

## Supporting Information

### Copper-Catalyzed Redox-Triggered Remote C-H Functionalization:

#### Highly Selective Formation of C-CF<sub>3</sub> and C-O Bonds

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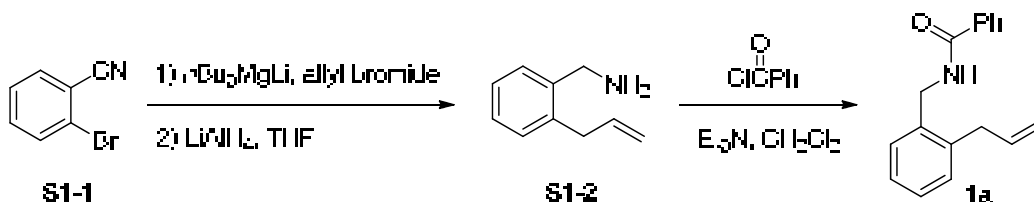
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### General information

All reactions were carried out under argon (Ar) atmosphere using Schlenk techniques with magnetic stirring. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. CuI was purchased from Aldrich, ethyl acetate (EA) was purchased anhydrous from commercial sources and transferred under an argon atmosphere. All alcohols are commercial available and were purified under standard methods or dried over molecular Sieves (4Å) before use. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 F254 plates. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040-0.063 mm). Visualization on TLC was achieved by use of UV light (254 nm) or iodine. NMR spectra were recorded on a Bruker DPX 400 spectrometer at 400 MHz for  $^1\text{H}$  NMR, 100 MHz for  $^{13}\text{C}$  NMR and 376 MHz for  $^{19}\text{F}$  NMR in  $\text{CDCl}_3$  with tetramethylsilane (TMS) as internal standard. Chemical shifts are reported in ppm and coupling constants are given in Hz. Data for  $^1\text{H}$  NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quarter; m, multiplet), coupling constant (Hz), integration. Data for  $^{13}\text{C}$  NMR are reported in terms of chemical shift ( $\delta$ , ppm).  $^{19}\text{F}$  NMR spectra were recorded on a Bruker DPX 400 MHz spectrometer [ $\text{CFCl}_3$  as an external reference (0 ppm)]. HMRS were obtained on a Bruker Apex IV RTMS.

### Synthetic procedures and spectral data for substrates

#### Representative procedure for the synthesis of 1a

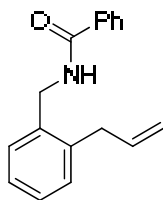


**N-(2-Allylbenzyl)benzamide (1a)** (2-Allylphenyl)methanamine was prepared according to literature procedure.<sup>S1</sup> Briefly, to a solution of *n*-butylmagnesium bromide in THF (10 mL of a 1 M solution in THF, 2.8 mmol) at 0 °C was added *n*-BuLi (8.0 mL of a 2.5 M solution in hexanes, 20 mmol). After being stirred for 20 min at 0 °C, the reaction mixture was cooled to -40 °C and 2-bromobenzonitrile (3.64 g, 20.0 mmol) was added slowly as a solution in THF (20 mL). After being stirred for an additional 30 min, a solution of  $\text{CuCN}\cdot 2\text{LiCl}$  (949.0 mg, 6.5 mmol) in THF (5 mL) was added, followed by allylbromide (6.93 mL, 80.0 mmol). After 20 min, the reaction mixture was quenched with aqueous  $\text{NH}_4\text{Cl}$  and EtOAc, warmed to room temperature, and stirred vigorously for 30 min. The aqueous layer was extracted with EtOAc ( $3\times 30$  mL), washed with brine, dried with anhydrous  $\text{Na}_2\text{SO}_4$ , filtered, and concentrated *in vacuo*. The crude material was purified by flash column chromatography (EtOAc/petroleum ether = 1/10) to afford 2-allylbenzamide (2.3 g, 81%) as a colorless oil.

To a suspension of  $\text{LiAlH}_4$  (425.4 mg, 11.2 mmol) in THF (15 mL) at 0 °C was slowly added a solution of 2-bromobenzonitrile (401.0 mg, 2.8 mmol) in THF (2.5 mL). After being stirred for 3 h at 0 °C, the reaction mixture was quenched by slow, sequential addition of water (0.5 mL) in  $\text{Na}_2\text{SO}_4$  (3.0 g). The reaction mixture was warmed to room temperature, stirred for an additional 30 min, filtered, and concentrated *in vacuo*. The crude material was purified by flash

chromatography on silicagel (EtOAc/petroleum ether = 1/2 to EtOAc/Et<sub>3</sub>N = 50/1) to afford 2-allylbenzylamine (315.1 mg, 79%) as a colorless oil.

To a solution of 2-allylbenzylamine (315.1 mg, 2.1 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was added Et<sub>3</sub>N (0.8 mL, 6.3 mmol) followed by slowly adding a solution of benzoyl chloride (323.3 mg, 2.3 mmol) at 0 °C, and then stirred for 2 h at room temperature. The reaction mixture was concentrated *in vacuo* and purified by flash chromatography on silica gel (EtOAc/petroleum ether = 1/8) to afford **1a** (464.4 mg, 88%) as a white solid.



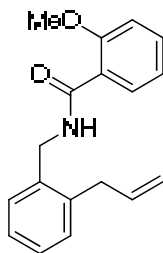
**1a**

***N*-(2-allylbenzyl)benzamide (1a)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.75-7.72 (m, 2H), 7.48-7.43 (m, 1H), 7.38-7.31 (m, 3H), 7.27-7.17 (m, 3H), 6.62 (d, *J* = 5.2 Hz, 1H), 5.98 (ddt, *J* = 17.2, 10.0, 6.0 Hz, 1H), 5.06 (dq, *J* = 10.0, 1.6 Hz, 1H), 4.96 (dq, *J* = 16.8, 1.6 Hz, 1H), 4.59 (d, *J* = 5.2 Hz, 2H), 3.44 (d, *J* = 6.4 Hz, 2H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 167.03, 137.93, 137.24, 135.79, 134.17, 131.34, 130.10, 129.15, 128.38, 127.90, 126.88, 126.73, 115.94, 41.68, 36.81.

HRMS (ESI) *m/z* calcd. for C<sub>17</sub>H<sub>17</sub>NO [M+Na]<sup>+</sup> 274.1208, found 274.1202.



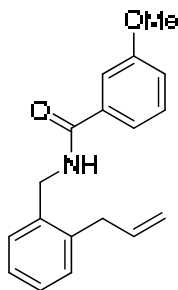
**1b**

***N*-(2-allylbenzyl)-2-methoxybenzamide (1b)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.23 (dd, *J* = 8.0, 2.0 Hz, 1H), 8.07 (s, 1H), 7.45-7.36 (m, 2H), 7.25-7.19 (m, 3H), 7.07 (td, *J* = 7.6, 0.8 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 5.98 (ddt, *J* = 16.8, 10.0, 6.4 Hz, 1H), 5.09-4.98 (m, 2H), 4.68 (d, *J* = 5.6 Hz, 2H), 3.87 (s, 3H), 3.48 (d, *J* = 6.4 Hz, 2H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.01, 157.33, 137.73, 136.67, 136.32, 132.70, 132.23, 129.83, 128.41, 127.53, 126.64, 121.19, 115.91, 111.16, 55.76, 41.17, 36.86.

HRMS (ESI) *m/z* calcd. for C<sub>18</sub>H<sub>19</sub>NO<sub>2</sub> [M+Na]<sup>+</sup> 304.1313, found 304.1293.



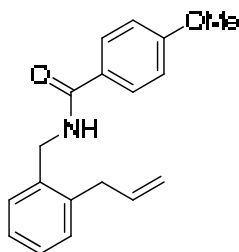
1c

***N*-(2-allylbenzyl)-3-methoxybenzamide (1c)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.37-7.33 (m, 2H), 7.31-7.20 (m, 5H), 7.01 (ddd,  $J = 7.6, 2.8, 1.6$  Hz, 1H), 6.43 (s, 1H), 6.00 (ddt,  $J = 16.8, 10.0, 6.4$  Hz, 1H), 5.08 (dq,  $J = 10.0, 1.6$  Hz, 1H), 4.98 (dq,  $J = 17.2, 2.0$  Hz, 1H), 4.62 (d,  $J = 5.2$  Hz, 2H), 3.81 (s, 3H), 3.47 (d,  $J = 6.0$  Hz, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.86, 159.72, 138.05, 137.35, 135.74, 135.70, 130.23, 129.45, 129.34, 128.05, 126.83, 118.61, 117.65, 116.02, 112.27, 55.34, 41.85, 36.88.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{18}\text{H}_{19}\text{NO}_2$   $[\text{M}+\text{Na}]^+$  304.1313, found 304.1270.



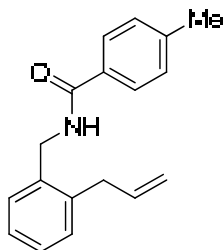
1d

***N*-(2-allylbenzyl)-4-methoxybenzamide (1d)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74-7.70 (m, 2H), 7.34-7.32 (m, 1H), 7.28-7.19 (m, 3H), 6.87 (d,  $J = 8.8$  Hz, 2H), 6.43 (s, 1H), 5.99 (ddt,  $J = 16.4, 10.0, 6.0$  Hz, 1H), 5.07 (dq,  $J = 10.0, 1.6$  Hz, 1H), 4.97 (dq,  $J = 16.8, 1.6$  Hz, 1H), 4.60 (d,  $J = 5.6$  Hz, 2H), 3.81 (s, 4H), 3.45 (d,  $J = 6.0$  Hz, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.54, 162.08, 138.04, 137.34, 136.03, 130.15, 129.26, 128.71, 127.91, 126.77, 126.56, 115.95, 113.63, 55.29, 41.73, 36.85.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{18}\text{H}_{19}\text{NO}_2$   $[\text{M}+\text{Na}]^+$  304.1313, found 304.1296.



1e

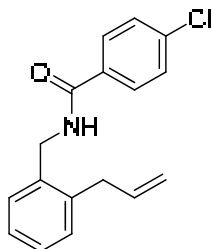
***N*-(2-allylbenzyl)-4-methylbenzamide (1e)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d,  $J = 8.4$  Hz, 2H), 7.34-7.32 (m, 1H), 7.28-7.17 (m, 5H), 6.44 (s, 1H), 5.99 (ddt,  $J = 17.2, 10.0, 6.0$  Hz, 1H), 5.05 (dq,  $J = 10.0, 1.6$  Hz, 1H), 4.97 (dq,  $J = 17.2, 2.0$  Hz, 1H), 4.61 (d,  $J = 5.6$  Hz, 2H), 3.45 (d,  $J = 6.0$  Hz, 2H), 2.37 (s, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.94, 141.78, 138.03, 137.31, 135.90, 131.35, 130.15, 129.28,

129.09, 127.94, 126.89, 126.78, 115.97, 41.73, 36.85, 21.36.

HRMS (ESI)  $m/z$  calcd. for  $C_{18}H_{19}NO$   $[M+Na]^+$  288.1364, found 288.1304.



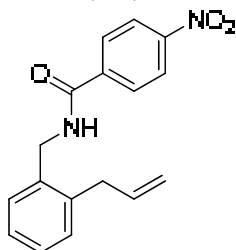
**1f**

***N*-(2-allylbenzyl)-4-chlorobenzamide (1f)**

$^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.68 (dt,  $J = 9.2, 2.4$  Hz, 2H), 7.40-7.36 (m, 2H), 7.35-7.33 (m, 1H), 7.29-7.27 (m, 1H), 7.25-7.22 (m, 2H), 6.33 (s, 1H), 6.01 (ddt,  $J = 17.2, 10.0, 6.0$  Hz, 1H), 5.08 (dq,  $J = 10.4, 1.6$  Hz, 1H), 4.97 (dq,  $J = 16.8, 1.6$  Hz, 1H), 4.63 (d,  $J = 5.6$  Hz, 2H), 3.47 (dt,  $J = 6.0, 1.6$  Hz, 2H).

$^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  166.04, 137.92, 137.54, 137.24, 135.60, 132.58, 130.16, 129.10, 128.60, 128.37, 127.99, 126.76, 115.95, 41.77, 36.81.

HRMS (ESI)  $m/z$  calcd. for  $C_{17}H_{16}ClNO$   $[M+Na]^+$  308.0818, found 308.0811.



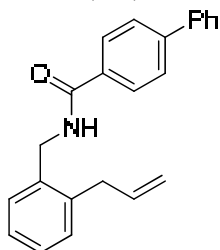
**1g**

***N*-(2-allylbenzyl)-4-nitrobenzamide (1g)**

$^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.23-8.20 (m, 2H), 7.91-7.88 (m, 2H), 7.34-7.21 (m, 4H), 6.70 (s, 1H), 6.00 (ddt,  $J = 17.2, 10.0, 6.0$  Hz, 1H), 5.07 (dq,  $J = 10.0, 1.6$  Hz, 1H), 4.96 (dq,  $J = 17.2, 1.6$  Hz, 1H), 4.63 (d,  $J = 5.2$  Hz, 2H), 3.46 (d,  $J = 6.0$  Hz, 2H).

$^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  164.98, 149.49, 139.81, 138.03, 137.44, 135.16, 130.44, 129.41, 128.35, 128.12, 126.95, 123.69, 116.05, 42.15, 36.91.

HRMS (ESI)  $m/z$  calcd. for  $C_{17}H_{16}N_2O_3$   $[M+Na]^+$  319.1059, found 319.1052.



**1h**

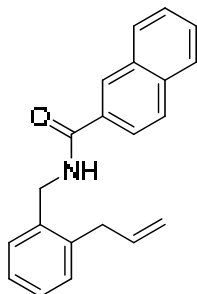
***N*-(2-allylbenzyl)-[1,1'-biphenyl]-4-carboxamide (1h)**

$^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.88-7.84 (m, 2H), 7.68-7.61 (m, 5H), 7.50-7.46 (m, 2H), 7.44-7.38 (m, 2H), 7.31 (dd,  $J = 6.8, 2.8$  Hz, 1H), 7.30-7.25 (m, 1H), 6.37 (s, 1H), 6.06 (ddt,  $J = 17.2, 10.0,$

6.0 Hz, 1H), 5.13 (dq,  $J = 10.4, 1.6$  Hz, 1H), 5.02 (dq,  $J = 17.2, 1.6$  Hz, 1H), 4.70 (d,  $J = 5.2$  Hz, 2H), 3.53 (dt,  $J = 6.0, 1.6$  Hz, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.75, 144.24, 139.93, 138.12, 137.39, 135.85, 132.91, 130.27, 129.39, 128.85, 128.08, 127.93, 127.45, 127.14, 127.13, 126.87, 116.04, 41.89, 36.91.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{23}\text{H}_{21}\text{NO}$   $[\text{M}+\text{Na}]^+$  350.1521, found 350.1515.



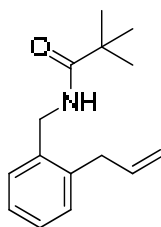
**1i**

***N*-(2-Allylbenzyl)-2-naphthamide (1i)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.28 (s, 1H), 7.90-7.81 (m, 5H), 7.58-7.50 (m, 2H), 7.40 (d,  $J = 7.2$  Hz, 1H), 7.33-7.24 (m, 2H), 6.49 (s, 1H), 6.04 (ddt,  $J = 17.2, 10.0, 6.0$  Hz, 1H), 5.10 (dq,  $J = 10.4, 1.6$  Hz, 1H), 5.01 (dq,  $J = 17.2, 1.6$  Hz, 1H), 4.71 (d,  $J = 5.2$  Hz, 2H), 3.51 (d,  $J = 6.4$  Hz, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.09, 138.10, 137.34, 135.86, 134.64, 132.53, 131.47, 130.22, 129.35, 128.83, 128.32, 128.02, 127.64, 127.54, 127.41, 126.82, 126.62, 123.55, 116.01, 41.93, 36.88.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{21}\text{H}_{19}\text{NO}$   $[\text{M}+\text{Na}]^+$  324.1364, found 324.1360.



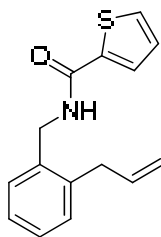
**1j**

***N*-(2-Allylbenzyl)pivalamide (1j)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.27-7.18 (m, 4H), 6.03-5.93 (m, 2H), 5.08 (dd,  $J = 10.0, 1.2$  Hz, 1H), 4.96 (dd,  $J = 16.8, 1.6$  Hz, 1H), 4.42 (d,  $J = 5.2$  Hz, 2H), 3.41 (d,  $J = 6.0$  Hz, 2H), 1.19 (s, 9H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  177.92, 137.85, 137.23, 136.12, 130.07, 128.91, 127.73, 126.68, 115.86, 41.24, 38.57, 36.68, 27.47.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{15}\text{H}_{21}\text{NO}$   $[\text{M}+\text{Na}]^+$  254.1521, found 254.1512.



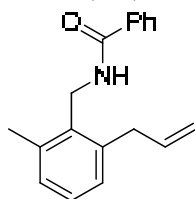
**1k**

***N*-(2-Allylphenyl)thiophene-2-carboxamide (1k)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.46-7.45 (m, 2H), 7.35 (d,  $J = 7.6$  Hz, 1H), 7.30-7.21 (m, 3H), 7.04 (dd,  $J = 5.2, 4.0$  Hz, 1H), 6.27 (s, 1H), 6.06-5.96 (m, 1H), 5.09 (dq,  $J = 10.0, 1.6$  Hz, 1H), 4.98 (dq,  $J = 16.8, 1.6$  Hz, 1H), 4.61 (d,  $J = 5.6$  Hz, 2H), 3.47 (d,  $J = 6.4$  Hz, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.49, 138.77, 138.02, 137.39, 135.68, 130.22, 129.88, 129.38, 128.05, 128.03, 127.52, 126.82, 116.02, 41.69, 36.89.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{15}\text{H}_{15}\text{NOS}$   $[\text{M}+\text{Na}]^+$  280.0772, found 280.0767.



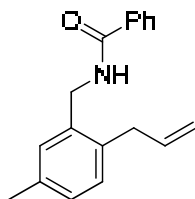
**1l**

***N*-(2-Allyl-6-methylphenyl)benzamide (1l)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.73-7.70 (m, 2H), 7.50-7.46 (m, 1H), 7.42-7.38 (m, 3H), 7.23-7.18 (m, 1H), 7.13-7.09 (m, 1H), 6.05-6.00 (m, 1H), 5.08 (dq,  $J = 10.0, 1.6$  Hz, 1H), 4.97 (dq,  $J = 17.2, 2.0$  Hz, 1H), 4.67 (d,  $J = 4.8$  Hz, 2H), 3.51 (dt,  $J = 6.0, 1.6$  Hz, 2H), 2.41 (s, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.96, 139.15, 138.08, 138.02, 134.20, 133.74, 131.31, 129.01, 128.40, 128.15, 128.07, 126.81, 115.82, 38.34, 37.49, 19.75.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{18}\text{H}_{19}\text{NO}$   $[\text{M}+\text{Na}]^+$  288.1364, found 288.1360.



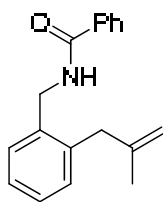
**1m**

***N*-(2-Allyl-5-methylphenyl)benzamide (1m)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74-7.72 (m, 2H), 7.47-7.43 (m, 1H), 7.36 (t,  $J = 7.6$  Hz, 3H), 7.21 (d,  $J = 8.4$  Hz, 1H), 7.02 (s, 1H), 6.53 (s, 1H), 5.97 (ddt,  $J = 17.2, 10.4, 6.0$  Hz, 1H), 5.06 (dq,  $J = 10.4, 1.6$  Hz, 1H), 4.97 (dq,  $J = 17.2, 2.0$  Hz, 1H), 4.56 (d,  $J = 5.6$  Hz, 2H), 3.41 (d,  $J = 6.4$  Hz, 2H), 2.31 (s, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.94, 137.88, 137.62, 137.42, 134.30, 132.79, 131.27, 130.8, 129.39, 128.36, 127.40, 126.87, 115.83, 41.52, 36.83, 20.94.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{18}\text{H}_{19}\text{NO}$   $[\text{M}+\text{Na}]^+$  288.1364, found 288.1361.



