# Supporting Information

## Photoinduced N-heterocyclic nitrenium-catalyzed single electron reduction of Se-Se bond for the generation of nucleophilic selenolates

Chao-Shen Zhang,[b‡] Kun-Quan Chen,[b‡] Ling Zhou,[a] Zhu-Sheng Yang,[a*] Zhi-Xiang Wang[b,c*] and Xiang-Yu Chen[b,c*]

[a]School of Materials and Architectural Engineering, Guizhou Normal University, Guiyang 550025, China

[b]School of Chemical Sciences, University of Chinese Academy of Sciences Beijing 100049, China

[c]Binzhou Institute of Technology, Weiqiao-UCAS Science and Technology Park, Binzhou, Shandong Province 256606, China

# contents

1. General information .....S 错误!未定义书签。
2. General procedure for the selenation of alkyl chlorides .....S4
3. The mechanism study of C-Se bond formation .....S5
    3.1. Radical inhibition experiment .....S5
    3.2. Radical clock experiment .....S5
    3.3. Reaction of tertiary alkyl chloride .....S6
    3.4. Examining other leaving groups .....S6
    3.5. Stern-Volmer luminescence quenching study .....S6
4. Preparation of alkyl chlorides .....S9
5. Compound characterization data .....S12
6. NMR spectra .....S21
7. References .....S56

# 1. General Information

Chemicals were purchased from Heowns，Innochem or Bidepharm and used without further purification unless otherwise noted. Solvents were purified using a solvent-purification system (VSPS-8, Vigor) that contained activated alumina and molecular sieves. Nitrenium saltswere prepared according to literature methods.[1,2]

Analytical thin layer chromatography was carried out with silica gel pre-coated glass plates (TLC-Silica gel GF254, coating thickness: 0.25 mm) purchased from Xinnuo Chemical (Yantai, China). Chromatographic purification of the products was performed on silica gel 200-300 mesh. Visualization of the developed TLC plates was performed with ultraviolet irradiation (254 nm) or by staining with basic potassium permanganate solution.

High-resolution mass spectra (HRMS) were obtained with the mass analyzer of an orbitrap. The calculated values are based on the most abundant isotope.

IR spectra were taken on a Vertex 70 spectrophotometer and reported as wave numbers ($cm^{-1}$).

The WRS-2 microcomputer melting point meter was used to measure the melting point of solids.

The GC-MS TQ8040 was used in the detection of the reaction mixture.

UV-vis absorption spectra were acquired on UV-1900 spectrophotometer (Shimadzu, Japan).

Cyclic voltammetry studies were carried out on a Shanghai Chen Hua CHI440E electrochemical workstation.

Fluoresence spectra were recorded on a Edingburg FS5 Fluorescent Spectrophotometer.

The luminescence decays were measured on an Edingburg FLS980 spectrometer.

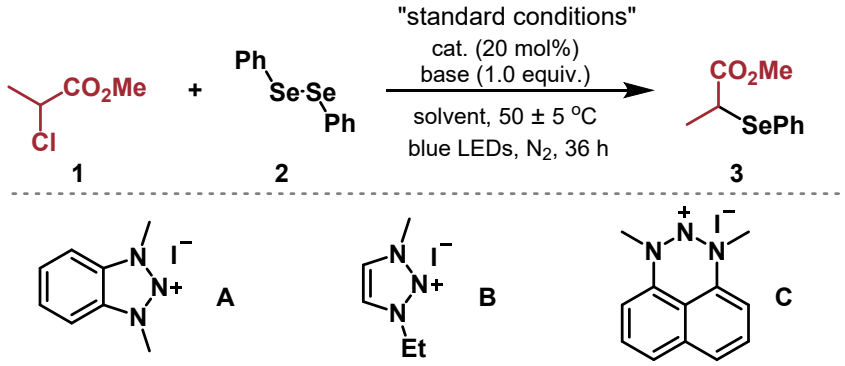
$^{1}H$- and $^{13}C$- NMR spectra were recorded at ambient temperature on a Shimadzu Avance 400/500 Spectrometer. The chemical shifts are reported in ppm downfield of tetramethylsilane (TMS) and referenced to residual solvent peaks resonance as internal standard. The order of citation in parentheses is a) multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd= doublet of doublet, ddd= doublet of doublet of doublet, td = triplet of doublet, m = multiplet, bs = broad signal), b) coupling constants, c) number of protons. Coupling constants (*J*) are reported in Hertz (Hz).

Photochemical experiments were performed magnetically stirred in 10 mL glass tubes, sealed with a rubber septum. The tubes were irradiated with blue light (450 nm) using a LED lamp with a power output of 100 W (see below pictures). The distance from the light source to the irradiation vessel is 2 cm to keep the reaction temperature with 50 ± 5 °C. (The purchase link of LED lamp is https://item.taobao.com/item.htm?spm=a230r.1.14.90.19013f311BIQEz&id=548675812368&ns=1&abbucket=0#detail).

**Figure S1.** The spectrum of blue LEDs employed in the reaction

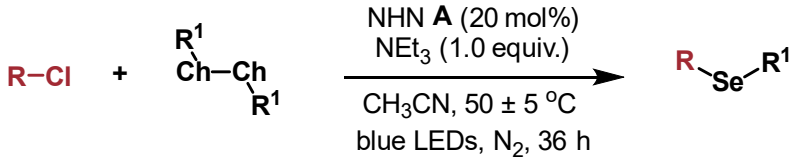
## 2. General procedure for the selenation of alkyl chlorides

**Scheme S1.** Optimization of the reaction conditions[a]




| Entry | Catalyst | Base | Solvent | Yield (%)[b] |
|---|---|---|---|---|
| 1 | **A** | $NEt_3$ | $CH_3CN$ | 84 |
| 2 | **B** | $NEt_3$ | $CH_3CN$ | 33 |
| 3 | **C** | $NEt_3$ | $CH_3CN$ | 50 |
| 4 | **A** | DBU | $CH_3CN$ | 27 |
| 5 | **A** | TMEDA | $CH_3CN$ | 49 |
| 6 | **A** | DABCO | $CH_3CN$ | 22 |
| 7 | **A** | pyridine | $CH_3CN$ | 32 |
| 8 | **A** | NaOAc | $CH_3CN$ | 27 |
| 9 | **A** | $Na_2CO_3$ | $CH_3CN$ | 26 |
| 10 | **A** | $NEt_3$ | DCM | 29 |
| 11 | **A** | $NEt_3$ | THF | 49 |
| 12 | **A** | $NEt_3$ | DMA | trace |
| 13 | **A** | $NEt_3$ | DMSO | trace |
| 14 | **A** | $NEt_3$ | Toluene | NR |
| 15 | **A** | $NEt_3$ | HFIP | NR |
| 16 | **A** | --- | $CH_3CN$ | 9 |
| 17[c] | **A** | $NEt_3$ | $CH_3CN$ | NR |
| 18[d] | **A** | $NEt_3$ | $CH_3CN$ | 84 |
| 19[e] | **A** | $NEt_3$ | $CH_3CN$ | 80 |

[a] Conditions: alkyl chloride **1** (0.2 mmol), diselenide **2** (0.1 mmol), catalyst (20 mol%), $NEt_3$ (0.1 mmol), $CH_3CN$ (0.4 M), 36 h. [b] Yield of isolated product given. [c] No irradiation, 60 °C. [d] Alkyl chloride **1** (0.4 mmol), diselenide **2** (0.2 mmol), catalyst (20 mol%), $NEt_3$ (0.2 mmol), $CH_3CN$ (0.4 M), 36 h. [e] Alkyl chloride **1** (0.2 mmol), diselenide **2** (0.4 mmol). NR, No Reaction. DIPEA = N,N-Diisopropylethylamine. TMEDA = N,N,N',N'-Tetramethylethylenediamine. DBU = 1,8-Diazabicyclo [5.4.0] undec-7-ene.



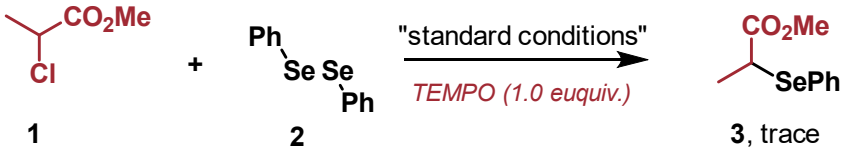

**General procedure A:** Alkyl chloride (0.4 mmol), diselenide (0.2 mmol), NHN **A1** (10.9 mg, 0.04 mmol,

20 mol%) and $NEt_3$ (20.3 mg, 0.2 mmol, 27.8 μL) were added sequentially into a 10 mL dry tube under nitrogen, then $CH_3CN$ (1 mL) was added by syringe. After degas, the resulting mixture was allowed to stir under blue LED irradiation for 36 h, where the distance from the light source to the irradiation vessel is 2 cm to keep the reaction temperature at 50 ± 5 °C. Upon completion, the solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate as eluent to afford the desired product.

**General procedure B (gram scale):** 2-chloropropanoate (390.4 mg, 3.2 mmol), 1,2-diphenyldiselane (499.2 mg, 1.6 mmol), NHN **A1** (87.2 mg, 0.32 mmol, 20 mol%) and $NEt_3$ (162.4 mg, 1.6 mmol, 222.4 μL) were added sequentially into a 30 mL dry tube under nitrogen, then $CH_3CN$ (8 mL) was added by syringe. After degas, the resulting mixture was allowed to stir under blue LED irradiation for 36 h, where the distance from the light source to the irradiation vessel is 2 cm to keep the reaction temperature at 50 ± 5 °C. Upon completion, the solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate as eluent to afford 406 mg the desired product, yield 52%.

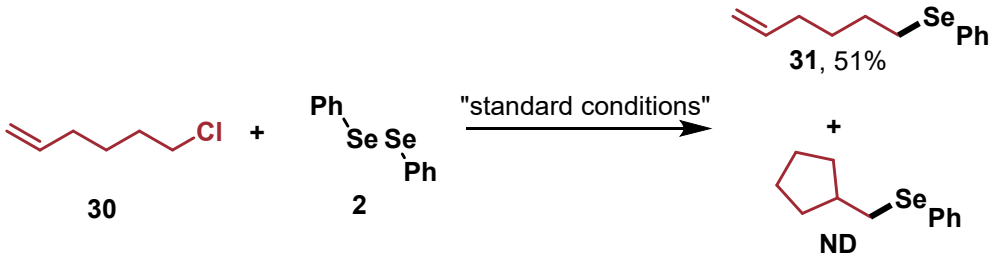
## 3. The mechanism study of C-Se bond formation

### 3.1. Radical inhibition experiment




In a nitrogen atmosphere, to a dry tube equipped with a stirring bar, methyl 2-chloropropanoate (48.8 mg, 0.4 mmol), 1,2-diphenyldiselane (62.4 mg, 0.2 mmol), NHN **A1** (10.9 mg, 0.04 mmol, 20 mol%), $NEt_3$ (20.3 mg, 0.2 mmol, 27.8 μL), TEMPO (31.2 mg, 0.2 mmol) and MeCN (1.0 mL) were added. Then, the resulting mixture was allowed to stir under blue LED irradiation for 36 h, and only trace amount of the product was observed.

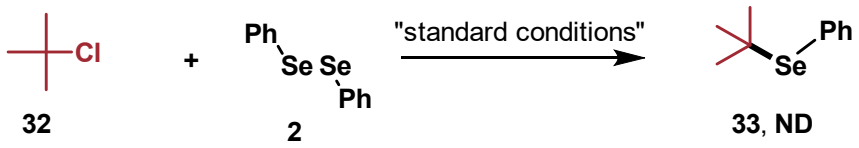
### 3.2. Radical clock experiment




The 6-chlorohex-1-ene (47.2 mg, 0.4 mmol), 1,2-diphenyldiselane (62.4 mg, 0.2 mmol), NHN **A1** (10.9 mg,

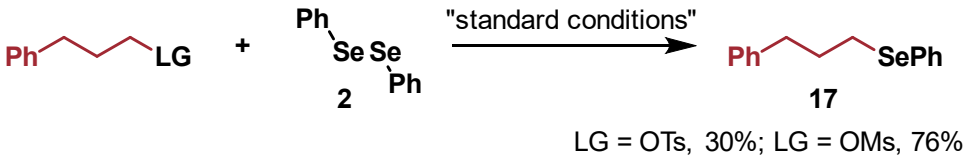
0.04 mmol, 20 mol%), $NEt_3$ (20.3 mg, 0.2 mmol, 27.8 μL) and MeCN (1.0 mL) were added sequentially into a 10 mL dry tube under nitrogen. The resulting mixture was allowed to stir under blue LED irradiation for 36 h giving the linear product **31** in 51% yield and no cyclization product was detected.

### 3.3. Reaction of tertiary alkyl chloride




Under the standard conditions, *t*BuCl (36.8 mg, 0.4 mmol), 1,2-diphenyldiselane (62.4 mg, 0.2 mmol), NHN **A1** (10.9 mg, 0.04 mmol, 20 mol%), $NEt_3$ (20.3 mg, 0.2 mmol, 27.8 μL) and MeCN (1.0 mL) were added sequentially into a 10 mL dry tube under nitrogen. The resulting mixture was allowed to stir under blue LED irradiation for 36 h and the desired product **33** was not observed.

### 3.4. Examining other leaving groups




3-Phenylpropyl 4-methylbenzenesulfonate (116.0 mg, 0.4 mmol) or 3-phenylpropyl methanesulfonate (85.6 mg, 0.4 mmol), NHN **A1** (10.9 mg, 0.04 mmol, 20 mol%), $NEt_3$ (20.3 mg, 0.2 mmol, 27.8 μL) and MeCN (1.0 mL) were added sequentially into a 10 mL dry tube under nitrogen. The resulting mixture was allowed to stir under blue LED irradiation for 36 h giving the desired product **17** in 30% (LG = OTs) or 76% (LG = OMs) yields, respectively.

