

## **Electronic Supplementary Information**

### **Visible light-induced carbene reactivity of acceptor diazoalkanes: deconstructive difunctionalizations of cyclic ethers with nucleophiles**

*Keyong Zhu, Xinlong Zhou, Yikun Ren, Linhui Dong, Guanzhen Zhao, Jingjing Zhao\* and Pan Li\**

*College of Chemistry and Chemical Engineering, Henan University, Kaifeng 475004, China*

Email: zhaojingjing@henu.edu.cn

Email: panli@henu.edu.cn

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## General Information:

All reagents purchased from commercial sources were used as received. The silica gel for column chromatography was supplied as 300–400 meshes. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker AVANCE III spectrometer and are referenced to the residual solvent signals (7.26 ppm for  $^1\text{H}$  and 77.0 ppm for  $^{13}\text{C}$  in  $\text{CDCl}_3$ ; 2.50 ppm for  $^1\text{H}$  and 39.5 ppm for  $^{13}\text{C}$  in  $d_6$ -DMSO). The HRMS spectra were recorded on a Bruker MicroTOF Q II spectrometer. All the carboxylic acids, nucleophiles and cyclic ethers were purchased from commercial sources. All diazoalkanes were synthesized according to our previous work.<sup>[1-2]</sup>

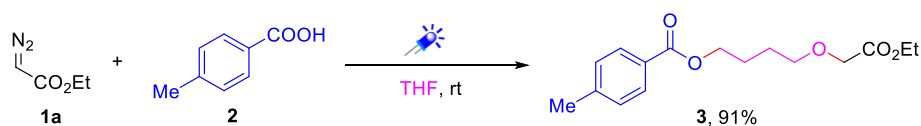
## Reaction Equipment and Light Source

We use RLH-18 8-position Photo Reaction System, which manufactured by Beijing Rogertech Co.ltd base in Beijing PRC. This Photo reactor we used have equipped 8 blue light 10W LED. This blue light 10 W LED's energy peak wavelength is 440 nm, peak width at half-height is 25 nm. Irradiation vessel is borosilicate glass test tube, LED irradiate through a high-reflection channel to the test tube, path length is 2 cm. No filter between LED and test tube.

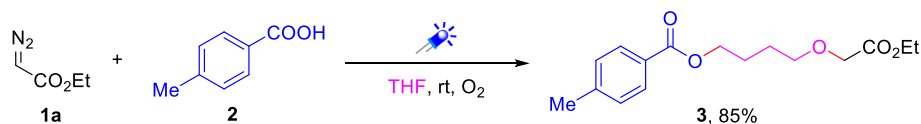


**Figure S1.** The Reaction Equipment and Light Source ( $\lambda_{\text{max}} = 440 \text{ nm}$ ,  $\Delta\lambda = 25 \text{ nm}$ )

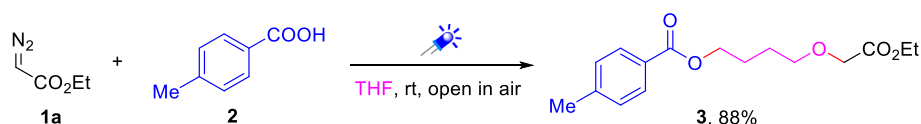
### Reaction of Ethyl Diazoacetate and 4-Methylbenzoic Acid in THF



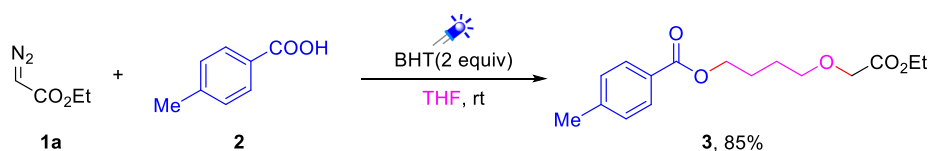
To a 10 mL Schlenk flask was added 4-methylbenzoic acid **2** (0.4 mmol, 54 mg), THF (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **3** (107 mg, 91% yield).



To a 10 mL Schlenk flask was added 4-methylbenzoic acid **2** (0.4 mmol, 54 mg), THF (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was vacuumed and purged with oxygen three times before it was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **3** (100 mg, 85% yield).

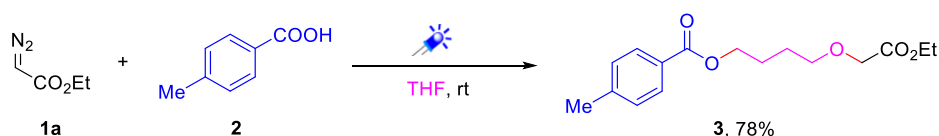


To a 10 mL Schlenk flask was added 4-methylbenzoic acid **2** (0.4 mmol, 54 mg), THF (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was open in air. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **3** (104 mg, 88% yield).



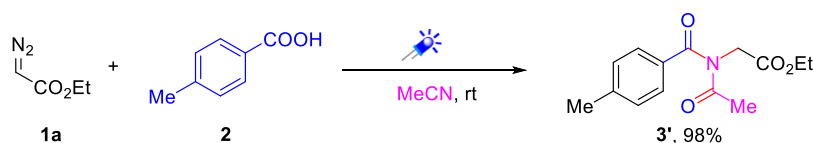
To a 10 mL Schlenk flask was added 4-methylbenzoic acid **2** (0.4 mmol, 54 mg), BHT (0.8 mmol, 176 mg) and THF (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **3** (100 mg, 85% yield).

### Gram Scale Experiment



To a 100 mL Schlenk flask was added 4-methylbenzoic acid **2** (5 mmol, 0.6 g), THF (20 mL), followed by ethyl diazoacetate **1a** (10 mmol, 1.14 g). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 48 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **3** (1.15 g, 78% yield). *It's worth noting that no reaction occurs in dark!*

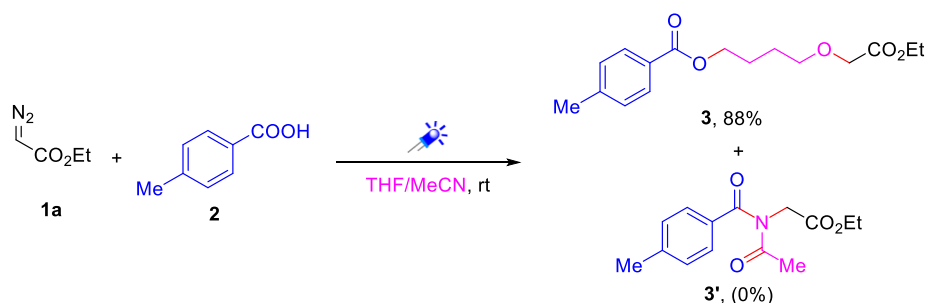
### Reaction of Ethyl Diazoacetate and 4-Methylbenzoic Acid in MeCN



To a 10 mL Schlenk flask was added 4-methylbenzoic acid **2** (0.4 mmol, 54 mg), MeCN (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **3'** (103 mg, 98% yield). *It's worth noting that no reaction occurs in dark!*

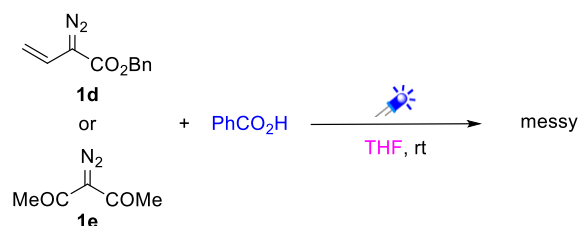


### Reaction of Ethyl Diazoacetate and 4-Methylbenzoic Acid in THF/MeCN.

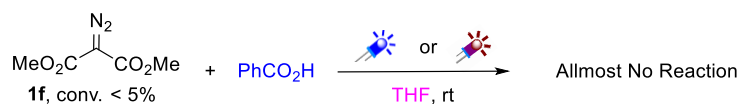


To a 10 mL Schlenk flask was added 4-methylbenzoic acid **2** (0.4 mmol, 54 mg), THF/MeCN = 1 : 1 (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **3** (104 mg, 88% yield).

### Control Experiments

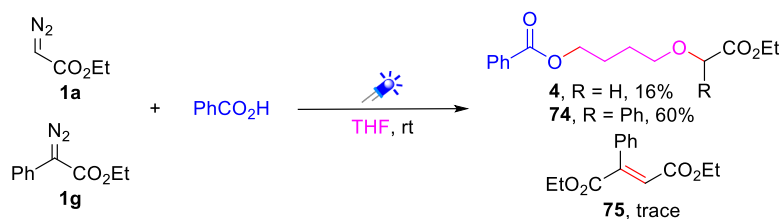


To a 10 mL Schlenk flask was added benzoic acid (0.4 mmol, 49 mg), THF (2 mL), followed by vinyl diazoacetate **1d** (0.8 mmol, 162 mg) or 3-diazopentane-2,4-dione **1e** (0.8 mmol, 101 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h, giving the messy reactions.



To a 10 mL Schlenk flask was added benzoic acid (0.4 mmol, 49 mg), THF (2 mL), followed by dimethyl 2-diazomalonate **1f** (0.8 mmol, 126 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs or purple LEDs ( $\lambda_{\text{max}} = 400 \text{ nm}$ ) for 12 h, giving the no reaction.

## Competitive Experiment



To a 10 mL Schlenk flask was added benzoic acid (0.4 mmol, 49 mg), THF (2 mL), followed by ethyl diazoacetate **1a** (0.4 mmol, 46 mg) and aryl diazoacetate **1g** (0.4 mmol, 76 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The desired products **4** and **74** were obtained in 16% and 60% yields. In addition, these products **4**, **74** and **75** were detected by GC–MS (Figure S2–S5).

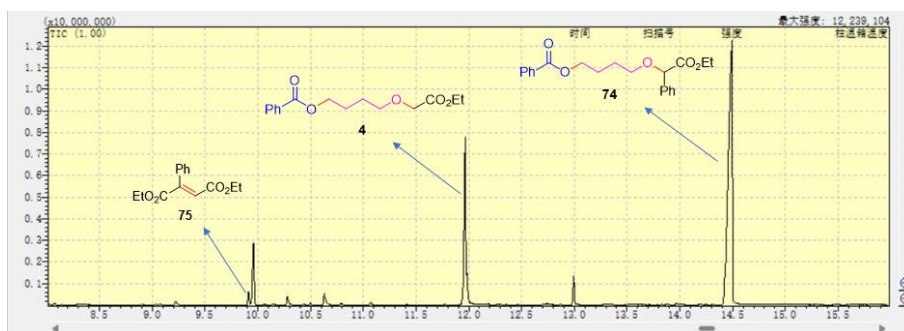


Figure S2 GC–MS of Competitive Experiment

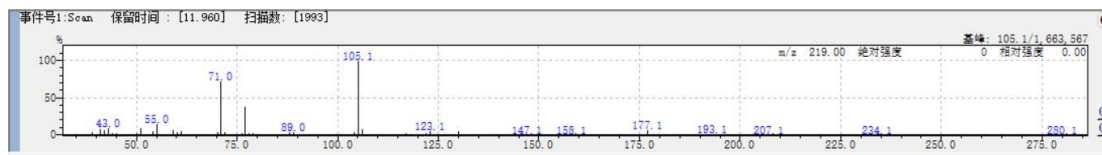


Figure S3 GC–MS of Compound **4** ( $m/z$ : 280, 234, 207, 193.)

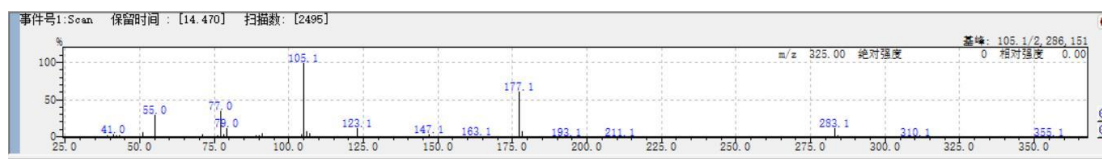


Figure S4 GC–MS of Compound **74** ( $m/z$ : 355, 310, 283.)

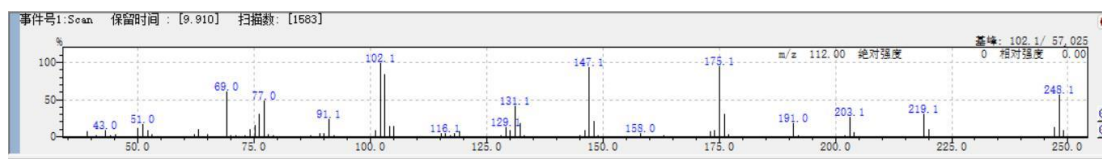
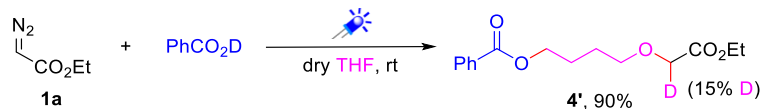


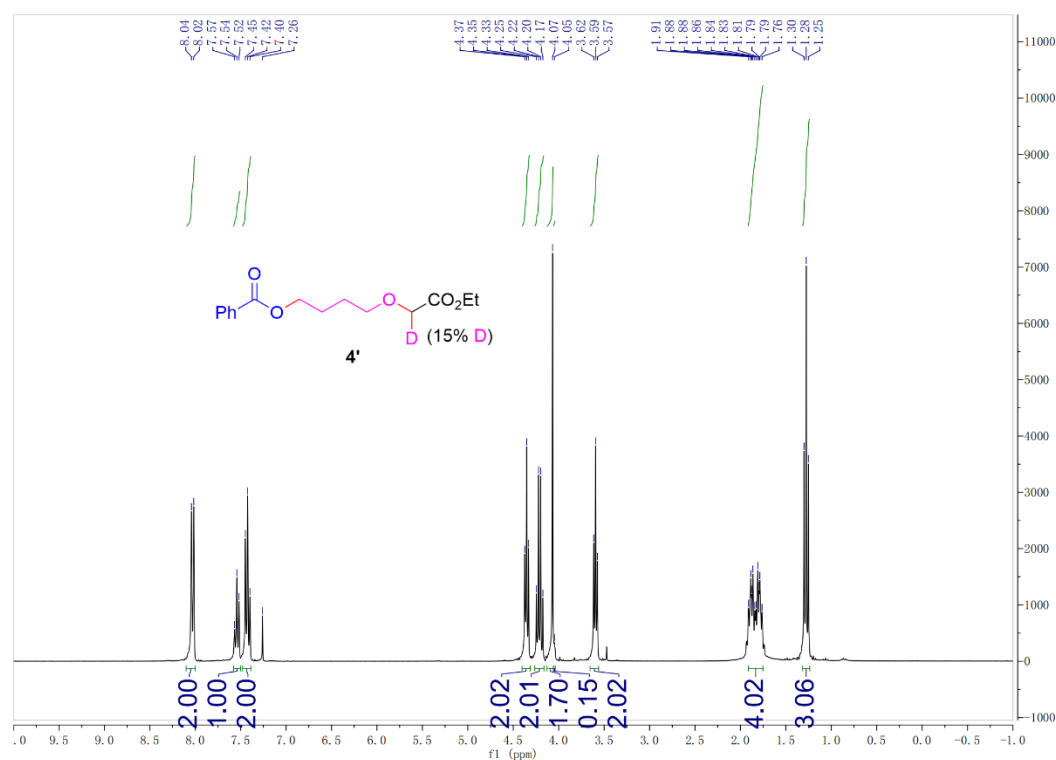
Figure S5 GC–MS of Compound **75** ( $m/z$ : 248, 219, 203, 175.)

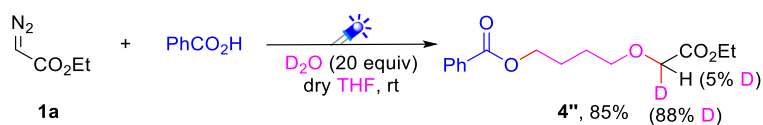
## Isotope-Labeling Experiments



To a 10 mL Schlenk flask was added *d*-benzoic acid (0.4 mmol, 50 mg), dry THF (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **4'** (101 mg, 90% yield).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03 (d,  $J = 7.4$  Hz, 2 H), 7.54 (t,  $J = 7.4$  Hz, 1 H), 7.42 (t,  $J = 7.5$  Hz, 2 H), 4.35 (t,  $J = 6.3$  Hz, 2 H), 4.21 (q,  $J = 7.1$  Hz, 2 H), 4.07 (s, 1.70 H), 4.05 (t,  $J = 2.5$  Hz, 0.15 H), 3.59 (t,  $J = 6.2$  Hz, 2 H), 1.92 – 1.75 (m, 4 H), 1.28 (t,  $J = 7.1$  Hz, 3 H).

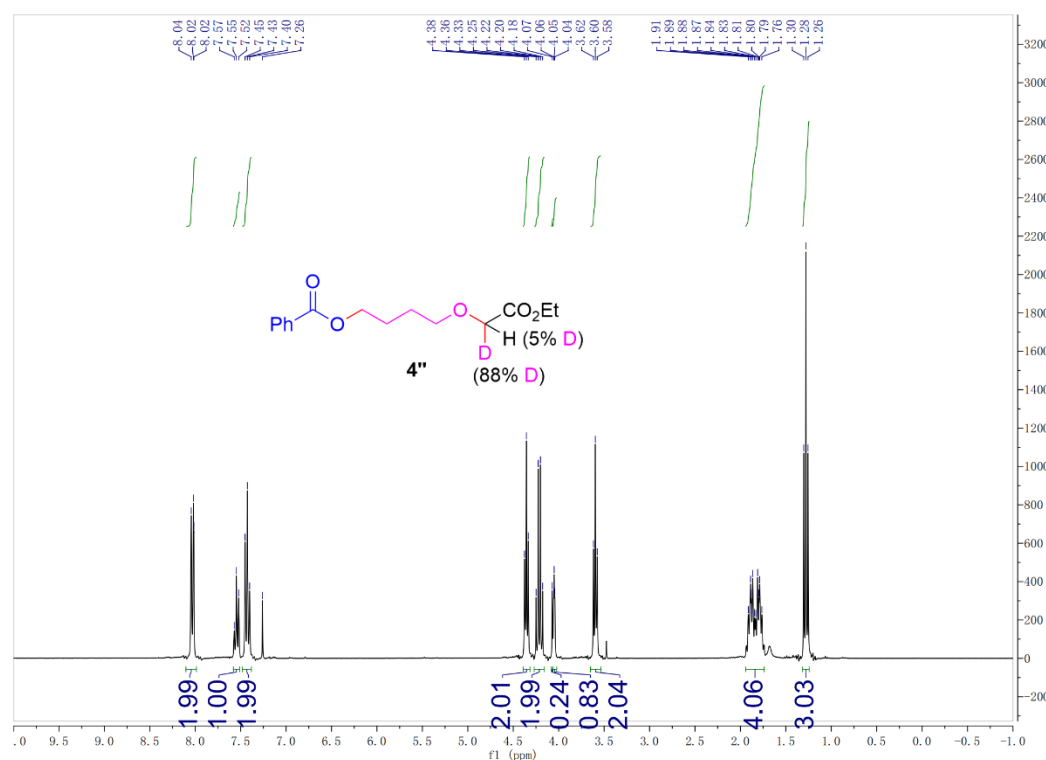
### $^1\text{H}$ NMR (300 MHz, $\text{CDCl}_3$ ) Spectrum of **4'**



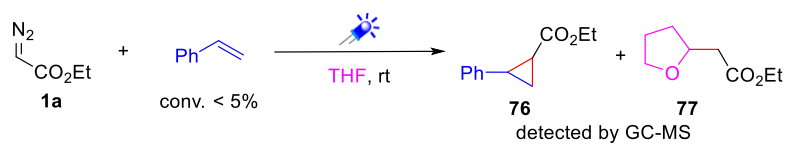


To a 10 mL Schlenk flask was added benzoic acid (0.4 mmol, 49 mg), dry THF (2 mL) and  $\text{D}_2\text{O}$  (8 mmol, 160 mg), followed by ethyl diazoacetate **1a** (0.8 mmol, 91 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product **4''** (96 mg, 85% yield).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.07 – 8.00 (m, 2 H), 7.55 (t,  $J = 7.4$  Hz, 1 H), 7.43 (t,  $J = 7.6$  Hz, 2 H), 4.36 (t,  $J = 6.3$  Hz, 2 H), 4.21 (q,  $J = 7.1$  Hz, 2 H), 4.07 (s, 0.26 H), 4.05 (t,  $J = 2.5$  Hz, 0.78 H), 3.60 (t,  $J = 6.2$  Hz, 2 H), 1.92 – 1.75 (m, 4 H), 1.28 (t,  $J = 7.1$  Hz, 3 H).

**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of **4''****



## Styrene instead of carboxylic acid



To a 10 mL Schlenk flask was added styrene (0.4 mmol, 42 mg), THF (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 93 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The products **76** and **77** were detected by GC–MS (Figure S6–S8).

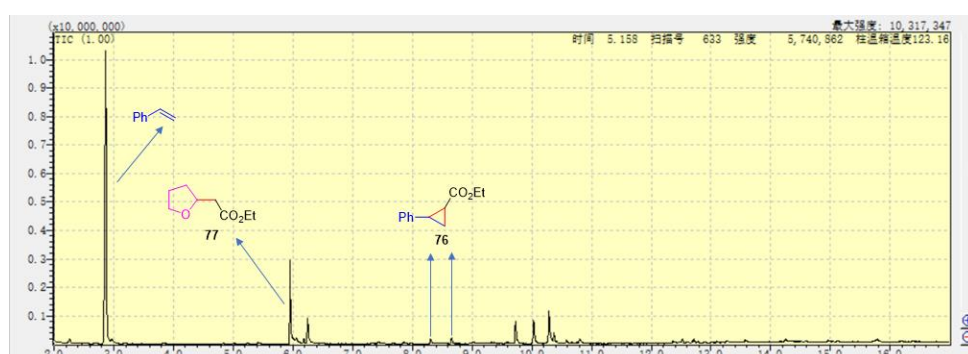


Figure S6 GC–MS of Styrene instead of Carboxylic Acid

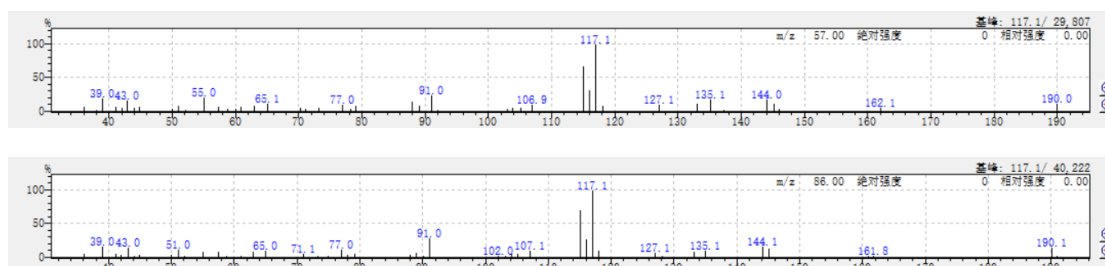


Figure S7 GC–MS of Compound **76** (two isomers: m/z: 190, 162, 144, 135.)

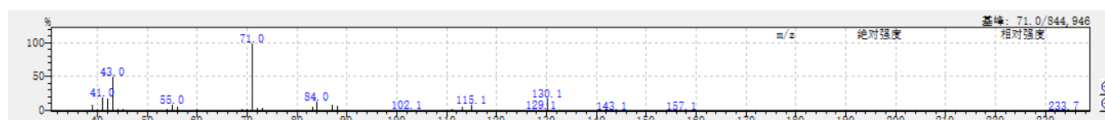
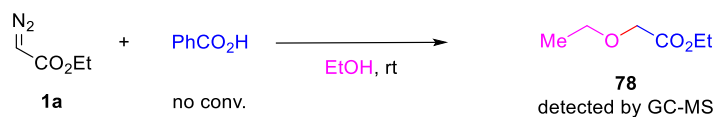


Figure S8 GC–MS of Compound **77** (m/z: 157, 143, 130, 129)

## EtOH instead of THF



To a 10 mL Schlenk flask was added benzoic acid (0.4 mmol, 49 mg), EtOH (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 93 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The products **78** was detected by GC–MS (Figure S9–S10).

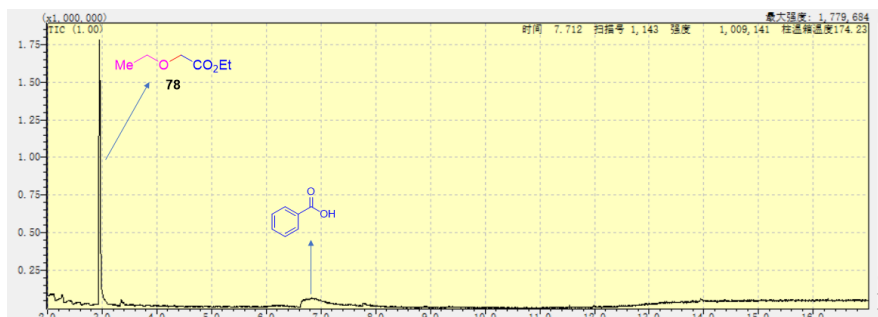


Figure S9 GC–MS of EtOH instead of THF

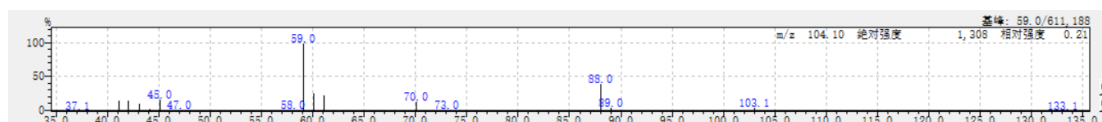
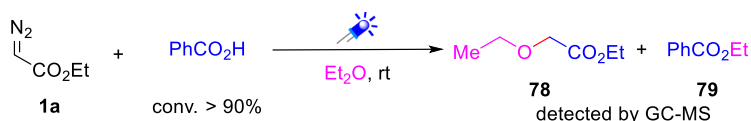


Figure S10 GC–MS of Compound **78** (m/z: 133, 103, 89, 88)

### Et<sub>2</sub>O instead of THF



To a 10 mL Schlenk flask was added benzoic acid (0.4 mmol, 49 mg), Et<sub>2</sub>O (2 mL), followed by ethyl diazoacetate **1a** (0.8 mmol, 93 mg). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The products **78** and **79** were detected by GC–MS (Figure S11–S12).

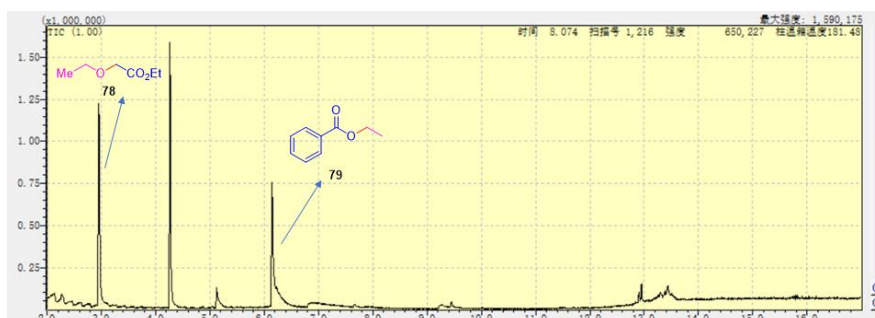
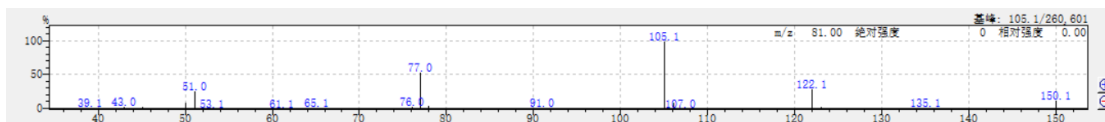


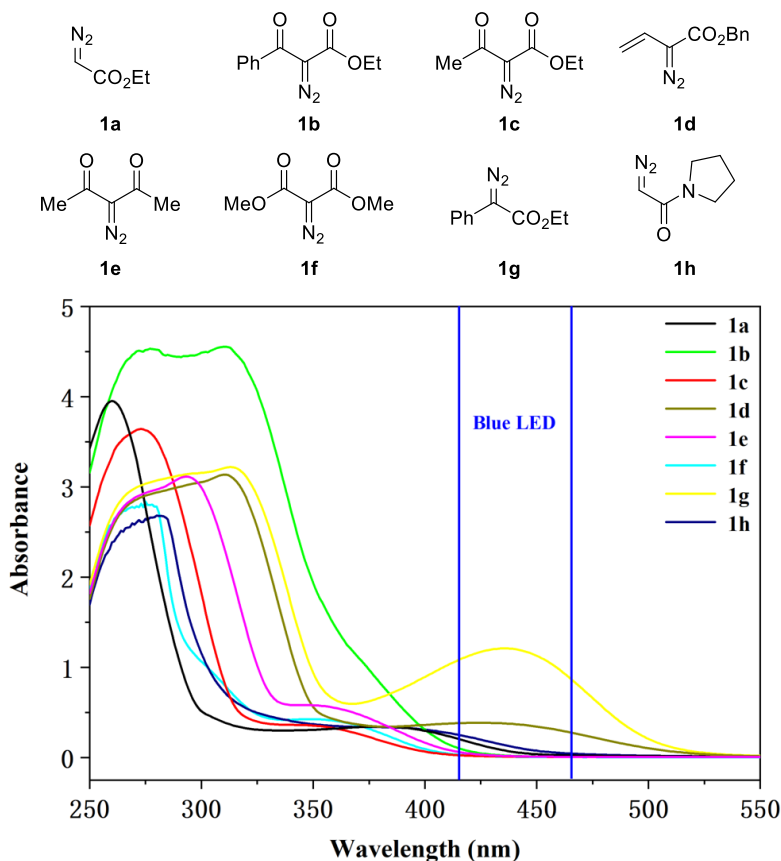
Figure S11 GC–MS of Et<sub>2</sub>O instead of THF



**Figure S12** GC–MS of Compound **79** ( $m/z$ : 150, 135, 122, 107)

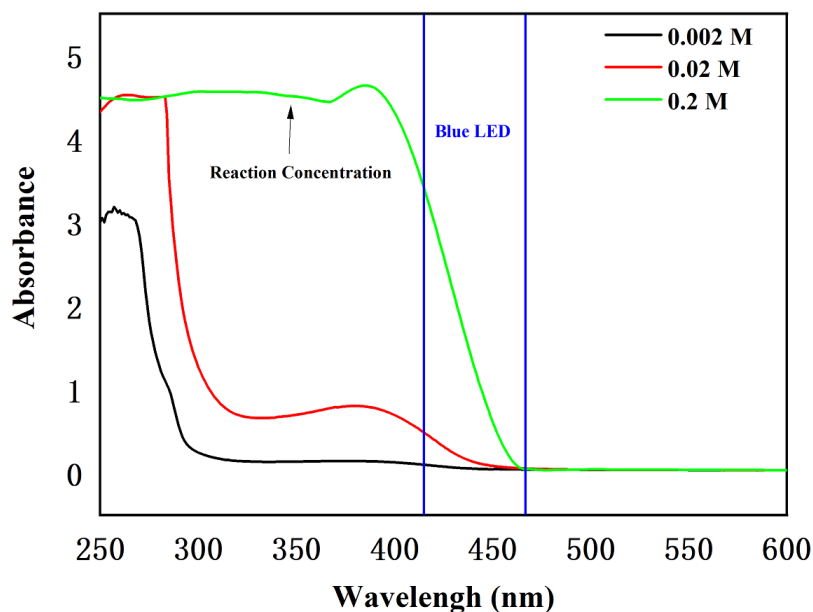
## UV-vis Absorbance Spectra

Emission intensities were recorded using a UV-6000PC visible spectrophotometer. This blue light 10 W LED's energy peak wavelength is 440 nm, peak width at half-height is 25 nm. At the outset of this investigation, the UV-vis absorbance spectra of different types of substrates were analyzed, including diazo compounds **1a–1h**. In a typical experiment, the spectrum of a 0.01 mol mL<sup>-1</sup> solution in THF was collected.



**Figure. S13** UV-vis Absorbance Spectra of Diazoalkanes in THF.

Emission intensities were recorded using a UV-6000PC visible spectrophotometer. This blue light 10 W LED's energy peak wavelength is 440 nm, peak width at half-height is 25 nm. In a typical experiment, the UV-vis absorbance spectra of EDA in different concentrations were collected. In a typical experiment, the spectrum of different concentration solution in THF was collected.

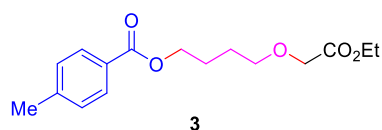


**Figure. S14** UV-vis Absorbance Spectra of Different Concentration **1a** in THF.

**General Procedure for Synthesis of Difunctionalized Ethers.**



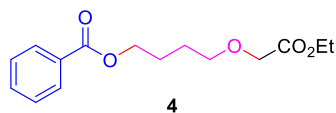
To a 10 mL Schlenk flask was added nucleophile (0.4 mmol), cyclic ether (2 mL), followed by diazoalkane (0.8 mmol). Then Schlenk tube was tightly screw capped. The mixture was stirred under the blue LEDs for 12 h. The solvent was evaporated in vacuo, and the residue was purified by flash column chromatography, eluting with petroleum ether and ethyl acetate (10:1) to afford the desired product.



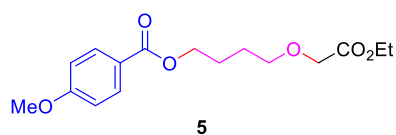
**4-(2-Ethoxy-2-oxoethoxy)butyl 4-methylbenzoate (3, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (107 mg, 91%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (d,  $J$  = 8.2 Hz, 2 H), 7.22 (d,  $J$  = 8.0 Hz, 2 H), 4.33 (t,  $J$  = 6.2 Hz, 2 H), 4.21 (q,  $J$  = 7.1 Hz, 2 H), 4.07 (s, 2 H), 3.59 (t,  $J$  = 6.1 Hz, 2 H), 2.40 (s, 3 H), 1.98 – 1.67 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 166.6, 143.4, 129.5, 129.0, 128.8, 128.7, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4, 127.3, 127.2, 127.1, 127.0, 126.9, 126.8, 126.7, 126.6, 126.5, 126.4, 126.3, 126.2, 126.1, 126.0, 125.9, 125.8, 125.7, 125.6, 125.5, 125.4, 125.3, 125.2, 125.1, 125.0, 124.9, 124.8, 124.7, 124.6, 124.5, 124.4, 124.3, 124.2, 124.1, 124.0, 123.9, 123.8, 123.7, 123.6, 123.5, 123.4, 123.3, 123.2, 123.1, 123.0, 122.9, 122.8, 122.7, 122.6, 122.5, 122.4, 122.3, 122.2, 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9.4, 9.3, 9.2, 9.1, 9.0, 8.9, 8.8, 8.7, 8.6, 8.5, 8.4, 8.3, 8.2, 8.1, 8.0, 7.9, 7.8, 7.7, 7.6, 7.5, 7.4, 7.3, 7.2, 7.1, 7.0, 6.9, 6.8, 6.7, 6.6, 6.5, 6.4, 6.3, 6.2, 6.1, 6.0, 5.9, 5.8, 5.7, 5.6, 5.5, 5.4, 5.3, 5.2, 5.1, 5.0, 4.9, 4.8, 4.7, 4.6, 4.5, 4.4, 4.3, 4.2, 4.1, 4.0, 3.9, 3.8, 3.7, 3.6, 3.5, 3.4, 3.3, 3.2, 3.1, 3.0, 2.9, 2.8, 2.7, 2.6, 2.5, 2.4, 2.3, 2.2, 2.1, 2.0, 1.9, 1.8, 1.7, 1.6, 1.5, 1.4, 1.3, 1.2, 1.1, 1.0, 0.9, 0.8, 0.7, 0.6, 0.5, 0.4, 0.3, 0.2, 0.1, 0.0.



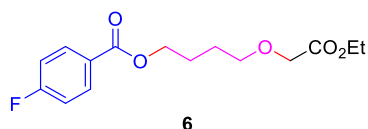
127.6, 71.2, 68.3, 64.4, 60.8, 26.2, 25.4, 21.6, 14.2. HRMS (ESI)  $m/z$ :  $[M + H]^+$  Calcd for  $C_{16}H_{23}O_5$  295.1540, found 295.1546.



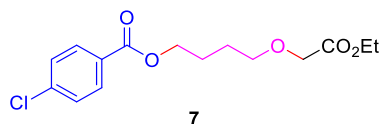
**4-(2-Ethoxy-2-oxoethoxy)butyl benzoate (4, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (103 mg, 92%);  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.02 (d,  $J$  = 7.3 Hz, 2 H), 7.53 (t,  $J$  = 7.4 Hz, 1 H), 7.41 (t,  $J$  = 7.6 Hz, 2 H), 4.34 (t,  $J$  = 6.4 Hz, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.06 (s, 2 H), 3.58 (t,  $J$  = 6.3 Hz, 2 H), 1.93 – 1.83 (m, 2 H), 1.81 – 1.74 (m, 2 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}C\{^1H\}$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  170.4, 166.5, 132.8, 130.3, 129.5, 128.2, 71.1, 68.3, 64.6, 60.7, 26.1, 25.4, 14.1. HRMS (ESI)  $m/z$ :  $[M + H]^+$  Calcd for  $C_{15}H_{21}O_5$  281.1384, found 281.1383.



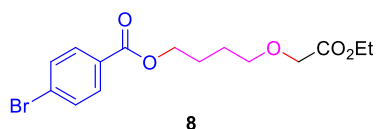
**4-(2-Ethoxy-2-oxoethoxy)butyl 4-methoxybenzoate (5, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (101 mg, 82%);  $^1H$  NMR (300 MHz,  $CDCl_3$ )  $\delta$  8.02 – 7.90 (m, 2 H), 6.96 – 6.84 (m, 2 H), 4.30 (t,  $J$  = 5.9 Hz, 2 H), 4.23 – 4.16 (m, 2 H), 4.06 (s, 2 H), 3.83 (d,  $J$  = 1.6 Hz, 3 H), 3.58 (t,  $J$  = 6.1 Hz, 2 H), 1.92 – 1.70 (m, 4 H), 1.29 – 1.24 (m, 3 H).  $^{13}C\{^1H\}$  NMR (75 MHz,  $CDCl_3$ )  $\delta$  170.4, 166.2, 163.2, 131.4, 122.6, 113.4, 71.1, 68.2, 64.2, 60.7, 55.3, 26.1, 25.4, 14.1. HRMS (ESI)  $m/z$ :  $[M + H]^+$  Calcd for  $C_{16}H_{23}O_6$  311.1489, found 311.1480.



**4-(2-Ethoxy-2-oxoethoxy)butyl 4-fluorobenzoate (6, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (117 mg, 98%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.08 – 8.00 (m, 2 H), 7.12 – 7.06 (m, 2 H), 4.38 – 4.29 (m, 2 H), 4.23 – 4.18 (m, 2 H), 4.06 (d,  $J$  = 1.1 Hz, 2 H), 3.59 (t,  $J$  = 6.2 Hz, 1 H), 1.91 – 1.84 (m, 2 H), 1.82 – 1.74 (m, 2 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H).  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -105.94.  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.24, 167.14, 165.46 (d,  $J$  = 253.5 Hz), 131.86 (d,  $J$  = 9.3 Hz), 126.41 (d,  $J$  = 3.0 Hz), 115.23 (d,  $J$  = 22.0 Hz), 70.91, 68.10, 64.59, 60.58, 26.01, 25.24, 13.99. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{20}\text{FO}_5$  299.1289, found 299.1283.

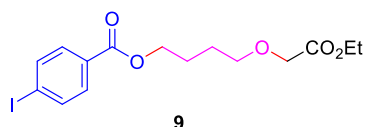


**4-(2-Ethoxy-2-oxoethoxy)butyl 4-chlorobenzoate (7, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (109 mg, 87%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96 (d,  $J$  = 8.5 Hz, 2 H), 7.40 (d,  $J$  = 8.5 Hz, 2 H), 4.35 (t,  $J$  = 6.3 Hz, 2 H), 4.21 (q,  $J$  = 7.1 Hz, 2 H), 4.07 (s, 2 H), 3.59 (t,  $J$  = 6.2 Hz, 2 H), 1.94 – 1.71 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 165.6, 139.1, 130.8, 128.7, 128.5, 71.0, 68.2, 64.8, 60.0, 26.1, 25.3, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{15}\text{H}_{19}\text{ClNaO}_5$  337.0813, found 337.0809.

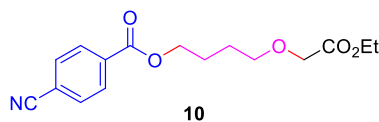


**4-(2-Ethoxy-2-oxoethoxy)butyl 4-bromobenzoate (8, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (141 mg, 98%);  $^1\text{H}$  NMR

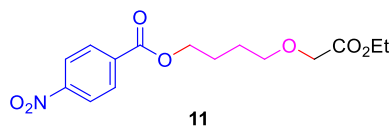
(300 MHz, CDCl<sub>3</sub>)  $\delta$  7.90 – 7.87 (m, 2 H), 7.61 – 7.52 (m, 2 H), 4.34 (q,  $J$  = 5.9 Hz, 2 H), 4.21 (q,  $J$  = 7.1 Hz, 2 H), 4.07 (s, 2 H), 3.59 (t,  $J$  = 6.2 Hz, 2 H), 1.95 – 1.71 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 165.7, 131.5, 131.0, 129.1, 127.8, 71.0, 68.2, 64.8, 60.7, 26.1, 25.3, 14.1. HRMS (ESI)  $m/z$ : [M + Na]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>19</sub>BrNaO<sub>5</sub> 381.0308, found 381.0318.



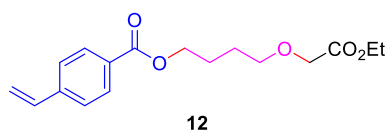
**4-(2-Ethoxy-2-oxoethoxy)butyl 4-iodobenzoate (9, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (140 mg, 86%); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 – 7.70 (m, 4 H), 4.34 (t,  $J$  = 6.3 Hz, 2 H), 4.25 – 4.16 (m, 2 H), 4.07 (s, 2 H), 3.59 (t,  $J$  = 6.1 Hz, 2 H), 1.95 – 1.71 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.4, 166.0, 137.6, 130.9, 129.7, 100.6, 71.1, 68.3, 64.9, 60.8, 26.1, 25.3, 14.2. HRMS (ESI)  $m/z$ : [M + Na]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>19</sub>INaO<sub>5</sub> 429.0169, found 429.0179.



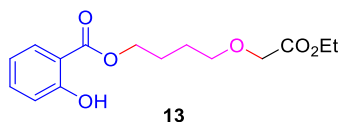
**4-(2-Ethoxy-2-oxoethoxy)butyl 4-cyanobenzoate (10, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (118 mg, 97%); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.12 (d,  $J$  = 8.3 Hz, 2 H), 7.73 (d,  $J$  = 8.3 Hz, 2 H), 4.38 (t,  $J$  = 6.4 Hz, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.06 (s, 2 H), 3.65 – 3.53 (m, 2 H), 1.96 – 1.83 (m, 2 H), 1.81 – 1.72 (m, 2 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 164.8, 134.0, 132.2, 129.9, 117.8, 116.1, 70.9, 68.2, 65.4, 60.7, 26.0, 25.2, 14.1. HRMS (ESI)  $m/z$ : [M + Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>19</sub>NNaO<sub>5</sub> 328.1155, found 328.1150.



**4-(2-Ethoxy-2-oxoethoxy)butyl 4-nitrobenzoate (11, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (72 mg, 55%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.31 – 8.21 (m, 2 H), 8.20 – 8.13 (m, 2 H), 4.40 (t,  $J$  = 6.5 Hz, 2 H), 4.27 – 4.13 (m, 2 H), 4.06 (s, 2 H), 3.59 (t,  $J$  = 6.2 Hz, 2 H), 1.95 – 1.85 (m, 2 H), 1.80 – 1.74 (m, 2 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 164.6, 150.4, 135.7, 130.6, 123.4, 71.0, 68.3, 65.6, 60.8, 26.1, 25.4, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{15}\text{H}_{19}\text{KNO}_7$  364.0793, found 364.0803.

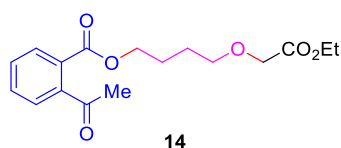


**4-(2-Ethoxy-2-oxoethoxy)butyl 4-vinylbenzoate (12, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (65 mg, 53%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98 (d,  $J$  = 8.3 Hz, 2 H), 7.44 (d,  $J$  = 8.3 Hz, 2 H), 6.74 (dd,  $J$  = 17.6, 10.9 Hz, 1 H), 5.85 (d,  $J$  = 17.6 Hz, 1 H), 5.37 (d,  $J$  = 10.9 Hz, 1 H), 4.34 (t,  $J$  = 6.3 Hz, 2 H), 4.21 (q,  $J$  = 7.1 Hz, 2 H), 4.07 (s, 2 H), 3.59 (t,  $J$  = 6.1 Hz, 2 H), 1.95 – 1.70 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 166.2, 141.7, 135.9, 129.7, 129.3, 125.9, 116.3, 71.0, 68.2, 64.5, 60.7, 26.1, 25.3, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{17}\text{H}_{23}\text{O}_5$  307.1540, found 307.1538.

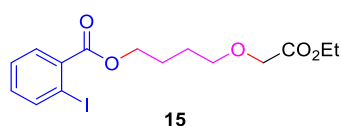


**4-(2-Ethoxy-2-oxoethoxy)butyl 2-hydroxybenzoate (13, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (106 mg, 98%);  $^1\text{H}$  NMR

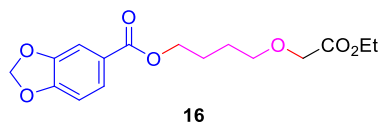
(300 MHz, CDCl<sub>3</sub>)  $\delta$  10.81 (s, 1 H), 7.82 (dd,  $J$  = 8.0, 1.5 Hz, 1 H), 7.50 – 7.38 (m, 1 H), 6.95 (d,  $J$  = 8.4 Hz, 1 H), 6.88 – 6.83 (m, 1 H), 4.38 (t,  $J$  = 6.4 Hz, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.06 (s, 2 H), 3.59 (t,  $J$  = 6.1 Hz, 2 H), 1.97 – 1.71 (m, 4 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 170.0, 161.5, 135.5, 129.7, 118.9, 117.4, 112.4, 70.9, 68.17, 64.9, 60.7, 26.0, 25.2, 14.1. HRMS (ESI)  $m/z$ : [M + H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>21</sub>O<sub>6</sub> 297.1333, found 297.1330.



**4-(2-Ethoxy-2-oxoethoxy)butyl 2-acetylbenzoate (14, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (101 mg, 78%); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 (d,  $J$  = 7.5 Hz, 1H), 7.61 – 7.43 (m, 2 H), 7.39 (d,  $J$  = 7.4 Hz, 1 H), 4.33 (t,  $J$  = 6.4 Hz, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.05 (s, 2 H), 3.57 (t,  $J$  = 6.1 Hz, 2 H), 2.53 (s, 3 H), 1.91 – 1.67 (m, 4 H), 1.27 (t,  $J$  = 7.1 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  202.9, 170.35, 166.8, 142.6, 131.9, 129.9, 129.6, 128.9, 126.3, 70.9, 68.2, 65.3, 60.7, 30.0, 26.0, 25.1, 14.1. HRMS (ESI)  $m/z$ : [M + H]<sup>+</sup> Calcd for C<sub>17</sub>H<sub>23</sub>O<sub>6</sub> 323.1489, found 323.1497.

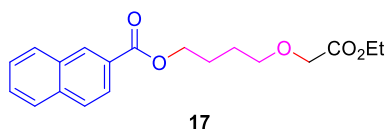


**4-(2-Ethoxy-2-oxoethoxy)butyl 2-iodobenzoate (15, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (146 mg, 90%); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.96 (d,  $J$  = 7.9 Hz, 1 H), 7.77 (dd,  $J$  = 7.8, 1.6 Hz, 1 H), 7.41 – 7.34 (m, 1 H), 7.15 – 7.10 (m, 1 H), 4.36 (t,  $J$  = 6.3 Hz, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.05 (s, 2 H), 3.58 (t,  $J$  = 6.1 Hz, 2 H), 1.97 – 1.69 (m, 4 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 166.4, 141.0, 135.1, 132.4, 130.7, 127.7, 93.8, 70.9, 68.1, 65.2, 60.6, 26.1, 25.1, 14.0. HRMS (ESI)  $m/z$ : [M + Na]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>19</sub>INaO<sub>5</sub> 429.0169, found 429.0170.

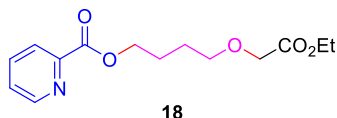


**4-(2-Ethoxy-2-oxoethoxy)butyl benzo[d][1,3]dioxole-5-carboxylate (16, new compound):**

The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (119 mg, 92%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63 (dd,  $J = 8.2, 1.6$  Hz, 1 H), 7.44 (d,  $J = 1.5$  Hz, 1 H), 6.81 (d,  $J = 8.2$  Hz, 1 H), 6.02 (s, 2 H), 4.30 (t,  $J = 6.2$  Hz, 2 H), 4.20 (q,  $J = 7.1$  Hz, 2 H), 4.06 (s, 2 H), 3.58 (t,  $J = 6.1$  Hz, 2 H), 1.90 – 1.70 (m, 4 H), 1.27 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 165.8, 151.4, 147.6, 125.1, 124.2, 109.3, 107.8, 101.7, 71.1, 68.2, 64.5, 60.7, 26.1, 25.3, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{16}\text{H}_{21}\text{O}_7$  325.1282, found 325.1291.

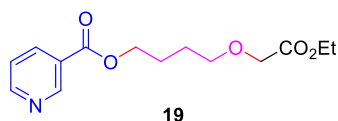


**4-(2-Ethoxy-2-oxoethoxy)butyl 2-naphthoate (17, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (127 mg, 96%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.60 (s, 1 H), 8.06 (dd,  $J = 8.6, 1.4$  Hz, 1 H), 7.95 (d,  $J = 7.7$  Hz, 1 H), 7.87 (d,  $J = 8.5$  Hz, 2 H), 7.65 – 7.45 (m, 2 H), 4.42 (t,  $J = 6.3$  Hz, 2 H), 4.28 – 4.16 (m, 2 H), 4.09 (s, 2 H), 3.63 (t,  $J = 6.2$  Hz, 2 H), 2.02 – 1.75 (m, 4 H), 1.28 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 166.5, 135.3, 132.3, 130.8, 129.1, 128.0, 127.9, 127.6, 127.4, 126.4, 125.0, 71.0, 68.2, 64.6, 60.6, 26.1, 25.3, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{19}\text{H}_{23}\text{O}_5$  331.1540, found 331.1540.

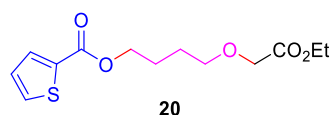


**4-(2-Ethoxy-2-oxoethoxy)butyl picolinate (18, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a slightly yellow oil (103 mg, 92%);  $^1\text{H}$  NMR (300 MHz,

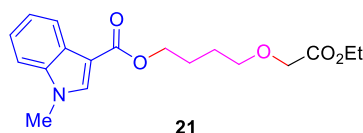
CDCl<sub>3</sub>)  $\delta$  8.75 (d,  $J$  = 4.7 Hz, 1 H), 8.11 (d,  $J$  = 7.8 Hz, 1 H), 7.86 – 7.80 (m, 1 H), 7.51 – 7.41 (m, 1 H), 4.44 (t,  $J$  = 6.6 Hz, 2 H), 4.19 (q,  $J$  = 7.1 Hz, 2 H), 4.05 (s, 2 H), 3.56 (q,  $J$  = 6.5 Hz, 2 H), 1.97 – 1.88 (m, 2 H), 1.81 – 1.72 (m, 2 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.2, 164.9, 149.6, 147.8, 136.8, 126.7, 124.9, 70.9, 68.1, 65.4, 60.6, 25.9, 25.2, 14.0. HRMS (ESI)  $m/z$ : [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>20</sub>NO<sub>5</sub> 282.1336, found 282.1339.



**4-(2-Ethoxy-2-oxoethoxy)butyl nicotinate (19, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a slightly yellow oil (84 mg, 75%); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  9.20 (s, 1 H), 8.76 (d,  $J$  = 4.8 Hz, 1 H), 8.29 (d,  $J$  = 7.9 Hz, 1 H), 7.39 (dd,  $J$  = 7.9, 4.9 Hz, 1 H), 4.38 (t,  $J$  = 6.4 Hz, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.06 (s, 2 H), 3.58 (t,  $J$  = 6.2 Hz, 2 H), 1.94 – 1.85 (m, 2 H), 1.81 – 1.72 (m, 2 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 165.0, 153.1, 150.6, 136.9, 126.1, 123.2, 70.9, 68.1, 65.0, 60.6, 26.0, 25.2, 14.0. HRMS (ESI)  $m/z$ : [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>20</sub>NO<sub>5</sub> 282.1336, found 282.1343.



**4-(2-Ethoxy-2-oxoethoxy)butyl thiophene-2-carboxylate (20, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (111 mg, 97%); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 (dd,  $J$  = 3.7, 1.1 Hz, 1 H), 7.54 (dd,  $J$  = 5.0, 1.1 Hz, 1 H), 7.09 (dd,  $J$  = 4.9, 3.8 Hz, 1 H), 4.33 (t,  $J$  = 6.3 Hz, 2 H), 4.21 (q,  $J$  = 7.1 Hz, 2 H), 4.07 (s, 2 H), 3.59 (t,  $J$  = 6.2 Hz, 2 H), 1.94 – 1.72 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 162.1, 133.8, 133.2, 132.2, 127.6, 71.0, 68.2, 64.7, 60.7, 26.0, 25.3, 14.1. HRMS (ESI)  $m/z$ : [M + Na]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>18</sub>NaO<sub>5</sub>S 309.0767, found 309.0772.



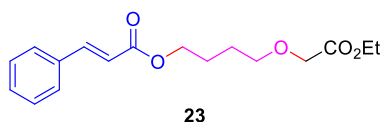
**4-(2-Ethoxy-2-oxoethoxy)butyl 1-methyl-1H-indole-3-carboxylate (21, new compound):**

The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a slightly yellow oil (93 mg, 70%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.20 – 8.10 (m, 1 H), 7.77 (s, 1 H), 7.38 – 7.22 (m, 3 H), 4.36 (q,  $J$  = 6.5 Hz, 2 H), 4.26 – 4.17 (m, 2 H), 4.07 (s, 2 H), 3.81 (s, 3 H), 3.61 (t,  $J$  = 6.3 Hz, 2 H), 1.92 – 1.87 (m, 2 H), 1.86 – 1.79 (m, 2 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 164.9, 137.0, 135.1, 126.4, 122.5, 121.7, 121.4, 109.6, 106.8, 71.1, 68.2, 63.2, 60.6, 33.2, 26.2, 25.5, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{18}\text{H}_{24}\text{NO}_5$  334.1649, found 334.1653.



**4-(2-Ethoxy-2-oxoethoxy)butyl 2-oxo-2-phenylacetate (22, new compound):**

The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (38 mg, 31%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.07 – 7.94 (m, 2 H), 7.66 (t,  $J$  = 7.4 Hz, 1 H), 7.51 (t,  $J$  = 7.7 Hz, 2 H), 4.44 (t,  $J$  = 6.5 Hz, 2 H), 4.21 (q,  $J$  = 7.1 Hz, 2 H), 4.06 (s, 2 H), 3.58 (t,  $J$  = 6.1 Hz, 2 H), 1.96 – 1.87 (m, 2 H), 1.81 – 1.67 (m, 2 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  186.4, 170.4, 163.9, 134.9, 132.4, 129.9, 128.9, 70.9, 68.2, 65.9, 60.8, 25.9, 25.2, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{16}\text{H}_{20}\text{NaO}_6$  331.1152, found 331.1153.

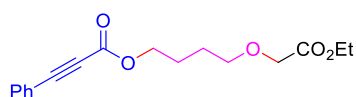


**4-(2-Ethoxy-2-oxoethoxy)butyl cinnamate (23, new compound):**

The reaction was performed following the general procedure. The residue was purified by flash column chromatography

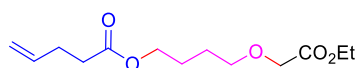


(10:1 PE/EA) to give the product as a colorless oil (76 mg, 62%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67 (d,  $J$  = 16.0 Hz, 1 H), 7.52 – 7.50 (m, 2 H), 7.42 – 7.33 (m, 3 H), 6.42 (d,  $J$  = 16.0 Hz, 1 H), 4.28 – 4.15 (m, 4 H), 4.06 (s, 2 H), 3.58 (t,  $J$  = 6.2 Hz, 2 H), 1.89 – 1.69 (m, 4 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 166.9, 144.6, 134.3, 130.1, 128.8, 128.0, 118.1, 71.1, 68.2, 64.1, 60.7, 26.1, 25.3, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{17}\text{H}_{22}\text{NaO}_5$  329.1359, found 329.1360.



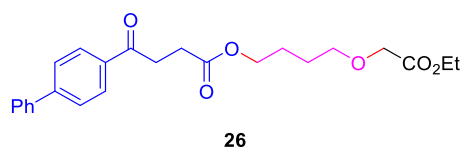
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**4-(2-Ethoxy-2-oxoethoxy)butyl 3-phenylpropiolate (24, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (118 mg, 97%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.55 – 7.53 (m, 2 H), 7.45 – 7.38 (m, 1 H), 7.35 – 7.31 (m, 2 H), 4.26 – 4.23 (m, 2 H), 4.18 (q,  $J$  = 7.1 Hz, 2 H), 4.03 (s, 2 H), 3.54 (t,  $J$  = 6.1 Hz, 2 H), 1.88 – 1.76 (m, 2 H), 1.74 – 1.68 (m, 2 H), 1.27 – 1.23 (m, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 153.9, 132.8, 130.5, 128.4, 119.4, 86.0, 80.5, 70.8, 68.2, 65.6, 60.6, 25.9, 25.1, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{17}\text{H}_{21}\text{O}_5$  305.1384, found 305.1383.

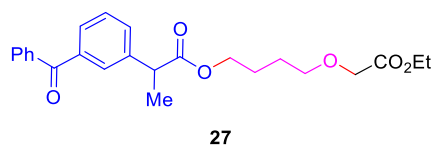


25

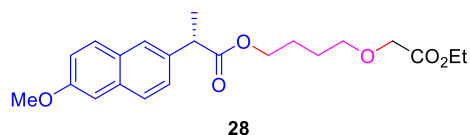
**4-(2-Ethoxy-2-oxoethoxy)butyl pent-4-enoate (25, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (77 mg, 75%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.86 – 5.71 (m, 1 H), 5.04 – 4.95 (m, 2 H), 4.18 (q,  $J$  = 7.1 Hz, 2 H), 4.09 – 4.04 (m, 2 H), 4.02 (s, 2 H), 3.52 (t,  $J$  = 6.1 Hz, 2 H), 2.41 – 2.29 (m, 4 H), 1.76 – 1.61 (m, 4 H), 1.28 – 1.22 (m, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  173.0, 170.4, 136.6, 115.4, 71.1, 68.2, 64.0, 60.7, 33.5, 28.8, 26.0, 25.2, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{13}\text{H}_{23}\text{O}_5$  259.1540, found 259.1532.



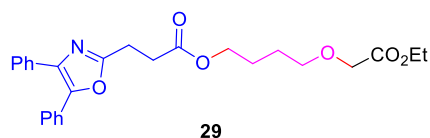
**4-(2-Ethoxy-2-oxoethoxy)butyl 4-([1,1'-biphenyl]-4-yl)-4-oxobutanoate (26, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a white solid (150 mg, 91%); mp 61 – 65 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05 (d,  $J$  = 8.4 Hz, 2 H), 7.69 (d,  $J$  = 8.4 Hz, 2 H), 7.66 – 7.57 (m, 2 H), 7.47 (t,  $J$  = 7.4 Hz, 2 H), 7.43 – 7.36 (m, 1 H), 4.21 (q,  $J$  = 7.1 Hz, 2 H), 4.15 (t,  $J$  = 6.3 Hz, 2 H), 4.05 (s, 2 H), 3.55 (t,  $J$  = 6.1 Hz, 2 H), 3.34 (t,  $J$  = 6.6 Hz, 2 H), 2.78 (t,  $J$  = 6.6 Hz, 2 H), 1.78 – 1.68 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  197.6, 172.9, 170.4, 145.8, 139.8, 135.2, 128.9, 128.6, 128.2, 127.2 (2C overlap), 71.1, 68.3, 64.4, 60.7, 33.4, 28.2, 26.0, 25.2, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{24}\text{H}_{29}\text{O}_6$  413.1959, found 413.1953.