**1n**

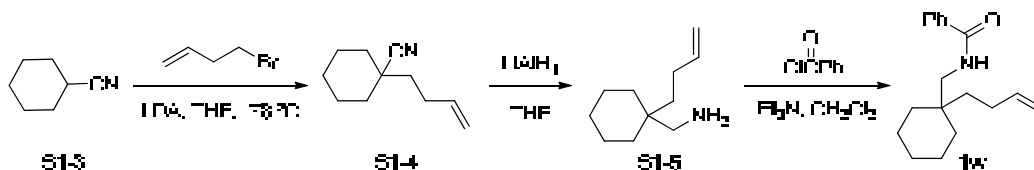
***N*-2-(2-Methylallyl)benzyl)benzamide (1n)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76-7.73 (m, 2H), 7.49-7.44 (m, 1H), 7.40-7.34 (m, 3H), 7.28-7.17 (m, 3H), 6.52 (s, 1H), 4.84 (s, 1H), 4.60 (d,  $J = 5.2$  Hz, 2H), 4.50 (d,  $J = 0.8$  Hz, 1H), 3.39 (s, 2H), 1.74 (d,  $J = 0.4$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.96, 145.78, 137.77, 136.12, 134.22, 131.36, 130.87, 129.41, 128.42, 127.81, 126.87, 126.82, 111.80, 41.93, 41.04, 22.90.

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{18}\text{H}_{19}\text{NO}$   $[\text{M}+\text{Na}]^+$  288.1364, found 288.1360.

**Synthetic procedure for 1w**



***N*-((1-(But-3-en-1-yl)cyclohexyl)methyl)benzamide**

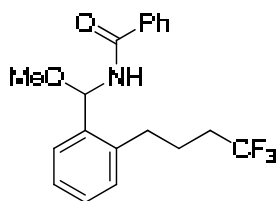
**(1w)**

(1-(But-3-en-1-yl)cyclohexyl)methanamine (**S1-5**) was synthesized according to the procedures previously reported by Widenhoefer.<sup>S2</sup> To a solution of **S1-5** (334.6 mg, 2.0 mmol) in  $\text{CH}_2\text{Cl}_2$  (10 mL) was added  $\text{Et}_3\text{N}$  (0.8 mL, 6.0 mmol) followed by slowly adding a solution of benzoyl chloride (309.3 mg, 2.2 mmol) at 0 °C, and then stirred for 2 h at room temperature. The reaction mixture was concentrated *in vacuo* and purified by flash chromatography on silica gel (EtOAc/petroleum ether = 1/8) to afford **1w** (488.5 mg, 90%) as a white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.73 (d,  $J = 7.6$  Hz, 2H), 7.49-7.38 (m, 3H), 6.19 (s, 1H), 5.82 (ddt,  $J = 16.8, 10.4, 6.0$  Hz, 1H), 5.02 (d,  $J = 17.2$  Hz, 1H), 4.93 (d,  $J = 10.4$  Hz, 1H), 3.37 (d,  $J = 6.0$  Hz, 2H), 2.04 (dd,  $J = 16.0, 6.8$  Hz, 2H), 1.55-1.29 (m, 12H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.82, 139.43, 135.24, 131.41, 128.69, 126.97, 114.44, 45.85, 36.67, 35.24, 33.84, 27.58, 26.34, 21.74, 21.59. HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{18}\text{H}_{25}\text{NO}$   $[\text{M}+\text{Na}]^+$  294.1834, found 294.1829.

**Synthetic Procedures and Spectral Data for *N,O*-aminals**

To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1** (0.2 mmol, 1.0 equiv.), Togni's reagent **2a** (88.5 mg, 0.28 mmol) and  $\text{CuI}$  (7.6 mg, 0.04 mmol). The tube was evacuated and backfilled with Ar for three times, and then alcohol (32.5  $\mu\text{L}$ , 0.8 mmol) and solvent (EtOAc, 1.0 mL) were added *via* syringe. The tube was stirred at 45 °C. After reaction completed (monitored by TLC), the reaction solution was concentrated *in vacuo* and the crude residue was purified by silica gel column chromatography (petroleum ether/EtOAc = 10/1-20/1) to give the corresponding *N,O*-amininal products.





**3a**

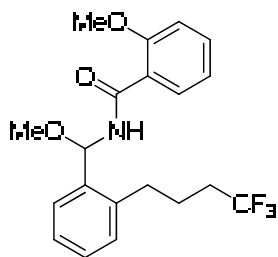
***N*-(Methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3a)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.79-7.76 (m, 2H), 7.65 (dd,  $J = 6.8, 2.0$  Hz, 1H), 7.54-7.50 (m, 1H), 7.45-7.41 (m, 2H), 7.34-7.27 (m, 2H), 7.23-7.20 (m, 1H), 6.61-6.57 (m, 1H), 6.51 (d,  $J = 9.2$  Hz, 1H), 3.52 (s, 3H), 2.82-2.69 (m, 2H), 2.20-2.07 (m, 2H), 1.92-1.75 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.95, 138.53, 137.01, 133.40, 132.07, 129.65, 128.74, 128.66, 126.99, 126.93 (q,  $J_{\text{CF}} = 274.6$  Hz), 126.87, 125.33, 78.92, 55.98, 33.39 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.86, 23.49 (d,  $J = 2.8$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.28 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{19}\text{H}_{20}\text{F}_3\text{NO}_2$  [ $\text{M}+\text{Na}$ ] $^+$  374.1344, found 374.1334.



**3b**

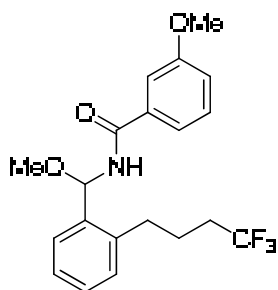
**2-Methoxy-*N*-(methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3b)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.37 (d,  $J = 9.2$  Hz, 1H), 8.23 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.63-7.60 (m, 1H), 7.48-7.44 (m, 1H), 7.32-7.27 (m, 2H), 7.22-7.19 (m, 1H), 7.09 (t,  $J = 7.6$  Hz, 1H), 6.95 (d,  $J = 8.4$  Hz, 1H), 6.55 (d,  $J = 9.2$  Hz, 1H), 3.87 (s, 3H), 3.53 (s, 3H), 2.85-2.73 (m, 2H), 2.18-2.04 (m, 2H), 1.94-1.77 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.06, 157.55, 138.58, 137.51, 133.35, 132.45, 129.60, 128.48, 126.99 (q,  $J_{\text{CF}} = 274.6$  Hz), 126.75, 125.37, 121.36, 120.62, 111.32, 79.02, 55.85, 33.32 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.97, 23.46 (d,  $J_{\text{CF}} = 2.8$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.32 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_3$  [ $\text{M}+\text{Na}$ ] $^+$  404.1449, found 404.1444.



**3c**

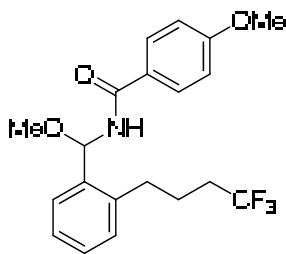
**3-Methoxy-*N*-(methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3c)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65-7.63 (m, 1H), 7.37 (t,  $J = 2.0$  Hz, 1H), 7.33-7.25 (m, 4H), 7.20 (dd,  $J = 7.2, 2.0$  Hz, 1H), 7.05 (ddd,  $J = 8.0, 2.4, 0.8$  Hz, 1H), 6.59 (d,  $J = 9.2$  Hz, 1H), 6.50 (d,  $J = 9.6$  Hz, 1H), 3.83 (s, 3H), 3.52 (s, 3H), 2.82-2.69 (m, 2H), 2.20-2.08 (m, 2H), 1.92-1.75 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.82, 159.83, 138.50, 136.94, 134.87, 129.60, 128.72, 126.93 (q,  $J_{\text{CF}} = 274.6$  Hz), 126.84, 125.32, 118.62, 118.23, 112.39, 78.94, 55.95, 55.35, 33.37 (q,  $J_{\text{CF}} = 28.5$  Hz), 30.85, 23.47 (d,  $J_{\text{CF}} = 2.8$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.29 (s, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_3$   $[\text{M}+\text{Na}]^+$  404.1449, found 404.1444.



**3c**

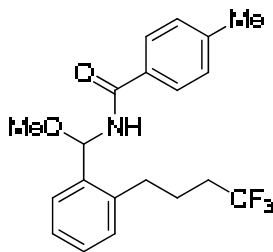
**4-Methoxy-*N*-(methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3d)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76-7.72 (m, 2H), 7.66-7.64 (m, 1H), 7.33-7.27 (m, 2H), 7.21-7.19 (m, 1H), 6.91 (d,  $J = 8.8$  Hz, 2H), 6.49 (s, 2H), 3.83 (s, 3H), 3.51 (s, 3H), 2.81-2.68 (m, 2H), 2.16-2.08 (m, 2H), 1.88-1.77 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.37, 162.61, 138.56, 137.17, 129.62, 128.91, 128.67, 126.94 (q,  $J_{\text{CF}} = 274.5$  Hz), 126.83, 125.53, 125.32, 113.81, 78.86, 55.92, 55.38, 33.38 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.85, 23.48 (d,  $J_{\text{CF}} = 2.8$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.29 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_3$   $[\text{M}+\text{Na}]^+$  404.1449, found 404.1441.



**3d**

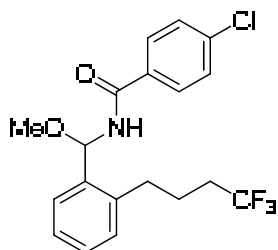
***N*-(Methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)-4-methylbenzamide (3e)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69-7.64 (m, 3H), 7.34-7.27 (m, 2H), 7.24-7.19 (m, 3H), 6.56 (d,  $J = 9.2$  Hz, 1H), 6.51 (d,  $J = 9.6$  Hz, 1H), 3.52 (s, 3H), 2.82-2.69 (m, 2H), 2.39 (s, 3H), 2.19-2.07 (m, 2H), 1.91-1.76 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.92, 142.70, 138.63, 137.20, 130.61, 129.68, 129.36, 128.75, 127.02 (q,  $J_{\text{CF}} = 274.8$  Hz), 127.09, 126.90, 125.42, 78.96, 56.00, 33.46 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.93, 23.55 (d,  $J_{\text{CF}} = 2.8$  Hz), 21.47.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.31 (s, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  388.1500, found 388.1495.



**3f**

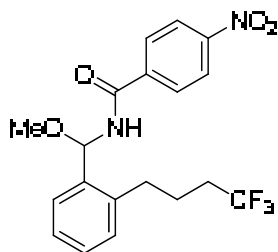
**4-Chloro-*N*-(methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3f)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.72-7.69 (m, 2H), 7.65-7.62 (m, 1H), 7.41-7.37 (m, 2H), 7.34-7.26 (m, 2H), 7.22-7.19 (m, 1H), 6.62 (d, *J* = 9.2 Hz, 1H), 6.48 (d, *J* = 9.6 Hz, 1H), 3.50 (s, 3H), 2.79-2.69 (m, 2H), 2.19-2.06 (m, 2H), 1.89-1.75 (m, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.92, 138.54, 138.36, 136.79, 131.76, 129.69, 128.90, 128.83, 128.47, 126.91 (q, *J*<sub>CF</sub> = 274.8 Hz), 126.90, 125.36, 79.07, 56.02, 33.38 (q, *J*<sub>CF</sub> = 28.4 Hz), 30.88, 23.50 (d, *J*<sub>CF</sub> = 2.8 Hz).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -66.26 (t, *J* = 10.9 Hz, 3F).

HRMS (ESI) *m/z* calcd. for C<sub>19</sub>H<sub>19</sub>ClF<sub>3</sub>NO<sub>2</sub> [M+Na]<sup>+</sup> 408.0954, found 408.0947.



**3g**

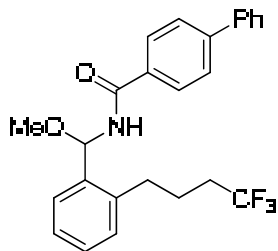
***N*-(Methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)-4-nitrobenzamide (3g)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.17 (d, *J* = 8.4 Hz, 2H), 7.86 (d, *J* = 8.8 Hz, 2H), 7.56 (d, *J* = 7.2 Hz, 1H), 7.26-7.19 (m, 2H), 7.13 (d, *J* = 6.8 Hz, 1H), 6.69 (d, *J* = 9.2 Hz, 1H), 6.40 (d, *J* = 9.2 Hz, 1H), 3.44 (s, 3H), 2.73-2.62 (m, 2H), 2.10-2.00 (m, 2H), 1.84-1.66 (m, 2H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.99, 149.81, 138.94, 138.51, 136.37, 129.79, 129.03, 128.27, 127.02, 126.88 (q, *J*<sub>CF</sub> = 274.7 Hz), 125.37, 123.85, 79.25, 56.20, 33.37 (q, *J*<sub>CF</sub> = 28.4 Hz), 30.93, 23.55 (d, *J*<sub>CF</sub> = 2.9 Hz).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -66.24 (t, *J* = 10.9 Hz, 3F).

HRMS (ESI) *m/z* calcd. for C<sub>19</sub>H<sub>19</sub>F<sub>3</sub>N<sub>2</sub>O<sub>4</sub> [M+Na]<sup>+</sup> 419.1195, found 419.1188.



**3h**

***N*-(Methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)-[1,1'-biphenyl]-4-carboxamide (3h)**

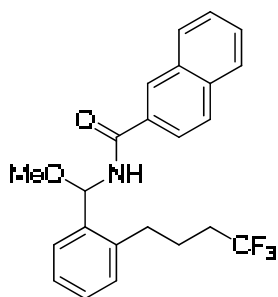
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.69-7.59 (m, 5H), 7.47 (t, *J* = 7.6 Hz,

2H), 7.40 (d,  $J = 6.8$  Hz, 1H), 7.35-7.30 (m, 2H), 7.24-7.21 (m, 1H), 6.60 (d,  $J = 9.6$  Hz, 1H), 6.54 (d,  $J = 9.6$  Hz, 1H), 3.55 (s, 3H), 2.85-2.71 (m, 2H), 2.21-2.10 (m, 2H), 1.93-1.80 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.64, 144.93, 139.78, 138.57, 137.06, 132.01, 129.68, 128.91, 128.77, 128.10, 127.56, 127.32, 127.19, 126.96 (q,  $J_{\text{CF}} = 274.8$  Hz), 126.91, 125.36, 78.98, 56.04, 33.43 (q,  $J_{\text{CF}} = 28.3$  Hz), 30.91, 23.52 (d,  $J_{\text{CF}} = 2.9$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.28 (s, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{25}\text{H}_{24}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  450.1657, found 450.1649.



**3i**

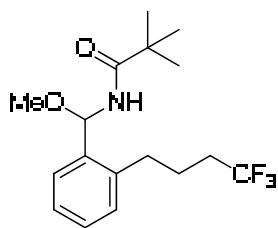
***N*-(Methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)-2-naphthamide (3i)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.29 (s, 1H), 7.89-7.82 (m, 4H), 7.73-7.70 (m, 1H), 7.55 (dt,  $J = 16.8, 6.4$  Hz, 2H), 7.35-7.30 (m, 2H), 7.23-7.21 (m, 1H), 6.79 (d,  $J = 9.2$  Hz, 1H), 6.58 (d,  $J = 9.2$  Hz, 1H), 3.57 (s, 3H), 2.86-2.72 (m, 2H), 2.22-2.11 (m, 2H), 1.95-1.78 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.06, 138.60, 137.03, 134.91, 132.45, 130.58, 129.65, 128.90, 128.75, 128.59, 127.92, 127.70, 127.68, 126.94 (q,  $J_{\text{CF}} = 274.7$  Hz), 126.87, 126.85, 125.40, 123.36, 79.04, 56.03, 33.40 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.89, 23.50 (d,  $J_{\text{CF}} = 2.7$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.28 (s, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{23}\text{H}_{22}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  424.1500, found 424.1495.



**3j**

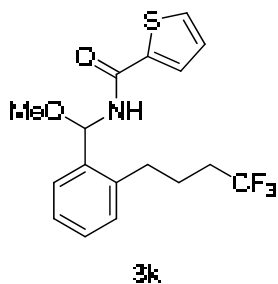
***N*-(Methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)pivalamide (3j)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58-7.56 (m, 1H), 7.34-7.27 (m, 2H), 7.22-7.20 (m, 1H), 6.29 (d,  $J = 9.6$  Hz, 1H), 6.01 (d,  $J = 9.2$  Hz, 1H), 3.42 (s, 3H), 2.71 (t,  $J = 8.0$  Hz, 2H), 2.22-2.09 (m, 2H), 1.89-1.80 (m, 2H), 1.23 (s, 9H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  178.21, 138.45, 137.16, 129.58, 128.60, 126.99 (q,  $J_{\text{CF}} = 274.6$  Hz), 126.85, 125.20, 78.44, 55.78, 38.88, 33.40 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.77, 27.36, 23.43 (d,  $J_{\text{CF}} = 2.8$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.21 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{17}\text{H}_{24}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  354.1657, found 354.1650.



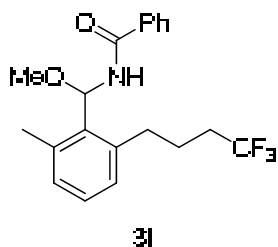
***N*-(Methoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)thiophene-2-carboxamide (3k)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.65 (dd, *J* = 7.2, 1.6 Hz, 1H), 7.51 (m, 2H), 7.33-7.27 (m, 2H), 7.20 (dd, *J* = 7.2, 2.0 Hz, 1H), 7.06 (dd, *J* = 4.8, 4.0 Hz, 1H), 6.46 (s, 2H), 3.52 (s, 3H), 2.82-2.67 (m, 2H), 2.19-2.06 (m, 2H), 1.92-1.75 (m, 2H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.36, 138.56, 137.85, 136.86, 130.97, 129.62, 128.75, 128.63, 127.69, 126.94 (q, *J*<sub>CF</sub> = 274.8 Hz), 126.80, 125.38, 78.99, 55.99, 33.37 (q, *J*<sub>CF</sub> = 28.4 Hz), 30.84, 23.50 (d, *J*<sub>CF</sub> = 2.7 Hz).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -66.31 (s, 3F).

HRMS (ESI) *m/z* calcd. for C<sub>17</sub>H<sub>18</sub>F<sub>3</sub>NO<sub>2</sub>S [M+Na]<sup>+</sup> 380.0908, found 380.0901.



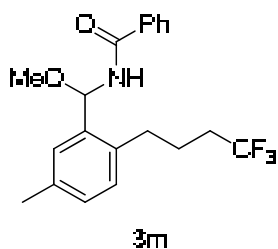
***N*-(Methoxy(2-methyl-6-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3l)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.77-7.75 (m, 2H), 7.54-7.51 (m, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.08 (dd, *J* = 14.4, 7.2 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 1H), 6.67 (d, *J* = 8.8 Hz, 1H), 3.51 (s, 3H), 2.93 (tq, *J* = 20.8, 7.6 Hz, 2H), 2.58 (s, 3H), 2.25-2.13 (m, 2H), 1.98-1.81 (m, 2H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 167.17, 139.18, 136.32, 134.86, 133.73, 132.00, 130.14, 128.69, 128.52, 128.47, 127.09 (q, *J*<sub>CF</sub> = 274.6 Hz), 126.95, 79.97, 56.06, 33.39 (q, *J*<sub>CF</sub> = 28.3 Hz), 32.72, 24.20 (d, *J*<sub>CF</sub> = 2.8 Hz), 20.74.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -66.39 (s, 3F).

HRMS (ESI) *m/z* calcd. for C<sub>20</sub>H<sub>22</sub>NO<sub>2</sub>F<sub>3</sub> [M+Na]<sup>+</sup> 388.1500, found 388.1380.



