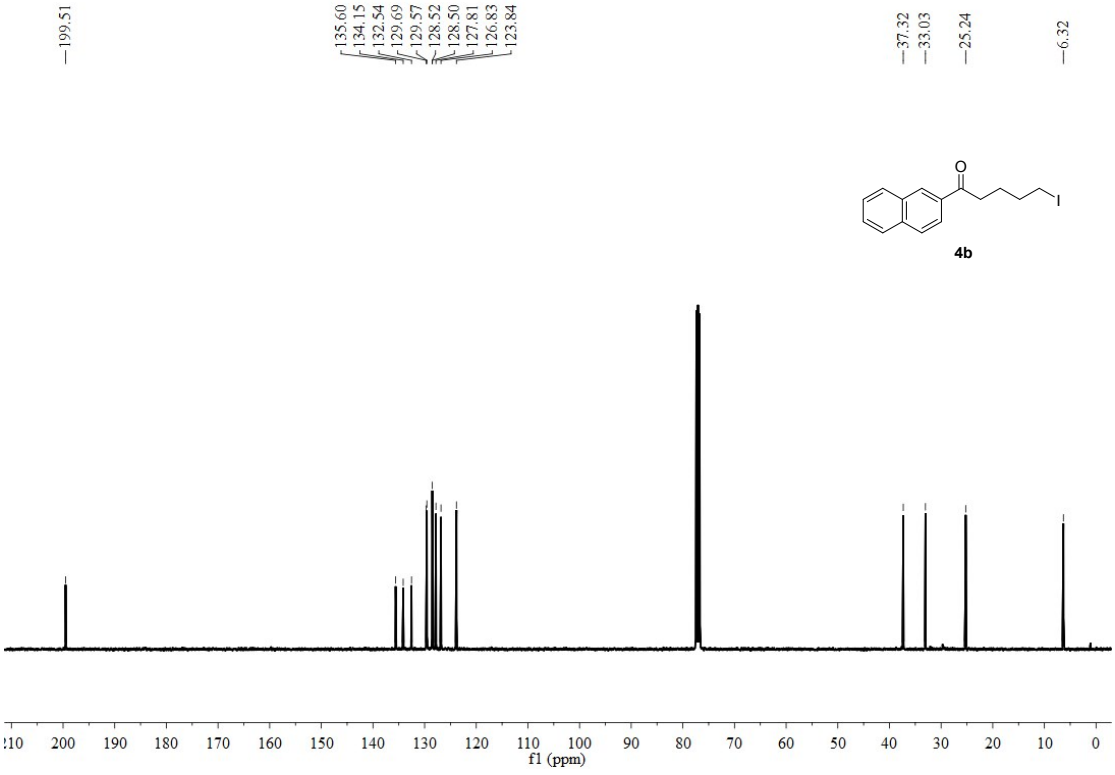
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4a**



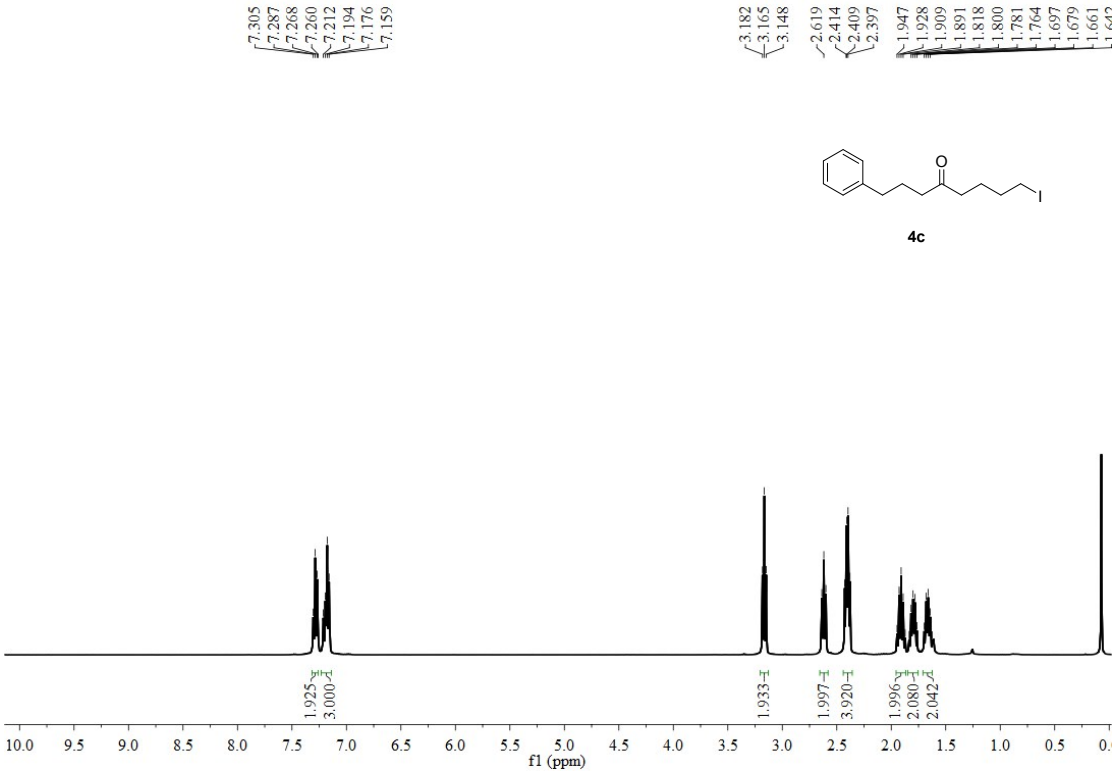


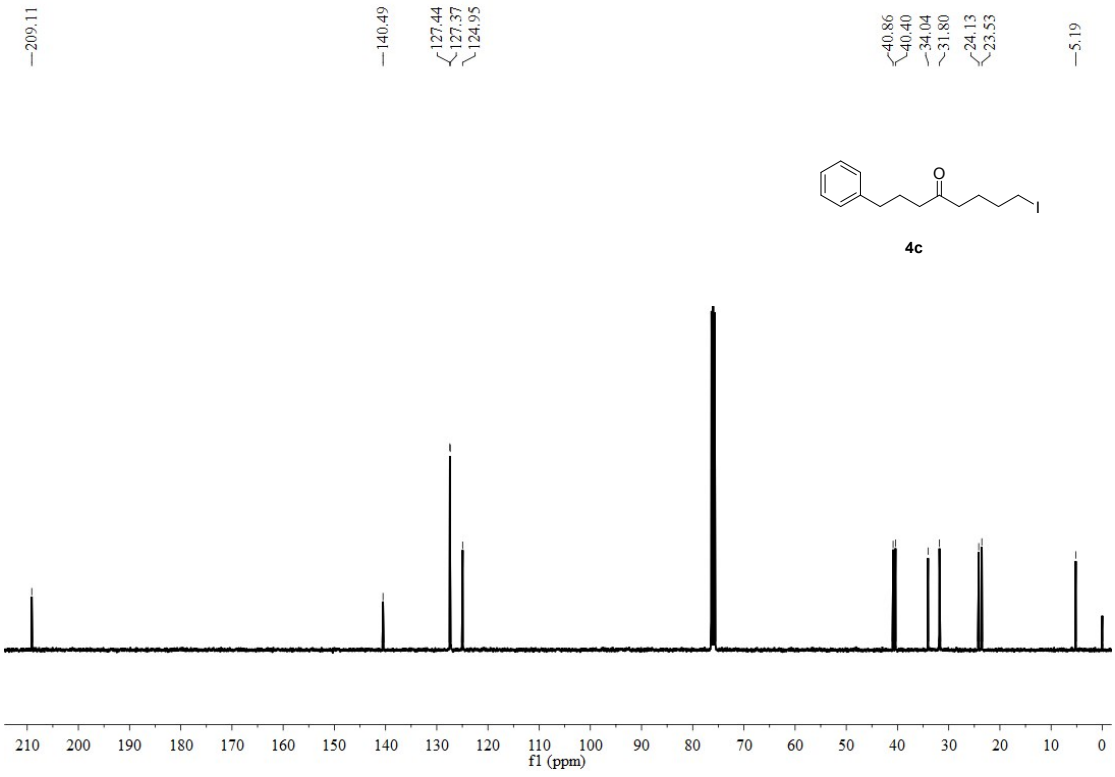
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4b**



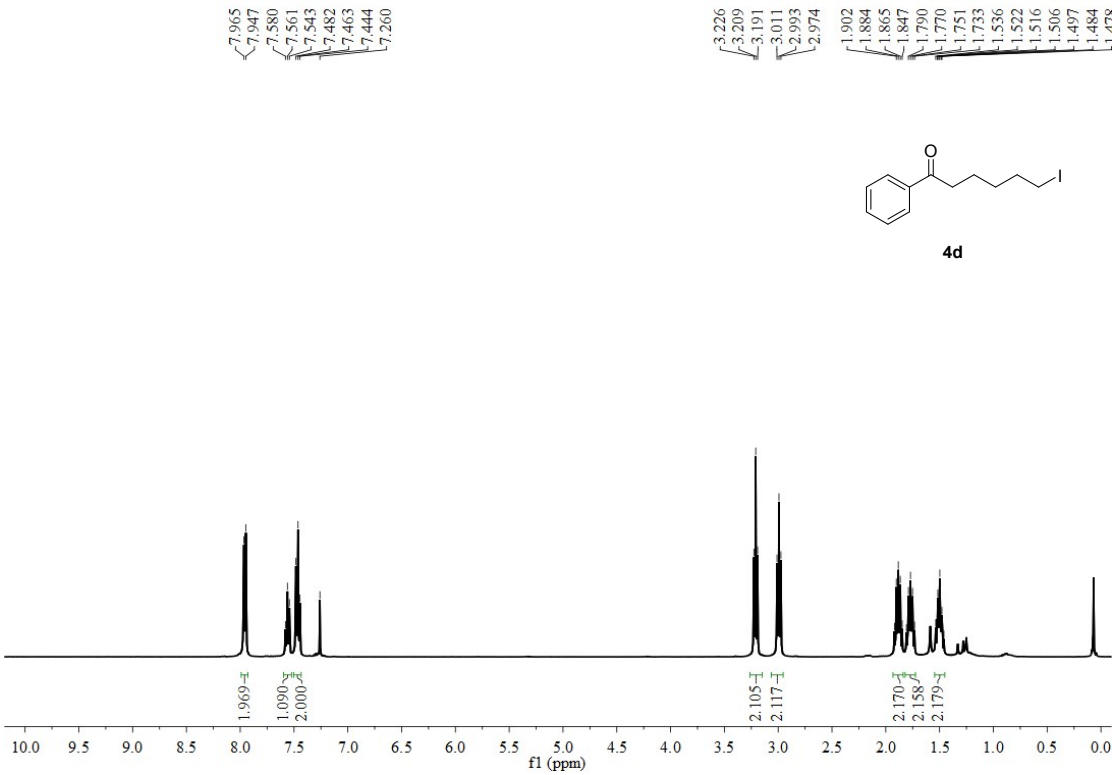


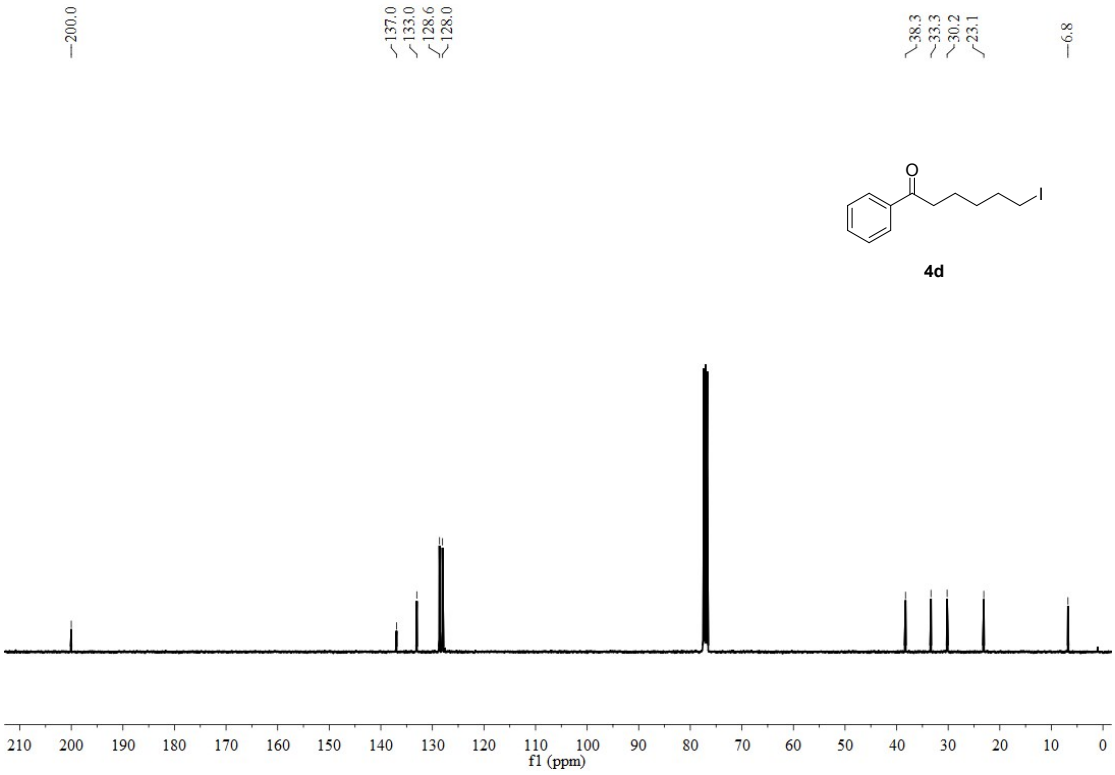
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4c**



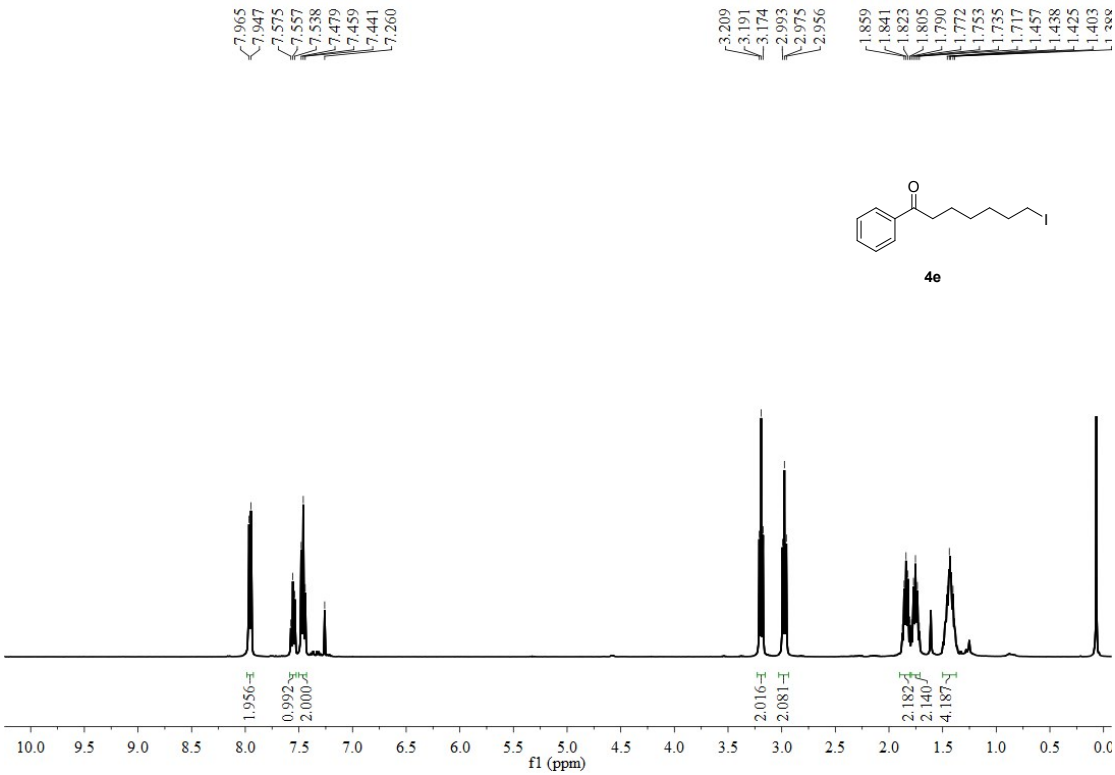


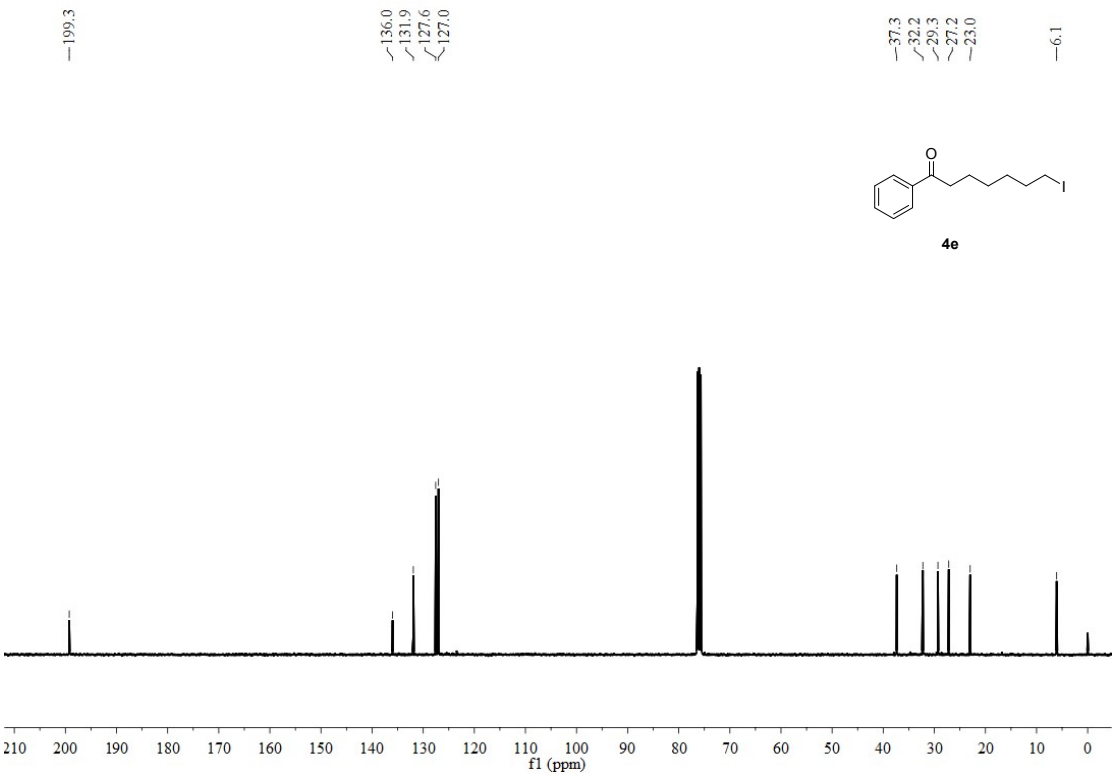
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4d**



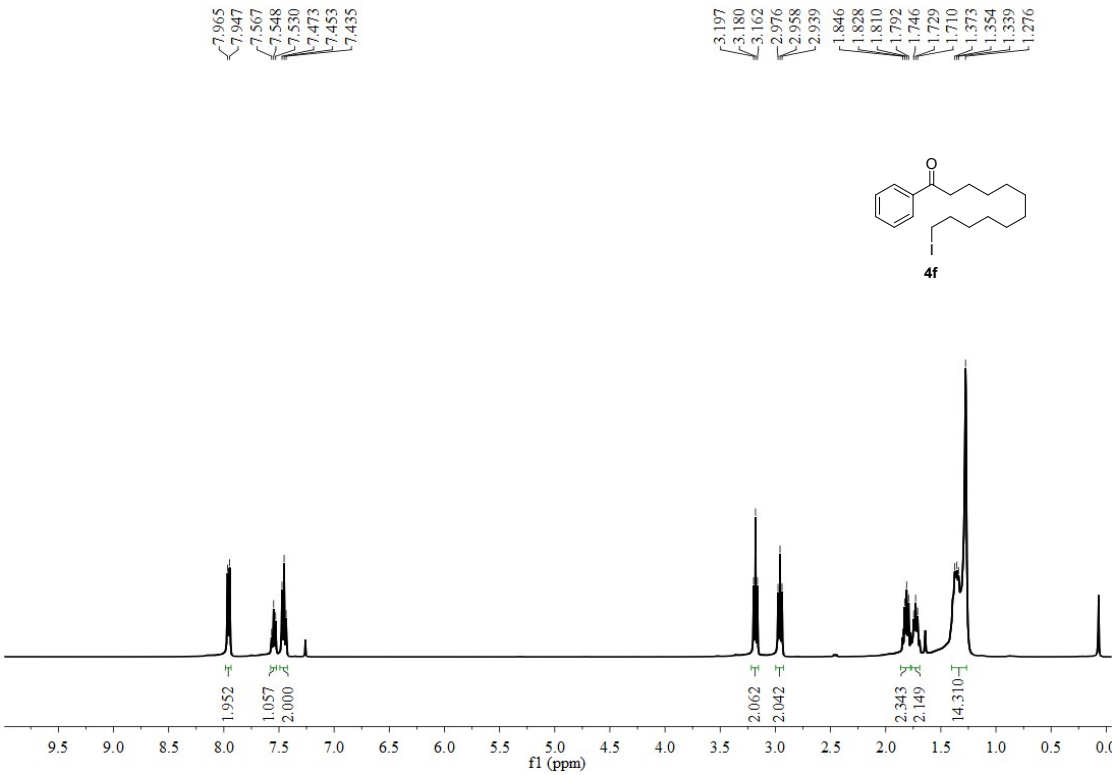


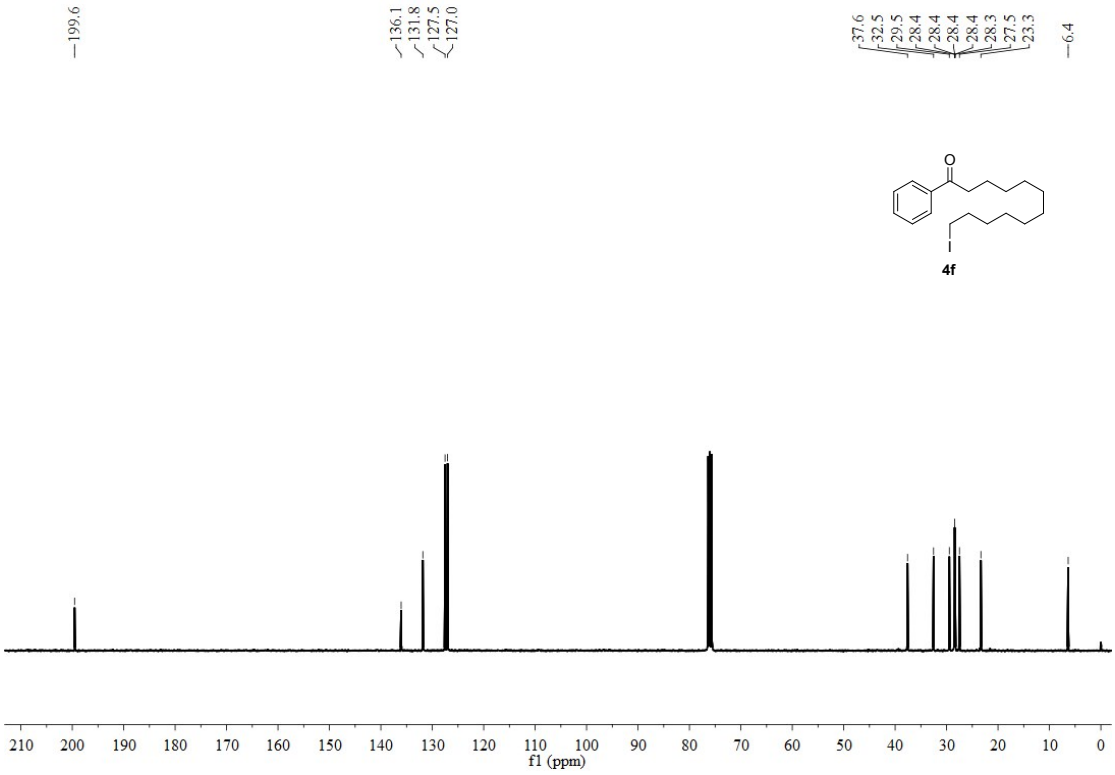
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4e**