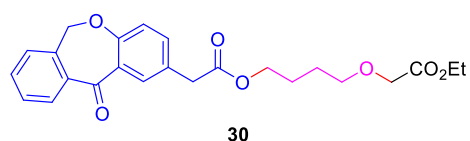
**4-(2-Ethoxy-2-oxoethoxy)butyl 2-(3-benzoylphenyl)propanoate (27, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (101 mg, 61%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (t,  $J$  = 6.9 Hz, 2 H), 7.72 (s, 1 H), 7.64 (dd,  $J$  = 7.6, 1.0 Hz, 1 H), 7.60 – 7.49 (m, 2 H), 7.49 – 7.35 (m, 3 H), 4.16 (q,  $J$  = 7.1 Hz, 2 H), 4.12 – 4.04 (m, 2 H), 3.98 (s, 2 H), 3.76 (q,  $J$  = 7.2 Hz, 1 H), 3.46 (t,  $J$  = 6.2 Hz, 2 H), 1.73 – 1.63 (m, 2 H), 1.62 – 1.53 (m, 2 H), 1.50 (d,  $J$  = 7.2 Hz, 3 H), 1.23 (t,  $J$  = 7.1, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  196.3, 173.9, 170.3, 140.8, 137.7, 137.4, 132.4, 131.4, 129.9, 129.0, 128.8, 128.4, 128.2, 70.9, 68.1, 64.5, 60.6, 45.3, 25.8, 25.2, 18.3, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{24}\text{H}_{29}\text{O}_6$  413.1959, found 413.1963.



**4-(2-Ethoxy-2-oxoethoxy)butyl (S)-2-(6-methoxynaphthalen-2-yl)propanoate (28, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (129 mg, 83%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72 – 7.64 (m, 3 H), 7.40 (dd,  $J$  = 8.5, 1.8 Hz, 1 H), 7.16 – 7.08 (m, 2 H), 4.19 (q,  $J$  = 7.1 Hz, 2 H), 4.15 – 4.07 (m, 2 H), 3.96 (s, 2 H), 3.90 (s, 3 H), 3.84 (q,  $J$  = 7.1 Hz, 1 H), 3.43 (t,  $J$  = 6.3 Hz, 2 H), 1.72 – 1.69 (m, 2 H), 1.62 – 1.53 (m, 5 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  174.6, 170.3, 157.5, 135.7, 133.6, 129.1, 128.8, 127.0, 126.1, 125.8, 118.8, 105.5, 70.9, 68.1, 64.4, 60.7, 55.1, 45.4, 25.8, 25.1, 18.4, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{22}\text{H}_{28}\text{NaO}_6$  411.1778, found 411.1782.

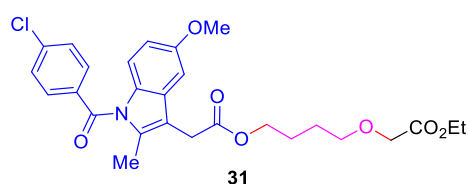


**4-(2-Ethoxy-2-oxoethoxy) butyl 3-(4,5-diphenyloxazol-2-yl) propanoate (29, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (117 mg, 65%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 – 7.59 (m, 2 H), 7.59 – 7.54 (m, 2 H), 7.38 – 7.27 (m, 6 H), 4.25 – 4.11 (m, 4 H), 4.01 (s, 2 H), 3.49 (t,  $J$  = 6.1 Hz, 2 H), 3.17 (t,  $J$  = 7.5 Hz, 2 H), 2.90 (t,  $J$  = 7.5 Hz, 2 H), 1.80 – 1.61 (m, 4 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.8, 170.3, 161.6, 145.2, 134.9, 132.3, 128.8, 128.5, 128.4, 128.3, 127.9, 127.7, 126.3, 70.9, 68.1, 64.3, 60.6, 31.0, 25.9, 25.1, 23.4, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{26}\text{H}_{30}\text{NO}_6$  452.2068, found 452.2067.

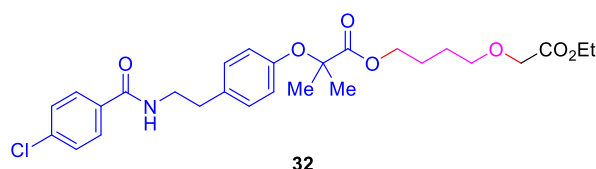


**4-(2-Ethoxy-2-oxoethoxy)butyl 2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-2-yl) acetate (30, new compound):** The reaction was performed following the general procedure. The residue

was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (130 mg, 76%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.09 (d,  $J$  = 2.2 Hz, 1 H), 7.86 (d,  $J$  = 7.5 Hz, 1 H), 7.52 (t,  $J$  = 7.2 Hz, 1 H), 7.48 – 7.36 (m, 2 H), 7.33 (d,  $J$  = 7.4 Hz, 1 H), 7.00 (d,  $J$  = 8.4 Hz, 1 H), 5.15 (s, 2 H), 4.18 (q,  $J$  = 7.1 Hz, 2 H), 4.12 (t,  $J$  = 6.4 Hz, 2 H), 4.02 (s, 2 H), 3.61 (s, 2 H), 3.51 (t,  $J$  = 6.2 Hz, 2 H), 1.80 – 1.58 (m, 4 H), 1.25 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  190.7, 171.3, 170.4, 160.3, 140.3, 136.2, 135.5, 132.7, 132.3, 129.4, 129.1, 127.8, 127.7, 125.0, 120.9, 73.5, 71.0, 68.2, 64.6, 60.7, 40.1, 25.9, 25.2, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{24}\text{H}_{26}\text{NaO}_7$  449.1571, found 449.1575.

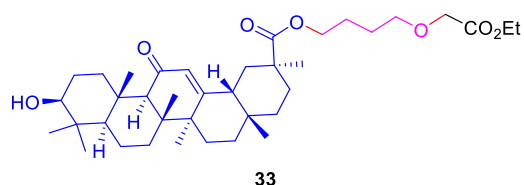


**4-(2-Ethoxy-2-oxoethoxy)butyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (31, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a slightly yellow oil (126 mg, 61%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 (dd,  $J$  = 8.5, 2.0 Hz, 2 H), 7.45 (dd,  $J$  = 8.5, 2.1 Hz, 2 H), 6.95 (s, 1 H), 6.86 (d,  $J$  = 9.0 Hz, 1 H), 6.65 (dd,  $J$  = 9.0, 2.2 Hz, 1 H), 4.27 – 4.15 (m, 2 H), 4.12 (t,  $J$  = 6.4 Hz, 2 H), 4.01 (d,  $J$  = 1.4 Hz, 2 H), 3.82 (d,  $J$  = 2.0 Hz, 3 H), 3.64 (s, 2 H), 3.49 (t,  $J$  = 6.1 Hz, 2 H), 2.36 (s, 3 H), 1.79 – 1.68 (m, 2 H), 1.67 – 1.56 (m, 2 H), 1.26 (t,  $J$  = 7.1, 1.8 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 170.3, 168.1, 155.9, 139.0, 135.7, 133.8, 131.0, 130.6, 130.5, 128.9, 114.8, 112.5, 111.5, 101.1, 70.9, 68.1, 64.6, 60.6, 55.5, 30.2, 25.9, 25.2, 14.0, 13.2. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{27}\text{H}_{30}\text{ClKNO}_7$  554.1342, found 554.1337.

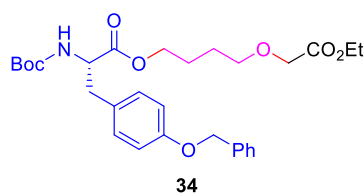


**4-(2-Ethoxy-2-oxoethoxy)butyl 2-(4-(2-(4-chlorobenzamido)ethyl)phenoxy)-2-methylpropanoate (32, new compound):** The reaction was performed following the general procedure.

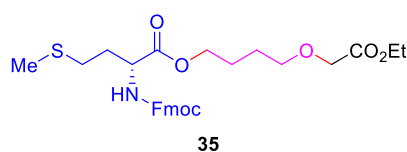
The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a white solid (187 mg, 90%); mp 48 – 52 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 – 7.57 (m, 2 H), 7.40 – 7.31 (m, 2 H), 7.05 (d,  $J$  = 8.5 Hz, 2 H), 6.76 (d,  $J$  = 8.5 Hz, 2 H), 6.35 – 6.34 (m, 1 H), 4.17 (q,  $J$  = 7.0 Hz, 4 H), 3.99 (s, 2 H), 3.64 – 3.59 (m, 2 H), 3.45 (t,  $J$  = 6.4 Hz, 2 H), 2.83 (t,  $J$  = 6.9 Hz, 2 H), 1.76 – 1.65 (m, 2 H), 1.59 – 1.49 (m, 8 H), 1.25 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  174.1, 170.3, 166.3, 153.8, 137.2, 132.8, 132.3, 129.2, 128.4, 128.2, 119.0, 78.9, 70.8, 68.0, 64.9, 60.6, 41.2, 34.4, 25.7, 25.2, 24.9, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{27}\text{H}_{34}\text{ClKNO}_7$  558.1655, found 558.1648.



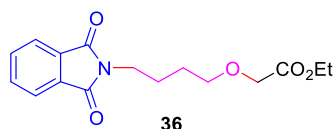
**4-(2-Ethoxy-2-oxoethoxy)butyl (2S,4aS,6aS,6bR,8aR,10S,12aS,12bR,14bR)-10-hydroxy-2,4a,6a,6b,9,9,12a-heptamethyl-13-oxo-1,2,3,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,12b,13,14b-icosahydricene-2-carboxylate (33, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (153 mg, 61%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.60 (s, 1 H), 4.19 (q,  $J$  = 7.1 Hz, 2 H), 4.13 – 4.06 (m, 2 H), 4.04 (s, 2 H), 3.54 (t,  $J$  = 5.9 Hz, 2 H), 3.22 – 3.18 (m, 1 H), 2.77 – 2.74 (m, 1 H), 2.31 (s, 1 H), 2.11 – 1.93 (m, 3 H), 1.93 – 1.78 (m, 2 H), 1.76 – 1.53 (m, 10 H), 1.41 – 1.32 (m, 6 H), 1.29 – 1.24 (m, 5 H), 1.17 – 1.09 (m, 10 H), 1.02 – 0.93 (m, 5 H), 0.77 (s, 6 H), 0.68 – 0.66 (m, 1 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  199.9, 176.1, 170.2, 169.0, 128.2, 78.3, 70.8, 68.0, 63.9, 61.5, 60.5, 54.7, 48.1, 45.1, 43.7, 42.9, 40.8, 38.9, 37.5, 36.8, 32.5, 31.6, 30.8, 28.3, 28.1, 27.9, 27.0, 26.2, 26.1, 25.9, 25.2, 23.1, 18.4, 17.2, 16.1, 15.4, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{38}\text{H}_{60}\text{KO}_7$  667.3971, found 667.3959.



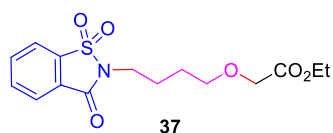
**4-(2-Ethoxy-2-oxoethoxy) butyl (S)-3-(4-(benzyloxy)phenyl)-2-((tert-butoxycarbonyl)amino)propanoate (34, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (171 mg, 81%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 – 7.28 (m, 5 H), 7.04 (d,  $J$  = 8.5 Hz, 2 H), 6.89 (d,  $J$  = 8.6 Hz, 2 H), 5.01 (d,  $J$  = 7.6 Hz, 3 H), 4.51 (d,  $J$  = 7.2 Hz, 1 H), 4.26 – 4.16 (m, 2 H), 4.15 – 4.06 (m, 2 H), 4.04 (s, 2 H), 3.51 (t,  $J$  = 6.1 Hz, 2 H), 3.09 – 2.94 (m, 2 H), 1.77 – 1.67 (m, 2 H), 1.65 – 1.60 (m, 2 H), 1.41 (s, 9 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.9, 170.3, 157.8, 155.0, 136.9, 130.2, 128.4, 128.2, 127.8, 127.3, 114.8, 79.7, 70.9, 69.9, 68.2, 64.9, 60.7, 54.5, 37.4, 28.2, 25.9, 25.1, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{29}\text{H}_{39}\text{NNaO}_8$  552.2568, found 552.2574.



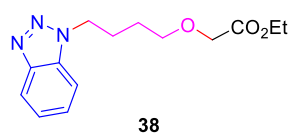
**4-(2-Ethoxy-2-oxoethoxy)butyl (((9H-fluoren-9-yl)methoxy)carbonyl)-D-methioninate (35, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (180 mg, 85%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J$  = 7.5 Hz, 2 H), 7.60 – 7.58 (m, 2 H), 7.43 – 7.34 (m, 2 H), 7.30 (t,  $J$  = 7.4 Hz, 2H), 5.53 (s, 1 H), 4.55 – 4.35 (m, 3 H), 4.23 – 4.17 (m, 5 H), 4.03 (s, 2 H), 3.58 – 3.48 (m, 2 H), 2.52 (t,  $J$  = 7.2 Hz, 2 H), 2.26 – 2.03 (m, 4 H), 1.96 (td,  $J$  = 14.4, 7.5 Hz, 1 H), 1.82 – 1.61 (m, 4 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.9, 170.2, 155.8, 143.7, 143.5, 141.2, 127.5, 126.8, 124.8, 119.8, 70.7, 68.0, 66.7, 65.2, 60.6, 53.0, 46.9, 31.7, 29.7, 25.8, 25.1, 15.2, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{28}\text{H}_{35}\text{KNO}_7\text{S}$  568.1766, found 568.1749.



**Ethyl 2-(4-(1,3-dioxoisindolin-2-yl)butoxy)acetate (36, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a white solid (107 mg, 88%); mp 60 – 63 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80 – 7.78 (m, 2 H), 7.68 – 7.66 (m, 2 H), 4.16 (q,  $J = 7.1$  Hz, 2 H), 4.01 (s, 2 H), 3.68 (t,  $J = 7.0$  Hz, 2 H), 3.53 (t,  $J = 6.3$  Hz, 2 H), 1.81 – 1.70 (m, 2 H), 1.66 – 1.59 (m, 2 H), 1.24 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 168.2, 133.7, 131.9, 123.0, 70.9, 68.2, 60.6, 37.4, 26.7, 25.1, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{16}\text{H}_{20}\text{NO}_5$  306.1336, found 306.1346.

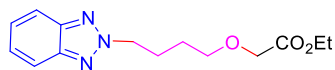


**Ethyl 2-(4-(1,1-dioxido-3-oxobenzo[d]isothiazol-2(3H)-yl)butoxy)acetate (37, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (111.8 mg, 82%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03 (d,  $J = 7.1$  Hz, 1 H), 7.95 – 7.77 (m, 3 H), 4.20 (q,  $J = 7.1$  Hz, 2 H), 4.05 (s, 2 H), 3.81 (t,  $J = 7.3$  Hz, 2 H), 3.58 (t,  $J = 6.2$  Hz, 2 H), 2.02 – 1.89 (m, 2 H), 1.79 – 1.68 (m, 2 H), 1.26 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 158.9, 137.6, 134.6, 134.2, 127.3, 125.0, 120.8, 70.8, 68.3, 60.7, 39.0, 26.7, 25.1, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{15}\text{H}_{19}\text{NNaO}_6\text{S}$  364.0825, found 364.0836.



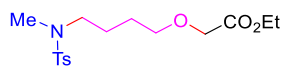
**Ethyl 2-(4-(2H-benzo[d][1,2,3]triazol-2-yl)butoxy)acetate (38, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (20:1 PE/EA) to give the product as a colorless oil (27 mg, 24%);  $^1\text{H}$  NMR (300

MHz, CDCl<sub>3</sub>) δ 8.04 (d, *J* = 8.3 Hz, 1 H), 7.57 (d, *J* = 8.3 Hz, 1 H), 7.47 (t, *J* = 7.6 Hz, 1 H), 7.35 (t, *J* = 7.6 Hz, 1 H), 4.71 (t, *J* = 7.1 Hz, 2 H), 4.20 (q, *J* = 7.1 Hz, 2 H), 4.03 (s, 2 H), 3.56 (t, *J* = 6.0 Hz, 2 H), 2.22 – 2.08 (m, 2 H), 1.74 – 1.58 (m, 2 H), 1.26 (t, *J* = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>) δ 170.3, 145.8, 132.8, 127.0, 123.6, 119.7, 109.4, 70.6, 68.1, 60.7, 47.6, 26.5, 26.4, 14.0. HRMS (ESI) *m/z*: [M + Na]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>19</sub>N<sub>3</sub>NaO<sub>3</sub> 300.1319, found 300.1311.



39

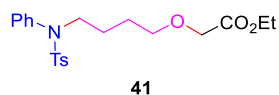
**Ethyl 2-(4-(2H-benzo[d][1,2,3]triazol-2-yl)butoxy)acetate (39, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (20:1 PE/EA) to give the product as a colorless oil (53 mg, 48%); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.82 – 7.79 (m, 2 H), 7.32 – 7.26 (m, 2 H), 4.75 (t, *J* = 7.0 Hz, 2 H), 4.15 (q, *J* = 7.1 Hz, 2 H), 4.00 (s, 2 H), 3.53 (t, *J* = 6.2 Hz, 2 H), 2.27 – 2.14 (m, 2 H), 1.69 – 1.53 (m, 2 H), 1.22 (t, *J* = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ 170.3, 144.1, 126.0, 117.8, 70.6, 68.1, 60.7, 56.0, 26.6, 26.4, 14.0. HRMS (ESI) *m/z*: [M + Na]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>19</sub>N<sub>3</sub>NaO<sub>3</sub> 300.1319, found 300.1312.



40

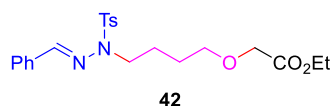
**Ethyl 2-(4-((N,4-dimethylphenyl)sulfonamido)butoxy)acetate (40, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (119 mg, 87%); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.63 (d, *J* = 8.2 Hz, 2 H), 7.29 (d, *J* = 8.1 Hz, 2 H), 4.24 – 4.12 (m, 2 H), 4.03 (s, 2 H), 3.59 – 3.48 (m, 2 H), 2.99 (t, *J* = 6.5 Hz, 2 H), 2.68 (s, 3 H), 2.40 (s, 3 H), 1.70 – 1.58 (m, 4 H), 1.26 (t, *J* = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ 169.9, 142.7, 133.9, 129.2, 126.8, 70.4, 67.7, 60.1, 49.2, 34.0, 25.9, 23.5, 20.9, 13.7. HRMS (ESI) *m/z*: [M + Na]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>25</sub>NNaO<sub>5</sub>S 366.1346, found 366.1356.



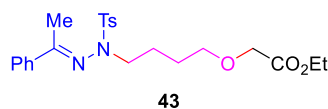


**Ethyl 2-(4-((4-methyl-N-phenylphenyl)sulfonamido)butoxy)acetate (41, new compound):**

The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (92 mg, 57%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45 (d,  $J$  = 8.2 Hz, 2 H), 7.33 – 7.27 (m, 3 H), 7.23 (d,  $J$  = 8.1 Hz, 2 H), 7.07 – 7.00 (m, 2 H), 4.19 (q,  $J$  = 7.1 Hz, 2 H), 4.00 (s, 2 H), 3.56 (t,  $J$  = 6.9 Hz, 2 H), 3.49 (t,  $J$  = 6.3 Hz, 2 H), 2.41 (s, 3 H), 1.70 – 1.60 (m, 2 H), 1.56 – 1.46 (m, 2 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 143.3, 139.0, 135.2, 129.3, 128.9, 128.7, 127.8, 127.7, 70.9, 68.2, 60.7, 50.0, 26.2, 24.6, 21.5, 14.2. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{21}\text{H}_{27}\text{NNaO}_5\text{S}$  428.1502, found 428.1507.

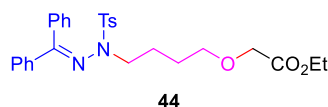


**Ethyl (E)-2-(4-(2-benzylidene-1-tosylhydrazinyl)butoxy)acetate (42, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (131 mg, 76%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 (s, 1 H), 7.75 (d,  $J$  = 8.3 Hz, 2 H), 7.69 – 7.62 (m, 2 H), 7.39 – 7.37 (m, 3 H), 7.28 (d,  $J$  = 8.1 Hz, 2 H), 4.19 (q,  $J$  = 7.1 Hz, 2 H), 4.05 (s, 2 H), 3.64 – 3.55 (m, 4 H), 2.39 (s, 3 H), 1.73 (t,  $J$  = 4.5 Hz, 4 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 149.1, 143.7, 134.3, 133.9, 130.1, 129.3, 128.5, 128.0, 127.4, 70.7, 68.0, 60.6, 47.9, 26.4, 24.2, 21.3, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{22}\text{H}_{28}\text{N}_2\text{NaO}_5\text{S}$  455.1611, found 455.1609.



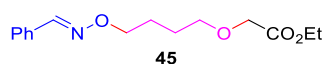
**Ethyl (E)-2-(4-(2-(1-phenylethylidene)-1-tosylhydrazinyl)butoxy)acetate (43, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a slightly yellow oil (148 mg, 83%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85 – 7.79 (m, 2 H), 7.67 (d,  $J$  = 8.2 Hz, 2 H),

7.50 – 7.37 (m, 3 H), 7.33 (d,  $J = 8.1$  Hz, 2 H), 4.18 (q,  $J = 7.1$  Hz, 2 H), 4.01 (s, 2 H), 3.50 (t,  $J = 6.4$  Hz, 2 H), 3.16 (t,  $J = 7.1$  Hz, 2 H), 2.64 (s, 3 H), 2.44 (s, 3 H), 1.70 – 1.60 (m, 2 H), 1.53 – 1.41 (m, 2 H), 1.25 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  177.8, 170.3, 143.8, 137.0, 131.9, 130.9, 129.2, 128.8, 128.3, 127.1, 70.9, 68.1, 60.6, 52.1, 26.6, 24.3, 21.5, 18.0, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{23}\text{H}_{31}\text{N}_2\text{O}_5\text{S}$  447.1948, found 447.1944.

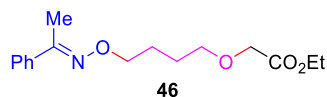


**Ethyl 2-(4-(2-(diphenylmethylene)-1-tosylhydrazinyl)butoxy)acetate (44, new compound):**

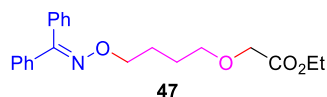
The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (157 mg, 77%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80 (d,  $J = 8.2$  Hz, 2 H), 7.61 – 7.54 (m, 2 H), 7.53 – 7.42 (m, 6 H), 7.39 – 7.32 (m, 4 H), 4.18 (q,  $J = 7.1$  Hz, 2 H), 3.93 (s, 2 H), 3.27 (t,  $J = 6.0$  Hz, 2 H), 2.97 (t,  $J = 6.8$  Hz, 2 H), 2.47 (s, 3 H), 1.29 – 1.14 (m, 7 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  174.8, 170.2, 143.8, 137.2, 135.0, 132.1, 131.0, 129.4, 129.3, 129.1, 129.01, 128.98, 128.0, 127.8, 70.7, 67.9, 60.5, 52.5, 26.3, 23.6, 21.5, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{28}\text{H}_{33}\text{N}_2\text{O}_5\text{S}$  509.2105, found 509.2113.



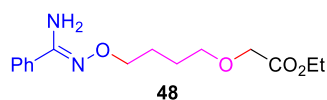
**Ethyl (E)-2-(4-((benzylideneamino)oxy)butoxy)acetate (45, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (102 mg, 91%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06 (s, 1 H), 7.61 – 7.53 (m, 2 H), 7.40 – 7.31 (m, 3 H), 4.24 – 4.18 (m, 4 H), 4.06 (s, 2 H), 3.58 (t,  $J = 6.3$  Hz, 2 H), 1.88 – 1.67 (m, 4 H), 1.28 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 148.3, 132.3, 129.6, 128.6, 126.9, 73.8, 71.4, 68.3, 60.7, 26.0, 25.7, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{22}\text{NO}_4$  280.1543, found 280.1552.



**Ethyl (E)-2-(4-(((1-phenylethylidene)amino)oxy)butoxy)acetate (46, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (77 mg, 65%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66 – 7.60 (m, 2 H), 7.39 – 7.31 (m, 3 H), 4.26 – 4.16 (m, 4 H), 4.07 (s, 2 H), 3.58 (t,  $J$  = 6.3 Hz, 2 H), 2.22 (s, 3 H), 1.87 – 1.71 (m, 4 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 154.2, 136.7, 128.8, 128.3, 125.9, 73.7, 71.5, 68.3, 60.7, 26.1, 25.8, 14.1, 12.6. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{16}\text{H}_{24}\text{NO}_4$  294.1700, found 294.1708.

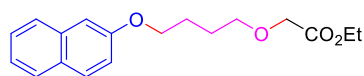


**Ethyl 2-(4-(((diphenylmethylene)amino)oxy)butoxy)acetate (47, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (88 mg, 62%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.50 – 7.46 (m, 2 H), 7.46 – 7.38 (m, 3 H), 7.38 – 7.28 (m, 5 H), 4.26 – 4.17 (m, 4 H), 4.03 (s, 2 H), 3.53 (t,  $J$  = 6.5 Hz, 2 H), 1.85 – 1.75 (m, 2 H), 1.74 – 1.64 (m, 2 H), 1.28 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 156.4, 136.6, 133.4, 129.2, 129.1, 128.6, 128.1, 128.0, 127.8, 74.2, 71.5, 68.3, 60.7, 26.1, 25.7, 14.2. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{21}\text{H}_{25}\text{NNaO}_4$  378.1676, found 378.1685.



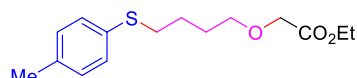
**Ethyl (Z)-2-(4-(((amino(phenyl)methylene)amino)oxy)butoxy)acetate (48, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (74 mg, 63%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 – 7.60 (m, 2 H), 7.44 – 7.32 (m, 3 H), 4.84 (s, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.11 (t,  $J$  = 6.2 Hz, 2 H), 4.05 (s, 2 H), 3.57 (t,  $J$  = 6.3 Hz, 2 H), 1.85 – 1.70 (m, 4 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 151.6, 132.3, 129.4, 128.2,

125.6, 72.8, 71.3, 68.0, 60.4, 25.9, 25.4, 13.9. HRMS (ESI)  $m/z$ :  $[M + Na]^+$  Calcd for  $C_{15}H_{22}N_2NaO_4$  317.1472, found 317.1462.



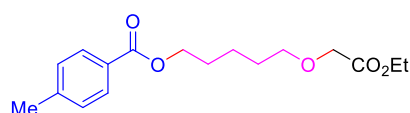
49

**Ethyl 2-(4-(naphthalen-2-yloxy)butoxy)acetate (49, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (98 mg, 81%);  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.80 – 7.70 (m, 3 H), 7.49 – 7.39 (m, 1 H), 7.37 – 7.29 (m, 1 H), 7.20 – 7.10 (m, 2 H), 4.23 (q,  $J = 7.1$  Hz, 2 H), 4.14 – 4.10 (m, 4 H), 3.64 (t,  $J = 6.3$  Hz, 2 H), 2.03 – 1.92 (m, 2 H), 1.91 – 1.80 (m, 2 H), 1.29 (t,  $J = 7.1$  Hz, 3 H).  $^{13}C\{^1H\}$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  170.3, 156.8, 134.4, 129.1, 128.7, 127.4, 126.5, 126.1, 123.2, 118.7, 106.3, 71.1, 68.1, 67.2, 60.5, 26.0, 25.7, 14.0. HRMS (ESI)  $m/z$ :  $[M + Na]^+$  Calcd for  $C_{18}H_{22}NaO_4$  325.1410, found 325.1412.



50

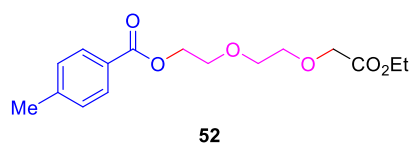
**Ethyl 2-(4-(p-tolylthio)butoxy)acetate (50, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (46 mg, 41%);  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.24 (d,  $J = 8.1$  Hz, 2 H), 7.07 (d,  $J = 8.0$  Hz, 2 H), 4.20 (q,  $J = 7.1$  Hz, 2 H), 4.03 (s, 2 H), 3.52 (t,  $J = 5.9$  Hz, 2 H), 2.90 (t,  $J = 6.8$  Hz, 2 H), 2.30 (s, 3 H), 1.78 – 1.68 (m, 4 H), 1.27 (t,  $J = 7.1$  Hz, 3 H).  $^{13}C\{^1H\}$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  170.4, 135.9, 132.7, 129.9, 129.5, 71.1, 68.2, 60.7, 34.0, 28.4, 25.7, 20.9, 14.1. HRMS (ESI)  $m/z$ :  $[M + K]^+$  Calcd for  $C_{15}H_{22}KO_3S$  321.0921, found 321.0916.



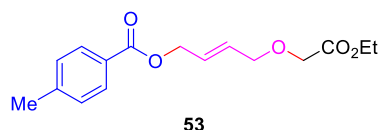
51

**5-(2-Ethoxy-2-oxoethoxy)pentyl 4-methylbenzoate (51, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column

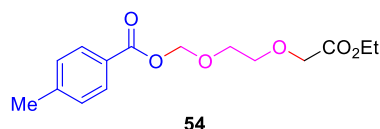
chromatography (10:1 PE/EA) to give the product as a colorless oil (68 mg, 55%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (d,  $J$  = 8.1 Hz, 2 H), 7.21 (d,  $J$  = 8.0 Hz, 2 H), 4.29 (t,  $J$  = 6.6 Hz, 2 H), 4.19 (q,  $J$  = 7.1 Hz, 2 H), 4.04 (s, 2 H), 3.54 (t,  $J$  = 6.5 Hz, 2 H), 2.38 (s, 2 H), 1.82 – 1.74 (m, 3 H), 1.73 – 1.64 (m, 2 H), 1.57 – 1.48 (m, 2 H), 1.26 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 166.6, 143.4, 129.5, 128.9, 127.6, 71.5, 68.3, 64.6, 60.7, 29.1, 28.5, 22.6, 21.5, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{17}\text{H}_{24}\text{NaO}_5$  331.1516, found 331.1513.



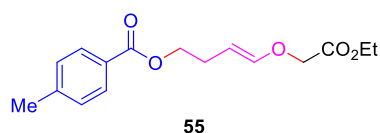
**2-(2-(2-Ethoxy-2-oxoethoxy)ethoxy)ethyl 4-methylbenzoate (52, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (77 mg, 62%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.95 – 7.89 (m, 2 H), 7.20 (d,  $J$  = 8.0 Hz, 2 H), 4.47 – 4.40 (m, 2 H), 4.21 – 4.10 (m, 4 H), 3.82 – 3.20 (m, 2 H), 3.72 (m, 4 H), 2.37 (d,  $J$  = 2.1 Hz, 3 H), 1.26 – 1.22 (m, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 166.5, 143.5, 129.6, 128.9, 127.2, 70.8, 70.6, 69.2, 68.6, 63.8, 60.7, 21.5, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{16}\text{H}_{22}\text{KO}_6$  349.1048, found 349.1039.



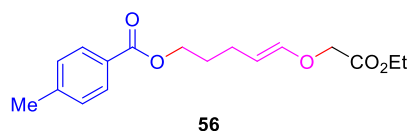
**(E)-4-(2-(2-Ethoxy-2-oxoethoxy)but-2-en-1-yl) 4-methylbenzoate (53, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (60 mg, 51%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (d,  $J$  = 8.1 Hz, 2 H), 7.22 (d,  $J$  = 8.1 Hz, 2 H), 5.94 – 5.75 (m, 2 H), 4.88 – 4.86 (d,  $J$  = 5.2 Hz, 2 H), 4.31 – 4.16 (m, 4 H), 4.09 (s, 2 H), 2.39 (s, 3 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 166.4, 143.7, 129.9, 129.6, 129.1, 127.7, 127.3, 67.5, 66.8, 60.9, 60.4, 21.6, 14.2. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{16}\text{H}_{20}\text{KO}_5$  331.0942, found 331.0936.



**2-((2-Ethoxy-2-oxoethoxy)methoxy)ethyl 4-methylbenzoate (54, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (97 mg, 82%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.93 (d,  $J = 8.1$  Hz, 2 H), 7.21 (d,  $J = 8.0$  Hz, 2 H), 5.54 (s, 2 H), 4.24 – 4.09 (m, 4 H), 3.92 – 3.89 (m, 2 H), 3.76 – 3.72 (m, 2 H), 2.38 (s, 3 H), 1.24 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 165.9, 143.9, 129.7, 129.0, 126.9, 89.6, 70.4, 69.6, 68.6, 60.7, 21.5, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{15}\text{H}_{20}\text{NaO}_6$  319.1152, found 319.1159.

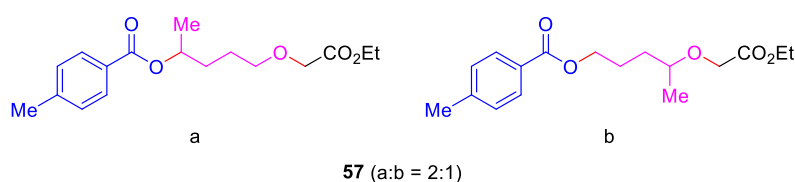


**(E)-4-(2-Ethoxy-2-oxoethoxy)but-1-en-1-yl 4-methylbenzoate (55, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (55 mg, 47%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.93 (d,  $J = 8.2$  Hz, 2 H), 7.22 (d,  $J = 8.0$  Hz, 2 H), 6.01 – 5.99 (m, 1 H), 4.6 – 4.54 (m, 1 H), 4.36 – 4.27 (m, 4 H), 4.22 (q,  $J = 7.1$  Hz, 2 H), 2.64 – 2.54 (m, 2 H), 2.39 (s, 3 H), 1.28 (t,  $J = 7.1$  Hz, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.3, 166.7, 146.2, 143.3, 129.6, 129.0, 127.7, 103.6, 68.5, 64.2, 61.1, 23.8, 21.6, 14.1. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{16}\text{H}_{20}\text{KO}_5$  331.0942, found 331.0936.

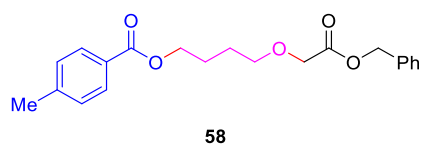


**(E)-5-(2-Ethoxy-2-oxoethoxy)pent-1-en-1-yl 4-methylbenzoate (56, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (49 mg, 40%);  $^1\text{H}$

NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.93 (d,  $J$  = 8.2 Hz, 2 H), 7.22 (d,  $J$  = 8.0 Hz, 2 H), 5.94 – 5.93 (m, 1 H), 4.53 – 4.48 (m, 1 H), 4.32 (t,  $J$  = 6.6 Hz, 2 H), 4.27 (d,  $J$  = 7.1 Hz, 2 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 2.39 (s, 3 H), 2.32 – 2.27 (m, 2 H), 1.88 – 1.81 (m, 2 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  169.4, 166.7, 145.1, 143.3, 129.5, 129.0, 127.8, 107.3, 68.5, 64.3, 61.1, 28.5, 21.6, 20.4, 14.1. HRMS (ESI)  $m/z$ : [M + K]<sup>+</sup> Calcd for C<sub>17</sub>H<sub>22</sub>KO<sub>5</sub> 345.1099, found 345.1110.

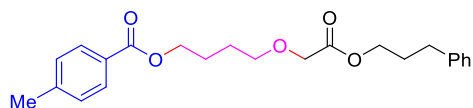


**5-(2-Ethoxy-2-oxoethoxy)pentan-2-yl 4-methylbenzoate (57, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (62 mg, 50%); Major isomer: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.92 – 7.90 (m, 2 H), 7.26 – 7.21 (m, 2 H), 4.40 – 4.25 (m, 1 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.13 – 4.03 (m, 2 H), 3.61 – 3.50 (m, 2 H), 2.39 (s, 3 H), 2.02 – 1.50 (m, 4 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H), 1.20 – 1.18 (m, 3 H). Minor isomer: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.92 – 7.90 (m, 2 H), 7.26 – 7.21 (m, 2 H), 5.27 – 4.98 (m, 1 H), 4.20 (q,  $J$  = 7.1 Hz, 2 H), 4.13 – 4.03 (m, 2 H), 3.69 – 3.40 (m, 2 H), 2.39 (s, 3 H), 2.05 – 1.55 (m, 4 H), 1.34 – 1.33 (m, 3 H), 1.27 (t,  $J$  = 7.1 Hz, 3 H). The mixture of two isomers: <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.8, 170.5, 166.7, 166.2, 143.4, 143.3, 129.51, 129.49, 129.0, 128.9, 128.0, 127.6, 75.9, 71.4, 71.0, 68.3, 65.94, 64.7, 60.7, 32.9, 32.5, 25.5, 24.8, 21.6, 20.1, 19.2, 14.1. HRMS (ESI)  $m/z$ : [M + K]<sup>+</sup> Calcd for C<sub>17</sub>H<sub>24</sub>KO<sub>5</sub> 347.1255, found 347.1254.



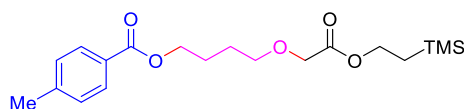
**4-(2-(Benzyloxy)-2-oxoethoxy)butyl 4-methylbenzoate (58, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (108 mg, 76%); <sup>1</sup>H NMR

(400 MHz, CDCl<sub>3</sub>)  $\delta$  7.92 (d,  $J$  = 8.2 Hz, 2 H), 7.39 – 7.31 (m, 5 H), 7.22 (d,  $J$  = 8.0 Hz, 2 H), 5.19 (s, 2 H), 4.33 (t,  $J$  = 6.3 Hz, 2 H), 4.13 (s, 2 H), 3.60 (t,  $J$  = 6.2 Hz, 2 H), 2.40 (s, 3 H), 1.92 – 1.82 (m, 2 H), 1.81 – 1.76 (m, 2 H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 166.6, 143.4, 135.4, 129.5, 129.0, 128.6, 128.40, 128.37, 127.6, 71.2, 68.3, 66.5, 64.4, 26.2, 25.4, 21.6. HRMS (ESI)  $m/z$ : [M + H]<sup>+</sup> Calcd for C<sub>21</sub>H<sub>25</sub>O<sub>5</sub> 357.1697, found 357.1702.



59

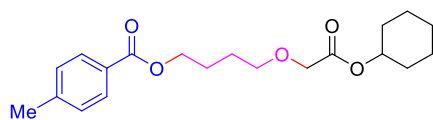
**4-(2-Oxo-2-(3-phenylpropoxy)ethoxy)butyl 4-methylbenzoate (59, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (112 mg, 73%); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.93 (d,  $J$  = 8.1 Hz, 2 H), 7.31 – 7.16 (m, 7 H), 4.35 (t,  $J$  = 6.2 Hz, 2 H), 4.19 (t,  $J$  = 6.6 Hz, 2 H), 4.08 (s, 2 H), 3.60 (t,  $J$  = 6.1 Hz, 2 H), 2.75 – 2.62 (m, 2 H), 2.40 (s, 3 H), 2.06 – 1.95 (m, 2 H), 1.91 – 1.71 (m, 4 H). <sup>13</sup>C{<sup>1</sup>H} NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  170.4, 166.6, 143.4, 140.9, 129.5, 128.9, 128.4, 128.3, 127.5, 126.0, 71.1, 68.2, 64.4, 64.1, 32.0, 30.0, 26.2, 25.4, 21.6. HRMS (ESI)  $m/z$ : [M + Na]<sup>+</sup> Calcd for C<sub>23</sub>H<sub>28</sub>NaO<sub>5</sub> 407.1829, found 407.1821.



60

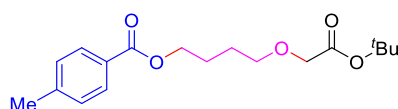
**4-(2-Oxo-2-(2-(trimethylsilyl)ethoxy)ethoxy)butyl 4-methylbenzoate (60, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (144 mg, 98%); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.90 (d,  $J$  = 8.2 Hz, 2 H), 7.20 (d,  $J$  = 8.0 Hz, 2 H), 4.37 – 4.29 (m, 2 H), 4.27 – 4.14 (m, 2 H), 4.03 (d,  $J$  = 6.3 Hz, 2 H), 3.61 – 3.55 (m, 2 H), 2.38 (s, 3 H), 1.89 – 1.83 (m, 2 H), 1.81 – 1.68 (m, 2 H), 1.04 – 0.94 (m, 2 H), 0.02 (s, 9 H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.5, 166.5, 143.3, 129.4, 128.9, 127.5, 71.0, 68.3, 64.3, 63.0, 26.1, 25.3, 21.5, 17.2, -1.7. HRMS (ESI)  $m/z$ : [M + Na]<sup>+</sup> Calcd for C<sub>19</sub>H<sub>30</sub>NaO<sub>5</sub>Si 389.1755, found 389.1748.





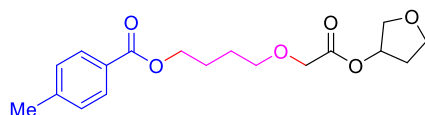
61

**4-(2-(Cyclohexyloxy)-2-oxoethoxy)butyl 4-methylbenzoate (61, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (113 mg, 81%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (d,  $J = 8.1$  Hz, 2 H), 7.22 (d,  $J = 8.1$  Hz, 2 H), 4.87 – 4.80 (m, 1 H), 4.33 (t,  $J = 6.3$  Hz, 2 H), 4.05 (s, 2 H), 3.59 (t,  $J = 6.1$  Hz, 2 H), 2.40 (s, 3 H), 1.96 – 1.65 (m, 8 H), 1.61 – 1.47 (m, 1 H), 1.47 – 1.18 (m, 5 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.9, 166.6, 143.4, 129.5, 128.9, 127.5, 73.2, 71.1, 68.4, 64.4, 31.5, 26.2, 25.4, 25.2, 23.6, 21.6. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{20}\text{H}_{28}\text{NaO}_5$  371.1829, found 371.1838.



62

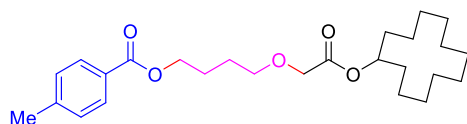
**4-(2-(Tert-butoxy)-2-oxoethoxy)butyl 4-methylbenzoate (62, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (125 mg, 97%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.90 (d,  $J = 8.2$  Hz, 2 H), 7.20 (d,  $J = 8.1$  Hz, 2 H), 4.32 (t,  $J = 6.4$  Hz, 2 H), 3.94 (s, 2 H), 3.56 (t,  $J = 6.3$  Hz, 2 H), 2.39 (s, 3 H), 1.90 – 1.83 (m, 2 H), 1.82 – 1.71 (m, 2 H), 1.46 (s, 9 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.7, 166.6, 143.4, 129.5, 128.9, 127.6, 81.5, 71.0, 68.7, 64.4, 28.0, 26.2, 25.4, 21.5. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{18}\text{H}_{26}\text{KO}_5$  361.1412, found 361.1405.



63

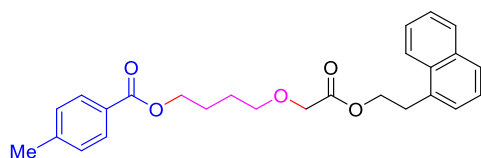
**4-(2-Oxo-2-((tetrahydrofuran-3-yl)oxy)ethoxy)butyl 4-methylbenzoate (63, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (77 mg, 57%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (d,  $J = 8.1$  Hz, 2 H), 7.22 (d,  $J = 8.0$  Hz, 2 H), 5.44

– 5.30 (m, 1 H), 4.33 (t,  $J = 6.4$  Hz, 2 H), 4.07 (s, 2 H), 3.93 – 3.89 (m, 2 H), 3.87 – 3.80 (m, 2 H), 3.58 (t,  $J = 6.2$  Hz, 2 H), 2.39 (s, 3 H), 2.23 – 2.14 (m, 1 H), 2.07 – 1.95 (m, 1 H), 1.90 – 1.83 (m, 2 H), 1.80 – 1.74 (m, 2 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 166.6, 143.4, 129.5, 129.0, 127.6, 75.3, 73.0, 71.2, 68.2, 66.9, 64.4, 32.6, 26.2, 25.4, 21.6. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{K}]^+$  Calcd for  $\text{C}_{18}\text{H}_{24}\text{KO}_6$  375.1204, found 375.1200.



64

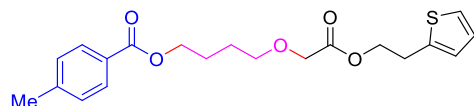
**4-(2-(Cyclododecyloxy)-2-oxoethoxy)butyl 4-methylbenzoate (64, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a slightly yellow oil (124 mg, 72%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (d,  $J = 7.9$  Hz, 2 H), 7.22 (d,  $J = 7.9$  Hz, 2 H), 5.21 – 5.05 (m, 1 H), 4.33 (t,  $J = 6.2$  Hz, 2 H), 4.04 (s, 2 H), 3.59 (t,  $J = 6.1$  Hz, 2 H), 2.40 (s, 3 H), 1.9 – 1.68 (m, 6 H), 1.59 – 1.24 (m, 20 H).  $^{13}\text{C}$  NMR (75 MHz,  $d_6$ -DMSO)  $\delta$  169.9, 165.7, 143.4, 129.2, 129.1, 127.2, 71.5, 70.2, 67.7, 64.2, 28.7, 25.7, 25.1, 23.5, 23.3, 23.0, 22.7, 21.1, 20.4. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{26}\text{H}_{40}\text{NaO}_5$  455.2768, found 455.2761.



65

**4-(2-(2-(Naphthalen-1-yl)ethoxy)-2-oxoethoxy)butyl 4-methylbenzoate (65, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (126 mg, 75%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.09 (d,  $J = 8.4$  Hz, 1 H), 7.94 (d,  $J = 8.2$  Hz, 2 H), 7.86 (d,  $J = 7.7$  Hz, 1 H), 7.76 (d,  $J = 8.0$  Hz, 1 H), 7.59 – 7.46 (m, 2 H), 7.44 – 7.34 (m, 2 H), 7.23 (d,  $J = 8.0$  Hz, 2 H), 4.52 (t,  $J = 7.4$  Hz, 2 H), 4.34 (t,  $J = 6.4$  Hz, 2 H), 4.07 (s, 2 H), 3.55 (t,  $J = 6.3$  Hz, 2 H), 3.45 (t,  $J = 7.4$  Hz, 2 H), 2.40 (s, 3 H), 1.91 – 1.82 (m, 2 H), 1.80 – 1.75 (m, 2 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 166.6, 143.4, 133.8, 133.3, 131.9, 129.5,

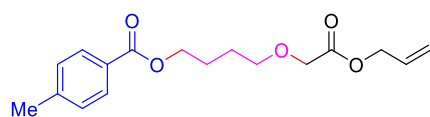
129.0, 128.7, 127.6, 127.5, 126.9, 126.1, 125.6, 125.4, 123.4, 71.1, 68.2, 64.6, 64.4, 32.1, 26.1, 25.3, 21.5. HRMS (ESI)  $m/z$ :  $[M + K]^+$  Calcd for  $C_{26}H_{28}KO_5$  459.1568, found 459.1578.



66

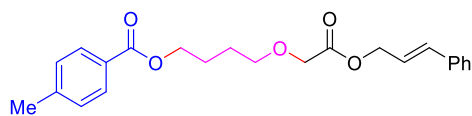
**4-(2-Oxo-2-(2-(thiophen-2-yl)ethoxy)ethoxy)butyl 4-methylbenzoate (66, new compound):**

The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (146 mg, 97%);  $^1H$  NMR (300 MHz,  $CDCl_3$ )  $\delta$  7.93 (d,  $J$  = 8.1 Hz, 2 H), 7.23 (d,  $J$  = 8.0 Hz, 2 H), 7.15 (d,  $J$  = 5.1 Hz, 1 H), 6.97 – 6.91 (m, 1 H), 6.86 (d,  $J$  = 3.3 Hz, 1 H), 4.44 – 4.29 (m, 4 H), 4.10 (s, 2 H), 3.57 (t,  $J$  = 6.1 Hz, 2 H), 3.18 (t,  $J$  = 6.7 Hz, 2 H), 2.40 (s, 3 H), 1.91 – 1.73 (m, 4 H).  $^{13}C\{^1H\}$  NMR (75 MHz,  $CDCl_3$ )  $\delta$  170.1, 166.4, 143.2, 139.3, 129.3, 128.8, 127.4, 126.7, 125.4, 123.9, 71.0, 68.0, 64.6, 64.2, 29.0, 26.0, 25.2, 21.4. HRMS (ESI)  $m/z$ :  $[M + Na]^+$  Calcd for  $C_{20}H_{24}NaO_5S$  399.1237, found 399.1242.



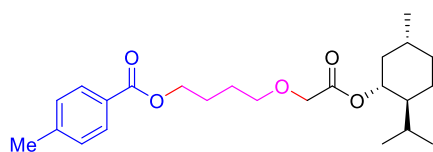
67

**4-(2-(Allyloxy)-2-oxoethoxy)butyl 4-methylbenzoate (67, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (65 mg, 53%);  $^1H$  NMR (300 MHz,  $CDCl_3$ )  $\delta$  7.92 (d,  $J$  = 8.2 Hz, 2 H), 7.23 (d,  $J$  = 8.1 Hz, 2 H), 5.99 – 5.86 (m, 1 H), 5.36 – 5.24 (m, 2 H), 4.65 (d,  $J$  = 5.8 Hz, 2 H), 4.33 (t,  $J$  = 6.2 Hz, 2 H), 4.11 (s, 2 H), 3.60 (t,  $J$  = 6.1 Hz, 2 H), 2.40 (s, 3 H), 1.98 – 1.74 (m, 4 H).  $^{13}C\{^1H\}$  NMR (75 MHz,  $CDCl_3$ )  $\delta$  170.1, 166.6, 143.5, 131.6, 129.5, 129.0, 127.6, 118.8, 71.2, 68.2, 65.4, 64.4, 26.2, 25.4, 21.6. HRMS (ESI)  $m/z$ :  $[M + K]^+$  Calcd for  $C_{17}H_{22}KO_5$  345.1099, found 345.1093.



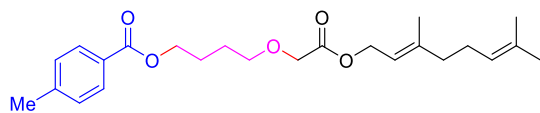
68

**4-(2-(Cinnamyloxy)-2-oxoethoxy)butyl 4-methylbenzoate (68, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (98 mg, 64%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (d,  $J$  = 8.1 Hz, 2 H), 7.43 – 7.18 (m, 7 H), 6.67 (d,  $J$  = 15.9 Hz, 1 H), 6.34 – 6.24 (m, 1 H), 4.82 (d,  $J$  = 6.5 Hz, 2 H), 4.34 (t,  $J$  = 6.2 Hz, 2 H), 4.13 (s, 2 H), 3.62 (t,  $J$  = 6.1 Hz, 2 H), 2.40 (s, 3 H), 1.95 – 1.75 (m, 4 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 166.6, 143.4, 135.9, 134.8, 129.5, 128.9, 128.5, 128.1, 127.5, 126.6, 122.5, 71.2, 68.2, 65.3, 64.4, 26.1, 25.4, 21.6. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{23}\text{H}_{26}\text{NaO}_5$  405.1672, found 405.1677.



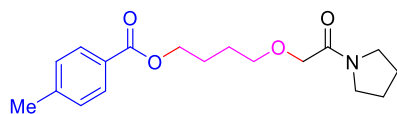
69

**4-(2-(((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)oxy)-2-oxoethoxy)butyl 4-methylbenzoate (69, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (141 mg, 87%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (d,  $J$  = 8.2 Hz, 1H), 7.22 (d,  $J$  = 8.1 Hz, 2 H), 4.82 – 4.73 (m, 1 H), 4.33 (t,  $J$  = 6.3 Hz, 2 H), 4.12 – 3.97 (m, 2 H), 3.59 (t,  $J$  = 6.1 Hz, 2 H), 2.39 (d,  $J$  = 6.7 Hz, 3 H), 2.04 – 1.98 (m, 1 H), 1.94 – 1.73 (m, 5 H), 1.72 – 1.61 (m, 2 H), 1.57 – 1.31 (m, 2 H), 1.15 – 0.95 (m, 2 H), 0.94 – 0.83 (m, 7 H), 0.80 – 0.71 (m, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.0, 166.6, 143.4, 129.5, 128.9, 127.5, 74.7, 71.1, 68.3, 64.4, 46.9, 40.8, 34.1, 31.3, 26.2, 25.4, 23.3, 21.9, 21.6, 20.6, 16.2. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{24}\text{H}_{36}\text{NaO}_5$  427.2455, found 427.2454.



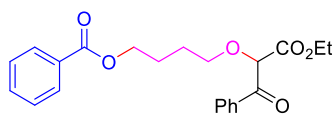
70

**(E)-4-(2-((3,7-dimethylocta-2,6-dien-1-yl)oxy)-2-oxoethoxy)butyl 4-methylbenzoate (70, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (158 mg, 95%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (d,  $J = 8.2$  Hz, 2 H), 7.22 (d,  $J = 8.1$  Hz, 2 H), 5.40 – 5.27 (m, 1 H), 5.06 (t,  $J = 5.9$  Hz, 1 H), 4.67 (d,  $J = 7.2$  Hz, 2 H), 4.33 (t,  $J = 6.2$  Hz, 2 H), 4.08 (s, 2 H), 3.59 (t,  $J = 6.1$  Hz, 2 H), 2.40 (s, 3 H), 2.15 – 2.00 (m, 4 H), 1.95 – 1.74 (m, 4 H), 1.70 (s, 3 H), 1.67 (s, 3 H), 1.59 (s, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 166.6, 143.4, 142.8, 131.8, 129.5, 129.0, 127.6, 123.6, 117.7, 71.1, 68.3, 64.4, 61.6, 39.4, 26.2 (2C, overlap), 25.6, 25.4, 21.6, 17.6, 16.4. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{24}\text{H}_{34}\text{NaO}_5$  425.2298, found 425.2293.



71

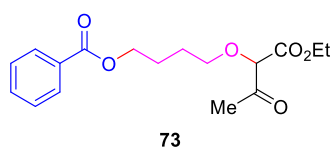
**4-(2-Oxo-2-(pyrrolidin-1-yl)ethoxy)butyl 4-methylbenzoate (71, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (88 mg, 69%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (d,  $J = 8.2$  Hz, 2 H), 7.22 (d,  $J = 8.0$  Hz, 2 H), 4.32 (t,  $J = 6.2$  Hz, 2 H), 4.08 (s, 2 H), 3.59 (t,  $J = 6.1$  Hz, 2 H), 3.45 (s, 4 H), 2.40 (s, 3 H), 1.99 – 1.71 (m, 8 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  167.6, 166.5, 143.3, 129.4, 128.9, 127.5, 70.8, 70.4, 64.4, 45.7, 45.5, 26.11, 26.06, 25.4, 23.7, 21.5. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{18}\text{H}_{25}\text{NNaO}_4$  342.1676, found 342.1675.



72

**4-((1-Ethoxy-1,3-dioxo-3-phenylpropan-2-yl)oxy)butyl benzoate (72, new compound):** The reaction was performed following the general procedure. The residue was purified by flash

column chromatography (10:1 PE/EA) to give the product as a colorless oil (65 mg, 42%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.08 – 7.99 (m, 4 H), 7.64 – 7.50 (m, 2 H), 7.49 – 7.36 (m, 4 H), 5.00 (s, 1 H), 4.33 – 4.11 (m, 4 H), 3.80 – 3.71 (m, 1 H), 3.67 – 3.61 (m, 1 H), 1.91 – 1.75 (m, 4 H), 1.22 – 1.12 (m, 3 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  192.6, 167.7, 166.5, 134.1, 133.9, 132.8, 130.3, 129.5, 129.4, 128.6, 128.3, 83.9, 70.8, 64.5, 61.9, 26.2, 25.4, 14.0. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{22}\text{H}_{24}\text{NaO}_6$  407.1465, found 407.1474.

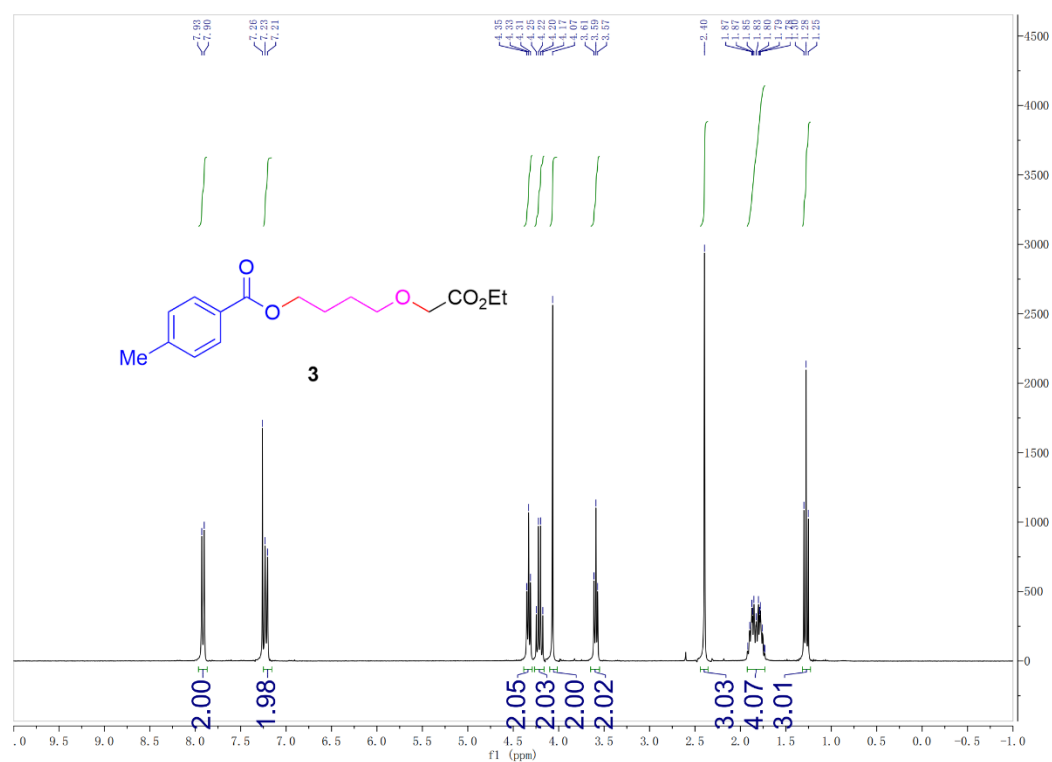


**4-((1-Ethoxy-1,3-dioxobutan-2-yl)oxy)butyl benzoate (73, new compound):** The reaction was performed following the general procedure. The residue was purified by flash column chromatography (10:1 PE/EA) to give the product as a colorless oil (49 mg, 38%);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03 (d,  $J = 7.5$  Hz, 2 H), 7.55 (t,  $J = 7.4$  Hz, 1 H), 7.43 (t,  $J = 7.5$  Hz, 2 H), 4.38 – 4.33 (m, 3 H), 4.31 – 4.20 (m, 2 H), 3.75 – 3.48 (m, 2 H), 2.26 (s, 3 H), 1.98 – 1.76 (m, 4 H), 1.28 (t,  $J = 7.1$  Hz, 1 H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  201.9, 167.0, 166.3, 132.7, 130.1, 129.3, 128.2, 85.5, 70.5, 64.3, 61.7, 26.1, 26.0, 25.3, 13.9. HRMS (ESI)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $\text{C}_{17}\text{H}_{22}\text{NaO}_6$  345.1309, found 345.1299.