### 3.5. Stern-Volmer luminescence quenching study

Luminescence intensities were recorded using an Edinburgh instruments FS5 spectrofluorometer excited at 280 nm. All luminescence measurements were recorded using a quartz cuvette (fluorescence quartz cuvette, 10 x 10 mm, 3.5 mL). Quenching was analyzed by plotting $\mathbf{I_0/I}$ according to the Stern-Volmer relationship: $\mathbf{I_0/I} = k_q\tau_0[Q]+1$ where $\mathbf{I_0}$ represents the integral of the luminescence over the range of 290 to 550 nm in the absence of a quencher, **I** is the integral of luminescence over the range of 290 to 550 nm in the presence of a quencher, $k_q$ represents the quenching rate constant, [Q] is the

concentration of a given quencher.

| NHN **A1** | **Quencher** | **Ratio (A1 : Q)** |
|---|---|---|
| **0.00001M** | **0.0001M** | **1 : 10** |
| | **0.0002M** | **1 : 20** |
| | **0.0003M** | **1 : 30** |
| | **0.0004M** | **1 : 40** |
| | **0.0005M** | **1 : 50** |

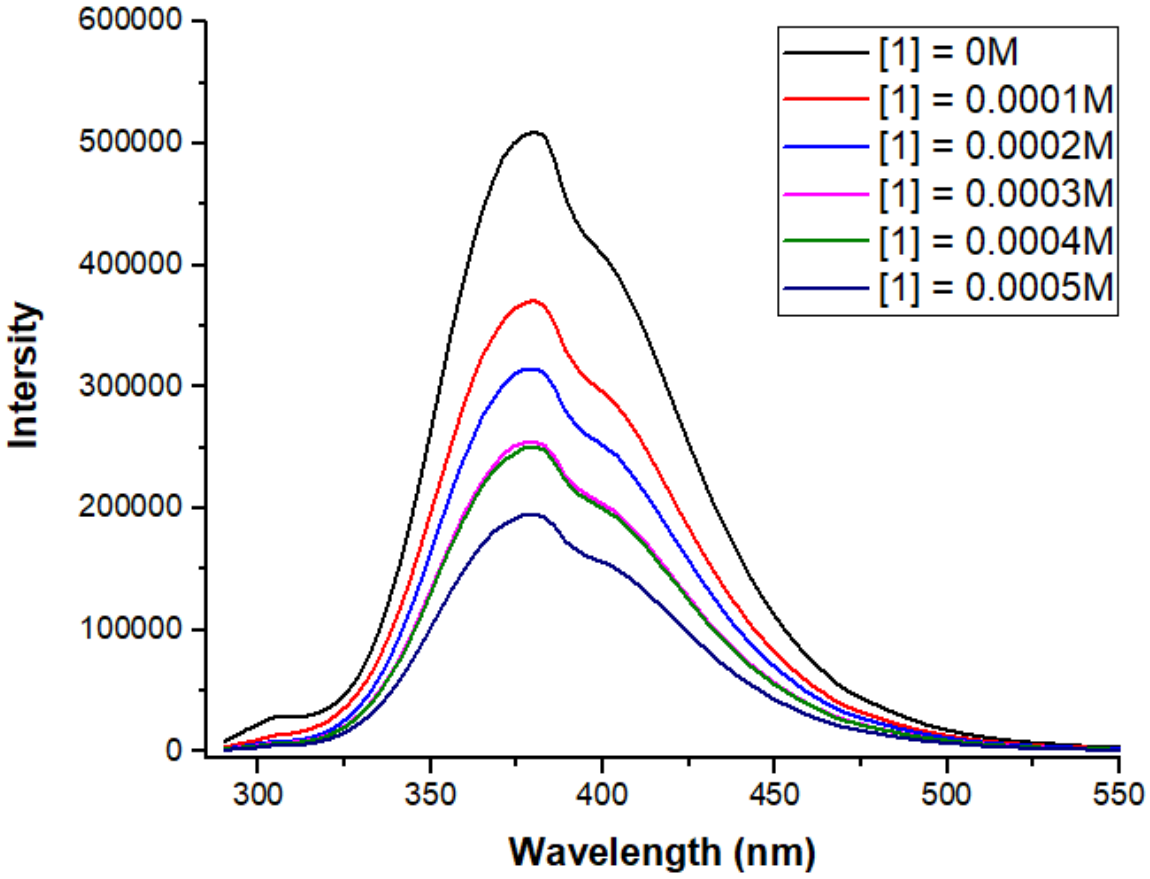



**Figure S2.** Fluorescence emission spectrum of NHN **A1** (0.01 mM) at different concentrations of alkyl chloride **1**.

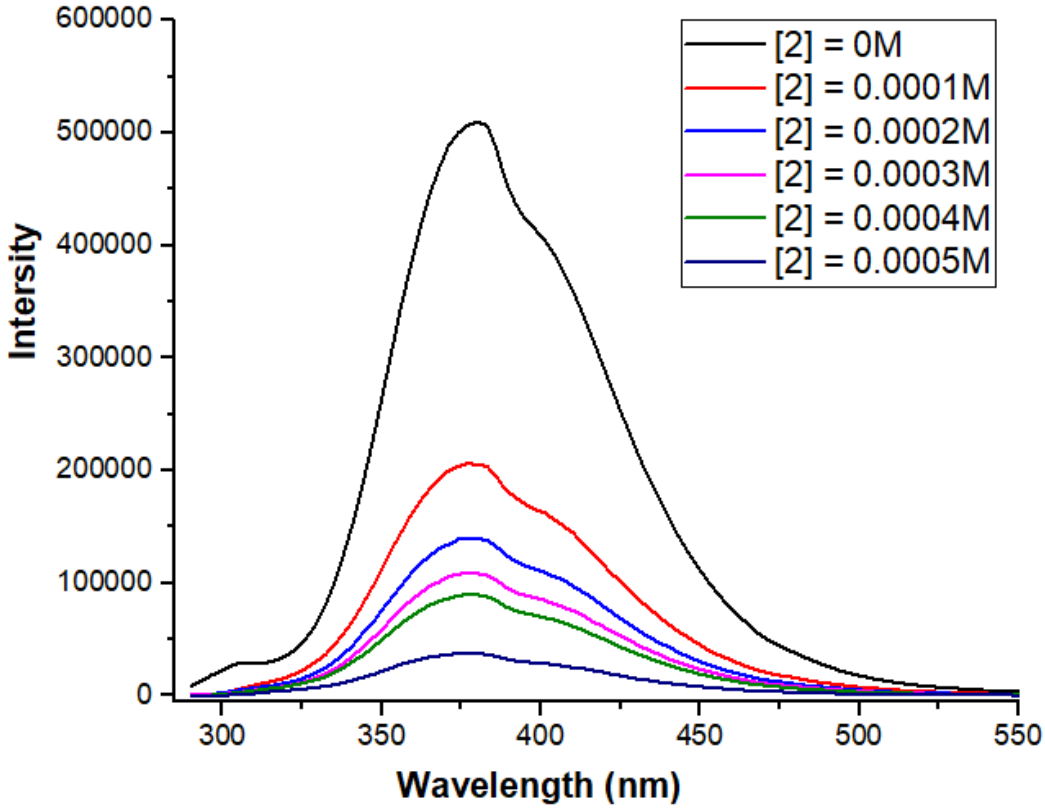



**Figure S3.** Fluorescence emission spectrum of NHN **A1** (0.01 mM) at different concentrations of diselenide **2**.

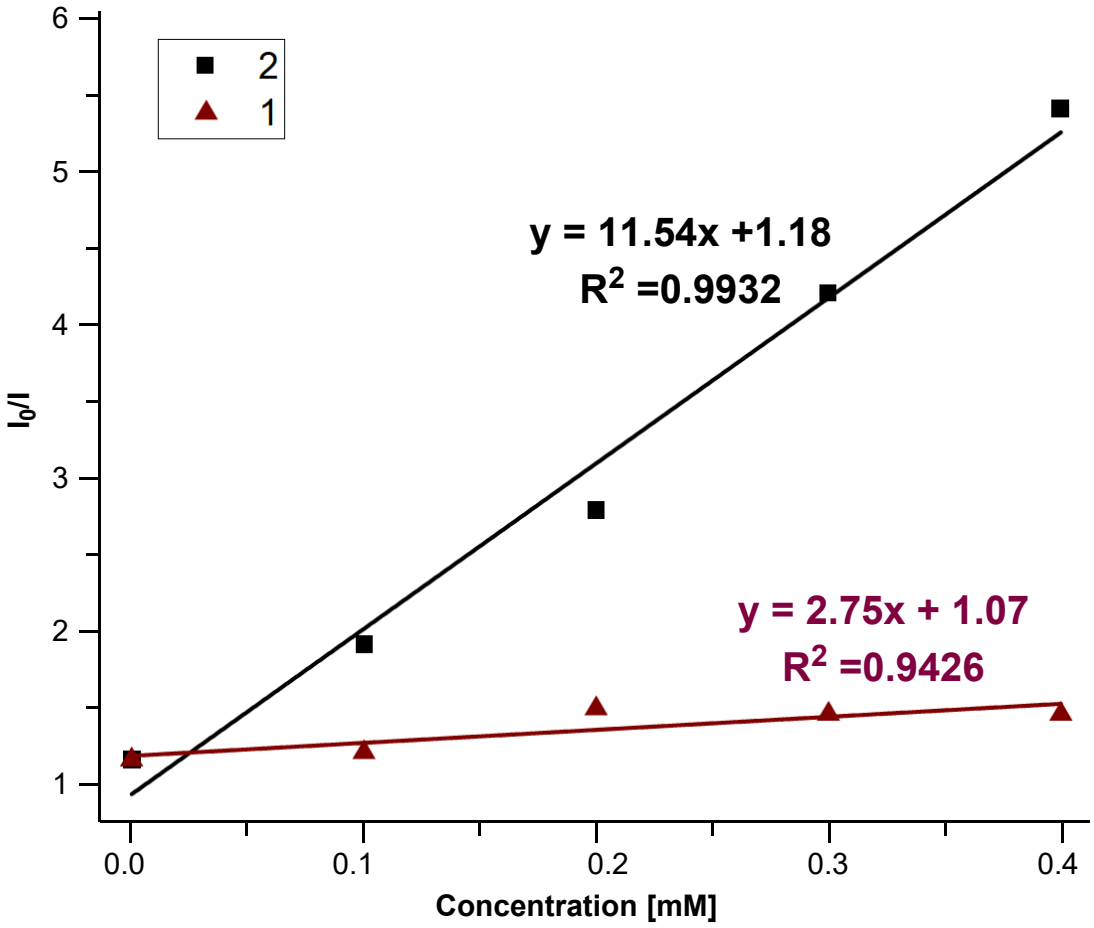
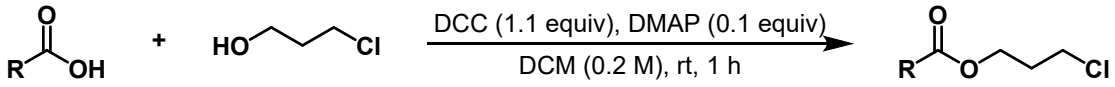



**Figure S4.** Stern-Volmer plot of NHN **A1** at different concentrations of quencher.

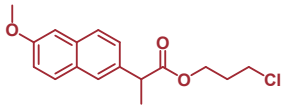
**4. Preparation of alkyl chlorides**

Alkyl chlorides **25-Cl** – **29-Cl** were prepared following literature procedure,[3] and other substrates were purchased from commercial sources and used directly without further purification.




**25-Cl - 29-Cl**

To a flask equipped with a stirrer-bar were added the corresponding carboxylic acid (3.0 mmol, 1.0 equiv.), *N,N'*-dicyclohexylcarbodiimide (749 mg, 3.3 mmol, 1.1 equiv.), 4-(dimethylamino)pyridine (36.6 mg, 0.3 mmol, 0.1 equiv.), 3-chloro-1-propanol (375 µL, 4.5 mmol, 1.5 equiv.), and $CH_2Cl_2$ (15.0 mL). The resulting mixture was stirred at room temperature for 1 h and filtered to remove the urea by-product. The resulting mixture was concentrated under reduced pressure and purified by flash column chromatography (silica gel, PE/EtOAc gradient elution) to afford the desired products **25-Cl** – **29-Cl**.

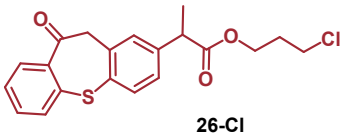


25-Cl

**3-Chloropropyl-2-(6-methoxynaphthalen-2-yl)propanoate (25-Cl)**

The title product was obtained after purification by column chromatography (PE/EA = 10:1) as a colorless oil (780.6 mg, 2.55 mmol, 85%).

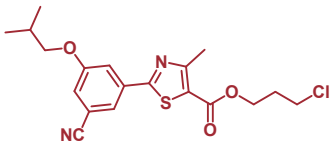
**$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.72 – 7.67 (m, 2H), 7.67 – 7.64 (m, 1H), 7.38 (dd, *J* = 8.5, 1.9 Hz, 1H), 7.16 – 7.08 (m, 2H), 4.29 – 4.16 (m, 2H), 3.89 (s, 3H), 3.85 (q, *J* = 7.2 Hz, 1H), 3.46 – 3.42 (m, 2H), 2.02 – 1.97 (m, 2H), 1.57 (d, *J* = 7.1 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 174.6, 157.7, 135.7, 133.8, 129.3, 129.0, 127.23 126.2, 126.0, 119.1, 105.6, 61.5, 55.4, 45.4, 41.2, 31.5, 18.5. **IR (ATR)**: 2966, 1729, 1605, 1505, 1483, 1452, 1028, 852 cm$^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{17}H_{19}ClO_3Na^+$:329.0915; found 329.0912.



