Supporting Information for

Palladium-Catalyzed *β*-Mesylation of Simple Amide via Primary sp^3C-H Activation

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I. General Information

NMR spectra were obtained on a Varian Mercury 400 plus instrument and Bruker AMX-400 instrument (400 MHz for ^1H, and 100 MHz for ^{13}C). ^{19}F NMR spectra were recorded on Bruker AMX-400 instrument (376 MHz). HRMS data were recorded on ACQUITYTM UPLC & Q-TOF MS Premier. Melting points were obtained on an INESA SGW X-4 melting point apparatus.

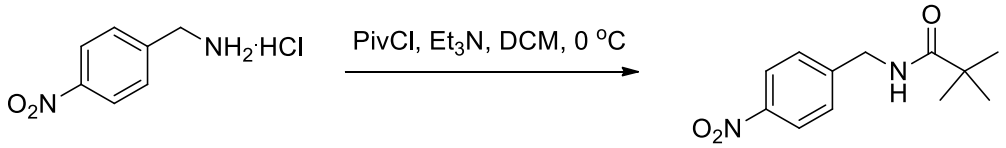
All reagents were from commercial sources. Solvents were distilled before use. Flash

Chromatography was performed on silica gel (200-300 mesh).

II. Experimental Section

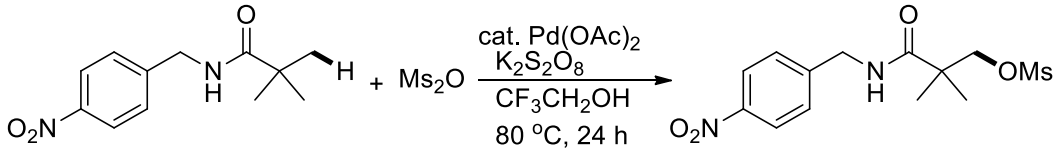
1. Typical procedures

(1) A typical procedure of preparation of amide substrates (*N*-(4-nitrobenzyl)pivalamide) (1a)



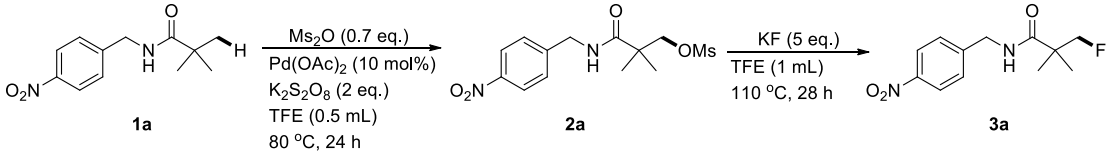
To a stirring solution of (4-nitrophenyl)methanamine hydrochloride (5.0 g, 26.5 mmol), Et_3N (6.7 g, 66.3 mmol) in DCM (50 mL), was added trimethylacetylchloride (PivCl) (3.8 g, 31.8 mmol) at 0 °C (ice/water bath). The reaction mixture was allowed to warm to room temperature, and stirred at rt overnight. After that, the reaction mixture was quenched with water (100 mL), and extracted with DCM (10 mL*3), washed with aq.K_2CO_3, brine, and dried over $MgSO_4$. Then, the combined extracts were concentrated and recrystallization from n-hexane/EtOAc (3:1, v/v) mixture to afford *N*-(4-nitrobenzyl)pivalamide (**1a**) (light yellow solid) (5.9 g, yield: 94%).

(2) A typical procedure of *β*-mesylation of amides (2,2-dimethyl-3-((4-nitrobenzyl)amino)-3-oxopropyl methanesulfonate) (2a)



The mixture of *N*-(4-nitrobenzyl)pivalamide (**1a**) (236 mg, 1.0 mmol), $Pd(OAc)_2$ (4.4 mg, 0.02 mmol, 2 mol %), $K_2S_2O_8$ (108 mg, 0.4 mmol) in TFE (800 mg, 8 mmol) were heated at 80 °C for 2 hours (oil bath). Then another batch of $Pd(OAc)_2$ (18 mg, 0.08 mmol, 8 mol %), $K_2S_2O_8$ (432 mg, 1.6 mmol) was added to the mixture and reacted at this temperature for another 22 hours. After that, the reaction mixture was added CH_2Br_2 (90 mg, 0.50 mmol) as internal standard and detected by ^1H NMR analysis (yield: 82%). The reaction mixture was transferred into a 50 mL round-bottom flask and the solvent was evaporated. The desired *β*-mesylated amide (**2a**) was isolated by flash column chromatography on silica gel using n-hexane/ethyl acetate (2:1, v/v) as eluant, slurry product (247 mg, 75%) recrysalized from (PE/EtOAc), to give a light yellow solid (200 mg, 61%).

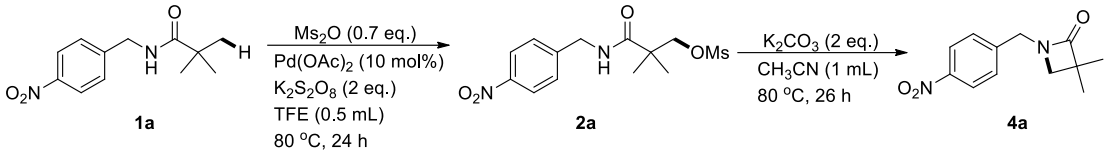
(3) A typical procedure of *β*-fluoride of amides (3-fluoro-2,2-dimethyl-*N*-(4-nitrobenzyl)propanamide) (3a)



The above mesylation reaction mixture (from substrate, 1 mmol) (without purification)

was cooled to room temperature and filtered through celite. The cake was washed with EtOAc (10 mL*3) and the combined filtrate was evaporated under reduced pressure. The crude *β*-mesylated amide (**2a**) was added TFE (1 mL), and KF (330 mg, 4.5 mmol). The mixture was heated to 110 °C and stirred for 28 hours to give the *β*-fluoroamide product (**3a**). The reaction mixture was filtered through celite, washed with EtOAc (10 mL*3) and the combined filtrate was evaporated under reduced pressure. The crude mixture was detected by ^{1}H NMR analysis (yield: 74%). Then the desired *β*- fluoride amide (**3a**) was purified by flash column chromatography on silica gel using n-hexane/ethyl acetate (5:1, v/v) as eluant, to give a light yellow solid (165 mg, 65%).

(4) A typical procedure of *β*-lactam (3,3-dimethyl-1-(4-nitrobenzyl)azetidin-2-one) (4a)



The crude *β*-mesylated amide (**2a**) (from substrate, 1.0 mmol) was dissolved in CH_3CN (1 mL), then K_2CO_3 (276 mg, 2.0 mmol) was added. The mixture was heated to 80 °C and stirred for 26 hours to give the *β*-lactam product (**4a**). The reaction mixture was detected by ^{1}H NMR analysis (yield: 73%). Then the desired *β*-fluoride amide (**4a**) was isolated by flash column chromatography on silica gel using n-hexane/ethyl acetate (5:1, v/v) as eluant, to give a light yellow solid (150 mg, 64%).

2. Screening the reaction conditions for *β*-mesylation products

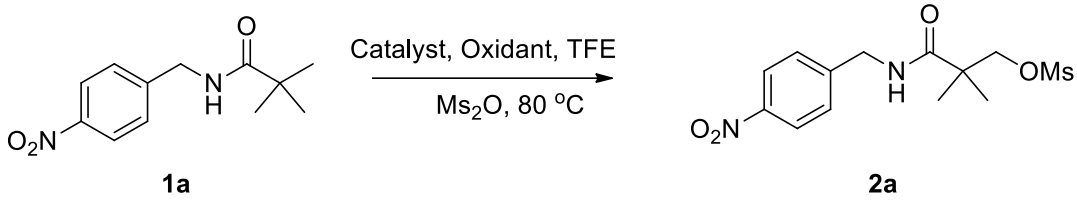


Table S-1 **Effects of solvents**[a]

Entry	Solvent	Yield (%)
1	EA	13
2	CH_3CN	0
3	DMF	0
4	DCE	36
5	THF	trace

6	Dioxane	0
7	DCM	66
8	$CHCl_3$	56
9	TFE	76

[a] Yields based on substrate and detected by ^1H NMR analysis using CH_2Br_2 as internal standard. Conditions: **1a** (0.1 mmol), $Pd(OAc)_2$ (0.01 mmol), $K_2S_2O_8$ (0.2 mmol), Ms_2O (0.07 mmol), solvent (0.3 mL), 80 °C, 24 h.

Table S-2 **Effects of catalyst and oxidants** [a]

Entry	Catalyst	Oxidant	Yield (%)
1	$Pd(OAc)_2$	$K_2S_2O_8$	74
2	$Pd(PPh_3)_4$	$K_2S_2O_8$	38
3	$Pd(PPh_3)_2Cl_2$	$K_2S_2O_8$	25
4	$Pd(TFA)_2$	$K_2S_2O_8$	60
5	$PdCl_2$	$K_2S_2O_8$	0
6	$Cu(OAc)_2$	$K_2S_2O_8$	0
7	$Co(OAc)_2·4H_2O$	$K_2S_2O_8$	0
8	$PtCl_2$	$K_2S_2O_8$	0
9	$Pd(OAc)_2$	Oxone (1.25 equiv)	36
10	$Pd(OAc)_2$	Oxone (2.5 equiv)	40
11	$Pd(OAc)_2$	TBHP	0
12	$Pd(OAc)_2$	$Na_2CO_3·1.5H_2O_2$	0
13	$Pd(OAc)_2$	AgOAc	0
14	$Pd(OAc)_2$	$Cu(OAc)_2$	0
15	$Pd(OAc)_2$	$PhI(OAc)_2$	38

[a] Yields based on substrate and detected by ^{1}H NMR analysis using CH_2Br_2 as internal standard. Conditions: **1a** (0.1 mmol), catalyst (0.01 mmol), oxidant (0.2 mmol), Ms_2O (0.07 mmol), TFE (0.3 mL), 80 °C, 24 h.

Table S-3 **Effects of the amount of $Pd(OAc)_2$** [a]

Entry	$Pd(OAc)_2$ (mol %)	Yield (%)
1	1	16
2	3	39
3	5	66
4	10	70
5	20	70

[a] Yields based on substrate and detected by ^{1}H NMR analysis using CH_2Br_2 as internal standard. Conditions: **1a** (0.1 mmol), $Pd(OAc)_2$, $K_2S_2O_8$ (0.2 mmol), Ms_2O (0.07 mmol), TFE (0.3 mL), 80 °C, 24 h.

Table S-4 **Effects of temperature** [a]

Entry	T (^{o}C)	Yield (%)
1	60	63
2	70	67
3	80	70
4	90	65
5	100	60

[a] Yields based on substrate and detected by ^{1}H NMR analysis using CH_2Br_2 as internal standard. Conditions: **1a** (1.0 mmol), $Pd(OAc)_2$ (0.01 mmol), $K_2S_2O_8$ (0.2 mmol), Ms_2O (0.07 mmol), TFE (0.3 mL), heat, 24 h.

Table S-5 **Effects of the amount of $K_2S_2O_8$** [a]

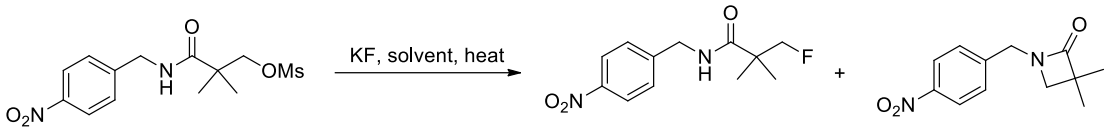
Entry	$K_2S_2O_8$ (equiv)	Yield (%)
1	1.0	45

2	1.5	52
3	2.0	70
4	2.5	69
5	3.0	61

[a] Yields based on substrate and detected by ^{1}H NMR analysis using CH_2Br_2 as internal standard. Conditions: **1a** (1.0 mmol), $Pd(OAc)_2$ (0.01 mmol), $K_2S_2O_8$, Ms_2O (0.07 mmol), TFE (0.3 mL), 80 °C, 24 h.

3. Screening the reaction conditions for *β*-fluoramides

Table S-6 **Optimization for 3-fluoro-2,2-dimethyl-*N*-(4-nitrobenzyl)propanamide (3a)** [a]



2a **3a** **4a**

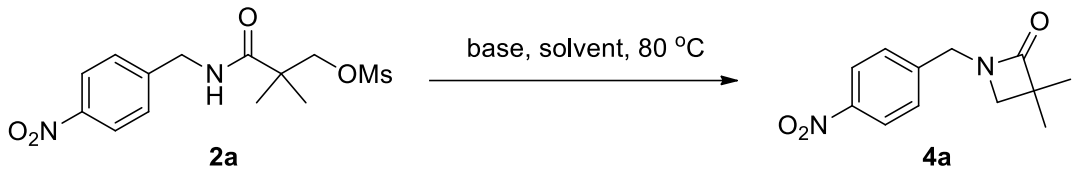
Entry	Solvent	T (^{o}C)	Yield
1	MeOH	80	7% **3a**+16% **4a**
2	EtOH	80	9% **3a** +37% **4a**
3	CH_3CN	80	trace
4	THF	80	0
5	Acetone	80	10% **4a**
6	$CHCl_3$	80	0
7	DMF	80	34% **4a**
8	DMSO	80	30% **4a**
9	TFE	80	14% **3a** +5% **4a**
10	EA	80	0
11	TFE	80	24% **3a** +10% **4a**
12	TFE [b]	80	20% **3a** +10% **4a**
13	HFIP	80	19% **4a**
14	i-PrOH	80	0

15	TFE	90	18% **3a** +8% **4a**
16	TFE	100	58% **3a**
17	TFE	110	78% **3a** +5%Etherification product
18	TFE [c]	110	83% **3a** +5% Etherification product

[a] Yields based on substrate and detected by ^{1}H NMR analysis using CH_2Br_2 as internal standard. Conditions: **2a** (0.1 mmol), KF (0.5 mmol), solvent (1 mL), 20 h. [b] KF (1 mmol) .[c] 26 h.

4. Screening the reaction conditions for *β*-lactams

Table S-7 **Optimization for 3,3-dimethyl-1-(4-nitrobenzyl)azetidin-2-one (4a)** [a]



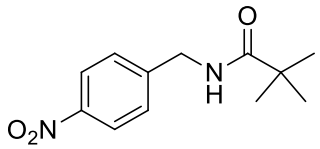
Entry	Base	Solvent	Yield (%)
1	TEA	EA	0
2	TEA	EtOH	12
3	TEA	$CHCl_3$	0
4	TEA	DMF	0
5	TEA	DCM	12
6	TEA	Acetone	0
7	TEA	DMSO	0
8	TEA	Toluene	0
9	K_2CO_3	MeOH	0
10	K_2CO_3	EtOH	0
11	K_2CO_3	DCM	25
12	K_2CO_3	CH_3CN	99

[a] Yields based on substrate and detected by ^{1}H NMR analysis using CH_2Br_2 as internal standard. Conditions: **2a** (0.1 mmol), TEA (0.5 mmol) or K_2CO_3 (0.2 mmol), solvent (1 mL), 80 °C, 22 h.

III. Preparations and Characterization of Compounds and NMR Spectra

1. Preparations and characterization of compounds

(1) The amide substrates



***N*-(4-Nitrobenzyl)pivalamide (1a)**

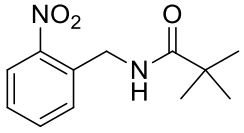
Light yellow solid.

Mp (melting point): 122-124 °C (ref 1: 119-121 °C).

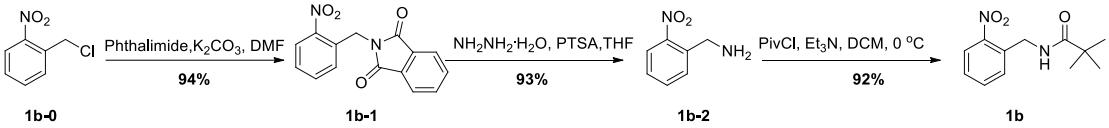
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.8 Hz, 2H), 7.41 (d, *J* = 8.8 Hz, 2H), 6.13 (br s, 1H), 4.53 (d, *J* = 6.0 Hz, 2H), 1.25 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.7 (C=O), 147.2 (C), 146.5 (C), 128.0 (CH), 123.9 (CH), 42.8 (CH_2), 38.8 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.0 (upward), 123.9 (upward), 42.8 (downward), 27.6 (upward).



Preparation of *N*-(2-nitrobenzyl)pivalamide (1b) (ref 2)



2-(2-Nitrobenzyl)isoindoline-1,3-dione (1b-1)

To a solution of 1-(chloromethyl)-2-nitrobenzene (**1b-0**) (5.0 g, 29.2 mmol) in DMF (40 mL) was added phthalimide (5.2 g, 35.4 mmol), and K_2CO_3 (8.4 g, 60.9 mmol). The mixture was stirred at room temperature overngiht. Water (200 mL) was added, and the precipitate was collected by filtration. The cake was washed with water (20 mL*2) and dried in vacuo to give a dark brown solid (**1b-1**) (7.7 g, 94%). Mp: 215-217 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.92-7.90 (m, 2H), 7.79-7.76 (m, 2H), 7.54 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.54 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 1H), 5.30 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9 (C=O), 148.1 (C), 134.4 (CH), 133.8 (CH), 131.9 (C), 131.7 (C), 128.5 (CH), 128.1

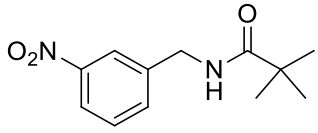
(CH), 125.4 (CH), 123.7 (CH), 38.7 (CH_2); DEPT-135 (100 MHz, $CDCl_3$) δ 134.4 (upward), 133.8 (upward), 128.5 (upward), 128.1 (upward), 125.4 (upward), 123.7 (upward), 38.7 (downward).

(2-Nitrophenyl)methanamine (1b-2)

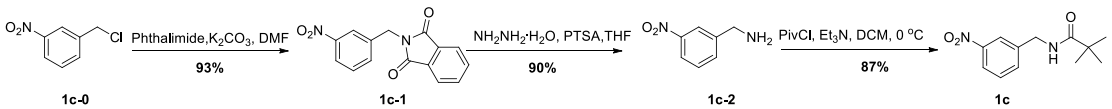
To a solution of **1b-1** (2.3 g, 8.2 mmol) in THF (20 mL) were added hydrazine monohydrate (2 g, 34 mmol) and p-toluenesulfonic acid monohydrate (PTSA) (150 mg, 0.8 mmol). The mixture was heated under reflux for 6 h. The resulting mixture was cooled to room temperature, basified with aqueous 5% potassium carbonate solution to pH=10, and extracted with dichloromethane three times. The organic layers were combined, washed with water, dried over magnesium sulfate, and concentrated in vacuo to give **1b-2** (1.2 g, 93%), which was used directly in the following step without further purification.

***N*-(2-Nitrobenzyl)pivalamide (1b)**

To a stirring solution of **1b-2** (1.2 g, 7.9 mmol), Et_3N (1.2 g, 11.9 mmol) in DCM (20 mL), was added trimethylacetylchloride (PivCl) (1.1 g, 9.1 mmol) at 0 ºC (ice/water bath). The reaction mixture was stirred at rt overnight. After that, the reaction mixture was quenched with water (20 mL), and extracted with DCM (10 mL*3), washed with aq.K_2CO_3, brine, and dried over $MgSO_4$. Then, the combined extracts were concentrated and recrystallization from hexane-EtOAc mixture to afford *N*-(2-nitrobenzyl)pivalamide (**1b**) (gray solid), (1.7 g, yield: 92%). Mp: 112-114 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.8 Hz, 1H), 7.67-7.60 (m, 2H), 7.49-7.45 (m, 1H), 6.56 (br s, 1H), 4.67 (d, *J* = 6.4 Hz, 2H), 1.20 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6 (C=O), 148.5 (C), 134.0 (CH), 133.9 (C), 132.5 (CH), 128.6 (CH), 125.0 (CH), 41.4 (CH_2), 38.8 (C), 27.5 (CH_3); DEPT-135 (100 MHz, $CDCl_3$) δ 134.1 (upward), 132.5 (upward), 128.6 (upward), 125.1 (upward), 41.4 (downward), 27.5 (upward).



Preparation of *N*-(3-nitrobenzyl)pivalamide (1c)



2-(3-Nitrobenzyl)isoindoline-1,3-dione (1c-1)

The similar method to **1b-1**, using **1c-0** (5 g, 33 mmol) as starting material to obtain **1c-1** as a light brown solid (8.6 g, 93%). Mp: 164-165 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (t, *J* = 1.2Hz, 1H), 8.16 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.91-7.87 (m, 2H), 7.79-7.75 (m, 3H), 7.53 (t, *J* = 8.0 Hz, 1H), 4.96 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8 (C=O), 148.4 (C), 134.7 (CH), 134.3 (CH), 131.9 (C), 129.8 (CH), 123.6 (CH), 123.5 (CH), 123.0 (CH), 40.8 (CH_2); DEPT-135 (100 MHz, $CDCl_3$) δ 134.7 (upward), 134.3

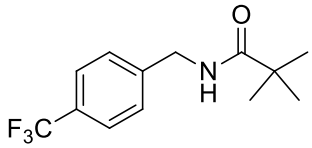
(upward), 129.8 (upward), 123.6 (upward), 123.5 (upward), 123.0 (upward), 40.8 (downward).

(3-Nitrophenyl)methanamine (1c-2)

The similar method to **1b-2**, giving **1c-2** as a brown solid (90%), which was used directly in the following step without further purification.

***N*-(3-Nitrobenzyl)pivalamide (1c)**

The similar method to **1b**, giving **1c** as a light yellow solid (87%). Mp: 75-78 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12-8.10 (m, 2H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 1H), 6.29 (br s, 1H), 4.54 (d, *J* = 6.0 Hz, 2H), 1.26 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.8 (C=O), 148.4 (C), 141.1 (C), 133.6 (CH), 129.6 (CH), 122.3 (CH), 122.0 (CH), 42.7 (CH_2), 38.8 (C), 27.6 (CH_3); DEPT-135 (100 MHz, $CDCl_3$) δ 133.6 (upward), 129.6 (upward), 122.3 (upward), 122.0 (upward), 42.7 (downward), 27.6 (upward).



***N*-(4-(Trifluoromethyl)benzyl)pivalamide (1d)**

White solid.

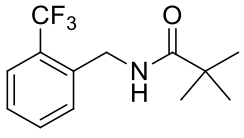
Mp: 111-114 °C (ref 3: 114-119 °C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 6.16 (br s, 1H), 4.50 (d, *J* = 6.0 Hz, 2H), 1.25 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6 (C=O), 142.9 (C), 129.6 (C), 127.7 (CH), 125.6 (CH), 124.1 (CF_3), 43.0 (CH_2), 38.8 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 127.7 (upward), 125.6 (upward), 43.0 (downward), 27.6 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.5.



***N*-(2-(Trifluoromethyl)benzyl)pivalamide (1e)**

White solid.

Mp: 81-85 °C (ref 3: 82-85 °C).

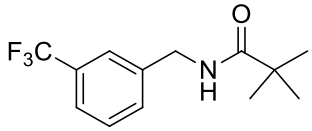
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.0 Hz, 2H), 7.52-7.50 (m, 2H), 7.40-7.36 (m, 1H), 6.14 (br s, 1H), 4.61 (d, *J* = 6.0 Hz, 2H), 1.21 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4 (C=O), 137.0 (C), 132.3 (CH), 130.4 (CH),

128.1 (C), 127.5 (CH), 126.0 (CH), 124.5 (CF_3), 40.2 (CH_2), 38.7 (C), 27.5 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 132.3 (upward), 130.4 (upward), 127.5 (upward), 126.0 (upward), 40.2 (downward), 27.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -59.6.



***N*-(3-(Trifluoromethyl)benzyl)pivalamide (1f)**

Light yellow solid.

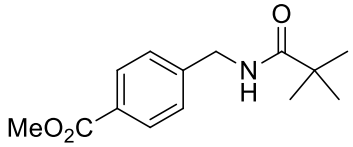
Mp: 72-75 °C (ref 3: 73-76 °C).

^1H NMR (400 MHz, $CDCl_3$) δ 7.54-7.28 (m, 4H), 6.16 (br s, 1H), 4.50 (d, *J* = 6.0 Hz, 2H), 1.25 (s, 9H).

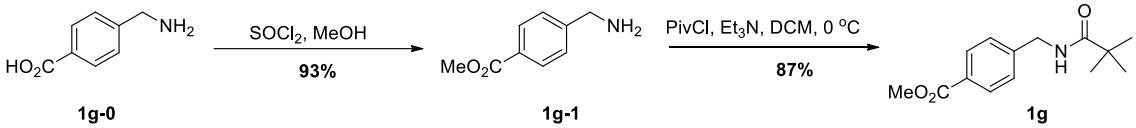
^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6 (C=O), 139.9 (C), 131.0 (C), 130.9 (CH), 129.2 (CH), 124.1 (CH), 124.0 (CF_3), 43.0 (CH_2), 38.8 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 130.8 (upward), 129.2 (upward), 124.1 (upward), 42.9 (downward), 27.6 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.7.



Preparation of methyl 4-(pivalamidomethyl)benzoate (1g) (ref 4)



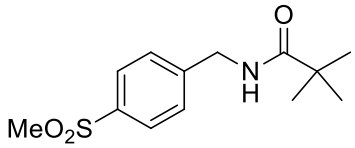
1g-0 **1g-1** **1g**

Methyl 4-(aminomethyl)benzoate (1g-1)

A solution of 4-(aminomethyl)benzoic acid (**1g-0**) (5.0 g, 33.1 mmol) in MeOH (30 mL) were cooled to 0 °C. To this solution, $SOCl_2$ (3.5 mL, 43.7 mmol) was added dropwise in 30 min. The cooling bath was removed, and the mixture was stirred at rt overnight. The volatile solvent was evaporated to give **1g-1** as hydrochloride salt (99%). Mp: 264-267 °C. ^1H NMR (400 MHz, DMSO-d_6) δ 8.80 (br s, 2H), 7.97 (d, *J* = 8.0 Hz, 2H), 7.68 (d, *J* = 8.0 Hz, 2H), 4.10 (s, 2H), 3.86 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.4 (C=O), 139.9 (C), 129.9 (CH), 129.7 (C), 52.7 (CH_3), 42.1 (CH_2); DEPT-135 (100 MHz, DMSO-d_6) δ 129.7 (upward), 52.7 (upward), 42.1 (downward).

Methyl 4-(pivalamidomethyl)benzoate (1g)

To a stirring solution of **1g-1** (1.65 g, 10 mmol), Et_3N (1.8 g, 18 mmol) in DCM (20 mL), was added trimethylacetylchloride (PivCl) (1.3 g, 11 mmol) at 0 °C (ice/water bath). The reaction mixture was stirred at rt overnight. After that, the reaction mixture was quenched with water (20 mL), and extracted with DCM (10 mL*3), washed with aq.K_2CO_3, brine, and dried over $MgSO_4$. Then, the combined extracts were concentrated and recrystallization from hexane-EtOAc mixture to afford methyl 4-(pivalamidomethyl)benzoate (**1g**) (white solid) (2.17 g, yield: 87%). Mp: 94-96 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 6.14 (br s, 1H), 4.49 (d, *J* = 6.4 Hz, 2H), 3.91 (s, 3H), 1.25 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.5 (C=O), 166.9 (CO_2), 144.1 (C), 130.0 (CH), 129.2 (C), 127.3 (CH), 52.1 (CH_3), 43.1 (CH_2), 38.8 (C), 27.6 (CH_3); DEPT-135 (100 MHz, $CDCl_3$) δ 130.0 (upward), 127.3 (upward), 52.1 (upward), 43.1 (downward), 27.6 (upward).



***N*-(4-(Methylsulfonyl)benzyl)pivalamide (1h)**

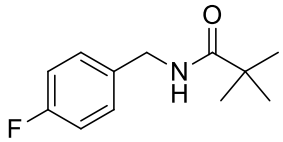
White solid.

Mp: 161-163 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 6.44 (br s, 1H), 4.50 (d, *J* = 6.0 Hz, 2H), 3.02 (s, 3H), 1.25 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.8 (C=O), 145.6 (C), 139.0 (C), 128.0 (CH), 127.6 (CH), 44.6 (CH_3), 42.8 (CH_2), 38.8 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.0 (upward), 127.6 (upward), 44.6 (upward), 42.8 (downward), 27.6 (upward).



***N*-(4-Fluorobenzyl)pivalamide (1i)**

Light yellow solid.

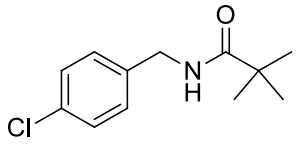
Mp: 79-82 °C (ref 3: 80-82 °C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.22 (m, 2H), 7.02 (t, *J* = 8.8 Hz, 2H), 6.02 (br s, 1H), 4.40 (d, *J* = 6.4 Hz, 2H), 1.23 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4 (C=O), 162.1 (CF), 134.5 (C), 129.2 (CH), 115.5 (CH), 42.8 (CH_2), 38.7 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 129.2 (upward), 115.5 (upward), 42.8 (downward), 27.6 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -115.2.



***N*-(4-Chlorobenzyl)pivalamide (1j)**

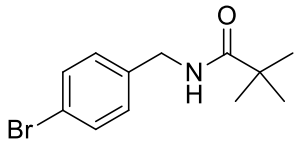
White solid.

Mp: 101-104 °C (ref 3: 101-105 °C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 5.95 (br s, 1H), 4.42 (d, *J* = 6.0 Hz, 2H), 1.25 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4 (C=O), 137.3 (C), 133.2 (C), 129.0 (CH), 128.8 (CH), 42.9 (CH_2), 38.7 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 129.0 (upward), 128.8 (upward), 42.9 (downward), 27.6 (upward).



***N*-(4-Bromobenzyl)pivalamide (1k)**

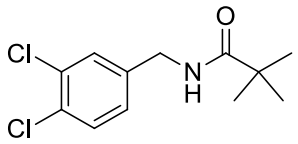
White crystal.

Mp: 106-109 °C (ref 3: 105-108 °C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 6.04 (br s, 1H), 4.39 (d, *J* = 6.0 Hz, 2H), 1.23 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4 (C=O), 137.8 (C), 131.8 (CH), 129.3 (CH), 121.2 (CH), 42.9 (CH_2), 38.7 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 131.8 (upward), 129.3 (upward), 42.9 (downward), 27.6 (upward).



***N*-(3,4-Dichlorobenzyl)pivalamide (1l)**

White solid.

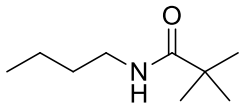
Mp: 121-123 °C (ref 3: 120-123 °C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 2.0 Hz, 1H), 7.08 (dd, *J* = 2.0, 8.0 Hz, 1H), 6.24 (br s, 1H), 4.36 (d, *J* = 6.0 Hz, 2H), 1.23 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6 (C=O), 139.2 (C), 132.6 (C), 131.2 (C), 130.6

(CH), 129.3 (CH), 126.8 (CH), 42.3 (CH_2), 38.7 (C), 27.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 130.6 (upward), 129.3 (upward), 126.8 (upward), 42.3 (downward), 27.6 (upward).



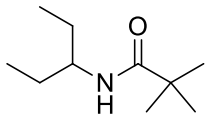
***N*-butylpivalamide (1m)**

Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.63 (br s, 1H), 3.25 (dd, *J* = 6.8, 12.8 Hz, 2H), 1.53-1.46 (m, 2H), 1.40-1.31 (m, 2H), 1.21 (s, 9H), 0.94 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3 (C=O), 39.3 (CH_2), 38.6 (C), 31.7 (CH_2), 27.6 (CH_3), 20.1 (CH_2), 13.8 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 39.3 (downward), 31.7 (downward), 27.6 (upward), 20.1 (downward), 13.8 (upward).



***N*-(Pentan-3-yl)pivalamide (1n)**

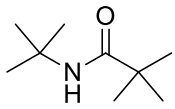
White crystal.

Mp: 114-117 ^{o}C (ref 3: 118-122 ^{o}C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.29 (br s, 1H), 3.82-3.76 (m, 1H), 1.62-1.51 (m, 2H), 1.42-1.31 (m, 2H), 1.22 (s, 9H), 0.89 (t, *J* = 7.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1 (C=O), 51.5 (CH), 38.7 (C), 27.7 (CH_3), 27.6 (CH_3), 10.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 51.5 (upward), 27.7 (upward), 27.6 (downward), 10.2 (upward).



***N*-(*tert*-Butyl)pivalamide (1o)**

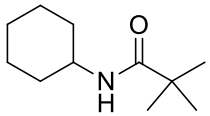
White crystal.

Mp: 115-117 ^{o}C (ref 5:117-118 ^{o}C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.41 (br s, 1H), 1.34 (s, 9H), 1.16 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.8 (C=O), 50.7 (C), 39.0 (C), 28.7 (CH_3), 27.7 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 28.7 (upward), 27.7 (upward).



***N*-Cyclohexylpivalamide (1p)**

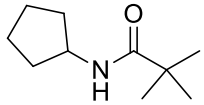
White solid.

Mp: 124-127 ºC (ref 3:122-124 ºC).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.48 (br s, 1H), 3.77-3.69 (m, 1H), 1.90-1.86 (m, 2H), 1.71-1.66 (m, 2H), 1.63-1.57 (m, 1H), 1.41-1.30 (m, 2H), 1.17 (s, 9H), 1.15-1.05 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.4 (C=O), 47.9 (CH), 38.5 (C), 33.1 (CH_2), 27.6 (CH_3), 25.6 (CH_2), 24.9 (CH_2).

DEPT-135 (100 MHz, $CDCl_3$) δ 47.9 (upward), 33.1 (downward), 27.6 (upward), 25.6 (downward), 24.9 (downward).



***N*-Cyclopentylpivalamide (1q)**

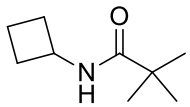
White crystal.

Mp: 125-128 ºC (ref 3:126-130 ºC).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.55 (br s, 1H), 4.20-4.12 (m, 1H), 2.04-1.94 (m, 2H), 1.70-1.56 (m, 4H), 1.37-1.28 (m, 2H), 1.17 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9 (C=O), 51.0 (CH), 38.4 (C), 33.2 (CH_2), 27.6 (CH_3), 23.8 (CH_2).

DEPT-135 (100 MHz, $CDCl_3$) δ 51.0 (upward), 33.1 (downward), 27.6 (upward), 23.8 (downward).



***N*-Cyclobutylpivalamide (1r)**

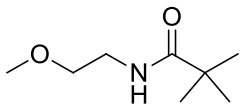
White crystal.

Mp: 133-135 ºC (ref 3:120-125 ºC).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.77 (br s, 1H), 4.41-4.31 (m, 1H), 2.37-2.30 (m, 2H), 1.87-1.77 (m, 2H), 1.73-1.65 (m, 2H), 1.17 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5 (C=O), 44.7 (CH), 38.4 (C), 31.3 (CH_2), 27.6 (CH_3), 15.0 (CH_2).

DEPT-135 (100 MHz, $CDCl_3$) δ 44.7 (upward), 31.2 (downward), 27.5 (upward), 15.0 (downward).



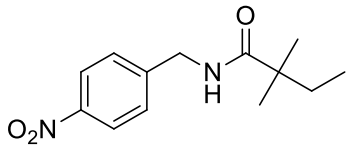
_N_-(2-Methoxyethyl)pivalamide (1s)

Light yellow liquid.

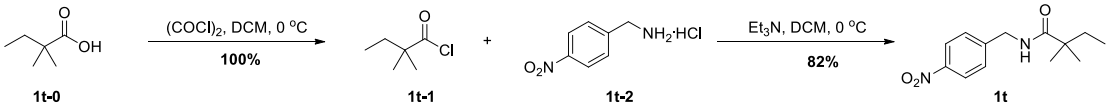
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.09 (br s, 1H), 3.35-3.28 (m, 4H), 3.24 (s, 3H), 1.08 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4 (C=O), 71.1 (CH_2), 58.6 (CH_3), 39.1 (CH_2), 38.5 (C), 27.4 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 71.1 (downward), 58.6 (upward), 39.1 (downward), 27.4 (upward).



Preparation of 2,2-dimethyl-_N_-(4-nitrobenzyl)butanamide (1t) (ref 6)



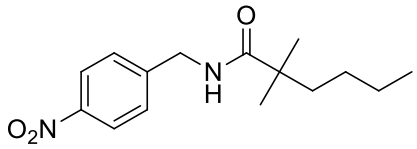
2,2-Dimethylbutanoyl chloride (1t-1)

Under nitrogen atmosphere, oxalyl chloride (640 mg, 5 mmol) was added dropwise to a solution of 2,2-dimethylbutyric acid (**1t-0**) (488 mg, 4.2 mmol) in dichloromethane (10 mL) over a period of 1 minute. The reaction mixture was stirred at rt for 60 minutes . Dichloromethane and oxalyl chloride were distilled under reduced pressure. The crude product (**1t-1**) (566 mg, yield: 100%) was used immediately in the following step without further purification.

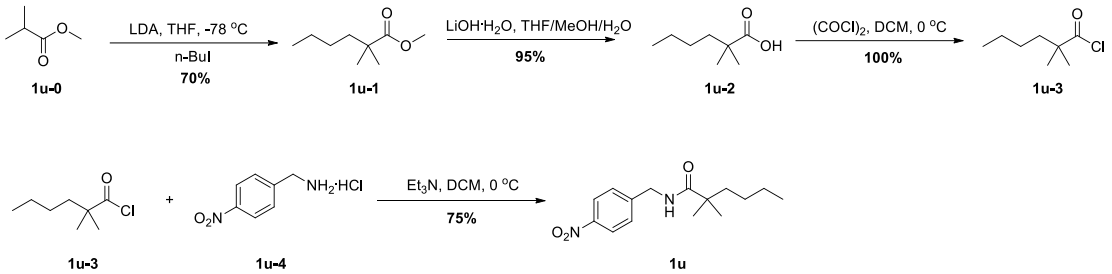
2,2-Dimethyl-_N_-(4-nitrobenzyl)butanamide (1t)

To a stirring solution of (4-nitrophenyl)methanamine hydrochloride (**1t-2**) (500 mg, 3.5 mmol), Et_3N (884 mg, 8.75 mmol) in DCM (20 mL), was added 2,2-dimethylbutanoyl chloride (**1t-1**) (566 mg, 4.2 mmol) at 0 °C (ice/water bath). The reaction mixture was allowed to warm to room temperature, and stirred at rt overnight. After that, the reaction mixture was quenched with water (30 mL), and extracted with DCM (5 mL*3), washed with aq.K_2CO_3, brine, and dried over $MgSO_4$. Then, the combined extracts were concentrated and recrystallization from hexane-EtOAc mixture to afford 2,2-dimethyl-_N_-(4-nitrobenzyl)butanamide (**1t**) (yellow solid) (720 mg, yield: 82%). Mp: 80-82 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, J = 8.4 Hz, 2H), 7.41 (d, J = 8.4 Hz, 2H), 6.31 (br s, 1H), 4.53 (d, J = 6.4 Hz, 2H), 1.59 (q, J = 7.2 Hz, 2H), 1.20 (s, 6H), 0.85 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1 (C=O), 147.1 (C), 146.6 (C), 128.1 (CH), 123.8 (CH), 42.8 (CH_2), 42.5 (C), 33.8 (CH_2), 24.9 (CH_3), 9.2 (CH_3); DEPT-135 (100 MHz, $CDCl_3$) δ 128.1 (upward),

123.8 (upward), 42.8 (downward), 33.8 (downward), 25.0 (upward), 9.2 (upward).



Preparation of 2,2-dimethyl-*N*-(4-nitrobenzyl)hexanamide (1u) (ref 7)



Methyl 2,2-dimethylhexanoate (1u-1)

A solution of lithium diisopropylamide (LDA) (2M in THF) (5 mL, 1.5 mmol) in THF (15 mL) was cooled to -78 °C. Then methylisobutyrate (**1u-0**) (1.02g, 10 mmol) was added dropwise at this temperature in 10 min. The resulting mixture was stirred at -78 °C for 30 min. Then a solution of n-BuI (2.76g, 15 mmol) in THF (5 mL) was added carefully. The reaction was continued for 2 h, and slowly warmed to rt. The reaction mixture was quenched with saturated NH_4Cl, extracted with EtOAc (3 × 15 mL). The organic layer was dried over $MgSO_4$, and the solvent was filtered, evaporated under reduced pressure to give the crude product (**1u-1**) (1.1 g, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.65 (s, 3H), 1.52-1.48 (m, 2H), 1.30-1.25 (m, 4H), 1.21-1.13 (m, 2H), 1.15 (s, 6H), 0.88 (t, *J* = 7.2 Hz, 3H).

2,2-Dimethylhexanoic acid (1u-2)

To a solution of **1u-1** (1.1g, 7 mmol) in THF/MeOH/H_2O (1:1:1) (6 mL) was added $LiOH·H_2O$ (1.26 g, 30 mmol). The mixture was stirred at rt overnight. The volatile was evaporated under reduced pressure. Then the reaction mixture was acidified with conc. HCl and extracted with CH_2Cl_2. The organic layer was dried over $MgSO_4$, filtered and concentrated under vaccum to give the desired carboxylic acid (**1u-2**) as a slurry (960 mg, 95%).

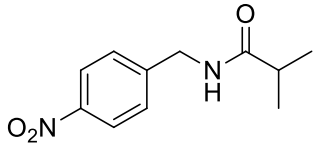
2,2-Dimethylhexanoyl chloride (1u-3)

Under nitrogen atmosphere, oxalyl chloride (1.1 g, 8.7 mmol) was added dropwise to a solution of 2,2-dimethylhexanoic acid (**1u-2**) (960 mg, 6.7 mmol) in dichloromethane (10 mL) over a period of 5 minutes. The reaction mixture was stirred at rt for 1.5 h. Dichloromethane and oxalyl chloride were distilled under reduced pressure. The crude product (**1u-3**) (1.09 g, yield: 100%) was used immediately in the following step without further purification.

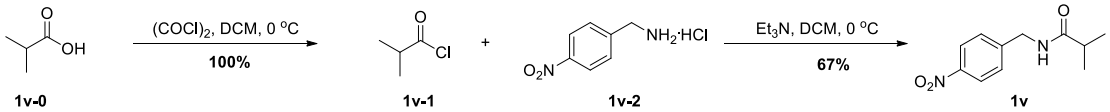
2,2-Dimethyl-*N*-(4-nitrobenzyl)hexanamide (1u)

To a stirring solution of (4-nitrophenyl)methanamine hydrochloride (**1u-4**) (890 mg,

4.7 mmol), Et_3N (1.52 g, 15 mmol) in DCM (20 mL), was added 2,2-dimethylhexanoyl chloride (**1u-3**) (1.09 g, 6.7 mmol) at 0 °C (ice/water bath). The reaction mixture was allowed to warm to room temperature, and stirred at rt overnight. After that, the reaction mixture was quenched with water (50 mL), and extracted with DCM (10 mL*3), washed with aq.K_2CO_3, brine, and dried over $MgSO_4$. The combined extracts were concentrated. The crude mixture was purified through silica gel column chromatography eluting with n-hexane/EtOAc (5:1) to afford 2,2-dimethyl-*N*-(4-nitrobenzyl)hexanamide (**1u**) (orange solid) (980 mg, yield: 75%). Mp: 57-60 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 6.10 (br s, 1H), 4.57 (d, *J* = 6.0 Hz, 2H), 1.58-1.53 (m, 2H), 1.32-1.21 (m, 4H), 1.23 (s, 6H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2 (C=O), 147.2 (C), 146.5 (C), 128.1 (CH), 123.9 (CH), 42.8 (CH_2), 42.2 (C), 41.1 (CH_2), 27.1 (CH_2), 25.4 (CH_3), 23.2 (CH_2), 14.0 (CH_3); DEPT-135 (100 MHz, $CDCl_3$) δ 128.1 (upward), 123.8 (upward), 42.8 (downward), 41.1 (downward), 27.1 (downward), 25.4 (upward), 23.2 (downward), 14.0 (upward).



Preparation of *N*-(4-nitrobenzyl)isobutyramide (1v)



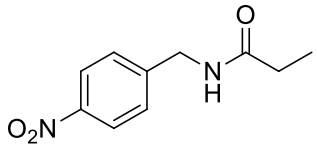
Isobutyryl chloride (1v-1)

Under nitrogen atmosphere, oxalyl chloride (640 mg, 5 mmol) was added dropwise to a solution of isobutyric acid (**1v-0**) (370 mg, 4.2 mmol) in dichloromethane (10 mL) over a period of 1 minute. The reaction mixture was stirred at rt for 60 minutes . Dichloromethane and oxalyl chloride were distilled under reduced pressure. The crude product (**1v-1**) (447 mg, yield: 100%) was used immediately in the following step without further purification.

***N*-(4-Nitrobenzyl)isobutyramide (1v)**

To a stirring solution of (4-nitrophenyl)methanamine hydrochloride (**1v-2**) (500 mg, 3.5 mmol), Et_3N (884 mg, 8.75 mmol) in DCM (20 mL), was added isobutyryl chloride (**1v-1**) (447 mg, 4.2 mmol) at 0 °C (ice/water bath). The reaction mixture was allowed to warm to room temperature, and stirred at rt overnight. After that, the reaction mixture was quenched with water (30 mL), and extracted with DCM (5 mL*3), washed with aq.K_2CO_3, brine, and dried over $MgSO_4$. Then, the combined extracts were concentrated and recrystallization from n-hexane/EtOAc mixture to afford *N*-(4-nitrobenzyl)isobutyramide (**1v**) (yellow solid) (624 mg, yield: 67%). Mp: 95-99 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 5.94 (br s, 1H), 4.57 (d, *J* = 6.0 Hz, 2H), 2.50-2.43 (m, 1H), 1.23 (d, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.4 (C=O), 147.1 (C), 146.5 (C), 128.2 (CH),

123.8 (CH), 42.7 (CH_2), 35.5 (CH), 19.7 (CH_3); DEPT-135 (100 MHz, $CDCl_3$) δ 128.2 (upward), 123.8 (upward), 42.7 (downward), 35.5 (upward), 19.7 (upward).



_N_-(4-nitrobenzyl)propionamide (1w)

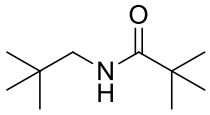
Light yellow solid.

Mp: 105-109 °C.

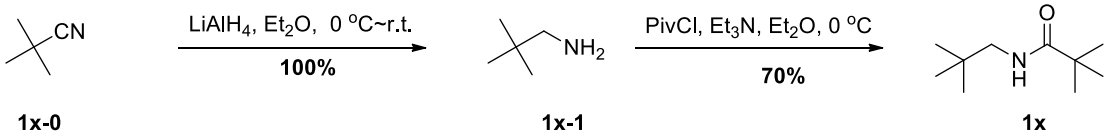
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.8 Hz, 2H), 7.41 (d, *J* = 8.8 Hz, 2H), 6.41 (br s, 1H), 4.51 (d, *J* = 6.4 Hz, 2H), 2.30 (q, *J* = 7.6 Hz, 2H), 1.18 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2 (C=O), 147.1 (C), 146.3 (C), 128.2 (CH), 123.8 (CH), 42.7 (CH_2), 29.5 (CH_2), 9.8 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.1 (upward), 123.8 (upward), 42.7 (downward), 29.5 (downward), 9.8 (upward).



Preparation of _N_-neopentylpivalamide (1x) (ref 8)



1x-0 **1x-1** **1x**

2,2-Dimethylpropan-1-amine (1x-1)

To a slurry of lithium aluminum hydride ($LiAlH_4$) (550 mg, 14.5 mmol) in anhydrous ether (10 mL) was added trimethylacetonitrile (**1x-0**) (1.0 g, 12 mmol) at 0 °C (ice/water bath) under nitrogen atmosphere. The cooling bath was removed and the mixture was stirred at rt overnight. The mixture was cooled to 0 °C again, and hydrolyzed with water (550 mg). After the addition of 15% sodium hydroxide solution (0.55 mL), water (1.75 g) was added again. The ether layer was separated, dried over $MgSO_4$. After filtration, the solution of **1x-1** was used directly in the following step.

_N_-Neopentylpivalamide (1x)

To the above solution of **1x-1** (1.04 g, 12 mmol), Et_3N (1.82 g, 18 mmol) and trimethylacetylchloride (PivCl) (1.75 g, 14.5 mmol) were added in sequence at 0 °C (ice/water bath). The reaction mixture was allowed to warm to room temperature, and stirred at rt overnight. After that, the reaction mixture was quenched with water (50 mL), and extracted with Et_2O (10 mL*3), washed with aq.K_2CO_3, brine, and dried over $MgSO_4$. The combined extracts were concentrated. The crude mixture was

purified through silica gel column chromatography eluting with n-hexane/EtOAc (1:1) to afford *N*-neopentylpivalamide (**1x**) (white solid) (1.4 g, yield: 70%). Mp: 89-90 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.70 (br s, 1H), 3.08 (d, *J* = 6.4 Hz, 2H), 1.23 (s, 9H), 0.92 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3 (C=O), 50.3 (CH_2), 38.9 (C), 32.0 (C), 27.7 (CH_3), 22.2 (CH_3); DEPT-135 (100 MHz, $CDCl_3$) δ 50.3 (downward), 27.7 (upward), 27.2 (upward).

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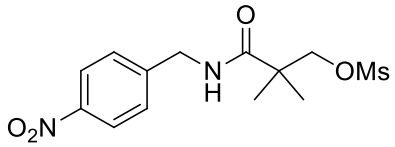
(2) *β*-Mesylated amides



2,2-Dimethyl-3-((4-nitrobenzyl)amino)-3-oxopropyl methanesulfonate (2a)

Gray solid (247 mg, 75%). R_f = 0.50, 66% EtOAc in n-hexane.

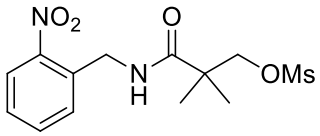
Mp: 81-83 ^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 6.27 (br s, 1H), 4.59 (d, *J* = 6.0 Hz, 2H), 4.26 (s, 2H), 3.00 (s, 3H), 1.35 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.7 (C=O), 147.3 (C), 145.6 (C), 128.0 (CH), 124.0 (CH), 76.0 (CH_2), 43.0 (CH_2), 42.9 (C), 37.0 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.0 (upward), 124.0 (upward), 76.0 (downward), 43.0 (downward), 37.0 (upward), 22.3 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{18}N_2O_6S$ $[M+Na]^+$: 353.0783; found: 353.0786.



2,2-Dimethyl-3-((2-nitrobenzyl)amino)-3-oxopropyl methanesulfonate (2b)

Brown solid (257 mg, 78%). R_f = 0.50, 66% EtOAc in n-hexane.

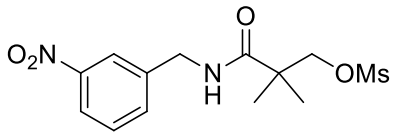
Mp: 89-91 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.6 Hz, 1H), 7.62-7.59 (m, 2H), 7.48-7.45 (m, 1H), 6.69 (br s, 1H), 4.68 (d, *J* = 6.0 Hz, 2H), 4.16 (s, 2H), 2.90 (s, 3H), 1.25 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6 (C=O), 148.4 (C), 134.1 (CH), 133.3 (C), 132.0 (CH), 128.9 (CH), 125.2 (CH), 75.7 (CH_2), 42.8 (C), 41.6 (CH_2), 36.9 (CH_3), 22.1 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 134.1 (upward), 132.0 (upward), 128.9 (upward), 125.3 (upward), 75.8 (downward), 41.6 (downward), 36.9 (upward), 22.1 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{18}N_2O_6S$ $[M+Na]^+$: 353.0783; found: 353.0788.



2,2-Dimethyl-3-((3-nitrobenzyl)amino)-3-oxopropyl methanesulfonate (2c)

White solid (231 mg, 70%). R_f = 0.50, 66% EtOAc in n-hexane.

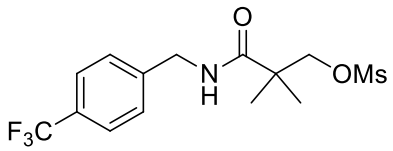
Mp: 128-130 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 8.10 (m, 2H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.53-7.49 (m, 1H), 6.56 (br s, 1H), 4.55 (d, *J* = 6.0 Hz, 2H), 4.24 (s, 2H), 2.99 (s, 3H), 1.32 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.8 (C=O), 148.4 (C), 140.5 (C), 133.6 (CH), 129.7 (CH), 122.4 (CH), 122.1 (CH), 75.9 (CH_2), 42.9 (CH_2), 42.8 (C), 37.0 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 133.5 (upward), 129.8 (upward), 122.6 (upward), 122.1 (upward), 75.9 (downward), 42.9 (downward), 37.0 (upward), 22.3 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{18}N_2O_6S$ $[M+Na]^+$: 353.0783; found: 353.0784.



2,2-Dimethyl-3-oxo-3-((4-(trifluoromethyl)benzyl)amino)propyl

methanesulfonate (2d)

Brown oil (204 mg, 58%). R_f = 0.55, 60% EtOAc in n-hexane.

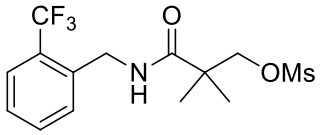
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 6.32 (br s, 1H), 4.52 (d, *J* = 6.0 Hz, 2H), 4.23 (s, 2H), 2.95 (s, 3H), 1.32 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6 (C=O), 142.2 (C), 129.8 (C), 127.6 (CH), 125.7 (CH), 124.0 (CF_3), 76.1 (CH_2), 43.2 (CH_2), 42.8 (C), 36.9 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 127.7 (upward), 125.7 (upward), 76.1 (downward), 43.2 (downward), 36.9 (upward), 22.2 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.5.

HRMS (ESI-Q/TOF MS) Calcd for $C_{14}H_{18}F_3NO_4S$ $[M+Na]^+$: 376.0806; found: 376.0816.



2,2-Dimethyl-3-oxo-3-((2-(trifluoromethyl)benzyl)amino)propyl methanesulfonate (2e)

Gray solid (211 mg, 60%). R_f = 0.60, 50% EtOAc in n-hexane.

Mp: 80-82 ºC.

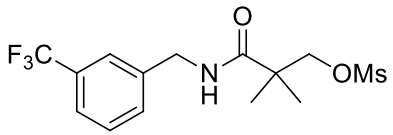
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.0 Hz,1H), 7.55-7.50 (m, 2H), 7.42-7.38 (m, 1H), 6.27 (br s, 1H), 4.64 (d, *J* = 6.0 Hz, 2H), 4.21 (s, 2H), 2.93 (s, 3H), 1.28 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4 (C=O), 136.3 (C), 132.4 (CH), 130.2 (CH), 128.1 (C), 127.8 (CH), 126.2 (CH), 124.4 (CF_3), 76.0 (CH_2), 42.8 (C), 40.5 (CH_2), 36.8 (CH_3), 22.1 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 132.4 (upward), 130.4 (upward), 127.8 (upward), 126.3 (upward), 76.0 (downward), 40.6 (downward), 36.9 (upward), 22.1 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -59.6.

HRMS (ESI-Q/TOF MS) Calcd for $C_{14}H_{18}F_3NO_4S$ $[M+Na]^+$: 376.0806; found: 376.0799.



2,2-Dimethyl-3-oxo-3-((3-(trifluoromethyl)benzyl)amino)propyl methanesulfonate (2f)

Gray solid (218 mg, 62%). R_f = 0.55, 60% EtOAc in n-hexane.

Mp: 67-69 ^{o}C.

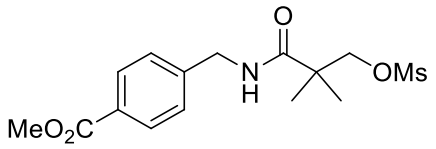
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.46 (m, 4H), 6.42 (br s, 1H), 4.50 (d, *J* = 5.6 Hz, 2H), 4.22 (s, 2H), 2.94 (s, 3H), 1.30 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6 (C=O), 139.3 (C), 131.0 (C), 130.8 (CH), 129.3 (CH), 124.3 (CH), 124.1 (C), 124.0 (CF_3), 75.9 (CH_2), 43.2 (CH_2), 42.8 (C), 36.9 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 130.8 (upward), 129.3 (upward), 124.3 (upward), 75.9 (downward), 43.3 (downward), 36.9 (upward), 22.3 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.7.

HRMS (ESI-Q/TOF MS) Calcd for $C_{14}H_{18}F_3NO_4S$ $[M+Na]^+$: 376.0806; found: 376.0815.



Methyl 4-((2,2-dimethyl-3-((methylsulfonyl)oxy)propanamido)methyl)benzoate (2g)

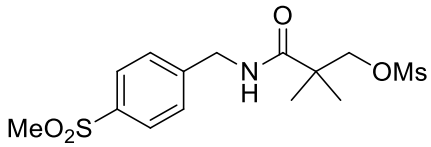
Brown slurry (154 mg, 45%). R_f = 0.50, 60% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 6.31 (br s, 1H), 4.52 (d, *J* = 6.0 Hz, 2H), 4.24 (s, 2H), 3.92 (s, 3H), 2.96 (s, 3H), 1.32 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6 (C=O), 166.8 (CO_2), 143.2 (C), 130.1 (CH), 129.4 (C), 127.3 (CH), 76.1 (CH_2), 52.2 (CH_3), 43.3 (CH_2), 42.8 (C), 36.9 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 130.0 (upward), 127.3 (upward), 76.0 (downward), 52.2 (upward), 43.4 (downward), 36.9 (upward), 22.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{15}H_{21}NO_6S$ $[M+Na]^+$: 366.0987; found: 366.0991.



2,2-Dimethyl-3-((4-(methylsulfonyl)benzyl)amino)-3-oxopropyl methanesulfonate (2h)

Light brown solid (228 mg, 63%). R_f = 0.40, 80% EtOAc in n-hexane.

Mp: 101-103 ^{o}C.

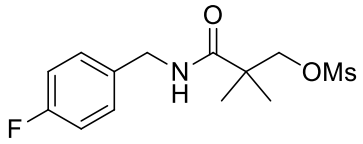
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 6.58 (br s, 1H), 4.53 (d, *J* = 6.0 Hz, 2H), 4.22 (s, 2H), 3.01 (s, 3H), 2.97 (s, 3H), 1.31 (s,

6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0 (C=O), 145.0 (C), 139.4 (C), 128.2 (CH), 127.9 (CH), 76.4 (CH_2), 44.8 (CH_3), 43.2 (CH_2), 43.1 (C), 37.2 (CH_3), 22.4 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.0 (upward), 127.8 (upward), 76.1 (downward), 44.5 (upward), 43.0 (downward), 37.0 (upward), 22.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{14}H_{21}NO_6S_2$ $[M+Na]^+$: 386.0708; found: 386.0714.



3-((4-Fluorobenzyl)amino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2i)

Brown slurry (127 mg, 42%). R_f = 0.50, 60% EtOAc in n-hexane.

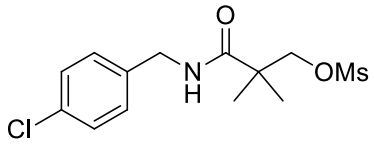
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.21 (m, 2H), 7.04-6.99 (m, 2H), 6.12 (br s, 1H), 4.41 (d, *J* = 6.0 Hz, 2H), 4.21 (s, 2H), 2.93 (s, 3H), 1.28 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3 (C=O), 162.2 (CF), 133.8 (C), 129.3 (CH), 115.6 (CH), 76.1 (CH_2), 43.1 (CH_2), 42.8 (C), 36.9 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 129.3 (upward), 115.6 (upward), 76.1 (downward), 43.1 (downward), 36.9 (upward), 22.2 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -114.7.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{18}FNO_4S$ $[M+Na]^+$: 326.0838; found: 326.0840.



3-((4-Chlorobenzyl)amino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2j)

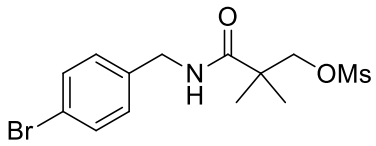
Dark slurry (143 mg, 45%). R_f = 0.60, 50% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.32 (m, 2H), 7.23-7.21 (m, 2H), 6.16 (br s, 1H), 4.44 (d, *J* = 5.6 Hz, 2H), 4.24 (s, 2H), 2.95 (s, 3H), 1.31 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4 (C=O), 133.6 (C), 133.4 (C), 128.9 (CH), 76.1 (CH_2), 43.1 (CH_2), 42.8 (C), 36.9 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.9 (upward), 76.1 (downward), 43.0 (downward), 36.9 (upward), 22.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{18}ClNO_4S$ $[M+Na]^+$: 342.0543; found: 342.0546.



3-((4-Bromobenzyl)amino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2k)

Light brown solid (182 mg, 50%). R_f = 0.50, 55% EtOAc in n-hexane.

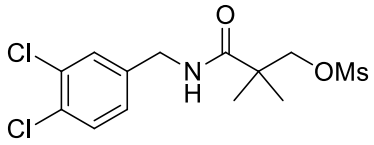
Mp: 52-54 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 6.30 (br s, 1H), 4.39 (d, *J* = 5.6 Hz, 2H), 4.21 (s, 2H), 2.94 (s, 3H), 1.28 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4 (C=O), 137.2 (C), 131.8 (CH), 129.2 (CH), 121.4 (C), 76.1 (CH_2), 43.0 (CH_2), 42.7 (C), 36.9 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 131.9 (upward), 129.2 (upward), 76.1 (downward), 43.1 (downward), 36.9 (upward), 22.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{18}BrNO_4S$ $[M+Na]^+$: 386.0038; found: 386.0041.



3-((3,4-Dichlorobenzyl)amino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2l)

Brown solid (194 mg, 55%). R_f = 0.50, 55% EtOAc in n-hexane.

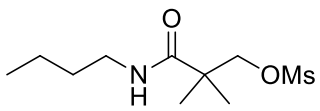
Mp: 75-78 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.0 Hz, 1H), 7.36 (d, *J* = 2.0 Hz, 1H), 7.11 (dd, *J* = 2.0, 8.0 Hz, 1H), 6.31 (br s, 1H), 4.41 (d, *J* = 6.0 Hz, 2H), 4.23 (s, 2H), 2.99 (s, 3H), 1.31 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6 (C=O), 138.5 (C), 132.7 (C), 131.5 (C), 130.7 (CH), 129.4 (CH), 126.8 (CH), 76.0 (CH_2), 42.8 (C), 42.6 (CH_2), 37.0 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 130.7 (upward), 129.4 (upward), 126.8 (upward), 75.9 (downward), 42.6 (downward), 37.0 (upward), 22.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{17}Cl_2NO_4S$ $[M+Na]^+$: 376.0153; found: 376.0157.



3-(Butylamino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2m)

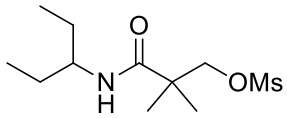
Yellow oil (140 mg, 56%). R_f = 0.55, 50% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.21 (br s, 1H), 4.08 (s, 2H), 3.11-3.08 (m, 2H), 2.90 (s, 3H), 1.38-1.34 (m, 2H), 1.23-1.18 (m, 2H), 1.13 (s, 6H), 0.80-0.77 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4 (C=O), 76.1 (CH_2), 42.5 (C), 39.4 (CH_2), 36.8 (CH_3), 31.3 (CH_2), 22.0 (CH_3), 19.8 (CH_2), 13.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.1 (downward), 39.6 (downward), 36.9 (upward), 31.5 (downward), 22.2 (upward), 20.0 (downward), 13.7 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{17}Cl_2NO_4S$ $[M+H]^+$: 252.1270; found: 252.1283.



2,2-Dimethyl-3-oxo-3-(pentan-3-ylamino)propyl methanesulfonate (2n)

White solid (132 mg, 50%). R_f = 0.55, 50% EtOAc in n-hexane.

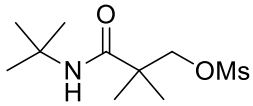
Mp: 70-72 ^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.39 (br s, 1H), 4.23 (s, 2H), 3.84-3.78 (m, 1H), 3.02 (s, 3H), 1.63-1.56 (m, 4H), 1.30 (s, 6H), 0.92 (t, *J* = 7.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1 (C=O), 76.1 (CH_2), 52.3 (CH), 42.7 (C), 36.9 (CH_3), 27.5 (CH_2), 22.4 (CH_3), 10.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.1 (downward), 52.2 (upward), 36.9 (upward), 27.5 (downward), 22.4 (upward), 10.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{11}H_{23}NO_4S$ $[M+Na]^+$: 288.1245; found: 288.1238.



3-(*tert*-Butylamino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2o)

White solid (50 mg, 20%). R_f = 0.45, 50% EtOAc in n-hexane.

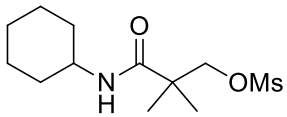
Mp: 42-44 ^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.54 (br s, 1H), 4.16 (s, 2H), 2.99 (s, 3H), 1.34 (s, 9H), 1.22 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6 (C=O), 76.4 (CH_2), 51.4 (C), 43.0 (C), 36.9 (CH_3), 28.6 (CH_3), 22.3 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.4 (downward), 36.9 (upward), 28.6 (upward), 22. (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{11}H_{23}NO_4S$ $[M+Na]^+$: 288.1245; found: 288.1249.



3-(Cyclohexylamino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2p)

White solid (116 mg, 42%). R_f = 0.50, 40% EtOAc in n-hexane.

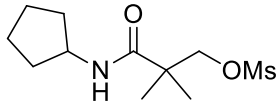
Mp: 77-79 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 5.59 (br s, 1H), 4.22 (s, 2H), 3.79-3.72 (m, 1H), 3.03 (s, 3H), 1.93-1.90 (m, 2H), 1.75-1.71 (m, 2H), 1.66-1.63 (m, 1H), 1.43-1.35 (m, 2H), 1.27 (s, 6H), 1.21-1.15 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4 (C=O), 76.1 (CH_2), 48.5 (CH), 42.5 (C), 36.9 (CH_3), 32.9 (CH_2), 25.5 (CH_2), 24.8 (CH_2), 22.3 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.2 (downward), 48.5 (upward), 36.9 (upward), 32.9 (downward), 25.5 (downward), 24.8 (downward), 22.3 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{23}NO_4S$ $[M+Na]^+$: 300.1245; found: 300.1238.



3-(Cyclopentylamino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2q)

Gray solid (92 mg, 35%). R_f = 0.55, 50% EtOAc in n-hexane.

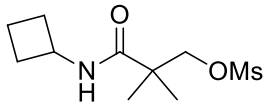
Mp: 68-71 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 5.70 (br s, 1H), 4.20 (s, 2H), 4.19-4.12 (m, 1H), 3.02 (s, 3H), 2.05-1.96 (m, 2H), 1.71-1.58 (m, 2H), 1.41-1.32 (m, 2H), 1.25 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9 (C=O), 76.2 (CH_2), 51.5 (CH), 42.5 (C), 36.9 (CH_3), 33.1 (CH_2), 23.8 (CH_2), 22.3 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.2 (downward), 51.5 (upward), 36.9 (upward), 33.1 (downward), 23.8 (downward), 22.3 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{11}H_{21}NO_4S$ $[M+Na]^+$: 286.1089; found: 286.1098.



3-(Cyclobutylamino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2r)

Light yellow solid (22 mg, 42%). R_f = 0.45, 50% EtOAc in n-hexane.

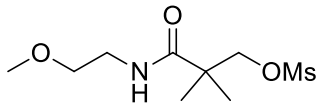
Mp: 97-101 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 5.83 (br s, 1H), 4.37-4.31 (m, 1H), 4.19 (s, 2H), 3.00 (s, 3H), 2.39-2.32 (m, 2H), 1.90-1.80 (m, 2H), 1.77-1.71 (m, 2H), 1.25 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5 (C=O), 76.1 (CH_2), 45.0 (CH), 42.4 (C), 36.9 (CH_3), 31.0 (CH_2), 22.2 (CH_3), 15.1 (CH_2).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.1 (downward), 45.0 (upward), 36.9 (upward), 31.1 (downward), 22.2 (upward), 15.1 (downward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{10}H_{19}NO_4S$ $[M+Na]^+$: 272.0932; found: 272.0928.



3-((2-Methoxyethyl)amino)-2,2-dimethyl-3-oxopropyl methanesulfonate (2s)

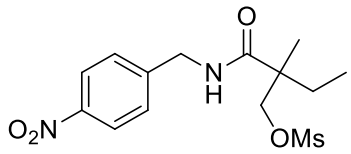
Light brown oil (106 mg, 42%). R_f = 0.30, 80% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.17 (br s, 1H), 4.22 (s, 2H), 3.48-3.43 (m, 2H), 3.37 (s, 3H), 3.02 (s, 3H), 1.28 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4 (C=O), 76.0 (CH_2), 70.9 (CH_2), 58.9 (CH_3), 42.7 (C), 39.5 (CH_2), 36.9 (CH_3), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.0 (downward), 70.9 (downward), 58.8 (upward), 39.5 (downward), 36.9 (upward), 22.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_9H_{19}NO_5S$ $[M+Na]^+$: 276.0882; found: 276.0893.



2-Methyl-2-((4-nitrobenzyl)carbamoyl)butyl methanesulfonate (2t)

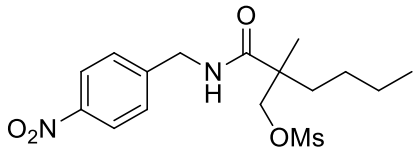
Dark slurry (200 mg, 58%). R_f = 0.50, 60% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.8 Hz, 2H), 7.44 (d, *J* = 8.8 Hz, 2H), 6.45 (br s, 1H), 4.63-4.52 (m, 2H), 4.41 (d, *J* = 9.2 Hz, 1H), 4.11 (d, *J* = 9.2 Hz, 1H), 2.98 (s, 3H), 1.81-1.72 (m, 1H), 1.57-1.48 (m, 1H), 1.33 (s, 3H), 0.92 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2 (C=O), 147.3 (C), 145.8 (C), 128.1 (CH), 123.9 (CH), 75.1 (CH_2), 46.7 (C), 43.0 (CH_2), 36.9 (CH_3), 28.7 (CH_2), 18.5 (CH_3), 8.4 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.1 (upward), 123.9 (upward), 75.1 (downward), 43.0 (downward), 36.9 (upward), 28.7 (downward), 18.5 (upward), 8.4 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{14}H_{20}N_2O_6S$ $[M+Na]^+$: 367.0940; found: 367.0941.



2-Methyl-2-((4-nitrobenzyl)carbamoyl)hexyl methanesulfonate (2u)

Brown solid (178 mg, 48%). R_f = 0.50, 50% EtOAc in n-hexane.

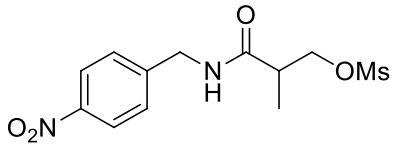
Mp: 89-92 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.8 Hz, 2H), 7.43 (d, *J* = 8.8 Hz, 2H), 6.50 (br s, 1H), 4.62-4.39 (m, 2H), 4.40 (d, *J* = 9.2 Hz, 1H), 4.08 (d, *J* = 9.6 Hz, 1H), 2.97 (s, 3H), 1.71-1.63 (m, 1H), 1.48-1.40 (m, 1H), 1.33 (s, 3H), 1.31-1.25 (m, 3H), 1.21-1.16 (m, 1H), 0.88 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3 (C=O), 147.2 (C), 145.9 (C), 128.1 (CH), 123.9 (CH), 75.4 (CH_2), 46.3 (C), 43.0 (CH_2), 36.9 (CH_3), 35.7 (CH_2), 26.1 (CH_2), 23.0 (CH_2), 18.9 (CH_3), 13.9 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.1 (upward), 123.9 (upward), 75.3 (downward), 43.0 (downward), 36.9 (upward), 35.7 (downward), 26.1 (downward), 23.0 (downward), 19.0 (upward), 13.9 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{16}H_{24}N_2O_6S$ $[M+Na]^+$: 395.1253; found: 395.1246.



2-Methyl-3-((4-nitrobenzyl)amino)-3-oxopropyl methanesulfonate (2v)

Brown solid (25 mg, 8%). R_f = 0.40, 80% EtOAc in n-hexane.

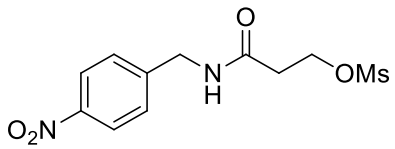
Mp: 112-114 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 6.45 (br s, 1H), 4.65-4.49 (m, 2H), 4.39-4.26 (m, 2H), 3.00 (s, 3H), 2.87-2.78 (m, 1H), 1.25 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.0 (C=O), 147.3 (C), 145.6 (C), 128.1 (CH), 123.9 (CH), 71.5 (CH_2), 42.9 (CH_2), 41.1 (CH), 37.2 (CH_3), 13.9 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.1 (upward), 123.9 (upward), 71.4 (downward), 42.9 (downward), 41.1 (upward), 37.2 (upward), 13.9 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{16}N_2O_6S$ $[M+Na]^+$: 339.0627; found: 339.0622.



3-((4-Nitrobenzyl)amino)-3-oxopropyl methanesulfonate (2w)

Light yellow solid (9 mg, 3%). R_f = 0.25, 80% EtOAc in n-hexane.

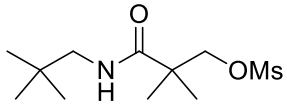
Mp: 109-112 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 6.30 (br s, 1H), 4.60 (s, 2H), 4.58 (t, *J* = 6.4 Hz, 2H), 3.04 (s, 3H), 2.73 (t, *J* = 5.6 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 169.0 (C=O), 147.4 (C), 145.4 (C), 128.2 (CH), 124.0 (CH), 66.2 (CH_2), 43.0 (CH_2), 37.3 (CH_3), 36.2 (CH_2).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.2 (upward), 124.0 (upward), 66.1 (downward), 43.0 (downward), 37.3 (upward), 36.3 (downward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{11}H_{14}N_2O_6S$ $[M+Na]^+$: 325.0470; found: 325.0464.



2,2-Dimethyl-3-(neopentylamino)-3-oxopropyl methanesulfonate (2x)

White solid (156mg, 59%). R_f = 0.60, 50% EtOAc in n-hexane.

Mp: 60-62 °C.

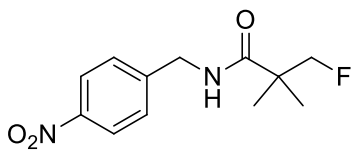
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.81 (br s, 1H), 4.24 (s, 2H), 3.11 (d, *J* = 6.4 Hz, 2H), 3.02 (s, 3H), 1.31 (s, 3H), 0.93 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3 (C=O), 76.0 (CH_2), 50.5 (CH_2), 42.8 (C), 36.9 (CH_3), 32.0 (C), 27.1 (CH_3), 22.3 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 76.1 (downward), 50.6 (downward), 36.9 (upward), 27.1 (upward), 22.4 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{11}H_{23}NO_4S$ $[M+Na]^+$: 288.1245; found: 288.1249.

(3) *β*-Fluoramides



***N*-(4-Nitrobenzyl)-3-fluoro-2,2-dimethylpropanamide (3a)**

Gray solid (165 mg, 65%). R_f = 0.50, 50% EtOAc in n-hexane.

Mp: 72-75 ^{o}C.

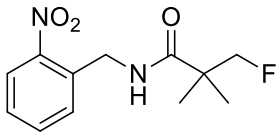
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.8 Hz, 2H), 7.39 (d, *J* = 8.8 Hz, 2H), 6.43 (br s, 1H), 4.55 (d, *J* = 6.0 Hz, 2H), 4.41 (d, *J* = 47.6 Hz, 2H), 1.25 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.6 (C=O), 147.4 (C), 146.2 (C), 128.1 (CH), 124.1 (CH), 89.3 (CH_2), 43.1 (CH_2), 21.7 (CH_3), 21.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.0 (upward), 124.0 (upward), 89.1 (downward), 42.9 (downward), 21.5 (upward), 21.4 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.0.

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}FN_2O_3$ $[M+Na]^+$: 277.0964; found: 277.0958.



***N*-(2-Nitrobenzyl)-3-fluoro-2,2-dimethylpropanamide (3b)**

Brown oil (152 mg, 60%). R_f = 0.50, 50% EtOAc in n-hexane.

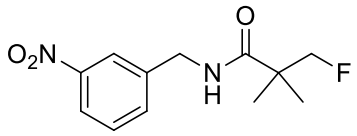
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08-8.06 (m, 1H), 7.65-7.63 (m, 2H), 7.50-7.47 (m, 1H), 6.76 (br s, 1H), 4.71 (d, *J* = 6.4 Hz, 2H), 4.38 (d, *J* = 47.6 Hz, 2H), 1.22 (d, *J* = 2.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.3 (C=O), 148.4 (C), 134.1 (CH), 133.6 (C), 132.1 (CH), 128.7 (CH), 125.1 (CH), 89.0 (CH_2), 43.3 (C), 41.4 (CH_2), 21.4 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 134.1 (upward), 132.1 (upward), 128.7 (upward), 125.1 (upward), 89.0 (downward), 41.4 (downward), 21.4 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}FN_2O_3$ $[M+Na]^+$: 277.0964; found: 277.0968.



***N*-(3-Nitrobenzyl)-3-fluoro-2,2-dimethylpropanamide (3c)**

Brown oil (172 mg, 68%). R_f = 0.50, 50% EtOAc in n-hexane.

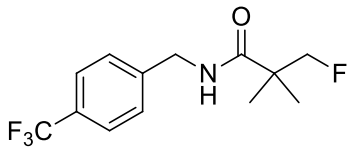
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.0 Hz, 1H), 8.11 (s, 1H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 8.0 Hz, 1H), 6.39 (br s, 1H), 4.56 (d, *J* = 6.0 Hz, 2H), 4.42 (d, *J* = 47.6 Hz, 2H), 1.26 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.6 (C=O), 148.3 (C), 140.8 (C), 133.4 (CH), 129.6 (CH), 122.3 (CH), 121.9 (CH), 89.1 (CH_2), 43.3 (C), 42.7 (CH_2), 21.4 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 133.5 (upward), 129.7 (upward), 122.4 (upward), 122.1 (upward), 89.1 (downward), 42.7 (downward), 21.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}FN_2O_3$ $[M+Na]^+$: 277.0964; found: 277.0974.



3-Fluoro-2,2-dimethyl-*N*-(4-(trifluoromethyl)benzyl)propanamide (3d)

Light yellow oil. R_f = 0.50, 33% EtOAc in n-hexane.

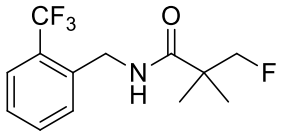
^1H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 6.31 (br s, 1H), 4.55 (d, *J* = 5.6 Hz, 2H), 4.44 (d, *J* = 47.6 Hz, 2H), 1.28 (d, *J* = 1.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.2 (C=O), 142.4 (C), 129.7 (C), 127.6 (CH), 125.7 (CH), 124.1 (CF_3), 89.2 (CH_2), 43.3 (C), 43.1 (CH_2), 21.5 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 127.6 (upward), 125.7 (upward), 89.2 (downward), 43.1 (downward), 21.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.5, -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{15}F_4NO$ $[M+Na]^+$: 300.0987; found: 300.0982.



3-Fluoro-2,2-dimethyl-*N*-(2-(trifluoromethyl)benzyl)propanamide (3e)

White solid. R_f = 0.55, 33% EtOAc in n-hexane.

Mp: 60-62 °C.

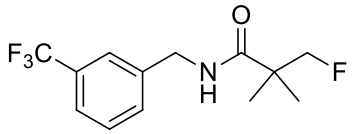
^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.0 Hz, 1H), 7.55-7.51 (m, 2H), 7.42-7.38 (m, 1H), 6.31 (br s, 1H), 4.66 (d, *J* = 6.0 Hz, 2H), 4.41 (d, *J* = 47.6 Hz, 2H), 1.24 (d, *J* = 2.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1 (C=O), 136.6 (C), 132.4 (CH), 130.4 (CH), 128.1 (C), 127.6 (CH), 126.0 (CH), 124.4 (CF_3), 89.1 (CH_2), 43.2 (C), 40.3 (CH_2), 21.4 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 132.4 (upward), 130.4 (upward), 127.6 (upward), 126.0 (upward), 89.1 (downward), 40.3 (downward), 21.4 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -59.6, -74.2.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{15}F_4NO$ $[M+Na]^+$: 300.0987; found: 300.0994.



3-Fluoro-2,2-dimethyl-*N*-(3-(trifluoromethyl)benzyl)propanamide (3f)

Yellow oil. R_f = 0.50, 33% EtOAc in n-hexane.

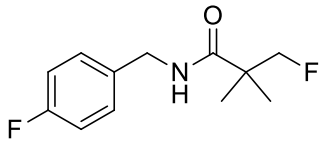
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.52 (m, 2H), 7.48-7.47 (m, 2H), 6.31 (br s, 1H), 4.55 (d, *J* = 6.0 Hz, 2H), 4.44 (d, *J* = 47.6 Hz, 2H), 1.28 (d, *J* = 1.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.2 (C=O), 139.4 (C), 130.9 (C), 130.8 (CH), 129.2 (CH), 124.3 (CH), 124.1 (CF_3), 89.1 (CH_2), 43.3 (C), 43.0 (CH_2), 21.5 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 130.8 (upward), 129.2 (upward), 124.3 (upward), 89.1 (downward), 43.0 (downward), 21.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.7, -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{15}F_4NO$ $[M+Na]^+$: 300.0987; found: 300.0981.



***N*-(4-Fluorobenzyl)-3-fluoro-2,2-dimethylpropanamide (3i)**

Light brown oil. R_f = 0.50, 33% EtOAc in n-hexane.

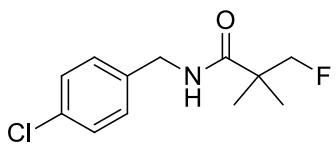
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.23 (m, 2H), 7.05-7.01 (m, 2H), 6.21 (br s, 1H), 4.44 (d, *J* = 5.6 Hz, 2H), 4.42 (d, *J* = 47.6 Hz, 2H), 1.26 (d, *J* = 1.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0 (C=O), 162.2 (CF), 134.1 (C), 129.2 (CH), 115.5 (CH), 89.2 (CH_2), 43.2 (C), 42.9 (CH_2), 21.5 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 129.2 (upward), 115.5 (upward), 89.2 (downward), 42.9 (downward), 21.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1, -115.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}F_2NO$ $[M+Na]^+$: 250.1019; found: 250.1014.



***N*-(4-Chlorobenzyl)-3-fluoro-2,2-dimethylpropanamide (3j)**

Light brown oil. R_f = 0.50, 40% EtOAc in n-hexane.

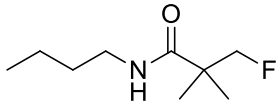
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.31 (m, 2H), 7.22-7.20 (m, 2H), 6.22 (br s, 1H), 4.44 (d, *J* = 5.6 Hz, 2H), 4.42 (d, *J* = 47.6 Hz, 2H), 1.26 (d, *J* = 2.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1 (C=O), 136.9 (C), 133.3 (C), 128.8 (CH), 89.2 (CH_2), 43.2 (C), 42.9 (CH_2), 21.5 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.9 (upward), 89.2 (downward), 42.9 (downward), 21.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}F_2NO$ $[M+Na]^+$: 250.1019; found: 250.1014.



***N*-Butyl-3-fluoro-2,2-dimethylpropanamide (3m)**

Colorless oil. R_f = 0.70, 40% EtOAc in n-hexane.

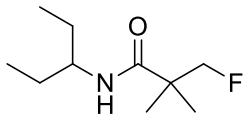
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.87 (br s, 1H), 4.39 (d, *J* = 47.6 Hz, 2H), 3.30-3.25 (m, 2H), 1.53-1.47 (m, 2H), 1.39-1.33 (m, 2H), 1.22 (d, *J* = 1.6 Hz, 6H), 0.94 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0 (C=O), 89.2 (CH_2), 43.2 (C), 39.3 (CH_2), 31.5 (CH_2), 21.5 (CH_3), 20.0 (CH_2), 13.8 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 89.2 (downward), 39.4 (downward), 31.6 (downward), 21.5 (upward), 20.0 (downward), 13.8 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_9H_{18}FNO$ $[M+Na]^+$: 198.1270; found: 198.1259.



3-Fluoro-2,2-dimethyl-N-(pentan-3-yl)propanamide (3n)

White solid. R_f = 0.60, 40% EtOAc in n-hexane.

Mp: 90-93 ^{o}C.

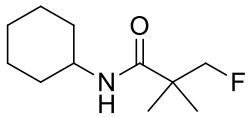
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.54 (br s, 1H), 4.40 (d, *J* = 47.6 Hz, 2H), 3.86-3.79 (m, 1H), 1.63-1.52 (m, 2H), 1.43-1.32 (m, 2H), 1.24 (d, *J* = 2.0 Hz, 6H), 0.90 (t, *J* = 7.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.8 (C=O), 89.3 (CH_2), 51.8 (CH), 43.2 (CH), 27.5 (CH_2), 21.7 (CH_3), 21.6 (CH_2), 10.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 89.3 (downward), 51.8 (upward), 27.5 (downward), 21.7 (upward), 21.6 (downward), 10.2 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{10}H_{20}FNO$ $[M+Na]^+$: 212.1427; found: 212.1421.



***N*-Cyclohexyl-3-fluoro-2,2-dimethylpropanamide (3p)**

White solid. R_f = 0.70, 40% EtOAc in n-hexane.

Mp: 88-91 °C.

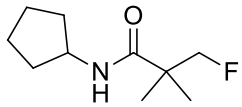
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.72 (br s, 1H), 4.38 (d, *J* = 47.6 Hz, 2H), 3.80-3.58 (m, 1H), 1.92-1.88 (m, 2H), 1.73-1.68 (m, 2H), 1.42-1.32 (m, 2H), 1.20 (d, 6H), 1.18-1.10 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1 (C=O), 89.3 (CH_2), 48.1 (CH), 43.0 (C), 33.0 (CH_2), 25.6 (CH_2), 24.8 (CH_2), 21.6 (CH_3), 21.5 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 89.3 (downward), 48.1 (upward), 33.0 (downward), 25.6 (downward), 24.8 (downward), 21.6 (upward), 21.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{11}H_{20}FNO$ $[M+Na]^+$: 224.1427; found: 224.1421.



***N*-Cyclopentyl-3-fluoro-2,2-dimethylpropanamide (3q)**

White solid. R_f = 0.60, 40% EtOAc in n-hexane.

Mp: 97-100 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 (br s, 1H), 4.38 (d, *J* = 47.6 Hz, 2H), 4.25-4.16 (m, 1H), 2.05-1.97 (m, 2H), 1.71-1.59 (m,4H), 1.41-1.33 (m, 2H), 1.21 (d, *J* = 1.6 Hz, 6H).

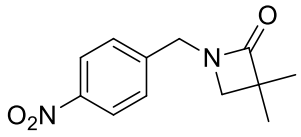
^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6 (C=O), 89.2 (CH_2), 51.2 (CH), 43.0 (C), 33.1 (CH_2), 23.8 (CH_2), 21.6 (CH_3), 21.5 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 89.3 (downward), 51.2 (upward), 33.1 (downward), 23.8 (downward), 21.6 (upward), 21.5 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{10}H_{18}FNO$ $[M+Na]^+$: 210.1270; found: 210.1263.

(4) *β*-Lactams



3,3-Dimethyl-1-(4-nitrobenzyl)azetidin-2-one (4a)

Light yellow solid (150 mg, 64%). R_f = 0.50, 50% EtOAc in n-hexane.

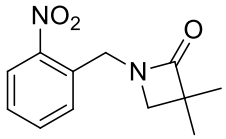
Mp: 73-76 ^oC.

^1H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.8 Hz, 2H), 7.41 (d, *J* = 8.8 Hz, 2H), 4.47 (s, 2H), 2.99 (s, 2H), 1.32 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1 (C=O), 147.5 (C), 143.5 (C), 128.7 (CH), 124.1 (CH), 54.3 (CH_2), 51.6 (C), 45.1 (CH_2), 21.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.7 (upward), 124.1 (upward), 54.3 (downward), 45.1 (downward), 21.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{14}N_2O_3$ $[M+Na]^+$: 257.0902; found: 257.0893.



3,3-Dimethyl-1-(2-nitrobenzyl)azetidin-2-one (4b)

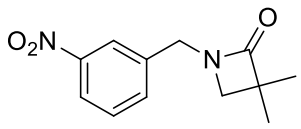
Brown oil (140 mg, 60%). R_f = 0.50, 50% EtOAc in n-hexane.

^1H NMR (400 MHz, $CDCl_3$) δ 8.06 (dd, *J* =1.2, 8.0 Hz, 1H), 7.69-7.65 (m, 1H), 7.54-7.48 (m, 2H), 4.74 (s, 2H), 3.12 (s, 2H), 1.35 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4 (C=O), 148.5 (C), 133.9 (CH), 131.8 (C), 130.7 (CH), 128.7 (CH), 125.1 (CH), 55.4 (CH_2), 51.4 (C), 42.8 (CH_2), 21.3 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 133.9 (upward), 130.7 (upward), 128.7 (upward), 125.1 (upward), 55.4 (downward), 42.8 (downward), 21.3 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{14}N_2O_3$ $[M+Na]^+$: 257.0902; found: 257.0908.



3,3-Dimethyl-1-(3-nitrobenzyl)azetidin-2-one (4c)

Brown oil (164 mg, 67%). R_f = 0.50, 50% EtOAc in n-hexane.

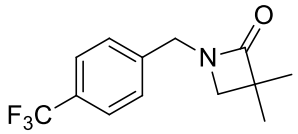
^1H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.0 Hz, 1H), 8.07 (s, 1H), 7.60 (d, *J* = 7.6

Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 1H), 4.47 (s, 2H), 3.01 (s, 2H), 1.31 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1 (C=O), 148.5 (C), 138.3 (C), 134.0 (CH), 130.0 (CH), 122.7 (CH), 122.6 (CH), 54.2 (CH_2), 51.5 (C), 44.9 (CH_2), 21.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 134.0 (upward), 130.0 (upward), 122.8 (upward), 122.7 (upward), 54.3 (downward), 44.9 (downward), 21.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{14}N_2O_3$ $[M+Na]^+$: 257.0902; found: 257.0902.



3,3-Dimethyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (4d)

Brown oil. R_f = 0.50, 33% EtOAc in n-hexane.

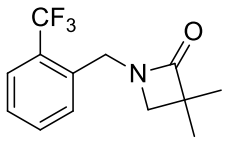
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 4.45 (s, 2H), 2.99 (s, 2H), 1.34 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0 (C=O), 140.1 (C), 130.0 (C), 128.3 (CH), 125.8 (CH), 122.7 (CF_3), 54.0 (CH_2), 51.3 (C), 45.2 (CH_2), 21.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.3 (upward), 125.8 (upward), 54.1 (downward), 45.2 (downward), 21.2 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.6.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{14}F_3NO$ $[M+Na]^+$: 280.0925; found: 280.0929.



3,3-Dimethyl-1-(2-(trifluoromethyl)benzyl)azetidin-2-one (4e)

Colorless oil. R_f = 0.55, 33% EtOAc in n-hexane.

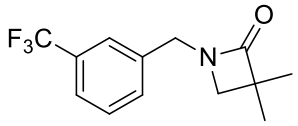
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.0 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.49 (d, *J* = 6.07.6 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 4.58 (s, 2H), 2.98 (s, 2H), 1.32 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2 (C=O), 134.8 (C), 132.5 (CH), 130.4 (CH), 128.5 (C), 127.7 (CH), 126.0 (CH), 124.2 (CF_3), 54.3 (CH_2), 51.1 (C), 41.9 (CH_2), 21.1 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 132.4 (upward), 130.4 (upward), 127.7 (upward), 126.0 (upward), 54.3 (downward), 41.9 (downward), 21.1 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -59.1.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{14}F_3NO$ $[M+Na]^+$: 280.0925; found: 280.0932.



3,3-Dimethyl-1-(3-(trifluoromethyl)benzyl)azetidin-2-one (4f)

Brown slurry. R_f = 0.50, 33% EtOAc in n-hexane.

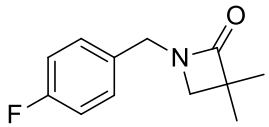
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.6 Hz, 1H), 7.53-7.45 (m, 3H), 4.45 (s, 2H), 2.99 (s, 2H), 1.34 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1 (C=O), 137.1 (C), 131.3 (CH), 131.0 (C), 129.4 (CH), 124.6 (CH), 123.9 (CF_3), 54.0 (CH_2), 51.3 (C), 45.2 (CH_2), 21.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 131.3 (upward), 129.4 (upward), 124.6 (upward), 54.0 (downward), 45.2 (downward), 21.2 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.7.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{14}F_3NO$ $[M+Na]^+$: 280.0925; found: 280.0931.



1-(4-Fluorobenzyl)-3,3-dimethylazetidin-2-one (4i)

Brown oil. R_f = 0.50, 33% EtOAc in n-hexane.

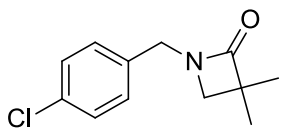
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.20 (m, 2H), 7.07-7.03 (t, *J* = 8.8 Hz, 2H), 4.35 (s, 2H), 2.94 (s, 2H), 1.31 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9 (C=O), 162.3 (CF), 131.7 (C), 129.7 (CH), 115.7 (CH), 53.7 (CH_2), 51.0 (C), 44.9 (CH_2), 21.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 129.7 (upward), 115.7 (upward), 53.7 (downward), 44.9 (downward), 21.2 (upward).

^{19}F NMR (376 MHz, $CDCl_3$) δ -114.7.

HRMS (ESI-Q/TOF MS) Calcd for $C_{13}H_{17}Cl_2NO_4S$ $[M+H]^+$: 208.1138; found: 208.1145.



1-(4-Chlorobenzyl)-3,3-dimethylazetidin-2-one (4j)

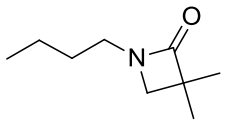
Brown oil. R_f = 0.50, 40% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.4 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 4.34 (s, 2H), 2.94 (s, 2H), 1.30 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0 (C=O), 134.4 (C), 133.5 (C), 129.4 (CH), 129.0 (CH), 53.8 (CH_2), 51.1 (C), 45.0 (CH_2), 21.1 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 129.4 (upward), 129.0 (upward), 53.8 (downward), 45.0 (downward), 21.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{14}ClNO$ $[M+Na]^+$: 246.0662; found: 246.0658.



1-Butyl-3,3-dimethylazetidin-2-one (4m)

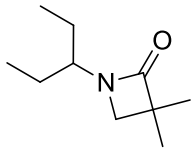
Colorless oil. R_f = 0.70, 20% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13-4.06 (m, 1H), 3.02 (s, 2H), 1.86-1.82 (m, 2H), 1.71-1.59 (m, 6H), 1.29 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9 (C=O), 54.1 (CH_2), 50.3 (C), 41.0 (CH), 29.7 (CH_2), 21.3 (CH_3), 20.1 (CH_2), 13.7 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 54.2 (downward), 41.0 (downward), 29.7 (downward), 21.3 (upward), 20.1 (downward), 13.7 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_9H_{17}NO$ $[M+Na]^+$: 178.1208; found: 178.1195.



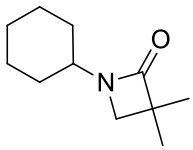
3,3-Dimethyl-1-(pentan-3-yl)azetidin-2-one (4n)

Colorless oil. R_f = 0.60, 40% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.52-3.45 (m, 1H), 2.97 (s, 2H), 1.56-1.39 (m, 4H), 1.33 (s, 6H), 0.95 (t, *J* = 8.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6 (C=O), 54.1 (CH), 50.7 (CH_2), 49.2 (C), 25.8 (CH_2), 21.6 (CH_3), 11.0 (CH_3).

HRMS (ESI-Q/TOF MS) Calcd for $C_{10}H_{19}NO$ $[M+Na]^+$: 192.1364; found: 192.1360.



1-Cyclohexyl-3,3-dimethylazetidin-2-one (4p)

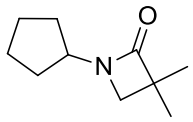
Colorless oil. R_f = 0.70, 40% EtOAc in n-hexane.

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.59-3.52 (m, 1H), 3.01 (s, 2H), 1.86-1.83 (m, 2H), 1.79-1.76 (m, 2H), 1.67-1.61 (m, 2H), 1.35-1.31 (m, 3H), 1.29 (s, 6H), 1.18-1.11 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2 (C=O), 51.5 (CH_2), 50.1 (CH), 49.0 (CH), 30.7 (CH_2), 25.3 (CH_2), 24.9 (CH_2), 21.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 51.5 (downward), 50.1 (upward), 30.7 (downward), 25.3 (downward), 24.9 (downward), 21.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{11}H_{19}NO$ $[M+Na]^+$: 204.1364; found: 204.1356.



1-Cyclopentyl-3,3-dimethylazetidin-2-one (4q)

Colorless oil. R_f = 0.60, 40% EtOAc in n-hexane.

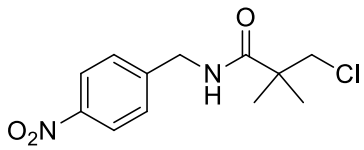
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13-4.06 (m, 1H), 3.02 (s, 2H), 1.86-1.82 (m, 2H), 1.71-1.59 (m, 6H), 1.29 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5 (C=O), 52.3 (CH_2), 52.0 (CH), 48.9 (C), 30.1 (CH_2), 24.1 (CH_2), 22.2 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 52.3 (downward), 52.0 (upnward), 30.1 (downward), 24.1 (downward), 21.2 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{10}H_{17}NO$ $[M+Na]^+$: 190.1208; found: 190.1200.

(5) Other halogenated amides



3-Chloro-2,2-dimethyl-*N*-(4-nitrobenzyl)propanamide (5a)

White solid. R_f = 0.55, 50% EtOAc in n-hexane.

Mp: 79-83 ^{o}C.

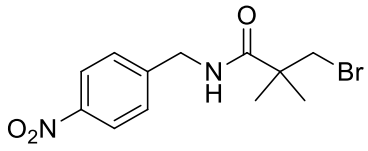
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 6.39 (br s, 1H), 4.58 (d, *J* = 6.0 Hz, 2H), 3.67 (s, 2H), 1.36 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1 (C=O), 147.2 (C), 145.9 (C), 128.1 (CH), 123.9 (CH), 52.8 (CH_2), 44.4 (C), 43.0 (CH_2), 23.6 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.1 (upward), 123.9 (upward), 52.8 (downward),

43.0 (downward), 23.6 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}ClN_2O_3$ $[M+Na]^+$: 293.0669; found: 293.0682.



3-Bromo-2,2-dimethyl-*N*-(4-nitrobenzyl)propanamide (6a)

White solid. R_f = 0.55, 50% EtOAc in n-hexane.

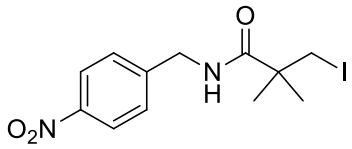
Mp: 88-90 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.8 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 6.39 (br s, 1H), 4.57 (d, *J* = 6.4 Hz, 2H), 3.56 (s, 2H), 1.39 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0 (C=O), 147.2 (C), 145.9 (C), 128.2 (CH), 123.8 (CH), 43.9 (C), 43.1 (CH_2), 42.3 (CH_2), 24.4 (CH_3).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.2 (upward), 123.9 (upward), 43.1 (downward), 42.2 (downward), 24.4 (upward).

HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}BrN_2O_3$ $[M+Na]^+$: 337.0164; found: 337.0163.



3-Iodo-2,2-dimethyl-*N*-(4-nitrobenzyl)propanamide (7a)

White cystal. R_f = 0.55, 50% EtOAc in n-hexane.

Mp: 105-108 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.8 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 6.20 (br s, 1H), 4.56 (d, *J* = 6.0 Hz, 2H), 3.39 (s, 2H), 1.40 (s, 6H).

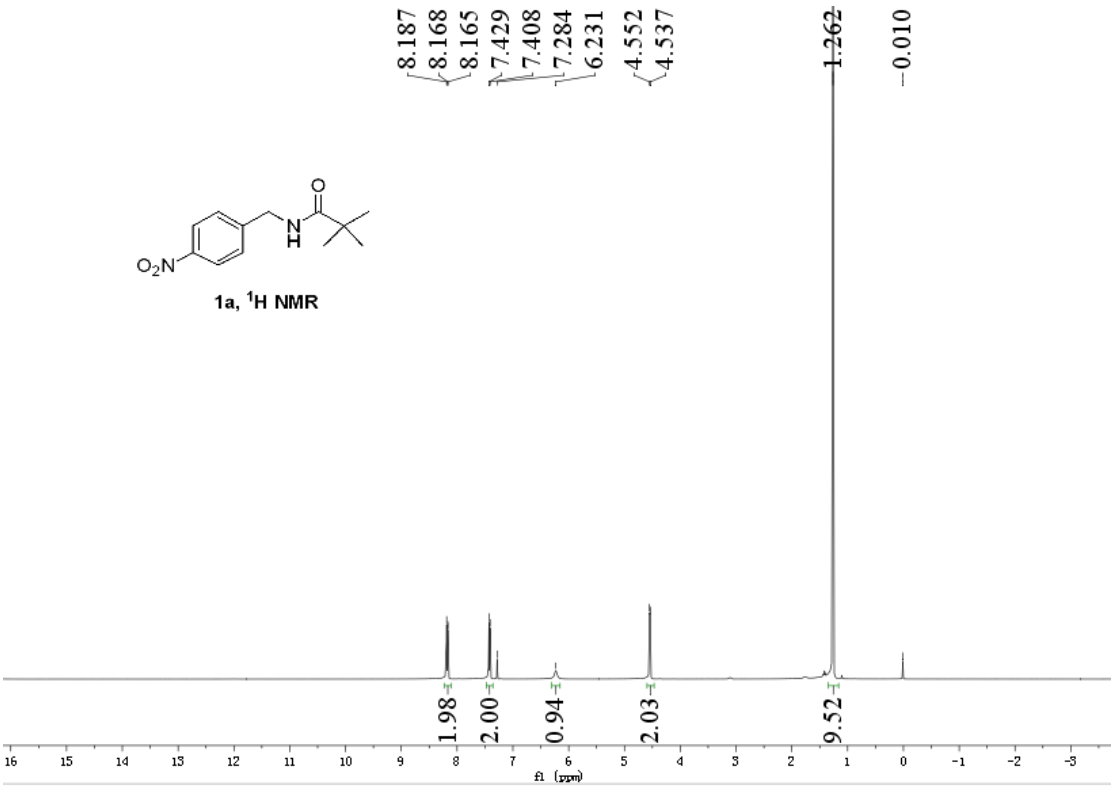
^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1 (C=O), 147.5 (C), 145.9 (C), 128.6 (CH), 124.1 (CH), 43.4 (C), 43.3 (CH_2), 25.8 (CH_3), 17.7 (CH_2).

DEPT-135 (100 MHz, $CDCl_3$) δ 128.4 (upward), 123.9 (upward), 43.1 (downward), 25.6 (upward), 17.6 (downward).

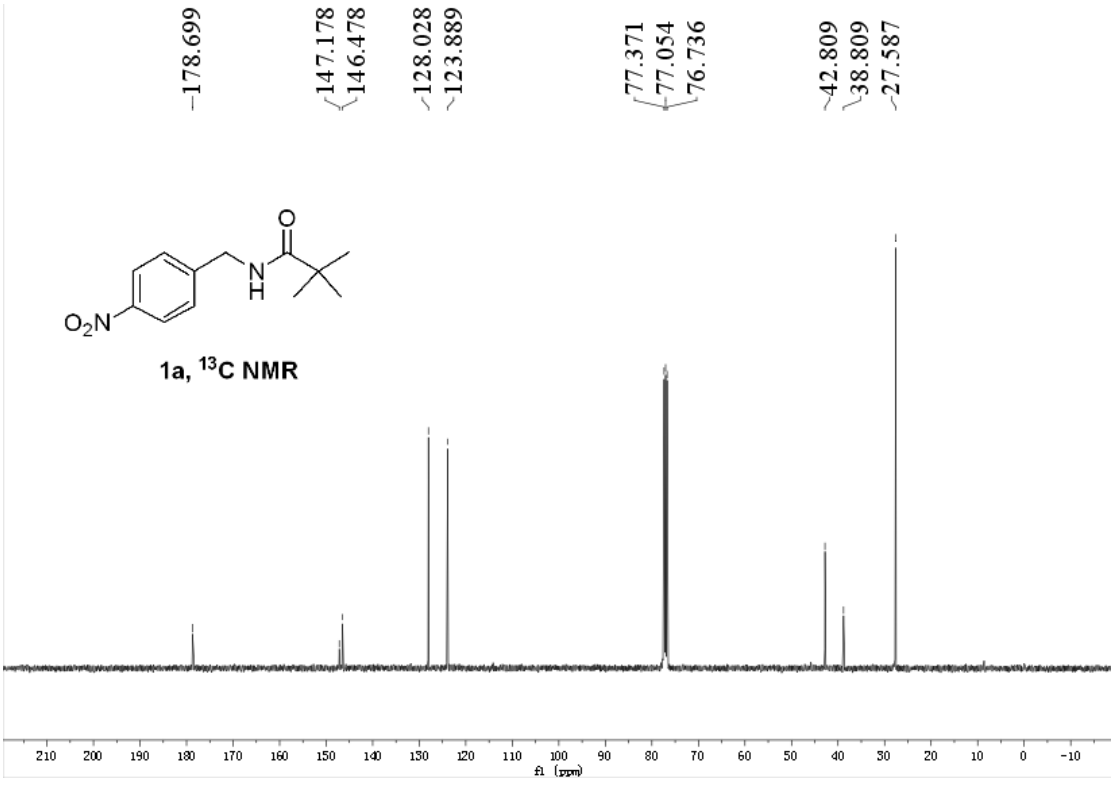
HRMS (ESI-Q/TOF MS) Calcd for $C_{12}H_{15}IN_2O_3$ $[M+Na]^+$: 385.0025; found: 385.0036.