***N*-(Methoxy(4-methyl-2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3m)**

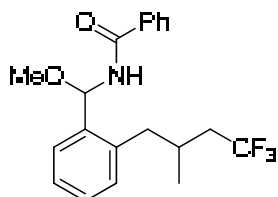
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 7.2 Hz, 2H), 7.52 (t, *J* = 8.4 Hz, 2H), 7.43 (t, *J* = 7.6 Hz,

2H), 7.02 (s, 1H), 6.55 (d,  $J = 9.2$  Hz, 1H), 6.47 (d,  $J = 9.2$  Hz, 1H), 3.51 (s, 3H), 2.78-2.66 (m, 2H), 2.35 (s, 3H), 2.20-2.07 (m, 2H), 1.91-1.74 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.89, 138.52, 138.43, 134.11, 133.50, 132.02, 130.38, 128.64, 127.56, 126.99 (q,  $J_{\text{CF}} = 274.5$  Hz), 126.98, 125.30, 78.96, 55.95, 33.44 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.88, 23.59 (d,  $J_{\text{CF}} = 2.8$  Hz), 21.07.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.28 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{22}\text{NO}_2\text{F}_3$   $[\text{M}+\text{Na}]^+$  388.1500, found 388.1492.



**3n** (dr = 1:1)

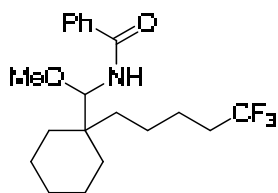
### ***N*-(Methoxy(2-(4,4,4-trifluoro-2-methylbutyl)phenyl)methyl)benzamide (3n)**

For two isomer:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77 (d,  $J = 7.2$  Hz, 4H), 7.67-7.64 (m, 2H), 7.51 (t,  $J = 7.6$  Hz, 2H), 7.42 (t,  $J = 8.0$  Hz, 4H), 7.34-7.28 (m, 4H), 7.20-7.17 (m, 2H), 6.62 (s, 2H), 6.52 (d,  $J = 5.6$  Hz, 1H), 6.50 (d,  $J = 5.6$  Hz, 1H), 3.51 (s, 3H), 3.51 (s, 3H), 2.83-2.57 (m, 4H), 2.30-2.10 (m, 4H), 2.04-1.88 (m, 2H), 1.08 (d,  $J = 6.4$  Hz, 3H), 0.51 (d,  $J = 6.0$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.03, 166.95, 137.57, 137.32 (d,  $J = 9.1$  Hz), 133.55, 133.47, 131.98, 131.95, 130.45, 130.38, 128.59, 128.52, 128.48, 127.01 (q,  $J_{\text{CF}} = 275.5$  Hz), 126.99 (q,  $J_{\text{CF}} = 275.8$  Hz), 126.98 (d,  $J = 2.0$  Hz), 126.94, 125.35, 79.03, 78.86, 55.93, 55.80, 39.57 (q,  $J_{\text{CF}} = 27$  Hz), 39.09 (q,  $J_{\text{CF}} = 34.8$  Hz), 29.63 (d,  $J_{\text{CF}} = 2.1$  Hz), 29.43 (d,  $J_{\text{CF}} = 2.2$  Hz) 19.38, 19.36.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.96 (t,  $J = 10.9$  Hz, 3F), -63.05 (t,  $J = 10.5$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  388.1500, found 388.1495.



**3w**

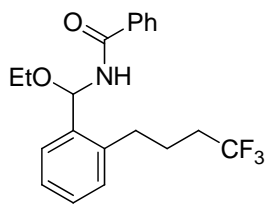
### ***N*-(Methoxy(1-(5,5,5-trifluoropentyl)cyclohexyl)methyl)benzamide (3w)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80-7.82 (m, 2H), 7.56-7.52 (m, 1H), 7.47 (t,  $J = 7.6$  Hz, 2H), 6.27 (d,  $J = 10.0$  Hz, 1H), 5.24 (d,  $J = 10.0$  Hz, 1H), 3.36 (s, 3H), 2.16-2.03 (m, 2H), 1.63-1.25 (m, 16H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.68, 134.13, 131.85, 128.73, 127.19 (q,  $J_{\text{CF}} = 274.6$  Hz), 126.86, 85.33, 56.41, 40.38, 33.61 (q,  $J_{\text{CF}} = 28.1$  Hz), 31.97, 30.177 (d,  $J = 49.8$  Hz), 26.02, 22.87 (q,  $J_{\text{CF}} = 2.6$  Hz), 22.45, 21.19 (d,  $J_{\text{CF}} = 9.9$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.37 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{28}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  394.1970, found 394.1946.



**3o**

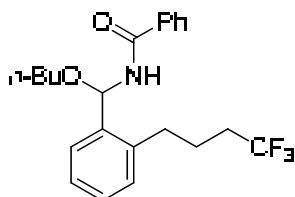
***N*-(Ethoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3o)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.78 (d,  $J = 7.2$  Hz, 2H), 7.71-7.67 (m, 1H), 7.51 (t,  $J = 7.2$  Hz, 1H), 7.43 (t,  $J = 7.6$  Hz, 2H), 7.34-7.27 (m, 2H), 7.23-7.19 (m, 1H), 6.62 (s, 2H), 3.87-3.80 (m, 1H), 3.72-3.64 (m, 1H), 2.84-2.70 (m, 2H), 2.20-2.07 (m, 2H), 1.93-1.76 (m, 2H), 1.29 (t,  $J = 6.8$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.75, 138.50, 137.41, 133.48, 132.00, 129.61, 128.63, 126.95 (q,  $J_{\text{CF}} = 274.7$  Hz), 126.98, 126.84, 125.57, 125.49, 77.43, 63.91, 33.42 (q,  $J_{\text{CF}} = 28.5$  Hz), 30.88, 23.50 (d,  $J_{\text{CF}} = 2.8$  Hz), 15.08.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.30 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  388.1500, found 388.1497.



**3p**

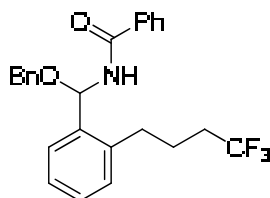
***N*-(Butoxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3p)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77 (d,  $J = 7.2$  Hz, 2H), 7.70-7.69 (m, 1H), 7.53-7.50 (m, 1H), 7.45-7.41 (m, 2H), 7.33-7.28 (m, 2H), 7.20 (d,  $J = 7.2$  Hz, 1H), 6.61-6.55 (m, 2H), 3.80-3.74 (m, 1H), 3.66-3.60 (m, 1H), 2.83-2.69 (m, 2H), 2.17-2.11 (m, 2H), 1.89-1.79 (m, 2H), 1.65 (dt,  $J = 14.0, 6.8$  Hz, 2H), 1.47-1.38 (m, 2H), 0.93 (td,  $J = 7.2, 0.8$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.73, 138.49, 137.49, 133.52, 132.02, 129.59, 128.65, 128.62, 126.98, 126.94 (q,  $J_{\text{CF}} = 274.9$  Hz), 126.83, 125.53, 77.58, 68.31, 33.44 (q,  $J_{\text{CF}} = 28.4$  Hz), 31.73, 30.86, 23.51 (d,  $J = 2.8$  Hz), 19.37, 13.86.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.33 (s, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{22}\text{H}_{26}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  416.1813, found 416.1807.



**3q**

***N*-(Benzyloxy(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3q)**

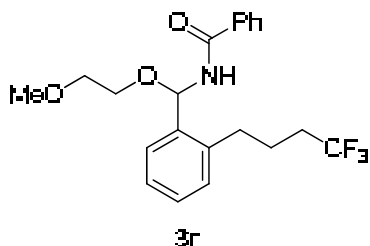
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80-7.76 (m, 3H), 7.55-7.51 (m, 1H), 7.45-7.42 (m, 4H), 7.38-7.29 (m, 5H), 7.23-7.20 (m, 1H), 6.76 (dd,  $J = 9.6, 2.4$  Hz, 1H), 6.71 (d,  $J = 9.6$  Hz, 1H), 4.80 (dd,  $J =$

31.6, 12.0 Hz, 2H), 2.78-2.63 (m, 2H), 2.13-1.96 (m, 2H), 1.82-1.73 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.86, 138.47, 137.69, 137.15, 133.39, 132.06, 129.59, 128.70, 128.63, 128.37, 127.86, 127.74, 126.99, 126.90 (q,  $J_{\text{CF}} = 274.6$  Hz), 126.84, 125.60, 77.10, 70.29, 33.30 (q,  $J_{\text{CF}} = 28.3$  Hz), 30.78, 23.40 (d,  $J_{\text{CF}} = 2.7$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.18 (t,  $J = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{25}\text{H}_{24}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  450.1657, found 450.1650.



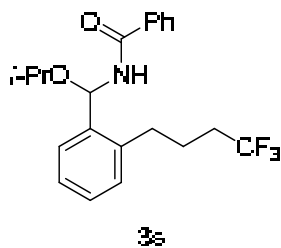
***N*-((2-Methoxyethoxy)(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3r)**

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.79-7.77 (m, 2H), 7.71-7.69 (m, 1H), 7.52-7.49 (m, 1H), 7.43-7.40 (m, 2H), 7.32-7.27 (m, 2H), 7.20-7.18 (m, 1H), 6.83 (t,  $J = 8.8$  Hz, 1H), 6.63 (d,  $J = 9.2$  Hz, 1H), 3.94-3.79 (m, 2H), 3.66-3.56 (m, 2H), 3.37 (s, 3H), 2.83-2.69 (m, 2H), 2.20-2.08 (m, 2H), 1.93-1.76 (m, 2H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.78, 138.51, 137.01, 133.39, 131.98, 129.53, 128.65, 128.57, 127.00, 126.96 (q,  $J_{\text{CF}} = 274.8$  Hz), 126.75, 125.72, 78.20, 71.66, 67.62, 58.89, 33.37 (q,  $J_{\text{CF}} = 28.3$  Hz), 30.84, 23.42 (d,  $J_{\text{CF}} = 2.8$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.25 (t,  $J = 10.5$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{21}\text{H}_{24}\text{F}_3\text{NO}_3$   $[\text{M}+\text{Na}]^+$  418.1606, found 418.1600.



***N*-((Isopropoxy)(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3s)**

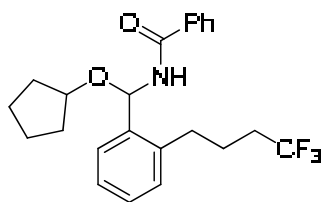
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77-7.70 (m, 3H), 7.53-7.49 (m, 1H), 7.44-7.41 (m, 2H), 7.32-7.28 (m, 2H), 7.22-7.18 (m, 1H), 6.72 (d,  $J = 9.2$  Hz, 1H), 6.58 (d,  $J = 9.2$  Hz, 1H), 4.00 (tt,  $J = 12.4$ , 6.0 Hz, 1H), 2.86-2.70 (m, 2H), 2.22-2.07 (m, 2H), 1.93-1.76 (m, 2H), 1.29 (dd,  $J = 9.2$ , 6.4 Hz, 6H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.56, 138.33, 137.99, 133.53, 131.97, 129.58, 128.62, 128.53, 126.97, 126.94 (q,  $J_{\text{CF}} = 274.6$  Hz), 126.84, 125.76, 75.12, 69.24, 33.43 (q,  $J_{\text{CF}} = 28.4$  Hz), 30.91, 23.57 (d,  $J = 2.8$  Hz), 23.01, 21.77.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.32 (t,  $J_{\text{CF}} = 10.9$  Hz, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{21}\text{H}_{24}\text{F}_3\text{NO}_2$   $[\text{M}+\text{Na}]^+$  402.1657, found 402.1650.





**3t**

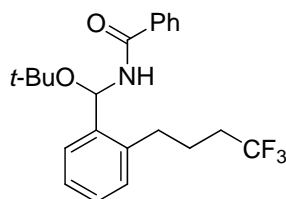
***N*-((Cyclopentyloxy)(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3t)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 6.8 Hz, 2H), 7.71 (d, *J* = 4.0 Hz, 1H), 7.52-7.41 (m, 3H), 7.30 (d, *J* = 2.4 Hz, 2H), 7.20 (d, *J* = 2.0 Hz, 1H), 6.66 (d, *J* = 9.2 Hz, 1H), 6.56 (d, *J* = 8.8 Hz, 1H), 4.29 (s, 1H), 2.84-2.69 (m, 2H), 2.17-2.12 (m, 2H), 1.85-1.67 (m, 8H), 1.55 (s, 2H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.51, 138.40, 137.91, 133.59, 131.94, 129.53, 128.61, 128.50, 126.97, 126.95 (q, *J*<sub>CF</sub> = 274.8 Hz), 126.79, 125.84, 79.17, 76.04, 33.44 (q, *J*<sub>CF</sub> = 28.3 Hz), 32.94, 32.26, 30.87, 23.54 (dd, *J*<sub>CF</sub> = 13.6, 10.8 Hz).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -66.32 (t, *J* = 10.5 Hz, 3F).

HRMS (ESI) *m/z* calcd. for C<sub>23</sub>H<sub>26</sub>F<sub>3</sub>NO<sub>2</sub>[M+Na]<sup>+</sup> 428.1813, found 428.1806.



**3u**

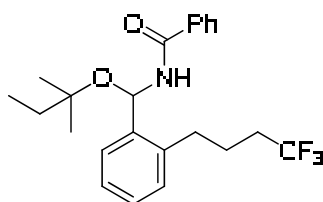
***N*-((Tert-butoxy)(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3u)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.77-7.72 (m, 3H), 7.44 (dt, *J* = 32.4, 6.8 Hz, 3H), 7.27 (dd, *J* = 4.4, 2.4 Hz, 2H), 7.17 (s, 1H), 6.79 (d, *J* = 9.2 Hz, 1H), 6.55 (d, *J* = 8.8 Hz, 1H), 2.86-2.71 (m, 2H), 2.18-2.11 (m, 2H), 1.91-1.80 (m, 2H), 1.31 (s, 9H).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.28, 139.85, 137.85, 133.97, 131.80, 129.49, 128.59, 128.16, 126.99 (q, *J*<sub>CF</sub> = 274.5 Hz), 126.91, 126.63, 126.13, 75.30, 72.13, 33.54 (q, *J*<sub>CF</sub> = 28.4 Hz), 30.86, 28.58, 23.56 (d, *J*<sub>CF</sub> = 2.8 Hz).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -66.34 (t, *J* = 10.9 Hz, 3F).

HRMS (ESI) *m/z* calcd. for C<sub>22</sub>H<sub>26</sub>F<sub>3</sub>NO<sub>2</sub> [M+Na]<sup>+</sup> 416.1813, found 416.1808.



**3v**

***N*-((Tert-pentyloxy)(2-(4,4,4-trifluorobutyl)phenyl)methyl)benzamide (3v)**

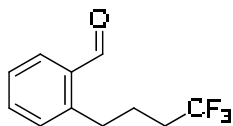
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.80-7.77 (m, 1H), 7.73-7.71 (m, 2H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 2H), 7.29-7.24 (m, 2H), 7.17-7.15 (m, 1H), 6.79 (d, *J* = 9.6 Hz, 1H), 6.58 (s, 1H), 2.87-2.71 (m, 2H), 2.22-2.10 (m, 2H), 1.91-1.745 (m, 2H), 1.73-1.57 (m, 2H), 1.28 (s, 3H), 1.24 (s,

3H), 0.88 (t,  $J_{CF} = 7.6$  Hz, 3H).

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.22, 139.93, 137.81, 134.01, 131.75, 129.46, 128.57, 128.08, 127.00 (q,  $J_{CF} = 274.5$  Hz), 126.58, 126.13, 125.62, 77.65, 71.70, 34.08, 33.52 (q,  $J_{CF} = 28.4$  Hz), 30.84, 25.97, 25.54, 23.58 (d,  $J = 2.8$  Hz), 8.54.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.23 (s, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{23}\text{H}_{28}\text{NO}_2\text{F}_3$   $[\text{M}+\text{Na}]^+$  430.1970, found 430.1964.



5

### 2-(4,4,4-Trifluorobutyl)benzaldehyde (5)

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  10.18 (s, 1H), 7.82 (dd,  $J = 7.6, 1.2$  Hz, 1H), 7.53 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.43 (td,  $J = 7.6, 1.2$  Hz, 1H), 7.28 (d,  $J = 7.6$  Hz, 1H), 3.11 (t,  $J = 7.6$  Hz, 2H), 2.23-2.10 (m, 2H), 1.91-1.83 (m, 2H).

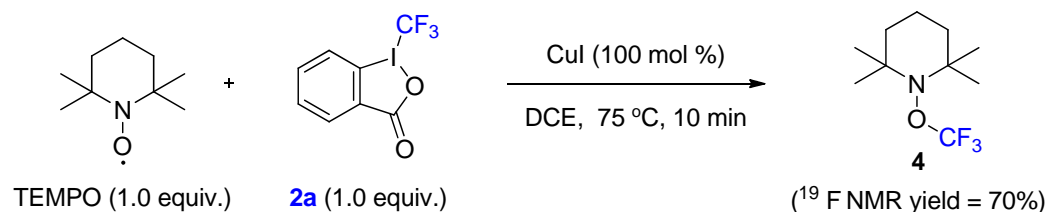
$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  192.81, 143.28, 134.05, 133.82, 133.70, 131.06, 127.04 (q,  $J_{CF} = 274.7$  Hz), 127.02, 33.34 (q,  $J_{CF} = 28.4$  Hz), 31.68, 23.78 (d,  $J_{CF} = 2.9$  Hz).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -66.17 (s, 3F).

HRMS (ESI)  $m/z$  calcd. for  $\text{C}_{11}\text{H}_{11}\text{F}_3\text{O}$   $[\text{M}-\text{H}]^-$  215.0684, found 215.0690.

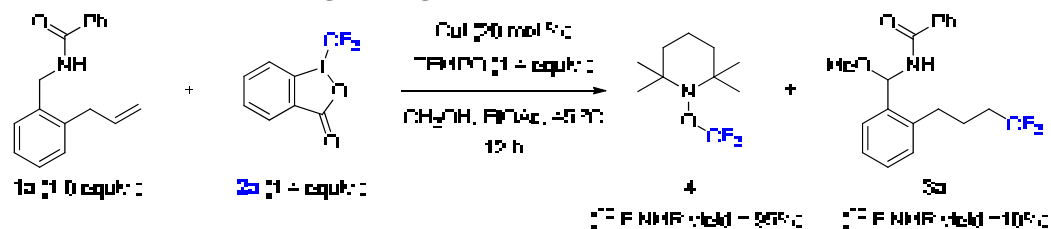
### Control experiments:

#### Reaction of TEMPO with Togni's reagent 2a



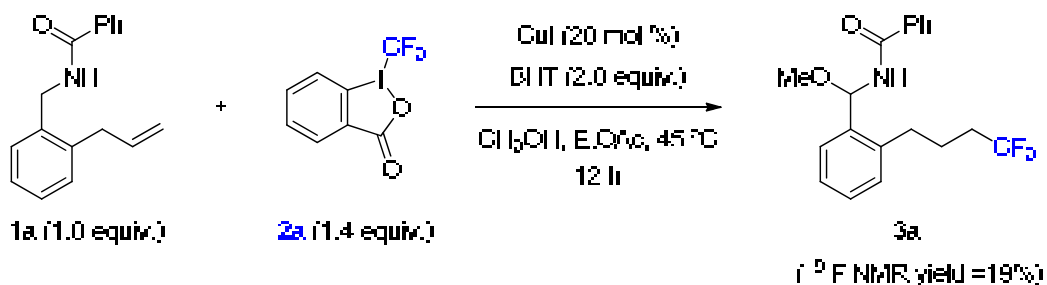
**Procedure:** To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **2a** (62.2 mg, 0.2 mmol), CuI (38.1 mg, 0.2 mmol), and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), 31.3 mg, 0.2 mmol). The tube was evacuated and backfilled with Ar for three times, and then solvent (DCE, 1.0 mL) was added *via* syringe. The sealed tube was stirred at 45 °C for 10 min, and then cooled to room temperature.  $\alpha,\alpha,\alpha$ -Trifluorotoluene (internal standard, 29.2 mg, 0.2 mmol) was added.  $^{19}\text{F}$  NMR analysis of this reaction mixture showed that TEMPO- $\text{CF}_3$  was formed in 70% yield.

#### Reaction of TEMPO and Togni's reagent 2a with 1a



**Procedure:** To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1a** (50.2 mg, 0.2 mmol), **2a** (88.5 mg, 0.28 mmol), CuI (7.6 mg, 0.04 mmol), and TEMPO (35.4 mg, 0.28 mmol). The tube was evacuated and backfilled with Ar for three times, and then alcohol (32.5  $\mu$ L, 0.8 mmol) and EtOAc (1.0 mL) were added *via* syringe. The sealed tube was stirred at 45  $^{\circ}$ C for 12 h, and then cooled to room temperature.  $\alpha,\alpha,\alpha$ -Trifluorotoluene (internal standard, 29.2 mg, 0.2 mmol) was added.  $^{19}$ F NMR analysis of this reaction mixture showed that TEMPO-CF<sub>3</sub> was formed in 95% yield and **3a** in 10% yield.

