# Supporting Information

**Radical Cascade Synthesis of γ-Amino Acids or γ-Lactams via Carboxyl-Mediated Intramolecular C−H Amination**

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# 1 Supplementary Methods

## 1.1 General Considerations

Unless otherwise noted, all reactions for substrates preparation were conducted in a flame-dried glass tube under an Ar atmosphere using anhydrous solvents. Commercially available chemicals were obtained from Adamas-beta, Energy Chemical, Bidepharm, Leyan, TCI or J&K Scientific and used as received unless otherwise stated. Anhydrous ethyl acetate (EA) was purchased from Energy Chemical. Flash column chromatography was performed over silica gel (300-400 mesh). Organic solutions were concentrated under reduced pressure on an IKA MVP 10 rotary evaporating using a temperature-controlled water bath.

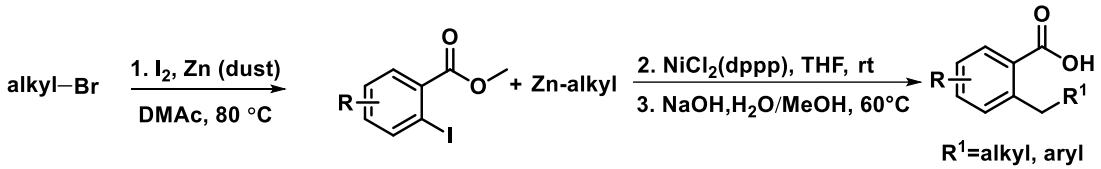
$^{1}H$, $^{19}F$, and $^{13}C$ NMR spectra were recorded on a Bruker AVANCE 600 MHz or a Bruker AVANCE 400 MHz spectrometer. Chemical shifts (δ) for $^{1}H$ and $^{13}C$ NMR spectra are given in ppm relative to TMS. The residual solvent signals were used as references for $^{1}H$ and $^{13}C$ NMR spectra and the chemical shifts converted to the TMS scale ($CDCl_3$ referenced at 7.26 ppm and 77.16 ppm, respectively; methanol-d4 referenced at 3.31 and 49.00 ppm, respectively; DMSO-d6 referenced at 2.50 and 39.52 ppm, respectively; $D_2O$ referenced at 4.79). $^{1}H$ and $^{19}F$ NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, h = hextet, hept = heptet, m = multiplet, b = broad), coupling constant (Hz), and integration. Data for $^{13}C$ NMR are reported in terms of chemical shift. High-resolution mass spectra (HRMS) were obtained from the Lanzhou University Analysis and Testing Center on Thermo Scientific Q Exactive Orbitrap LC-MS/MS System.

# 2 Supplementary Discussion

## 2.1 Preparation of substrates

Substrates s1-s100 were prepared according to the previously reported literature procedures.

### General synthetic methods for primary alkyl-benzoic acids




Step 1. Preparation of the Organozinc Reagents

Following the general procedure with slight modification.[1]A 25 mL round-bottomed flask was charged with zinc powder (1.5 equiv.) and heated to 70 ℃ under high vacuum for 30 min. After back-filling with argon, $I_2$ (0.05 equiv.) and DMAc (1.5 M) were added, and the resulting heterogenous red mixture was allowed to stir until the red color of the iodine had faded. Then, the alkyl halide (1 equiv.) was added. The colorless reaction mixture was allowed to stir for 12 h at 70 ℃, then the mixture was allowed to cool to room temperature. The gray solution was passed through dry celite and stored under argon.

Step 2. Negishi coupling of primary alkyl zinc reagents.

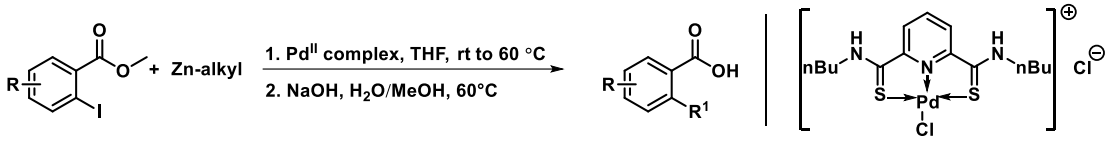
Following the general procedure with slight modification.[2,3] A 100 mL Schlenk tube was charged with $NiCl_2$(dppp) (406 mg, 0.75 mmol) and a stir bar in the air. The tube was sealed with a septum and backfilled with Ar. A solution of the methyl 2-iodobenzoate (15 mmol) in dry THF (10 mL) was added via a syringe. After stirring for 3-4 min at rt, the corresponding alkylzinc bromide (0.5 M THF solution, 30 mmol) was added, and the reaction mixture was stirred at room temperature for the indicated time and the corresponding Negishi coupling products in 23-90% yield after 18 hours. Besides the unconverted starting materials, side products included the biaryl coupling adduct and the dehalogenated esters. The reaction mixture was then transferred with EtOAc (40 mL) to a separatory funnel containing water (40 mL), the product was extracted with EtOAc (3×30 mL) and the combined organic extracts were washed with water (60 mL) and brine (80 mL). After drying (anhydrous $Na_2SO_4$), the solution was filtered and concentrated, and the residue was purified by

silica gel column chromatography (petroleum ether: EtOAc = 20:1).

Step 3. General procedure for ester hydrolysis

Following the general procedure with slight modification.[4] To a solution of methyl 2-alkyl benzoate (10 mmol) in MeOH (20 mL) was added 20 mL $H_2O$ and sodium hydroxide (5 equiv.), and the resulting mixture was stirred at 60 ℃ for 4 hours before adjusted pH to 2 with 1M HCl. The reaction mixture was extracted with EtOAc (3 × 10 mL). The organic phase was combined and dried over anhydrous $Na_2SO_4$, filtered, and evaporated to dryness *in vacuo*, affording the compound as a solid. Further crystallization from EtOAc–petroleum ether gave pure 2-alkylbenzoic acid.

## General synthetic methods for secondary alkyl-benzoic acids (S52-S63)

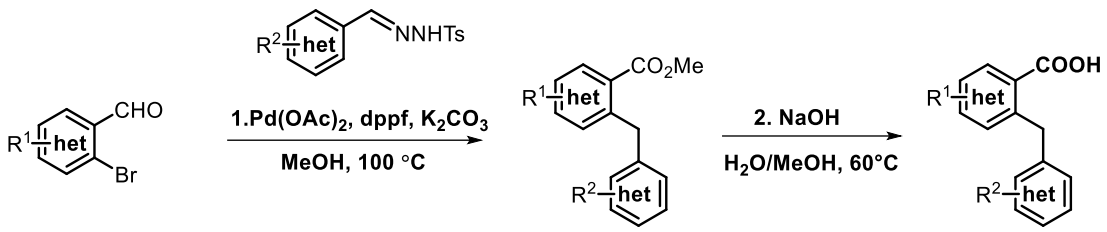



Step 1. Negishi coupling of secondary alkyl zinc reagents.

Following the general procedure with slight modification.[5] Zinc chloride (24.0 mmol) was added to a Schlenk tube, which was sealed with a rubber septum and transferred out. After THF (12.0 mL) was injected into the tube, alkylmagnesium chloride (1.0 M, 24 mL) was added dropwise. Then the mixture was stirred for 1 h at r.t., and then pincer thioamide $Pd^{II}$ complex (0.1 mol%) and 24 mmol methyl 2-iodobenzoate derivatives were added. The resultant mixture was stirred at 60 ℃. After the reaction was completed, the mixture was cooled to 0 ℃, quenched with saturated $NH_4Cl$ and extracted with ethyl acetate three times. The combined organic phase was washed with brine, and dried over $Na_2SO_4$. Then, it was concentrated *in vacuo*, and the resulting crude product was purified by silica gel column chromatography (petroleum ether: EtOAc = 20:1).

Step 2. To a solution of methyl 2-alkylbenzoate (10 mmol) in MeOH (20 mL) was added 20 mL $H_2O$ and NaOH (5 equiv.) and the resulting mixture stirred at 60 ℃ for 4 hours before adjusted pH to 2 with HCl (1N). The reaction mixture was extracted with ethyl acetate three times. The organic phase was combined and dried over anhydrous $Na_2SO_4$, filtered, and evaporated to dryness *in vacuo*, further crystallization from EtOAc/petroleum ether gave pure products, as white solids.

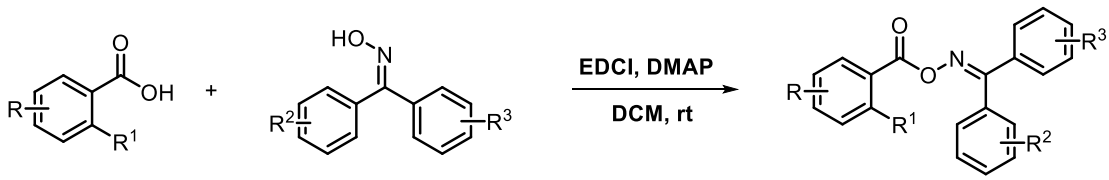
## Synthesis of substrate S17, S18




Step 1. Following the general procedure with slight modification.[6] An oven-dried Schlenk tube was charged with *N*-tosylhydrazone (15 mmol, 1.5 equiv.), $K_2CO_3$ (30 mmol, 3.0 equiv.), Pd $(OAc)_2$ (5 mol%), dppf (7.5 mol%). The tube was evacuated and backfilled with argon, and this procedure was repeated three times. To this mixture was added MeOH (100 mL) and 2-bromobenzaldehyde (10 mmol, 1.0 equiv.). The reaction was stirring at the indicated temperature. After the reaction was completed, the reaction mixture was quenched with water (40 mL) and extracted with ethyl acetate three times, dried over $Na_2SO_4$. The combined filtrate was concentrated, and the residue was purified by silica gel column chromatography to give the pure products.

Step 2. To a solution of methyl 2-alkyl benzoates (10 mmol) in MeOH (20 mL) was added 20 mL $H_2O$ and NaOH (5 equiv.), and the resulting mixture was stirred at 60 ℃ for 4 hours before adjusted pH to 2 with HCl (1N). The reaction mixture was extracted with ethyl acetate three times. The organic phase was combined and dried over anhydrous $Na_2SO_4$, filtered, and evaporated to dryness *in vacuo*, further crystallization from EtOAc/petroleum ether gave pure products as white solids.

## General synthetic methods for substrates S1-S63

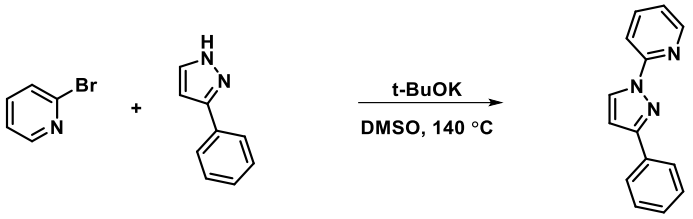



Oxime esters were prepared following the reported literature procedure.[7] To a solution of oximes (10.0 mmol) and carboxylic acid (10.0 mmol) in $CH_2Cl_2$ (30 mL), DMAP (10 mol%) and EDCI (25 mmol) was added. The mixture was stirred at room temperature under an inert atmosphere until the reaction was complete, as observed from TLC monitoring. The mixture was diluted with distilled water (25 mL) and the $CH_2Cl_2$ layer was separated, dried over anhydrous $Na_2SO_4$, and concentrated. The crude mass was treated with pentane (10 mL) and sonicated for 15 minutes. The resultant solid was filtered and dried under vacuum to obtain the pure oxime esters in most cases. In some cases,

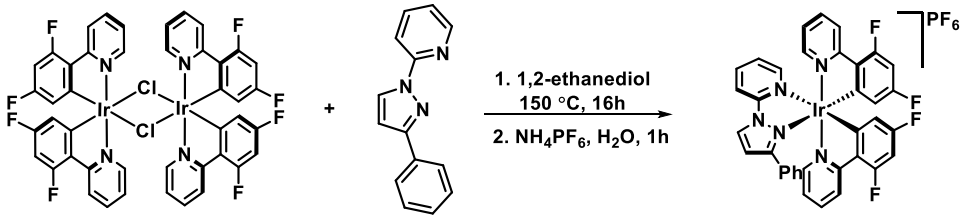
the final compound was purified by flash column chromatography using pentane/dichloromethane (DCM) or pentane/ethyl acetate (EtOAc) as eluent.

## 2.2 Synthesis of Ir-1, Ir-4

## Synthesis of 2-(3-Phenyl-1*H*-pyrazol-1-yl)pyridine (phpzpy).

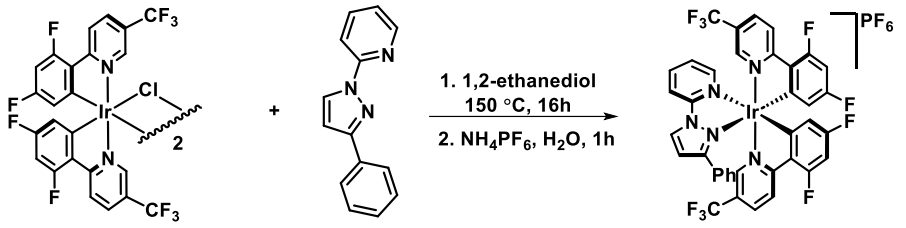



The ancillary ligand, phpzpy, was synthesized by using the reported procedure.[8,9] 3-Phenyl-1*H*-pyrazole (15 mmol, 1.0 equiv.) and potassium *tert*-butoxide (17 mmol, 1.13 equiv.) were dissolved at room temperature in dry DMSO (15 mL). To the basic solution, 2-bromopyridine (16 mmol, 1.1 equiv.) was added slowly under constant stirring. The reaction mixture was refluxed at 140 °C for 12 h under nitrogen and then cooled to room temperature, after which it was extracted with water and ether. The organic layer was washed with water and ether to remove DMSO as well as excess potassium *tert*-butoxide. The organic layer was then isolated, dried over anhydrous $Na_2SO_4$, and filtered. The solvent was then removed under reduced pressure. The crude product was purified by column chromatography on silica gel with hexane/ethyl acetate (20:1) as the eluent, to yield colorless oil that crystallized into colorless solid crystals after a while.




A solution of $[Ir(dFppy)_2Cl]_2$ (2 mmol, 1.0 equiv.) and phpzpy (2.3 equiv.) in 1,2-ethanediol (150 mL) The reaction mixture was heated to reflux at 150 °C for 16 h under nitrogen atmosphere with constant stirring and then cooled to room temperature. A concentrated solution of $NH_4PF_6$ (60 equiv.) in deionized water (100 mL) was added slowly to the reaction mixture under stirring for 1 h, resulting in a yellow suspension. The suspension was filtered, and the resultant precipitate was

washed with plenty of deionized water. The yellow precipitate obtained was dried under vacuum at 100 °C for 12 h. The crude material was subsequently crystallized from dichloromethane/hexane, yielding a yellow powder.[8,10]

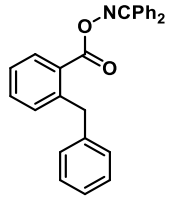



[Ir(dF(CF$_3$)ppy)$_2$(phpzpy)]PF$_6$
(**Ir-4**)

The same method as **Ir-1**, [Ir(dF(CF$_3$)ppy)$_2$Cl]$_2$ (1.12 g, 0.75 mmol) and 2-(3-phenyl-1*H*-pyrazol-1-yl) pyridine (0.35 g, 1.6 mmol) in 1,2-ethanediol (50 mL) under nitrogen. Subsequently it crystallized from dichloromethane/hexane, yielding a bright light-yellow powder.

## 2.3 Characterization data of substrates

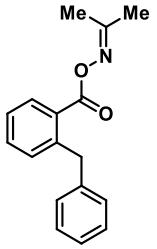
**diphenylmethanone *O*-(2-benzylbenzoyl) oxime (S1)**



**Chemical Formula:** $C_{27}H_{21}NO_2$

**S1** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.70 – 7.61 (m, 2H), 7.54 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.51 – 7.40 (m, 4H), 7.40 – 7.29 (m, 5H), 7.28 – 7.21 (m, 2H), 7.21 – 7.10 (m, 5H), 4.29 (s, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.7, 143.1, 140.6, 134.6, 132.9, 132.2, 131.4, 131.0, 130.5, 129.6, 129.2, 129.1, 128.6, 128.4, 128.3, 128.3, 128.2, 126.1, 126.0, 39.2.
**HRMS (ESI)**: m/z calculated for $[C_{27}H_{21}NO_2Na]^+$ [M + Na]$^+$: 414.1465, found: 414.1460.

**propan-2-one *O*-(2-benzylbenzoyl) oxime (S1-1)**



**Chemical Formula:** $C_{17}H_{17}NO_2$

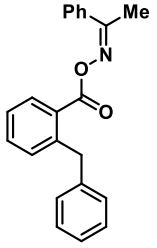
**S1-1** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.43 (td, *J* = 7.6, 1.5 Hz, 1H), 7.36 – 7.07 (m, 7H), 4.38 (s, 2H), 2.08 (s, 3H), 1.94 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.1, 164.6, 142.3, 140.5, 132.0, 131.4, 130.1, 129.0, 128.9, 128.3, 126.1, 125.9, 39.2, 22.0, 17.1.

**HRMS (ESI)**: m/z calculated for $[C_{17}H_{17}NO_2Na]^+$ $[M + Na]^+$: 290.1152, found: 290.1152.

**1-phenylethan-1-one *O*-(2-benzylbenzoyl) oxime (S1-2)**



**Chemical Formula:** $C_{22}H_{19}NO_2$

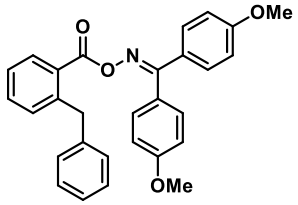
**S1-2** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.93 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.82 – 7.73 (m, 2H), 7.47 – 7.34 (m, 4H), 7.33 – 7.09 (m, 7H), 4.42 (s, 2H), 2.31 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 164.8, 163.4, 142.5, 140.5, 134.7, 132.2, 131.5, 130.6, 130.2, 129.0, 128.8, 128.5, 128.3, 127.0, 126.2, 125.9, 39.2, 14.6.

**HRMS (ESI)**: m/z calculated for $[C_{22}H_{19}NO_2Na]^+$ $[M + Na]^+$: 352.1308, found: 352.1308.

**bis(4-methoxyphenyl)methanone *O*-(2-benzylbenzoyl) oxime (S1-3)**



**Chemical Formula:** $C_{29}H_{25}NO_4$

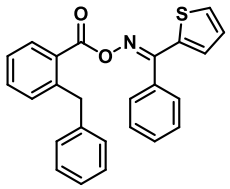
**S1-3** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.68 – 7.54 (m, 3H), 7.36 (td, *J* = 7.6, 1.5 Hz, 1H), 7.31 – 7.20 (m, 4H), 7.20 – 7.07 (m, 5H), 6.97 – 6.83 (m, 4H), 4.29 (s, 2H), 3.82 (s, 6H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.2, 165.1, 161.9, 160.5, 143.0, 140.7, 132.2, 131.4, 130.9, 130.8, 130.5, 129.3, 128.6, 128.4, 127.5, 126.2, 126.0, 125.2, 113.8, 113.6, 55.4, 55.3, 39.2.

**HRMS (ESI)**: m/z calculated for $[C_{29}H_{25}NO_4Na]^+$ $[M + Na]^+$: 474.1676, found: 474.1676.

**phenyl(thiophen-2-yl)methanone *O*-(2-benzylbenzoyl) oxime (S1-4)**



**Chemical Formula:** $C_{25}H_{19}NO_2S$

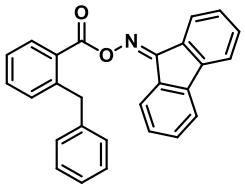
**S1-4** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 8.07 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.69 – 7.59 (m, 2H), 7.54 – 7.31 (m, 6H), 7.31 – 7.20 (m, 4H), 7.20 – 6.96 (m, 4H), 4.35 (d, *J* = 69.3 Hz, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) major-**S1-4**: δ 164.7, 161.2, 158.1, 143.3, 140.6, 137.9, 135.4, 133.0, 132.4, 132.3, 131.5, 130.8, 130.2, 129.9, 129.3, 128.4, 128.4, 127.3, 126.3, 126.1, 39.3. miner-**S1-4**: δ 164.4, 143.1, 135.4, 132.7, 132.1, 131.5, 131.4, 130.5, 130.3, 129.8, 129.2, 128.6, 128.3, 128.1, 126.3, 126.1, 126.1, 126.0, 39.1.

**HRMS (ESI)**: m/z calculated for $[C_{25}H_{19}NO_2SNa]^+$ $[M + Na]^+$: 420.1029, found: 420.1030.

### 9*H*-fluoren-9-one *O*-(2-benzylbenzoyl) oxime (S1-5)



**Chemical Formula:** $C_{27}H_{19}NO_2$

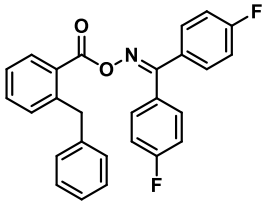
**S1-5** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 7.7 Hz, 1H), 7.99 (dd, *J* = 7.6, 1.8 Hz, 2H), 7.58 (dd, *J* = 12.8, 7.5 Hz, 2H), 7.51 (td, *J* = 7.6, 1.5 Hz, 1H), 7.47 – 7.35 (m, 3H), 7.35 – 7.28 (m, 2H), 7.27 – 7.19 (m, 5H), 7.14 (h, *J* = 4.3 Hz, 1H), 4.45 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 164.9, 158.9, 143.2, 142.7, 141.2, 140.5, 134.5, 132.6, 132.5, 131.8, 131.7, 130.2, 130.1, 130.1, 129.2, 128.6, 128.5, 126.5, 126.2, 123.5, 120.4, 120.2, 39.2.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{19}NO_2Na]^+$ $[M + Na]^+$: 412.1308, found: 412.1310.

### bis(4-fluorophenyl)methanone *O*-(2-benzylbenzoyl) oxime (S1-6)



**Chemical Formula:** $C_{27}H_{19}F_2NO_2$

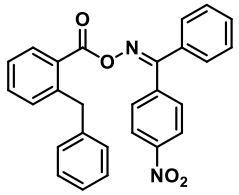
**S1-6** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.63 (dd, *J* = 8.7, 5.5 Hz, 2H), 7.59 – 7.51 (m, 1H), 7.44 – 7.35 (m, 1H), 7.35 – 7.22 (m, 4H), 7.22 – 7.03 (m, 9H), 4.31 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.2 (d, *J* = 137.36 Hz), 164.6, 163.6, 162.7 (d, *J* = 135.34 Hz), 143.2, 140.54, 132.5, 131.6, 131.2, 131.2, 131.0, 130.9, 130.7 (d, *J* = 3.03 Hz), 130.4, 129.2, 128.5 (d, *J* = 3.03 Hz), 128.4, 128.0, 126.2 (d, *J* = 11.11 Hz), 115.8 (d, *J* = 3.03 Hz), 115.6 (d, *J* = 2.02 Hz), 39.3.

**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -108.5, -109.8.
**HRMS (ESI)**: m/z calculated for $[C_{27}H_{19}F_2NO_2Na]^+$ $[M + Na]^+$: 450.1276, found: 450.1274.

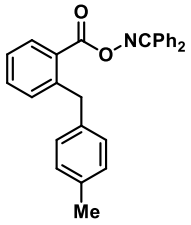
**(4-nitrophenyl)(phenyl)methanone *O*-(2-benzylbenzoyl) oxime (S1-7)**




**Chemical Formula:** $C_{27}H_{20}N_2O_4$

**S1-7** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 8.32 – 8.19 (m, 2H), 7.88 – 7.80 (m, 1H), 7.64 – 7.57 (m, 2H), 7.54 – 7.44 (m, 4H), 7.44 – 7.36 (m, 2H), 7.30 – 7.05 (m, 7H), 4.29 (d, *J* = 15.4 Hz, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) major-**S1-7**: δ 164.3, 163.5, 148.3, 143.0, 140.4, 139.3, 133.3, 132.6, 131.6, 130.2, 130.0, 129.6, 129.1, 128.8, 128.7, 128.4, 127.7, 126.3, 126.1, 123.7, 39.2. miner- **S1-7**: δ 164.2, 149.2, 143.4, 140.7, 131.7, 130.5, 130.3, 129.1, 128.7, 128.6, 127.5, 126.2, 126.0, 123.6, 39.3.
**HRMS (ESI)**: m/z calculated for $[C_{27}H_{20}N_2O_4Na]^+$ $[M + Na]^+$: 459.1315, found: 459.1314.

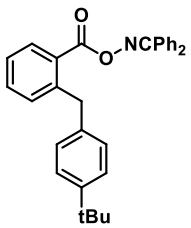
**diphenylmethanone *O*-(2-(4-methylbenzyl)benzoyl) oxime (S2)**




**Chemical Formula:** $C_{28}H_{23}NO_2$

**S2** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.72 – 7.65 (m, 2H), 7.56 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.51 – 7.44 (m, 4H), 7.44 – 7.32 (m, 5H), 7.18 – 7.12 (m, 2H), 7.12 – 7.02 (m, 4H), 4.27 (s, 2H), 2.32 (s, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.8, 143.5, 137. 6, 135.4, 134.7, 133.0, 132.2, 131.3, 131.0, 130. 5, 129.6, 129.1, 129.1, 129.1, 128.7, 128.5, 128. 5, 128.3, 128.2, 126.1, 38.8, 21.1.
**HRMS (ESI)**: m/z calculated for $[C_{28}H_{23}NO_2Na]^+$ $[M + Na]^+$: 428.1621, found: 428.1621.

**diphenylmethanone *O*-(2-(4-(tert-butyl)benzyl)benzoyl) oxime (S3)**

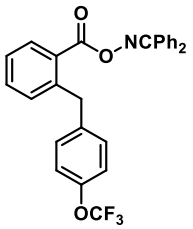



**Chemical Formula:** $C_{31}H_{29}NO_2$

**S3** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.68 – 7.61 (m, 2H), 7.52 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.49 – 7.41 (m,

4H), 7.36 (dddd, *J* = 14.3, 7.9, 3.5, 1.6 Hz, 5H), 7.30 – 7.23 (m, 2H), 7.19 – 7.10 (m, 2H), 7.10 – 7.02 (m, 2H), 4.26 (s, 2H), 1.29 (s, 9H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.8, 148.7, 143.4, 137.5, 134.7, 133.0, 132.2, 131.4, 131.0, 130.4, 129.6, 129.1, 128.8, 128.7, 128.4, 128.3, 128.2, 126.0, 125.3, 38.7, 34.4, 31.4.
**HRMS (ESI)**: m/z calculated for $[C_{31}H_{30}NO_2]^+$ $[M + H]^+$: 448.2271, found: 448.2269.

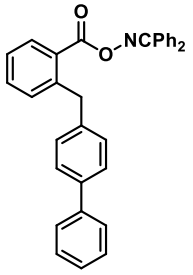
**diphenylmethanone *O*-(2-(4-(trifluoromethoxy)benzyl)benzoyl) oxime (S4)**




**Chemical Formula:** $C_{28}H_{20}F_3NO_3$

**S4** was obtained as a white solid.
**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.72 – 7.61 (m, 2H), 7.56 (d, *J* = 7.1 Hz, 1H), 7.52 – 7.29 (m, 10H), 7.23 – 7.02 (m, 7H), 4.32 (s, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.8, 164.5, 147.5, 147.5, 142.5, 139.5, 134.6, 132.9, 132.5, 131.6, 131.1, 130.7, 130.3, 129.7, 129.1, 128.6, 128.5, 128.3, 128.1, 126.5, 120.8, 120.5 (q, *J* = 257.55 Hz), 38.6.
**$^{19}$F NMR** (376 MHz, CDCl3) δ -57.8.
**HRMS (ESI)**: m/z calculated for $[C_{28}H_{21}F_3NO_3]^+$ $[M + H]^+$: 476.1468, found: 476.1466.

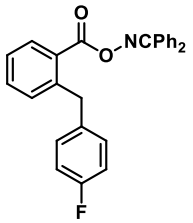
**diphenylmethanone *O*-(2-([1,1'-biphenyl]-4-ylmethyl)benzoyl) oxime (S5)**




**Chemical Formula:** $C_{33}H_{25}NO_2$

**S5** was obtained as a white solid.
**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.70 – 7.62 (m, 2H), 7.60 – 7.51 (m, 3H), 7.51 – 7.26 (m, 14H), 7.26 – 7.10 (m, 4H), 4.34 (s, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.7, 143.0, 141.0, 139.7, 138.8, 134.6, 132.9, 132.4, 131.5, 131.0, 130.5, 129.6, 129.5, 129.1, 128.7, 128.6, 128.4, 128.3, 128.1, 127.1, 127.0, 127.0, 126.2, 38.9.
**HRMS (ESI)**: m/z calculated for $[C_{33}H_{26}NO_2]^+$ $[M + H]^+$: 468.1958, found: 468.1957.

**diphenylmethanone *O*-(2-(4-fluorobenzyl)benzoyl) oxime (S6)**



**Chemical Formula:** $C_{27}H_{20}FNO_2$

**S6** was obtained as a white solid.

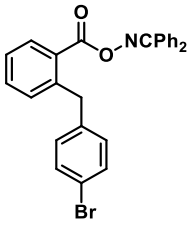
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.64 (m, 2H), 7.62 – 7.53 (m, 1H), 7.54 – 7.31 (m, 10H), 7.21 – 7.06 (m, 4H), 7.01 – 6.88 (m, 2H), 4.29 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.1 (d, $J$ = 109.08 Hz), 161.3 (d, $J$ = 244.42 Hz), 142.9, 136.2 (d, J = 3.03 Hz), 134.5, 132.8, 132.3, 131.3, 131.0, 130.5 (d, $J$ = 3.03 Hz), 130.4, 129.6, 129.0, 128.6, 128.4, 128.3, 128.0, 126.3, 115.0 (d, $J$ = 21.21 Hz), 38.4.

**$^{19}$F NMR** (376 MHz, CDCl3) δ -117.5.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{21}FNO_2]^+$ $[M + H]^+$: 410.1551, found: 410.1549.

**diphenylmethanone *O*-(2-(4-bromobenzyl)benzoyl) oxime (S7)**



**Chemical Formula:** $C_{27}H_{20}BrNO_2$

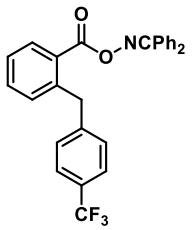
**S7** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.69 – 7.61 (m, 2H), 7.55 (dd, $J$ = 7.8, 1.5 Hz, 1H), 7.51 – 7.43 (m, 4H), 7.36 (dddd, $J$ = 18.3, 7.9, 4.0, 2.1 Hz, 7H), 7.21 – 7.12 (m, 2H), 7.07 – 6.97 (m, 2H), 4.25 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.8, 164.6, 142.5, 139.7, 134.6, 132.9, 132.5, 131.5, 131.4, 131.1, 130.9, 130.7, 129.7, 129.1, 128.7, 128.5, 128.4, 128.0, 126.5, 119.9, 38.7.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{20}BrNO_2Na]^+$ $[M + Na]^+$: 492.0570, found: 492.0569.

**diphenylmethanone *O*-(2-(4-(trifluoromethyl)benzyl)benzoyl) oxime (S8)**



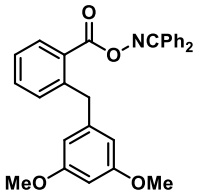
**Chemical Formula:** $C_{28}H_{20}F_3NO_2$

**S8** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.64 (dt, $J$ = 7.1, 1.4 Hz, 2H), 7.56 (dd, $J$ = 7.8, 1.5 Hz, 1H), 7.52 – 7.27 (m, 11H), 7.24 (d, $J$ = 8.0 Hz, 2H), 7.21 – 7.11 (m, 2H), 4.36 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.9, 164.5, 144.9 (d, *J* = 1.21 Hz), 142.0, 134.6, 132.9, 132.6, 131.7, 131.1, 130.8, 129.7, 129.3, 129.1, 128.6, 128.5, 128.4, 128.1, 128.3 (q, *J* = 32.32 Hz), 126.7, 125.2 (q, *J* = 3.03 Hz), 124.4 (q, *J* = 273.71 Hz), 39.2.
**$^{19}$F NMR** (376 MHz, CDCl3) δ -62.3.
**HRMS (ESI)**: m/z calculated for $[C_{28}H_{21}F_3NO_2]^+$ $[M + H]^+$: 460.1519, found: 460.1517.

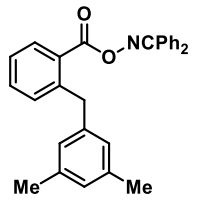
**diphenylmethanone *O*-(2-(3,5-dimethoxybenzyl)benzoyl) oxime (S9)**



**Chemical Formula:** $C_{29}H_{25}NO_4$

**S9** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.65 (dt, *J* = 8.6, 1.5 Hz, 2H), 7.52 (dt, *J* = 7.9, 1.9 Hz, 1H), 7.49 – 7.27 (m, 10H), 7.12 (ddd, *J* = 13.0, 6.8, 2.9 Hz, 2H), 6.30 (q, *J* = 2.1 Hz, 3H), 4.22 (s, 2H), 3.70 (s, 6H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.7, 164.9, 160.8, 143.0, 142.7, 134.7, 133.0, 132.3, 131.4, 131.1, 130.4, 129.7, 129.4, 129.3, 129.1, 129.0, 128.7, 128.5, 128.4, 128.3, 128.2, 127.9, 126.2, 107.4, 98.2, 55.3, 39.4.
**HRMS (ESI)**: m/z calculated for $[C_{29}H_{26}NO_4]^+$ $[M + H]^+$: 452.1856, found: 452.1856.

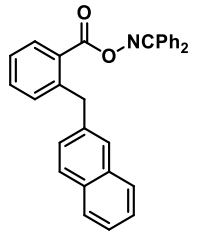
**diphenylmethanone *O*-(2-(3,5-dimethylbenzyl)benzoyl) oxime (S10)**



**Chemical Formula:** $C_{29}H_{25}NO_2$

**S10** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.77 – 7.62 (m, 2H), 7.58 – 7.51 (m, 1H), 7.51 – 7.31 (m, 9H), 7.15 (dt, *J* = 7.6, 3.6 Hz, 2H), 6.84 (s, 1H), 6.78 (s, 2H), 4.23 (s, 2H), 2.26 (s, 6H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.8, 143.4, 140.4, 137.8, 134.7, 132.9, 132.2, 131.4, 131.0, 130.4, 129.6, 129.1, 128.7, 128.4, 128.3, 128.1, 127.7, 127.1, 126.0, 38.9, 21.3.
**HRMS (ESI)**: m/z calculated for $[C_{29}H_{26}NO_2]^+$ $[M + H]^+$: 420.1958, found: 420.1960.

**diphenylmethanone *O*-(2-(naphthalen-2-ylmethyl)benzoyl) oxime (S11)**



**Chemical Formula:** $C_{31}H_{23}NO_2$

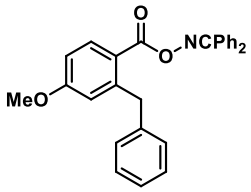
**S11** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.82 – 7.76 (m, 1H), 7.72 (d, *J* = 7.9 Hz, 2H), 7.68 – 7.60 (m, 2H), 7.60 – 7.52 (m, 2H), 7.51 – 7.33 (m, 11H), 7.33 – 7.21 (m, 4H), 7.16 (t, *J* = 7.6 Hz, 2H), 4.44 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.7, 164.9, 143.0, 138.2, 134.6, 133.6, 133.0, 132.3, 132.1, 131.5, 131.0, 130.6, 129.6, 129.2, 129.1, 128.8, 128.6, 128.5, 128.4, 128.3, 128.0, 127.9, 127.7, 127.6, 127.4, 126.2, 125.89, 125.3, 39.4.

**HRMS (ESI)**: m/z calculated for $[C_{31}H_{23}NO_2Na]^+$ $[M + Na]^+$: 464.1621, found: 464.1618.

**diphenylmethanone *O*-(2-benzyl-4-methoxybenzoyl) oxime (S12)**




**Chemical Formula:** $C_{28}H_{23}NO_3$

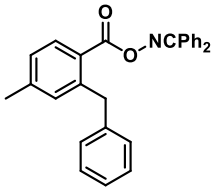
**S12** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.73 – 7.60 (m, 2H), 7.54 (d, *J* = 9.5 Hz, 1H), 7.48 – 7.41 (m, 4H), 7.40 – 7.31 (m, 4H), 7.25 (ddd, *J* = 8.1, 6.4, 1.1 Hz, 2H), 7.16 (td, *J* = 7.4, 1.6 Hz, 3H), 6.69 – 6.57 (m, 2H), 4.32 (s, 2H), 3.72 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.1, 164.1, 162.6, 146.3, 140.5, 134.8, 133.2, 133.0, 130.9, 129.5, 129.2, 129.1, 128.7, 128.4, 128.4, 128.3, 126.0, 120.0, 117.2, 111.1, 55.3, 39.5.

**HRMS (ESI)**: m/z calculated for $[C_{28}H_{23}NO_3Na]^+$ $[M + Na]^+$: 482.1338, found: 482.1335.

**diphenylmethanone *O*-(2-benzyl-4-methylbenzoyl) oxime (S13)**




**Chemical Formula:** $C_{28}H_{23}NO_2$

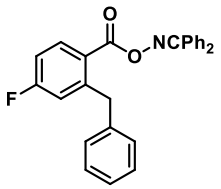
**S13** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.69 – 7.61 (m, 2H), 7.48 – 7.39 (m, 5H), 7.39 – 7.28 (m, 4H), 7.28 – 7.19 (m, 2H), 7.15 (td, *J* = 7.2, 1.6 Hz, 3H), 6.99 – 6.90 (m, 2H), 4.28 (s, 2H), 2.25 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.4, 164.7, 143.4, 143.0, 140.9, 134.8, 133.1, 132.3, 131.0, 130.8, 129.6, 129.2, 129.1, 128.7, 128.4, 128.3, 127.0, 126.0, 125.2, 39.2, 21.6.

**HRMS (ESI)**: m/z calculated for $[C_{28}H_{24}NO_2]^+$ $[M + H]^+$: 406.1802, found: 406.1779.

**diphenylmethanone *O*-(2-benzyl-4-fluorobenzoyl) oxime (S14)**



**Chemical Formula:** $C_{27}H_{20}FNO_2$

**S14** was obtained as a white solid.

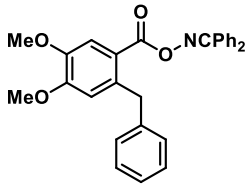
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.69 – 7.62 (m, 2H), 7.56 (dd, *J* = 8.6, 5.9 Hz, 1H), 7.46 (qd, *J* = 4.5, 1.5 Hz, 4H), 7.42 – 7.31 (m, 5H), 7.30 – 7.23 (m, 2H), 7.23 – 7.17 (m, 1H), 7.14 (dd, *J* = 6.9, 1.8 Hz, 2H), 6.85 – 6.75 (m, 2H), 4.29 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) major-**S14**: δ165.8, 163.8, 147.2(d, *J* = 8.08 Hz), 139.7, 134.5, 133.1(d, *J* = 9.09 Hz), 133.0, 131.2, 129.7, 129.3, 129.1, 128.6, 128.6, 128.5, 128.4, 126.4, 124.1, 118.2 (d, *J* = 22.22 Hz), 113.3 (d, *J* = 22.22 Hz), 39.3. minor-**S14**: 166.2, 163.7, 131.2, 129.1, 128.5, 128.5, 124.0, 39.0.

**$^{19}$F NMR** (376 MHz, CDCl3) δ -106.0.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{20}FNO_2Na]^+$ $[M + Na]^+$: 432.1370, found: 432.1368.

**diphenylmethanone *O*-(2-benzyl-4,5-dimethoxybenzoyl) oxime (S15)**



**Chemical Formula:** $C_{29}H_{25}NO_4$

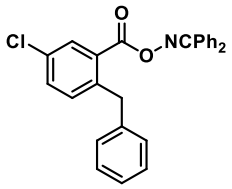
**S15** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.71 – 7.61 (m, 2H), 7.53 – 7.41 (m, 4H), 7.41 – 7.33 (m, 4H), 7.30 – 7.20 (m, 2H), 7.20 – 7.12 (m, 3H), 6.99 (s, 1H), 6.62 (s, 1H), 4.37 (s, 2H), 3.78 (s, 3H), 3.58 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 164.9, 163.4, 152.2, 146.6, 141.0, 138.5, 134.5, 133.6, 131.0, 129.3, 129.0, 128.9, 128.5, 128.4, 128.4, 128.3, 125.9, 123.8, 118.9, 114.1, 112.8, 55.8, 55.8, 39.0.

**HRMS (ESI)**: m/z calculated for $[C_{29}H_{26}NO_4]^+$ $[M + H]^+$: 474.1676, found: 474.1676.

**diphenylmethanone *O*-(2-benzyl-5-chlorobenzoyl) oxime (S16)**



**Chemical Formula:** $C_{27}H_{20}ClNO_2$

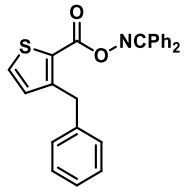
**S16** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.68 – 7.64 (m, 2H), 7.50 – 7.44 (m, 5H), 7.41 – 7.35 (m, 2H), 7.35 – 7.29 (m, 3H), 7.28 – 7.21 (m, 2H), 7.21 – 7.14 (m, 1H), 7.13 – 7.08 (m, 2H), 7.06 (d, *J* = 8.3 Hz, 1H), 4.25 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.2, 163.5, 141.7, 140.1, 134.4, 132.8, 132.2, 132.0, 131.2,

130.5, 129.8, 129.6, 129.12, 129.1, 128.6, 128.5, 128.5, 128.5, 126.2, 38.6.
**HRMS (ESI)**: m/z calculated for $[C_{27}H_{21}ClNO_2]^+$ $[M + H]^+$: 426.1255, found: 426.1256.

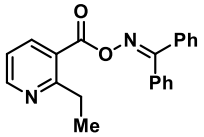
**diphenylmethanone *O*-(3-benzylthiophene-2-carbonyl) oxime (S17)**




**Chemical Formula:** $C_{25}H_{19}NO_2S$

**S17** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.71 – 7.62 (m, 2H), 7.51 – 7.42 (m, 4H), 7.42 – 7.33 (m, 4H), 7.30 (d, *J* = 5.1 Hz, 1H), 7.28 – 7.22 (m, 2H), 7.22 – 7.14 (m, 3H), 6.79 (d, *J* = 5.1 Hz, 1H), 4.27 (s, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.2, 160.0, 150.6, 140.0, 134.6, 132.8, 131.2, 131.0, 129.6, 129.1, 129.0, 128.9, 128.5, 128.5, 128.3, 126.2, 124.4, 35.3.
**HRMS (ESI)**: m/z calculated for $[C_{25}H_{20}NO_2S]^+$ $[M + H]^+$: 398.1209, found: 398.1206.

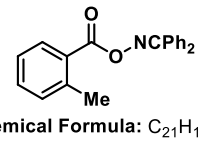
**diphenylmethanone *O*-(2-ethylnicotinoyl) oxime (S18)**




**Chemical Formula:** $C_{21}H_{18}N_2O_2$

**S18** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 8.58 (dd, *J* = 4.8, 1.8 Hz, 1H), 7.75 (dd, *J* = 7.9, 1.8 Hz, 1H), 7.69 – 7.60 (m, 2H), 7.51 – 7.41 (m, 4H), 7.41 – 7.30 (m, 4H), 7.06 (dd, *J* = 7.9, 4.8 Hz, 1H), 3.03 (q, *J* = 7.5 Hz, 2H), 1.19 (t, *J* = 7.5 Hz, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.0, 164.8, 163.8, 152.0, 138.1, 134.2, 132.7, 131.2, 129.6, 128.9, 128.4, 128.4, 128.3, 123.7, 120.6, 30.0, 13.7.
**HRMS (ESI)**: m/z calculated for $[C_{21}H_{19}N_2O_2]^+$ $[M + H]^+$: 331.1441, found: 331.1438.

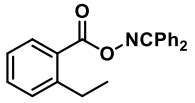
**diphenylmethanone *O*-(2-methylbenzoyl) oxime (S19)**




**Chemical Formula:** $C_{21}H_{17}NO_2$

**S19** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.77 – 7.63 (m, 2H), 7.57 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.53 – 7.30 (m, 9H), 7.25 – 7.06 (m, 2H), 2.49 (s, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.4, 164.7, 140.8, 134.6, 133.1, 132.2, 131.7, 131.0, 130.4, 129.5, 129.0, 128.6, 128.4, 128.3, 128.0, 125.6, 21.5.
**HRMS (ESI)**: m/z calculated for $[C_{21}H_{18}NO_2]^+$ $[M + H]^+$: 316.1332, found: 316.1329.

**diphenylmethanone O-(2-ethylbenzoyl) oxime (S20)**



**Chemical Formula:** $C_{22}H_{19}NO_2$

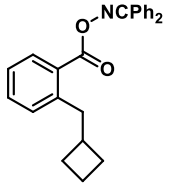
**S20** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.73 – 7.63 (m, 2H), 7.55 – 7.42 (m, 5H), 7.42 – 7.31 (m, 5H), 7.23 (d, *J* = 7.9 Hz, 1H), 7.17 – 7.04 (m, 1H), 2.89 (q, *J* = 7.5 Hz, 2H), 1.16 (t, *J* = 7.5 Hz, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.8, 146.8, 134.7, 133.1, 132.3, 131.0, 130.4, 130.3, 129.6, 129.1, 128.6, 128.4, 128.4, 127.7, 125.7, 27.4, 15.8.

**HRMS (ESI)**: m/z calculated for $[C_{22}H_{20}NO_2]^+$ $[M + H]^+$: 330.1489, found: 330.1490.

**diphenylmethanone *O*-(2-(cyclobutylmethyl)benzoyl) oxime (S21)**



**Chemical Formula:** $C_{25}H_{23}NO_2$

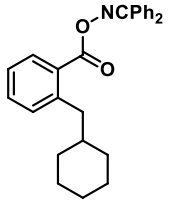
**S21** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.80 – 7.64 (m, 2H), 7.57 – 7.31 (m, 10H), 7.25 – 7.07 (m, 2H), 3.02 (d, *J* = 7.5 Hz, 2H), 2.61 (p, J = 7.8 Hz, 1H), 1.97 (tt, *J* = 10.8, 4.9 Hz, 2H), 1.87 – 1.60 (m, 4H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.3, 164.8, 143.3, 134.6, 132.8, 131.8, 130.9, 130.7, 130.2, 129.5, 128.9, 128.5, 128.3, 128.2, 127.8, 125.6, 40.5, 36.8, 28.1, 18.2.

**HRMS (ESI)**: m/z calculated for $[C_{25}H_{24}NO_2]^+$ $[M + H]^+$: 370.1802, found: 370.1800.

**diphenylmethanone *O*-(2-(cyclohexylmethyl)benzoyl) oxime (S22)**



**Chemical Formula:** $C_{27}H_{27}NO_2$

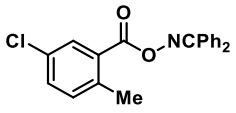
**S22** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.75 – 7.66 (m, 2H), 7.56 – 7.45 (m, 5H), 7.45 – 7.34 (m, 5H), 7.18 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.11 (td, *J* = 7.6, 1.3 Hz, 1H), 2.83 (d, *J* = 7.0 Hz, 2H), 1.72 – 1.53 (m, 6H), 1.24 – 1.09 (m, 3H), 0.95 (ddt, *J* = 17.8, 9.6, 4.9 Hz, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.4, 164.7, 144.0, 134.7, 132.9, 132.0, 131.6, 130.9, 130.3, 129.6, 129.1, 128.7, 128.4, 128.3, 128.1, 125.6, 41.7, 39.4, 33.1, 26.5, 26.3.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{28}NO_2]^+$ $[M + H]^+$: 398.2115, found: 398.2115.

**diphenylmethanone *O*-(5-chloro-2-methylbenzoyl) oxime (S23)**




**Chemical Formula:** $C_{21}H_{16}ClNO_2$

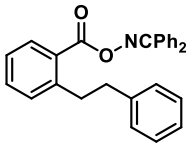
**S23** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.72 – 7.65 (m, 2H), 7.56 – 7.43 (m, 5H), 7.43 – 7.34 (m, 4H), 7.31 (dd, *J* = 8.2, 2.4 Hz, 1H), 7.12 (d, *J* = 8.3 Hz, 1H), 2.43 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.0, 163.5, 139.3, 134.3, 133.1, 132.9, 132.2, 131.4, 131.2, 130.4, 129.8, 129.3, 129.1, 128.5, 128.5, 21.0.

**HRMS (ESI)**: m/z calculated for $[C_{21}H_{16}ClNO_2Na]^+$ $[M + Na]^+$: 372.0762, found: 372.0761.

**diphenylmethanone *O*-(2-phenethylbenzoyl) oxime (S24)**




**Chemical Formula:** $C_{28}H_{23}NO_2$

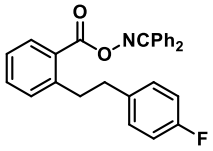
**S24** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.62 (m, 2H), 7.57 – 7.31 (m, 11H), 7.31 – 7.09 (m, 8H), 3.27 – 3.14 (m, 2H), 2.96 – 2.80 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.4, 144.6, 141.9, 134.7, 132.9, 132.3, 131.4, 131.0, 130.6, 129.6, 129.1, 128.7, 128.4, 128.3, 128.2, 127.6, 126.0, 125.8, 38.0, 36.8.

**HRMS (ESI)**: m/z calculated for $[C_{28}H_{23}NO_2Na]^+$ $[M + Na]^+$: 428.1621, found: 428.1617.

**diphenylmethanone *O*-(2-(4-fluorophenethyl)benzoyl) oxime (S25)**




**Chemical Formula:** $C_{28}H_{22}FNO_2$

**S25** was obtained as a white solid.

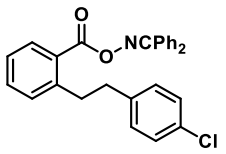
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.71 – 7.67 (m, 2H), 7.53 – 7.42 (m, 5H), 7.42 – 7.34 (m, 5H), 7.20 – 7.10 (m, 4H), 6.97 – 6.89 (m, 2H), 3.24 – 3.13 (m, 2H), 2.95 – 2.79 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) maJor-**S24**: δ 165.7, 164.4, 144.4, 137.5 (d, *J* = 2.02 Hz), 134.7, 133.0, 132.4, 131.4, 131.1, 130.6, 130.1, 129.7, 129.1, 128.7, 128.5, 128.4, 127.6, 126.1, 114.9 (d, *J* = 14.14 Hz), 37.1, 36.9. miner-**S24**:162.1, 160.5, 132.2, 130.0, 129.2, 128.9, 128.6, 128.3, 128.3, 125.9.

**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -117.8.

**HRMS (ESI)**: m/z calculated for $[C_{28}H_{23}FNO_2]^+$ $[M + H]^+$: 452.1856, found: 452.1856.

**diphenylmethanone *O*-(2-(4-chlorophenethyl)benzoyl) oxime (S26)**



**Chemical Formula:** $C_{28}H_{22}ClNO_2$

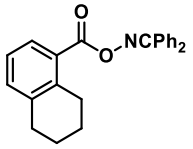
**S26** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.74 – 7.67 (m, 2H), 7.56 – 7.34 (m, 11H), 7.28 – 7.20 (m, 2H), 7.20 – 7.09 (m, 4H), 3.25 – 3.15 (m, 2H), 2.94 – 2.80 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.7, 164.3, 144.3, 140.3, 134.6, 132.9, 132.4, 131.5, 131.4, 131.1, 130.6, 130.1, 129.6, 129.1, 128.7, 128.5, 128.3, 128.3, 127.5, 126.2, 37.3, 36.7.

**HRMS (ESI)**: m/z calculated for $[C_{28}H_{23}ClNO_2]^+$ $[M + H]^+$: 440.1412, found: 440.1413.

**diphenylmethanone *O*-(5,6,7,8-tetrahydronaphthalene-1-carbonyl) oxime (S27)**



**Chemical Formula:** $C_{24}H_{21}NO_2$

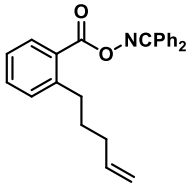
**S27** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.70 (dt, $J$ = 7.1, 1.4 Hz, 2H), 7.55 – 7.44 (m, 4H), 7.44 – 7.33 (m, 5H), 7.24 – 7.12 (m, 1H), 7.04 (t, $J$ = 7.7 Hz, 1H), 3.03 – 2.87 (m, 2H), 2.87 – 2.71 (m, 2H), 1.73 (p, $J$ = 3.4 Hz, 4H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.2, 165.1, 139.1, 138.4, 134.6, 133.4, 133.0, 130.9, 129.4, 128.9, 128.5, 128.4, 128.3, 128.2, 127.8, 124.8, 30.1, 27.5, 22.9, 22.2.

**HRMS (ESI)**: m/z calculated for $[C_{24}H_{22}NO_2]^+$ $[M + H]^+$: 356.1645, found: 356.1644.

**diphenylmethanone *O*-(2-(pent-4-en-1-yl)benzoyl) oxime (S28)**



**Chemical Formula:** $C_{25}H_{23}NO_2$

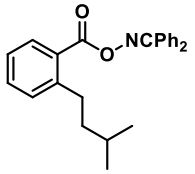
**S28** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.68 (dt, $J$ = 8.7, 1.7 Hz, 2H), 7.53 – 7.29 (m, 11H), 7.28 – 7.18 (m, 1H), 7.10 (tt, $J$ = 7.6, 1.6 Hz, 1H), 5.82 (ddtd, $J$ = 16.9, 10.2, 6.6, 1.7 Hz, 1H), 5.10 – 4.88 (m, 2H), 2.92 (td, $J$ = 7.7, 1.6 Hz, 2H), 2.19 – 2.01 (m, 2H), 1.76 – 1.59 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.6, 145.3, 138.7, 134.7, 133.0, 132.2, 131.1, 131.0, 130.5, 129.6, 129.1, 128.7, 128.5, 128.3, 127.8, 125.8, 114.6, 33.9, 33.8, 30.8.

**HRMS (ESI)**: m/z calculated for $[C_{25}H_{24}NO_2]^+$ $[M + H]^+$: 370.1802, found: 370.1801.

**diphenylmethanone *O*-(2-isopentylbenzoyl) oxime (S29)**



**Chemical Formula:** $C_{25}H_{25}NO_2$

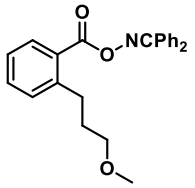
**S29** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.68 (dt, *J* = 8.6, 1.5 Hz, 2H), 7.53 – 7.41 (m, 5H), 7.42 – 7.31 (m, 6H), 7.26 – 7.19 (m, 1H), 7.14 – 7.03 (m, 1H), 2.97 – 2.86 (m, 2H), 1.66 – 1.58 (m, 1H), 1.52 – 1.41 (m, 2H), 0.93 (d, *J* = 6.6, 1.5 Hz, 6H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.4, 164.5, 146.0, 134.7, 132.9, 132.1, 130.9, 130.9, 130.3, 129.6, 129.1, 128.7, 128.4, 128.3, 125.5, 40.9, 32.3, 28.2, 22.5, 22.5.

**HRMS (ESI)**: m/z calculated for $[C_{25}H_{26}NO_2]^+$ $[M + H]^+$: 372.1958, found: 372.1957.

**diphenylmethanone *O*-(2-(3-methoxypropyl)benzoyl) oxime (S30)**



**Chemical Formula:** $C_{24}H_{23}NO_3$

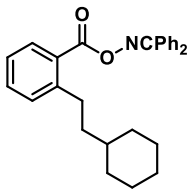
**S30** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.67 (dt, *J* = 7.2, 1.4 Hz, 2H), 7.53 – 7.42 (m, 5H), 7.42 – 7.33 (m, 5H), 7.25 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.11 (td, *J* = 7.6, 1.3 Hz, 1H), 3.38 (t, *J* = 6.4 Hz, 2H), 3.32 (s, 3H), 3.04 – 2.91 (m, 2H), 1.92 – 1.80 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.5, 144.9, 134.7, 132.9, 132.3, 131.3, 131.0, 130.5, 129.7, 129.1, 128.7, 128.5, 128.5, 128.4, 127.7, 125.9, 72.2, 58.5, 31.3, 30.9.

**HRMS (ESI)**: m/z calculated for $[C_{24}H_{24}NO_3]^+$ $[M + H]^+$: 374.1751, found: 374.1751.

**diphenylmethanone *O*-(2-(2-cyclohexylethyl)benzoyl) oxime (S31)**



**Chemical Formula:** $C_{28}H_{29}NO_2$

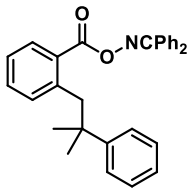
**S31** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.75 – 7.63 (m, 2H), 7.54 – 7.44 (m, 5H), 7.44 – 7.32 (m, 5H), 7.23 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.10 (td, *J* = 7.6, 1.3 Hz, 1H), 3.00 – 2.87 (m, 2H), 1.84 – 1.59 (m, 5H), 1.55 – 1.43 (m, 2H), 1.39 – 1.08 (m, 4H), 0.95 (qd, *J* = 11.8, 3.2 Hz, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.6, 146.2, 134.8, 133.0, 132.2, 131.0, 131.0, 130.4, 129.6, 129.1, 128.8, 128.4, 128.3, 127.7, 125.6, 39.6, 37.9, 33.3, 31.8, 26.8, 26.4.

**HRMS (ESI)**: m/z calculated for $[C_{28}H_{30}NO_2]^+$ $[M + H]^+$: 412.2271, found: 412.2271.

**diphenylmethanone *O*-(2-(2-methyl-2-phenylpropyl)benzoyl) oxime (S32)**



**Chemical Formula:** $C_{30}H_{27}NO_2$

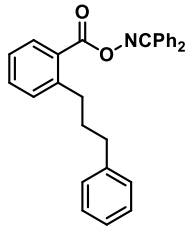
**S32** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.73 – 7.63 (m, 2H), 7.50 – 7.31 (m, 9H), 7.29 – 7.19 (m, 4H), 7.19 – 7.10 (m, 2H), 7.05 (td, *J* = 7.6, 1.4 Hz, 1H), 6.68 (dd, *J* = 7.7, 1.4 Hz, 1H), 3.32 (s, 2H), 1.26 (s, 6H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 165.4, 149.1, 140.5, 134.8, 133.0, 132.7, 131.0, 130.8, 130.1, 129.8, 129.6, 129.2, 128.8, 128.5, 128.3, 128.0, 126.3, 125.9, 125.7, 46.0, 39.6, 27.9.

**HRMS (ESI)**: m/z calculated for $[C_{30}H_{28}NO_2]^+$ $[M + H]^+$: 434.2115, found: 434.2114.

**diphenylmethanone *O*-(2-(3-phenylpropyl)benzoyl) oxime (S33)**



**Chemical Formula:** $C_{29}H_{25}NO_2$

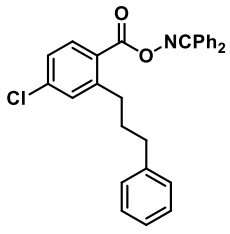
**S33** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.72 – 7.64 (m, 2H), 7.50 – 7.41 (m, 5H), 7.36 (dddd, *J* = 9.2, 7.4, 5.8, 1.6 Hz, 5H), 7.28 – 7.21 (m, 2H), 7.21 – 7.07 (m, 5H), 2.99 – 2.92 (m, 2H), 2.70 – 2.61 (m, 2H), 1.96 – 1.86 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.7, 145.1, 142.4, 134.7, 133.0, 132.2, 131.1, 130.5, 129.6, 129.1, 128.7, 128.5, 128.5, 128.4, 128.3, 127.8, 125.9, 125.7, 35.9, 34.1, 33.3.

**HRMS (ESI)**: m/z calculated for $[C_{29}H_{25}NO_2Na]^+$ $[M + Na]^+$: 442.1778, found: 442.1777.

**diphenylmethanone *O*-(4-chloro-2-(3-phenylpropyl)benzoyl) oxime (S34)**



**Chemical Formula:** $C_{29}H_{24}ClNO_2$

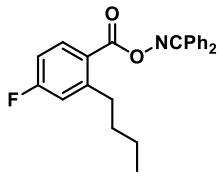
**S34** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.73 – 7.65 (m, 2H), 7.52 – 7.43 (m, 4H), 7.43 – 7.34 (m, 5H), 7.31 (dd, *J* = 8.3, 2.4 Hz, 1H), 7.25 (dd, *J* = 8.2, 6.9 Hz, 2H), 7.14 (dd, *J* = 18.3, 7.8 Hz, 4H), 2.96 – 2.88 (m, 2H), 2.68 – 2.60 (m, 2H), 1.93 – 1.82 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.1, 163.3, 143.5, 142.1, 134.4, 132.7, 132.3, 132.1, 131.5,

131.2, 130.4, 129.8, 129.1, 129.1, 128.5, 128.5, 128.4, 128.3, 125.7, 35.8, 33.4, 33.1.
**HRMS (ESI)**: m/z calculated for $[C_{29}H_{24}ClNO_2Na]^+$ $[M + Na]^+$: 476.1388, found: 476.1382.

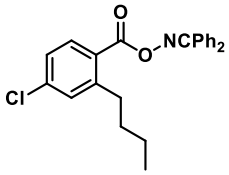
**diphenylmethanone *O*-(2-butyl-4-fluorobenzoyl) oxime (S35)**




**Chemical Formula:** $C_{24}H_{22}FNO_2$

**S35** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.75 – 7.60 (m, 2H), 7.54 – 7.43 (m, 5H), 7.43 – 7.34 (m, 4H), 6.93 (dd, *J* = 9.8, 2.6 Hz, 1H), 6.78 (ddd, *J* = 8.8, 7.9, 2.7 Hz, 1H), 2.99 – 2.84 (m, 2H), 1.64 – 1.47 (m, 2H), 1.36 (h, *J* = 7.3 Hz, 2H), 0.91 (t, *J* = 7.3 Hz, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 164.7 (d, *J* = 254.52Hz), 164.5 (d, J = 191.9 Hz), 163.6, 149.5 (d, *J* = 9.09 Hz), 134.6, 133.0, 132.9, 131.0, 129.6, 129.0, 128.6, 128.4, 128.3, 123.7 (d, *J* = 3.03 Hz), 117.6 (d, *J* = 21.21 Hz), 112.7 (d, *J* = 21.21 Hz), 34.1, 33.4, 22.7, 13.9.
**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -106.9.
**HRMS (ESI)**: m/z calculated for $[C_{24}H_{23}FNO_2]^+$ $[M + H]^+$: 376.1707, found: 376.1706.

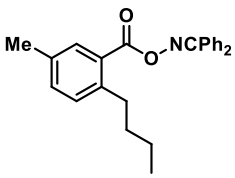
**diphenylmethanone *O*-(2-butyl-4-chlorobenzoyl) oxime (S36)**




**Chemical Formula:** $C_{24}H_{22}ClNO_2$

**S36** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.73 – 7.63 (m, 2H), 7.53 – 7.43 (m, 4H), 7.43 – 7.32 (m, 5H), 7.22 (d, *J* = 2.2 Hz, 1H), 7.08 (dd, *J* = 8.4, 2.2 Hz, 1H), 2.98 – 2.78 (m, 2H), 1.62 – 1.47 (m, 2H), 1.36 (dq, *J* = 14.6, 7.3 Hz, 2H), 0.91 (t, *J* = 7.3 Hz, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.7, 142.7, 135.1, 134.7, 133.1, 133.0, 131.1, 131.0, 131.0, 129.5, 129.1, 128.7, 128.4, 128.3, 127.4, 34.0, 33.6, 22.8, 20.7, 14.0.
**HRMS (ESI)**: m/z calculated for $[C_{24}H_{22}ClNO_2Na]^+$ $[M + Na]^+$: 414.1231, found: 414.1230.

**diphenylmethanone *O*-(2-butyl-5-methylbenzoyl) oxime (S37)**

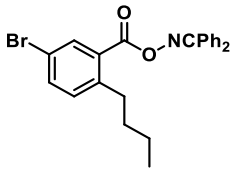



**Chemical Formula:** $C_{25}H_{25}NO_2$

**S37** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.76 – 7.67 (m, 2H), 7.55 – 7.44 (m, 4H), 7.44 – 7.35 (m, 4H), 7.28 (d, *J* = 2.0 Hz, 1H), 7.21 – 7.16 (m, 1H), 7.12 (d, *J* = 7.8 Hz, 1H), 2.93 – 2.82 (m, 2H), 2.21 (s, 3H), 1.60 – 1.47 (m, 2H), 1.35 (dq, *J* = 14.6, 7.3 Hz, 2H), 0.91 (t, *J* = 7.3 Hz, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.7, 142.7, 135.1, 134.7, 133.1, 132.9, 131.1, 131.0, 131.0, 129.5, 129.1, 128.7, 128.4, 128.3, 127.4, 33.9, 33.6, 22.8, 20.7, 14.0.
**HRMS (ESI)**: m/z calculated for $[C_{25}H_{26}NO_2]^+$ $[M + H]^+$: 372.1958, found: 372.1957.

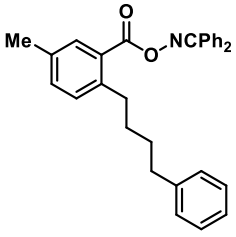
**diphenylmethanone *O*-(5-bromo-2-butylbenzoyl) oxime (S38)**




**Chemical Formula:** $C_{24}H_{22}BrNO_2$

**S38** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.75 – 7.65 (m, 2H), 7.56 (d, *J* = 2.3 Hz, 1H), 7.55 – 7.44 (m, 5H), 7.44 – 7.34 (m, 4H), 7.09 (d, *J* = 8.2 Hz, 1H), 2.94 – 2.77 (m, 2H), 1.52 (tt, *J* = 7.8, 6.4 Hz, 2H), 1.34 (dq, *J* = 14.6, 7.3 Hz, 2H), 0.90 (t, *J* = 7.3 Hz, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.1, 163.2, 144.6, 135.0, 134.4, 133.3, 132.8, 132.7, 131.2, 129.8, 129.4, 129.1, 128.6, 128.5, 128.5, 119.1, 33.7, 33.5, 22.7, 14.0.
**HRMS (ESI)**: m/z calculated for $[C_{24}H_{23}BrNO_2]^+$ $[M + H]^+$: 436.0907, found: 436.0906.

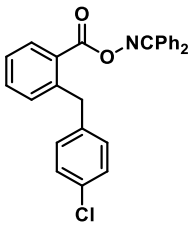
**diphenylmethanone *O*-(5-methyl-2-(4-phenylbutyl)benzoyl) oxime (S39)**




**Chemical Formula:** $C_{31}H_{29}NO_2$

**S39** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.73 – 7.66 (m, 2H), 7.53 – 7.43 (m, 4H), 7.42 – 7.35 (m, 4H), 7.25 (ddd, *J* = 8.9, 5.5, 3.3 Hz, 3H), 7.20 – 7.11 (m, 4H), 7.08 (d, *J* = 7.8 Hz, 1H), 2.88 (t, *J* = 7.3 Hz, 2H), 2.60 (t, *J* = 7.3 Hz, 2H), 2.19 (s, 3H), 1.62 (tdd, *J* = 15.9, 6.6, 2.7 Hz, 4H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.6, 142.8, 142.4, 135.2, 134.6, 133.1, 133.0, 131.1, 131.0, 131.0, 129.5, 129.1, 128.7, 128.4, 128.3, 128.2, 127.4, 125.6, 35.9, 33.7, 31.5, 31.5, 20.7.
**HRMS (ESI)**: m/z calculated for $[C_{31}H_{30}NO_2]^+$ $[M + H]^+$: 448.2271, found: 448.2271.

**diphenylmethanone *O*-(2-(4-chlorobenzyl)benzoyl) oxime (S42)**



**Chemical Formula:** $C_{27}H_{20}ClNO_2$

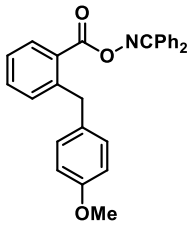
**S42** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.69 – 7.63 (m, 2H), 7.55 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.51 – 7.43 (m, 4H), 7.43 – 7.36 (m, 3H), 7.36 – 7.30 (m, 2H), 7.24 – 7.12 (m, 4H), 7.10 – 7.04 (m, 2H), 4.27 (s, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.7, 164.6, 142.6, 139.1, 134.5, 132.9, 132.4, 131.7, 131.5, 131.1, 130.6, 130.5, 129.6, 129.1, 128.6, 128.4, 128.4, 128.3, 128.1, 126.4, 38.6.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{20}ClNO_2Na]^+$ $[M + Na]^+$: 448.1075, found: 448.1072.

**diphenylmethanone *O*-(2-(4-methoxybenzyl)benzoyl) oxime (S45)**



**Chemical Formula:** $C_{28}H_{23}NO_3$

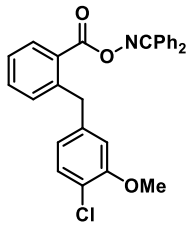
**S45** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ $^1$H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 2H), 7.71 – 7.63 (m, 1H), 7.62 – 7.51 (m, 4H), 7.51 – 7.32 (m, 6H), 7.15 (ddd, *J* = 7.1, 3.8, 2.6 Hz, 2H), 7.11 – 7.04 (m, 2H), 6.85 – 6.77 (m, 2H), 4.23 (s, 2H), 3.77 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 178.8, 165.6, 164.8, 143.6, 134.7, 133.0, 132.3, 131.3, 131.0, 130.5, 129.6, 129.1, 128.7, 128.5, 126.1, 55.3, 38.3.

**HRMS (ESI)**: m/z calculated for $[C_{28}H_{24}NO_3]^+$ $[M + H]^+$: 422.1751, found: 422.1754.

**diphenylmethanone *O*-(2-(4-chloro-3-methoxybenzyl)benzoyl) oxime (S46)**



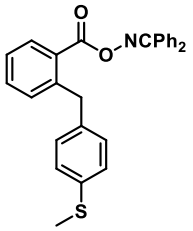
**Chemical Formula:** $C_{28}H_{22}ClNO_3$

**S46** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.71 – 7.61 (m, 2H), 7.59 – 7.52 (m, 1H), 7.52 – 7.29 (m, 8H), 7.24 – 7.12 (m, 3H), 7.03 (s, 1H), 6.77 (d, *J* = 2.0 Hz, 1H), 6.70 – 6.62 (m, 1H), 4.26 (s, 2H), 3.82 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.8, 164.7, 154.8, 142.5, 140.8, 134.6, 132.9, 131.3, 130.5, 129.9, 129.9, 129.1, 128.6, 128.5, 126.4, 121.9, 113.2, 56.1, 39.1.
**HRMS (ESI)**: m/z calculated for $[C_{28}H_{22}ClNO_3Na]^+$ $[M + Na]^+$: 478.1180, found: 478.1191.

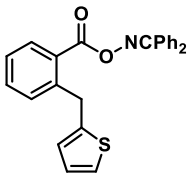
**diphenylmethanone *O*-(2-(4-(methylthio)benzyl)benzoyl) oxime (S47)**




**Chemical Formula:** $C_{28}H_{23}NO_2S$

**S47** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.70 – 7.64 (m, 2H), 7.55 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.50 – 7.43 (m, 4H), 7.43 – 7.31 (m, 5H), 7.16 (td, *J* = 6.9, 1.7 Hz, 4H), 7.12 – 7.05 (m, 2H), 4.26 (s, 2H), 2.45 (s, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.7, 143.0, 137.7, 135.5, 134.6, 132.9, 132.3, 131.4, 131.0, 130.5, 129.7, 129.6, 129.0, 128.6, 128.4, 128.3, 128.1, 126.9, 126.2, 38.7, 16.1.
**HRMS (ESI)**: m/z calculated for $[C_{28}H_{24}NO_2S]^+$ $[M + H]^+$: 460.1342, found: 460.1340.

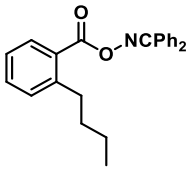
**diphenylmethanone *O*-(2-(thiophen-2-ylmethyl)benzoyl) oxime (S48)**




**Chemical Formula:** $C_{25}H_{19}NO_2S$

**S48** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.70 – 7.62 (m, 2H), 7.54 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.49 – 7.42 (m, 4H), 7.42 – 7.30 (m, 5H), 7.27 – 7.21 (m, 1H), 7.15 (td, *J* = 7.6, 1.3 Hz, 1H), 7.10 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.88 (dd, *J* = 5.2, 3.5 Hz, 1H), 6.78 (dq, *J* = 3.3, 1.1 Hz, 1H), 4.46 (s, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 164.5, 143.2, 142.7, 134.6, 132.9, 132.5, 131.0, 131.0, 130.6, 129.6, 129.1, 128.6, 128.4, 128.3, 127.6, 126.7, 126.5, 125.6, 123.9, 33.6.
**HRMS (ESI)**: m/z calculated for $[C_{25}H_{20}NO_2S]^+$ $[M + H]^+$: 398.1209, found: 398.1207.

**diphenylmethanone *O*-(2-butylbenzoyl) oxime (S50)**




**Chemical Formula:** $C_{24}H_{23}NO_2$

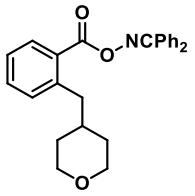
**S50** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.68 (dd, *J* = 8.3, 1.5 Hz, 2H), 7.54 – 7.27 (m, 10H), 7.22 (t, *J* = 7.1

Hz, 1H), 7.09 (td, *J* = 7.6, 1.4 Hz, 1H), 2.96 – 2.83 (m, 2H), 1.61 – 1.48 (m, 2H), 1.35 (q, *J* = 7.5 Hz, 2H), 0.90 (t, *J* = 7.3 Hz, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.4, 164.6, 145.6, 134.7, 132.9, 132.1, 131.0, 130.9, 130.3, 129.5, 129.0, 128.7, 128.4, 128.3, 127.7, 125.6, 34.0, 33.8, 22.7, 14.0.

**HRMS (ESI)**: m/z calculated for $[C_{24}H_{24}NO_2]^+$ $[M + H]^+$: 358.1802, found: 358.1801.

**diphenylmethanone *O*-(2-((tetrahydro-2*H*-pyran-4-yl)methyl)benzoyl) oxime (S51)**




**Chemical Formula:** $C_{26}H_{25}NO_3$

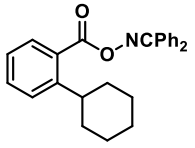
**S51** was obtained as a white solid.

**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ7.73 – 7.61 (m, 2H), 7.55 – 7.43 (m, 5H), 7.37 (ddt, *J* = 10.9, 5.1, 1.8 Hz, 5H), 7.22 – 7.03 (m, 2H), 3.89 (ddd, *J* = 11.4, 4.5, 1.8 Hz, 2H), 3.28 (td, *J* = 11.8, 2.1 Hz, 2H), 2.88 (d, *J* = 7.0 Hz, 2H), 1.86 (ttt, *J* = 11.0, 7.1, 3.8 Hz, 1H), 1.52 (ddd, *J* = 12.9, 4.0, 2.0 Hz, 2H), 1.44 – 1.20 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.3, 142.9, 134.6, 132.8, 132.1, 131.8, 131.0, 130.5, 129.6, 129.0, 128.6, 128.4, 128.2, 127.7, 126.0, 67.9, 41.5, 36.5, 33.0.

**HRMS (ESI)**: m/z calculated for $[C_{26}H_{26}NO_3]^+$ $[M + H]^+$: 400.1907, found: 400.1906.

**diphenylmethanone *O*-(2-cyclohexylbenzoyl) oxime (S52)**




**Chemical Formula:** $C_{26}H_{25}NO_2$

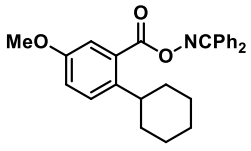
**S46** was obtained as a white solid.

**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ7.76 – 7.63 (m, 2H), 7.55 – 7.29 (m, 11H), 7.10 (td, *J* = 7.4, 1.6 Hz, 1H), 3.34 (tt, *J* = 11.3, 2.8 Hz, 1H), 1.90 – 1.67 (m, 5H), 1.49 – 1.33 (m, 4H), 1.25 (tt, *J* = 13.0, 2.8 Hz, 1H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 165.4, 149.3, 134.7, 132.7, 131.9, 130.9, 129.7, 129.5, 129.0, 128.6, 128.4, 128.2, 128.2, 126.9, 125.3, 40.0, 34.3, 26.8, 26.2.

**HRMS (ESI)**: m/z calculated for $[C_{26}H_{26}NO_2]^+$ $[M + H]^+$: 384.1958, found: 384.1956.

**diphenylmethanone *O*-(2-cyclohexyl-5-methoxybenzoyl) oxime (S53)**




**Chemical Formula:** $C_{27}H_{27}NO_3$

**S53** was obtained as a white solid.

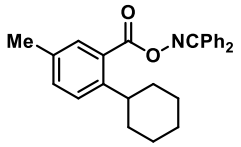
**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ7.75 – 7.62 (m, 2H), 7.53 – 7.31 (m, 8H), 7.26 (d, *J* = 8.6 Hz, 1H),

6.96 (dd, *J* = 8.7, 2.9 Hz, 1H), 6.88 (d, *J* = 2.9 Hz, 1H), 3.60 (s, 3H), 3.30 (tt, *J* = 8.6, 3.3 Hz, 1H), 1.83 – 1.65 (m, 5H), 1.48 – 1.09 (m, 5H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 164.9, 156.8, 142.0, 134.6, 133.0, 131.0, 129.4, 129.0, 128.6, 128.5, 128.4, 128.3, 128.1, 119.1, 113.5, 55.2, 39.3, 34.5, 26.9, 26.3.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{28}NO_3]^+$ $[M + H]^+$: 414.2064, found: 414.2062.

**diphenylmethanone *O*-(2-cyclohexyl-5-methylbenzoyl) oxime (S54)**




**Chemical Formula:** $C_{27}H_{27}NO_2$

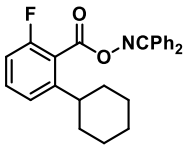
**S54** was obtained as a white solid.

**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ7.76 – 7.61 (m, 2H), 7.45 (ddd, *J* = 6.4, 2.9, 1.6 Hz, 4H), 7.42 – 7.33 (m, 4H), 7.27 – 7.12 (m, 3H), 3.28 (tt, *J* = 11.1, 2.8 Hz, 1H), 2.20 (s, 3H), 1.83 – 1.62 (m, 5H), 1.47 – 1.09 (m, 5H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.5, 165.4, 146.5, 134.8, 134.6, 132.9, 132.7, 131.0, 130.4, 129.5, 129.0, 128.7, 128.4, 128.2, 127.9, 126.9, 39.6, 34.4, 26.9, 26.2, 20.7.

**HRMS (ESI)**: m/z calculated for $[C_{27}H_{28}NO_2]^+$ $[M + H]^+$: 398.2115, found: 398.2113.

**diphenylmethanone *O*-(2-cyclohexyl-6-fluorobenzoyl) oxime (S55)**




**Chemical Formula:** $C_{26}H_{24}FNO_2$

**S55** was obtained as a white solid.

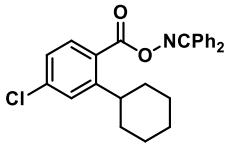
**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ7.51 (td, *J* = 7.8, 4.2 Hz, 1H), 7.40 (s, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 8.8 Hz, 1H), 1.93 – 1.81 (m, 5H), 1.65 – 1.47 (m, 4H), 1.46 – 1.33 (m, 1H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.8, 163.8, 160.6, 158.1, 148.3 (d, *J* = 2.02 Hz), 134.6, 132.4, 131.5 (d, *J* = 9.09 Hz), 131.1, 129.7, 129.2, 128.8, 128.4, 128.2, 121.9 (d, *J* = 3.03 Hz), 119.8 (d, *J* = 16.16 Hz), 112.9 (d, *J* = 21.21 Hz), 41.5, 34.1, 26.6, 26.0.

**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -114.2.

**HRMS (ESI)**: m/z calculated for $[C_{26}H_{25}FNO_2]^+$ $[M + H]^+$: 402.1864, found: 402.1862.

**diphenylmethanone *O*-(4-chloro-2-cyclohexylbenzoyl) oxime (S56)**



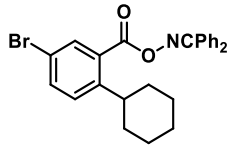

**Chemical Formula:** $C_{26}H_{24}ClNO_2$

**S56** was obtained as a white solid.

**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ7.72 – 7.63 (m, 2H), 7.52 – 7.43 (m, 4H), 7.43 – 7.29 (m, 6H), 7.07 (dd, *J* = 8.4, 2.1 Hz, 1H), 3.41 – 3.26 (m, 1H), 1.86 – 1.67 (m, 5H), 1.46 – 1.14 (m, 5H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.7, 164.5, 151.7, 138.4, 134.5, 132.7, 131.3, 131.1, 129.7, 129.1, 128.6, 128.4, 128.3, 127.5, 126.4, 125.7, 39.9, 34.2, 26.7, 26.1.
**HRMS (ESI)**: m/z calculated for $[C_{26}H_{25}ClNO_2]^+$ $[M + H]^+$: 418.1568, found: 418.1566.

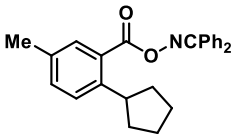
**diphenylmethanone *O*-(5-bromo-2-cyclohexylbenzoyl) oxime (S57)**




**Chemical Formula:** $C_{26}H_{24}BrNO_2$

**S57** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.69 (d, *J* = 7.7 Hz, 2H), 7.55 – 7.34 (m, 10H), 7.23 (d, *J* = 8.4 Hz, 1H), 3.28 (t, *J* = 11.6 Hz, 1H), 1.85 – 1.68 (m, 5H), 1.45 – 1.26 (m, 4H), 1.21 (q, *J* = 12.8 Hz, 1H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.2, 163.9, 148.4, 134.8, 134.4, 132.6, 132.6, 131.2, 129.9, 129.8, 129.1, 128.8, 128.5, 128.5, 128.4, 118.8, 39.7, 34.2, 26.7, 26.1.
**HRMS (ESI)**: m/z calculated for $[C_{26}H_{25}BrNO_2]^+$ $[M + H]^+$: 462.1063, found: 462.1063.

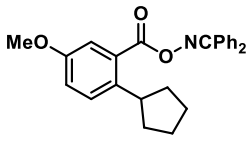
**diphenylmethanone *O*-(2-cyclopentyl-5-methylbenzoyl) oxime (S58)**




**Chemical Formula:** $C_{26}H_{25}NO_2$

**S58** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.68 (dd, *J* = 7.2, 1.8 Hz, 2H), 7.52 – 7.42 (m, 4H), 7.38 (td, *J* = 6.9, 2.3 Hz, 4H), 7.25 (d, *J* = 8.7 Hz, 1H), 7.20 (dd, *J* = 6.5, 2.0 Hz, 2H), 3.60 (tt, *J* = 9.6, 7.5 Hz, 1H), 2.21 (s, 3H), 2.01 – 1.88 (m, 2H), 1.80 – 1.54 (m, 4H), 1.54 – 1.38 (m, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.7, 165.6, 144.9, 134.8, 134.6, 133.0, 132.7, 131.0, 130.2, 129.5, 129.0, 128.8, 128.6, 128.4, 128.3, 126.8, 41.3, 34.8, 25.6, 20.6.
**HRMS (ESI)**: m/z calculated for $[C_{26}H_{26}NO_2]^+$ $[M + H]^+$: 384.1958, found: 384.1958.

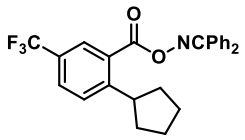
**diphenylmethanone *O*-(2-cyclopentyl-5-methoxybenzoyl) oxime (S59)**




**Chemical Formula:** $C_{26}H_{25}NO_3$

**S59** was obtained as a white solid.
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.74 – 7.59 (m, 2H), 7.46 (qd, *J* = 5.3, 1.7 Hz, 4H), 7.42 – 7.34 (m, 4H), 7.31 – 7.24 (m, 1H), 6.96 (dd, *J* = 8.7, 2.9 Hz, 1H), 6.88 (d, *J* = 2.9 Hz, 1H), 3.62 (s, 3H), 2.02 – 1.90 (m, 2H), 1.82 – 1.54 (m, 4H), 1.46 (ddt, *J* = 16.7, 11.2, 7.8 Hz, 2H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.6, 165.2, 156.8, 140.2, 134.6, 133.1, 131.1, 129.4, 129.4, 129.0, 128.5, 128.4, 128.3, 128.1, 119.2, 113.3, 55.3, 41.0, 34.8, 25.6.
**HRMS (ESI)**: m/z calculated for $[C_{26}H_{26}NO_3]^+$ $[M + H]^+$: 384.1958, found: 384.1958.

**diphenylmethanone *O*-(2-cyclopentyl-5-(trifluoromethyl)benzoyl) oxime (S60)**



**Chemical Formula:** $C_{26}H_{22}F_3NO_2$

**S60** was obtained as a white solid.

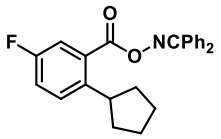
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.76 – 7.56 (m, 4H), 7.56 – 7.33 (m, 9H), 3.77 (ddd, *J* = 17.2, 9.5, 7.5 Hz, 1H), 2.04 (dtt, *J* = 10.9, 5.7, 2.4 Hz, 2H), 1.88 – 1.59 (m, 4H), 1.59 – 1.43 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.4, 164.1, 152.5, 134.3, 132.6, 131.3, 129.8, 129.3, 129.1, 128.5, 128.4, 128.4, 128.2, 127.7 (q, *J* = 33.33 Hz), 127.7, 126.8 (q, *J* = 4.04 Hz), 123.6 (q, *J* = 272.7 Hz), 41.6, 34.9, 25.8.

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

**HRMS (ESI)**: m/z calculated for $[C_{26}H_{23}F_3NO_2]^+$ $[M + H]^+$: 438.1675, found: 438.1674.

**diphenylmethanone *O*-(2-cyclopentyl-5-fluorobenzoyl) oxime (S61)**



**Chemical Formula:** $C_{25}H_{22}FNO_2$

**S61** was obtained as a white solid.

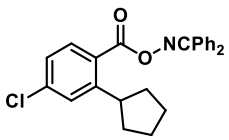
**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.68 (dt, *J* = 7.2, 1.4 Hz, 2H), 7.53 – 7.44 (m, 4H), 7.44 – 7.29 (m, 5H), 7.09 (ddd, *J* = 16.8, 8.5, 2.8 Hz, 2H), 3.62 (tt, *J* = 9.7, 7.5 Hz, 1H), 2.05 – 1.91 (m, 2H), 1.75 (ttd, *J* = 9.5, 6.9, 4.2 Hz, 2H), 1.64 (dtd, *J* = 12.3, 8.0, 3.2 Hz, 2H), 1.53 – 1.39 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.0, 164.5 (d, *J* = 3.03 Hz), 161.2, 158.7, 143.8 (d, *J* = 3.03 Hz), 134.4, 132.7, 131.2, 130.3 (d, *J* = 7.07 Hz), 129.7, 129.1, 128.7 (d, *J* = 7.07 Hz), 128.5, 128.5, 128.4, 119.0 (d, *J* = 21.21 Hz), 116.1 (d, *J* = 23.23 Hz), 41.1, 34.9, 25.6.

**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -117.2.

**HRMS (ESI)**: m/z calculated for $[C_{25}H_{23}FNO_2]^+$ $[M + H]^+$: 388.1707, found: 388.1707.

**diphenylmethanone *O*-(4-chloro-2-cyclopentylbenzoyl) oxime (S62)**



**Chemical Formula:** $C_{25}H_{22}ClNO_2$

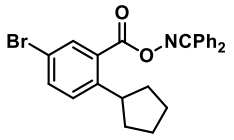
**S62** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.67 (dt, *J* = 7.1, 1.4 Hz, 2H), 7.54 – 7.29 (m, 10H), 7.08 (dd, *J* = 8.4, 2.1 Hz, 1H), 3.65 (tt, *J* = 9.6, 7.5 Hz, 1H), 1.99 (dq, *J* = 16.7, 5.9 Hz, 2H), 1.76 (qdt, *J* = 10.4, 7.9, 4.1 Hz, 2H), 1.63 (qd, *J* = 8.1, 3.9 Hz, 2H), 1.55 – 1.37 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.8, 164.9, 150.4, 138.3, 134.5, 132.8, 131.2, 131.2, 129.7, 129.1, 128.6, 128.5, 128.4, 127.3, 125.7, 41.6, 34.7, 25.7.

**HRMS (ESI)**: m/z calculated for $[C_{25}H_{23}ClNO_2]^+$ $[M + H]^+$: 404.1412, found: 404.1408.

**diphenylmethanone *O*-(5-bromo-2-cyclopentylbenzoyl) oxime (S63)**




**Chemical Formula:** $C_{25}H_{22}BrNO_2$

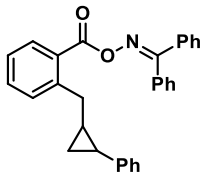
**S63** was obtained as a white solid.

**$^1$H NMR** (400 MHz, $CDCl_3$) δ7.75 – 7.61 (m, 2H), 7.55 – 7.44 (m, 6H), 7.44 – 7.32 (m, 4H), 7.23 (d, *J* = 8.2 Hz, 1H), 3.60 (ddd, *J* = 17.1, 9.6, 7.5 Hz, 1H), 1.97 (dqd, *J* = 10.7, 6.4, 2.4 Hz, 2H), 1.83 – 1.54 (m, 4H), 1.53 – 1.37 (m, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.2, 164.2, 147.1, 134.8, 134.4, 132.7, 132.4, 131.2, 130.8, 129.8, 129.1, 128.8, 128.5, 128.5, 128.5, 118.7, 41.3, 34.8, 25.7.

**HRMS (ESI)**: m/z calculated for $[C_{25}H_{23}BrNO_2]^+$ $[M + H]^+$: 448.0907, found: 448.0905.

**diphenylmethanone O-(2-((2-phenylcyclopropyl)methyl)benzoyl) oxime (S64)**



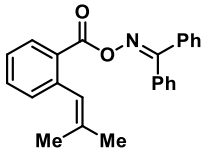

**Chemical Formula:** $C_{30}H_{25}NO_2$

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.62 (m, 2H), 7.57 – 7.28 (m, 13H), 7.27 – 7.06 (m, 4H), 7.06 – 6.93 (m, 2H), 3.01 (qd, *J* = 15.1, 6.8 Hz, 2H), 1.76 (dt, *J* = 9.1, 4.9 Hz, 1H), 1.48 – 1.30 (m, 1H), 0.91 (ddt, *J* = 14.1, 8.5, 5.1 Hz, 2H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.51, 164.86, 143.68, 143.40, 134.64, 132.97, 132.31, 131.01, 130.38, 129.58, 129.19, 129.05, 128.99, 128.64, 128.42, 128.33, 128.27, 128.22, 128.20, 127.90, 127.81, 125.96, 125.74, 125.26, 37.77, 23.78, 23.46, 16.03.

**HRMS (ESI)**: m/z calculated for $[C_{30}H_{26}NO_2]^+$ $[M + H]^+$: 432.1958, found: 432.1958.

**diphenylmethanone *O*-(2-(2-methylprop-1-en-1-yl)benzoyl) oxime (S65)**




Chemical Formula: $C_{24}H_{21}NO_2$

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.70 – 7.65 (m, 2H), 7.56 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.50 – 7.43 (m, 4H), 7.42 – 7.34 (m, 5H), 7.22 (d, *J* = 7.7 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.55 (s, 1H), 1.84 (d, *J* = 1.5 Hz, 3H), 1.66 (d, *J* = 1.4 Hz, 3H).

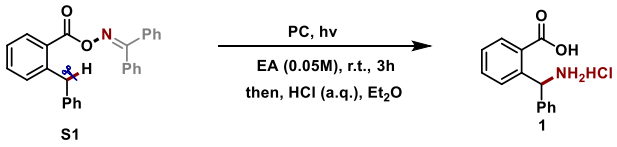
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 165.1, 164.7, 140.4, 135.2, 134.8, 132.8, 131.6, 131.1, 131.1, 130.9, 130.0, 129.5, 129.0, 128.8, 128.3, 128.2, 128.2, 127.9, 125.9, 124.3, 26.2, 19.2.

**HRMS (ESI)**: m/z calculated for $[C_{24}H_{22}NO_2]^+$ $[M + H]^+$: 356.1645, found: 356.1645.

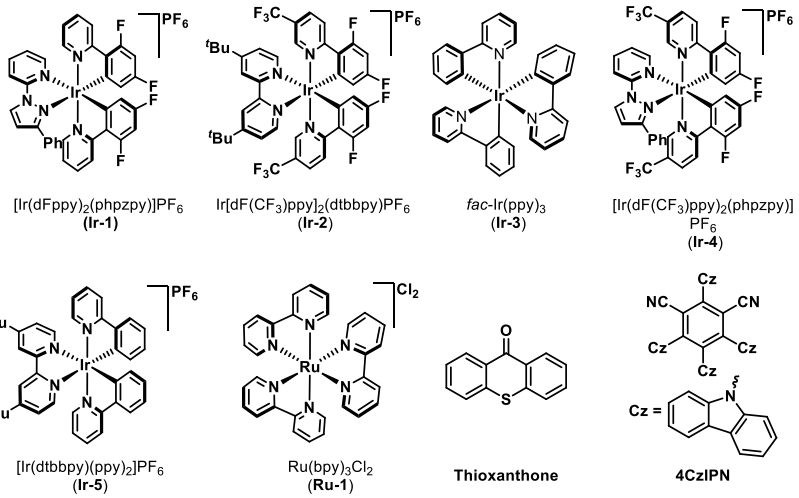
## 2.4 Additional Reaction Optimization

### Screening reaction parameters for γ C−H amination

**Supplementary Table 1: Screening of photocatalyst[a]**

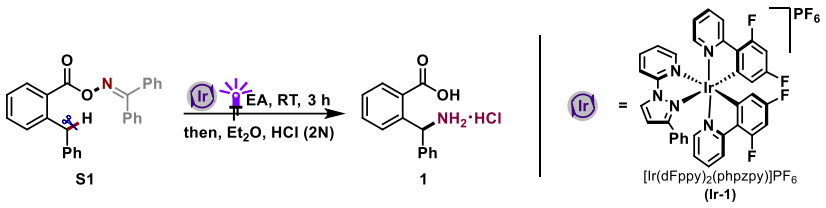



| Entry | PC | Yield |
|---|---|---|
| 1 | $[Ir(dFppy)_2(phpzpy)]PF_6$ (**Ir-1**) | 82% |
| 2 | $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (**Ir-2**) | 57% |
| 3[b] | *fac*-$Ir(ppy)_3$ (**Ir-3**) | N.D. |
| 4 | $[Ir(dF(CF_3)ppy)_2(phpzpy)]PF_6$ (**Ir-4**) | 64% |
| 5 | $[Ir(dtbbpy)(ppy)_2]PF_6$ (**Ir-5**) | 24% |
| 6[b] | $Ru(bpy)_3Cl_2$ (**Ru-1**) | N.D. |
| 7[c] | Thioxanthone | 73% |
| 8[c] | 4CzIPN | 58% |




[a]Reaction conditions: **S1** (0.2 mmol, 1.0 equiv.), PC (1 mol%), EA (4 mL), irradiation by purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature under argon for 3 h, then the solvents were removed under reduced pressure, acidified by 2 mL HCl (2 N) and stirred in $Et_2O$ (2 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (2.0 mL) and extracted with $Et_2O$ (10 mL x 3). The water layer was combined, and water was removed *in vacuo*, and product **1** was obtained as a white solid. [b]The reaction was irradiated by blue LEDs ($\lambda_{max}$ = 450 nm). [c]PC (5 mol%) was used. N.D. = not detected.

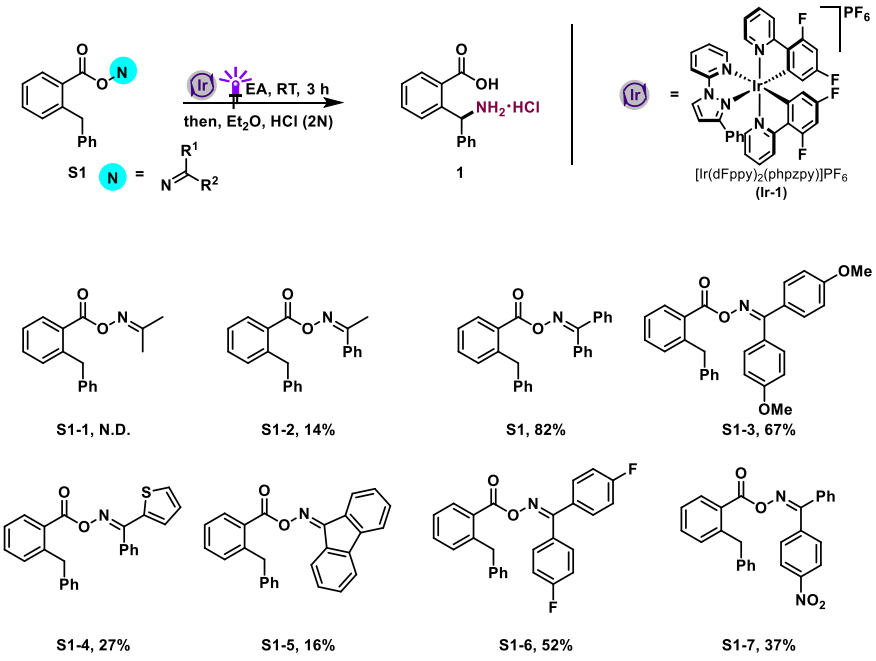
**Supplementary Table 2: Screening of solvents[a]**




| Entry | Solvnt | Yield |
|---|---|---|
| 1 | EA | 82% |
| 2 | $CH_3CN$ | 44% |
| 3 | DCM | 60% |
| 4 | $CH_3COCH_3$ | 45% |
| 5 | DCE | 57% |
| 6 | THF | 68% |
| 7 | MeOH | 49% |
| 8 | Toluene | 58% |
| 9 | 1,4-dioxane | 64% |

[a]Reaction conditions: **S1** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), solvent (4 mL), irradiation by purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature under argon for 3 h, then the solvents were removed under reduced pressure, acidified by 2 mL HCl (2 N) and stirred in $Et_2O$ (2 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (2.0 mL) and extracted with $Et_2O$ (10 mL x 3). The water layer was combined, and water was removed *in vacuo*, and product **1** was obtained as a white solid.

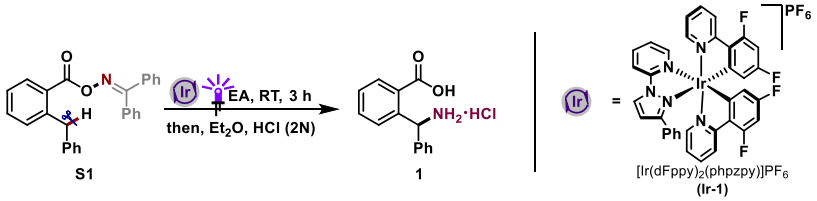
**Supplementary Figure 1: Screening the effect of different activators[a]**




[a]Reaction conditions: **S1** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), EA (4 mL),

irradiation by purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature under argon for 3 h, then the solvents were removed under reduced pressure, acidified by 2 mL HCl (2 N) and stirred in $Et_2O$ (2 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (2.0 mL) and extracted with $Et_2O$ (10 mL x 3). The water layer was combined, and water was removed *in vacuo*, and product **1** was obtained as a white solid.

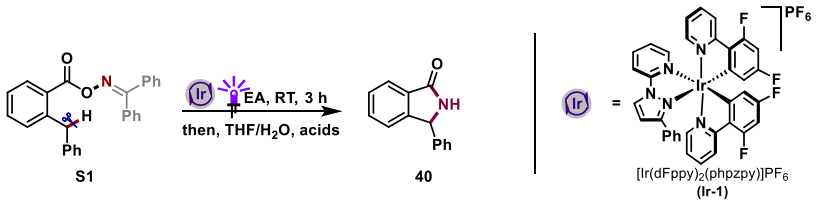
**Supplementary Table 3: Screening the effect of triplet state inhibitor**[a]




| Entry | Conditions | Yield |
|---|---|---|
| 1 | none | 82% |
| 2 | no light | N.D. |
| 3 | add 3 equiv. *E*-stilbene | 54% |
| 4 | in air | 39% |

[a]Reaction conditions: **S1** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), EA (4 mL), irradiation by purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature under argon for 3 h, then the solvents were removed under reduced pressure, acidified by 2 mL HCl (2 N) and stirred in $Et_2O$ (2 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (2.0 mL) and extracted with $Et_2O$ (10 mL x 3). The water layer was combined, and water was removed *in vacuo*, and product **1** was obtained as a white solid.

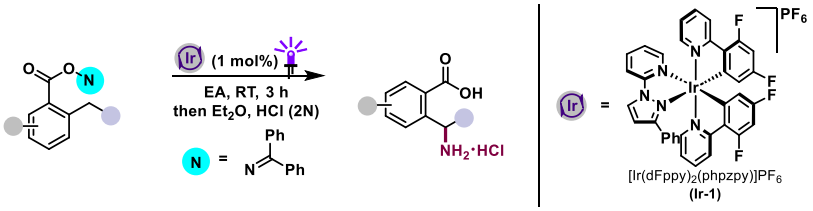
**Supplementary Table 4: Screening of acids for the synthesis of γ-lactams**[a]



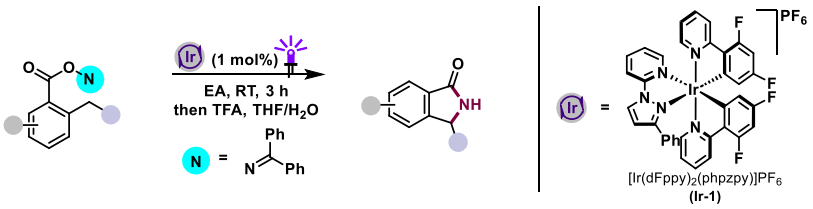

| Entry | Acid | Yield |
|---|---|---|
| 1 | TFA | 71% |
| 2 | HCl (2N) | 41% |
| 3 | AcOH | 56% |
| 4 | $H_2SO_4$ | 45% |
| 5 | PTSA | 37% |

[a]Reaction conditions: **S1** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), EA (4 mL), irradiation by purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature under argon for 3 h, then the solvents were removed under reduced pressure, stirred in THF (2 mL)/$H_2O$ (50 equiv. 180μL), acid (1 equiv.) for 12 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (eluent: *n*-hexanes/10% methanol-EtOAc solution = 2:1), and product **40** was obtained as a white solid. TFA, trifluoroacetic acid. PTSA, *p*-toluenesulfonic acid.

## 2.5 Experimental Procedures



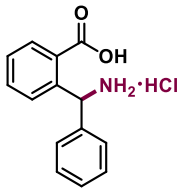

**Procedure:** To an oven-dried glass tube (10 mL) equipped with a magnetic stir bar was added the oxime esters (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), and EA (0.05 M). Then the vessel was bubbled with a stream of argon for 20 min via a syringe needle. The tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 3 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion of the reaction, the solvents were removed under reduced pressure, acidified by 2 mL HCl (2 N) and stirred in $Et_2O$ (2 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (2.0 mL) and extracted with $Et_2O$ (10 mL x 3). The water layer was combined, and water was removed *in vacuo*, and the product was obtained as a white solid.



**Procedure:** To an oven-dried glass tube (10 mL) equipped with a magnetic stir bar were added the oxime esters (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), and EA (0.05 M). Then the vessel was bubbled with a stream of argon for 20 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 3 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion of the reaction, the solvents were removed under reduced pressure, and stirred in THF (2 mL)/$H_2O$ (50 equiv. 180μL), TFA (1.0 equiv.) for 12 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (eluent: *n*-hexanes/10% methanol-EtOAc solution = 2:1), and the product was obtained as a white solid.

## 2.6 Characterization data of products

**2-(amino(phenyl)methyl)benzoic acid hydrochloride (1)**




**Chemical Formula:** $C_{14}H_{14}ClNO_2$

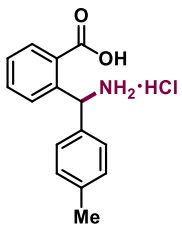
Product **1** was obtained as a white solid (43.2 mg, 82%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.95 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.63 (td, *J* = 7.6, 1.5 Hz, 1H), 7.51 (td, *J* = 7.7, 1.2 Hz, 1H), 7.47 – 7.35 (m, 4H), 7.29 (dd, *J* = 7.5, 2.2 Hz, 2H), 6.35 (s, 1H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 170.9, 136.4, 135.9, 133.1, 131.4, 130.1, 129.3, 129.0, 128.9, 128.5, 127.3, 54.9.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{14}NO_2]^+$ $[M]^+$: 228.1019, found: 228.1018.

**2-(amino(*p*-tolyl)methyl)benzoic acid hydrochloride (2)**




**Chemical Formula:** $C_{15}H_{16}ClNO_2$

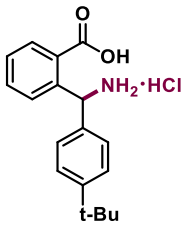
Product **2** was obtained as a white solid (41.8 mg, 75%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.95 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.63 (td, *J* = 7.7, 1.4 Hz, 1H), 7.56 – 7.47 (m, 1H), 7.43 (d, *J* = 7.9 Hz, 1H), 7.23 (d, *J* = 7.9 Hz, 2H), 7.16 (d, *J* = 7.9 Hz, 2H), 6.31 (s, 1H), 2.28 (s, 3H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.9, 139.3, 136.6, 133.0, 132.9, 131.4, 130.1, 129.6, 129.3, 128.3, 127.3, 54.7, 20.1.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{16}NO_2]^+$ $[M]^+$: 242.1176, found: 242.1175.

**2-(amino(4-(tert-butyl)phenyl)methyl)benzoic acid hydrochloride (3)**




**Chemical Formula:** $C_{18}H_{22}ClNO_2$

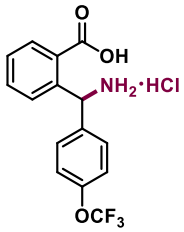
Product **3** was obtained as a white solid (43.7 mg, 68%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.94 (d, *J* = 7.7 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 1H), 7.37 (dd, *J* = 12.6, 8.0 Hz, 3H), 7.18 (d, *J* = 8.2 Hz, 2H), 6.32 (s, 1H), 1.16 (s, 9H).

$^{13}$C NMR (151 MHz, $D_2O$) δ 170.6, 152.3, 136.8, 133.2, 133.0, 131.6, 129.8, 129.3, 128.6, 127.1, 126.0, 54.5, 34.0, 30.4.

**HRMS (ESI)**: m/z calculated for $[C_{18}H_{22}NO_2]^+$ $[M]^+$: 284.1645, found: 284.1641.

**2-(amino(4-(trifluoromethoxy)phenyl)methyl)benzoic acid hydrochloride (4)**




**Chemical Formula:** $C_{15}H_{13}ClF_3NO_3$

Product **4** was obtained as a white solid (42.0 mg, 60%)

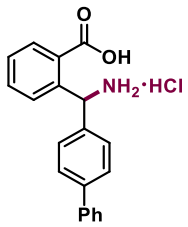
**$^1$H NMR** (600 MHz, $D_2O$) δ 8.02 (d, *J* = 7.7 Hz, 1H), 7.71 (t, *J* = 7.7 Hz, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.48 (d, *J* = 7.9 Hz, 1H), 7.41 (d, *J* = 8.7 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 6.44 (s, 1H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.9, 149.0, 136.1, 134.8, 133.2, 131.6, 130.3, 129.6, 129.2, 128.5, 121.4, 121.2 (q, *J* = 256.7 Hz), 54.5.

**$^{19}$F NMR** (565 MHz, $D_2O$) δ -56.85.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{13}F_3NO_3]^+$ $[M]^+$: 312.0842, found: 312.0839.

**2-([1,1'-biphenyl]-4-yl(amino)methyl)benzoic acid hydrochloride (5)**




**Chemical Formula:** $C_{20}H_{18}ClNO_2$

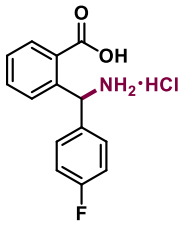
Product **5** was obtained as a white solid (49.5 mg, 73%)

**$^1$H NMR** (600 MHz, MeOD) δ 8.15 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.68 (td, *J* = 8.1, 1.7 Hz, 3H), 7.64 – 7.60 (m, 2H), 7.58 – 7.50 (m, 2H), 7.49 – 7.41 (m, 4H), 7.37 – 7.32 (m, 1H), 6.65 (s, 1H).

**$^{13}$C NMR** (151 MHz, MeOD) δ 170.2, 142.9, 141.3, 139.3, 136.8, 134.1, 133.1, 131.4, 131.0, 130.4, 130.1, 130.0, 129.0, 128.8, 128.5, 128.5, 128.0, 55.6.

**HRMS (ESI)**: m/z calculated for $[C_{20}H_{18}NO_2]^+$ $[M]^+$: 304.1332, found: 304.1328.

**2-(amino(4-fluorophenyl)methyl)benzoic acid hydrochloride (6)**




**Chemical Formula:** $C_{14}H_{13}ClFNO_2$

Product **6** was obtained as a white solid (33.2mg, 59%)

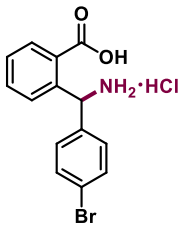
**$^1$H NMR** (400 MHz, $D_2O$) δ 7.94 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.67 (td, *J* = 7.7, 1.5 Hz, 1H), 7.55 (td, *J* = 7.6, 1.2 Hz, 1H), 7.46 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.37 – 7.26 (m, 2H), 7.20 – 7.11 (m, 2H), 6.33 (s, 1H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.4, 163.7, 161.3, 135.9, 132.7, 131.9 (d, *J* = 3.03 Hz), 131.2, 130.9, 129.5 (d, *J* = 9.09 Hz), 129.4, 128.1, 115.8 (d, *J* = 22.22 Hz), 54.6.

**$^{19}$F NMR** (376 MHz, $D_2O$) δ -113.49.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{13}FNO_2]^+$ $[M]^+$: 246.0925, found: 246.0924.

**2-(amino(4-bromophenyl)methyl)benzoic acid hydrochloride (7)**




**Chemical Formula:** $C_{14}H_{13}BrClNO_2$

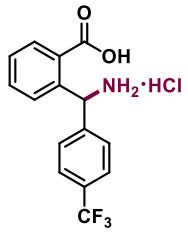
Product **7** was obtained as a white solid (46.1mg, 67%)

$^1$H NMR (400 MHz, $D_2O$) δ 7.94 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.61 (td, *J* = 7.7, 1.5 Hz, 1H), 7.55 – 7.46 (m, 3H), 7.43 – 7.33 (m, 1H), 7.19 – 7.09 (m, 2H), 6.30 (s, 1H).

$^{13}$C NMR (101 MHz, $D_2O$) δ 170.6, 136.0, 135.0, 133.1, 131.9, 131.6, 129.9, 129.4, 129.1, 128.31, 122.4, 54.4.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{13}BrNO_2]^+$ $[M]^+$: 306.0124, found: 306.0124.

**2-(amino(4-(trifluoromethyl)phenyl)methyl)benzoic acid hydrochloride (8)**




**Chemical Formula:** $C_{15}H_{13}ClF_3NO_2$

Product **8** was obtained as a white solid (37.7 mg, 57%)

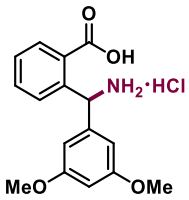
**$^1$H NMR** (600 MHz, $D_2O$) δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.69 (td, *J* = 7.7, 1.5 Hz, 1H), 7.60 (td, *J* = 7.7, 1.2 Hz, 1H), 7.53 – 7.47 (m, 2H), 7.47 – 7.42 (m, 1H), 6.45 (s, 1H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 171.1, 134.0, 135.7, 133.0, 131.5, 130.7, 130.1 (q, *J* = 33.22 Hz), 129.7, 128.8, 127.8, 125.9 (q, *J* = 4.53 Hz), 123.9 (q, *J* = 271.8 Hz), 54.8.

**$^{19}$F NMR** (565 MHz, $D_2O$) δ -62.6.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{13}F_3NO_2]^+$ $[M]^+$: 296.0893, found: 296.0890.

**2-(amino(3,5-dimethoxyphenyl)methyl)benzoic acid hydrochloride (9)**




**Chemical Formula:** $C_{16}H_{18}ClNO_4$

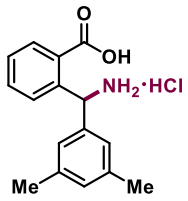
Product **9** was obtained as a white solid (41.3 mg, 64%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.94 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.59 (td, *J* = 7.7, 1.5 Hz, 1H), 7.48 (td, *J* = 7.6, 1.3 Hz, 1H), 7.39 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.49 (q, *J* = 2.0 Hz, 3H), 6.26 (s, 1H), 3.71 (s, 6H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.0, 160.6, 138.4, 136.0, 133.0, 131.4, 130.4, 129.5, 128.6, 105.9, 100.3, 55.4, 54.8.

**HRMS (ESI)**: m/z calculated for $[C_{16}H_{18}NO_4]^+$ $[M]^+$: 288.1230, found: 288.1229.

**2-(amino(3,5-dimethylphenyl)methyl)benzoic acid hydrochloride (10)**




**Chemical Formula:** $C_{16}H_{18}ClNO_2$

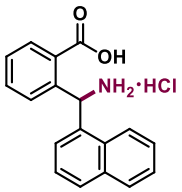
Product **10** was obtained as a white solid (45.3 mg, 78%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.94 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.61 (td, *J* = 7.7, 1.5 Hz, 1H), 7.49 (td, *J* = 7.7, 1.2 Hz, 1H), 7.41 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.02 (d, *J* = 1.9 Hz, 1H), 6.97 – 6.79 (m, 2H), 6.27 (s, 1H), 2.21 (s, 6H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 170.9, 139.2, 136.5, 136.0, 133.0, 131.4, 130.1, 130.1, 129.3, 128.5, 124.8, 54.8, 20.3.

**HRMS (ESI)**: m/z calculated for $[C_{16}H_{18}NO_2]^+$ $[M]^+$: 256.1332, found: 256.1334.

**2-(amino(naphthalen-1-yl)methyl)benzoic acid hydrochloride (11)**




**Chemical Formula:** $C_{18}H_{16}ClNO_2$

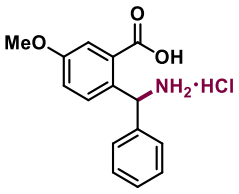
Product **11** was obtained as a white solid (40.8 mg, 65%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 8.02 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.88 (dh, *J* = 7.2, 3.6 Hz, 3H), 7.84 – 7.79 (m, 1H), 7.65 (td, *J* = 7.7, 1.5 Hz, 1H), 7.61 – 7.53 (m, 3H), 7.47 (d, *J* = 7.9 Hz, 1H), 7.34 (dd, *J* = 8.6, 2.0 Hz, 1H), 6.55 (s, 1H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.7, 136.5, 133.5, 133.2, 132.7, 132.6, 131.6, 130.0, 129.5, 128.9, 128.7, 128.1, 127.7, 127.2, 127.1, 126.2, 125.0, 55.0.

**HRMS (ESI)**: m/z calculated for $[C_{18}H_{16}NO_2]^+$ $[M]^+$: 278.1176, found: 278.1175.

**2-(amino(phenyl)methyl)-5-methoxybenzoic acid hydrochloride (12)**




**Chemical Formula:** $C_{15}H_{16}ClNO_3$

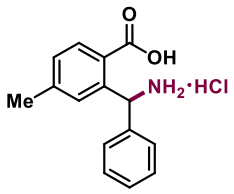
Product **12** was obtained as a white solid (41.0 mg, 70%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 8.01 (d, *J* = 8.7 Hz, 1H), 7.55 – 7.39 (m, 3H), 7.39 – 7.31 (m, 2H), 7.03 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.95 (d, *J* = 2.6 Hz, 1H), 6.44 (s, 1H), 3.83 (s, 3H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.1, 162.7, 139.4, 135.7, 134.5, 129.1, 128.9, 127.2, 121.5, 115.3, 113.5, 55.6, 54.9.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{16}NO_3]^+$ $[M]^+$: 258.1125, found: 258.1123.

**2-(amino(phenyl)methyl)-4-methylbenzoic acid hydrochloride (13)**




**Chemical Formula:** $C_{15}H_{16}ClNO_2$

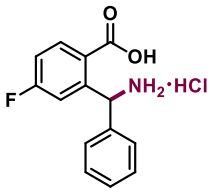
Product **13** was obtained as a white solid (37.8 mg, 68%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.90 (d, *J* = 8.0 Hz, 1H), 7.49 – 7.40 (m, 3H), 7.40 – 7.31 (m, 3H), 7.29 – 7.23 (m, 1H), 6.38 (s, 1H), 2.37 (s, 3H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.9, 144.6, 136.7, 136.0, 131.7, 129.8, 129.5, 129.3, 129.0, 128.8, 127.2, 127.0, 54.9, 20.7.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{16}NO2]^+$ $[M]^+$: 277.0864, found: 277.0859.

**2-(amino(phenyl)methyl)-4-fluorobenzoic acid hydrochloride (14)**




**Chemical Formula:** $C_{14}H_{13}ClFNO_2$

Product **14**was obtained as a white solid (37.8 mg, 67%)

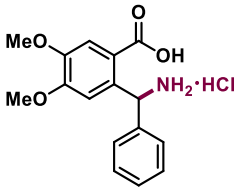
**$^1$H NMR** (400 MHz, $D_2O$) δ 8.02 (dd, *J* = 8.8, 5.9 Hz, 1H), 7.45 – 7.36 (m, 3H), 7.33 – 7.26 (m, 2H), 7.21 (td, *J* = 8.3, 2.6 Hz, 1H), 7.10 (dd, *J* = 10.0, 2.6 Hz, 1H), 6.38 (s, 1H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 169.7, 166.0, 163.4, 139.9 (d, *J* = 8.08 Hz), 135.2, 134.6 (d, *J* = 10.1 Hz), 129.1, 129.0, 127.2, 126.1 (d, *J* = 4.04 Hz), 116.3 (d, *J* = 8.08 Hz), 116.1 (d, *J* = 6.06 Hz), 54.5.

**$^{19}$F NMR** (376 MHz, $D_2O$) δ -105.0.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{13}FNO_2]^+$ $[M]^+$: 246.0925, found: 276.0924.

**2-(amino(phenyl)methyl)-4,5-dimethoxybenzoic acid hydrochloride (15)**




**Chemical Formula:** $C_{16}H_{18}ClNO_4$

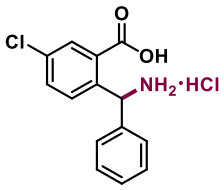
Product **15** was obtained as a white solid (58.1 mg, 90%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.48 (s, 1H), 7.40 (ddd, *J* = 13.2, 8.0, 6.3 Hz, 3H), 7.32 – 7.27 (m, 2H), 6.86 (s, 1H), 6.39 (s, 1H), 3.75 (s, 3H), 3.74 (s, 3H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 169.9, 151.7, 147.8, 136.0, 131.4, 129.1, 128.8, 126.9, 122.3, 114.4, 111.8, 55.7, 54.6.

**HRMS (ESI)**: m/z calculated for $[C_{16}H1_8NO_4]^+$ $[M]^+$: 288.1230, found: 288.1227.

**2-(amino(phenyl)methyl)-5-chlorobenzoic acid hydrochloride (16)**




**Chemical Formula:** $C_{14}H_{13}Cl_2NO_2$

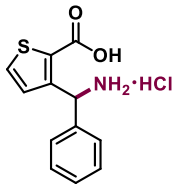
Product **16** was obtained as a white solid (36.9 mg, 62%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.99 (d, *J* = 2.3 Hz, 1H), 7.64 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.50 – 7.42 (m, 3H), 7.38 (d, *J* = 8.5 Hz, 1H), 7.35 – 7.29 (m, 2H), 6.32 (s, 1H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.1, 135.5, 134.8, 134.8, 132.9, 132.4, 131.1, 130.2, 129.1, 128.9, 127.2, 54.6.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{13}ClNO_2]^+$ $[M]^+$: 262.0629, found: 262.0628.