2. NMR Spectra

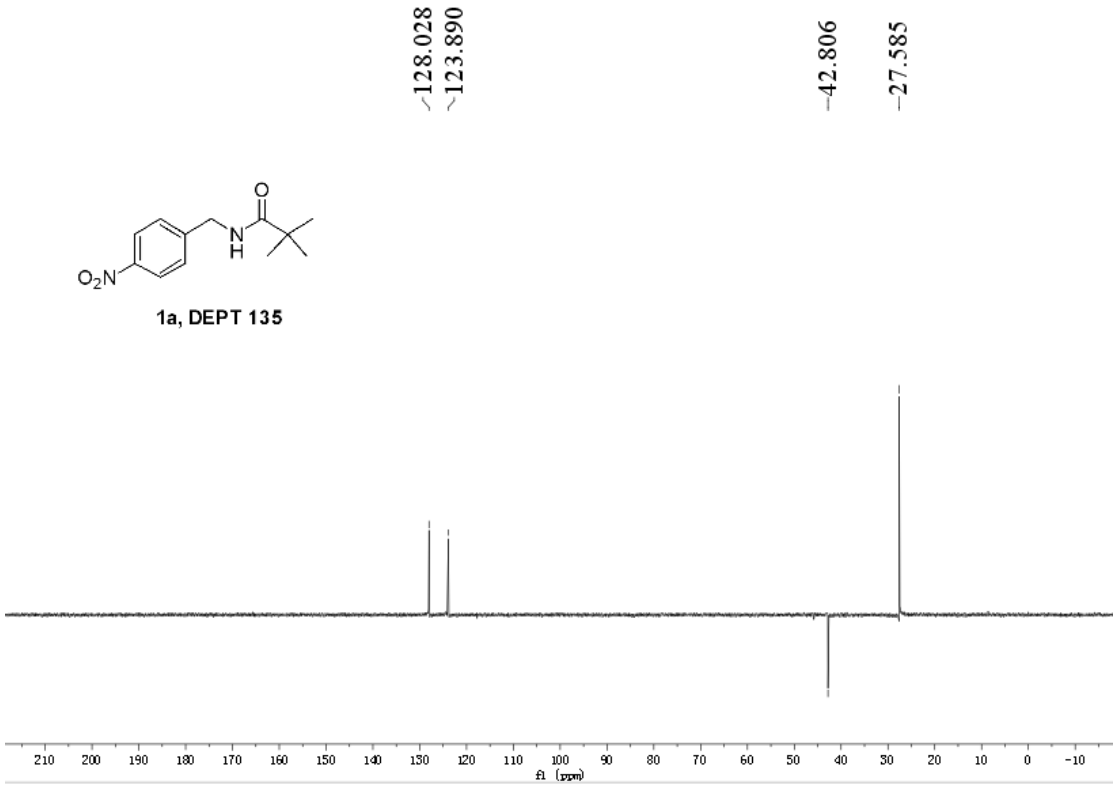
(1) The amide substrates



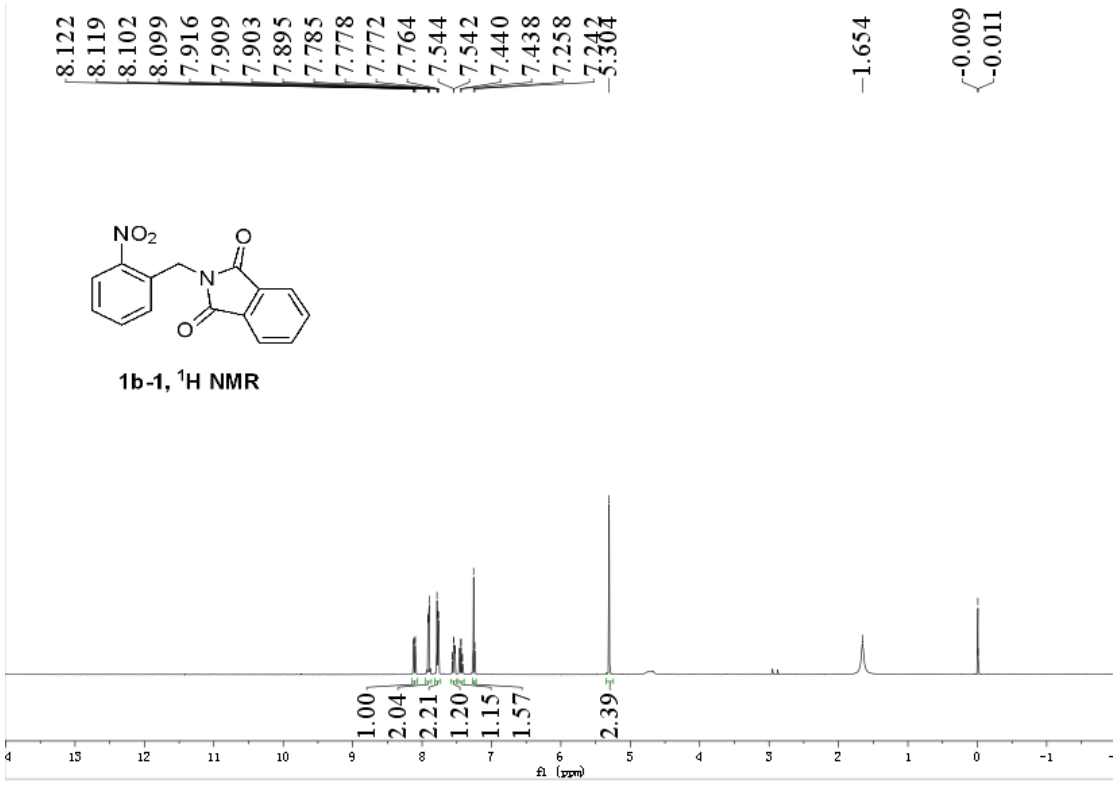
1a, ^{1}H NMR



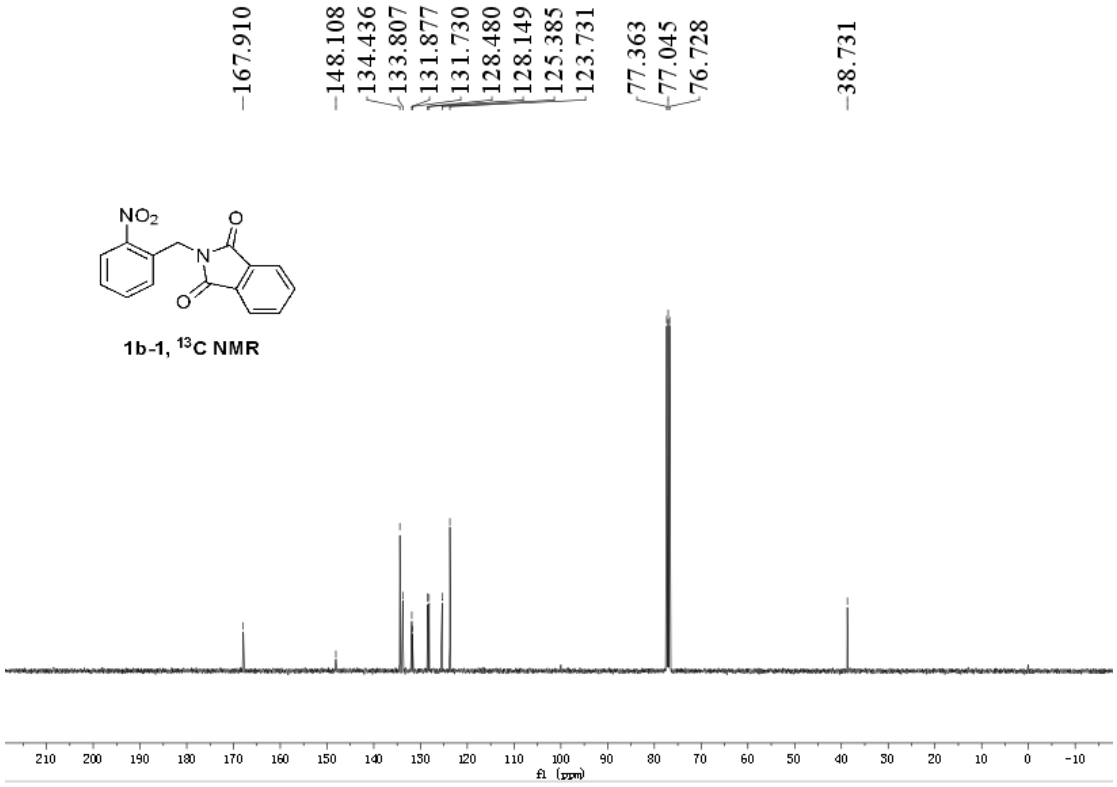
1a, ^{13}C NMR



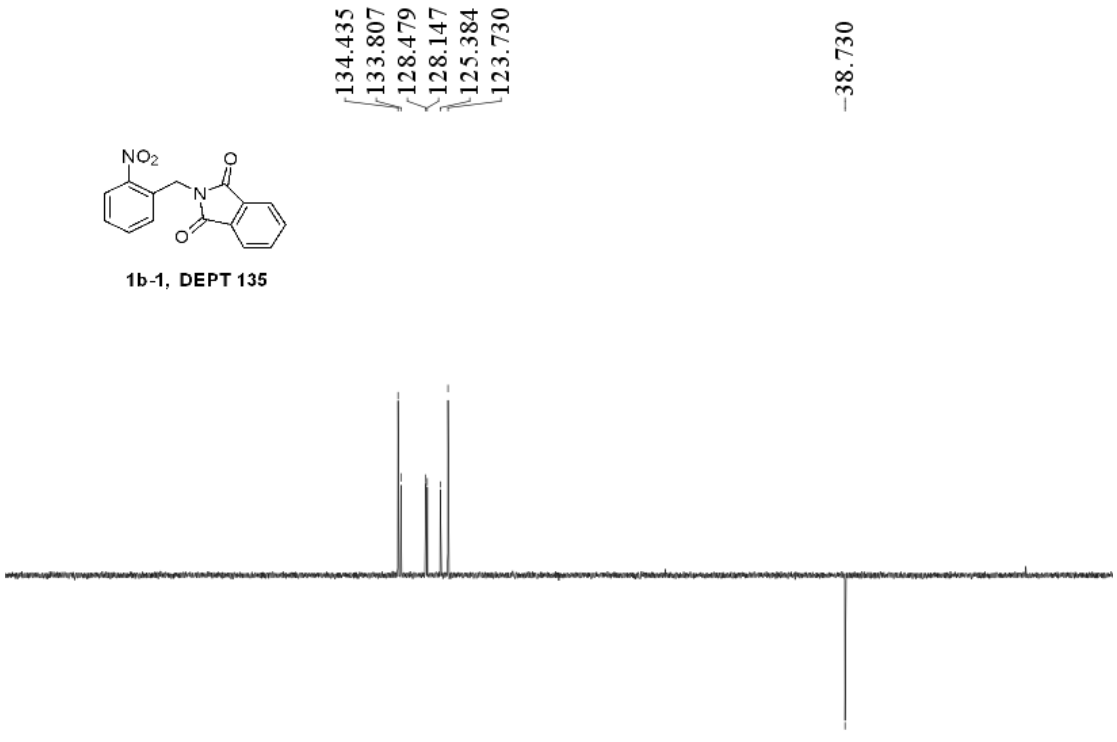
1a, DEPT 135



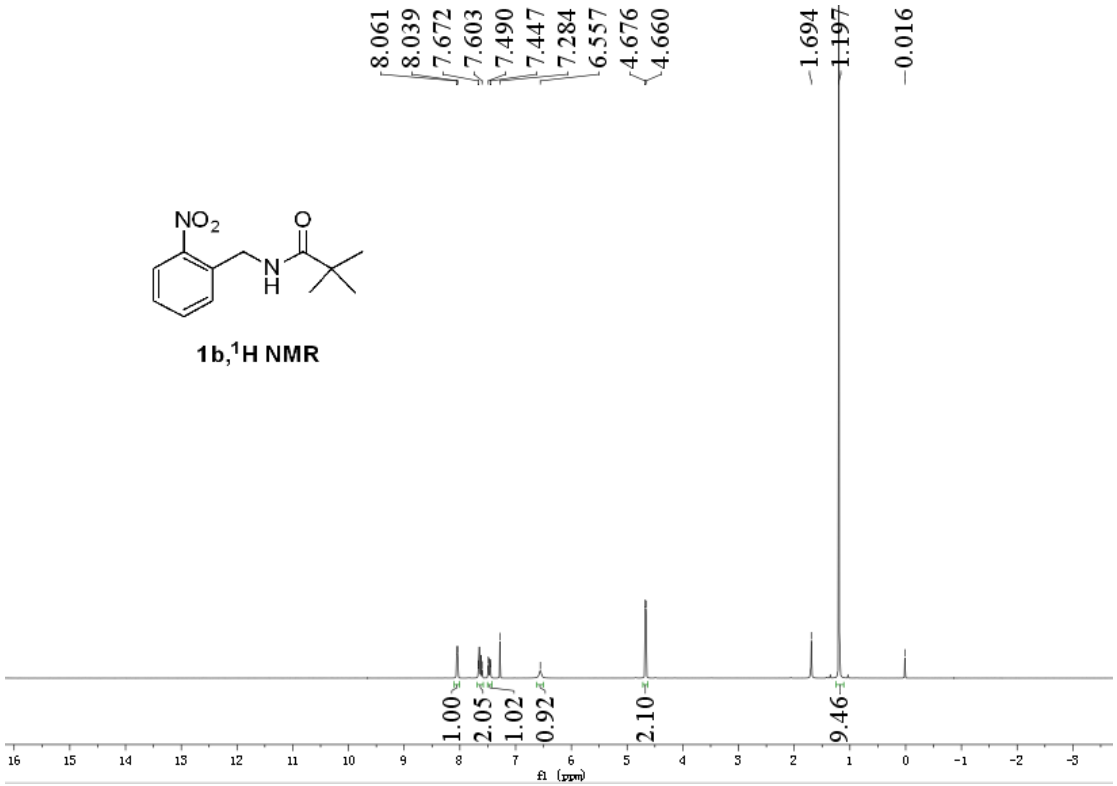
1b-1, ^{1}H NMR

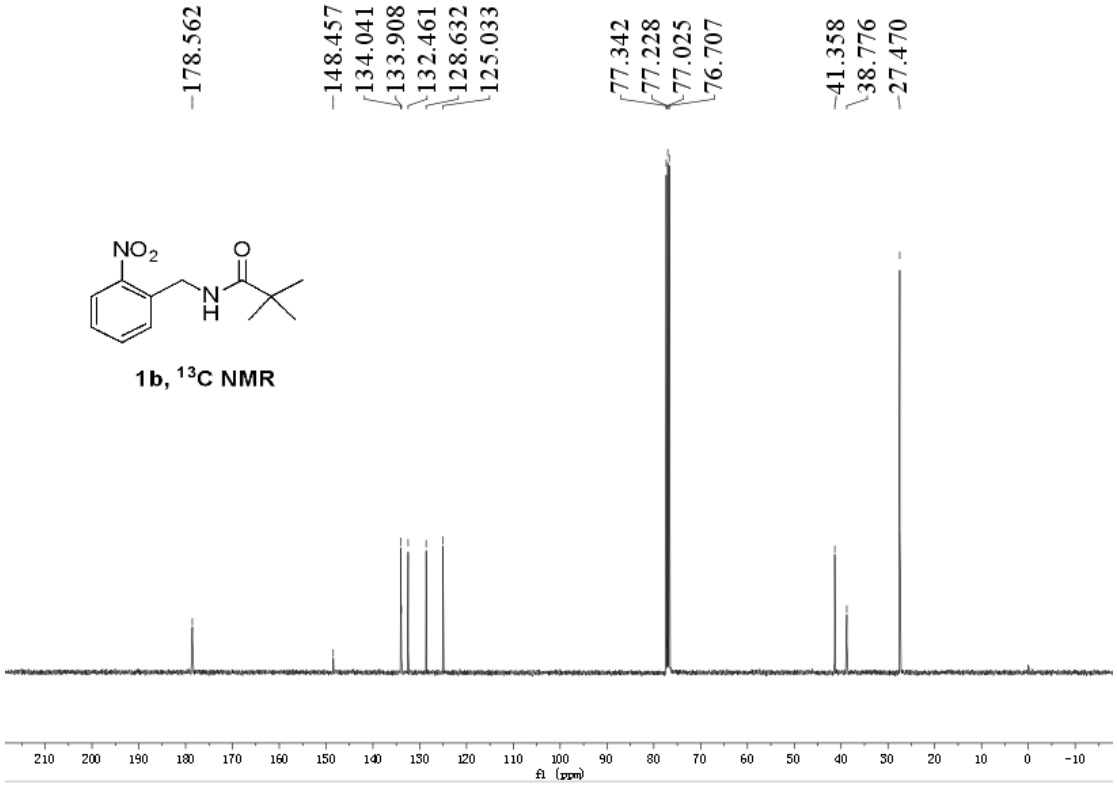


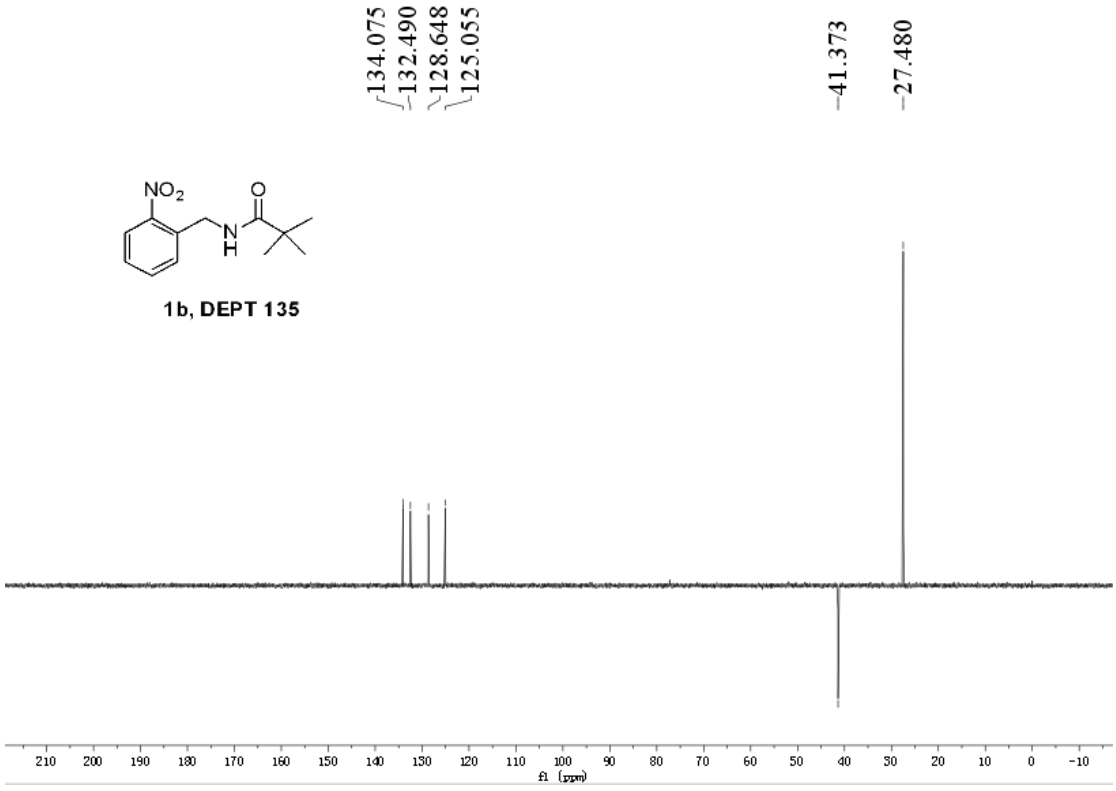
1b-1, ^{13}C NMR



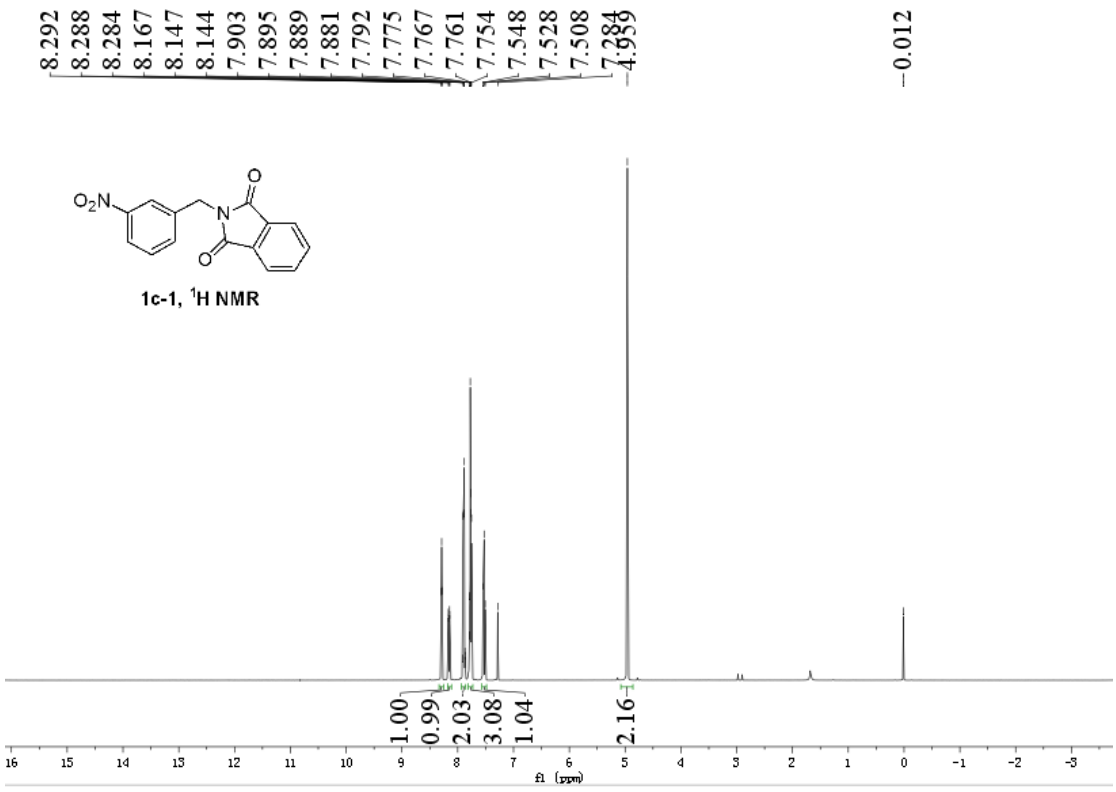
1b-1, DEPT 135


8.061
8.039
7.672
7.603
7.490
7.447
7.284
6.557
4.676
4.660
1.694
1.197
0.016
NO2
O
N
H
1b,1H NMR
1.00
2.05
1.02
0.92
2.10
9.46
f1 (ppm)

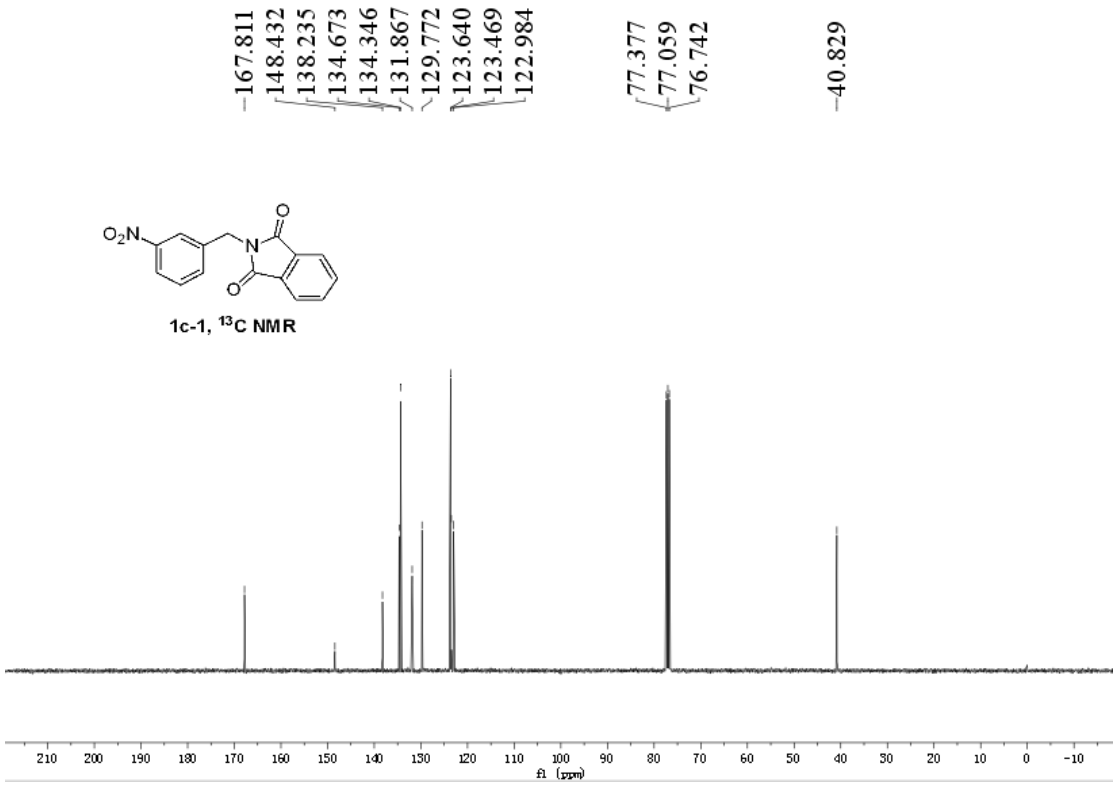

178.562
148.457
134.041
133.908
132.461
128.632
125.033
77.342
77.228
77.025
76.707
41.358
38.776
27.470
NO2
O
N
H
1b, 13C NMR
f1 (ppm)

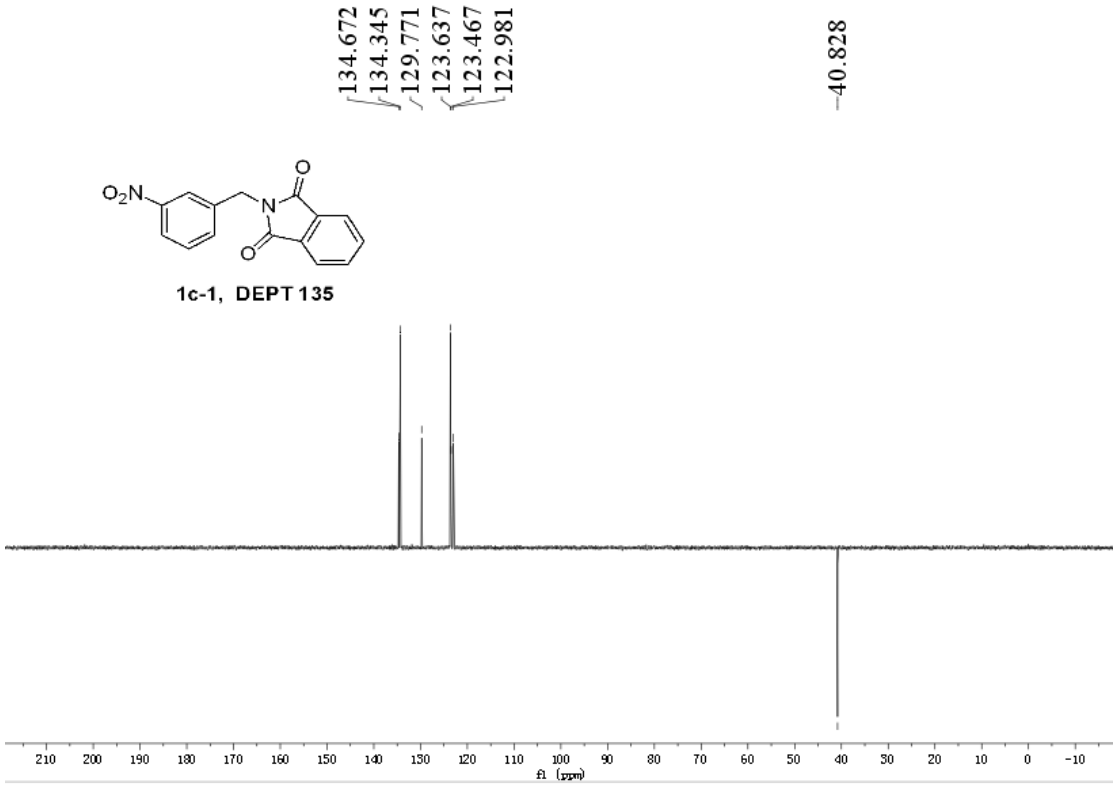


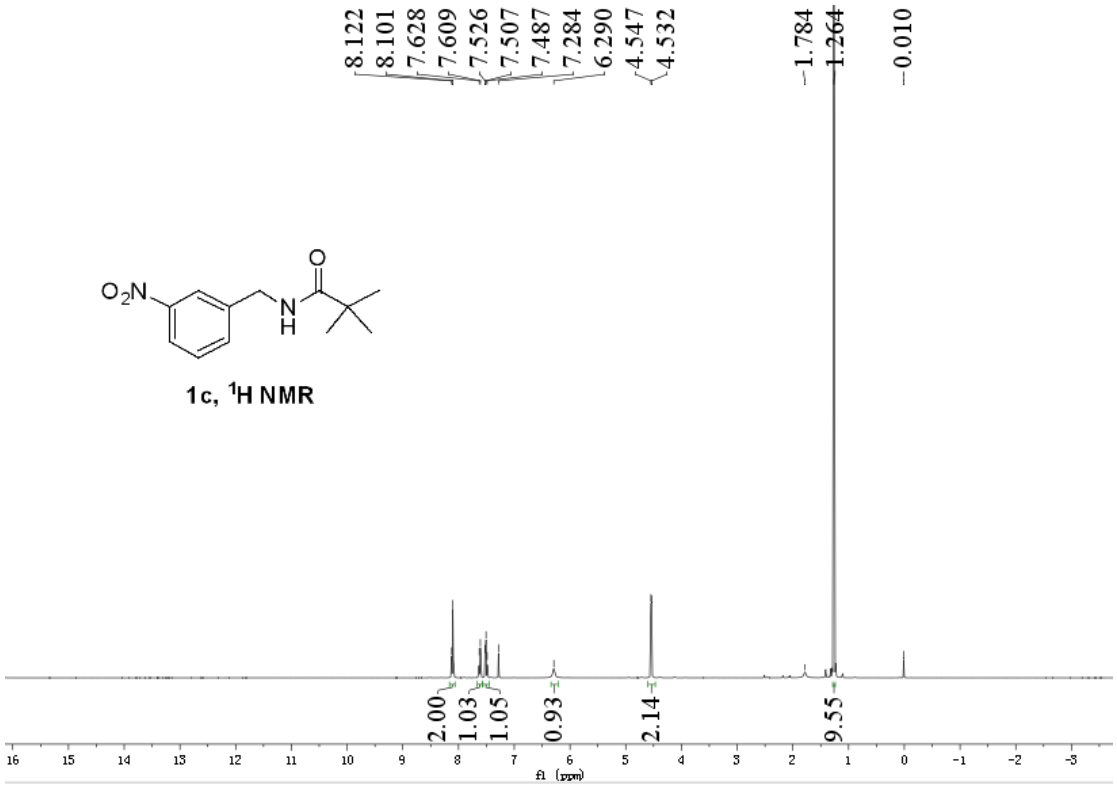
1b, DEPT 135



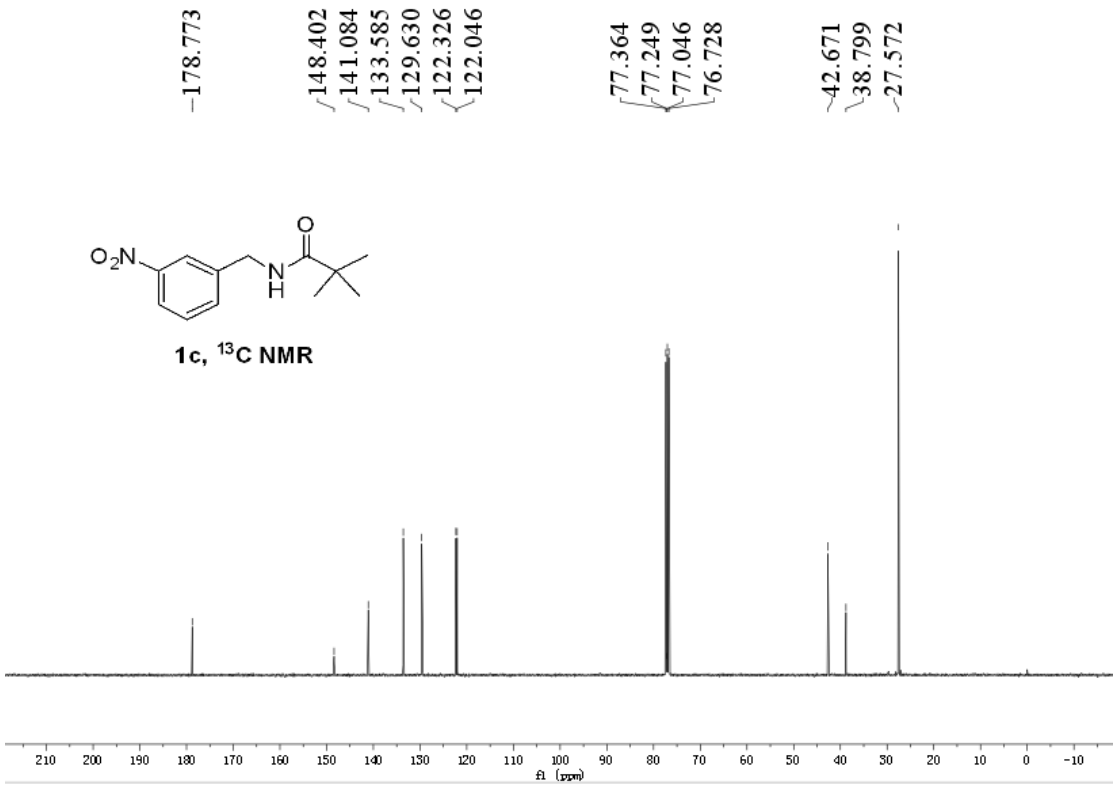
1c-1, ^{1}H NMR


167.811
148.432
138.235
134.673
134.346
131.867
129.772
123.640
123.469
122.984
77.377
77.059
76.742
40.829
O2N
O
N
O
1c-1, 13C NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

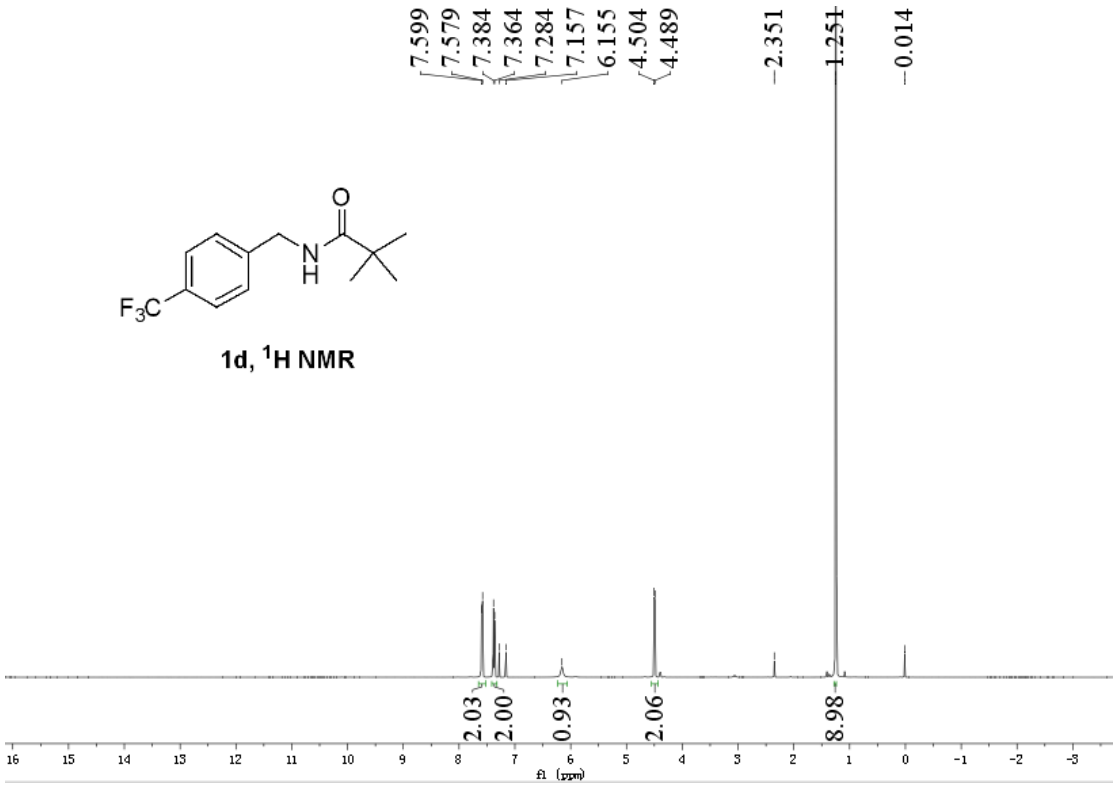

134.672
134.345
129.771
123.637
123.467
122.981
40.828
O2N
O
N
O
1c-1, DEPT 135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



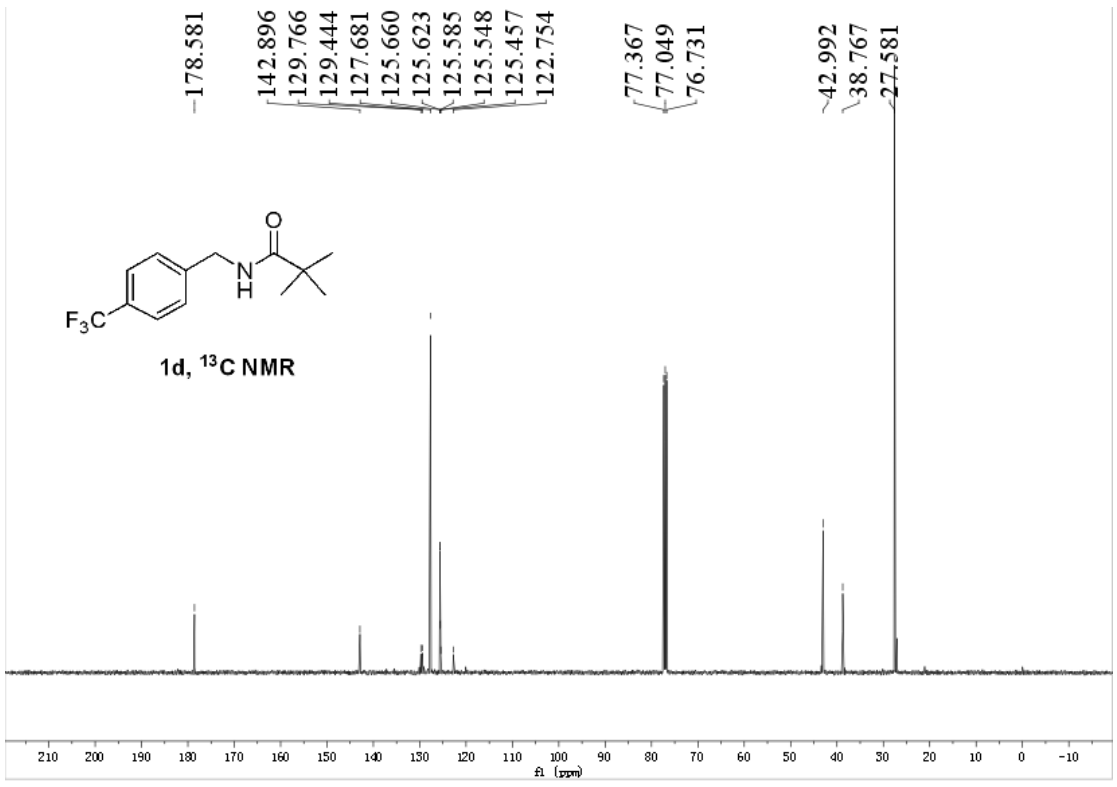
1c, ^{1}H NMR



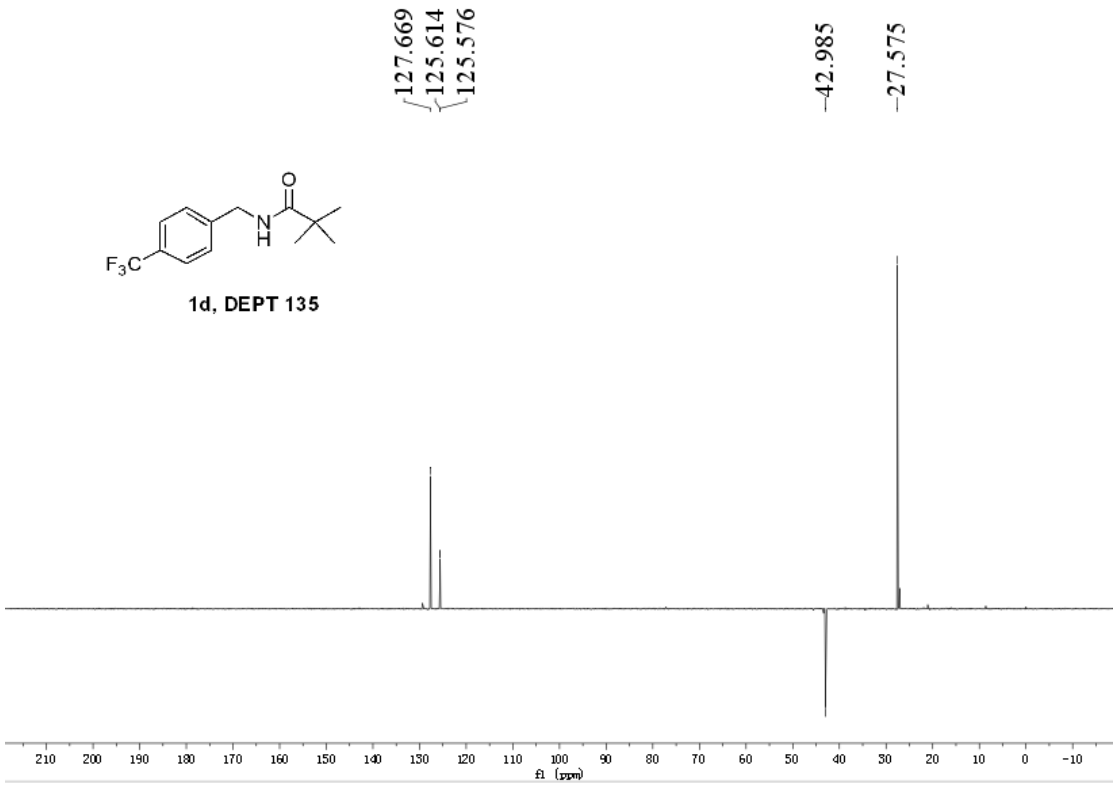
1c, ^{13}C NMR

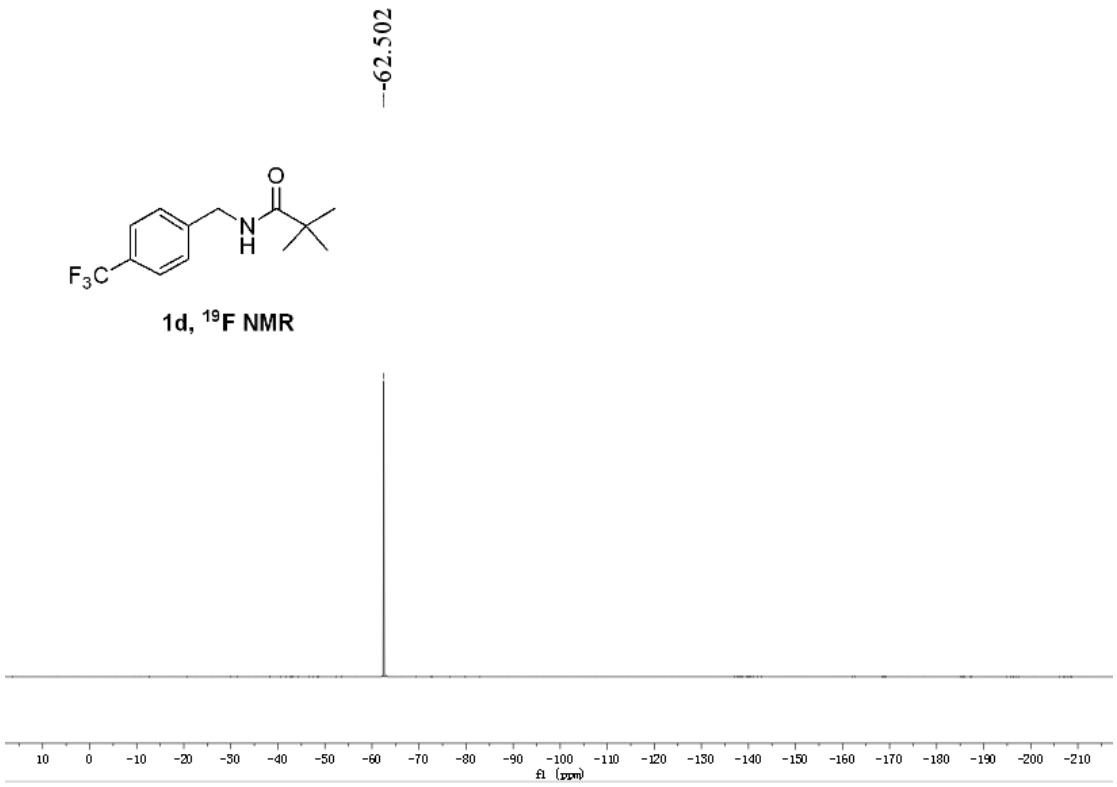


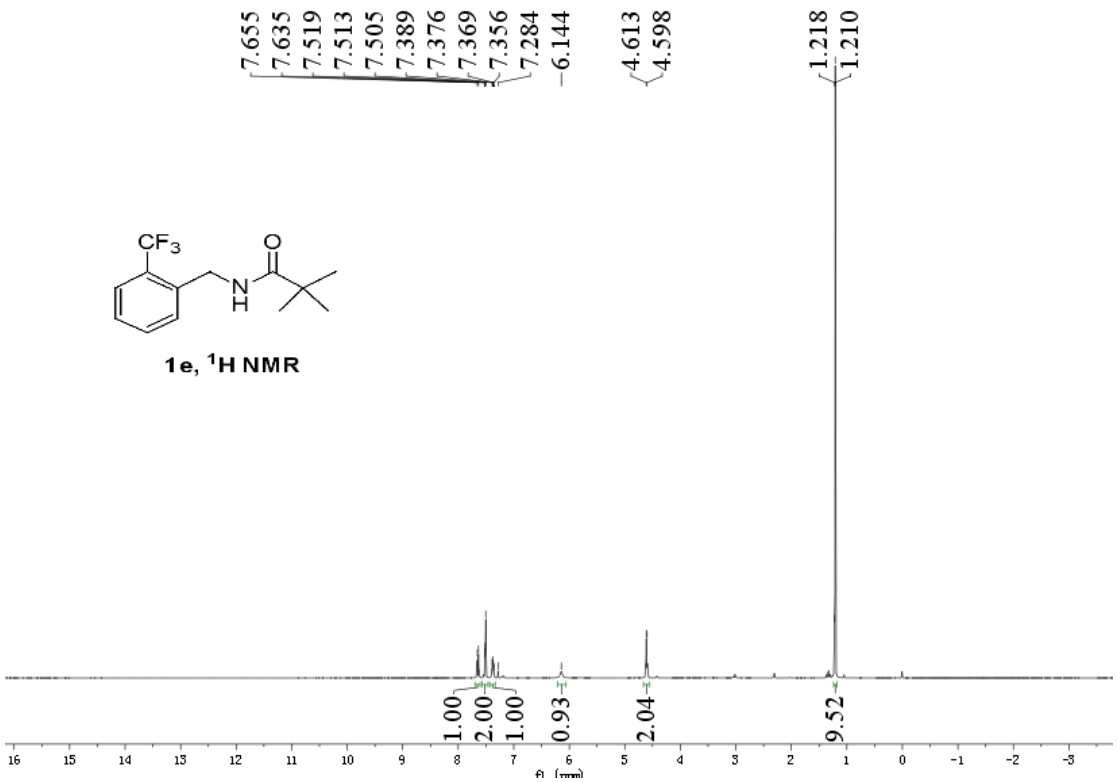
1d, ^{1}H NMR



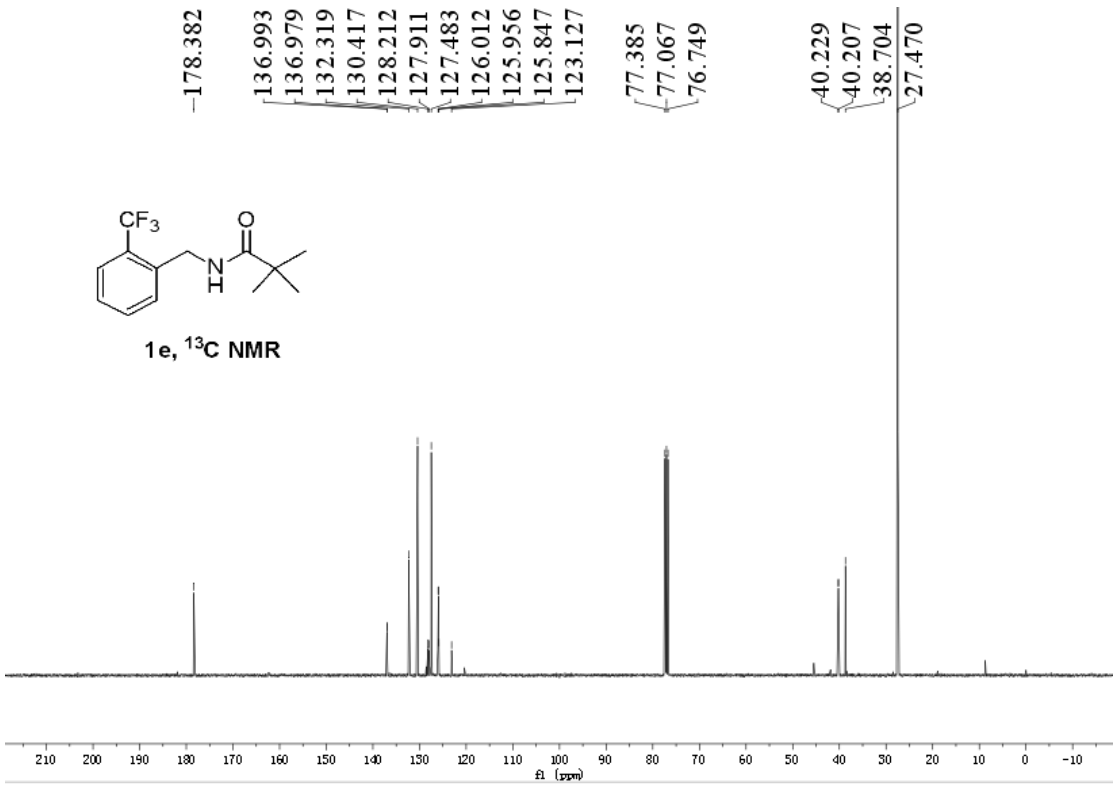
1d, ^{13}C NMR


127.669
125.614
125.576
42.985
27.575
F3C
O
N
H
1d, DEPT 135
f1 (ppm)

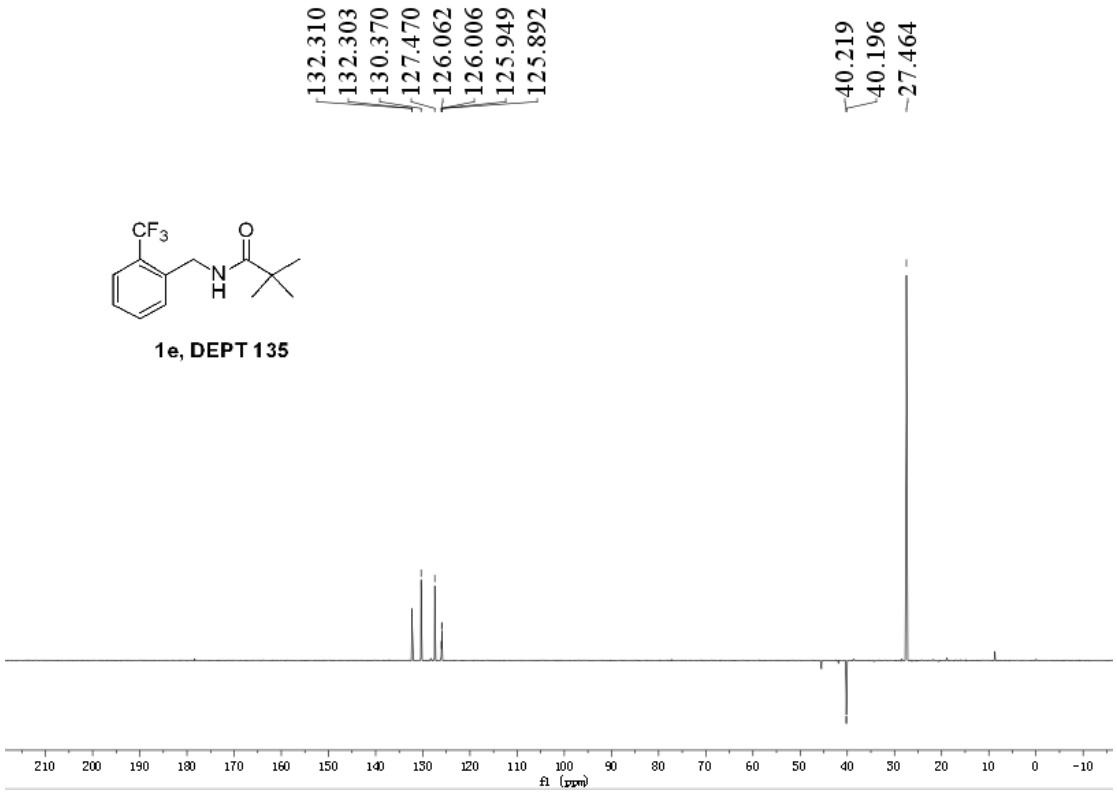

-62.502
F3C
O
N
H
1d, 19F NMR
f1 (ppm)

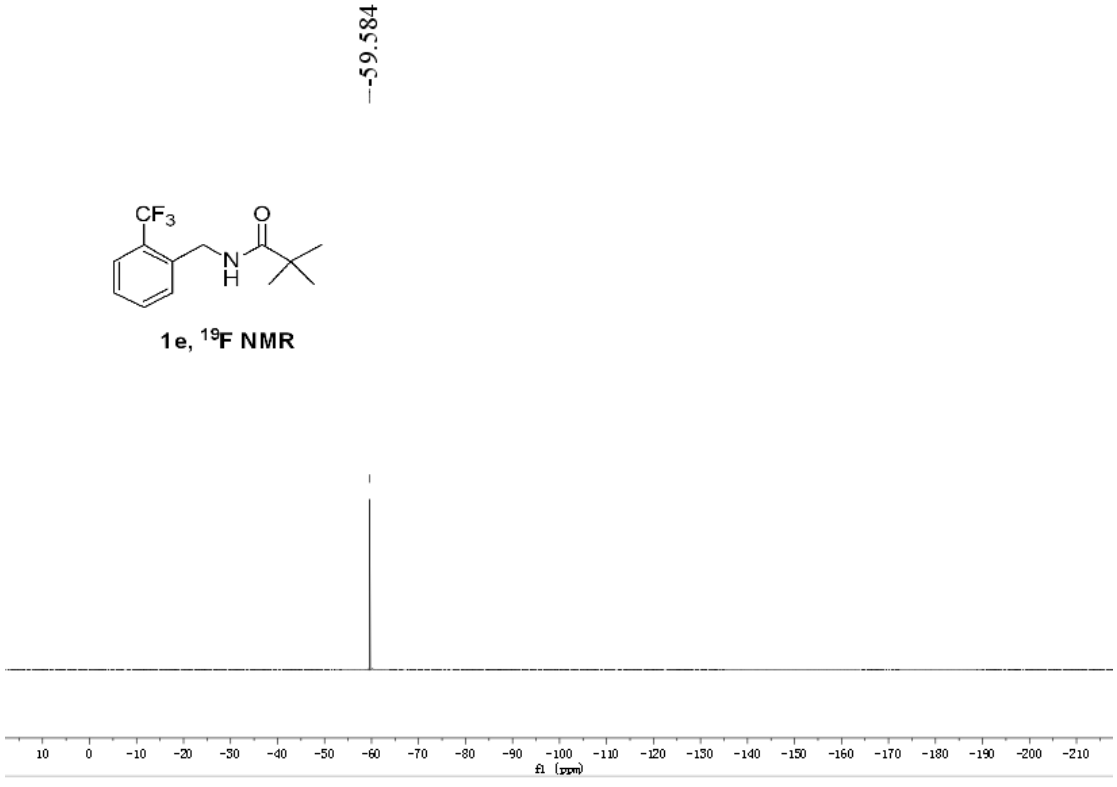


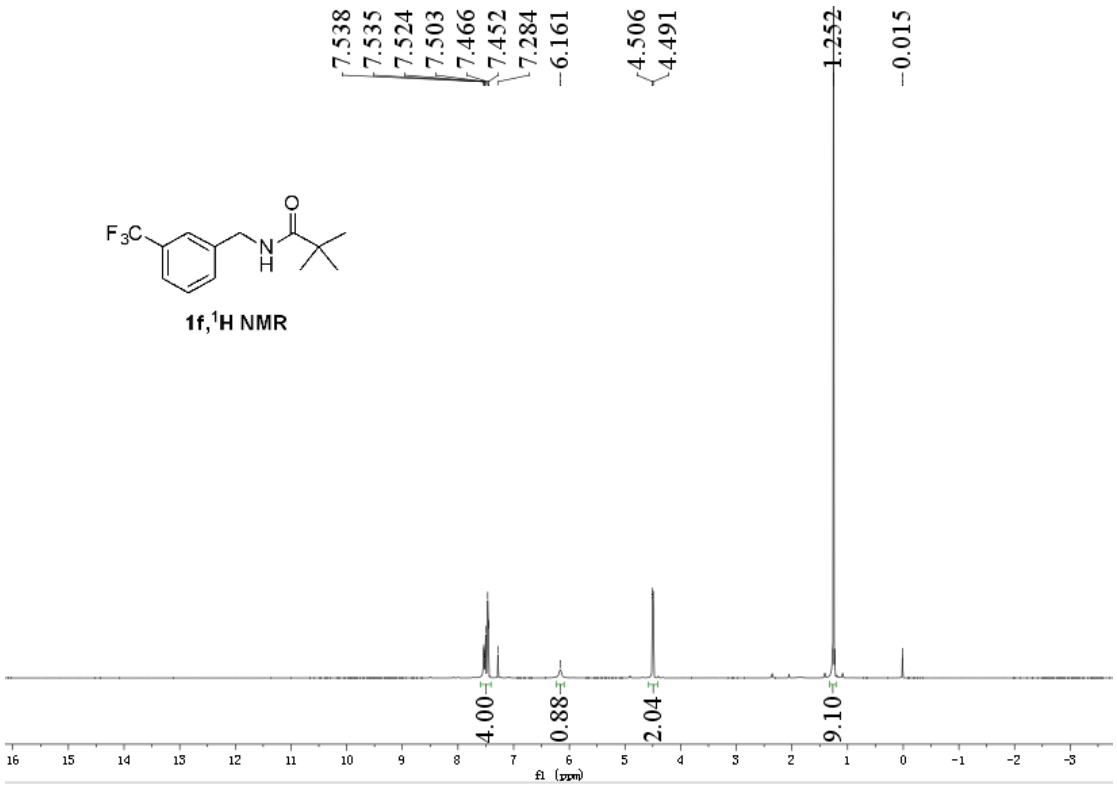
1e, ^{1}H NMR



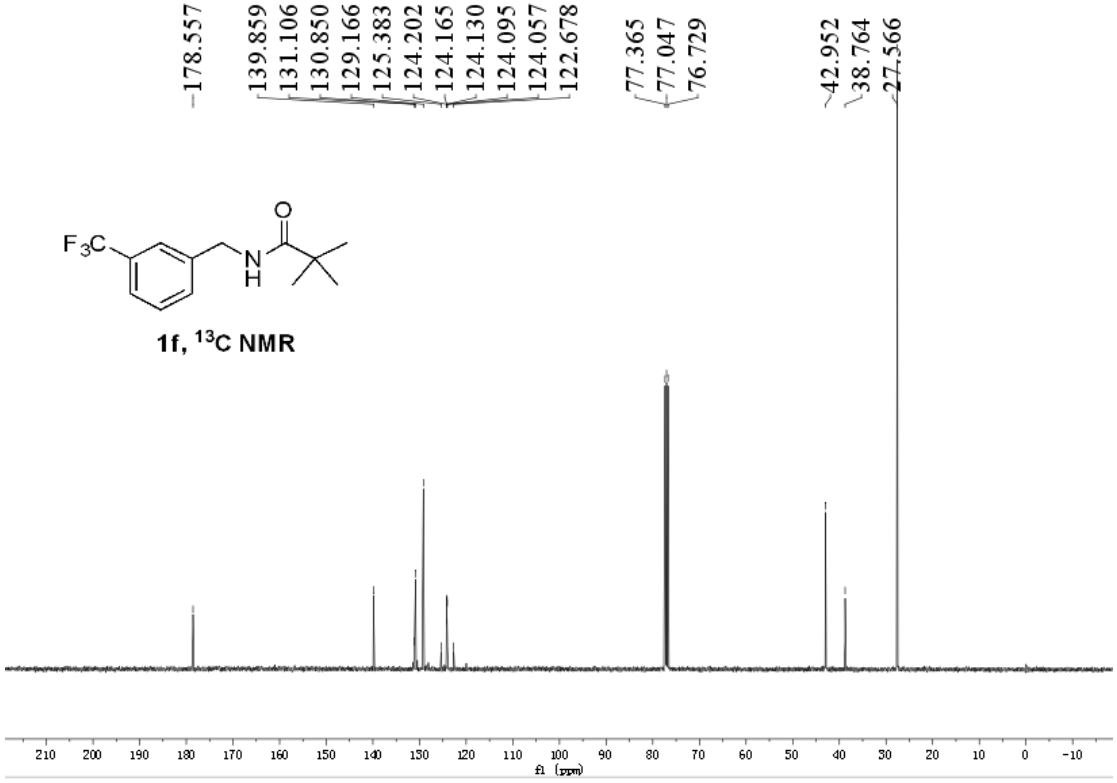
1e, ^{13}C NMR


132.310
132.303
130.370
127.470
126.062
126.006
125.949
125.892
40.219
40.196
27.464
CF3
O
N
H
1e, DEPT 135
f1 (ppm)

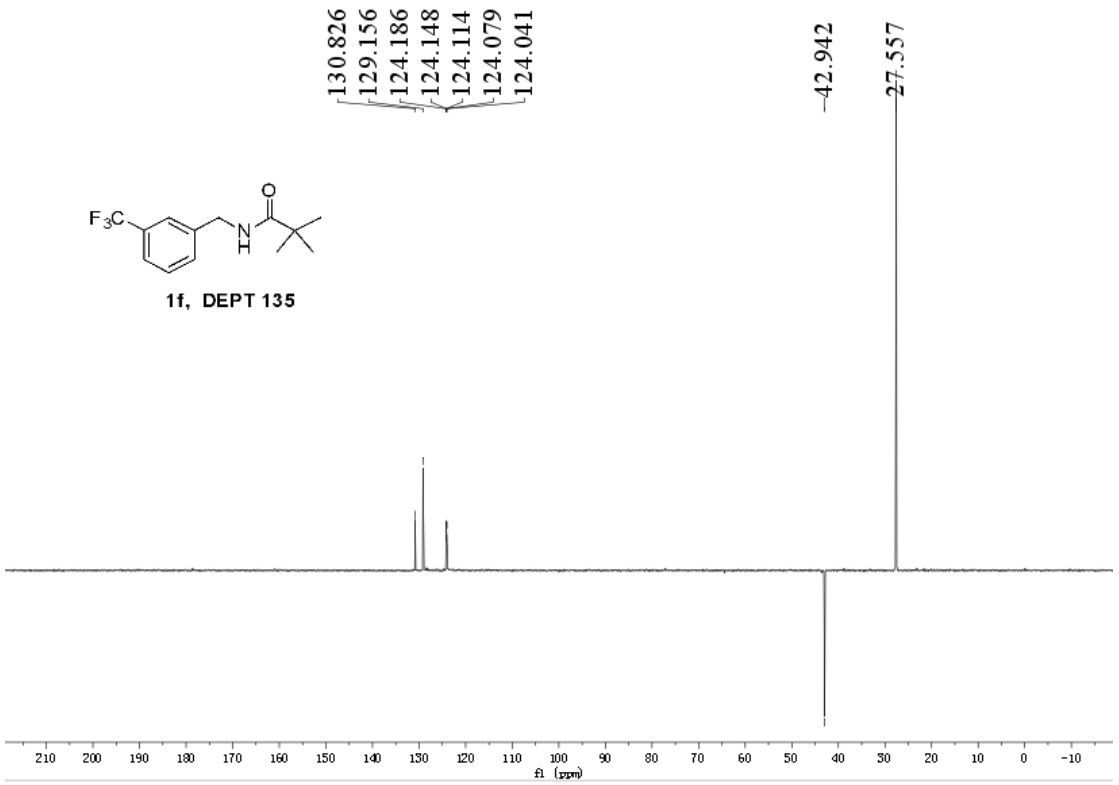

-59.584
CF3
O
N
H
1e, 19F NMR
f1 (ppm)

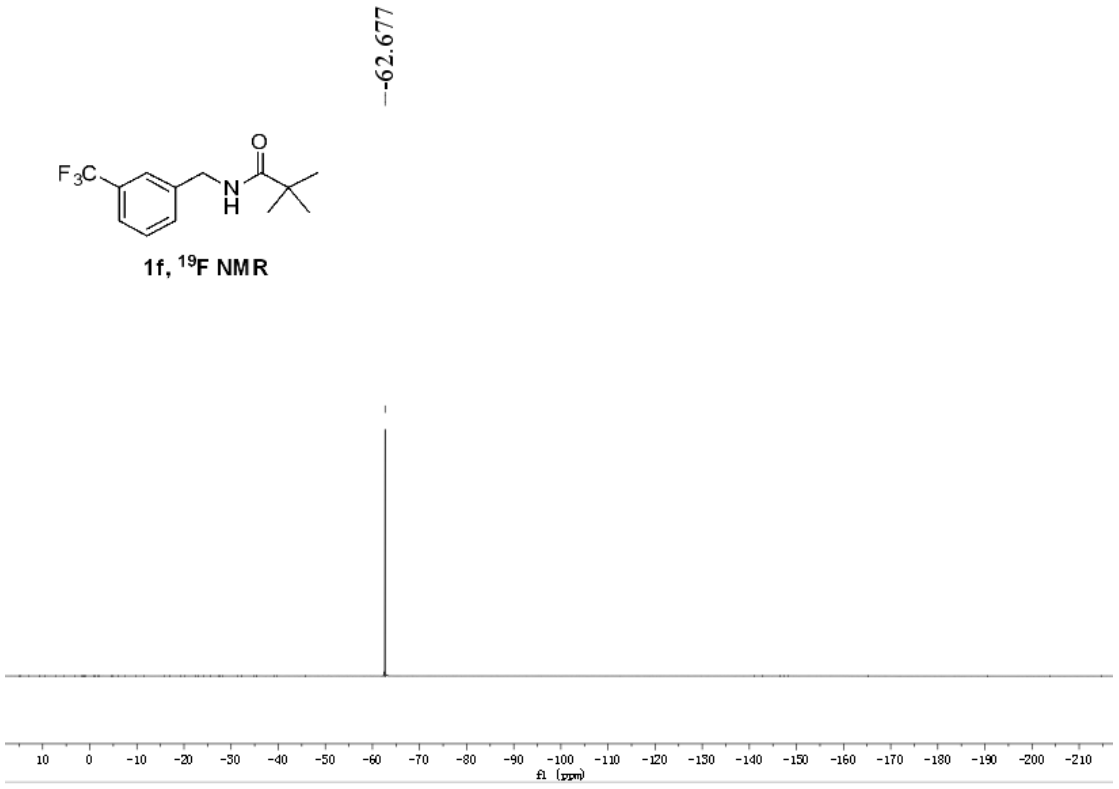


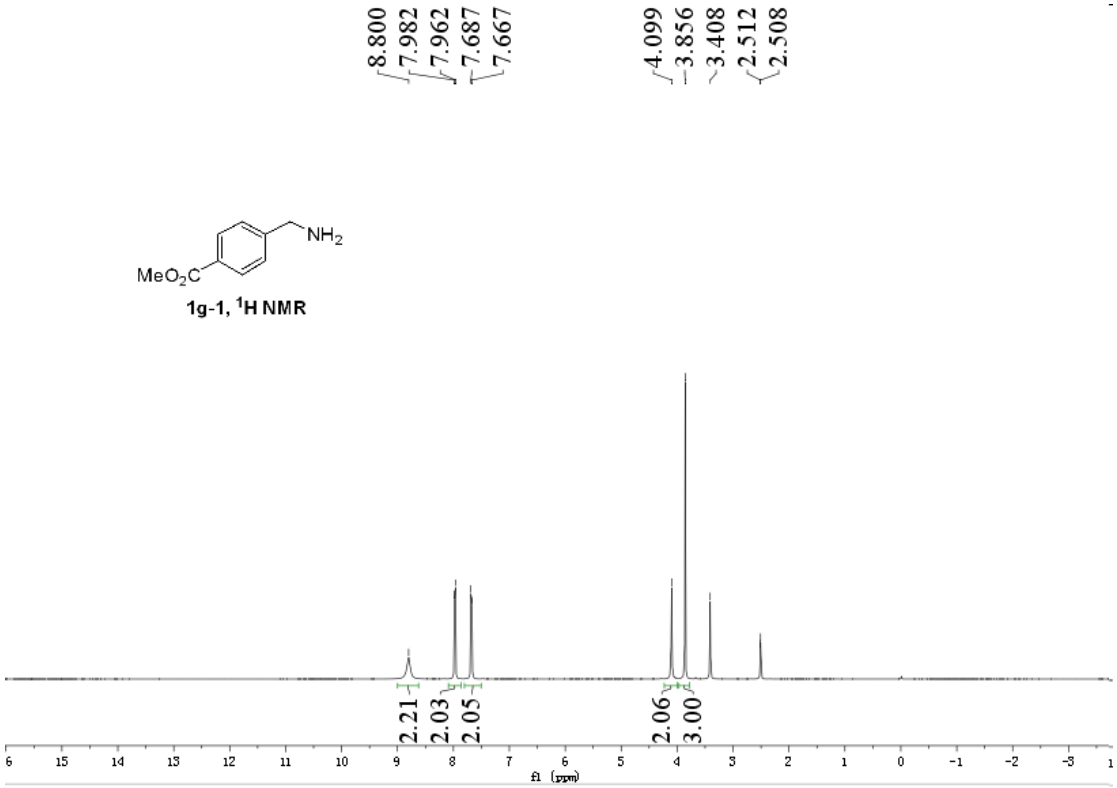
1f,^{1}H NMR



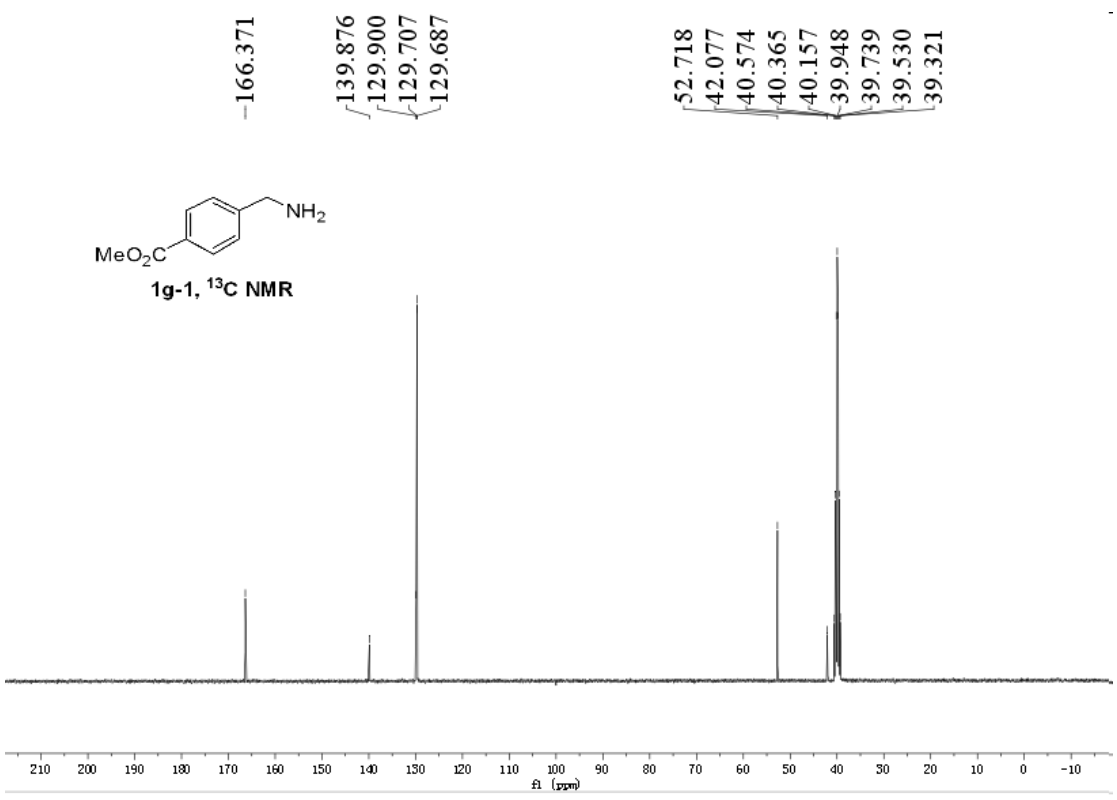
1f, ^{13}C NMR


130.826
129.156
124.186
124.148
124.114
124.079
124.041
42.942
27.557
F3C
O
N
H
1f, DEPT 135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

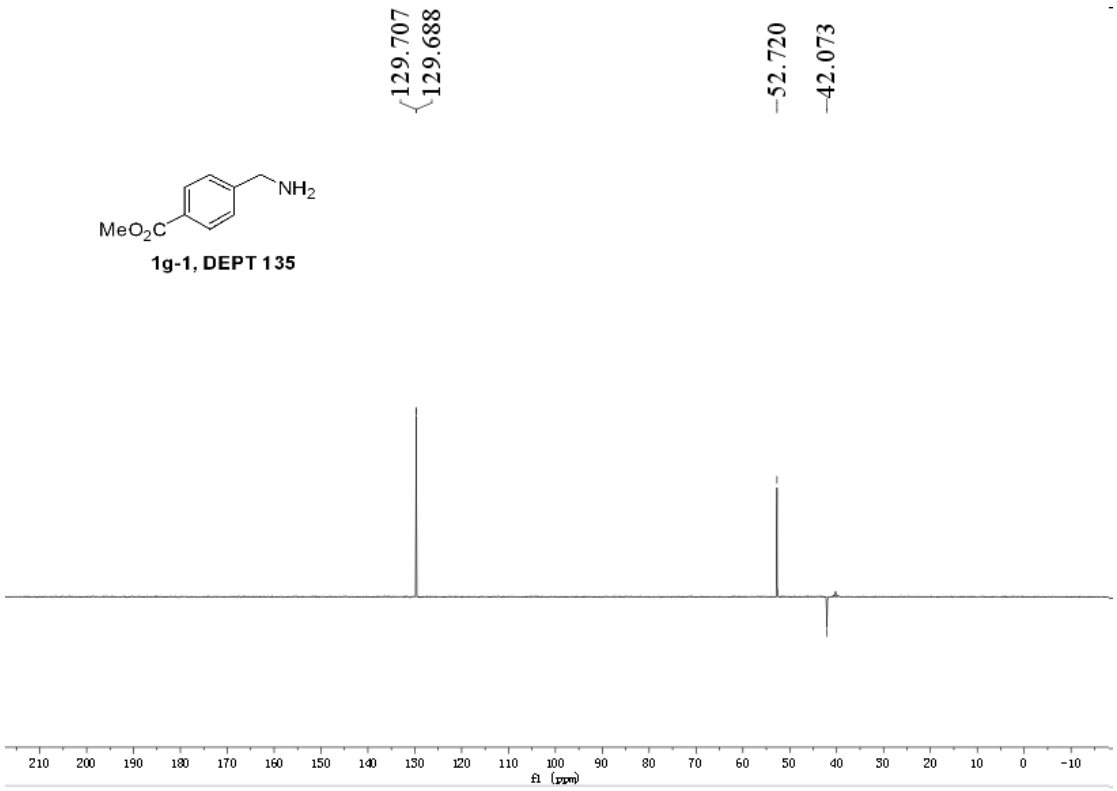

-62.677
F3C
O
N
H
1f, 19F NMR
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

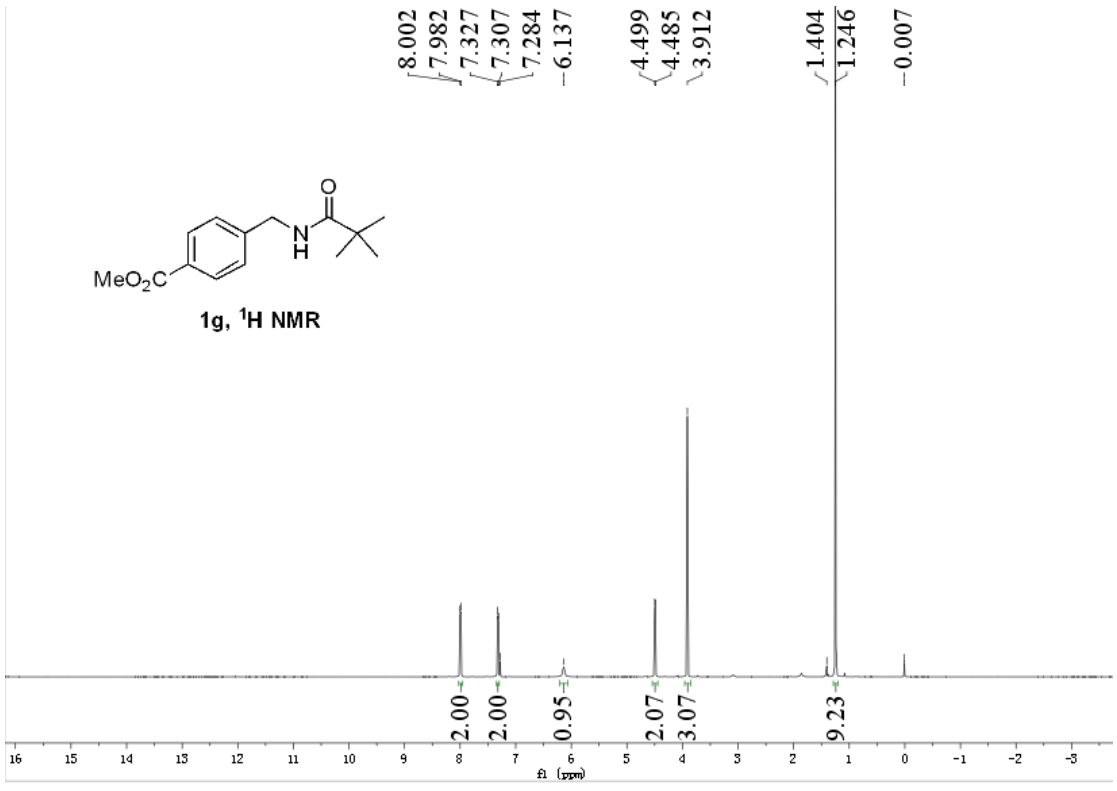


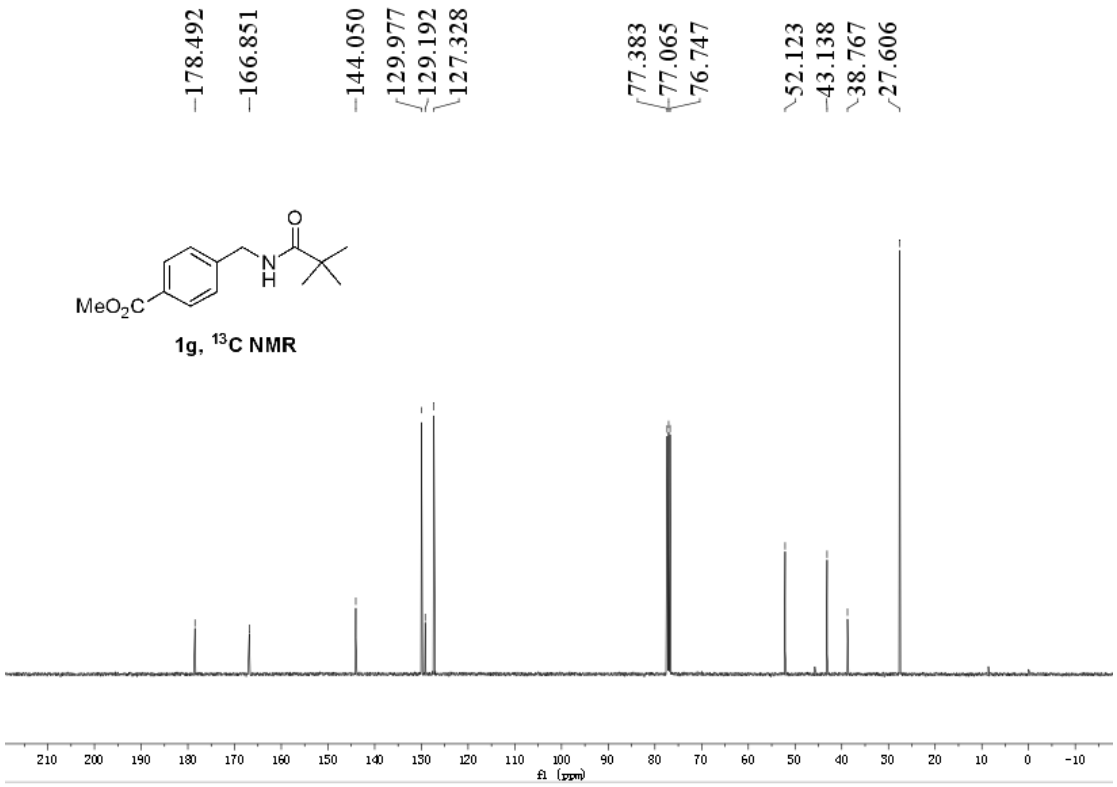
1g-1, ^{1}H NMR

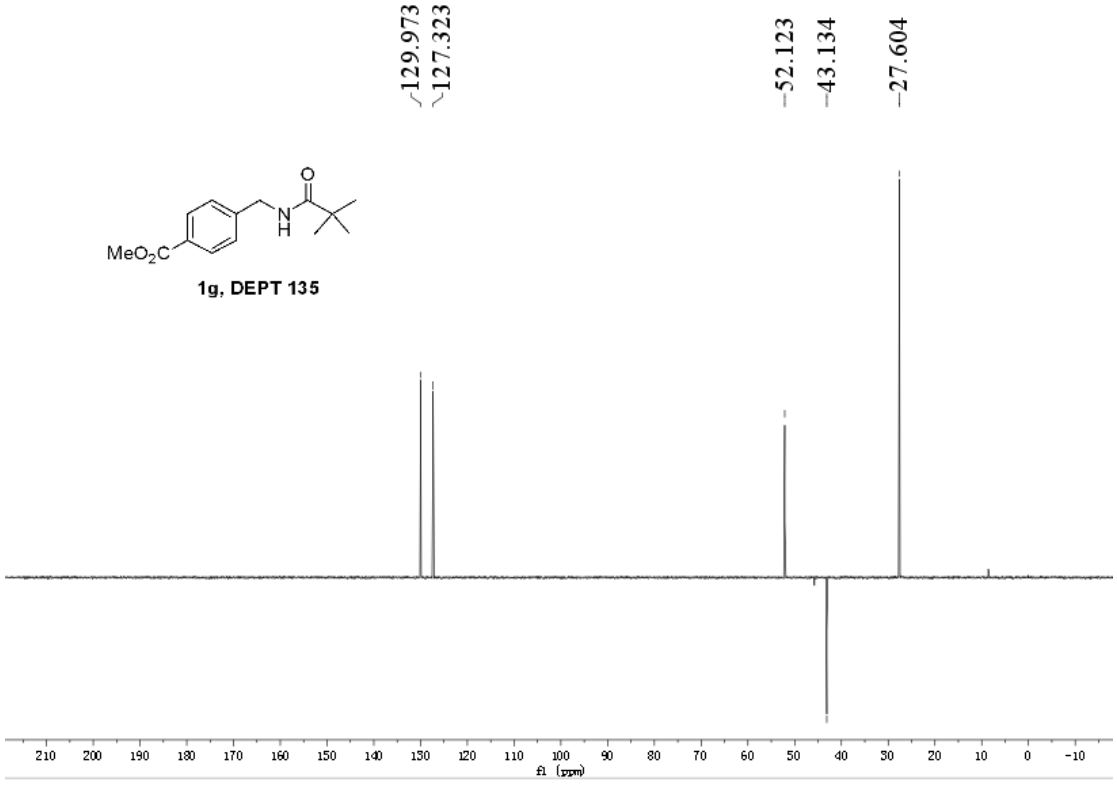


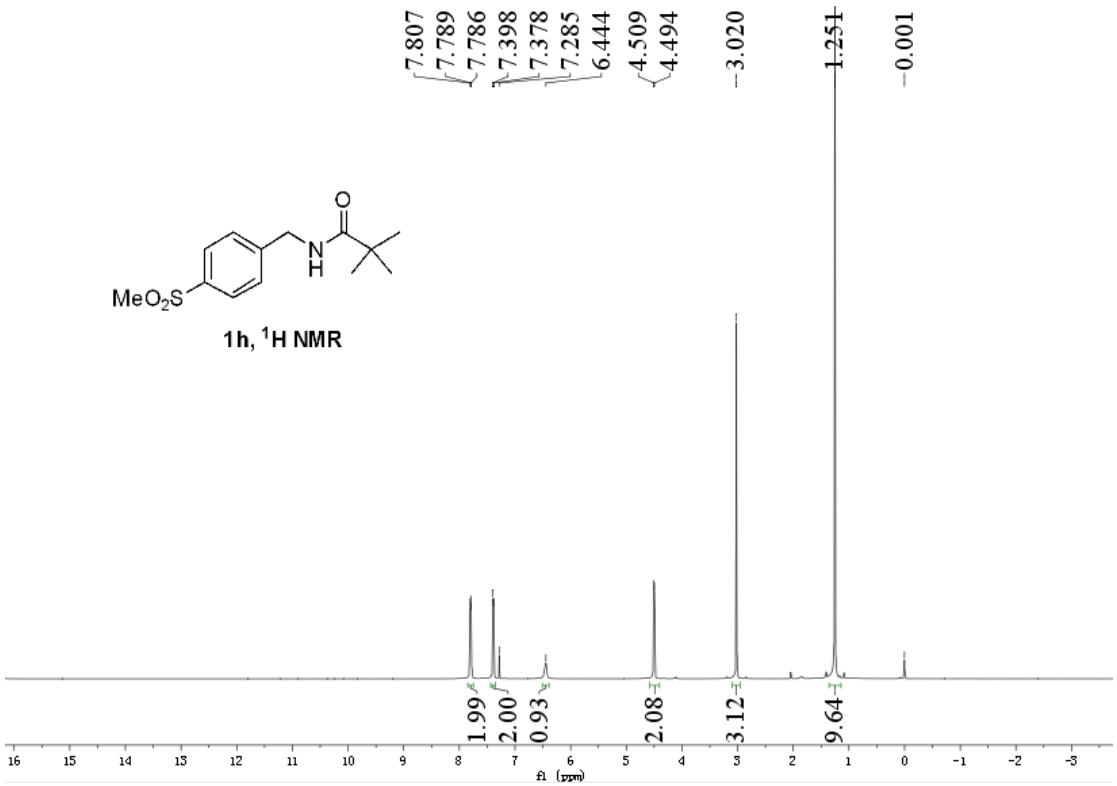
1g-1, ^{13}C NMR


129.707
129.688
52.720
42.073
NH2
MeO2C
1g-1, DEPT 135
f1 (ppm)

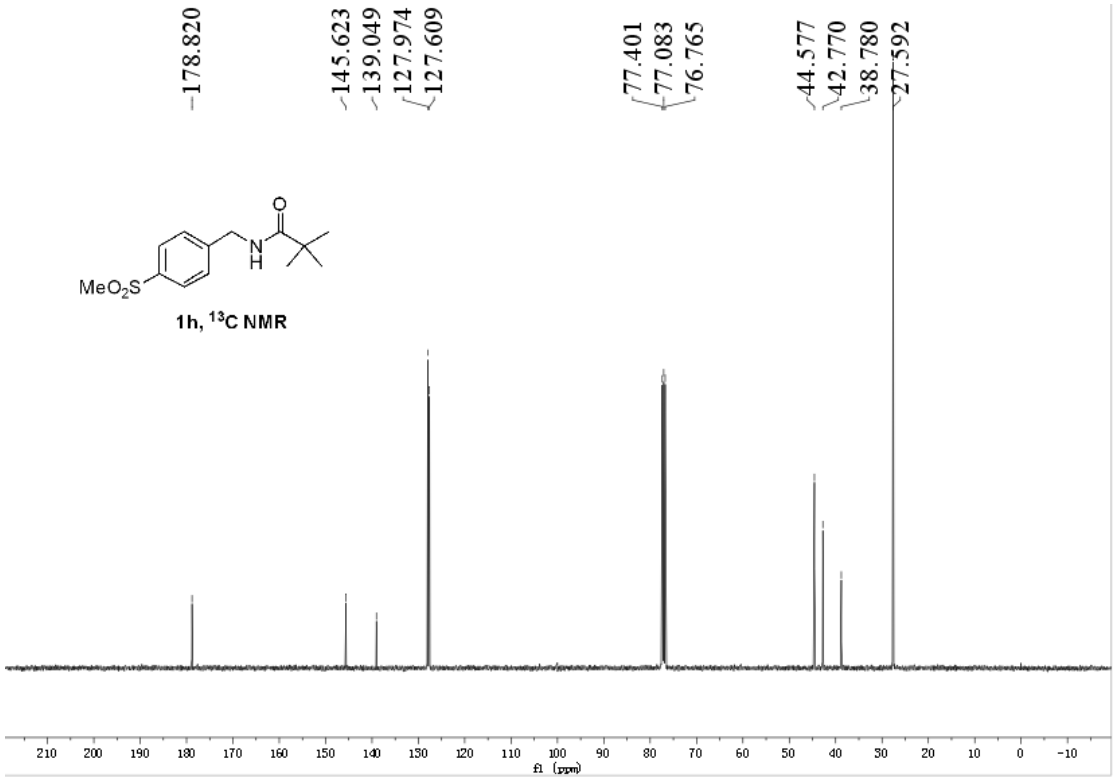

8.002
7.982
7.327
7.307
7.284
6.137
4.499
4.485
3.912
1.404
1.246
0.007
O
N
H
MeO2C
1g, 1H NMR
2.00
2.00
0.95
2.07
3.07
9.23
f1 (ppm)


178.492
166.851
144.050
129.977
129.192
127.328
77.383
77.065
76.747
52.123
43.138
38.767
27.606
MeO2C
N
H
O
1g, 13C NMR
f1 (ppm)

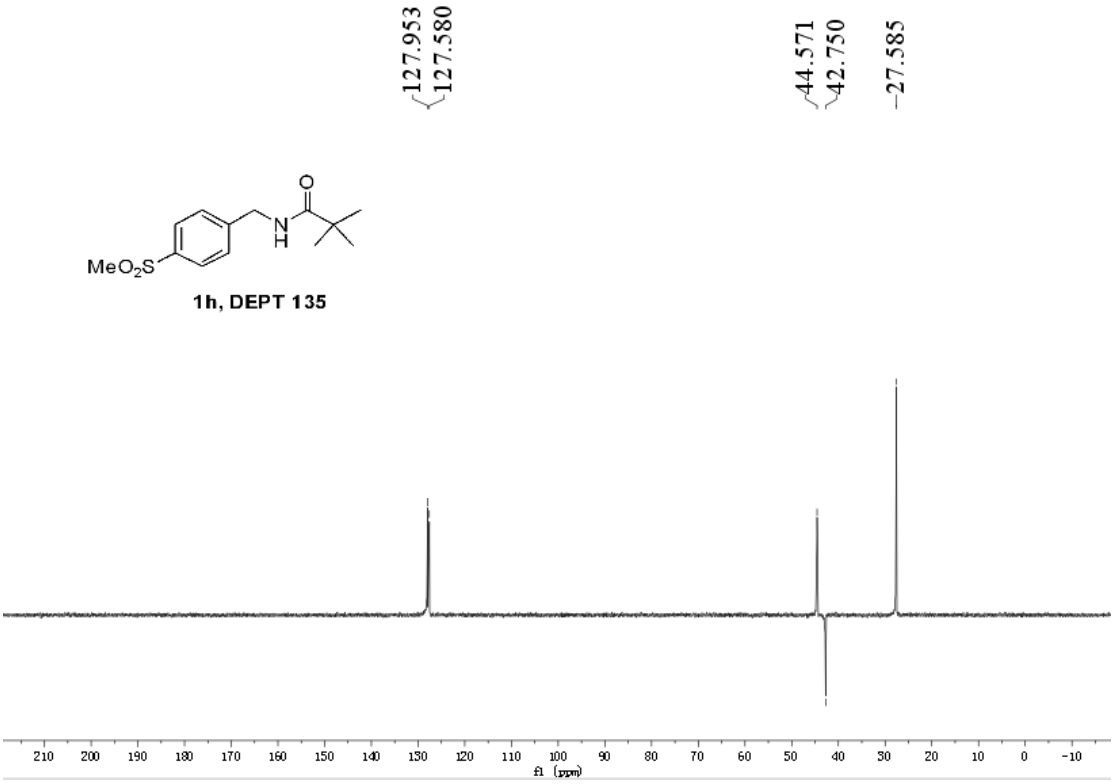

129.973
127.323
52.123
43.134
27.604
MeO2C
N
H
O
1g, DEPT 135
f1 (ppm)

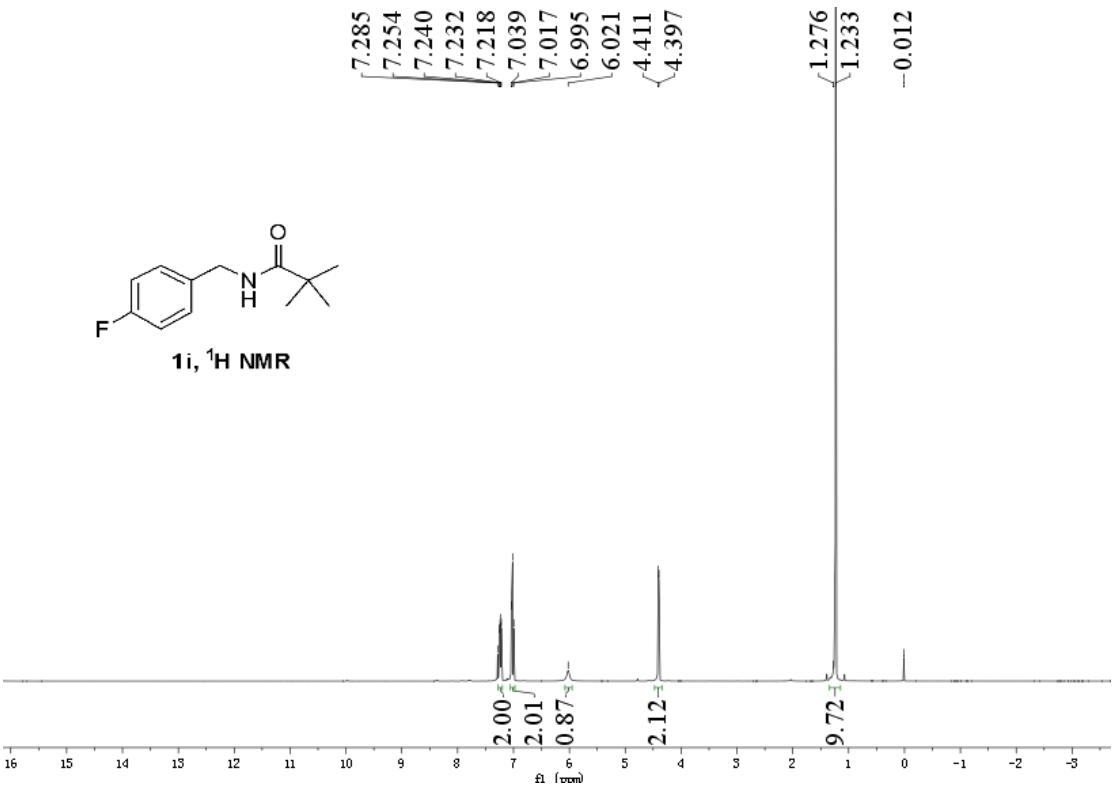


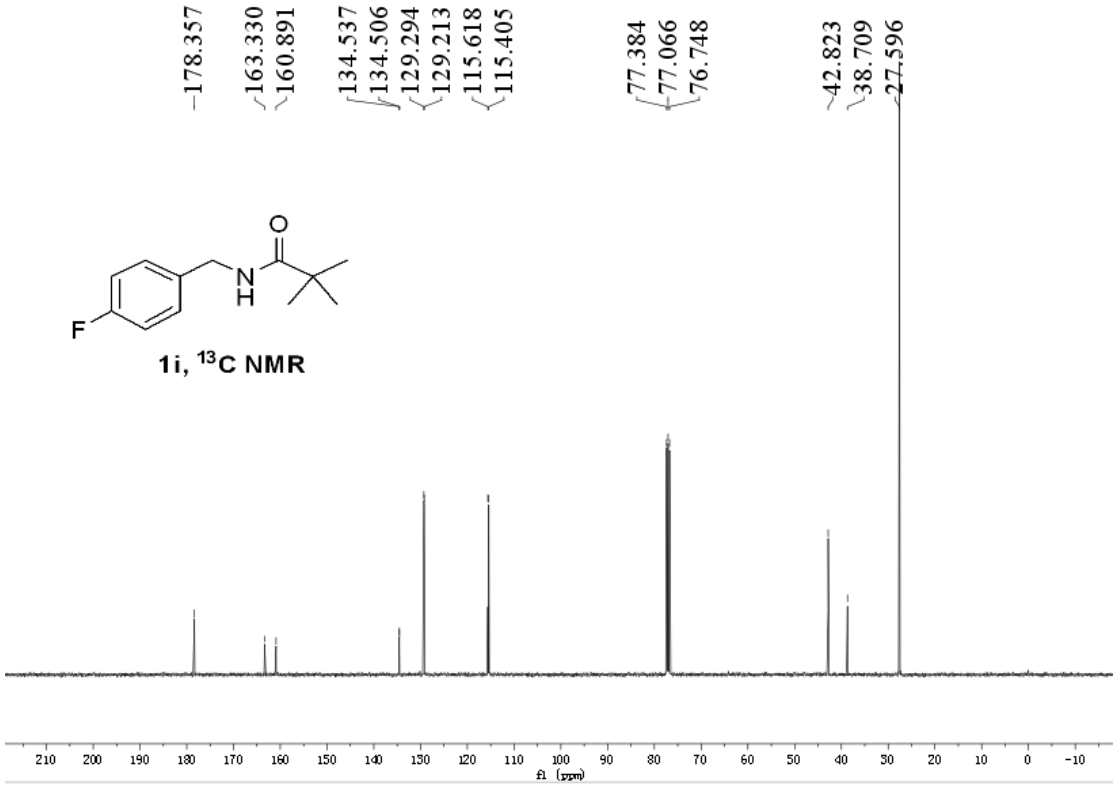
1h, ^{1}H NMR

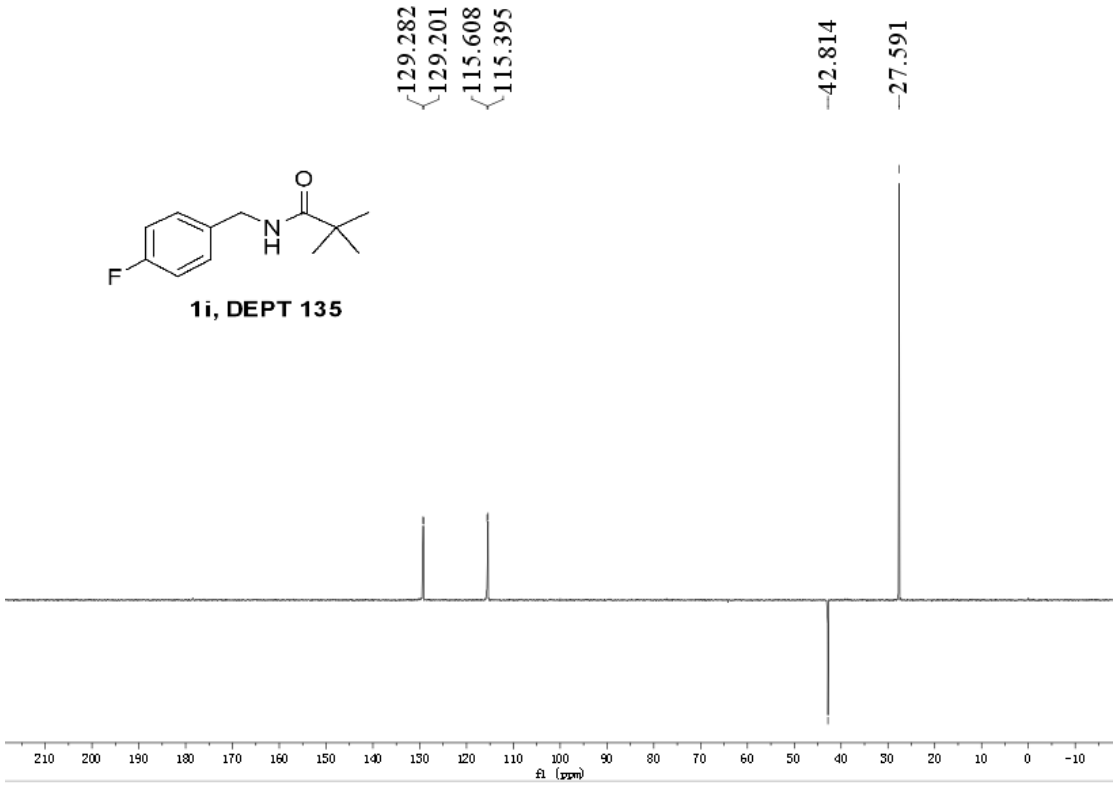


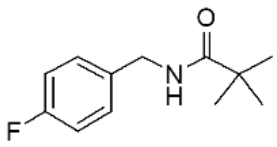
1h, ^{13}C NMR


127.953
127.580
44.571
42.750
27.585
MeO2S
1h, DEPT 135
f1 (ppm)

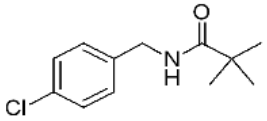

7.285
7.254
7.240
7.232
7.218
7.039
7.017
6.995
6.021
4.411
4.397
1.276
1.233
0.012
2.00
2.01
0.87
2.12
9.72
F
1i, 1H NMR
f1 (ppm)


178.357
163.330
160.891
134.537
134.506
129.294
129.213
115.618
115.405
77.384
77.066
76.748
42.823
38.709
27.596
O
N
H
F
1i, ^{13}C NMR
f1 (ppm)

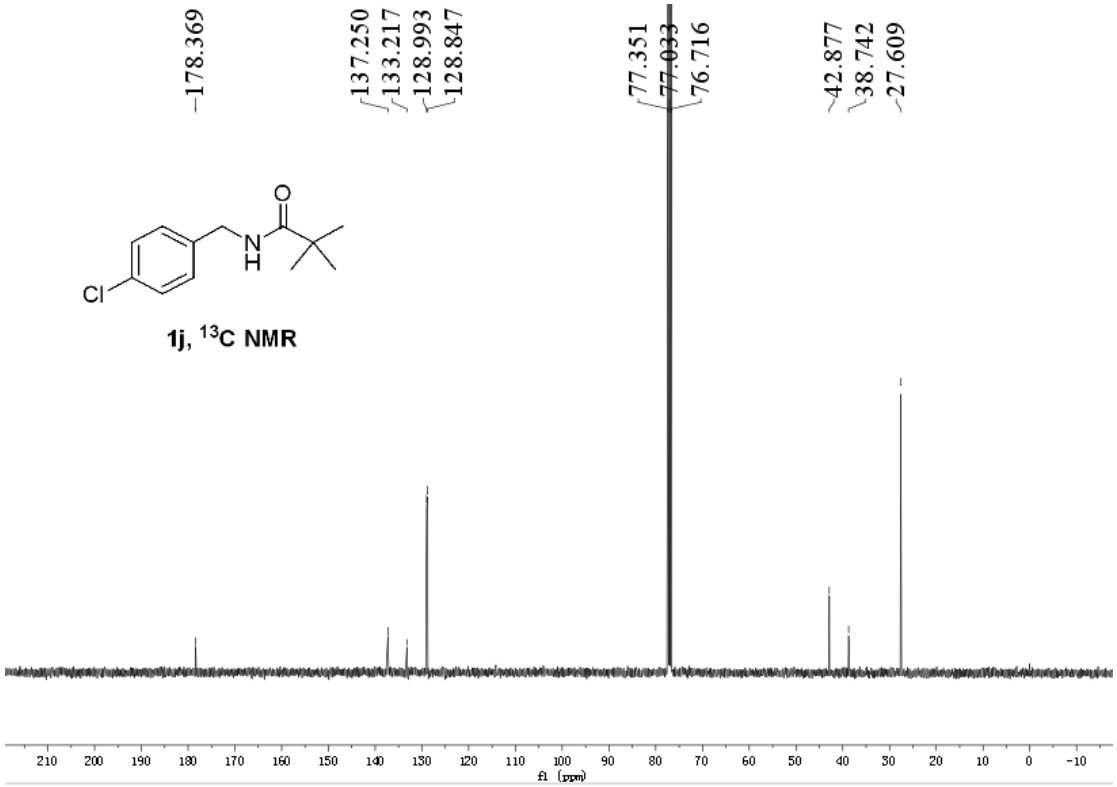

129.282
129.201
115.608
115.395
42.814
27.591
O
N
H
F
1i, DEPT 135
f1 (ppm)

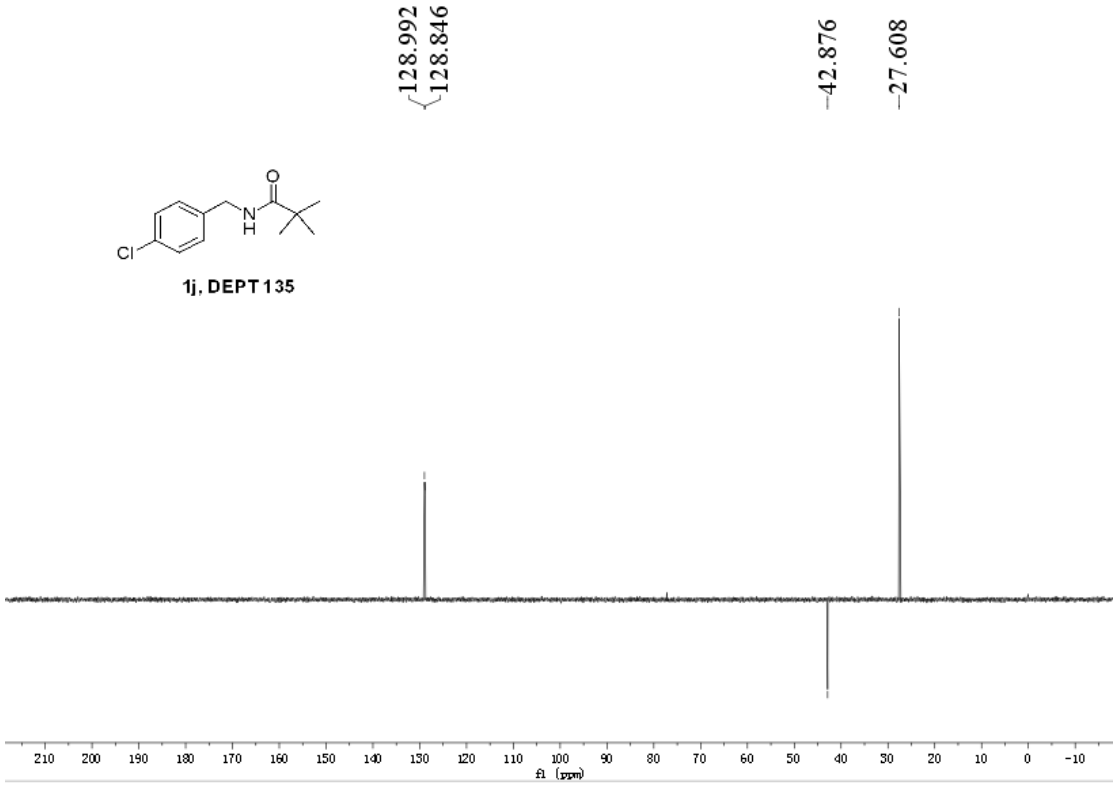


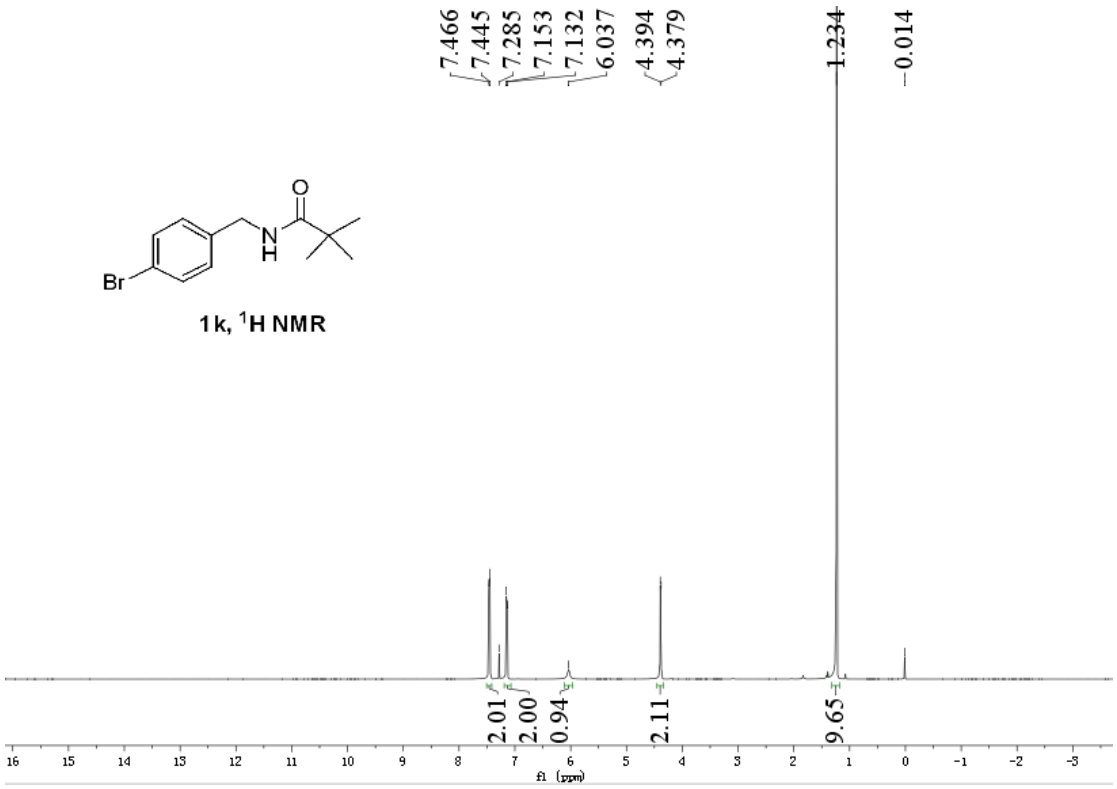
1i, ^{19}F NMR



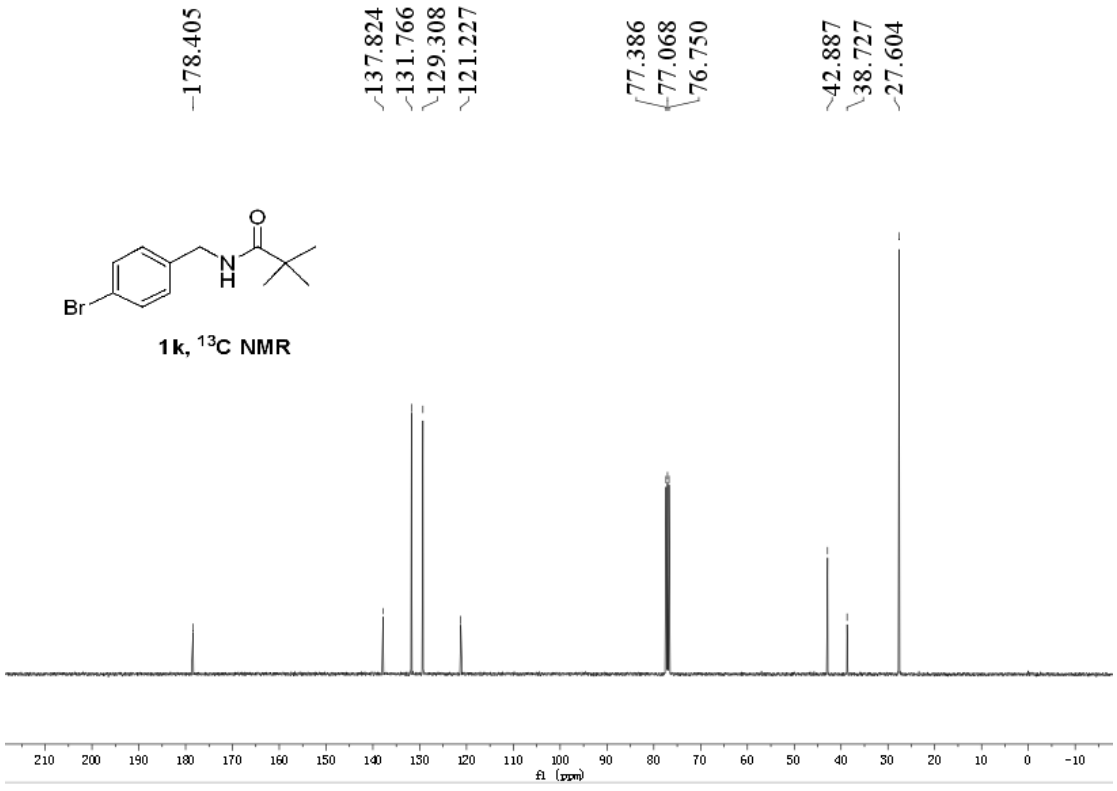
1j, ^{1}H NMR


178.369
137.250
133.217
128.993
128.847
77.351
77.033
76.716
42.877
38.742
27.609
Cl
O
N
H
1j, ^{13}C NMR
f1 (ppm)

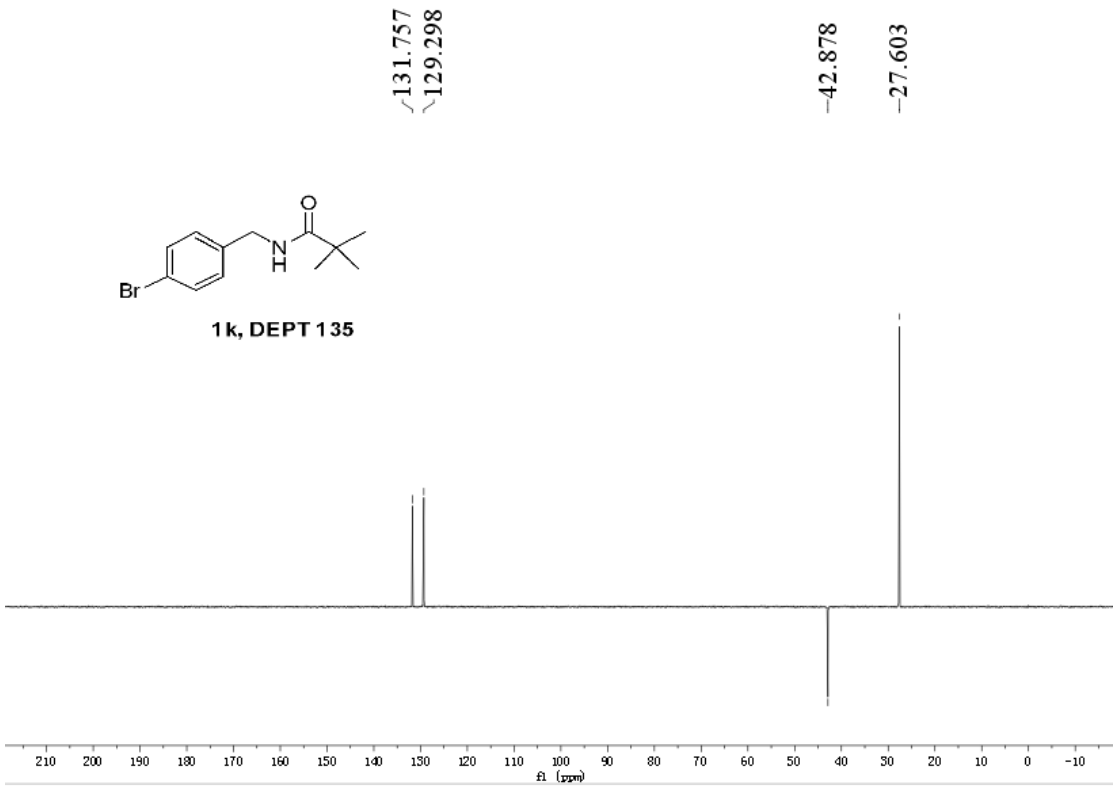

128.992
128.846
42.876
27.608
Cl
O
N
H
1j, DEPT135
f1 (ppm)