#### Reaction of BHT and Togni's reagent 2a with 1a

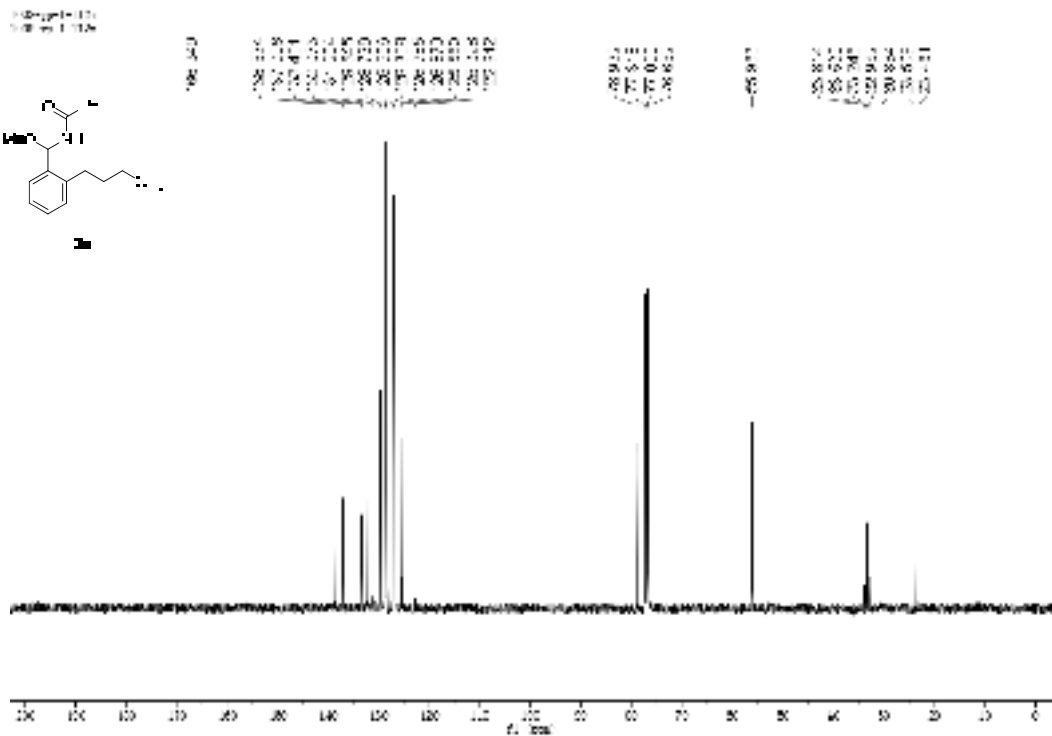
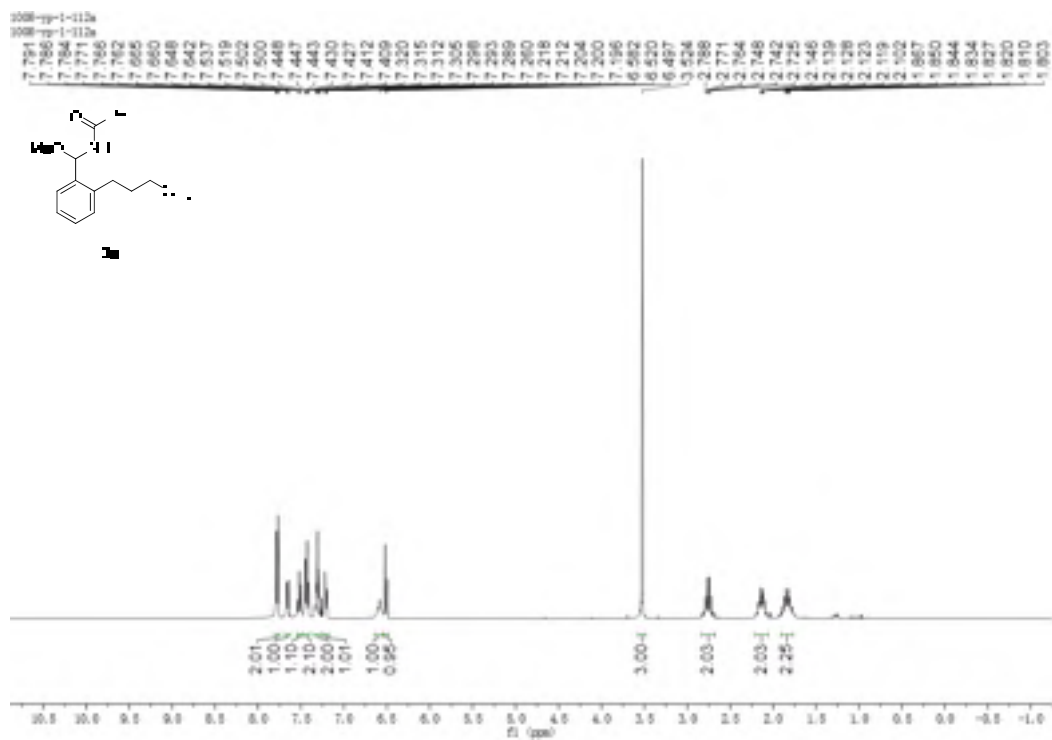


**Procedure:** To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1a** (25.1 mg, 0.1 mmol), **2a** (44.2 mg, 0.14 mmol), CuI (3.8 mg, 0.02 mmol) and BHT (41.3 mg, 0.20 mmol). The tube was evacuated and backfilled with Ar for three times, and then alcohol (32.5  $\mu$ L, 0.8 mmol) and EtOAc (1.0 mL) were added *via* syringe. The sealed tube was stirred at 45  $^{\circ}$ C for 12 h, and then cooled to room temperature.  $\alpha,\alpha,\alpha$ -Trifluorotoluene (29.2 mg, 0.2 mmol) was added as internal standard.  $^{19}$ F NMR analysis of this reaction mixture showed that **3a** was formed in 19% yield.

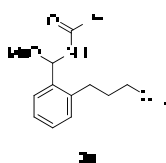
#### References

- S1) Julian, L. D.; Hartwig, J. F. *J. Am. Chem. Soc.* **2010**, *132*, 13813.  
S2) Bender, C. F.; Widenhoefer, R. A. *J. Am. Chem. Soc.* **2005**, *127*, 1070.

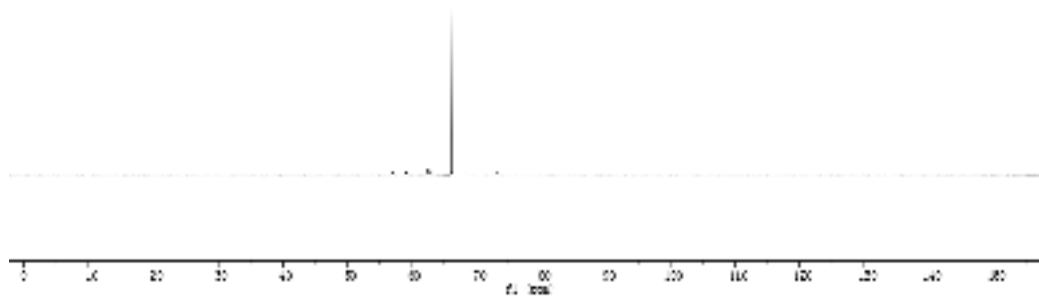
# NMR spectra



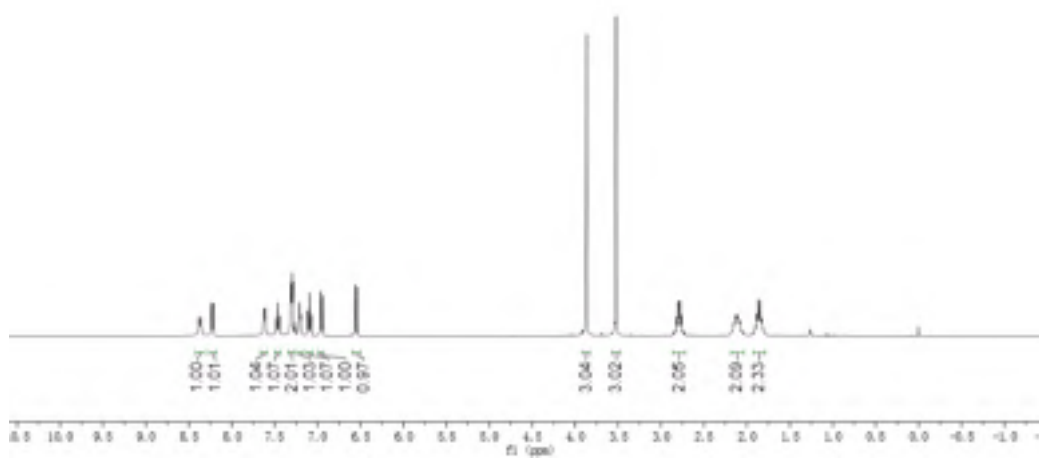
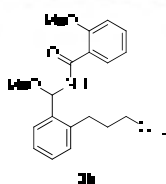
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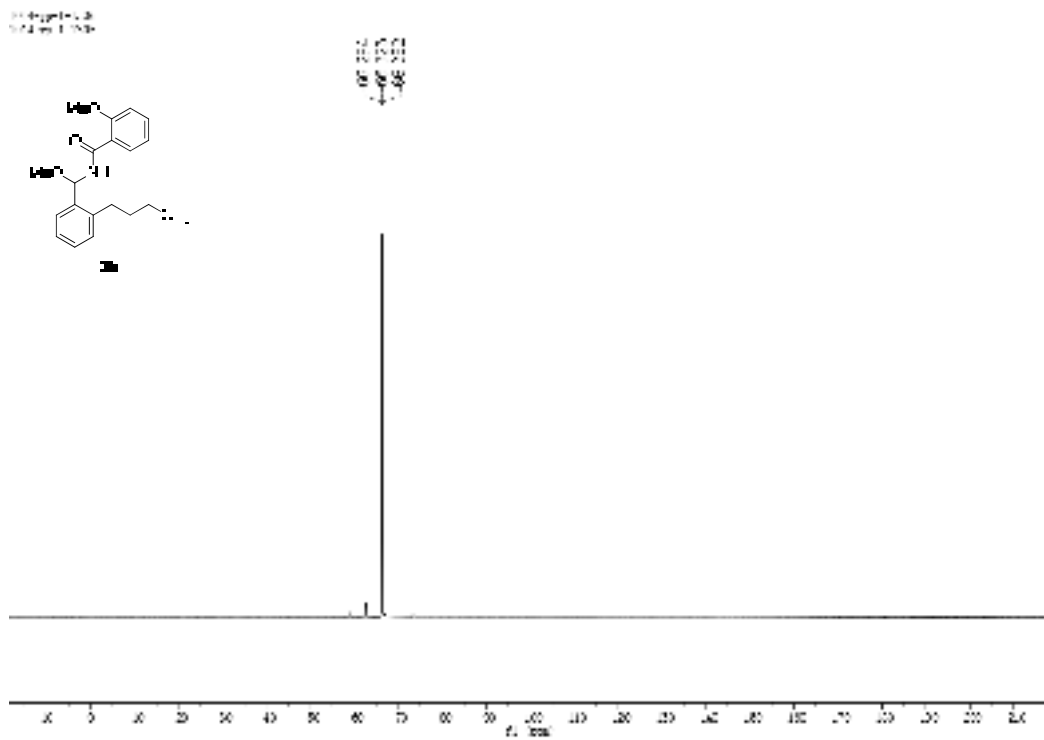
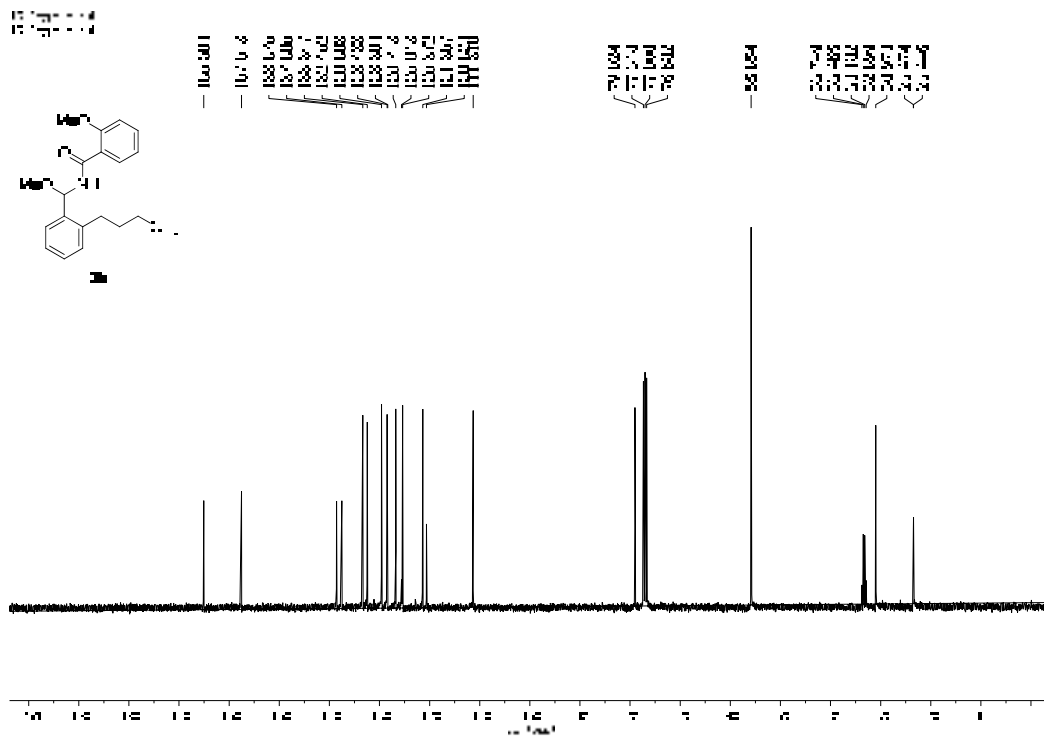


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10:27:13  
10:27:13



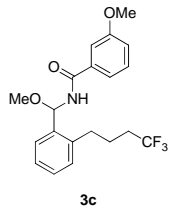
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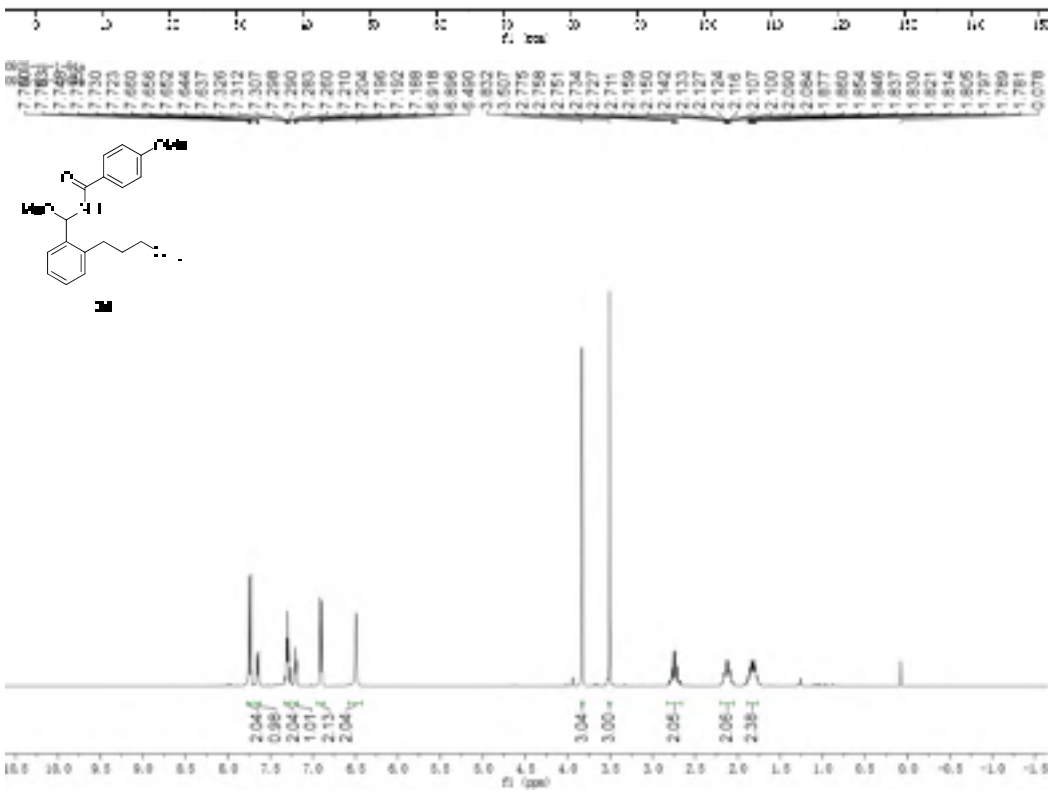




1H NMR (400 MHz, CDCl3)



1H NMR

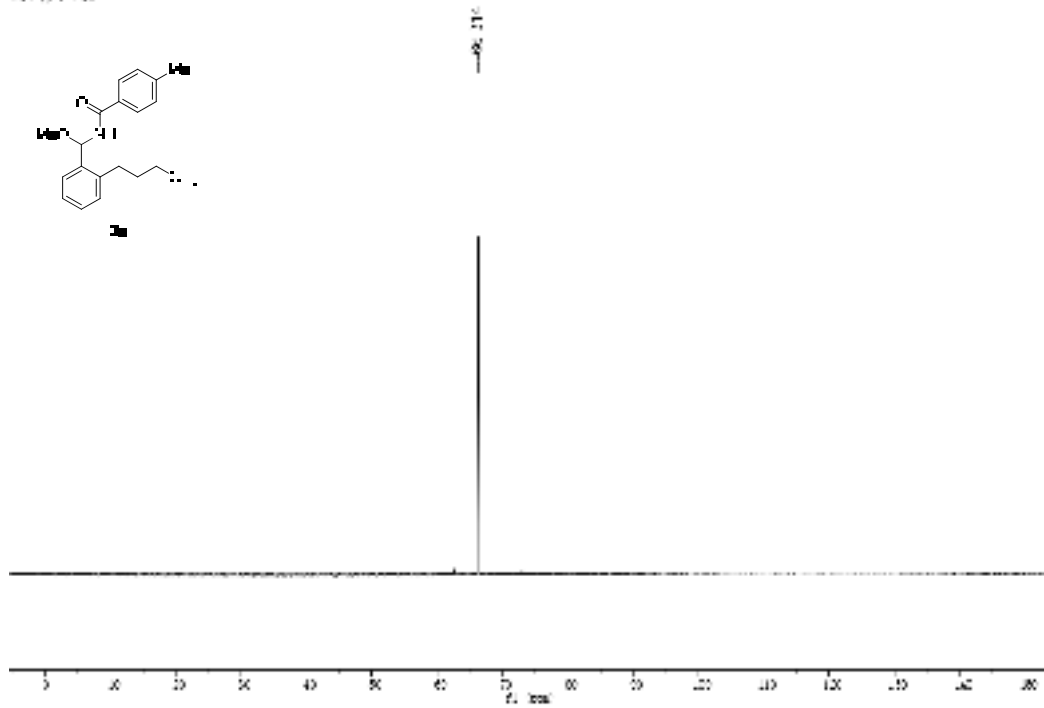
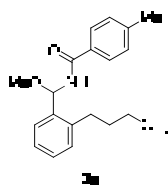








