

## Supporting Information

### **Electrochemical $\alpha$ -C(sp<sup>3</sup>)-H/O-H Cross-coupling of Isochromans and Alcohols Assisted by Benzoic Acid**

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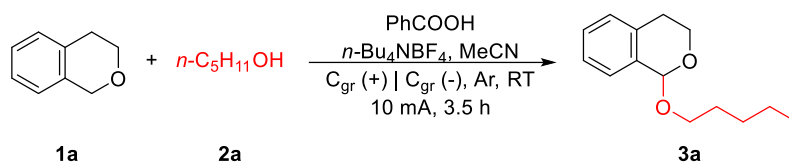
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### 3) Optimization of reaction conditions

Table S1. Optimization of reaction conditions<sup>a</sup>



Entry	Variation from the standard condition	Yield (%)
1	None	93 (92 <sup>b</sup> )
2	PhCOOH (0.8 eq, 0.4 mmol)	72
3	PhCOOH (1.2 eq, 0.6 mmol)	85
4	4-MeOPhCOOH instead of PhCOOH	85
5	4-CF <sub>3</sub> PhCOOH instead of PhCOOH	37
6	CH <sub>3</sub> COOH instead of PhCOOH	51
7	TFA instead of PhCOOH	51
8	TsOH instead of PhCOOH	63
9	PhCOONa instead of PhCOOH	36
10	Pt plate as the anode	69
11	Ni plate as the cathode	52
12	Pt plate as the cathode	44
13	Pt plate as both the anode and cathode	73
14	<i>n</i> -Bu <sub>4</sub> NPF <sub>6</sub> as the electrolyte	68
15	Et <sub>4</sub> NBF <sub>4</sub> as the electrolyte	72
16	LiClO <sub>4</sub> as the electrolyte	85
17	DCM as the solvent	51
18	DCE as the solvent	69
19	Acetone as the solvent	73
20	I = 5 mA	38
21	I = 15 mA	59
22	Air atmosphere	13
23	No PhCOOH	trace
24	No electric current	0
25	No electric current and electrolyte	0

<sup>a</sup>Standard conditions: undivided cell, graphite plate electrodes, **1a** (0.5 mmol), **2a** (1 mmol, 2 eq), PhCOOH (0.5 mmol, 1 eq), *n*-Bu<sub>4</sub>NBF<sub>4</sub> (1 mmol, 2 eq), 5 mL of MeCN, 10 mA, Ar atmosphere, stirring for 3.5 h at rt. Yields were determined by <sup>1</sup>H NMR spectroscopy using dibromomethane as an internal standard. Abbreviations: TFA, trifluoroacetic acid; TsOH, toluenesulfonic acid; DCM, dichloromethane; DCE, 1,2-dichloroethane. <sup>b</sup>Isolated yield.

## 4) Control experiments and mechanistic studies

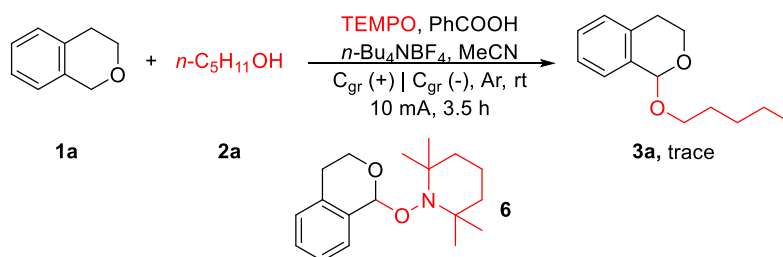
### A) Aerobic electrooxidation of isochroman



An oven-dried 25 mL undivided cell equipped with two graphite sheet electrodes (10 mm × 10 mm × 3 mm) was charged with PhCOOH (61.1 mg, 0.5 mmol, 1 eq.), *n*-Bu<sub>4</sub>NBF<sub>4</sub> (329.3 mg, 1 mmol, 2 eq.), MeCN (5 mL), and isochromane (67.1 mg, 0.5 mmol, 1 eq.). The electrolysis was carried out in air atmosphere at room temperature using a constant current of 10 mA for 3.5 hours.

The solvent in the system is removed under reduced pressure, and the crude product is purified by preparative TLC with ethyl acetate/petroleum ether as the eluent.

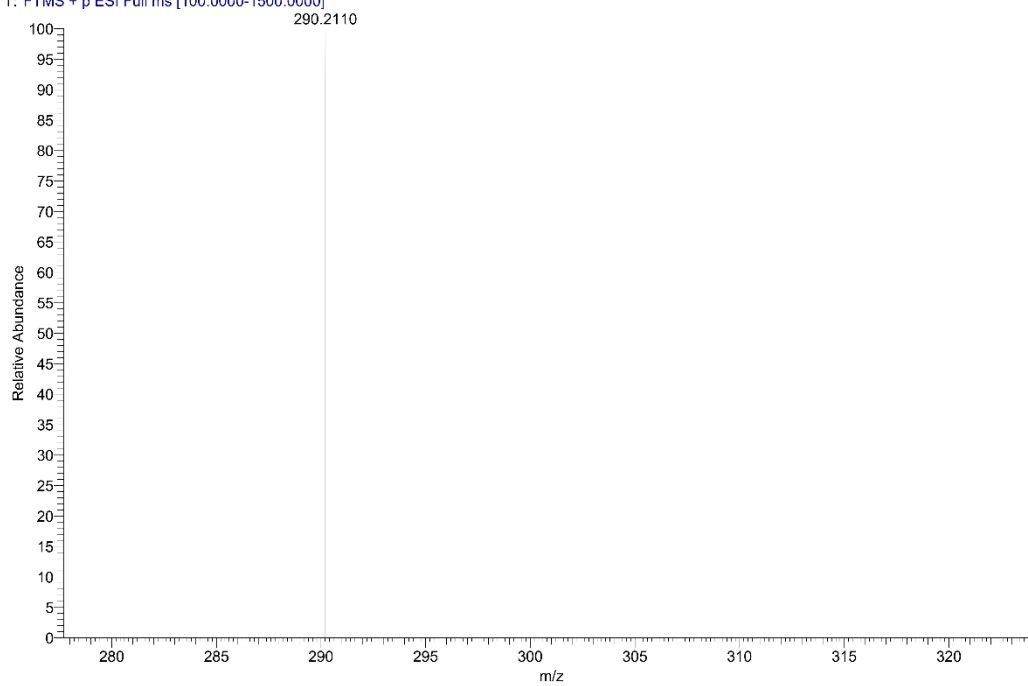
### B) Radical-trapping studies



HRMS (ESI) *m/z* calcd for C<sub>18</sub>H<sub>28</sub>NO<sub>2</sub><sup>+</sup> (M+H)<sup>+</sup> 290.2115, found 290.2110

PhCOOH (61.1 mg, 0.5 mmol, 1 eq.), *n*-Bu<sub>4</sub>NBF<sub>4</sub> (329.3 mg, 1 mmol, 2 eq.), TEMPO (156.3 mg, 1 mmol, 2 eq.), MeCN (5 mL), isochromane (67.1 mg, 0.5 mmol, 1 eq.) and *n*-C<sub>5</sub>H<sub>11</sub>OH (88.2 mg, 1 mmol, 2 eq.) were subsequently added into a 25 mL undivided cell. Insert the graphite plate electrodes (10 mm × 10 mm × 3 mm) into the cell, and purge the cell with argon for 10 s. The electrolysis was carried out in Ar atmosphere at room temperature using a constant current of 10 mA for 3.5 hours. We detected the radical trapping product **6** by HRMS analysis, confirming involvement of the radical intermediate. The HRMS spectrum of **6** is illustrated as below.

1-189-3 #21-25 RT: 0.09-0.11 AV: 5 NL: 4.29E5  
T: FTMS + p ESI Full ms [100.0000-1500.0000]



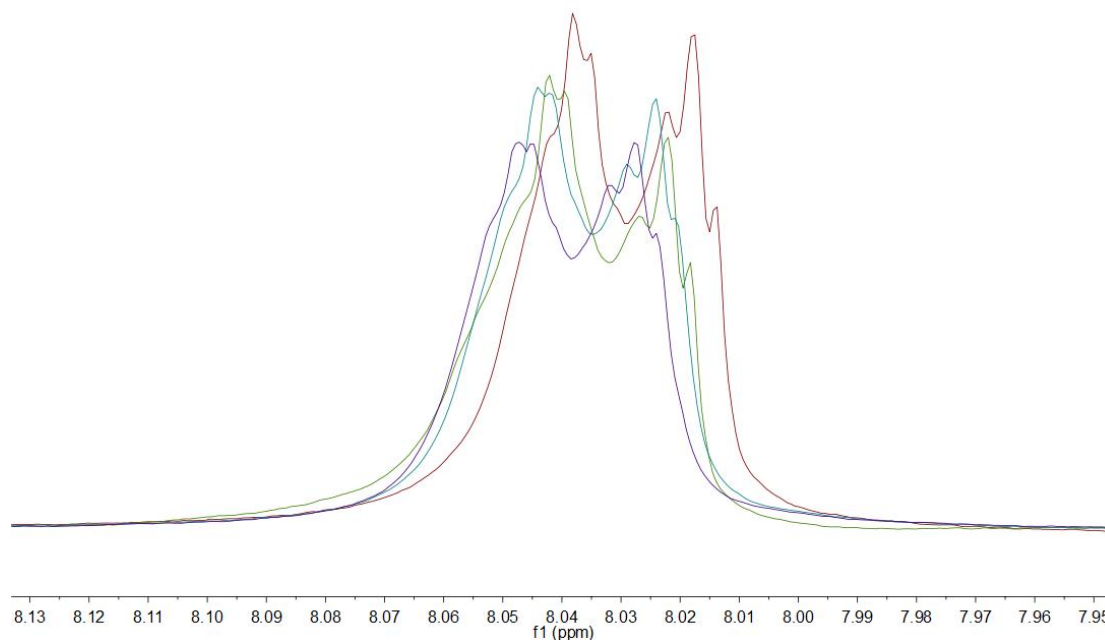
## 5) NMR spectroscopic investigation

For each sample, benzoic acid (0.5 mmol, 1 eq), isochroman (0–1 mmol, 0–2 eq) and 0.5 mL of CD<sub>3</sub>CN were added into the NMR tube, and the average chemical shift of 2-H of benzoic acid was investigated.

Table S2. The concentration dependence of isochroman of the chemical shift of benzoic acid

Entry	Isochroman (eq.)	Average chemical shift of 2-H of benzoic acid (ppm)
1	0	8.028
2	0.2	8.027
3	0.4	8.029
4	0.6	8.029
5	0.8	8.030
6	1	8.032
7	1.2	8.033
8	1.5	8.034
9	2	8.037

Figure S1. Stacked <sup>1</sup>H NMR spectrum of 2-H of benzoic acid<sup>a</sup>

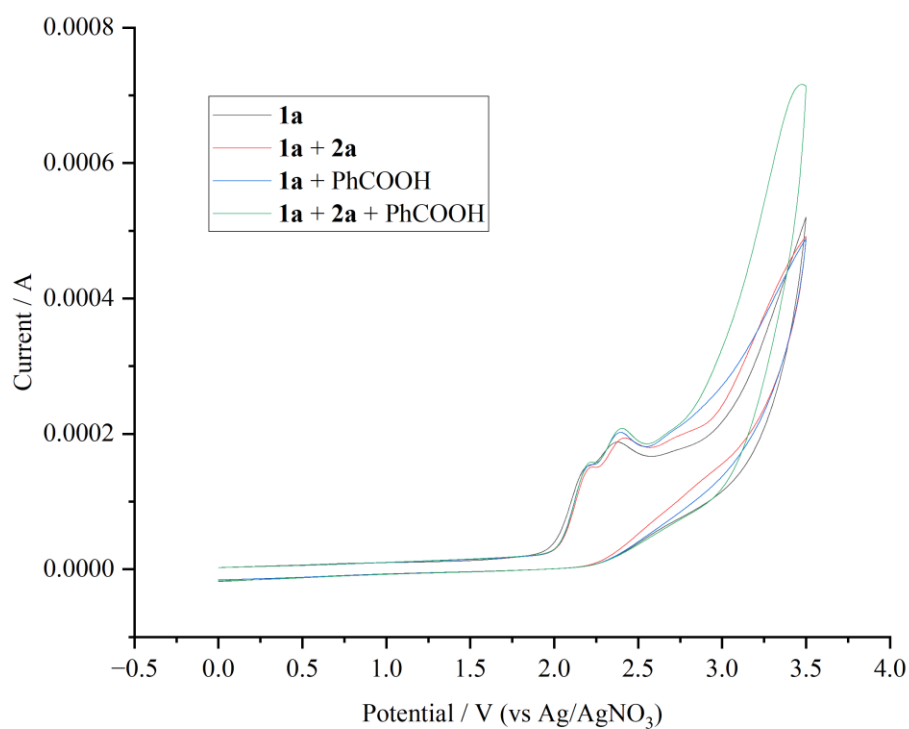


<sup>a</sup>Red: Entry 1; Green: Entry 6; Cyan: Entry 8; Purple: Entry 9.

## 6) Cyclic Voltammetry experiment

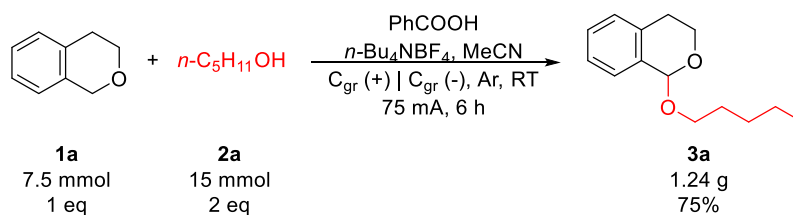
Cyclic voltammetry was performed in a three-electrode cell connected to a Schlenk line under air at room temperature. The working electrode was a glassy carbon electrode, the counter electrode a platinum wire. The reference was an Ag/AgNO<sub>3</sub> electrode submerged in saturated aqueous AgNO<sub>3</sub> solution, and separated from reaction by a salt bridge. 10 mL of CH<sub>3</sub>CN containing 0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub> were poured into the electrochemical cell in all experiments, and the concentration of all tested compounds was 2 mmol/L. The scan rate is 0.1 V/s, ranging from 0 V to 3.5 V. The peak potentials vs. Ag/AgNO<sub>3</sub> for used.

Figure S2. Cyclic Voltammetry of **1a** under different conditions





## 7) Gram-scale synthesis

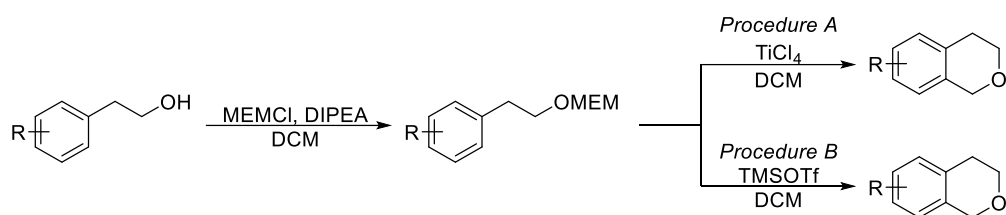


PhCOOH (0.92 g, 7.5 mmol, 1 eq.),  $n\text{-Bu}_4\text{NBF}_4$  (2.47 g, 7.5 mmol, 1 eq.), MeCN (40 mL), isochromane (1.01 g, 7.5 mmol, 1 eq.) and  $n\text{-C}_5\text{H}_{11}\text{OH}$  (1.32 g, 15 mmol, 2 eq.) were subsequently added into a 100 mL undivided cell. Insert the graphite plate electrodes (10 mm  $\times$  30 mm  $\times$  3 mm) into the cell, and purge the cell with argon for 20 s. The electrolysis was carried out in Ar atmosphere at room temperature using a constant current of 75 mA for 6 hours.

The solvent in the system is removed under reduced pressure, and the crude product is purified by column chromatography.

## 8) Synthesis of substrates

General procedure for the preparation of substituted isochroman (**1b-1d**)<sup>[1]</sup>



Under argon atmosphere, substituted phenethyl alcohol (10 mmol, 1 eq), dry DCM (15 mL), and DIPEA (15 mmol, 1.5 eq) are added into a dry 50 mL flask subsequently. The reaction system is cooled in ice bath and stirs at 0 °C, then MEMCl (15 mmol, 1.5 eq) is added dropwise. The mixture is stirred at room temperature for 2.5 h.

After the completion of reaction, 20 mL of saturated Na<sub>2</sub>CO<sub>3</sub> solution is added, and the mixture is extracted with 20 mL × 3 of DCM. The organic phase is dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated in *vacuo*. The crude product (MEM acetal) is directly applied to the further reaction without purification (Procedure A) or purified with column chromatography (Procedure B).

### Procedure A (**1b**, **1c**):

Under argon atmosphere, dry DCM (15 mL) and TiCl<sub>4</sub> (15 mmol, 1.5 eq) are subsequently added into a dry 50 mL flask. The reaction system is cooled in ice bath and stir at 0°C. The MEM acetal is added, and the mixture is stirred in ice bath for 2.5h.

After the completion of reaction, 20 mL of saturated Na<sub>2</sub>CO<sub>3</sub> solution is added, and the mixture is extracted with 20 mL × 3 of DCM. The organic phase is dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated in *vacuo*. The crude product (substituted isochroman) is purified with column chromatography (PE:EA = 20:1).

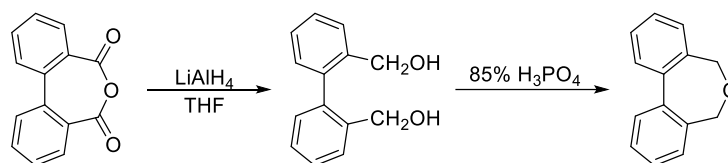
### Procedure B (**1d**):

To the MEM acetal (1.0 equiv) in DCM at 0 °C was added TMSOTf (1 mmol, 0.1 eq) dropwise. The mixture is stirred at room temperature for 10 h.

After the completion of reaction, 20 mL of saturated Na<sub>2</sub>CO<sub>3</sub> solution is added, and the mixture is extracted with 20mL × 3 of DCM. The organic phase is dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated in *vacuo*. The crude product (substituted

isochroman) is purified with column chromatography (PE:EA = 20:1).

Procedure for the preparation of 5,7-dihydrodibenzo[*c,e*]oxepine (**1e**)<sup>[1]</sup>

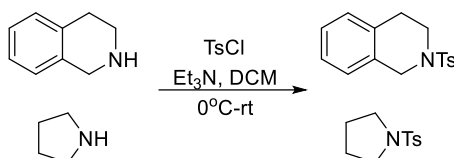


Under argon atmosphere, LiAlH<sub>4</sub> (3.4 g, 0.09 mol) and dry THF (250 mL) are added to a dry 500 mL flask and stirring. Diphenic anhydride (10.0 g, 0.045 mol) was added in 1 g portion over 2 h, and then the mixture is refluxed for 16 h.

The reaction is quenched with dropwise addition of water, and the precipitate is removed with vacuum filtering. The mixture is extracted with 20 mL × 3 of DCM. The organic phase is dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated in *vacuo*. The crude product was recrystallised from benzene for the further reaction.

To 50 mL of 85% H<sub>3</sub>PO<sub>4</sub>, the diol (8.0 g, 0.037 mmol) is added and the mixture is refluxed for 1 h. After the reaction is over, the mixture is extracted with Et<sub>2</sub>O, dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated in *vacuo*. The crude product is recrystallised from hexane and yields as a white solid.

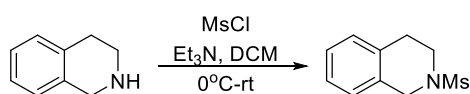
Procedure for the preparation of **1h** and **1j**<sup>[2]</sup>



1,2,3,4-tetrahydroisoquinoline or pyrrolidine (1 eq, 20 mmol), Et<sub>3</sub>N (1.2 eq) and DCM (40 mL) are added to a dry 100 mL flask and stirring at 0°C. TsCl (4.58 g, 1.2 eq) is added, and then the mixture is stirred for 1.5 h at room temperature.

The mixture is extracted with DCM, the organic phase is dried with anhydrous Na<sub>2</sub>SO<sub>4</sub> and then concentrated in *vacuo*. The crude product is purified with column chromatography (PE:EA = 5:1).

Procedure for the preparation of **1i**<sup>[2]</sup>

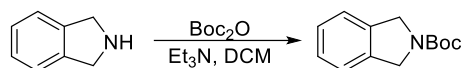


1,2,3,4-tetrahydroisoquinoline (1 eq, 20 mmol), Et<sub>3</sub>N (1.2 eq) and DCM (40 mL)

are added to a dry 100 mL flask and stirring at 0°C. MsCl (1.86 mL, 1.2 eq) was added, and then the mixture is stirred for 1.5 h at room temperature.

The mixture is extracted with DCM, the organic phase dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, concentrated in *vacuo*. The crude product is purified with column chromatography (PE:EA = 5:1).

Procedure for the preparation of **4k**<sup>[3]</sup>

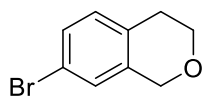


Isoindoline (1 eq, 20 mol), Et<sub>3</sub>N (1.2 eq) and DCM (40 mL) are added to a dry 100 mL flask and stirring. Boc<sub>2</sub>O (5.24 g, 1.2 eq) was added, and then the mixture is stirred overnight.

The mixture is concentrated in *vacuo*. The crude product is purified with column chromatography. (PE:EA = 5:1)

## 9) Characterization data for the substrates and products

### 7-Bromoisochromane (**1b**)<sup>[1]</sup>



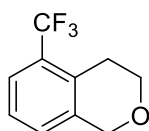
Colorless oil. Acquired via Procedure A with 63% Yield.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.26 (d, *J* = 8.2 Hz, 1H), 7.11 (s, 1H), 6.97 (d, *J* = 8.1 Hz, 1H), 4.71 (s, 2H), 3.93 (t, *J* = 5.7 Hz, 2H), 2.78 (t, *J* = 5.5 Hz, 2H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 137.1, 132.2, 130.6, 129.5, 127.4, 119.5, 67.4, 65.2, 27.8.

The spectral data obtained were identical with those reported in literature.

### 5-(Trifluoromethyl)isochromane (**1c**)<sup>[1]</sup>



Colorless oil. Acquired via Procedure A with 51% Yield.

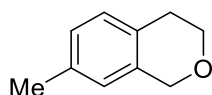
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.49 (d, *J* = 7.7 Hz, 1H), 7.13 (d, *J* = 7.7 Hz, 1H), 4.80 (s, 2H), 3.97 (t, *J* = 5.8 Hz, 2H), 3.00 (t, *J* = 5.7 Hz, 2H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 136.5, 132.1 (d, *J* = 1.5 Hz), 128.8 (q, *J* = 29.7 Hz), 128.2 (q, *J* = 0.9 Hz), 125.8, 124.1 (q, *J* = 5.8 Hz), 68.0, 64.7, 25.3 (q, *J* = 2.0 Hz).

**<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -61.45.

The spectral data obtained were identical with those reported in literature.

### 7-Methylisochromane (**1d**)<sup>[1]</sup>



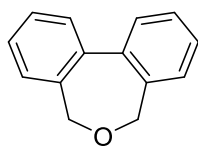
Colorless oil. Acquired via Procedure B with 63% Yield.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 6.99 (q, *J* = 7.9 Hz, 2H), 6.79 (s, 1H), 4.73 (s, 2H), 3.95 (t, *J* = 5.7 Hz, 2H), 2.81 (t, *J* = 5.6 Hz, 2H), 2.29 (s, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 135.5, 134.7, 130.1, 128.8, 127.2, 124.9, 68.0, 65.6, 28.0, 21.1.

The spectral data obtained were identical with those reported in literature.

5,7-Dihydrodibenzo[*c,e*]oxepine (**1e**)<sup>[1]</sup>



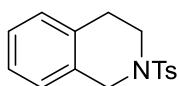
White Solid. Acquired with 70% Yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.56 (d, *J* = 7.6 Hz, 2H), 7.53 – 7.47 (m, 2H), 7.45 – 7.38 (m, 4H), 4.36 (s, 4H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 141.2, 135.2, 129.7, 129.0, 128.3, 127.5, 67.6.

The spectral data obtained were identical with those reported in literature.

2-Tosyl-1,2,3,4-tetrahydroisoquinoline (**1h**)<sup>[2]</sup>



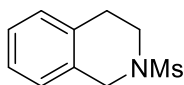
White Solid. Acquired with 99% Yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.18 – 7.10 (m, 2H), 7.10 – 7.05 (m, 1H), 7.05 – 6.99 (m, 1H), 4.24 (s, 2H), 3.35 (t, *J* = 5.9 Hz, 2H), 2.93 (t, *J* = 5.8 Hz, 2H), 2.42 (s, 3H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 143.7, 133.2, 133.1, 131.6, 129.7, 128.8, 127.8, 126.7, 126.4, 126.3, 47.6, 43.7, 28.9, 21.5.

The spectral data obtained were identical with those reported in literature.

2-(Methylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (**1i**)<sup>[2]</sup>



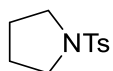
White Solid. Acquired with 98% Yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.23 – 7.13 (m, 3H), 7.12 – 7.07 (m, 1H), 4.46 (s, 2H), 3.56 (t, *J* = 6.0 Hz, 2H), 2.98 (t, *J* = 5.9 Hz, 2H), 2.84 (s, 3H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 133.1, 131.7, 129.1, 127.0, 126.6, 126.4, 47.3, 43.4, 35.9, 28.7.

The spectral data obtained were identical with those reported in literature.

1-Tosylpyrrolidine (**1j**)<sup>[2]</sup>



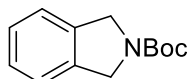
White Solid. Acquired with 99% Yield.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 (d,  $J = 8.1$  Hz, 2H), 7.33 (d,  $J = 8.1$  Hz, 2H), 3.23 (t,  $J = 6.7$  Hz, 4H), 2.43 (s, 3H), 1.88 – 1.56 (m, 4H).

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  143.4, 133.8, 129.6, 127.5, 47.9, 25.2, 21.5.

The spectral data obtained were identical with those reported in literature.

*t*-Butyl isoindoline-2-carboxylate (**1k**)<sup>[3]</sup>



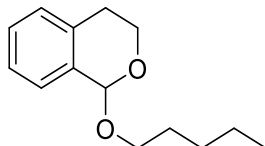
White Solid. Acquired with 99% Yield.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 – 7.19 (m, 4H), 4.67 (d,  $J = 15.2$  Hz, 4H), 1.52 (s, 9H).

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  154.6, 137.3, 137.0, 127.3, 127.2, 122.8, 122.5, 79.7, 52.3, 52.0, 28.6.

The spectral data obtained were identical with those reported in literature.

1-(Pentyloxy)isochromane (**3a**)



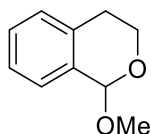
Colorless oil.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.27 – 7.16 (m, 3H), 7.16 – 7.05 (m, 1H), 5.54 (s, 1H), 4.15 (td,  $J = 11.6, 3.4$  Hz, 1H), 3.88 (ddd,  $J = 13.0, 8.2, 4.1$  Hz, 2H), 3.63 (dt,  $J = 9.6, 6.7$  Hz, 1H), 3.08 – 2.94 (m, 1H), 2.62 (dd,  $J = 16.5, 1.4$  Hz, 1H), 1.72 – 1.61 (m, 2H), 1.45 – 1.29 (m, 4H), 0.91 (t,  $J = 7.0$  Hz, 3H).

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  134.5, 134.1, 128.5, 128.0, 127.5, 126.3, 96.8, 68.3, 57.8, 29.5, 28.5, 28.1, 22.5, 14.1

**HRMS** (ESI)  $m/z$  calcd for  $\text{C}_{14}\text{H}_{20}\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 243.1356, found: 243.1351.

1-(Methyloxy)isochromane (**3b**)<sup>[4]</sup>



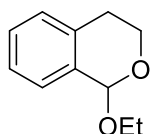
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.29 – 7.16 (m, 3H), 7.10 (d, *J* = 6.9 Hz, 1H), 5.44 (s, 1H), 4.11 (td, *J* = 11.7, 3.3 Hz, 1H), 3.96 – 3.84 (m, 1H), 3.53 (s, 3H), 3.08 – 2.95 (m, 1H), 2.60 (dd, *J* = 16.5, 1.5 Hz, 1H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 134.2, 134.1, 128.5, 128.2, 127.5, 126.4, 97.8, 57.8, 55.3, 28.0.

The spectral data obtained were identical with those reported in literature.

#### 1-(Ethyloxy)isochromane (**3c**)



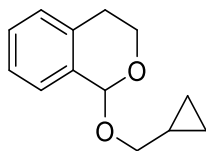
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.35 – 7.19 (m, 3H), 7.16 – 7.04 (m, 1H), 5.56 (s, 1H), 4.16 (td, *J* = 11.6, 3.4 Hz, 1H), 4.01 – 3.84 (m, 2H), 3.70 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.07 – 2.96 (m, 1H), 2.71 – 2.55 (m, 1H), 1.30 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.4, 134.1, 128.5, 128.1, 127.5, 126.4, 96.6, 63.5, 57.9, 28.1, 15.4.

**HRMS** (ESI) *m/z* calcd for C<sub>11</sub>H<sub>14</sub>NaO<sub>2</sub><sup>+</sup> (*M*+Na<sup>+</sup>): 201.0886, found: 201.0887.

#### 1-(Cyclopropylmethoxy)isochromane (**3d**)



Colorless oil.

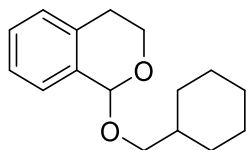
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.26-7.20 (m, 3H), 7.14 – 7.06 (m, 1H), 5.60 (s, 1H), 4.25-4.14 (m, 1H), 3.91-3.85 (m, 1H), 3.64-3.52 (m, 2H), 3.10 – 2.92 (m, 1H), 2.61 (dd, *J* = 16.5, 1.8 Hz, 1H), 1.22 – 1.10 (m, 1H), 0.63 – 0.50 (m, 2H), 0.34 – 0.21 (m, 2H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.4, 134.1, 128.5, 128.1, 127.6, 126.4, 96.3, 72.7, 57.8, 28.0, 10.8, 3.4, 3.1.



**HRMS** (ESI)  $m/z$  calcd for  $C_{13}H_{16}NaO_2^+$  ( $M+Na^+$ ): 227.1043, found: 227.1042.

1-(Cyclohexylmethoxy)isochromane (**3e**)



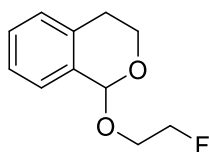
Colorless oil.

**$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta$  7.28 – 7.15 (m, 3H), 7.14 – 7.03 (m, 1H), 5.51 (s, 1H), 4.13 (td,  $J = 11.6, 3.4$  Hz, 1H), 3.87 (ddd,  $J = 11.2, 6.0, 1.6$  Hz, 1H), 3.68 (dd,  $J = 9.5, 6.5$  Hz, 1H), 3.41 (dd,  $J = 9.5, 6.7$  Hz, 1H), 3.07 – 2.93 (m, 1H), 2.60 (dd,  $J = 16.5, 1.5$  Hz, 1H), 1.90 – 1.76 (m, 2H), 1.77 – 1.60 (m, 4H), 1.22 (qdd,  $J = 12.3, 9.2, 3.2$  Hz, 3H), 0.99 (td,  $J = 12.1, 2.6$  Hz, 2H).

**$^{13}C$  NMR** (100 MHz,  $CDCl_3$ )  $\delta$  134.5, 134.1, 128.5, 128.0, 127.6, 126.3, 97.0, 74.1, 57.8, 38.1, 30.3, 30.2, 28.1, 26.7, 25.9.

**HRMS** (ESI)  $m/z$  calcd for  $C_{16}H_{22}NaO_2^+$  ( $M+Na^+$ ): 269.1512, found: 269.1511.

1-(2-Fluoroethoxy)isochromane (**3f**)



Colorless oil.

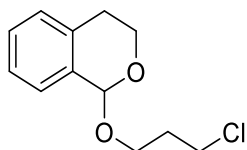
**$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta$  7.34 – 7.17 (m, 3H), 7.16 – 7.07 (m, 1H), 5.62 (s, 1H), 4.70 (dd,  $J = 5.2, 3.3$  Hz, 1H), 4.58 (dd,  $J = 5.0, 3.5$  Hz, 1H), 4.21 – 3.88 (m, 4H), 3.09 – 2.96 (m, 1H), 2.63 (dd,  $J = 16.6, 1.8$  Hz, 1H).

**$^{13}C$  NMR** (100 MHz,  $CDCl_3$ )  $\delta$  134.1, 133.7, 128.5, 128.3, 127.6, 126.4, 97.1, 83.0 (d,  $J = 170$  Hz), 66.9 (d,  $J = 20$  Hz), 58.0, 27.9.

**$^{19}F$  NMR** (377 MHz,  $CDCl_3$ )  $\delta$  -223.59.

**HRMS** (ESI)  $m/z$  calcd for  $C_{11}H_{13}FNaO_2$  ( $M+Na^+$ ): 219.0792, found: 219.0793.

1-(3-Chloropropoxy)isochromane (**3g**)



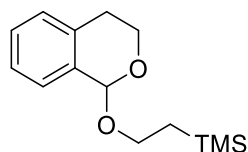
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.27 – 7.18 (m, 3H), 7.16 – 7.08 (m, 1H), 5.55 (s, 1H), 4.12 (td, *J* = 11.6, 3.4 Hz, 1H), 4.02 (dt, *J* = 10.2, 5.7 Hz, 1H), 3.91 (ddd, *J* = 11.2, 6.0, 1.5 Hz, 1H), 3.82 – 3.73 (m, 1H), 3.73 – 3.63 (m, 2H), 3.09 – 2.95 (m, 1H), 2.62 (ddd, *J* = 16.5, 3.1, 1.4 Hz, 1H), 2.10 (p, *J* = 6.2 Hz, 2H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.1, 128.5, 128.2, 127.5, 126.4, 97.0, 64.5, 58.0, 42.0, 32.7, 28.0.

**HRMS** (ESI) *m/z* calcd for C<sub>12</sub>H<sub>15</sub>ClNaO<sub>2</sub><sup>+</sup> (M+Na<sup>+</sup>): 249.0653, found: 249.0652.

(2-(Isochroman-1-yloxy)ethyl)trimethylsilane (**3h**)



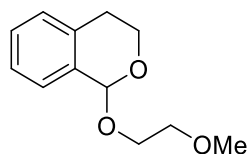
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.21 (dd, *J* = 8.4, 7.1 Hz, 3H), 7.14 – 7.05 (m, 1H), 5.55 (s, 1H), 4.14 (td, *J* = 11.6, 3.3 Hz, 1H), 4.02 – 3.81 (m, 2H), 3.77 – 3.63 (m, 1H), 3.08 – 2.90 (m, 1H), 2.60 (d, *J* = 16.4 Hz, 1H), 1.11-0.95 (m, 2H), 0.03 (s, 9H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 135.8, 135.4, 129.8, 129.3, 128.8, 127.6, 97.6, 66.7, 59.2, 29.4, 19.7, 0.0.

**HRMS** (ESI) *m/z* calcd for C<sub>14</sub>H<sub>22</sub>NaO<sub>2</sub>Si<sup>+</sup> (M+Na<sup>+</sup>): 273.1281, found: 273.1279.

1-(2-Methoxyethoxy)isochromane (**3i**)



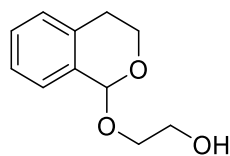
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.33 – 7.15 (m, 3H), 7.15 – 7.05 (m, 1H), 5.60 (s, 1H), 4.16 (td, *J* = 11.6, 3.4 Hz, 1H), 3.99 (dt, *J* = 11.1, 4.6 Hz, 1H), 3.90 (ddd, *J* = 11.2, 6.0, 1.6 Hz, 1H), 3.86 – 3.79 (m, 1H), 3.64 (t, *J* = 4.9 Hz, 2H), 3.40 (s, 3H), 3.08 – 2.95 (m, 1H), 2.62 (dd, *J* = 16.6, 1.7 Hz, 1H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.1, 134.0, 128.4, 128.2, 127.7, 126.3, 97.1, 72.1, 67.1, 59.0, 57.9, 28.0.

**HRMS** (ESI) *m/z* calcd for C<sub>12</sub>H<sub>16</sub>NaO<sub>3</sub><sup>+</sup> (M+Na<sup>+</sup>): 231.0992, found: 231.0993.

2-(Isochroman-1-yloxy)ethan-1-ol (**3j**)



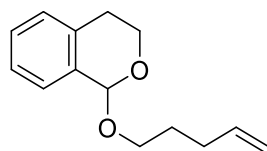
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.31 – 7.18 (m, 3H), 7.13 (d, *J* = 7.2 Hz, 1H), 5.59 (s, 1H), 4.15 (td, *J* = 11.5, 3.4 Hz, 1H), 3.99 – 3.89 (m, 2H), 3.88 – 3.70 (m, 3H), 3.07 – 2.96 (m, 1H), 2.74 (d, *J* = 5.4 Hz, 1H), 2.64 (d, *J* = 16.5 Hz, 1H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.0, 133.8, 128.5, 128.4, 127.5, 126.4, 97.4, 70.5, 62.3, 58.2, 27.9.

**HRMS** (ESI) *m/z* calcd for C<sub>11</sub>H<sub>14</sub>NaO<sub>3</sub><sup>+</sup> (M+Na<sup>+</sup>): 217.0835, found: 217.0836.

1-(Pent-4-en-1-yloxy)isochromane (**3k**)



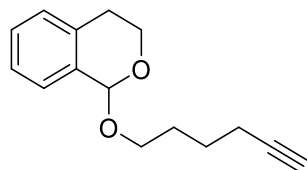
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.29 – 7.15 (m, 3H), 7.15 – 7.06 (m, 1H), 5.84 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.53 (s, 1H), 5.05 (dd, *J* = 17.1, 1.6 Hz, 1H), 4.98 (dd, *J* = 10.2, 1.0 Hz, 1H), 4.14 (td, *J* = 11.6, 3.4 Hz, 1H), 3.98 – 3.80 (m, 2H), 3.64 (dt, *J* = 9.7, 6.6 Hz, 1H), 3.09 – 2.92 (m, 1H), 2.61 (dd, *J* = 16.5, 1.5 Hz, 1H), 2.19 (dd, *J* = 14.6, 7.0 Hz, 2H), 1.83 – 1.69 (m, 2H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 138.3, 134.5, 134.1, 128.5, 128.1, 127.5, 126.3, 114.9, 96.8, 67.5, 57.9, 30.5, 29.0, 28.1.

**HRMS** (ESI) *m/z* calcd for C<sub>14</sub>H<sub>18</sub>NaO<sub>2</sub><sup>+</sup> (M+Na<sup>+</sup>): 241.1199, found: 241.1197.

1-(Hex-5-yn-1-yloxy)isochromane (**3l**)



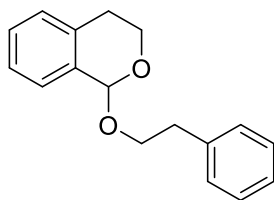
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.27 – 7.17 (m, 3H), 7.16 – 7.07 (m, 1H), 5.54 (s, 1H), 4.14 (td, *J* = 11.6, 3.3 Hz, 1H), 3.96 – 3.84 (m, 2H), 3.66 (dt, *J* = 9.8, 6.3 Hz, 1H), 3.09 – 2.94 (m, 1H), 2.62 (d, *J* = 15.2 Hz, 1H), 2.24 (td, *J* = 7.0, 2.5 Hz, 2H), 1.96 (t, *J* = 2.6 Hz, 1H), 1.78 (dd, *J* = 8.1, 6.3 Hz, 2H), 1.67 (dd, *J* = 11.3, 4.0 Hz, 2H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.3, 134.1, 128.5, 128.1, 127.5, 126.3, 96.8, 84.4, 68.5, 67.5, 57.9, 28.9, 28.0, 25.3, 18.2

**HRMS** (ESI) *m/z* calcd for C<sub>15</sub>H<sub>18</sub>NaO<sub>2</sub><sup>+</sup> (M+Na<sup>+</sup>): 253.1199, found: 253.1197.

1-Phenethoxyisochromane (**3m**)



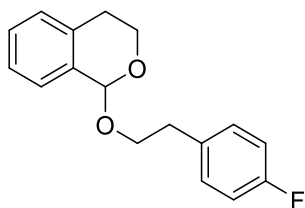
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.33 – 7.24 (m, 4H), 7.24 – 7.15 (m, 3H), 7.10 (dd, *J* = 12.6, 7.2 Hz, 2H), 5.52 (s, 1H), 4.09 (dt, *J* = 20.6, 9.6 Hz, 2H), 3.94 – 3.77 (m, 2H), 2.98 (p, *J* = 10.5 Hz, 3H), 2.58 (d, *J* = 16.4 Hz, 1H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 139.1, 134.2, 134.2, 129.1, 128.5, 128.4, 128.2, 127.6, 126.4, 126.3, 97.0, 69.0, 58.0, 36.5, 28.1.

**HRMS** (ESI) *m/z* calcd for C<sub>17</sub>H<sub>18</sub>NaO<sub>2</sub><sup>+</sup> (M+Na<sup>+</sup>): 277.1199, found: 277.1196.

