

## Supporting information

### Visible-light-promoted N-H functionalization of O-substituted hydroxamic acid with diazo esters

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## 1. General information

Unless otherwise indicated, all the reagents and solvents were purchased from commercial suppliers and used without any further purification. Thin layer chromatography (TLC) was performed on commercial silica gel plates and flash column chromatography was performed with 300–400 mesh silica gel cartridge. Visualization of TLC achieved using ultraviolet light (254 nm). NMR spectra were recorded on a Bruker Avance 400 MHz (400 MHz for  $^1\text{H}$ ; 376 MHz for  $^{19}\text{F}$ ; 100 MHz for  $^{13}\text{C}$ ) instruments and are referenced relative to  $\text{CDCl}_3$  ( $\delta$  7.26 ppm for  $^1\text{H}$ , 77.16 ppm for  $^{13}\text{C}$ ) using TMS as internal standard. Data are recorded as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = penta), coupling constant ( $J$ ) in Hertz (Hz), and integration. High-resolution mass spectra (HRMS) were obtained on an Agilent mass spectrometer using ESI (electrospray ionization).

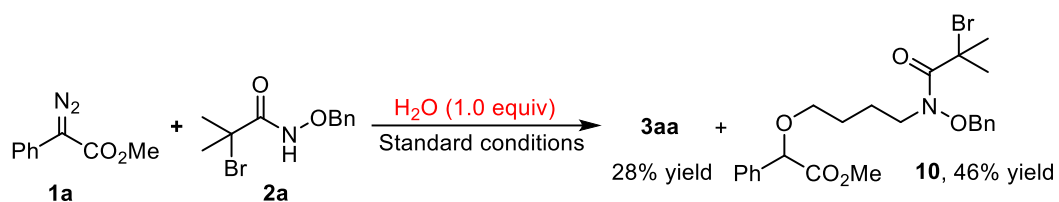
## 2. Optimization of reaction conditions

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

Entry	Base	Solvent	Yield (%) <sup>b</sup>	
			3aa	4aa
1	NaHCO <sub>3</sub>	THF	trace	n.d.
2	NaOH	THF	63	n.d.
3	DBU	THF	50	n.d.
4	KO <sup>t</sup> Bu	THF	90	trace
5 <sup>c</sup>	KO <sup>t</sup> Bu	THF	n.d.	n.d.
6 <sup>d</sup>	KO <sup>t</sup> Bu	THF	85	n.d.
7 <sup>e</sup>	KO <sup>t</sup> Bu	THF	29	n.d.
8 <sup>f</sup>	KO <sup>t</sup> Bu	THF	6	n.d.
9 <sup>g</sup>	KO <sup>t</sup> Bu	THF	18	n.d.
10 <sup>h</sup>	KO <sup>t</sup> Bu	THF	27	n.d.
11	KO <sup>t</sup> Bu	1,4-dioxane	n.d.	60
12	Cs <sub>2</sub> CO <sub>3</sub>	1,4-dioxane	n.d.	49
13	NaOH	1,4-dioxane	n.d.	43
14	NaHCO <sub>3</sub>	1,4-dioxane	n.d.	trace
15	KO <sup>t</sup> Bu	DCE	n.d.	n.d.
16	KO <sup>t</sup> Bu	DMF	n.d.	n.d.
17	KO <sup>t</sup> Bu	CH <sub>3</sub> CN	n.d.	n.d.
18 <sup>c</sup>	KO <sup>t</sup> Bu	1,4-dioxane	n.d.	n.d.

<sup>a</sup>Reaction conditions: **1a** (0.2 mmol), **2a** (1.5 equiv), base (1.2 equiv), solvent (2 mL), 14 h, room temperature, 30 W blue LEDs ( $\lambda \approx 440\text{-}450$  nm). <sup>b</sup>Isolated yields. <sup>c</sup>Without light irradiation. <sup>d</sup>Under N<sub>2</sub> atmosphere. <sup>e</sup>10 W green light ( $\lambda = 520$  nm). <sup>f</sup>12 W CFL. <sup>g</sup>2 W blue light ( $\lambda = 495$  nm). <sup>h</sup>5 W blue light ( $\lambda = 495$  nm). n.d. = no desired product was detected.

## Studies on the effect of water:



The yields of **3aa** and **10** were determined by <sup>1</sup>H NMR

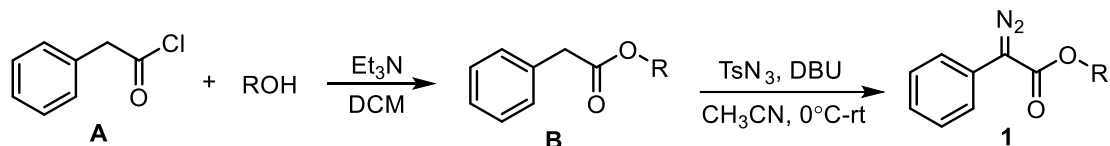
Methyl 2-diazo-2-phenylacetate **1a** (0.2 mmol), N-(benzyloxy)-2-bromo-2-methylpropanamide **2a** (0.3 mmol), KO<sup>t</sup>Bu (0.24 mmol) and H<sub>2</sub>O (0.2 mmol, 3.6 μL) were combined in 2 mL of THF, and the mixture was stirred under the irradiation of 30 W blue LEDs ( $\lambda \approx 440\text{-}450$  nm) for 14 hours at room temperature.

The reaction resulted in the formation of **3aa** in 28% yield and a N-H insertion product **10** that still brominated in 46% yield (The yields of products **3aa** and **10** were determined by <sup>1</sup>H NMR). TLC result showed that products **3aa** and **10** are difficult to separate because of resemble structure and close characters. This result suggested that water has much influence on the efficiency and chemoselectivity of this reaction.

### 3. Experimental section

#### (1) Preparation of starting materials

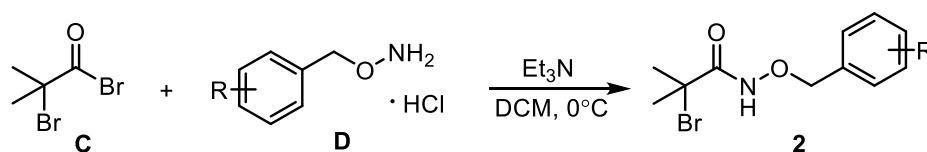
##### Synthesis of substrate **1**<sup>[1-2]</sup>:



To a flask containing ROH (1 equiv), DCM and Et<sub>3</sub>N (1.2 equiv), 2-phenylacetyl chloride **A** (1.2 equiv) was added and the reaction was kept at 0 °C for 2 h. Then the solution was warmed to room temperature and kept for 1 h. Afterwards, the solution was filtered and the filtrate was concentrated. Then it was extracted with ethyl acetate and dried over Na<sub>2</sub>SO<sub>4</sub> to give the crude product, which was next purified in flash column chromatography on silica gel using ethyl acetate/petroleum ether (v/v, 1:100) as eluent to get compound **B**.

DBU (1.5 equiv) was added dropwise to a solution of compound **B** (1 equiv) and TsN<sub>3</sub> (1.5 equiv) in dry MeCN at room temperature under nitrogen atmosphere. After stirring for 24 h the mixture was diluted with water and the aqueous phase was extracted with ethyl acetate. The combined organic layers were washed with brine and dried over Na<sub>2</sub>SO<sub>4</sub>. The solvent was evaporated and purification of the crude product by column chromatography (PE: EA = 95:5) yielded **1** as a red-orange oil.

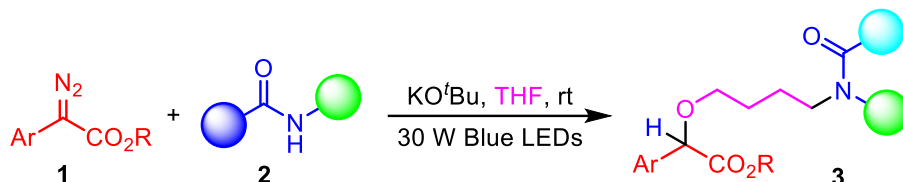
##### Synthesis of substrate **2**<sup>[3]</sup>:



To a solution of O-benzyloxyamine hydrochloride **D** (2.0 g, 12.5 mmol, 1.0 eq) in dichloromethane (50 ml), triethylamine (1.75 ml, 12.5 mmol, 1.0 eq) was added. The reaction mixture was then cooled to 0 °C in an ice water bath. Next,  $\alpha$ -haloacid halide **C** (1.5 ml, 12.5 mmol, 1.0 eq) was added dropwise to the reaction mixture. The reaction was stirred for 4 hours at 0 °C. After 4 hours, the mixture was allowed to warm to room temperature. The reaction was then quenched with water. The resulting mixture was diluted with DCM and was washed with water (3 × 50 mL), followed by brine. The

organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated under vacuum. The crude reaction mixture was purified by silica gel chromatography (ethyl acetate/hexanes) to give compound **2** as a white solid.

**(2) General procedure of this visible-light-promoted N-H functionalization of O-substituted hydroxamic acid with diazo esters.**

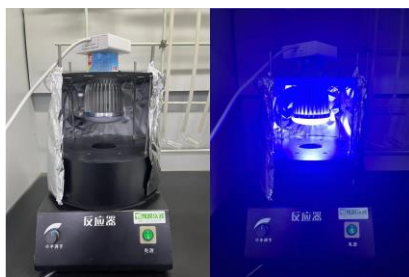


Aryldiazoacetates **1** (0.2 mmol),  $\alpha$ -halohydroxamates **2** (0.3 mmol) and KO<sup>t</sup>Bu (0.24 mmol) were combined in 2 mL of THF, and the mixture was stirred under the irradiation of 30 W blue LEDs ( $\lambda \approx 440$ -450 nm) for 14 hours at room temperature. As the reaction completed, the solution was concentrated under reduced pressure, then the residue was purified by column chromatography (PE/EA = 25/1) to afford the target product **3**.

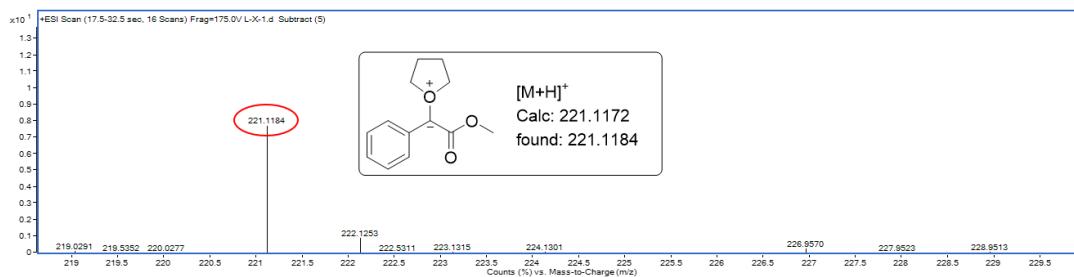
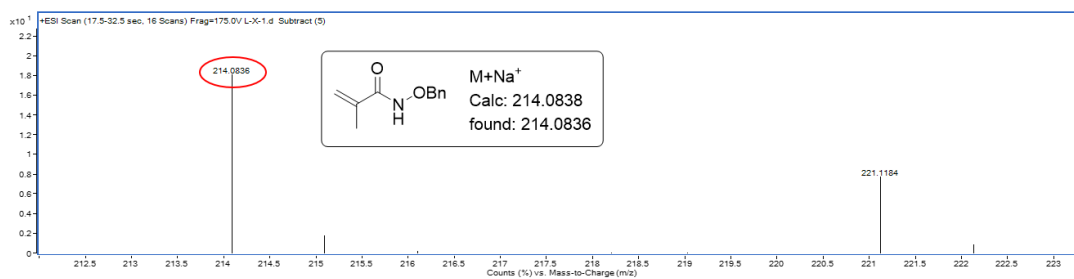
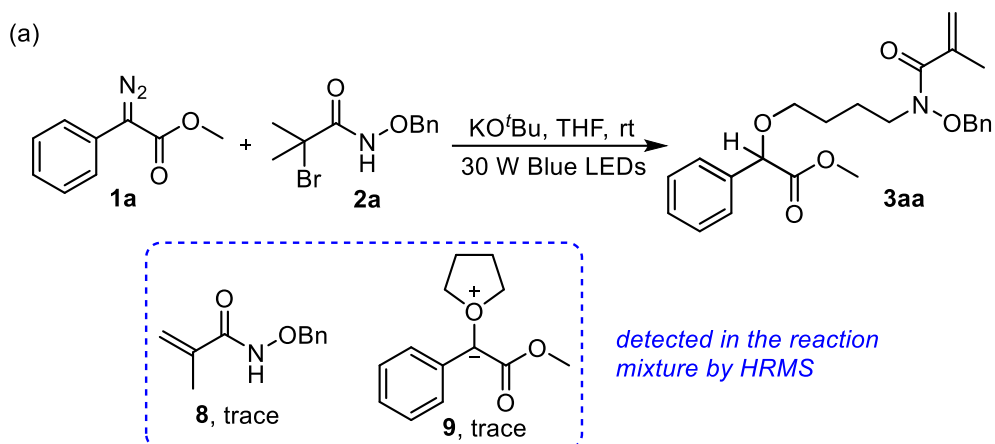


Aryldiazoacetates **1** (0.2 mmol),  $\alpha$ -halohydroxamates **2** (0.3 mmol) and KO<sup>t</sup>Bu (0.24 mmol) were combined in 2 mL of dry 1,4-dioxane, and the mixture was stirred under the irradiation of 30 W blue LEDs ( $\lambda \approx 440$ -450 nm) for 14 hours at room temperature. The solution was next concentrated under reduced pressure, then the residue was purified by column chromatography (PE/EA = 25/1) to afford the target product **4**.

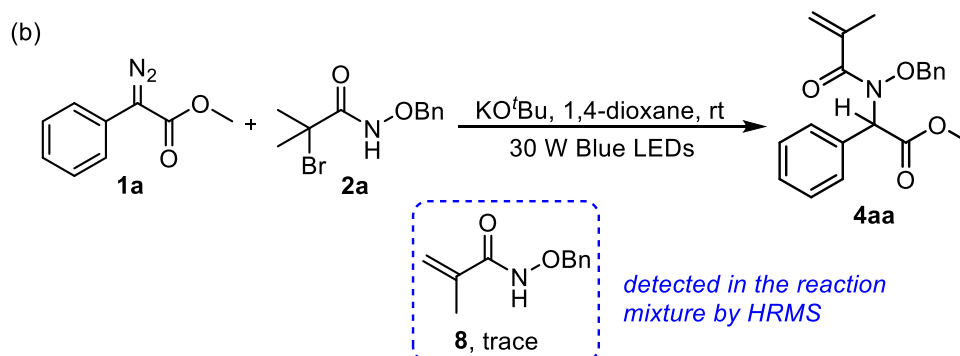
**The experimental setup:**

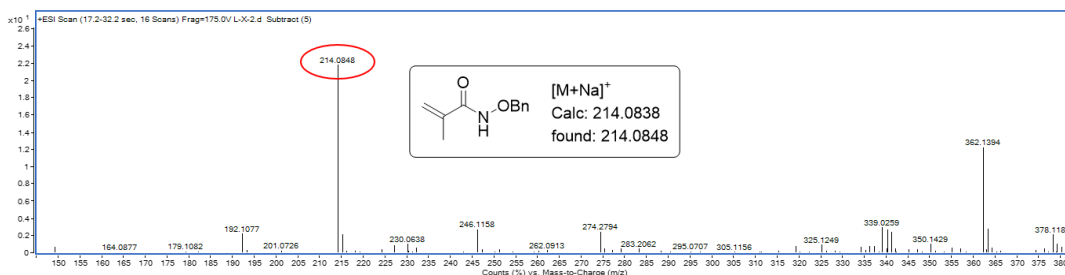


### (3) Mechanistic Studies

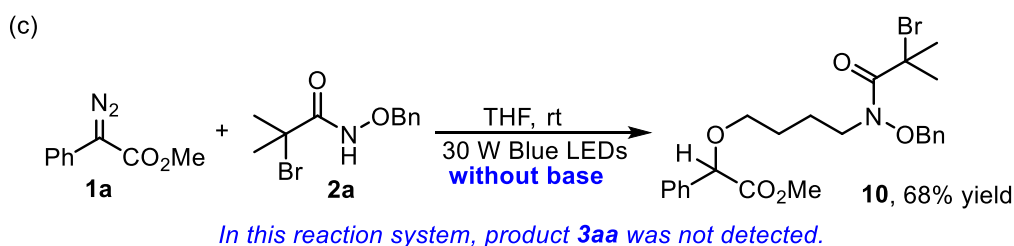


**Fig. S1 HRMS spectrum of compound 8 and 9**

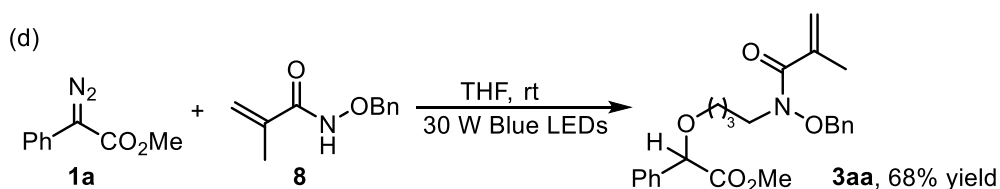




**Fig. S2 HRMS spectrum for compound 8**



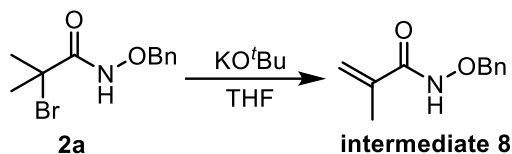
Methyl 2-diazo-2-phenylacetate **1a** (0.2 mmol), N-(benzyloxy)-2-bromo-2-methylpropanamide **2a** (0.3 mmol) were combined in 2 mL of THF, the mixture was stirred under the irradiation of 30 W blue LEDs ( $\lambda \approx 440\text{-}450$  nm) for 14 hours at room temperature. As the reaction completed, the solution was concentrated under reduced pressure, then the residue was purified by column chromatography (PE/EA = 25/1) affording the target product **10** in 68% yield.



Methyl 2-diazo-2-phenylacetate **1a** (0.2 mmol), N-(benzyloxy)methacrylamide **8** (0.3 mmol) were combined in 2 mL of THF, and the mixture was stirred under the irradiation of 30 W blue LEDs ( $\lambda \approx 440\text{-}450$  nm) for 14 hours at room temperature. As the reaction completed, the solution was concentrated under reduced pressure, then the residue was purified by column chromatography (PE/EA = 25/1) to afford the desired product **3aa** in 68% yield.

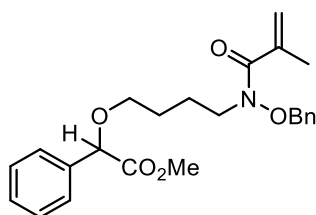


Synthesis of N-(benzyloxy)methacrylamide **8**:



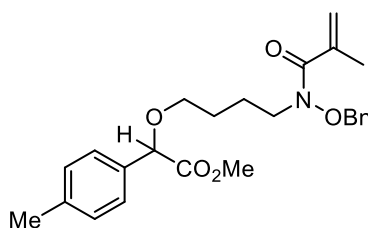
To a dry glass tube (35 mL, 18 x 180 mm) was added **2a** (0.3 mmol), KO<sup>t</sup>Bu (0.3 mmol) and 2 mL THF, then the mixture was stirred for 5 hours at room temperature. The resulting mixture was then concentrated in vacuo to afford crude product. The crude product further purified by column chromatography (PE/EA = 4/1) to afford N-(benzyloxy)methacrylamide **8**. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.56 (s, 1H), 7.41-7.34 (m, 5H), 5.57 (s, 1H), 5.31 (s, 1H), 4.94 (s, 2H), 1.91 (s, 3H) ppm. <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 167.1, 137.8, 135.3, 129.3, 128.8, 128.6, 120.4, 78.2, 18.5 ppm. HRMS (ESI) (m/z): calculated for C<sub>11</sub>H<sub>12</sub>NO<sub>2</sub>Na<sup>+</sup> [M + Na]<sup>+</sup> 241.0838; found, 241.0836.

#### 4. Analytical data of products



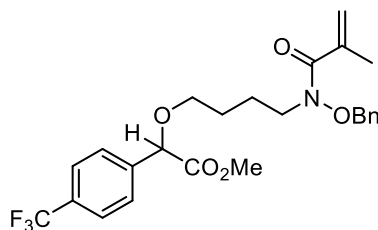
##### Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3aa)

Yellow oil (90%, 74.2 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.41 (m, 2H), 7.38-7.35 (m, 4H), 7.33-7.30 (m, 4H), 5.57 (s, 1H), 5.17 (t,  $J = 1.63$  Hz, 1H), 5.02 (s, 2H), 4.84 (s, 1H), 4.14 (t,  $J = 5.8$  Hz, 2H), 3.70 (s, 3H), 3.55-3.50 (m, 1H), 3.45-3.39 (m, 1H), 1.87 (s, 3H), 1.77-1.72 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 155.8, 137.7, 136.6, 136.0, 128.6, 128.6, 128.3, 128.3, 127.8, 127.1, 118.0, 81.0, 76.5, 71.5, 69.4, 52.2, 26.8, 26.0, 18.8 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{24}\text{H}_{29}\text{NO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  434.1938; found, 434.1938.



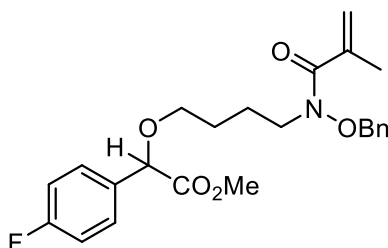
##### Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(*p*-tolyl)acetate (3ba)

Yellow oil (81%, 68.9 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36-7.30 (m, 7H), 7.16 (d,  $J = 7.8$  Hz, 2H), 5.57-5.56 (m, 1H), 5.17-5.16 (m, 1H), 5.02 (s, 2H), 4.80 (s, 1H), 4.13 (t,  $J = 6.0$  Hz, 2H), 3.69 (s, 3H), 3.53-3.47 (m, 1H), 3.43-3.38 (m, 1H), 2.34 (s, 3H), 1.87 (s, 3H), 1.76-1.71 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.5, 155.9, 138.5, 137.7, 136.0, 133.6, 129.3, 128.3, 128.3, 127.8, 127.1, 118.0, 80.9, 76.5, 71.5, 69.2, 52.2, 26.8, 26.0, 21.2, 18.8 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{25}\text{H}_{31}\text{NO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  448.2094; found, 448.2098.



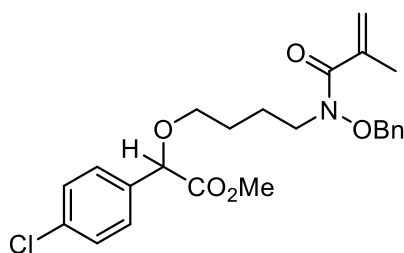
**Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(4-(trifluoromethyl)phenyl)acetate (3ca)**

Yellow oil (60%, 57.6 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61 (d,  $J = 8.2$  Hz, 2H), 7.56 (d,  $J = 8.3$  Hz, 2H), 7.38-7.35 (m, 3H), 7.33-7.31 (m, 2H), 5.56 (s, 1H), 5.17 (t,  $J = 1.6$  Hz, 1H), 5.03 (s, 2H), 4.89 (s, 1H), 4.16-4.13 (m, 2H), 3.71 (s, 3H), 3.59-3.54 (m, 1H), 3.46-3.41 (m, 1H), 1.87 (s, 3H), 1.78-1.74 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 155.8, 140.5, 137.7, 136.1, 130.9, 130.6, 128.3, 128.3, 127.8, 127.4, 125.5 (q,  $J_{\text{CF}} = 3.8$  Hz), 117.9, 80.4, 76.6, 71.4, 69.8, 52.4, 26.8, 26.0, 18.8 ppm.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.66 (3F, s) ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{25}\text{H}_{28}\text{F}_3\text{NO}_5\text{Na}^+ [\text{M} + \text{Na}]^+$  502.1812; found, 502.1816.



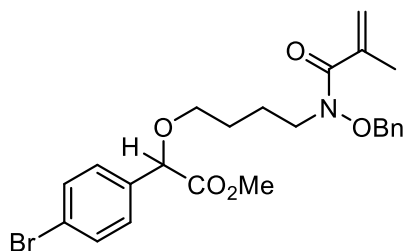
**Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(4-fluorophenyl)acetate (3da)**

Yellow oil (75%, 64.4 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.38 (m, 2H), 7.35-7.31 (m, 5H), 7.05-7.01 (m, 2H), 5.56 (s, 1H), 5.18-5.16 (m, 1H), 5.03 (s, 2H), 4.81 (s, 1H), 4.15-4.12 (m, 2H), 3.70 (s, 3H), 3.55-3.50 (m, 1H), 3.44-3.39 (m, 1H), 1.87 (s, 3H), 1.76-1.73 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.3, 162.9 (d,  $J_{\text{CF}} = 245.8$  Hz), 155.8, 137.7, 136.1, 132.5 (d,  $J_{\text{CF}} = 3.3$  Hz), 128.9 (d,  $J_{\text{CF}} = 8.3$  Hz), 128.3, 128.3, 127.8, 117.9, 115.5 (d,  $J_{\text{CF}} = 21.5$  Hz), 80.3, 76.5, 71.5, 69.5, 52.3, 26.8, 26.0, 18.8 ppm.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -113.23 (1F, s) ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{24}\text{H}_{28}\text{FNO}_5\text{Na}^+ [\text{M} + \text{Na}]^+$  452.1844; found, 452.1848.



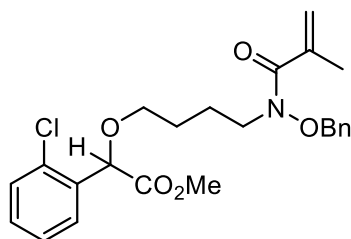
**Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(4-chlorophenyl)acetate (3ea)**

Yellow oil (87%, 77.5 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38-7.37 (m, 1H), 7.36-7.33 (m, 6H), 7.31-7.30 (m, 2H), 5.56 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.03 (s, 2H), 4.80 (s, 1H), 4.15-4.13 (m, 2H), 3.69 (s, 3H), 3.55-3.50 (m, 1H), 3.45-3.38 (m, 1H), 1.87 (s, 3H), 1.76-1.73 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.0, 155.8, 137.7, 136.0, 135.1, 134.5, 128.8, 128.4, 128.3, 128.3, 127.8, 117.9, 80.3, 76.5, 71.5, 69.5, 52.3, 26.8, 26.0, 18.8 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{24}\text{H}_{28}\text{ClNO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  468.1548; found, 468.1547.



**Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(4-bromophenyl)acetate (3fa)**

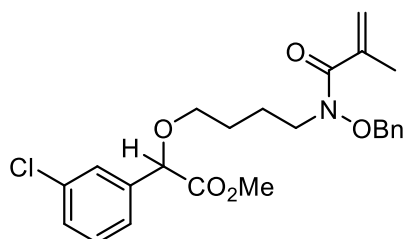
Yellow oil (76%, 74.4 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49-7.47 (m, 2H), 7.36-7.33 (m, 3H), 7.32-7.29 (m, 4H), 5.56 (s, 1H), 5.18-5.17 (m, 1H), 5.03 (s, 2H), 4.78 (s, 1H), 4.15-4.12 (m, 2H), 3.69 (s, 3H), 3.55-3.50 (m, 1H), 3.43-3.38 (m, 1H), 1.87 (s, 3H), 1.76-1.72 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.0, 155.8, 137.7, 136.1, 135.7, 131.7, 128.7, 128.3, 128.3, 127.8, 122.7, 117.9, 80.4, 76.5, 71.5, 69.6, 52.3, 26.8, 26.0, 18.8 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{24}\text{H}_{28}\text{BrNO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  512.1043; found, 512.1045.



**Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(2-chlorophenyl)acetate (3ga)**

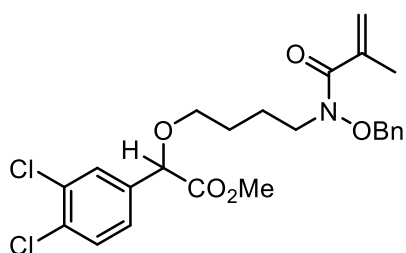
Yellow oil (83%, 73.9 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51-7.49 (m, 1H), 7.39-

7.35 (m, 4H), 7.33-7.28 (m, 4H), 5.56 (d,  $J = 1.0$  Hz, 1H), 5.32 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.02 (s, 2H), 4.14-4.12 (m, 2H), 3.71 (s, 3H), 3.61-3.56 (m, 1H), 3.47-3.41 (m, 1H), 1.87 (s, 3H), 1.75-1.71 (m, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 155.9, 137.7, 136.0, 134.7, 133.7, 129.8, 129.6, 128.7, 128.3, 128.3, 127.8, 127.2, 118.0, 77.2, 76.5, 71.5, 69.8, 52.3, 26.8, 26.00, 18.8 ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{24}\text{H}_{28}\text{ClNO}_5\text{Na}^+ [\text{M} + \text{Na}]^+$  468.1548; found, 468.1553.



**Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(3-chlorophenyl)acetate (3ha)**

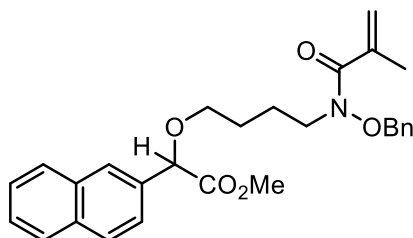
Yellow oil (79%, 70.4 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.43 (m, 1H), 7.38-7.33 (m, 4H), 7.32-7.29 (m, 4H), 5.57 (s, 1H), 5.18 (t,  $J = 1.6$  Hz, 1H), 5.03 (s, 2H), 4.79 (s, 1H), 4.16-4.13 (m, 2H), 3.71 (s, 3H), 3.56-3.50 (m, 1H), 3.45-3.39 (m, 1H), 1.87 (s, 3H), 1.76-1.73 (m, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.9, 155.8, 138.6, 137.7, 136.0, 134.5, 129.8, 128.8, 128.3, 128.3, 127.8, 127.2, 125.2, 118.0, 80.3, 76.5, 71.4, 69.6, 52.4, 26.7, 26.0, 18.8 ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{24}\text{H}_{28}\text{ClNO}_5\text{Na}^+ [\text{M} + \text{Na}]^+$  468.1548; found, 468.1550.



**Methyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-(3,4-dichlorophenyl)acetate (3ia)**

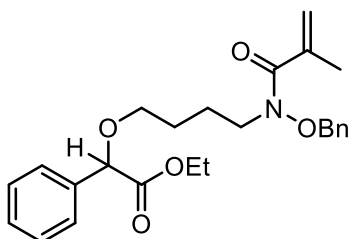
Yellow oil (74%, 70.9 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 (d,  $J = 2.0$  Hz, 1H), 7.42 (d,  $J = 8.3$  Hz, 1H), 7.38-7.33 (m, 4H), 7.32-7.30 (m, 1H), 7.29-7.27 (m, 1H), 5.56 (d,  $J = 1.0$  Hz, 1H), 5.18 (t,  $J = 1.7$  Hz, 1H), 5.03 (s, 2H), 4.77 (s, 1H), 4.16-4.13 (m, 2H), 3.71 (s, 3H), 3.56-3.51 (m, 1H), 3.44-3.39 (m, 1H), 1.88 (s, 3H), 1.76-1.73 (m, 4H)

ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.6, 155.8, 137.6, 136.8, 136.0, 132.7, 132.8, 130.6, 129.0, 128.3, 128.2, 127.8, 126.3, 117.9, 79.7, 76.6, 71.4, 69.8, 52.5, 26.7, 26.0, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{24}\text{H}_{27}\text{Cl}_2\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  502.1158; found, 502.1158.



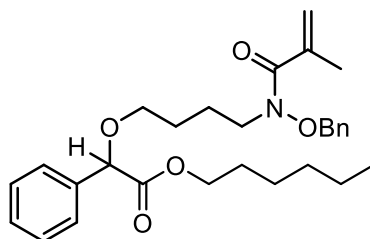
**Methyl 2-(4-(N-(benzyloxy)methacrylamido)butoxy)-2-(naphthalen-2-yl)acetate (3ja)**

Yellow oil (79%, 72.9 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 (s, 1H), 7.85-7.81 (m, 3H), 7.56-7.53 (m, 1H), 7.50-7.47 (m, 2H), 7.37-7.33 (m, 3H), 7.32-7.29 (m, 2H), 5.56 (s, 1H), 5.16 (t,  $J = 1.7$  Hz, 1H), 5.01 (s, 2H), 5.00 (s, 1H), 4.16-4.13 (m, 2H), 3.70 (s, 3H), 3.59-3.54 (m, 1H), 3.49-3.44 (m, 1H), 1.86 (s, 3H), 1.78-1.75 (m, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.3, 155.8, 137.7, 136.0, 134.0, 133.4, 133.1, 128.5, 128.3, 128.3, 128.1, 127.8, 127.7, 126.6, 126.4, 126.3, 124.5, 118.0, 81.2, 76.5, 71.5, 69.4, 52.3, 26.8, 26.0, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{28}\text{H}_{31}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  484.2094; found, 484.2095.



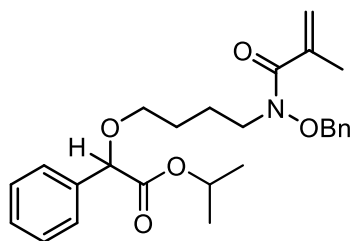
**Ethyl 2-(4-(N-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3ka)**

Yellow oil (76%, 64.6 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45-7.42 (m, 2H), 7.38-7.34 (m, 4H), 7.33-7.30 (m, 4H), 5.57 (s, 1H), 5.17 (t,  $J = 1.6$  Hz, 1H), 5.02 (s, 2H), 4.81 (s, 1H), 4.18-4.11 (m, 4H), 3.56-3.51 (m, 1H), 3.45-3.40 (m, 1H), 1.87 (s, 3H), 1.77-1.73 (m, 4H), 1.20 (t,  $J = 7.1$  Hz, 3H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.0, 155.9, 137.7, 136.7, 136.0, 128.5, 128.3, 128.3, 127.8, 127.1, 118.0, 81.1, 76.5, 71.5, 69.3, 61.1, 26.8, 26.0, 18.8, 14.1 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{25}\text{H}_{31}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  448.2094; found, 448.2096.



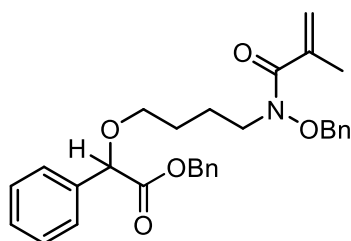
**Hexyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3la)**

Yellow oil (67%, 64.5 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.42 (m, 2H), 7.38-7.34 (m, 4H), 7.33-7.30 (m, 4H), 5.57 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.02 (s, 2H), 4.82 (s, 1H), 4.16-4.13 (m, 2H), 4.09 (t,  $J = 6.7$  Hz, 2H), 3.56-3.51 (m, 1H), 3.45-3.40 (m, 1H), 1.87 (s, 3H), 1.77-1.72 (m, 4H), 1.57-1.52 (m, 2H), 1.23-1.18 (m, 6H), 0.84 (t,  $J = 6.6$  Hz, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.0, 155.9, 137.7, 136.8, 136.0, 128.5, 128.3, 128.3, 127.8, 127.1, 118.0, 81.1, 76.5, 71.6, 69.3, 65.2, 31.3, 28.4, 26.8, 26.0, 25.3, 22.5, 18.8, 14.0 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{29}\text{H}_{39}\text{NO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  504.2720; found, 504.2719.



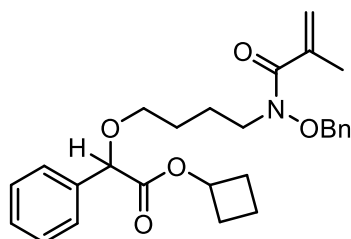
**Isopropyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3ma)**

Yellow oil (51%, 44.8 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.42 (m, 2H), 7.38-7.35 (m, 3H), 7.34-7.30 (m, 5H), 5.57 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.03 (s, 2H), 5.06-5.00 (m, 1H), 4.78 (s, 1H), 4.15 (t,  $J = 5.9$  Hz, 2H), 3.57-3.51 (m, 1H), 3.46-3.41 (m, 1H), 1.87 (s, 3H), 1.78-1.72 (m, 4H), 1.23 (d,  $J = 6.3$  Hz, 3H), 1.11 (d,  $J = 6.2$  Hz, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.6, 155.9, 137.7, 136.8, 136.1, 128.5, 128.4, 128.3, 128.3, 127.8, 127.0, 118.0, 81.2, 76.5, 71.6, 69.3, 68.7, 26.9, 26.0, 21.8, 21.5, 18.8 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{26}\text{H}_{33}\text{NO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  462.2251; found, 462.2251.



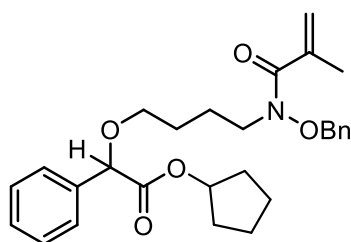
**Benzyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3na)**

Yellow oil (79%, 77.0 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.41 (m, 2H), 7.37-7.32 (m, 7H), 7.30-7.28 (m, 4H), 7.22-7.19 (m, 2H), 5.56 (s, 1H), 5.17-5.08 (m, 3H), 5.02 (s, 2H), 4.87 (s, 1H), 4.14-4.11 (m, 2H), 3.55-3.50 (m, 1H), 3.45-3.40 (m, 1H), 1.87 (s, 3H), 1.75-1.72 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 155.8, 137.7, 136.5, 136.0, 135.5, 128.6, 128.6, 128.5, 128.3, 128.3, 128.2, 127.9, 127.8, 127.1, 118.0, 81.0, 76.5, 71.5, 69.4, 66.7, 26.8, 26.0, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{30}\text{H}_{33}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  510.2251; found, 510.2253.



**Cyclobutyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3oa)**

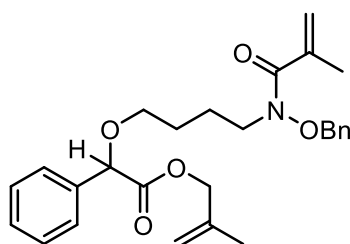
Yellow oil (80%, 72.2 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.42 (m, 2H), 7.38-7.35 (m, 4H), 7.33-7.30 (m, 4H), 5.57 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.02 (s, 2H), 5.01-4.97 (m, 1H), 4.78 (s, 1H), 4.14 (t,  $J = 5.9$  Hz, 2H), 3.56-3.50 (m, 1H), 3.45-3.40 (m, 1H), 2.35-2.31 (m, 1H), 2.29-2.24 (m, 1H), 2.09-1.99 (m, 1H), 1.98-1.91 (m, 1H), 1.87 (s, 3H), 1.78-1.71 (m, 5H), 1.62-1.54 (m, 1H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 155.9, 137.7, 136.7, 136.0, 128.5, 128.5, 128.3, 128.3, 127.8, 127.1, 118.0, 81.0, 76.5, 71.6, 69.5, 69.3, 30.2, 30.1, 26.8, 26.0, 18.8, 13.5 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{27}\text{H}_{33}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  474.2251; found, 474.2247.



**Cyclopentyl 2-(4-(*N*-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3pa)**

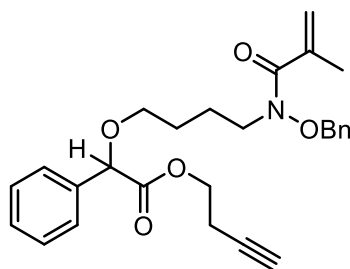


Yellow oil (91%, 84.7 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.43-7.41 (m, 2H), 7.38-7.35 (m, 3H), 7.33-7.30 (m, 5H), 5.57 (s, 1H), 5.20-5.17 (m, 2H), 5.02 (s, 2H), 4.77 (s, 1H), 4.15 (t,  $J = 5.9$  Hz, 2H), 3.56-3.51 (m, 1H), 3.45-3.40 (m, 1H), 1.87 (s, 3H), 1.77-1.73 (m, 4H), 1.71-1.61 (m, 3H), 1.59-1.46 (m, 5H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 155.9, 137.7, 136.9, 136.0, 128.4, 128.4, 128.3, 128.3, 127.8, 127.0, 118.0, 81.1, 77.9, 76.5, 71.6, 69.3, 32.5, 32.5, 26.8, 26.0, 23.6, 23.5, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{28}\text{H}_{35}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  488.2407; found, 488.2408.



**2-Methylallyl 2-(4-(N-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3qa)**

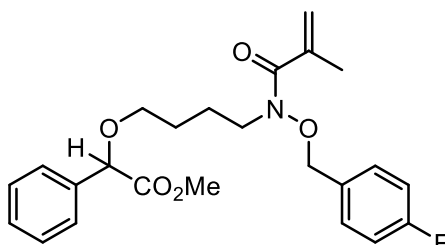
Yellow oil (60%, 54.2 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.46-7.43 (m, 2H), 7.38-7.35 (m, 4H), 7.33-7.30 (m, 4H), 5.57 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.02 (s, 2H), 4.86 (s, 1H), 4.83 (d,  $J = 7.2$  Hz, 2H), 4.56-4.48 (m, 2H), 4.14 (t,  $J = 5.8$  Hz, 2H), 3.58-3.53 (m, 1H), 3.46-3.41 (m, 1H), 1.87 (s, 3H), 1.77-1.72 (m, 4H), 1.61 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.6, 155.8, 139.4, 137.7, 136.7, 136.0, 128.6, 128.6, 128.3, 128.3, 127.8, 127.1, 118.0, 113.1, 81.1, 76.5, 71.5, 69.4, 68.1, 26.8, 26.0, 19.3, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{27}\text{H}_{33}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  474.2251; found, 474.2248.



**But-3-yn-1-yl 2-(4-(N-(benzyloxy)methacrylamido)butoxy)-2-phenylacetate (3ra)**

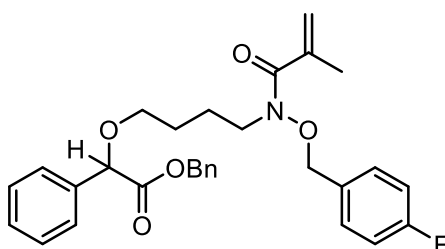
Yellow oil (71%, 63.8 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.46-7.43 (m, 2H), 7.38-7.35 (m, 4H), 7.33-7.31 (m, 4H), 5.57 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.02 (s, 2H), 4.85 (s, 1H), 4.24-4.17 (m, 2H), 4.14 (t,  $J = 5.9$  Hz, 2H), 3.58-3.53 (m, 1H), 3.46-3.41 (m, 1H), 2.49-2.45 (m, 2H), 1.93-1.91 (m, 1H), 1.87 (s, 3H), 1.78-1.72 (m, 4H) ppm.

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 155.8, 137.7, 136.5, 136.0, 128.6, 128.6, 128.3, 128.3, 127.8, 127.1, 118.0, 80.9, 79.6, 76.5, 71.5, 70.0, 69.4, 62.6, 26.8, 26.0, 18.9, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{27}\text{H}_{31}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  472.2094; found, 472.2098.



**Methyl 2-(4-(N-((4-fluorobenzyl)oxy)methacrylamido)butoxy)-2-phenylacetate (3ab)**

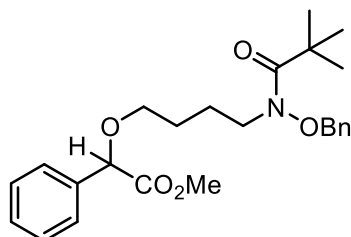
Yellow oil (83%, 71.3 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.42 (m, 2H), 7.38-7.35 (m, 2H), 7.34-7.32 (m, 3H), 7.04-6.99 (m, 2H), 5.56 (s, 1H), 5.18 (t,  $J = 1.7$  Hz, 1H), 4.97 (s, 2H), 4.84 (s, 1H), 4.12 (t,  $J = 5.8$  Hz, 2H), 3.70 (s, 3H), 3.55-3.50 (m, 1H), 3.45-3.40 (m, 1H), 1.87 (s, 3H), 1.75 (t,  $J = 3.0$  Hz, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 162.5 (d,  $J_{\text{CF}} = 245.8$  Hz), 156.0, 136.6, 135.9, 133.5 (d,  $J_{\text{CF}} = 3.2$  Hz), 130.2 (d,  $J_{\text{CF}} = 8.1$  Hz), 128.7, 128.6, 127.1, 118.2, 115.1 (d,  $J_{\text{CF}} = 21.5$  Hz), 81.0, 75.7, 71.5, 69.4, 52.2, 26.8, 26.0, 18.8 ppm.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -114.50 (1F, s) ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{24}\text{H}_{28}\text{FNO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  452.1844; found, 452.1847.



**Benzyl 2-(4-(N-((4-fluorobenzyl)oxy)methacrylamido)butoxy)-2-phenylacetate (3nb)**

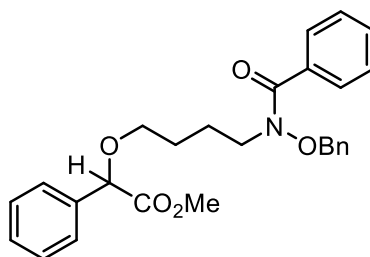
Yellow oil (65%, 65.7 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.41 (m, 2H), 7.34-7.32 (m, 4H), 7.31-7.29 (m, 4H), 7.22-7.20 (m, 2H), 7.03-6.98 (m, 2H), 5.56 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.18-5.09 (m, 2H), 4.97 (s, 2H), 4.87 (s, 1H), 4.12-4.09 (m,

2H), 3.56-3.50 (m, 1H), 3.45-3.40 (m, 1H), 1.86 (s, 3H), 1.75-1.72 (m, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 163.5 (d,  $J_{\text{CF}} = 246.1$  Hz) 156.0, 136.5, 135.9, 135.5, 133.5 (d,  $J_{\text{CF}} = 3.2$  Hz), 130.2 (d,  $J_{\text{CF}} = 8.2$  Hz), 128.6, 128.6, 128.5, 128.2, 128.0, 127.2, 118.2, 115.1 (d,  $J_{\text{CF}} = 21.3$  Hz), 81.1, 75.7, 71.5, 69.4, 66.7, 26.8, 26.0, 18.8 ppm.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -114.50 (1F, s) ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{30}\text{H}_{32}\text{FNO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  528.2157; found, 528.2162.



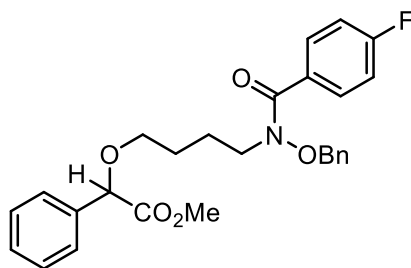
**Methyl 2-(4-(N-(benzyloxy)pivalamido)butoxy)-2-phenylacetate (3ad)**

Yellow oil (58%, 49.6 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44-7.42 (m, 2H), 7.37-7.32 (m, 7H), 7.30-7.28 (m, 1H), 4.91 (s, 2H), 4.84 (s, 1H), 4.27-4.24 (m, 2H), 3.69 (s, 3H), 3.54-3.50 (m, 1H), 3.45-3.39 (m, 1H), 1.72-1.69 (m, 4H), 1.10 (s, 9H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 162.3, 137.9, 136.6, 128.6, 128.6, 128.3, 128.2, 127.6, 127.1, 81.1, 76.0, 72.2, 69.5, 52.2, 36.5, 27.6, 26.9, 26.0 ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{25}\text{H}_{33}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  450.2251; found, 450.2249.



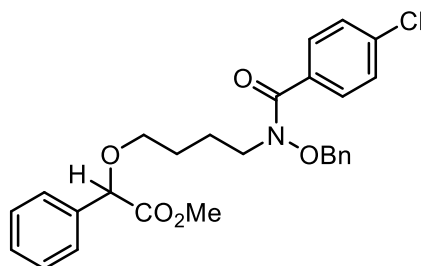
**Methyl 2-(4-(N-(benzyloxy)benzamido)butoxy)-2-phenylacetate (3ag)**

Yellow oil (56%, 50.1 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69-7.66 (m, 2H), 7.43-7.40 (m, 4H), 7.37-7.34 (m, 5H), 7.33-7.31 (m, 4H), 5.10 (s, 2H), 4.84 (s, 1H), 4.27 (t,  $J = 6.0$  Hz, 2H), 3.69 (s, 3H), 3.57-3.51 (m, 1H), 3.46-3.41 (m, 1H), 1.83-1.76 (m, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 154.5, 137.8, 136.6, 131.4, 129.9, 128.6, 128.6, 128.6, 128.3, 128.3, 127.8, 127.1, 127.0, 81.0, 76.6, 71.7, 69.3, 52.2, 26.9, 26.0 ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{27}\text{H}_{29}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  470.1938; found, 470.1937.