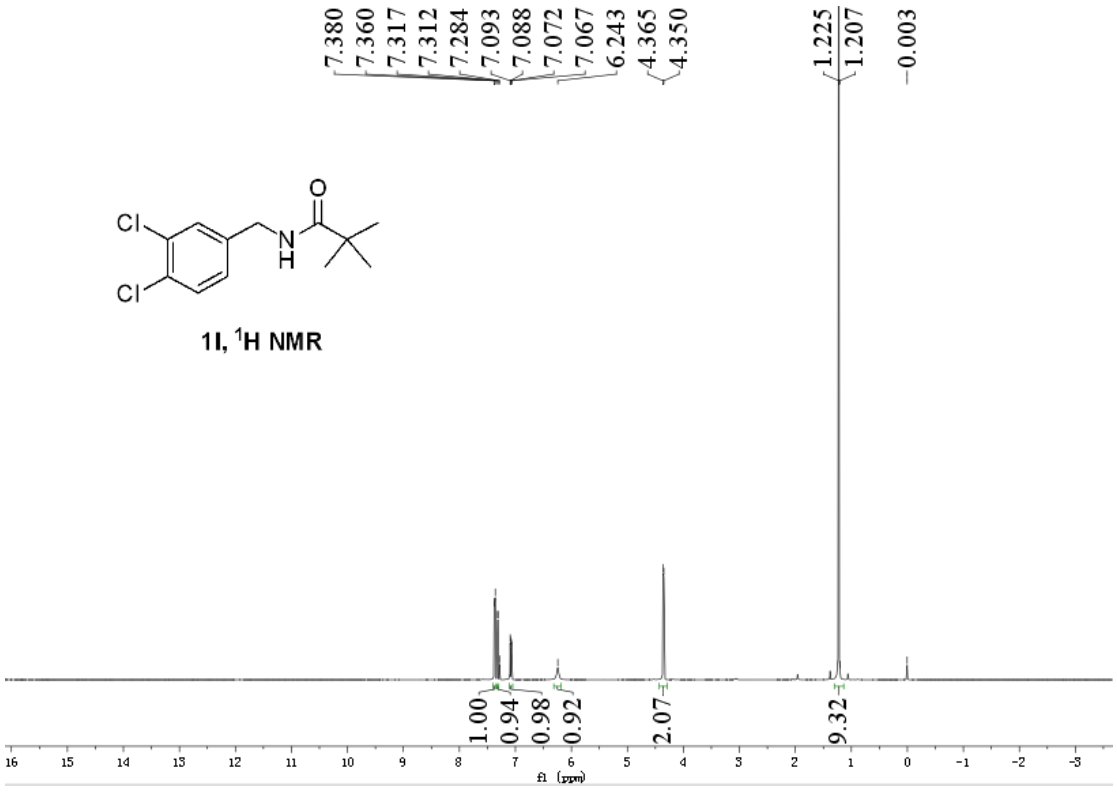


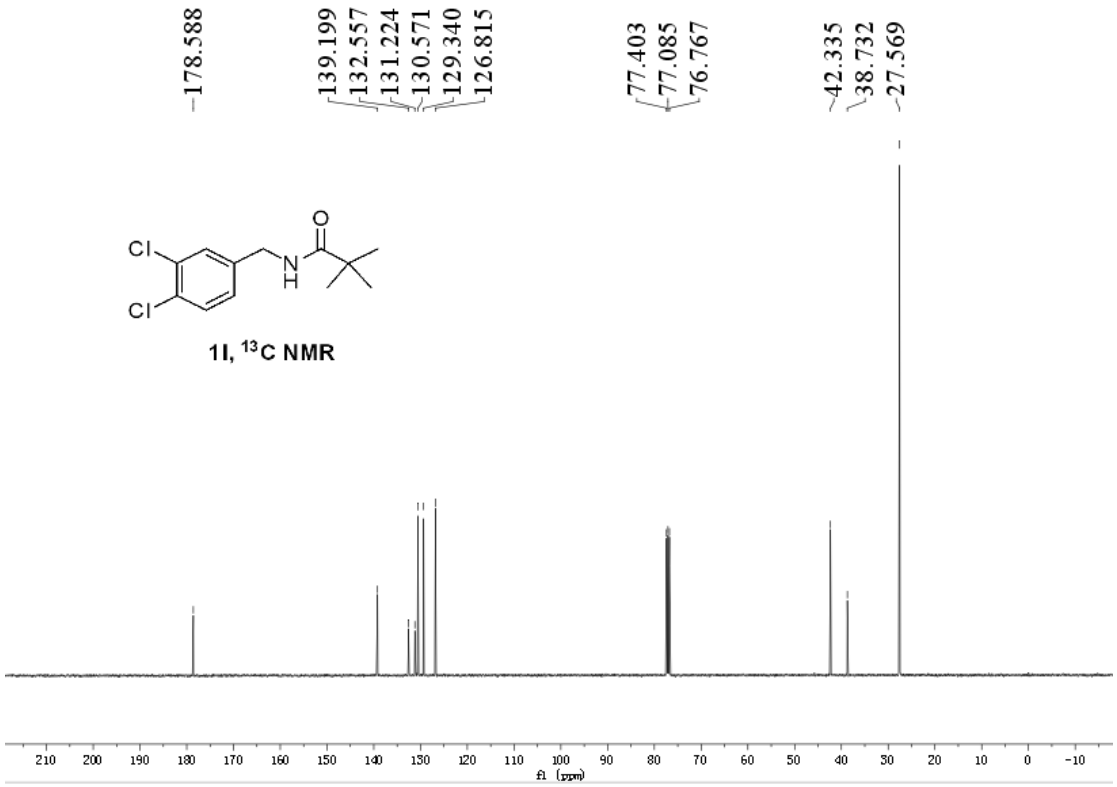
1k, ^{1}H NMR



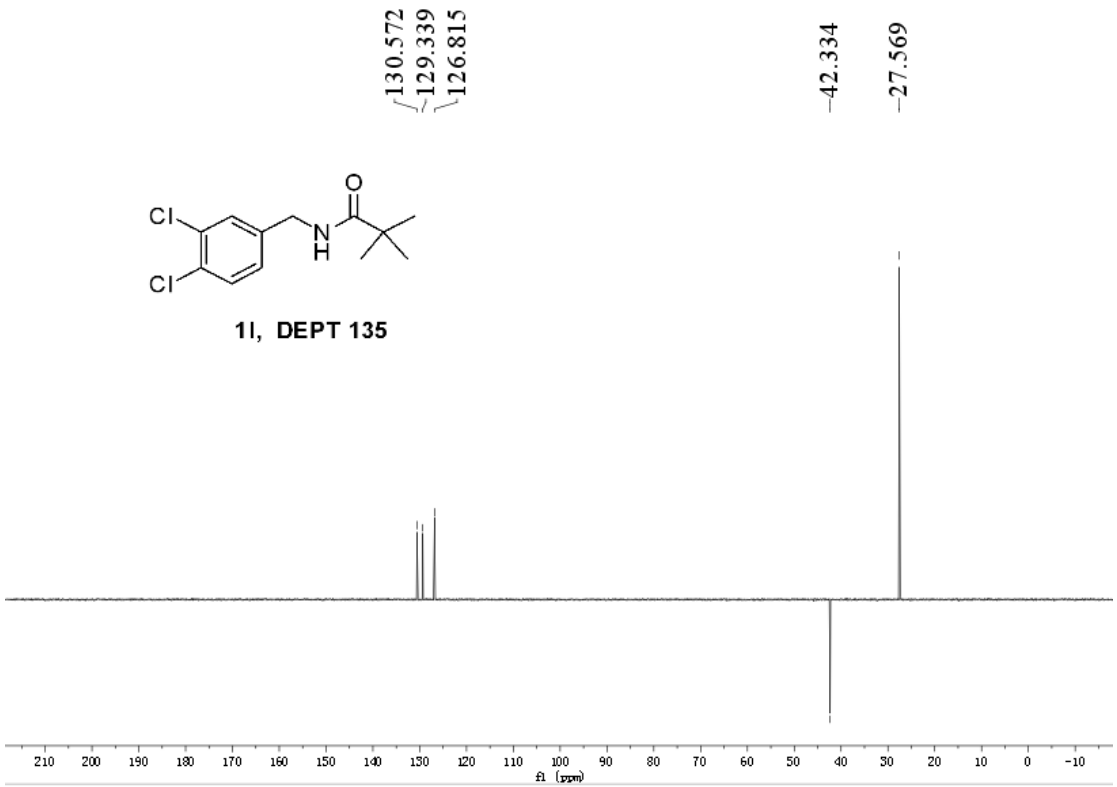
1k, ^{13}C NMR


131.757
129.298
42.878
27.603
Br
1k, DEPT 135
f1 (ppm)

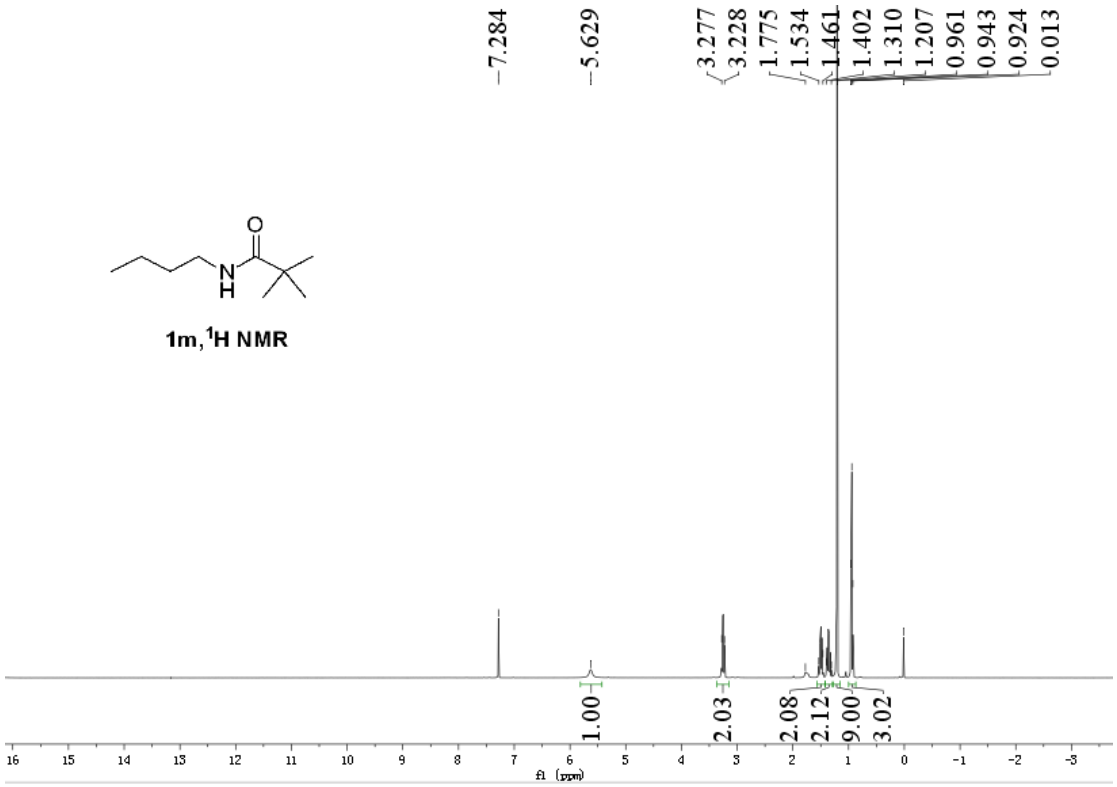

7.380
7.360
7.317
7.312
7.284
7.093
7.088
7.072
7.067
6.243
4.365
4.350
1.225
1.207
0.003
Cl
Cl
1l, 1H NMR
1.00
0.94
0.98
0.92
2.07
9.32
f1 (ppm)



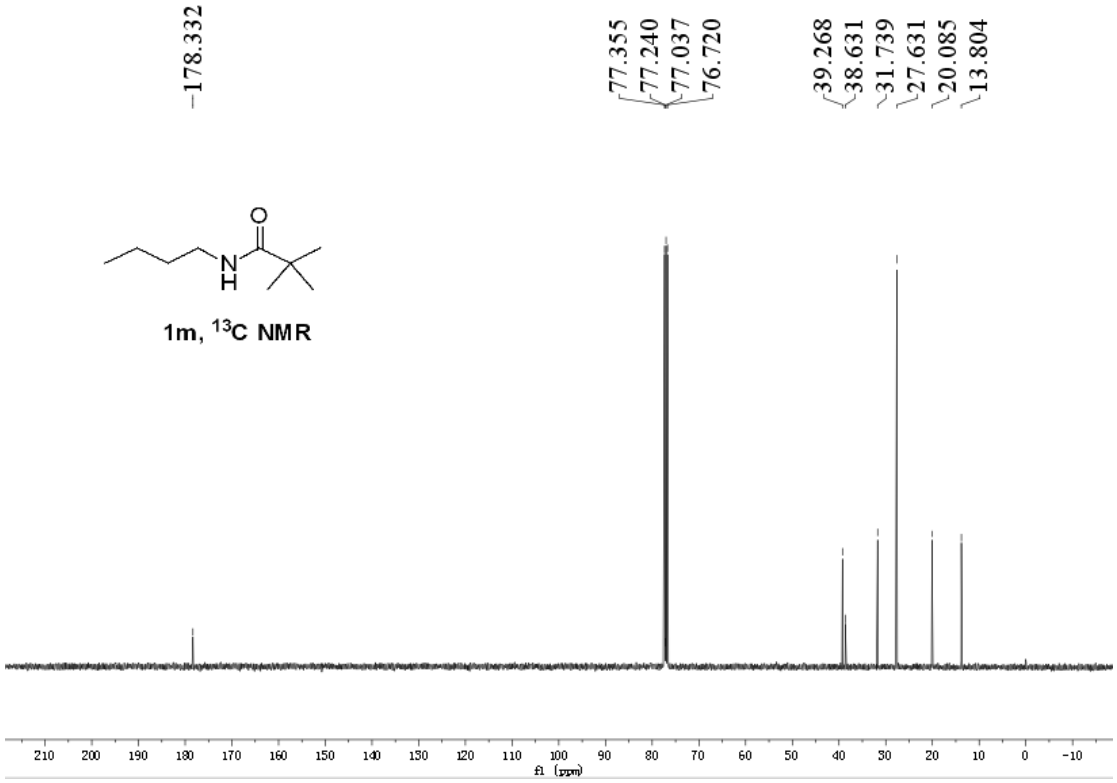
1l, ^{13}C NMR



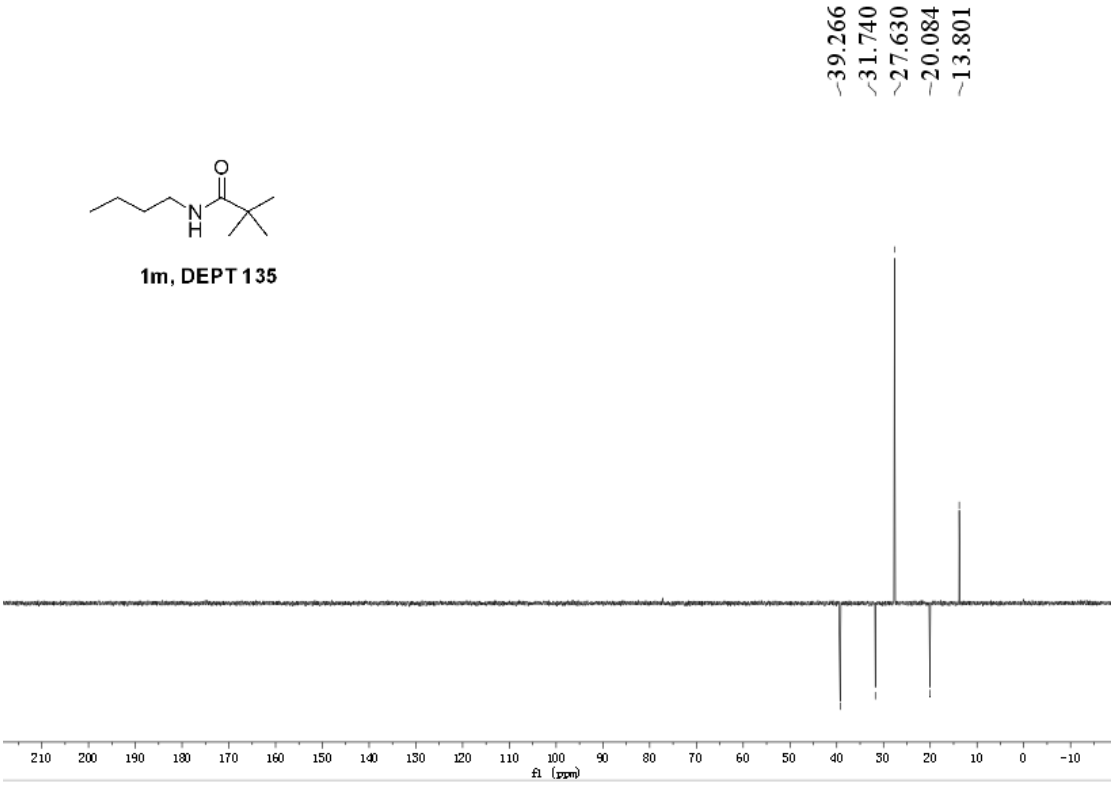
1l, DEPT 135



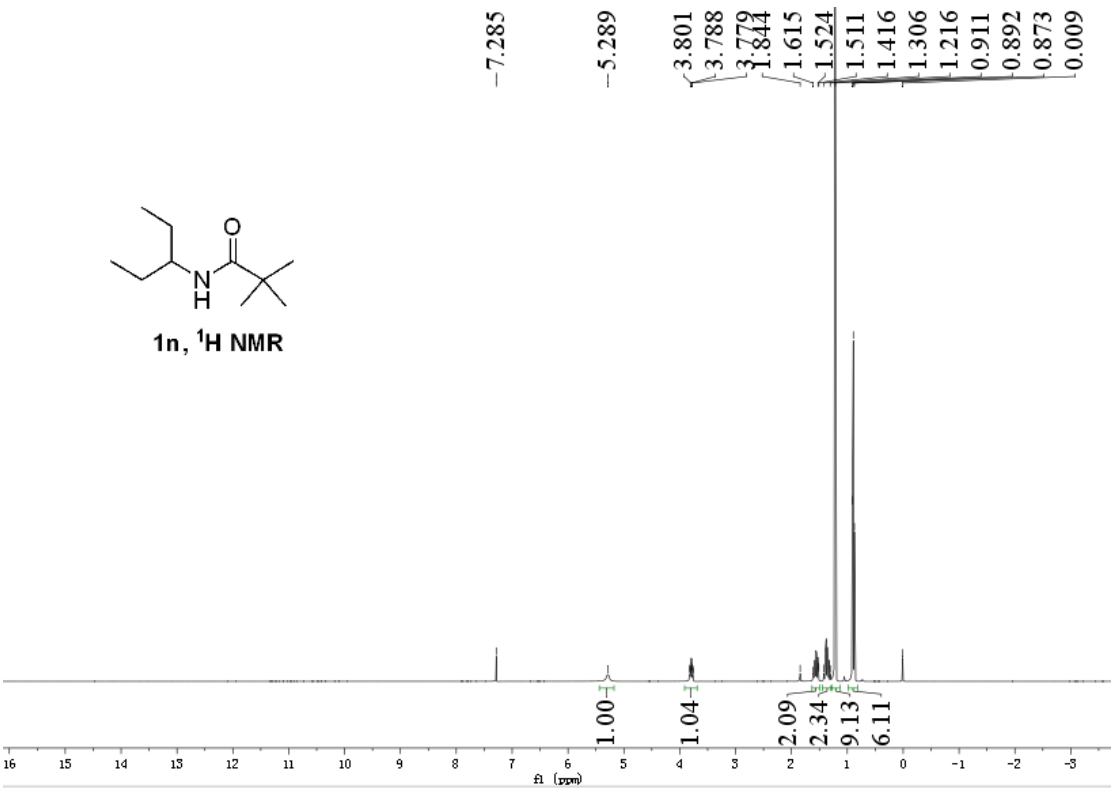
1m, ^{1}H NMR



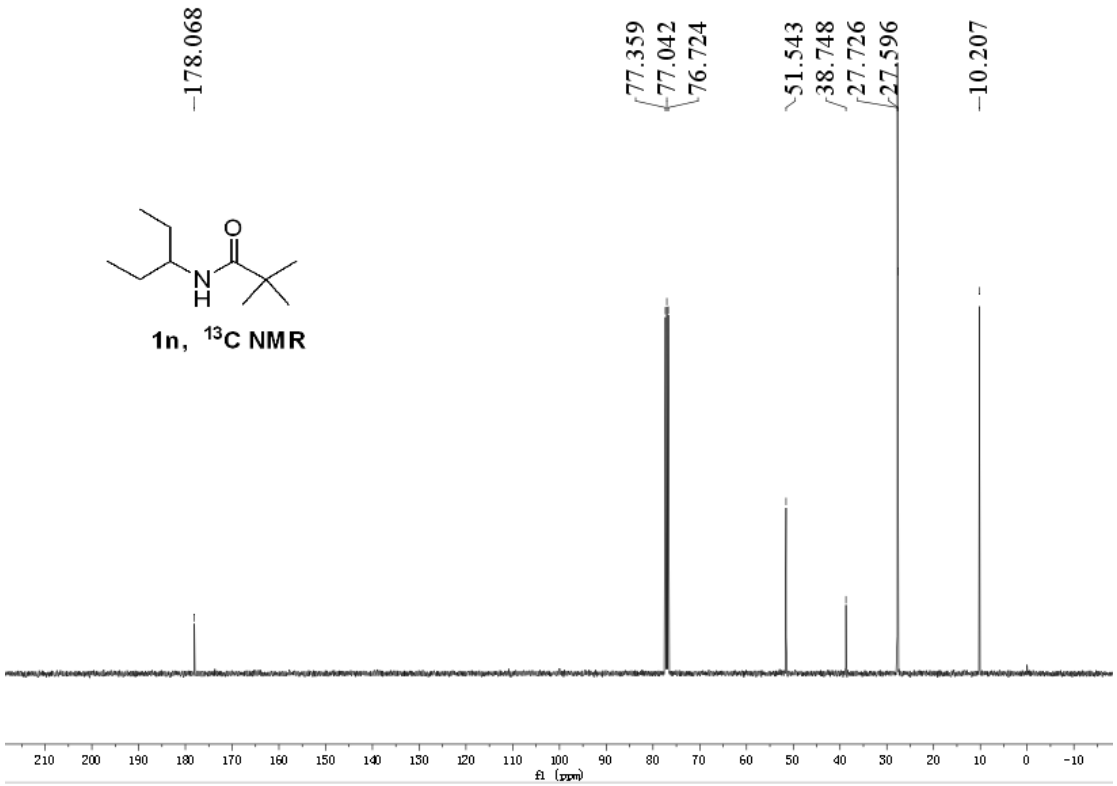
1m, ^{13}C NMR

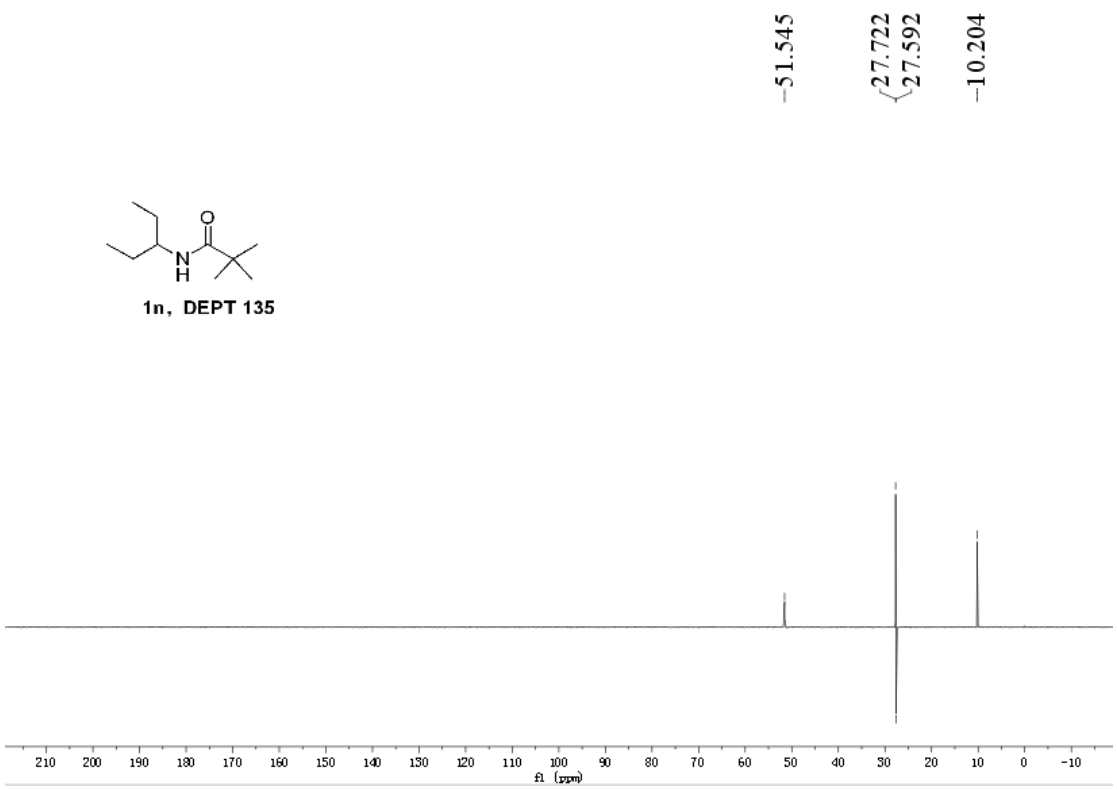


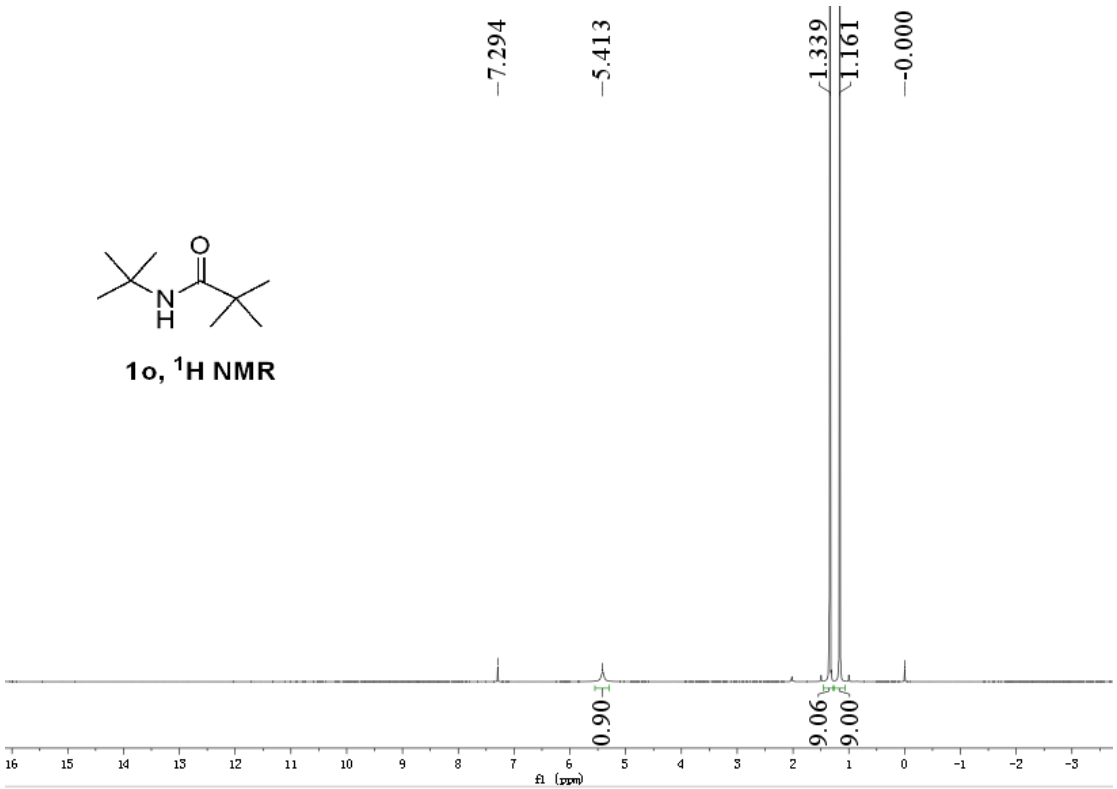
1m, DEPT 135



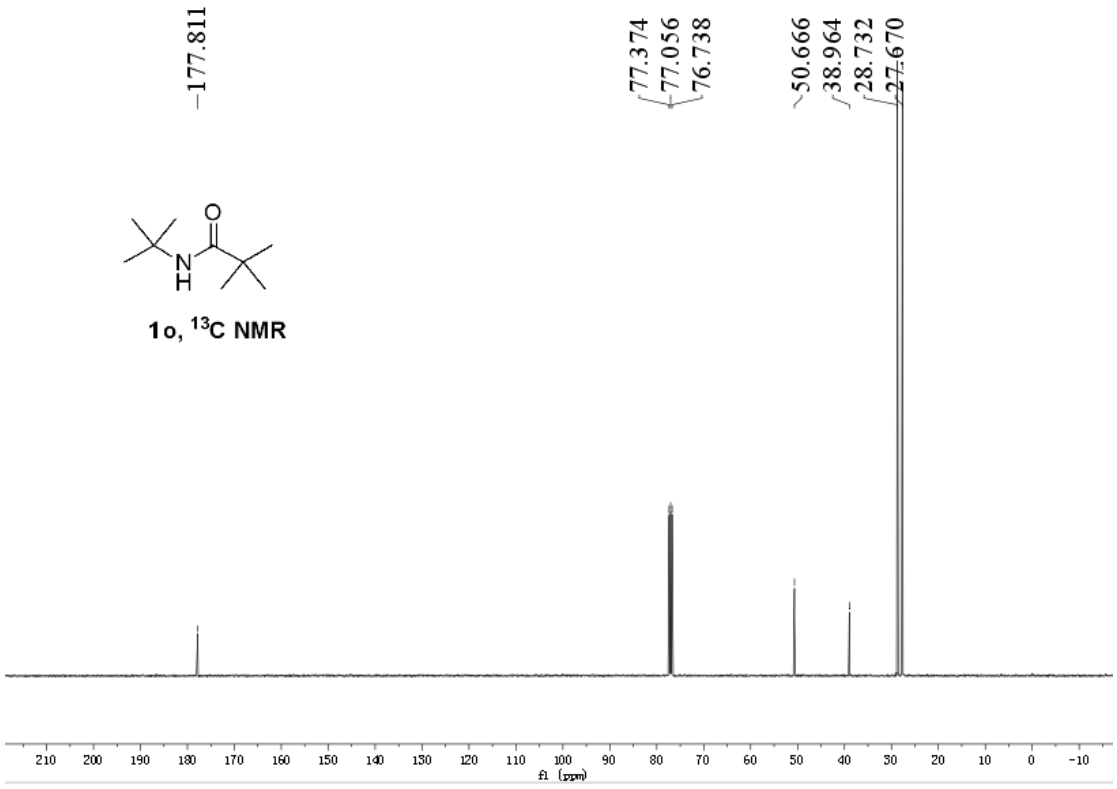
1n, ^{1}H NMR


178.068
77.359
77.042
76.724
51.543
38.748
27.726
27.596
10.207
O
N
H
1n, ^{13}C NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

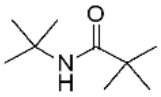

51.545
27.722
27.592
10.204
O
N
H
1n, DEPT 135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



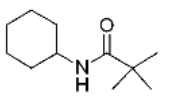
1o, ^{1}H NMR



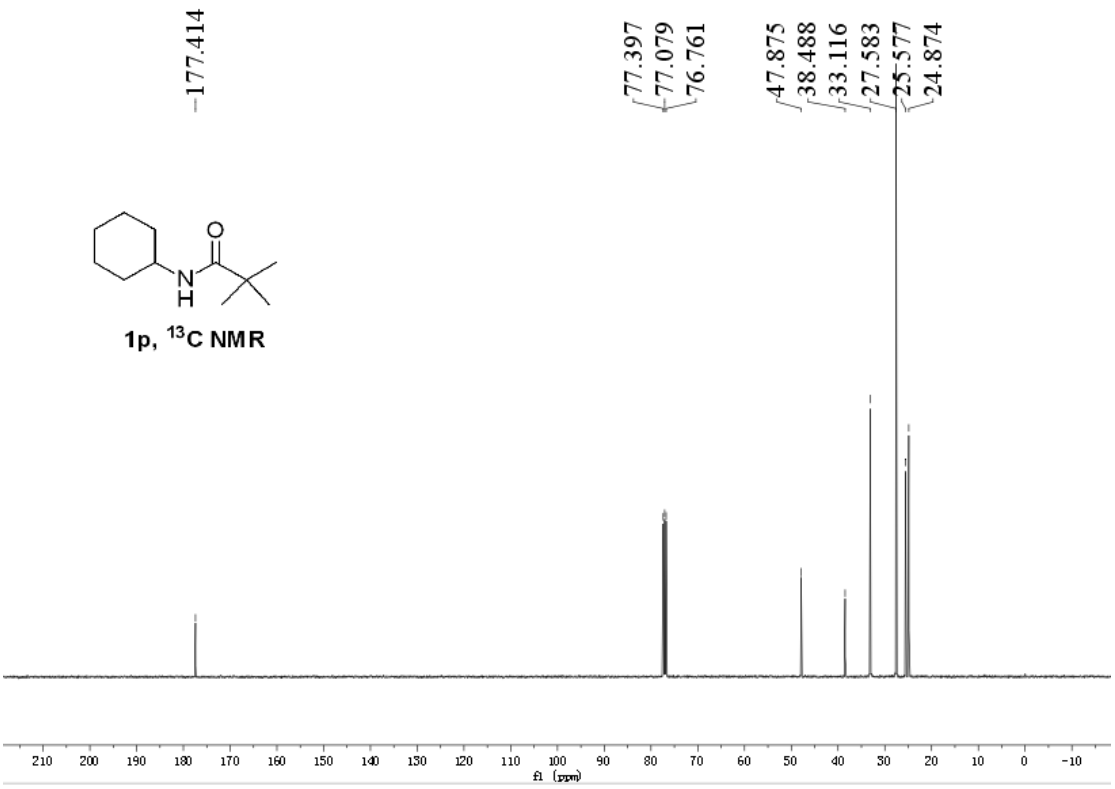
1o, ^{13}C NMR

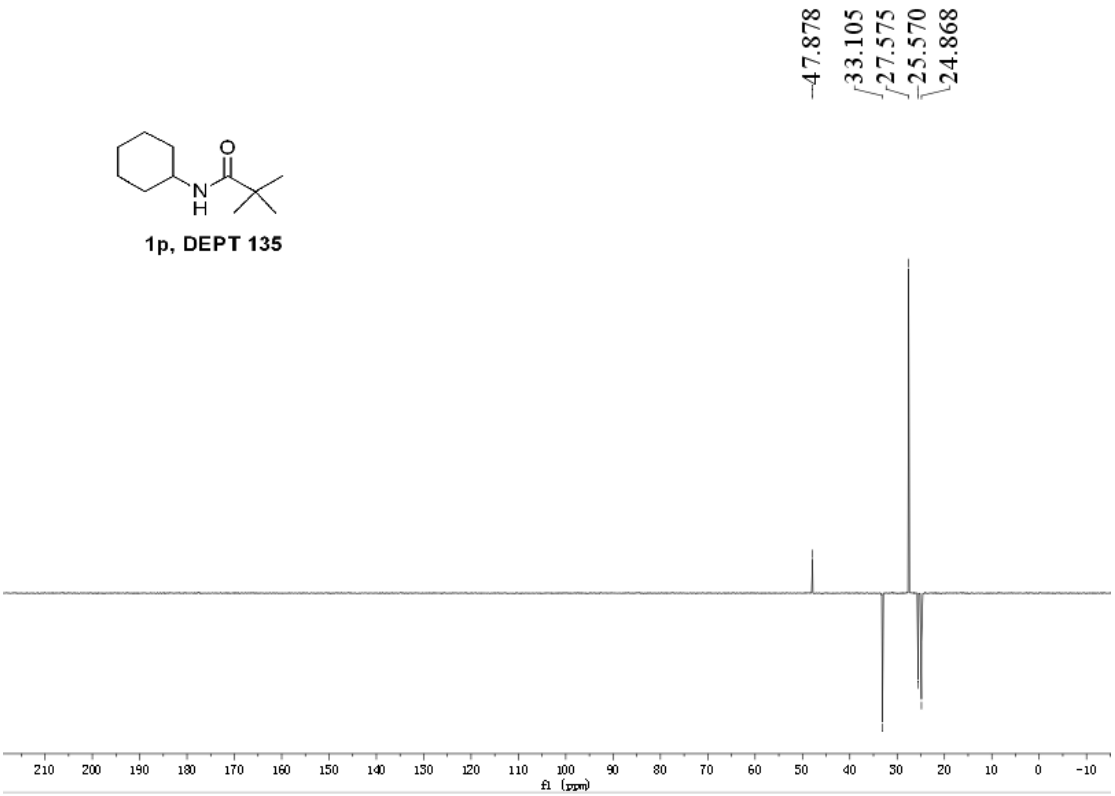


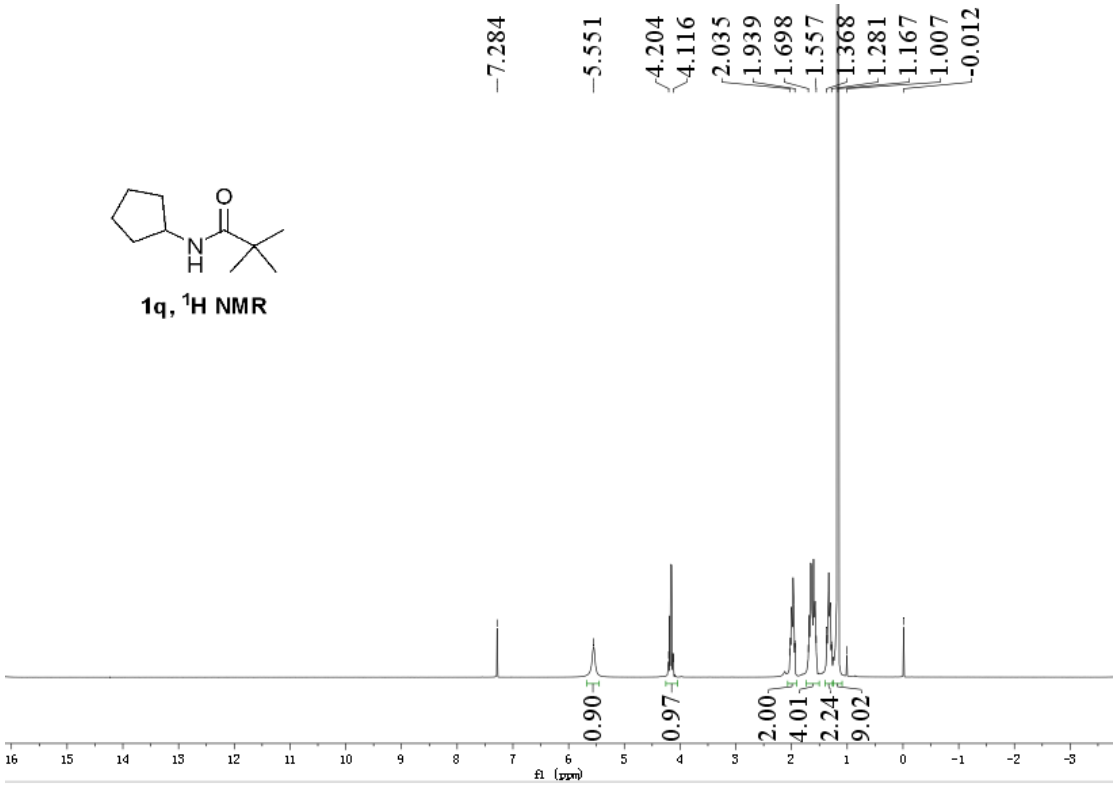
1o, DEPT 135



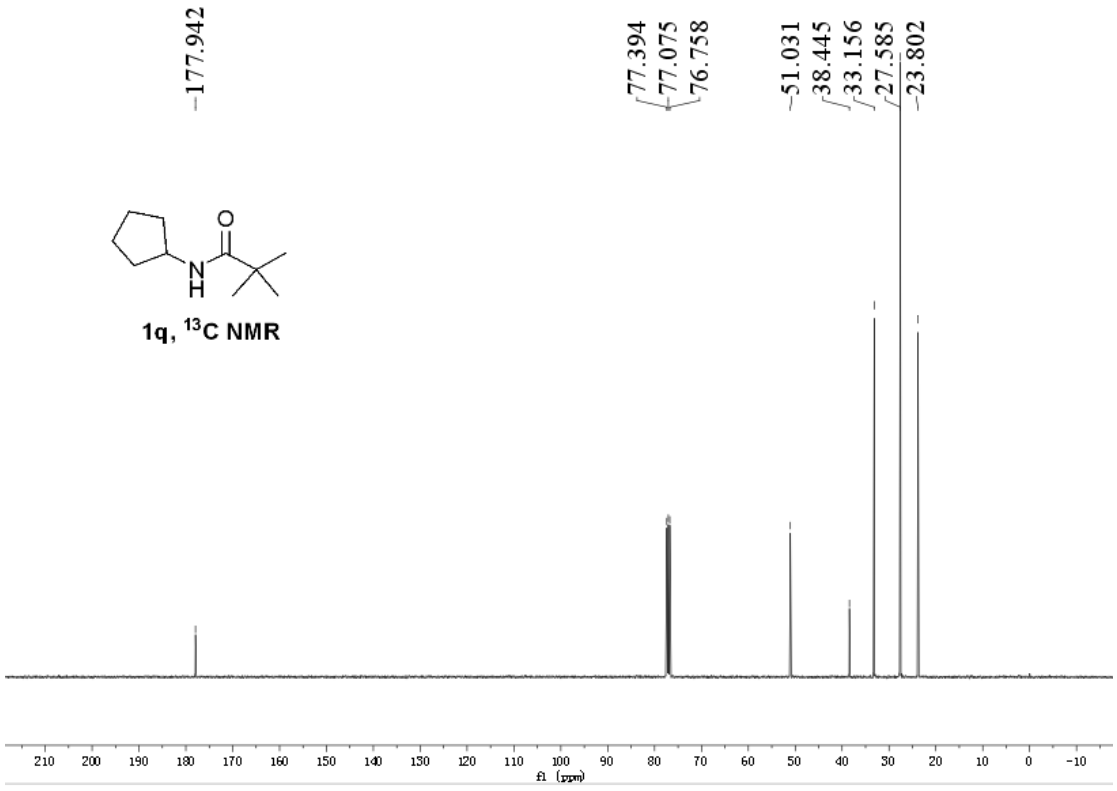
1p, ^{1}H NMR


177.414
77.397
77.079
76.761
47.875
38.488
33.116
27.583
25.577
24.874
O
N
H
1p, ^{13}C NMR
f1 (ppm)

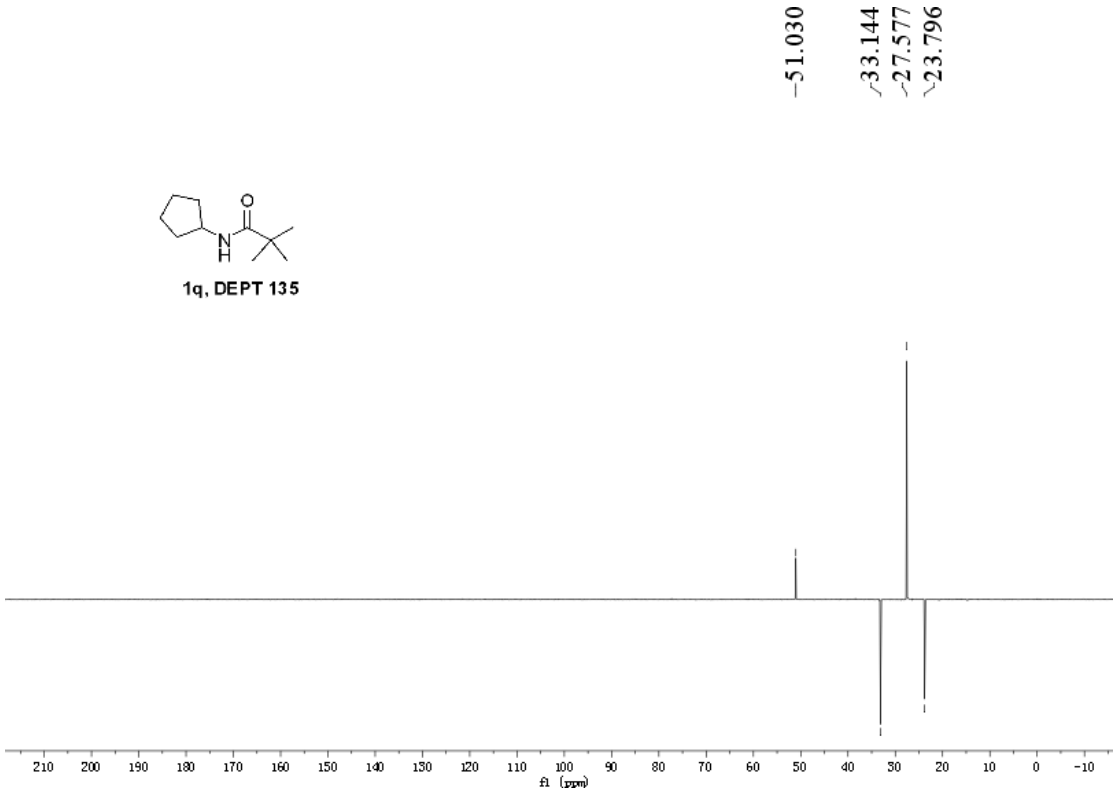

47.878
33.105
27.575
25.570
24.868
O
N
H
1p, DEPT 135
f1 (ppm)

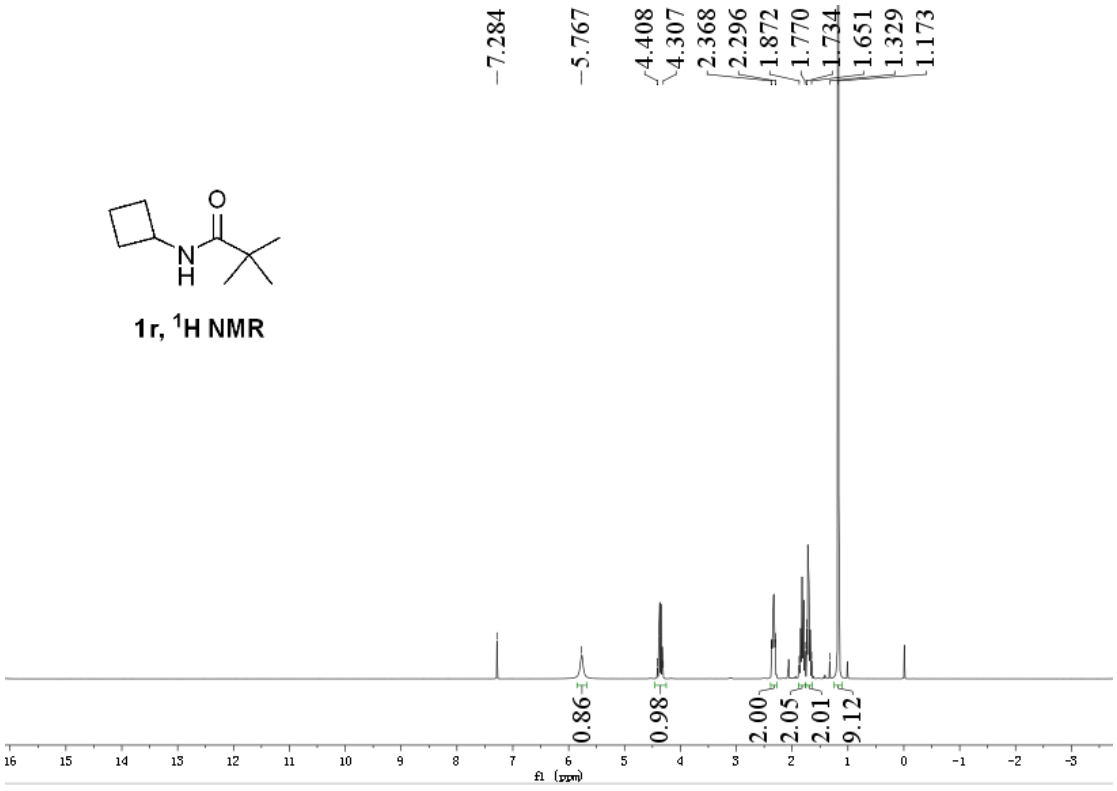


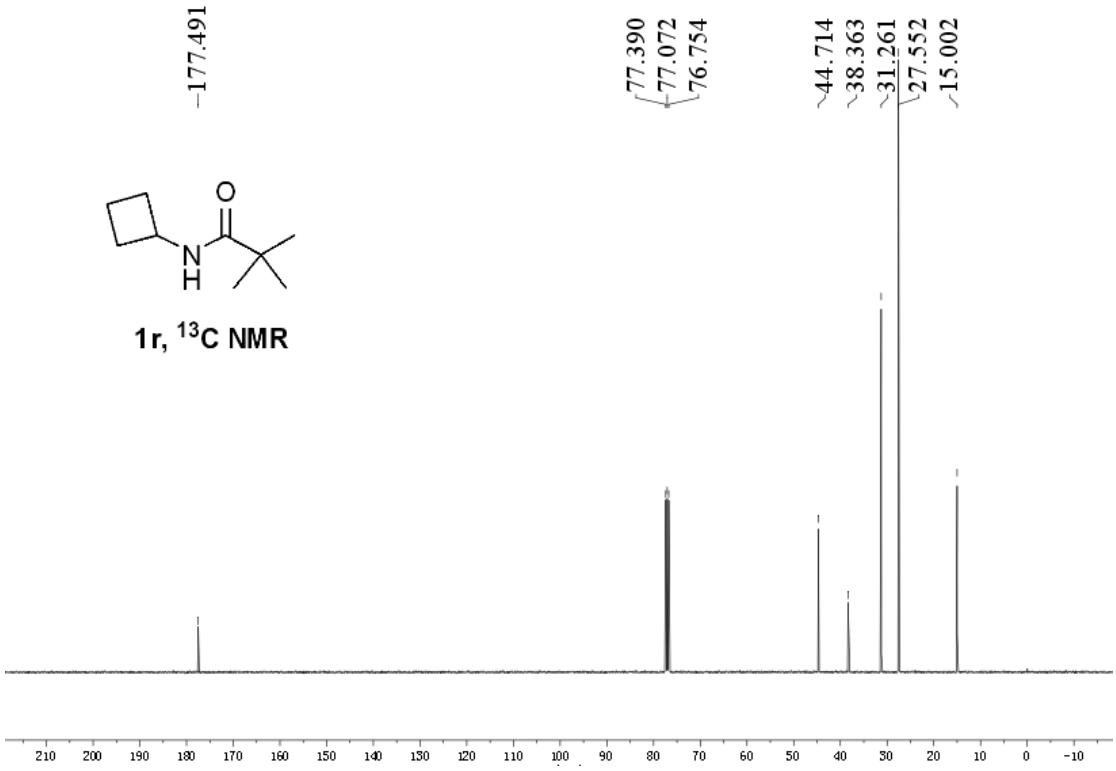
1q, ^{1}H NMR

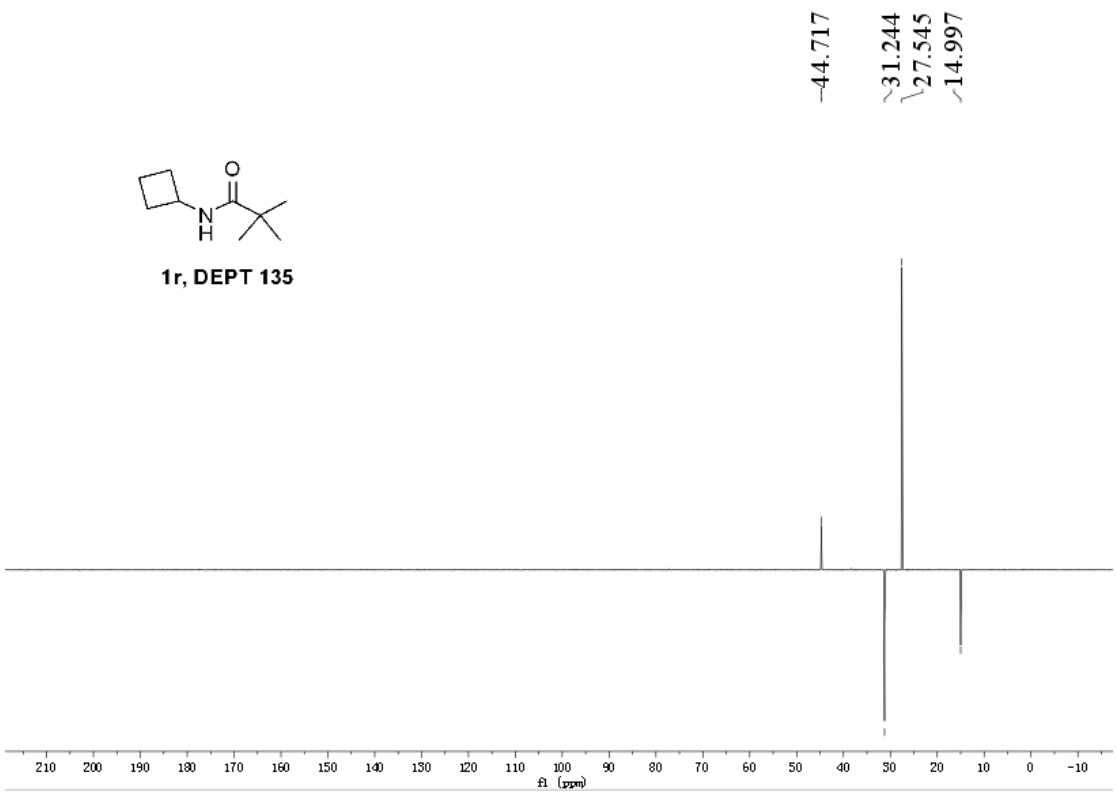


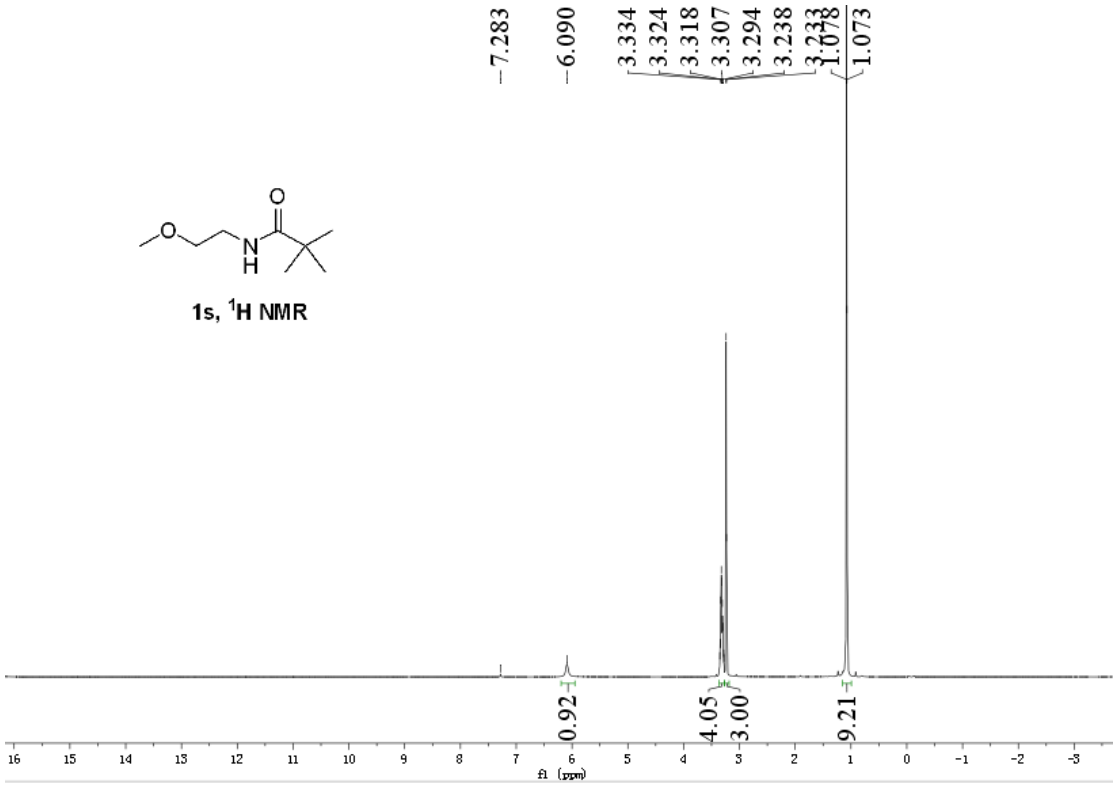
1q, ^{13}C NMR


51.030
33.144
27.577
23.796
1q, DEPT 135
f1 (ppm)

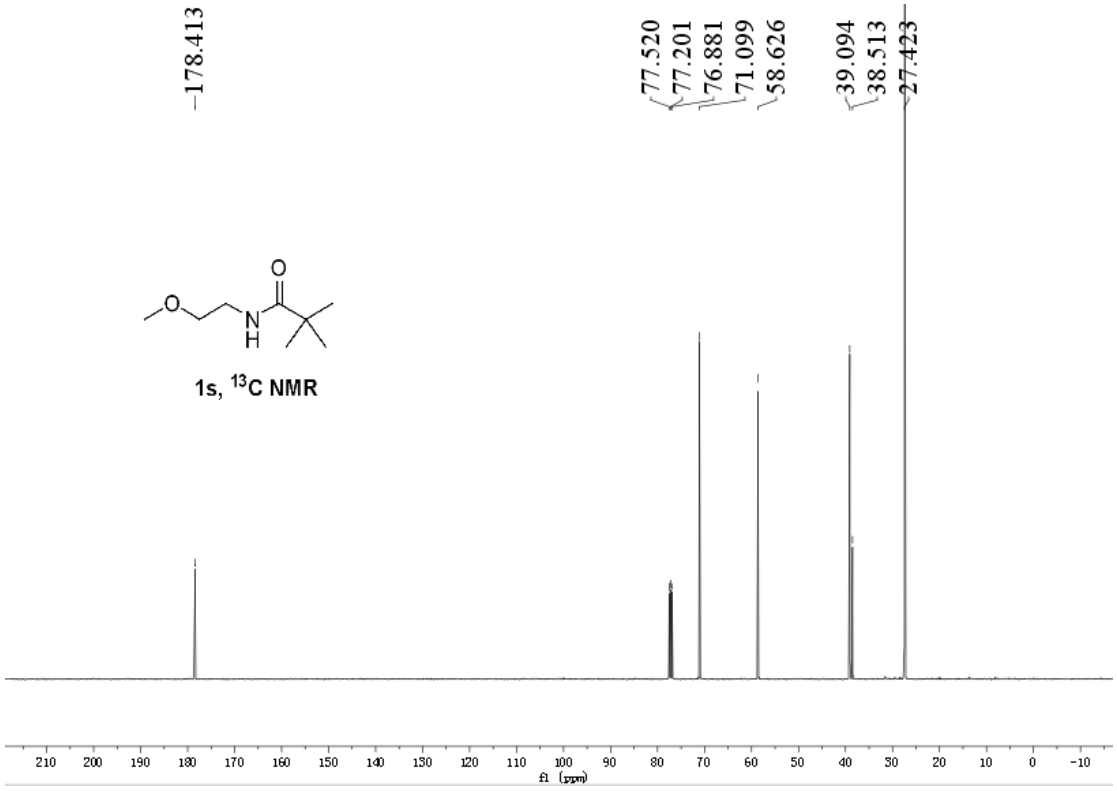

7.284
5.767
4.408
4.307
2.368
2.296
1.872
1.770
1.734
1.651
1.329
1.173
1r, 1H NMR
0.86
0.98
2.00
2.05
2.01
9.12
f1 (ppm)


177.491
77.390
77.072
76.754
44.714
38.363
31.261
27.552
15.002
O
N
H
1r, ^{13}C NMR

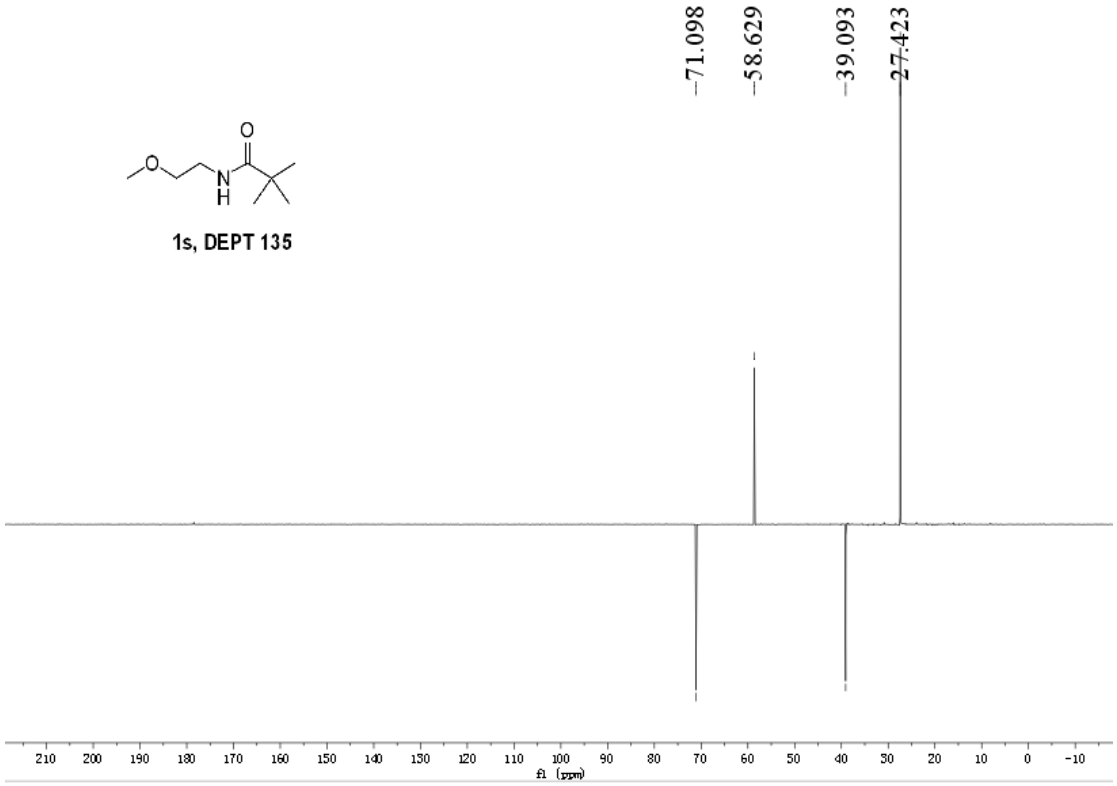

44.717
31.244
27.545
14.997
O
N
H
1r, DEPT 135
f1 (ppm)



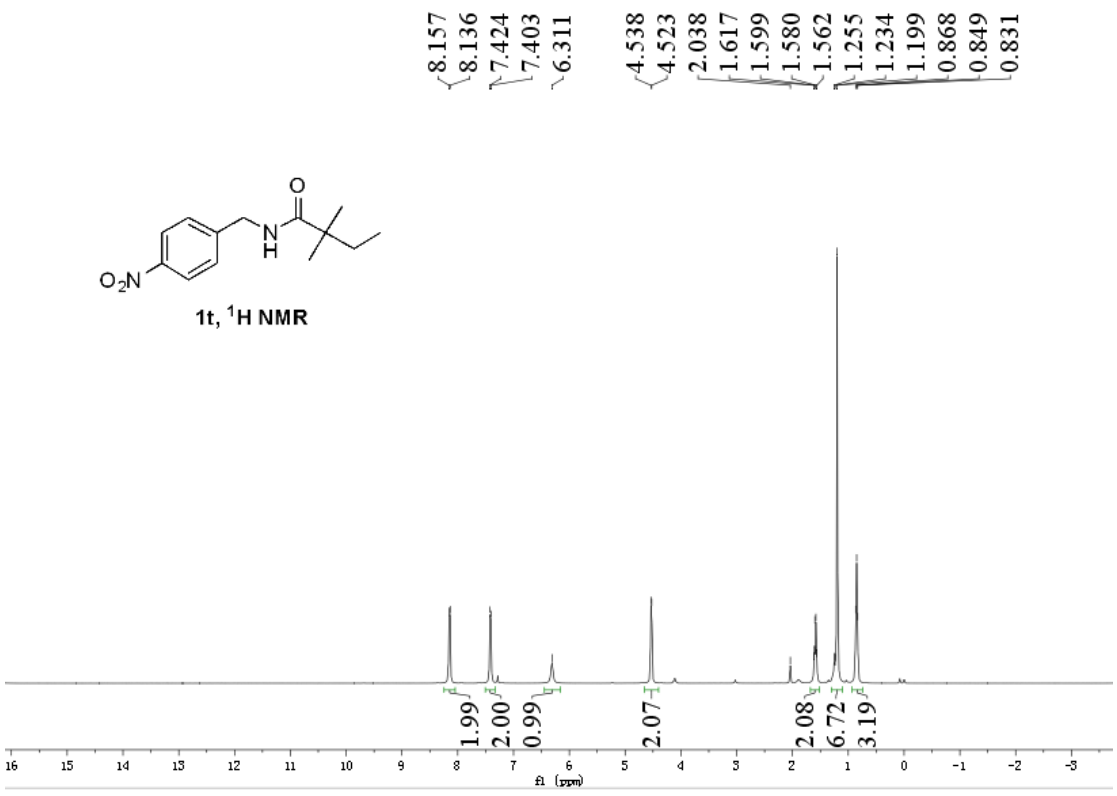
1s, ^{1}H NMR



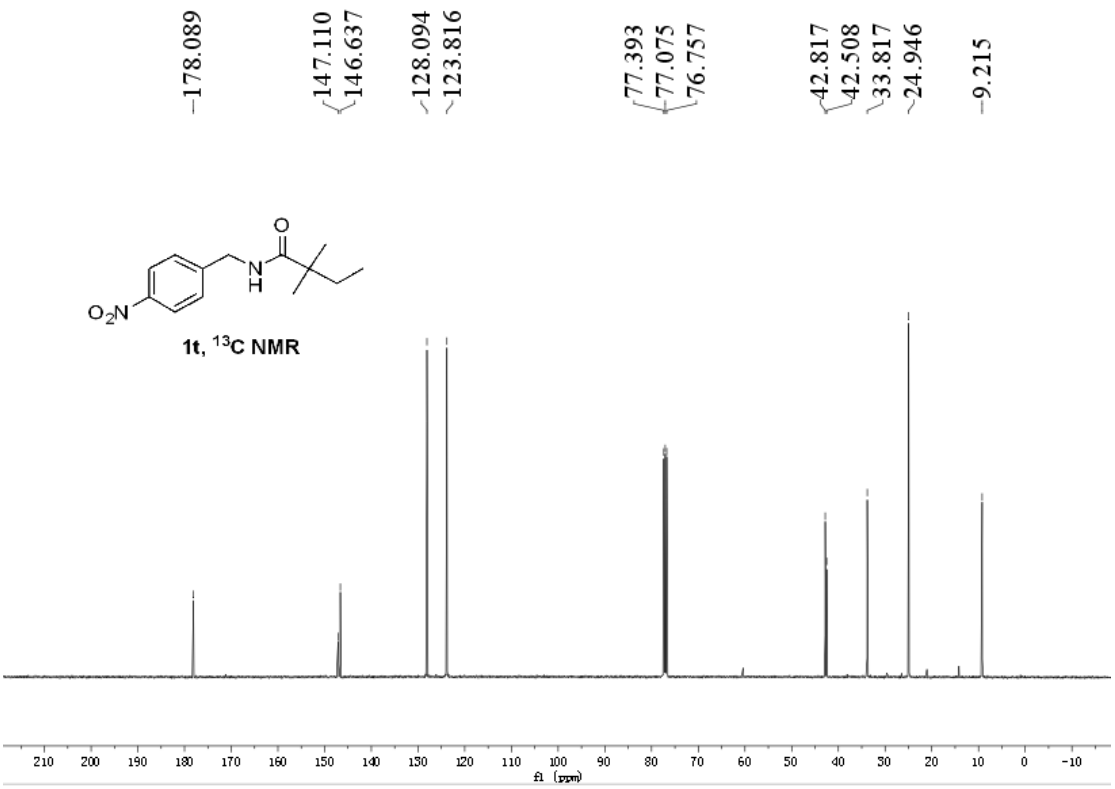
1s, ^{13}C NMR

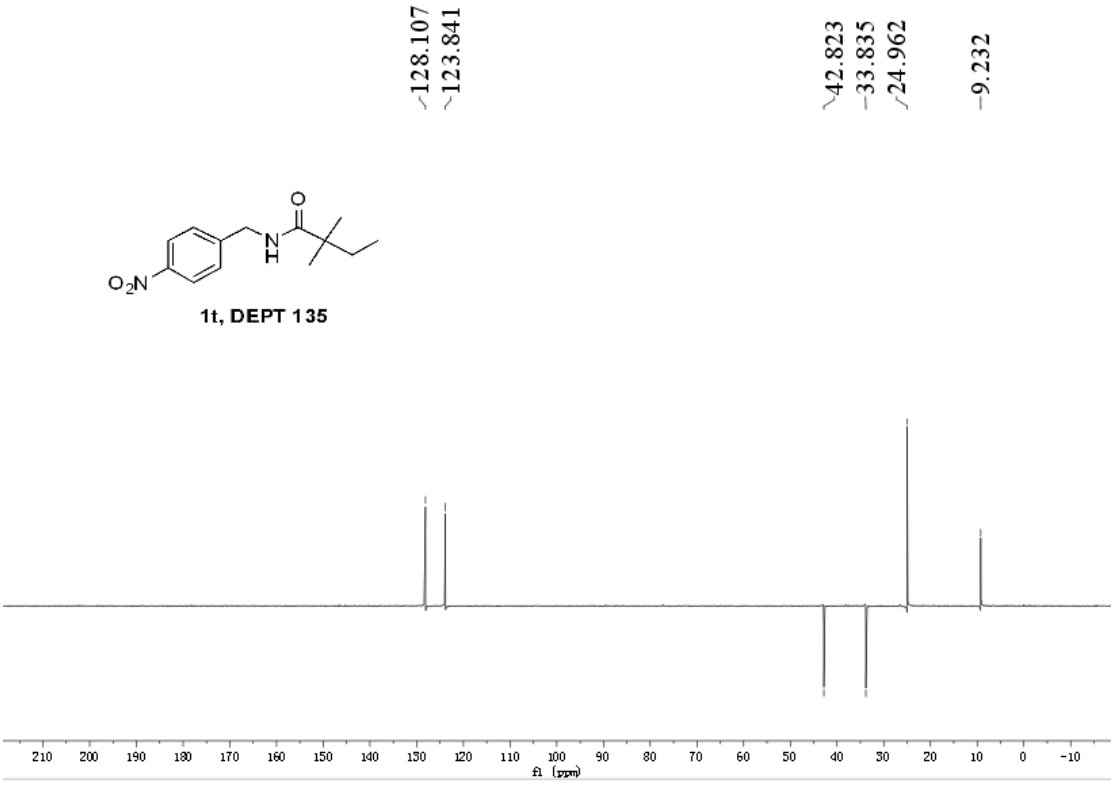


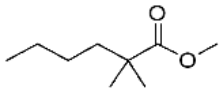
1s, DEPT 135



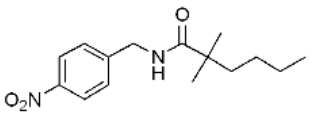
1t, ^{1}H NMR


178.089
147.110
146.637
128.094
123.816
77.393
77.075
76.757
42.817
42.508
33.817
24.946
9.215
O2N
1t, 13C NMR
fl (ppm)

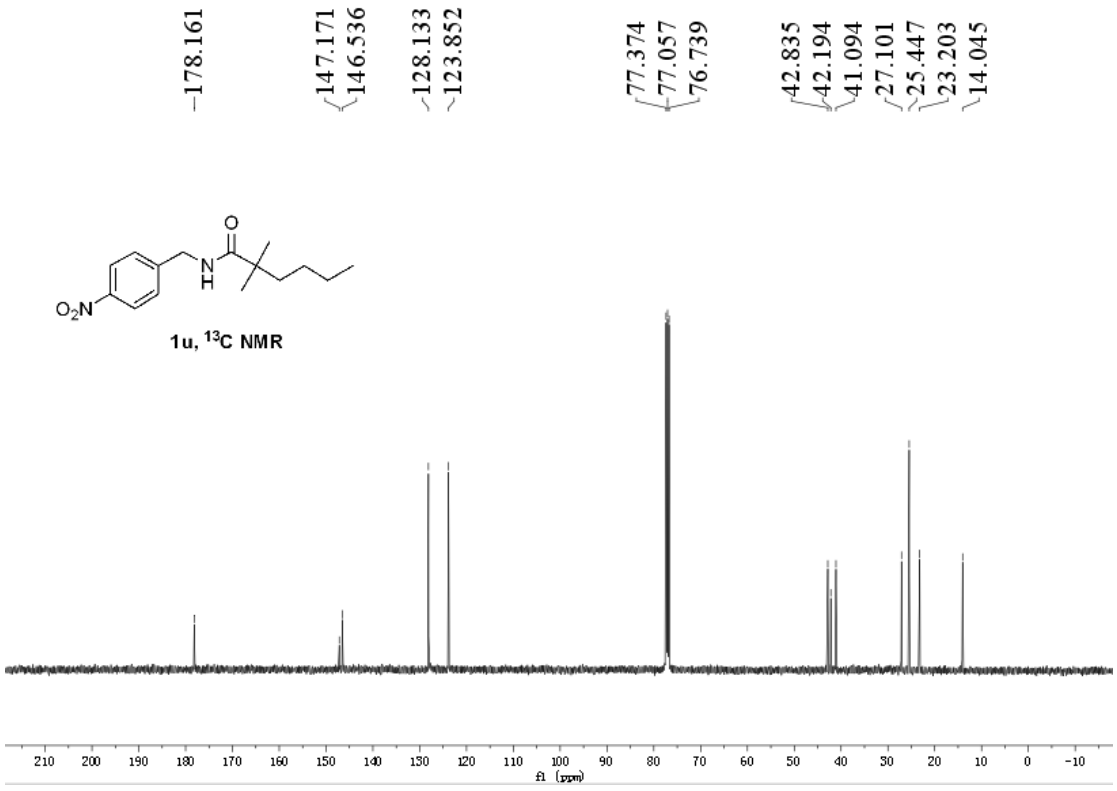

128.107
123.841
42.823
33.835
24.962
9.232
O2N
1t, DEPT 135
fl (ppm)

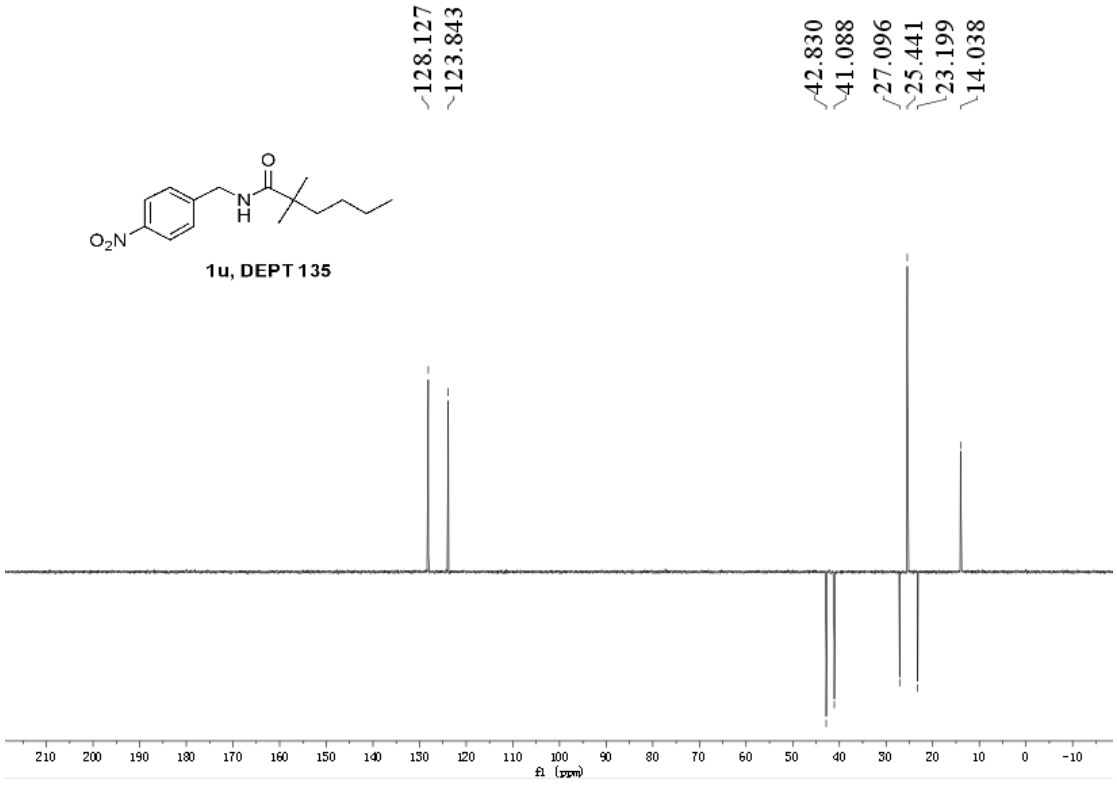


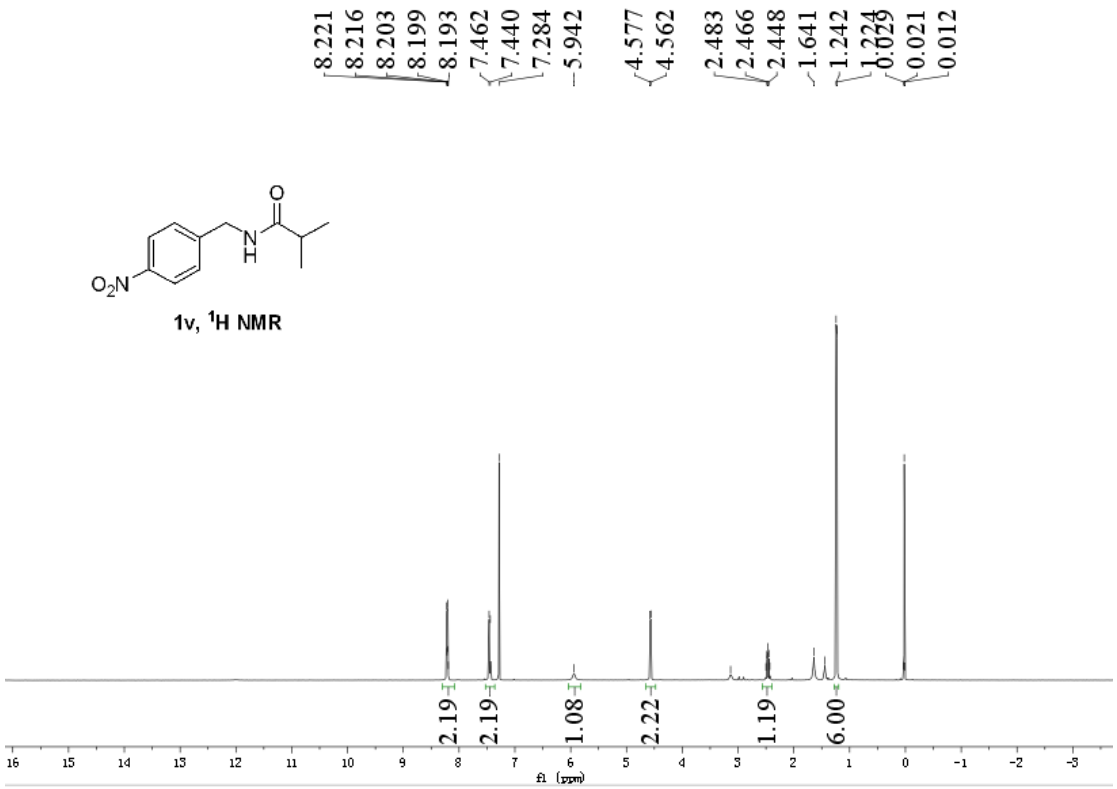
1u-1, ^{1}H NMR



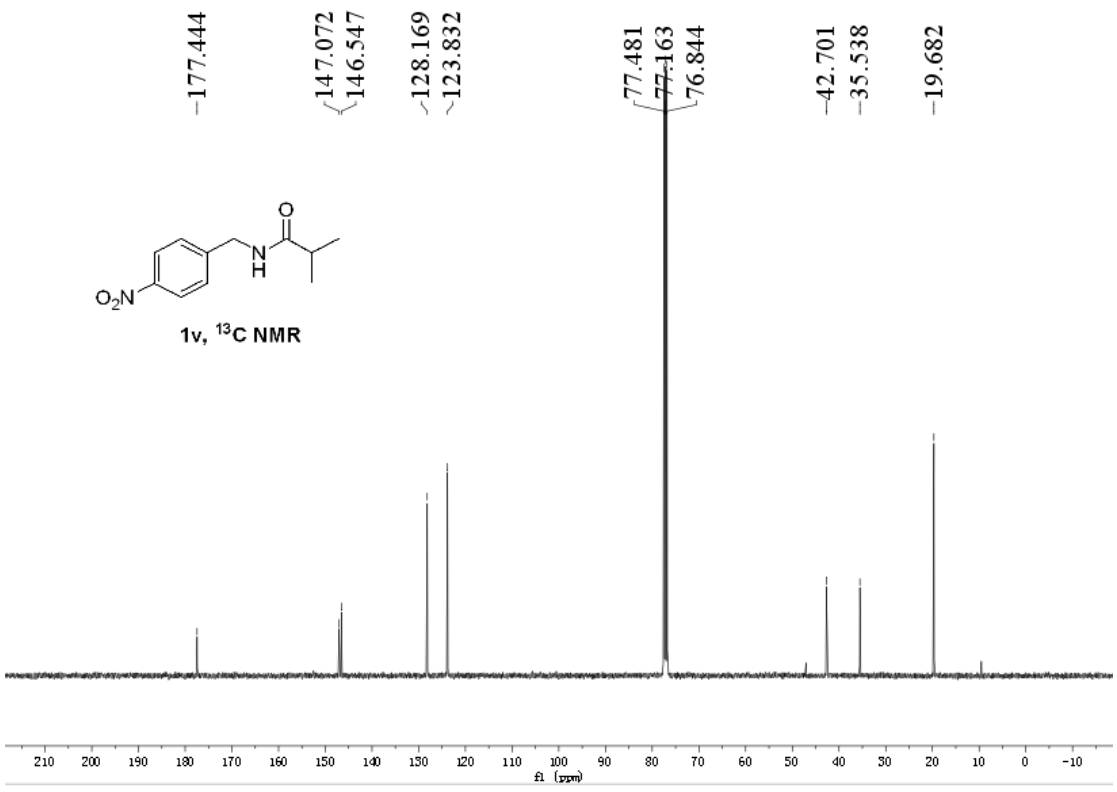
1u, ^{1}H NMR


178.161
147.171
146.536
128.133
123.852
77.374
77.057
76.739
42.835
42.194
41.094
27.101
25.447
23.203
14.045
1u, 13C NMR
f1 (ppm)

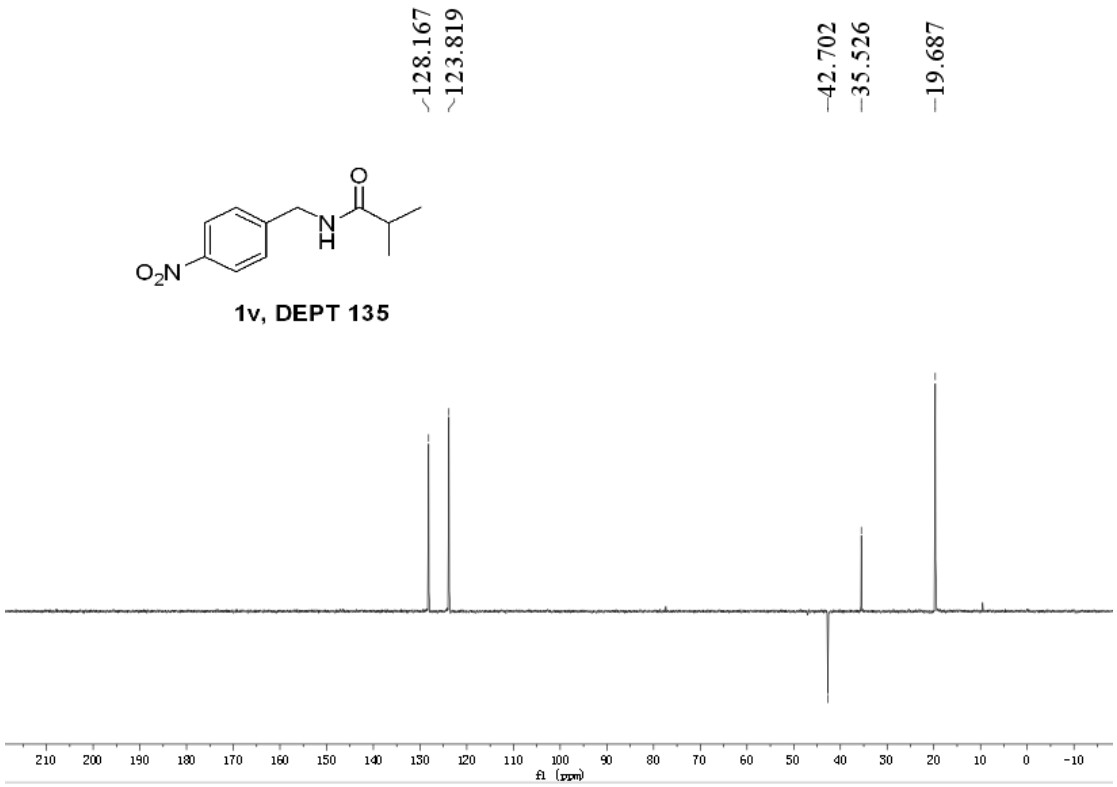

128.127
123.843
42.830
41.088
27.096
25.441
23.199
14.038
1u, DEPT 135
f1 (ppm)

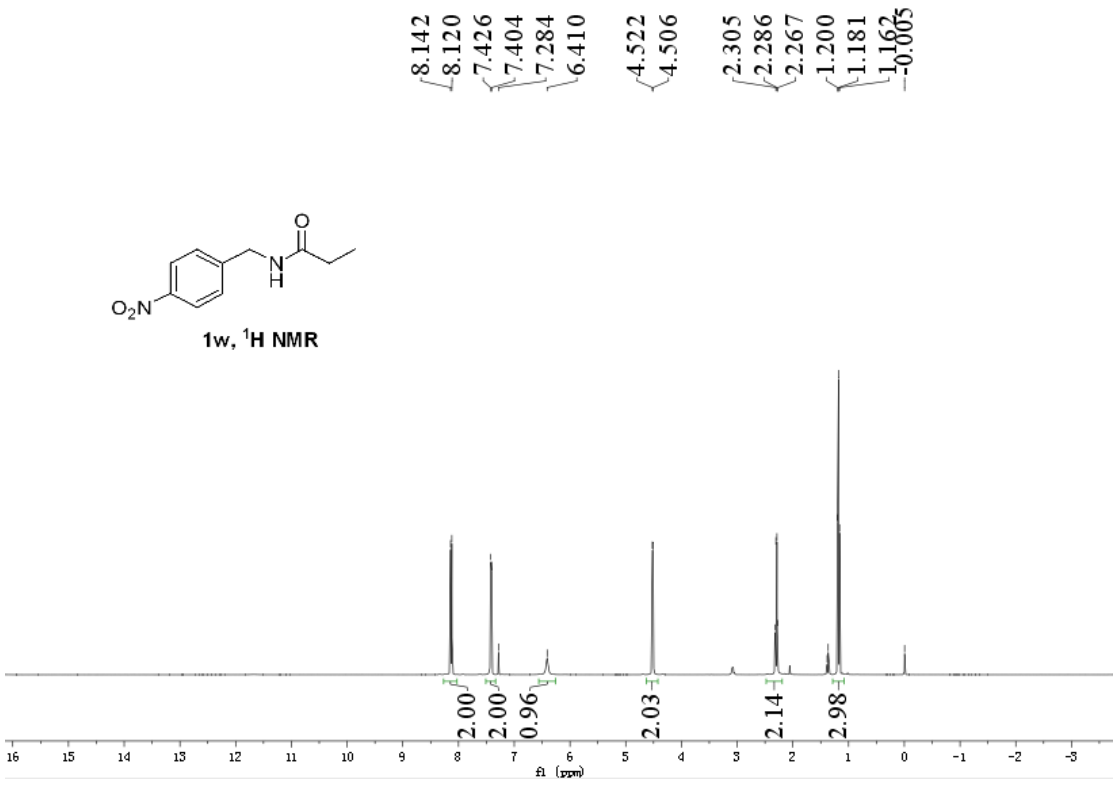


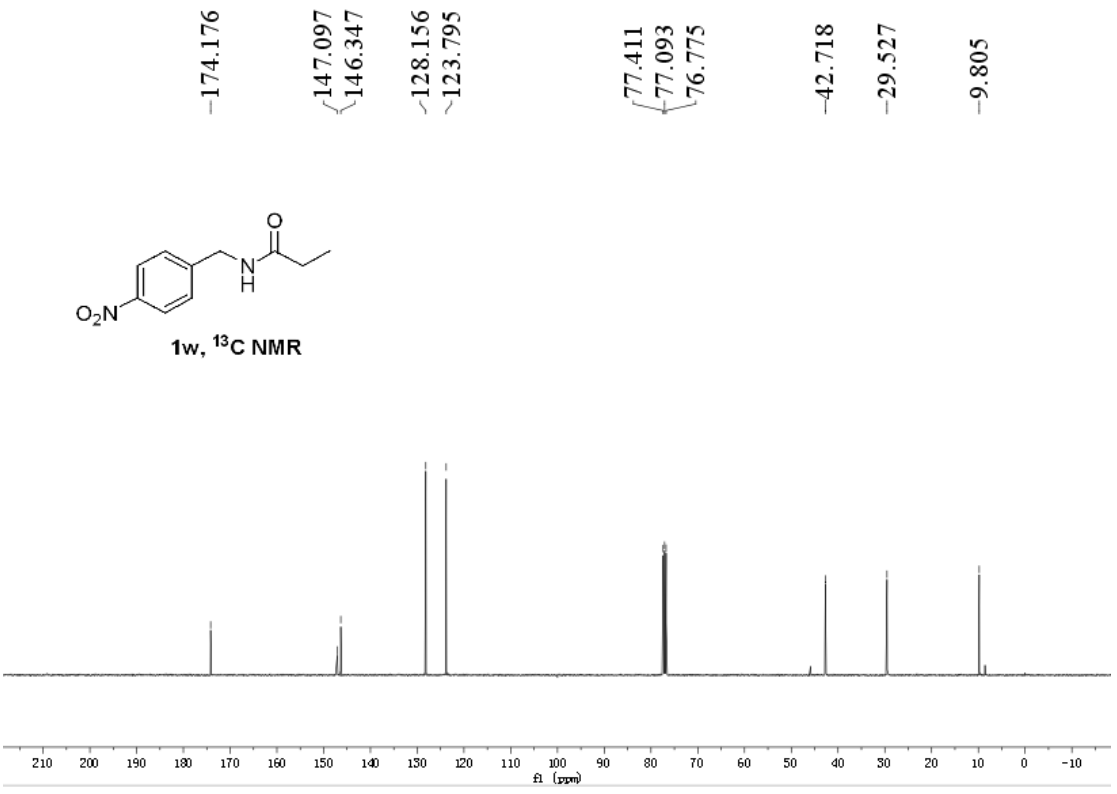
1v, ^{1}H NMR



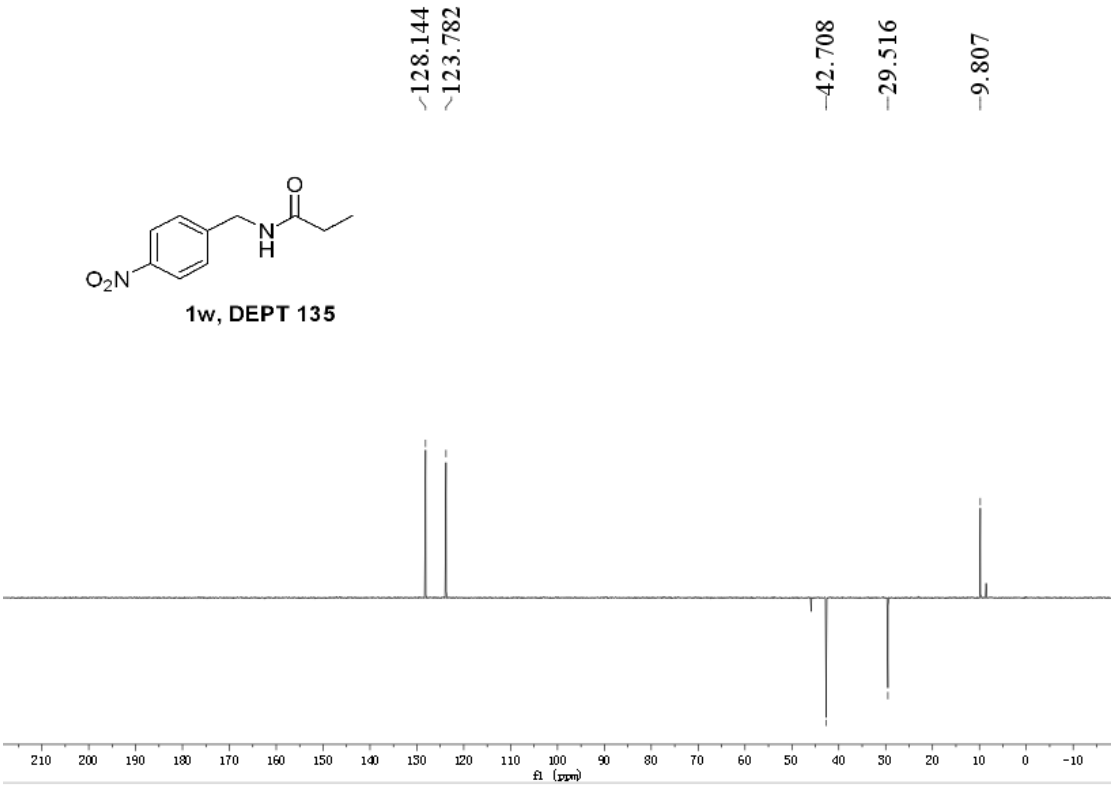
1v, ^{13}C NMR


128.167
123.819
42.702
35.526
19.687
O
N
H
O2N
1v, DEPT 135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

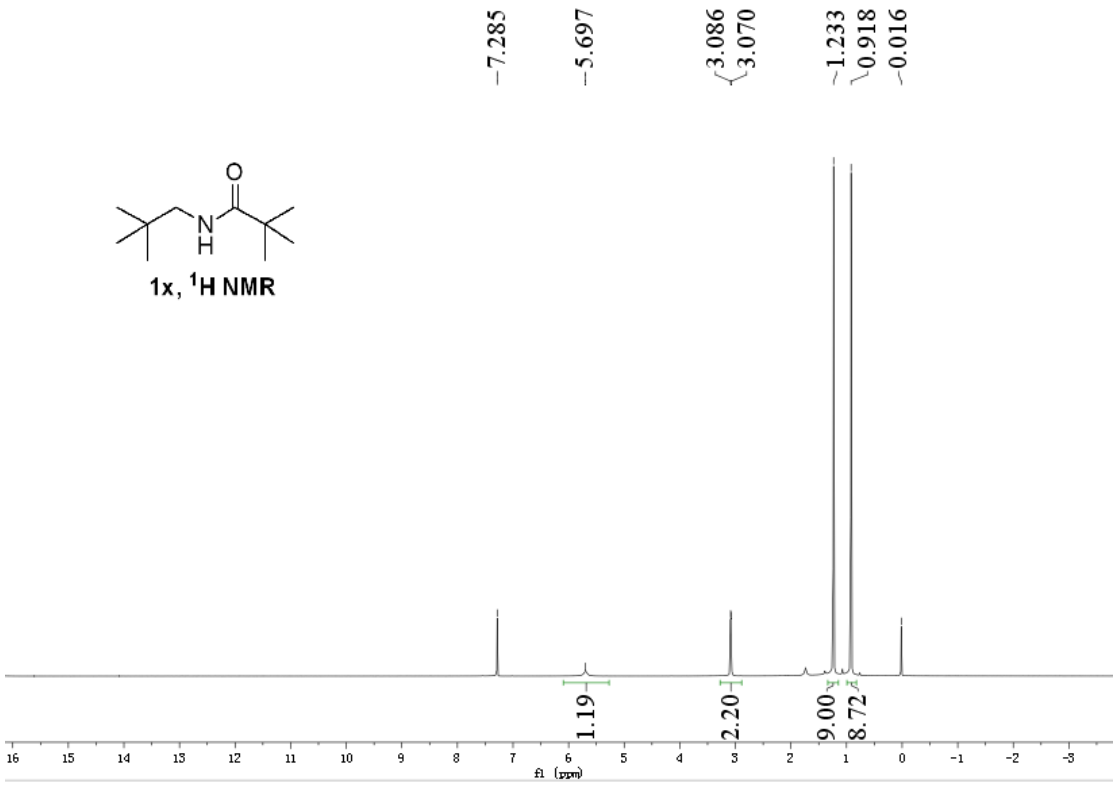

8.142
8.120
7.426
7.404
7.284
6.410
4.522
4.506
2.305
2.286
2.267
1.200
1.181
1.162
0.005
O
N
H
O2N
1w, 1H NMR
2.00
2.00
0.96
2.03
2.14
2.98
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)



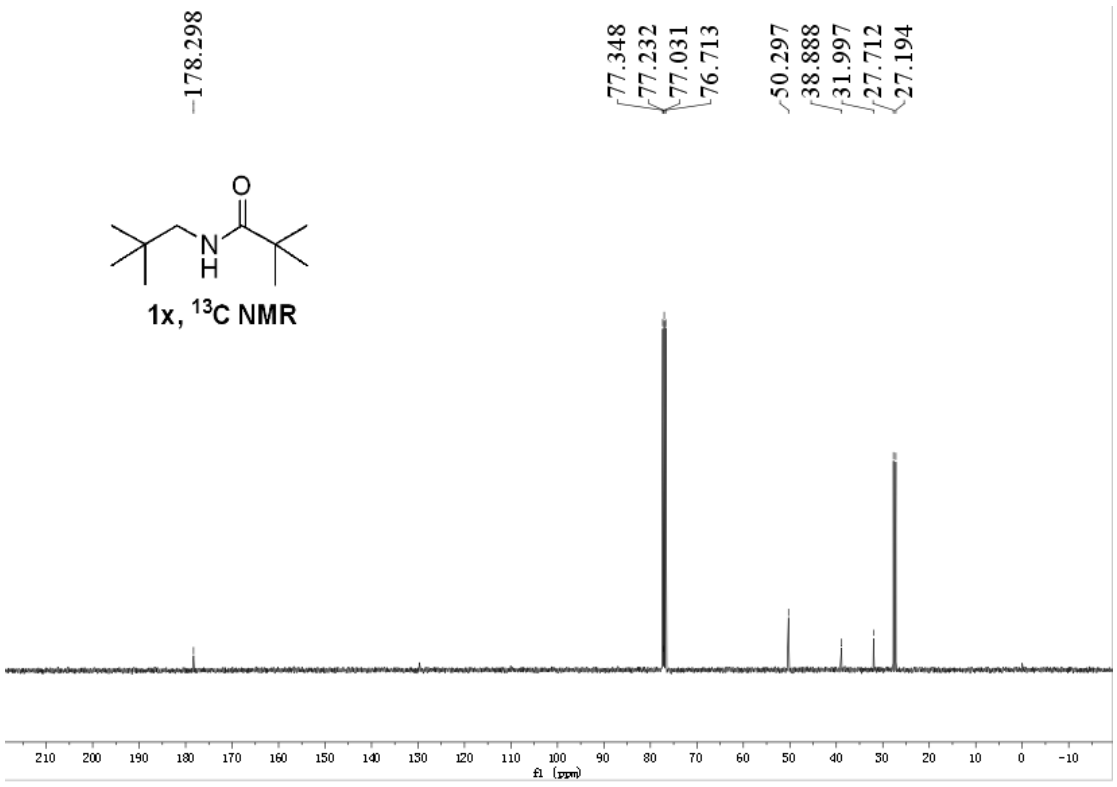
1w, ^{13}C NMR



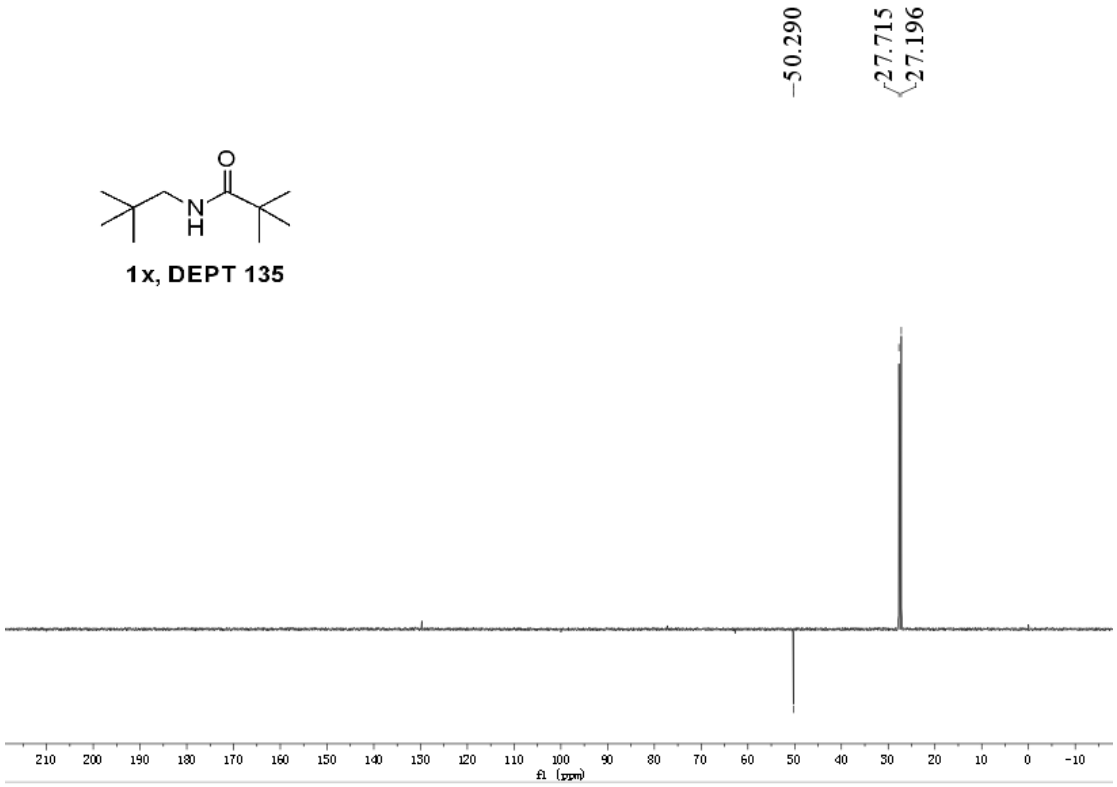
1w, DEPT 135



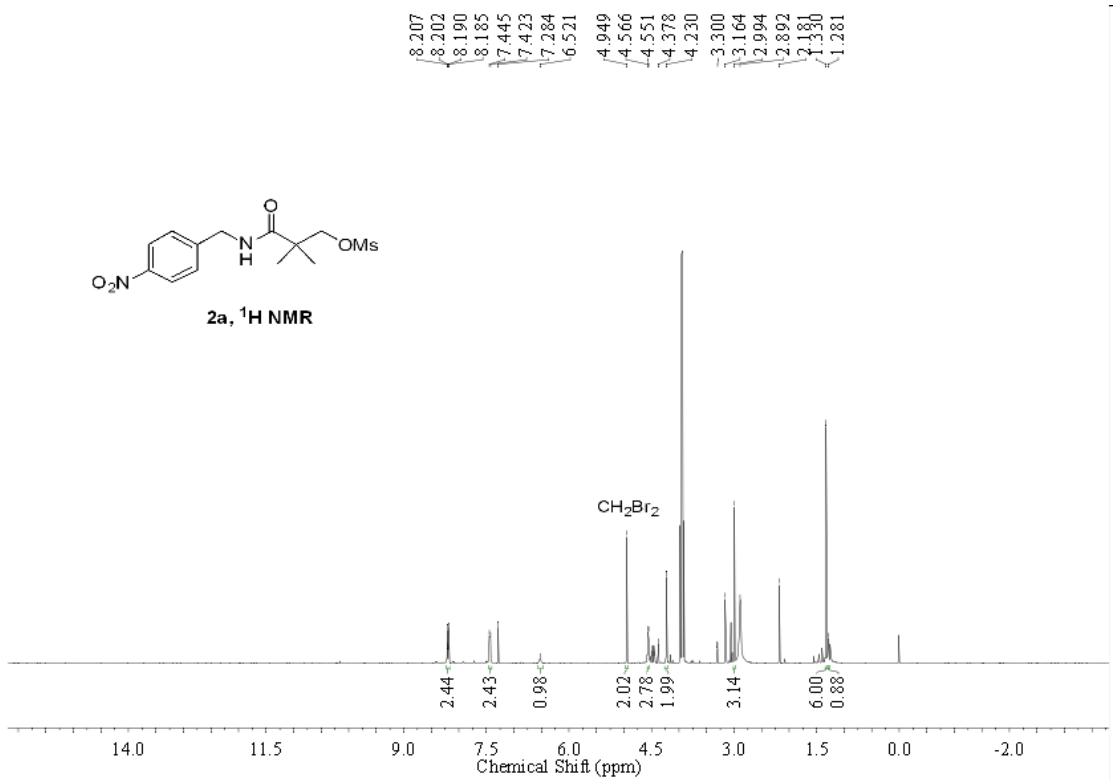
1x, ^{1}H NMR

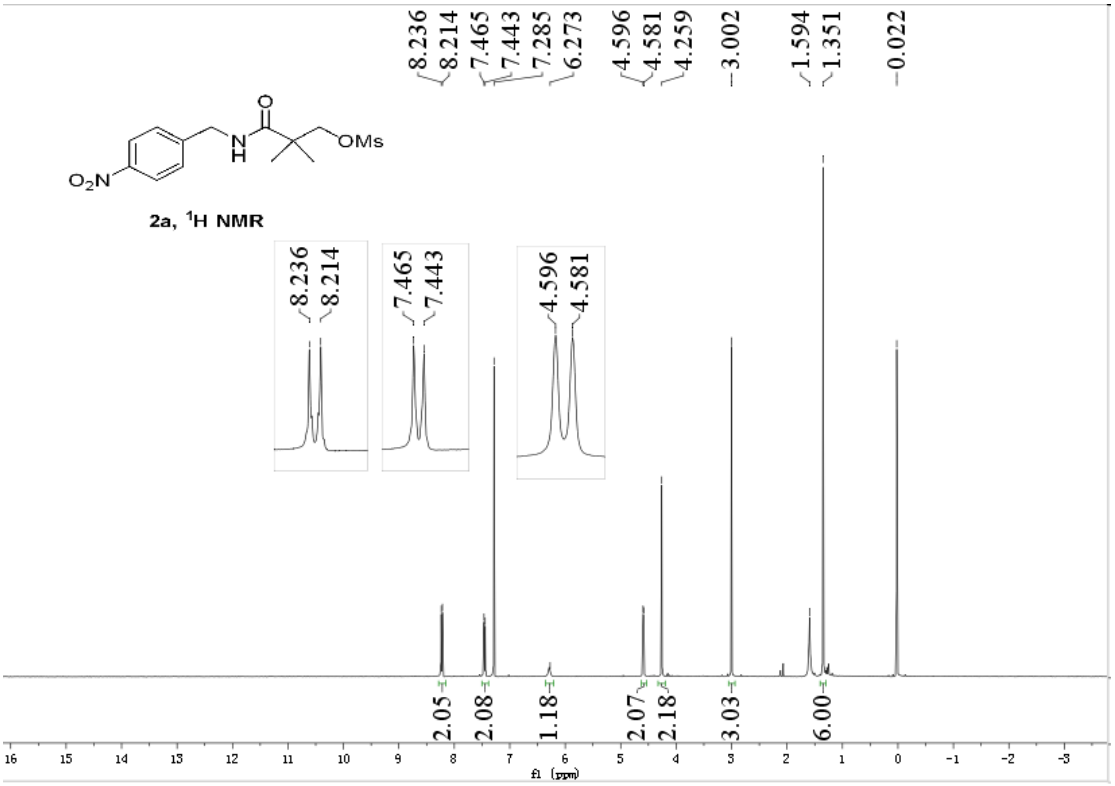


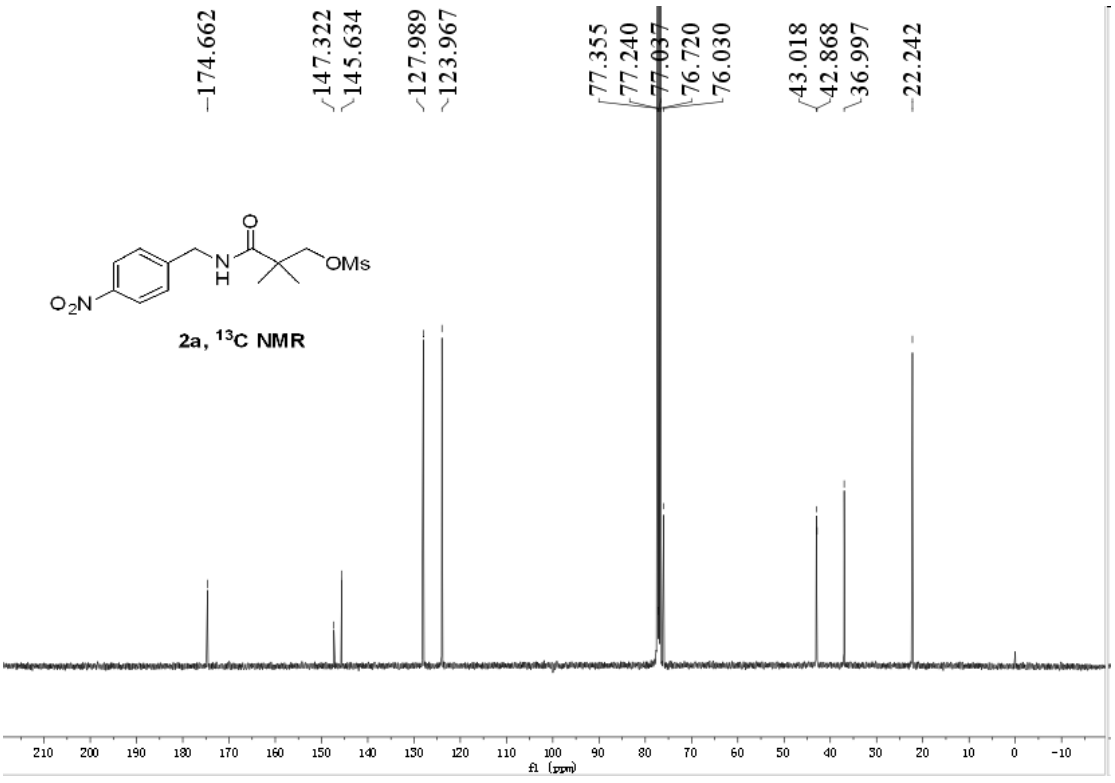
1x, ^{13}C NMR

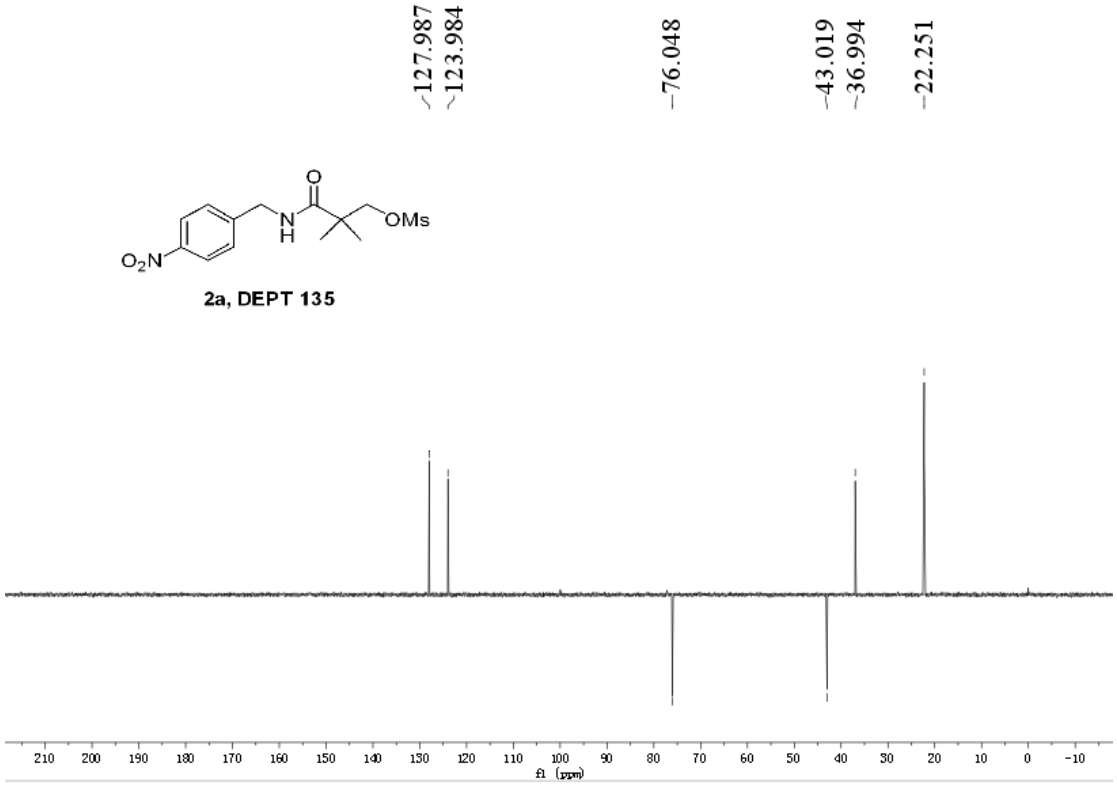


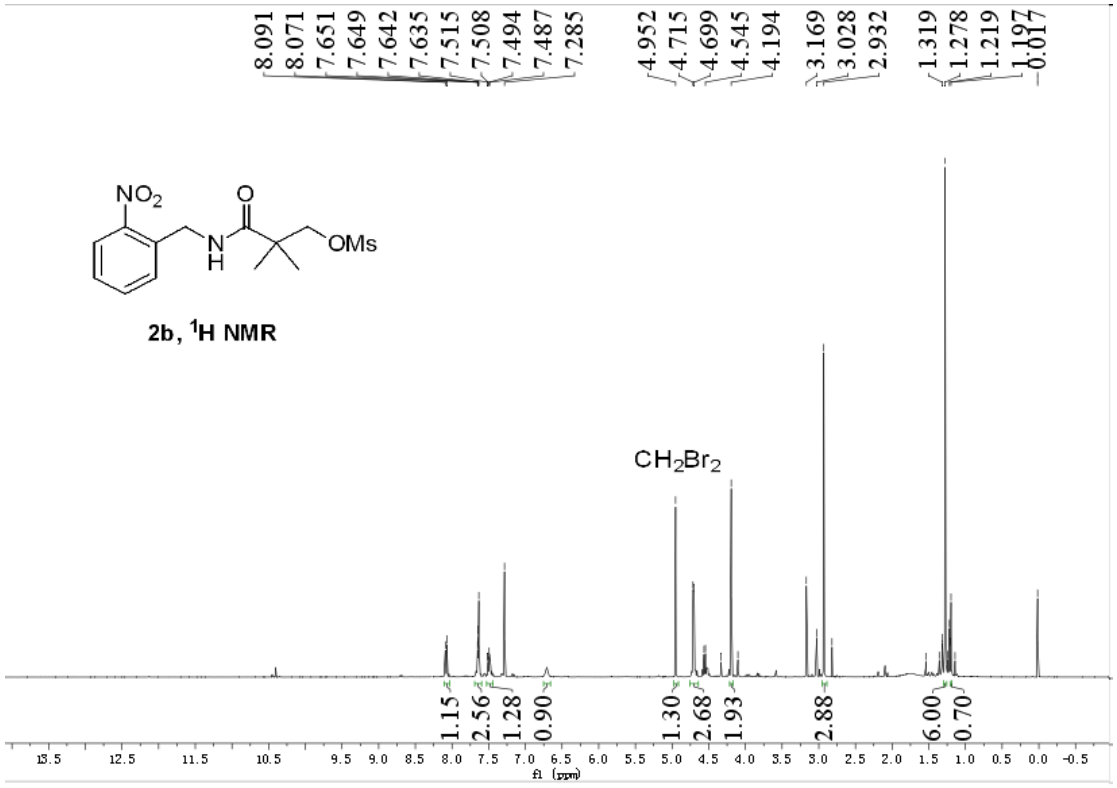
(2) *β*-Mesylated amides

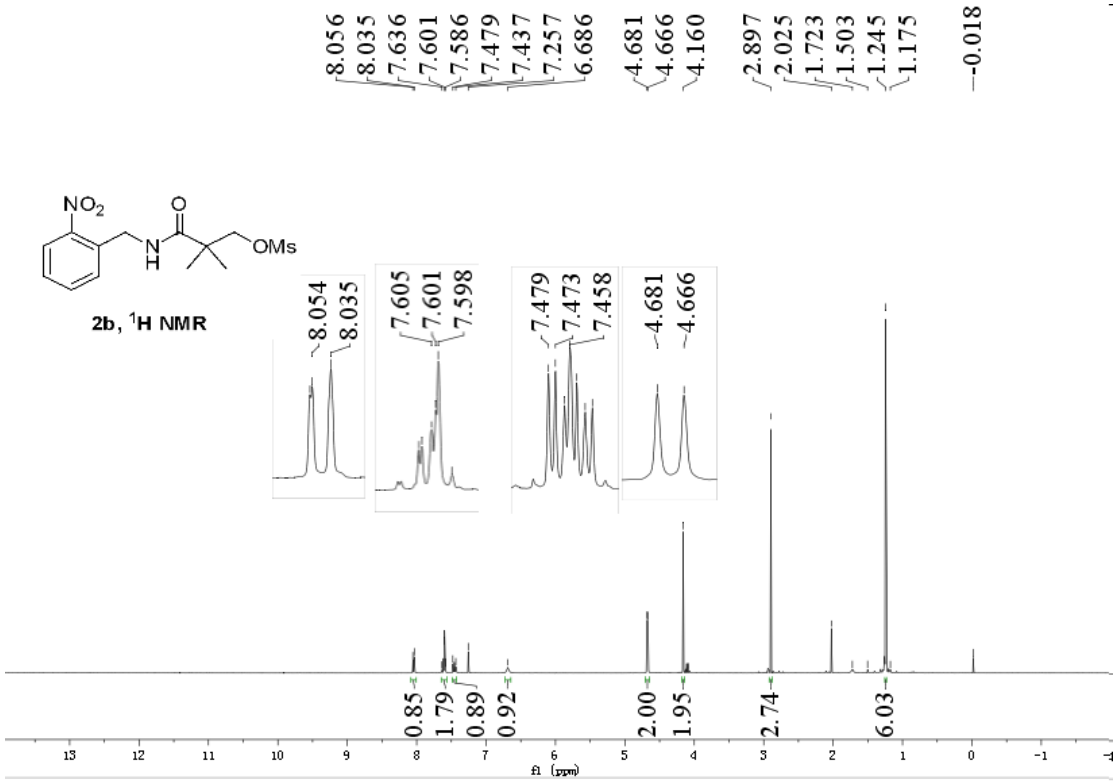


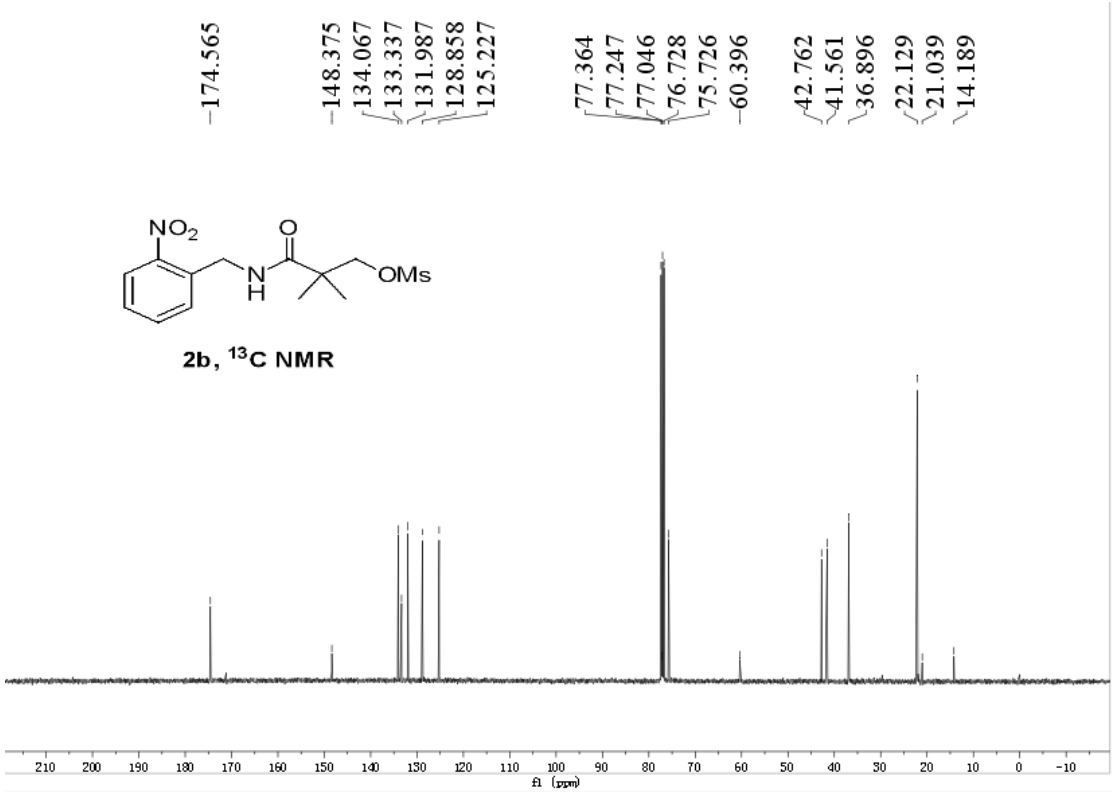

8.236
8.214
7.465
7.443
7.285
6.273
4.596
4.581
4.259
3.002
1.594
1.351
0.022
OMs
O2N
2a, 1H NMR
2.05
2.08
1.18
2.07
2.18
3.03
6.00
f1 (ppm)