1-(4-Fluorophenethoxy)isochromane (**3n**)



Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.20 (dd, *J* = 13.1, 7.3 Hz, 4H), 7.10 (t, *J* = 6.1 Hz, 2H), 6.97 (t, *J* = 8.6 Hz, 2H), 5.51 (s, 1H), 4.14 – 3.95 (m, 2H), 3.89 – 3.76 (m, 2H), 2.97 (ddd, *J* = 20.7, 13.0, 6.5 Hz, 3H), 2.58 (d, *J* = 16.3 Hz, 1H).

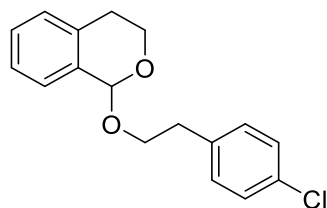
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 161.57 (d, *J* = 243.7 Hz), 134.8 (d, *J* = 3.1 Hz), 134.1, 130.4 (d, *J* = 7.8 Hz), 128.5, 128.2, 127.5, 126.3, 115.1 (d, *J* = 21.1 Hz), 97.0, 68.9,

57.9, 35.6, 28.0.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -117.18.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{FNaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 295.1105, found: 295.1102.

1-(4-Chlorophenoxy)isochromane (**3o**)



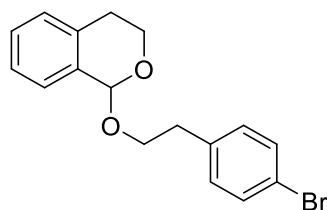
Colorless oil.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.21 (dt,  $J = 11.1, 8.3$  Hz, 6H), 7.10 (t,  $J = 6.3$  Hz, 2H), 5.50 (s, 1H), 4.14 – 3.97 (m, 2H), 3.91 – 3.74 (m, 2H), 2.96 (ddd,  $J = 21.7, 12.9, 6.4$  Hz, 3H), 2.58 (d,  $J = 16.0$  Hz, 1H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  137.7, 134.1, 134.1, 132.0, 130.4, 128.5, 128.4, 128.2, 127.5, 126.4, 97.0, 68.6, 58.0, 35.7, 28.0.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{ClNaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 311.0809, found: 311.0808.

1-(4-Bromophenoxy)isochromane (**3p**)

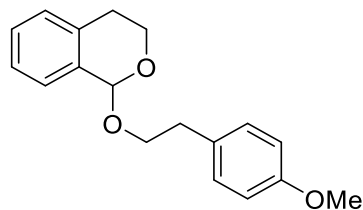


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 – 7.35 (m, 2H), 7.26 – 7.16 (m, 2H), 7.13 (dd,  $J = 8.5, 1.9$  Hz, 4H), 5.50 (s, 1H), 4.17 – 3.93 (m, 2H), 3.83 (ddd,  $J = 12.0, 8.1, 4.6$  Hz, 2H), 3.07 – 2.75 (m, 3H), 2.58 (d,  $J = 16.3$  Hz, 1H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  138.2, 134.1, 134.1, 131.4, 130.8, 128.5, 128.2, 127.5, 126.4, 120.1, 97.0, 68.5, 58.0, 35.8, 28.0.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{BrNaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 355.0304, found: 355.0301.

1-(4-Methoxyphenoxy)isochromane (**3q**)



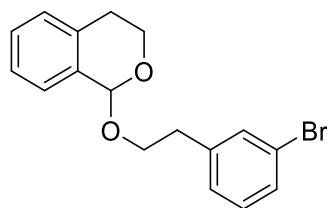
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.27 – 7.05 (m, 6H), 6.84 (d, *J* = 8.6 Hz, 2H), 5.52 (s, 1H), 4.07 (ddt, *J* = 11.0, 9.8, 5.3 Hz, 2H), 3.88 – 3.81 (m, 2H), 3.78 (s, 3H), 3.05 – 2.83 (m, 3H), 2.65 – 2.54 (m, 1H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 158.1, 134.2, 134.1, 131.1, 130.0, 128.5, 128.1, 127.5, 126.3, 113.8, 96.9, 69.2, 57.9, 55.3, 35.5, 28.0.

**HRMS** (ESI) *m/z* calcd for C<sub>18</sub>H<sub>20</sub>NaO<sub>3</sub><sup>+</sup> (*M*+Na<sup>+</sup>): 307.1305, found: 307.1302.

1-(3-Bromophenethoxy)isochromane (**3r**)



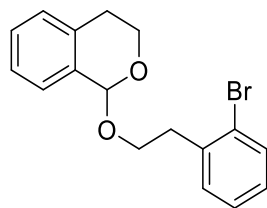
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.44 (s, 1H), 7.34 (dt, *J* = 7.4, 1.6 Hz, 1H), 7.25 – 7.06 (m, 6H), 5.50 (s, 1H), 4.14 – 3.98 (m, 2H), 3.89 – 3.78 (m, 2H), 3.03 – 2.86 (m, 3H), 2.64 – 2.52 (m, 1H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 141.6, 134.1, 134.0, 132.2, 129.9, 129.4, 128.5, 128.2, 127.7, 127.6, 126.4, 122.4, 97.0, 68.5, 58.0, 36.0, 28.0

**HRMS** (ESI) *m/z* calcd for C<sub>17</sub>H<sub>17</sub>BrNaO<sub>2</sub><sup>+</sup> (*M*+Na<sup>+</sup>): 355.0304, found: 355.0304.

1-(2-Bromophenethoxy)isochromane (**3s**)



Colorless oil.

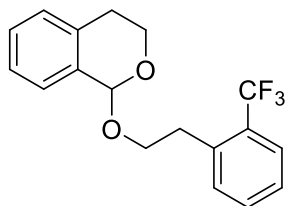
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.53 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 6.5 Hz, 1H), 7.26

– 7.01 (m, 6H), 5.54 (s, 1H), 4.17 – 3.98 (m, 2H), 3.98 – 3.78 (m, 2H), 3.12 (t,  $J = 7.1$  Hz, 2H), 3.05 – 2.89 (m, 1H), 2.58 (d,  $J = 16.0$  Hz, 1H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  138.3, 134.2, 132.9, 131.3, 128.5, 128.2, 128.1, 127.6, 127.4, 126.4, 97.0, 67.1, 58.0, 36.7, 28.1.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{BrNaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 355.0304, found: 355.0301.

1-(2-(Trifluoromethyl)phenoxy)isochromane (**3t**)



Colorless oil.

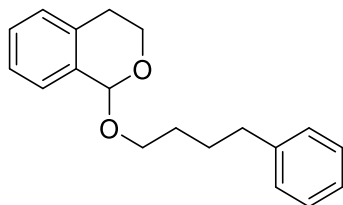
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 (d,  $J = 7.8$  Hz, 1H), 7.46 (q,  $J = 7.5$  Hz, 2H), 7.31 (t,  $J = 7.2$  Hz, 1H), 7.27 – 7.14 (m, 3H), 7.11 (d,  $J = 6.6$  Hz, 1H), 5.55 (s, 1H), 4.10 (dt,  $J = 15.4, 5.1$  Hz, 2H), 3.96 – 3.76 (m, 2H), 3.18 (t,  $J = 7.0$  Hz, 2H), 3.10 – 2.89 (m, 1H), 2.61 (d,  $J = 16.4$  Hz, 1H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  137.5, 134.2, 134.1, 131.9, 131.6, 128.9 (q,  $J = 25.6$  Hz), 128.5, 128.2, 127.5, 126.4, 126.3, 126.0 (q,  $J = 5.7$  Hz), 96.9, 68.3, 58.0, 33.1, 28.0.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -59.57.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{17}\text{F}_3\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 345.1073, found: 345.1069.

1-(4-Phenylbutoxy)isochromane (**3u**)



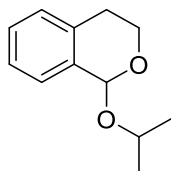
Colorless oil.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.27 (t,  $J = 7.6$  Hz, 2H), 7.24 – 7.12 (m, 6H), 7.10 (d,  $J = 5.3$  Hz, 1H), 5.52 (s, 1H), 4.13 (td,  $J = 11.5, 2.6$  Hz, 1H), 3.89 (dq,  $J = 12.4, 6.3$  Hz, 2H), 3.68 – 3.59 (m, 1H), 3.07 – 2.93 (m, 1H), 2.71 – 2.54 (m, 3H), 1.74 (dd,  $J = 11.4, 4.4$  Hz, 4H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  142.6, 134.4, 134.2, 128.5, 128.4, 128.2, 127.6, 126.4, 125.8, 96.9, 68.1, 57.9, 35.8, 29.5, 28.3, 28.1.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{19}\text{H}_{22}\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 305.1512, found: 305.1510.

1-Isopropoxyisochromane (**3v**)



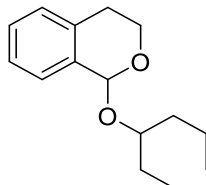
Colorless oil.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26 – 7.14 (m, 3H), 7.15 – 7.05 (m, 1H), 5.64 (s, 1H), 4.26 – 4.06 (m, 2H), 3.88 (ddd,  $J = 11.2, 6.0, 1.3$  Hz, 1H), 3.08 – 2.93 (m, 1H), 2.61 (dd,  $J = 16.5, 1.9$  Hz, 1H), 1.34 – 1.22 (m, 6H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  134.6, 134.2, 128.5, 127.9, 127.4, 126.3, 95.0, 69.5, 57.7, 28.1, 23.7, 22.0.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{12}\text{H}_{16}\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 215.1043, found: 215.1042.

1-(Hexan-3-yloxy)isochromane (**3w**)



Colorless oil.

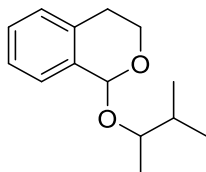
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 – 7.14 (m, 3H), 7.15 – 7.04 (m, 1H), 5.63 (s, 1H), 4.20 (ddd,  $J = 11.9, 5.9, 2.9$  Hz, 1H), 3.88 (dd,  $J = 11.2, 6.0$  Hz, 1H), 3.84 – 3.73 (m, 1H), 3.09 – 2.90 (m, 1H), 2.60 (dd,  $J = 16.6, 2.0$  Hz, 1H), 1.74 – 1.30 (m, 6H), 0.97 (ddt,  $J = 19.4, 11.9, 7.3$  Hz, 6H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  134.7, 134.3, 128.5, 127.9, 127.5, 126.3, 95.9, 95.3, 79.2, 78.1, 57.9, 36.5, 36.0, 28.2, 27.8, 26.4, 19.0, 18.6, 14.5, 14.2, 10.1, 9.3.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{15}\text{H}_{22}\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 257.1512, found: 257.1513.

1-((3-Methylbutan-2-yl)oxy)isochromane (**3x**)





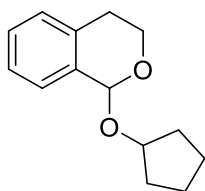
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.30 – 7.14 (m, 3H), 7.15 – 7.06 (m, 1H), 5.62 (d, *J* = 7.6 Hz, 1H), 4.24 – 4.13 (m, 1H), 3.93 – 3.82 (m, 1H), 3.74 (dp, *J* = 18.6, 6.2 Hz, 1H), 3.01 (ddd, *J* = 22.7, 12.0, 5.9 Hz, 1H), 2.60 (dt, *J* = 16.5, 3.3 Hz, 1H), 1.84 (dtt, *J* = 45.2, 13.3, 6.8 Hz, 1H), 1.23 (dd, *J* = 10.3, 6.3 Hz, 3H), 1.05 – 0.87 (m, 6H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.8, 134.7, 134.4, 134.2, 128.6, 128.4, 128.0, 127.8, 127.6, 127.4, 126.3, 97.0, 94.3, 79.5, 58.0, 57.7, 33.8, 33.2, 28.2, 28.1, 18.9, 18.7, 18.5, 18.1, 17.9, 16.3.

**HRMS** (ESI) *m/z* calcd for C<sub>15</sub>H<sub>22</sub>NaO<sub>2</sub><sup>+</sup> (*M*+Na<sup>+</sup>): 243.1356, found: 243.1354.

1-(Cyclopentyloxy)isochromane (**3y**)



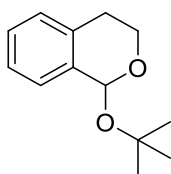
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.25 – 7.13 (m, 3H), 7.13 – 7.05 (m, 1H), 5.60 (s, 1H), 4.43 (t, *J* = 9.2 Hz, 1H), 4.15 (td, *J* = 11.7, 3.3 Hz, 1H), 3.92 – 3.83 (m, 1H), 3.06 – 2.93 (m, 1H), 2.64 – 2.55 (m, 1H), 1.94 – 1.65 (m, 6H), 1.65 – 1.46 (m, 2H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 134.7, 134.2, 128.5, 127.9, 127.5, 126.3, 95.5, 79.1, 57.9, 33.4, 32.3, 28.1, 23.6, 23.3.

**HRMS** (ESI) *m/z* calcd for C<sub>14</sub>H<sub>18</sub>NaO<sub>2</sub><sup>+</sup> (*M*+Na<sup>+</sup>): 241.1199, found: 241.1198.

1-(*t*-Butoxy)isochromane (**3z**)



Colorless oil.

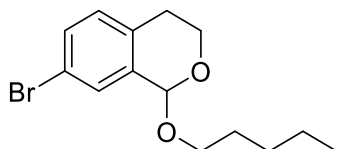
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.23 – 7.12 (m, 3H), 7.12 – 7.06 (m, 1H), 5.84 (s, 1H),

4.21 (td,  $J = 11.5, 3.4$  Hz, 1H), 3.86 (ddd,  $J = 11.3, 6.0, 1.8$  Hz, 1H), 3.04 – 2.88 (m, 1H), 2.61 (ddd,  $J = 16.5, 3.0, 1.8$  Hz, 1H), 1.39 (s, 9H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  135.7, 134.5, 128.6, 127.7, 127.4, 126.2, 91.5, 74.9, 57.6, 29.2, 28.2.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{13}\text{H}_{18}\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 229.1199, found: 229.1199.

#### 7-Bromo-1-(pentyloxy)isochromane (**4b**)



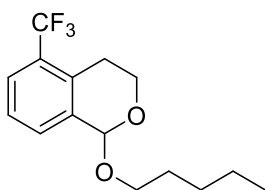
Colorless oil.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34 (dd,  $J = 11.7, 3.6$  Hz, 2H), 6.98 (d,  $J = 8.1$  Hz, 1H), 5.48 (s, 1H), 4.10 (td,  $J = 11.6, 3.3$  Hz, 1H), 3.94 – 3.79 (m, 2H), 3.61 (dt,  $J = 9.5, 6.7$  Hz, 1H), 3.00 – 2.85 (m, 1H), 2.57 (d,  $J = 16.6$  Hz, 1H), 1.75 – 1.56 (m, 2H), 1.36 (dd,  $J = 11.5, 8.0$  Hz, 4H), 0.92 (t,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  136.4, 133.1, 131.2, 130.5, 130.2, 119.8, 96.1, 68.5, 57.6, 29.5, 28.4, 27.6, 22.5, 14.1.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{14}\text{H}_{19}\text{BrNaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 321.0461, found: 321.0458.

#### 1-(Pentyloxy)-5-(trifluoromethyl)isochromane (**4c**)



Colorless oil.

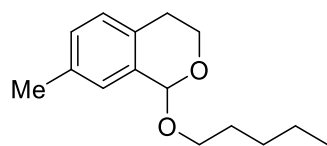
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (d,  $J = 7.7$  Hz, 1H), 7.41 (d,  $J = 7.6$  Hz, 1H), 7.31 (t,  $J = 7.7$  Hz, 1H), 5.55 (s, 1H), 4.14 (td,  $J = 11.7, 3.4$  Hz, 1H), 3.97 – 3.83 (m, 2H), 3.63 (dt,  $J = 9.6, 6.7$  Hz, 1H), 3.05 (ddd,  $J = 17.7, 11.9, 6.0$  Hz, 1H), 2.88 (d,  $J = 17.3$  Hz, 1H), 1.73 – 1.61 (m, 2H), 1.46 – 1.29 (m, 4H), 0.92 (t,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  135.9, 133.2, 131.5, 128.3 (q,  $J = 30.0$  Hz), 126.1, 125.7 (q,  $J = 5.6$  Hz), 123.0 (q,  $J = 283.5$  Hz), 96.4, 68.4, 57.0, 29.5, 28.5, 25.1, 22.5, 14.0.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.54.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{15}\text{H}_{19}\text{F}_3\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 311.1229, found: 311.1225.

7-Methyl-1-(pentyloxy)isochromane (**4d**)



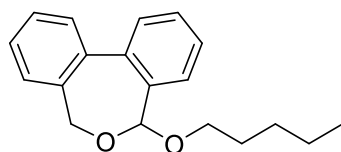
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.11 – 6.90 (m, 3H), 5.50 (s, 1H), 4.12 (td, *J* = 11.7, 3.3 Hz, 1H), 3.95 – 3.77 (m, 2H), 3.62 (dt, *J* = 9.4, 6.7 Hz, 1H), 3.04 – 2.86 (m, 1H), 2.56 (d, *J* = 15.7 Hz, 1H), 2.31 (s, 3H), 1.77 – 1.55 (m, 2H), 1.46 – 1.26 (m, 4H), 0.92 (t, *J* = 6.8 Hz, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 135.8, 134.2, 131.0, 129.0, 128.4, 127.9, 96.9, 68.3, 58.0, 29.6, 28.5, 27.7, 22.6, 21.1, 14.1.

**HRMS** (ESI) *m/z* calcd for C<sub>15</sub>H<sub>22</sub>NaO<sub>2</sub><sup>+</sup> (*M*+Na<sup>+</sup>): 257.1512, found: 257.1512.

5-(Pentyloxy)-5,7-dihydrodibenzo[*c,e*]oxepine (**4e**)



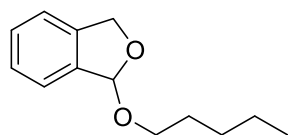
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.64 – 7.28 (m, 8H), 5.49 (s, 1H), 4.54 – 4.38 (m, 2H), 3.68 (dt, *J* = 9.2, 6.8 Hz, 1H), 3.31 (dt, *J* = 9.2, 6.2 Hz, 1H), 1.42 – 1.30 (m, 2H), 1.21 – 1.09 (m, 2H), 1.02 – 0.91 (m, 2H), 0.79 (t, *J* = 7.3 Hz, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 141.3, 138.6, 136.4, 136.3, 129.1, 128.9, 128.5, 128.0, 127.7, 127.6, 127.2, 101.8, 67.8, 66.8, 29.3, 28.1, 22.5, 14.1.

**HRMS** (ESI) *m/z* calcd for C<sub>19</sub>H<sub>22</sub>NaO<sub>2</sub><sup>+</sup> (*M*+Na<sup>+</sup>): 305.1512, found: 305.1508.

1-(Pentyloxy)-1,3-dihydroisobenzofuran (**4f**)



Colorless oil.

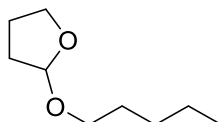
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.27 (m, 3H), 7.27 – 7.22 (m, 1H), 6.24 (d, *J* = 2.0 Hz, 1H), 5.20 (dd, *J* = 12.6, 1.2 Hz, 1H), 5.02 (d, *J* = 12.6 Hz, 1H), 3.69 (dd, *J* =

11.4, 4.7 Hz, 1H), 3.62 – 3.53 (m, 1H), 1.67 – 1.56 (m, 2H), 1.41 – 1.26 (m, 4H), 0.89 (t,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  140.7, 138.5, 129.8, 128.3, 123.7, 121.7, 107.6, 72.9, 68.3, 30.3, 29.0, 23.2, 14.8.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{13}\text{H}_{18}\text{NaO}_2^+$  ( $\text{M}+\text{Na}^+$ ): 229.1199, found: 229.1199.

2-(Pentyloxy)tetrahydrofuran (**4g**)<sup>[5]</sup>



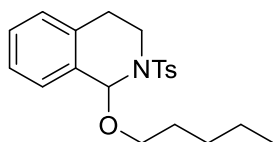
Colorless oil.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.10 (dd,  $J = 4.2, 1.5$  Hz, 1H), 3.94 – 3.78 (m, 2H), 3.71 – 3.59 (m, 1H), 3.43 – 3.29 (m, 1H), 2.06 – 1.73 (m, 4H), 1.64 – 1.47 (m, 2H), 1.32 (dd,  $J = 4.1, 2.8$  Hz, 4H), 0.90 (dd,  $J = 9.7, 4.0$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  103.7, 67.2, 66.7, 32.3, 29.4, 28.3, 23.5, 22.4, 14.0.

The spectral data obtained were identical with those reported in literature.

1-(Pentyloxy)-2-tosyl-1,2,3,4-tetrahydroisoquinoline (**4h**)



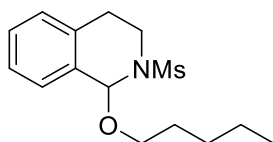
Colorless oil.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 (d,  $J = 8.3$  Hz, 2H), 7.31 – 7.12 (m, 5H), 6.96 (d,  $J = 6.9$  Hz, 1H), 6.03 (s, 1H), 3.76 (ddt,  $J = 16.2, 9.4, 4.3$  Hz, 2H), 3.66 – 3.51 (m, 2H), 2.61 – 2.42 (m, 2H), 2.35 (s, 3H), 1.66 – 1.52 (m, 2H), 1.39 – 1.23 (m, 5H), 0.89 (dd,  $J = 9.1, 4.9$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  143.3, 138.0, 133.7, 133.6, 129.5, 128.7, 128.5, 128.4, 126.9, 126.5, 83.3, 68.2, 38.4, 29.2, 28.4, 26.8, 22.5, 21.5, 14.1.

HRMS (ESI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{17}\text{NNaO}_3\text{S}^+$  ( $\text{M}+\text{Na}^+$ ): 396.1604, found: 396.1602.

2-(Methylsulfonyl)-1-(pentyloxy)-1,2,3,4-tetrahydroisoquinoline (**4i**)



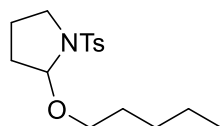
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.31 – 7.21 (m, 3H), 7.17 (d, *J* = 6.8 Hz, 1H), 5.82 (s, 1H), 3.89 – 3.79 (m, 1H), 3.76 – 3.55 (m, 3H), 3.03 (ddd, *J* = 17.5, 11.5, 6.4 Hz, 1H), 2.91 – 2.81 (m, 4H), 1.65 – 1.55 (m, 2H), 1.40 – 1.25 (m, 4H), 0.89 (td, *J* = 7.3, 4.0 Hz, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 133.6, 133.3, 129.0, 128.8, 128.5, 126.6, 83.2, 68.2, 40.5, 38.8, 29.3, 28.4, 27.6, 22.5, 14.1.

**HRMS** (ESI) *m/z* calcd for C<sub>15</sub>H<sub>23</sub>NNaO<sub>3</sub>S<sup>+</sup> (M+Na<sup>+</sup>): 320.1291, found: 320.1290.

2-(Pentyloxy)-1-tosylpyrrolidine (**4j**)



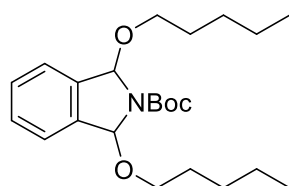
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.72 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.19 (d, *J* = 5.0 Hz, 1H), 3.73 (dt, *J* = 9.5, 6.8 Hz, 1H), 3.51 – 3.36 (m, 2H), 3.14 (td, *J* = 9.7, 7.6 Hz, 1H), 2.42 (s, 3H), 2.10 – 1.96 (m, 1H), 1.90 – 1.80 (m, 1H), 1.80 – 1.70 (m, 1H), 1.60 – 1.51 (m, 2H), 1.43 – 1.25 (m, 5H), 0.90 (t, *J* = 7.0 Hz, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 143.4, 136.0, 129.6, 127.4, 90.3, 67.7, 47.2, 32.8, 29.2, 28.4, 23.2, 22.5, 21.5, 14.1.

**HRMS** (ESI) *m/z* calcd for C<sub>16</sub>H<sub>25</sub>NNaO<sub>3</sub>S<sup>+</sup> (M+Na<sup>+</sup>): 334.1447, found: 334.1443.

*t*-Butyl 1,3-bis(pentyloxy)isoindoline-2-carboxylate (**4k**)



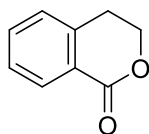
Colorless oil.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.39 (s, 4H), 6.19 (s, 2H), 3.61 (dd, *J* = 42.4, 35.4 Hz, 4H), 1.65 – 1.49 (m, 13H), 1.39 – 1.26 (m, 8H), 0.88 (t, *J* = 7.0 Hz, 6H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 155.1, 129.3, 123.8, 89.1, 80.9, 29.7, 28.4, 22.5, 14.1.

**HRMS** (ESI) *m/z* calcd for C<sub>23</sub>H<sub>37</sub>NNaO<sub>4</sub><sup>+</sup> (M+Na<sup>+</sup>): 414.2615, found: 414.2613.

Isochroman-1-one (**5**)<sup>[6]</sup>



Colorless oil.

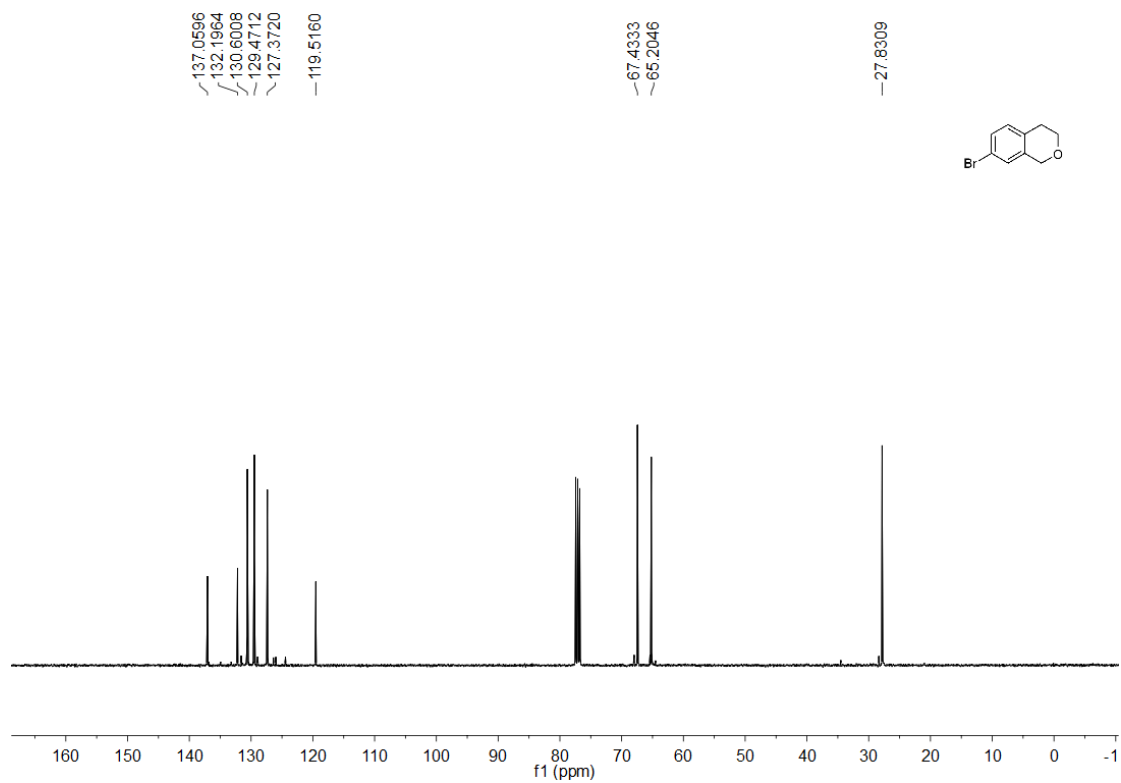
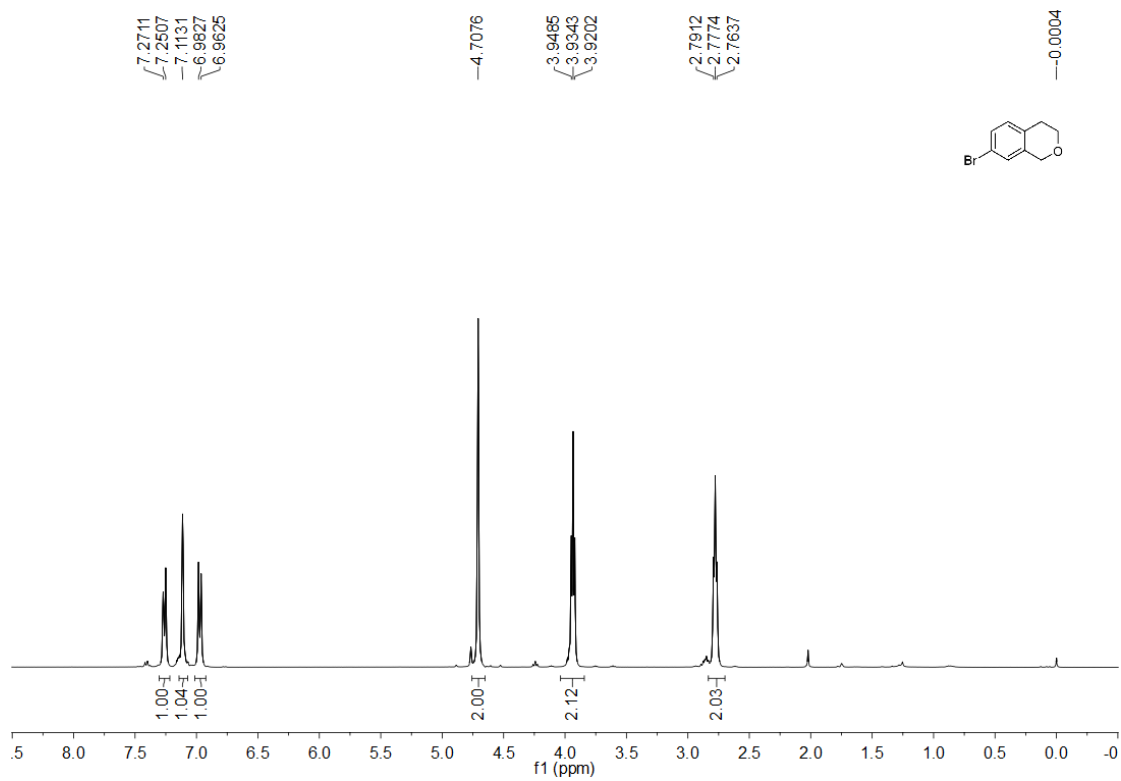
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.09 (d,  $J = 7.5$  Hz, 1H), 7.54 (t,  $J = 7.2$  Hz, 1H), 7.40 (t,  $J = 7.3$  Hz, 1H), 7.28 (d,  $J = 7.4$  Hz, 1H), 4.54 (s, 2H), 3.07 (s, 2H).

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.1, 139.6, 133.7, 130.4, 127.7, 127.3, 125.3, 67.3, 27.8.

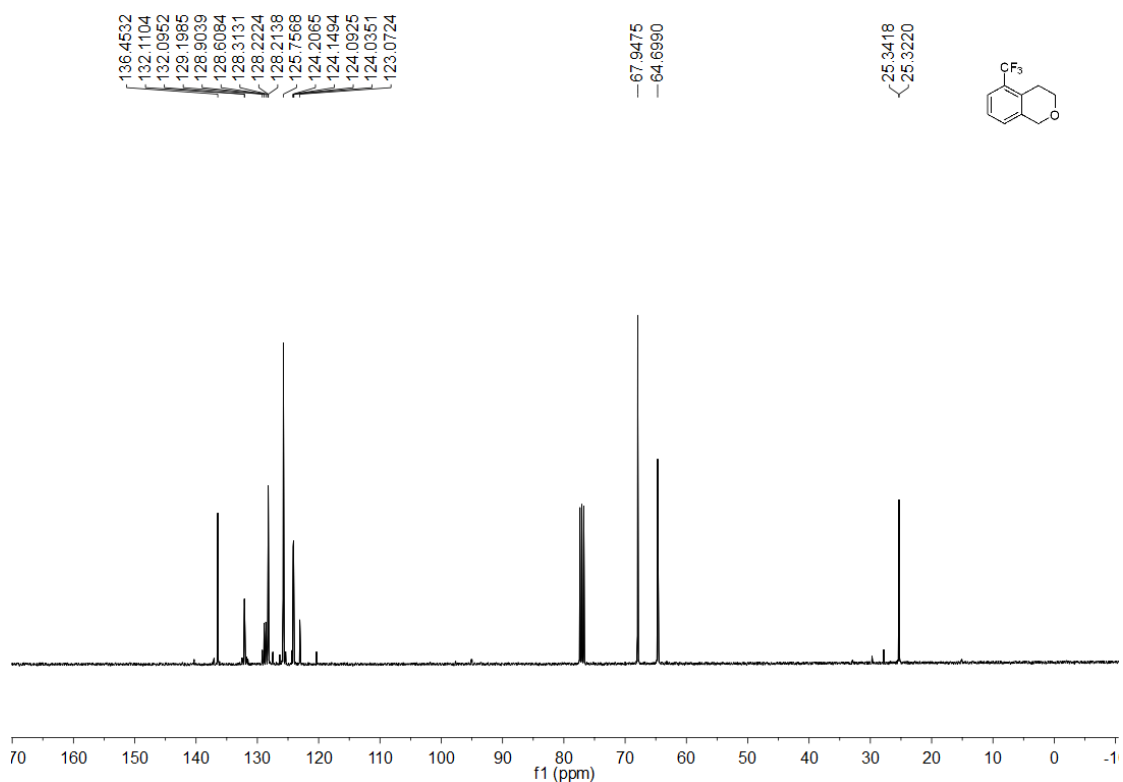
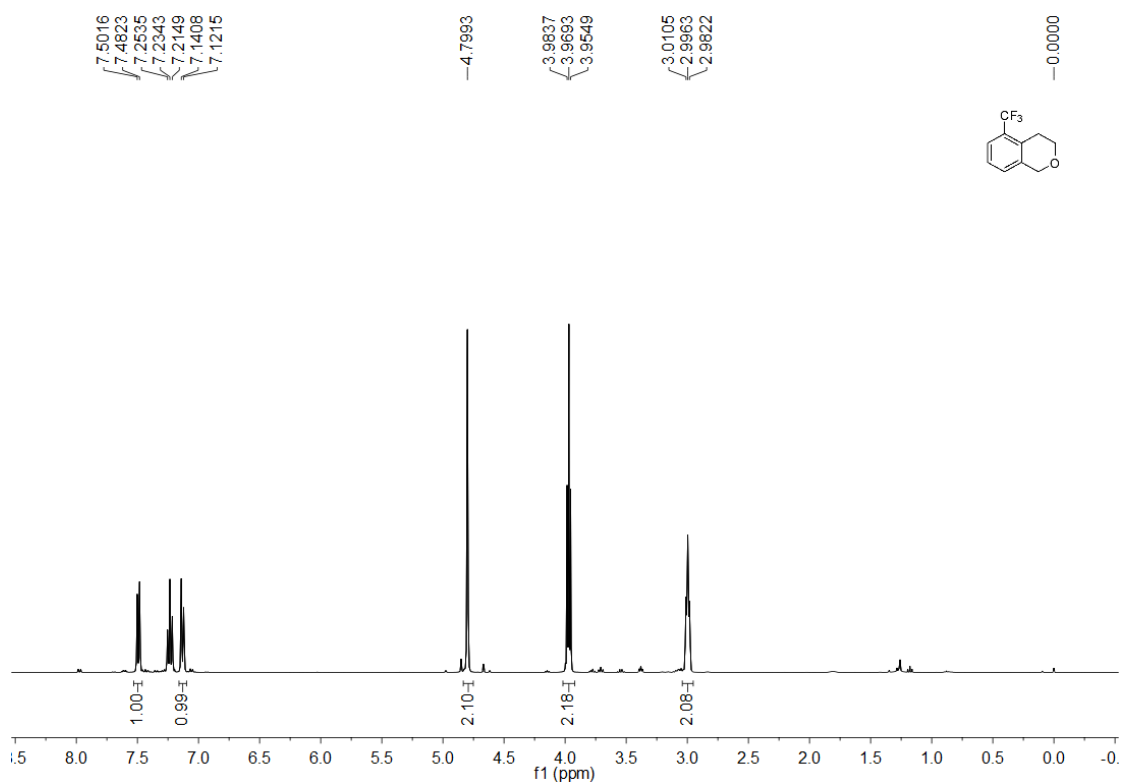
The spectral data obtained were identical with those reported in literature.