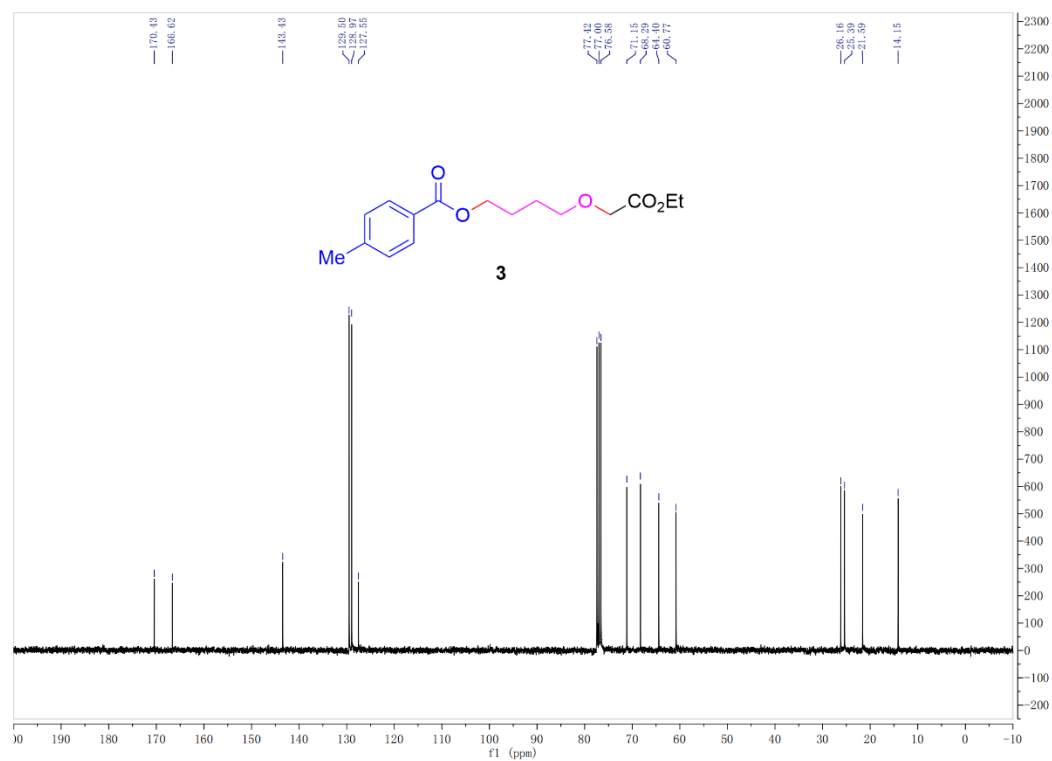
## References

- [1] Y. Liu; K. Zhu, J. Zhao and P. Li, *Org. Lett.*, 2022, **24**, 6834–6838.
- [2] K. Zhu, M. Cao, G. Zhao, J. Zhao and P. Li, *Org. Lett.*, 2022, **24**, 5855–5859.

**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 3**

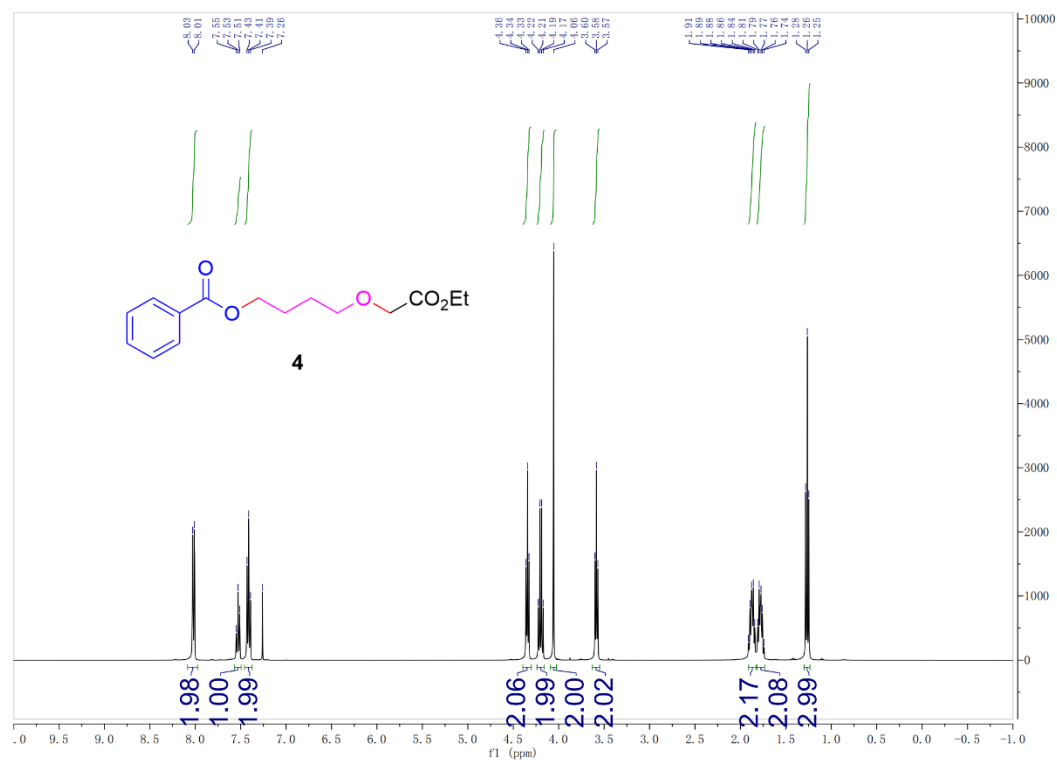


**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 3**

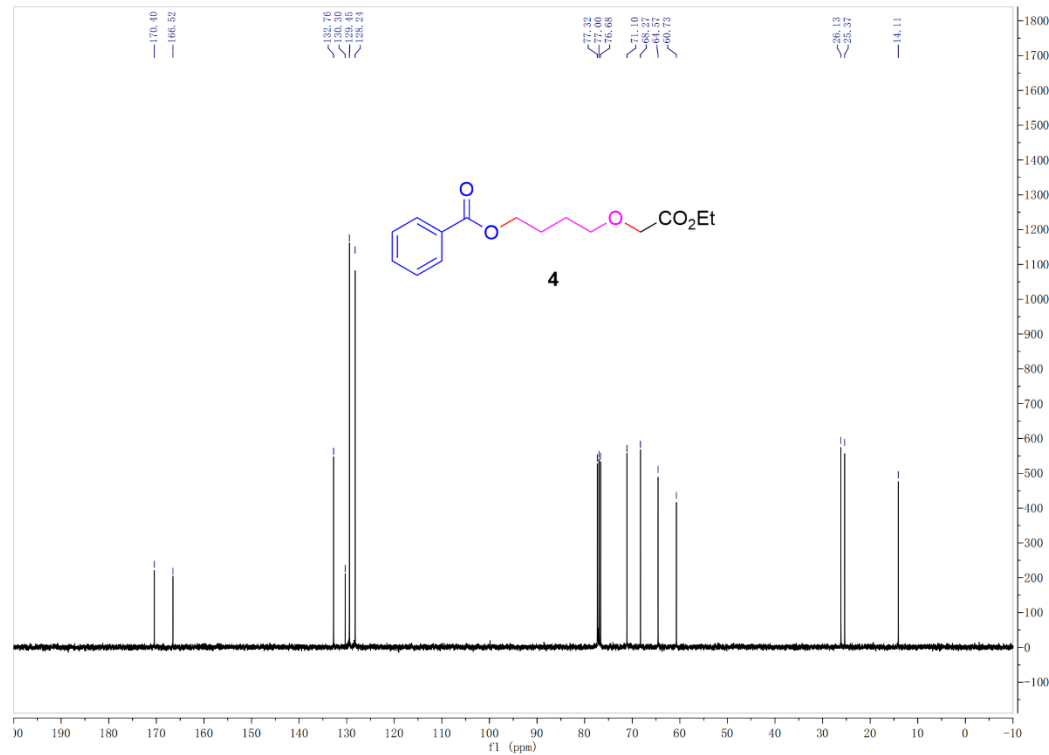




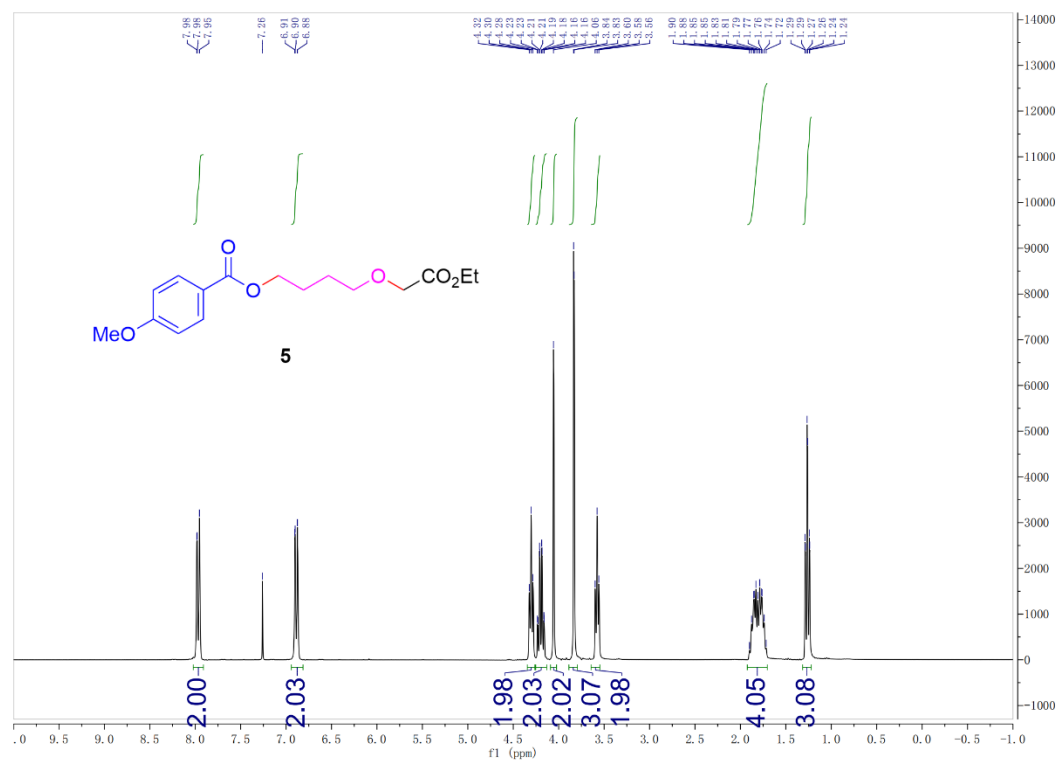
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 4**



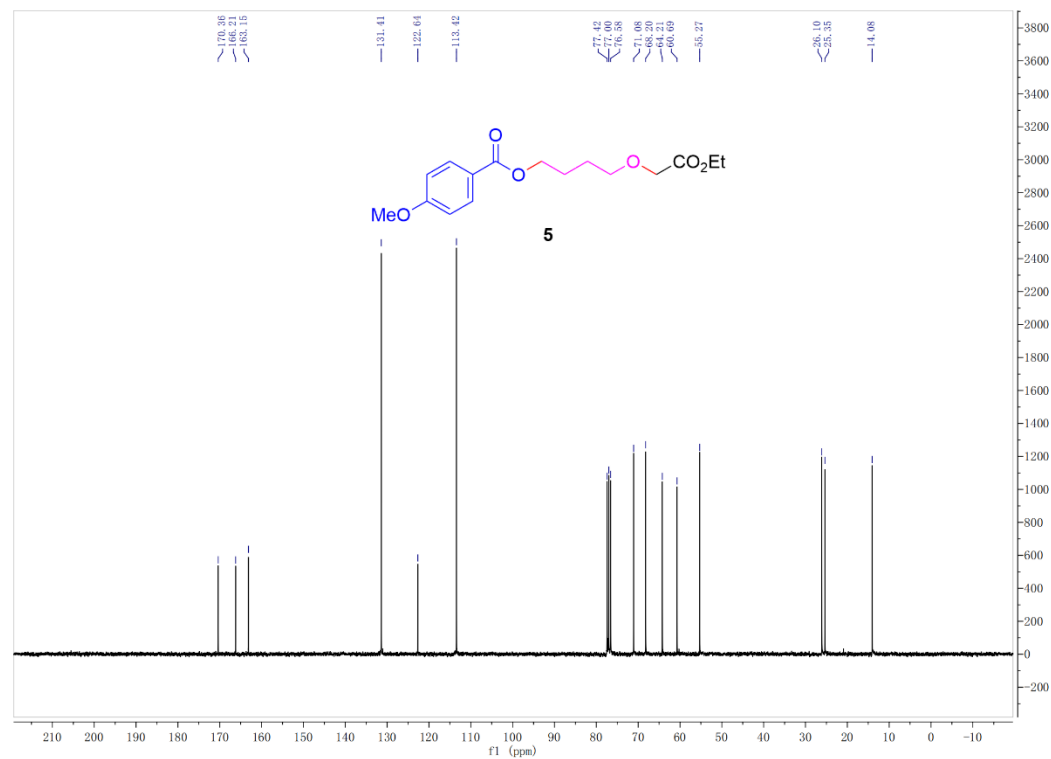
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 4**



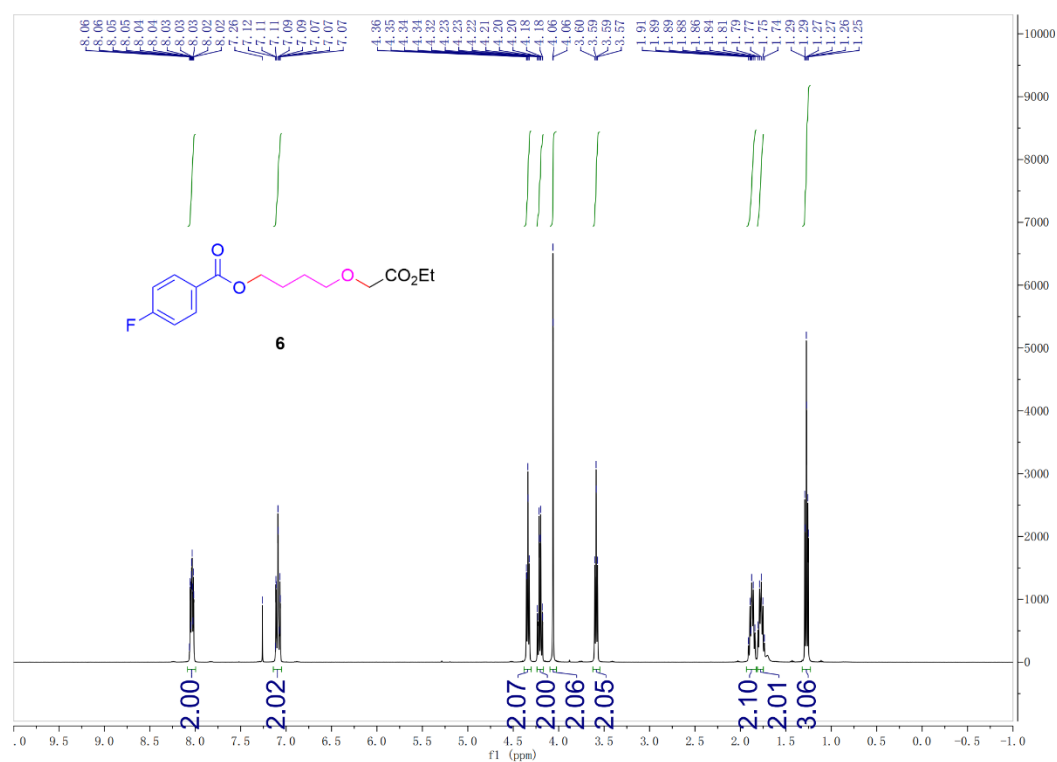
**<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) Spectrum of 5**



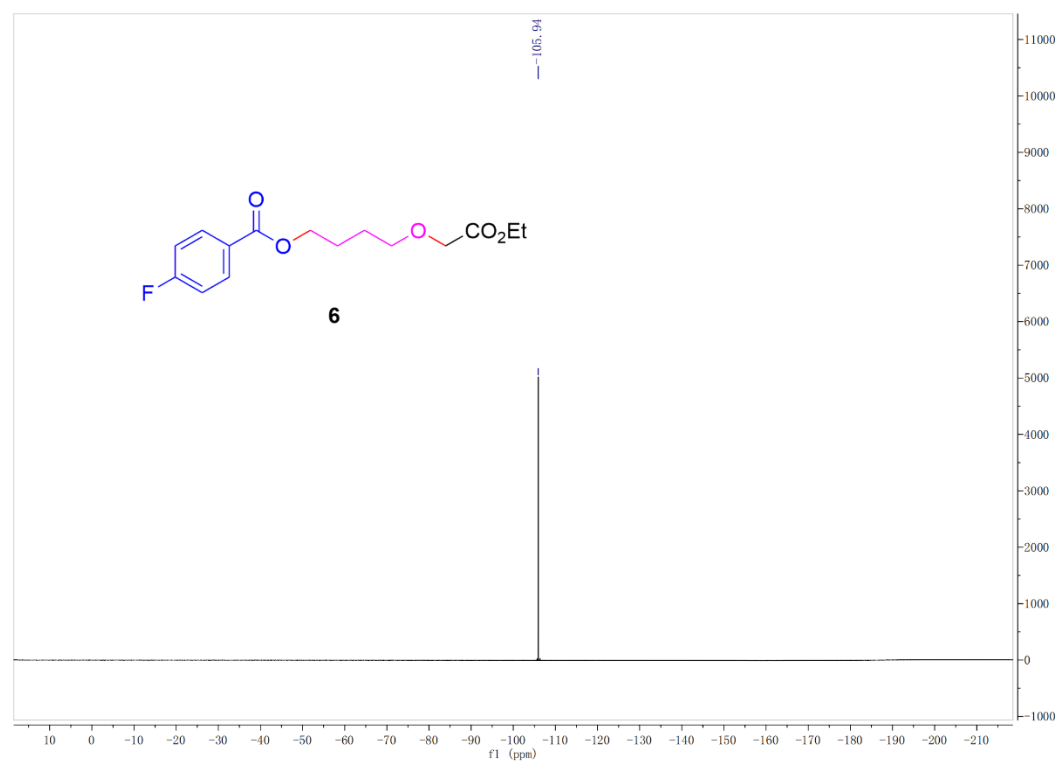
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 5**



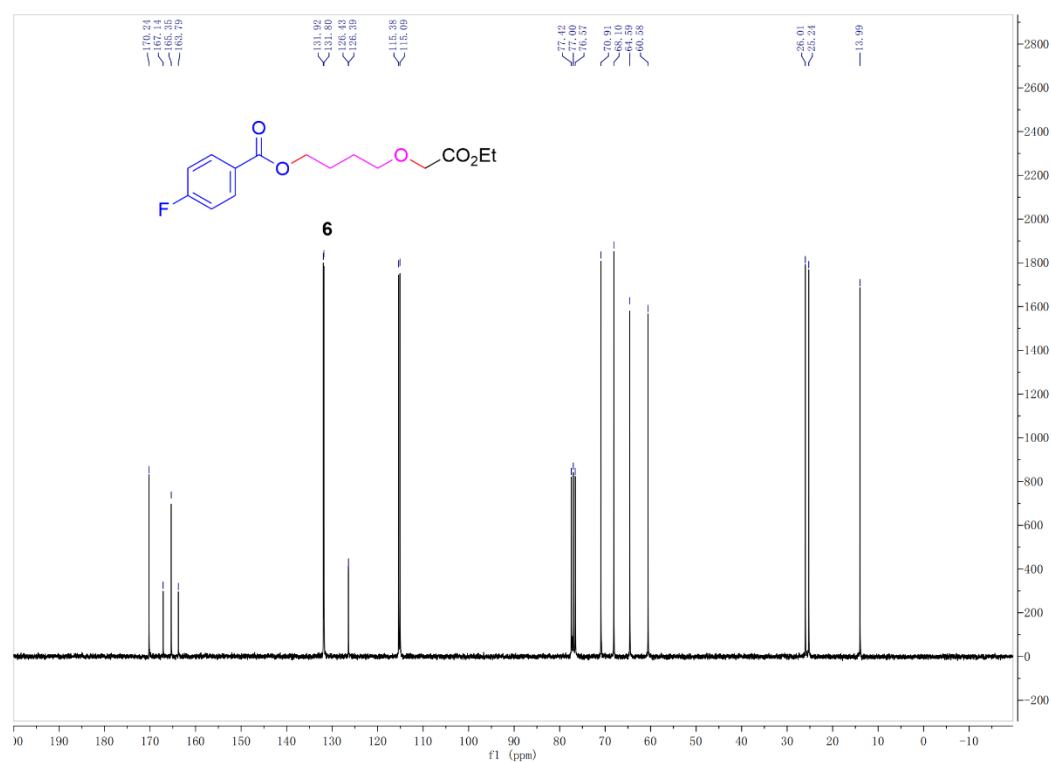
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 6**



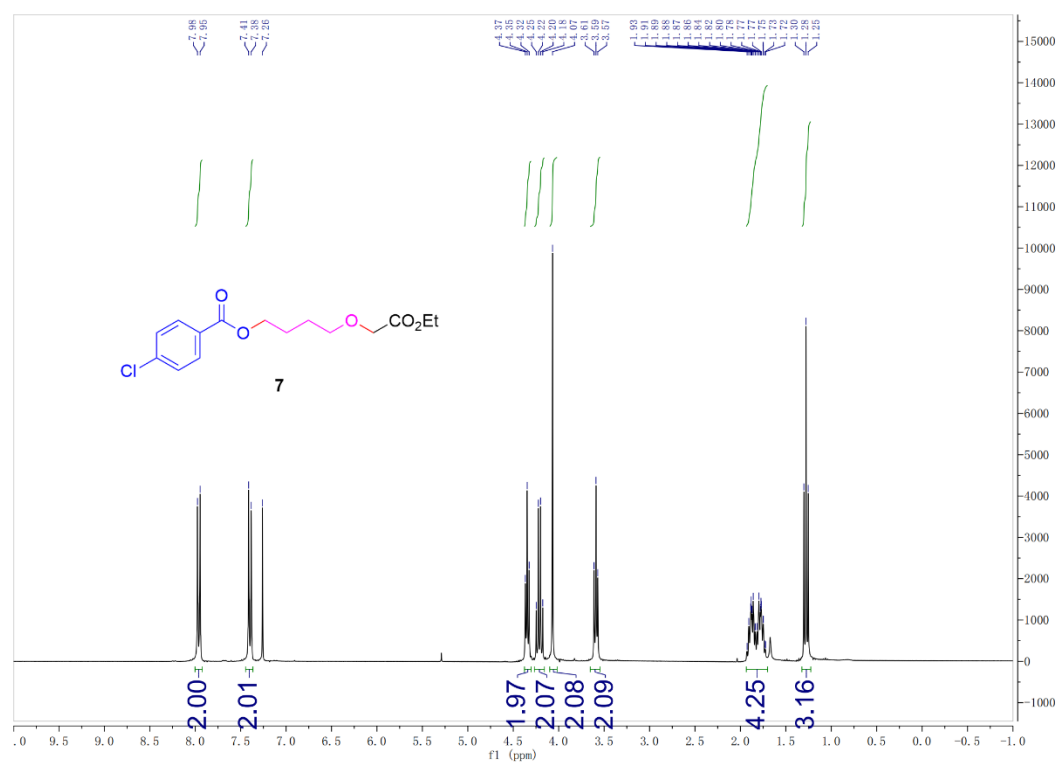
**<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) Spectrum of 6**



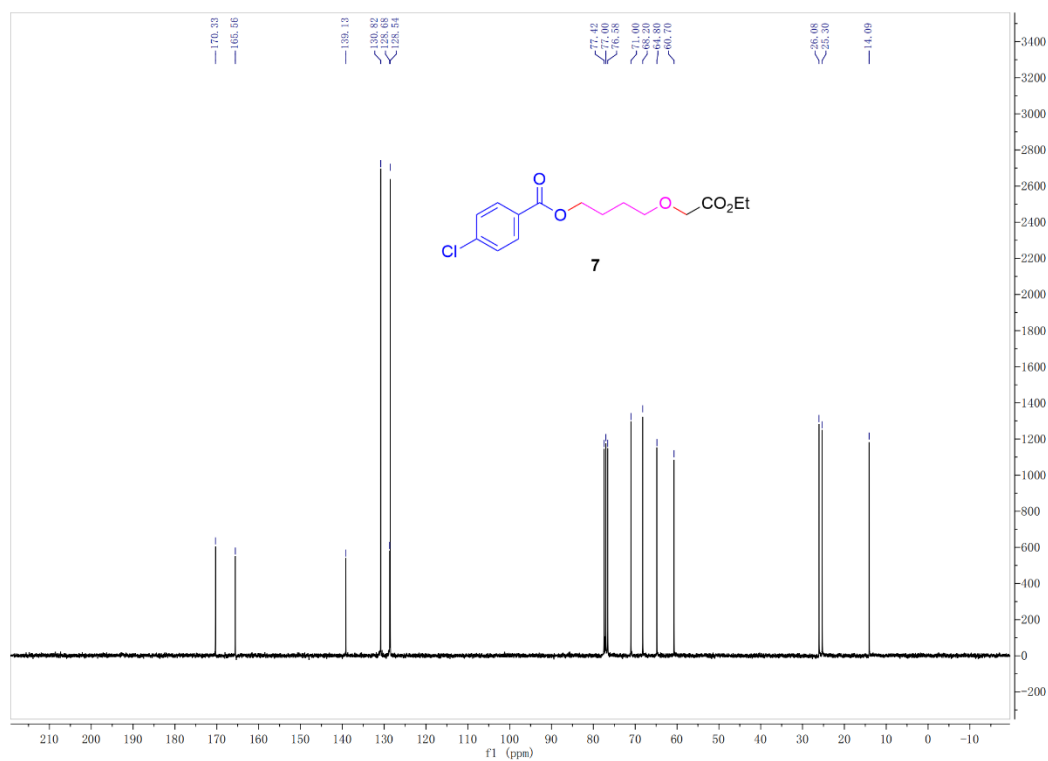
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 6**



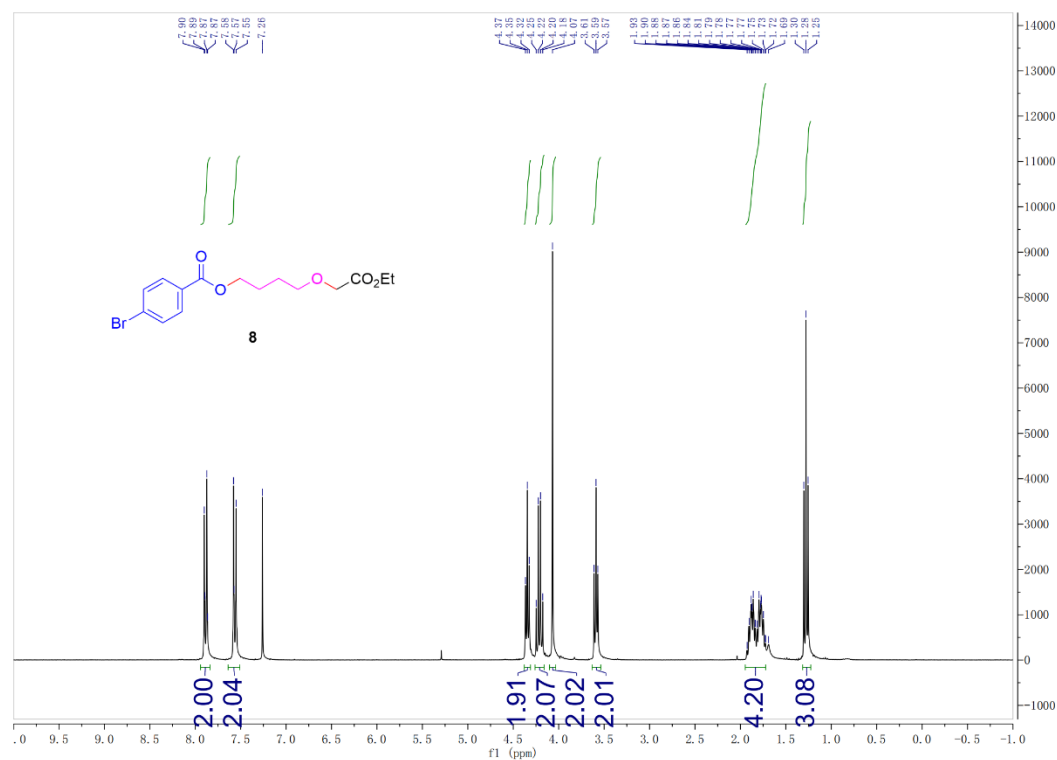
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 7**



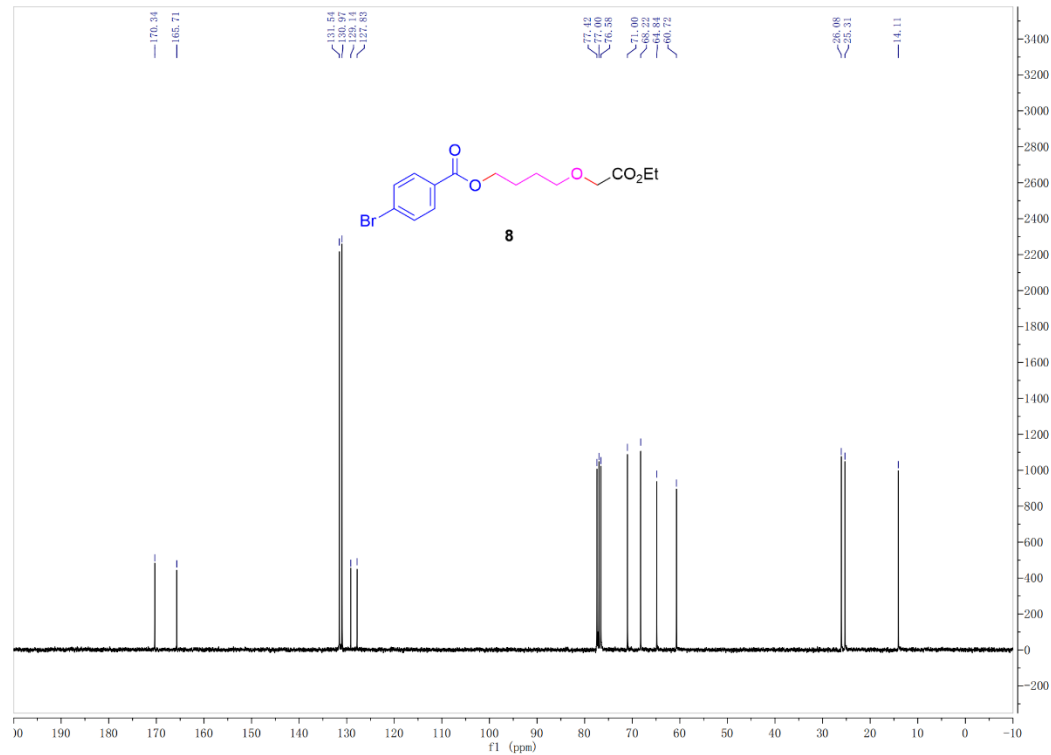
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 7**



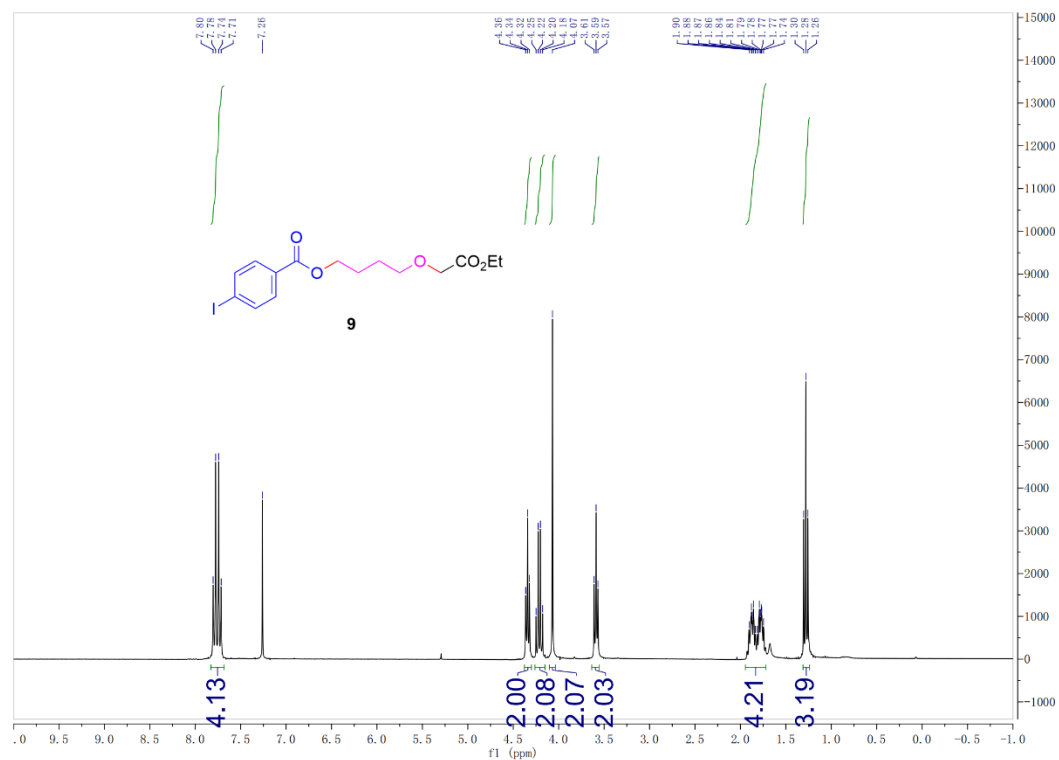
**<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) Spectrum of 8**



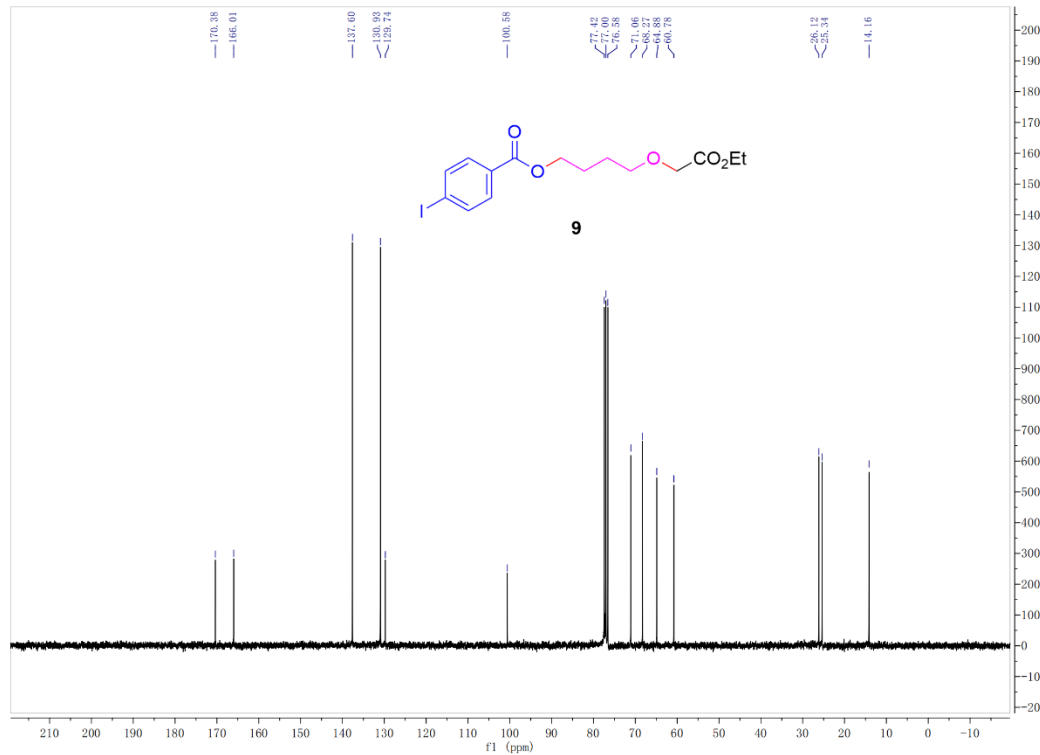
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 8**



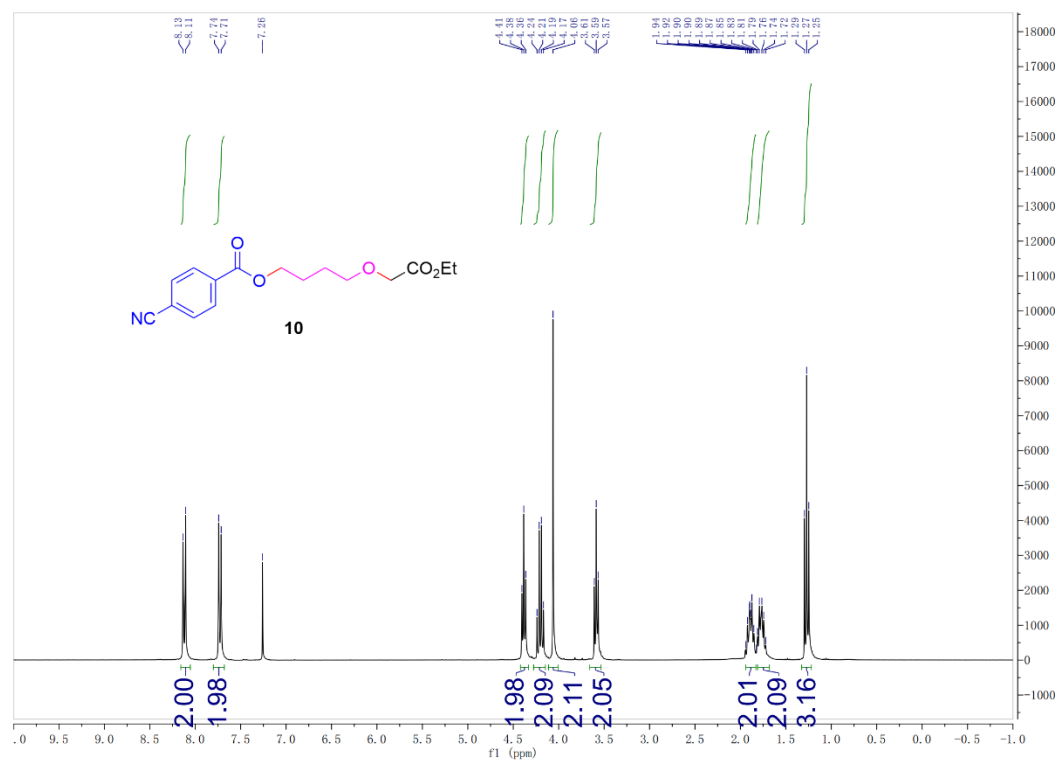
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 9**



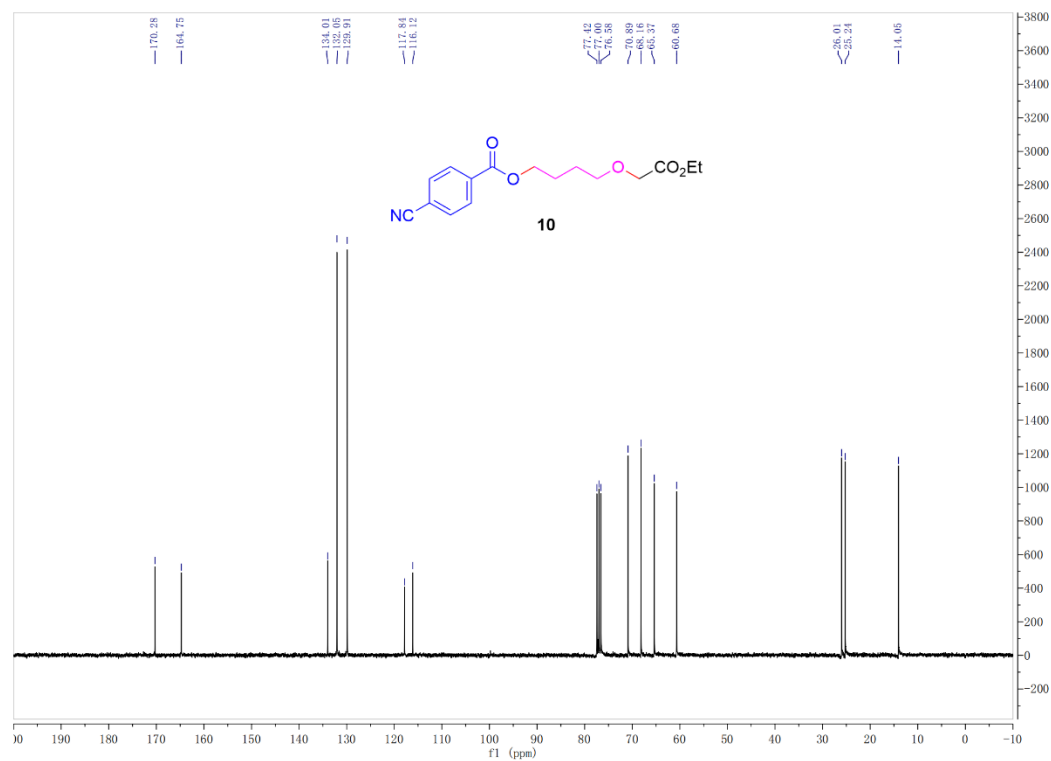
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 9**



**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 10**

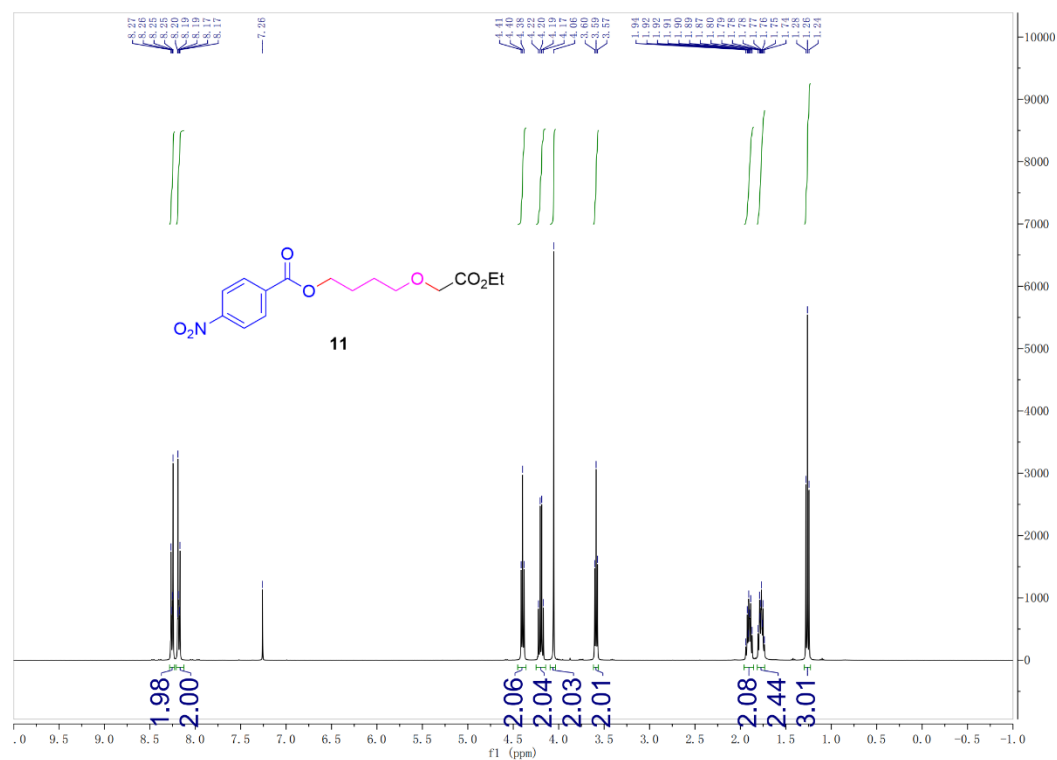


**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 10**

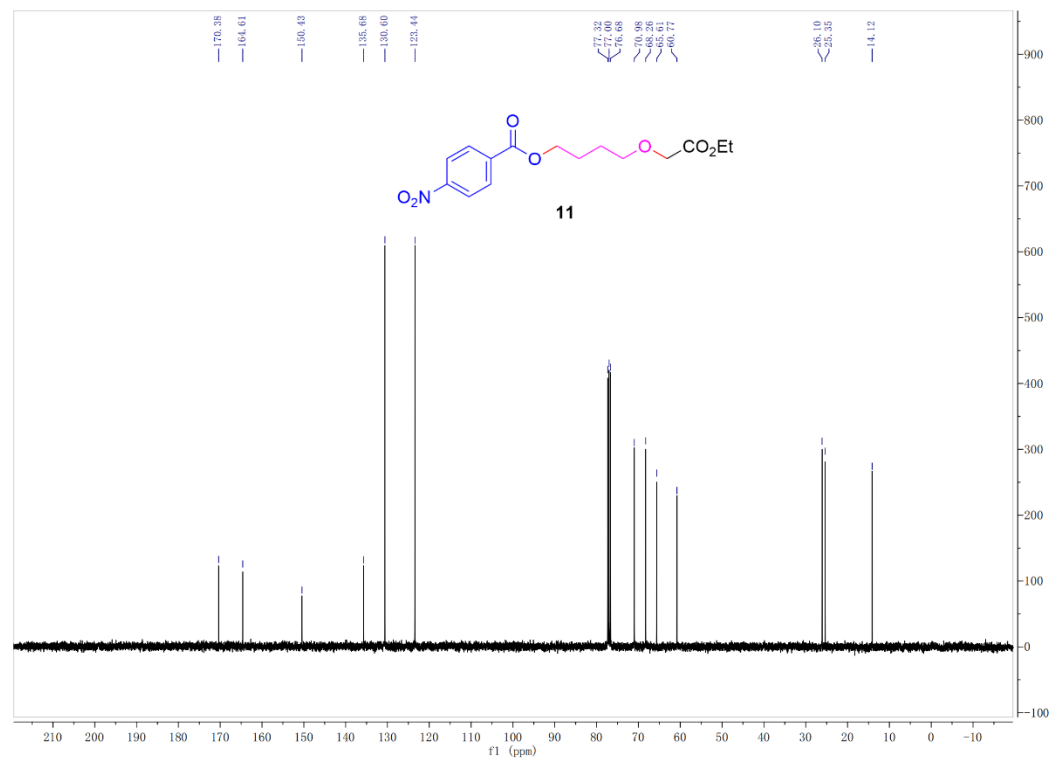




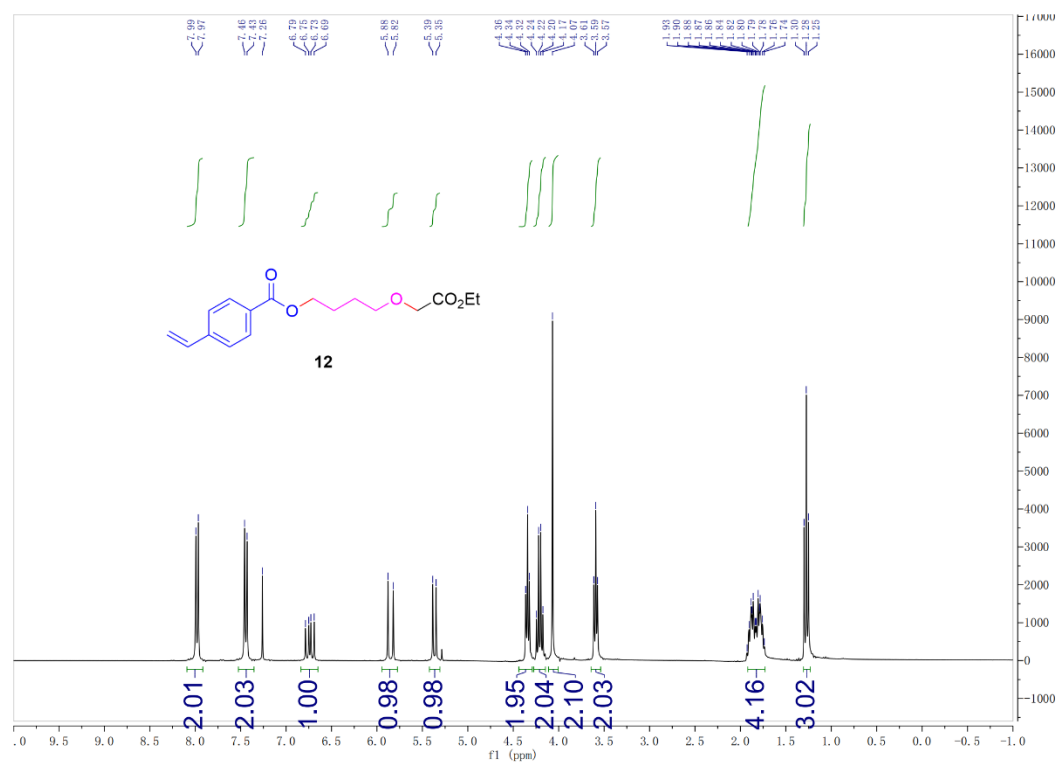
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 11**



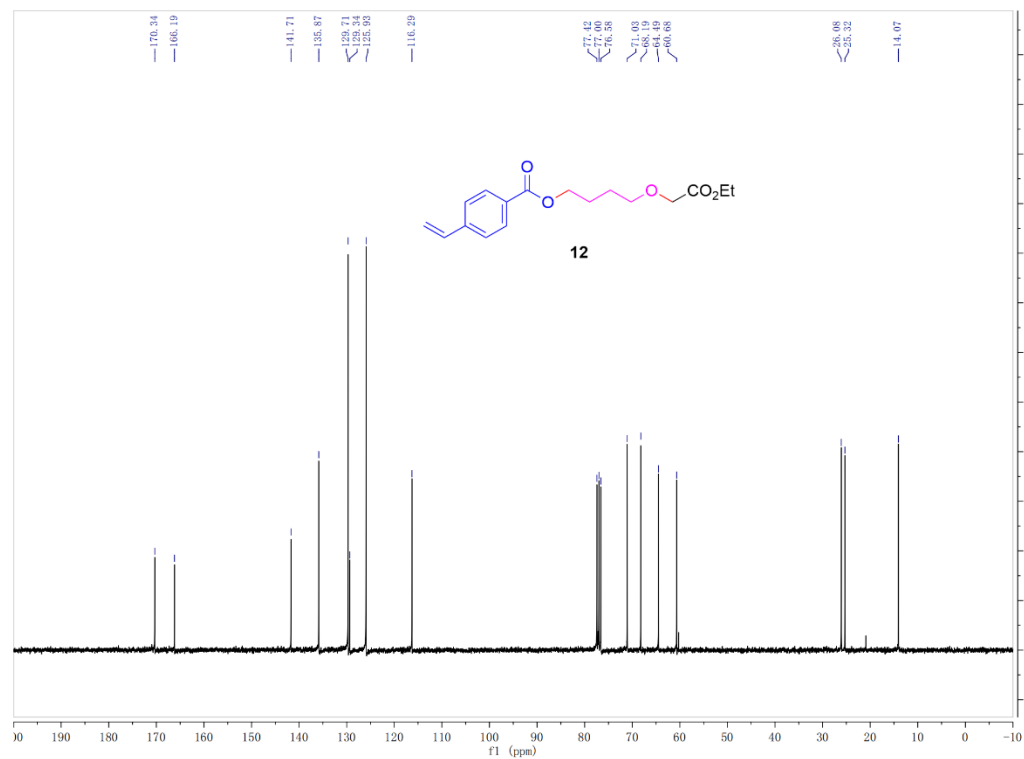
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 11**



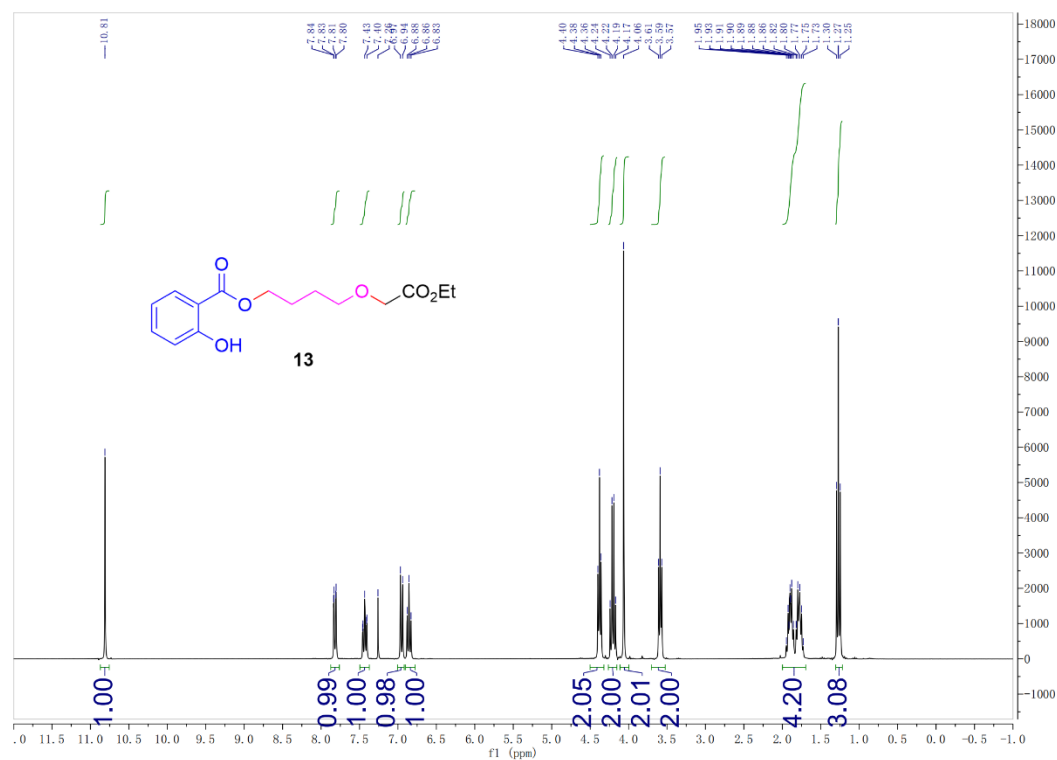
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 12**



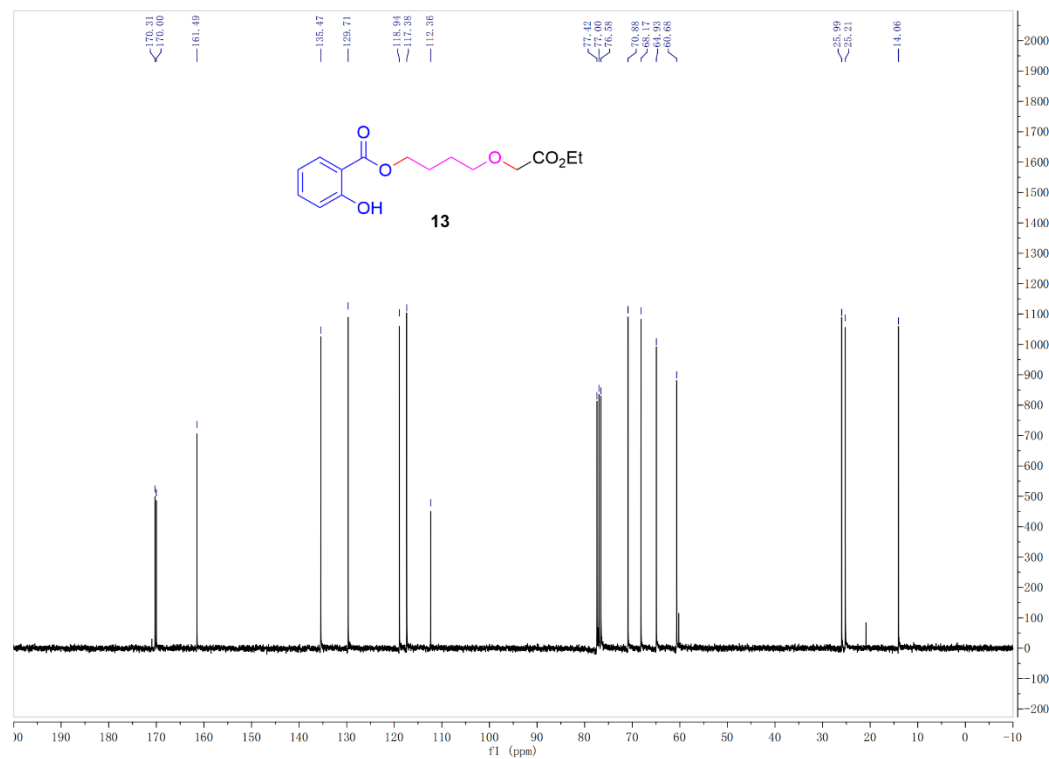
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 12**



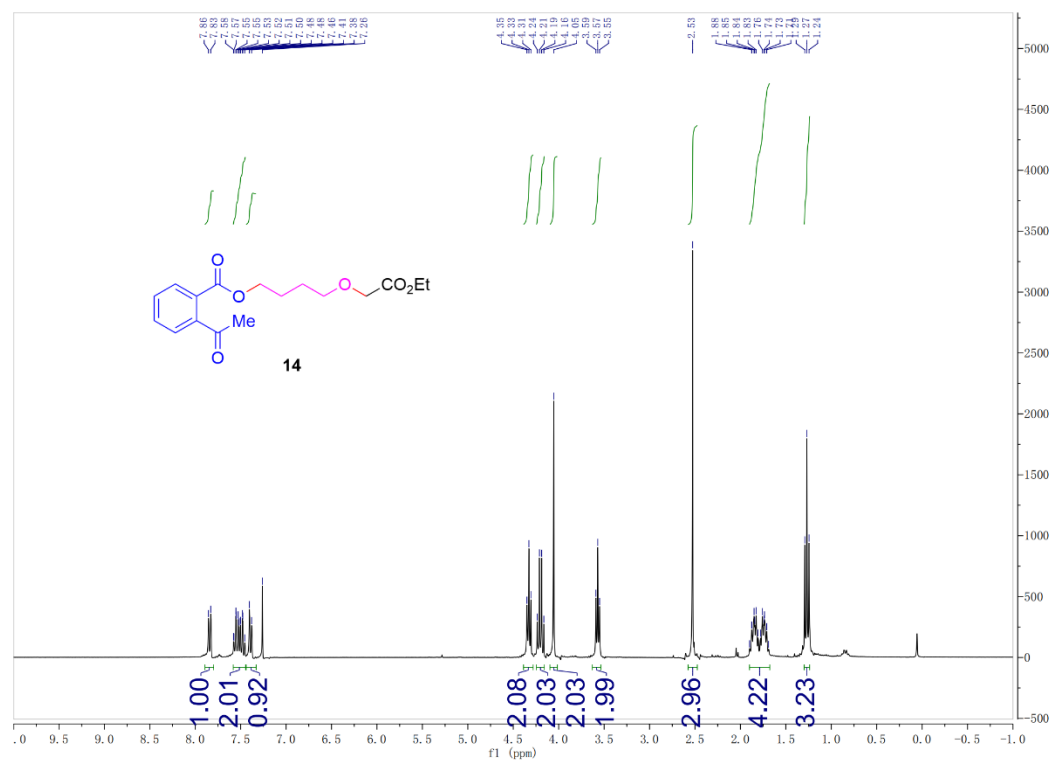
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 13**