26-Cl

**3-chloropropyl 2-(10-oxo-10,11-dihydrodibenzo[*b,f*]thiepin-2-yl)propanoate (26-Cl)**

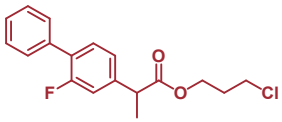
The title product was obtained after purification by column chromatography (PE/EA = 5:1) as a colorless oil (908.6 mg, 2.43 mmol, 81%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 8.15 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.59 – 7.53 (m, 2H), 7.42 – 7.33 (m, 2H), 7.28 – 7.25 (m, 1H), 7.11 (dd, *J* = 8.0, 2.0 Hz, 1H), 4.32 (s, 2H), 4.18 (t, *J* = 6.1 Hz, 2H), 3.70 (q, *J* = 7.2 Hz, 1H), 3.45 – 3.42 (m, 2H), 2.03 – 1.96 (m, 2H), 1.46 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 191.2, 173.7, 142.6, 140.1, 138.0, 136.1, 133.3, 132.5, 131.5, 131.5, 130.9, 128.6, 126.9, 126.3, 61.6, 51.0, 45.1, 41.1, 31.4, 18.4. **IR (ATR)**: 2968, 1730, 1670, 1586, 1458, 1427, 1282, 1155, 1071, 831, 754 cm$^{-1}$ **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{20}H_{19}ClO_3SNa^+$: 397.0636; found 397.0635.



27-Cl

**3-chloropropyl 2-(3-cyano-5-isobutoxyphenyl)-4-methylthiazole-5-carboxylate (27-Cl)**

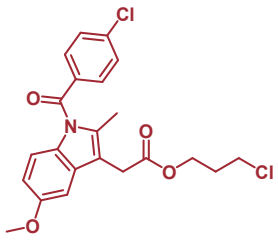
The title product was obtained after purification by column chromatography (PE/EA = 5:1) as white solid (1070.4 mg, 2.73 mmol, 91%). **Melting Point:** 121 - 123 °C. **$^1$H NMR (500 MHz, $CDCl_3$)** δ 8.18 – 7.96 (m, 2H), 6.99 – 6.97 (m, 1H), 4.43 (t, *J* = 6.7 Hz, 2H), 3.87 (d, *J* = 7.6 Hz, 2H), 3.67 (t, *J* = 6.4 Hz, 2H), 2.73 (s, 3H), 2.23 – 2.13 (m, 3H), 1.06 (d, *J* = 6.7 Hz, 6H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 167.5, 162.6, 161.9, 161.6, 132.7, 132.1, 125.9, 121.4, 115.5, 112.7, 103.0, 75.8, 62.1, 41.2, 31.6, 28.2, 19.1, 17.6. **IR (ATR)**: 2967, 2227, 1687, 1601, 1508, 1468, 1042, 761 cm$^{-1}$ **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{19}H_{21}ClN_2O_3SNa^+$: 415.0854; found 415.0849.




28-Cl

**3-chloropropyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (28-Cl)**

The title product was obtained after purification by column chromatography (PE/EA = 10:1) as colorless oil (835.5 mg, 2.61 mmol, 87%). **$^{1}$H NMR (500 MHz, $CDCl_3$)** δ 7.54 -7.52 (m, 2H), 7.45 – 7.33 (m, 4H), 7.17 – 7.08 (m, 2H), 4.24 (t, *J* = 6.1 Hz, 2H), 3.75 (q, *J* = 7.2 Hz, 1H), 3.51 – 3.47 (m, 2H), 2.07 – 2.02 (m, 2H), 1.53 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 173.9, 159.8 (d, *J* = 248.9 Hz), 141.8 (d, *J* = 7.7 Hz), 135.6, 131.0 (d, *J* = 3.8 Hz), 129.1 (d, *J* = 3.0 Hz), 128.0 (d, *J* = 13.4 Hz), 128.0, 127.9, 123.6 (d, *J* = 3.2 Hz), 115.4 (d, *J* = 23.8 Hz), 61.7, 45.0, 41.1, 31.5, 18.3. **$^{19}$F NMR (471 MHz, $CDCl_3$)** δ -117.38 (t, *J* = 9.3 Hz). **IR (ATR)**: 2970, 1732, 1581, 1483, 1170, 1074, 765 $cm^{-1}$ **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{18}H_{18}ClFO_2Na^+$:343,0872; found 343.0869.

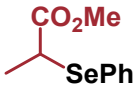



29-Cl

**3-chloropropyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (29-Cl)**

The title product was obtained after purification by column chromatography (PE/EA = 5:1) as pale-yellow solid (896.1 mg, 2.07 mmol, 69%). **Melting Point:** 73 - 75 °C. **$^{1}$H NMR (500 MHz, $CDCl_3$)** δ 7.67 – 7.60 (m, 2H), 7.49 – 7.42 (m, 2H), 6.96 – 6.83 (m, 2H), 6.65 (dd, *J* = 9.0, 2.5 Hz, 1H), 4.24 (t, *J* = 6.1 Hz, 2H), 3.82 (s, 3H), 3.66 (s, 2H), 3.50 (t, *J* = 6.4 Hz, 2H), 2.37 (s, 3H), 2.08 – 2.03 (m, 2H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 170.7, 168.3, 156.1, 139.3, 136.0, 133.9, 131.2, 130.8, 130.6, 129.2, 115.1, 112.5, 111.8, 101.2, 61.7, 55.7, 41.1, 31.5, 30.3, 13.4. **IR (ATR)**: 3022, 1716, 1667, 1602, 1476, 1448, 1356, 1088, 959, 755, 602 $cm^{-1}$ **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{22}H_{21}Cl_2NO_4Na^+$:456.0740; found 456.0739.

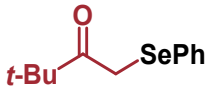
## 5. Compound characterization data



3

**methyl 2-(phenylselanyl)propanoate (3)**

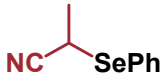
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow oil (41.0 mg, 0.168 mmol, 84%). **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.59 – 7.57 (m, 2H), 7.34 – 7.24 (m, 3H), 3.77 (q, *J* = 7.2 Hz, 1H), 3.63 (s, 3H), 1.53 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 174.0, 135.9, 129.1, 128.7, 127.7, 52.2, 37.2, 17.8. These data are in agreement with those reported previously in the literature.[4]



4

**3,3-dimethyl-1-(phenylselanyl)butan-2-one (4)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (36.9 mg, 0.144 mmol, 72%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.57 – 7.55 (m, 2H), 7.30 – 7.26 (m, 3H), 3.88 (s, 2H), 1.19 (s, 9H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 210.9, 133.7, 129.7, 129.3, 127.8, 44.4, 32.8, 27.0. These data are in agreement with those reported previously in the literature.[5]

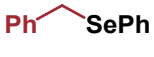


5

**2-(phenylselanyl)propanenitrile (5)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (23.6 mg, 0.112 mmol, 56%).

**$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.76 – 7.70 (m, 2H), 7.46 – 7.35 (m, 3H), 3.71 (q, *J* = 7.4 Hz, 1H), 1.66 (d, *J* = 7.3 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 136.6, 129.8, 129.6, 126.0, 120.9, 19.6, 19.4. These data are in agreement with those reported previously in the literature.[6]

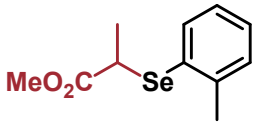


6

**benzyl(phenyl)selane (6)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (33.7 mg, 0.136 mmol, 68%). **$^1$H NMR (500 MHz, $CDCl_3$)**

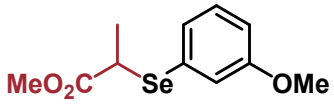
δ 7.48 – 7.42 (m, 2H), 7.26 – 7.19 (m, 8H), 4.11 (s, 2H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 138.7, 133.7, 130.5, 129.1, 129.0, 128.6, 127.4, 127.0, 32.4. These data are in agreement with those reported previously in the literature.[7]




**7**

**methyl 2-(o-tolylselanyl)propanoate (7)**

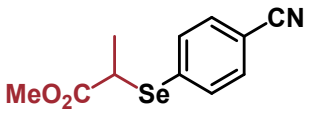
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (44.4 mg, 0.172 mmol, 86%). **$^{1}$H NMR (500 MHz, $CDCl_3$)** δ 7.44 – 7.36 (m, 2H), 7.22 – 7.12 (m, 2H), 3.77 (q, *J* = 7.1 Hz, 1H), 3.65 (s, 3H), 2.34 (s, 3H), 1.55 (d, *J* = 7.1 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 174.1, 138.9, 136.5, 132.8, 129.5, 128.9, 127.6, 52.2, 37.3, 21.4, 17.9. **IR (ATR)**: 2918, 1729, 1433, 1330, 1255, 1059, 995, 853 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{11}H_{14}O_2SeNa^+$:281.0051; found 281.0049.




**8**

**methyl 2-((3-methoxyphenyl)selanyl)propanoate (8)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (42.7 mg, 0.156 mmol, 78%). **$^{1}$H NMR (500 MHz, $CDCl_3$)** δ 7.25 – 7.12 (m, 3H), 6.89 – 6.87 (m, 1H), 3.82 – 3.77 (m, 4H), 3.67 (s, 3H), 1.56 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 174.1, 159.7, 129.9, 128.8, 127.8, 120.8, 114.6, 55.5, 52.3, 37.4, 17.9. **IR (ATR)**: 2950, 1727, 1586, 1475, 1330, 1210, 1035, 992, 838 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{11}H_{14}O_3SeNa^+$:297.0000; found 296.9997.

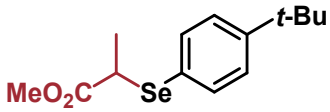



**9**

**methyl 2-((4-cyanophenyl)selanyl)propanoate (9)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (40.9 mg, 0.152 mmol, 76%). **$^{1}$H NMR (500 MHz, $CDCl_3$)** δ 7.69 – 7.64 (m, 2H), 7.58 – 7.55 (m, 2H), 3.90 (q, *J* = 7.2 Hz, 1H), 3.69 (s, 3H), 1.61 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 173.5, 135.7, 134.3, 133.4, 133.1, 132.4, 118.6, 111.7, 52.6, 37.4, 17.8. **IR (ATR)**:

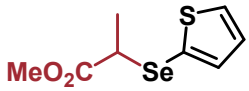
2226, 1726, 1586, 1486, 1330, 1258, 1061, 821 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{11}H_{11}O_2NSeNa^+$:291.9847; found 291.9846.




**10**

**methyl 2-((4-(tert-butyl)phenyl)selanyl)propanoate (10)**

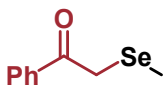
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (38.2 mg, 0.142 mmol, 71%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 3.75 (q, *J* = 7.1 Hz, 1H), 3.65 (s, 3H), 1.53 (d, *J* = 7.1 Hz, 3H), 1.31 (s, 9H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 174.2, 152.1, 135.9, 126.2, 124.2, 52.2, 37.2, 34.8, 31.4, 17.9. **IR (ATR)**: 2959, 1730, 1449, 1330, 1206, 1143, 824 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{14}H_{20}O_2SeNa^+$:323.0521; found 323.0518.




**11**

**methyl 2-(thiophen-2-ylselanyl)propanoate (11)**

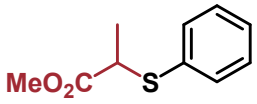
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (40.0 mg, 0.160 mmol, 80%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.46 (dd, *J* = 5.3, 1.2 Hz, 1H), 7.24 (dd, *J* = 3.5, 1.2 Hz, 1H), 7.03 (dd, *J* = 5.3, 3.5 Hz, 1H), 3.68 (q, *J* = 7.2, 1H), 3.68 (s, 3H) 1.53 (d, *J* = 7.1 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 173.4, 138.3, 132.8, 128.3, 121.0, 52.4, 39.2, 17.5. **IR (ATR)**: 2917, 2848, 1728, 1644, 1433, 1329, 962, 845 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_8H_{10}O_2SSeNa^+$:272.9459; found 272.9456.




**12**

**2-(methylselanyl)-1-phenylethan-1-one (12)**

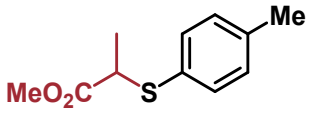
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (31.7 mg, 0.148 mmol, 74%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.96 – 7.93 (m, 2H), 7.58 – 7.53 (m, 1H), 7.48 – 7.44 (m, 2H), 3.75 (s, 2H), 2.07 (s, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 194.7, 135.3, 133.3, 128.8, 128.8, 28.0, 6.0. These data are in agreement with those reported previously in the literature.[8]



**13**

**methyl 2-(phenylthio)propanoate (13)**

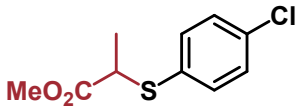
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (22.4 mg, 0.114 mmol, 57% **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.47 – 7.43 (m, 2H), 7.34 – 7.28 (m, 3H), 3.80 (q, *J* = 7.1 Hz, 1H), 3.67 (s, 3H), 1.49 (d, *J* = 7.1 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 173.2, 133.2, 133.1, 129.1, 128.2, 52.4, 45.3, 17.6. These data are in agreement with those reported previously in the literature.[9]



**14**

**methyl 2-(p-tolylthio)propanoate (14)**

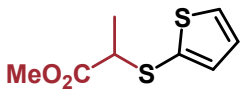
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (26.0 mg, 0.124mmol, 62%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.35 (d, *J* = 8.1 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 2H), 3.72 (q, *J* = 7.1 Hz, 1H), 3.67 (s, 3H), 2.34 (s, 3H), 1.45 (d, *J* = 7.1 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 173.3, 138.6, 134.0, 129.9, 129.2, 52.3, 45.6, 21.3, 17.5. These data are in agreement with those reported previously in the literature.[4]



**15**

**methyl 2-((4-chlorophenyl)thio)propanoate (15)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (26.2 mg, 0.114 mmol, 57%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.40 – 7.36 (m, 2H), 7.30 – 7.26 (m, 2H), 3.76 (q, *J* = 7.1 Hz, 1H), 3.68 (s, 3H), 1.47 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 173.0, 134.7, 134.6, 131.6, 129.3, 52.5, 45.4, 17.4. These data are in agreement with those reported previously in the literature.[4]



**16**

**methyl 2-(thiophen-2-ylthio)propanoate (16)**

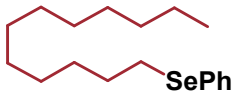
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (20.6 mg, 0.102 mmol, 51%).