**3-(amino(phenyl)methyl)thiophene-2-carboxylic acid hydrochloride (17)**




**Chemical Formula:** $C_{12}H_{12}ClNO_2S$

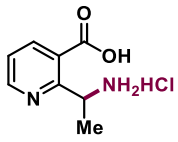
Product **17** was obtained as a white solid (36.1 mg, 67%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.70 (d, *J* = 5.2 Hz, 1H), 7.47 – 7.32 (m, 5H), 7.12 (d, *J* = 5.2 Hz, 1H), 6.32 (s, 1H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 165.1, 142.7, 135.5, 133.1, 130.2, 129.2, 129.2, 128.4, 127.1, 52.4.

**HRMS (ESI)**: m/z calculated for $[C_{12}H_{12}NO_2S]^+$ $[M]^+$: 234.0583, found: 234.0582.

**2-(1-aminoethyl)nicotinic acid hydrochloride (18)**




**Chemical Formula:** $C_8H_{11}ClN_2O_2$

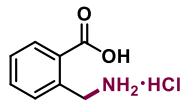
Product **18** was obtained as a white solid (36.5 mg, 90%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 8.75 (dd, *J* = 4.9, 1.7 Hz, 1H), 8.43 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.56 (dd, *J* = 8.0, 4.9 Hz, 1H), 5.45 (t, *J* = 6.7 Hz, 1H), 1.59 (d, *J* = 6.7 Hz, 3H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 168.5, 156.4, 151.8, 140.8, 124.9, 124.1, 49.0, 18.9.

**HRMS (ESI)**: m/z calculated for $[C_8H_{11}N_2O_2]^+$ $[M]^+$: 167.0815, found: 167.0815.

**2-(aminomethyl)benzoic acid hydrochloride (19)**




**Chemical Formula:** $C_8H_{10}ClNO_2$

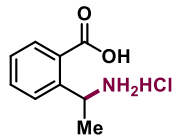
Product **19** was obtained as a white solid (25.7 mg, 68%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 8.08 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.67 (td, *J* = 7.6, 1.4 Hz, 1H), 7.58 (td, *J* = 7.7, 1.3 Hz, 1H), 7.53 (dd, *J* = 7.6, 1.3 Hz, 1H), 4.37 (s, 2H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.4, 133.6, 133.3, 132.3, 131.7, 130.0, 129.9, 42.6.

**HRMS (ESI)**: m/z calculated for $[C_8H_{10}NO_2]^+$ $[M]^+$: 152.0706, found: 152.0705.

**2-(1-aminoethyl)benzoic acid hydrochloride (20)**




**Chemical Formula:** $C_9H_{12}ClNO_2$

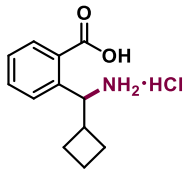
Product **20** was obtained as a white solid (37.8 mg, 94%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.97 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.67 (dtd, *J* = 15.9, 7.9, 1.5 Hz, 2H), 7.53 (td, *J* = 7.5, 1.5 Hz, 1H), 5.13 (q, *J* = 6.9 Hz, 1H), 1.66 (d, *J* = 6.9 Hz, 3H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.1, 137.5, 133.3, 131.2, 130.0, 129.3, 127.2, 47.7, 18.1.

**HRMS (ESI)**: m/z calculated for $[C_9H_{12}NO_2]^+$ $[M]^+$: 166.0863, found: 166.0862.

**2-(amino(cyclobutyl)methyl)benzoic acid hydrochloride (21)**




**Chemical Formula:** $C_{12}H_{16}ClNO_2$

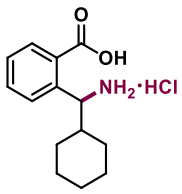
Product **21** was obtained as a white solid (42.8 mg, 89%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.95 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.65 (td, *J* = 7.6, 1.5 Hz, 1H), 7.52 (ddd, *J* = 11.1, 7.4, 2.2 Hz, 2H), 4.94 (d, *J* = 10.7 Hz, 1H), 3.16 – 3.01 (m, 1H), 2.25 (m, 1H), 2.03 – 1.85 (m, 2H), 1.79 (m, 2H), 1.63 – 1.48 (m, 1H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.3, 134.9, 133.1, 131.2, 131.1, 129.4, 128.3, 56.9, 36.6, 25.7, 24.6, 16.9.

**HRMS (ESI)**: m/z calculated for $[C_{12}H_{16}NO_2]^+$ $[M]^+$: 206.1176, found: 206.1174.

**2-(amino(cyclohexyl)methyl)benzoic acid hydrochloride (22)**



**Chemical Formula:** $C_{14}H_{20}ClNO_2$

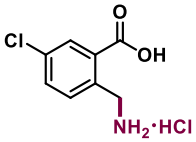
Product **22** was obtained as a white solid (48.6 mg, 90%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.94 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.69 (td, *J* = 7.7, 1.4 Hz, 1H), 7.61 – 7.51 (m, 2H), 4.74 (s, 1H), 2.08 – 1.93 (m, 2H), 1.82 (m, 1H), 1.60 (m, 2H), 1.29 (m, 1H), 1.25 – 1.06 (m, 4H), 0.87 (m, 1H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 171.8, 136.0, 132.9, 131.5, 130.8, 129.2, 40.4, 29.1, 29.0, 25.3, 25.0, 25.0.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{20}NO_2]^+$ $[M]^+$: 234.1489, found: 234.1488.

**2-(aminomethyl)-5-chlorobenzoic acid hydrochloride (23)**



**Chemical Formula:** $C_8H_9Cl_2NO_2$

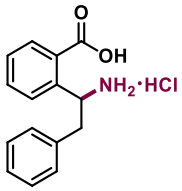
Product **23** was obtained as a white solid (32.0 mg, 72%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 8.03 (d, *J* = 2.3 Hz, 1H), 7.62 (dd, *J* = 8.2, 2.3 Hz, 1H), 7.45 (d, *J* = 8.2 Hz, 1H), 4.32 (s, 2H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 169.2, 135.2, 133.7, 133.1, 132.0, 131.8, 131.4, 41.9.

**HRMS (ESI)**: m/z calculated for $[C_8H_9ClNO_2]^+$ $[M]^+$: 186.0316, found: 186.0317.

**2-(1-amino-2-phenylethyl)benzoic acid hydrochloride (24)**



**Chemical Formula:** $C_{15}H_{16}ClNO_2$

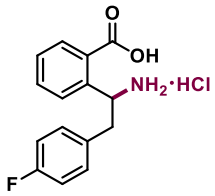
Product **24** was obtained as a white solid (48.1 mg, 87%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.85 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.70 – 7.56 (m, 2H), 7.47 (ddd, *J* = 7.7, 7.0, 1.6 Hz, 1H), 7.32 – 7.20 (m, 3H), 7.18 – 7.08 (m, 2H), 5.40 (t, *J* = 7.8 Hz, 1H), 3.28 (qd, *J* = 13.8, 7.8 Hz, 2H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 170.9, 135.9, 135.4, 133.2, 131.1, 130.2, 129.3, 129.2, 128.8, 127.6, 127.4, 53.1, 39.3.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{16}NO_2]^+$ $[M]^+$: 242.1176, found: 242.1173.

**2-(1-amino-2-(4-fluorophenyl)ethyl)benzoic acid hydrochloride (25)**




**Chemical Formula:** $C_{15}H_{15}ClFNO_2$

Product **25** was obtained as a white solid (41.6 mg, 70%)

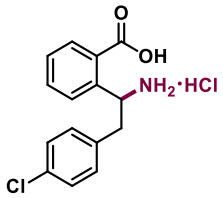
**$^1$H NMR** (400 MHz, $D_2O$) δ 7.84 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.71 – 7.53 (m, 2H), 7.48 (td, *J* = 7.5, 1.4 Hz, 1H), 7.16 – 7.04 (m, 2H), 7.03 – 6.89 (m, 2H), 5.37 (t, *J* = 7.8 Hz, 1H), 3.36 – 3.14 (m, 2H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 170.9, 161.8 (d, *J* = 244.42 Hz), 135.7, 133.2 (d, *J* = 4.04 Hz), 131.2, 131.1 (d, *J* = 3.03 Hz), 131.0, 130.4, 129.2, 127.5, 115.4 (d, *J* = 22.22 Hz), 53.1, 38.6.

**$^{19}$F NMR** (376 MHz, $D_2O$) δ -116.1.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{15}FNO_2]^+$ $[M]^+$: 260.1081, found: 260.1078.

**2-(1-amino-2-(4-chlorophenyl)ethyl)benzoic acid hydrochloride (26)**




**Chemical Formula:** $C_{15}H_{15}Cl_2NO_2$

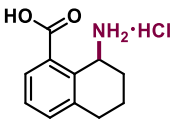
Product **26** was obtained as a white solid (43.4 mg, 70%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.74 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.59 – 7.45 (m, 2H), 7.38 (td, *J* = 7.6, 1.4 Hz, 1H), 7.18 – 7.08 (m, 2H), 7.01 – 6.89 (m, 2H), 5.34 – 5.19 (m, 1H), 3.23 (dd, *J* = 13.7, 6.9 Hz, 1H), 3.13 (dd, *J* = 13.7, 8.9 Hz, 1H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.1, 135.5, 134.0, 133.1, 132.5, 131.0, 130.8, 130.7, 129.3, 128.6, 127.7, 52.9, 38.6.

**HRMS (ESI)**: m/z calculated for $[C_{15}H_{15}ClNO_2]^+$ $[M]^+$: 276.0786, found: 276.0783.

**8-amino-5,6,7,8-tetrahydronaphthalene-1-carboxylic acid hydrochloride (27)**




**Chemical Formula:** $C_{11}H_{14}ClNO_2$

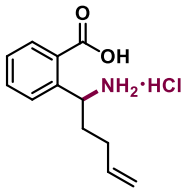
Product **27** was obtained as a white solid (44.9 mg, 99%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.83 (dd, *J* = 7.3, 1.8 Hz, 1H), 7.49 – 7.37 (m, 2H), 4.93 (t, *J* = 3.4 Hz, 1H), 2.98 (m, 1H), 2.84 (ddd, *J* = 17.8, 11.8, 6.4 Hz, 1H), 2.22 (m, 1H), 2.03 – 1.90 (m, 2H), 1.90 – 1.74 (m, 1H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.3, 139.4, 134.9, 130.8, 130.4, 129.4, 129.0, 46.4, 28.4, 26.5, 15.7.

**HRMS (ESI)**: m/z calculated for $[C_{11}H_{14}NO_2]^+$ $[M]^+$: 192.1019, found: 192.1018.

**2-(1-aminopent-4-en-1-yl)benzoic acid hydrochloride (28)**




**Chemical Formula:** $C_{12}H_{16}ClNO_2$

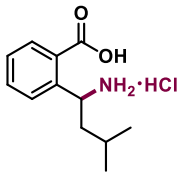
Product **28** was obtained as a white solid (43.9 mg, 91%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.96 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.70 (td, *J* = 7.6, 1.4 Hz, 1H), 7.63 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.55 (td, *J* = 7.6, 1.4 Hz, 1H), 5.17 – 5.04 (m, 1H), 2.00 (ddd, *J* = 13.8, 8.9, 6.4 Hz, 1H), 1.84 (ddd, *J* = 14.1, 7.8, 6.6 Hz, 1H), 1.52 – 1.36 (m, 1H), 0.87 (dd, *J* = 8.6, 6.6 Hz, 6H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.4, 136.3, 133.2, 131.1, 130.7, 129.4, 128.0, 50.2, 41.4, 24.3, 21.7, 21.1.

**HRMS (ESI)**: m/z calculated for $[C_{12}H_{16}NO_2]^+$ $[M]^+$: 206.1176, found: 206.1176.

**2-(1-amino-3-methylbutyl)benzoic acid hydrochloride (29)**




**Chemical Formula:** $C_{12}H_{18}ClNO_2$

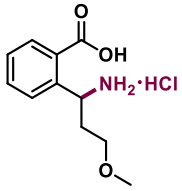
Product **29** was obtained as a white solid (45.5 mg, 93%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.94 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.67 (td, *J* = 7.6, 1.5 Hz, 1H), 7.63 – 7.58 (m, 1H), 7.53 (td, *J* = 7.5, 1.4 Hz, 1H), 5.05 (dd, *J* = 8.9, 6.6 Hz, 1H), 1.98 (m, 1H), 1.82 (m, 1H), 1.47 – 1.36 (m, 1H), 0.85 (dd, *J* = 8.5, 6.6 Hz, 6H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.6, 136.1, 133.0, 131.0, 130.9, 129.3, 127.9, 50.2, 41.3, 24.2, 21.6, 21.0.

**HRMS (ESI)**: m/z calculated for $[C_{12}H_{18}NO_2]^+$ $[M]^+$: 208.1332, found: 208.1331.

**2-(1-amino-3-methoxypropyl)benzoic acid hydrochloride (30)**



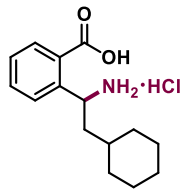

**Chemical Formula:** $C_{11}H_{16}ClNO_3$

Product **30** was obtained as a white solid (48.3 mg, 98%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.97 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.69 (td, *J* = 7.7, 1.5 Hz, 1H), 7.63 – 7.58 (m, 1H), 7.54 (td, *J* = 7.6, 1.4 Hz, 1H), 5.18 (t, *J* = 7.4 Hz, 1H), 3.48 (m, 1H), 3.32 (m, 1H),

3.23 (s, 3H), 2.42 – 2.14 (m, 2H).
**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.0, 135.8, 133.3, 131.3, 130.3, 129.5, 127.8, 68.7, 58.0, 50.2, 32.2.
**HRMS (ESI)**: m/z calculated for $[C_{11}H_{16}NO_3]^+$ $[M]^+$: 210.1125, found: 210.1124.

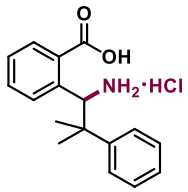
**2-(1-amino-2-cyclohexylethyl)benzoic acid hydrochloride (31)**




**Chemical Formula:** $C_{15}H_{22}ClNO_2$

Product **31** was obtained as a white solid (45.6 mg, 80%)
**$^{1}$H NMR** (600 MHz, $D_2O$) δ 7.95 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.70 (td, *J* = 7.7, 1.4 Hz, 1H), 7.66 – 7.61 (m, 1H), 7.55 (td, *J* = 7.6, 1.3 Hz, 1H), 5.12 (t, *J* = 7.8 Hz, 1H), 1.99 (ddd, *J* = 13.7, 9.3, 5.8 Hz, 1H), 1.86 (ddd, *J* = 14.0, 7.9, 6.3 Hz, 1H), 1.71 – 1.48 (m, 5H), 1.19 – 1.01 (m, 4H), 1.01 – 0.80 (m, 2H).
**$^{13}$C NMR** (151 MHz, $D_2O$) δ 171.6, 136.2, 133.1, 131.0, 131.0, 129.3, 127.9, 49.7, 40.1, 33.6, 32.7, 32.1, 25.8, 25.5, 25.4.
**HRMS (ESI)**: m/z calculated for $[C_{15}H_{22}NO_2]^+$ $[M]^+$: 248.1645, found: 248.1644.

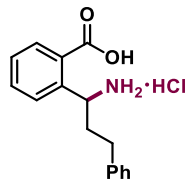
**2-(1-amino-2-methyl-2-phenylpropyl)benzoic acid hydrochloride (32)**




**Chemical Formula:** $C_{17}H_{20}ClNO_2$

Product **32** was obtained as a white solid (53.8 mg, 88%)
**$^{1}$H NMR** (600 MHz, DMSO-d6) δ 8.92 (s, 1H), 7.58 (d, *J* = 7.5 Hz, 1H), 7.43 (d, *J* = 7.4 Hz, 2H), 7.37 (q, *J* = 7.6 Hz, 3H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.28 – 7.21 (m, 1H), 6.02 (d, *J* = 7.7 Hz, 1H), 4.97 (s, 1H), 3.36 (d, *J* = 1.5 Hz, 1H), 1.48 (s, 3H), 0.92 (s, 3H).
**$^{13}$C NMR** (151 MHz, DMSO-d6) δ 169.8, 146.0, 145.2, 133.2, 130.6, 128.2, 127.9, 126.6, 126.6, 123.3, 122.5, 64.8, 41.5, 28.9, 19.7.
**HRMS (ESI)**: m/z calculated for $[C_{17}H_{20}NO_2]^+$ $[M]^+$: 270.1489, found: 270.1486.

**2-(1-amino-3-phenylpropyl)benzoic acid hydrochloride (33)**




**Chemical Formula:** $C_{16}H_{18}ClNO_2$

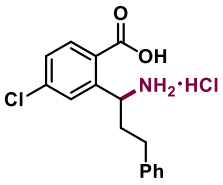
Product **33** was obtained as a white solid (52.7 mg, 91%)

**$^1$H NMR** (400 MHz, $D_2O$) δ 7.92 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.65 (td, *J* = 7.6, 1.4 Hz, 1H), 7.58 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.50 (td, *J* = 7.6, 1.3 Hz, 1H), 7.26 – 7.09 (m, 3H), 7.03 – 6.91 (m, 2H), 4.87 (dd, *J* = 8.7, 5.9 Hz, 1H), 2.52 – 2.21 (m, 4H).

**$^{13}$C NMR** (101 MHz, $D_2O$) δ 170.8, 140.0, 135.4, 133.4, 131.3, 130.8, 129.5, 128.6, 128.3, 128.1, 126.4, 51.1, 33.4, 31.1.

**HRMS (ESI)**: m/z calculated for $[C_{16}H_{18}NO_2]^+$ $[M]^+$: 256.1332, found: 256.1329.

**2-(1-amino-3-phenylpropyl)-4-chlorobenzoic acid hydrochloride (34)**




**Chemical Formula:** $C_{16}H_{17}Cl_2NO_2$

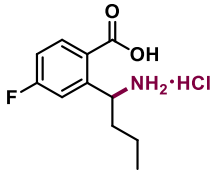
Product **34** was obtained as a white solid (47.0 mg, 72%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.91 (d, *J* = 2.3 Hz, 1H), 7.62 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.53 (d, *J* = 8.5 Hz, 1H), 7.24 – 7.11 (m, 3H), 7.02 – 6.95 (m, 2H), 4.83 (dd, *J* = 9.9, 5.4 Hz, 1H), 2.55 (ddd, *J* = 13.5, 8.3, 5.2 Hz, 1H), 2.46 – 2.39 (m, 1H), 2.39 – 2.33 (m, 1H), 2.33 – 2.24 (m, 1H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 169.4, 140.0, 134.9, 134.0, 133.1, 132.5, 131.1, 129.9, 128.7, 128.4, 126.4, 50.4, 33.1, 30.9.

**HRMS (ESI)**: m/z calculated for $[C_{16}H_{17}ClNO_2]^+$ $[M]^+$: 290.0942, found: 290.0939.

**2-(1-aminobutyl)-4-fluorobenzoic acid hydrochloride (35)**




**Chemical Formula:** $C_{11}H_{15}ClFNO_2$

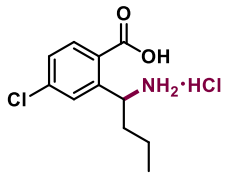
Product **35** was obtained as a white solid (41.4 mg, 85%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 8.05 (dd, *J* = 8.8, 5.9 Hz, 1H), 7.38 (dd, *J* = 10.0, 2.6 Hz, 1H), 7.27 (ddd, *J* = 8.7, 8.0, 2.6 Hz, 1H), 5.07 (t, *J* = 7.6 Hz, 1H), 2.12 – 1.91 (m, 2H), 1.45 – 1.09 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.3, 165.7, 164.1, 139.7 (d, *J* = 9.06 Hz), 134.2 (d, *J* = 9.06 Hz), 127.0 (d, *J* = 3.02 Hz), 116.2 (d, *J* = 21.14 Hz), 115.2 (d, *J* = 24.16 Hz), 51.3, 34.3, 18.5, 12.7.

**$^{19}$F NMR** (565 MHz, $D_2O$) δ -105.5.
**HRMS (ESI)**: m/z calculated for $[C_{11}H_{15}FNO_2]^+$ $[M]^+$: 212.1081, found: 212.1080.

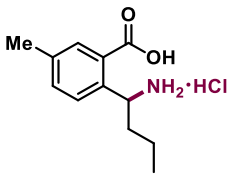
**2-(1-aminobutyl)-4-chlorobenzoic acid hydrochloride (36)**




**Chemical Formula:** $C_{11}H_{15}Cl_2NO_2$

Product**36** was obtained as a white solid (46.2 mg, 87%)
**$^1$H NMR** (600 MHz, $D_2O$) δ 7.94 (d, *J* = 8.4 Hz, 1H), 7.65 (d, *J* = 2.1 Hz, 1H), 7.56 (dd, *J* = 8.4, 2.1 Hz, 1H), 5.00 (t, *J* = 7.6 Hz, 1H), 2.14 – 1.88 (m, 2H), 1.42 – 1.16 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).
**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.7, 138.5, 138.1, 132.7, 129.9, 129.4, 128.1, 51.5, 34.3, 18.5, 12.7.
**HRMS (ESI)**: m/z calculated for $[C_{11}H_{15}ClNO_2]^+$ $[M]^+$: 228.0786, found: 228.0784.

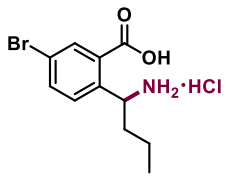
**2-(1-aminobutyl)-5-methylbenzoic acid hydrochloride (37)**




**Chemical Formula:** $C_{12}H_{18}ClNO_2$

Product **37** was obtained as a white solid (32.7 mg, 67%)
**$^1$H NMR** (400 MHz, $D_2O$) δ 7.72 (d, *J* = 1.7 Hz, 1H), 7.47 – 7.37 (m, 2H), 4.86 (dd, *J* = 8.6, 6.8 Hz, 1H), 2.30 (s, 3H), 2.01 – 1.83 (m, 2H), 1.28 – 1.06 (m, 2H), 0.80 (td, *J* = 7.4, 3.6 Hz, 3H).
**$^{13}$C NMR** (101 MHz, $D_2O$) δ 171.6, 139.8, 133.5, 133.0, 131.4, 130.7, 127.7, 51.4, 34.4, 19.9, 18.5, 12.6.
**HRMS (ESI)**: m/z calculated for $[C_{12}H_{18}NO_2]^+$ $[M]^+$: 208.1332, found: 208.1330.

**2-(1-aminobutyl)-5-bromobenzoic acid hydrochloride (38)**




**Chemical Formula:** $C_{11}H_{15}BrClNO_2$

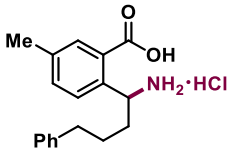
Product **38** was obtained as a white solid (45.9 mg, 74%)
**$^1$H NMR** (600 MHz, $D_2O$) δ 8.10 (d, *J* = 2.2 Hz, 1H), 7.82 (dd, *J* = 8.4, 2.2 Hz, 1H), 7.50 (d, *J* =

8.5 Hz, 1H), 4.97 (t, $J$ = 7.6 Hz, 1H), 2.11 – 1.89 (m, 2H), 1.35 – 1.15 (m, 2H), 0.88 (t, $J$ = 7.4 Hz, 3H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 170.0, 135.7, 135.3, 133.8, 133.0, 129.6, 122.6, 51.3, 34.4, 18.6, 12.7.

**HRMS (ESI)**: m/z calculated for $[C_{11}H_{15}BrNO_2]^+$ $[M]^+$: 272.0281, found: 272.0277.

**2-(1-amino-4-phenylbutyl)-5-methylbenzoic acid hydrochloride (39)**




**Chemical Formula:** $C_{18}H_{22}ClNO_2$

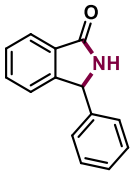
Product **39** was obtained as a white solid (49.0 mg, 77%)

**$^1$H NMR** (600 MHz, $D_2O$) δ 7.63 (d, $J$ = 1.9 Hz, 1H), 7.31 (d, $J$ = 8.0 Hz, 1H), 7.24 – 7.20 (m, 1H), 6.94 (ddd, $J$ = 8.6, 6.9, 2.1 Hz, 2H), 6.88 – 6.80 (m, 3H), 4.87 (t, $J$ = 6.9 Hz, 1H), 2.42 (ddd, $J$ = 14.5, 8.9, 6.0 Hz, 1H), 2.36 – 2.29 (m, 1H), 2.08 (d, $J$ = 1.9 Hz, 3H), 2.00 (p, $J$ = 7.4 Hz, 3H), 1.43 – 1.34 (m, 1H), 1.26 – 1.18 (m, 1H).

**$^{13}$C NMR** (151 MHz, $D_2O$) δ 171.0, 141.6, 139.6, 139.5, 133.6, 132.9, 131.9, 130.6, 128.2, 128.2, 125.7, 51.7, 34.4, 31.6, 27.1, 20.1.

**HRMS (ESI)**: m/z calculated for $[C_{18}H_{22}NO_2]^+$ $[M]^+$: 284.1645, found: 284.1643.

**3-phenylisoindolin-1-one (40)**




**Chemical Formula:** $C_{14}H_{11}NO$

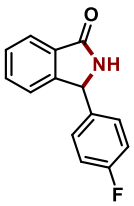
Product **40** was obtained as a white solid (29.7 mg, 71%)

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.99 – 7.78 (m, 1H), 7.55 – 7.43 (m, 2H), 7.40 – 7.20 (m, 6H), 6.77 (s, 1H), 5.63 (s, 1H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 171.0, 148.0, 138.4, 132.3, 130.8, 129.1, 128.6, 128.4, 126.8, 123.9, 123.3, 60.8.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{12}NO]^+$ $[M + H]^+$: 210.0913, found: 210.0913.

**3-(4-fluorophenyl)isoindolin-1-one (41)**

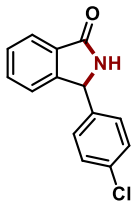



**Chemical Formula:** $C_{14}H_{10}FNO$

Product **41** was obtained as a white solid (34.6 mg, 76%)

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.6 Hz, 2H), 7.60 – 7.38 (m, 2H), 7.32 – 7.16 (m, 3H), 7.02 (t, *J* = 8.6 Hz, 2H), 5.62 (s, 1H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 171.5, 163.9, 161.5, 147.9, 134.2 (d, *J* = 4.04 Hz), 132.4, 130.9, 128.6, 128.5 (d, *J* = 5.05 Hz), 123.5 (d, *J* = 55.55 Hz), 116.0 (d, *J* = 22.22 Hz), 60.2.
**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -113.4.
**HRMS (ESI)**: m/z calculated for $[C_{14}H_{11}FNO]^+$ $[M + H]^+$: 250.0639, found: 250.0639.

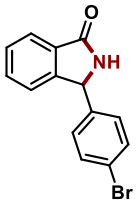
**3-(4-chlorophenyl)isoindolin-1-one (42)**




**Chemical Formula:** $C_{14}H_{10}ClNO$

Product **42** was obtained as a white solid (40.8 mg, 84%)
**$^1$H NMR** (400 MHz, DMSO-d6) δ 9.11 (s, 1H), 7.72 (d, *J* = 7.4 Hz, 1H), 7.57 – 7.45 (m, 2H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.31 (dd, *J* = 11.2, 7.8 Hz, 3H), 5.77 (s, 1H).
$^{13}$C NMR (101 MHz, DMSO-d6) δ 169.7, 147.8, 138.7, 132.5, 132.0, 131.3, 128.8, 128.5, 128.3, 123.5, 123.0, 58.8.
**HRMS (ESI)**: m/z calculated for $[C_{14}H_{11}ClNO]^+$ $[M + H]^+$: 244.0523, found: 244.0532.

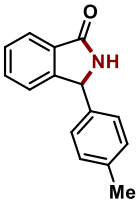
**3-(4-bromophenyl)isoindolin-1-one (43)**




**Chemical Formula:** $C_{14}H_{10}BrNO$

Product **43** was obtained as a white solid (52.1 mg, 89%)
**$^1$H NMR** (600 MHz, DMSO-d6) δ 9.10 (s, 1H), 7.71 (d, *J* = 7.5 Hz, 1H), 7.63 – 7.43 (m, 4H), 7.27 (dd, *J* = 21.0, 7.8 Hz, 3H), 5.75 (s, 1H).
**$^{13}$C NMR** (151 MHz, DMSO-d6) δ 169.7, 147.7, 139.2, 132.0, 131.7, 131.3, 128.8, 128.3, 123.5, 123.0, 121.0, 58.8.
**HRMS (ESI)**: m/z calculated for $[C_{14}H_{11}BrNO]^+$ $[M + H]^+$: 288.0019, found: 288.0023.

**3-(p-tolyl)isoindolin-1-one (44)**

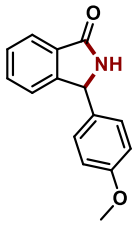



**Chemical Formula:** $C_{15}H_{13}NO$

Product **44** was obtained as a white solid (30.0 mg, 67%)
**$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.4 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.32 (s, 1H), 7.23 (d,

$J$ = 7.5 Hz, 1H), 7.15 (d, $J$ = 1.8 Hz, 4H), 5.60 (s, 1H), 2.33 (s, 3H).
**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 171.4, 148.2, 138.4, 135.3, 132.3, 130.9, 129.7, 128.3, 126.8, 123.8, 123.3, 60.7, 21.2.
**HRMS (ESI)**: m/z calculated for $[C_{15}H_{14}NO]^+$ $[M + H]^+$: 224.1070, found: 224.1073.

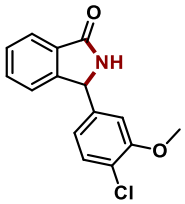
**3-(4-methoxyphenyl)isoindolin-1-one (45)**




**Chemical Formula:** $C_{15}H_{13}NO_2$

Product **45** was obtained as a white solid (28.5 mg, 60%)
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.92 – 7.82 (m, 1H), 7.55 – 7.41 (m, 2H), 7.24 – 7.12 (m, 3H), 6.92 – 6.80 (m, 3H), 5.58 (s, 1H), 3.79 (s, 3H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 171.0, 159.8, 148.3, 132.3, 130.9, 130.2, 128.3, 128.1, 123.7, 123.3, 114.4, 60.3, 55.4.
**HRMS (ESI)**: m/z calculated for $[C_{15}H_{14}NO]^+$ $[M + H]^+$: 240.1019, found: 240.1023.

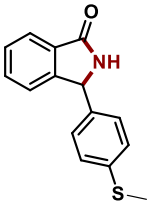
**3-(4-chloro-3-methoxyphenyl)isoindolin-1-one (46)**




**Chemical Formula:** $C_{15}H_{12}ClNO_2$

Product **46** was obtained as a white solid (45.6 mg, 83%)
**$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.88 (d, $J$ = 7.5 Hz, 1H), 7.51 (dt, $J$ = 23.4, 7.5 Hz, 2H), 7.35 (dd, $J$ = 8.1, 1.7 Hz, 1H), 7.23 (d, $J$ = 7.5 Hz, 1H), 7.04 (s, 1H), 6.88 (d, $J$ = 8.1 Hz, 1H), 6.75 (s, 1H), 5.60 (s, 1H), 3.82 (d, $J$ = 1.7 Hz, 3H).
**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 171.2, 155.6, 147.5, 138.5, 132.5, 130.7, 130.6, 128.7, 124.0, 123.2, 122.7, 119.8, 109.8, 60.5, 56.2.
**HRMS (ESI)**: m/z calculated for $[C_{15}H_{13}ClNO_2]^+$ $[M + H]^+$: 274.0629, found: 274.0637.

**3-(4-(methylthio)phenyl)isoindolin-1-one (47)**

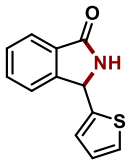



**Chemical Formula:** $C_{15}H_{13}NOS$

Product **47** was obtained as a white solid (32.4 mg, 63%)
**$^1$H NMR** (400 MHz, DMSO-d6) δ 9.07 (s, 1H), 7.70 (dd, $J$ = 7.3, 1.3 Hz, 1H), 7.57 – 7.44 (m,

2H), 7.30 – 7.18 (m, 5H), 5.70 (s, 1H), 2.45 (s, 3H).
**$^{13}$C NMR** (101 MHz, DMSO-d6) δ 169.7, 148.2, 137.8, 136.2, 131.9, 131.4, 128.2, 127.2, 126.2, 123.5, 122.9, 59.1, 14.7.
**HRMS (ESI)**: m/z calculated for $[C_{15}H_{14}NOS]^+$ $[M + H]^+$: 256.0791, found: 256.0798.

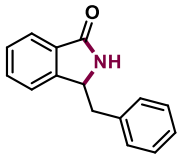
**3-(thiophen-2-yl)isoindolin-1-one (48)**




**Chemical Formula:** $C_{12}H_9NOS$

Product **48** was obtained as a white solid (20.5 mg, 47%)
**$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.89 (dt, *J* = 7.3, 1.0 Hz, 1H), 7.61 – 7.47 (m, 2H), 7.38 (dq, *J* = 7.6, 1.0 Hz, 1H), 7.31 – 7.24 (m, 1H), 7.14 (dt, *J* = 3.5, 0.9 Hz, 1H), 7.00 (dd, *J* = 5.1, 3.5 Hz, 1H), 6.91 (s, 1H), 5.95 (s, 1H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 170.4, 147.1, 141.8, 132.5, 130.6, 128.8, 127.1, 125.9, 125.8, 123.9, 123.4, 56.2.
**HRMS (ESI)**: m/z calculated for $[C_{12}H_{10}NOS]^+$ $[M + H]^+$: 216.0478, found: 216.0486.

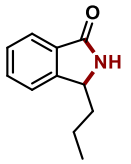
**3-benzylisoindolin-1-one (49)**




**Chemical Formula:** $C_{15}H_{13}NO$

Product **49** was obtained as a white solid (34.4 mg, 77%)
**$^{1}$H NMR** (600 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.5 Hz, 1H), 7.58 – 7.51 (m, 1H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.37 – 7.30 (m, 3H), 7.30 – 7.25 (m, 1H), 7.25 – 7.21 (m, 2H), 6.79 (s, 1H), 4.81 (dd, *J* = 9.0, 5.4 Hz, 1H), 3.22 (dd, *J* = 13.6, 5.3 Hz, 1H), 2.83 (dd, *J* = 13.6, 8.9 Hz, 1H).
**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 170.4, 146.8, 136.9, 131.9, 131.7, 129.2, 128.8, 128.3, 127.1, 123.9, 122.7, 58.0, 41.3.
**HRMS (ESI)**: m/z calculated for $[C_{15}H_{14}NO]^+$ $[M + H]^+$: 224.1070, found: 224.1070.

**3-propylisoindolin-1-one (50)**

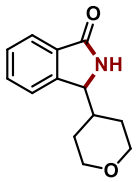



**Chemical Formula:** $C_{11}H_{13}NO$

Product **50** was obtained as a white solid (19.6 mg, 56%)
**$^{1}$H NMR** (600 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.49 – 7.40 (m, 2H), 6.91 (s, 1H), 4.62 (dd, *J* = 8.2, 4.3 Hz, 1H), 1.93 (m, 1H), 1.62 (m, 1H), 1.56 – 1.43 (m, 1H), 1.39 (m, 1H), 0.97 (m, 3H).
**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 147.8, 131.8, 128.1, 123.8, 122.4, 56.8, 36.8, 19.1, 14.1.

**HRMS (ESI)**: m/z calculated for $[C_{11}H_{14}NO]^+$ $[M + H]^+$: 176.1070, found: 176.1075.

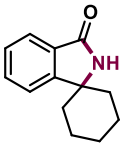
**3-(tetrahydro-2H-pyran-4-yl)isoindolin-1-one (51)**



**Chemical Formula:** $C_{13}H_{15}NO_2$

Product **51** was obtained as a white solid (32.6 mg, 75%)
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.5 Hz, 1H), 7.57 (td, *J* = 7.5, 1.2 Hz, 1H), 7.53 – 7.38 (m, 3H), 4.55 (d, *J* = 4.1 Hz, 1H), 4.11 – 3.87 (m, 2H), 3.37 (m, 2H), 2.10 (m, 1H), 1.83 – 1.70 (m, 1H), 1.66 – 1.40 (m, 2H), 1.18 (m, 1H).
**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 171.3, 145.6, 132.5, 131.9, 128.4, 124.0, 122.9, 67.8, 67.7, 61.0, 39.4, 29.9, 26.8.
**HRMS (ESI)**: m/z calculated for $[C_{13}H_{16}NO_2]^+$ $[M + H]^+$: 218.1176, found: 218.1183.

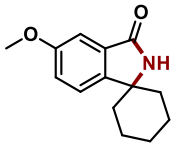
**spiro[cyclohexane-1,1'-isoindolin]-3'-one (52)**



**Chemical Formula:** $C_{13}H_{15}NO$

Product **52** was obtained as a white solid (25.2 mg, 63%)
**$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.83 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.63 (s, 1H), 7.55 (td, *J* = 7.5, 1.2 Hz, 1H), 7.48 – 7.36 (m, 2H), 1.93 – 1.81 (m, 6H), 1.62 – 1.54 (m, 4H), 1.48 – 1.35 (m, 1H).
**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 170.1, 153.0, 131.9, 130.9, 128.1, 124.0, 121.3, 61.7, 36.7, 25.0, 23.4.
**HRMS (ESI)**: m/z calculated for $[C_{13}H_{16}NO]^+$ $[M + H]^+$: 202.1226, found: 202.1226.

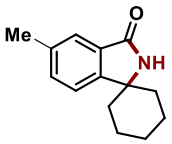
**5'-methoxyspiro[cyclohexane-1,1'-isoindolin]-3'-one (53)**



**Chemical Formula:** $C_{14}H_{17}NO_2$

Product **53** was obtained as a white solid (36.1 mg, 78%)
**$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.61 (s, 1H), 7.33 – 7.27 (m, 2H), 7.10 (dd, *J* = 8.4, 2.5 Hz, 1H), 3.85 (s, 3H), 1.92 – 1.78 (m, 5H), 1.62 – 1.50 (m, 4H), 1.40 (m, 1H).
**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 170.0, 159.9, 145.5, 132.1, 122.2, 120.2, 106.5, 61.4, 55.7, 36.8, 25.0, 23.5.
**HRMS (ESI)**: m/z calculated for $[C_{14}H_{18}NO_2]^+$ $[M + H]^+$: 232.1332, found: 232.1336.

**5'-methylspiro[cyclohexane-1,1'-isoindolin]-3'-one (54)**




**Chemical Formula:** $C_{14}H_{17}NO$

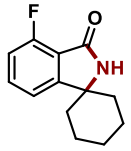
Product **54** was obtained as a white solid (31.8 mg, 74%)

**$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 9.1 Hz, 2H), 7.36 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.29 (d, *J* = 7.7 Hz, 1H), 2.43 (s, 3H), 1.91 – 1.80 (m, 5H), 1.62 – 1.51 (m, 4H), 1.46 – 1.35 (m, 1H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 170.2, 150.4, 138.0, 132.8, 131.0, 124.2, 121.1, 61.5, 36.7, 25.0, 23.4, 21.3.

**HRMS (ESI)**: m/z calculated for $[C_{14}H_{18}NO]^+$ $[M + H]^+$: 216.1383, found: 216.1389.

**4'-fluorospiro[cyclohexane-1,1'-isoindolin]-3'-one (55)**




**Chemical Formula:** $C_{13}H_{14}FNO$

Product **55** was obtained as a white solid (30.1 mg, 69%)

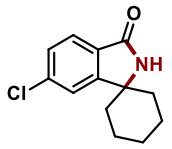
**$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.51 (td, *J* = 7.8, 4.2 Hz, 1H), 7.40 (s, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 8.8 Hz, 1H), 1.93 – 1.81 (m, 5H), 1.65 – 1.47 (m, 4H), 1.46 – 1.33 (m, 1H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 166.9, 160.0, 158.3, 155.9 (d, *J* = 3.02 Hz), 134.0 (d, *J* = 7.55 Hz), 118.0 (d, *J* = 13.59 Hz), 117.3 (d, *J* = 4.53 Hz), 115.3 (d, *J* = 10.57 Hz), 61.6, 36.7, 24.9, 23.3.

**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -114.2.

**HRMS (ESI)**: m/z calculated for $[C_{13}H_{15}FNO]^+$ $[M + H]^+$: 220.1132, found: 220.1142.

**6'-chlorospiro[cyclohexane-1,1'-isoindolin]-3'-one (56)**




**Chemical Formula:** $C_{13}H_{14}ClNO$

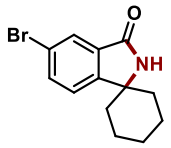
Product **56** was obtained as a white solid (31.3 mg, 66%)

**$^1$H NMR** (600 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.50 – 7.32 (m, 2H), 2.01 – 1.76 (m, 5H), 1.72 – 1.51 (m, 4H), 1.40 (m, 1H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 169.1, 154.6, 138.2, 129.5, 128.6, 125.3, 122.0, 61.7, 36.6, 24.9, 23.2.

**HRMS (ESI)**: m/z calculated for $[C_{13}H_{15}ClNO]^+$ $[M + H]^+$: 236.0837, found: 236.0842.

**5'-bromospiro[cyclohexane-1,1'-isoindolin]-3'-one (57)**




**Chemical Formula:** $C_{13}H_{14}BrNO$

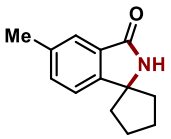
Product **57** was obtained as a white solid (36.2 mg, 65%)

**$^1$H NMR** (600 MHz, $CDCl_3$) δ 8.34 (s, 1H), 7.96 (d, *J* = 2.1 Hz, 1H), 7.65 (dd, *J* = 8.0, 2.1 Hz, 1H), 7.29 (dd, *J* = 8.1, 1.9 Hz, 1H), 1.86 m, 5H), 1.61 (m, 4H), 1.45 – 1.35 (m, 1H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 168.7, 151.7, 134.8, 133.1, 127.1, 123.0, 121.9, 62.0, 36.6, 25.0, 23.2.

**HRMS (ESI)**: m/z calculated for $[C_{13}H_{15}BrNO]^+$ $[M + H]^+$: 280.0332, found: 280.0339.

**5'-methylspiro[cyclopentane-1,1'-isoindolin]-3'-one (58)**




**Chemical Formula:** $C_{13}H_{15}NO$

Product **58** was obtained as a white solid (35.0 mg, 87%)

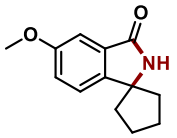
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.62 (s, 1H), 7.38 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.31 – 7.25 (m, 1H), 7.08 (s, 1H), 2.44 (s, 3H), 2.15 – 1.87 (m, 8H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 148.8, 137.9, 133.1, 131.4, 123.9, 120.9, 69.2, 39.2, 24.8, 21.3.

**HRMS (ESI, m/z)**: $[M+H]^+$ Calcd for ; Found.]

**HRMS (ESI)**: m/z calculated for $[C_{13}H_{16}NO]^+$ $[M + H]^+$: 202.1226, found: 202.1233.

**5'-methoxyspiro[cyclopentane-1,1'-isoindolin]-3'-one (59)**




**Chemical Formula:** $C_{13}H_{15}NO_2$

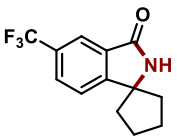
Product **59** was obtained as a white solid (35.3 mg, 81%)

**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.48 (s, 1H), 7.31 – 7.26 (m, 2H), 7.12 (dd, *J* = 8.4, 2.5 Hz, 1H), 3.86 (s, 3H), 2.13 – 2.04 (m, 2H), 2.04 – 1.88 (m, 6H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 169.9, 159.9, 143.7, 132.6, 122.1, 120.5, 106.2, 69.1, 55.7, 38.9, 24.7.

**HRMS (ESI)**: m/z calculated for $[C_{13}H_{16}NO_2]^+$ $[M + H]^+$: 218.1176, found: 218.1186.

**5'-(trifluoromethyl)spiro[cyclopentane-1,1'-isoindolin]-3'-one (60)**




**Chemical Formula:** $C_{13}H_{12}F_3NO$

Product **60** was obtained as a white solid (30.7 mg, 60%)

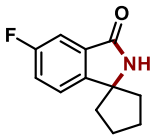
**$^1$H NMR** (600 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.98 (s, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 2.21 – 1.90 (m, 8H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 168.6, 154.9, 132.2, 130.8 (q, *J* = 33.22 Hz), 129.0 (q, *J* = 3.02 Hz), 123.9 (q, *J* = 273.31 Hz), 121.9, 121.1 (q, *J* = 4.53 Hz), 69.7, 39.2, 24.9.

**$^{19}$F NMR** (565 MHz, $CDCl_3$) δ -62.2.

**HRMS (ESI)**: m/z calculated for $[C_{13}H_{13}F_3NO]^+$ $[M + H]^+$: 256.0944, found: 256.0954.

**5'-fluorospiro[cyclopentane-1,1'-isoindolin]-3'-one (61)**




**Chemical Formula:** $C_{12}H_{12}FNO$

Product **61** was obtained as a white solid (31.3 mg, 76%)

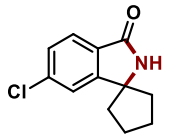
**$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.61 (s, 1H), 7.47 (dd, *J* = 7.7, 2.4 Hz, 1H), 7.36 (dd, *J* = 8.4, 4.4 Hz, 1H), 7.31 – 7.22 (m, 1H), 2.17 – 1.88 (m, 8H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 168.7, 162.7 (d, *J* = 247.45), 146.9 (d, *J* = 2.02 Hz), 133.4 (d, *J* = 8.08 Hz), 122.7 (d, *J* = 8.08 Hz), 119.5 (d, *J* = 23.23 Hz), 110.3 (d, *J* = 23.23 Hz), 69.3, 39.1, 24.7.

**$^{19}$F NMR** (376 MHz, $CDCl_3$) δ -113.9.

**HRMS (ESI)**: m/z calculated for $[C_{12}H_{13}FNO]^+$ $[M + H]^+$: 206.0976, found: 206.0983.

**6'-chlorospiro[cyclopentane-1,1'-isoindolin]-3'-one (62)**




**Chemical Formula:** $C_{12}H_{12}ClNO$

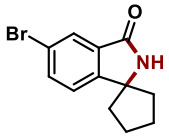
Product **62** was obtained as a white solid (34.2 mg, 77%)

**$^1$H NMR** (600 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.72 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.43 – 7.36 (m, 2H), 2.08 (m, 2H), 2.04 – 1.94 (m, 6H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 169.1, 153.3, 138.4, 129.9, 128.4, 124.9, 121.8, 69.3, 39.1, 24.8.

**HRMS (ESI)**: m/z calculated for $[C_{12}H_{13}ClNO]^+$ $[M + H]^+$: 222.0680, found: 250. 222.0690.

**5'-bromospiro[cyclopentane-1,1'-isoindolin]-3'-one (63)**



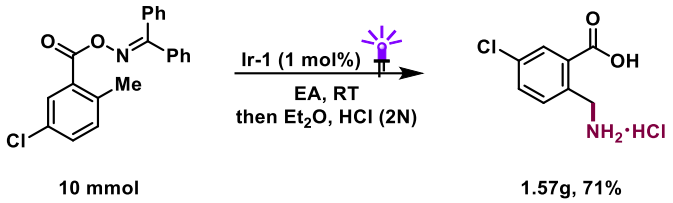

**Chemical Formula:** $C_{12}H_{12}BrNO$

Product **63** was obtained as a white solid (38.5 mg, 72%)

**$^1$H NMR** (600 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.96 – 7.90 (m, 1H), 7.69 – 7.63 (m, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 2.01 (m, 8H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 168.6, 150.2, 135.0, 133.5, 126.8, 122.9, 121.8, 69.5, 39.0, 24.8.
**HRMS (ESI)**: m/z calculated for $[C_{12}H_{13}BrNO]^+$ $[M + H]^+$: 266.0175, found: 266.0182.

## 2.7 Gram scale of S23

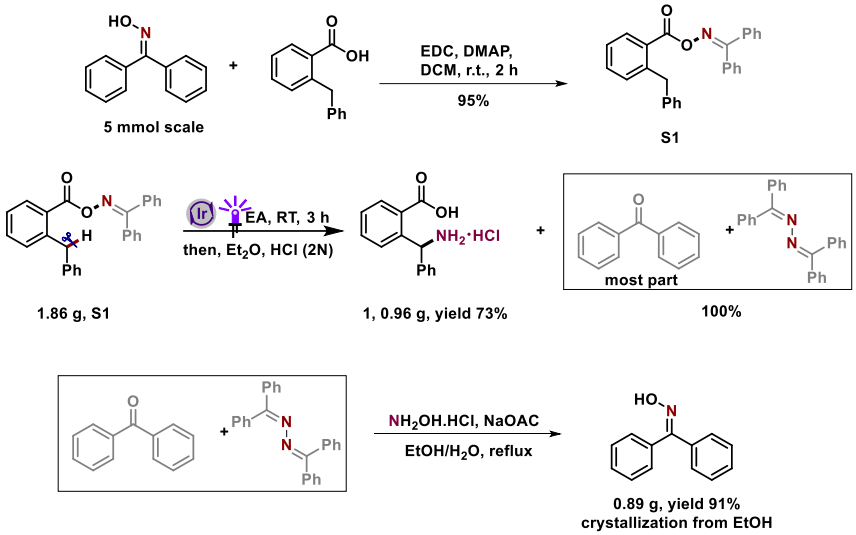



10 mmol 1.57g, 71%

To an oven-dried glass flask (250 mL) equipped with a magnetic stir bar were added the oxime esters **S23** (10 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), and EA (0.05 M). Then the vessel was bubbled with a stream of argon for 30 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 12 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion of the reaction, the solvents were removed under reduced pressure, acidified by 20 mL HCl (2 N) and stirred in $Et_2O$ (30 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (20 mL) and extracted with $Et_2O$ (30 mL x 3). The water layer was combined, and water was removed *in vacuo*, and the product was obtained as a white solid **23** (1.57g, 71%).

## 2.8 Recycling of the benzophenone

To verify the recovery of benzophenone byproducts, we also conducted a large-scale experiment on the template substrate **S1** (5 mmol) under the standard procedure.




To a solution of benzophenone oxime (5.0 mmol) and 2-benzylbenzoic acid (5.0 mmol) in $CH_2Cl_2$ (30 mL), DMAP (10 mol%) and EDCI•HCl (12.5 mmol) was added. The mixture was stirred at room temperature under inert atmosphere until the reaction was complete as observed from TLC monitoring. The mixture was diluted with distilled water (25 mL) and the $CH_2Cl_2$ layer was separated, dried over anhydrous $Na_2SO_4$ and concentrated. The crude mass was treated with pentane (3 mL) and sonicated for 15 minutes. The resultant solid was filtered and dried under vacuum to obtain the diphenylmethanone *O*-(2-benzylbenzoyl) oxime (**S1**).

To an oven-dried glass flask (250 mL) equipped with a magnetic stir bar were added the oxime esters **S1** (1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), and EA (0.05 M). Then the flask was bubbled with a stream of argon for 30 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 12 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion of the reaction, the solvents were removed under reduced pressure, acidified by 10 mL HCl (2 N) and stirred in $Et_2O$ (30 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (20 mL) and extracted with $Et_2O$ (30 mL x 3). The water layer was combined, and water was removed *in vacuo*, and the product **1** was obtained as a white solid **1** (0.96 g, 73%). And the organic phase was concentrated *in vacuo*, and the mixture of imine self-coupling byproducts and benzophenone was obtained as a yellow solid.

And then, add hydroxylamine hydrochloride (1.5 equiv.), sodium acetate (2 equiv.) and EtOH/$H_2O$ (4/1; 50 mL) to this yellow mixture, and the mixture was stirred at 80 °C overnight. The mixture

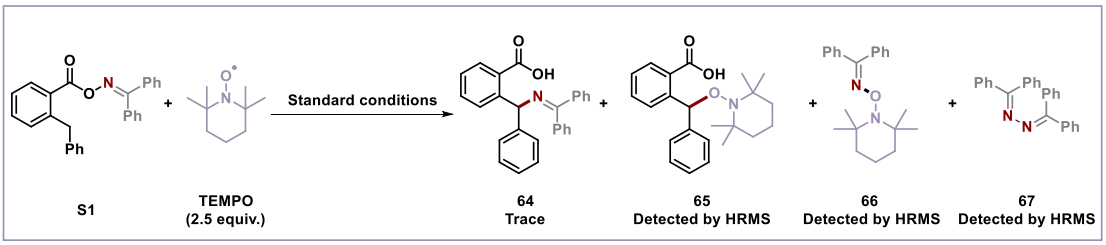
was cooled down to r.t. and EtOH was removed under reduced pressure. $H_2O$ (50 mL) was added, and the resulting mixture was extracted with EtOAc (3x 20 mL). The combined organic layers were washed with $H_2O$ (2x 50 mL) and brine (50 mL) and were dried over $Na_2SO_4$. the organic phase was concentrated *in vacuo*, and the product was obtained as a white solid. Further crystallization from EtOH gave pure benzophenone oxime as a white solid (0.89 g, 91 %).

# 3 Supplementary Notes

## 3.1 Mechanistic Investigations

### 3.1.1 Trapping Experiments

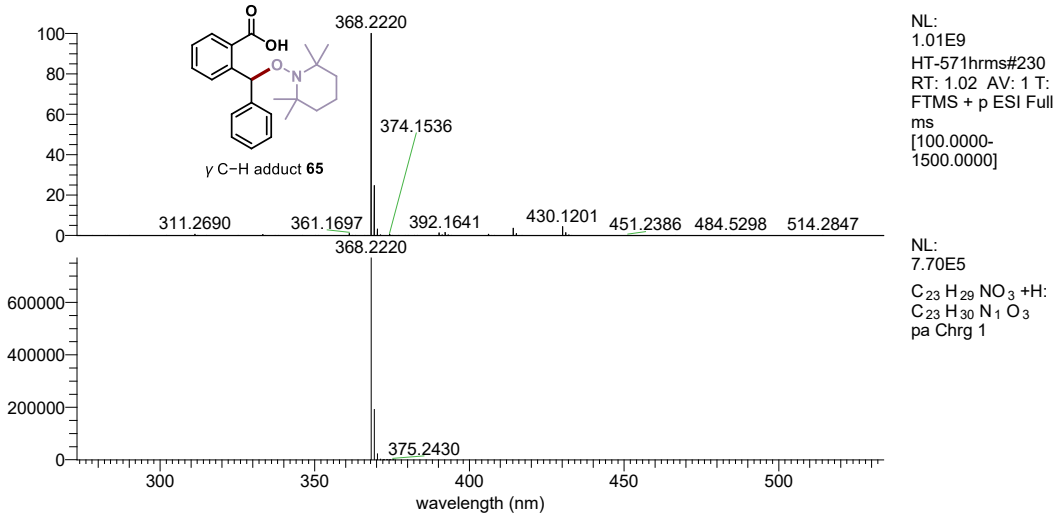
To investigate the intermediacy of radicals which are involved in the presented methodology, a trapping experiment for the intramolecular C−H amination using 2,2,6,6-tetramethylpiperidine-N-oxyl (TEMPO) in (over-)stoichiometric quantities was performed.



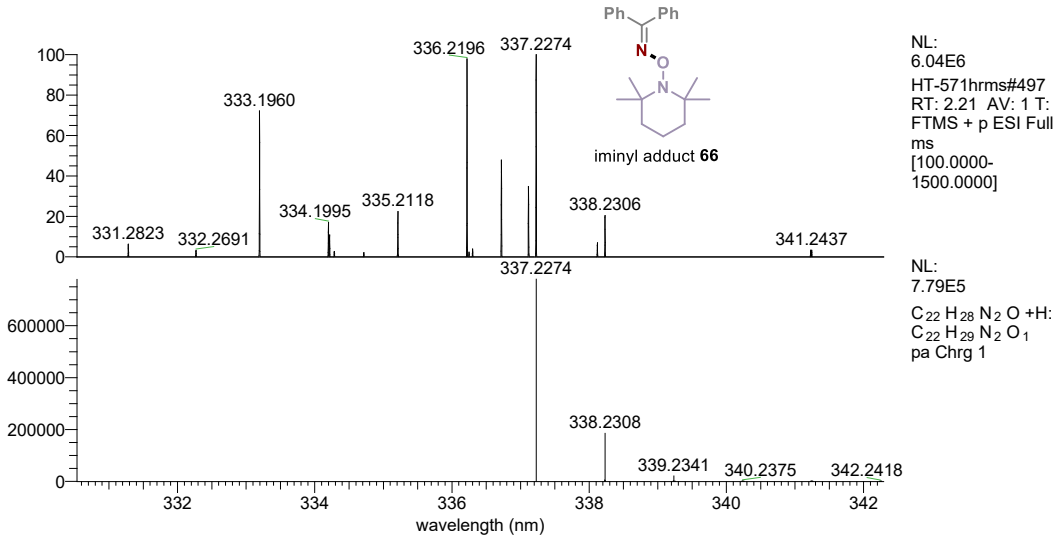

**Supplementary Figure 2. TEMPO-trapping experiment for γ C−H amination.**

To an oven-dried glass tube (10 mL) equipped with a magnetic stir bar were added the oxime esters **S1** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), and TEMPO (2.5 equiv.) in EA (0.05 M). Then the vessel was bubbled with a stream of argon for 20 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 3 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion, the crude reaction mixture was filtered over basified alumina and washed with EtOAc. Both, the adduct of the transient carboxyl oxygen radical and of the persistent iminyl radical with TEMPO could be observed in HRMS (γ C−H adduct **65**: $[M+H]^+$ calc 368.2220, found 368.2220; iminyl adduct **66**: $[M+H]^+$ calc 337.2274, found 337.2274; radical–radical self-coupled byproduct **67:** $[M+H]^+$ calc

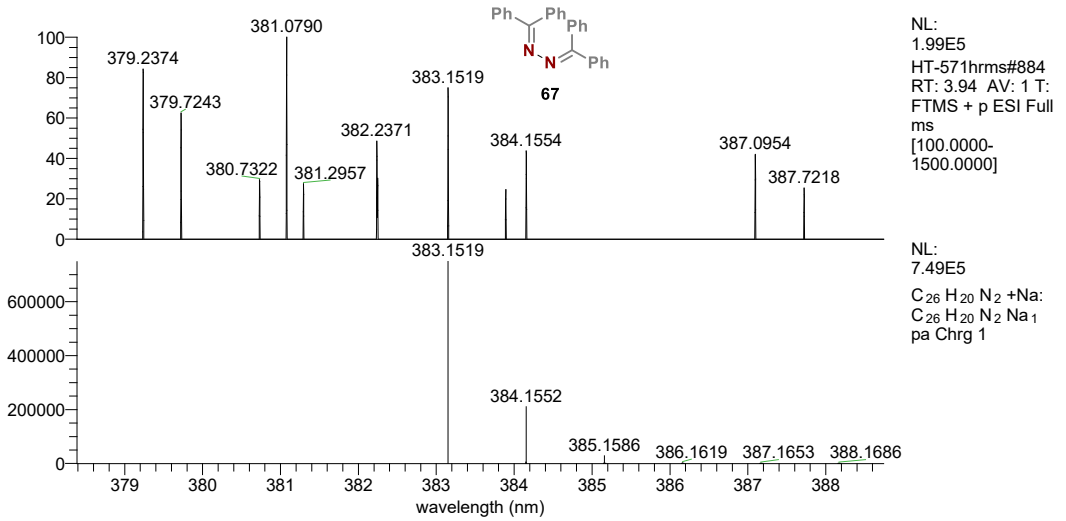
383.1519, found 383.1519 ). No product formation could be observed by NMR analysis.




**Supplementary Figure 3. HRMS of TEMPO-trapping experiment (γ C−H adduct 65)**



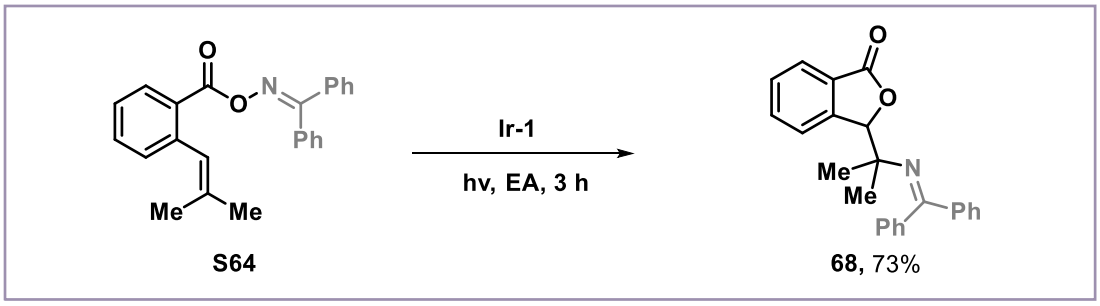

**Supplementary Figure 4. HRMS of TEMPO-trapping experiment (iminyl adduct 66)**




**Supplementary Figure 5. HRMS of TEMPO-trapping experiment (radical–radical self-coupled byproduct 67)**

*Therefore, the TEMPO trapping experiment supports the intermediacy of both aroyloxy radical and persistent iminyl radical and their participation for product formation.*

### 3.1.2 Radical Probe Experiment




**Supplementary Figure 6. Radical Probe Experiment of S64**

To an oven-dried glass tube (10 mL) equipped with a magnetic stir bar were added the oxime esters **S64** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), and EA (0.05 M). Then the vessel was bubbled with a stream of argon for 20 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 3 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion of the reaction, the solvents were removed under reduced pressure, column chromatography on silica gel pre-basified with $NEt_3$

using pentane: EtOAc mixtures afforded 3-(2-((diphenylmethylene)amino)propan-2-yl)isobenzofuran-1(3*H*)-one (**68**, 51.9 mg, 73 %) as a white solid.

**$^1$H NMR (600 MHz, $CDCl_3$)** δ 7.97 – 7.84 (m, 1H), 7.78 (d, *J* = 7.7 Hz, 1H), 7.66 – 7.56 (m, 3H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.45 – 7.31 (m, 6H), 7.22 – 7.08 (m, 2H), 5.65 (s, 1H), 1.40 (s, 3H), 0.59 (s, 3H).

**$^{13}$C NMR (151 MHz, $CDCl_3$)** δ 171.1, 166.1, 148.9, 140.8, 139.1, 133.7, 130.1, 129.0, 128.3, 128.2, 128.1, 128.0, 127.2, 125.3, 124.1, 88.0, 62.7, 26.4, 21.2.

**HRMS (ESI)**: m/z calculated for $[C_{24}H_{22}NO_2]^+$ $[M + H]^+$: 356.1645, found: 356.1645.

*This result suggests the involvement of aroyloxy radicals through a photo-induced EnT process.*

### 3.1.3 Radical Clock Experiment


**Supplementary Figure 7. Radical clock experiment of S64**

To an oven-dried glass tube (10 mL) equipped with a magnetic stir bar were added the oxime esters **S65** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), and EA (0.05 M). Then the vessel was bubbled with a stream of argon for 20 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 3 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion of the reaction, the solvents were removed under reduced pressure, Column chromatography on basified silica gel (hexane/EA = 5/1 – 1/1) afforded 2-(4-((diphenylmethylene)amino)-4-phenylbut-1-en-1-yl)benzoic acid (**69**, 21.58 mg, 25 %) as a colorless oil.

**$^1$H NMR** (600 MHz, $CDCl_3$) δ 9.35 (s, 1H), 7.98 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.59 (dd, *J* = 12.5, 7.4 Hz, 2H), 7.53 – 7.36 (m, 5H), 7.34 – 7.17 (m, 9H), 7.07 – 7.02 (m, 1H), 6.90 (d, *J* = 11.7 Hz, 1H), 5.57 (dt, *J* = 11.7, 7.7 Hz, 1H), 4.60 – 4.34 (m, 1H), 2.72 (ddt, *J* = 90.6, 13.7, 7.1

Hz, 2H).

**$^{13}$C NMR** (151 MHz, $CDCl_3$) δ 171.8, 167.1, 144.2, 139.7, 139.5, 137.6, 136.9, 132.4, 132.2, 131.2, 131.0, 130.9, 130.1, 129.9, 128.6, 128.4, 128.3, 128.3, 128.3, 127.9, 127.8, 127.1, 126.8, 126.7, 66.5, 37.9.

**HRMS (ESI)**: m/z calculated for $[C_{30}H_{26}NO_2]^+$ $[M + H]^+$: 432.1958, found: 432.1958.

*This result further suggests the involvement of benzyl radicals formed from the γ-position of aroyloxy radicals via 1,5-intramolecular HAT.*

### 3.1.4 Quantum Yield Measurement

A solution of ferrioxalate was chosen as an actinometer following the procedure described by the IUPAC (subcommittee on photochemistry).[11,12] The procedure is based on the decomposition under irradiation of ferric ions to ferrous ions which are complexed by 1,10-phenanthroline. This photochemical transformation has a known quantum yield, and the complexation of $Fe^{2+}$ with 1,10-phenanthroline can be monitored by UV-visible absorption since its extinction coefficient at 510 nm is known (ε = 11100 $M^{-1}$ $cm^{-1}$).[13] Therefore, the moles of the iron-phenanthroline complex formed are related to moles of photons absorbed. 0.006 M, 0.012 M, or 0.15 M solutions of ferrioxalate can be used for actinometry. In this case we chose a concentration of 0.15 M. The solutions were prepared and stored in a dark laboratory as follows:

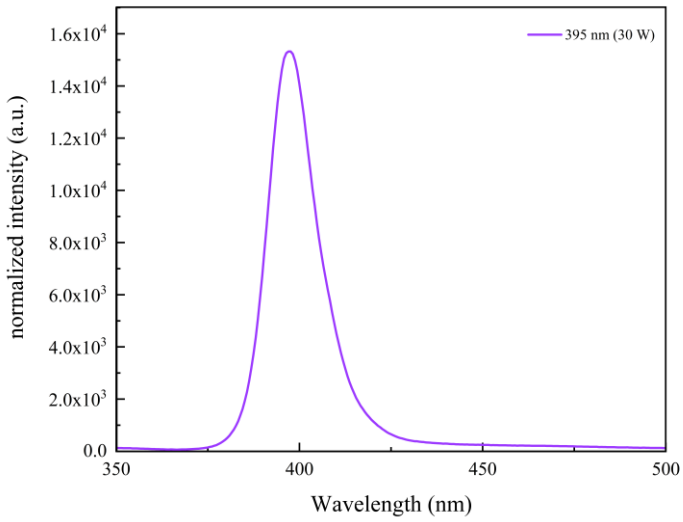
**Solution A**: 1.84 g of $K_3[Fe(C_2O_4)_3]$•$3H_2O$ and 1.75 mL of $H_2SO_4$ were added into a 25 mL volumetric flack and filled to the mark with ultrapure water.

**Solution B**: 49.6 mg of 1,10-phenanthroline monohydrate, 10.0 g of NaOAc, and 1.0 mL of H2SO4 were added to a 100-mL volumetric flask and filled to the mark with ultrapure water.

**Model reaction solution**: Oxime ester **S1** (78.2 mg, 0.2 mmol, 1.0 equiv.), and $[Ir(dFppy)_2(phpzpy)]PF_6$ (1mol %), were added to an oven-dried quartz cuvette (l = 1 cm) equipped with a magnetic stir bar and a plastic plug. The combined materials were dissolved in deoxygenated EA (4.0 mL) under argon atmosphere.

**Actinometry procedure**: The simultaneous irradiation of both the actinometer solution and model reaction by the self-same reactor setup is not feasible. However, the stability of the irradiation light

was checked through radiometer measurements (recorded on an AVANTES® AvaSpec-ULS2048 spectrometer instrument). Therefore, we assumed that consecutive measurements of both actinometer and model reaction are comparable. We used 30 W LED as a light source, detecting a maximum wavelength of emission of 395 nm (**Supplementary Figure 8**).




**Supplementary Figure 8.** The Emission spectrum of the photochemical reactor (λmax = 395 nm).

(1) 2 mL of solution A was added to a quartz cuvette under dark conditions while being stirred. Then, the actinometry solutions were irradiated with a 395 nm LED for specified time intervals (0, 5, 10, 15, 20 s) and a 0.1 mL aliquot was taken.

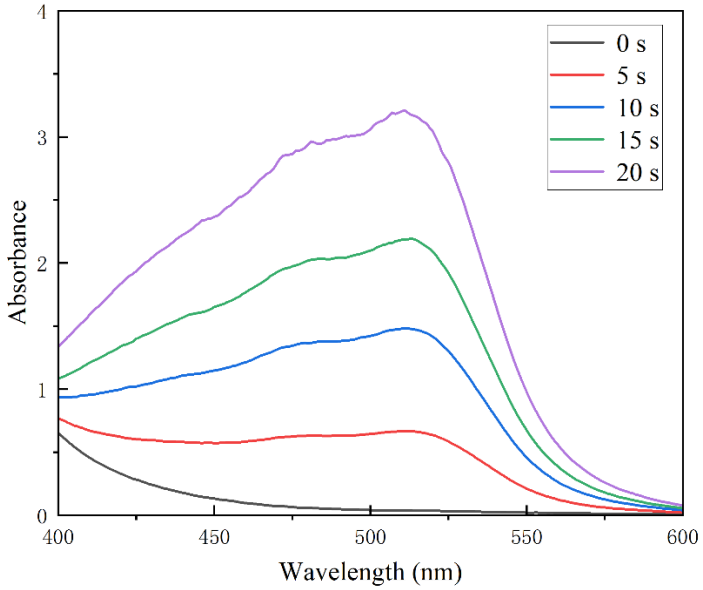
(2) 4 mL solution B was added to each aliquot, and the final volume was raised to 10 mL with ultrapure water. All samples were stored in the dark and stirred for one hour.

(3) The absorbance spectrum of each sample was monitored at 510 nm for each time interval by the UV-2600 spectrometer. The absorbance to each time was related to the photochemically produced $Fe^{2+}$ ions across the Lambert-Beer Law (Equation [1]):

$$\text{moles } Fe^{2+} = \frac{V1 \times V3 \times \Delta A(510\ nm)}{V2 \times 1 \times \varepsilon(510\ nm)} \qquad [1]$$

Where V1 is the irradiated volume (2 mL), V2 is the aliquot of the irradiated solution taken for the determination of the ferrous ions (0.1 mL), V3 is the final volume after complexation with phenanthroline (10 mL), l is the optical path-length of the irradiation quartz cuvette (1 cm), ΔA(510

nm) the optical difference in absorbance between the irradiated solution and that taken in the dark, ε(510 nm) is the extinction coefficient of the complex $Fe(phen)_3^{2+}$ (11100 $M^{-1}$ $cm^{-1}$ ).

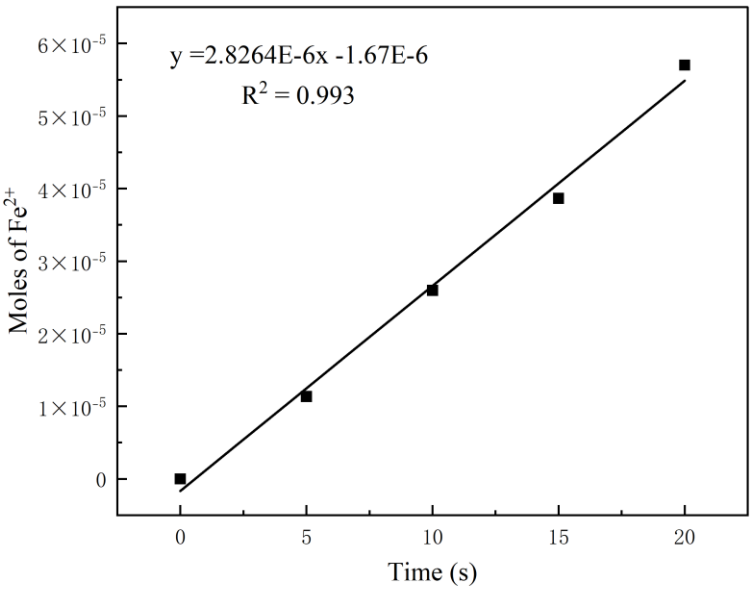



**Supplementary Figure 9.** The absorbance spectrum of each time interval.

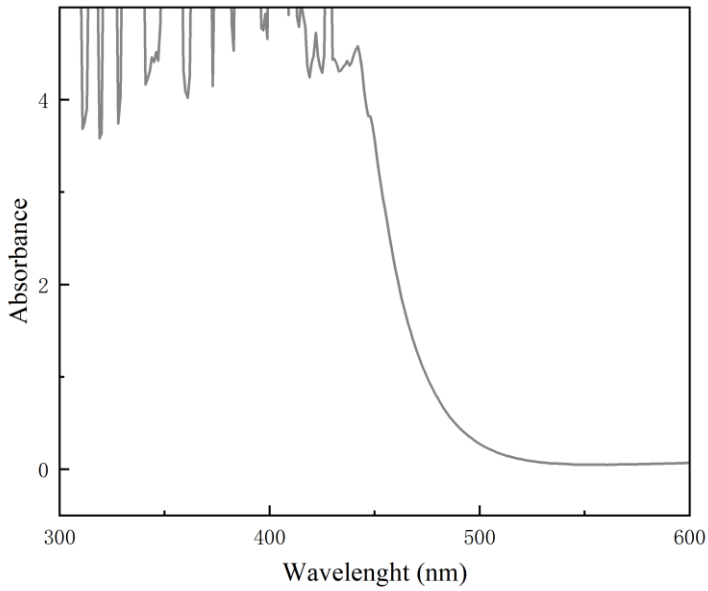
(4) The moles of $Fe^{2+}$ formed (x) are plotted as a function of time (t) (Figure S10.). The slope of the line (dx/dt) was correlated to the moles of incident photons by unit of time ($q_{n,p}$) using the following equation [2]:

$$q_{n,p} = \frac{dx/dt}{\Phi(\lambda) \times f} \quad , f = [1 - 10^{-A(\lambda)}] \qquad [2]$$

Where Φ (λ) is the quantum yield of the actinometer reaction at the irradiated wavelength, in this case being 1.13 at 395 nm.[12,14] The value of the photon flux must be divided by the fraction of absorbed light f at the irradiation wavelength, and A(λ) is the absorbance of the actinometer solution (ferrioxalate) at the irradiated wavelength (395 nm) obtaining a value of 6.4, f = 1. Therefore, the moles of incident photons by a unit of time were determined as $2.50 \times 10^{-6}$ einstein $s^{-1}$.



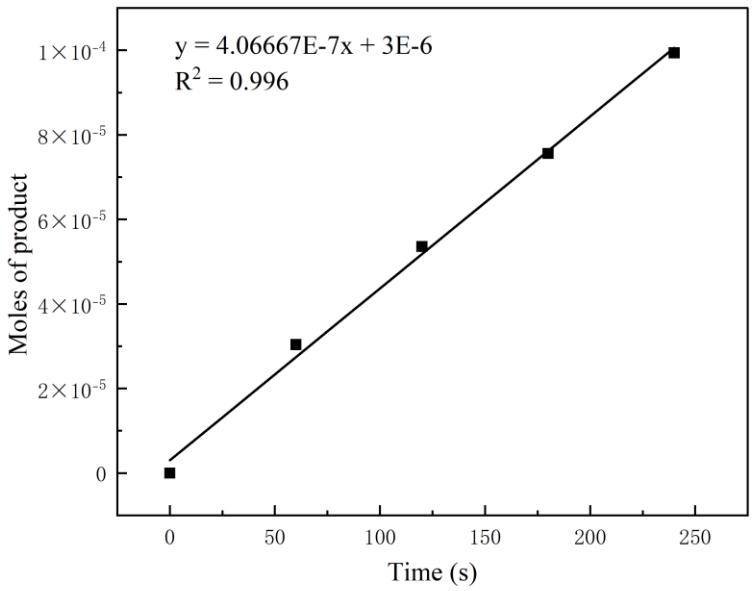

**Supplementary Figure 10.** The moles of $Fe^{2+}$ formed (x) are plotted as a function of time (t).




**Supplementary Figure 11.** Absorbance of a 0.15 M solution of $K_3[Fe(C_2O_4)_3]•3H_2O$ in ultrapure water.

The kinetics of the reaction under study were done by irradiating the actinometer solution described above. The model reaction solutions were irradiated using the same spectrometer with consecutive measurements every minute. The moles of products formed were determined by $^1$H-NMR spectrum

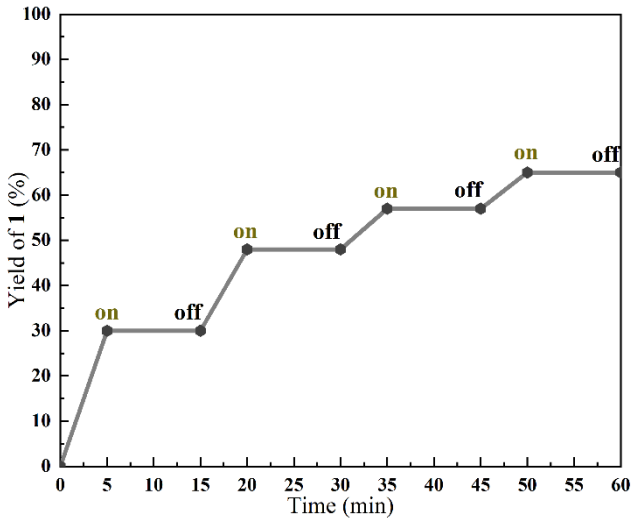
in $CDCl_3$. Plotting the moles of product versus the irradiation time, the slope dx/dt can be related to the quantum yield across the equation [2] being equal to $(q_{n,p})\times\Phi(\lambda)\times(1\text{-}10^{-A(\lambda)})$. The model reaction solution was added to a 1 cm optical pathway cuvette to measure A (395 nm), The quantum yield at 395 nm of the reaction by $[Ir(dFppy)_2(phpzpy)]PF_6$ was calculated as **0.24**.




**Supplementary Figure 12.** Plotting the moles of product versus the irradiation time

### 3.1.5 Light On/Off Experiment

Under standard conditions, the light On/Off experiments were carried out where the light was switched on every 5 min and off every 10 min with stirring maintained.

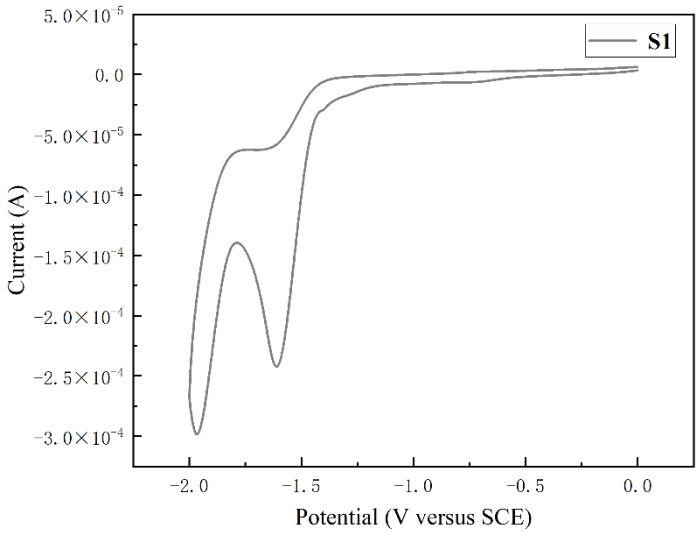



**Supplementary Figure 13.** Light On/Off experiment

*The results clearly show that the reaction only takes place when the light is on.*

### 3.1.6 Cyclic Voltammetry Studies

The experiments were conducted using a cyclic potentiometer with a glassy carbon working electrode polished by aluminum oxide (50 nm), a Pt counter electrode and a saturated calomel electrode (SCE) reference electrode. In the standard procedure, 0.1 mmol of substrate (**S1**) were dissolved in 10 mL of a 0.1 M $[N(Bu)_4]PF_6$. electrolyte solution in MeCN. Each measurement was conducted at 50 mV/s at room temperature without stirring.

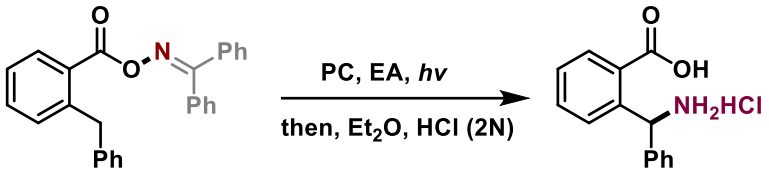



**Supplementary Figure 14.** The Cyclic Voltammetry (CV) experiments of substrates **S1** and $[N(Bu)_4]PF_6$ in MeCN, [**S1**] = $1.00\times10^{-2}$ M. **$E_{red}$ = -1.61 V vs SCE**

*Excited **Ir-1** not reducing enough to reduce the reagent. No product formation was observed by using more reducing photocatalysts (see section 3.1.6).*

### 3.1.7 Evaluation of C–H amination between yield and photocatalyst triplet energy

To investigate the substrate activation, present in the disclosed photocatalytic manifolds, photocatalysts of different triplet energy (and redox potentials) were evaluated in cross-relationship with product yield for the γ C–H amination. (**Supplementary Table 5**) Triplet energies and redox potentials (vs SCE) were adopted from the following sources.[15,16]




S1 1

| Entry | PC | $E_T$ (Kcal/mol) | $E_{1/2}^{[PC]^+/[PC]^*}$ (V) | $E_{1/2}^{[PC]^*/[PC]^-}$ (V) | Yield of 1 |
|---|---|---|---|---|---|
| 1[a] | Thioxanthone | 65.5 | -1.11 | +1.18 | 73% |
| 2 | $[Ir(dFppy)_2(phpzpy)]PF_6$ | 62.8 | -1.16 | +1.21 | 82% |
| 3[b] | $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ | 61.8 | -0.89 | +1.21 | 57% |
| 4[b] | *fac*-$Ir(ppy)_3$ | 58.1 | -1.73 | +0.31 | N.D. |
| 5[c] | 4CzIPN | 57.1 | -1.04 | +1.43 | 58% |
| 6[b] | $Ru(bpy)_3Cl_2$ | 46.5 | -0.81 | +0.77 | N.D. |

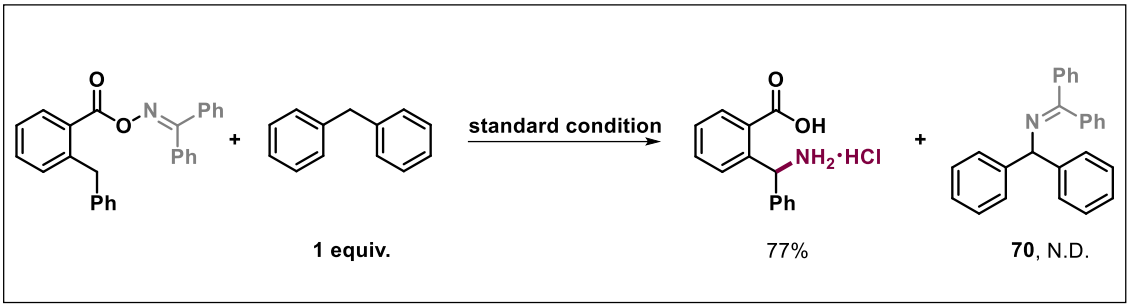
**Supplementary Table 5.** Evaluation of cross-relationship between yield and photocatalyst triplet energy.

For each entry, to an oven-dried glass tube (10 mL) equipped with a magnetic stir bar were added **S1** (0.2 mmol, 1.0 equiv.), PC (1 mol%), and EA (0.05 M). Then the vessel was bubbled with a stream of argon for 20 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 3 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated. Upon completion of the reaction, the solvents were removed under reduced pressure, acidified by 2 mL HCl (2 N) and stirred in $Et_2O$ (2 mL) for 2 h at room temperature. After consumption of the starting material was confirmed by TLC analysis, the solution was poured into water (2.0 mL) and extracted with $Et_2O$ (10 mL x 3). The water layer was combined, and water was removed *in vacuo*, and the product was obtained as a white solid. [a]5 mol% PC used. [b]The reaction was irradiated at 450nm. [c]5 mol% PC used, 450 nm.

*In summary, the correlation between triplet energy and the yield of the γ C-H amination product is shown. These results implied that an EnT process is likely to have been operational in the reaction.*

### 3.1.8 Regioselective experiment of aroyloxy radicals in HAT process

To demonstrate that this is an intramolecular hydrogen atom transfer (HAT) process rather than an intermolecular process, we artificially added an alkane with a lower bond dissociation energy (BDE) to the reaction system. Fortunately, no intermolecular HAT product **70** was formed, confirming that this is indeed an intramolecular HAT process.



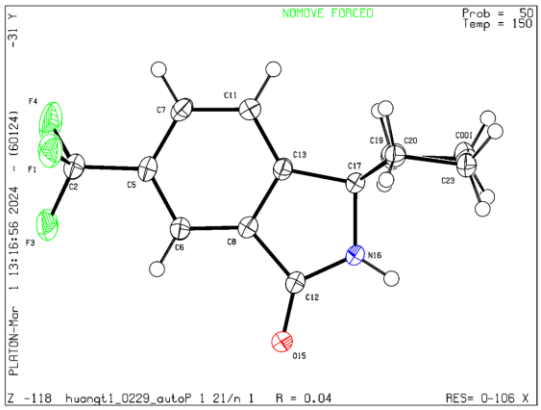

**Supplementary Figure 15.** Regioselective experiment of aroyloxy radicals in HAT process

To an oven-dried glass tube (10 mL) equipped with a magnetic stir bar were added the oxime esters **S1** (0.2 mmol, 1.0 equiv.), $[Ir(dFppy)_2(phpzpy)]PF_6$ (1 mol%), diphenylmethane (1 equiv.) and EA (0.05 M). Then the vessel was bubbled with a stream of argon for 20 min via a syringe needle and the tightly sealed tube was irradiated with two 30 W purple LEDs ($\lambda_{max}$ = 395 nm) at room temperature for 3 h (1 cm away, with a cooling fan to keep the reaction temperature at 25-30 °C and keeping the reaction region located in the center of LEDs lamp), unless otherwise stated.

After consumption of the starting material was confirmed by TLC analysis, the solvents were removed under reduced pressure, acidified by 2 mL HCl (2 N) and stirred in $Et_2O$ (2 mL) for 2 h at room temperature. the solution was poured into water (2.0 mL) and extracted with $Et_2O$ (10 mL x 3). The water layer was combined, and water was removed *in vacuo*, and the product was obtained as a white solid **1**(40.6 mg, 77%).

## 3.2 X-Ray Crystallographic Data

Single crystal of **60** was obtained by recrystallization from a mixed solvent of $CH_2Cl_2$/ petroleum ether at room temperature (evaporation in air). The X-ray single-crystal determination was performed on a Bruker APEX II X-ray single crystal diffractometer.




**Supplementary Figure 16.** X-Ray diffraction of **60** (CCDC-2336872).

| | | | |
|---|---|---|---|
| Bond precision: | C-C = 0. 0019 A | | Wavelength=0.71073 |
| Cell: | a=10.6674(5) | b=10.0950(4) | c=11.8549(6) |
| | alpha=90 | beta=115.445(6) | gamma=90 |
| Temperature: | 150 K | | |

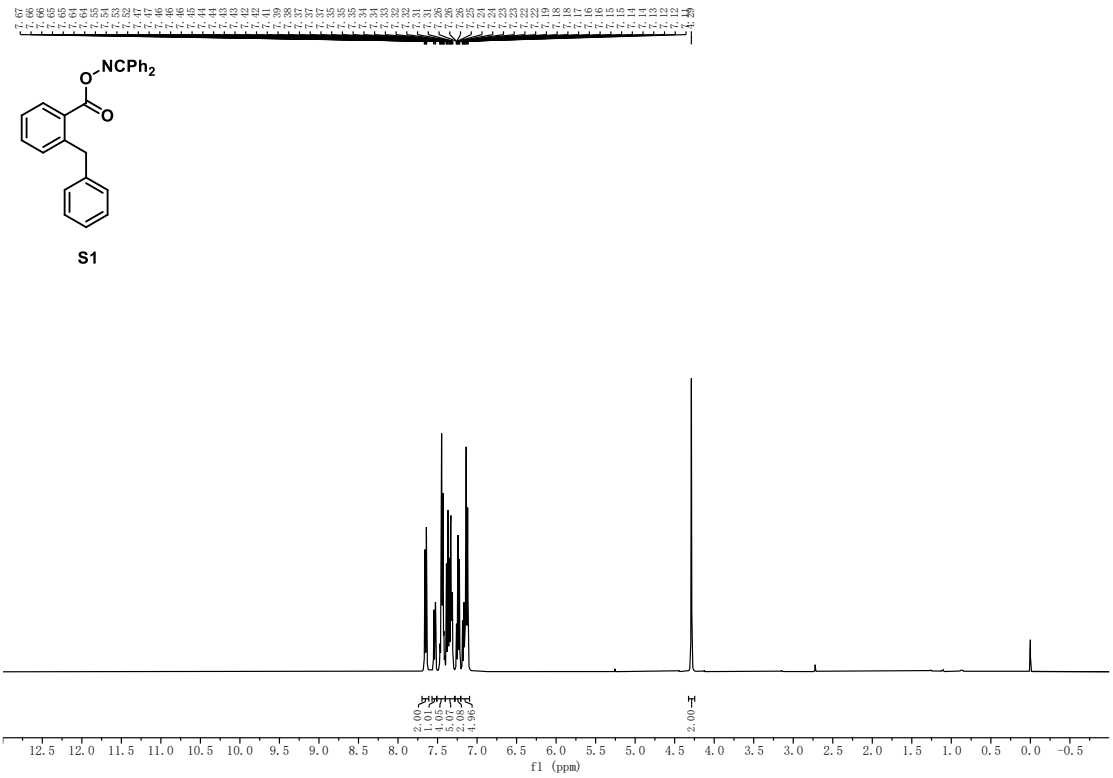
| | Calculated | Reported |
|---|---|---|
| Volume | 1152.79(11) | 1152.79(11) |
| Spacegroup | P 21/n | P 1 21/n 1 |
| Hall group | -P 2yn | -P 2yn |
| Moiety formula | C13 H12 F3 N O | C13 H12 F3 N O |
| Sum formula | C13 H12 F3 N O | C13 H12 F3 N O |
| Mr | 255.24 | 255.24 |
| Dx, g cm-3 | 1.471 | 1.471 |
| Z | 4 | 4 |
| Mu (mm-1) | 0.126 | 0.126 |
| F000 | 528.0 | 528.0 |
| F000’ | 528.37 | |
| h, k, lmax | 15,14,17 | 15,14,17 |
| Nref | 3579 | 2899 |
| Tmin, Tmax | 0.989,0.991 | 0.948,1.000 |
| Tmin’ | 0.989 | |

Correction method= # Reported T Limits: Tmin=0.948 Tmax=1.000
AbsCorr = MULTI-SCAN

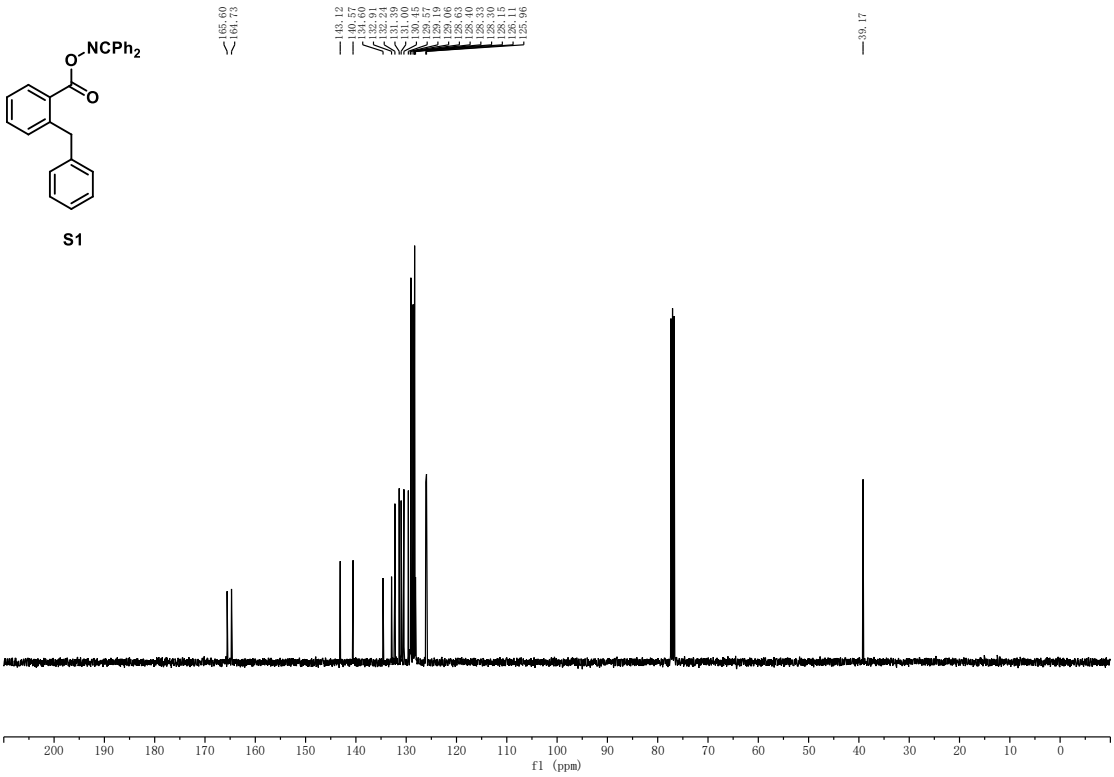
Data completeness= 0.810 Theta(max)= 30.707
R(reflections)= 0.0427 ( 2438) wR2(reflections)= 0.1118 ( 2899)