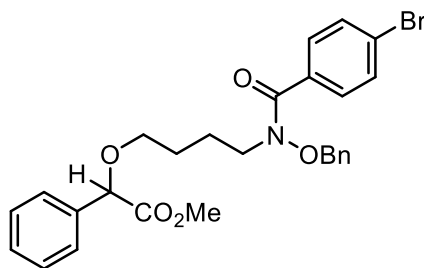
**Methyl 2-(4-(*N*-(benzyloxy)-4-fluorobenzamido)butoxy)-2-phenylacetate (3ah)**

Yellow oil (91%, 84.7 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70-7.66 (m, 2H), 7.43-7.39 (m, 4H), 7.37-7.33 (m, 5H), 7.32-7.30 (m, 1H), 7.03-6.99 (m, 2H), 5.08 (s, 2H), 4.84 (s, 1H), 4.30 (t,  $J = 6.1$  Hz, 2H), 3.69 (s, 3H), 3.57-3.52 (m, 1H), 3.47-3.41 (m, 1H), 1.84-1.74 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.3, 163.8 (d,  $J_{\text{CF}} = 249.7$  Hz), 153.4, 137.7, 136.6, 128.9 (d,  $J_{\text{CF}} = 8.4$  Hz), 128.7, 128.6, 128.4, 128.3, 127.9, 127.6 (d,  $J_{\text{CF}} = 3.1$  Hz), 127.1, 115.3 (d,  $J_{\text{CF}} = 21.8$  Hz), 81.0, 76.7, 72.0, 69.3, 52.2, 26.9, 26.0 ppm.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -110.95 (1F, s) ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{27}\text{H}_{28}\text{FNO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  488.1844; found, 488.1843.



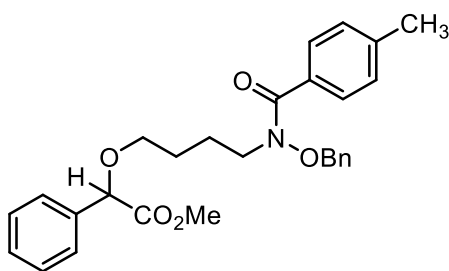
**Methyl 2-(4-(*N*-(benzyloxy)-4-chlorobenzamido)butoxy)-2-phenylacetate (3ai)**

Yellow oil (92%, 88.6 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64-7.62 (m, 2H), 7.43-7.42 (m, 1H), 7.41-7.37 (m, 4H), 7.35-7.33 (m, 4H), 7.32-7.28 (m, 3H), 5.09 (s, 2H), 4.83 (s, 1H), 4.31 (t,  $J = 6.1$  Hz, 2H), 3.69 (s, 3H), 3.57-3.51 (m, 1H), 3.46-3.41 (m, 1H), 1.84-1.74 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.3, 153.2, 137.6, 136.5, 135.8, 130.1, 128.6, 128.6, 128.5, 128.3, 128.3, 128.1, 127.9, 127.1, 81.0, 76.7, 72.0, 69.3, 52.2, 26.9, 25.9 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{27}\text{H}_{28}\text{ClNO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  504.1548; found, 504.1549.



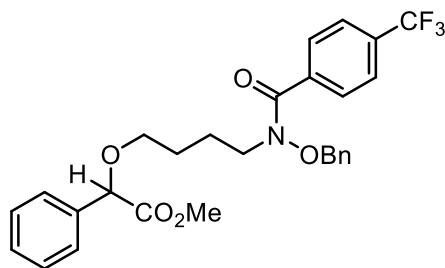
**Methyl 2-(4-(*N*-(benzyloxy)-4-bromobenzamido)butoxy)-2-phenylacetate (3aj)**

Yellow oil (82%, 86.3 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58-7.55 (m, 2H), 7.46-7.45 (m, 1H), 7.44-7.43 (m, 1H), 7.42-7.38 (m, 4H), 7.37-7.31 (m, 6H), 5.08 (s, 2H), 4.83 (s, 1H), 4.30 (t,  $J = 6.1$  Hz, 2H), 3.69 (s, 3H), 3.57-3.51 (m, 1H), 3.46-3.41 (m, 1H), 1.84-1.73 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 153.3, 137.6, 136.5, 131.4, 130.6, 128.7, 128.6, 128.4, 128.4, 128.3, 127.9, 127.1, 124.2, 81.0, 76.8, 72.0, 69.3, 52.2, 26.9, 26.0 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{27}\text{H}_{28}\text{BrNO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  548.1043; found, 548.1042.



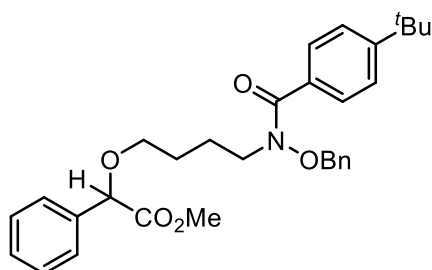
**Methyl 2-(4-(*N*-(benzyloxy)-4-methylbenzamido)butoxy)-2-phenylacetate (3ak)**

Yellow oil (81%, 74.7 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.56 (d,  $J = 8.0$  Hz, 2H), 7.43-7.39 (m, 4H), 7.37-7.29 (m, 6H), 7.14 (d,  $J = 7.9$  Hz, 2H), 5.09 (s, 2H), 4.83 (s, 1H), 4.24 (t,  $J = 6.0$  Hz, 2H), 3.69 (s, 3H), 3.56-3.51 (m, 1H), 3.46-3.40 (m, 1H), 2.35 (s, 3H), 1.82-1.74 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 154.7, 140.0, 137.8, 136.6, 129.0, 128.6, 128.6, 128.4, 128.3, 128.3, 127.8, 127.1, 127.0, 81.0, 76.5, 71.6, 69.3, 52.2, 26.9, 25.9, 21.4 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{28}\text{H}_{31}\text{NO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  484.2094; found, 484.2099.



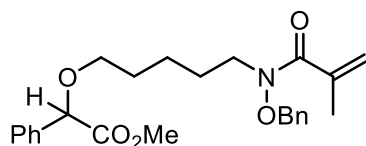
**Methyl 2-(4-(N-(benzyloxy)-4-(trifluoromethyl)benzamido)butoxy)-2-phenylacetate (3al)**

Yellow oil (88%, 90.7 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.82 (d,  $J = 8.2$  Hz, 2H), 7.57 (d,  $J = 8.3$  Hz, 2H), 7.43-7.39 (m, 4H), 7.38-7.35 (m, 3H), 7.34-7.32 (m, 3H), 5.11 (s, 2H), 4.84 (s, 1H), 4.36 (t,  $J = 6.1$  Hz, 2H), 3.69 (s, 3H), 3.58-3.52 (m, 1H), 3.47-3.42 (m, 1H), 1.85-1.75 (m, 4H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 152.7, 137.4, 136.6, 135.2, 131.7, 131.3, 128.7, 128.6, 128.4, 128.4, 128.0, 127.1, 127.0, 125.2 (q,  $J_{\text{CF}} = 3.96$  Hz), 81.1, 72.2, 69.3, 52.2, 26.9, 26.0 ppm.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.76 (3F, s) ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{28}\text{H}_{28}\text{F}_3\text{NO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  538.1812; found, 538.1810.



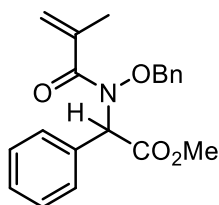
**Methyl 2-(4-(N-(benzyloxy)-4-(tert-butyl)benzamido)butoxy)-2-phenylacetate (3am)**

Yellow oil (86%, 86.6 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60-7.58 (m, 2H), 7.43-7.39 (m, 4H), 7.37-7.34 (m, 4H), 7.34-7.30 (m, 4H), 5.09 (s, 2H), 4.84 (s, 1H), 4.24 (t,  $J = 6.0$  Hz, 2H), 3.69 (s, 3H), 3.57-3.51 (m, 1H), 3.46-3.41 (m, 1H), 1.84-1.75 (m, 4H), 1.31 (s, 9H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 154.7, 153.1, 137.9, 136.6, 128.6, 128.6, 128.4, 128.3, 128.3, 127.7, 127.1, 126.8, 125.3, 81.0, 76.5, 71.6, 69.4, 52.2, 34.7, 31.2, 26.9, 26.0 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{31}\text{H}_{37}\text{NO}_5\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  526.2564; found, 526.2567.



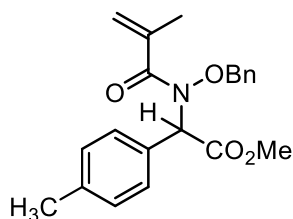
**Methyl (R)-2-((5-(N-(benzyloxy)methacrylamido)pentyl)oxy)-2-phenylacetate (5)**

Yellow oil (51%, 43.4 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45-7.42 (m, 2H), 7.38-7.35 (m, 4H), 7.34-7.30 (m, 4H), 5.57 (s, 1H), 5.17-5.16 (m, 1H), 5.03 (s, 2H), 4.85 (s, 1H), 4.11 (t,  $J = 6.6$  Hz, 2H), 3.70 (s, 3H), 3.53-3.47 (m, 1H), 3.42-3.37 (m, 1H), 1.87 (s, 3H), 1.68-1.61 (m, 4H), 1.48-1.41 (m, 2H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 155.9, 137.7, 136.7, 136.1, 128.6, 128.6, 128.3, 128.3, 127.8, 127.1, 117.9, 81.1, 76.5, 71.7, 69.7, 52.2, 29.8, 29.2, 22.3, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{25}\text{H}_{31}\text{NO}_5\text{Na}^+$   $[\text{M} + \text{Na}]^+$  448.2094; found, 448.2092.



**Methyl 2-(N-(benzyloxy)methacrylamido)-2-phenylacetate (4aa)**

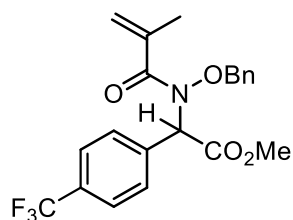
Yellow oil (60%, 40.7 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.40-7.36 (m, 6H), 7.35-7.32 (m, 4H), 5.96 (s, 1H), 5.67 (s, 1H), 5.19 (t,  $J = 1.7$  Hz, 1H), 5.00 (s, 2H), 3.48 (s, 3H), 1.87 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.1, 153.5, 136.9, 135.7, 135.6, 129.0, 128.8, 128.6, 128.4, 128.1, 127.4, 119.1, 81.0, 52.1, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{20}\text{H}_{21}\text{NO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  362.1363; found, 362.1367.



**Methyl 2-(N-(benzyloxy)methacrylamido)-2-(p-tolyl)acetate (4ba)**

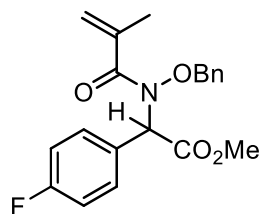
Yellow oil (55%, 38.8 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38-7.32 (m, 5H), 7.28 (d,  $J = 7.9$  Hz, 2H), 7.13 (d,  $J = 7.8$  Hz, 2H), 5.93 (s, 1H), 5.66 (s, 1H), 5.18 (s, 1H), 4.99 (s, 2H), 3.47 (s, 3H), 2.33 (s, 3H), 1.86 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 153.6, 138.9, 136.9, 135.7, 132.7, 129.3, 128.8, 128.3, 128.0, 127.4, 119.1, 80.8,

77.0, 52.1, 21.3, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for C<sub>21</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> [M + Na]<sup>+</sup> 376.1519; found, 376.1523.



**Methyl 2-(N-(benzyloxy)methacrylamido)-2-(4-(trifluoromethyl)phenyl)acetate (4ca)**

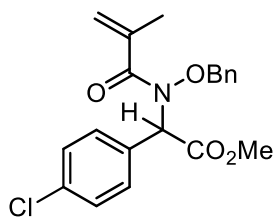
Yellow oil (51%, 41.5 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.57 (d, *J* = 8.3 Hz, 2H), 7.51 (d, *J* = 8.2 Hz, 2H), 7.37-7.33 (m, 5H), 6.01 (s, 1H), 5.70 (s, 1H), 5.23 (t, *J* = 1.6 Hz, 1H), 4.98 (d, *J* = 1.7 Hz, 2H), 3.50 (s, 3H), 1.88 (s, 3H) ppm. **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 169.5, 153.1, 139.5, 136.7, 135.5, 131.2, 130.9, 128.9, 128.4, 128.2, 127.6, 125.5 (q, *J*<sub>CF</sub> = 3.8 Hz), 119.2, 80.2, 77.2, 52.4, 18.8 ppm. **<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -62.72 (3F, s) ppm. **HRMS** (ESI) (m/z): calculated for C<sub>21</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>4</sub>Na<sup>+</sup> [M + Na]<sup>+</sup> 430.1237; found, 430.1234.



**Methyl 2-(N-(benzyloxy)methacrylamido)-2-(4-fluorophenyl)acetate (4da)**

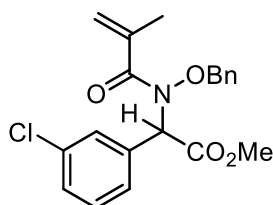
Yellow oil (54%, 38.6 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.38-7.34 (m, 7H), 7.02-6.97 (m, 2H), 5.95 (s, 1H), 5.66 (s, 1H), 5.19 (t, *J* = 1.6 Hz, 1H), 4.99 (s, 2H), 3.49 (s, 3H), 1.86 (s, 3H) ppm. **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 169.9, 163.0 (d, *J*<sub>CF</sub> = 248.4 Hz), 153.3, 136.8, 135.6, 131.5 (d, *J*<sub>CF</sub> = 3.3 Hz), 129.3 (d, *J*<sub>CF</sub> = 8.5 Hz), 128.9, 128.4, 128.2, 119.1, 115.6 (d, *J*<sub>CF</sub> = 21.6 Hz), 80.1, 77.1, 52.2, 18.8 ppm. **<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -112.37 (1F, s) ppm. **HRMS** (ESI) (m/z): calculated for C<sub>20</sub>H<sub>20</sub>FNO<sub>4</sub>Na<sup>+</sup> [M + Na]<sup>+</sup> 380.1269; found, 380.1268.





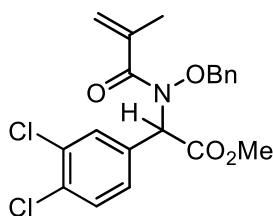
**Methyl 2-(*N*-(benzyloxy)methacrylamido)-2-(4-chlorophenyl)acetate (4ea)**

Yellow oil (56%, 41.8 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.37-7.35 (m, 5H), 7.33-7.29 (m, 4H), 5.93 (s, 1H), 5.66 (s, 1H), 5.20 (t,  $J = 1.6$  Hz, 1H), 4.98 (s, 2H), 3.49 (s, 3H), 1.87 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.7, 153.2, 136.7, 135.6, 135.0, 134.2, 128.9, 128.8, 128.7, 128.4, 128.2, 119.1, 80.1, 77.1, 52.2, 18.8 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{20}\text{H}_{20}\text{ClNO}_4\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  396.0973; found, 396.0976.



**Methyl 2-(*N*-(benzyloxy)methacrylamido)-2-(3-chlorophenyl)acetate (4fa)**

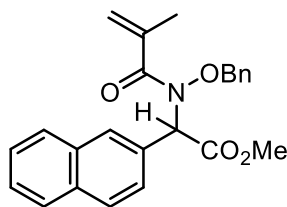
Yellow oil (44%, 32.8 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34-7.34 (m, 6H), 7.33-7.27 (m, 3H), 5.93 (s, 1H), 5.68 (s, 1H), 5.22 (t,  $J = 1.6$  Hz, 1H), 4.98 (d,  $J = 1.8$  Hz, 2H), 3.50 (s, 3H), 1.88 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.6, 153.2, 137.6, 136.7, 135.5, 134.4, 129.9, 129.2, 128.9, 128.4, 128.2, 127.5, 125.4, 119.2, 80.1, 77.1, 52.3, 18.8 ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{20}\text{H}_{20}\text{ClNO}_4\text{Na}^+$  [ $\text{M} + \text{Na}$ ] $^+$  396.0973; found, 396.0974.



**Methyl 2-(*N*-(benzyloxy)methacrylamido)-2-(3,4-dichlorophenyl)acetate (4ga)**

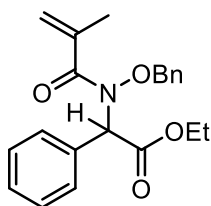
Yellow oil (46%, 37.5 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.48 (d,  $J = 2.0$  Hz, 1H), 7.39-7.33 (m, 6H), 7.22 (dd,  $J = 8.3, 2.1$  Hz, 1H), 5.90 (s, 1H), 5.69 (s, 1H), 5.23 (t,  $J = 1.7$  Hz, 1H), 4.97 (d,  $J = 2.4$  Hz, 2H), 3.51 (s, 3H), 1.88 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100

MHz, CDCl<sub>3</sub>)  $\delta$  169.3, 153.0, 136.6, 135.8, 135.5, 133.2, 132.7, 130.6, 129.3, 128.9, 128.4, 128.3, 126.5, 119.2, 79.5, 77.2, 52.4, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for C<sub>20</sub>H<sub>19</sub>Cl<sub>2</sub>NO<sub>4</sub>Na<sup>+</sup> [M + Na]<sup>+</sup> 430.0583; found, 430.0581.



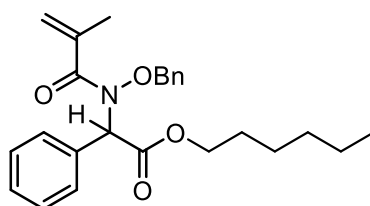
**Methyl 2-(N-(benzyloxy)methacrylamido)-2-(naphthalen-2-yl)acetate (4ha)**

Yellow oil (47%, 36.6 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.83-7.78 (m, 4H), 7.55-7.48 (m, 3H), 7.41-7.33 (m 5H), 6.12 (s, 1H), 5.70 (s, 1H), 5.18 (t, *J* = 1.6 Hz, 1H), 5.01 (d, *J* = 2.2 Hz, 2H), 3.49 (s, 3H), 1.87 (s, 3H) ppm. **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.1, 153.6, 136.9, 135.6, 133.5, 133.0, 133.0, 128.8, 128.5, 128.4, 128.3, 128.1, 127.7, 127.2, 126.6, 126.4, 124.5, 119.2, 81.1, 77.1, 52.2, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for C<sub>24</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> [M + Na]<sup>+</sup> 412.1519; found, 412.1522.



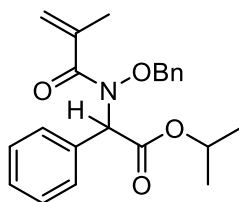
**Ethyl 2-(N-(benzyloxy)methacrylamido)-2-phenylacetate (4ia)**

Yellow oil (50%, 35.3 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.42-7.40 (m, 2H), 7.38-7.35 (m, 4H), 7.34-7.32 (m, 4H), 5.99 (s, 1H), 5.69 (s, 1H), 5.19 (t, *J* = 1.6 Hz, 1H), 5.00 (s, 2H), 4.04-3.94 (m, 2H), 1.86 (s, 3H), 1.10 (t, *J* = 7.1 Hz, 3H) ppm. **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  169.6, 153.4, 137.1, 135.8, 135.7, 128.9, 128.7, 128.5, 128.3, 128.0, 127.4, 119.1, 81.0, 76.9, 61.2, 18.8, 14.0 ppm. **HRMS** (ESI) (m/z): calculated for C<sub>21</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> [M + Na]<sup>+</sup> 376.1519; found, 376.1523.



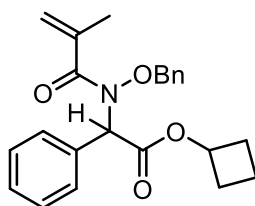
**Hexyl 2-(N-(benzyloxy)methacrylamido)-2-phenylacetate (4ja)**

Yellow oil (40%, 32.7 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.40 (m, 2H), 7.39-7.36 (m, 4H), 7.34-7.31 (m, 4H), 6.02 (s, 1H), 5.71 (s, 1H), 5.19 (t,  $J = 1.6$  Hz, 1H), 4.99 (s, 2H), 4.02-3.95 (m, 1H), 3.88-3.82 (m, 1H), 1.86 (s, 3H), 1.45 (q,  $J = 6.9$  Hz, 2H), 1.28-1.25 (m, 1H), 1.22-1.20 (m, 1H), 1.17-1.12 (m, 4H), 0.83 (t,  $J = 6.9$  Hz, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.7, 153.4, 137.1, 135.9, 135.8, 128.9, 128.6, 128.5, 128.3, 128.0, 127.4, 119.0, 81.0, 76.9, 65.3, 31.3, 28.3, 25.3, 22.5, 18.8, 14.0 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{25}\text{H}_{31}\text{NO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  432.2145; found, 432.2142.



**Isopropyl 2-(*N*-(benzyloxy)methacrylamido)-2-phenylacetate (4ka)**

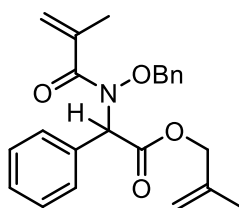
Yellow oil (55%, 40.4 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.36 (m, 6H), 7.34-7.31 (m, 4H), 6.02 (s, 1H), 5.72 (s, 1H), 5.19 (s, 1H), 5.00 (s, 2H), 4.97-4.91 (m, 1H), 1.86 (s, 3H), 1.14 (d,  $J = 6.3$  Hz, 3H), 1.05 (d,  $J = 6.3$  Hz, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.1, 153.3, 137.3, 135.9, 135.8, 128.8, 128.5, 128.5, 128.3, 127.9, 127.4, 119.0, 81.0, 76.8, 69.0, 21.6, 21.4, 18.8 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{22}\text{H}_{25}\text{NO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  390.1676; found, 390.1675.



**Cyclobutyl 2-(*N*-(benzyloxy)methacrylamido)-2-phenylacetate (4la)**

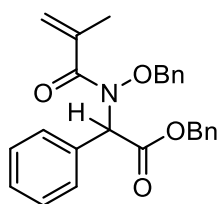
Yellow oil (47%, 35.6 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.39 (m, 2H), 7.38-7.35 (m, 4H), 7.34-7.32 (m, 4H), 6.01 (s, 1H), 5.70 (s, 1H), 5.19 (t,  $J = 1.6$  Hz, 1H), 5.00 (s, 2H), 4.84 (p,  $J = 7.4$  Hz, 1H), 2.26-2.19 (m, 2H), 1.98-1.89 (m, 1H), 1.86 (s, 3H), 1.86-1.79 (m, 1H), 1.73-1.65 (m, 1H), 1.57-1.49 (m, 1H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.9, 153.3, 137.2, 135.8, 135.7, 128.9, 128.6, 128.5, 128.3, 128.0, 127.5, 119.0, 80.8, 76.9, 69.7, 30.0, 30.0, 18.8, 13.5 ppm. **HRMS** (ESI) (m/z):

calculated for  $C_{23}H_{25}NO_4Na^+$   $[M + Na]^+$  402.1676; found, 402.1678.



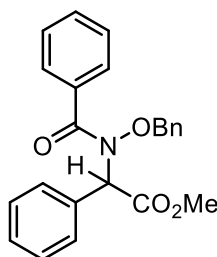
**2-Methylallyl 2-(N-(benzyloxy)methacrylamido)-2-phenylacetate (4ma)**

Yellow oil (33%, 25.0 mg).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.43-7.40 (m, 2H), 7.38-7.36 (m, 4H), 7.34-7.32 (m, 4H), 6.06 (s, 1H), 5.72 (s, 1H), 5.20 (t,  $J = 1.6$  Hz, 1H), 4.99 (d,  $J = 1.8$  Hz, 2H), 4.76 (d,  $J = 19.3$  Hz, 2H), 4.44 (d,  $J = 13.2$  Hz, 1H), 4.21 (d,  $J = 13.2$  Hz, 1H), 1.86 (s, 3H), 1.53 (s, 3H) ppm.  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  169.3, 153.3, 139.4, 137.0, 135.8, 135.7, 129.0, 128.7, 128.5, 128.3, 128.0, 127.4, 119.1, 113.0, 81.0, 76.9, 68.1, 19.2, 18.8 ppm. HRMS (ESI) (m/z): calculated for  $C_{23}H_{25}NO_4Na^+$   $[M + Na]^+$  402.1676; found, 402.1678.



**Benzyl 2-(N-(benzyloxy)methacrylamido)-2-phenylacetate (4na)**

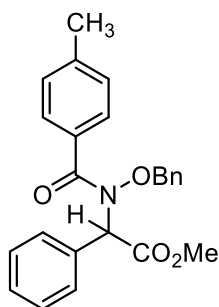
Yellow oil (58%, 48.2 mg).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.41-7.38 (m, 2H), 7.33-7.30 (m, 8H), 7.26-7.25 (m, 3H), 7.15-7.11 (m, 2H), 6.03 (s, 1H), 5.67 (s, 1H), 5.17 (t,  $J = 1.7$  Hz, 1H), 5.00-4.89 (m, 4H), 1.85 (s, 3H) ppm.  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  169.4, 153.4, 137.1, 135.7, 135.6, 135.4, 129.0, 128.7, 128.5, 128.4, 128.3, 128.1, 128.0, 128.0, 127.4, 119.1, 81.0, 76.8, 66.7, 18.8 ppm. HRMS (ESI) (m/z): calculated for  $C_{26}H_{25}NO_4Na^+$   $[M + Na]^+$  438.1676; found, 438.1675.



**Methyl 2-(N-(benzyloxy)benzamido)-2-phenylacetate (4ab)**

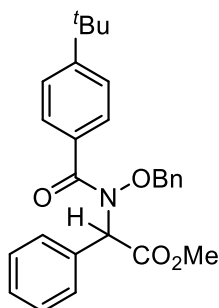
Yellow oil (51%, 38.3 mg).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.79-7.76 (m, 2H), 7.49-

7.46 (m, 2H), 7.44-7.39 (m, 3H), 7.38-7.35 (m, 6H), 7.33-7.31 (m, 2H), 6.24 (s, 1H), 5.08 (s, 2H), 3.51 (s, 3H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.9, 151.9, 137.0, 135.5, 131.3, 130.0, 129.1, 128.7, 128.7, 128.4, 128.1, 128.1, 127.4, 127.0, 81.2, 52.2 ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{23}\text{H}_{21}\text{NO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  398.1363; found, 398.1366.



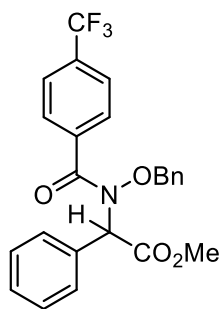
**Methyl 2-(*N*-(benzyloxy)-4-methylbenzamido)-2-phenylacetate (4ac)**

Yellow oil (43%, 33.5 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d,  $J = 8.2$  Hz, 2H), 7.48-7.45 (m, 2H), 7.43-7.39 (m, 3H), 7.38-7.34 (m, 5H), 7.11 (d,  $J = 8.0$  Hz, 2H), 6.20 (s, 1H), 5.06 (s, 2H), 3.51 (s, 3H), 2.34 (s, 3H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.0, 152.3, 140.2, 137.1, 135.6, 129.1, 128.9, 128.7, 128.7, 128.4, 128.4, 128.0, 127.4, 127.0, 81.1, 52.2, 21.4 ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{24}\text{H}_{23}\text{NO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  412.1519; found, 412.1520.



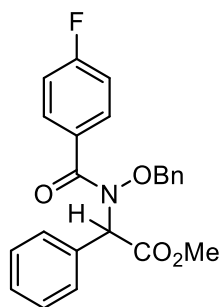
**Methyl 2-(*N*-(benzyloxy)-4-(tert-butyl)benzamido)-2-phenylacetate (4ad)**

Yellow oil (59%, 50.9 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 (d,  $J = 8.5$  Hz, 2H), 7.49-7.47 (m, 2H), 7.43-7.41 (m, 2H), 7.39-7.32 (m, 8H), 6.21 (s, 1H), 5.06 (s, 2H), 3.51 (s, 3H), 1.29 (s, 9H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.0, 153.3, 152.2, 137.2, 135.7, 129.1, 128.7, 128.7, 128.4, 128.4, 128.0, 127.4, 126.8, 125.1, 81.1, 77.0, 52.2, 34.7, 31.2 ppm. HRMS (ESI) (m/z): calculated for  $\text{C}_{27}\text{H}_{29}\text{NO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  454.1989; found, 454.1989.



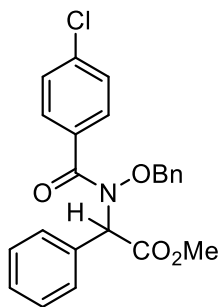
**Methyl 2-(*N*-(benzyloxy)-4-(trifluoromethyl)benzamido)-2-phenylacetate (4ae)**

Yellow oil (45%, 39.9 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (d,  $J = 8.2$  Hz, 2H), 7.57 (d,  $J = 8.3$  Hz, 2H), 7.47-7.44 (m, 2H), 7.43-7.39 (m, 4H), 7.38-7.34 (m, 4H), 6.33 (s, 1H), 5.09 (s, 2H), 3.53 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.7, 150.4, 136.7, 135.2, 135.0, 131.8, 131.5, 129.4, 128.8, 128.8, 128.5, 128.3, 127.4, 127.2, 125.1 (q,  $J_{\text{CF}} = 4.0$  Hz), 81.3, 77.5, 52.3 ppm.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.80 (3F, s) ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{24}\text{H}_{20}\text{F}_3\text{NO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  466.1237; found, 466.1240.



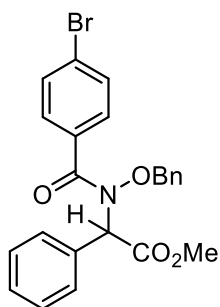
**Methyl 2-(*N*-(benzyloxy)-4-fluorobenzamido)-2-phenylacetate (4af)**

Yellow oil (37%, 29.1 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80-7.75 (m, 2H), 7.47-7.44 (m, 2H), 7.43-7.39 (m, 3H), 7.38-7.35 (m, 5H), 7.02-6.96 (m, 2H), 6.27 (s, 1H), 5.06 (s, 2H), 3.52 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.9, 164.0 (d,  $J_{\text{CF}} = 249.2$  Hz), 151.1, 136.9, 135.4, 129.2, 129.1 (d,  $J_{\text{CF}} = 8.7$  Hz), 128.8, 128.7, 128.4, 128.1, 127.5 (d,  $J_{\text{CF}} = 3.2$  Hz), 127.4, 115.2 (d,  $J_{\text{CF}} = 21.7$  Hz), 81.2, 77.2, 52.3 ppm.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -110.74 (1F, s) ppm. **HRMS** (ESI) ( $m/z$ ): calculated for  $\text{C}_{23}\text{H}_{20}\text{FNO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  416.1269; found, 416.1271.



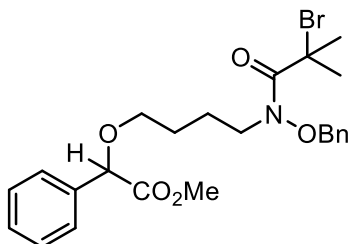
**Methyl 2-(*N*-(benzyloxy)-4-chlorobenzamido)-2-phenylacetate (4ag)**

Yellow oil (54%, 44.2 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75-7.71 (m, 2H), 7.46-7.43 (m, 2H), 7.42-7.38 (m, 4H), 7.37-7.34 (m, 4H), 7.30-7.26 (m, 2H), 6.27 (s, 1H), 5.06 (s, 2H), 3.52 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.8, 150.9, 136.8, 136.1, 135.3, 129.9, 129.3, 128.8, 128.7, 128.4, 128.4, 128.3, 128.2, 127.4, 81.2, 77.3, 52.3 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{23}\text{H}_{20}\text{ClNO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  432.0973; found, 432.0973.



**Methyl 2-(*N*-(benzyloxy)-4-bromobenzamido)-2-phenylacetate (4ah)**

Yellow oil (69%, 62.7 mg).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68-7.64 (m, 2H), 7.46-7.42 (m, 5H), 7.40 (s, 2H), 7.38-7.35 (m, 5H), 6.27 (s, 1H), 5.06 (s, 2H), 3.52 (s, 3H) ppm.  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.8, 151.0, 136.8, 135.3, 131.3, 130.4, 129.3, 128.8, 128.7, 128.5, 128.4, 128.2, 127.4, 124.5, 81.2, 77.3, 52.3 ppm. **HRMS** (ESI) (m/z): calculated for  $\text{C}_{23}\text{H}_{20}\text{BrNO}_4\text{Na}^+$   $[\text{M} + \text{Na}]^+$  476.0468; found, 476.0468.



**Methyl 2-(4-(*N*-(benzyloxy)-2-bromo-2-methylpropanamido)butoxy)-2-**

**phenylacetate (10)**

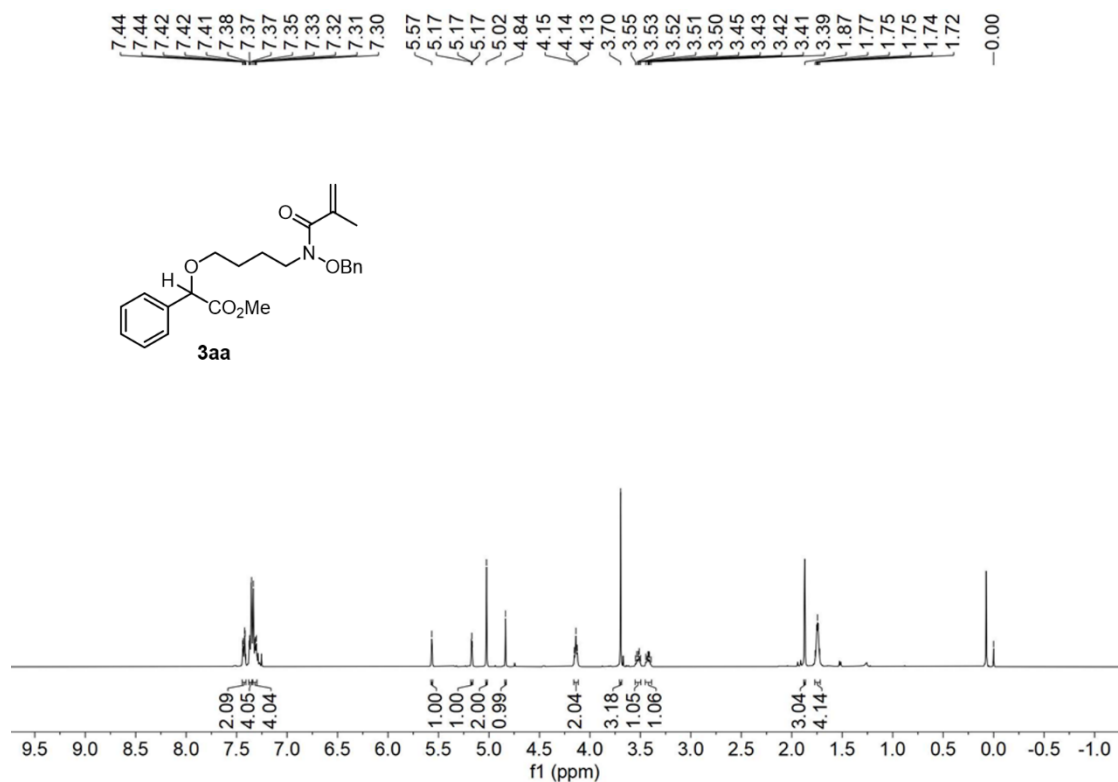
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.45-7.43 (m, 2H), 7.37-7.30 (m, 8H), 4.96 (s, 2H), 4.85 (s, 1H), 4.37-4.34 (m, 2H), 3.69 (s, 3H), 3.57-3.52 (m, 1H), 3.48-3.42 (m, 1H), 1.89 (s, 6H), 1.81-1.75 (m, 4H) ppm. **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.5, 157.4, 137.3, 136.6, 128.7, 128.6, 128.4, 128.4, 128.0, 127.2, 81.0, 73.5, 69.4, 61.0, 52.3, 31.5, 26.7, 25.9 ppm.



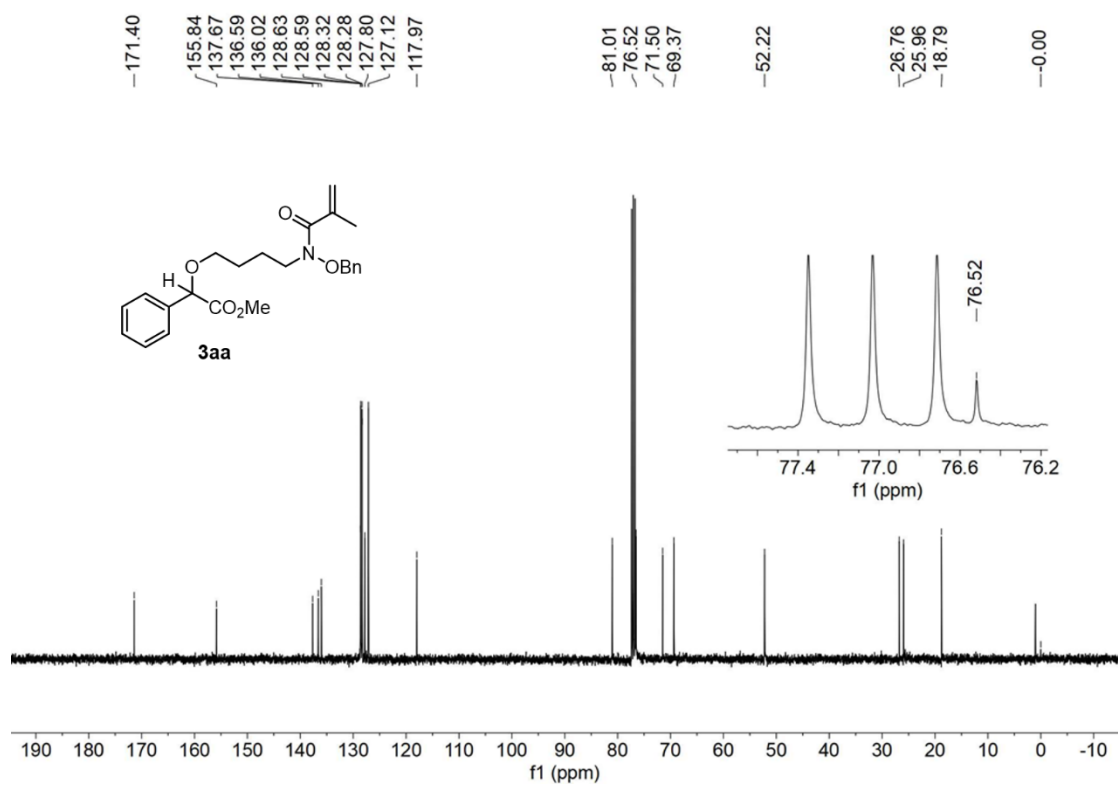
## 5. References

- [1] J. Zheng, J. Qi and S. Cui, Fe-Catalyzed olefin hydroamination with diazo compounds for hydrazone synthesis, *Org. Lett.*, 2016, **18**, 128–131.
- [2] V. Klöpfer, R. Eckl, J. Floß, P. M. C. Roth, O. Reiser and J. P. Barham, Catalyst-free, scalable heterocyclic flow photocyclopropanation, *Green Chem.*, 2021, **23**, 6366–6372.
- [3] M. C. DiPoto and J. Wu, Synthesis of 2-aminoimidazolones and imidazolones by (3 + 2) annulation of azaoxyallyl cations, *Org. Lett.*, 2018, **20**, 499–501.

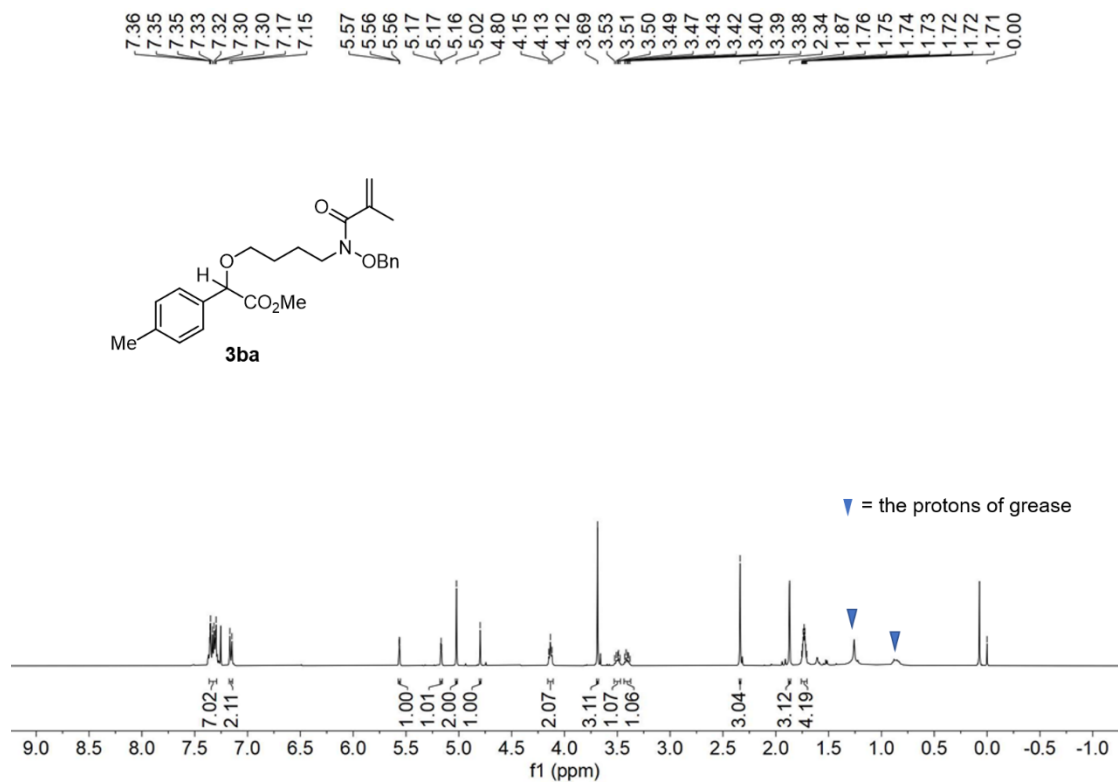
## 6. Copies of $^1\text{H}$ NMR and $^{13}\text{C}$ NMR Spectra



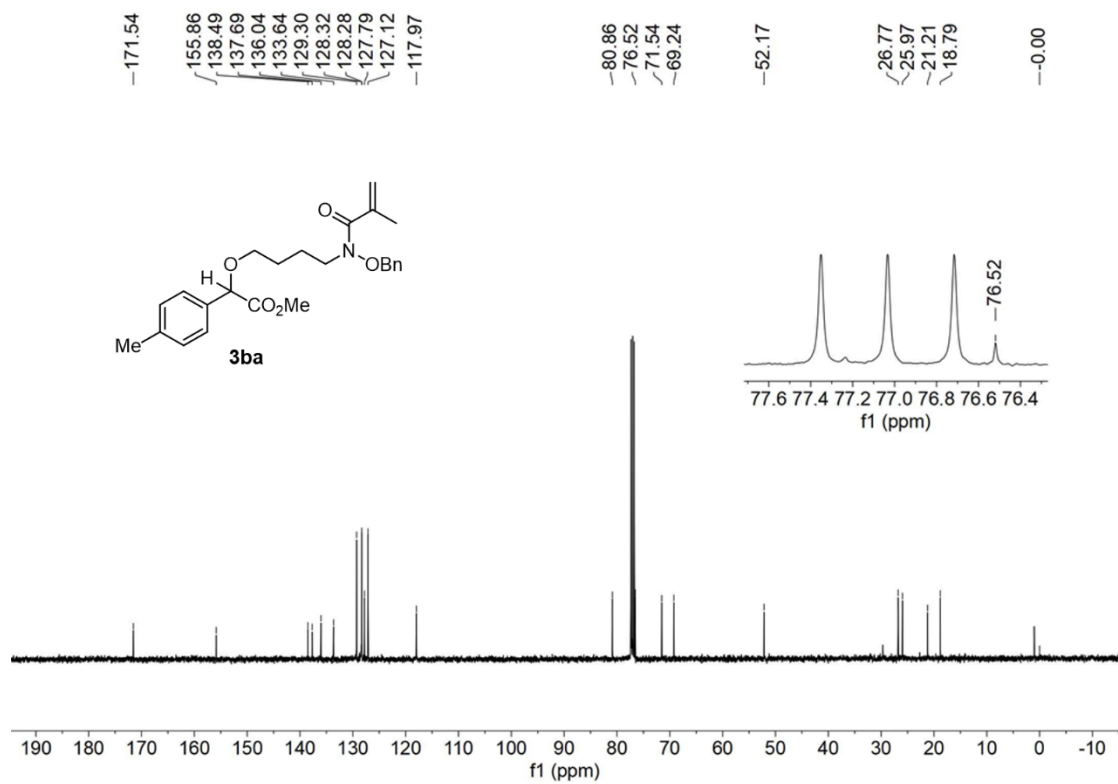
**Fig. S3**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3aa**.



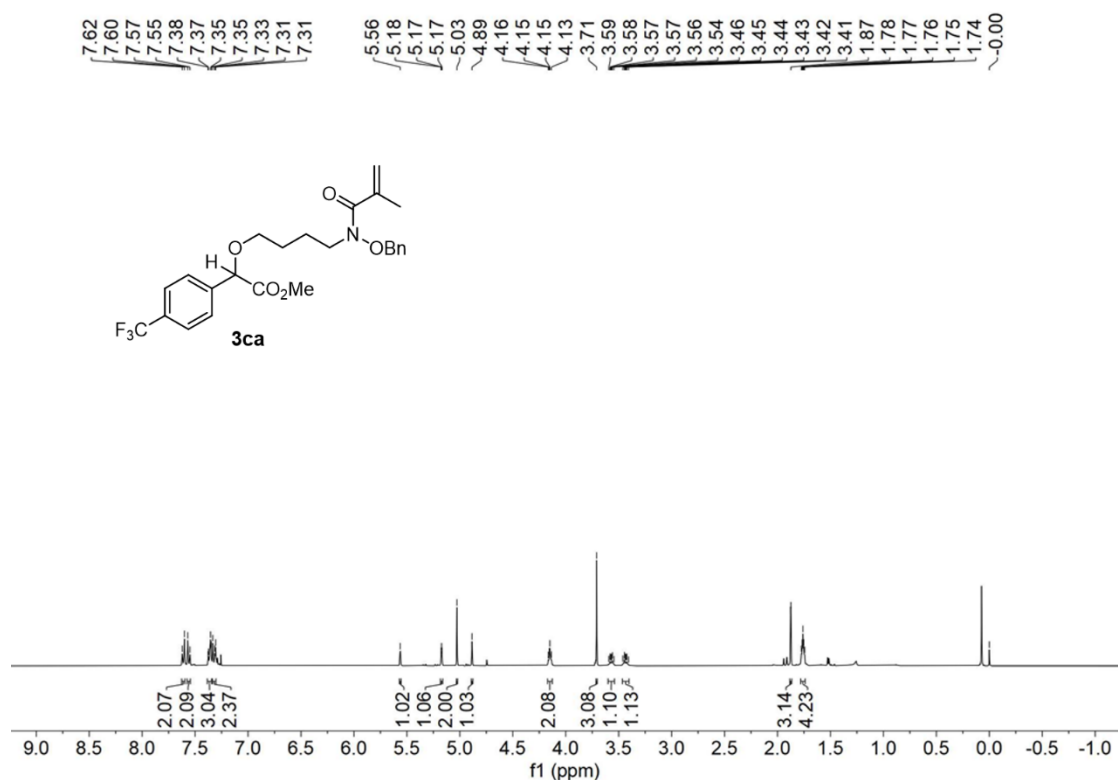
**Fig. S4**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3aa**.