**$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.43 (dd, *J* = 5.4, 1.2 Hz, 1H), 7.18 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.02 (dd, *J* = 5.4, 3.6 Hz, 1H), 3.70 (s, 3H), 3.63 (q, *J* = 7.1 Hz, 1H), 1.46 (d, *J* = 7.1 Hz, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 172.6, 136.8, 131.6, 130.2, 127.8, 52.5, 47.5, 17.1. **IR (ATR)**: 2949, 1731, 1433, 1400, 1217, 1065, 989, 847 $cm^{-1.}$ **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_8H_{10}O_2S_2Na^+$:225.0014; found 225.0012.


**17**

**phenyl(3-phenylpropyl)selane (17)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 100:1) as a colorless oil (30.9 mg, 0.112 mmol, 56%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.49 – 7.44 (m, 2H), 7.29 – 7.26 (m, 2H), 7.25 – 7.13 (m, 6H), 2.92 (t, *J* = 7.3 Hz, 2H), 2.73 (t, *J* = 7.5 Hz, 2H), 2.05 – 1.99 (m, 2H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 141.4, 132.7, 130.4, 129.2, 128.7, 128.5, 126.9, 126.1, 35.8, 31.7, 27.3. These data are in agreement with those reported previously in the literature.[10]

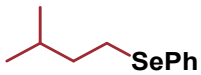



**18**

**dodecyl(phenyl)selane (18)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 100:1) as a colorless oil (42.4 mg, 0.130 mmol, 65%).

**$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.51 – 7.44 (m, 2H), 7.26 – 7.20 (m, 3H), 2.91 (t, *J* = 7.5 Hz, 2H), 1.73 – 1.67 (m, 2H), 1.42 – 1.36 (m, 2H), 1.31 – 1.25 (m, 16H), 0.88 (t, *J* = 6.9 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 132.5, 130.8, 129.1, 126.7, 32.1, 30.3, 30.0, 29.8, 29.8, 29.7, 29.6, 29.5, 29.2, 28.1, 22.8, 14.3. These data are in agreement with those reported previously in the literature.[11]




**19**

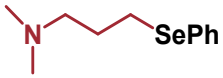
**isopentyl(phenyl)selane (19)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 100:1) as a colorless oil (26.9 mg, 0.118 mmol, 59%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.51 – 7.45 (m, 2H), 7.26 – 7.20 (m, 3H), 2.94 – 2.90 (m, 2H), 1.73 – 1.64 (m, 1H), 1.62 – 1.56 (m, 2H), 0.90 (d, *J* = 6.6 Hz, 6H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 132.4, 130.8, 129.1, 126.7, 39.2, 28.5, 26.0, 22.3. **IR (ATR)**: 2954, 1578, 1476, 1436, 1365, 1072, 730, 689 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+OH]^-$ calcd for $C_{11}H_{17}SeO^-$: 245.0450; found 245.0439.


**20**

**pentyl(phenyl)selane (20)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 100:1) as a colorless oil (22.8 mg, 0.10 mmol, 50%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.50 – 7.44 (m, 2H), 7.25 – 7.19 (m, 3H), 2.90 (t, *J* = 7.5 Hz, 2H), 1.73 – 1.67 (m, 2H), 1.41 – 1.26 (m, 4H), 0.87 (t, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 132.5, 130.8, 129.1, 126.7, 32.1, 30.0, 28.0, 22.3, 14.1. These data are in agreement with those reported previously in the literature.[12]




**21**

***N,N*-dimethyl-3-(phenylselanyl)propan-1-amine (21)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 10:1) as a colorless oil (34.0 mg, 0.14 mmol, 70%). **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.53 – 7.47 (m, 2H), 7.26 – 7.20 (m, 3H), 2.94 (t, *J* = 7.4 Hz, 2H), 2.36 (t, *J* = 7.1 Hz, 2H), 2.20 (s, 6H), 1.89 – 1.82 (m, 2H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 132.7, 130.5, 129.1, 126.9, 59.5, 45.6, 28.2, 25.7. These data are in agreement with those reported previously in the literature.[13]


**22**

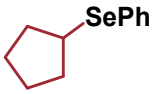
**methyl 3-(phenylselanyl)propanoate (22)**

Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (16.6 mg, 0.068 mmol, 34%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.56 – 7.49 (m, 2H), 7.28 – 7.26 (m, 3H), 3.67 (s, 3H), 3.10 (t, *J* = 7.4 Hz, 2H), 2.73 (t, *J* = 7.4 Hz, 2H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 172.8, 133.4, 129.3, 127.5, 51.9, 35.3, 21.9. These data are in agreement with those reported previously in the literature.[14]


**23**

**4-(phenylselanyl)butanenitrile (23)**

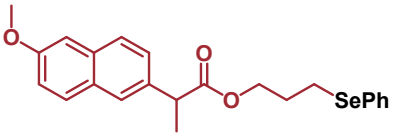
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless oil (28.8 mg, 0.128 mmol, 64%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.53 – 7.48 (m, 2H), 7.31 – 7.25 (m, 3H), 2.99 (t, *J* = 7.0 Hz, 2H), 2.50 (t, *J* = 7.1 Hz, 2H), 2.02 – 1.97 (m, 2H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 133.4, 129.4, 128.8, 127.6, 119.2, 26.2, 25.8, 17.1. These data are in agreement with those reported previously in the literature.[15]




**24**

**cyclopentyl(phenyl)selane (24)**

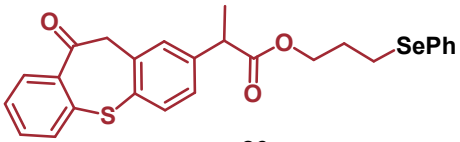
Following the general procedure A, the title product was obtained after purification by column chromatography (PE/EA = 100:1) as a colorless oil (18.1 mg, 0.08 mmol, 40%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.55 – 7.49 (m, 2H), 7.27 – 7.20 (m, 3H), 3.65 -3.60 (m, 1H), 2.13 – 1.98 (m, 2H), 1.82 – 1.63 (m, 4H), 1.61 - 1.55 (m, 2H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 133.6, 131.1, 129.0, 126.9, 41.9, 34.1, 24.9. These data are in agreement with those reported previously in the literature.[16]




**25**

**3-(phenylselanyl)propyl-2-(6-methoxynaphthalen-2-yl)propanoate (25)**

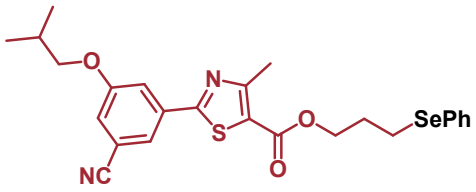
The title product was obtained after purification by column chromatography (PE/EA = 10:1) as a colorless oil (57.4 mg, 0.134 mmol, 67%). **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.72 – 7.62 (m, 3H), 7.42 – 7.32 (m, 3H), 7.22 – 7.06 (m, 5H), 4.17 – 4.13 (m, 2H), 3.89 (s, 3H), 3.83 (q, *J* = 7.2 Hz, 1H), 2.77 (t, *J* = 6.8 Hz, 2H), 1.97 – 1.86 (m, 2H), 1.56 (d, *J* = 7.1 Hz, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 174.7, 157.7, 135.8, 133.8, 132.7, 129.9, 129.4, 129.2, 129.0, 127.3, 127.0, 126.3, 126.0, 119.1, 105.7, 64.0, 55.5, 45.6, 29.2, 23.8, 18.5. **IR (ATR)**: 2972, 1727, 1604, 1505, 1478, 1071, 852, 734 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{23}H_{24}O_3SeNa^+$: 451.0783; found 451.0772.




**26**

**3-(phenylselanyl)propyl 2-(10-oxo-10,11-dihydrodibenzo[*b,f*]thiepin-2-yl)propanoate (26)**

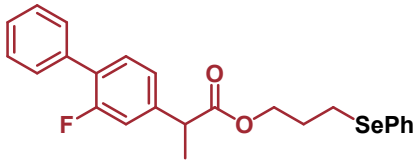
The title product was obtained after purification by column chromatography (PE/EA = 5:1) as a colorless oil (53.6 mg, 0.108 mmol, 54%). **$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.19 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.62 – 7.54 (m, 2H), 7.47 – 7.27 (m, 5H), 7.24 – 7.10 (m, 4H), 4.34 (s, 2H), 4.18 – 4.09 (m, 2H), 3.69 (q, *J* = 7.2 Hz, 1H), 2.80 (t, *J* = 7.2 Hz, 2H), 2.00 – 1.88 (m, 2H), 1.46 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 191.4, 173.9, 142.8, 140.3, 138.0, 136.2, 133.4, 132.8, 132.6, 131.7, 131.6, 131.0, 129.9, 129.2, 128.7, 127.1, 127.0, 126.4, 64.2, 51.2, 45.3, 29.2, 23.9, 18.5. **IR (ATR)**: 2973, 1729, 1671, 1586, 1475, 1427, 1071, 731, 690 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{26}H_{24}O_3SSeNa^+$: 519.0504; found 519.0491.




27

**3-(phenylselanyl)propyl 2-(3-cyano-5-isobutoxyphenyl)-4-methylthiazole-5-carboxylate (27)**

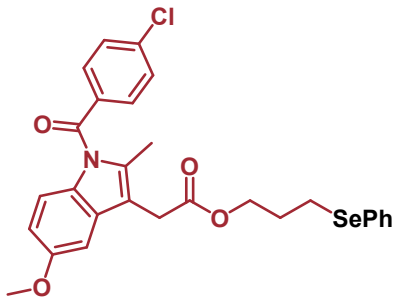
The title product was obtained after purification by column chromatography (PE/EA = 5:1) as white solid (50.4 mg, 0.098 mmol, 49%). **Melting Point:** 97 - 99 °C. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.17 (d, *J* = 2.3 Hz, 1H), 8.09 – 8.07 (m, 1H), 7.55 – 7.49 (m, 2H), 7.27 – 7.21 (m, 3H), 7.01 (d, *J* = 8.9 Hz, 1H), 4.38 (t, *J* = 6.1 Hz, 2H), 3.90 (d, *J* = 6.5 Hz, 2H), 3.02 (t, *J* = 7.2 Hz, 2H), 2.74 (s, 3H), 2.24 – 2.15 (m, 1H), 2.17 – 2.07 (m, 2H), 1.09 (d, *J* = 6.7 Hz, 6H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 167.4, 162.6, 162.0, 161.4, 133.0, 132.7, 132.2, 129.8, 129.3, 127.2, 126.0, 121.6, 115.5, 112.7, 103.1, 75.8, 64.6, 29.4, 28.2, 24.1, 19.2, 17.6. **IR (ATR)**: 2957, 2227, 1686, 1601, 1507, 1431, 1044, 736, 691 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{25}H_{26}N_2O_3SSeNa^+$: 537.0722; found 537.0717.




28

**3-(phenylselanyl)propyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (28)**

The title product was obtained after purification by column chromatography (PE/EA = 10:1) as colorless oil (52.2 mg, 0.118 mmol, 59%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.54 – 7.50 (m, 2H), 7.47 – 7.33 (m, 6H), 7.24 – 7.19 (m, 3H), 7.16 – 7.07 (m, 2H), 4.18 (t, *J* = 6.2 Hz, 2H), 3.73 (q, *J* = 7.2 Hz, 1H), 2.84 (t, *J* = 7.3 Hz, 2H), 2.03 – 1.92 (m, 2H), 1.52 (d, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 174.0, 159.8 (d, *J* = 249.7 Hz), 142.0 (d, *J* = 7.7 Hz), 135.6, 132.8, 131.0 (d, *J* = 4.1 Hz), 129.8, 129.2, 129.1 (d, *J* = 2.9 Hz), 128.6, 128.0 (d, *J* = 13.7 Hz), 127.8, 127.1, 123.6 (d, *J* = 3.4 Hz), 115.3 (d, *J* = 23.8 Hz), 64.3, 45.1, 29.2, 23.8, 18.4. **$^{19}$F NMR (471 MHz, $CDCl_3$)** δ -117.40 (t, *J* = 9.5 Hz). **IR (ATR)**: 2975, 1730, 1579, 1478, 1170, 1072, 873, 765 $cm^{-1}$ **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{24}H_{23}FO_2SeNa^+$: 465.0740; found 465.0730.




29

**3-(phenylselanyl)propyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate (29)**

The title product was obtained after purification by column chromatography (PE/EA = 5:1) as a yellow oil (62.2 mg, 0.112 mmol, 56%). **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.69 – 7.60 (m, 2H), 7.48 – 7.38 (m, 4H), 7.24 – 7.17 (m, 3H), 6.95 (d, *J* = 2.5 Hz, 1H), 6.86 (dd, *J* = 9.0, 0.5 Hz, 1H), 6.66 (dd, *J* = 9.0, 2.5 Hz, 1H), 4.18 (t, *J* = 6.2 Hz, 2H), 3.81 (s, 3H), 3.64 (s, 2H), 2.84 (t, *J* = 7.3 Hz, 2H), 2.37 (s, 3H), 2.03 – 1.94 (m, 2H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 170.9, 168.4, 156.2, 139.4, 136.0, 134.0, 132.8, 131.3, 130.9, 130.7, 129.8, 129.2, 127.1, 115.1, 112.6, 111.8, 101.4, 64.3, 55.8, 30.5, 29.2, 23.9, 13.5. **IR (ATR)**: 2929, 1731, 1678, 1589, 1475, 1355, 1087, 831, 733 $cm^{-1}$. **HRMS (ESI):** *m/z* $[M+Na]^+$ calcd for $C_{28}H_{26}ClNO_4SeNa^+$: 578.0608; found 578.0602.