## 10) NMR spectrum for the substrates and products

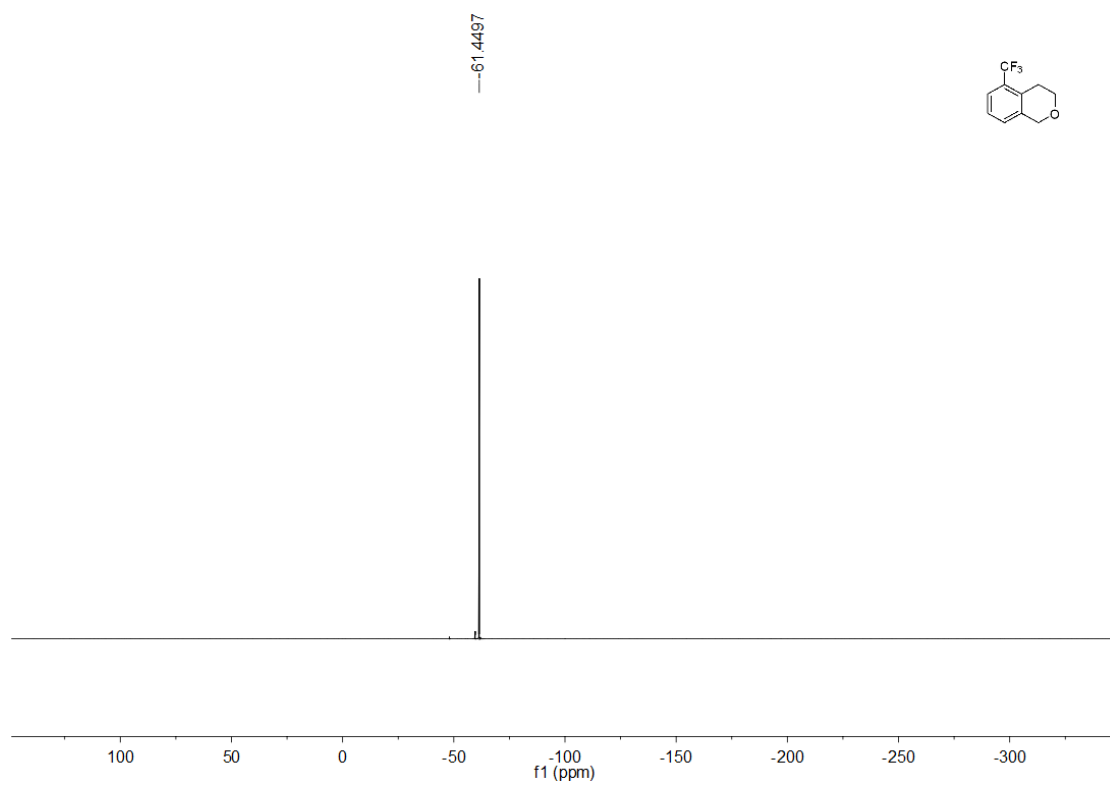
### 7-Bromoisochromane (**1b**)



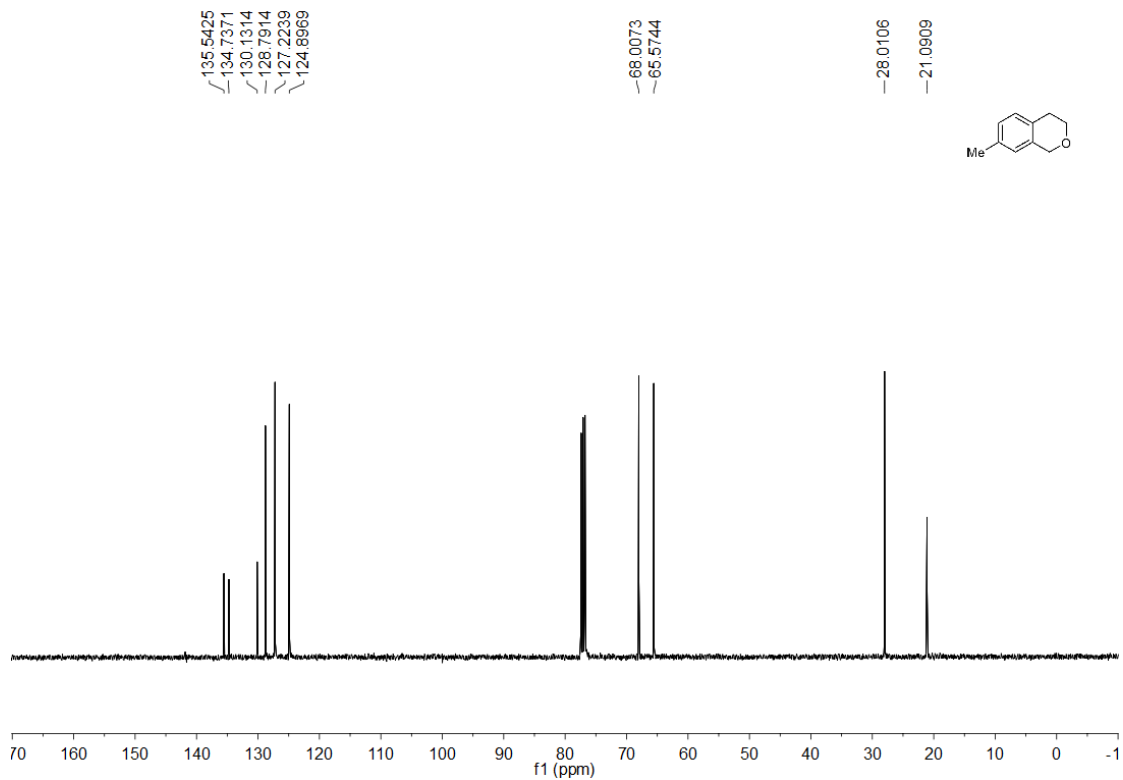
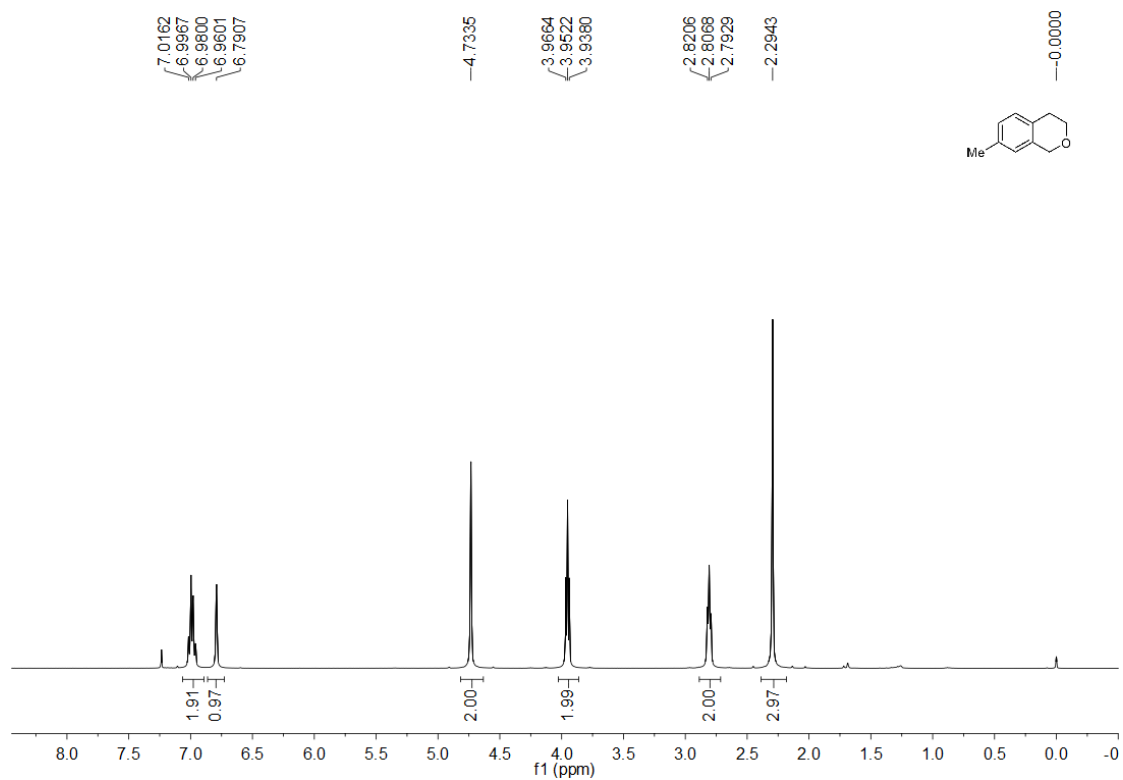
# 5-(Trifluoromethyl)isochromane (1c)



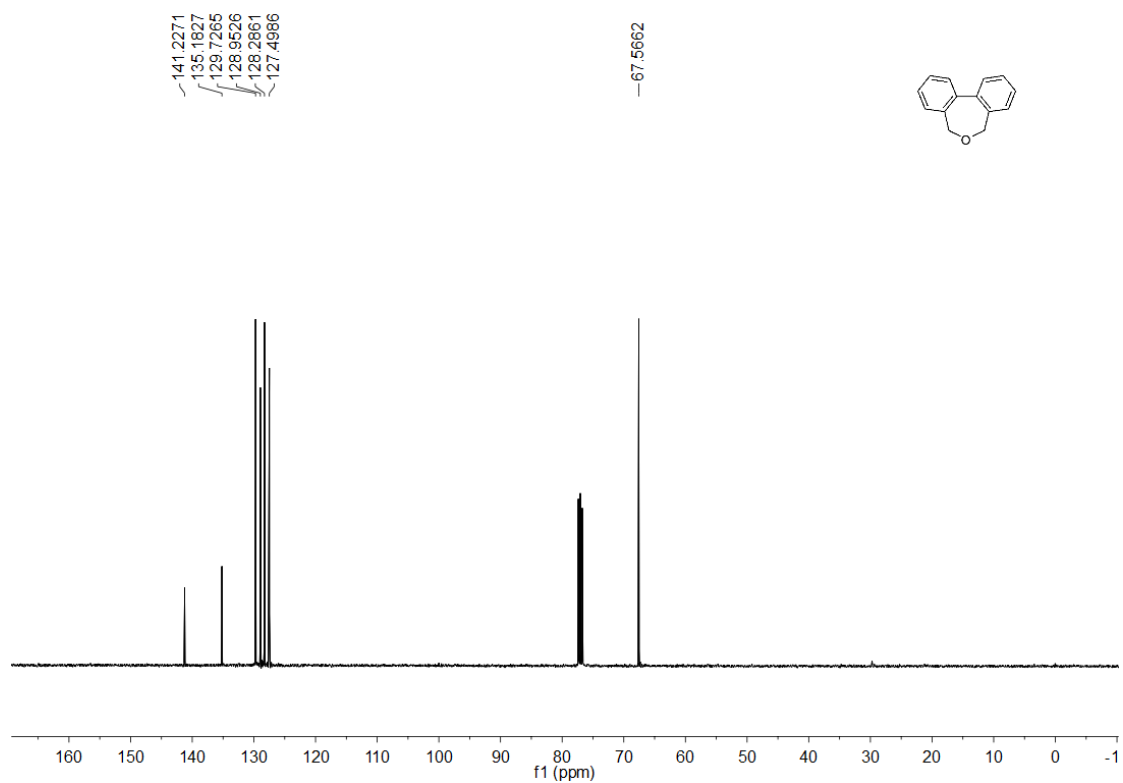
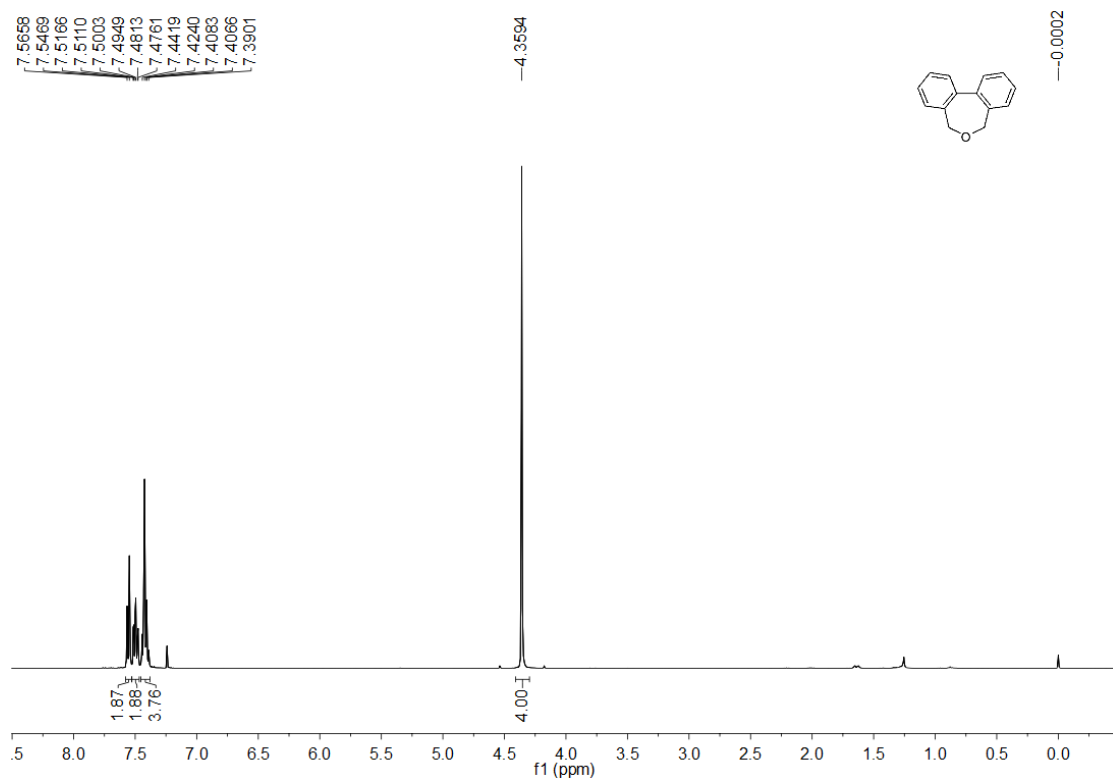




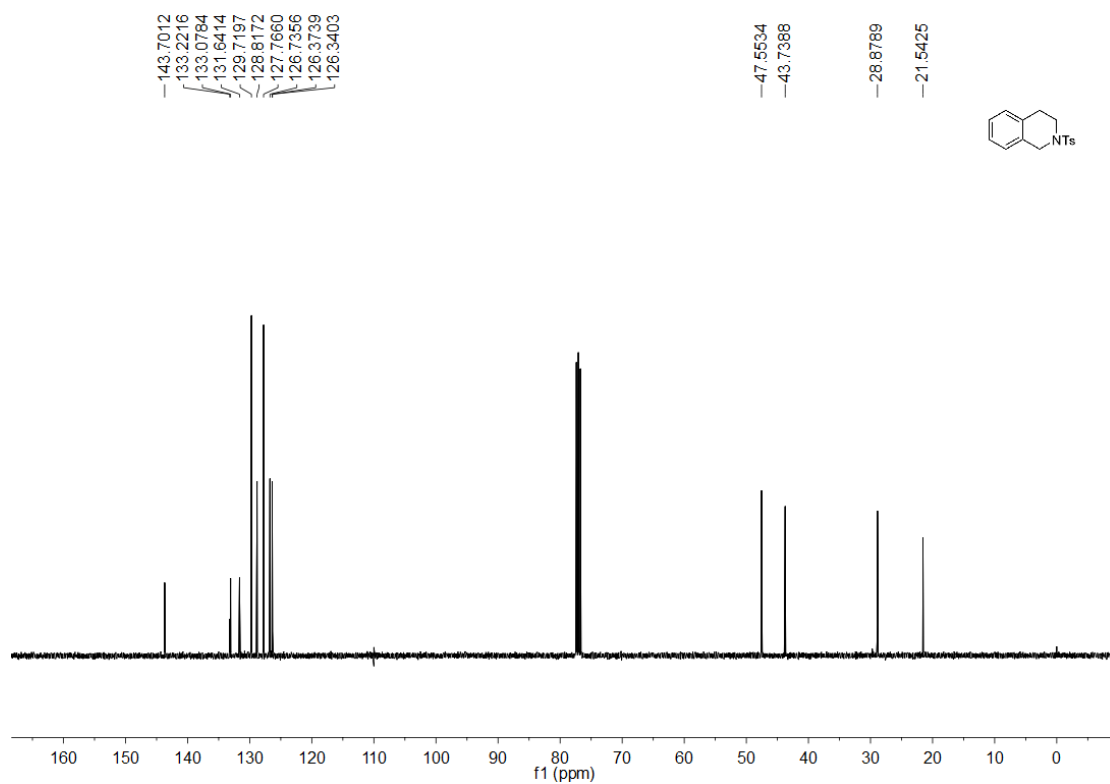
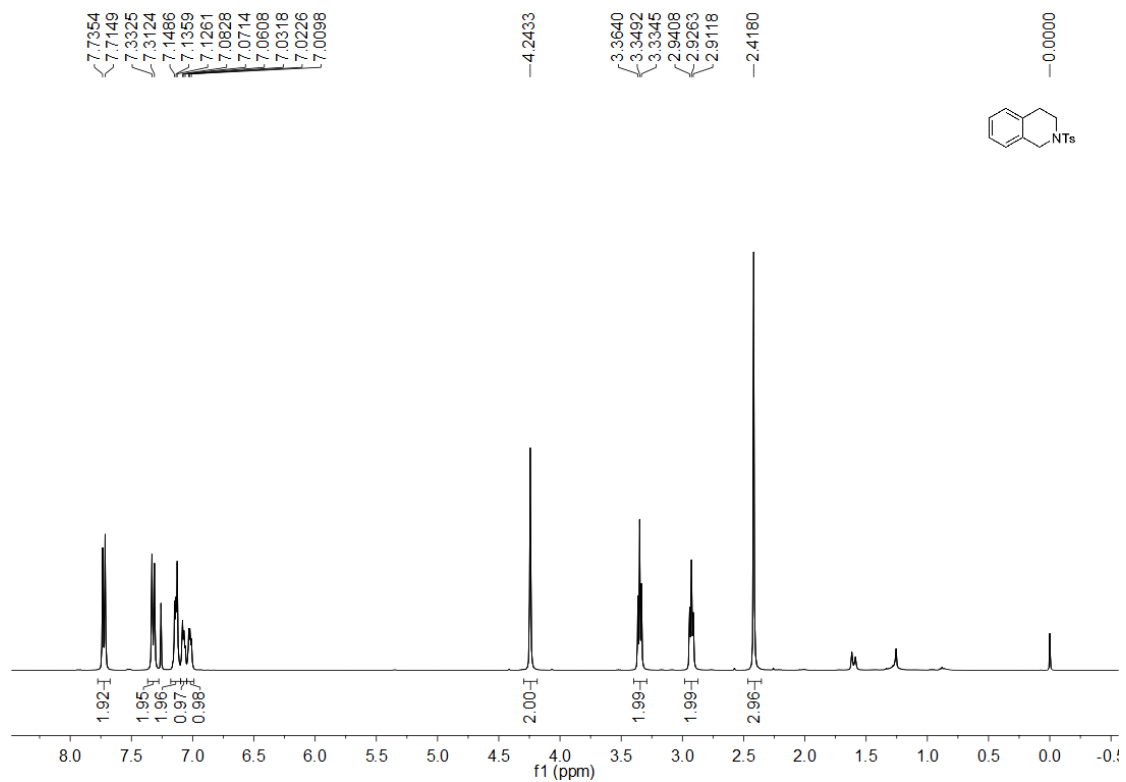
# 7-Methylisochromane (1d)



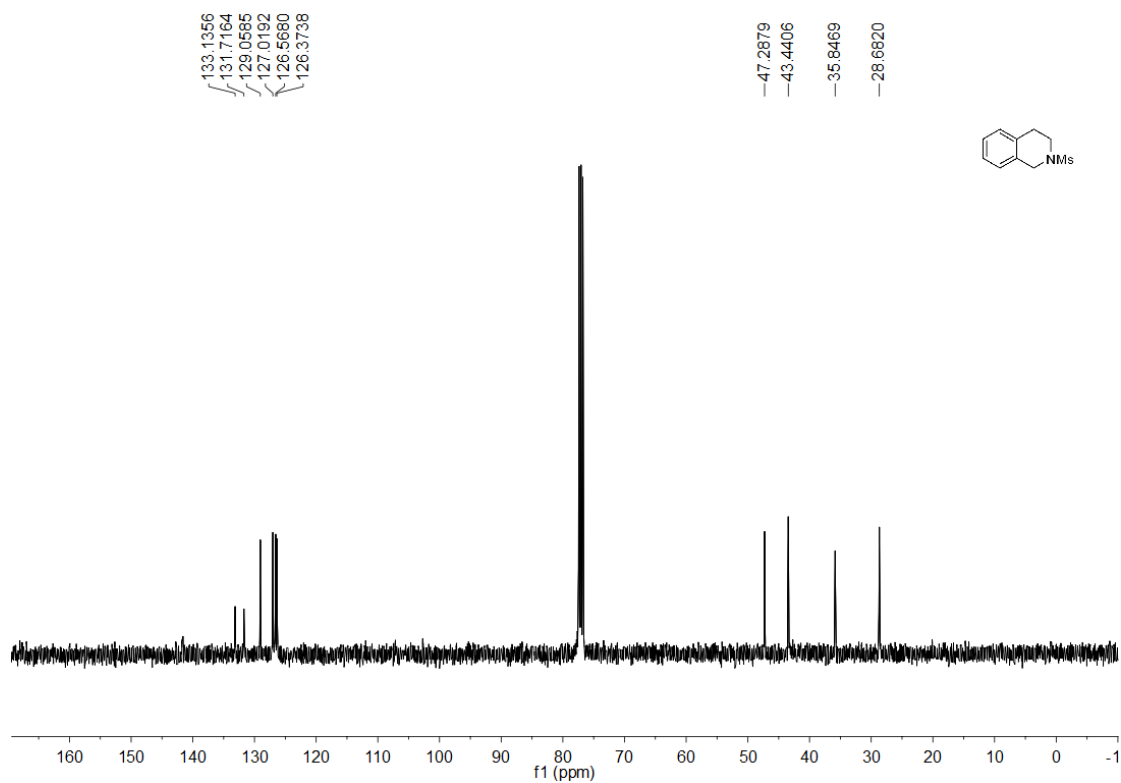
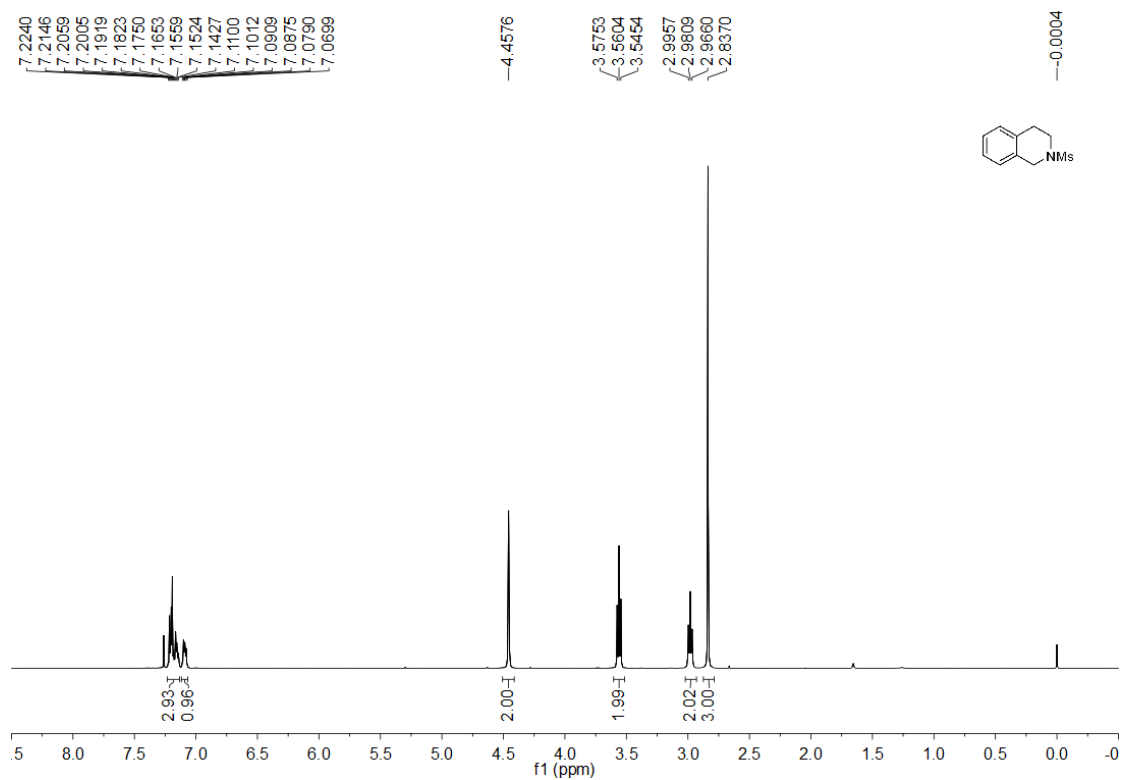
# 5,7-Dihydrodibenzo[c,e]oxepine (**1e**)



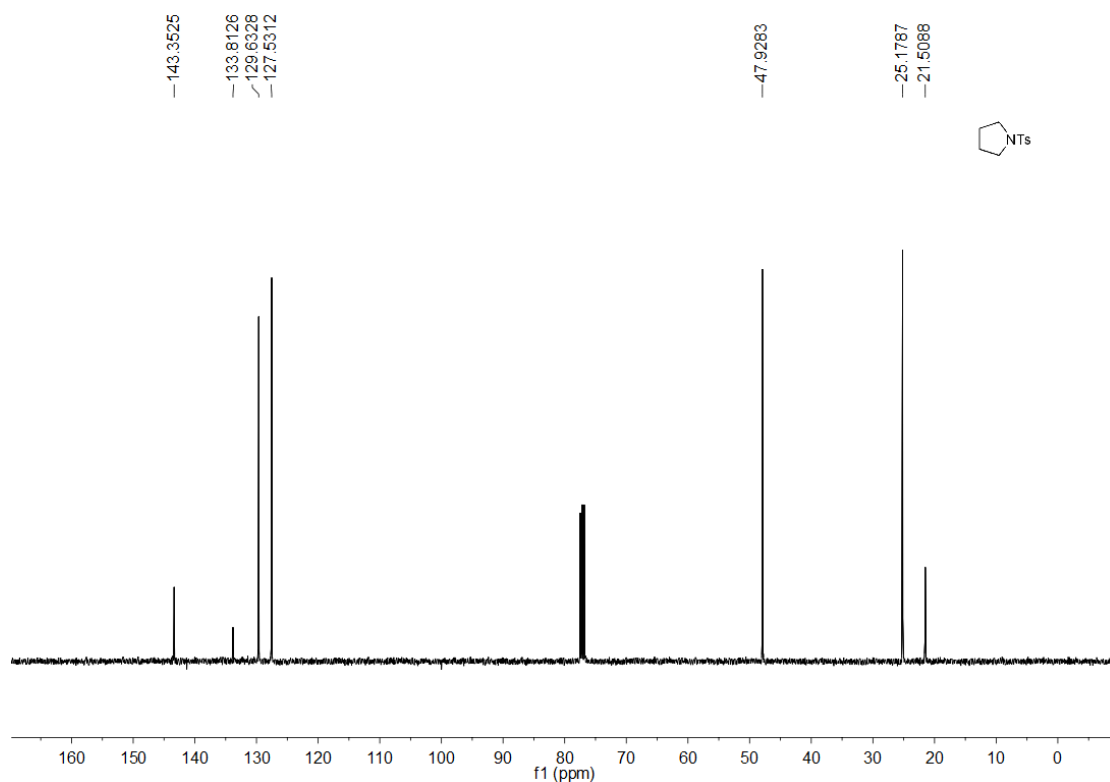
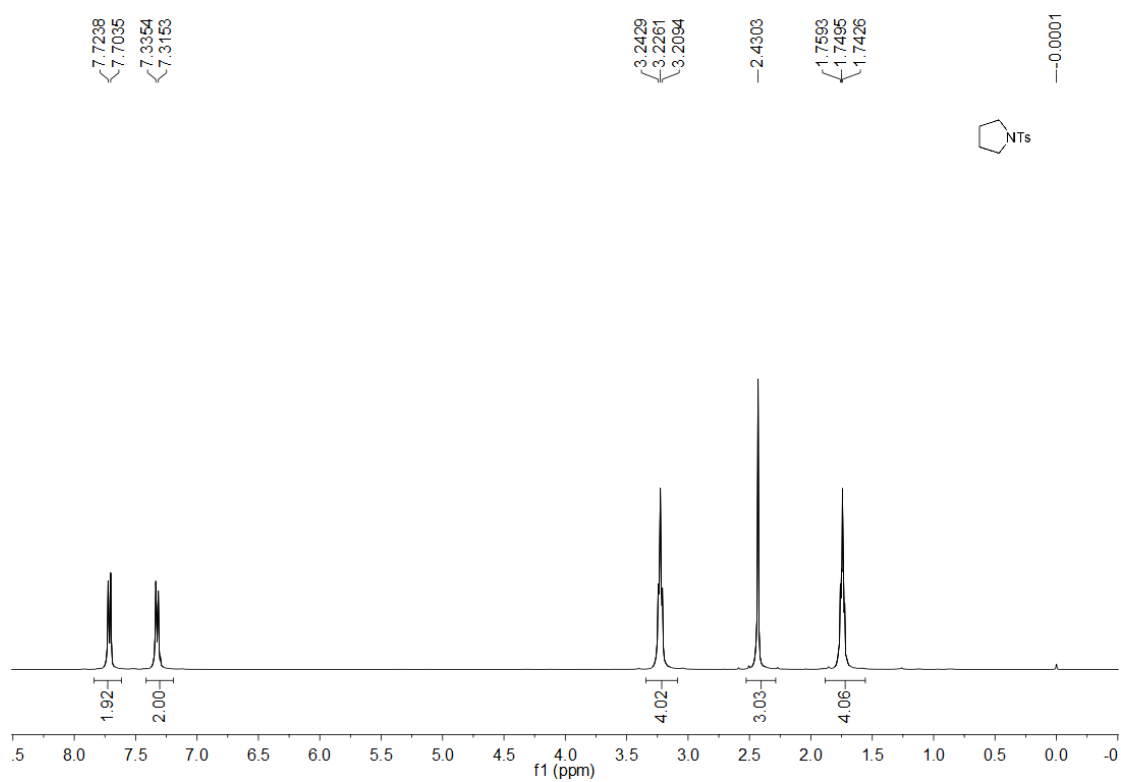
# 2-Tosyl-1,2,3,4-tetrahydroisoquinoline (**1h**)



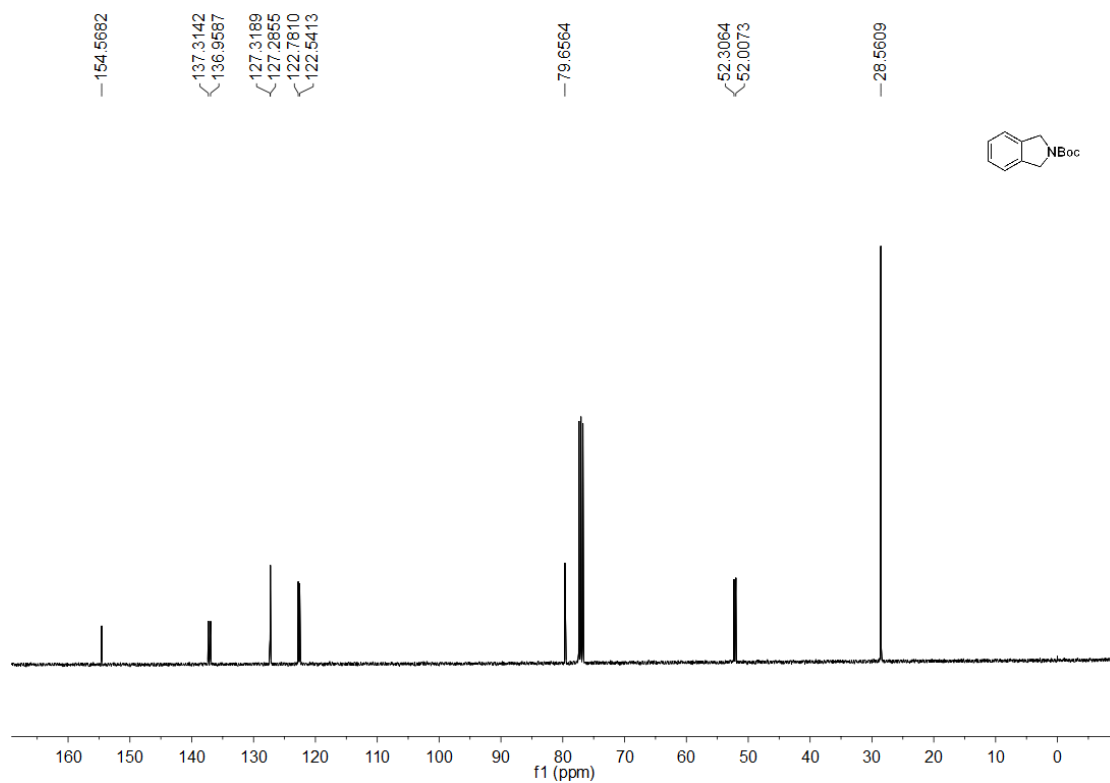
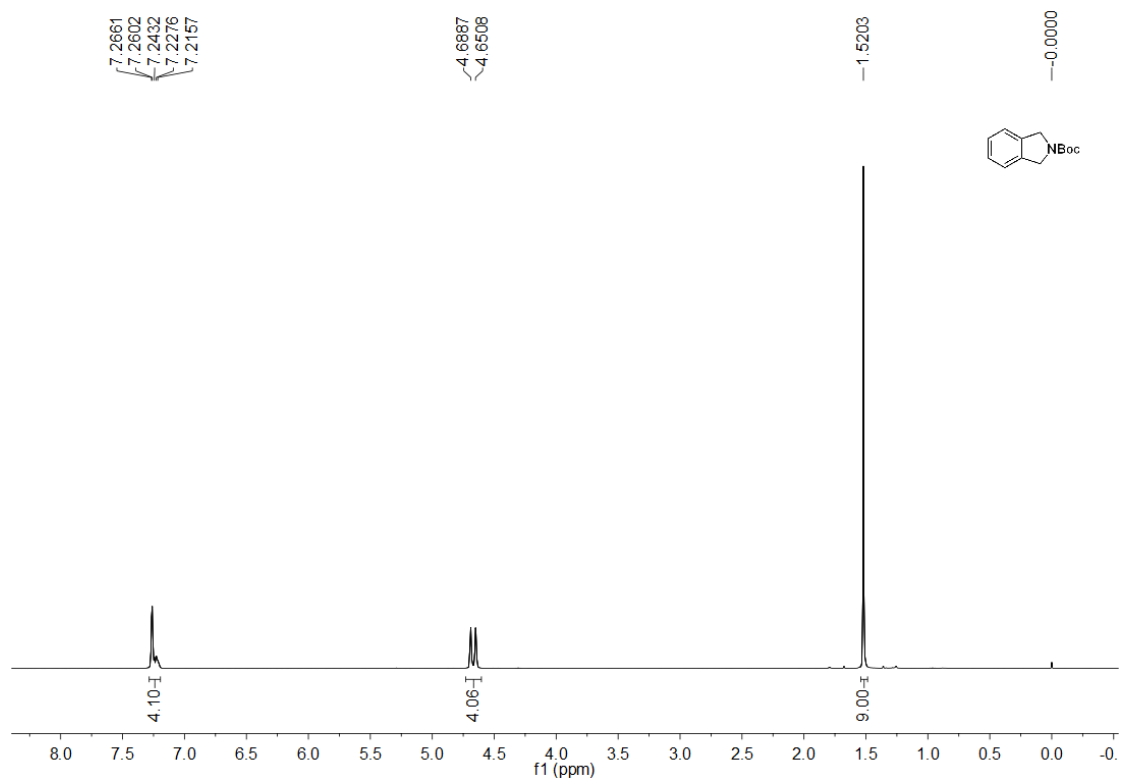
2-(Methylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (**1i**)



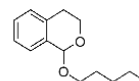
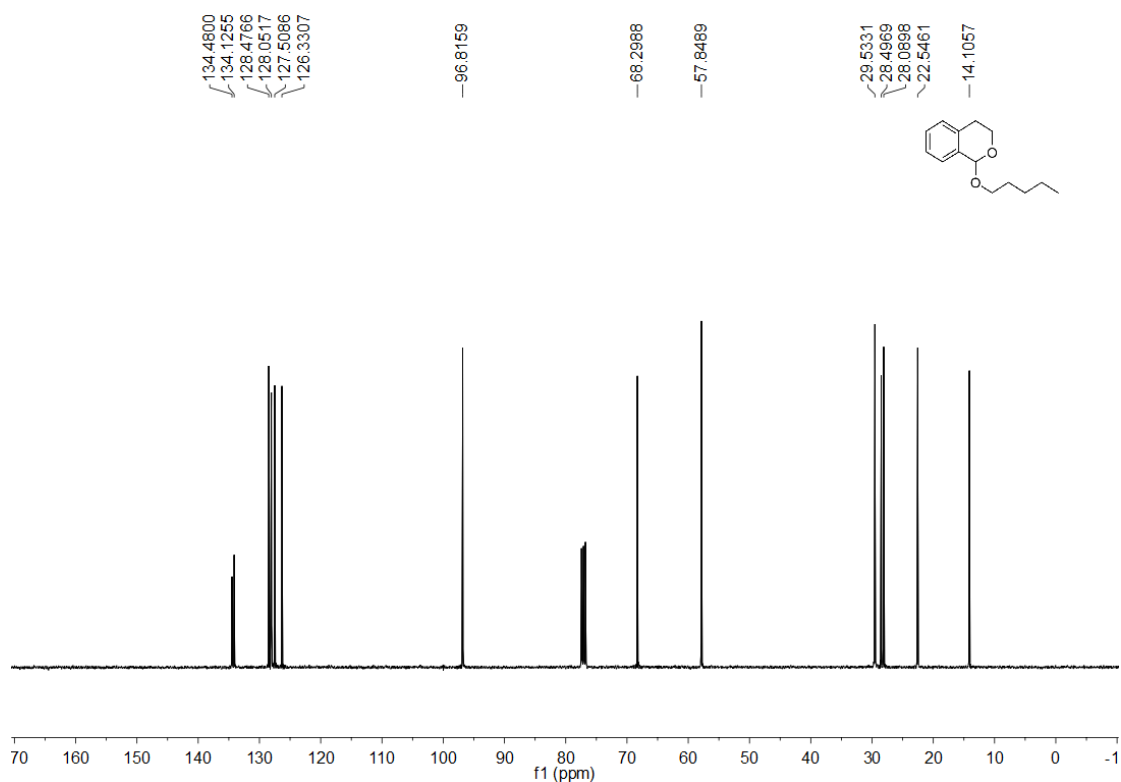
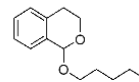
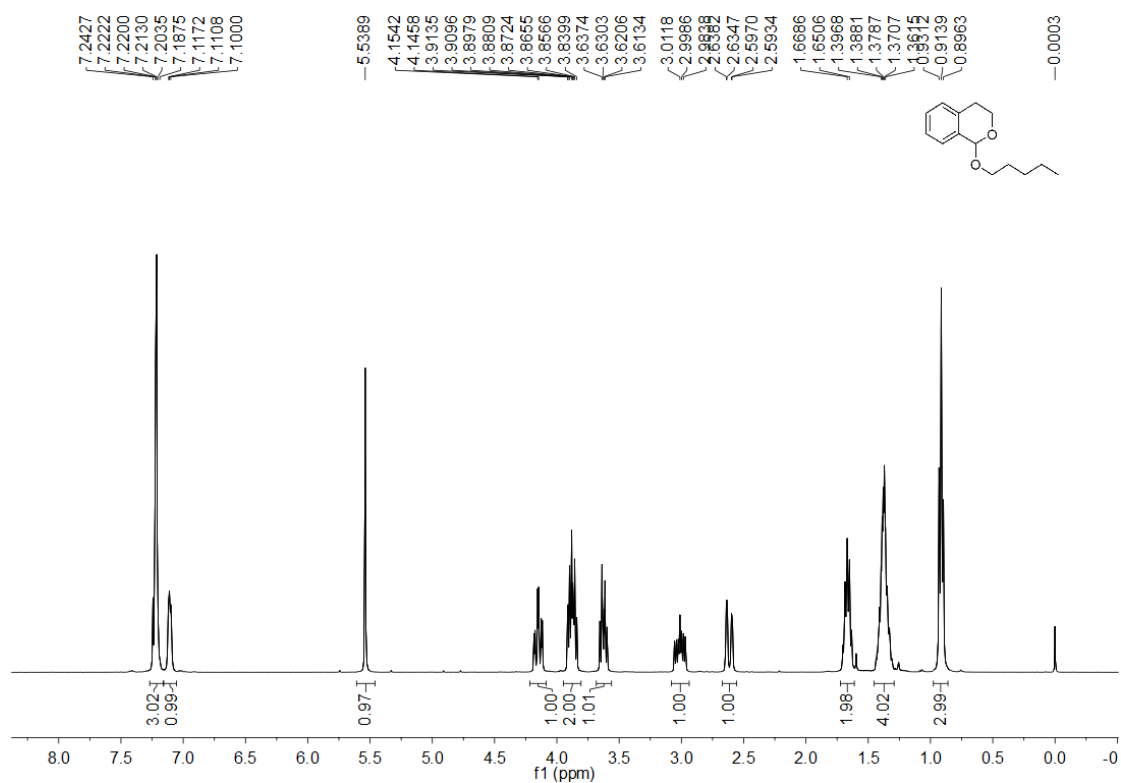
# 1-Tosylpyrrolidine (**1j**)



*t*-Butyl isoindoline-2-carboxylate (**1k**)

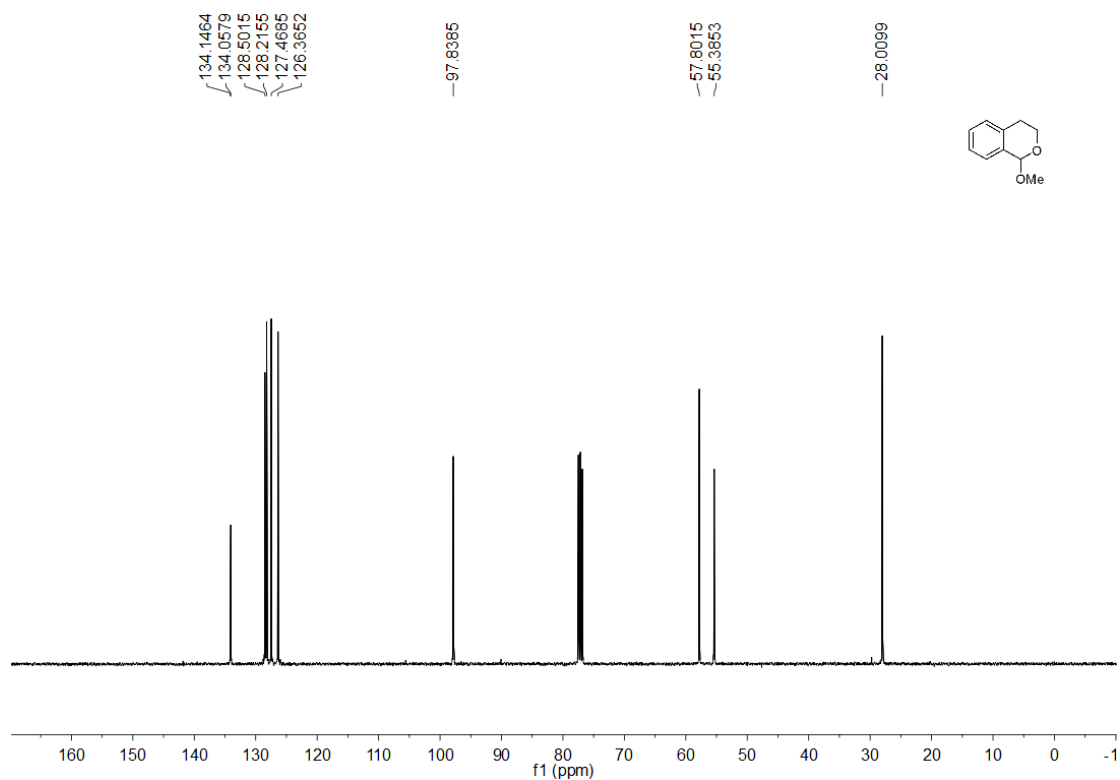
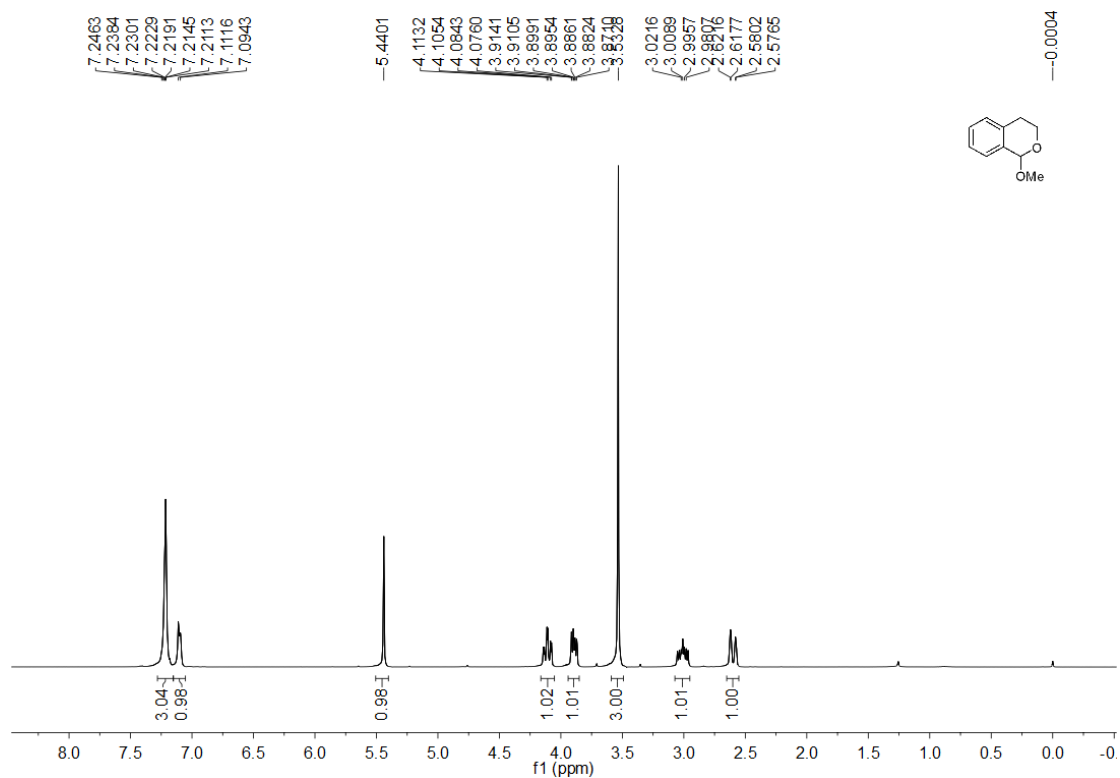