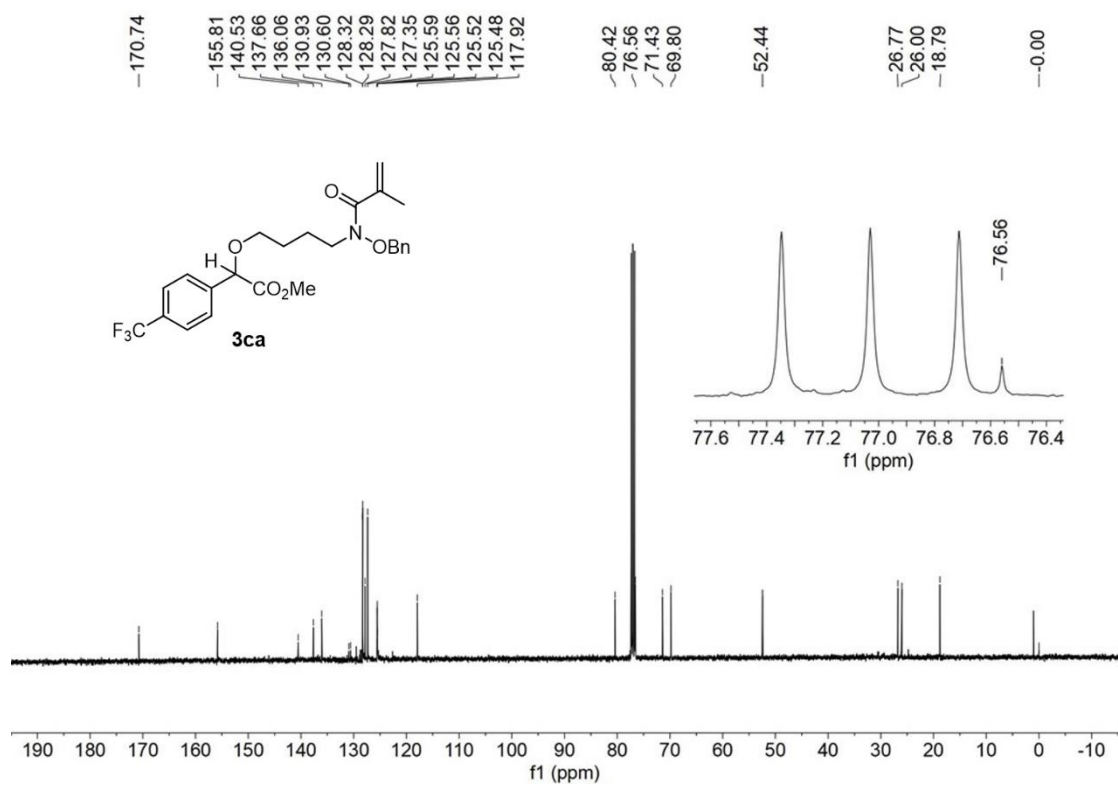
**Fig. S5**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3ba**.



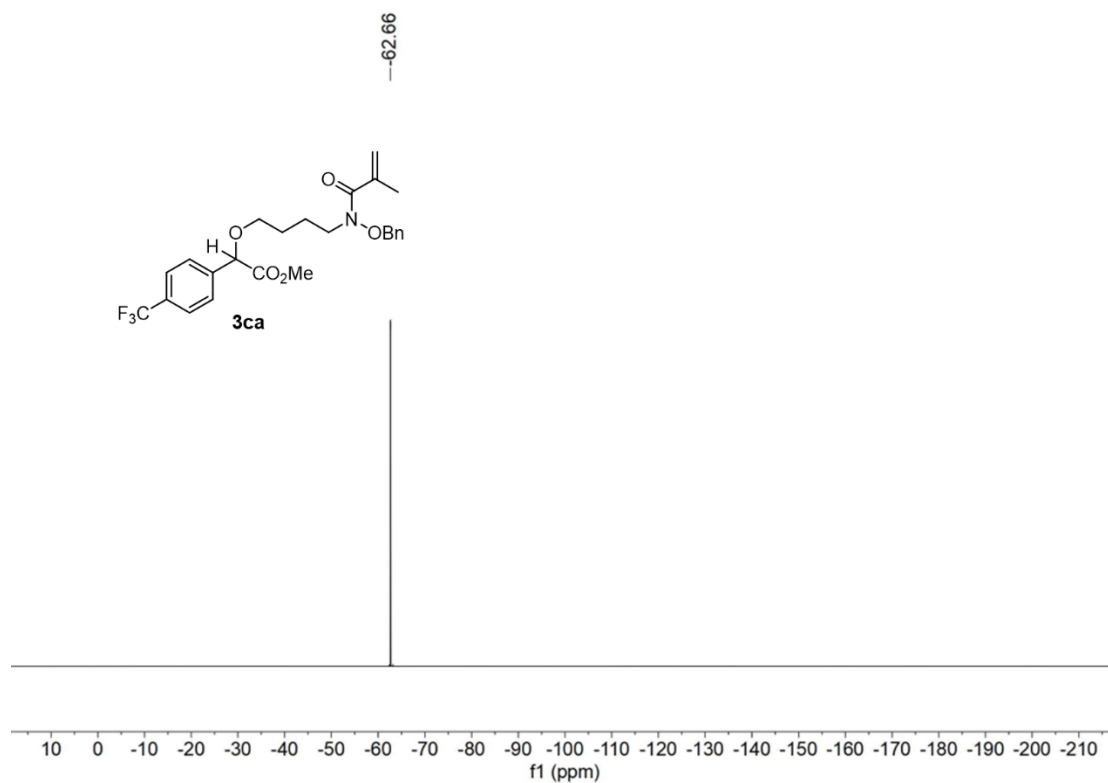
**Fig. S6**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3ba**.



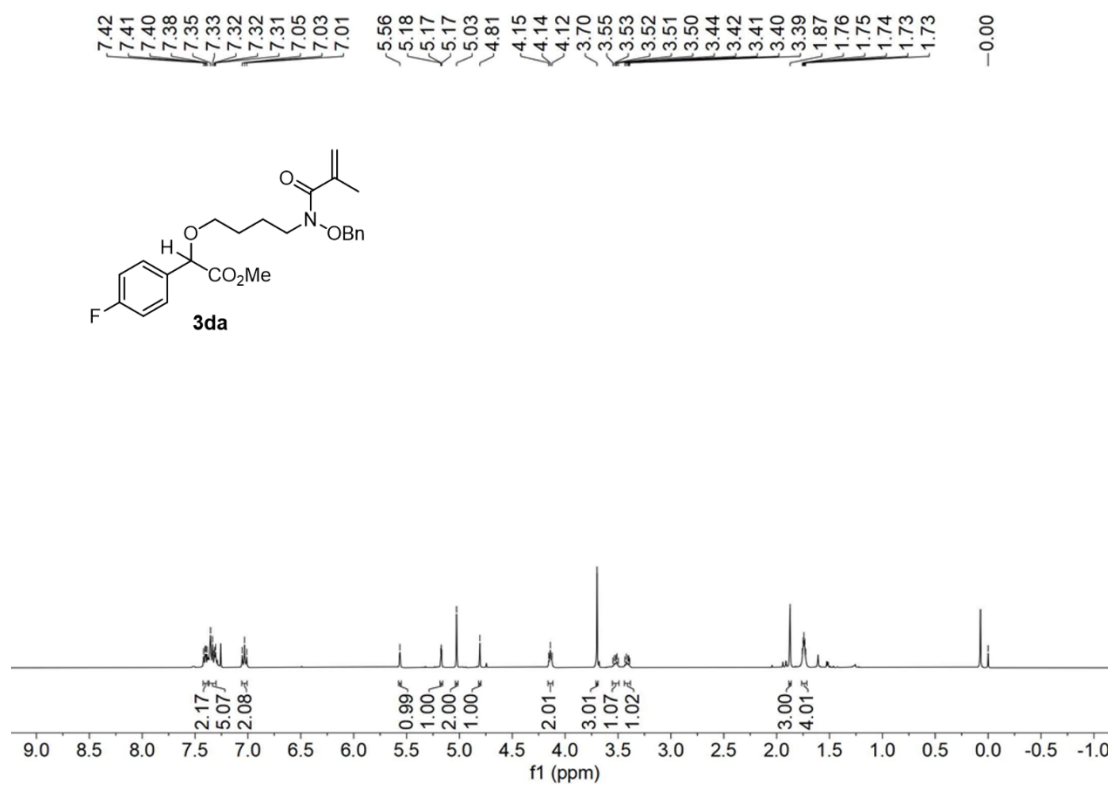
**Fig. S7** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ca**.



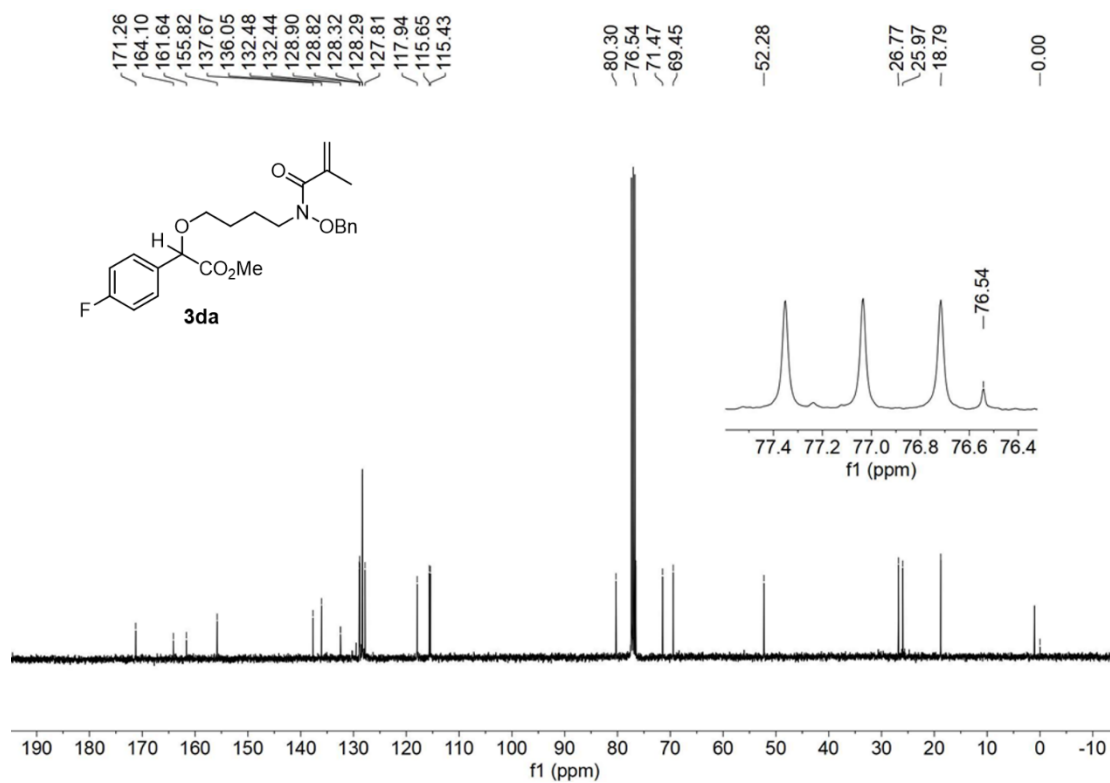
**Fig. S8** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ca**.



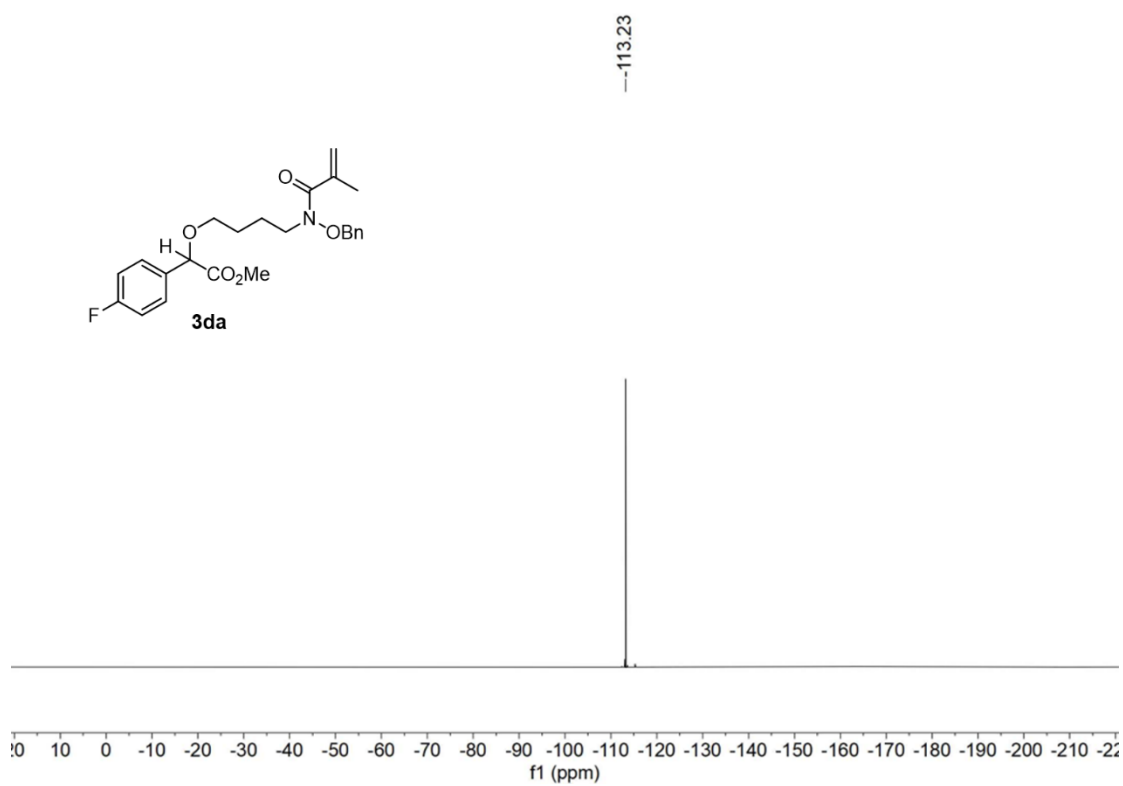
**Fig. S9**  $^{19}\text{F}$  NMR spectrum (376 MHz,  $\text{CDCl}_3$ ) of compound **3ca**.



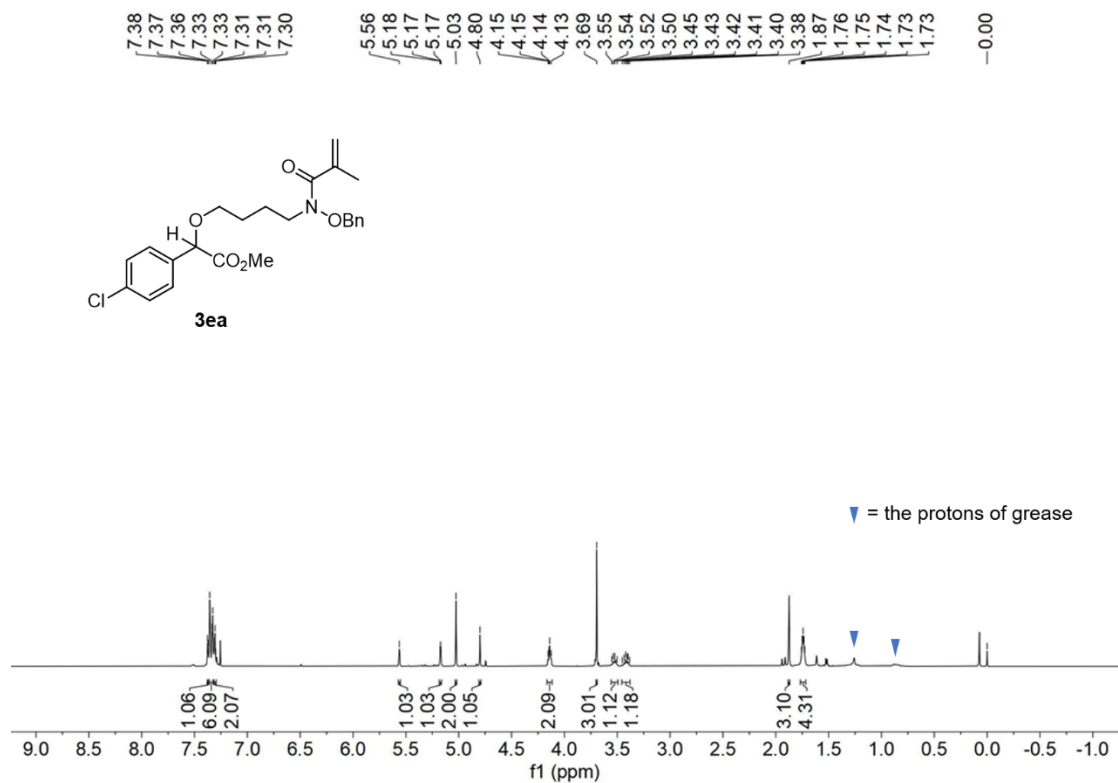
**Fig. S10**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3da**.



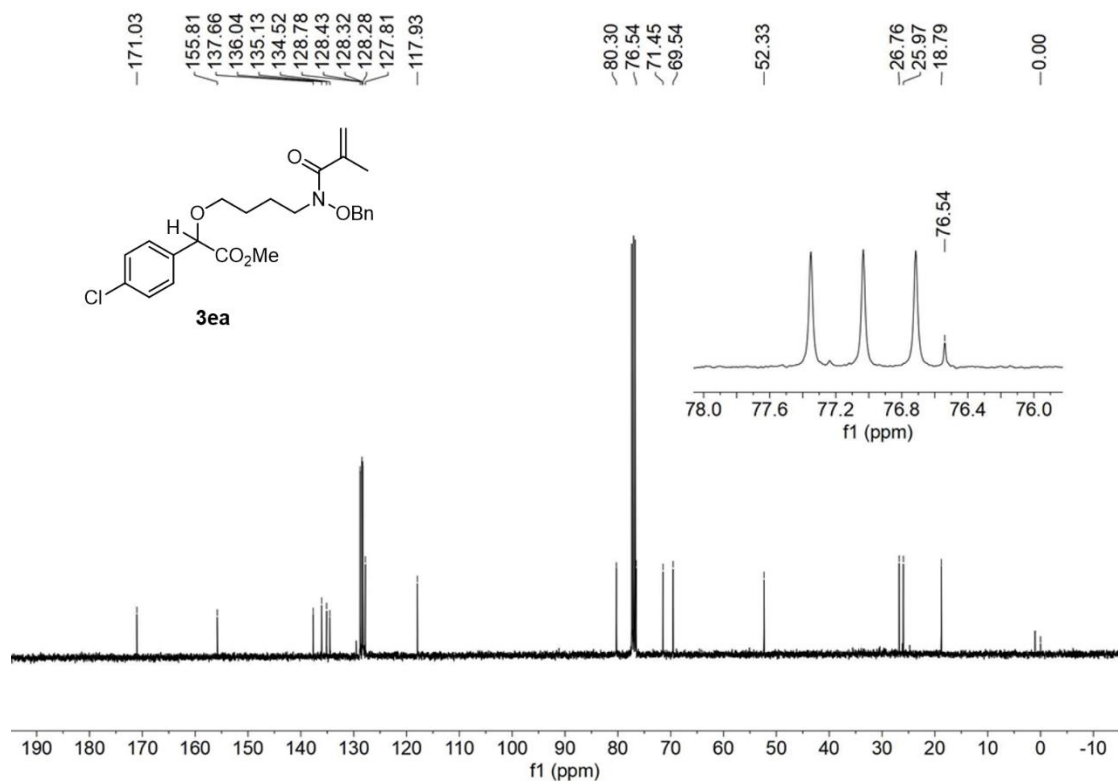
**Fig. S11** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3da**.



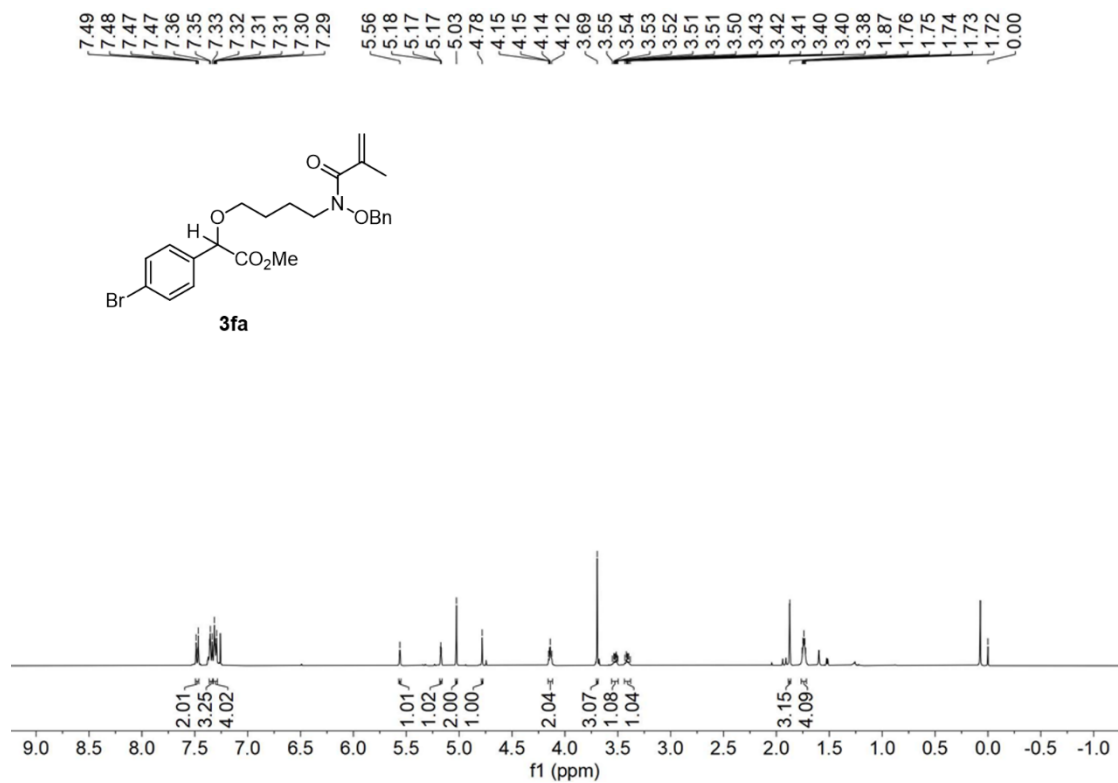
**Fig. S12** <sup>19</sup>F NMR spectrum (376 MHz, CDCl<sub>3</sub>) of compound **3da**.



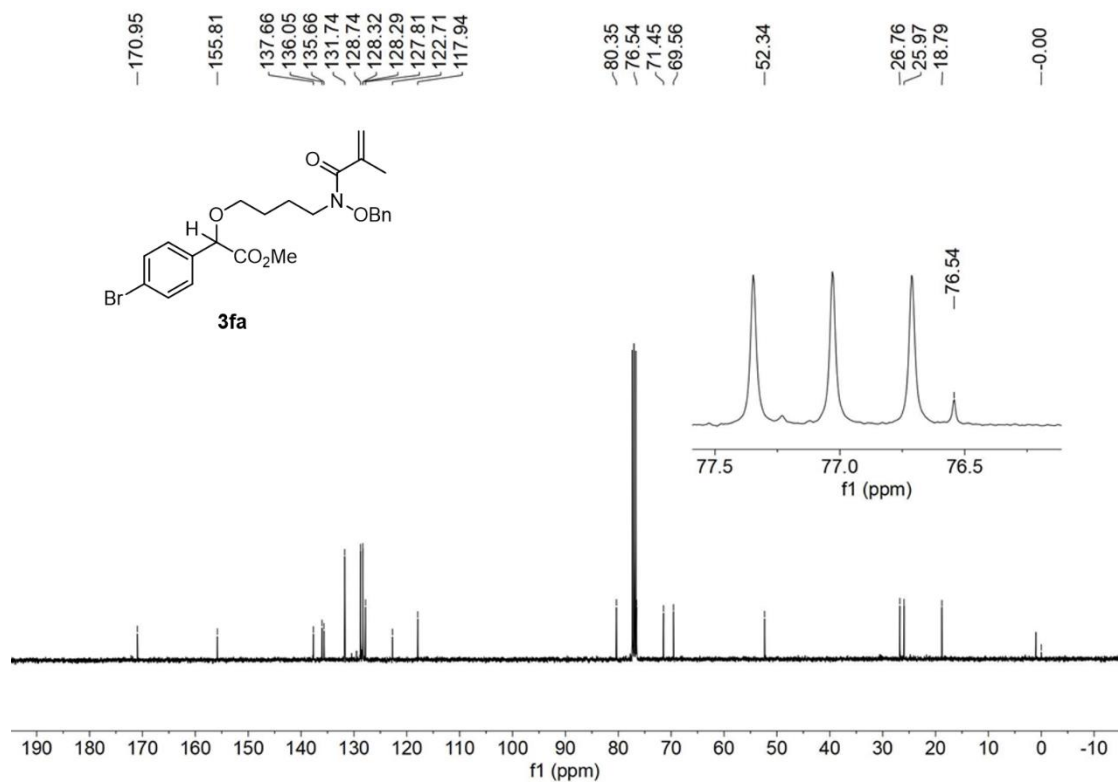
**Fig. S13**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3ea**.



**Fig. S14**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3ea**.

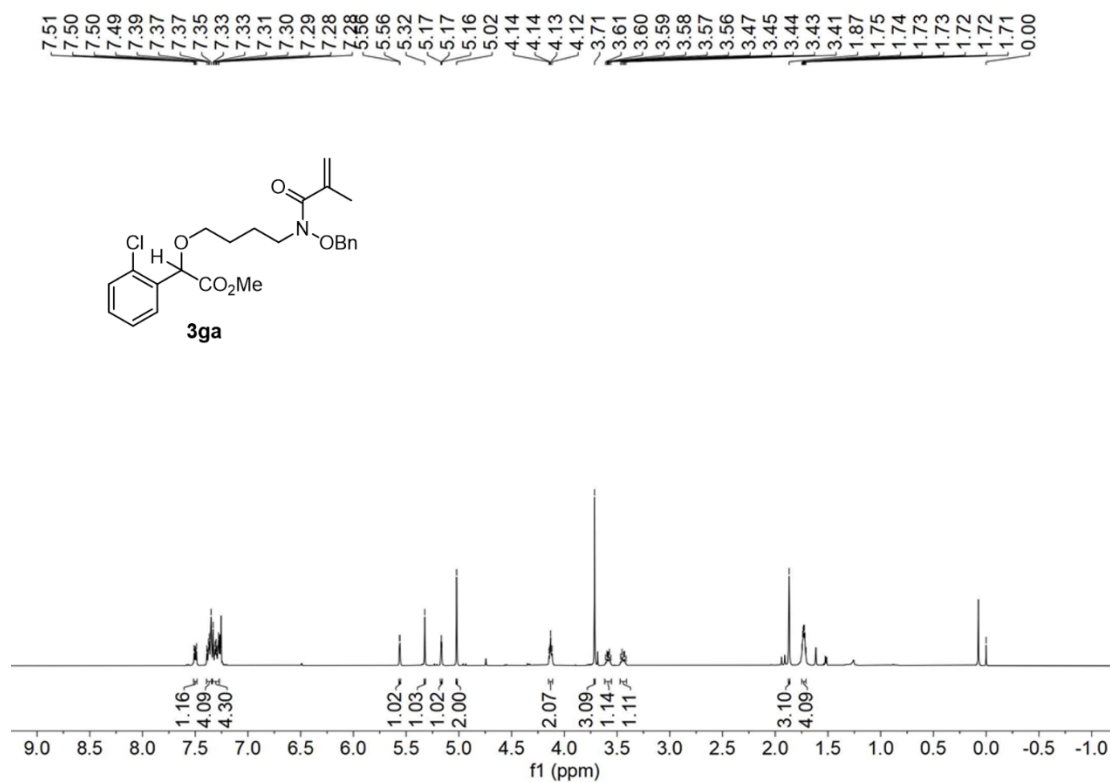


**Fig. S15**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3fa**.

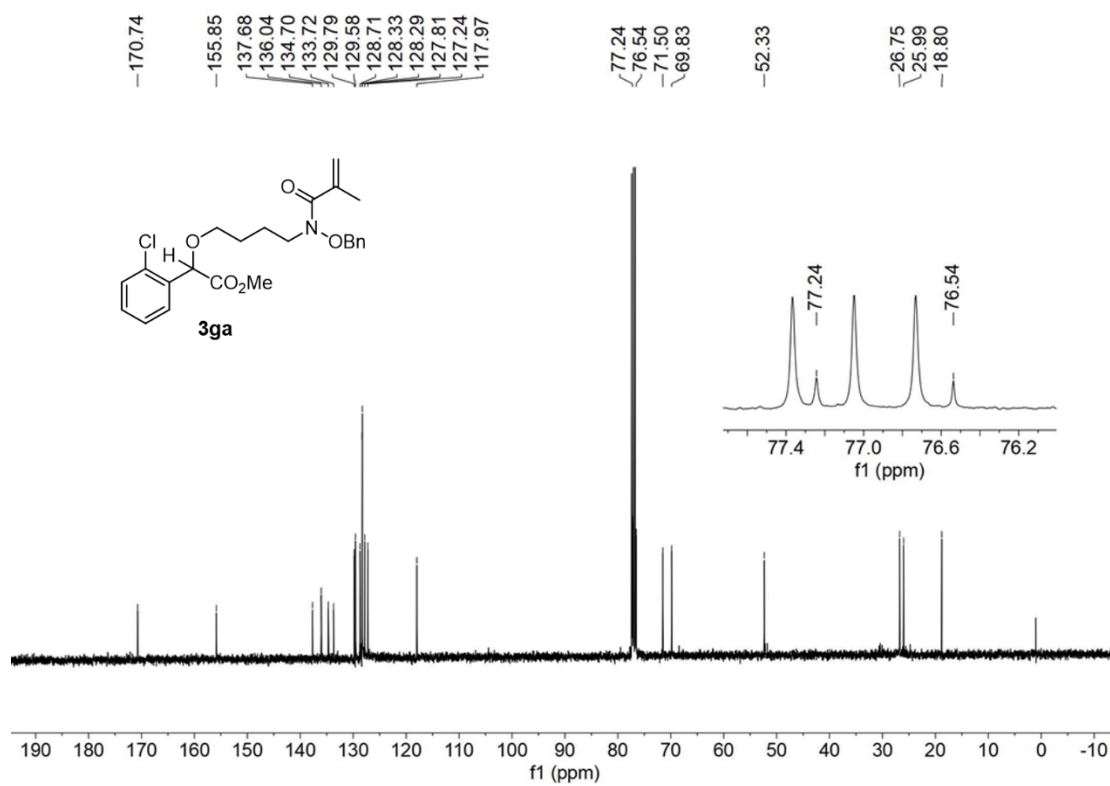


**Fig. S16**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3fa**.

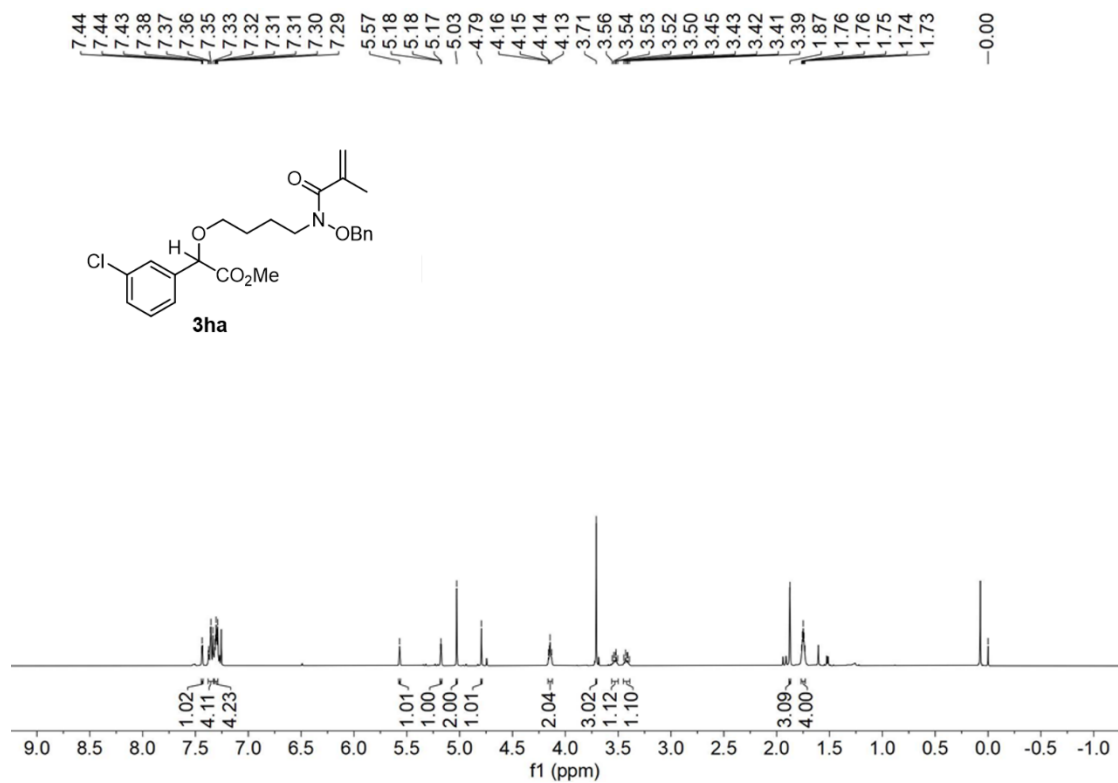




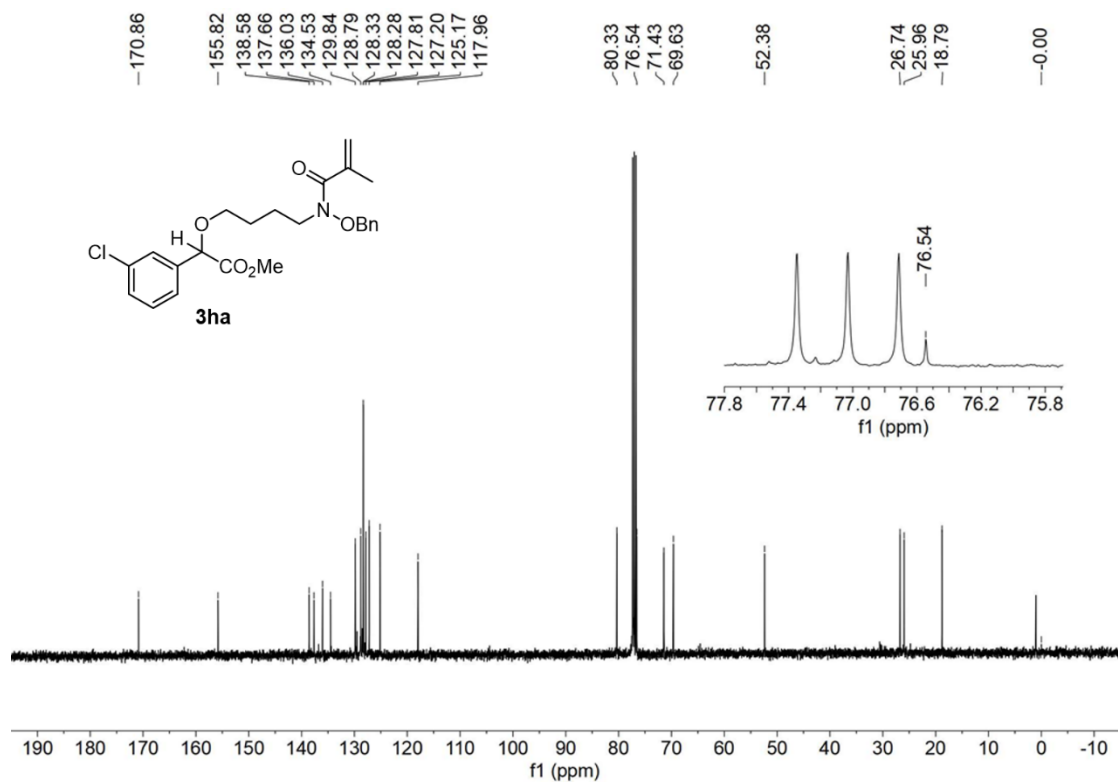
**Fig. S17** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ga**.



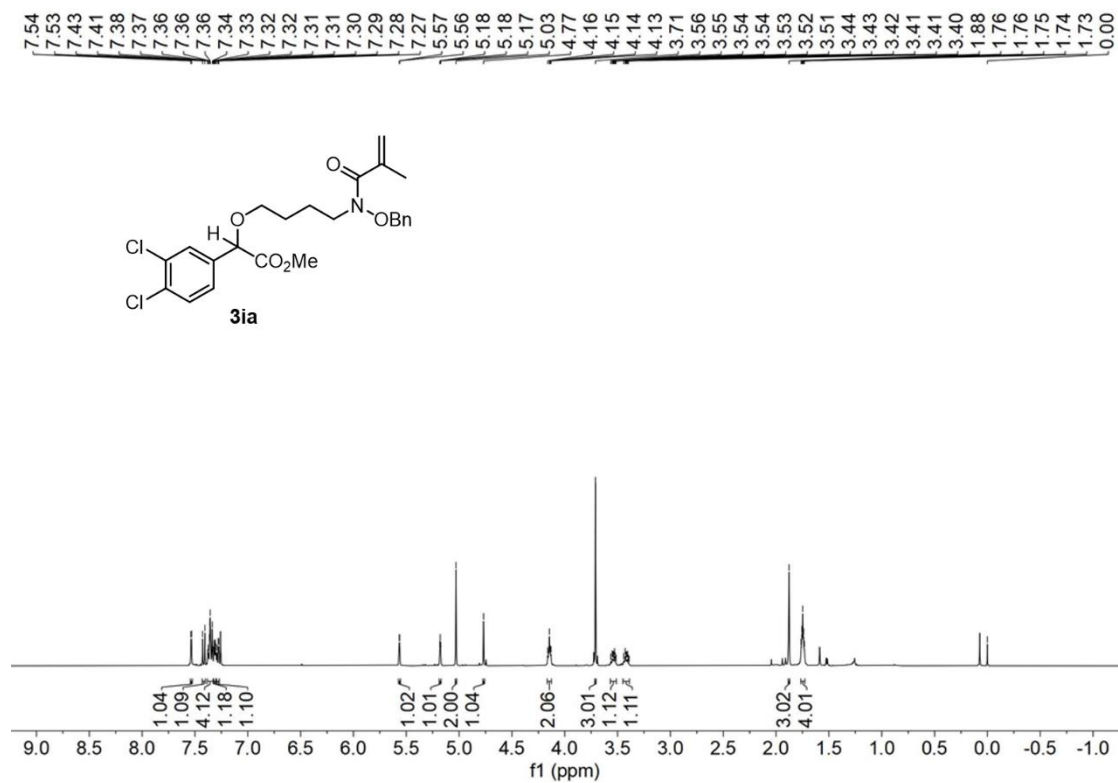
**Fig. S18** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ga**.



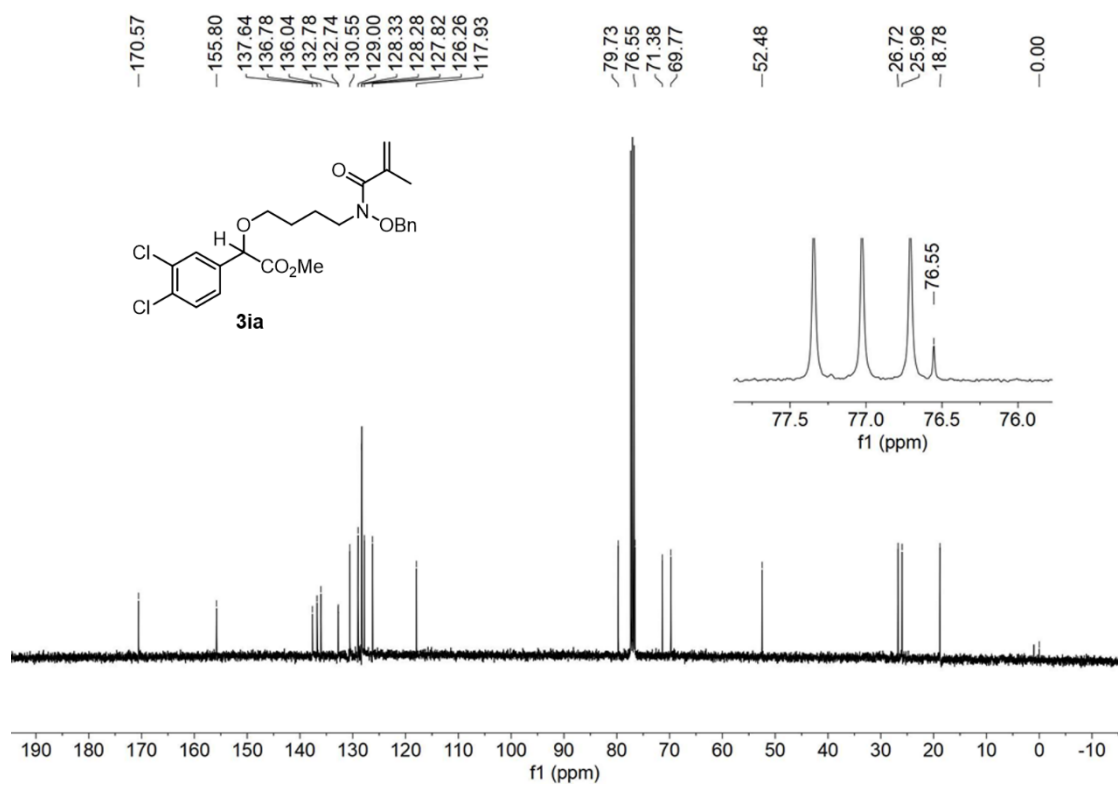
**Fig. S19** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ha**.



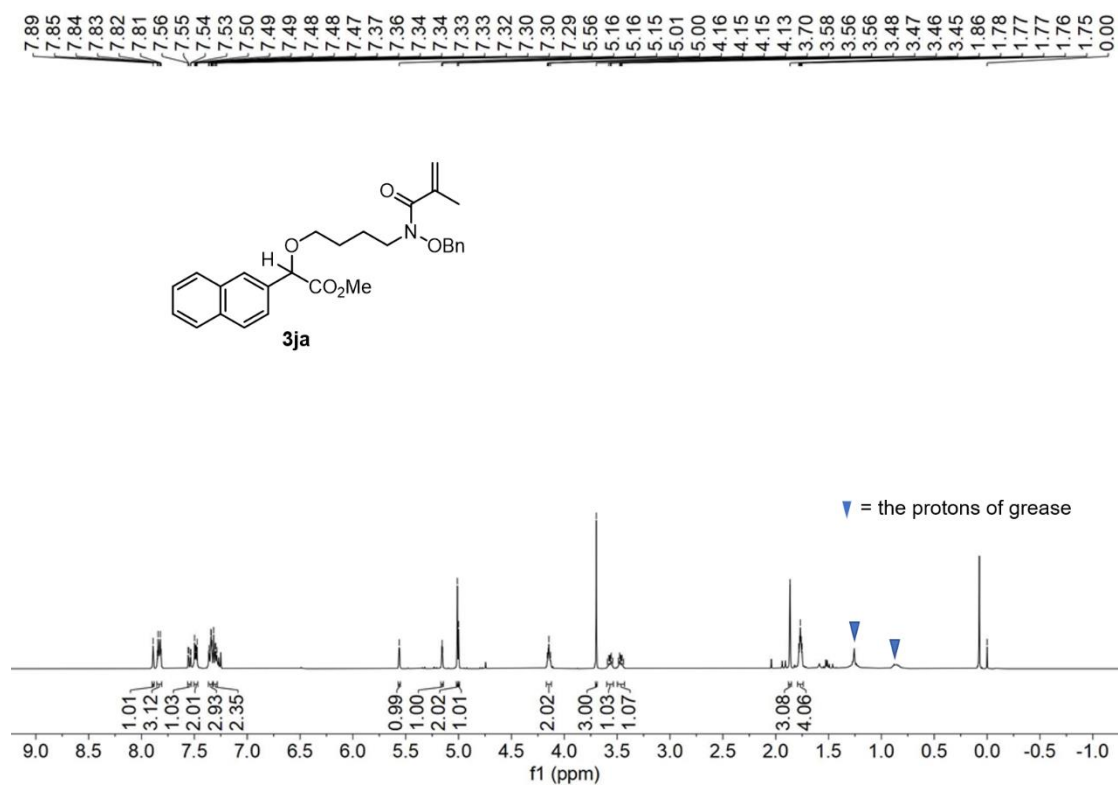
**Fig. S20** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ha**.



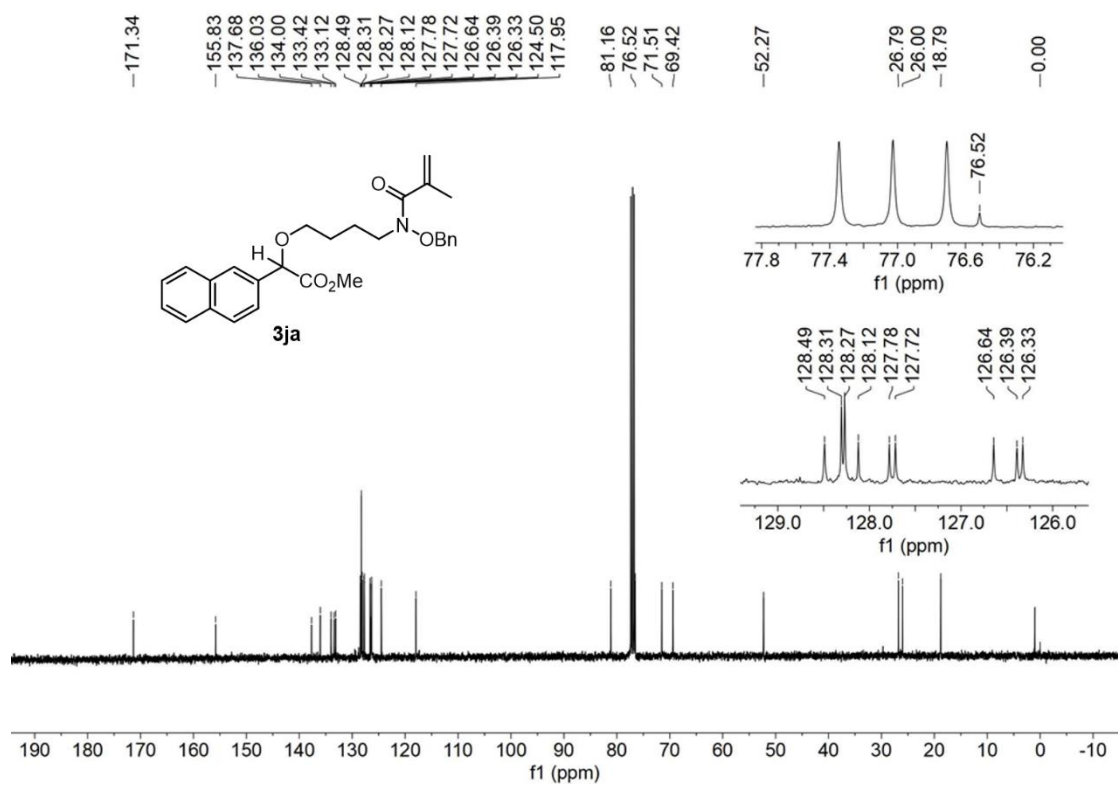
**Fig. S21** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ia**.



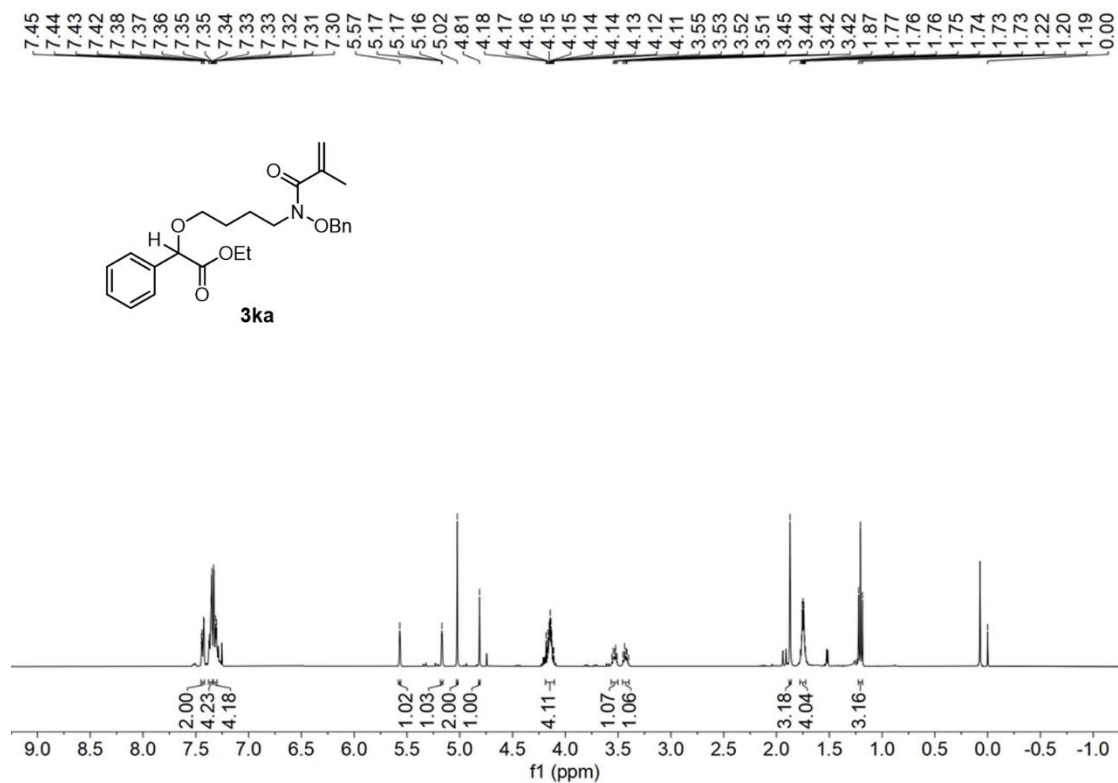
**Fig. S22** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ia**.