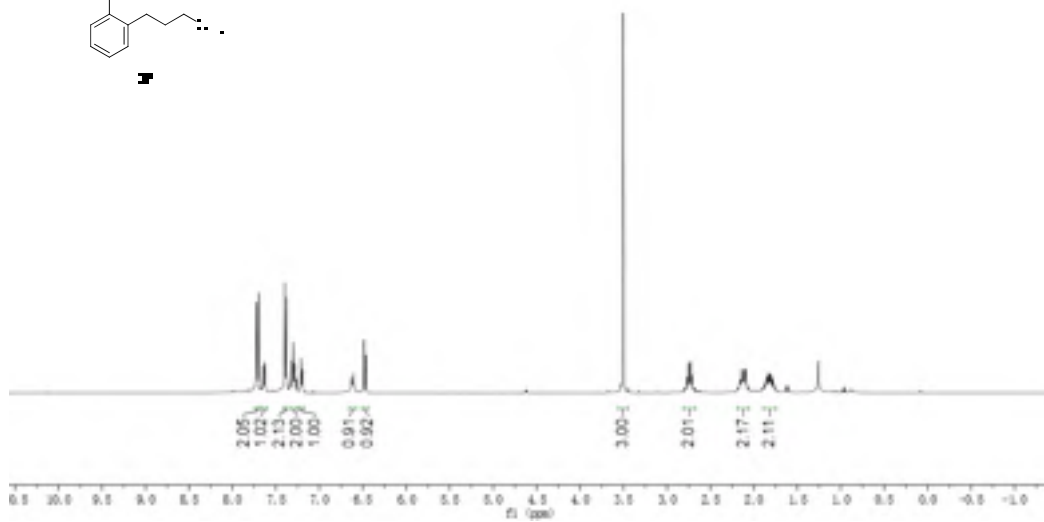
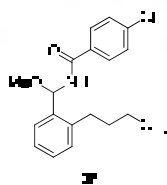
1H NMR (400 MHz, CDCl3)  
1.287 (s, 3H)



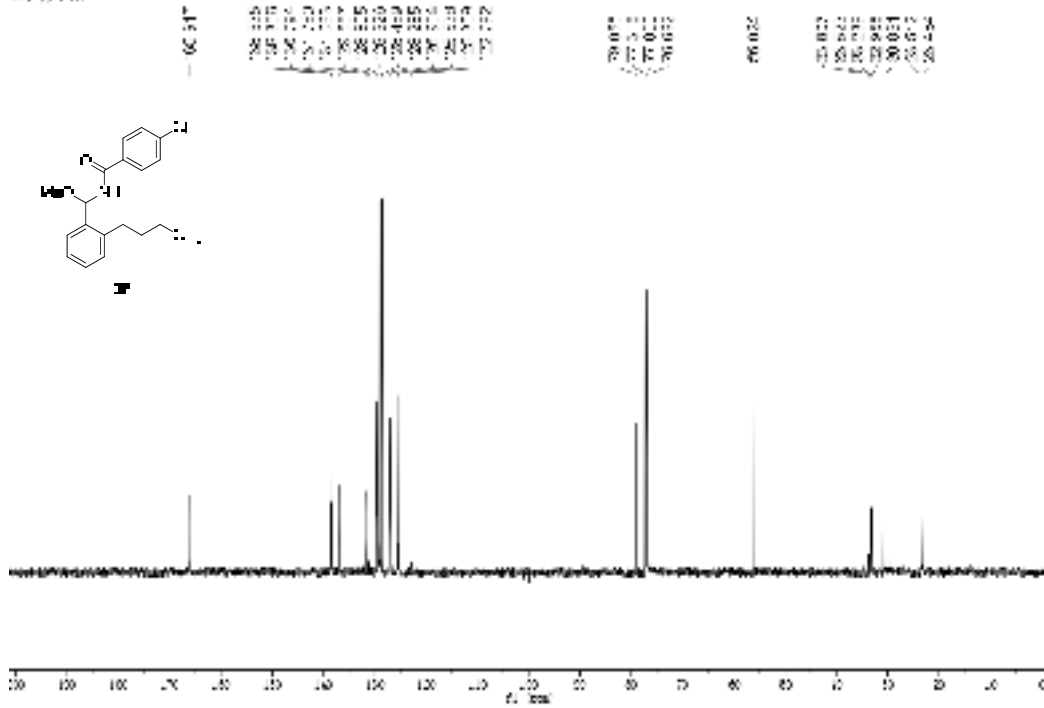
13C NMR (100 MHz, CDCl3)



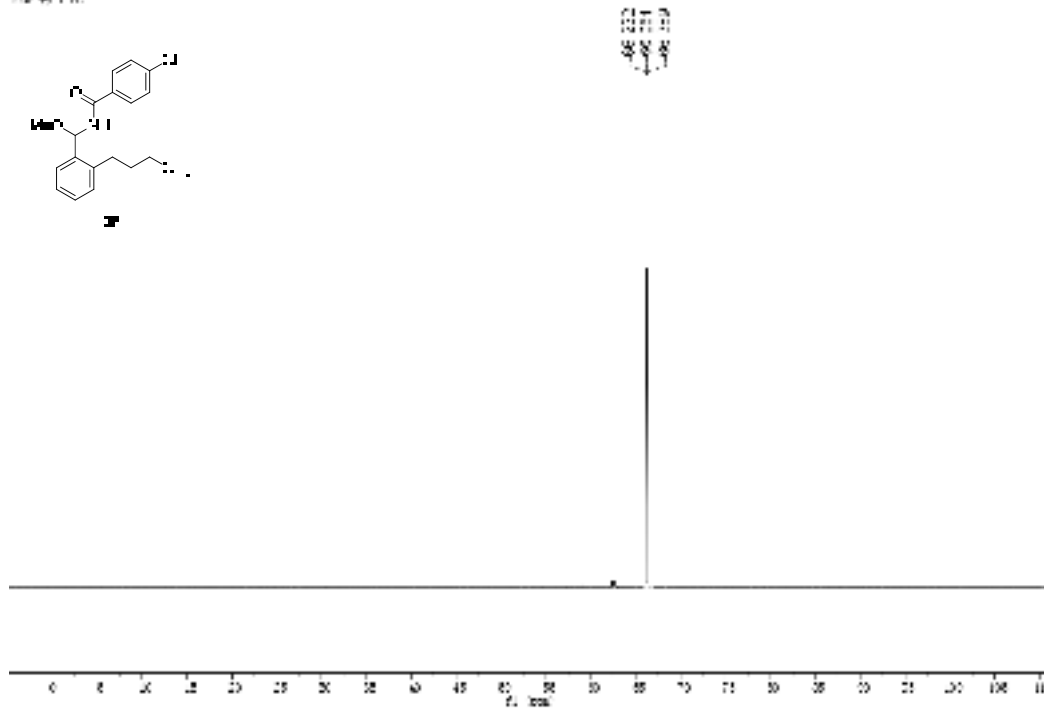
1H NMR (400 MHz, CDCl3)



13C NMR (101 MHz, CDCl<sub>3</sub>)

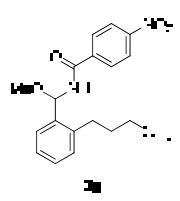


13C NMR (101 MHz, CDCl<sub>3</sub>)

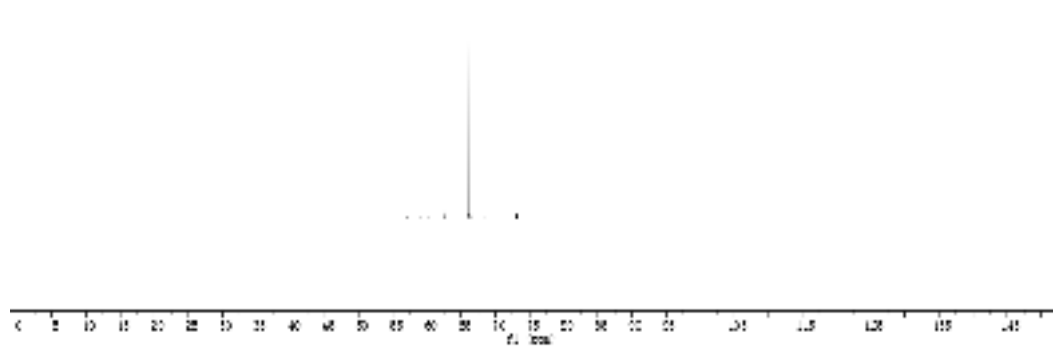




D:\31\p144a  
 P144a 1.70a



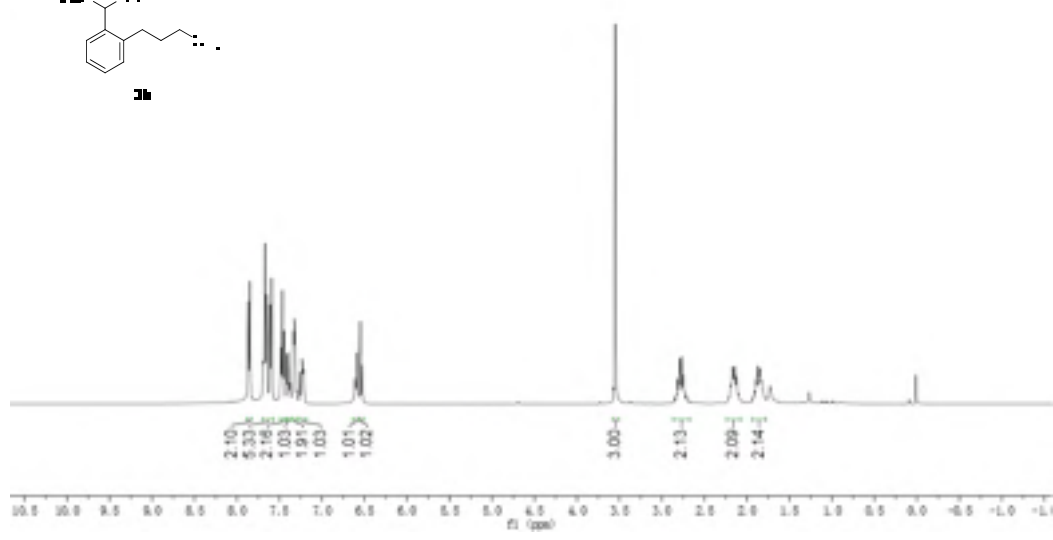
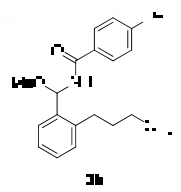
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 7.334  
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308-1-118  
 308-1-118

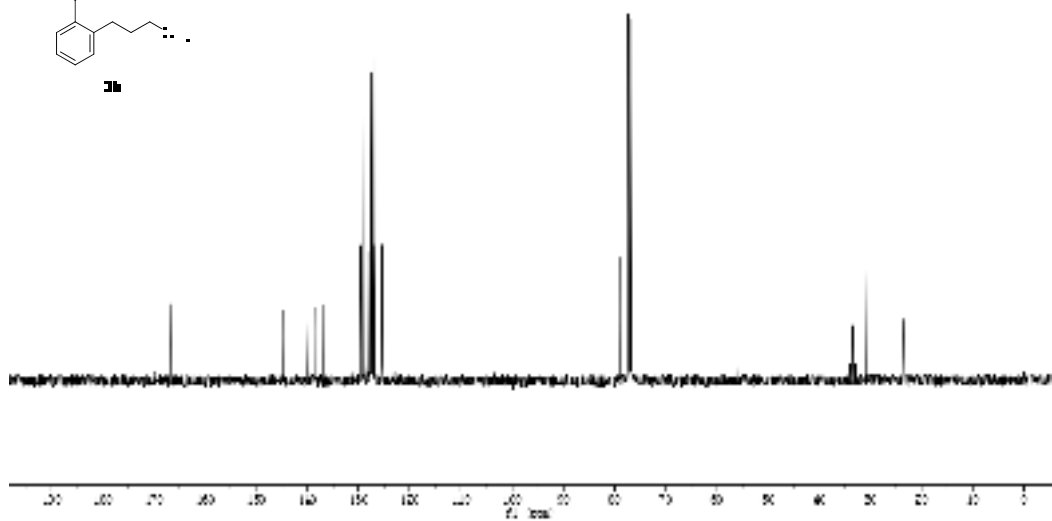
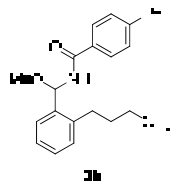
7.869 7.852 7.837 7.649 7.612 7.593 7.484 7.465 7.446 7.411 7.354 7.334 7.325 7.315 7.235 6.892 6.880 6.851 6.827

3.551 2.812 2.788 2.771 2.748 2.753 2.768 2.744 2.723 2.697 1.911 1.889 1.872 1.848 1.831 0.819

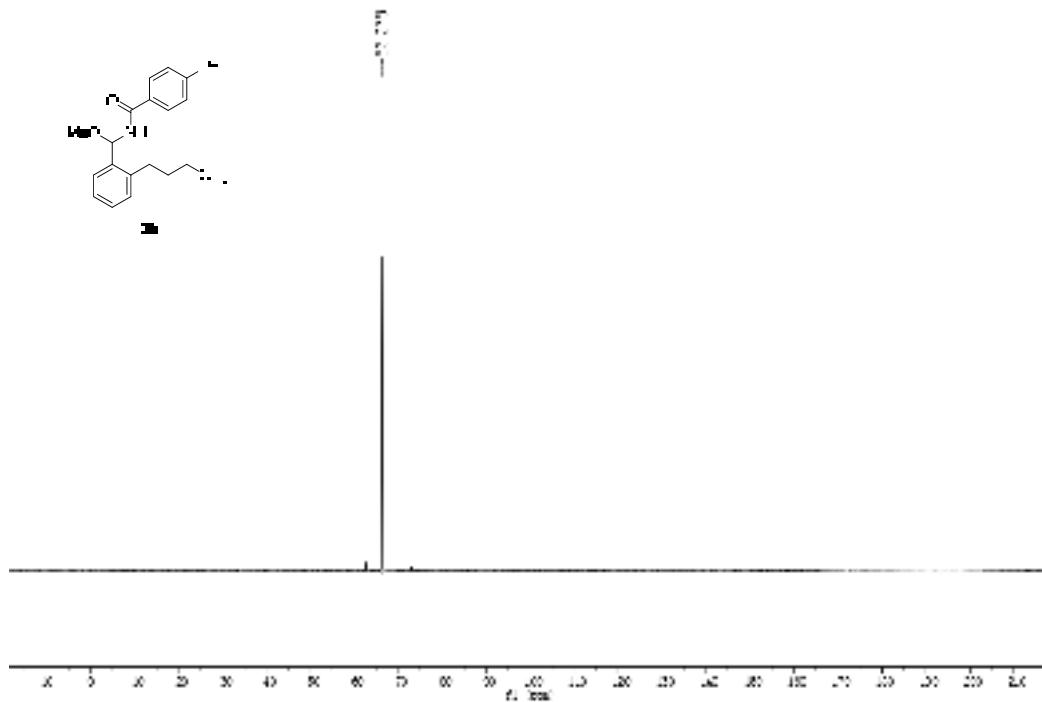
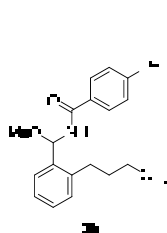


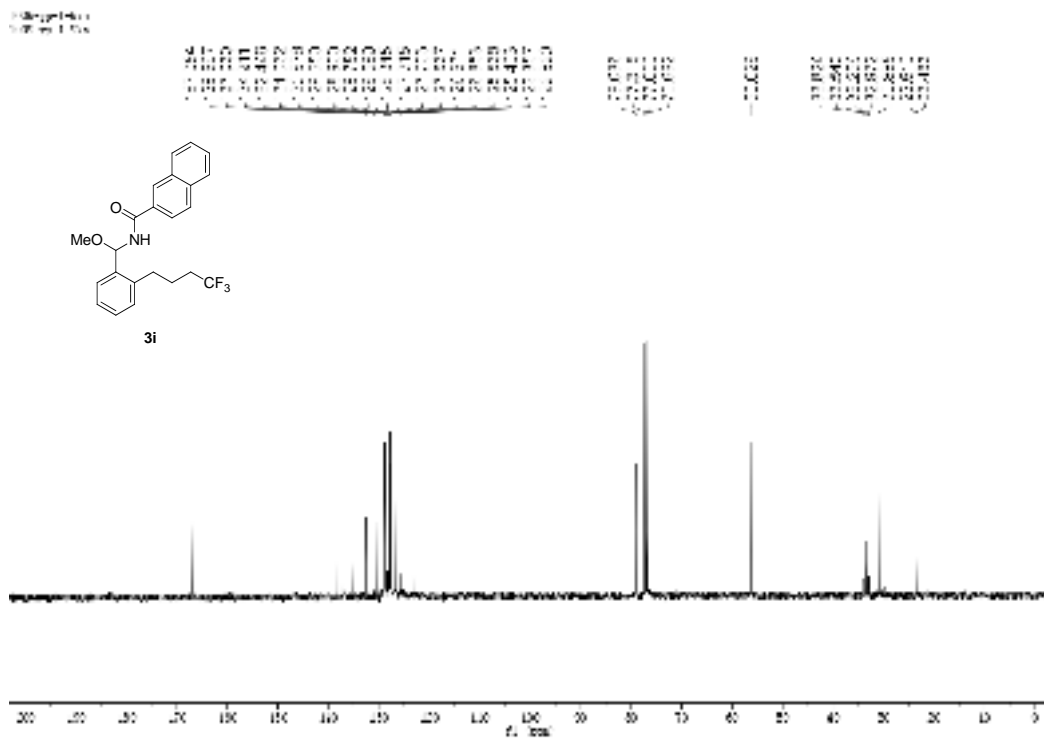
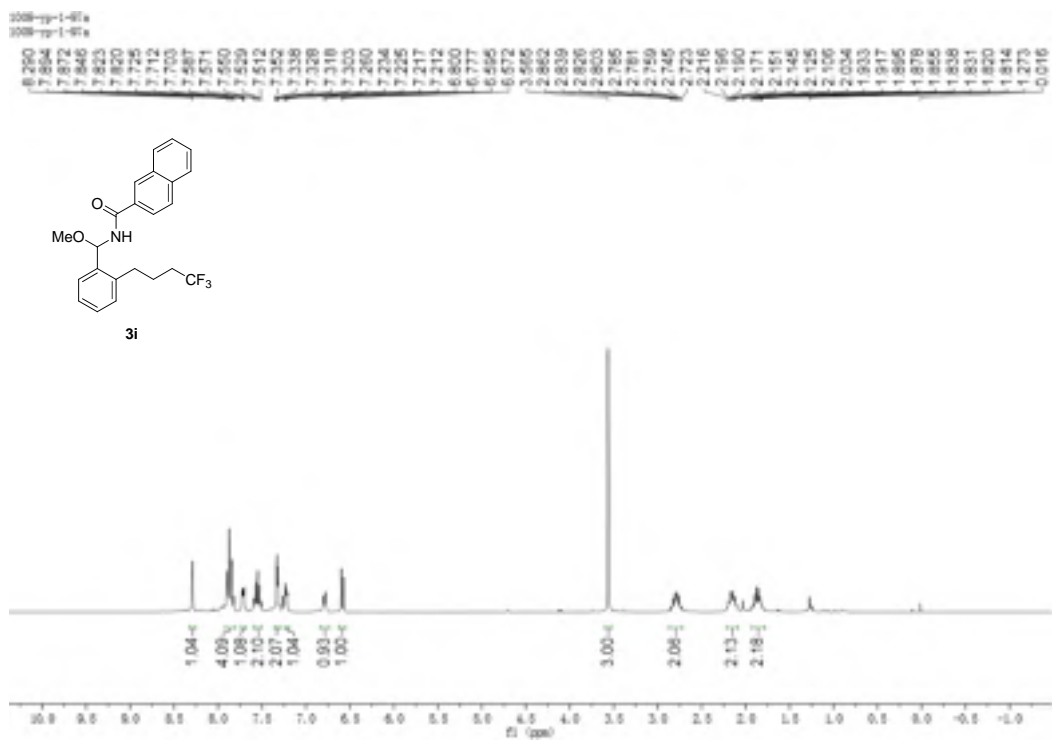
1H NMR (400 MHz, CDCl<sub>3</sub>)