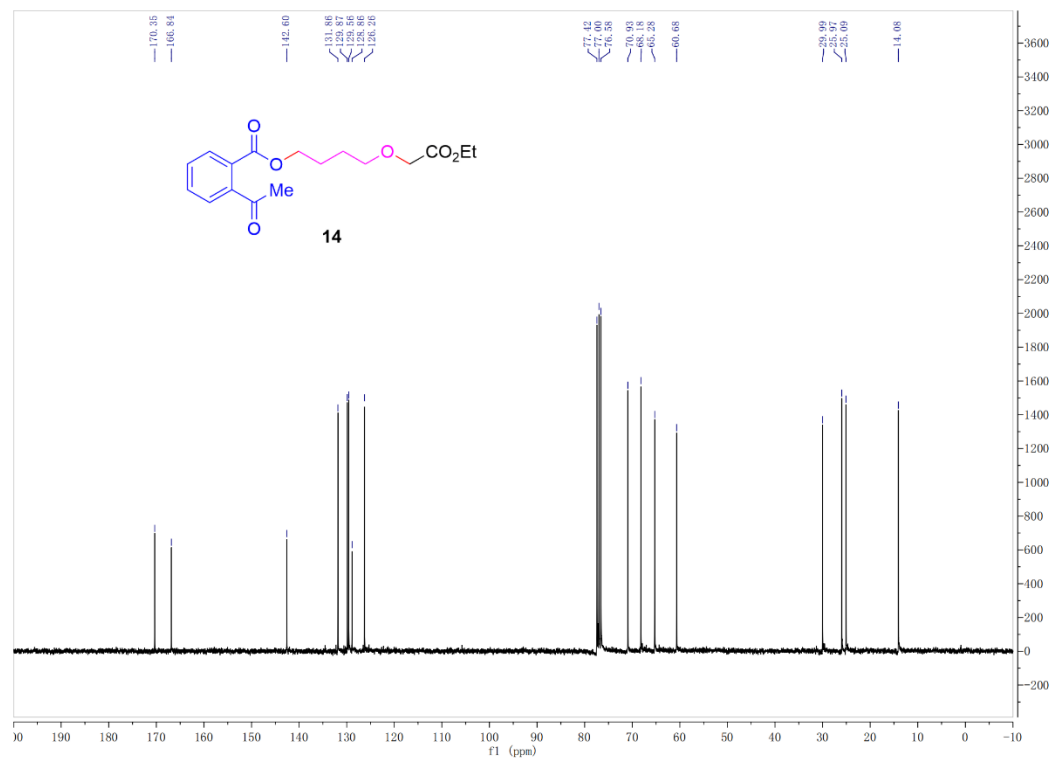
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 13**



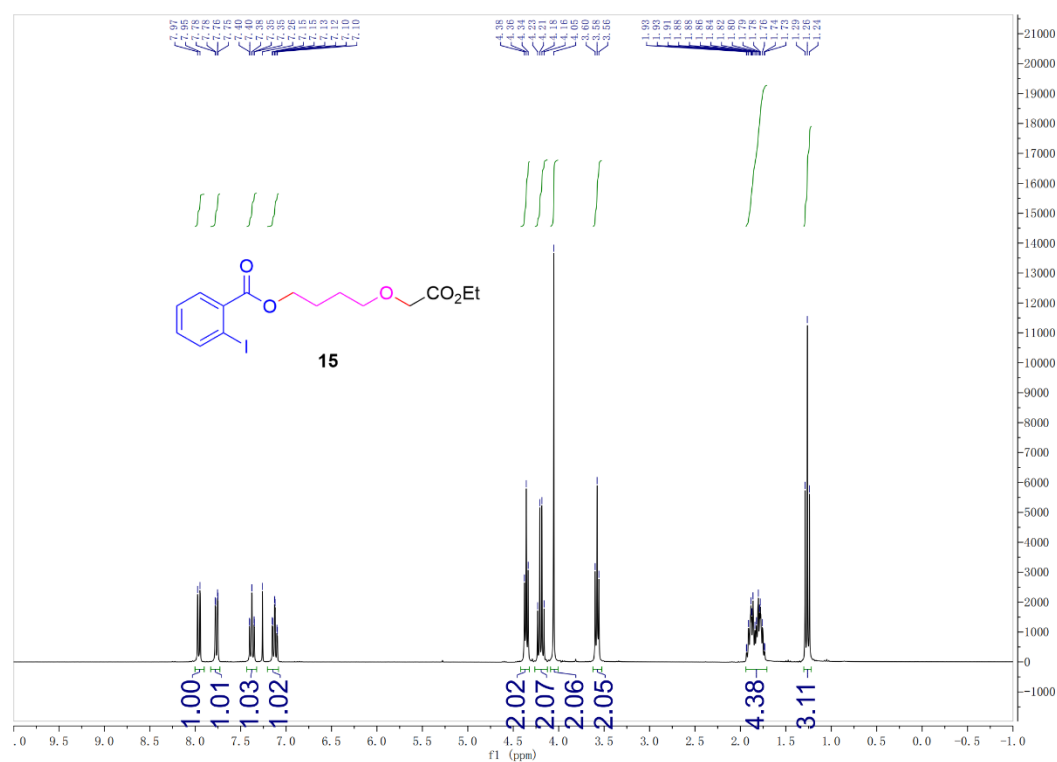
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 14**



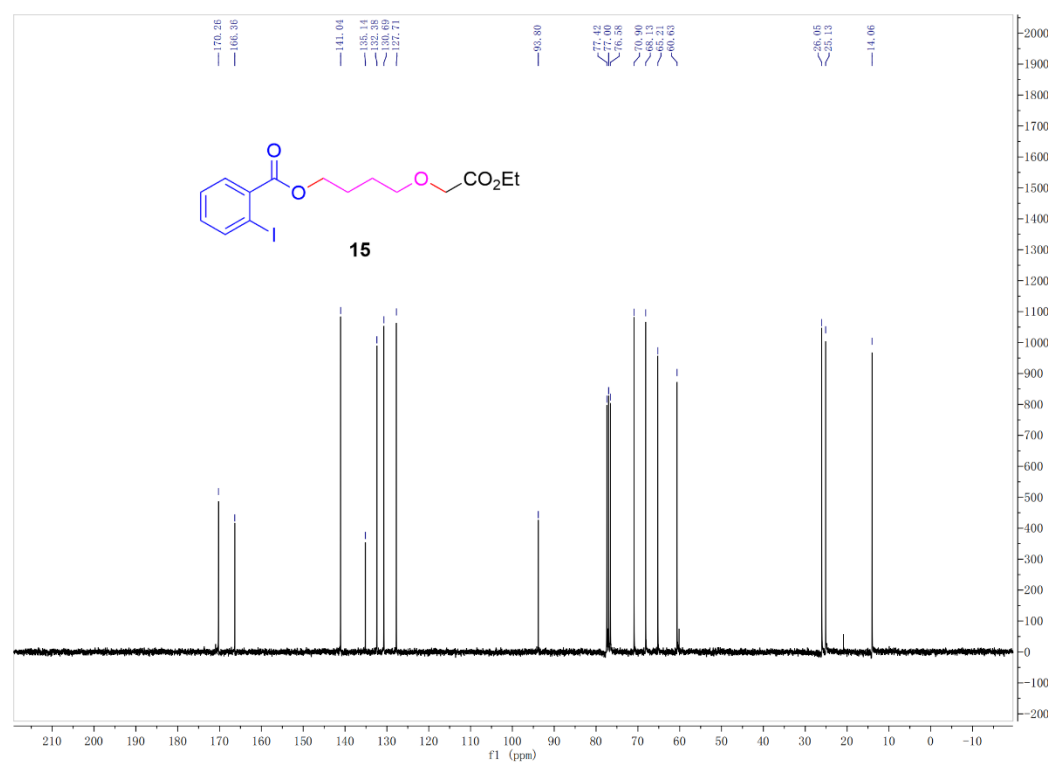
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 14**



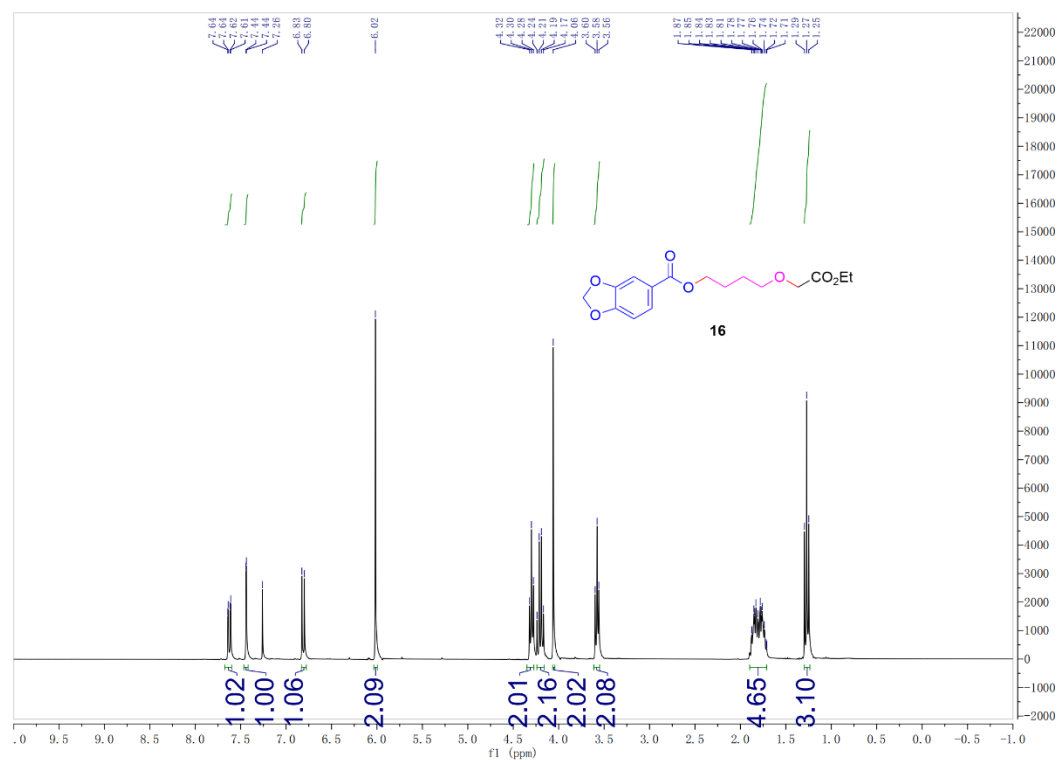
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 15**



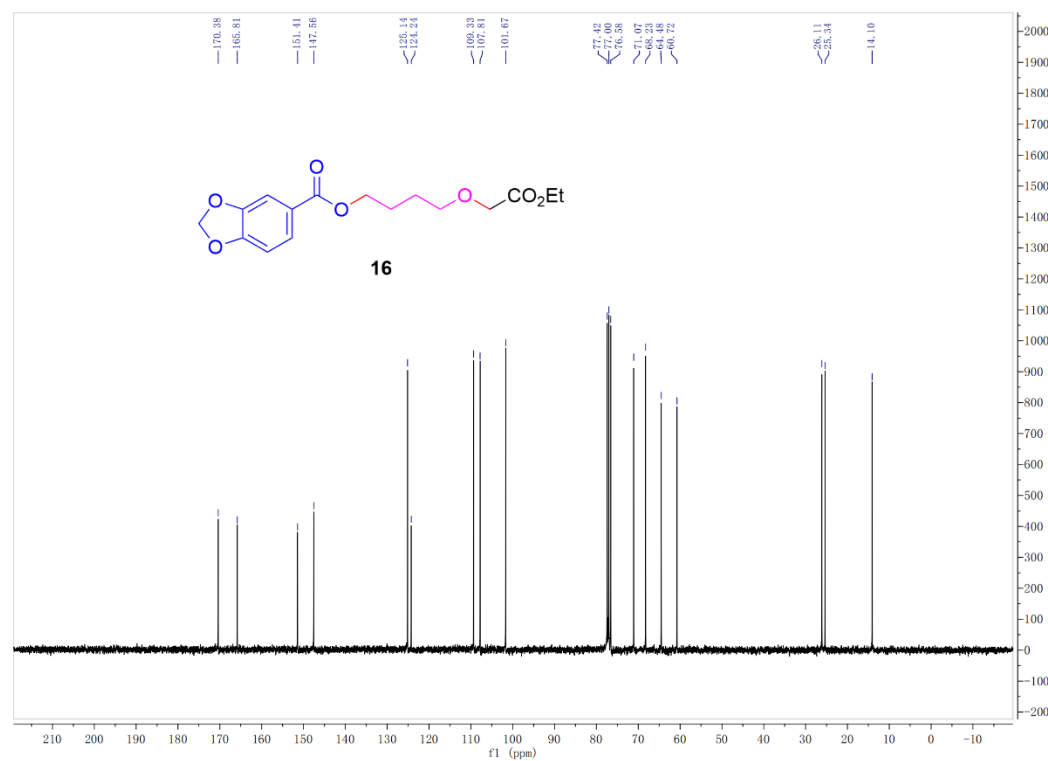
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 15**



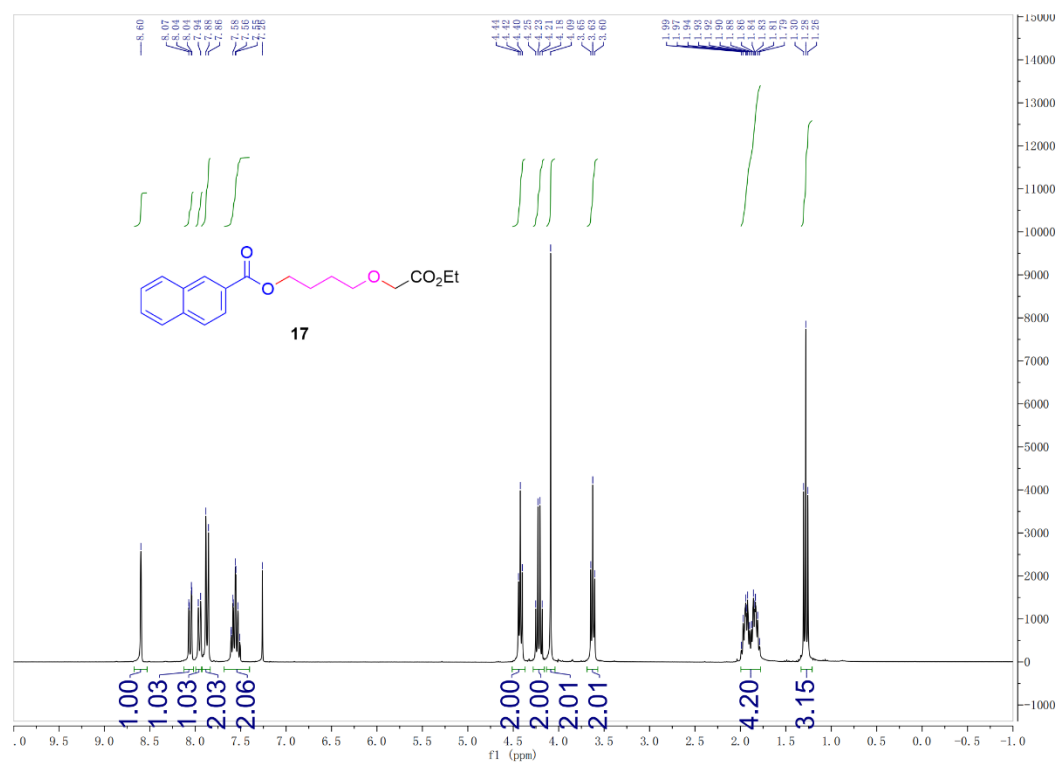
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 16**



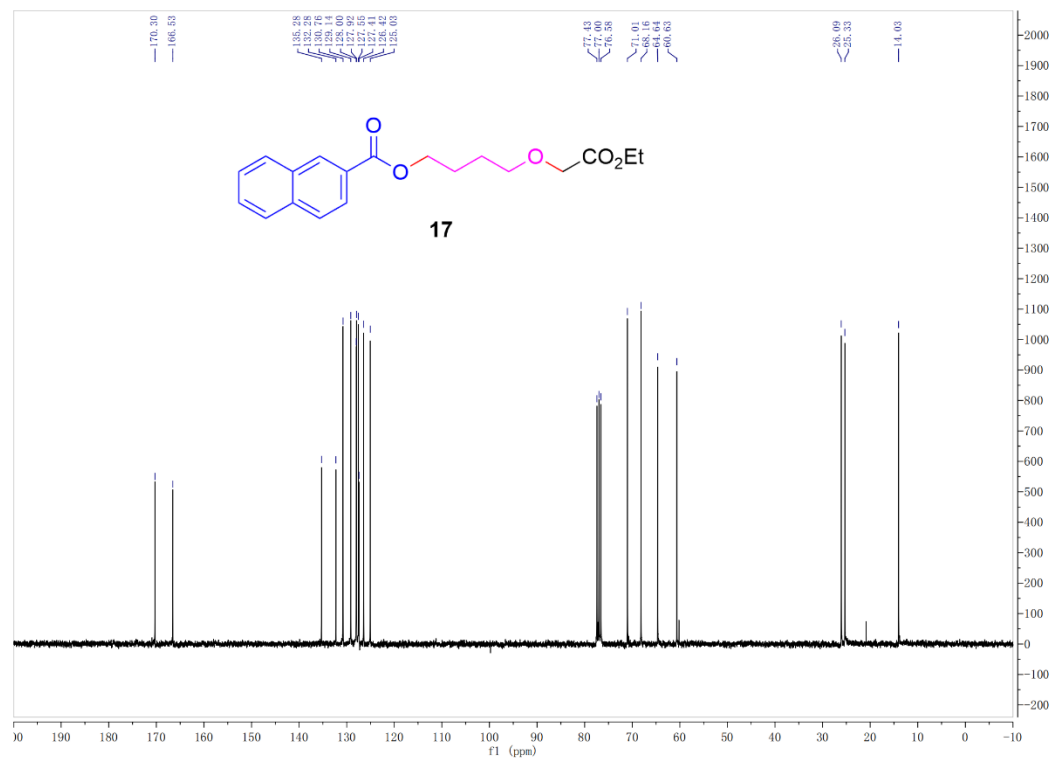
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 16**



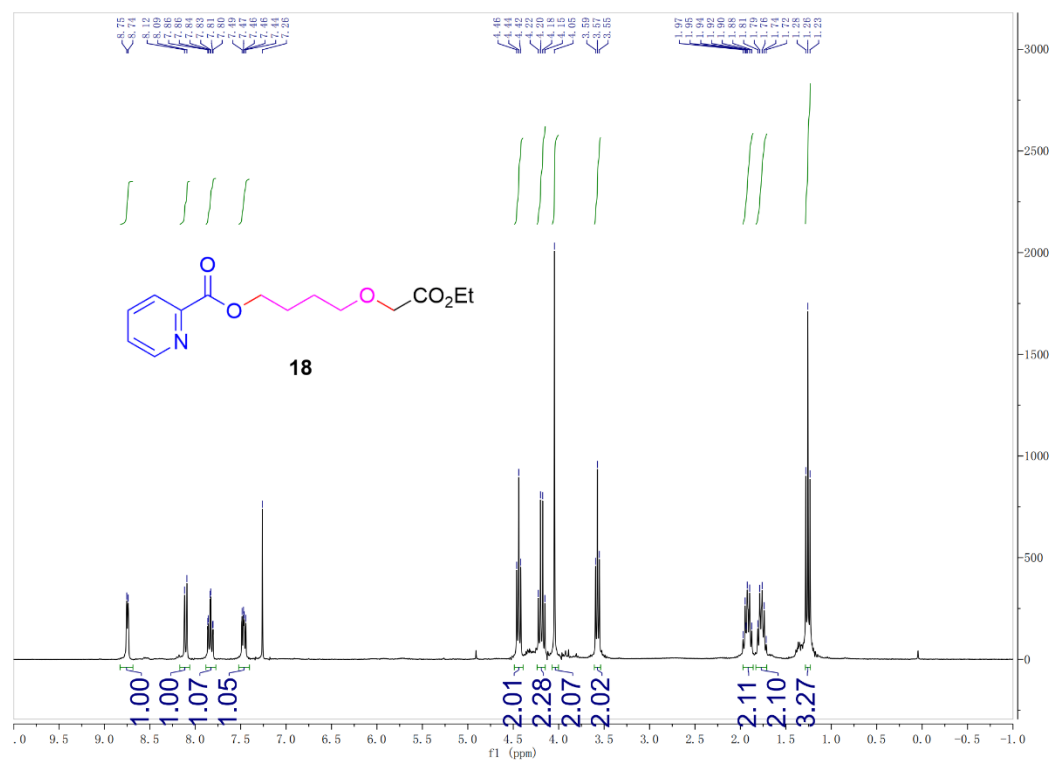
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 17**



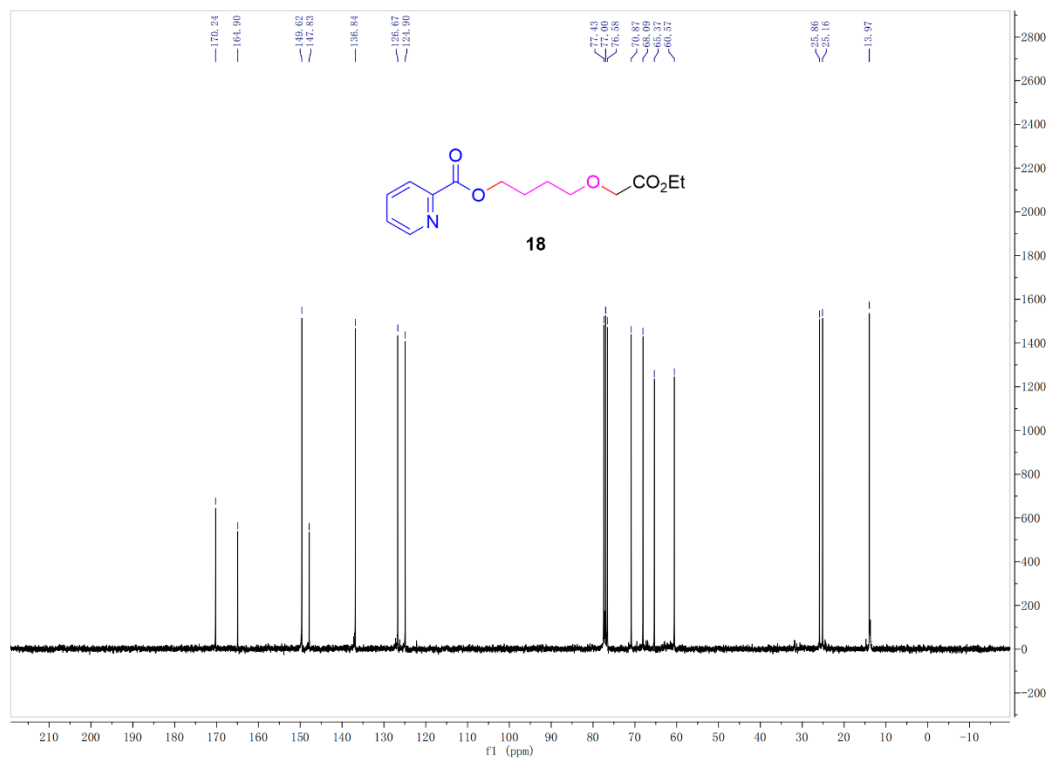
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 17**



**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 18**

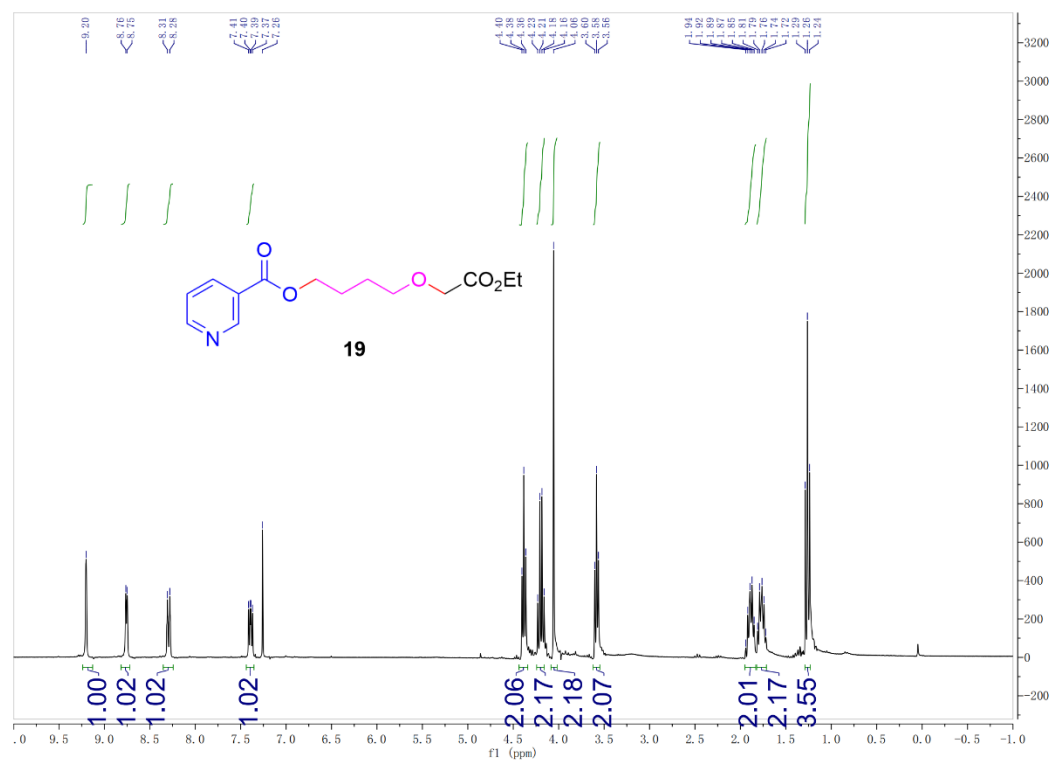


**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 18**

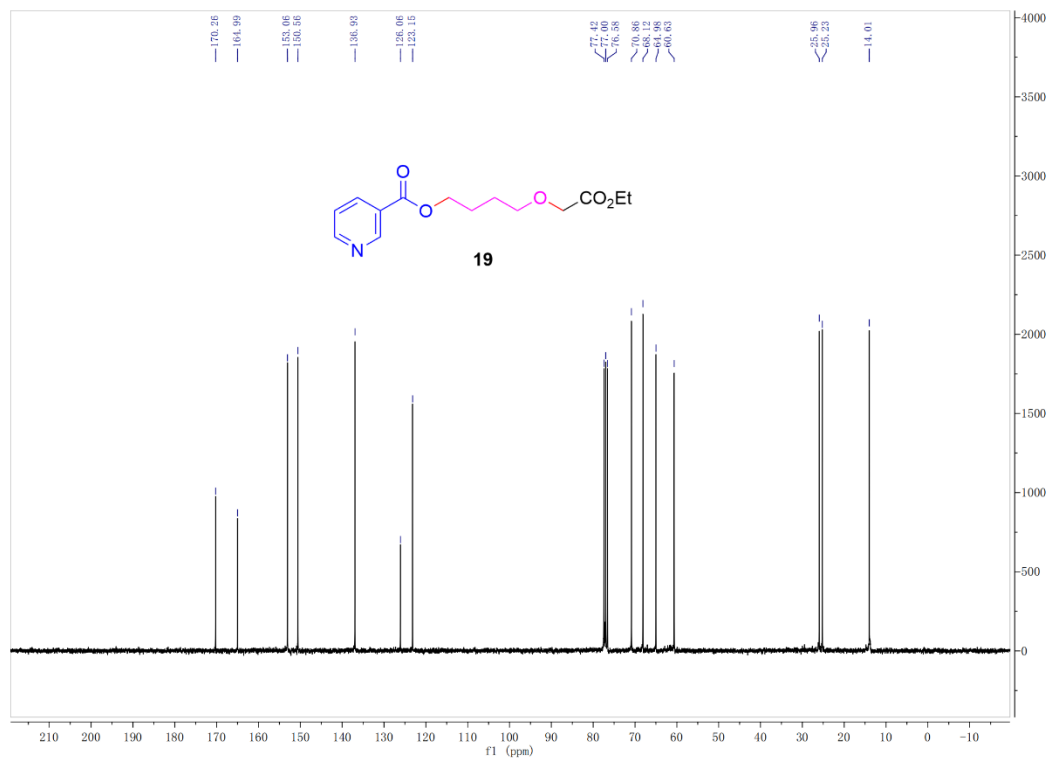




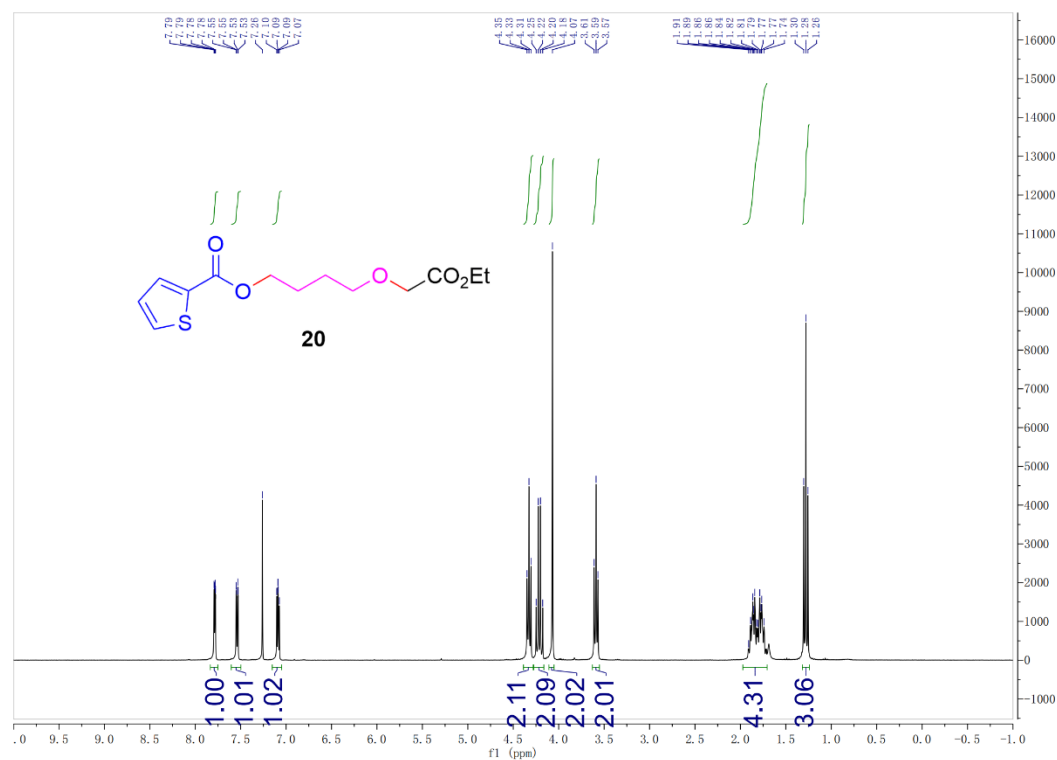
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 19**



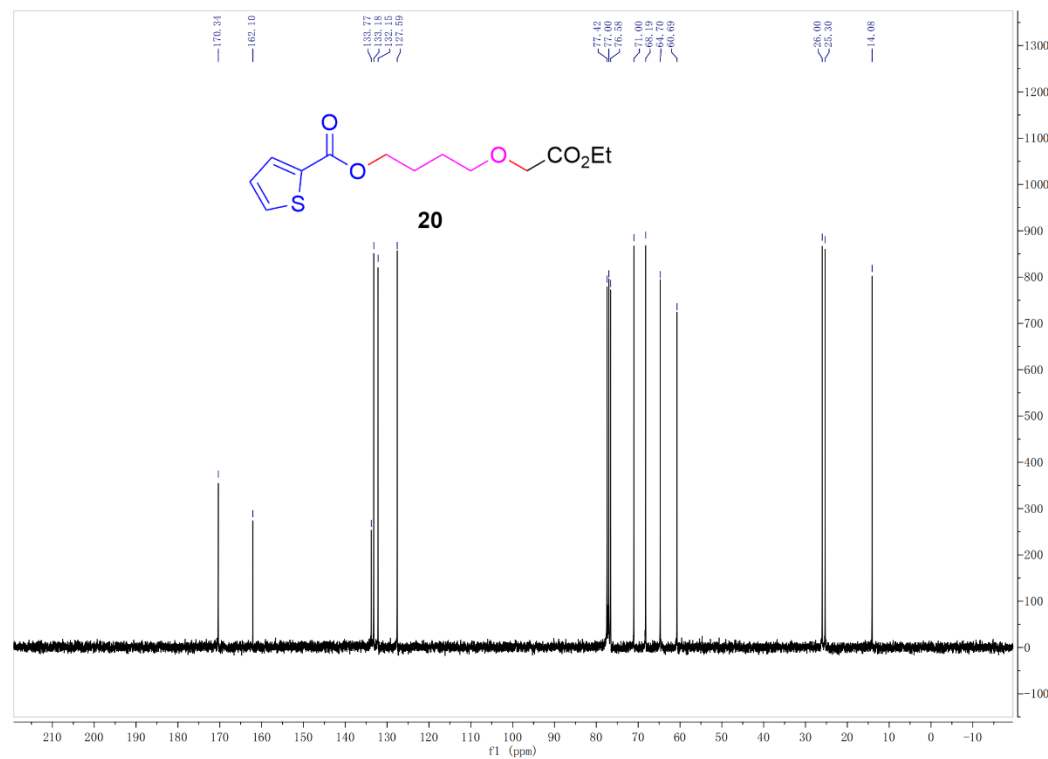
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 19**



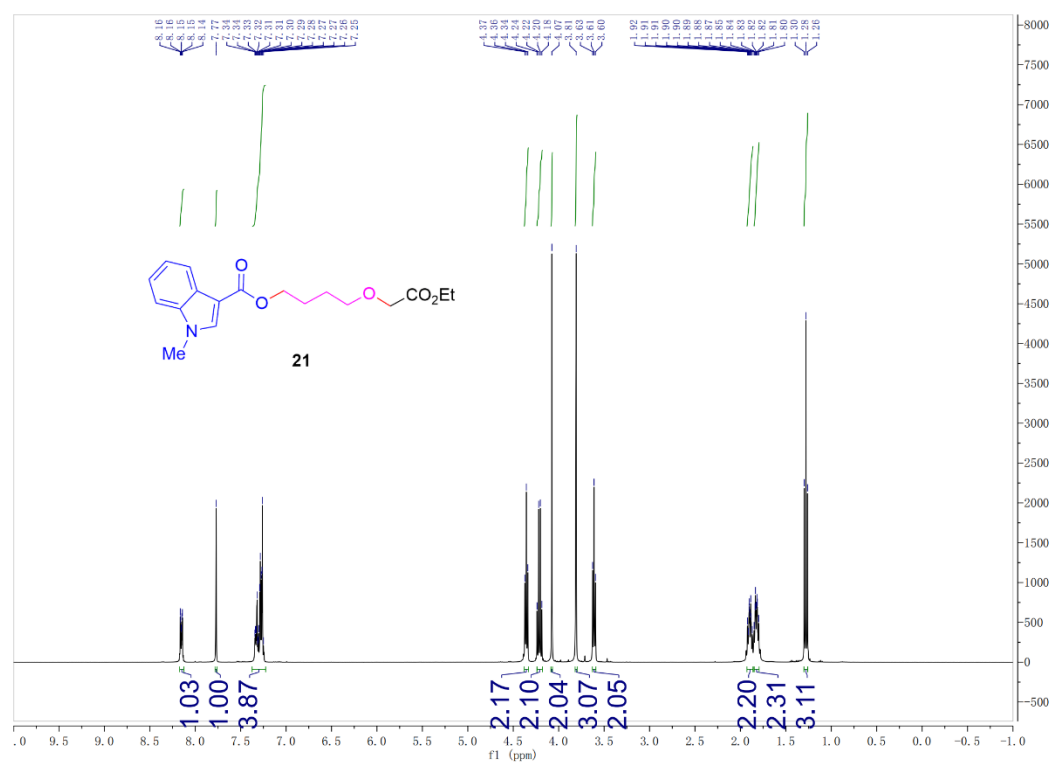
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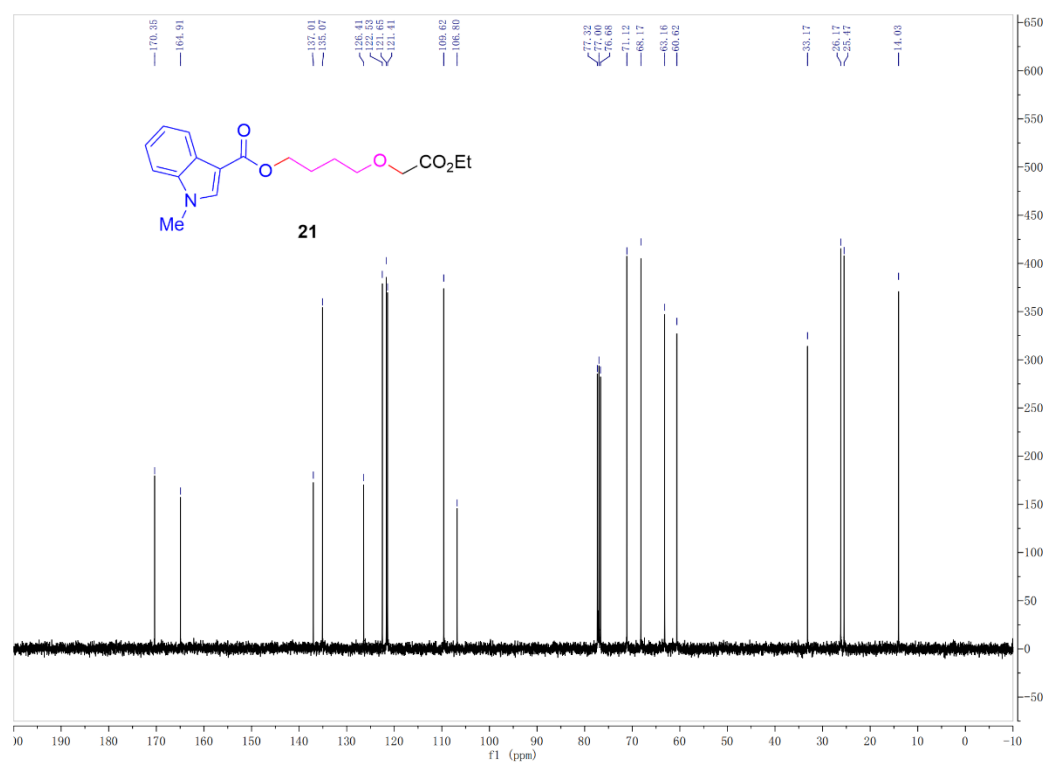
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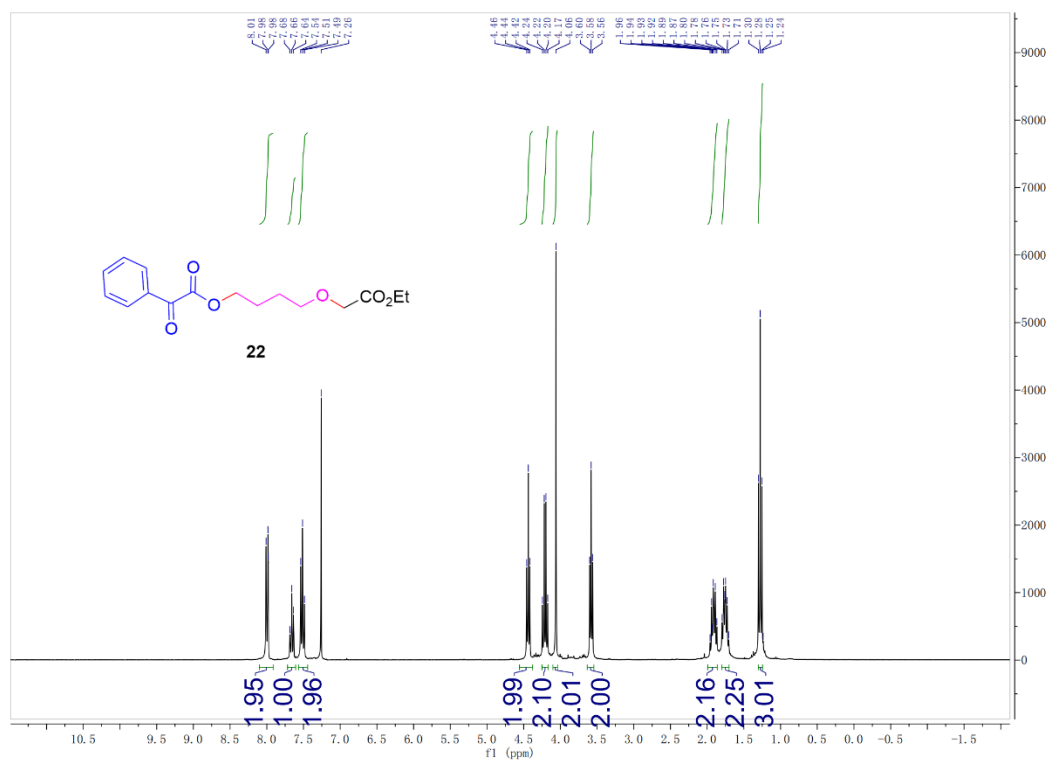
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 21**



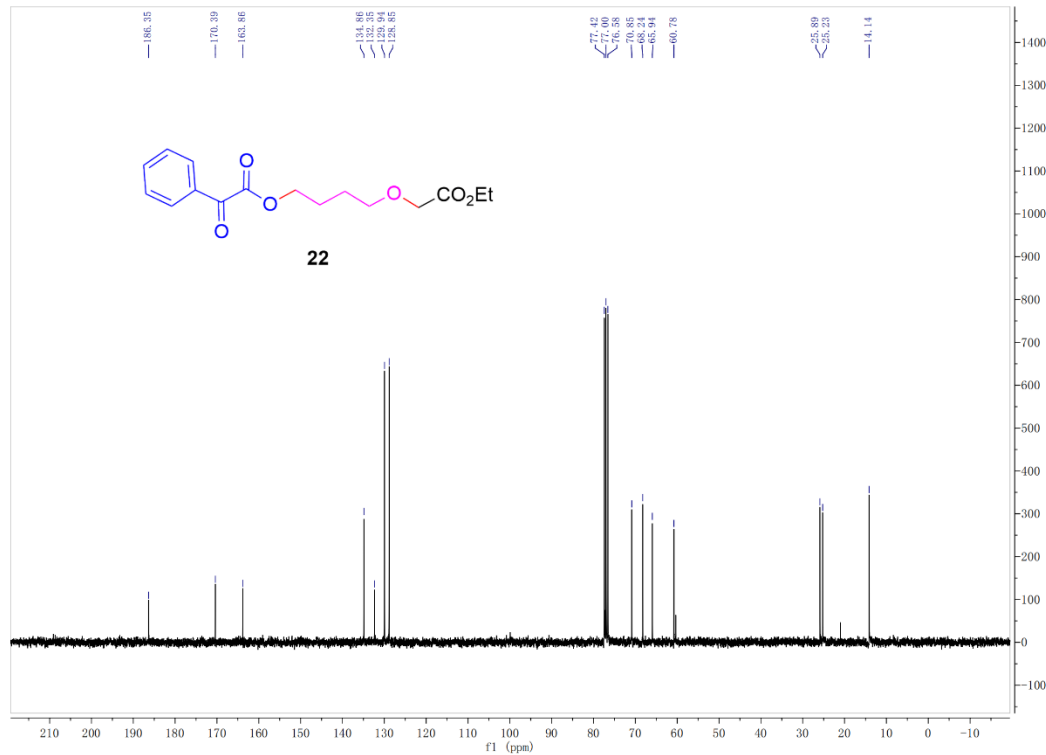
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 21**



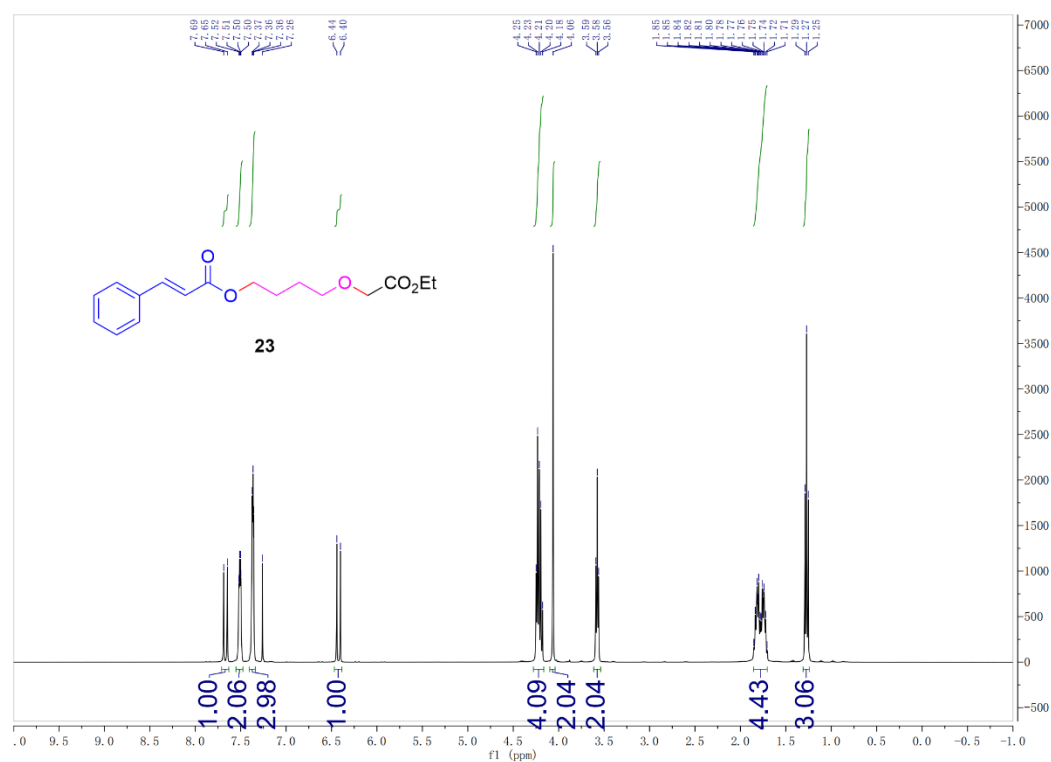
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 22**



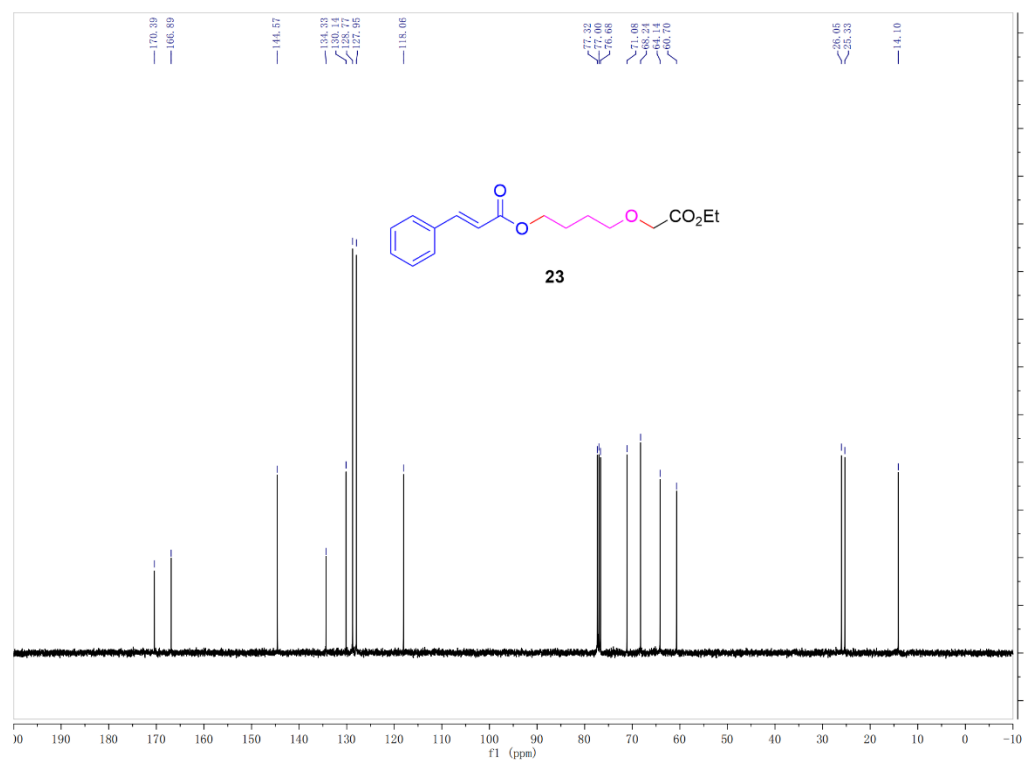
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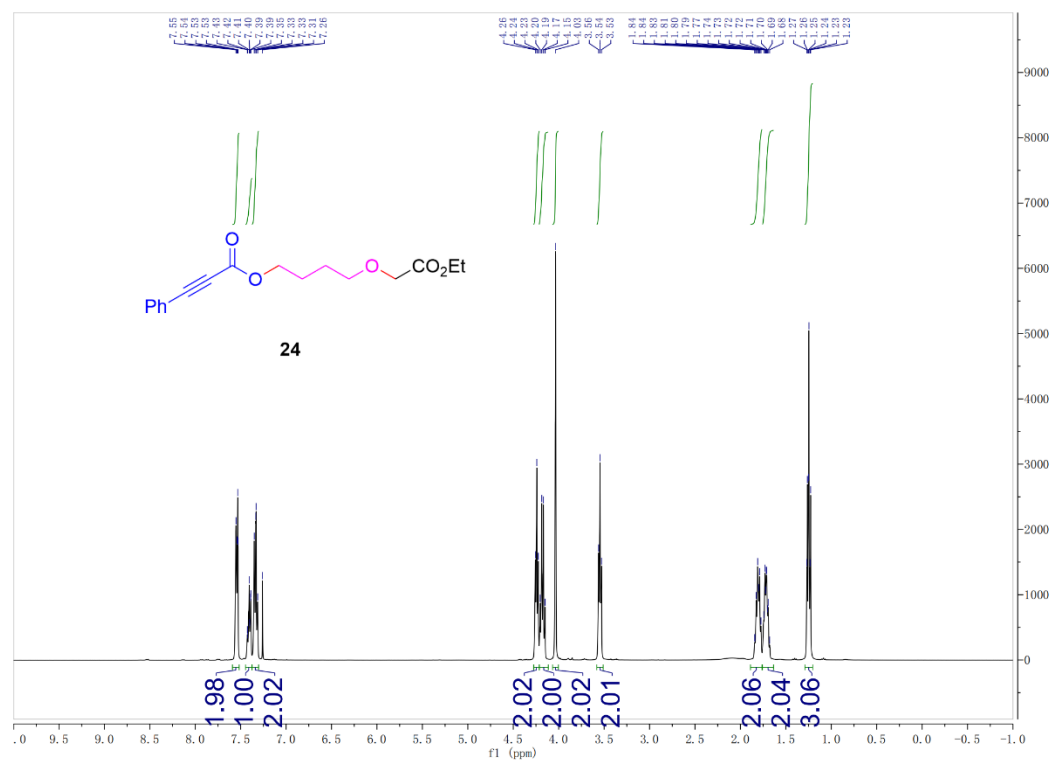
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 23**



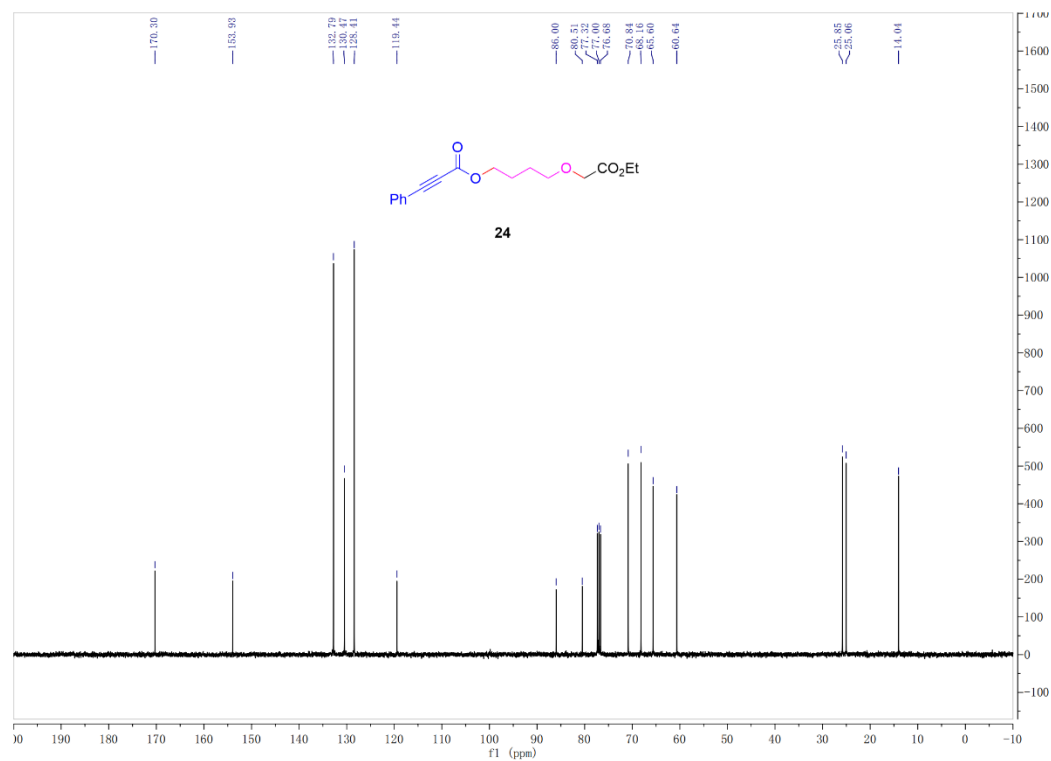
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 23**



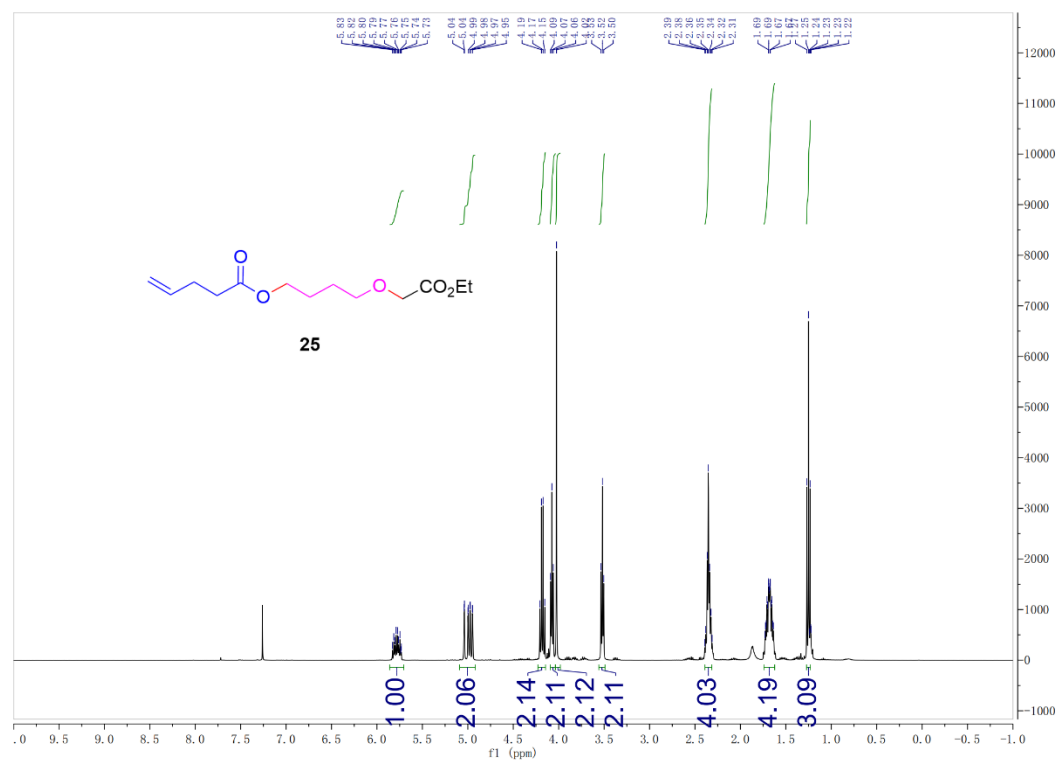
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 24**



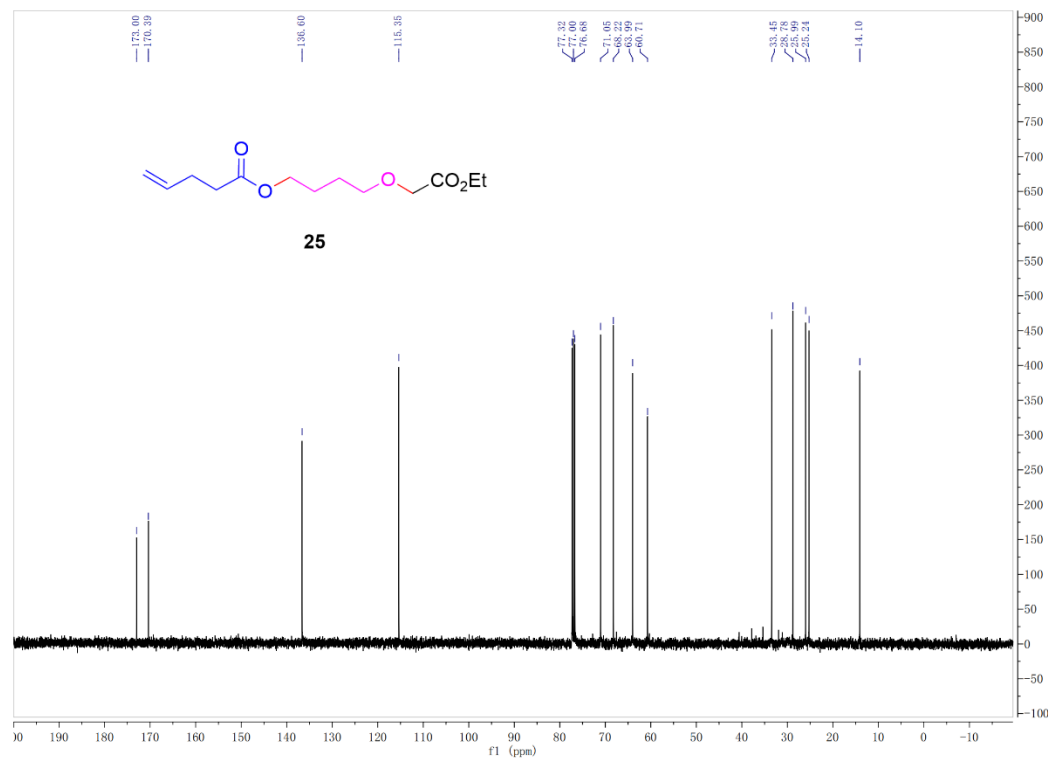
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 24**



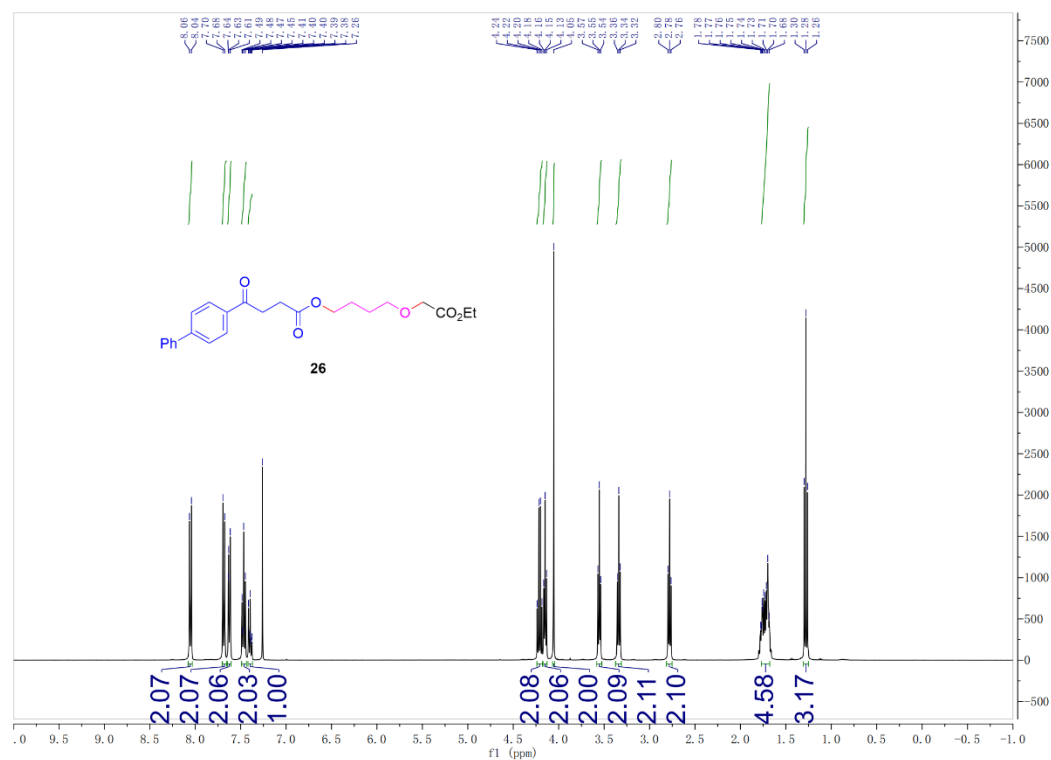
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 25**