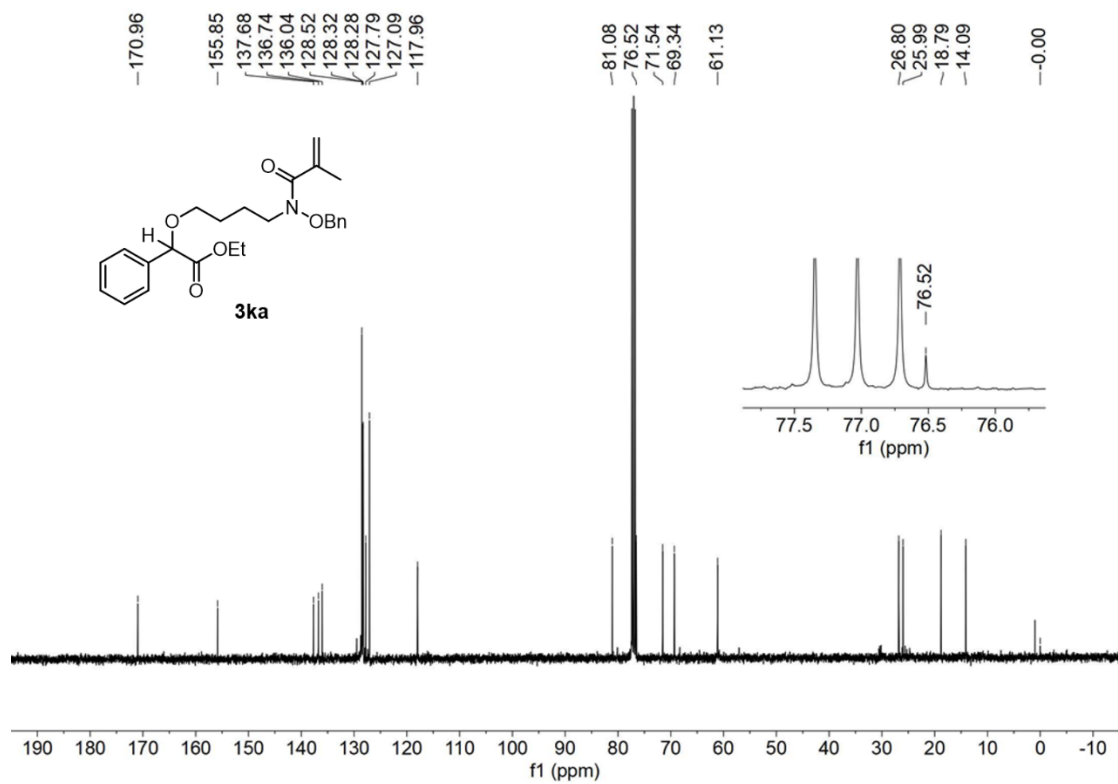
**Fig. S23**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3ja**.



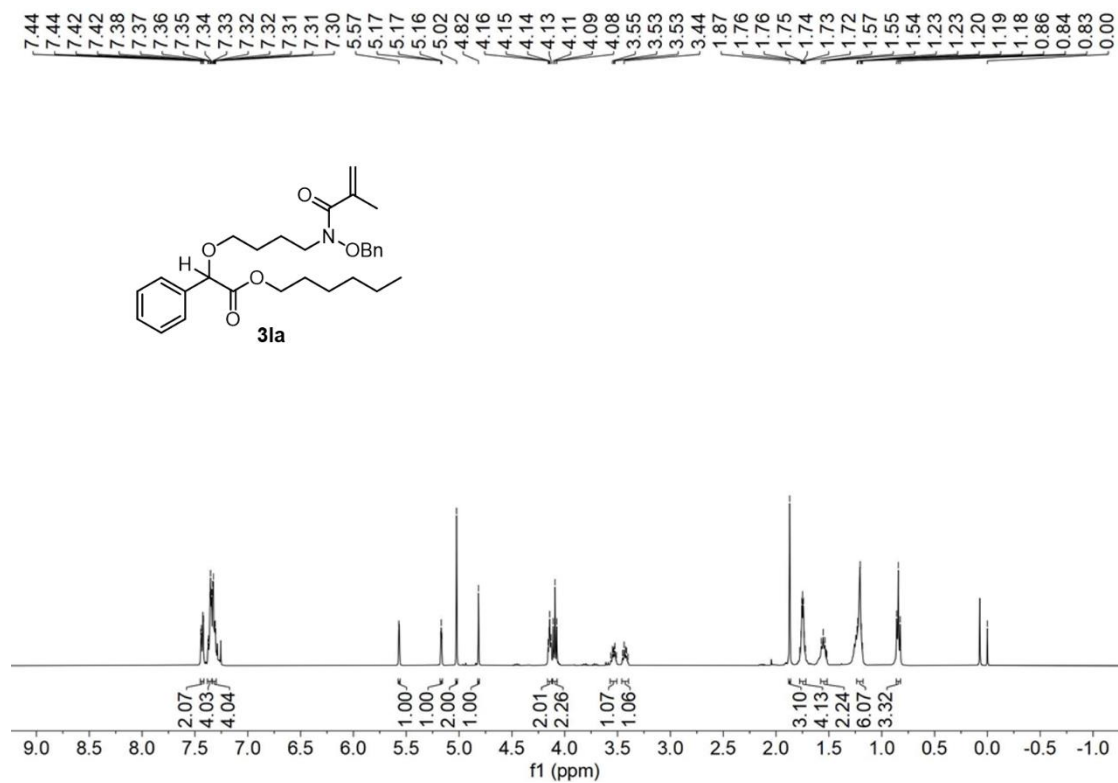
**Fig. S24**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3ja**.



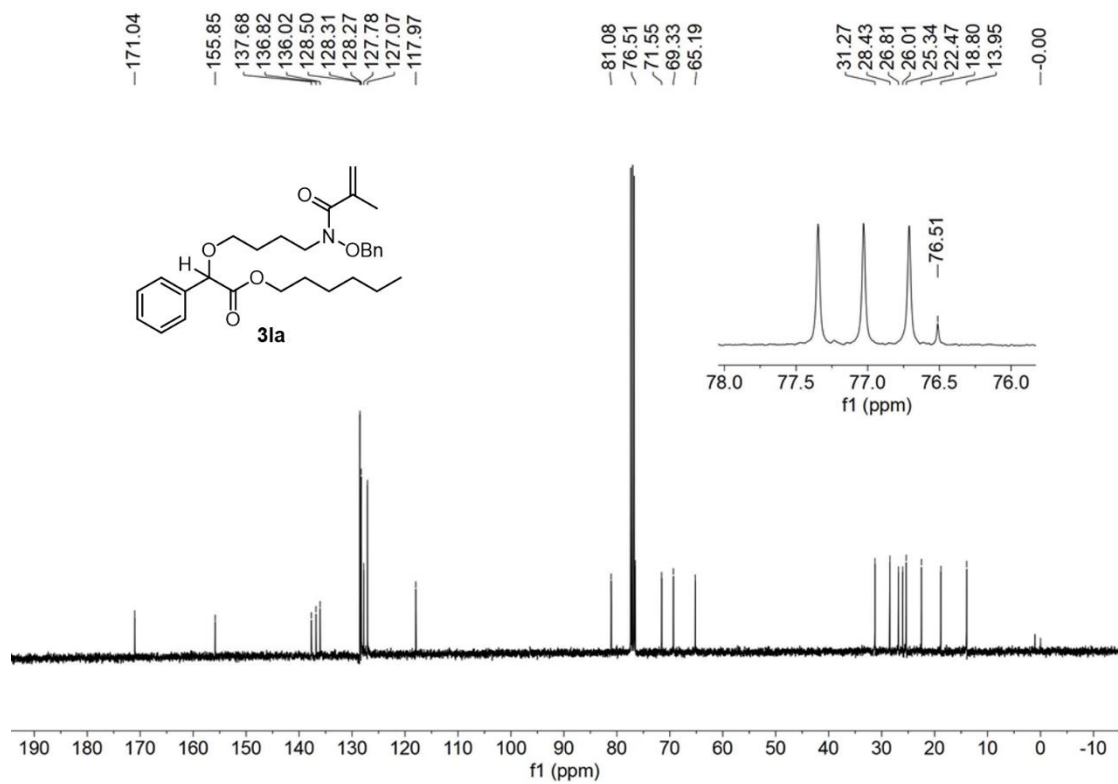
**Fig. S25** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ka**.



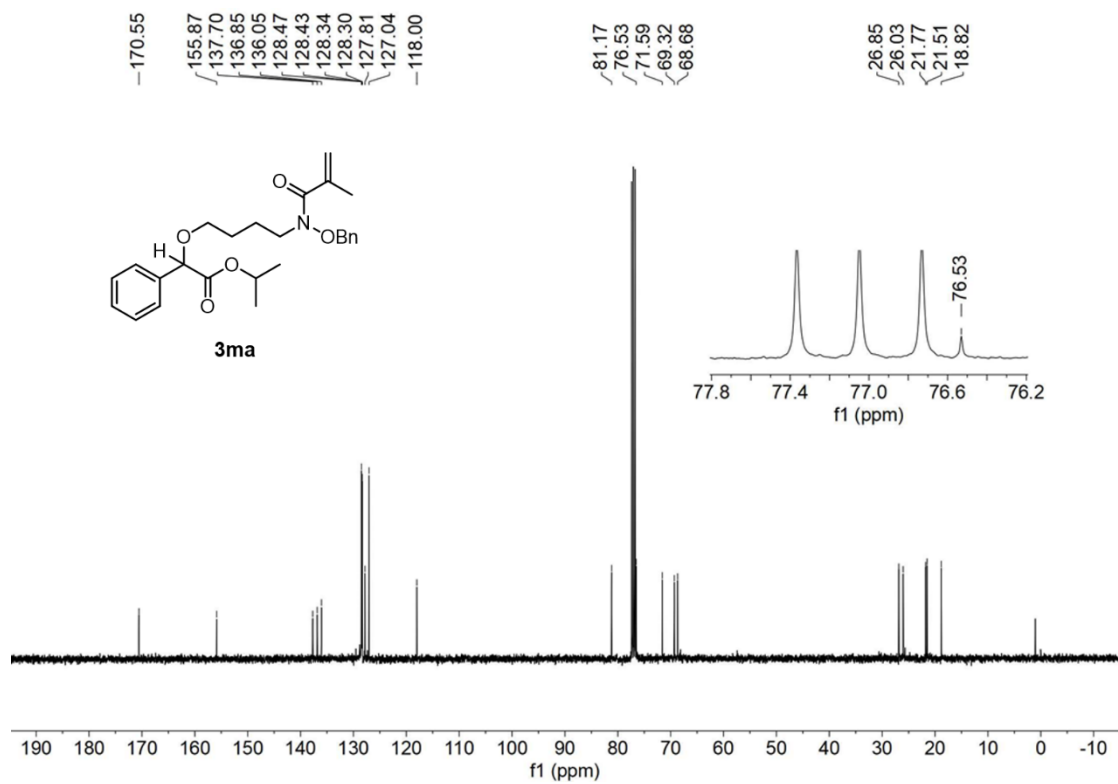
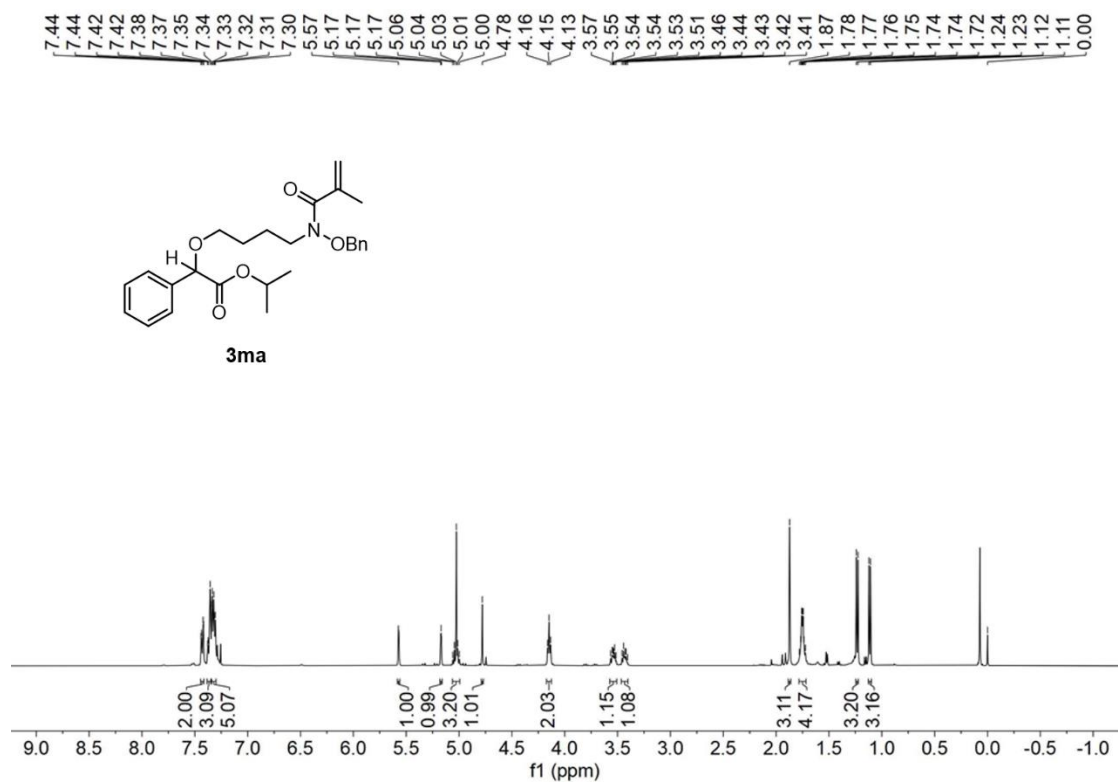
**Fig. S26** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ka**.

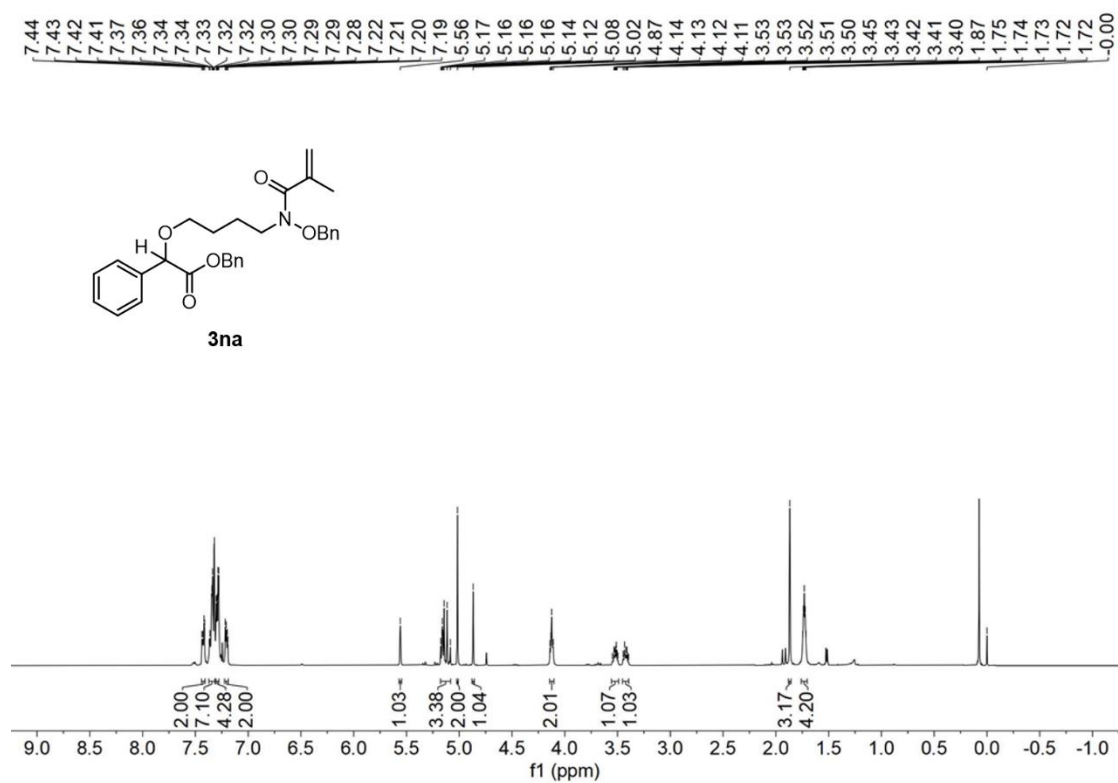


**Fig. S27**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3la**.

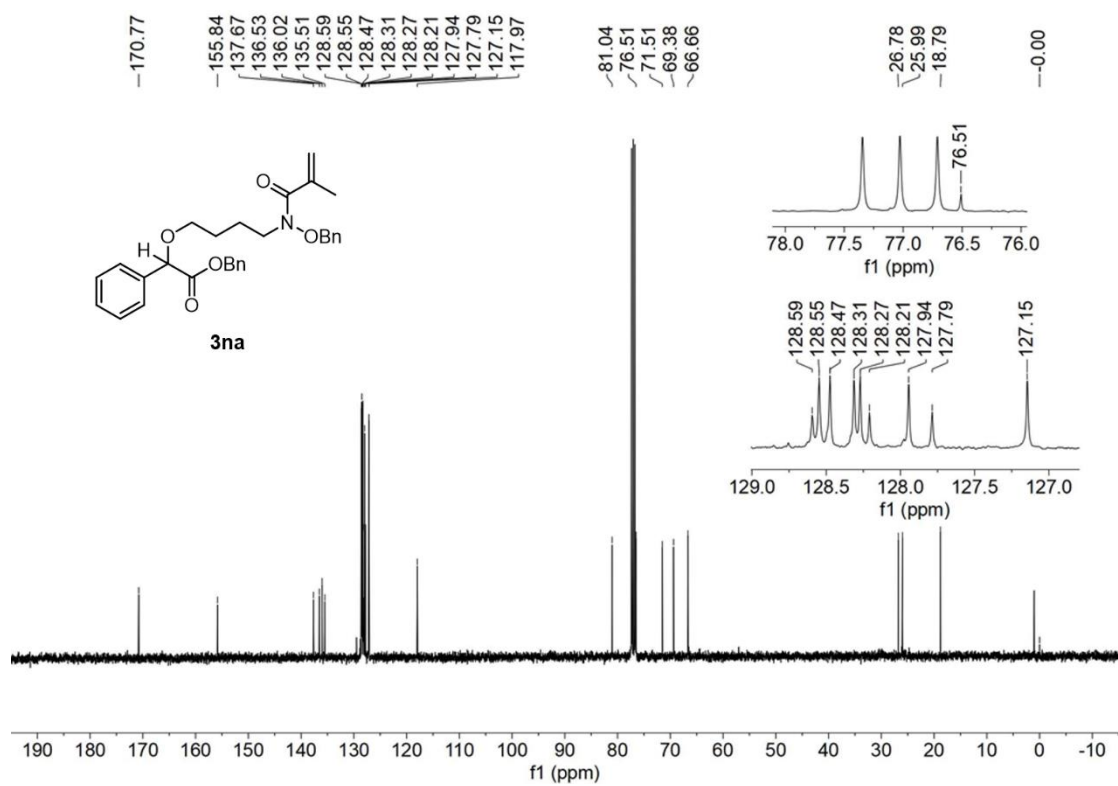


**Fig. S28**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3la**.



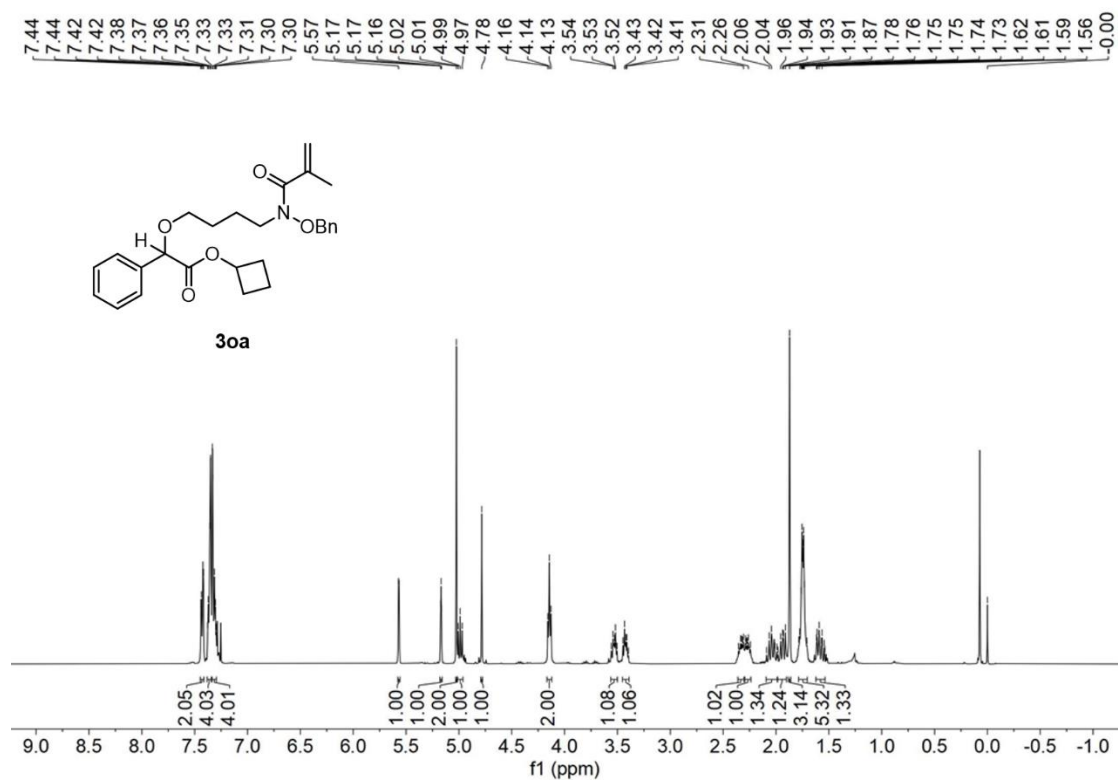


**Fig. S31**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3na**.

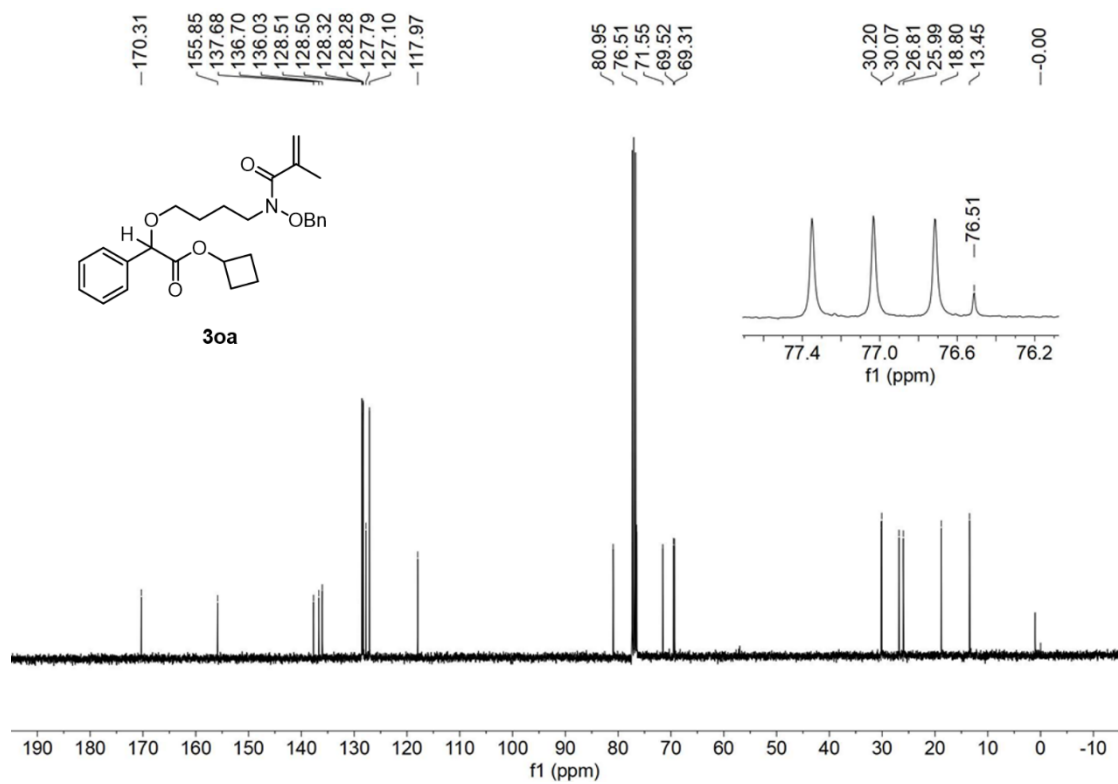


**Fig. S32**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3na**.

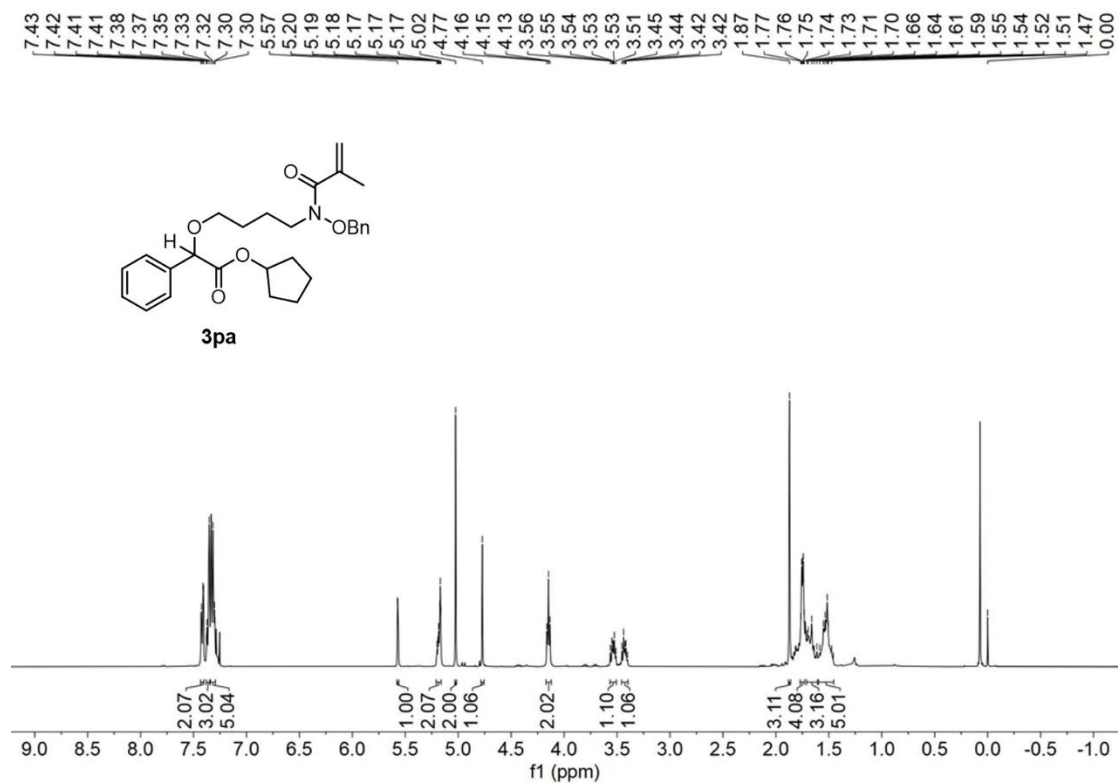




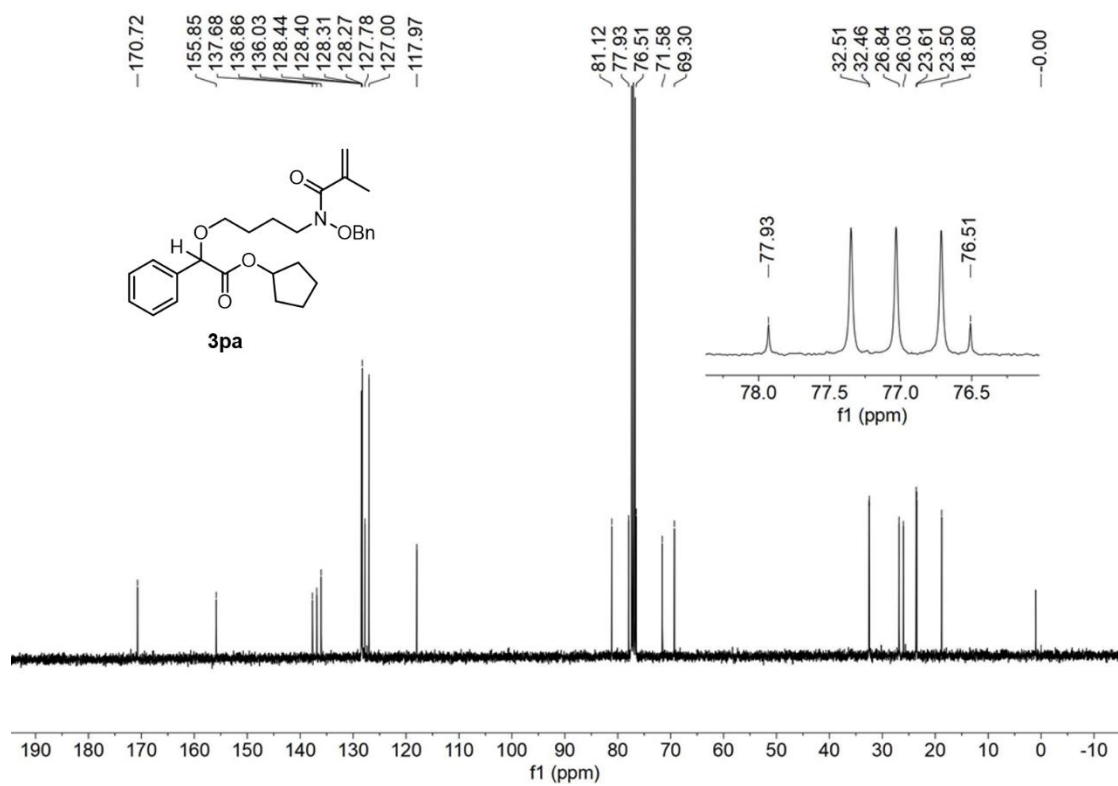
**Fig. S33** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **30a**.



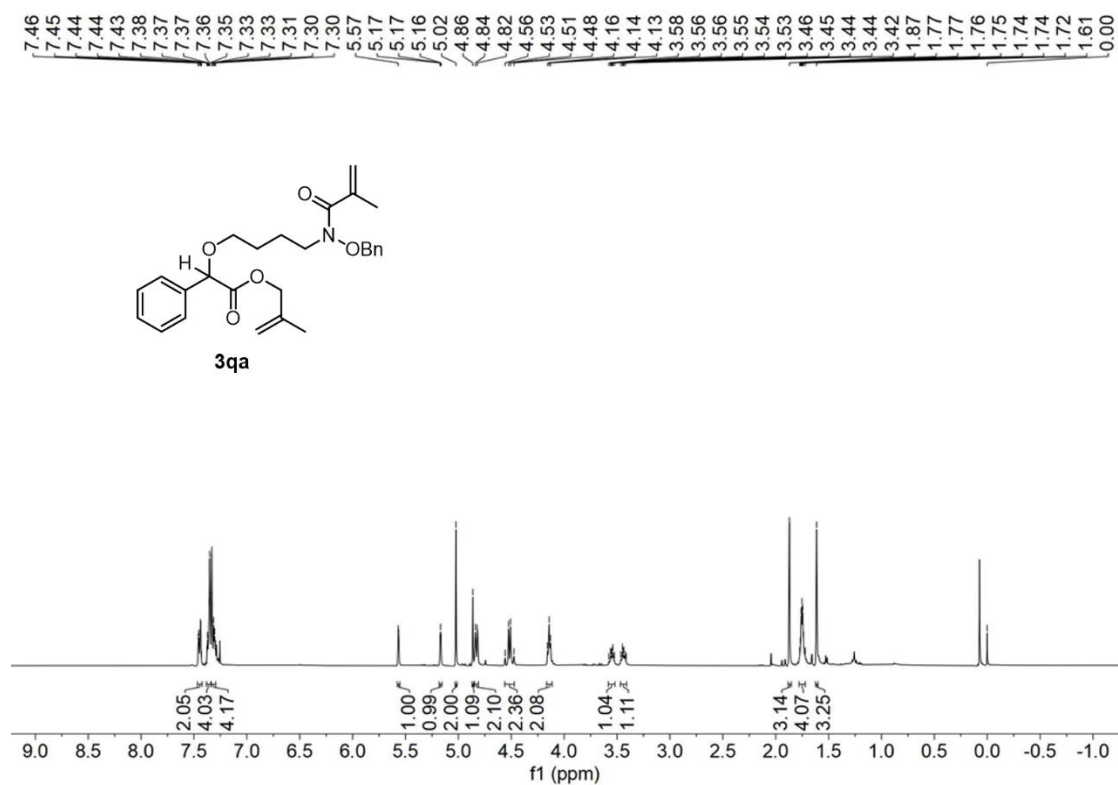
**Fig. S34** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **30a**.



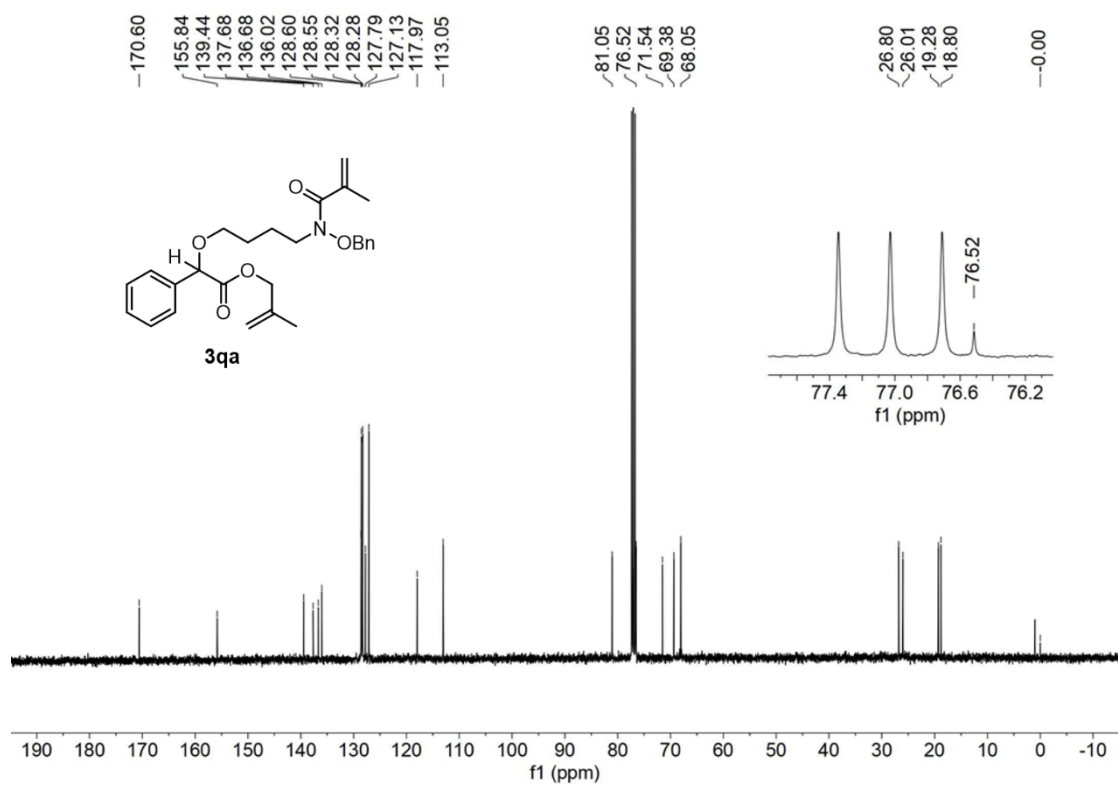
**Fig. S35** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3pa**.



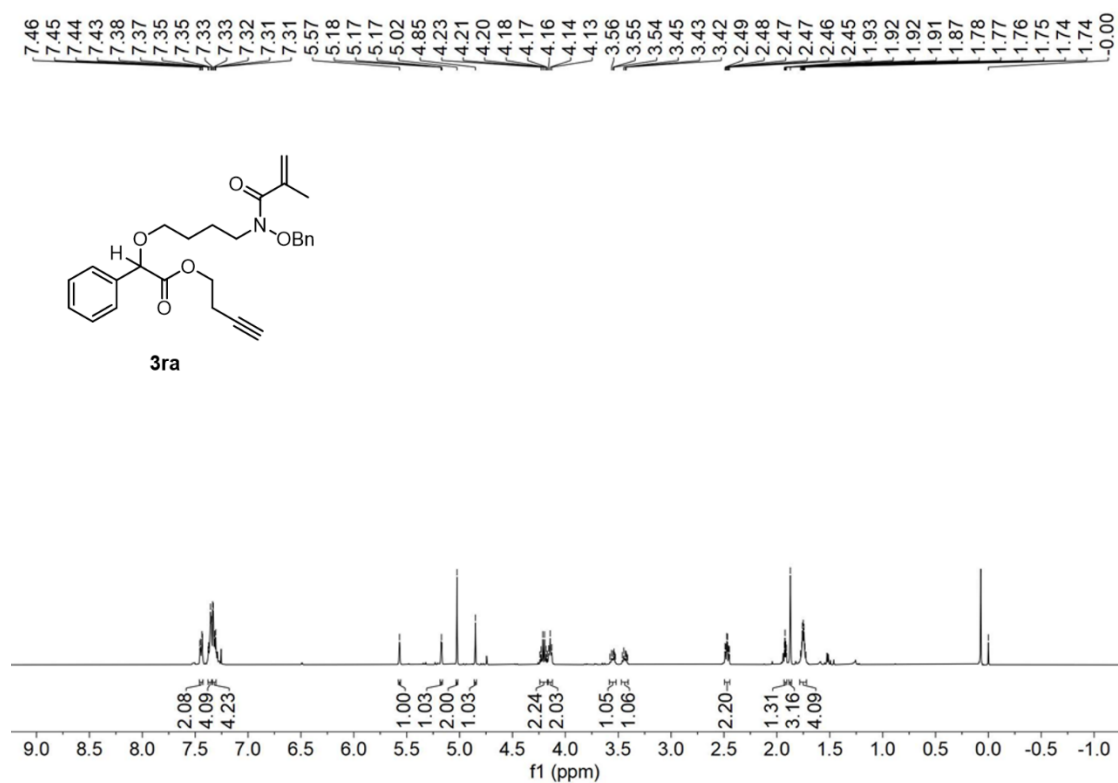
**Fig. S36** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3pa**.



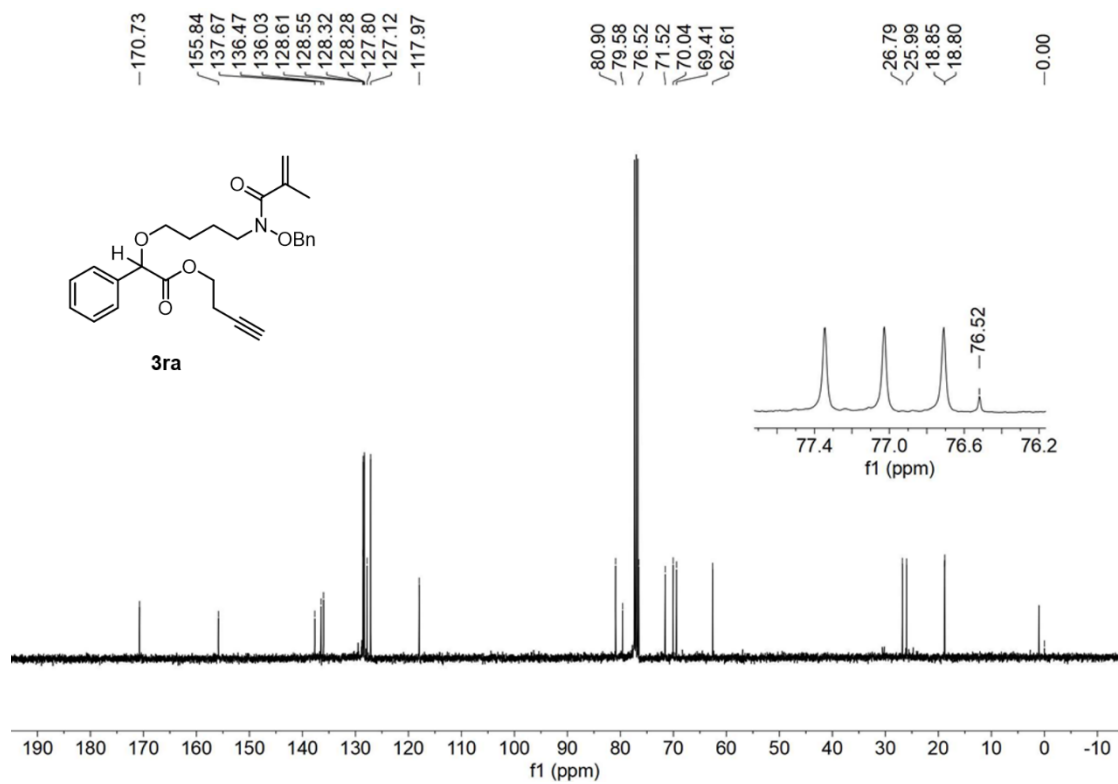
**Fig. S37** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3qa**.



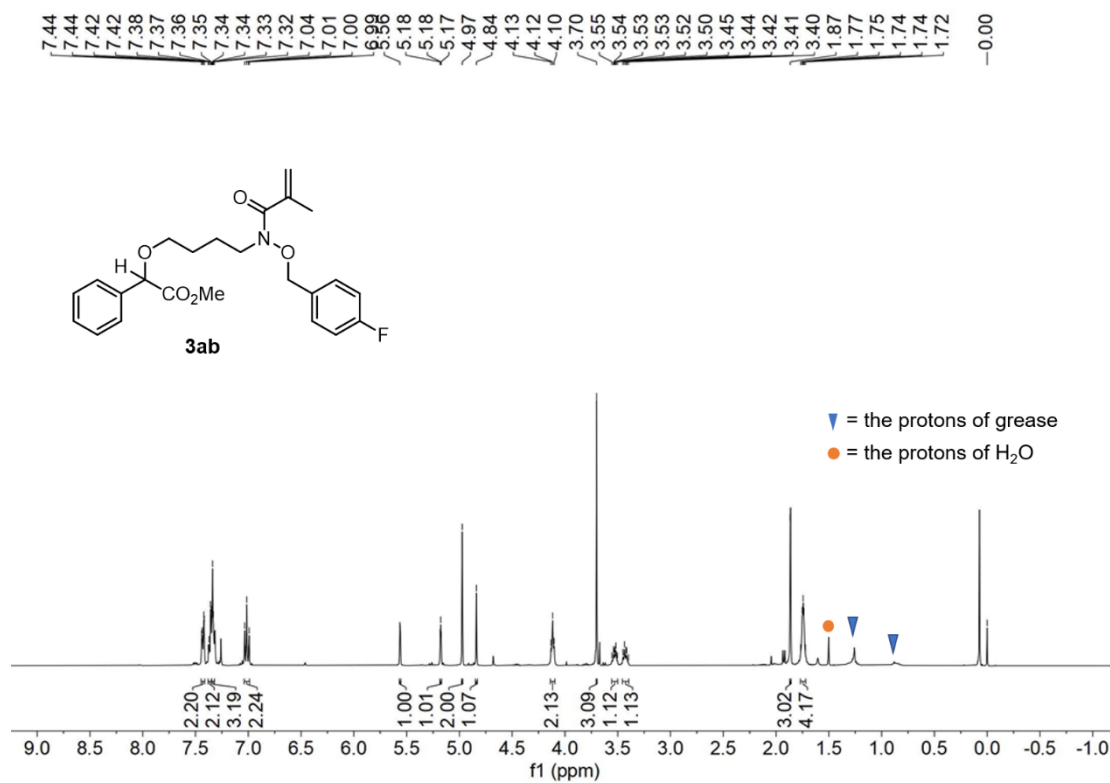
**Fig. S38** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3qa**.



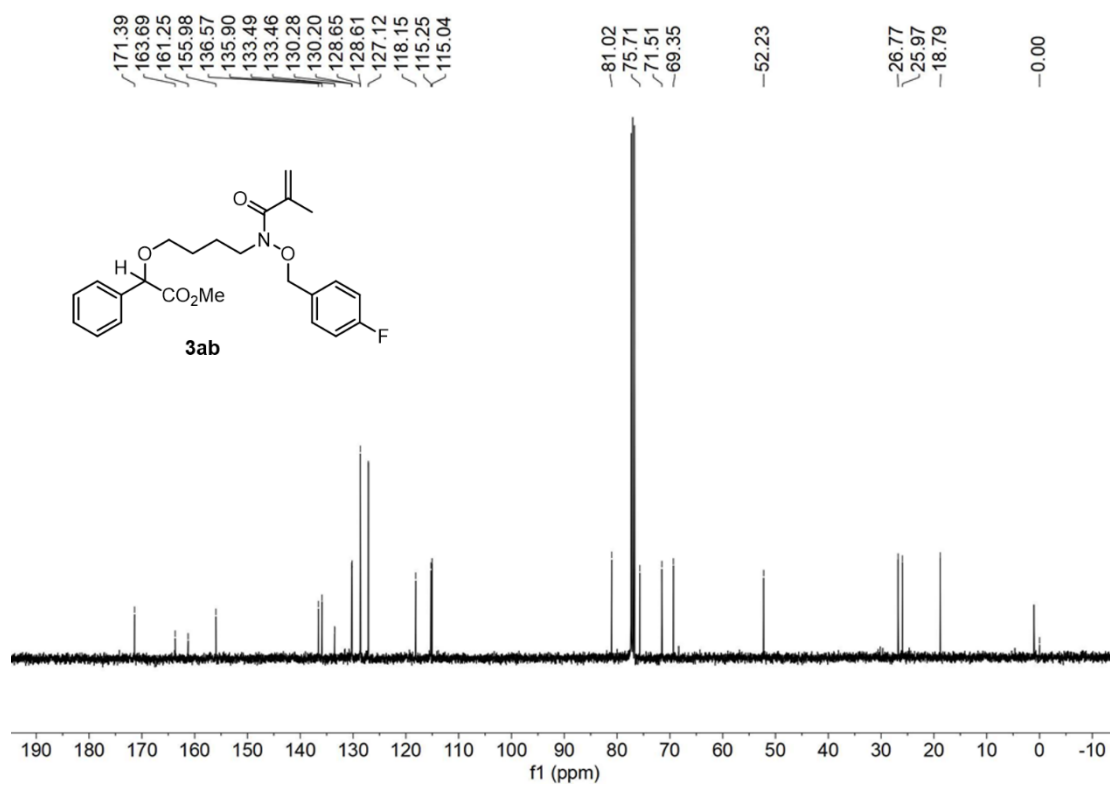
**Fig. S39** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ra**.