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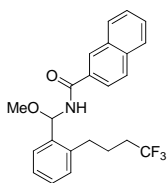
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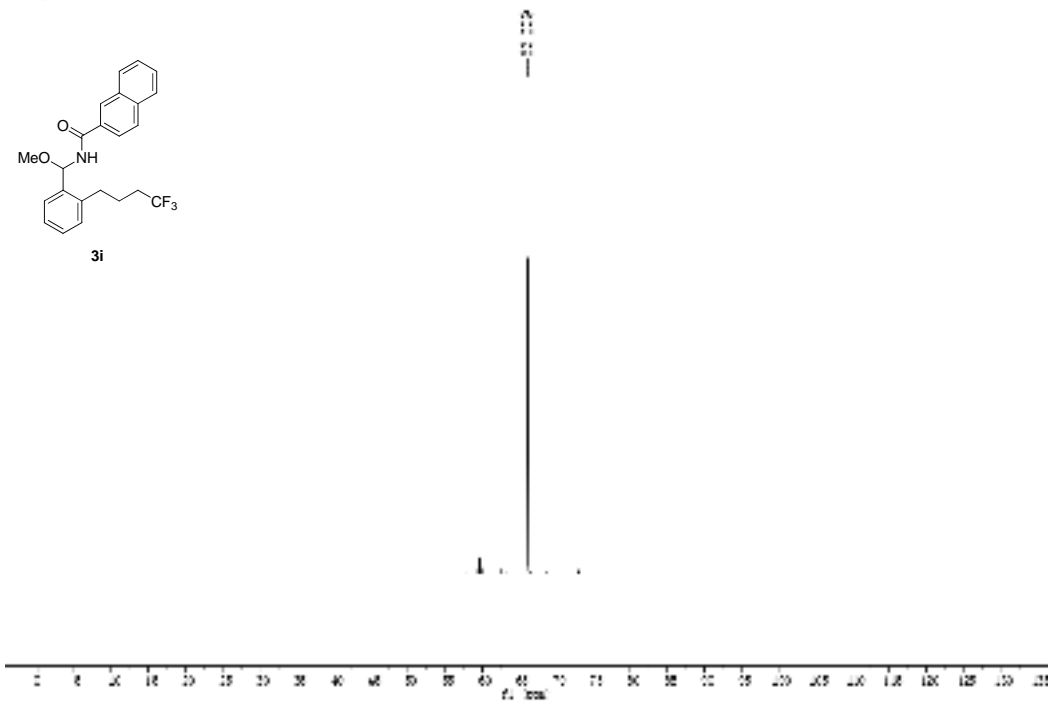




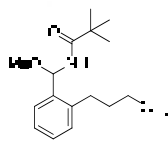
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100% MeOH



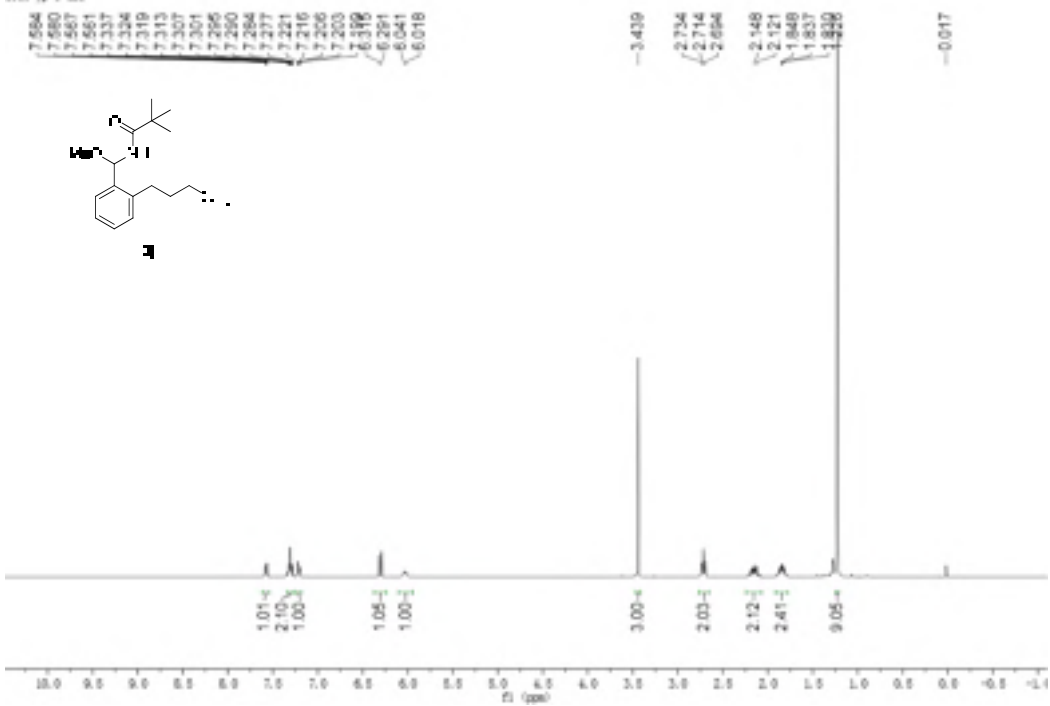
3i



100% MeOH  
100% MeOH



3j



1H NMR (400 MHz, CDCl3)

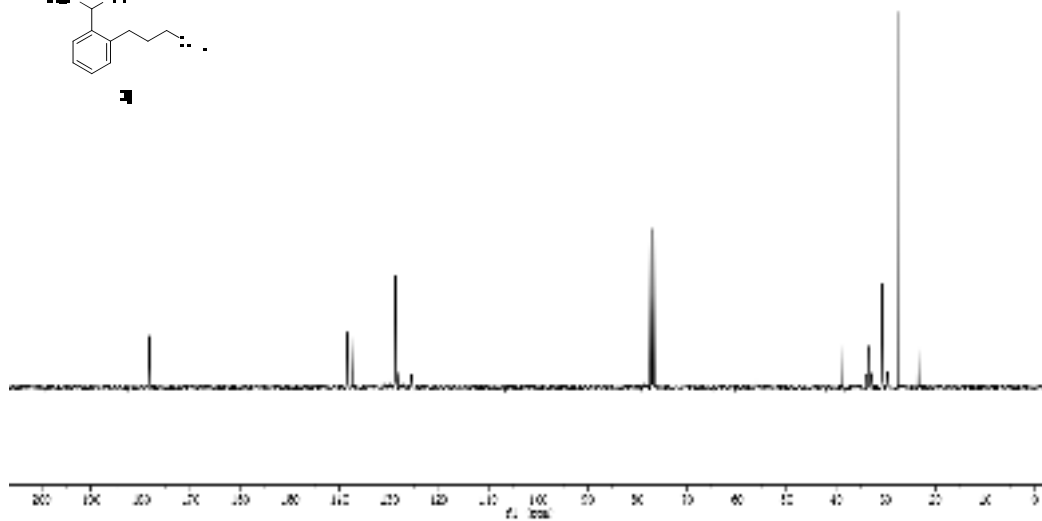
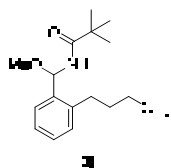
1.12 (s, 9H)

1.25 (t, 3H)

1.45 (s, 3H)

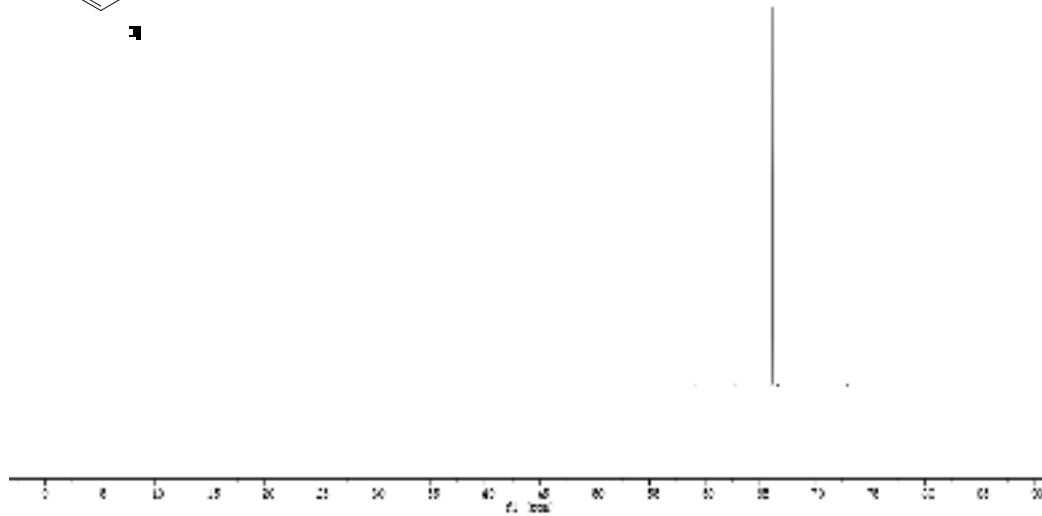
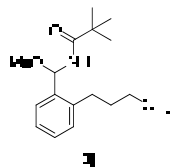
1.65 (s, 3H)

1.75 (s, 3H)



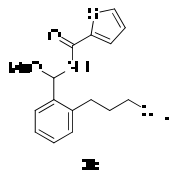
13C NMR (100 MHz, CDCl3)

16.5

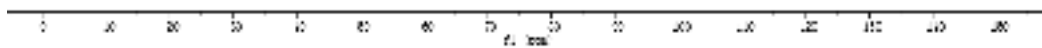




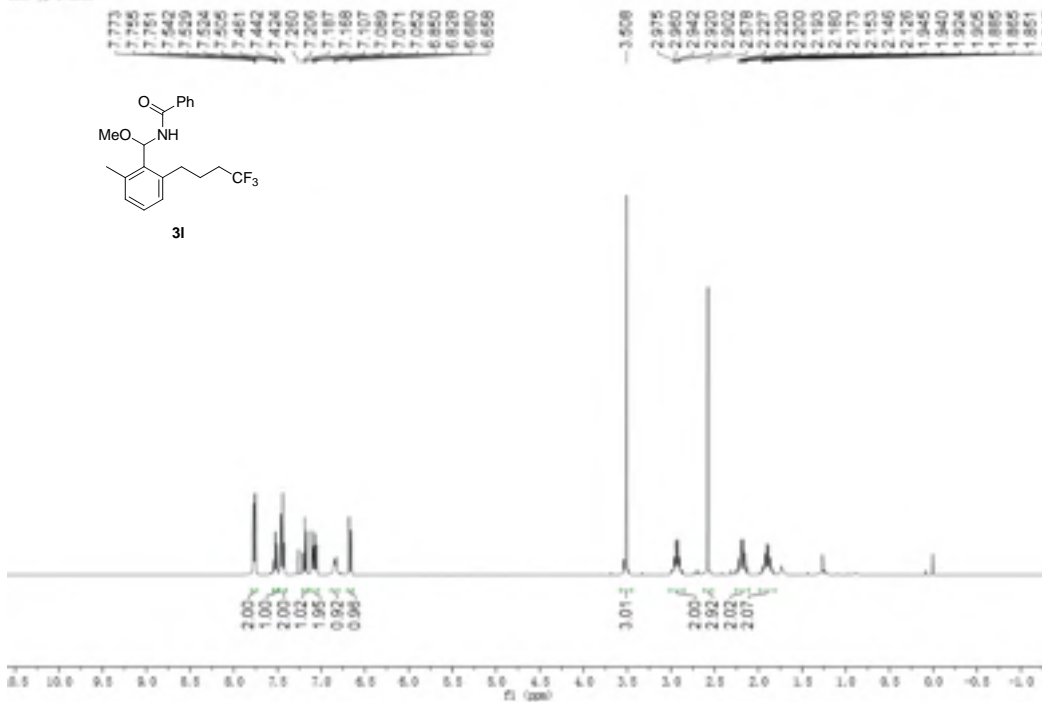
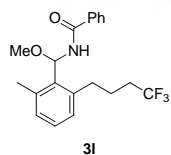
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13 Me



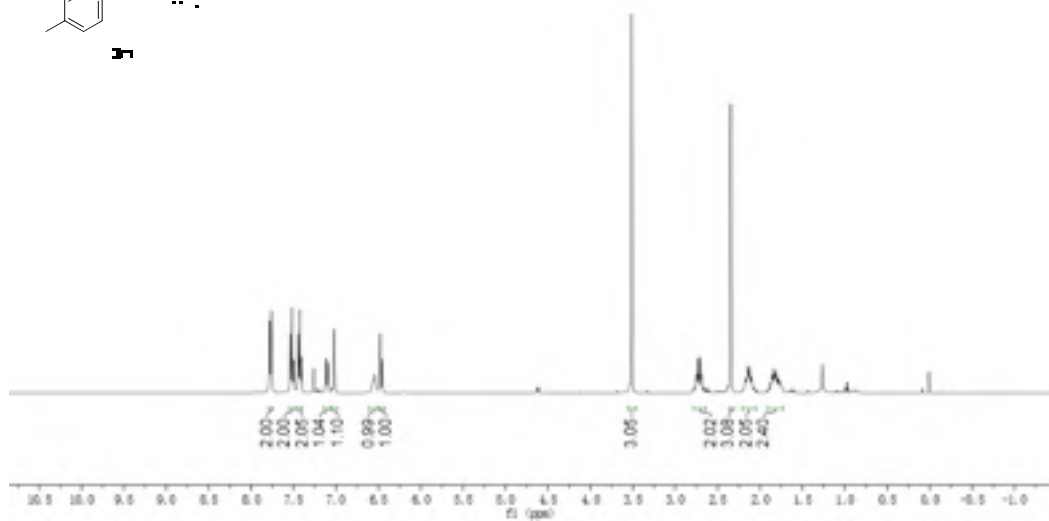
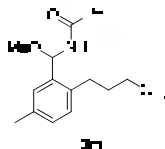
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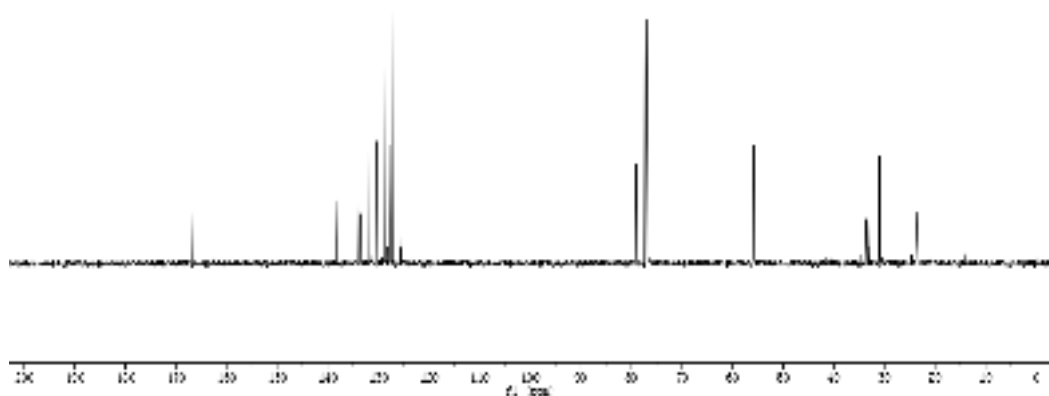
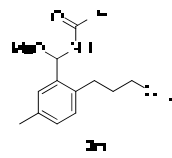
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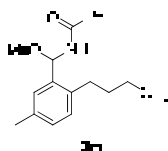


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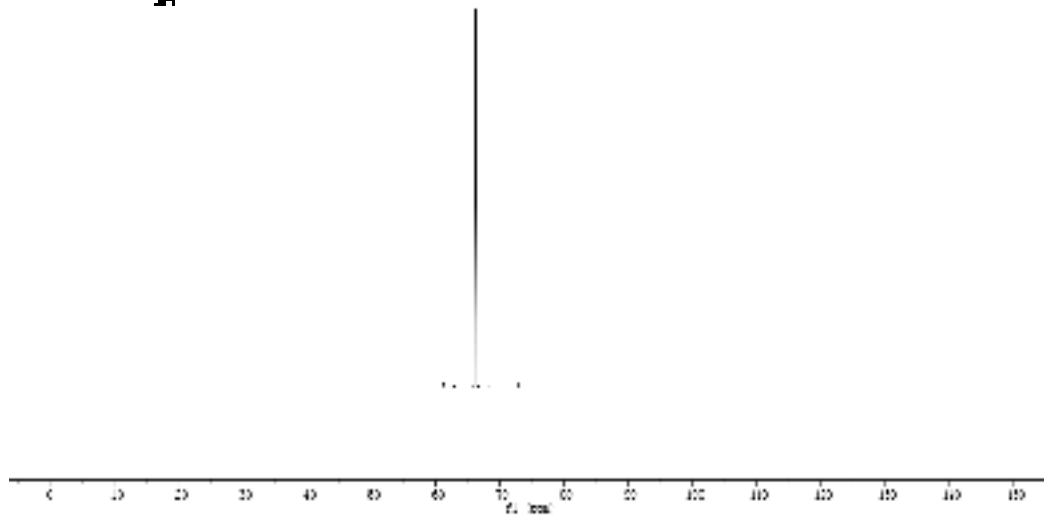
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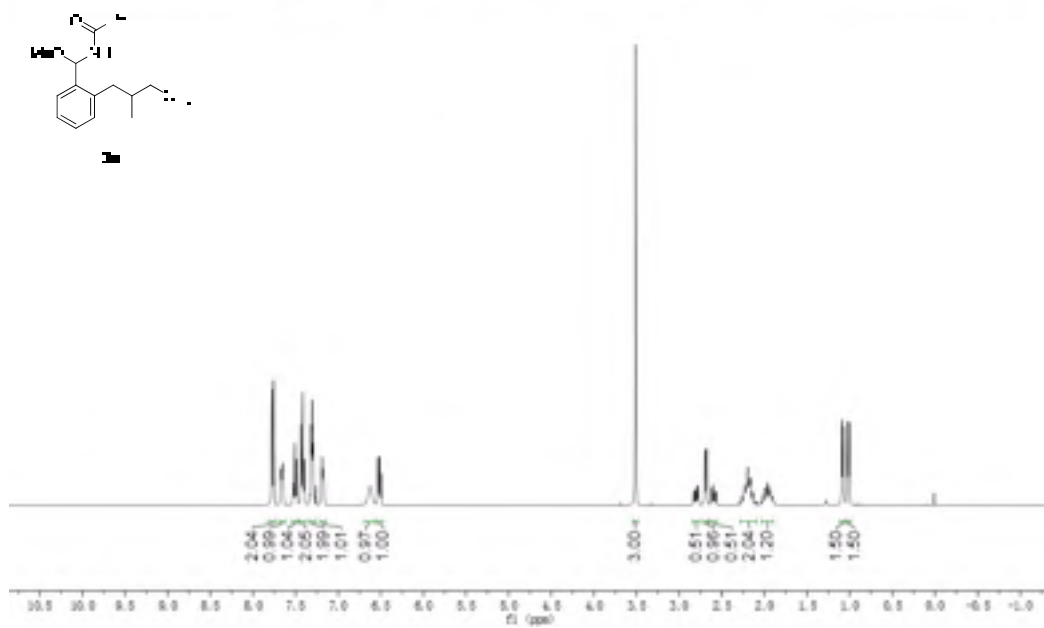
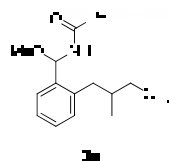
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7.656