S = 1.049 Npar= 163

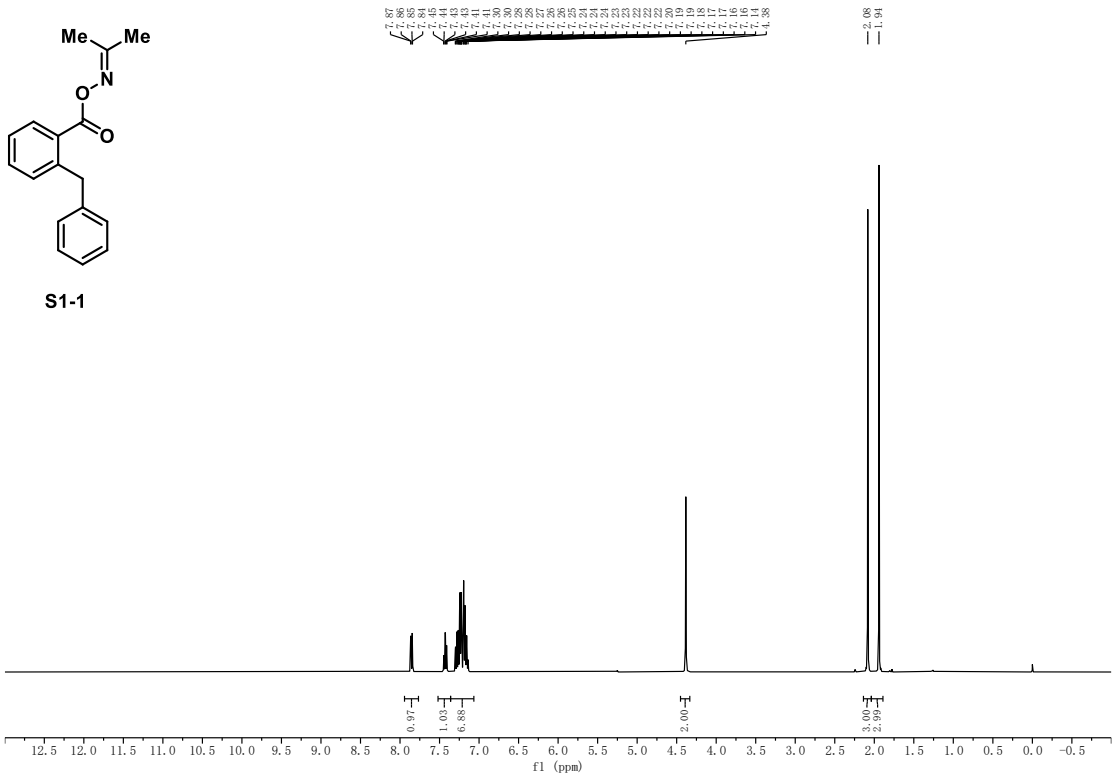
## 3.3 NMR spectra



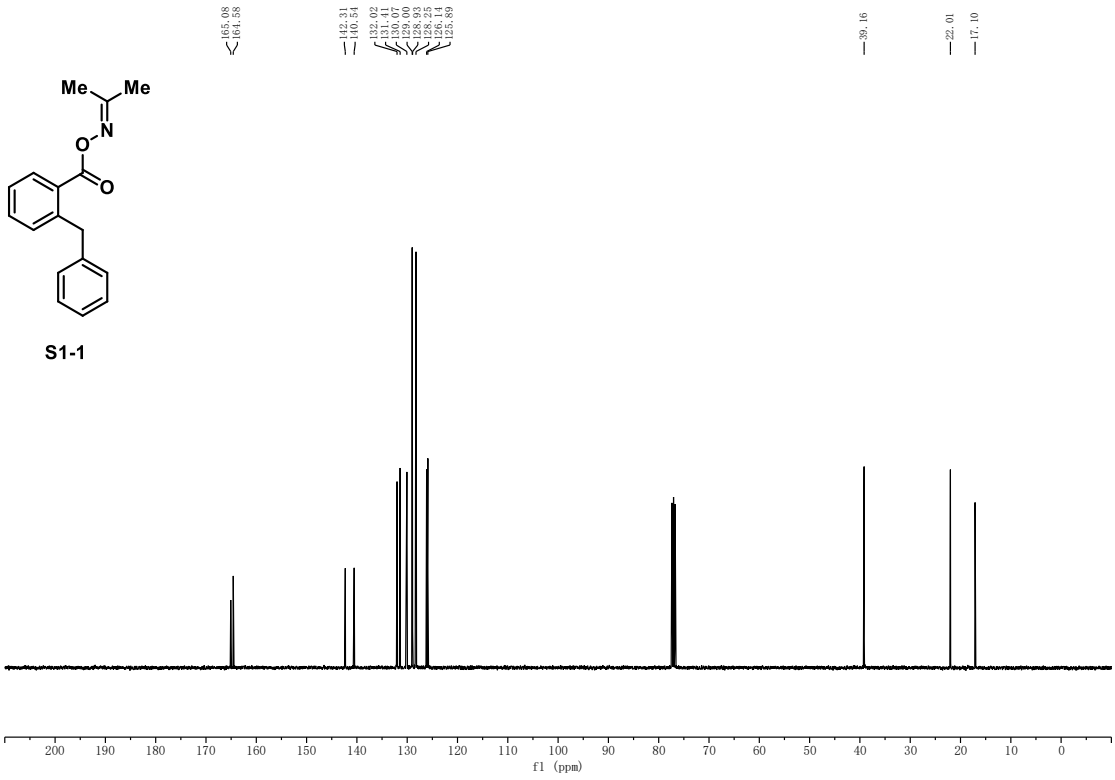

**Supplementary Figure 17** $^{1}$H NMR spectrum of **S1**



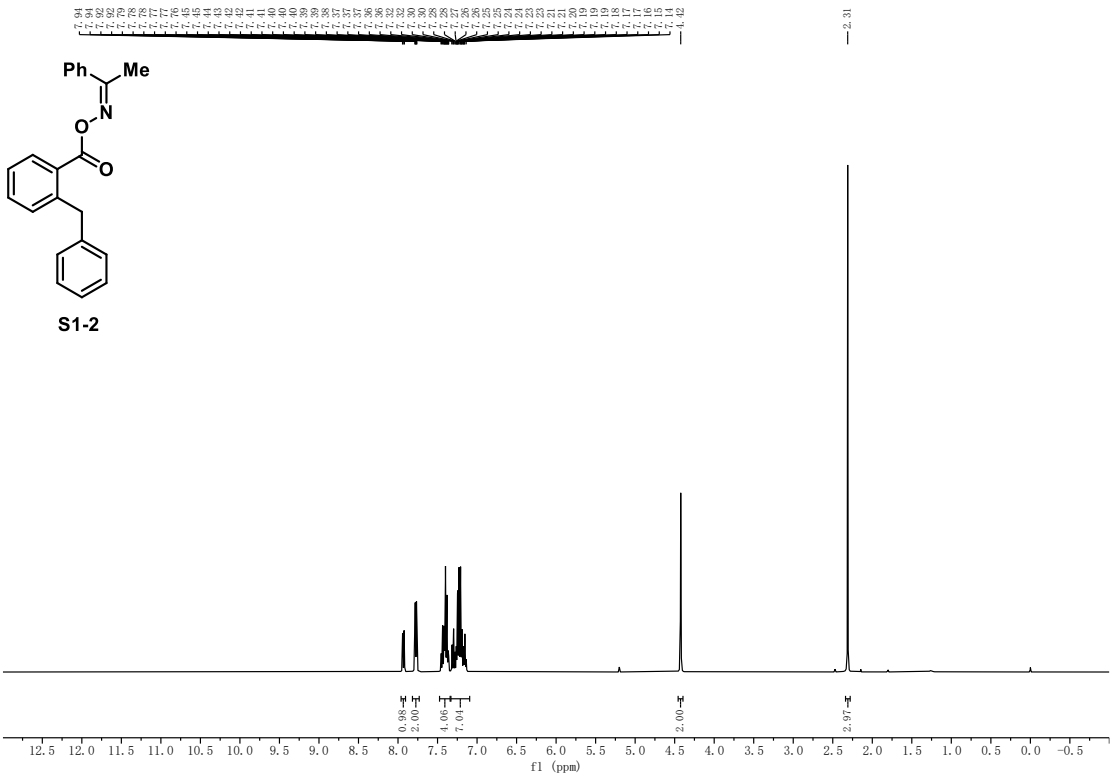

**Supplementary Figure 18** $^{13}$C NMR spectrum of **S1**



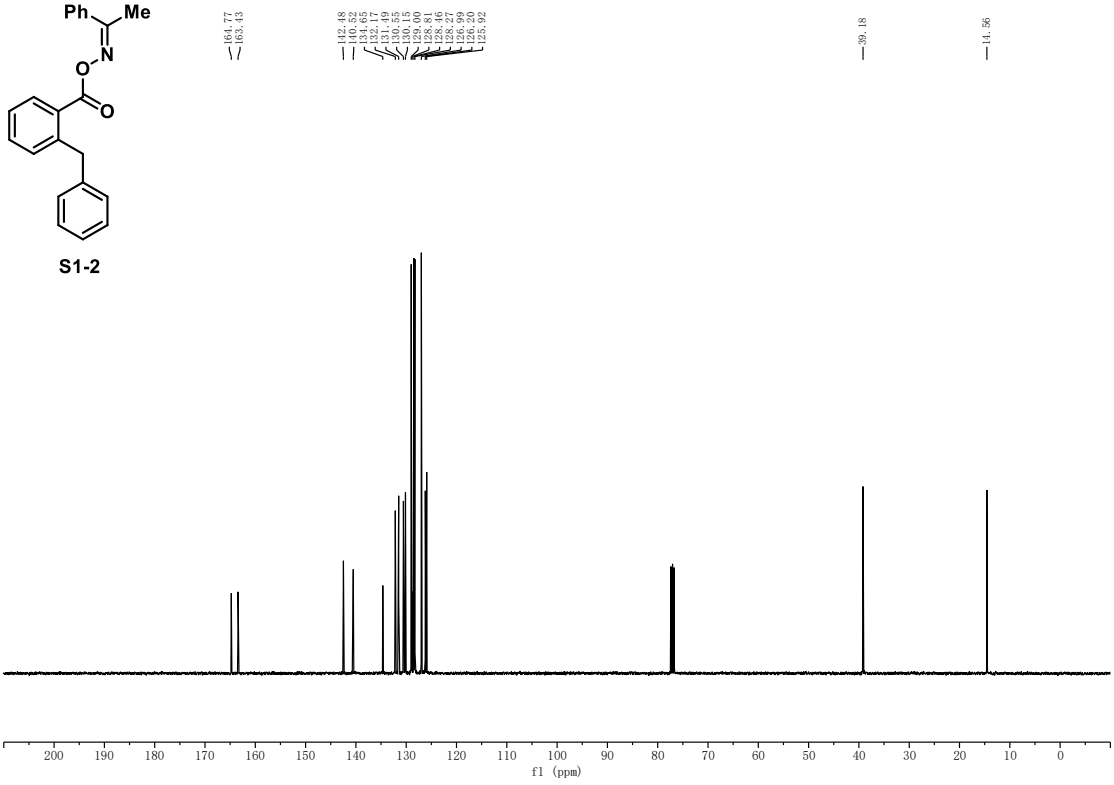

**Supplementary Figure 19** $^{1}$H NMR spectrum of **S1-1**



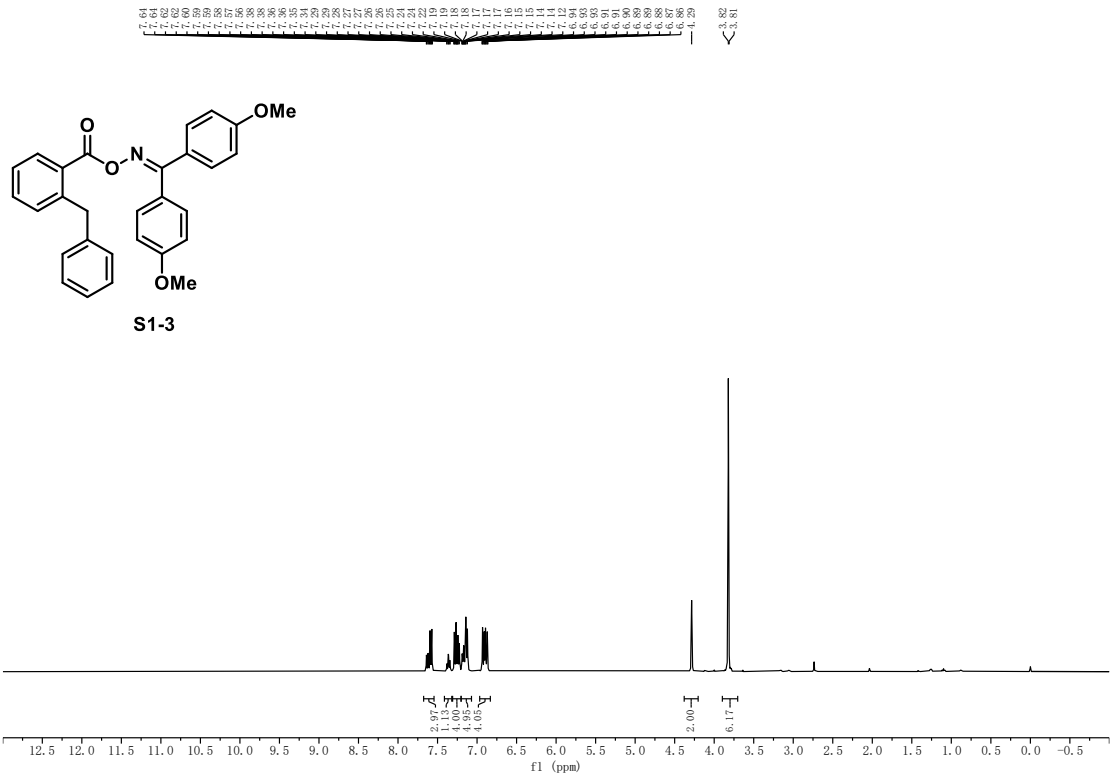

**Supplementary Figure 20** $^{13}$C NMR spectrum of **S1-1**



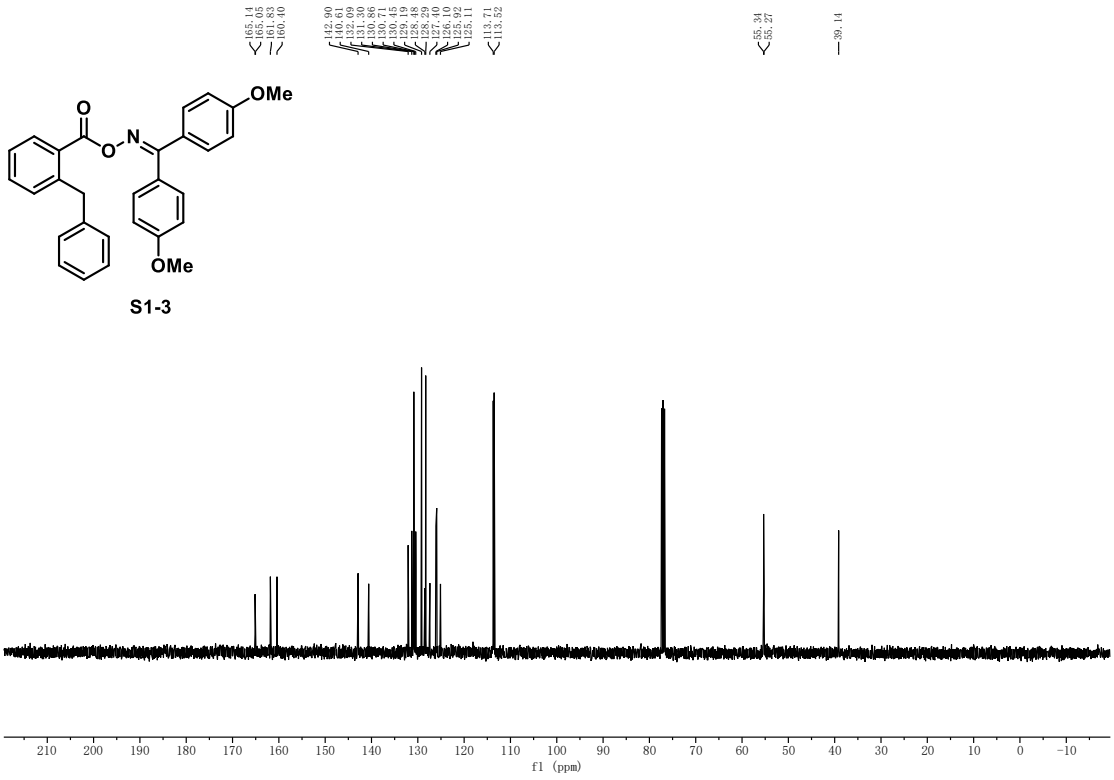

**Supplementary Figure 21** $^{1}$H NMR spectrum of **S1-2**



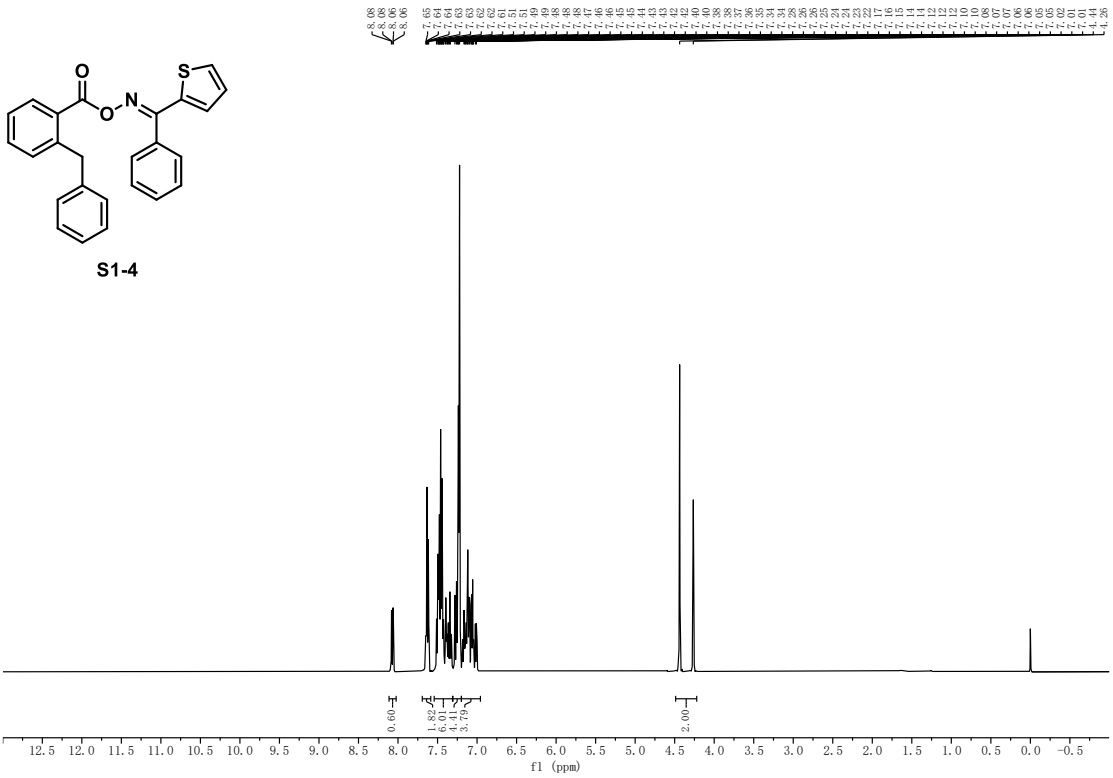

**Supplementary Figure 22** $^{13}$C NMR spectrum of **S1-2**



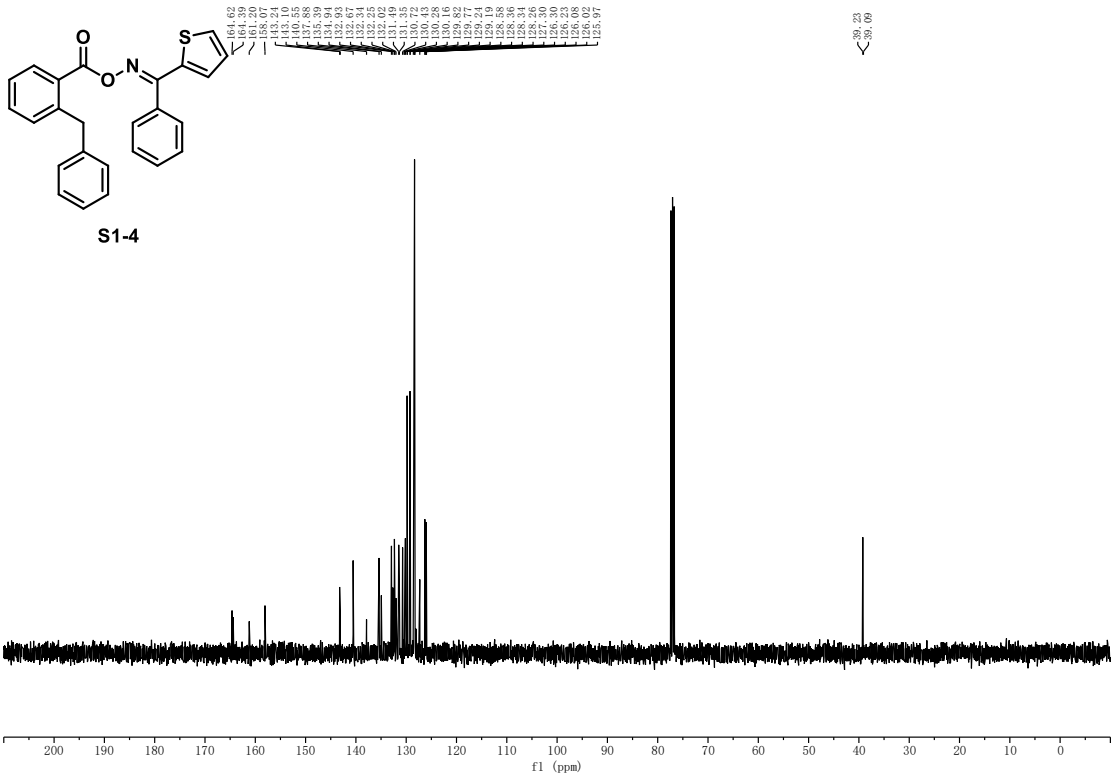

**Supplementary Figure 23** $^{1}$H NMR spectrum of **S1-3**



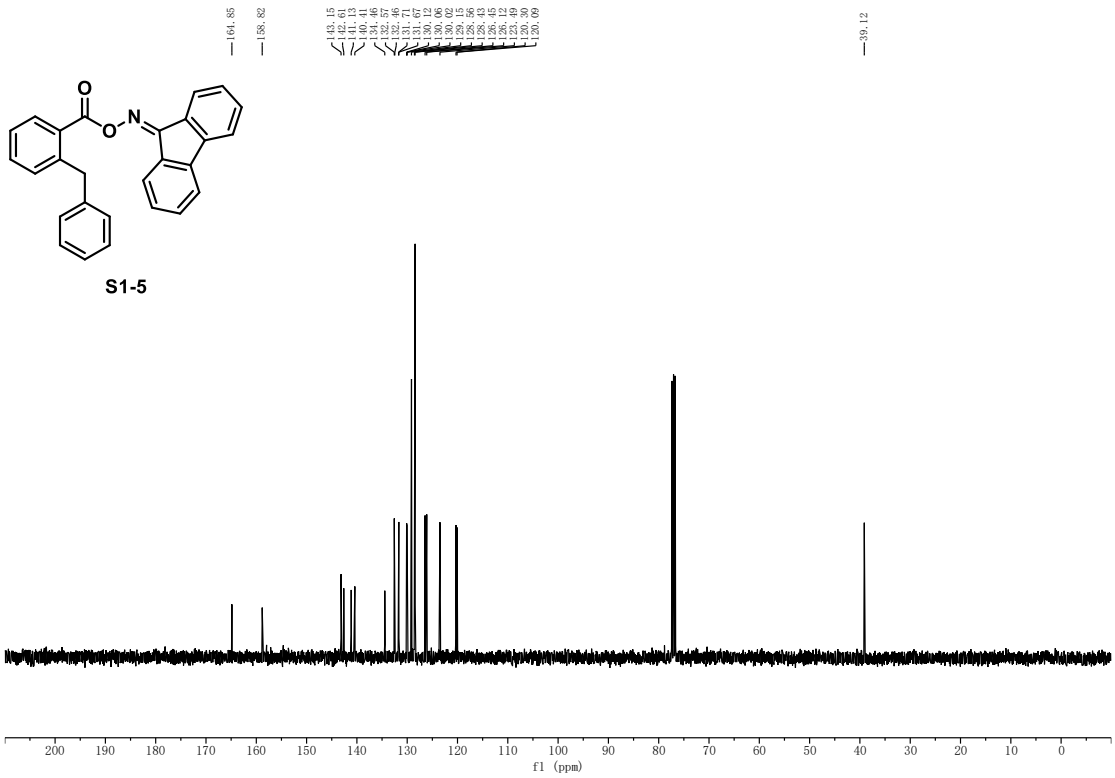

**Supplementary Figure 24** $^{13}$C NMR spectrum of **S1-3**



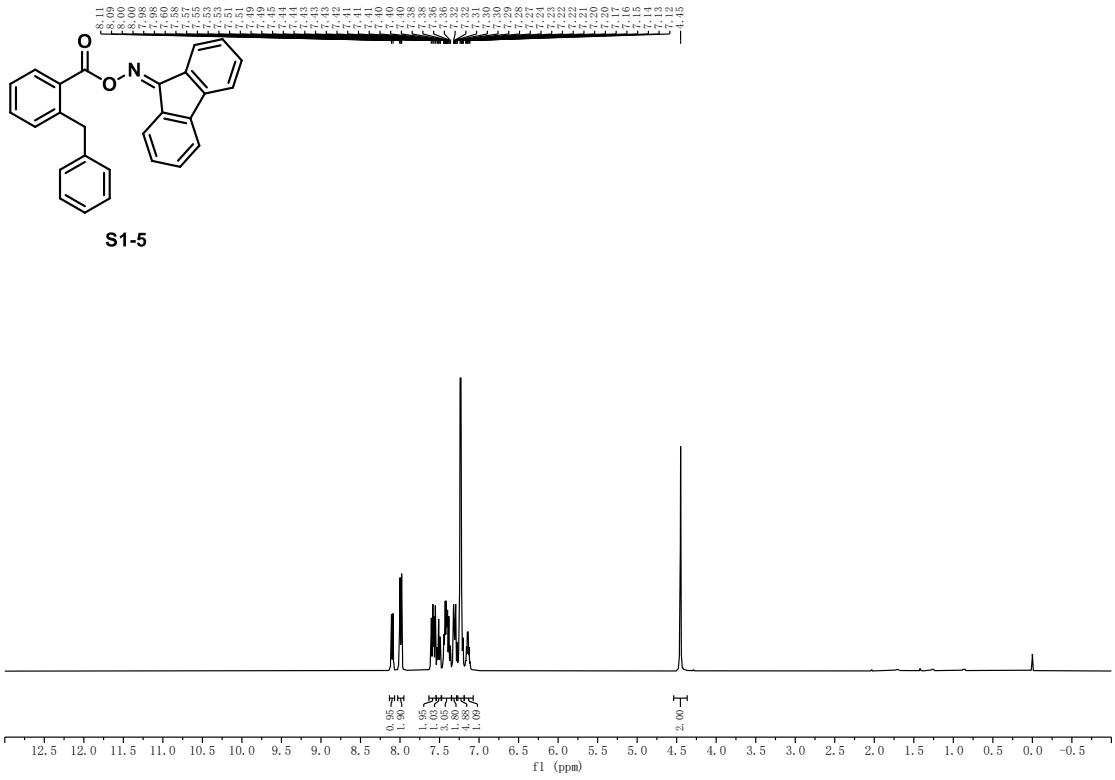

**Supplementary Figure 25** $^{1}$H NMR spectrum of **S1-2**



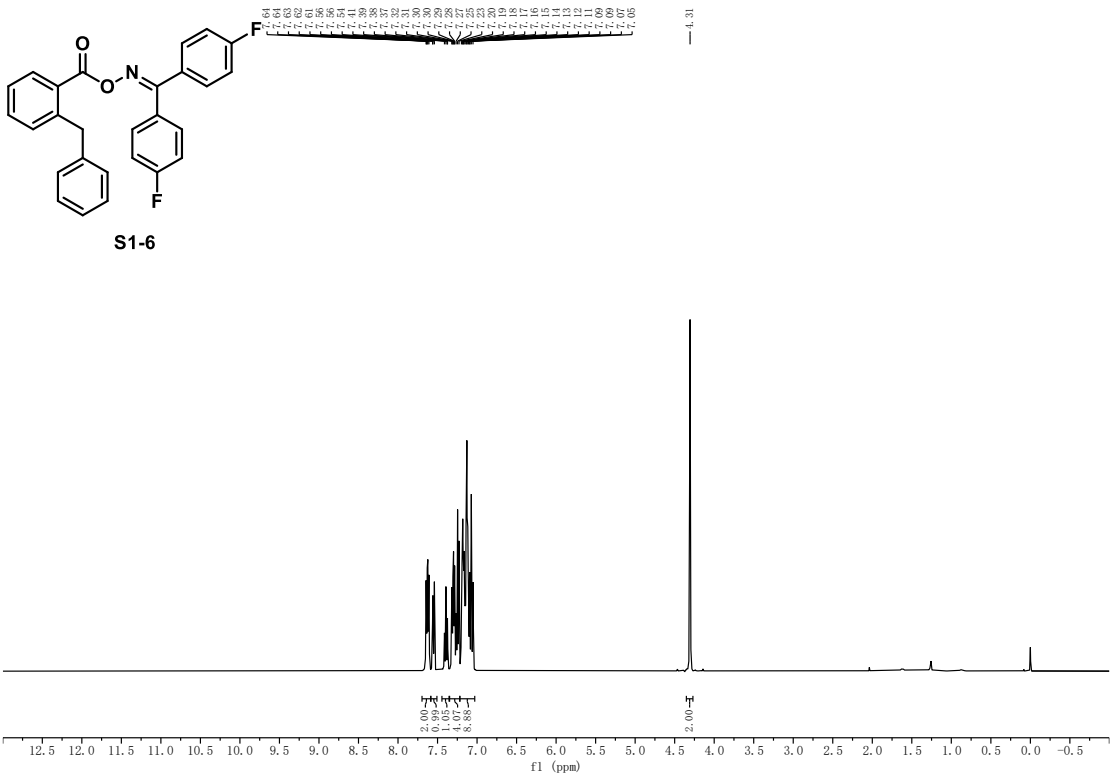

**Supplementary Figure 26** $^{13}$C NMR spectrum of **S1-4**



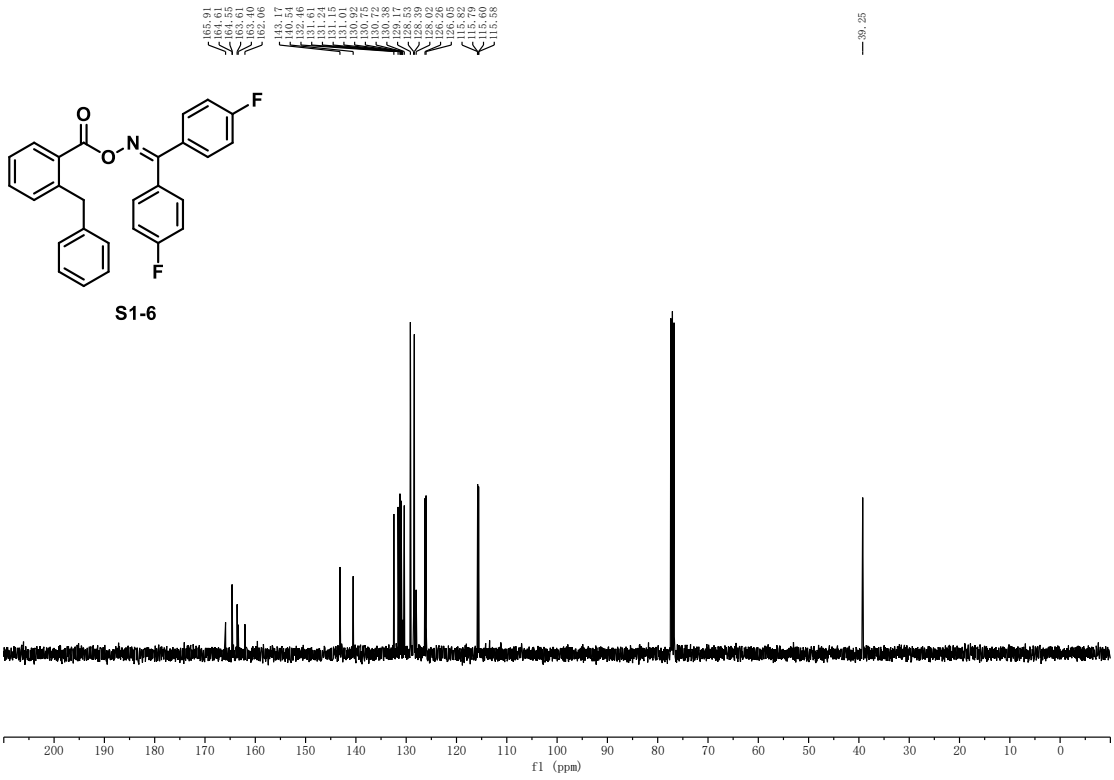

**Supplementary Figure 27** $^{1}$H NMR spectrum of **S1-5**



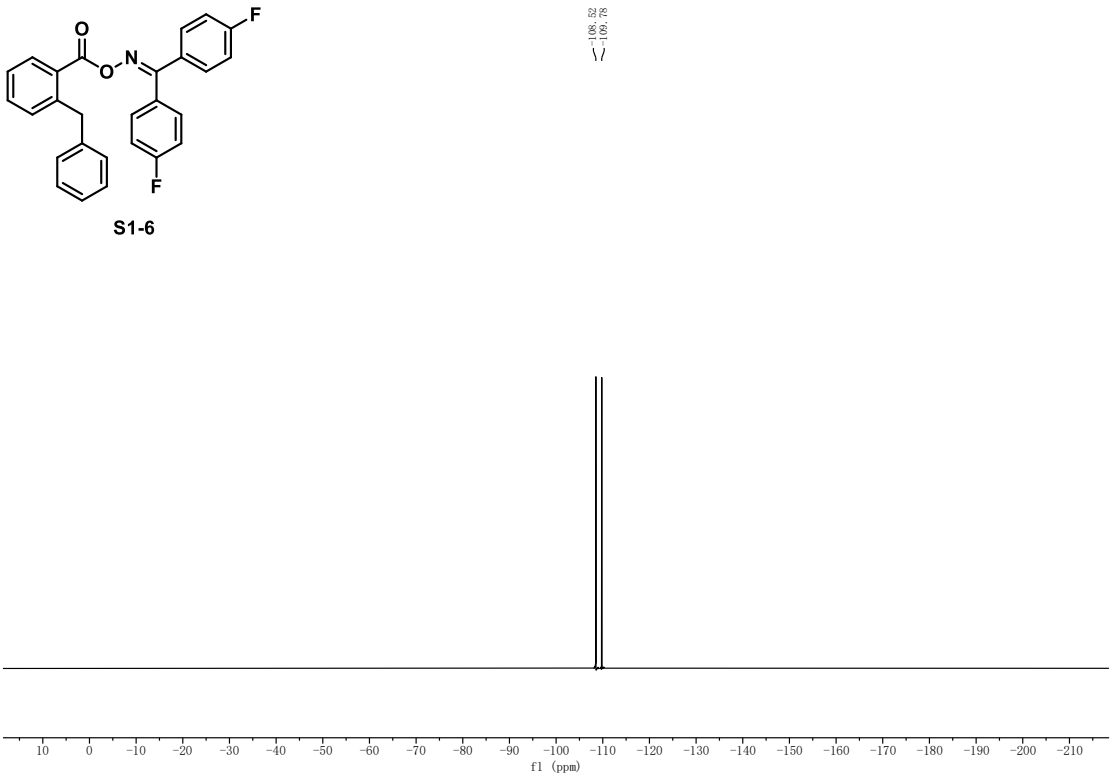

**Supplementary Figure 28** $^{13}$C NMR spectrum of **S1-5**



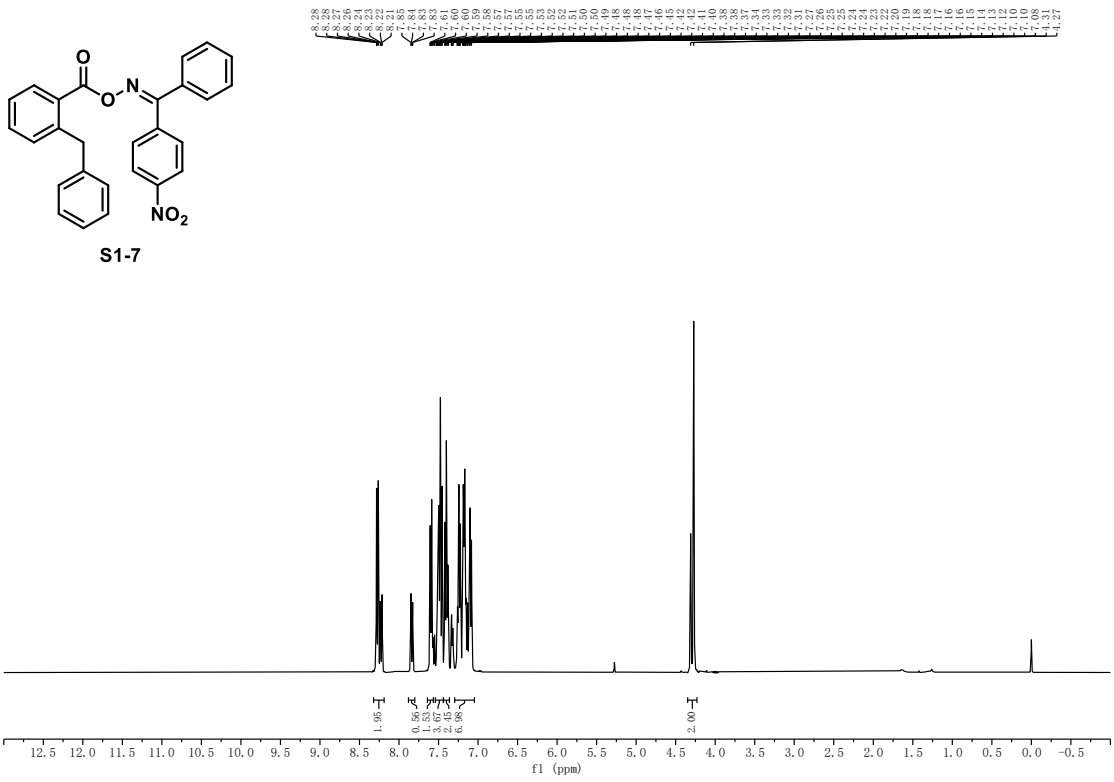

**Supplementary Figure 29** $^{1}$H NMR spectrum of **S1-6**



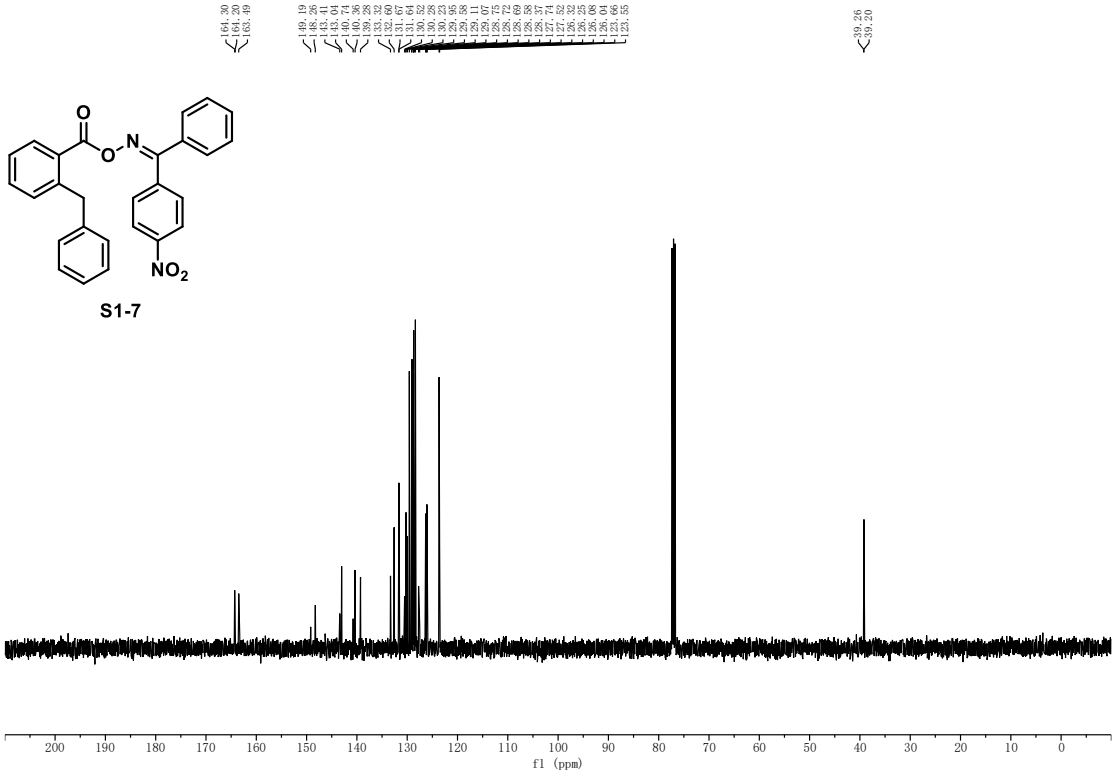

**Supplementary Figure 30** $^{13}$C NMR spectrum of **S1-6**



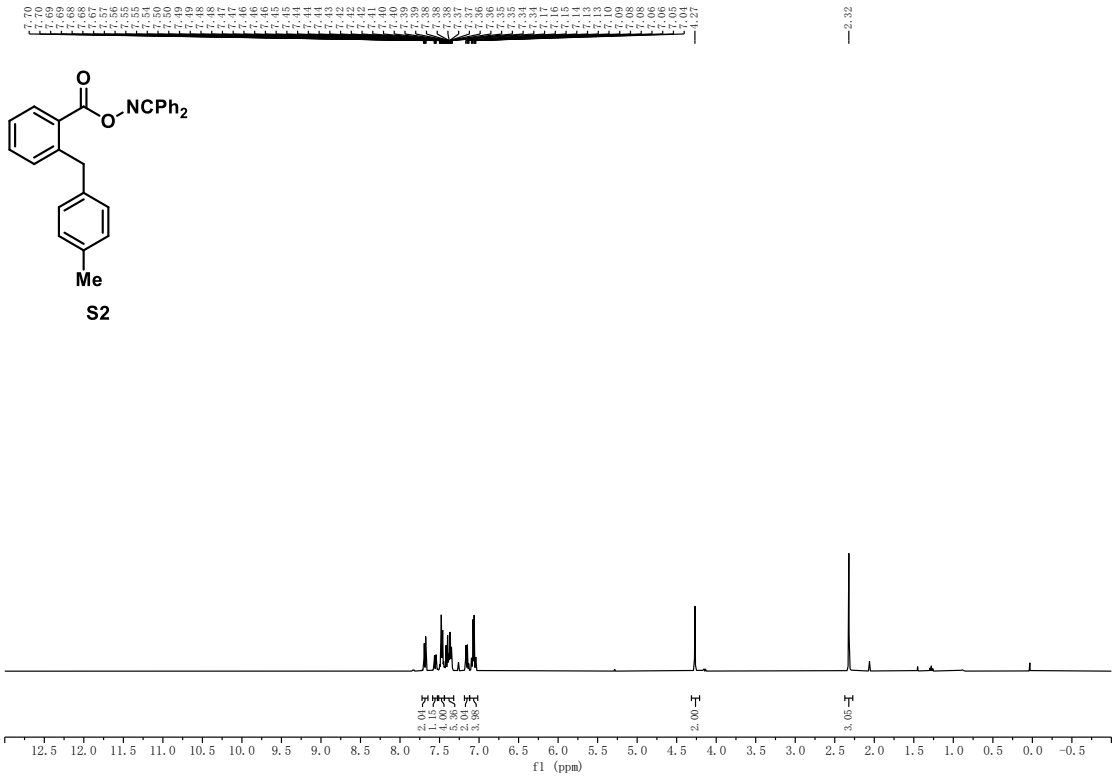

**Supplementary Figure 31** $^{19}$F NMR spectrum of **S1-6**



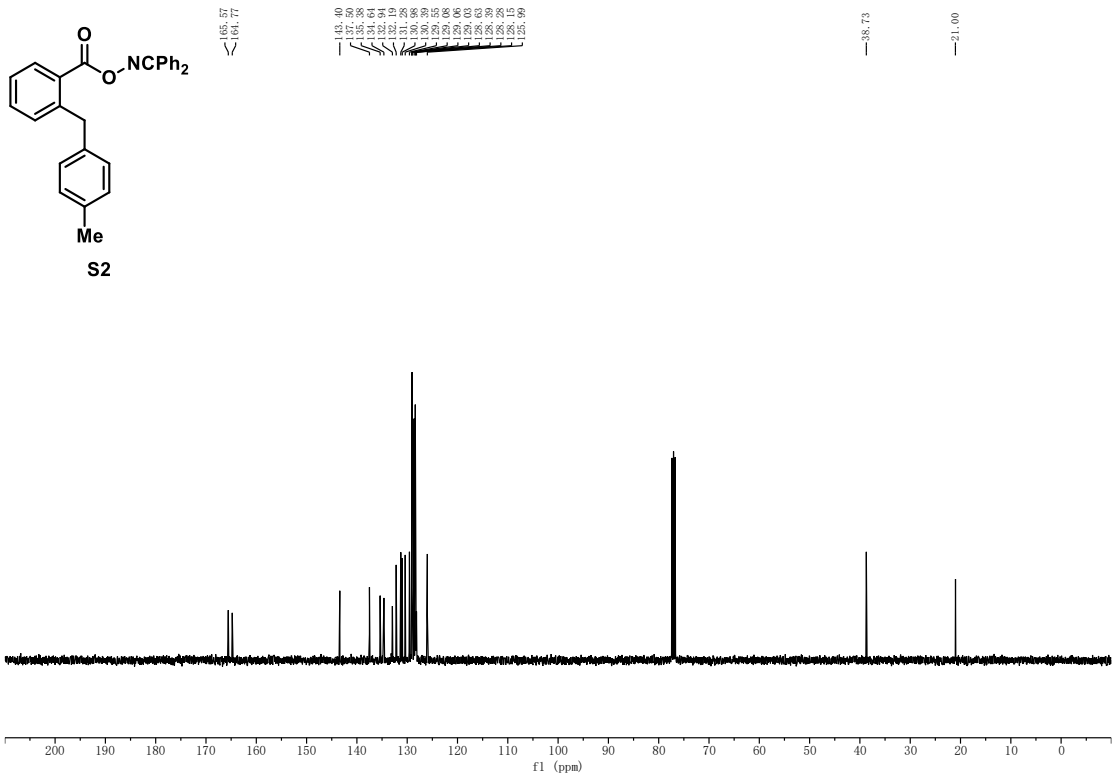

**Supplementary Figure 32** $^{1}$H NMR spectrum of **S1-7**



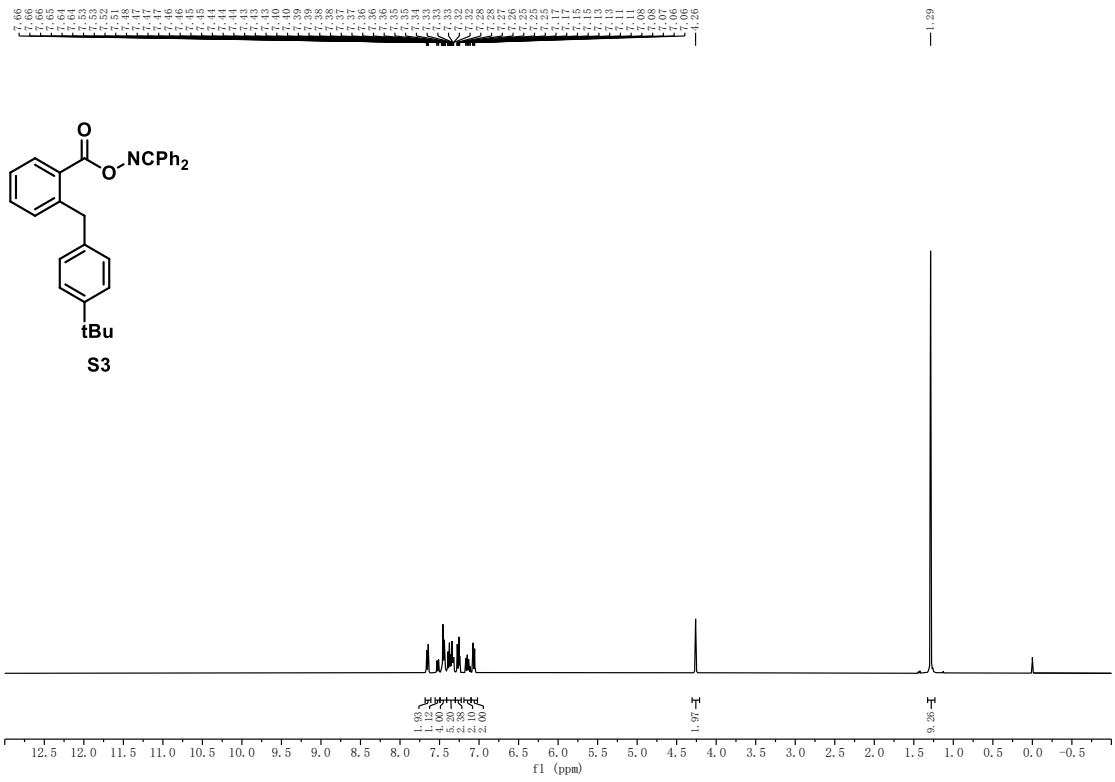

**Supplementary Figure 33** $^{13}$C NMR spectrum of **S1-7**



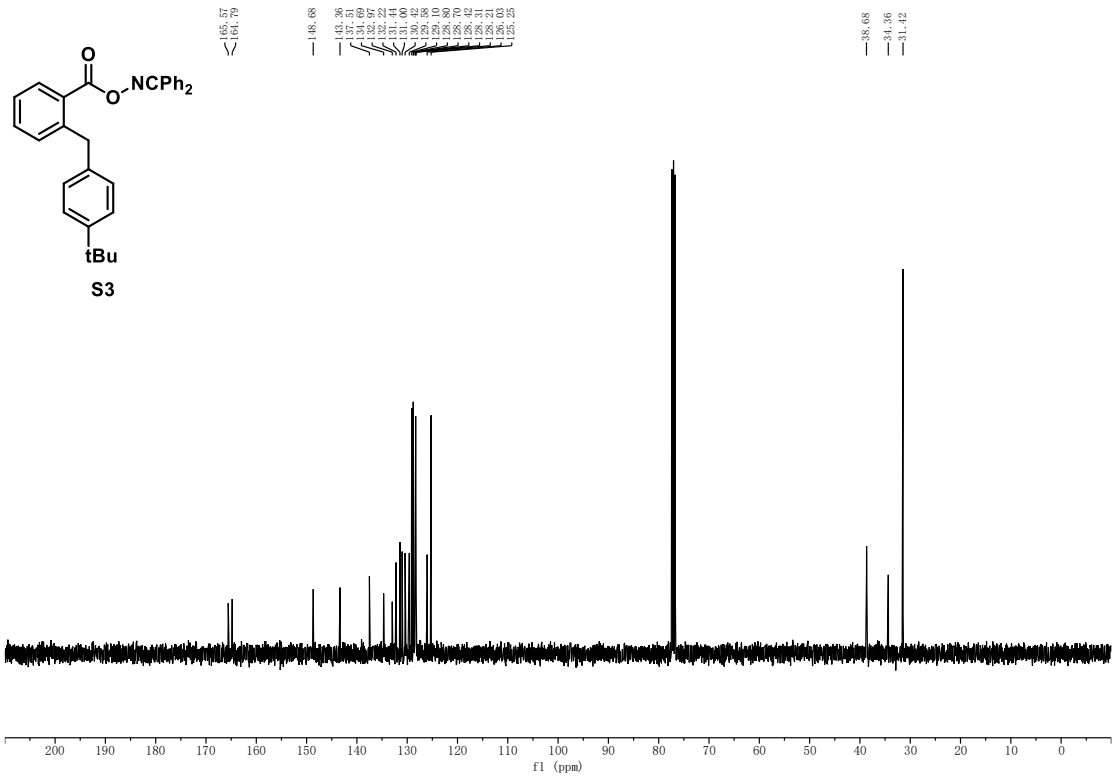

**Supplementary Figure 34** $^{1}$H NMR spectrum of **S2**



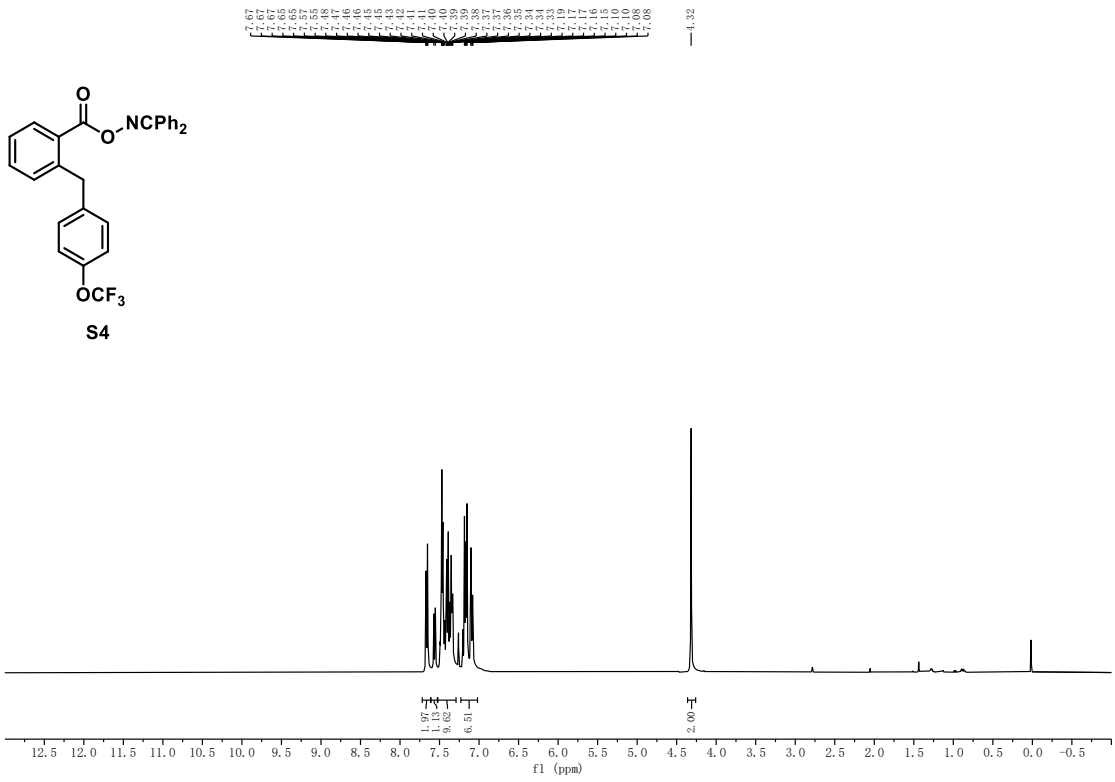

**Supplementary Figure 35** $^{13}$C NMR spectrum of **S2**



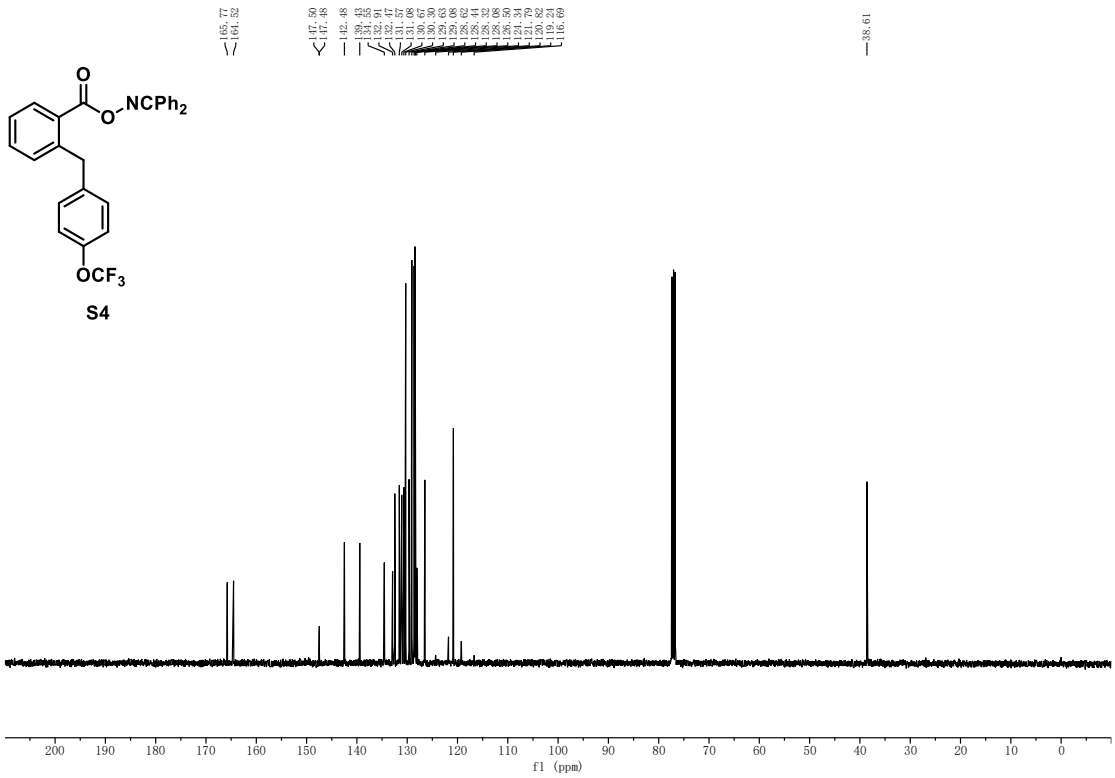

**Supplementary Figure 36** $^{1}$H NMR spectrum of **S3**



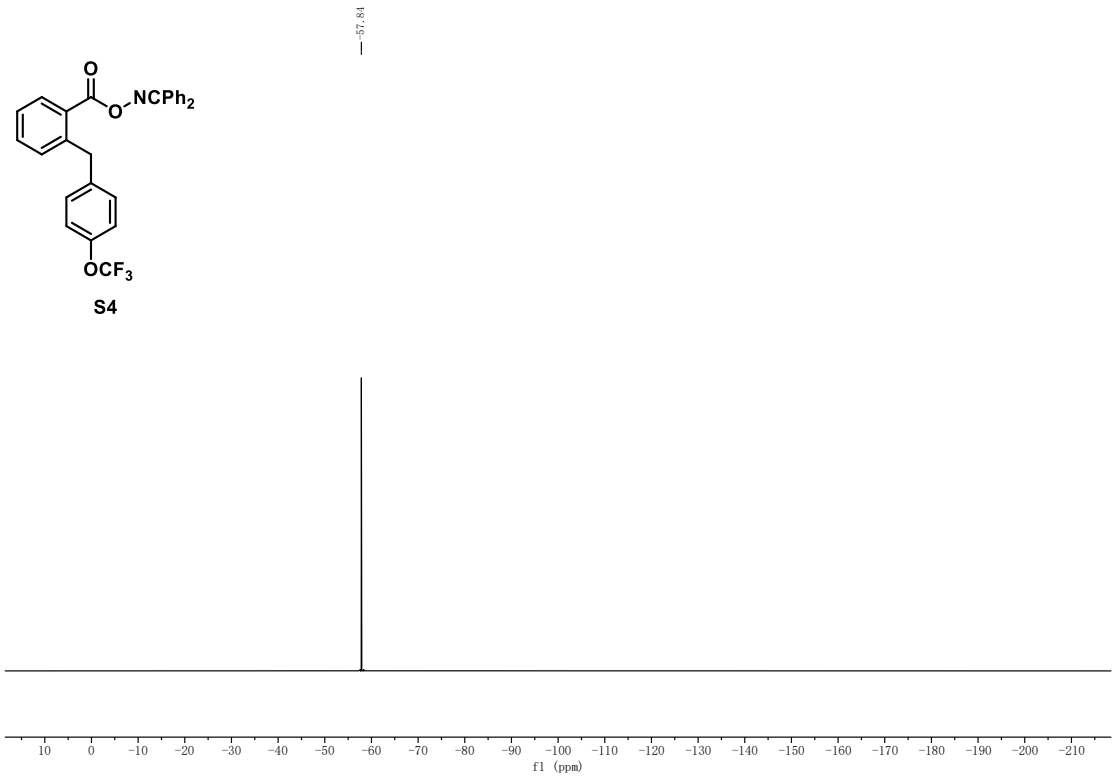

**Supplementary Figure 37** $^{13}$C NMR spectrum of **S3**



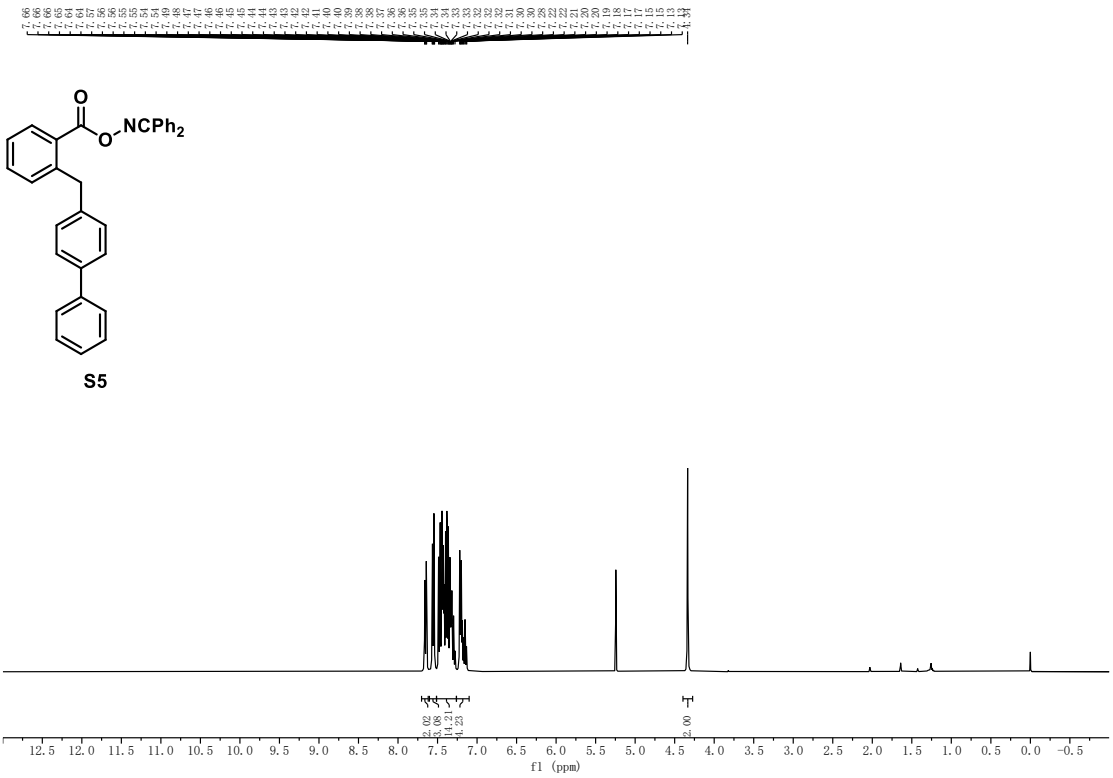

**Supplementary Figure 38** $^{1}$H NMR spectrum of **S4**



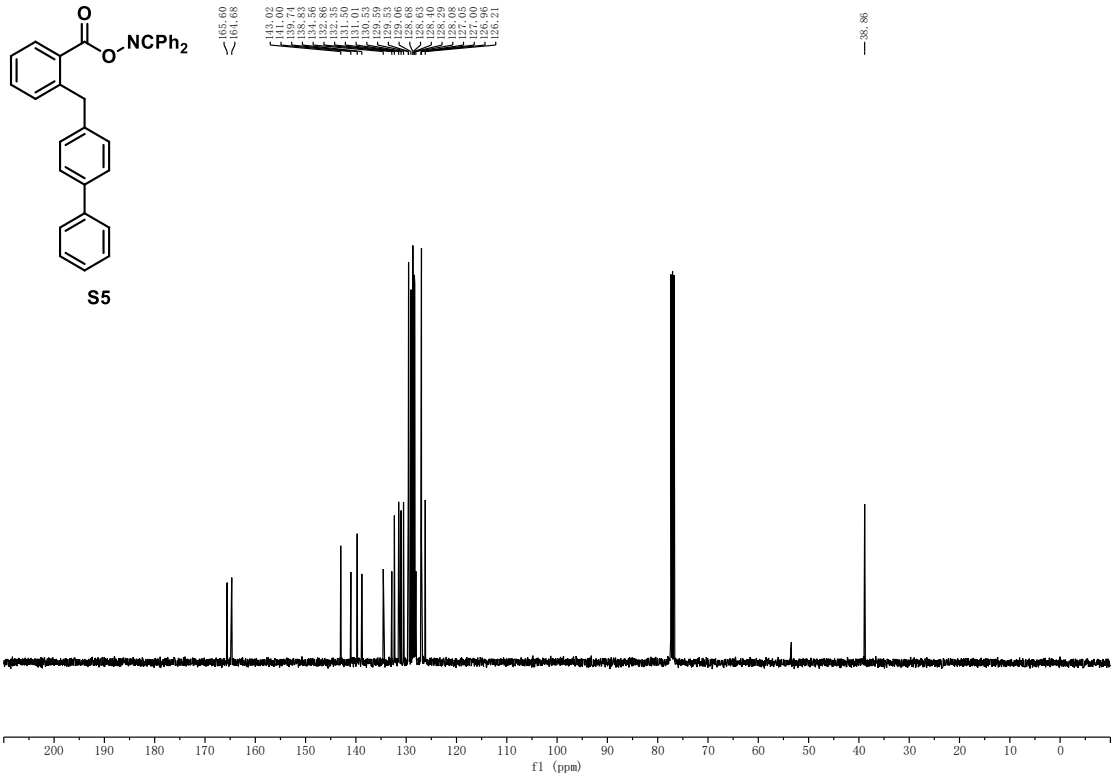

**Supplementary Figure 39** $^{13}$C NMR spectrum of **S4**



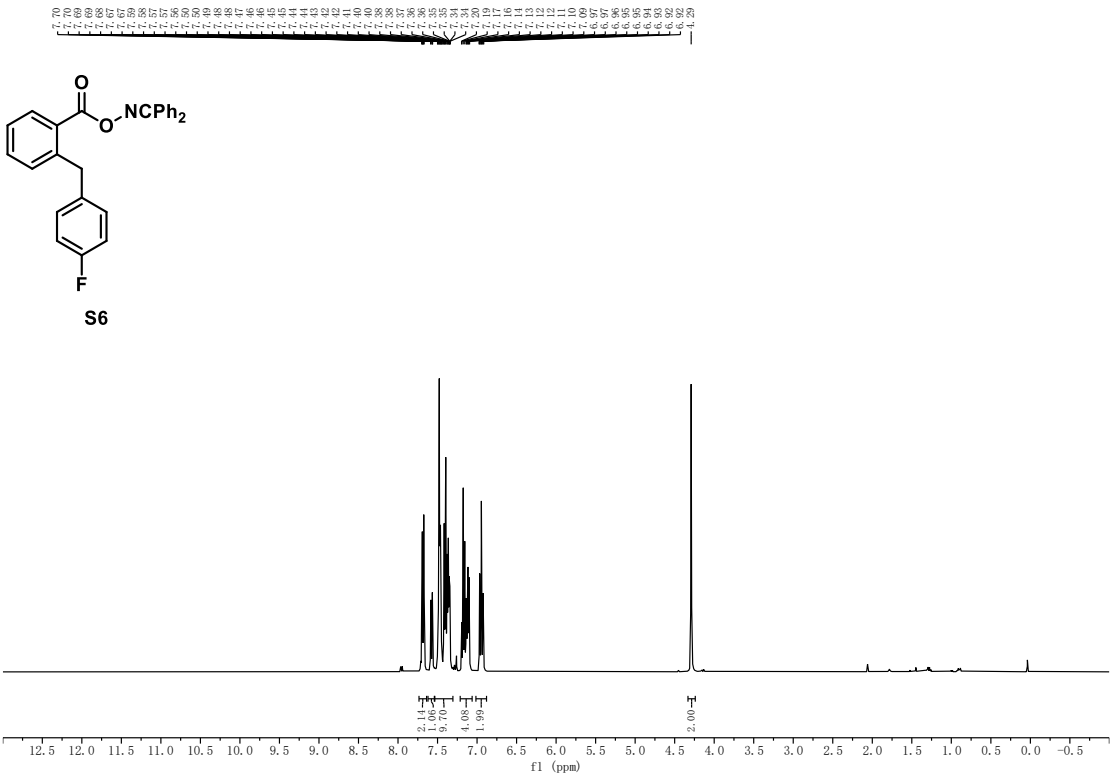

**Supplementary Figure 40** $^{19}$F NMR spectrum of **S4**



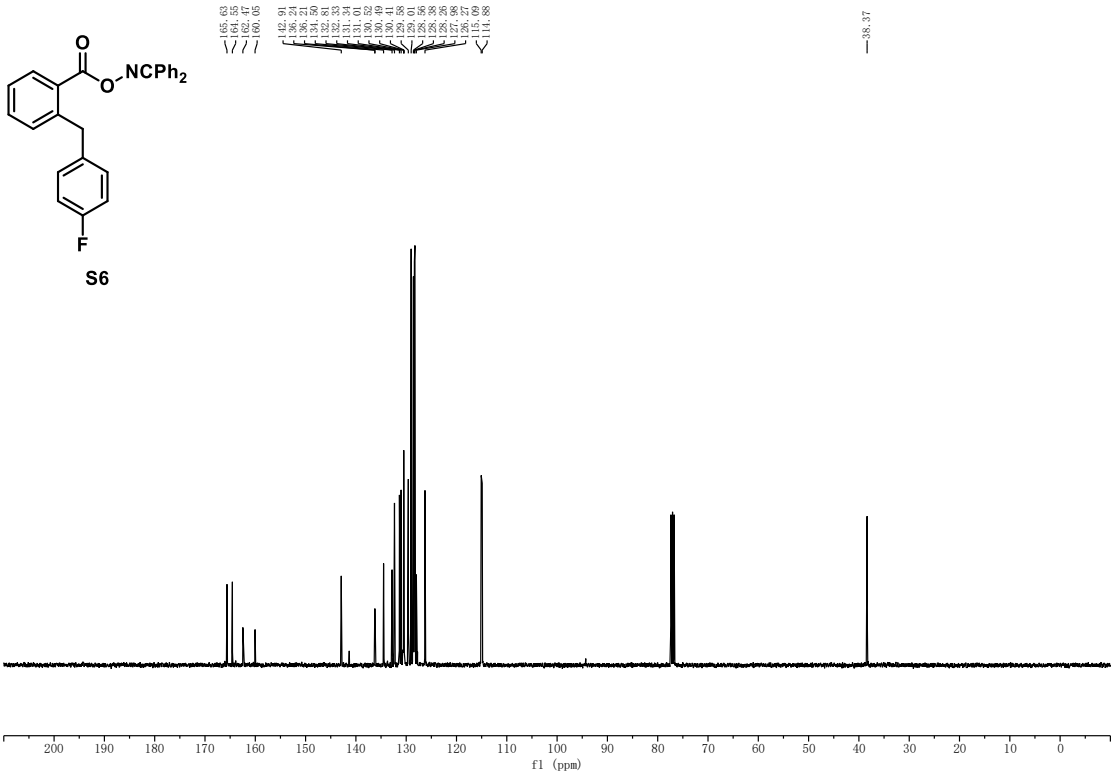

**Supplementary Figure 41** $^{1}$H NMR spectrum of **S5**



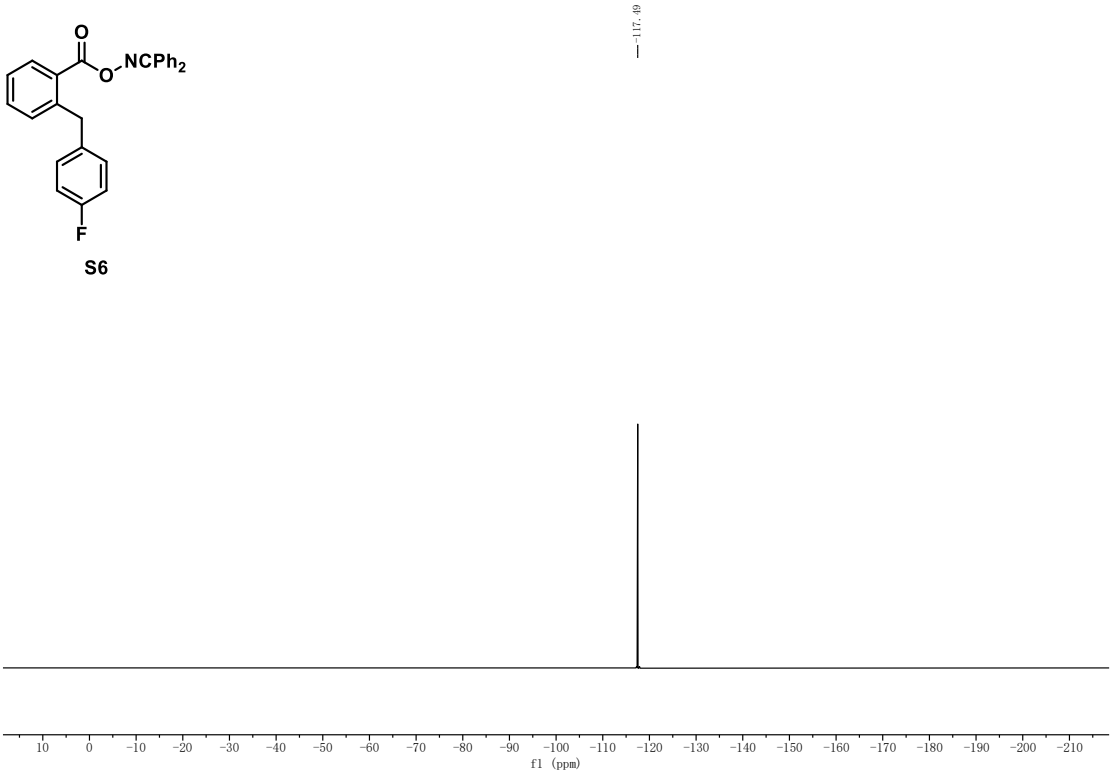

**Supplementary Figure 42** $^{13}$C NMR spectrum of **S5**



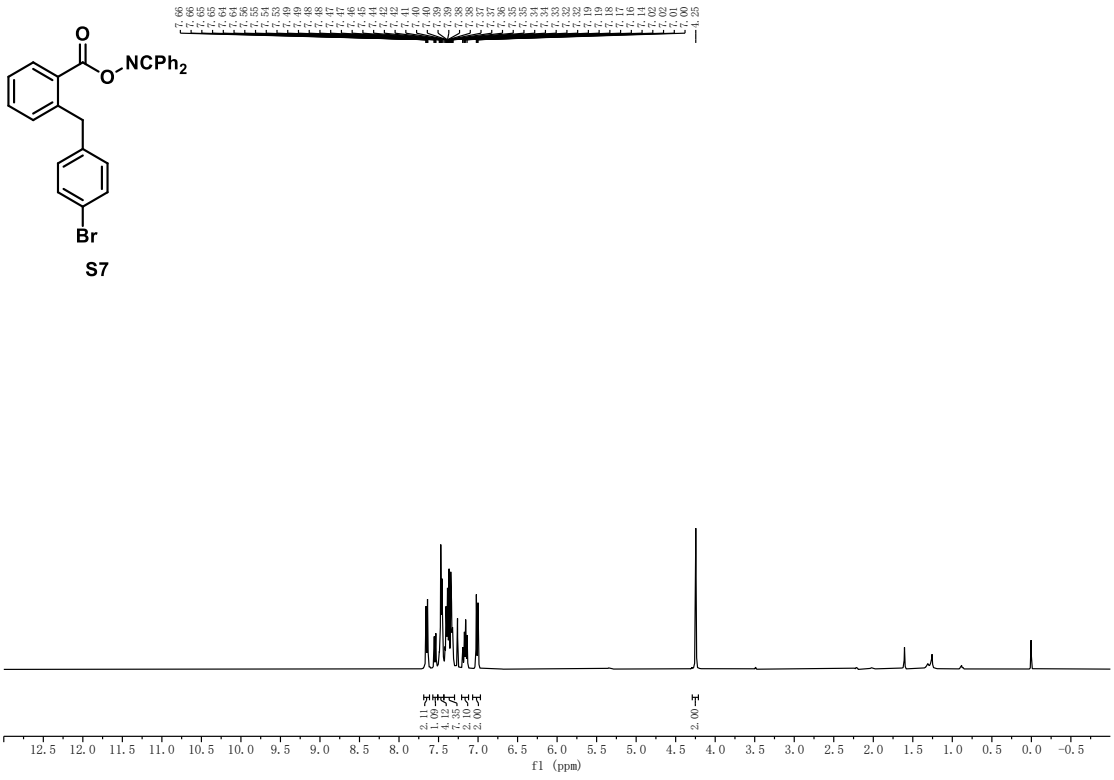

**Supplementary Figure 43** $^{1}$H NMR spectrum of **S6**



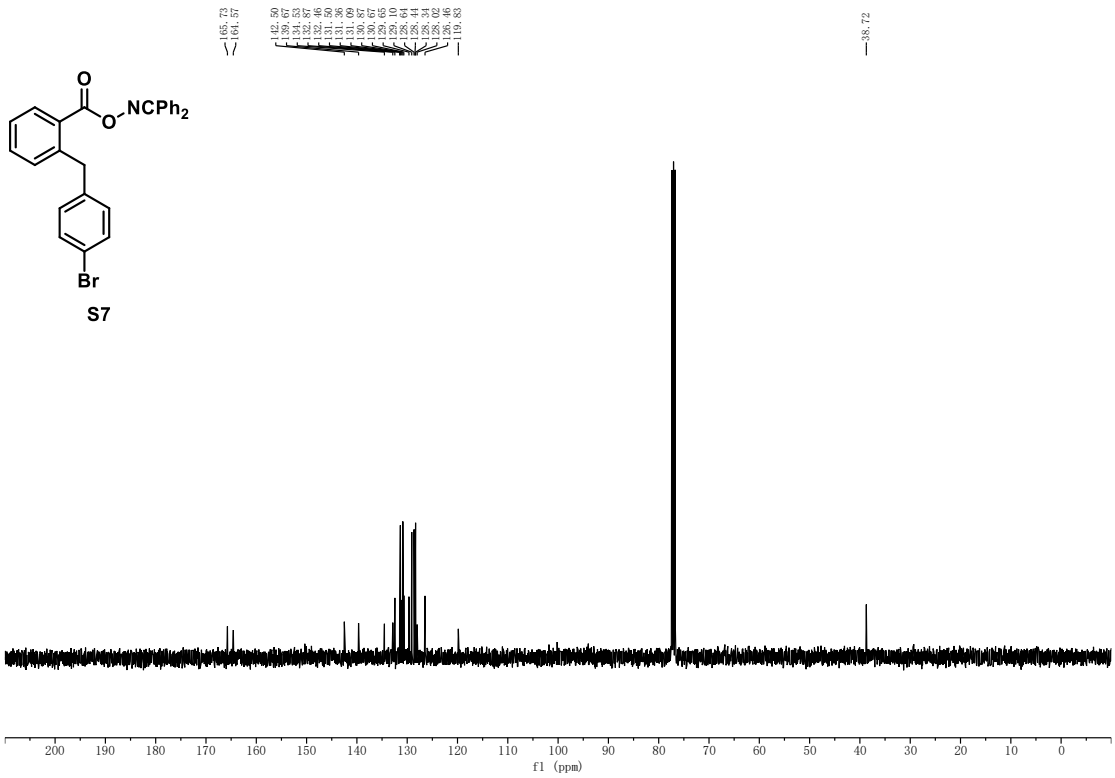

**Supplementary Figure 44** $^{13}$C NMR spectrum of **S6**



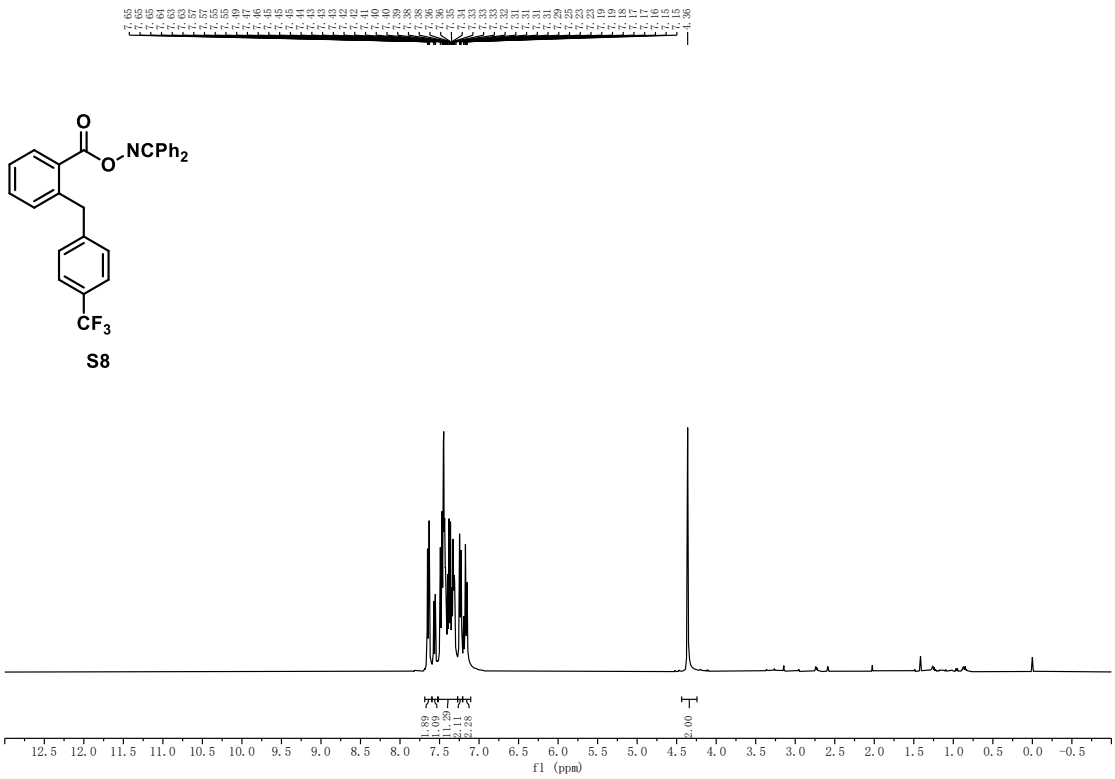

**Supplementary Figure 45** $^{19}$F NMR spectrum of **S6**



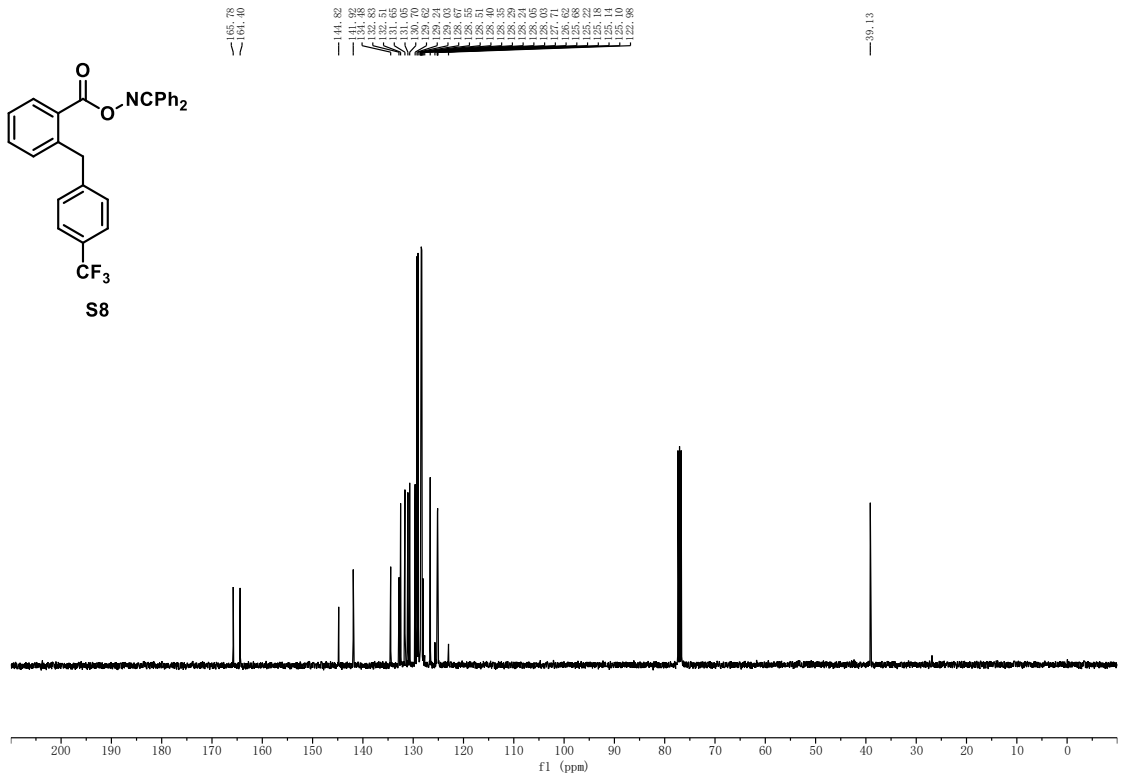

**Supplementary Figure 46** $^{1}$H NMR spectrum of **S7**



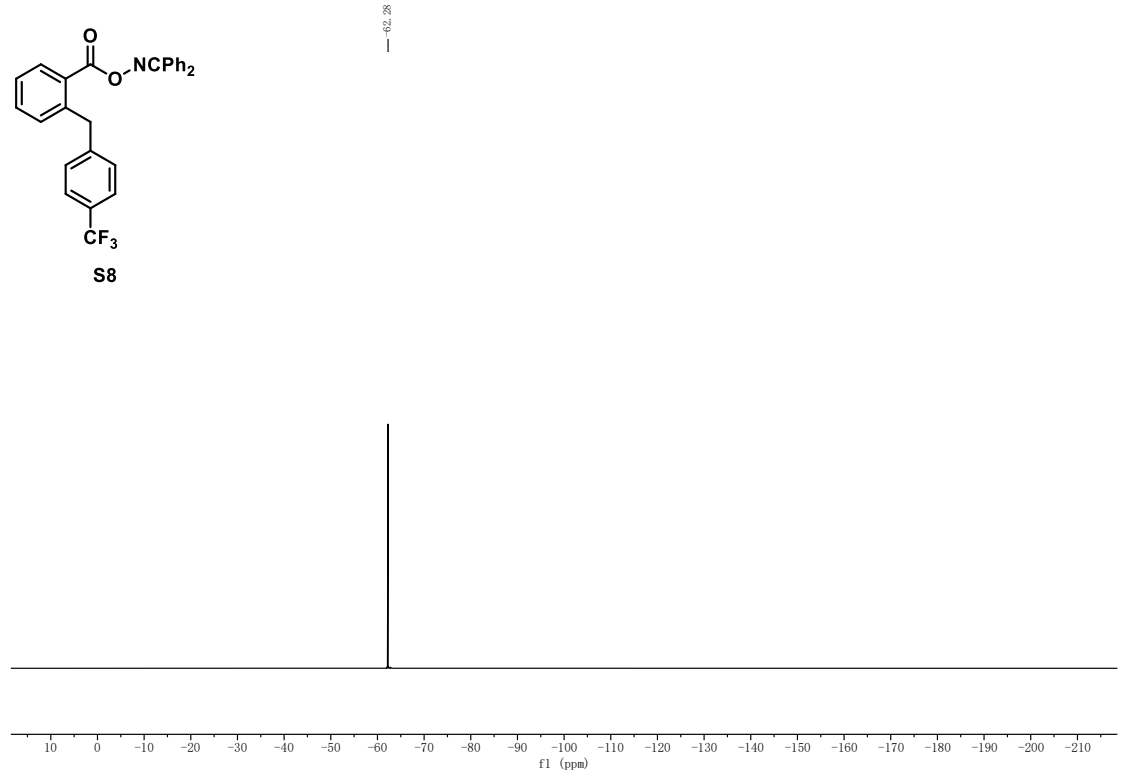

**Supplementary Figure 47** $^{13}$C NMR spectrum of **S7**



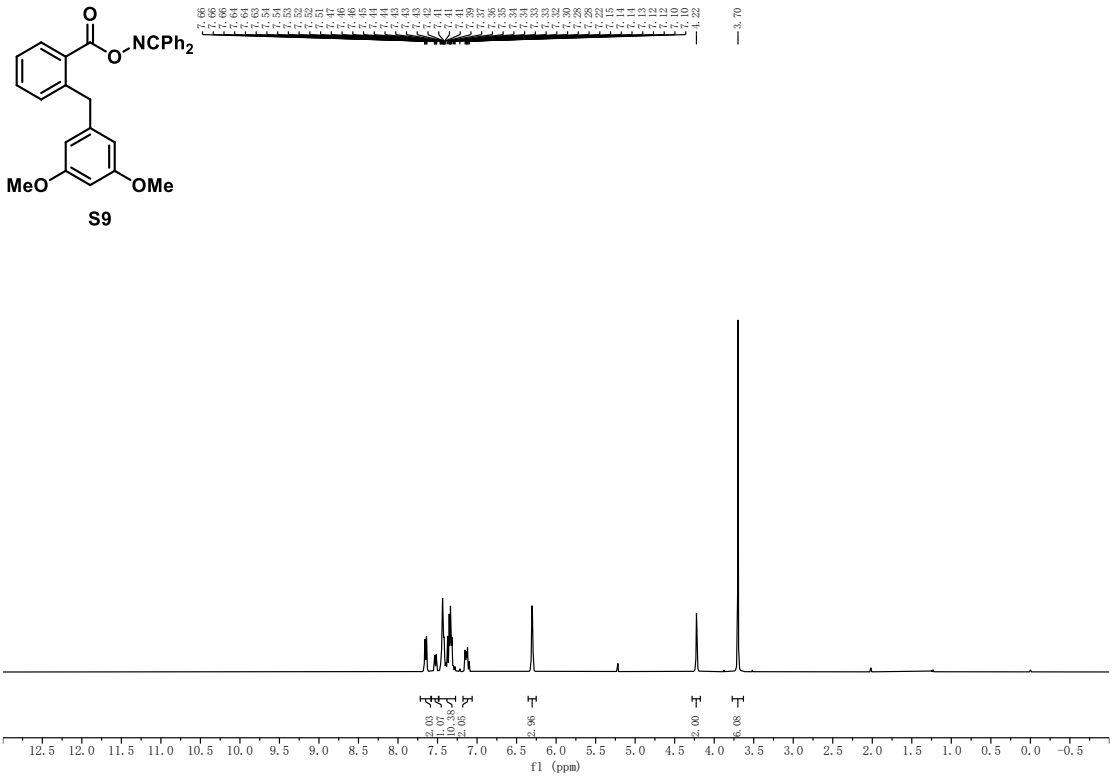

**Supplementary Figure 48** $^{1}$H NMR spectrum of **S8**



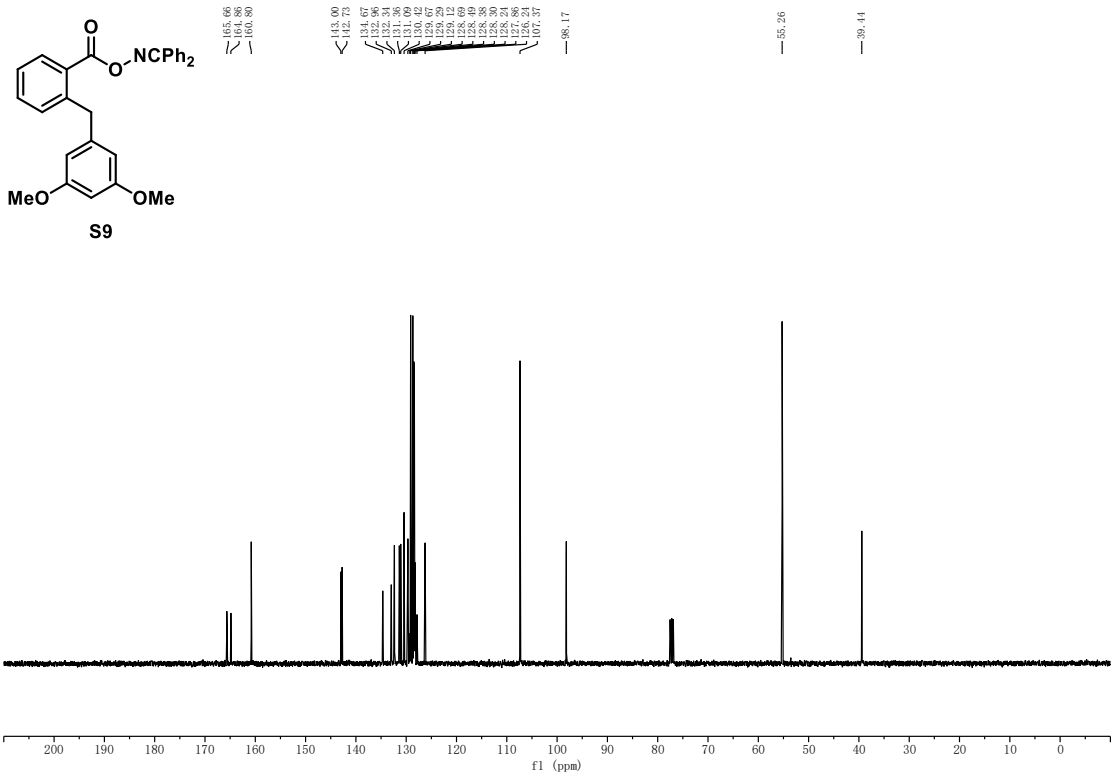

**Supplementary Figure 49** $^{13}$C NMR spectrum of **S8**



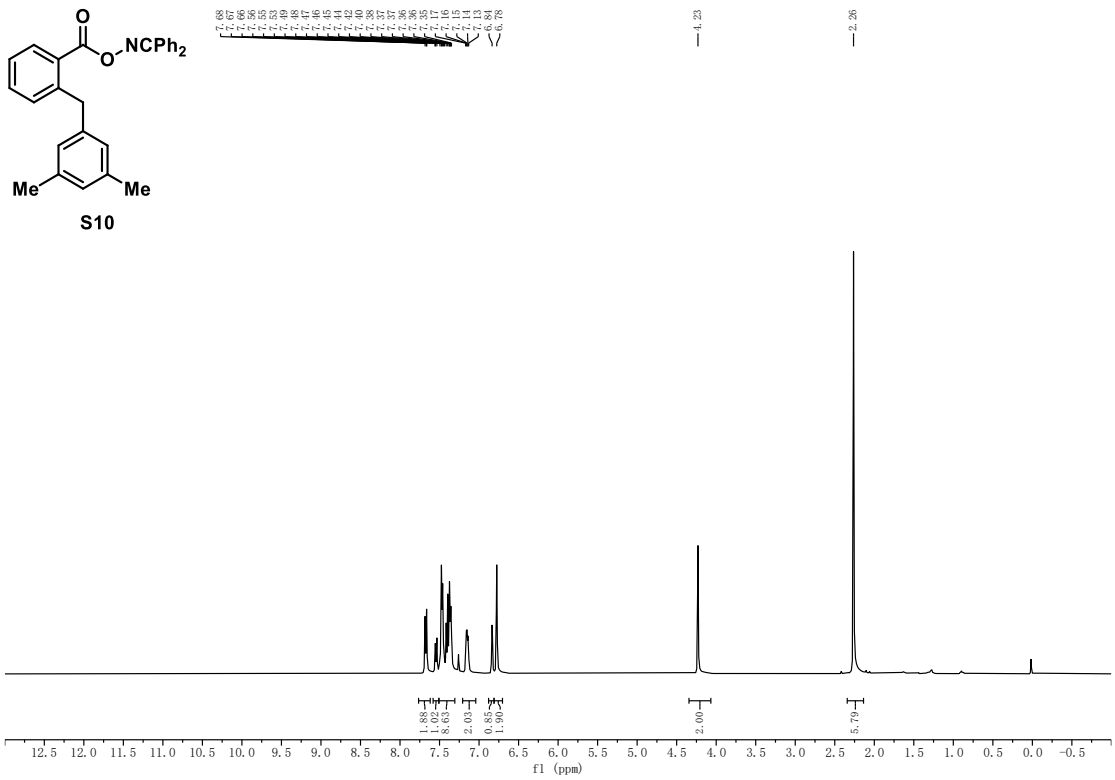

**Supplementary Figure 50** $^{19}$F NMR spectrum of **S8**



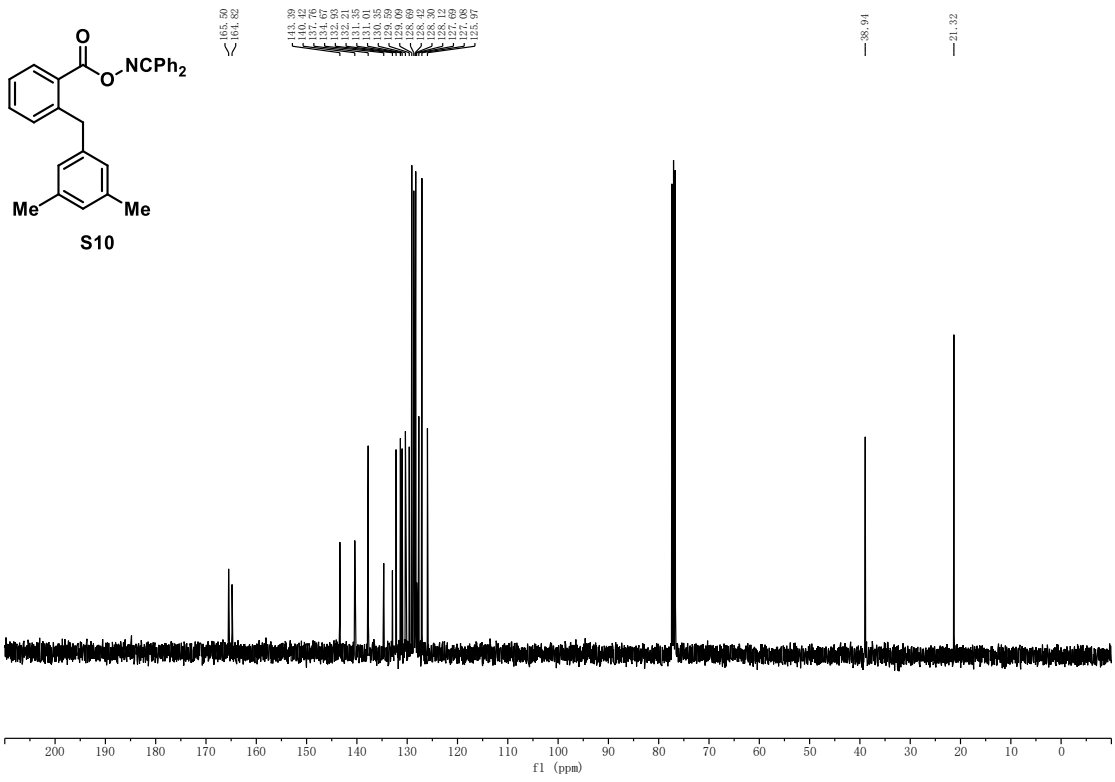

**Supplementary Figure 51** $^{1}$H NMR spectrum of **S9**



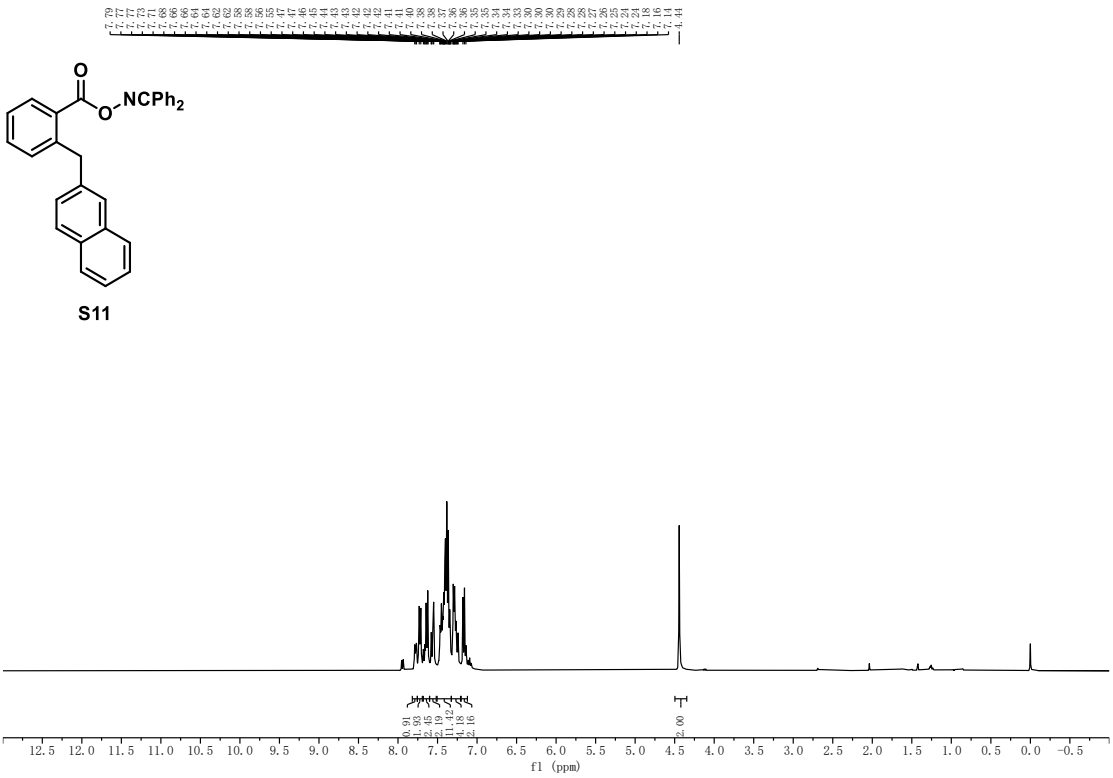

**Supplementary Figure 52** $^{13}$C NMR spectrum of **S9**



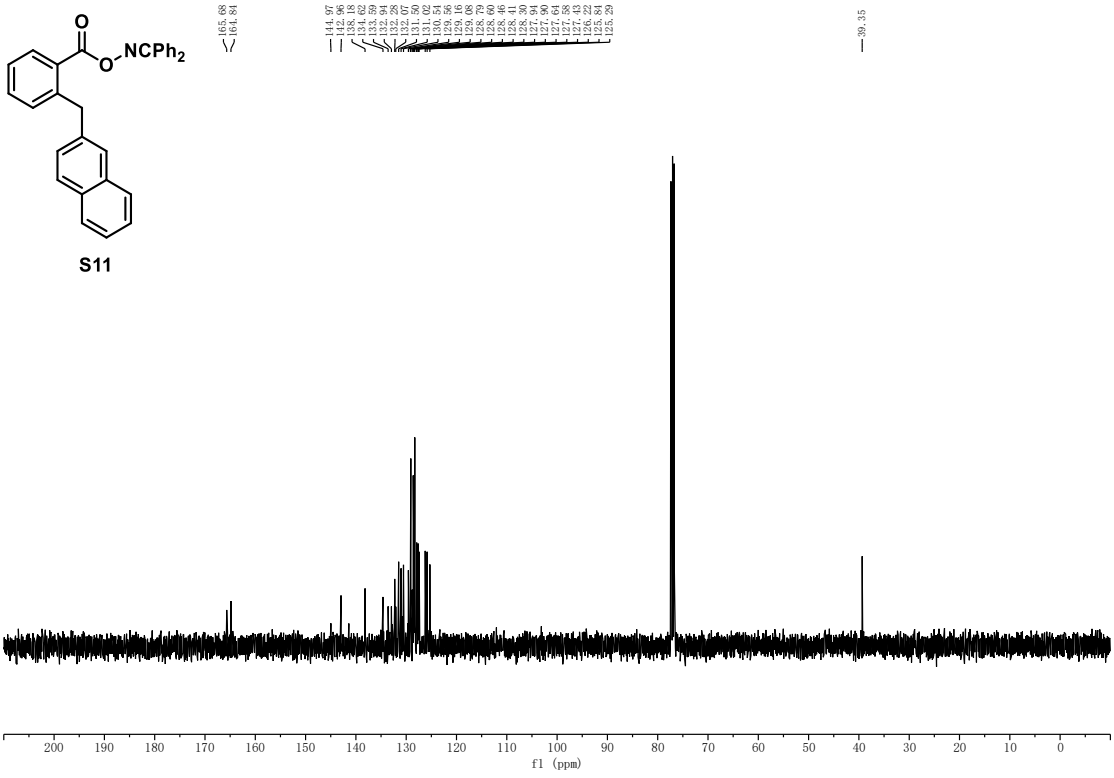

**Supplementary Figure 53** $^{1}$H NMR spectrum of **S10**



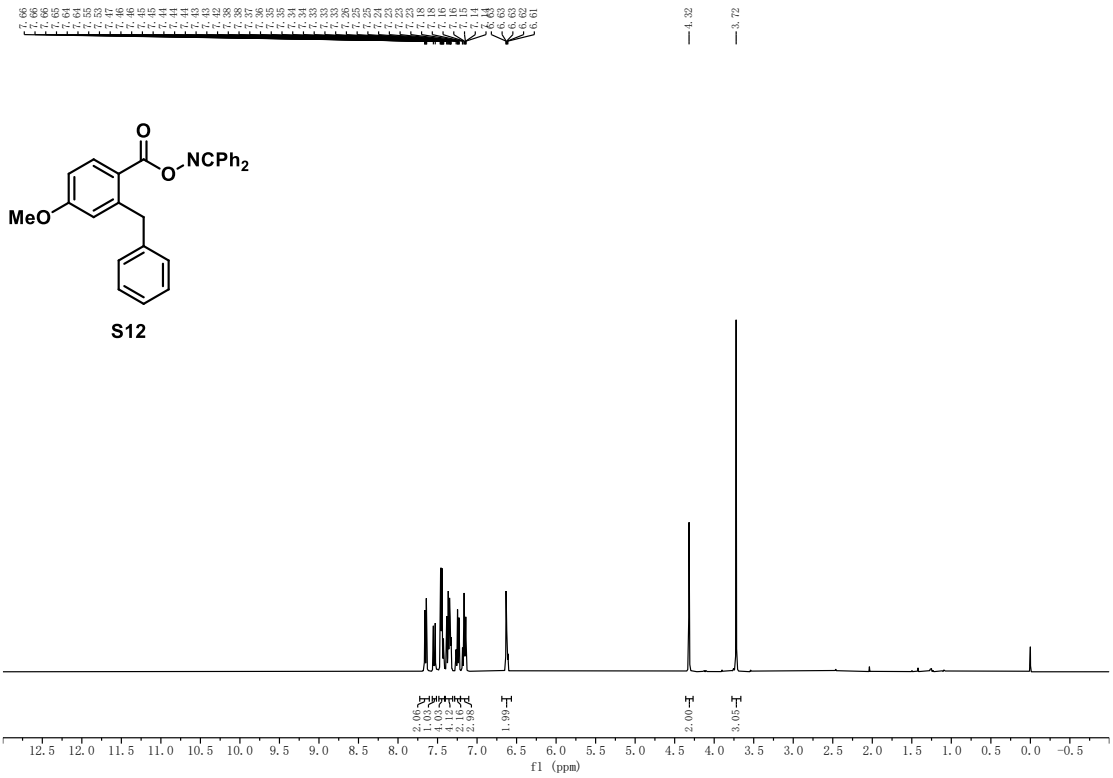

**Supplementary Figure 54** $^{13}$C NMR spectrum of **S10**



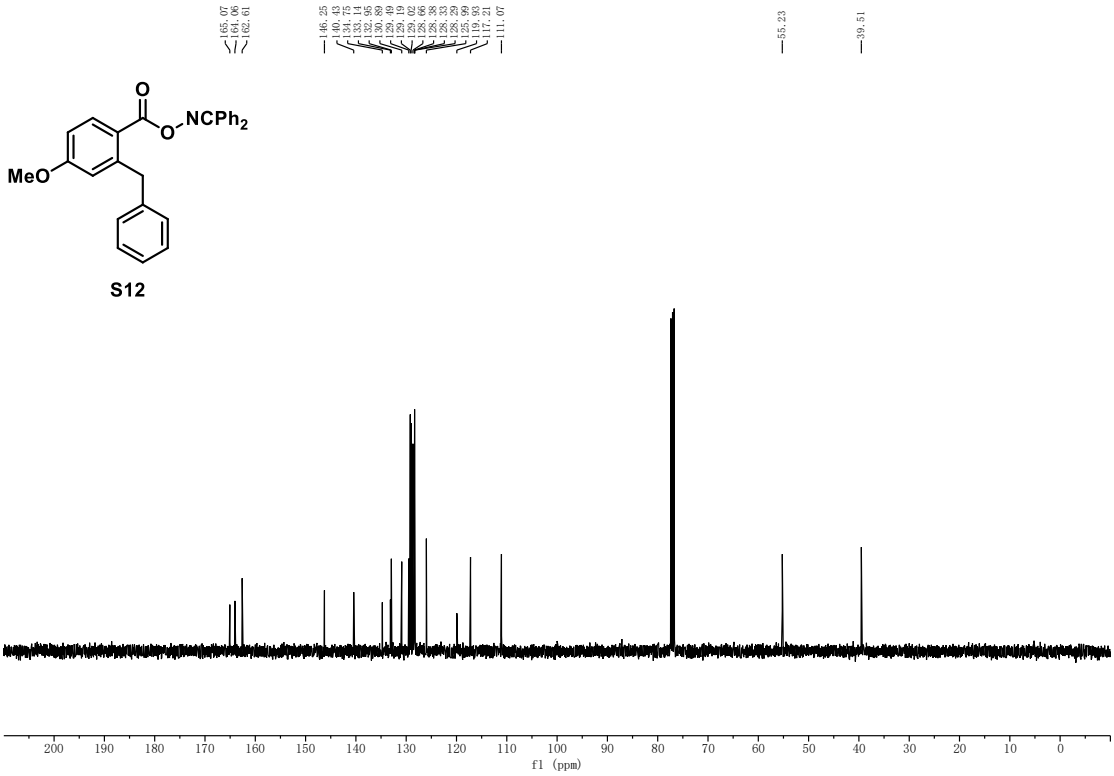

**Supplementary Figure 55** $^{1}$H NMR spectrum of **S11**



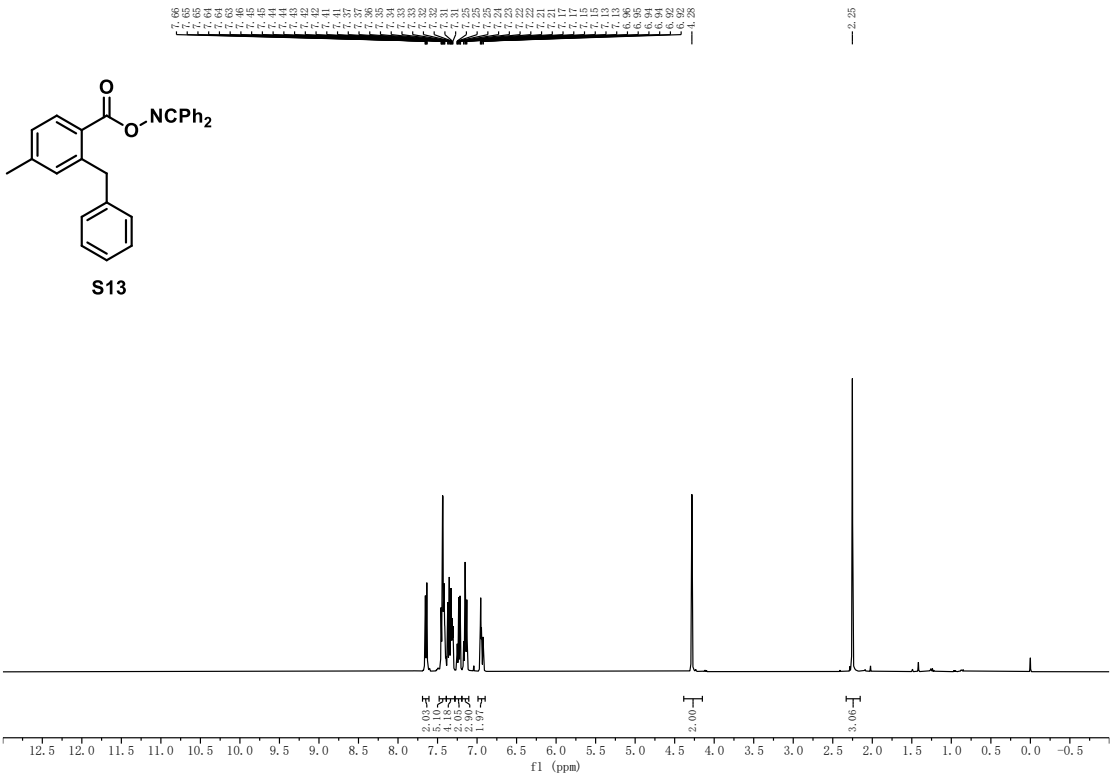

**Supplementary Figure 56** $^{13}$C NMR spectrum of **S11**



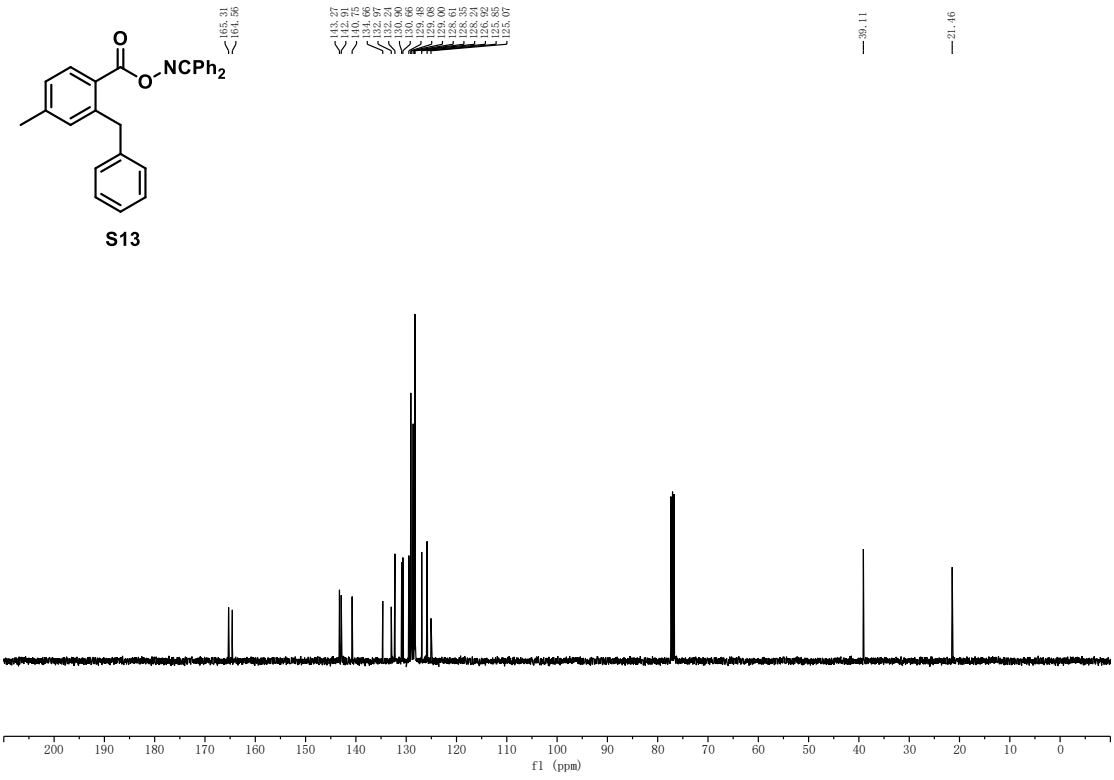

**Supplementary Figure 57** $^{1}$H NMR spectrum of **S12**



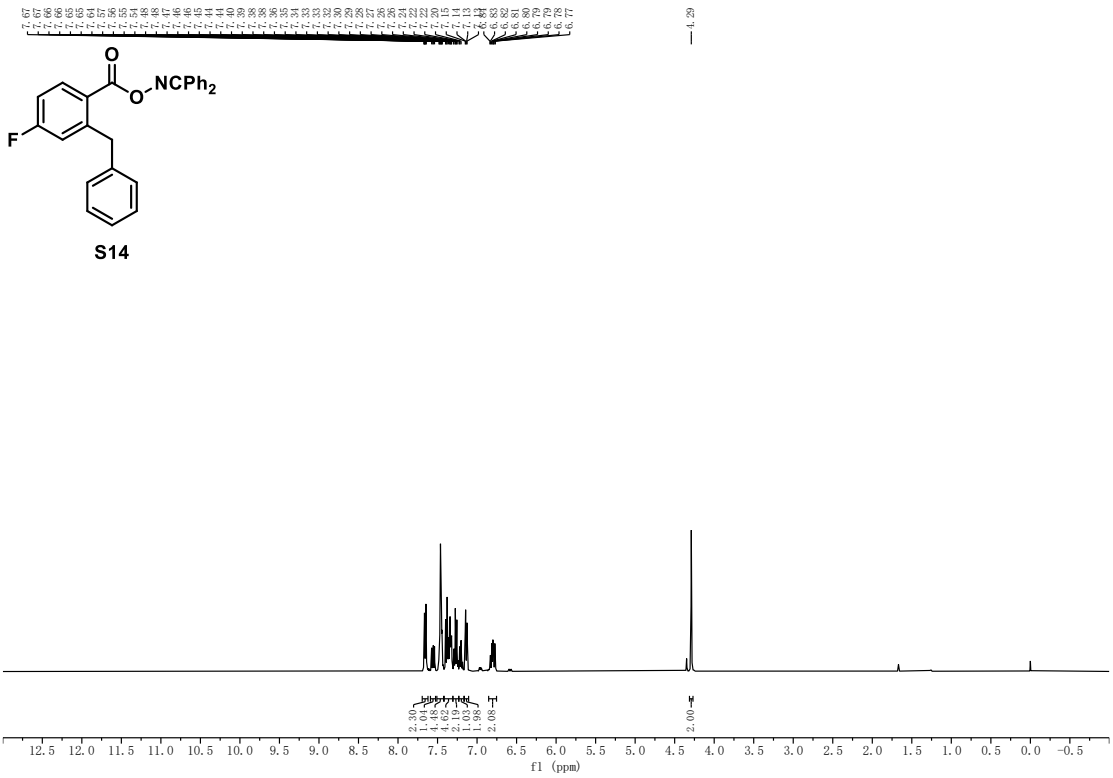

**Supplementary Figure 58** $^{13}$C NMR spectrum of **S12**



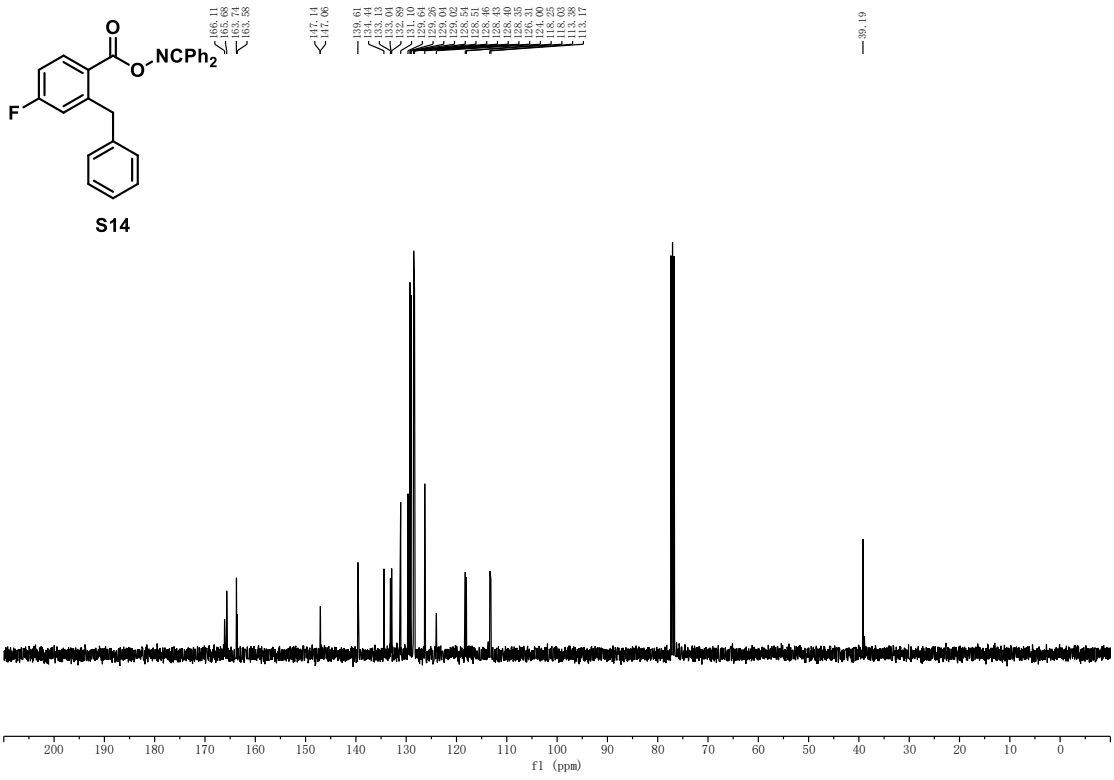

**Supplementary Figure 59** $^{1}$H NMR spectrum of **S13**



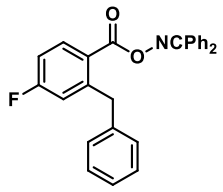