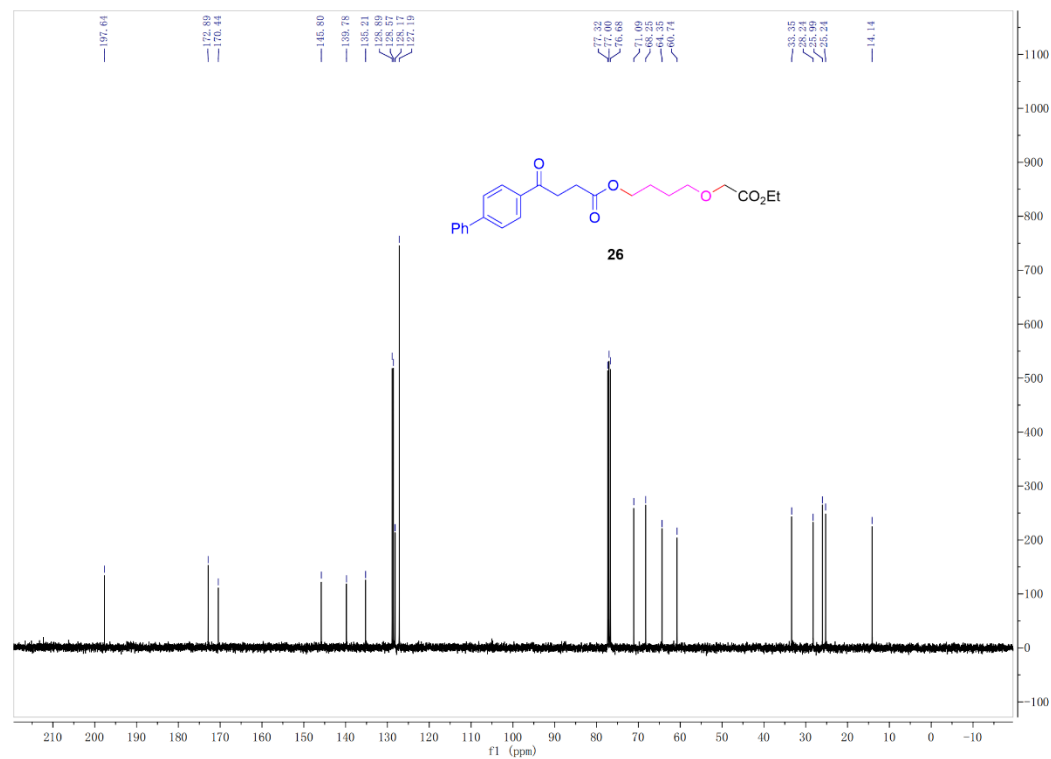
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 25**



**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 26**

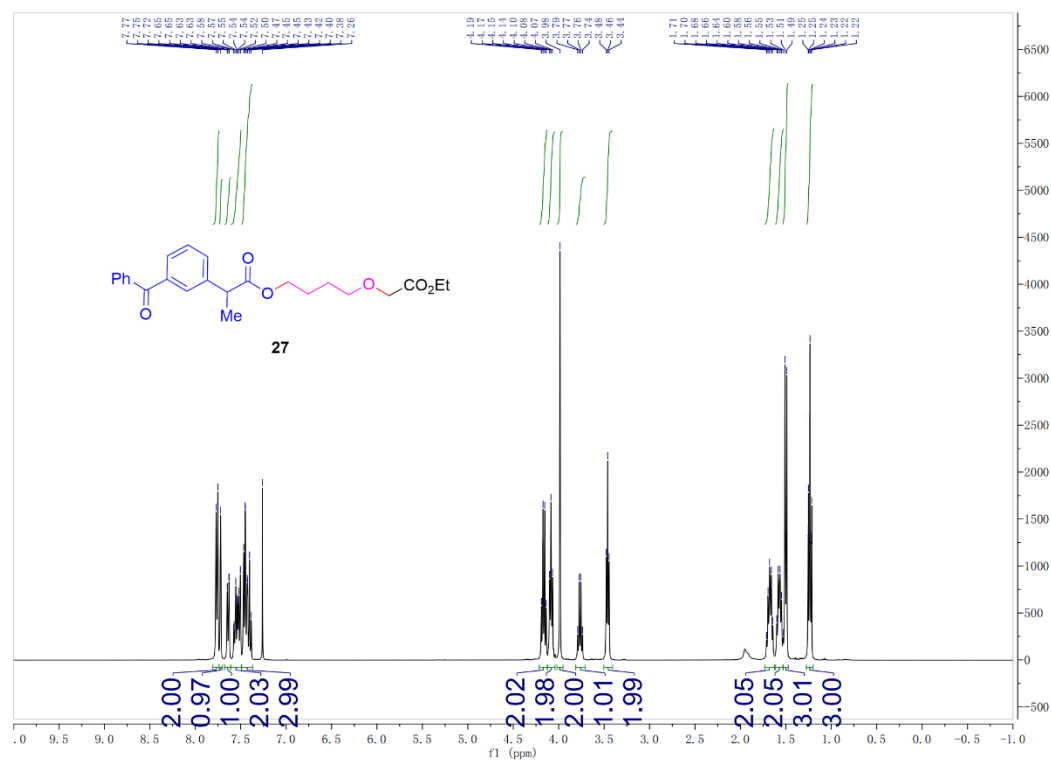


**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 26**

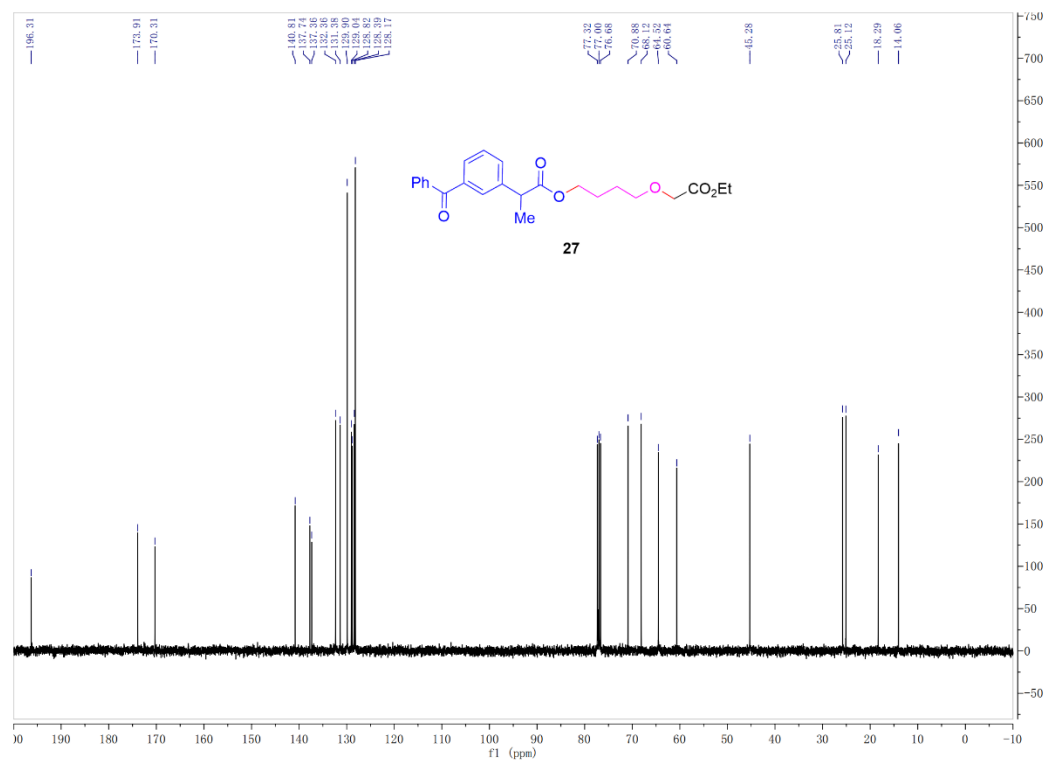




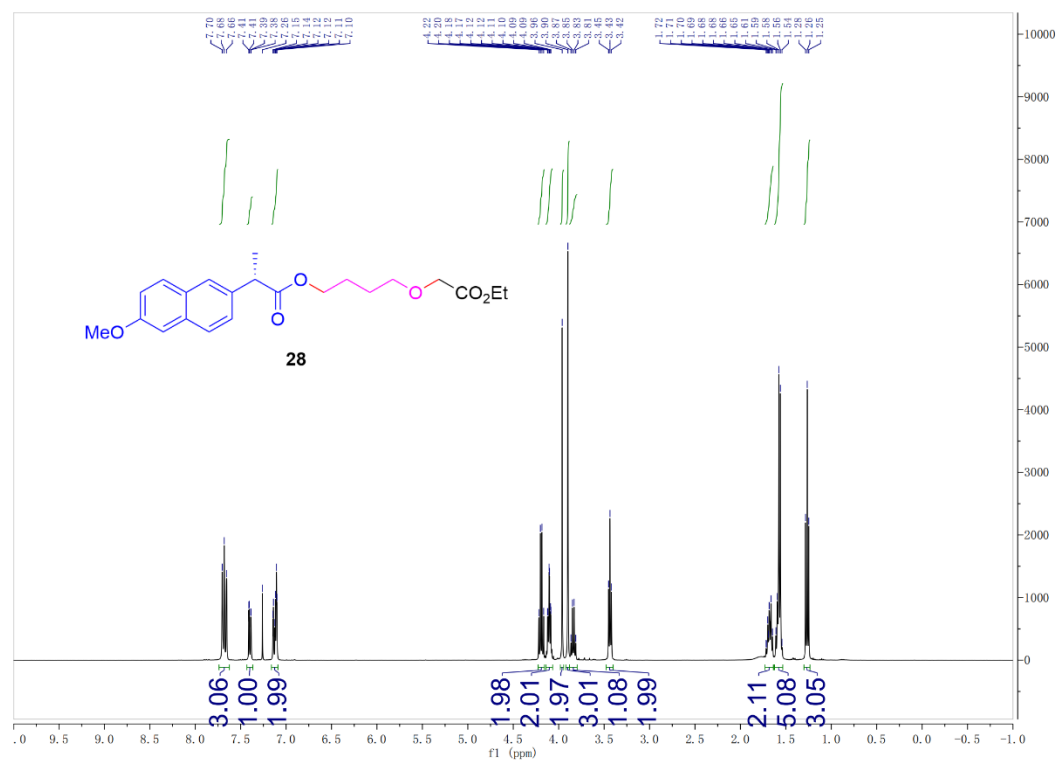
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 27**



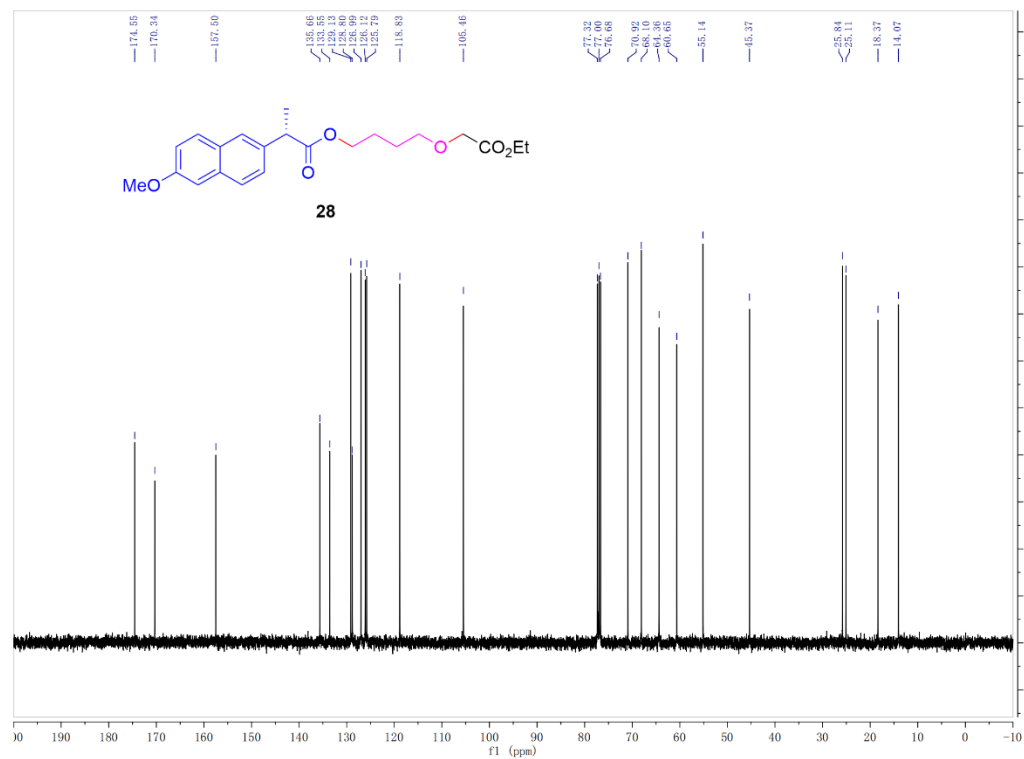
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 27**



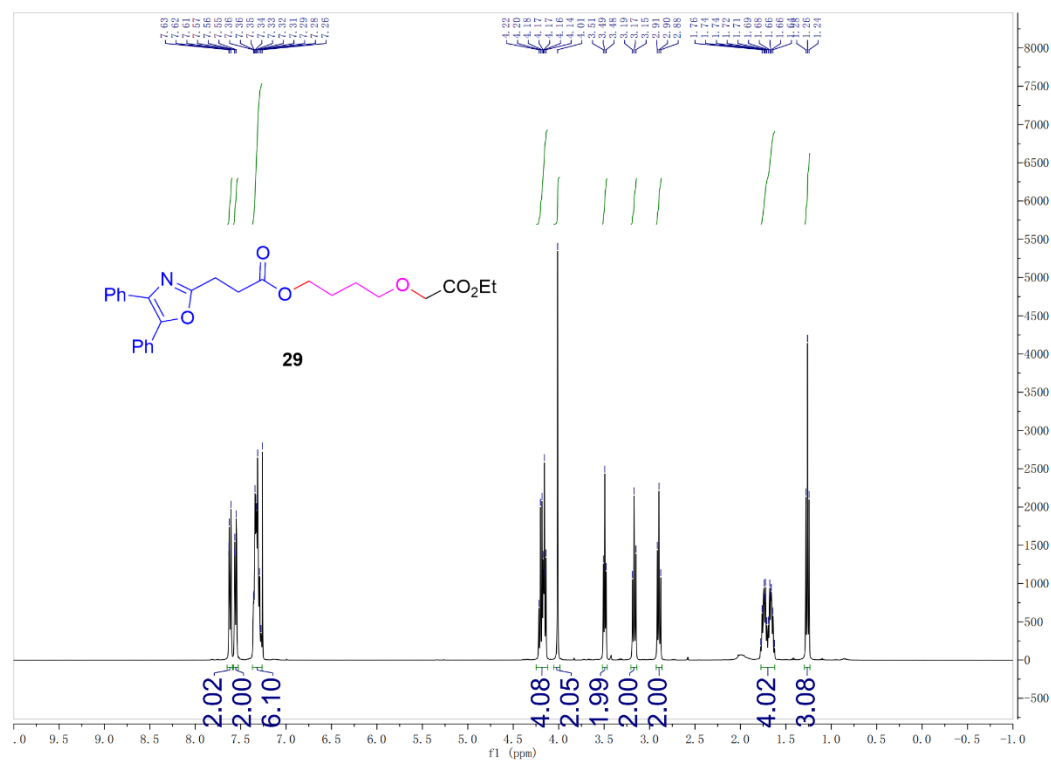
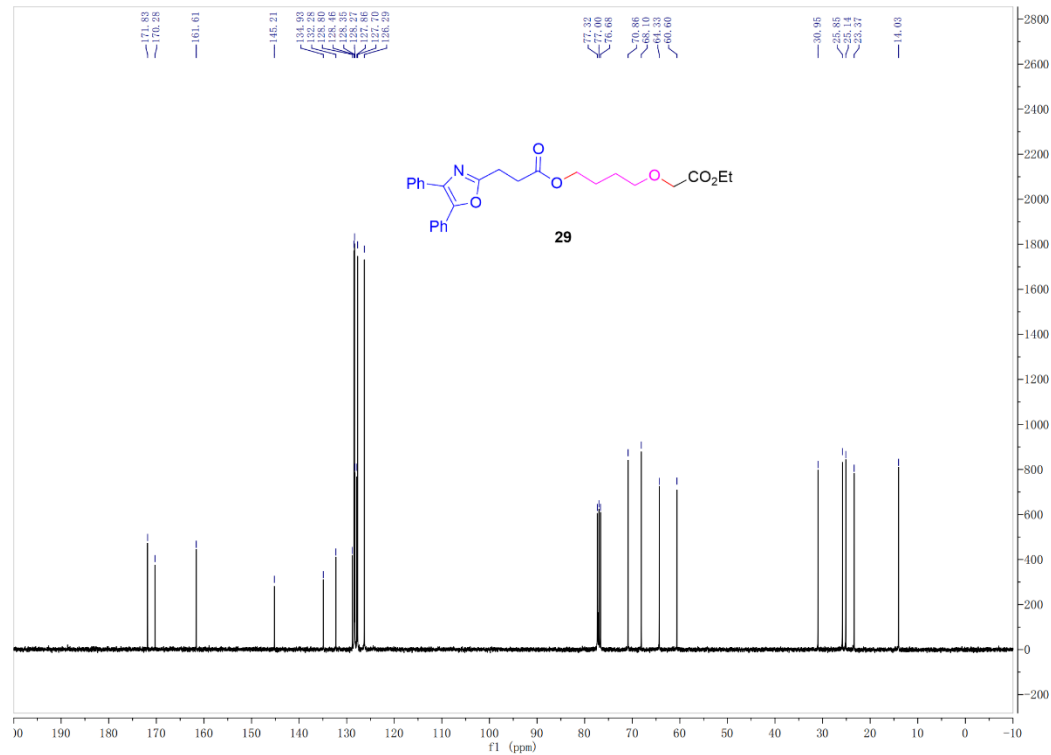
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 28**



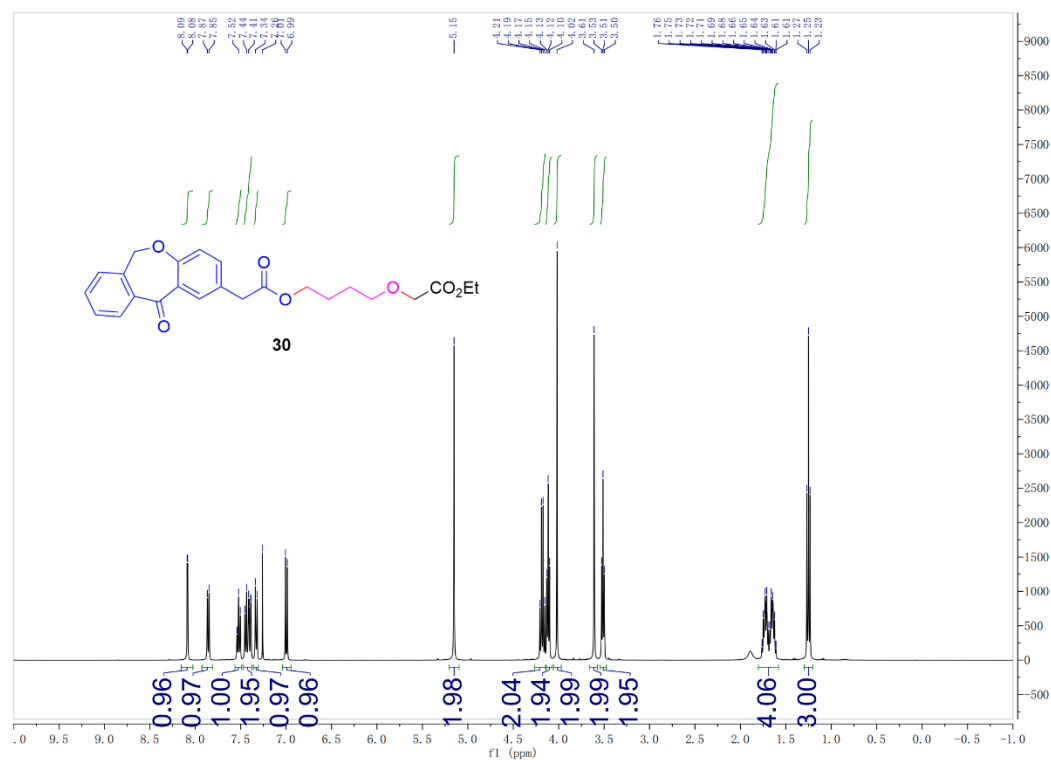
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 28**



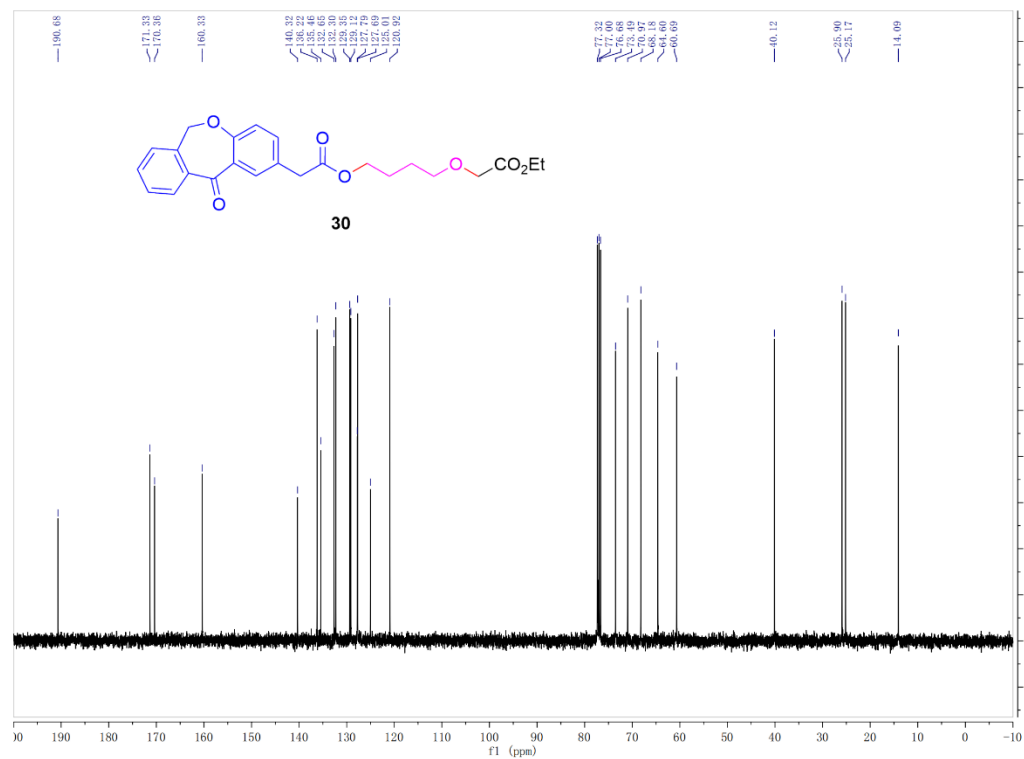
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 29**

 $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 29

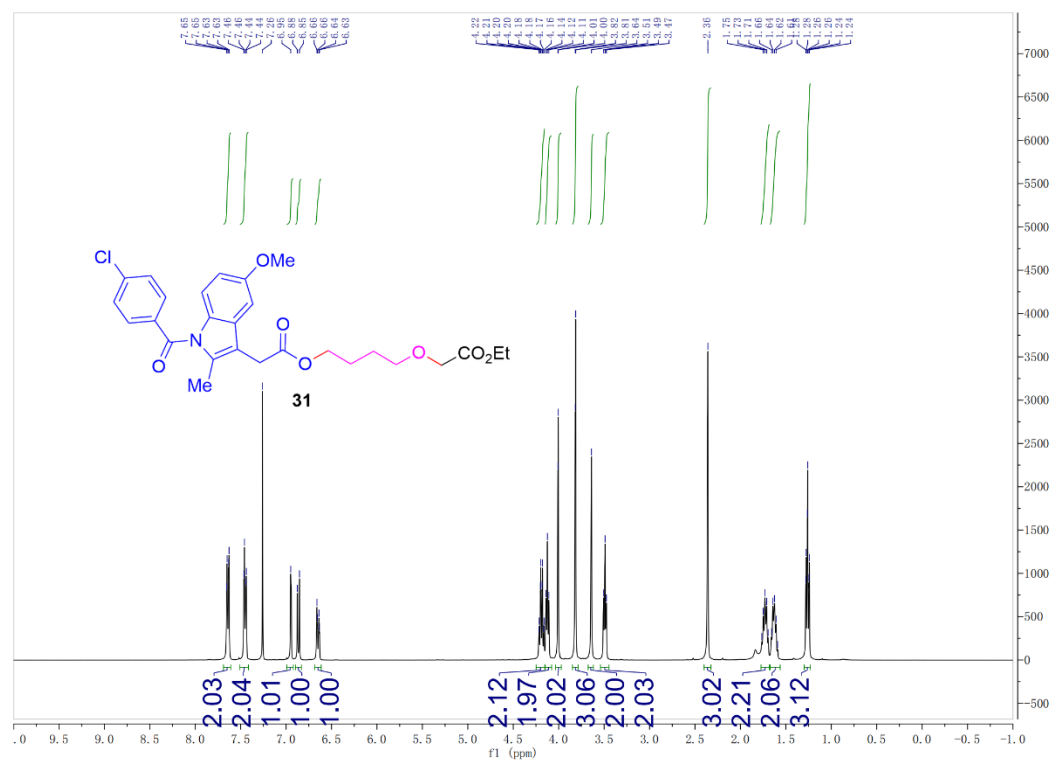
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 30**



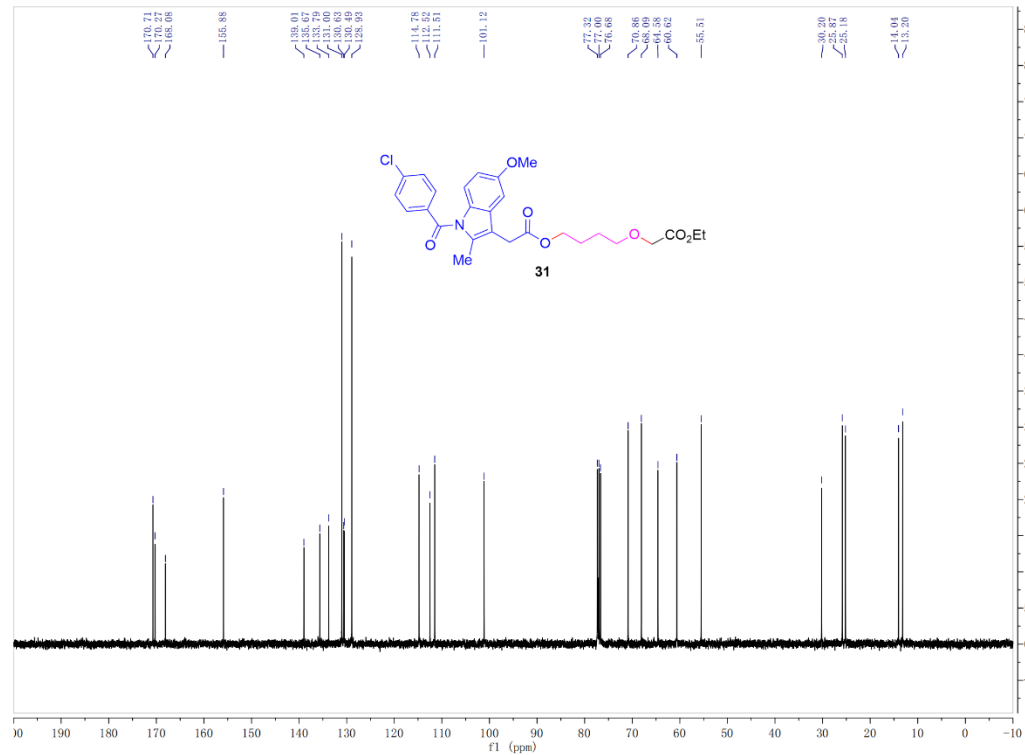
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 30**



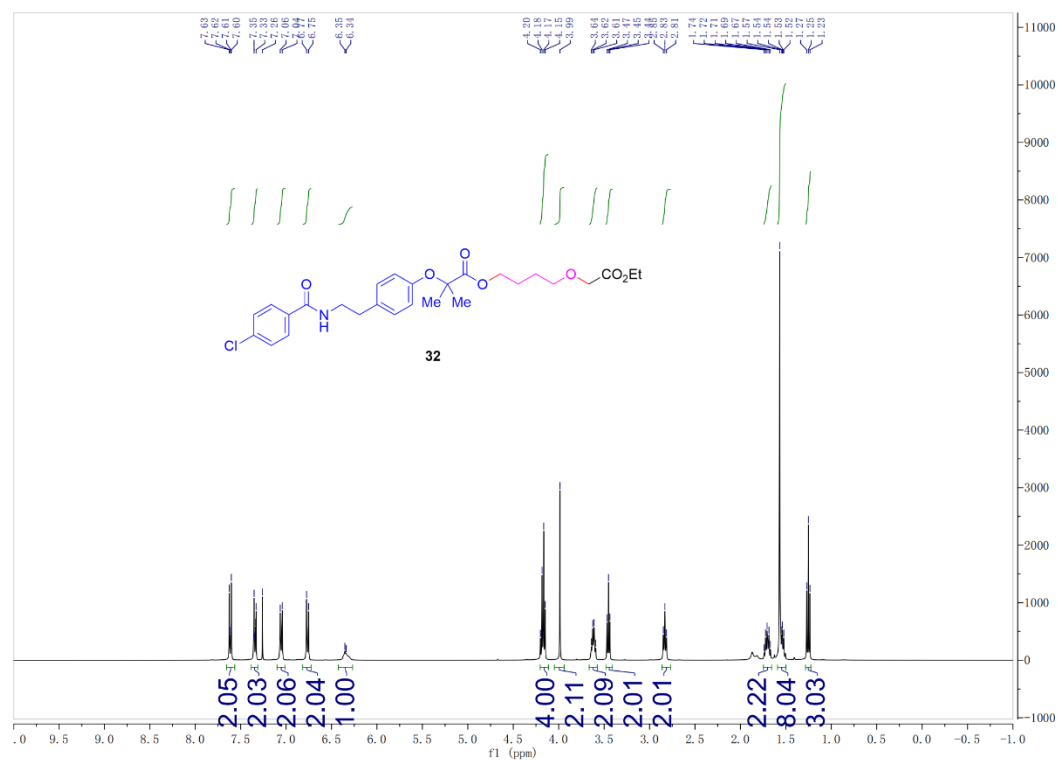
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 31**



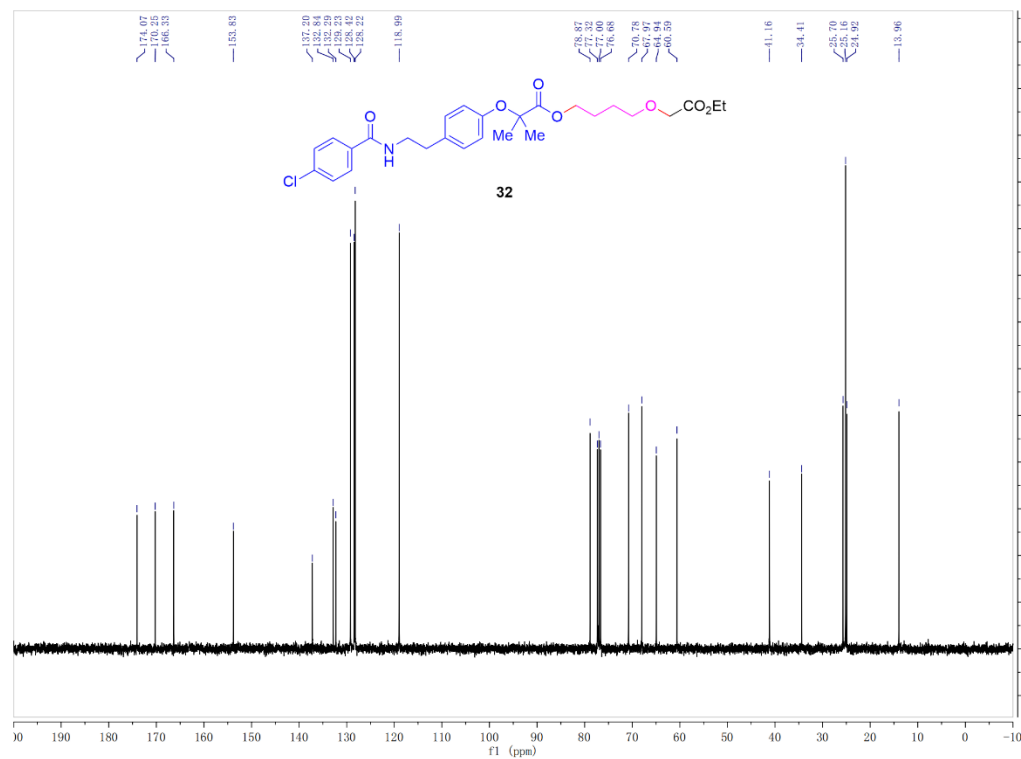
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 31**



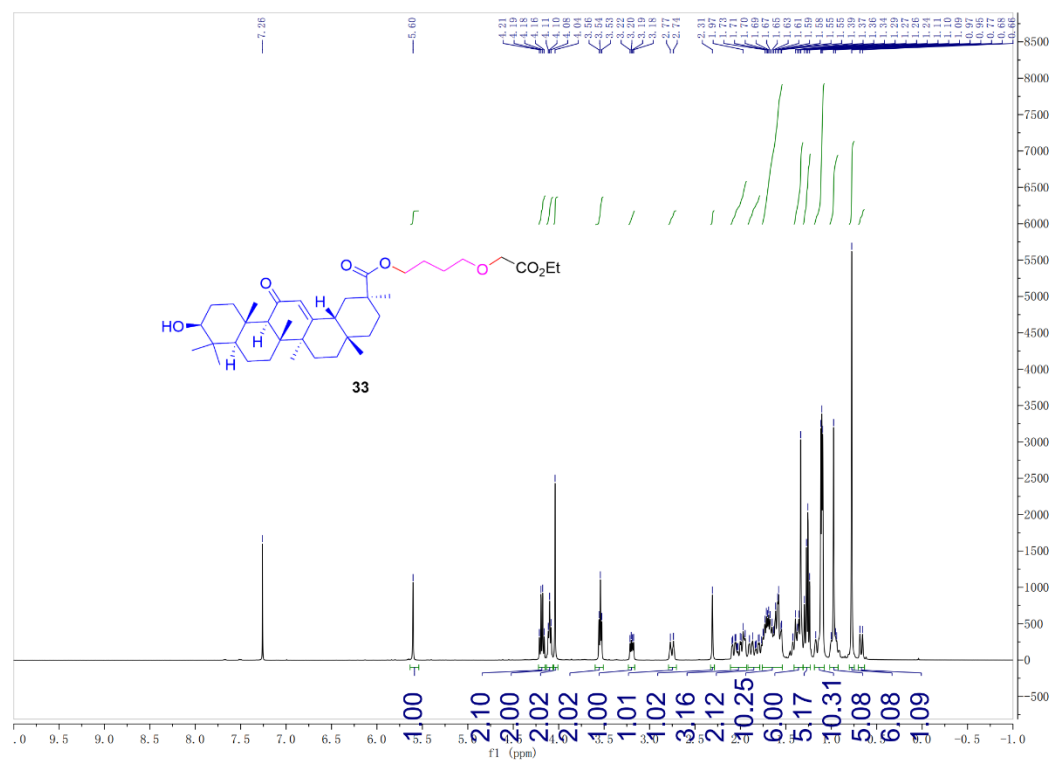
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 32**



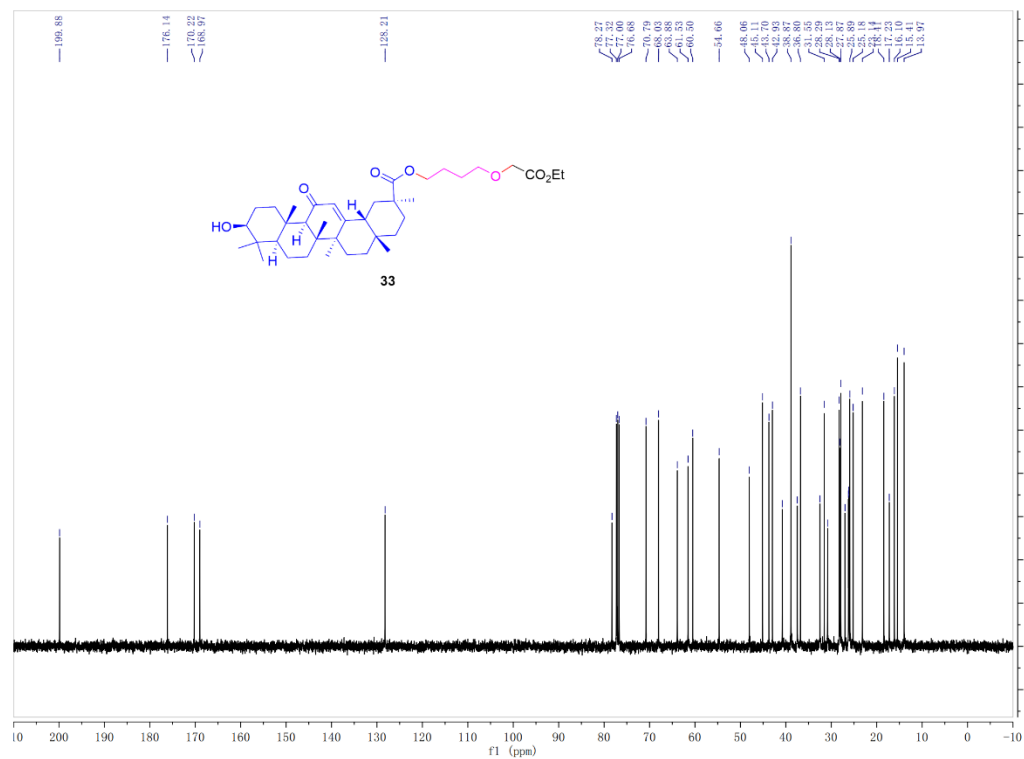
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 32**



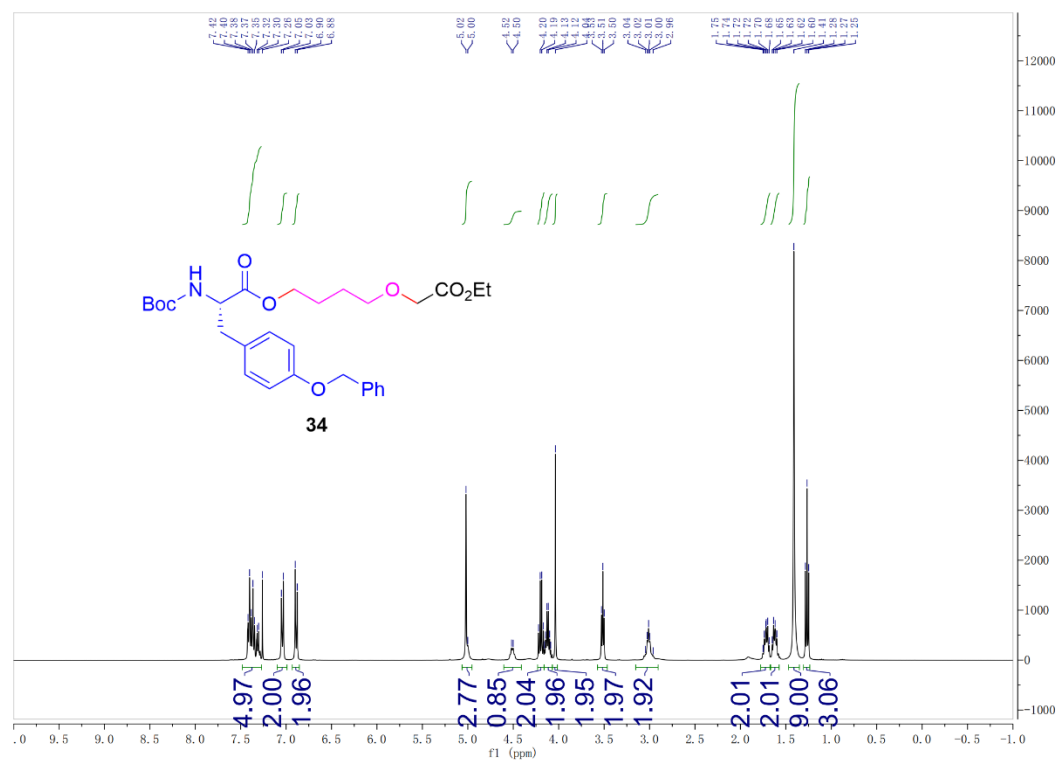
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 33**



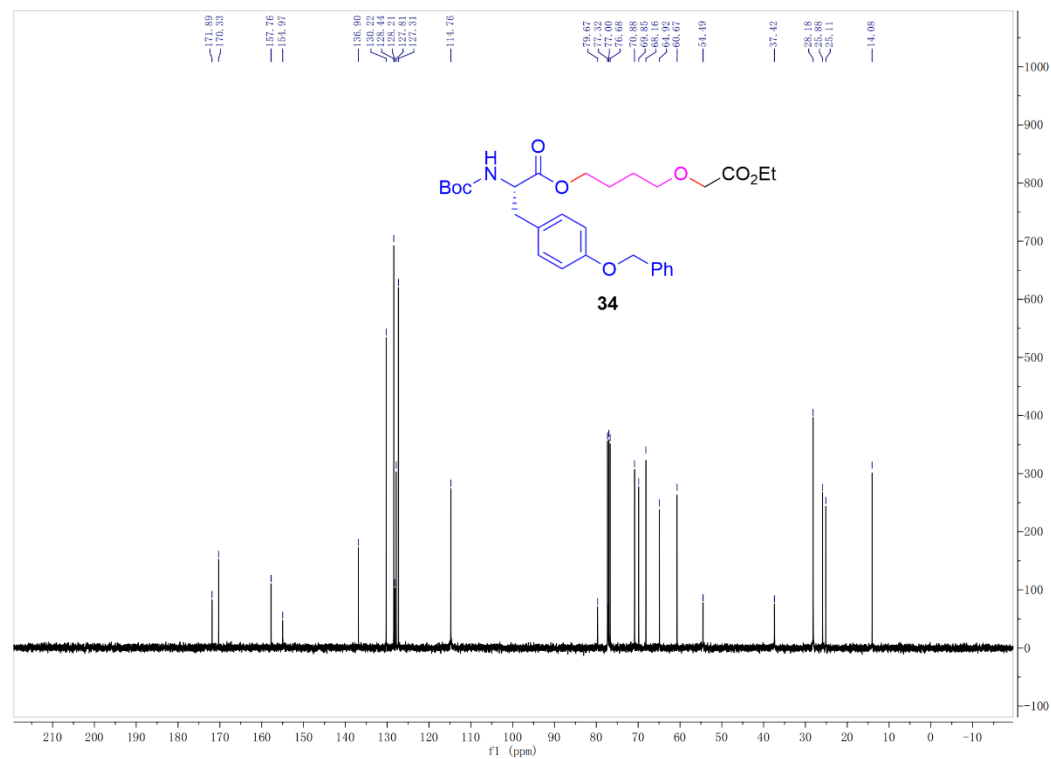
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 33**



**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 34**

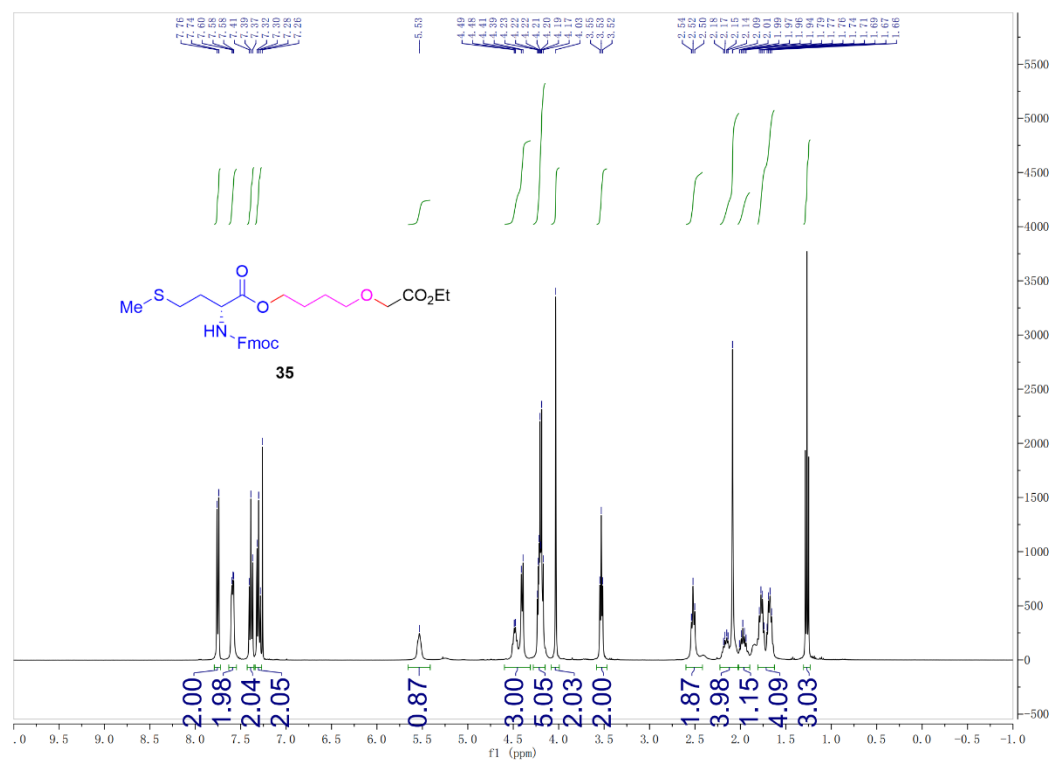


**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 34**

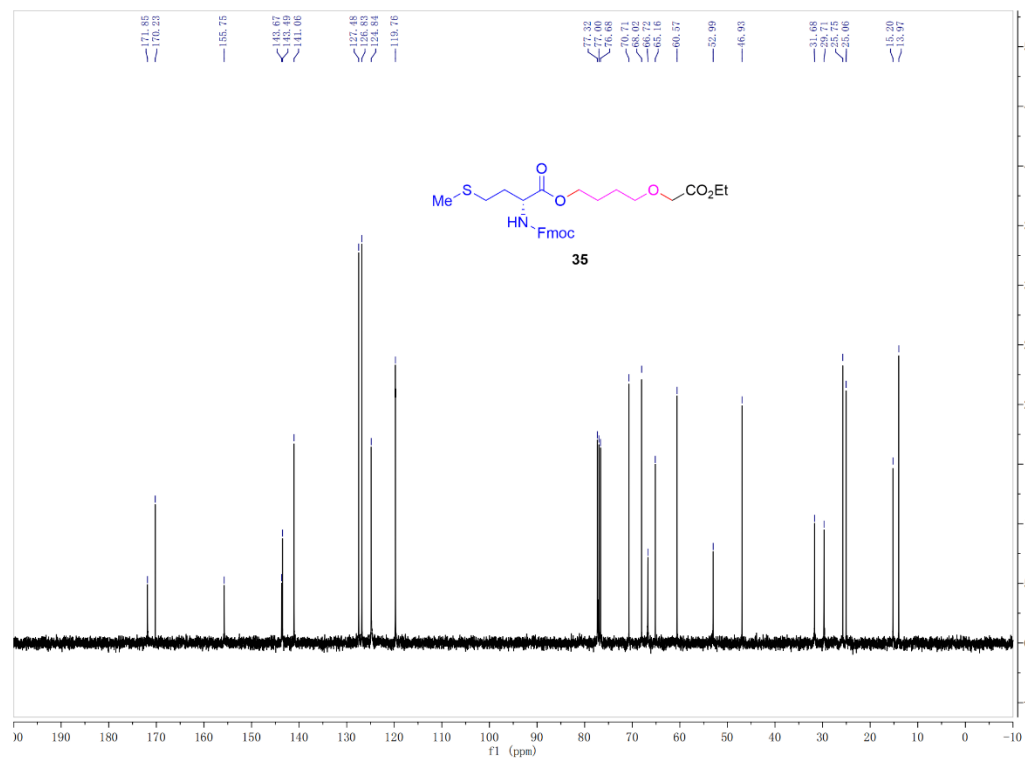




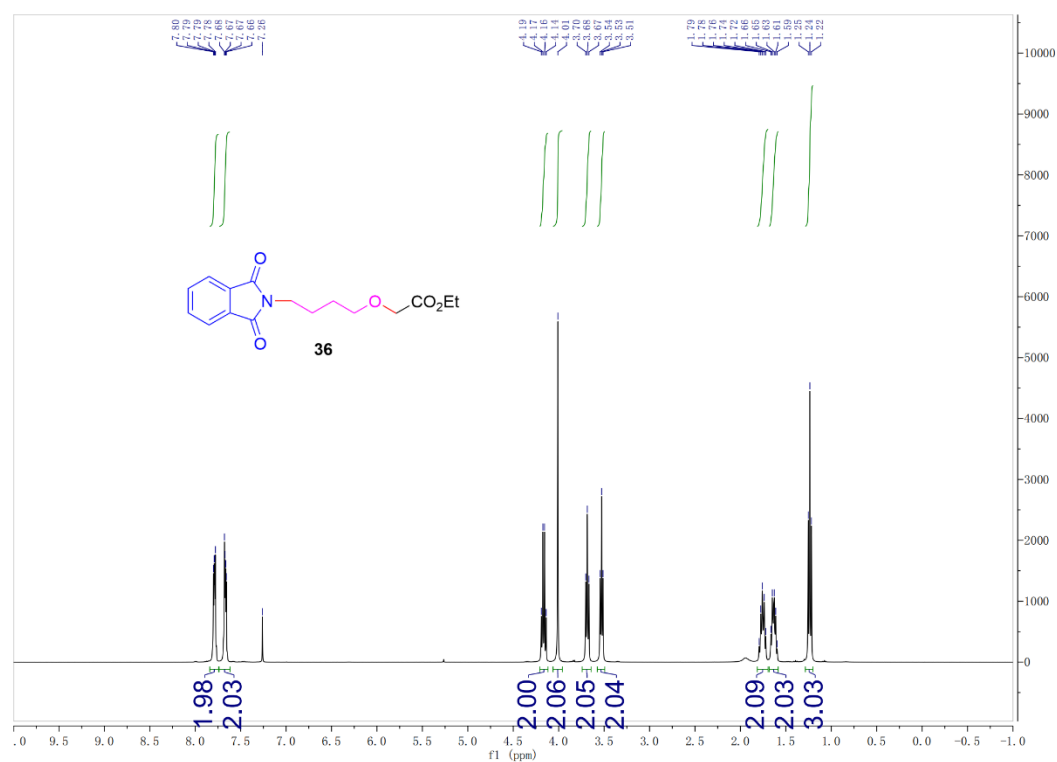
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 35**



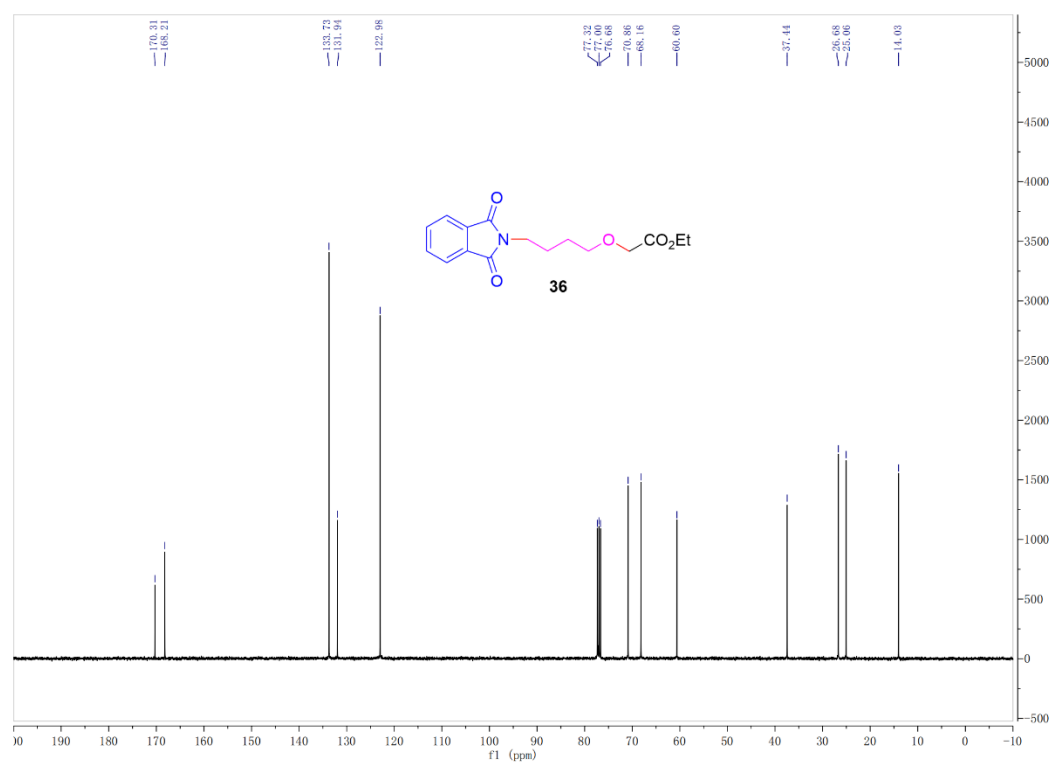
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 35**



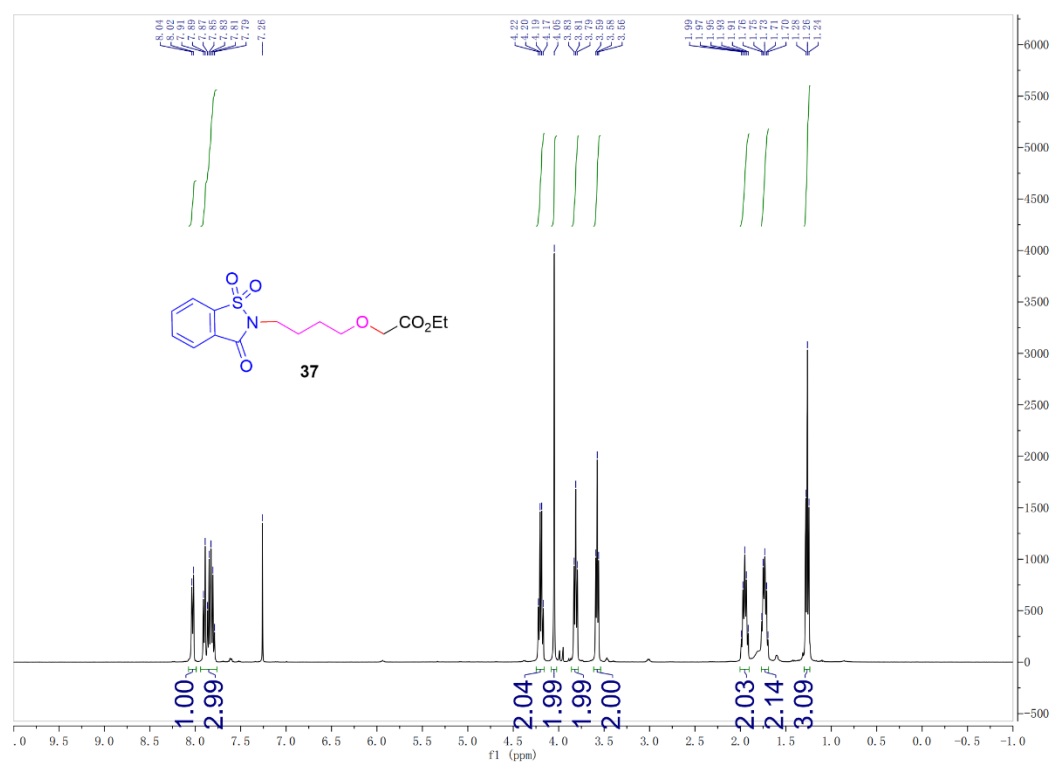
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 36**



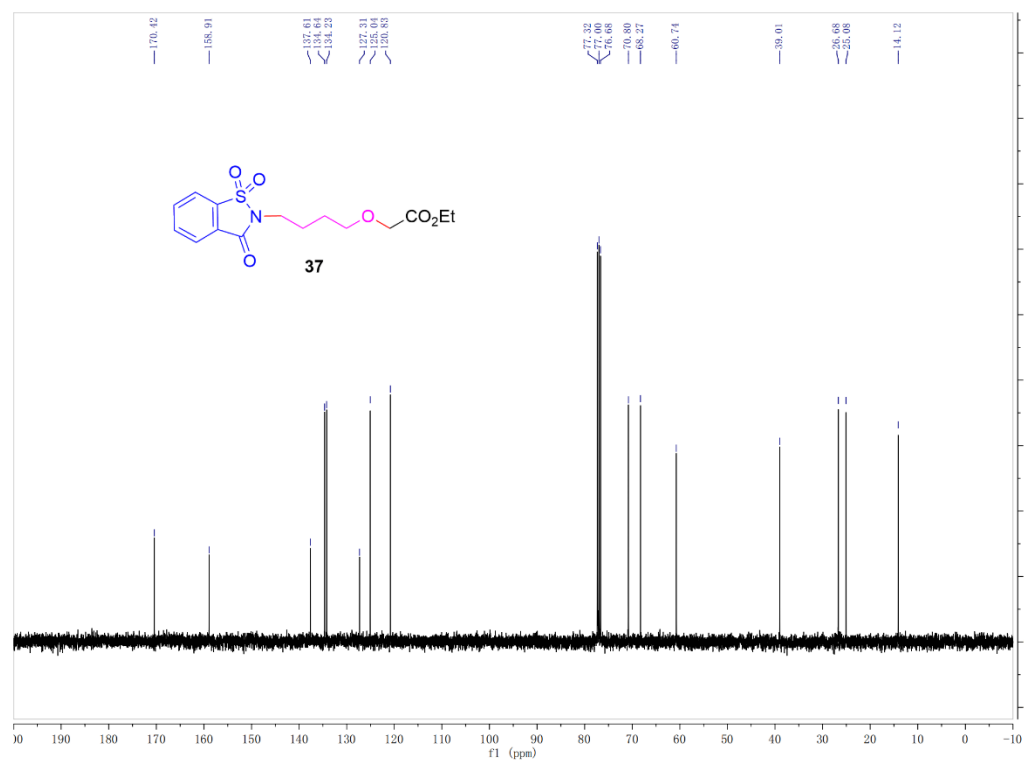
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 36**



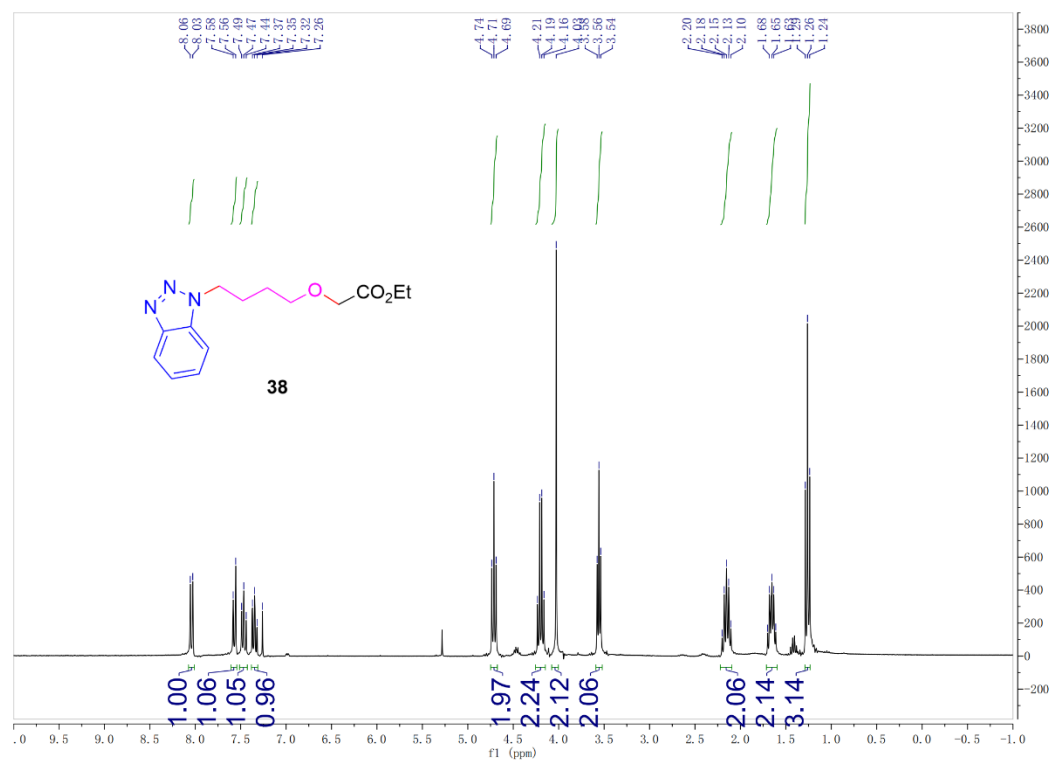
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 37**