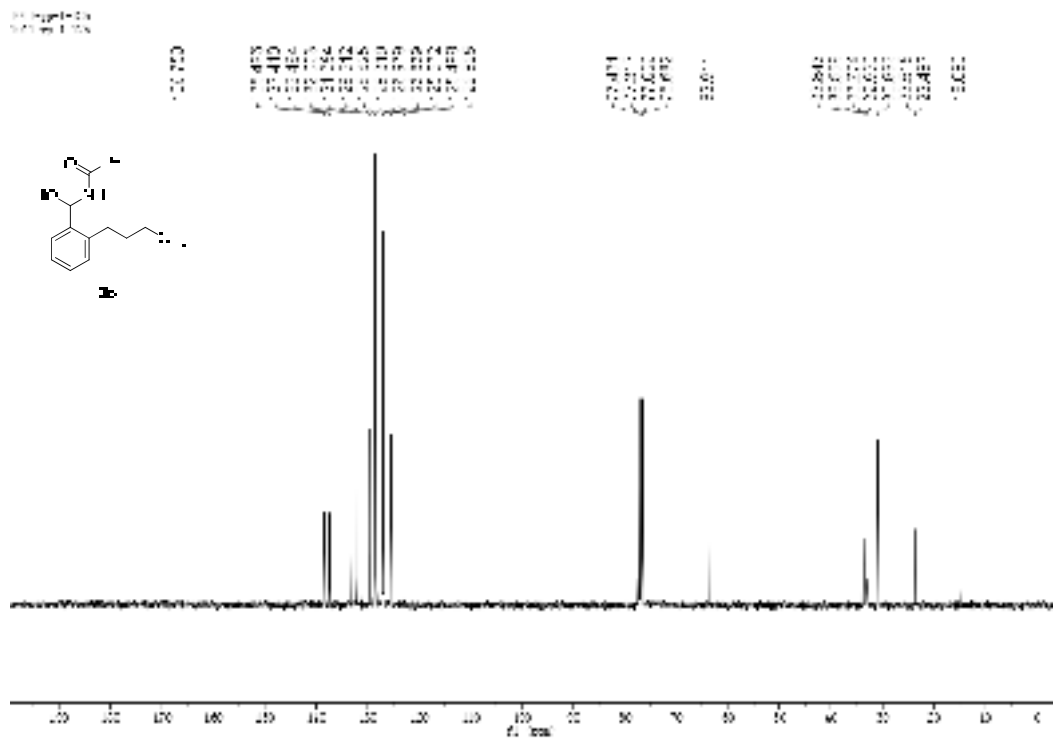
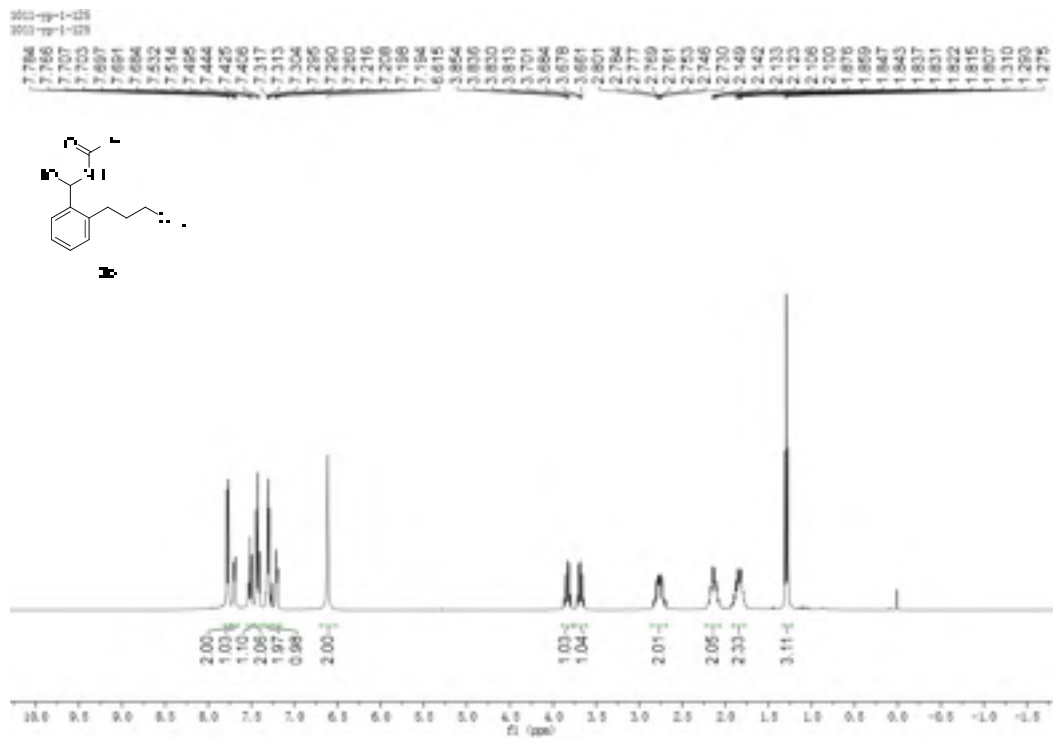


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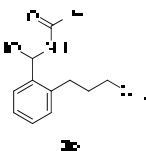








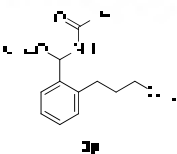
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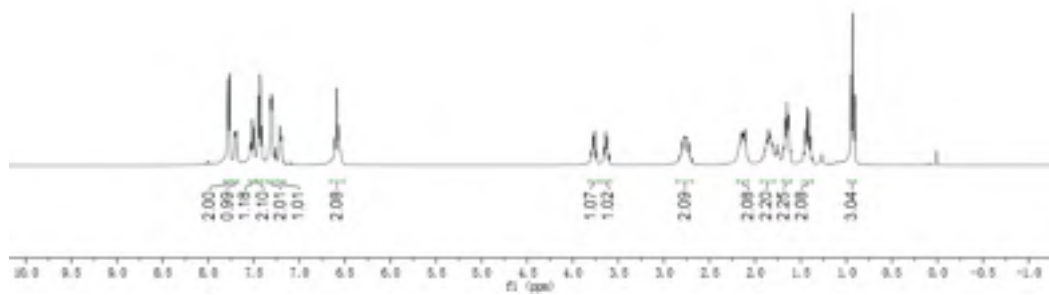
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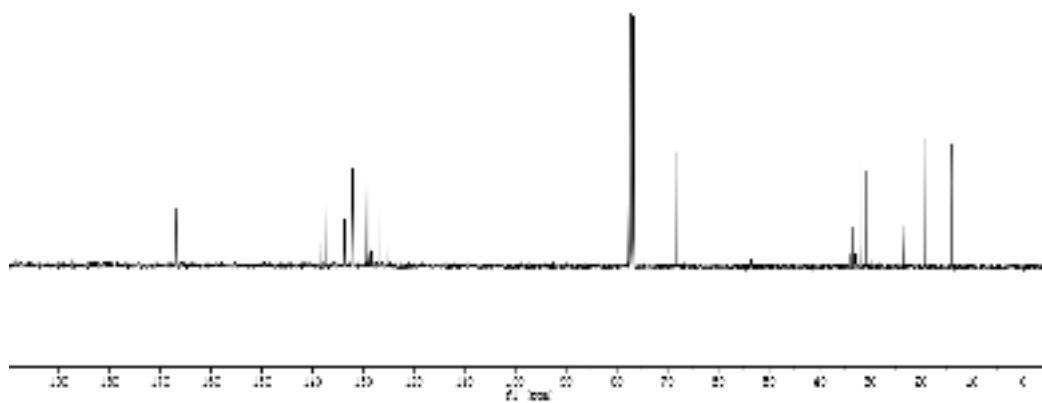
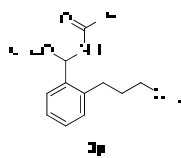
100 MHz, CDCl<sub>3</sub>



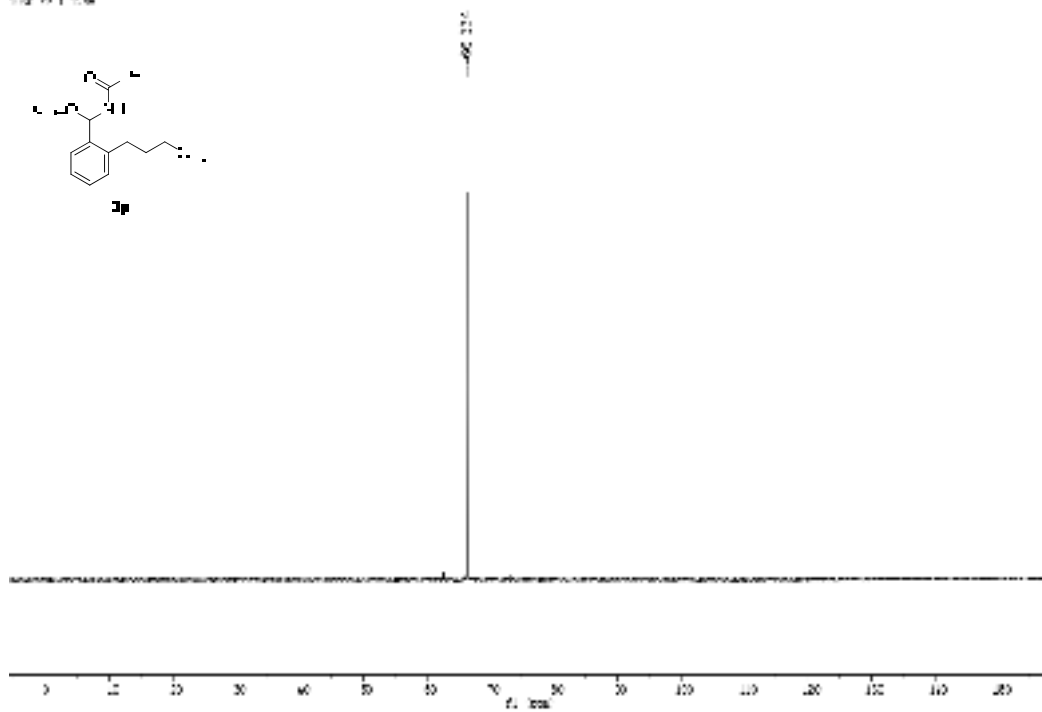
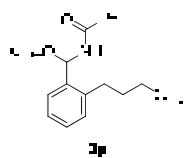
10

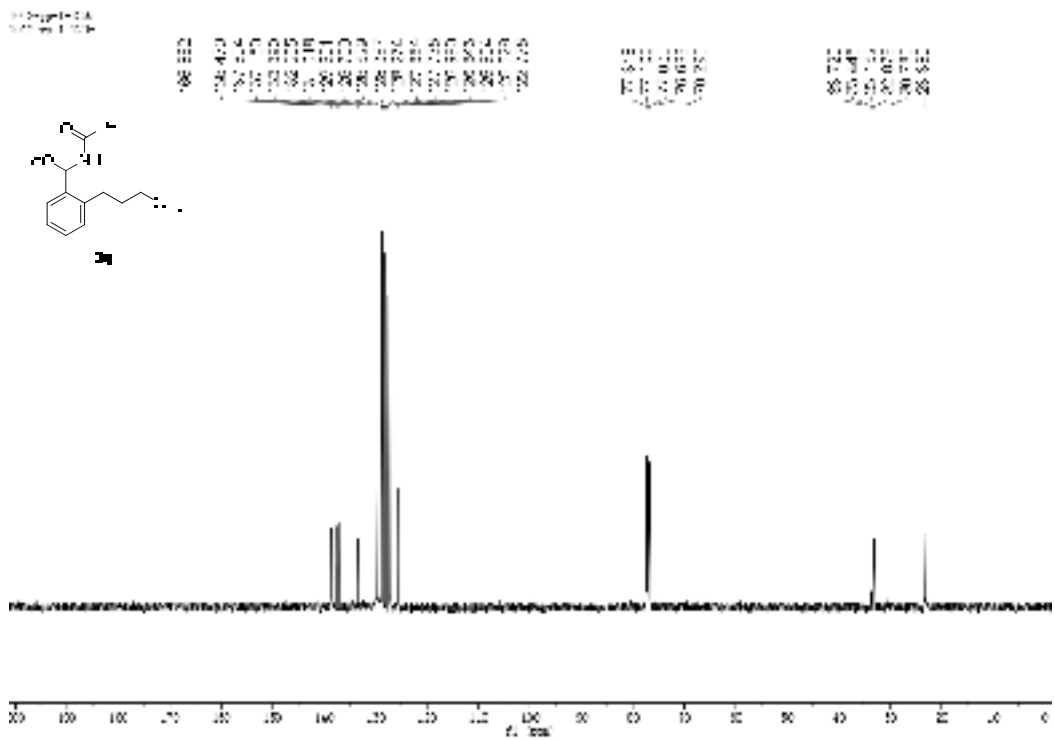
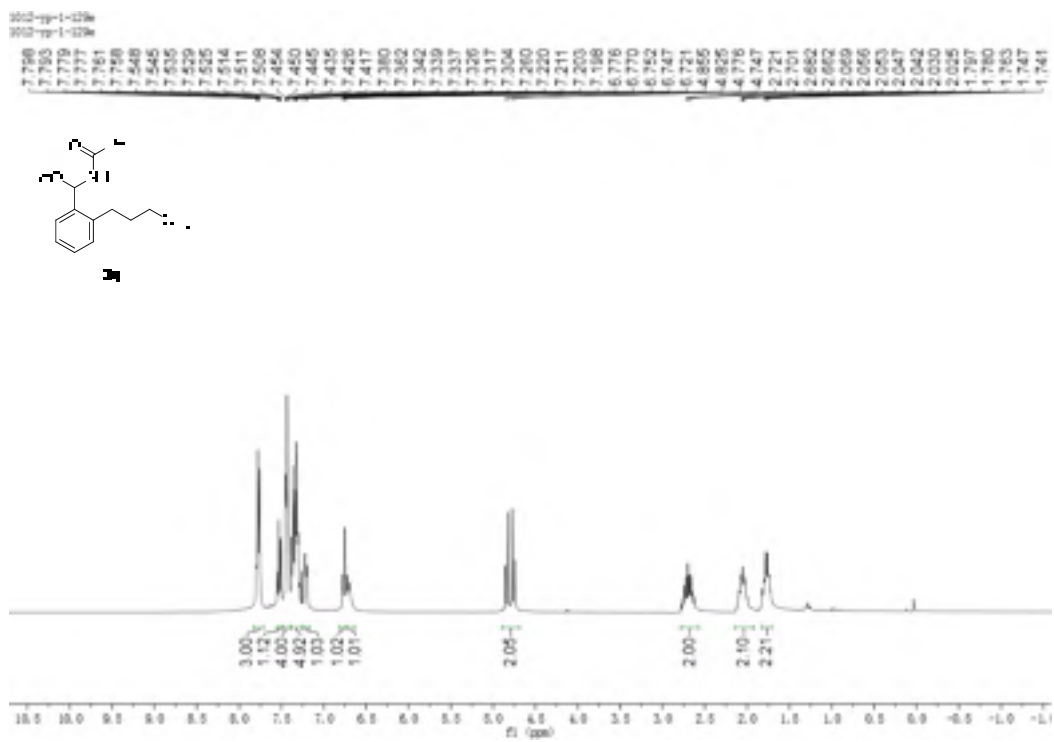


13C NMR (CDCl<sub>3</sub>)  
170.0, 168.0, 167.0, 166.0, 165.0, 164.0, 163.0, 162.0, 161.0, 160.0, 159.0, 158.0, 157.0, 156.0, 155.0, 154.0, 153.0, 152.0, 151.0, 150.0, 149.0, 148.0, 147.0, 146.0, 145.0, 144.0, 143.0, 142.0, 141.0, 140.0, 139.0, 138.0, 137.0, 136.0, 135.0, 134.0, 133.0, 132.0, 131.0, 130.0, 129.0, 128.0, 127.0, 126.0, 125.0, 124.0, 123.0, 122.0, 121.0, 120.0, 119.0, 118.0, 117.0, 116.0, 115.0, 114.0, 113.0, 112.0, 111.0, 110.0, 109.0, 108.0, 107.0, 106.0, 105.0, 104.0, 103.0, 102.0, 101.0, 100.0, 99.0, 98.0, 97.0, 96.0, 95.0, 94.0, 93.0, 92.0, 91.0, 90.0, 89.0, 88.0, 87.0, 86.0, 85.0, 84.0, 83.0, 82.0, 81.0, 80.0, 79.0, 78.0, 77.0, 76.0, 75.0, 74.0, 73.0, 72.0, 71.0, 70.0, 69.0, 68.0, 67.0, 66.0, 65.0, 64.0, 63.0, 62.0, 61.0, 60.0, 59.0, 58.0, 57.0, 56.0, 55.0, 54.0, 53.0, 52.0, 51.0, 50.0, 49.0, 48.0, 47.0, 46.0, 45.0, 44.0, 43.0, 42.0, 41.0, 40.0, 39.0, 38.0, 37.0, 36.0, 35.0, 34.0, 33.0, 32.0, 31.0, 30.0, 29.0, 28.0, 27.0, 26.0, 25.0, 24.0, 23.0, 22.0, 21.0, 20.0, 19.0, 18.0, 17.0, 16.0, 15.0, 14.0, 13.0, 12.0, 11.0, 10.0, 9.0, 8.0, 7.0, 6.0, 5.0, 4.0, 3.0, 2.0, 1.0, 0.0

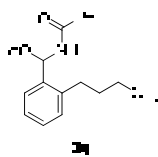


13C NMR (CDCl<sub>3</sub>)  
170.0, 168.0, 167.0, 166.0, 165.0, 164.0, 163.0, 162.0, 161.0, 160.0, 159.0, 158.0, 157.0, 156.0, 155.0, 154.0, 153.0, 152.0, 151.0, 150.0, 149.0, 148.0, 147.0, 146.0, 145.0, 144.0, 143.0, 142.0, 141.0, 140.0, 139.0, 138.0, 137.0, 136.0, 135.0, 134.0, 133.0, 132.0, 131.0, 130.0, 129.0, 128.0, 127.0, 126.0, 125.0, 124.0, 123.0, 122.0, 121.0, 120.0, 119.0, 118.0, 117.0, 116.0, 115.0, 114.0, 113.0, 112.0, 111.0, 110.0, 109.0, 108.0, 107.0, 106.0, 105.0, 104.0, 103.0, 102.0, 101.0, 100.0, 99.0, 98.0, 97.0, 96.0, 95.0, 94.0, 93.0, 92.0, 91.0, 90.0, 89.0, 88.0, 87.0, 86.0, 85.0, 84.0, 83.0, 82.0, 81.0, 80.0, 79.0, 78.0, 77.0, 76.0, 75.0, 74.0, 73.0, 72.0, 71.0, 70.0, 69.0, 68.0, 67.0, 66.0, 65.0, 64.0, 63.0, 62.0, 61.0, 60.0, 59.0, 58.0, 57.0, 56.0, 55.0, 54.0, 53.0, 52.0, 51.0, 50.0, 49.0, 48.0, 47.0, 46.0, 45.0, 44.0, 43.0, 42.0, 41.0, 40.0, 39.0, 38.0, 37.0, 36.0, 35.0, 34.0, 33.0, 32.0, 31.0, 30.0, 29.0, 28.0, 27.0, 26.0, 25.0, 24.0, 23.0, 22.0, 21.0, 20.0, 19.0, 18.0, 17.0, 16.0, 15.0, 14.0, 13.0, 12.0, 11.0, 10.0, 9.0, 8.0, 7.0, 6.0, 5.0, 4.0, 3.0, 2.0, 1.0, 0.0

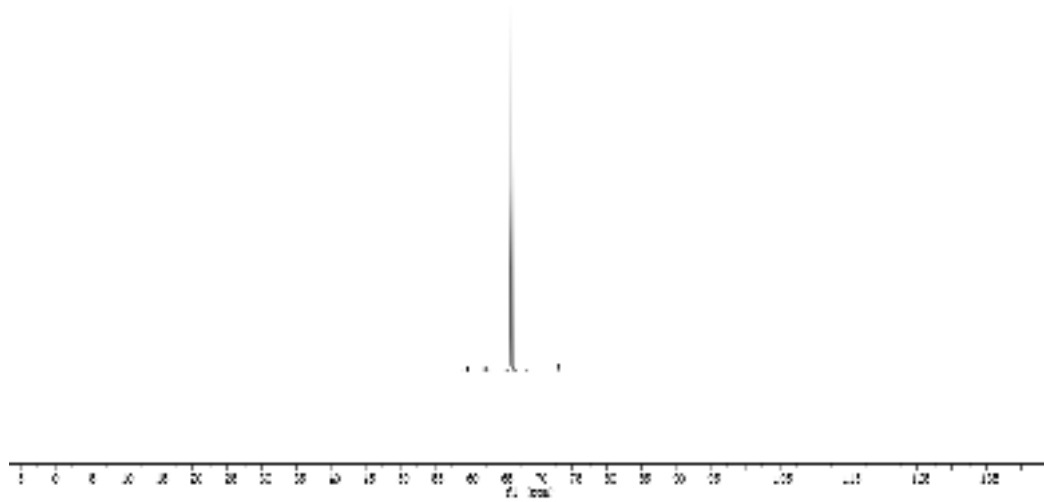




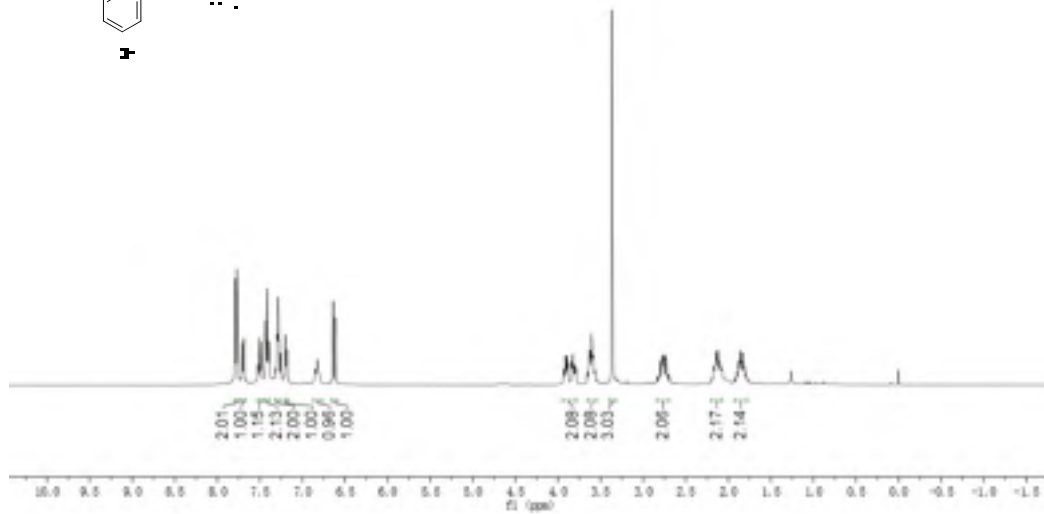
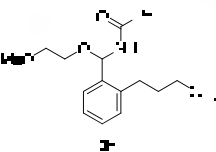
100% MeOH  
100% MeOH