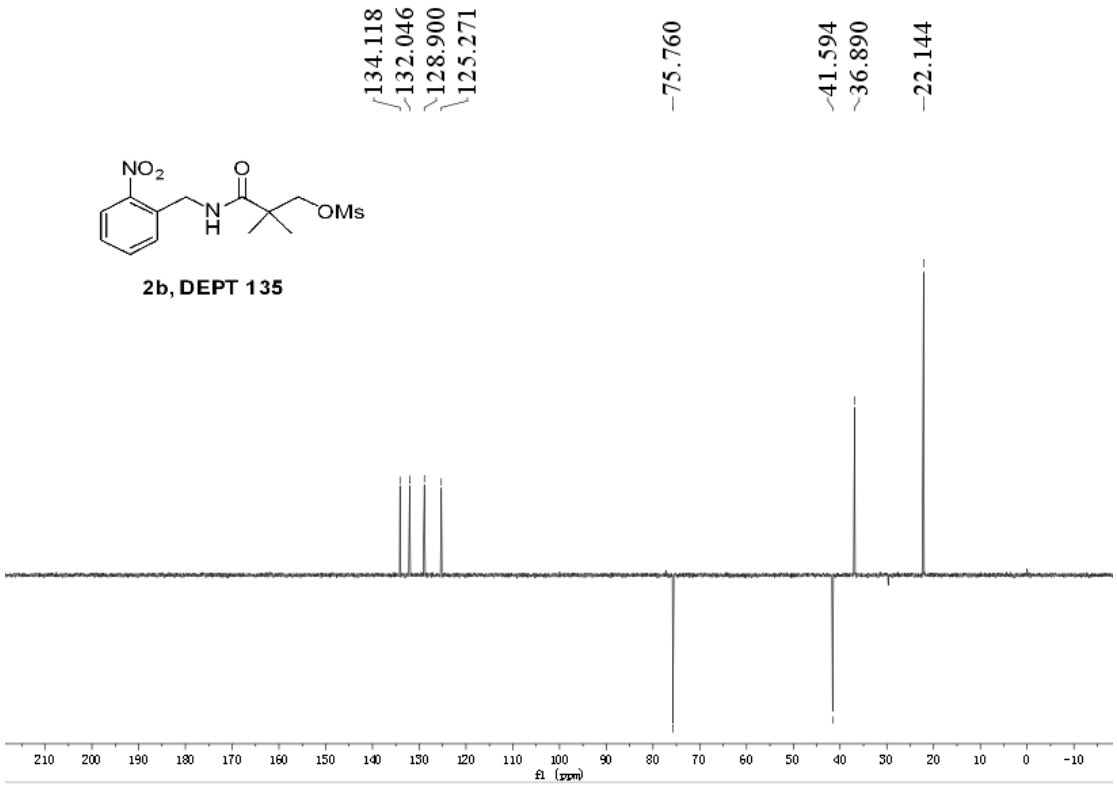

174.662
147.322
145.634
127.989
123.967
77.355
77.240
77.037
76.720
76.030
43.018
42.868
36.997
22.242
OMs
O2N
2a, 13C NMR
f1 (ppm)

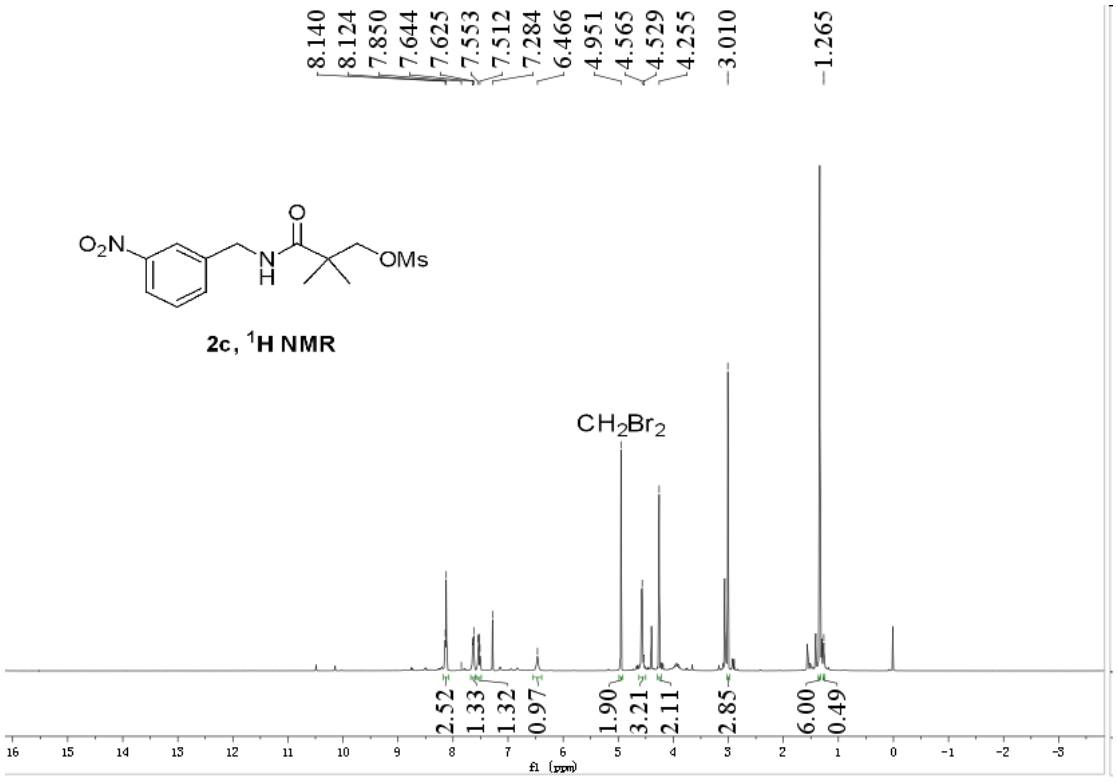

127.987
123.984
76.048
43.019
36.994
22.251
O
N
H
OMs
O2N
2a, DEPT 135
f1 (ppm)

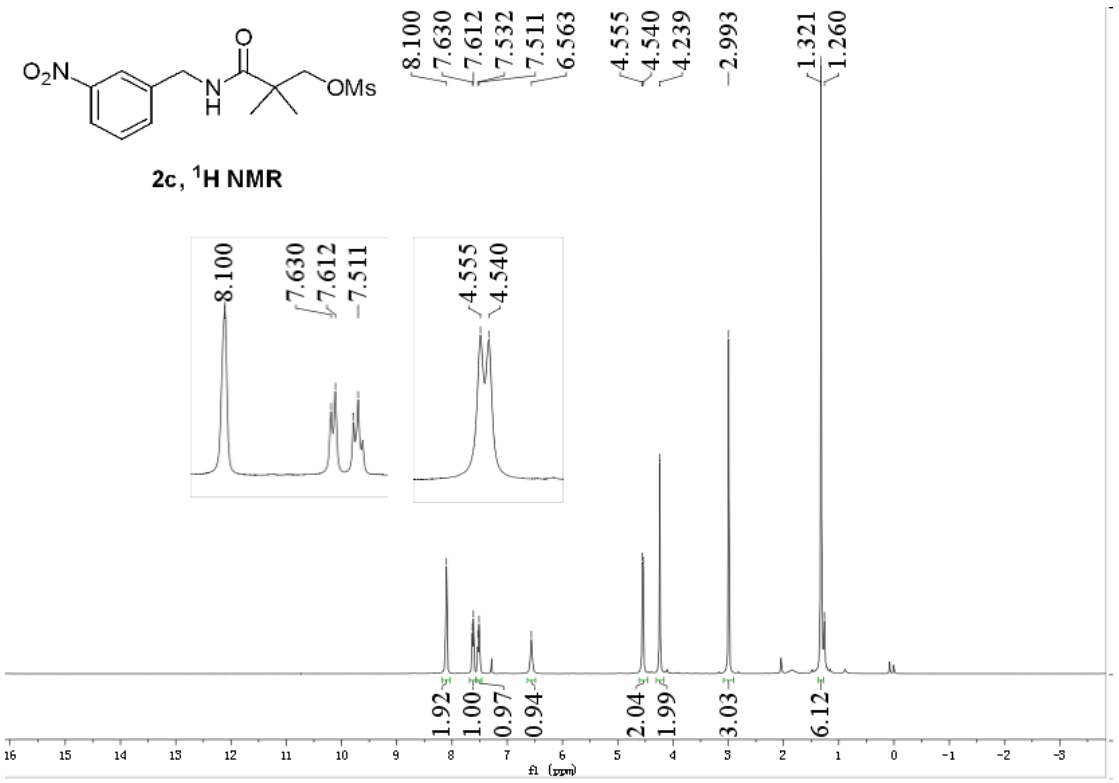

8.091
8.071
7.651
7.649
7.642
7.635
7.515
7.508
7.494
7.487
7.285
4.952
4.715
4.699
4.545
4.194
3.169
3.028
2.932
1.319
1.278
1.219
1.197
0.017
NO2
O
N
H
OMs
2b, 1H NMR
CH2Br2
1.15
2.56
1.28
0.90
1.30
2.68
1.93
2.88
6.00
0.70
f1 (ppm)


8.056
8.035
7.636
7.601
7.586
7.479
7.437
7.257
6.686
4.681
4.666
4.160
2.897
2.025
1.723
1.503
1.245
1.175
-0.018
NO2
O
N
H
OMs
2b, 1H NMR
8.054
8.035
7.605
7.601
7.598
7.479
7.473
7.458
4.681
4.666
0.85
1.79
0.89
0.92
2.00
1.95
2.74
6.03
f1 (ppm)

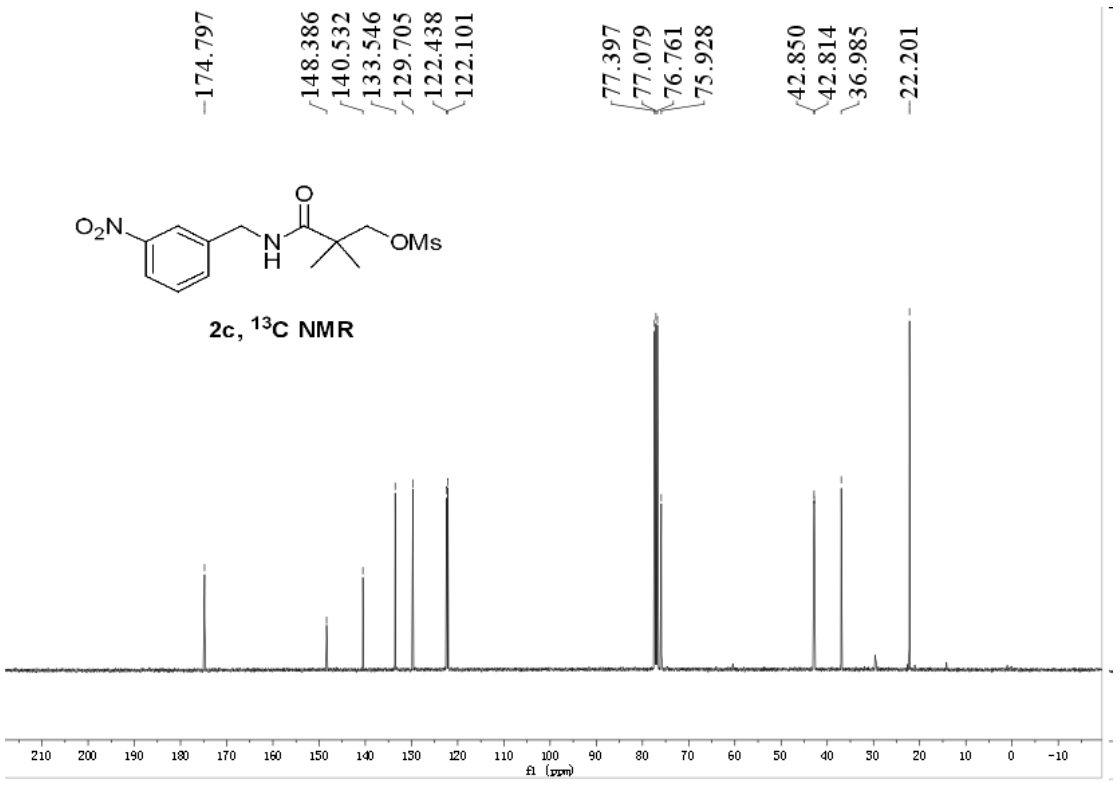

174.565
148.375
134.067
133.337
131.987
128.858
125.227
77.364
77.247
77.046
76.728
75.726
60.396
42.762
41.561
36.896
22.129
21.039
14.189
NO2
O
N
H
OMs
2b, 13C NMR
f1 (ppm)


134.118
132.046
128.900
125.271
75.760
41.594
36.890
22.144
NO2
O
N
H
OMs
2b, DEPT 135
f1 (ppm)

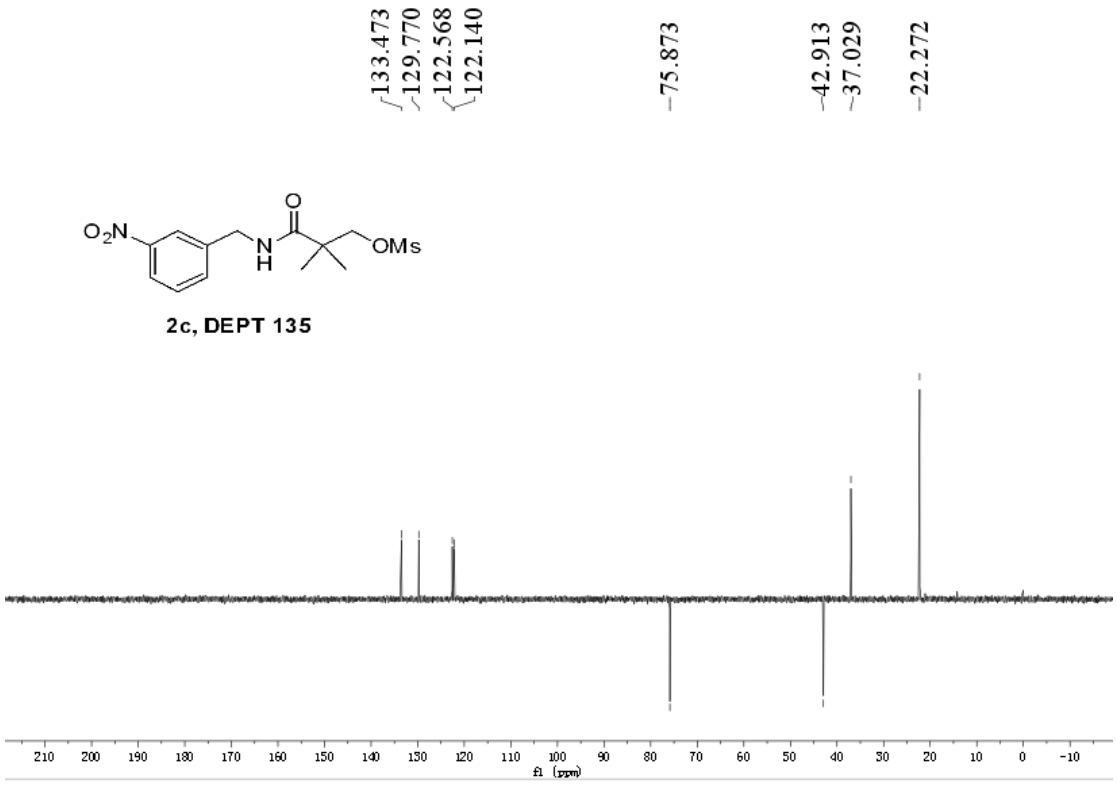

8.140
8.124
7.850
7.644
7.625
7.553
7.512
7.284
6.466
4.951
4.565
4.529
4.255
3.010
1.265
O2N
O
N
H
OMs
2c, 1H NMR
CH2Br2
2.52
1.33
1.32
0.97
1.90
3.21
2.11
2.85
6.00
0.49
f1 (ppm)

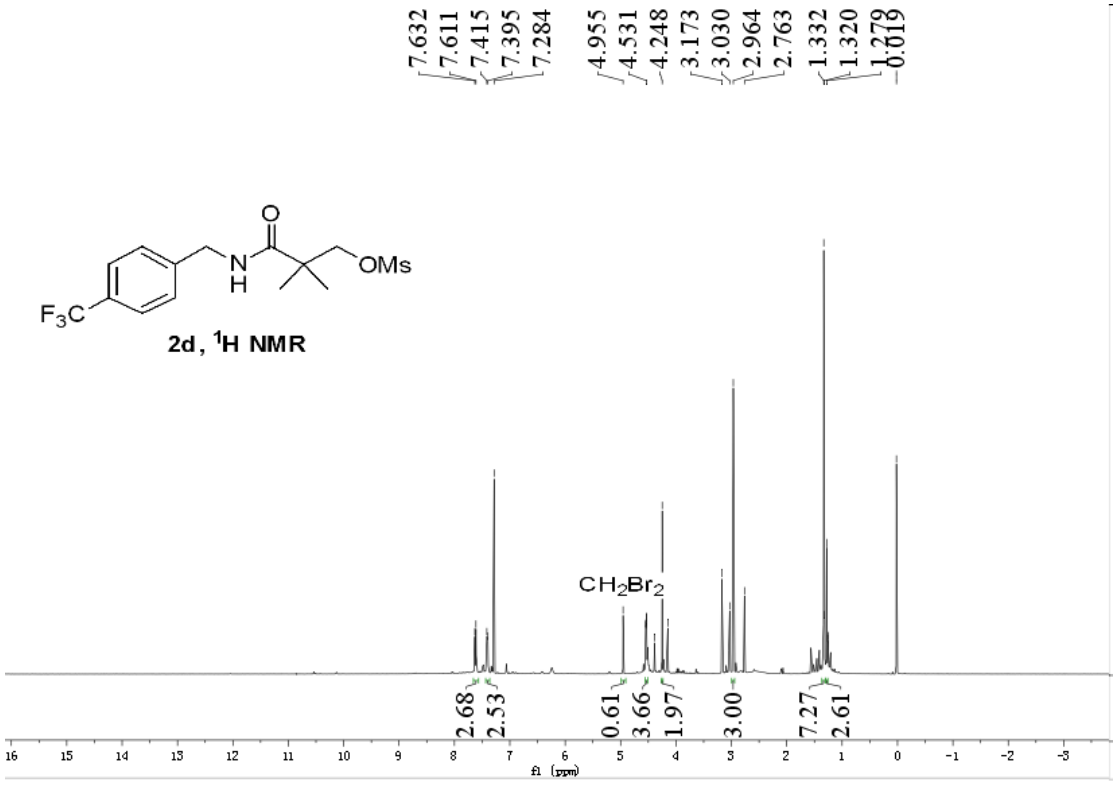


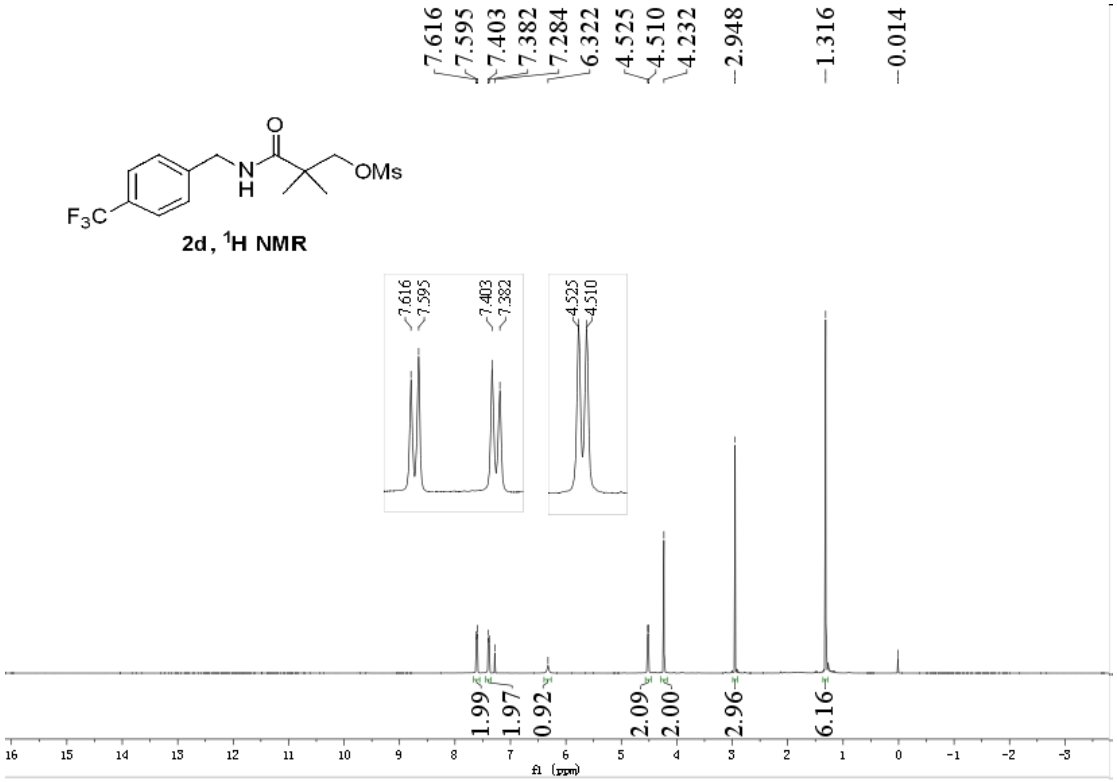
2c, ^{1}H NMR

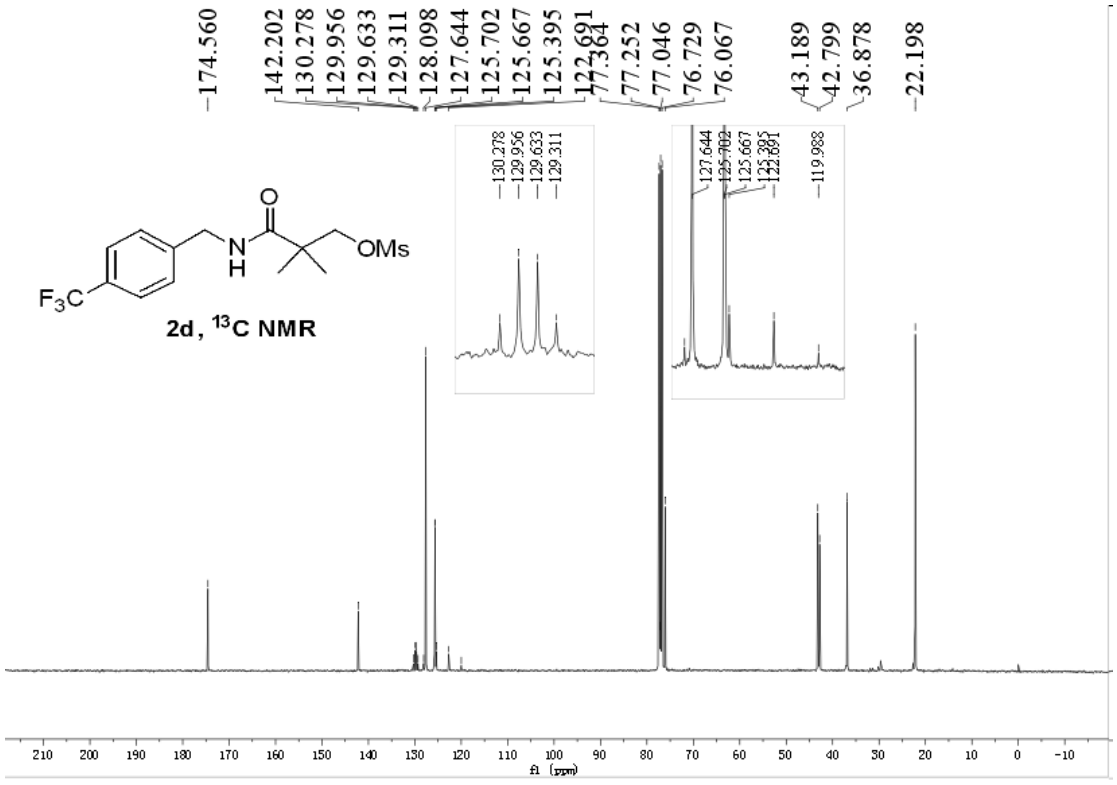


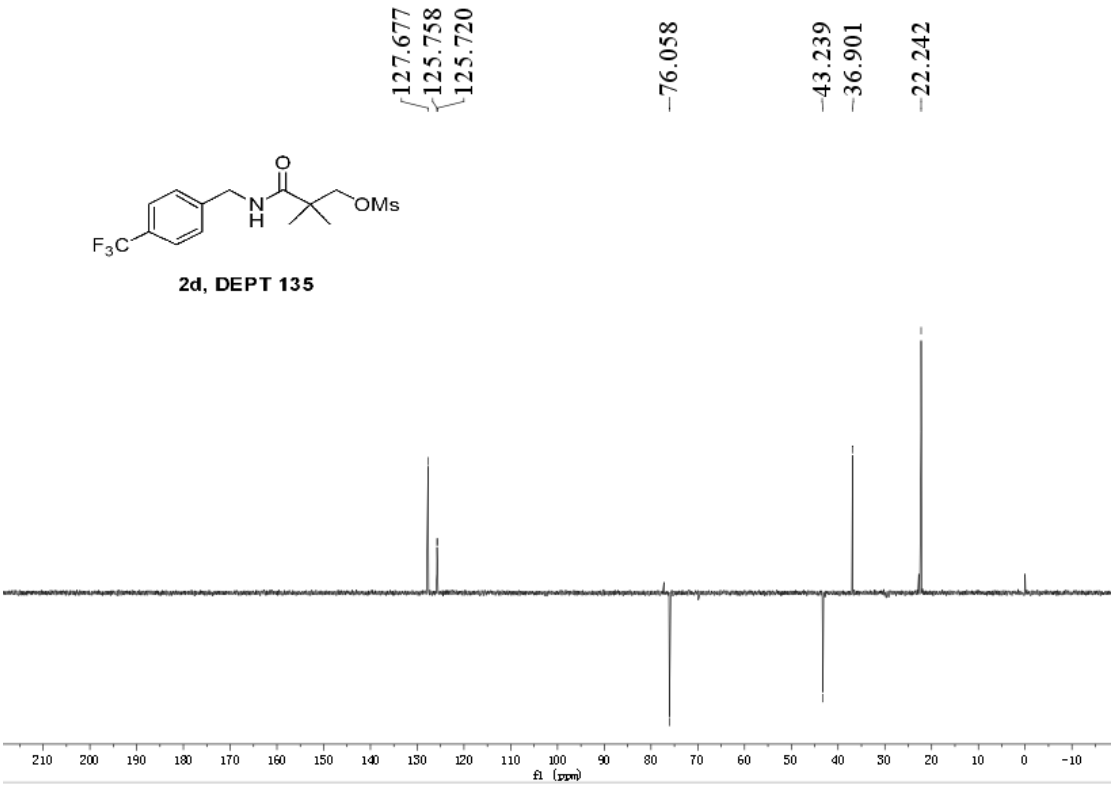
2c, ^{13}C NMR

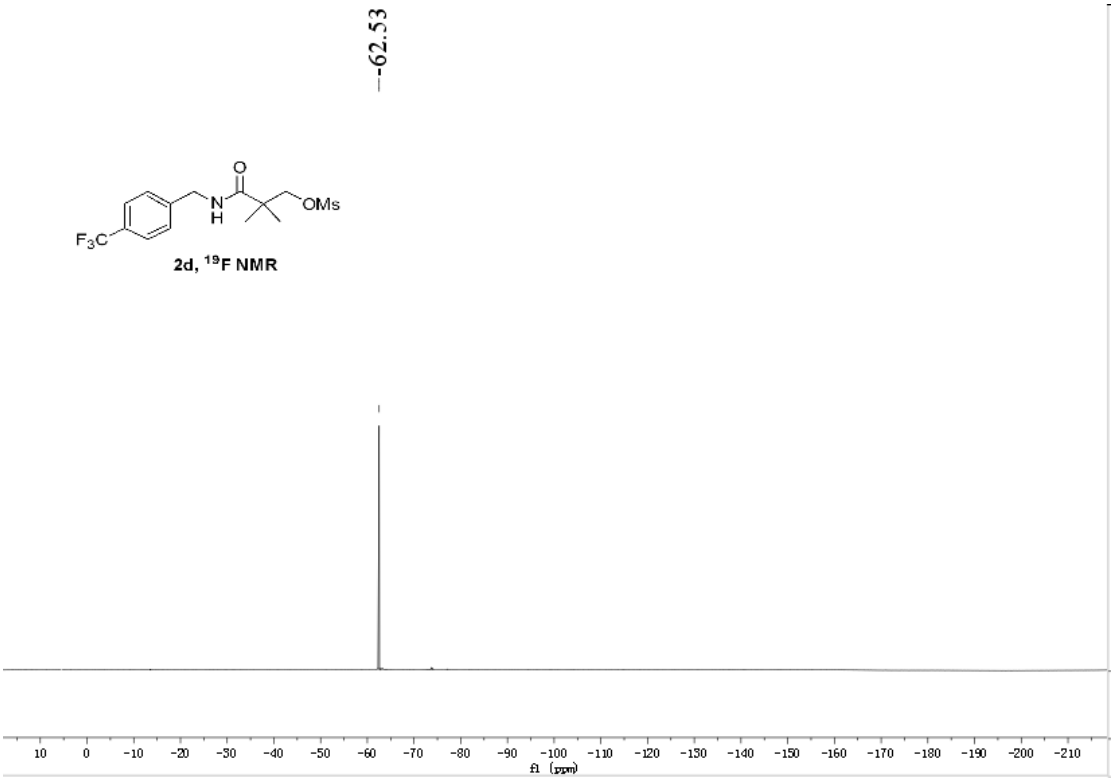

133.473
129.770
122.568
122.140
75.873
42.913
37.029
22.272
O2N
N
H
O
OMs
2c, DEPT 135
f1 (ppm)

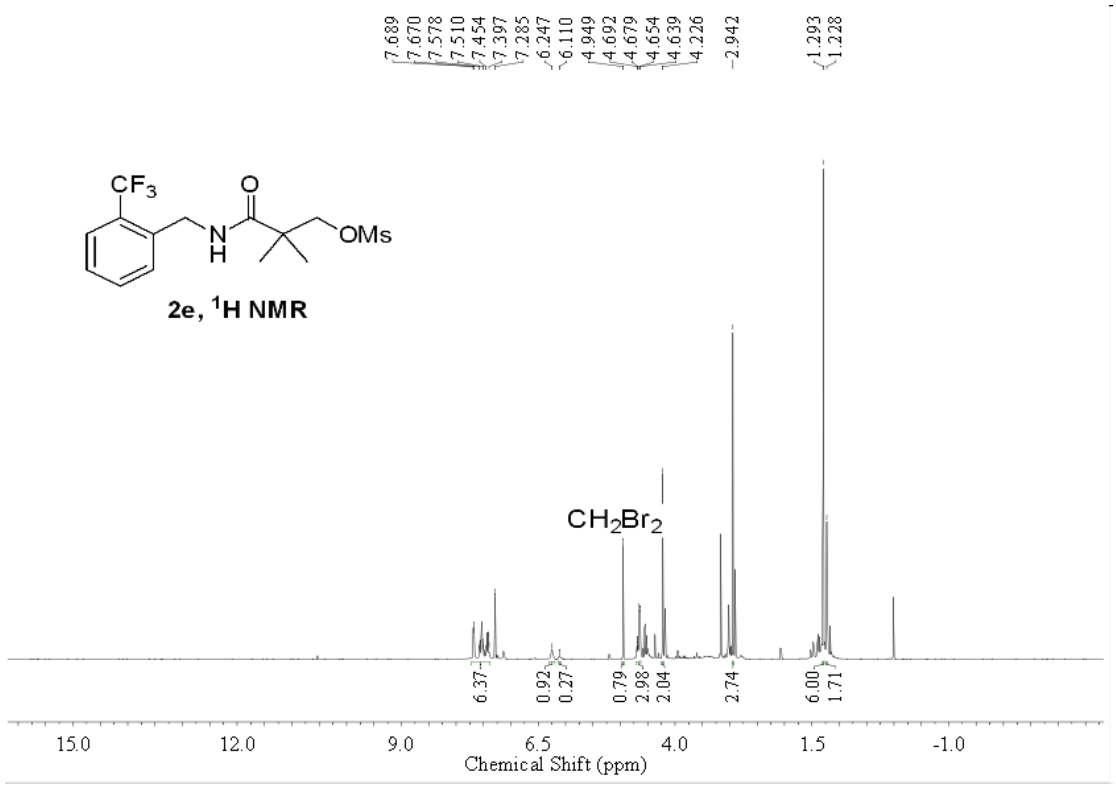

7.632
7.611
7.415
7.395
7.284
4.955
4.531
4.248
3.173
3.030
2.964
2.763
1.332
1.320
1.279
0.019
F3C
N
H
O
OMs
2d, 1H NMR
CH2Br2
2.68
2.53
0.61
3.66
1.97
3.00
7.27
2.61
f1 (ppm)

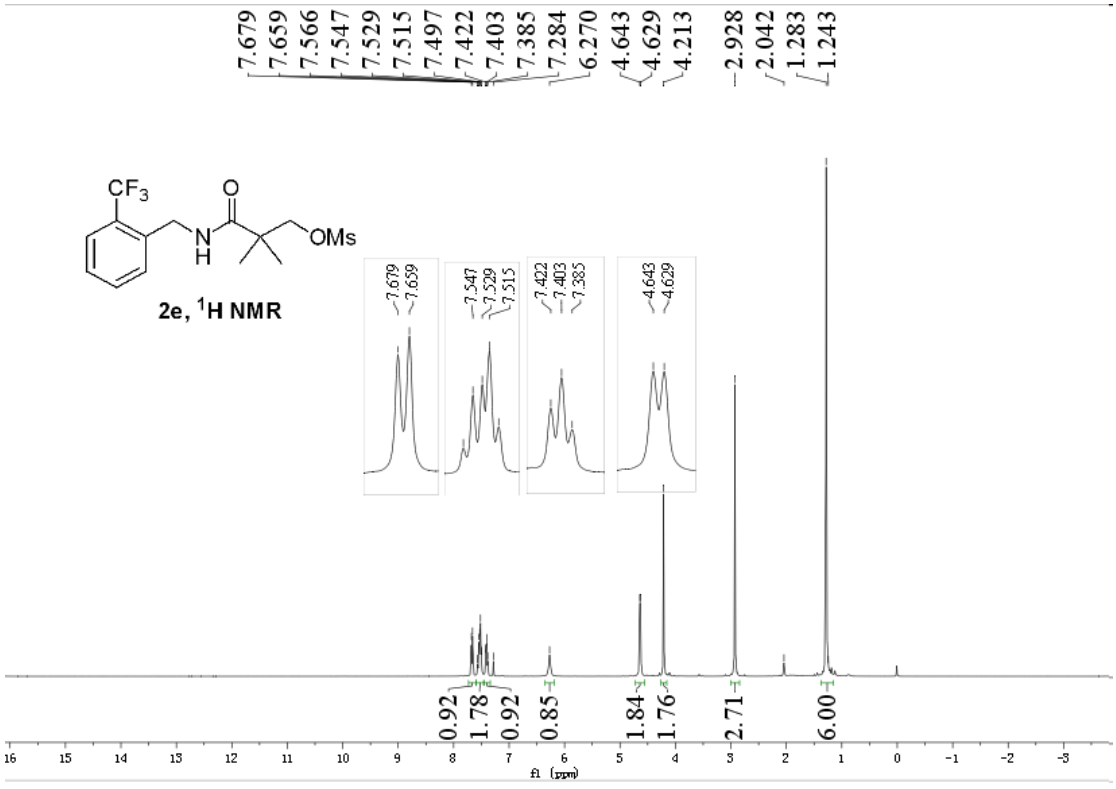

7.616
7.595
7.403
7.382
7.284
6.322
4.525
4.510
4.232
2.948
1.316
0.014
O
N
H
OMs
F3C
2d, 1H NMR
1.99
1.97
0.92
2.09
2.00
2.96
6.16
f1 (ppm)

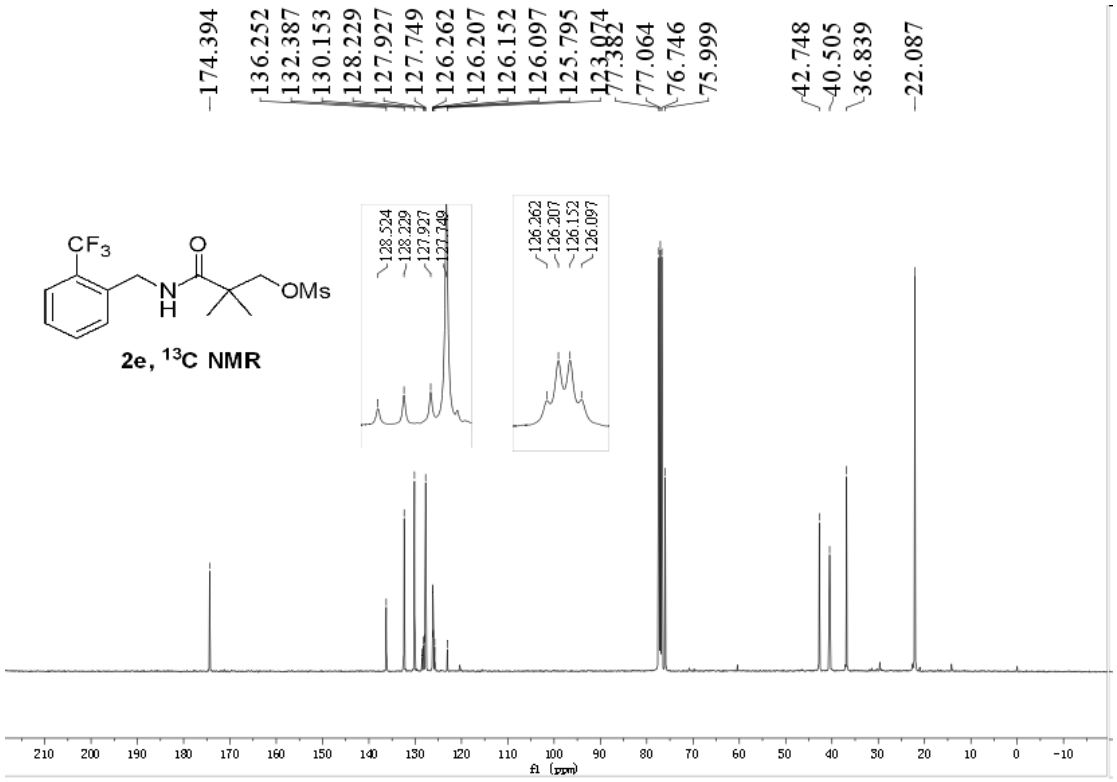

174.560
142.202
130.278
129.956
129.633
129.311
128.098
127.644
125.702
125.667
125.395
122.691
77.364
77.252
77.046
76.729
76.067
43.189
42.799
36.878
22.198
O
N
H
OMs
F3C
2d, 13C NMR
f1 (ppm)

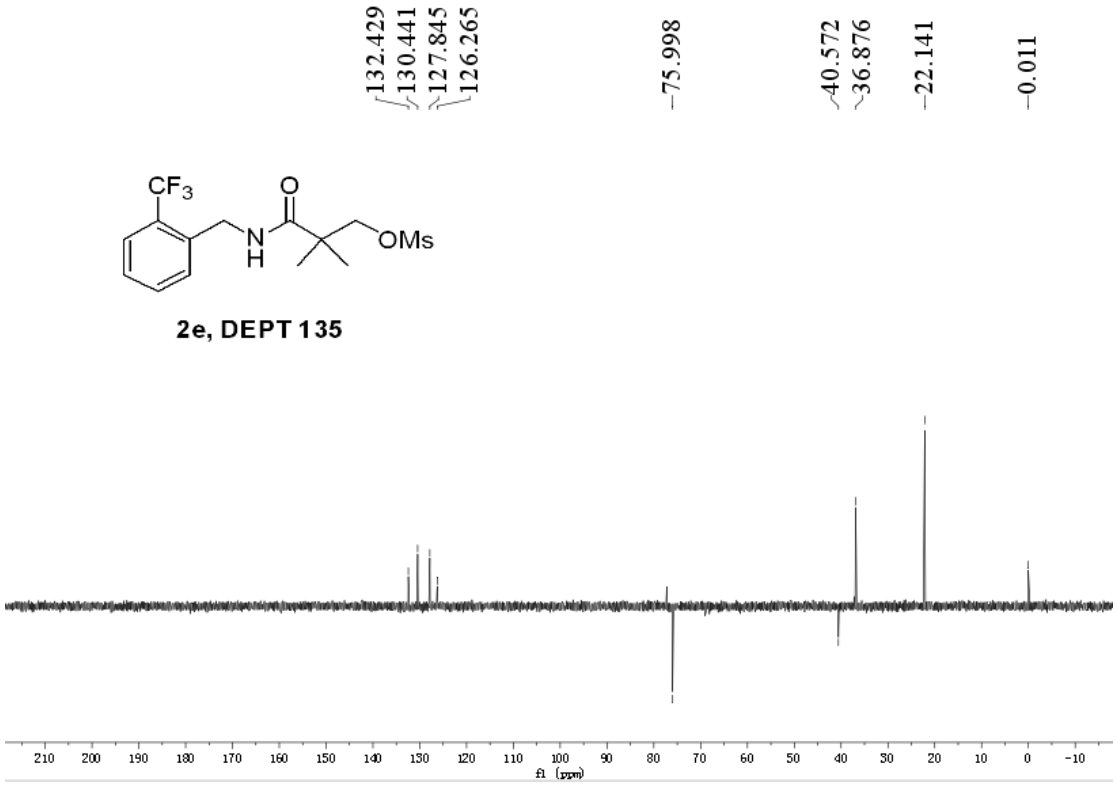

127.677
125.758
125.720
76.058
43.239
36.901
22.242
O
N
H
OMs
F3C
2d, DEPT 135
f1 (ppm)

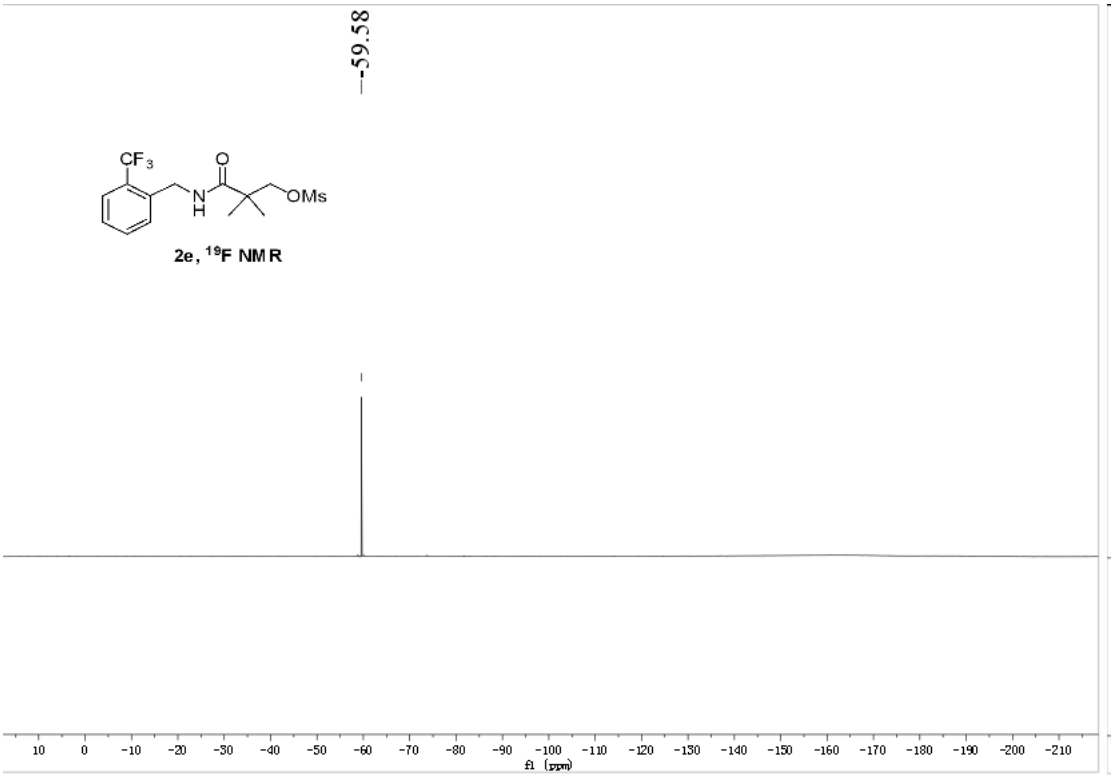

-62.53
O
N
H
OMs
F3C
2d, 19F NMR
f1 (ppm)

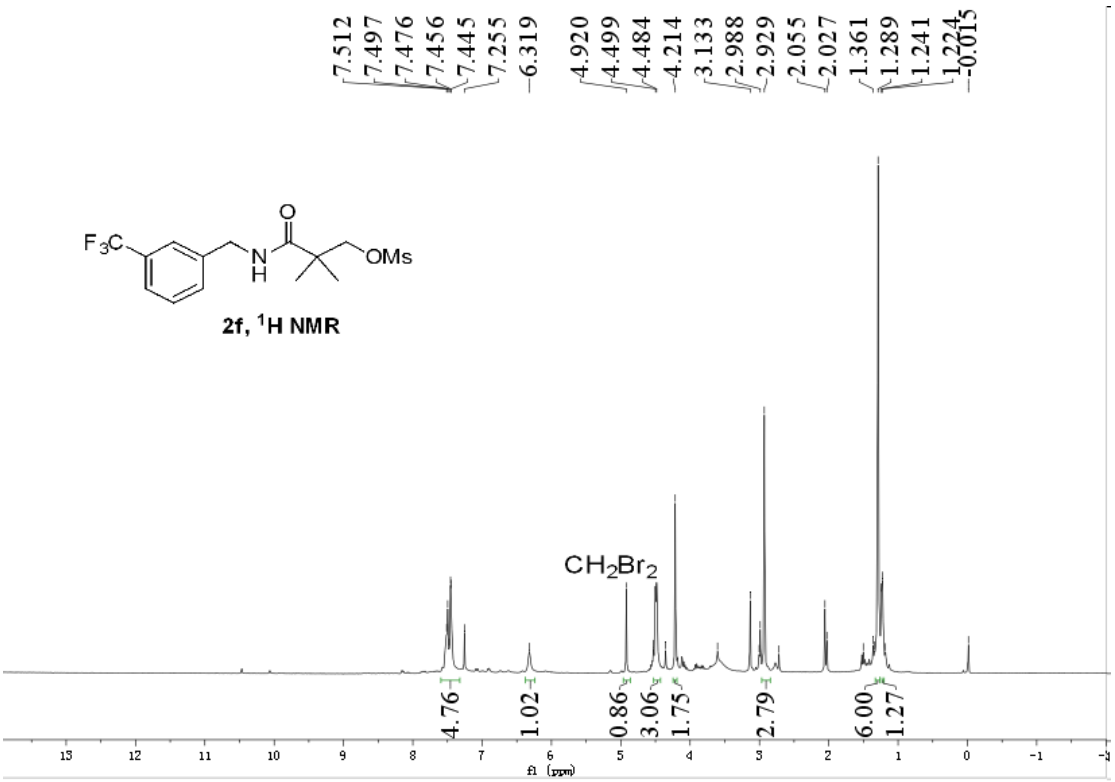

7.689
7.670
7.578
7.510
7.454
7.397
7.285
6.247
6.110
4.949
4.692
4.679
4.654
4.639
4.226
2.942
1.293
1.228
CF3
O
N
H
OMs
2e, 1H NMR
CH2Br2
6.37
0.92
0.27
0.79
2.98
2.04
2.74
6.00
1.71
15.0
12.0
9.0
6.5
4.0
1.5
-1.0
Chemical Shift (ppm)

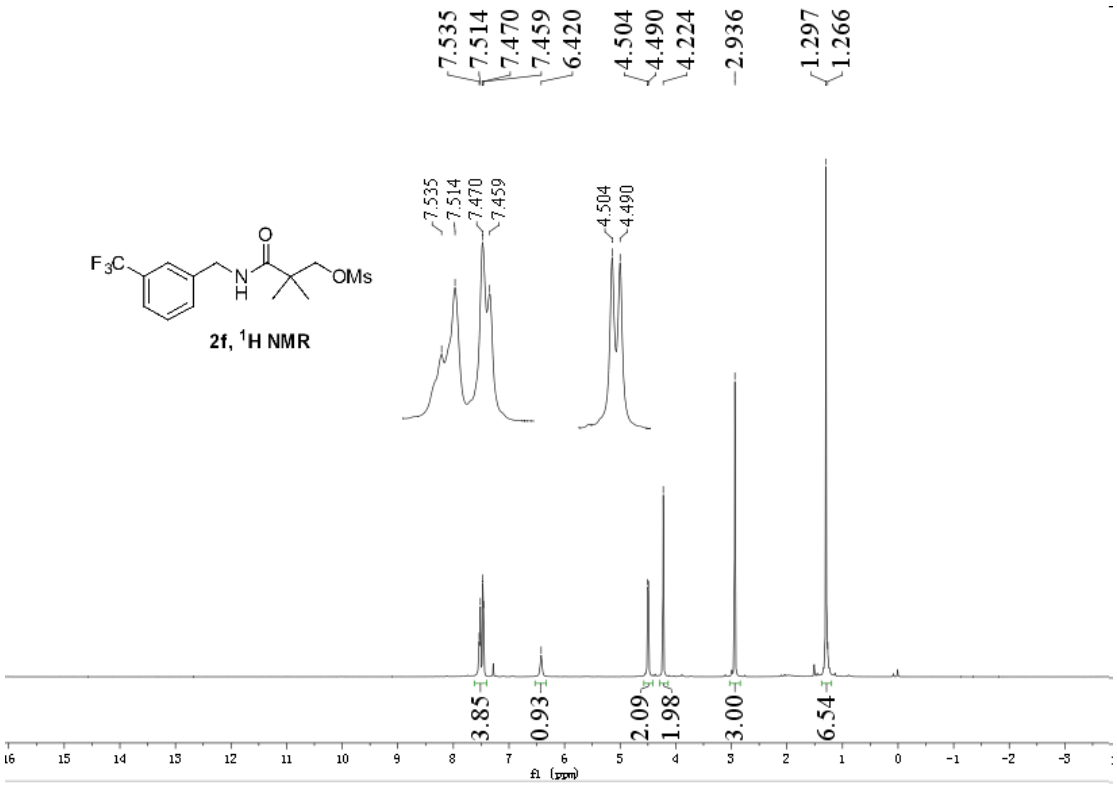

7.679
7.659
7.566
7.547
7.529
7.515
7.497
7.422
7.403
7.385
7.284
6.270
4.643
4.629
4.213
2.928
2.042
1.283
1.243
CF3
O
N
H
OMs
2e, 1H NMR
0.92
1.78
0.92
0.85
1.84
1.76
2.71
6.00
f1 (ppm)

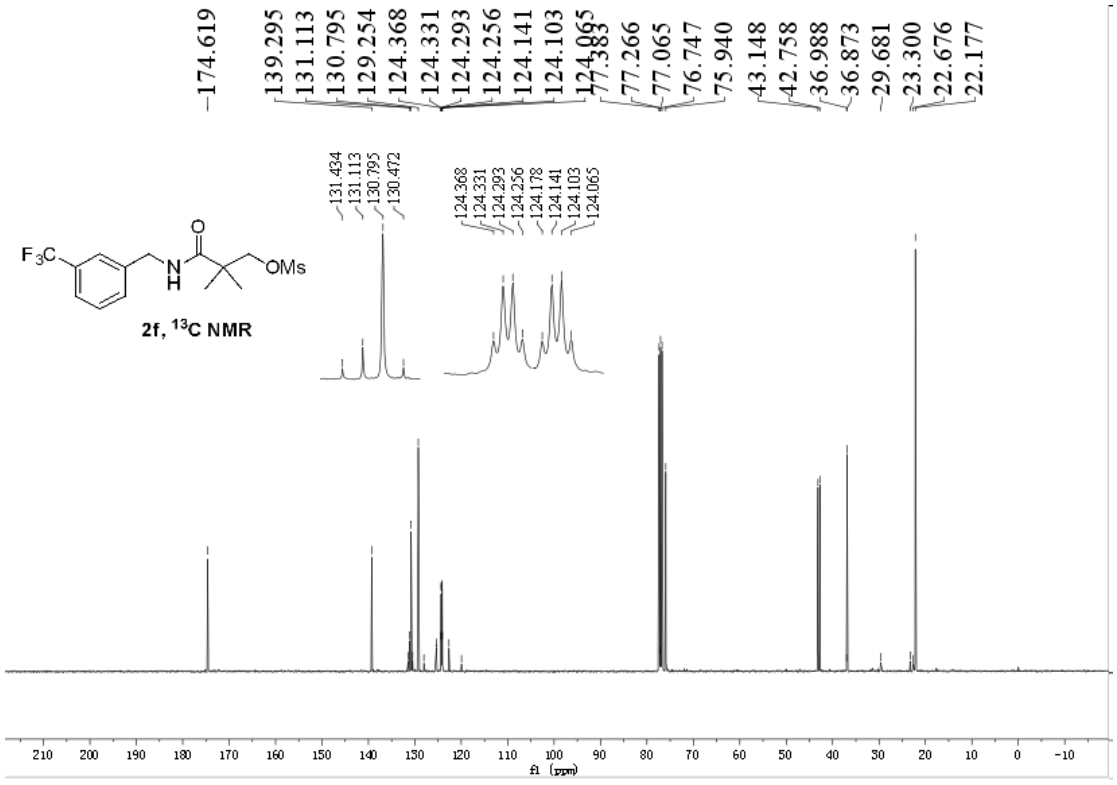

174.394
136.252
132.387
130.153
128.229
127.927
127.749
126.262
126.207
126.152
126.097
125.795
123.074
77.382
77.064
76.746
75.999
42.748
40.505
36.839
22.087
CF3
O
N
H
OMs
2e, 13C NMR
128.524
128.229
127.927
127.749
126.262
126.207
126.152
126.097
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

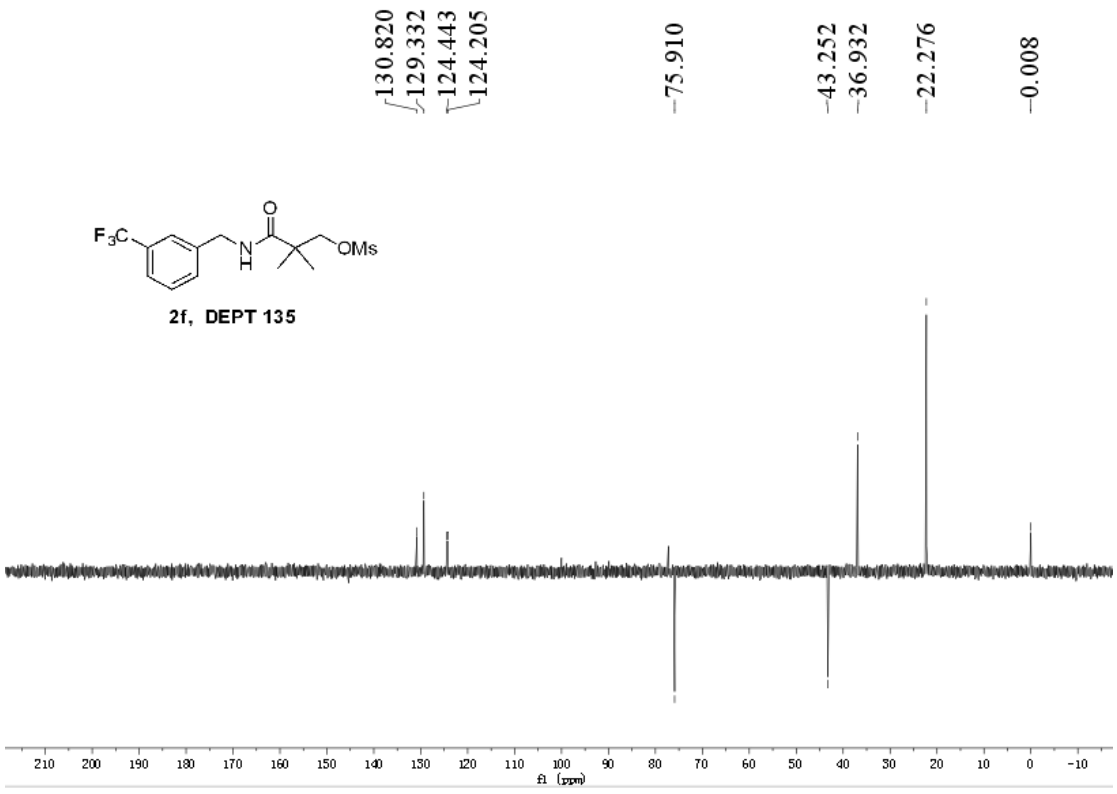

132.429
130.441
127.845
126.265
75.998
40.572
36.876
22.141
0.011
CF3
O
N
H
OMs
2e, DEPT 135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-59.58
CF3
O
N
H
OMs
2e, 19F NMR
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

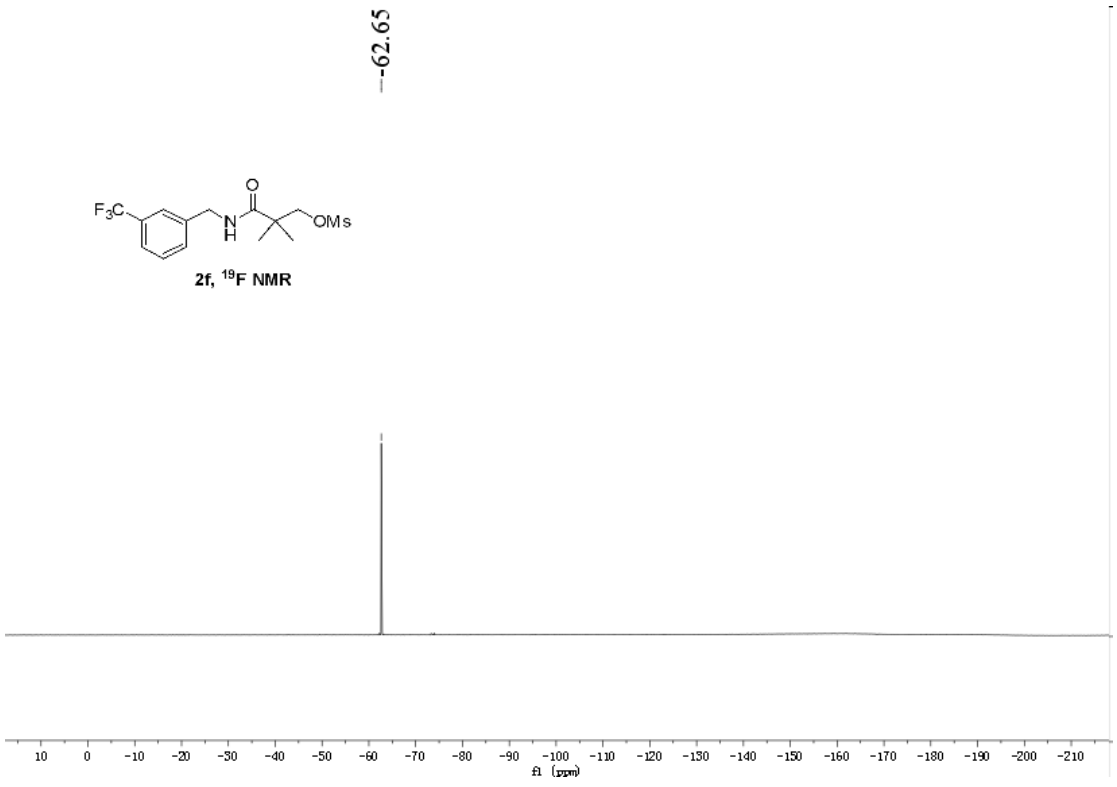

7.512
7.497
7.476
7.456
7.445
7.255
6.319
4.920
4.499
4.484
4.214
3.133
2.988
2.929
2.055
2.027
1.361
1.289
1.241
1.224
0.015
F3C
O
N
H
OMs
2f, 1H NMR
CH2Br2
4.76
1.02
0.86
3.06
1.75
2.79
6.00
1.27
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


7.535
7.514
7.470
7.459
6.420
4.504
4.490
4.224
2.936
1.297
1.266
F3C
O
N
H
OMs
2f, 1H NMR
3.85
0.93
2.09
1.98
3.00
6.54
f1 (ppm)

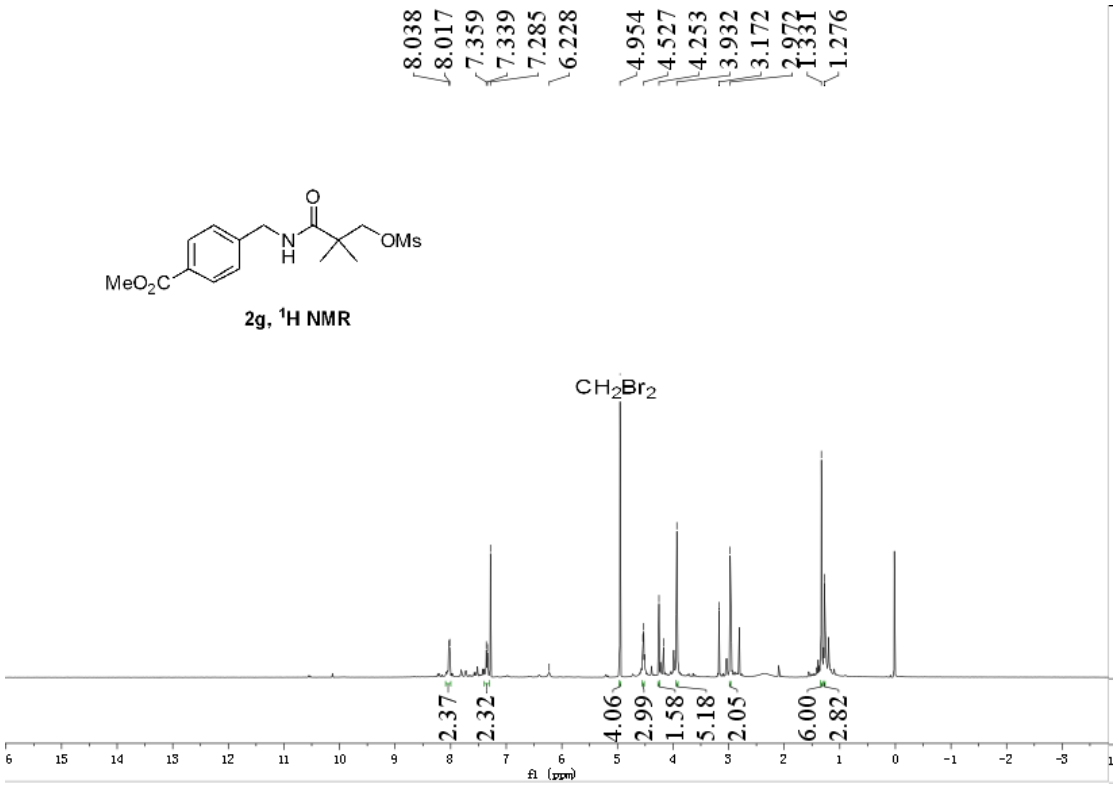

174.619
139.295
131.113
130.795
129.254
124.368
124.331
124.293
124.256
124.141
124.103
124.065
77.383
77.266
77.065
76.747
75.940
43.148
42.758
36.988
36.873
29.681
23.300
22.676
22.177
F3C
O
N
H
OMs
2f, 13C NMR
f1 (ppm)



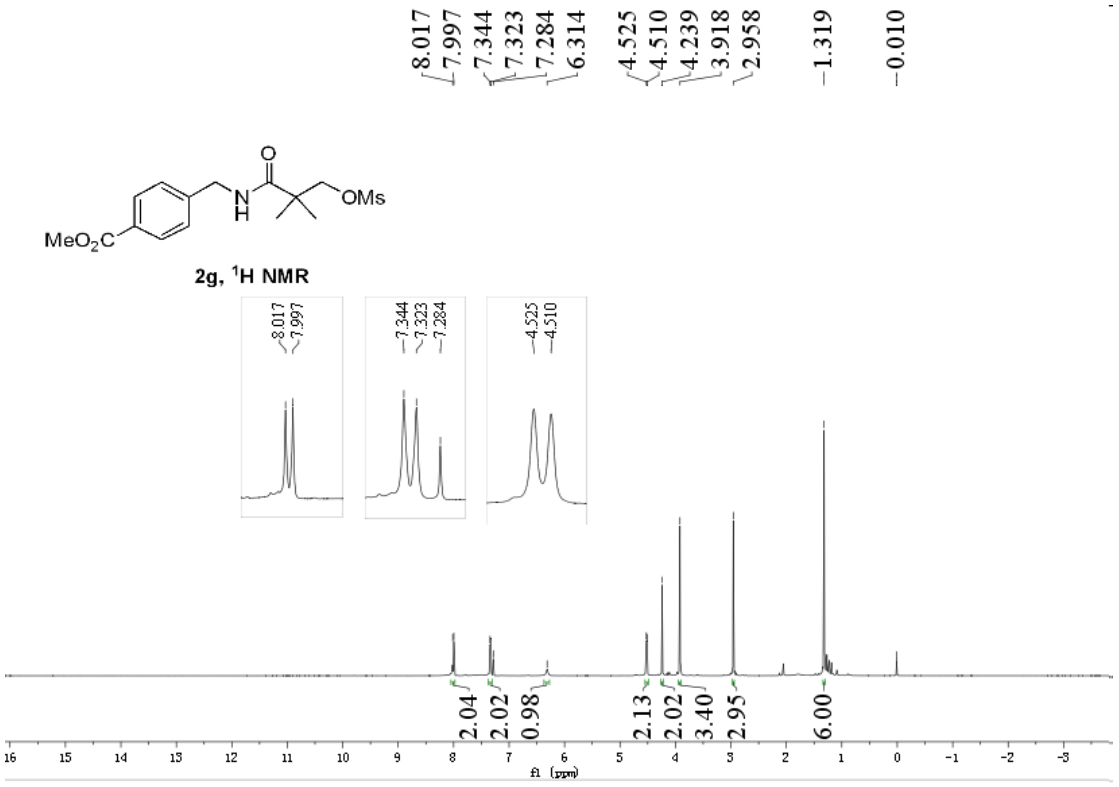
2f, DEPT 135



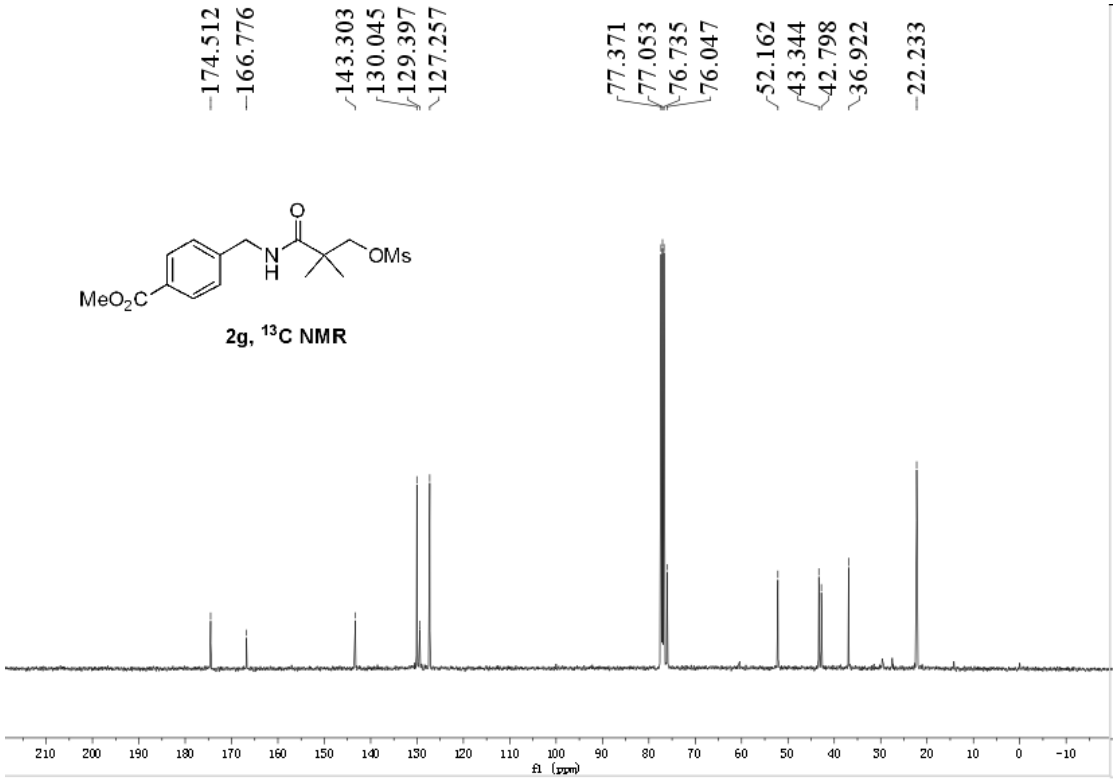
2f, ^{19}F NMR



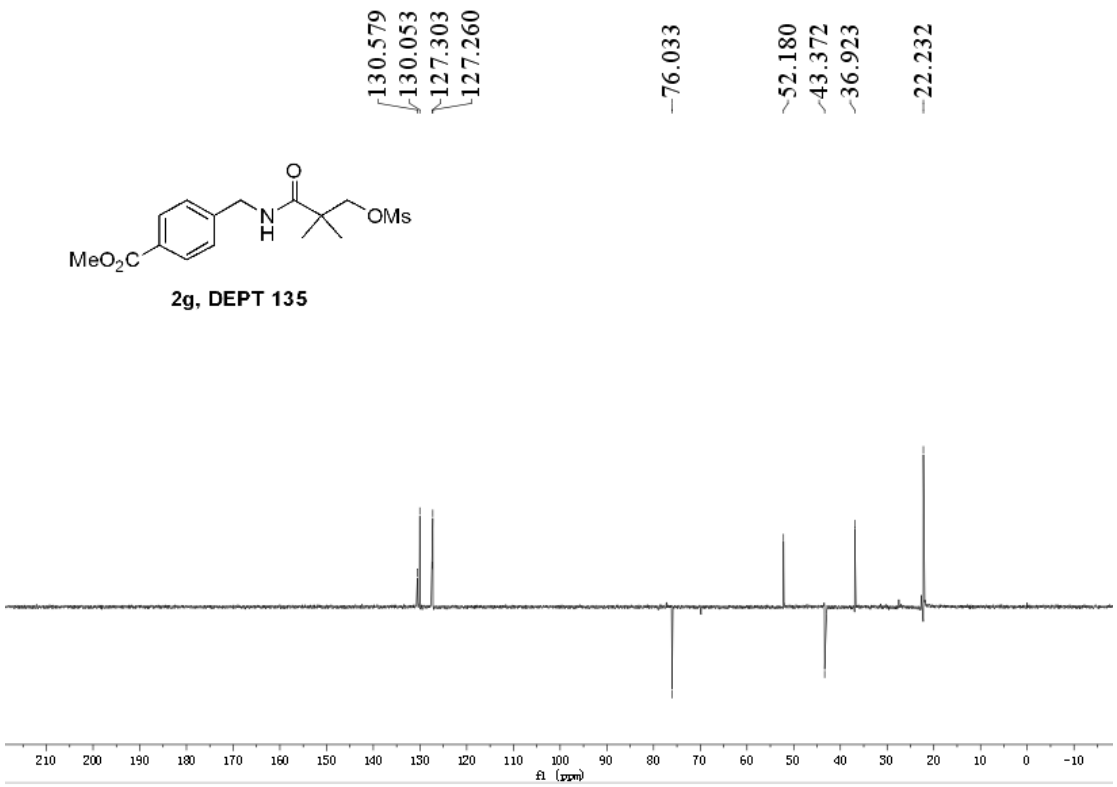
2g, ^{1}H NMR



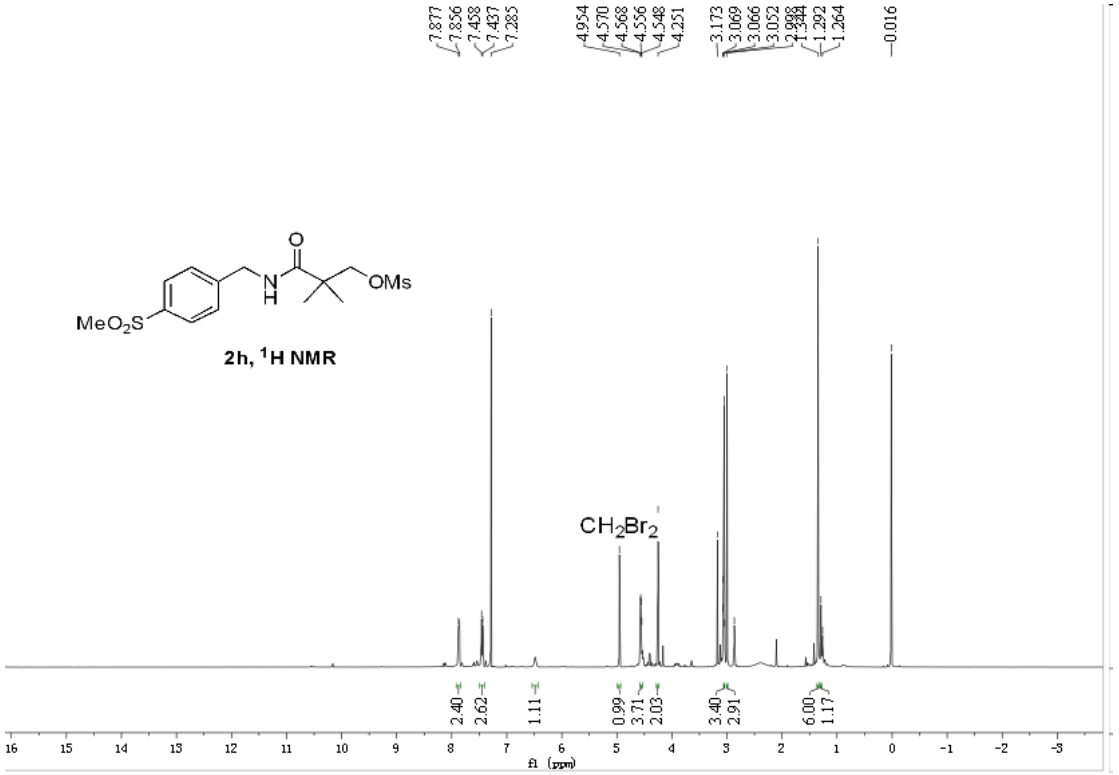
2g, ^{1}H NMR

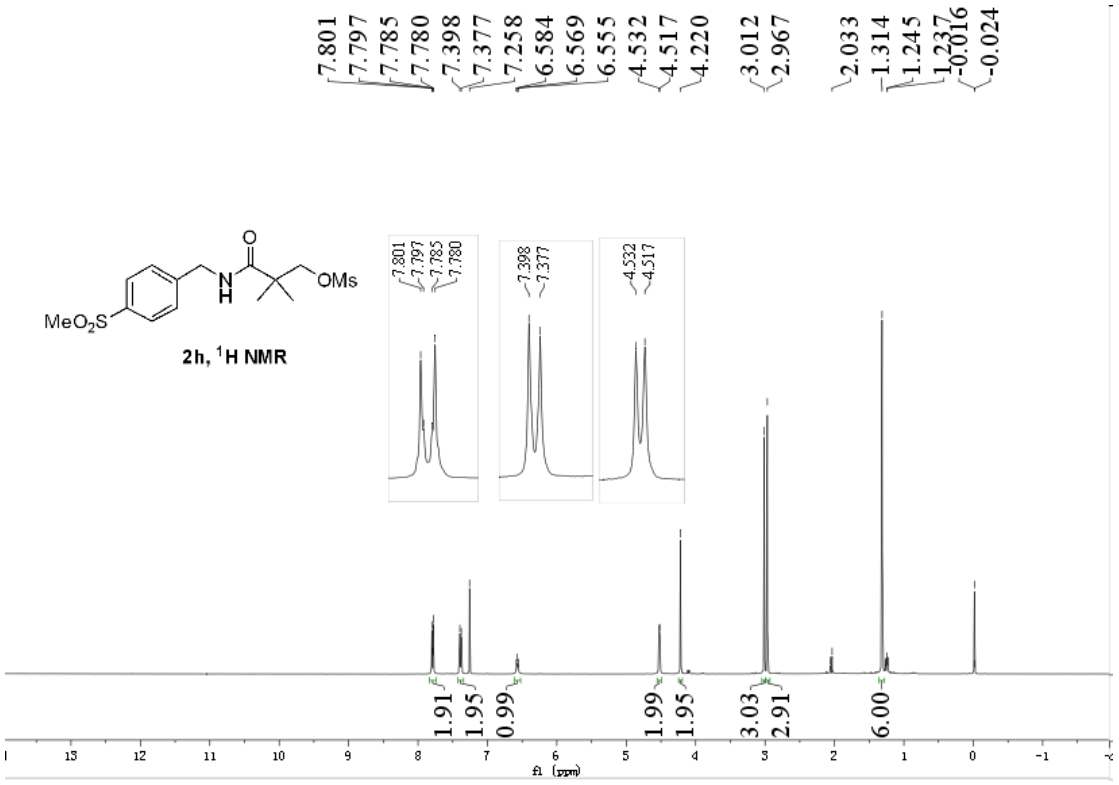


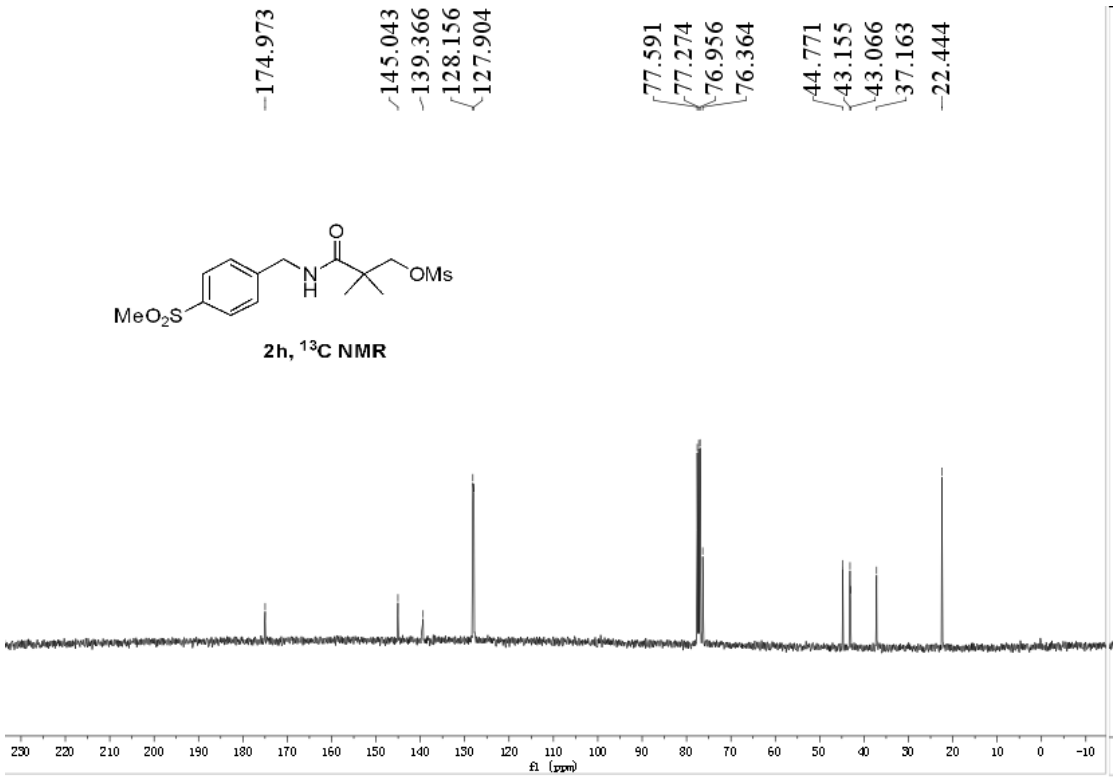
2g, ^{13}C NMR



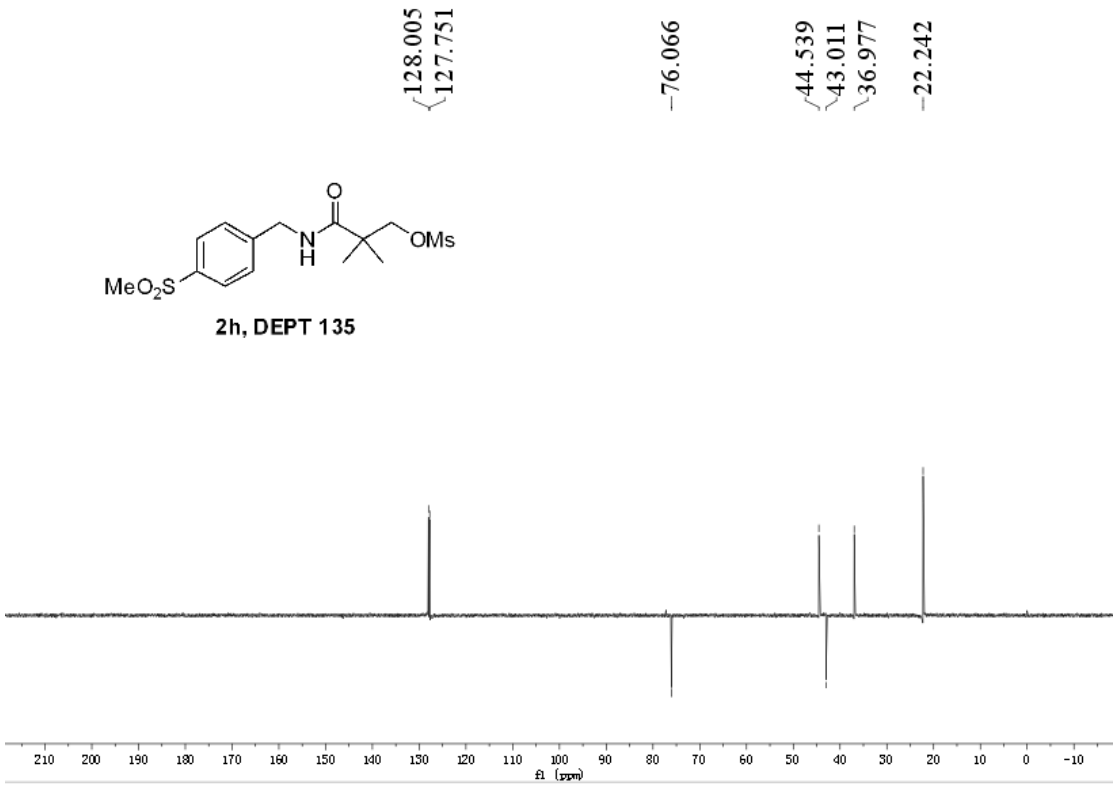
2g, DEPT 135


7.877
7.856
7.458
7.437
7.285
4.954
4.570
4.568
4.556
4.548
4.251
3.173
3.069
3.066
3.052
2.988
1.344
1.292
1.264
0.016
OMs
MeO2S
2h, 1H NMR
CH2Br2
2.40
2.62
1.11
0.99
3.71
2.03
3.40
2.91
6.00
1.17
f1 (ppm)

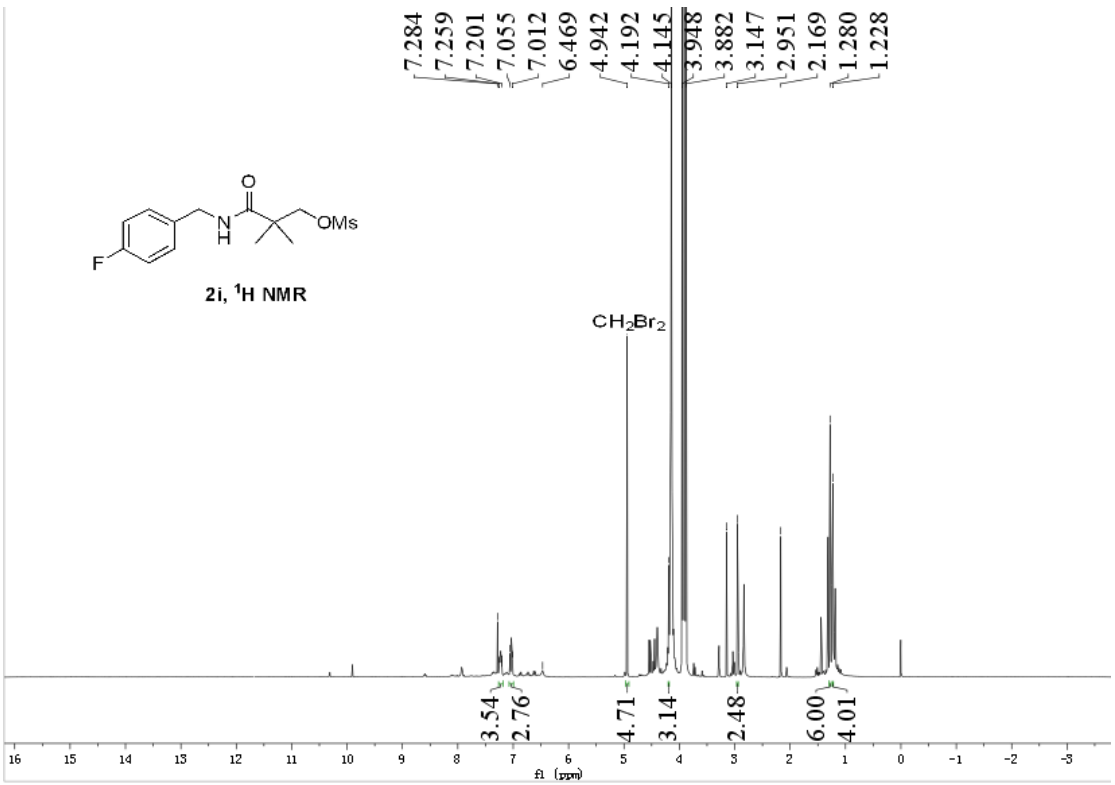

7.801
7.797
7.785
7.780
7.398
7.377
7.258
6.584
6.569
6.555
4.532
4.517
4.220
3.012
2.967
2.033
1.314
1.245
1.237
-0.016
-0.024
OMs
MeO2S
2h, 1H NMR
1.91
1.95
0.99
1.99
1.95
3.03
2.91
6.00
f1 (ppm)

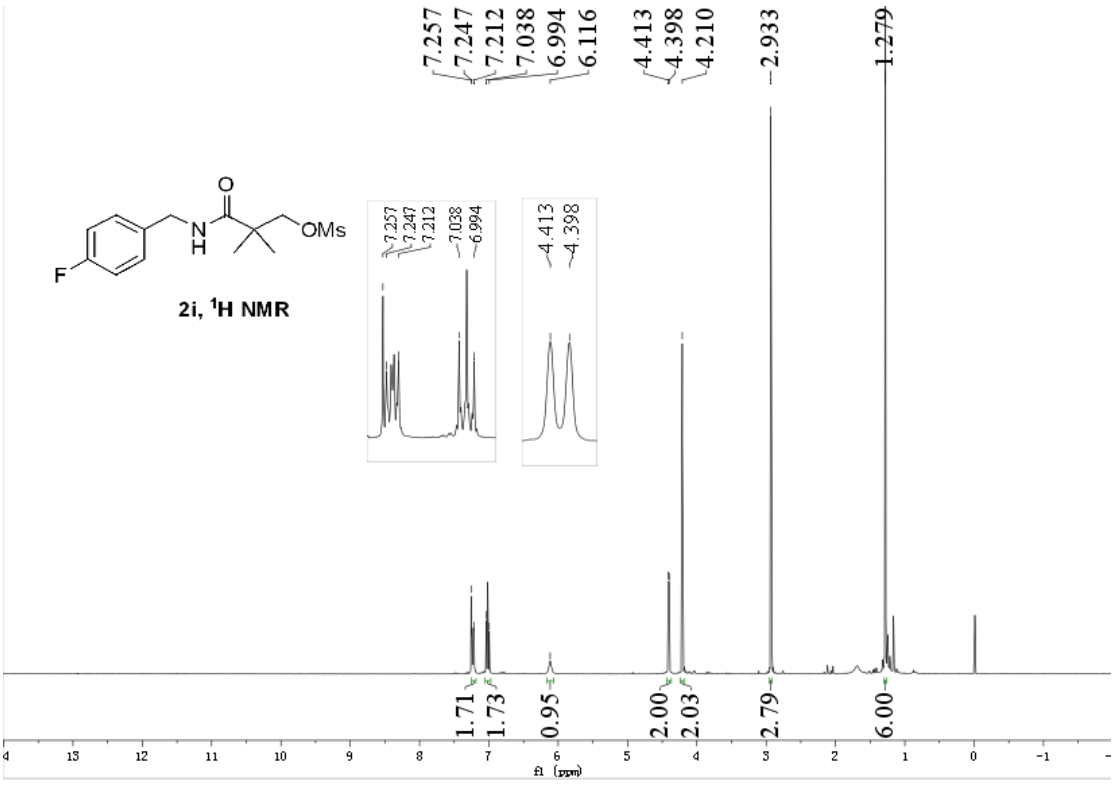


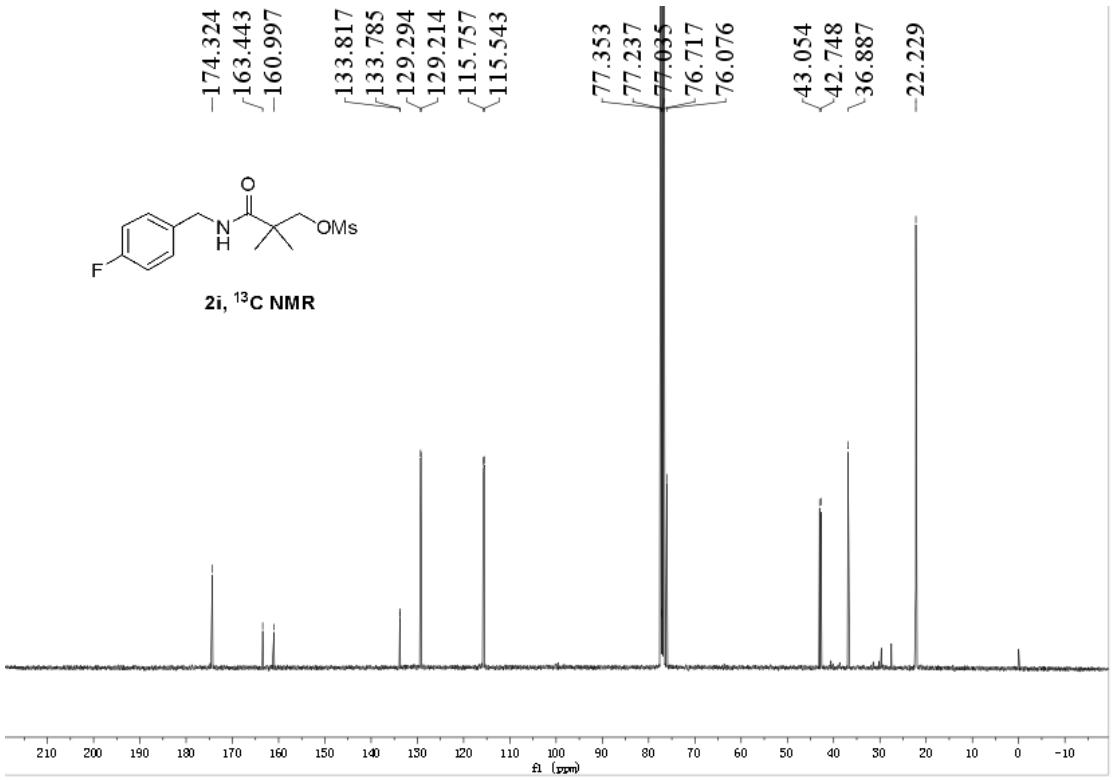
2h, ^{13}C NMR



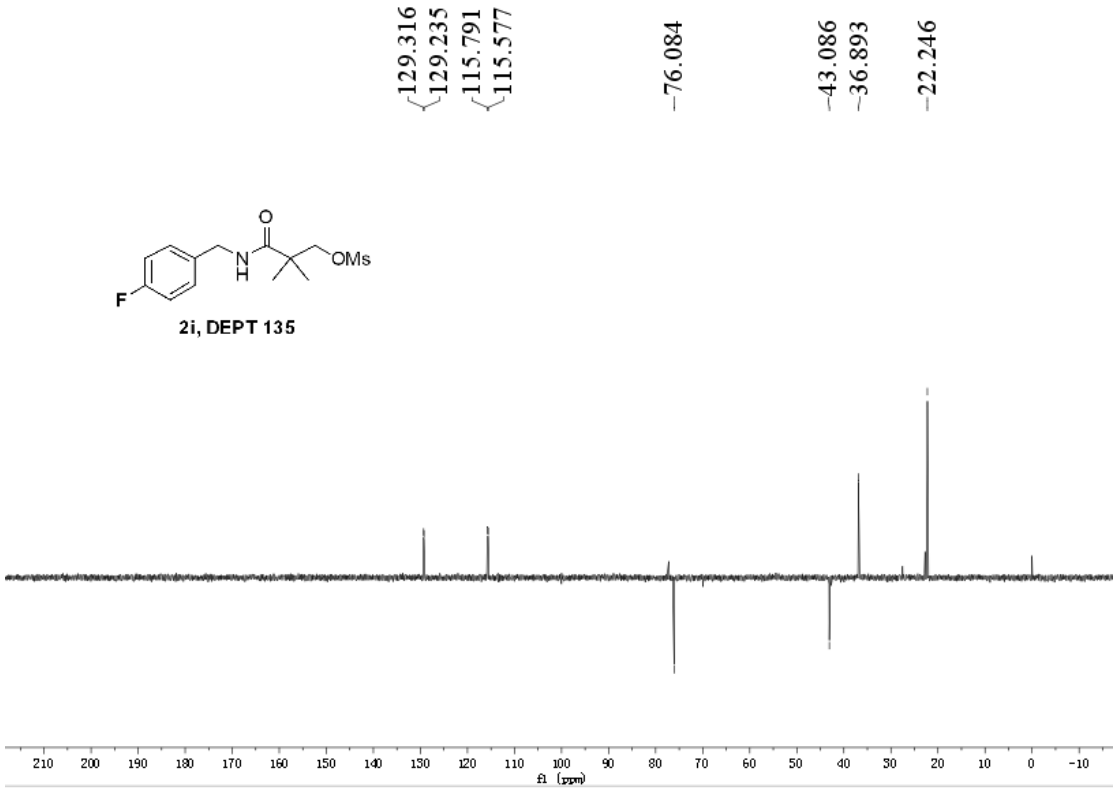
2h, DEPT 135


7.284
7.259
7.201
7.055
7.012
6.469
4.942
4.192
4.145
3.948
3.882
3.147
2.951
2.169
1.280
1.228
O
N
H
OMs
F
2i, ^{1}H NMR
CH_2Br_2
3.54
2.76
4.71
3.14
2.48
6.00
4.01
f1 (ppm)

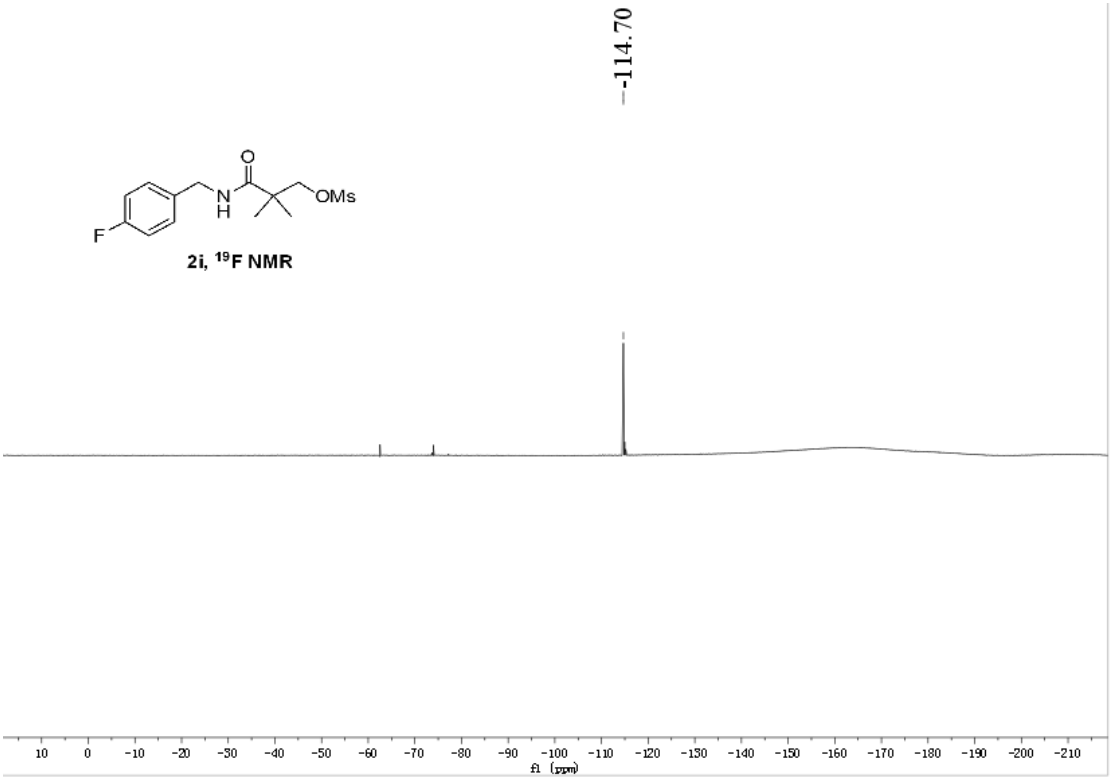

7.257
7.247
7.212
7.038
6.994
6.116
4.413
4.398
4.210
2.933
1.279
O
N
H
OMs
F
2i, ^{1}H NMR
1.71
1.73
0.95
2.00
2.03
2.79
6.00
f1 (ppm)



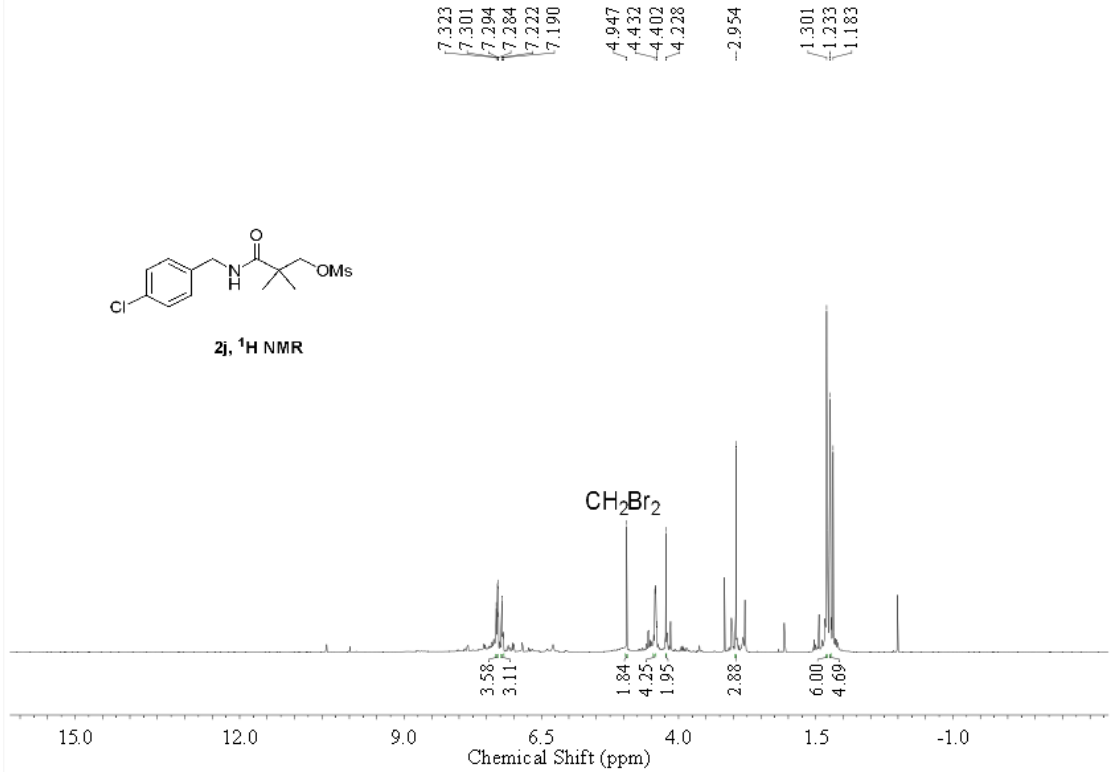
2i, ^{13}C NMR



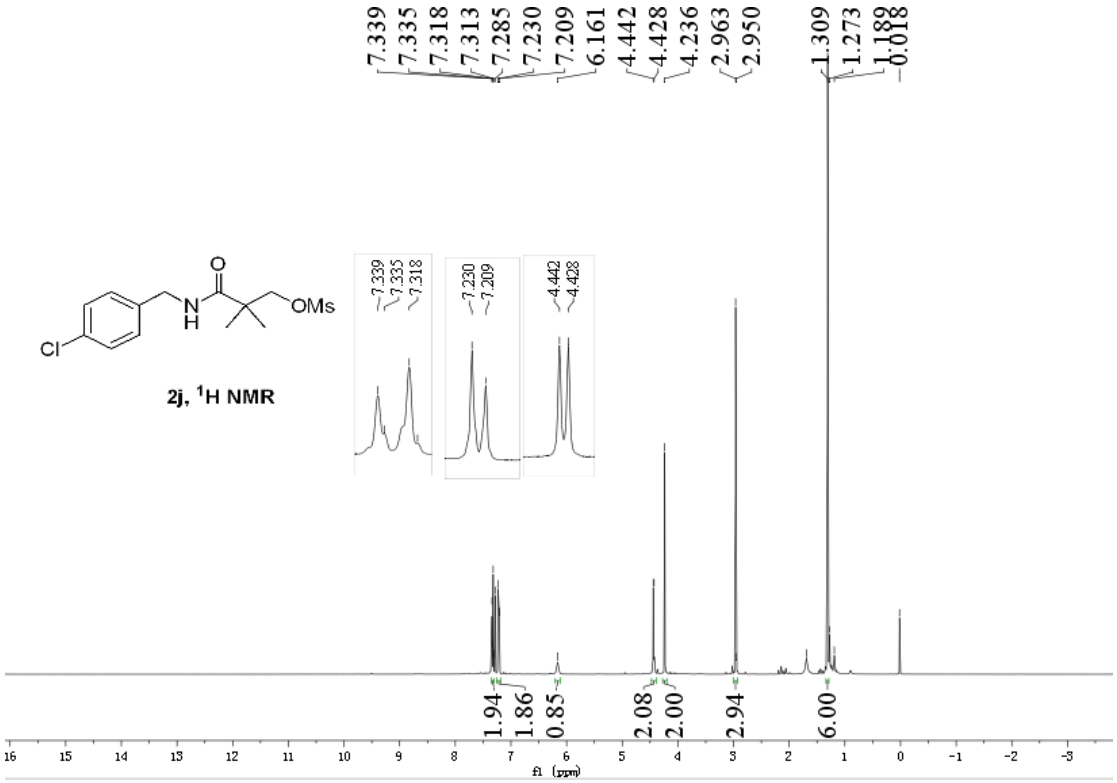
2i, DEPT 135

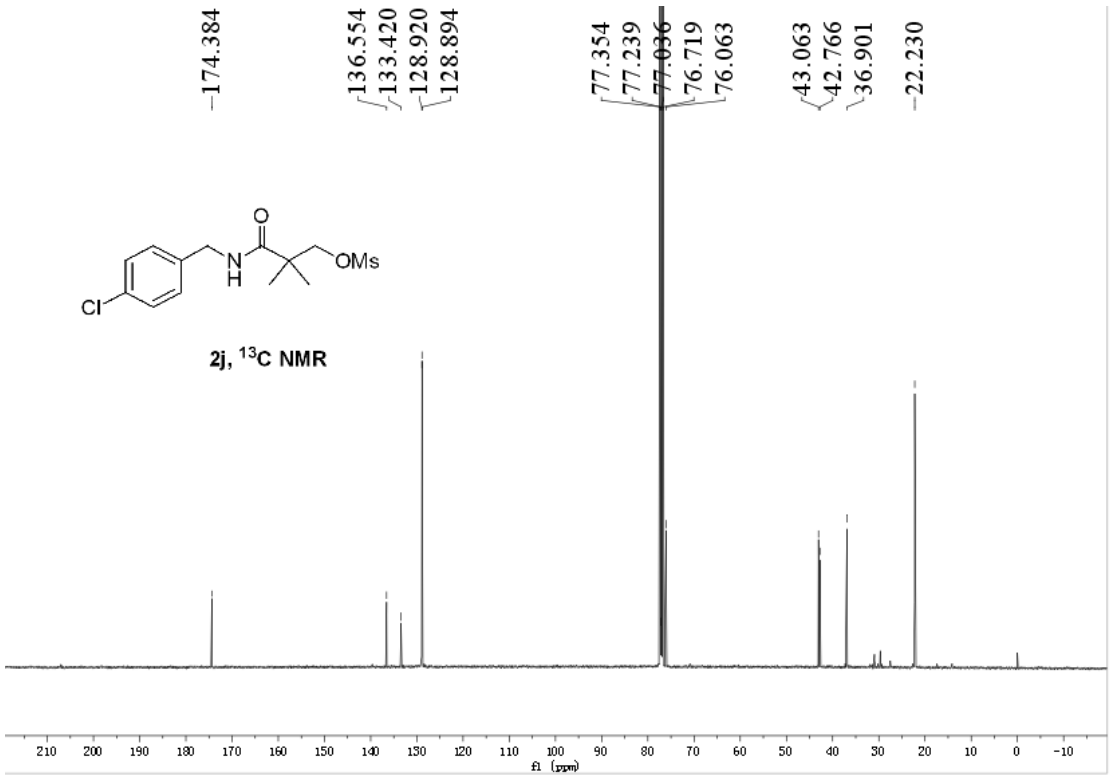


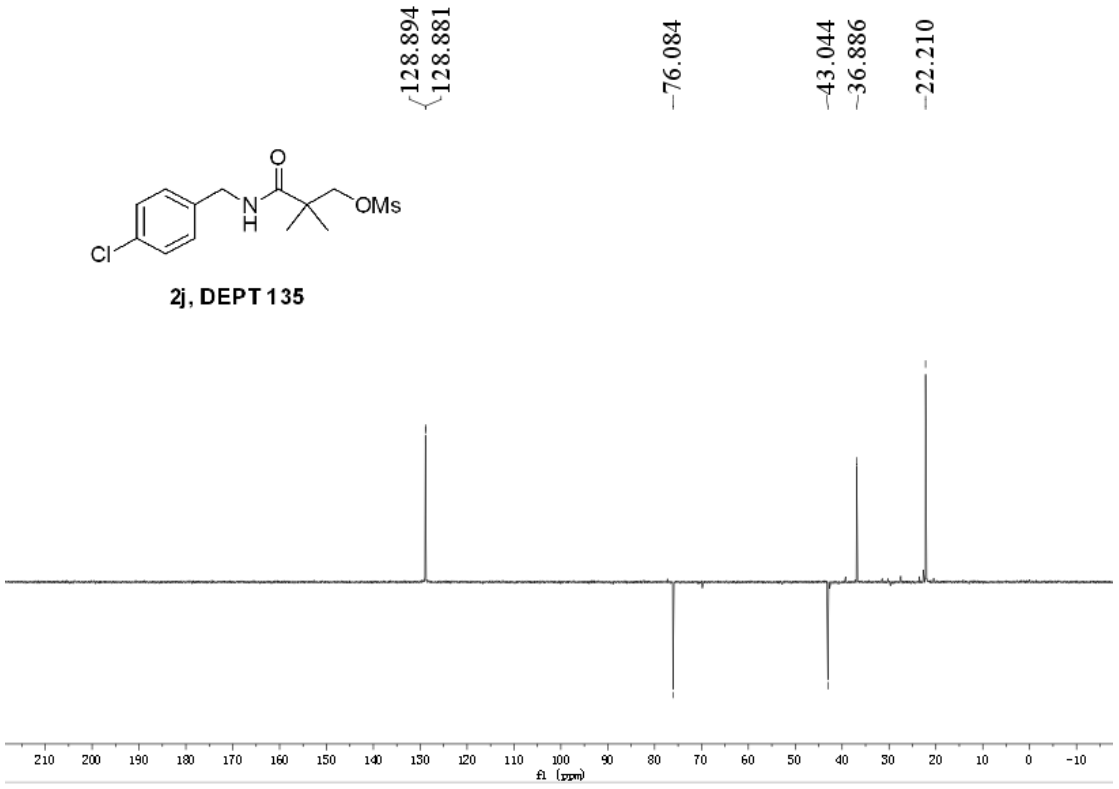
2i, ^{19}F NMR

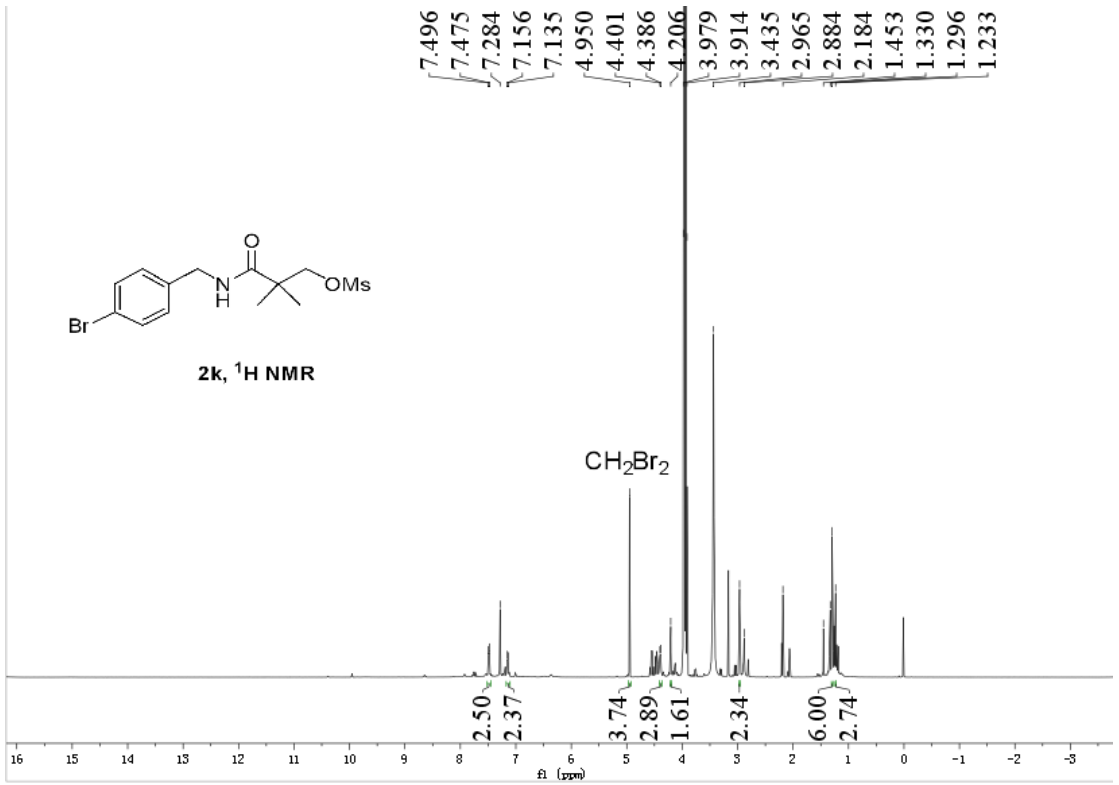


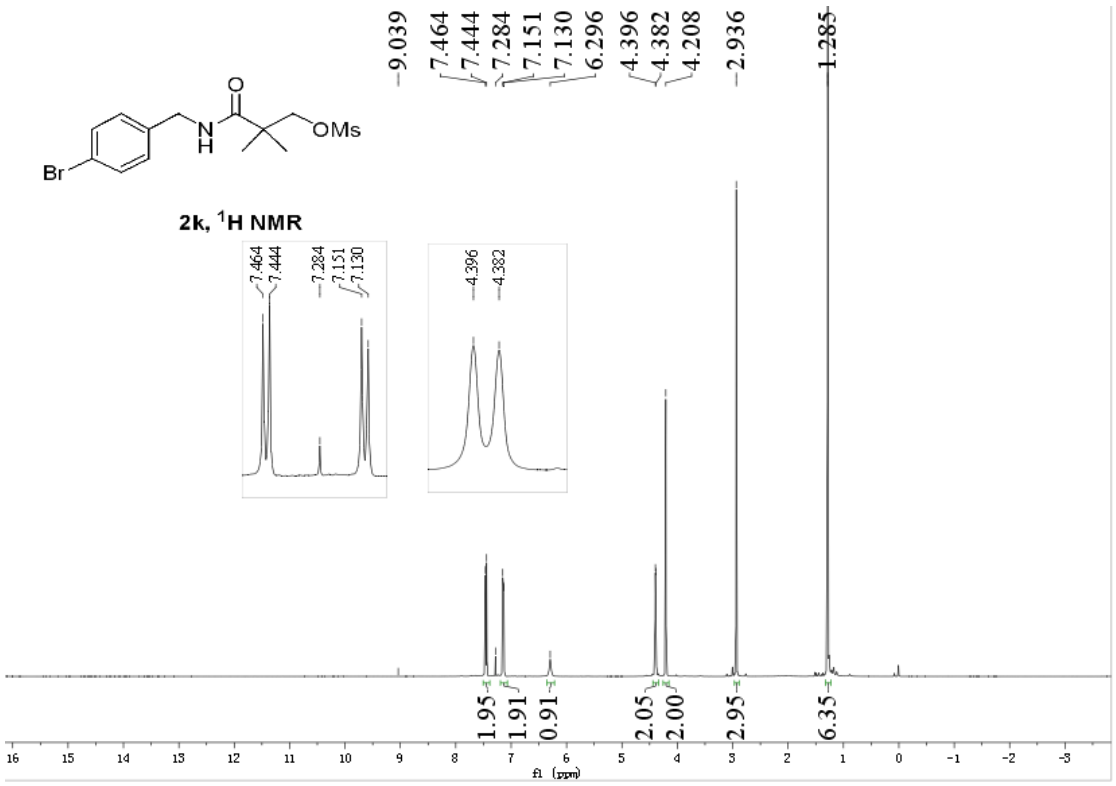
2j, ^{1}H NMR

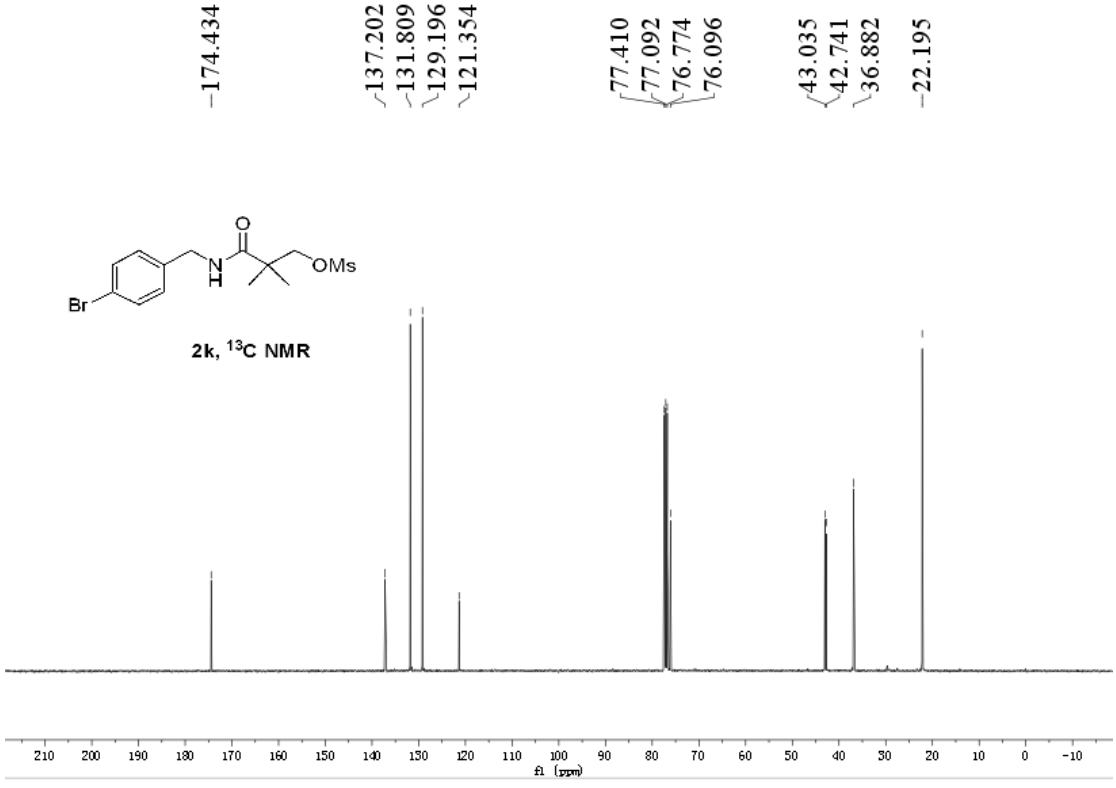

7.339
7.335
7.318
7.313
7.285
7.230
7.209
6.161
4.442
4.428
4.236
2.963
2.950
1.309
1.273
1.189
0.018
OMs
Cl
2j, 1H NMR
1.94
1.86
0.85
2.08
2.00
2.94
6.00
f1 (ppm)