# 1-(Pentyloxy)isochromane (3a)

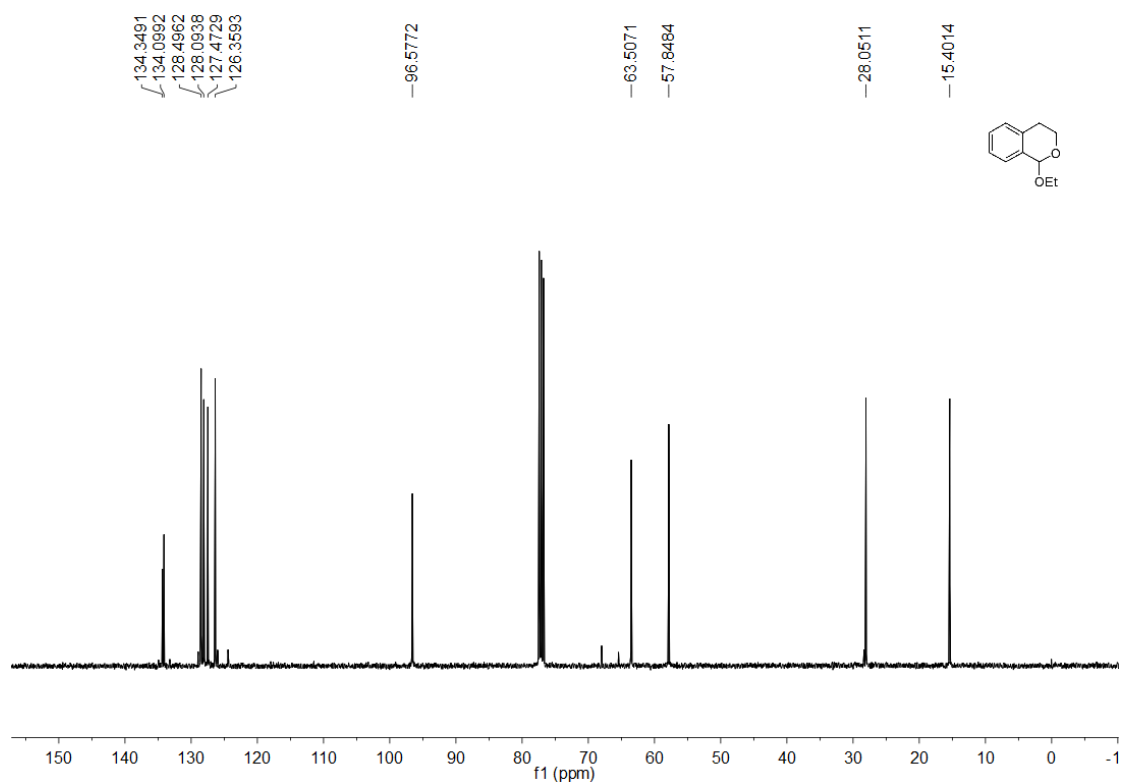
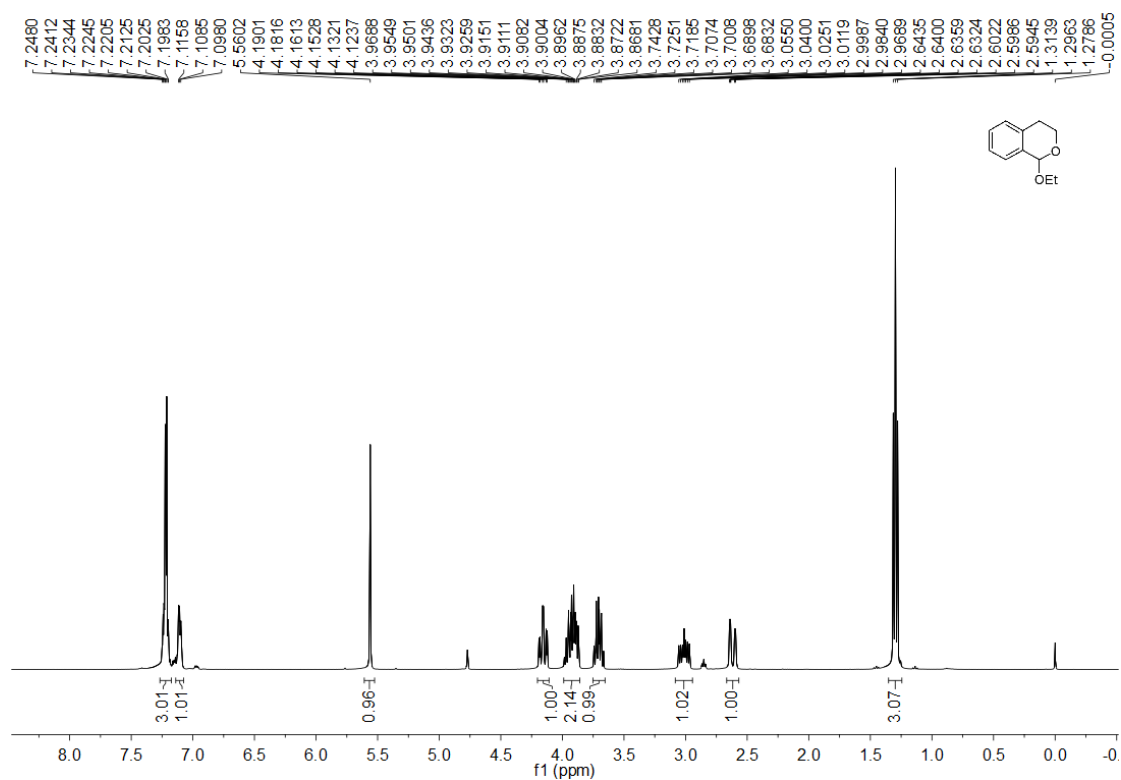




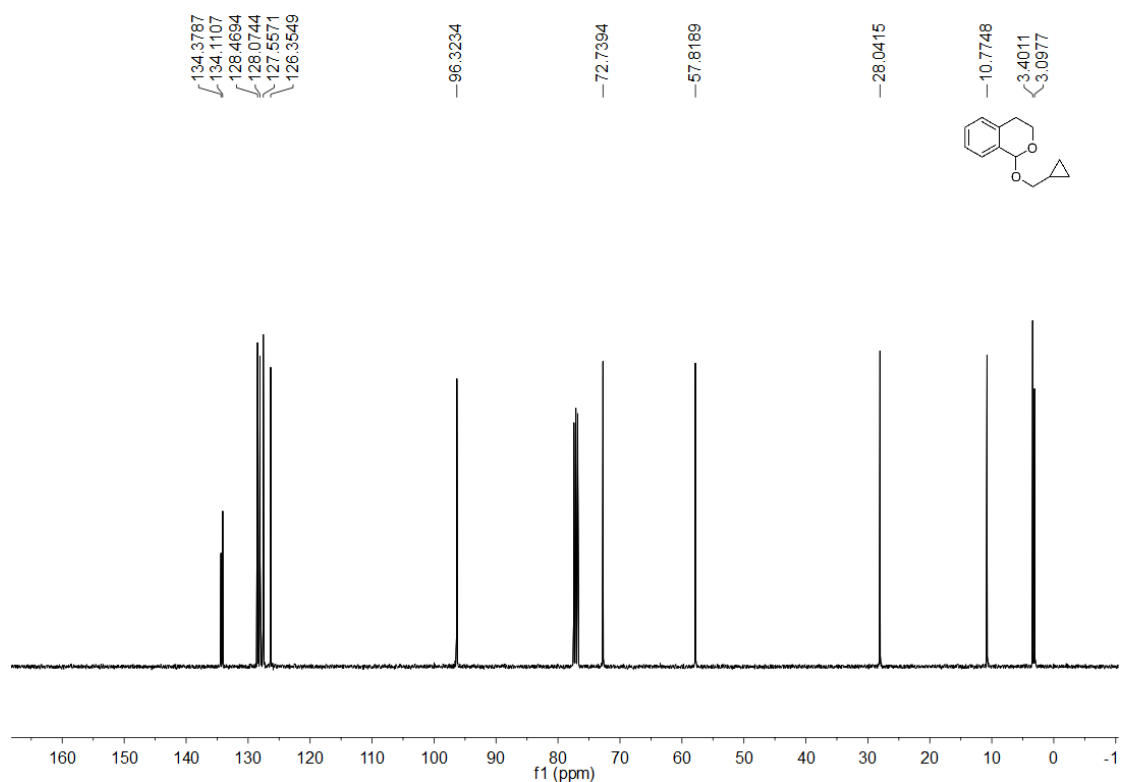
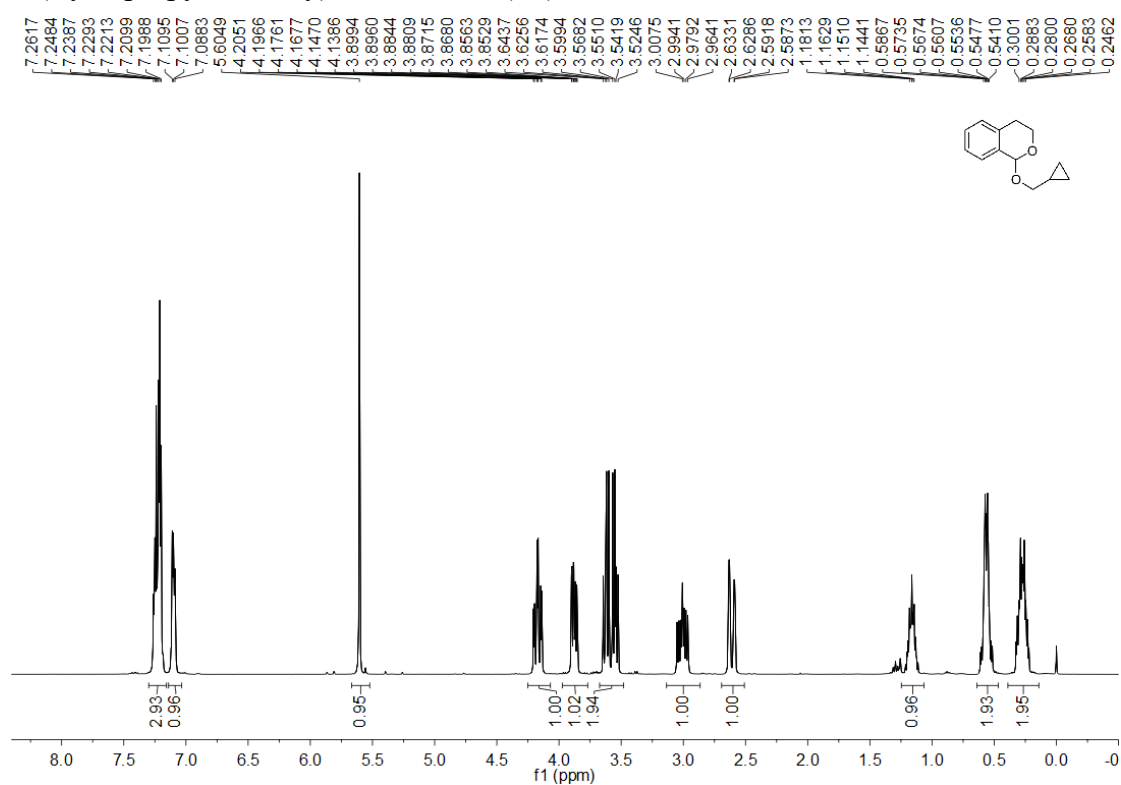
# 1-(Methoxy)isochromane (**3b**)



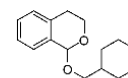
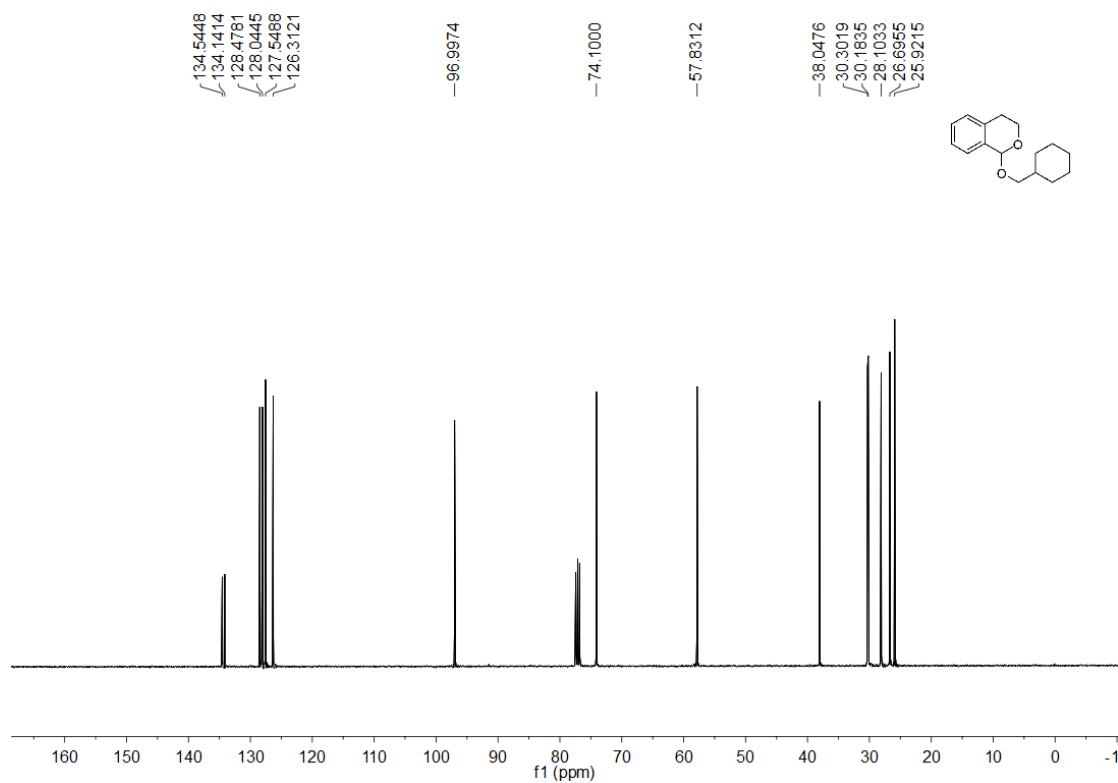
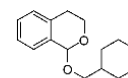
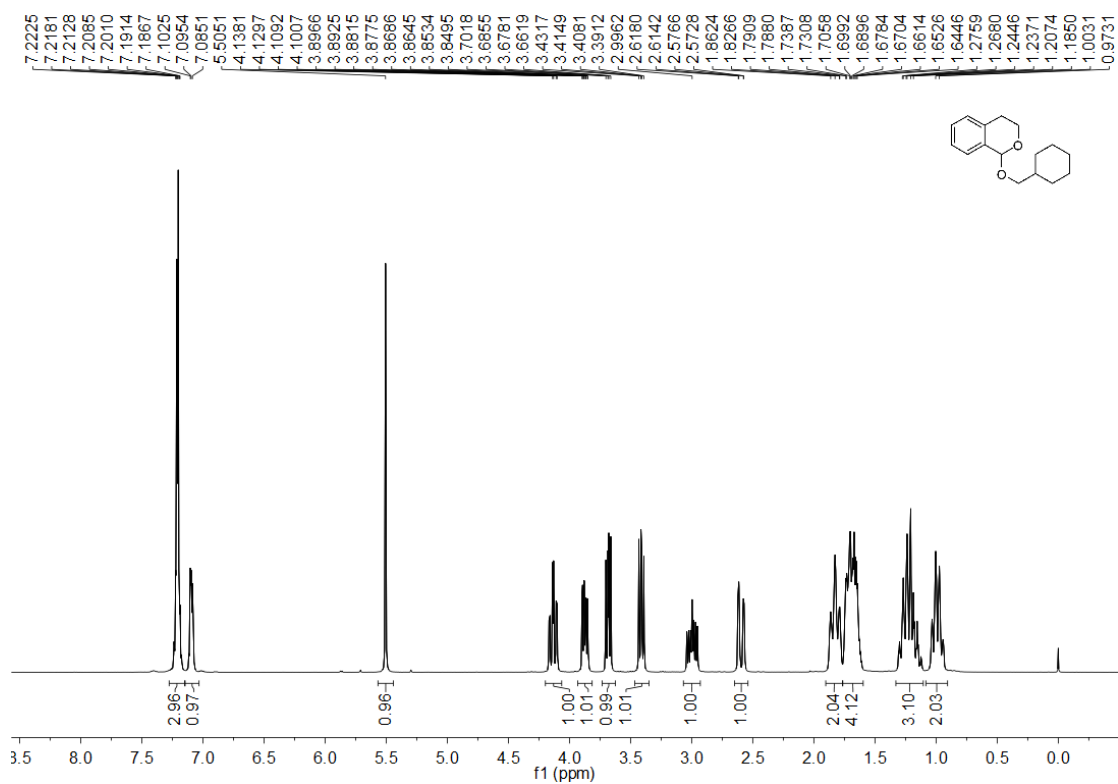
# 1-(Ethoxy)isochromane (3c)



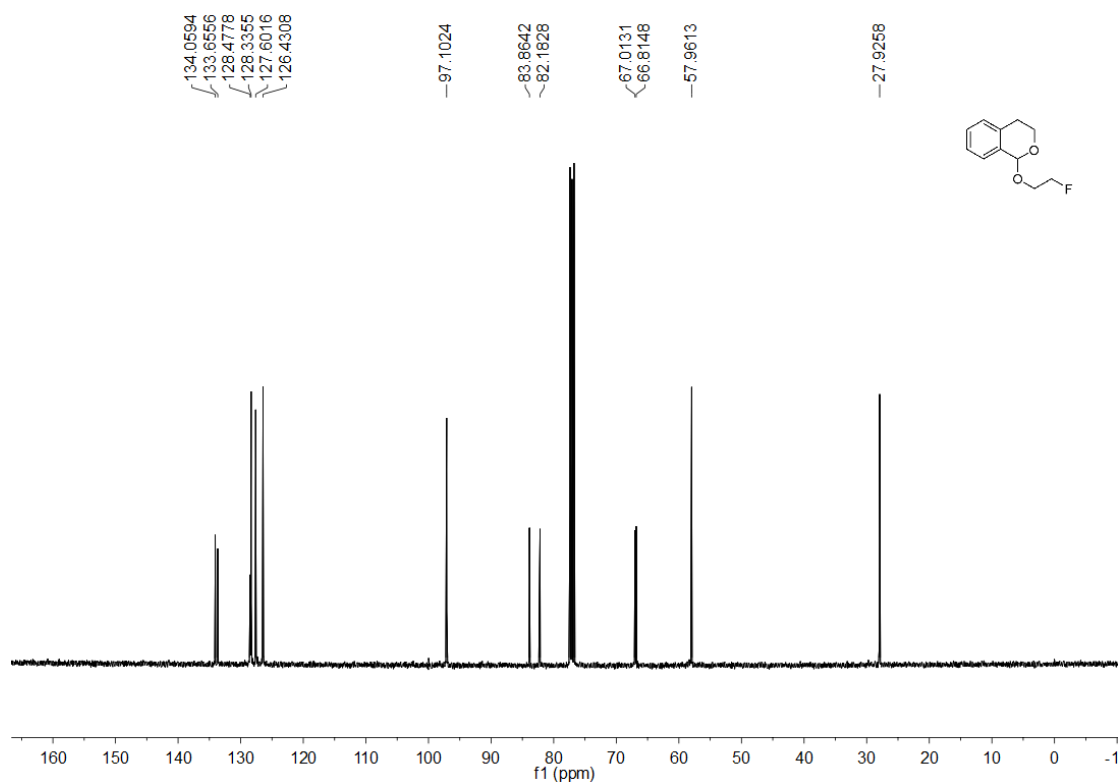
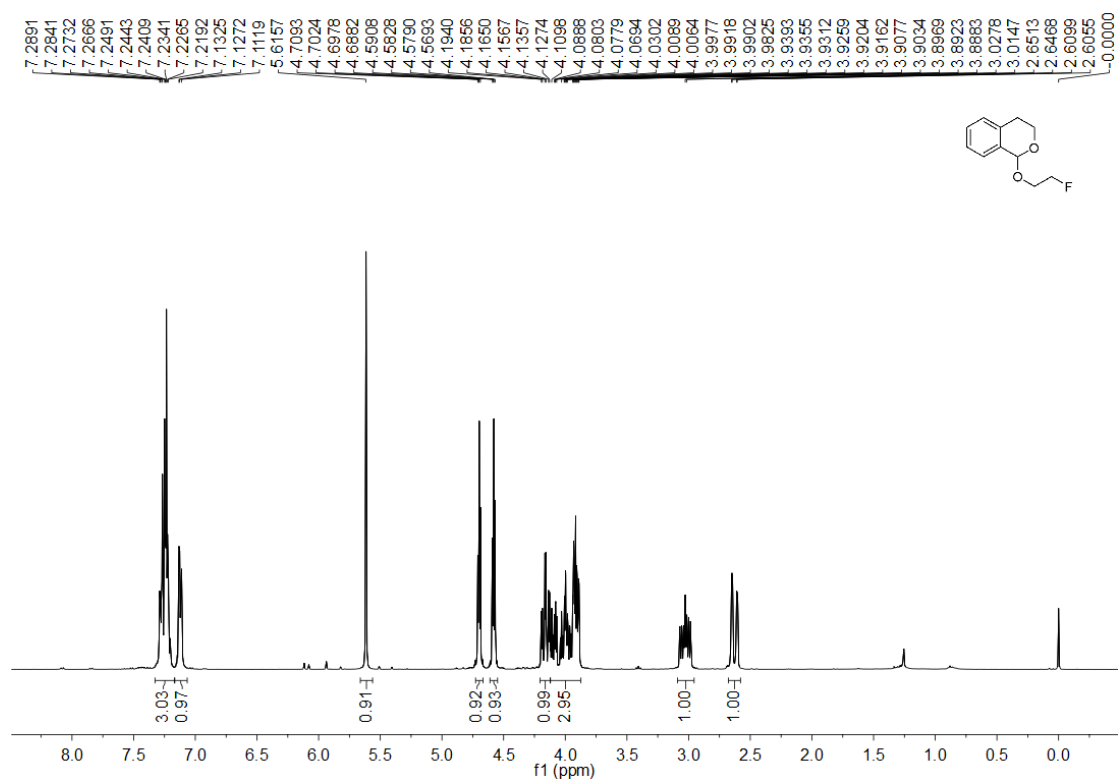
# 1-(Cyclopropylmethoxy)isochromane (3d)

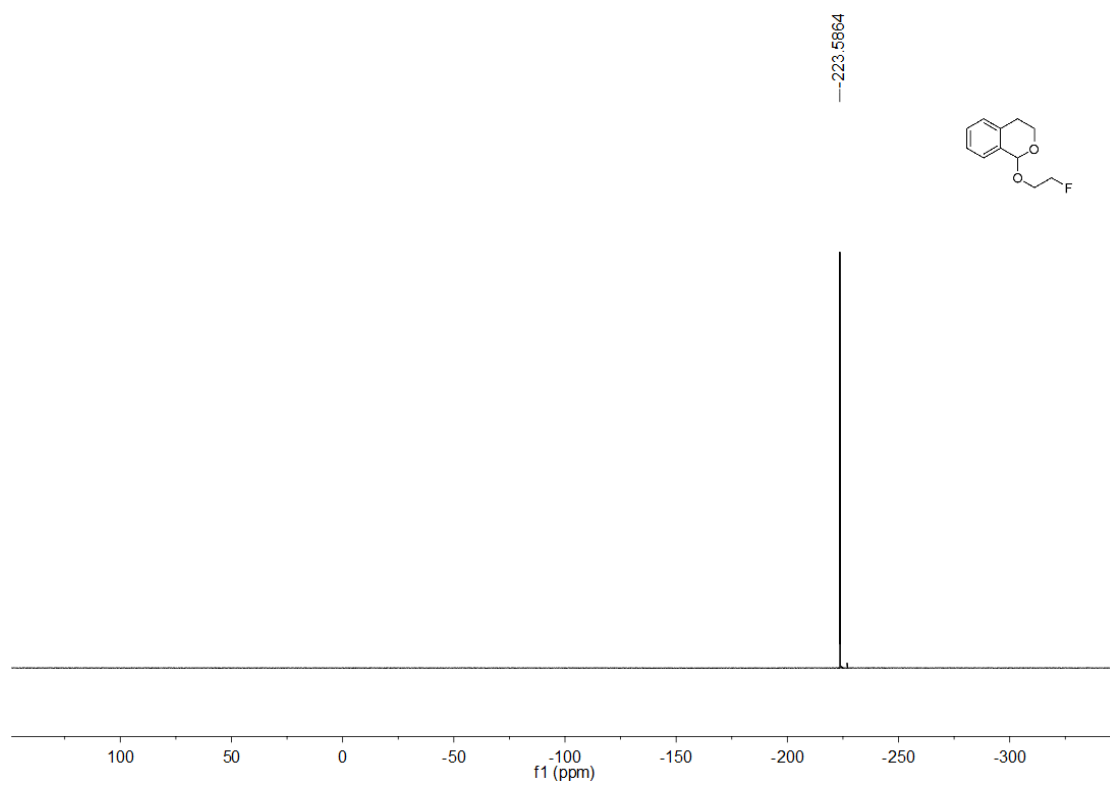


# 1-(Cyclohexylmethoxy)isochromane (3e)

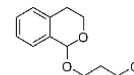
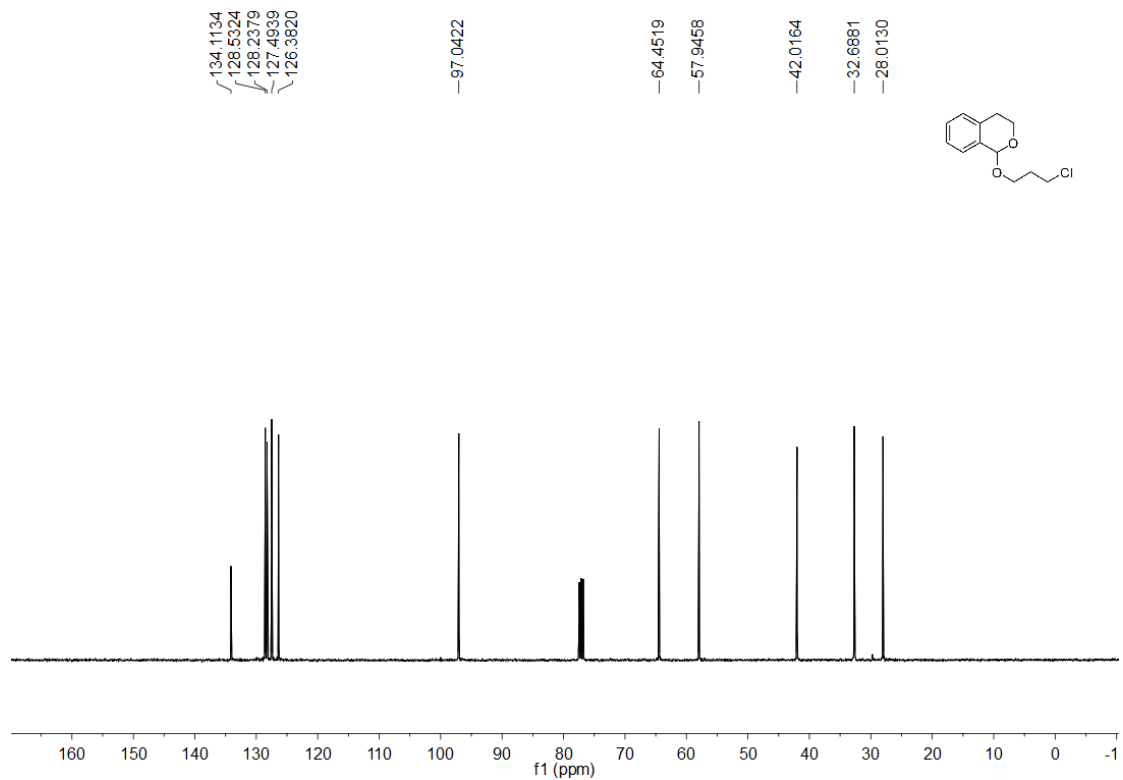
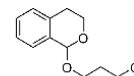
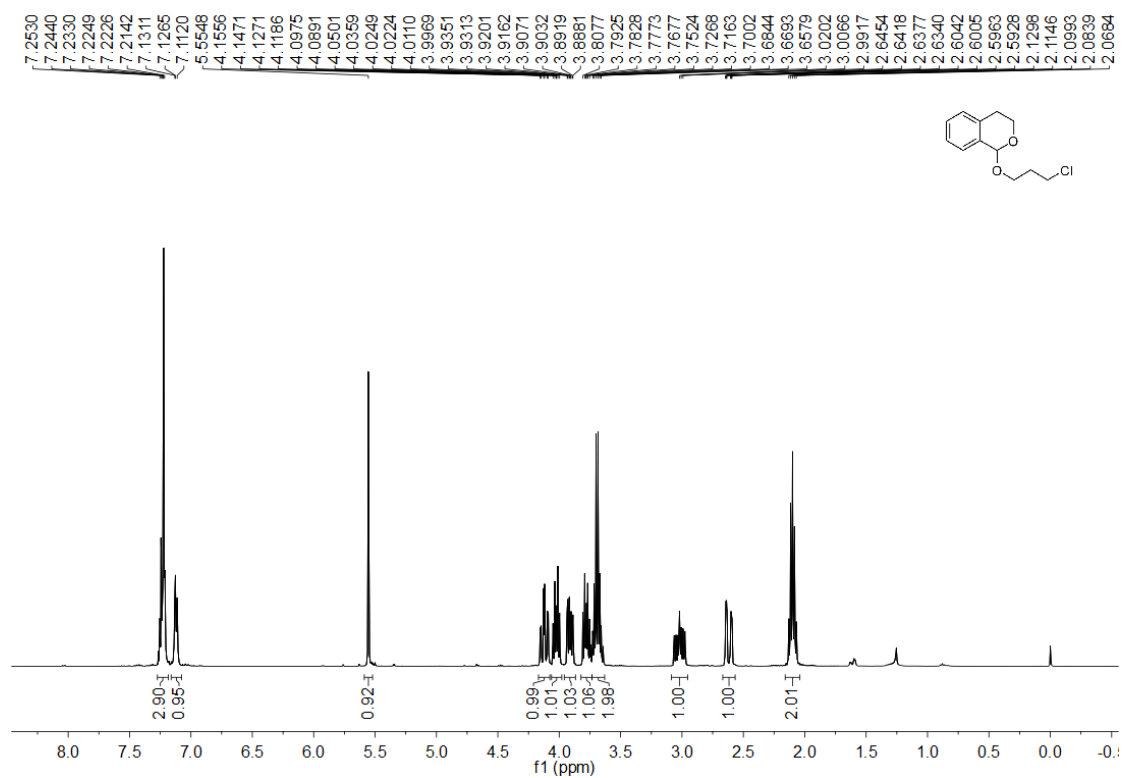


# 1-(2-Fluoroethoxy)isochromane (3f)

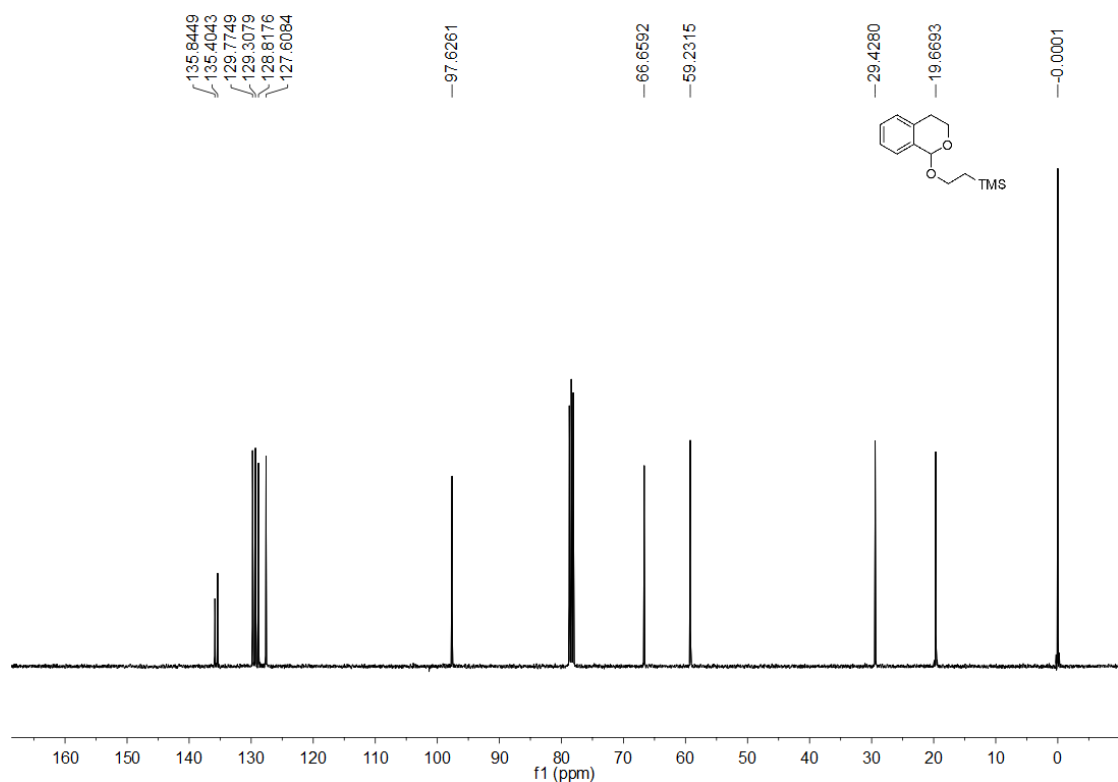
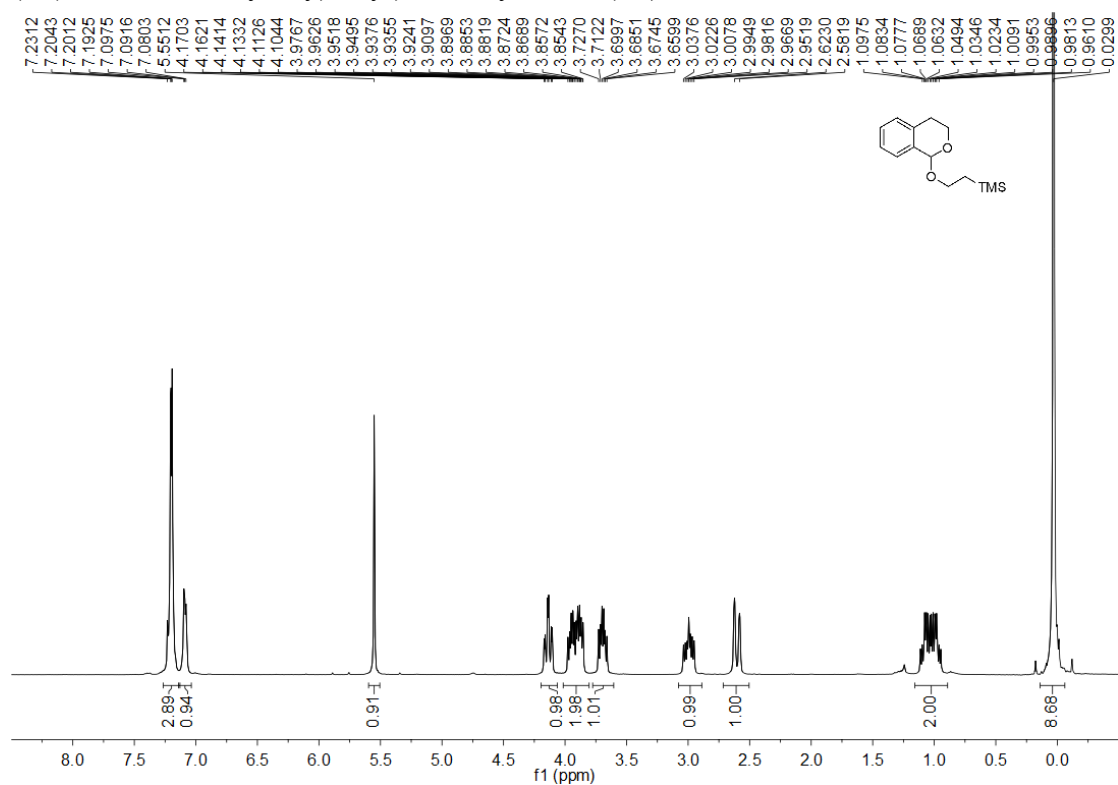




# 1-(3-Chloropropoxy)isochromane (3g)

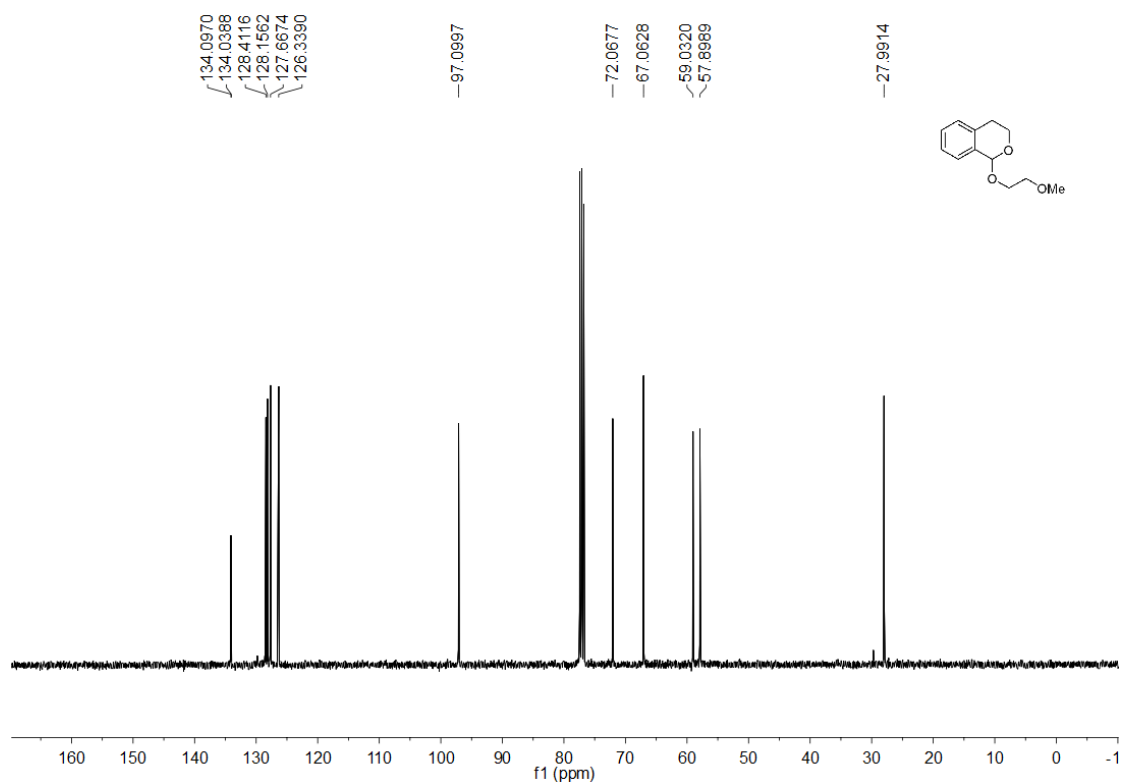
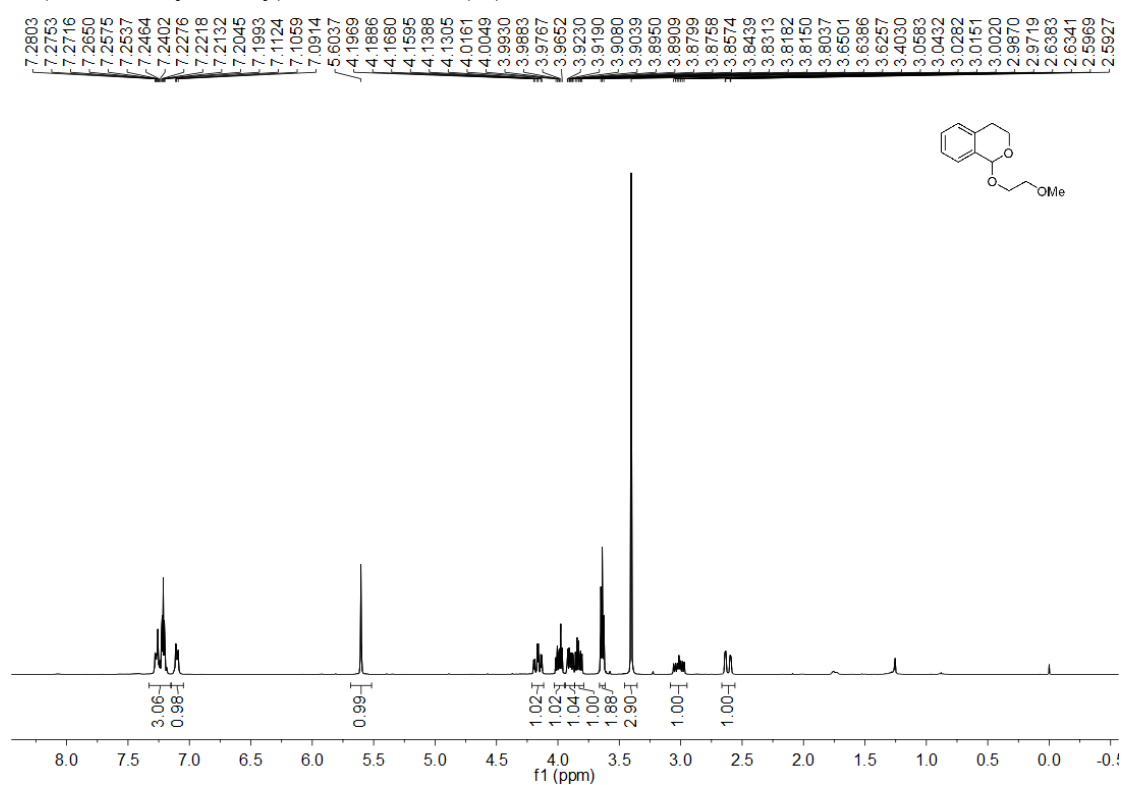