**Supplementary Figure 60** $^{13}$C NMR spectrum of **S13**




**Supplementary Figure 61** $^{1}$H NMR spectrum of **S14**



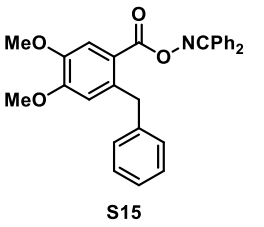

**Supplementary Figure 62** $^{13}$C NMR spectrum of **S14**




S14



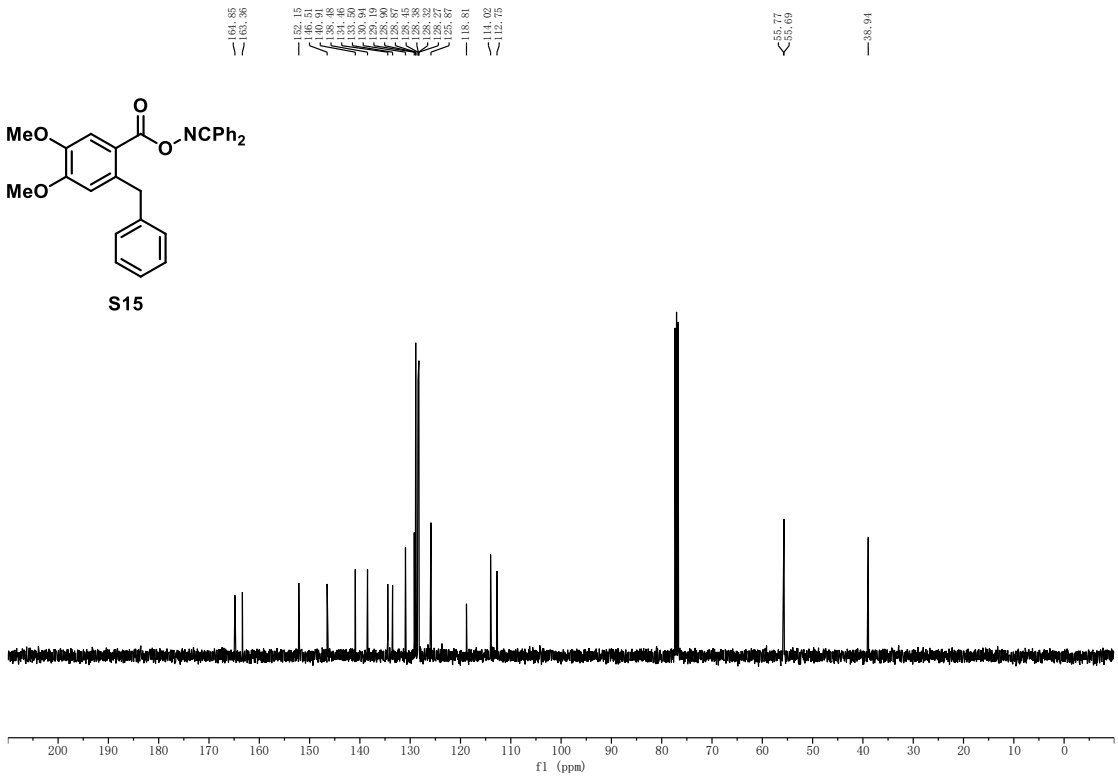

**Supplementary Figure 63** $^{19}$F NMR spectrum of **S14**



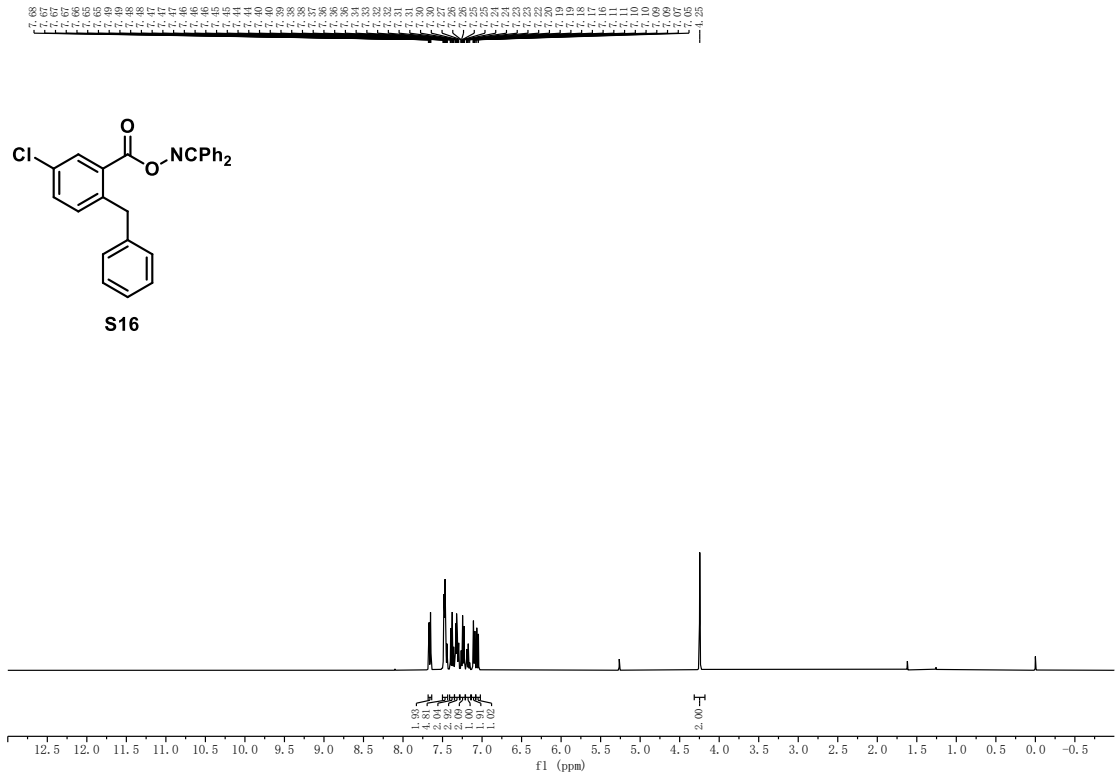

S15



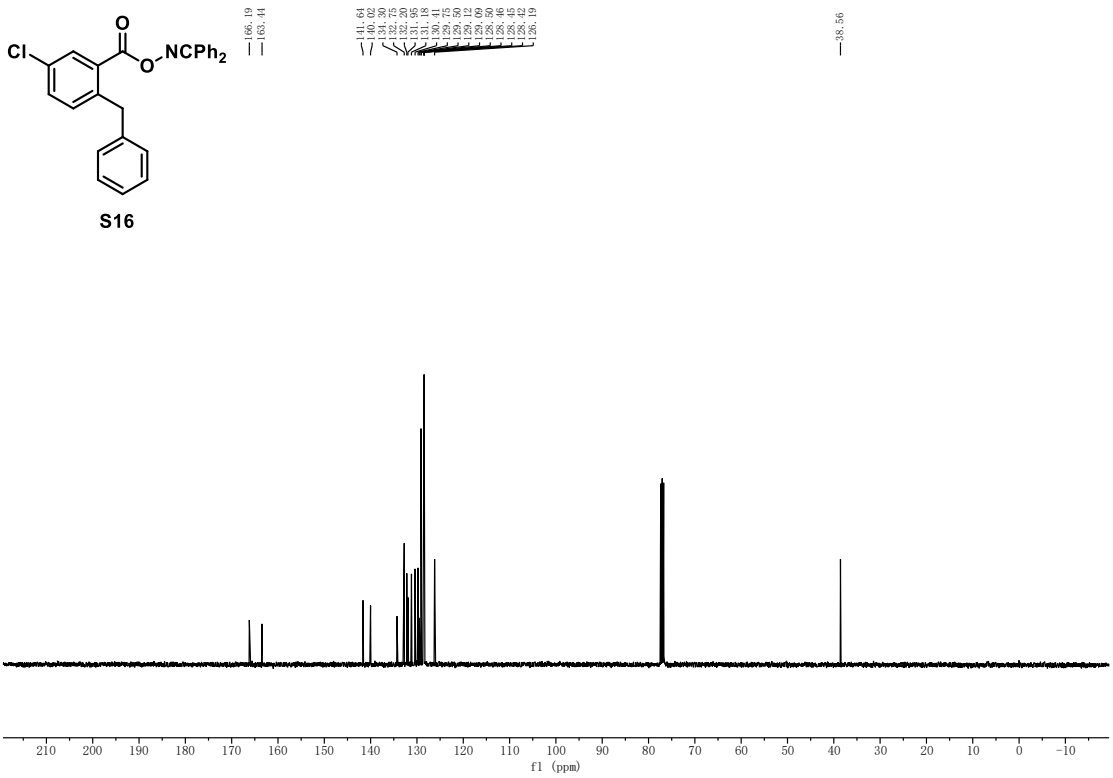

**Supplementary Figure 64** $^{1}$H NMR spectrum of **S15**



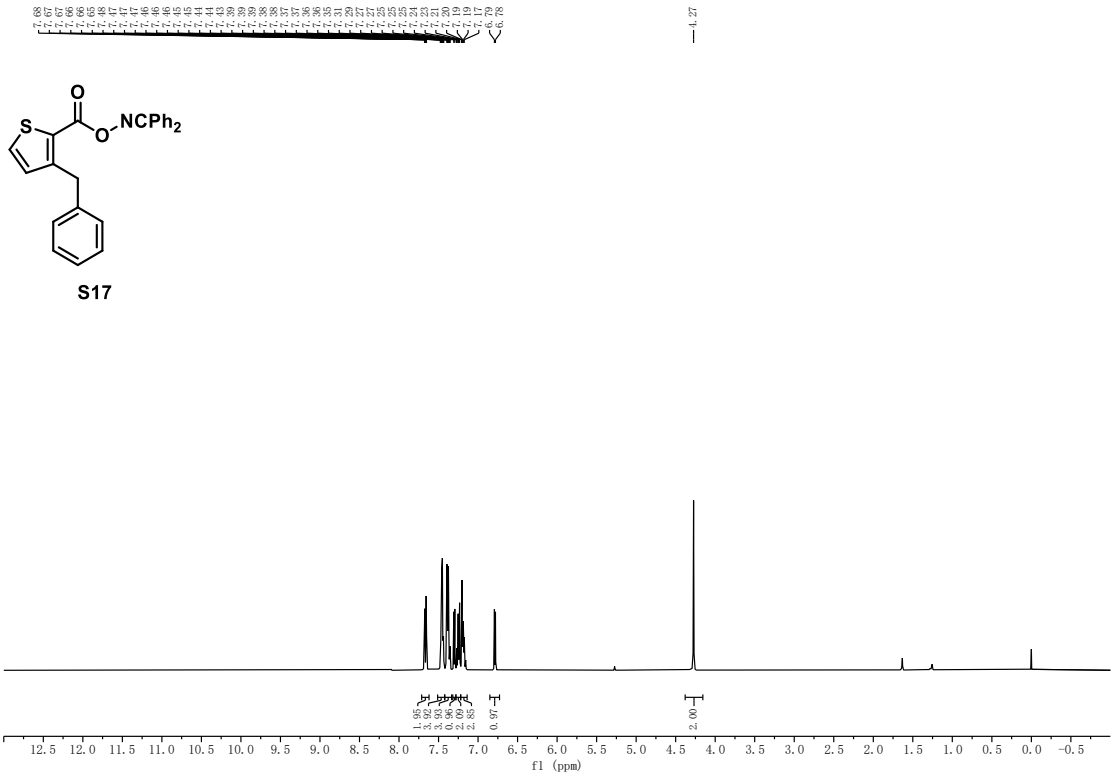

**Supplementary Figure 65** $^{13}$C NMR spectrum of **S15**



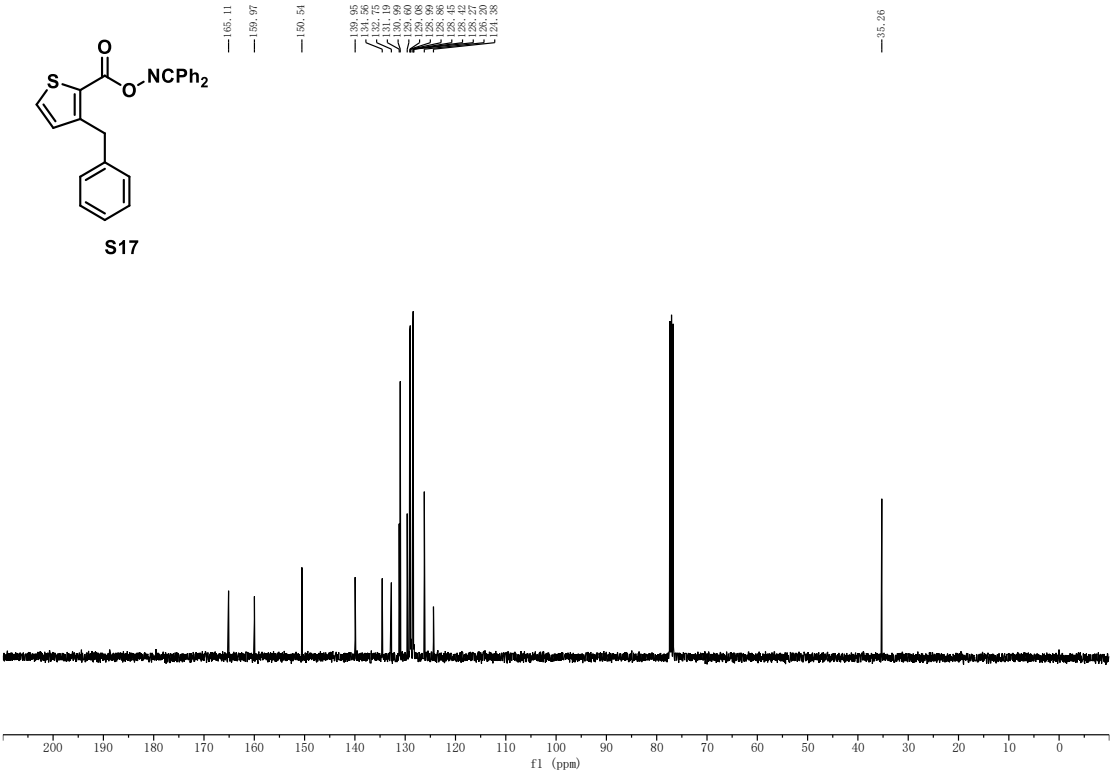

**Supplementary Figure 66** $^{1}$H NMR spectrum of **S16**



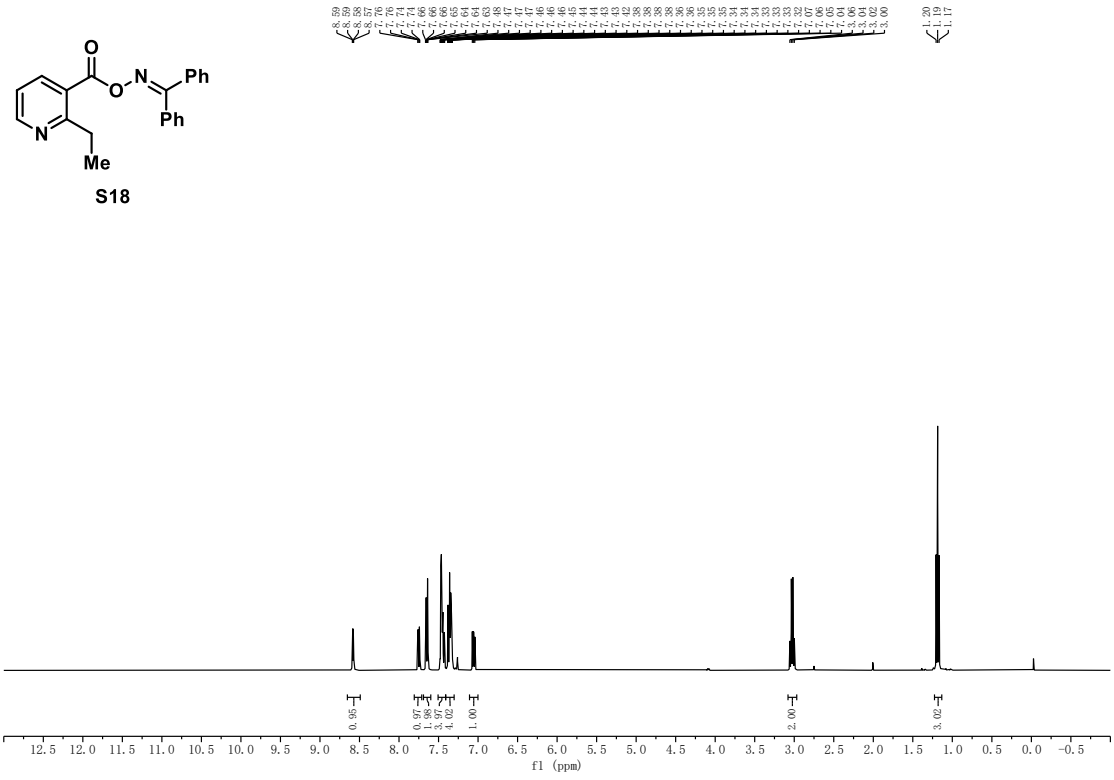

**Supplementary Figure 67** $^{13}$C NMR spectrum of **S16**



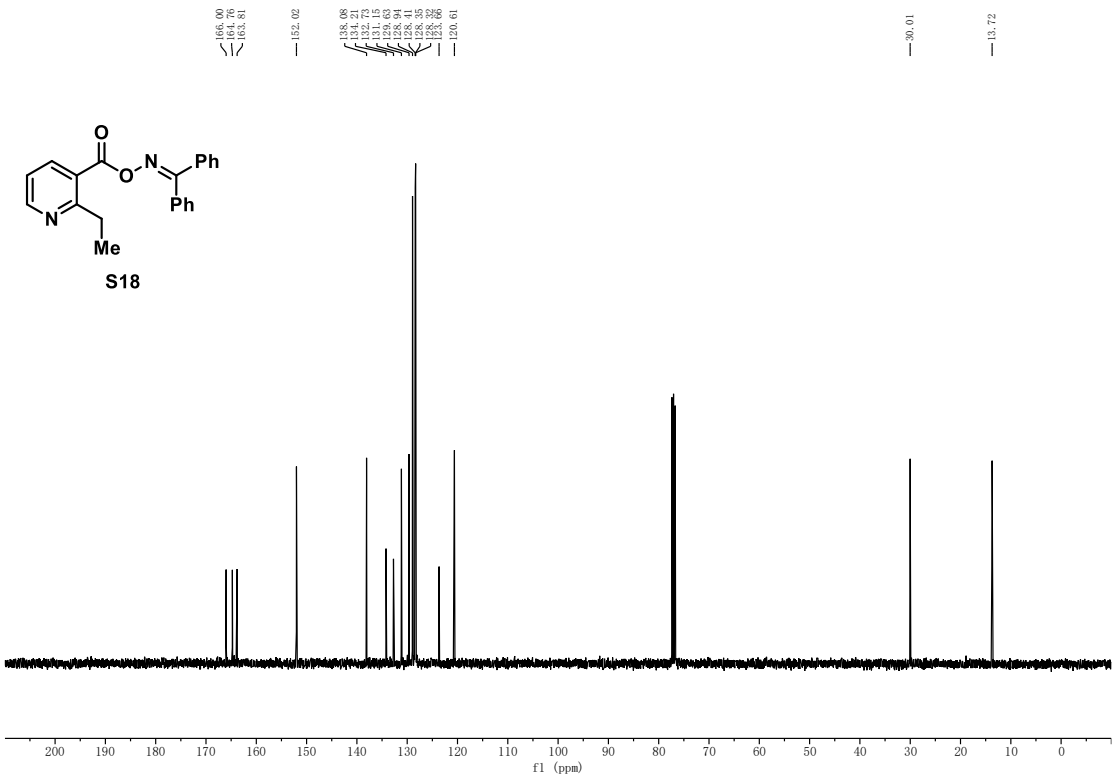

**Supplementary Figure 68** $^{1}$H NMR spectrum of **S17**



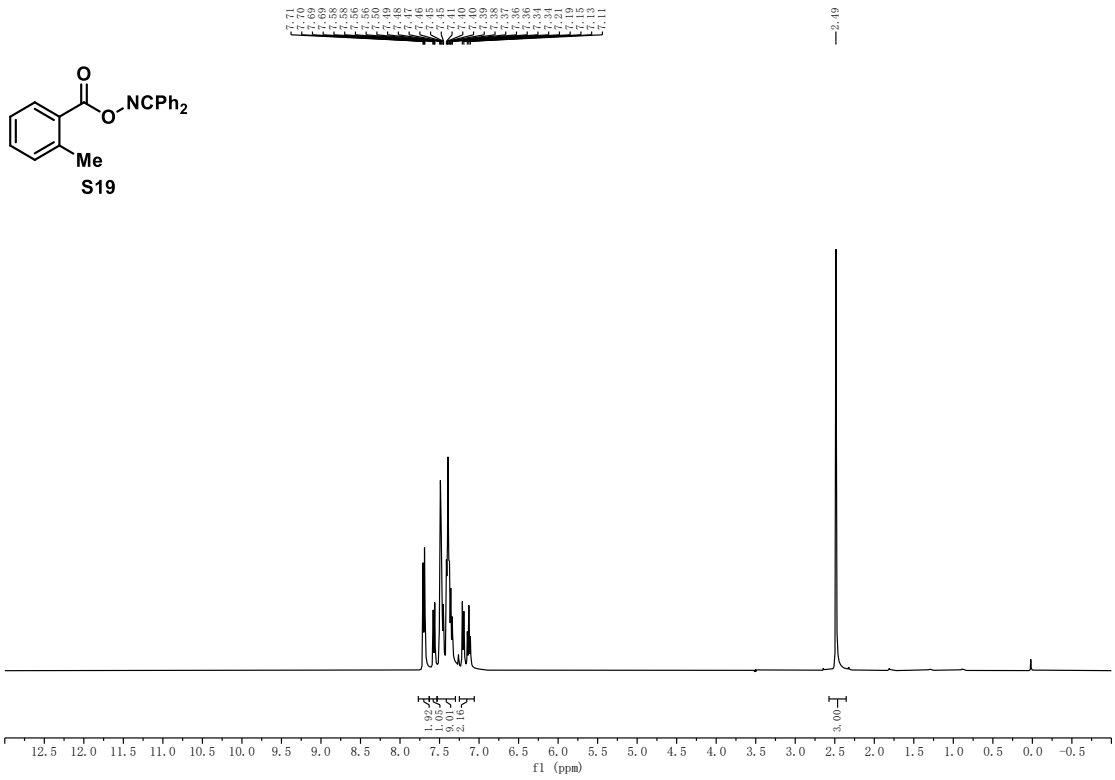

**Supplementary Figure 69** $^{13}$C NMR spectrum of **S17**



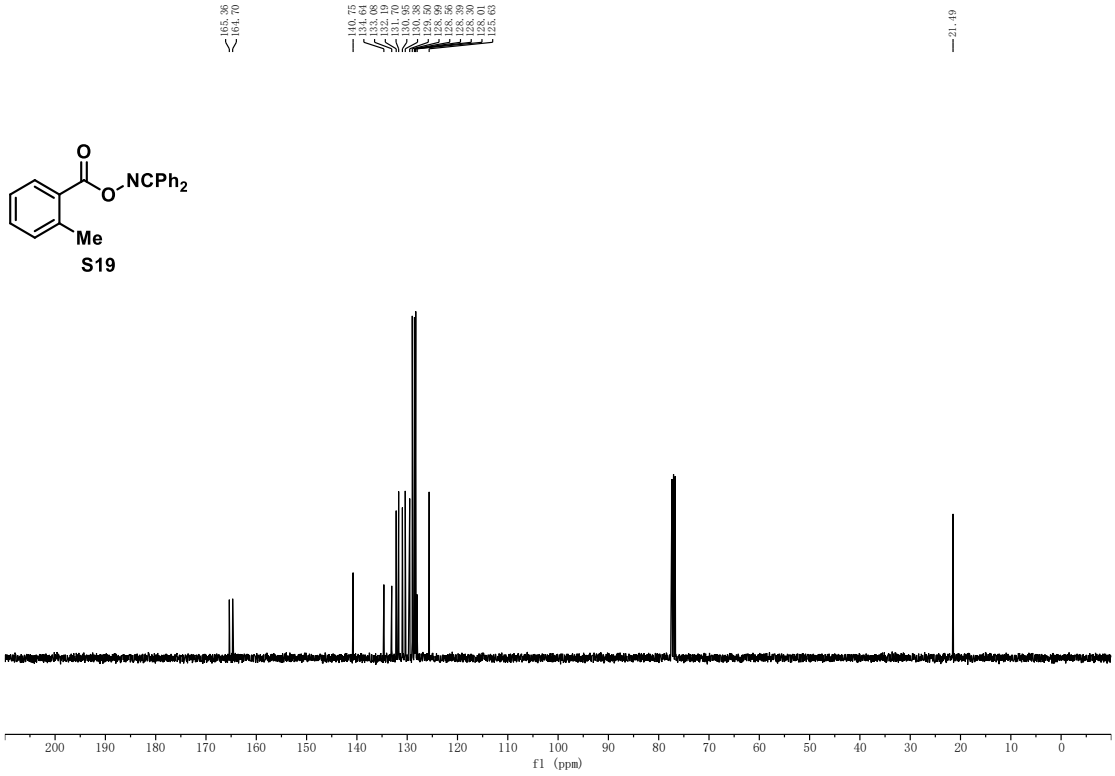

**Supplementary Figure 70** $^{1}$H NMR spectrum of **S18**



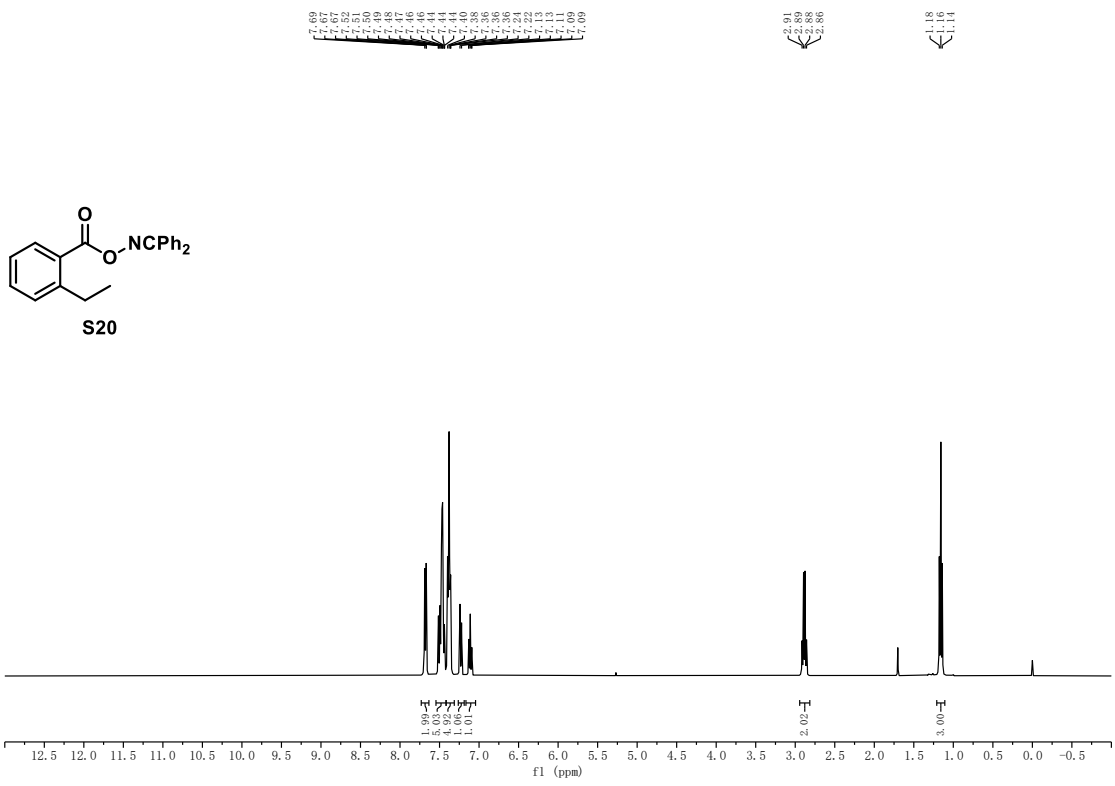

**Supplementary Figure 71** $^{13}$C NMR spectrum of **S18**



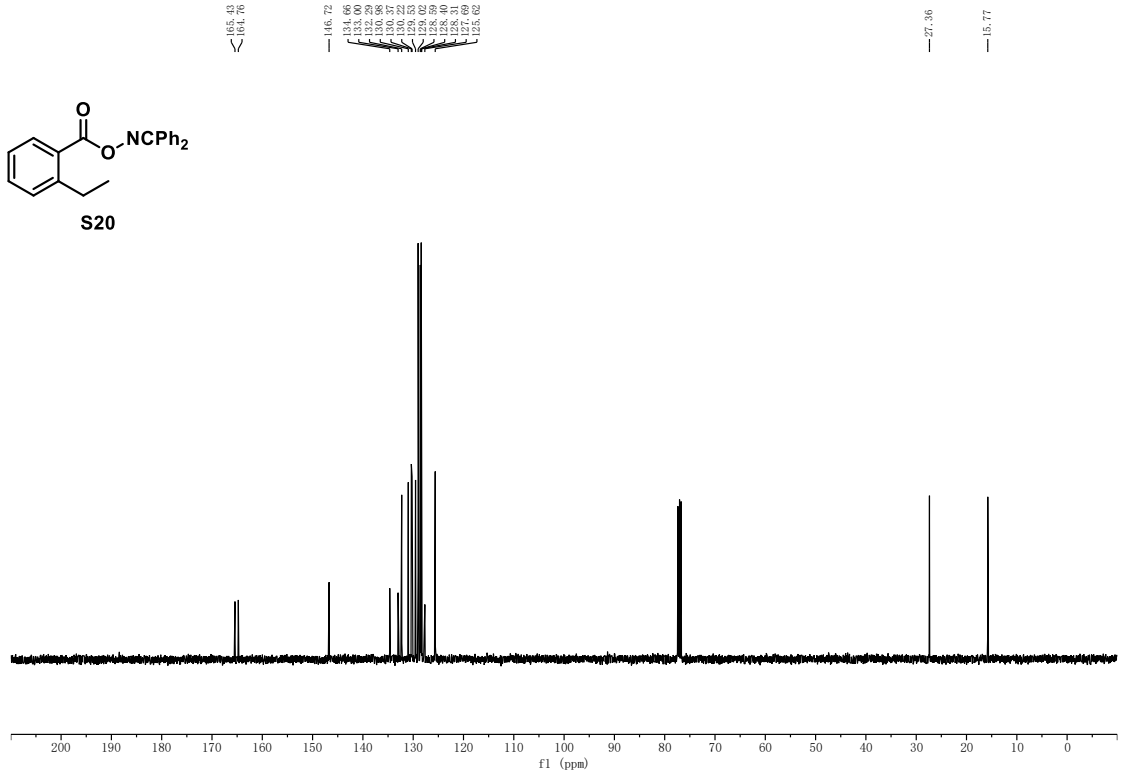

**Supplementary Figure 72** $^{1}$H NMR spectrum of **S19**



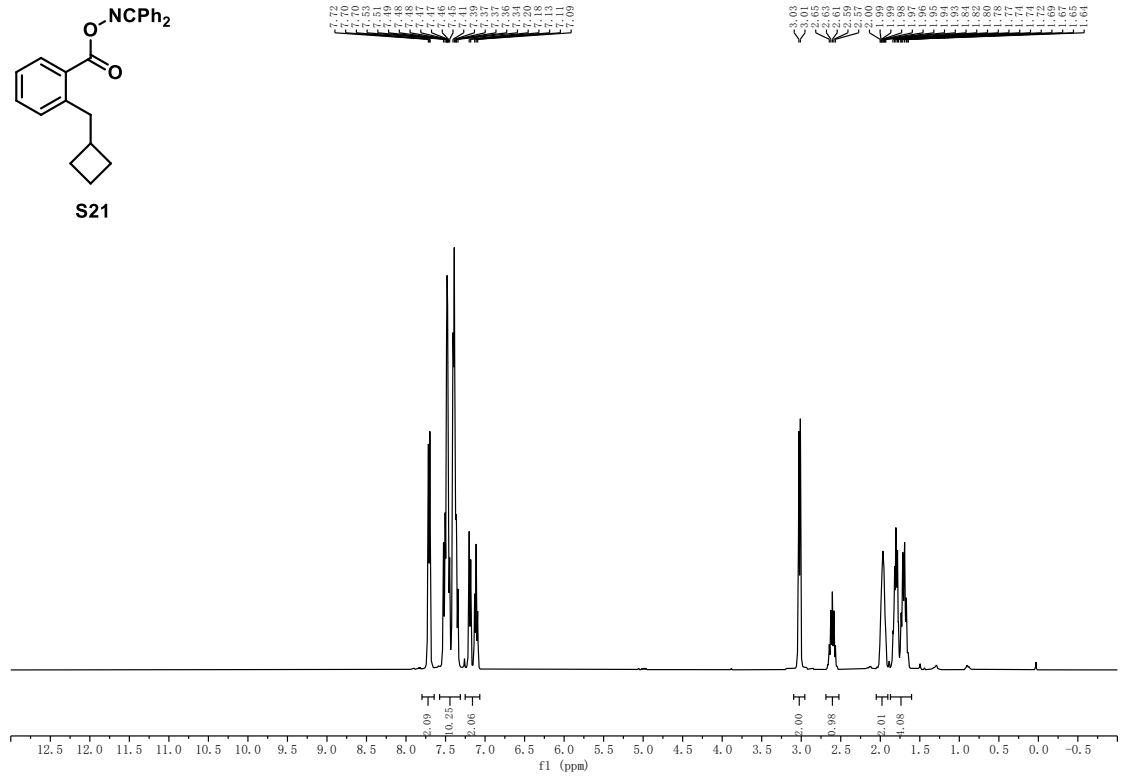

**Supplementary Figure 73** $^{13}$C NMR spectrum of **S19**



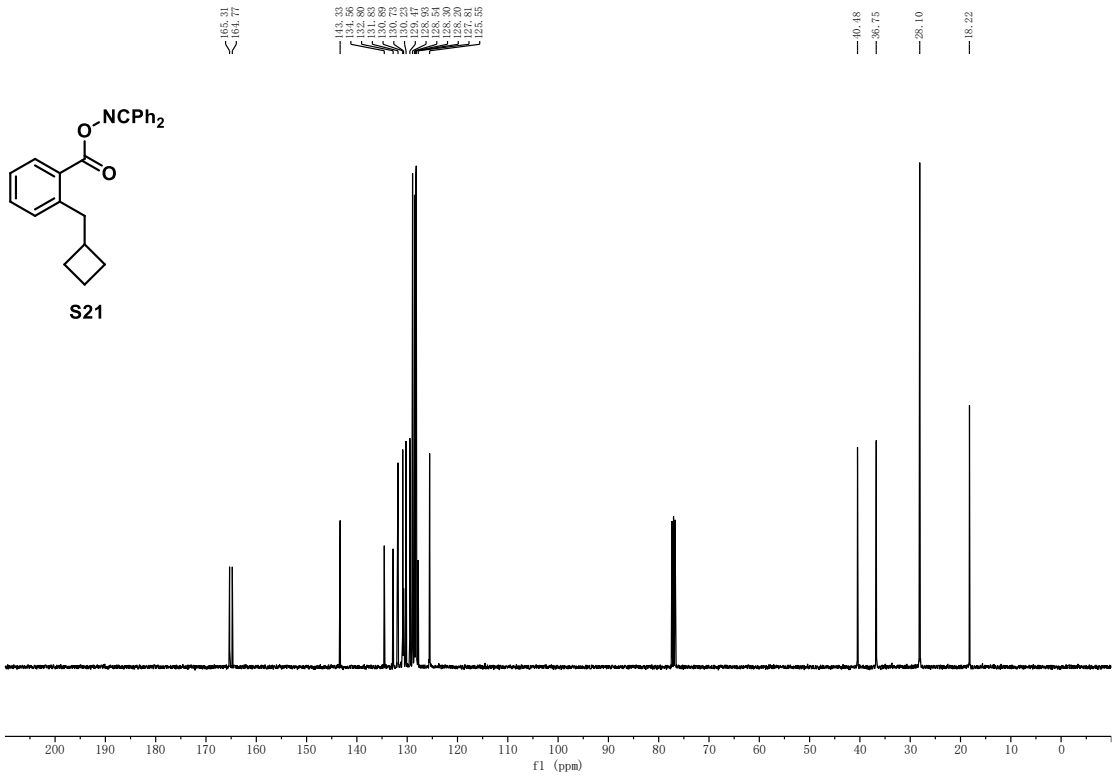

**Supplementary Figure 74** $^{1}$H NMR spectrum of **S20**



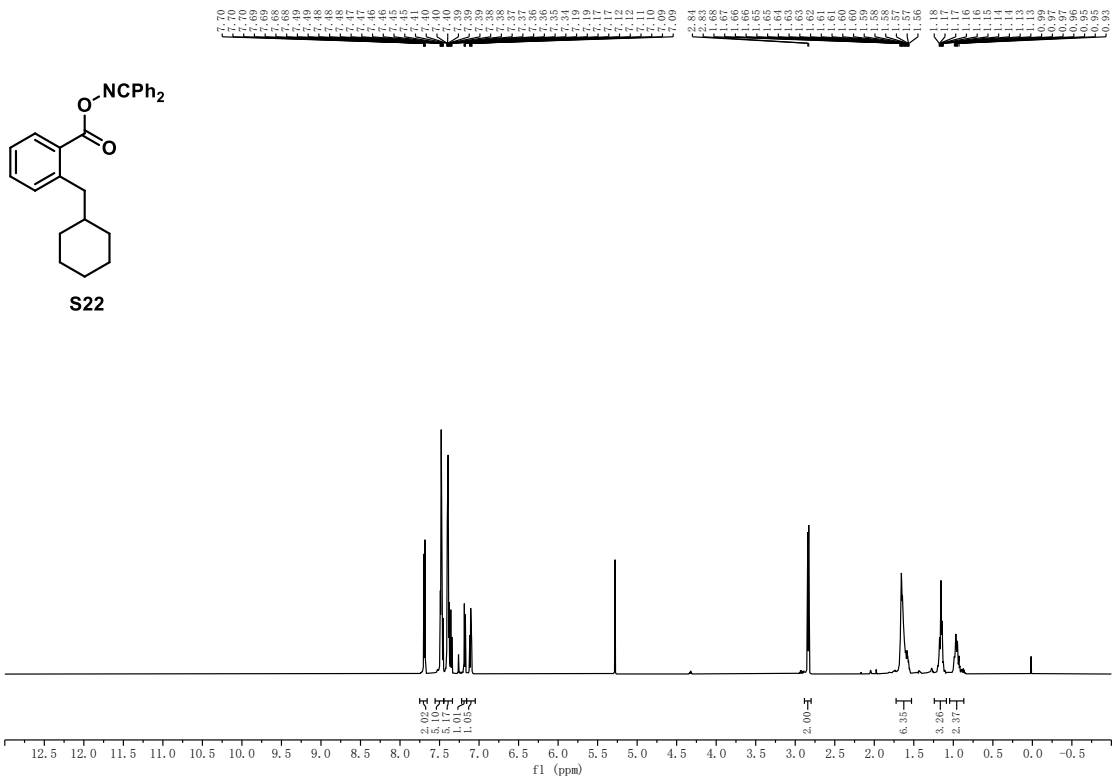

**Supplementary Figure 75** $^{13}$C NMR spectrum of **S20**



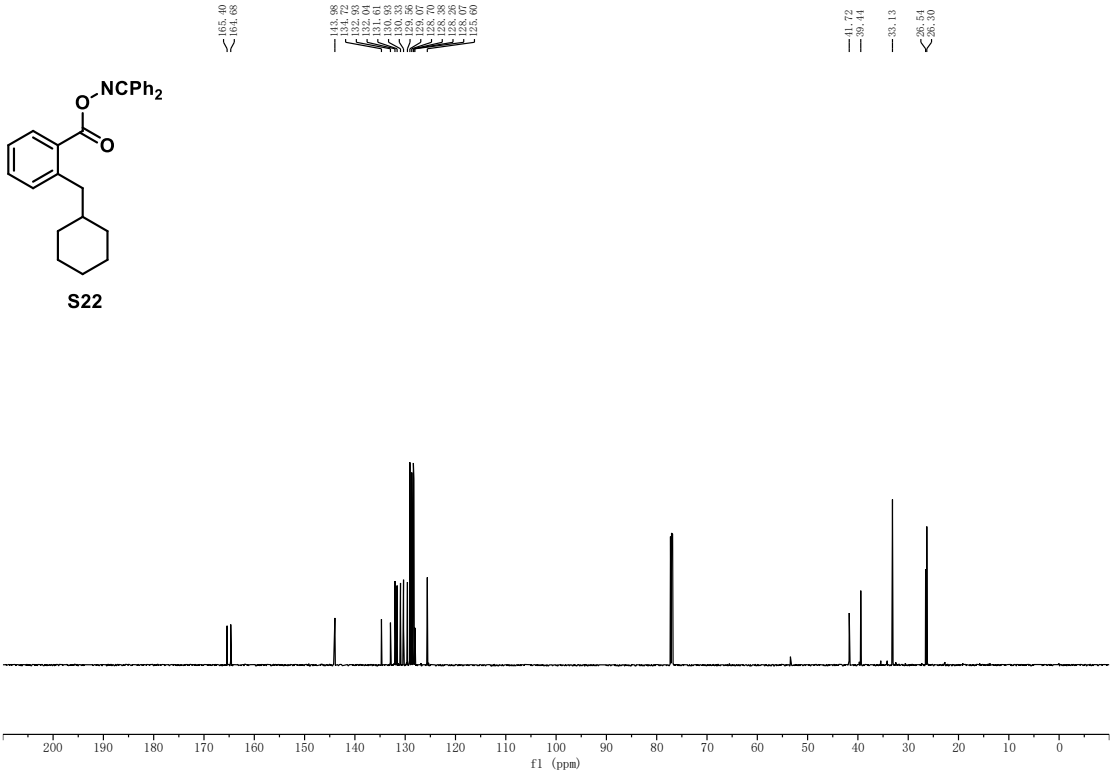

**Supplementary Figure 76** $^{1}$H NMR spectrum of **S21**



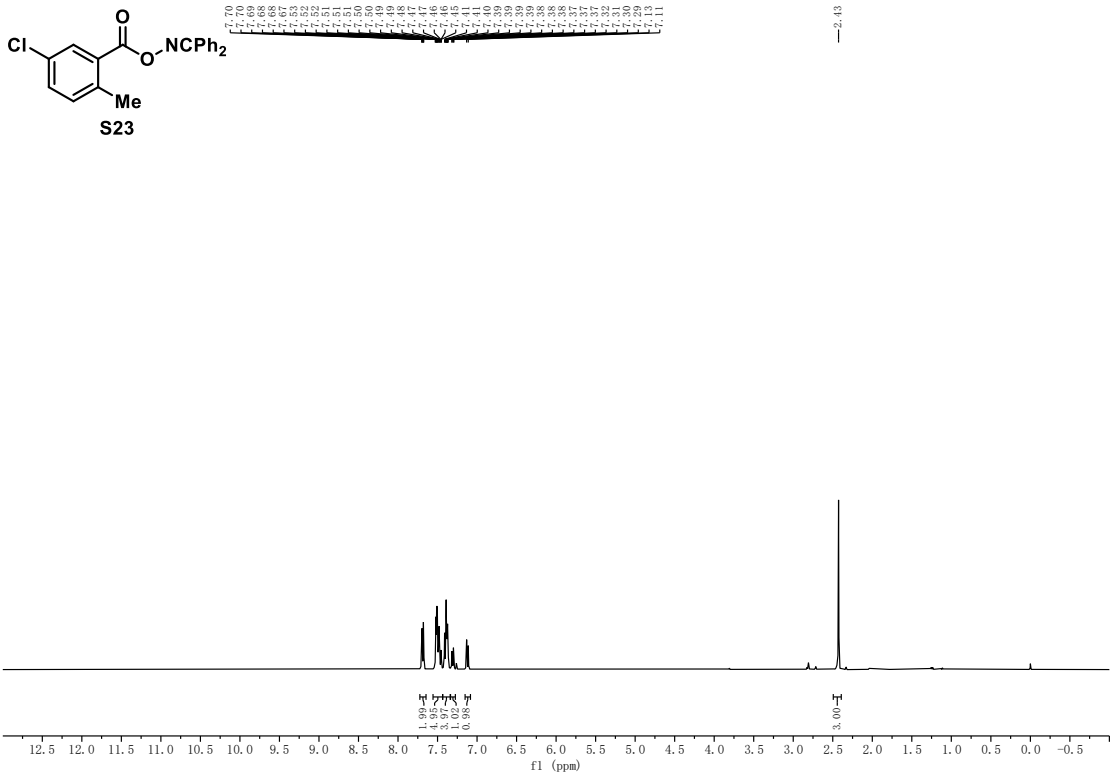

**Supplementary Figure 77** $^{13}$C NMR spectrum of **S21**



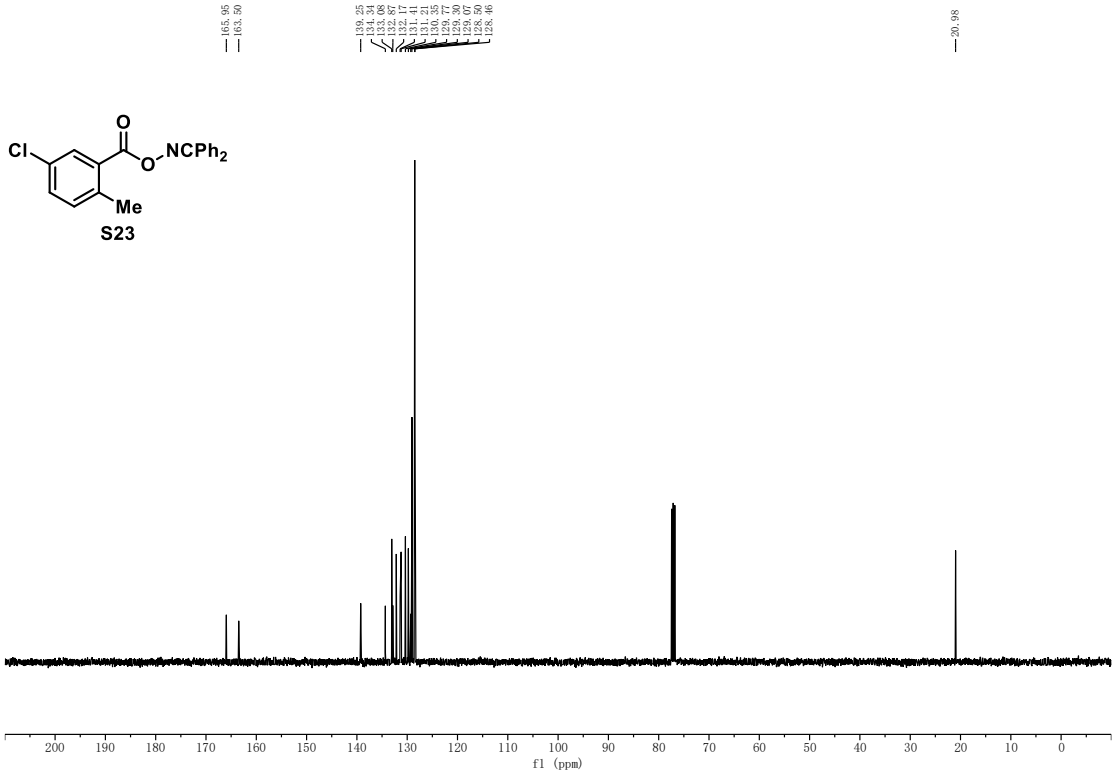

**Supplementary Figure 78** $^{1}$H NMR spectrum of **S22**



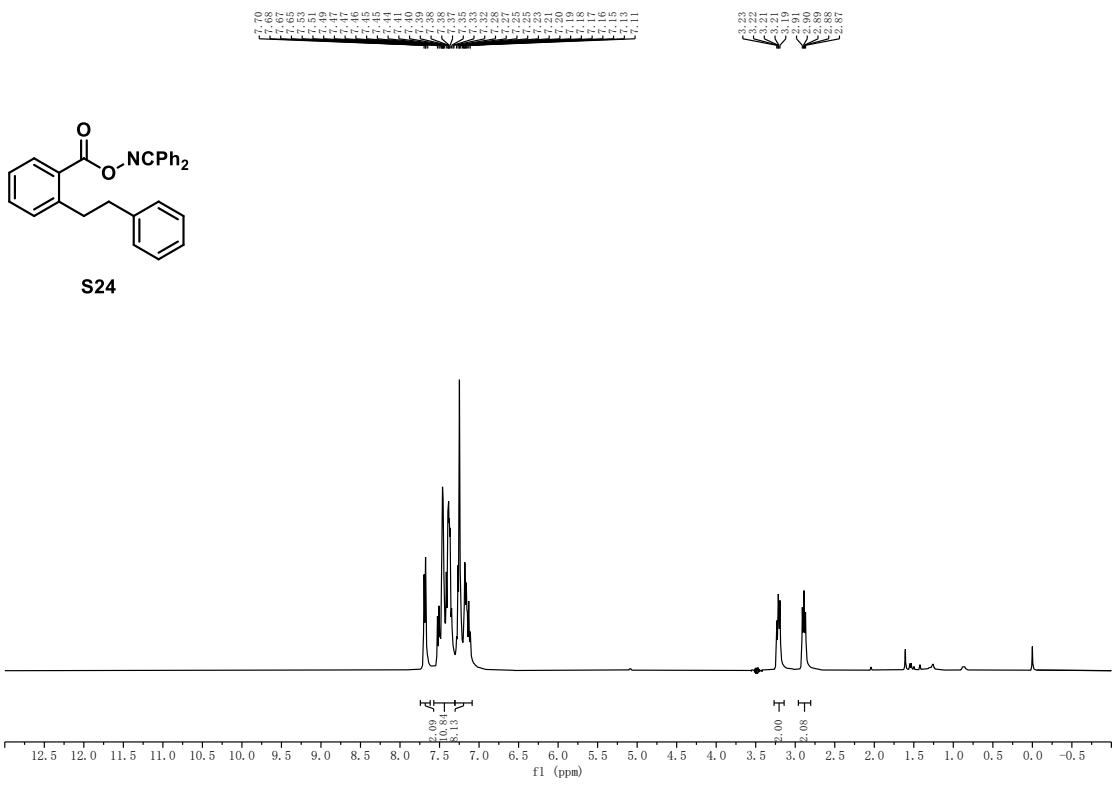

**Supplementary Figure 79** $^{13}$C NMR spectrum of **S22**



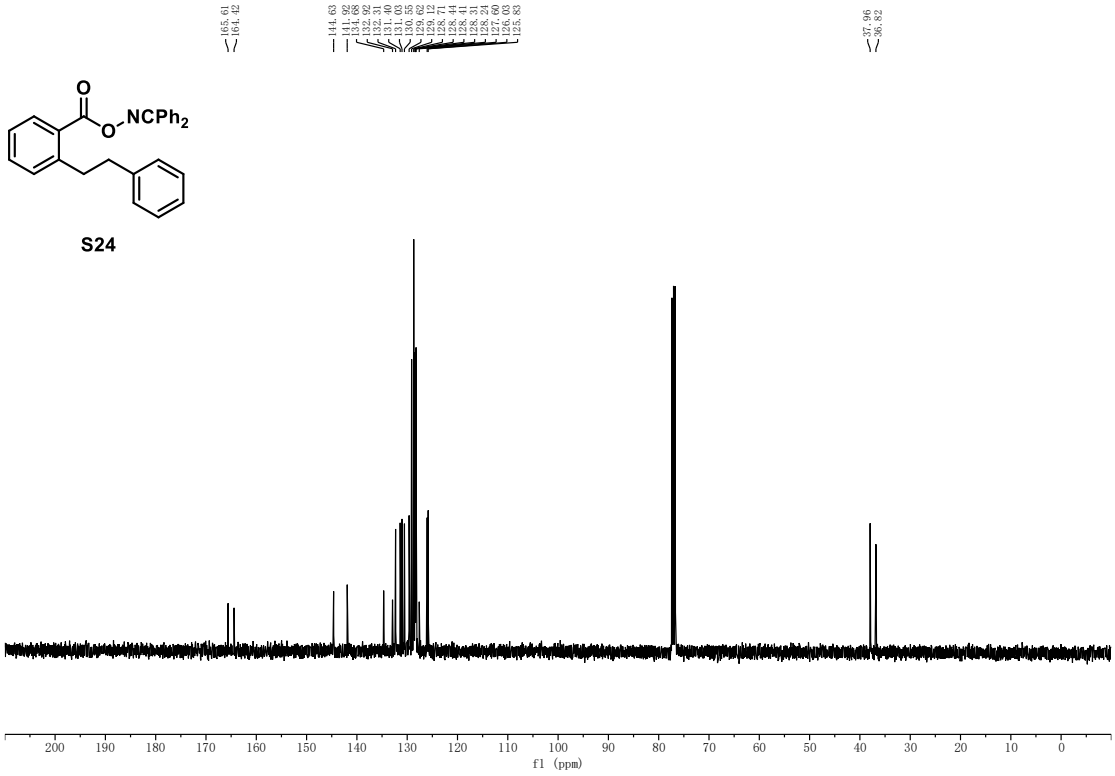

**Supplementary Figure 80** $^{1}$H NMR spectrum of **S23**



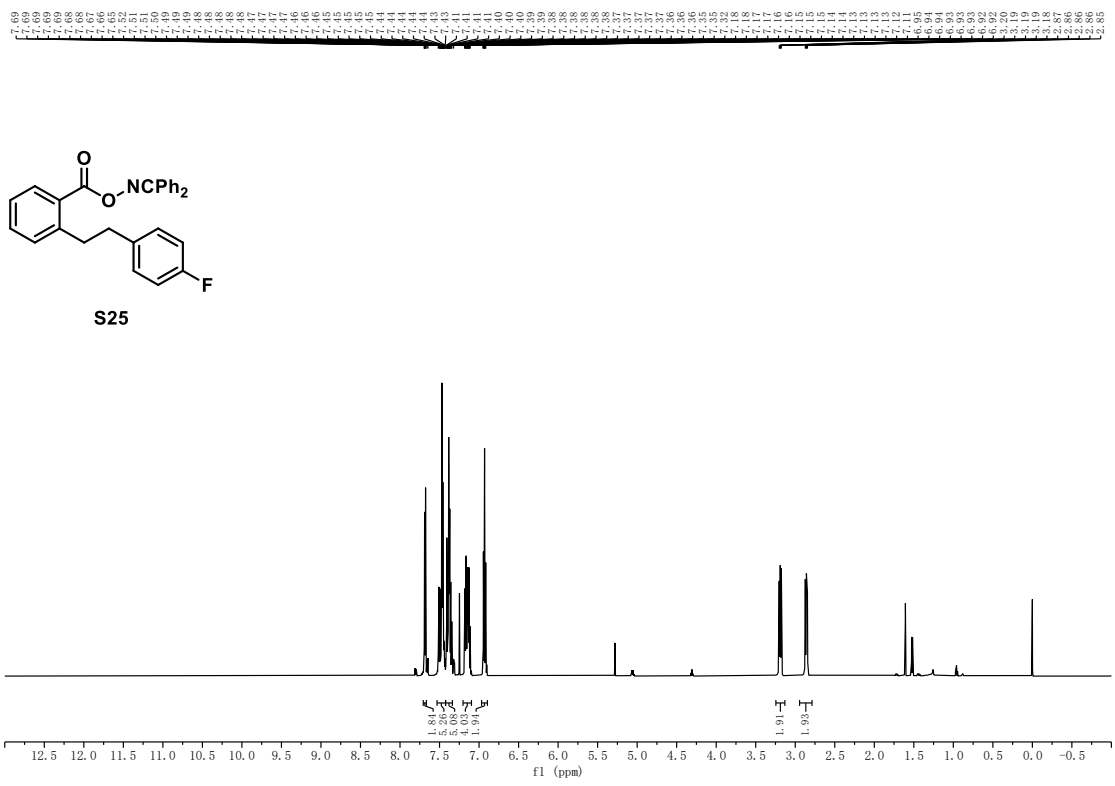

**Supplementary Figure 81** $^{13}$C NMR spectrum of **S23**



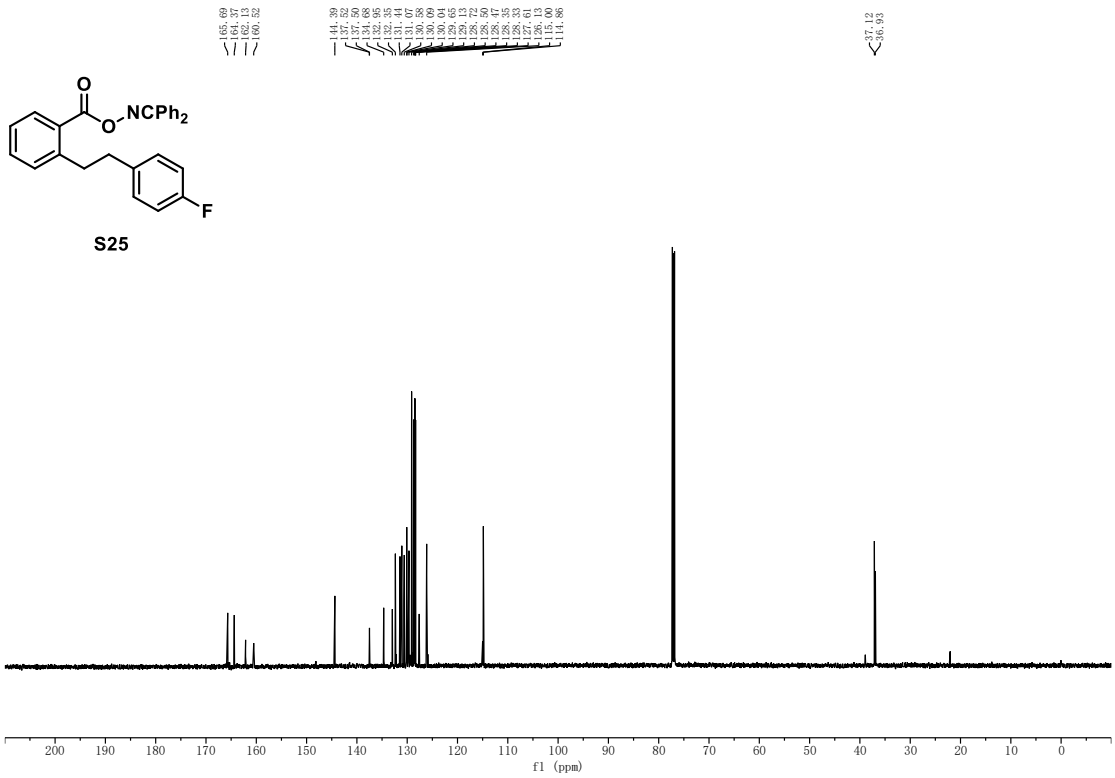

**Supplementary Figure 82** $^{1}$H NMR spectrum of **S24**



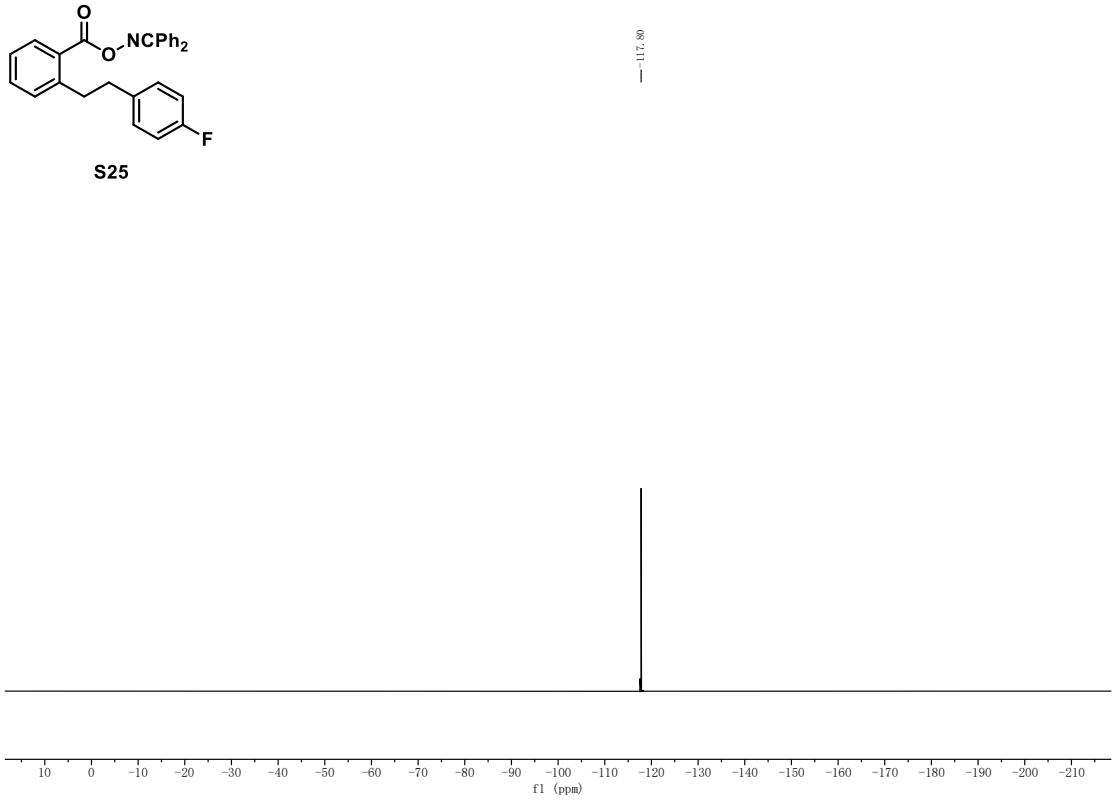

**Supplementary Figure 83** $^{13}$C NMR spectrum of **S24**



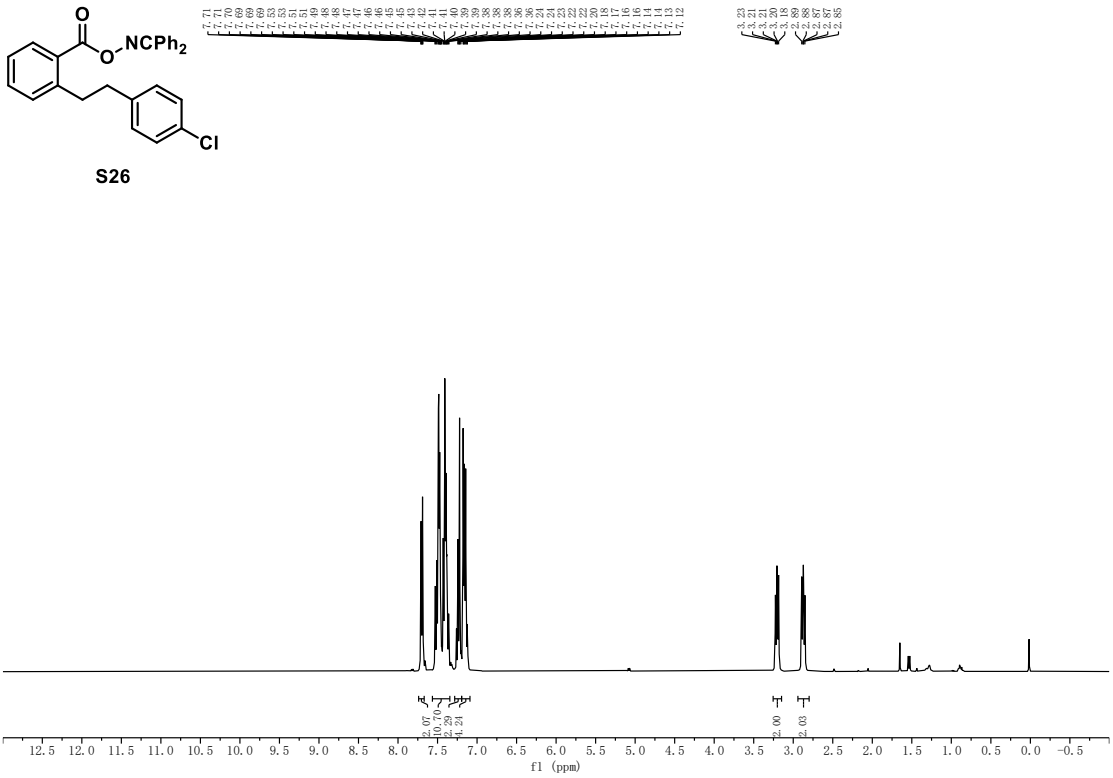

**Supplementary Figure 84** $^{1}$H NMR spectrum of **S25**



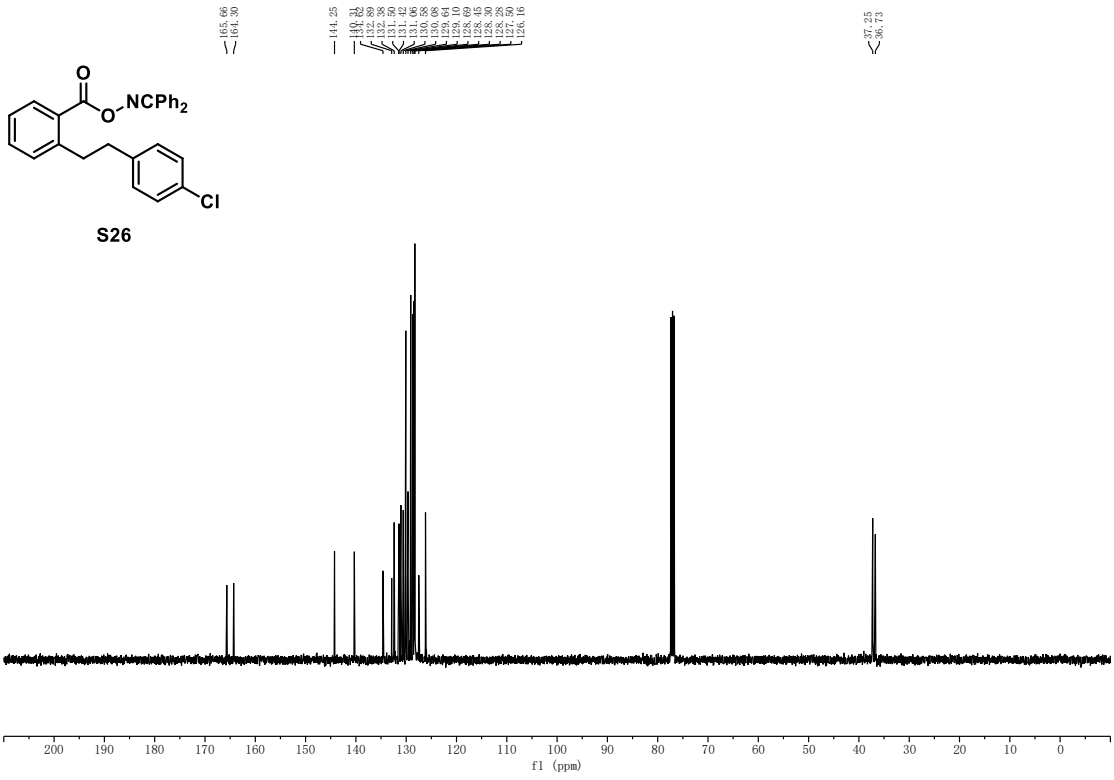

**Supplementary Figure 85** $^{13}$C NMR spectrum of **S25**



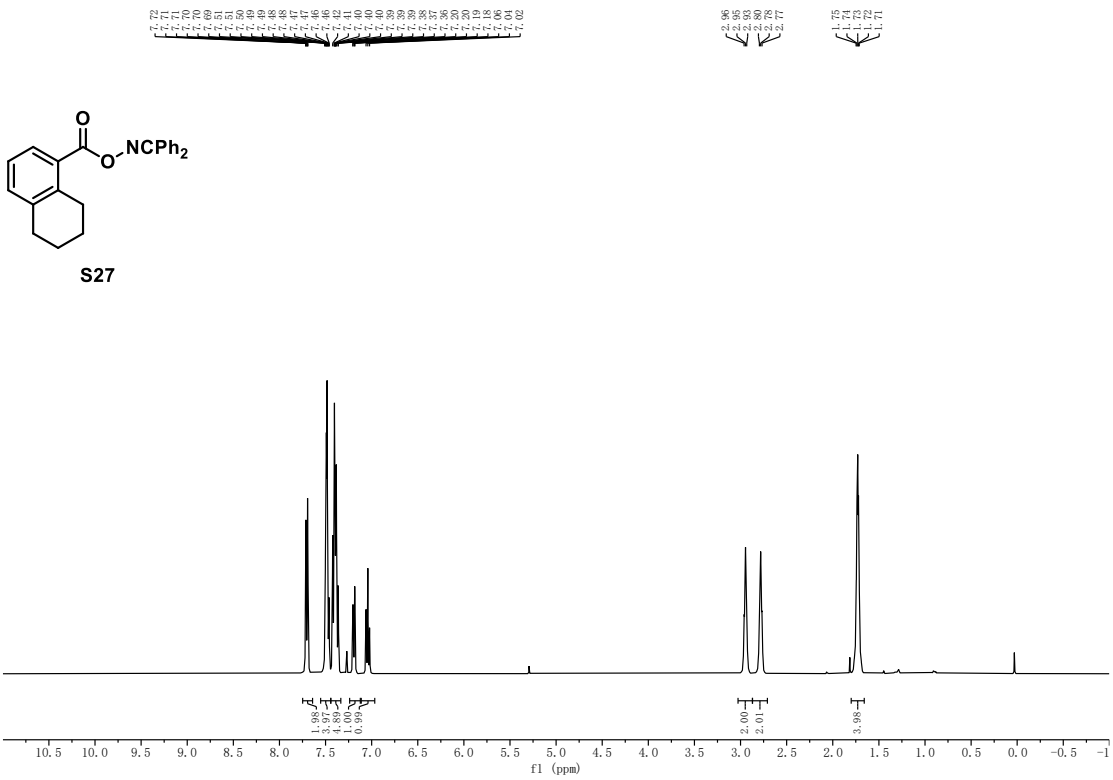

**Supplementary Figure 86** $^{19}$F NMR spectrum of **S25**



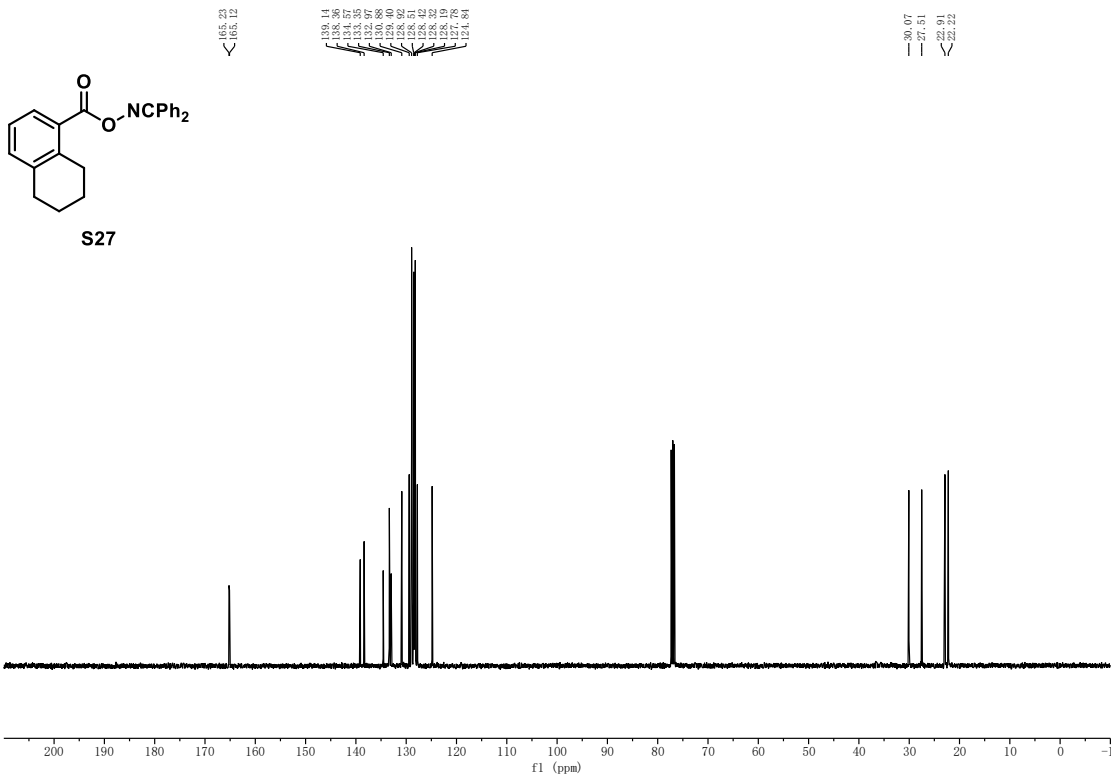

**Supplementary Figure 87** $^{1}$H NMR spectrum of **S26**



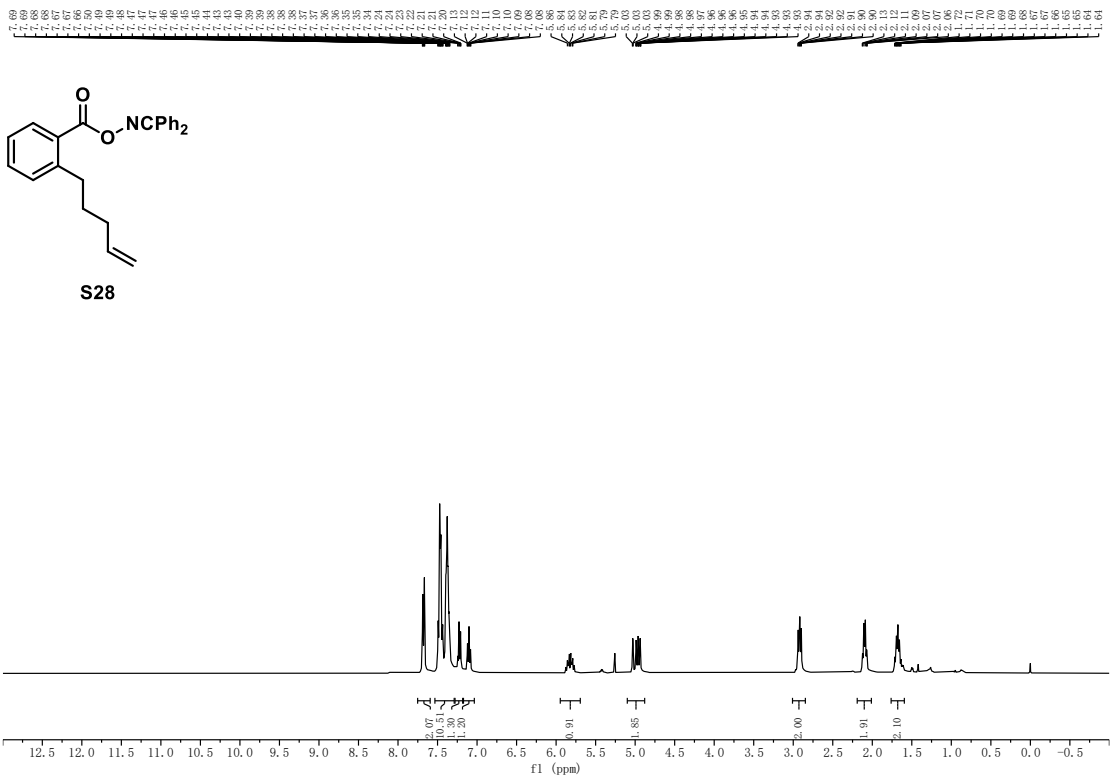

**Supplementary Figure 88** $^{13}$C NMR spectrum of **S26**



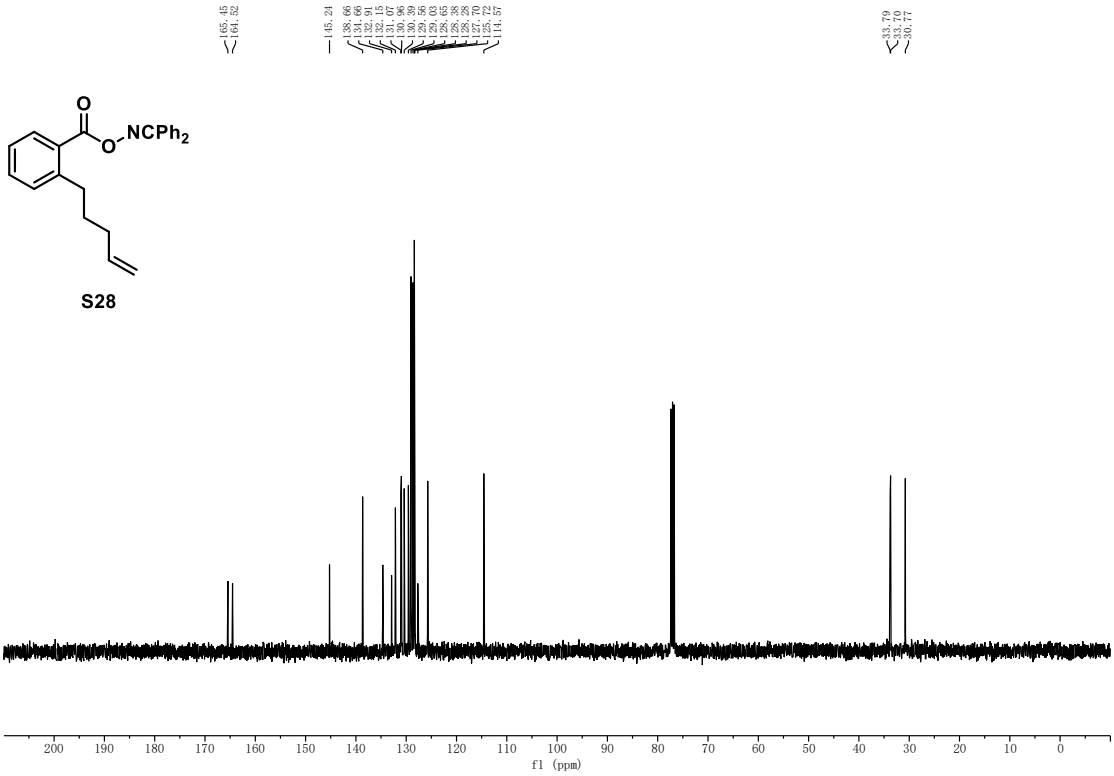

**Supplementary Figure 89** $^{1}$H NMR spectrum of **S27**



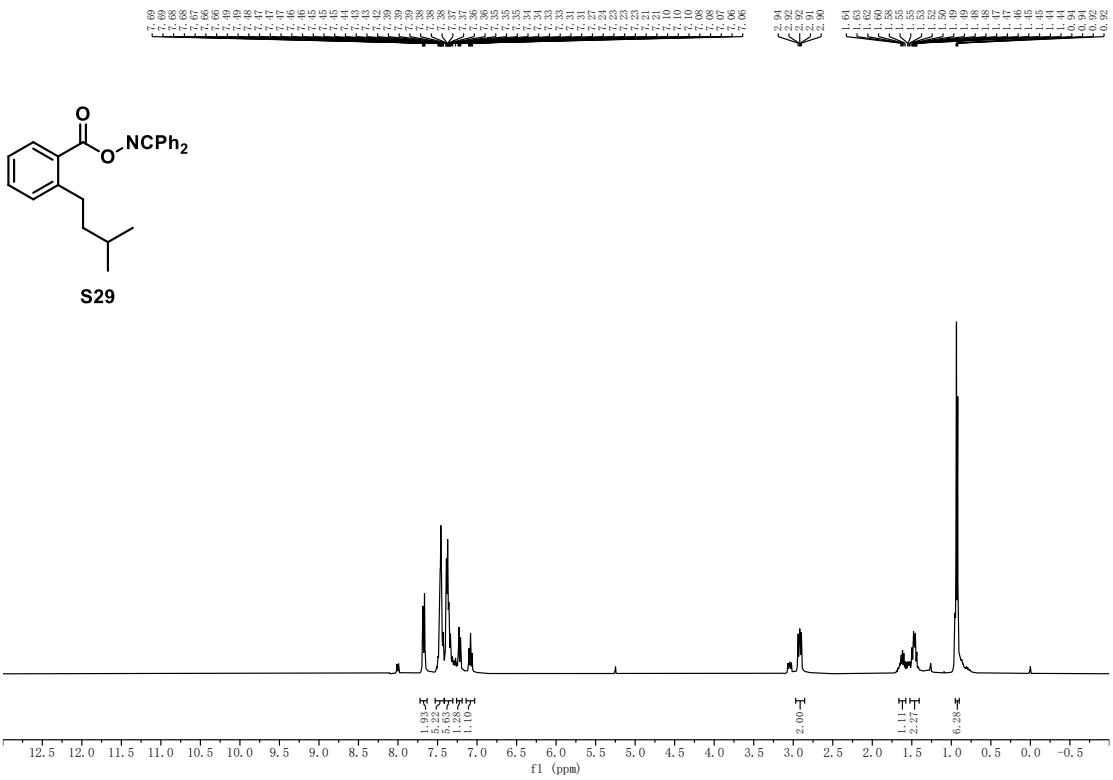

**Supplementary Figure 90** $^{13}$C NMR spectrum of **S27**



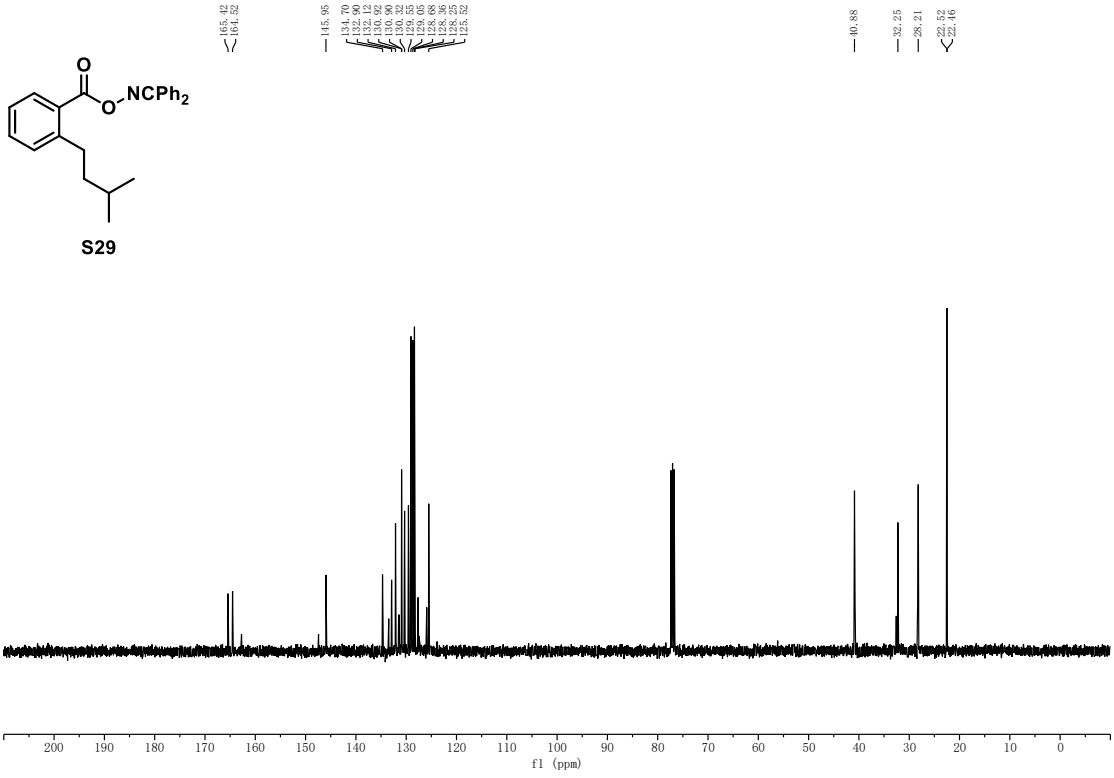

**Supplementary Figure 91** $^{1}$H NMR spectrum of **S28**



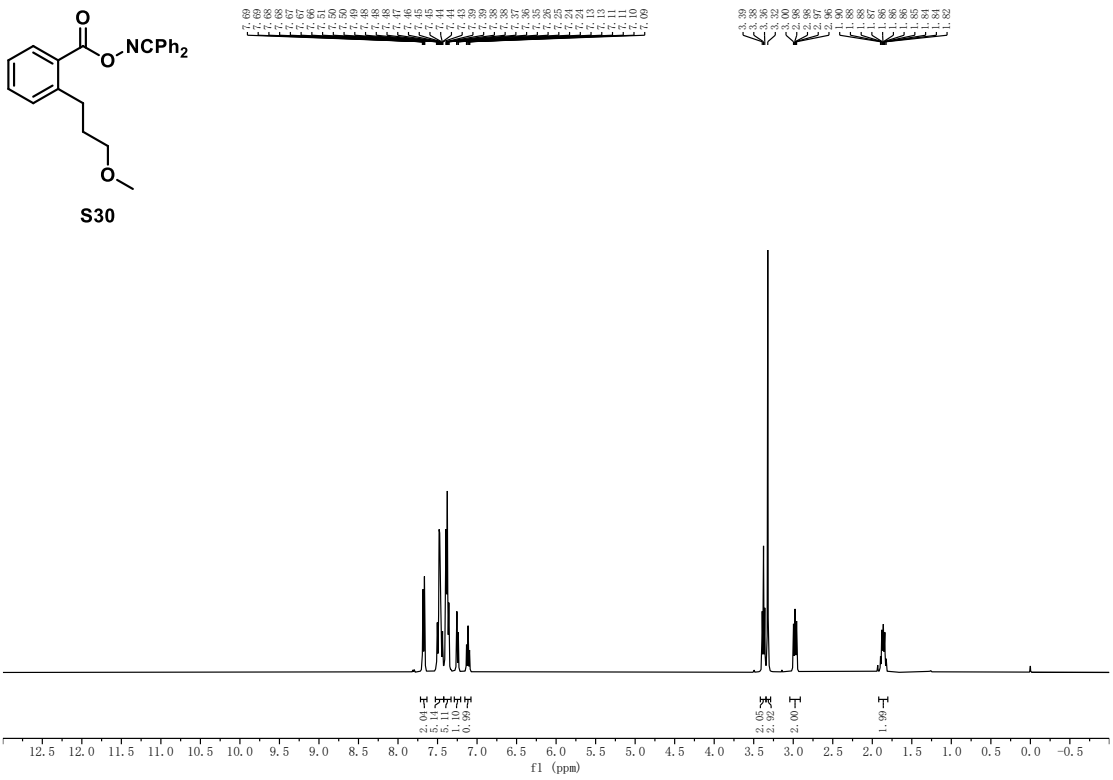

**Supplementary Figure 92** $^{13}$C NMR spectrum of **S28**



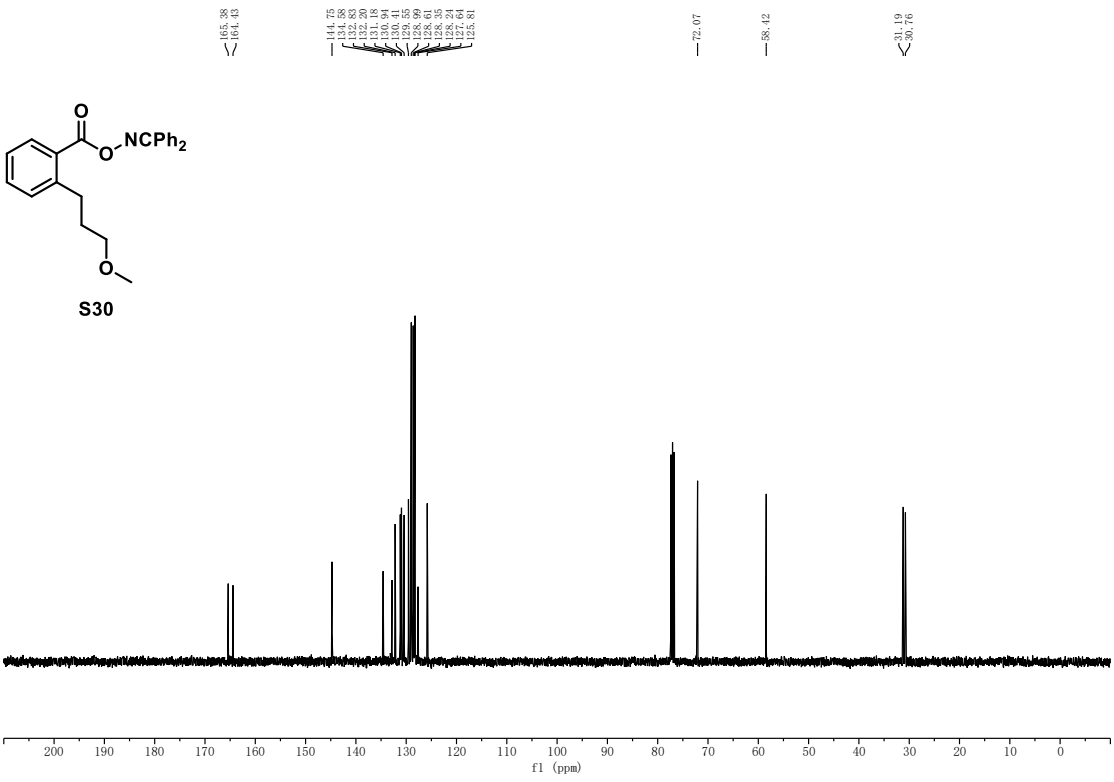

**Supplementary Figure 93** $^{1}$H NMR spectrum of **S29**



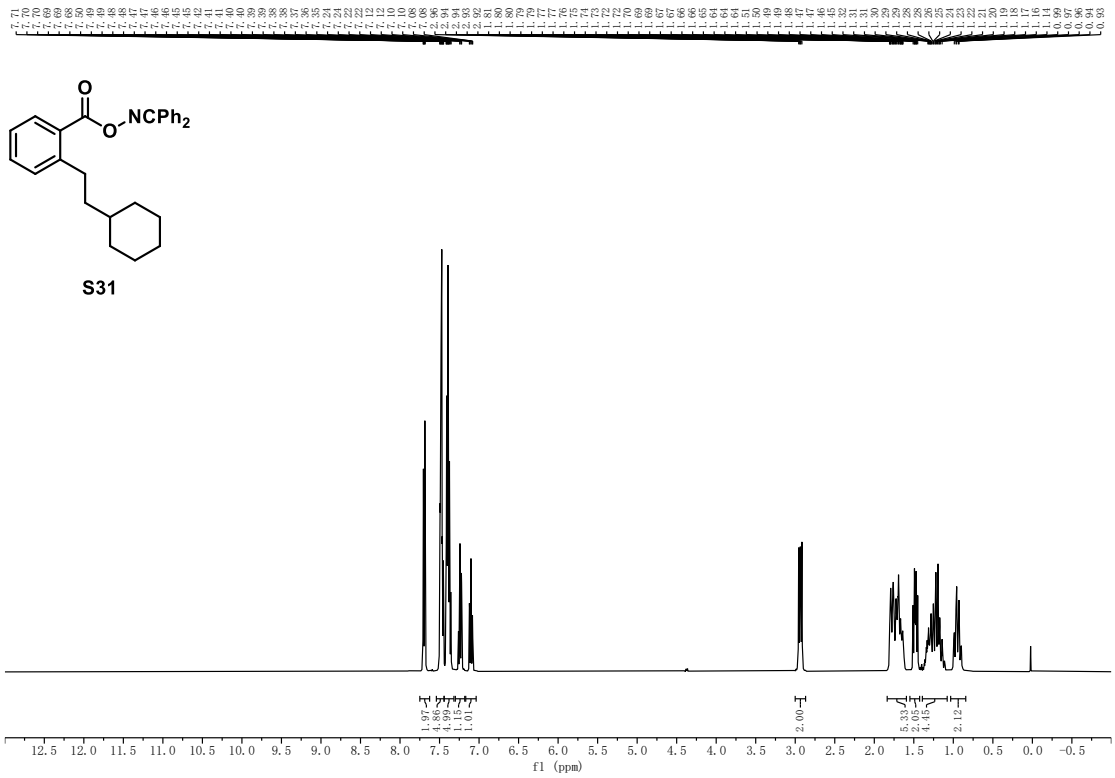

**Supplementary Figure 94** $^{13}$C NMR spectrum of **S29**



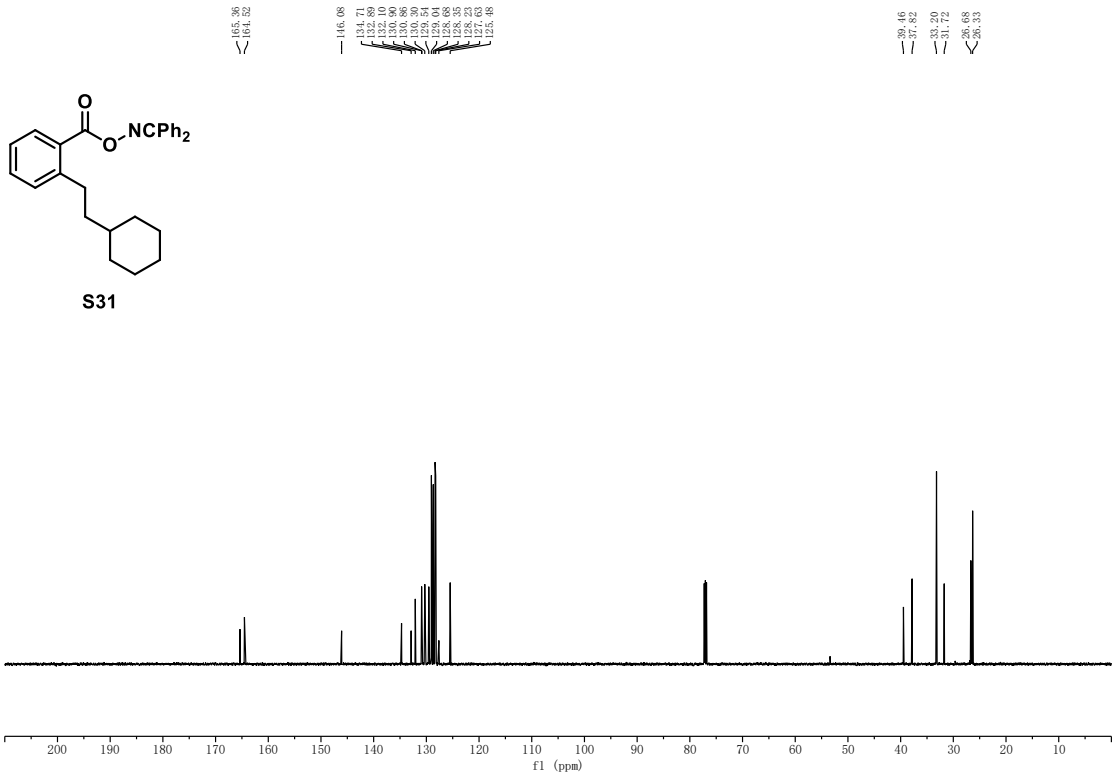

**Supplementary Figure 95** $^{1}$H NMR spectrum of **S30**



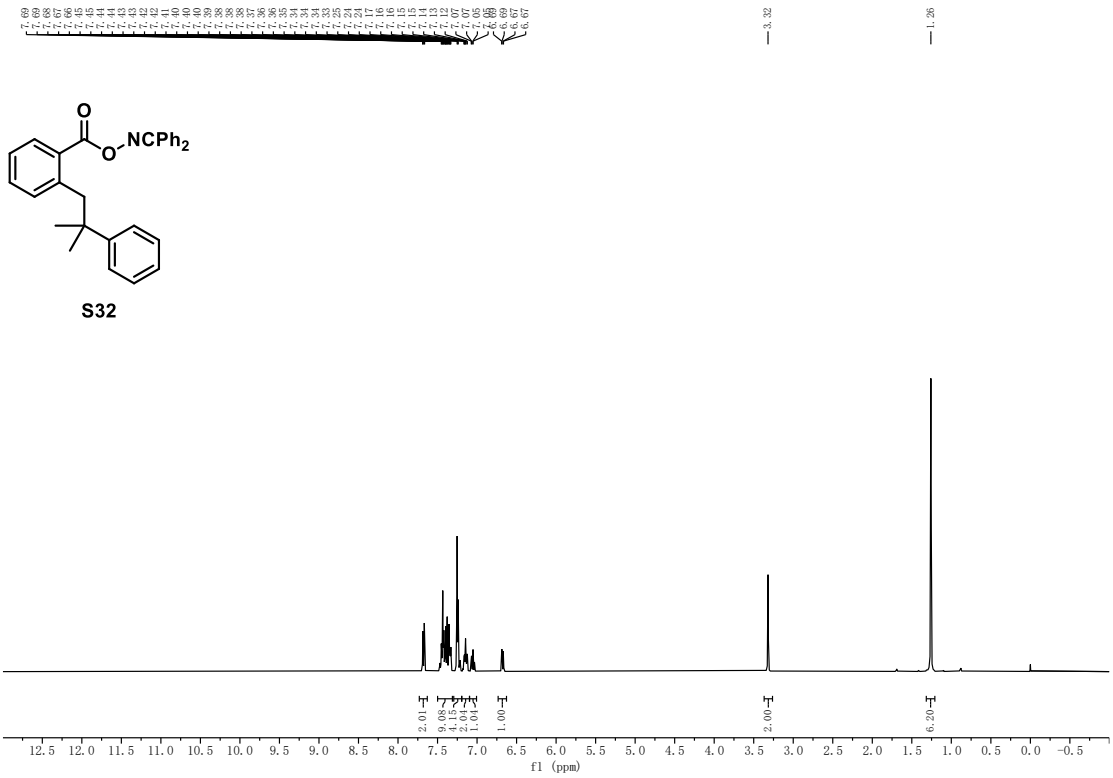

**Supplementary Figure 96** $^{13}$C NMR spectrum of **S30**



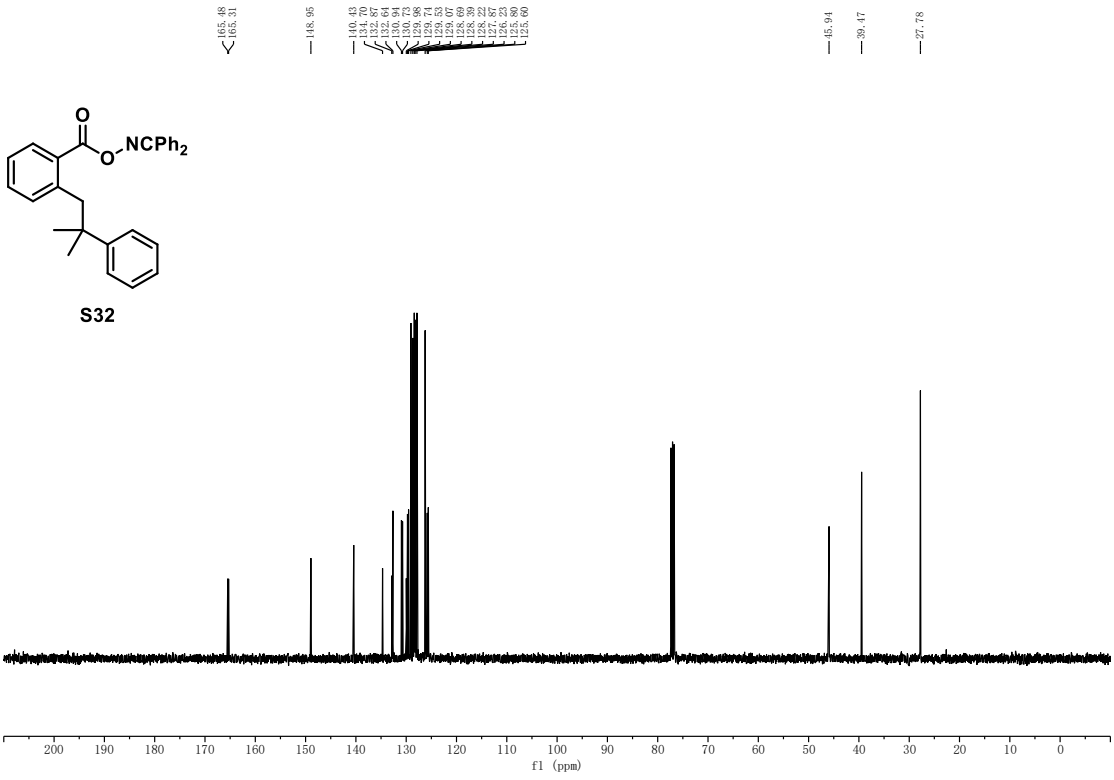

**Supplementary Figure 97** $^{1}$H NMR spectrum of **S31**



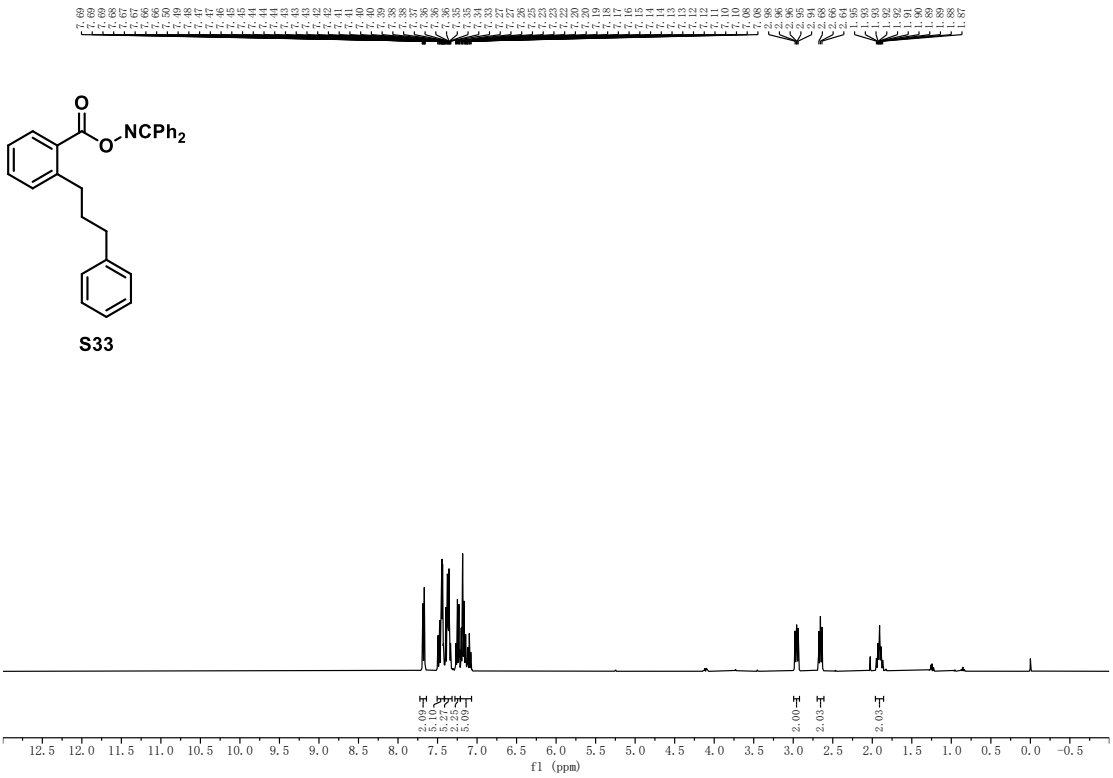

**Supplementary Figure 98** $^{13}$C NMR spectrum of **S31**



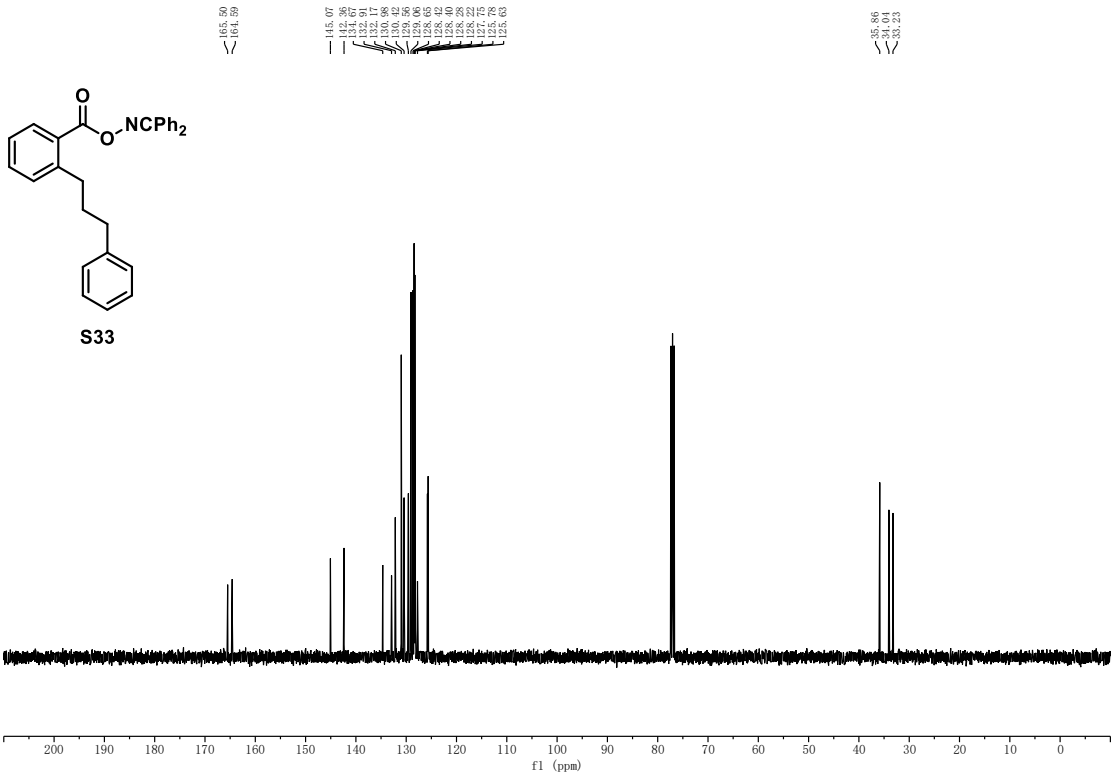

**Supplementary Figure 99** $^{1}$H NMR spectrum of **S32**



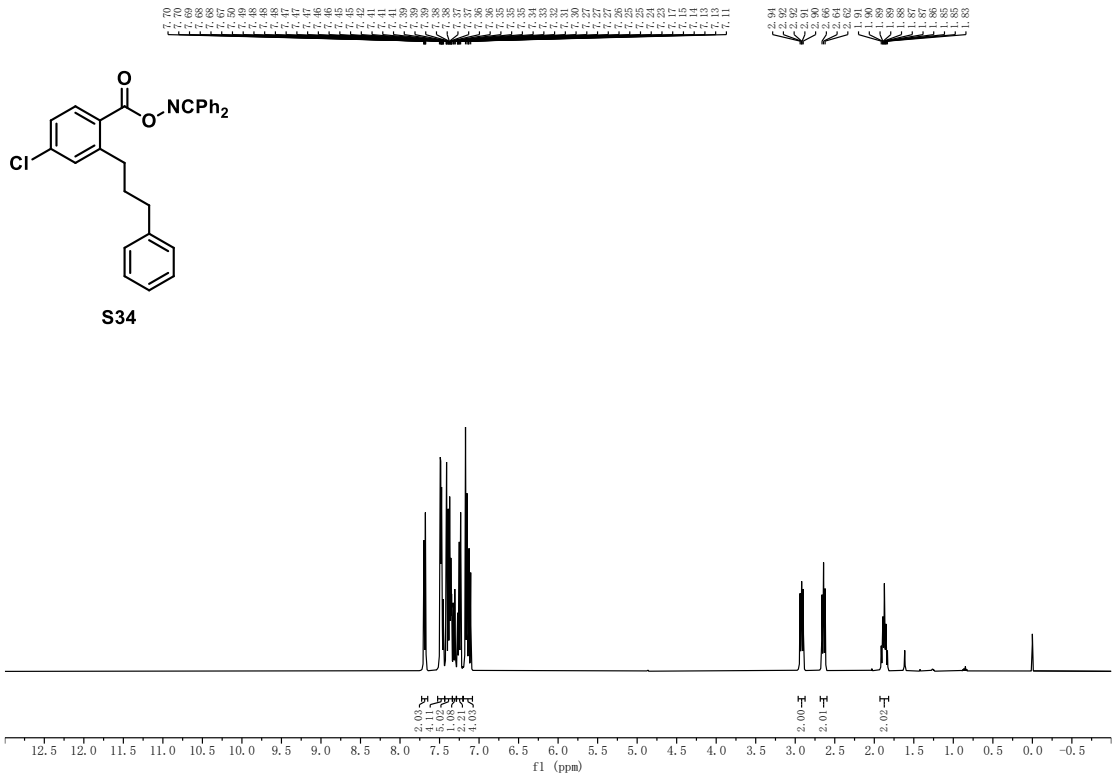

**Supplementary Figure 100** $^{13}$C NMR spectrum of **S32**



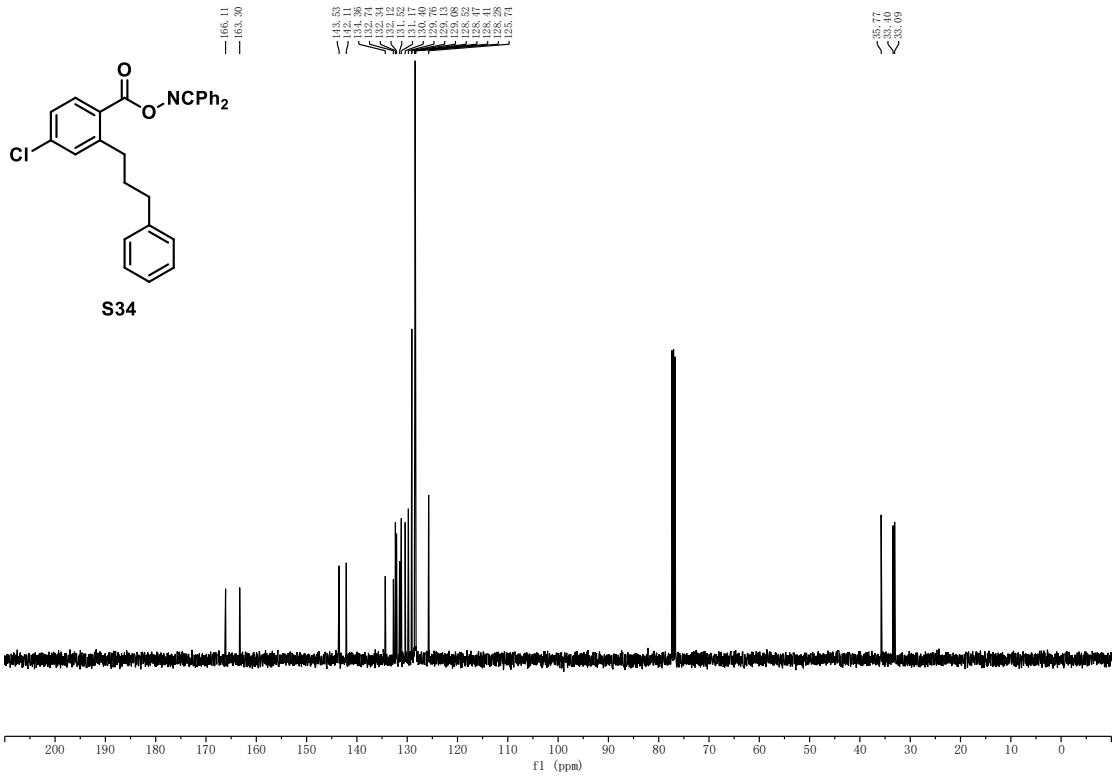

**Supplementary Figure 101** $^{1}$H NMR spectrum of **S33**



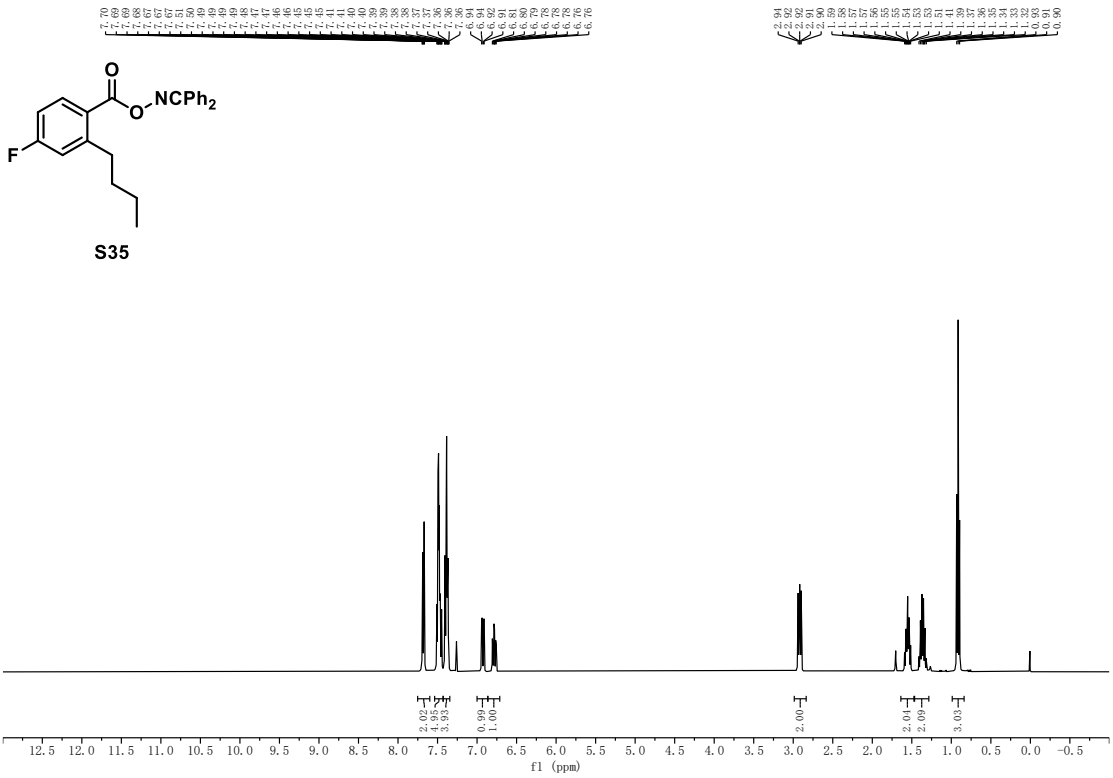

**Supplementary Figure 102** $^{13}$C NMR spectrum of **S33**



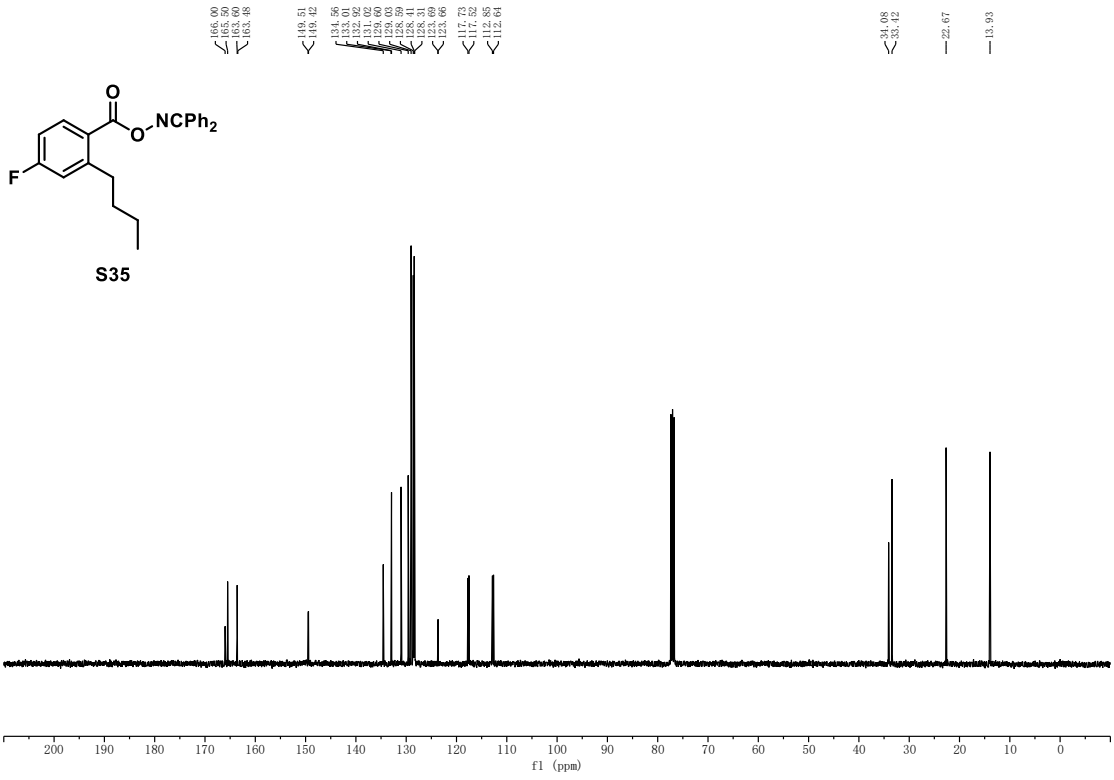

**Supplementary Figure 103** $^{1}$H NMR spectrum of **S34**



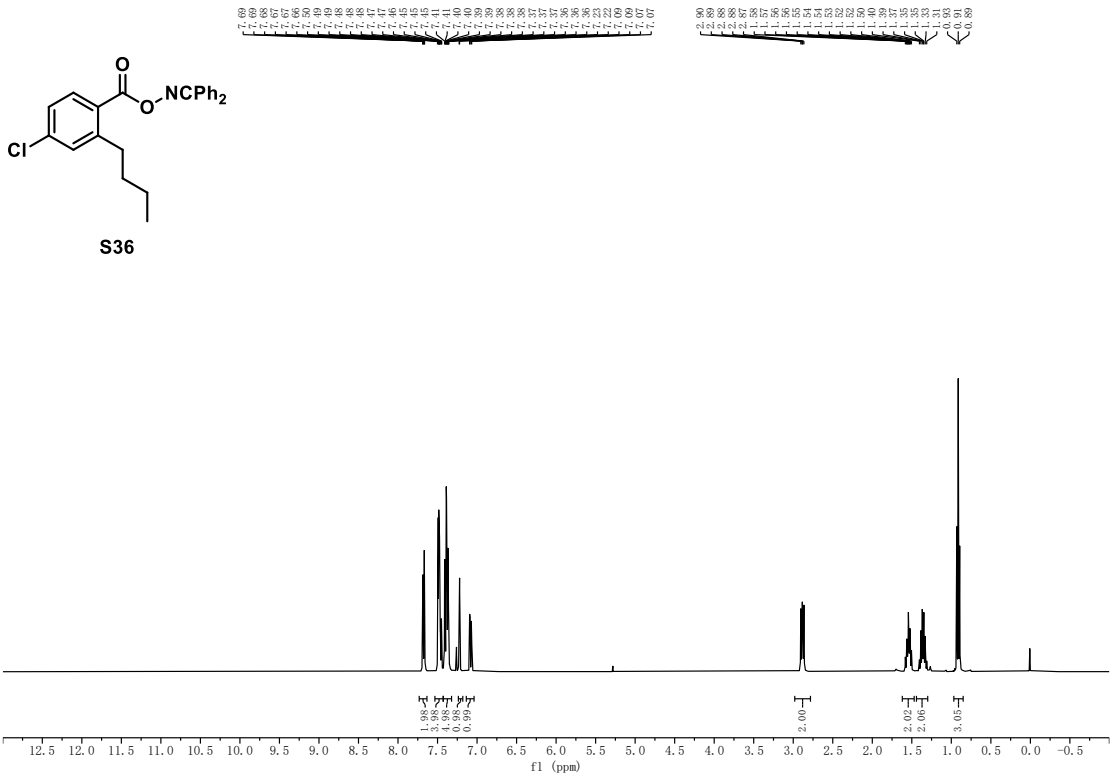

**Supplementary Figure 104** $^{13}$C NMR spectrum of **S34**



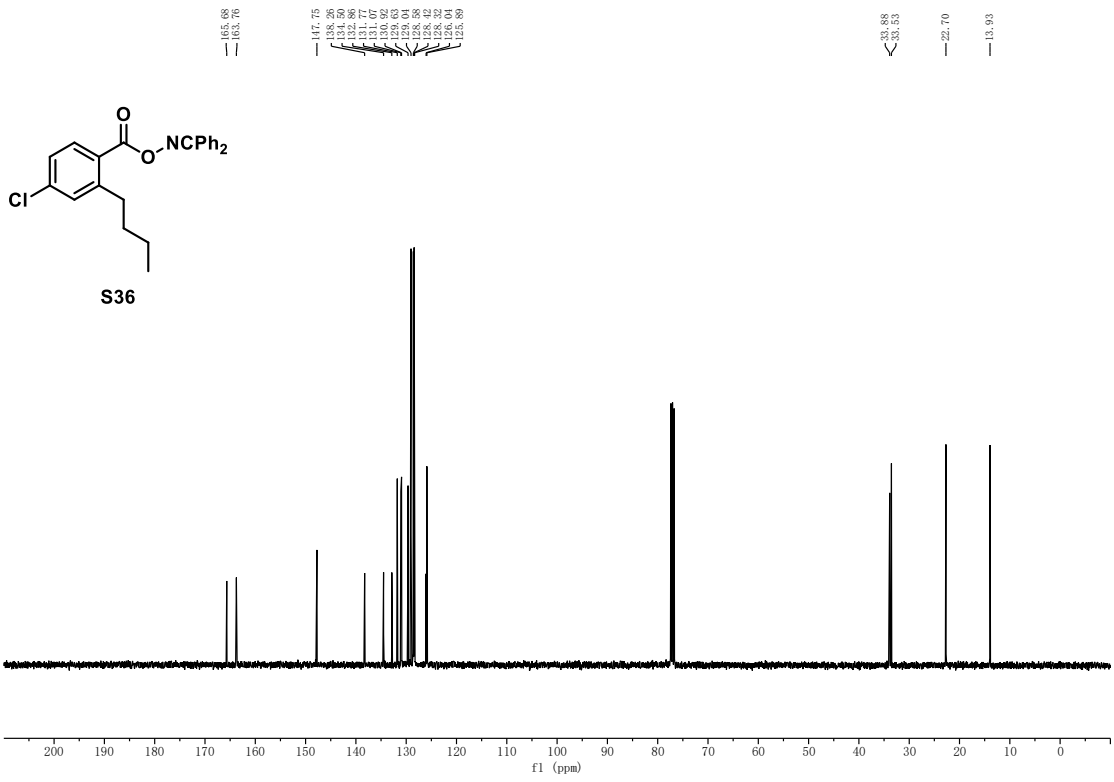