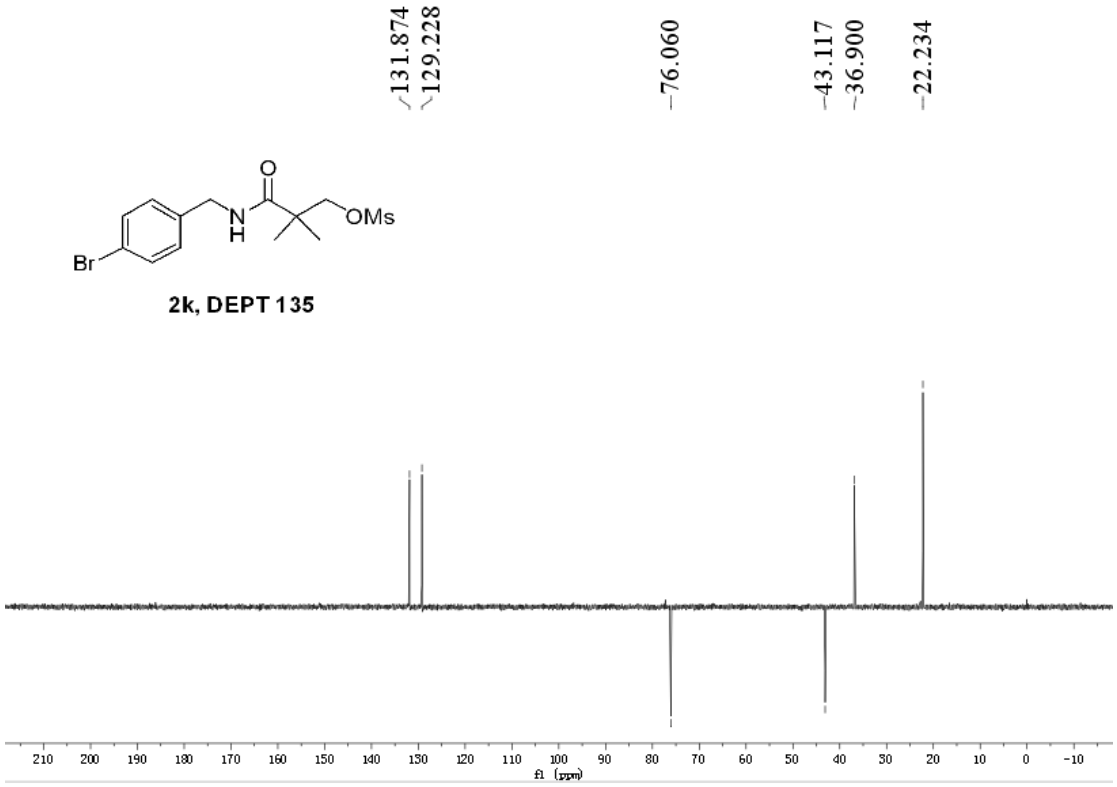

174.384
136.554
133.420
128.920
128.894
77.354
77.239
77.036
76.719
76.063
43.063
42.766
36.901
22.230
OMs
Cl
2j, 13C NMR
f1 (ppm)


128.894
128.881
76.084
43.044
36.886
22.210
Cl
N
H
O
OMs
2j, DEPT 135
f1 (ppm)


7.496
7.475
7.284
7.156
7.135
4.950
4.401
4.386
4.206
3.979
3.914
3.435
2.965
2.884
2.184
1.453
1.330
1.296
1.233
Br
N
H
O
OMs
2k, 1H NMR
CH2Br2
2.50
2.37
3.74
2.89
1.61
2.34
6.00
2.74
f1 (ppm)


9.039
7.464
7.444
7.284
7.151
7.130
6.296
4.396
4.382
4.208
2.936
1.285
Br
OMs
2k, ^{1}H NMR
1.95
1.91
0.91
2.05
2.00
2.95
6.35
f1 (ppm)


174.434
137.202
131.809
129.196
121.354
77.410
77.092
76.774
76.096
43.035
42.741
36.882
22.195
Br
OMs
2k, ^{13}C NMR
f1 (ppm)


131.874
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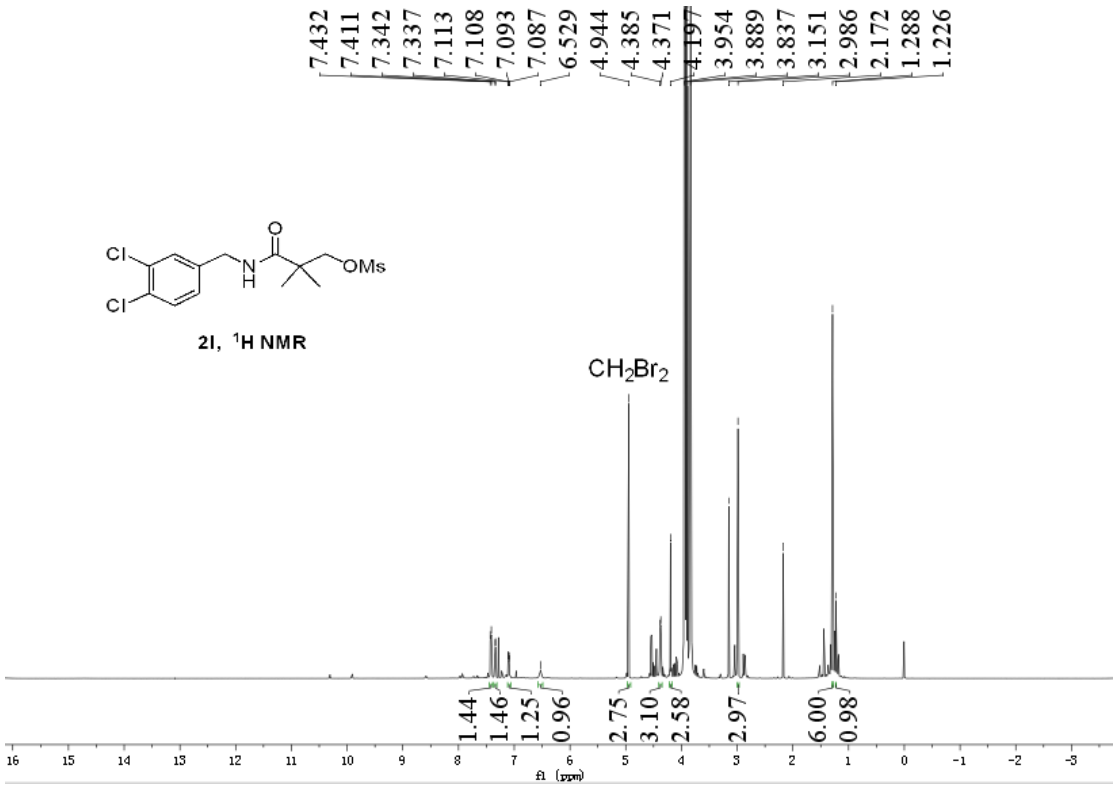
76.060
43.117
36.900
22.234
Br
O
N
H
OMs
2k, DEPT 135
f1 (ppm)


7.432
7.411
7.342
7.337
7.113
7.108
7.093
7.087
6.529
4.944
4.385
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4.197
3.954
3.889
3.837
3.151
2.986
2.172
1.288
1.226
Cl
Cl
O
N
H
OMs
2l, 1H NMR
CH2Br2
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1.25
0.96
2.75
3.10
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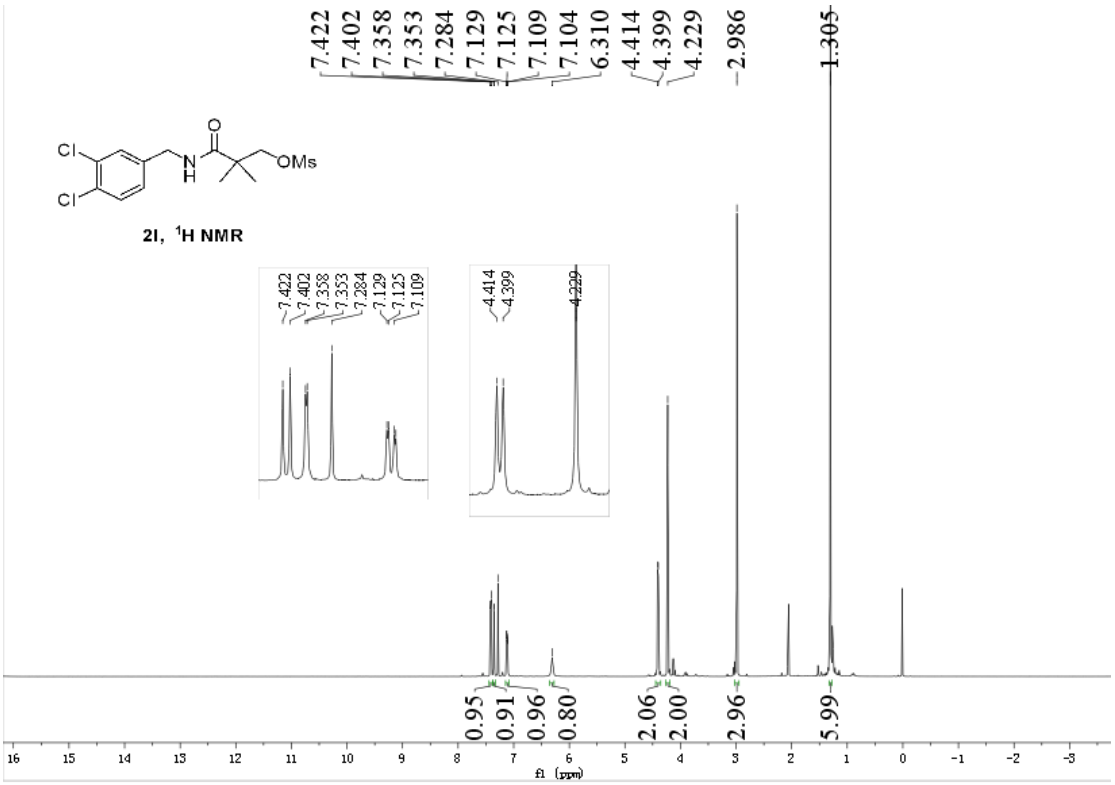
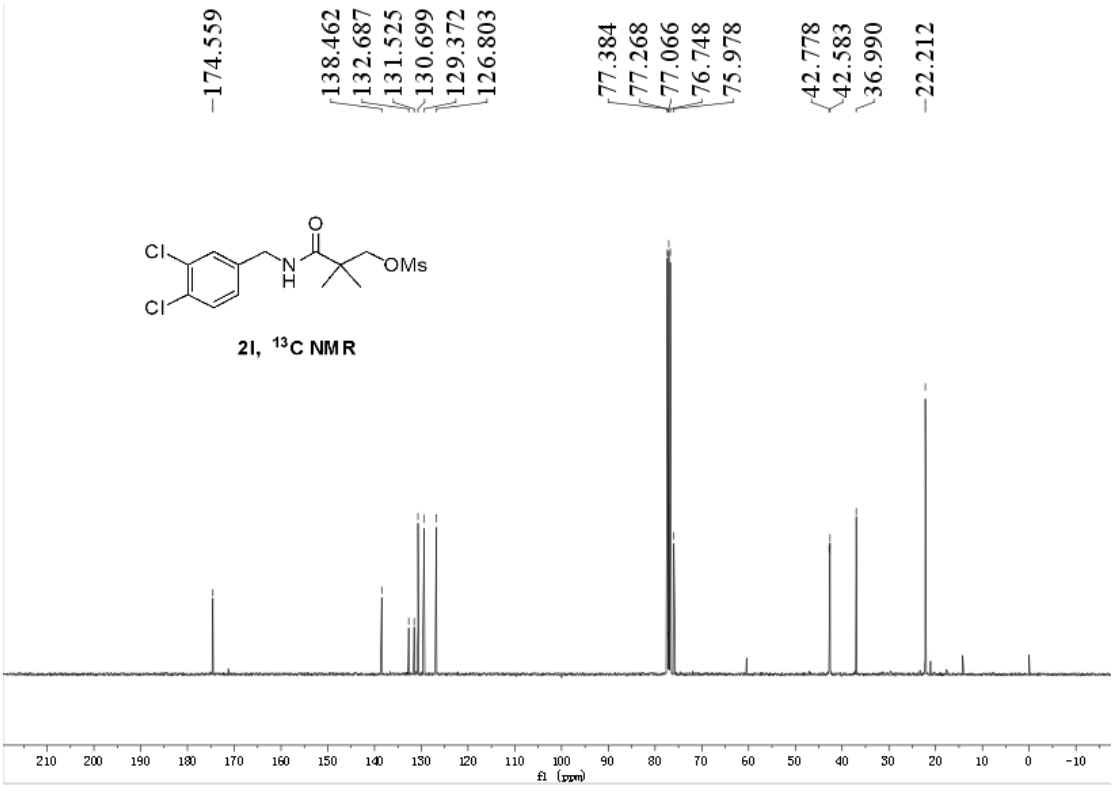
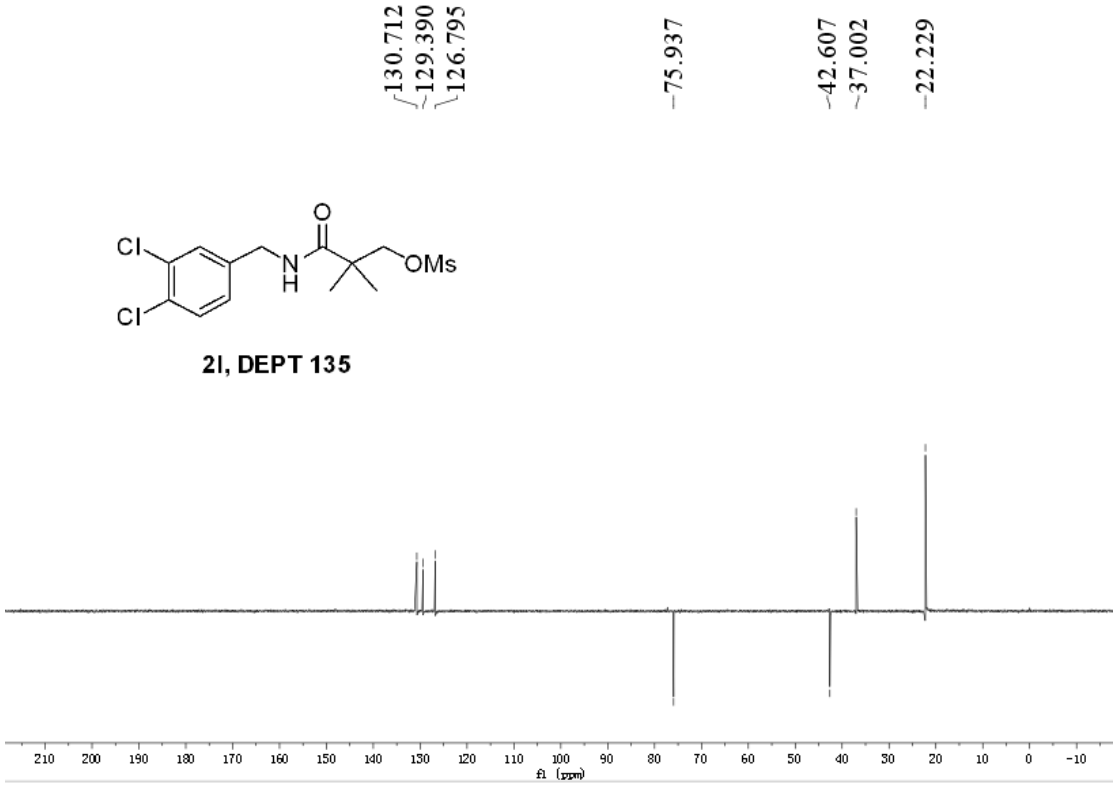
2.97
6.00
0.98
f1 (ppm)



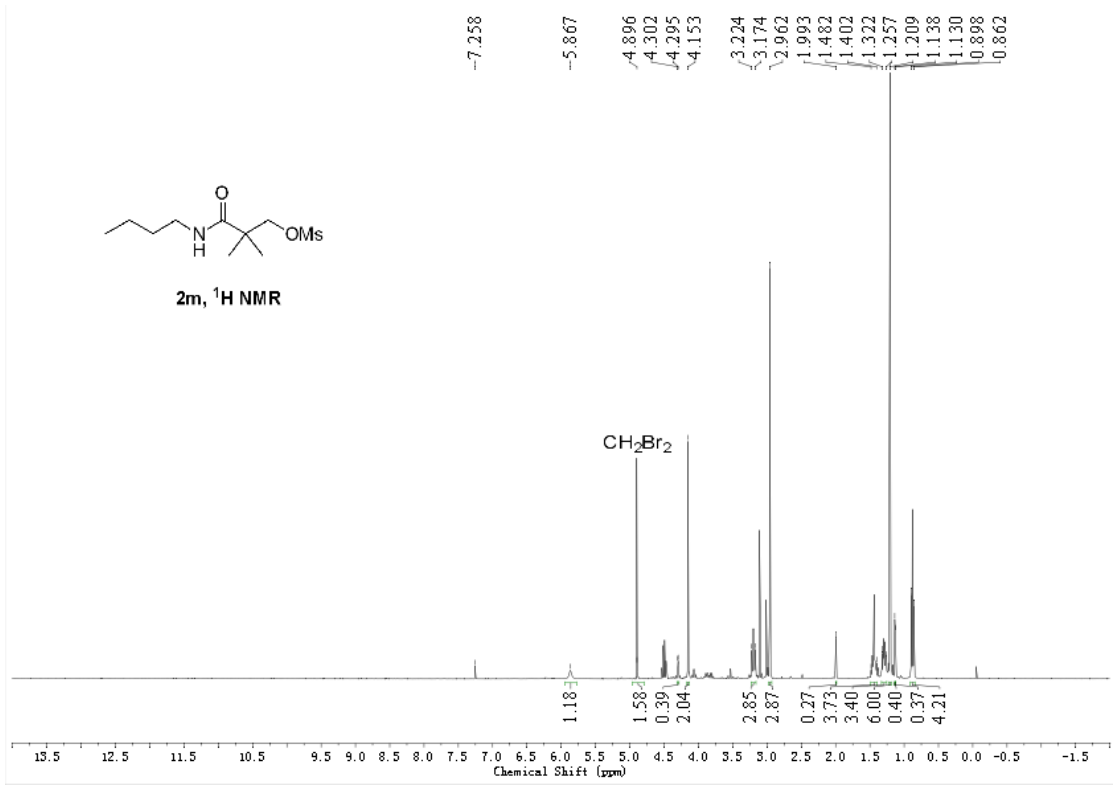
2l, ^{1}H NMR



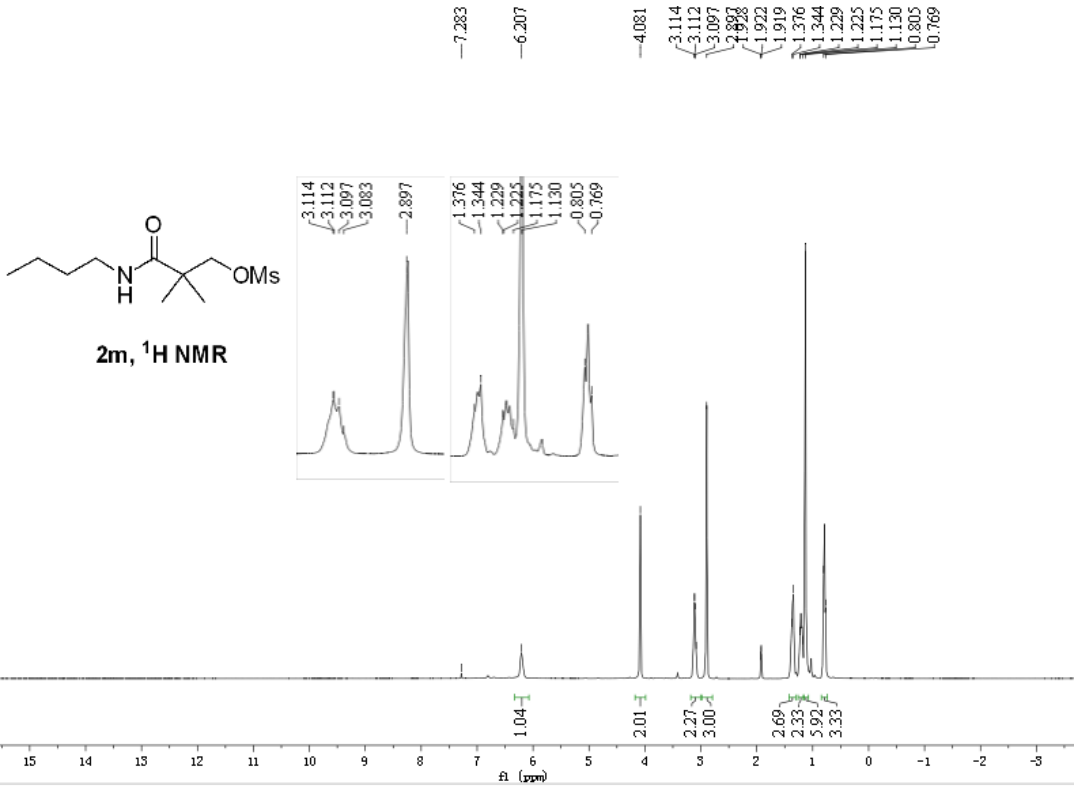
2l, ^{13}C NMR

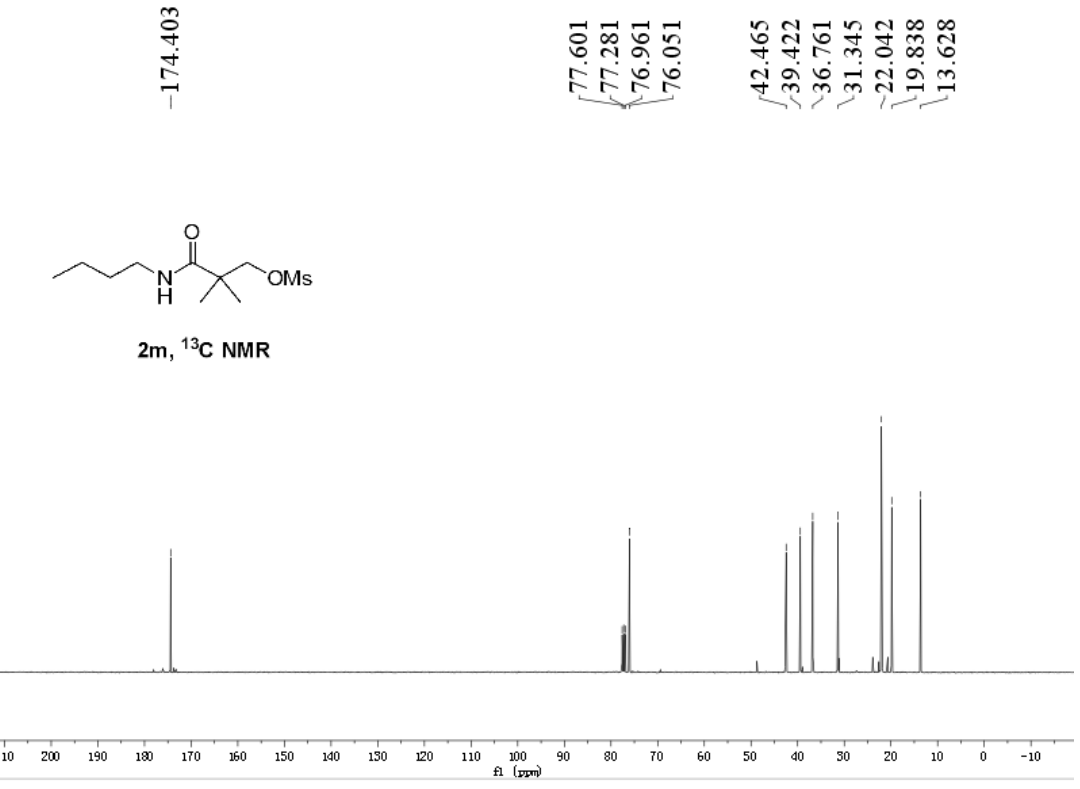


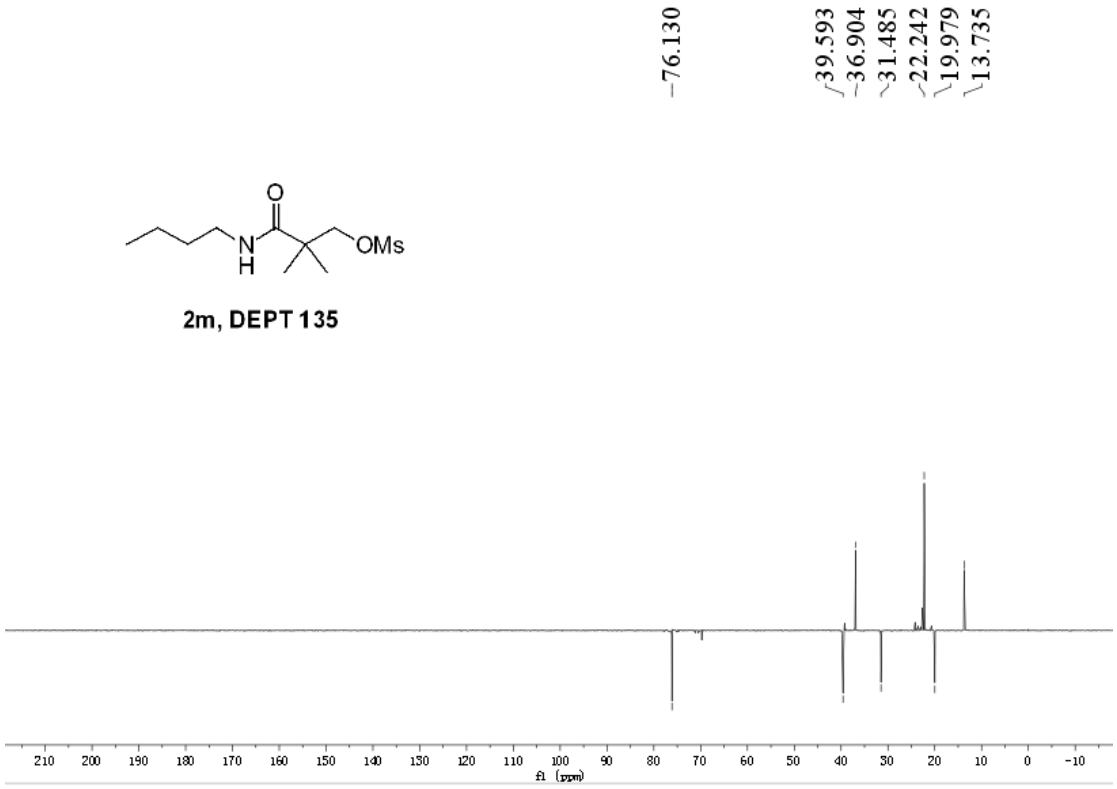
2l, DEPT 135

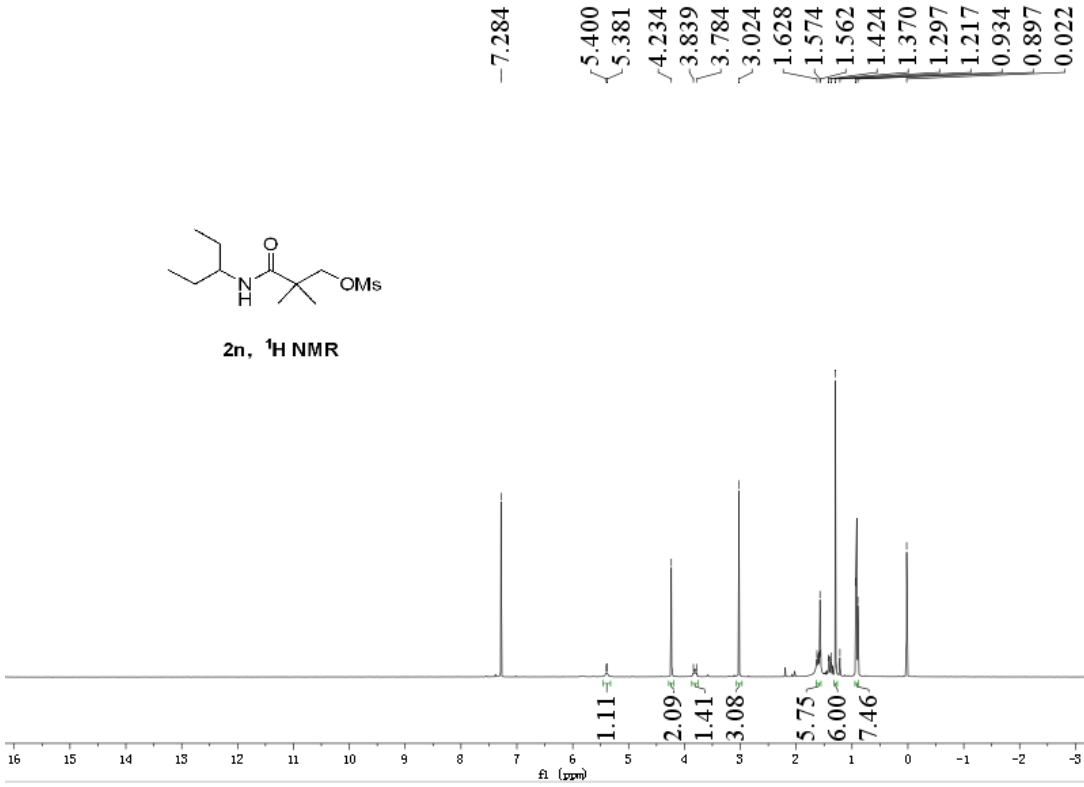


2m, ^{1}H NMR

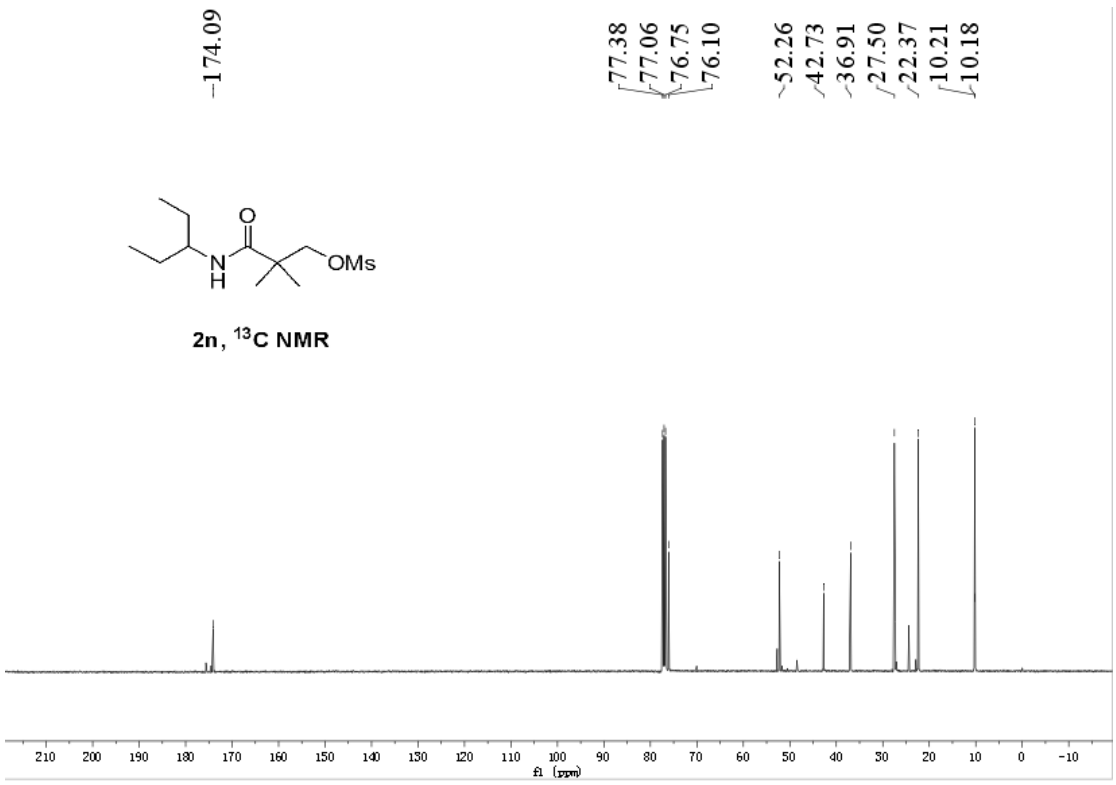

7.283
6.207
4.081
3.114
3.112
3.097
3.083
2.897
1.922
1.919
1.376
1.344
1.229
1.225
1.175
1.130
0.805
0.769
OMs
2m, 1H NMR
1.04
2.01
2.27
3.00
2.69
2.33
5.92
3.33
f1 (ppm)


174.403
77.601
77.281
76.961
76.051
42.465
39.422
36.761
31.345
22.042
19.838
13.628
OMs
2m, 13C NMR
f1 (ppm)

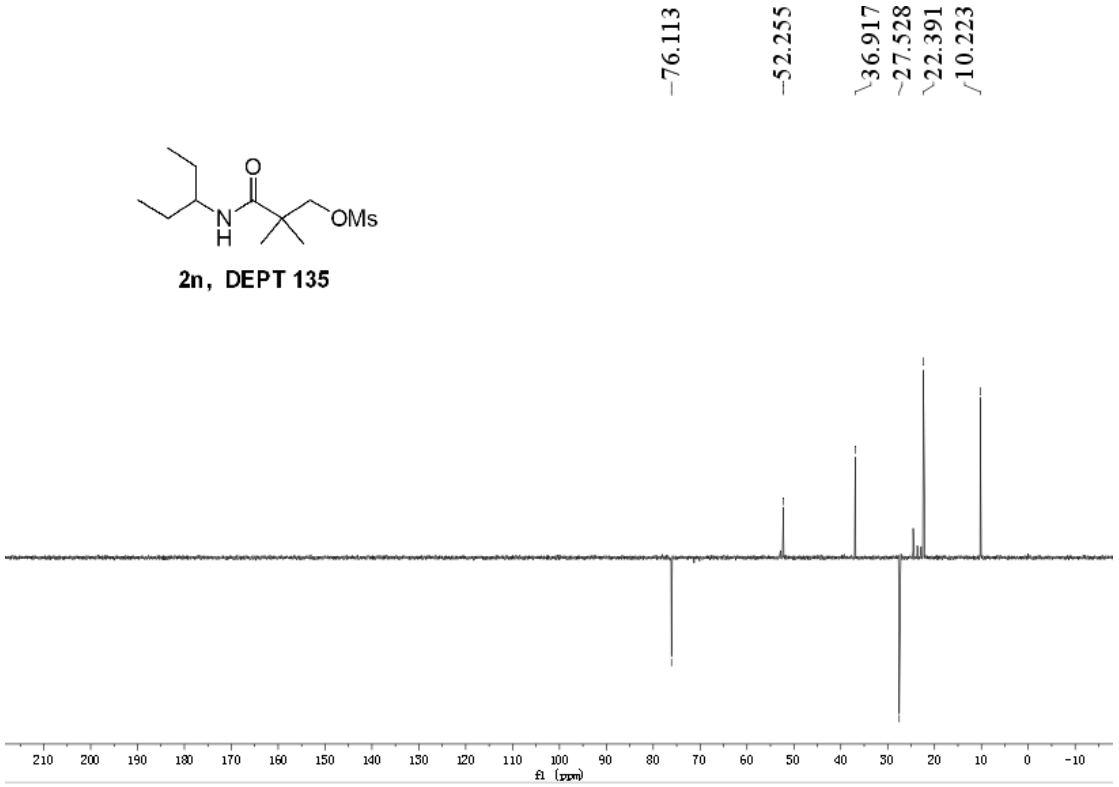

76.130
39.593
36.904
31.485
22.242
19.979
13.735
OMs
2m, DEPT 135
7.258
4.924
4.175
3.961
3.939
3.918
3.896
3.275
3.269
3.142
2.987
1.258
1.191
1.182
1.177
0.891
0.872
0.870
0.854
0.060
OMs
2n, 1H NMR
CH2Br2
2.00
1.75
2.84
2.13
4.72
4.92
4.13
0.99
11.94
f1 (ppm)

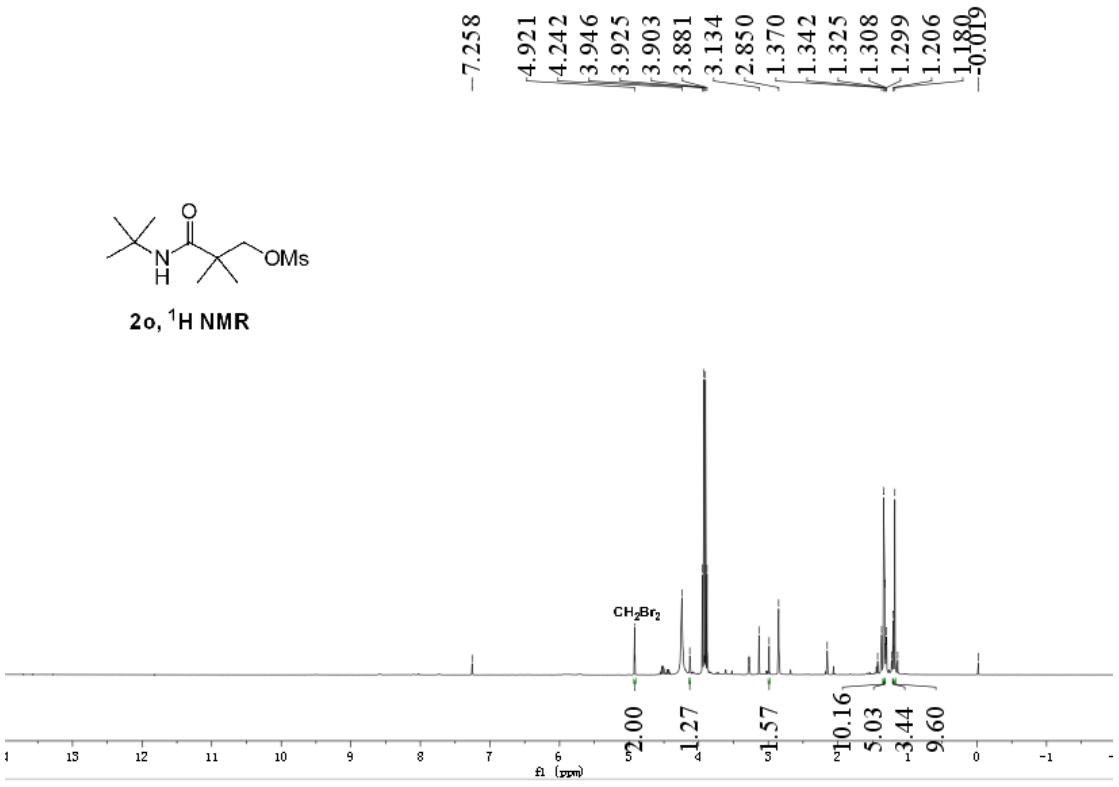


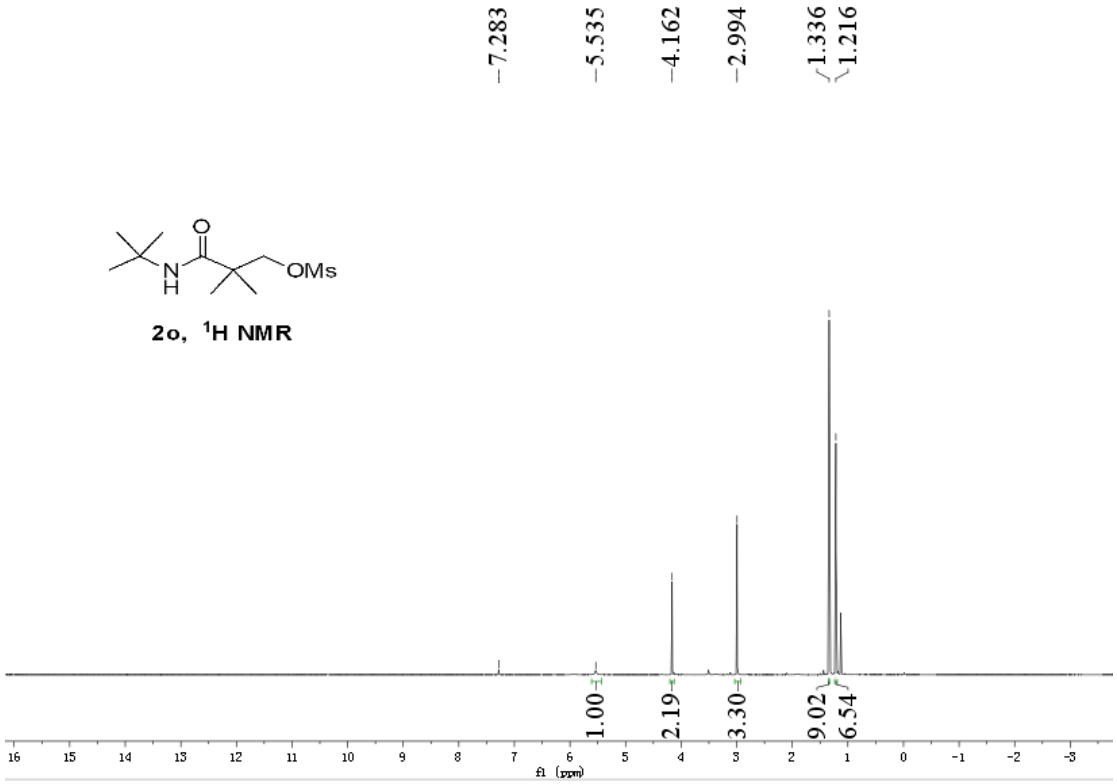
2n, ^{1}H NMR



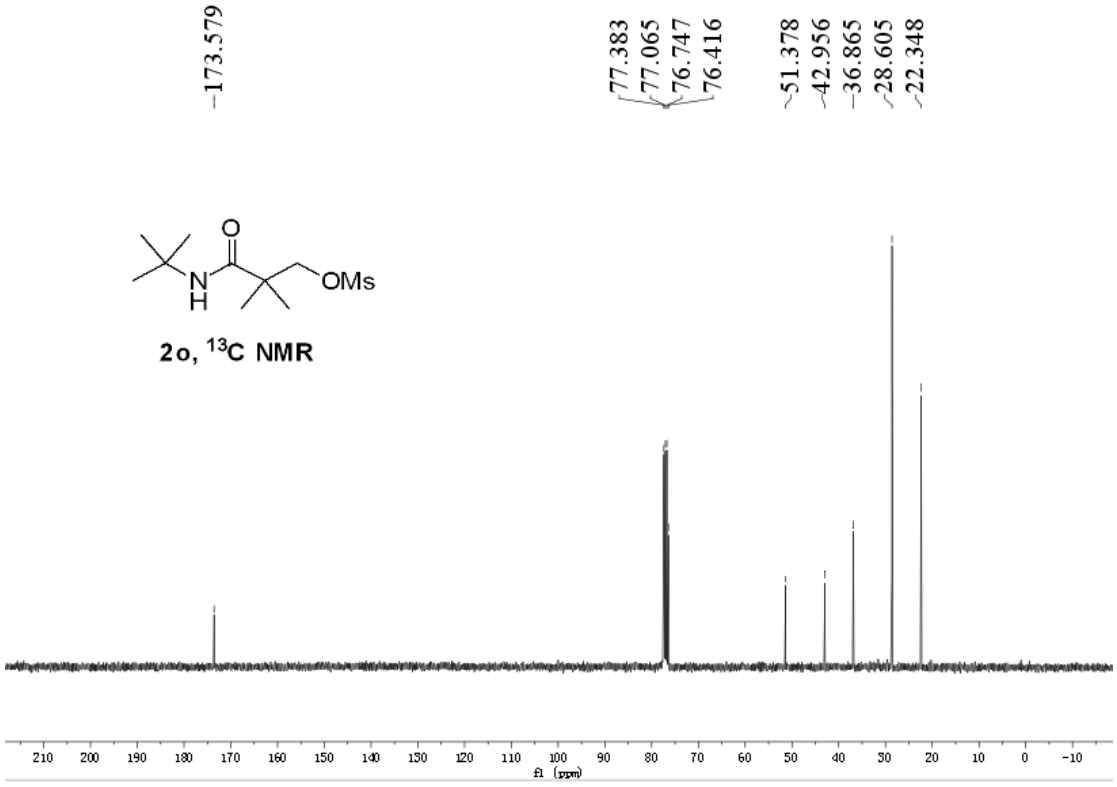
2n, ^{13}C NMR


76.113
52.255
36.917
27.528
22.391
10.223
OMs
2n, DEPT 135
f1 (ppm)

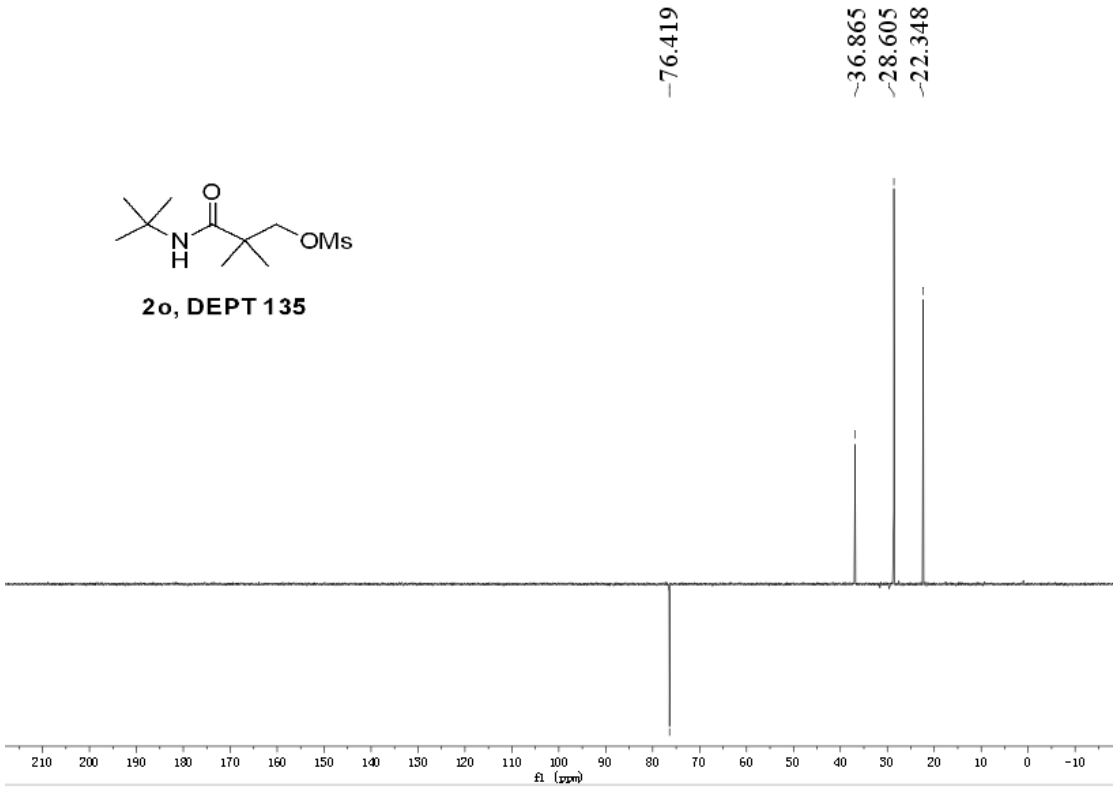

7.258
4.921
4.242
3.946
3.925
3.903
3.881
3.134
2.850
1.370
1.342
1.325
1.308
1.299
1.206
1.180
0.019
OMs
2o, 1H NMR
CH2Br2
2.00
1.27
1.57
10.16
5.03
3.44
9.60
f1 (ppm)

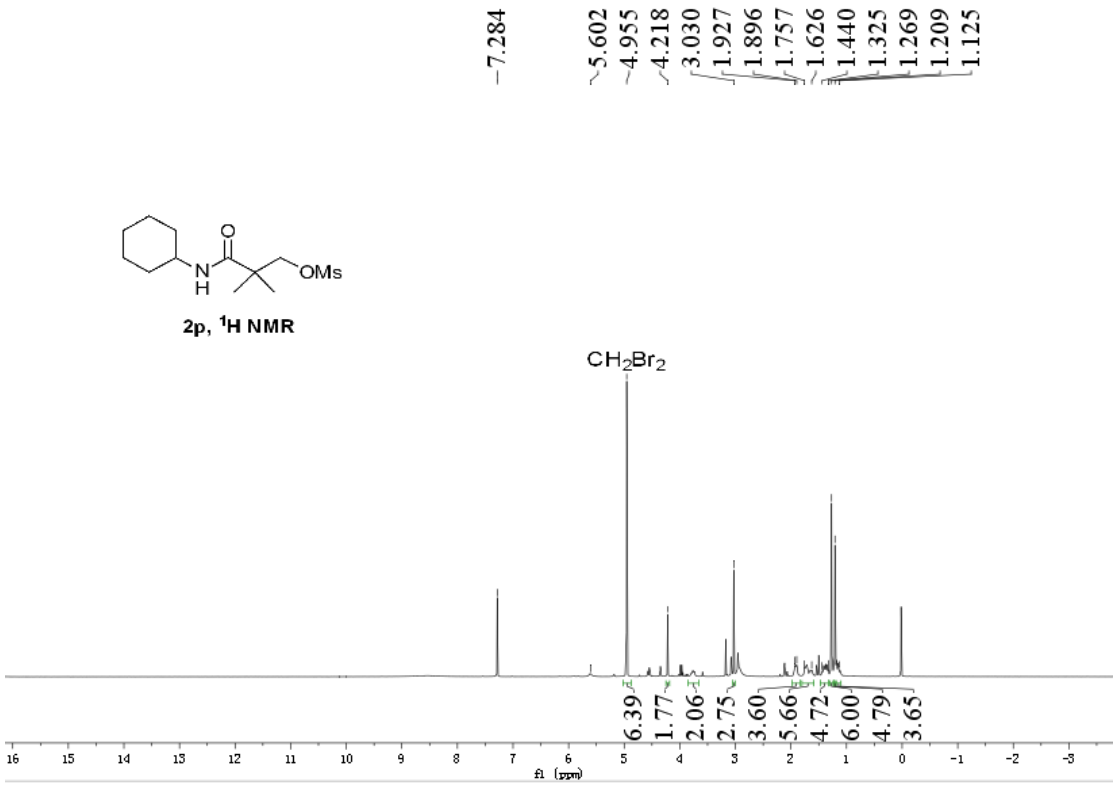


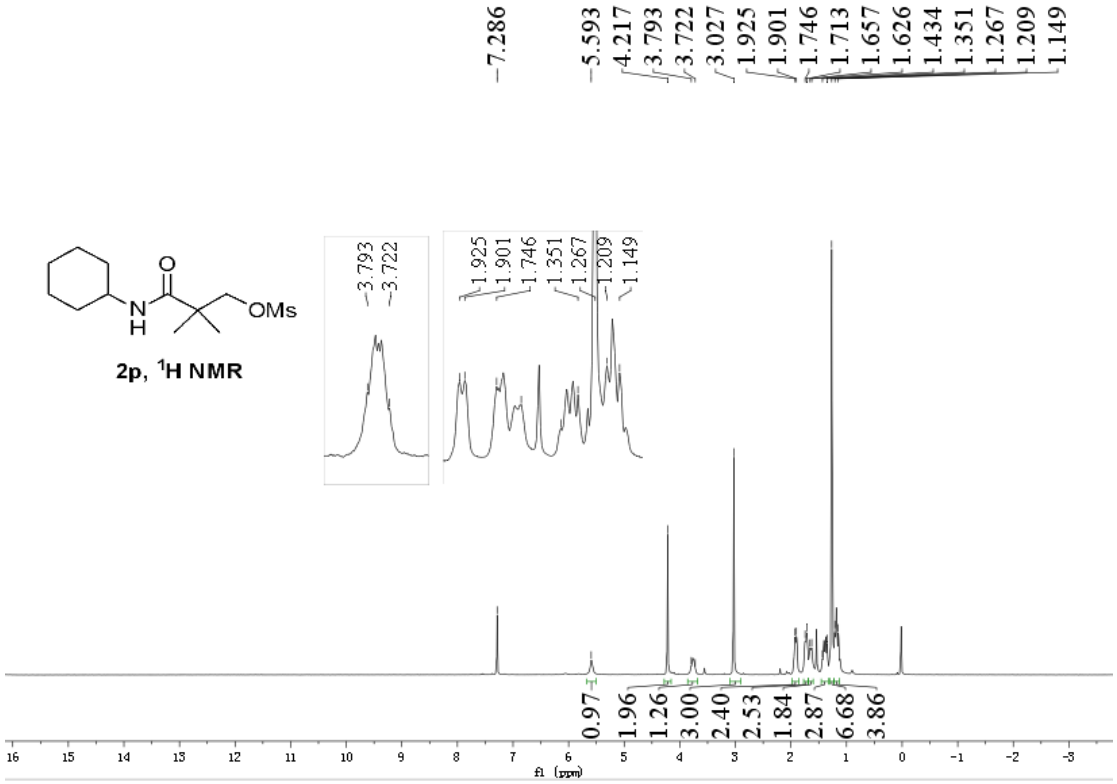
2o, ^{1}H NMR

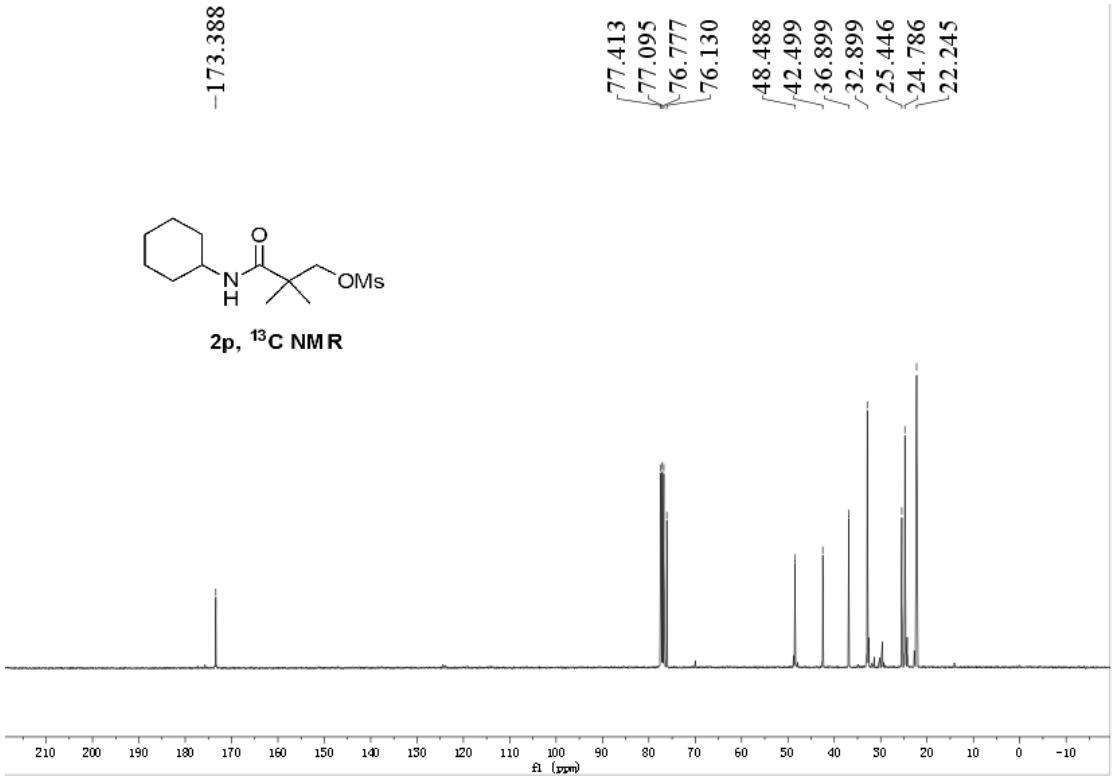


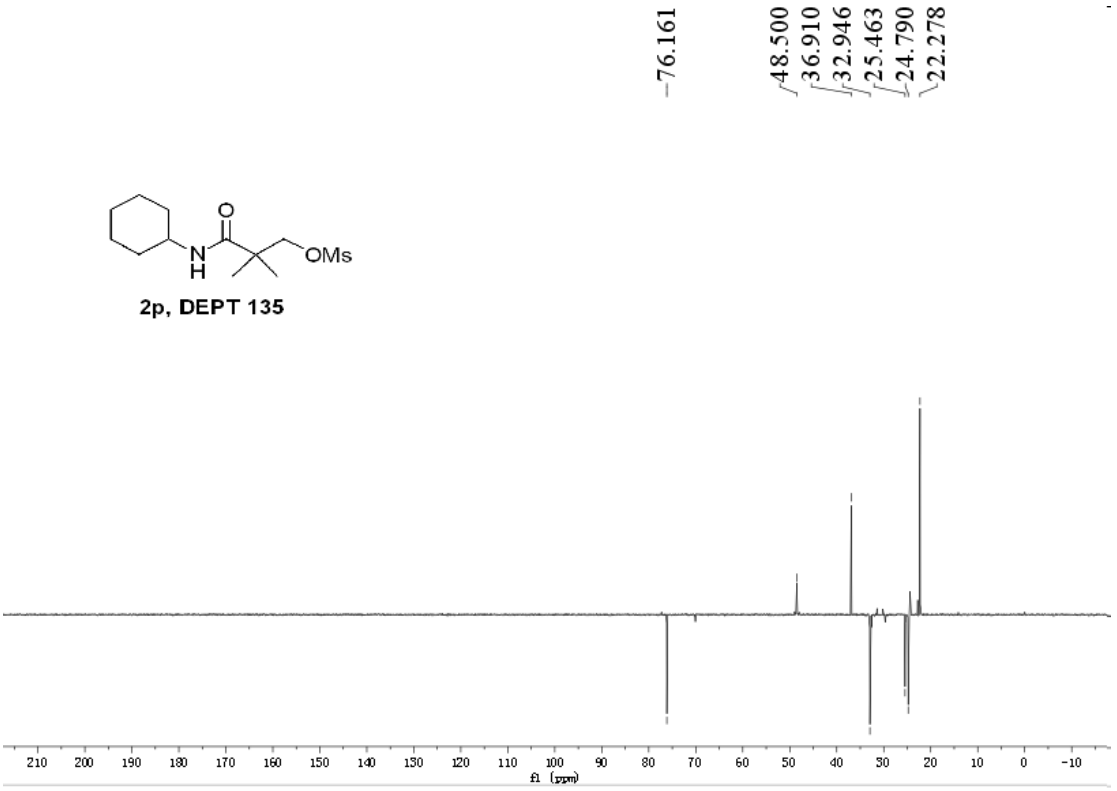
2o, ^{13}C NMR


76.419
36.865
28.605
22.348
OMs
2o, DEPT 135
f1 (ppm)

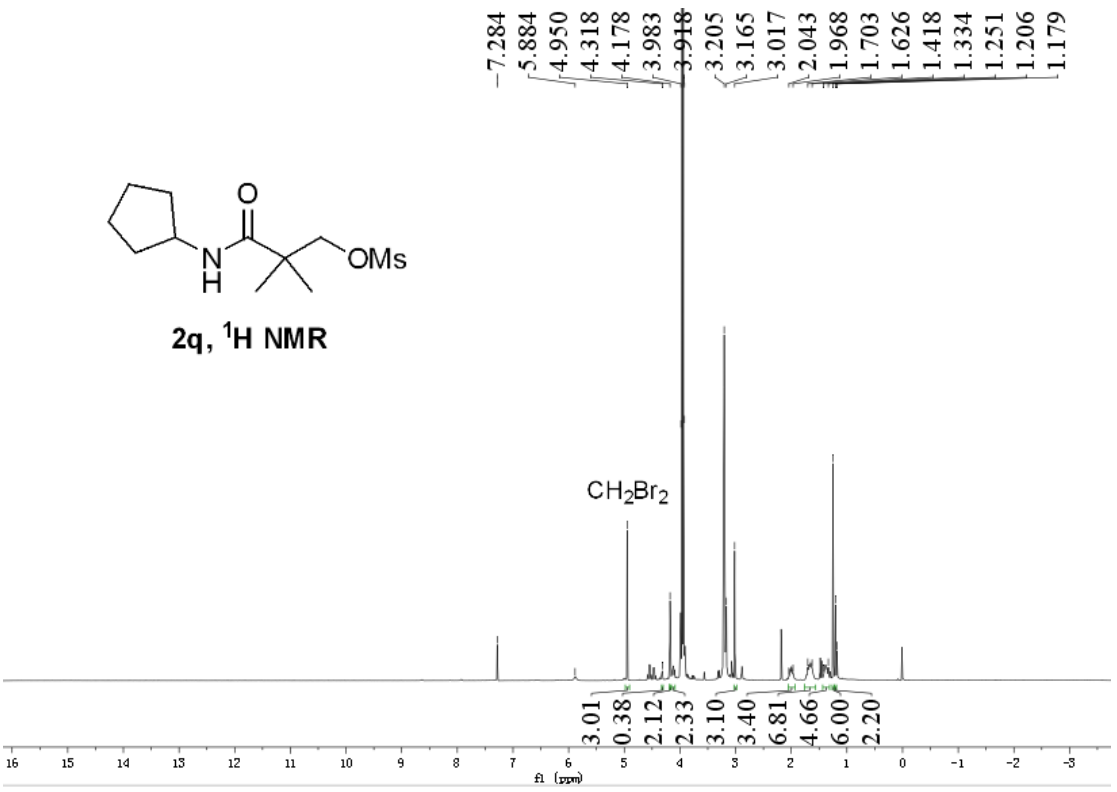

7.284
5.602
4.955
4.218
3.030
1.927
1.896
1.757
1.626
1.440
1.325
1.269
1.209
1.125
OMs
2p, ^{1}H NMR
CH_2Br_2
6.39
1.77
2.06
2.75
3.60
5.66
4.72
6.00
4.79
3.65
f1 (ppm)


7.286
5.593
4.217
3.793
3.722
3.027
1.925
1.901
1.746
1.713
1.657
1.626
1.434
1.351
1.267
1.209
1.149
OMs
2p, ^{1}H NMR
0.97
1.96
1.26
3.00
2.40
2.53
1.84
2.87
6.68
3.86
f1 (ppm)

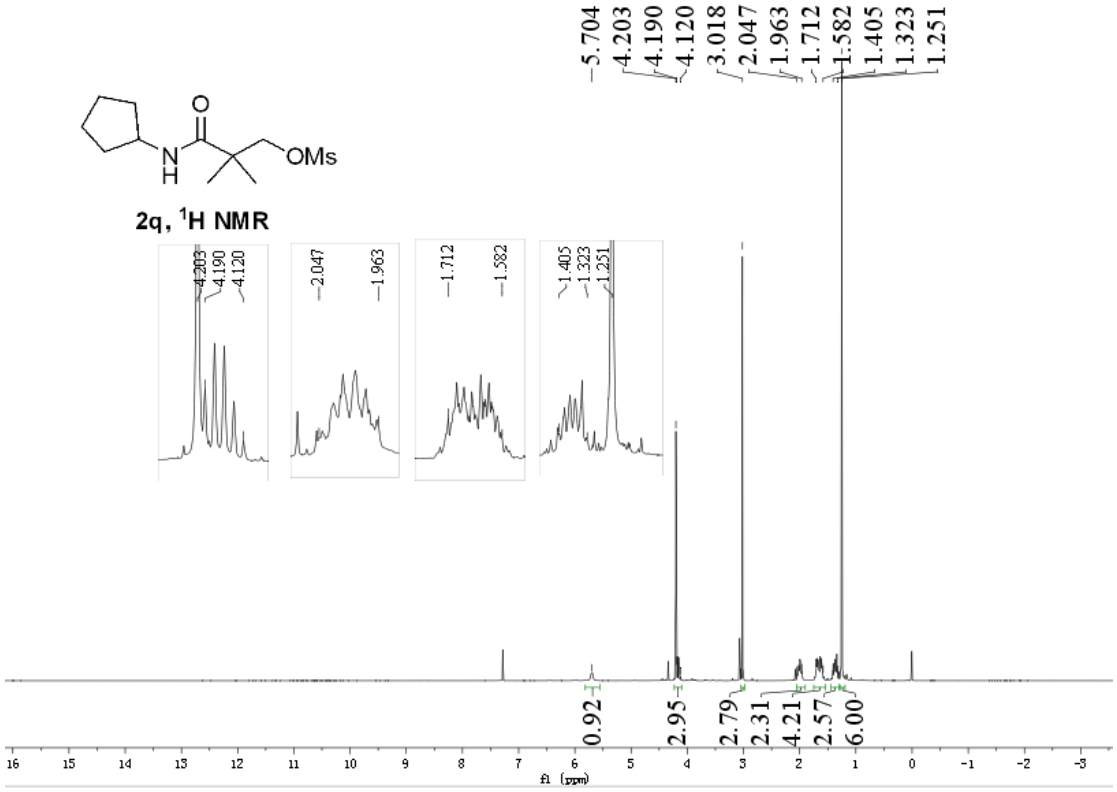

173.388
77.413
77.095
76.777
76.130
48.488
42.499
36.899
32.899
25.446
24.786
22.245
OMs
2p, ^{13}C NMR
f1 (ppm)

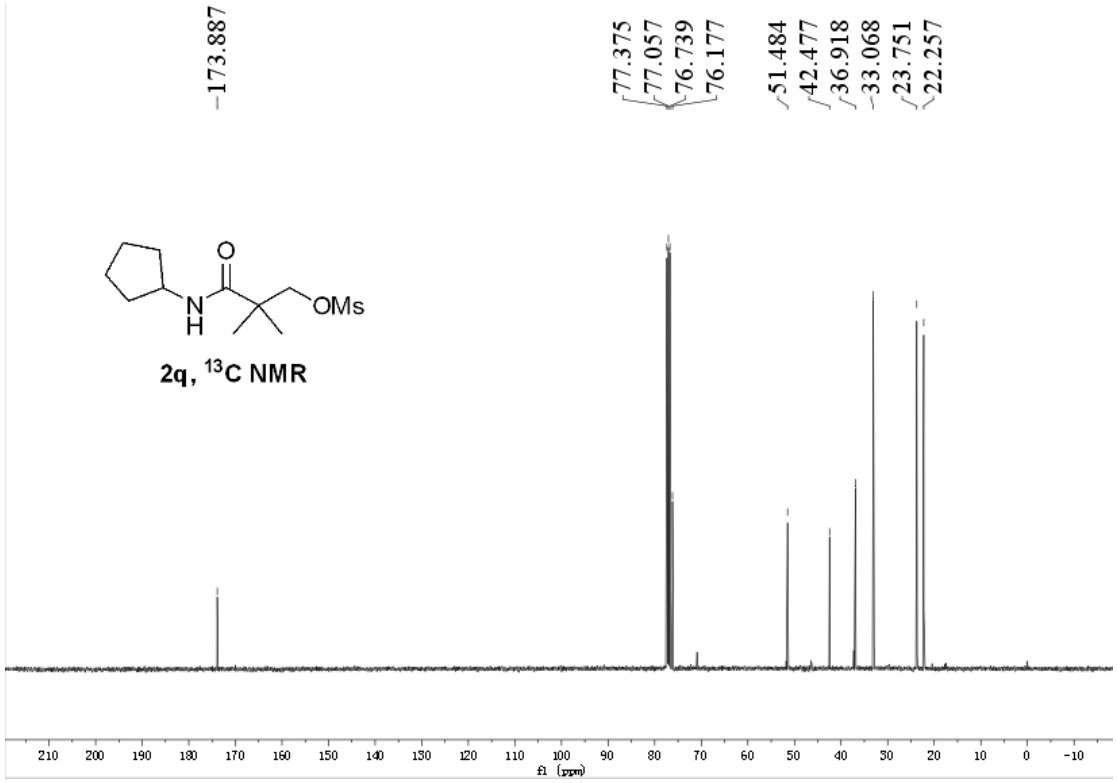


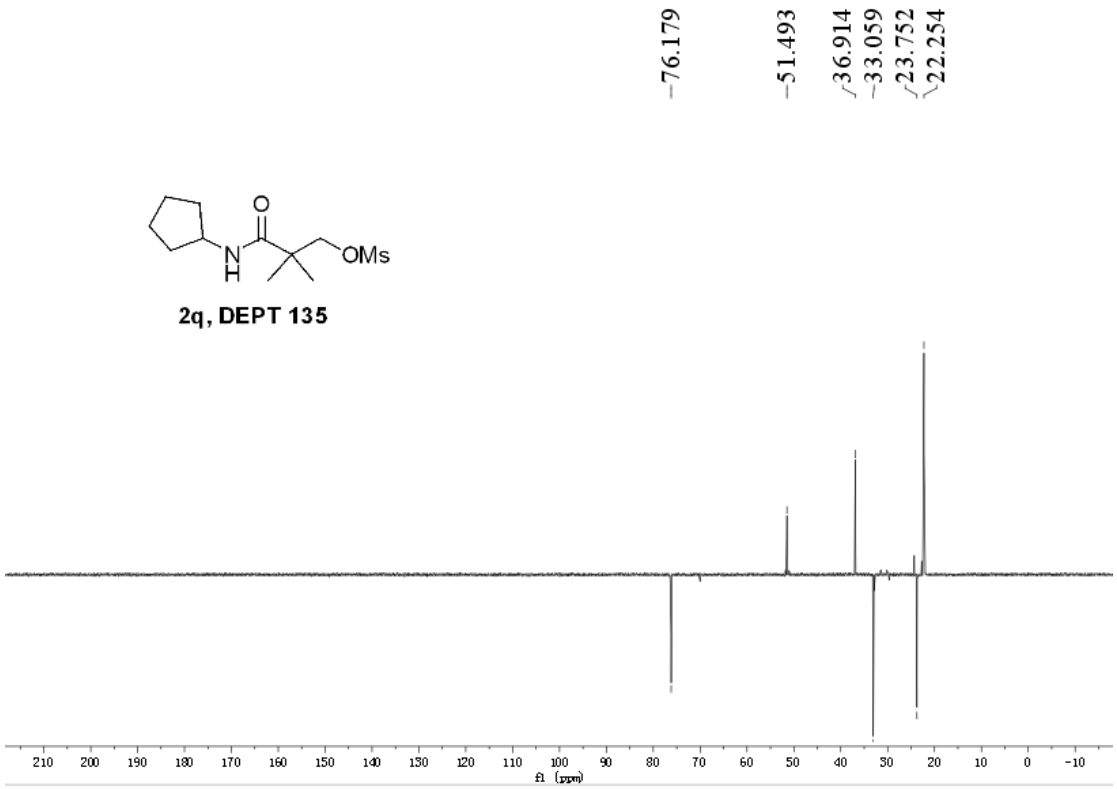
2p, DEPT 135



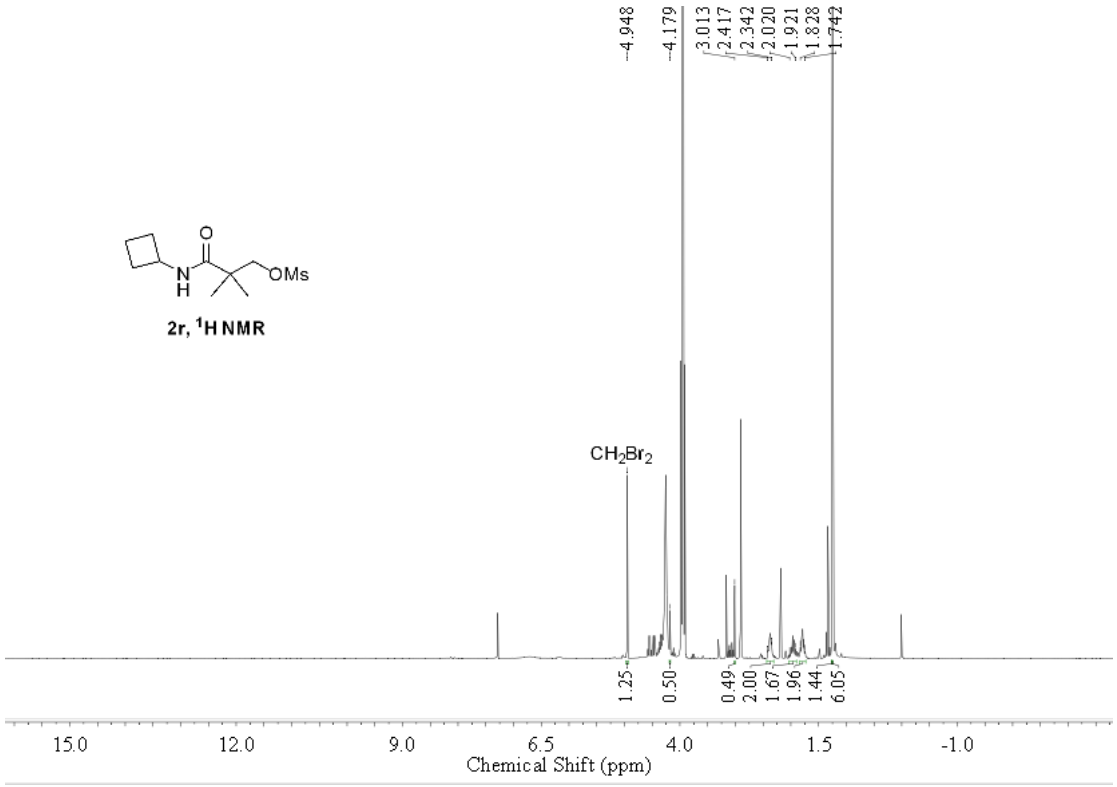
2q, ^{1}H NMR


5.704
4.203
4.190
4.120
3.018
2.047
1.963
1.712
1.582
1.405
1.323
1.251
OMs
2q, 1H NMR
0.92
2.95
2.79
2.31
4.21
2.57
6.00
f1 (ppm)

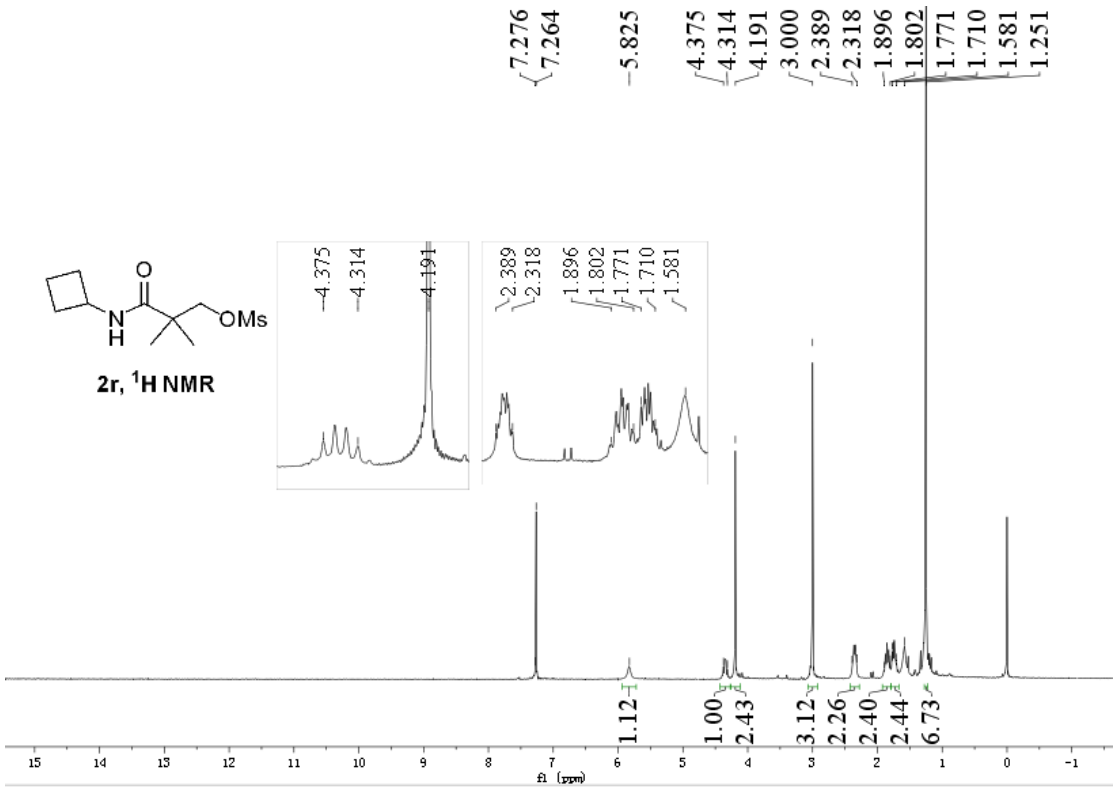

173.887
77.375
77.057
76.739
76.177
51.484
42.477
36.918
33.068
23.751
22.257
OMs
2q, 13C NMR
f1 (ppm)



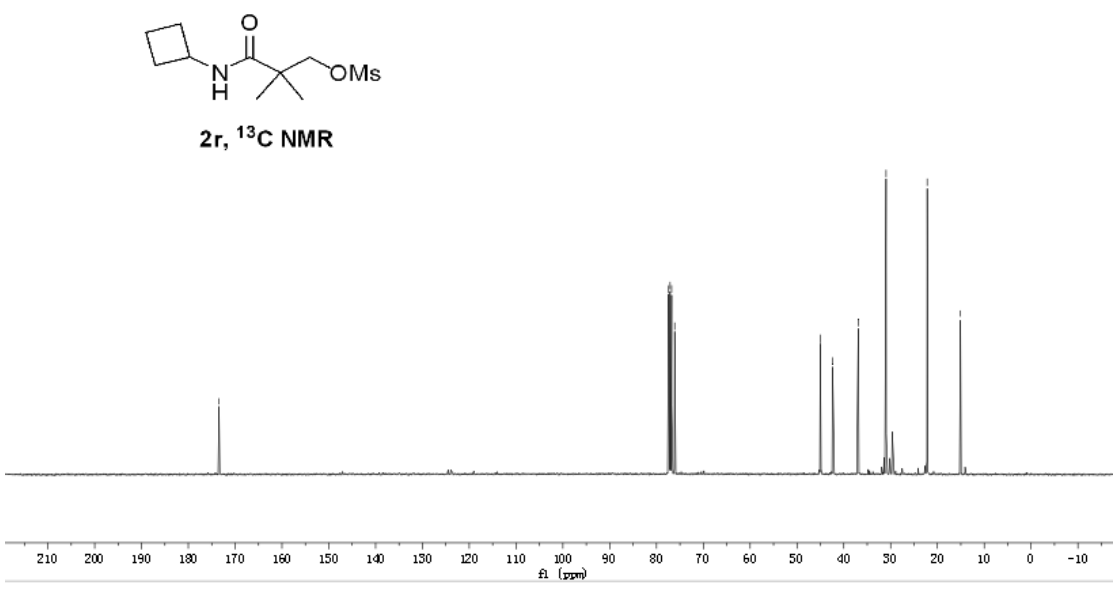
2q, DEPT 135



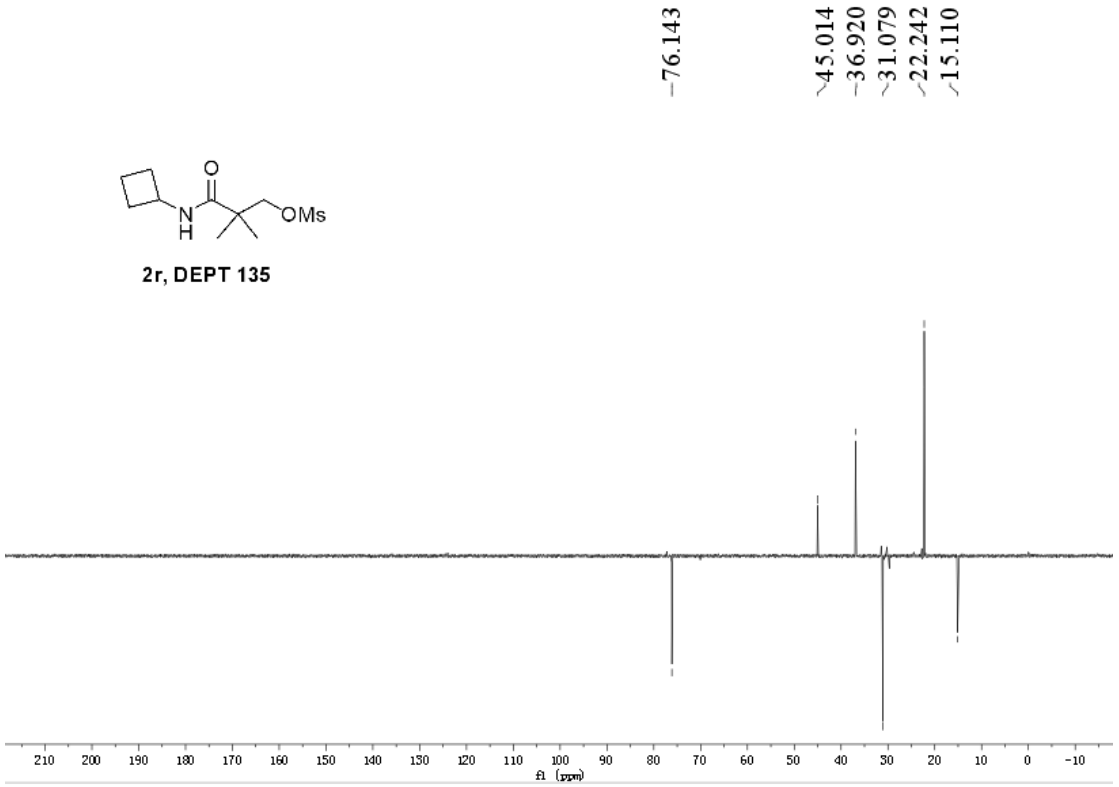
2r, ^{1}H NMR



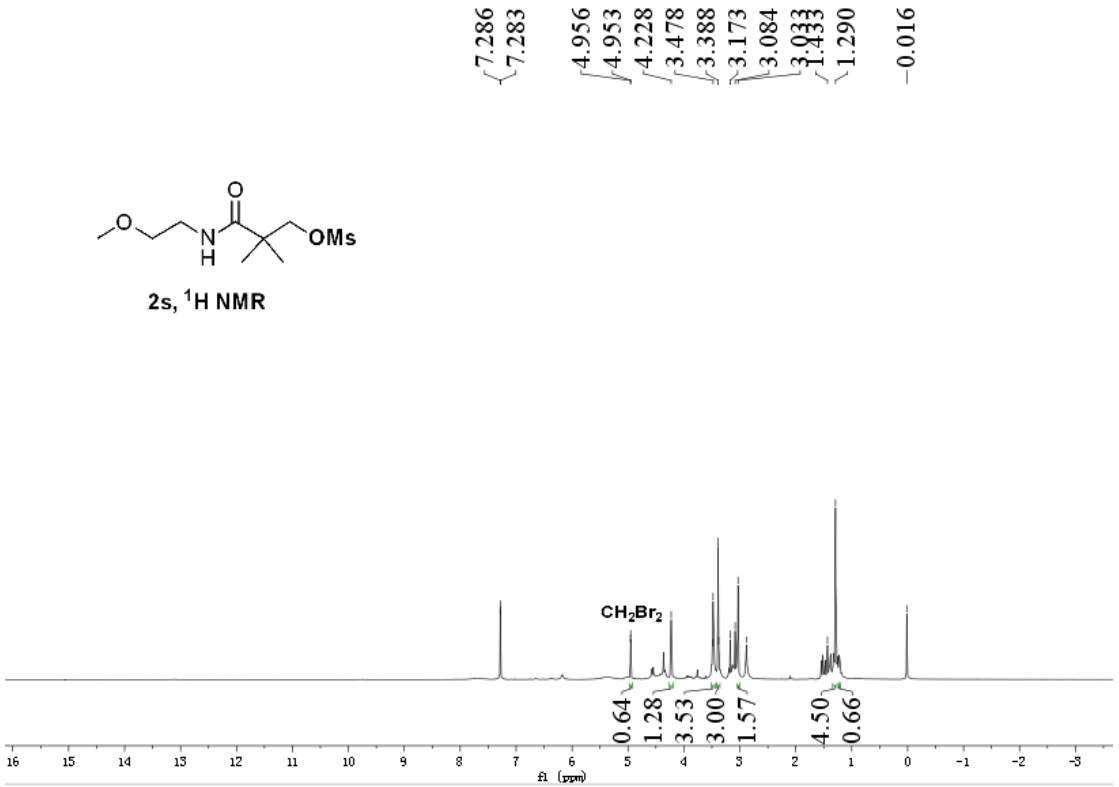
2r, ^{1}H NMR



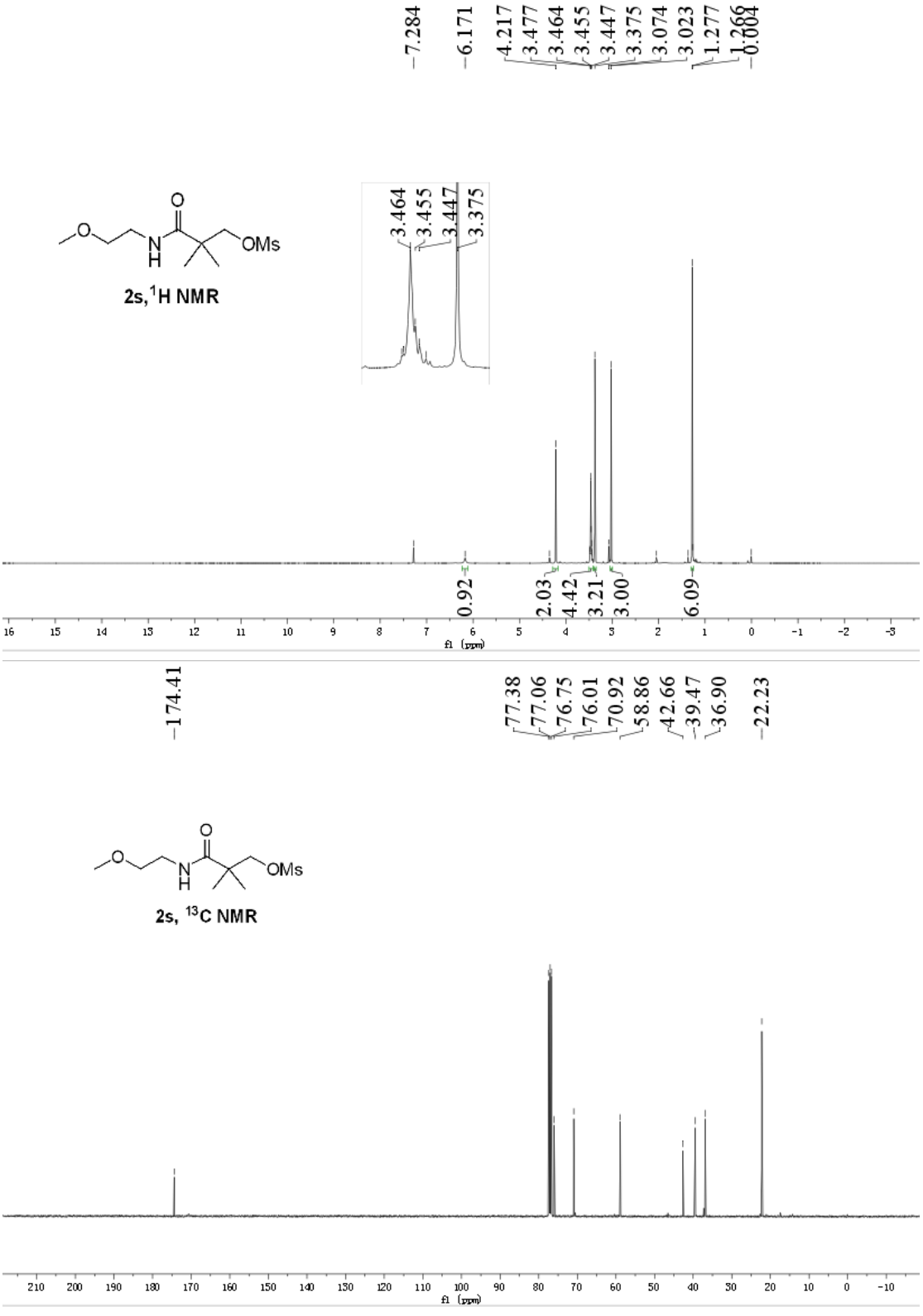
2r, ^{13}C NMR

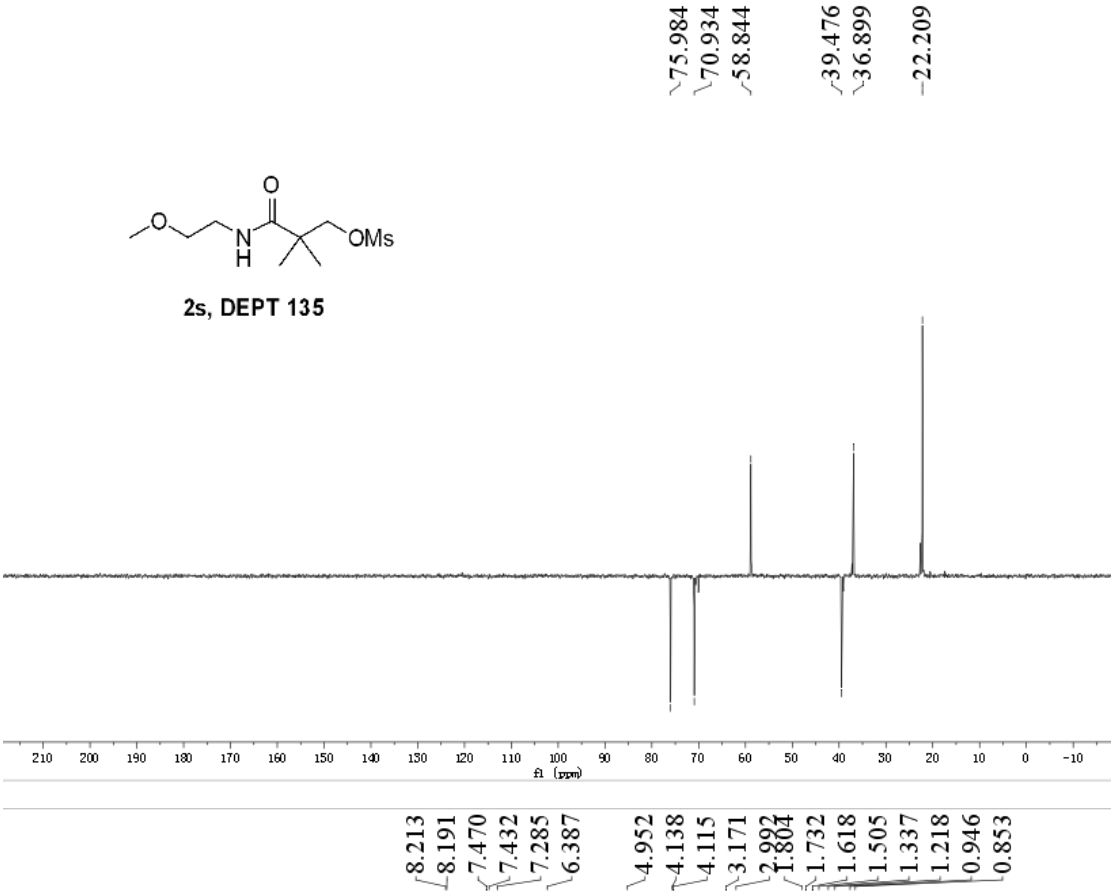


2r, DEPT 135

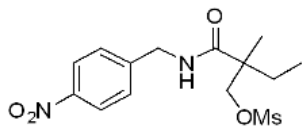


2s, ^{1}H NMR

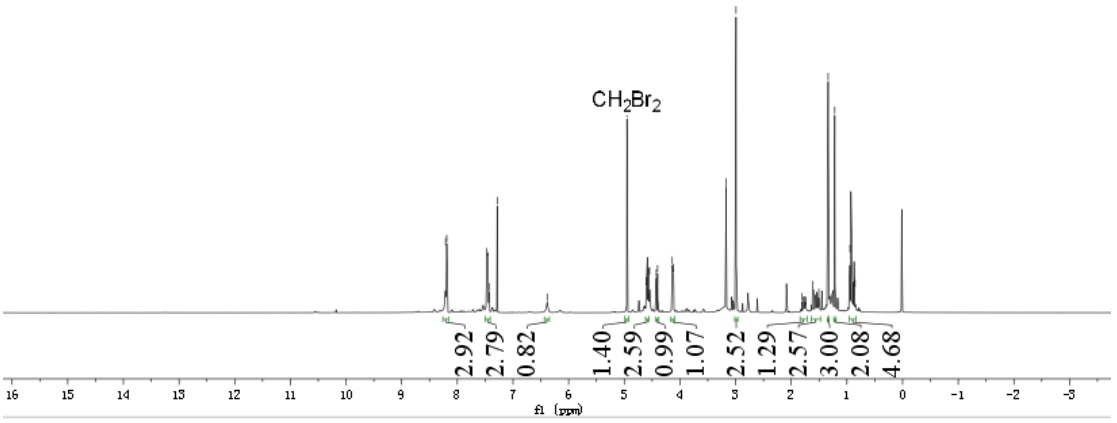

7.284
6.171
4.217
3.477
3.464
3.455
3.447
3.375
3.074
3.023
1.277
1.266
0.004
OMs
2s, ^{1}H NMR
3.464
3.455
3.447
3.375
0.92
2.03
4.42
3.21
3.00
6.09
f1 (ppm)
174.41
77.38
77.06
76.75
76.01
70.92
58.86
42.66
39.47
36.90
22.23
OMs
2s, ^{13}C NMR
f1 (ppm)