(2-(Isochroman-1-yloxy)ethyl)trimethylsilane (**3h**)

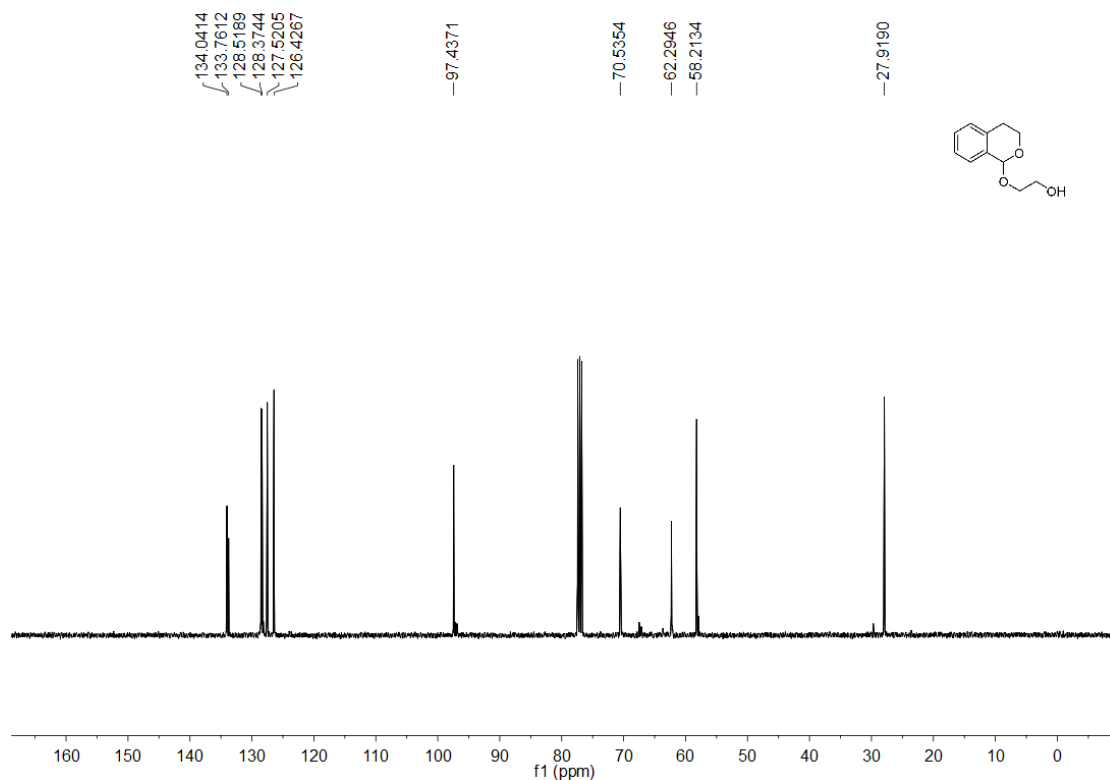
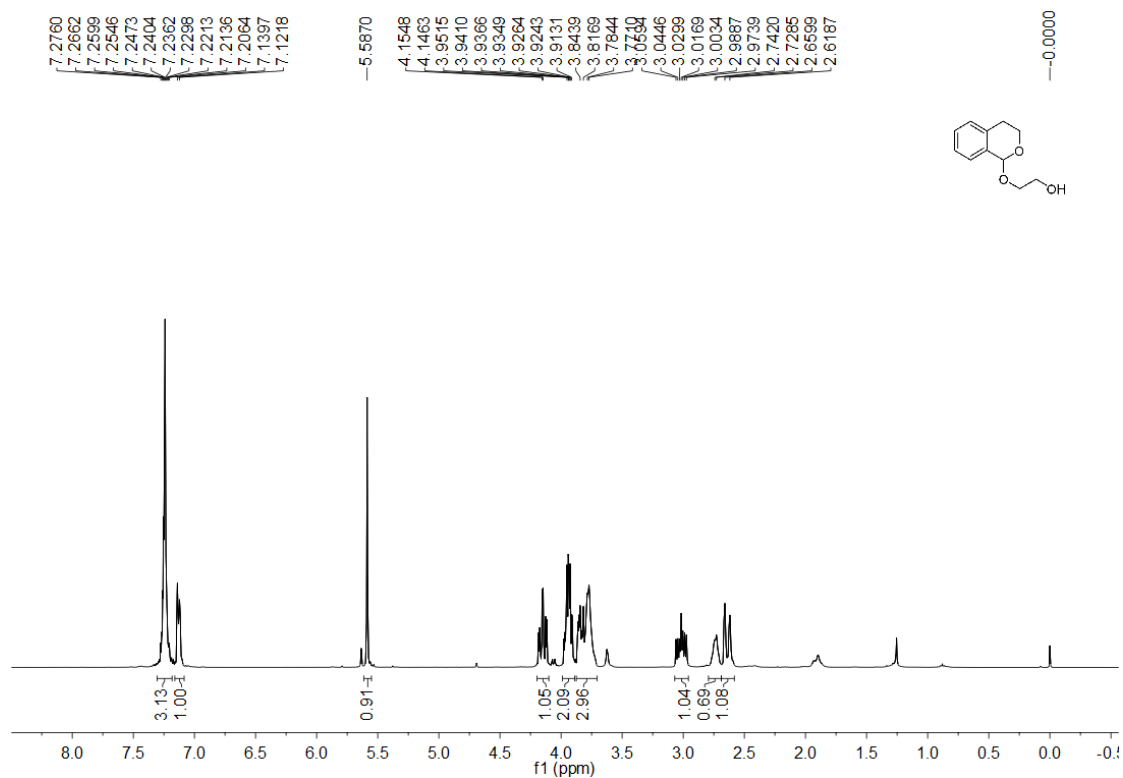




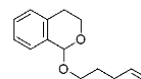
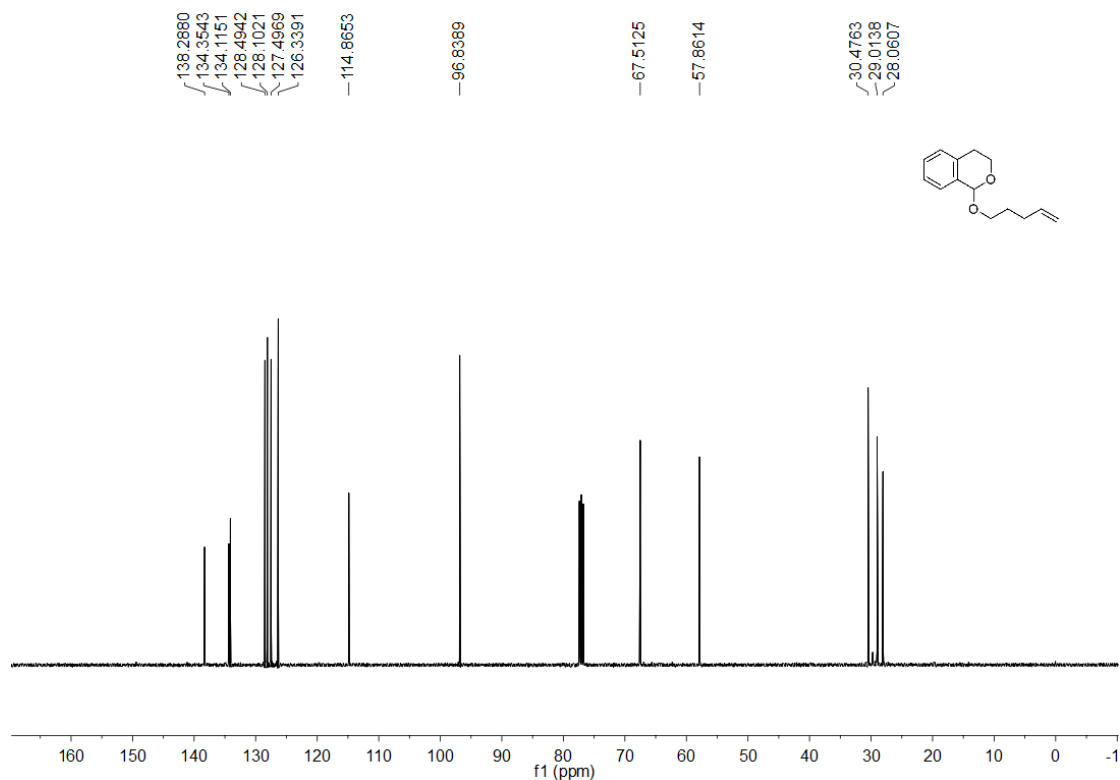
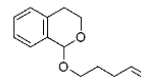
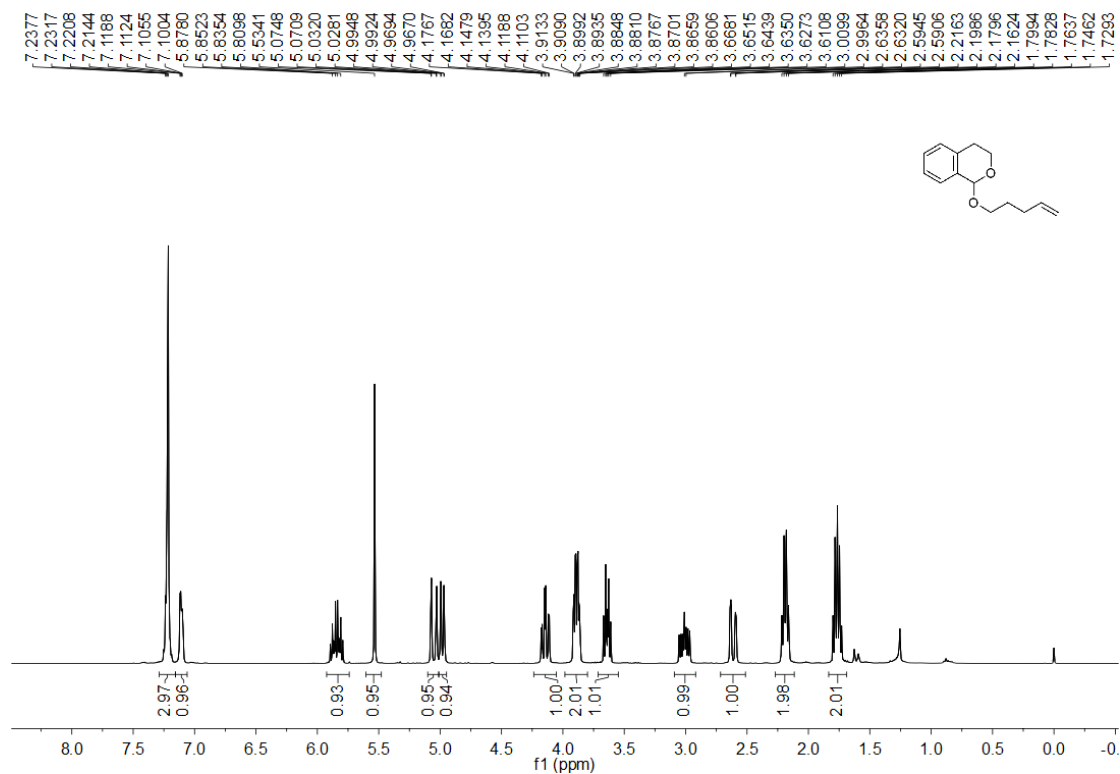
# 1-(2-Methoxyethoxy)isochromane (**3i**)



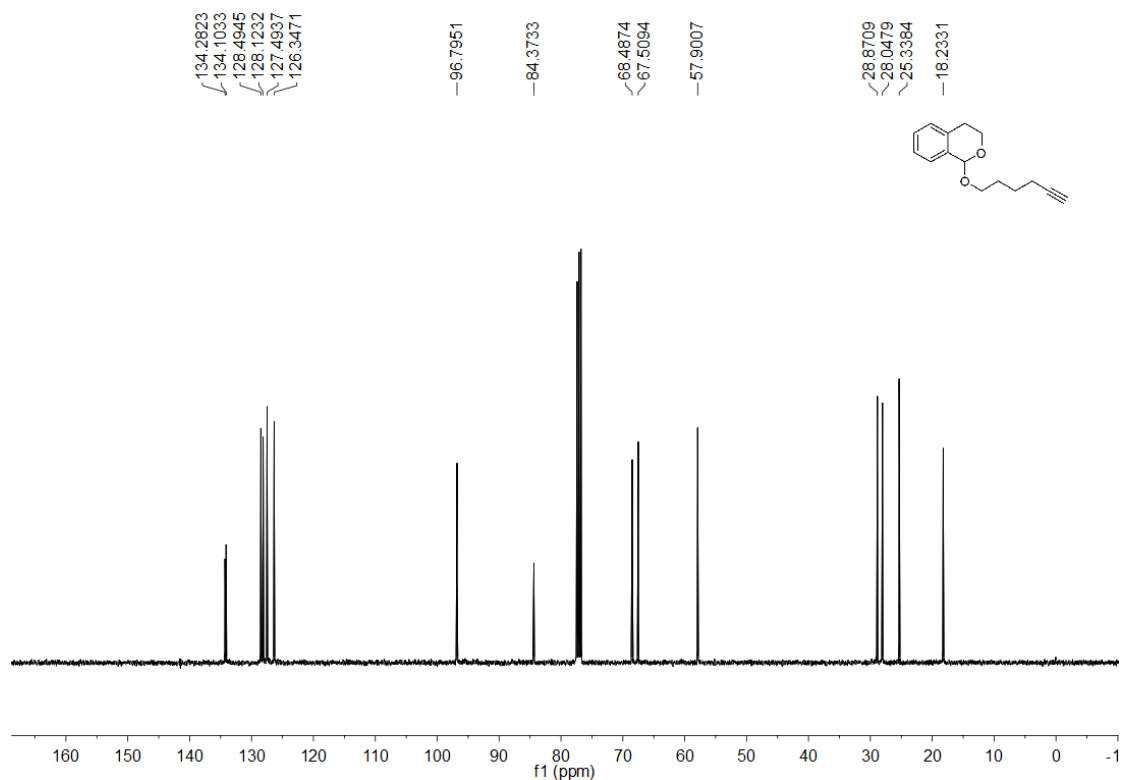
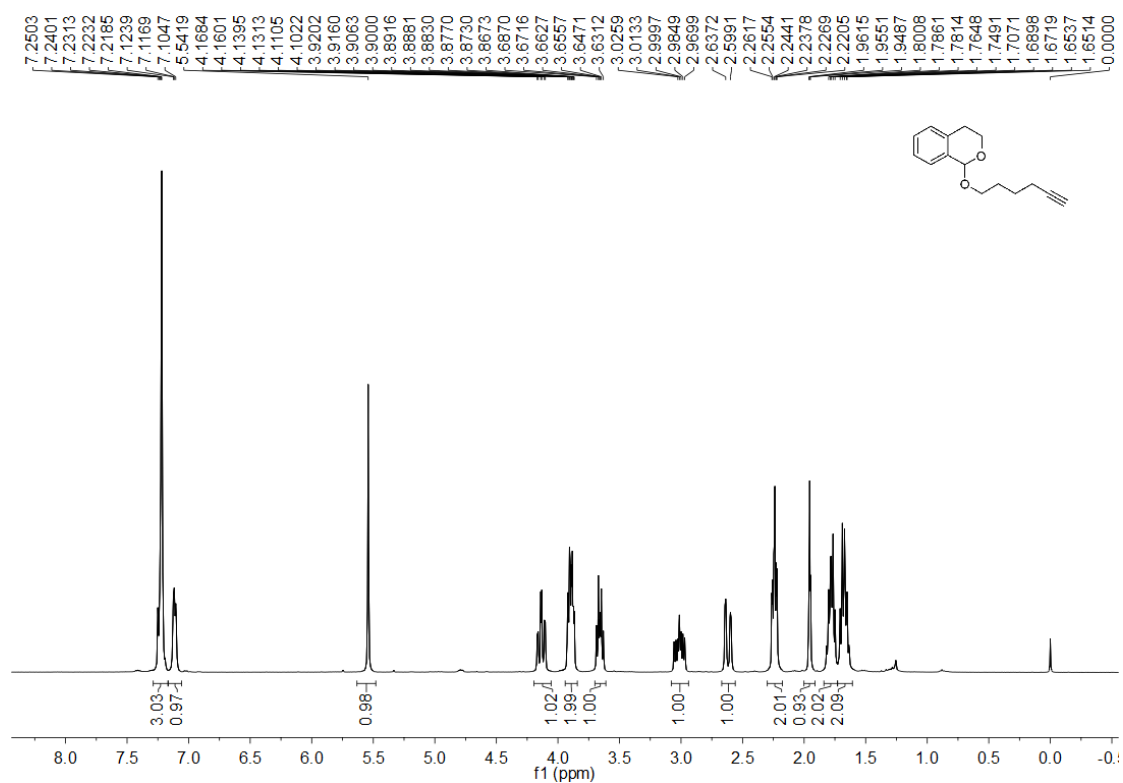
2-(Isochroman-1-yloxy)ethan-1-ol (**3j**)



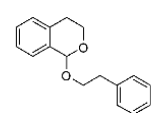
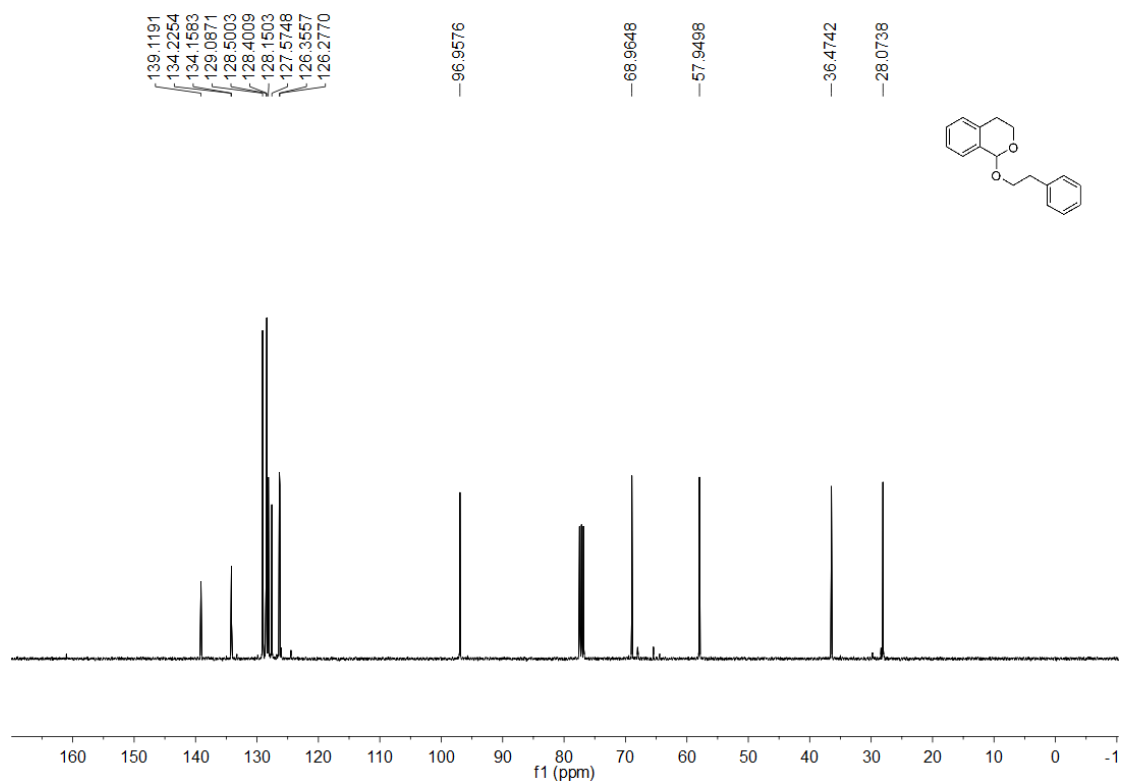
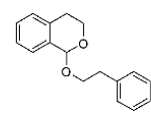
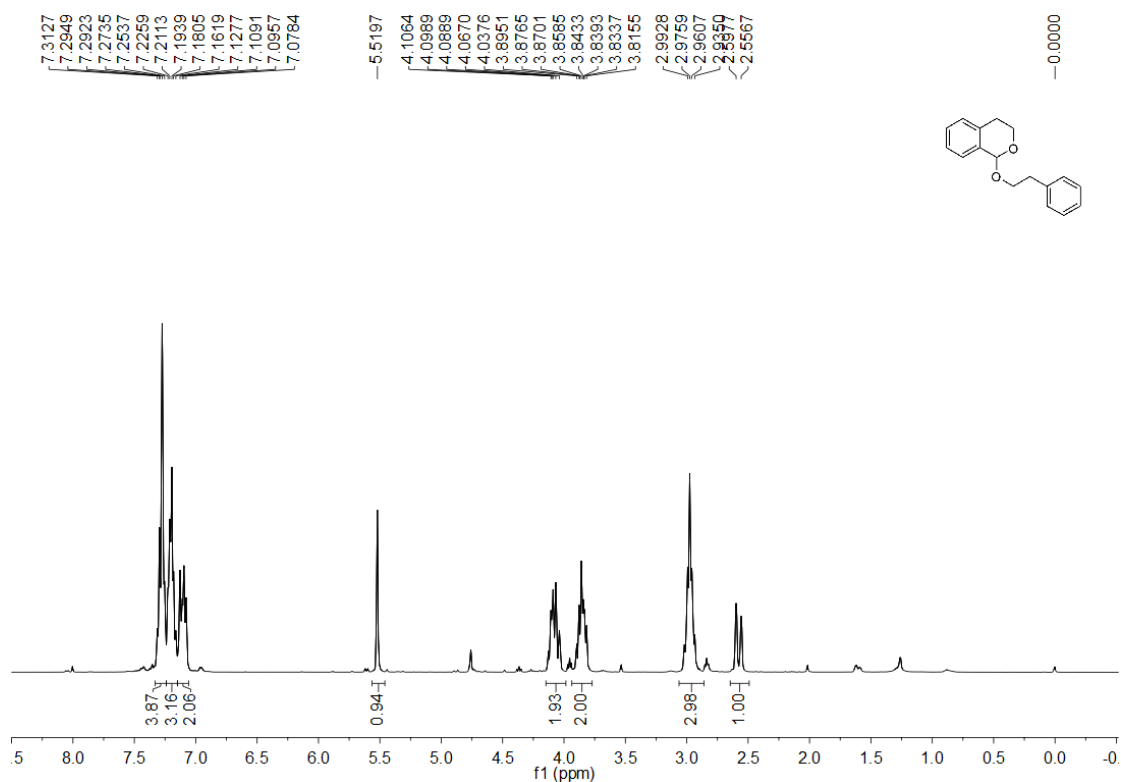
1-(Pent-4-en-1-yloxy)isochromane (**3k**)



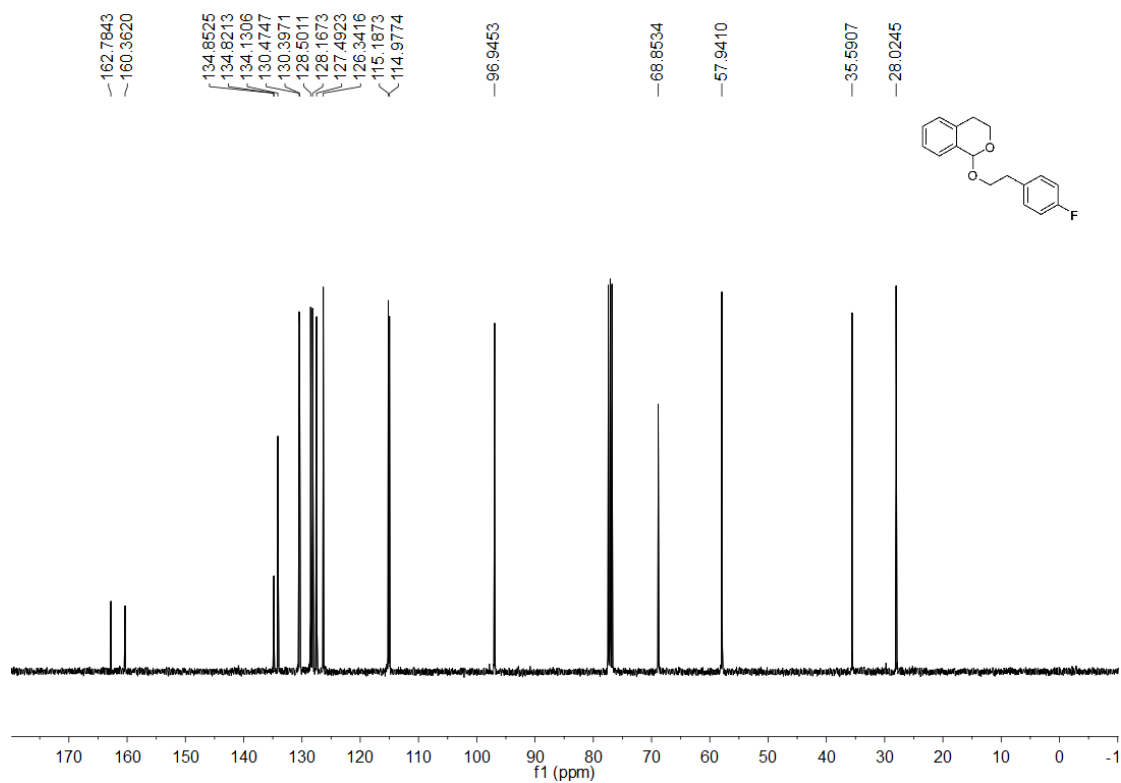
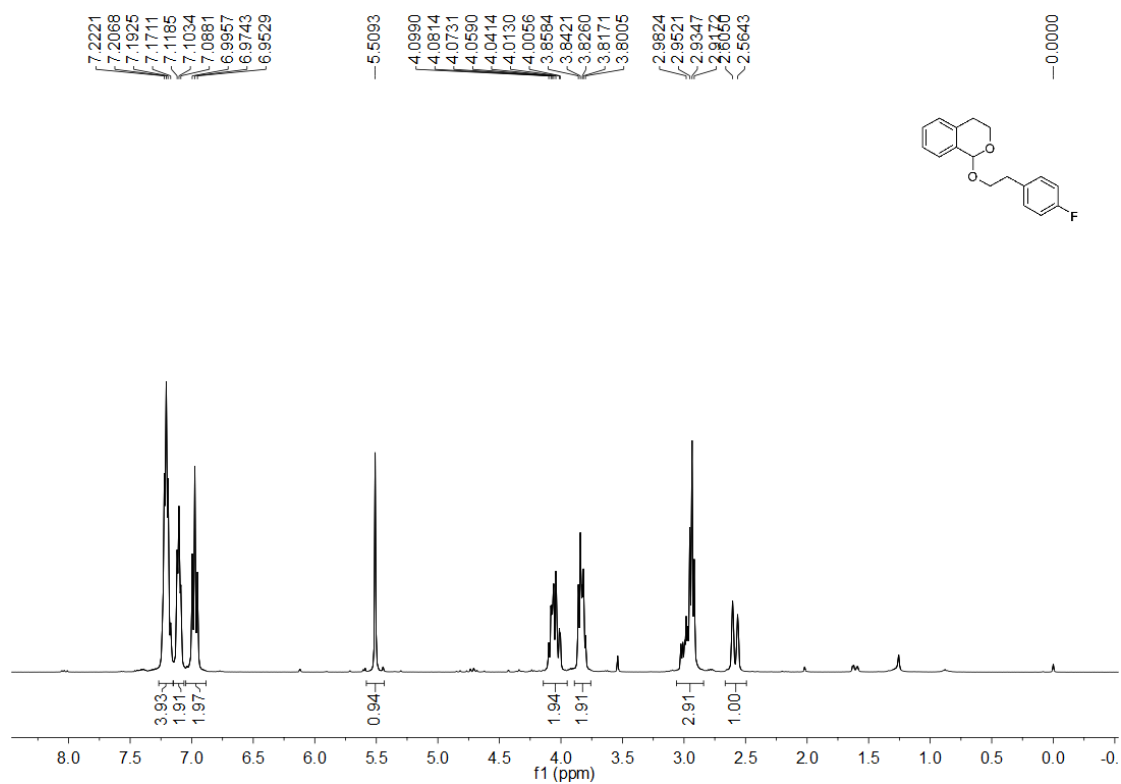
# 1-(Hex-5-yn-1-yloxy)isochromane (31)

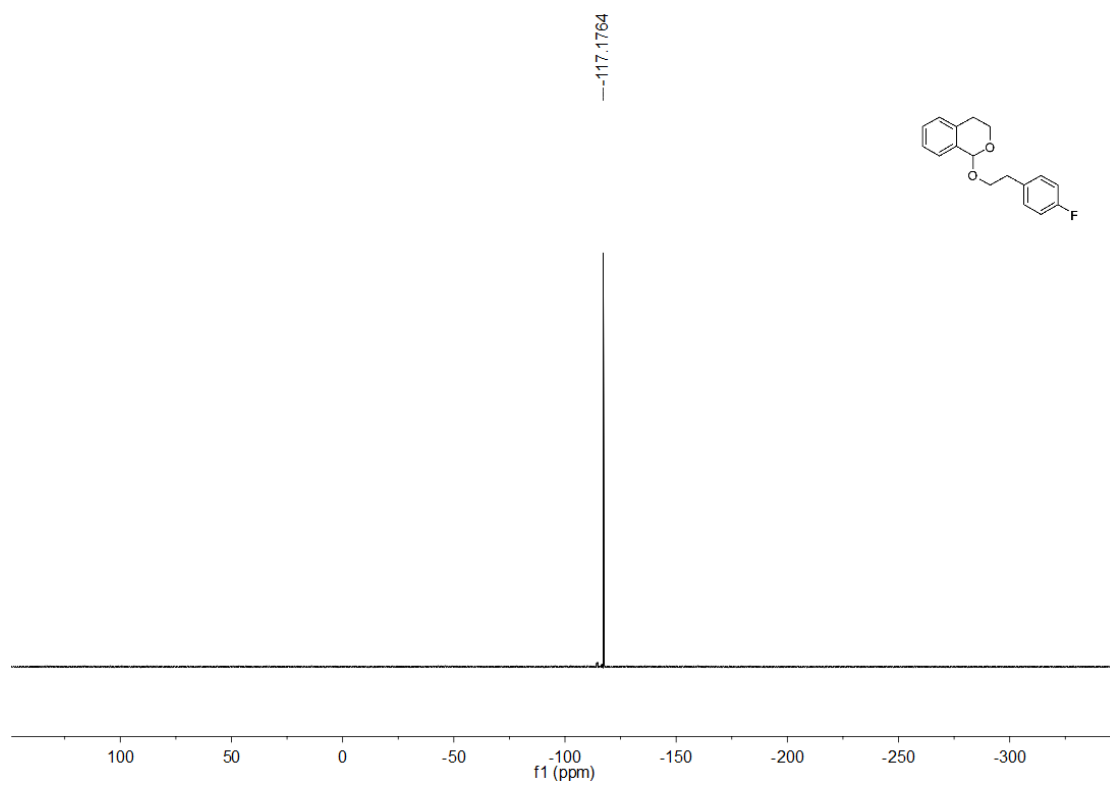


# 1-Phenethoxyisochromane (3m)

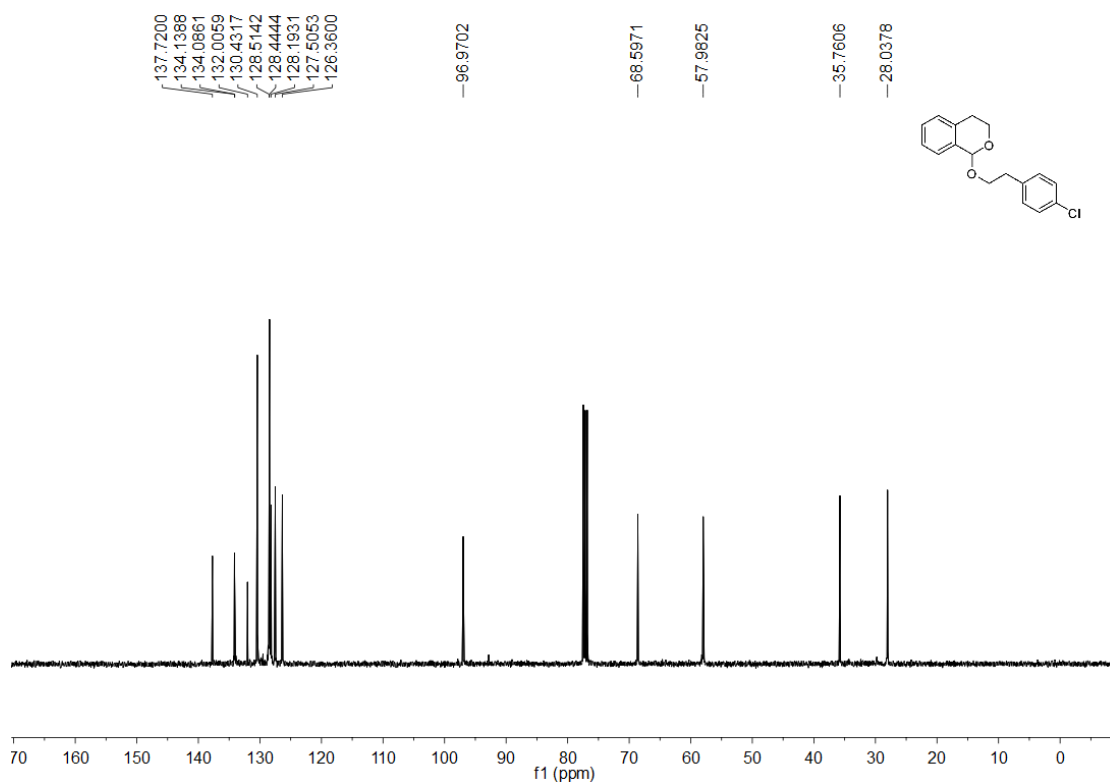
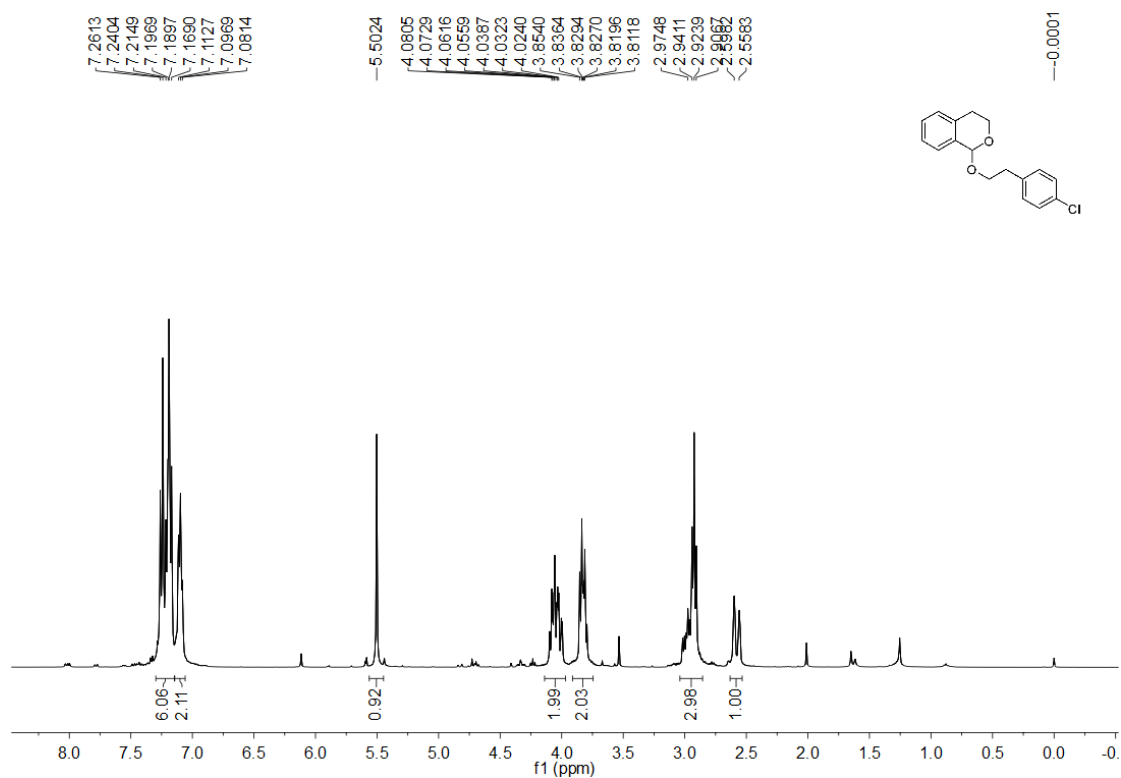


# 1-(4-Fluorophenoxy)isochromane (**3n**)



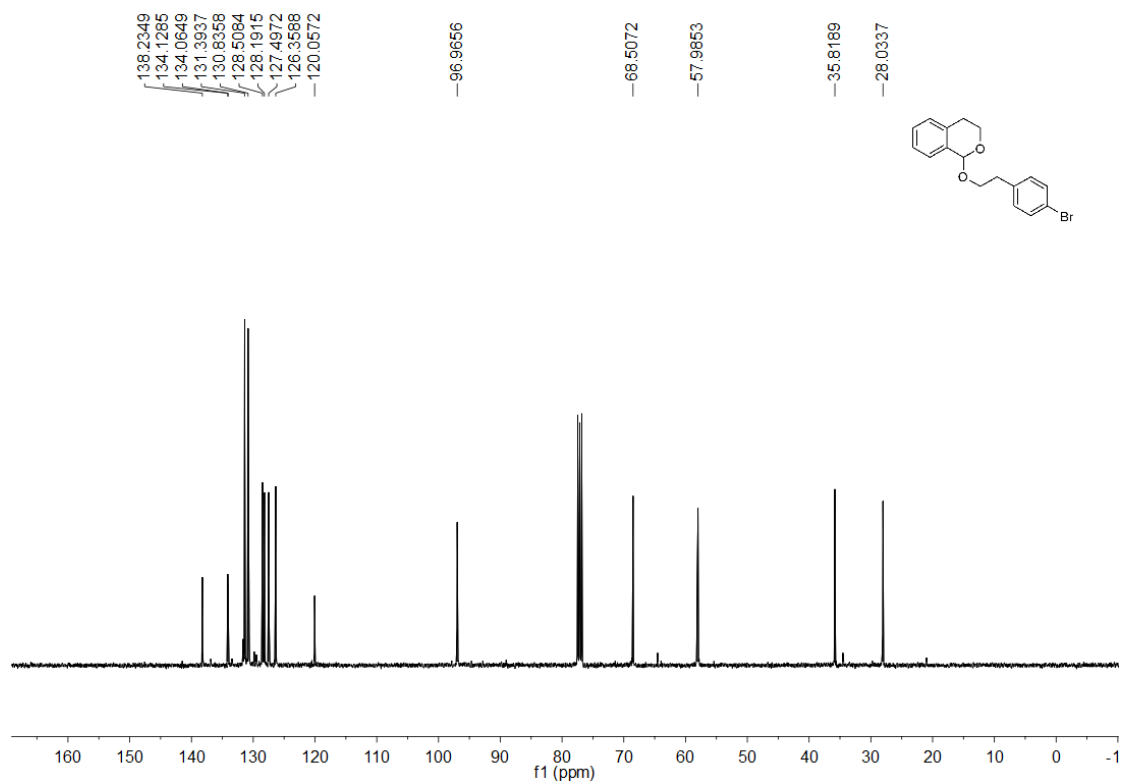
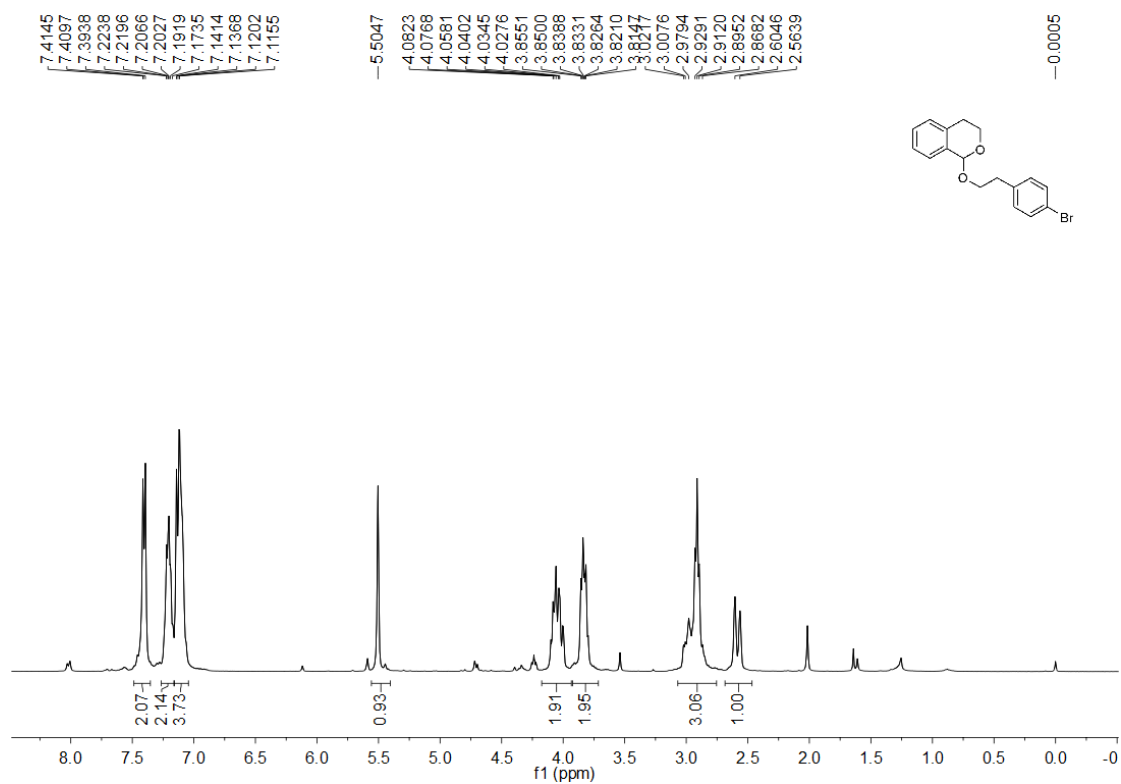