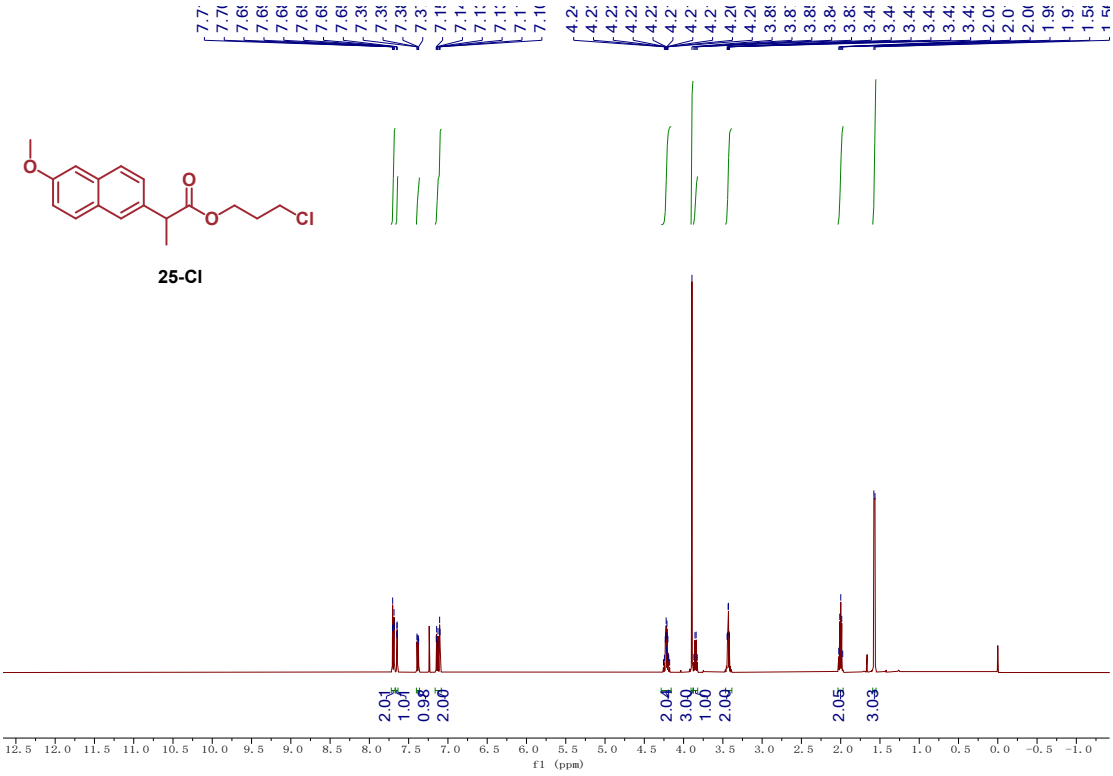

**31**

**hex-5-en-1-yl(phenyl)selane (31)**

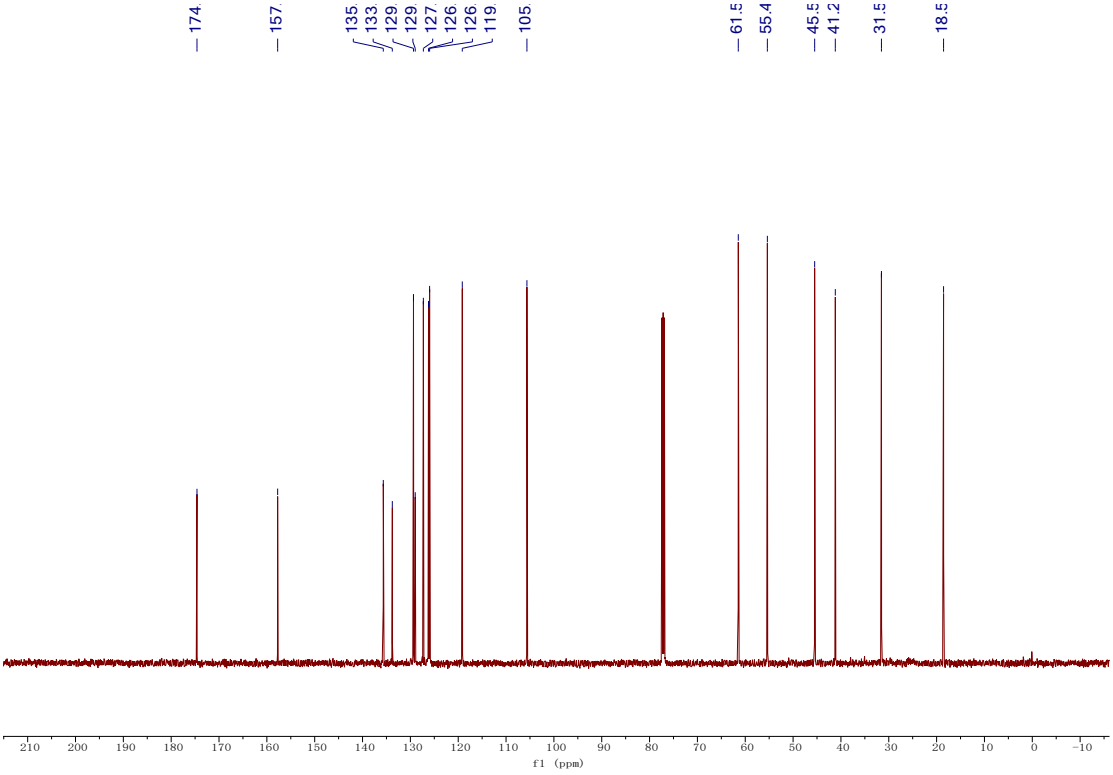
Following the procedure C, using alkyl chloride **30** (Scheme S4B), the title product was obtained after purification by column chromatography (PE/EA = 100:1) as a colorless oil (24.5 mg, 0.102 mmol, 51%). **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.56 – 7.40 (m, 2H), 7.27 – 7.21 (m, 3H), 5.81 - 5.73 (m, 1H), 5.07 – 4.87 (m, 2H), 2.90 (t, *J* = 7.5 Hz, 2H), 2.13 – 1.97 (m, 2H), 1.75 – 1.69 (m, 2H), 1.56 – 1.46 (m, 2H). **$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 138.5, 132.5, 130.7, 129.1, 126.7, 114.8, 33.3, 29.7, 29.1, 27.8. These data are in agreement with those reported previously in the literature.[17]

# 6. NMR spectra

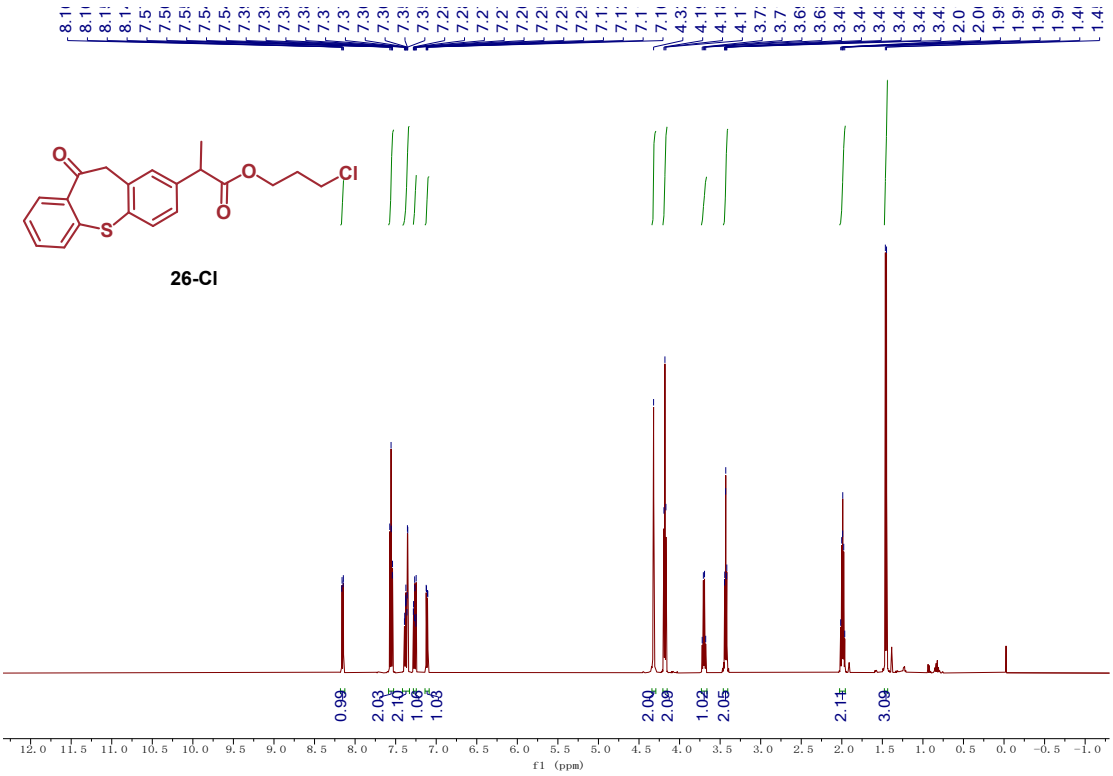
## $^{1}$H NMR of 25-Cl (500 MHz, $CDCl_3$)



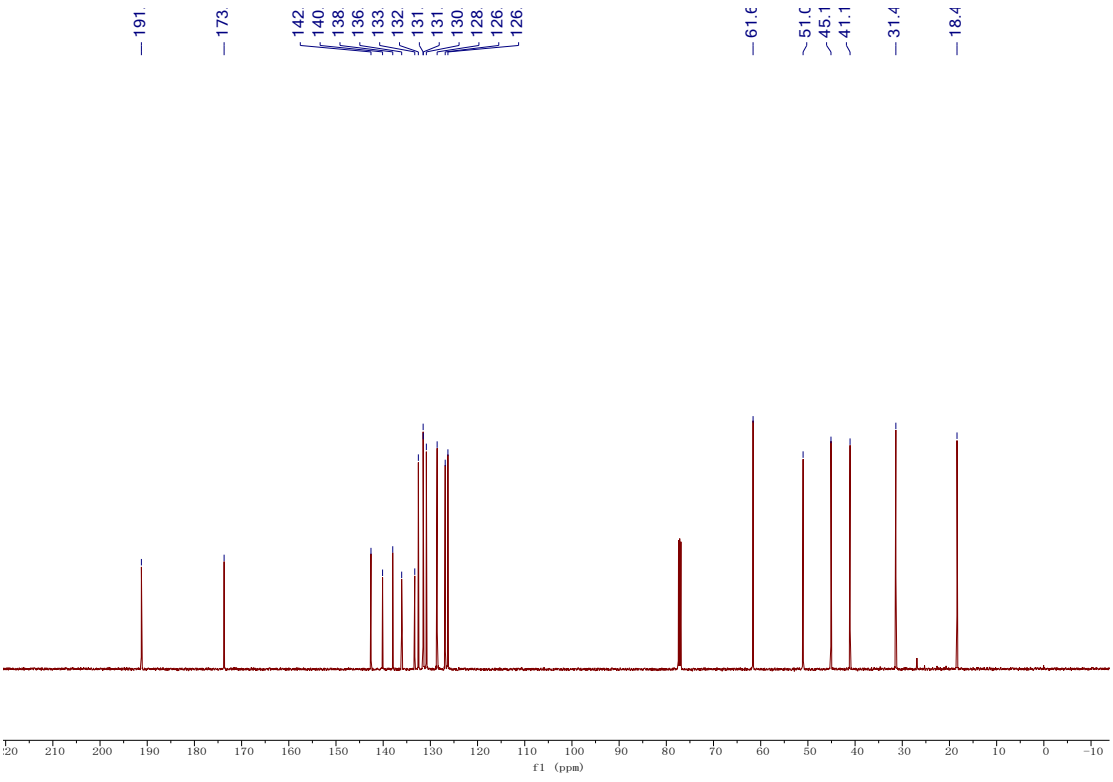

## $^{13}$C NMR of 25-Cl (126 MHz, $CDCl_3$)



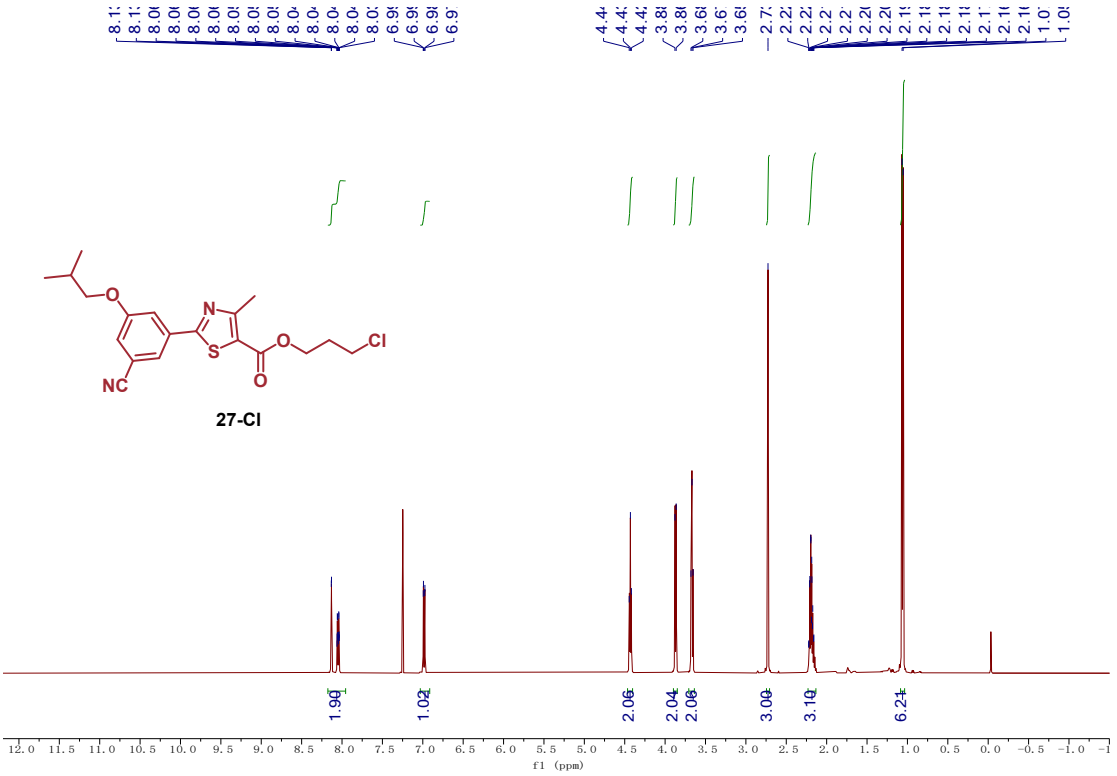
**$^{1}$H NMR of 26-Cl (500 MHz, $CDCl_3$)**