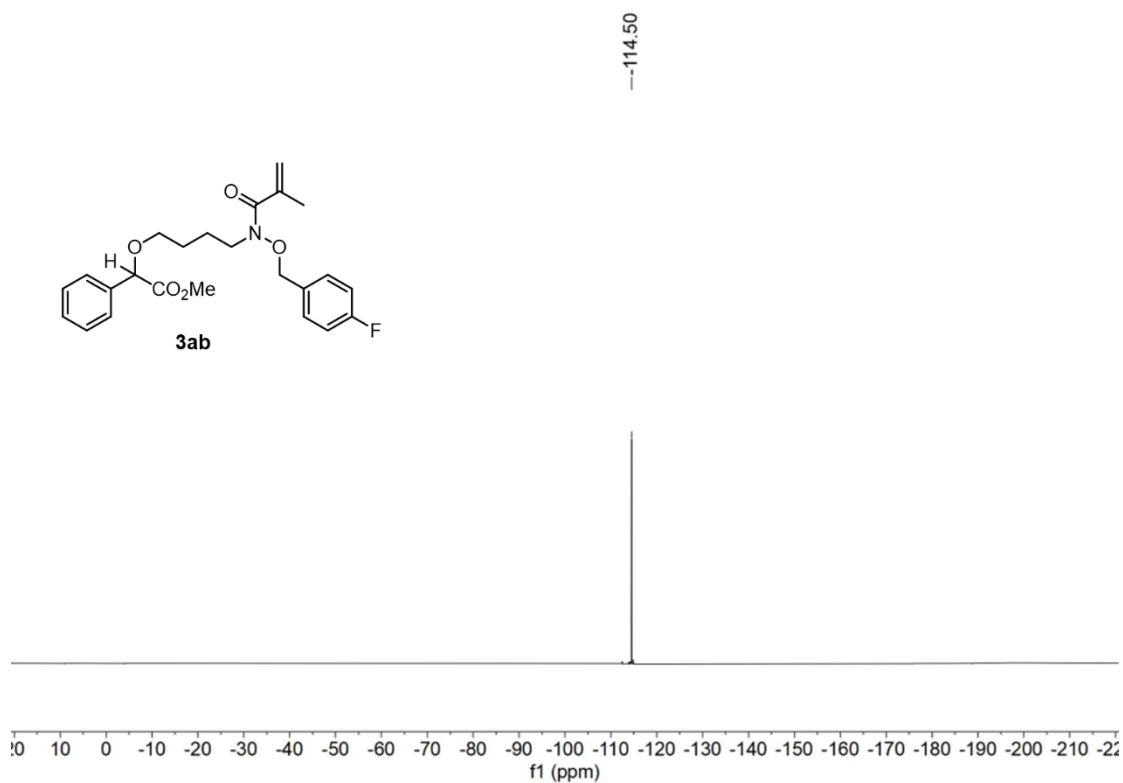
**Fig. S40** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ra**.



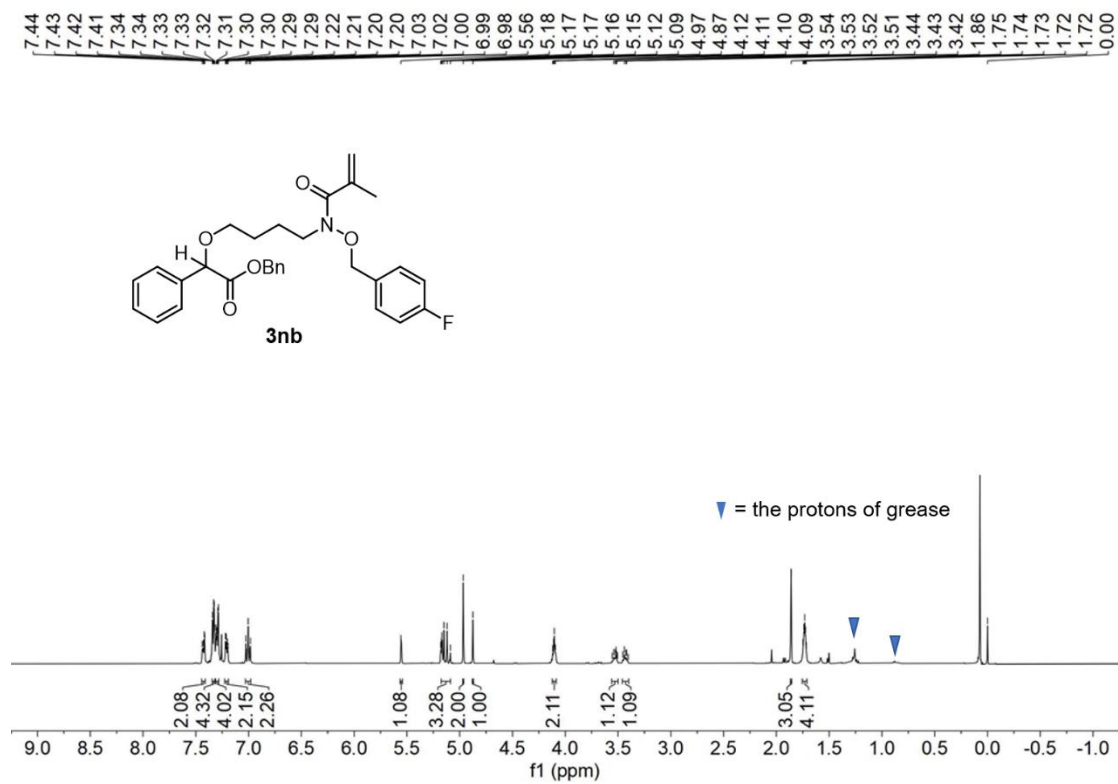
**Fig. S41** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ab**.



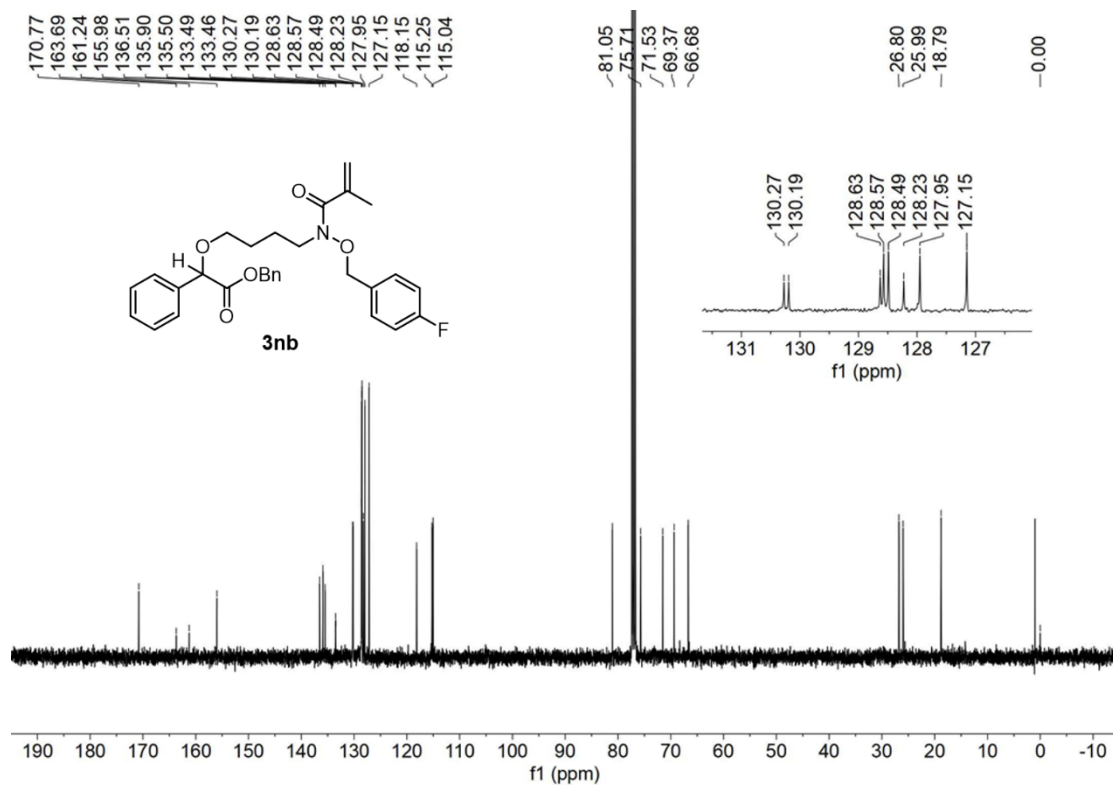
**Fig. S42** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ab**.



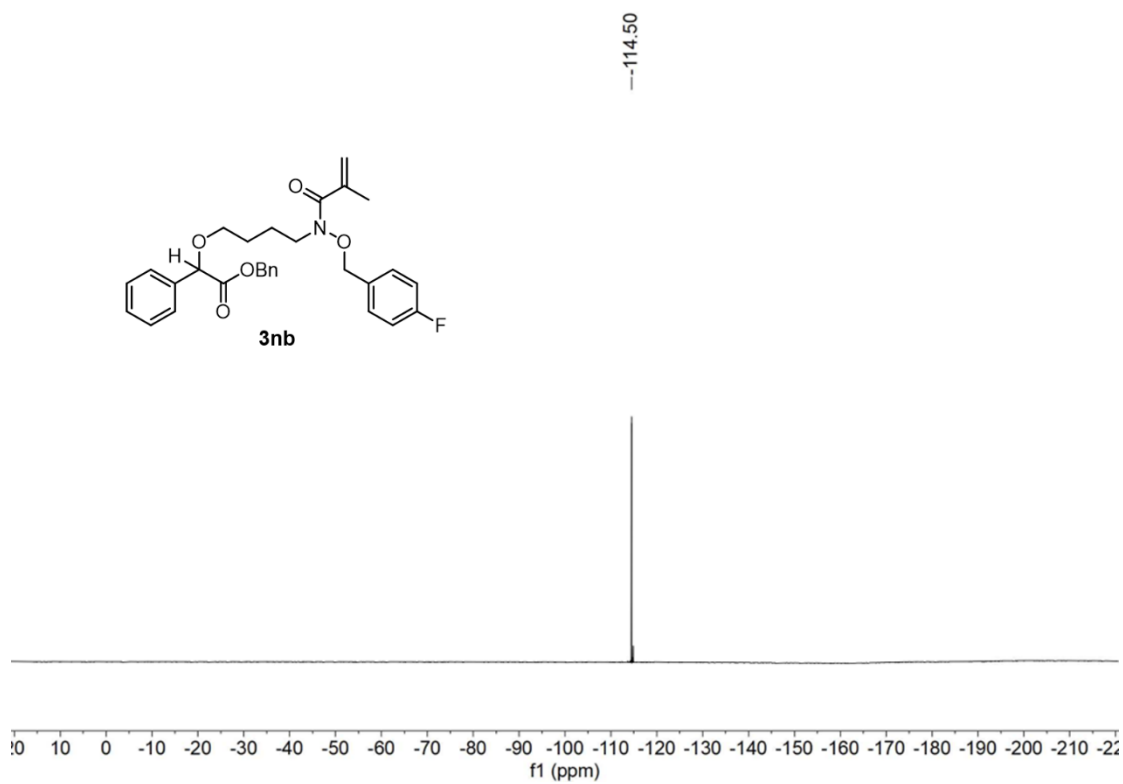
**Fig. S43**  $^{19}\text{F}$  NMR spectrum (376 MHz,  $\text{CDCl}_3$ ) of compound **3ab**.



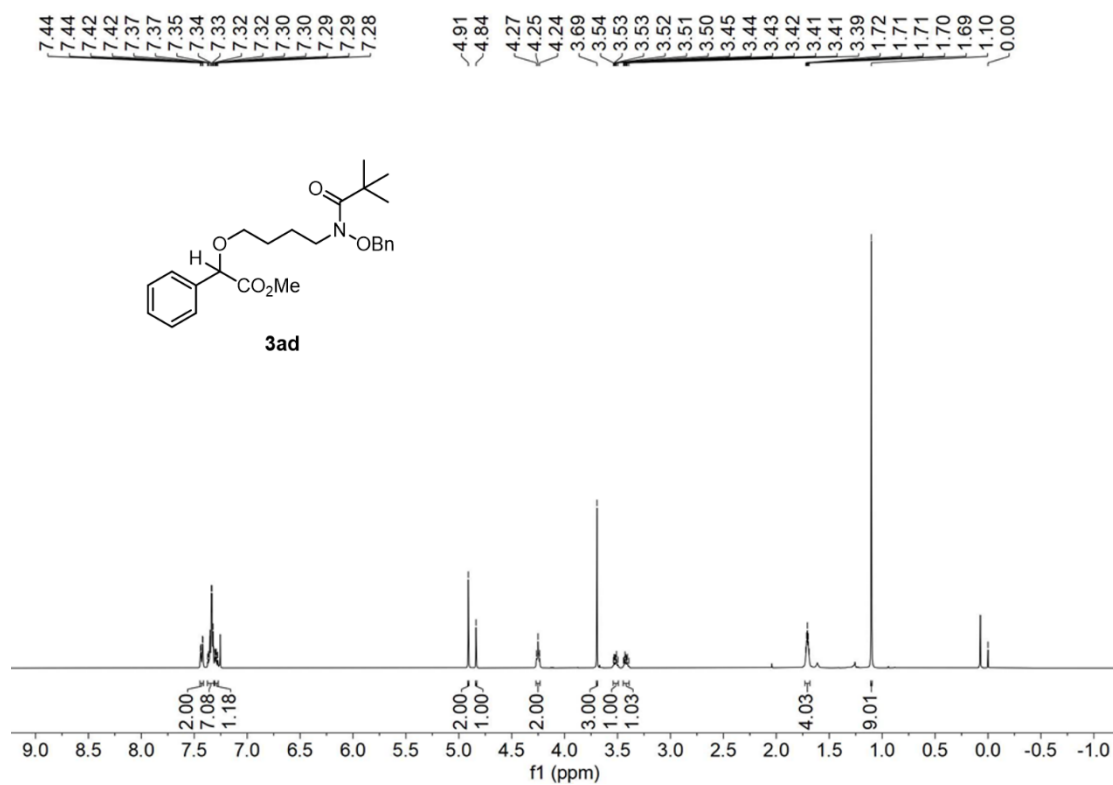
**Fig. S44**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3nb**.



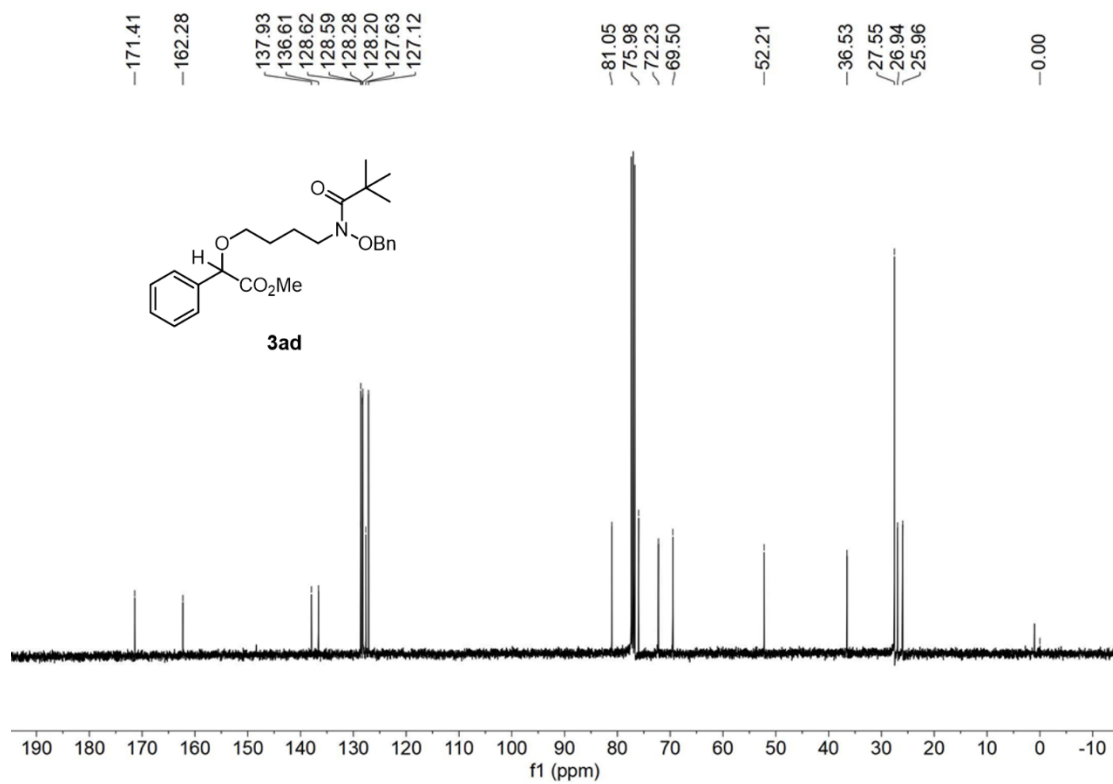
**Fig. S45** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3nb**.



**Fig. S46** <sup>19</sup>F NMR spectrum (376 MHz, CDCl<sub>3</sub>) of compound **3nb**.

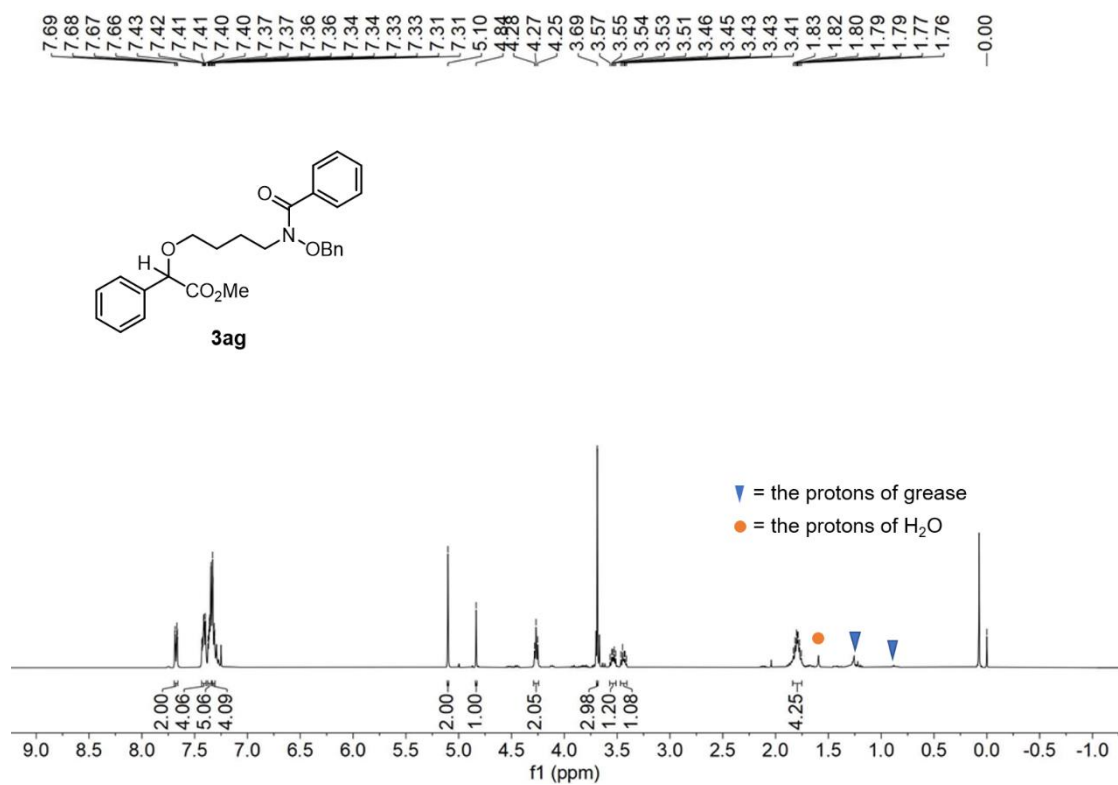


**Fig. S47** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ad**.

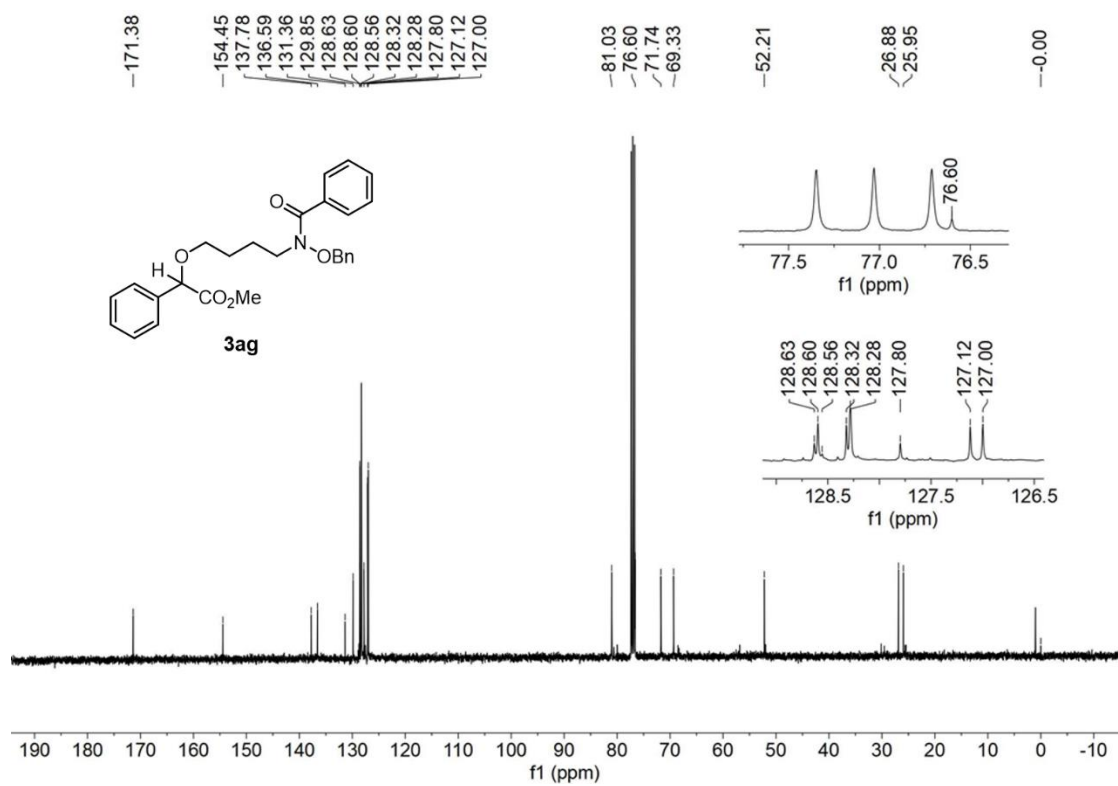


**Fig. S48** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ad**.

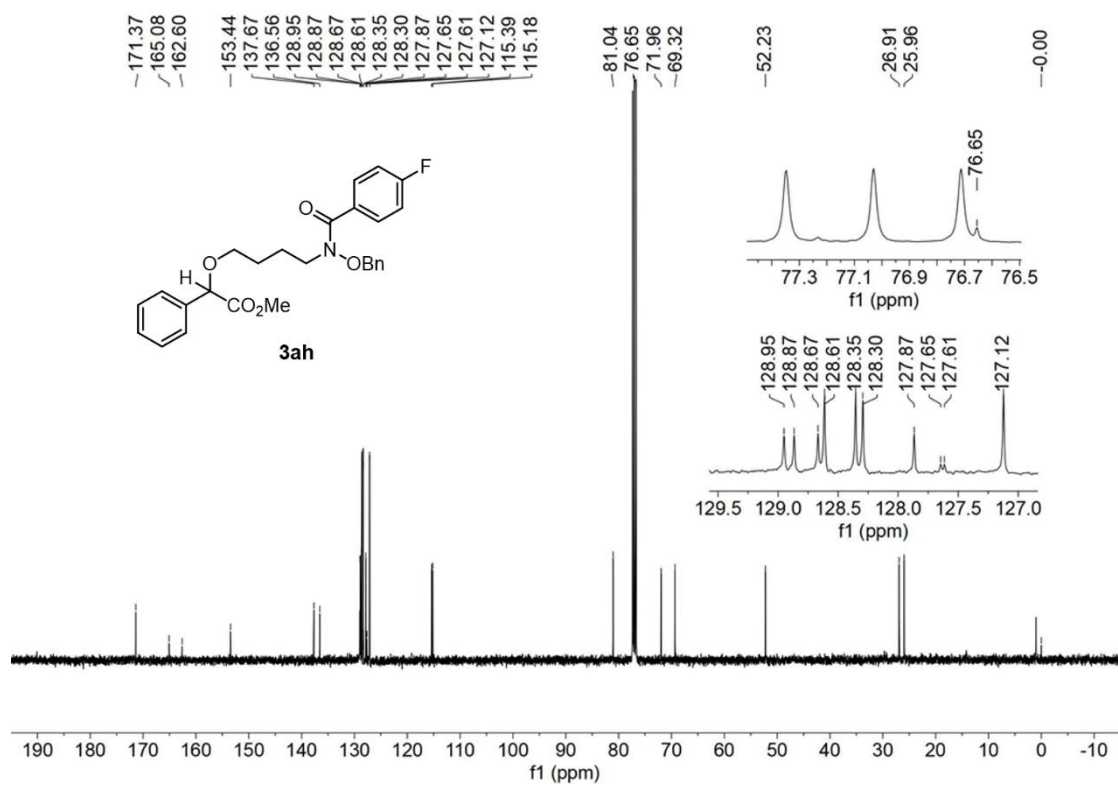
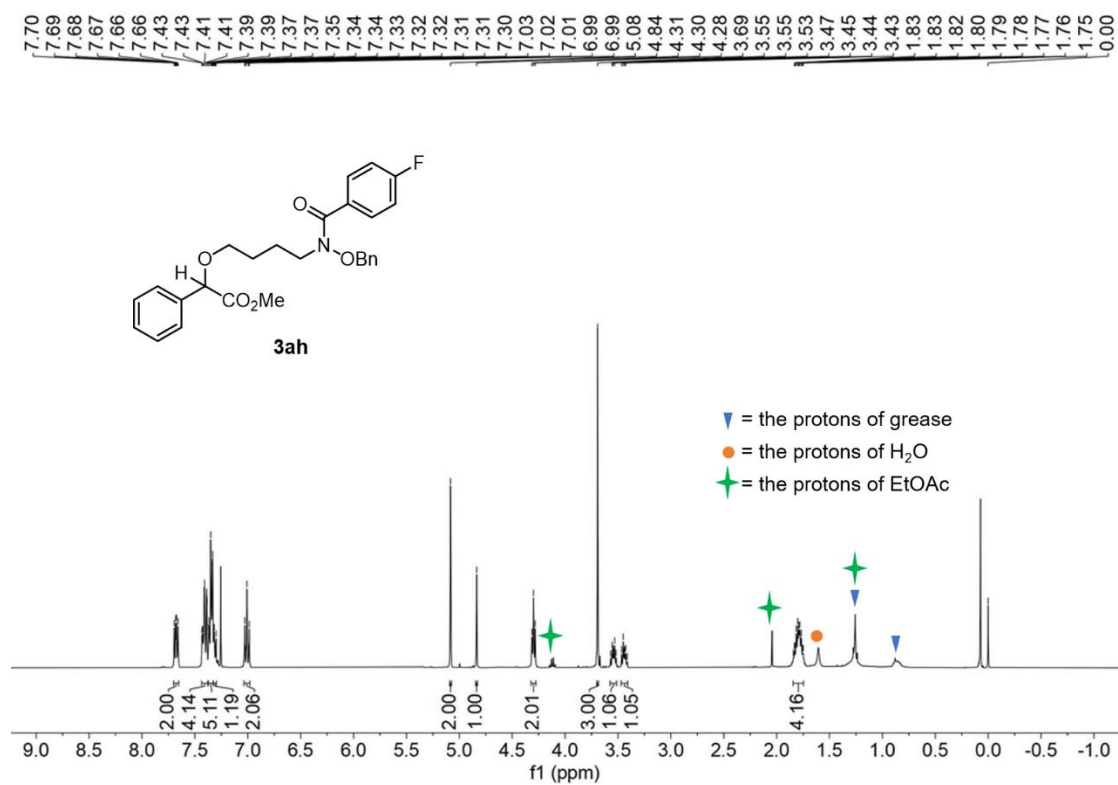


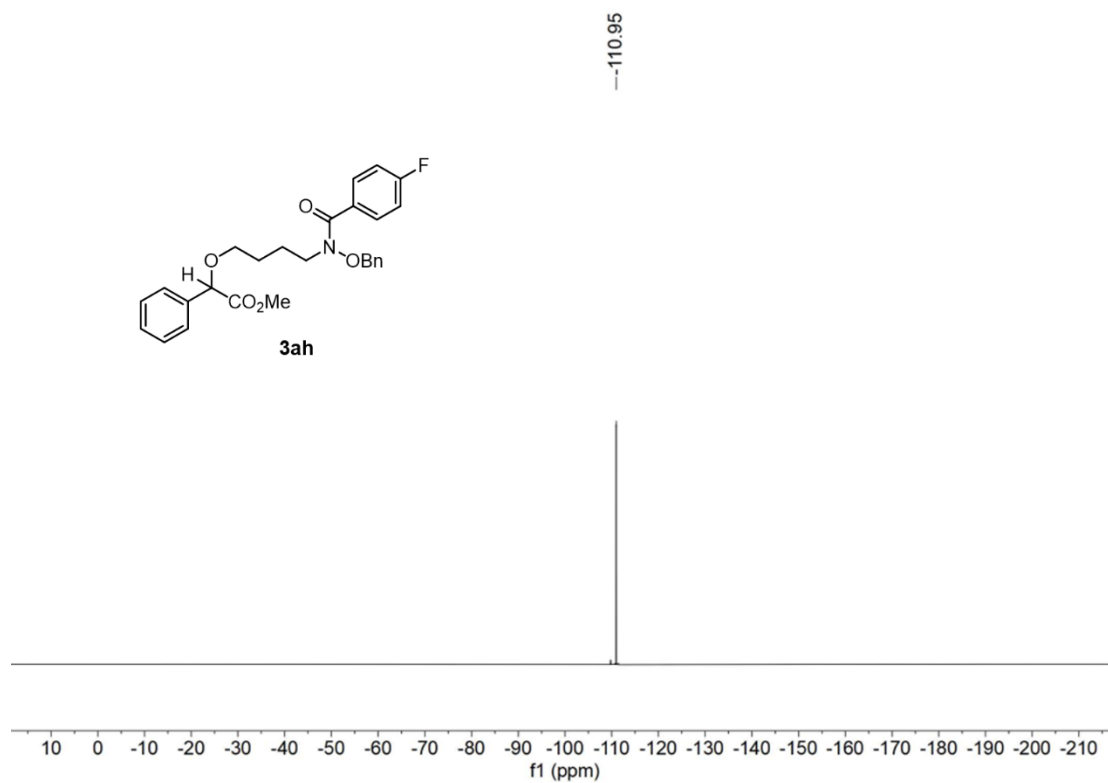


**Fig. S49** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ag**.

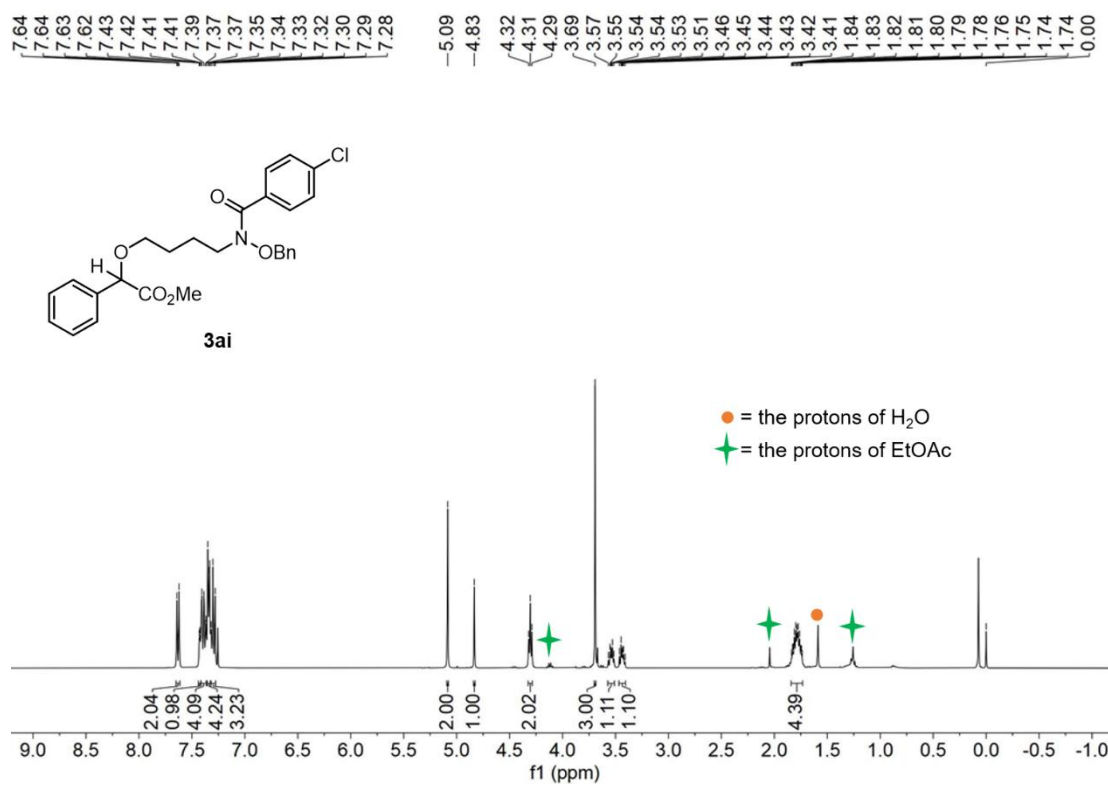


**Fig. S50** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ag**.

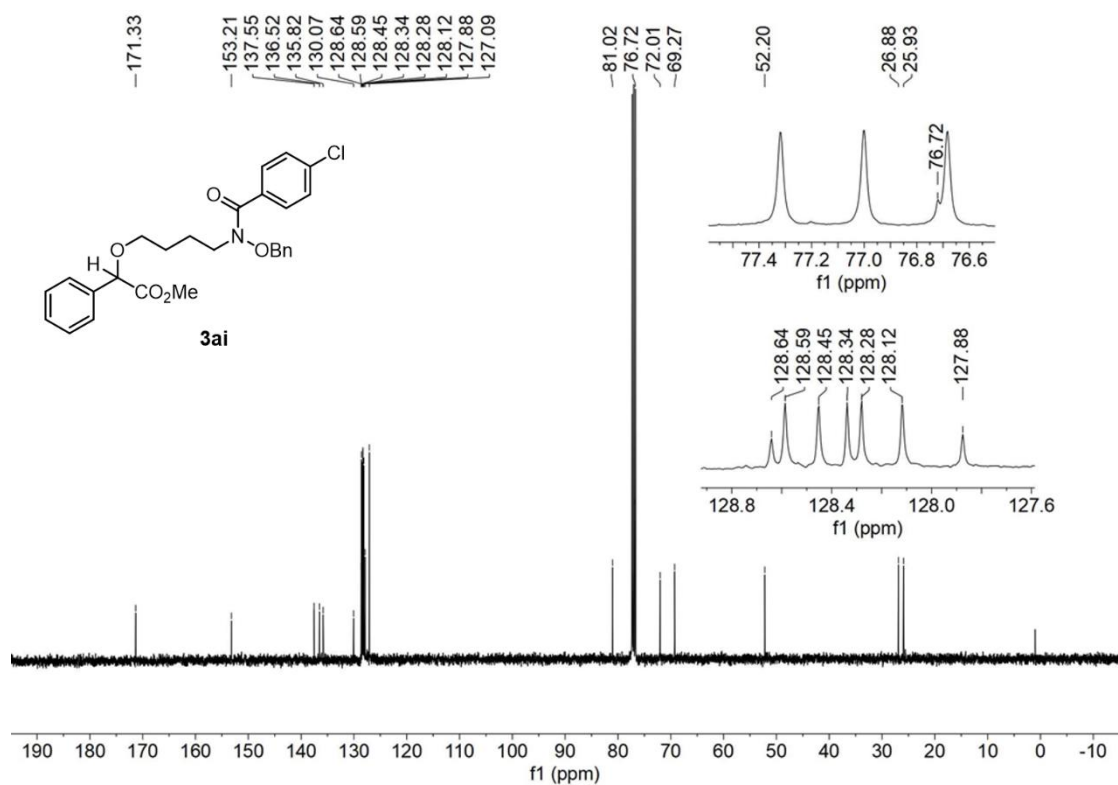




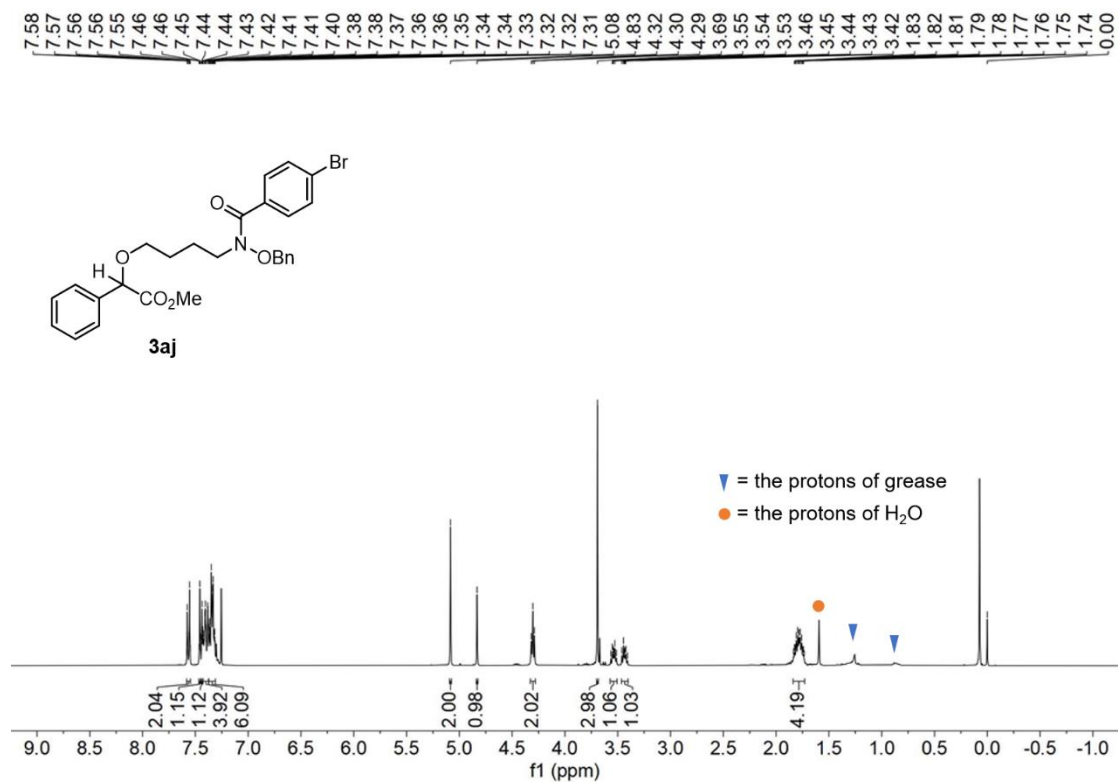
**Fig. S53**  $^{19}\text{F}$  NMR spectrum (376 MHz,  $\text{CDCl}_3$ ) of compound **3ah**.



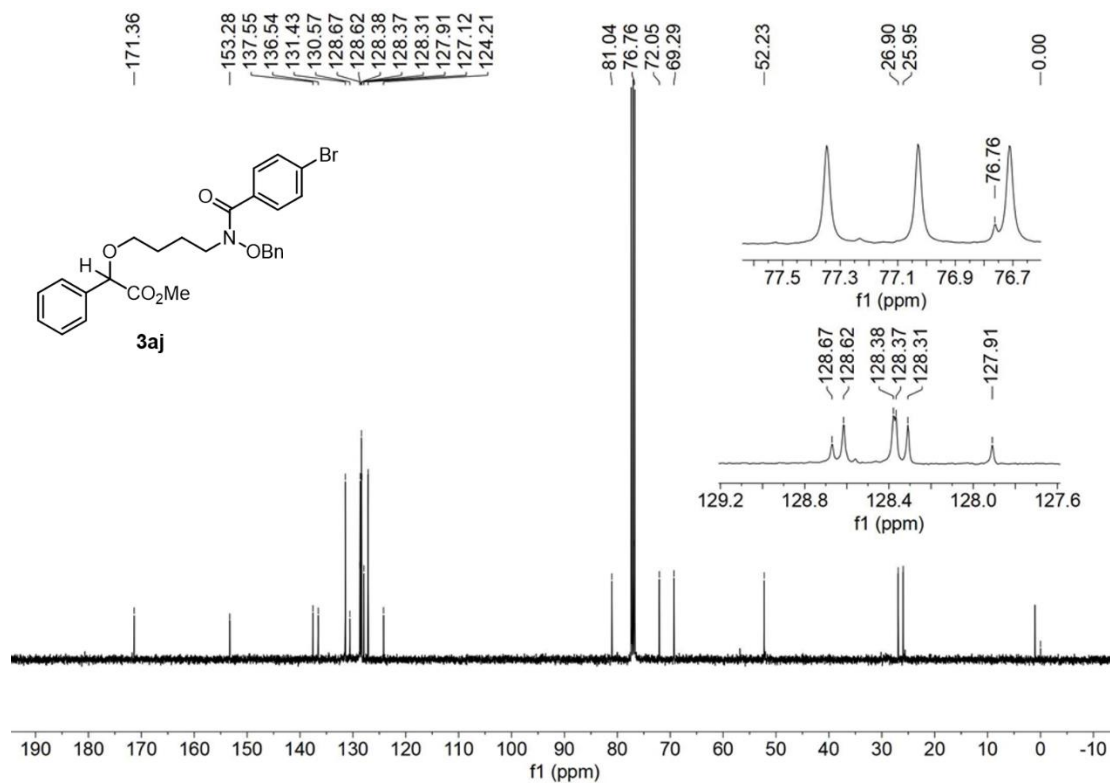
**Fig. S54**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **3ai**.



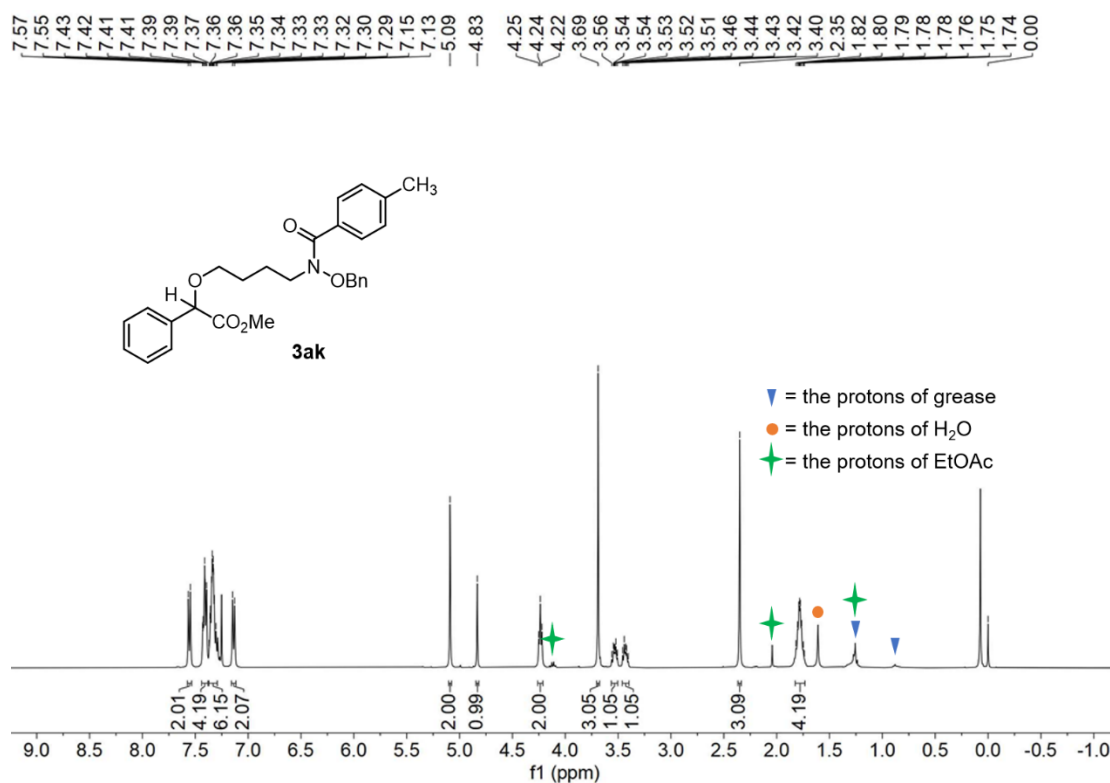
**Fig. S55** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ai**.



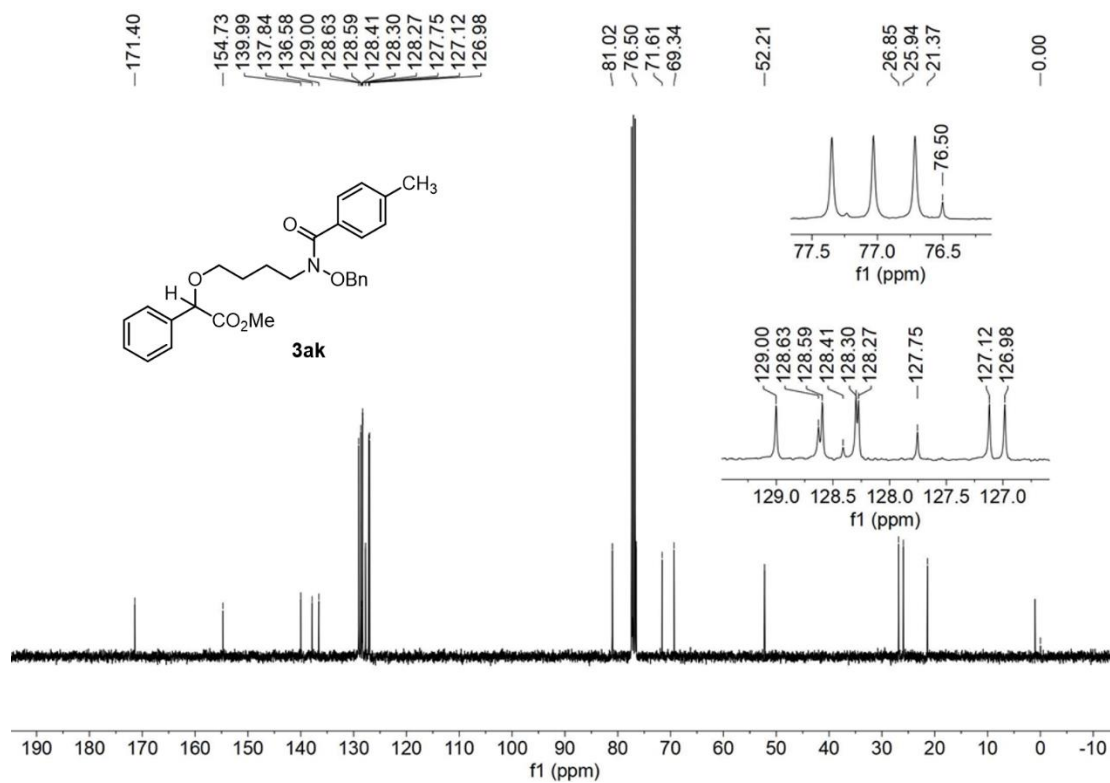
**Fig. S56** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3aj**.



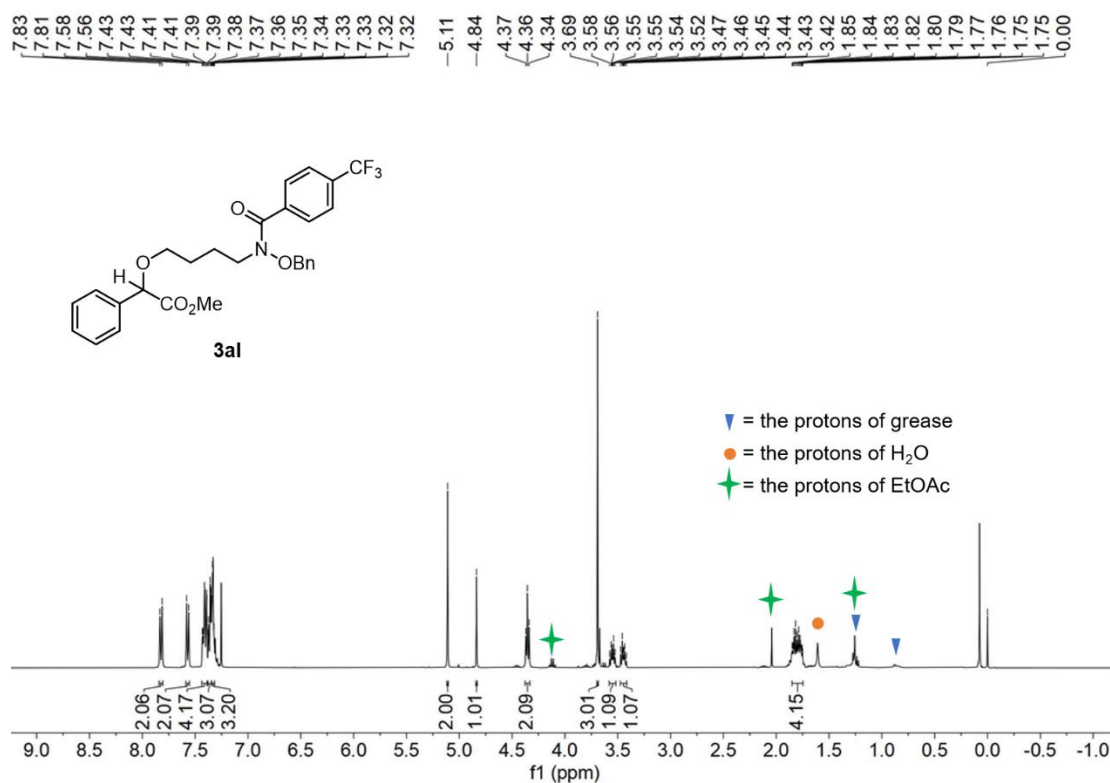
**Fig. S57** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3aj**.