# 1-(4-Chlorophenoxy)isochromane (30)

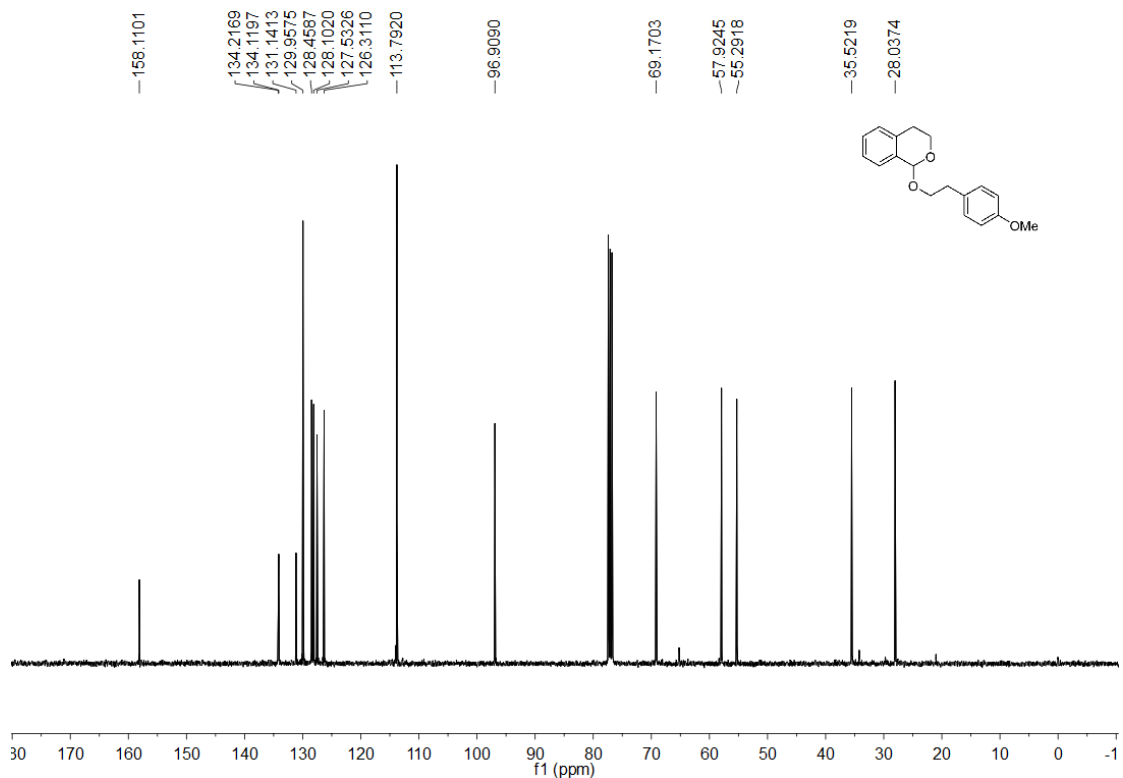
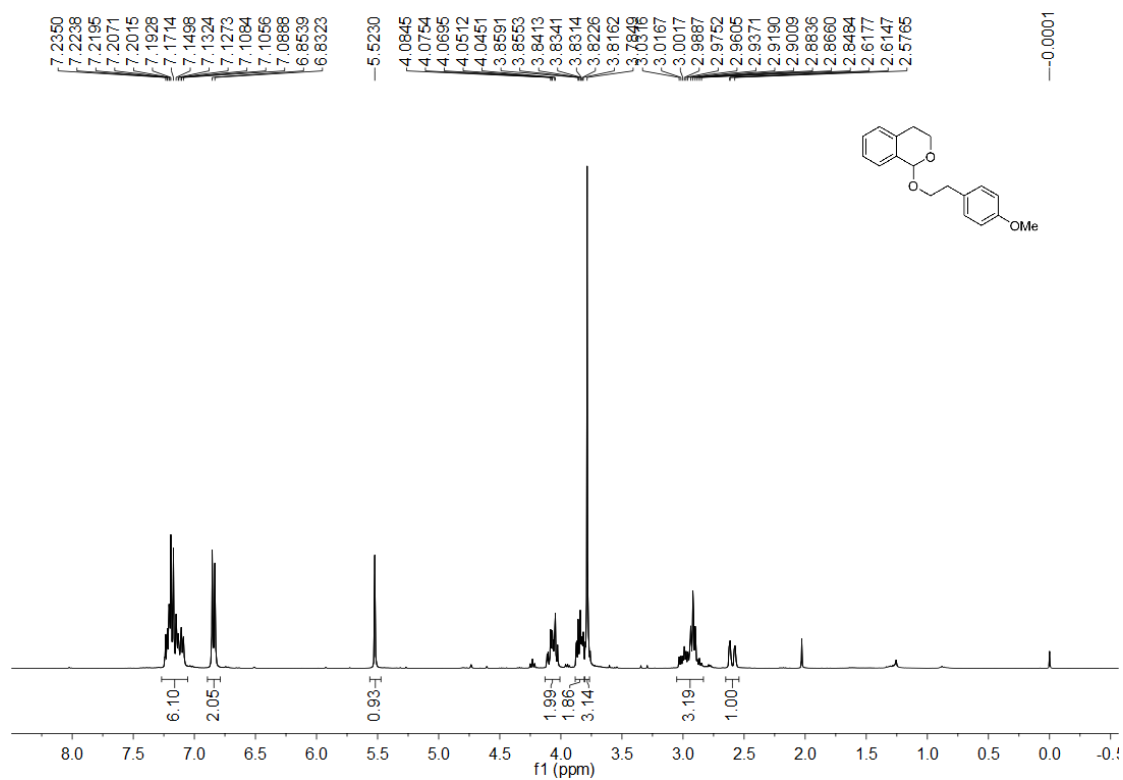




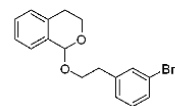
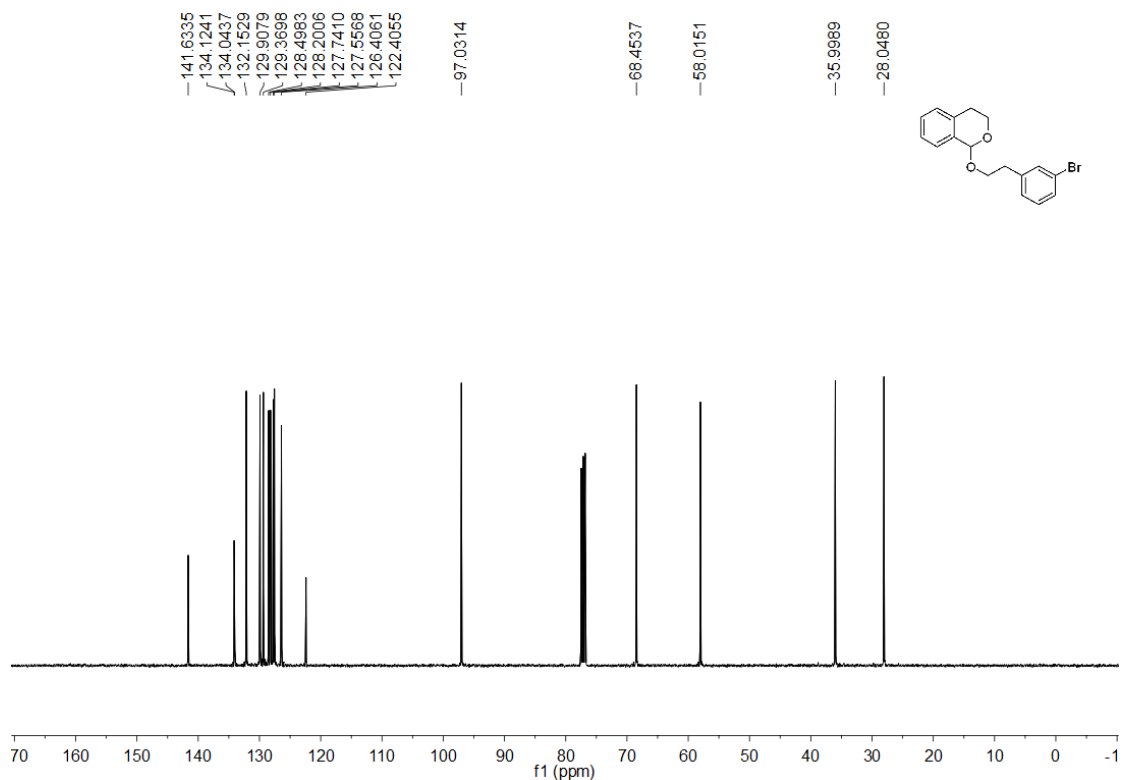
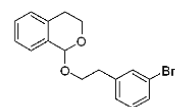
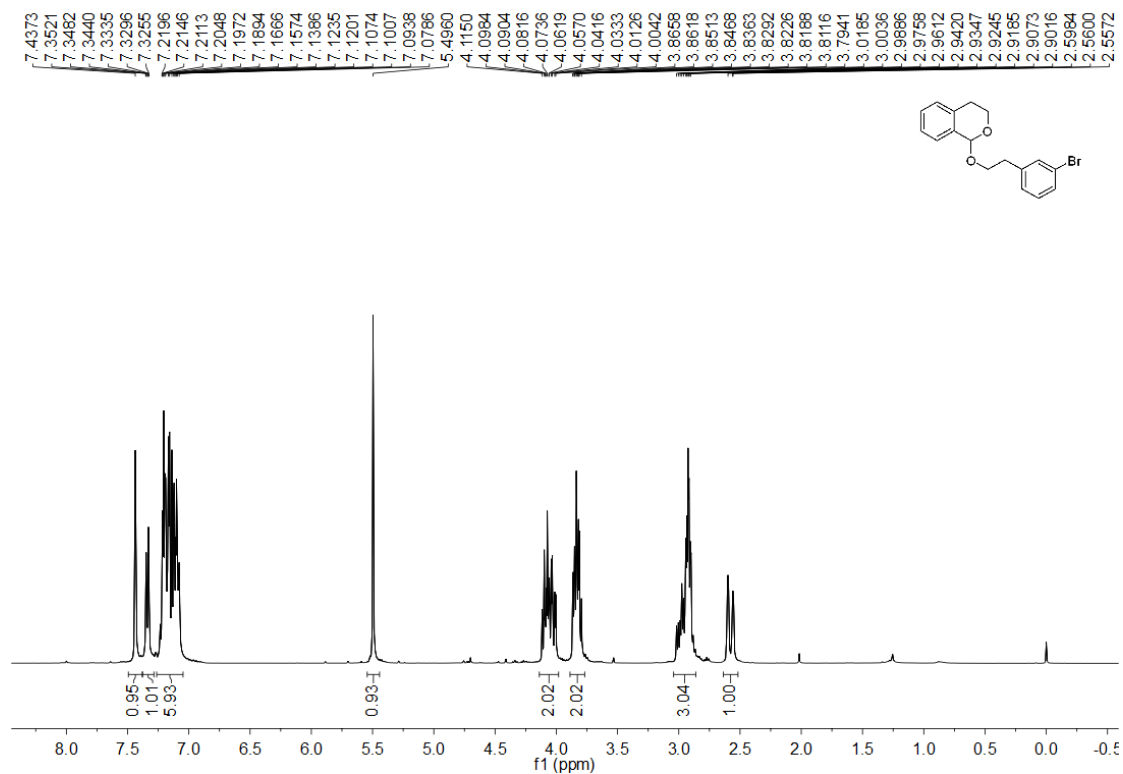
# 1-(4-Bromophenoxy)isochromane (**3p**)



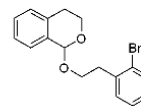
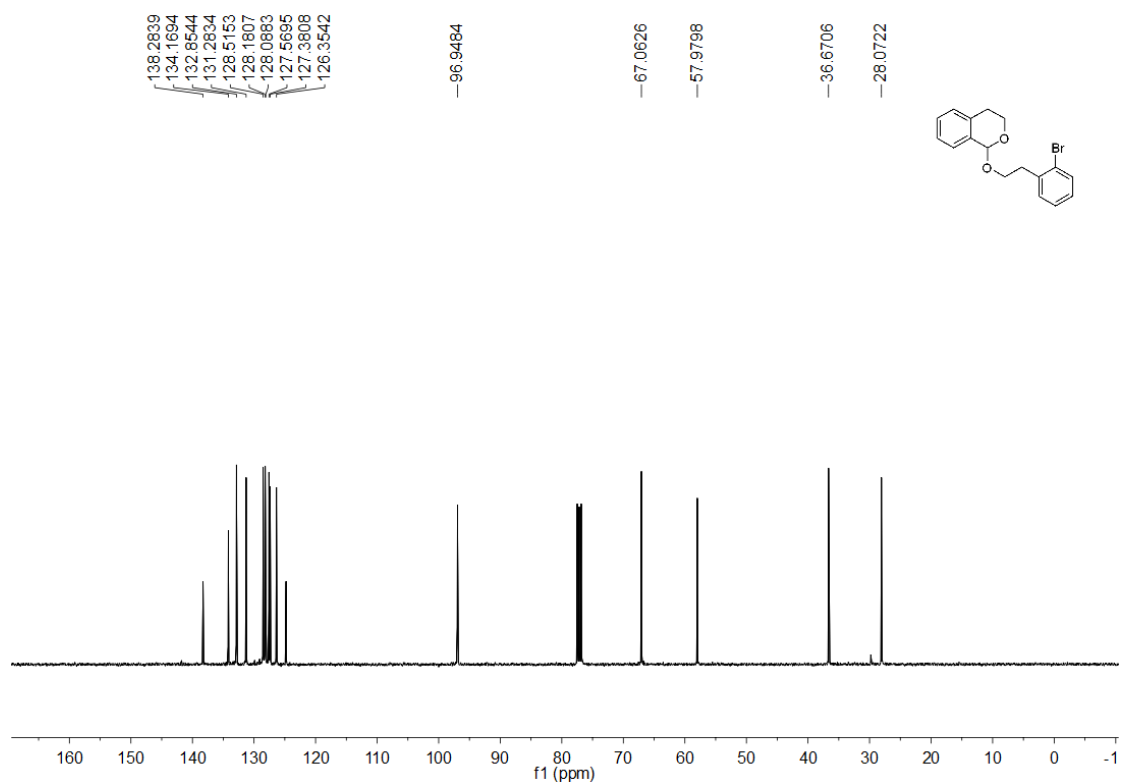
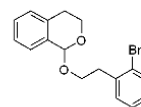
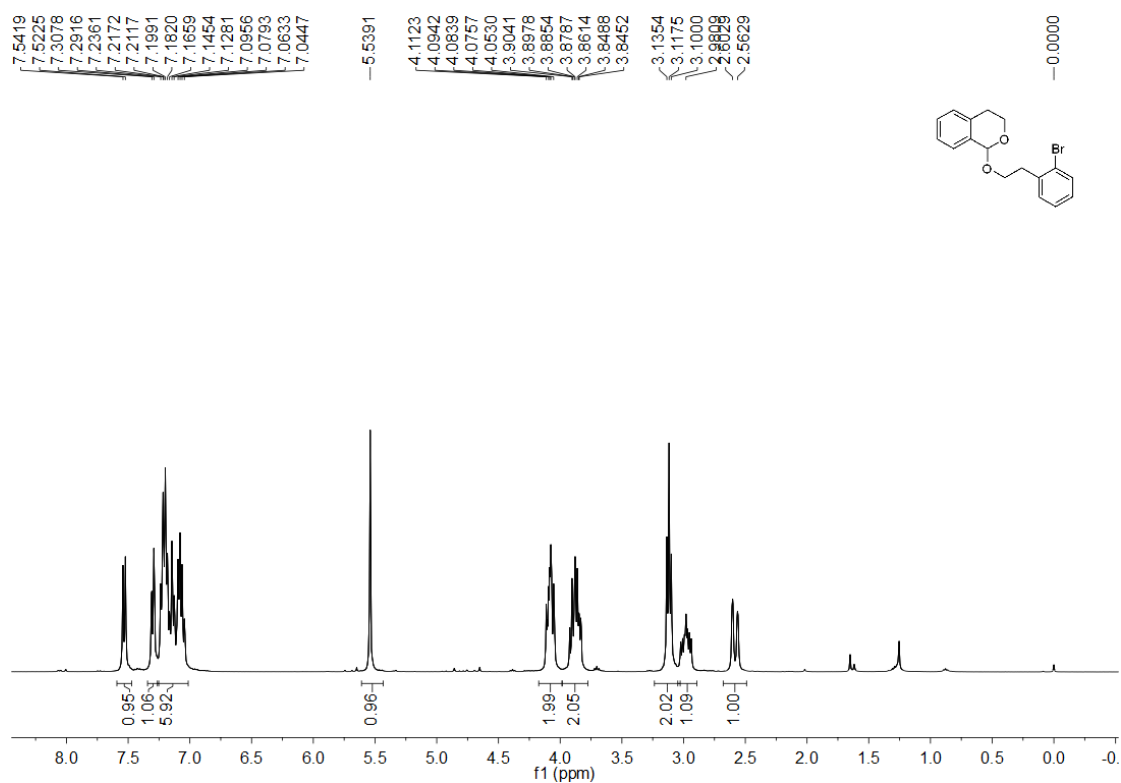
# 1-(4-Methoxyphenethoxy)isochromane (3q)



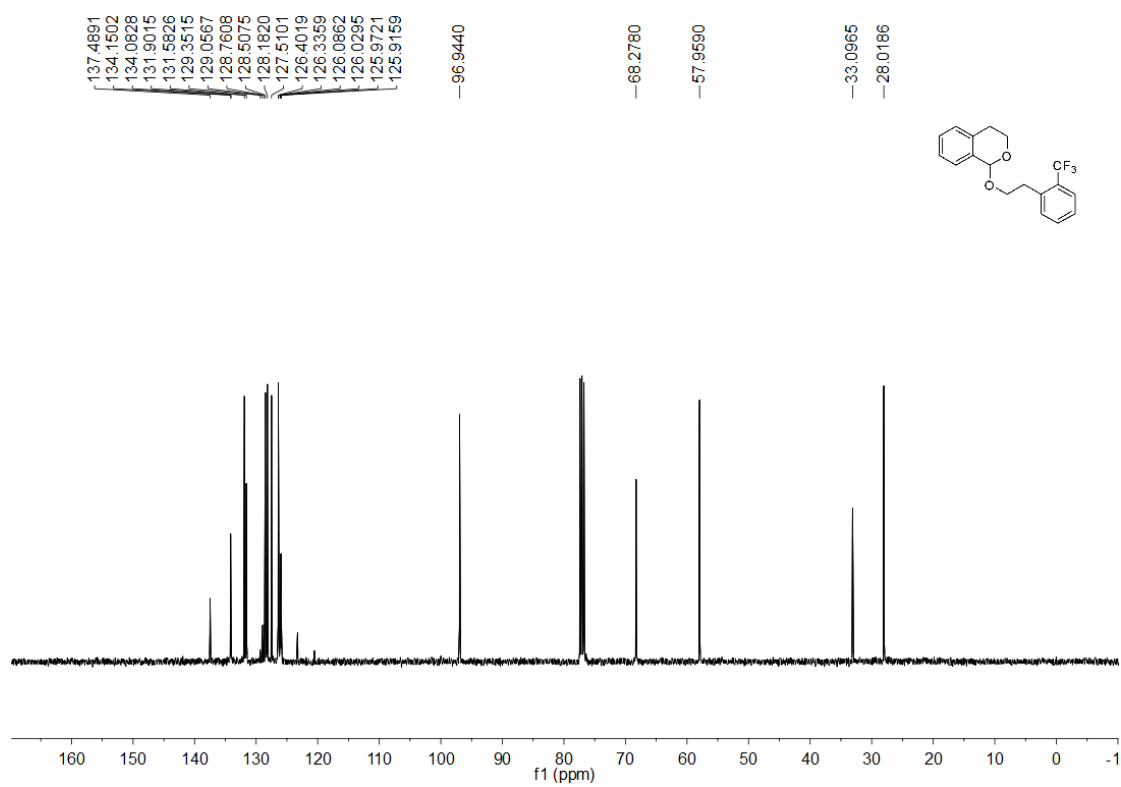
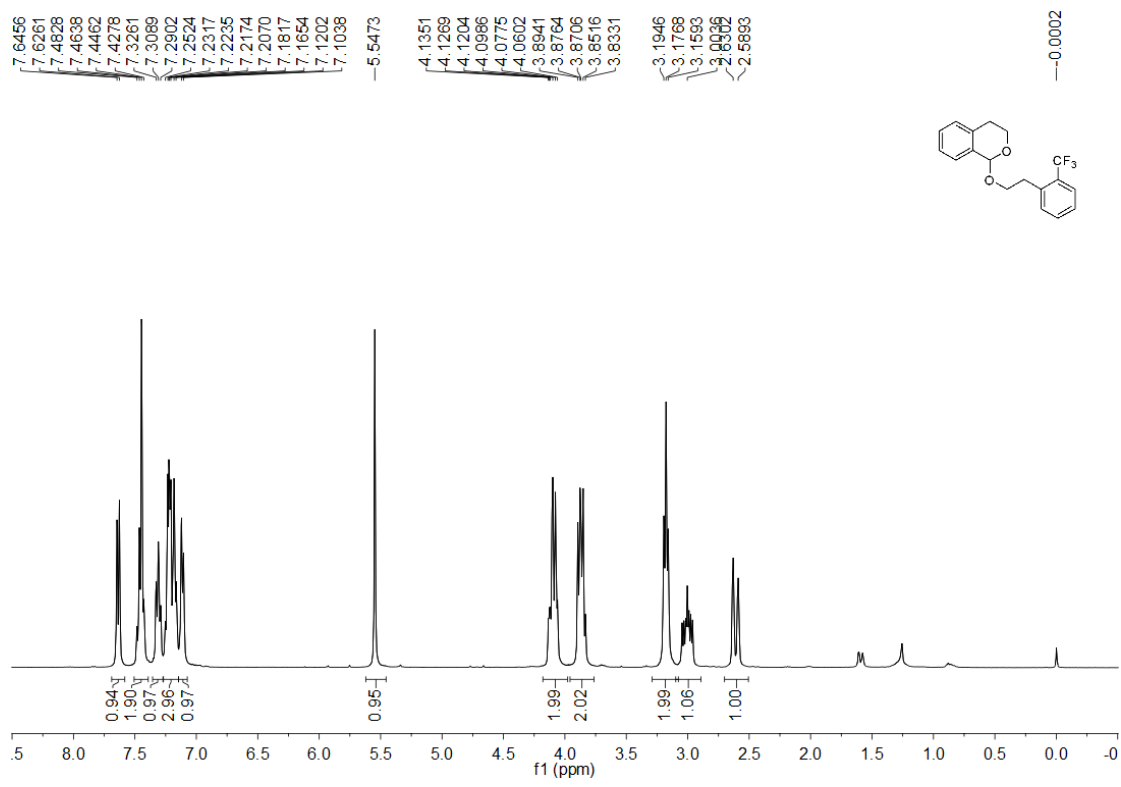
# 1-(3-Bromophenoxy)isochromane (**3r**)

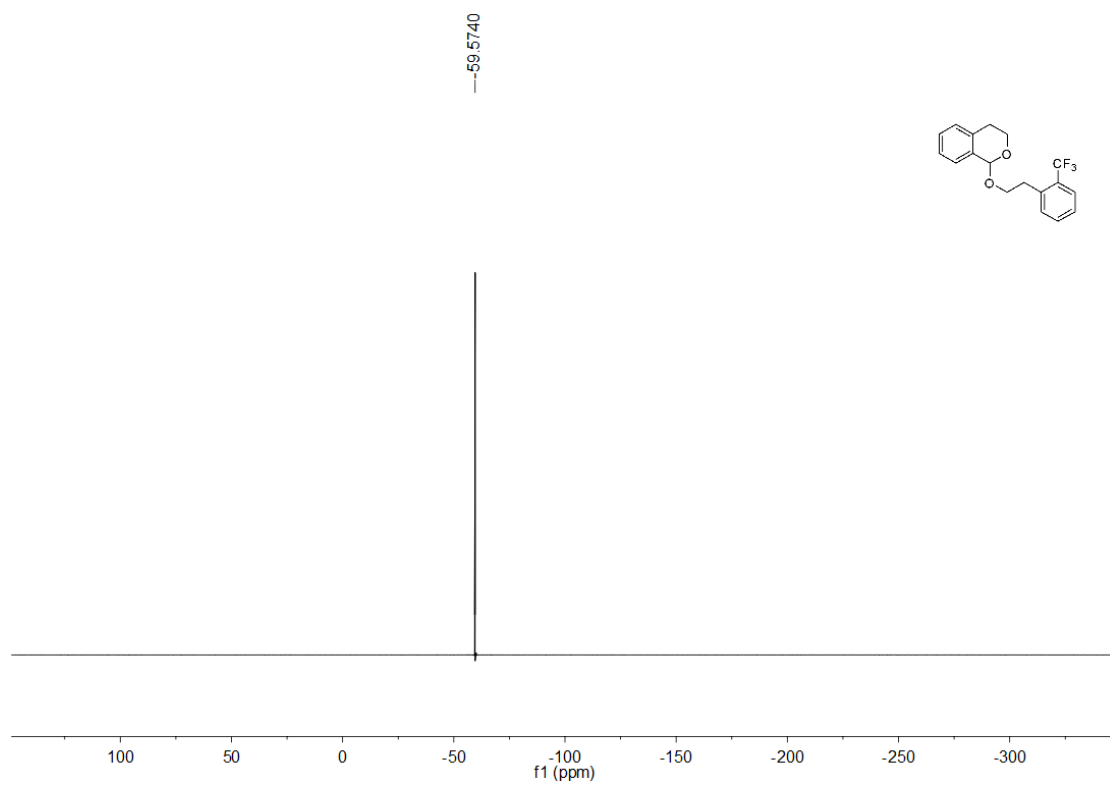


# 1-(2-Bromophenoxy)isochromane (**3s**)

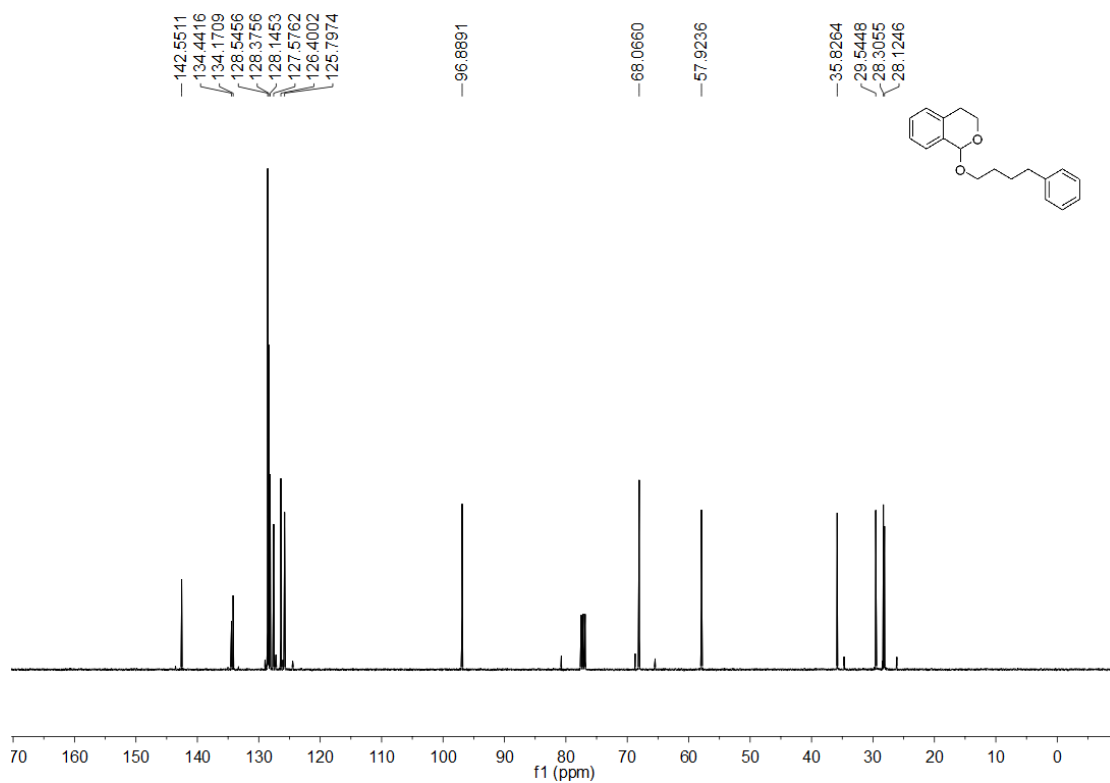
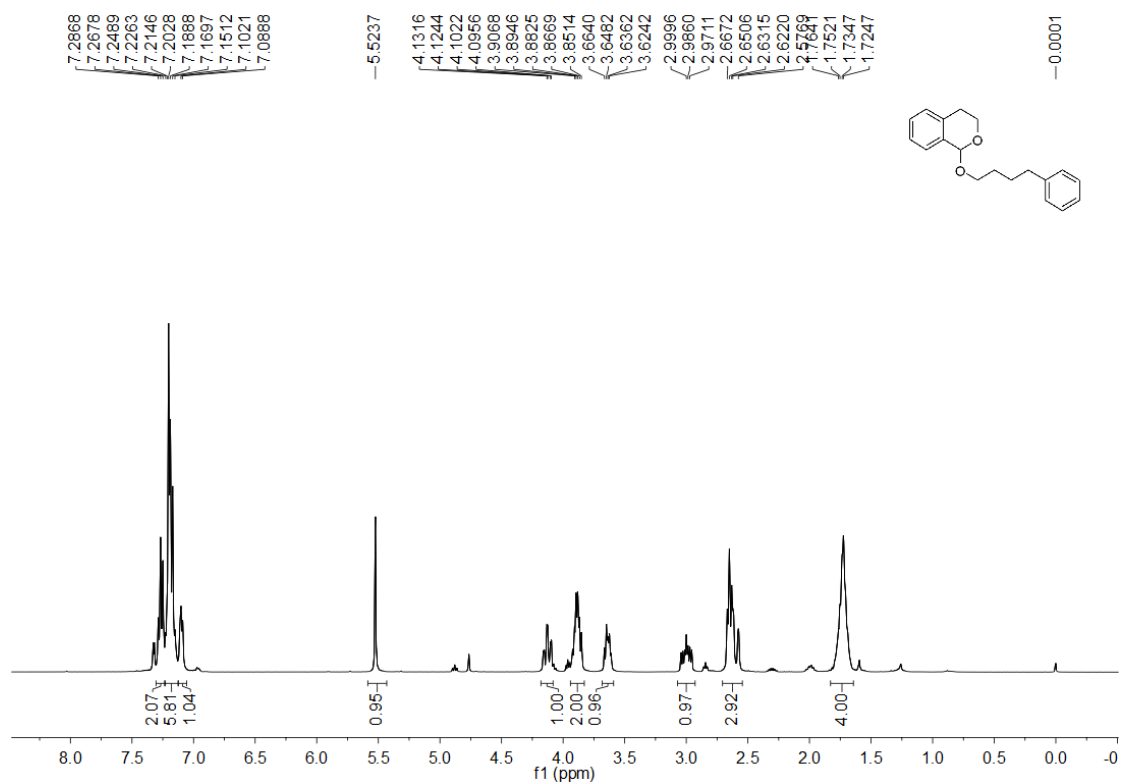


# 1-(2-(Trifluoromethyl)phenoxy)isochromane (3t)

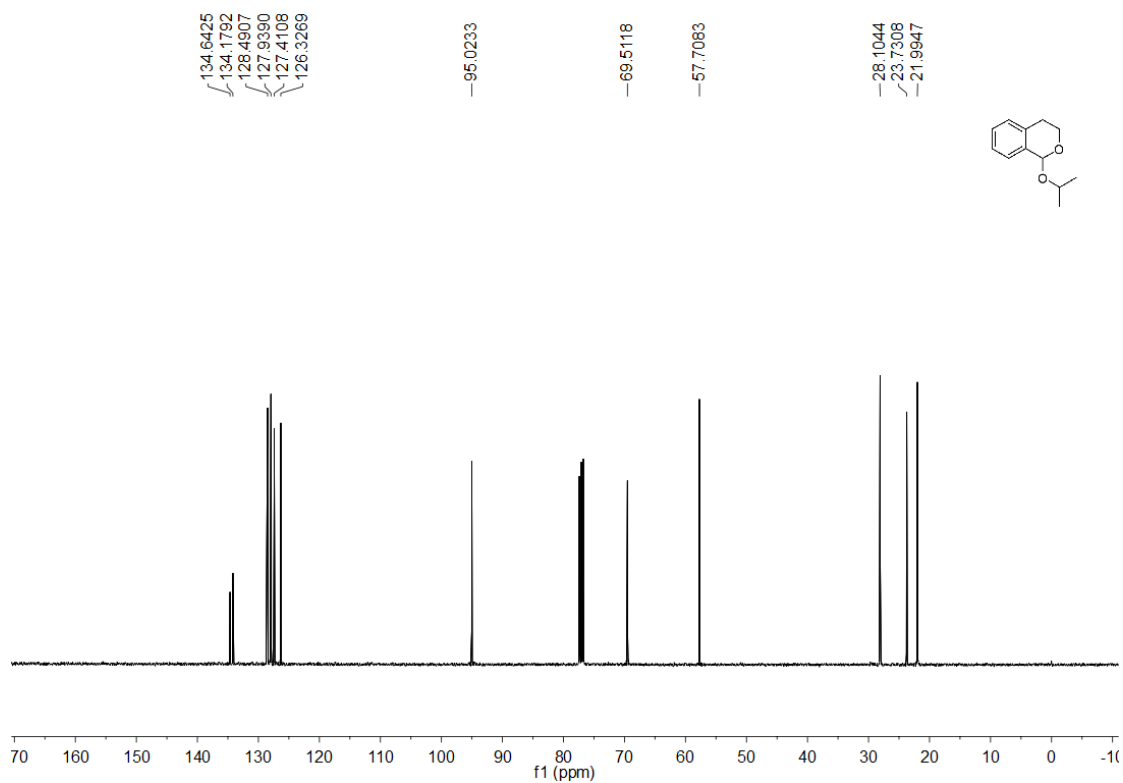
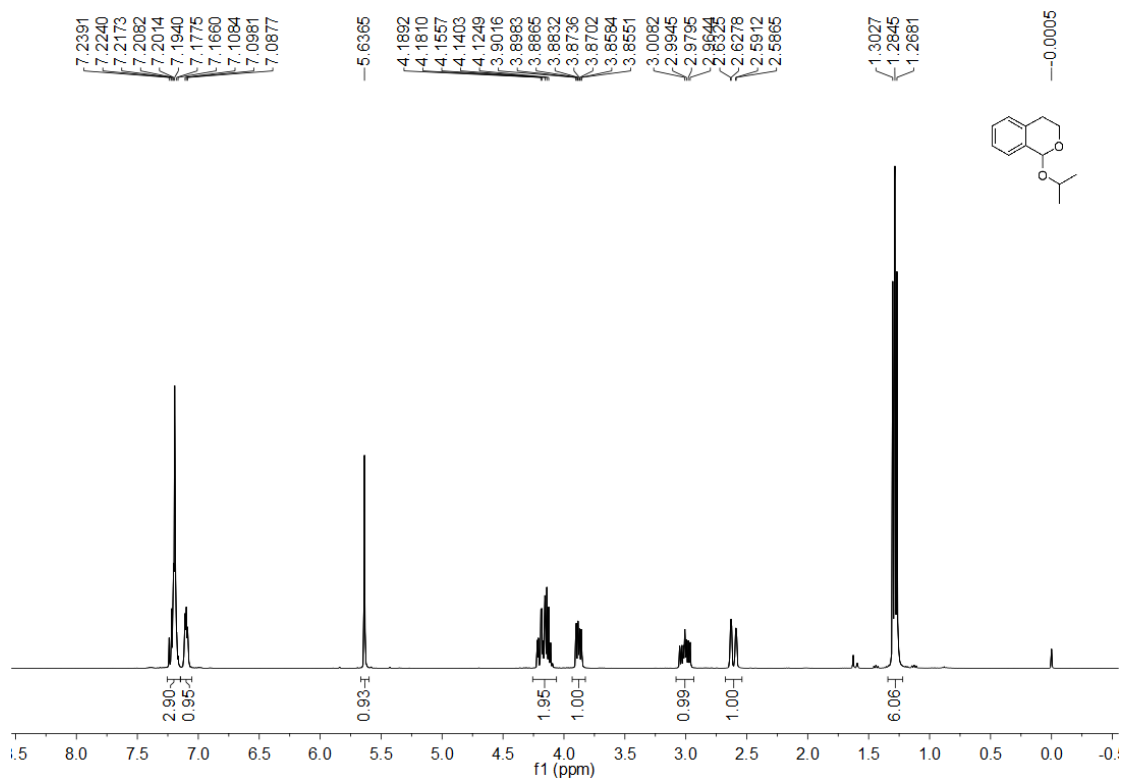