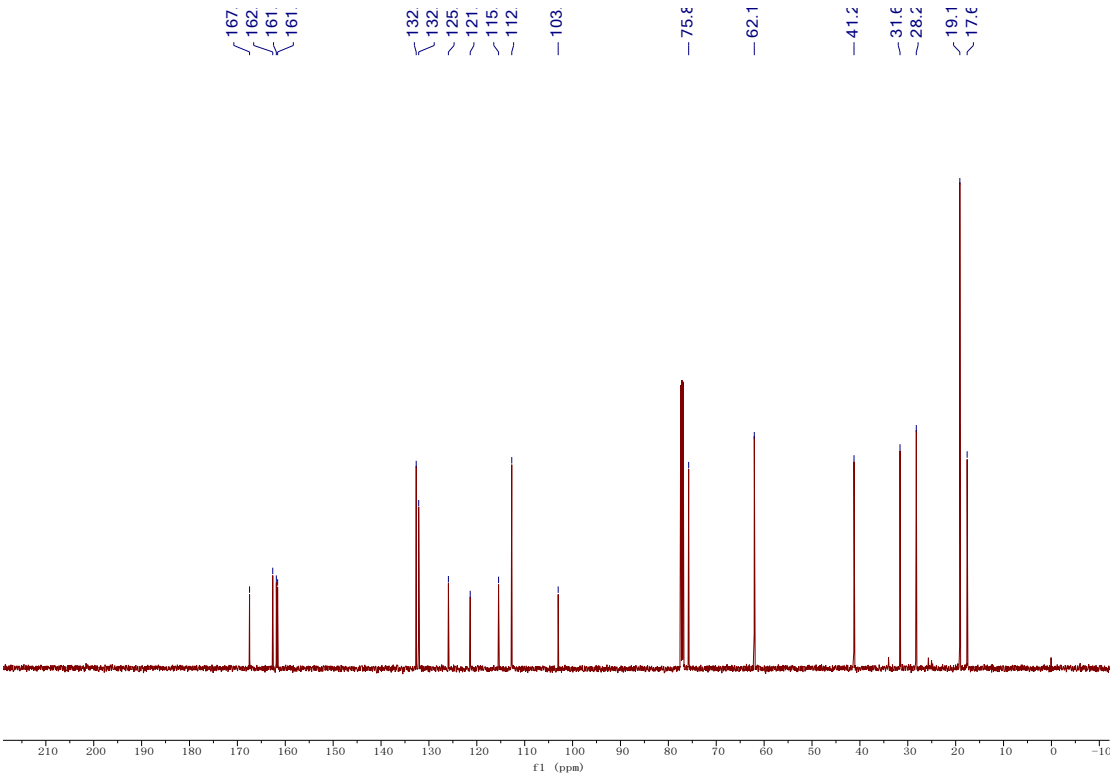

**$^{13}$C NMR of 26-Cl (126 MHz, $CDCl_3$)**



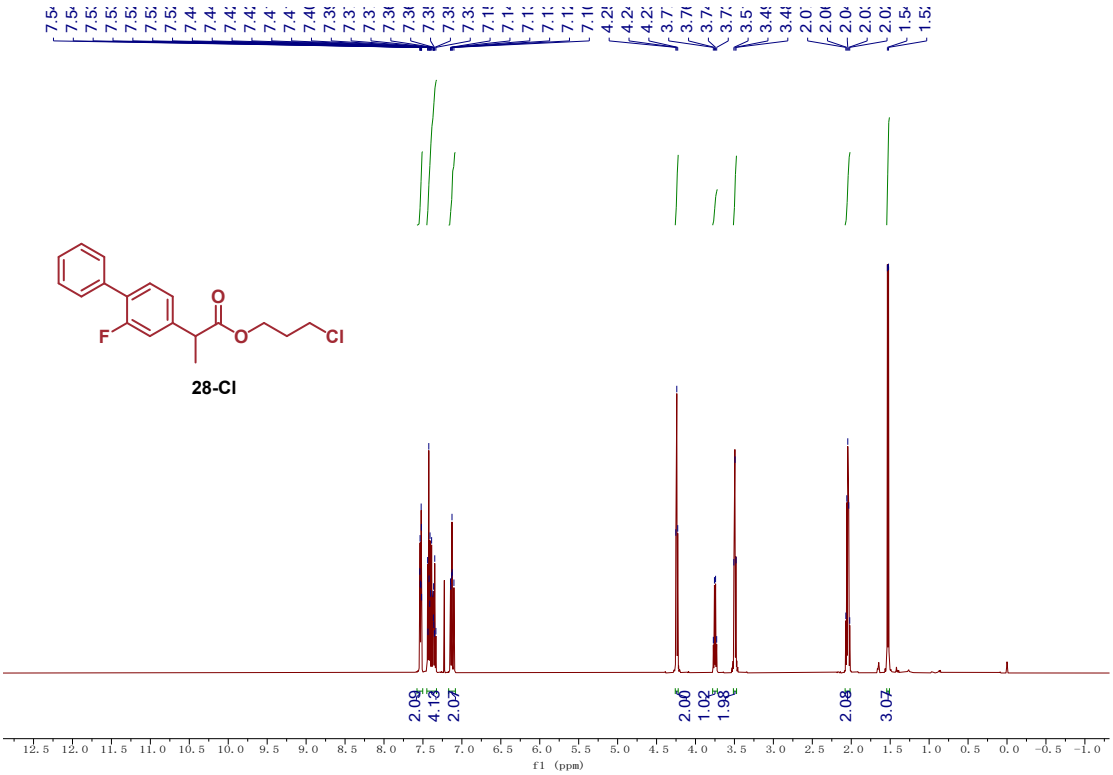
**$^{1}$H NMR of 27-Cl (500 MHz, $CDCl_3$)**



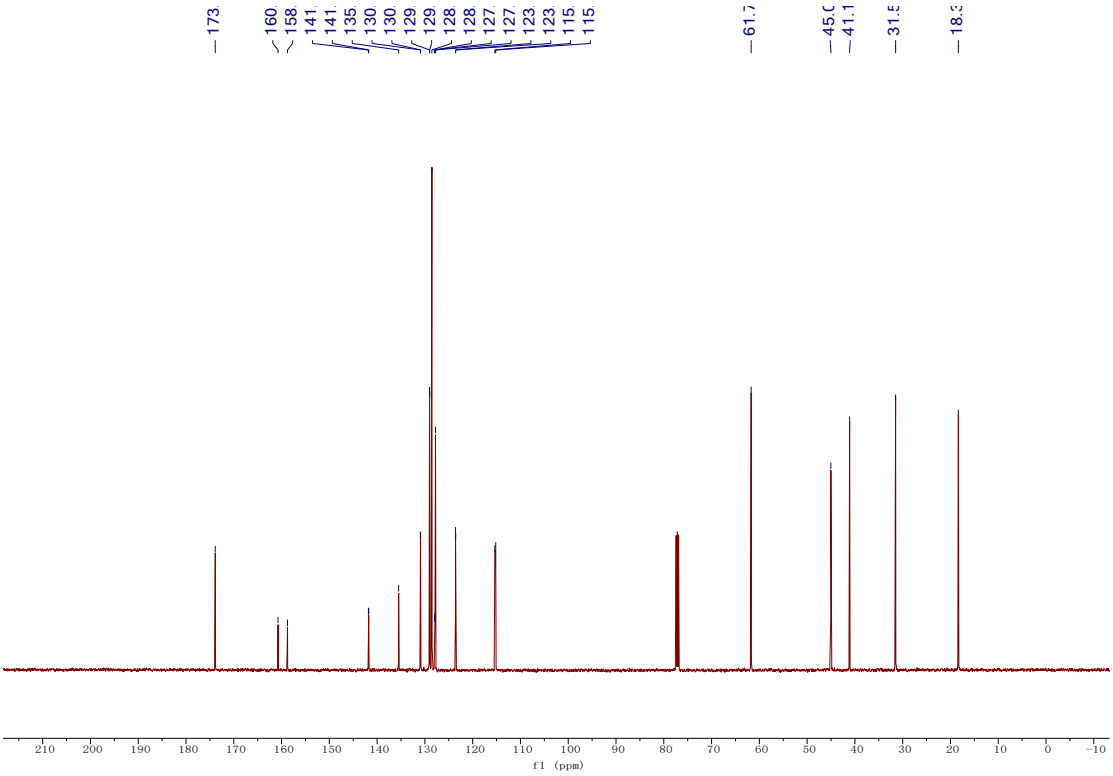

**$^{13}$C NMR of 27-Cl (126 MHz, $CDCl_3$)**



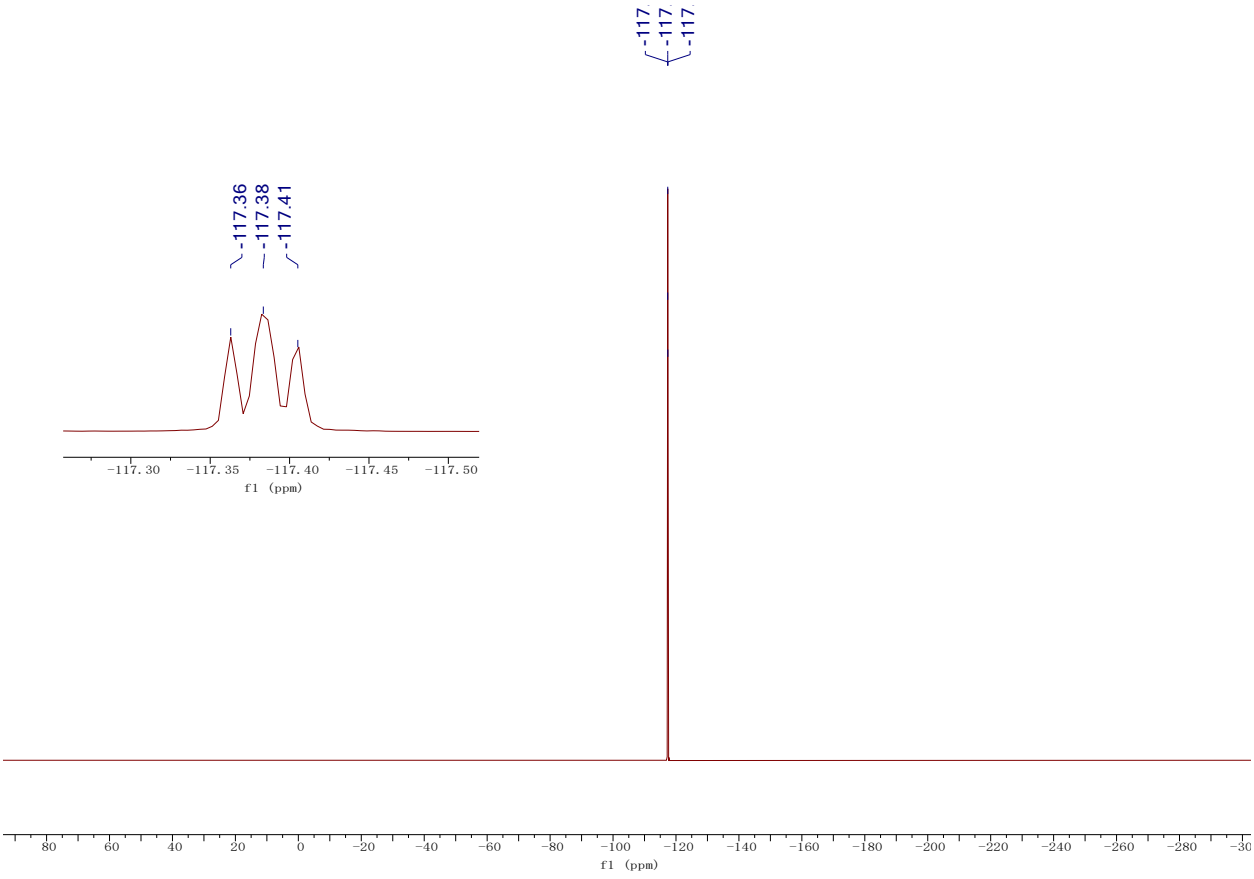
**$^{1}$H NMR of 28-Cl (500 MHz, $CDCl_3$)**