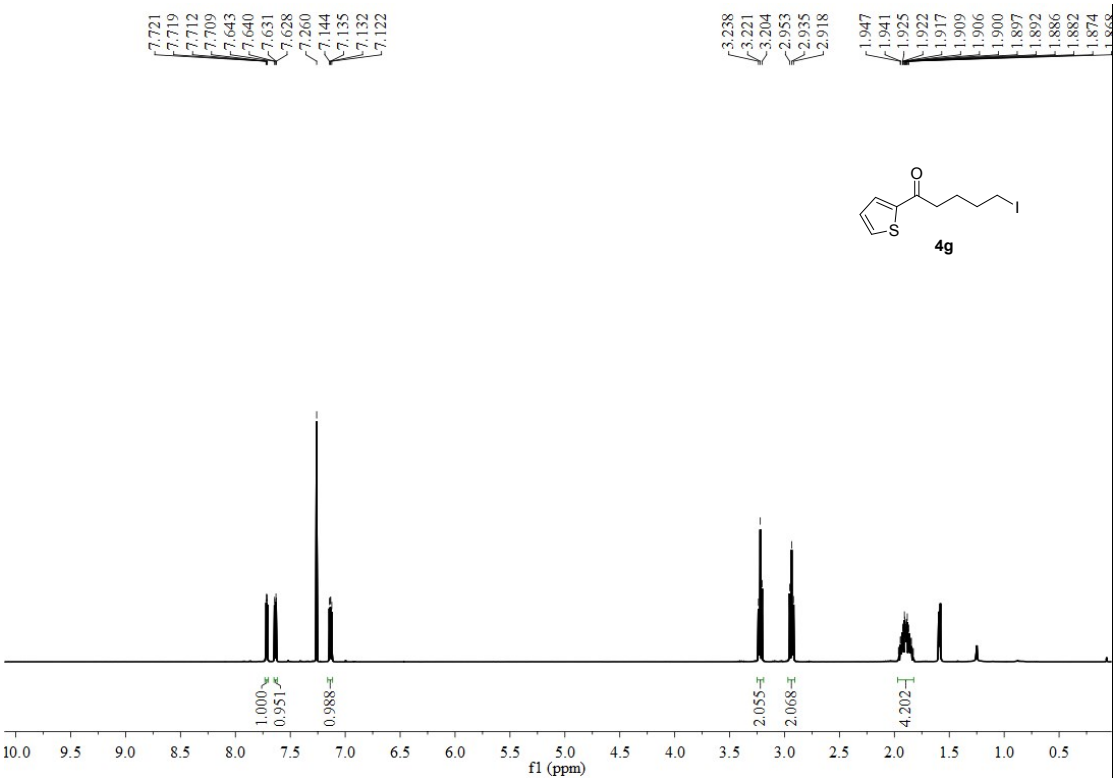


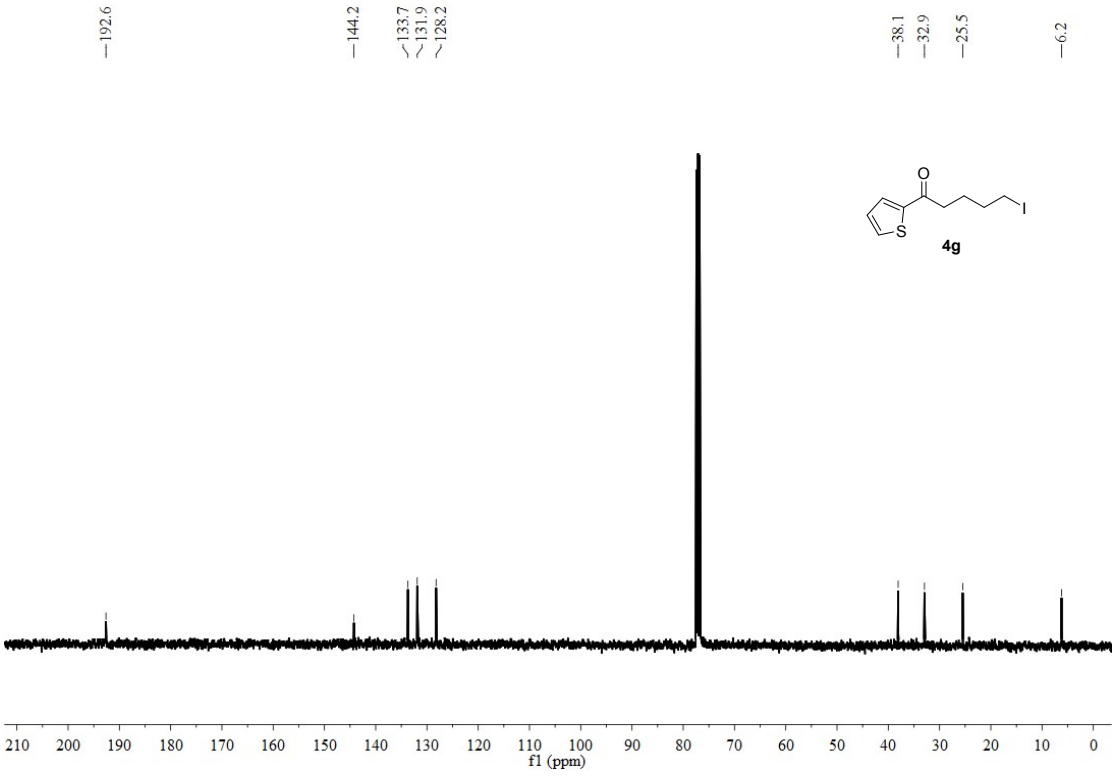
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4f**



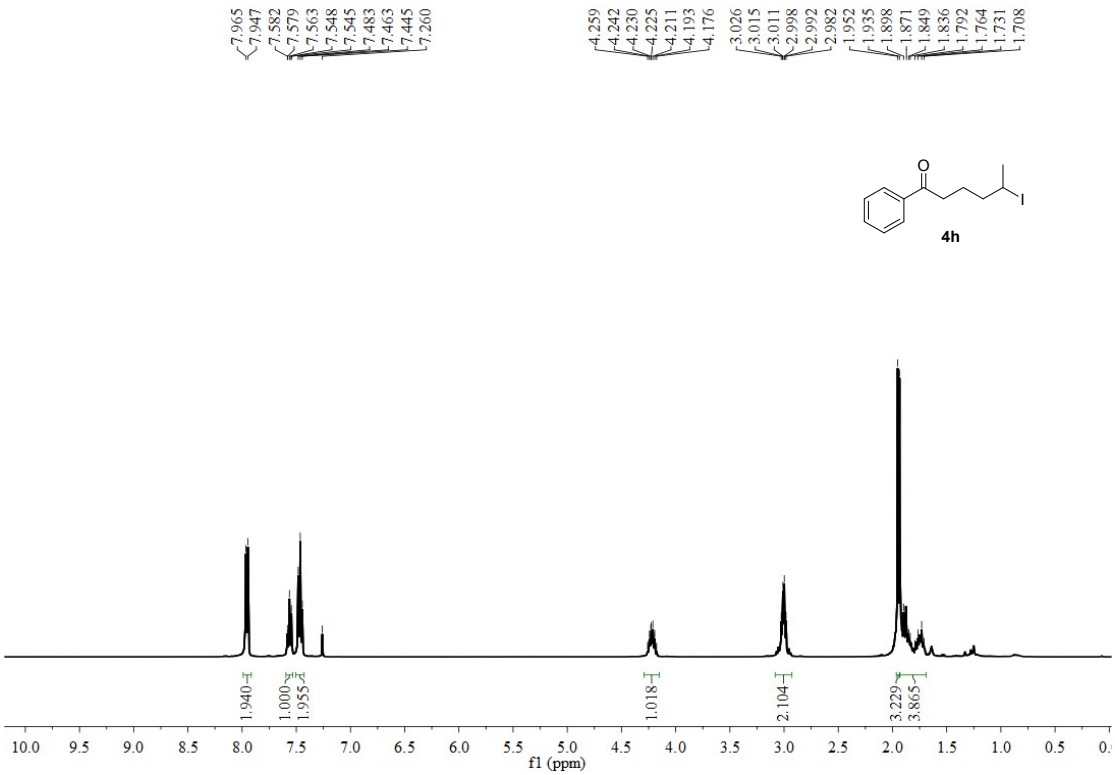


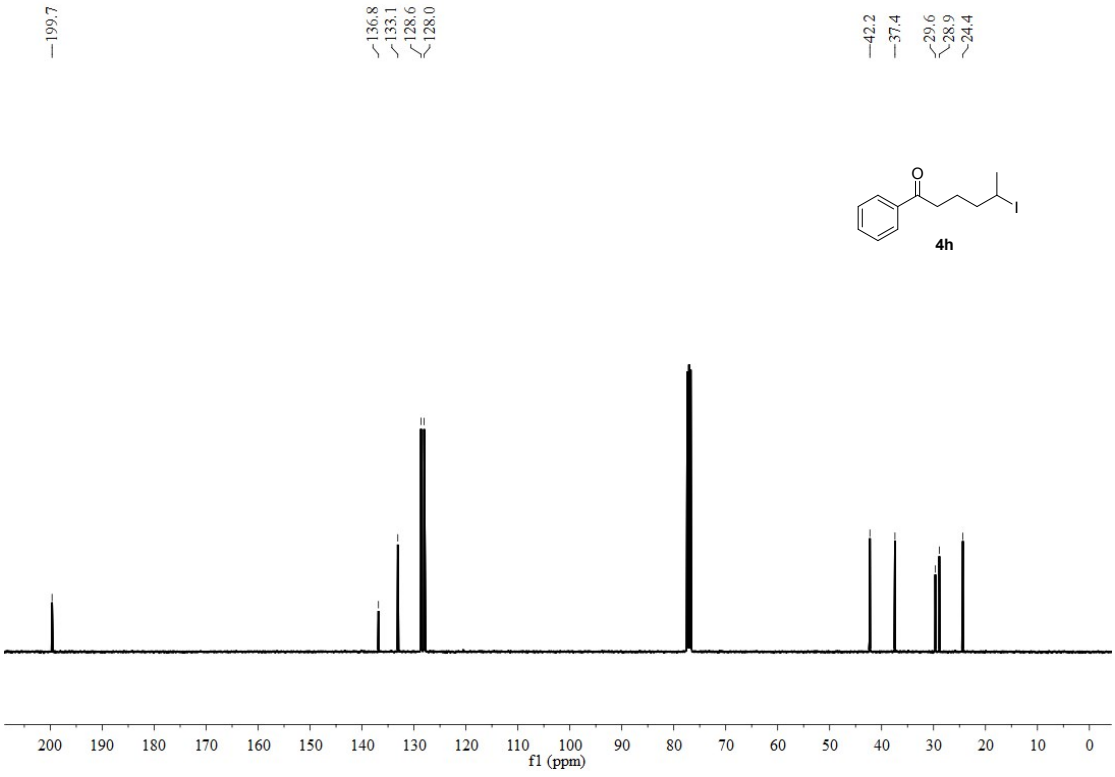
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4g**



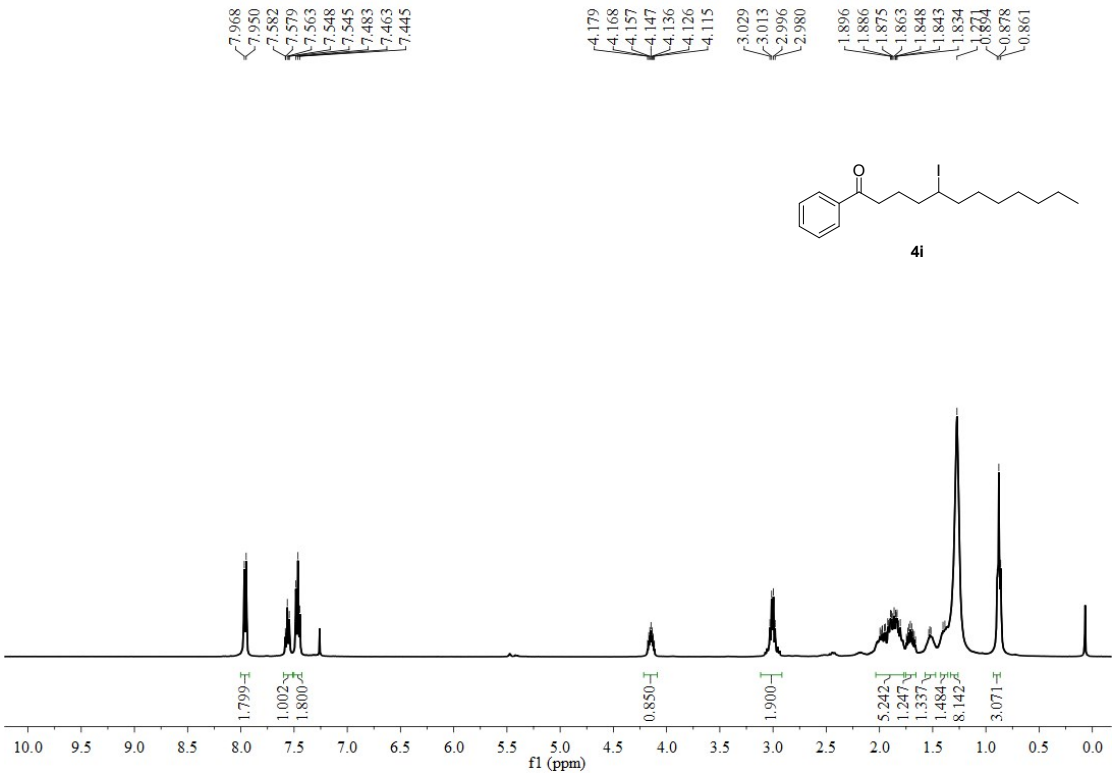


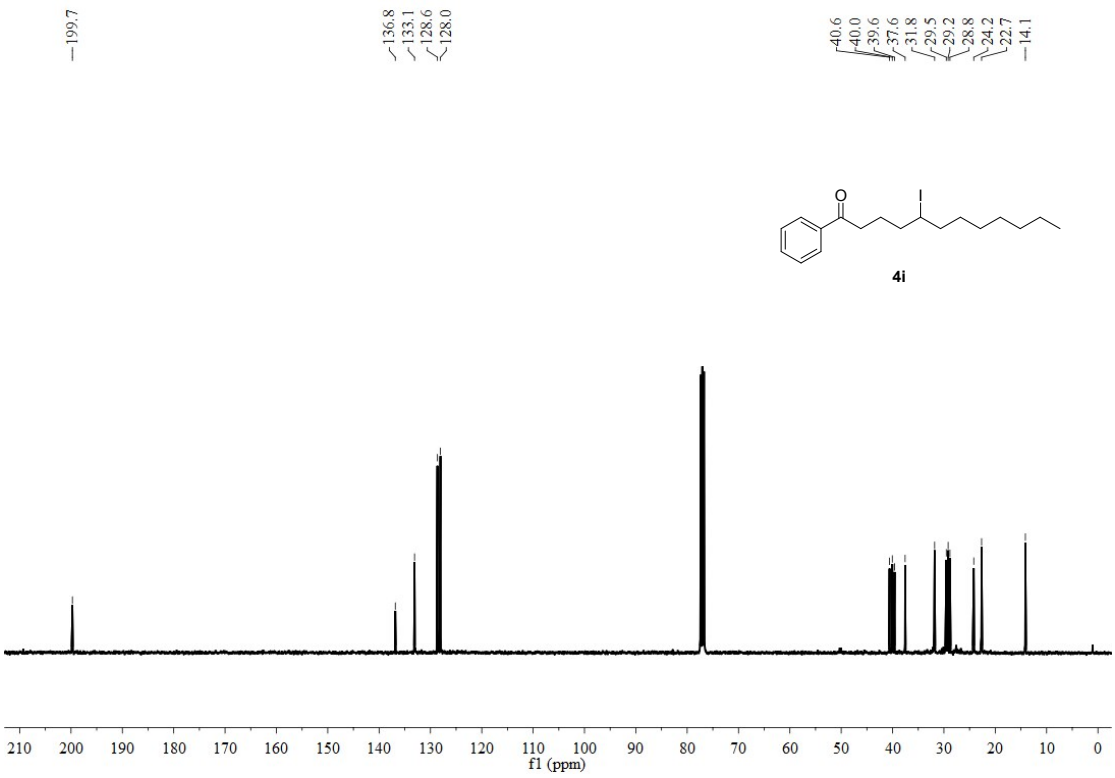
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4h**



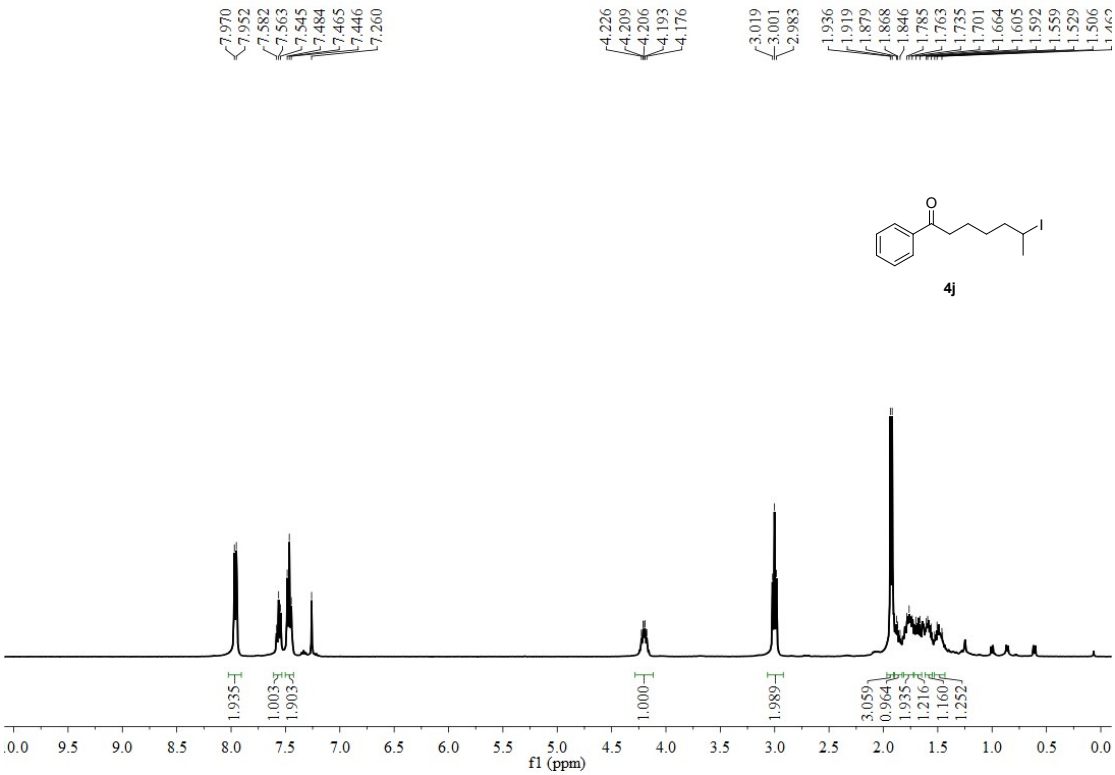


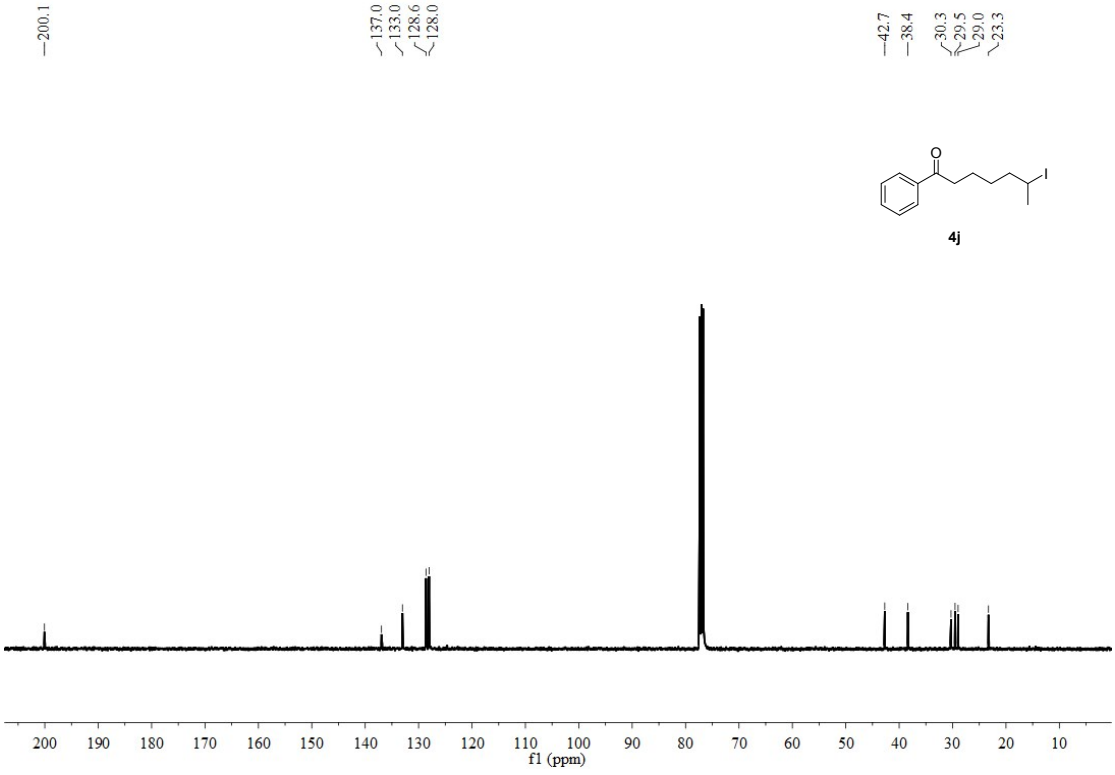
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4i**