**Supplementary Figure 105** $^{1}$H NMR spectrum of **S35**



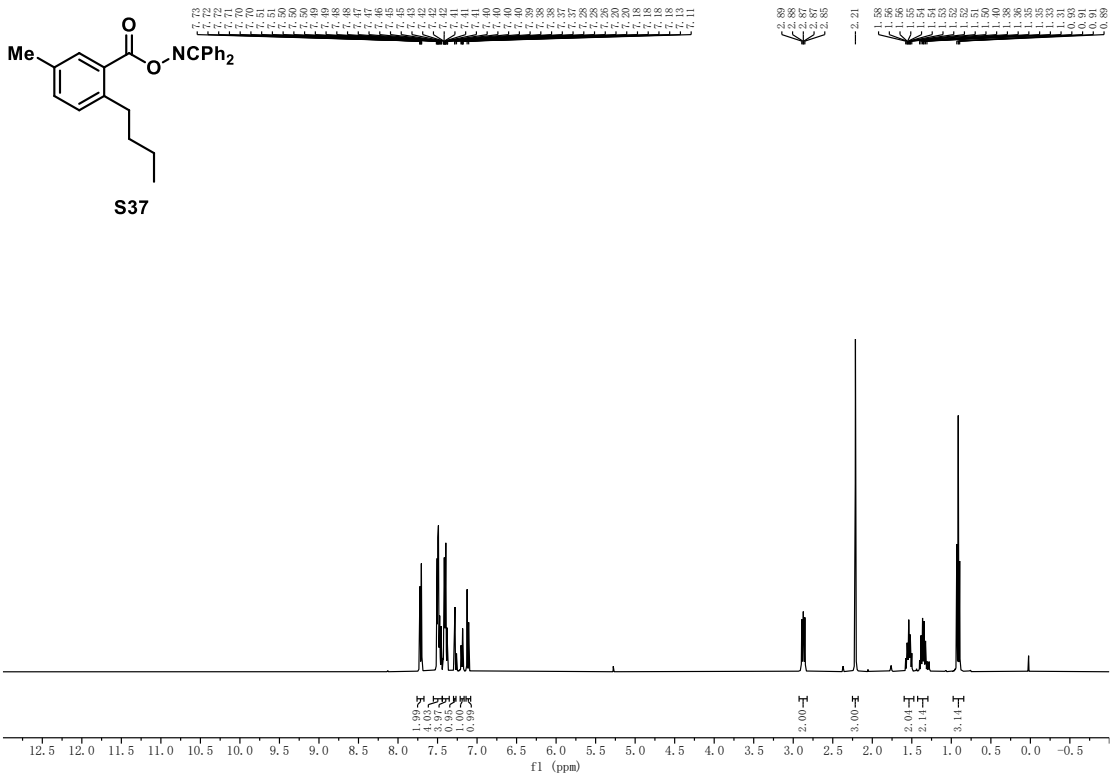

**Supplementary Figure 106** $^{13}$C NMR spectrum of **S35**



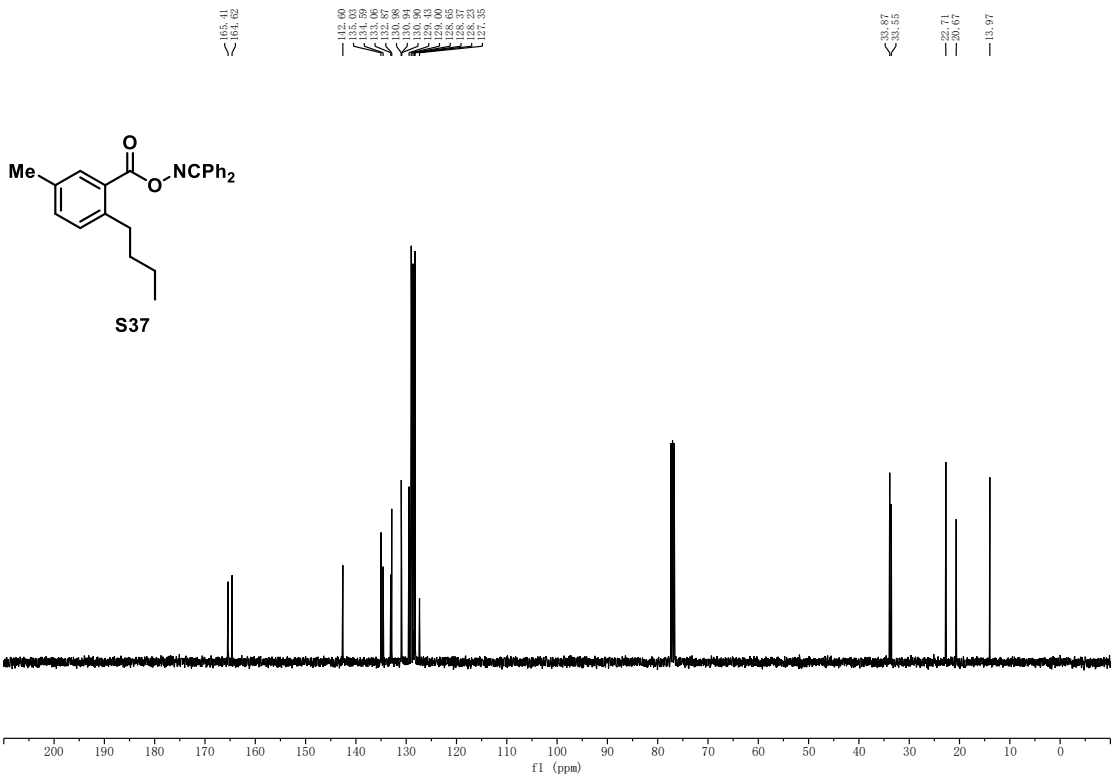

**Supplementary Figure 107** $^{1}$H NMR spectrum of **S36**



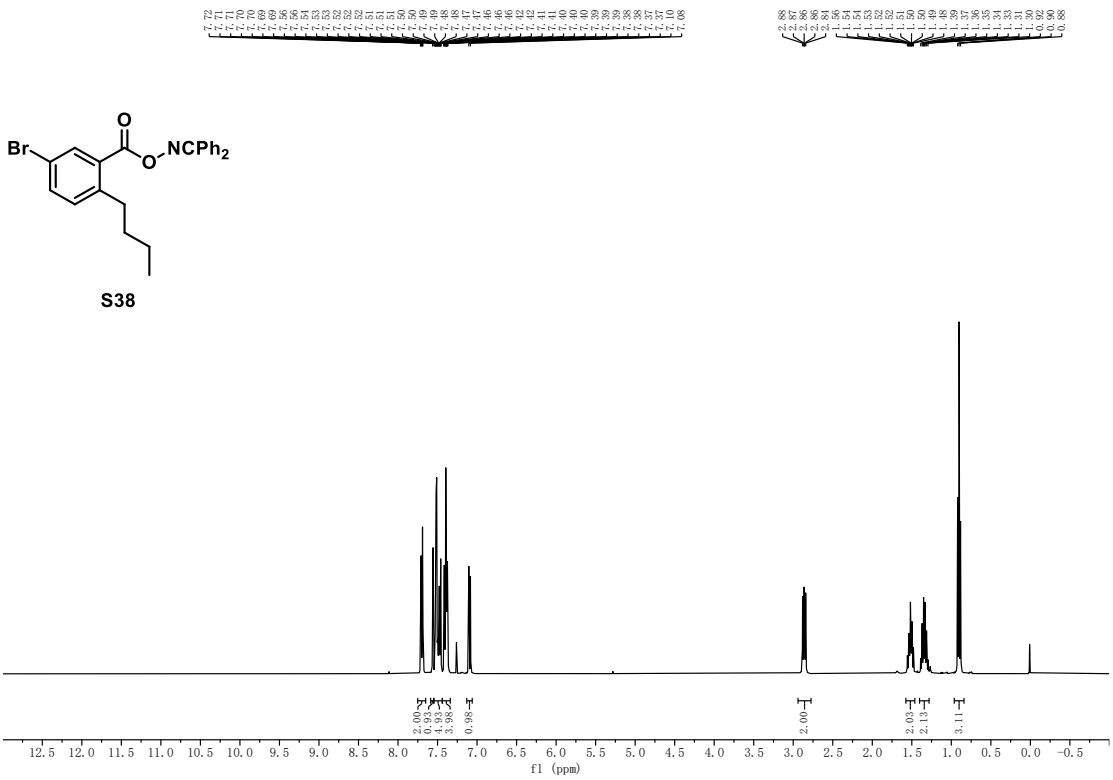

**Supplementary Figure 108** $^{13}$C NMR spectrum of **S36**



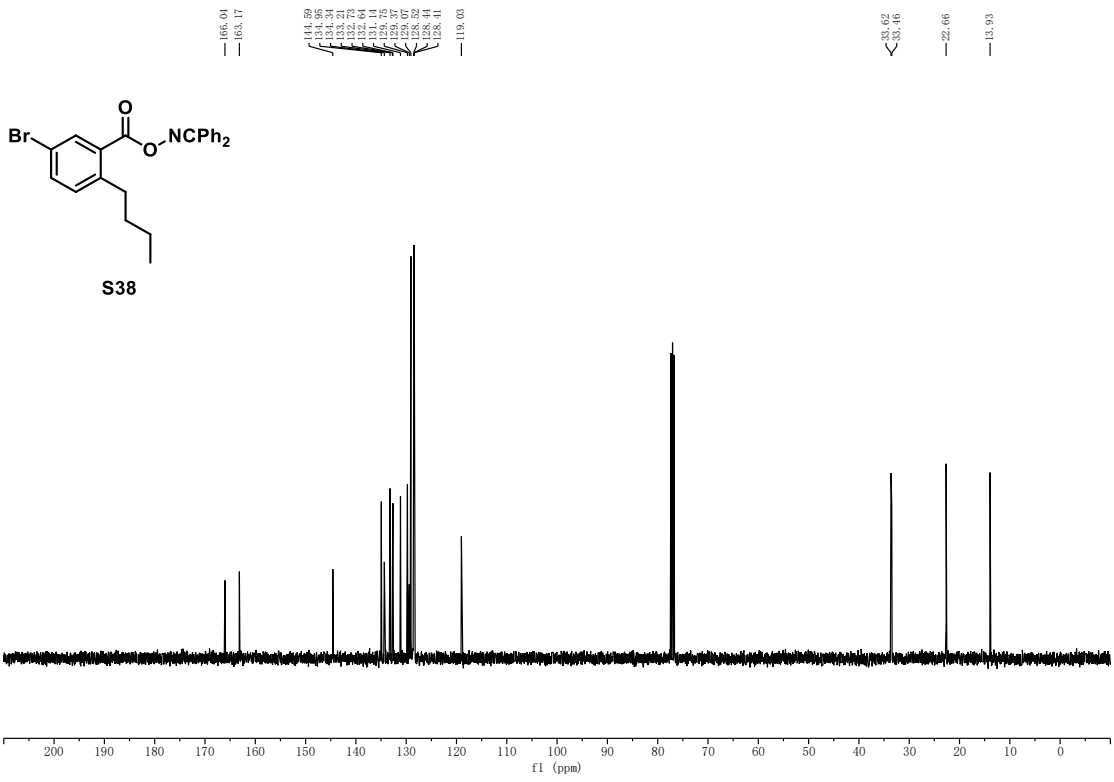

**Supplementary Figure 109** $^{1}$H NMR spectrum of **S37**



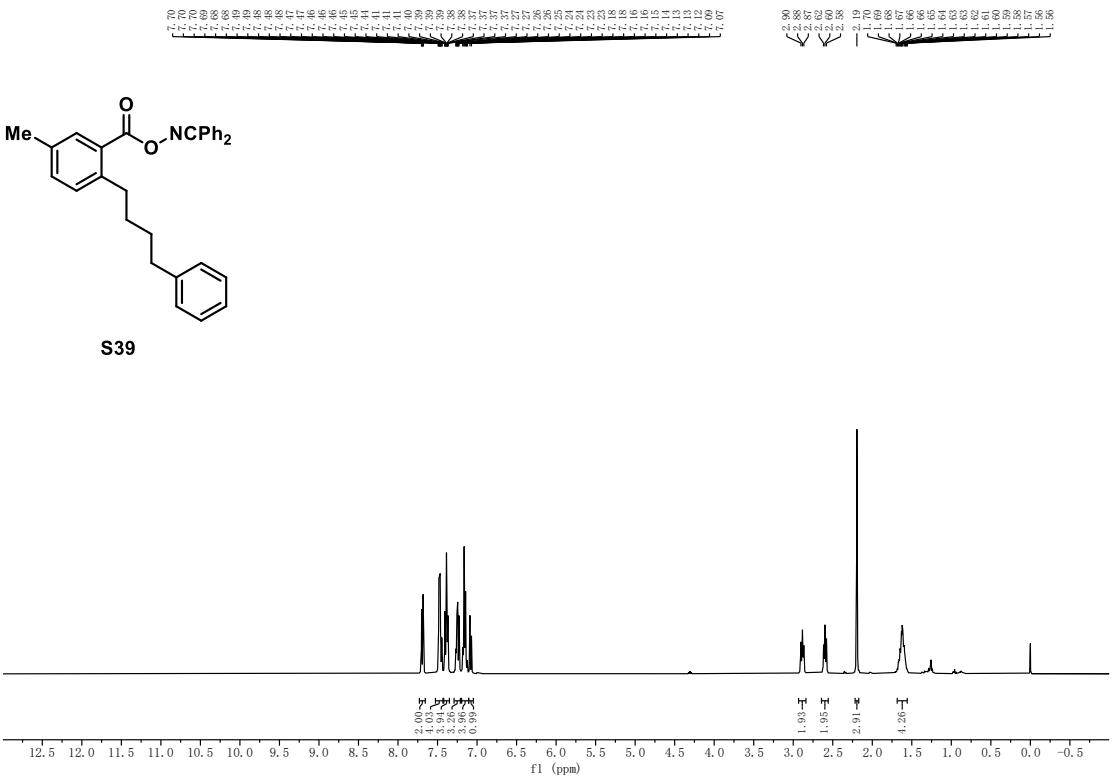

**Supplementary Figure 110** $^{13}$C NMR spectrum of **S37**



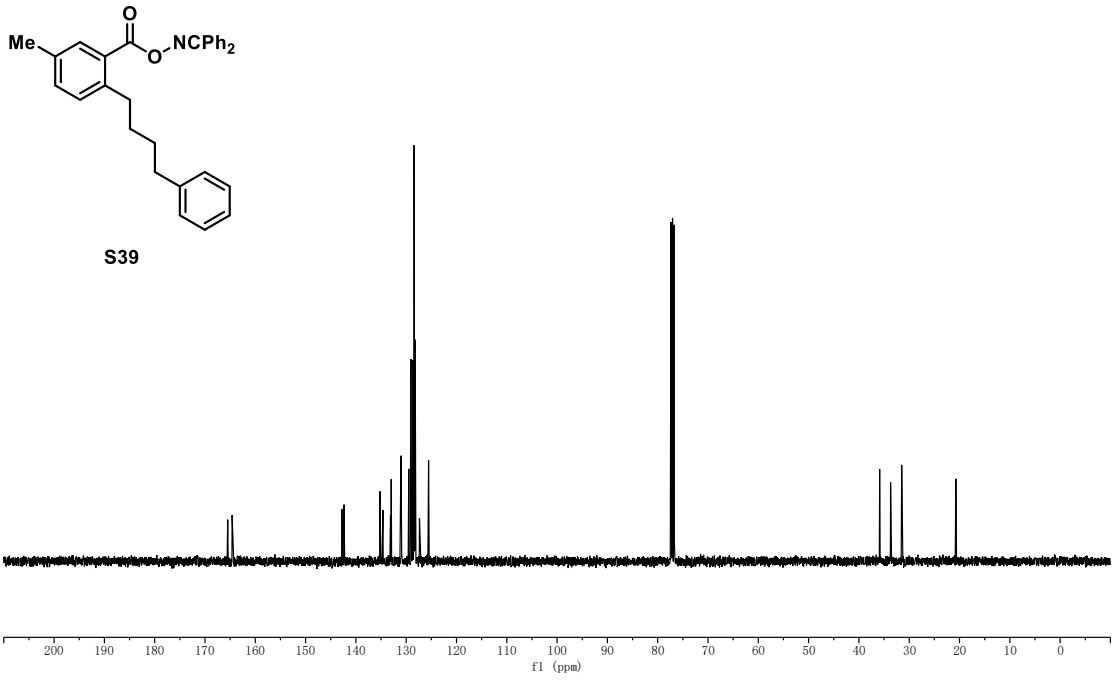

**Supplementary Figure 111** $^{1}$H NMR spectrum of **S38**



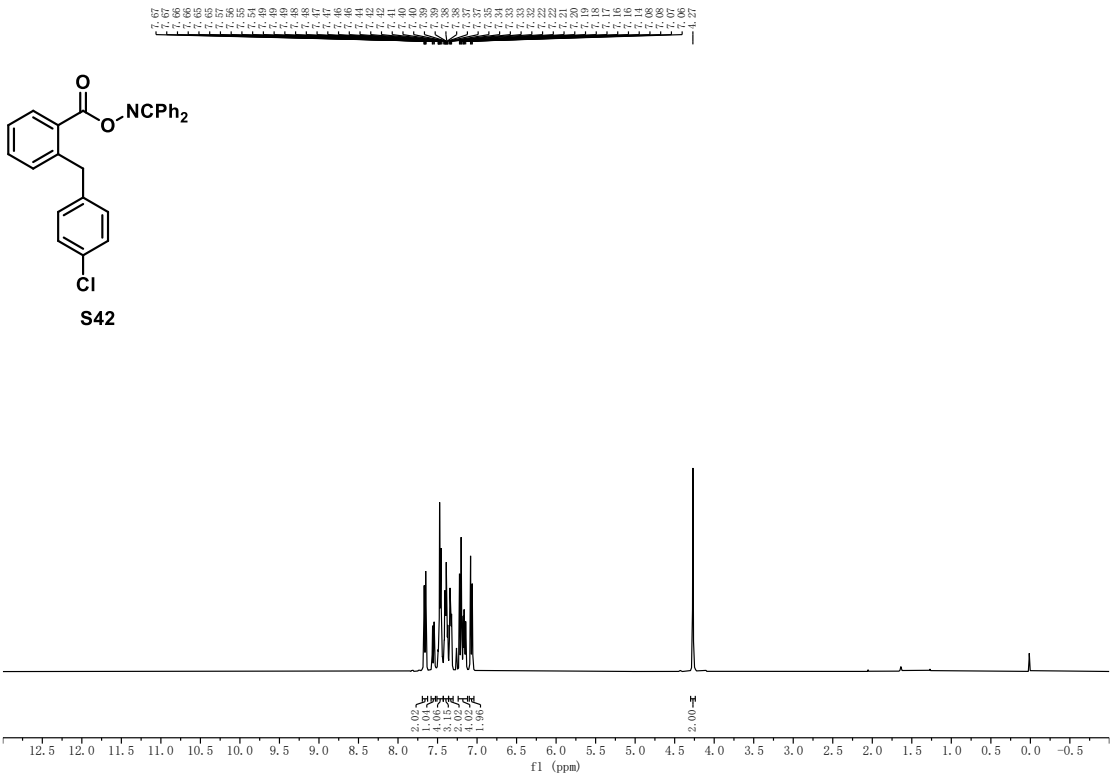

**Supplementary Figure 112** $^{13}$C NMR spectrum of **S38**



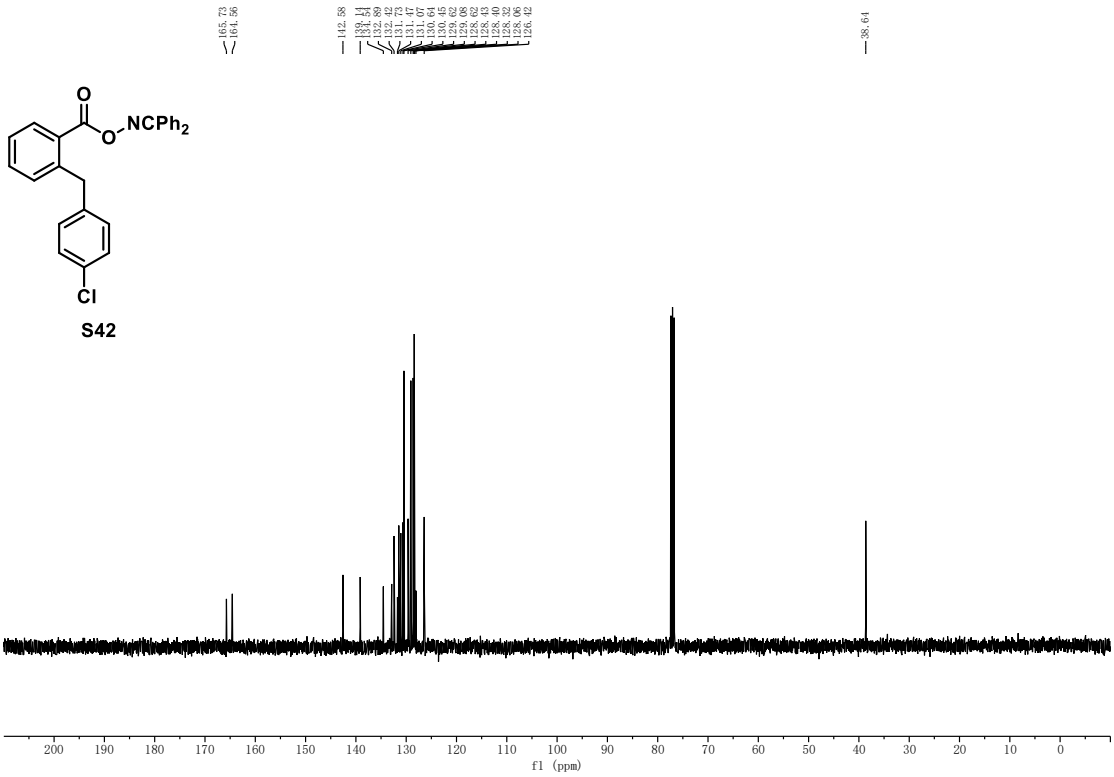

**Supplementary Figure 113** $^{1}$H NMR spectrum of **S39**



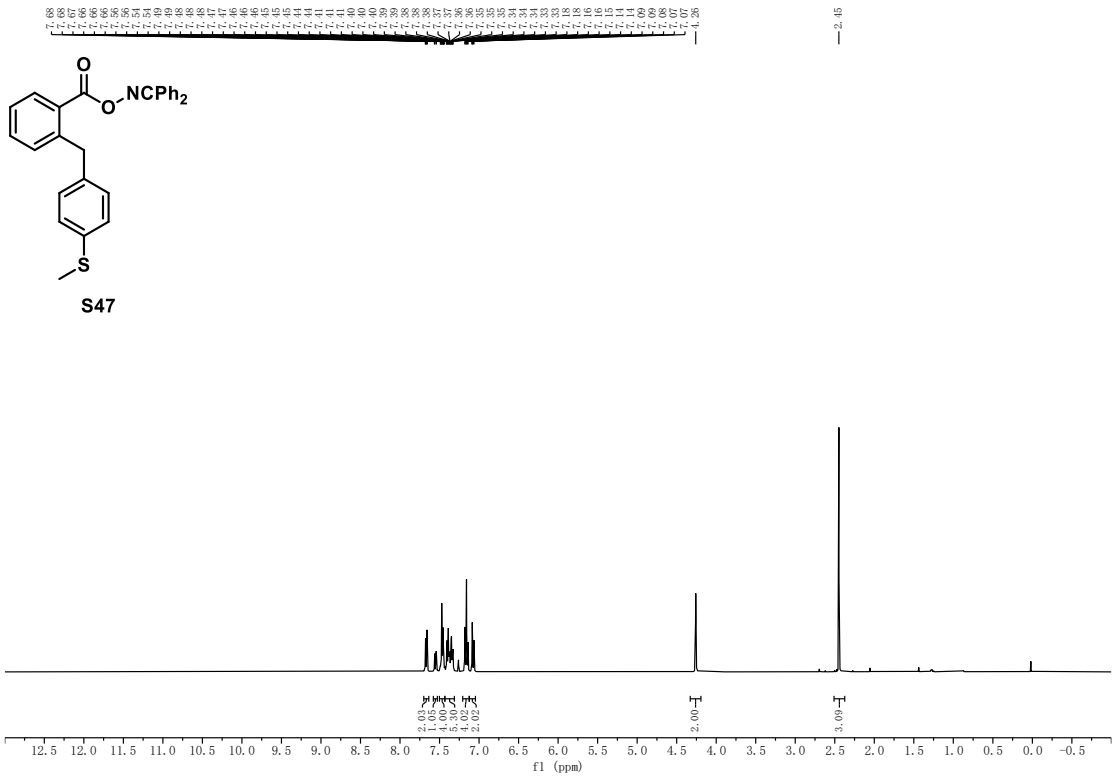

**Supplementary Figure 114** $^{13}$C NMR spectrum of **S39**



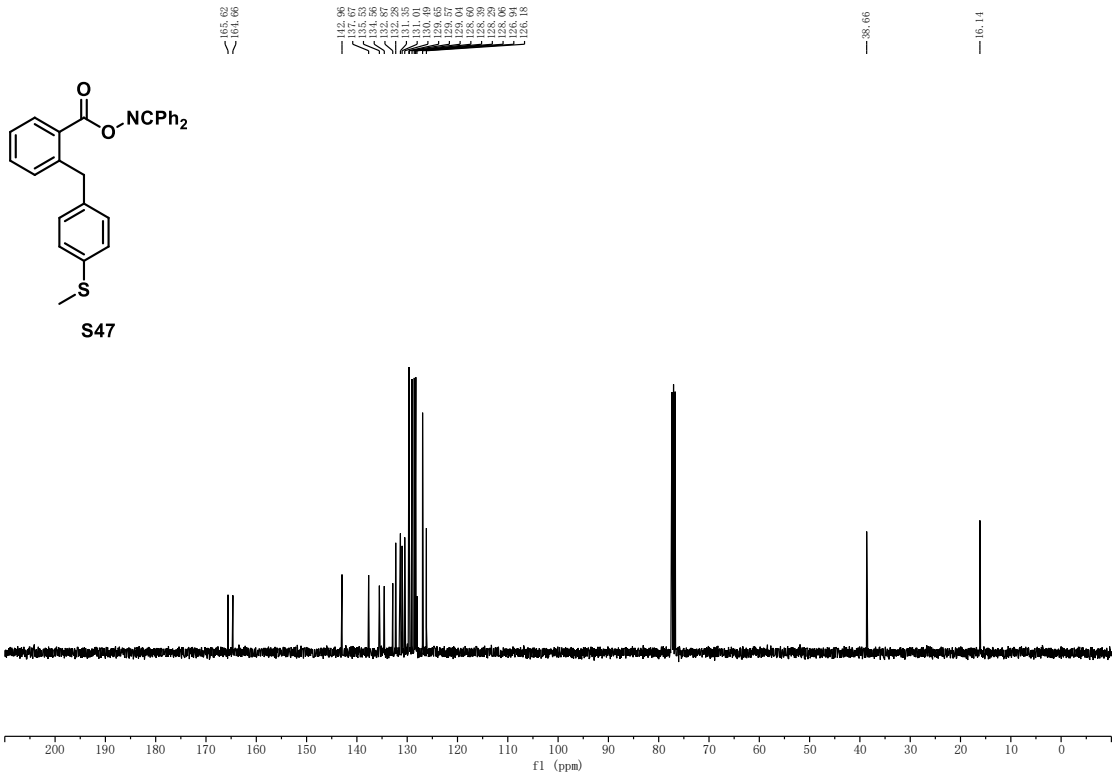

**Supplementary Figure 115** $^{1}$H NMR spectrum of **S42**



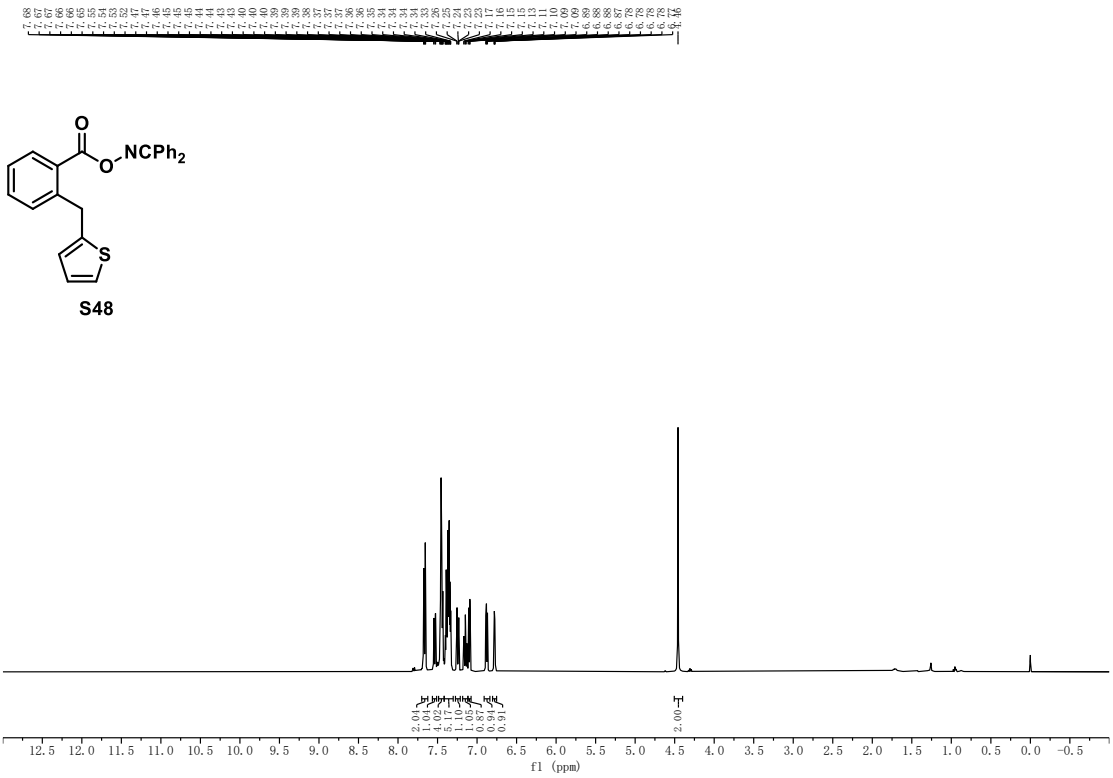

**Supplementary Figure 116** $^{13}$C NMR spectrum of **S42**



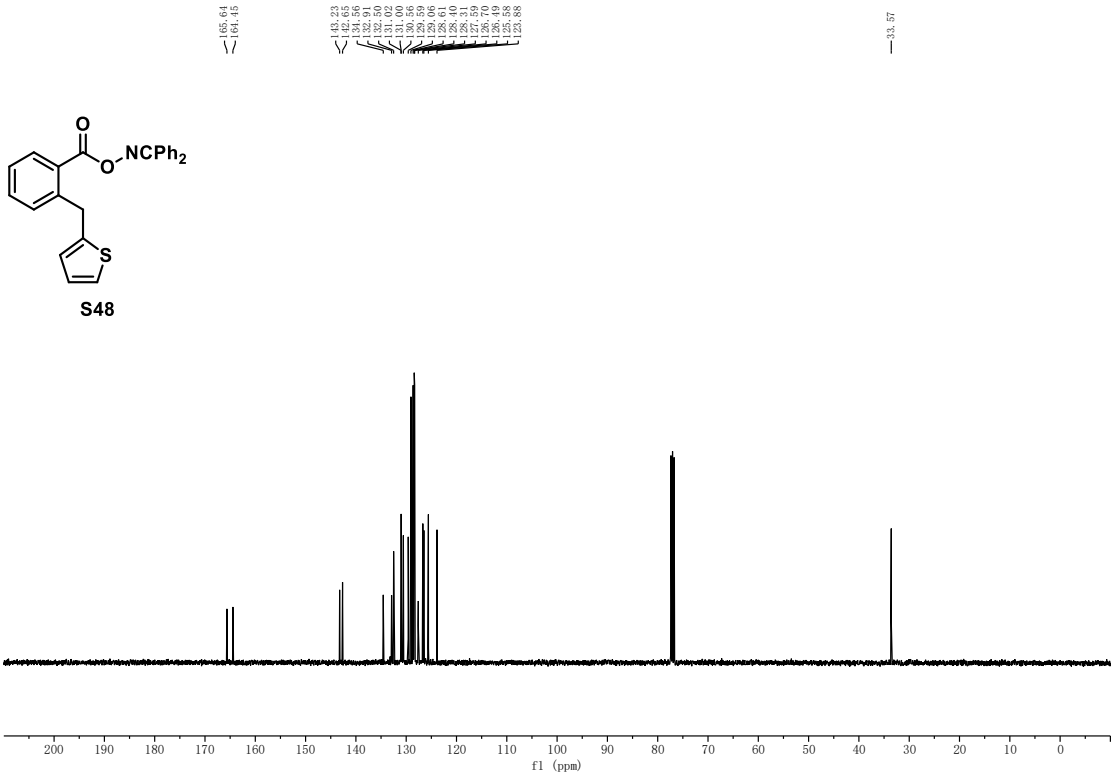

**Supplementary Figure 117** $^{1}$H NMR spectrum of **S47**



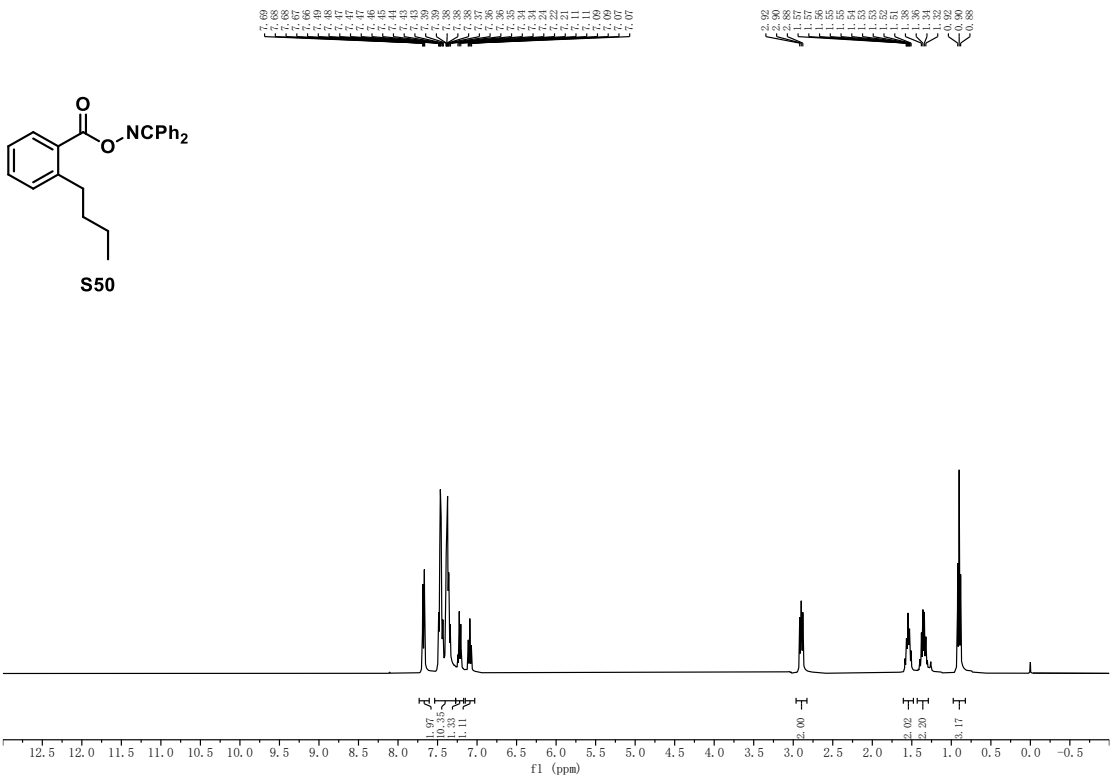

**Supplementary Figure 118** $^{13}$C NMR spectrum of **S47**



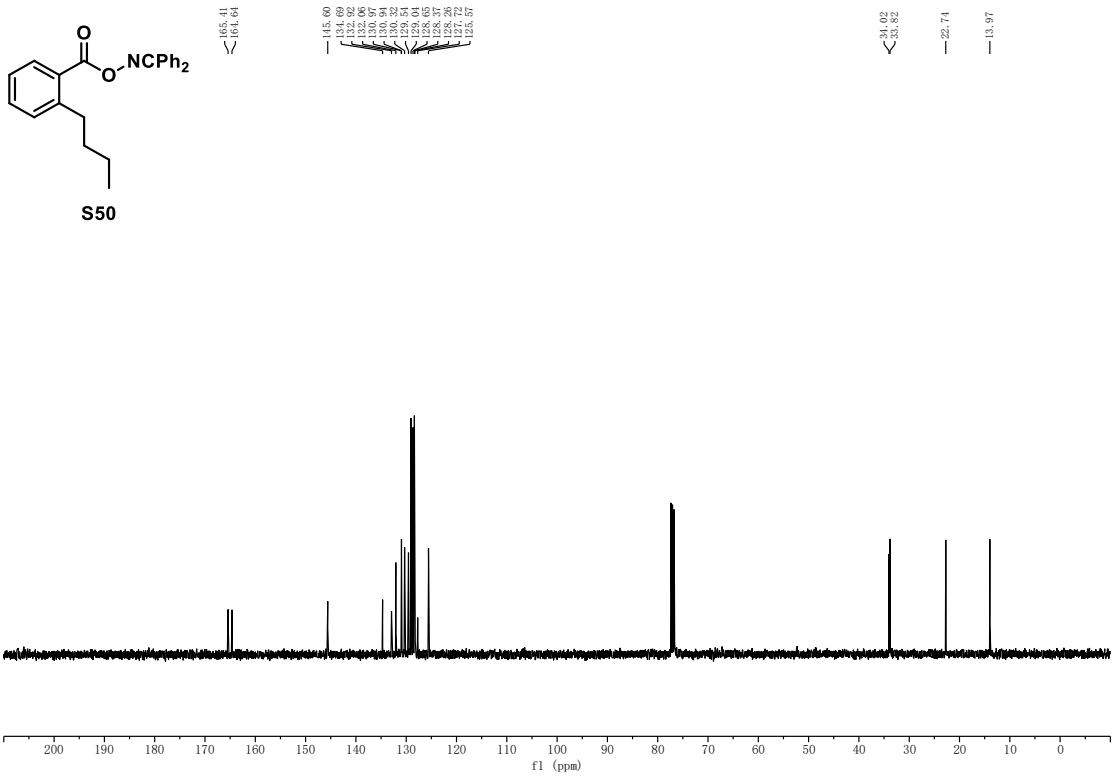

**Supplementary Figure 119** $^{1}$H NMR spectrum of **S48**



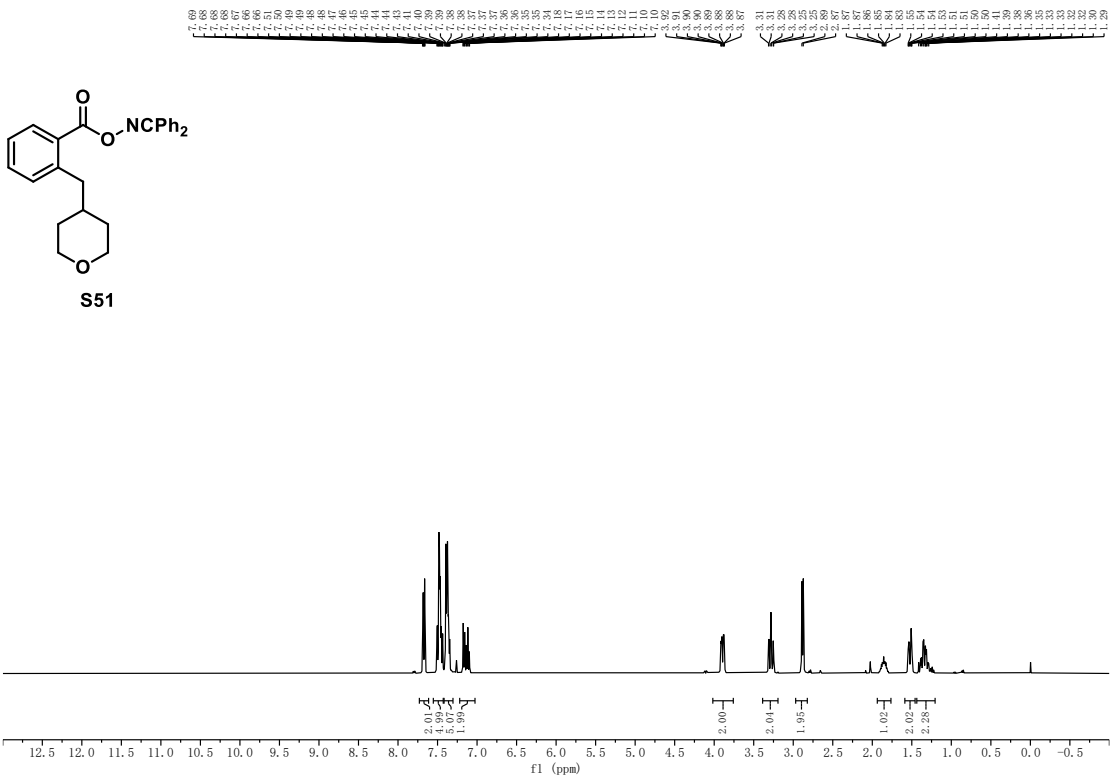

**Supplementary Figure 120** $^{13}$C NMR spectrum of **S48**



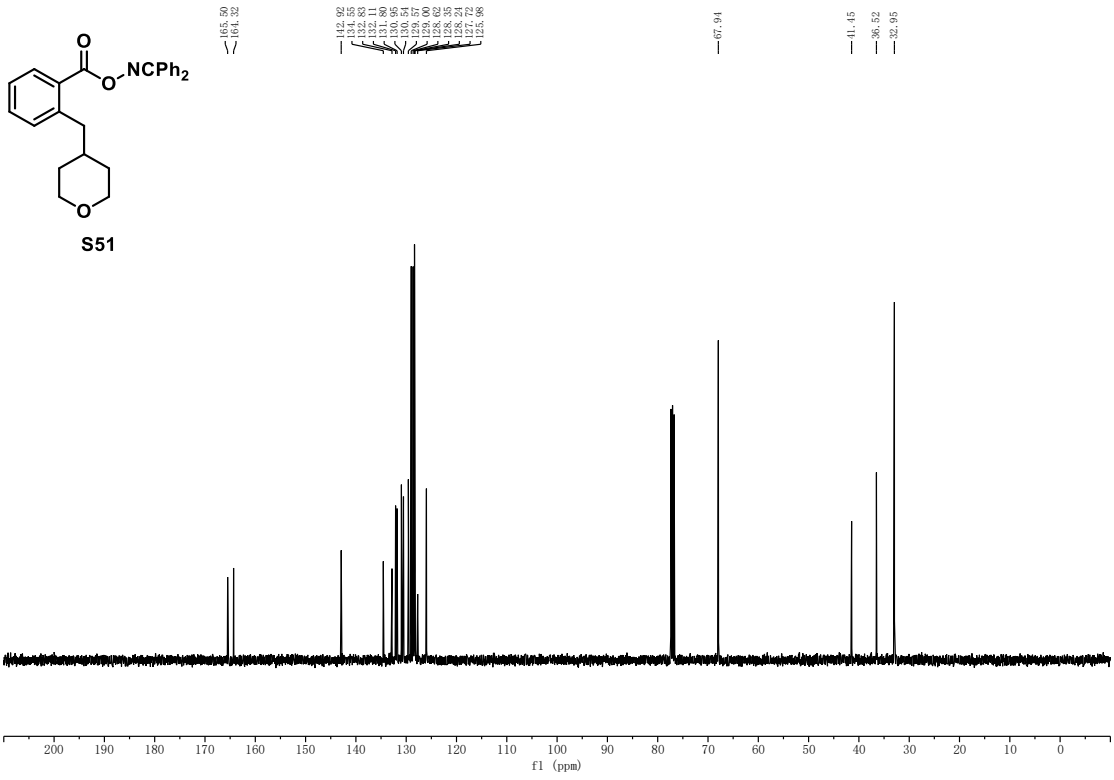

**Supplementary Figure 121** $^{1}$H NMR spectrum of **S50**



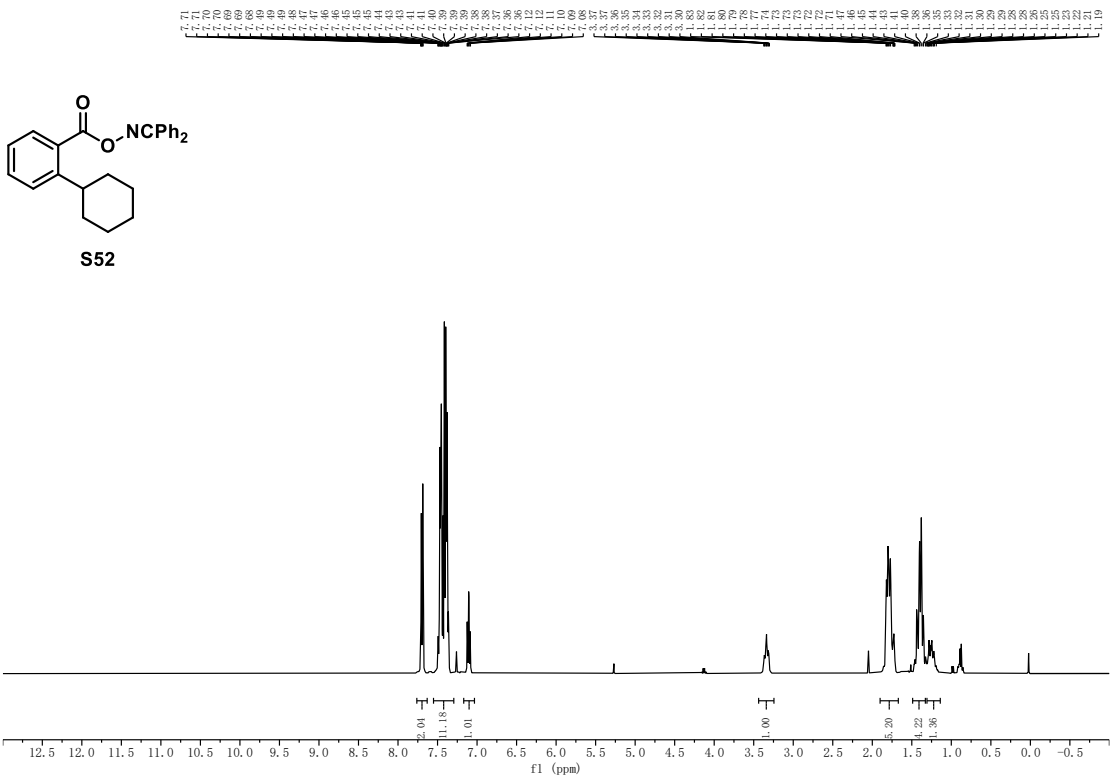

**Supplementary Figure 122** $^{13}$C NMR spectrum of **S50**



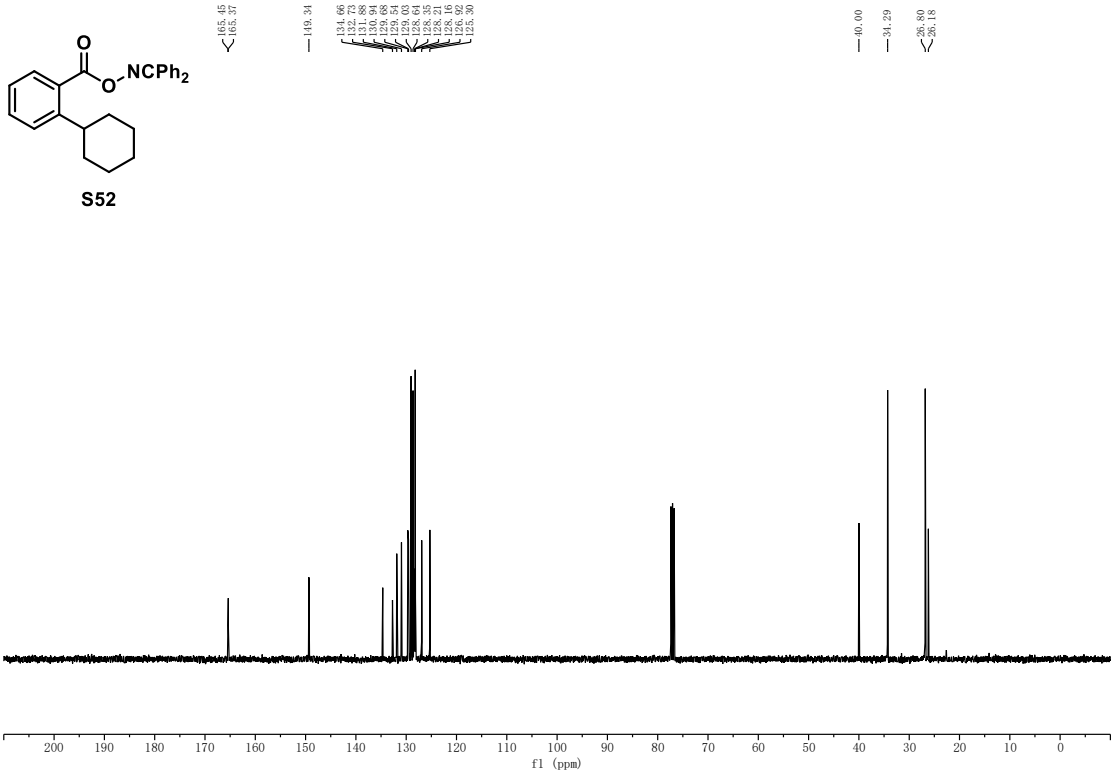

**Supplementary Figure 123** $^{1}$H NMR spectrum of **S51**



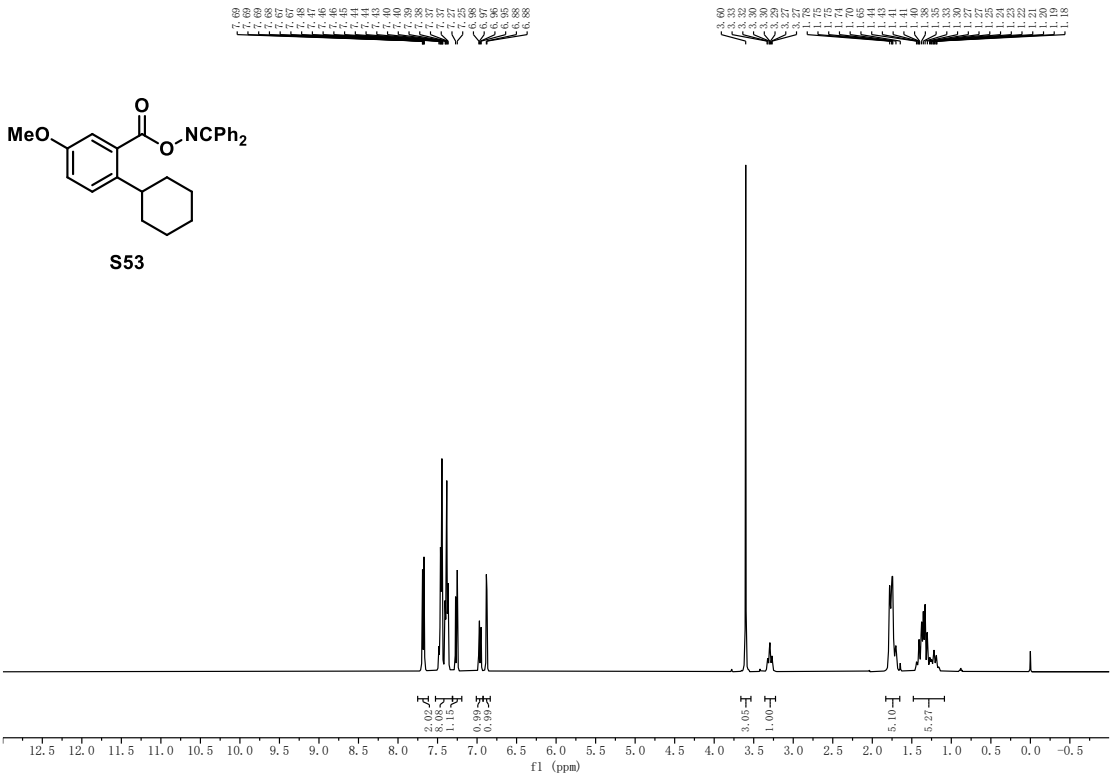

**Supplementary Figure 124** $^{13}$C NMR spectrum of **S51**



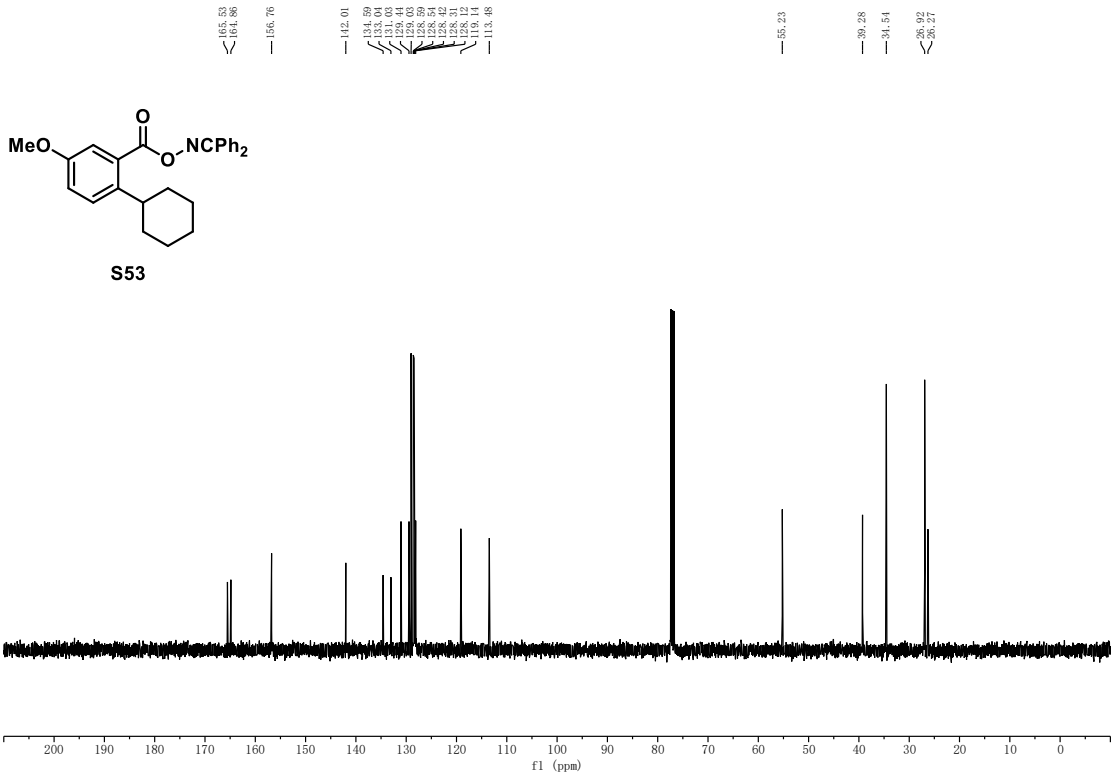

**Supplementary Figure 125** $^{1}$H NMR spectrum of **S52**



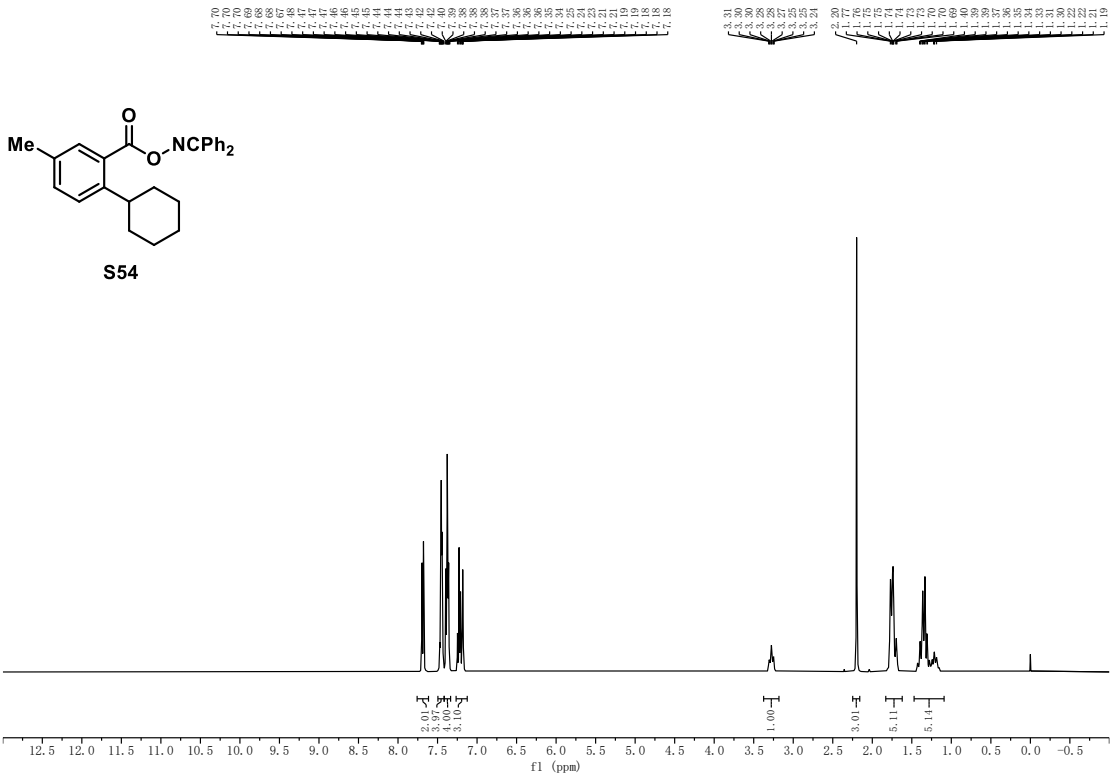

**Supplementary Figure 126** $^{13}$C NMR spectrum of **S52**



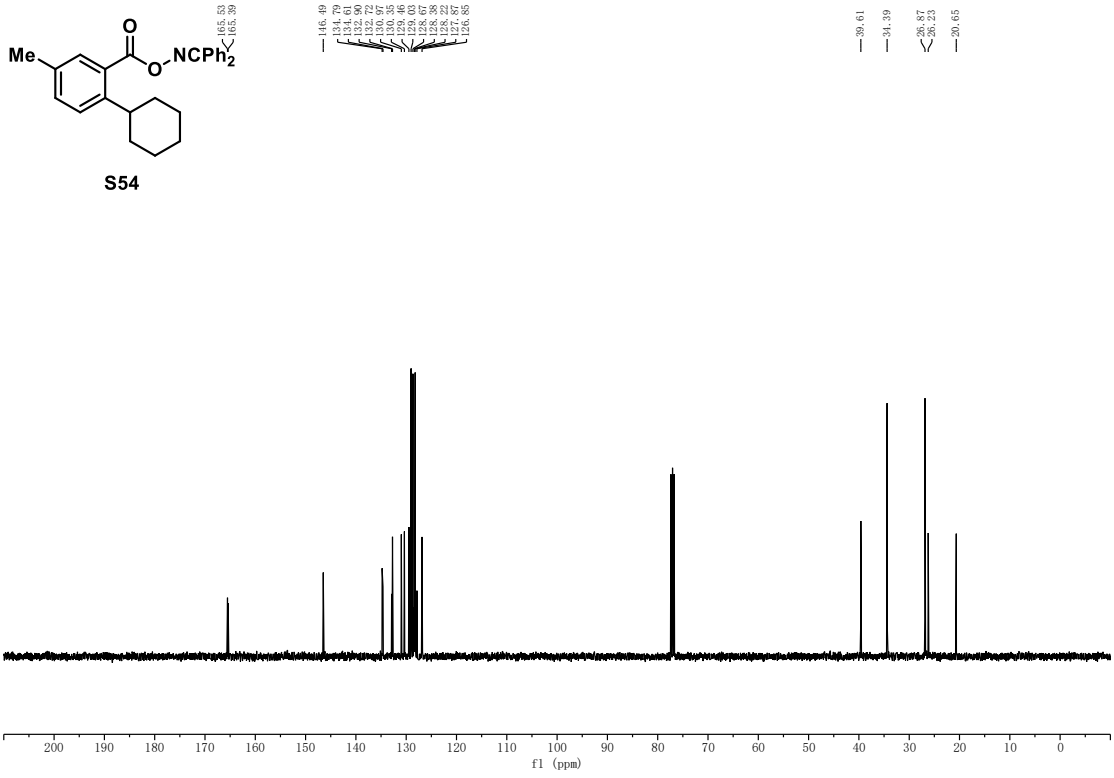

**Supplementary Figure 127** $^{1}$H NMR spectrum of **S53**



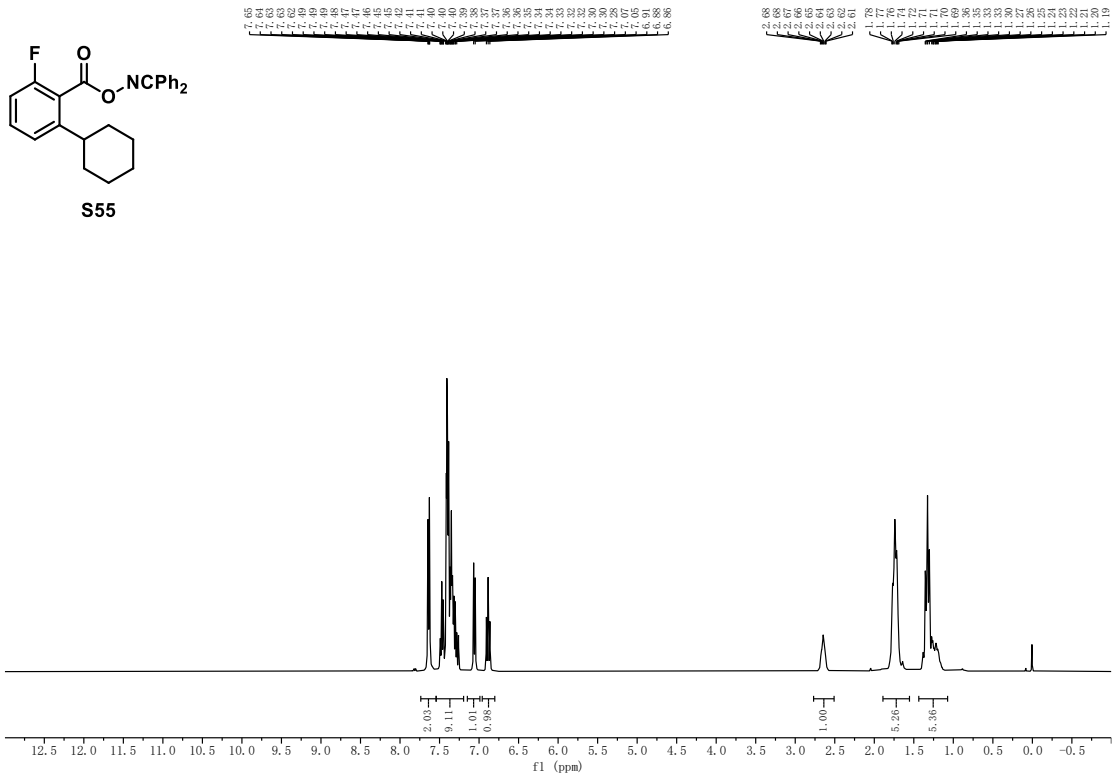

**Supplementary Figure 128** $^{13}$C NMR spectrum of **S53**



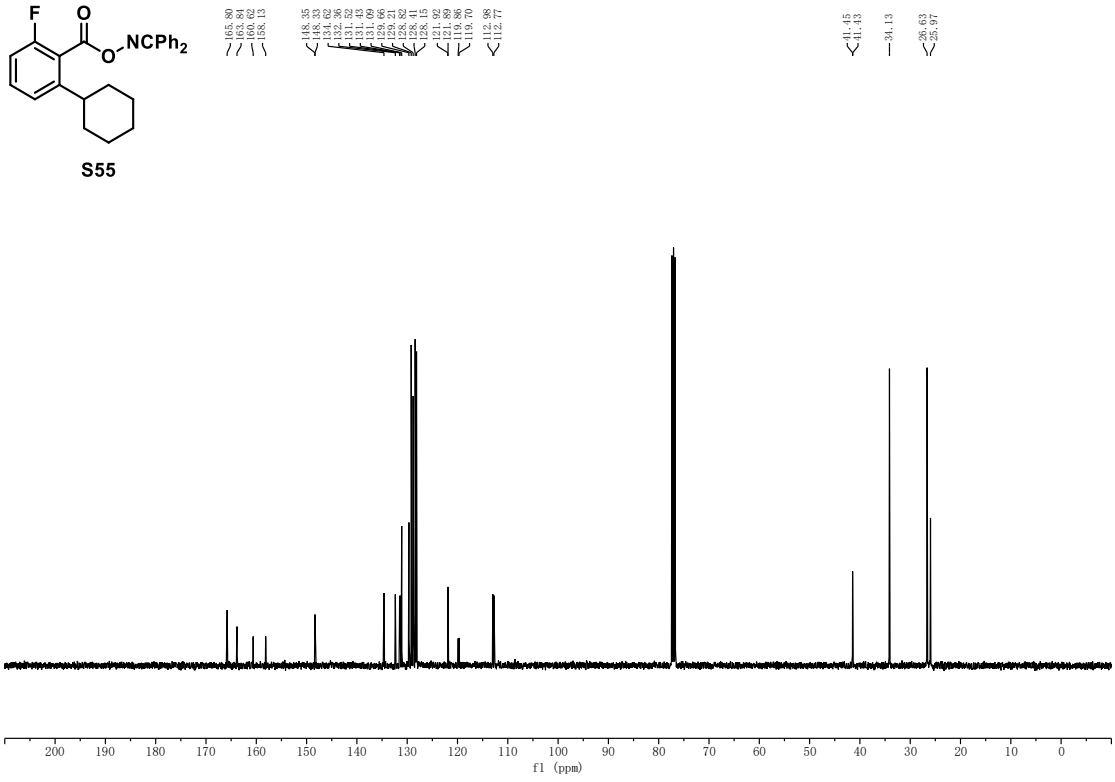

**Supplementary Figure 129** $^{1}$H NMR spectrum of **S54**



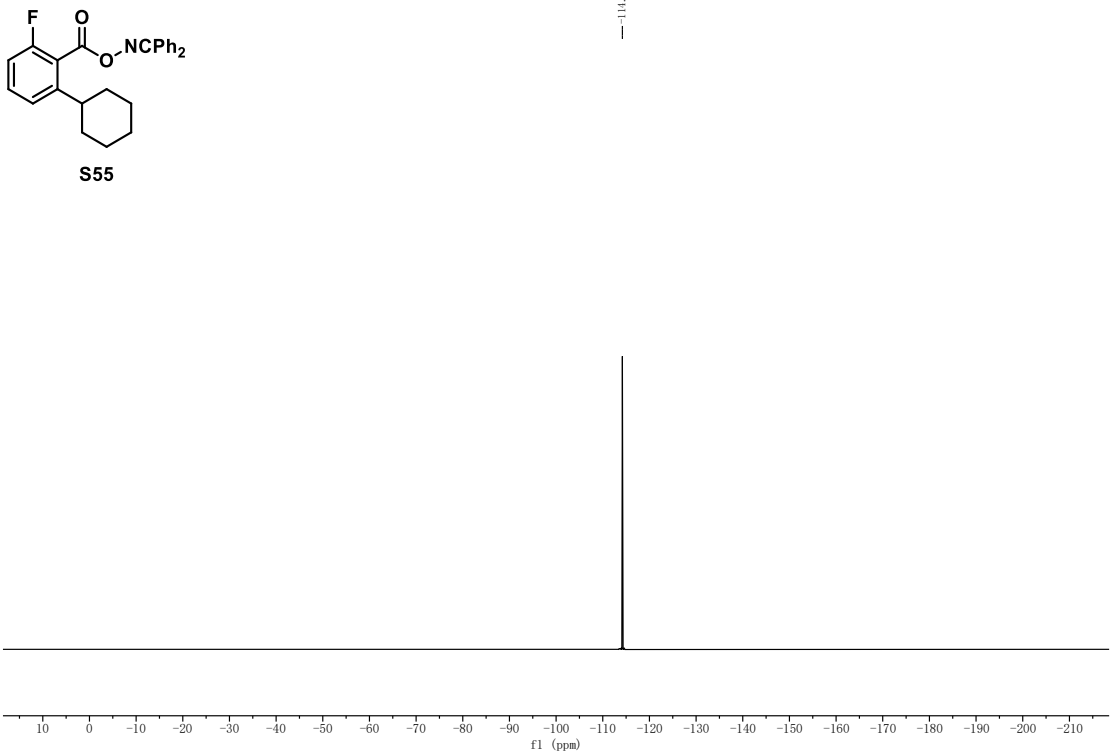

**Supplementary Figure 130** $^{13}$C NMR spectrum of **S54**



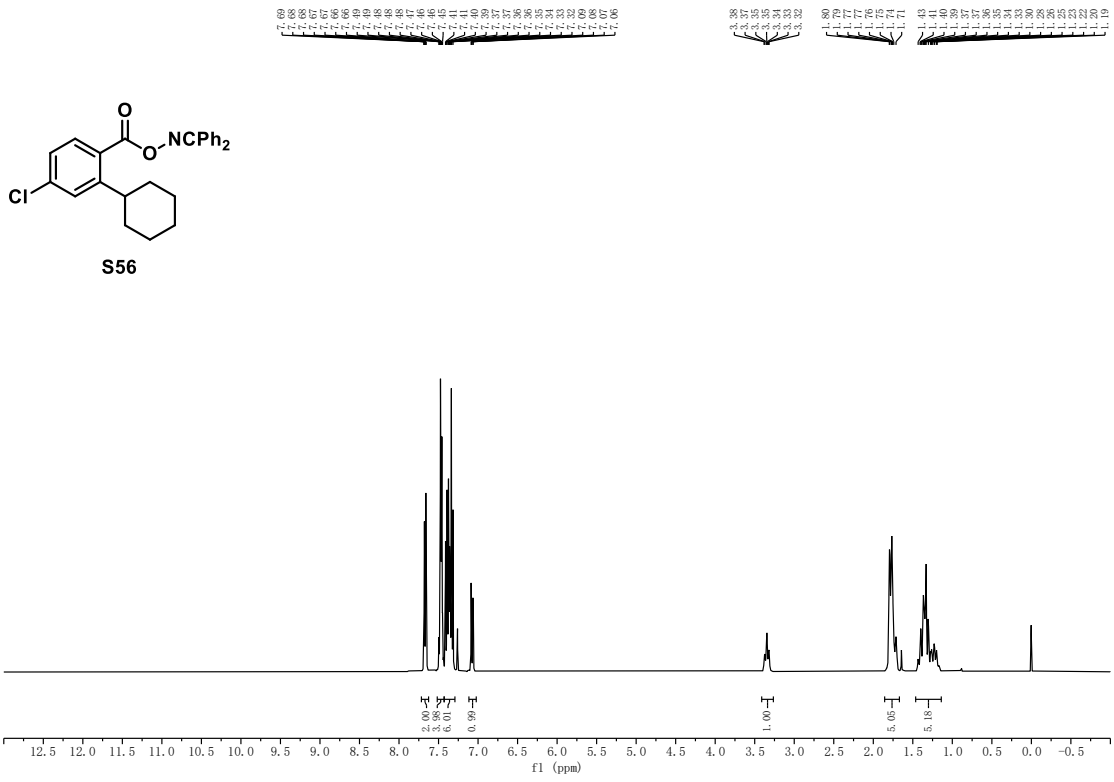

**Supplementary Figure 131** $^{1}$H NMR spectrum of **S55**



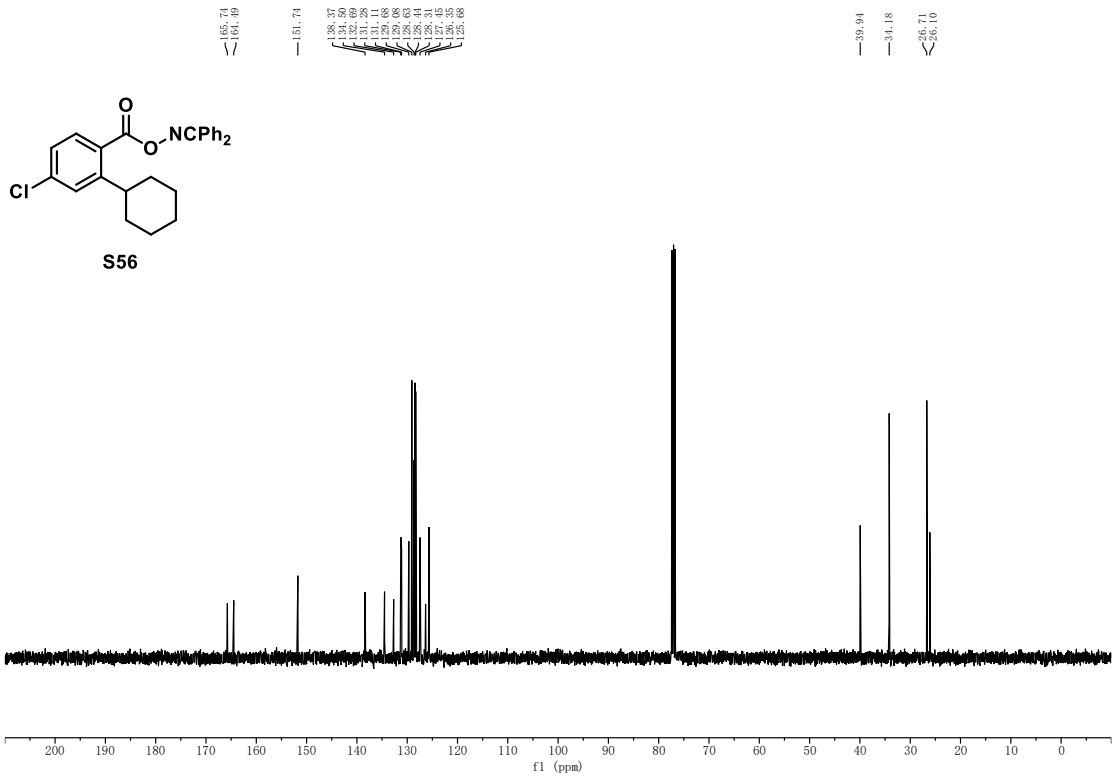

**Supplementary Figure 132** $^{13}$C NMR spectrum of **S55**



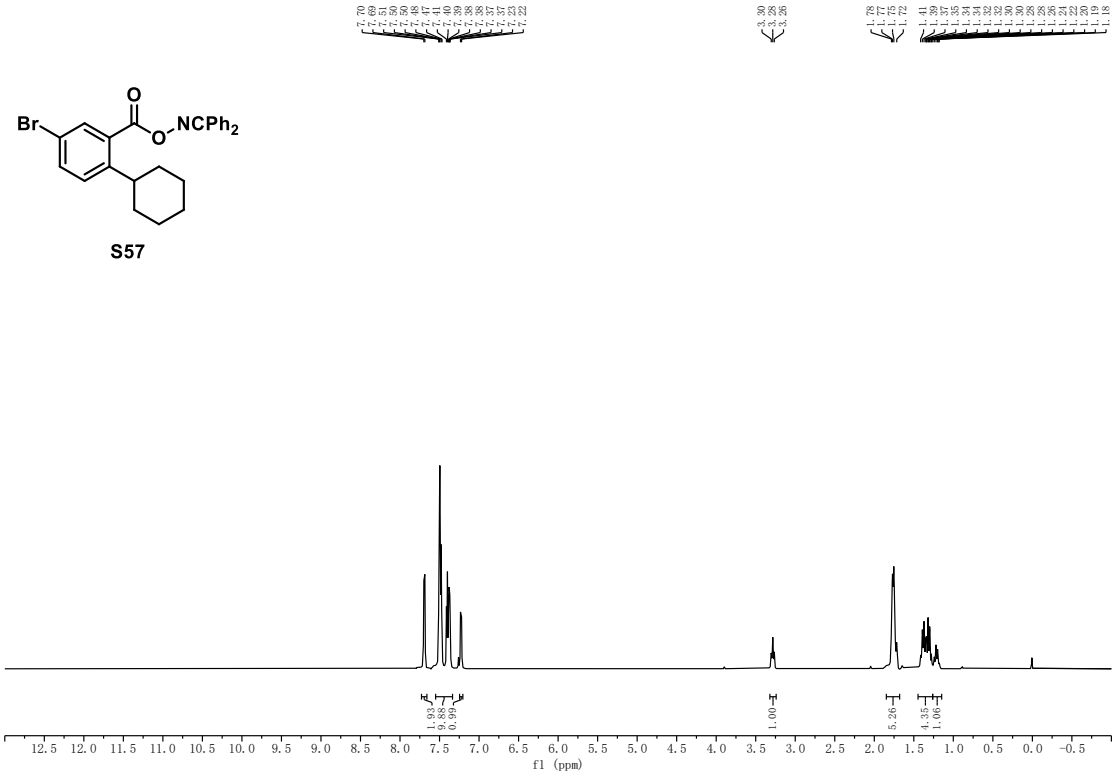

**Supplementary Figure 133** $^{19}$F NMR spectrum of **S55**



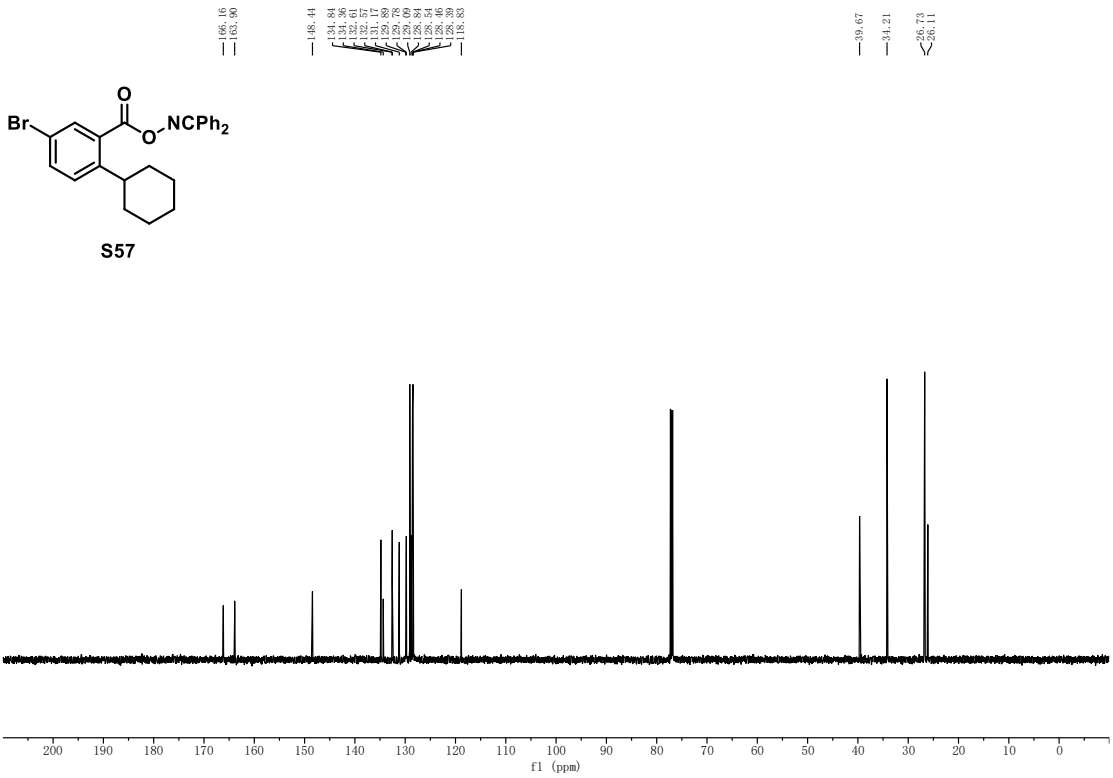

**Supplementary Figure 134** $^{1}$H NMR spectrum of **S56**



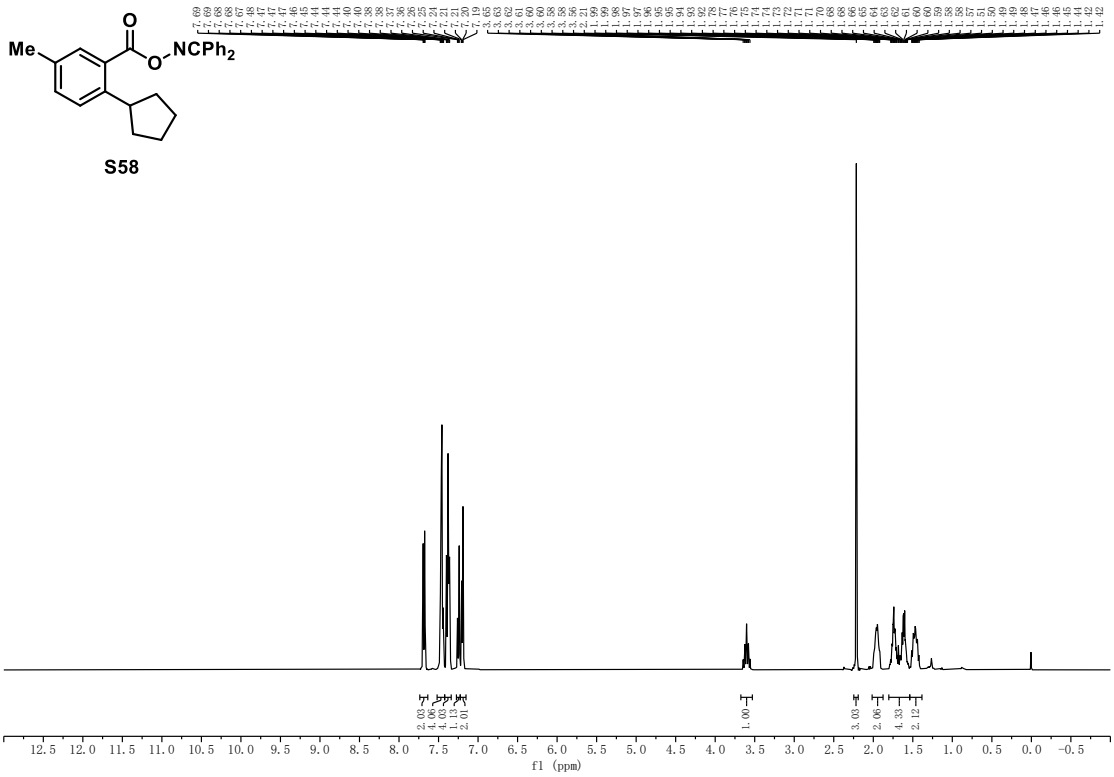

**Supplementary Figure 135** $^{13}$C NMR spectrum of **S56**



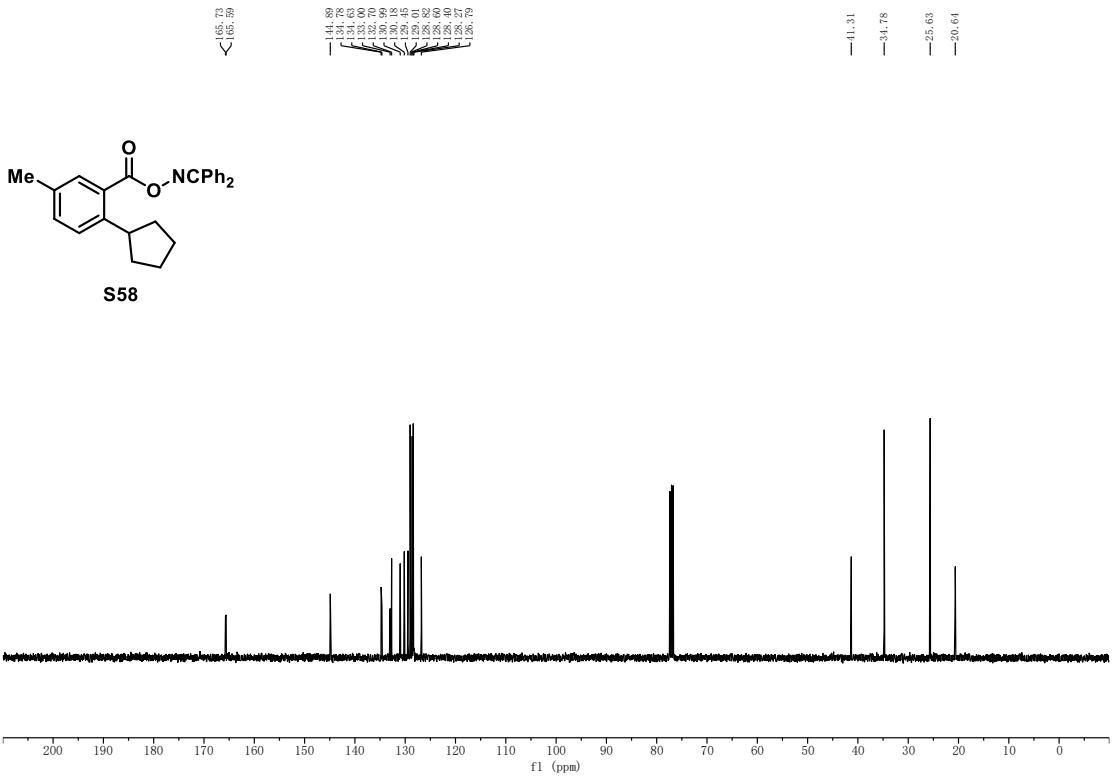

**Supplementary Figure 136** $^{1}$H NMR spectrum of **S57**



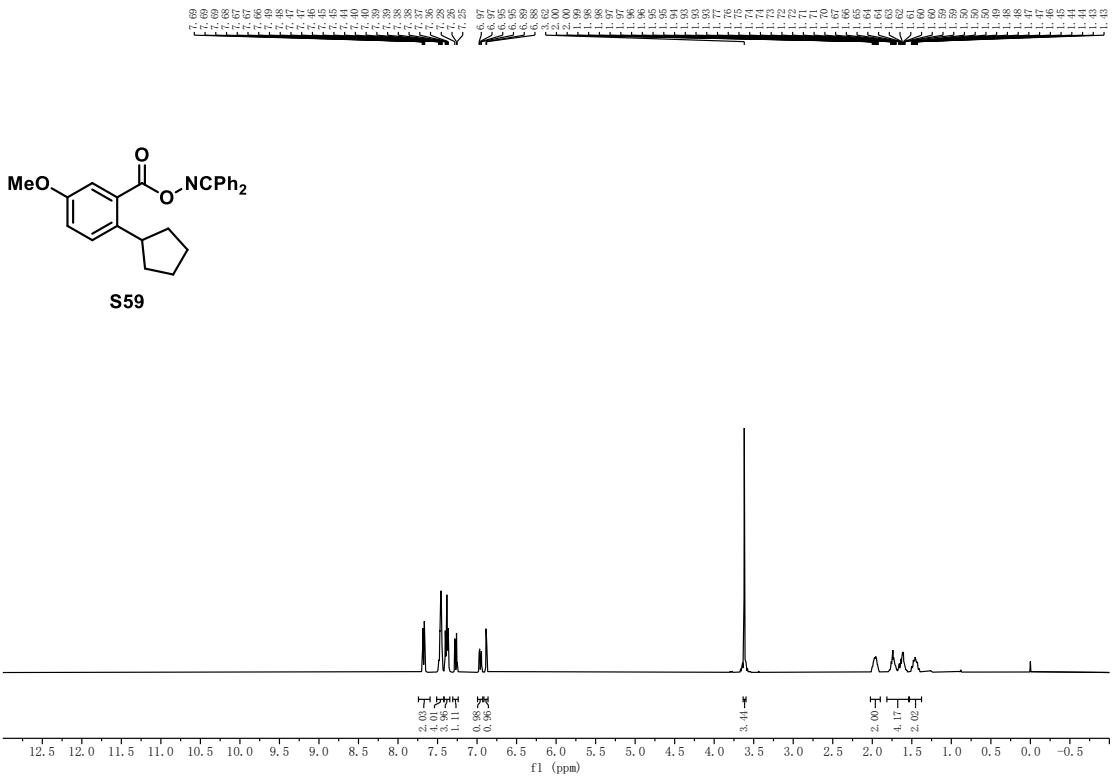

**Supplementary Figure 137** $^{13}$C NMR spectrum of **S57**



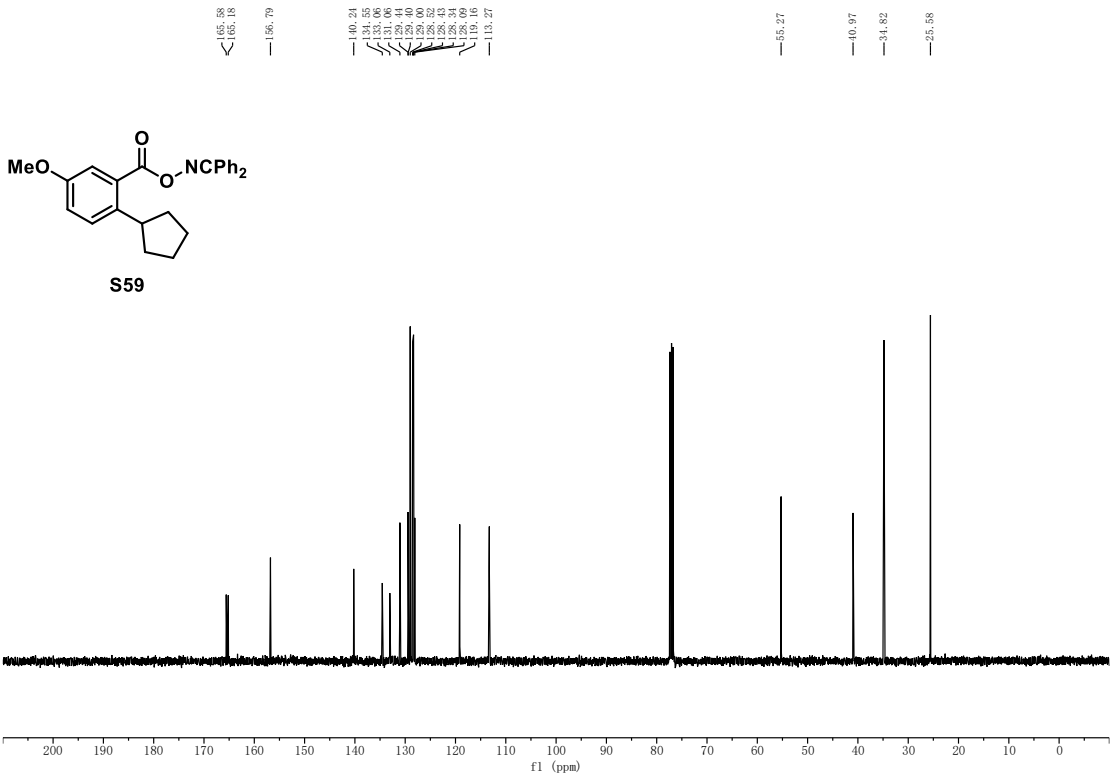

**Supplementary Figure 138** $^{1}$H NMR spectrum of **S58**



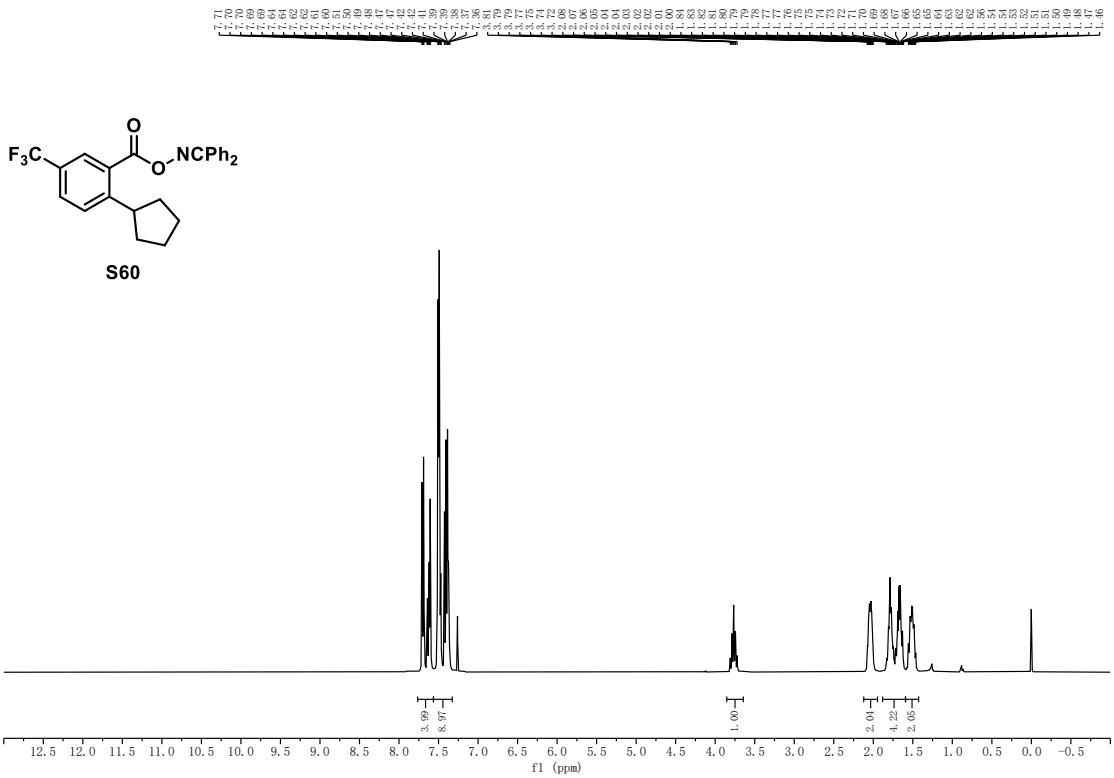

**Supplementary Figure 139** $^{13}$C NMR spectrum of **S58**



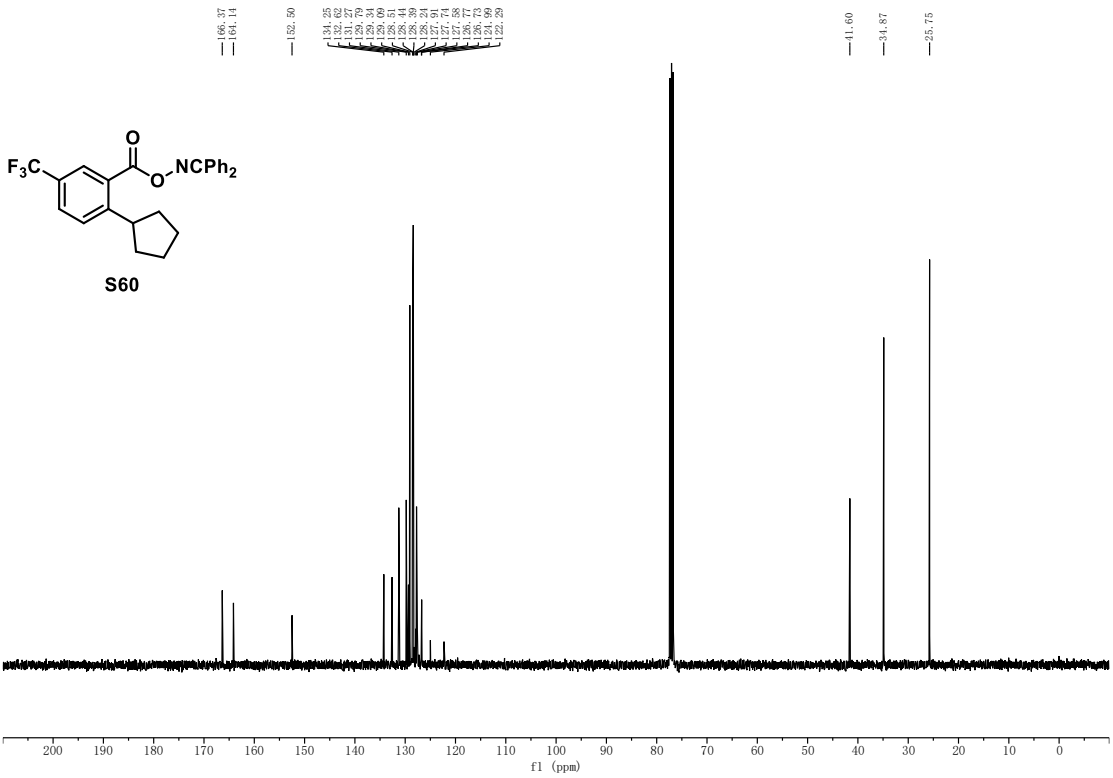

**Supplementary Figure 140** $^{1}$H NMR spectrum of **S59**



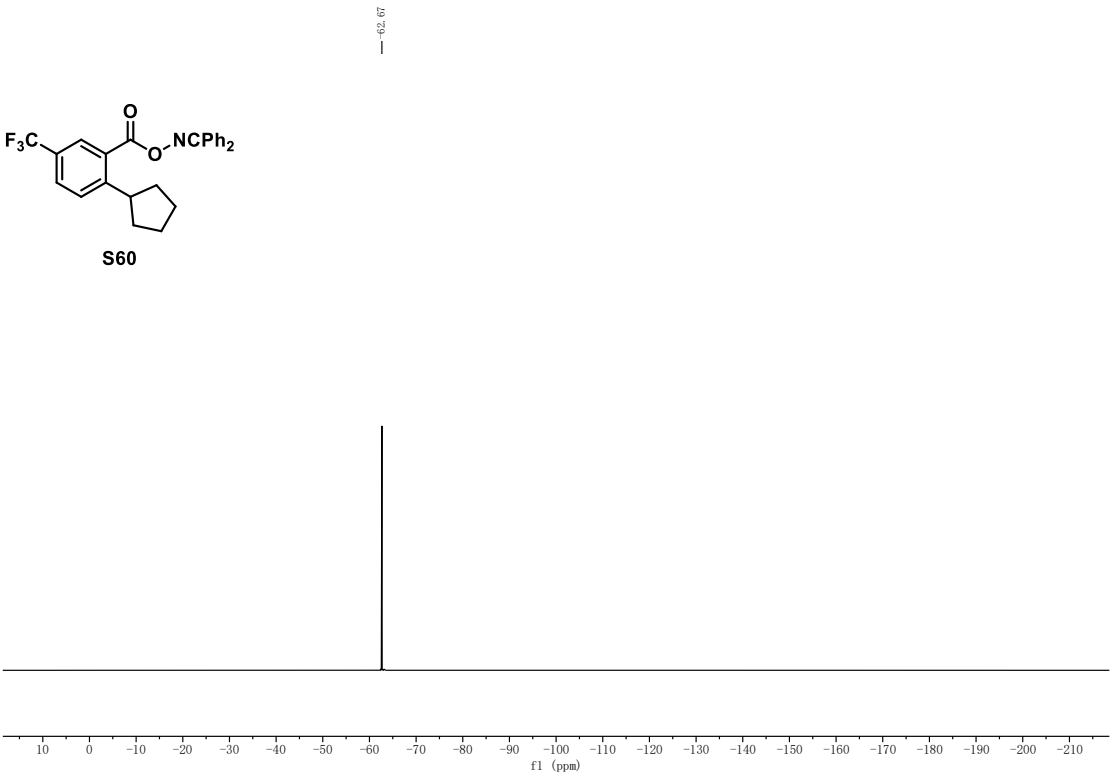

**Supplementary Figure 141** $^{13}$C NMR spectrum of **S59**



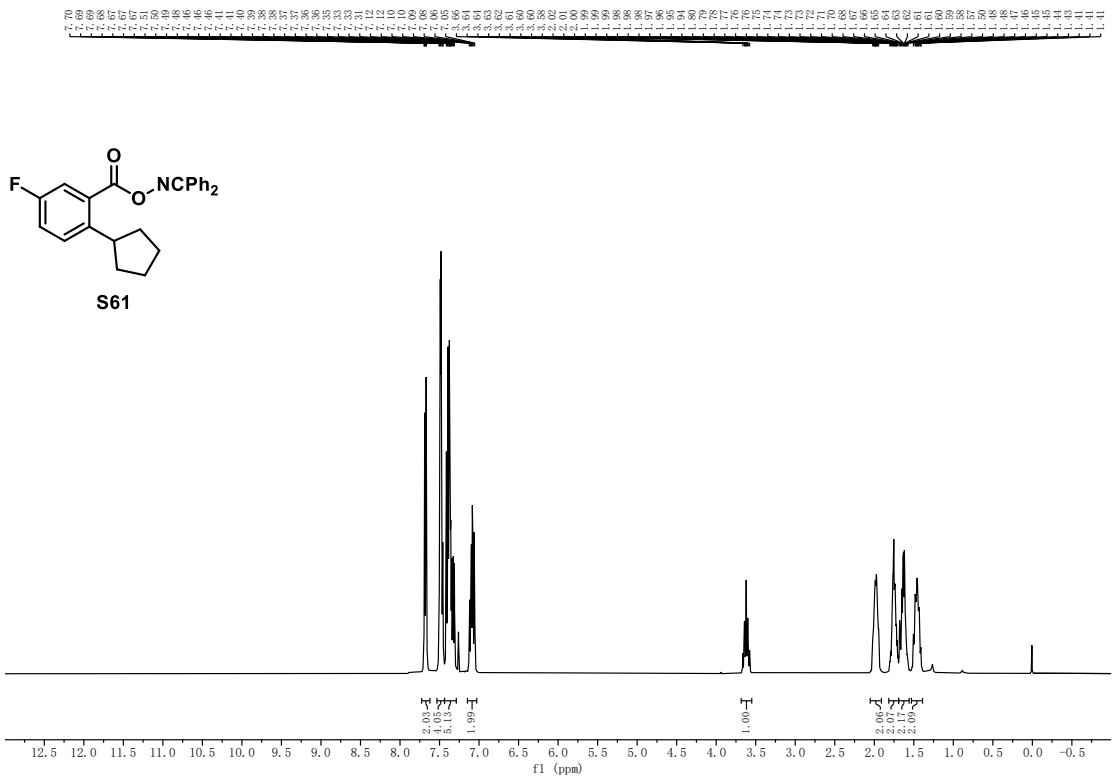

**Supplementary Figure 142** $^{1}$H NMR spectrum of **S60**



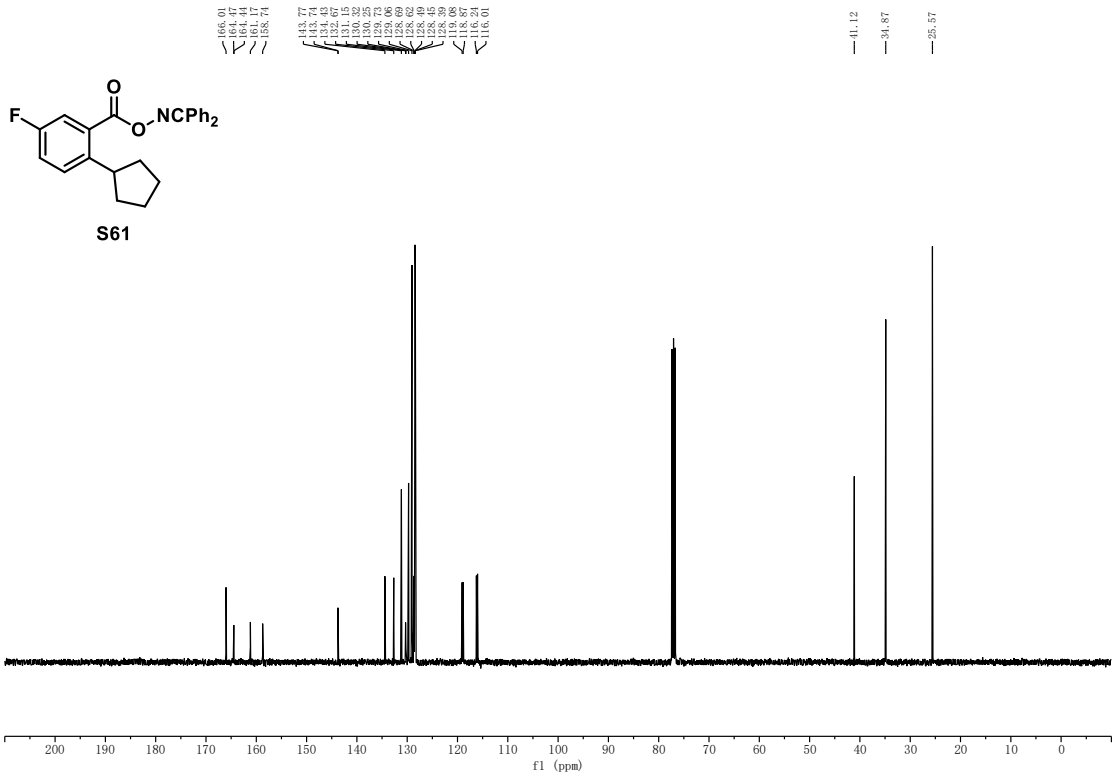

**Supplementary Figure 143** $^{13}$C NMR spectrum of **S60**



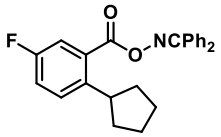

**Supplementary Figure 144** $^{19}$F NMR spectrum of **S60**




**Supplementary Figure 145** $^{1}$H NMR spectrum of **S61**



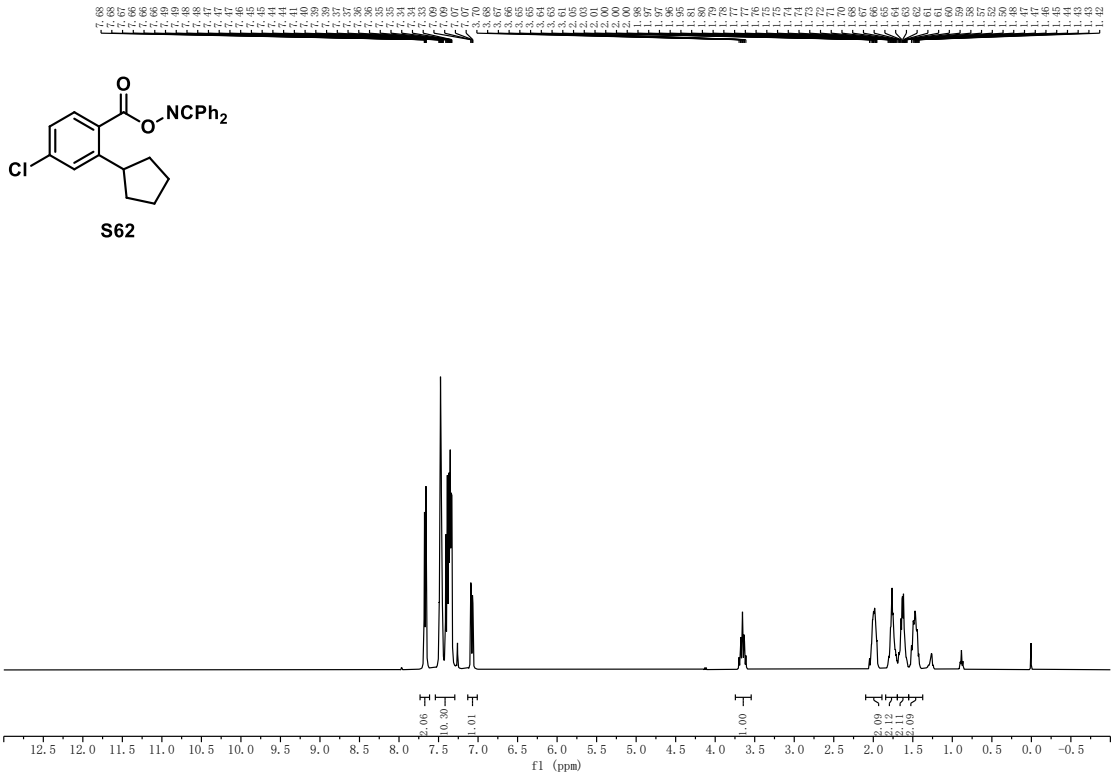

**Supplementary Figure 146** $^{13}$C NMR spectrum of **S61**




S61

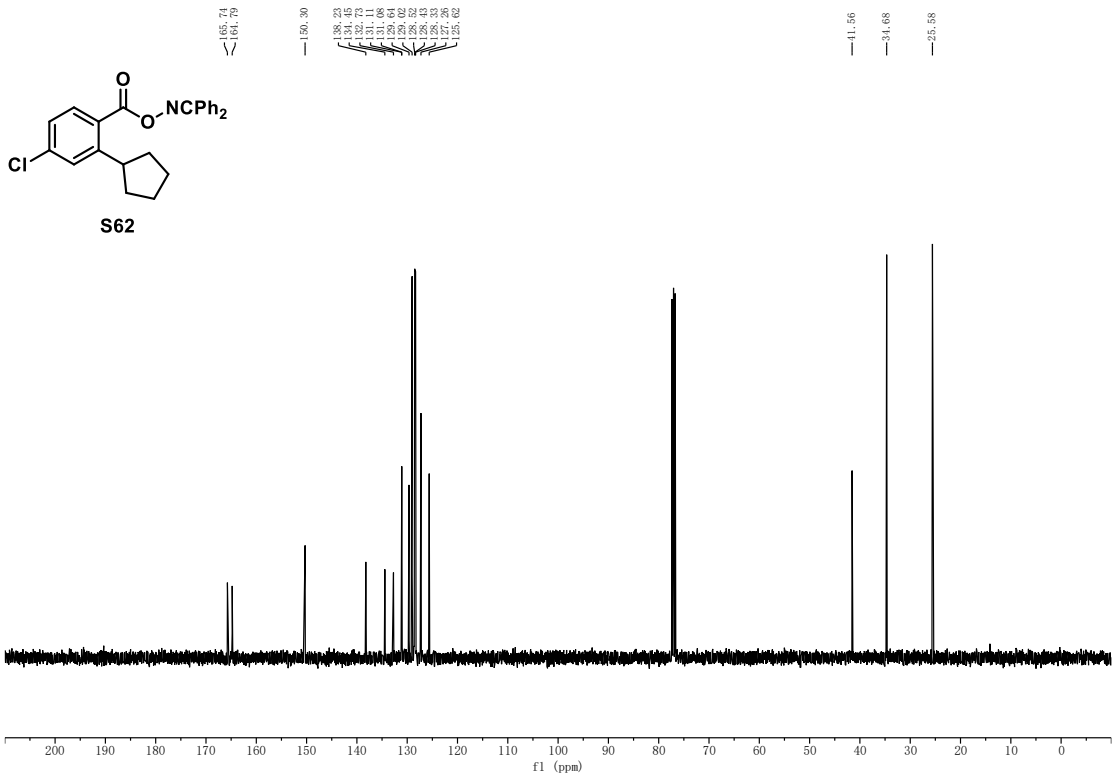



**Supplementary Figure 147** $^{19}$F NMR spectrum of **S61**

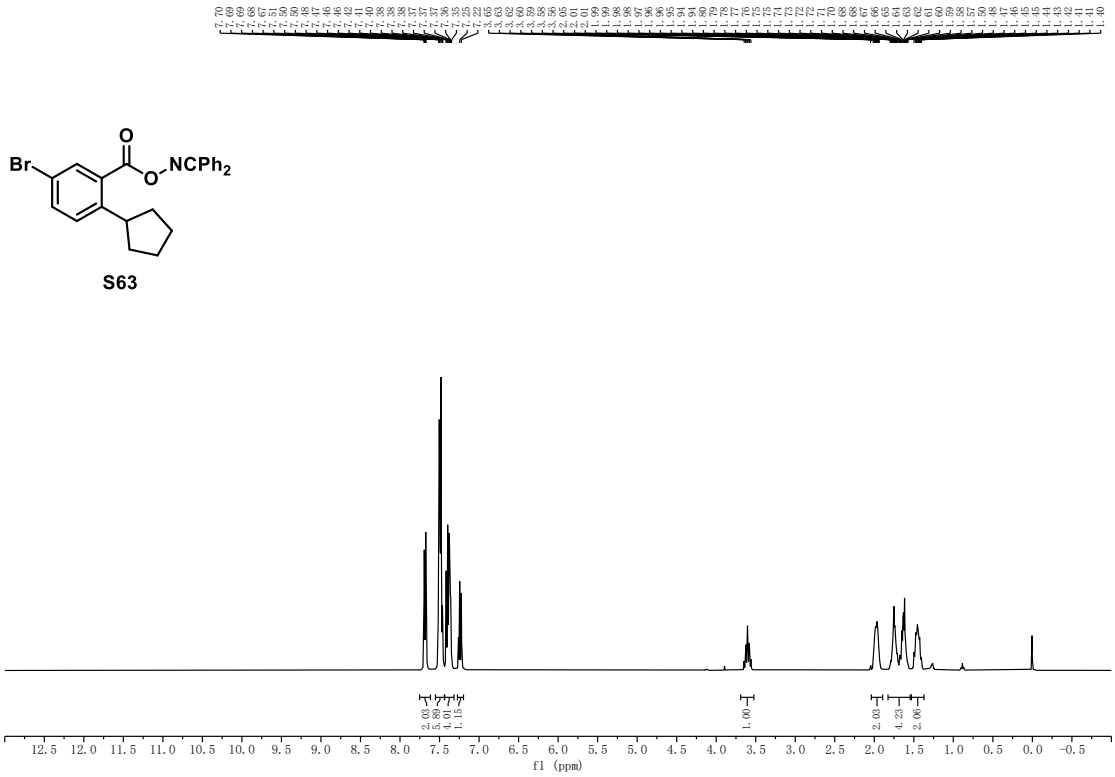



S62

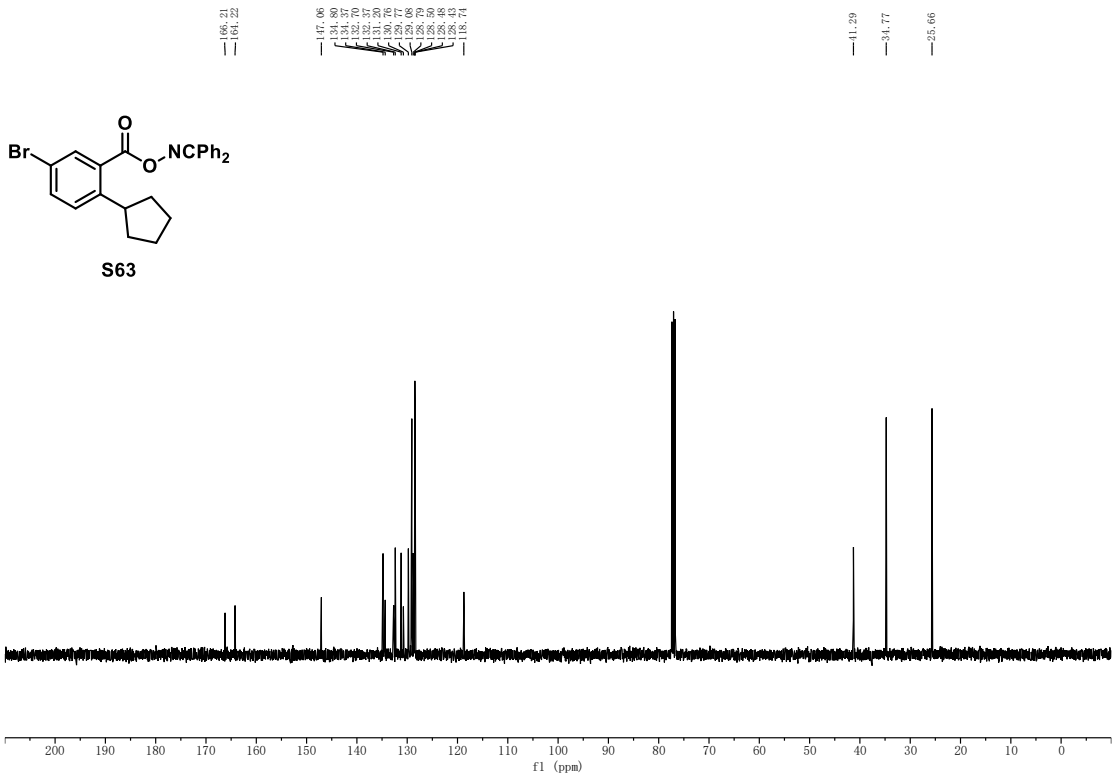
**Supplementary Figure 148** $^{1}$H NMR spectrum of **S62**