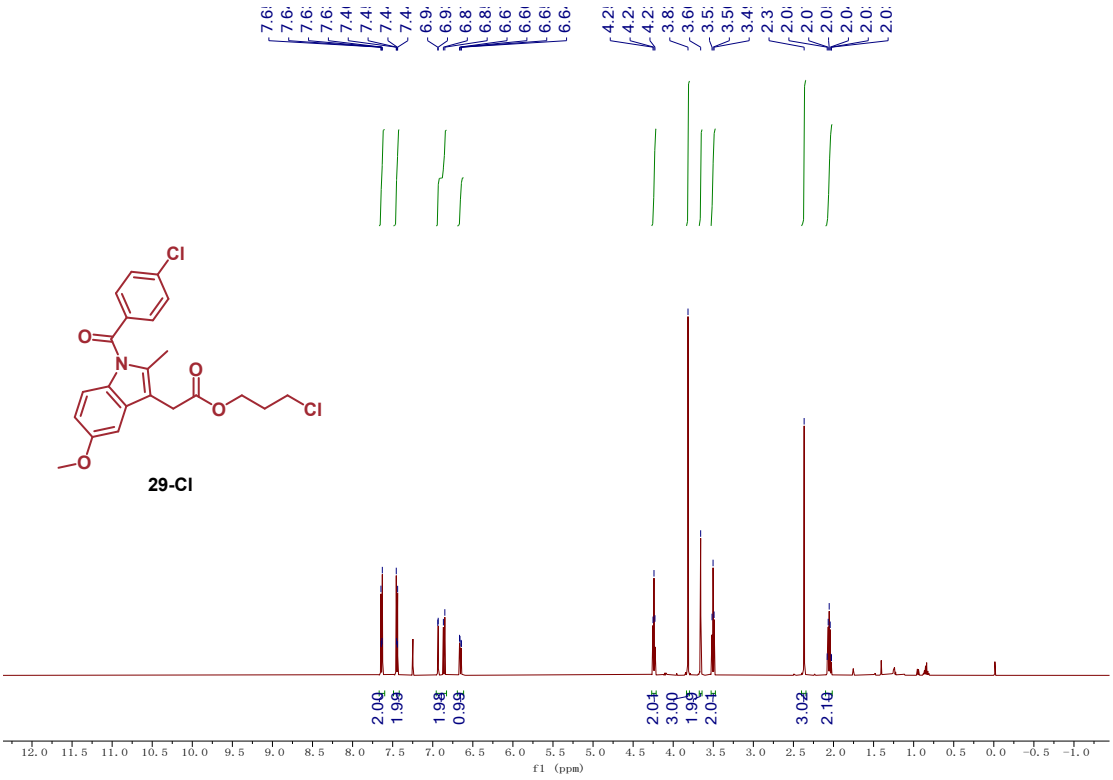

**$^{13}$C NMR of 28-Cl (126 MHz, $CDCl_3$)**



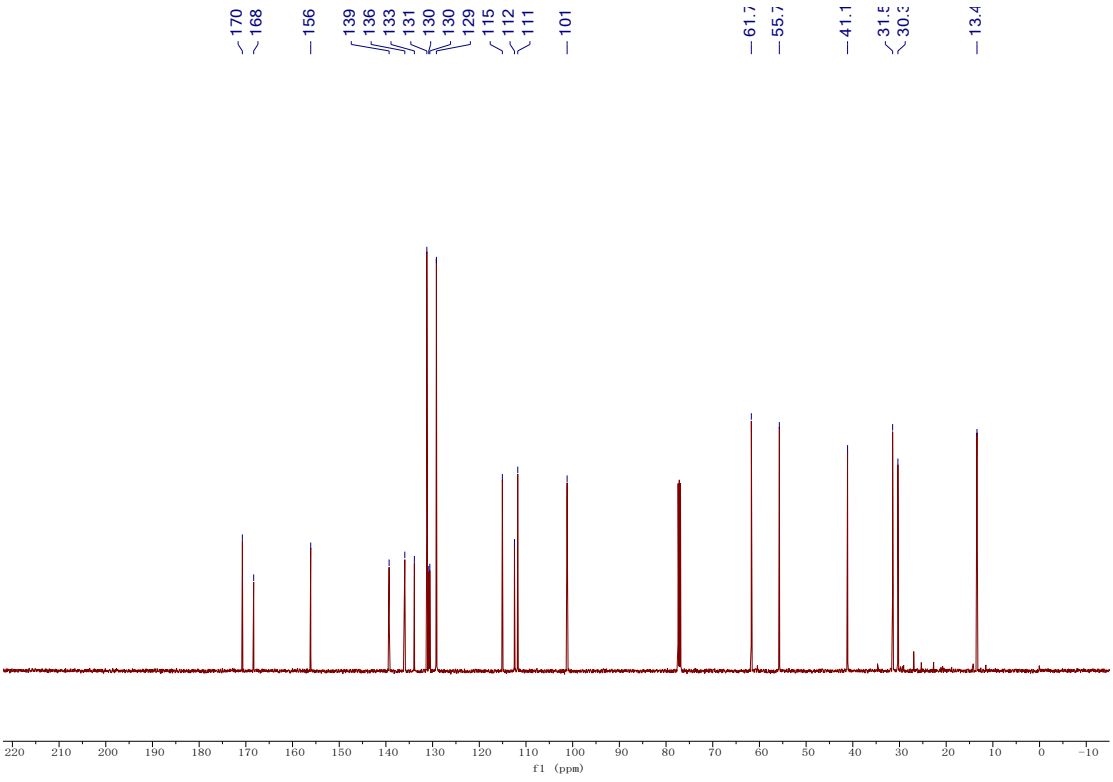

**$^{19}$F NMR of 28-Cl (471 MHz, $CDCl_3$)**


-117
-117
-117
-117.36
-117.38
-117.41
−117.30
−117.35
−117.40
−117.45
−117.50
f1 (ppm)
80
60
40
20
0
−20
−40
−60
−80
−100
−120
−140
−160
−180
−200
−220
−240
−260
−280
−30
f1 (ppm)

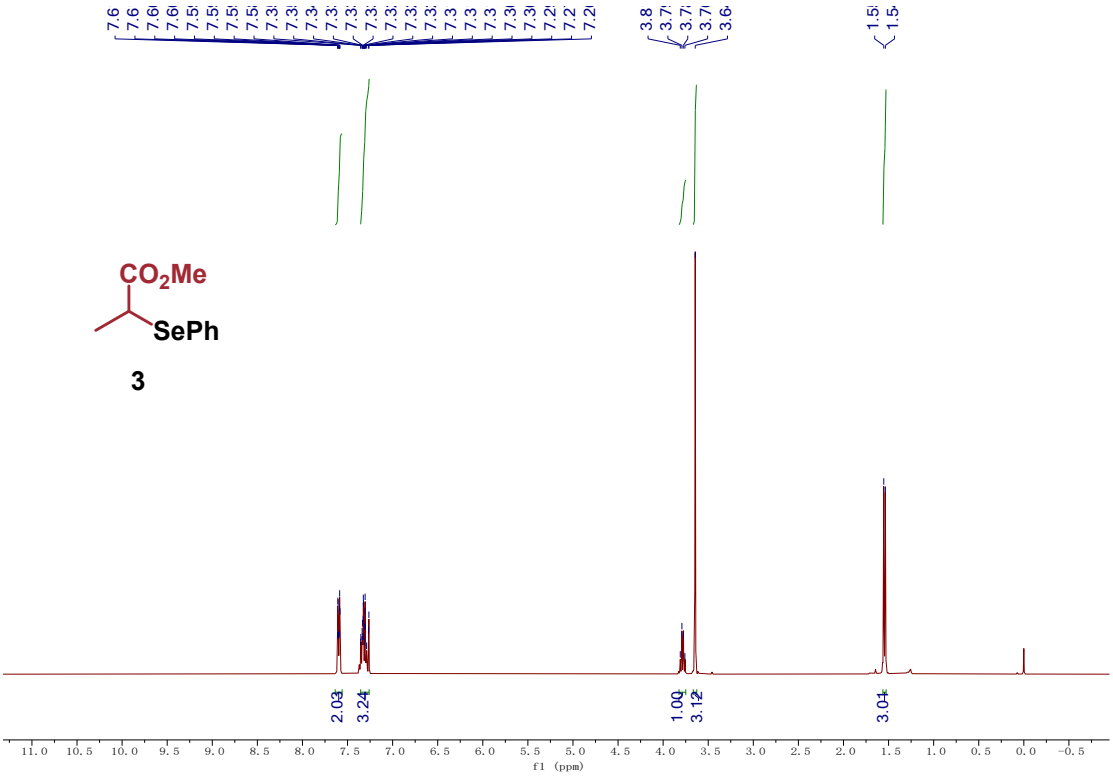
**$^{1}$H NMR of 29-Cl (500 MHz, $CDCl_3$)**



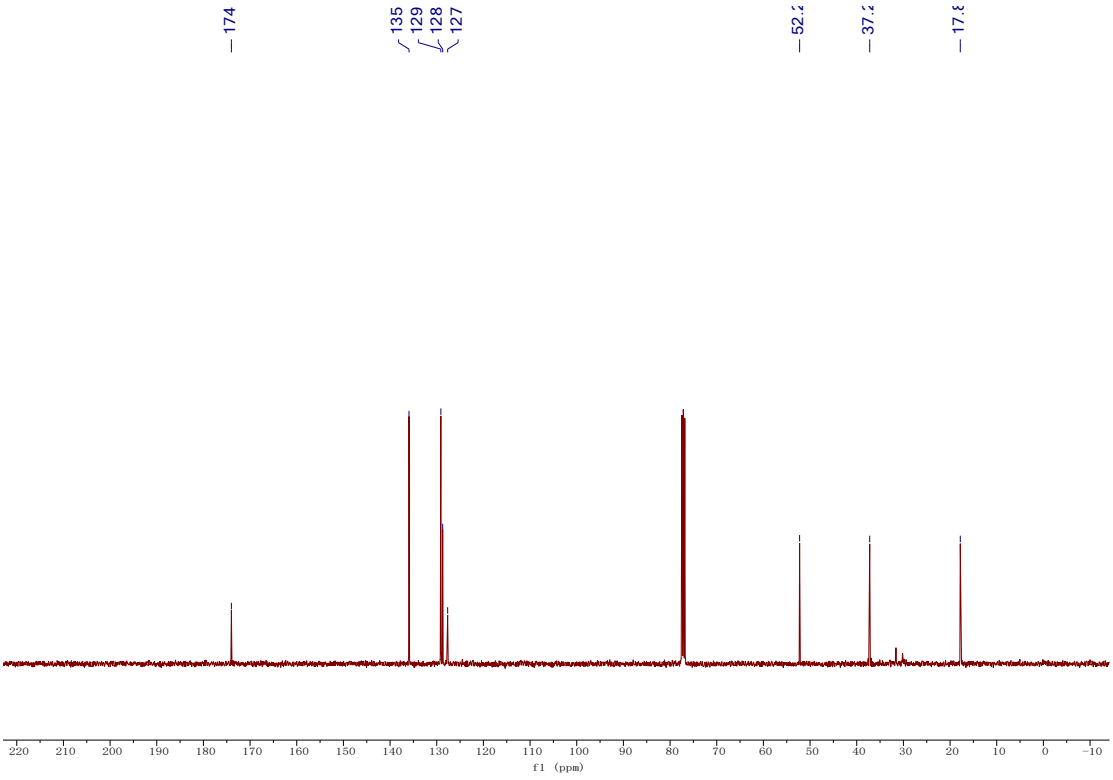

**$^{13}$C NMR of 29-Cl (126 MHz, $CDCl_3$)**



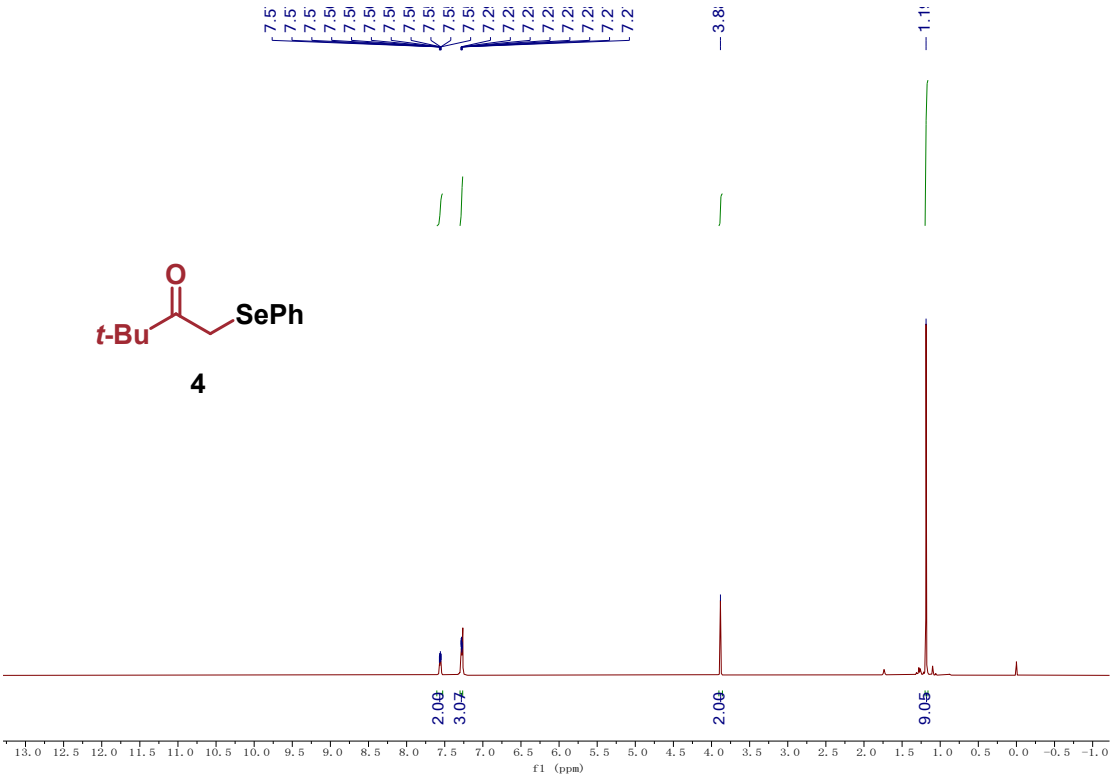
**$^{1}$H NMR of 3 (400 MHz, $CDCl_3$)**