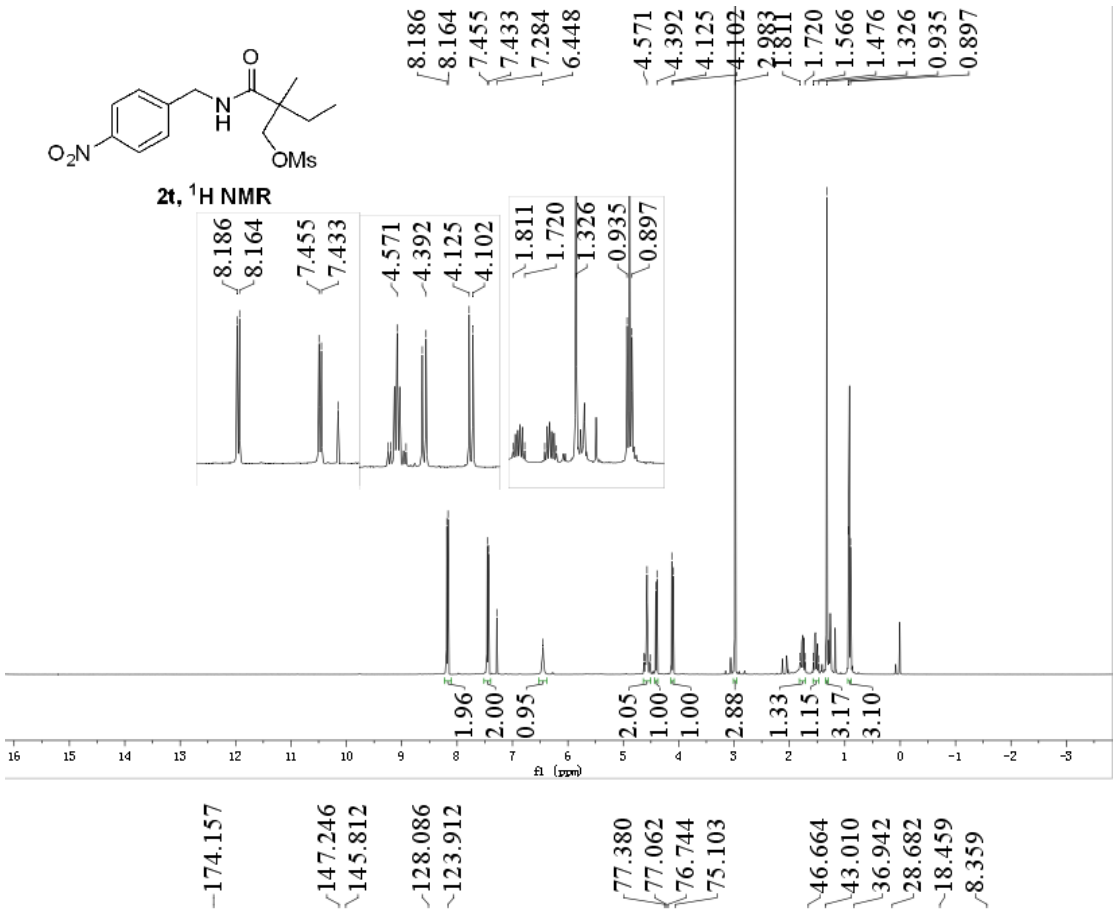


2s, DEPT 135

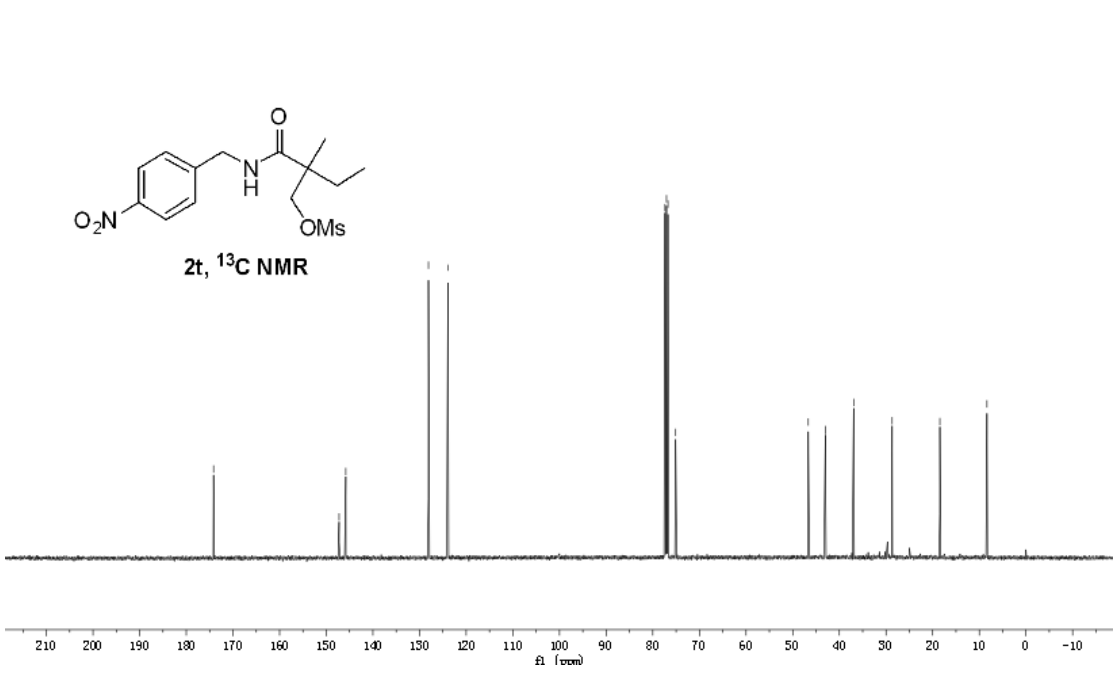


2t, ^{1}H NMR

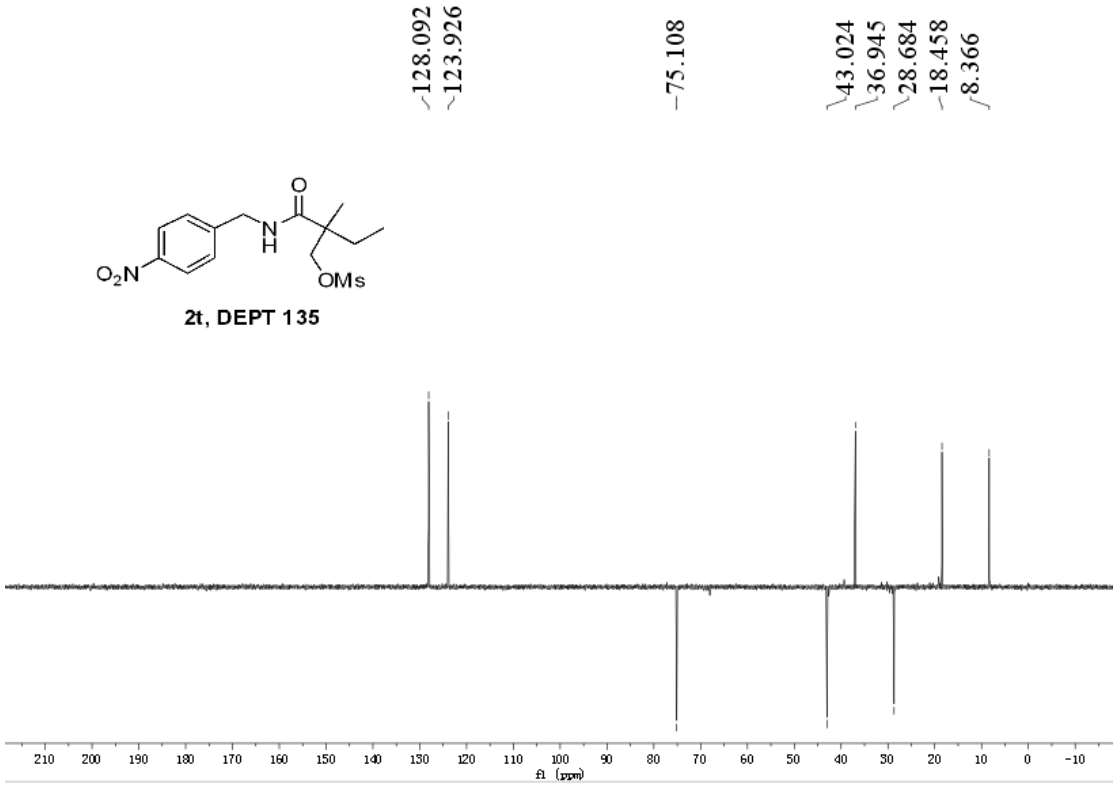




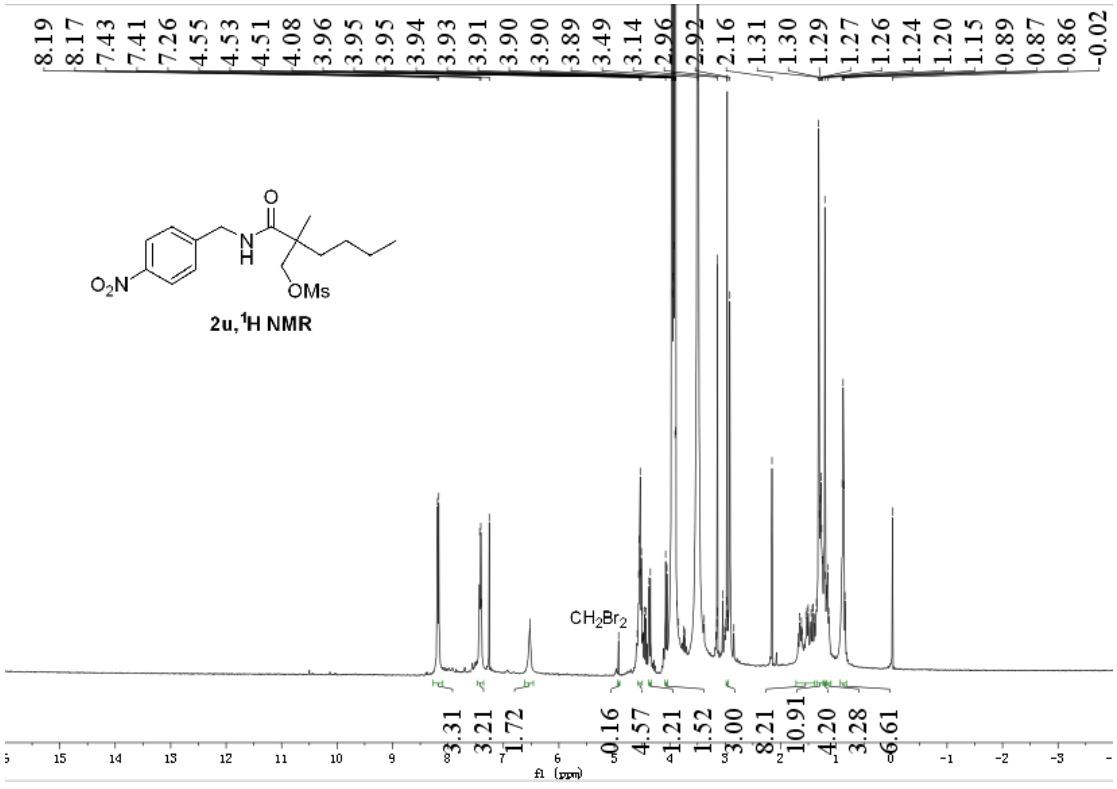
2t, ^{1}H NMR



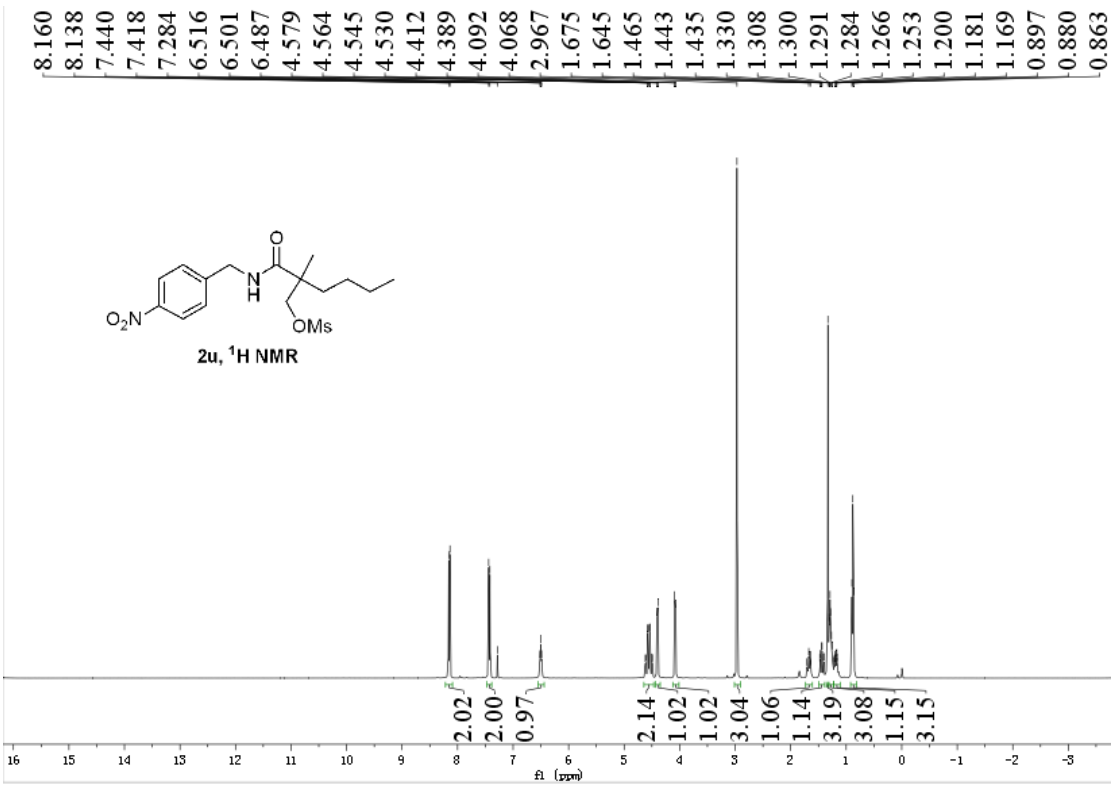
2t, ^{13}C NMR

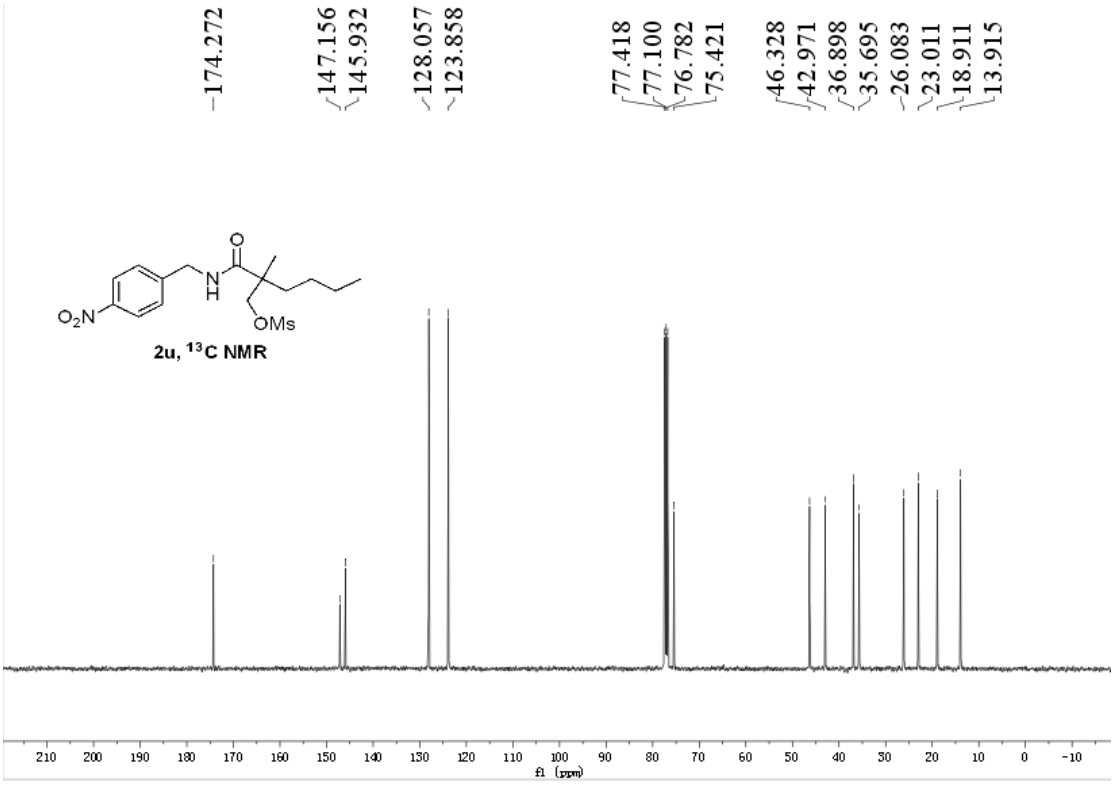


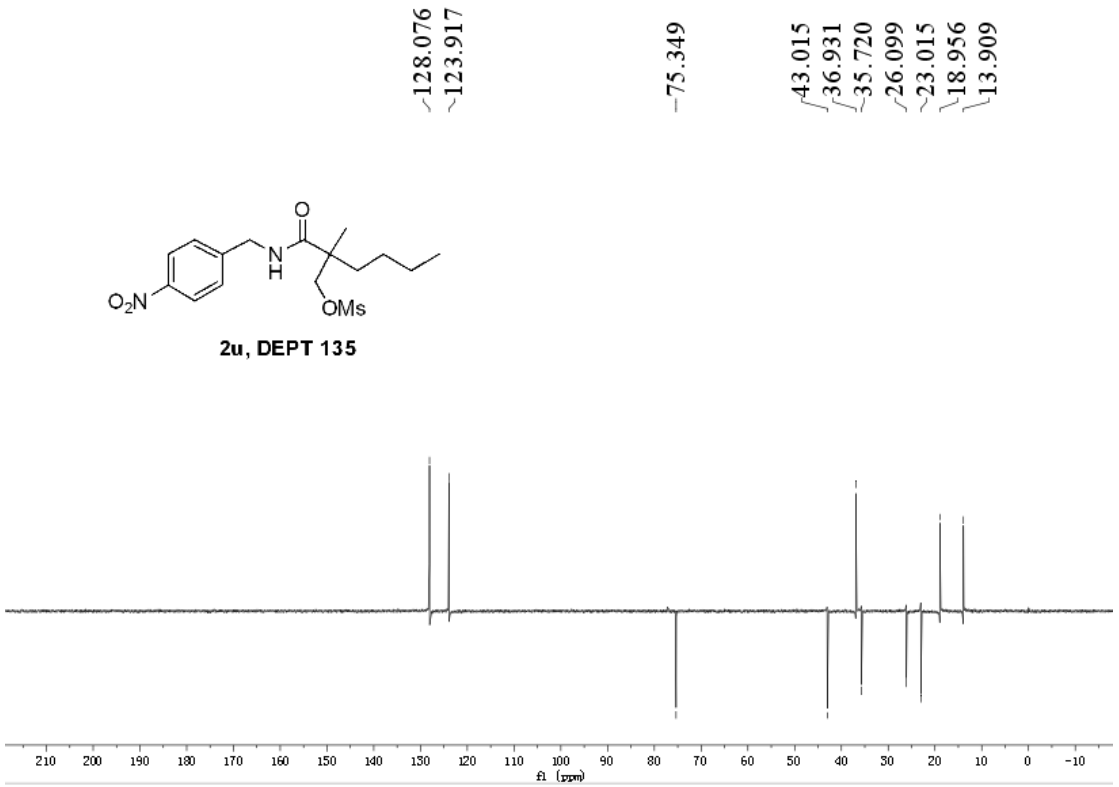
2t, DEPT 135



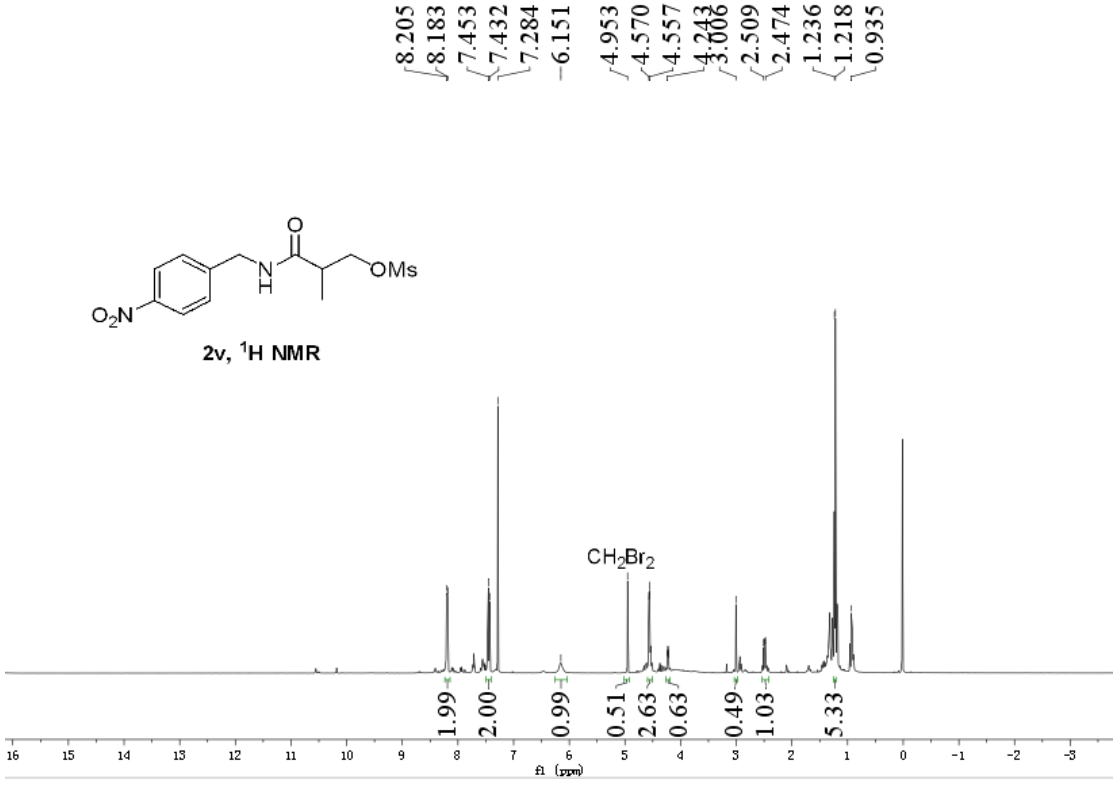
2u, ^{1}H NMR


8.160
8.138
7.440
7.418
7.284
6.516
6.501
6.487
4.579
4.564
4.545
4.530
4.412
4.389
4.092
4.068
2.967
1.675
1.645
1.465
1.443
1.435
1.330
1.308
1.300
1.291
1.284
1.266
1.253
1.200
1.181
1.169
0.897
0.880
0.863
O2N
N
H
O
OMs
2u, 1H NMR
2.02
2.00
0.97
2.14
1.02
1.02
3.04
1.06
1.14
3.19
3.08
1.15
3.15
f1 (ppm)

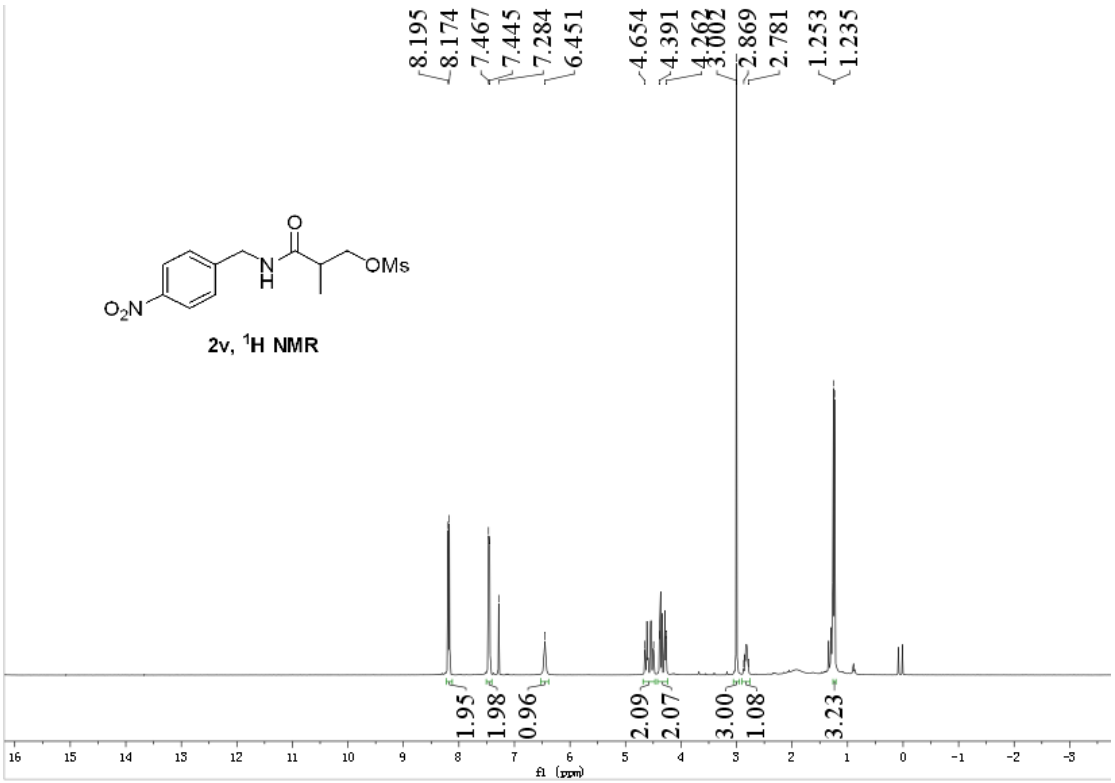

174.272
147.156
145.932
128.057
123.858
77.418
77.100
76.782
75.421
46.328
42.971
36.898
35.695
26.083
23.011
18.911
13.915
O2N
N
H
O
OMs
2u, 13C NMR
f1 (ppm)



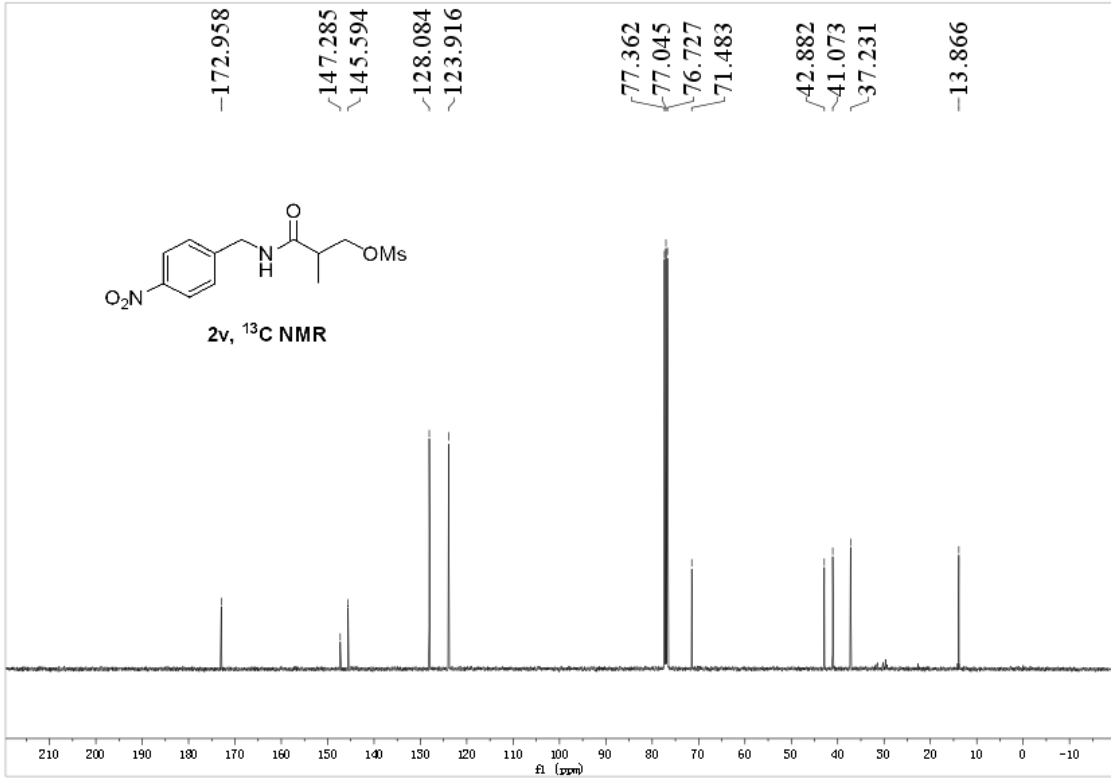
2u, DEPT 135



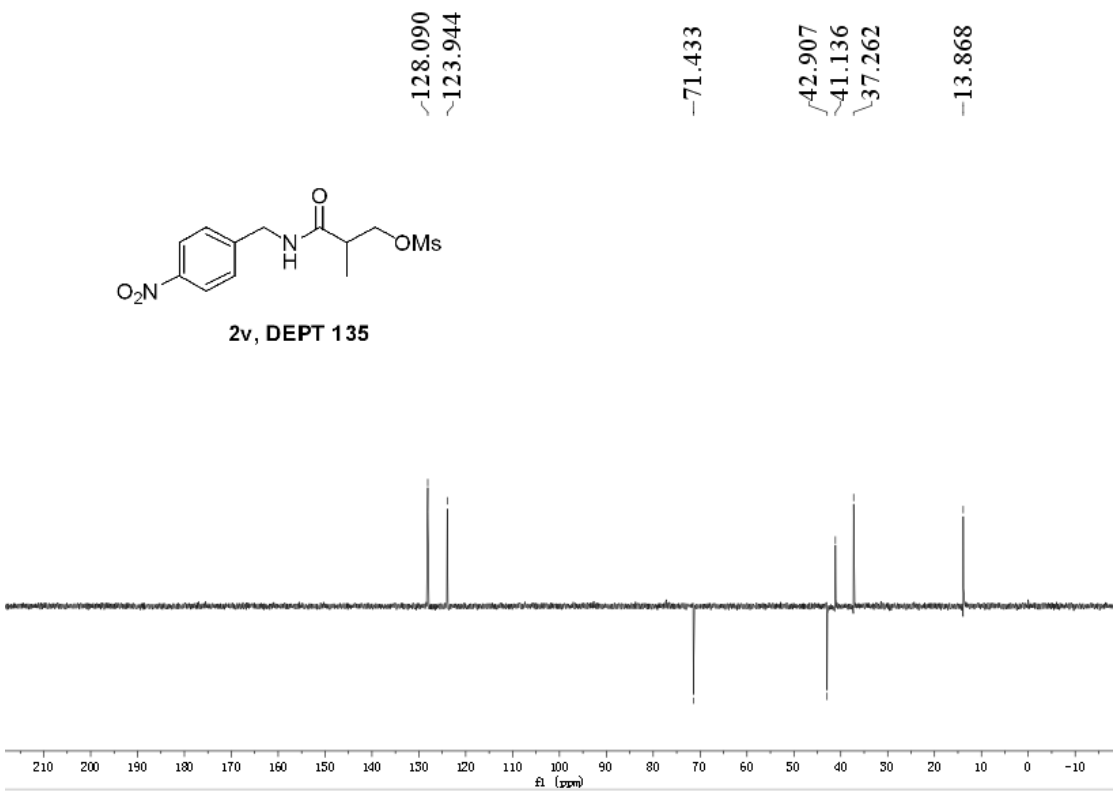
2v, ^{1}H NMR

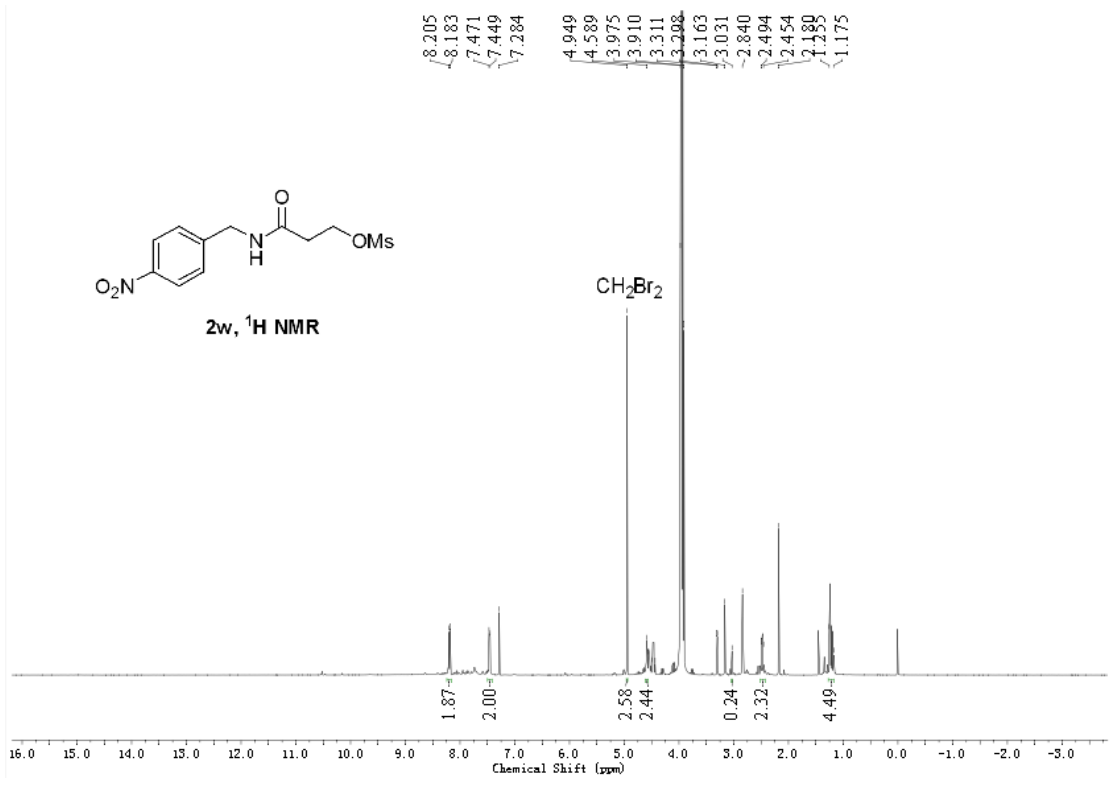


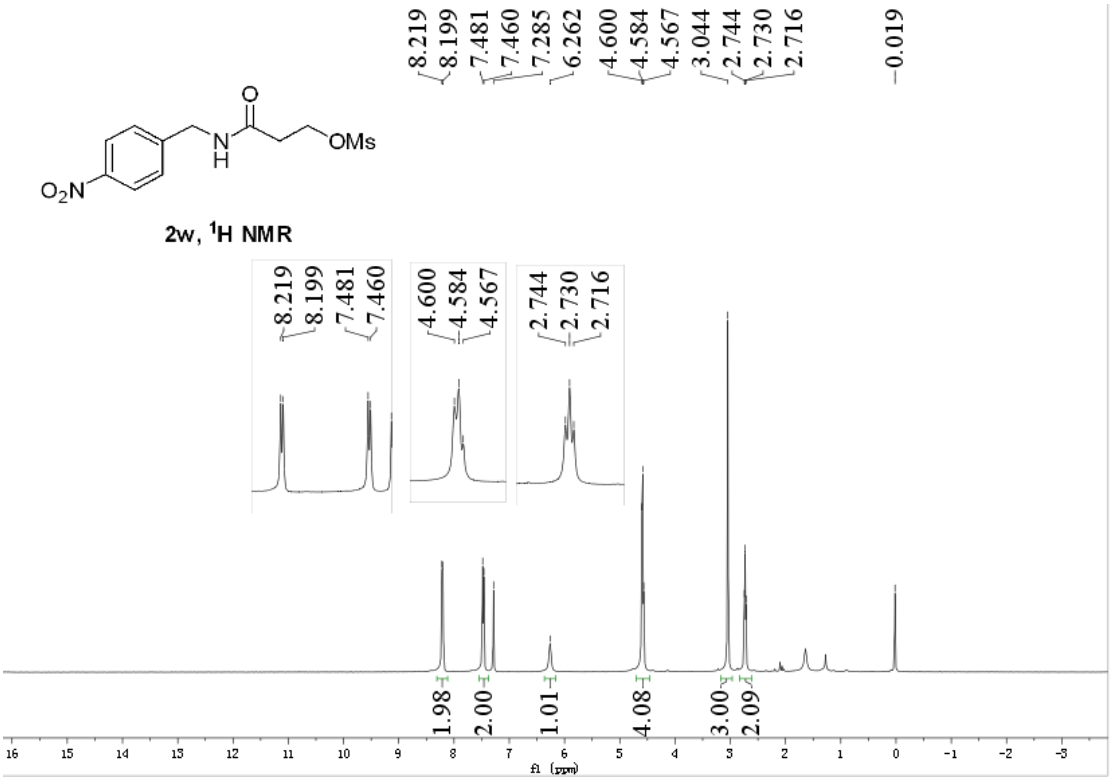
2v, ^{1}H NMR



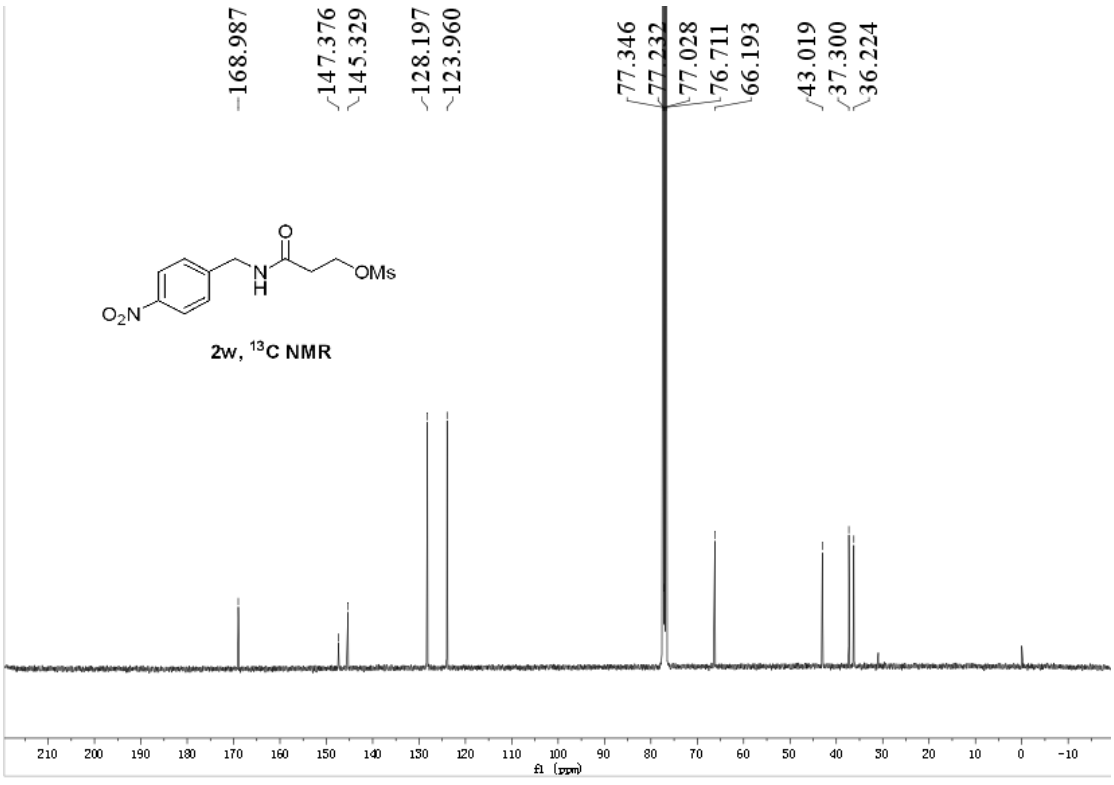
2v, ^{13}C NMR


128.090
123.944
71.433
42.907
41.136
37.262
13.868
O
N
H
OMs
O2N
2v, DEPT 135
fl (ppm)

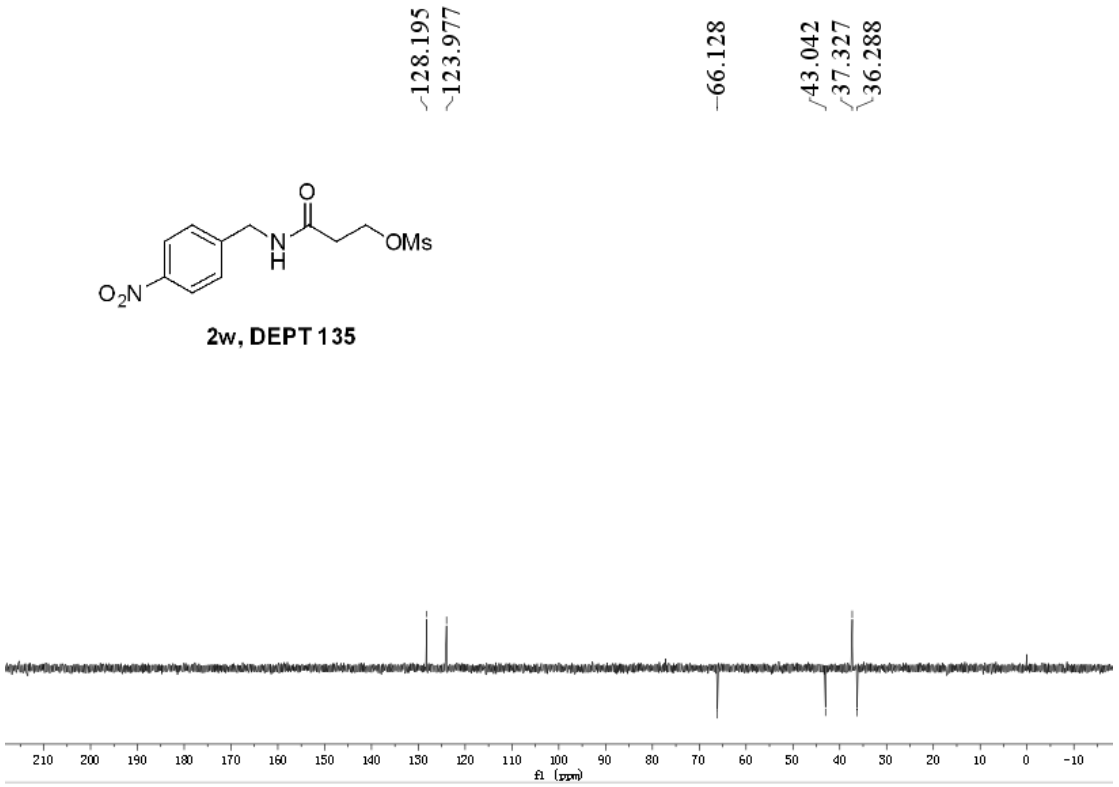

8.205
8.183
7.471
7.449
7.284
4.949
4.589
3.975
3.910
3.311
3.208
3.163
3.031
2.840
2.494
2.454
2.180
1.175
O
N
H
OMs
O2N
2w, 1H NMR
CH2Br2
1.87
2.00
2.58
2.44
0.24
2.32
4.49
Chemical Shift (ppm)



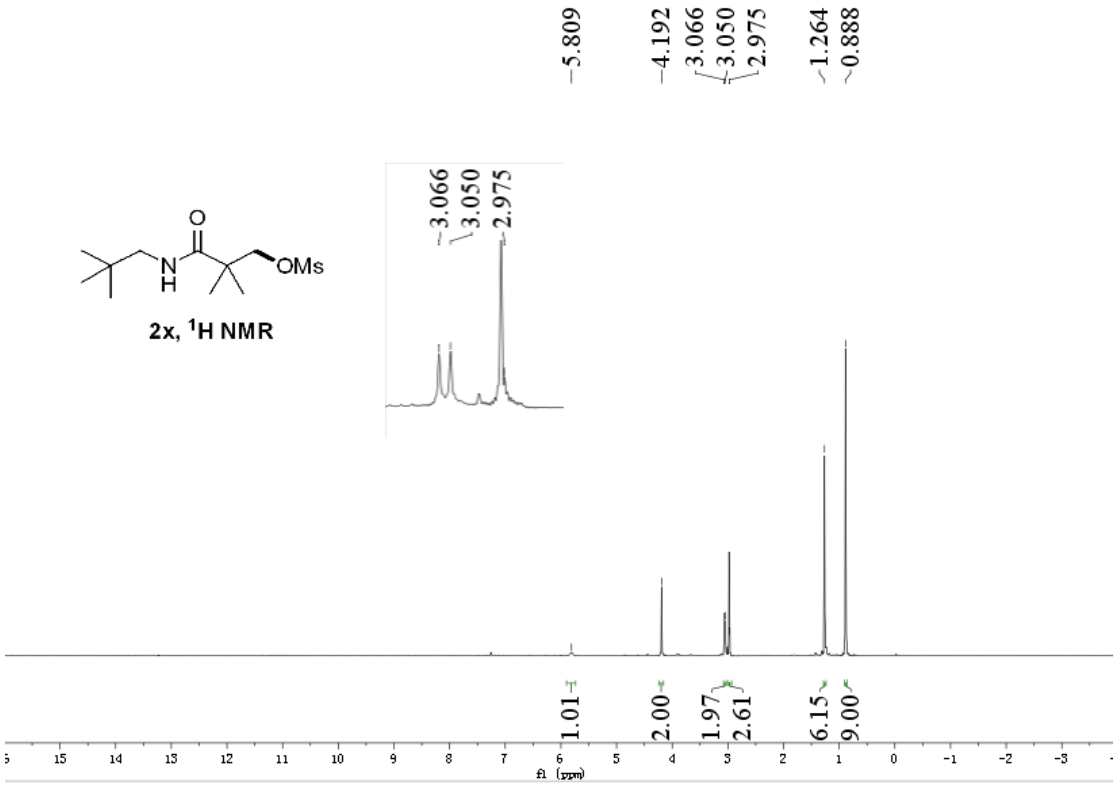
2w, ^{1}H NMR

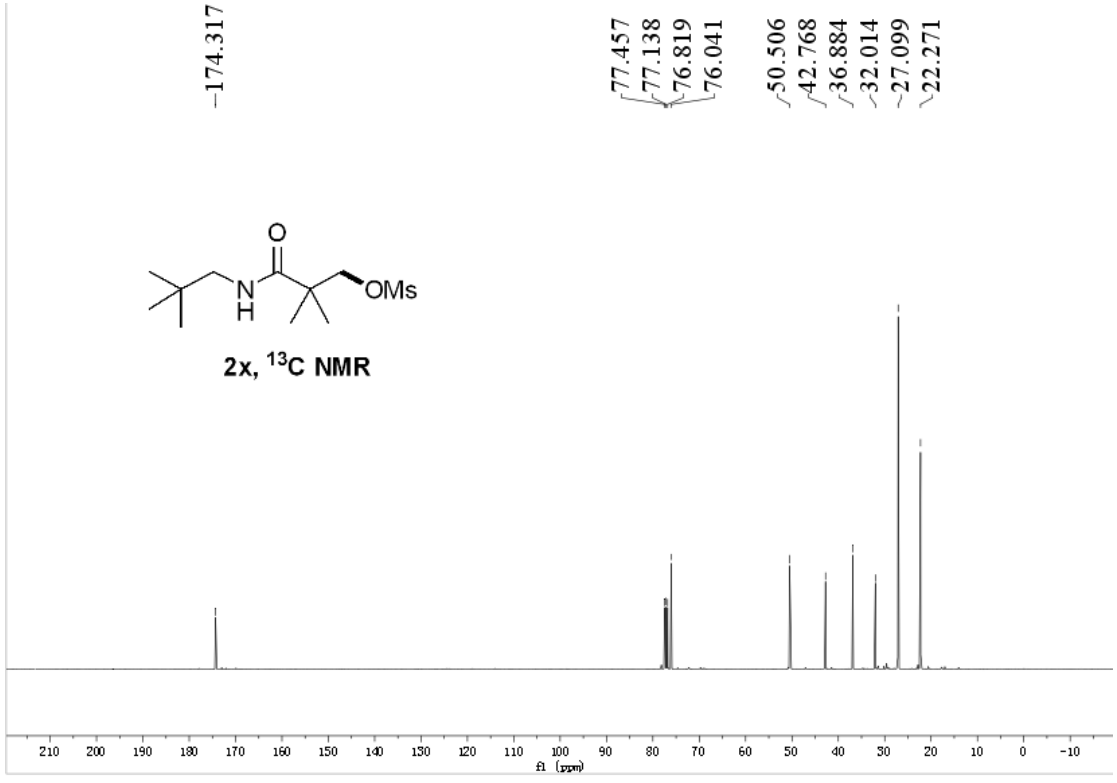


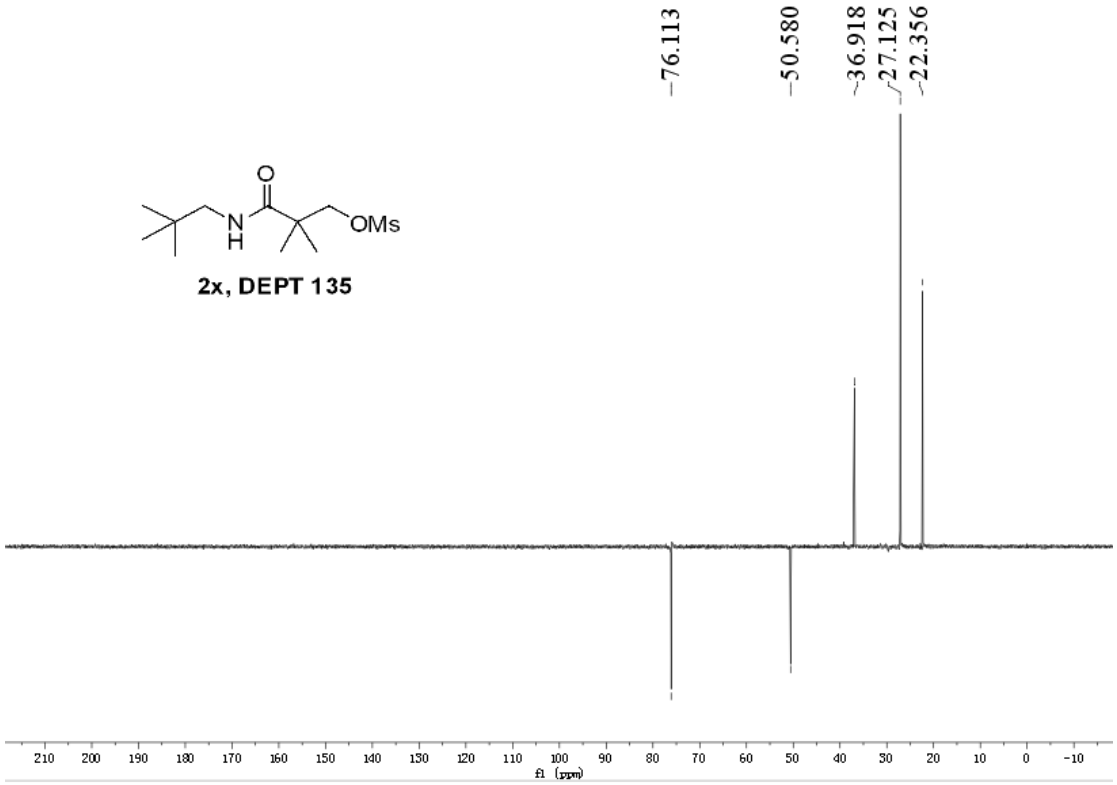
2w, ^{13}C NMR


128.195
123.977
66.128
43.042
37.327
36.288
O
N
H
OMs
O2N
2w, DEPT 135
7.284
6.182
4.942
4.336
4.186
3.946
3.881
3.145
2.999
1.308
1.283
0.942
0.912
CH2Br2
0.85
1.32
2.00
2.35
2.86
3.65
5.80
3.42
11.94
f1 (ppm)

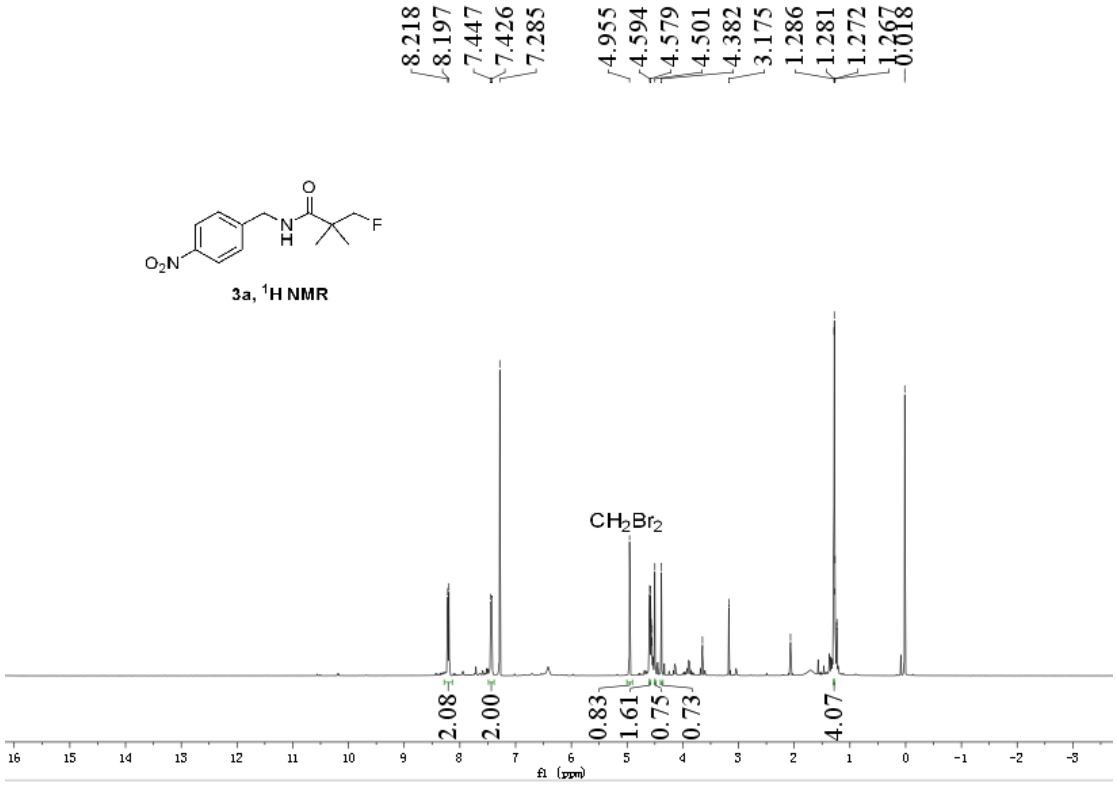
2x, ^{1}H NMR

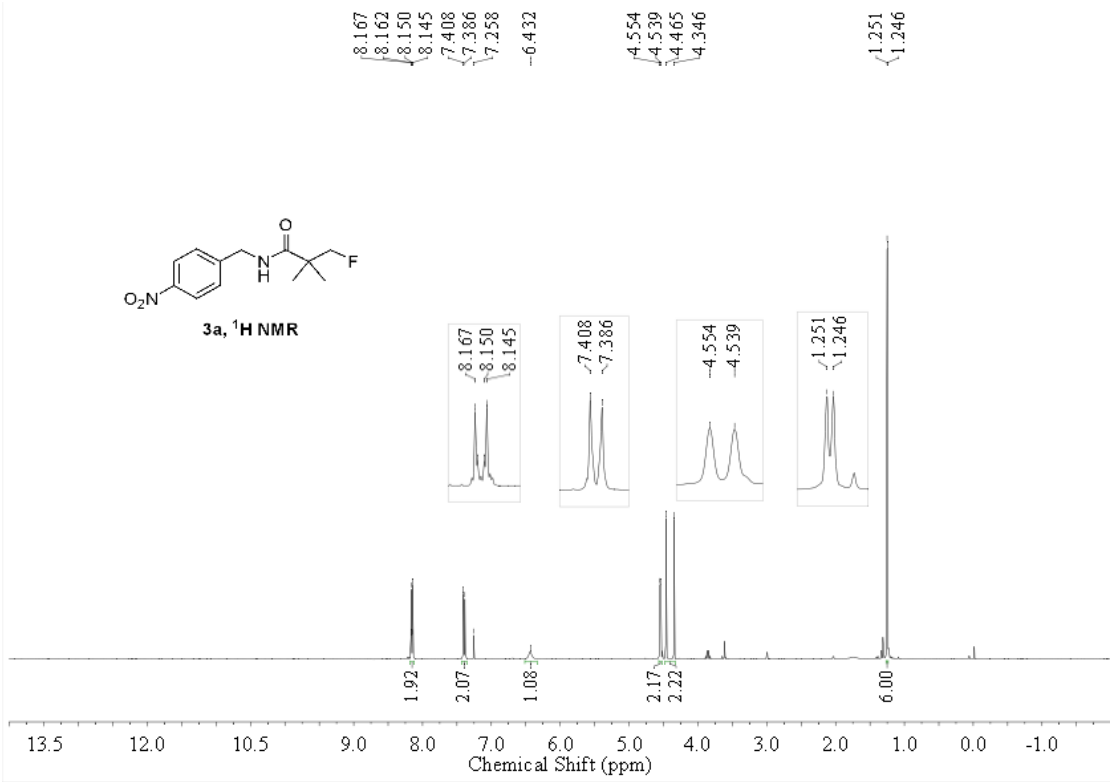

5.809
4.192
3.066
3.050
2.975
1.264
0.888
OMs
2x, 1H NMR
3.066
3.050
2.975
1.01
2.00
1.97
2.61
6.15
9.00
f1 (ppm)

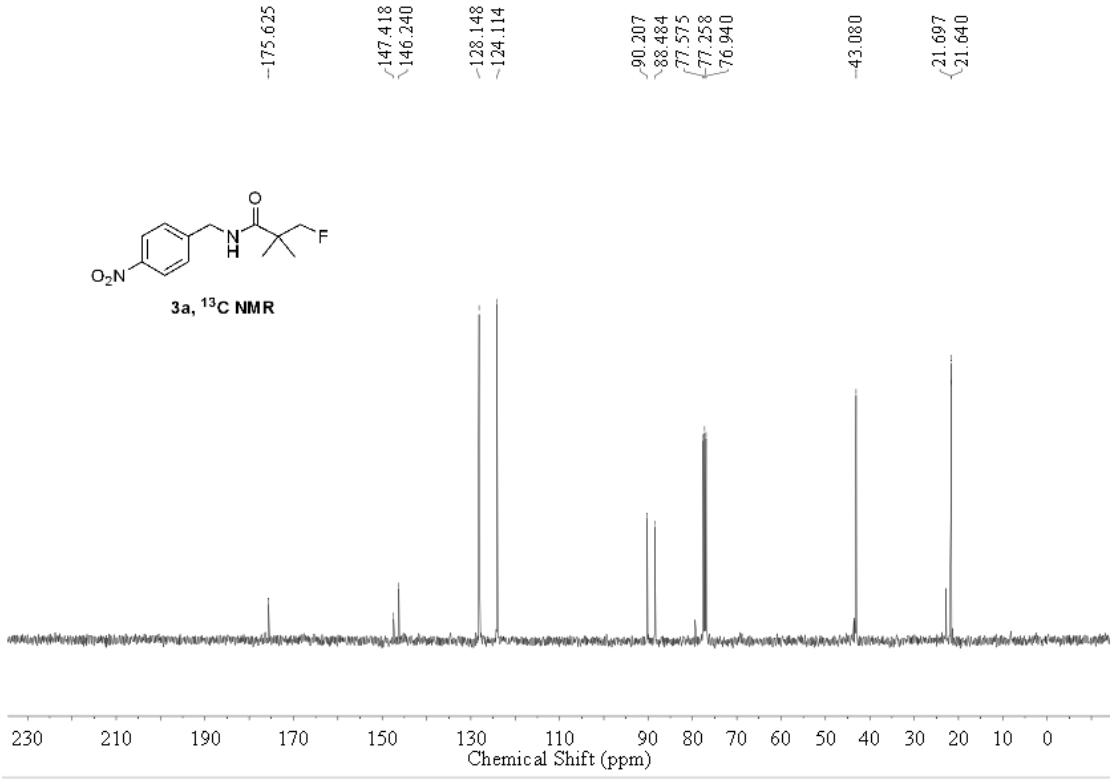

174.317
77.457
77.138
76.819
76.041
50.506
42.768
36.884
32.014
27.099
22.271
OMs
2x, 13C NMR
f1 (ppm)

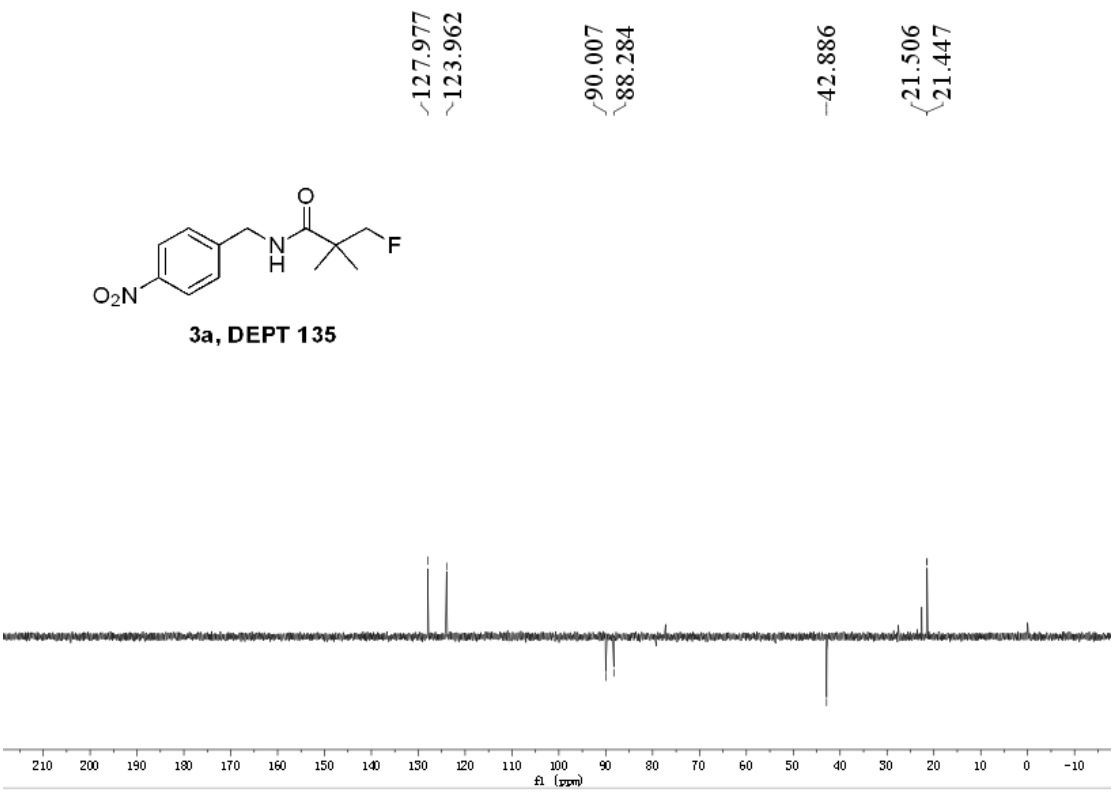


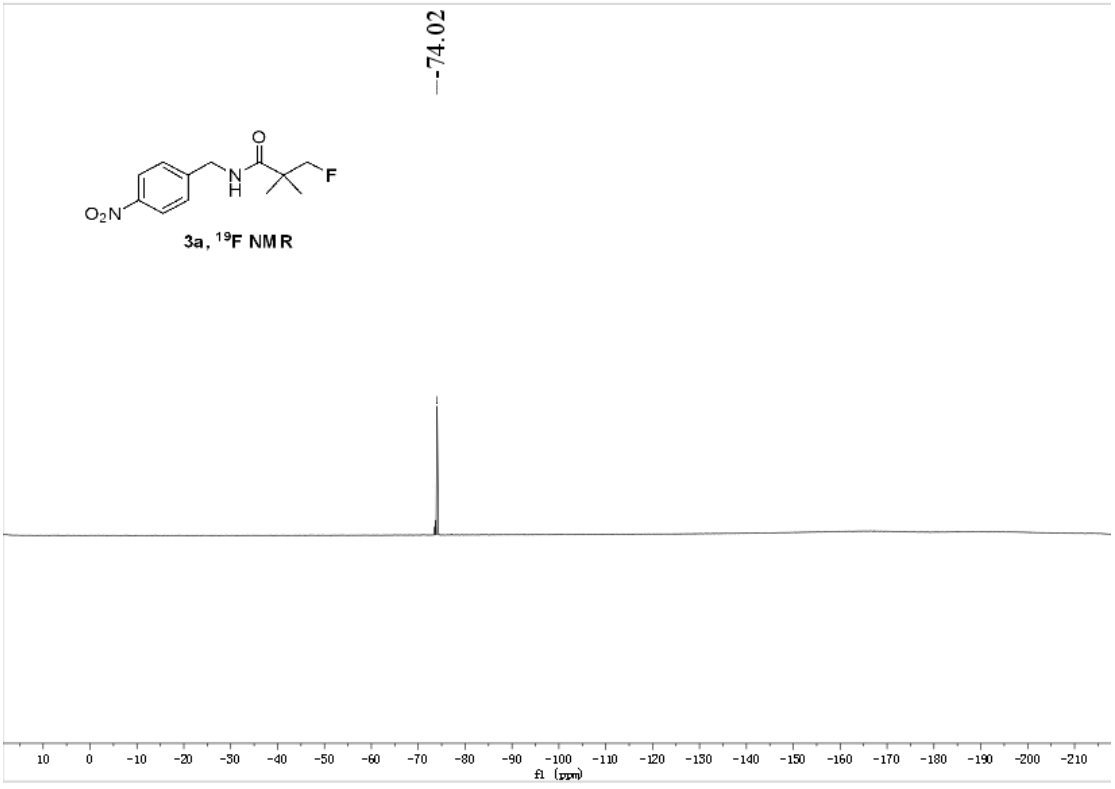
(3) *β*-Fluoramides

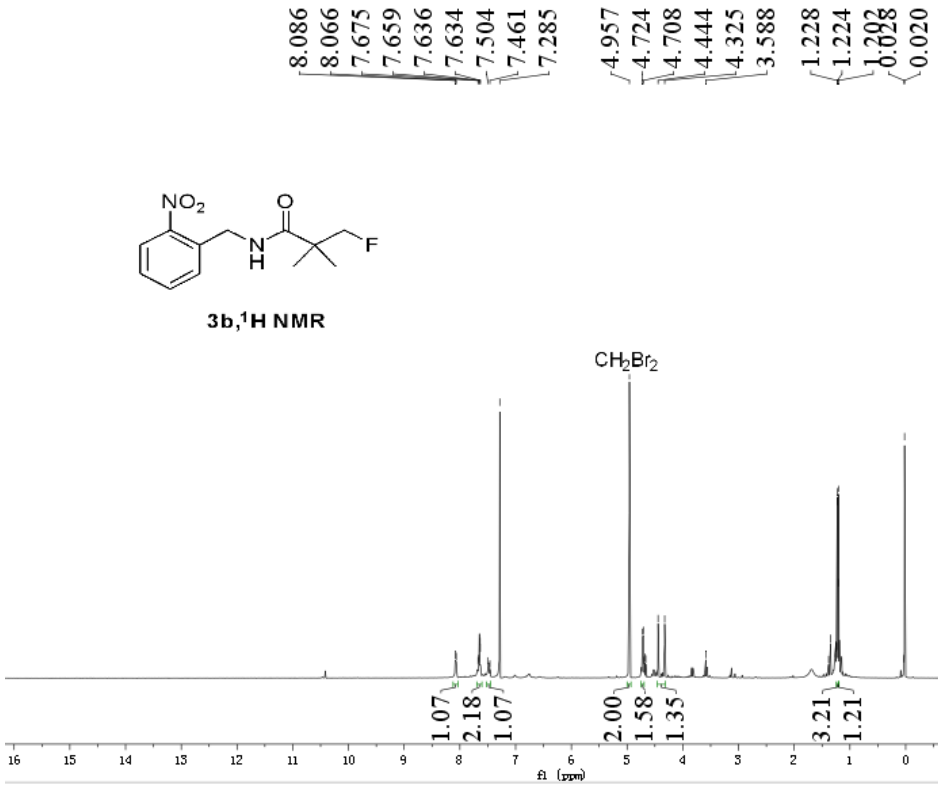


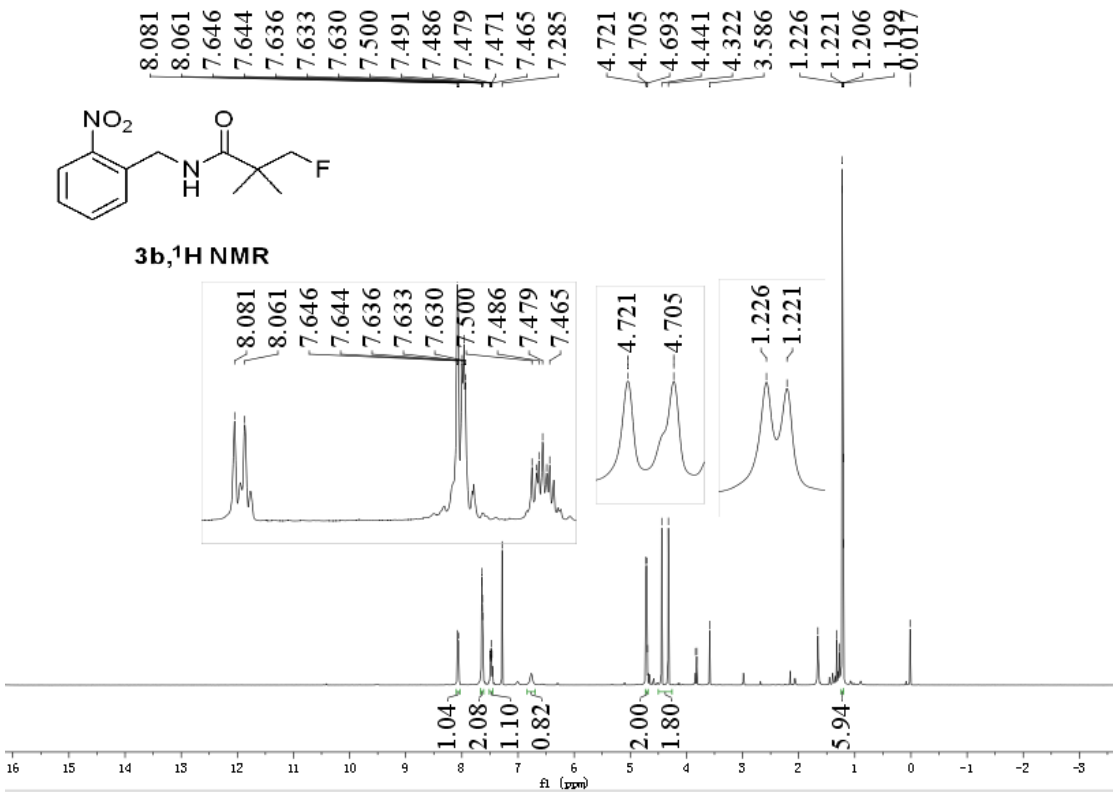

8.167
8.162
8.150
8.145
7.408
7.386
7.258
6.432
4.554
4.539
4.465
4.346
1.251
1.246
O
N
H
F
O2N
3a, 1H NMR
1.92
2.07
1.08
2.17
2.22
6.00
13.5
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
Chemical Shift (ppm)

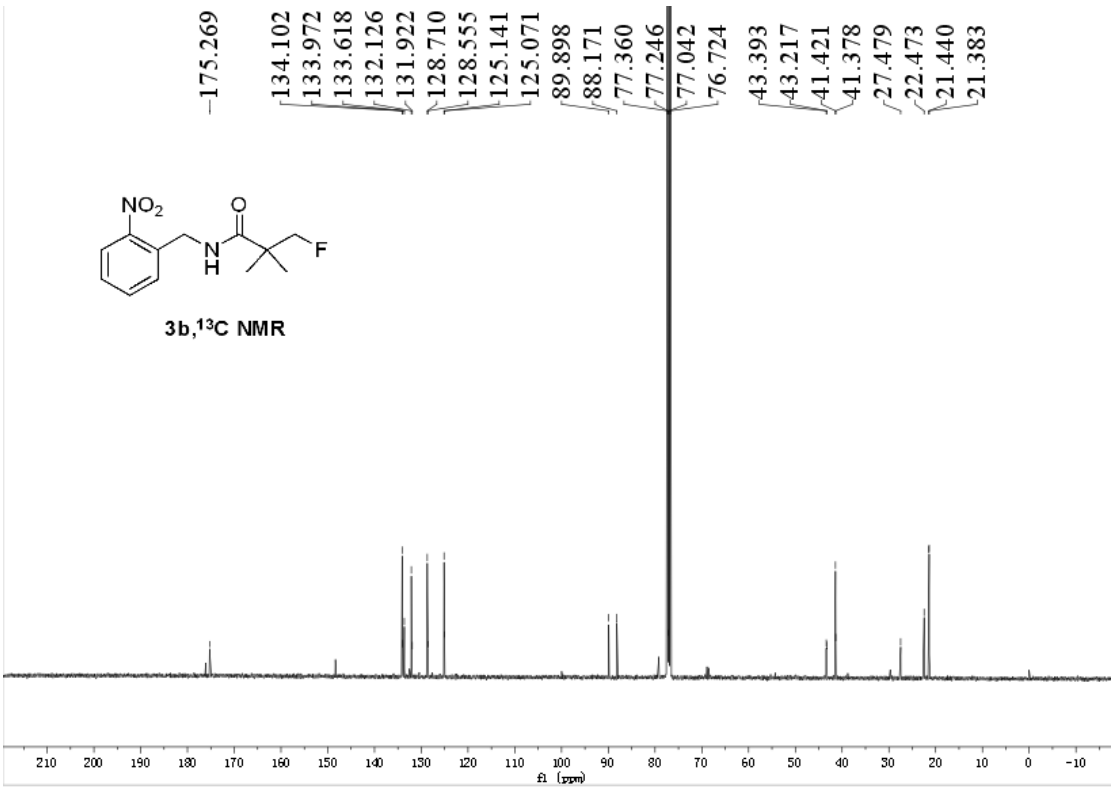

175.625
147.418
146.240
128.148
124.114
90.207
88.484
77.575
77.258
76.940
43.080
21.697
21.640
O
N
H
F
O2N
3a, 13C NMR
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)


127.977
123.962
90.007
88.284
42.886
21.506
21.447
O
N
H
F
O2N
3a, DEPT 135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

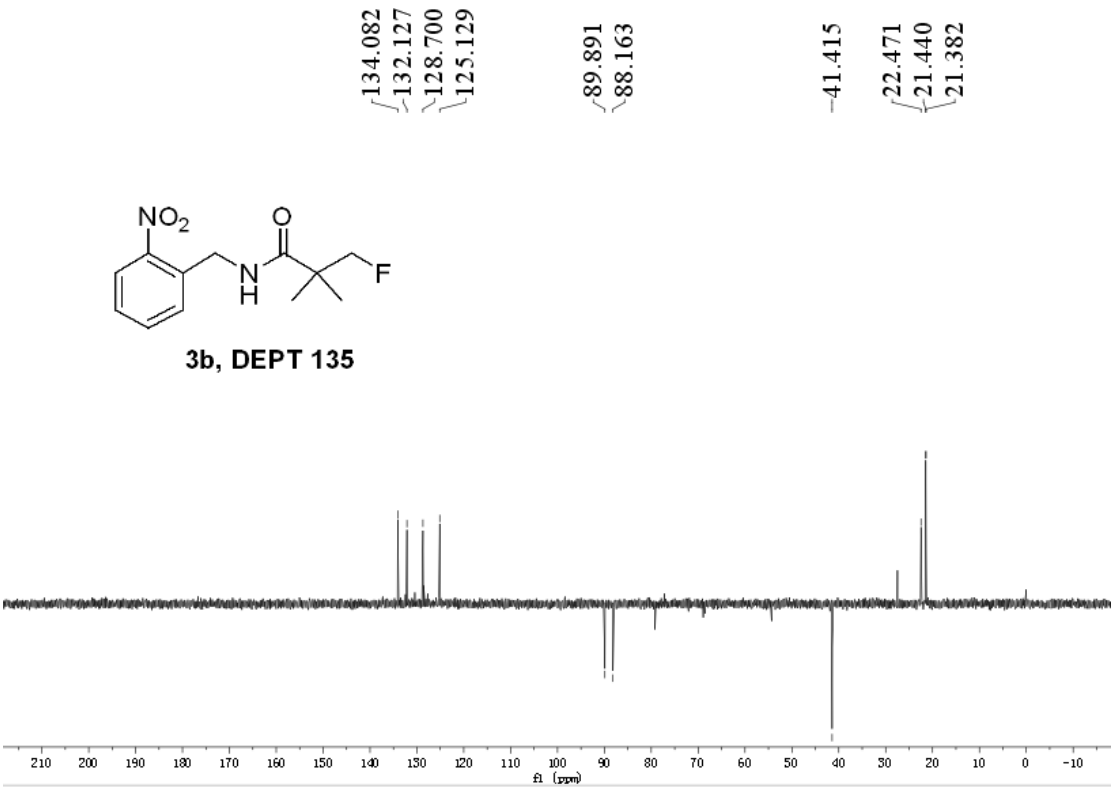

-74.02
O
N
H
F
O2N
3a, 19F NMR
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


8.086
8.066
7.675
7.659
7.636
7.634
7.504
7.461
7.285
4.957
4.724
4.708
4.444
4.325
3.588
1.228
1.224
1.202
0.028
0.020
NO2
O
N
H
F
3b,1H NMR
CH2Br2
1.07
2.18
1.07
2.00
1.58
1.35
3.21
1.21
f1 (ppm)

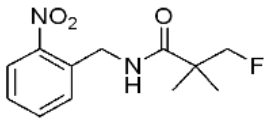

8.081
8.061
7.646
7.644
7.636
7.633
7.630
7.500
7.491
7.486
7.479
7.471
7.465
7.285
4.721
4.705
4.693
4.441
4.322
3.586
1.226
1.221
1.206
1.192
0.017
NO2
O
N
H
F
3b,1H NMR
1.04
2.08
1.10
0.82
2.00
1.80
5.94
f1 (ppm)



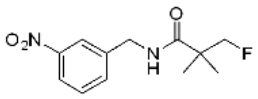
3b,^{13}C NMR



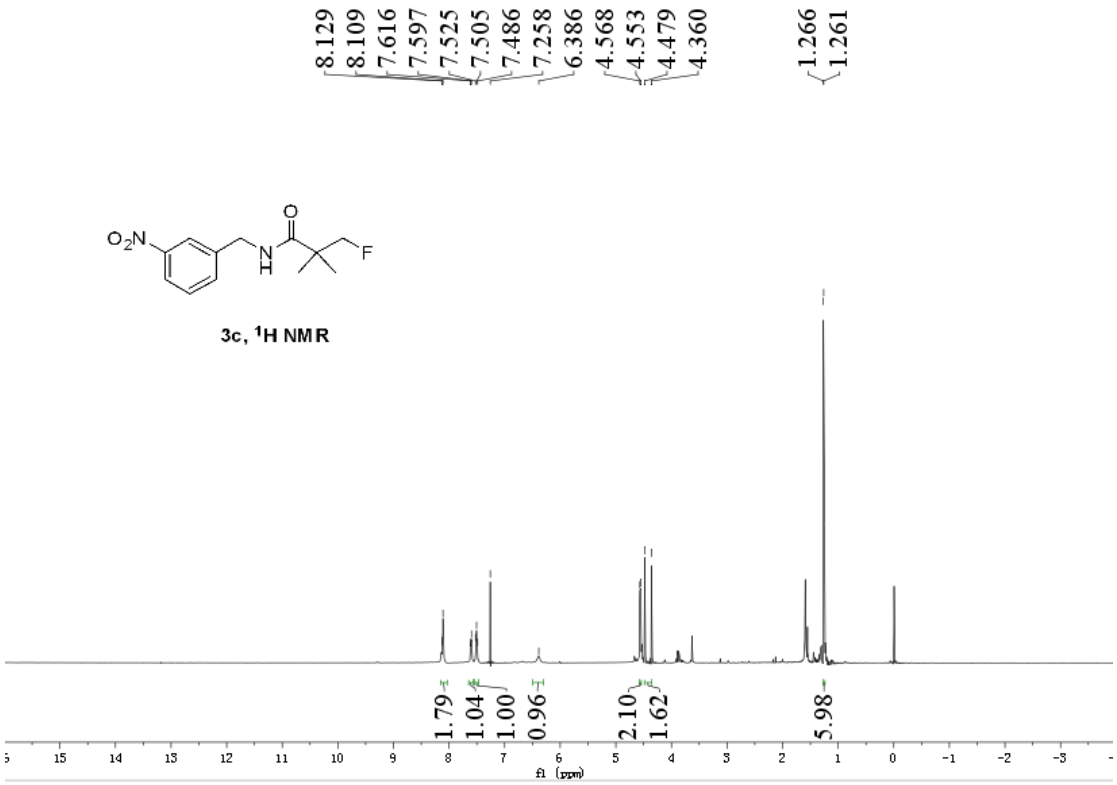
3b, DEPT 135



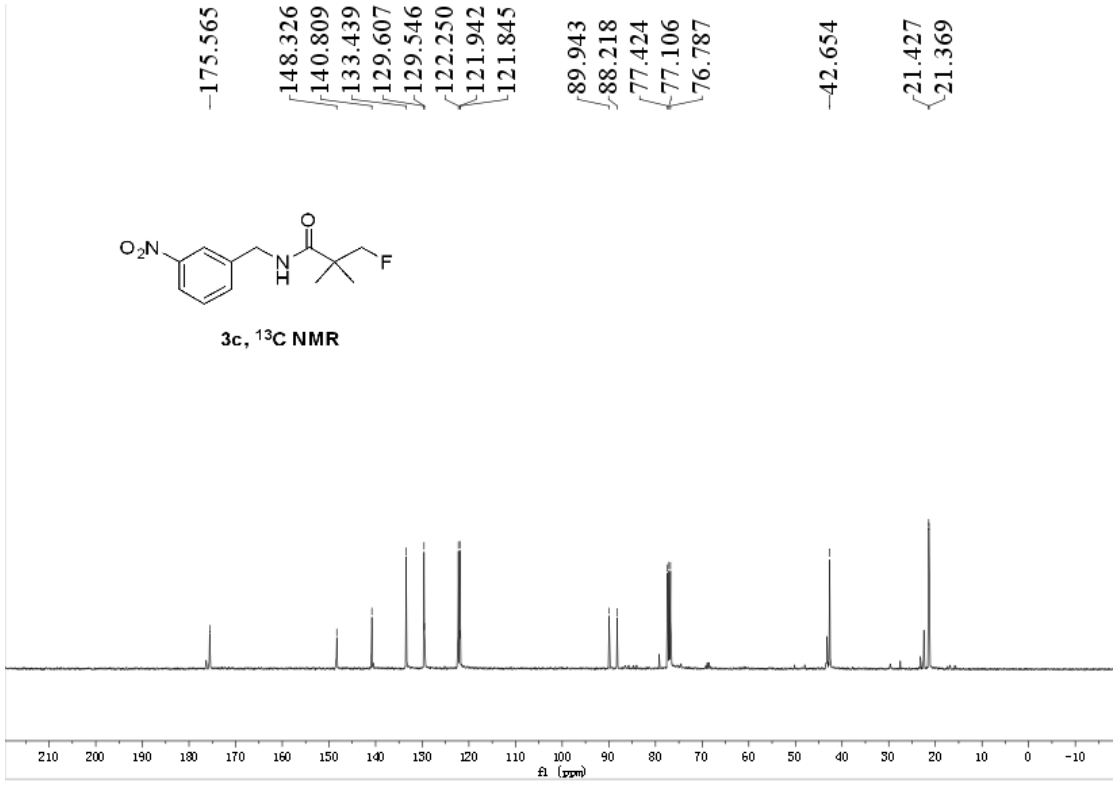
3b,^{19}F NMR



3c, ^{1}H NMR

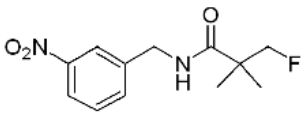


3c, ^{1}H NMR

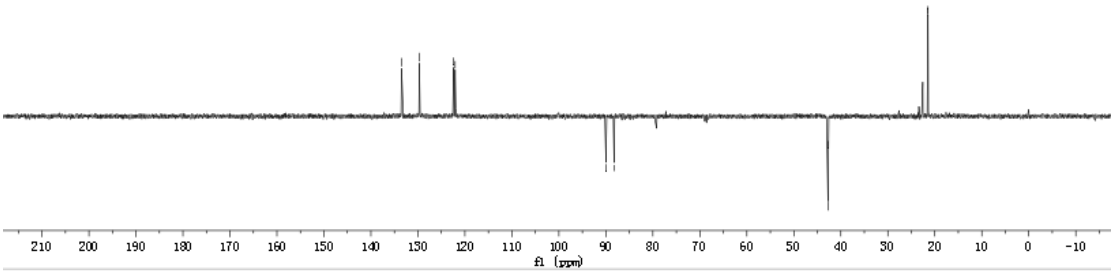


3c, ^{13}C NMR

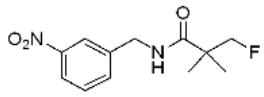
133.475
129.691
122.421
122.066
89.981
88.257
42.733
42.661
21.508
21.450



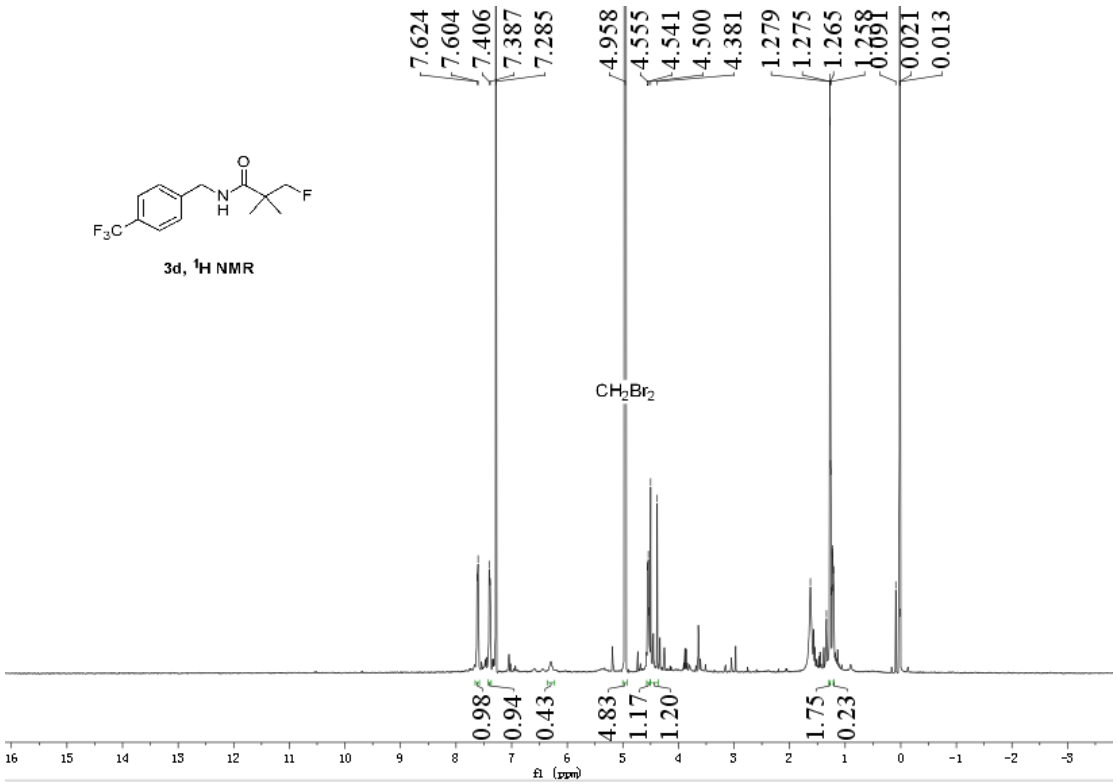
3c, DEPT 135



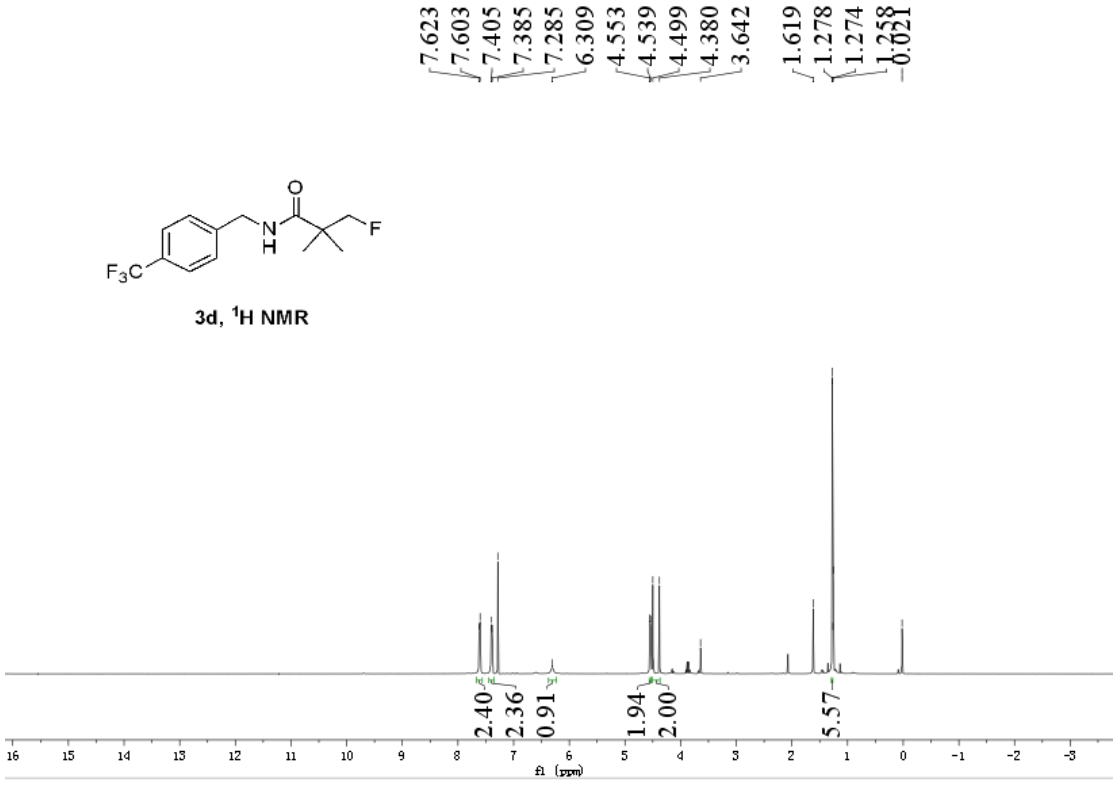
-74.01



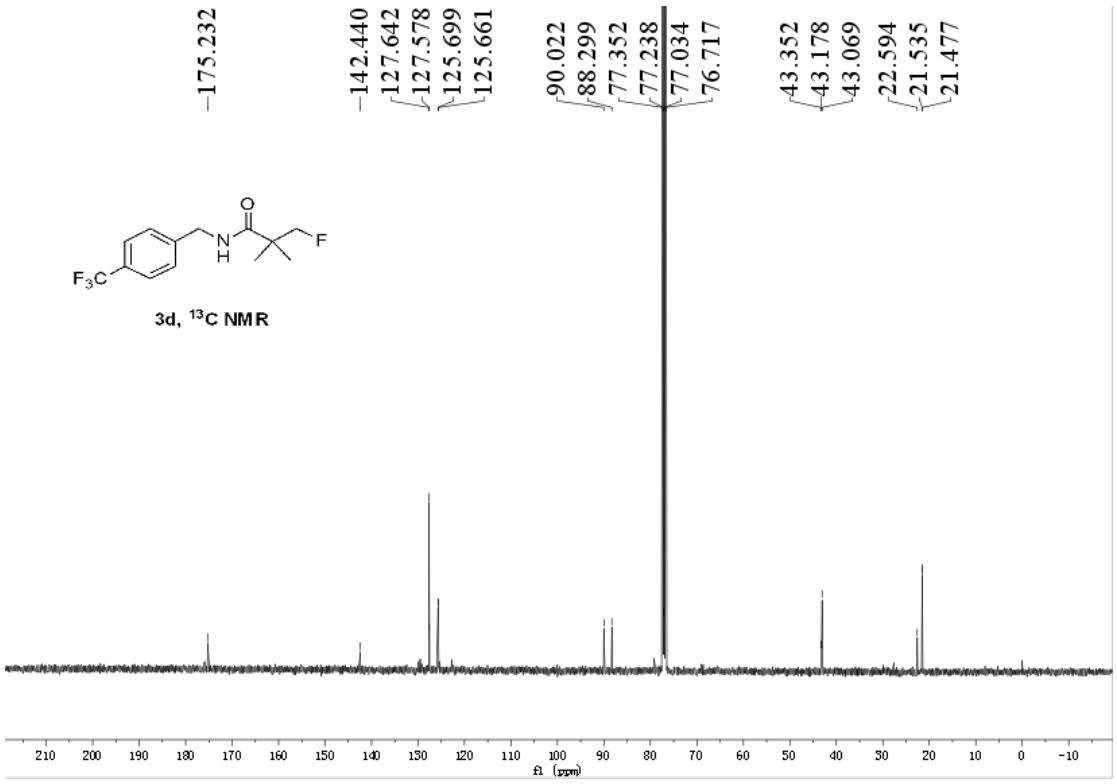
3c, ^{19}F NMR



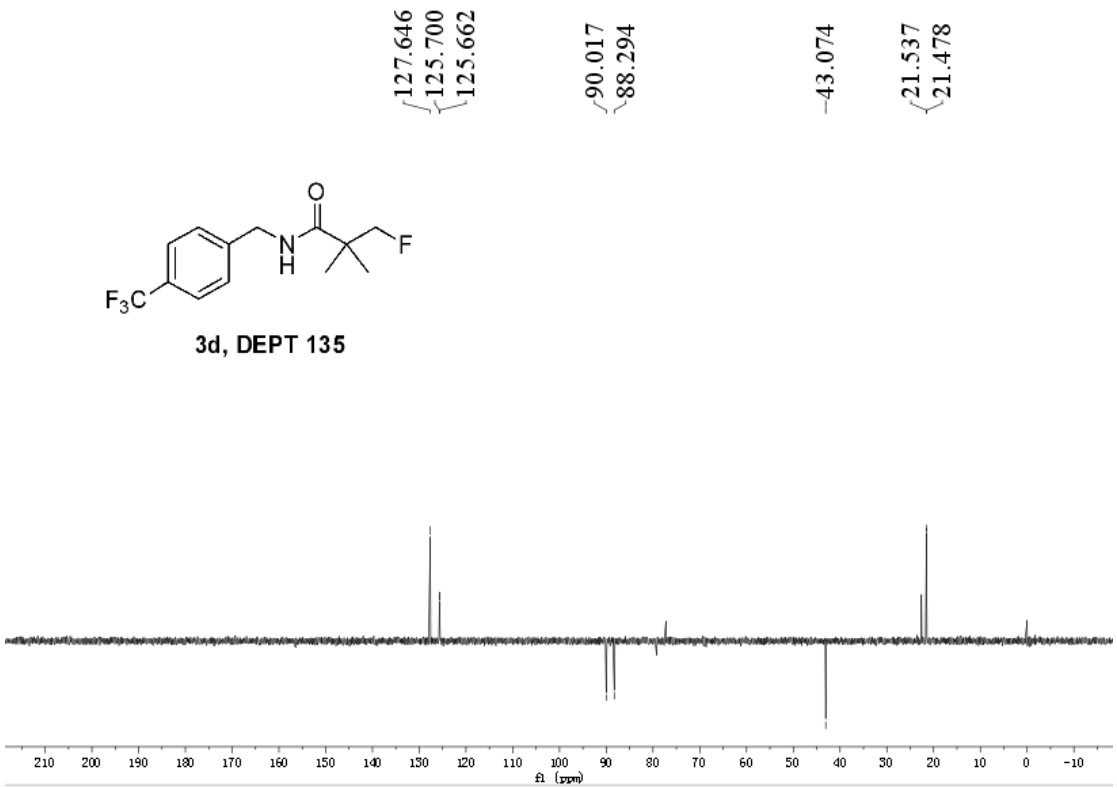
3d, ^{1}H NMR



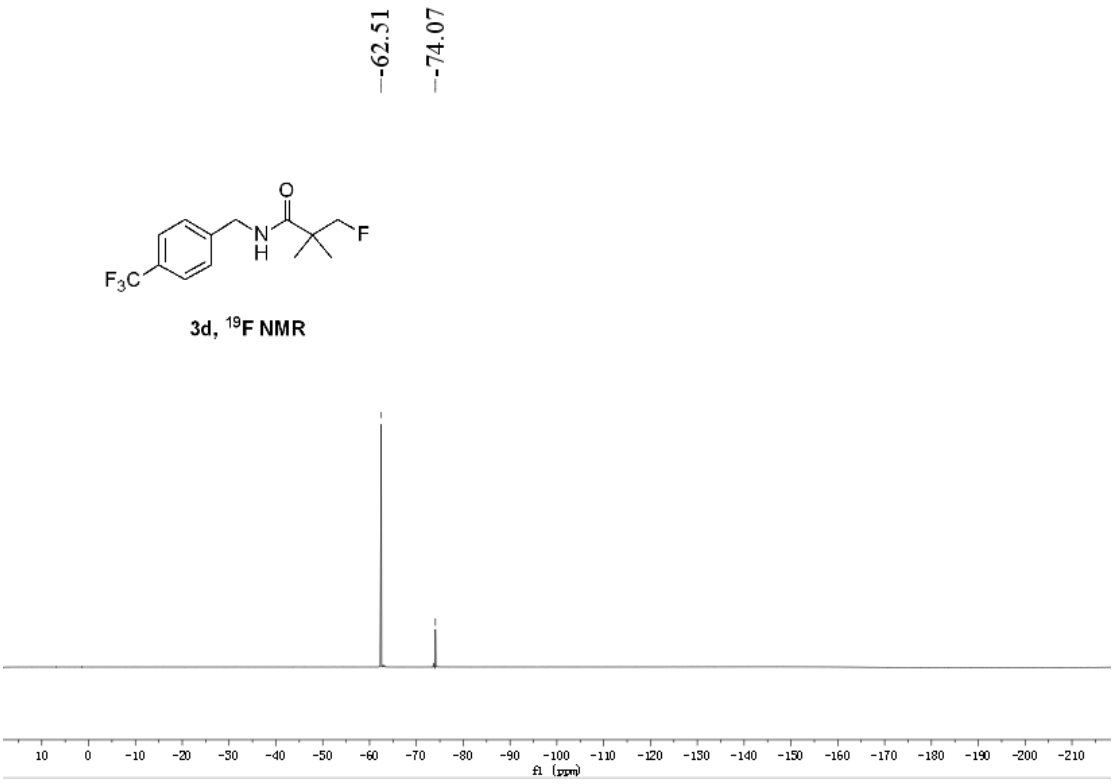
3d, ^{1}H NMR



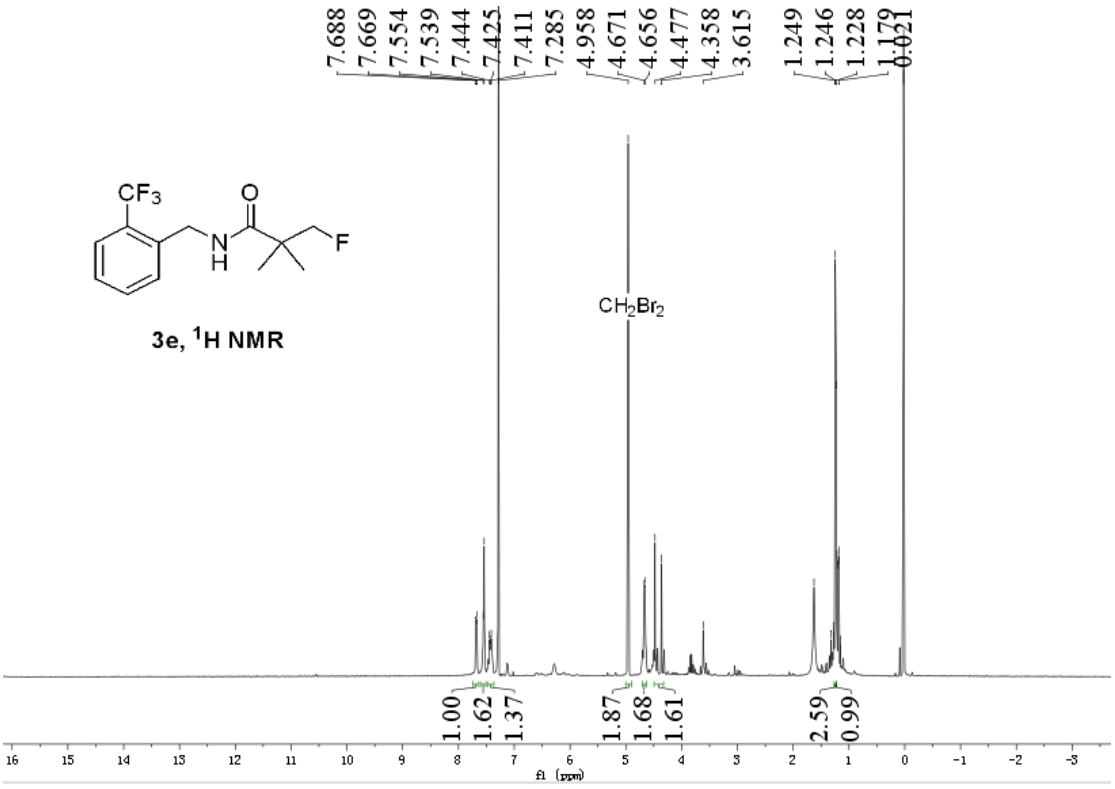
3d, ^{13}C NMR



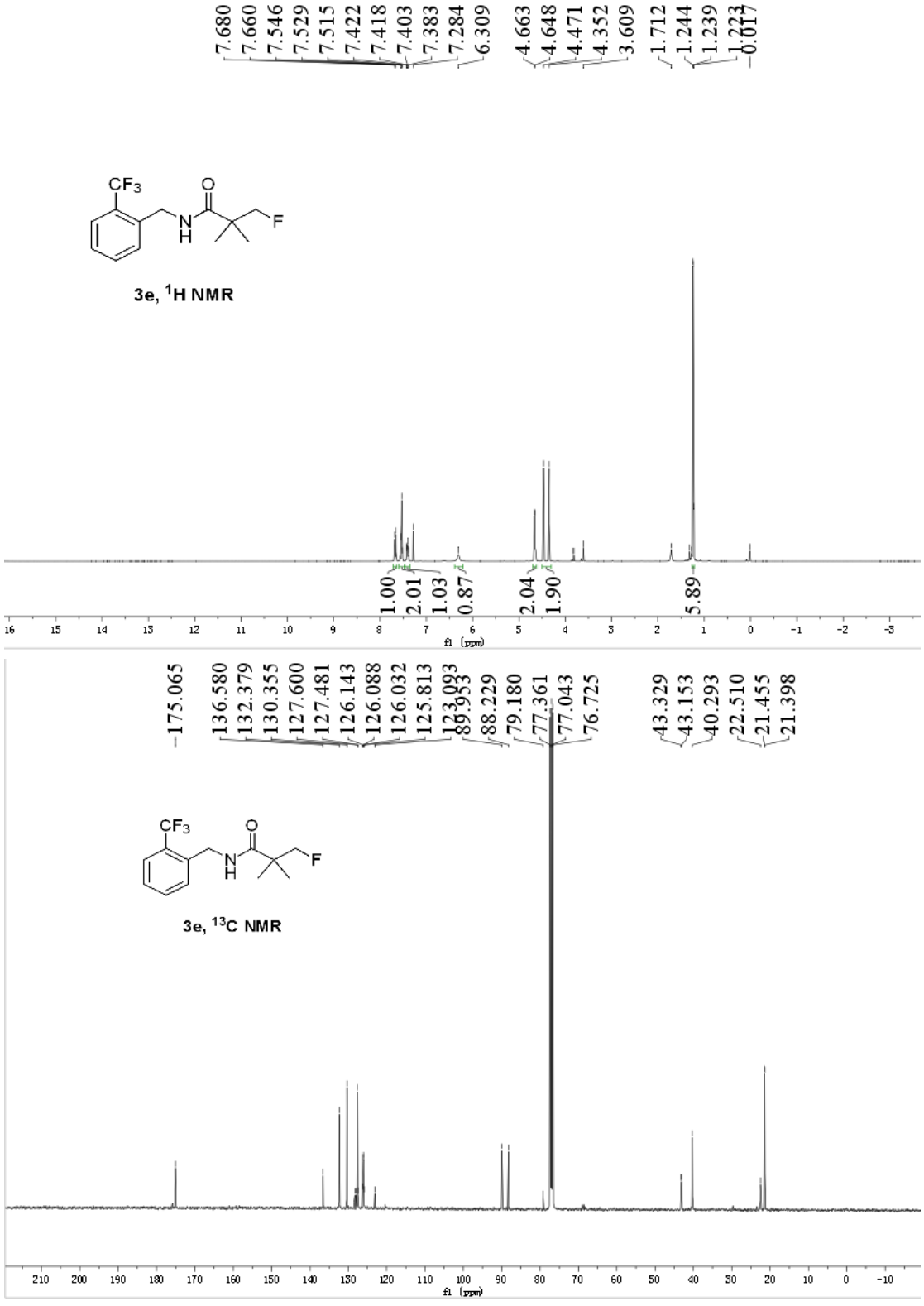
3d, DEPT 135

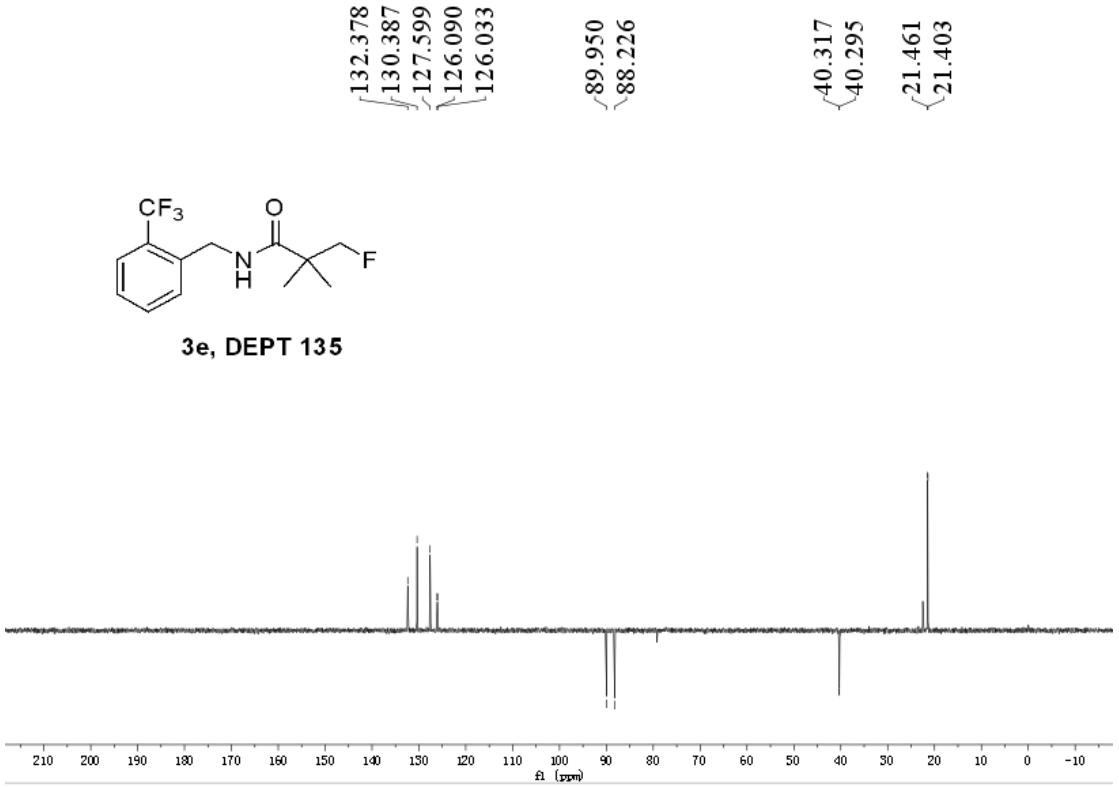


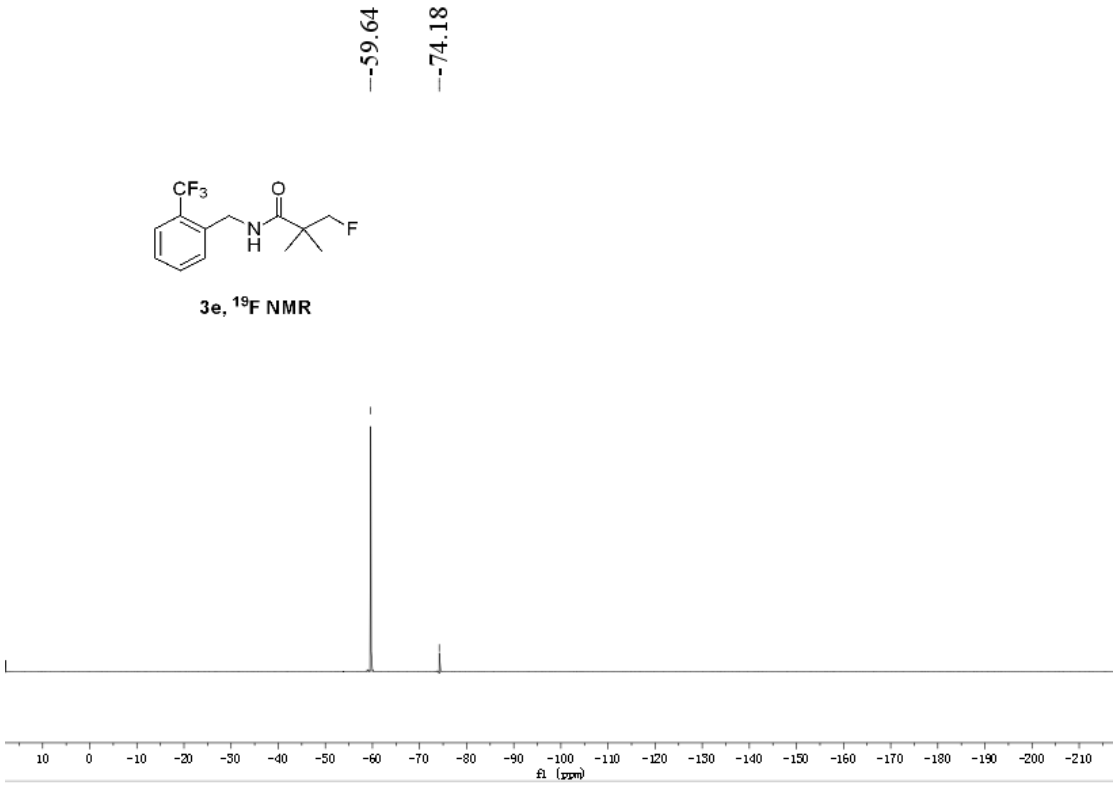
3d, ^{19}F NMR

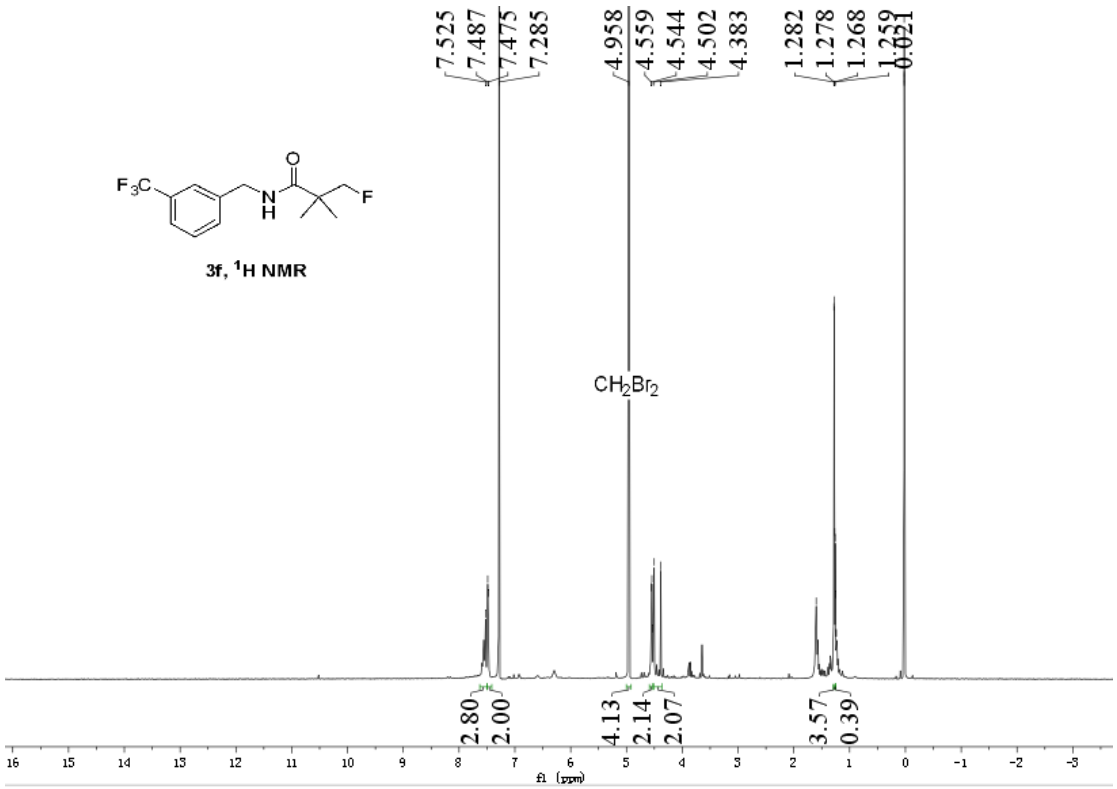


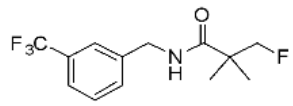
3e, ^{1}H NMR


7.680
7.660
7.546
7.529
7.515
7.422
7.418
7.403
7.383
7.284
6.309
4.663
4.648
4.471
4.352
3.609
1.712
1.244
1.239
1.223
0.017
CF3
O
N
H
F
3e, 1H NMR
1.00
2.01
1.03
0.87
2.04
1.90
5.89
f1 (ppm)
175.065
136.580
132.379
130.355
127.600
127.481
126.143
126.088
126.032
125.813
123.093
89.953
88.229
79.180
77.361
77.043
76.725
43.329
43.153
40.293
22.510
21.455
21.398
CF3
O
N
H
F
3e, 13C NMR
f1 (ppm)


132.378
130.387
127.599
126.090
126.033
89.950
88.226
40.317
40.295
21.461
21.403
CF3
O
N
H
F
3e, DEPT 135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

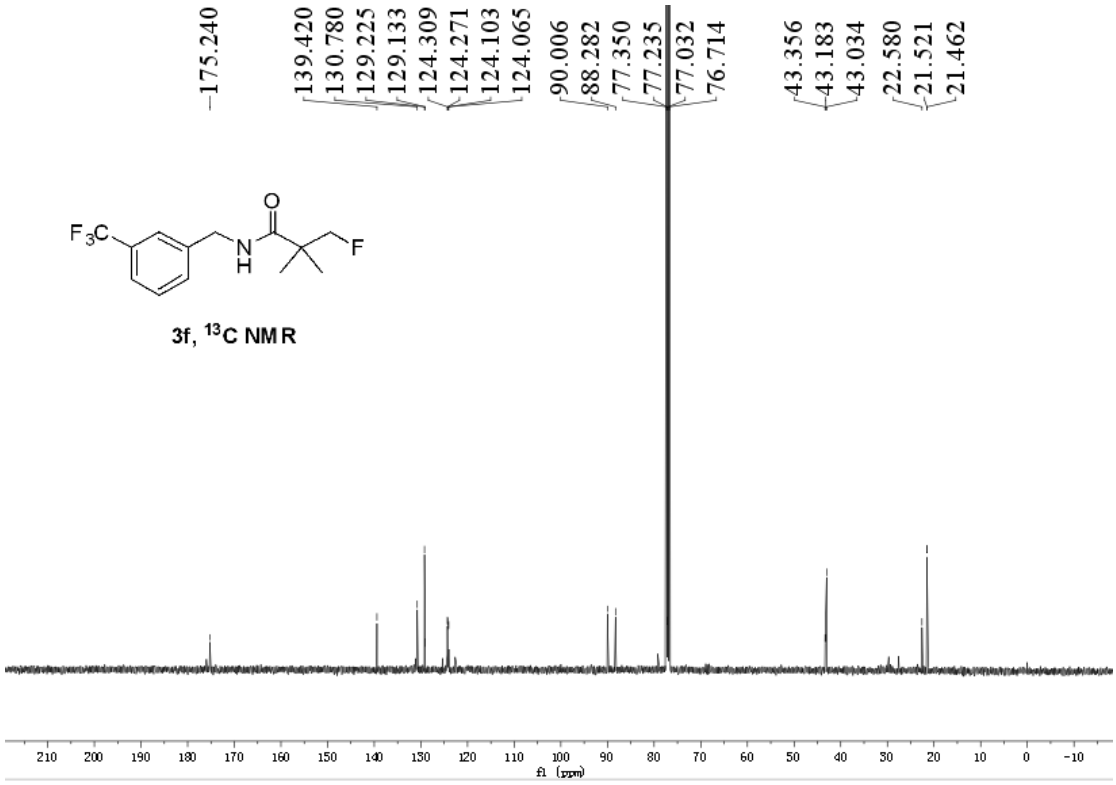

-59.64
-74.18
CF3
O
N
H
F
3e, 19F NMR
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


7.525
7.487
7.475
7.285
4.958
4.559
4.544
4.502
4.383
1.282
1.278
1.268
1.259
0.021
CH2Br2
2.80
2.00
4.13
2.14
2.07
3.57
0.39
f1 (ppm)

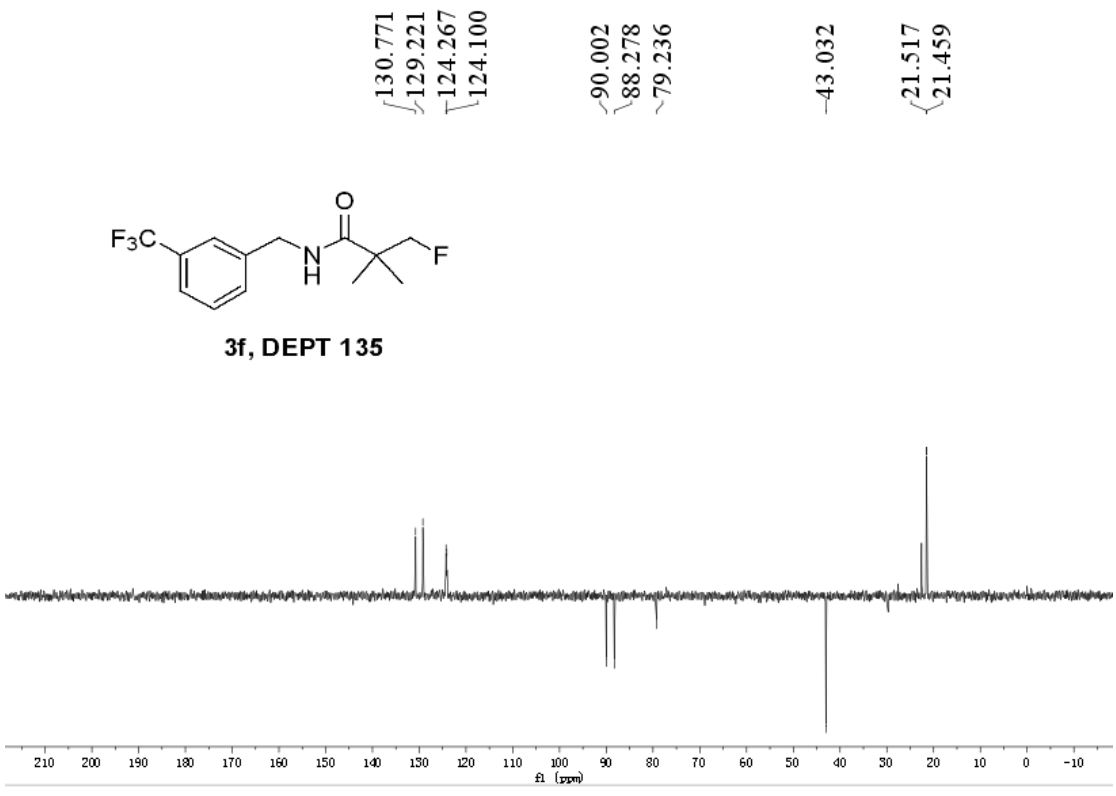


3f, ^{1}H NMR

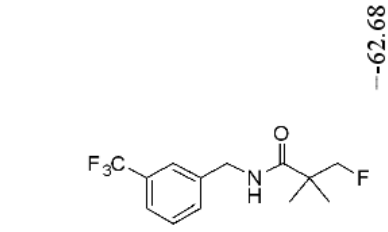
7.563
7.557
7.554
7.543
7.523
7.502
7.484
7.472
7.285
6.309
4.555
4.540
4.499
4.380
1.279
1.275
2.54
2.35
1.00
1.94
2.07
6.51
f1 (ppm)