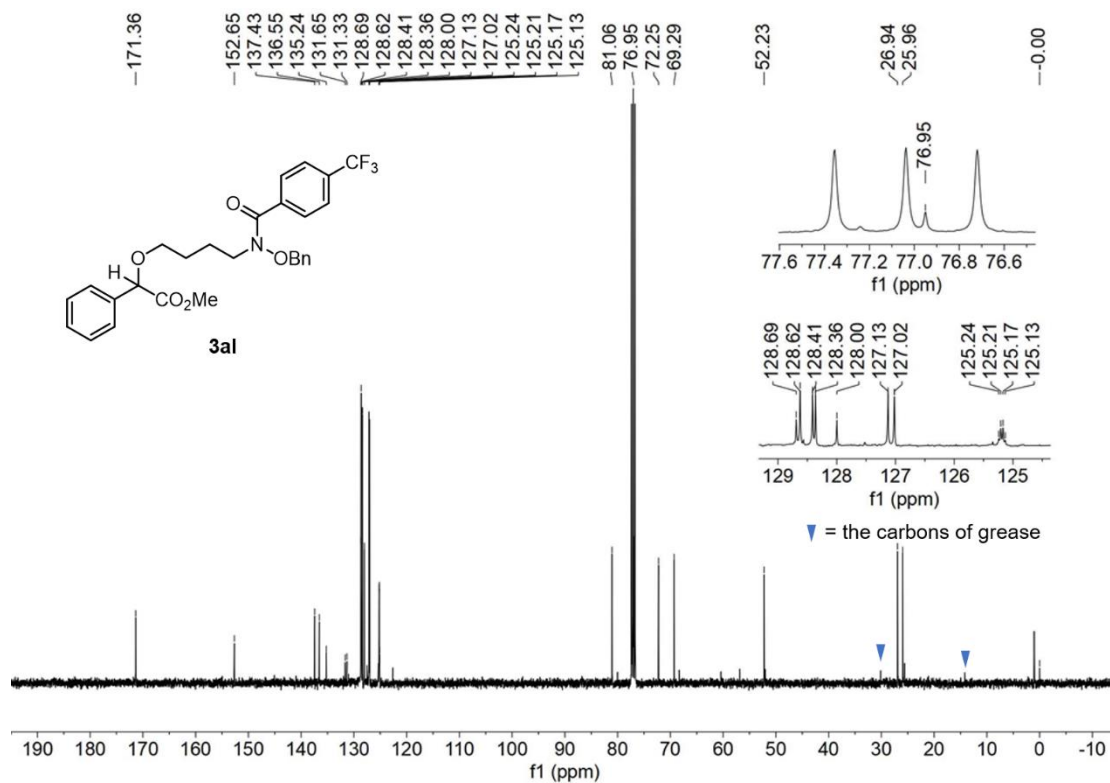
**Fig. S58** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3ak**.



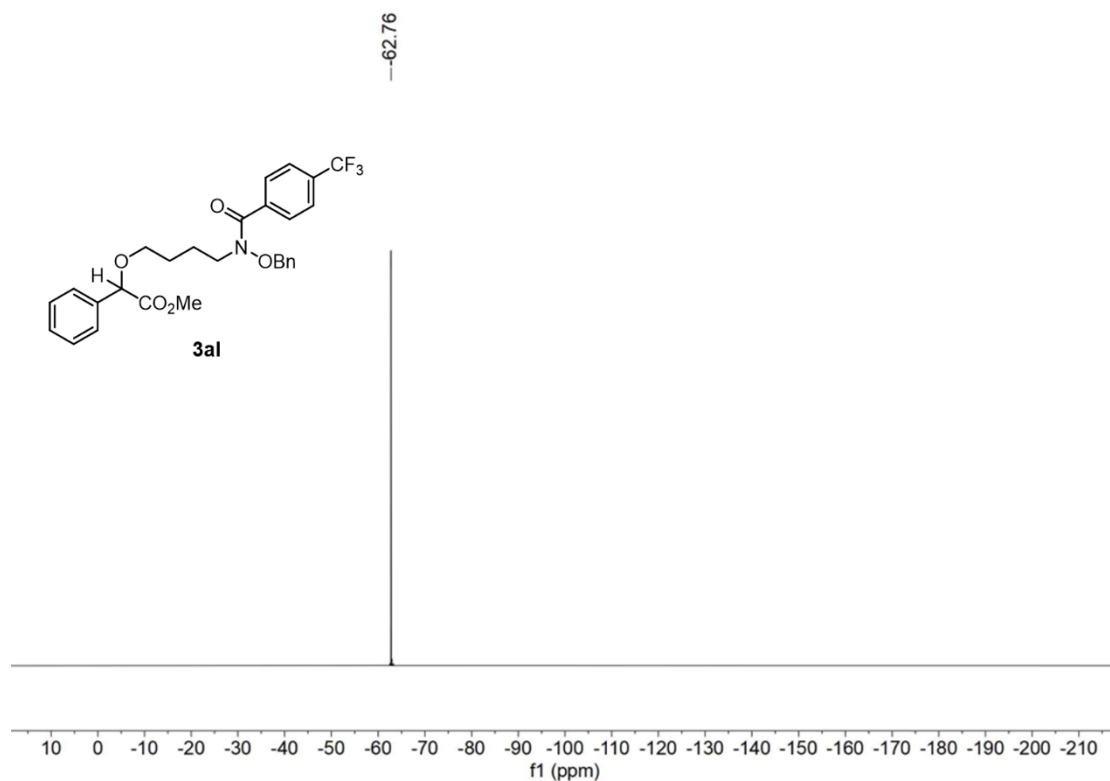
**Fig. S59** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3ak**.



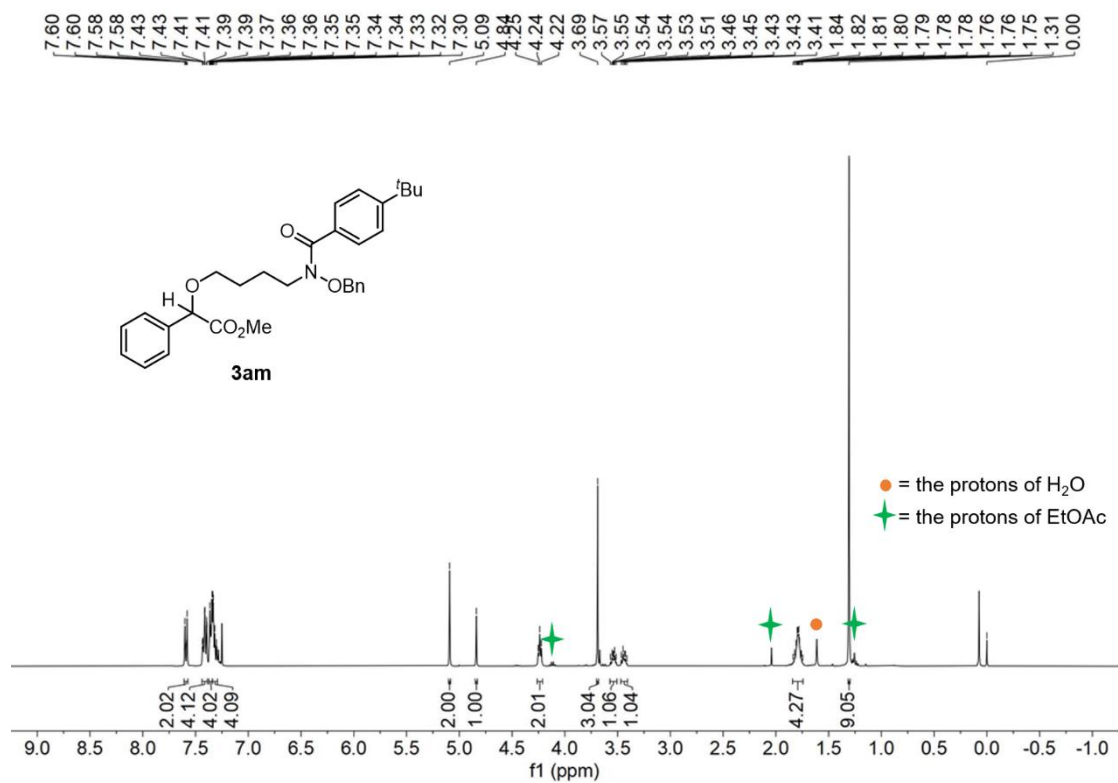
**Fig. S60** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3al**.



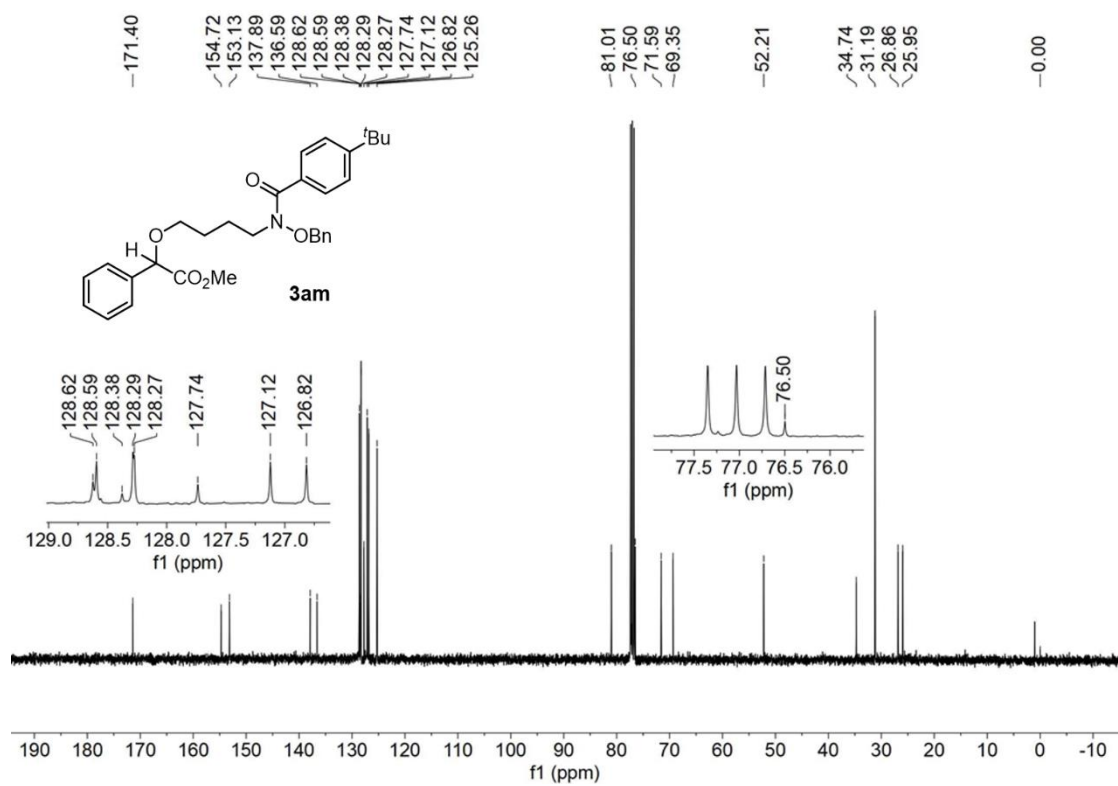
**Fig. S61**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **3al**.



**Fig. S62**  $^{19}\text{F}$  NMR spectrum (376 MHz,  $\text{CDCl}_3$ ) of compound **3al**.

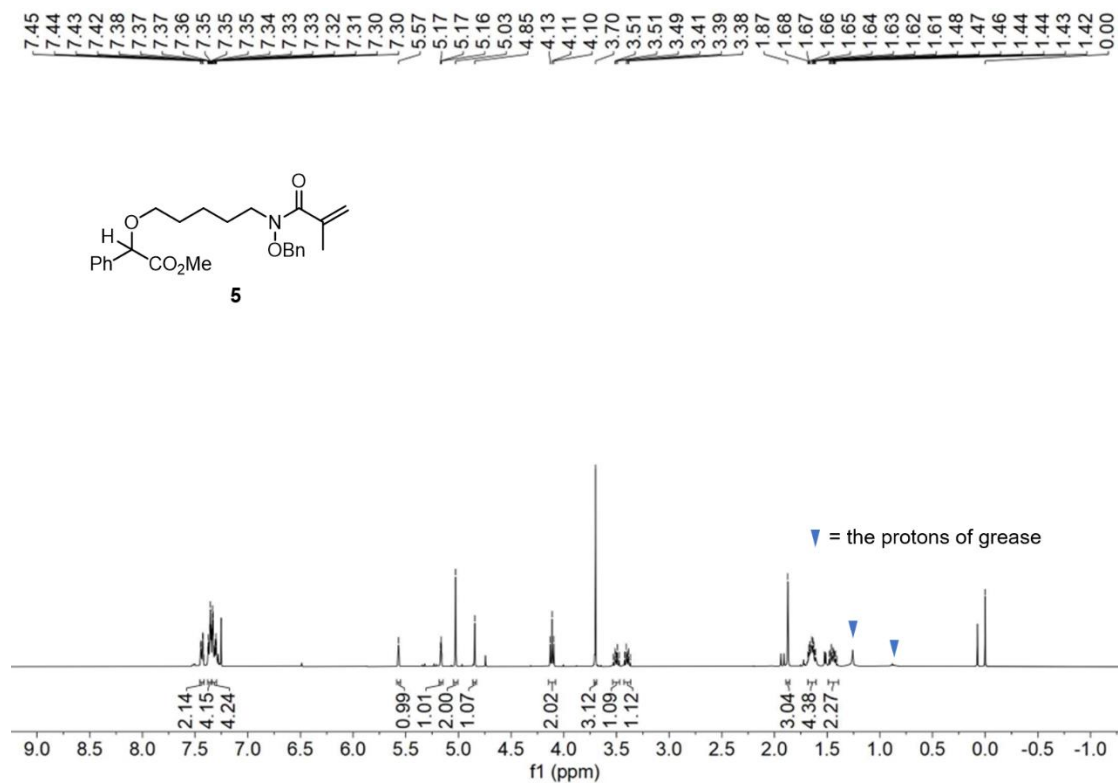


**Fig. S63** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **3am**.

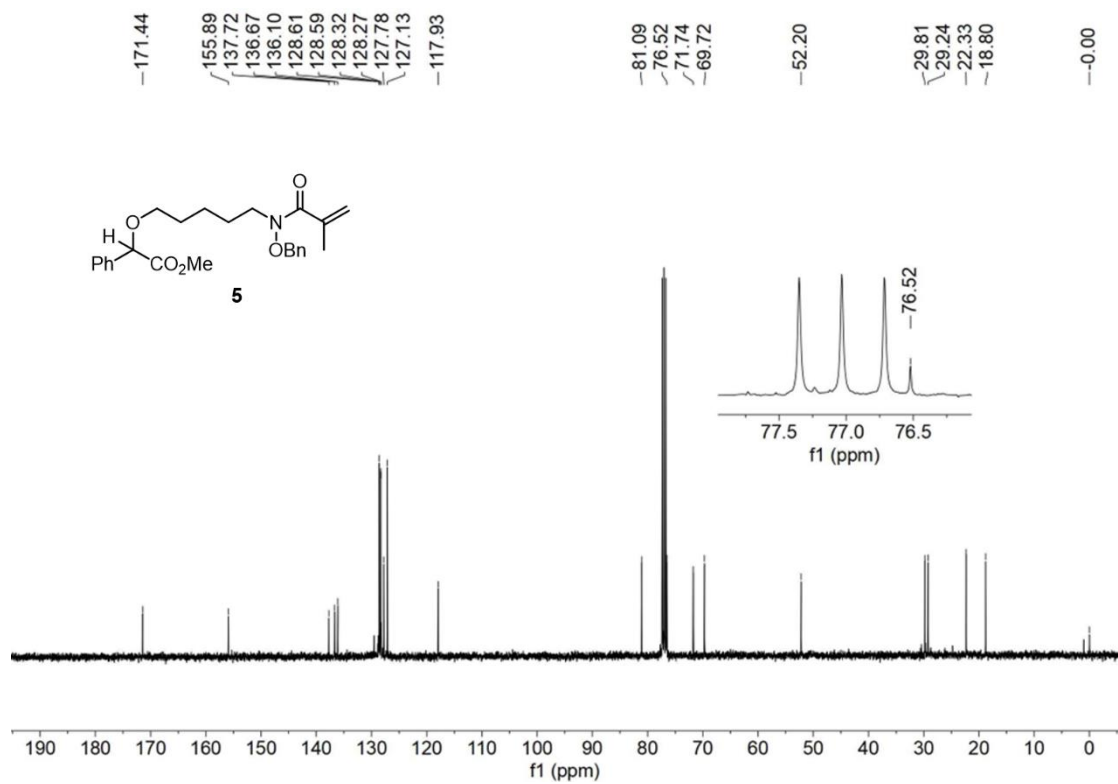


**Fig. S64** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **3am**.

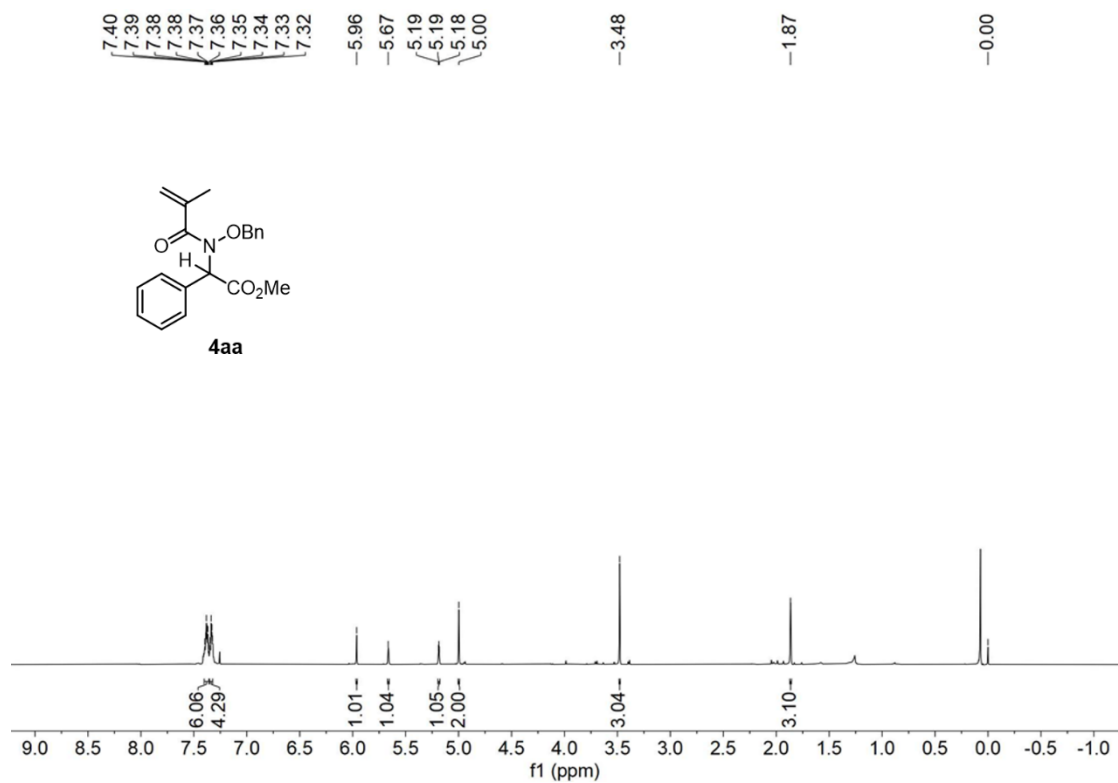




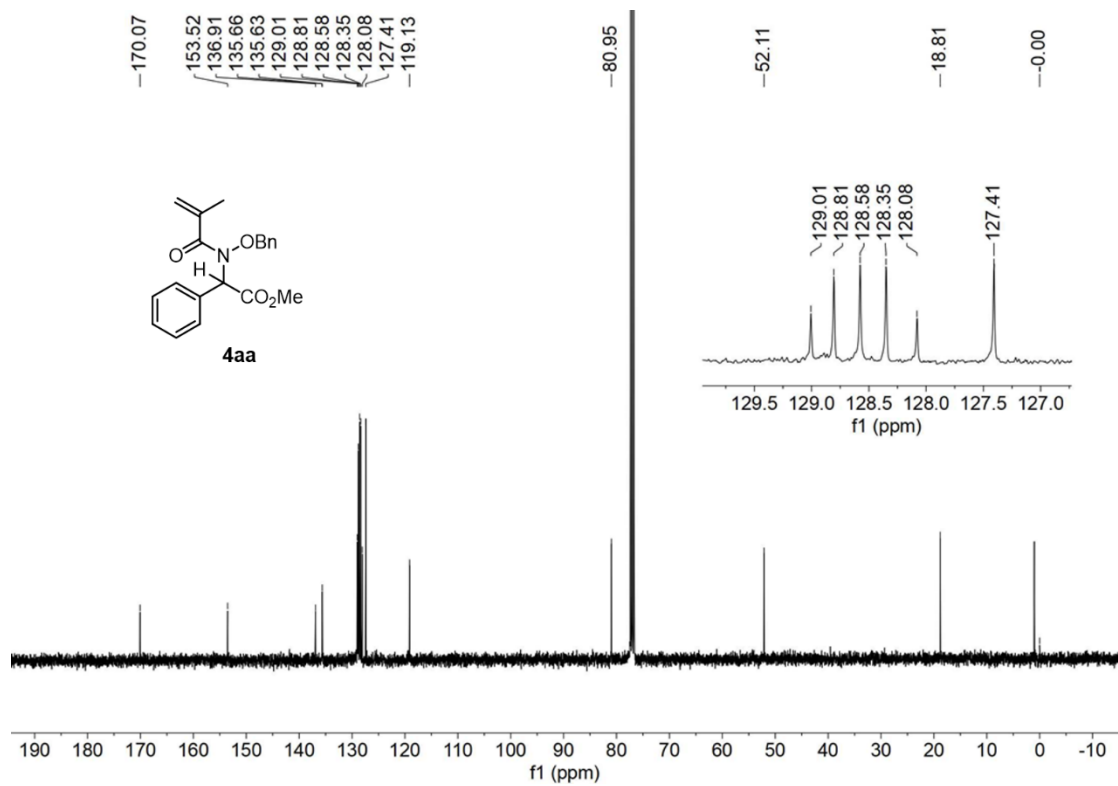
**Fig. S65**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **5**.



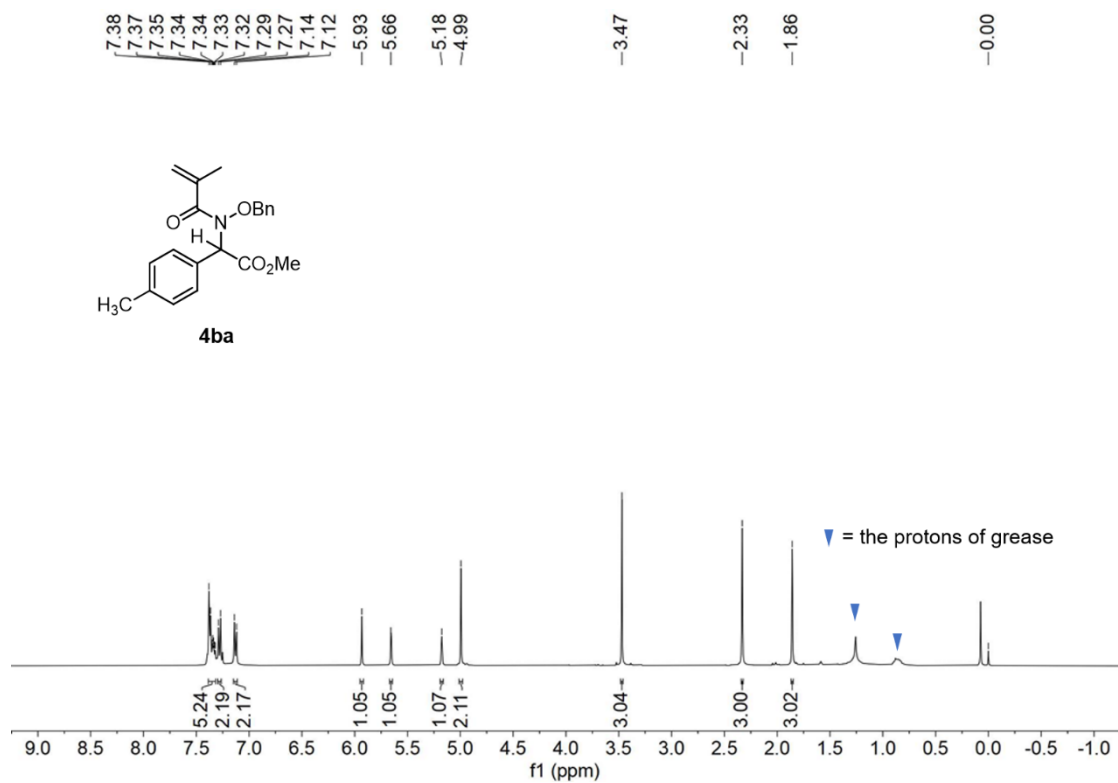
**Fig. S66**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **5**.



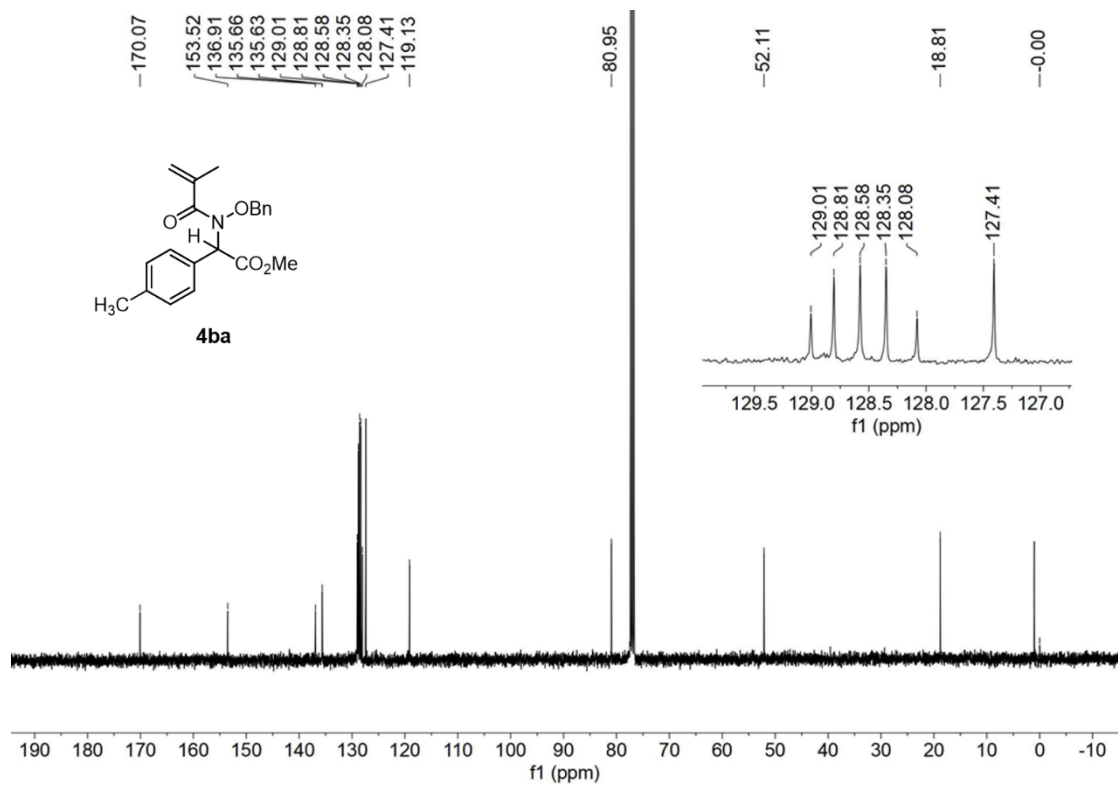
**Fig. S67** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4aa**.



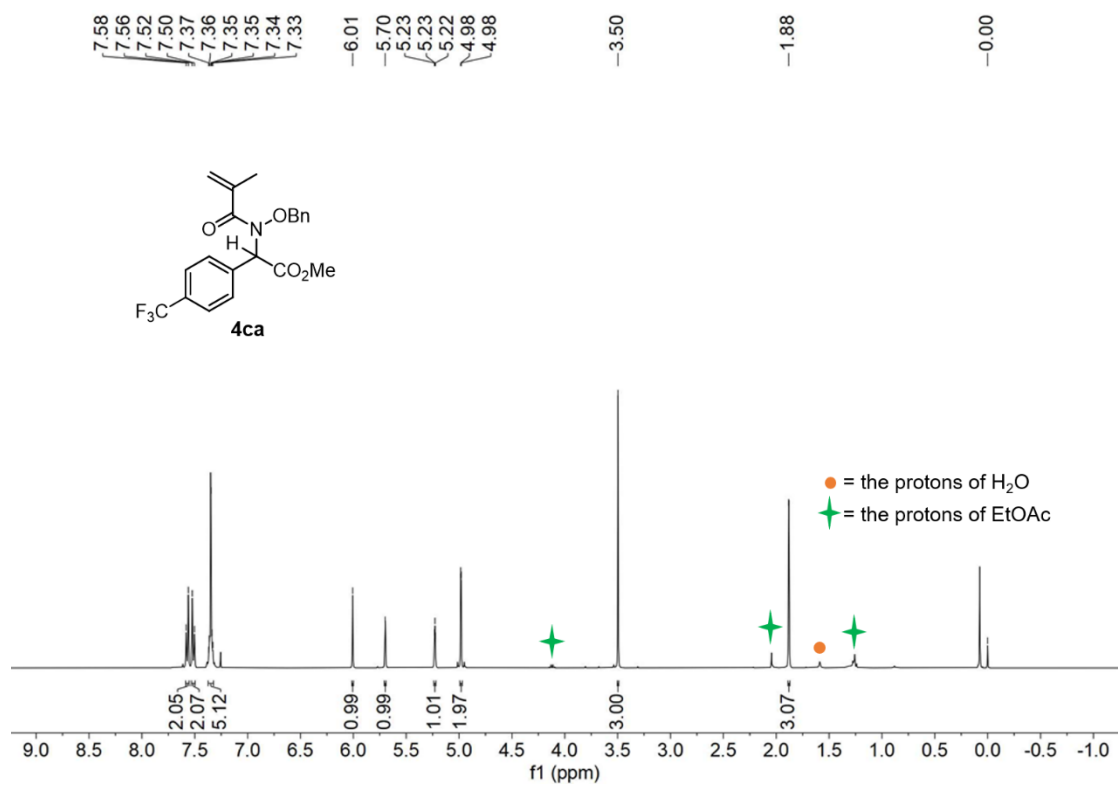
**Fig. S68** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4aa**.



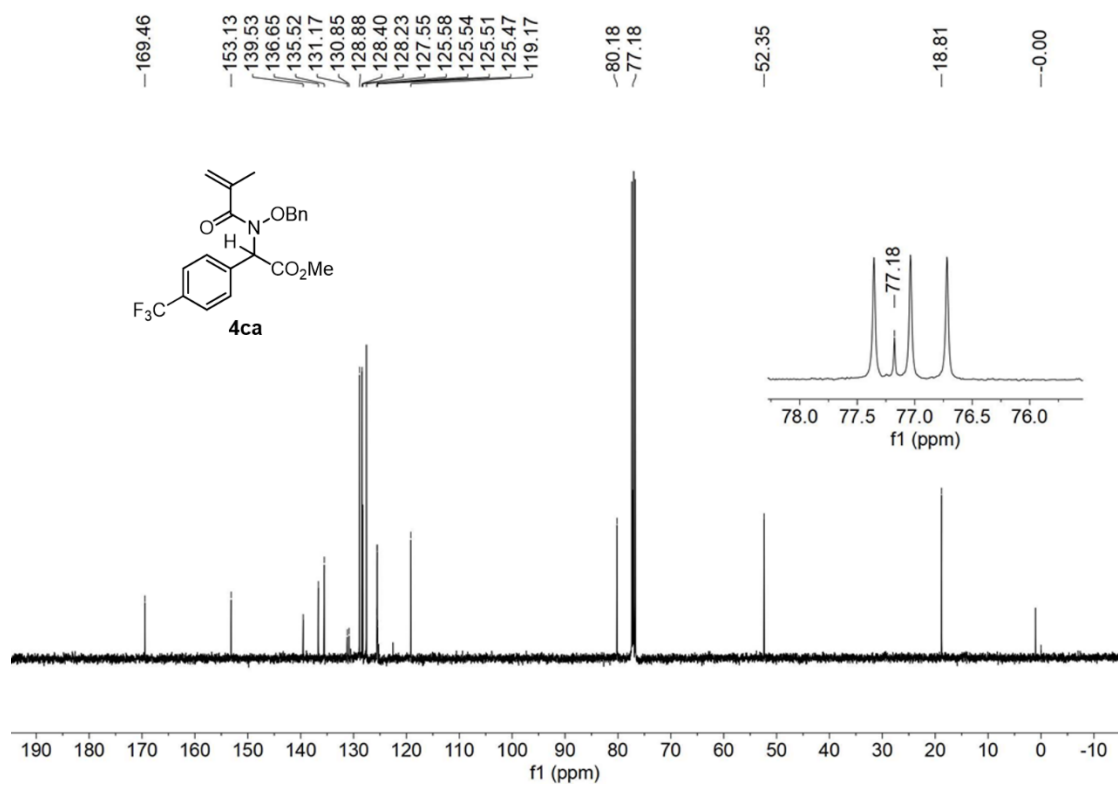
**Fig. S69** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ba**.



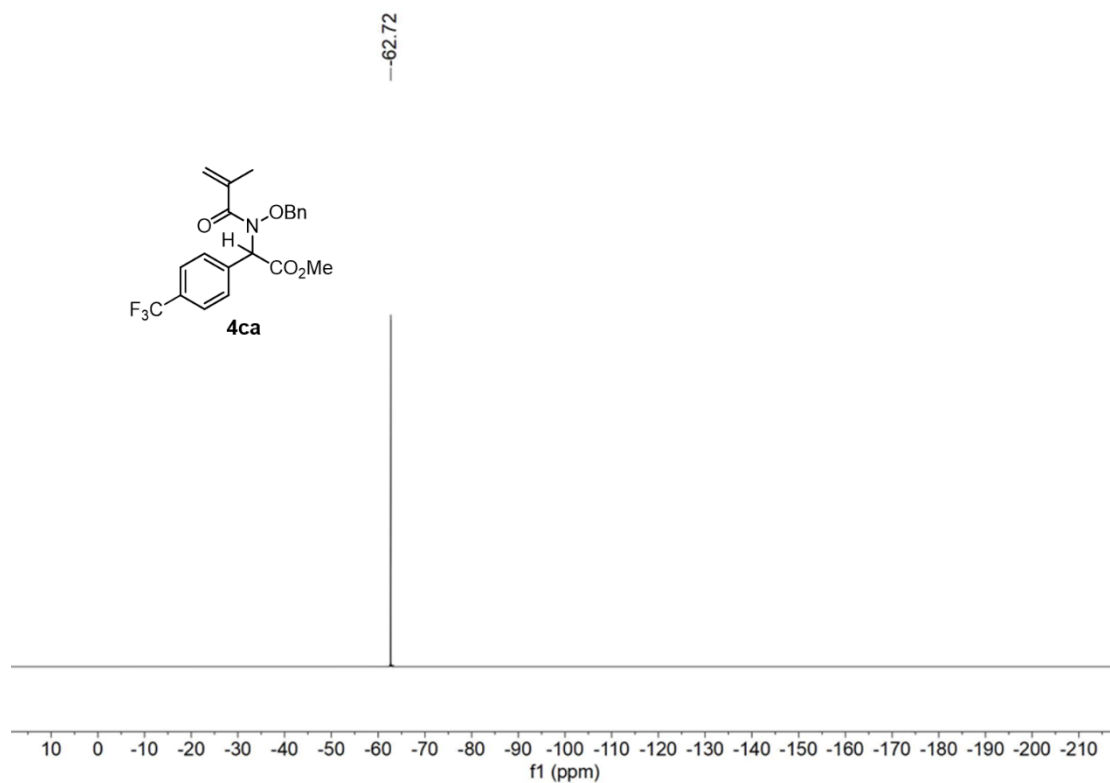
**Fig. S70** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ba**.



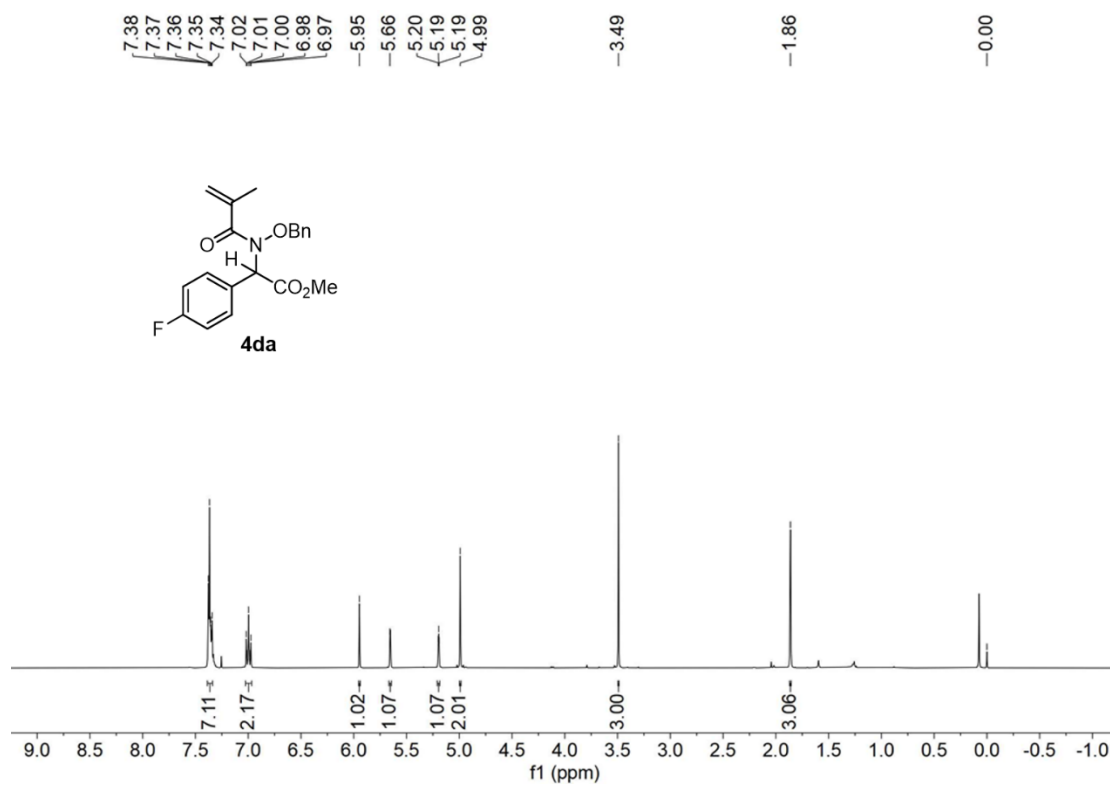
**Fig. S71** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ca**.



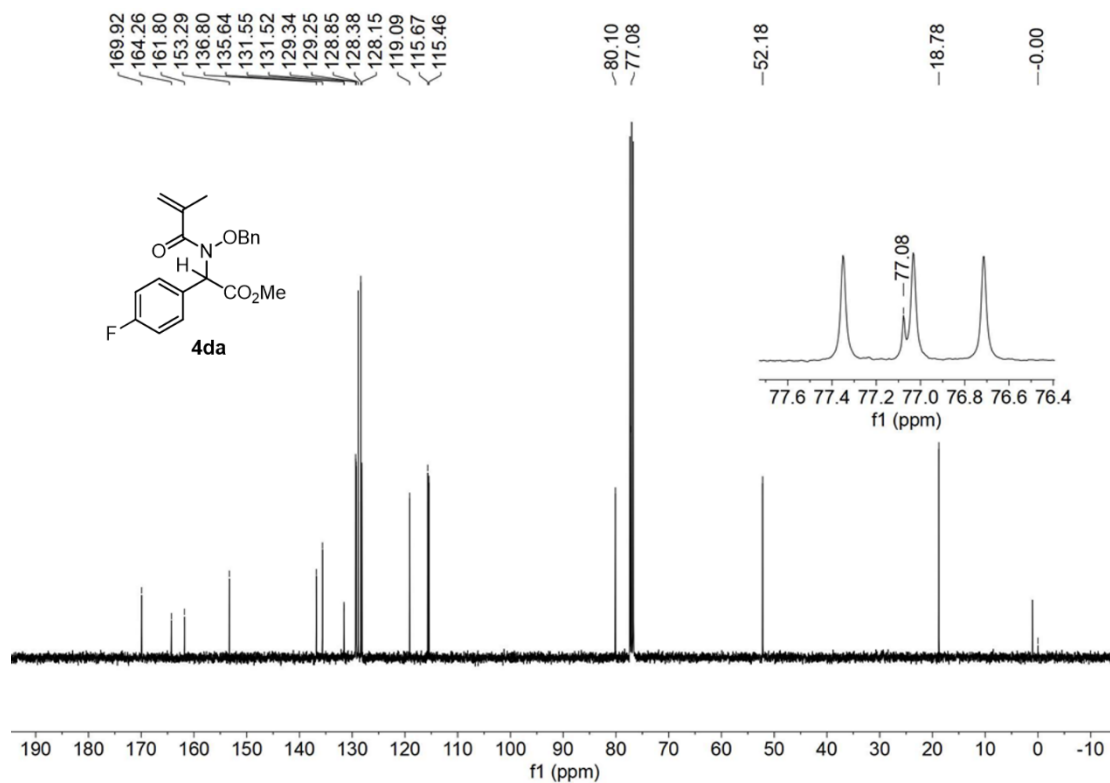
**Fig. S72** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ca**.



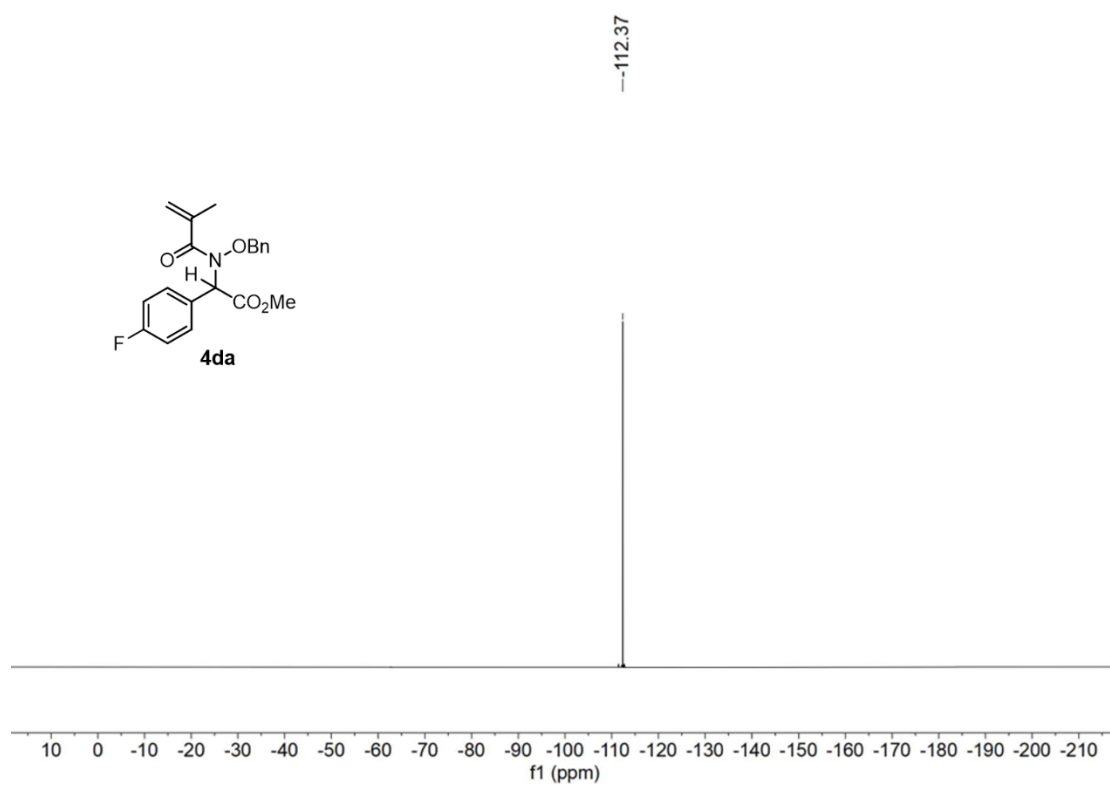
**Fig. S73**  $^{19}\text{F}$  NMR spectrum (376 MHz,  $\text{CDCl}_3$ ) of compound **4ca**.



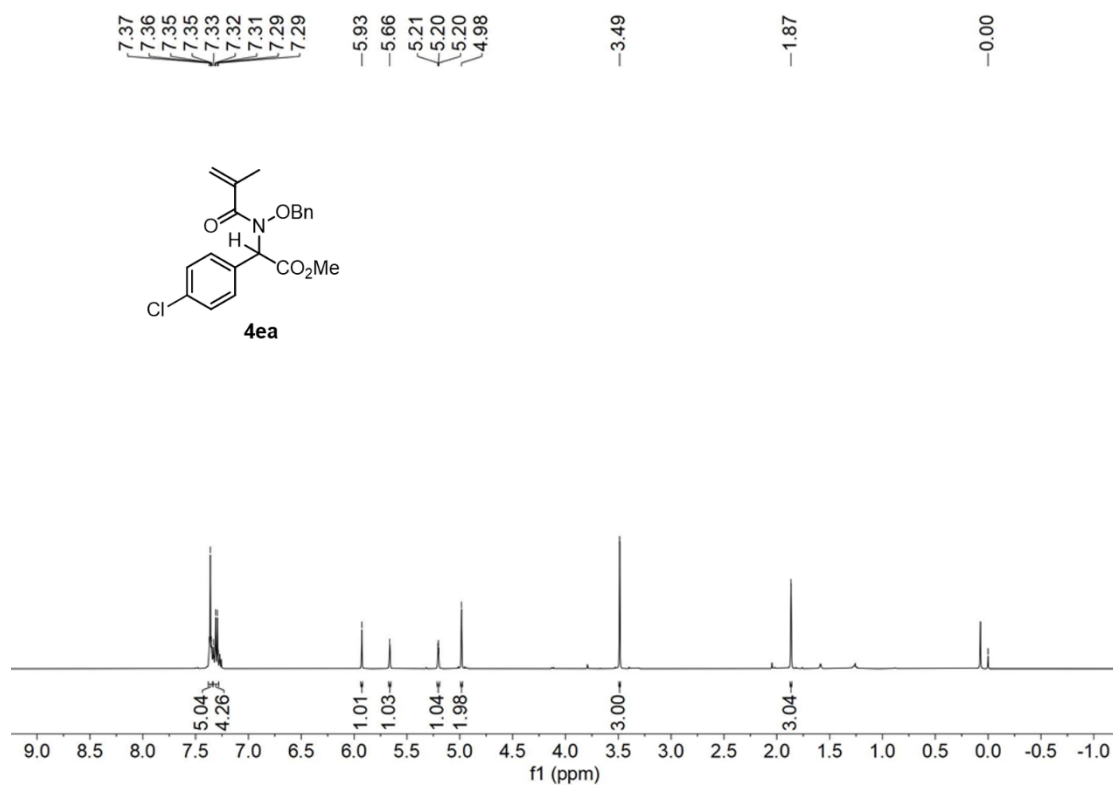
**Fig. S74**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **4da**.