# 1-(4-Phenylbutoxy)isochromane (**3u**)

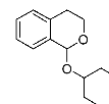
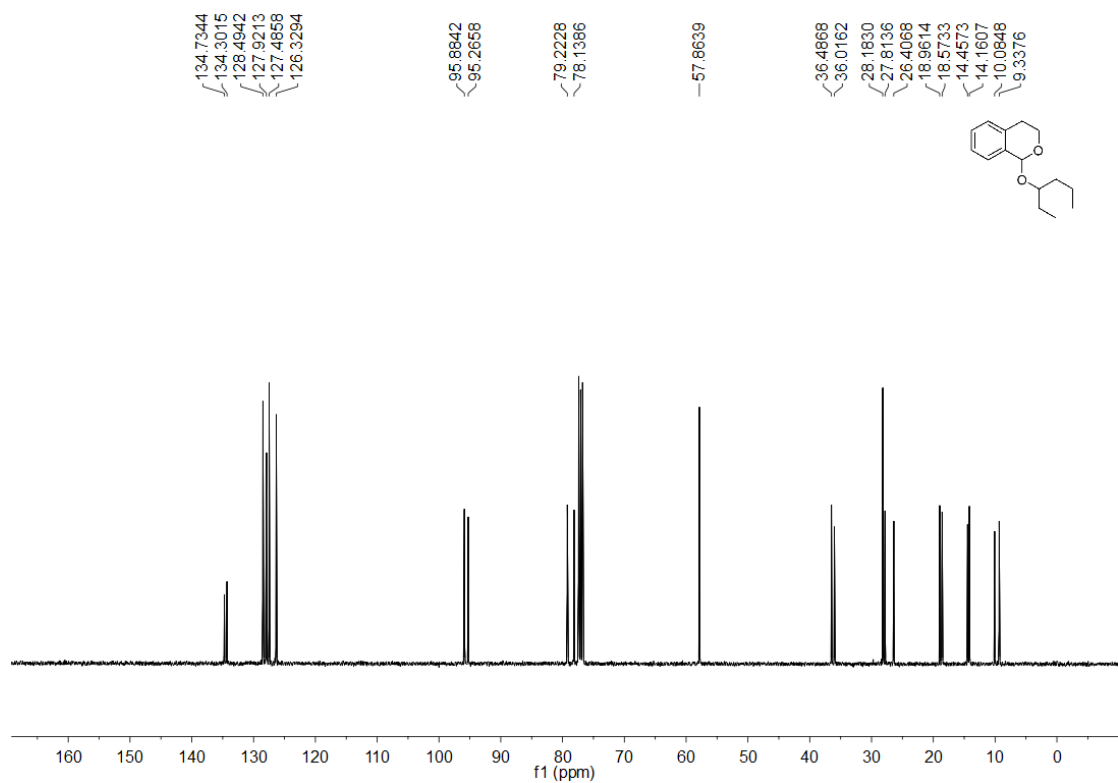
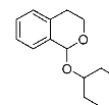
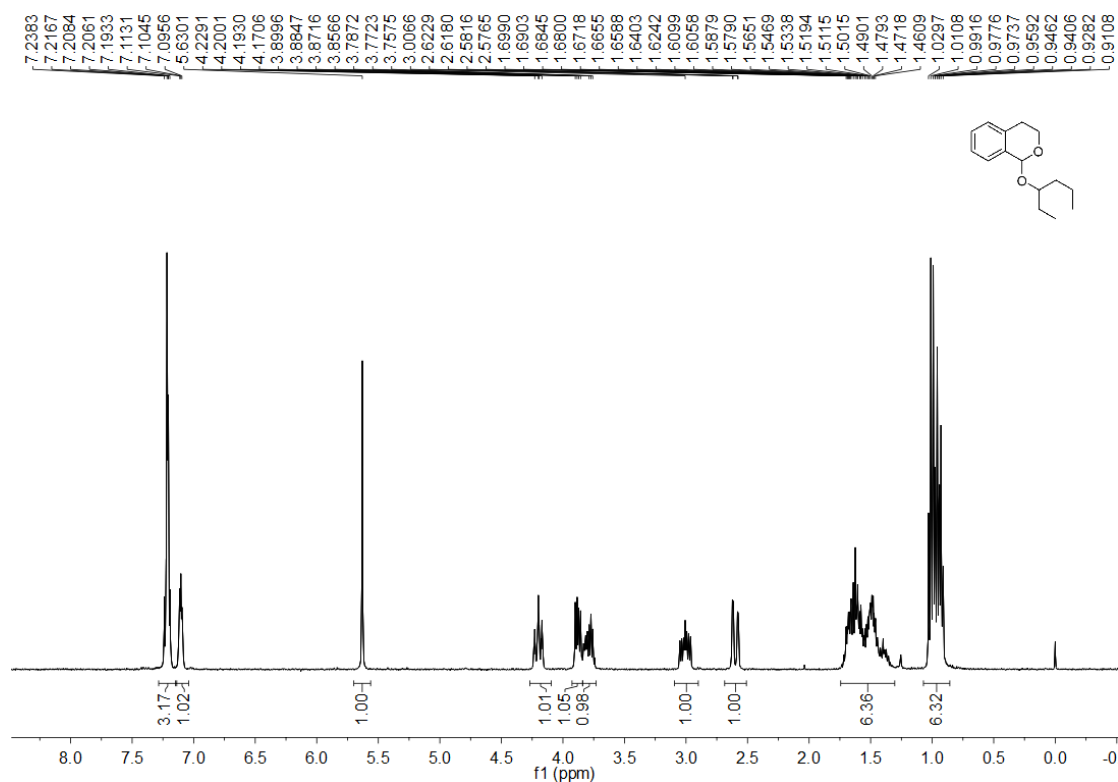


# 1-Isopropoxyisochromane (**3v**)

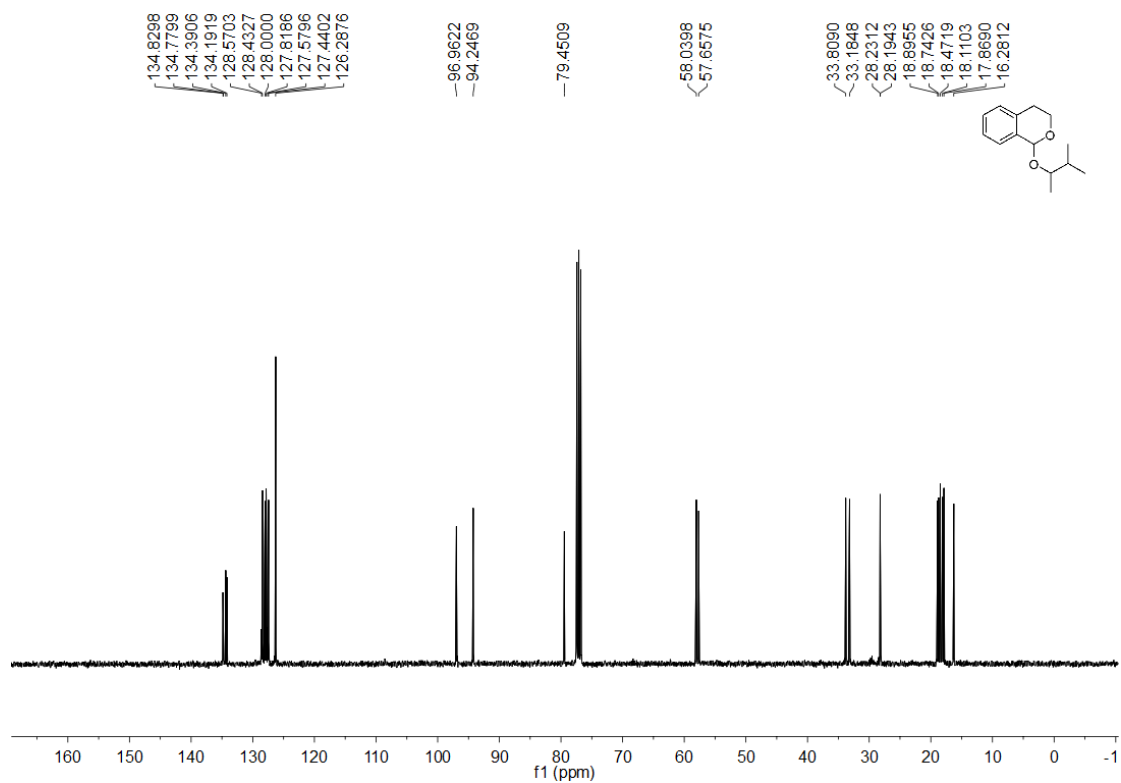
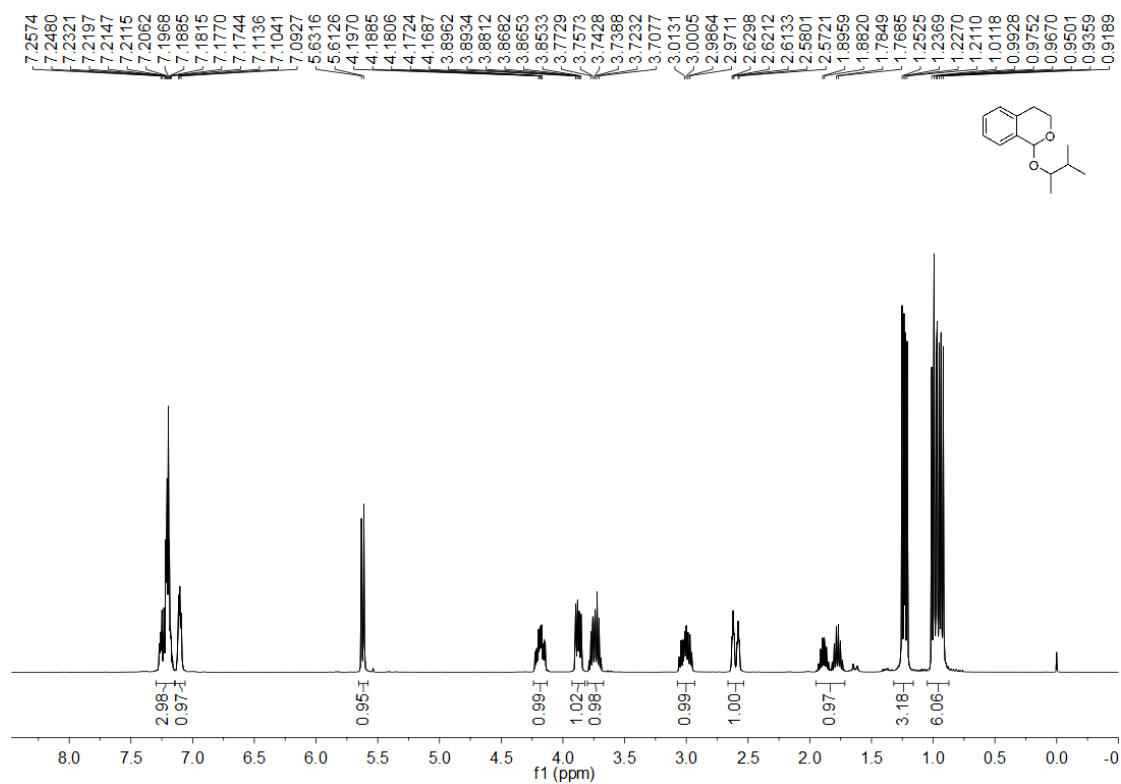




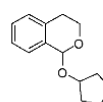
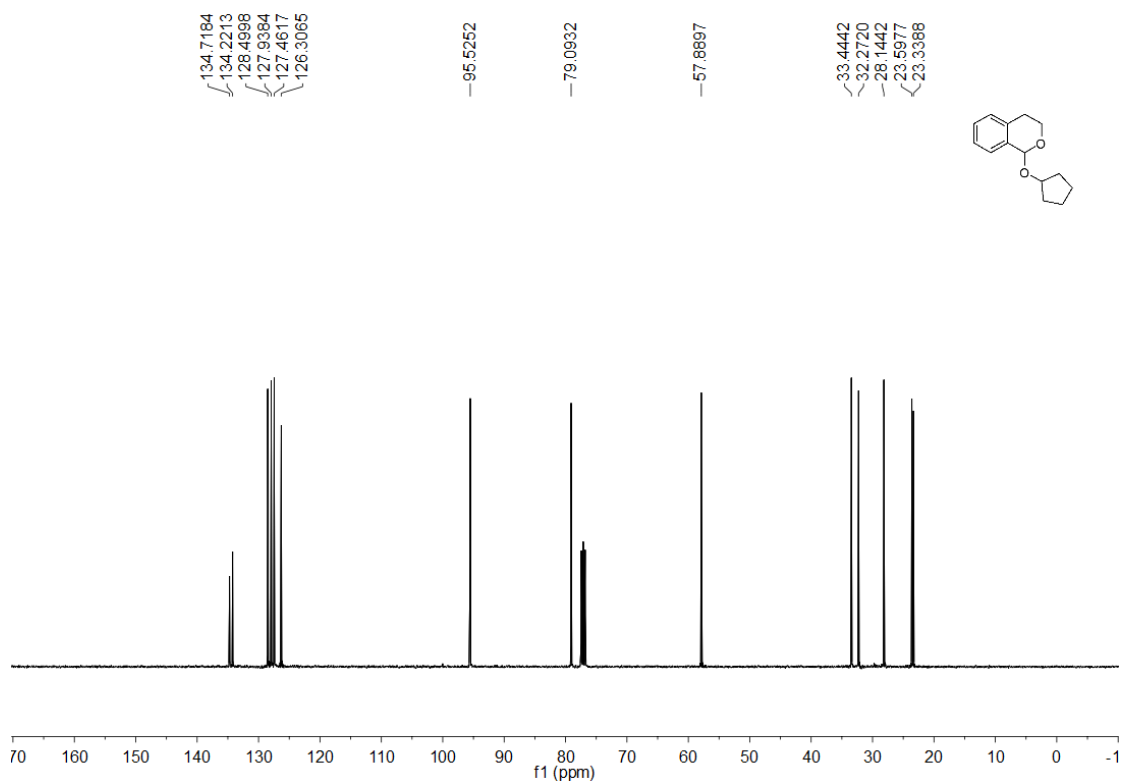
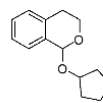
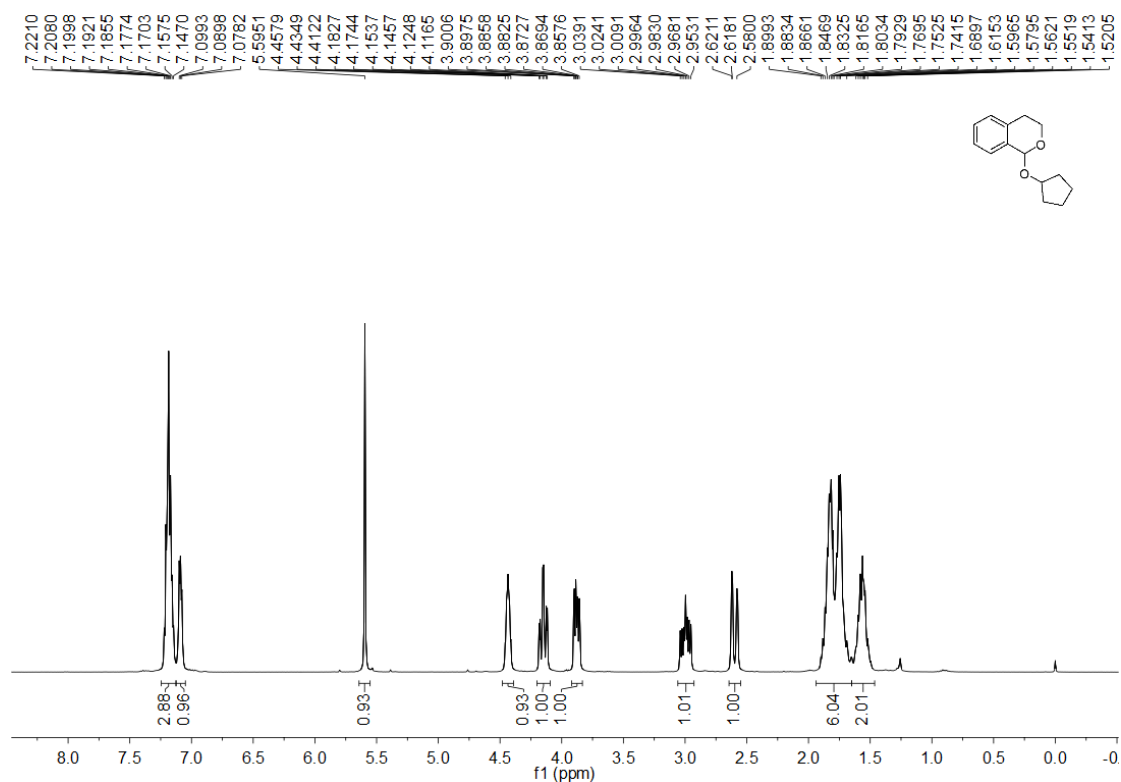
# 1-(Hexan-3-yloxy)isochromane (**3w**)



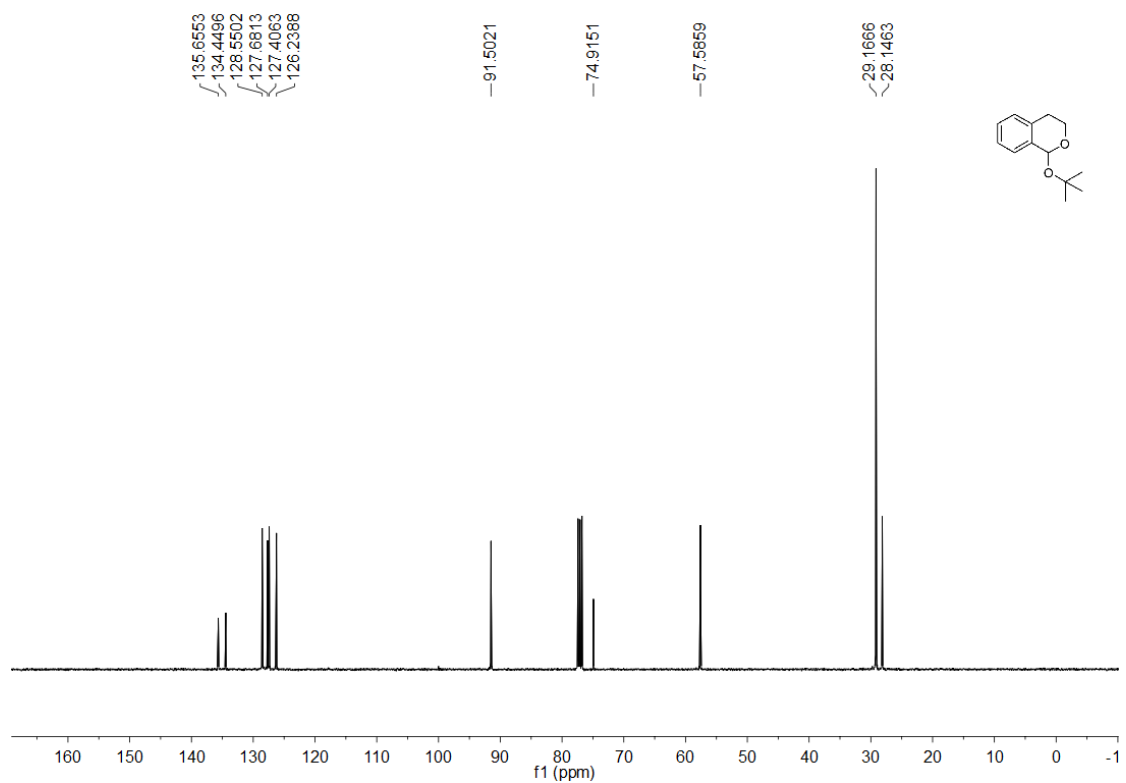
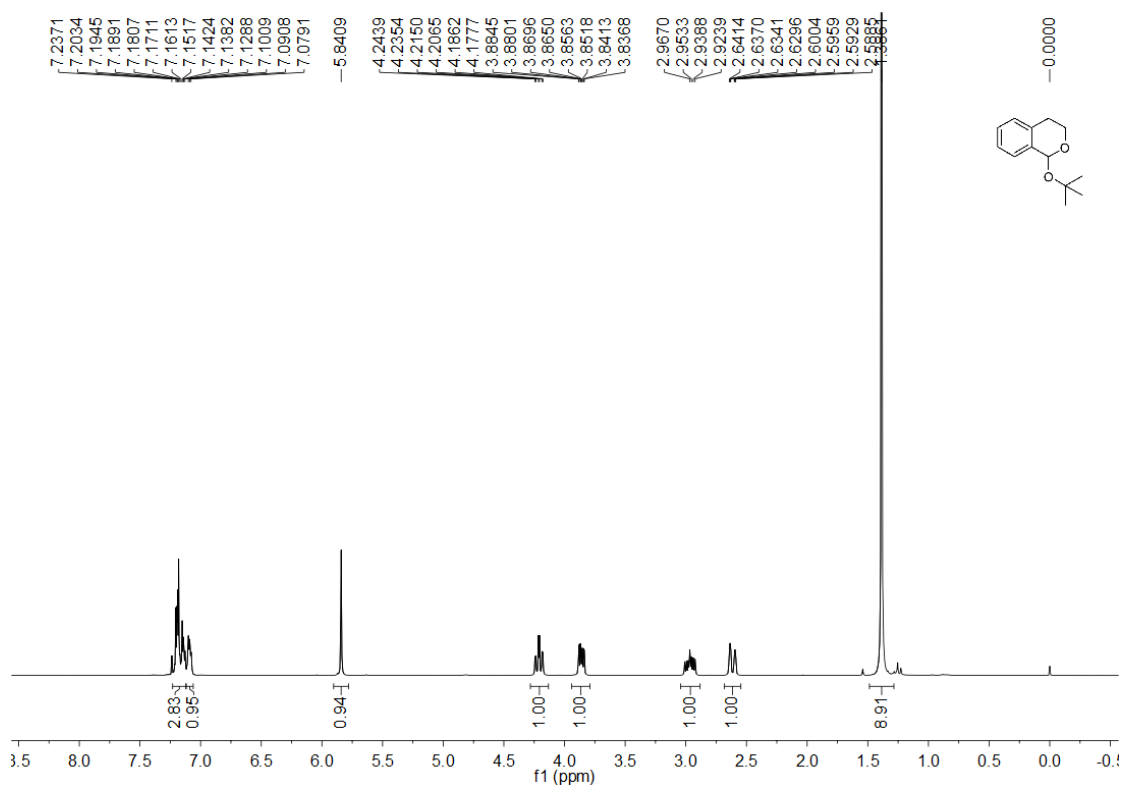
# 1-((3-Methylbutan-2-yl)oxy)isochromane (3x)



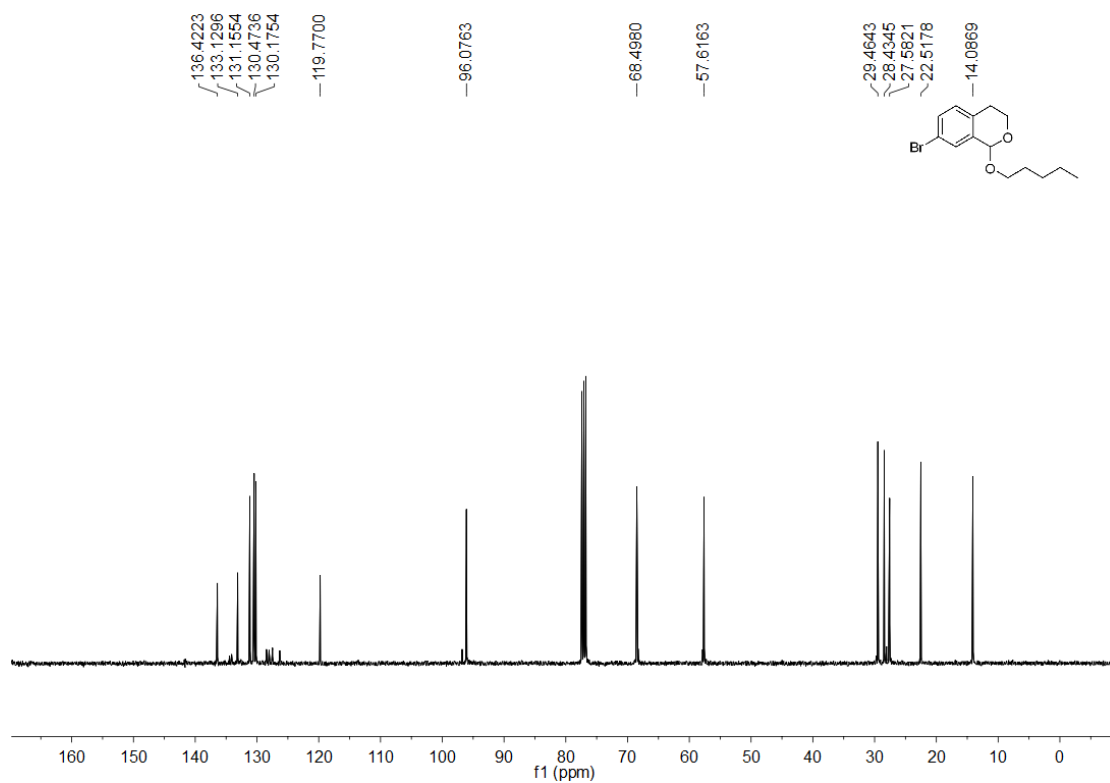
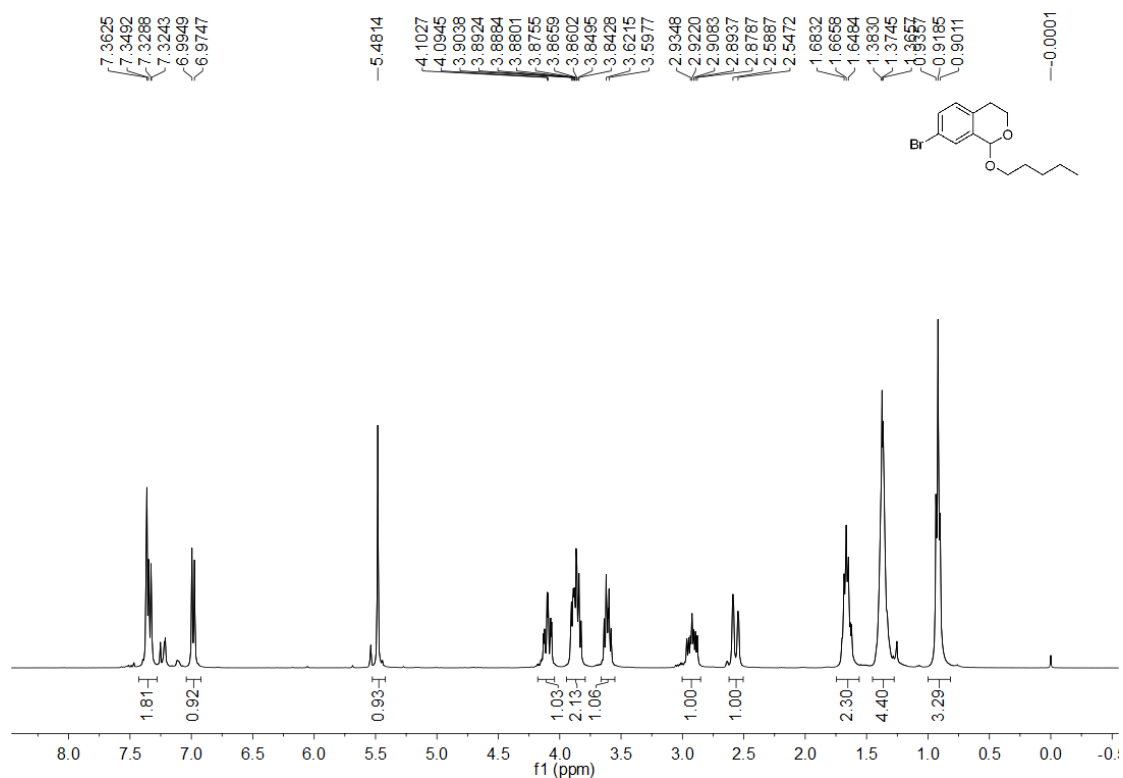
# 1-(Cyclopentyloxy)isochromane (**3y**)



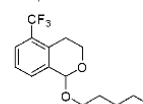
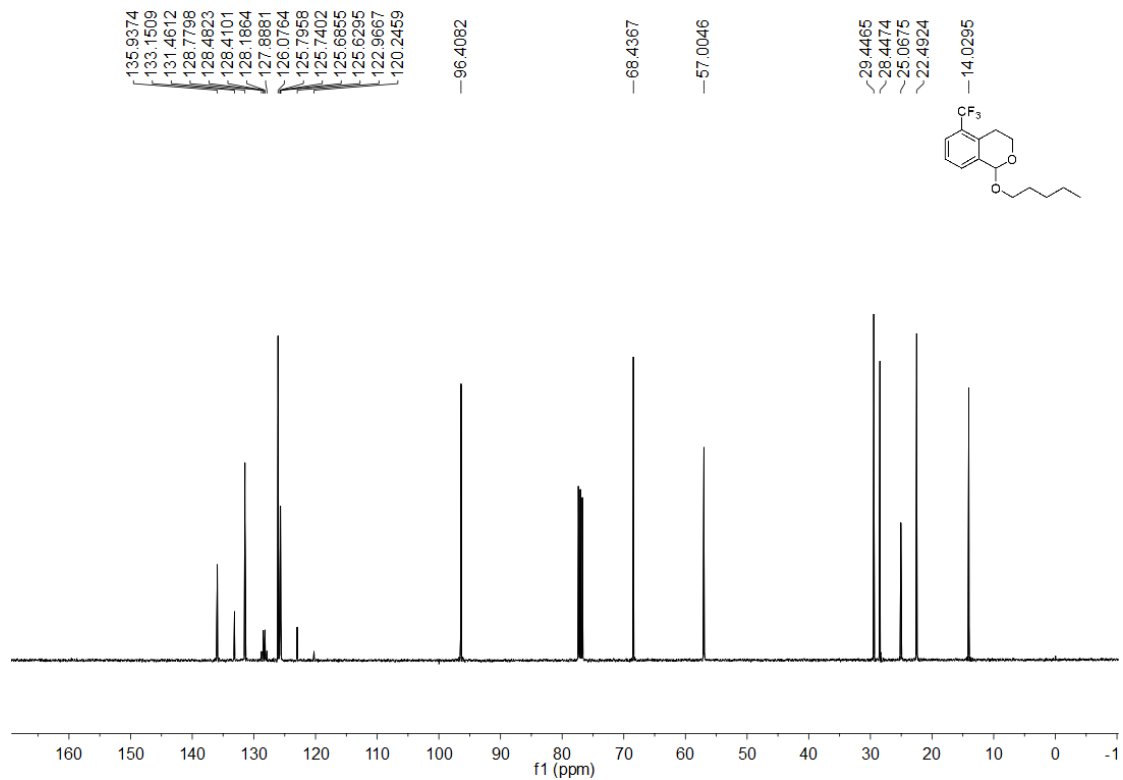
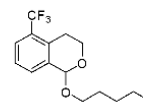
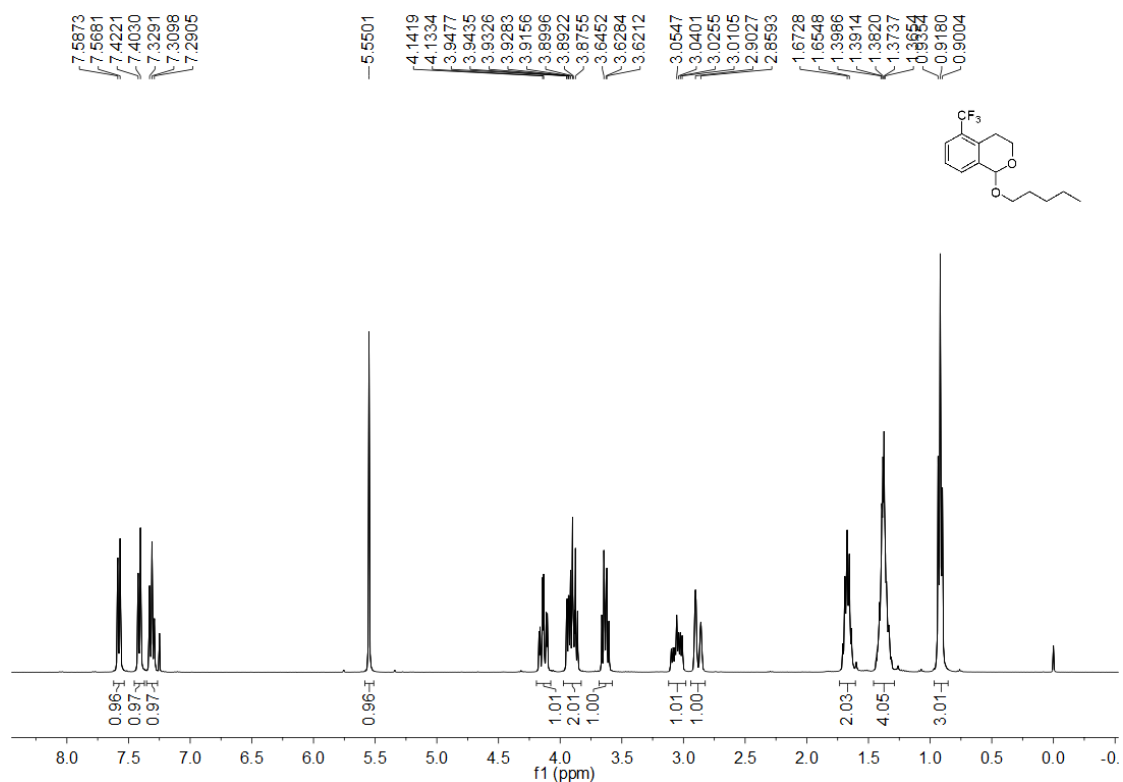
1-(*t*-Butoxy)isochromane (**3z**)

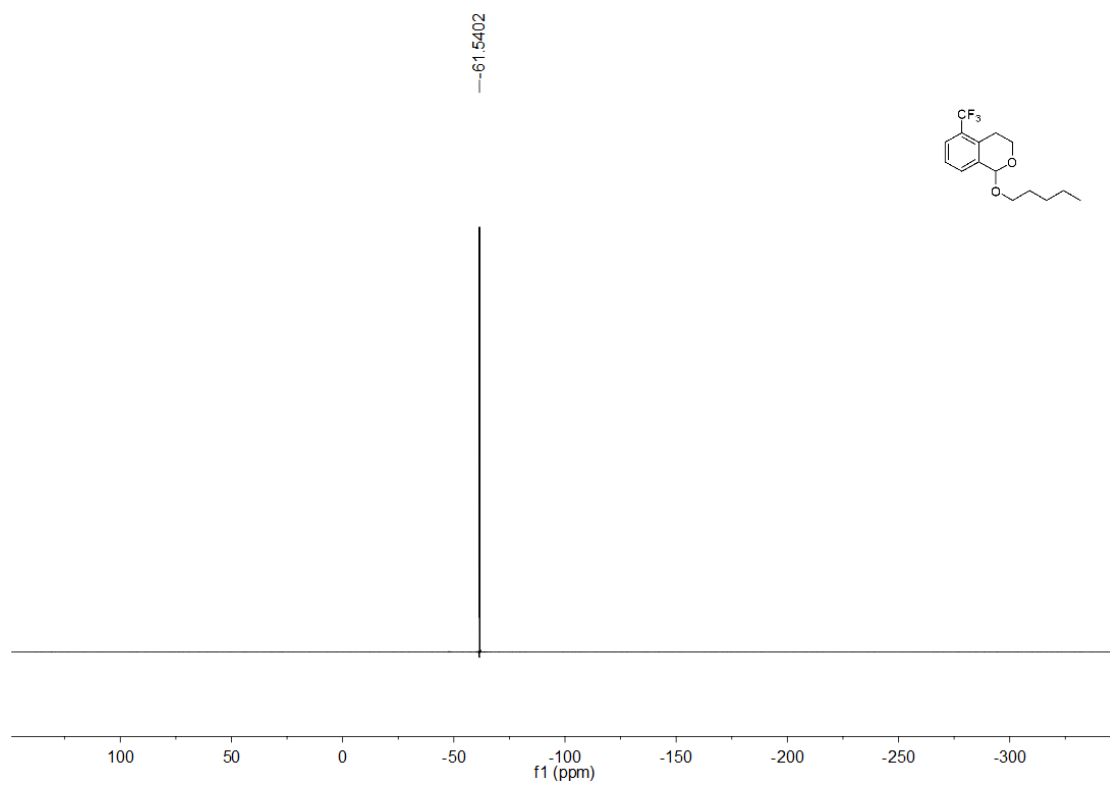


# 7-Bromo-1-(pentyloxy)isochromane (4b)

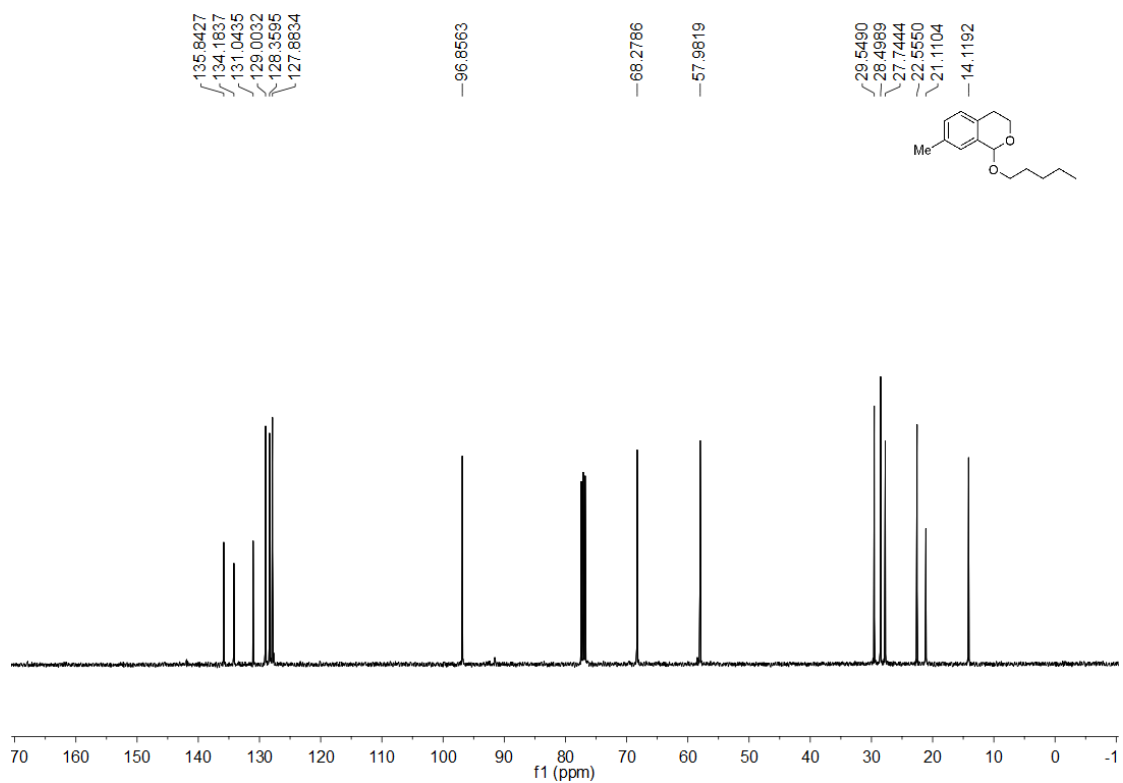
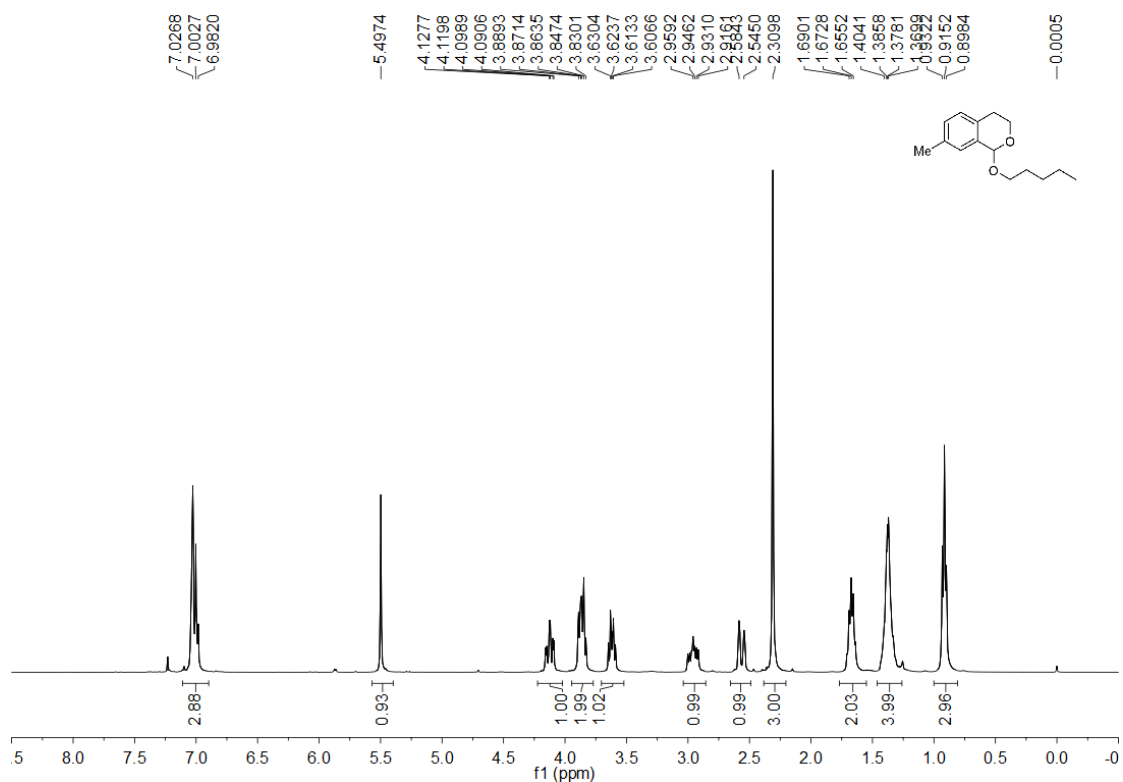