3.908  
3.889  
3.882  
3.883



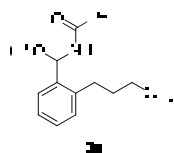
100% MeOH  
100% MeOH



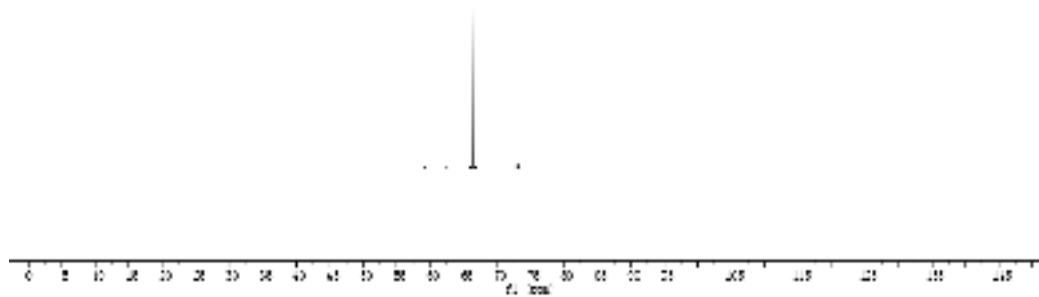




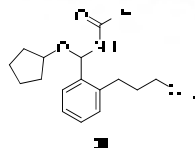
13C NMR (CDCl<sub>3</sub>)



171.25  
169.25  
141.11  
137.92

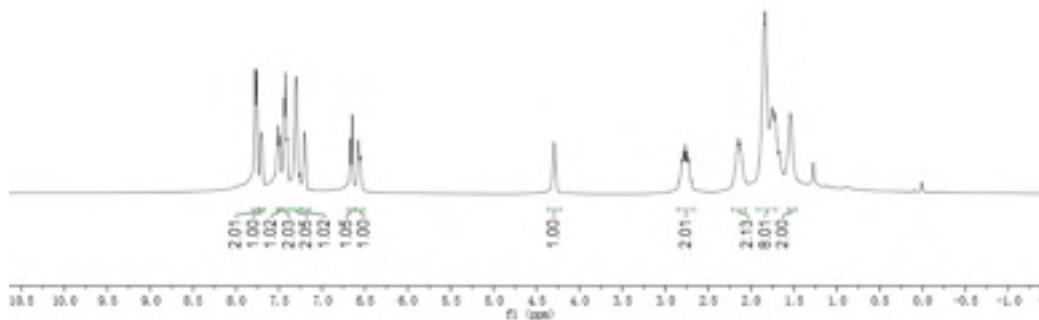


13C NMR (CDCl<sub>3</sub>)



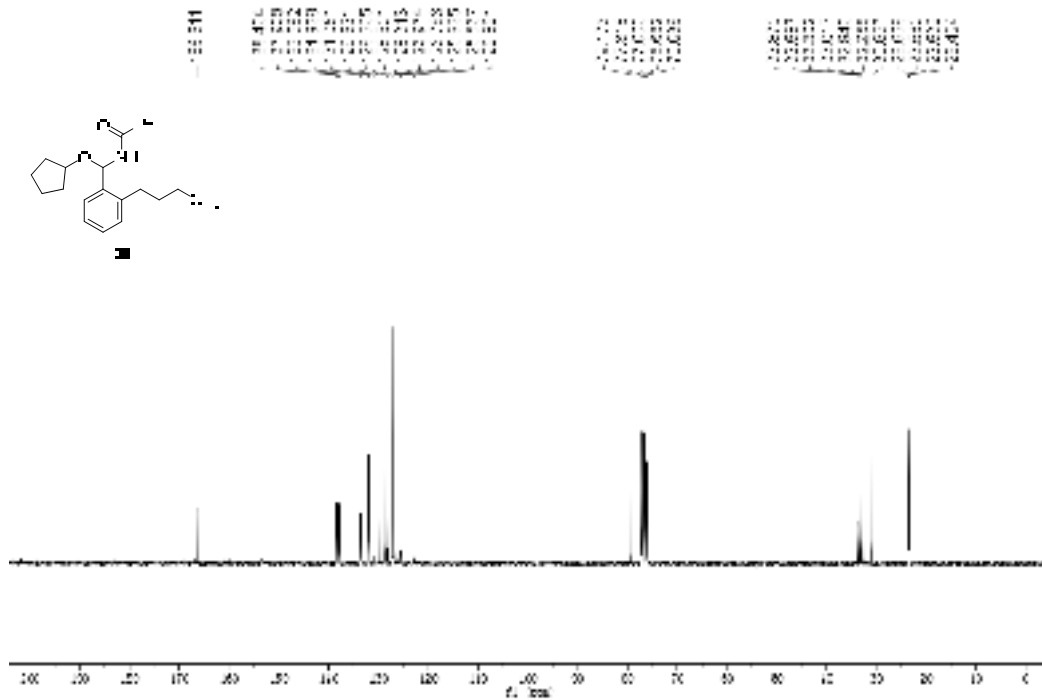
177.77  
177.60  
177.14  
175.04  
175.08  
174.80  
174.41  
174.22  
174.05  
173.07  
173.01  
172.80  
172.03  
171.98  
66.69  
66.66  
65.75  
65.53

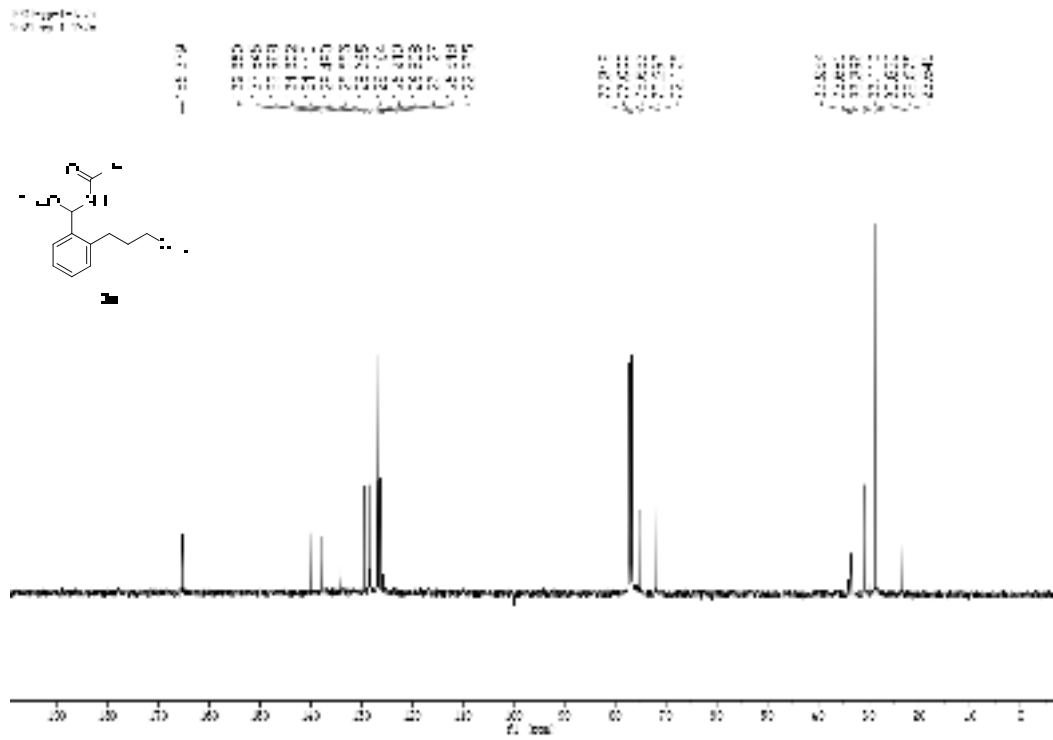
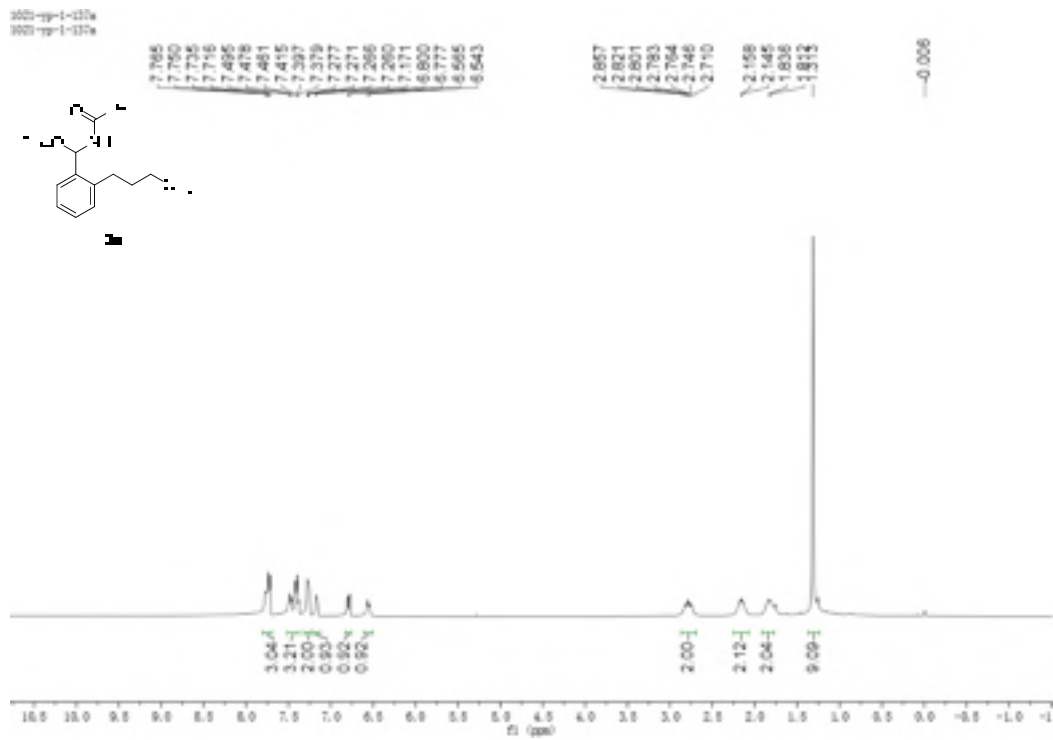
4.262  
2.840  
2.804  
2.786  
2.767  
2.747  
2.729  
2.693  
2.187  
2.154  
2.145  
2.131  
2.117  
1.848  
1.834  
1.745  
1.733  
1.654  
1.642  
1.545  
1.272





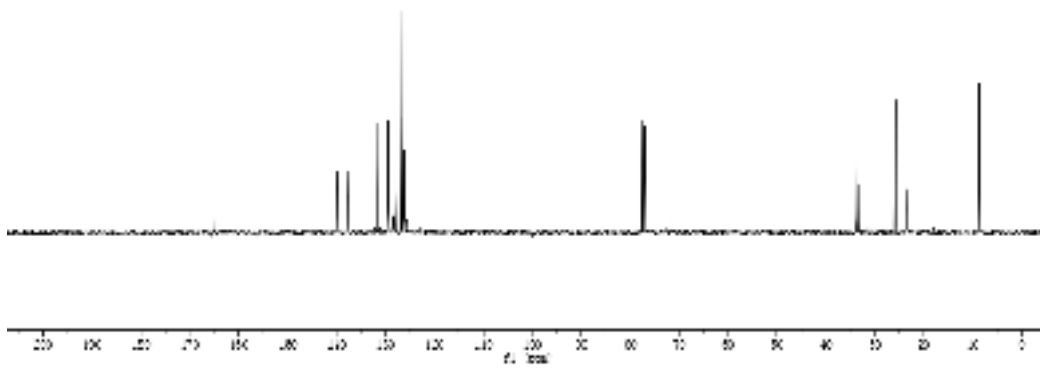
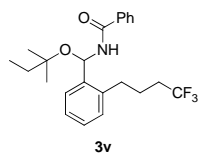
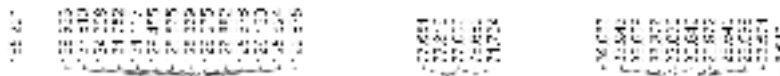
010mg010010





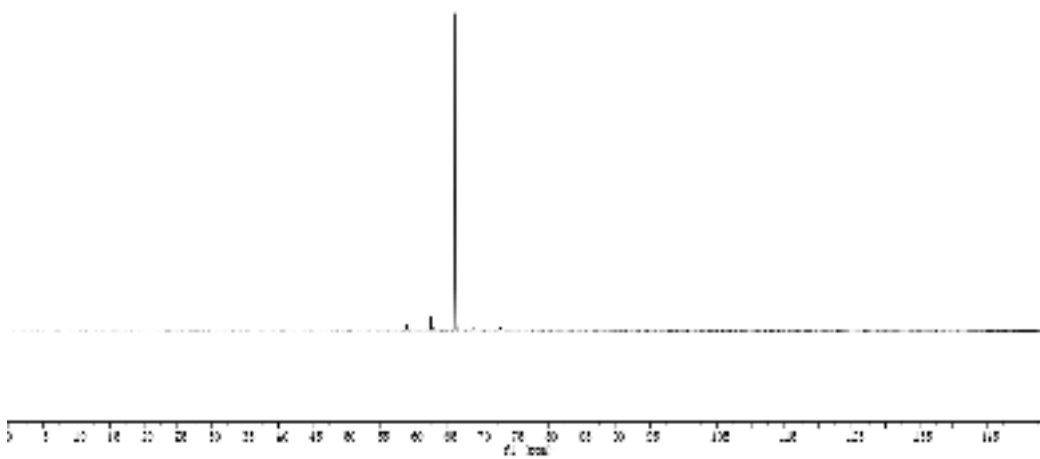
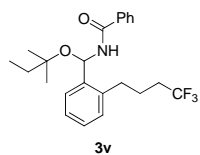


1H NMR (CDCl3)

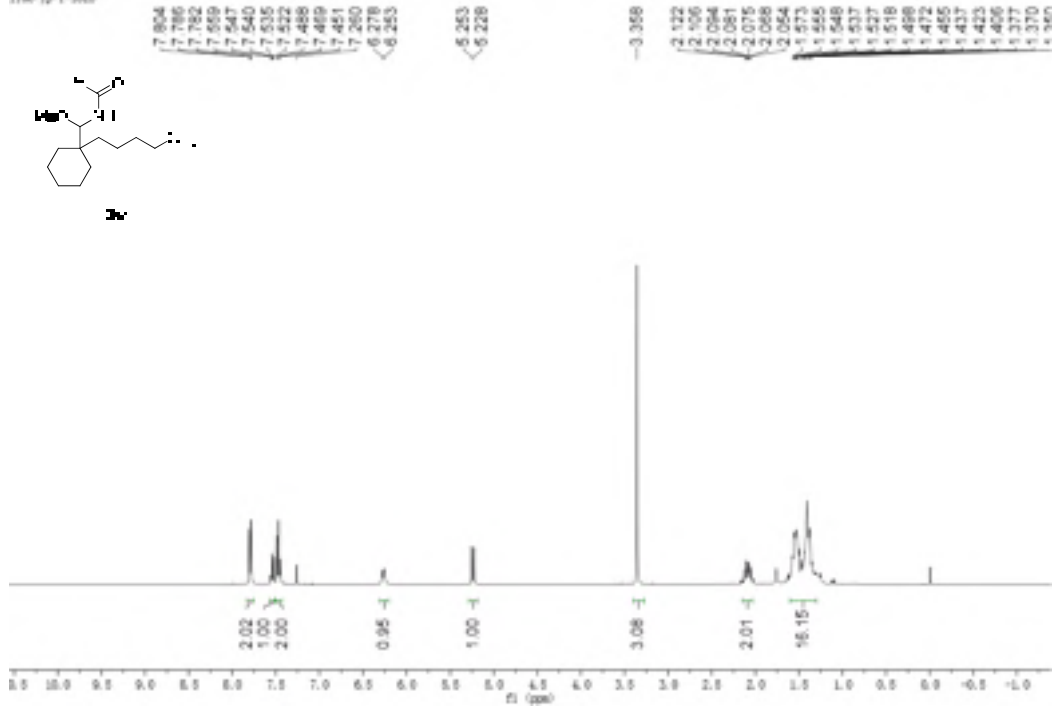


13C NMR (CDCl3)

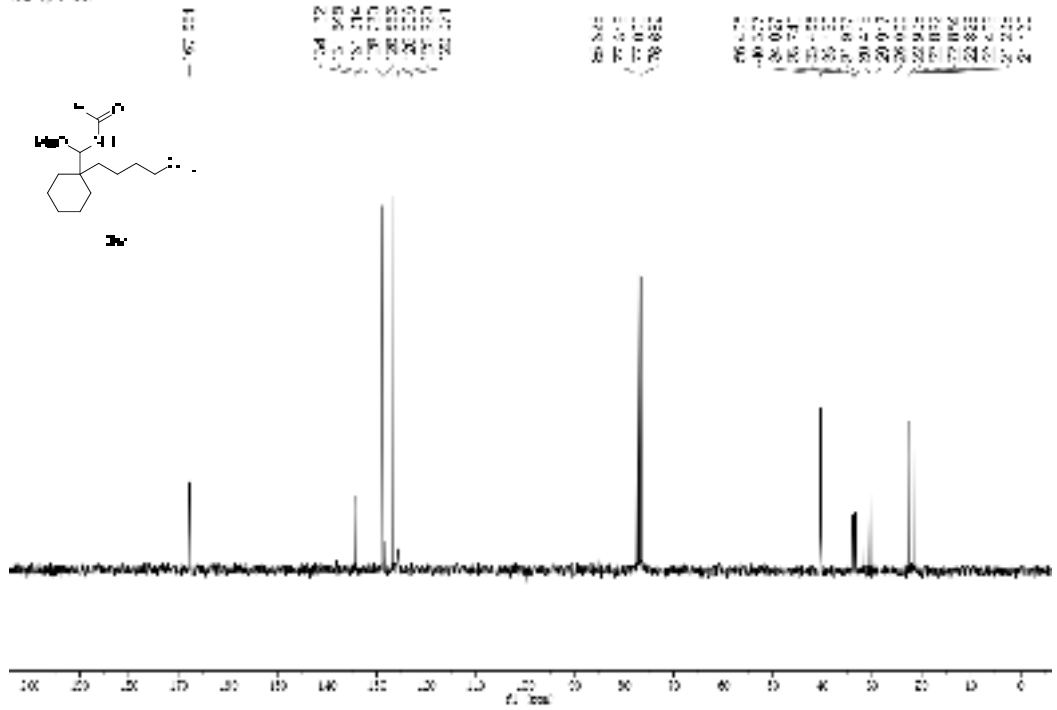
165.0



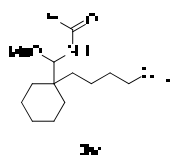
1188-pp1-1-02b  
1188-pp1-1-02b



1188-pp1-1-03  
1188-pp1-1-03



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7.091  
7.072  
7.053  
7.034