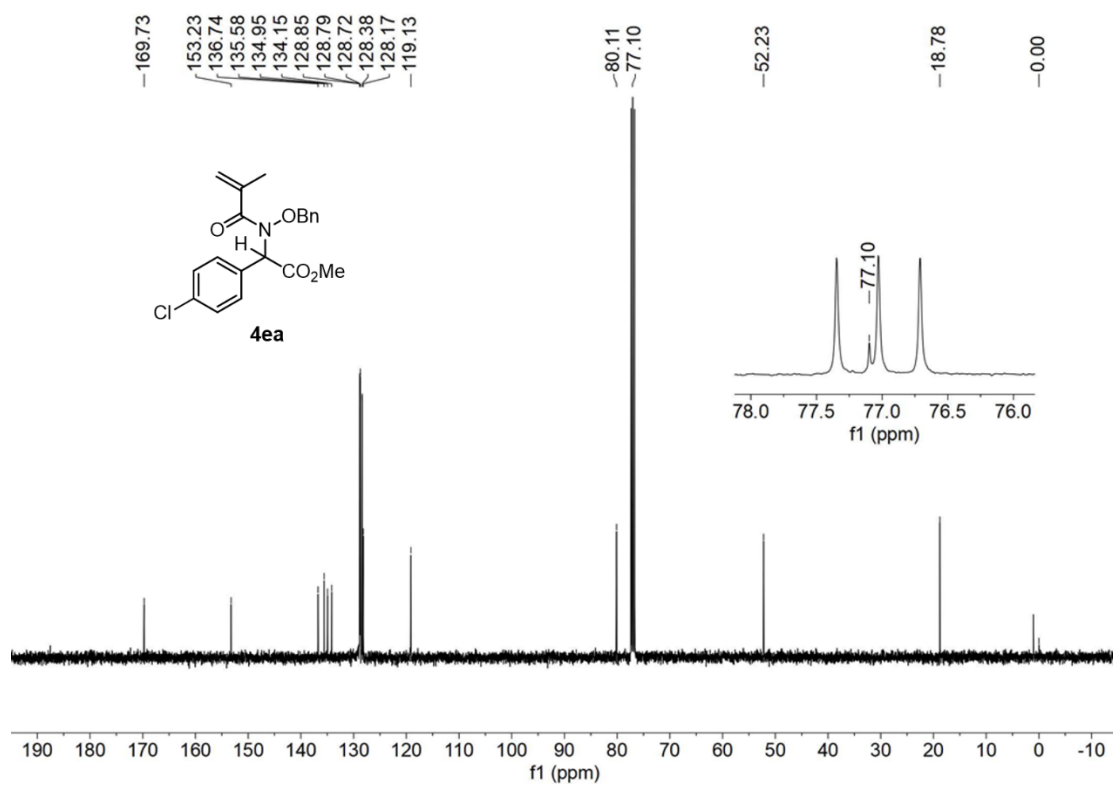
**Fig. S75** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4da**.



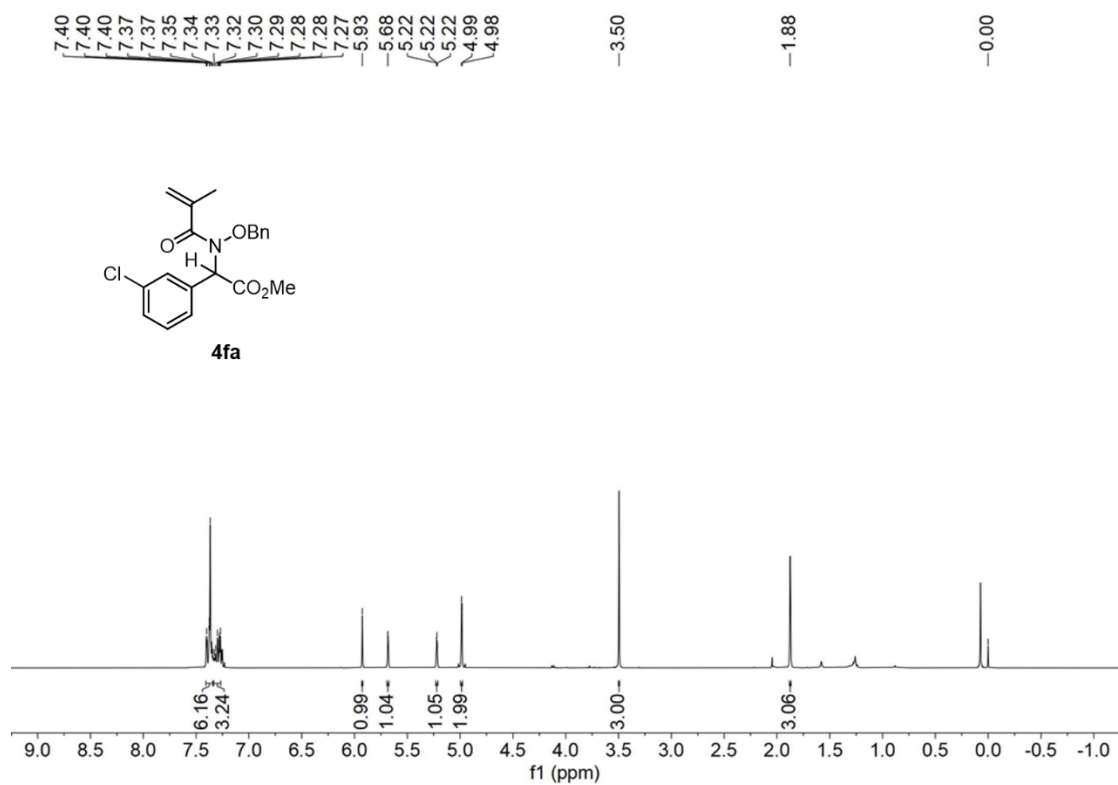
**Fig. S76** <sup>19</sup>F NMR spectrum (376 MHz, CDCl<sub>3</sub>) of compound **4da**.



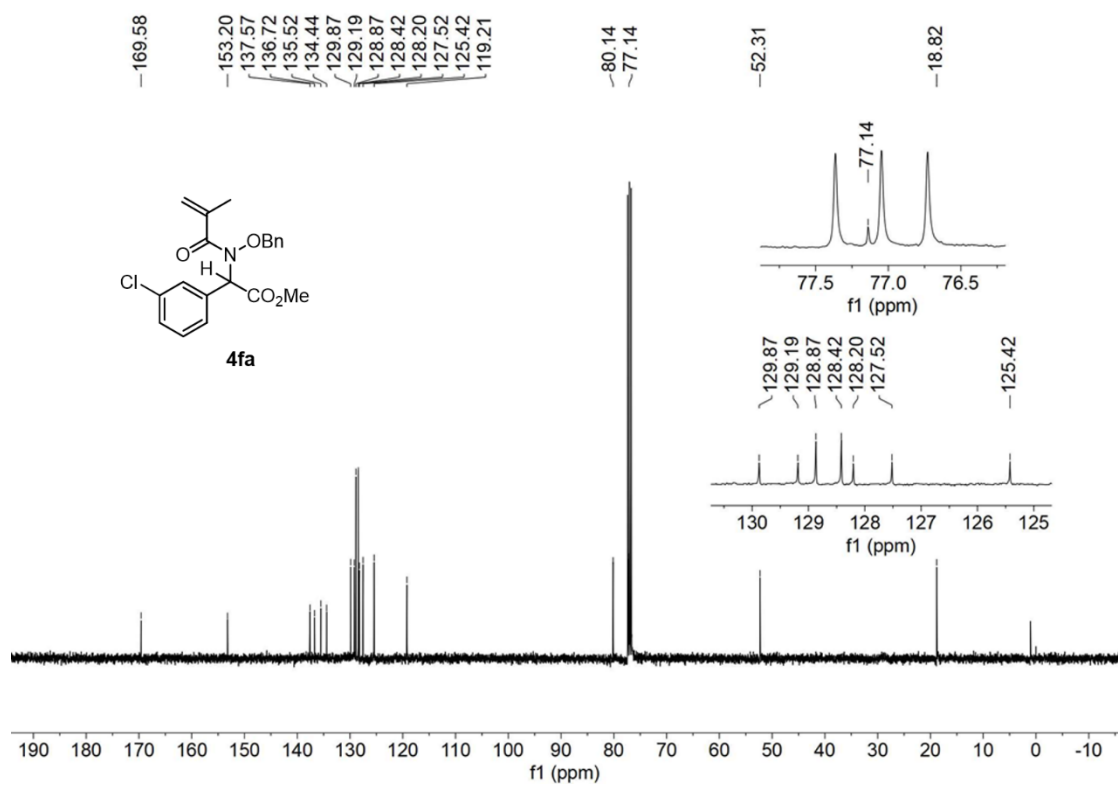
**Fig. S77** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ea**.



**Fig. S78** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ea**.

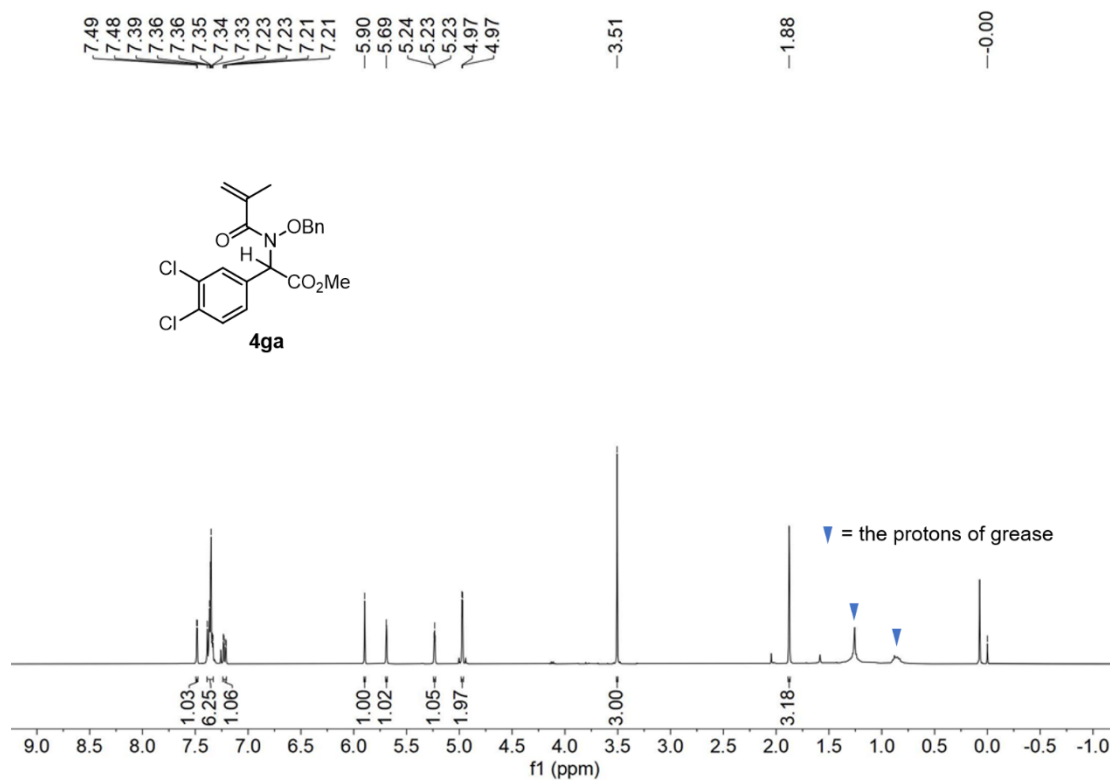


**Fig. S79** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4fa**.

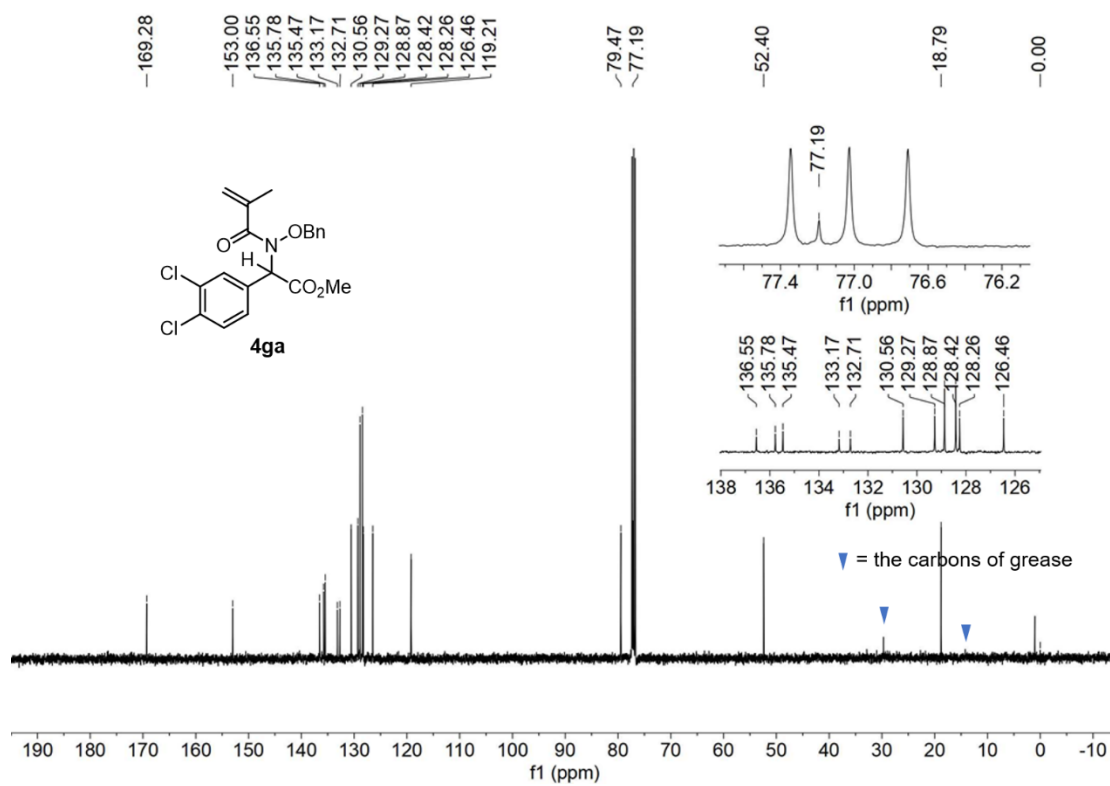


**Fig. S80** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4fa**.

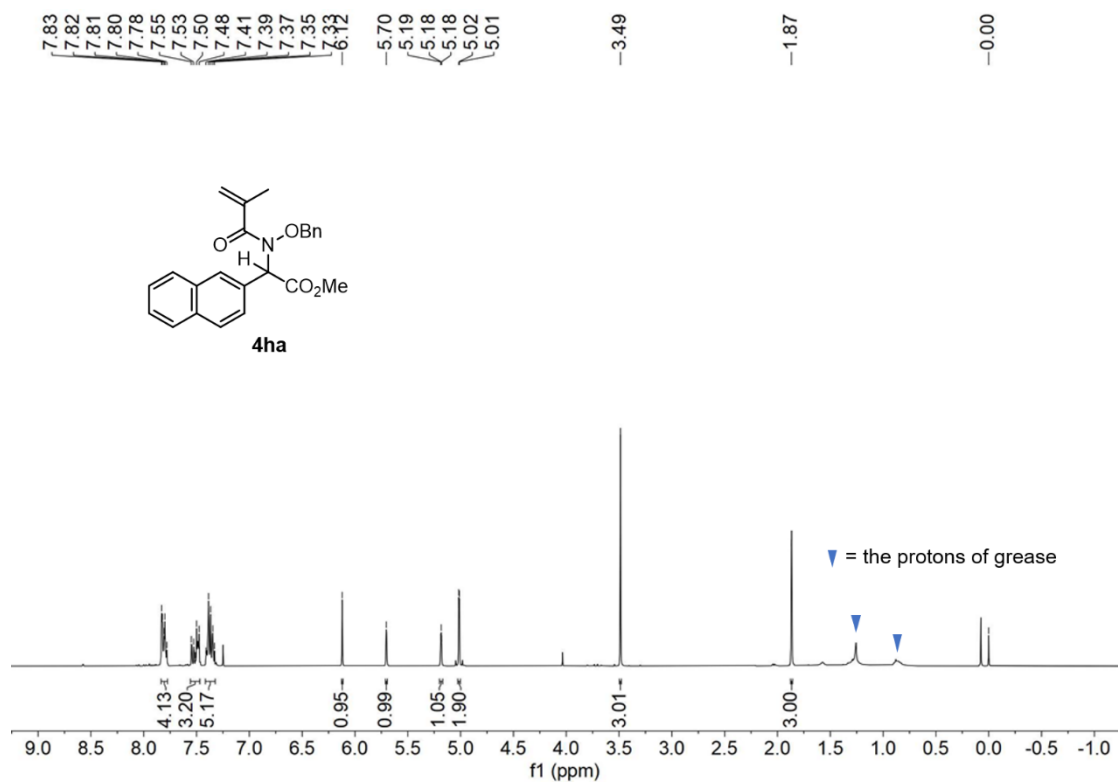




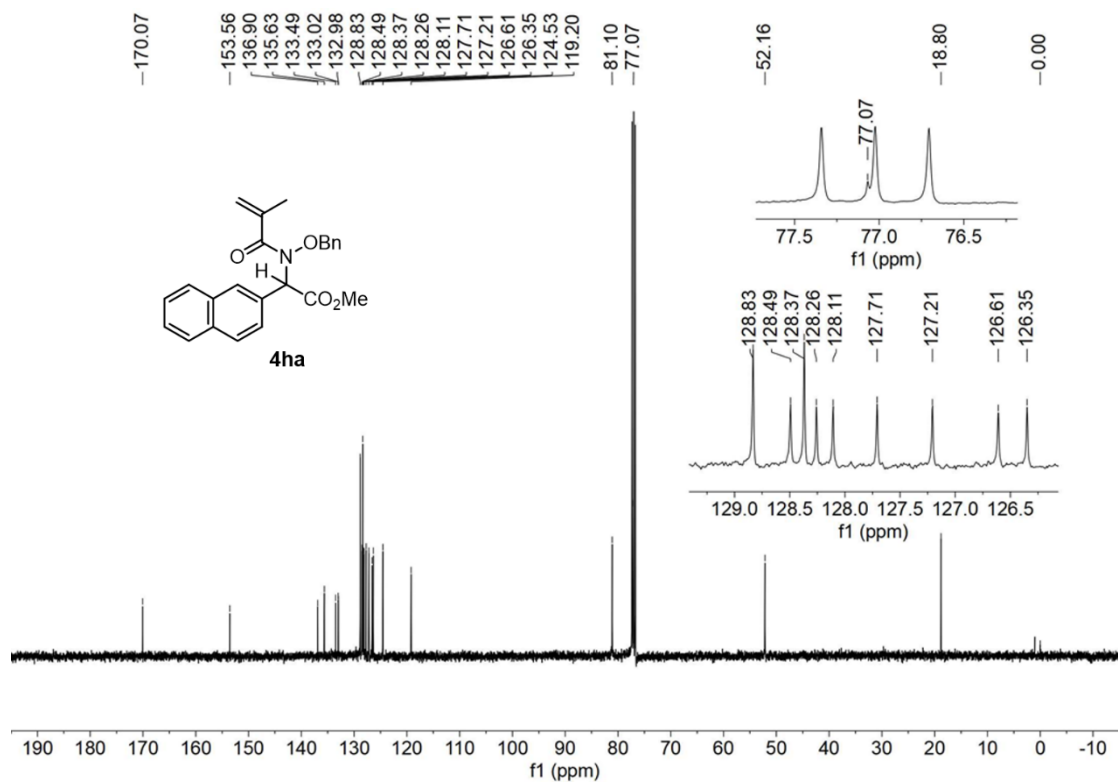
**Fig. S81** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ga**.



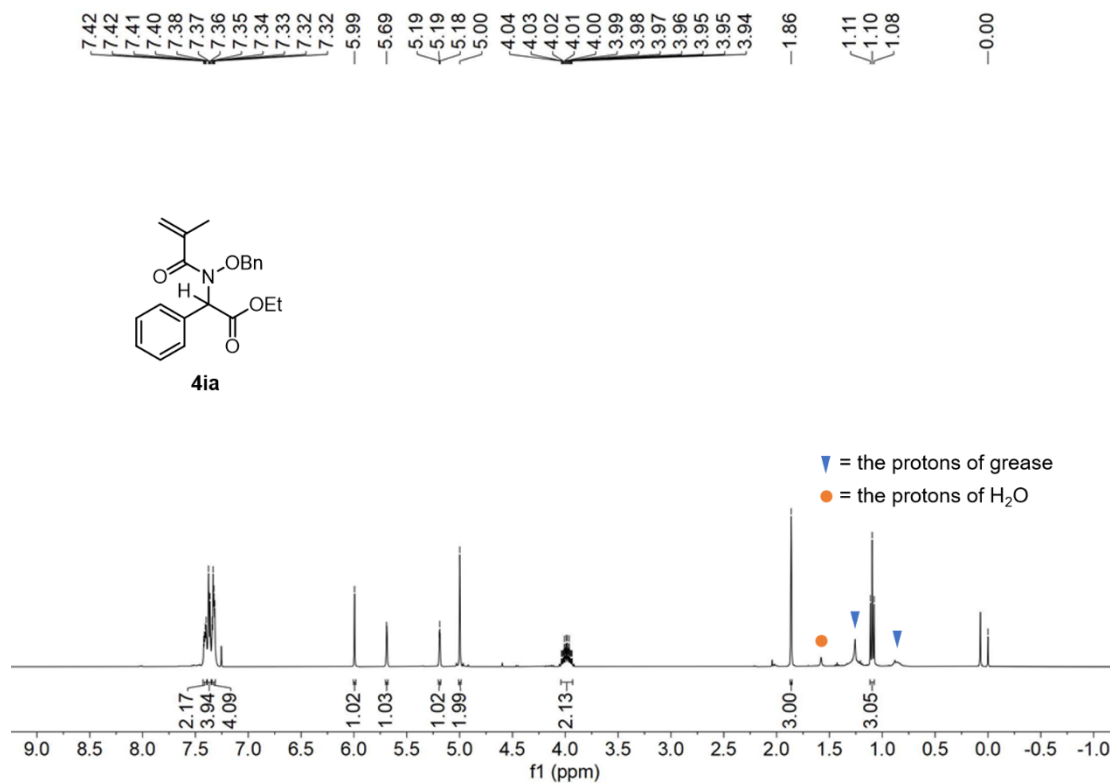
**Fig. S82** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ga**.



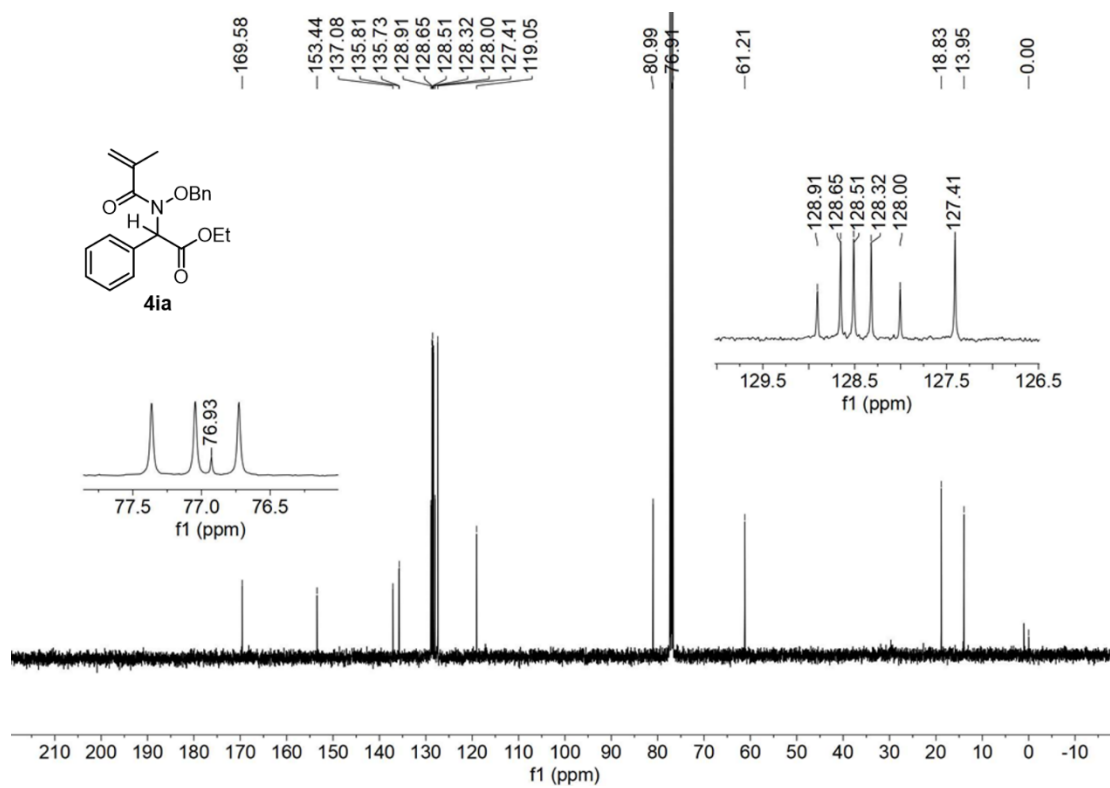
**Fig. S83** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ha**.



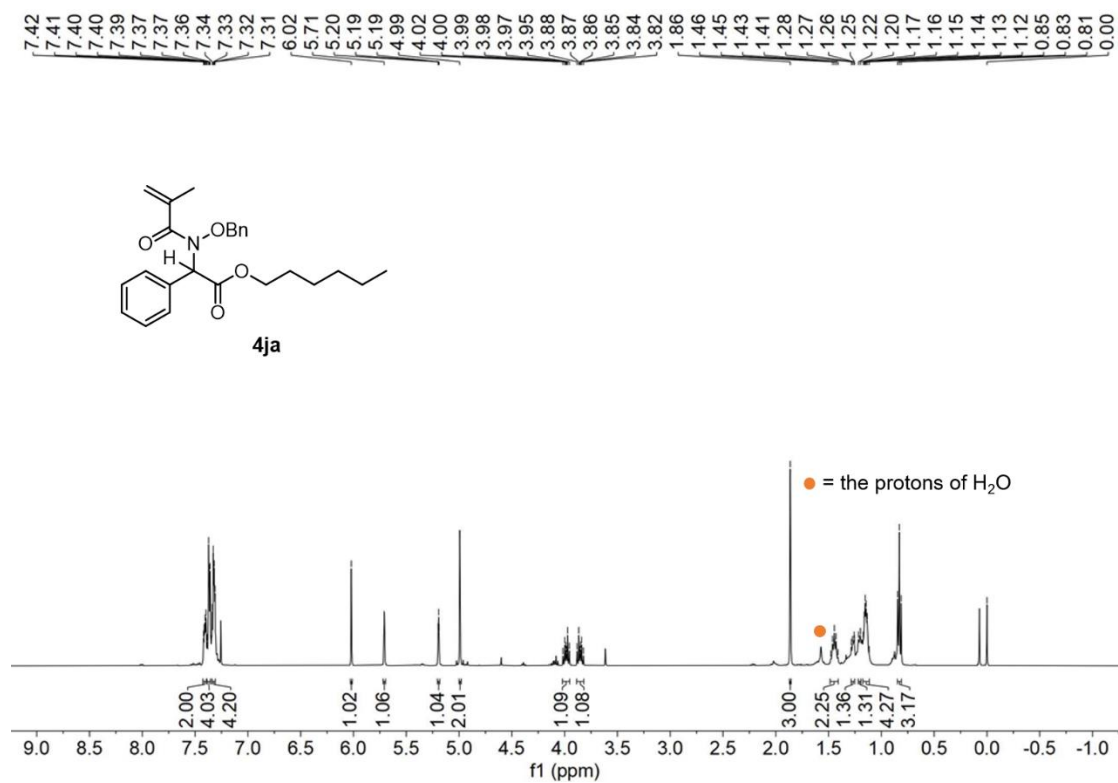
**Fig. S84** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ha**.



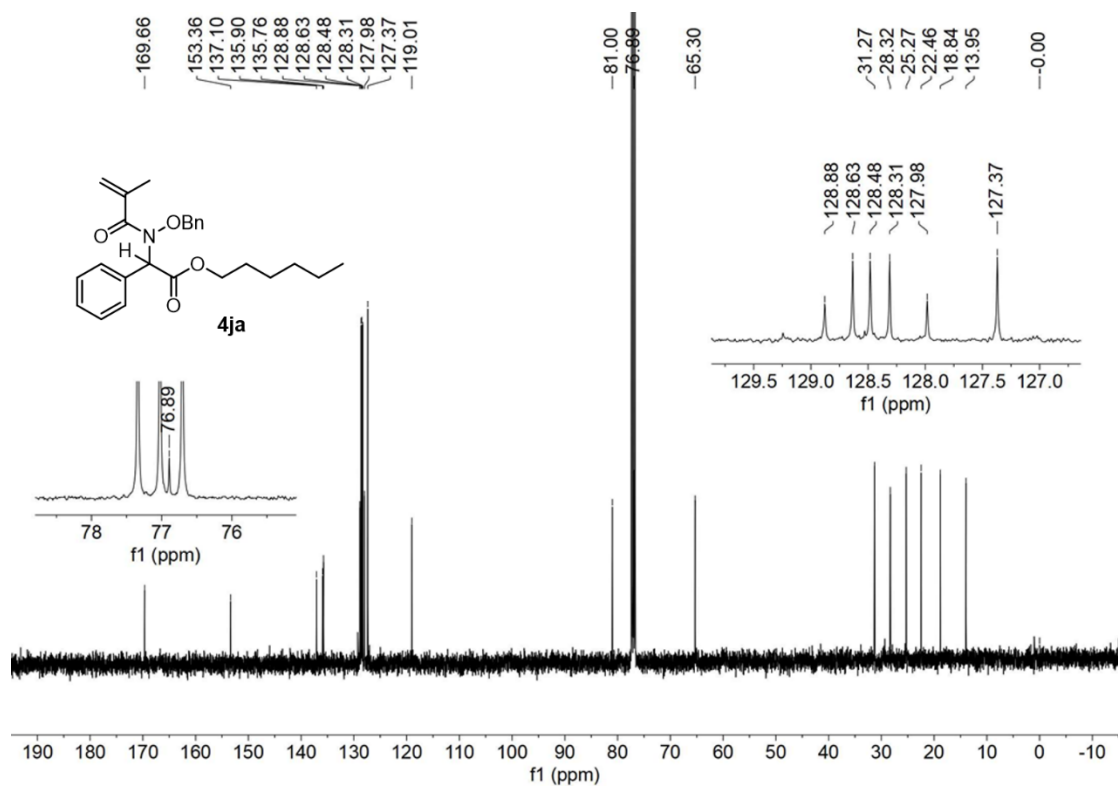
**Fig. S85** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ia**.



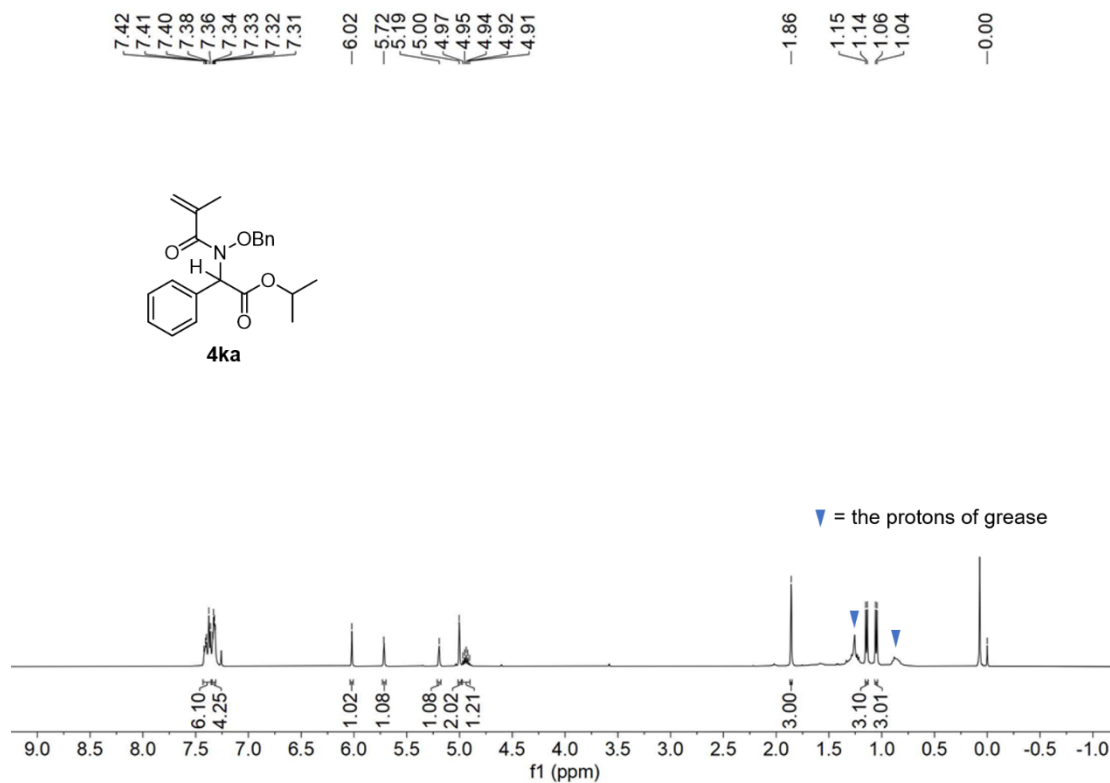
**Fig. S86** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ia**.



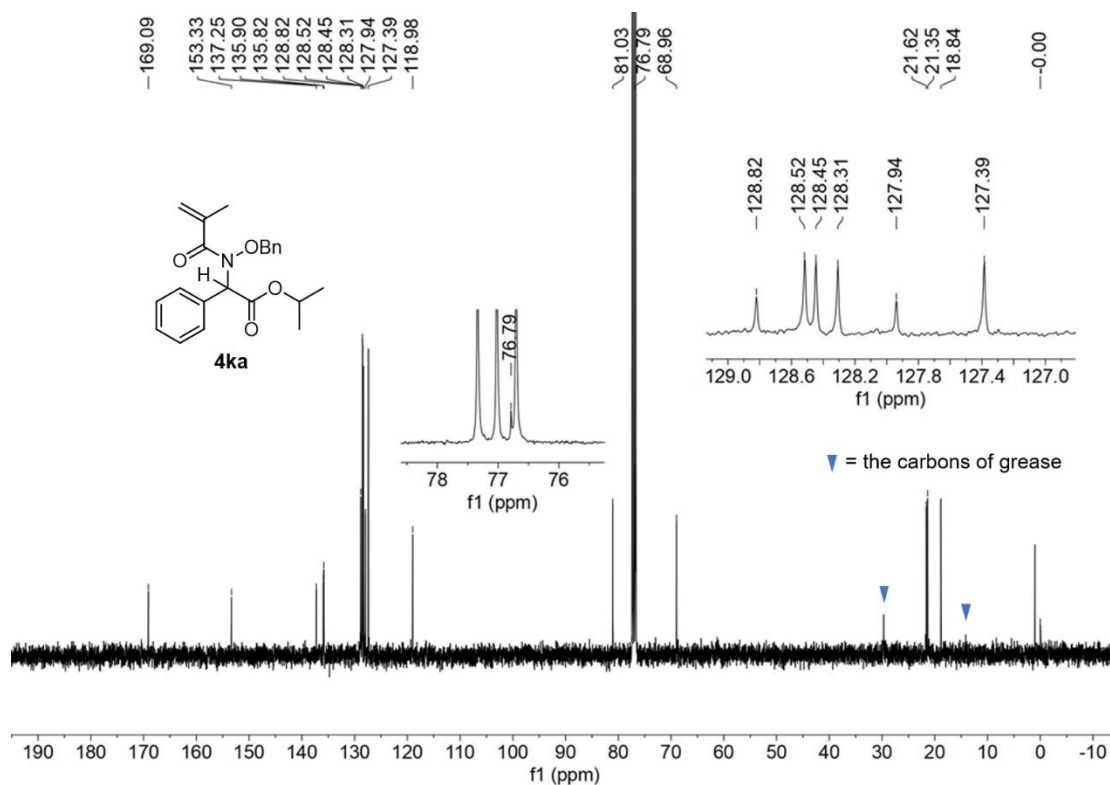
**Fig. S87** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ja**.



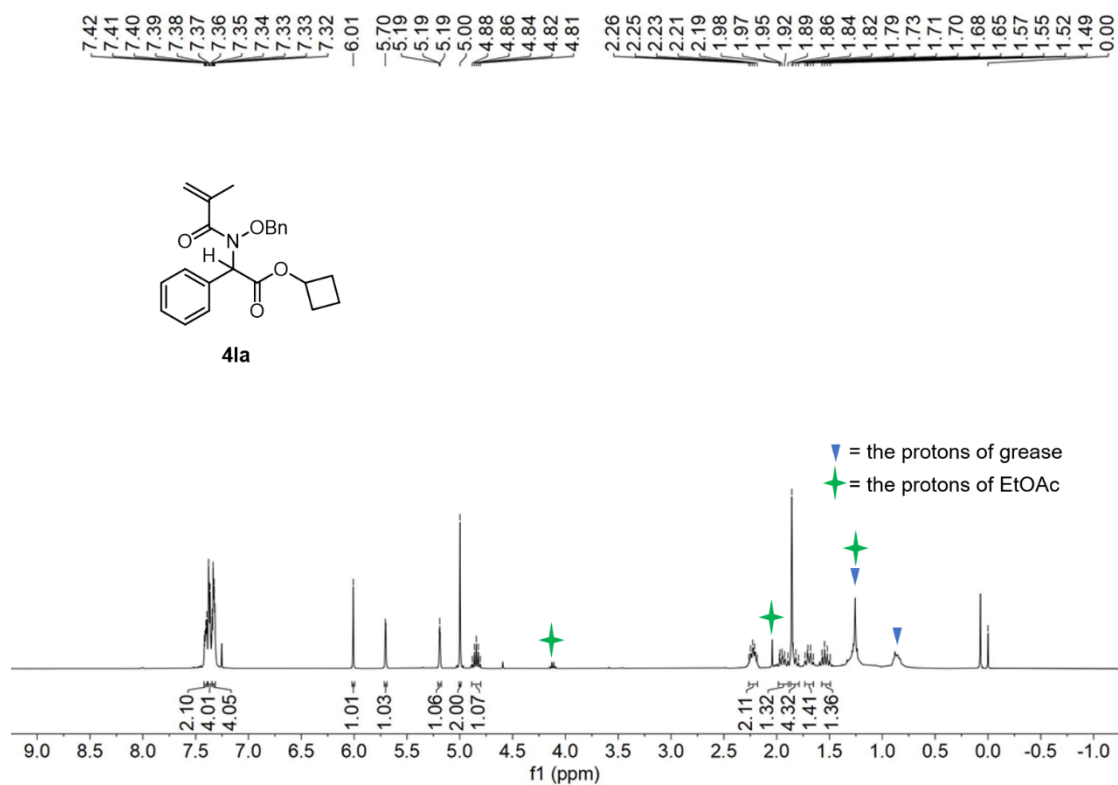
**Fig. S88** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ja**.



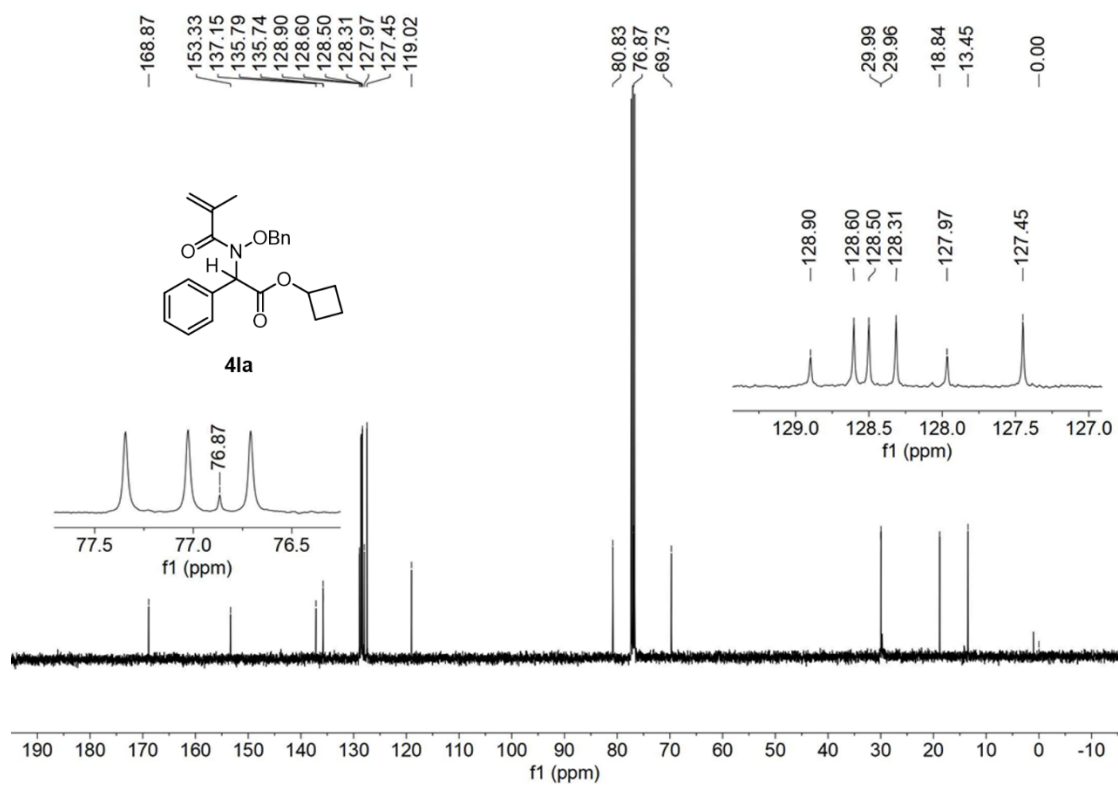
**Fig. S89**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **4ka**.



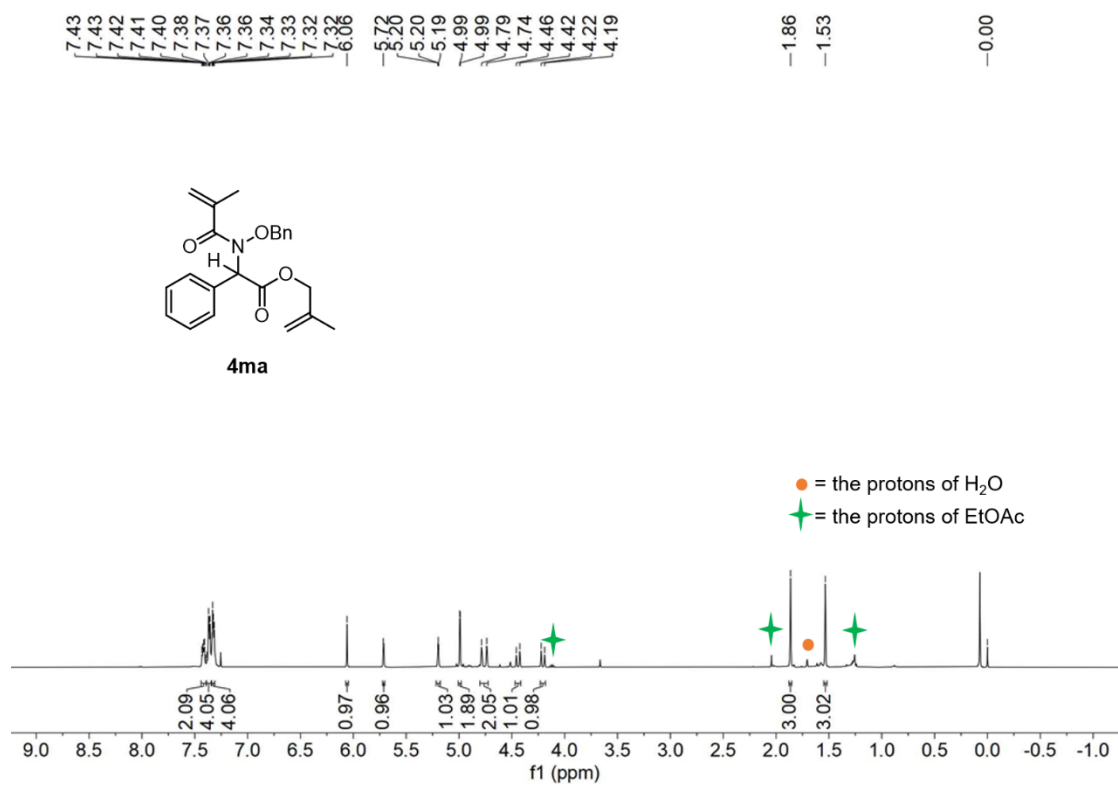
**Fig. S90**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **4ka**.



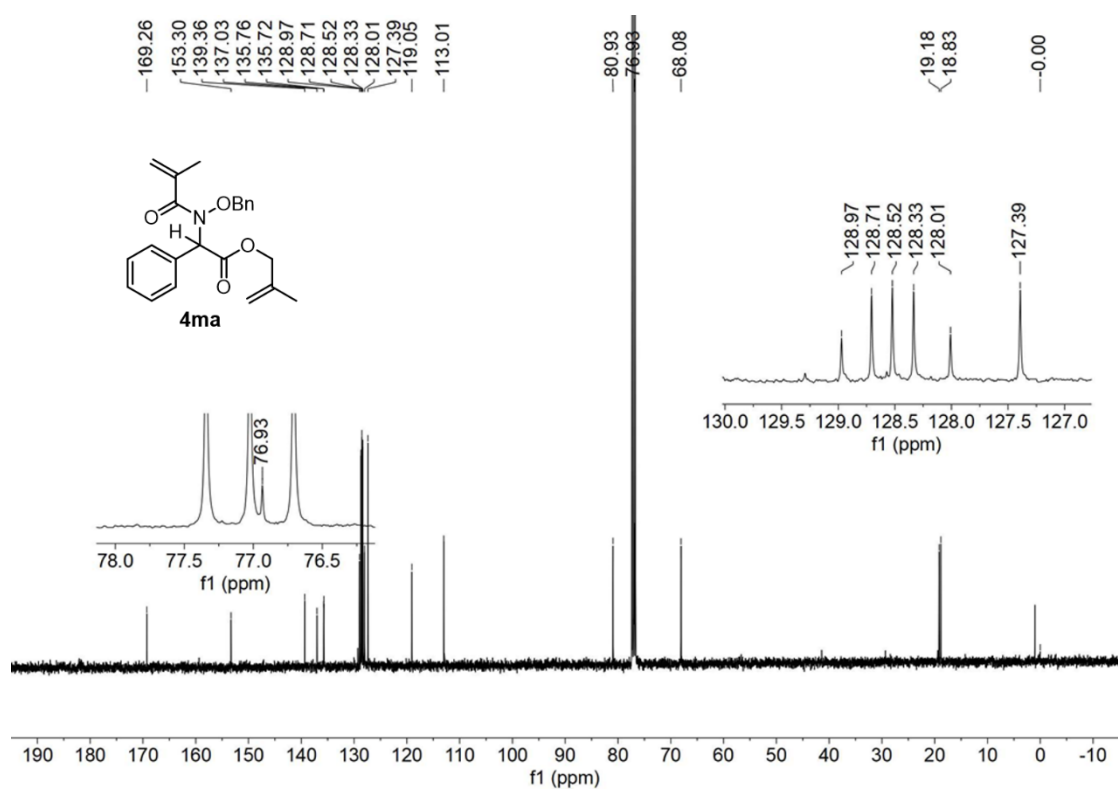
**Fig. S91**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **4la**.



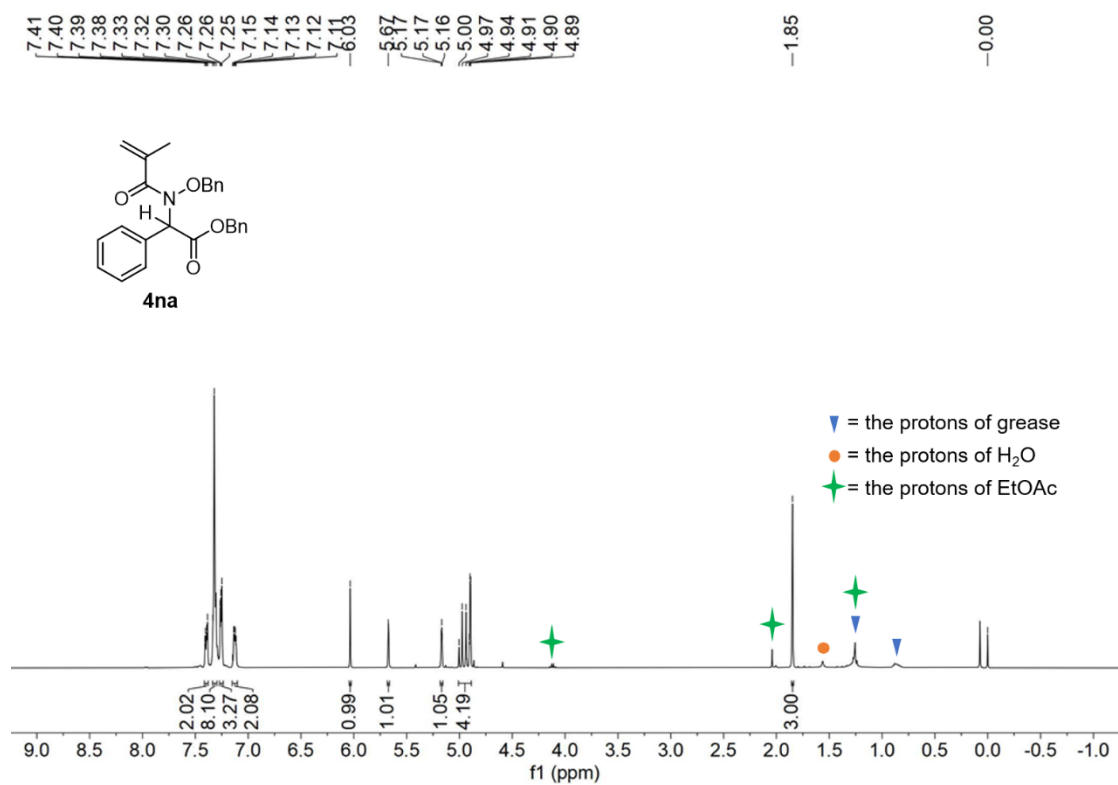
**Fig. S92**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{CDCl}_3$ ) of compound **4la**.