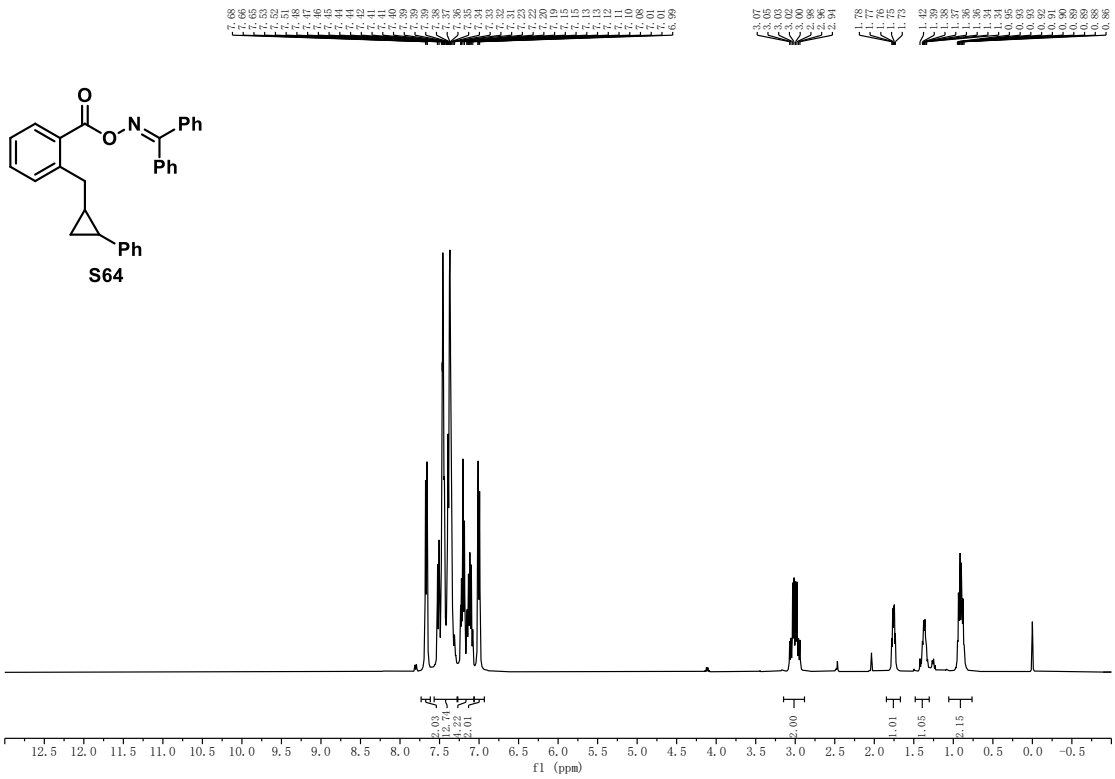

**Supplementary Figure 149** $^{13}$C NMR spectrum of **S62**



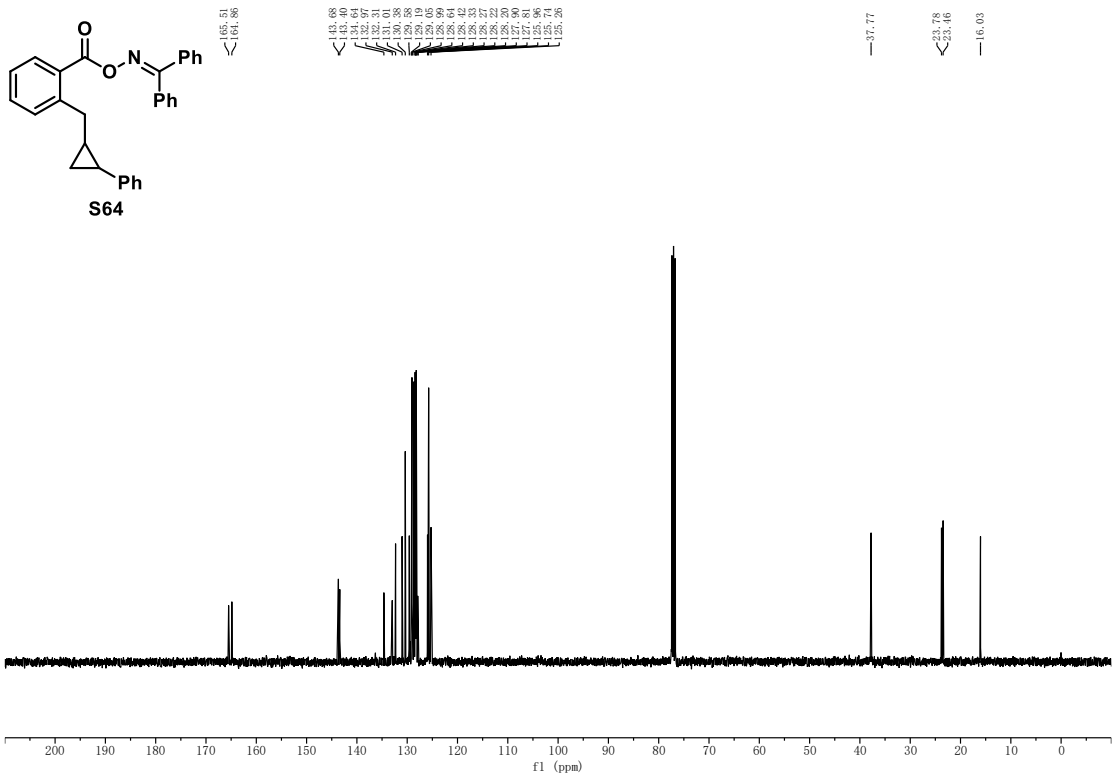

**Supplementary Figure 150** $^{1}$H NMR spectrum of **S63**



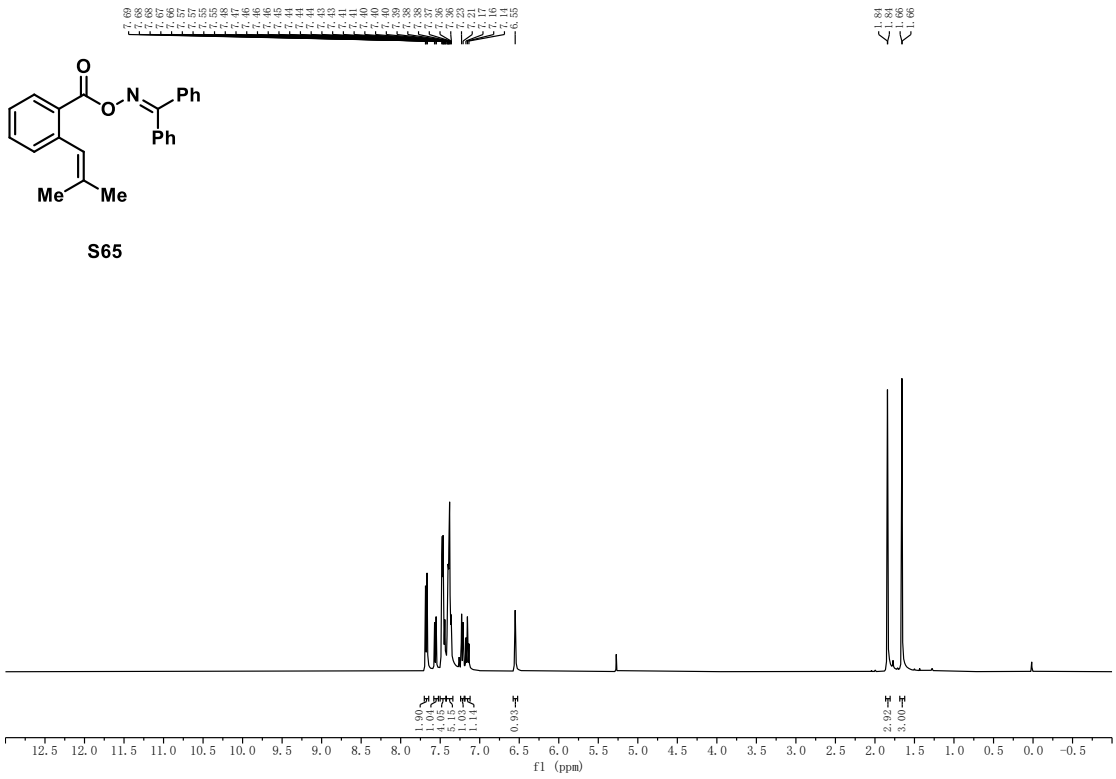

**Supplementary Figure 151** $^{13}$C NMR spectrum of **S63**



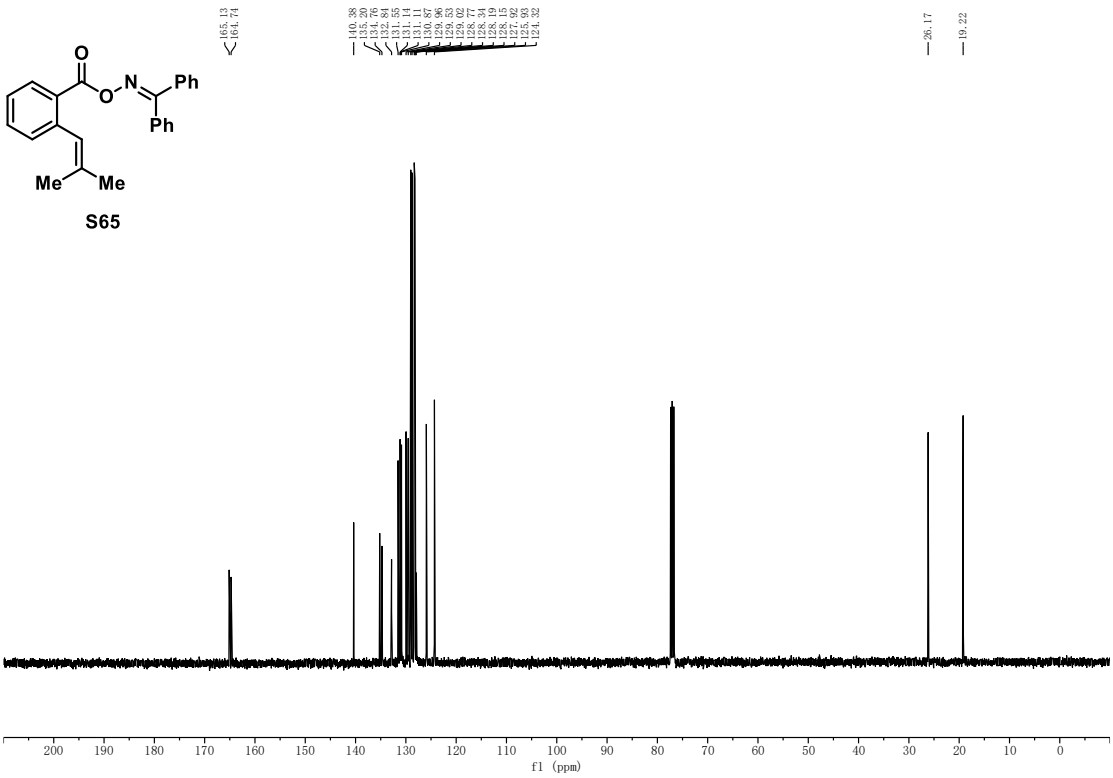

**Supplementary Figure 152** $^{1}$H NMR spectrum of **S64**



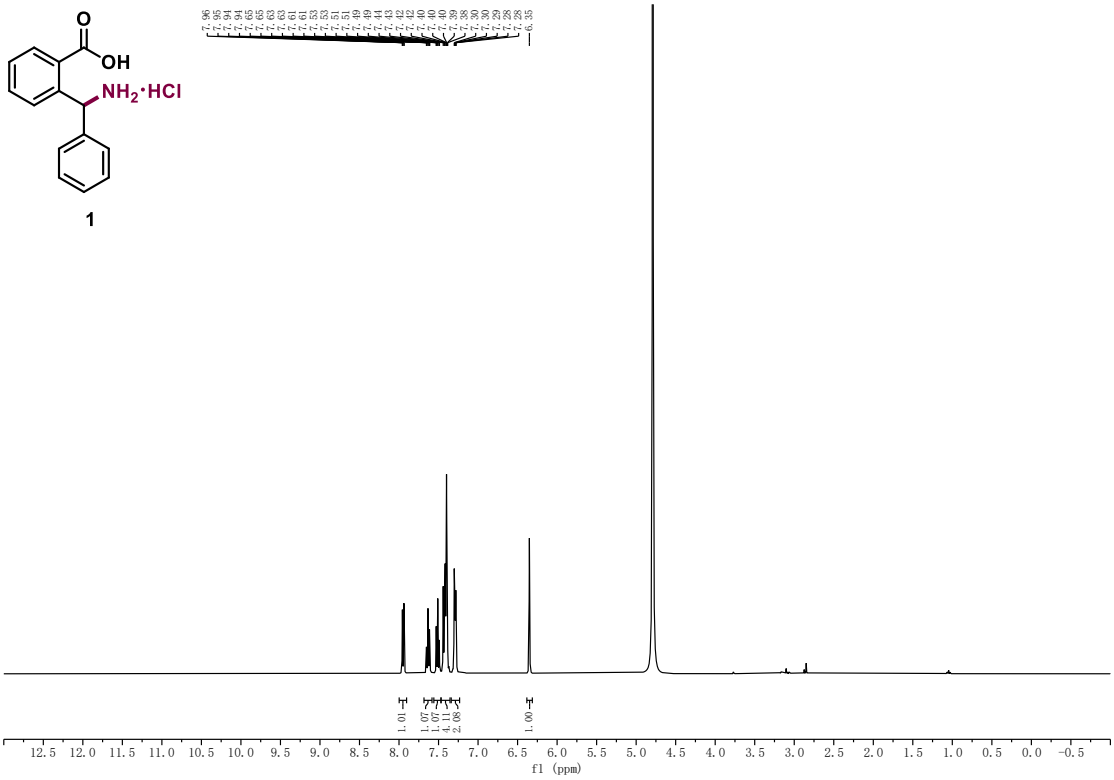

**Supplementary Figure 153** $^{13}$C NMR spectrum of **S64**



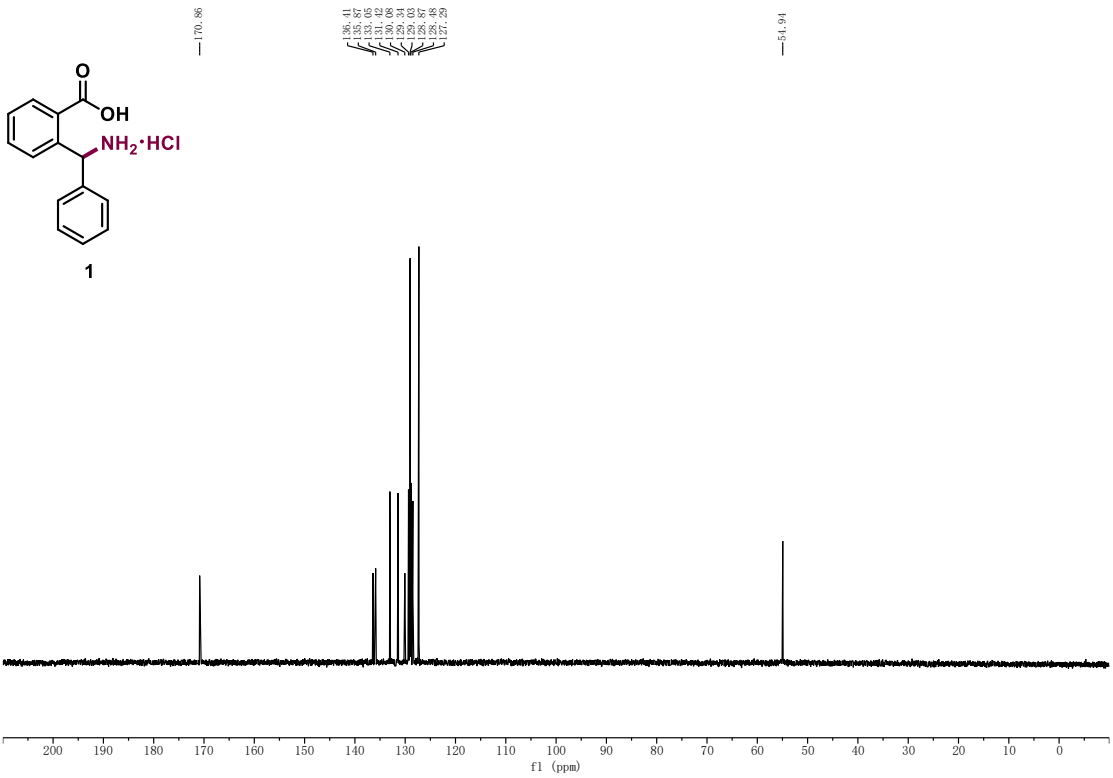

**Supplementary Figure 154** $^{1}$H NMR spectrum of **S65**



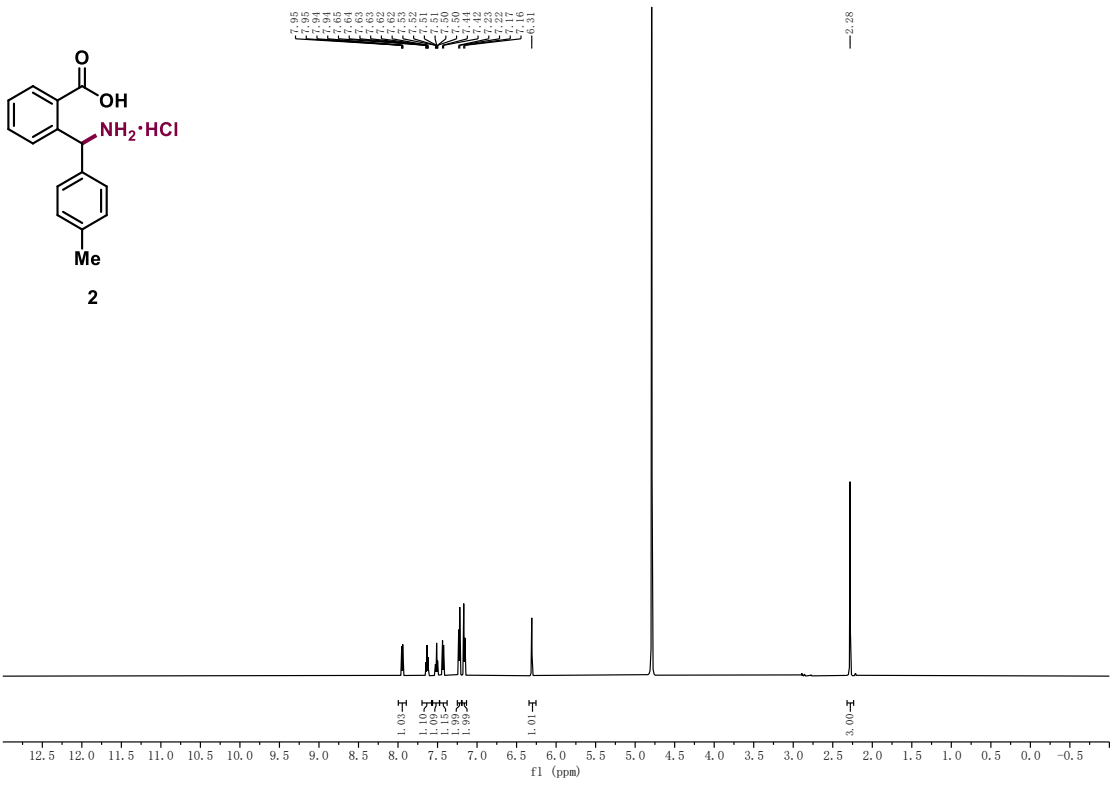

**Supplementary Figure 155** $^{13}$C NMR spectrum of **S65**



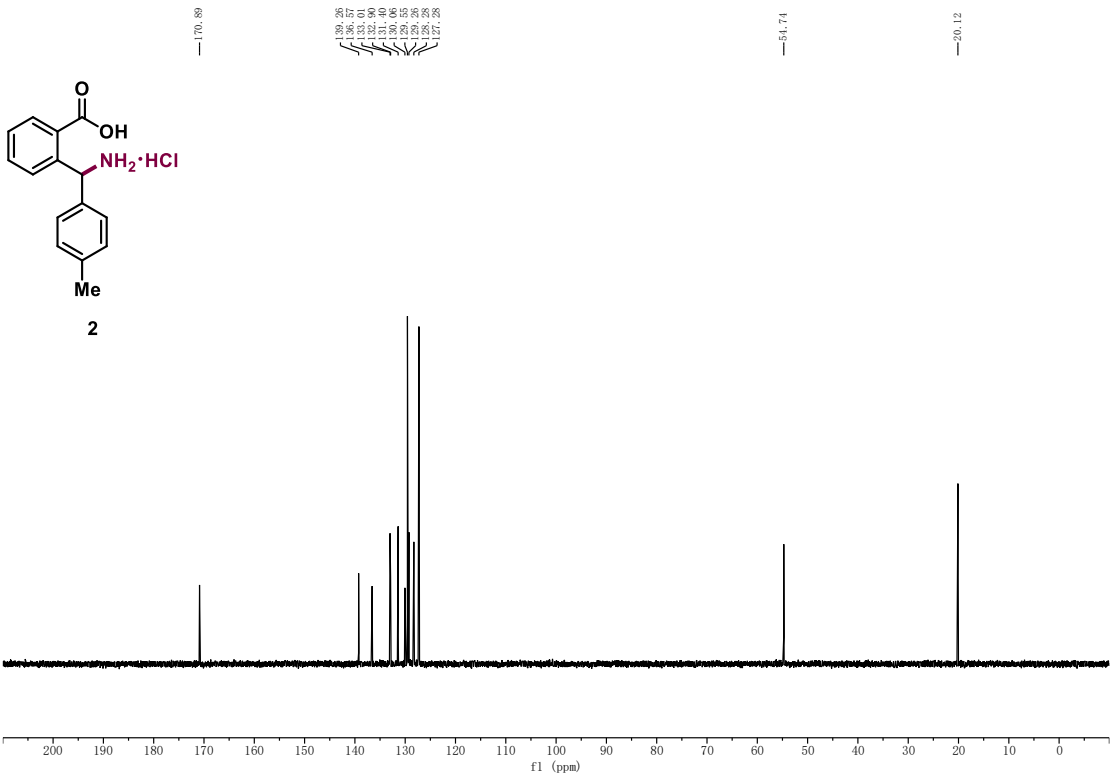

**Supplementary Figure 156** $^{1}$H NMR spectrum of compound **1**



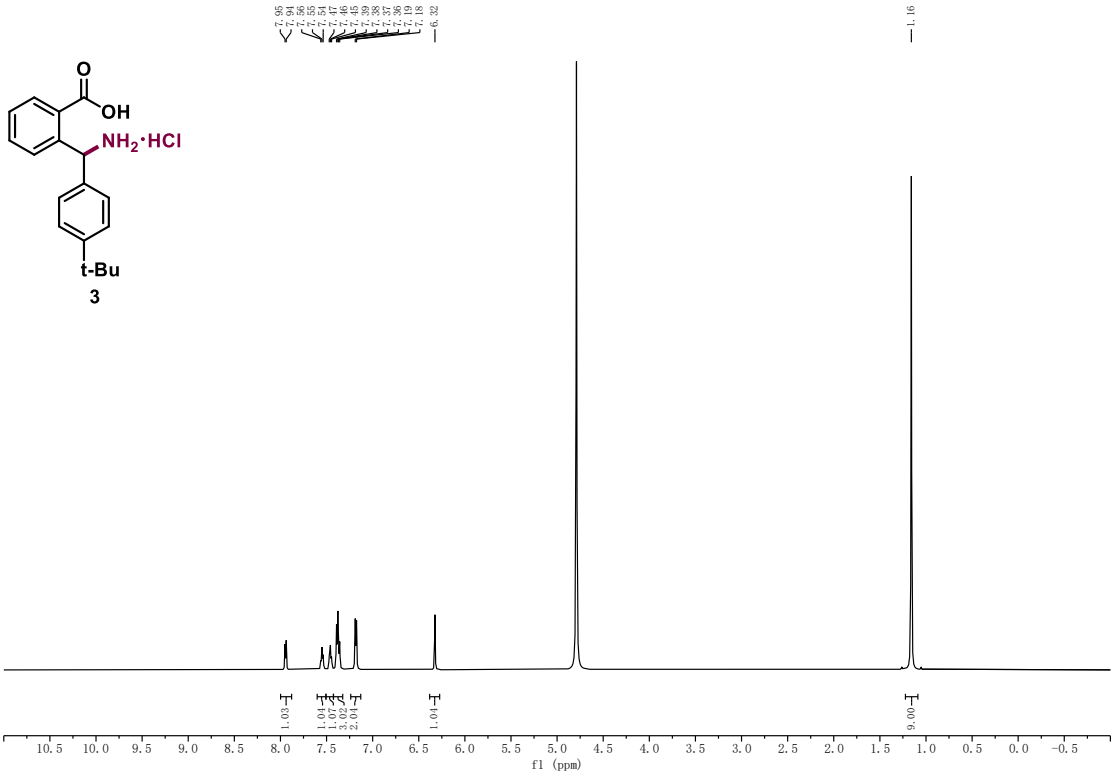

**Supplementary Figure 157** $^{13}$C NMR spectrum of compound **1**



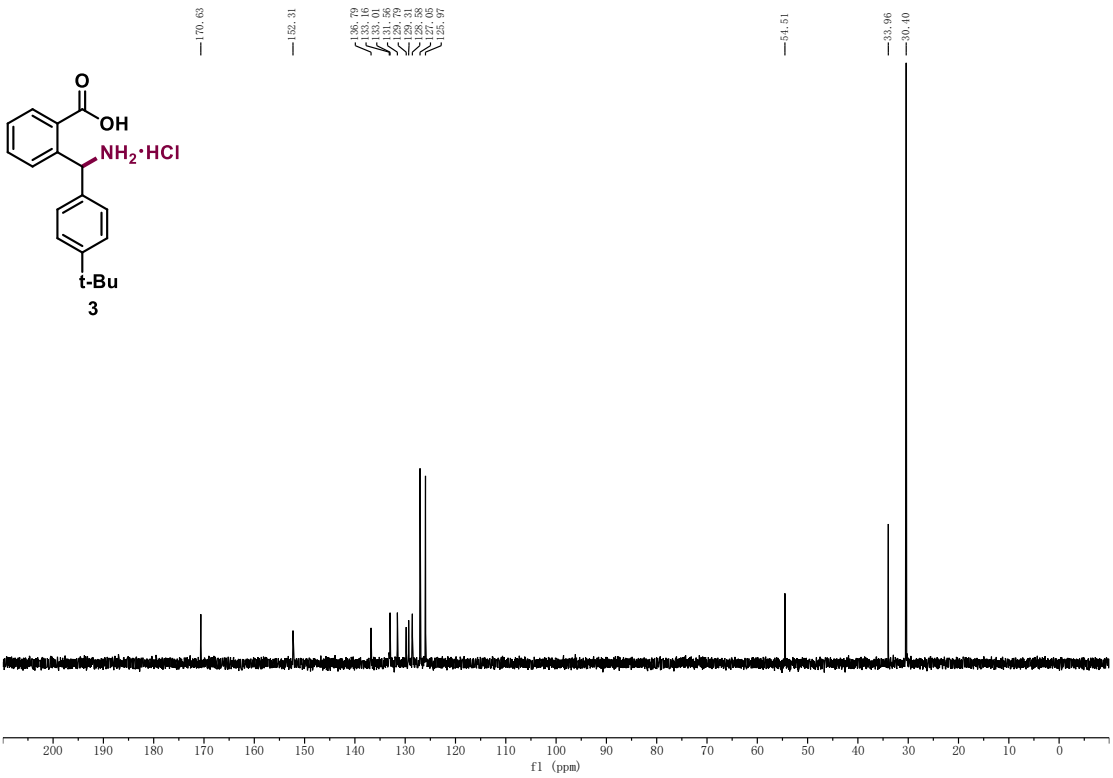

**Supplementary Figure 158** $^{1}$H NMR spectrum of compound **2**



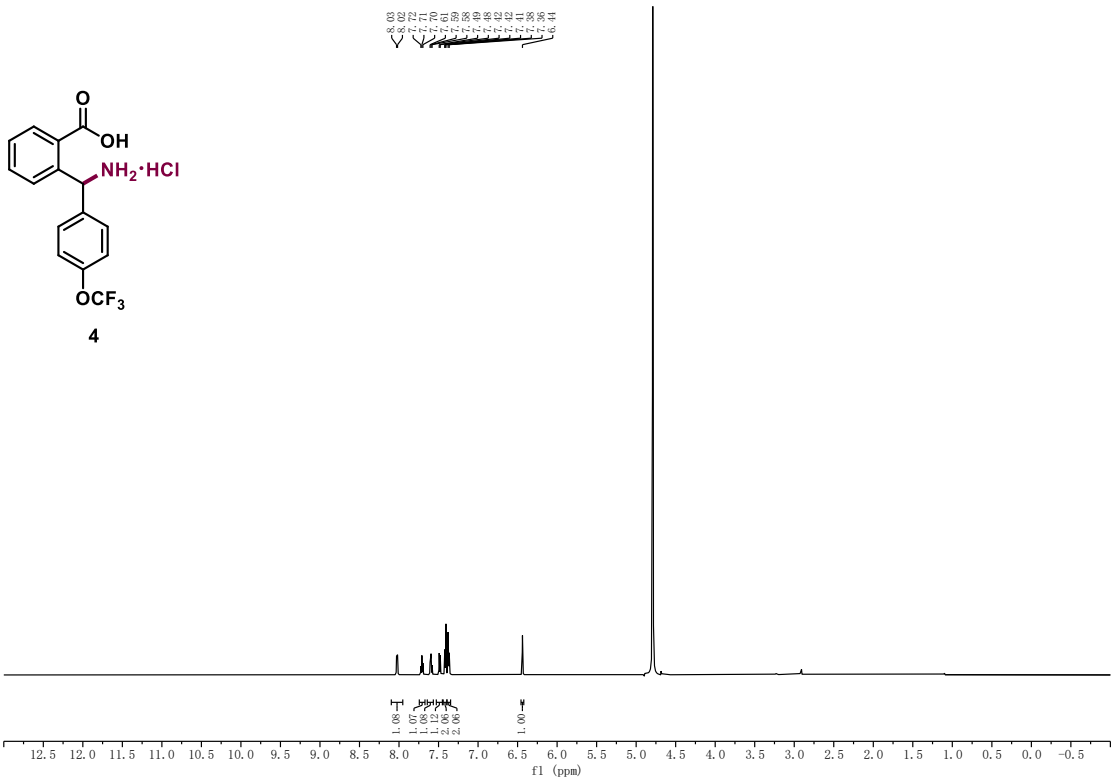

**Supplementary Figure 159** $^{13}$C NMR spectrum of compound **2**



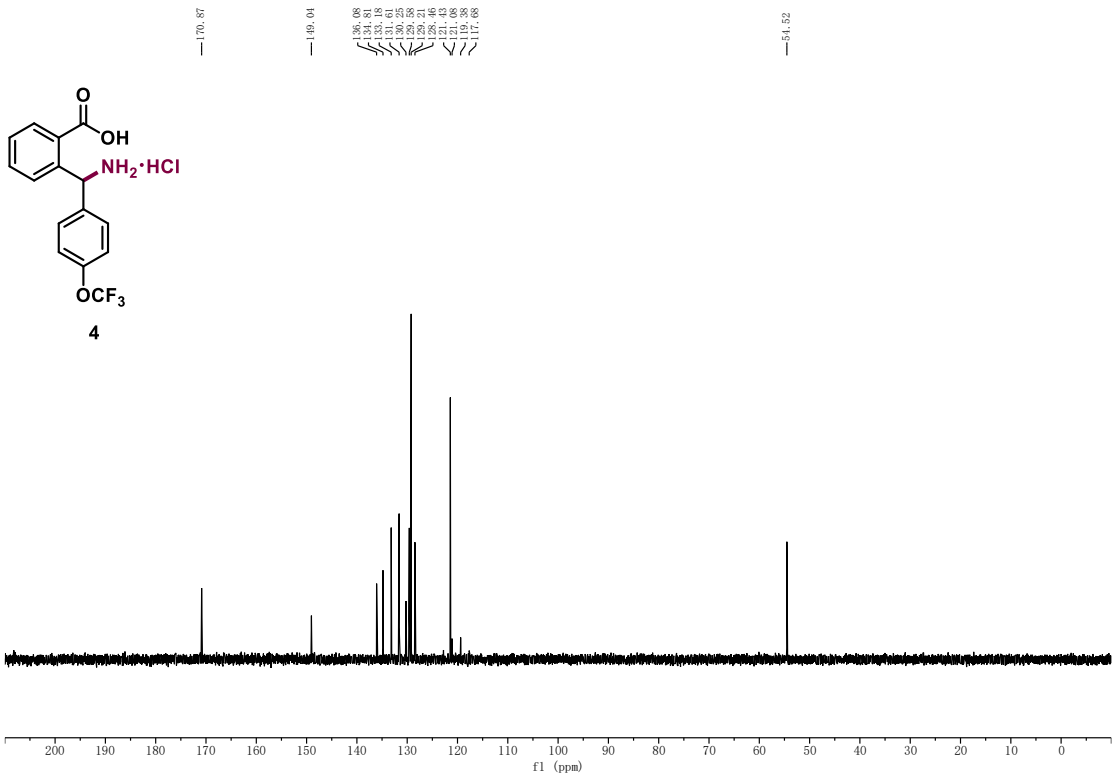

**Supplementary Figure 160** $^{1}$H NMR spectrum of compound **3**



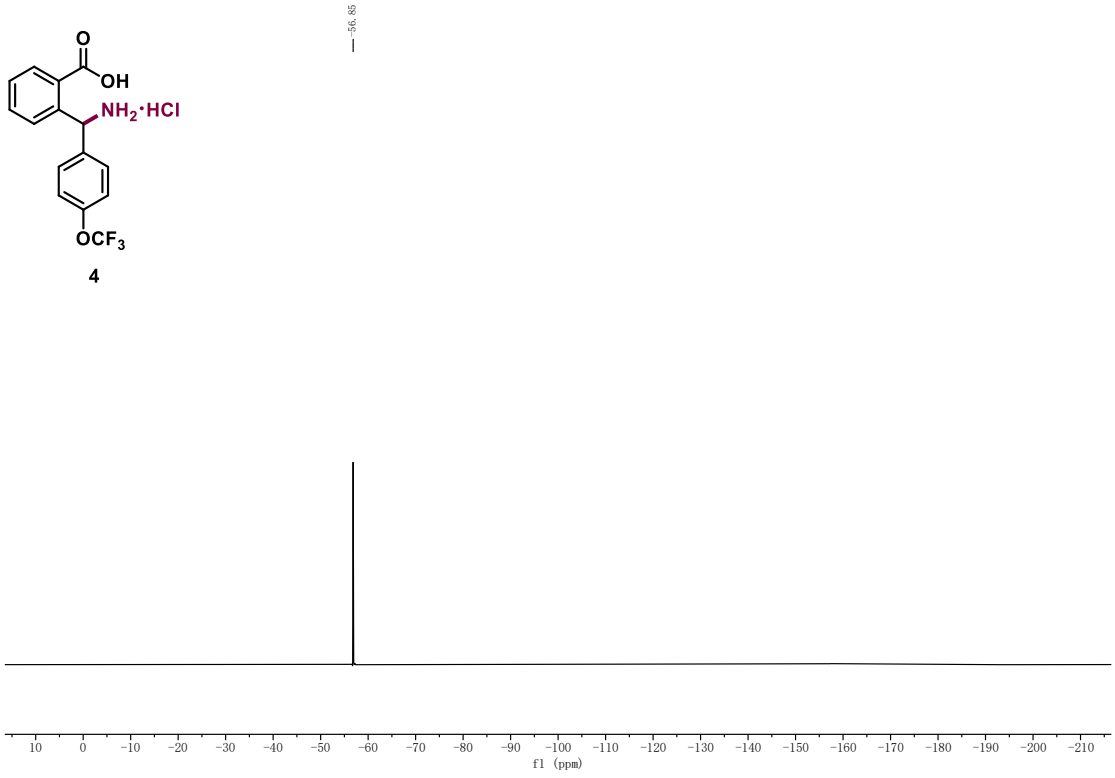

**Supplementary Figure 161** $^{13}$C NMR spectrum of compound **3**



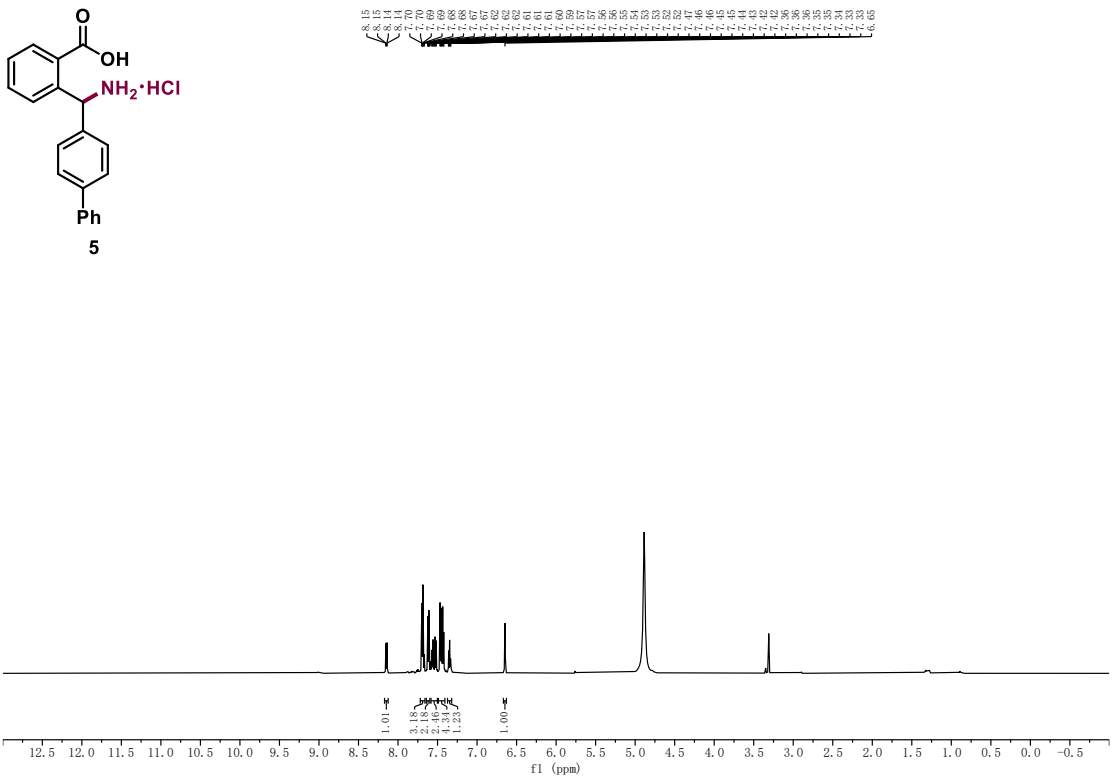

**Supplementary Figure 162** $^{1}$H NMR spectrum of compound **4**



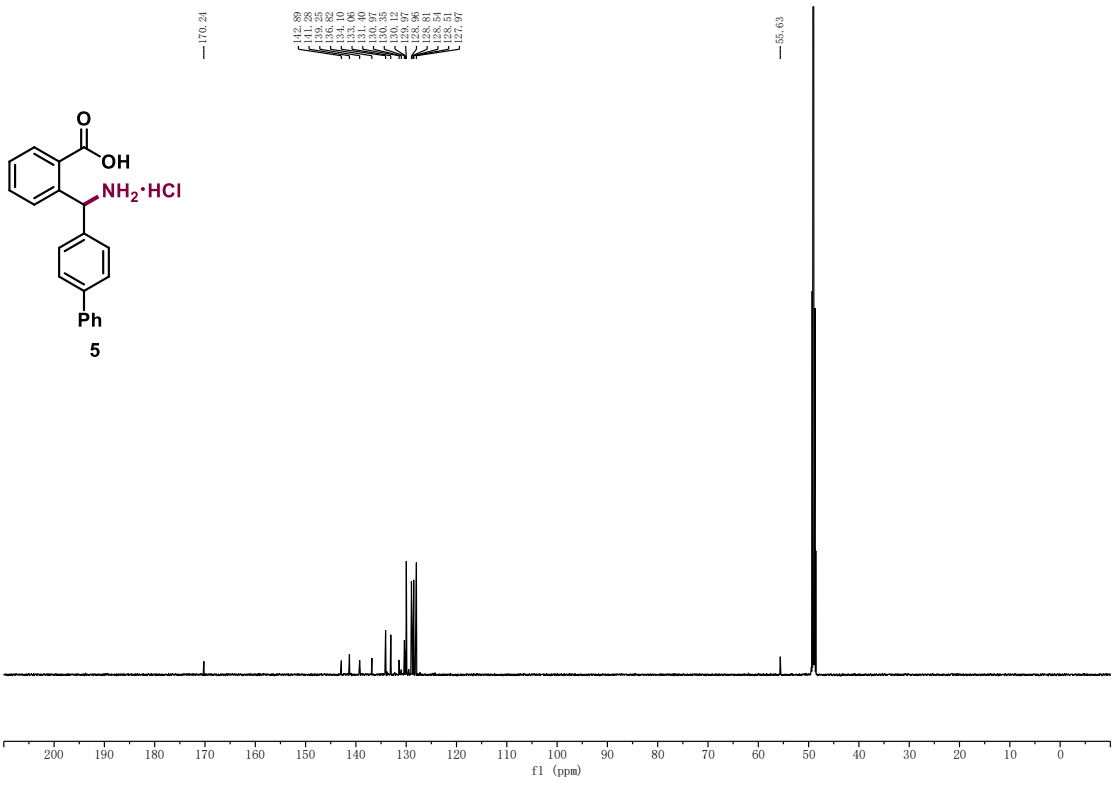

**Supplementary Figure 163** $^{13}$C NMR spectrum of compound **4**



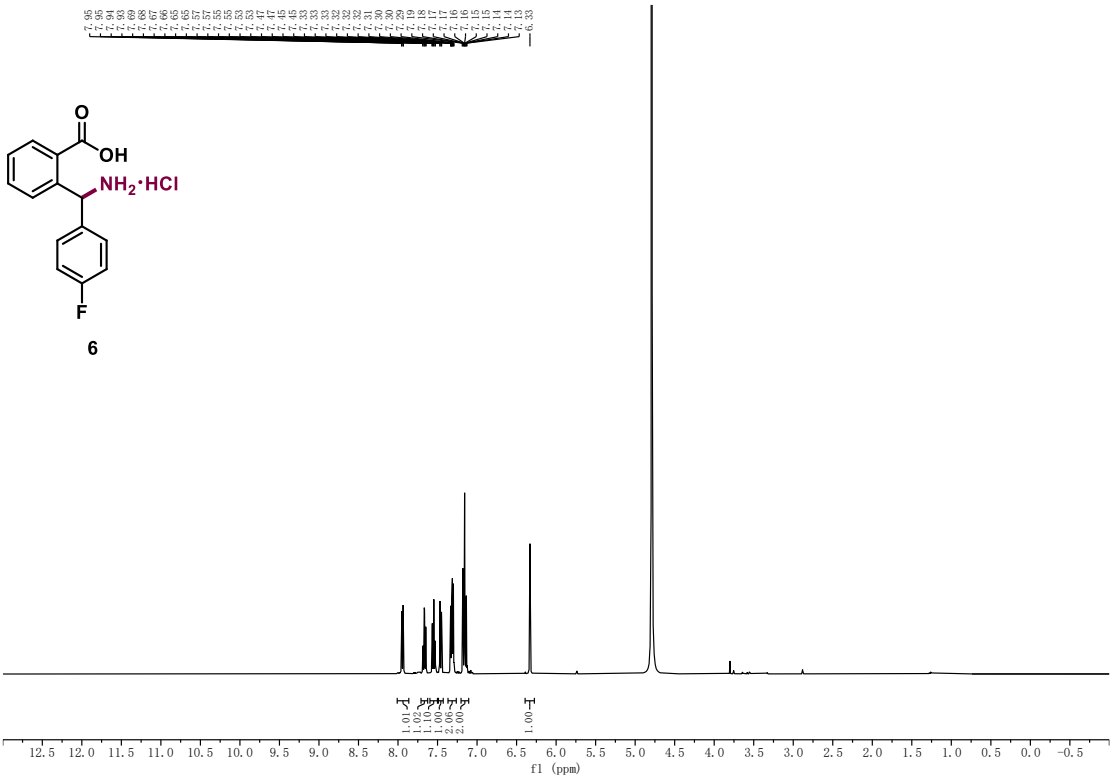

**Supplementary Figure 164** $^{19}$F NMR spectrum of compound **4**



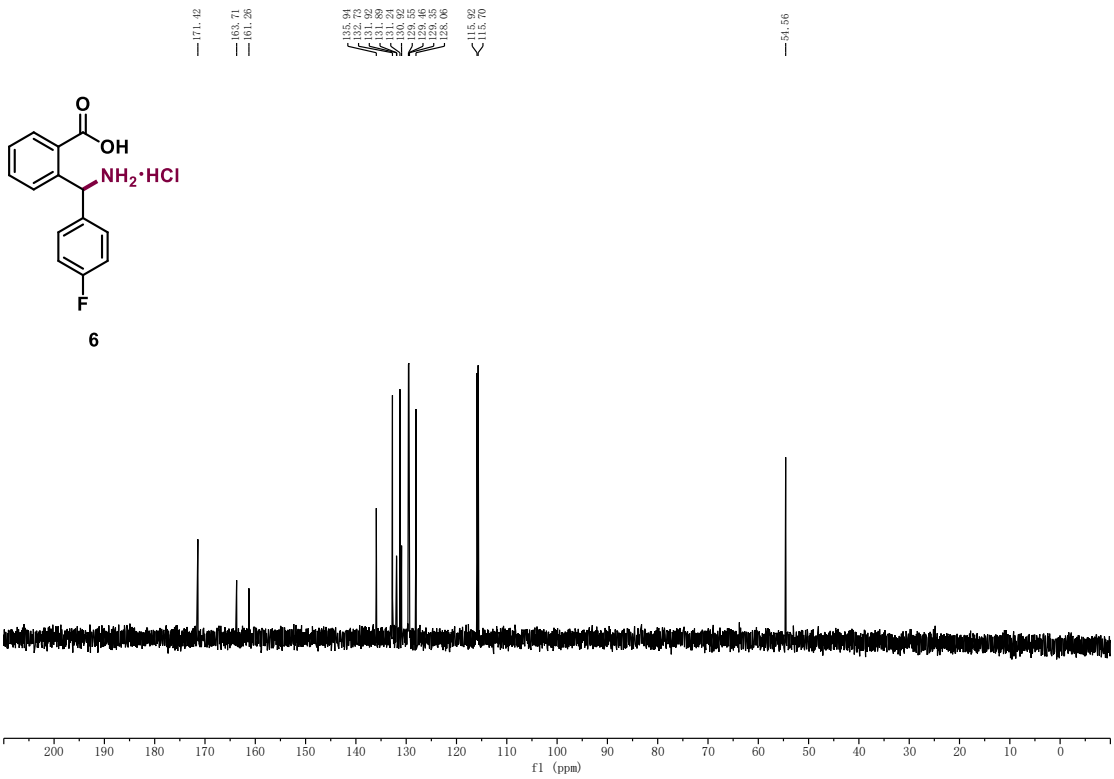

**Supplementary Figure 165** $^{1}$H NMR spectrum of compound **5**



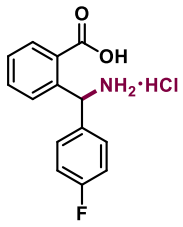

**Supplementary Figure 166** $^{13}$C NMR spectrum of compound **5**




**Supplementary Figure 167** $^{1}$H NMR spectrum of compound **6**



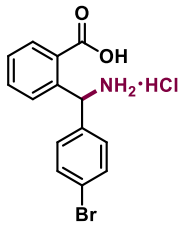

**Supplementary Figure 168** $^{13}$C NMR spectrum of compound **6**




6



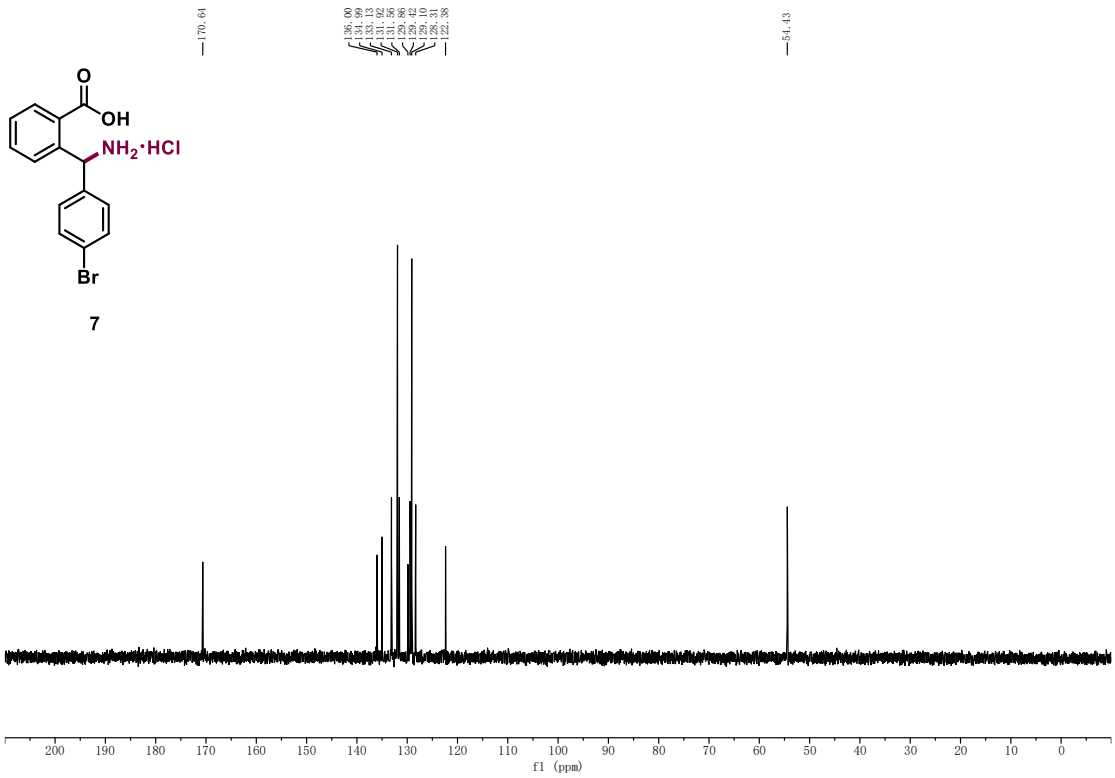

**Supplementary Figure 169** $^{19}$F NMR spectrum of compound **6**



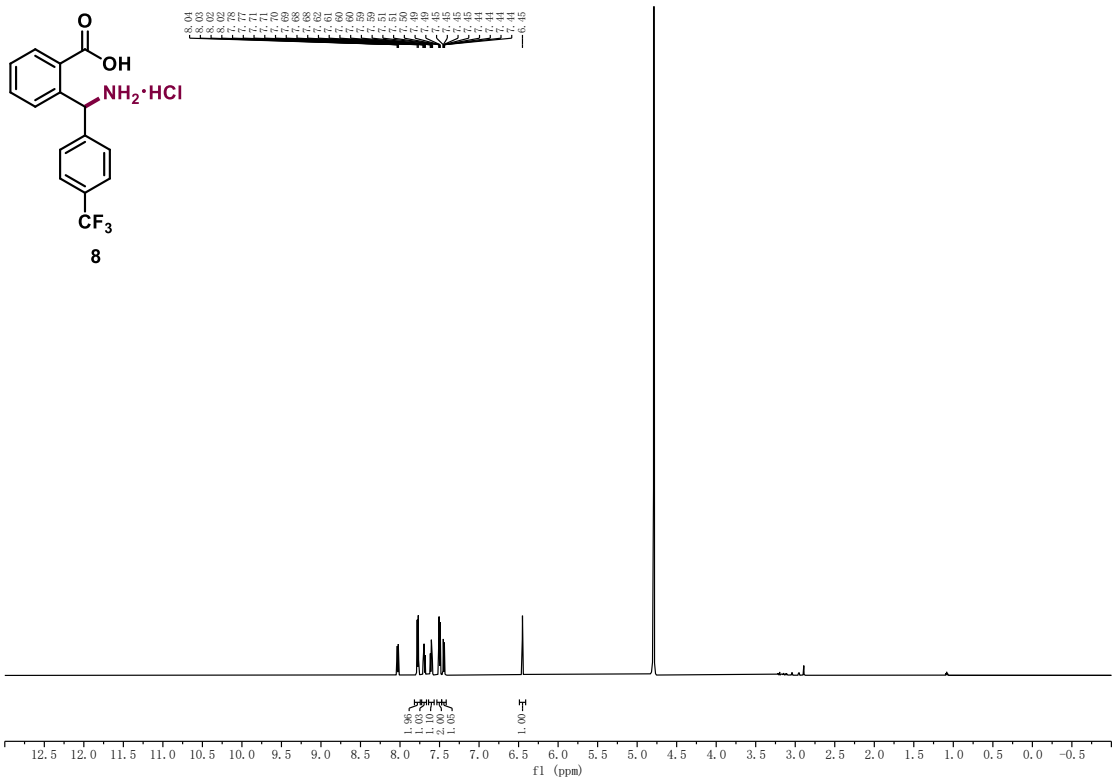

7



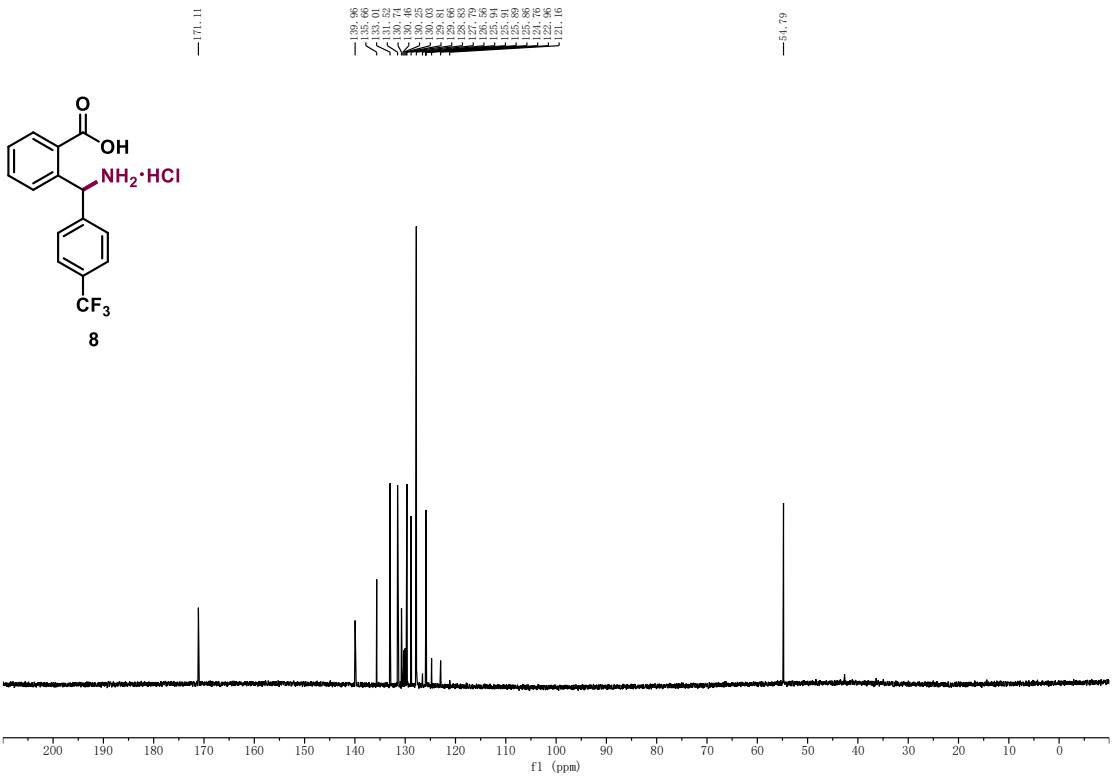

**Supplementary Figure 170** $^{1}$H NMR spectrum of compound **7**



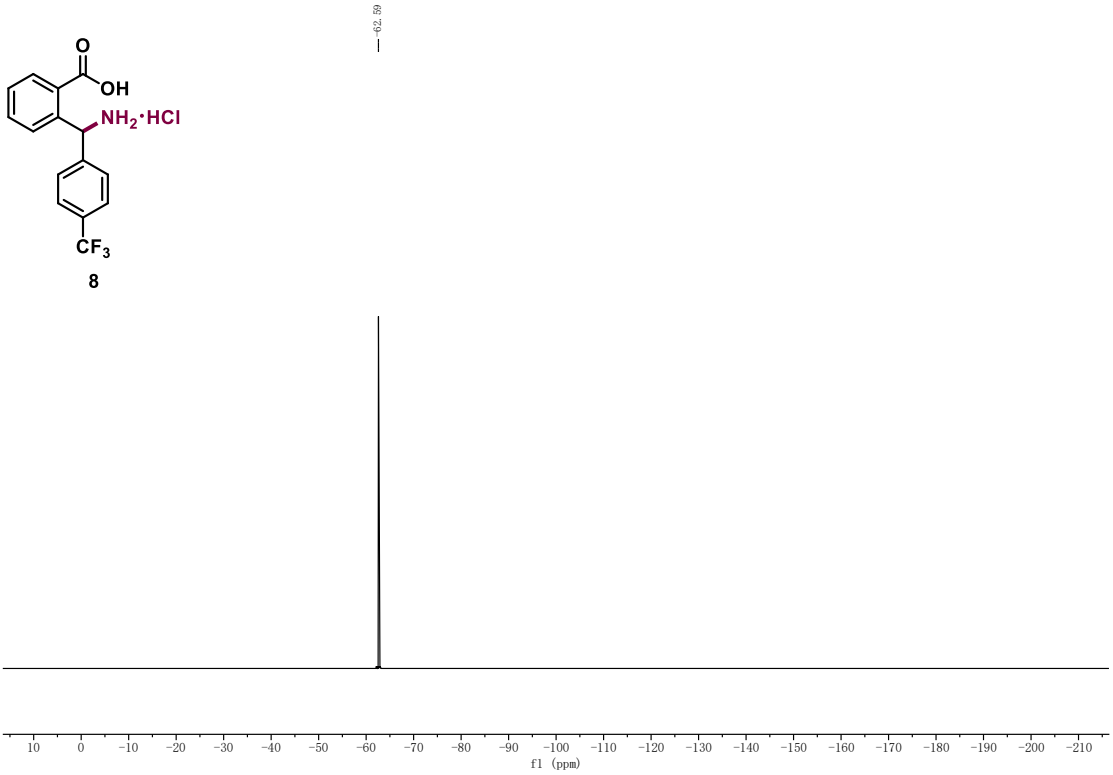

**Supplementary Figure 171** $^{13}$C NMR spectrum of compound **7**



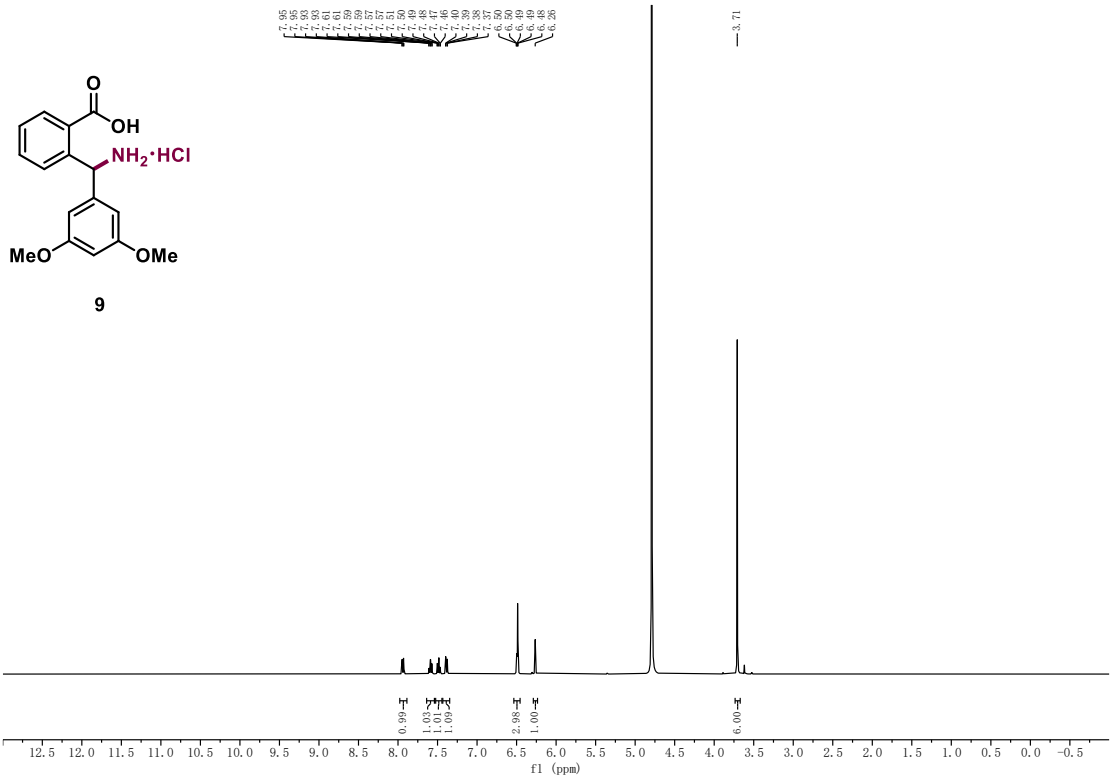

**Supplementary Figure 172** $^{1}$H NMR spectrum of compound **8**



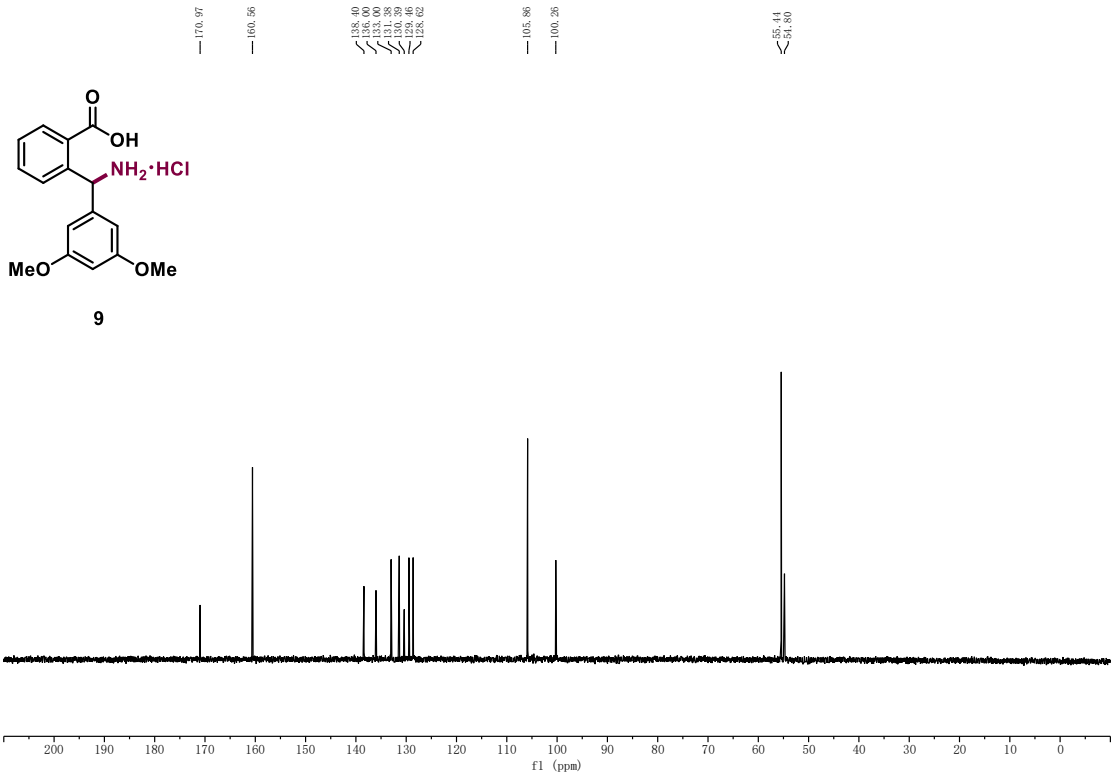

**Supplementary Figure 173** $^{13}$C NMR spectrum of compound **8**



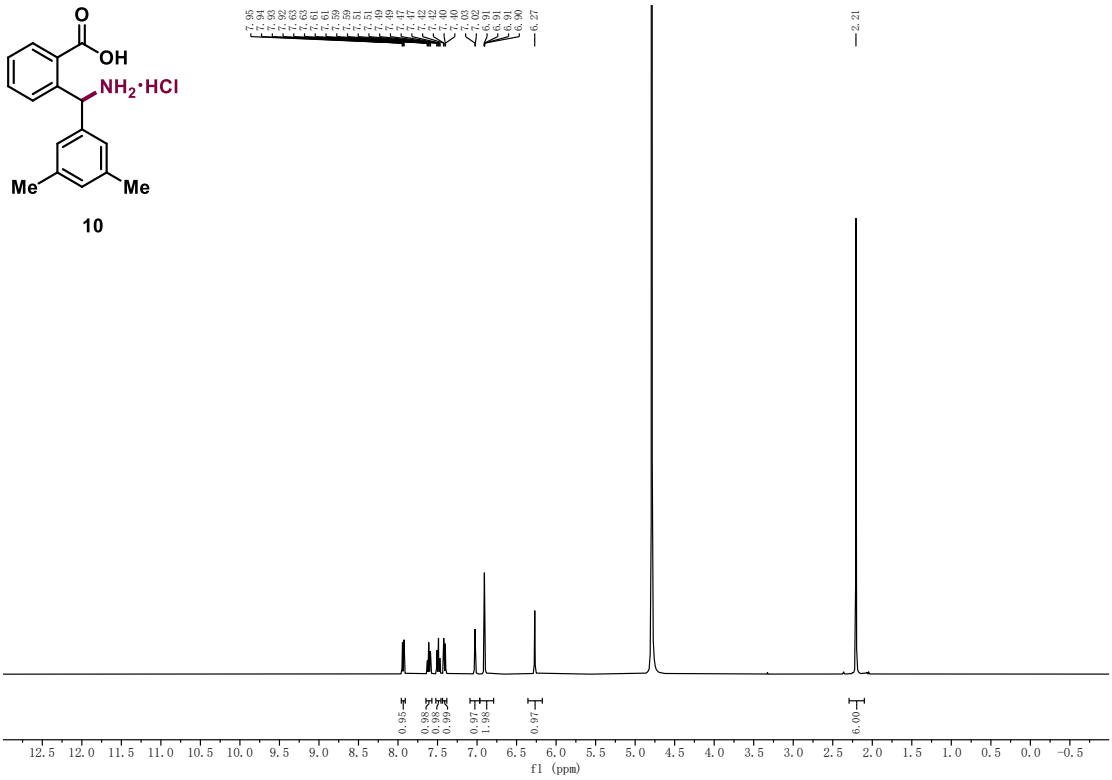

**Supplementary Figure 174** $^{19}$F NMR spectrum of compound **8**



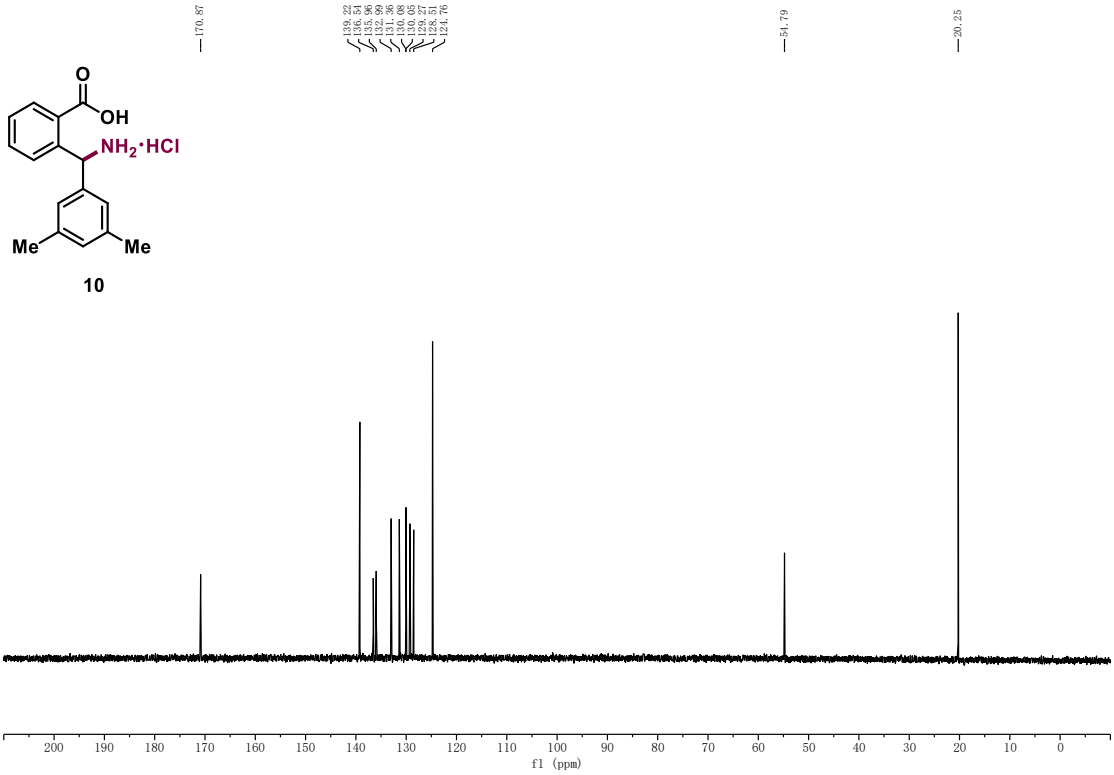

**Supplementary Figure 175** $^{1}$H NMR spectrum of compound **9**



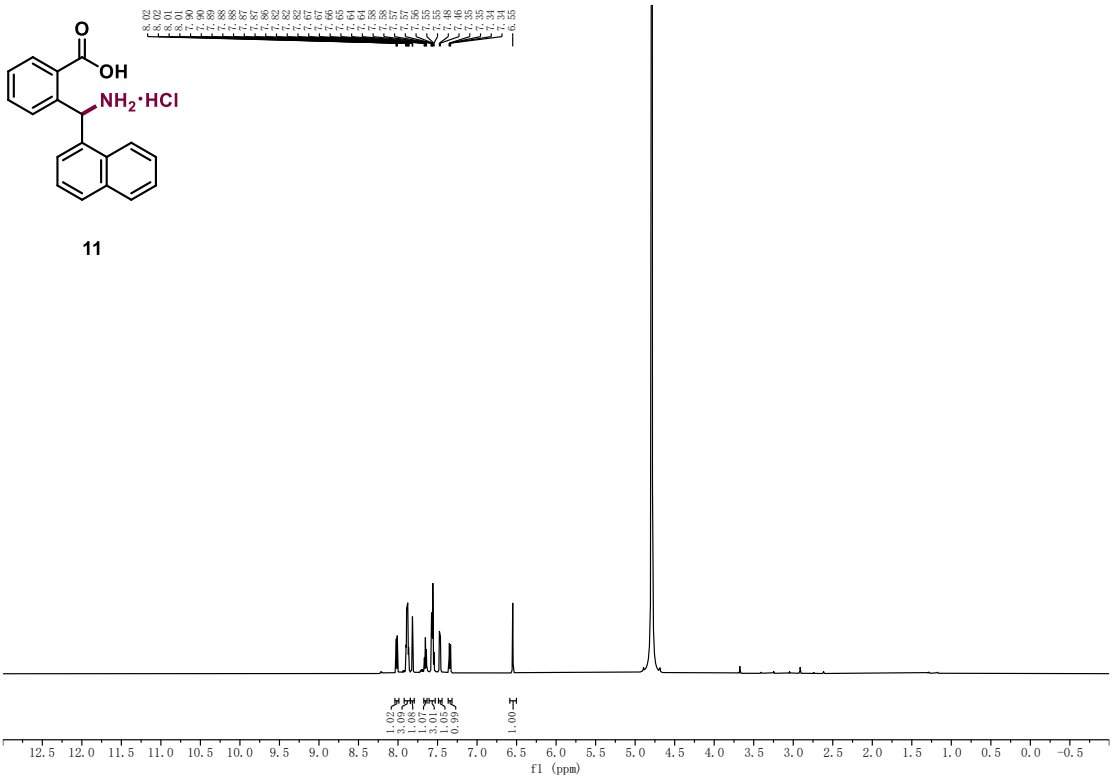

**Supplementary Figure 176** $^{13}$C NMR spectrum of compound **9**



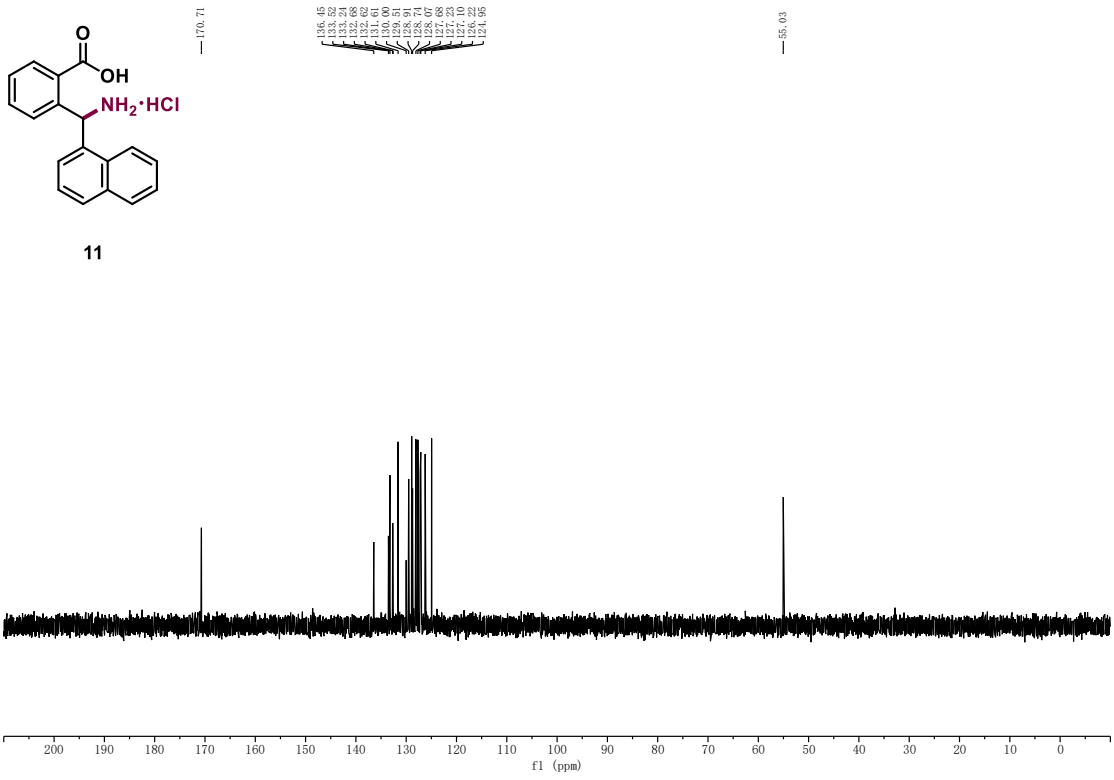

**Supplementary Figure 177** $^{1}$H NMR spectrum of compound **10**



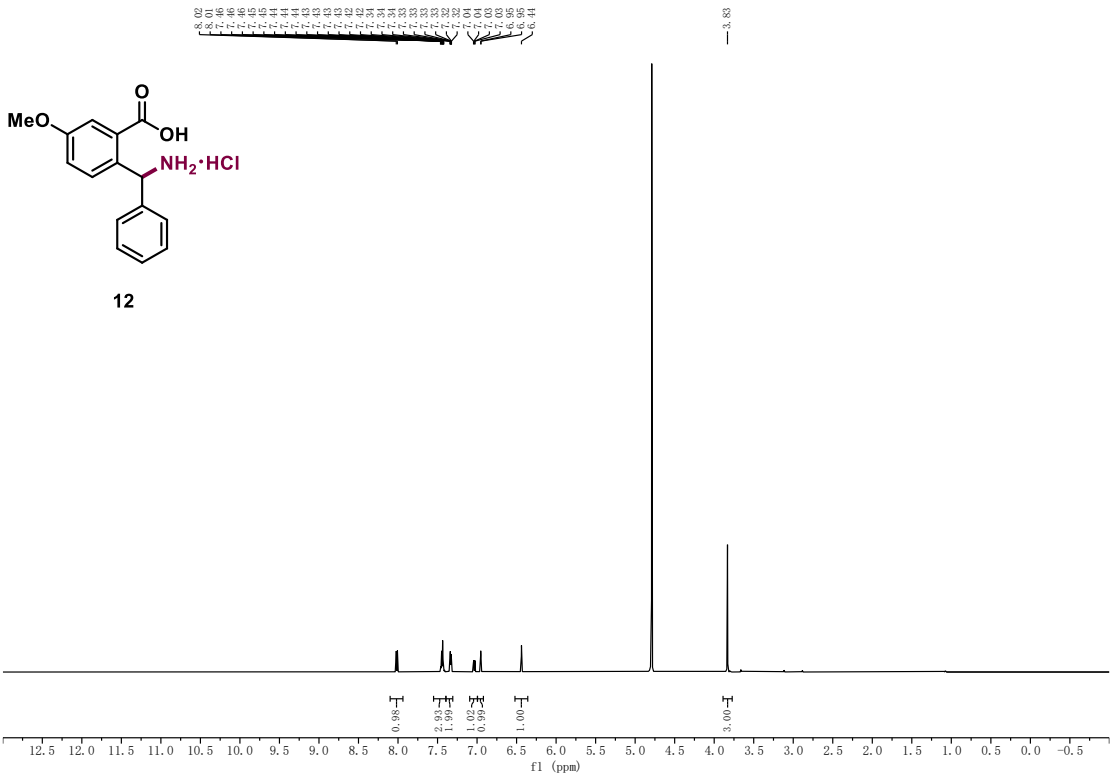

**Supplementary Figure 178** $^{13}$C NMR spectrum of compound **10**



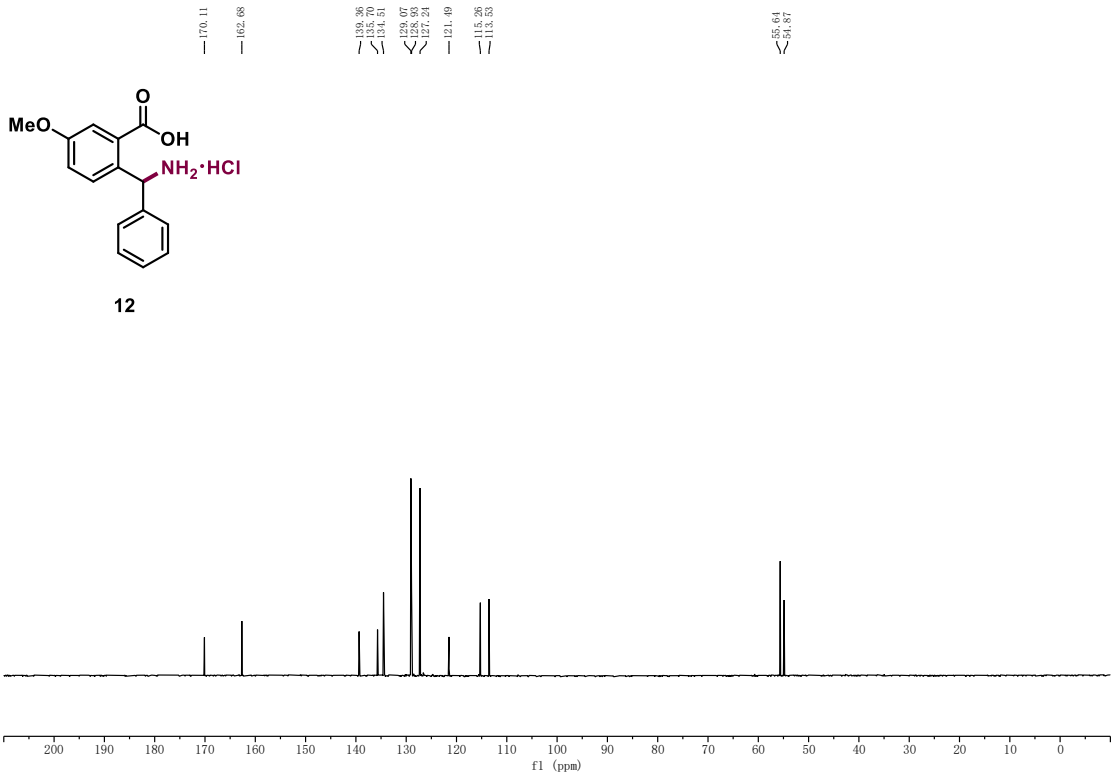

**Supplementary Figure 179** $^{1}$H NMR spectrum of compound **11**



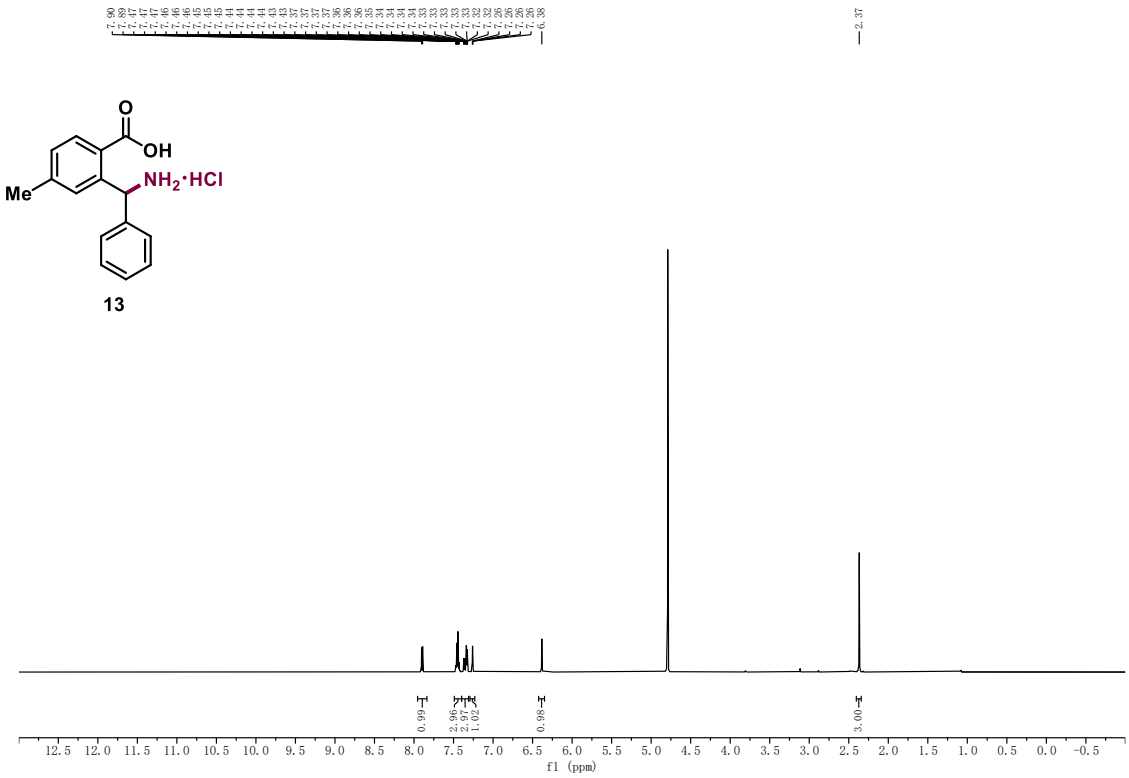

**Supplementary Figure 180** $^{13}$C NMR spectrum of compound **11**



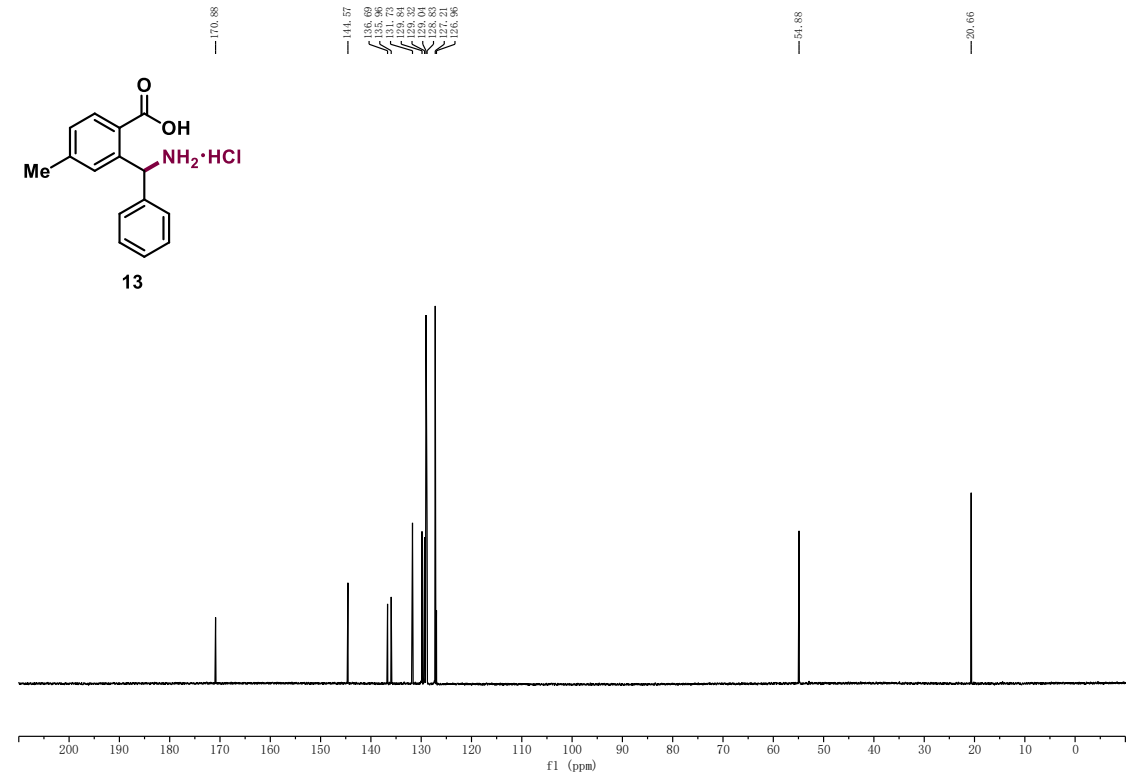

**Supplementary Figure 181** $^{1}$H NMR spectrum of compound **12**



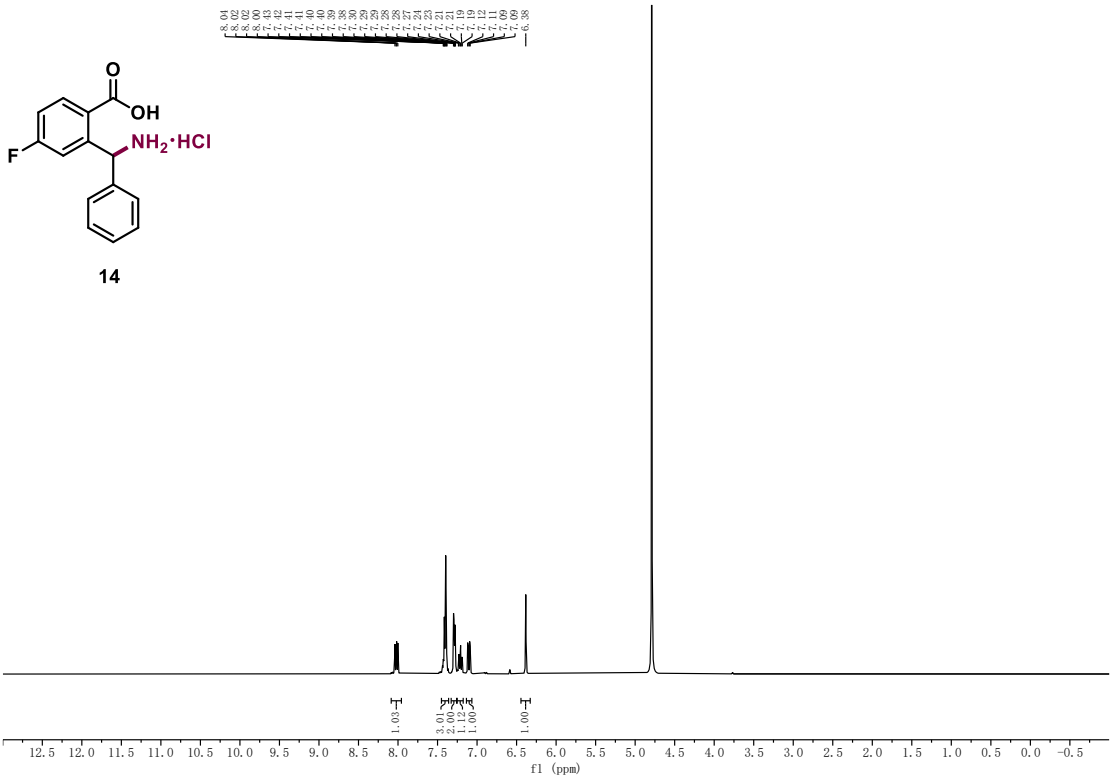

**Supplementary Figure 182** $^{13}$C NMR spectrum of compound **12**



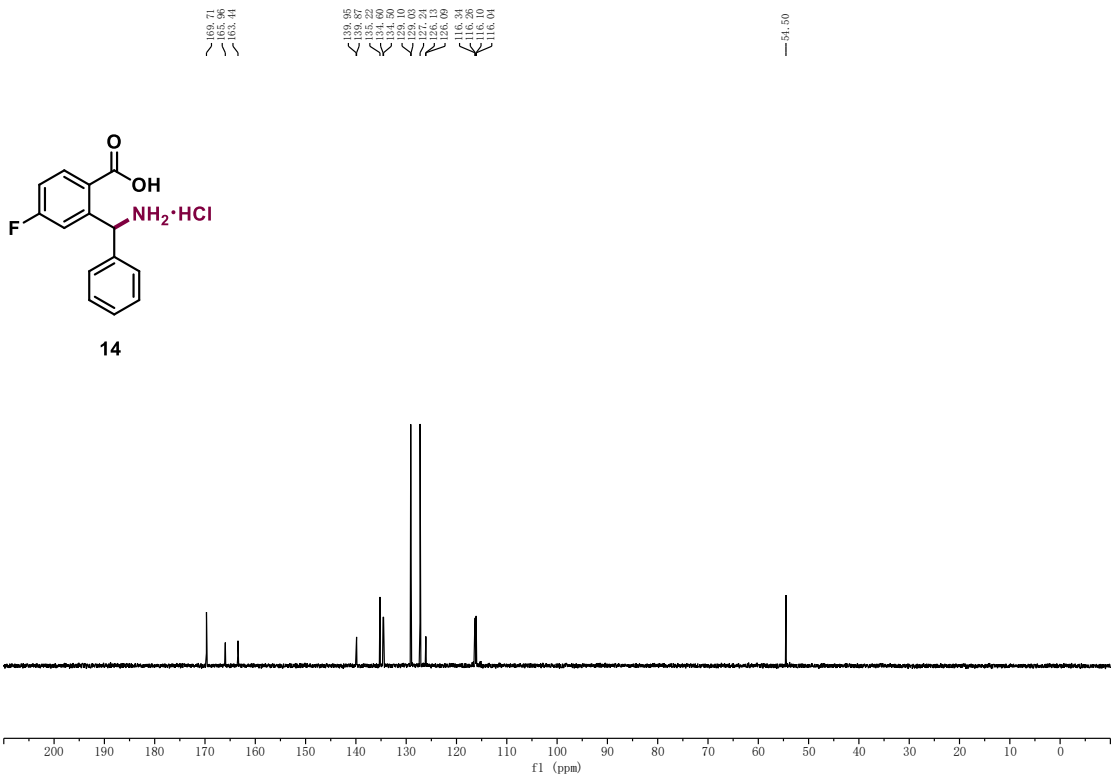

**Supplementary Figure 183** $^{1}$H NMR spectrum of compound **13**



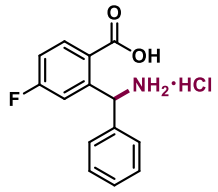

**Supplementary Figure 184** $^{13}$C NMR spectrum of compound **13**




**Supplementary Figure 185** $^{1}$H NMR spectrum of compound **14**



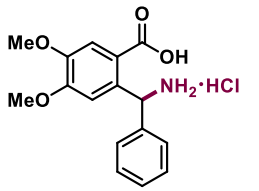

**Supplementary Figure 186** $^{13}$C NMR spectrum of compound **14**




14



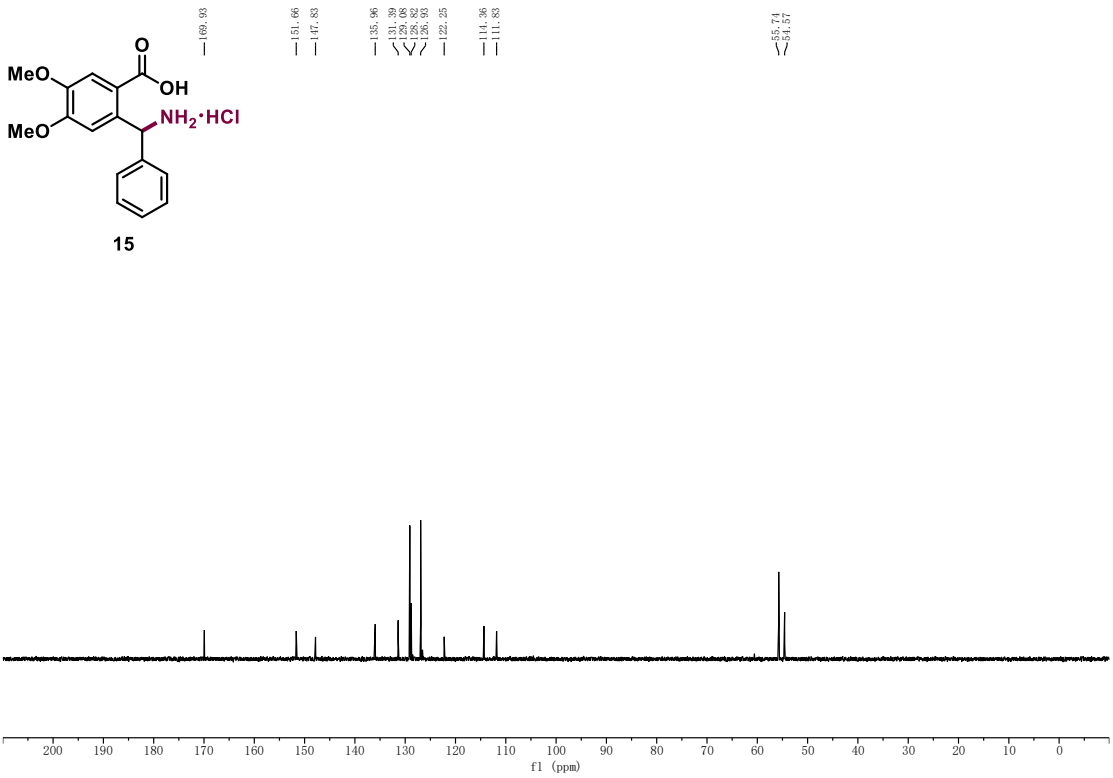

**Supplementary Figure 187** $^{19}$F NMR spectrum of compound **14**



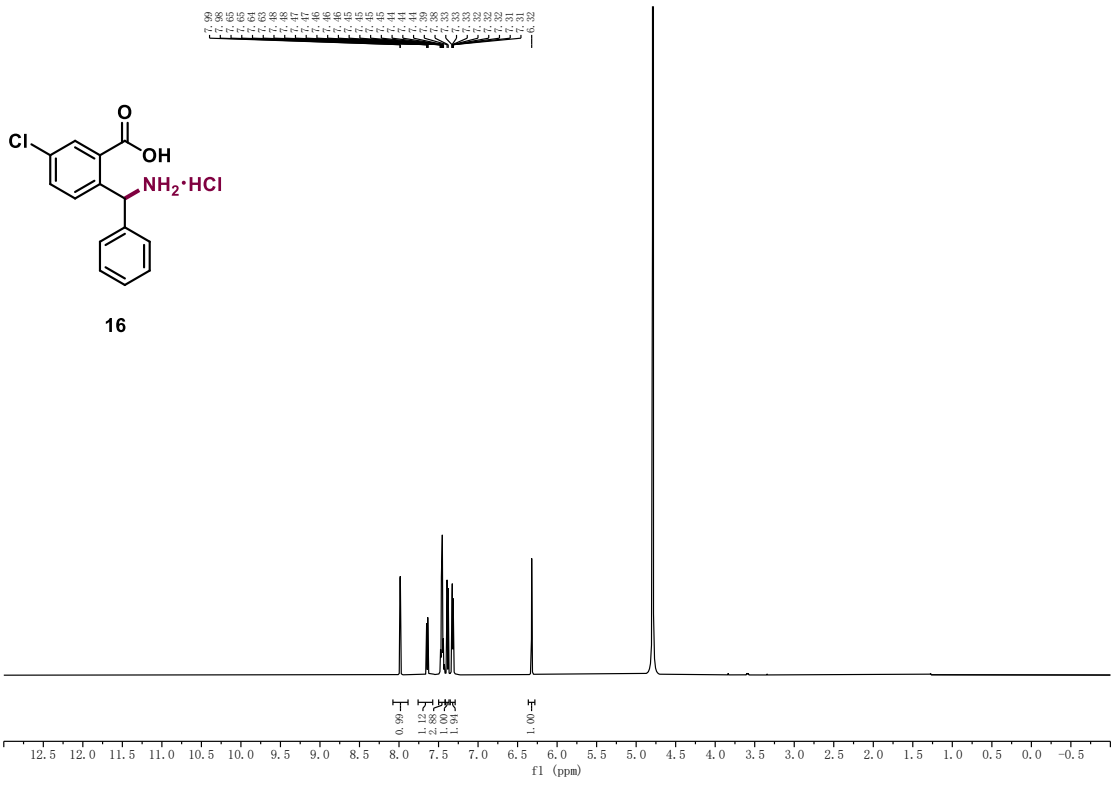

15



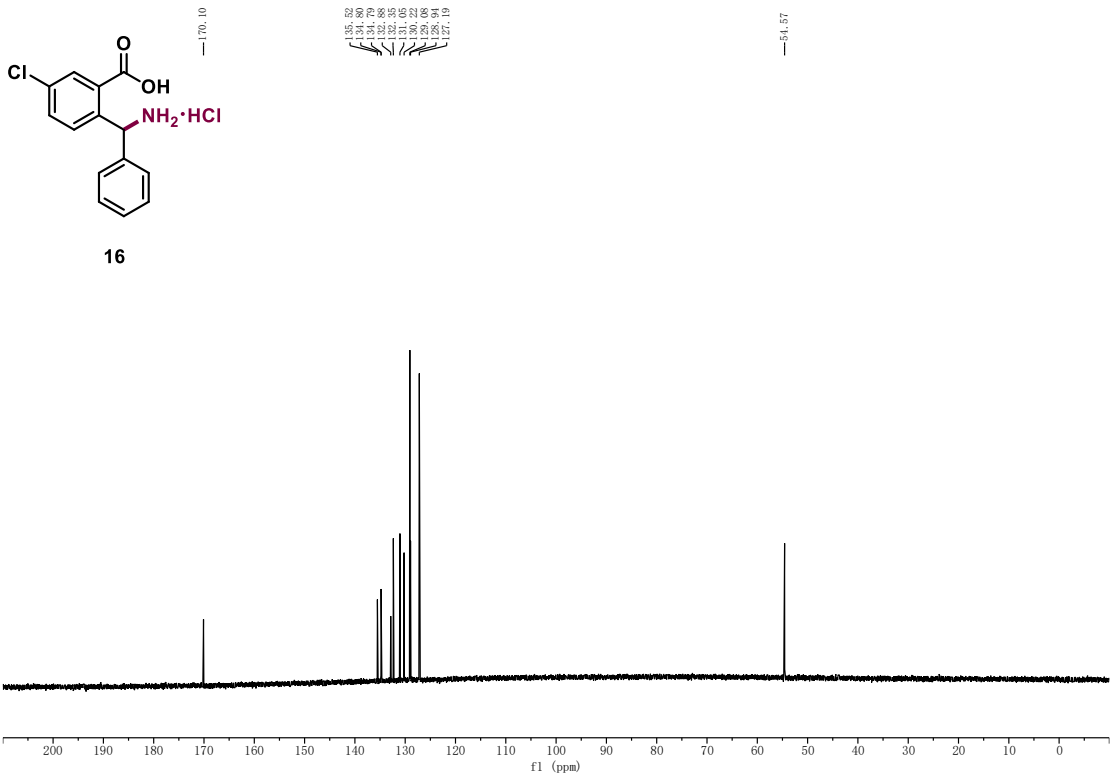

**Supplementary Figure 188** $^{1}$H NMR spectrum of compound **15**



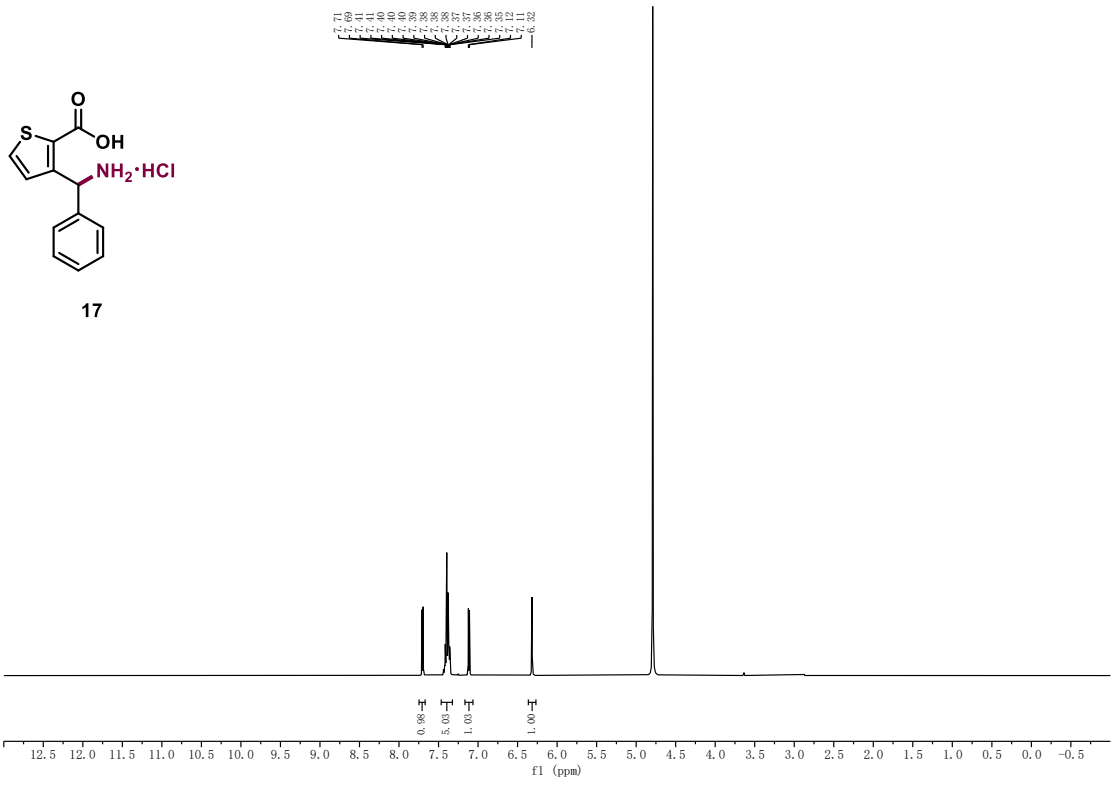

**Supplementary Figure 189** $^{13}$C NMR spectrum of compound **15**



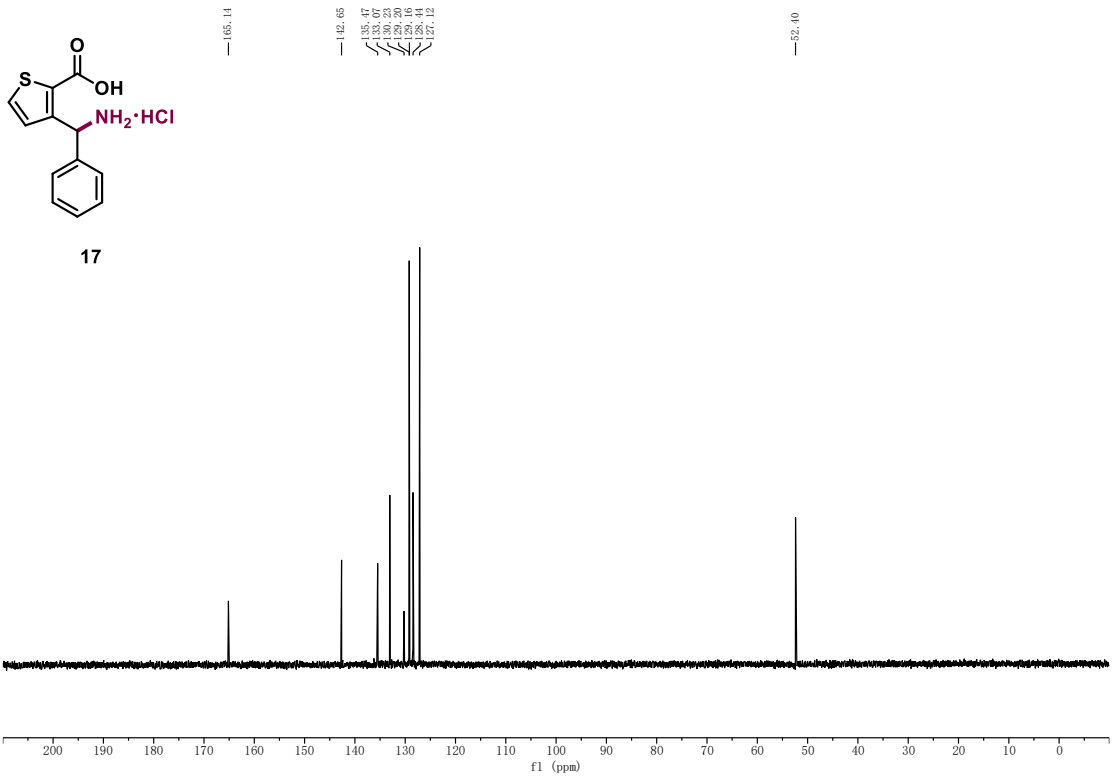

**Supplementary Figure 190** $^{1}$H NMR spectrum of compound **16**



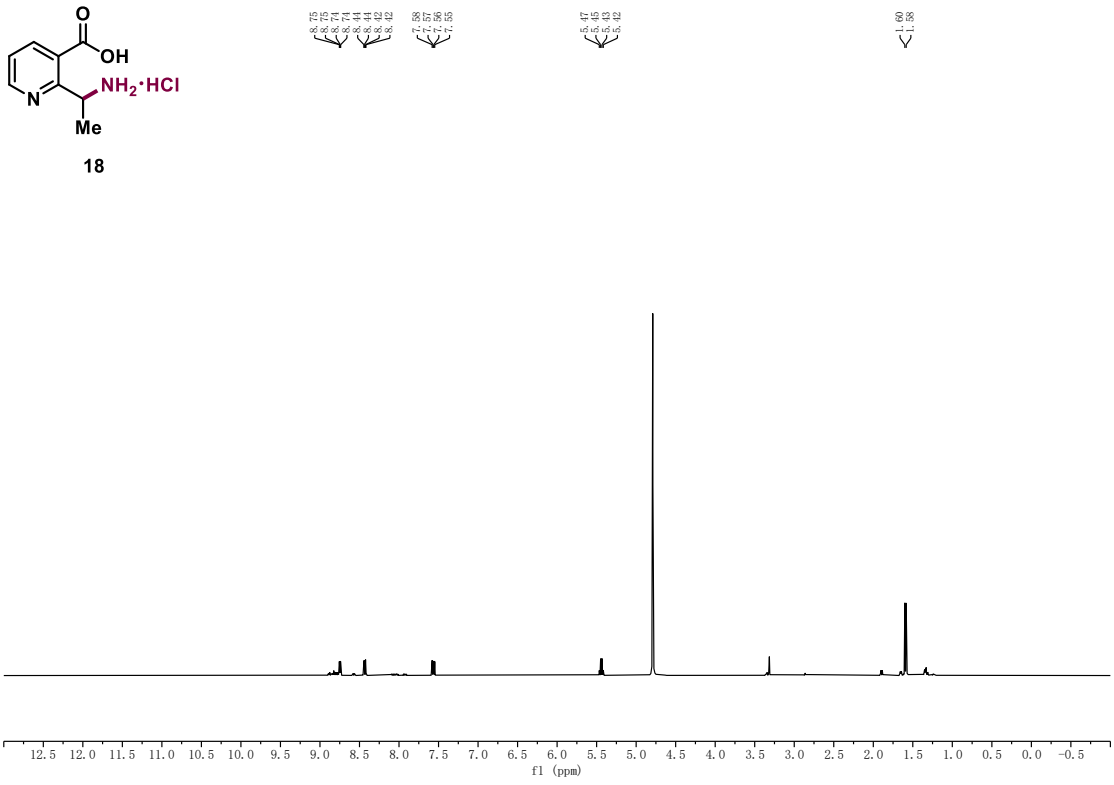

**Supplementary Figure 191** $^{13}$C NMR spectrum of compound **16**



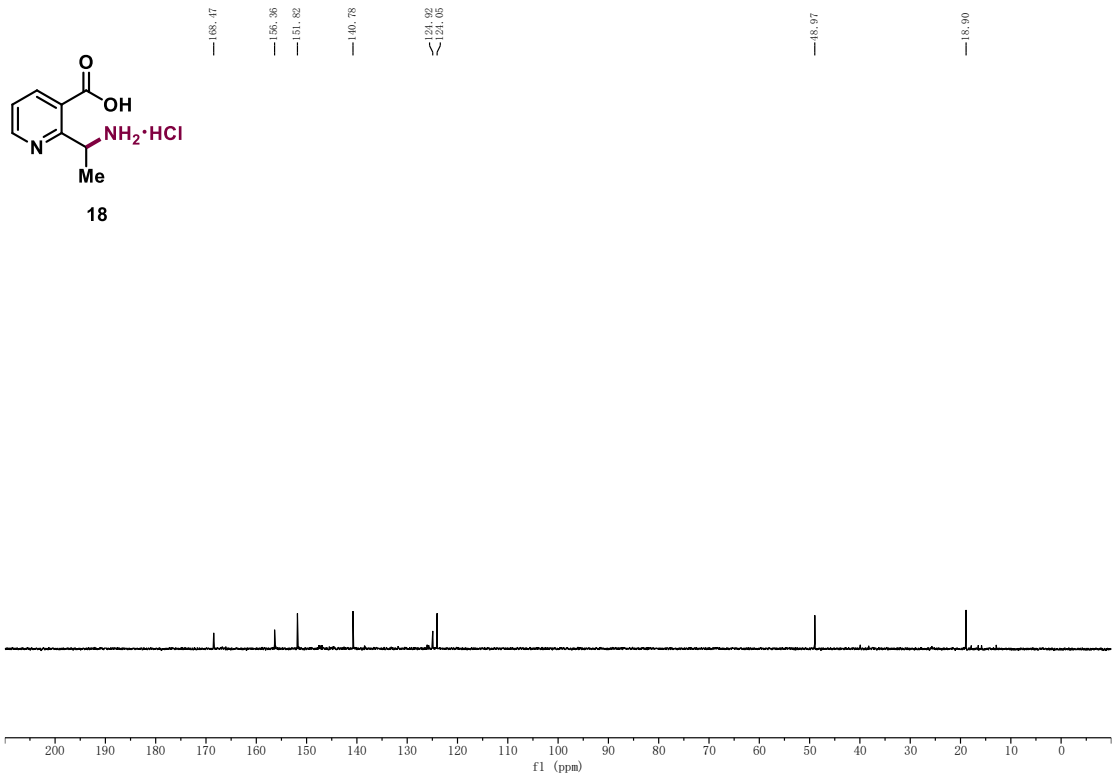

**Supplementary Figure 192** $^{1}$H NMR spectrum of compound **17**



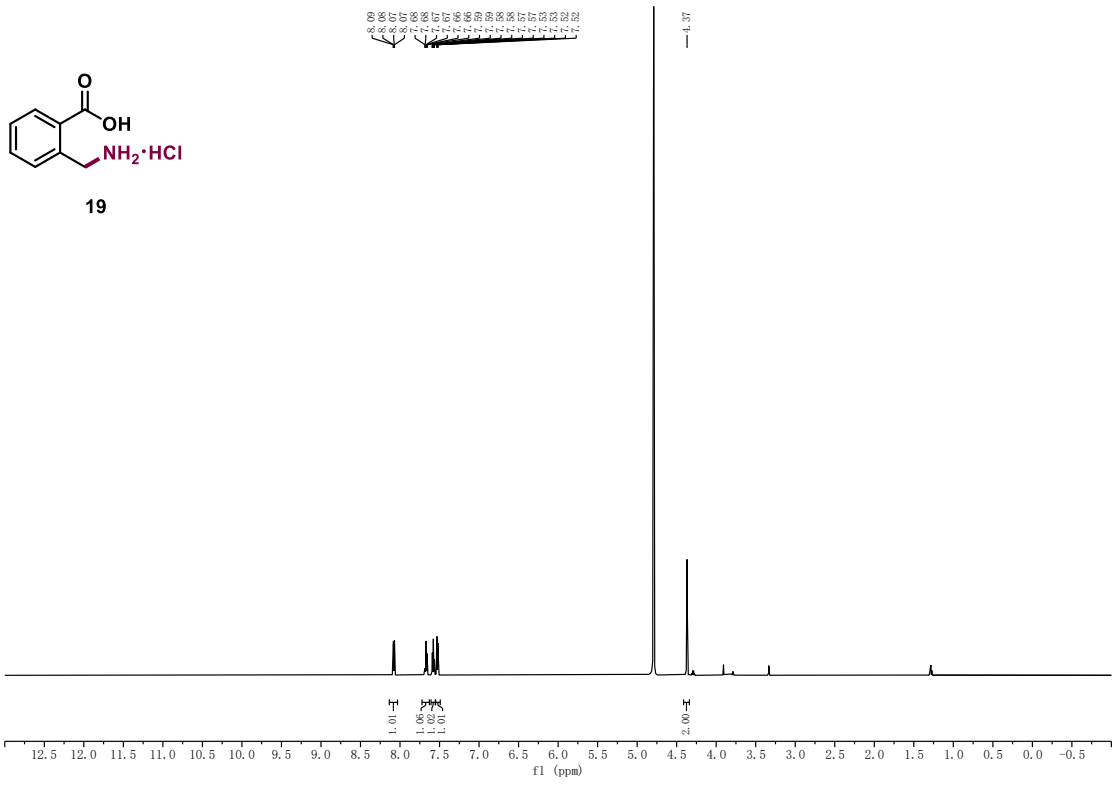

**Supplementary Figure 193** $^{13}$C NMR spectrum of compound **17**



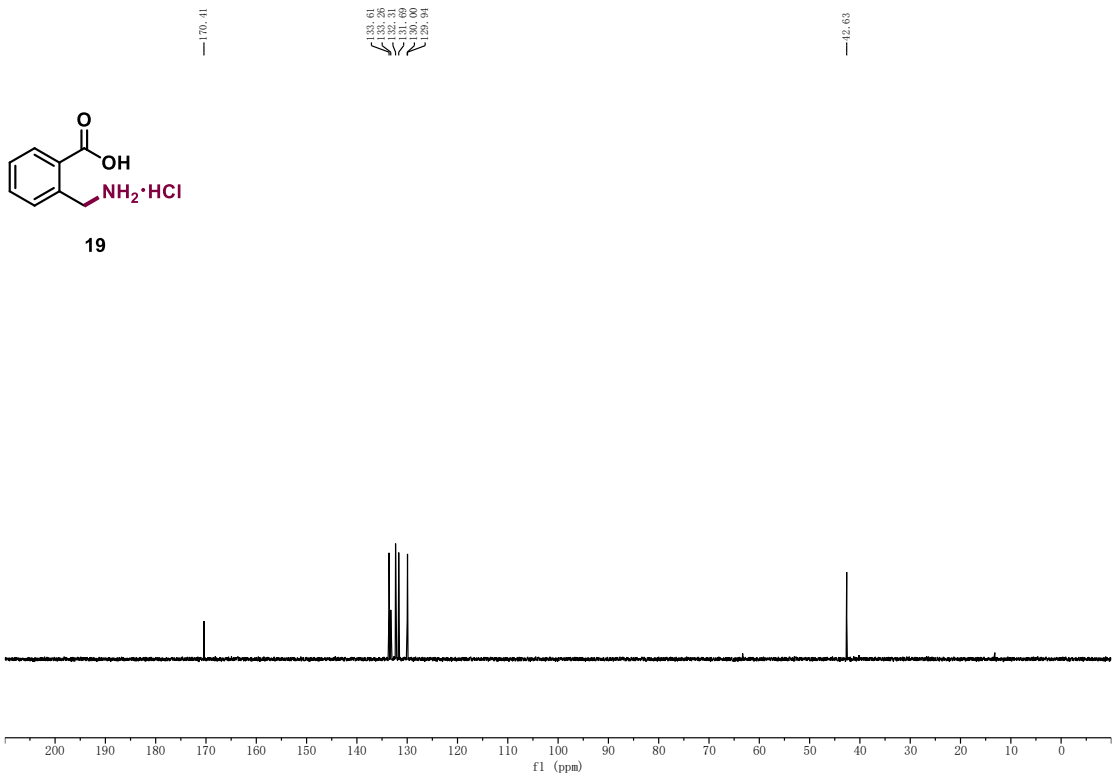

**Supplementary Figure 194** $^{1}$H NMR spectrum of compound **18**



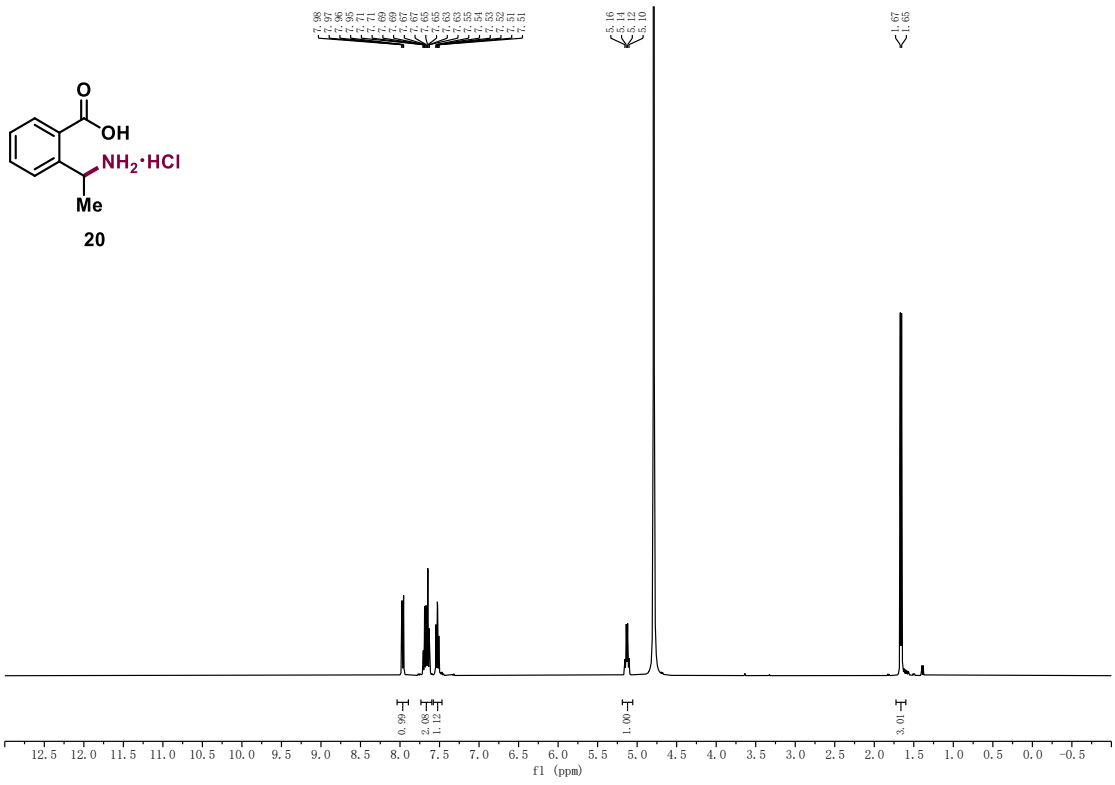

**Supplementary Figure 195** $^{13}$C NMR spectrum of compound **18**



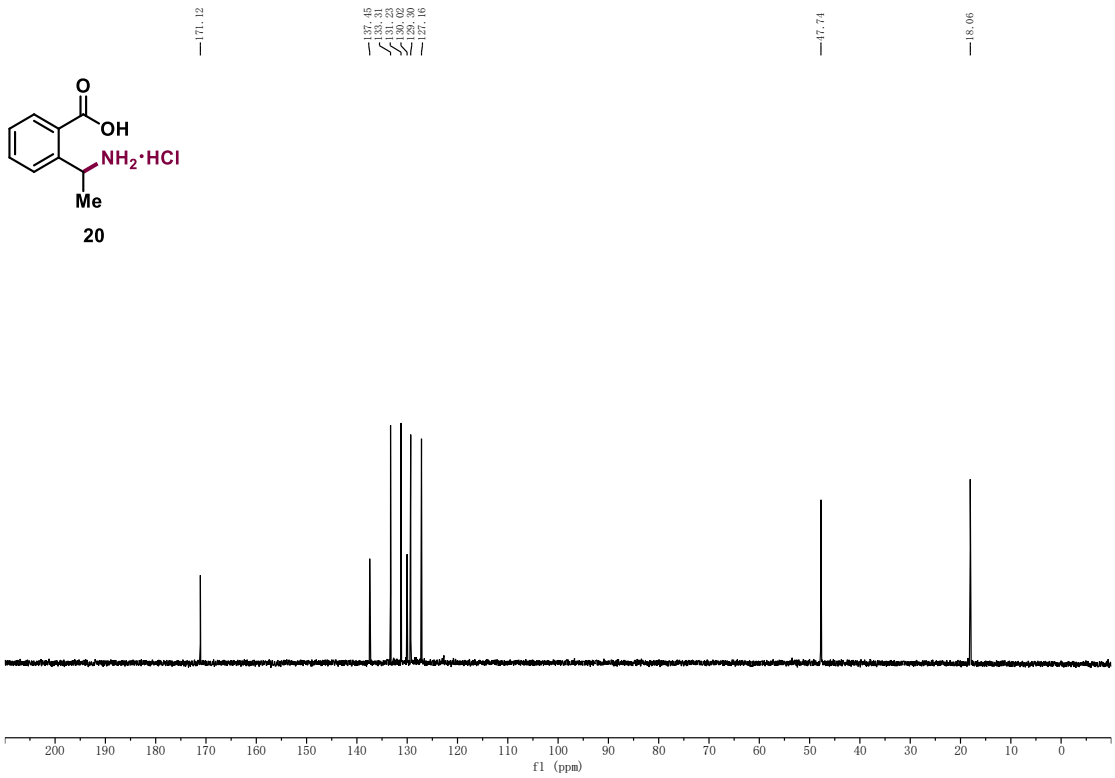

**Supplementary Figure 196** $^{1}$H NMR spectrum of compound **19**



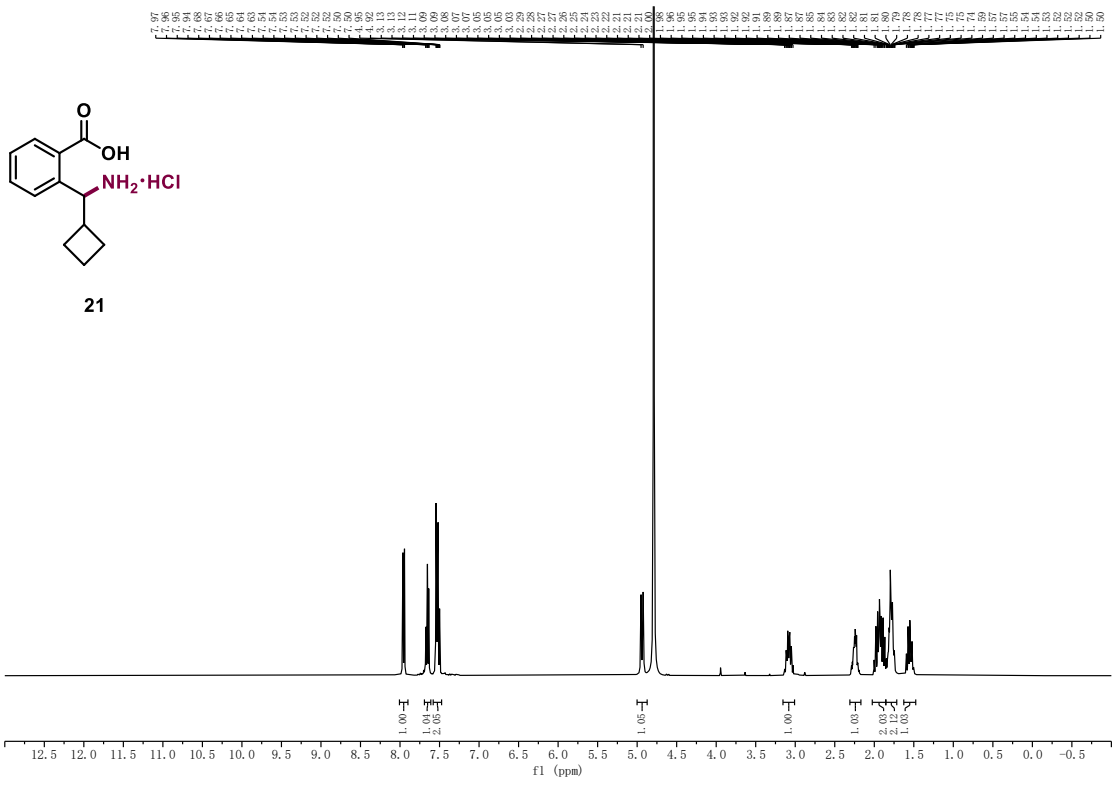

**Supplementary Figure 197** $^{13}$C NMR spectrum of compound **19**



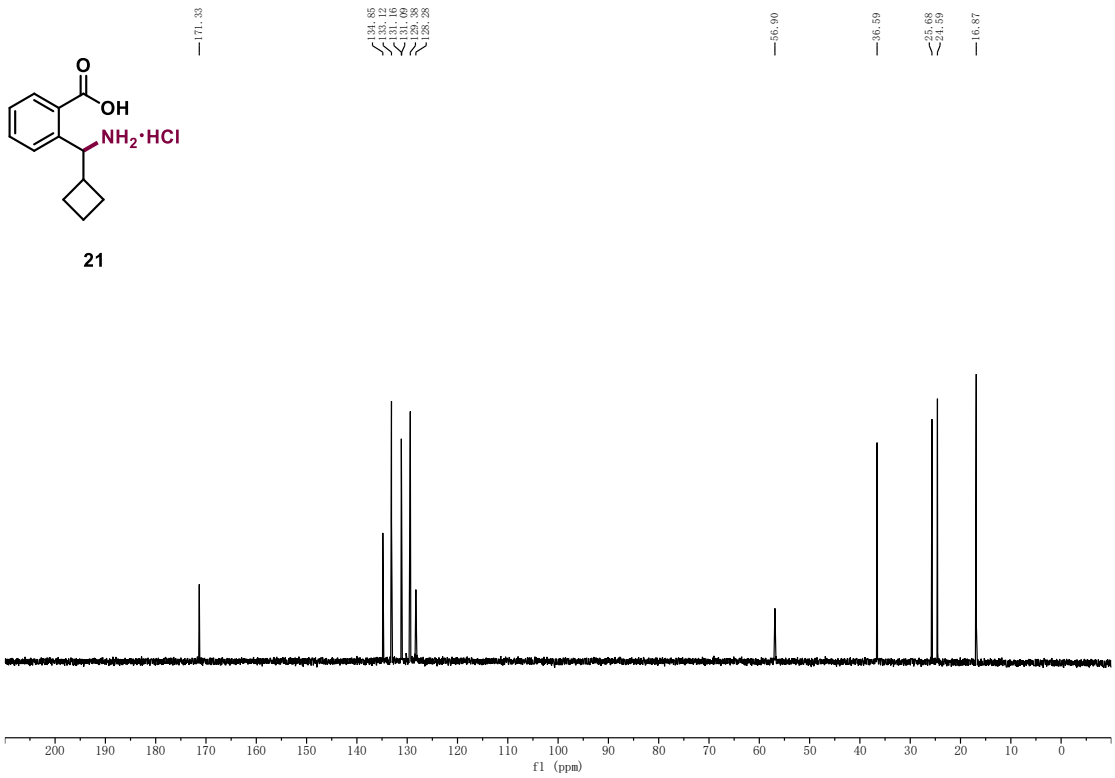

**Supplementary Figure 198** $^{1}$H NMR spectrum of compound **20**



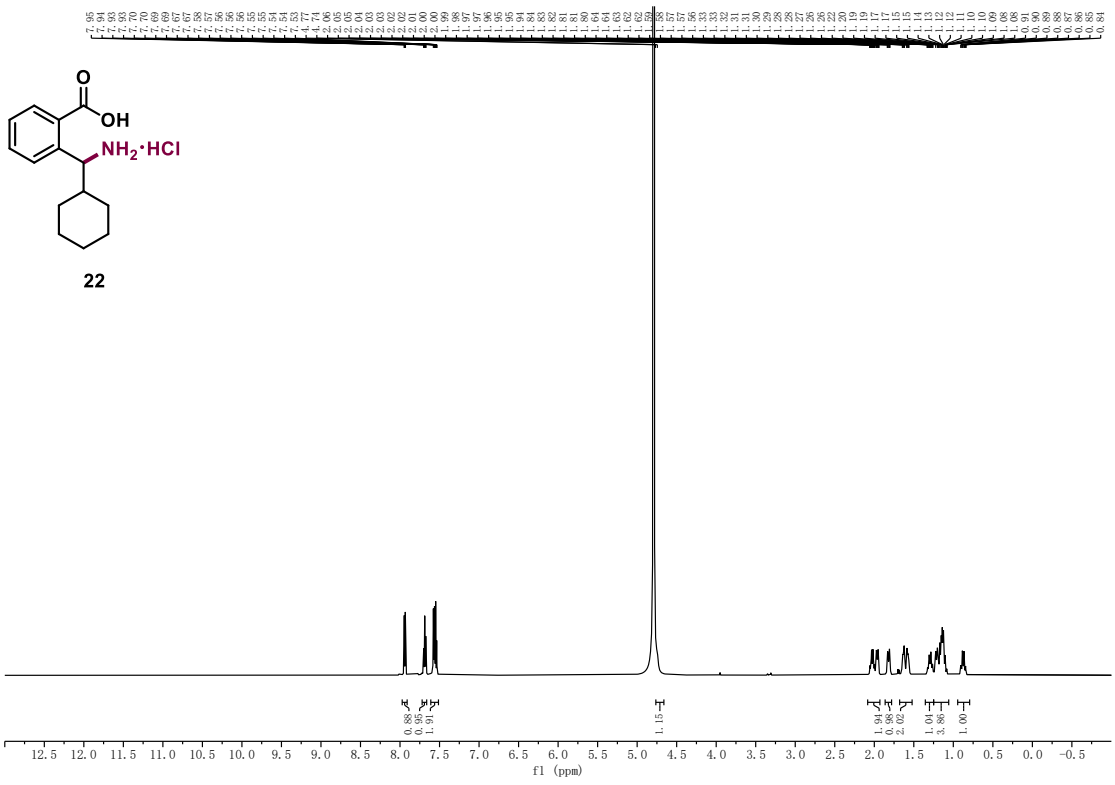