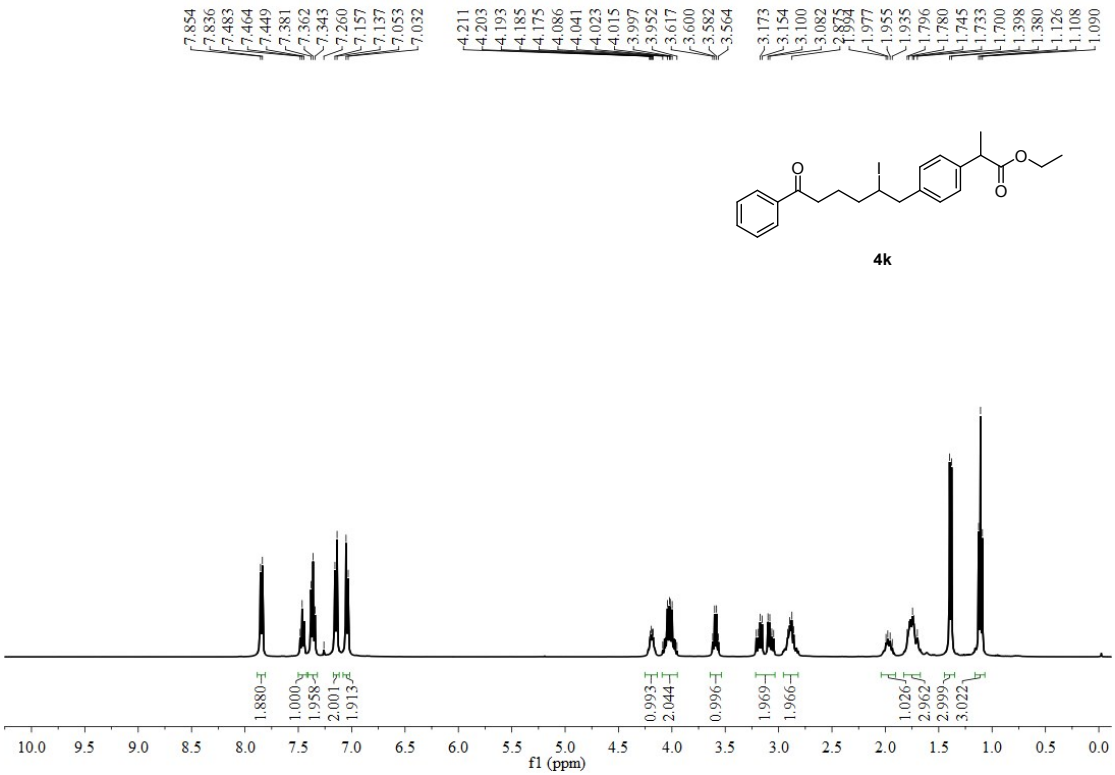


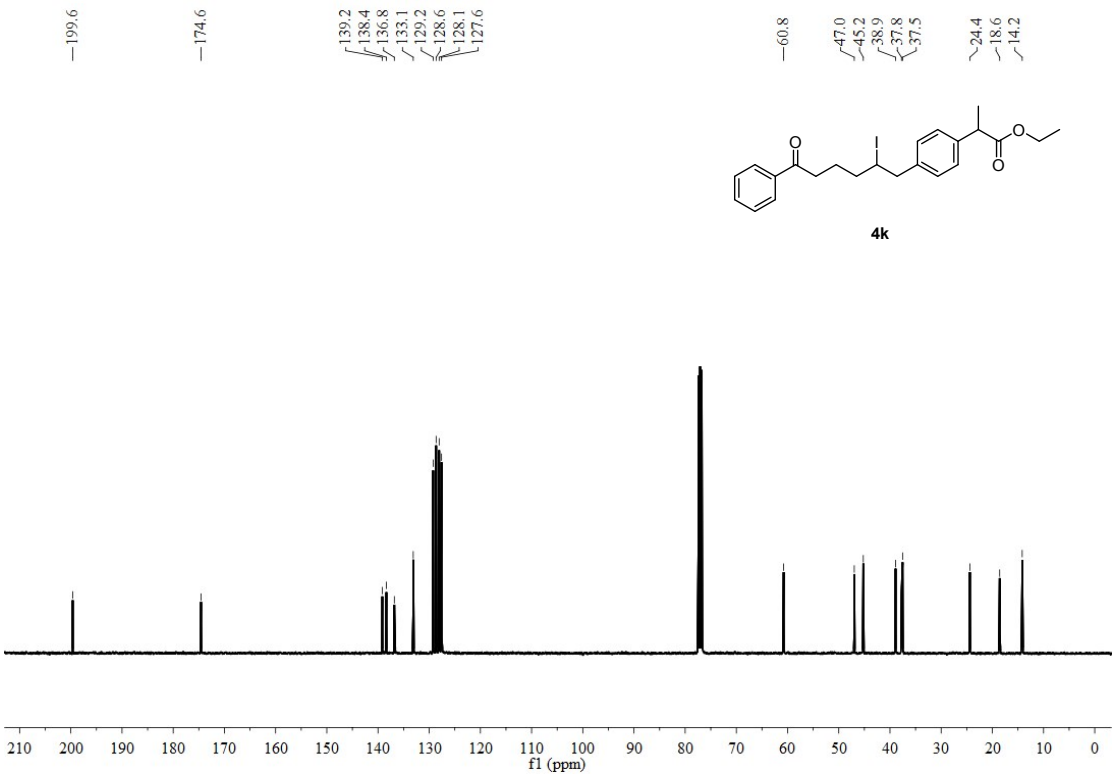
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4j**





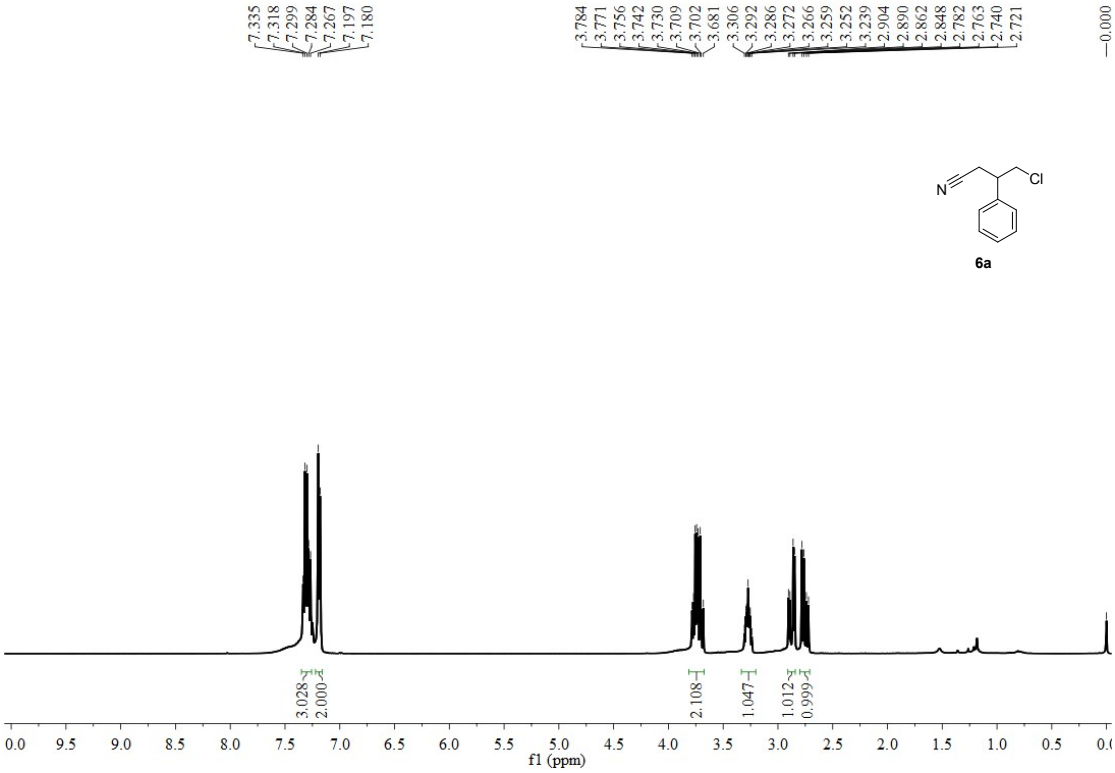
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **4k**

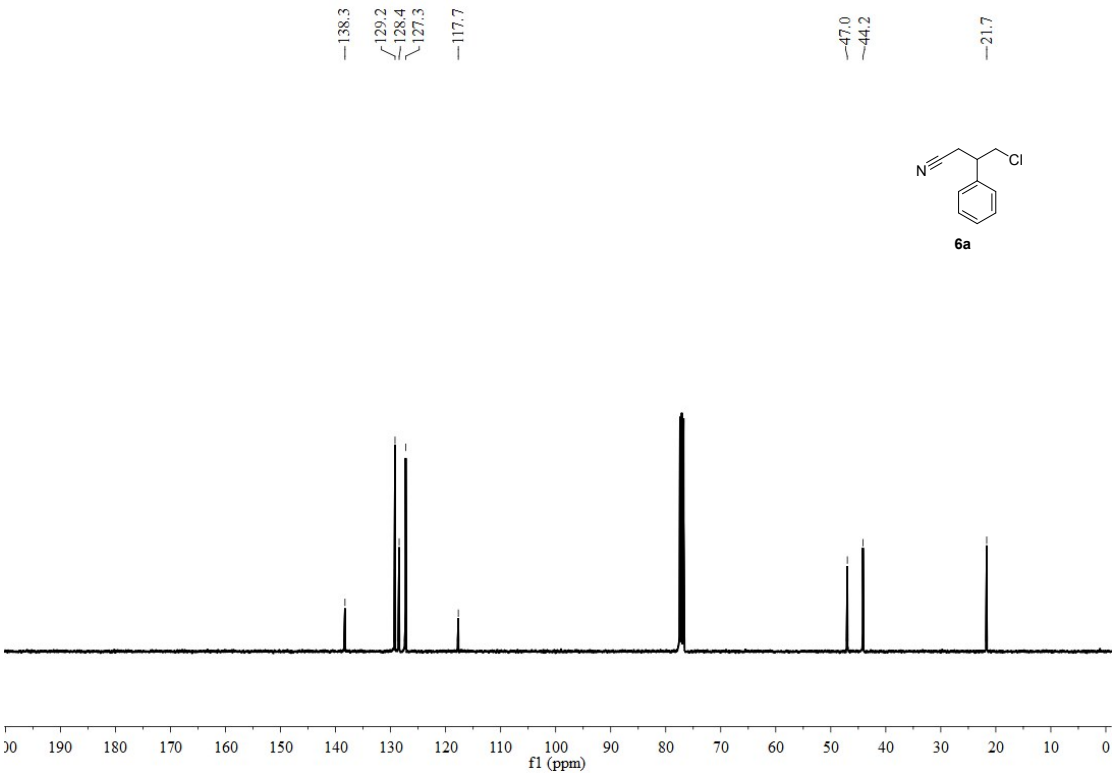




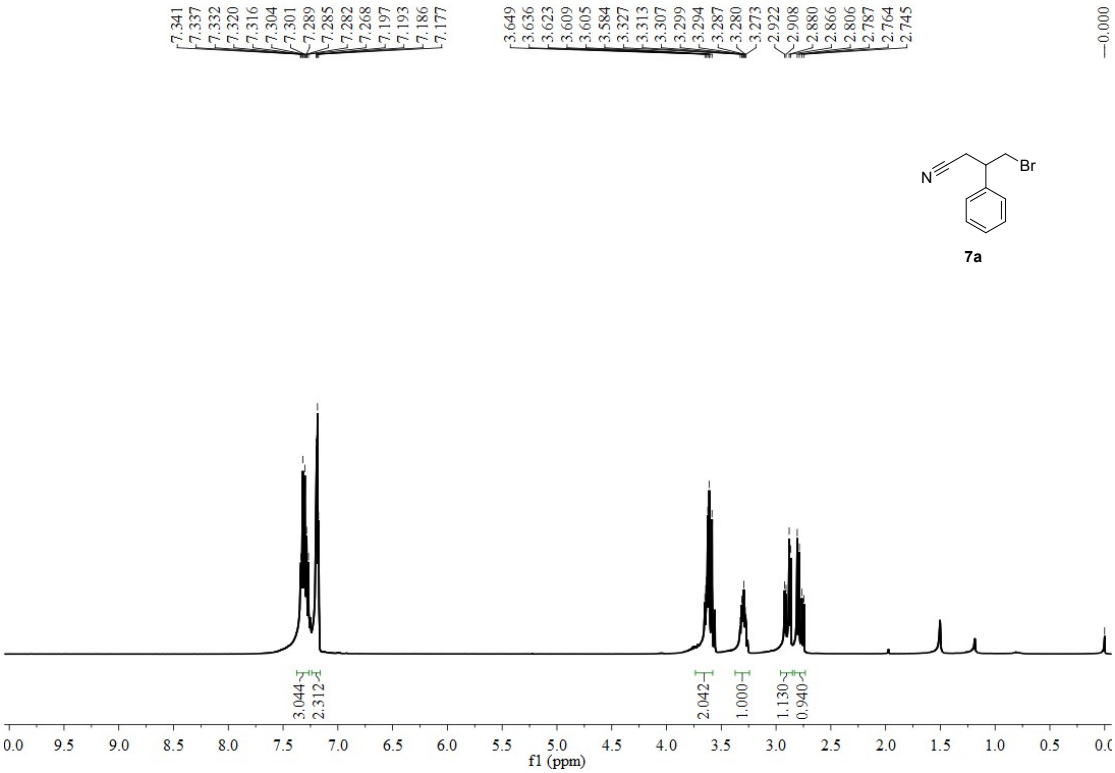
17. ^{1}H NMR and ^{13}C NMR Spectra of Products 6-8

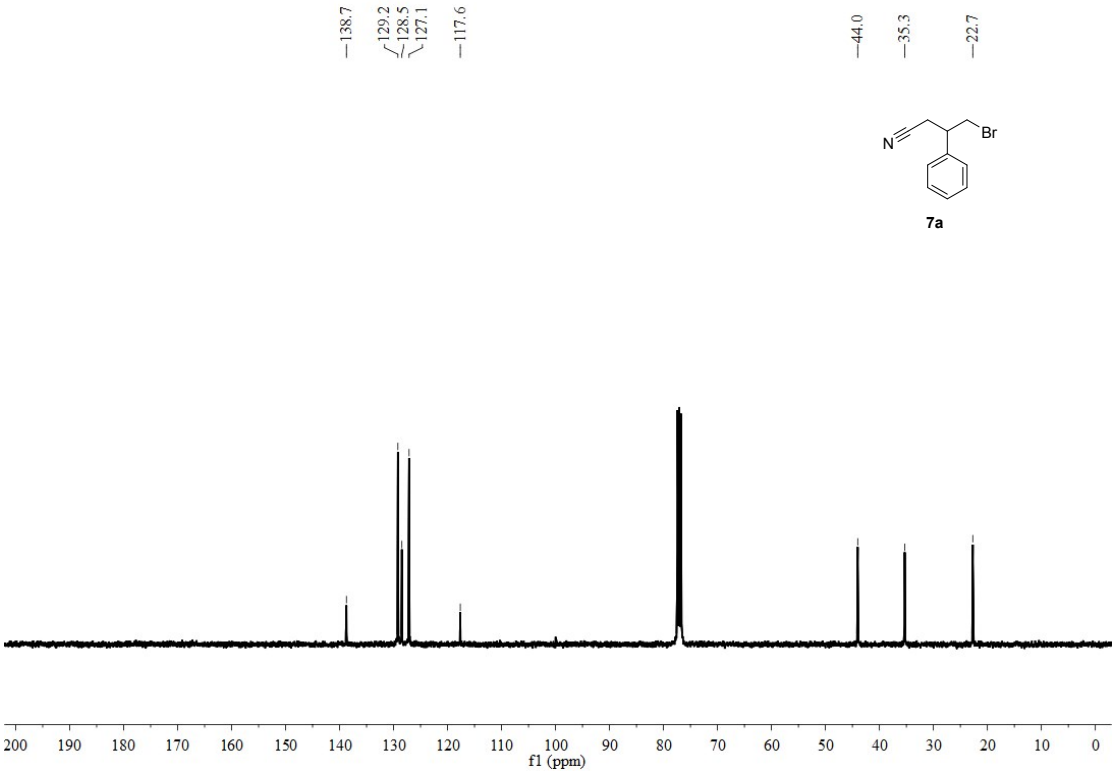
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **6a**



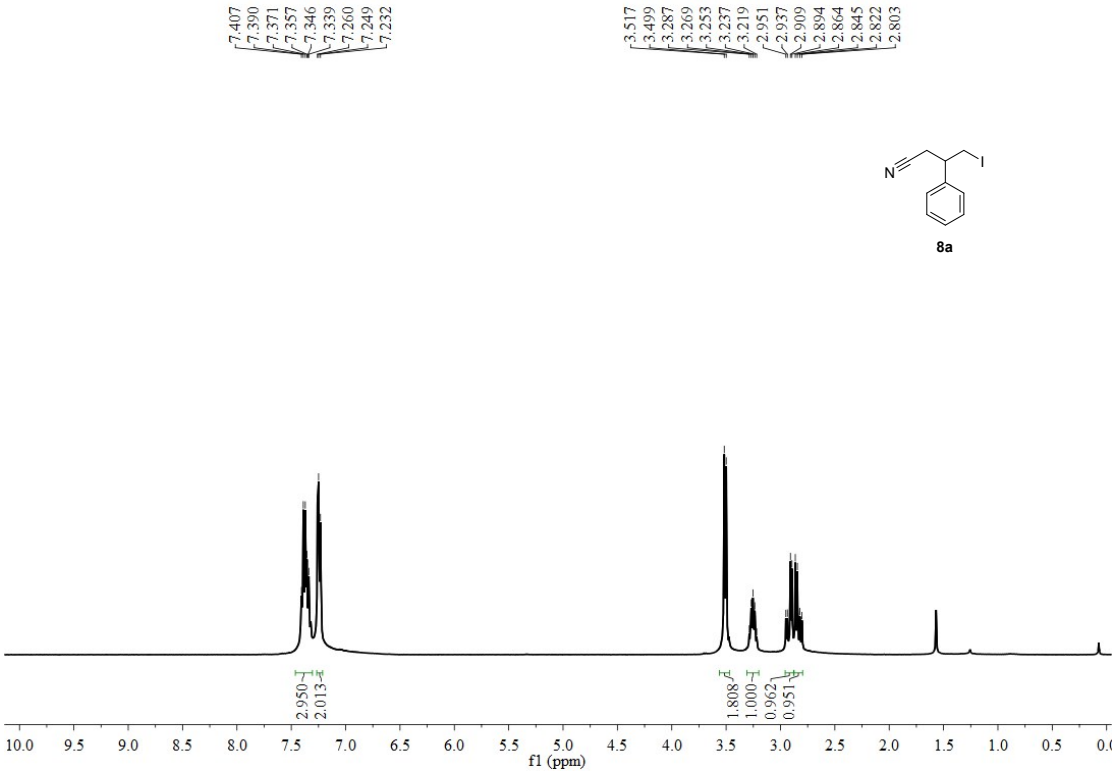


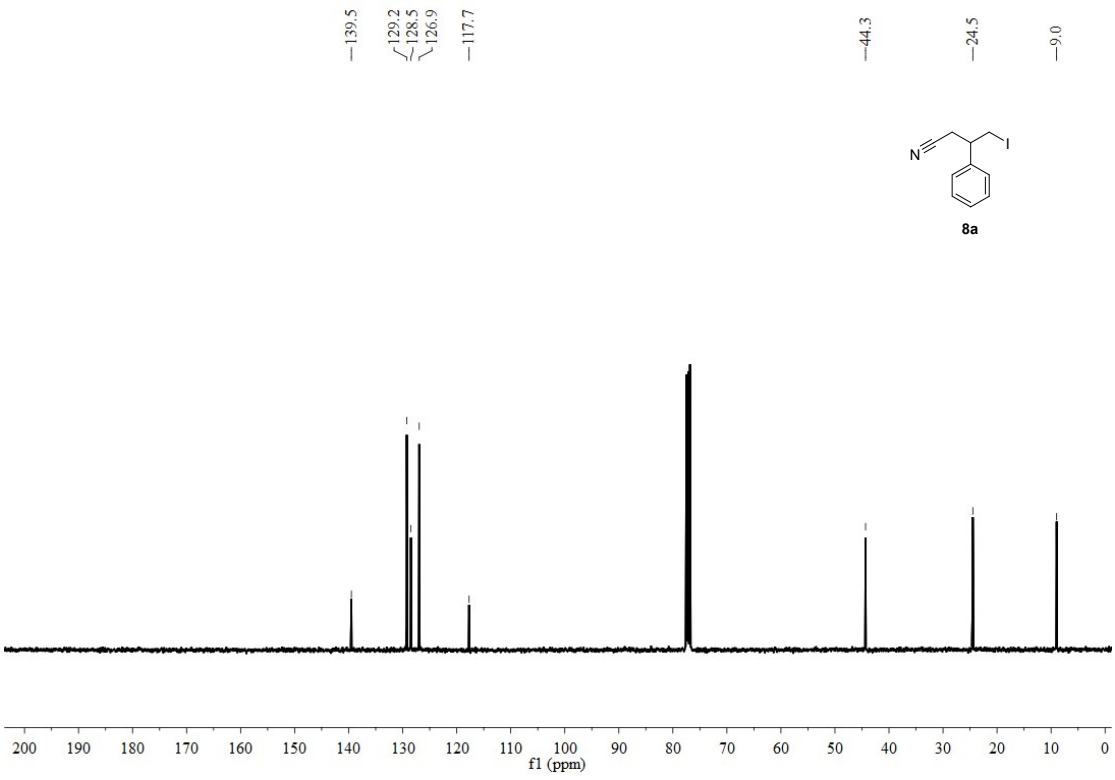
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **7a**