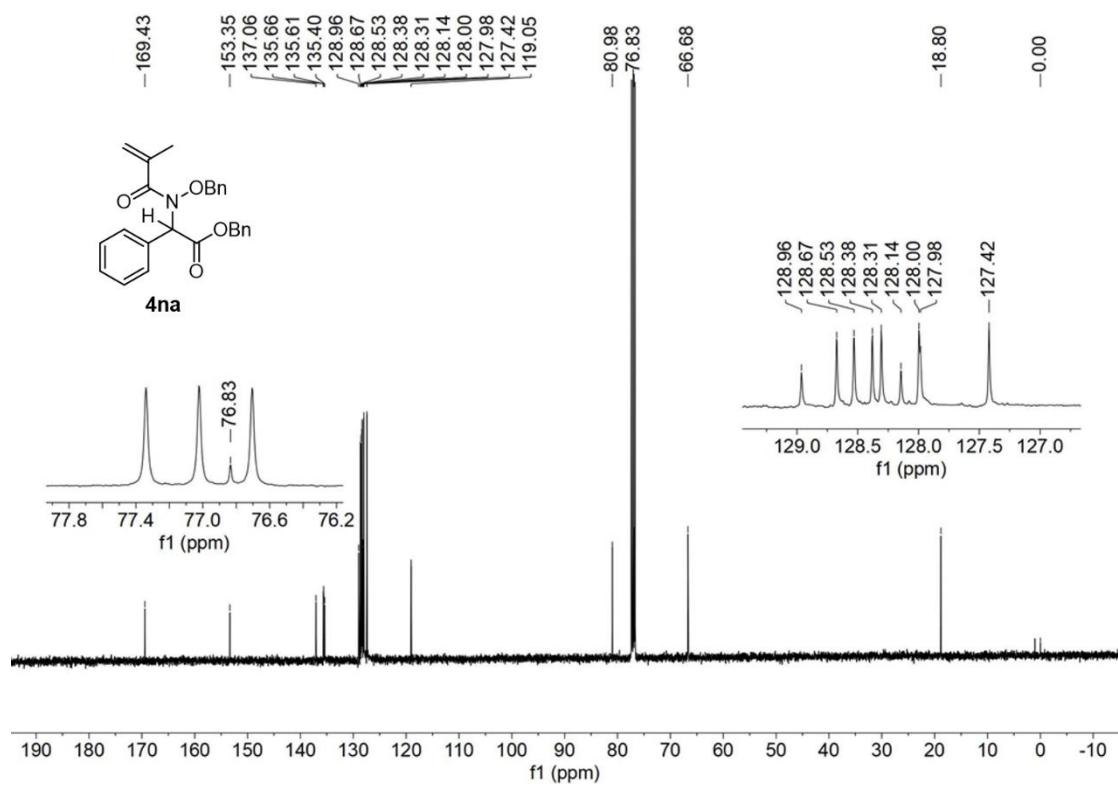
**Fig. S93** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ma**.



**Fig. S94** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ma**.

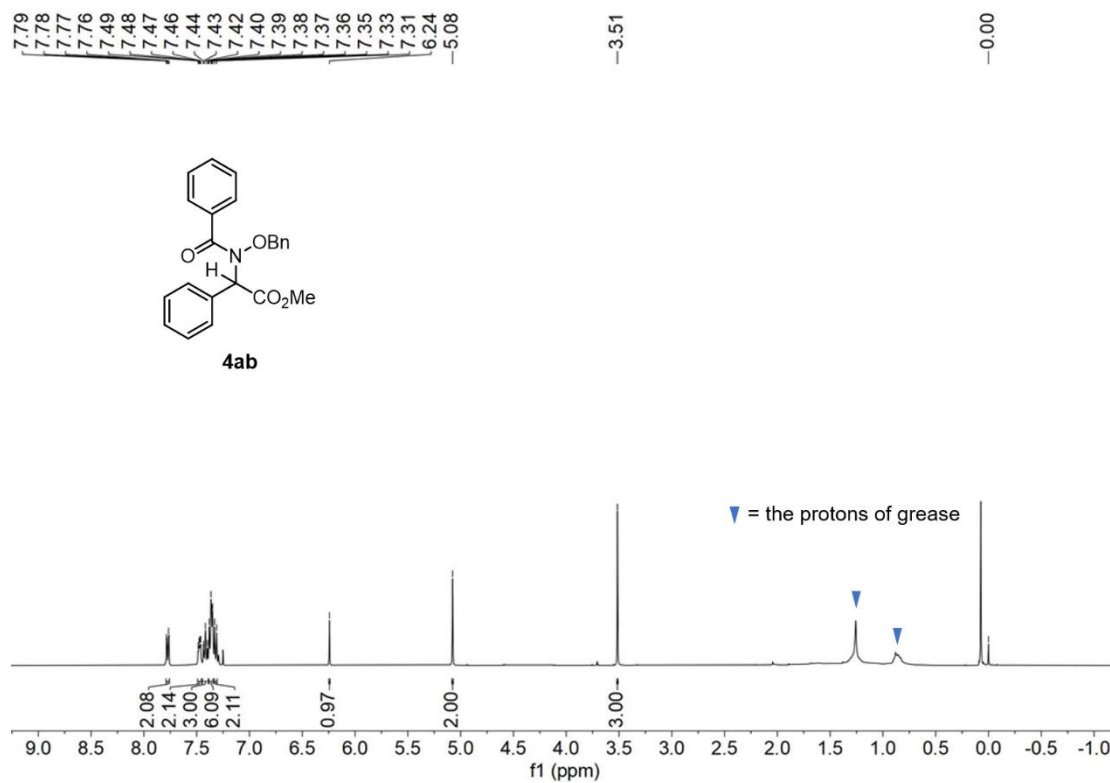


**Fig. S95** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4na**.

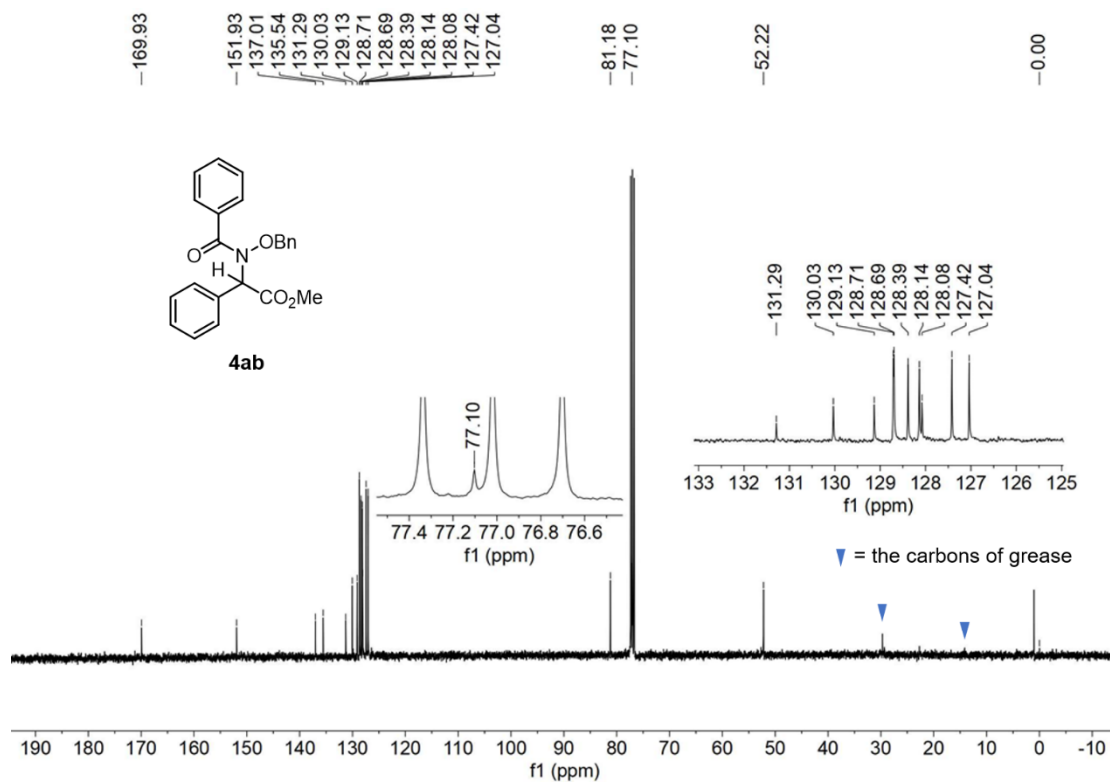


**Fig. S96** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4na**.

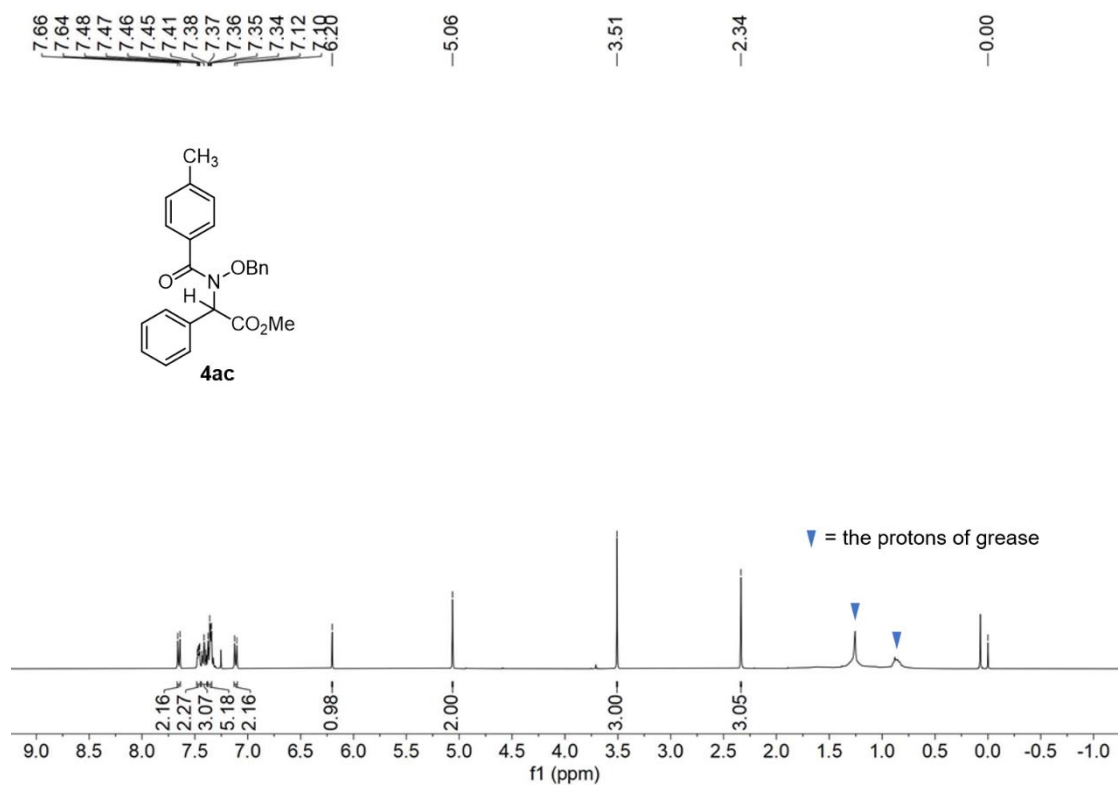




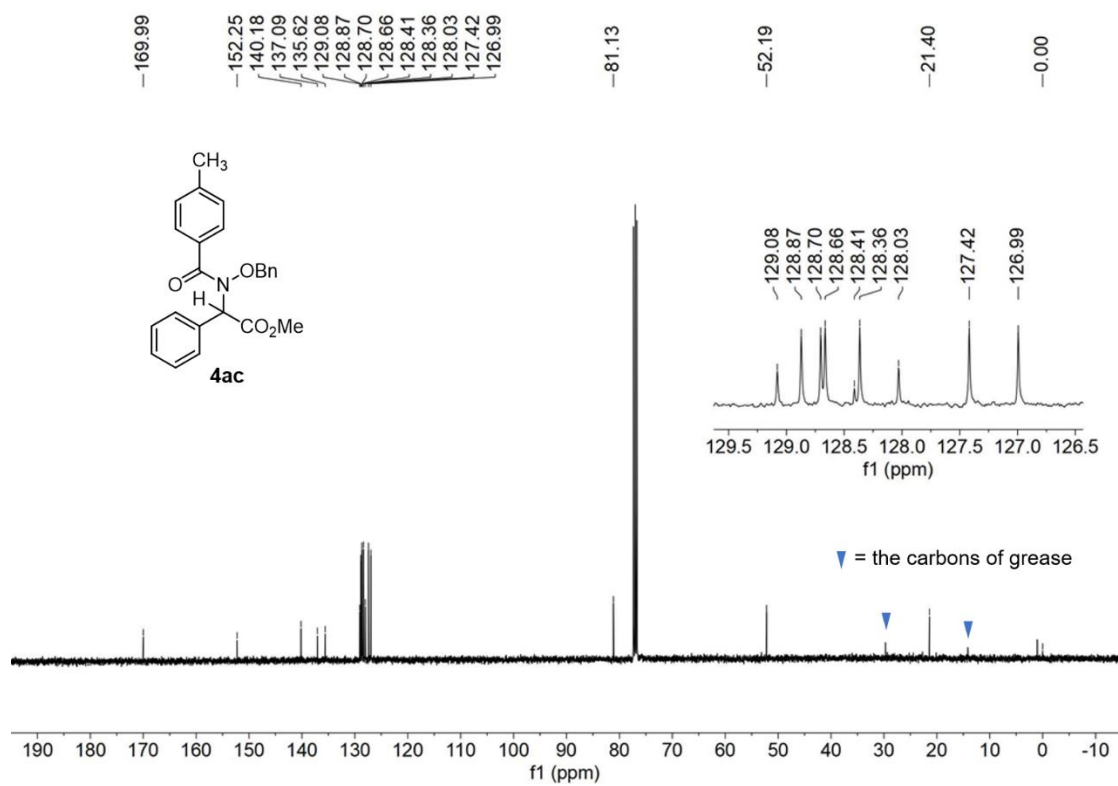
**Fig. S97** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ab**.



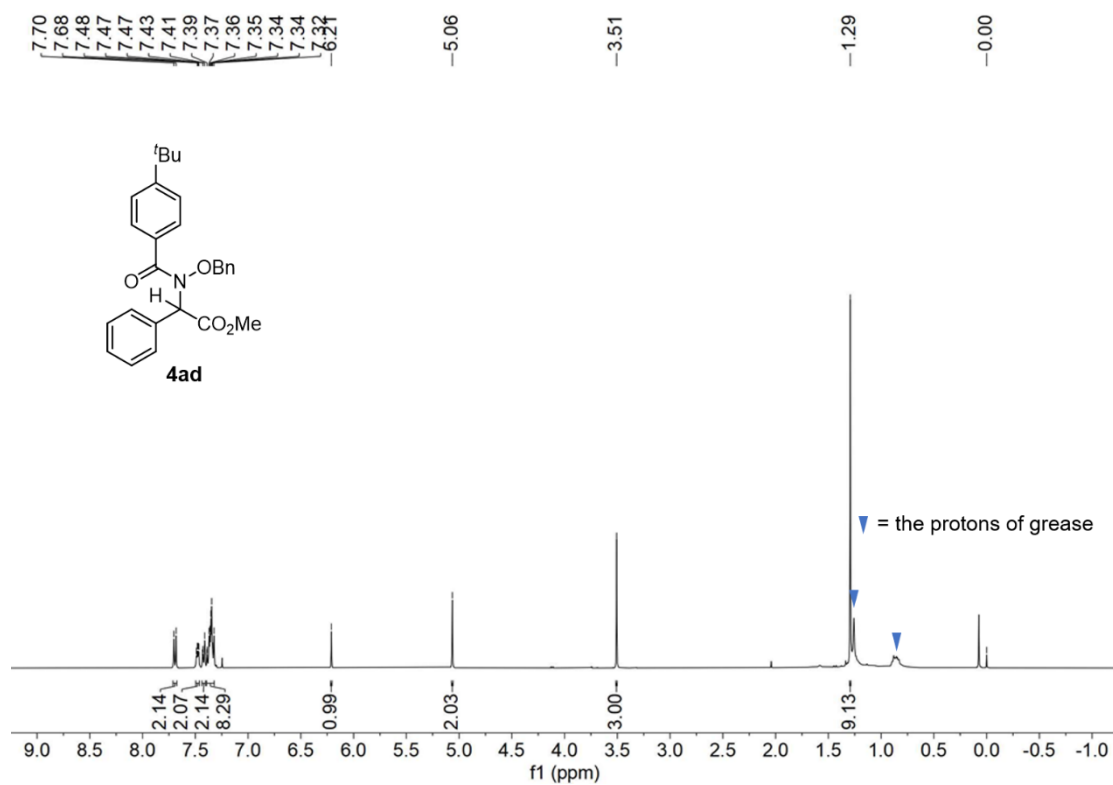
**Fig. S98** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ab**.



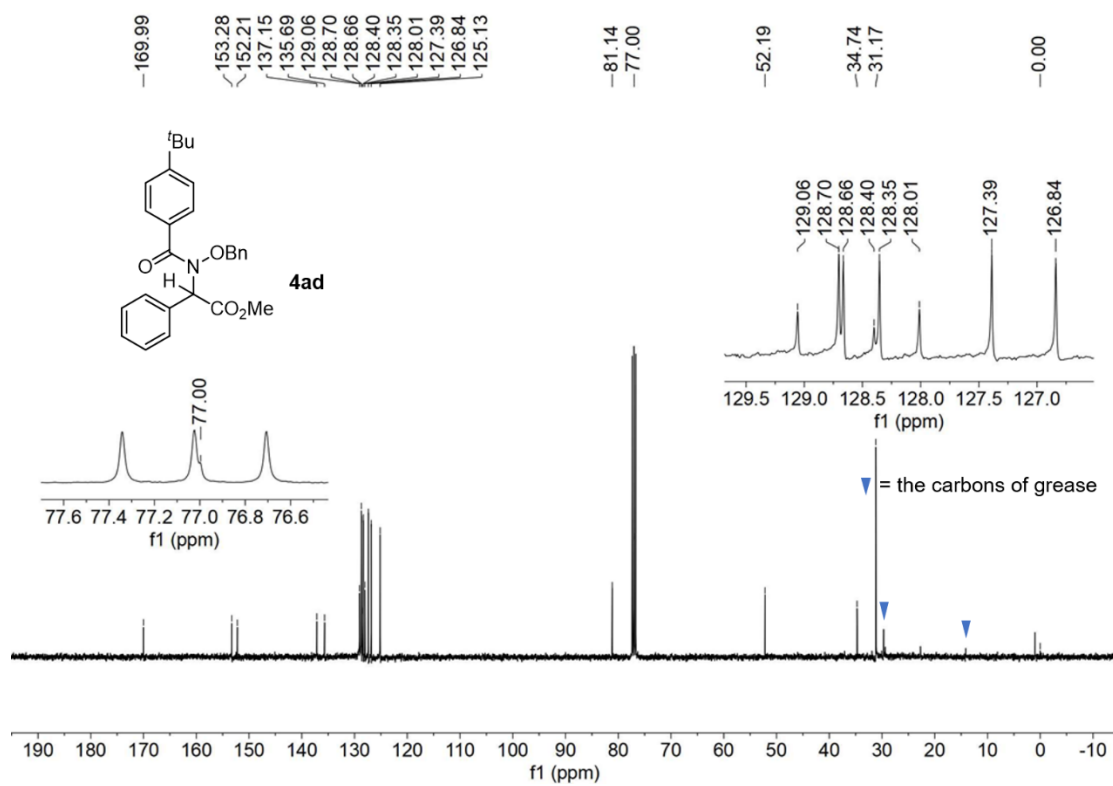
**Fig. S99** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ac**.



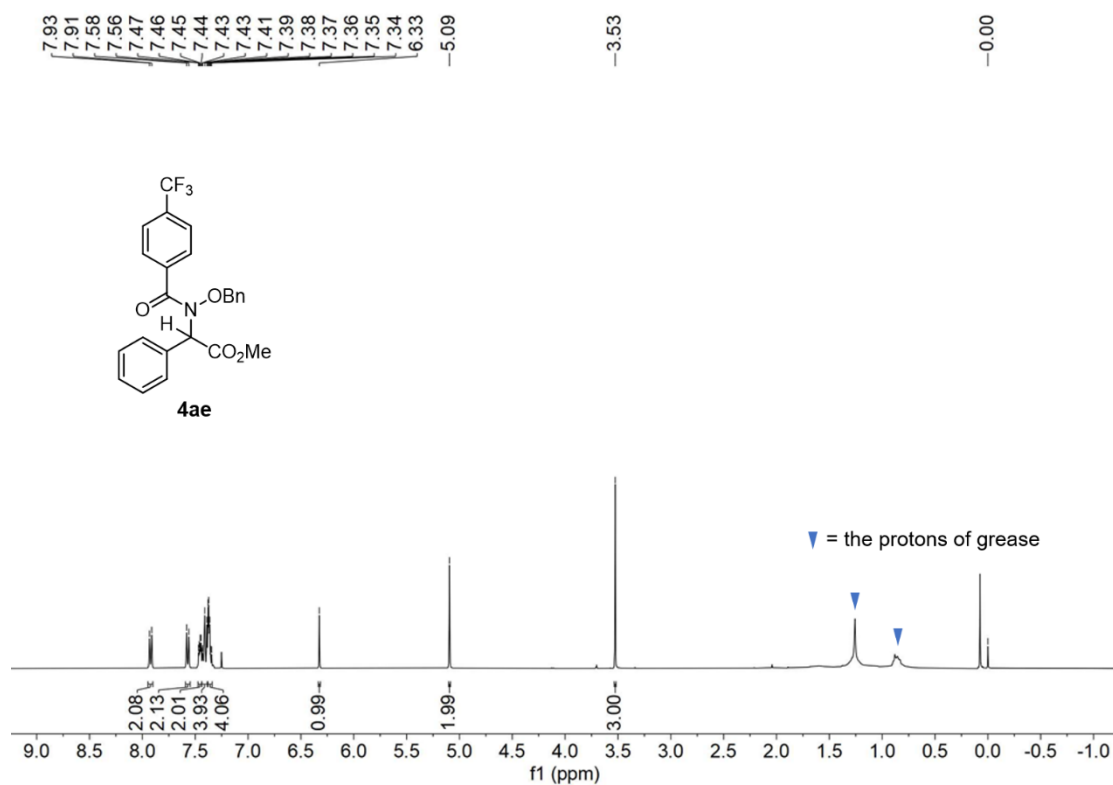
**Fig. S100** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ac**.



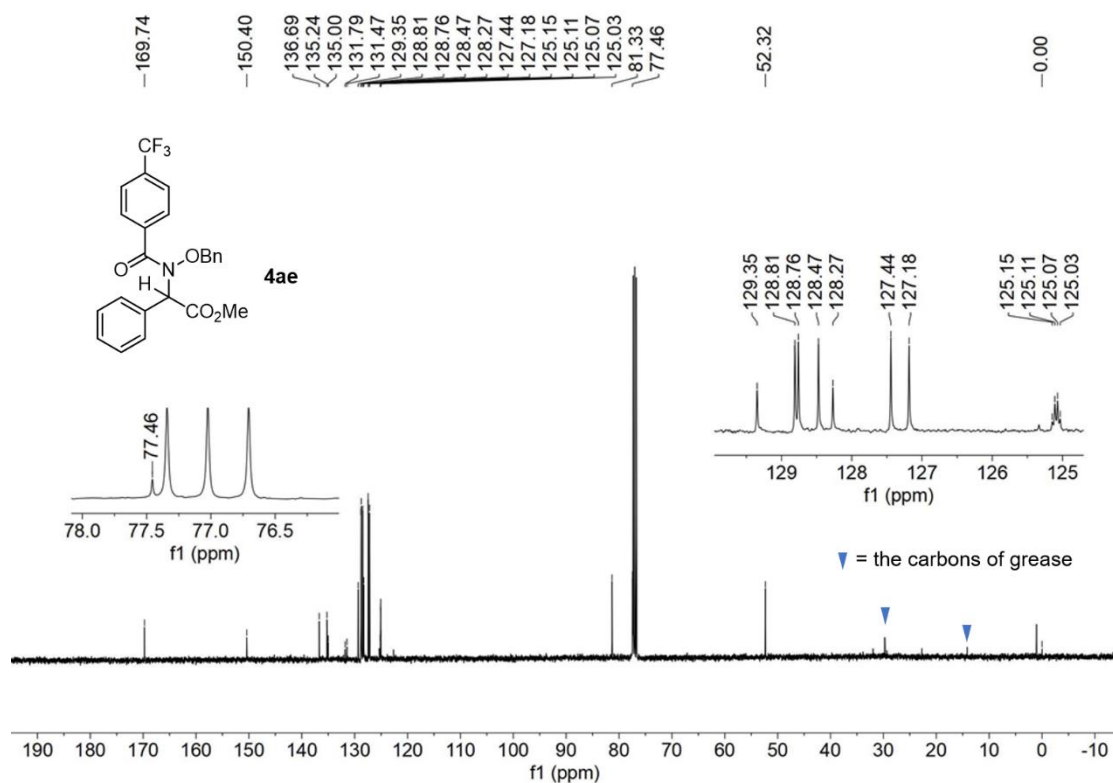
**Fig. S101** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ad**.



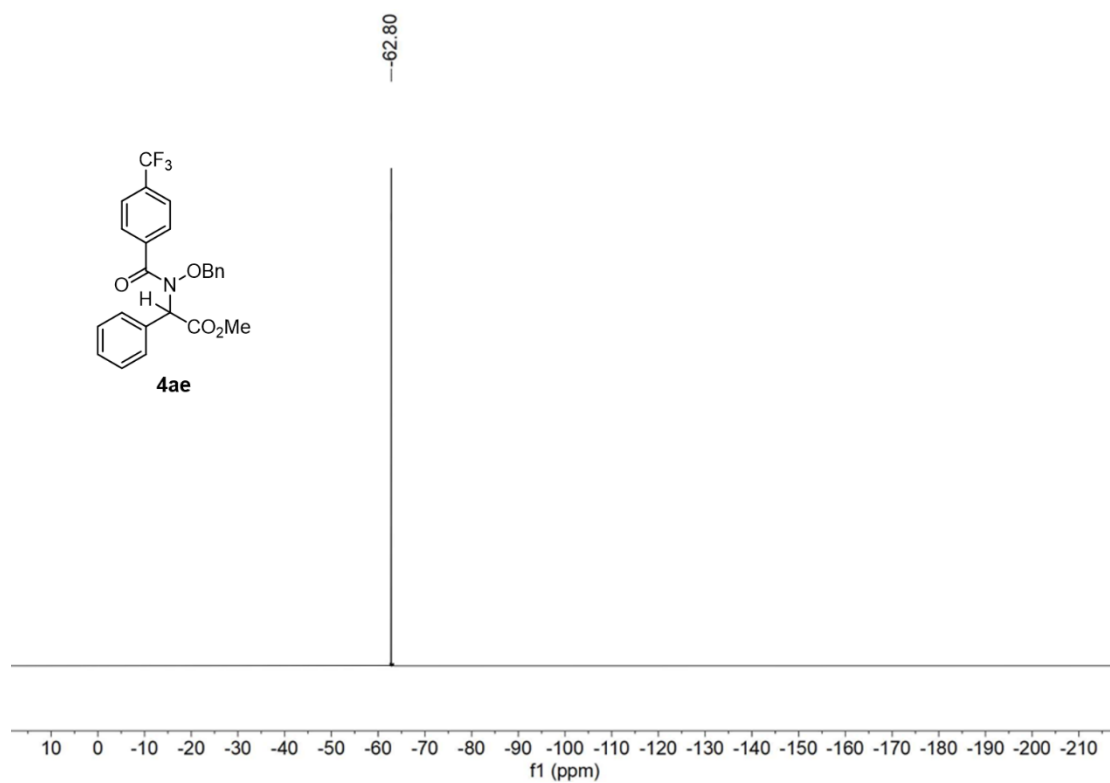
**Fig. S102** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ad**.



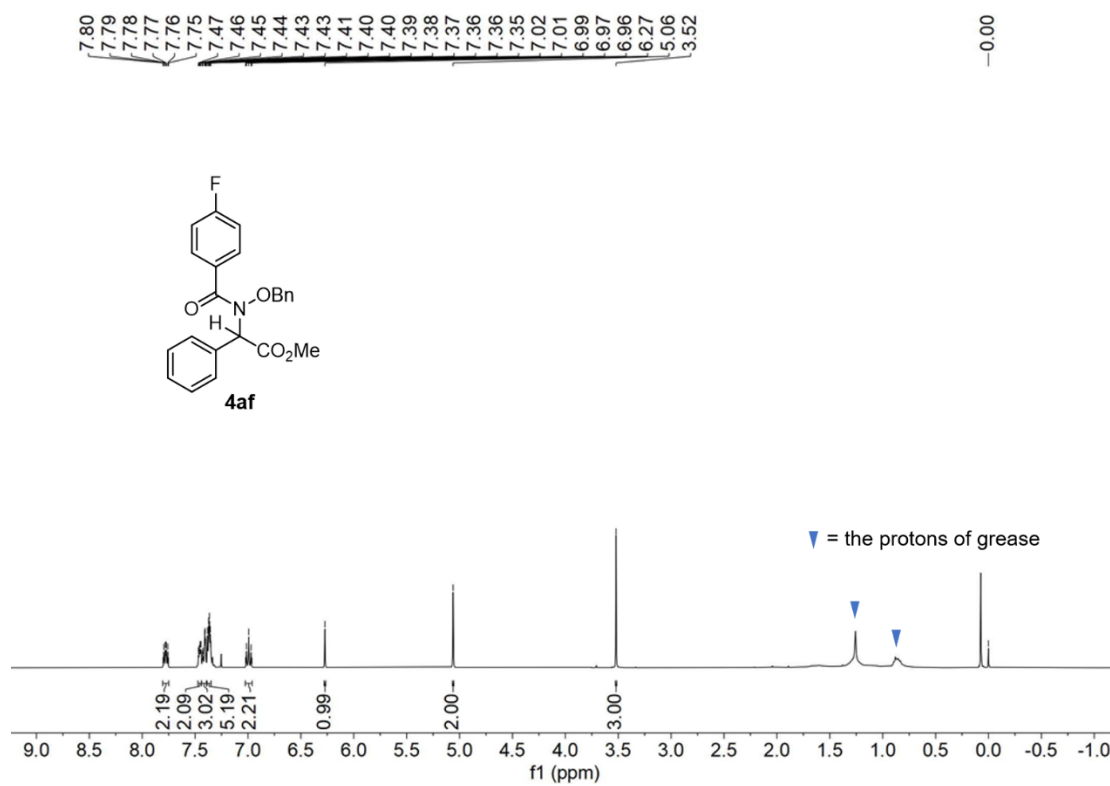
**Fig. S103** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ae**.



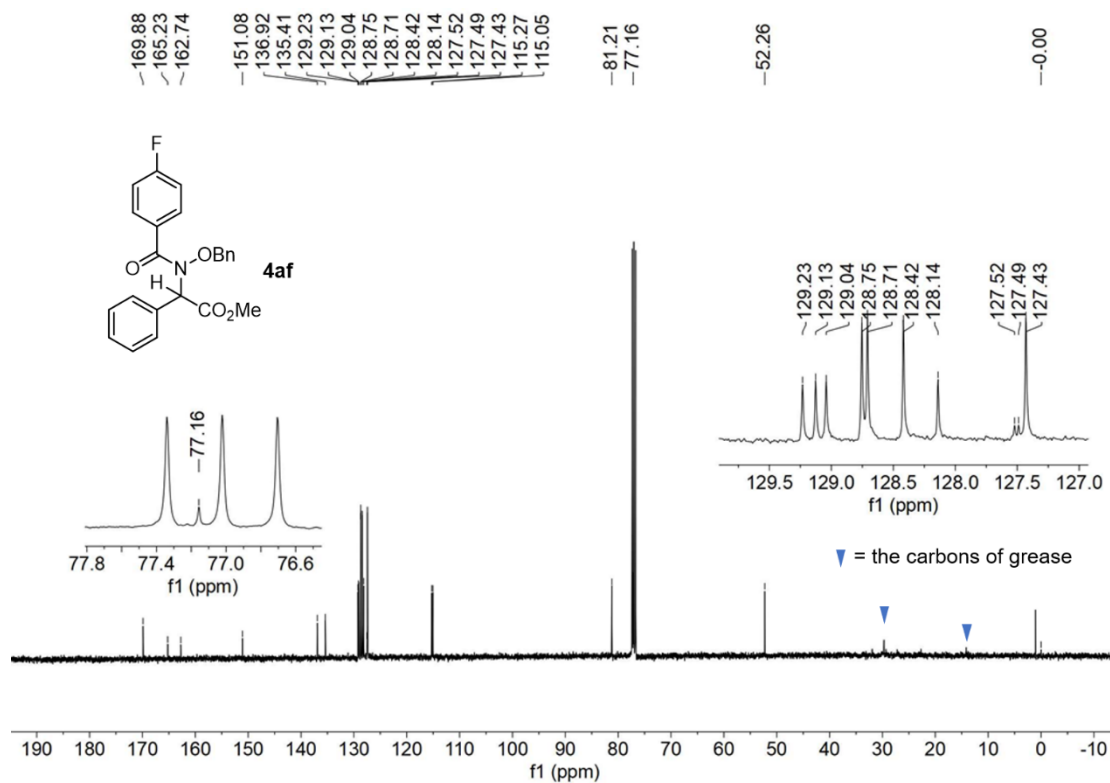
**Fig. S104** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ae**.



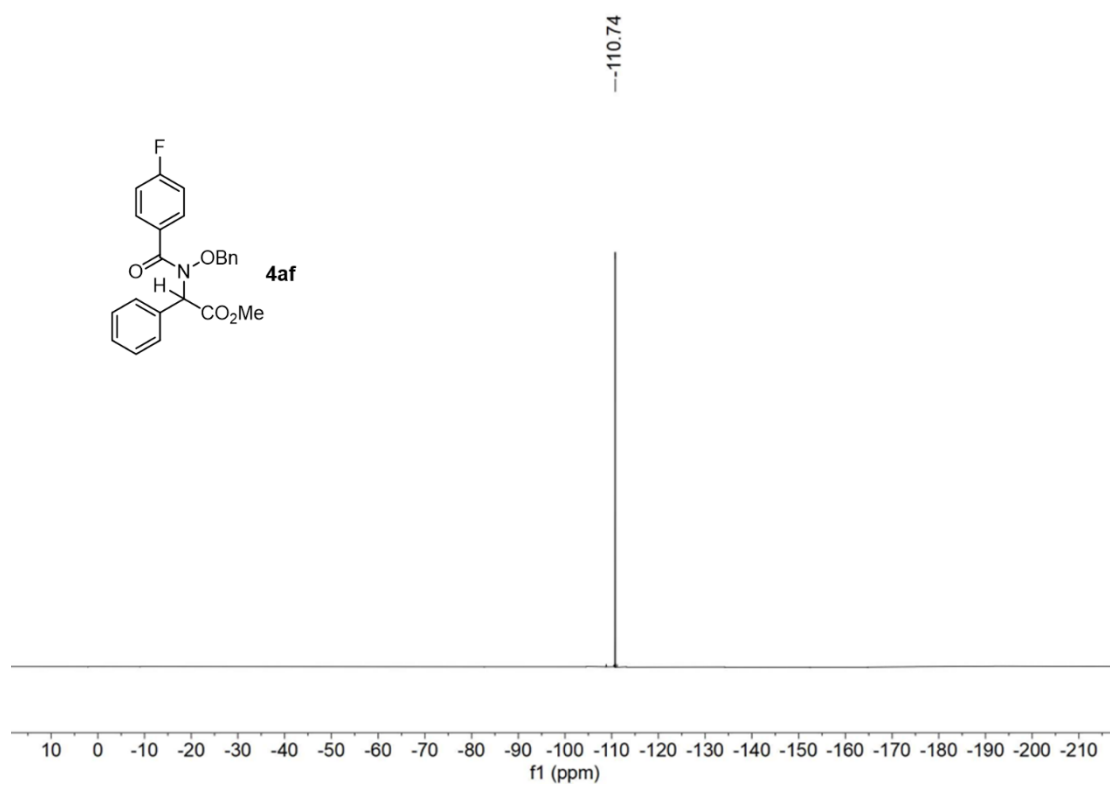
**Fig. S105**  $^{19}\text{F}$  NMR spectrum (376 MHz,  $\text{CDCl}_3$ ) of compound **4ae**.



**Fig. S106**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ) of compound **4af**.

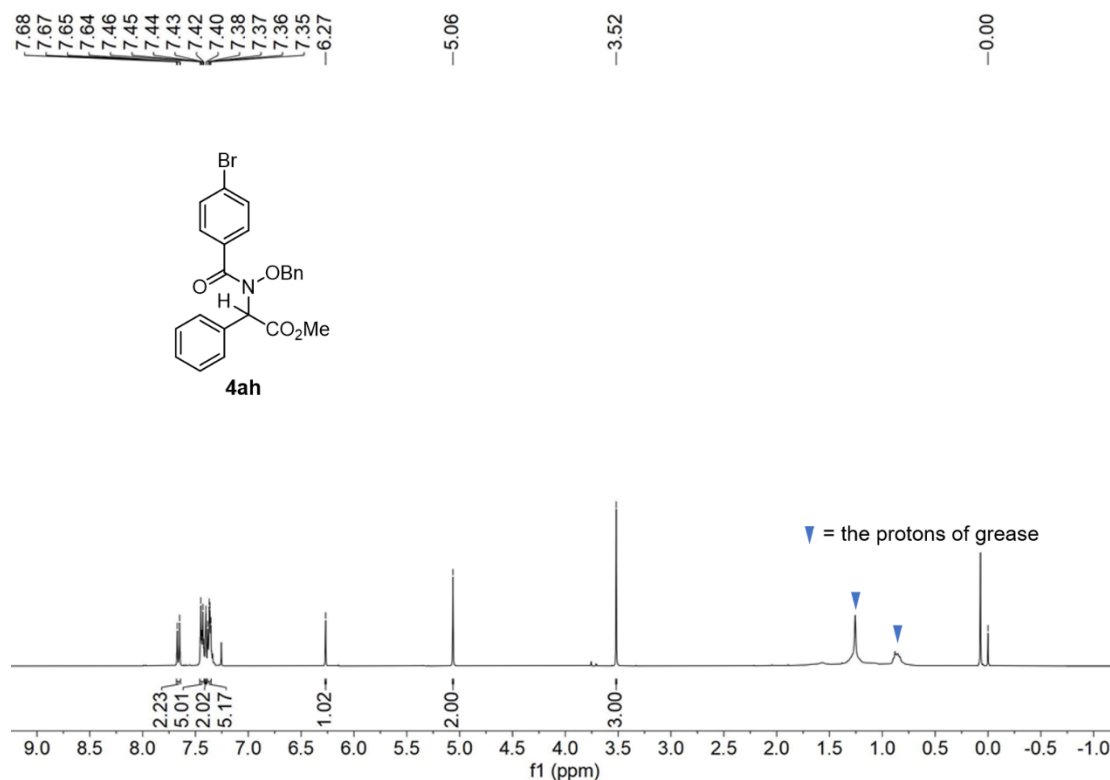


**Fig. S107** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4af**.

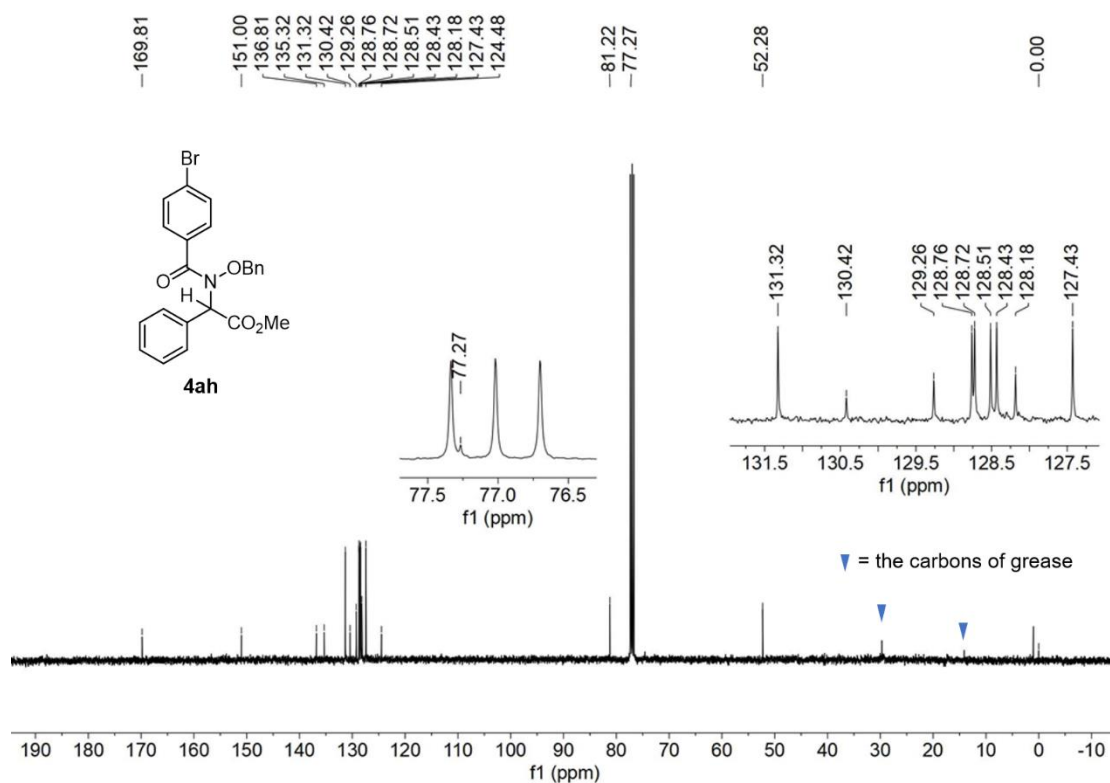


**Fig. S108** <sup>19</sup>F NMR spectrum (376 MHz, CDCl<sub>3</sub>) of compound **4af**.



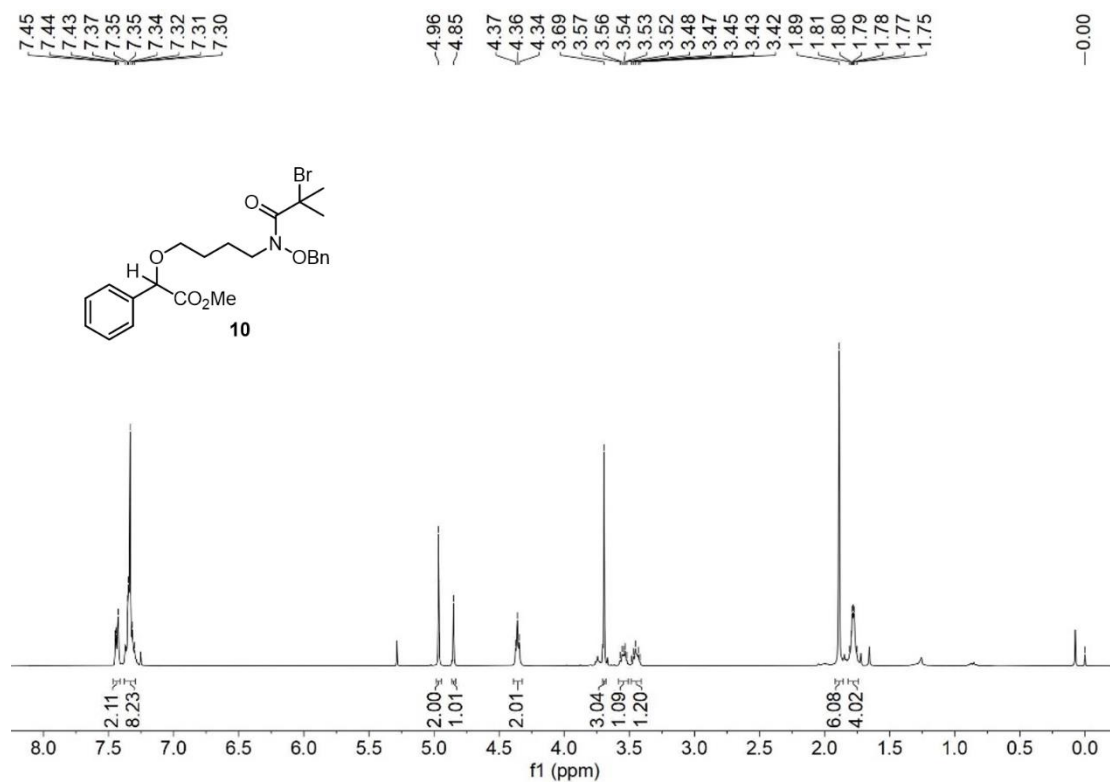


**Fig. S111** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **4ah**.

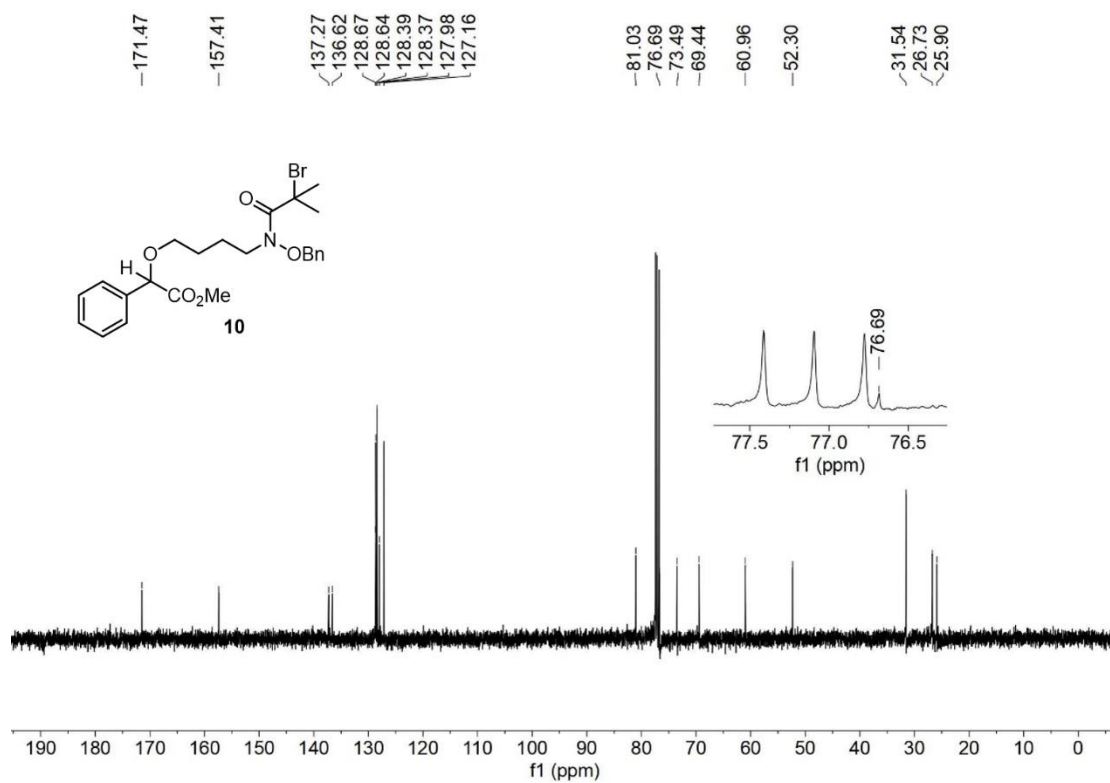


**Fig. S112** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound **4ah**.





**Fig. S113** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound 10.



**Fig. S114** <sup>13</sup>C NMR spectrum (100 MHz, CDCl<sub>3</sub>) of compound 10.