**Supplementary Figure 199** $^{13}$C NMR spectrum of compound **20**



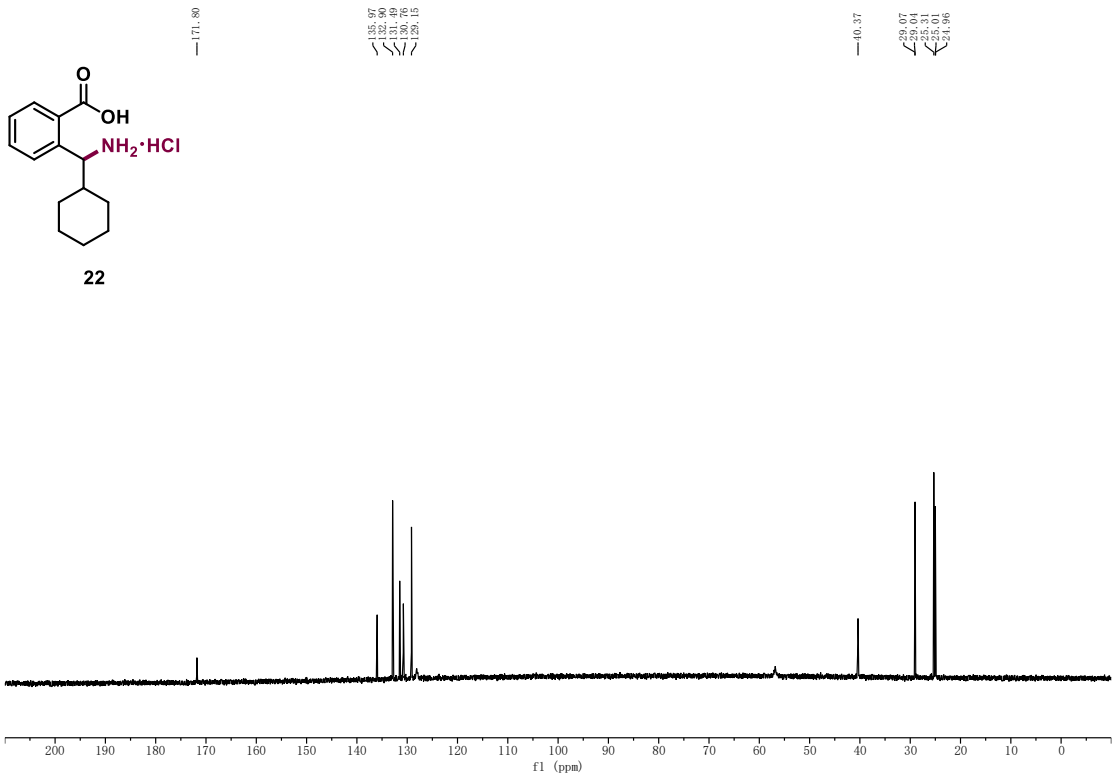

**Supplementary Figure 200** $^{1}$H NMR spectrum of compound **21**



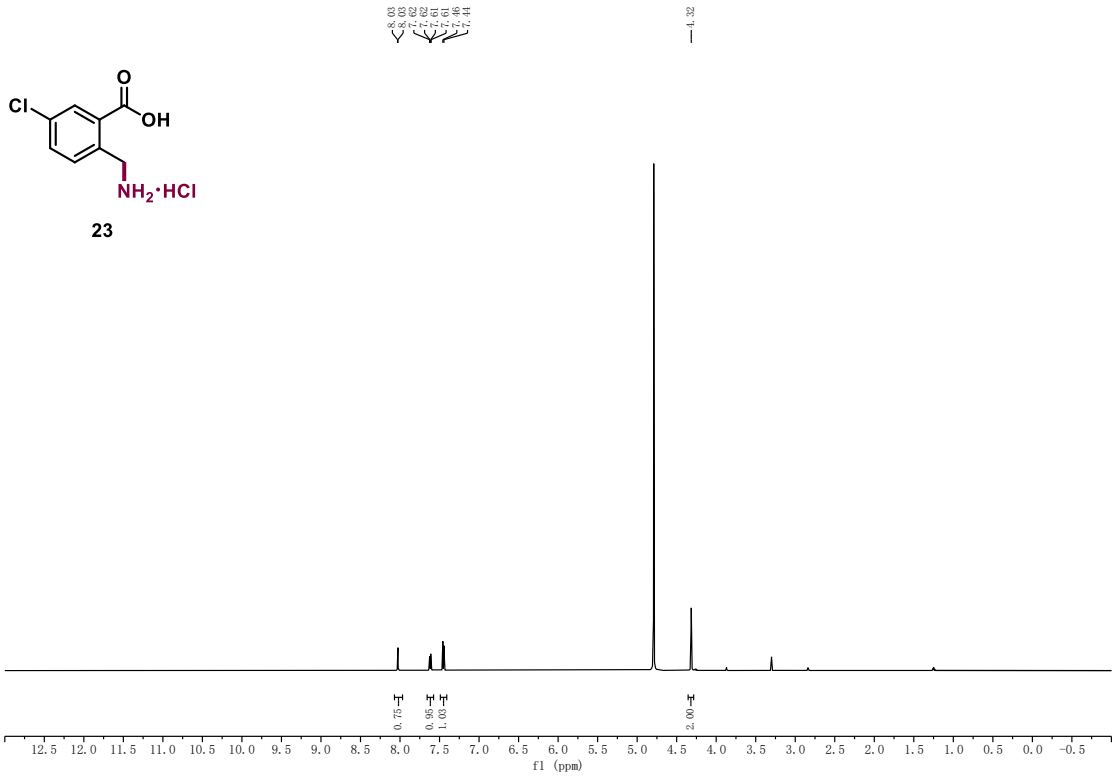

**Supplementary Figure 201** $^{13}$C NMR spectrum of compound **21**



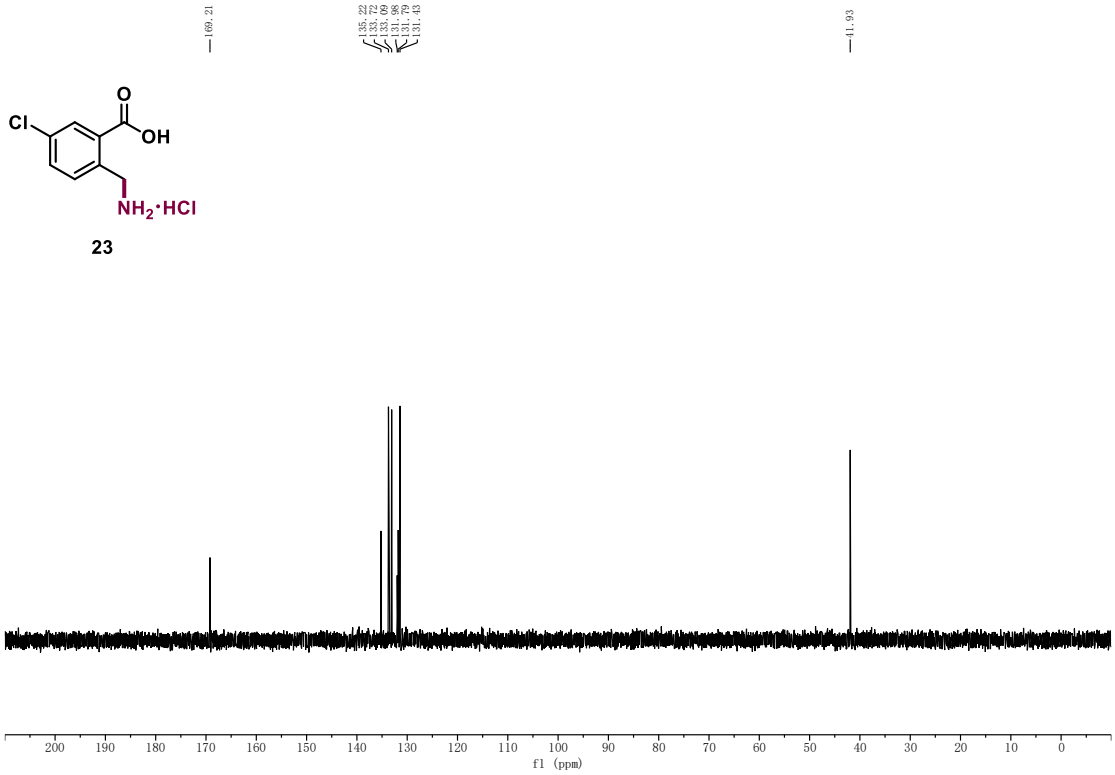

**Supplementary Figure 202** $^{1}$H NMR spectrum of compound **22**



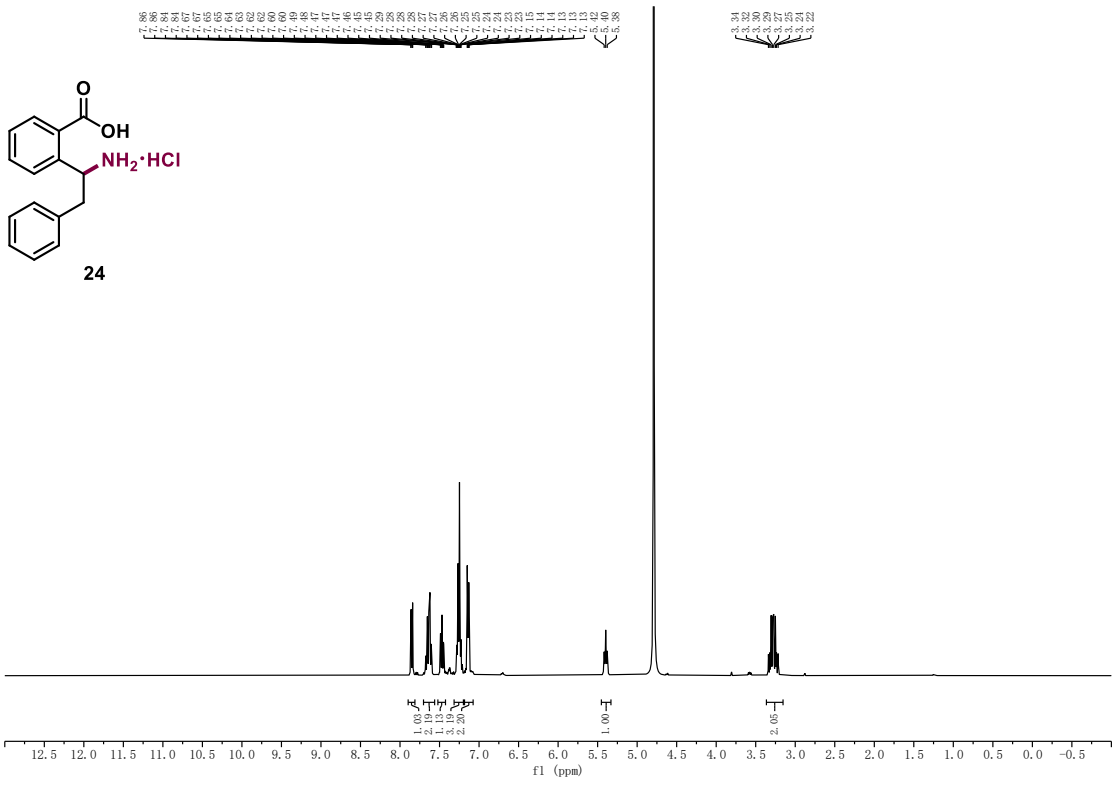

**Supplementary Figure 203** $^{13}$C NMR spectrum of compound **22**



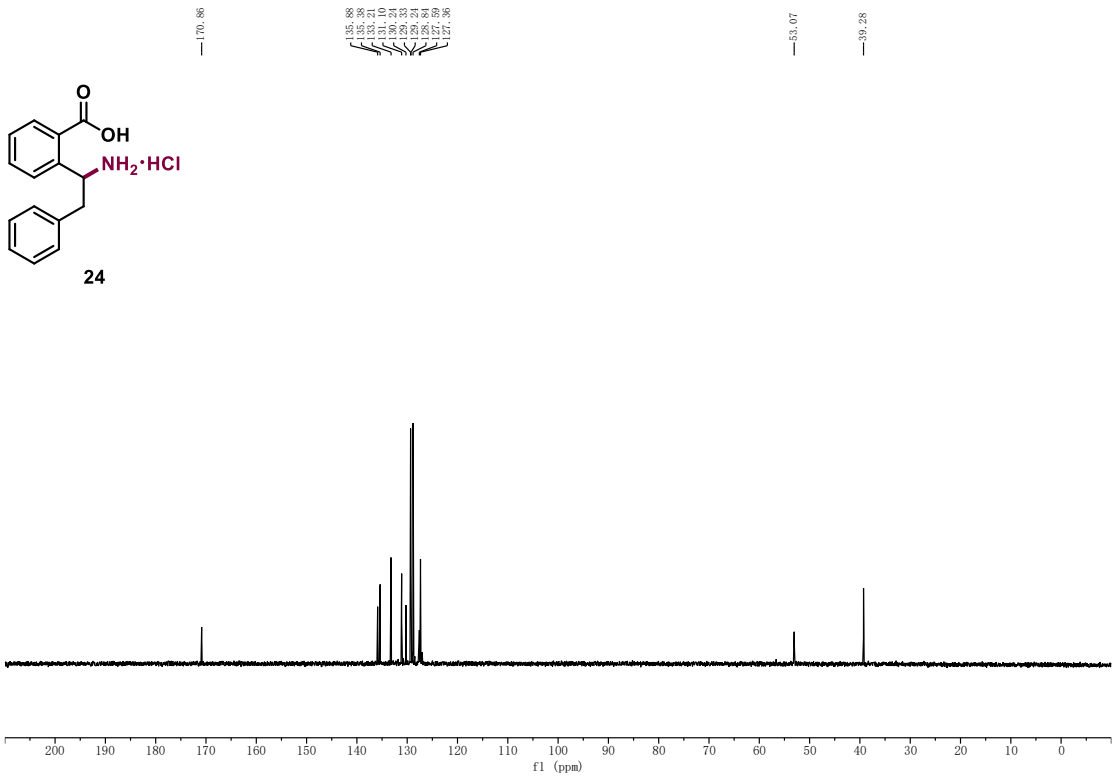

**Supplementary Figure 204** $^{1}$H NMR spectrum of compound **23**



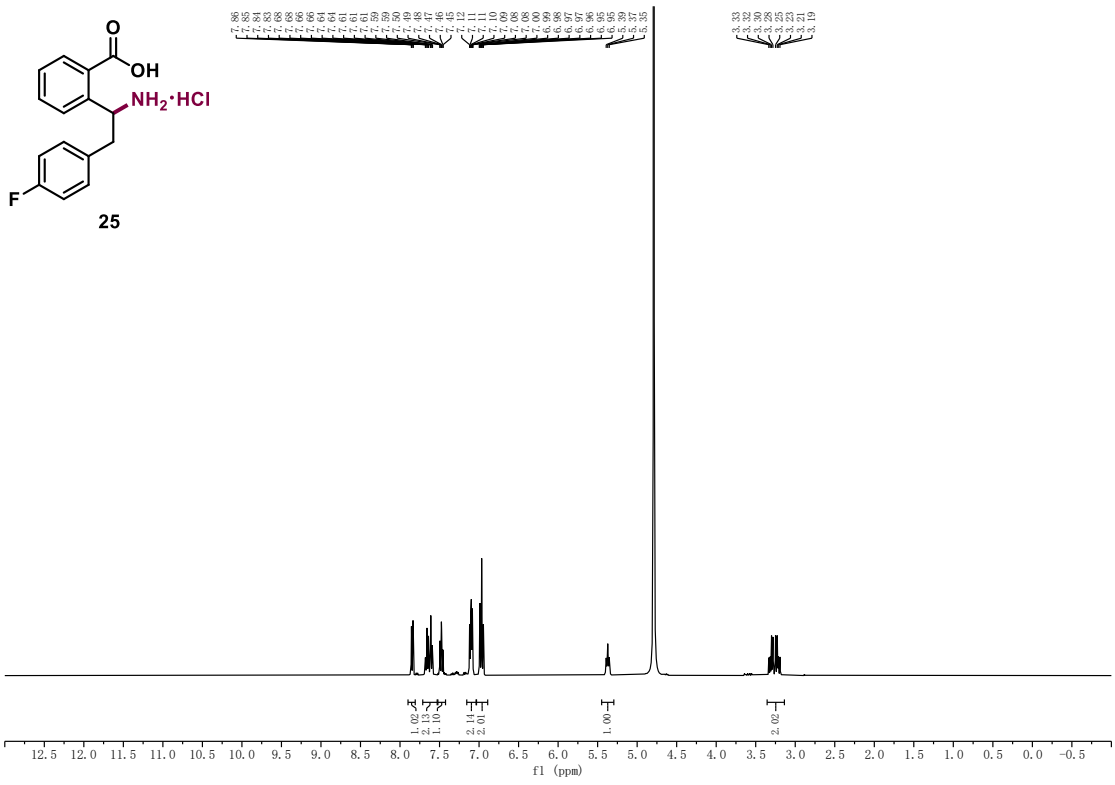

**Supplementary Figure 205** $^{13}$C NMR spectrum of compound **23**



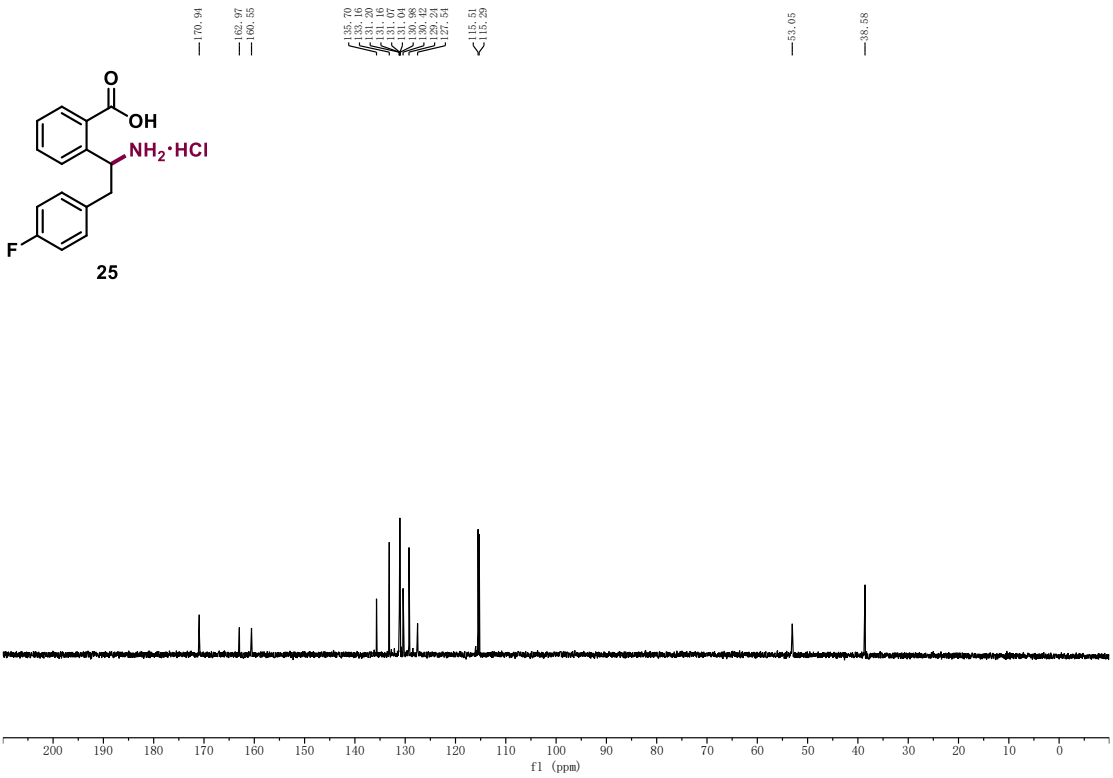

**Supplementary Figure 206** $^{1}$H NMR spectrum of compound **24**



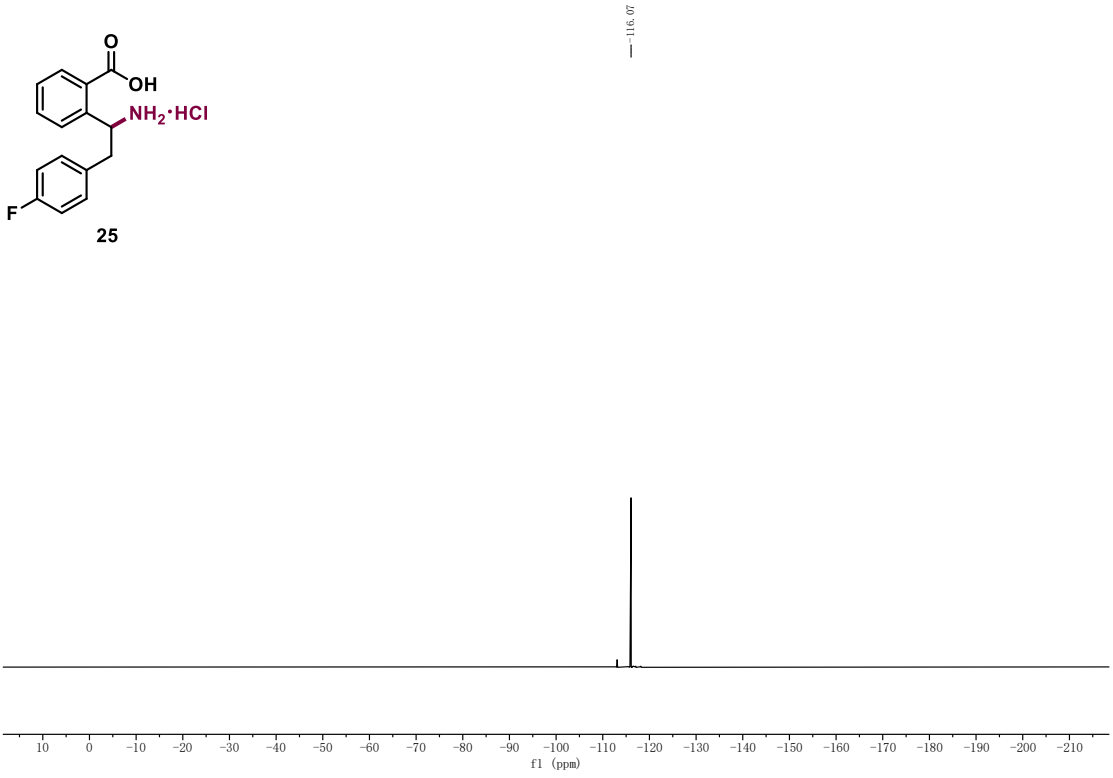

**Supplementary Figure 207** $^{13}$C NMR spectrum of compound **24**



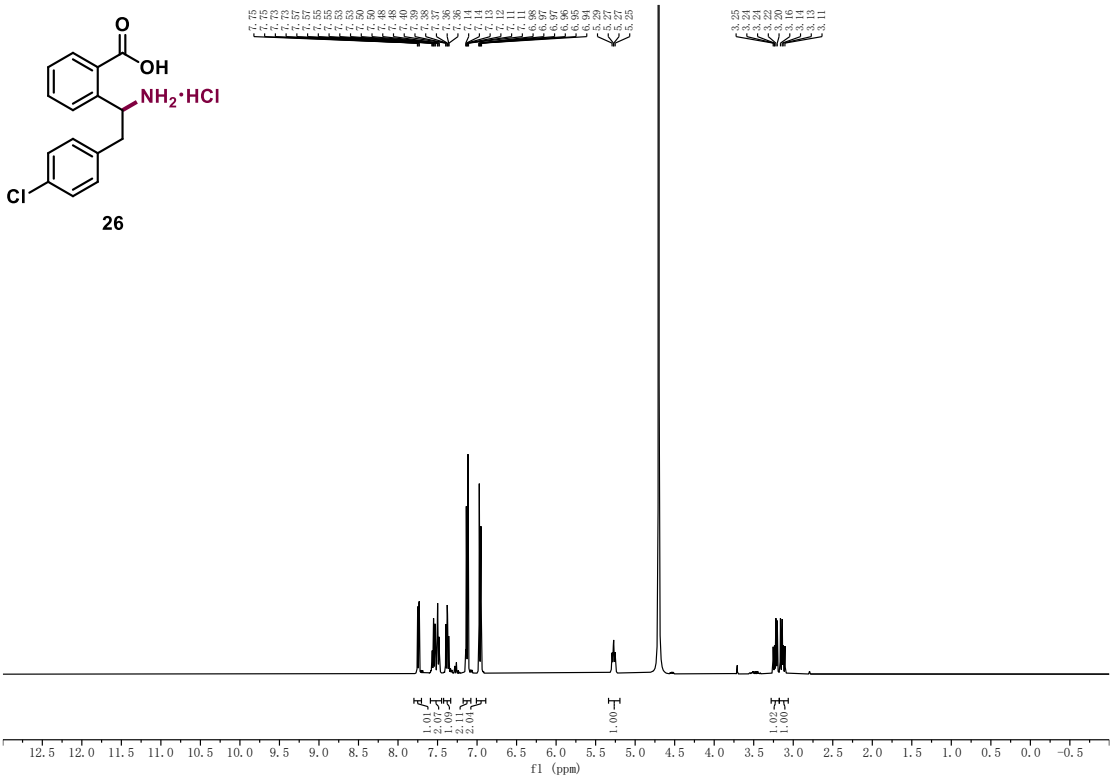

**Supplementary Figure 208** $^{1}$H NMR spectrum of compound **25**



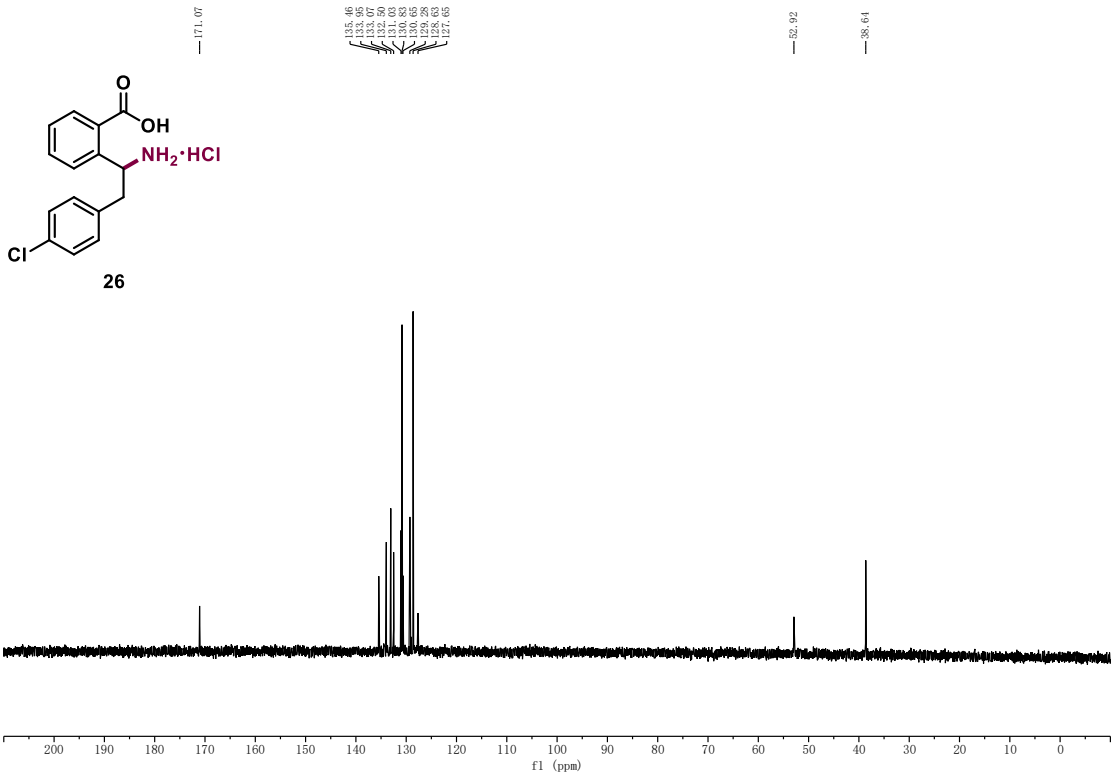

**Supplementary Figure 209** $^{13}$C NMR spectrum of compound **25**



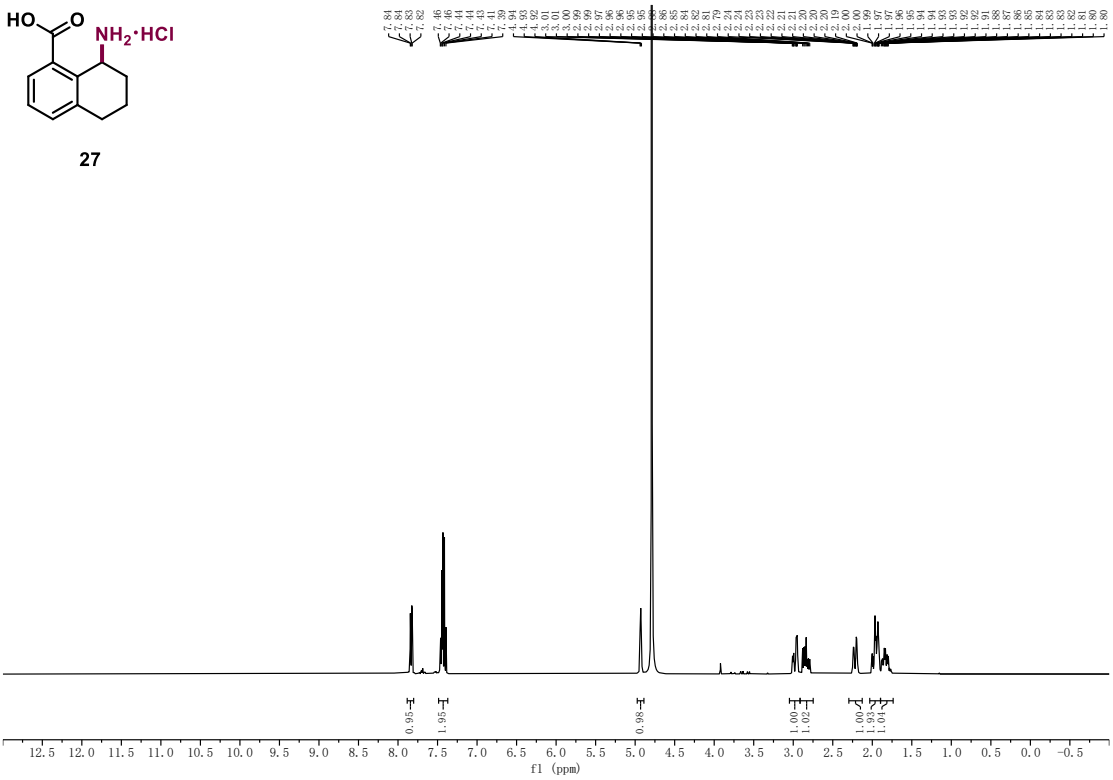

**Supplementary Figure 210** $^{19}$F NMR spectrum of compound **25**



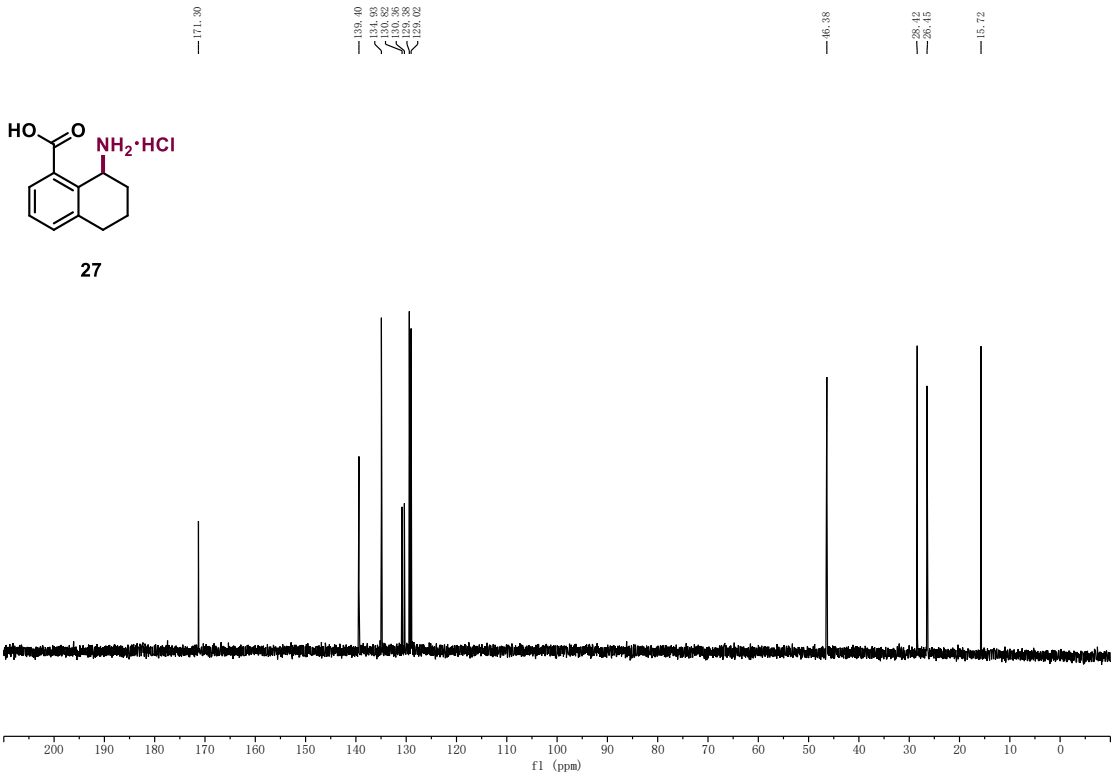

**Supplementary Figure 211** $^{1}$H NMR spectrum of compound **26**



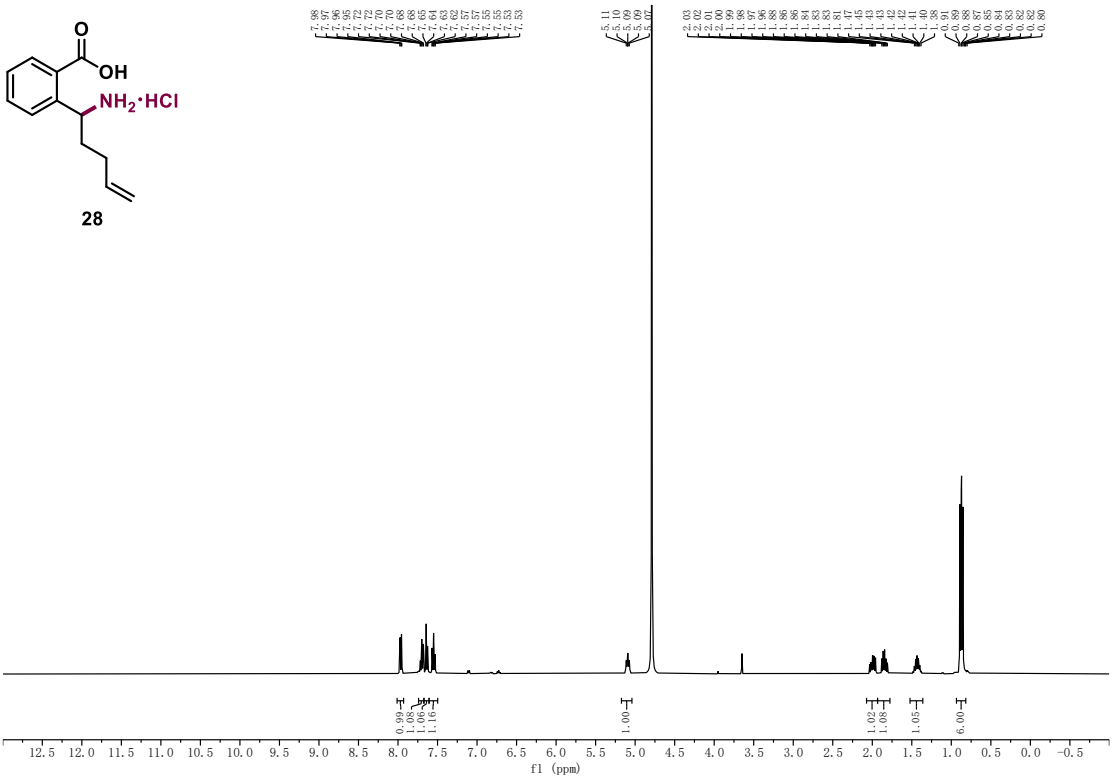

**Supplementary Figure 212** $^{13}$C NMR spectrum of compound **26**



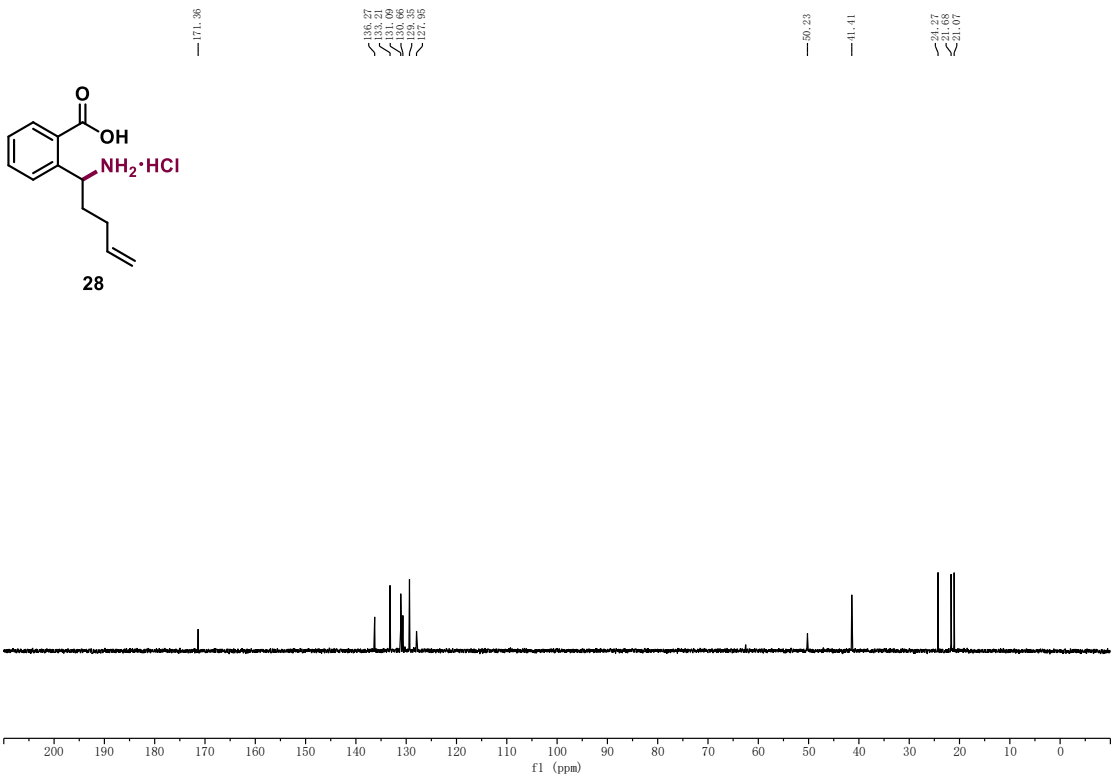

**Supplementary Figure 213** $^{1}$H NMR spectrum of compound **27**



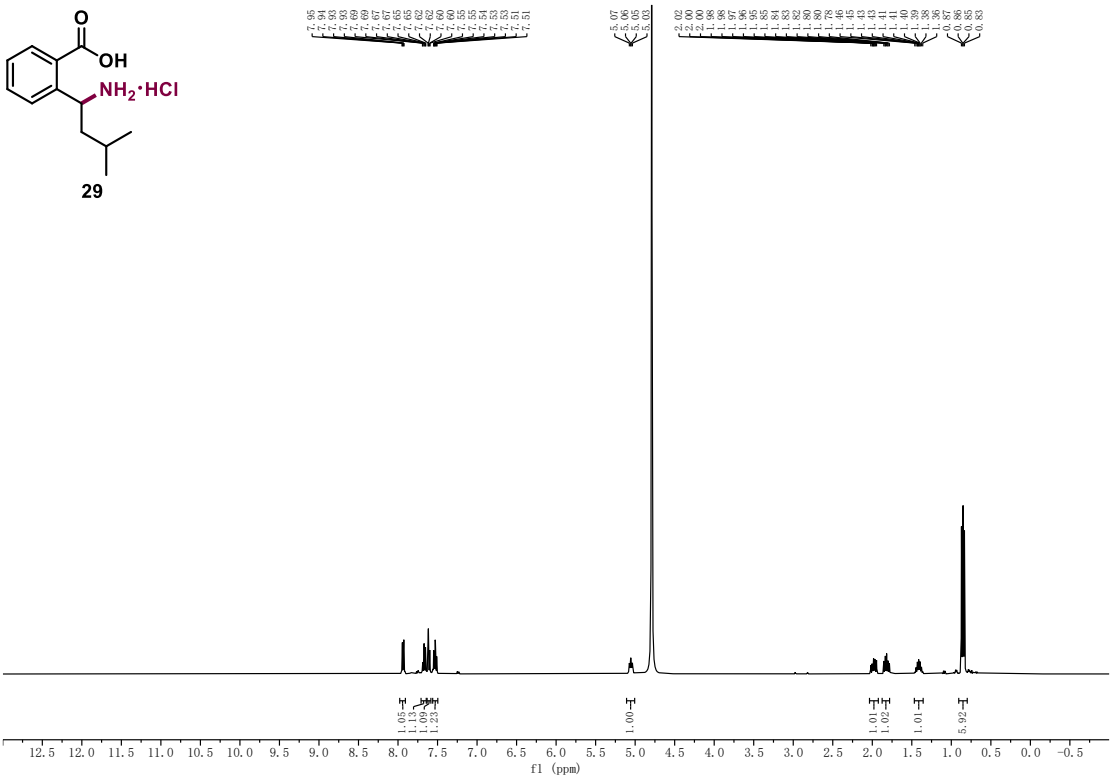

**Supplementary Figure 214** $^{13}$C NMR spectrum of compound **27**



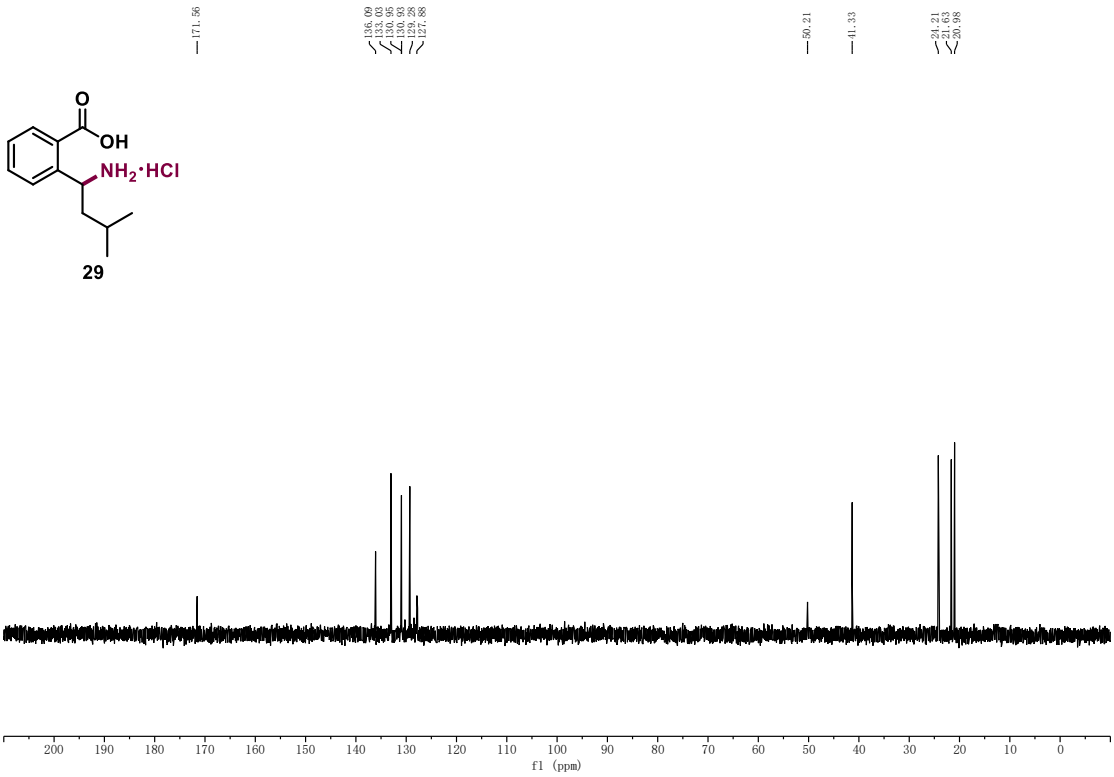

**Supplementary Figure 215** $^{1}$H NMR spectrum of compound **28**



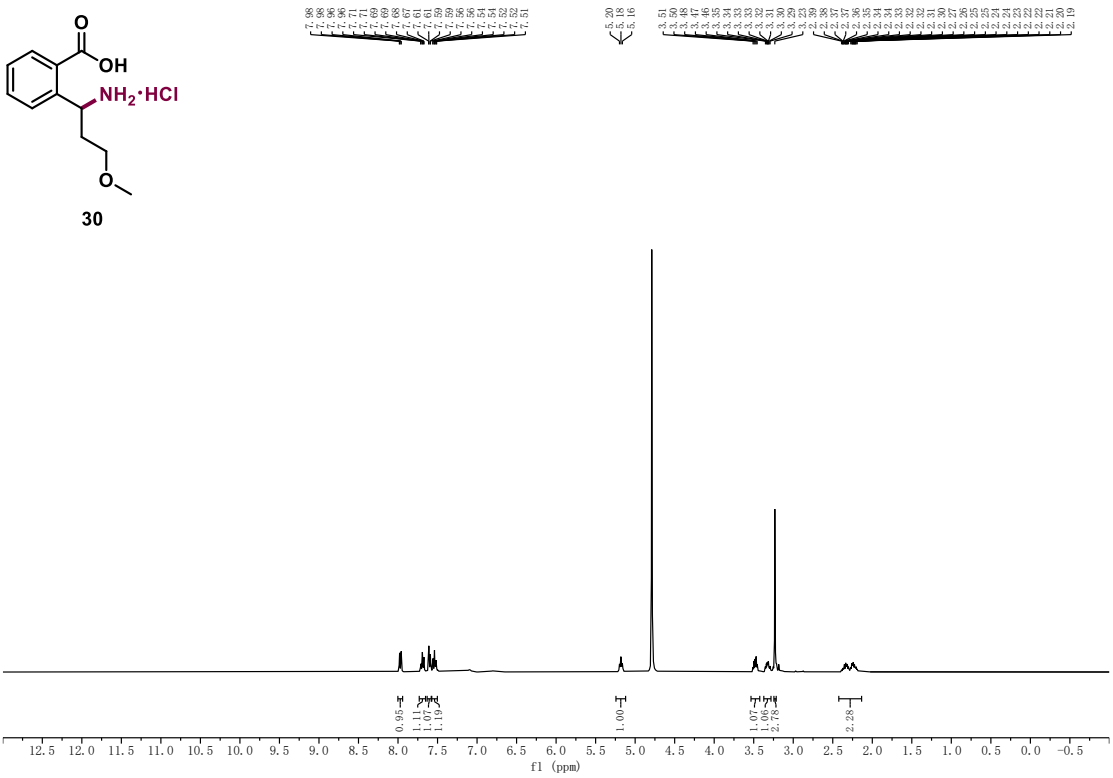

**Supplementary Figure 216** $^{13}$C NMR spectrum of compound **28**



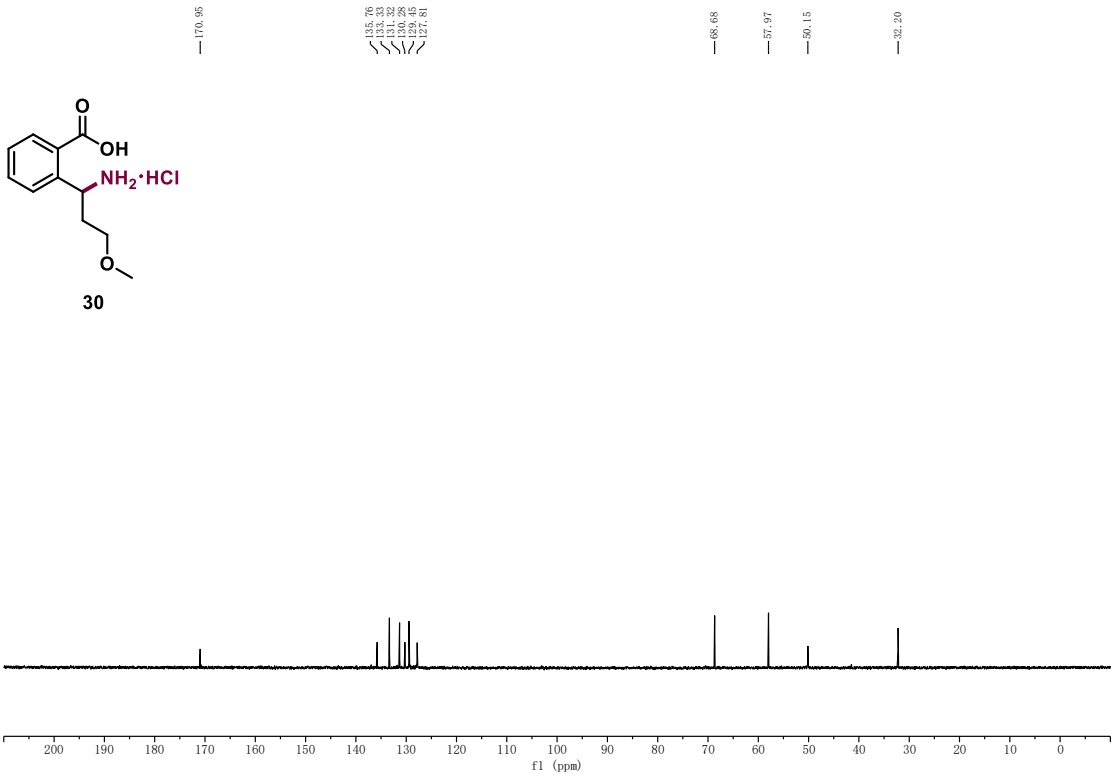

**Supplementary Figure 217** $^{1}$H NMR spectrum of compound **29**



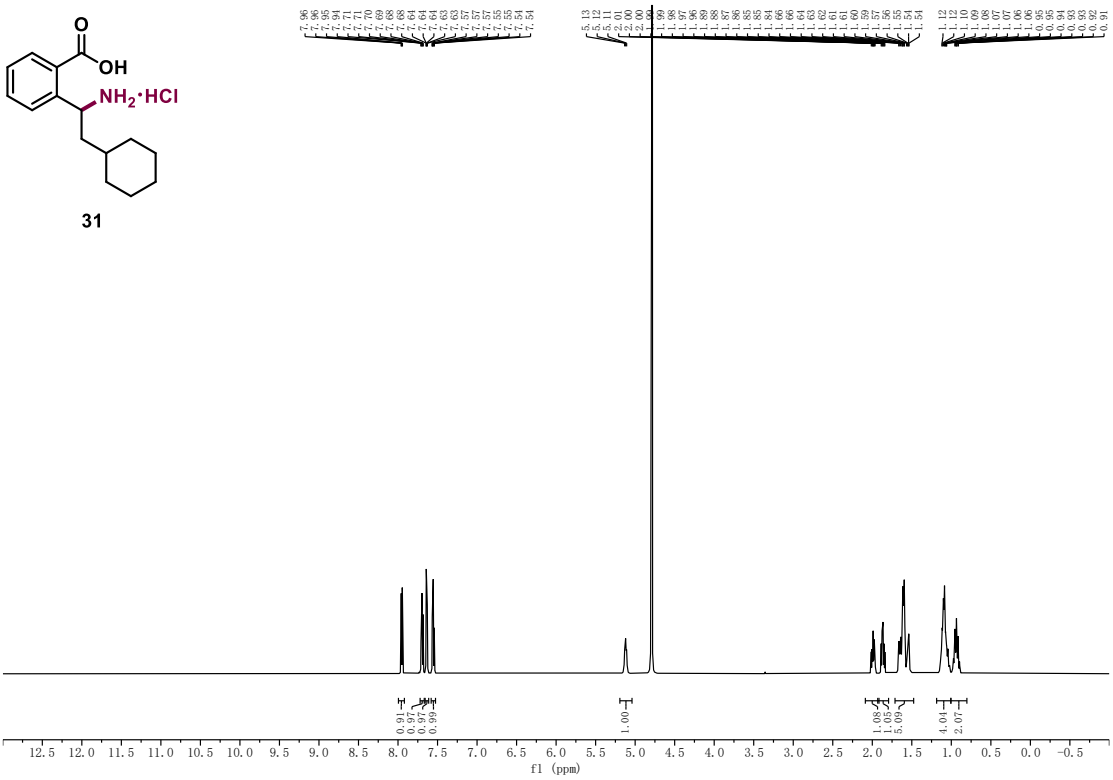

**Supplementary Figure 218** $^{13}$C NMR spectrum of compound **29**



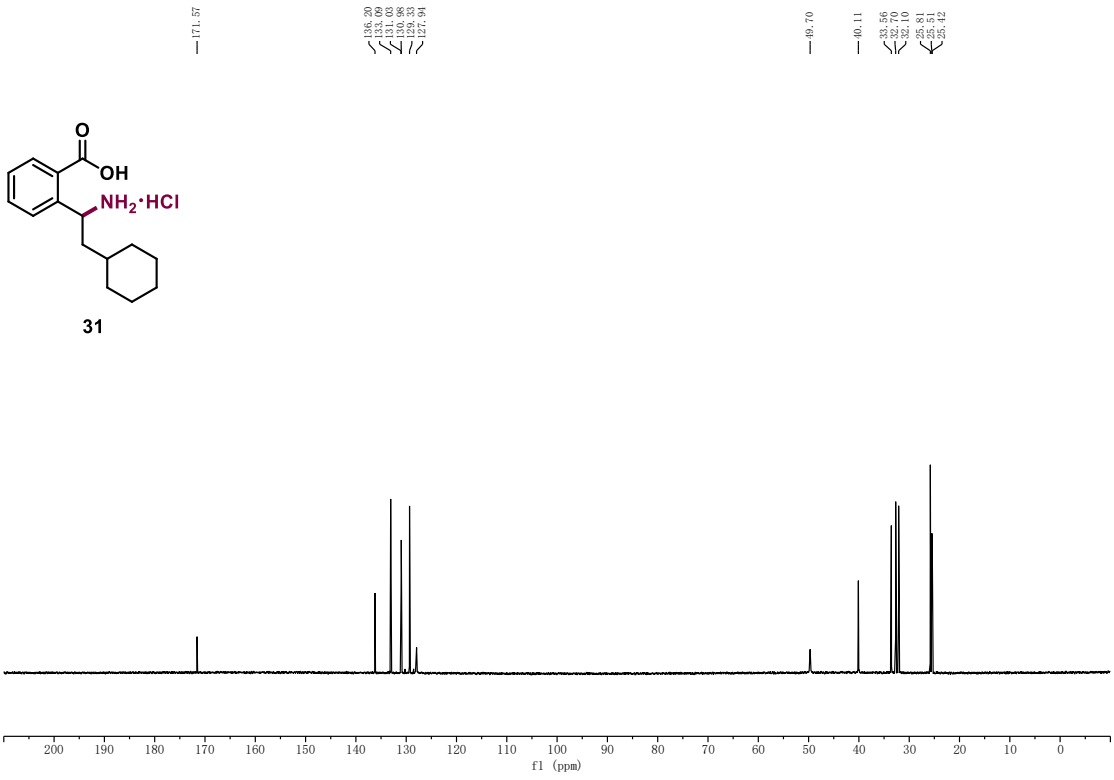

**Supplementary Figure 219** $^{1}$H NMR spectrum of compound **30**



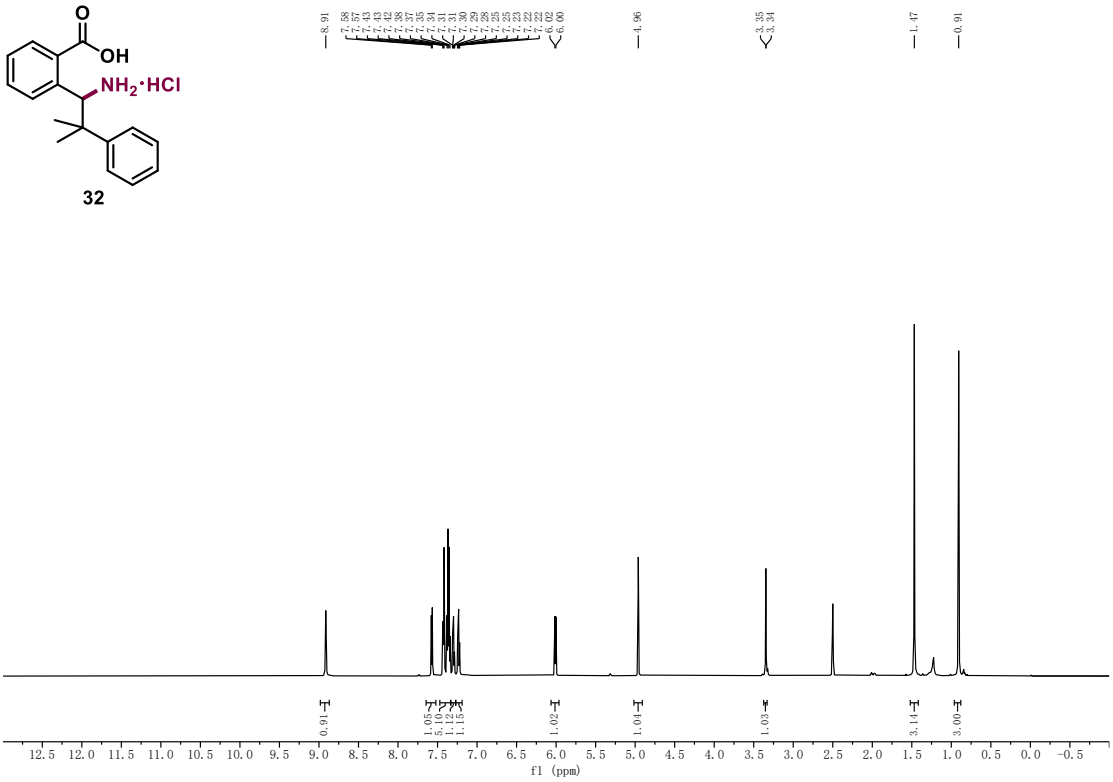

**Supplementary Figure 220** $^{13}$C NMR spectrum of compound **30**



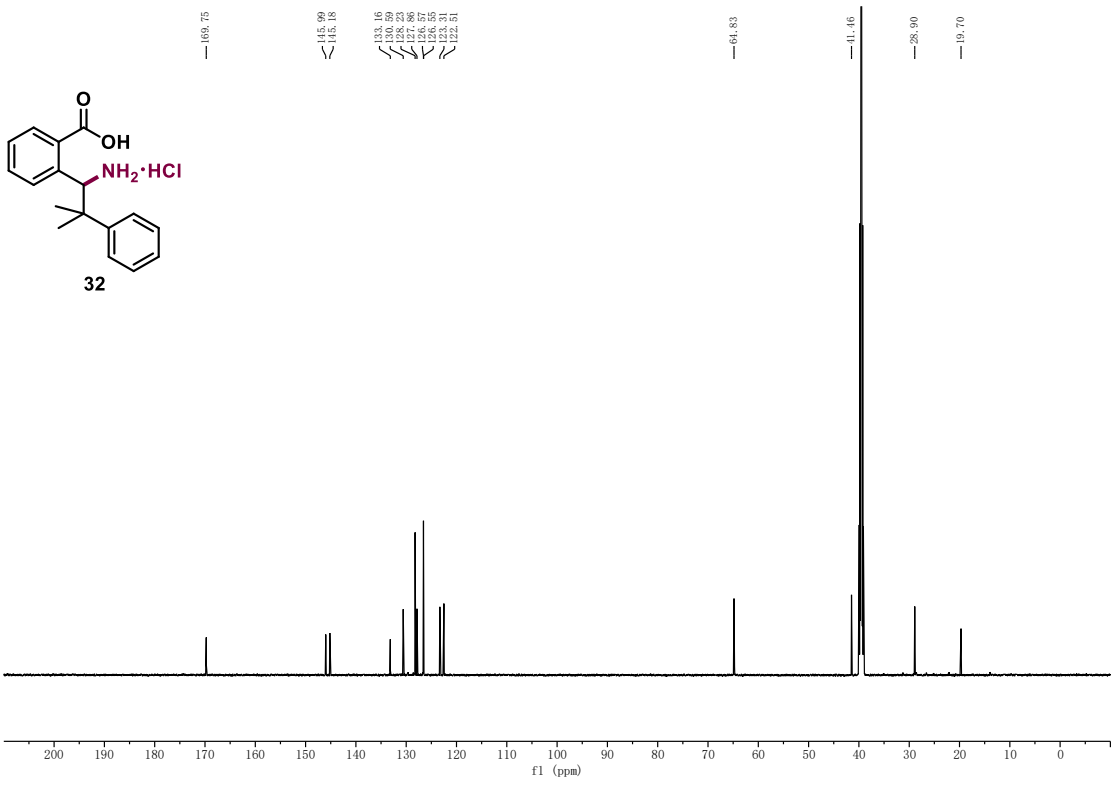

**Supplementary Figure 221** $^{1}$H NMR spectrum of compound **31**



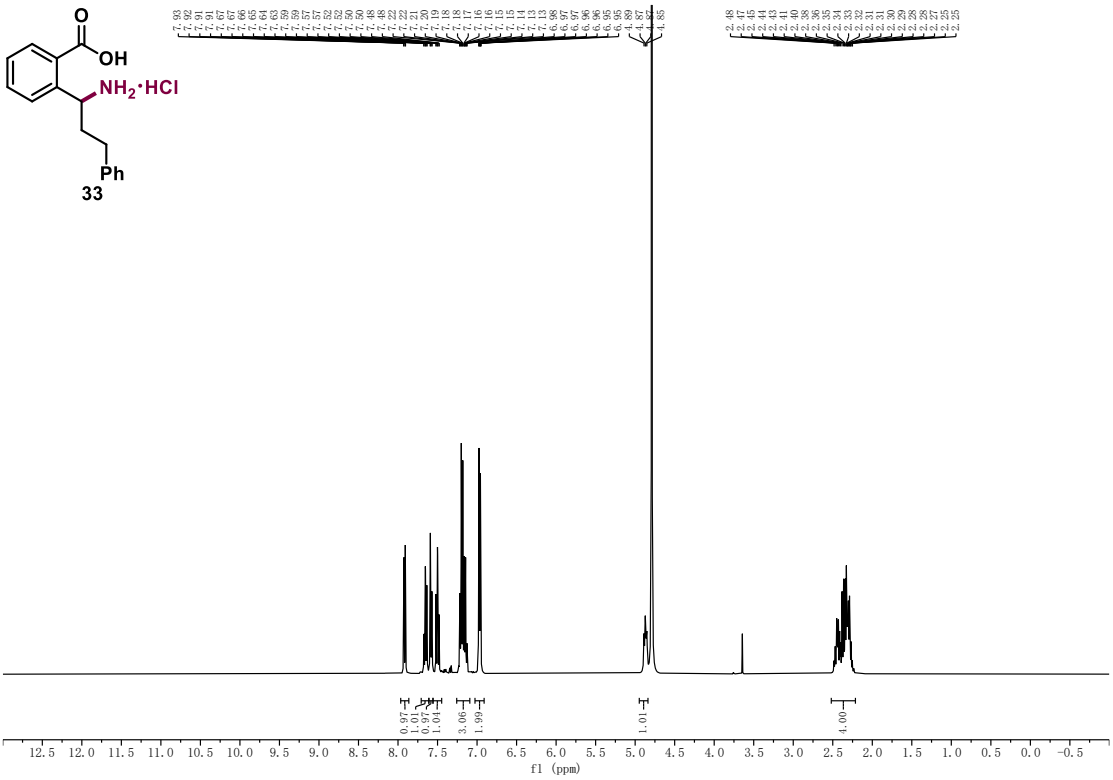

**Supplementary Figure 222** $^{13}$C NMR spectrum of compound **31**



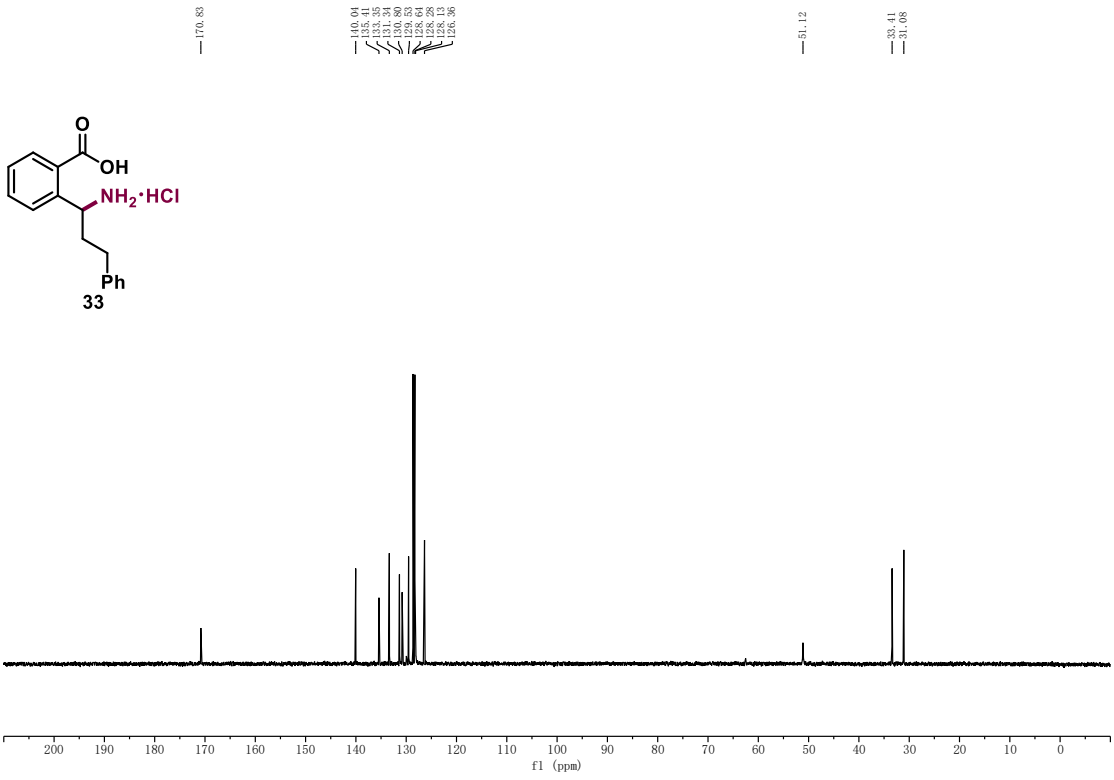

**Supplementary Figure 223** $^{1}$H NMR spectrum of compound **32**



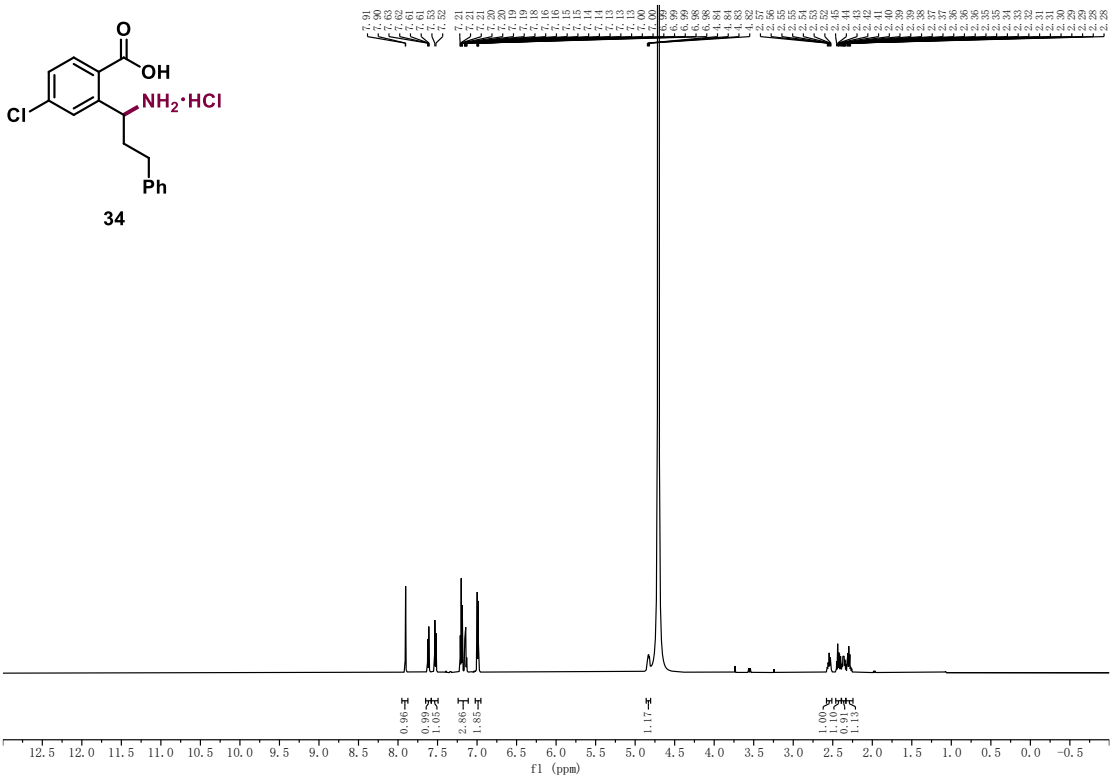

**Supplementary Figure 224** $^{13}$C NMR spectrum of compound **32**



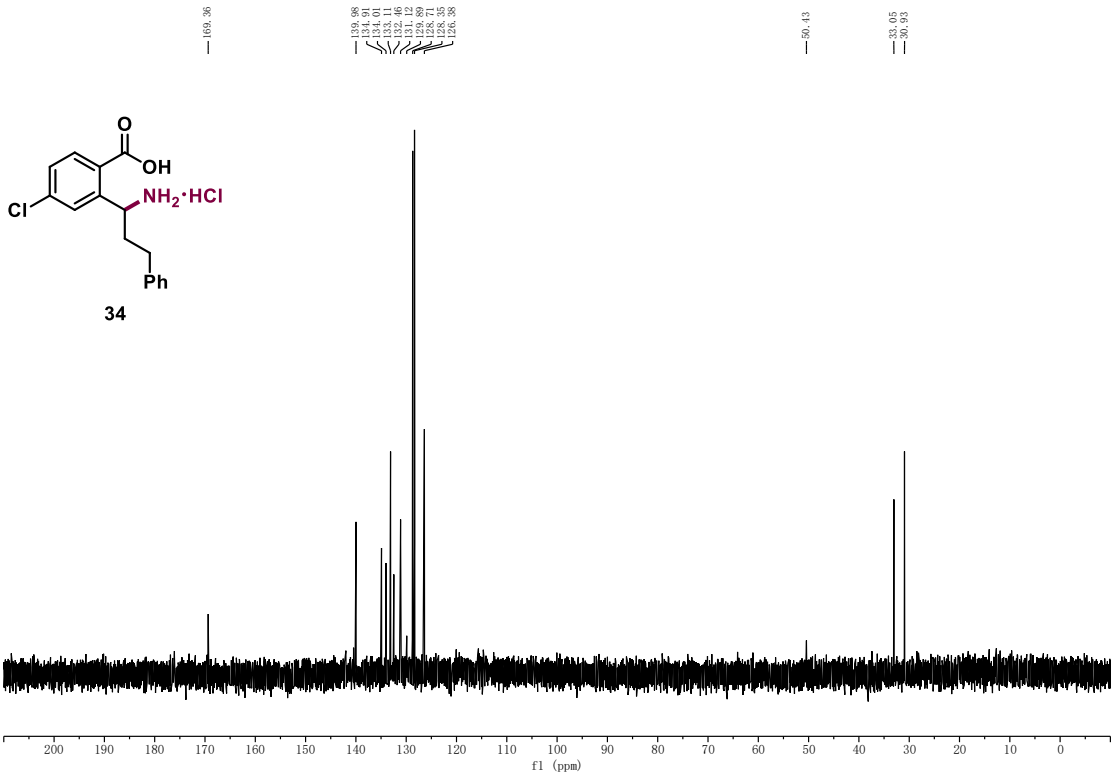

**Supplementary Figure 225** $^{1}$H NMR spectrum of compound **33**



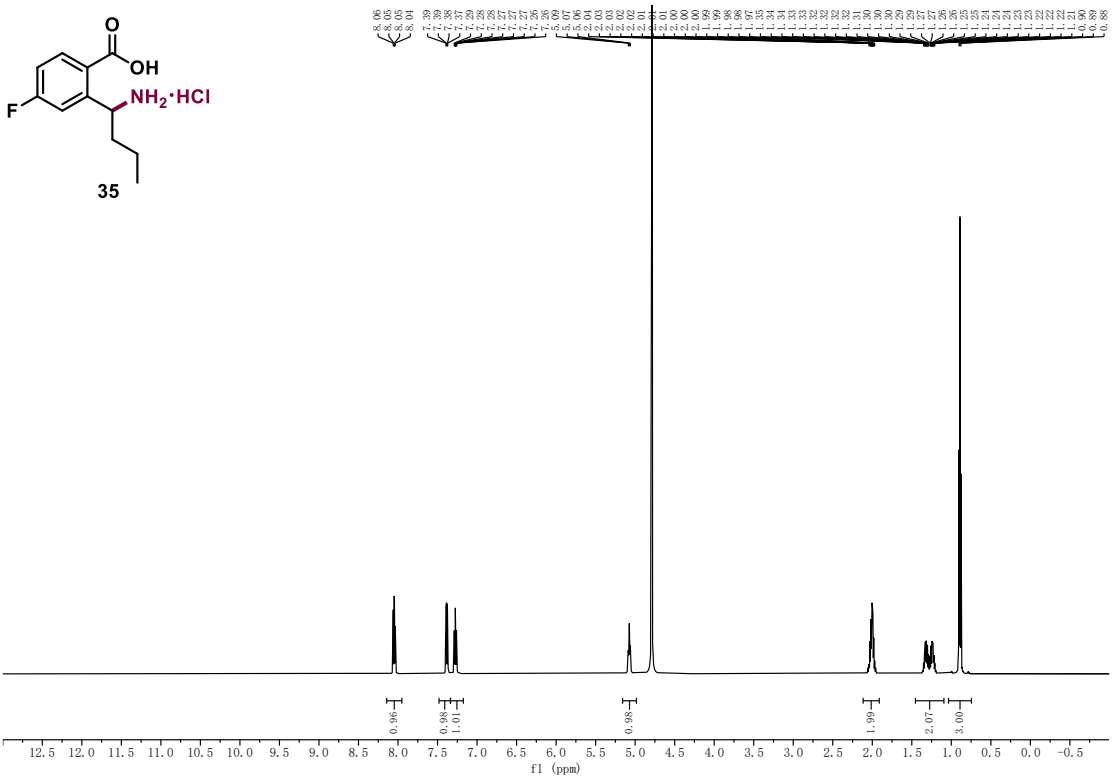

**Supplementary Figure 226** $^{13}$C NMR spectrum of compound **33**



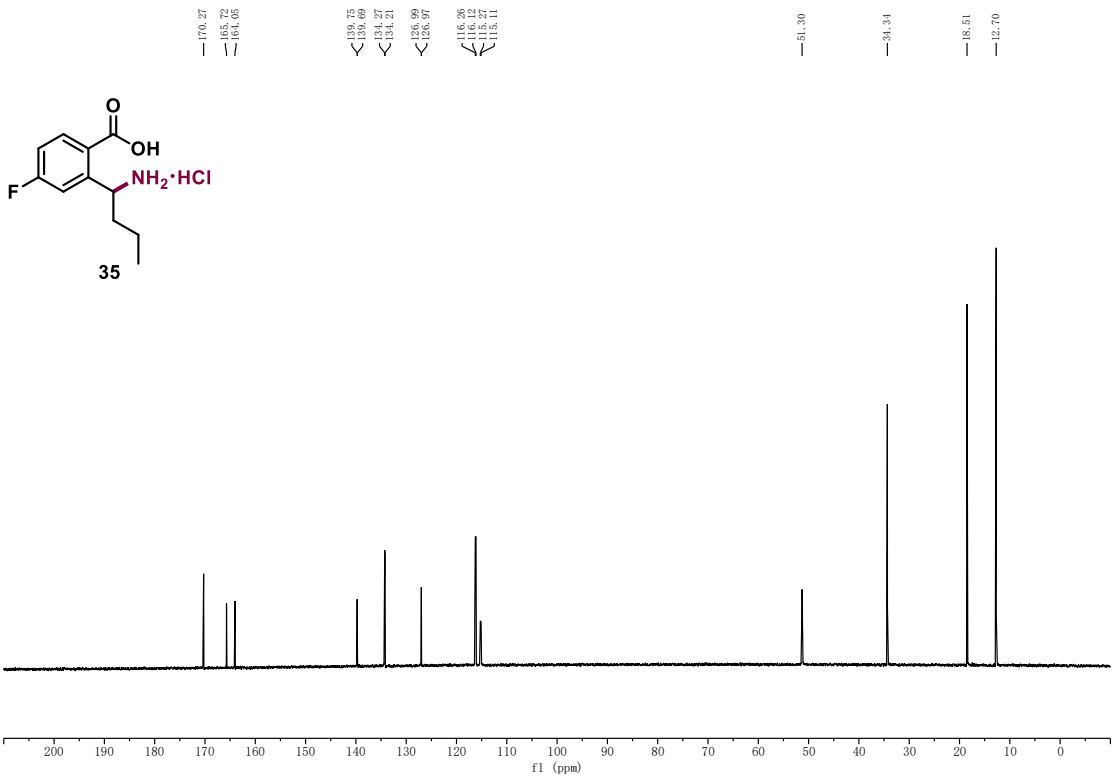

**Supplementary Figure 227** $^{1}$H NMR spectrum of compound **34**



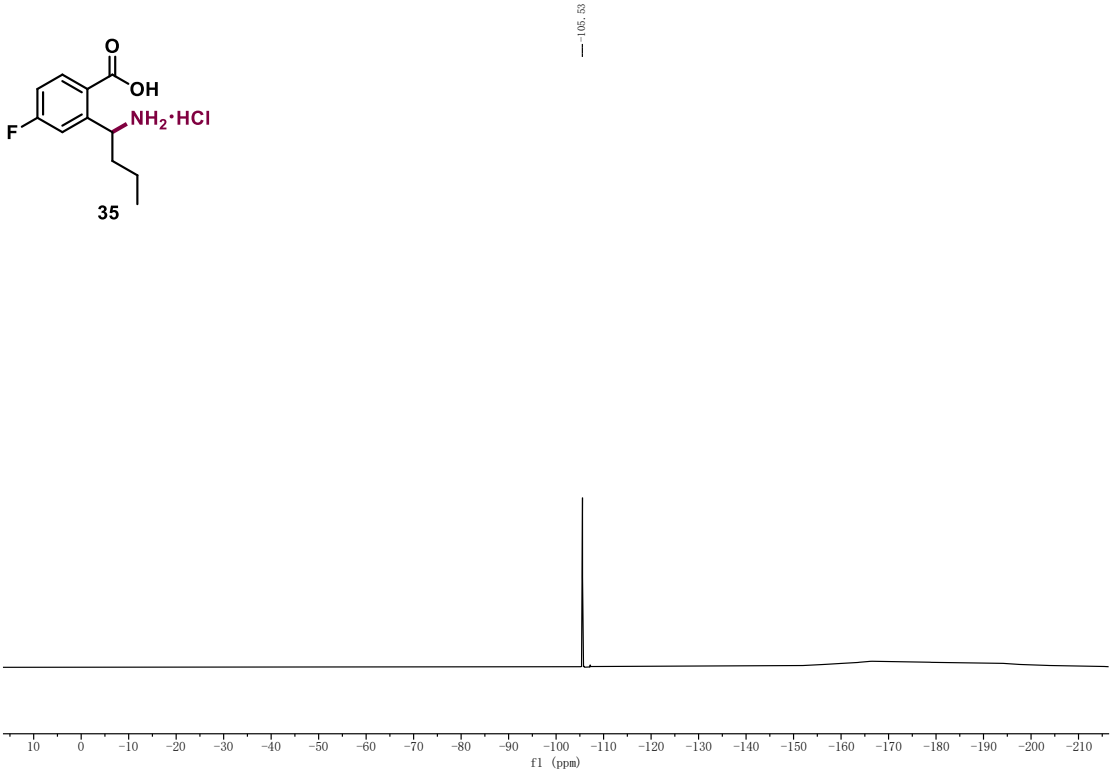

**Supplementary Figure 228** $^{13}$C NMR spectrum of compound **34**



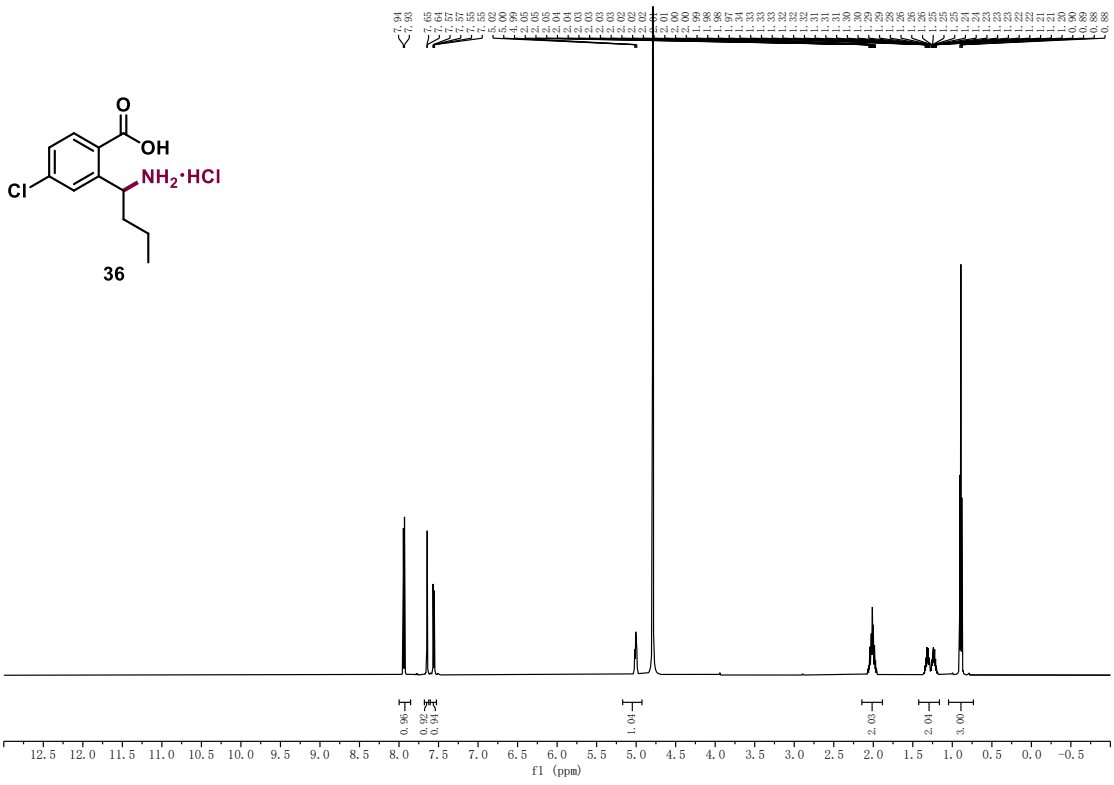

**Supplementary Figure 229** $^{1}$H NMR spectrum of compound **35**



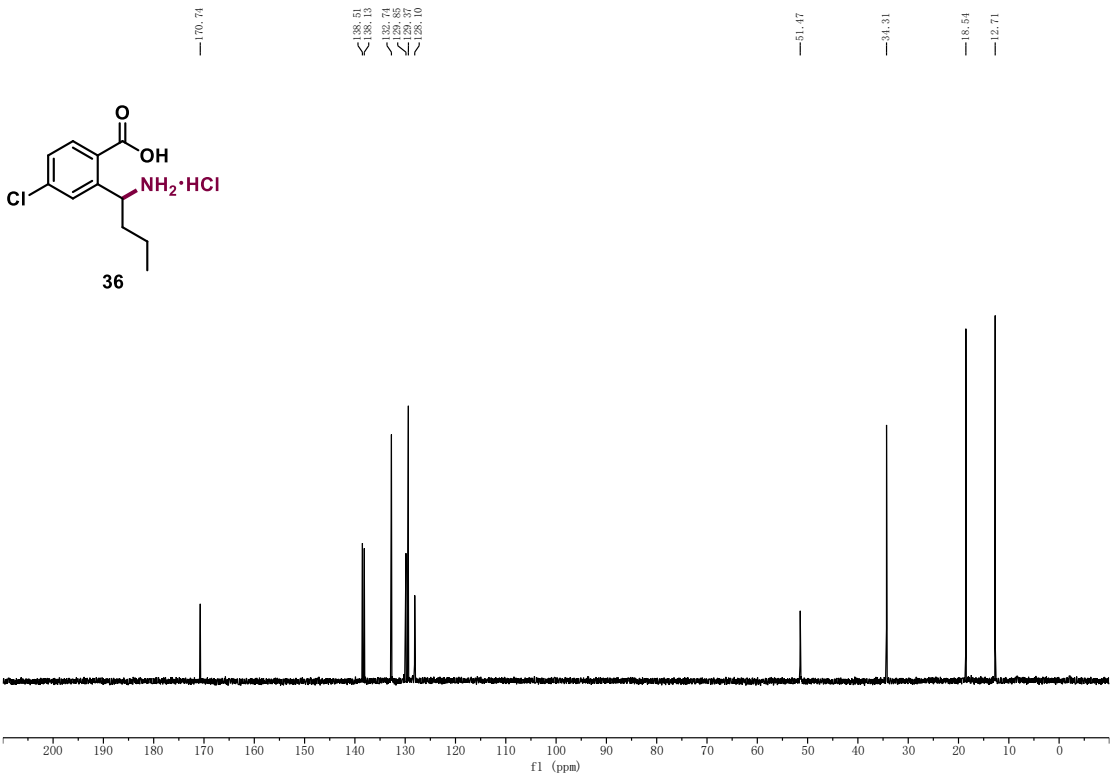

**Supplementary Figure 230** $^{13}$C NMR spectrum of compound **35**



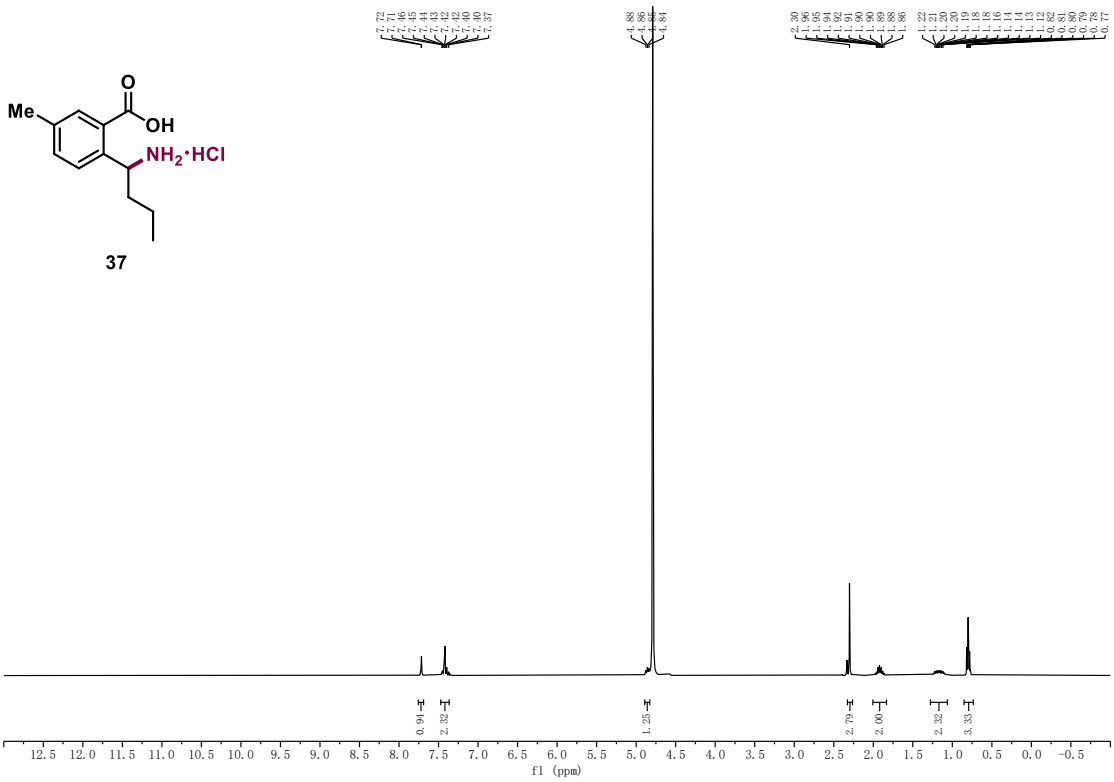

**Supplementary Figure 231** $^{19}$F NMR spectrum of compound **35**



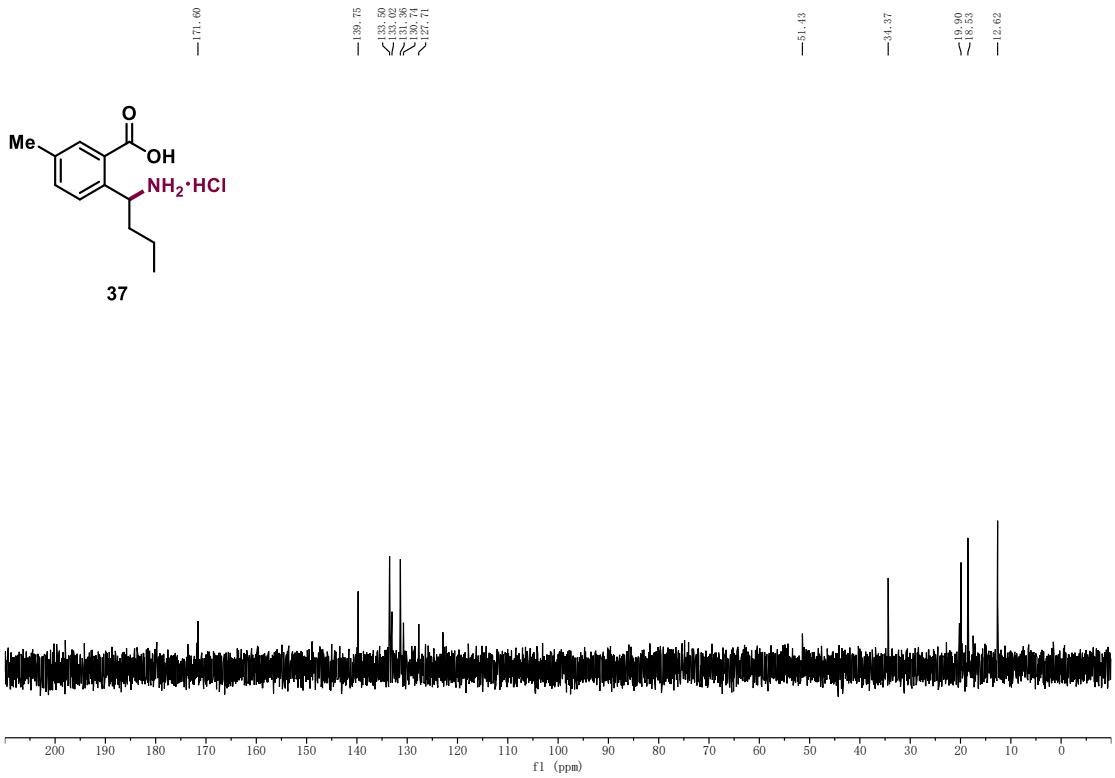

**Supplementary Figure 232** $^{1}$H NMR spectrum of compound **36**



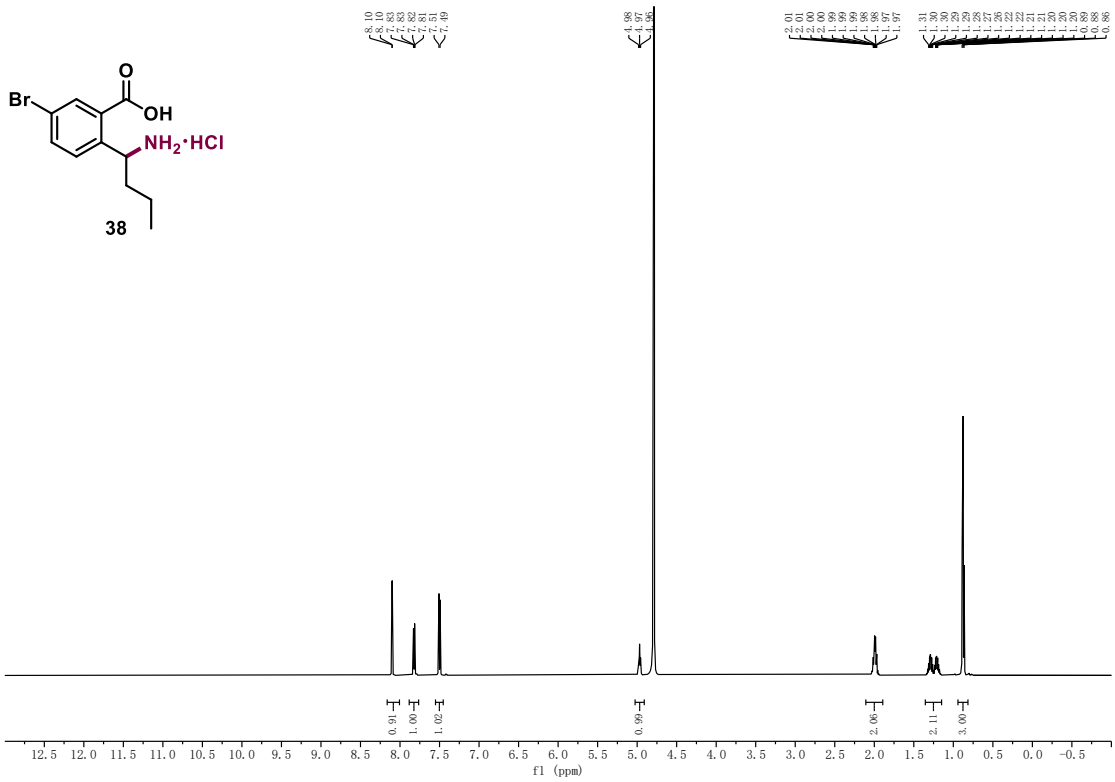

**Supplementary Figure 233** $^{13}$C NMR spectrum of compound **36**



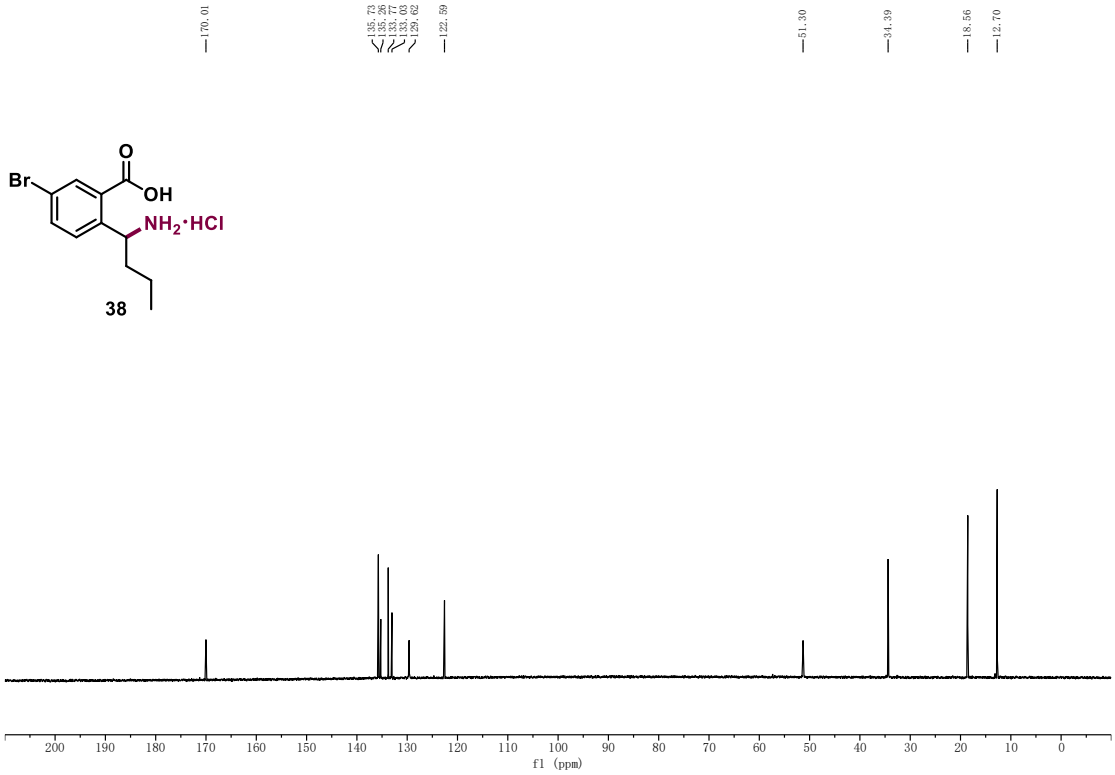

**Supplementary Figure 234** $^{1}$H NMR spectrum of compound **37**



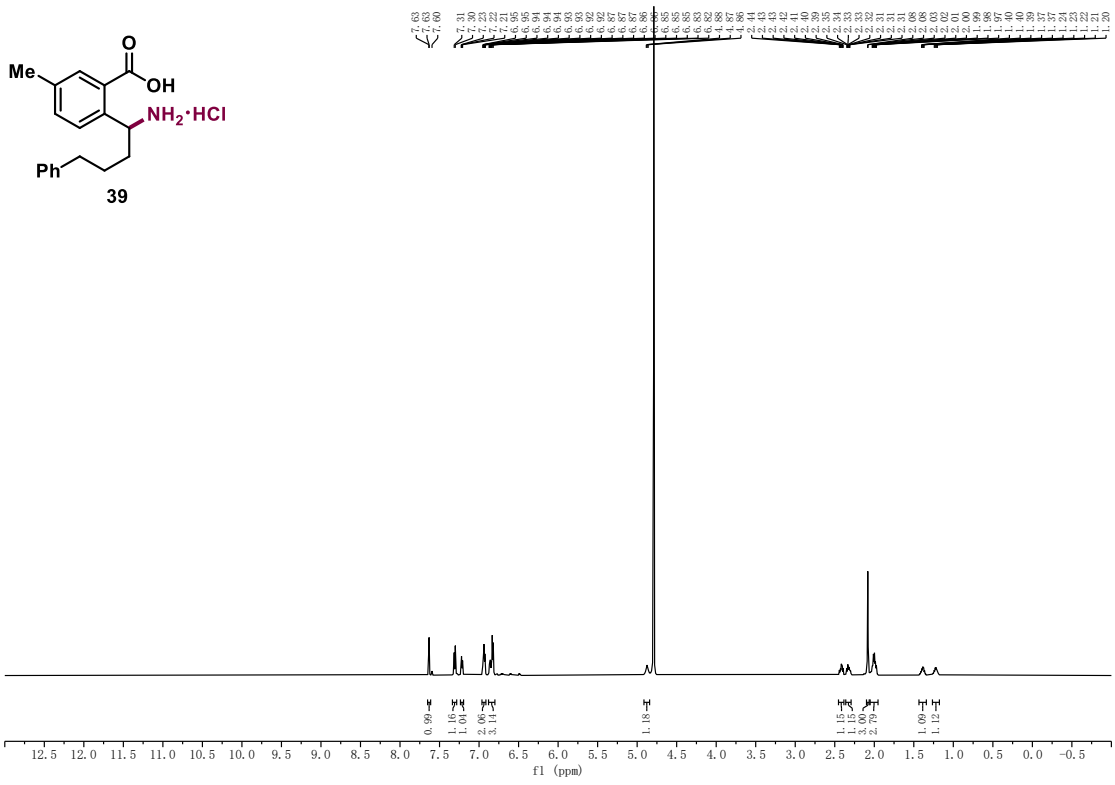

**Supplementary Figure 235** $^{13}$C NMR spectrum of compound **37**



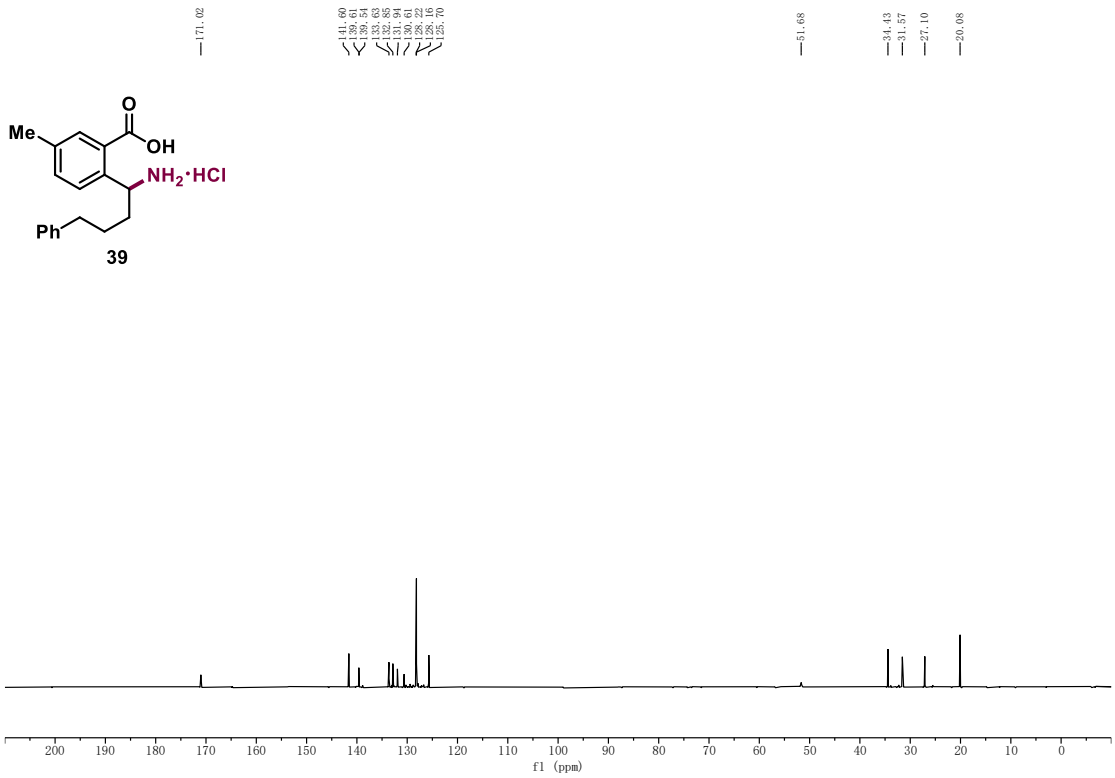

**Supplementary Figure 236** $^{1}$H NMR spectrum of compound **38**



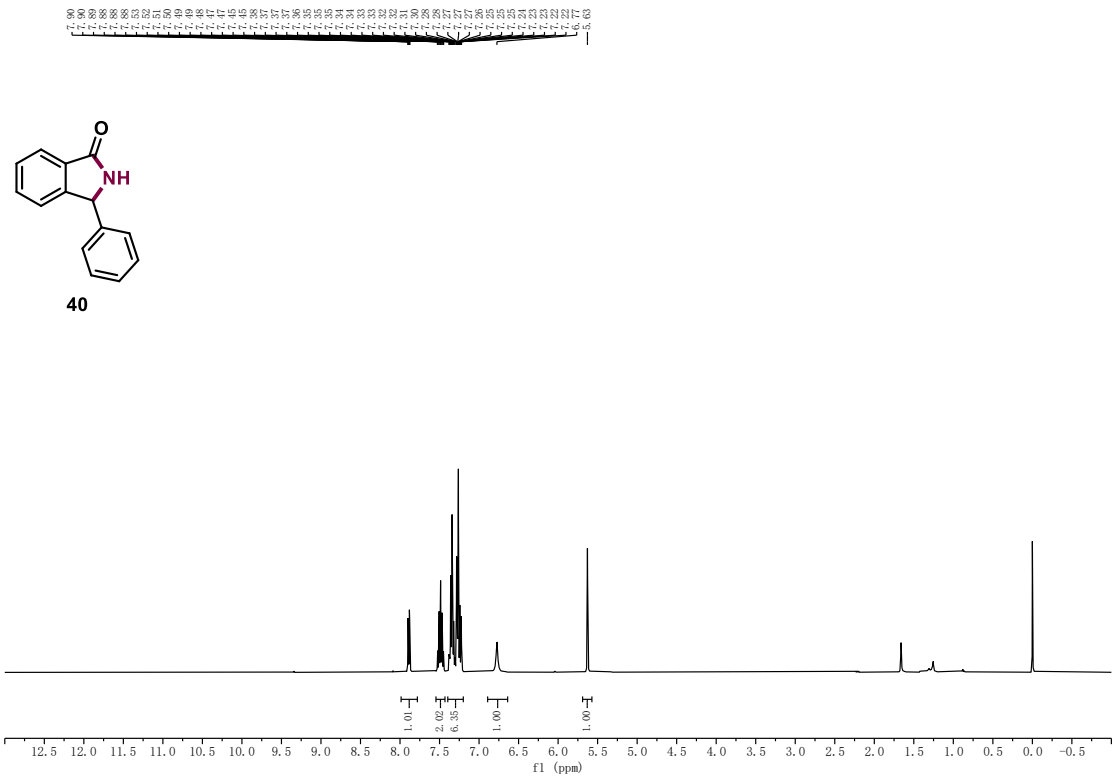

**Supplementary Figure 237** $^{13}$C NMR spectrum of compound **38**



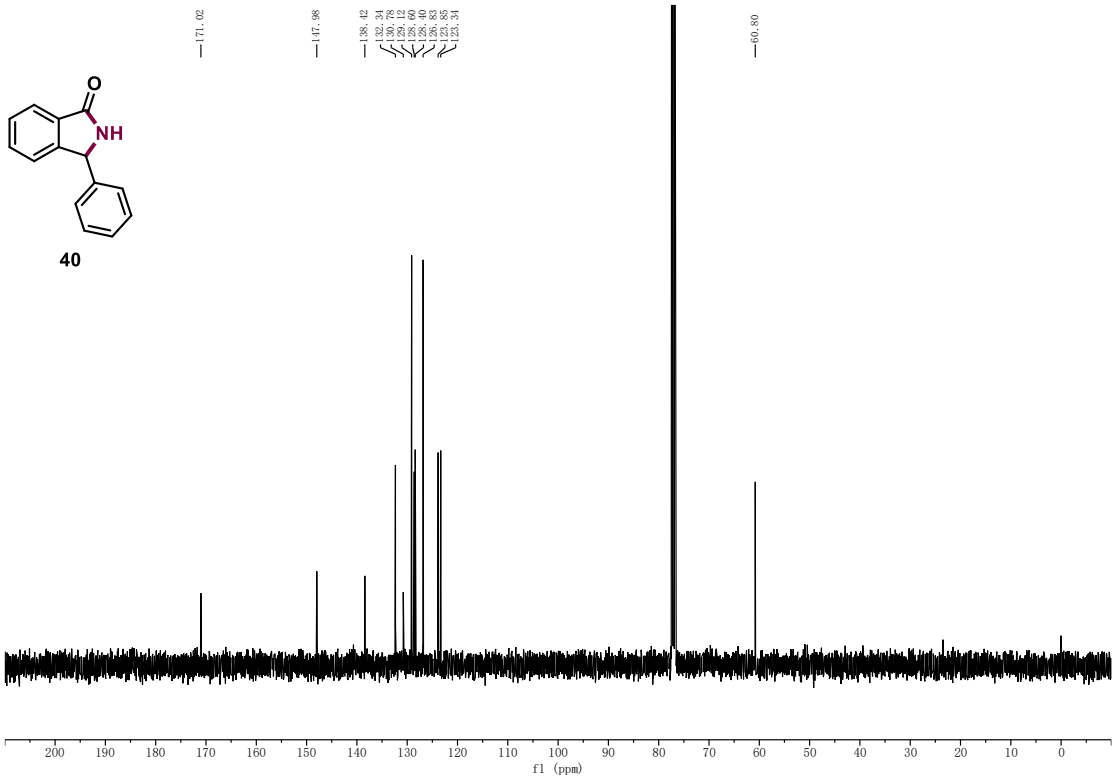

**Supplementary Figure 238** $^{1}$H NMR spectrum of compound **39**



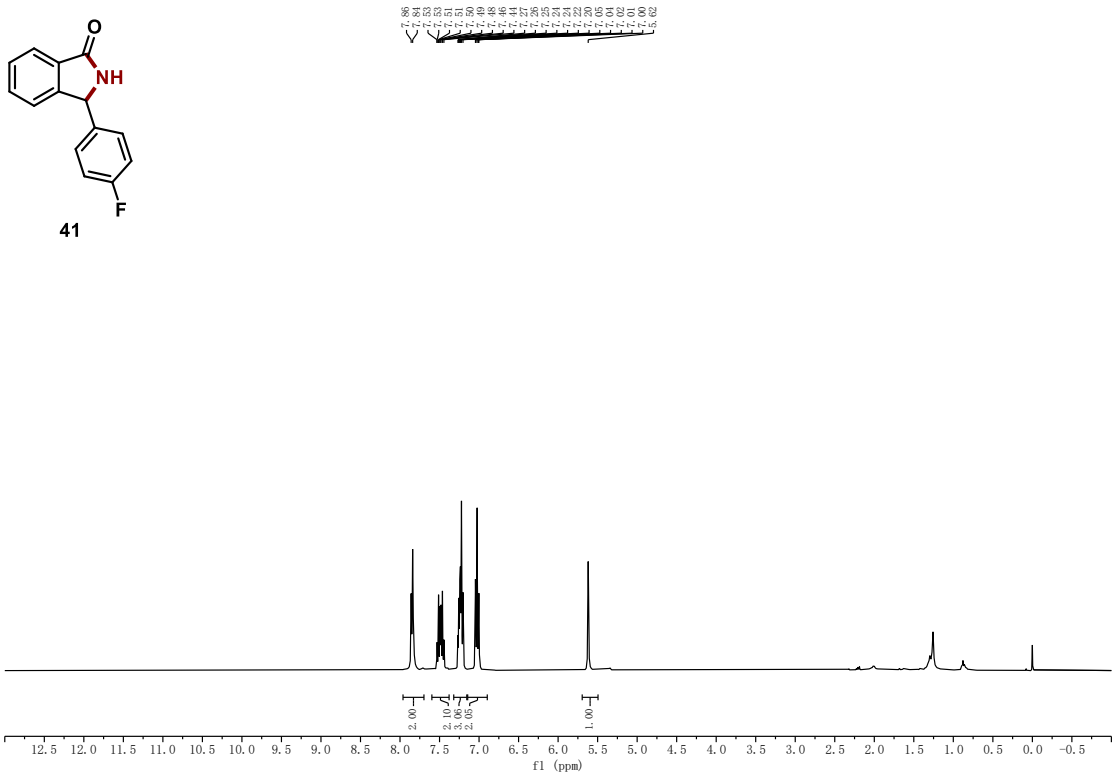

**Supplementary Figure 239** $^{13}$C NMR spectrum of compound **39**



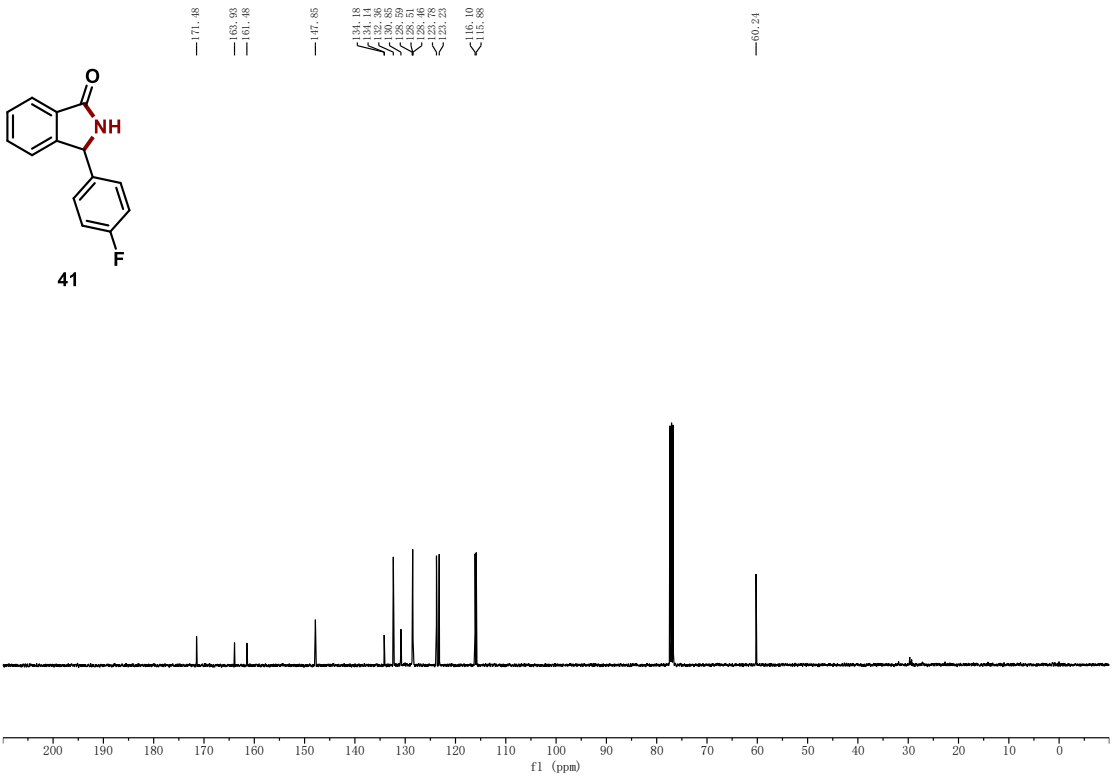

**Supplementary Figure 240** $^{1}$H NMR spectrum of compound **40**



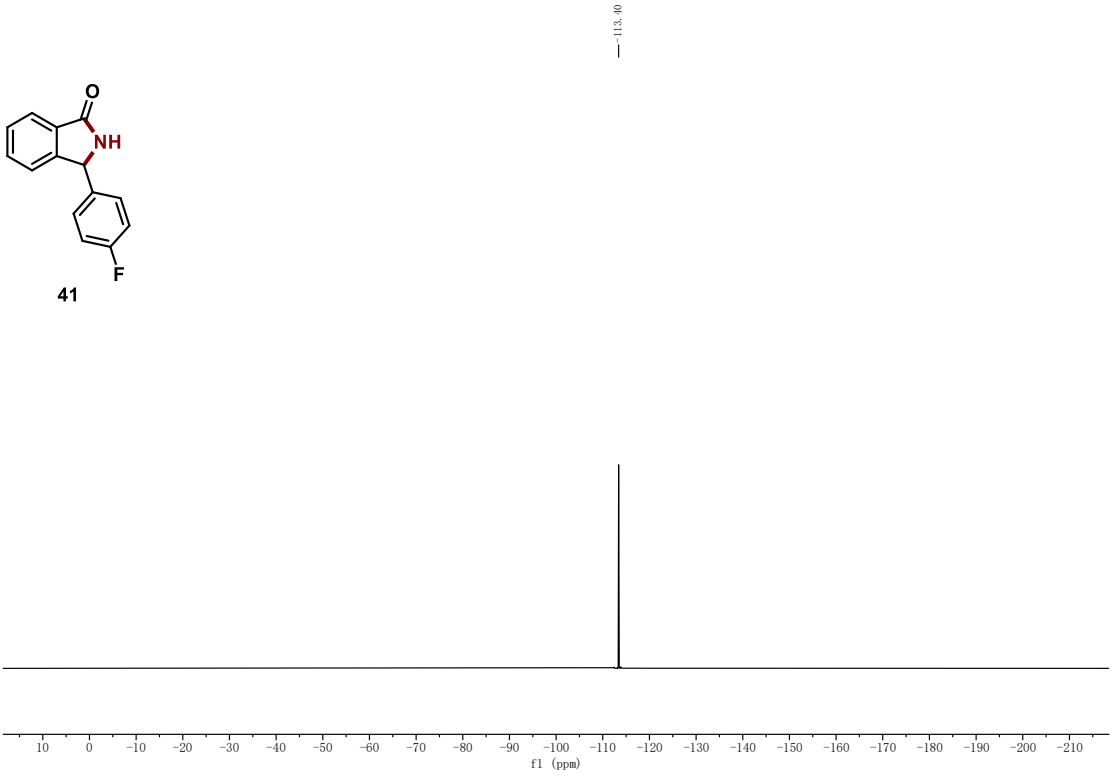

**Supplementary Figure 241** $^{13}$C NMR spectrum of compound **40**



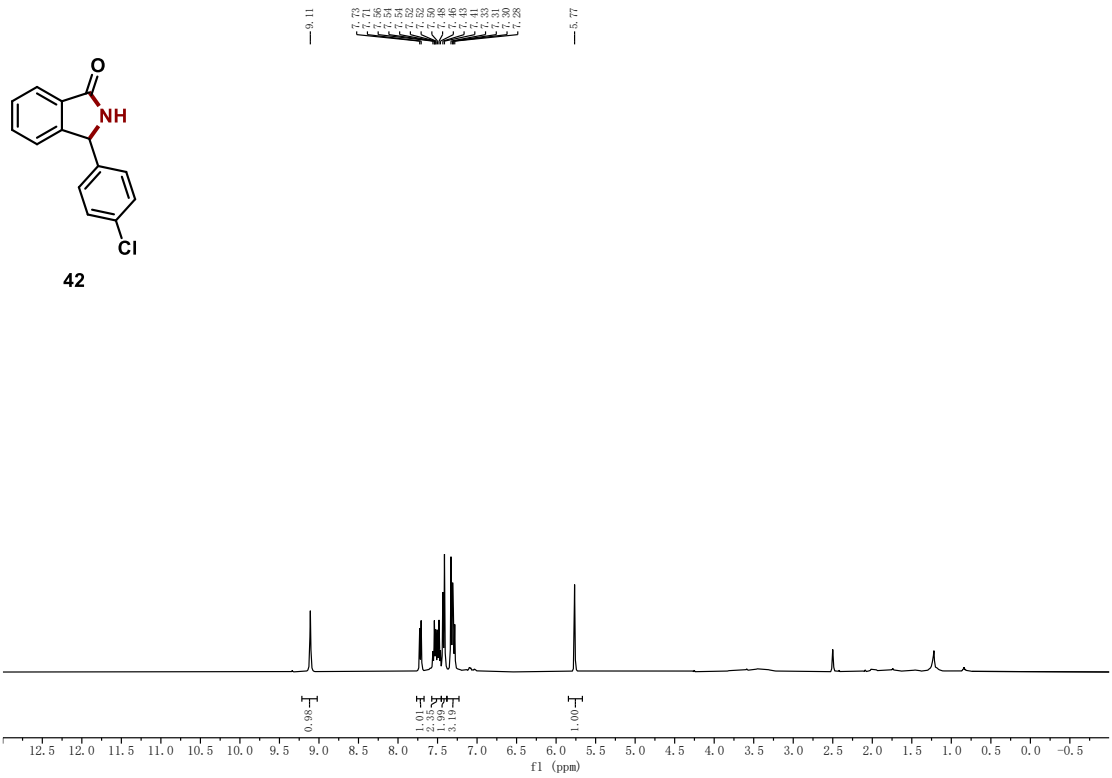

**Supplementary Figure 242** $^{1}$H NMR spectrum of compound **41**



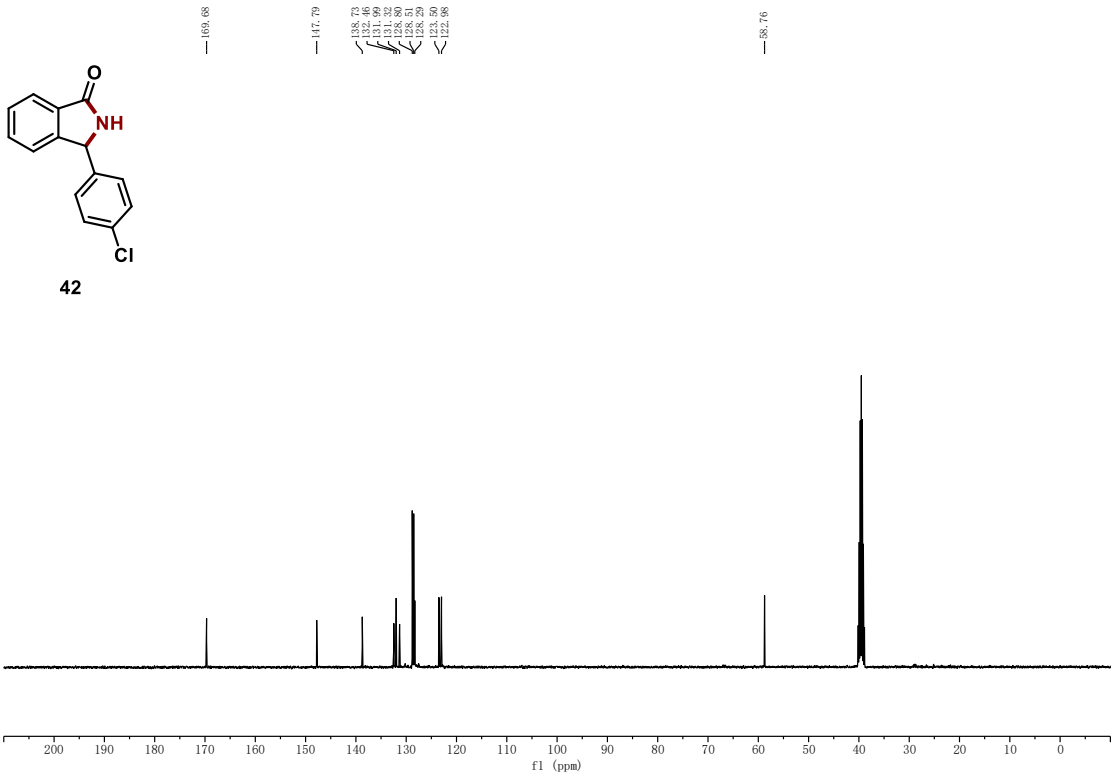

**Supplementary Figure 243** $^{13}$C NMR spectrum of compound **41**



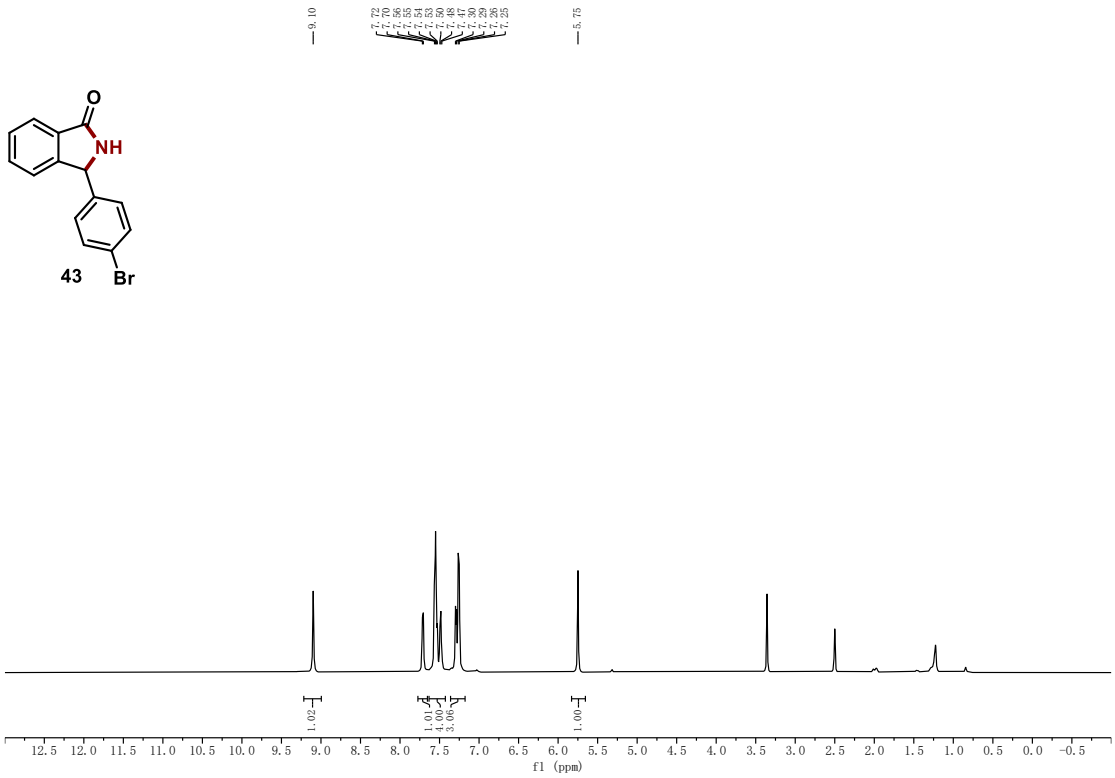

**Supplementary Figure 244** $^{19}$F NMR spectrum of compound **41**



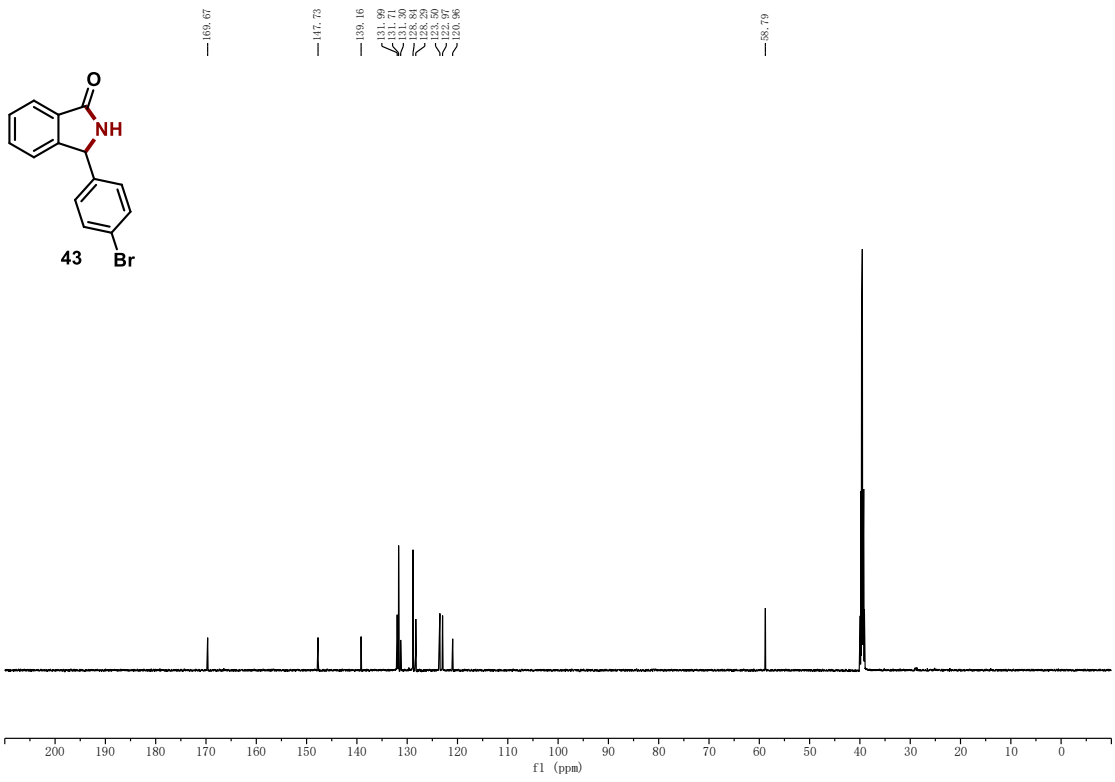

**Supplementary Figure 245** $^{1}$H NMR spectrum of compound **42**



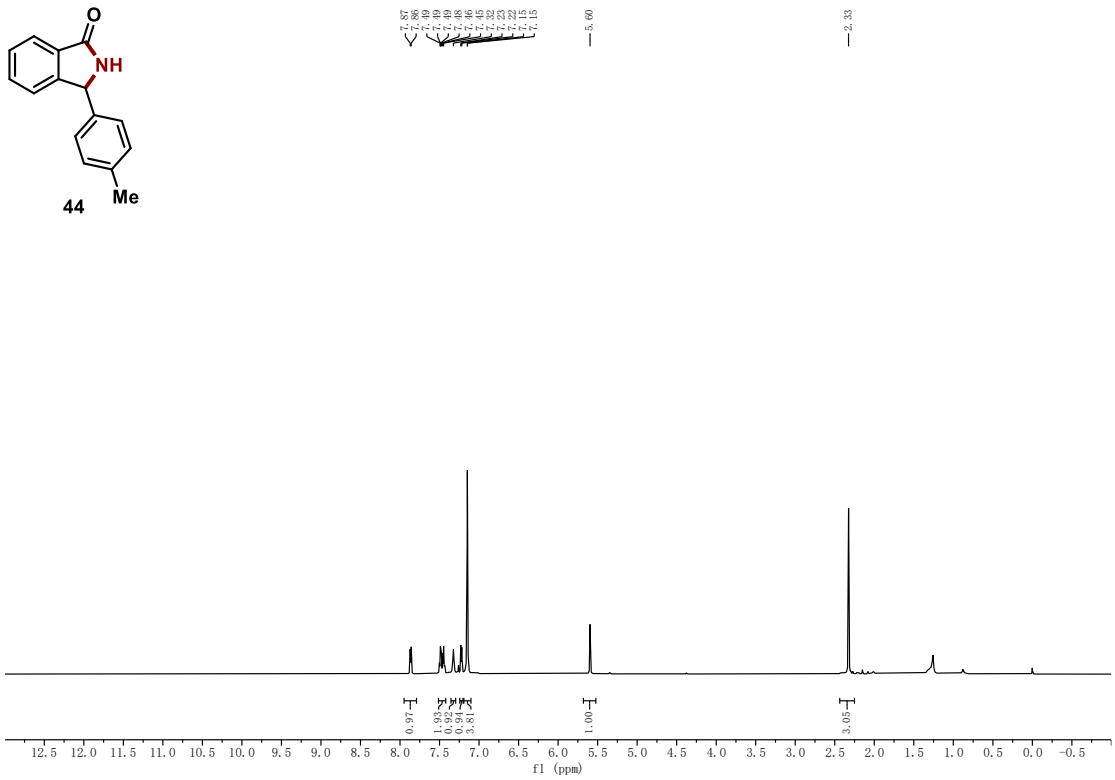

**Supplementary Figure 246** $^{13}$C NMR spectrum of compound **42**



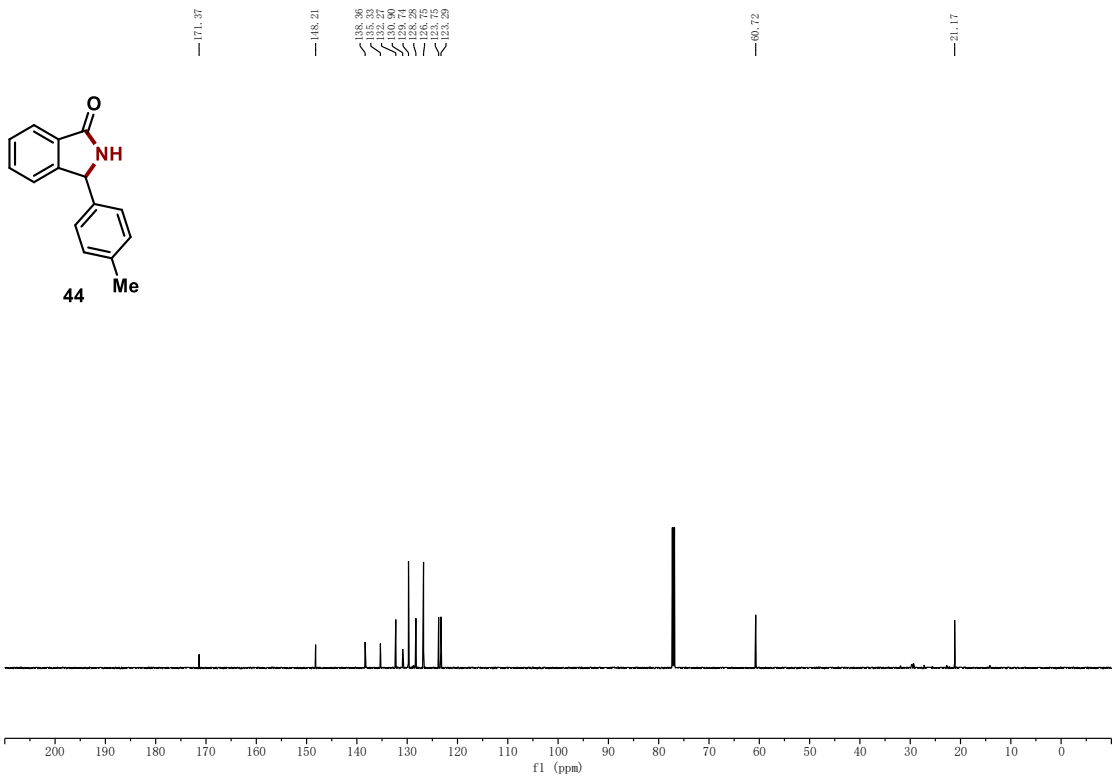

**Supplementary Figure 247** $^{1}$H NMR spectrum of compound **43**



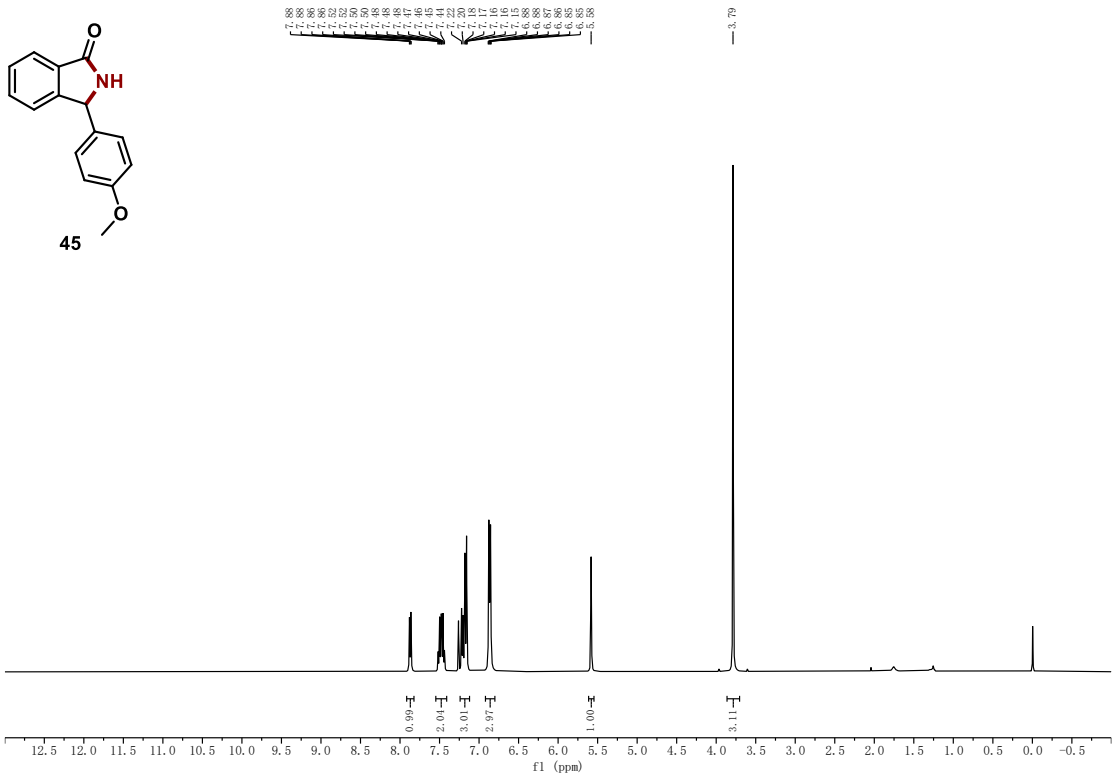