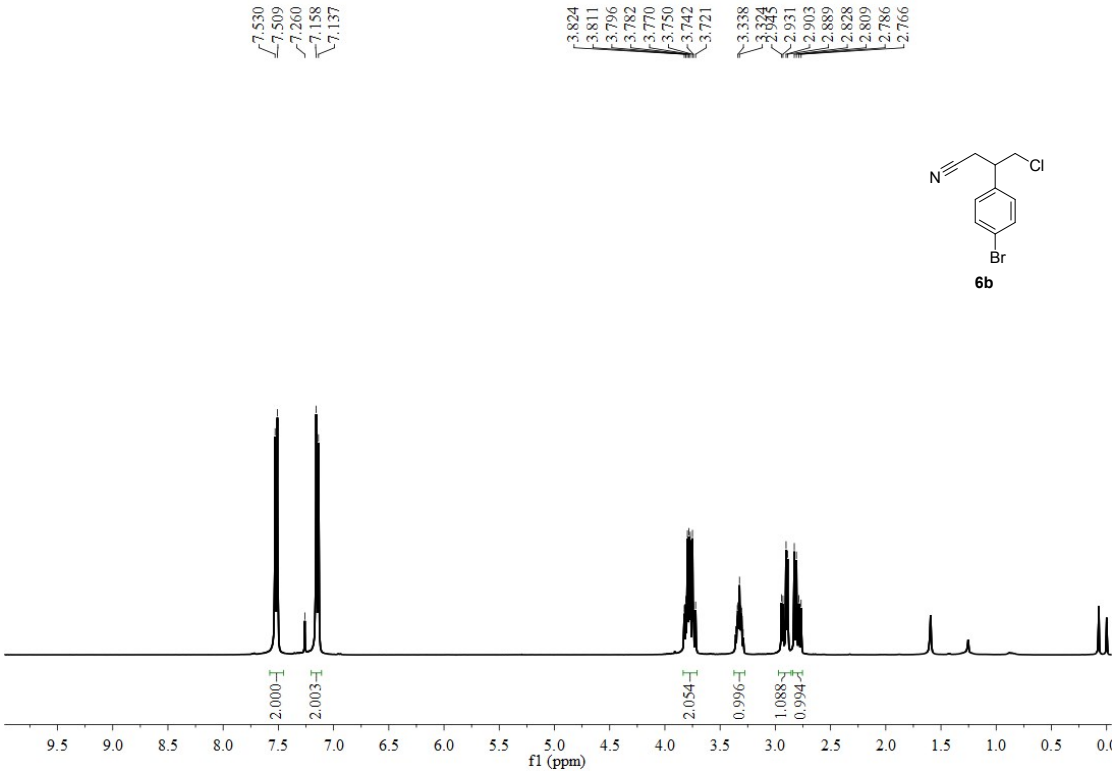


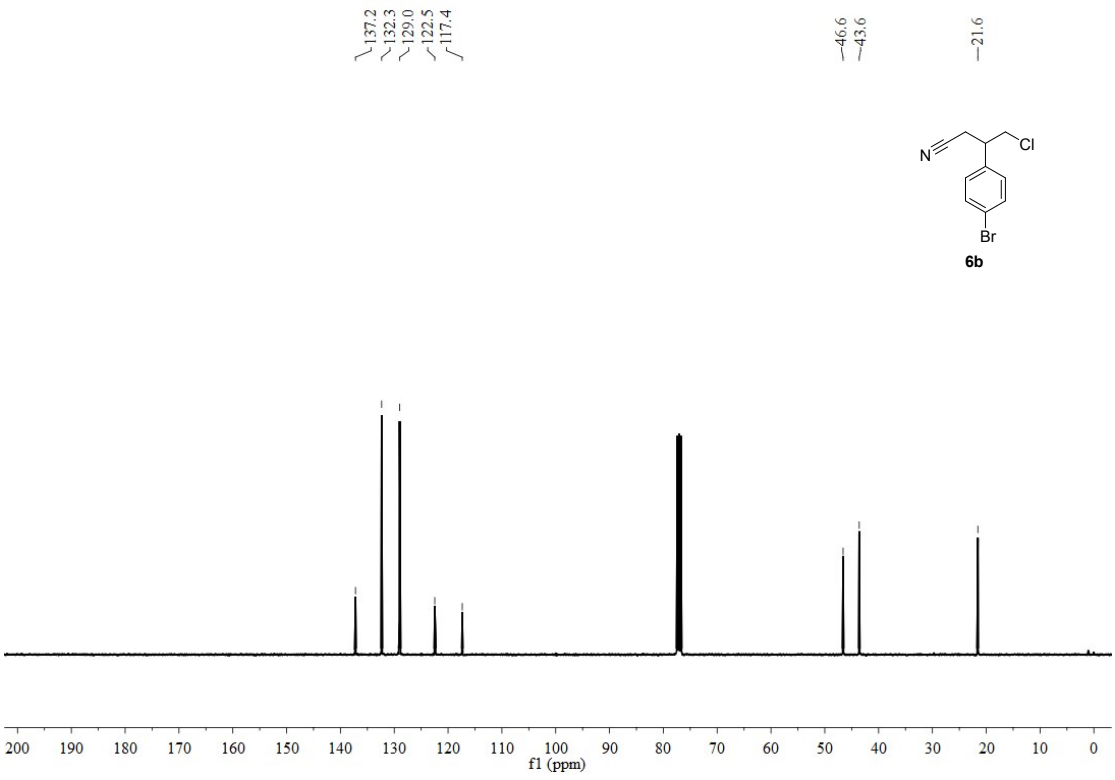
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **8a**



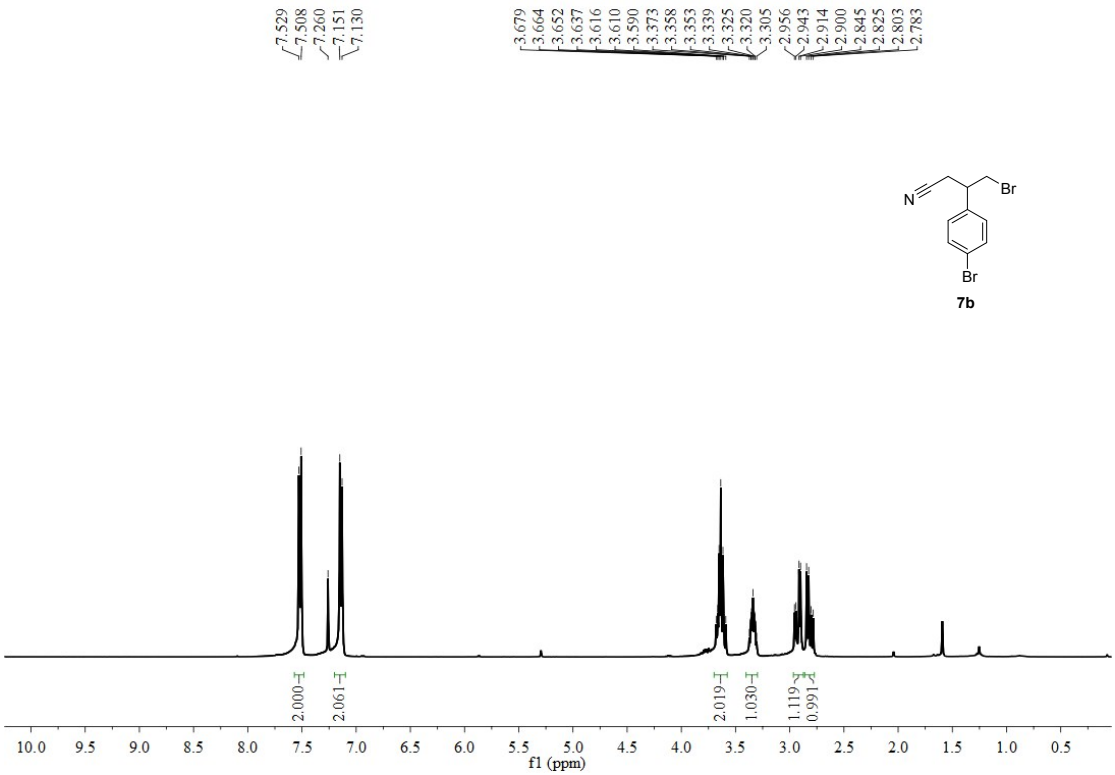


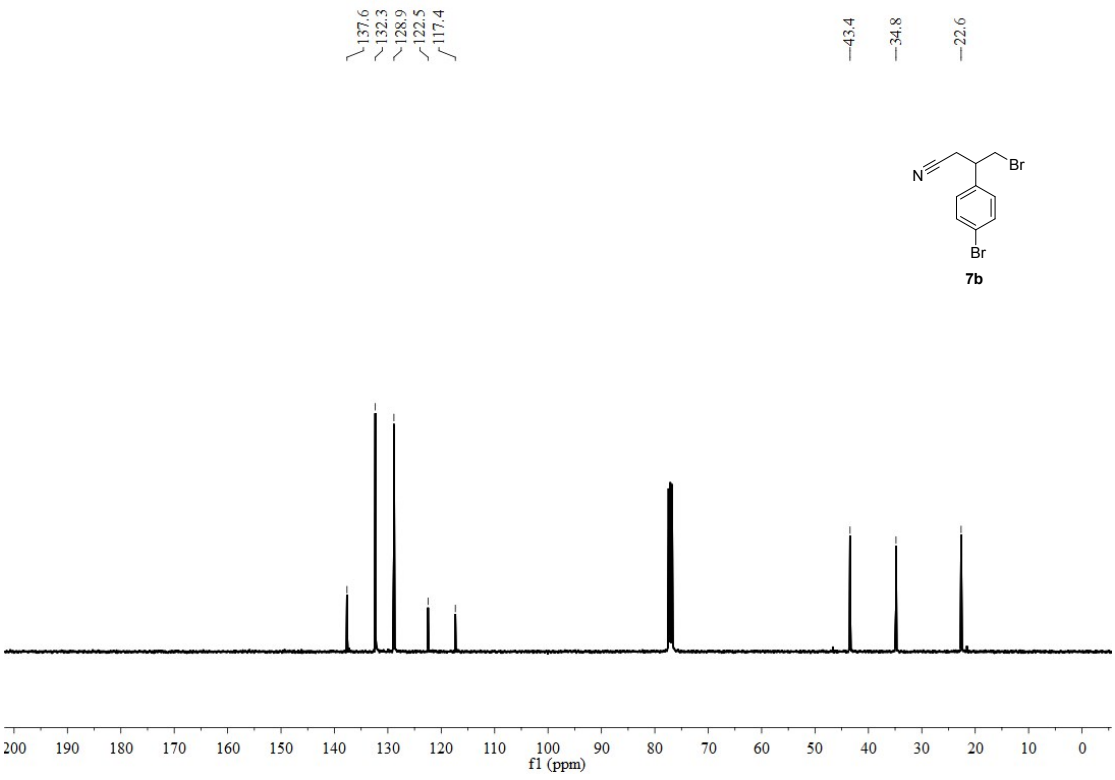
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **6b**



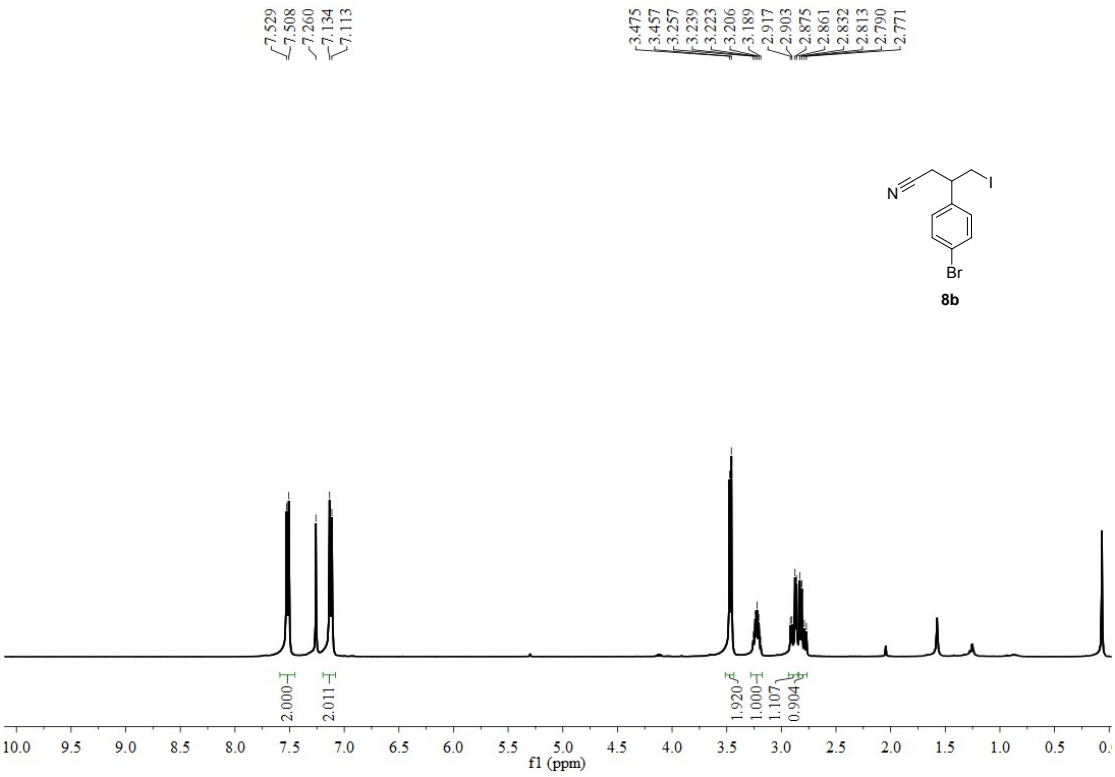


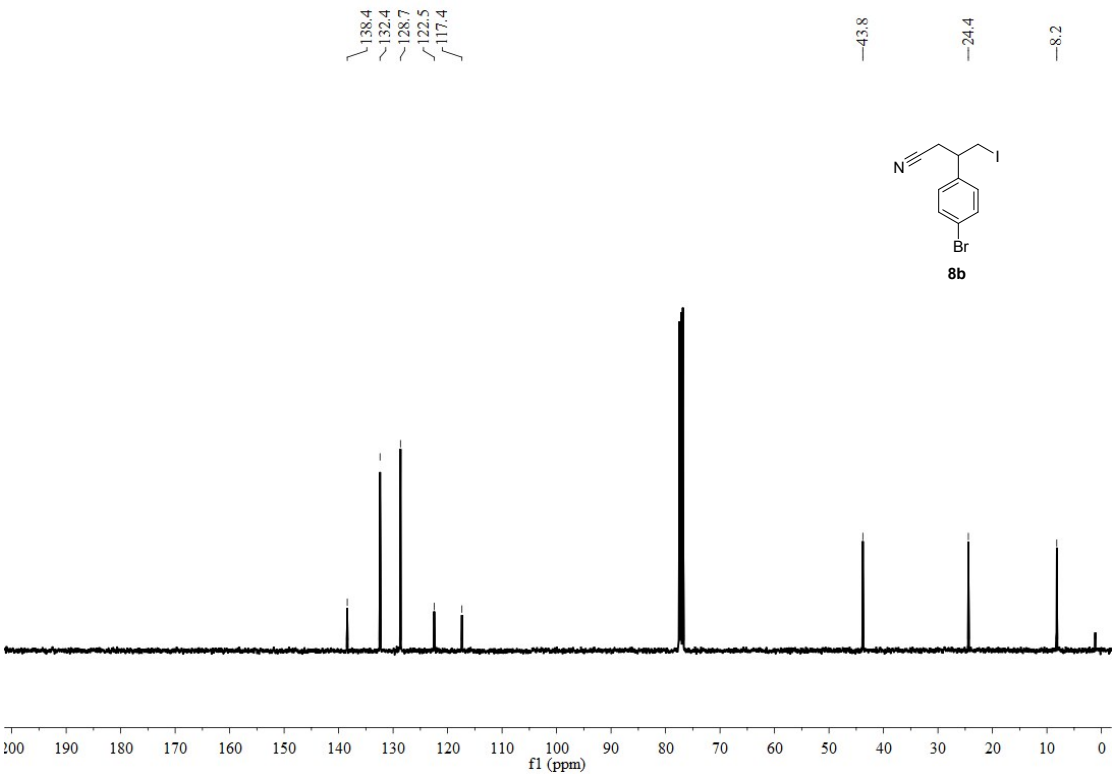
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **7b**



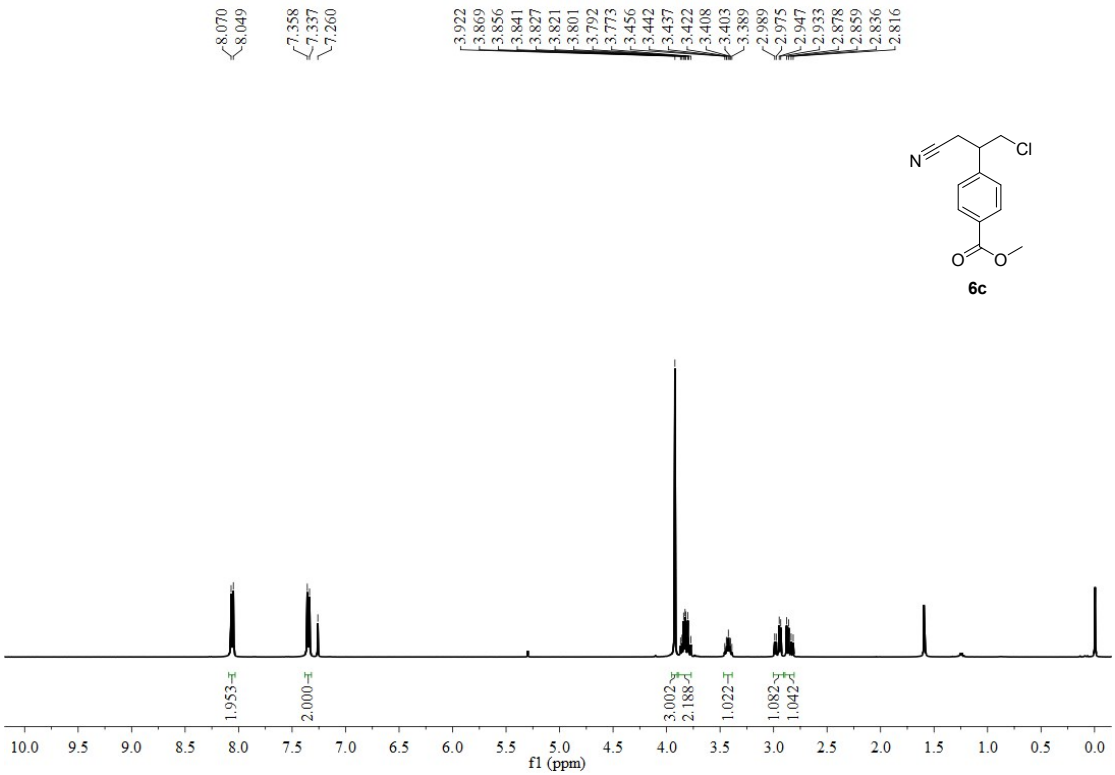


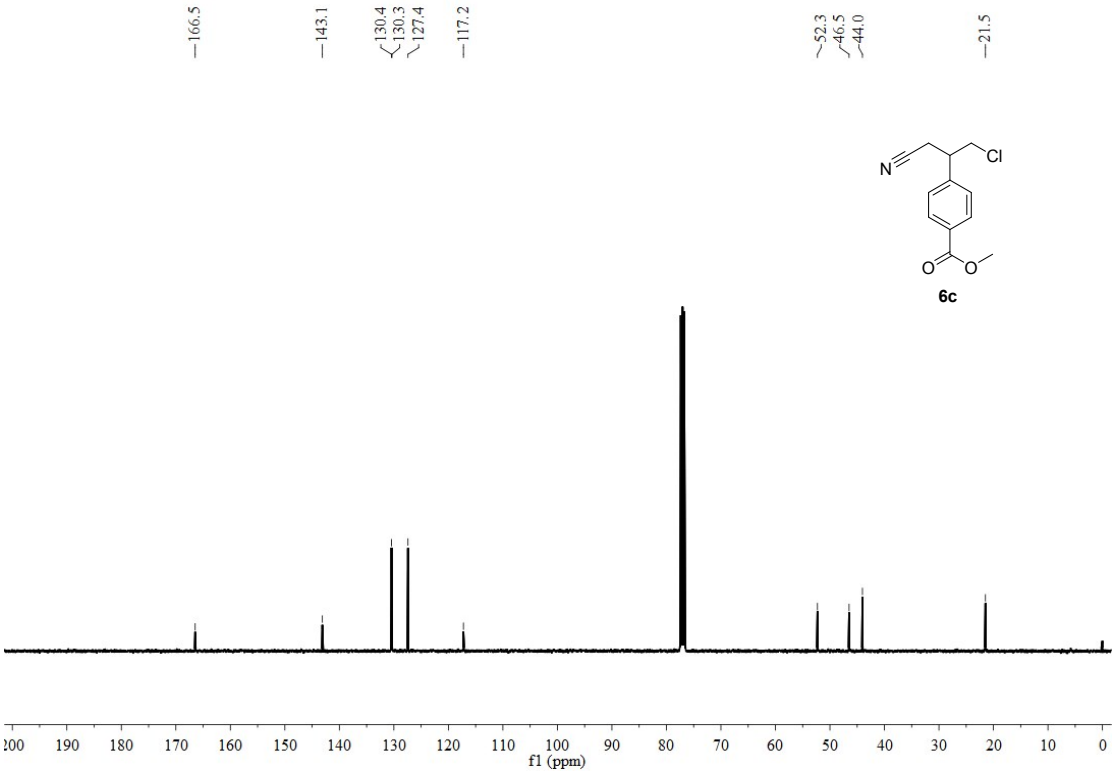
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **8b**