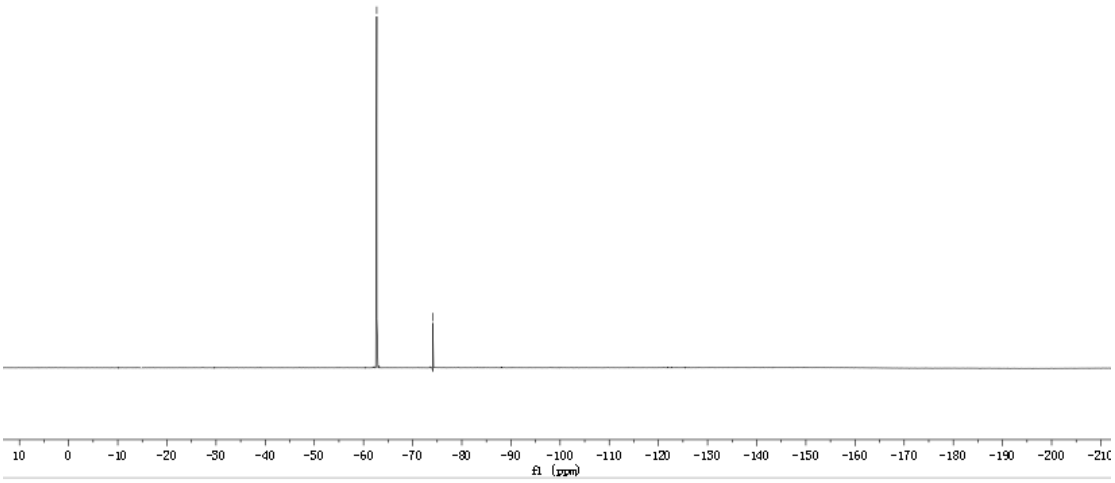
3f, ^{13}C NMR

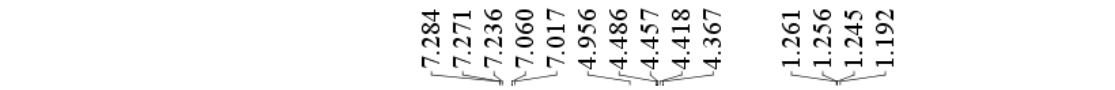


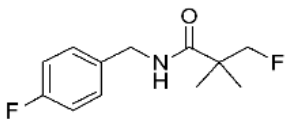
3f, DEPT 135



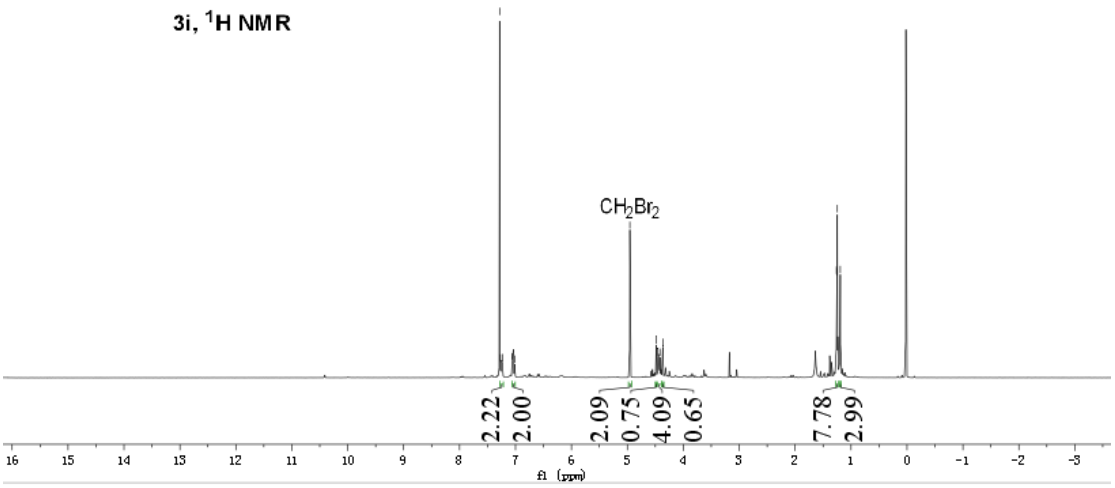
3f, ^{19}F NMR

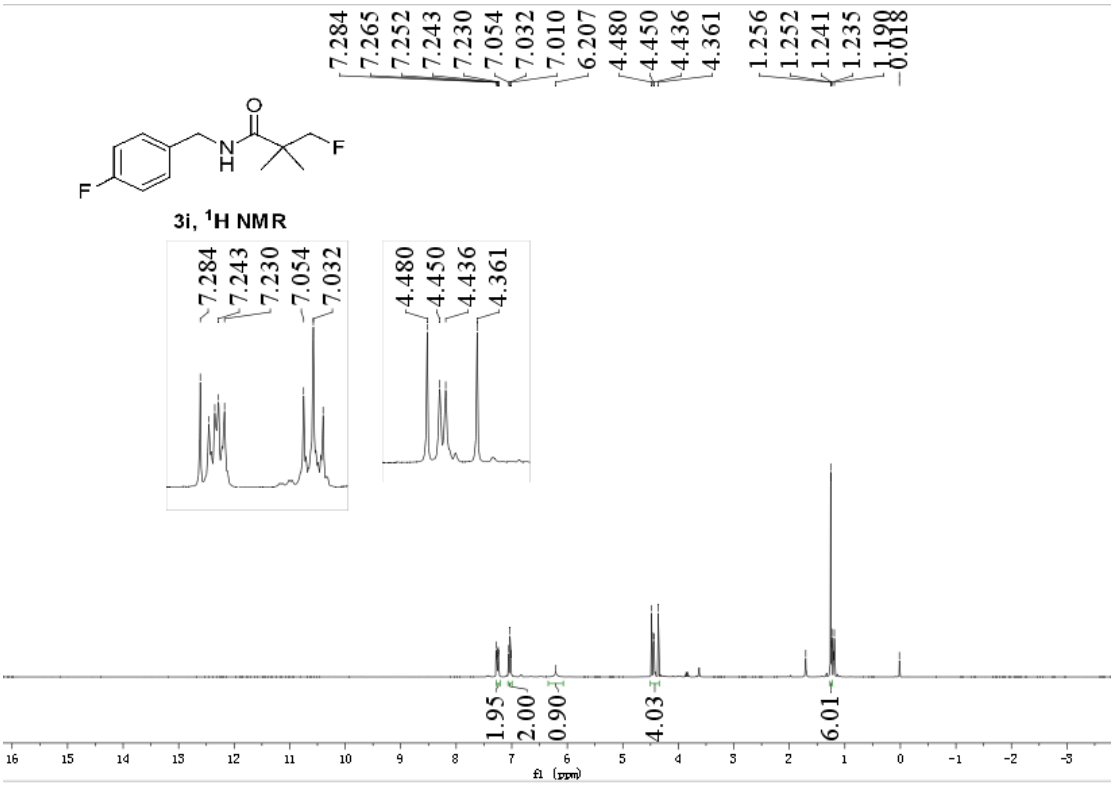




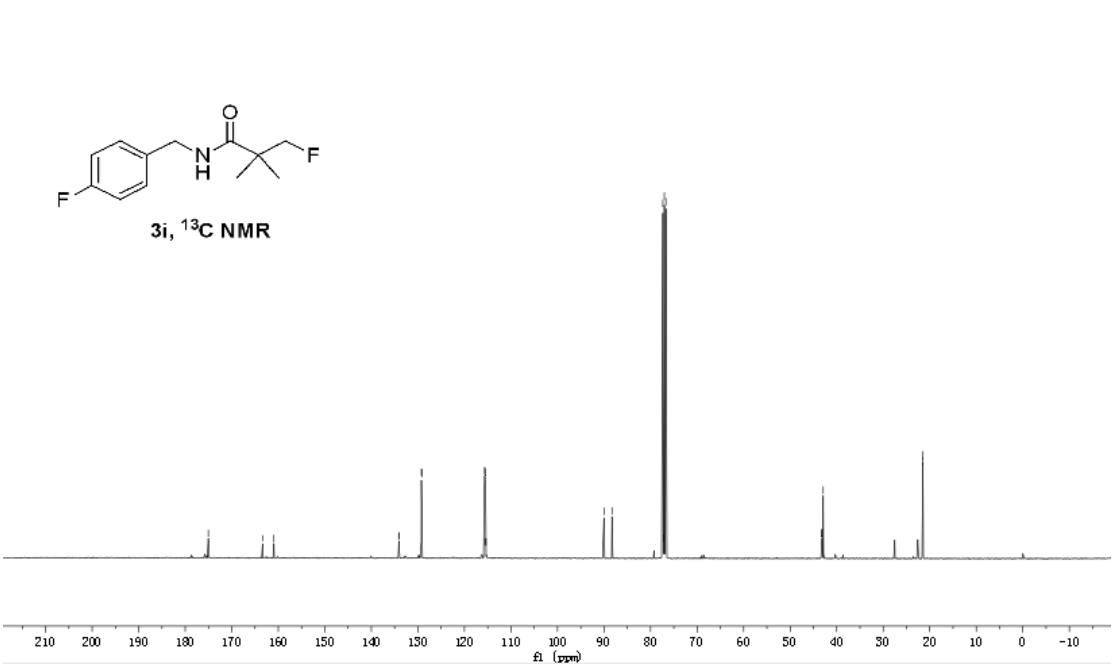


3i, ^{1}H NMR



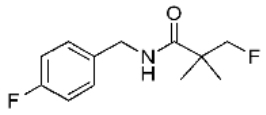


3i, ^{1}H NMR

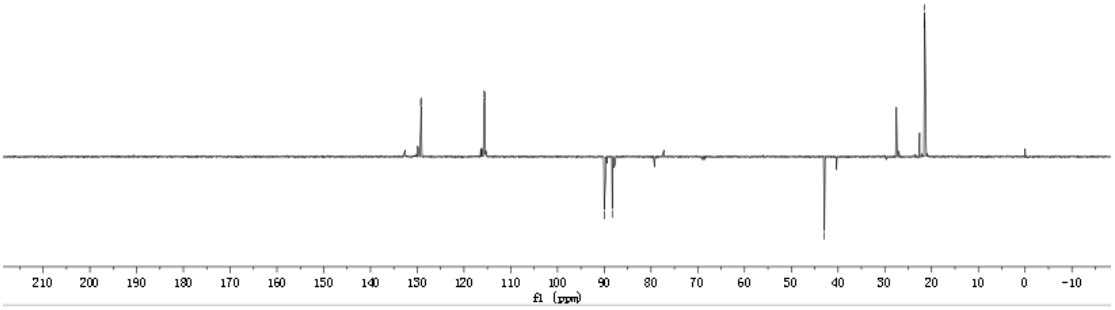


3i, ^{13}C NMR

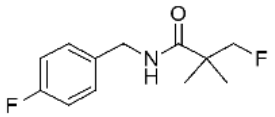
129.256
129.176
115.686
115.472
90.014
88.290
42.916
21.530
21.472



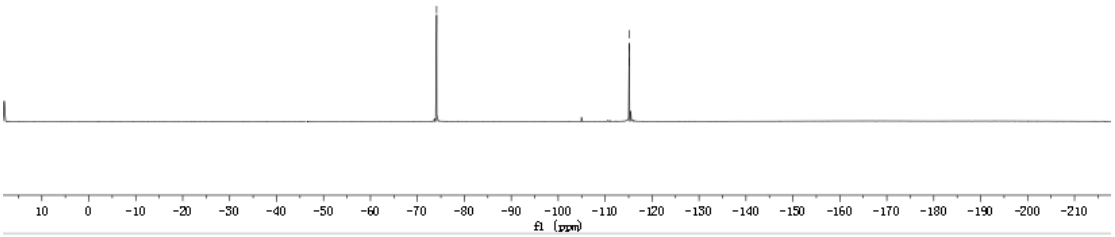
3i, DEPT 135

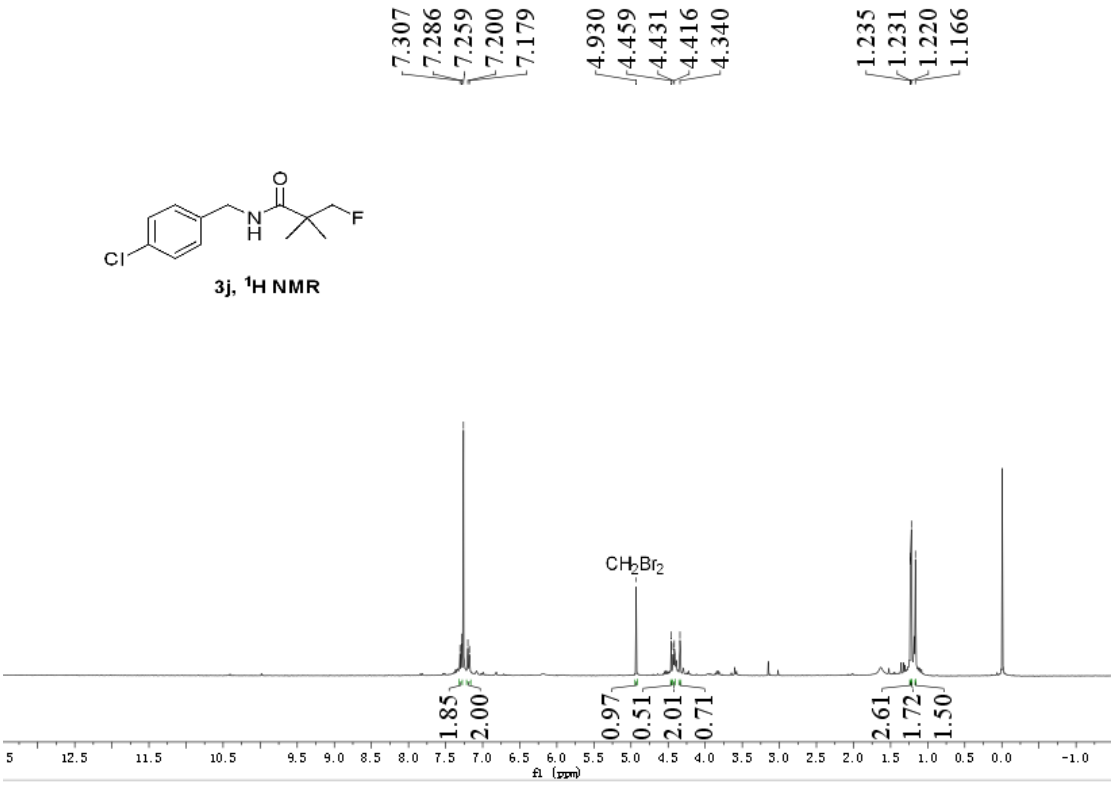


-74.09
-115.13
-115.13
-115.14

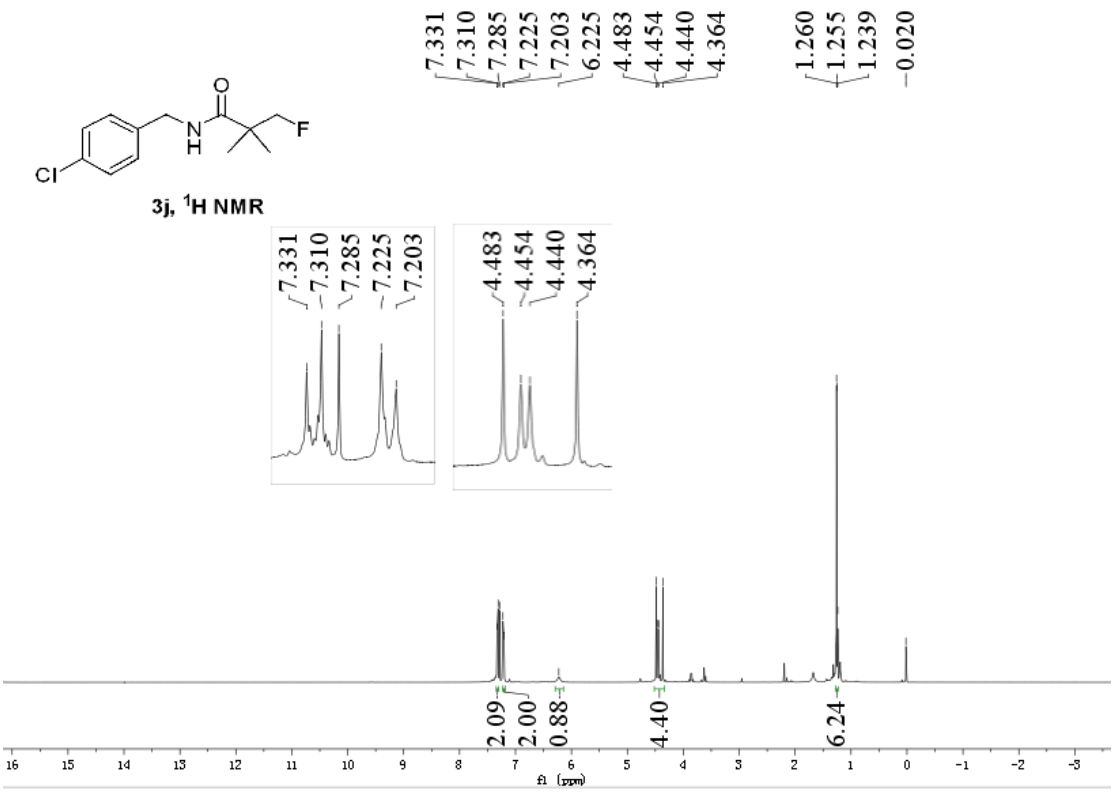


3i, ^{19}F NMR

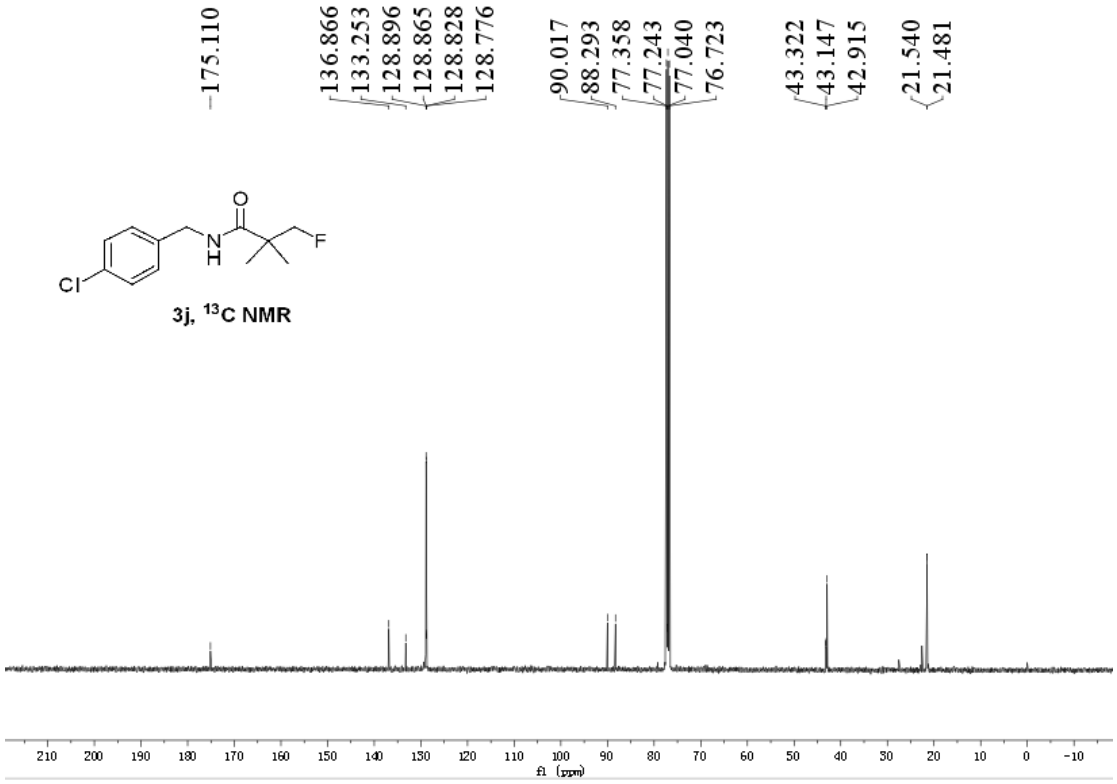




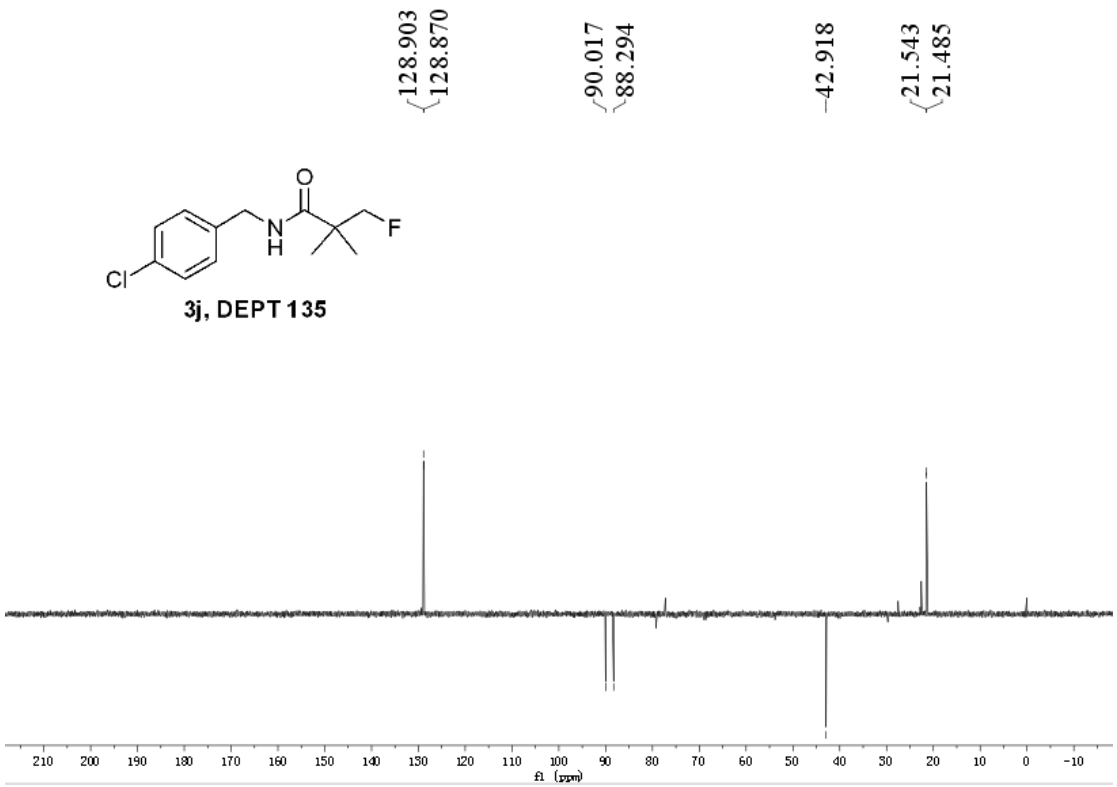
3j, ^{1}H NMR



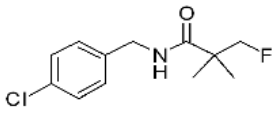
3j, ^{1}H NMR



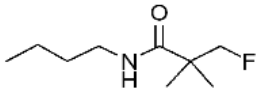
3j, ^{13}C NMR



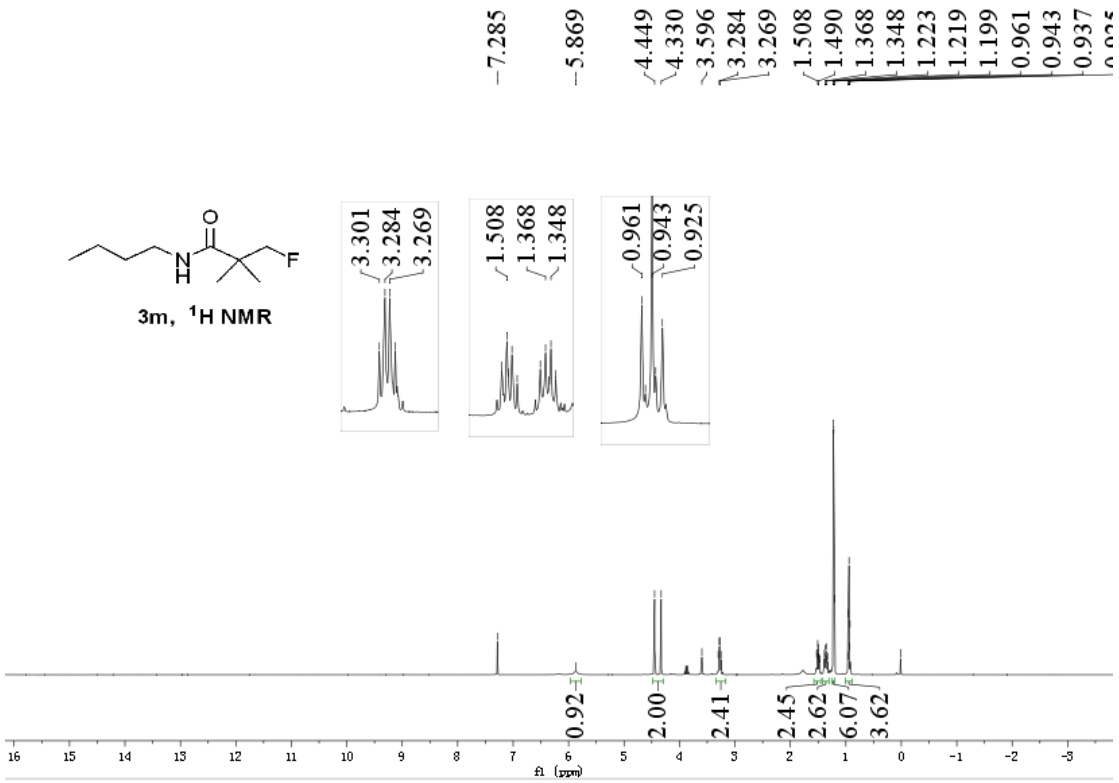
3j, DEPT 135

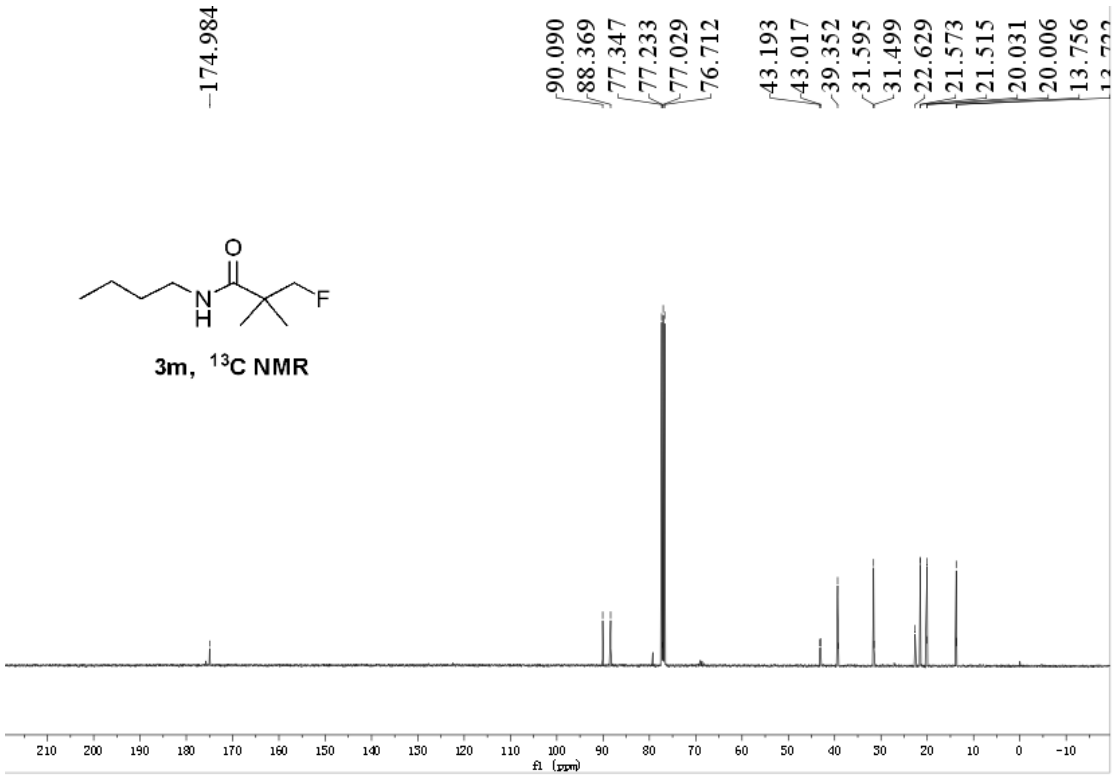


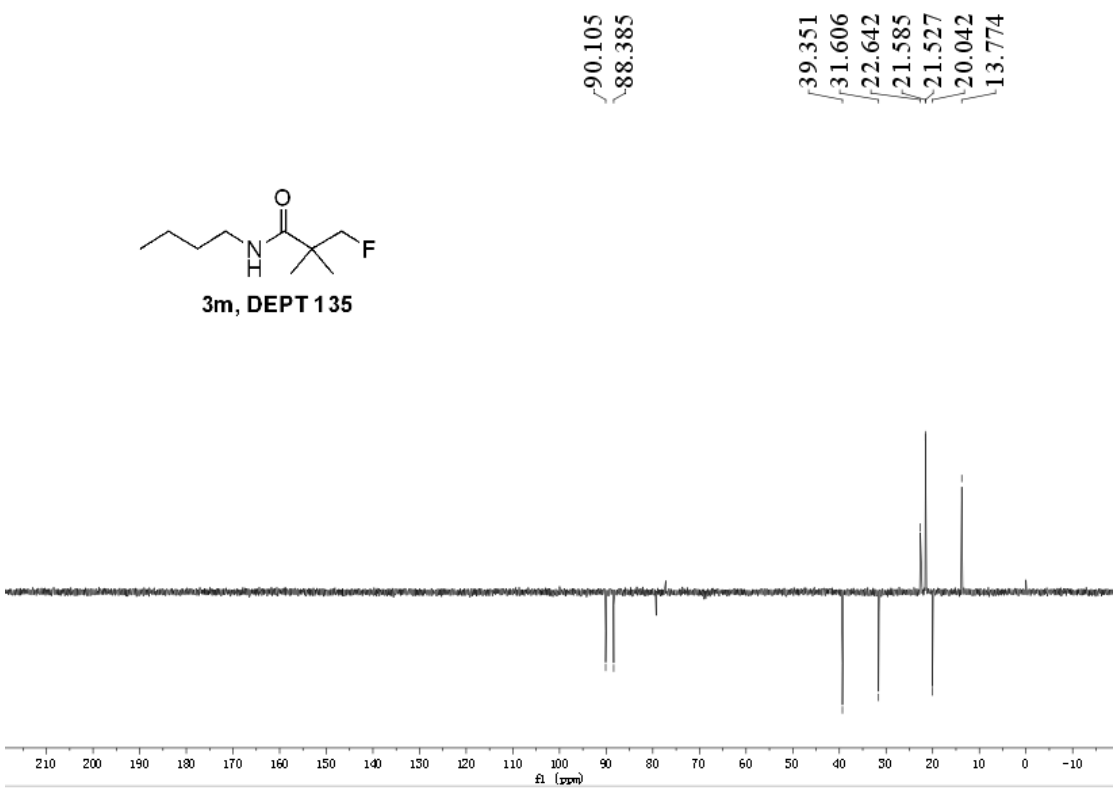
3j, ^{19}F NMR

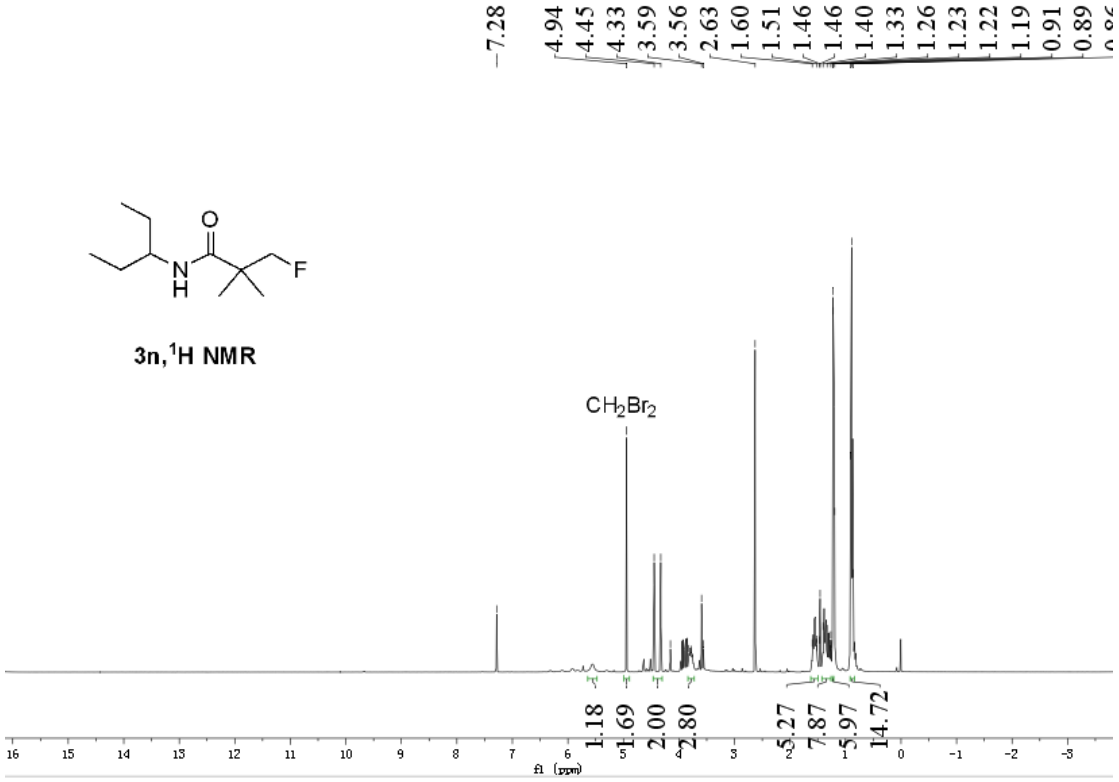


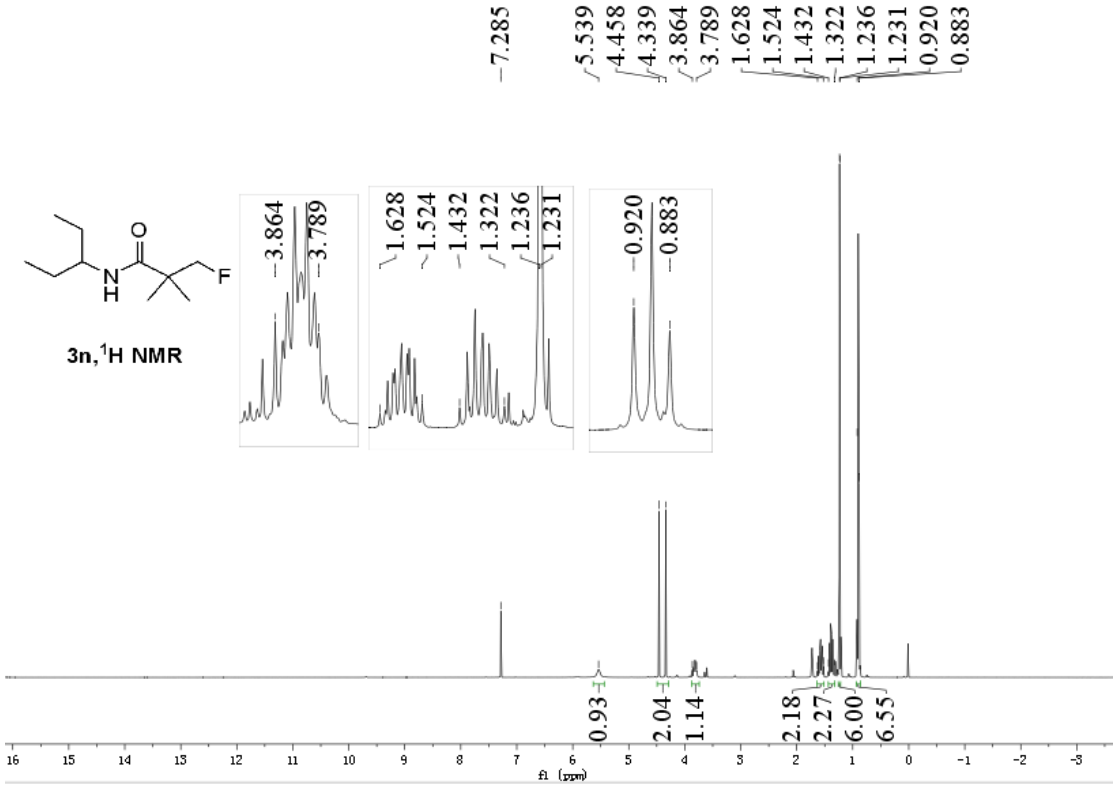
3m, ^{1}H NMR

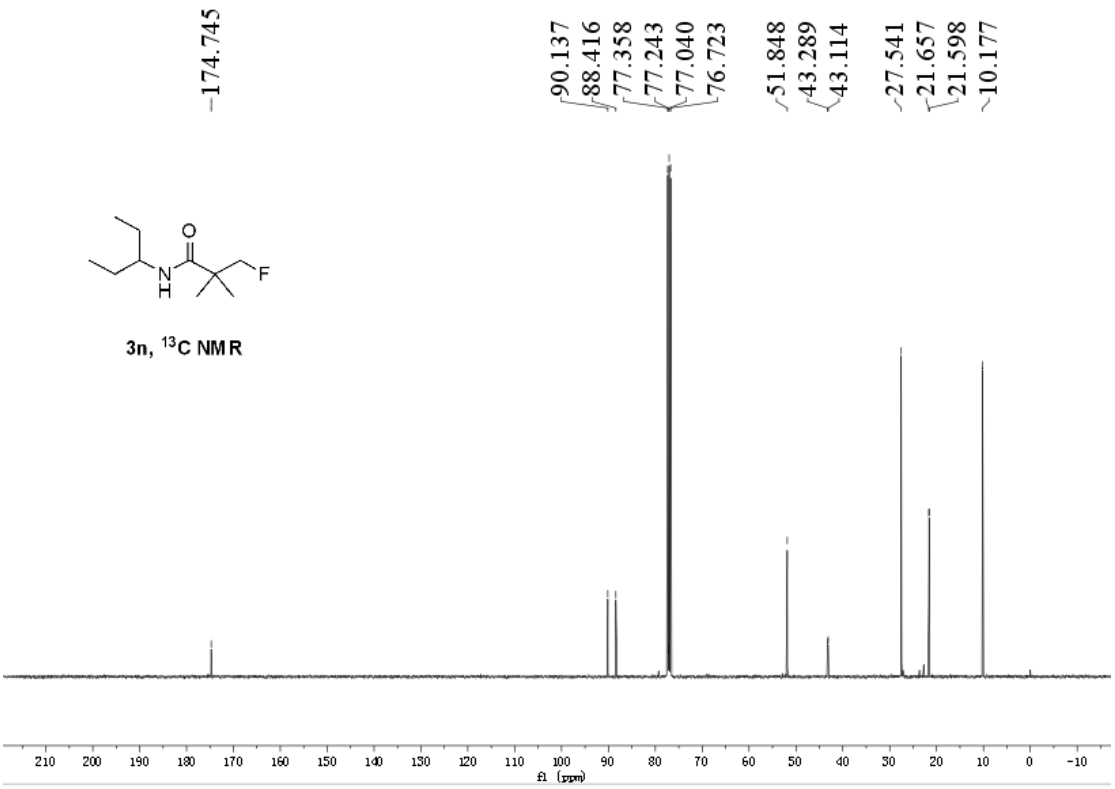

7.285
5.869
4.449
4.330
3.596
3.284
3.269
1.508
1.490
1.368
1.348
1.223
1.219
1.199
0.961
0.943
0.937
3m, 1H NMR
3.301
3.284
3.269
1.508
1.368
1.348
0.961
0.943
0.925
0.92
2.00
2.41
2.45
2.62
6.07
3.62
f1 (ppm)


174.984
90.090
88.369
77.347
77.233
77.029
76.712
43.193
43.017
39.352
31.595
31.499
22.629
21.573
21.515
20.031
20.006
13.756
3m, 13C NMR
f1 (ppm)

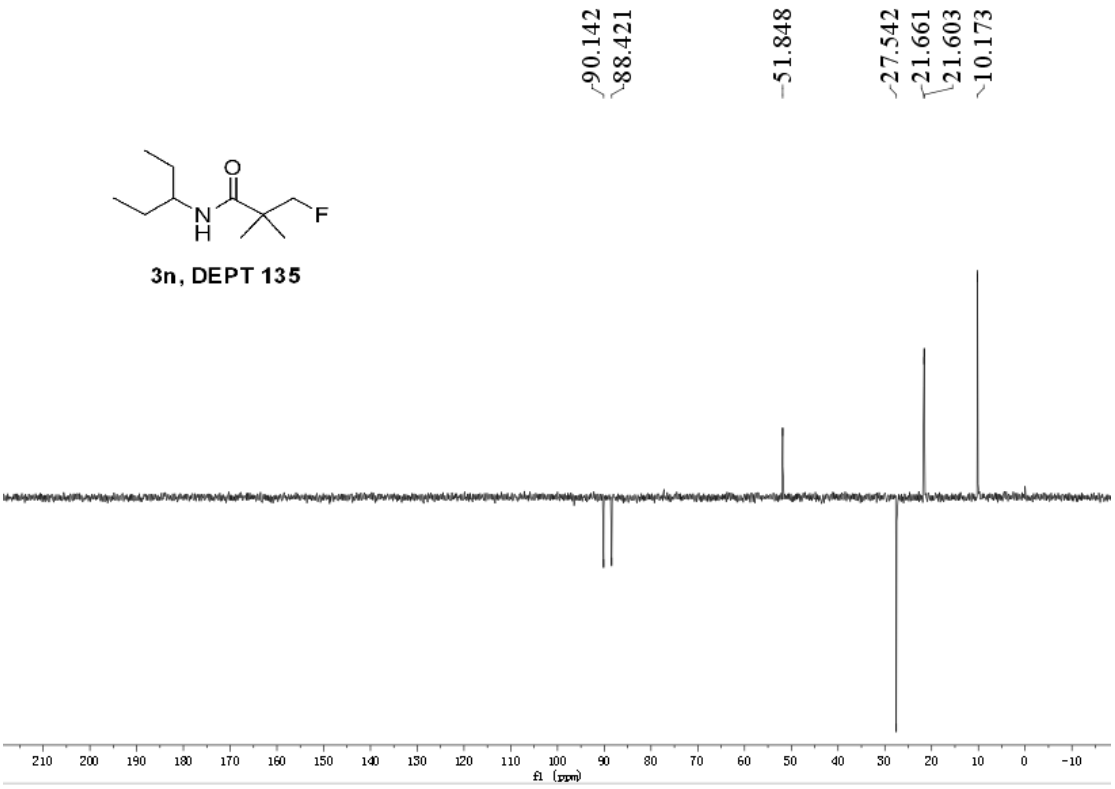

90.105
88.385
39.351
31.606
22.642
21.585
21.527
20.042
13.774
O
N
H
F
3m, DEPT 135
f1 (ppm)
-74.14
3m, 19F NMR
f1 (ppm)


7.28
4.94
4.45
4.33
3.59
3.56
2.63
1.60
1.51
1.46
1.46
1.40
1.33
1.26
1.23
1.22
1.19
0.91
0.89
O
N
H
F
3n,1H NMR
CH2Br2
1.18
1.69
2.00
2.80
5.27
7.87
5.97
14.72
f1 (ppm)

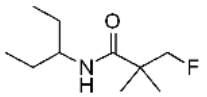

7.285
5.539
4.458
4.339
3.864
3.789
1.628
1.524
1.432
1.322
1.236
1.231
0.920
0.883
O
N
H
F
3n,1H NMR
3.864
3.789
1.628
1.524
1.432
1.322
1.236
1.231
0.920
0.883
0.93
2.04
1.14
2.18
2.27
6.00
6.55
f1 (ppm)



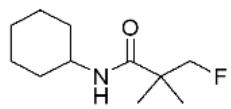
3n, ^{13}C NMR



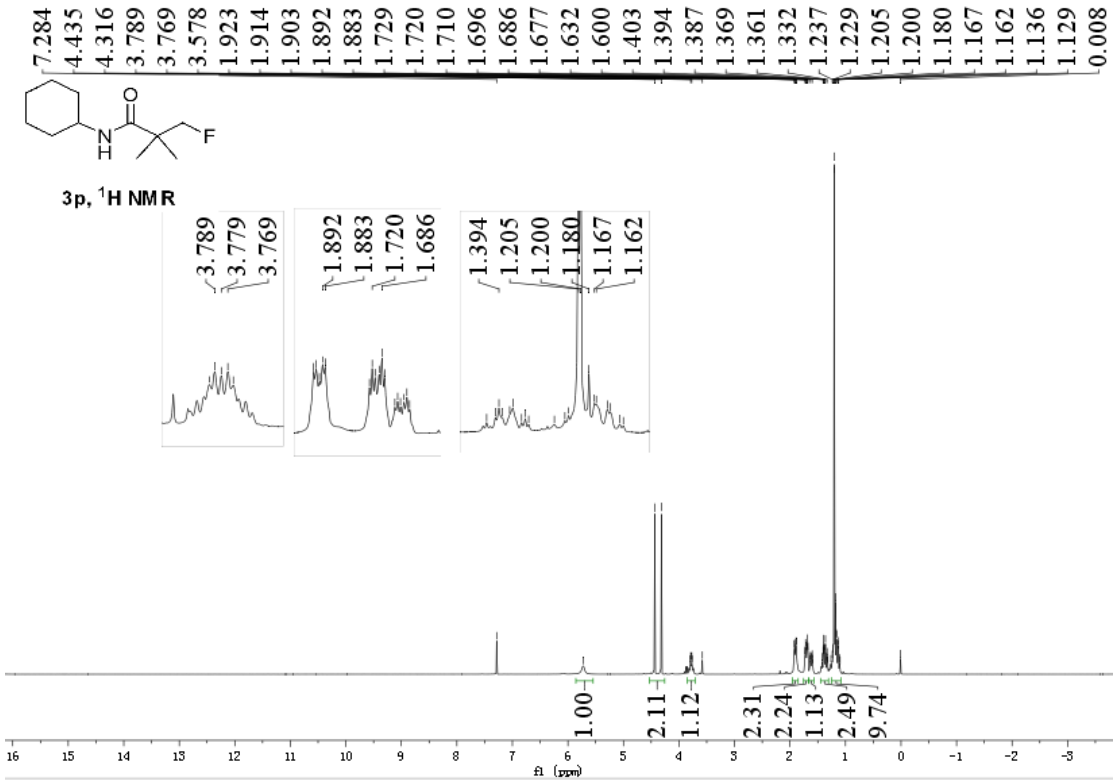
3n, DEPT 135

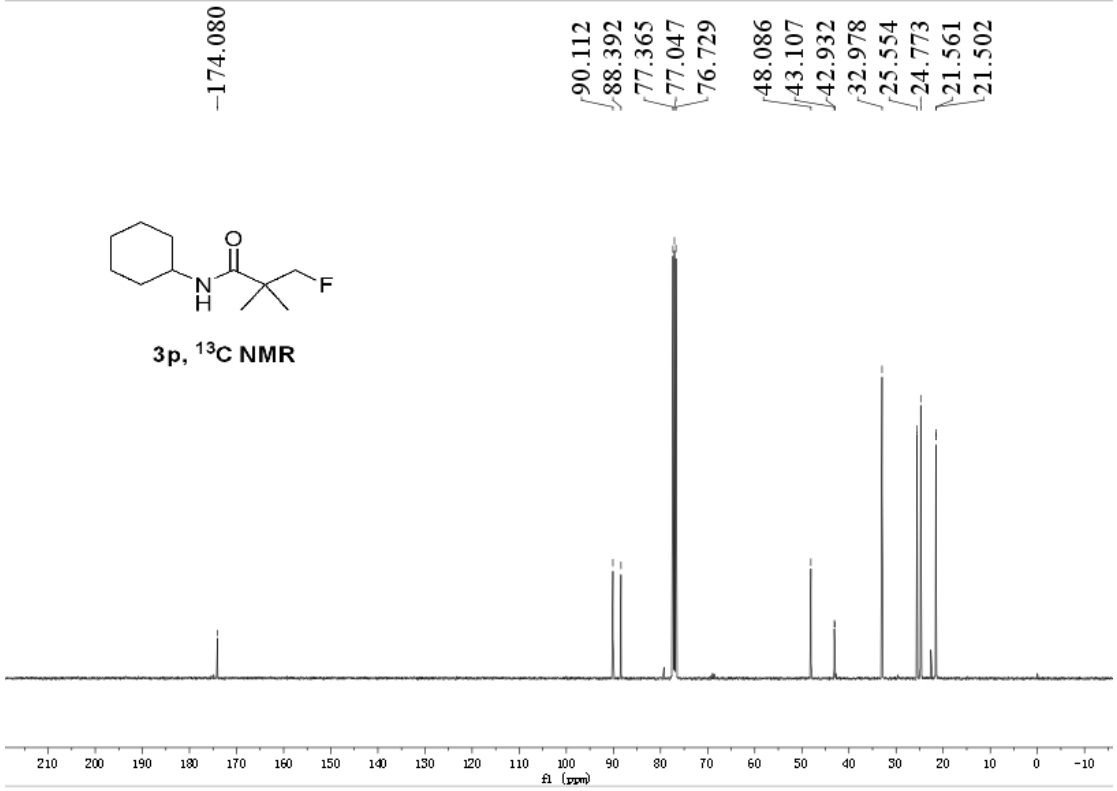


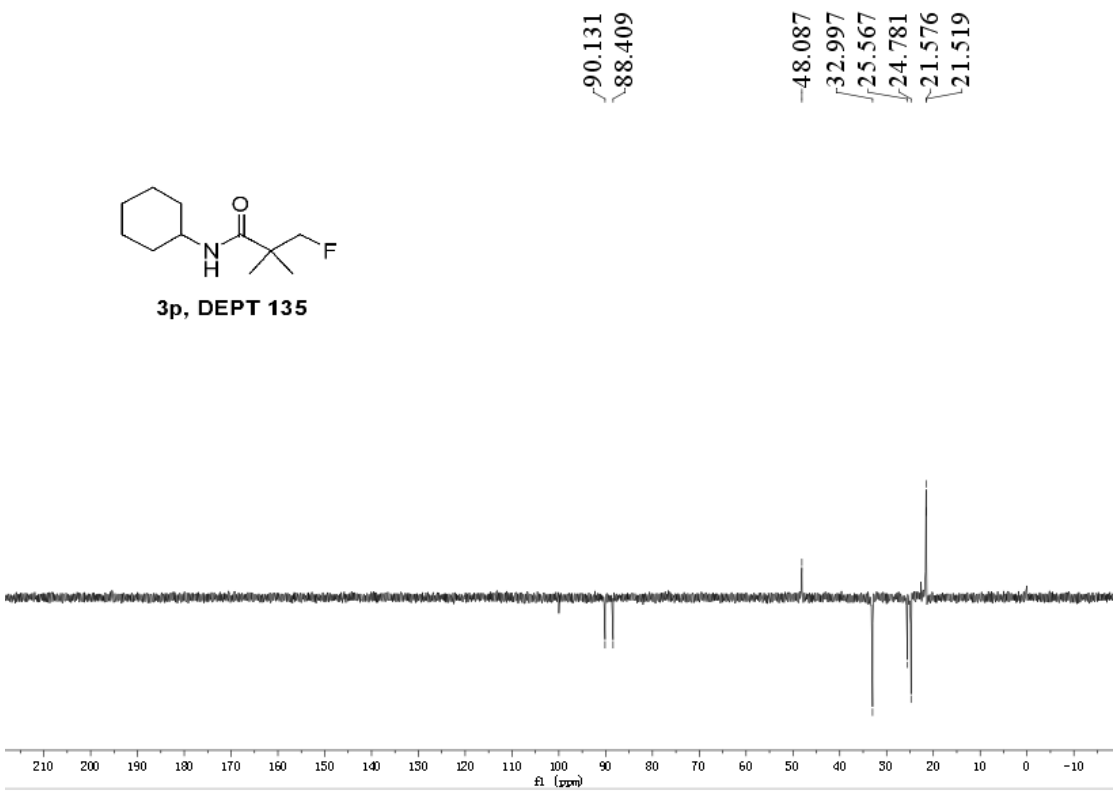
3n, ^{19}F NMR



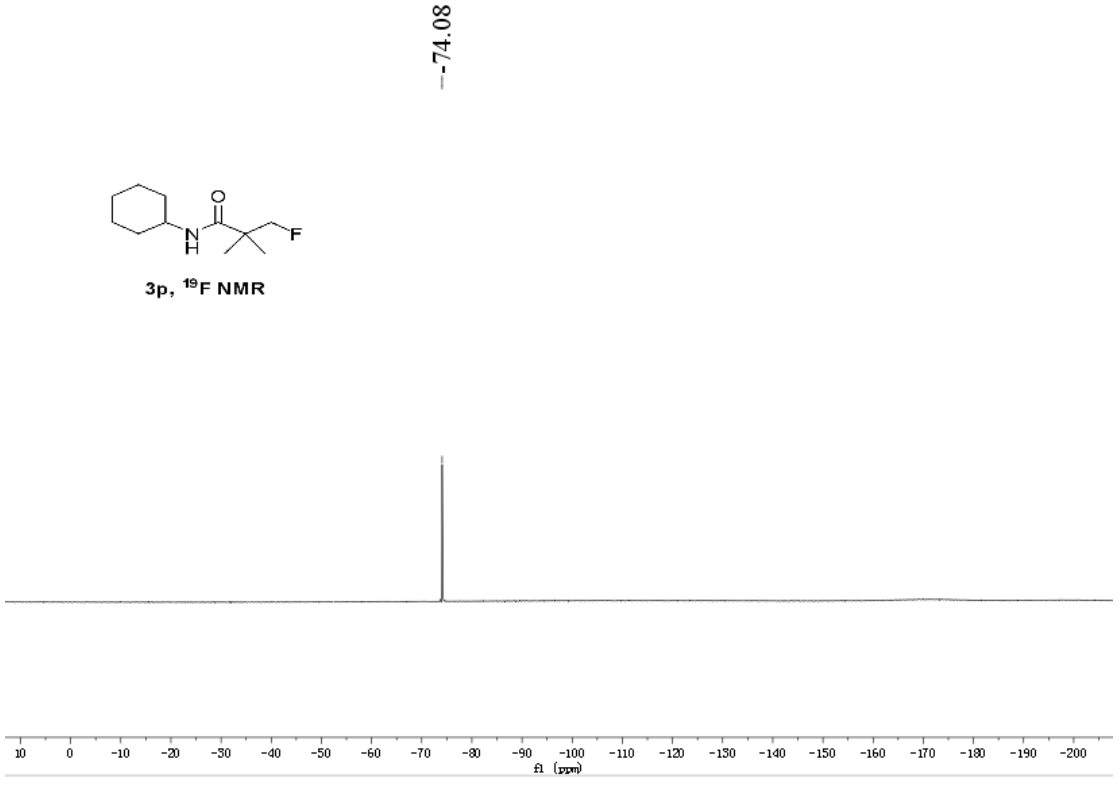
3p, ^{1}H NMR


7.284
4.435
4.316
3.789
3.769
3.578
1.923
1.914
1.903
1.892
1.883
1.729
1.720
1.710
1.696
1.686
1.677
1.632
1.600
1.403
1.394
1.387
1.369
1.361
1.332
1.237
1.229
1.205
1.200
1.180
1.167
1.162
1.136
1.129
0.008
O
N
H
F
3p, 1H NMR
3.789
3.779
3.769
1.892
1.883
1.720
1.686
1.394
1.205
1.200
1.180
1.167
1.162
1.00
2.11
1.12
2.31
2.24
1.13
2.49
9.74
f1 (ppm)

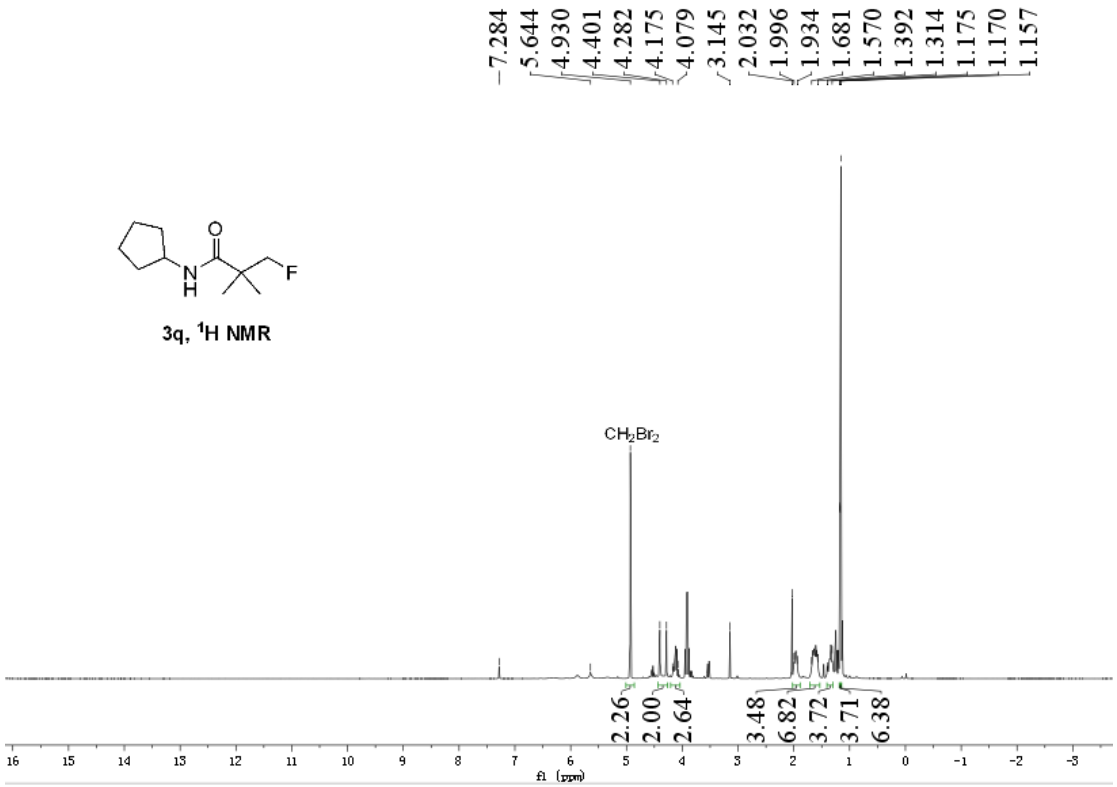

174.080
90.112
88.392
77.365
77.047
76.729
48.086
43.107
42.932
32.978
25.554
24.773
21.561
21.502
O
N
H
F
3p, 13C NMR
f1 (ppm)

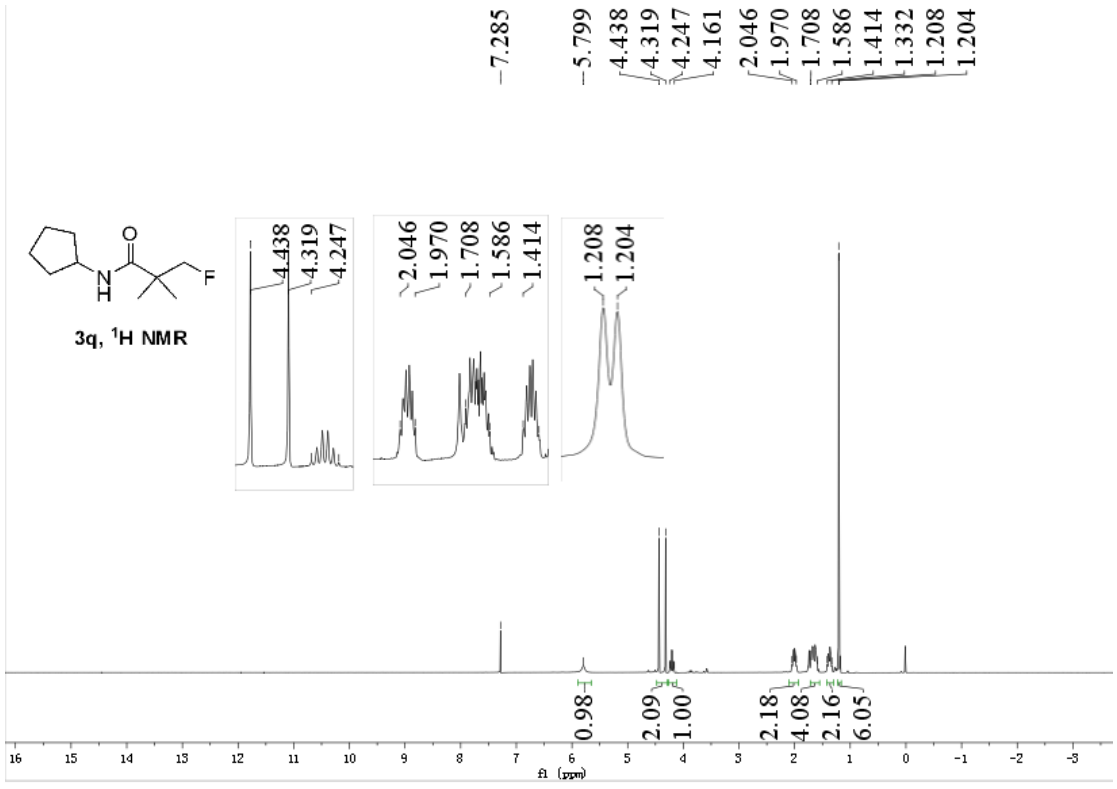


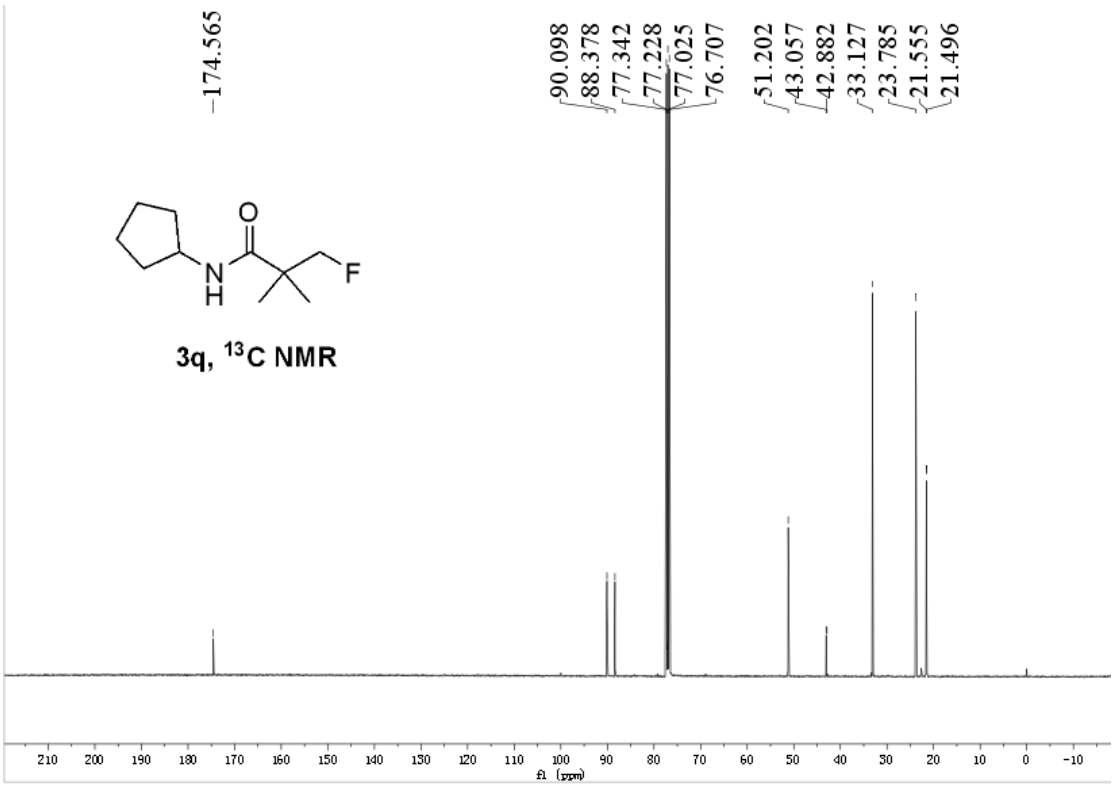
3p, DEPT 135



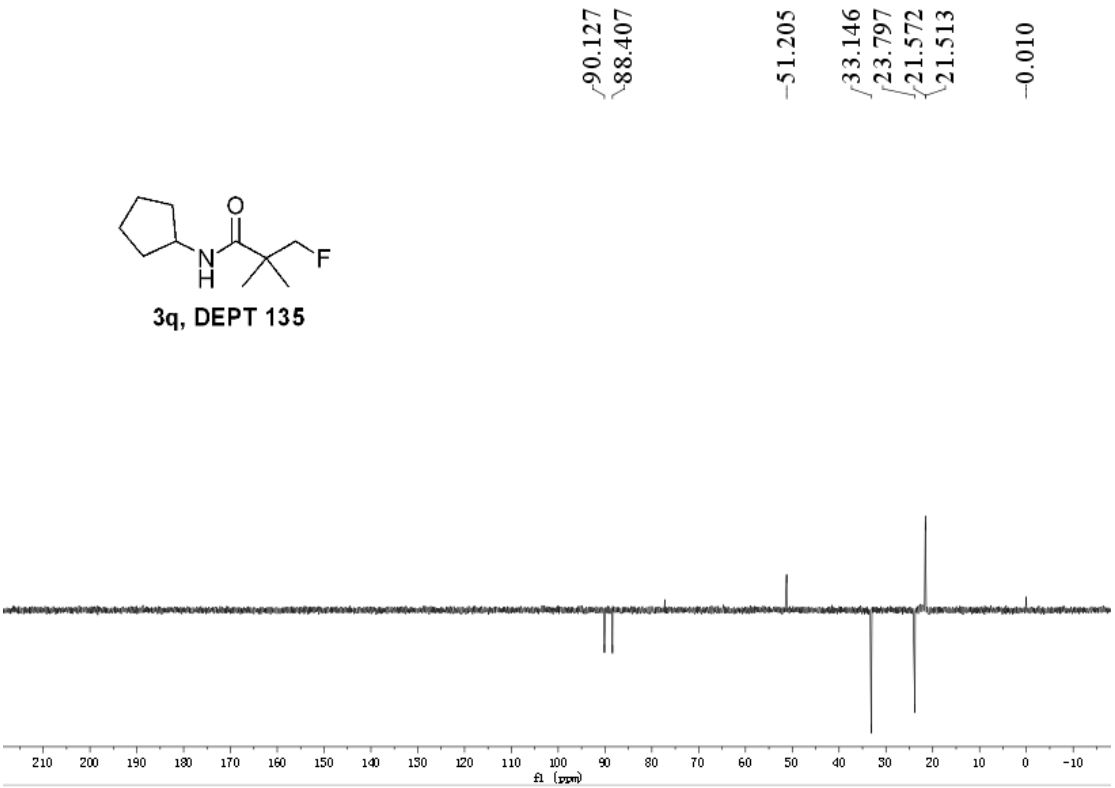
3p, ^{19}F NMR


7.284
5.644
4.930
4.401
4.282
4.175
4.079
3.145
2.032
1.996
1.934
1.681
1.570
1.392
1.314
1.175
1.170
1.157
O
N
H
F
3q, 1H NMR
CH2Br2
2.26
2.00
2.64
3.48
6.82
3.72
3.71
6.38
f1 (ppm)

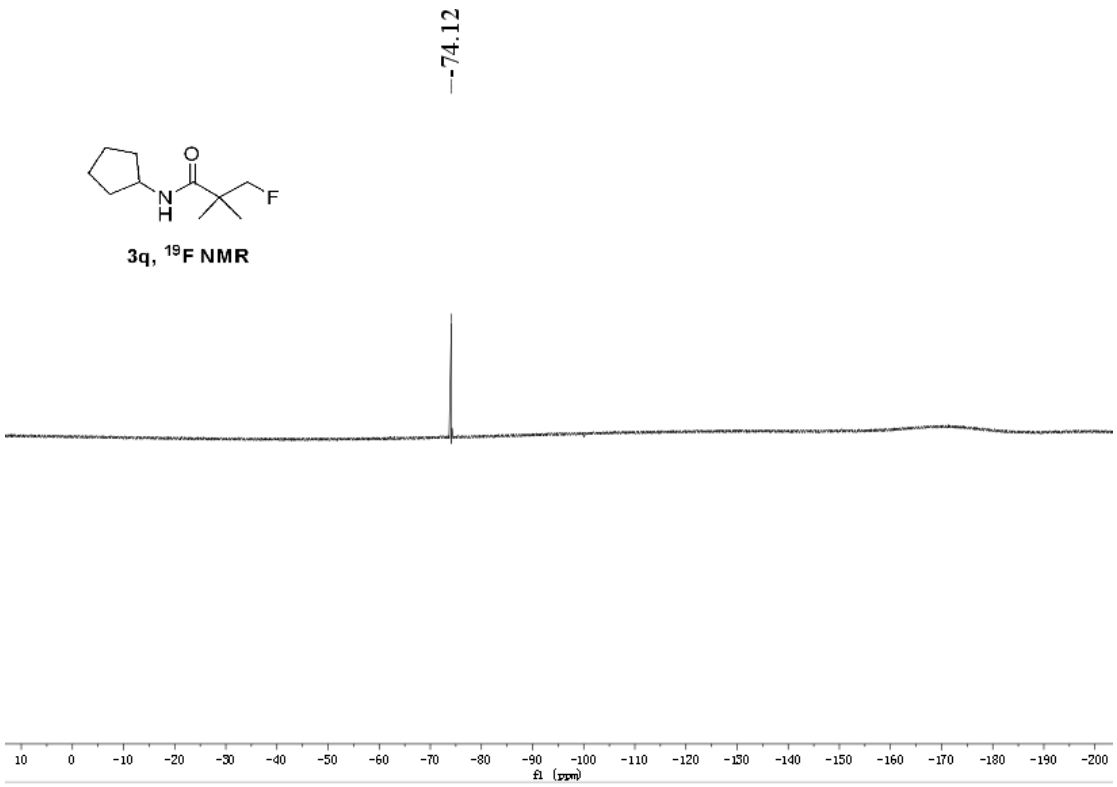

7.285
5.799
4.438
4.319
4.247
4.161
2.046
1.970
1.708
1.586
1.414
1.332
1.208
1.204
O
N
H
F
3q, 1H NMR
0.98
2.09
1.00
2.18
4.08
2.16
6.05
f1 (ppm)



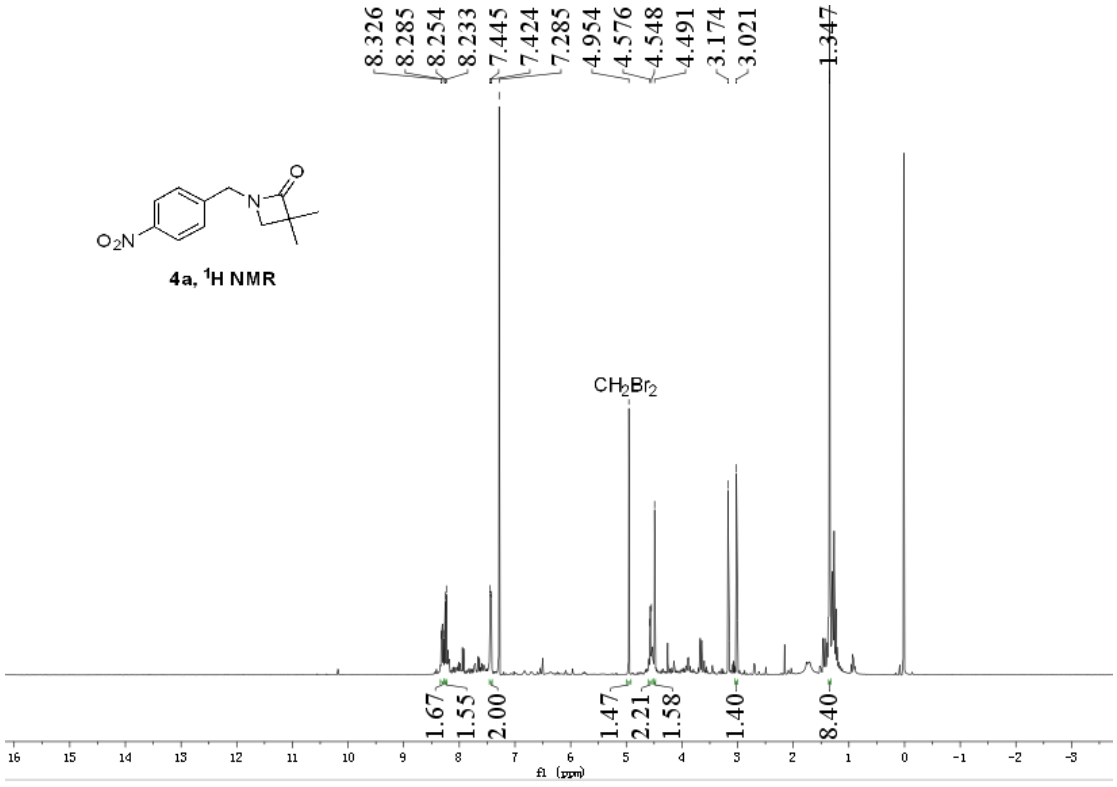
3q, ^{13}C NMR

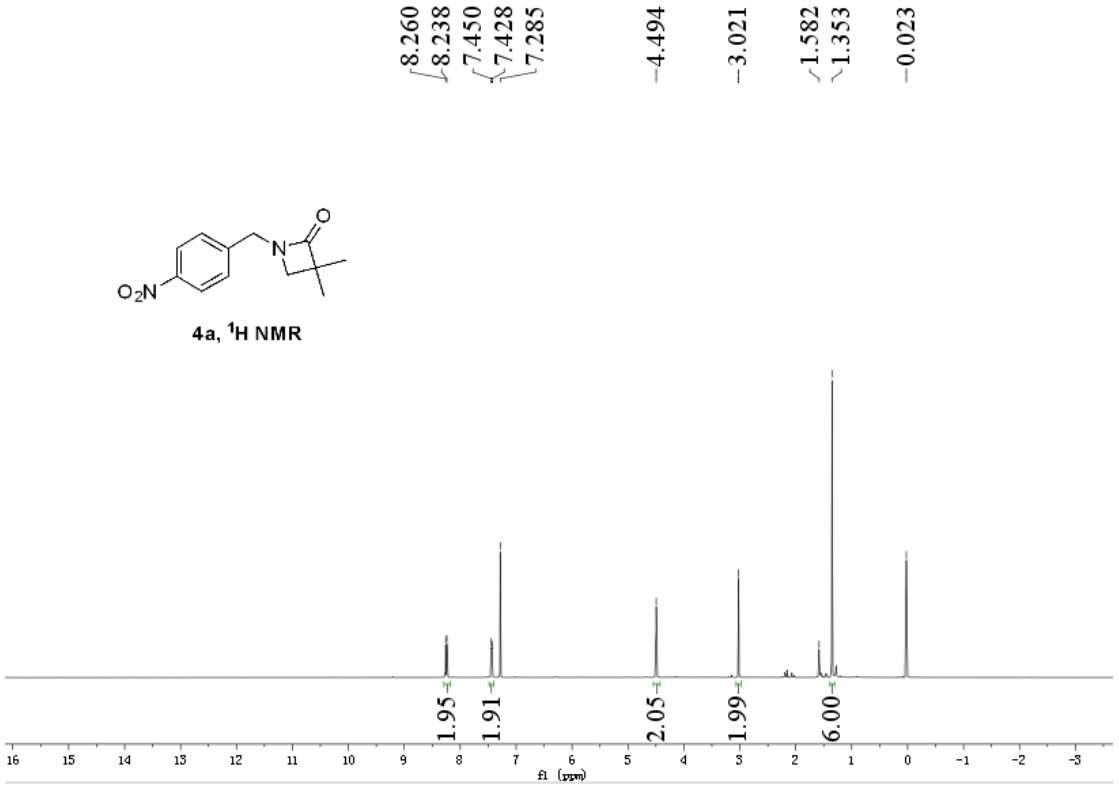


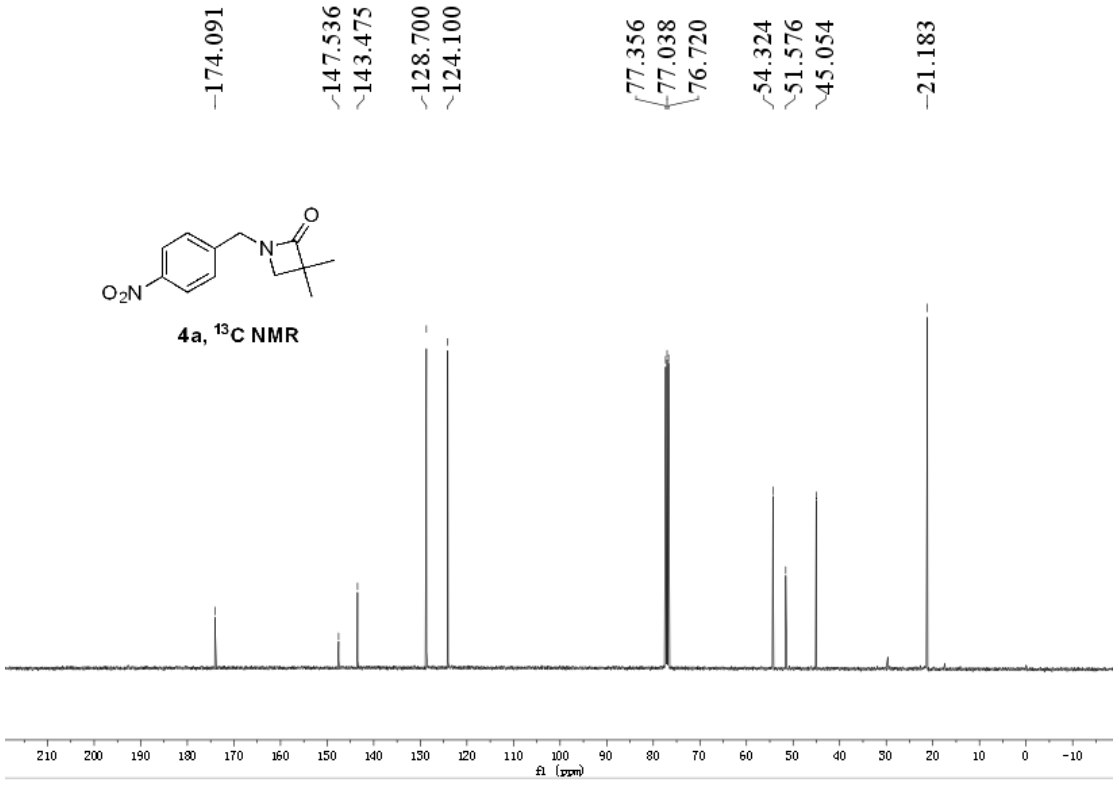
3q, DEPT 135

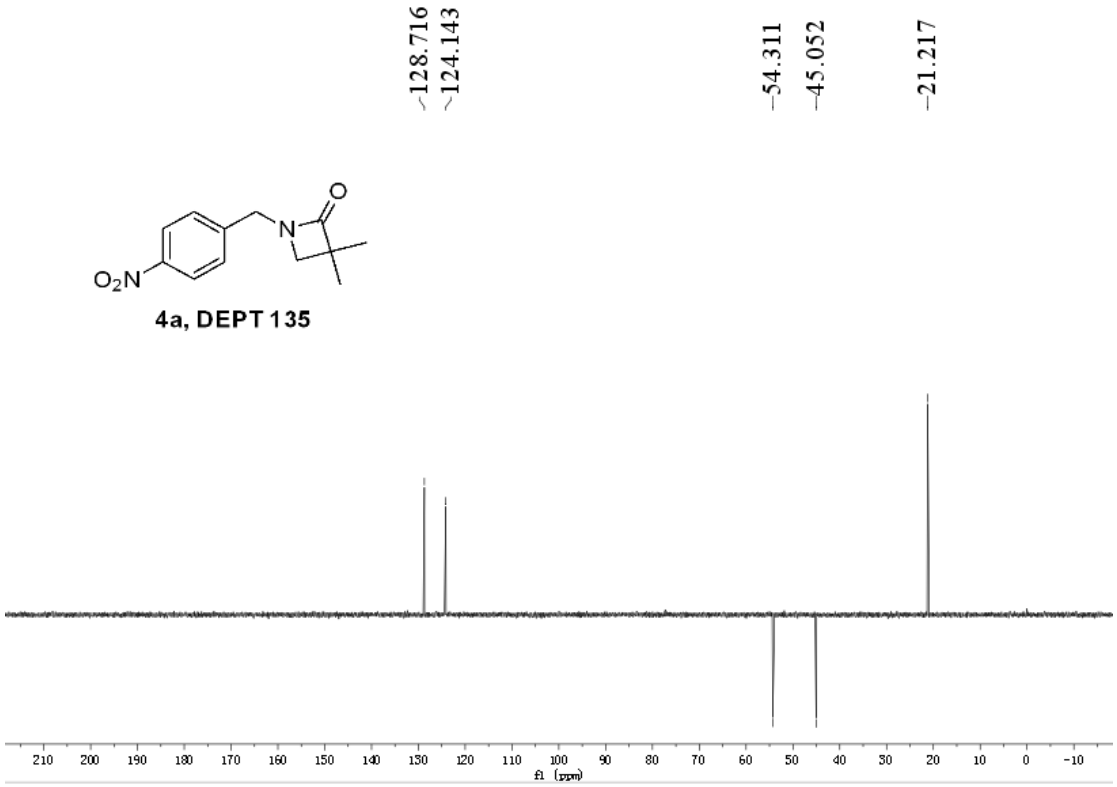

-74.12
3q, 19F NMR
f1 (ppm)

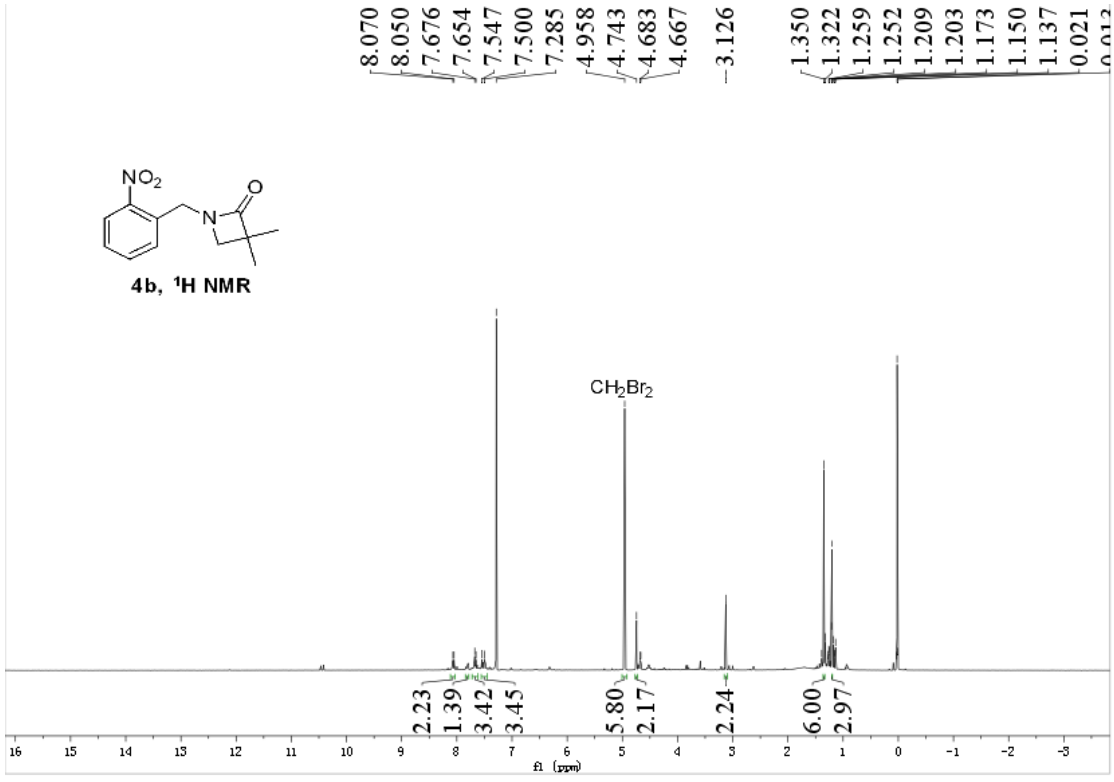
(4) *β*-Lactams

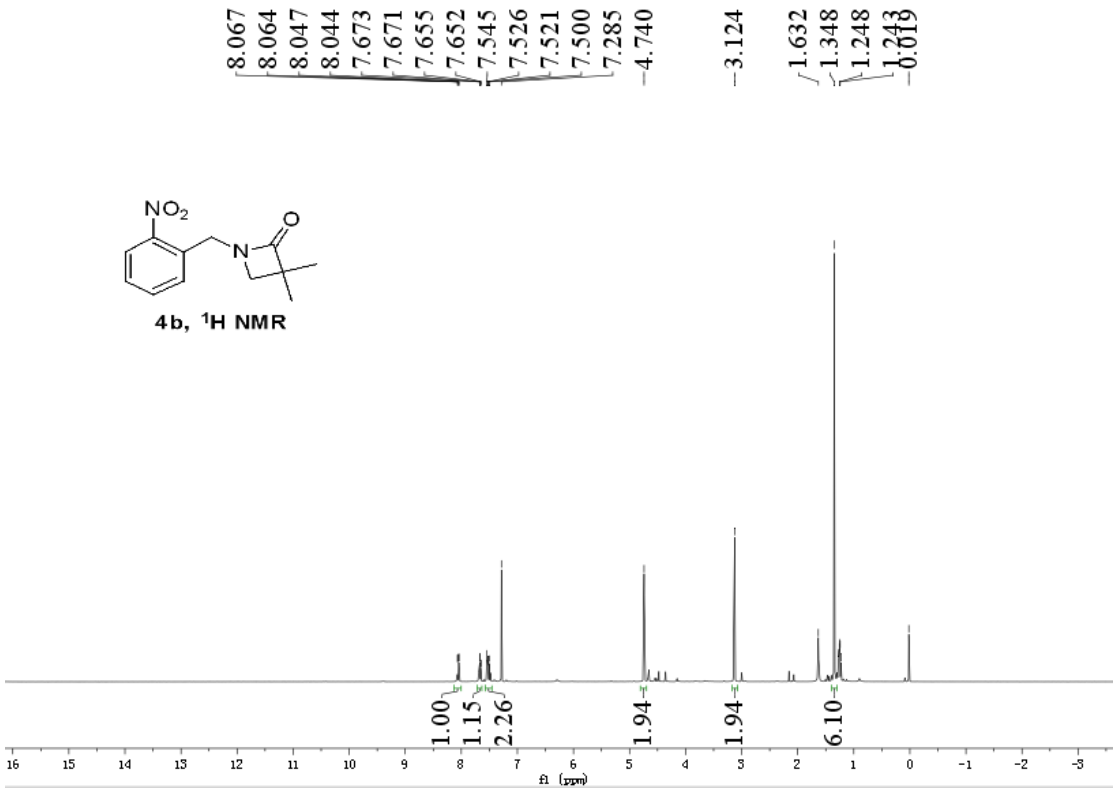

8.326
8.285
8.254
8.233
7.445
7.424
7.285
4.954
4.576
4.548
4.491
3.174
3.021
1.347
4a, 1H NMR
CH2Br2
1.67
1.55
2.00
1.47
2.21
1.58
1.40
8.40
f1 (ppm)

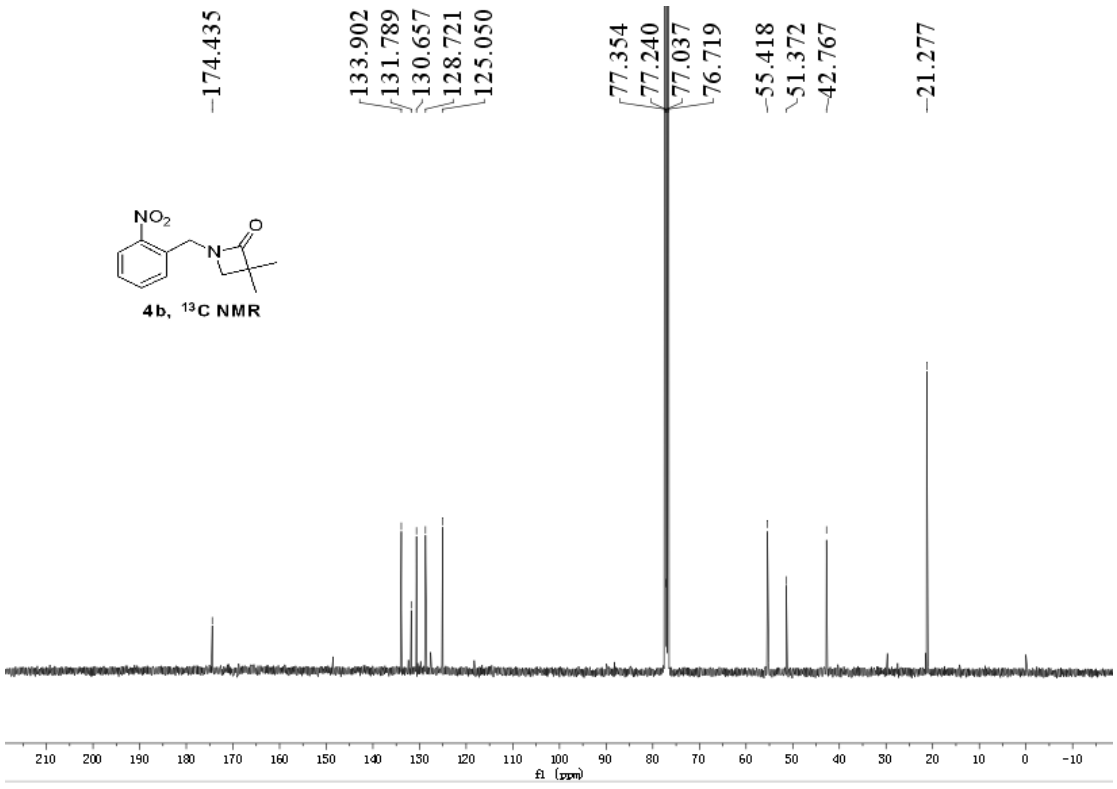

8.260
8.238
7.450
7.428
7.285
4.494
3.021
1.582
1.353
0.023
O2N
N
O
4a, 1H NMR
1.95
1.91
2.05
1.99
6.00
f1 (ppm)

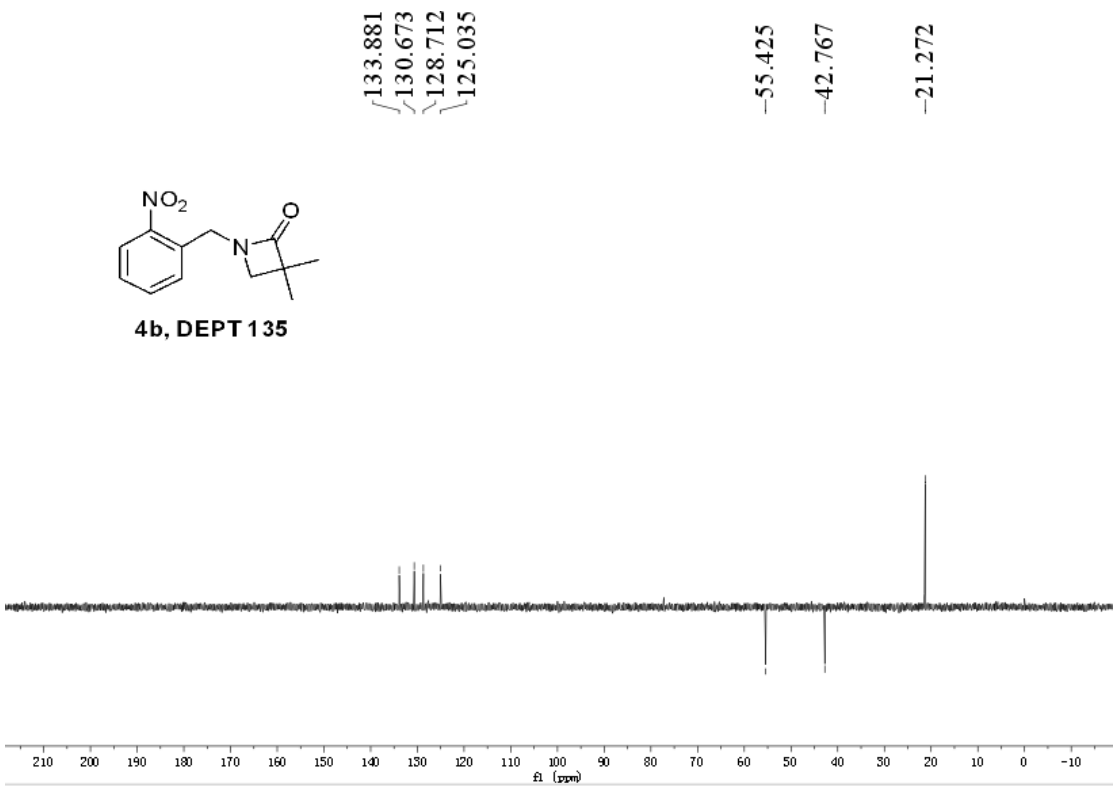

174.091
147.536
143.475
128.700
124.100
77.356
77.038
76.720
54.324
51.576
45.054
21.183
O2N
N
O
4a, 13C NMR
f1 (ppm)

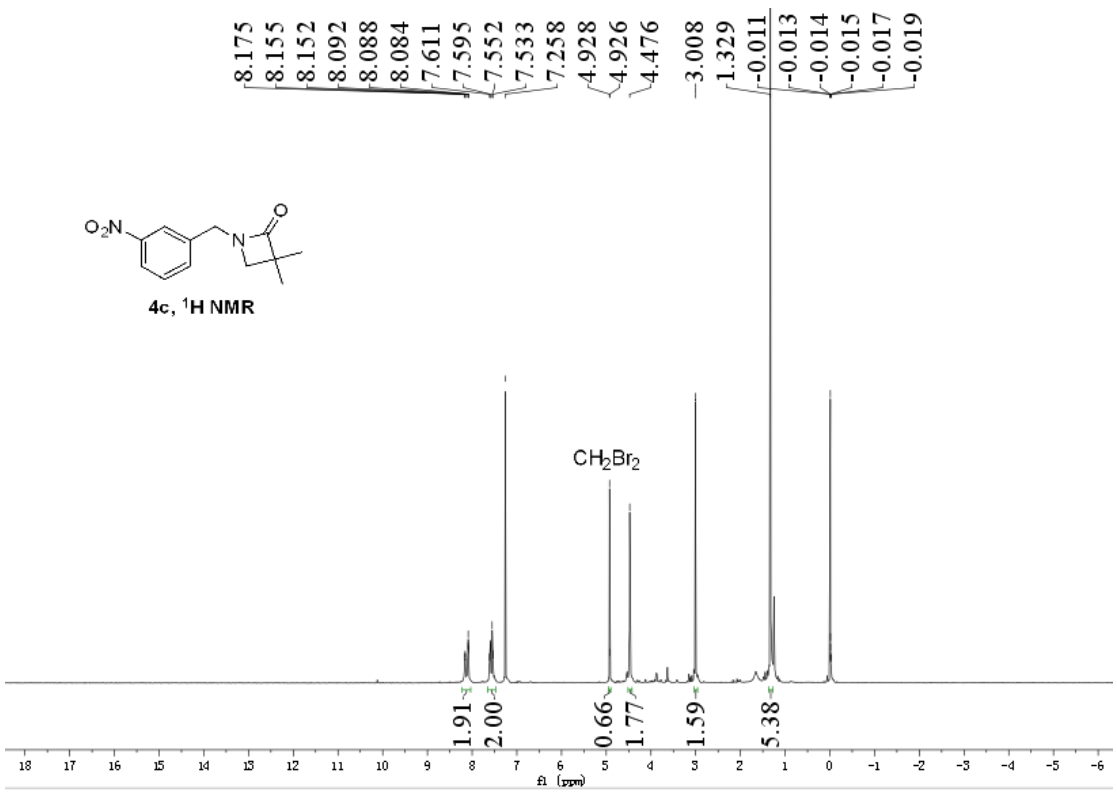

128.716
124.143
54.311
45.052
21.217
O2N
4a, DEPT 135

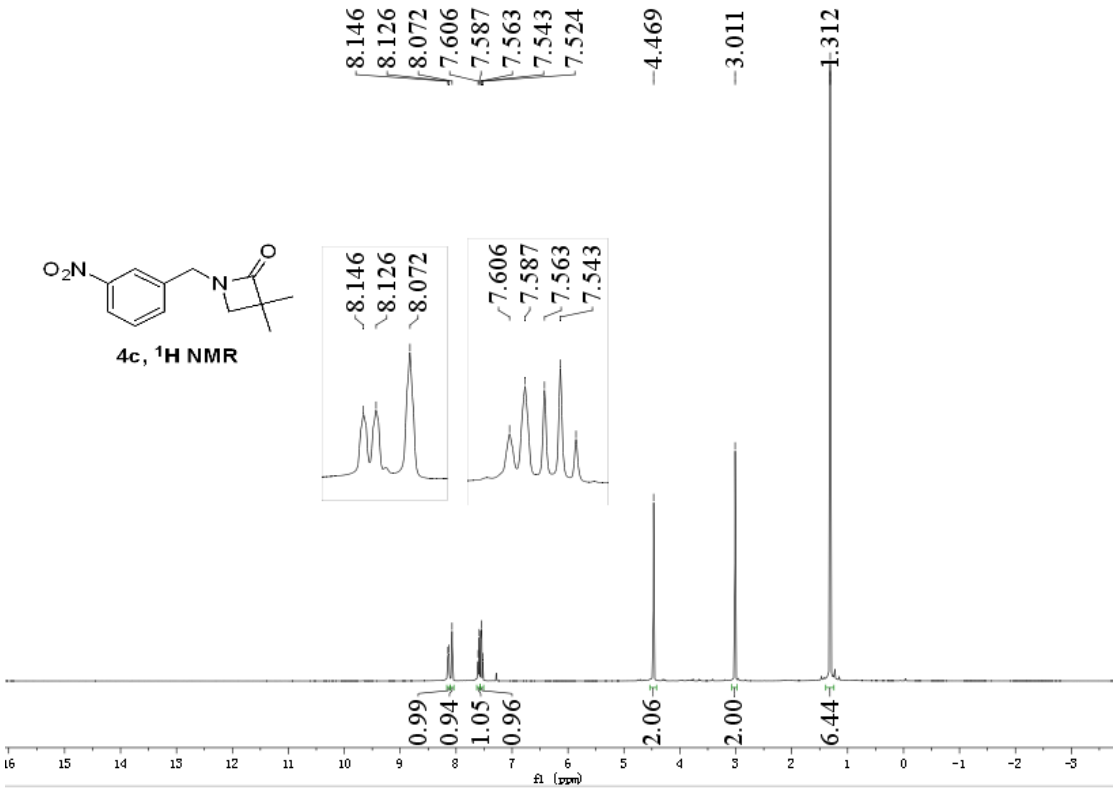

8.070
8.050
7.676
7.654
7.547
7.500
7.285
4.958
4.743
4.683
4.667
3.126
1.350
1.322
1.259
1.252
1.209
1.203
1.173
1.150
1.137
0.021
NO2
4b, 1H NMR
CH2Br2
2.23
1.39
3.42
3.45
5.80
2.17
2.24
6.00
2.97


8.067
8.064
8.047
8.044
7.673
7.671
7.655
7.652
7.545
7.526
7.521
7.500
7.285
4.740
3.124
1.632
1.348
1.248
1.243
0.019
NO2
N
O
4b, 1H NMR
1.00
1.15
2.26
1.94
1.94
6.10
f1 (ppm)


174.435
133.902
131.789
130.657
128.721
125.050
77.354
77.240
77.037
76.719
55.418
51.372
42.767
21.277
NO2
N
O
4b, 13C NMR
f1 (ppm)


133.881
130.673
128.712
125.035
55.425
42.767
21.272
NO2
O
N
4b, DEPT 135
f1 (ppm)


8.175
8.155
8.152
8.092
8.088
8.084
7.611
7.595
7.552
7.533
7.258
4.928
4.926
4.476
3.008
1.329
-0.011
-0.013
-0.014
-0.015
-0.017
-0.019
O2N
O
N
4c, 1H NMR
CH2Br2
1.91
2.00
0.66
1.77
1.59
5.38
f1 (ppm)


8.146
8.126
8.072
7.606
7.587
7.563
7.543
7.524
4.469
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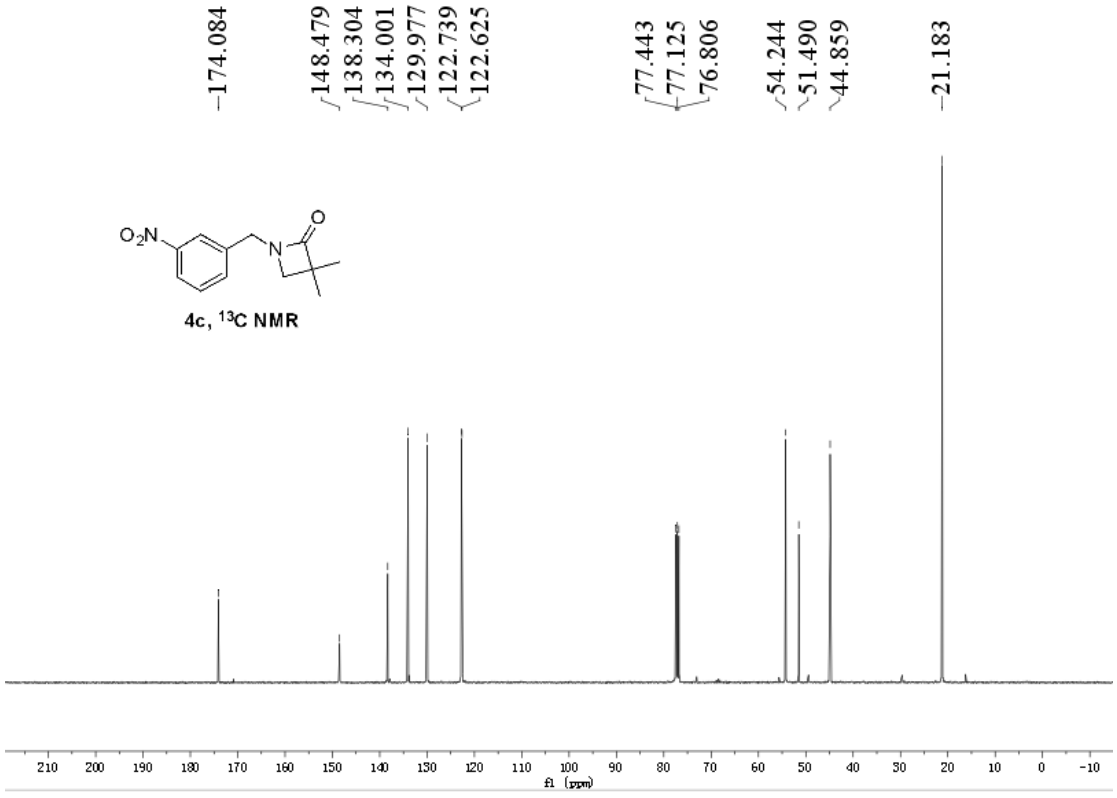
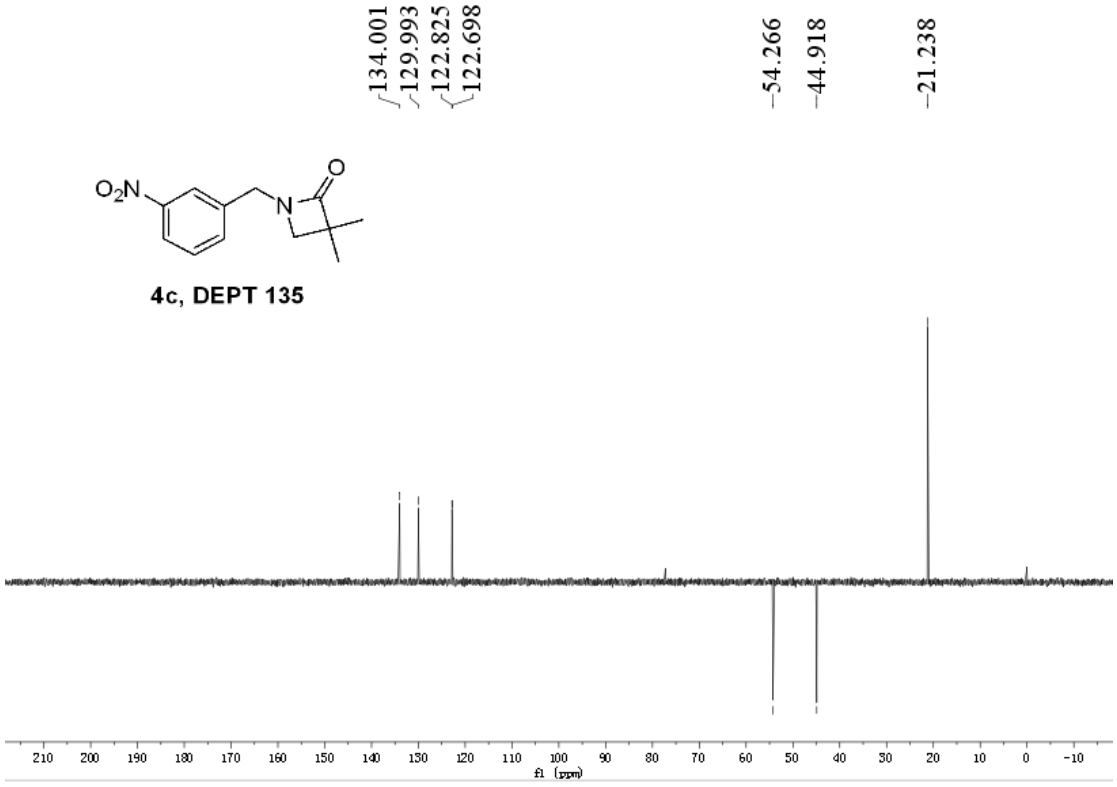
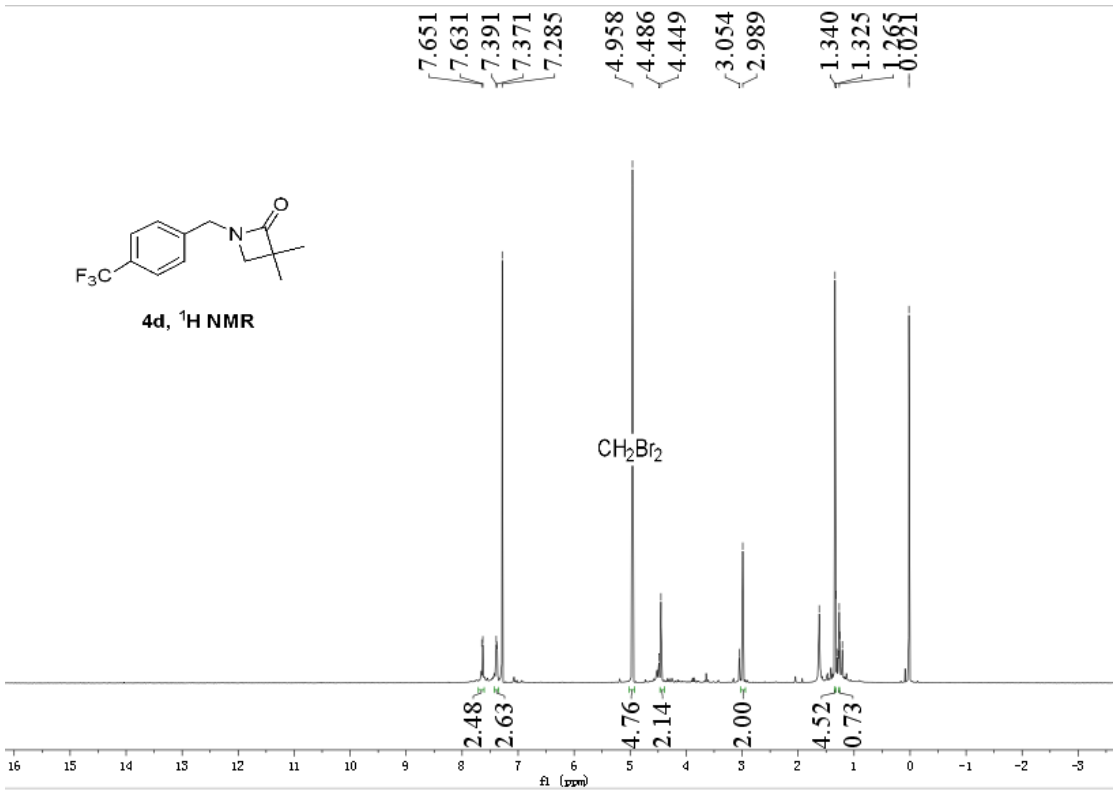
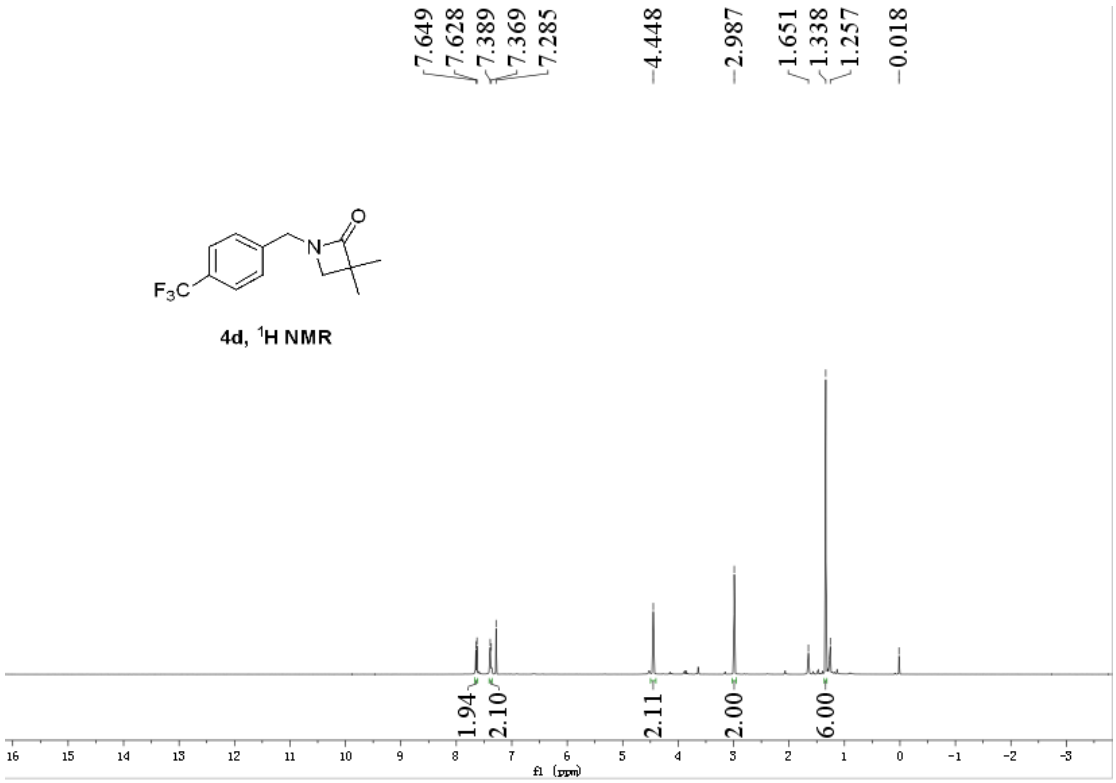
4c, 1H NMR
0.99
0.94
1.05
0.96
2.06
2.00
6.44
f1 (ppm)

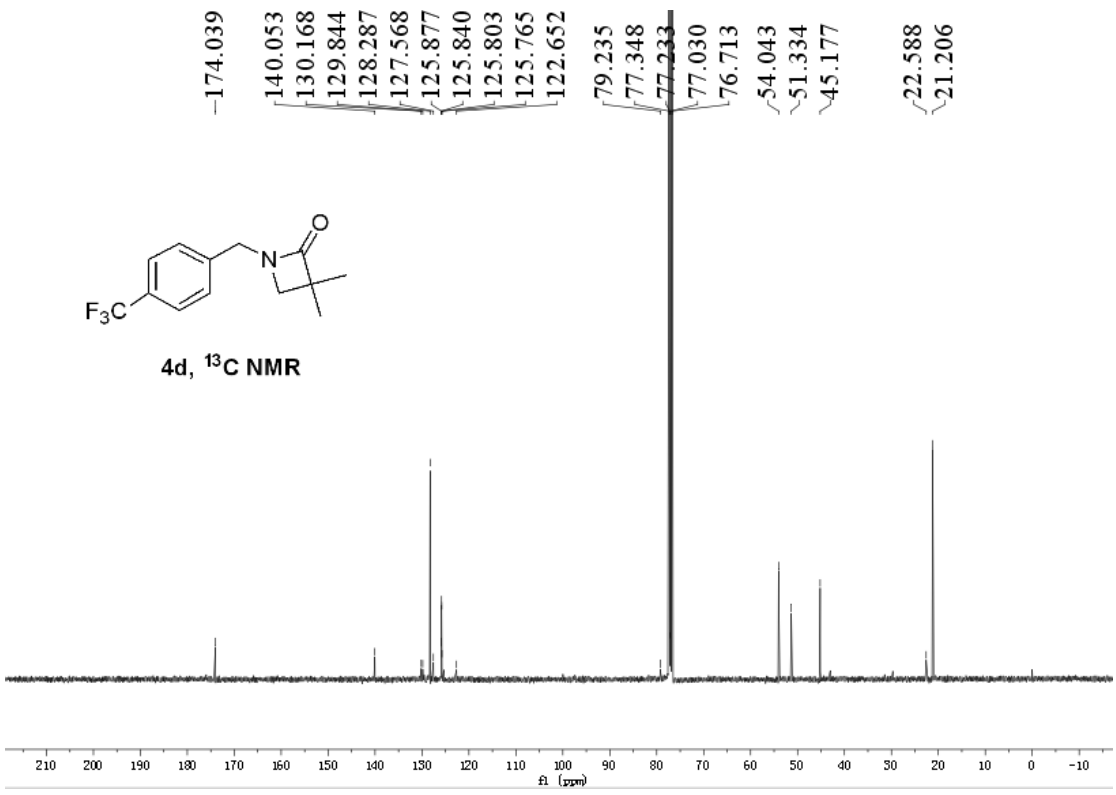

174.084
148.479
138.304
134.001
129.977
122.739
122.625
77.443
77.125
76.806
54.244
51.490
44.859
21.183
4c, 13C NMR
f1 (ppm)


134.001
129.993
122.825
122.698
54.266
44.918
21.238
O2N
N
O
4c, DEPT 135
f1 (ppm)

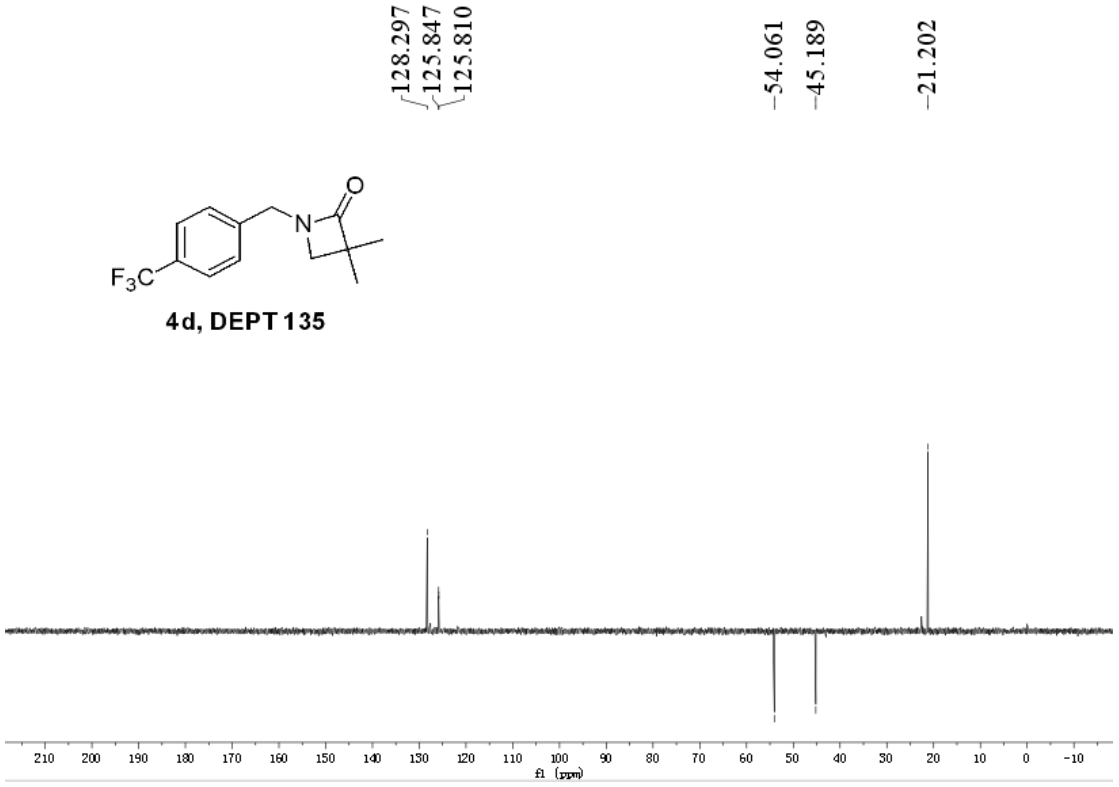

7.651
7.631
7.391
7.371
7.285
4.958
4.486
4.449
3.054
2.989
1.340
1.325
1.265
0.021
F3C
N
O
4d, 1H NMR
CH2Br2
2.48
2.63
4.76
2.14
2.00
4.52
0.73
f1 (ppm)