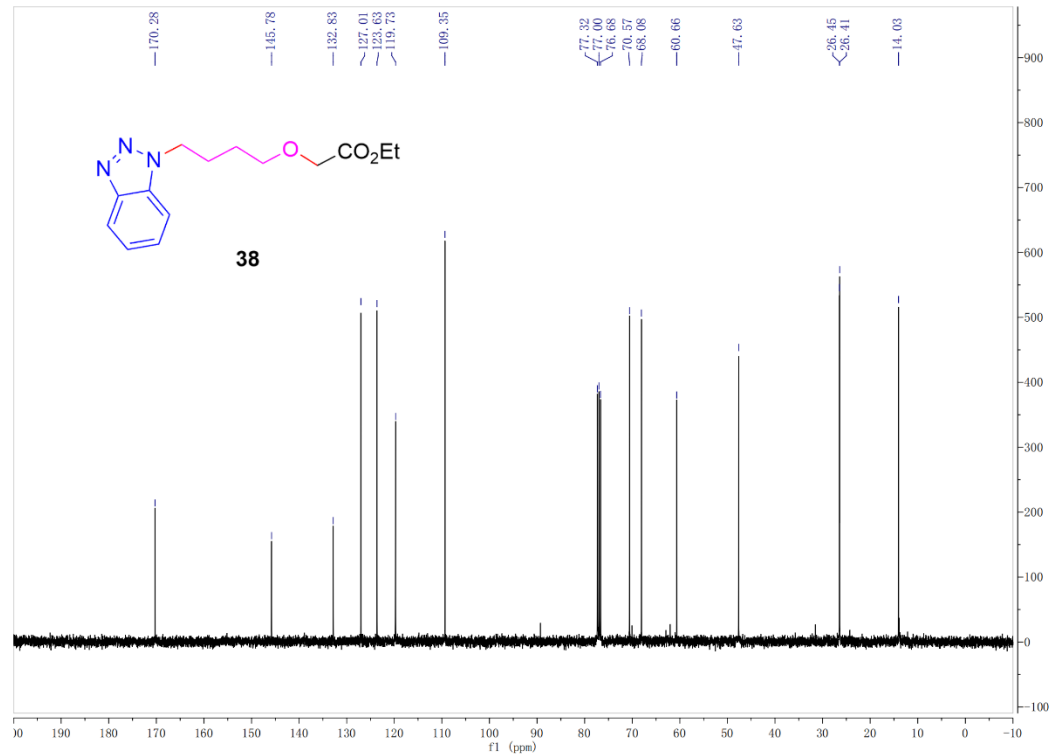
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 37**



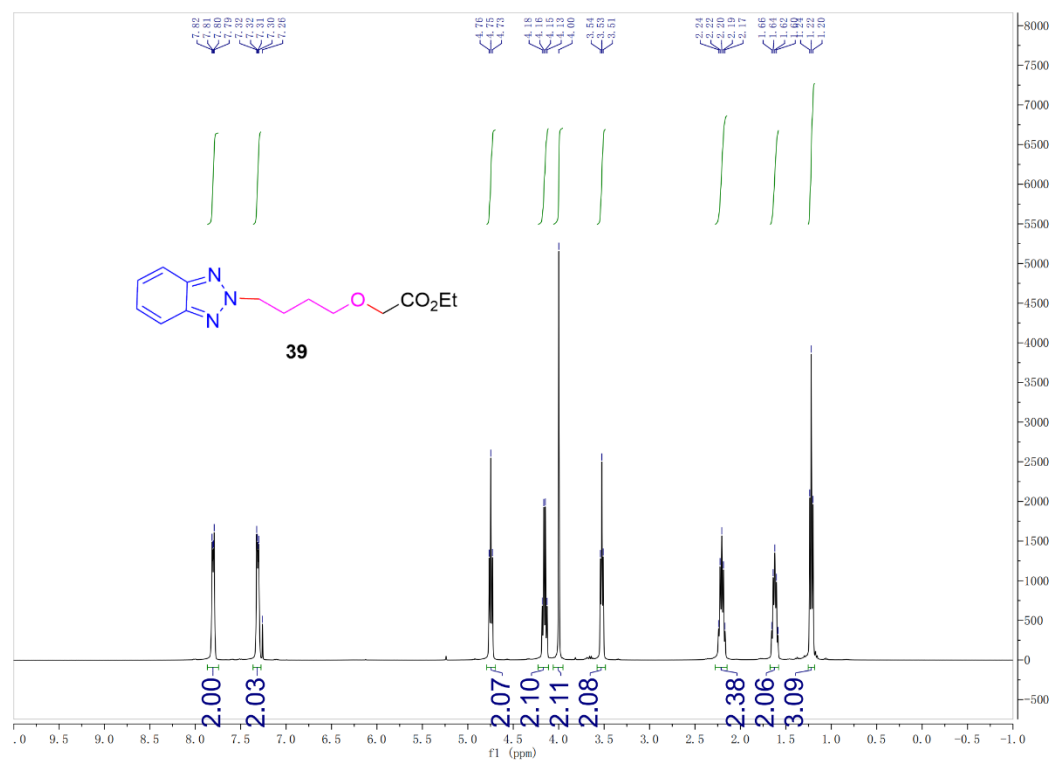
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 38**



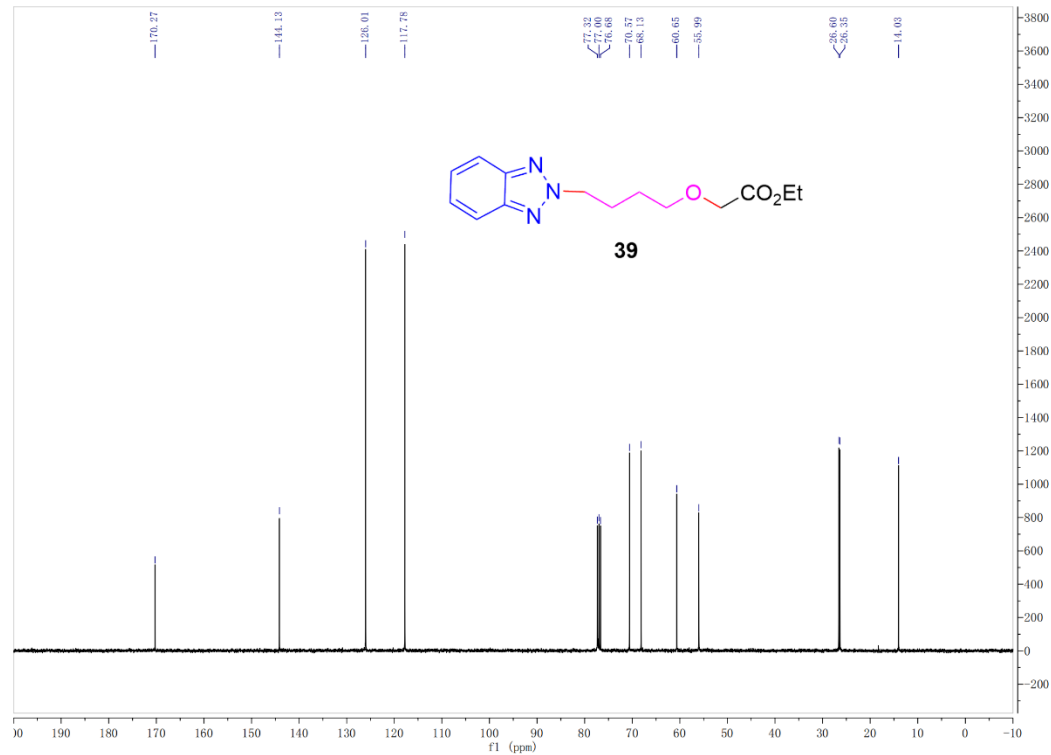
**$^{13}\text{C}\{^1\text{H}\}$  NMR (750 MHz,  $\text{CDCl}_3$ ) Spectrum of 38**



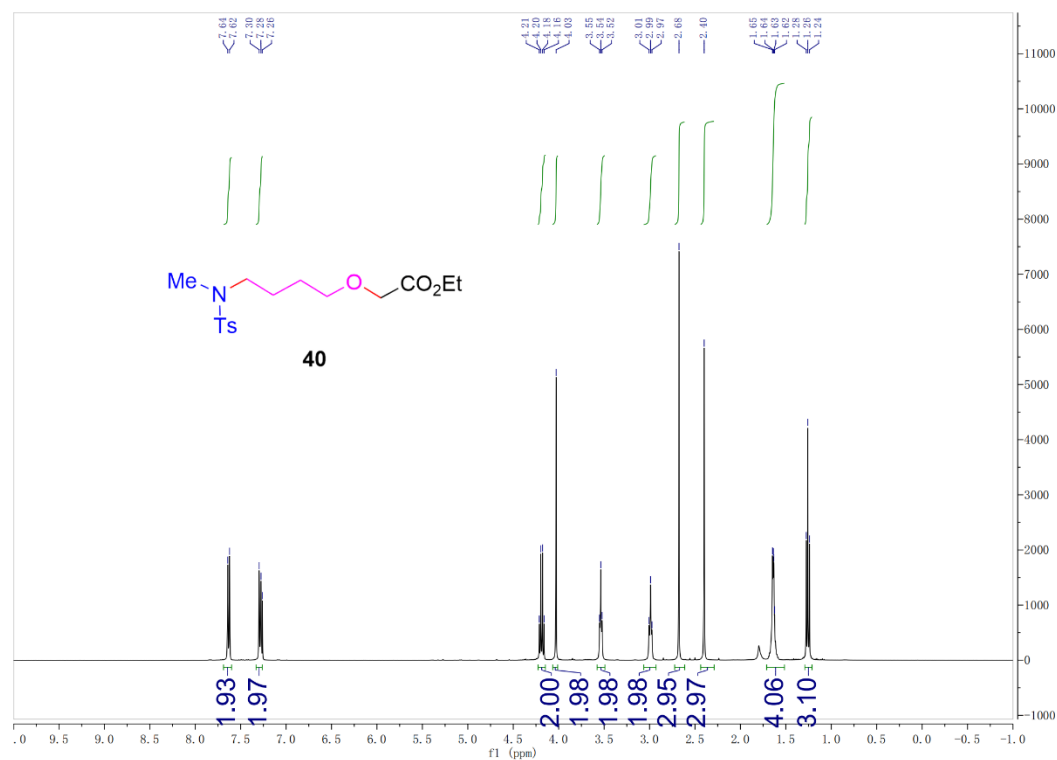
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 39**



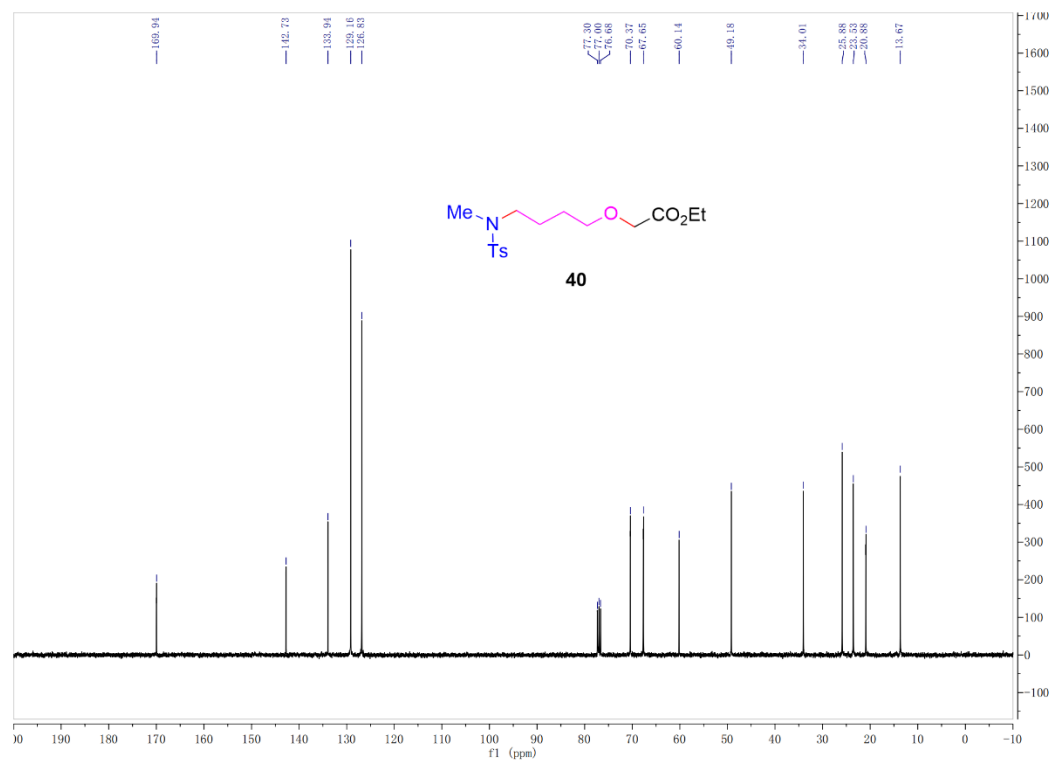
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 39**



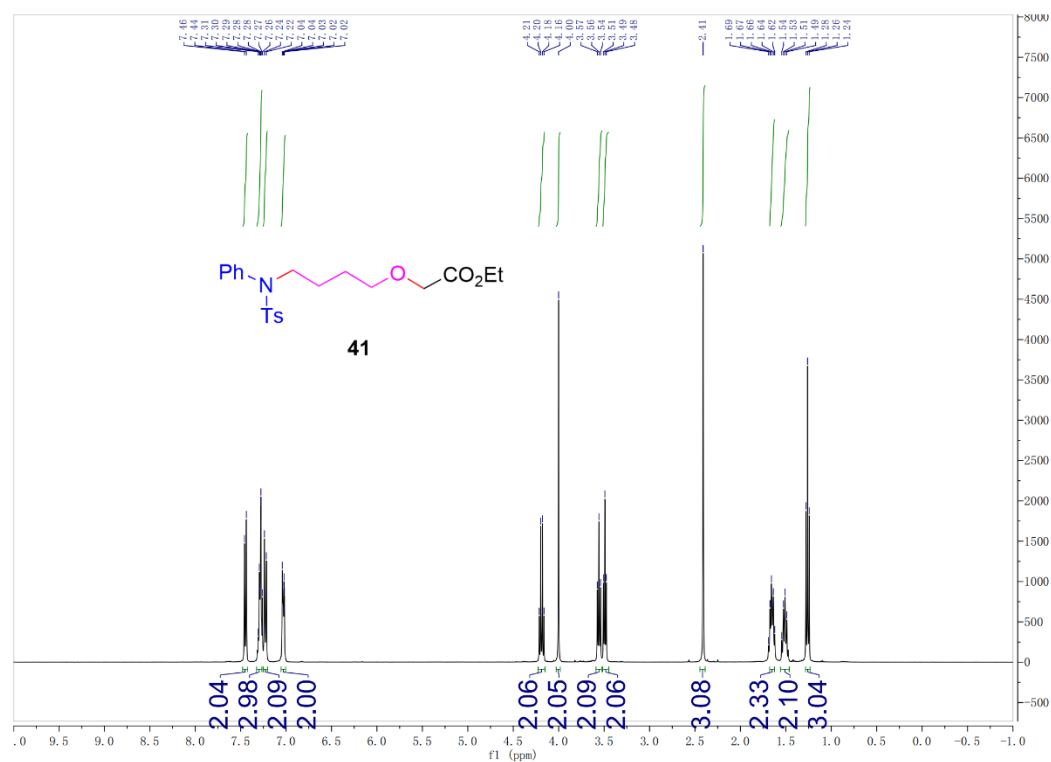
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 40**



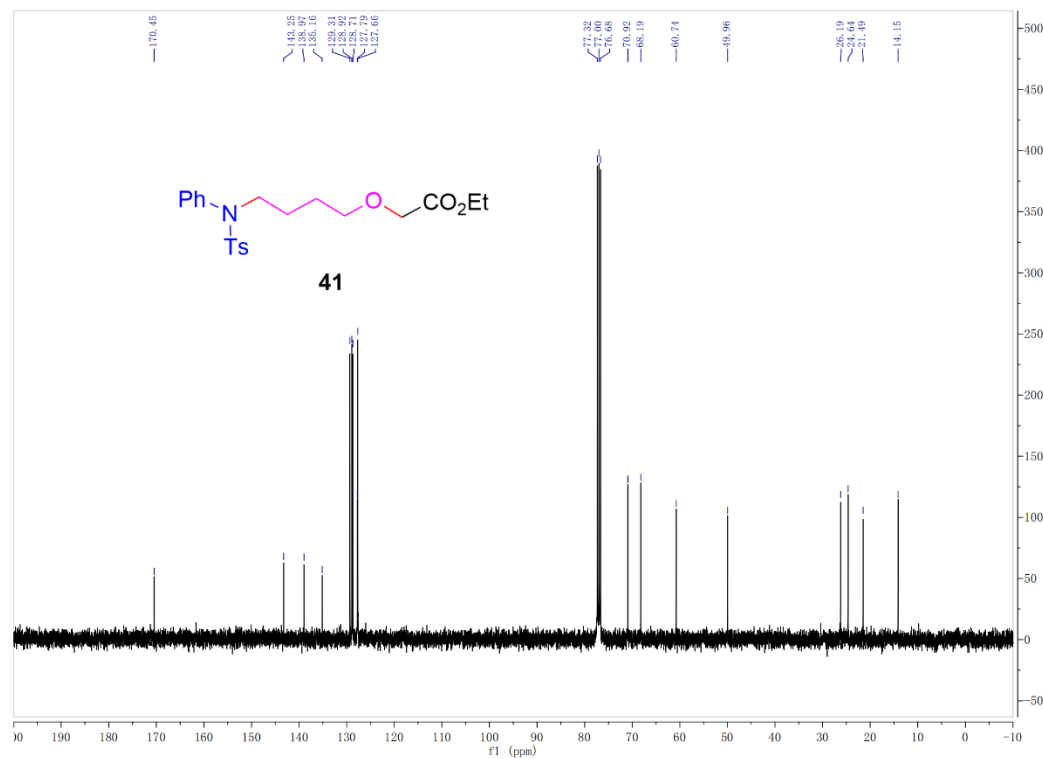
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 40**



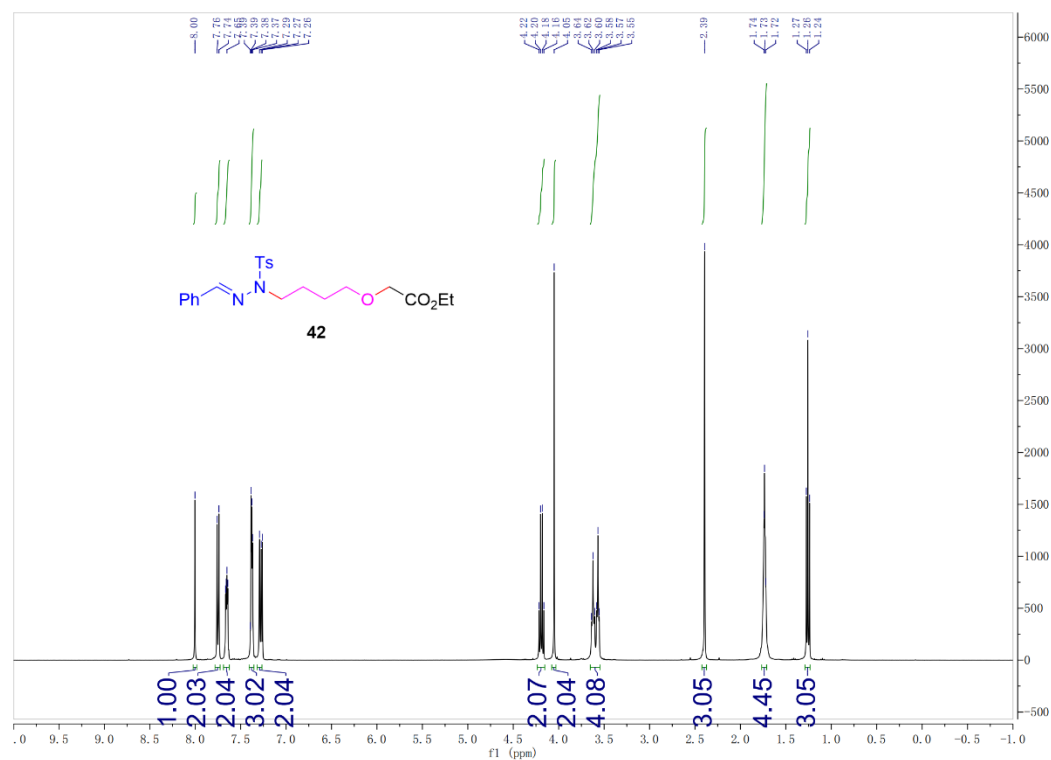
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 41**



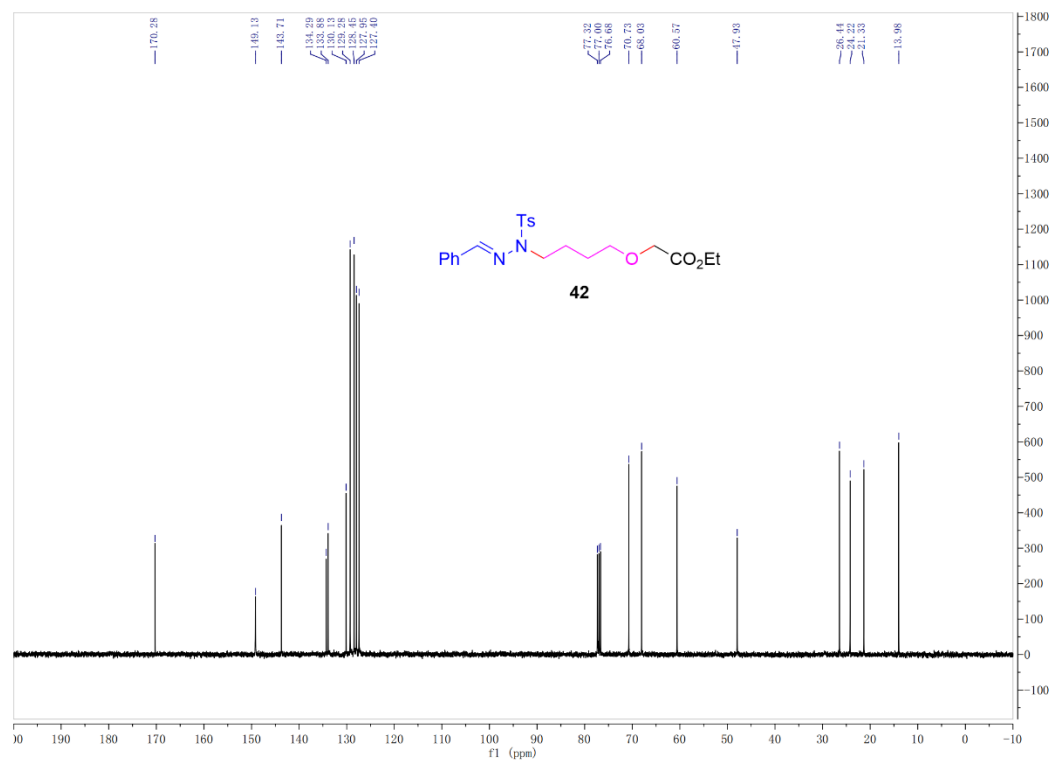
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 41**



**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 42**

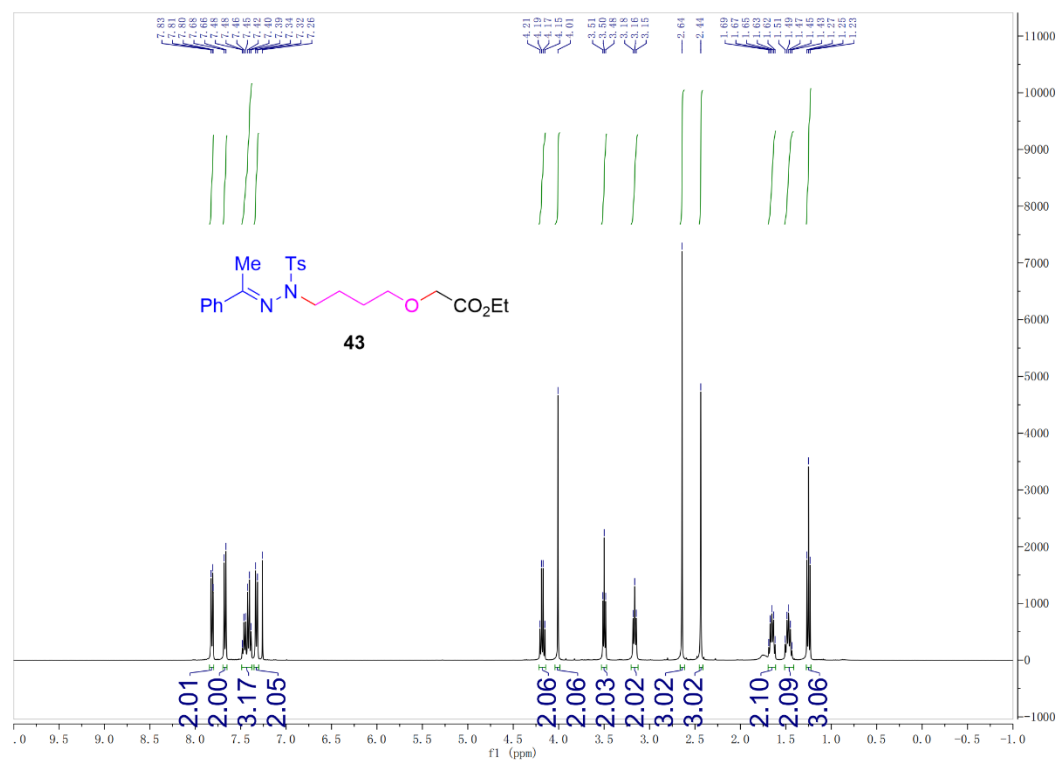


**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 42**

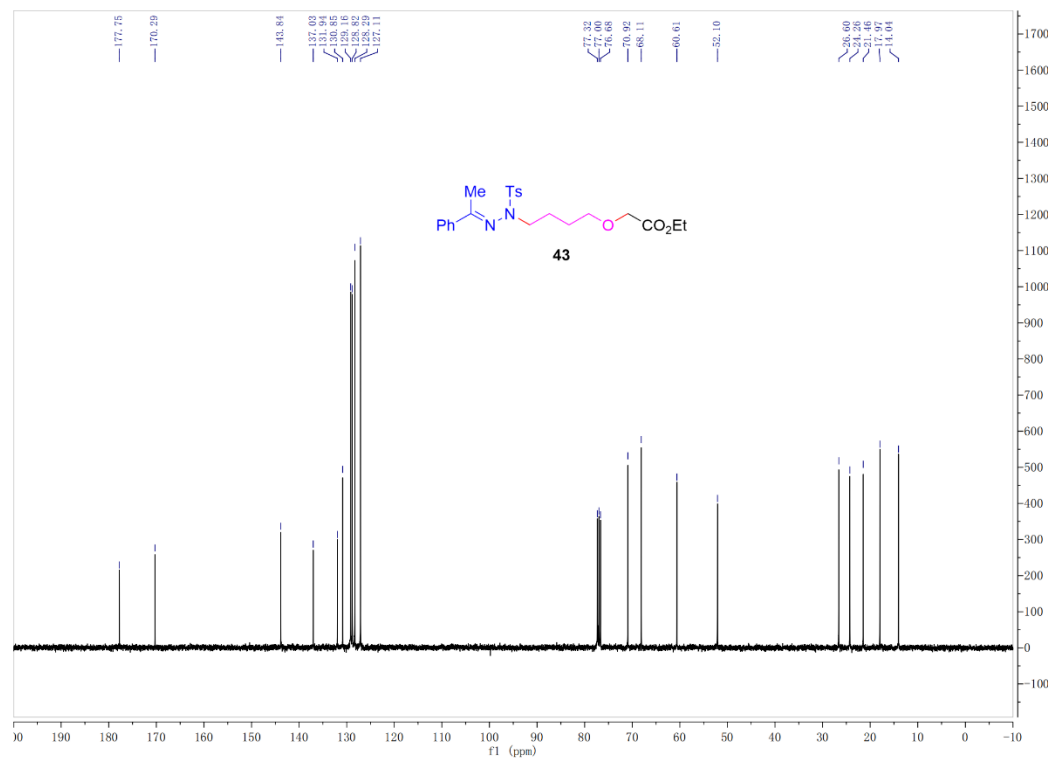




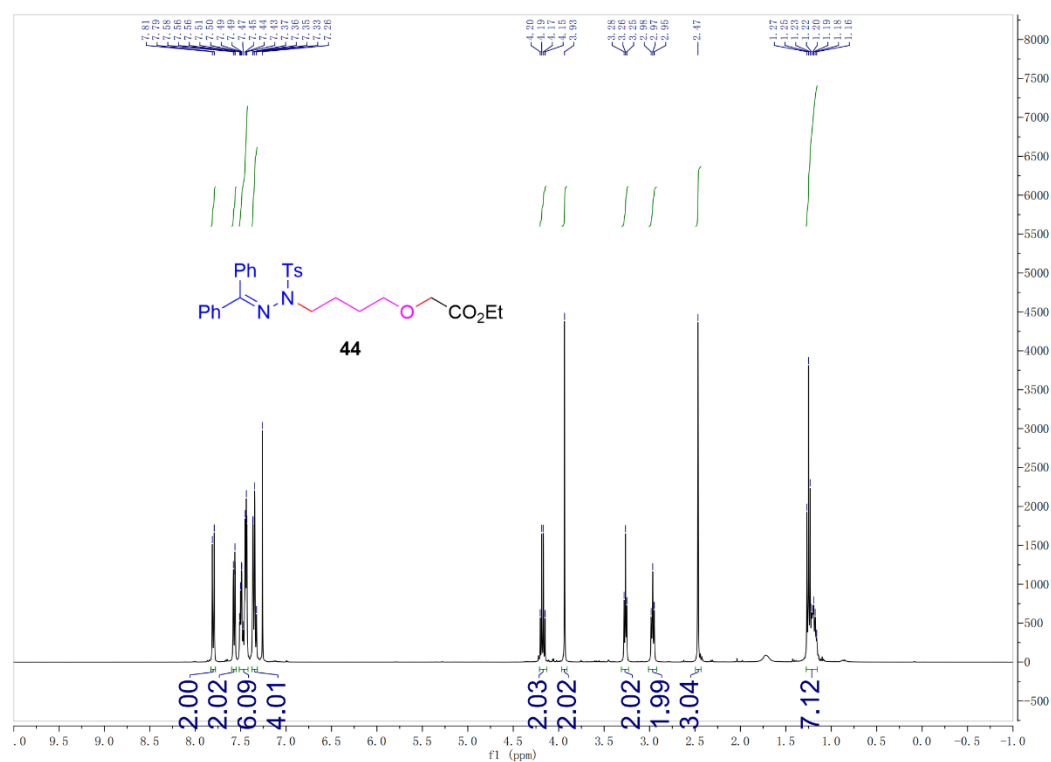
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 43**



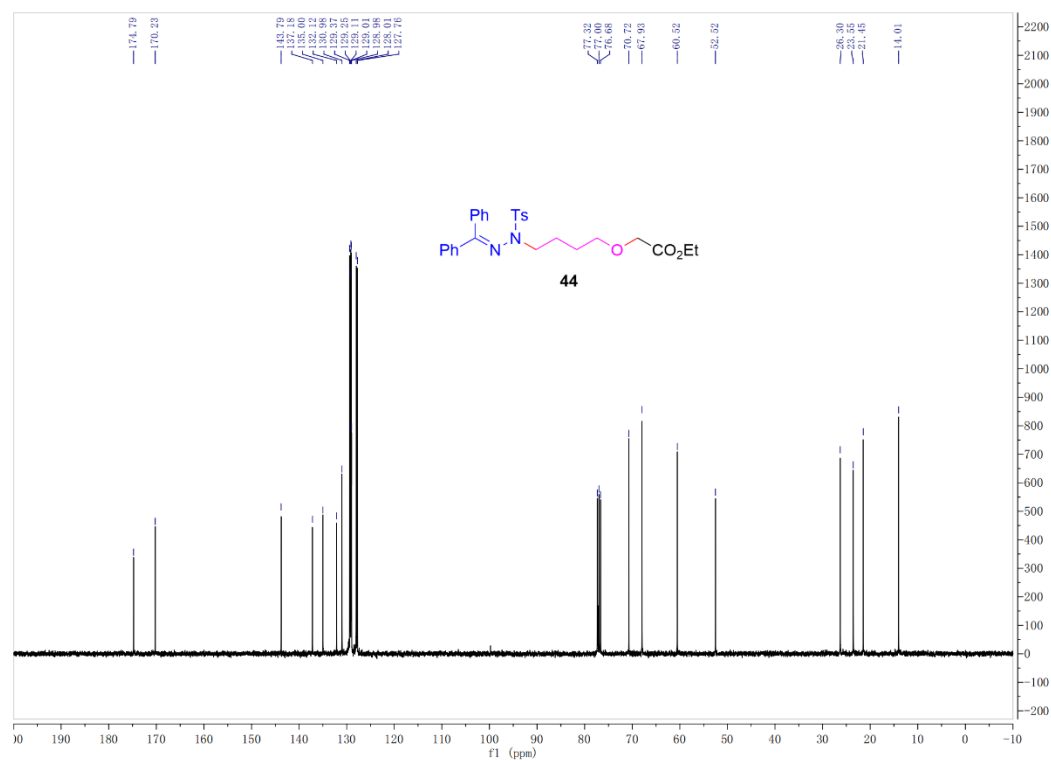
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 43**



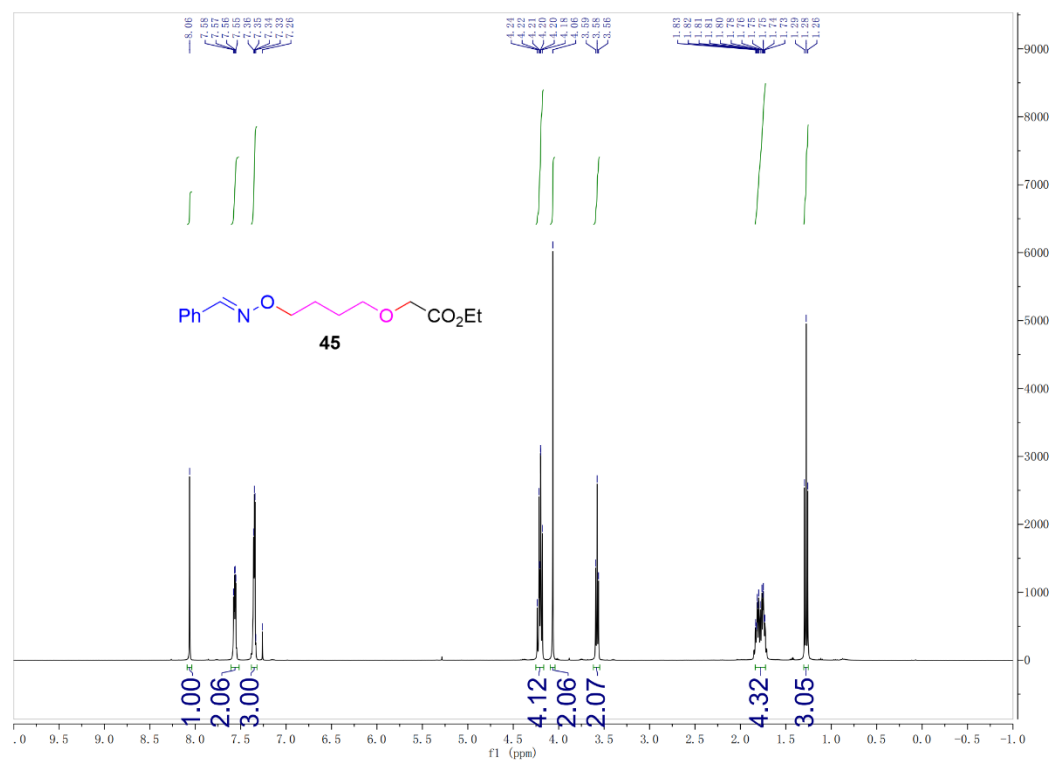
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 44**



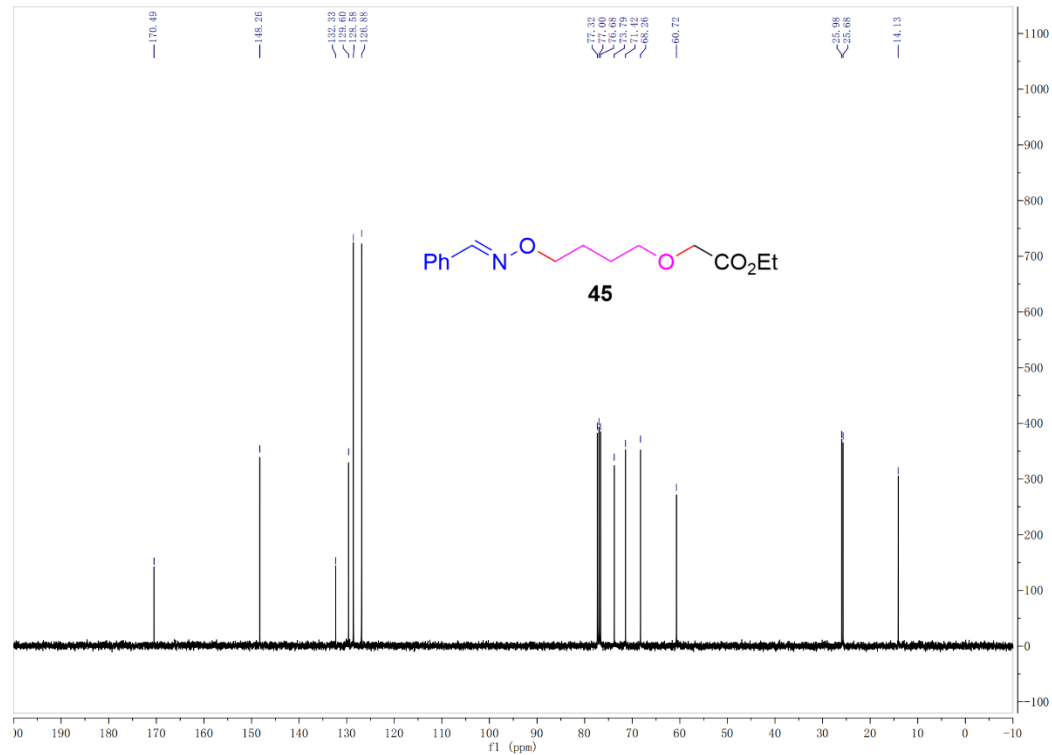
**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) Spectrum of 44**



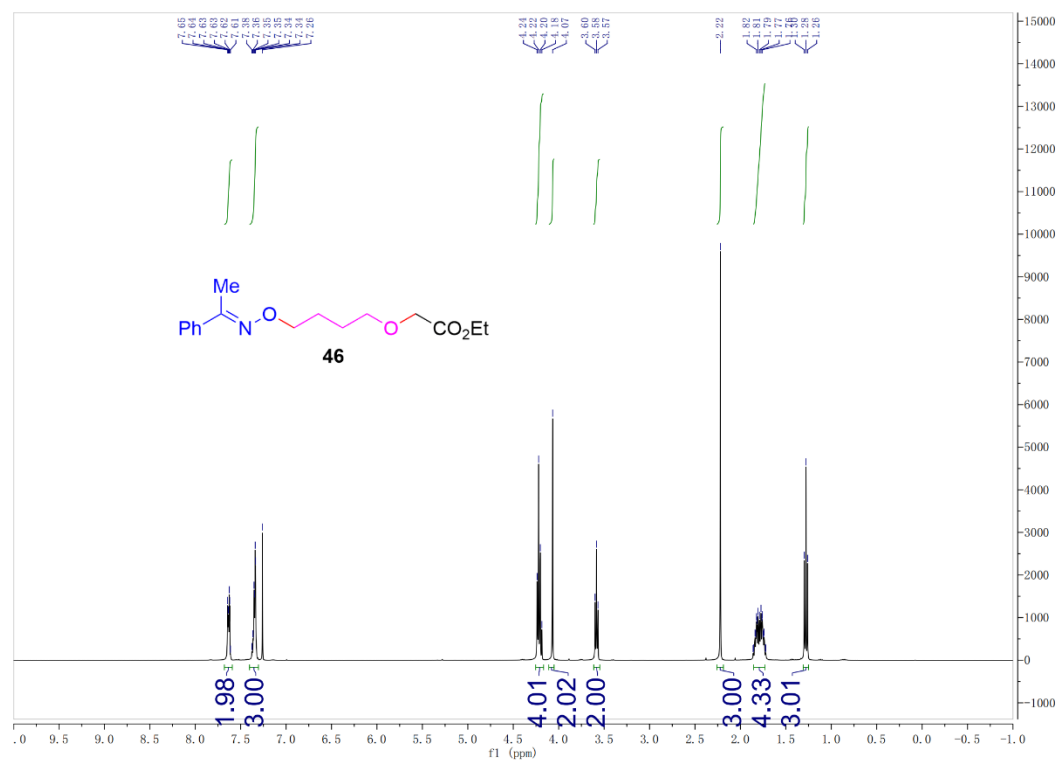
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 45**



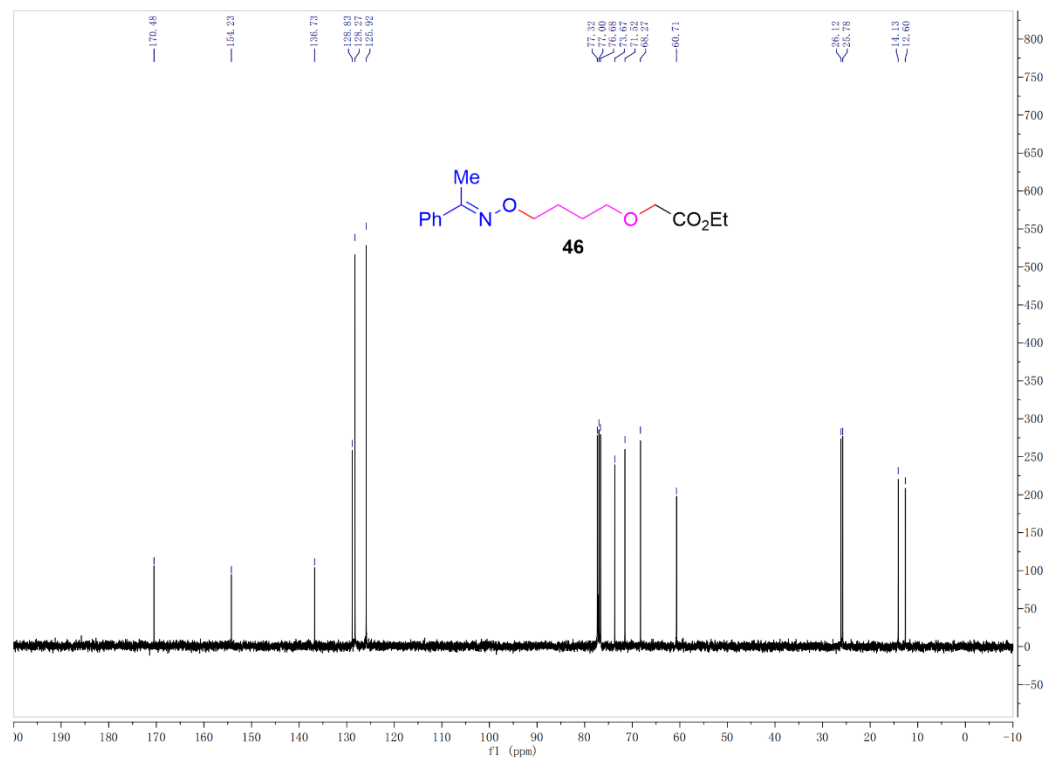
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 45**



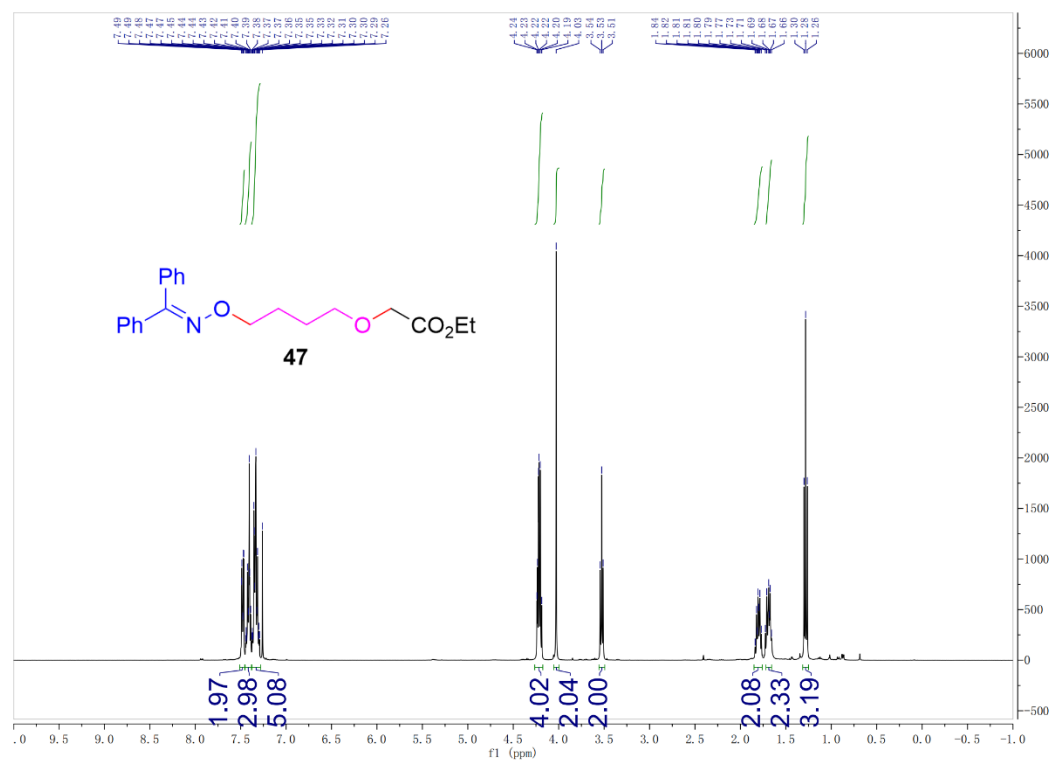
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 46**



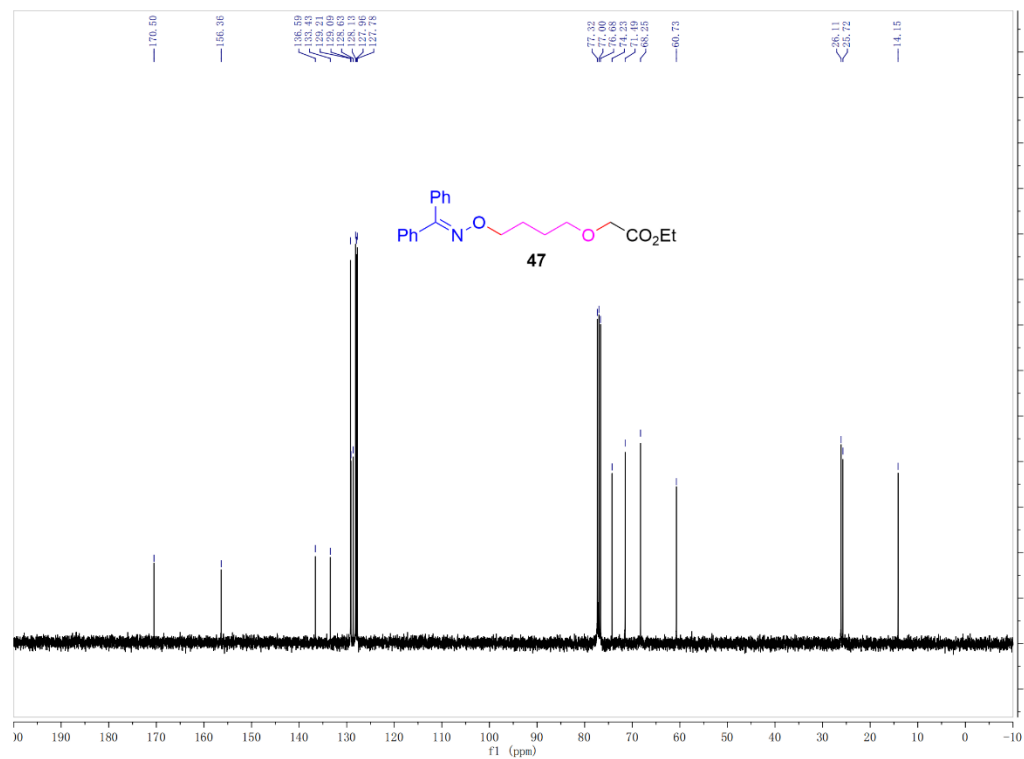
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 46**



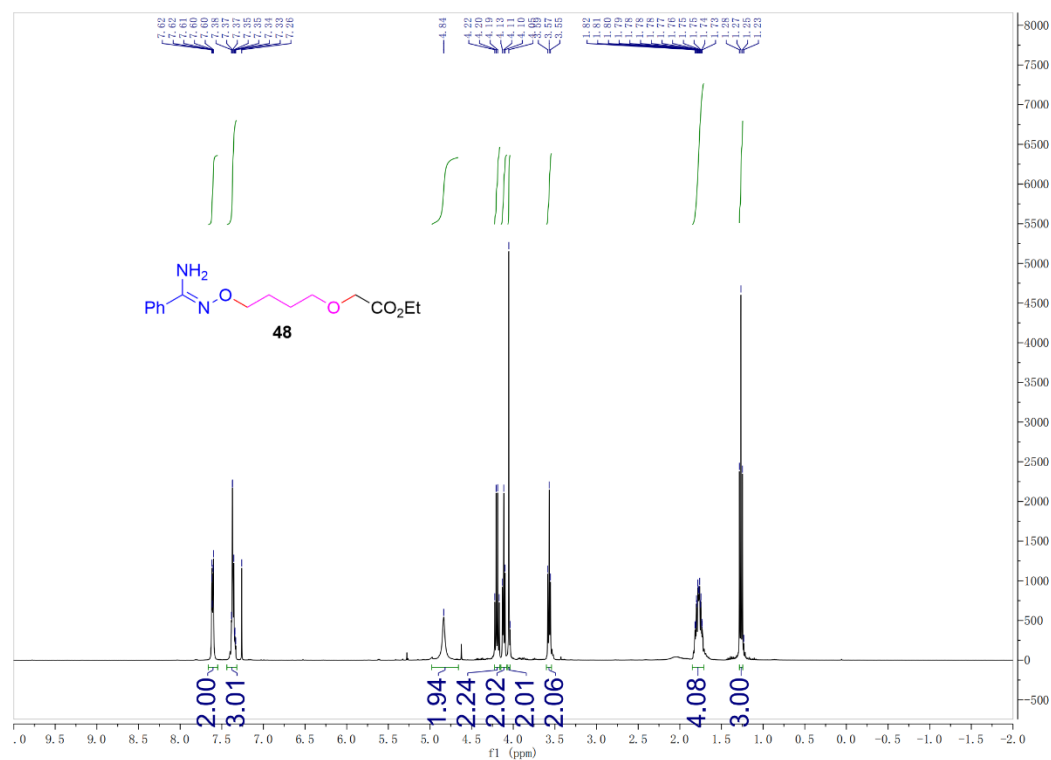
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 47**



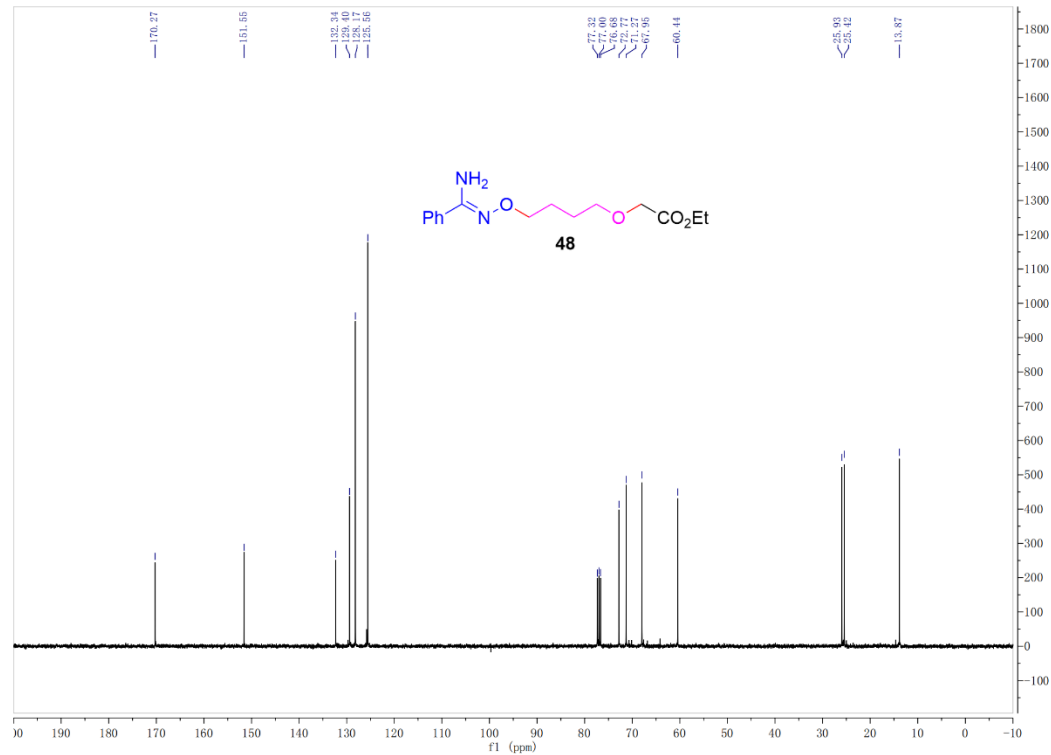
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 47**



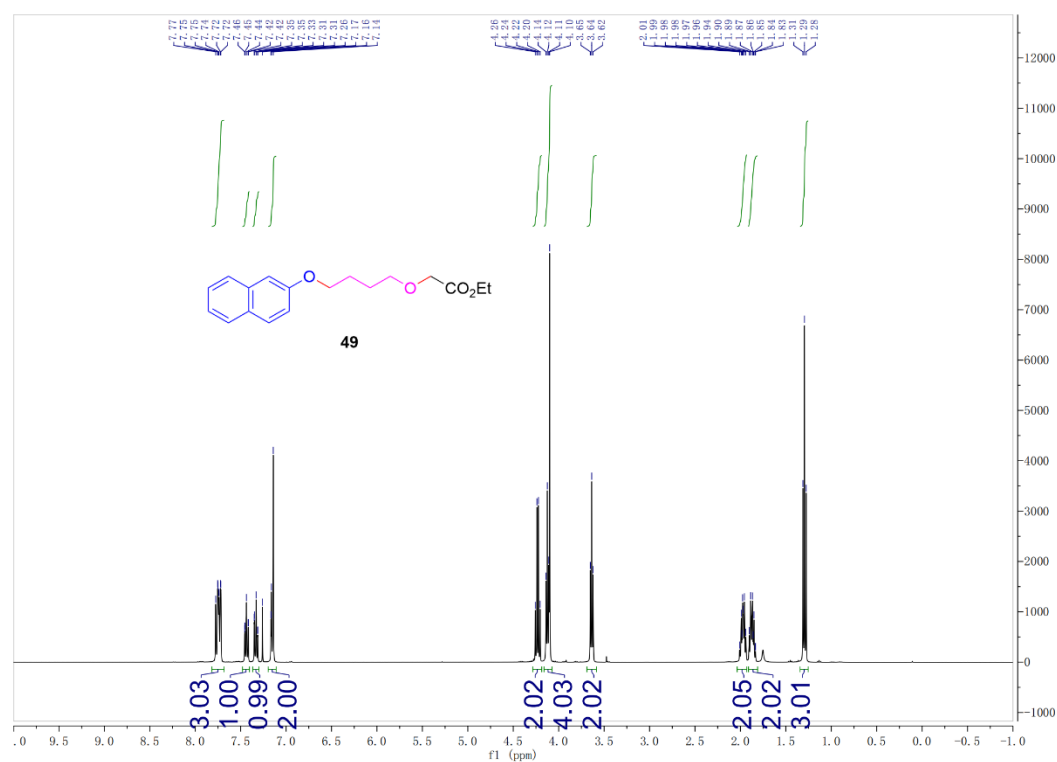
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 48**



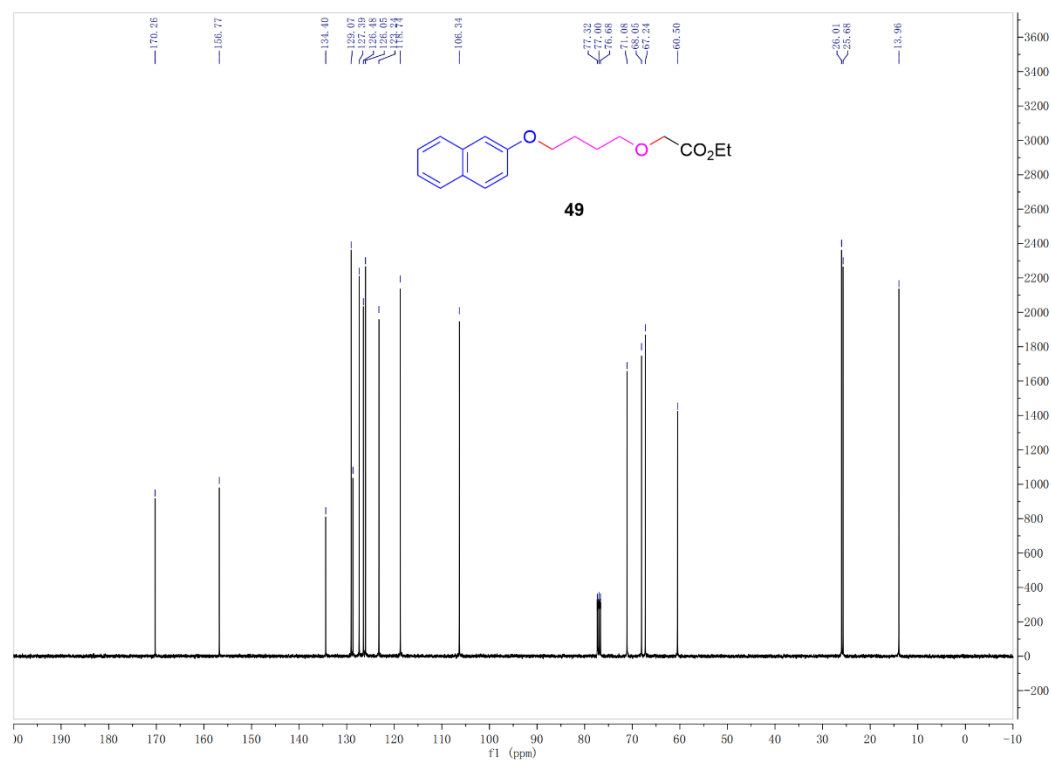
**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) Spectrum of 48**