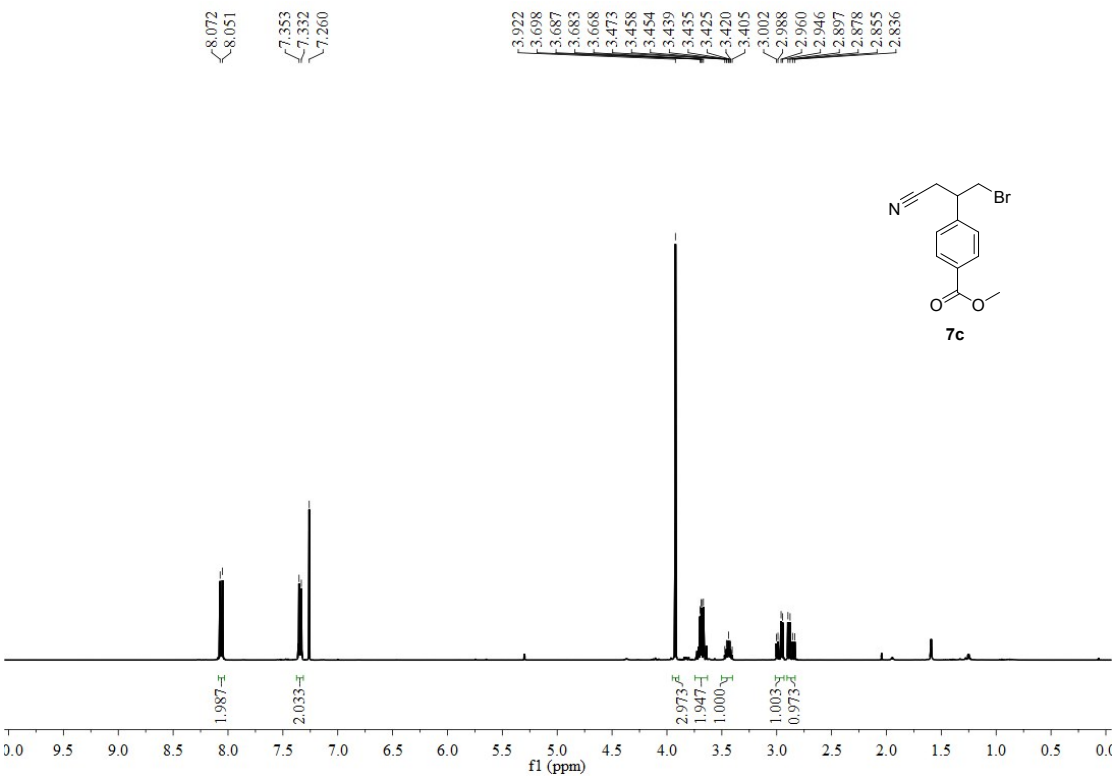


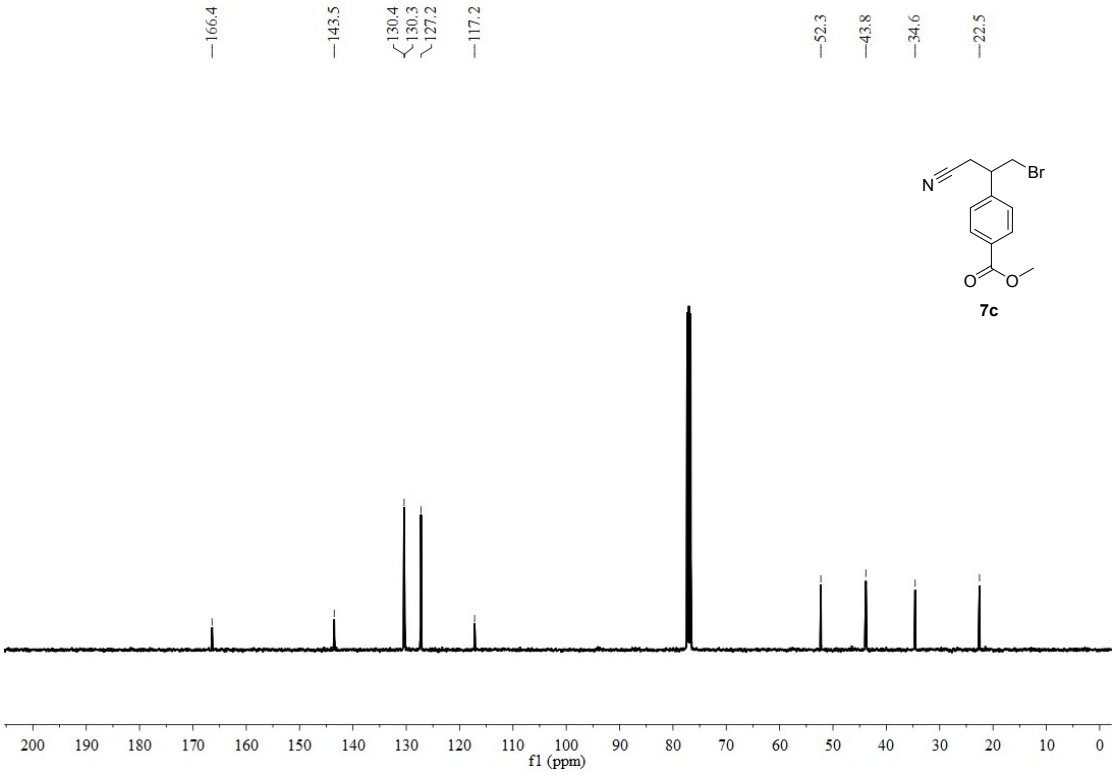
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **6c**



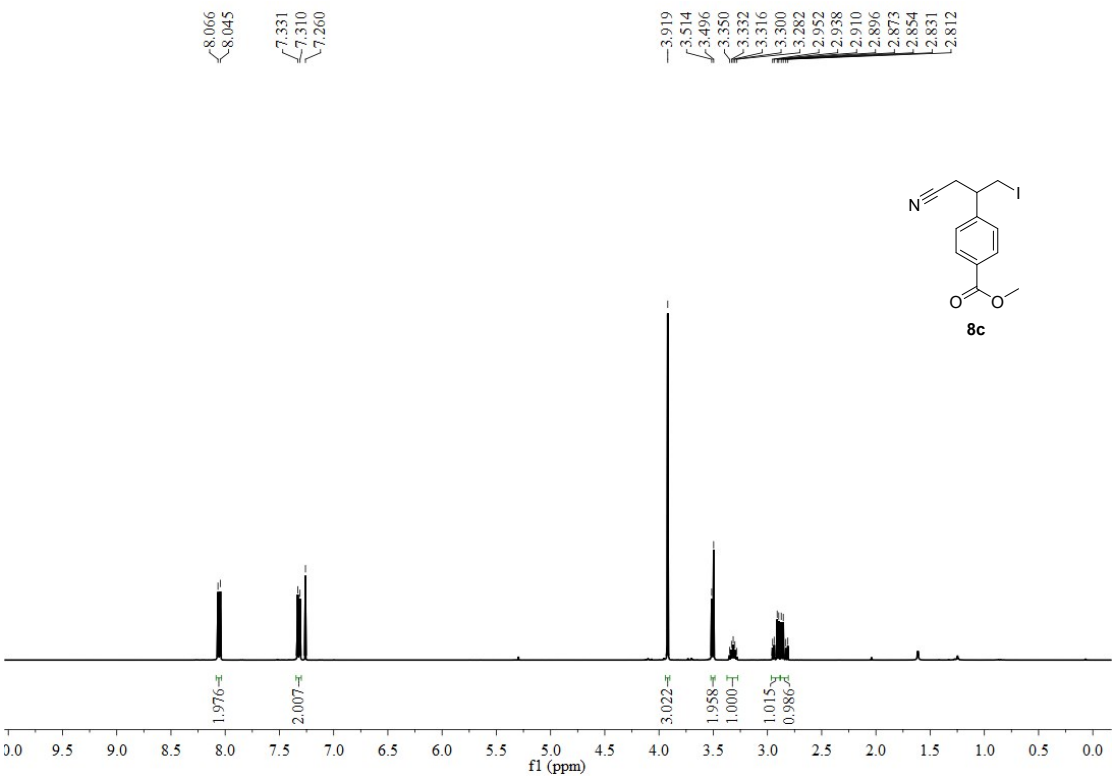


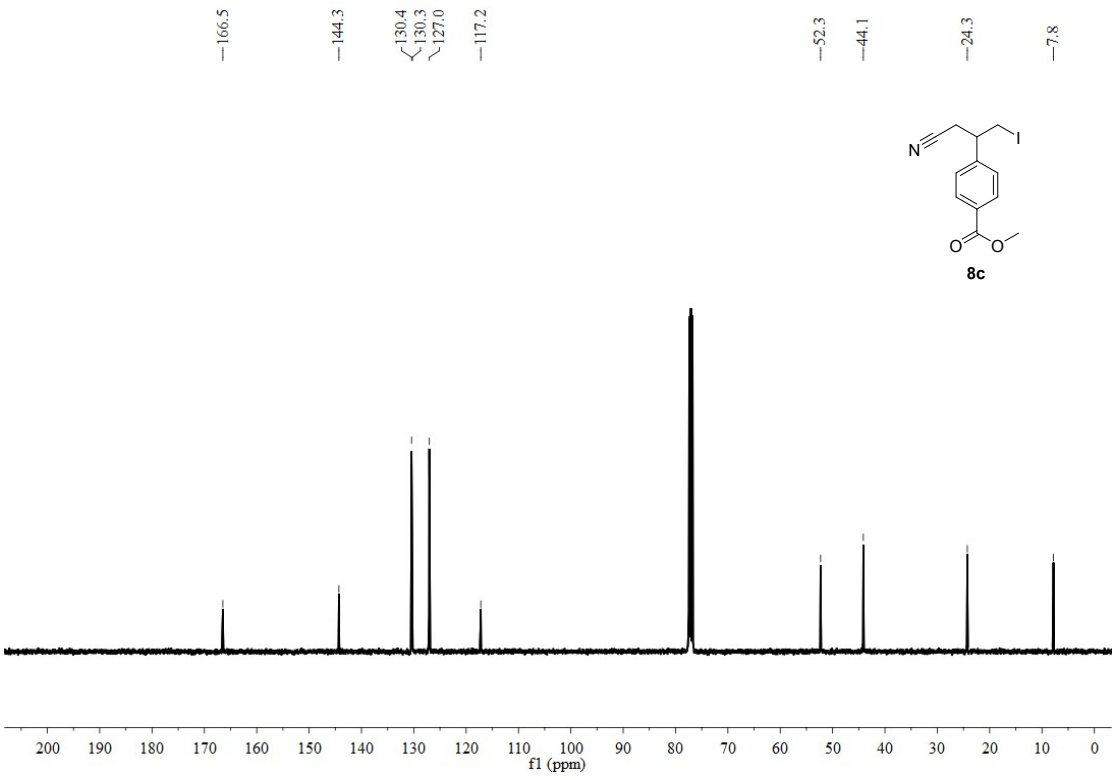
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **7c**



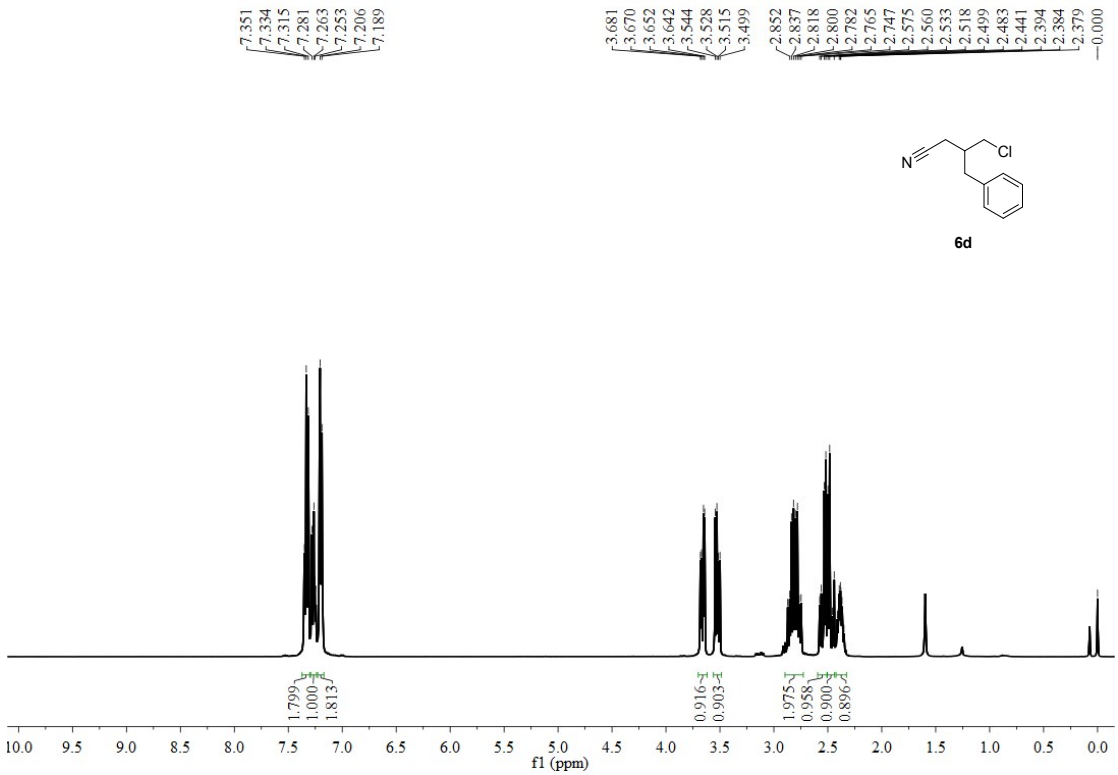


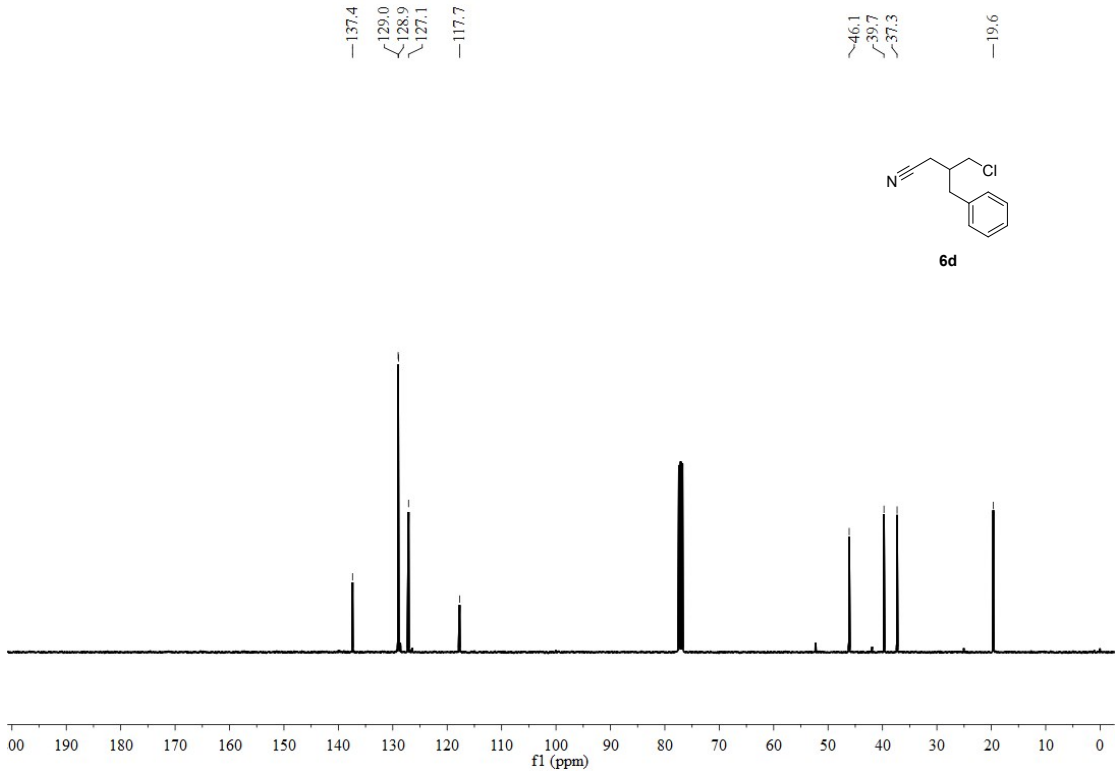
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **8c**



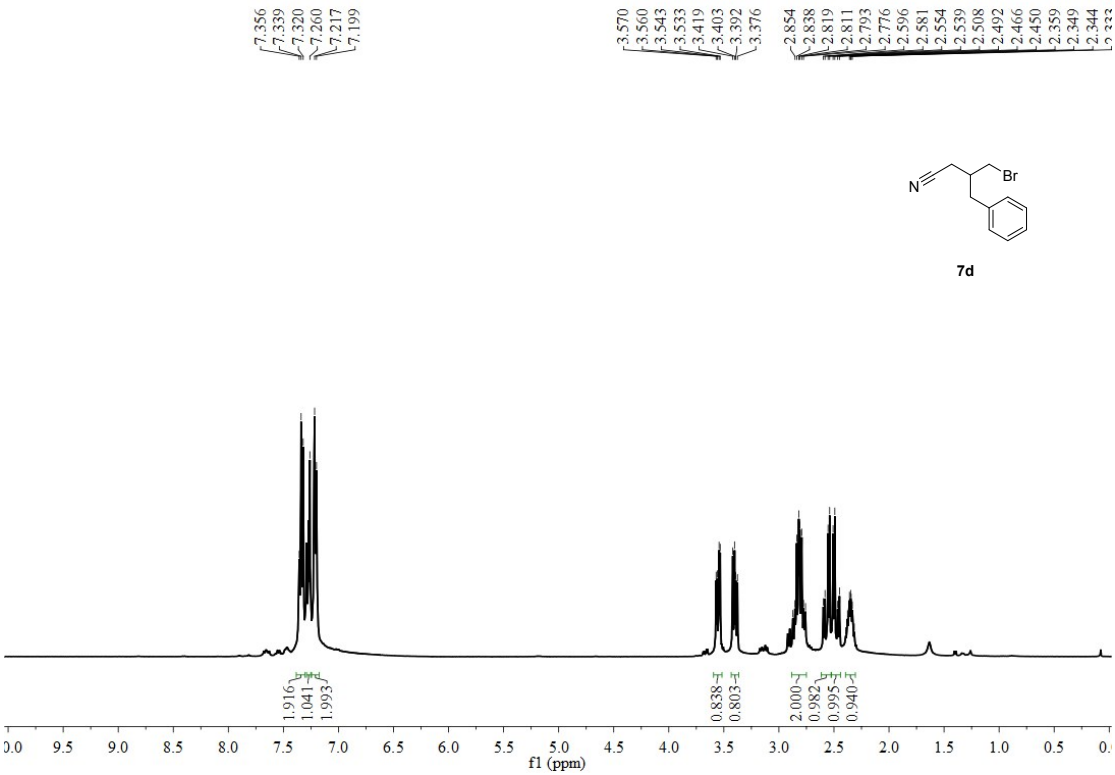


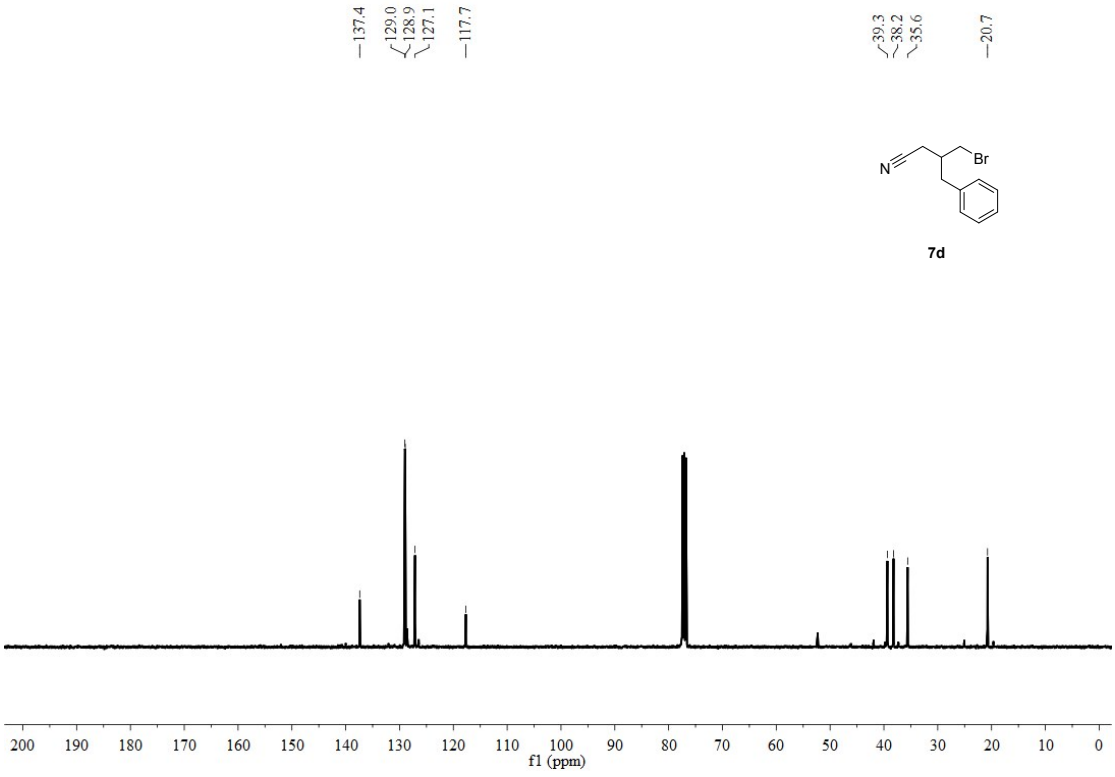
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **6d**