# 1-(Pentyloxy)-5-(trifluoromethyl)isochromane (4c)



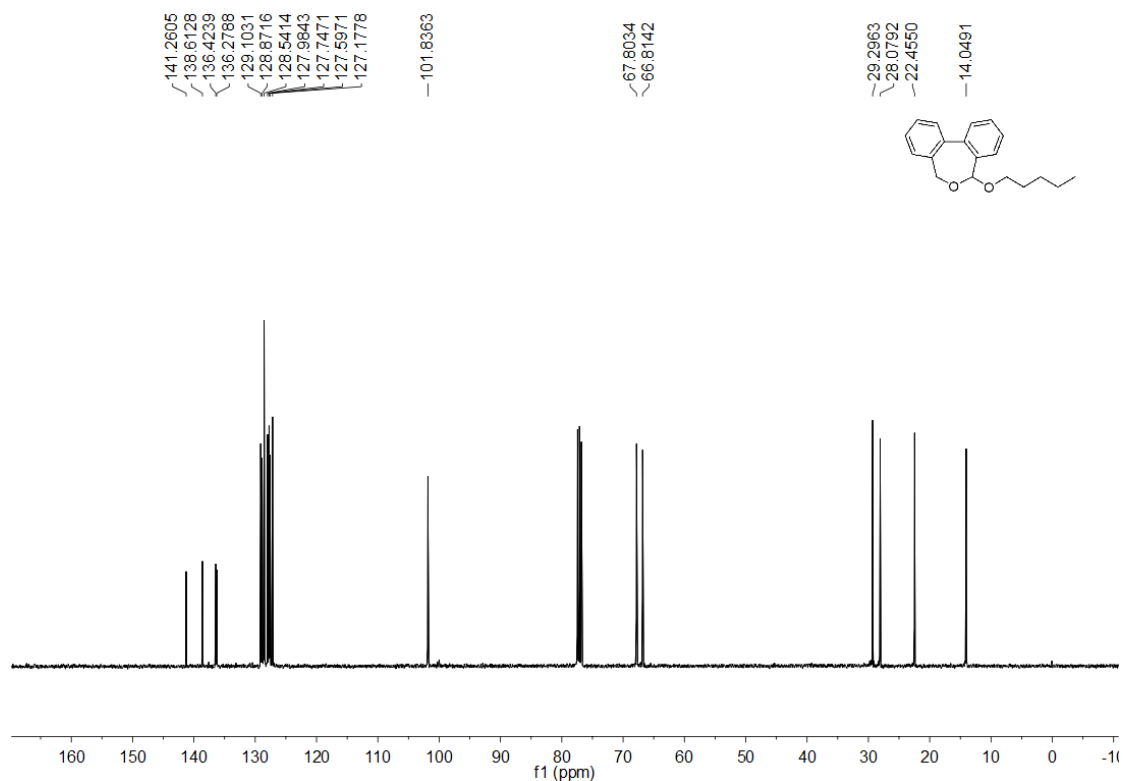
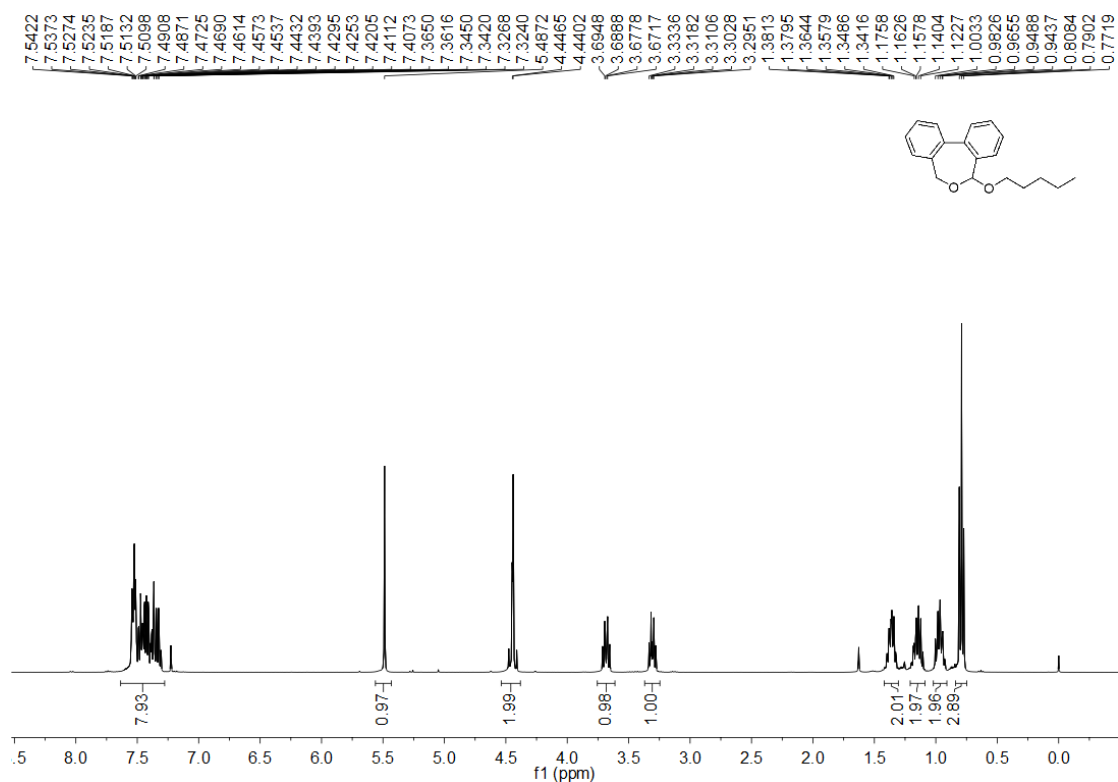


# 7-Methyl-1-(pentyloxy)isochromane (**4d**)

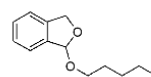
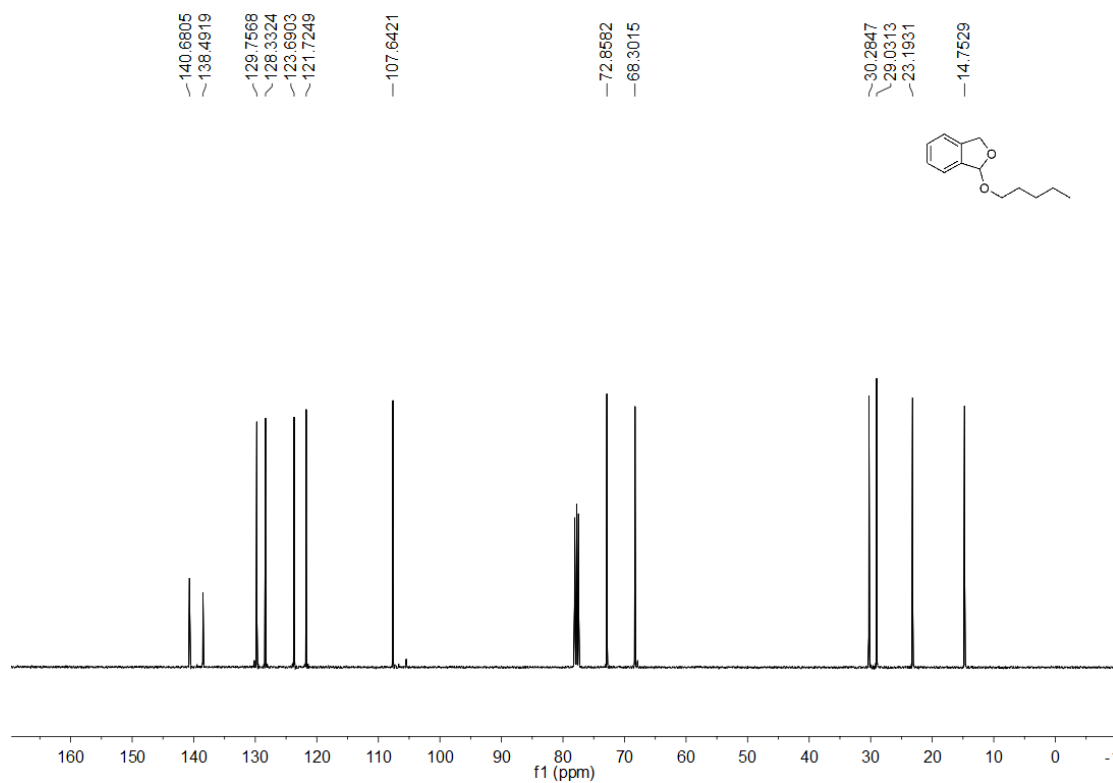
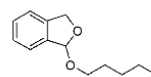
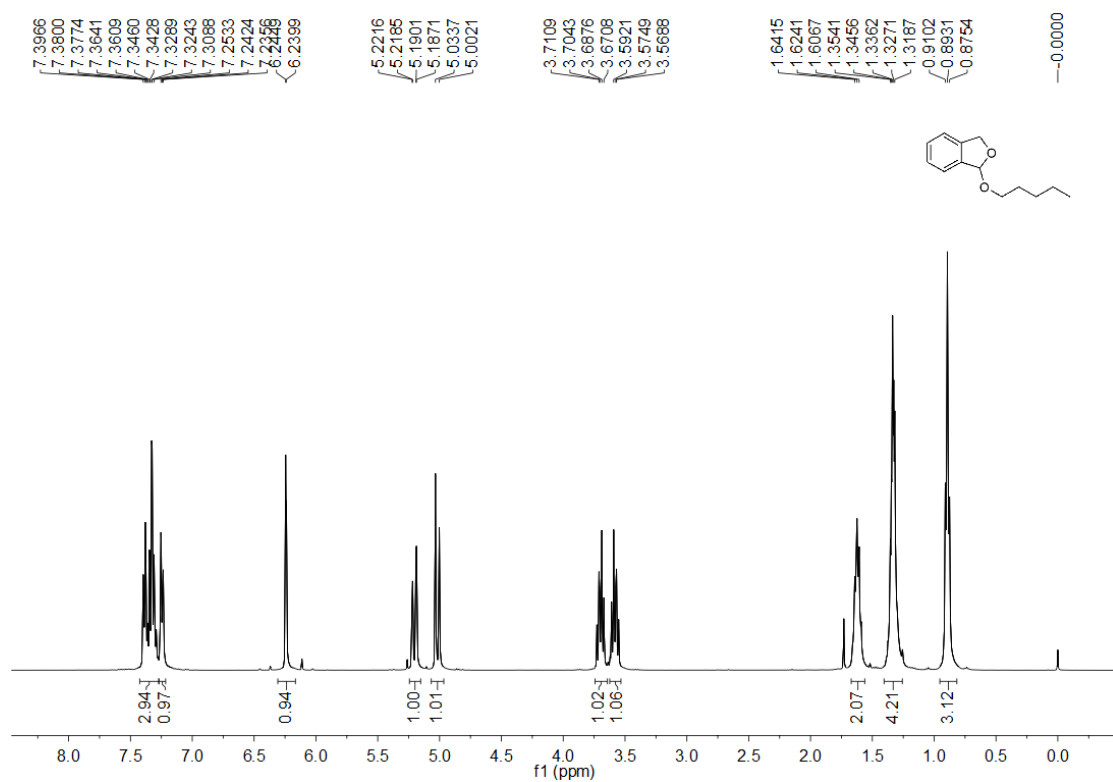




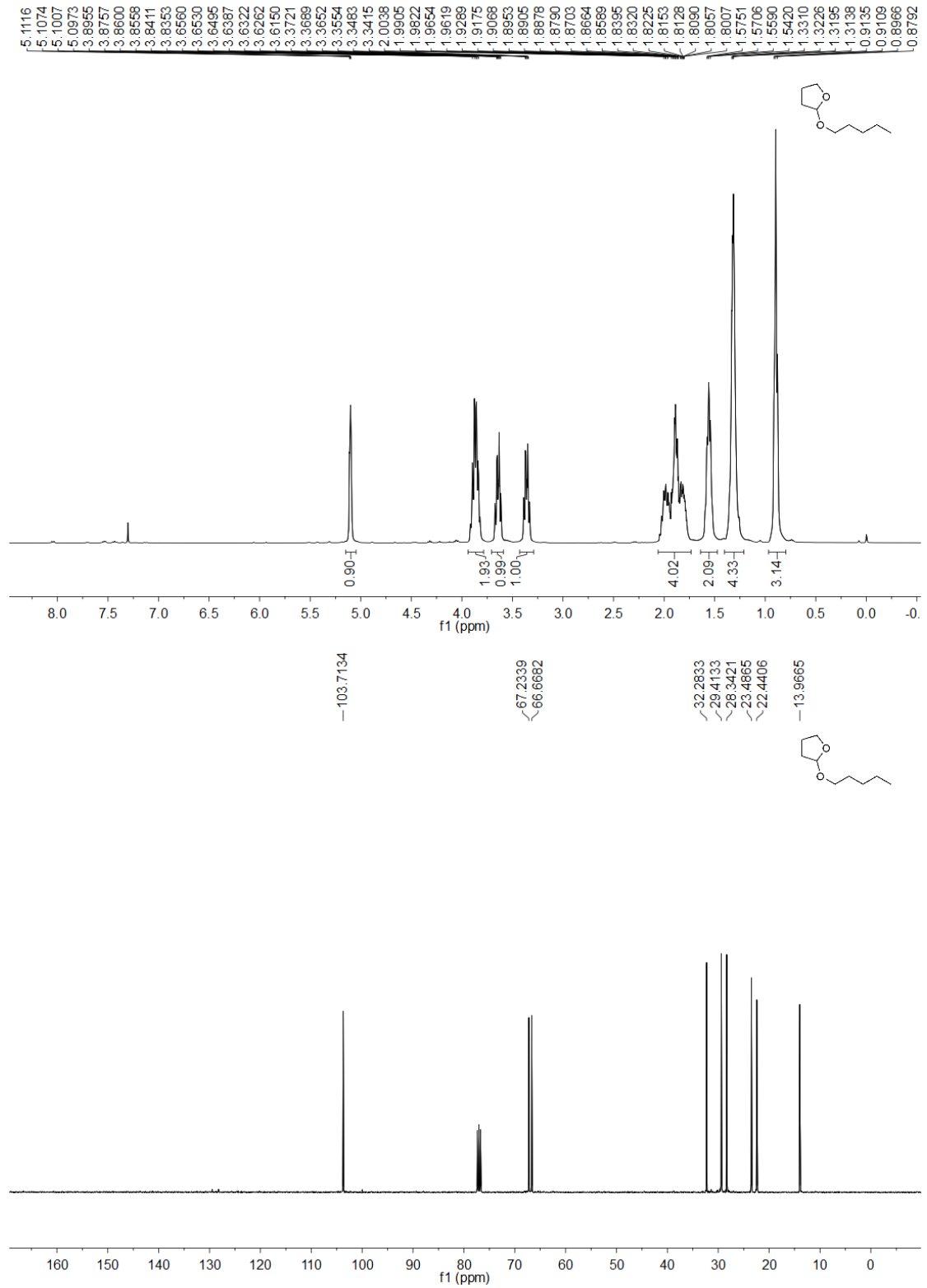
5-(Pentyloxy)-5,7-dihydrodibenzo[c,e]oxepine (**4e**)



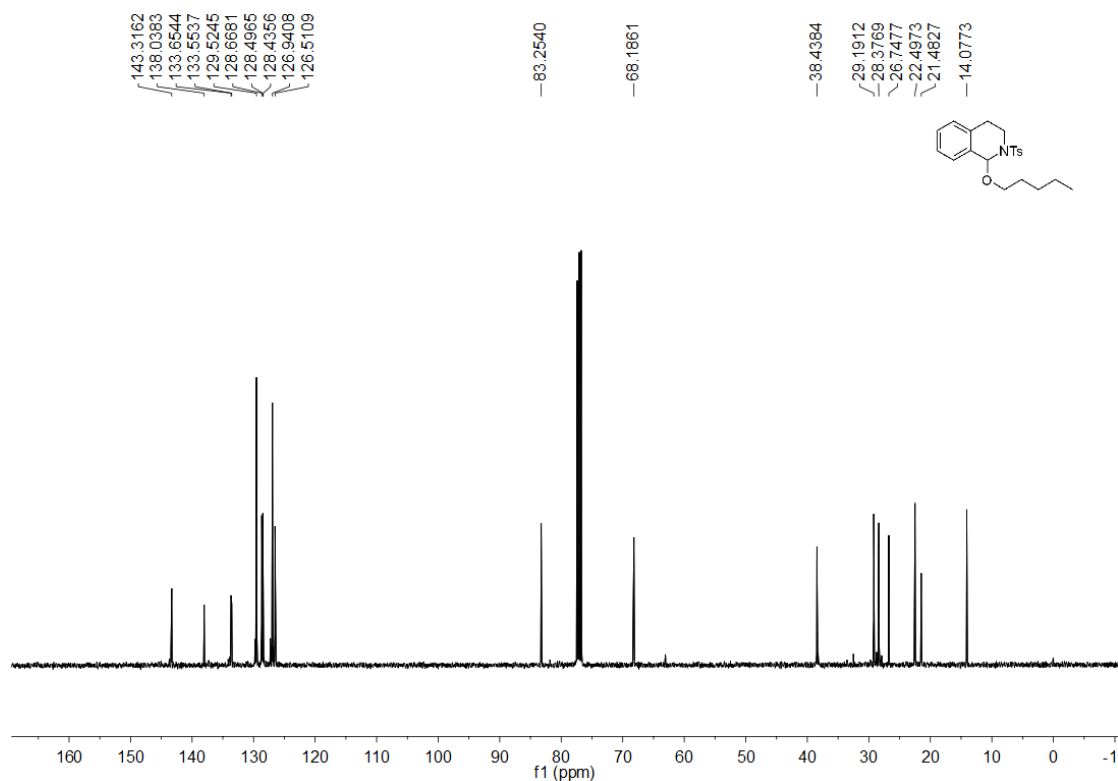
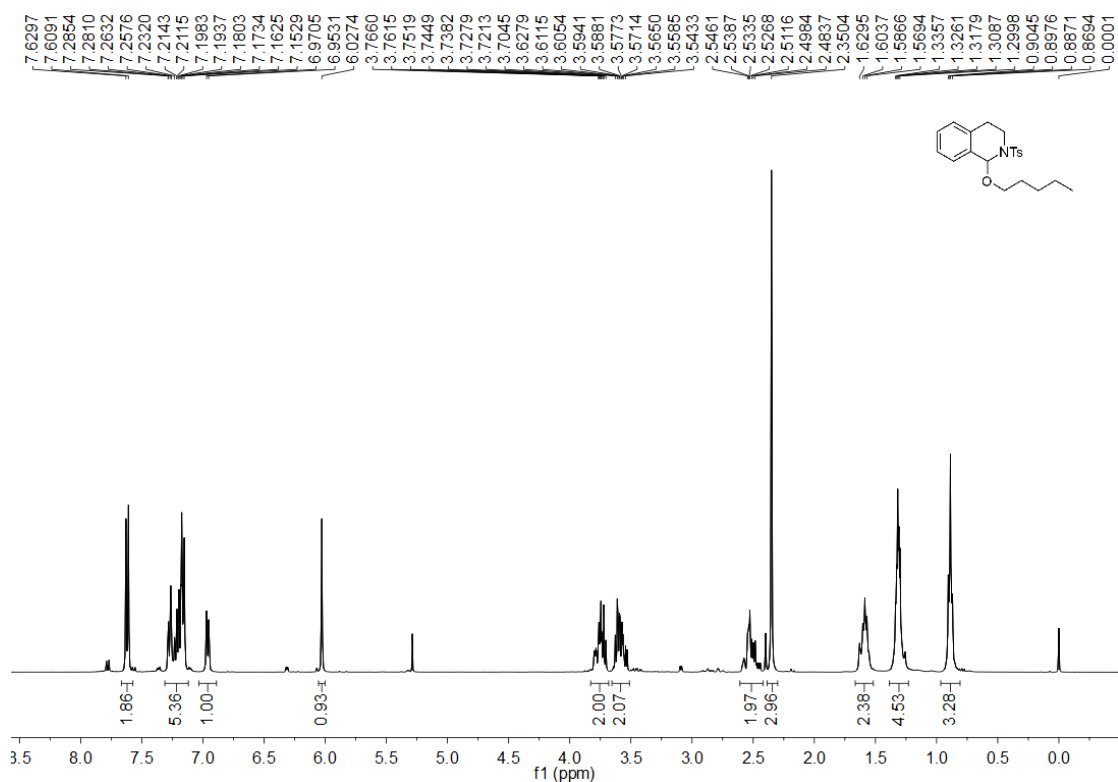
# 1-(Pentyloxy)-1,3-dihydroisobenzofuran (**4f**)



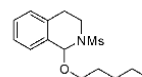
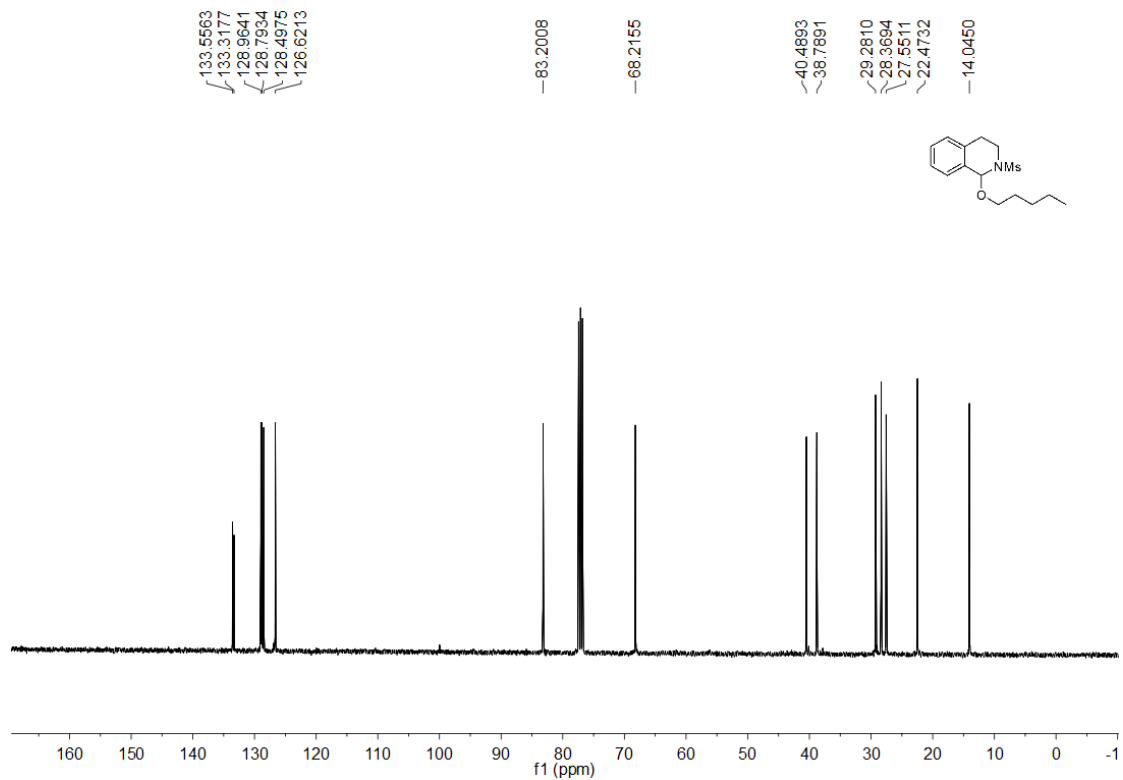
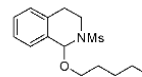
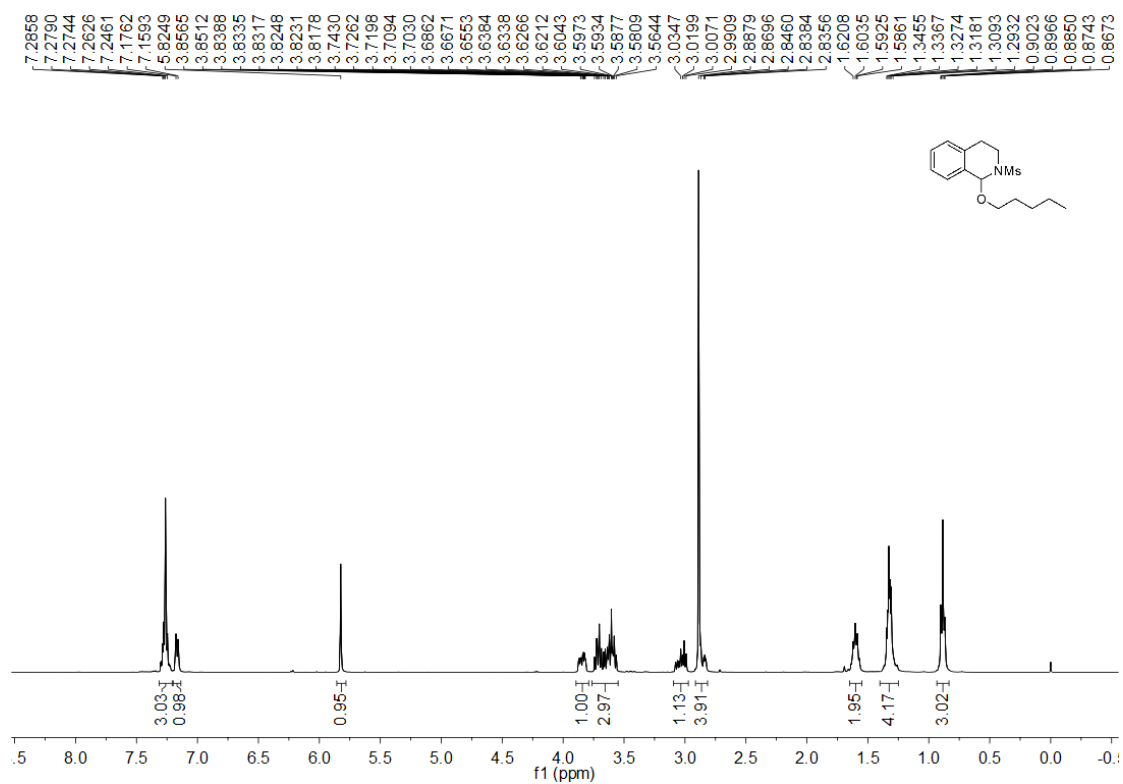
2-(Pentyloxy)tetrahydrofuran (**4g**)



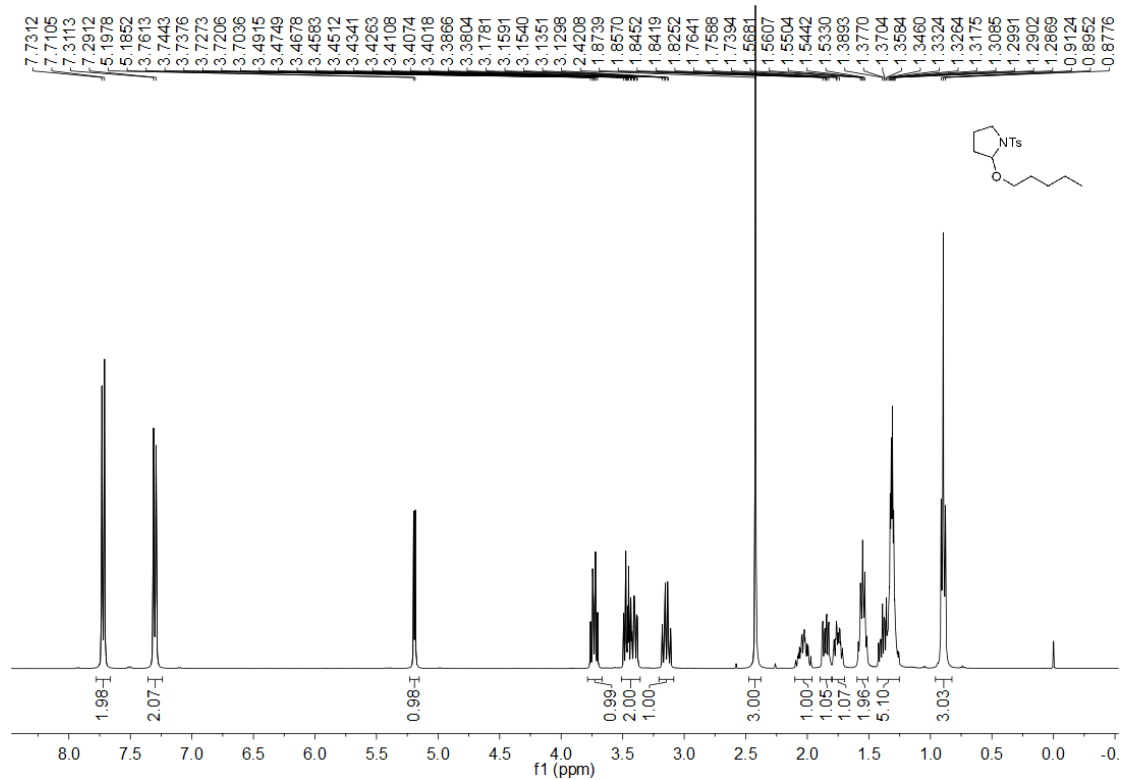
1-(Pentyloxy)-2-tosyl-1,2,3,4-tetrahydroisoquinoline (**4h**)



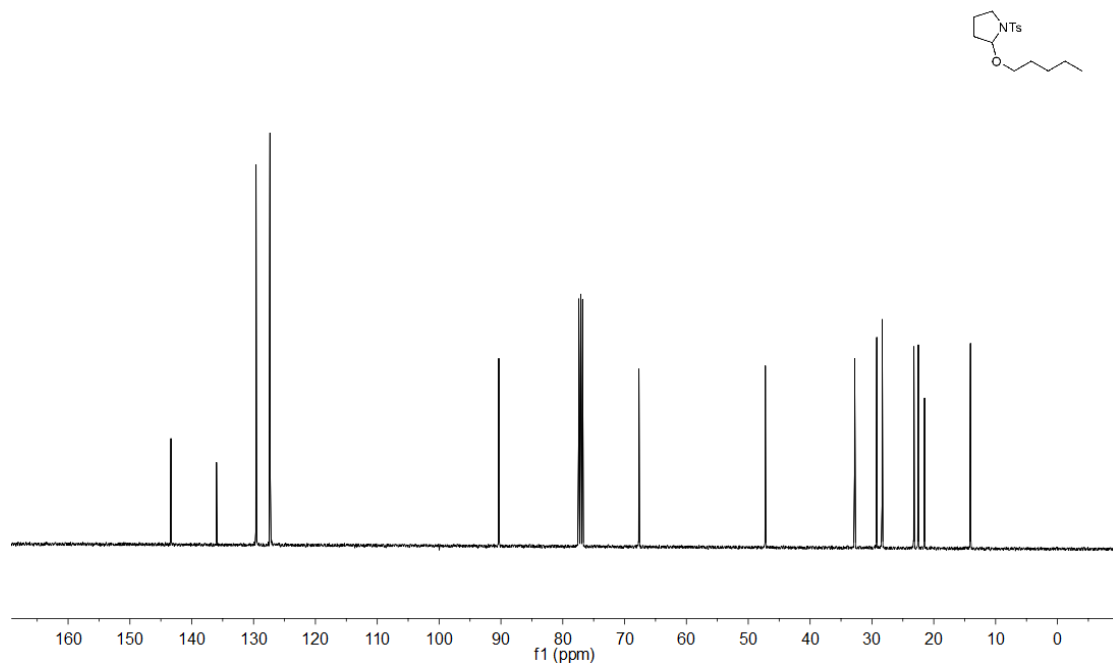
2-(Methylsulfonyl)-1-(pentyloxy)-1,2,3,4-tetrahydroisoquinoline (**4i**)



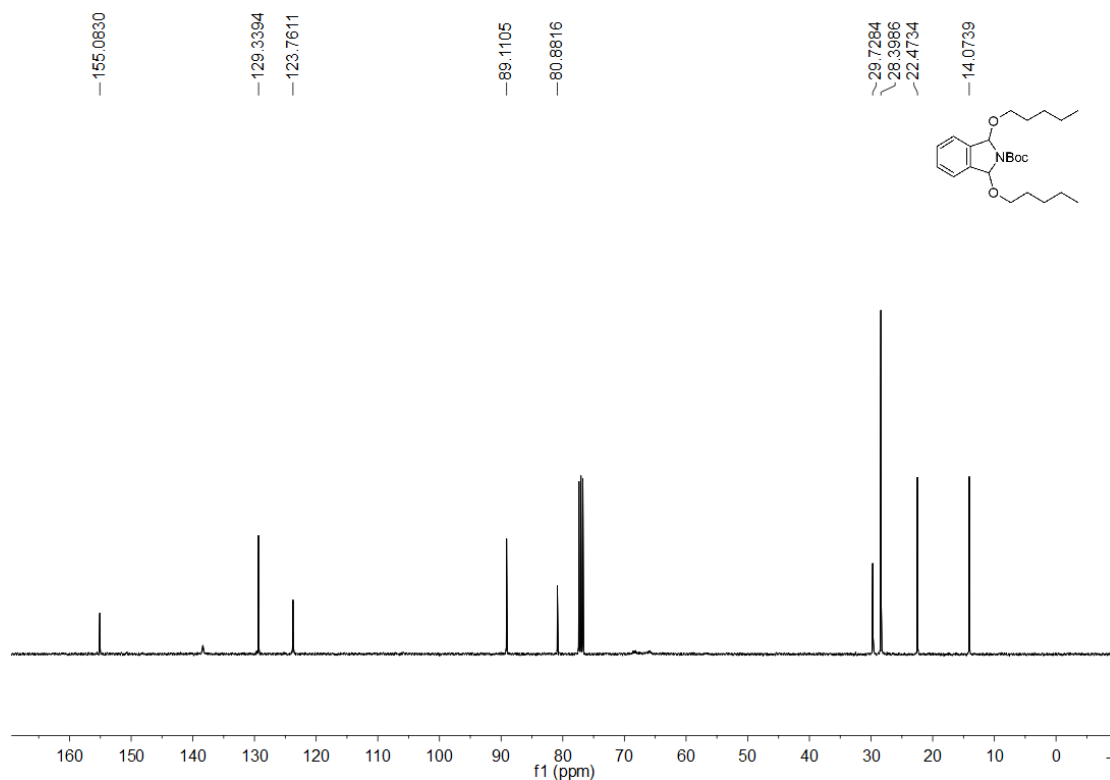
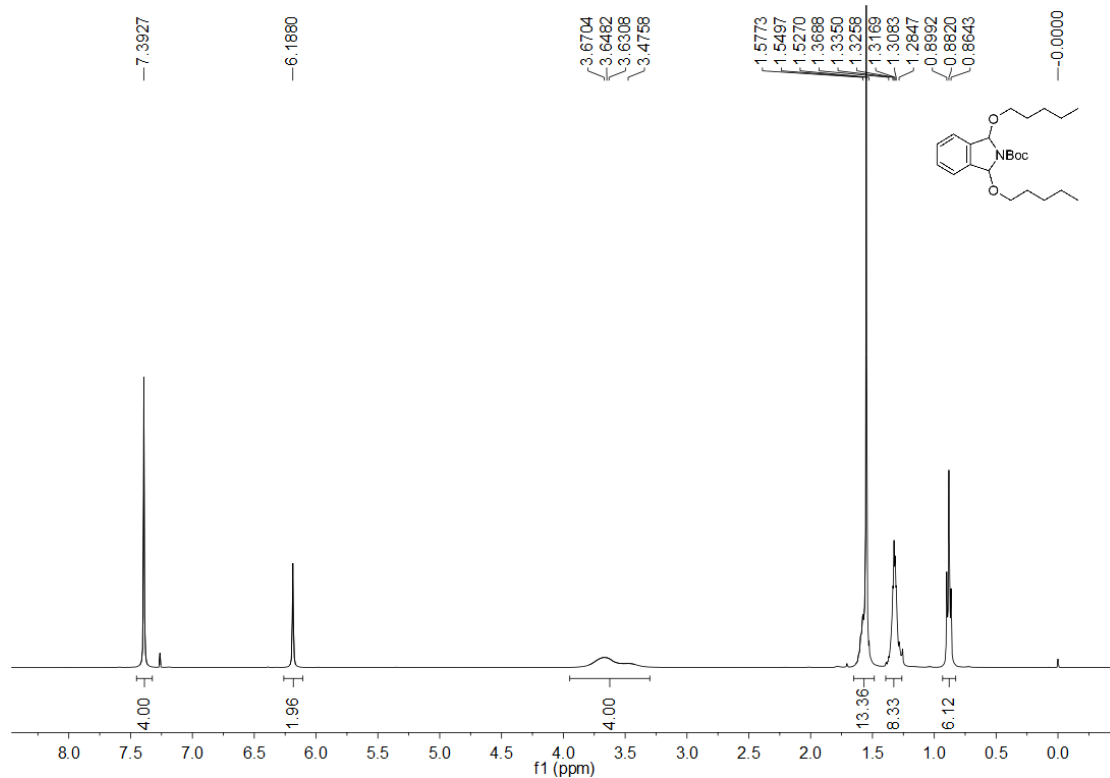
2-(Pentyloxy)-1-tosylpyrrolidine (4j)



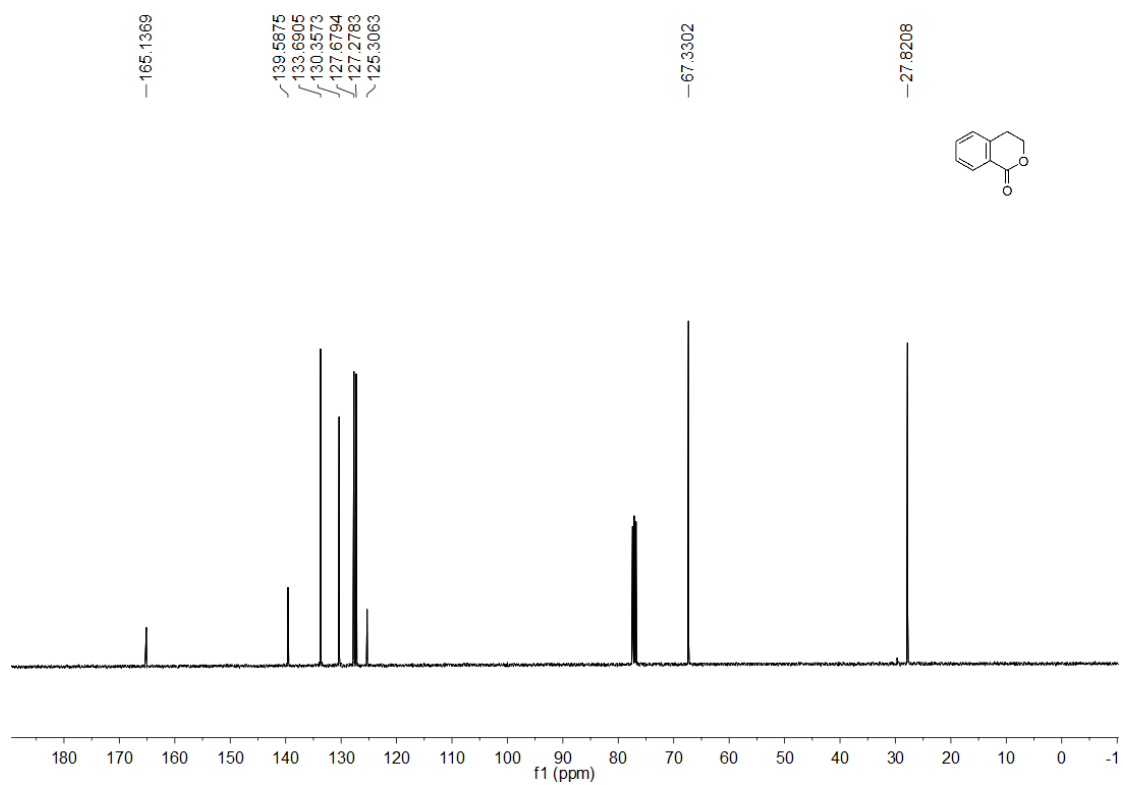
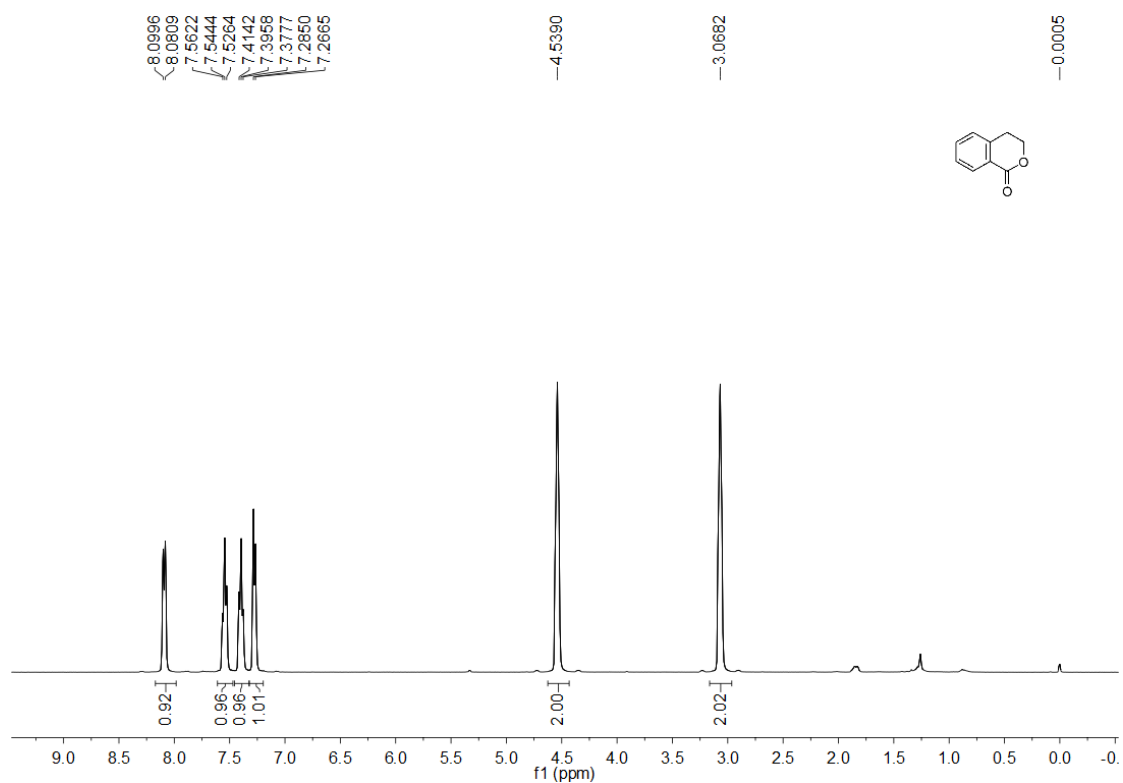
- 143.3611
- 136.0063
- 129.6046
- 127.3482
- 90.3445
- 67.6803
- 47.2391
- 32.7950
- 29.2157
- 28.3453
- 23.2133
- 22.4939
- 21.4873
- 14.0552



*t*-Butyl 1,3-bis(pentyloxy)isoindoline-2-carboxylate (**4k**)



# Isochroman-1-one (5)





## References

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