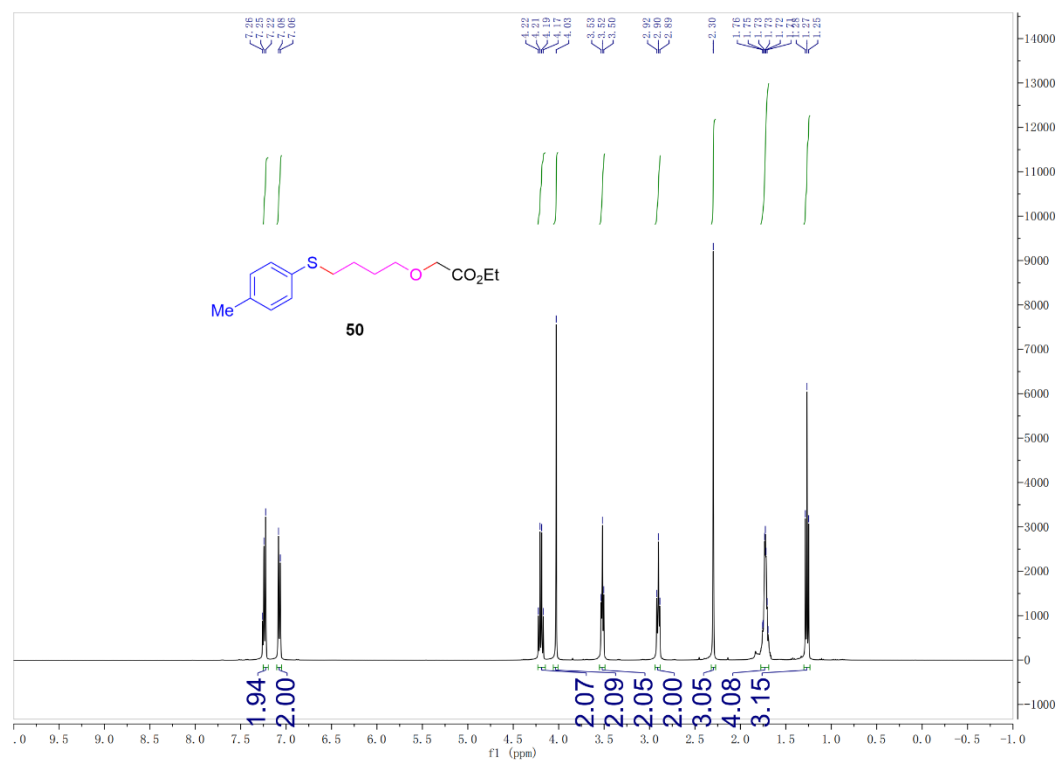
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 49**



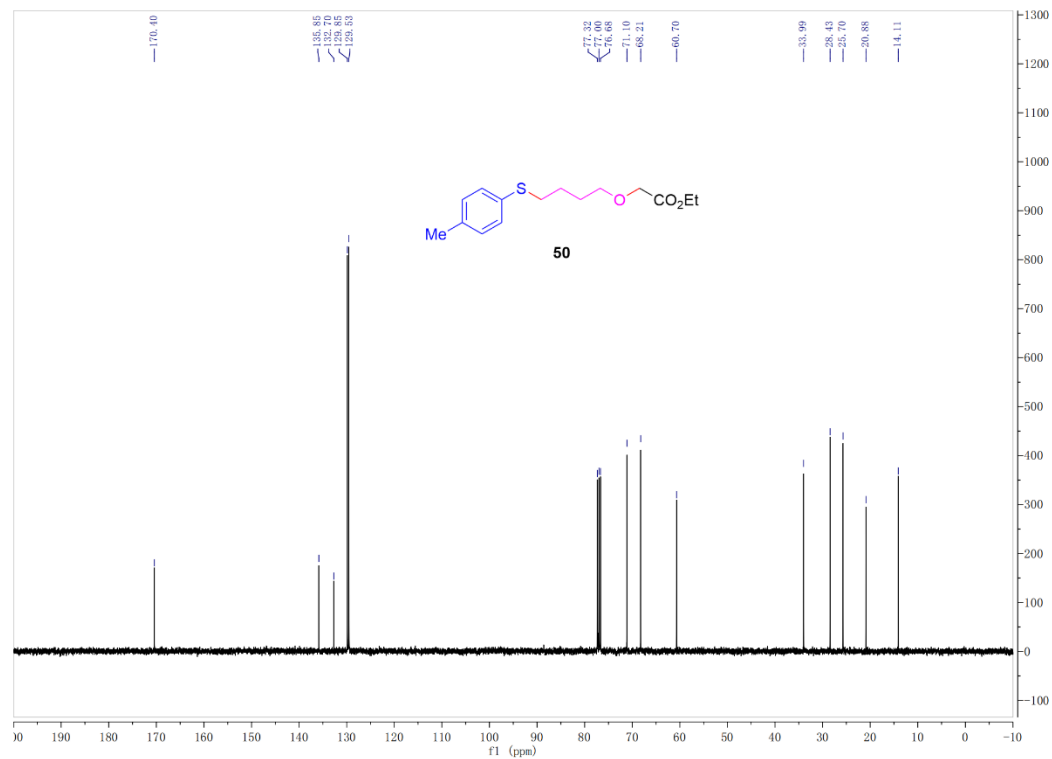
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 49**



**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 50**

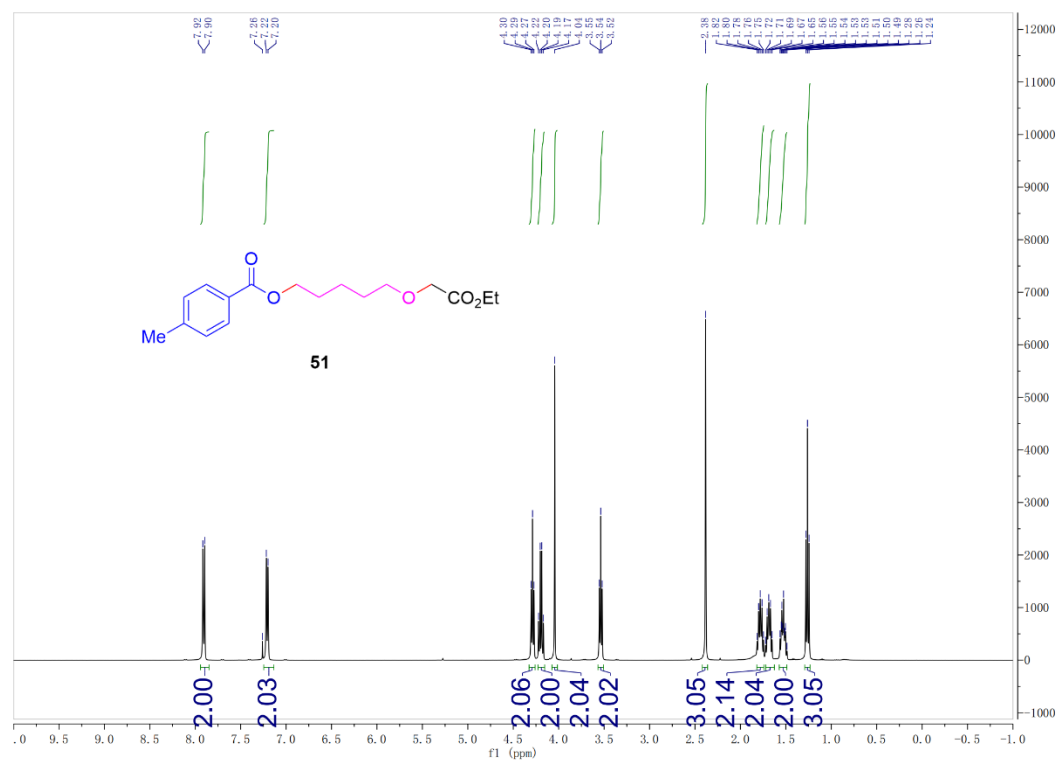


**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 50**

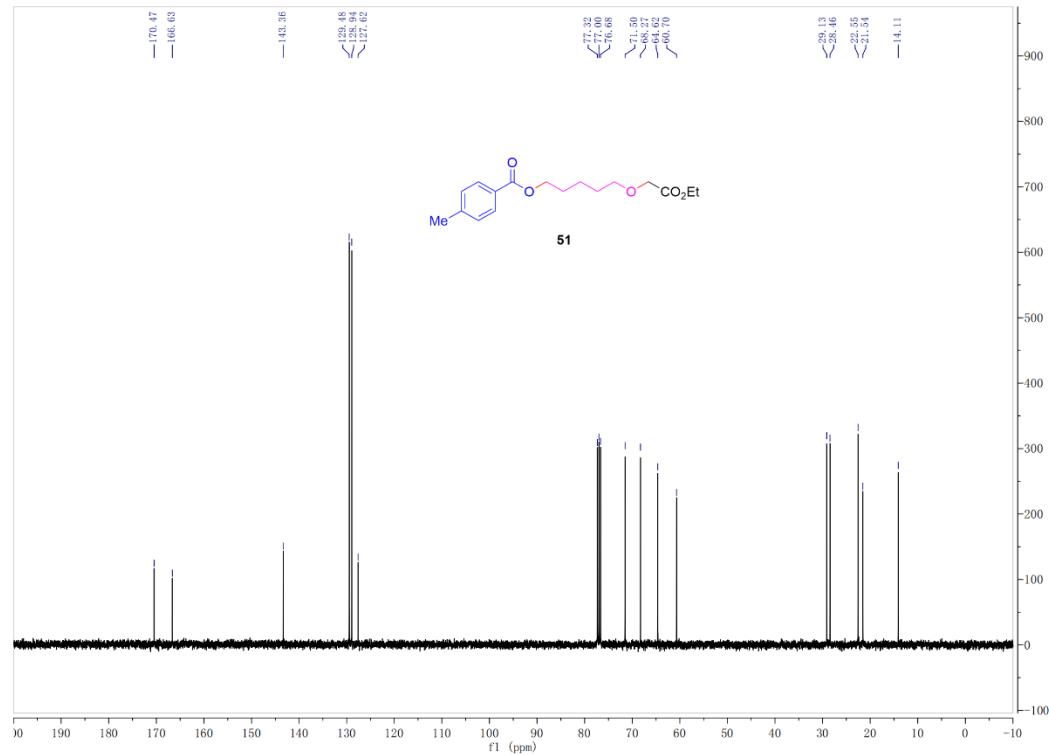




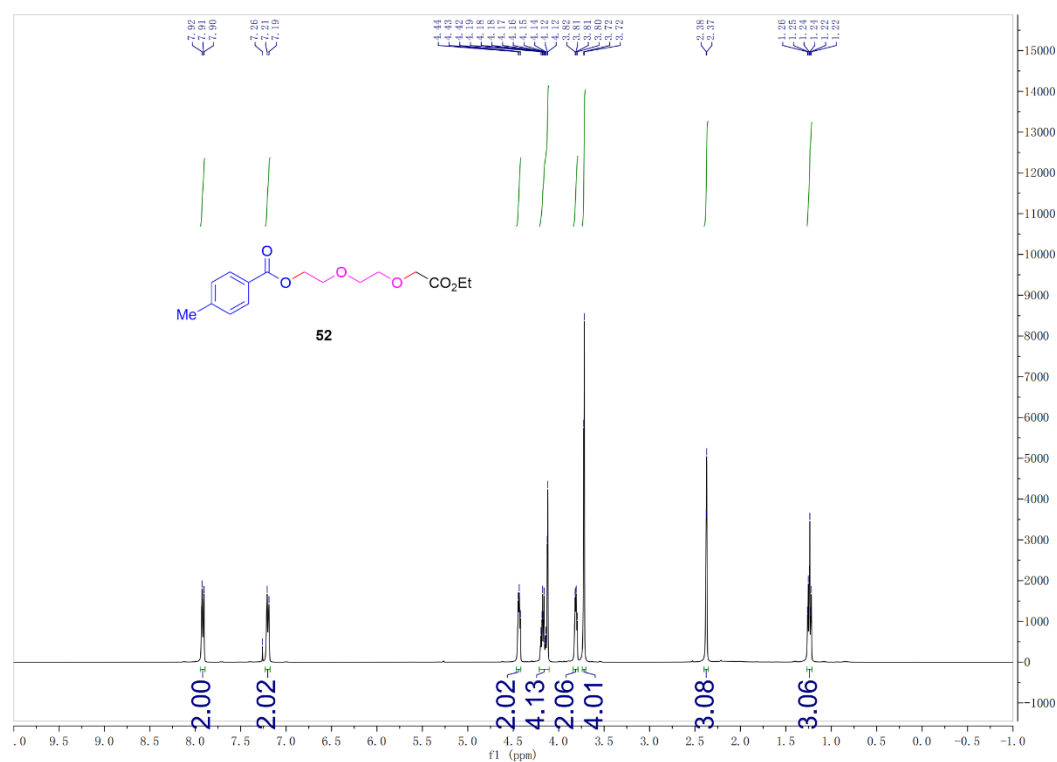
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 51**



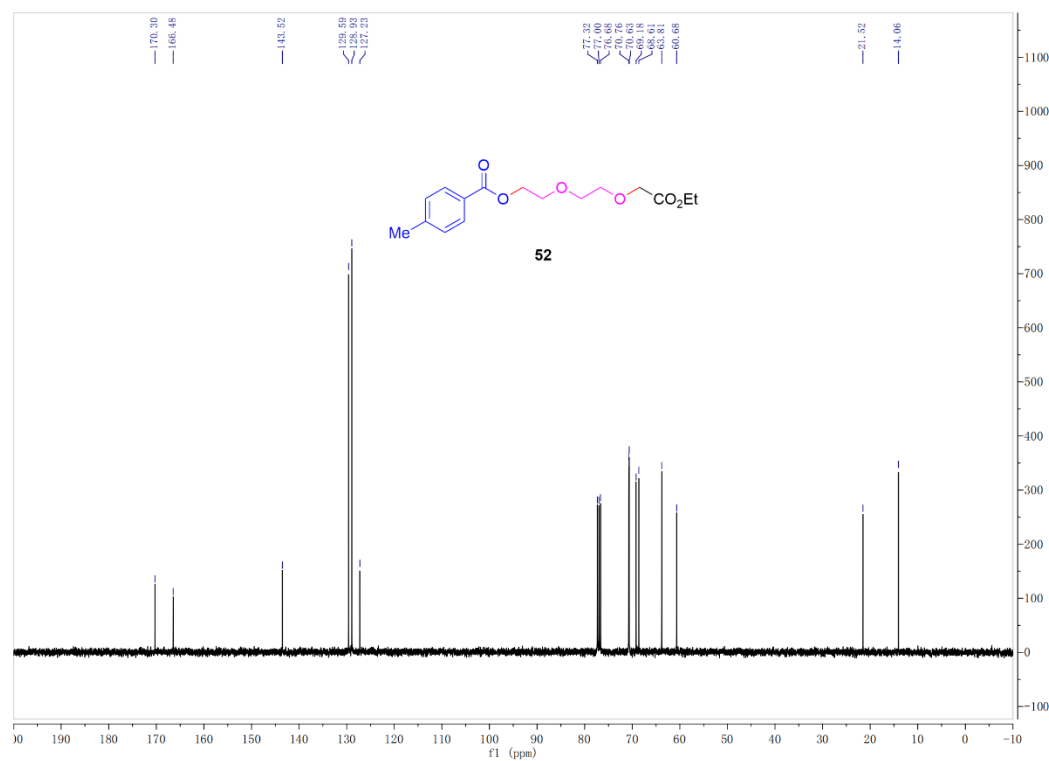
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 51**



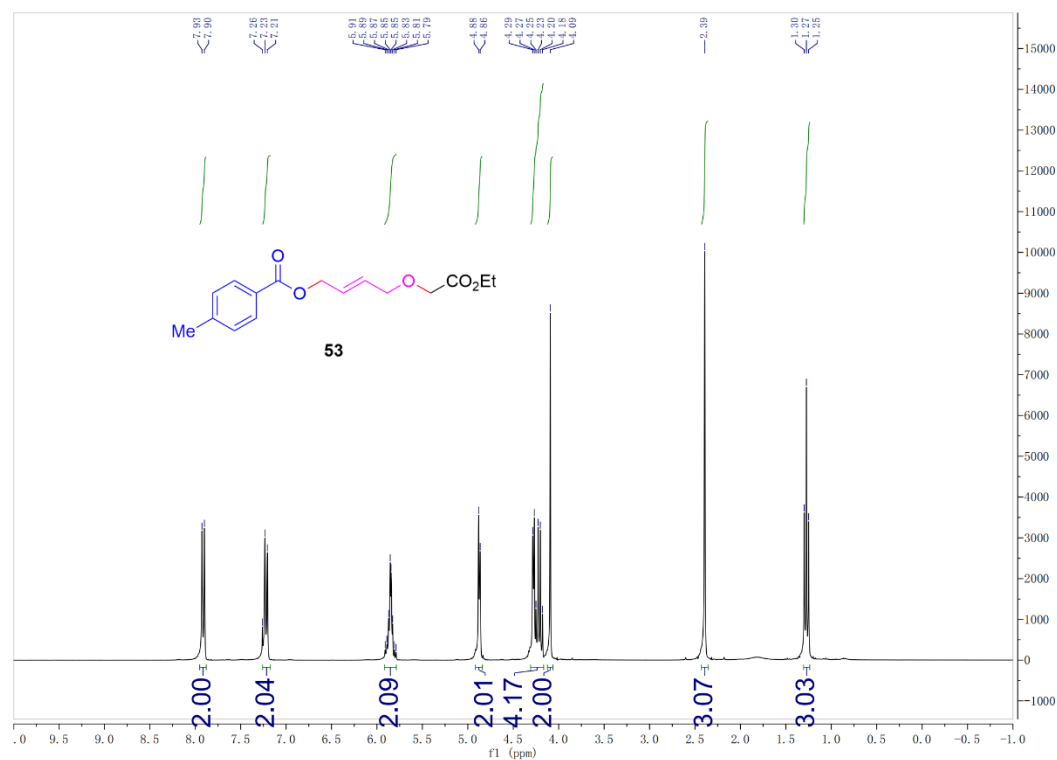
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 52**



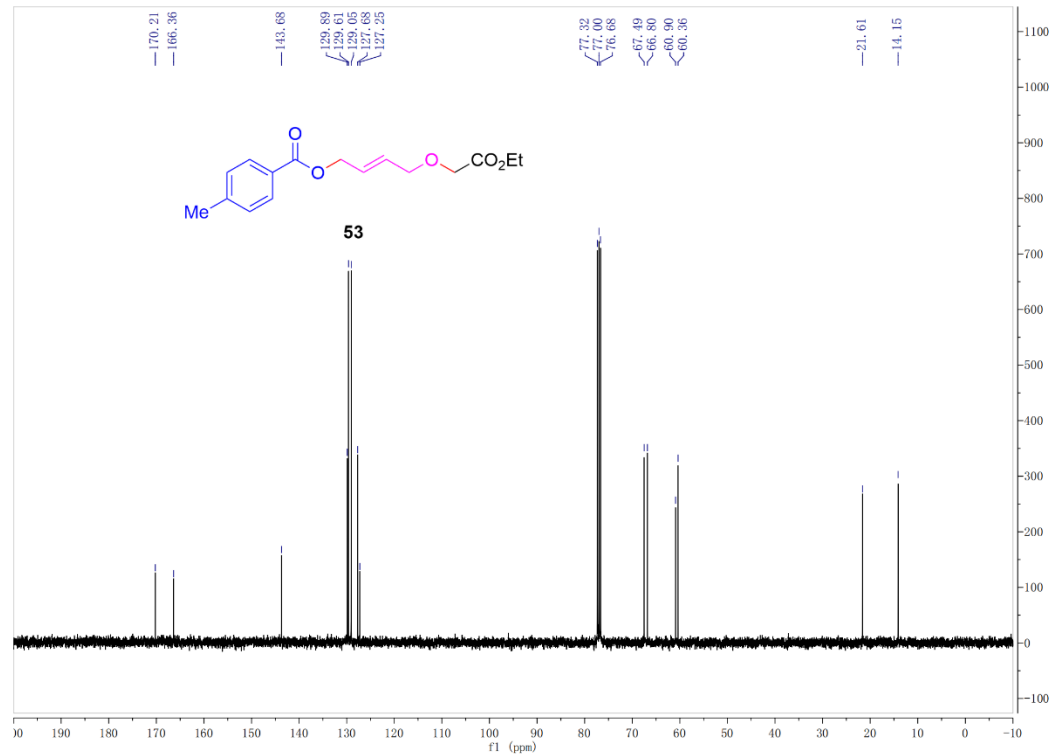
**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) Spectrum of 52**



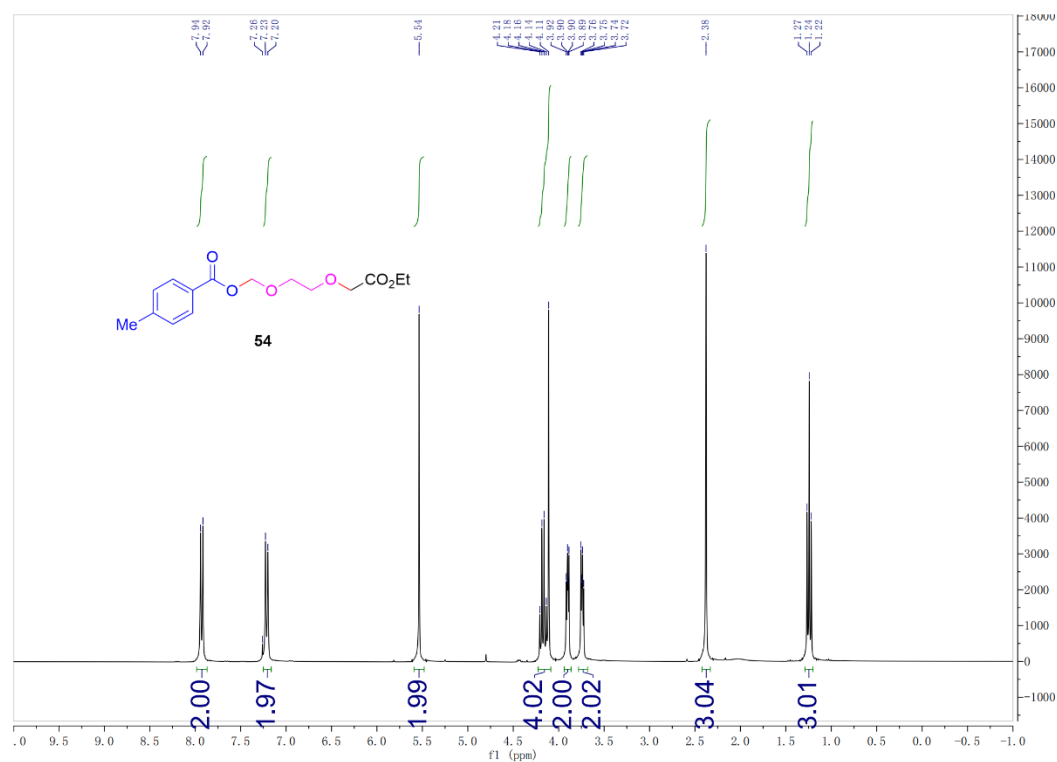
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 53**



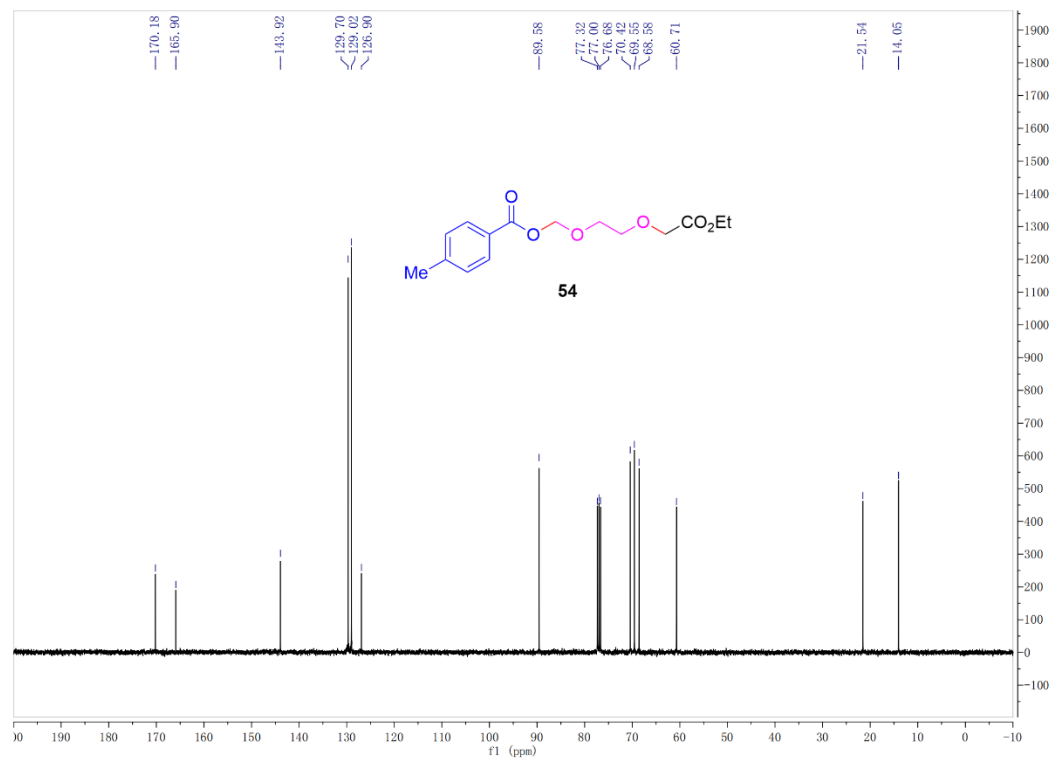
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 53**



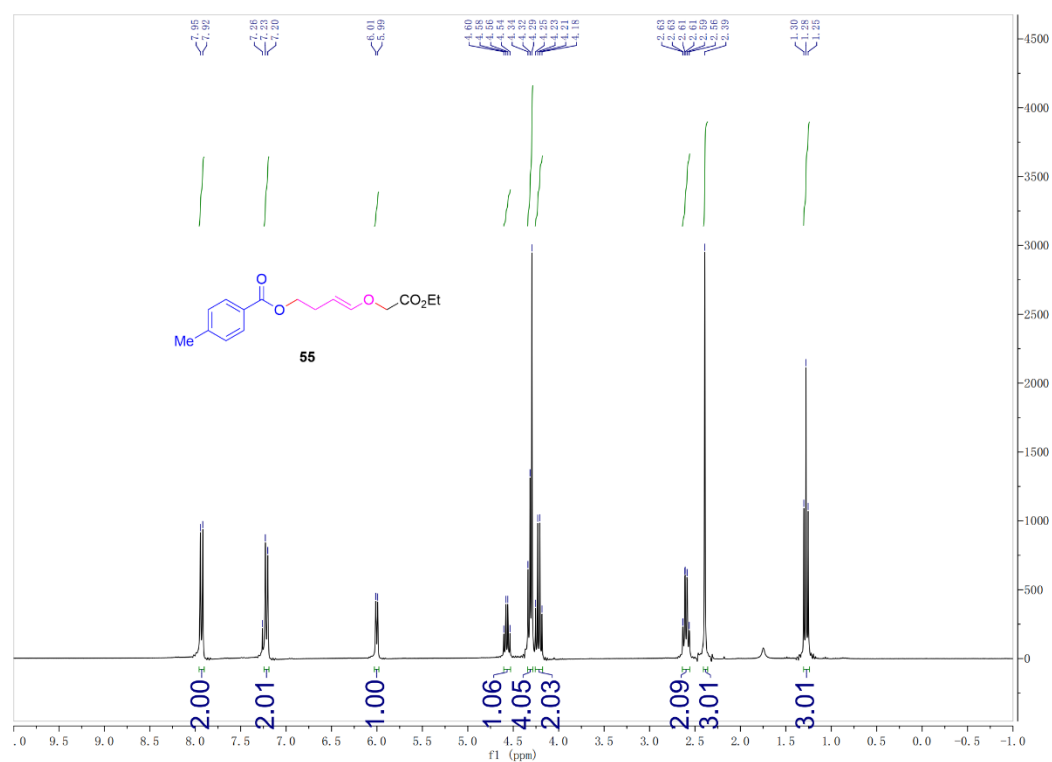
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 54**



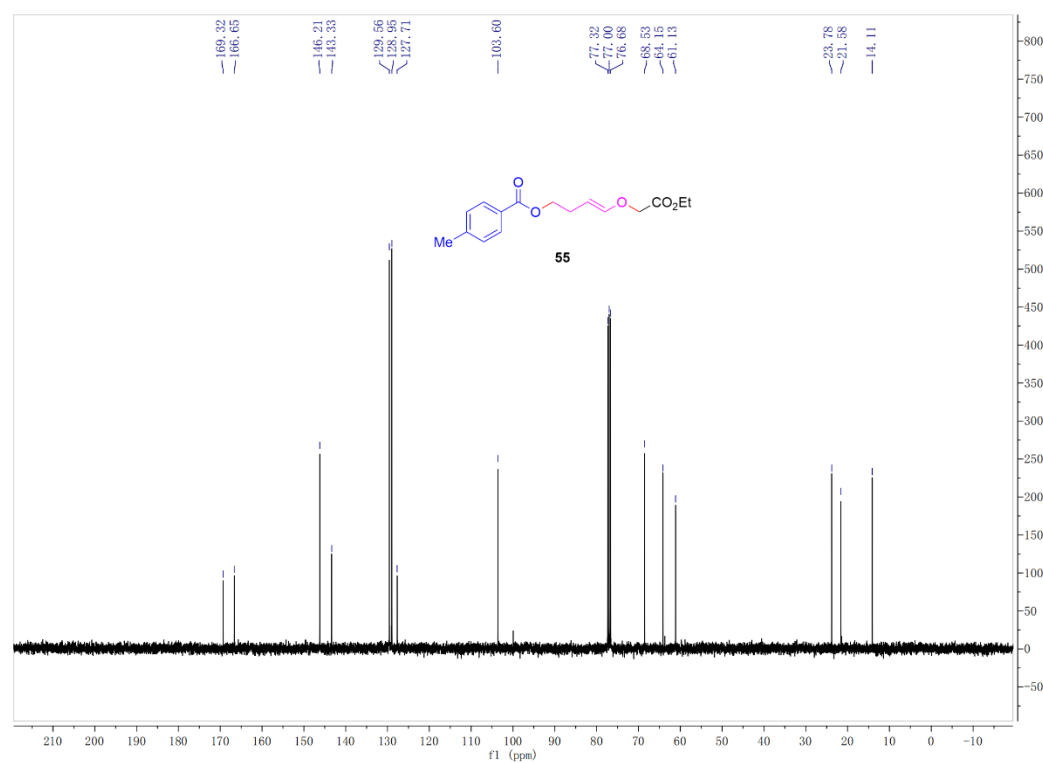
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 54**



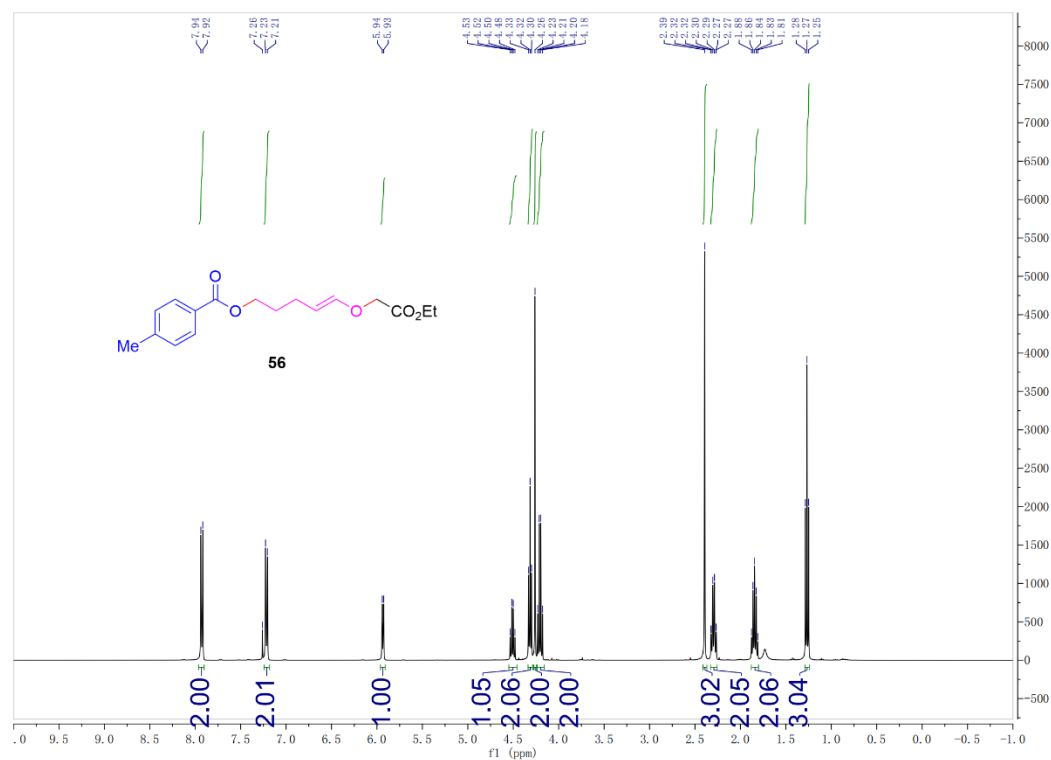
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 55**



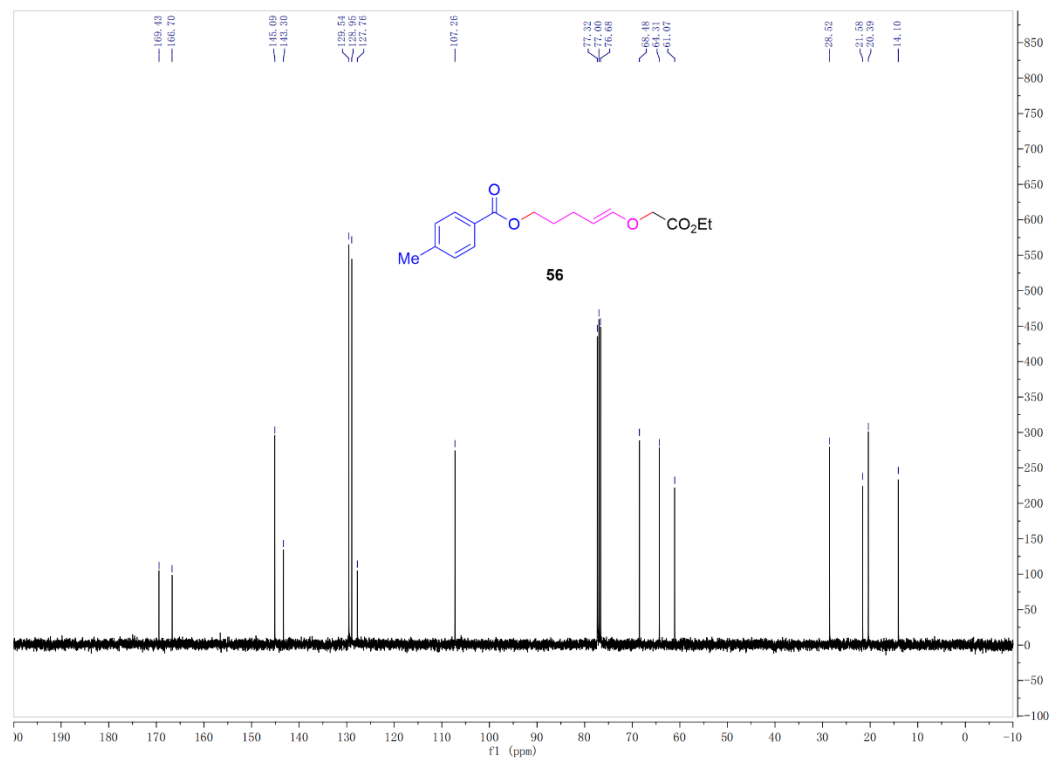
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 55**



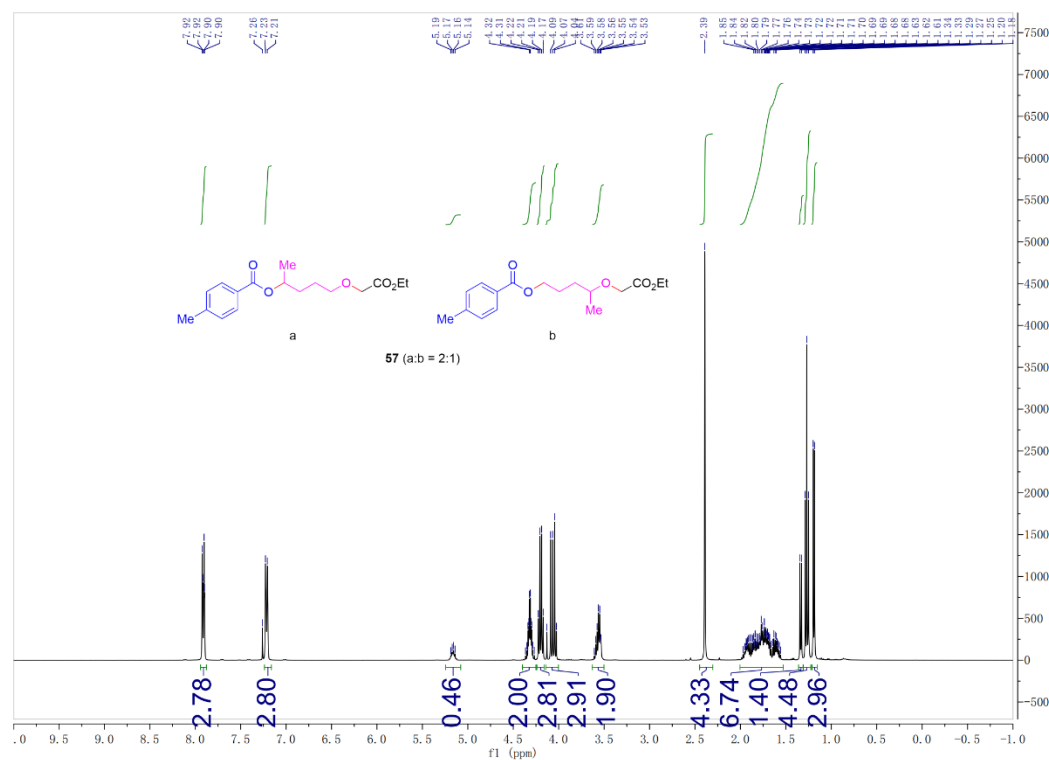
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 56**



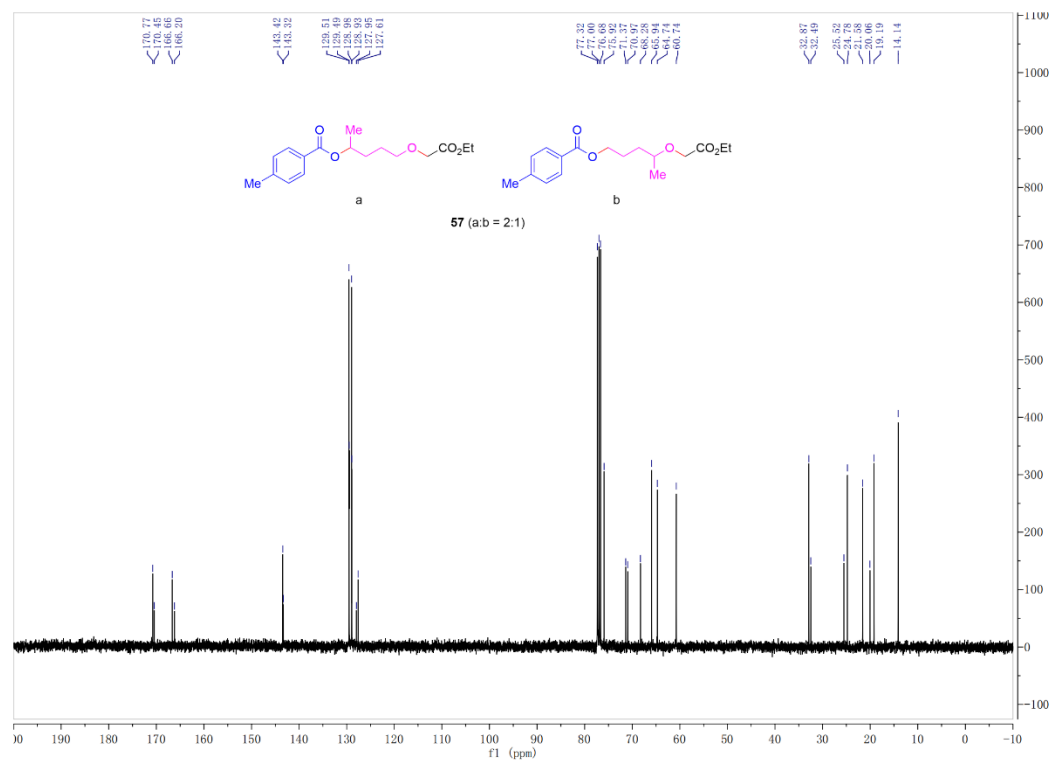
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 56**



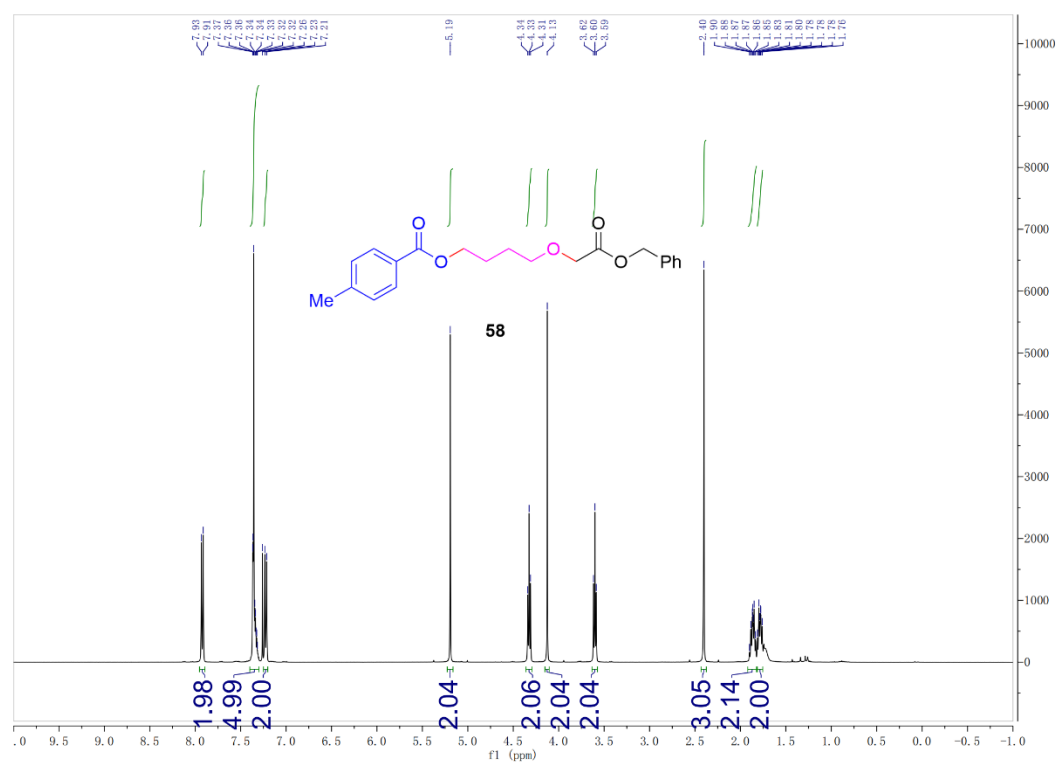
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) Spectrum of 57**



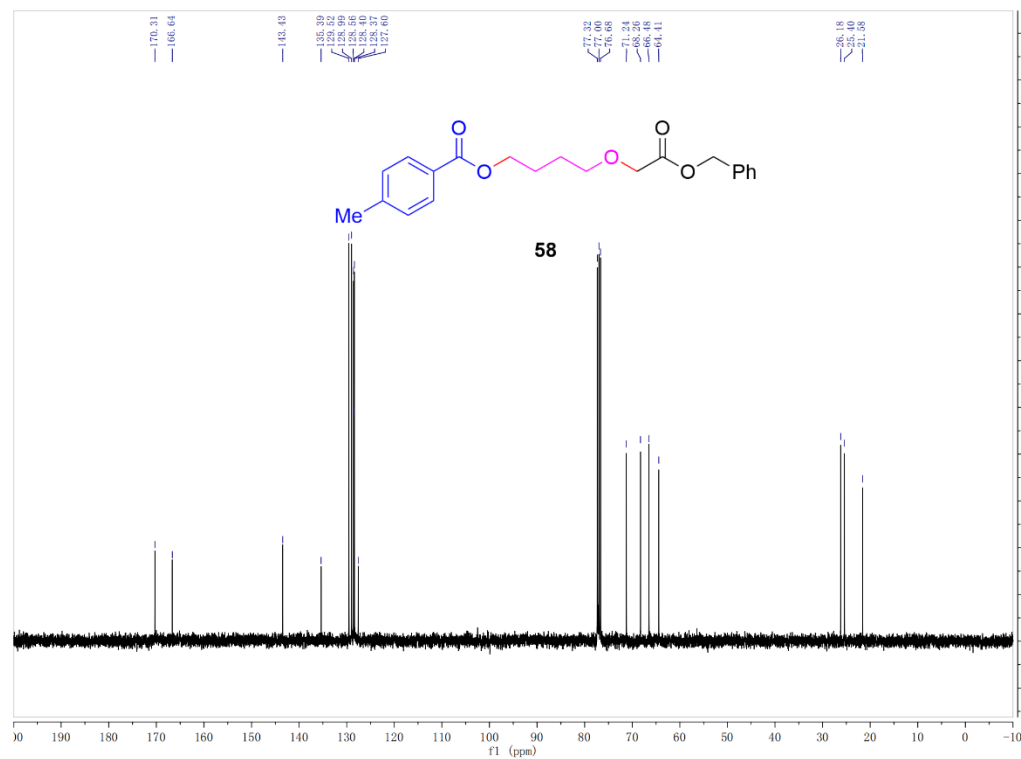
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 57**



**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 58**

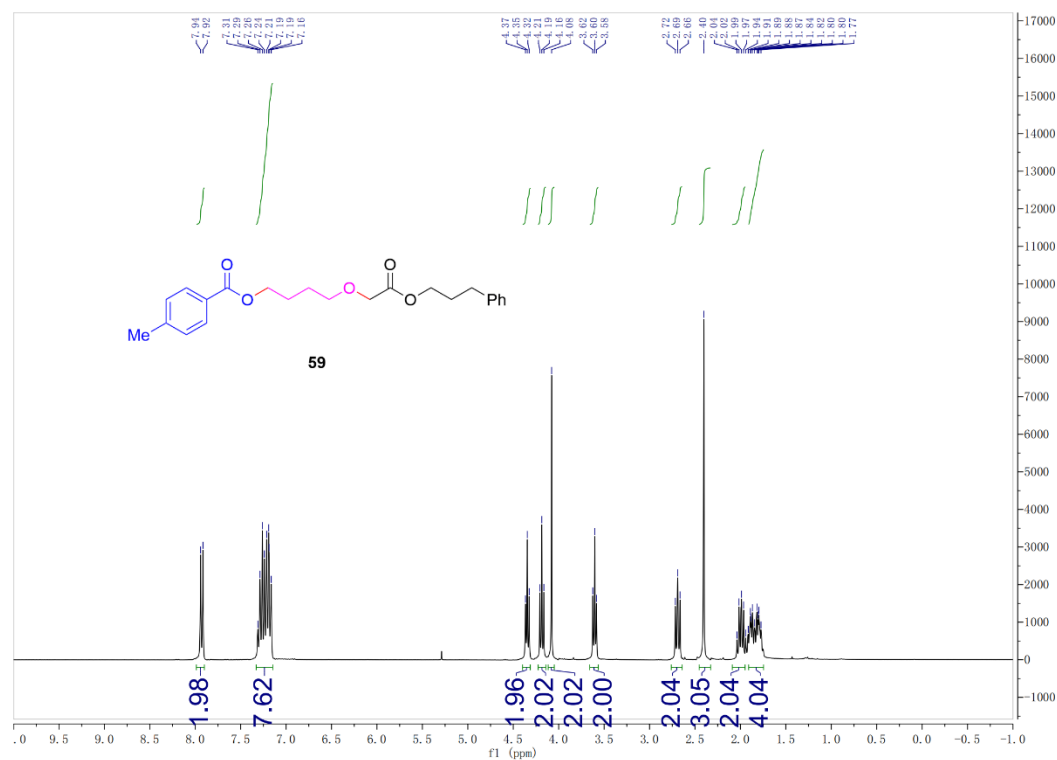


**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 58**

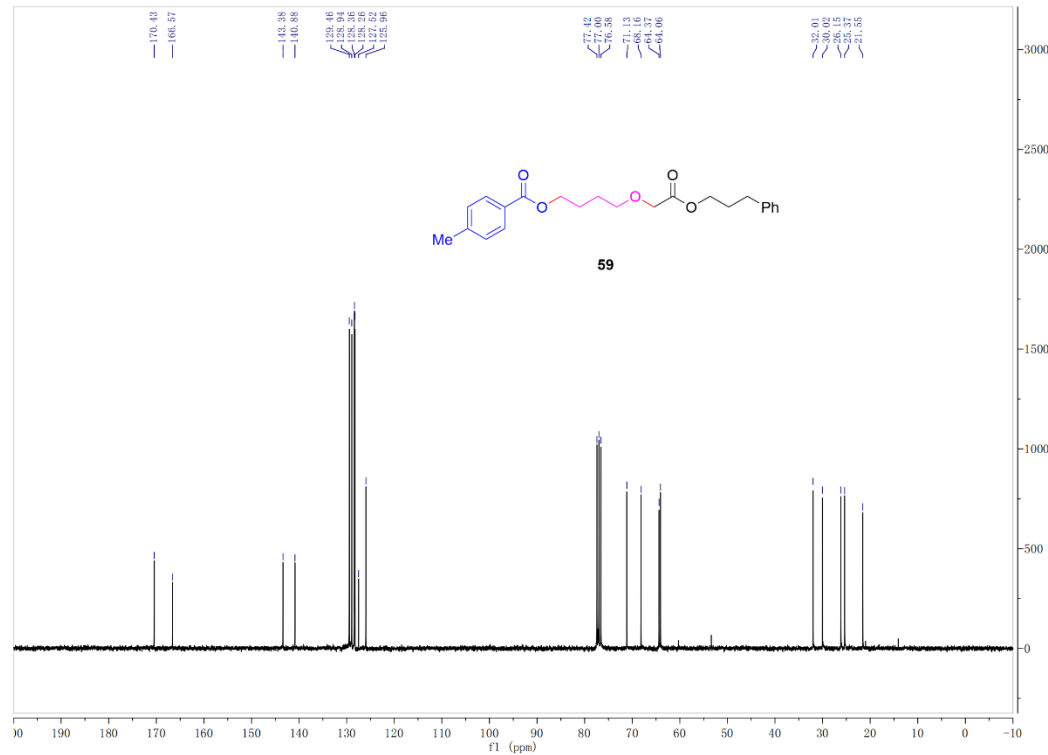




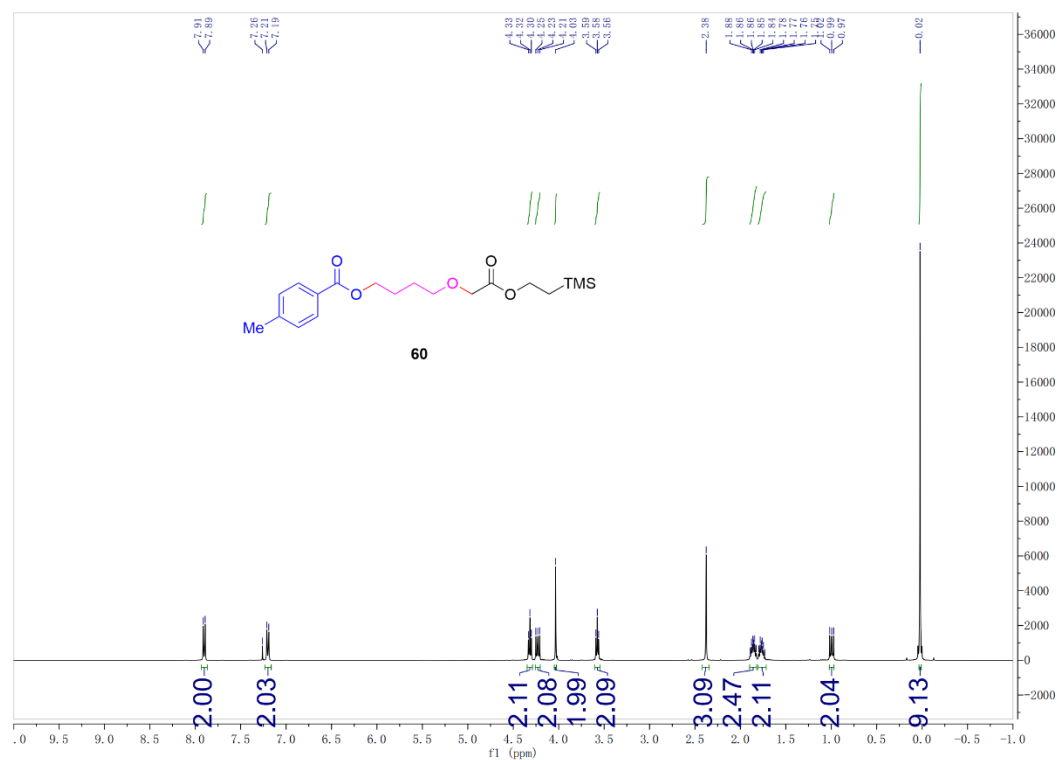
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 59**



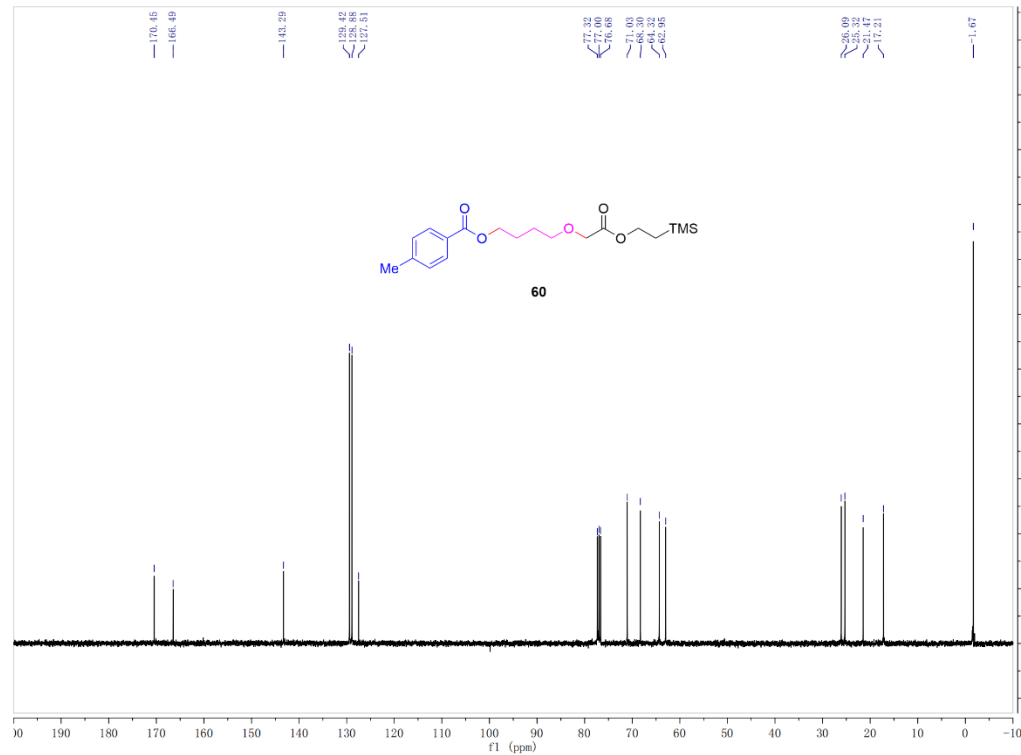
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 59**



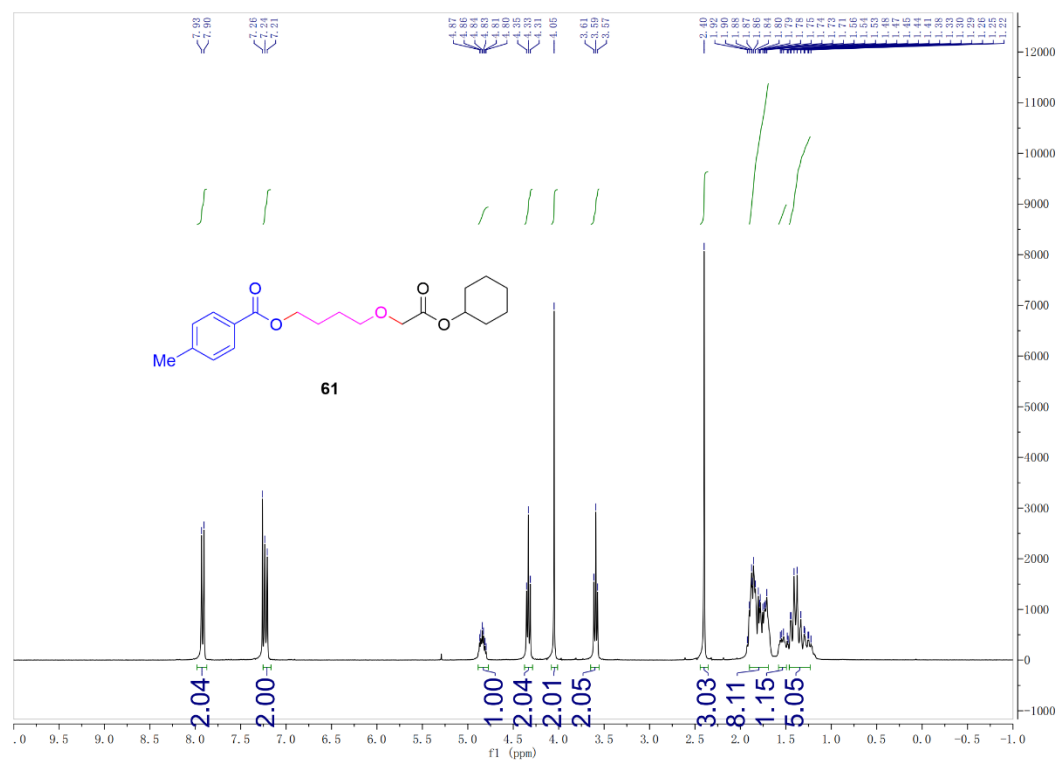
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 60**



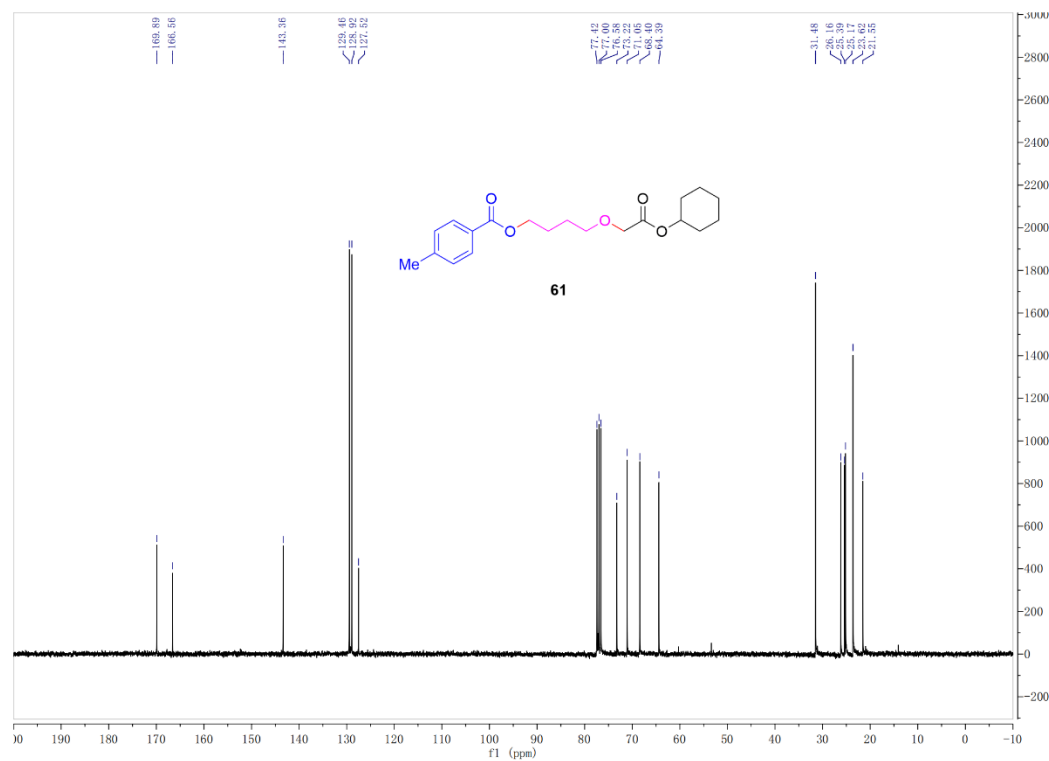
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 60**