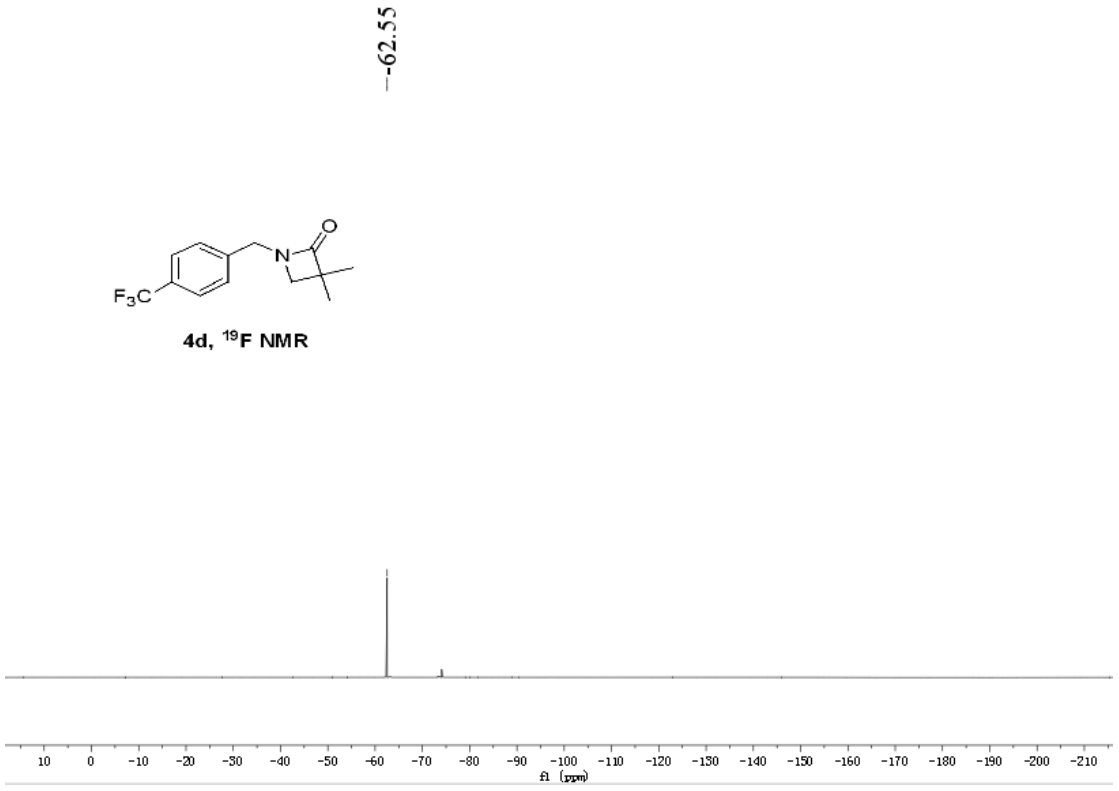
4d, ^{1}H NMR



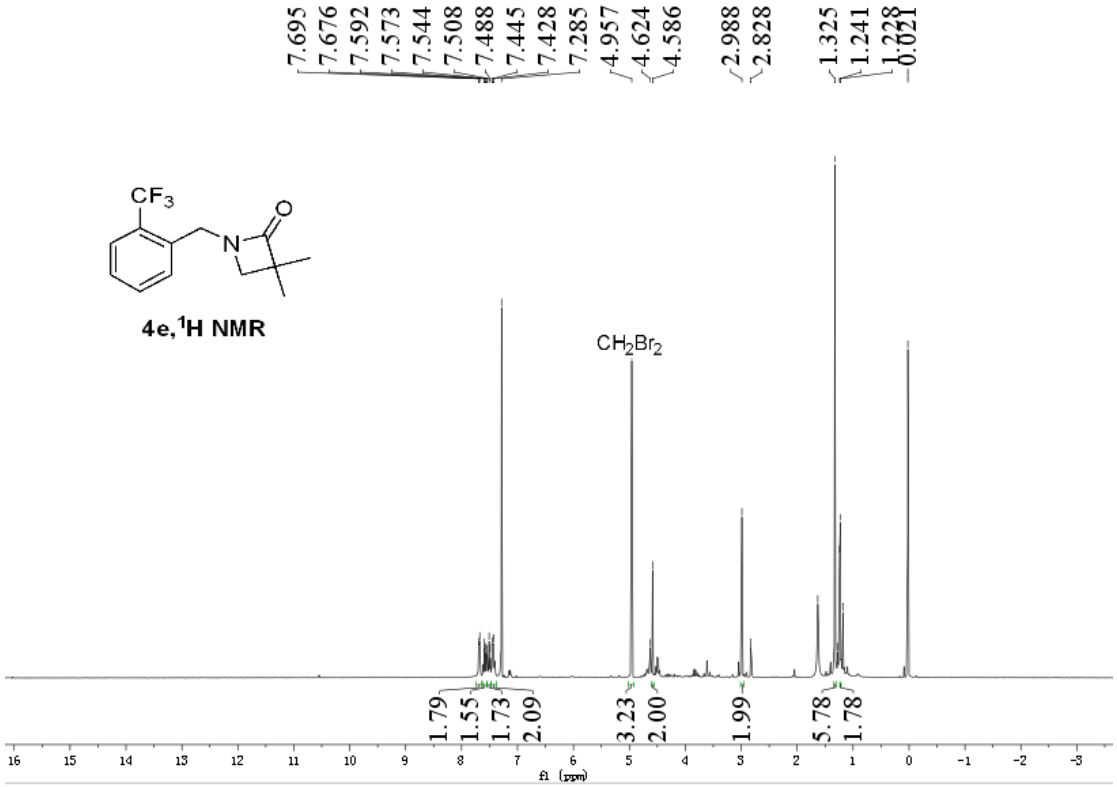
4d, ^{13}C NMR

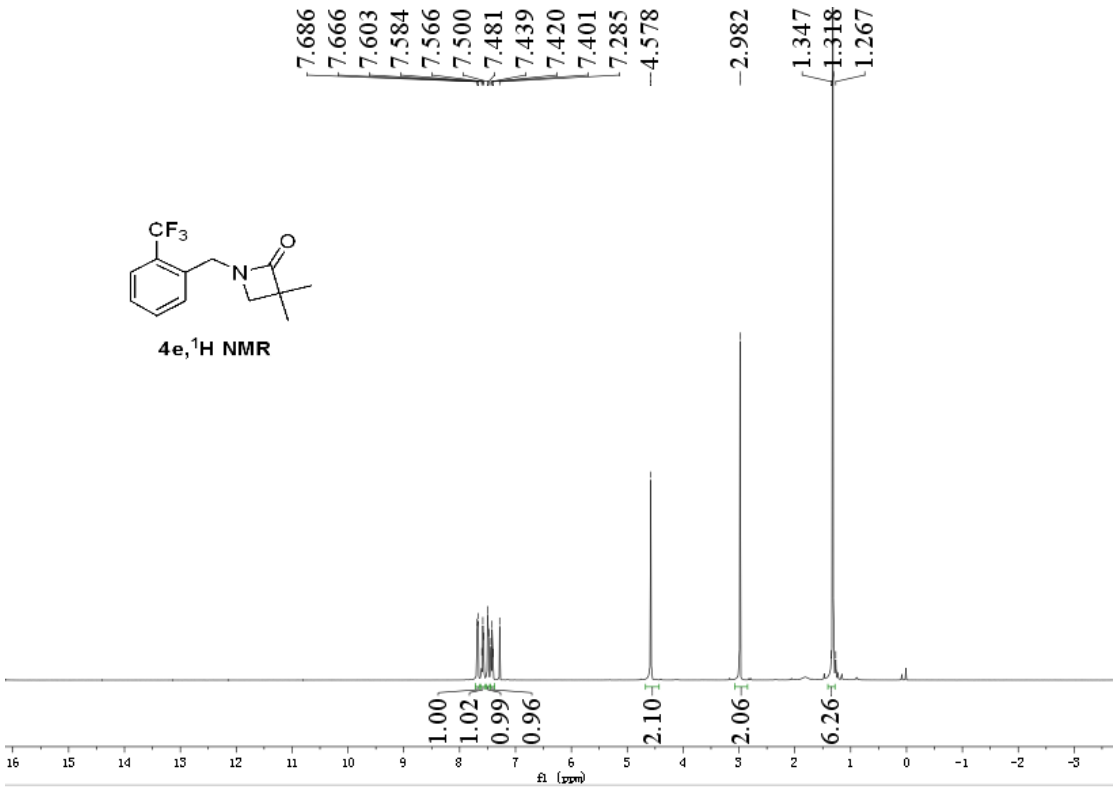


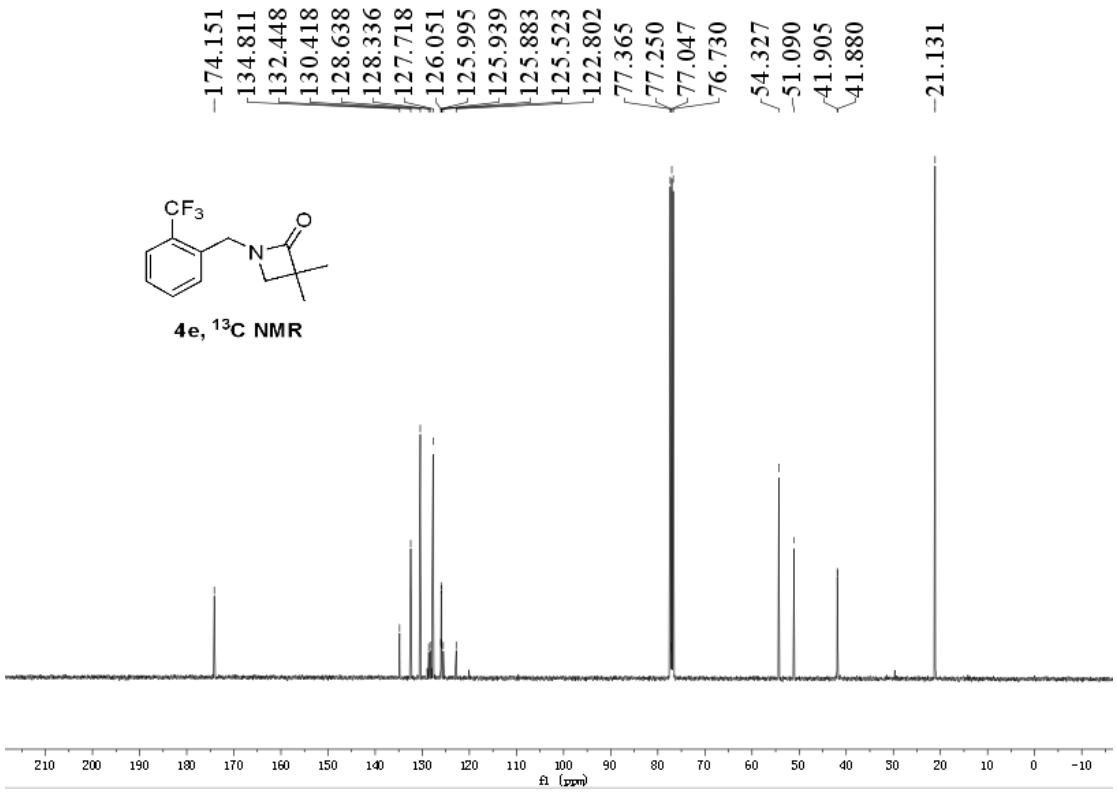
4d, DEPT 135

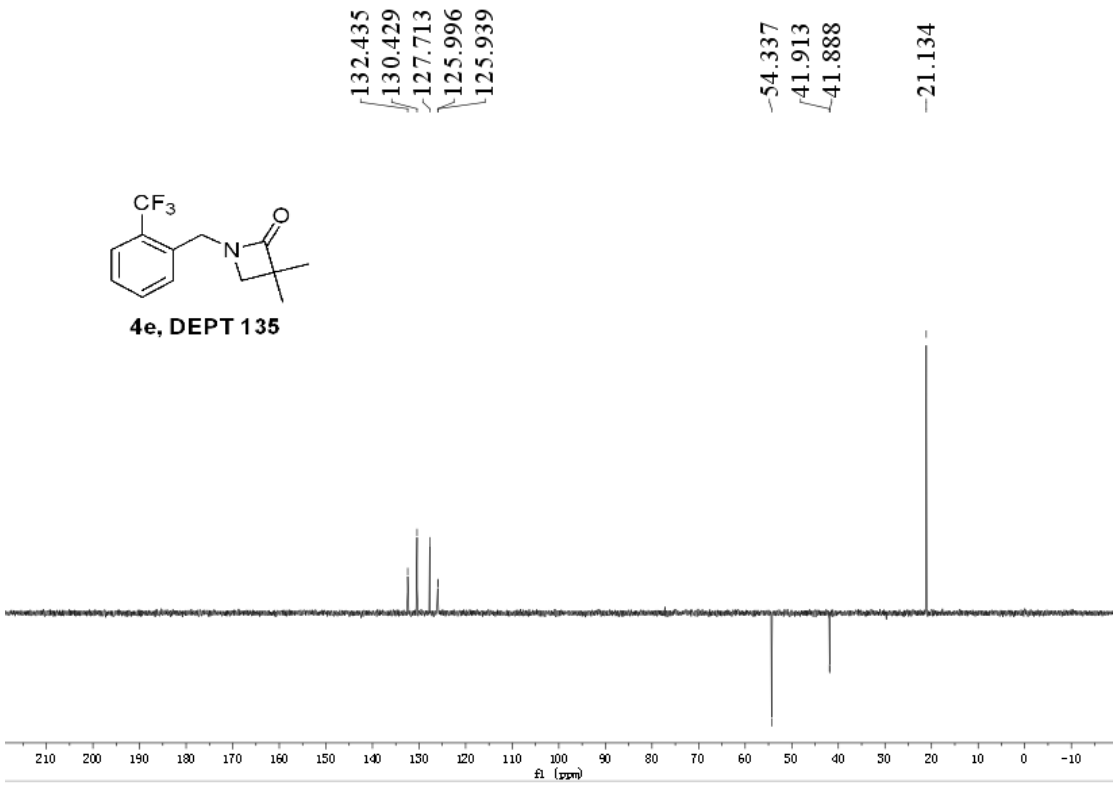


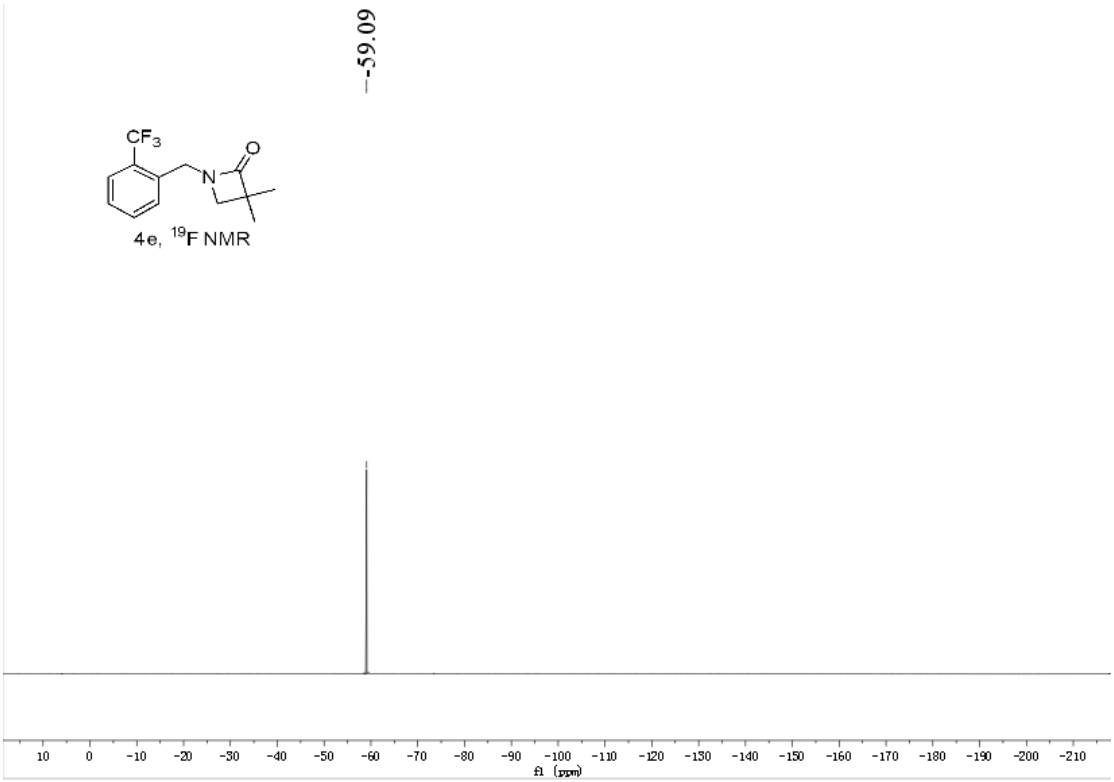
4d, ^{19}F NMR

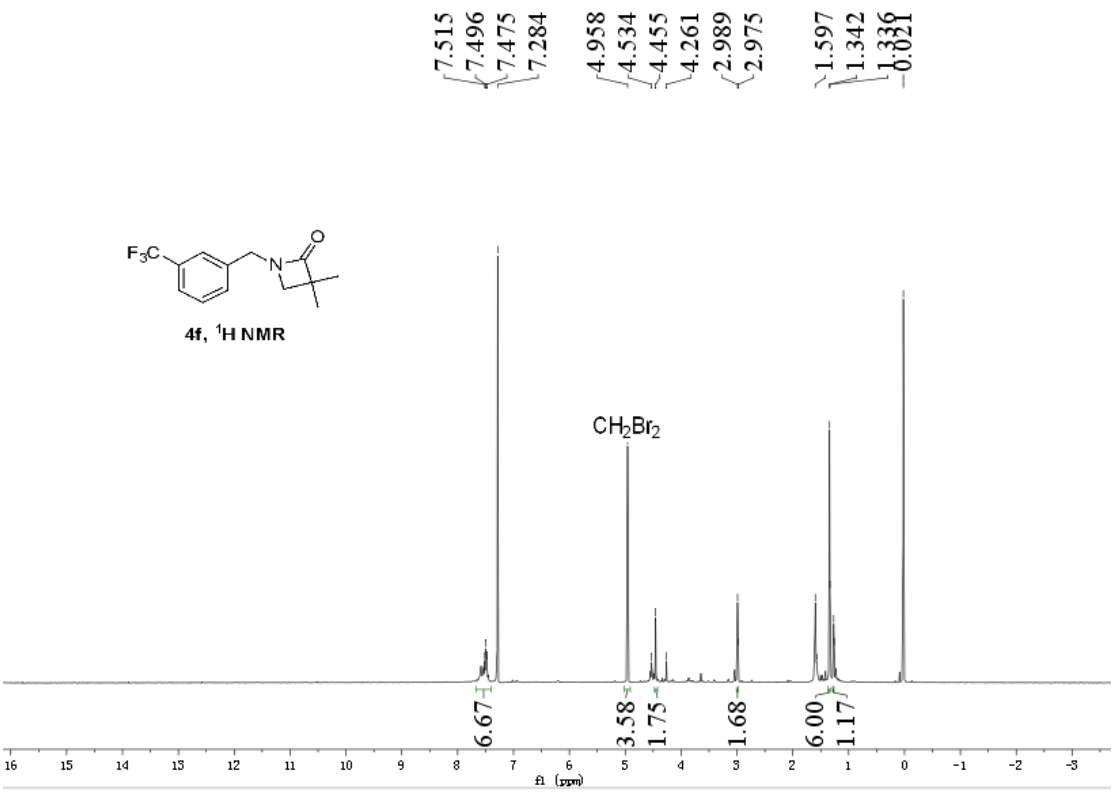

7.695
7.676
7.592
7.573
7.544
7.508
7.488
7.445
7.428
7.285
4.957
4.624
4.586
2.988
2.828
1.325
1.241
1.228
-0.021
CF3
O
N
4e,1H NMR
CH2Br2
1.79
1.55
1.73
2.09
3.23
2.00
1.99
5.78
1.78
f1 (ppm)

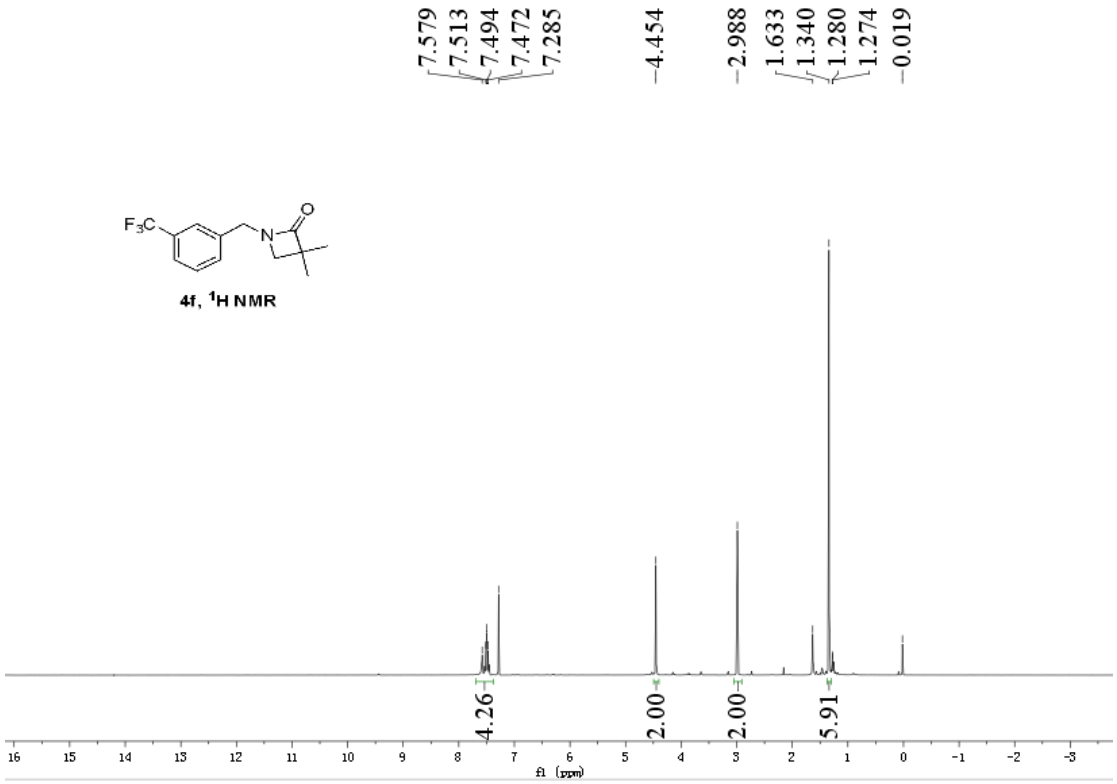

7.686
7.666
7.603
7.584
7.566
7.500
7.481
7.439
7.420
7.401
7.285
4.578
2.982
1.347
1.318
1.267
CF3
O
N
4e,1H NMR
1.00
1.02
0.99
0.96
2.10
2.06
6.26
f1 (ppm)


174.151
134.811
132.448
130.418
128.638
128.336
127.718
126.051
125.995
125.939
125.883
125.523
122.802
77.365
77.250
77.047
76.730
54.327
51.090
41.905
41.880
21.131
CF3
N
O
4e, 13C NMR
f1 (ppm)

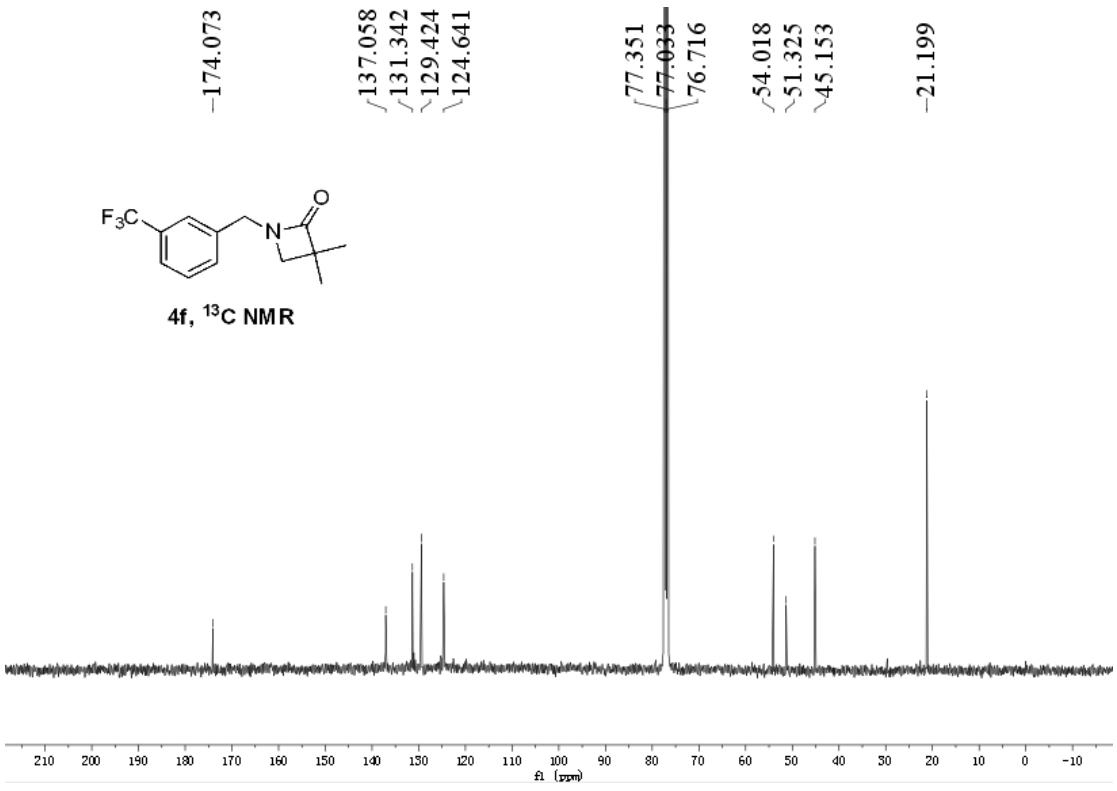

132.435
130.429
127.713
125.996
125.939
54.337
41.913
41.888
21.134
CF3
N
O
4e, DEPT 135
f1 (ppm)


-59.09
CF3
N
O
4e, 19F NMR
f1 (ppm)


7.515
7.496
7.475
7.284
4.958
4.534
4.455
4.261
2.989
2.975
1.597
1.342
1.336
0.021
F3C
N
O
4f, 1H NMR
CH2Br2
6.67
3.58
1.75
1.68
6.00
1.17
f1 (ppm)

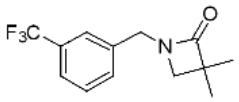


4f, ^{1}H NMR

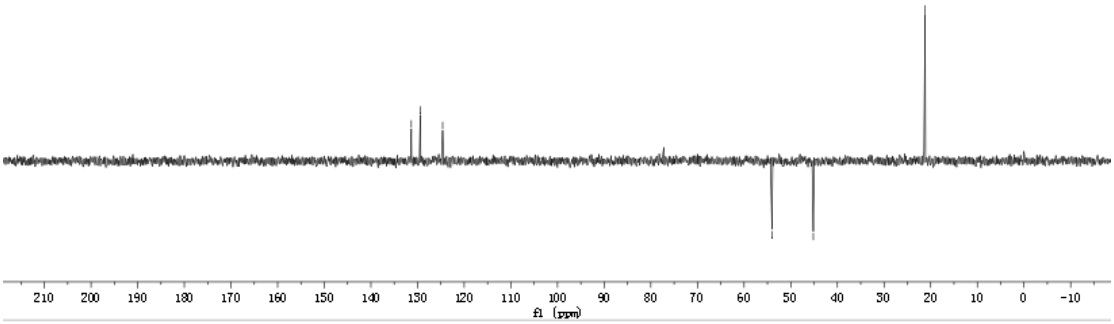


4f, ^{13}C NMR

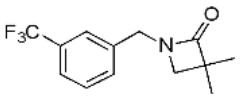
131.337
129.422
124.621
54.025
45.160
21.195



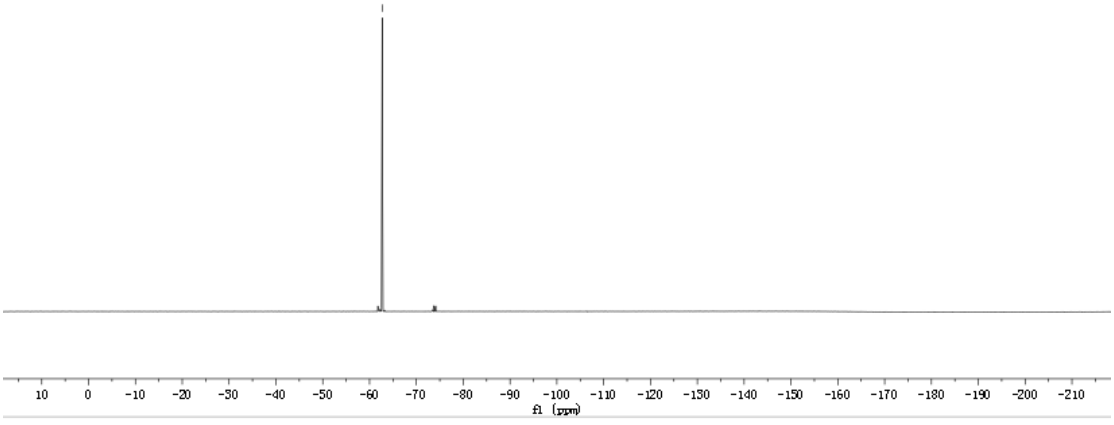
4f, DEPT 135

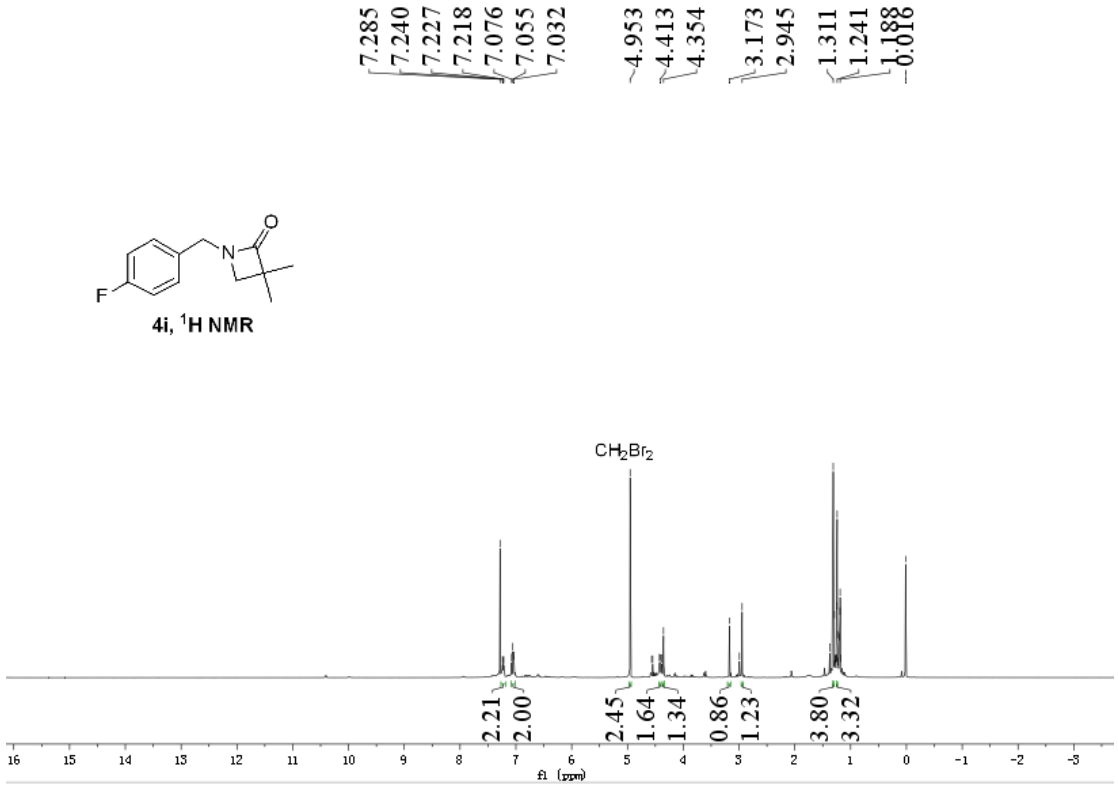


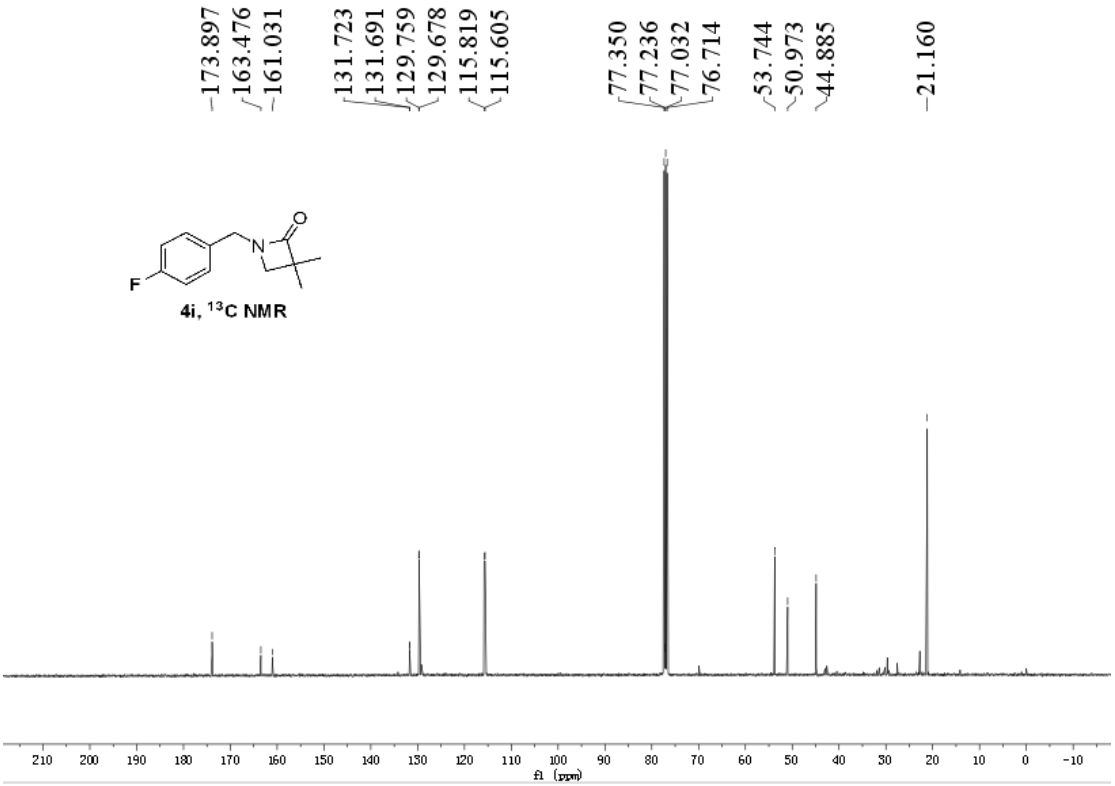
-62.689



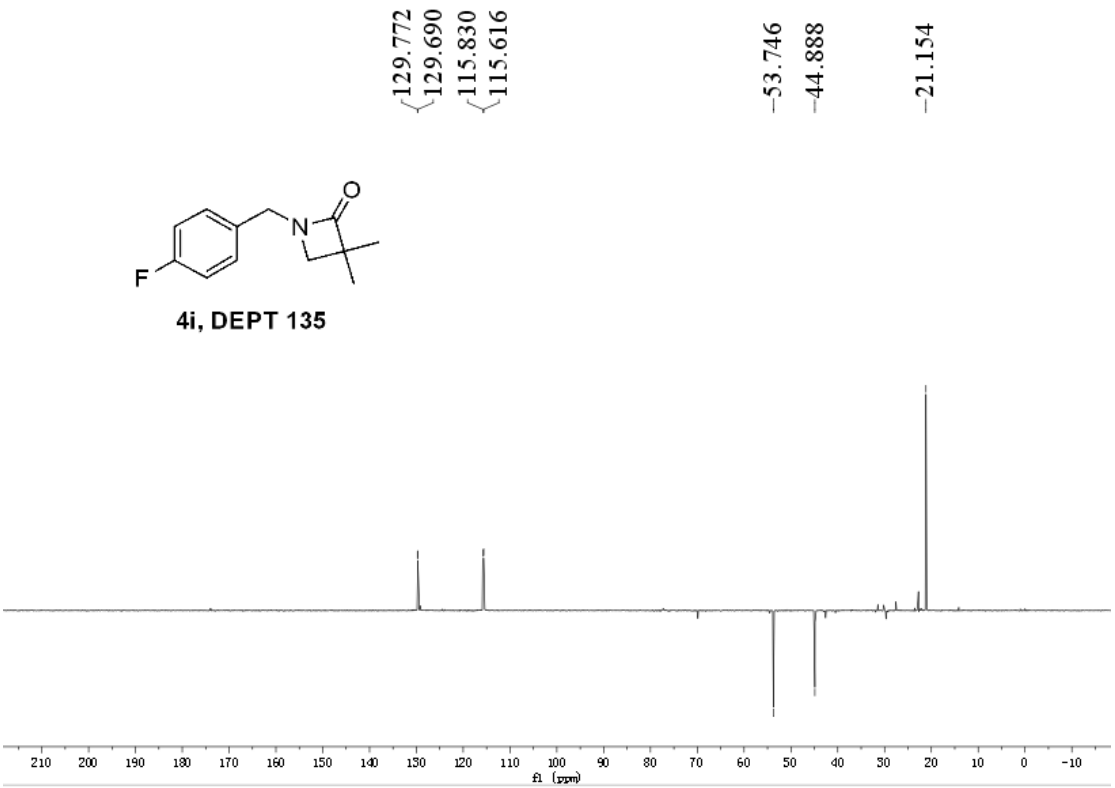
4f, ^{19}F NMR



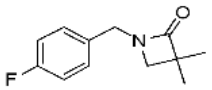

7.285
7.240
7.227
7.218
7.076
7.055
7.032
4.953
4.413
4.354
3.173
2.945
1.311
1.241
1.188
0.018
F
O
N
4i, ^{1}H NMR
CH_2Br_2
2.21
2.00
2.45
1.64
1.34
0.86
1.23
3.80
3.32
f1 (ppm)
7.284
7.239
7.226
7.218
7.204
7.075
7.053
7.031
4.352
2.944
1.310
1.301
1.271
1.222
0.016
F
O
N
4i, ^{1}H NMR
2.02
2.07
1.95
2.00
6.22
f1 (ppm)



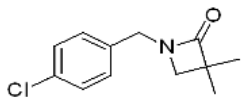
4i, ^{13}C NMR



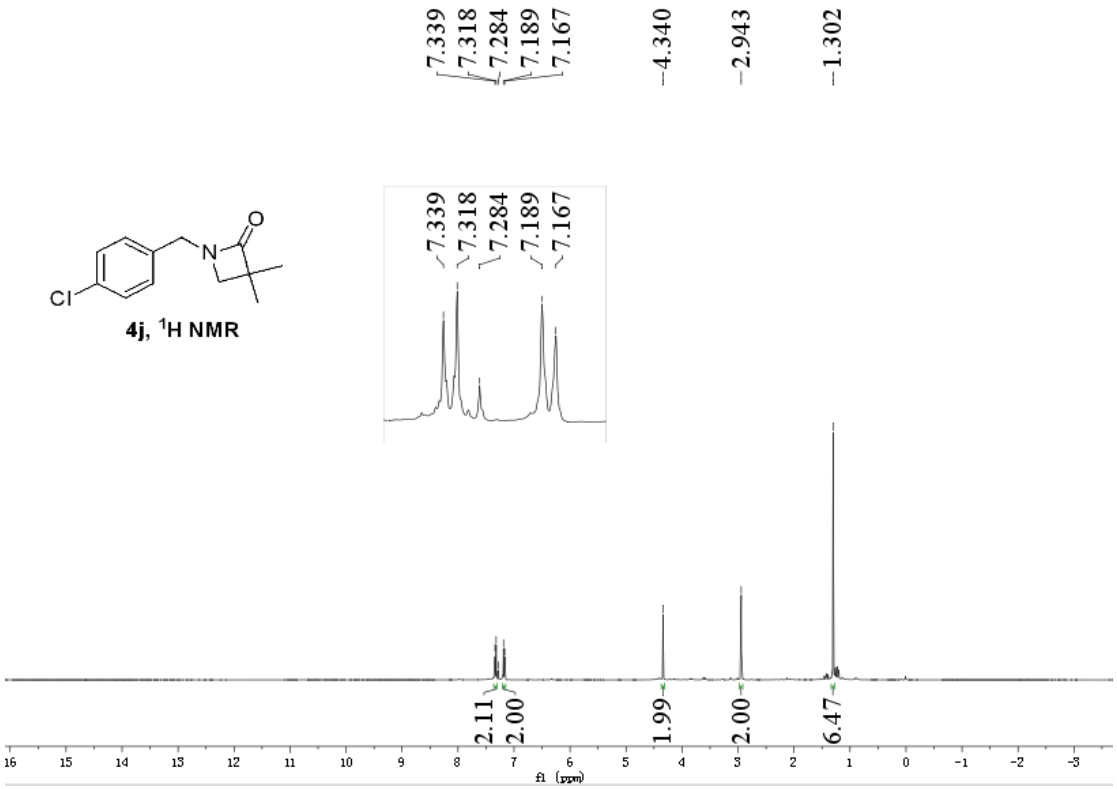
4i, DEPT 135

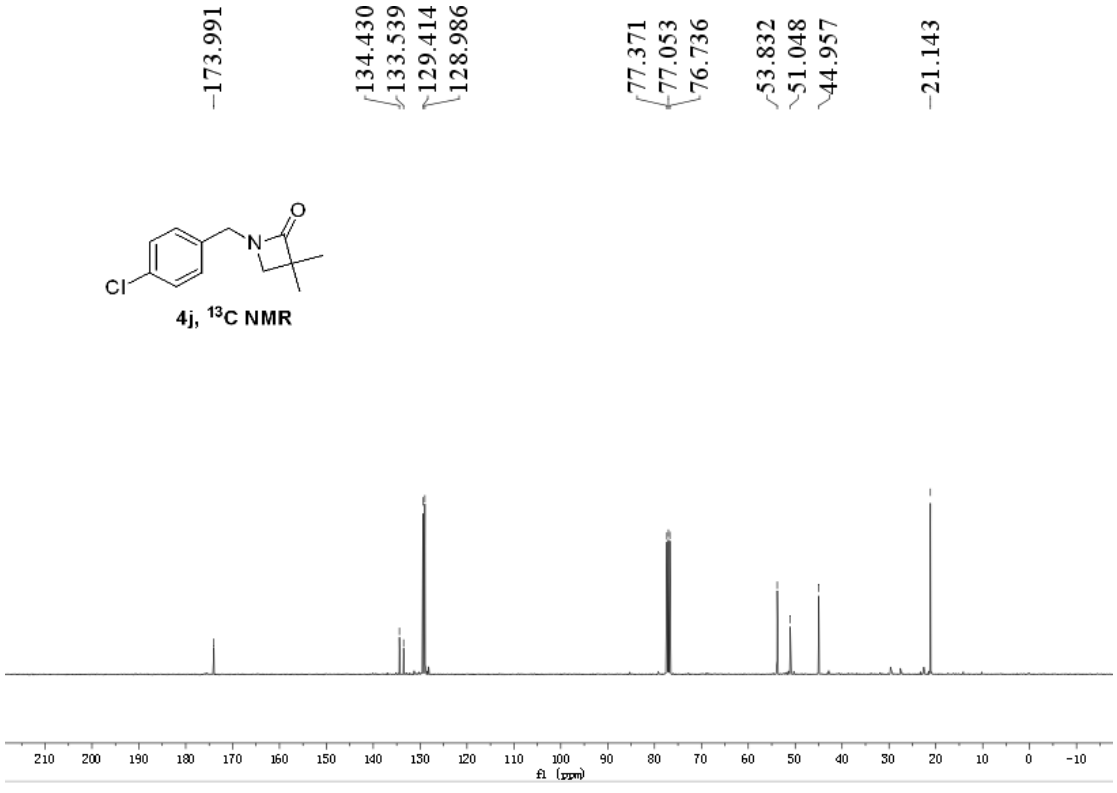


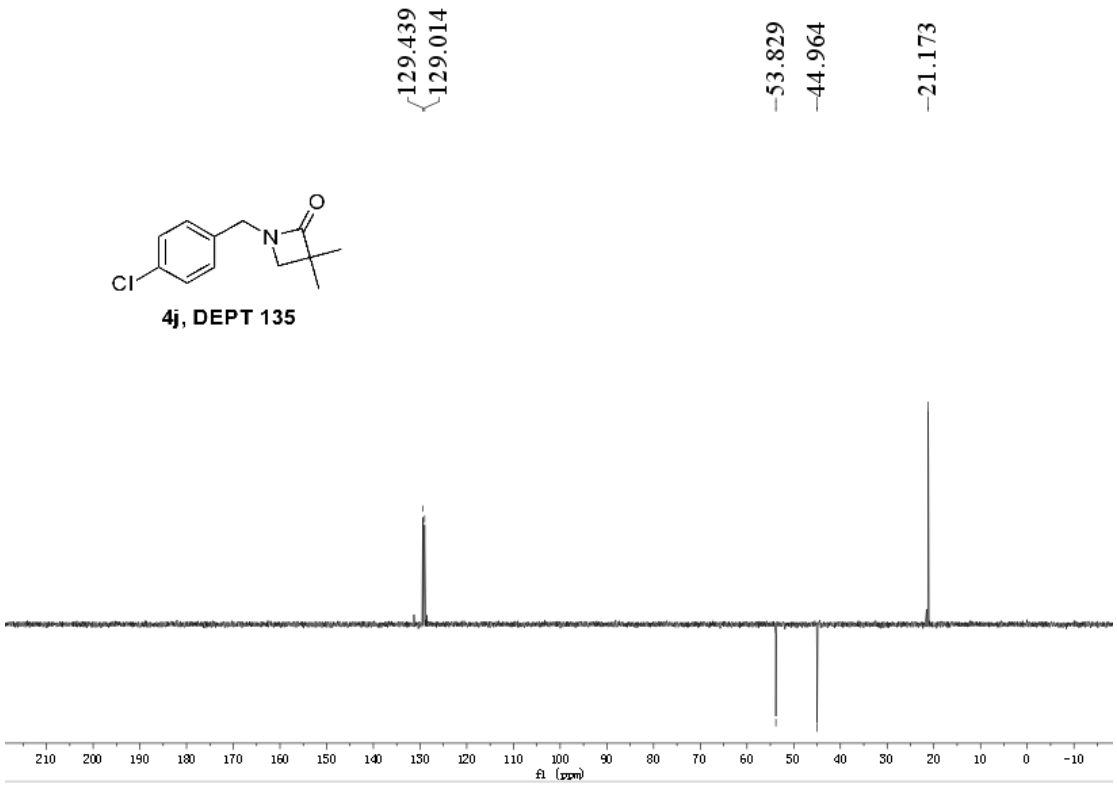
4i, ^{19}F NMR



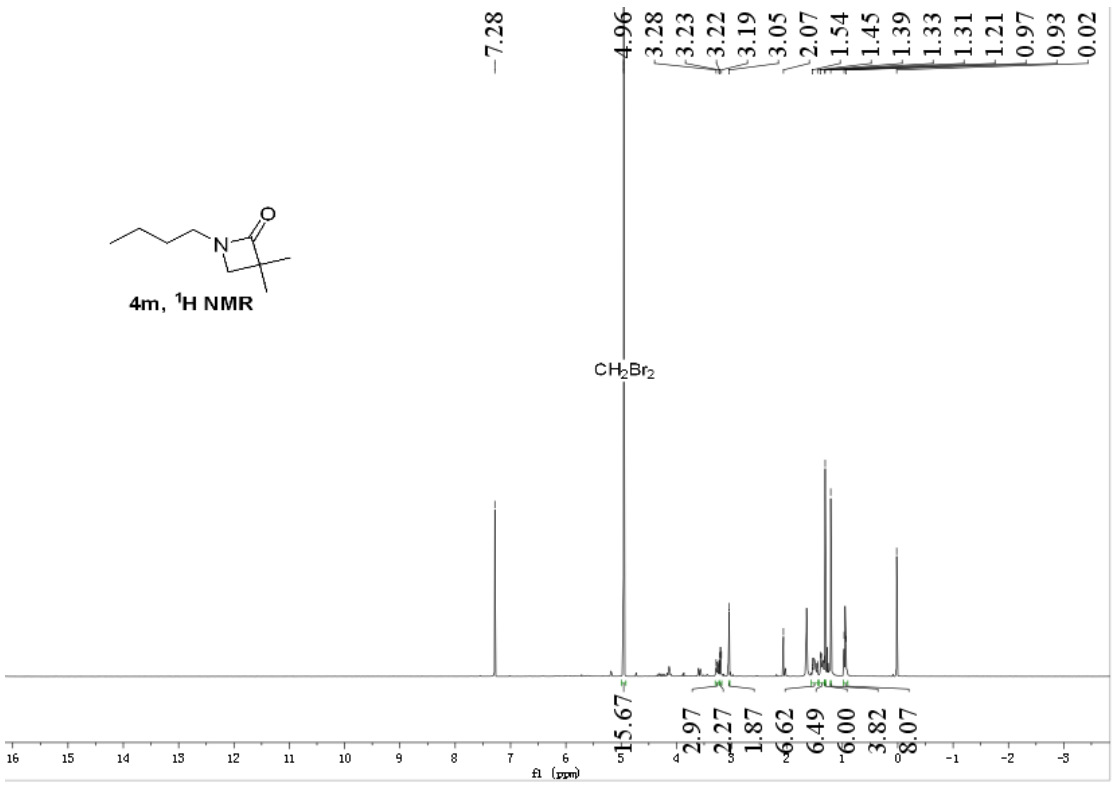
4j, ^{1}H NMR


7.339
7.318
7.284
7.189
7.167
4.340
2.943
1.302
Cl
O
N
4j, ^{1}H NMR
2.11
2.00
1.99
2.00
6.47
f1 (ppm)

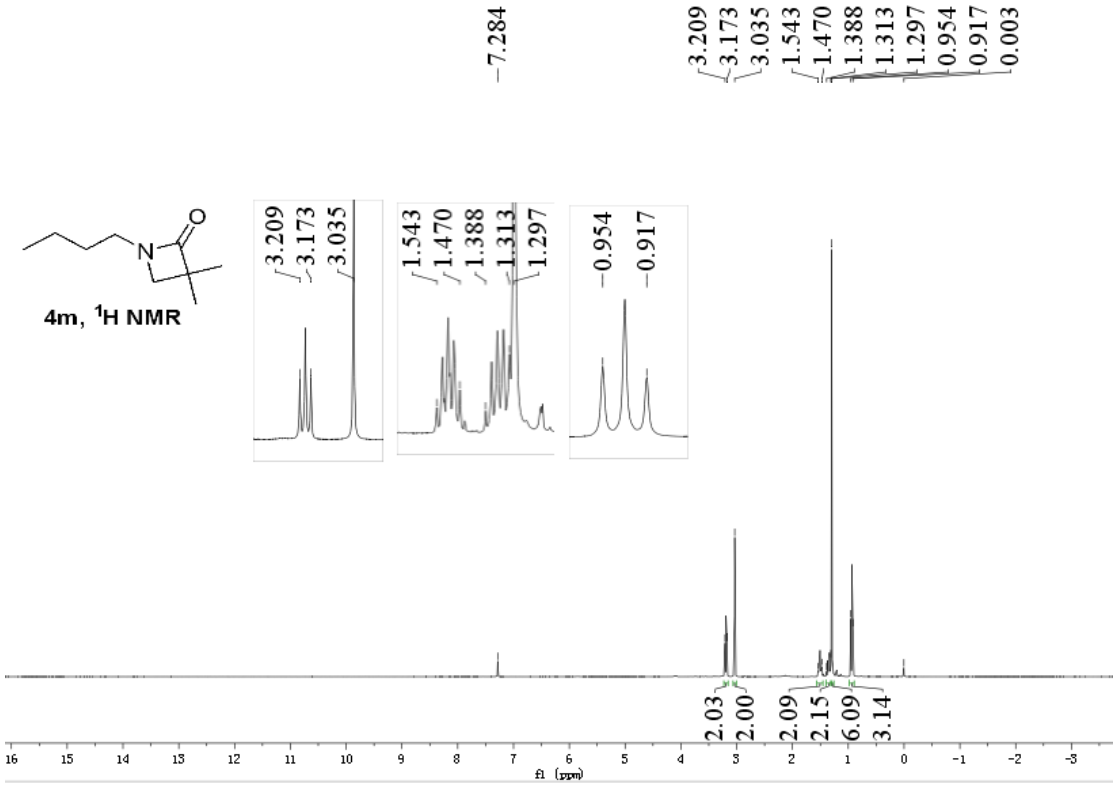

173.991
134.430
133.539
129.414
128.986
77.371
77.053
76.736
53.832
51.048
44.957
21.143
Cl
O
N
4j, ^{13}C NMR
f1 (ppm)

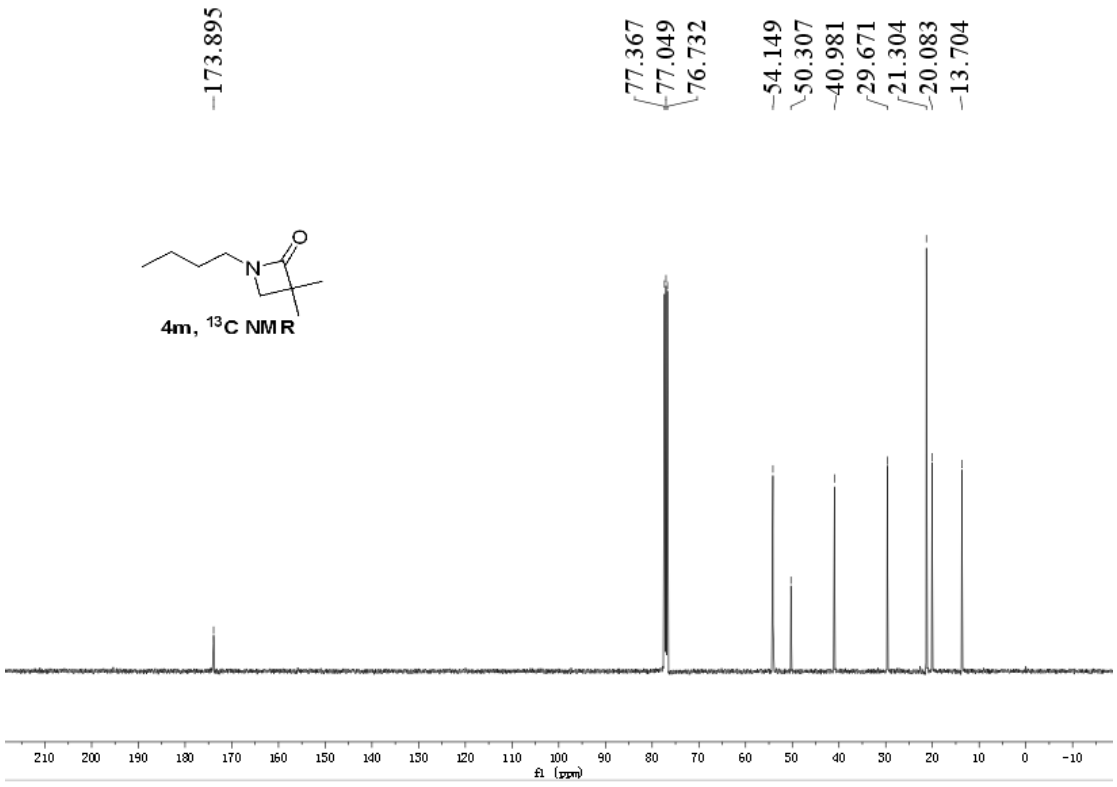


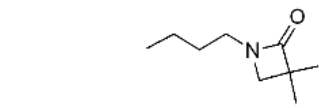
4j, DEPT 135



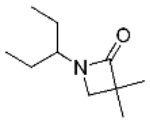
4m, ^{1}H NMR


7.284
3.209
3.173
3.035
1.543
1.470
1.388
1.313
1.297
0.954
0.917
0.003
4m, 1H NMR
2.03
2.00
2.09
2.15
6.09
3.14
f1 (ppm)

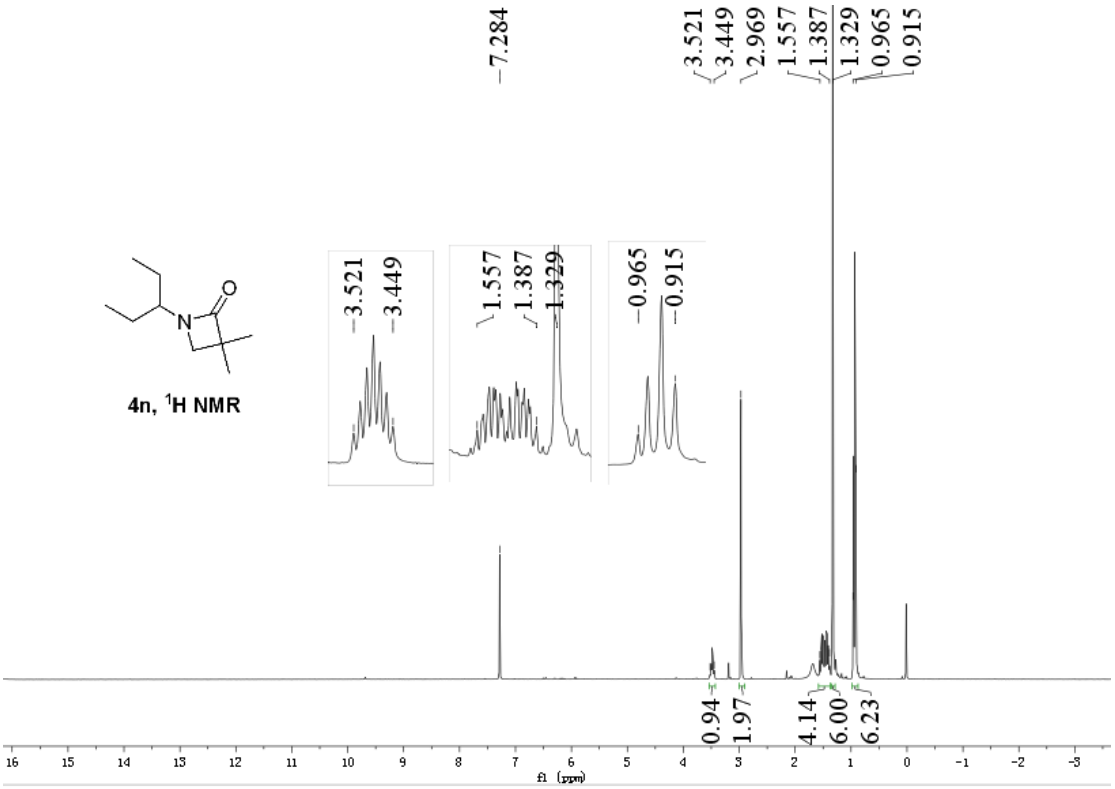

173.895
77.367
77.049
76.732
54.149
50.307
40.981
29.671
21.304
20.083
13.704
4m, 13C NMR
f1 (ppm)



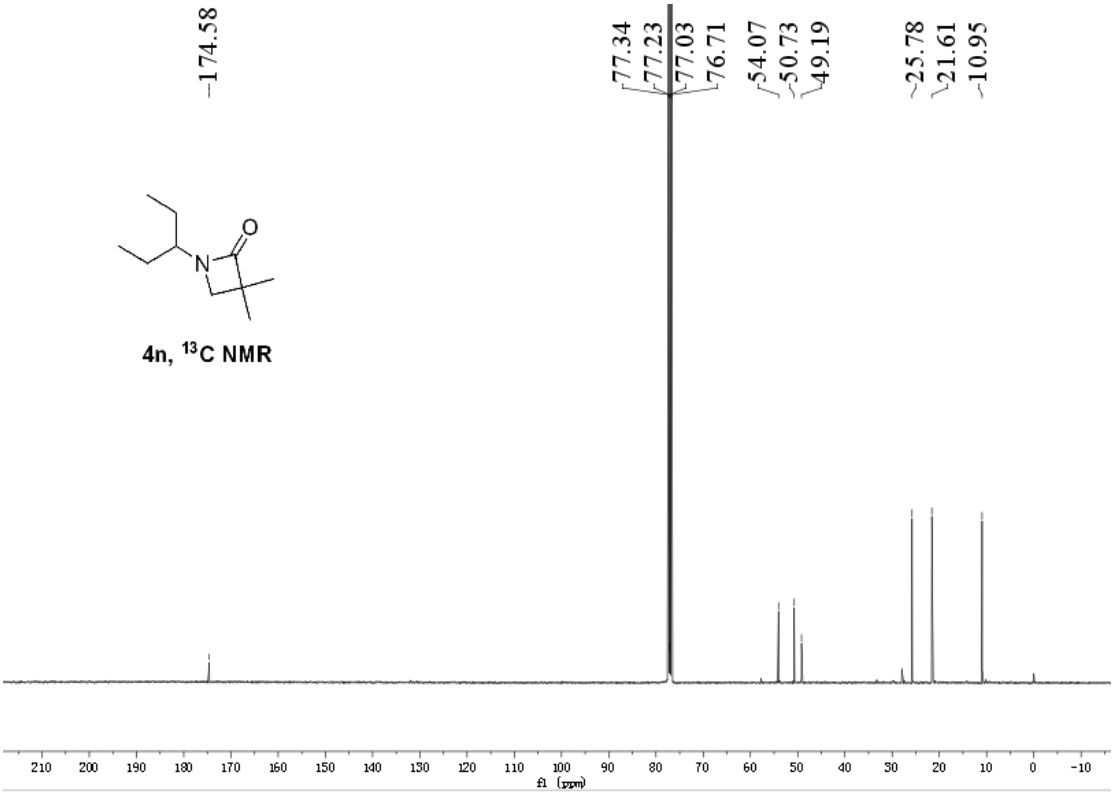
4m, DEPT 135



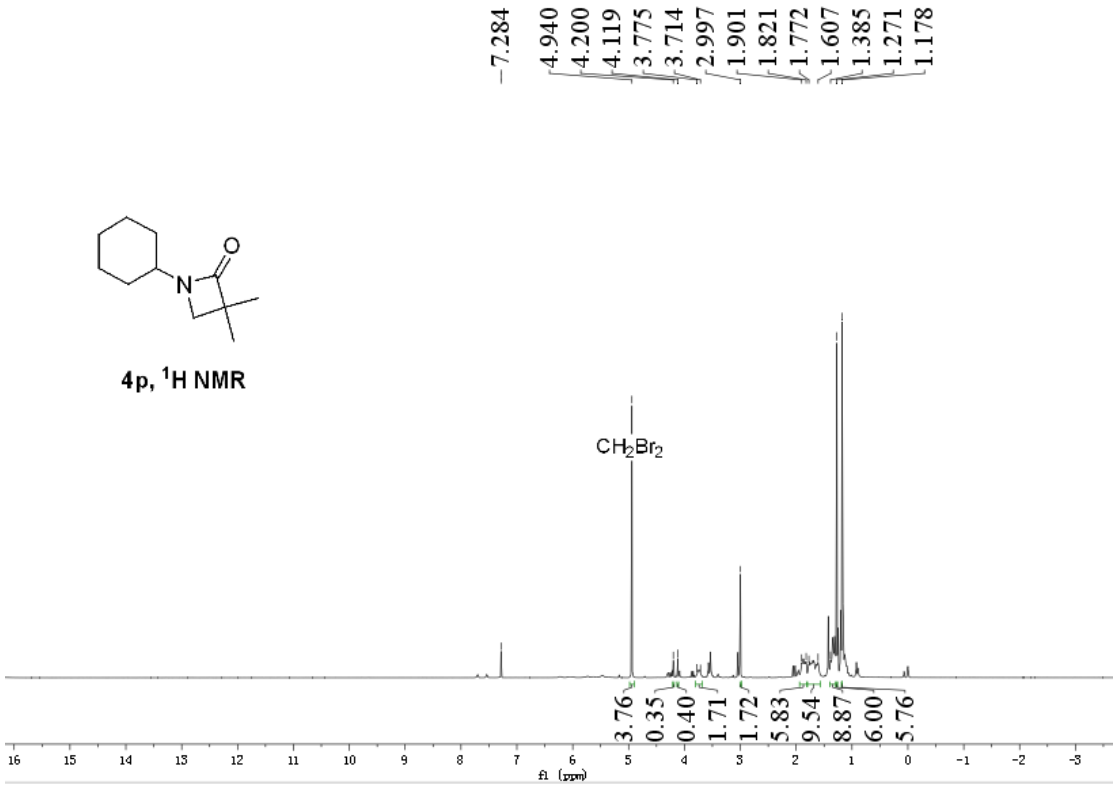
4n, ^{1}H NMR

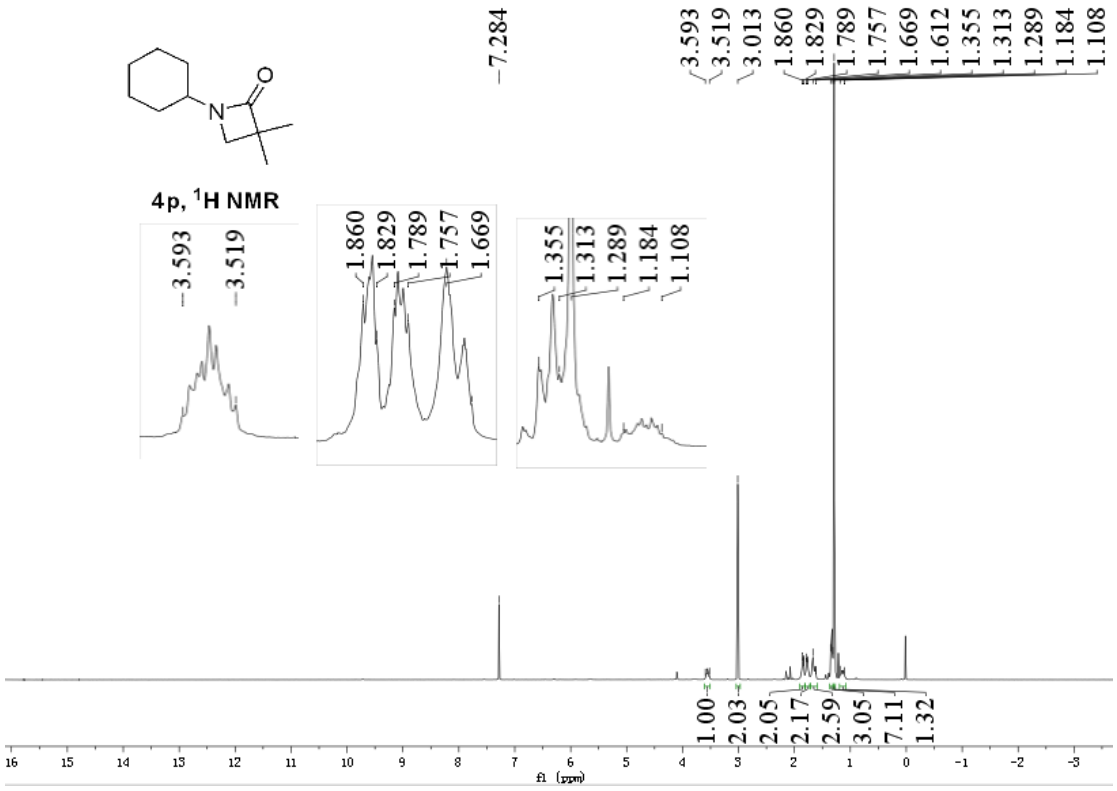


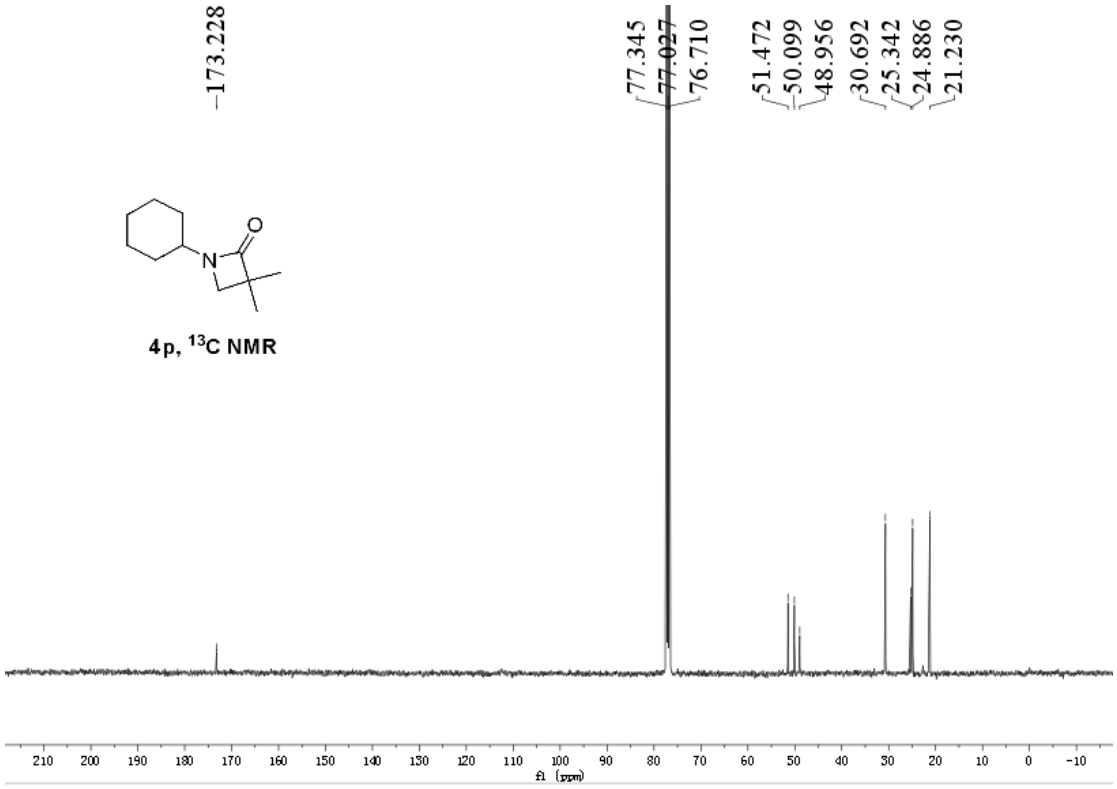
4n, ^{1}H NMR



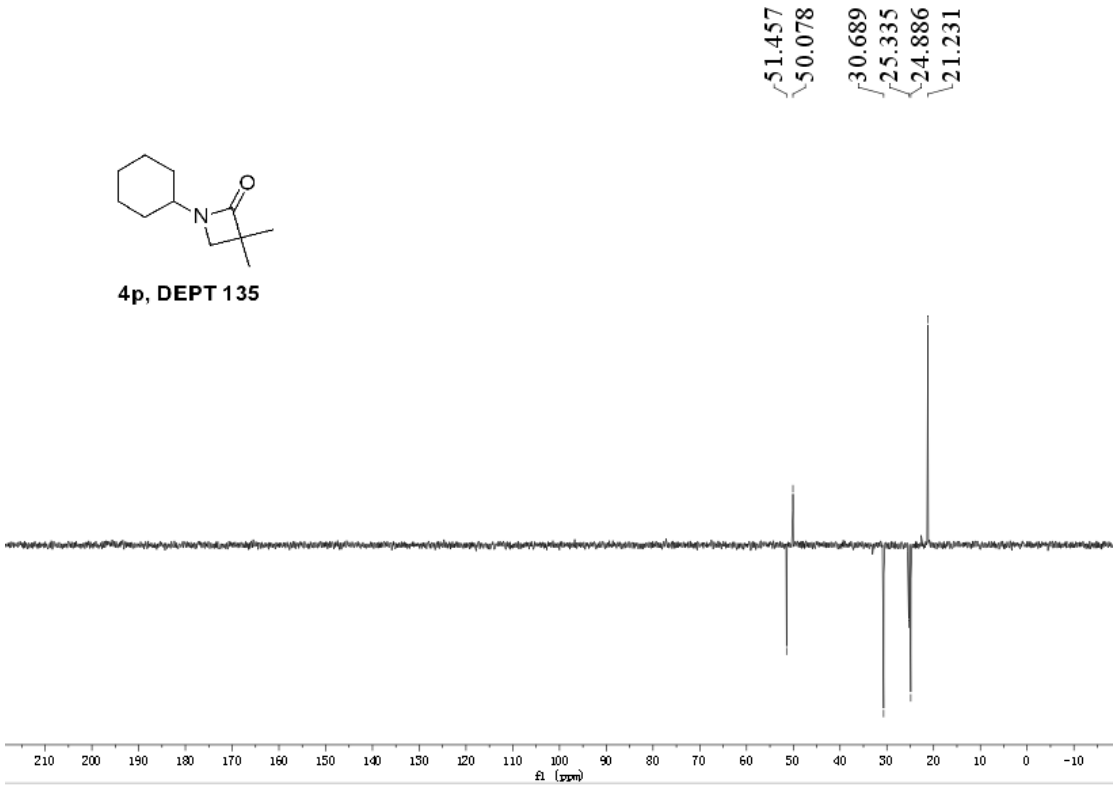
4n, ^{13}C NMR


7.284
4.940
4.200
4.119
3.775
3.714
2.997
1.901
1.821
1.772
1.607
1.385
1.271
1.178
O
N
4p, ^{1}H NMR
CH_2Br_2
3.76
0.35
0.40
1.71
1.72
5.83
9.54
8.87
6.00
5.76
f1 (ppm)

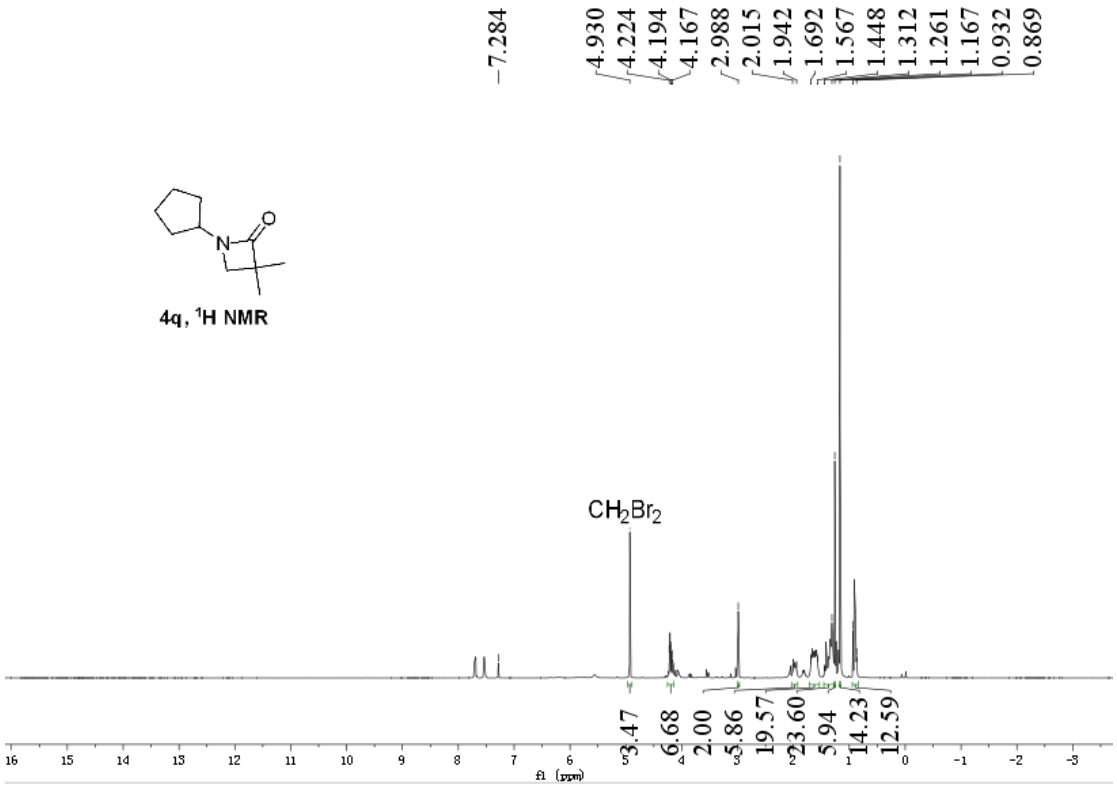

7.284
3.593
3.519
3.013
1.860
1.829
1.789
1.757
1.669
1.612
1.355
1.313
1.289
1.184
1.108
O
N
4p, ^{1}H NMR
3.593
3.519
1.860
1.829
1.789
1.757
1.669
1.355
1.313
1.289
1.184
1.108
1.00
2.03
2.05
2.17
2.59
3.05
7.11
1.32
f1 (ppm)

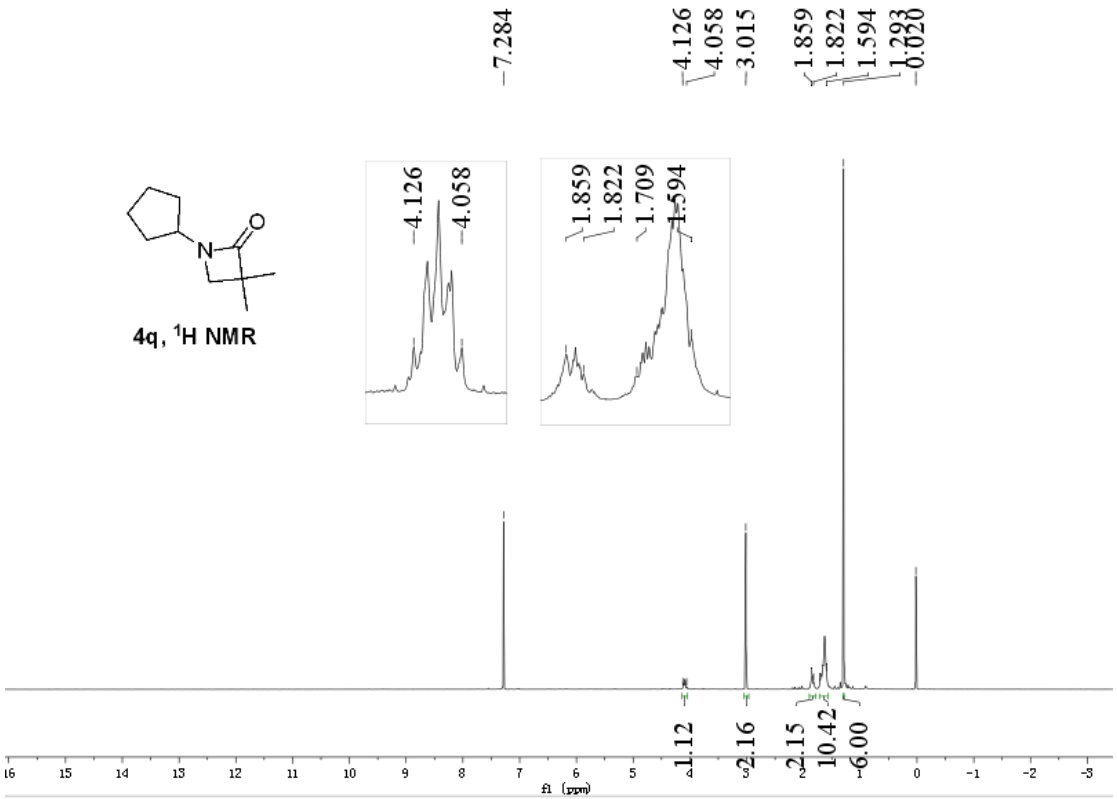


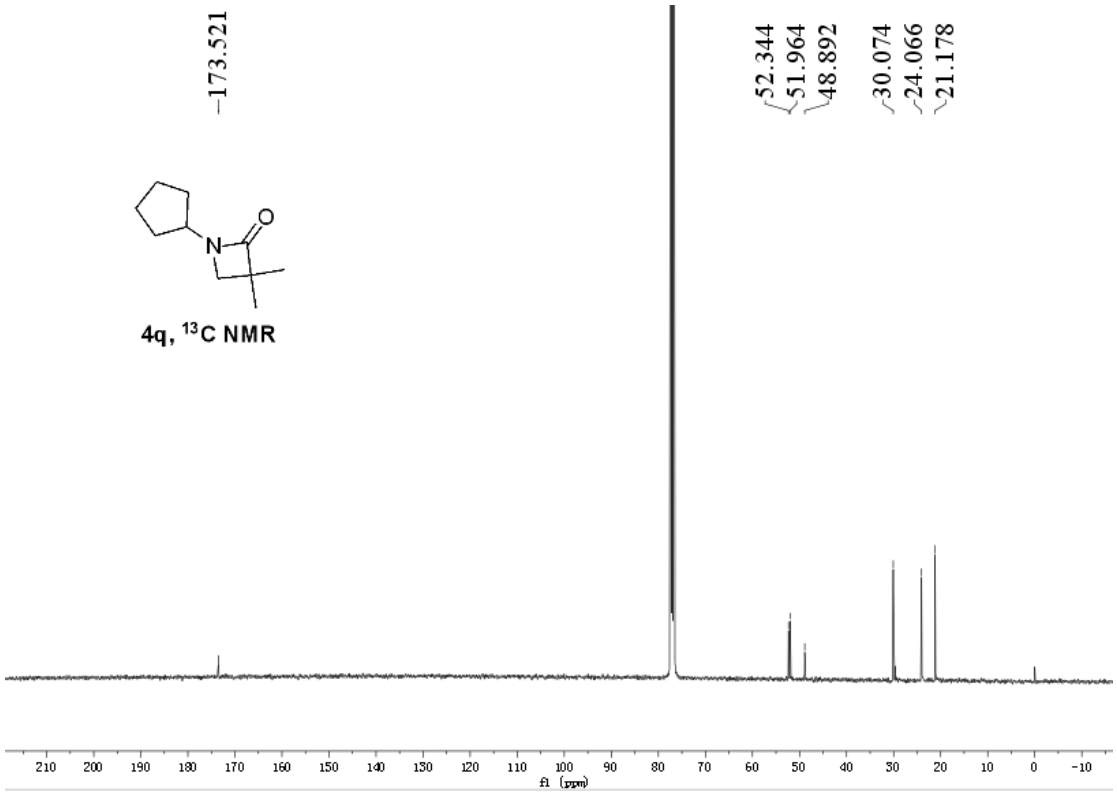
4p, ^{13}C NMR

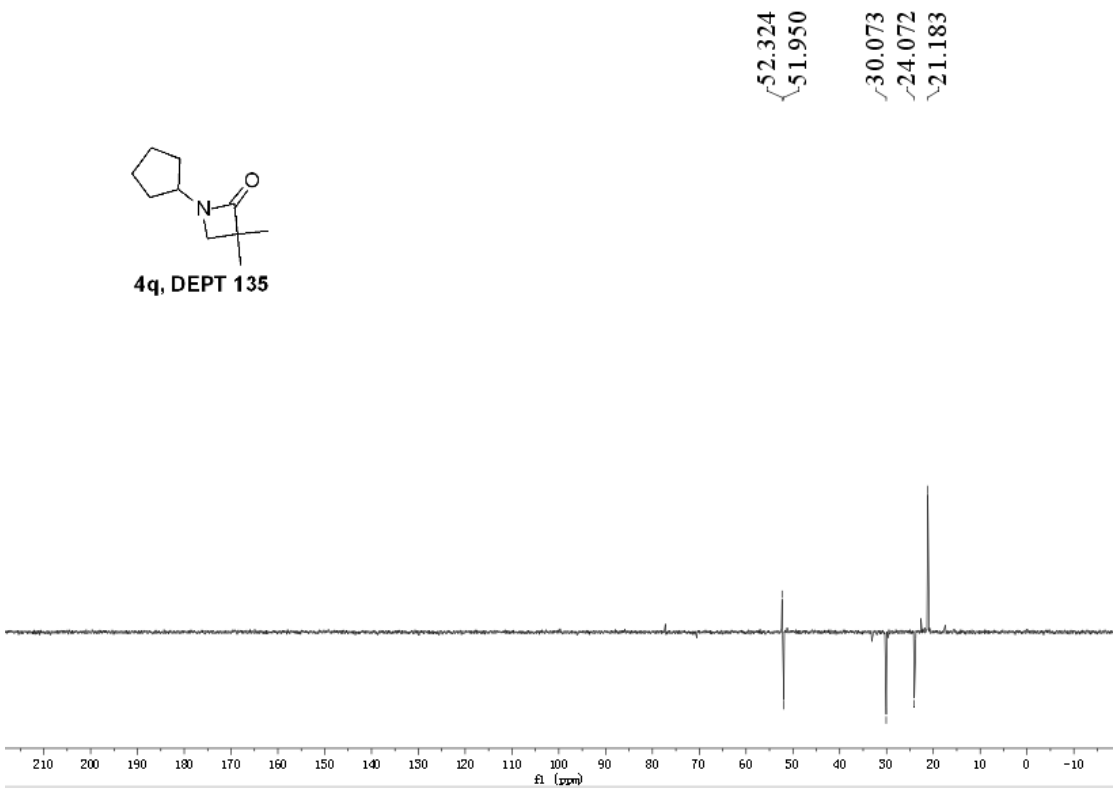


4p, DEPT 135

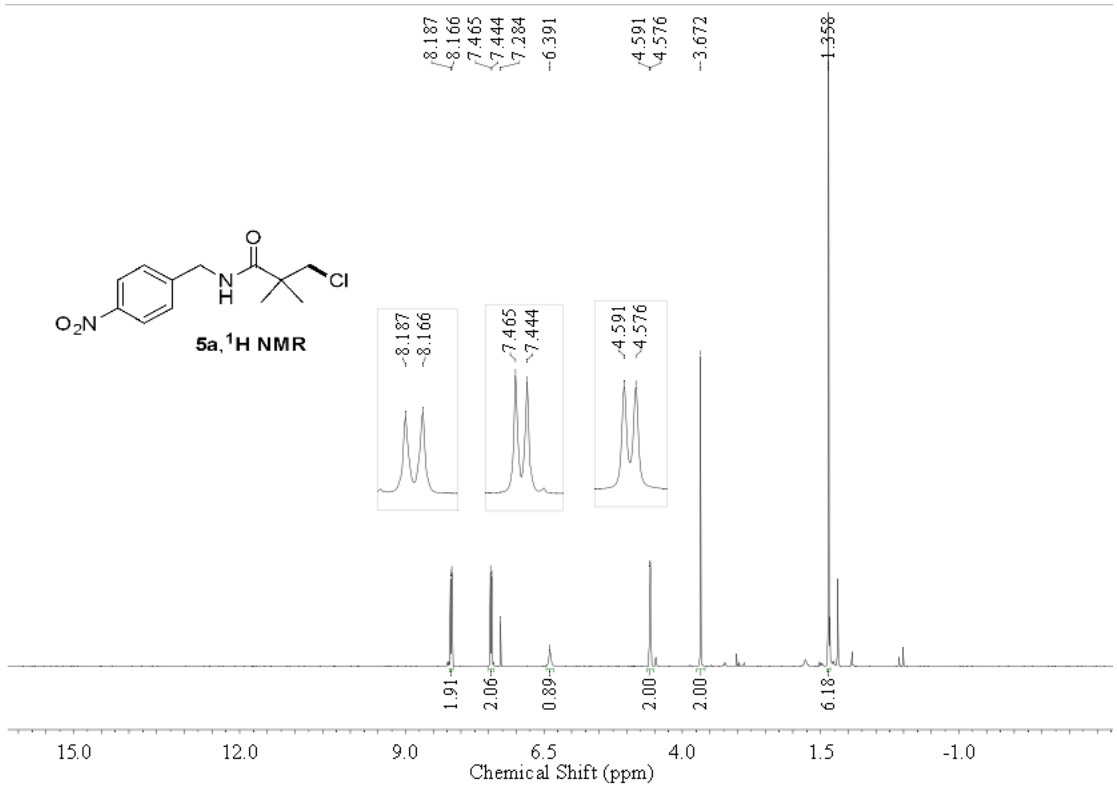

7.284
4.930
4.224
4.194
4.167
2.988
2.015
1.942
1.692
1.567
1.448
1.312
1.261
1.167
0.932
0.869
4q, 1H NMR
CH2Br2
3.47
6.68
2.00
5.86
19.57
23.60
5.94
14.23
12.59
f1 (ppm)

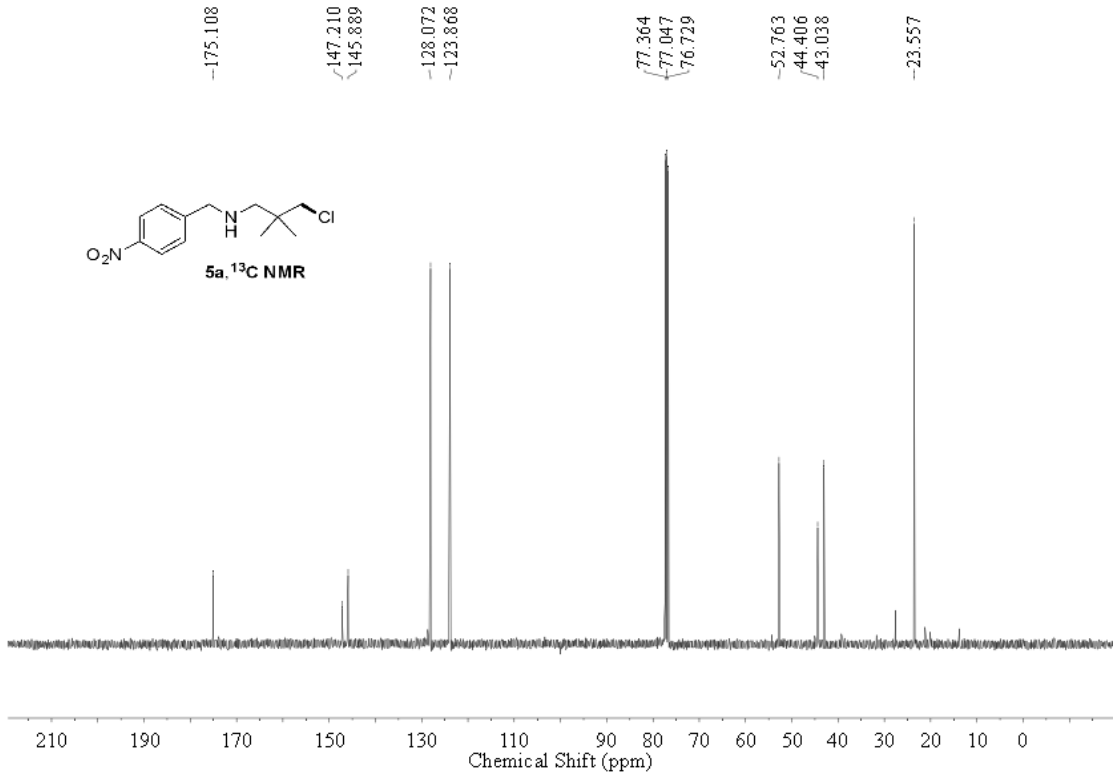

7.284
4.126
4.058
3.015
1.859
1.822
1.594
1.293
0.020
4q, 1H NMR
4.126
4.058
1.859
1.822
1.709
1.594
1.12
2.16
2.15
10.42
6.00
f1 (ppm)

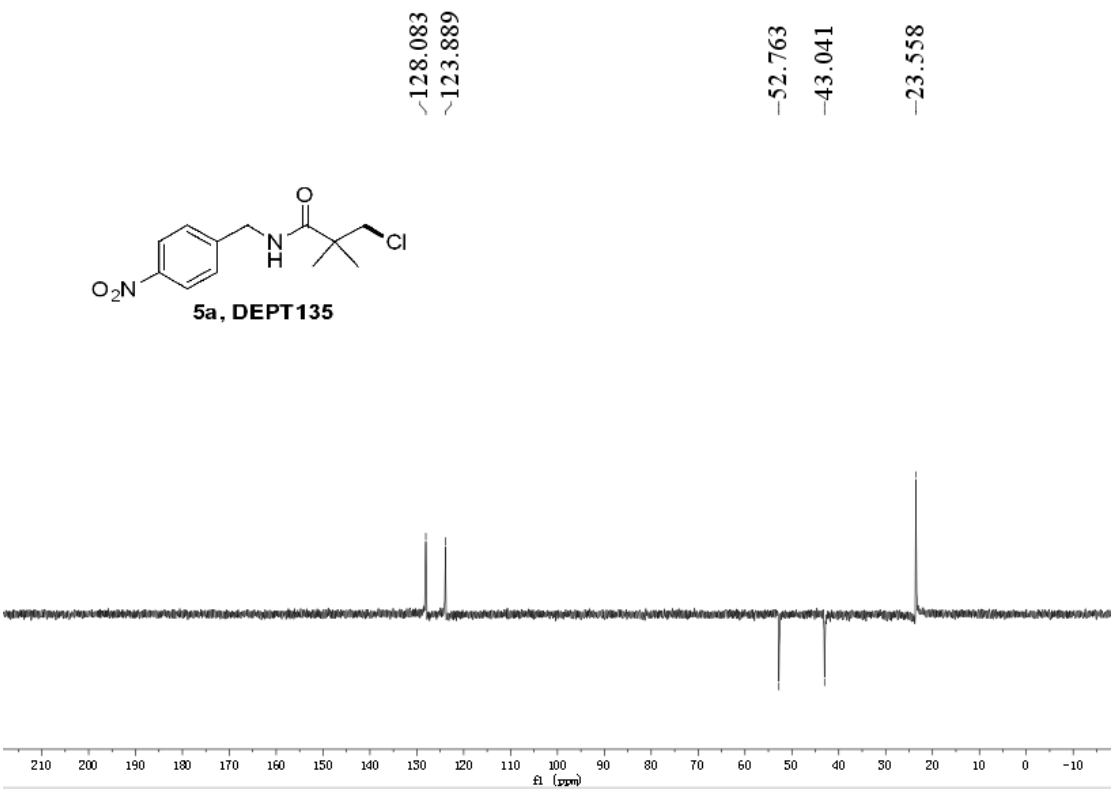


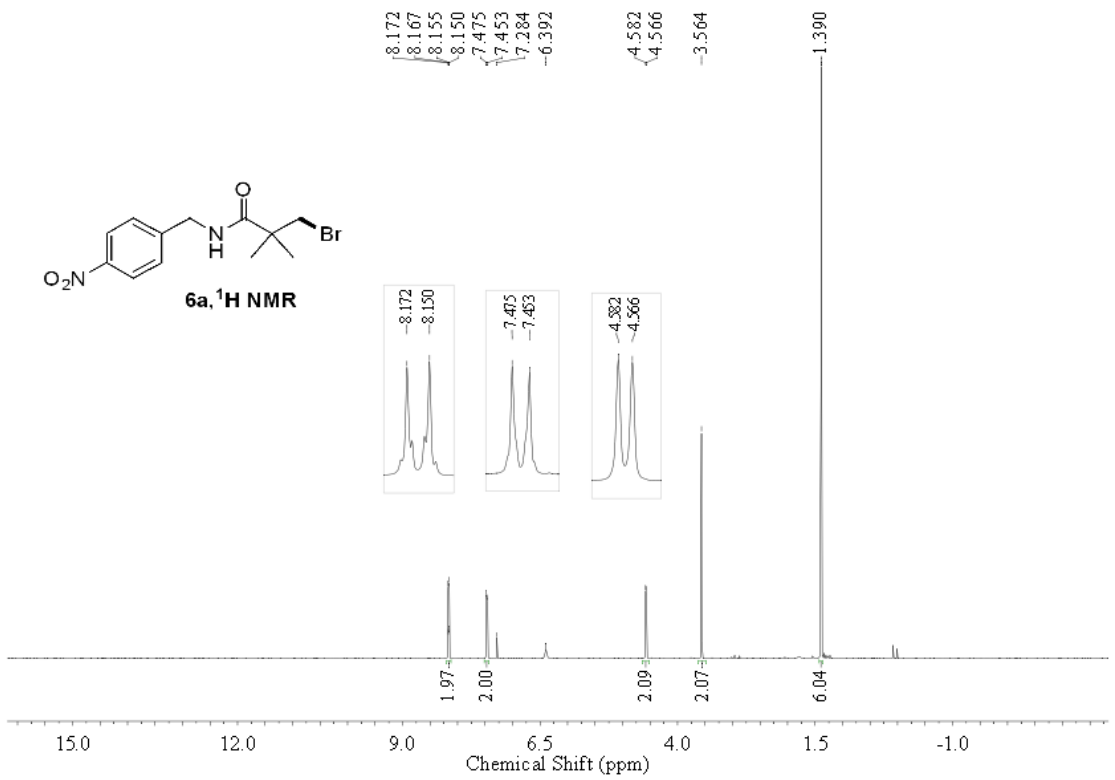


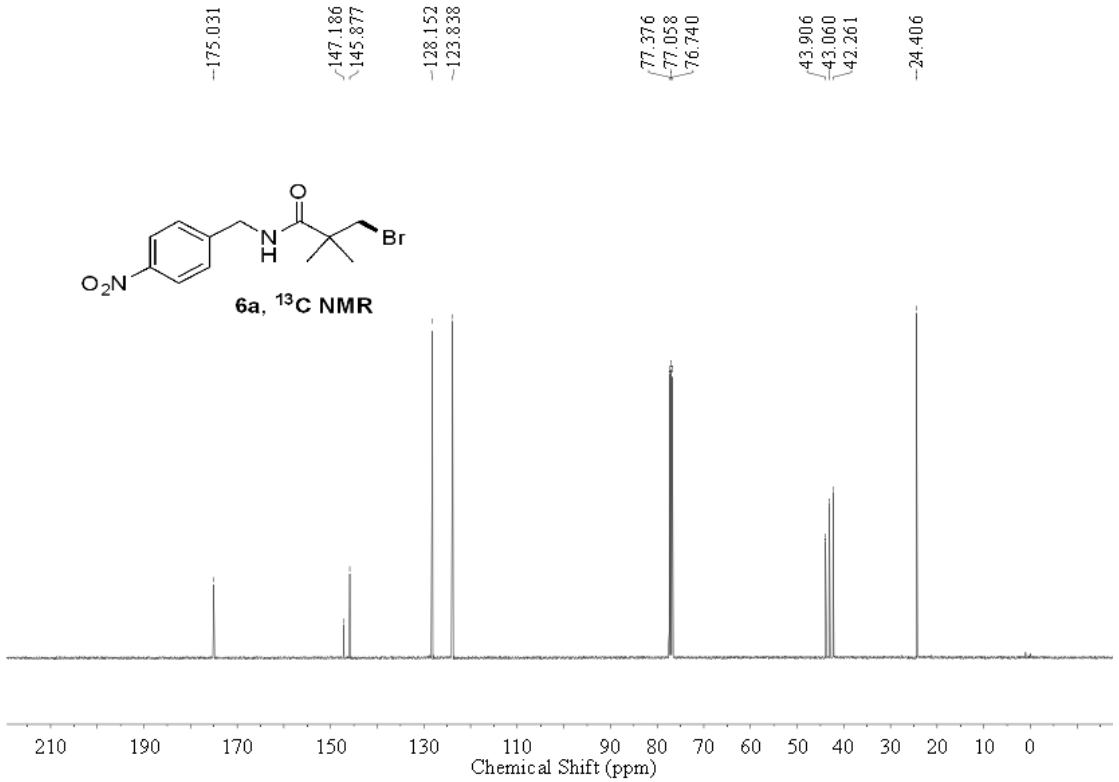
(5) Other halogenated amides

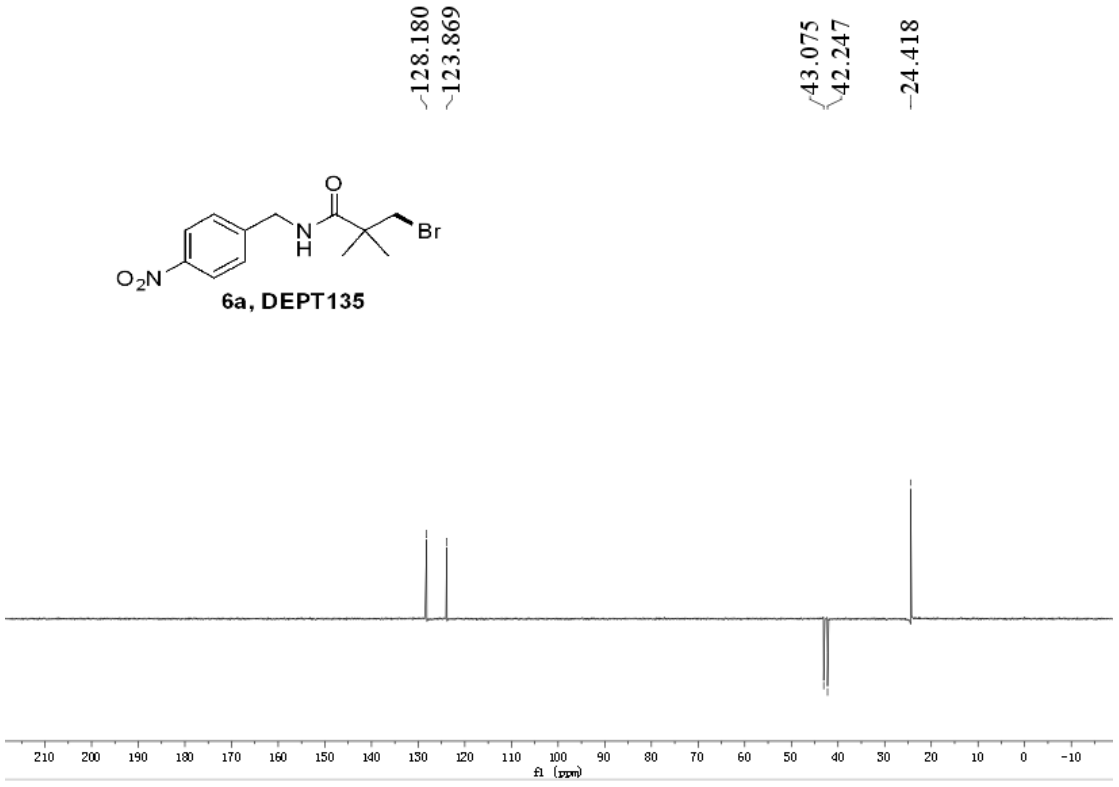

8.187
8.166
7.465
7.444
7.284
6.391
4.591
4.576
3.672
1.358
O
N
H
Cl
O2N
5a, 1H NMR
1.91
2.06
0.89
2.00
2.00
6.18
15.0
12.0
9.0
6.5
4.0
1.5
-1.0
Chemical Shift (ppm)

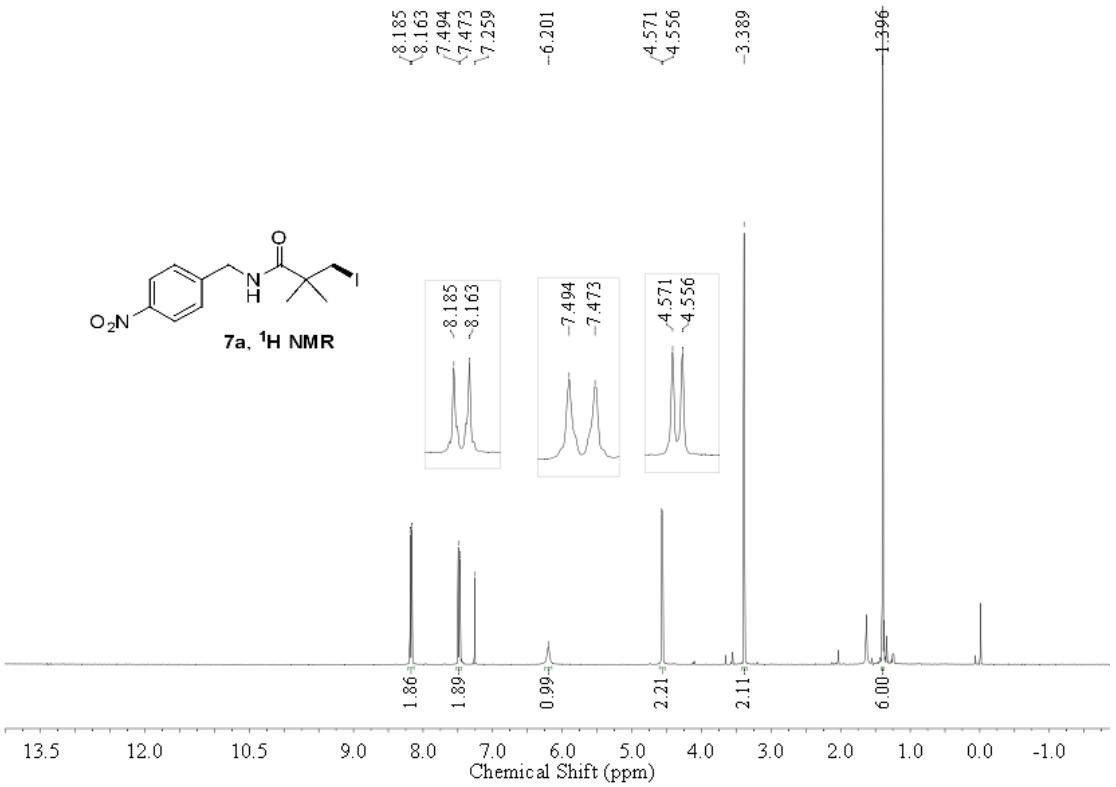

175.108
147.210
145.889
128.072
123.868
77.364
77.047
76.729
52.763
44.406
43.038
23.557
N
H
Cl
O2N
5a, 13C NMR
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

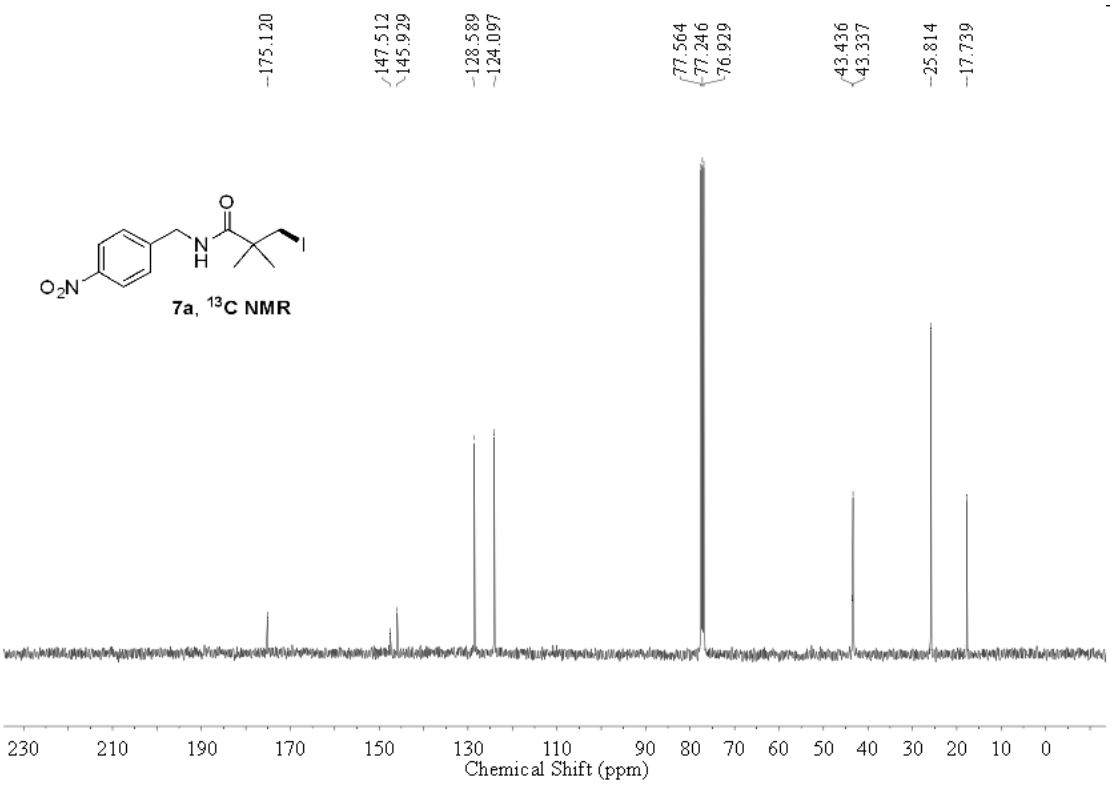

128.083
123.889
52.763
43.041
23.558
O
N
H
Cl
O2N
5a, DEPT135
f1 (ppm)

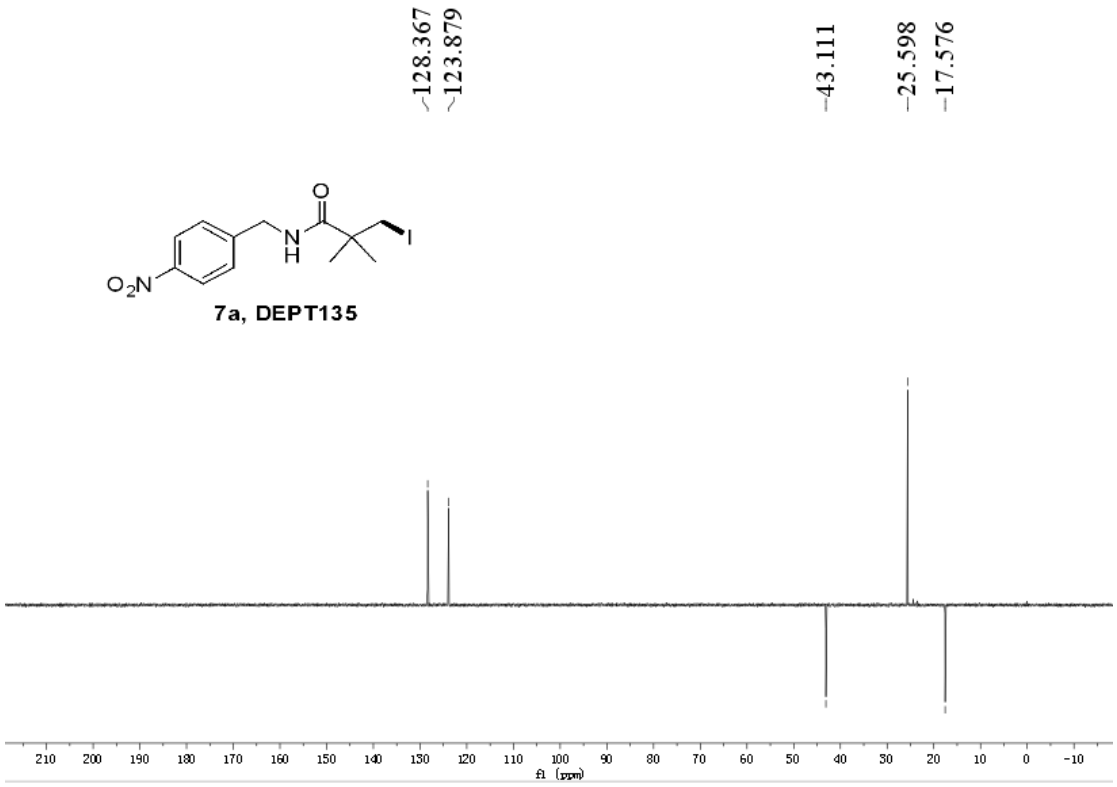

8.172
8.167
8.155
8.150
7.475
7.453
7.284
6.392
4.582
4.566
3.564
1.390
O
N
H
Br
O2N
6a, 1H NMR
1.97
2.00
2.09
2.07
6.04
15.0
12.0
9.0
6.5
4.0
1.5
-1.0
Chemical Shift (ppm)


175.031
147.186
145.877
128.152
123.838
77.376
77.058
76.740
43.906
43.060
42.261
24.406
O
N
H
Br
O2N
6a, 13C NMR
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)


128.180
123.869
43.075
42.247
24.418
O
N
H
Br
O2N
6a, DEPT135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.185
8.163
7.494
7.473
7.259
6.201
4.571
4.556
3.389
1.396
7a, 1H NMR
1.86
1.89
0.99
2.21
2.11
6.00
13.5
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
Chemical Shift (ppm)


175.120
147.512
145.929
128.589
124.097
77.564
77.246
76.929
43.436
43.337
25.814
17.739
7a, 13C NMR
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)


128.367
123.879
43.111
25.598
17.576
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O2N
7a, DEPT135
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)