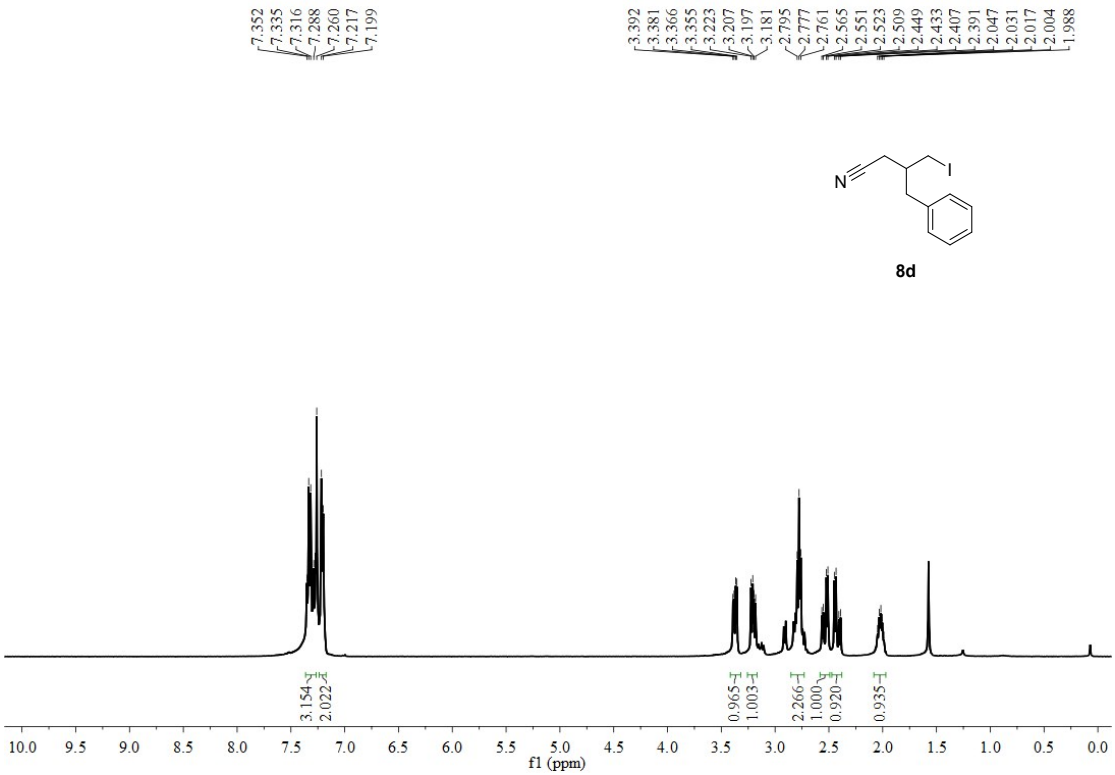


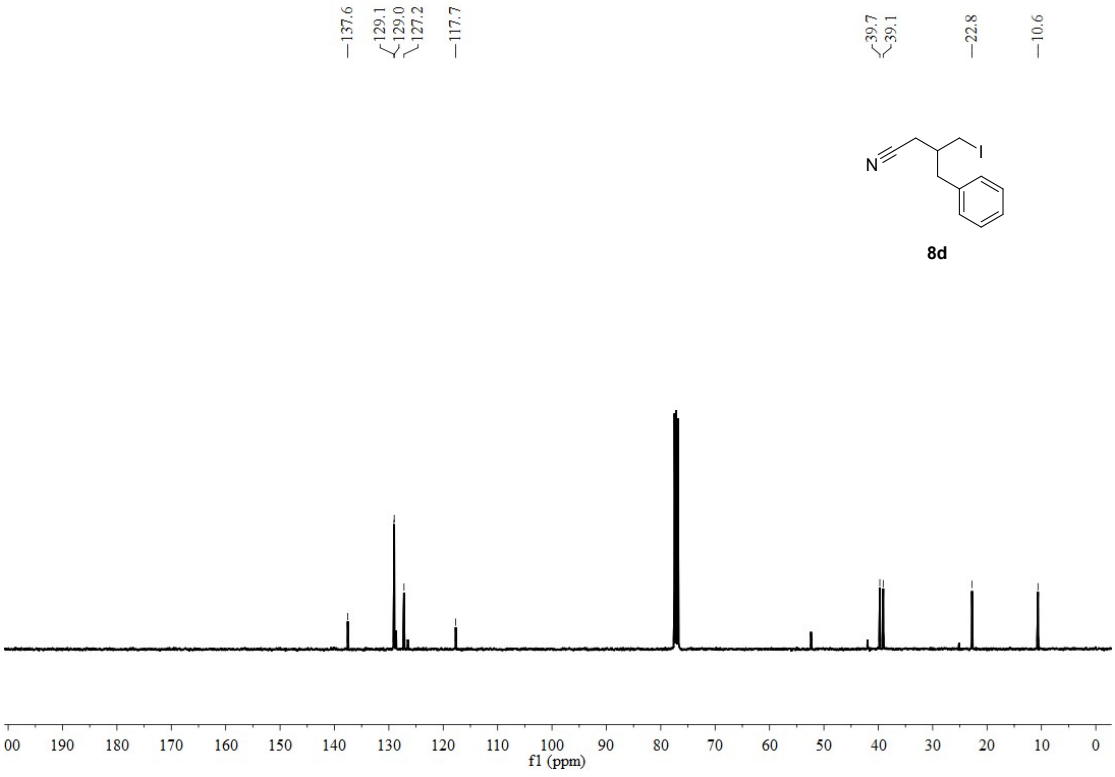
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **7d**



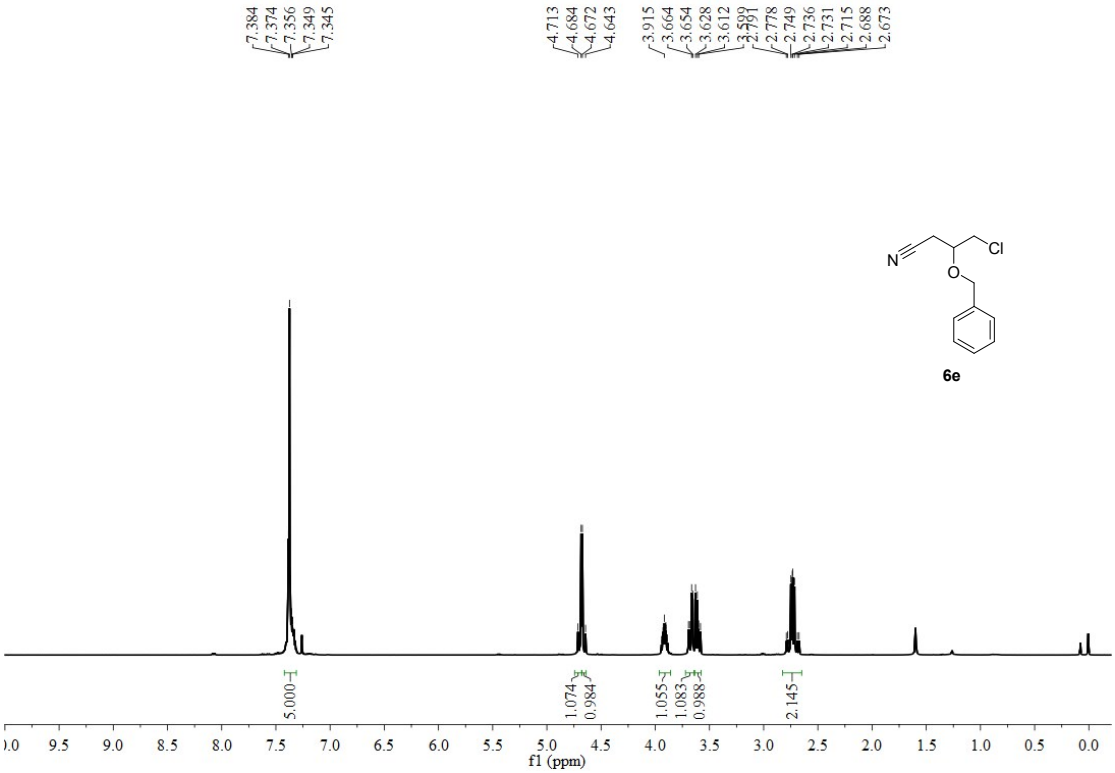


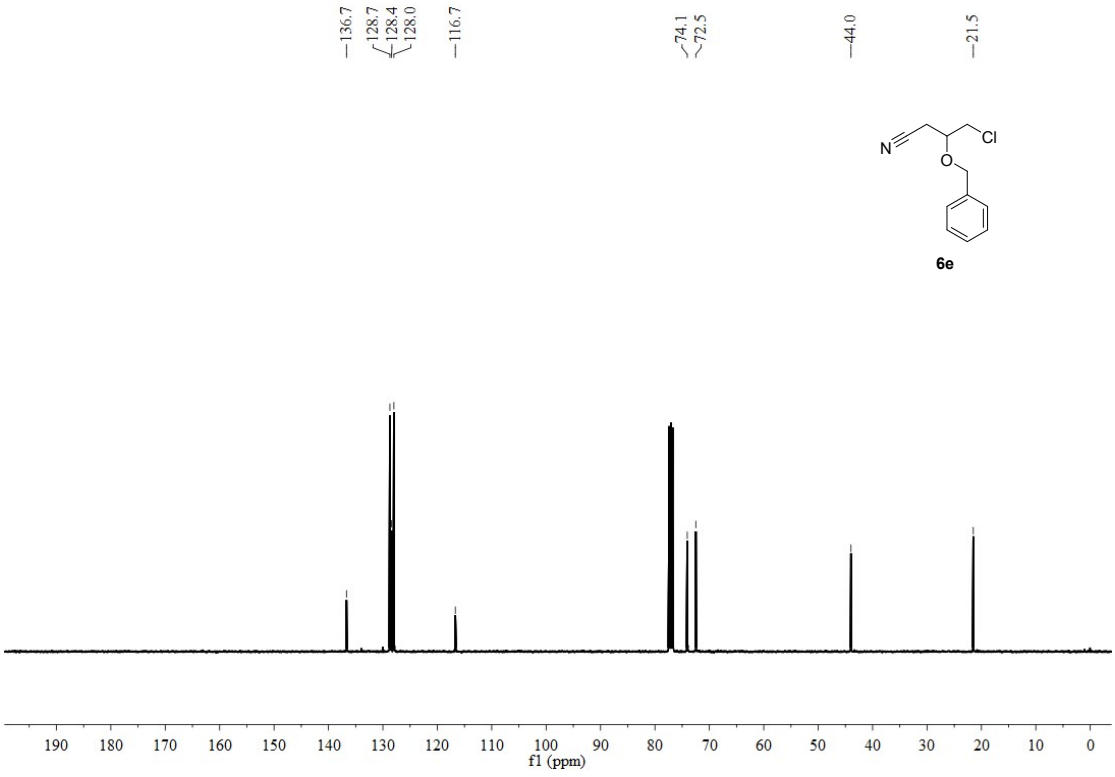
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **8d**



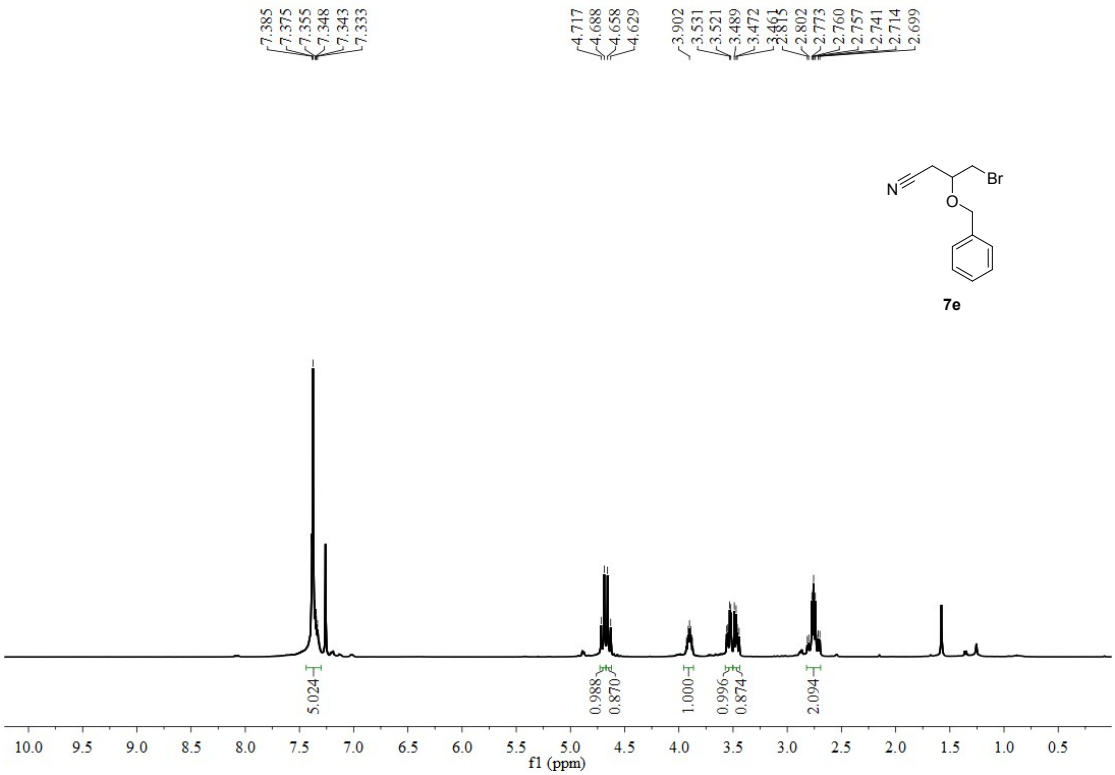


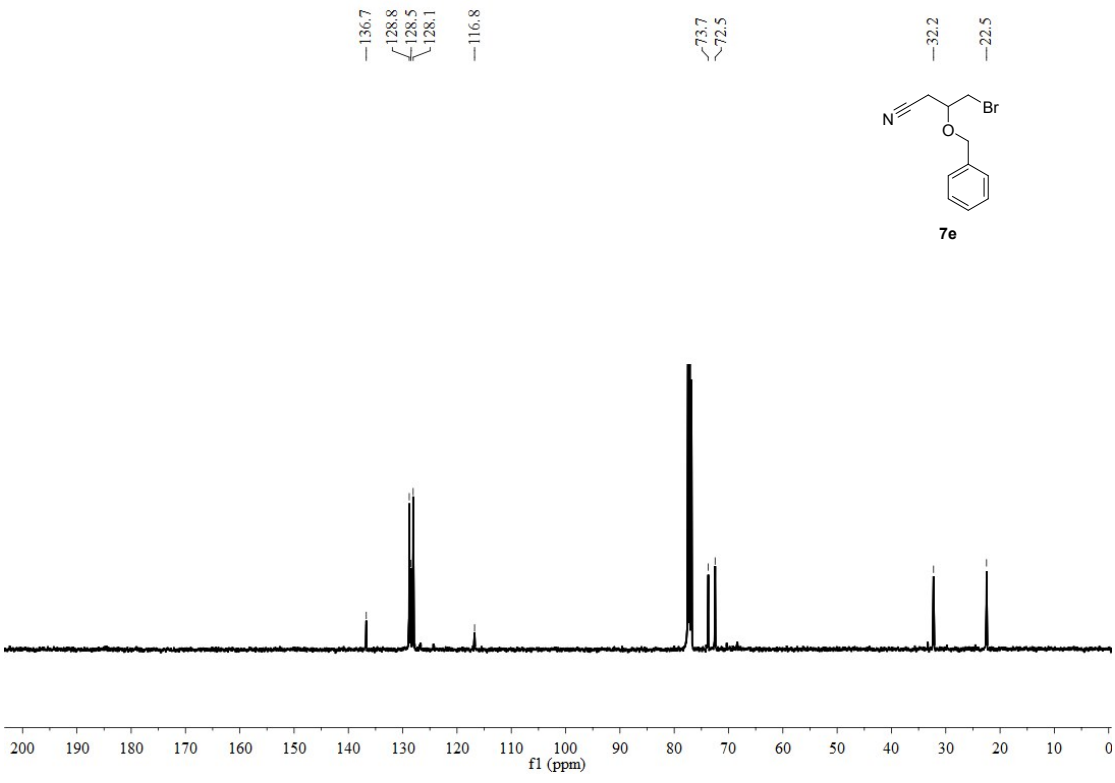
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **6e**



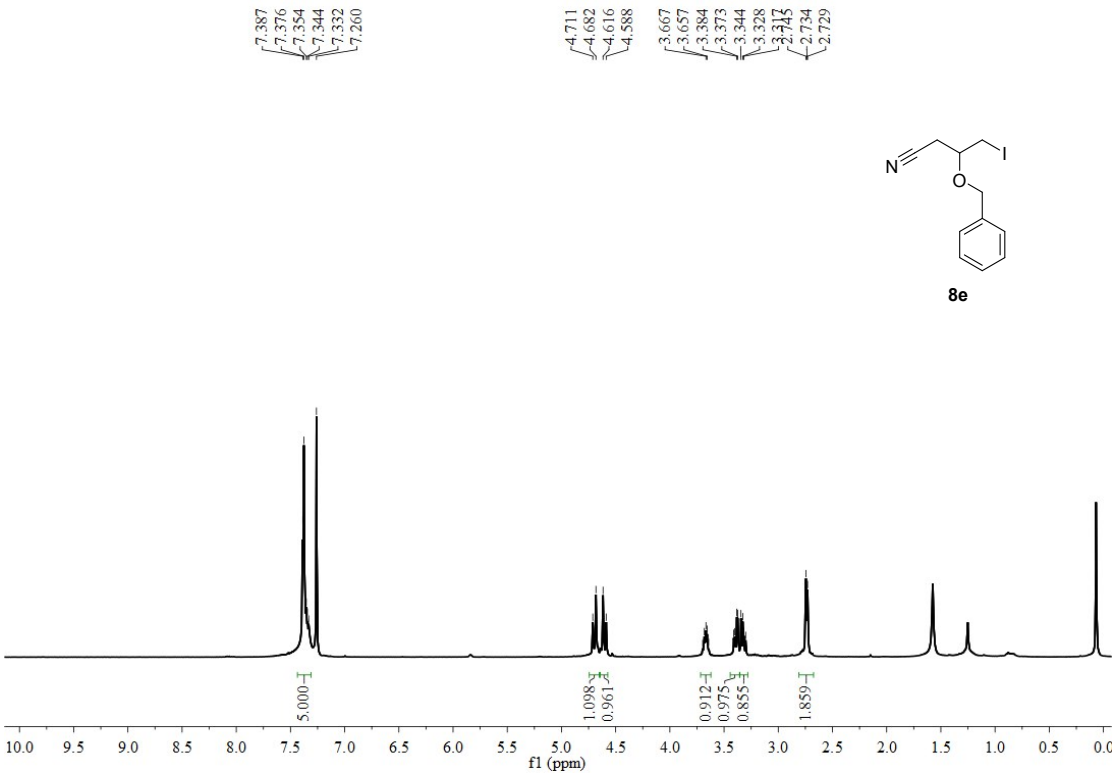


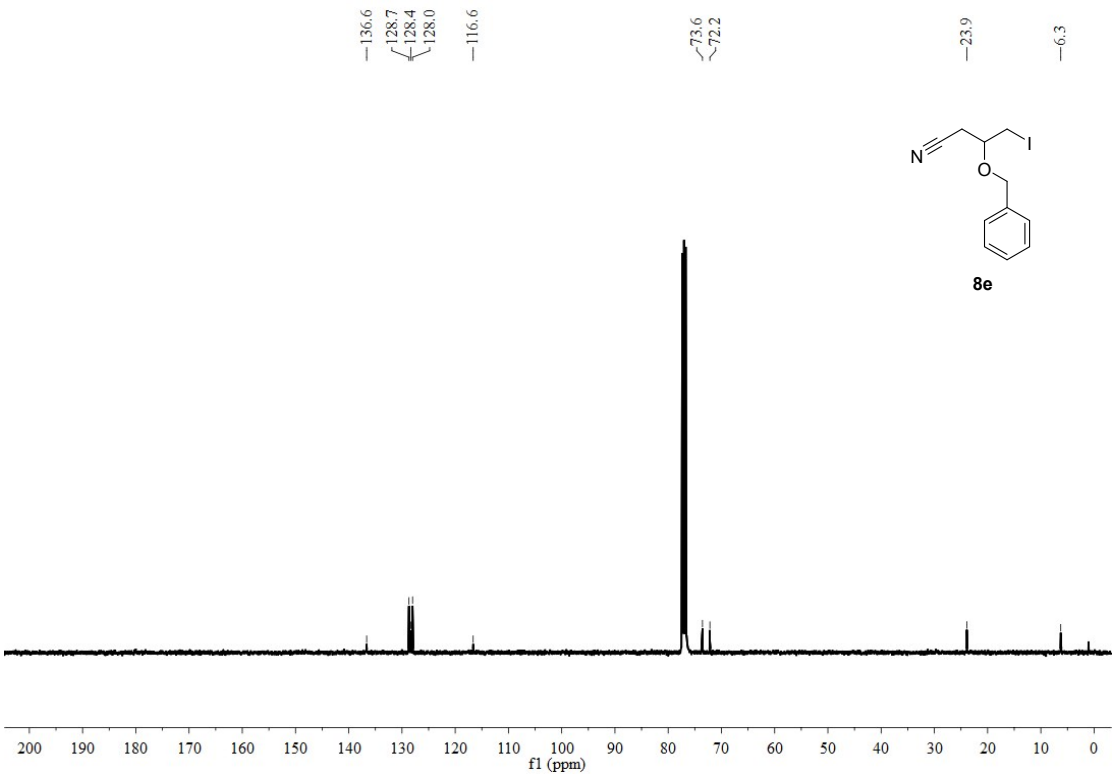
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **7e**