**Supplementary Figure 248** $^{13}$C NMR spectrum of compound **43**



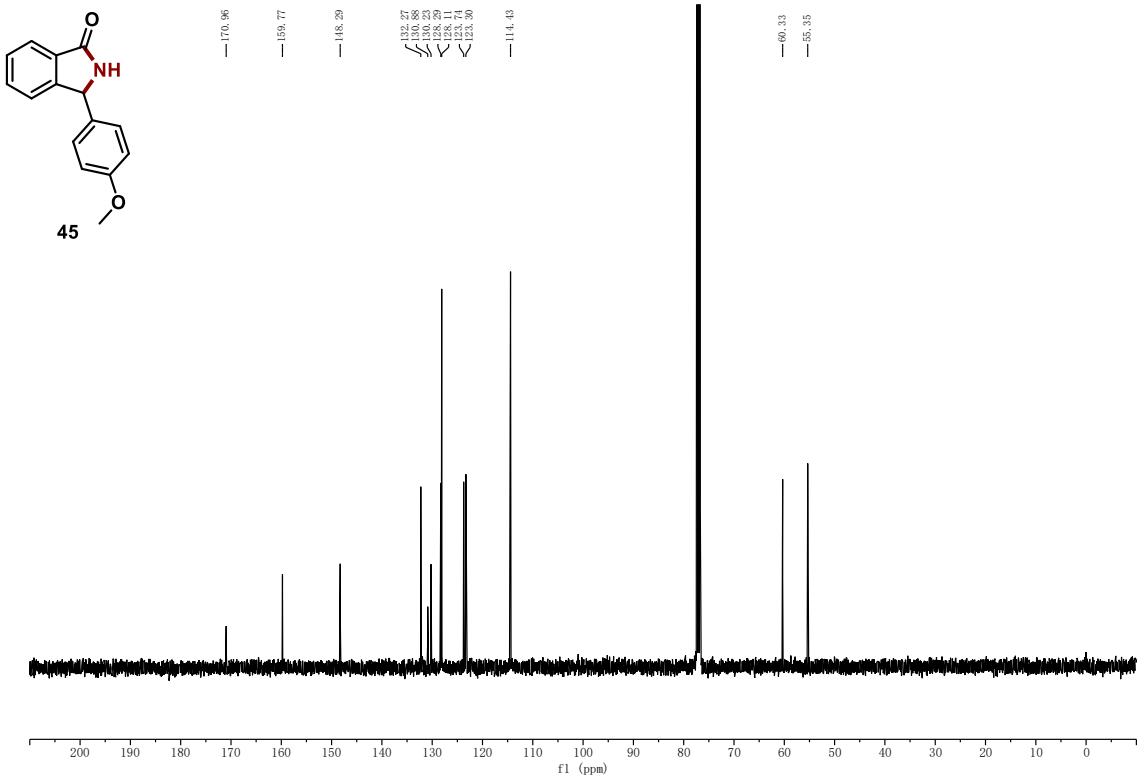

**Supplementary Figure 249** $^{1}$H NMR spectrum of compound **44**



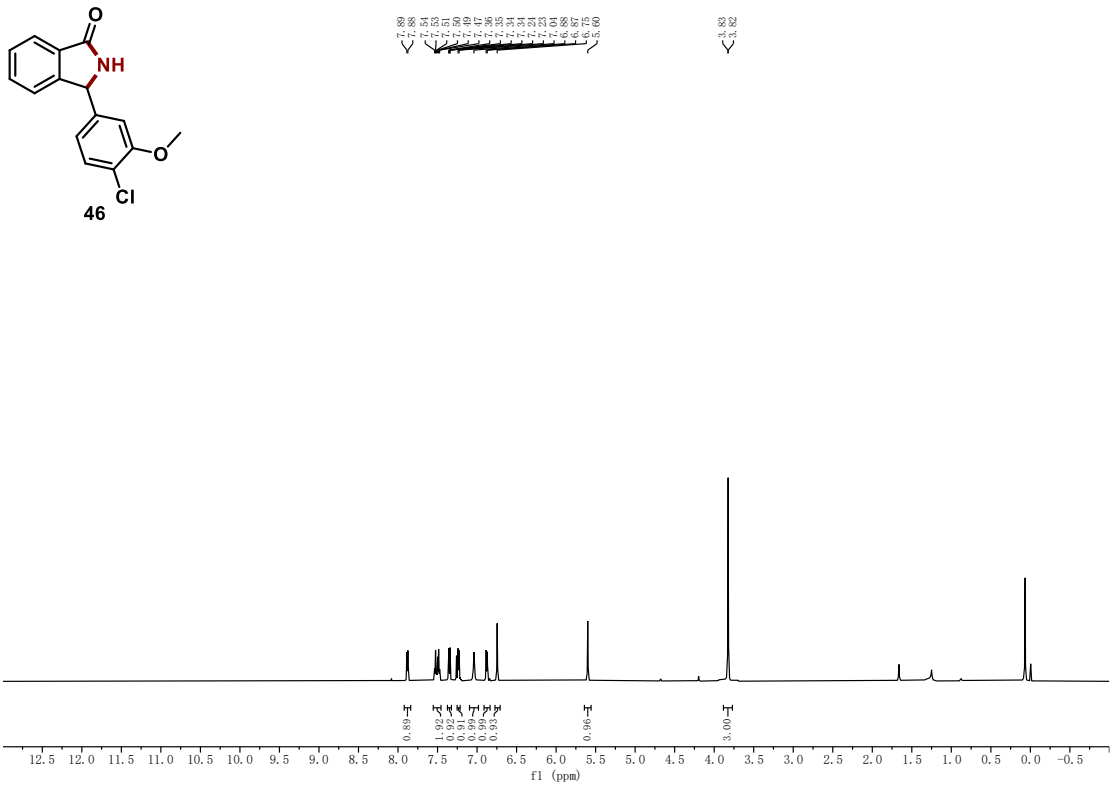

**Supplementary Figure 250** $^{13}$C NMR spectrum of compound **44**



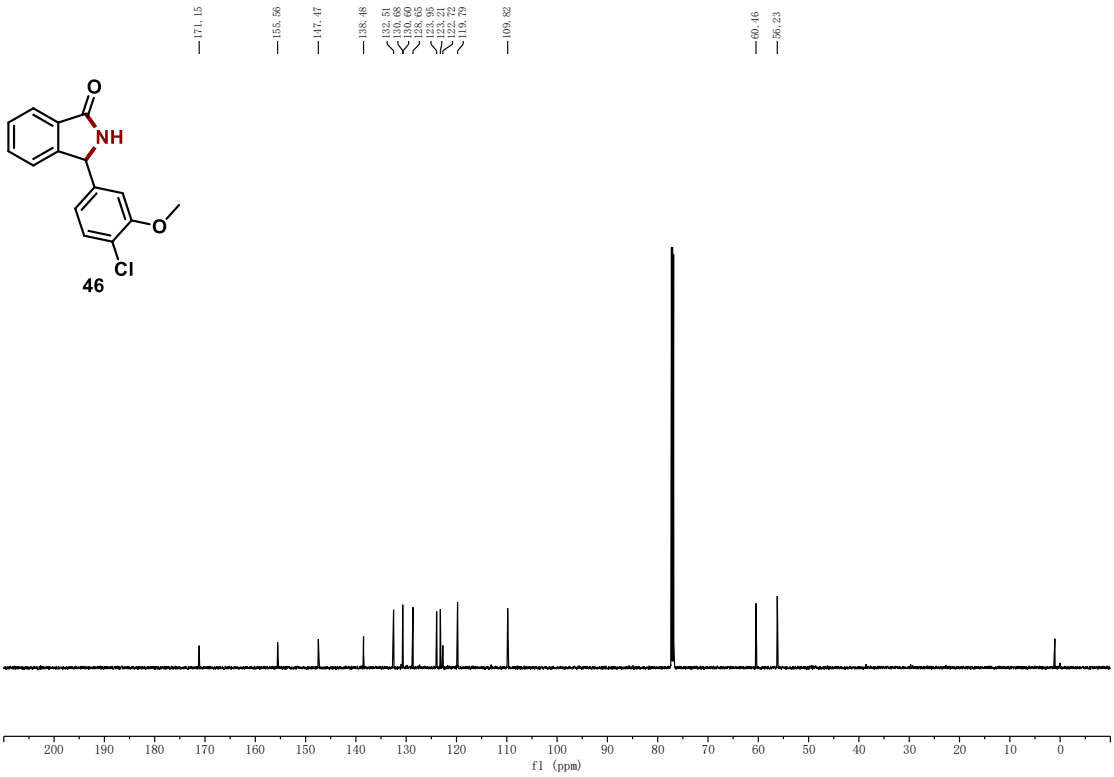

**Supplementary Figure 251** $^{1}$H NMR spectrum of compound **45**



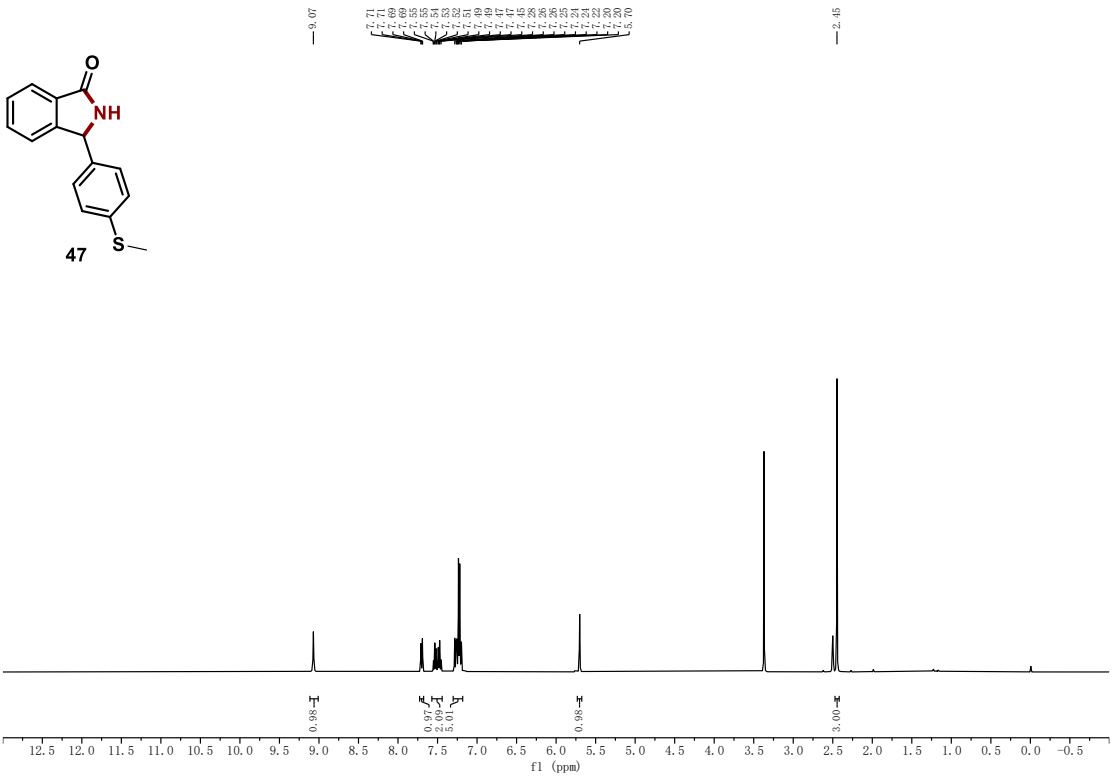

**Supplementary Figure 252** $^{13}$C NMR spectrum of compound **45**



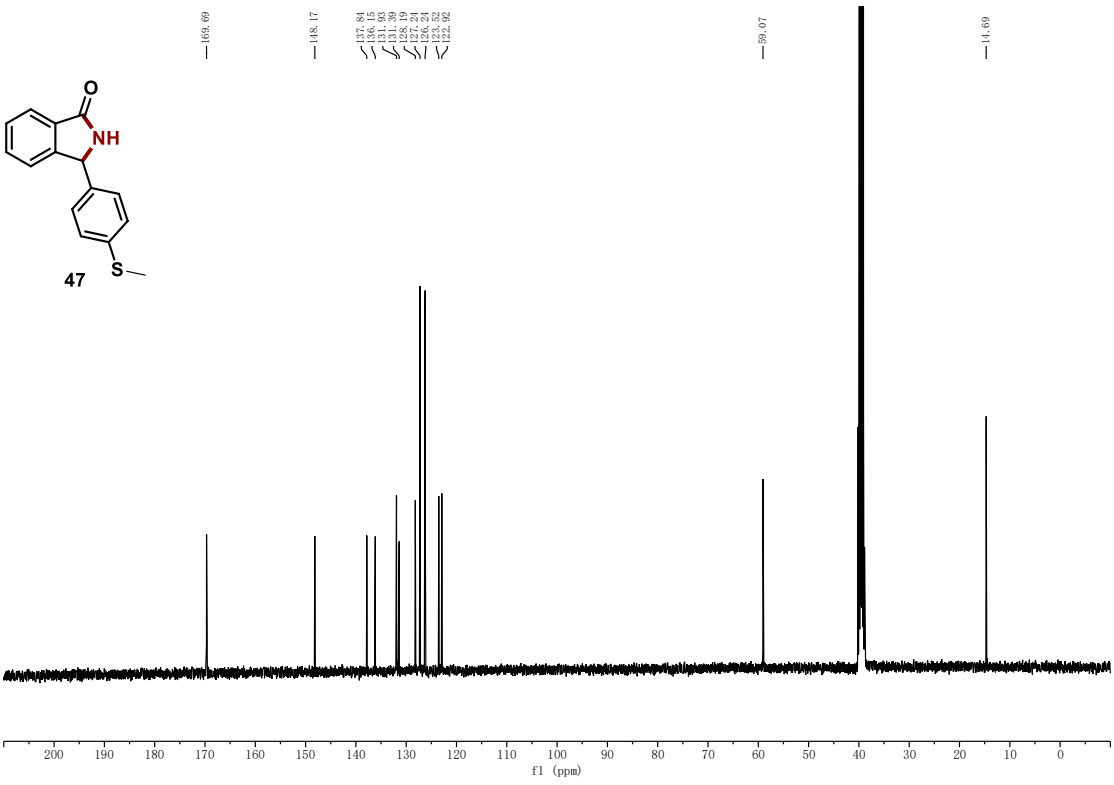

**Supplementary Figure 253** $^{1}$H NMR spectrum of compound **46**



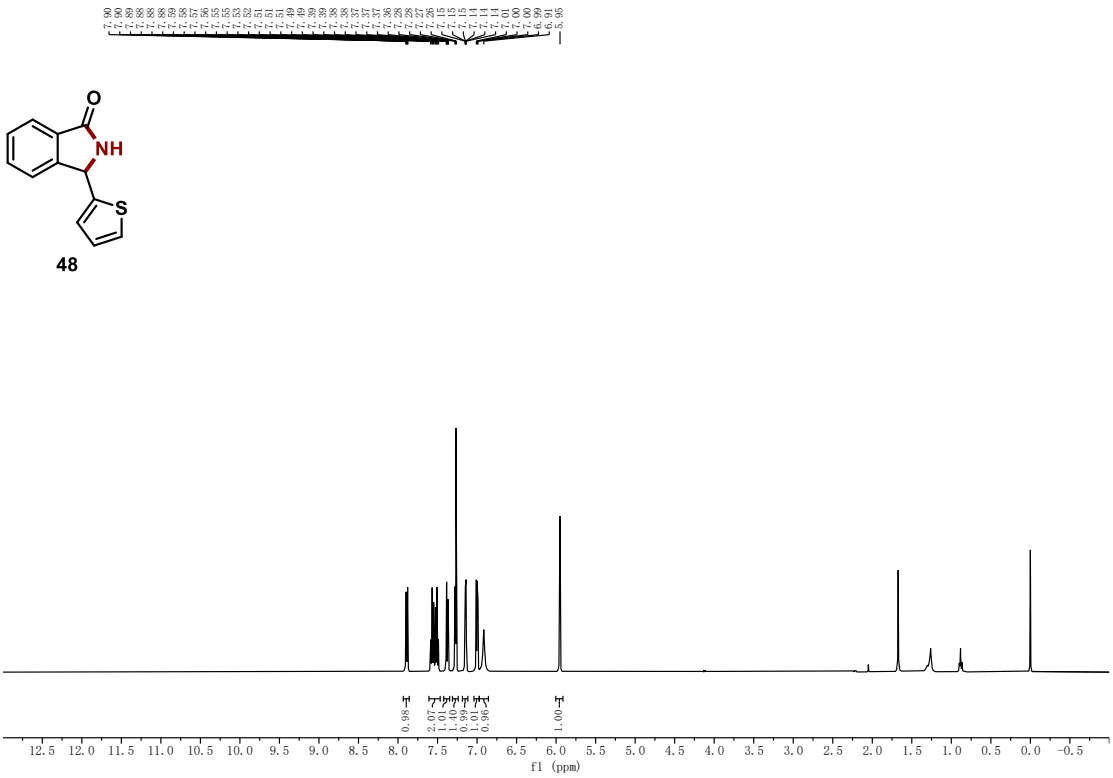

**Supplementary Figure 254** $^{13}$C NMR spectrum of compound **46**



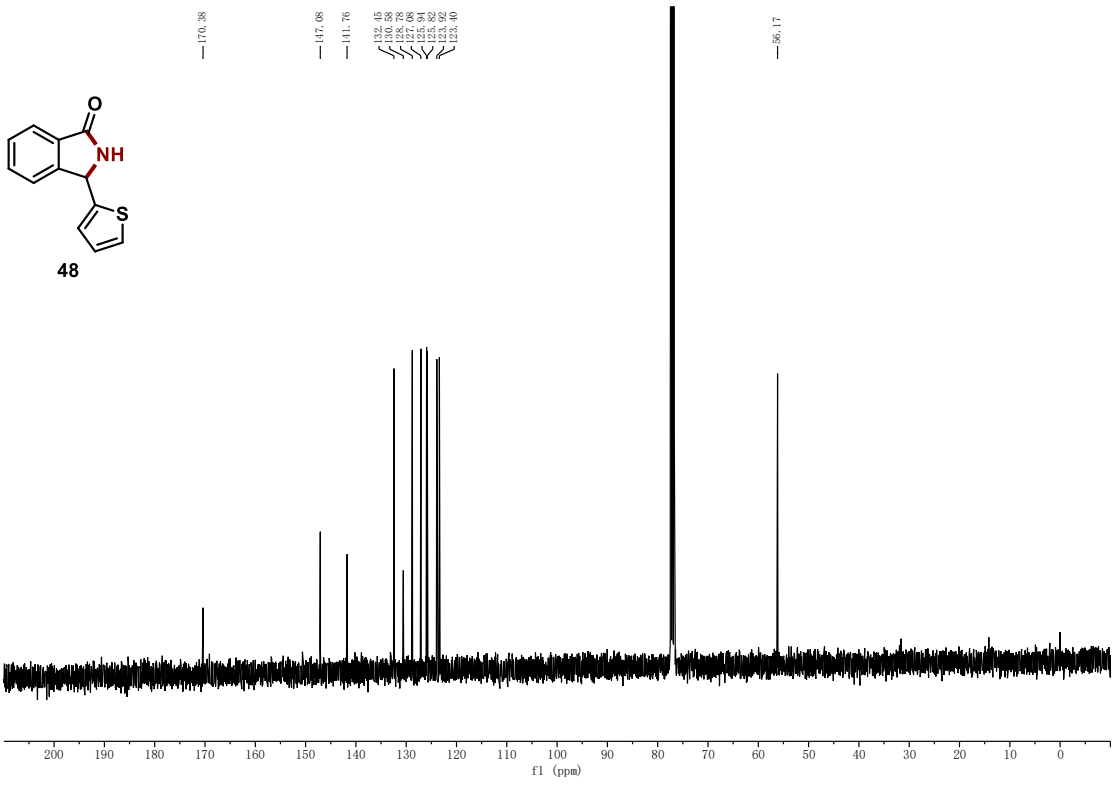

**Supplementary Figure 255** $^{1}$H NMR spectrum of compound **47**



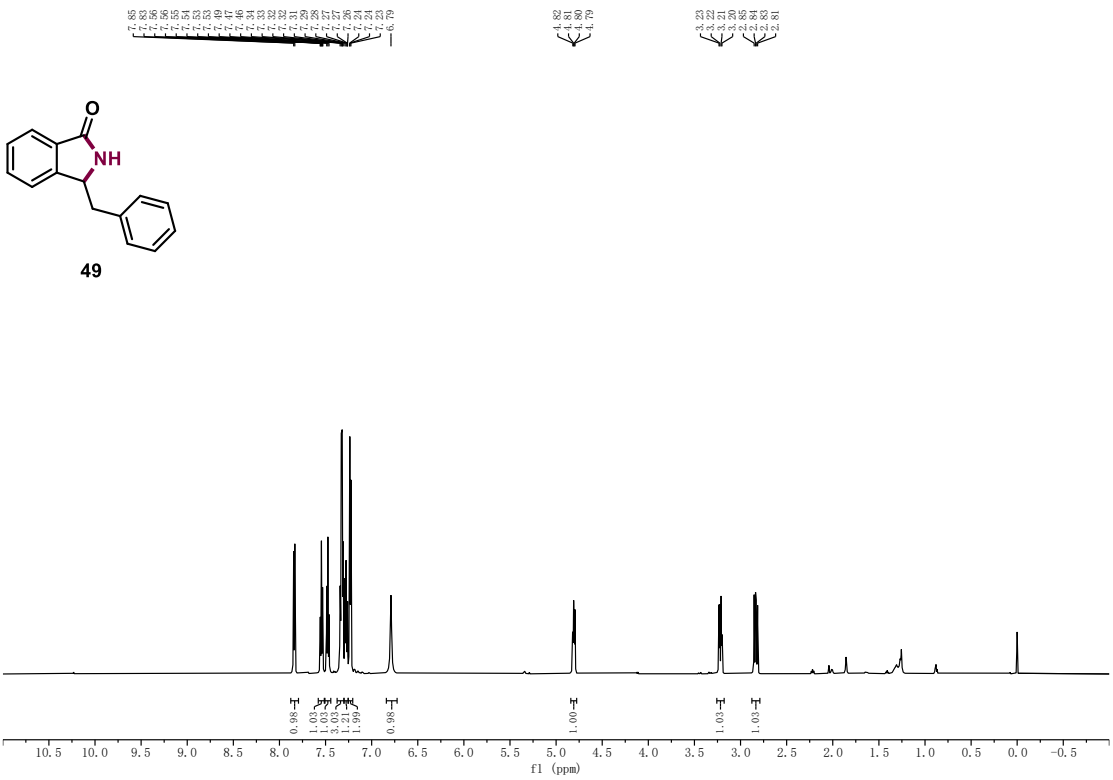

**Supplementary Figure 256** $^{13}$C NMR spectrum of compound **47**



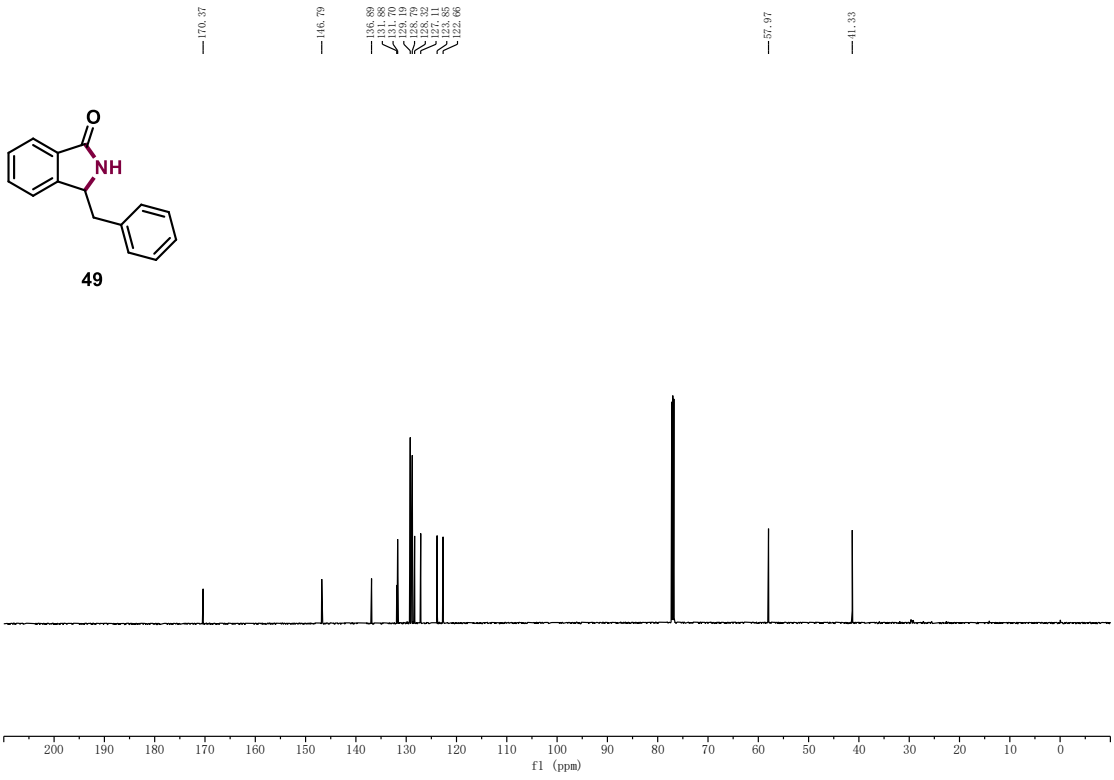

**Supplementary Figure 257** $^{1}$H NMR spectrum of compound **48**



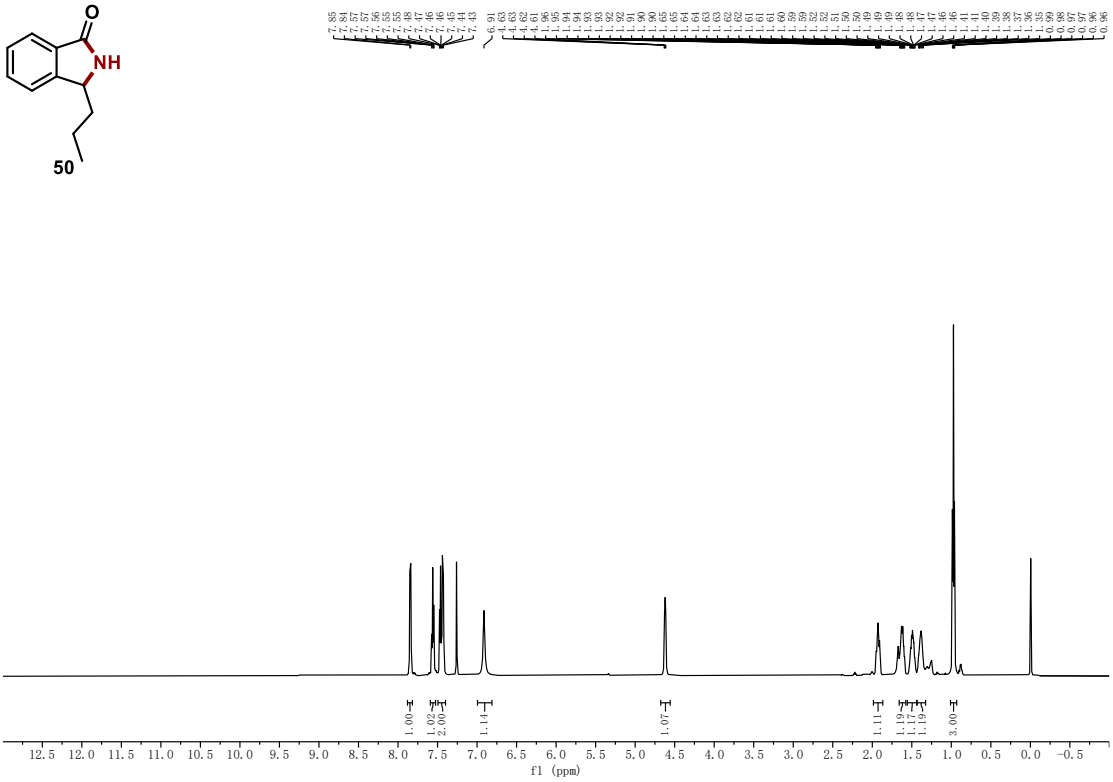

**Supplementary Figure 258** $^{13}$C NMR spectrum of compound **48**



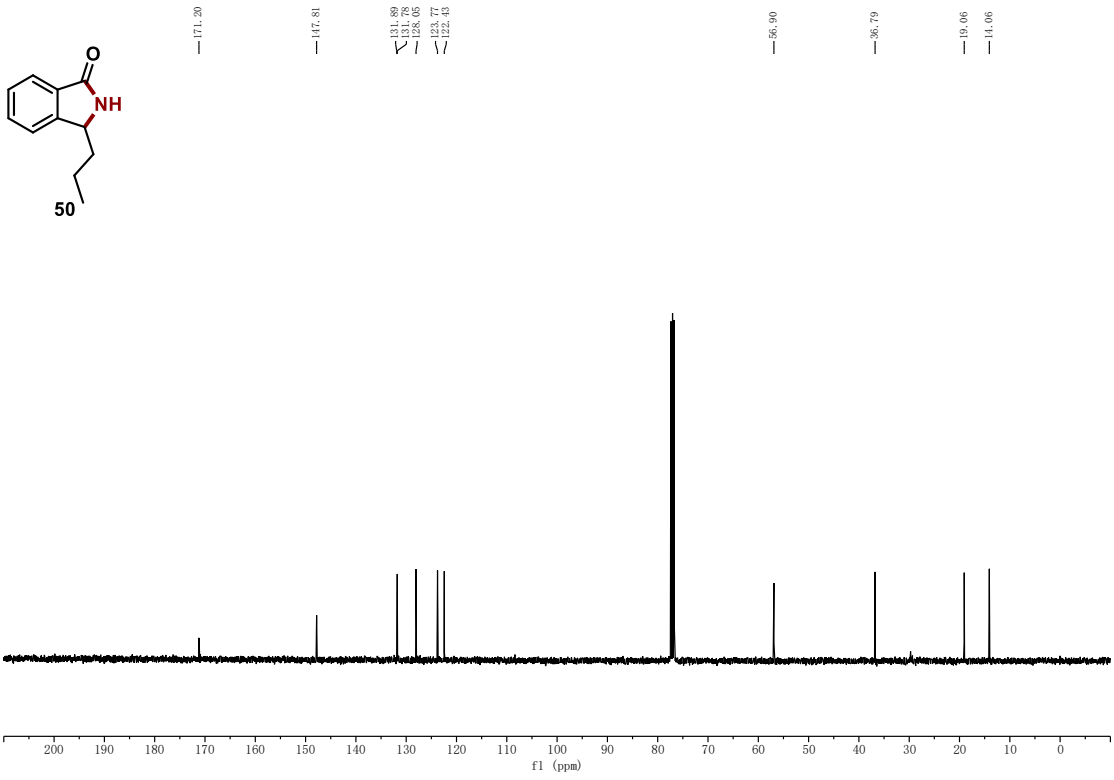

**Supplementary Figure 259** $^{1}$H NMR spectrum of compound **49**



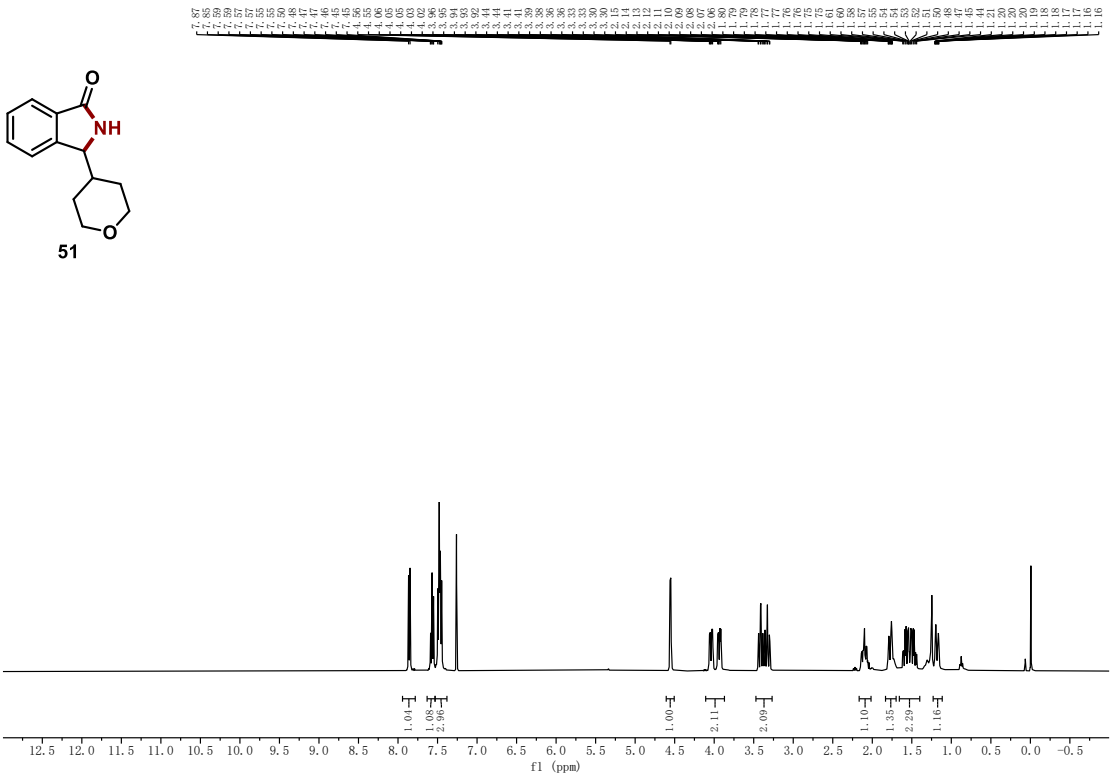

**Supplementary Figure 260** $^{13}$C NMR spectrum of compound **49**



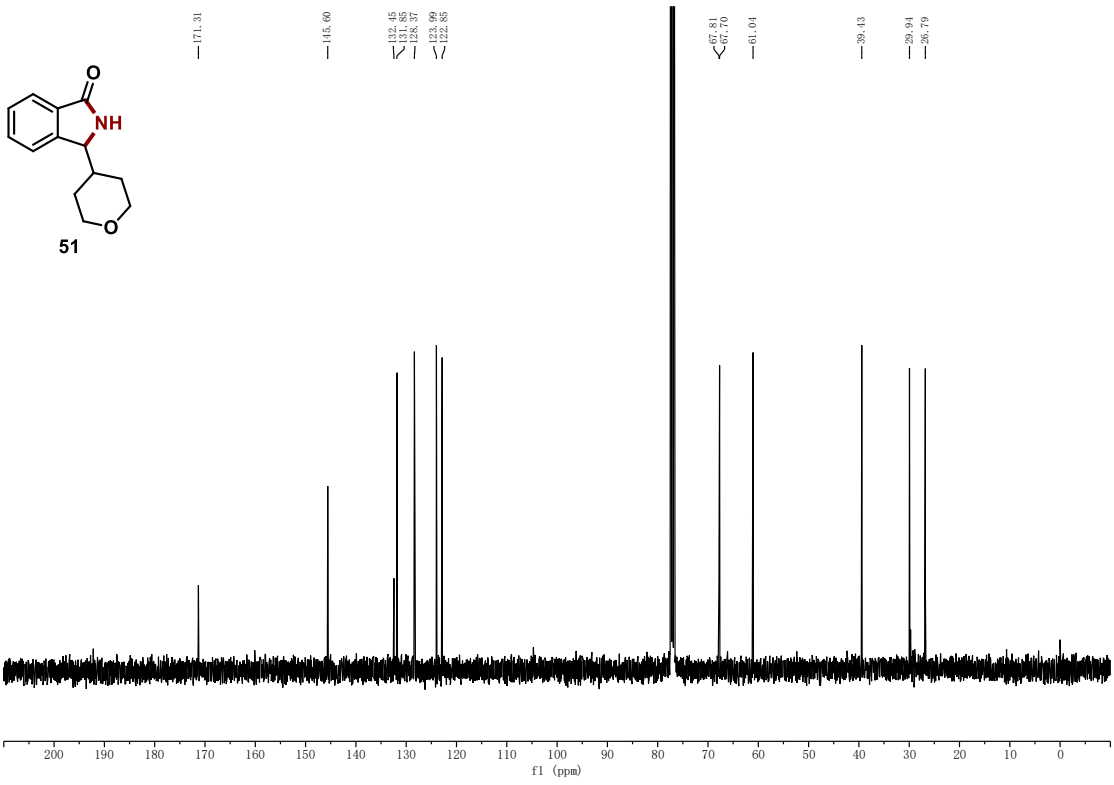

**Supplementary Figure 261** $^{1}$H NMR spectrum of compound **50**



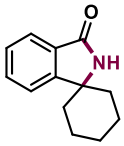

**Supplementary Figure 262** $^{13}$C NMR spectrum of compound **50**




**Supplementary Figure 263** $^{1}$H NMR spectrum of compound **51**



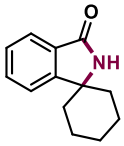

**Supplementary Figure 264** $^{13}$C NMR spectrum of compound **51**




52



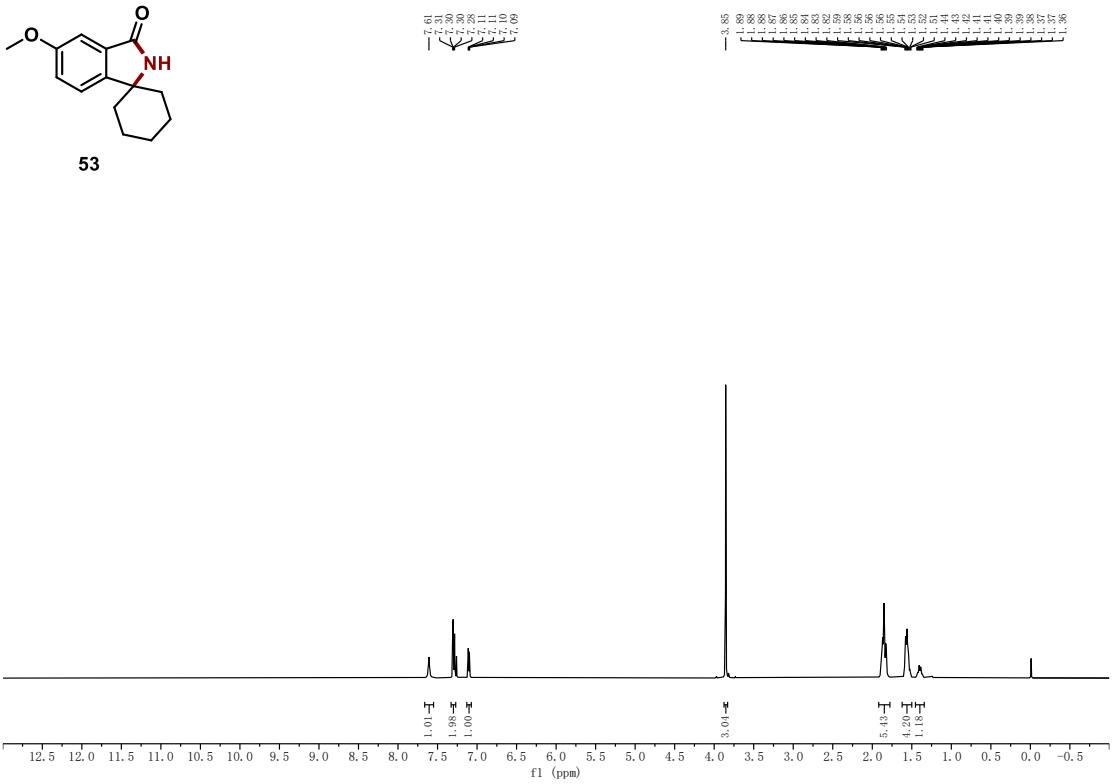

**Supplementary Figure 265** $^{1}$H NMR spectrum of compound **52**



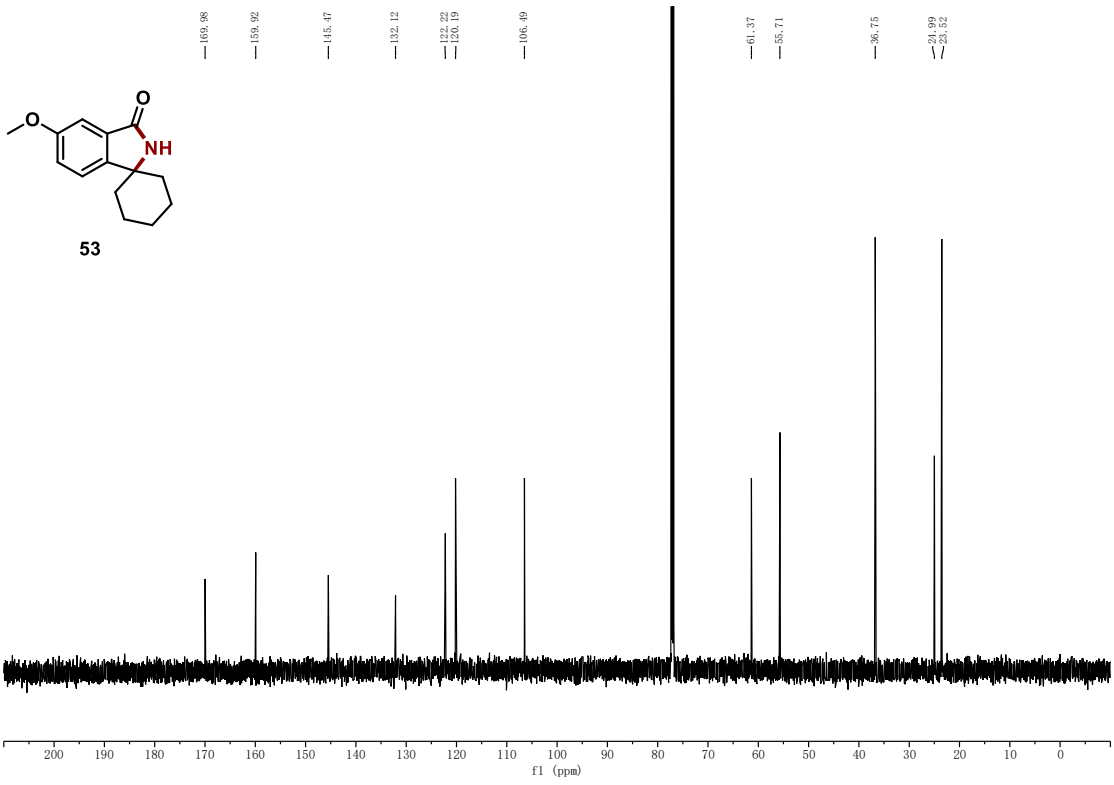

52



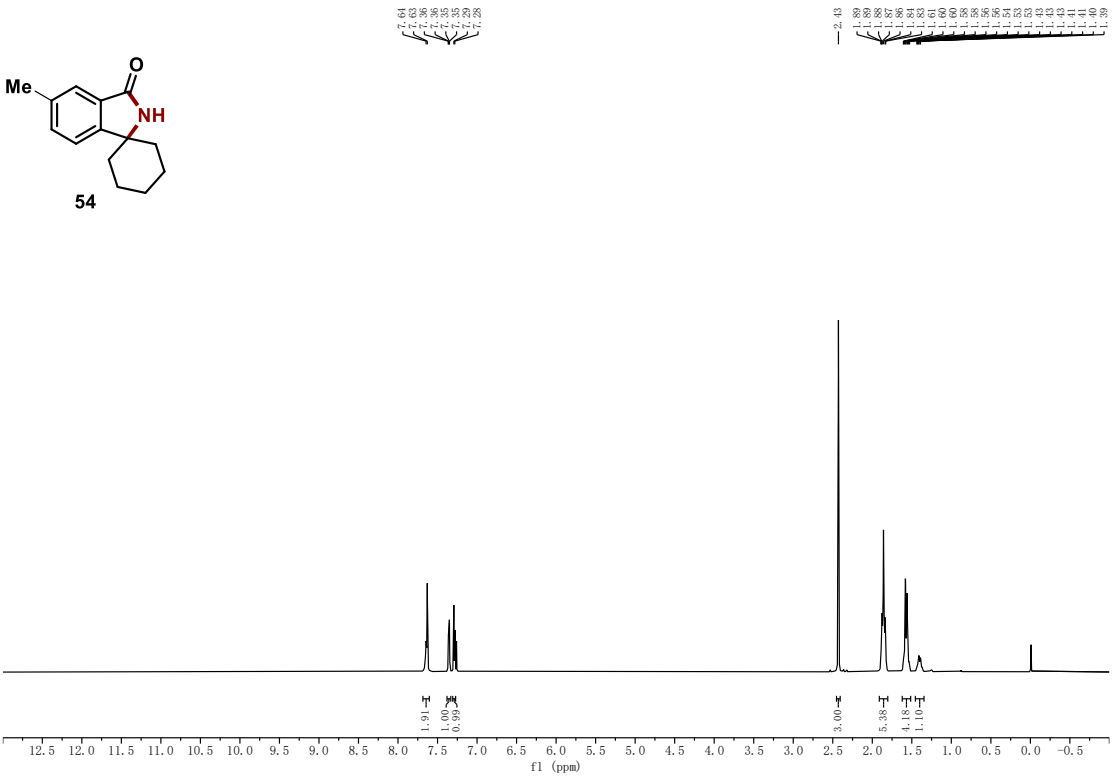

**Supplementary Figure 266** $^{13}$C NMR spectrum of compound **52**



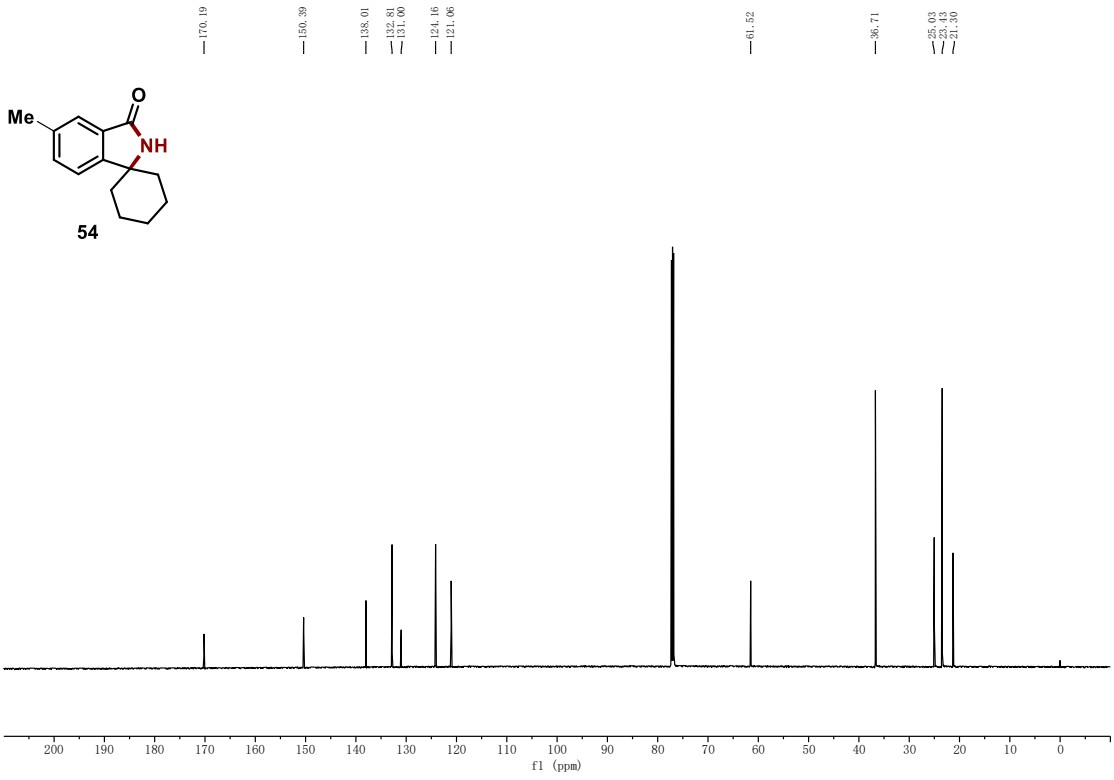

**Supplementary Figure 267** $^{1}$H NMR spectrum of compound **53**



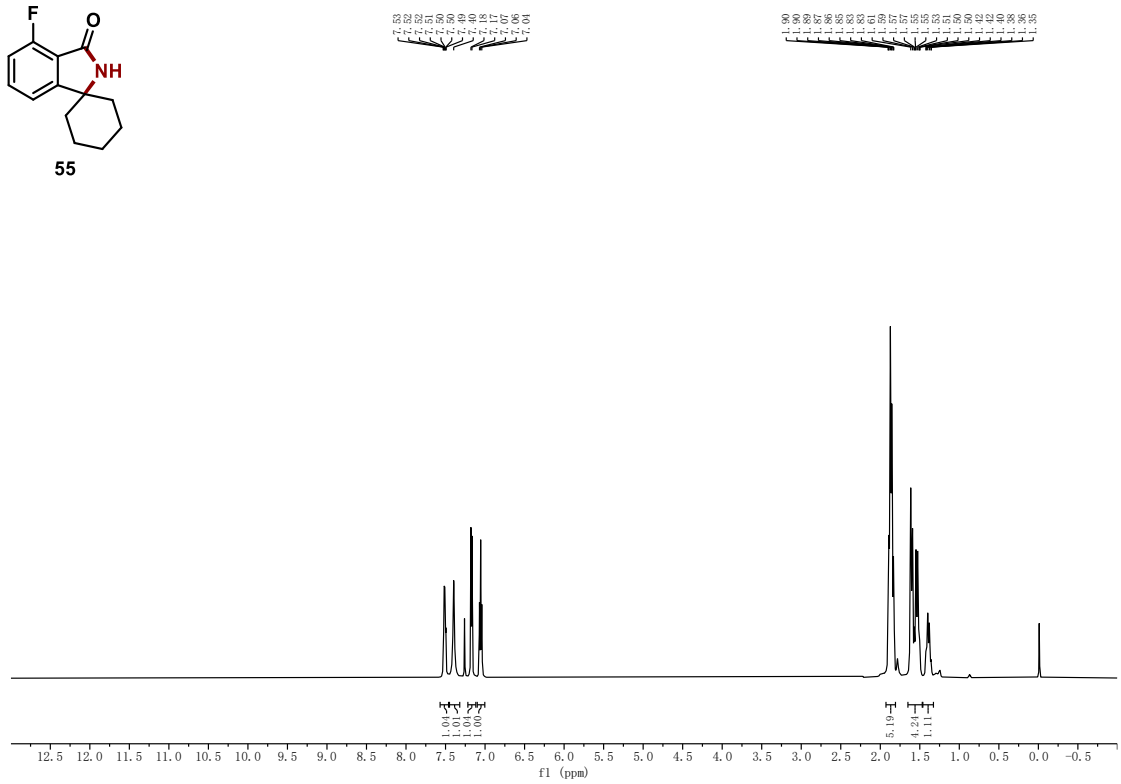

**Supplementary Figure 268** $^{13}$C NMR spectrum of compound **53**



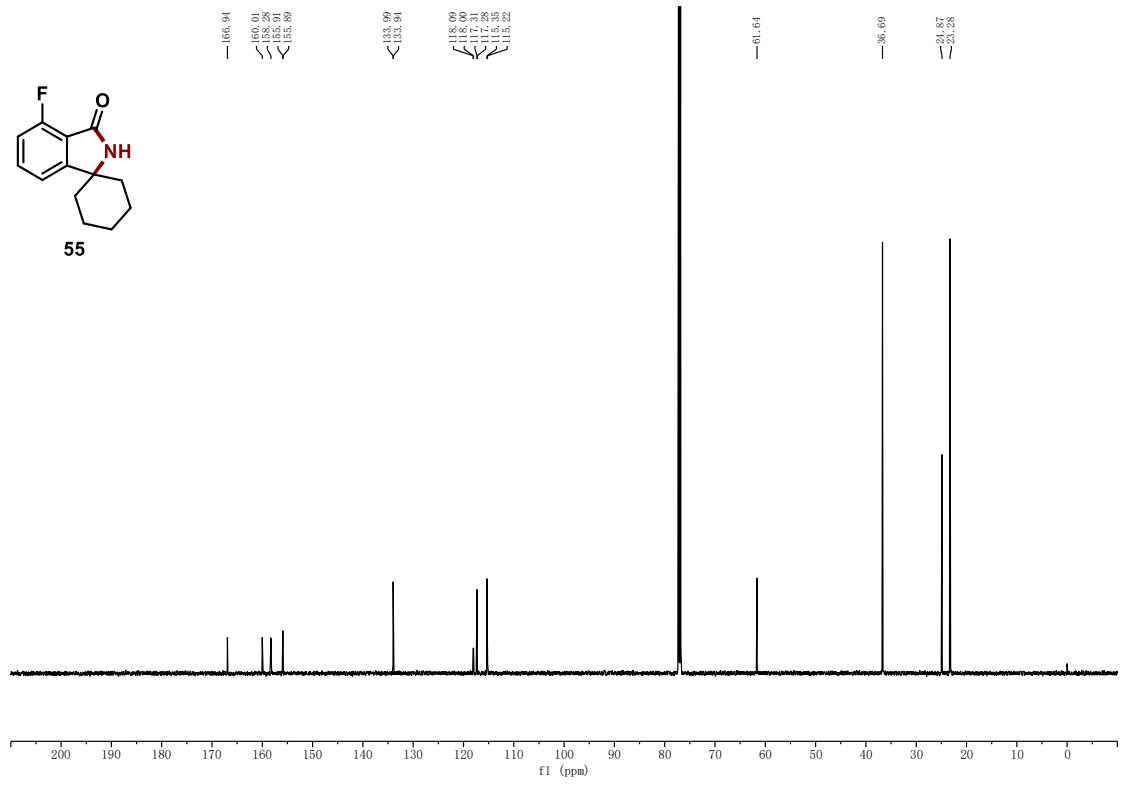

**Supplementary Figure 269** $^{1}$H NMR spectrum of compound **54**



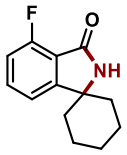

**Supplementary Figure 270** $^{13}$C NMR spectrum of compound **54**




**Supplementary Figure 271** $^{1}$H NMR spectrum of compound **55**



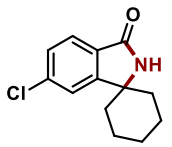

**Supplementary Figure 272** $^{13}$C NMR spectrum of compound **55**




55



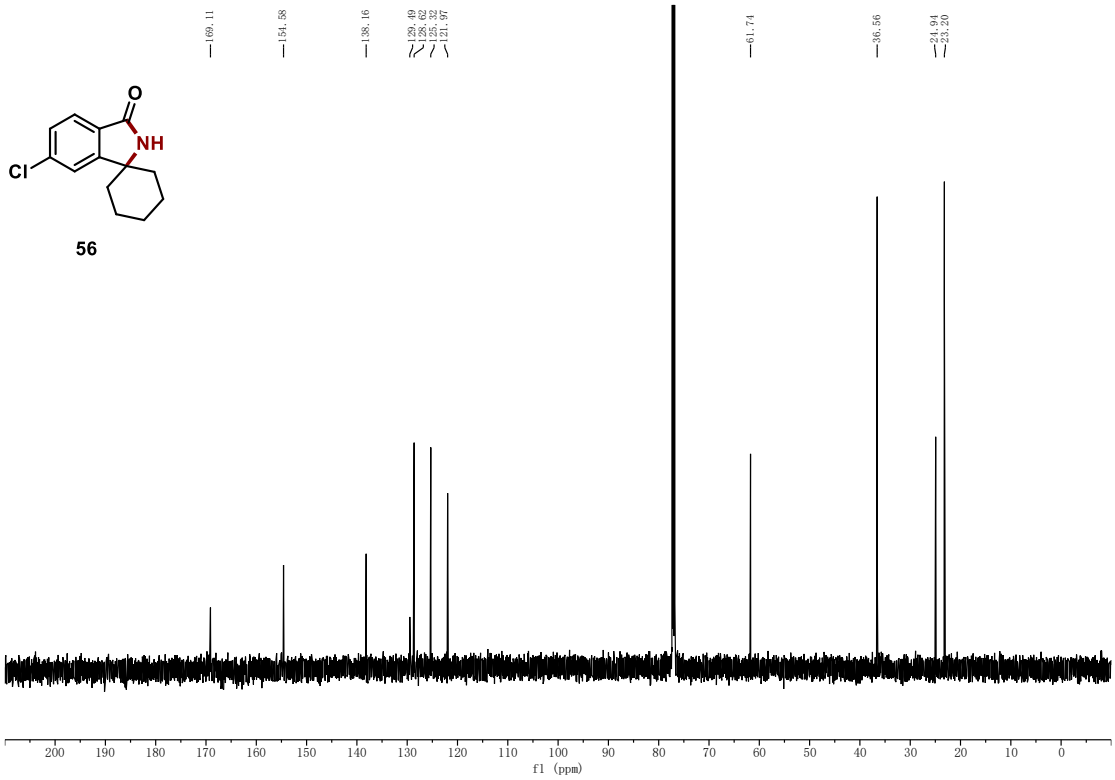

**Supplementary Figure 273** $^{19}$F NMR spectrum of compound **55**



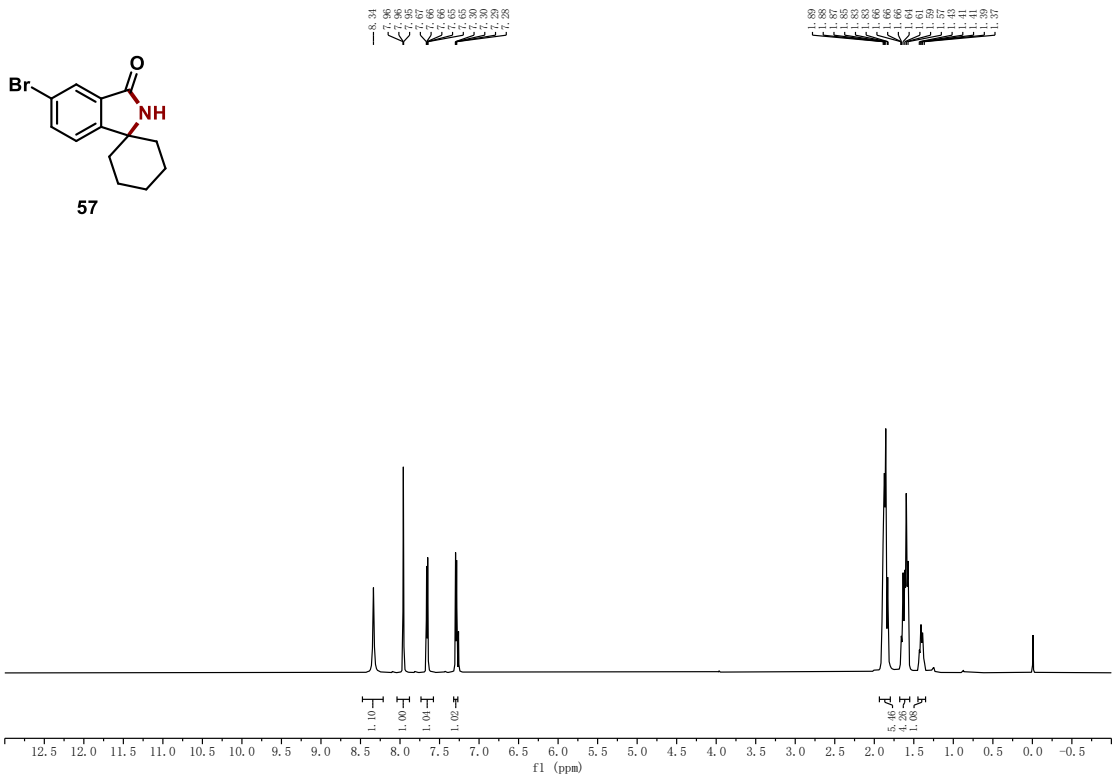

56



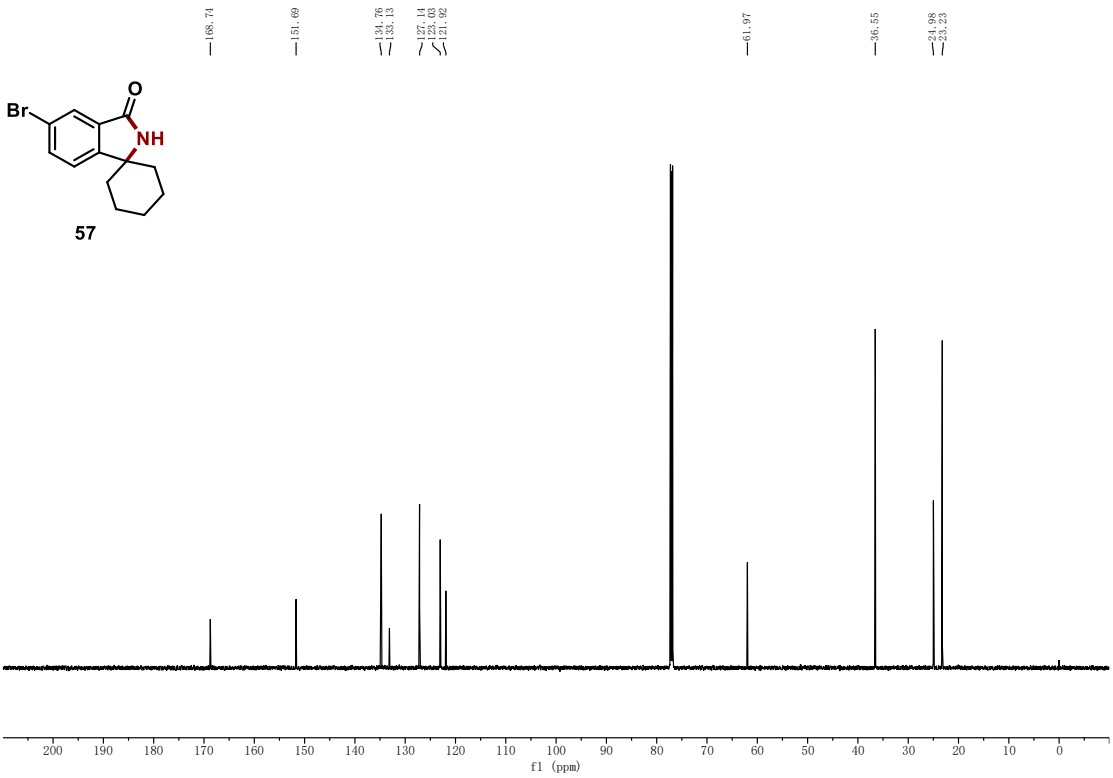

**Supplementary Figure 274** $^{1}$H NMR spectrum of compound **56**



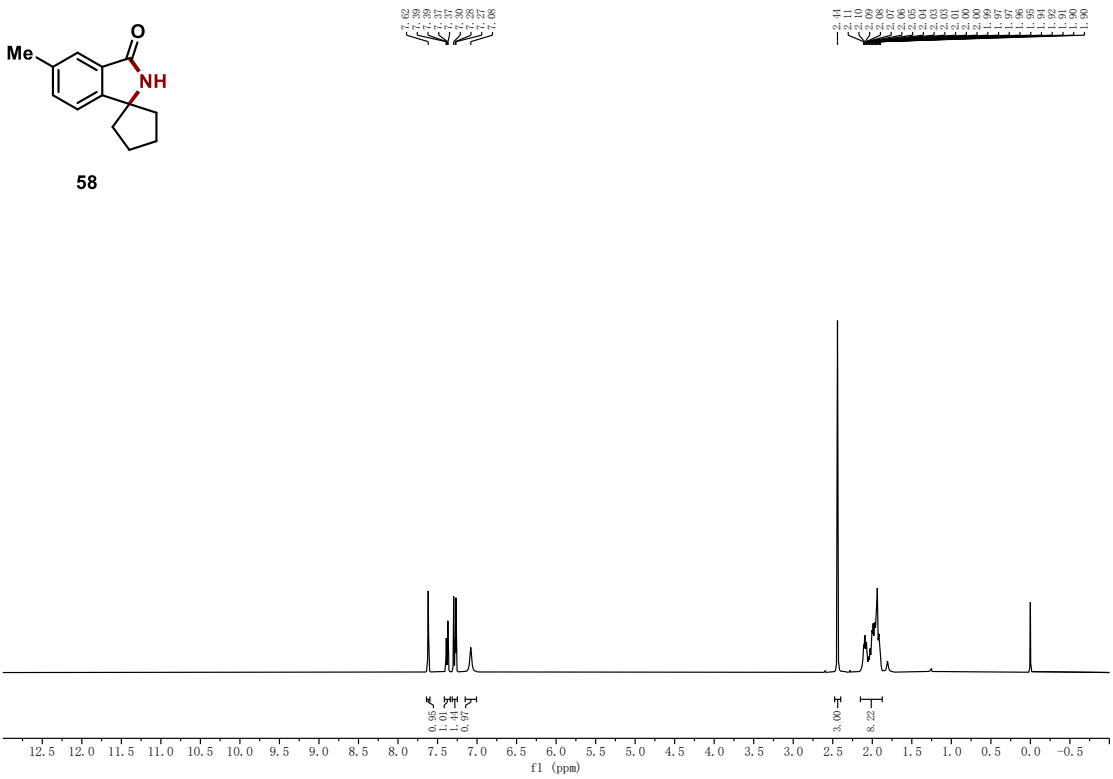

**Supplementary Figure 275** $^{13}$C NMR spectrum of compound **56**



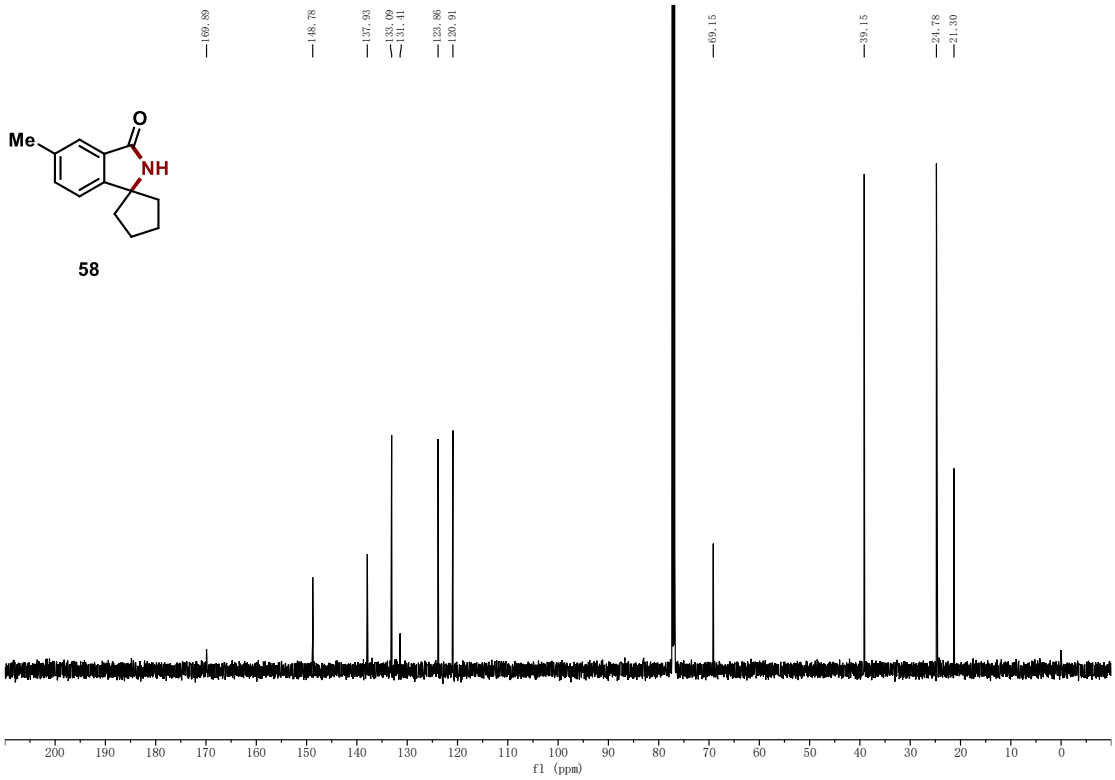

**Supplementary Figure 276** $^{1}$H NMR spectrum of compound **57**



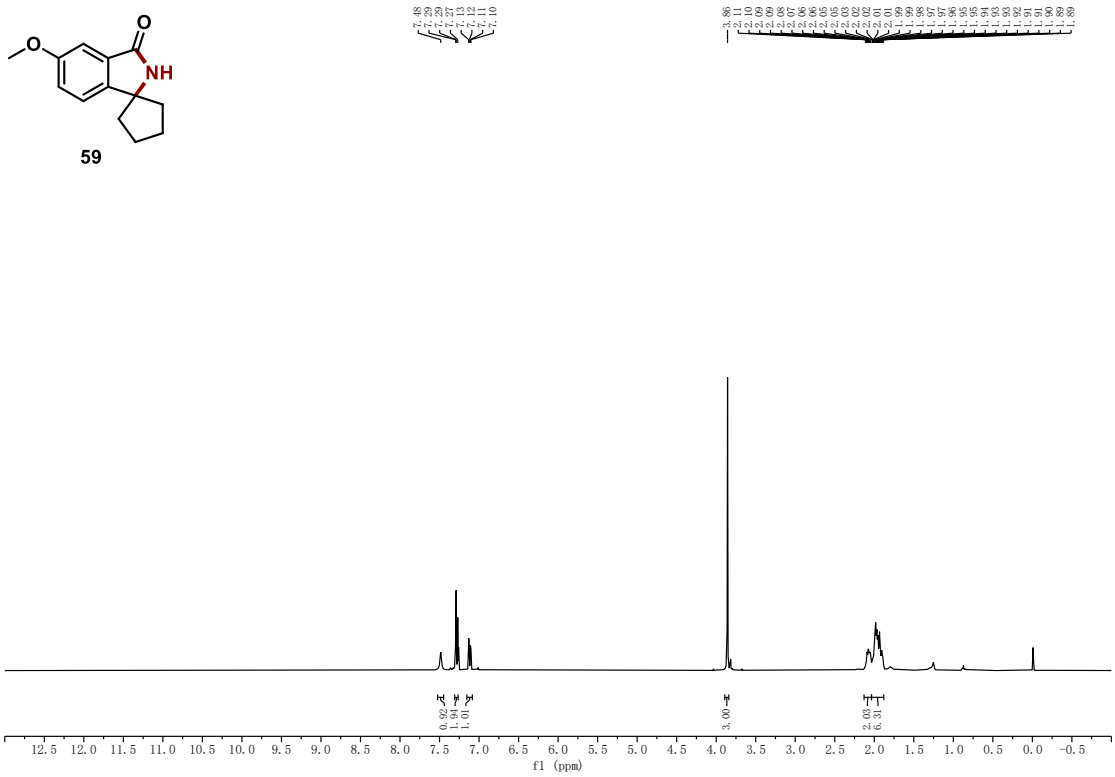

**Supplementary Figure 277** $^{13}$C NMR spectrum of compound **57**



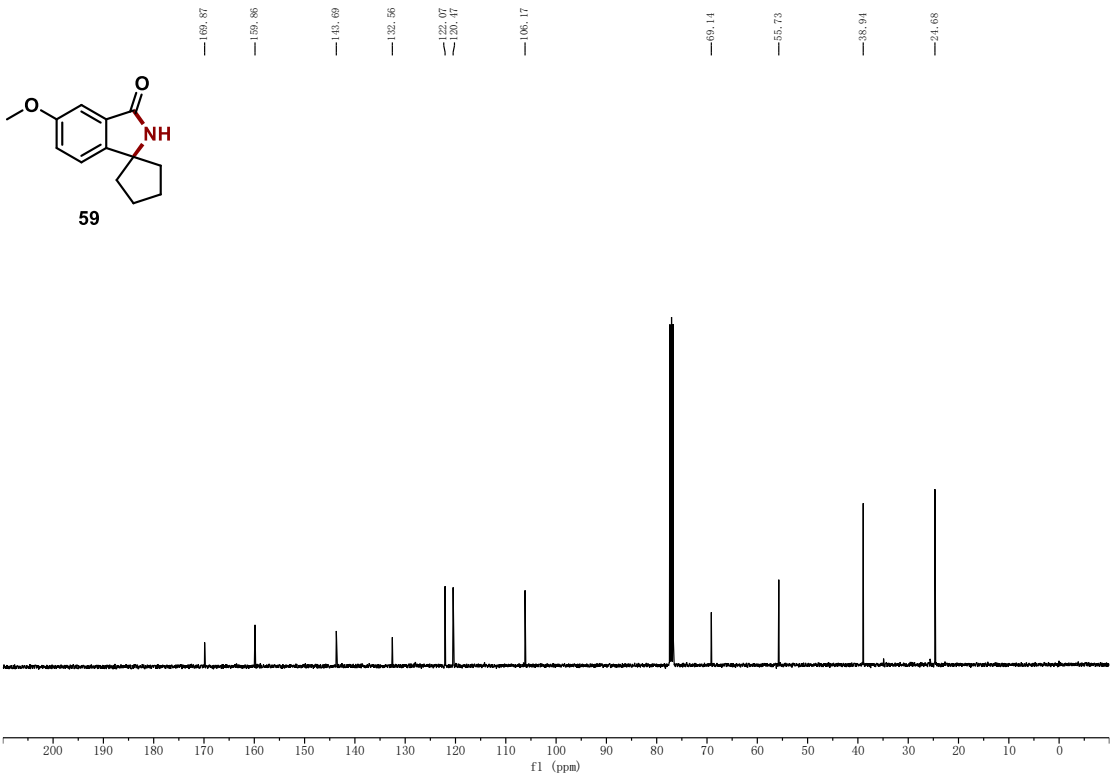

**Supplementary Figure 278** $^{1}$H NMR spectrum of compound **58**



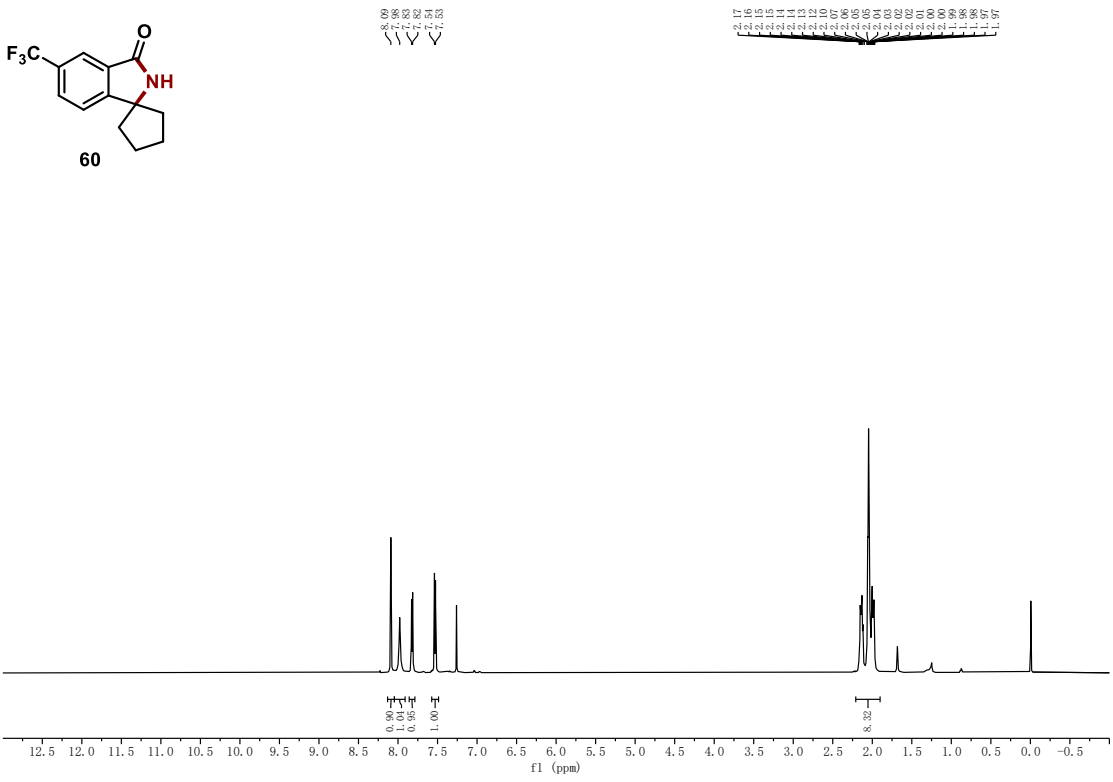

**Supplementary Figure 279** $^{13}$C NMR spectrum of compound **58**



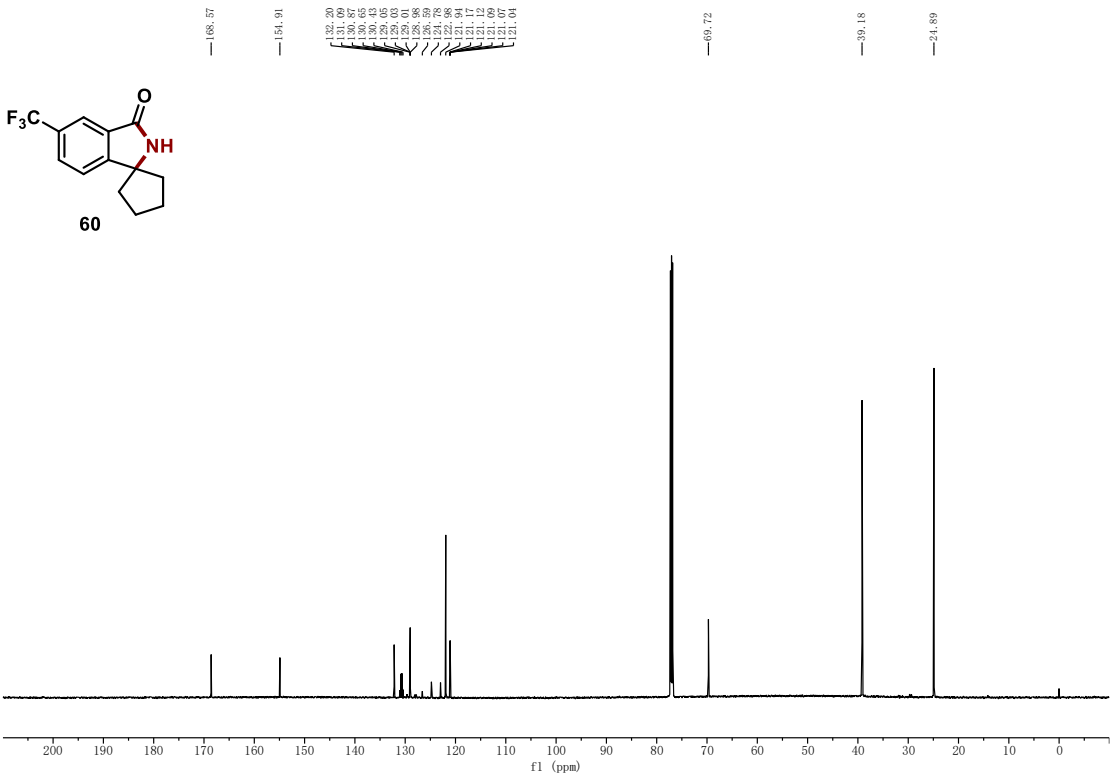

**Supplementary Figure 280** $^{1}$H NMR spectrum of compound **59**



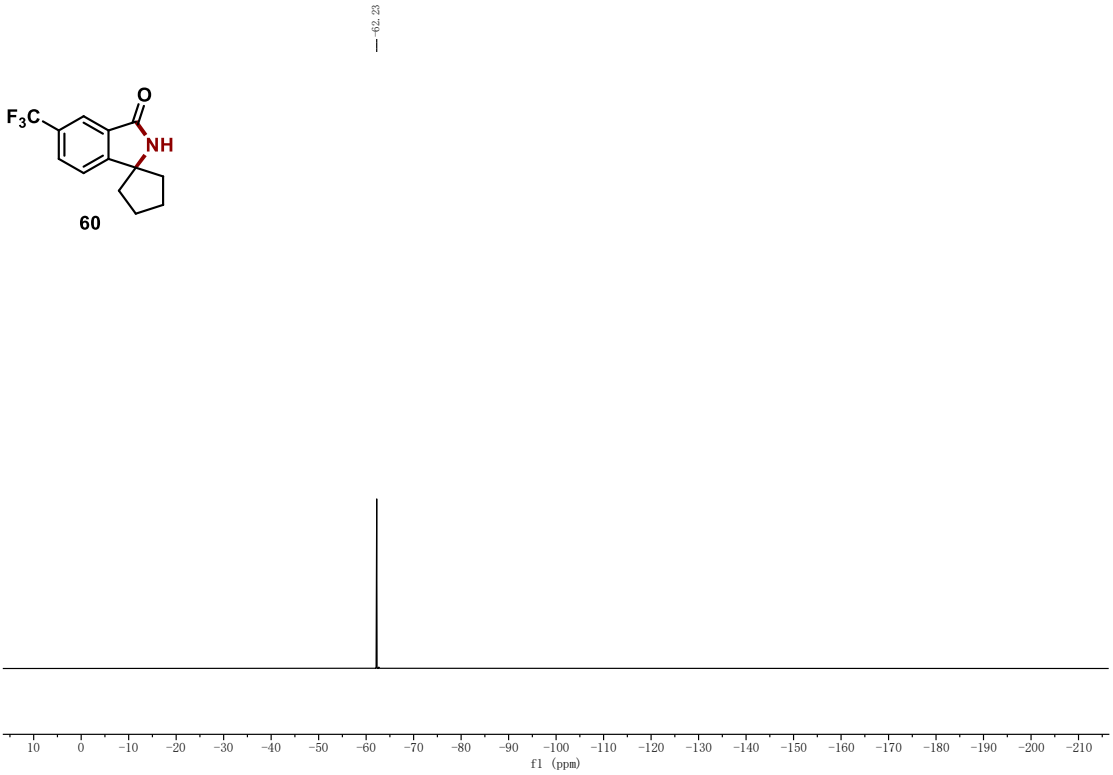

**Supplementary Figure 281** $^{13}$C NMR spectrum of compound **59**



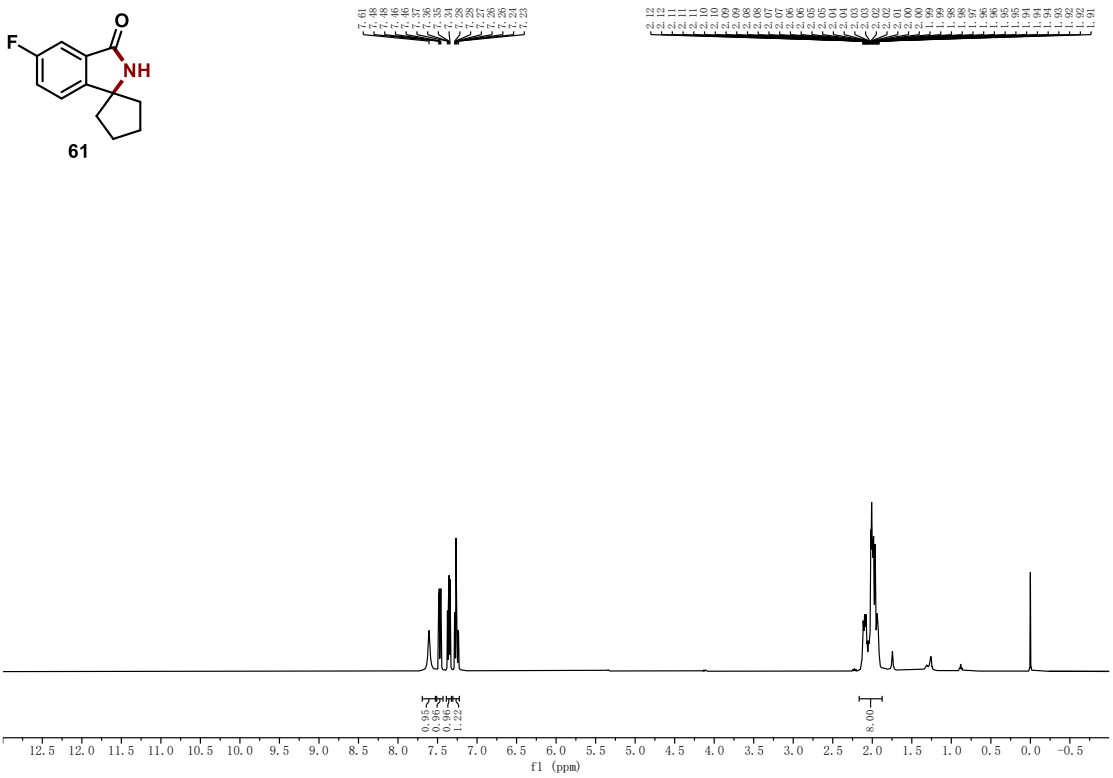

**Supplementary Figure 282** $^{1}$H NMR spectrum of compound **60**



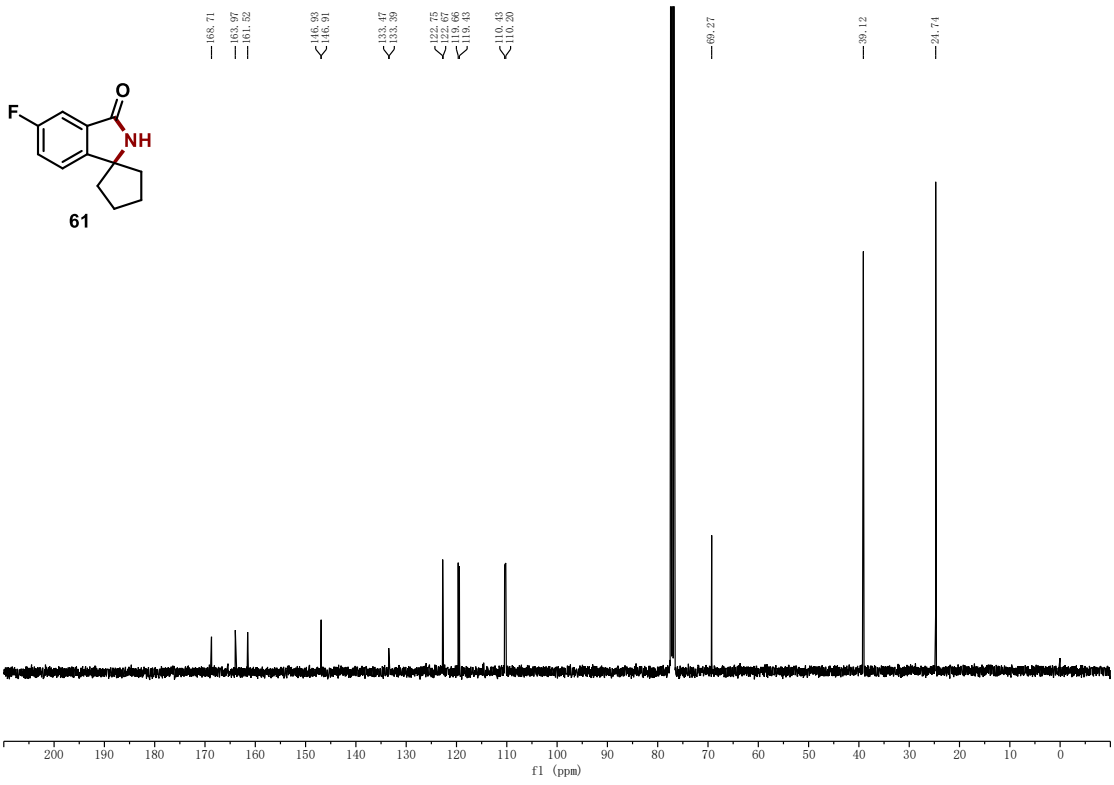

**Supplementary Figure 283** $^{13}$C NMR spectrum of compound **60**



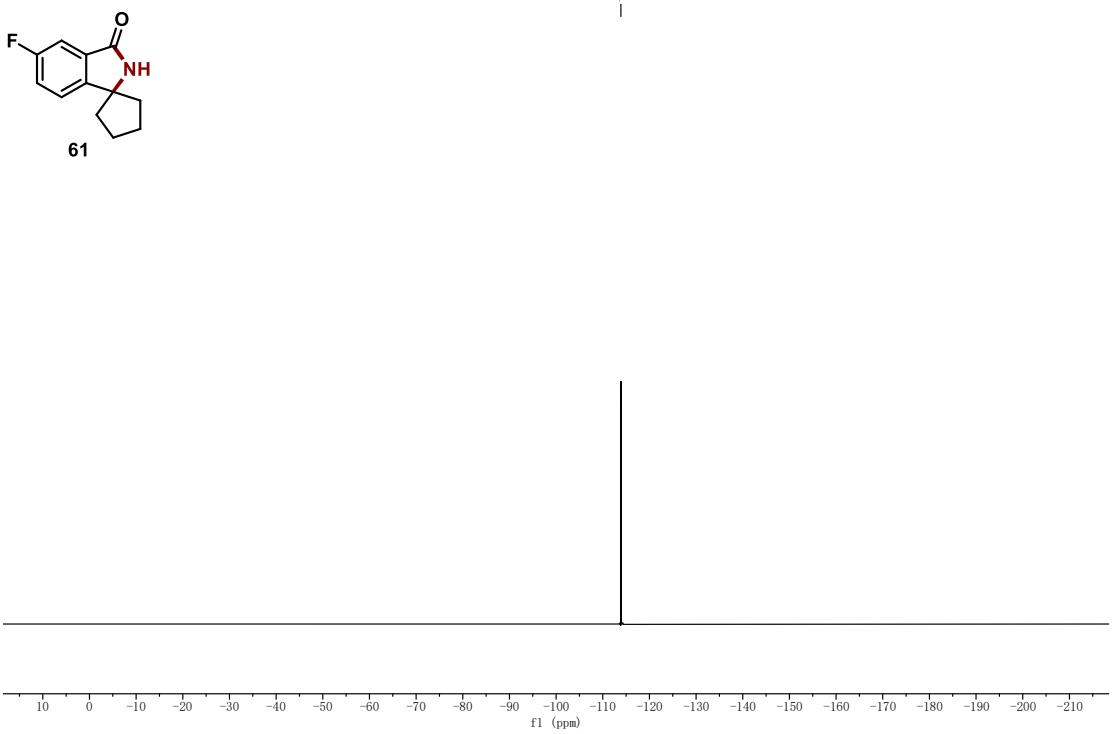

**Supplementary Figure 284** $^{19}$F NMR spectrum of compound **60**



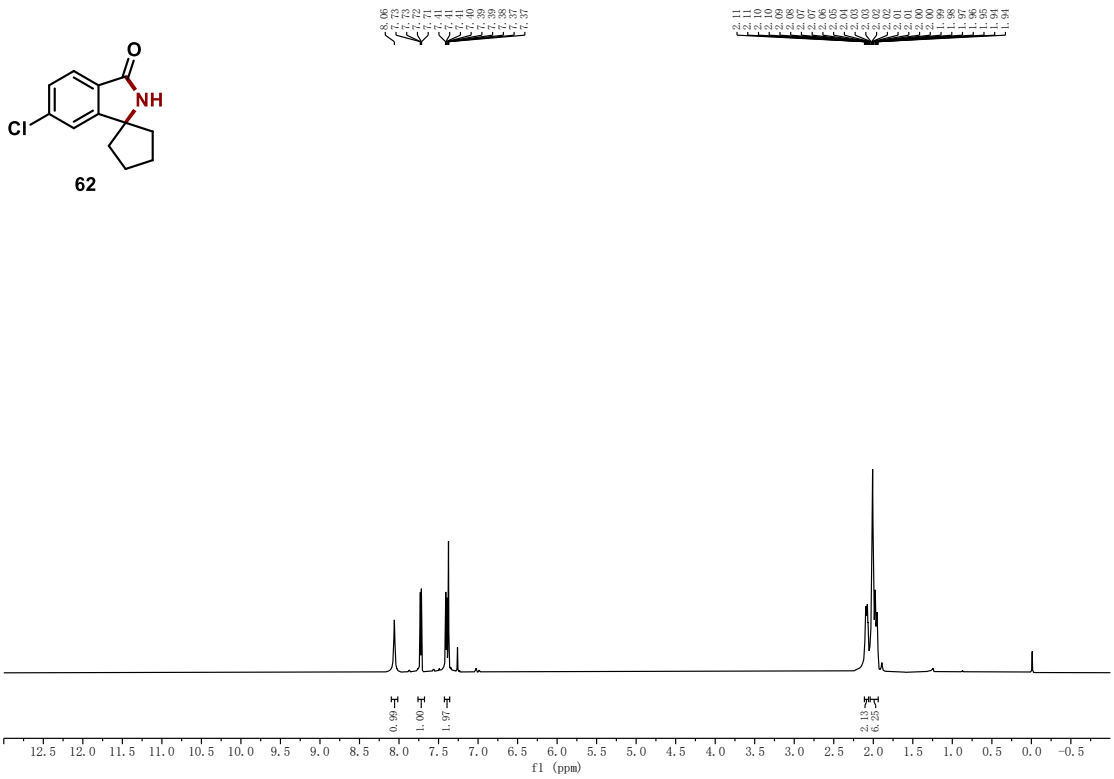

**Supplementary Figure 285** $^{1}$H NMR spectrum of compound **61**



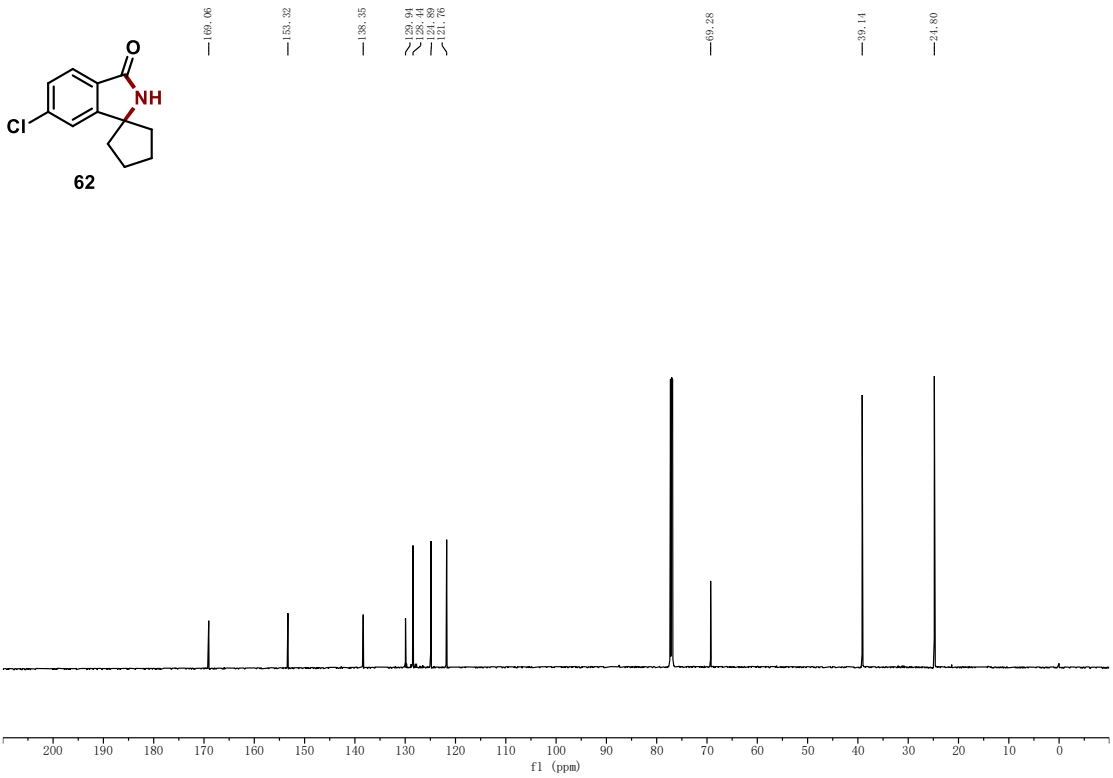

**Supplementary Figure 286** $^{13}$C NMR spectrum of compound **61**



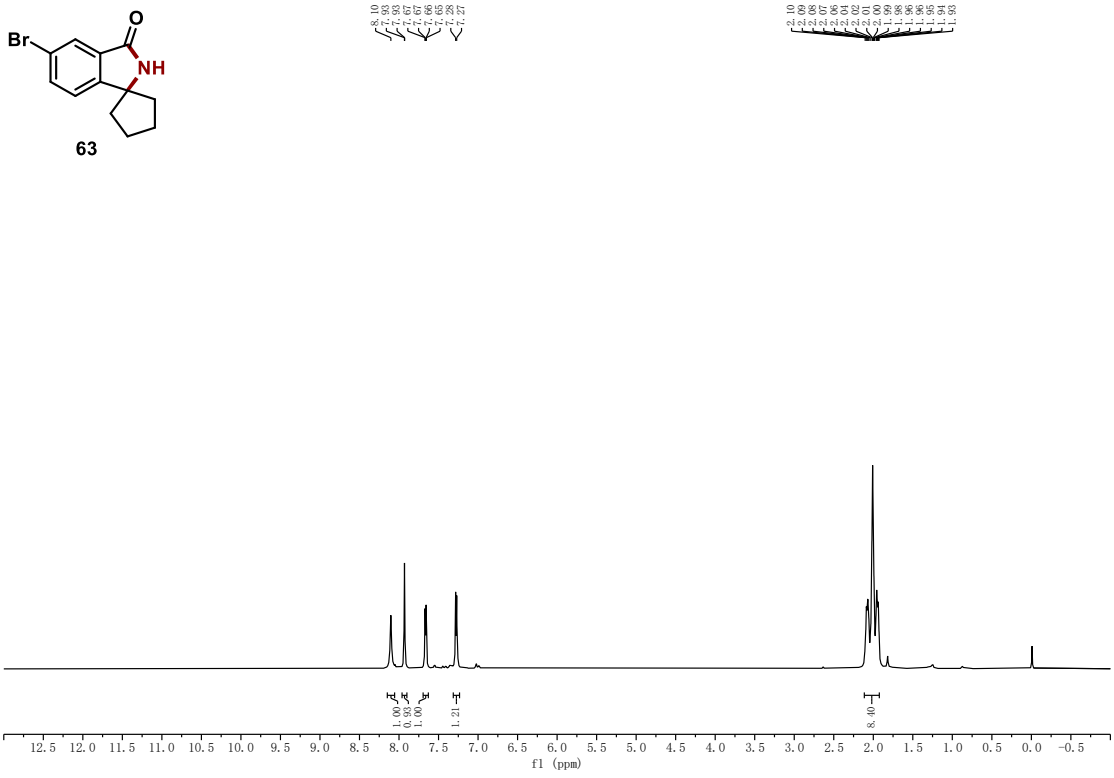

**Supplementary Figure 287** $^{19}$F NMR spectrum of compound **61**



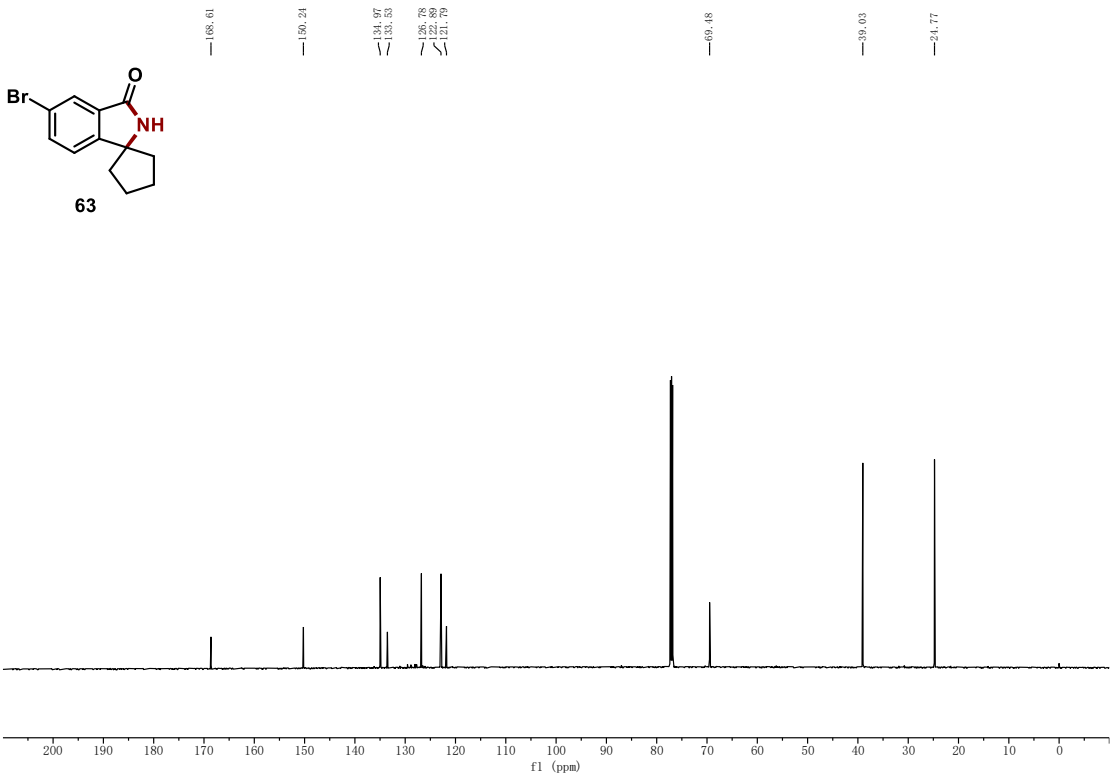

**Supplementary Figure 288** $^{1}$H NMR spectrum of compound **62**



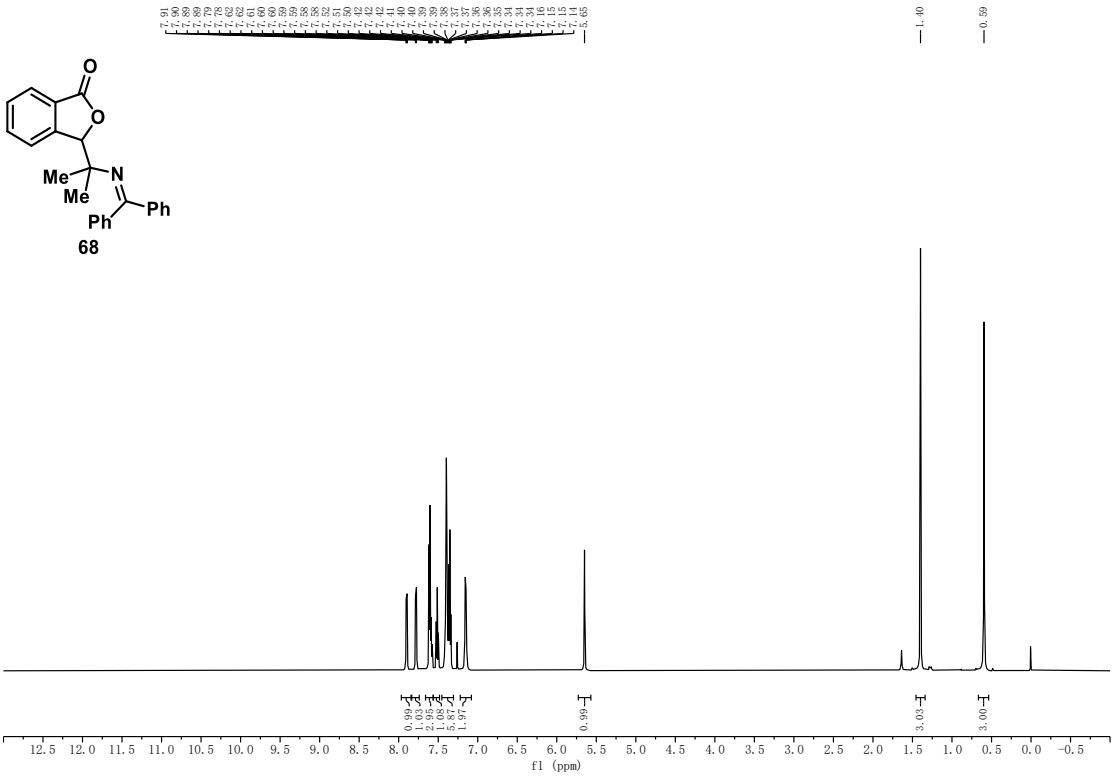

**Supplementary Figure 289** $^{13}$C NMR spectrum of compound **62**



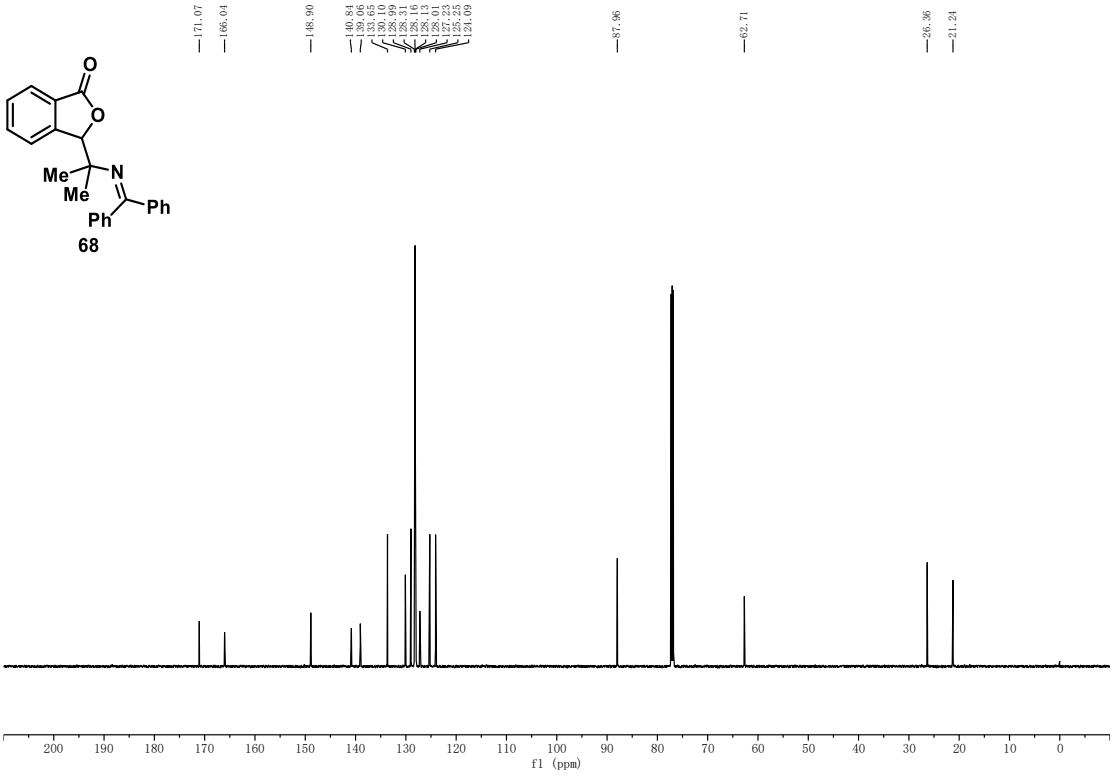

**Supplementary Figure 290** $^{1}$H NMR spectrum of compound **63**



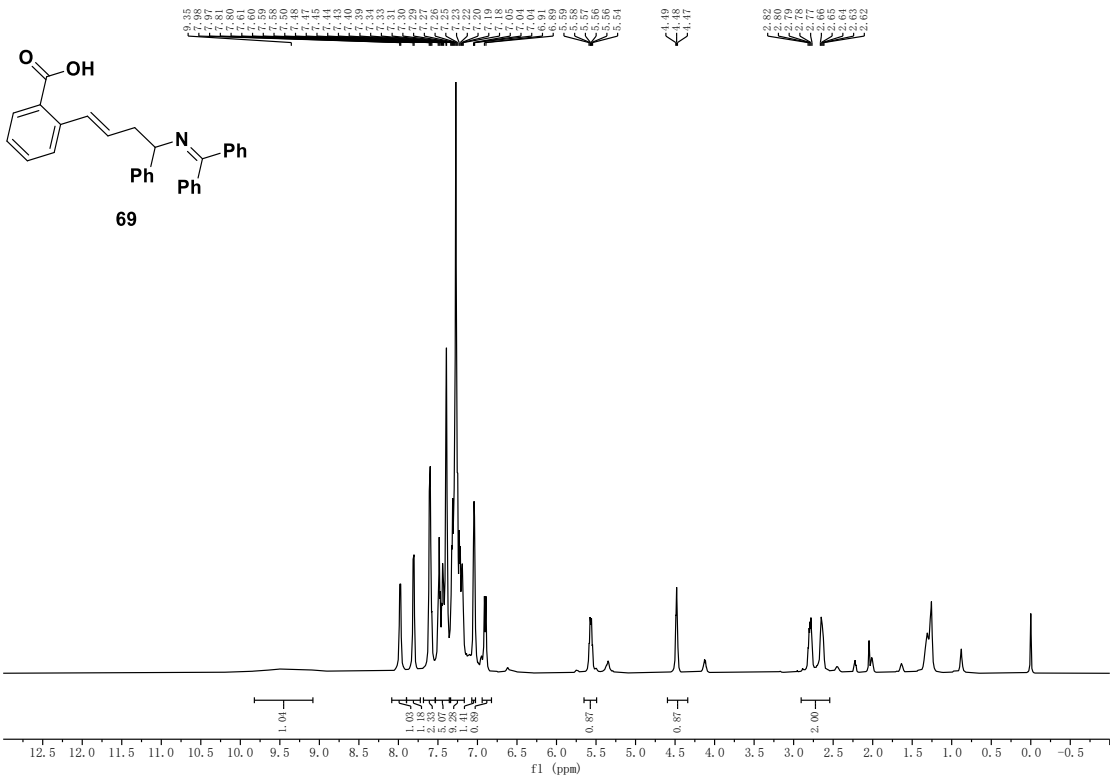

**Supplementary Figure 291** $^{13}$C NMR spectrum of compound **63**



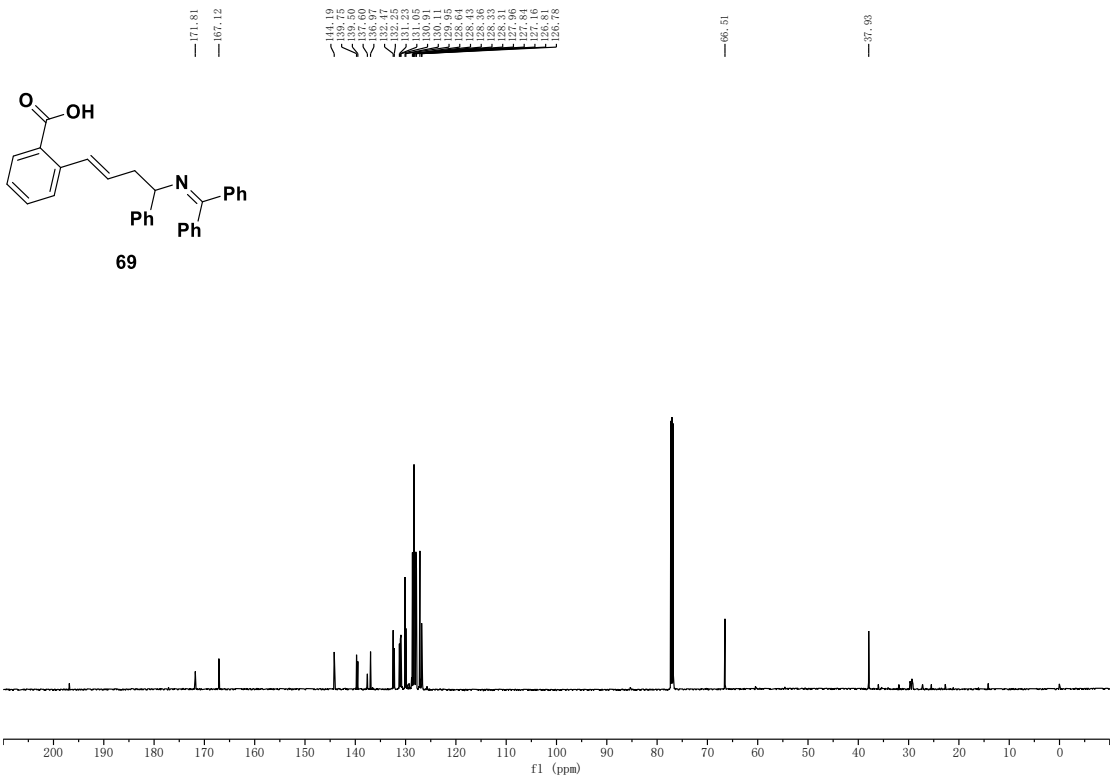

**Supplementary Figure 292** $^{1}$H NMR spectrum of compound **68**




**Supplementary Figure 293** $^{13}$C NMR spectrum of compound **68**



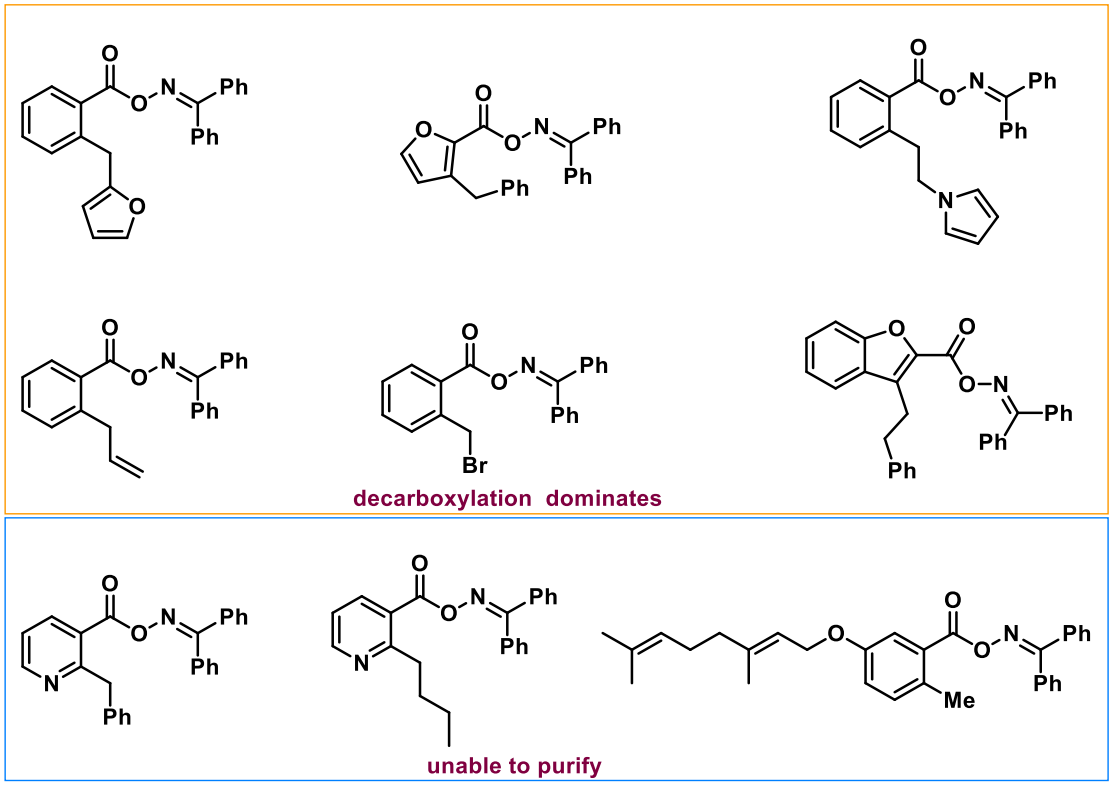

**Supplementary Figure 294** $^{1}$H NMR spectrum of compound **69**




**Supplementary Figure 295** $^{13}$C NMR spectrum of compound **69**

## 3.4 Unsuccessful Substrates

The unsuccessful substrates for the desired γ C-H Amination reactions are listed in Supplementary **Figure 296.**




**Supplementary Figure 296**. Unsuccessful substrates for the desired γ C-H amination reactions.

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