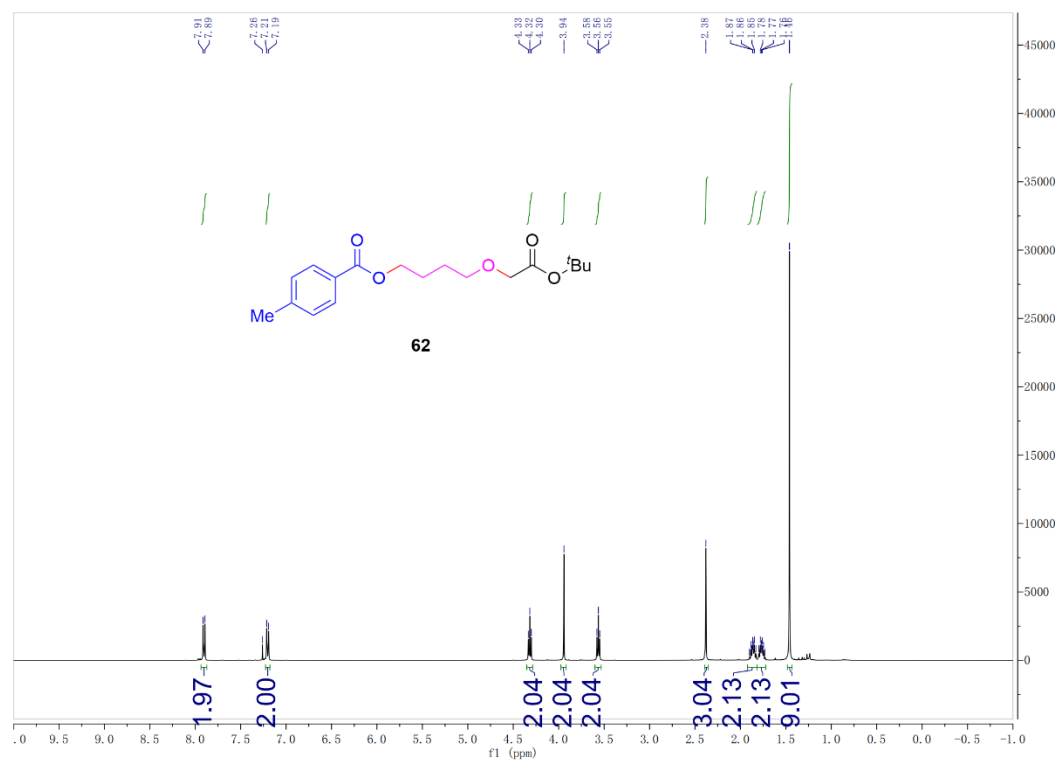
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 61**



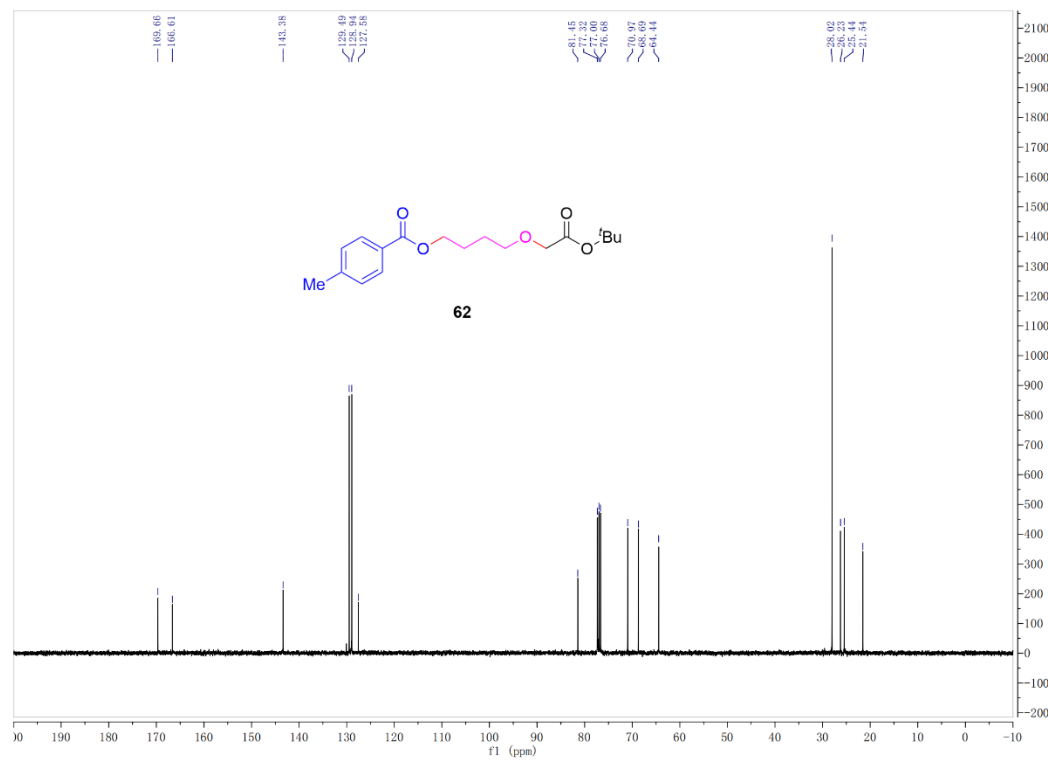
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 61**



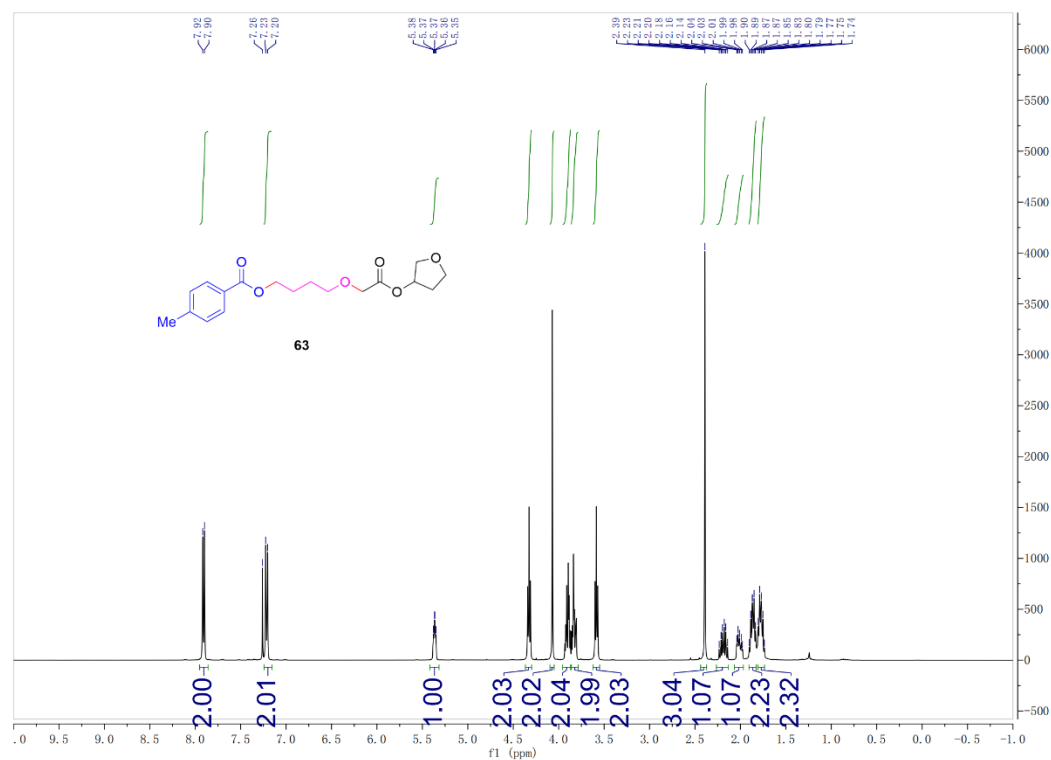
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 62**



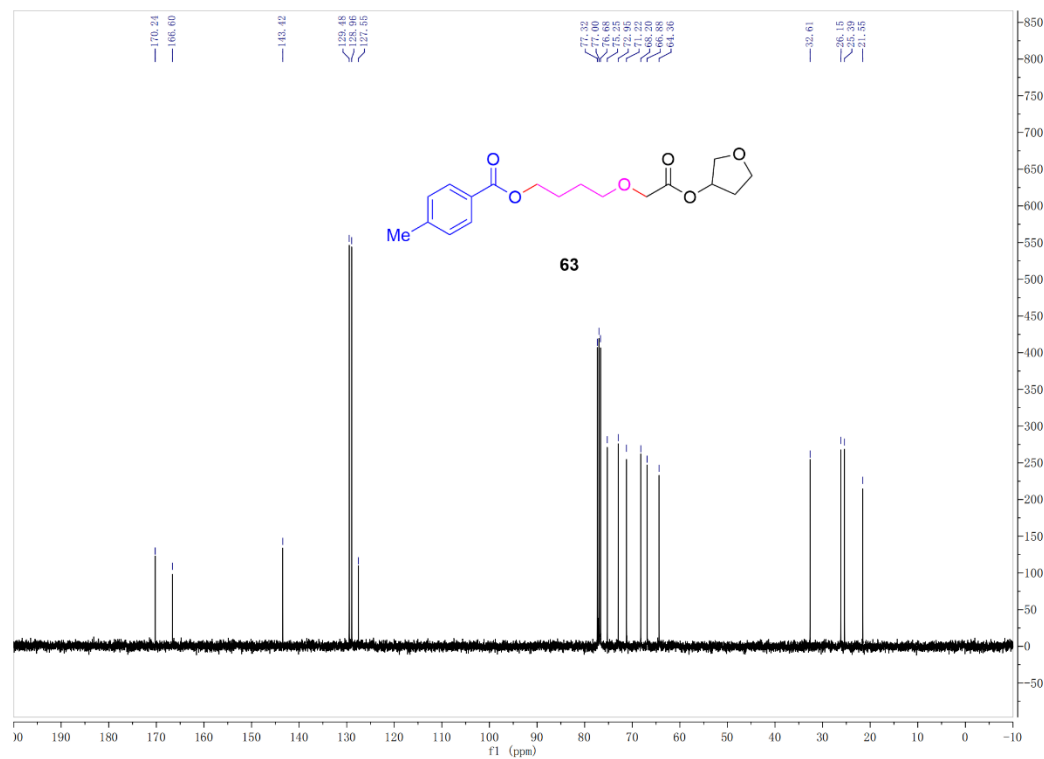
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 62**



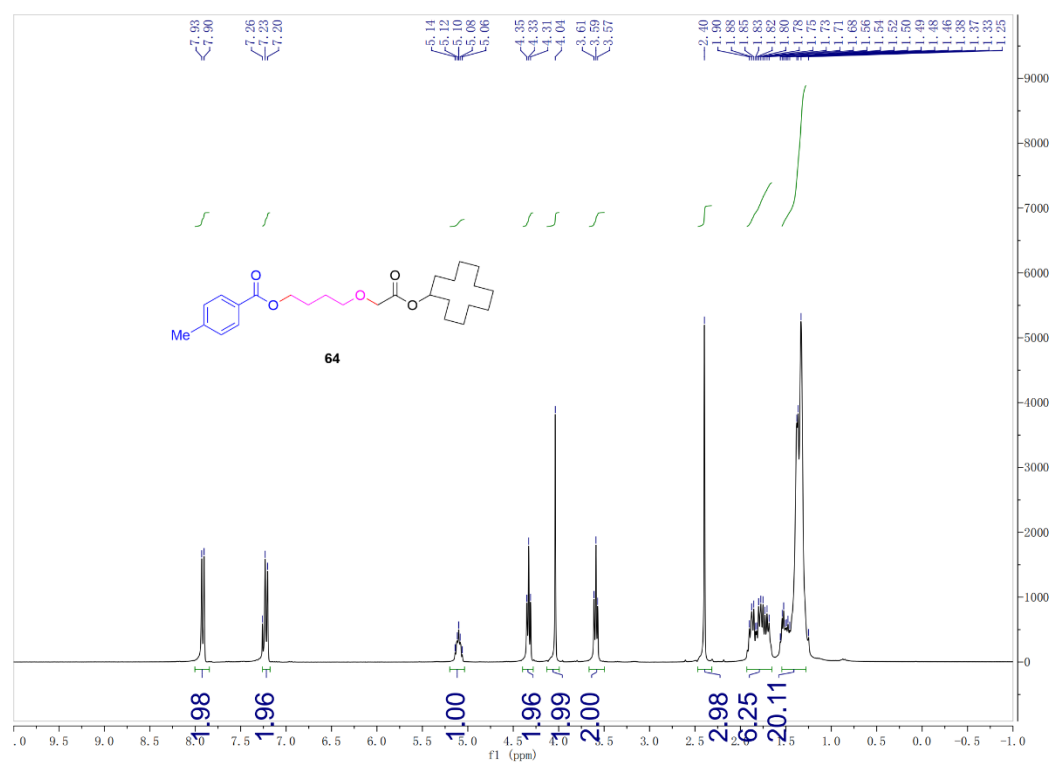
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 63**



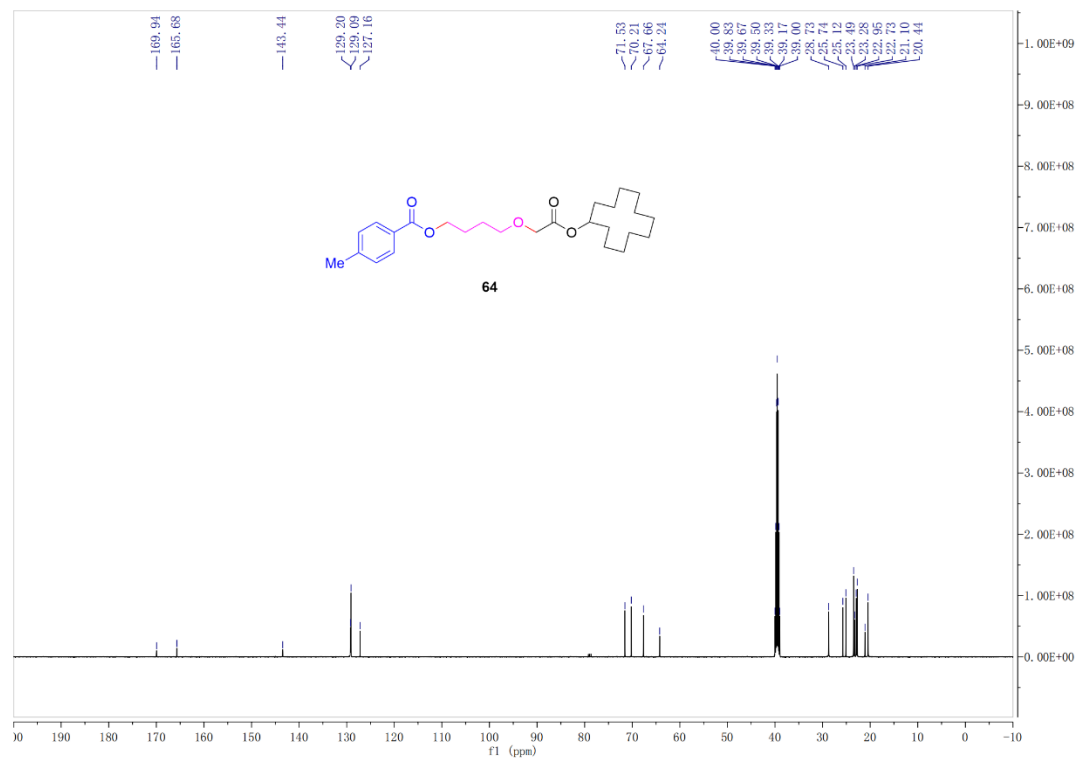
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 63**



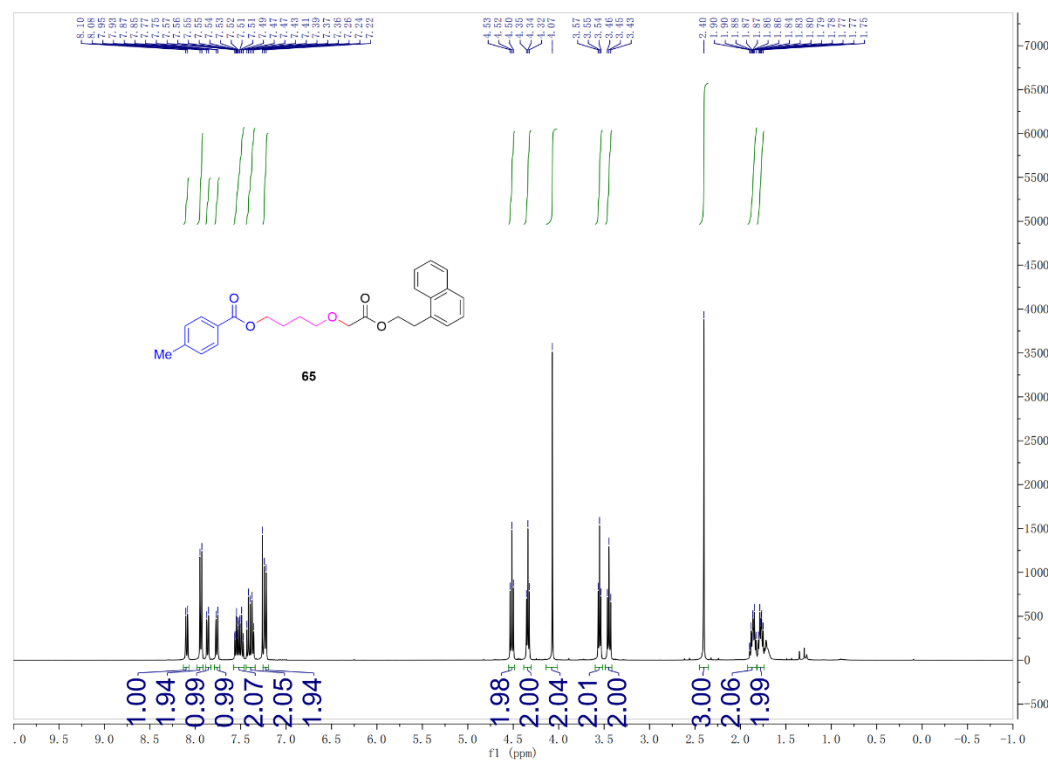
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 64**



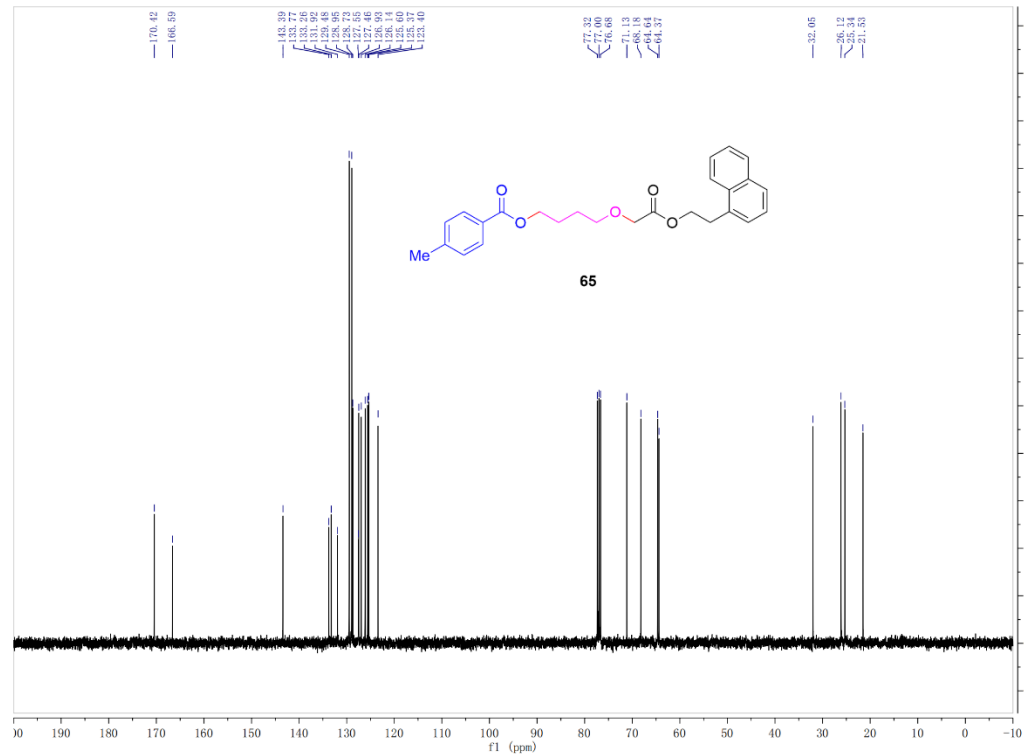
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $d_6$ -DMSO) Spectrum of 64**



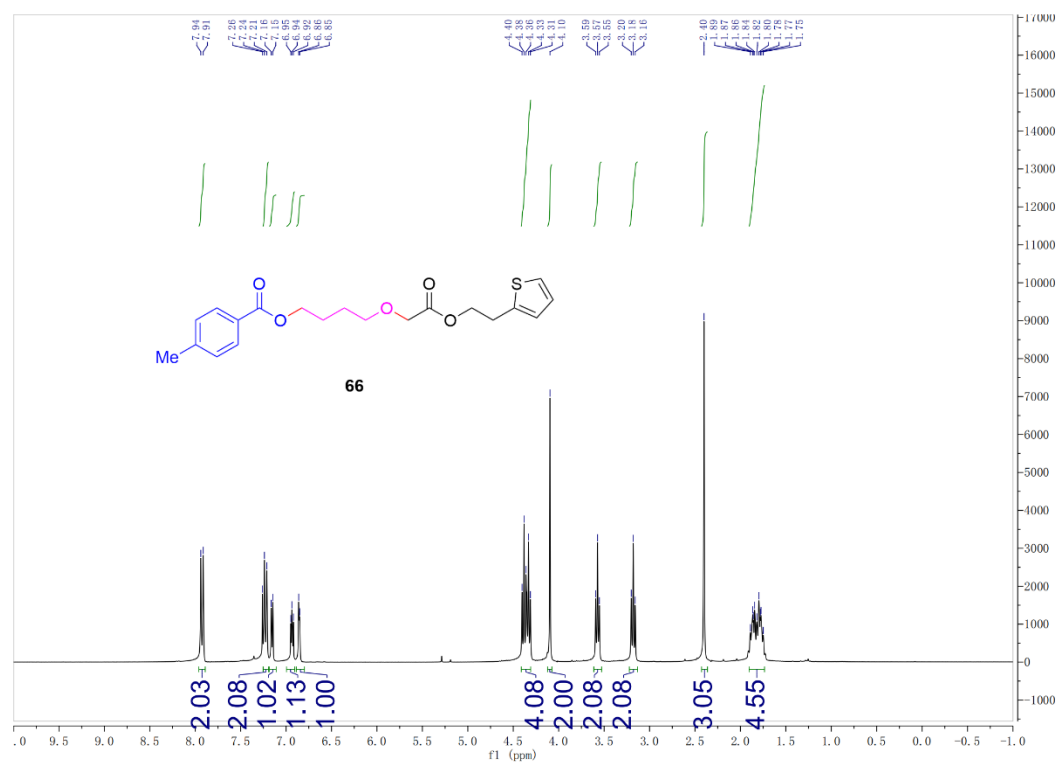
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) Spectrum of 65**



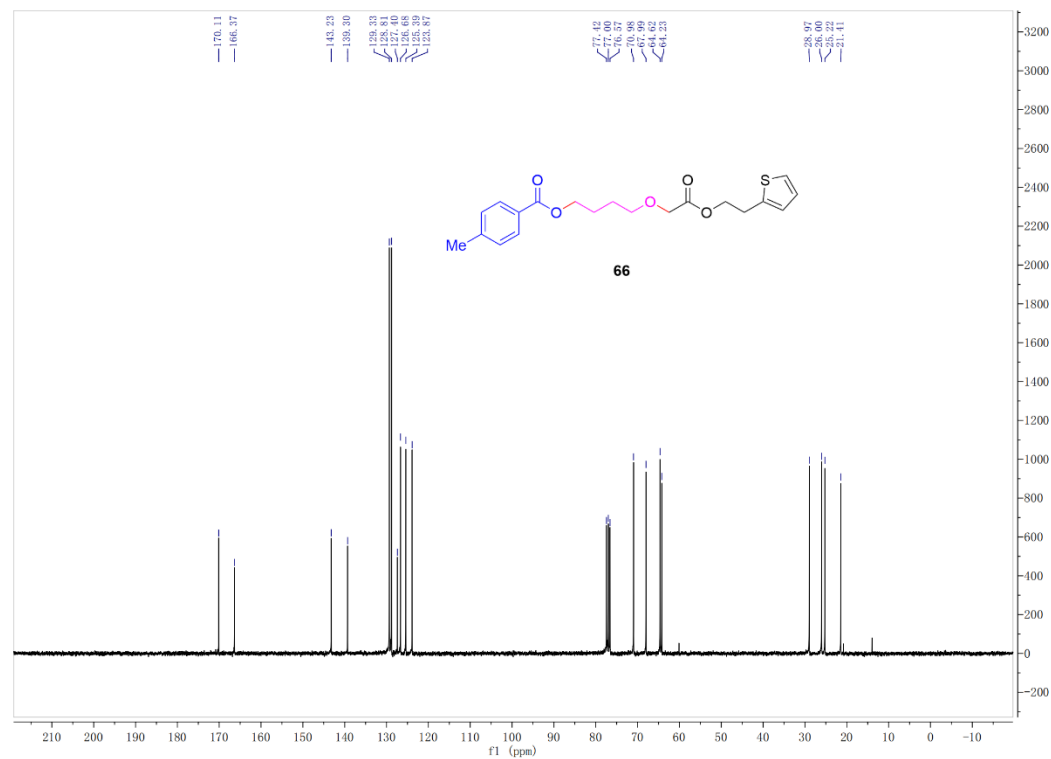
**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) Spectrum of 65**



**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 66**

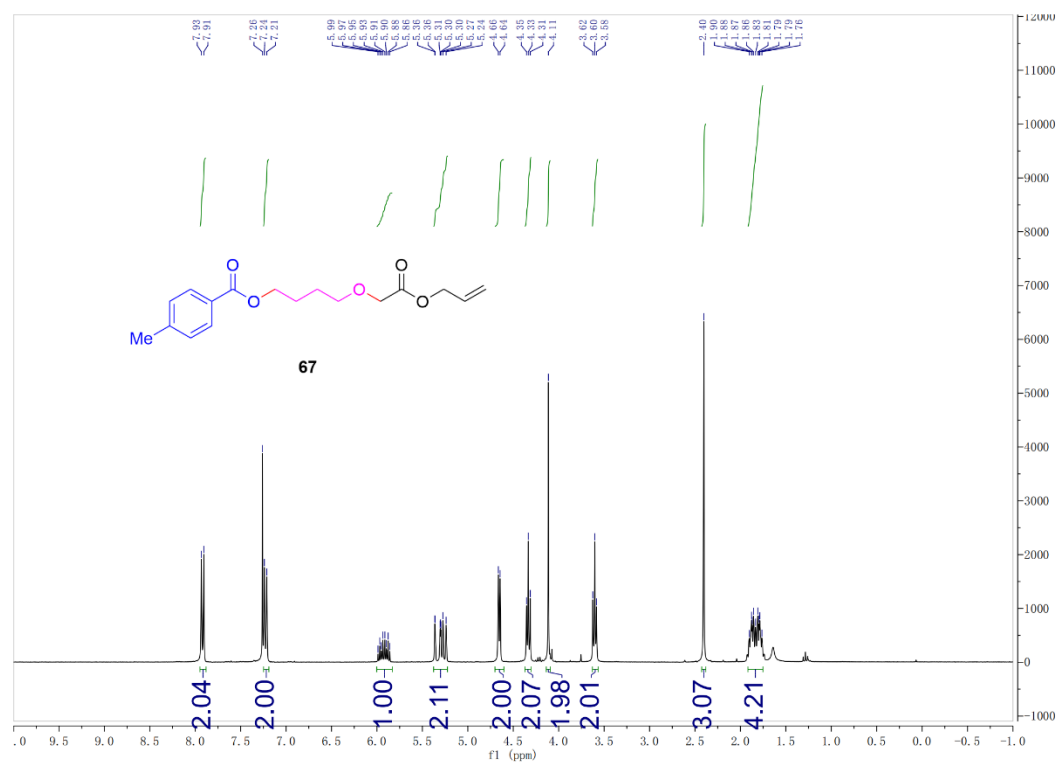


**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 66**

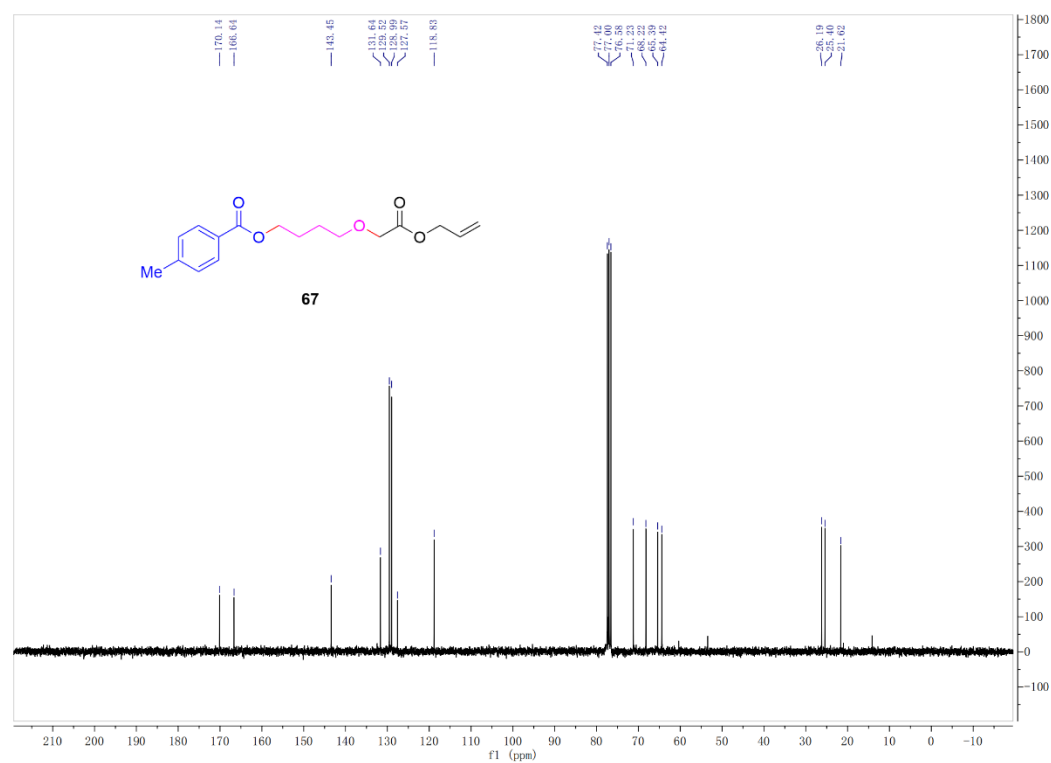




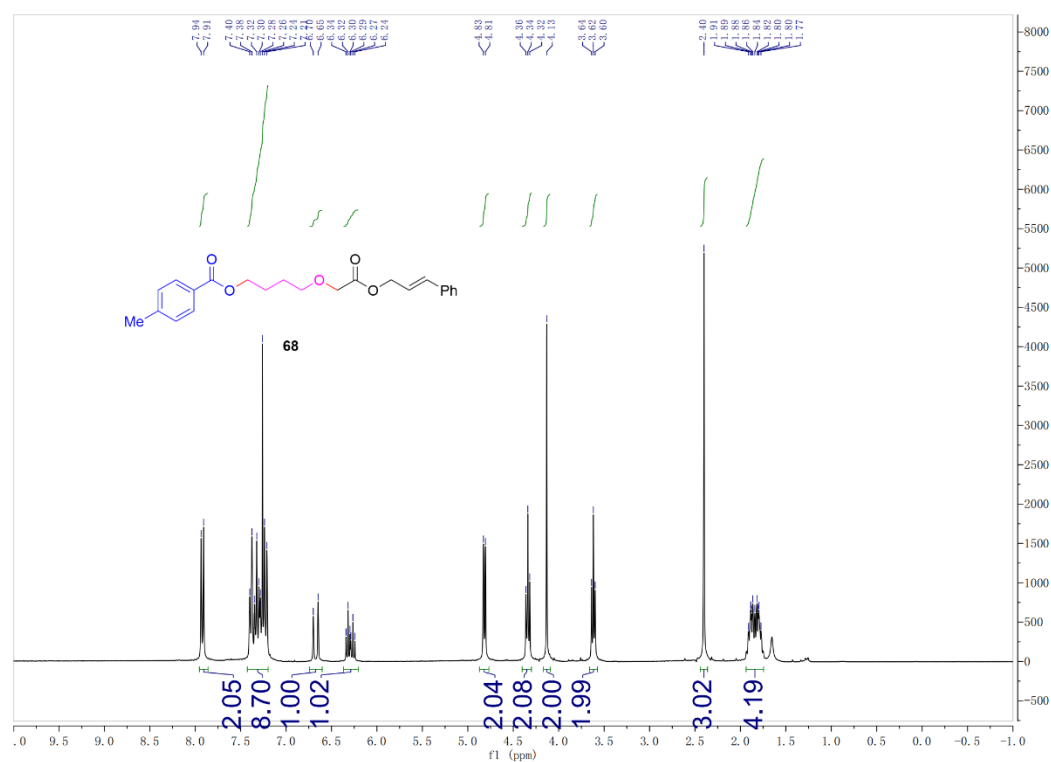
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 67**



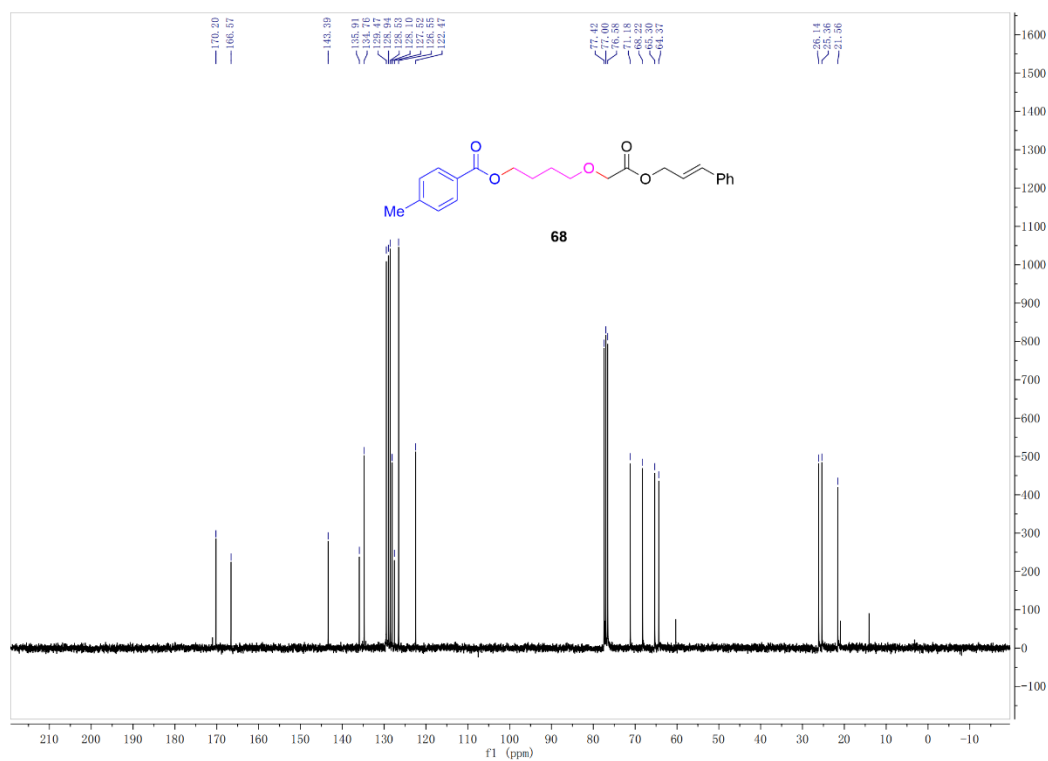
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 67**



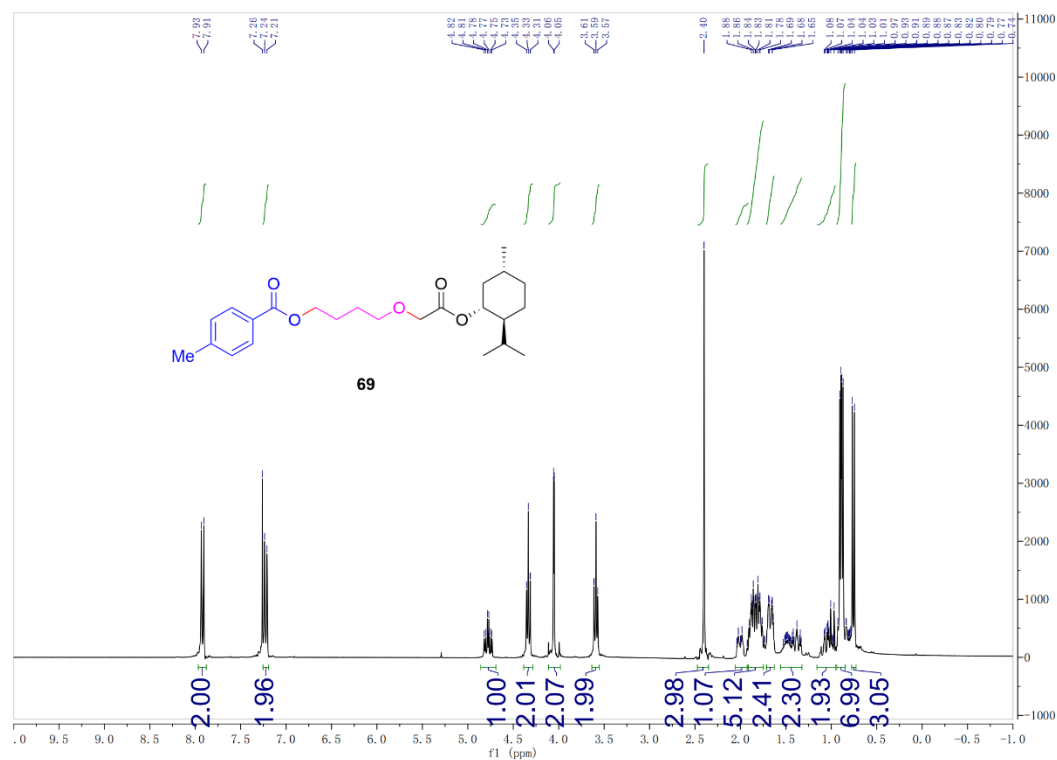
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 68**



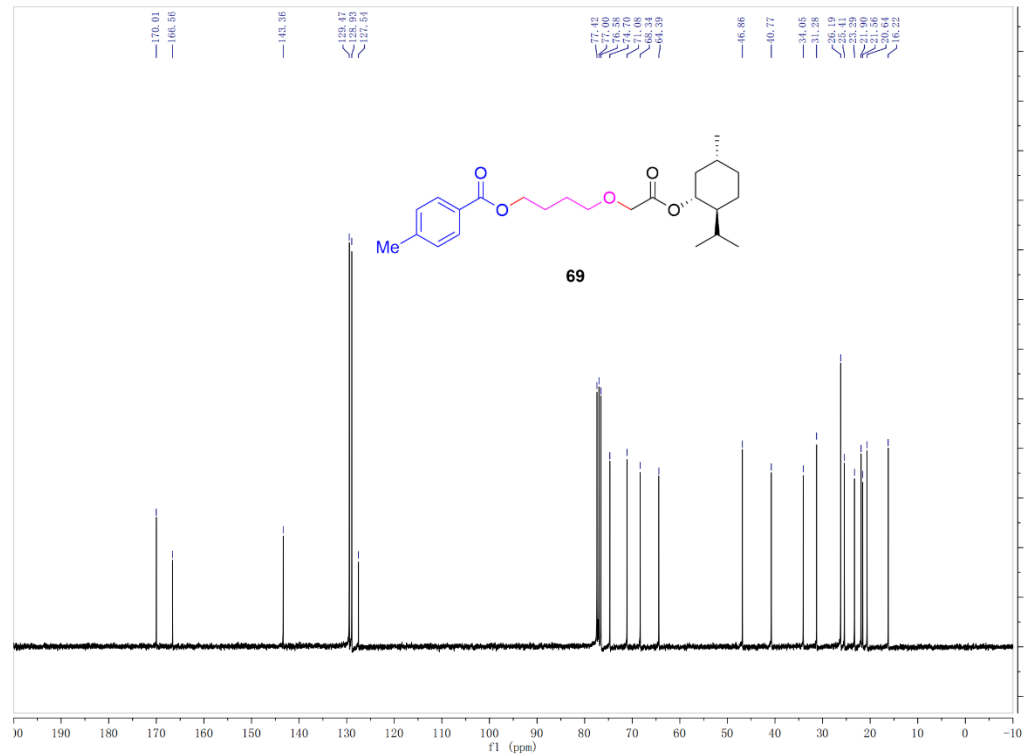
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 68**



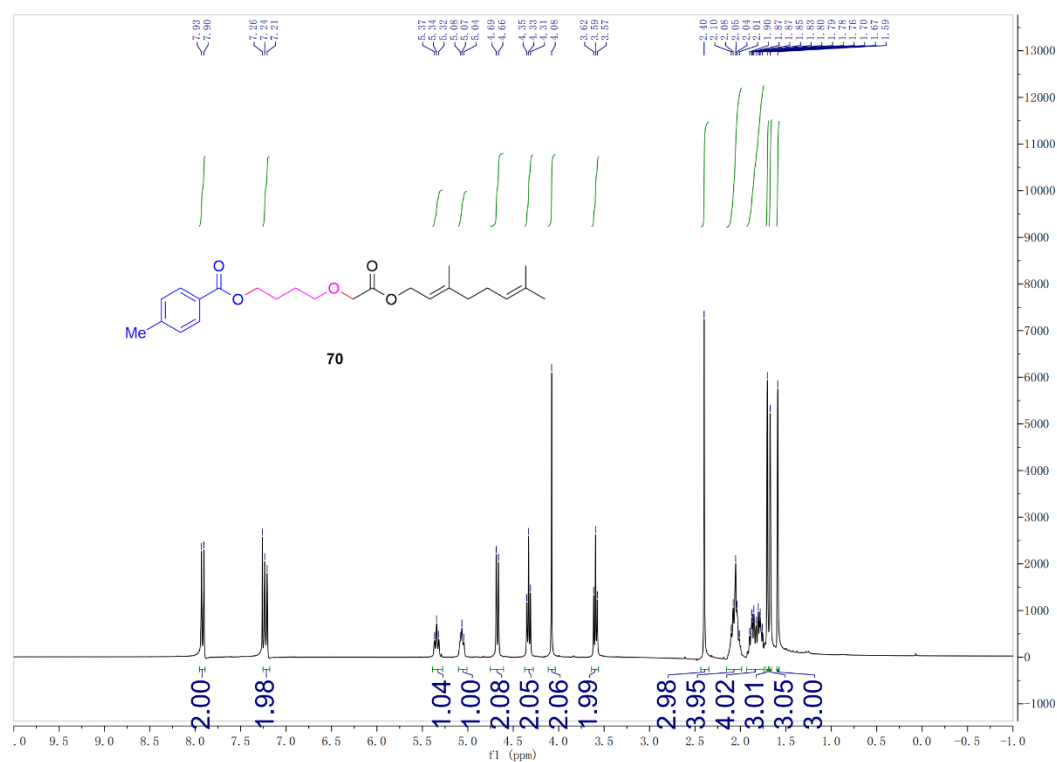
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 69**



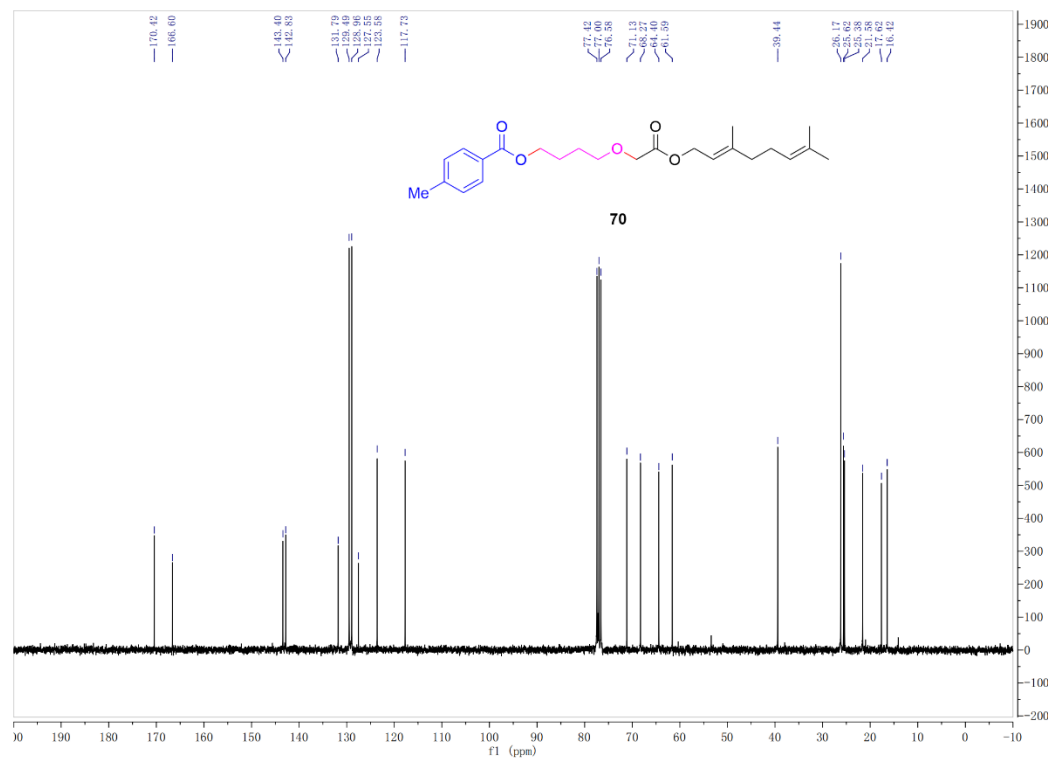
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 69**



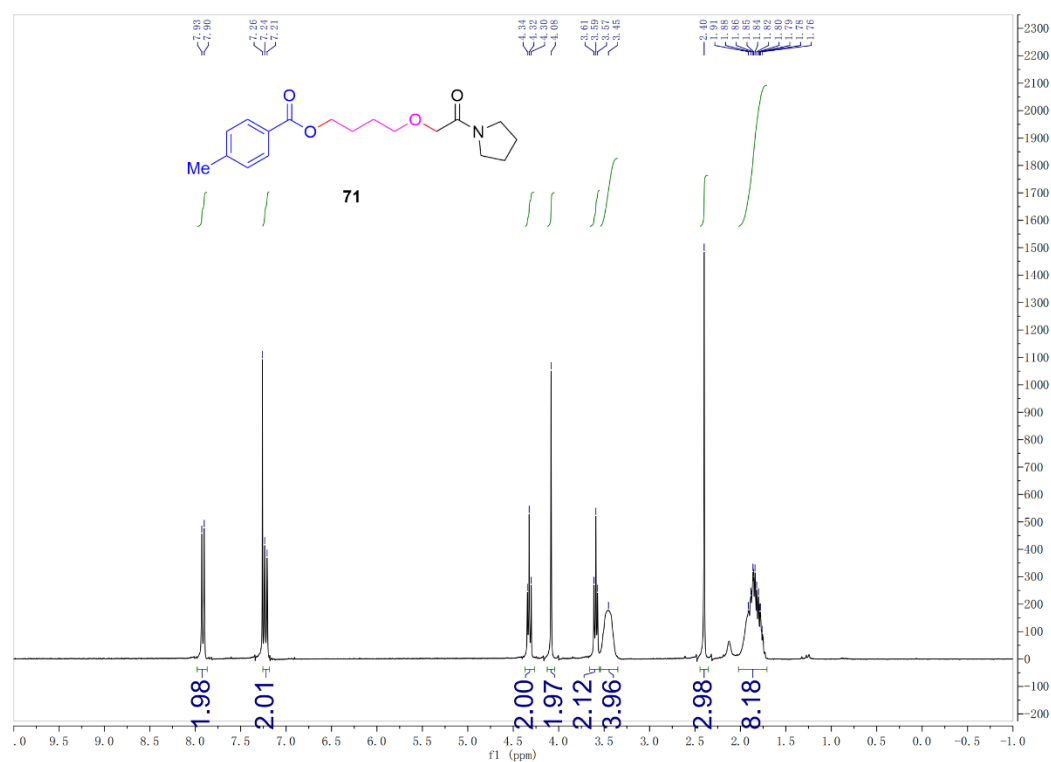
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 70**



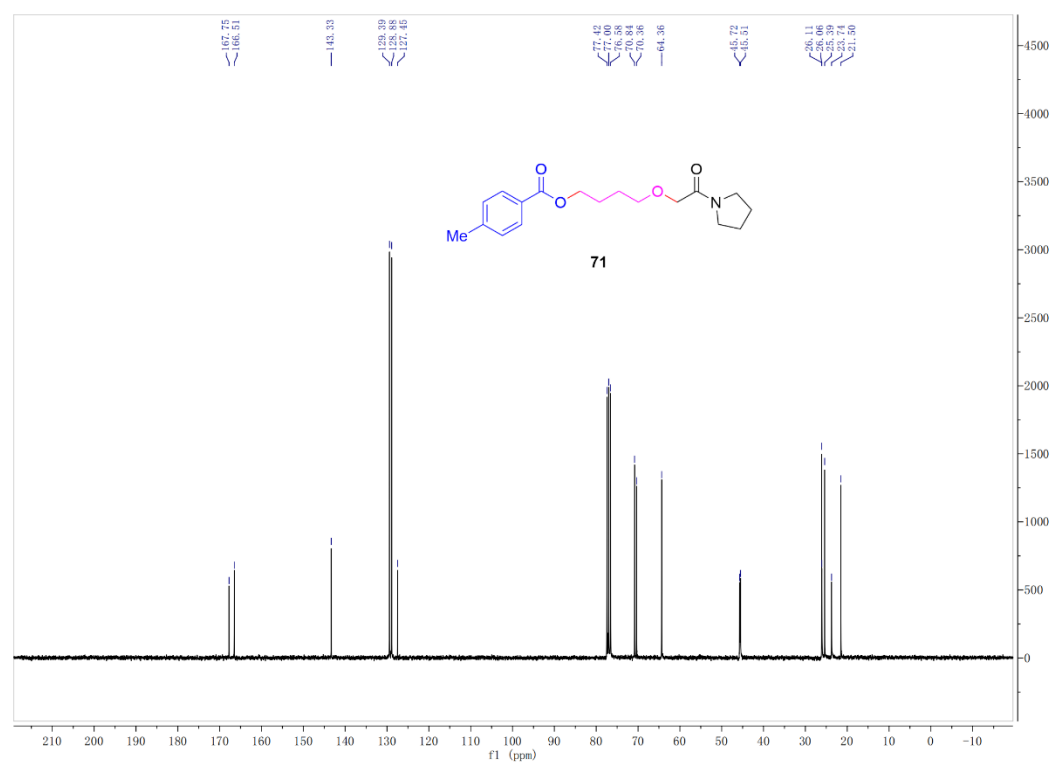
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 70**



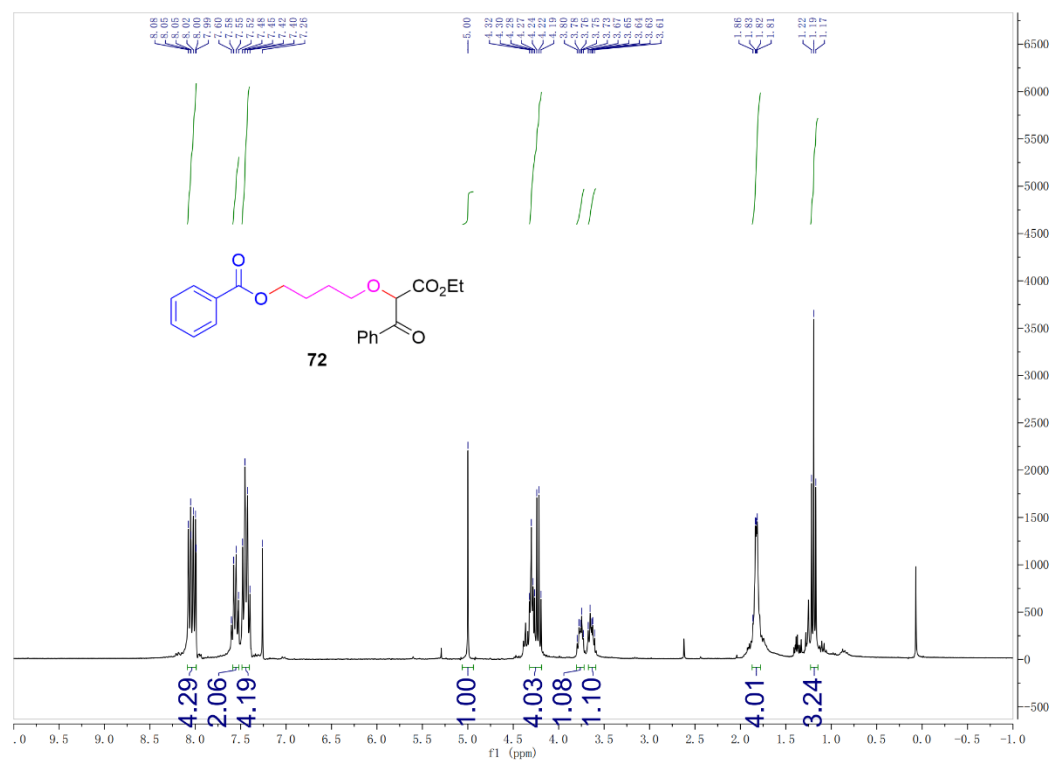
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 71**



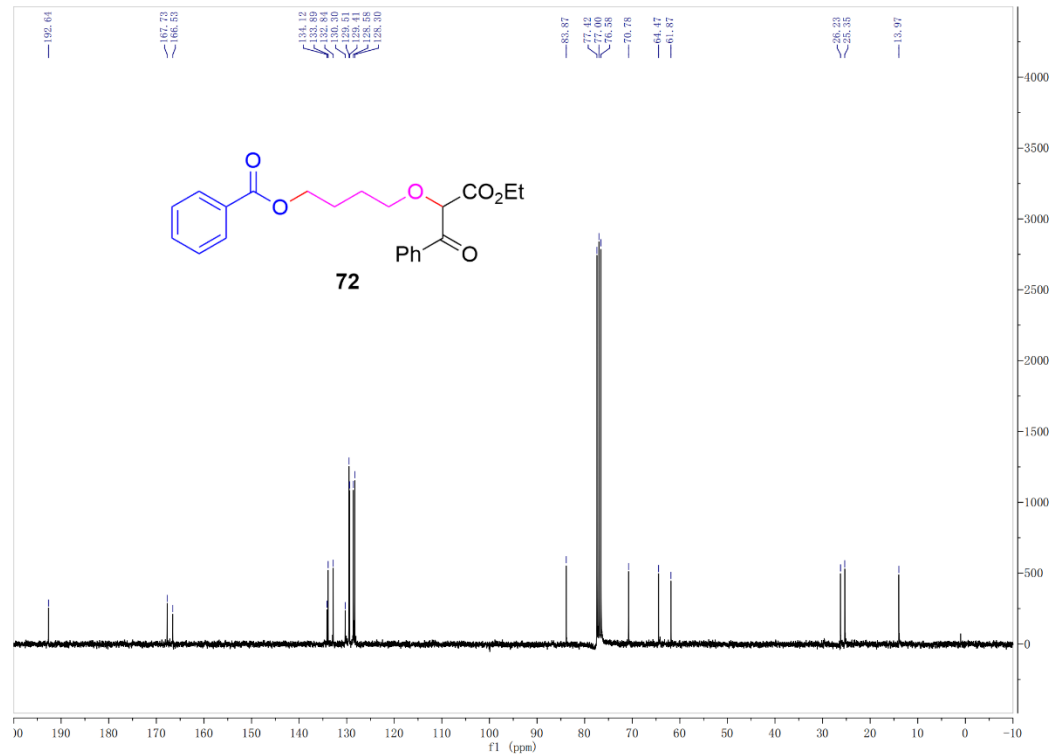
**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 71**



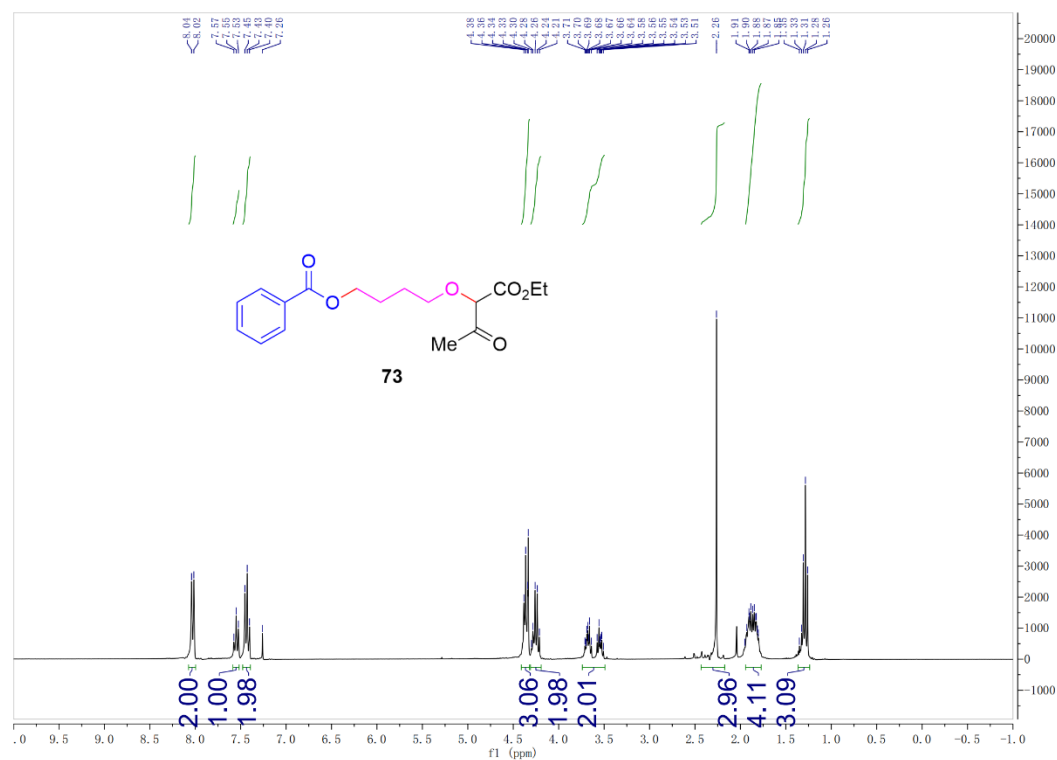
**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 72**



**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 72**



**$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) Spectrum of 73**



**$^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CDCl}_3$ ) Spectrum of 73**