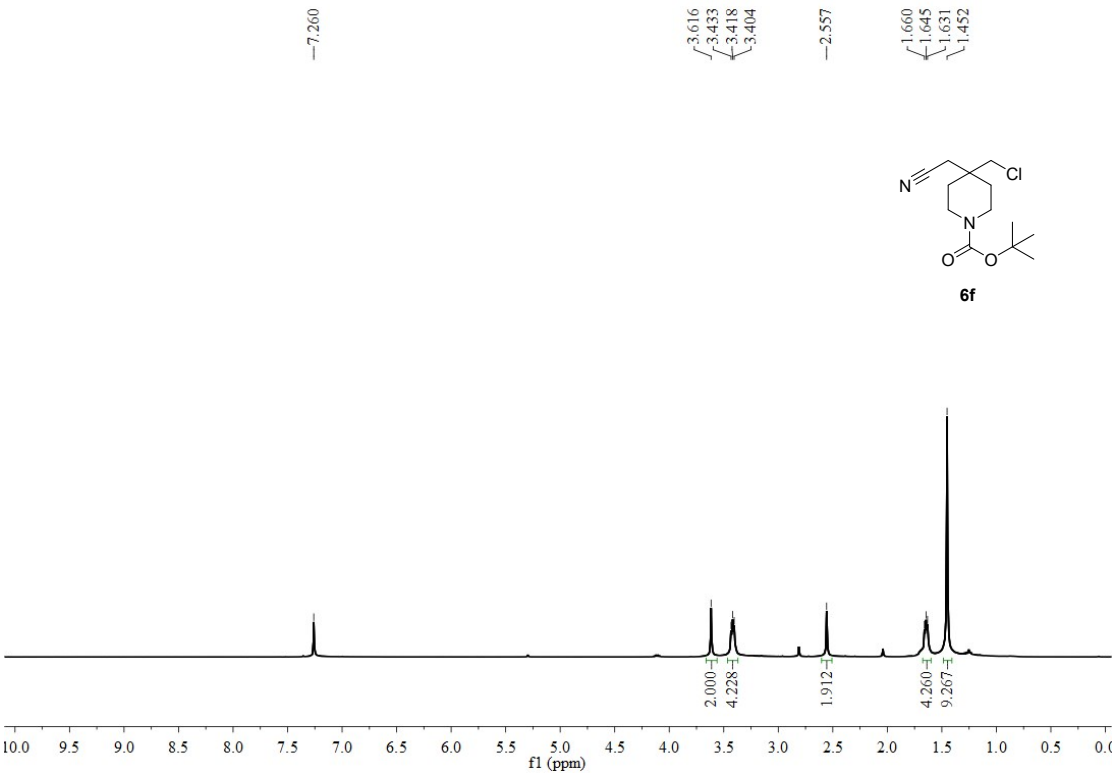


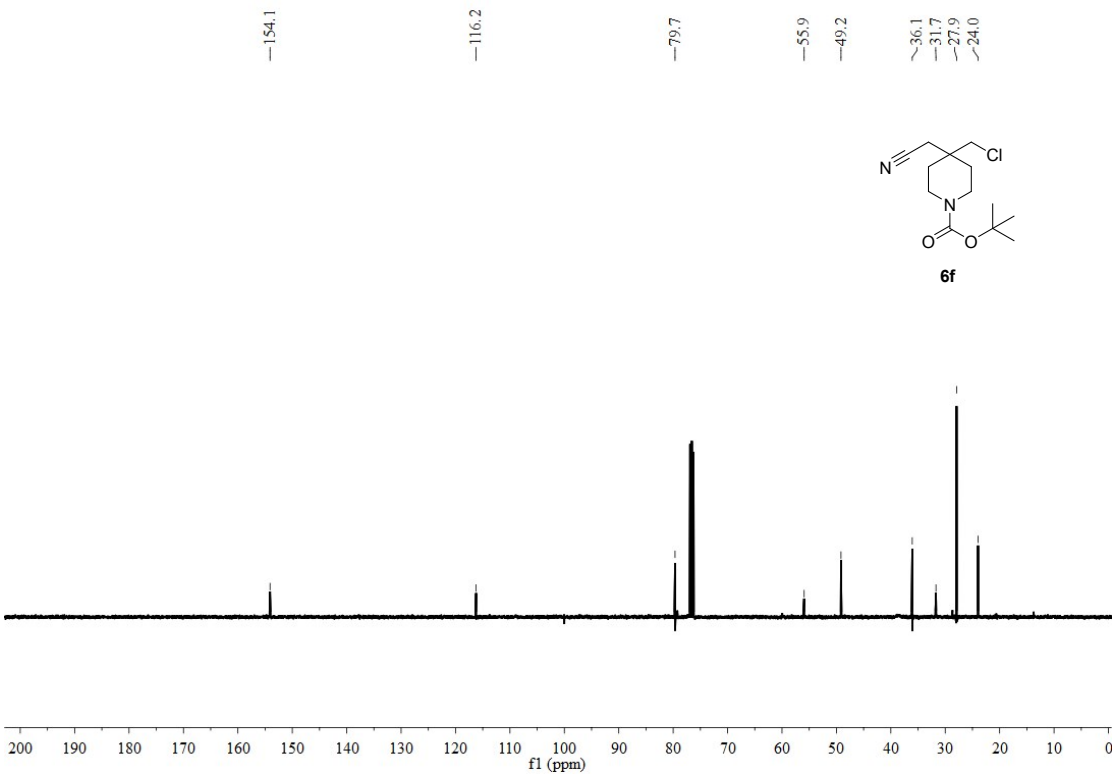
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **8e**



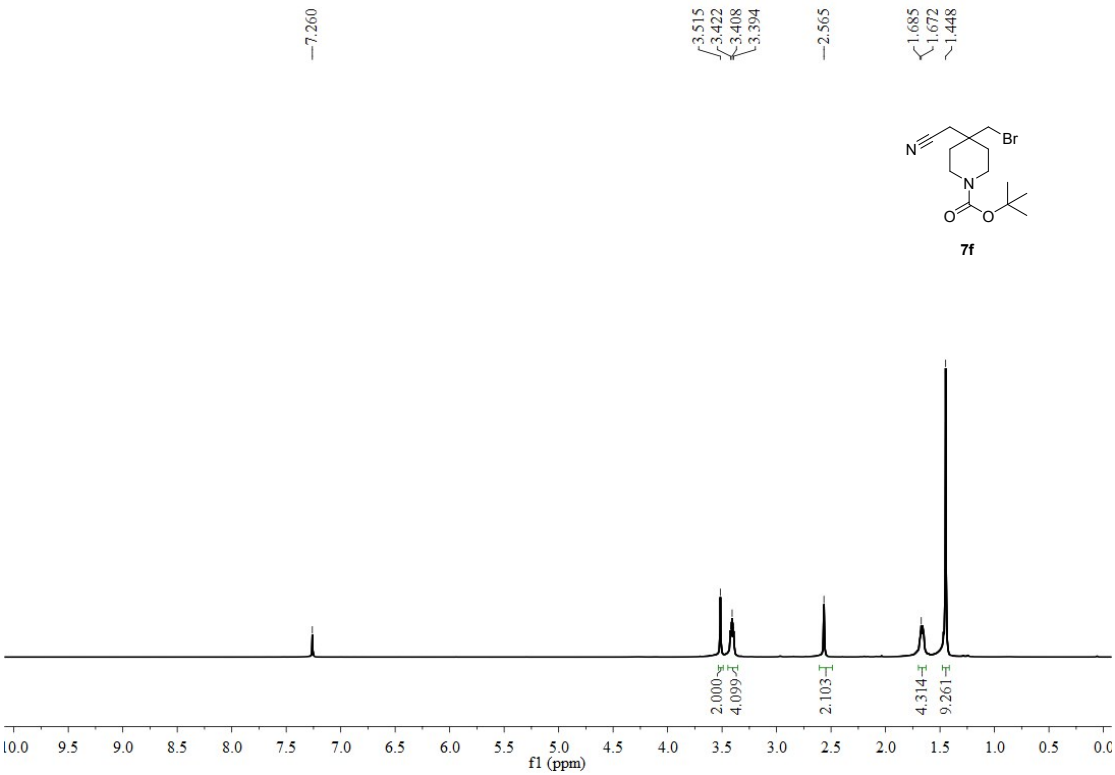


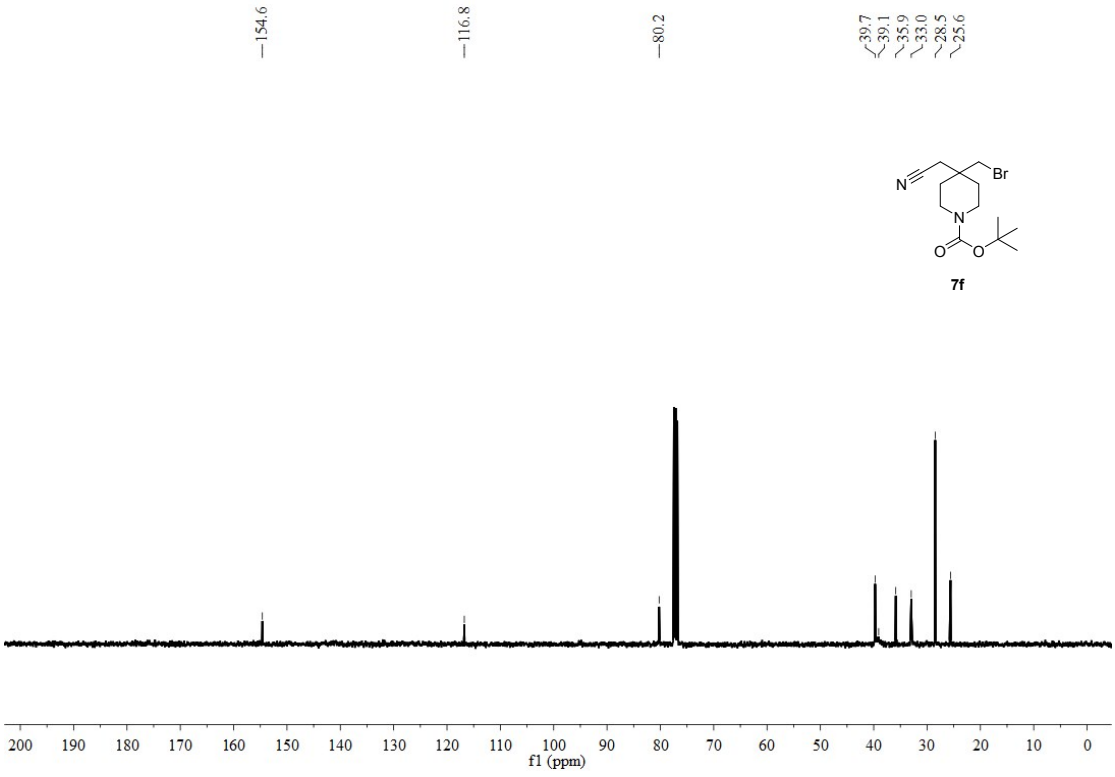
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **6f**



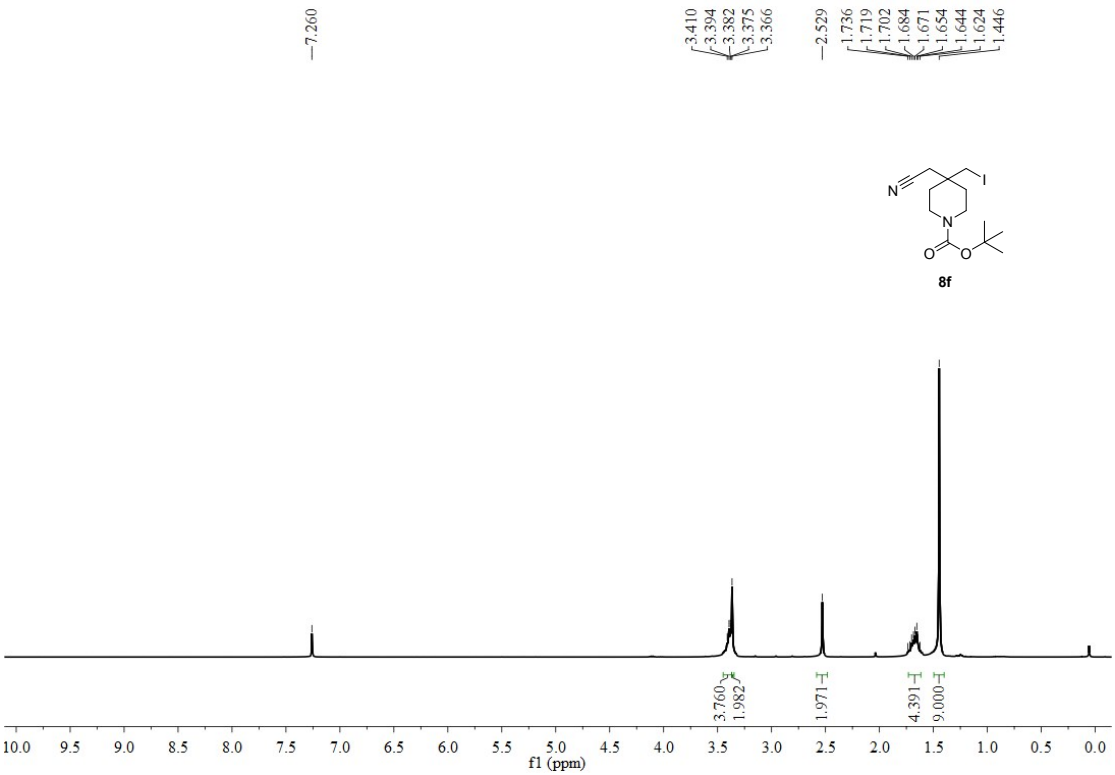


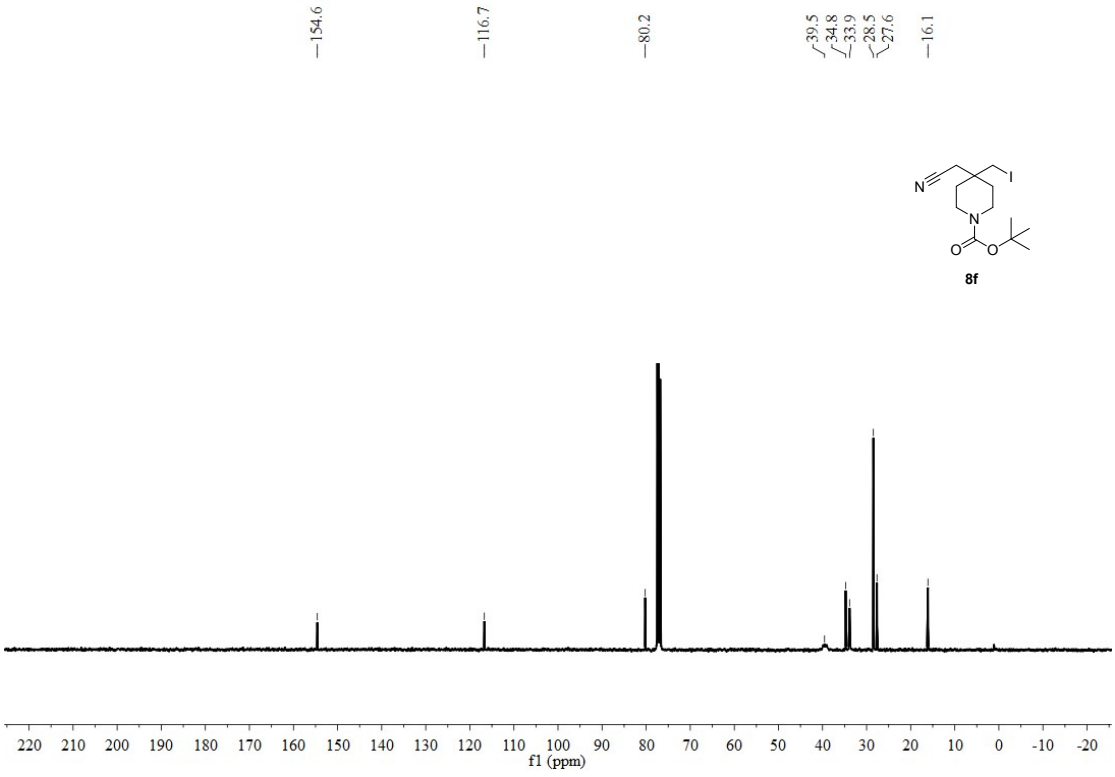
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **7f**



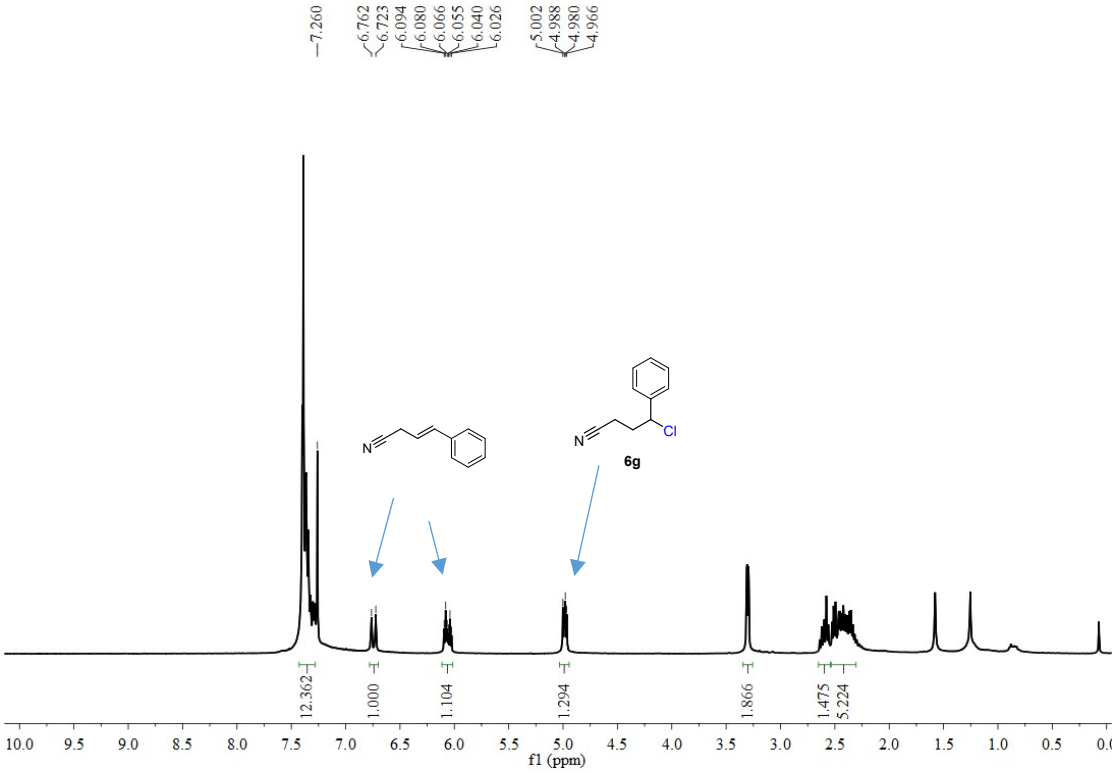


^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **8f**





^{1}H NMR (400 MHz, $CDCl_3$) spectra of product **6g**



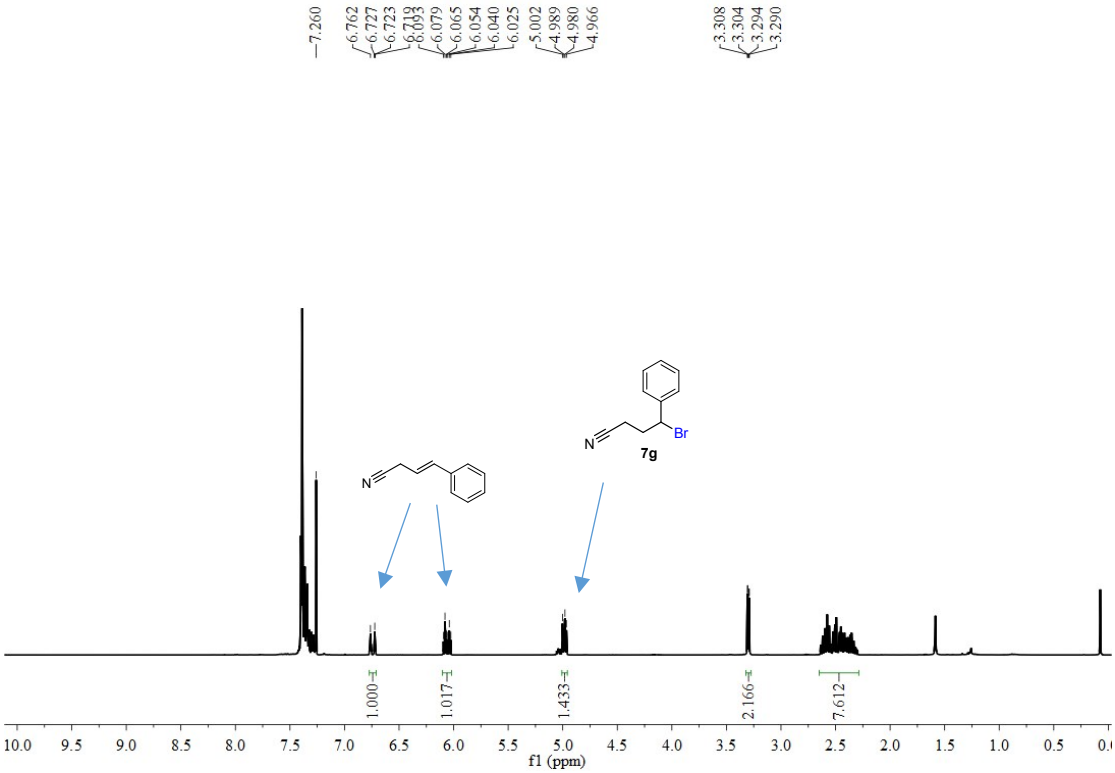
$1.294n_1 \times 179 + 1.000n_2 \times 143 = 22$ mg

$n_1 = 1.294\ n_2$

$n_1 = 0.076$ mmol, $n_2 = 0.059$ mmol

n_1Yield = 38%, n_2 Yield = 30%

^{1}H NMR (400 MHz, $CDCl_3$) spectra of product **7g**



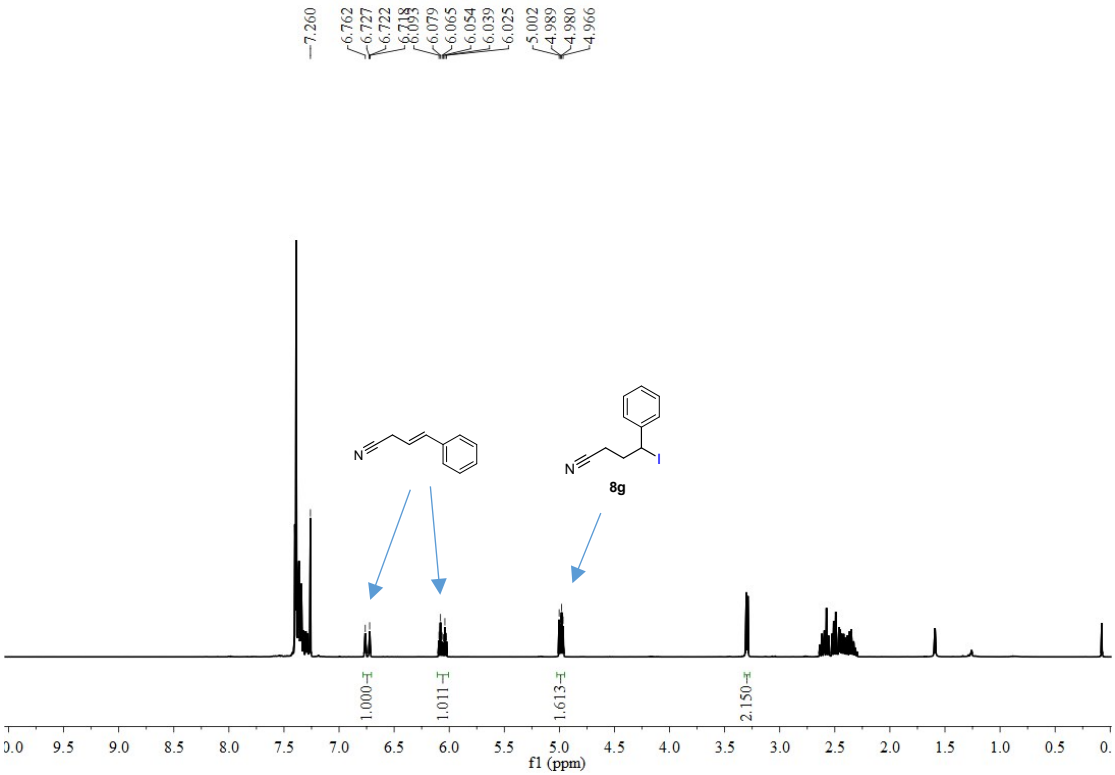
$1.433n_1 \times 233 + 1.000n_2 \times 143 = 16$ mg

$n_1 = 1.433n_2$

$n_1 = 0.0382$ mmol, $n_2 = 0.0266$ mmol

n_1 yield = 19%, n_2 yield = 13%

^{1}H NMR (400 MHz, $CDCl_3$) spectra of product **8g**



$1.613n_1 \times 271 + 1.000n_2 \times 143 = 18.8$ mg

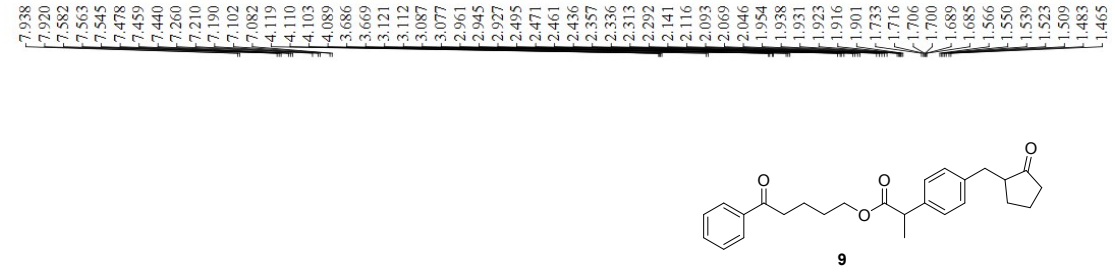
$n_1 = 1.613n_2$

$n_1 = 0.0523$ mmol, $n_2 = 0.0324$ mmol

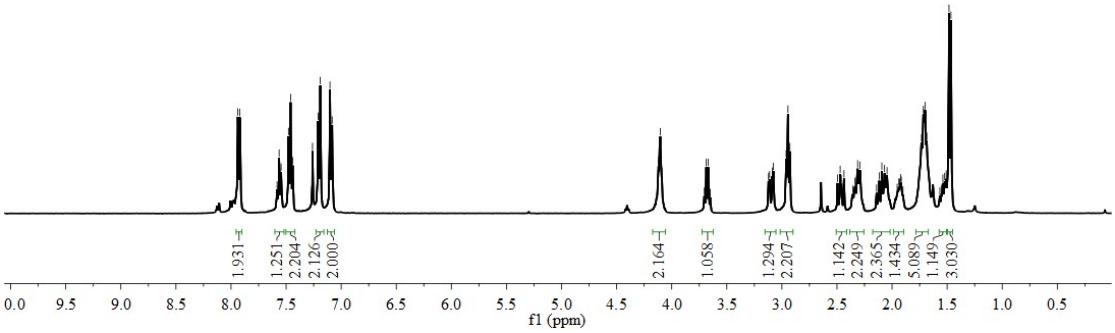
n_1 yield = 19%, n_2 yield = 13%

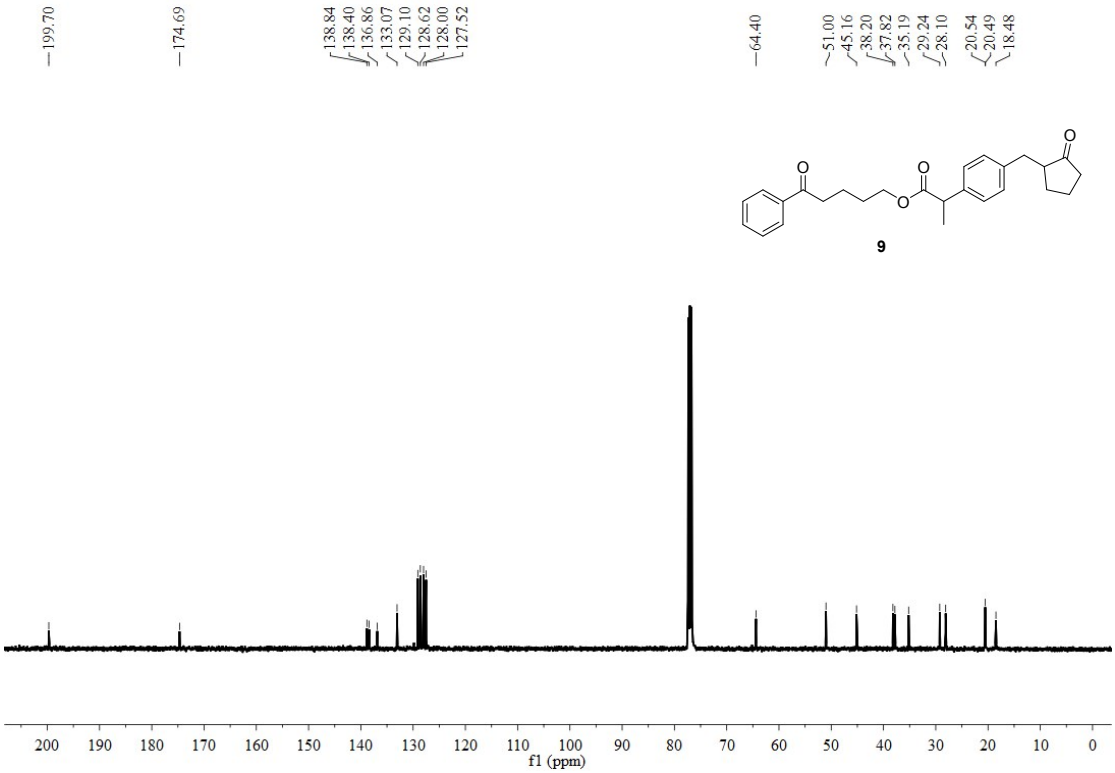
18. ^{1}H NMR and ^{13}C NMR Spectra of Products 9-12

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **9**



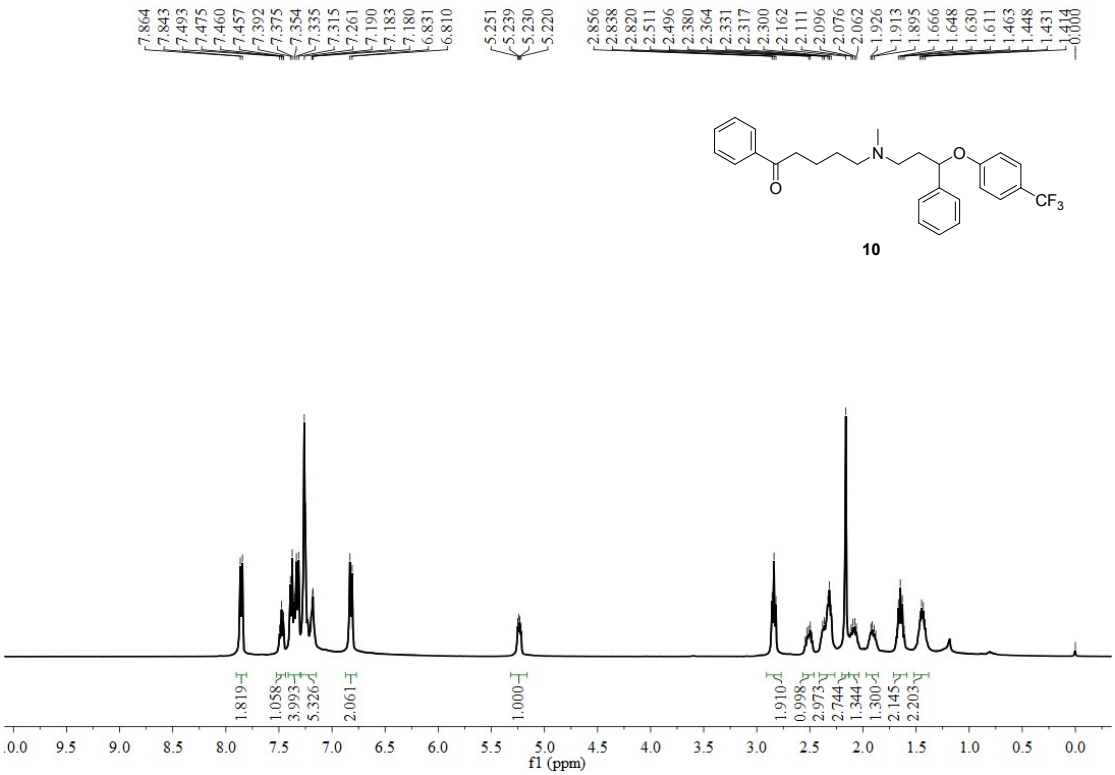
9

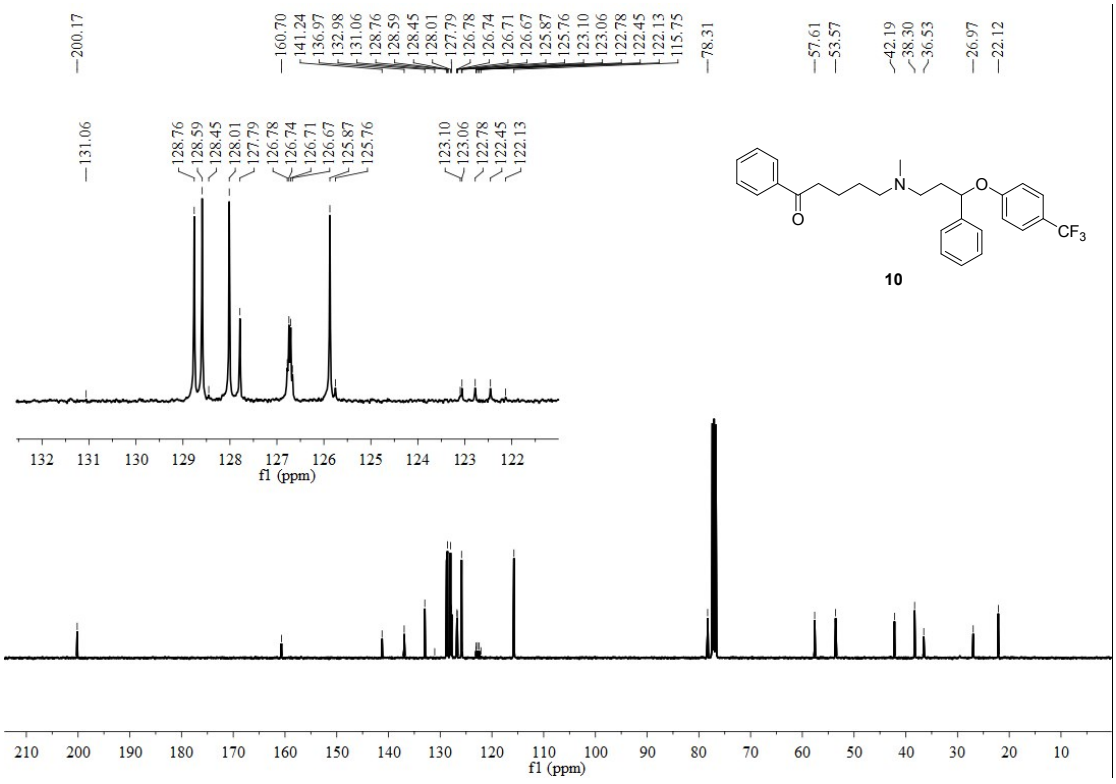




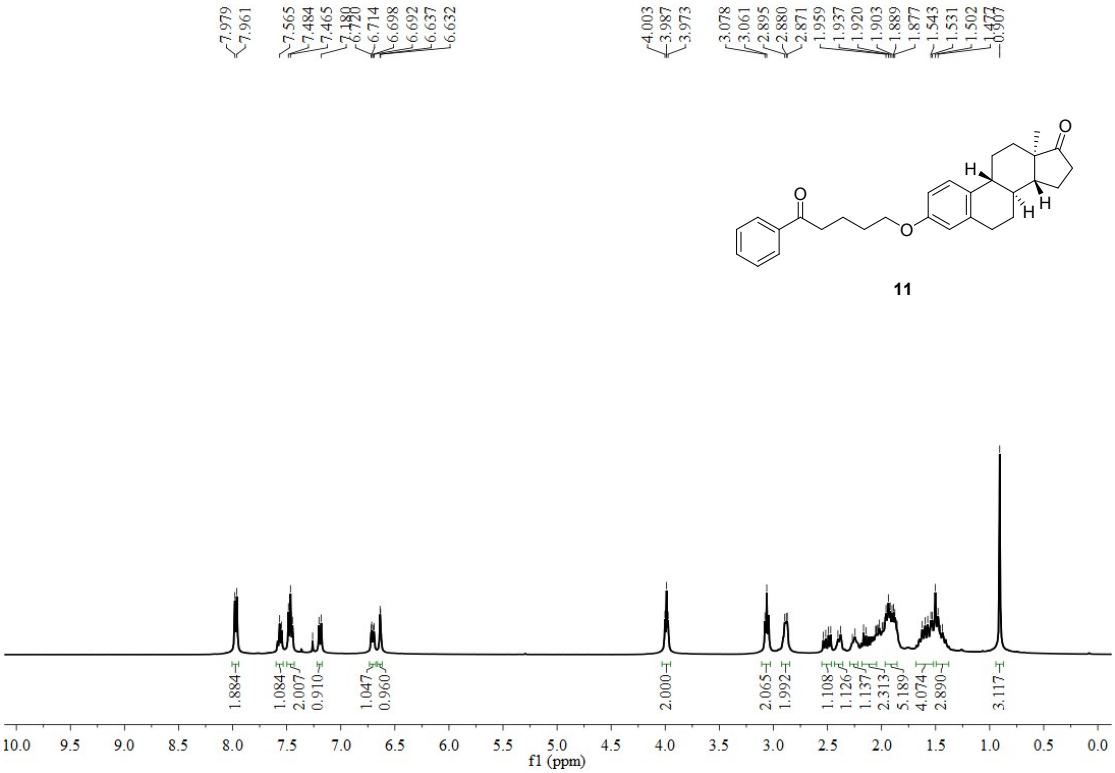
9

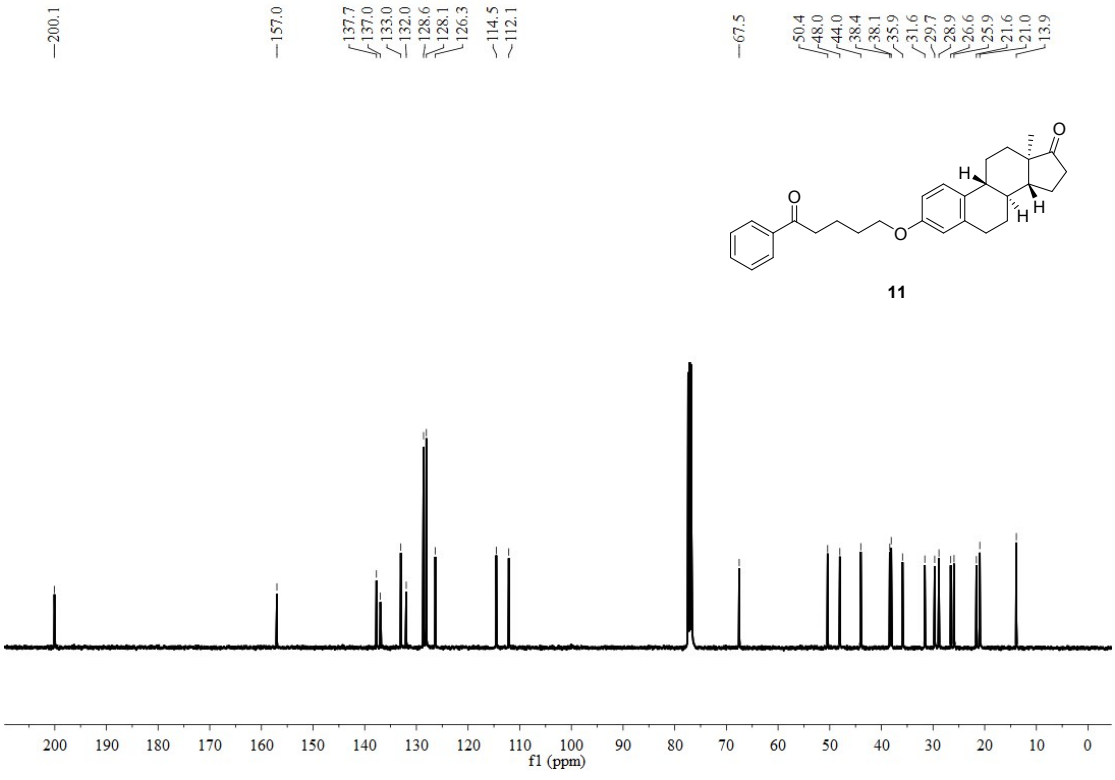
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **10**





^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **11**





^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product **12**