**$^{13}$C NMR of 3 (101 MHz, $CDCl_3$)**

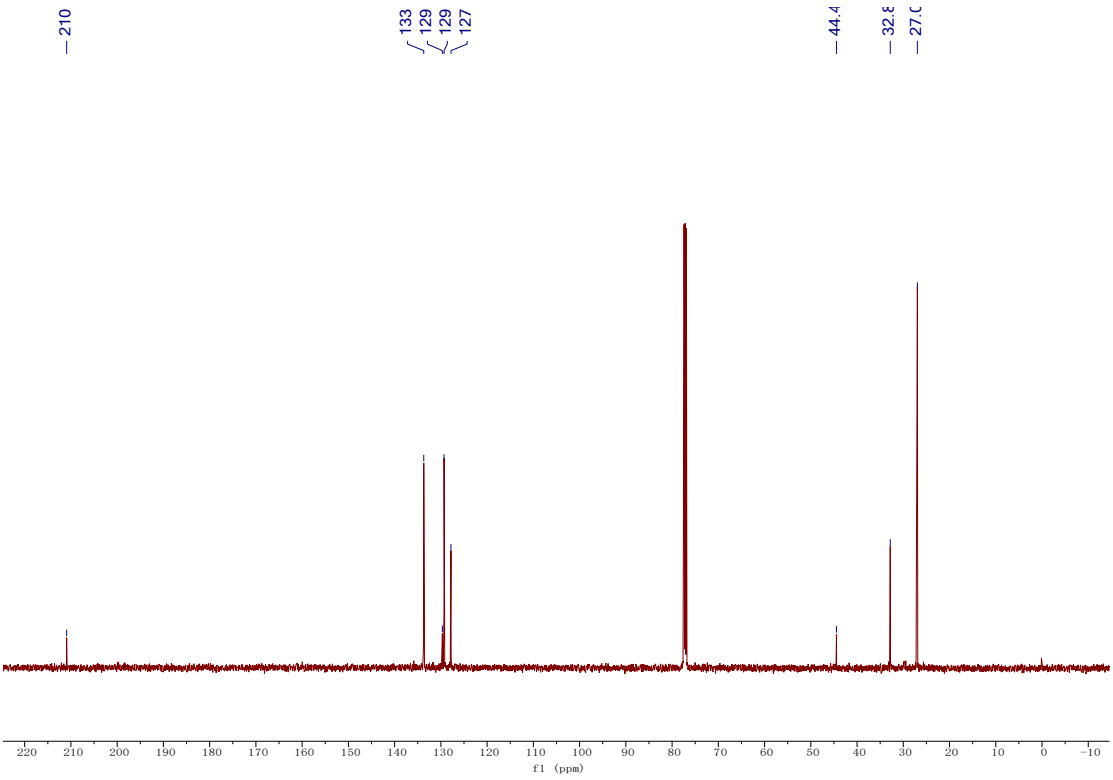


**$^{1}$H NMR of 4 (500 MHz, $CDCl_3$)**

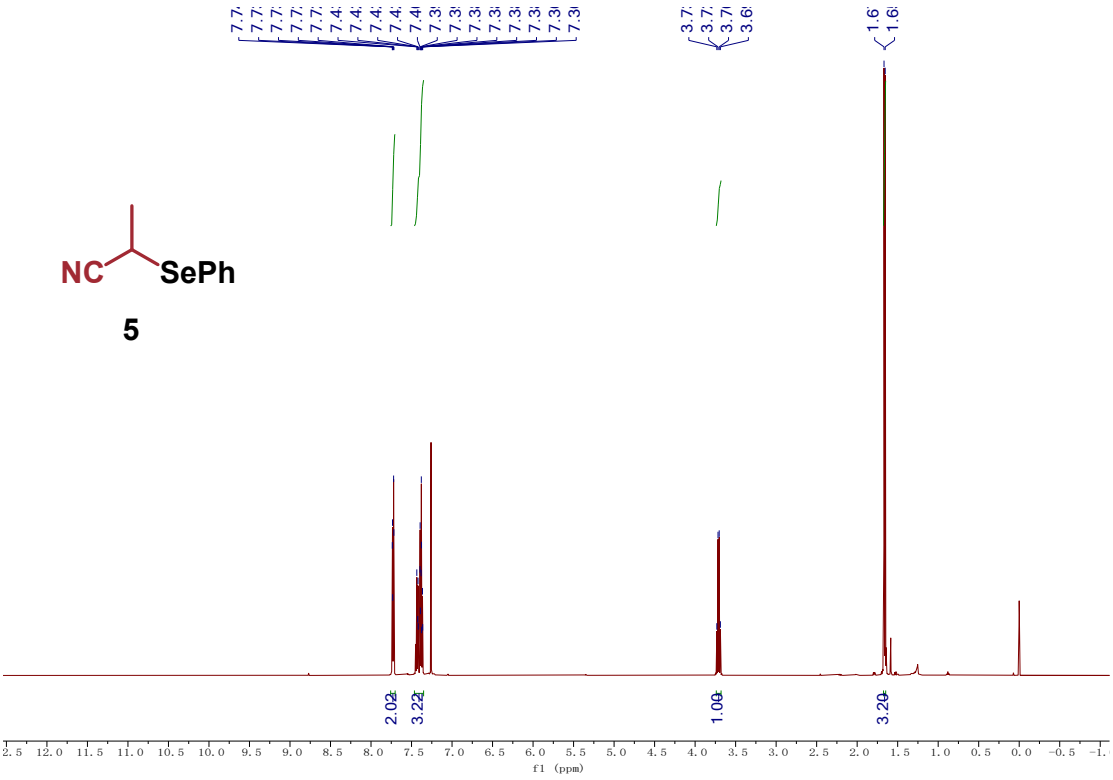



4

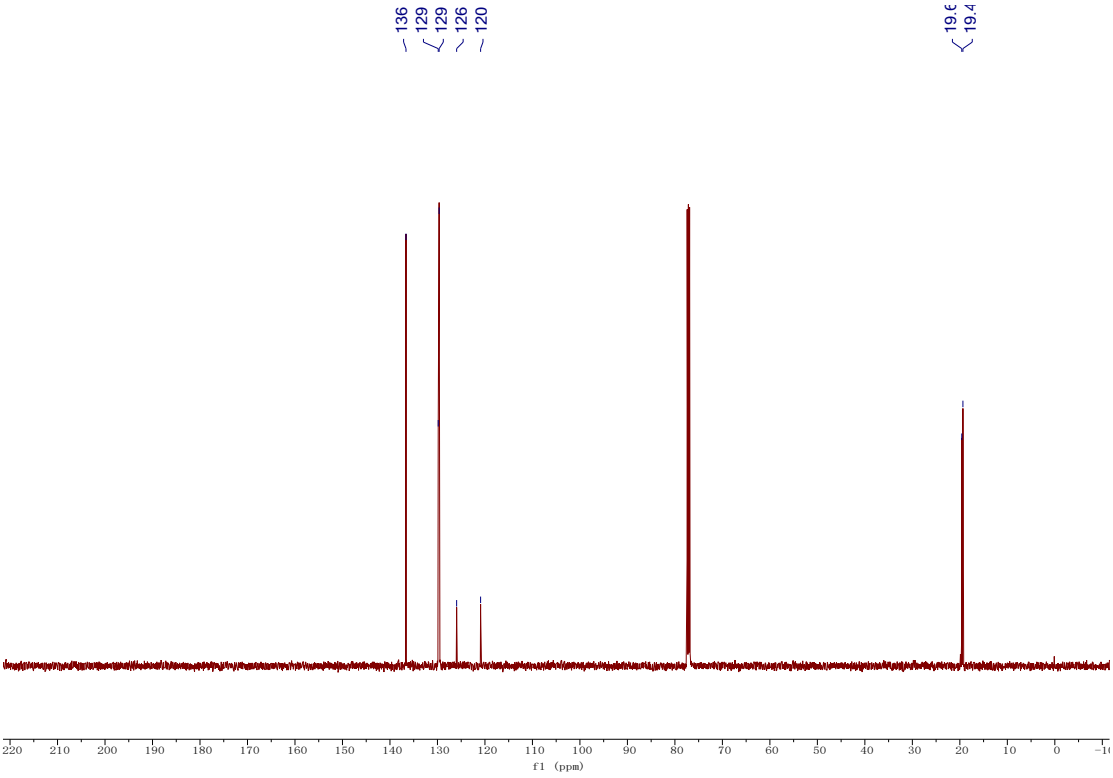
**$^{13}$C NMR of 4 (126 MHz, $CDCl_3$)**



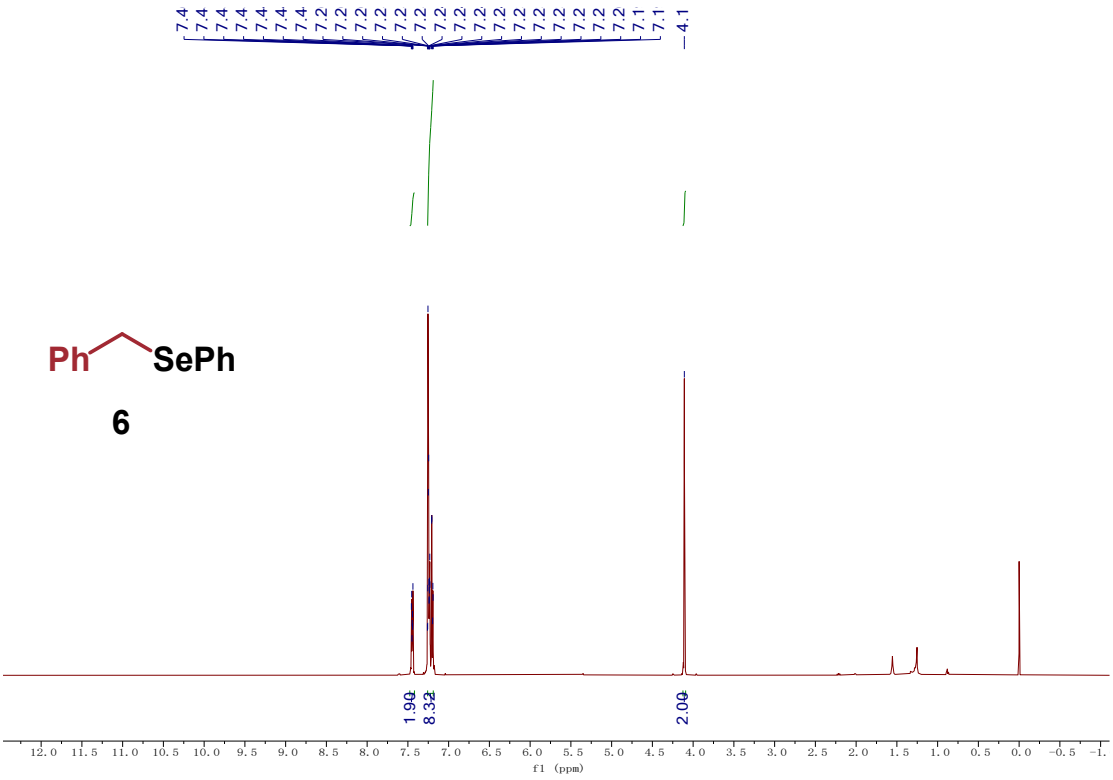
**$^{1}$H NMR of 5 (500 MHz, $CDCl_3$)**



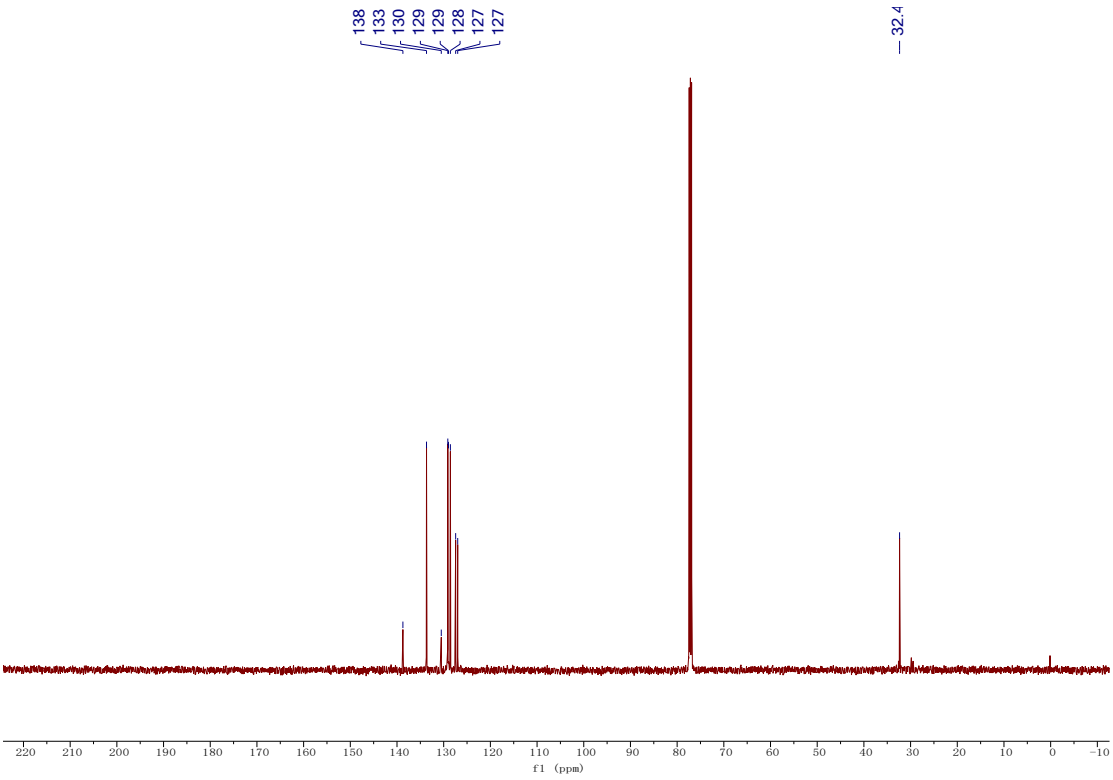

**$^{13}$C NMR of 5 (126 MHz, $CDCl_3$)**



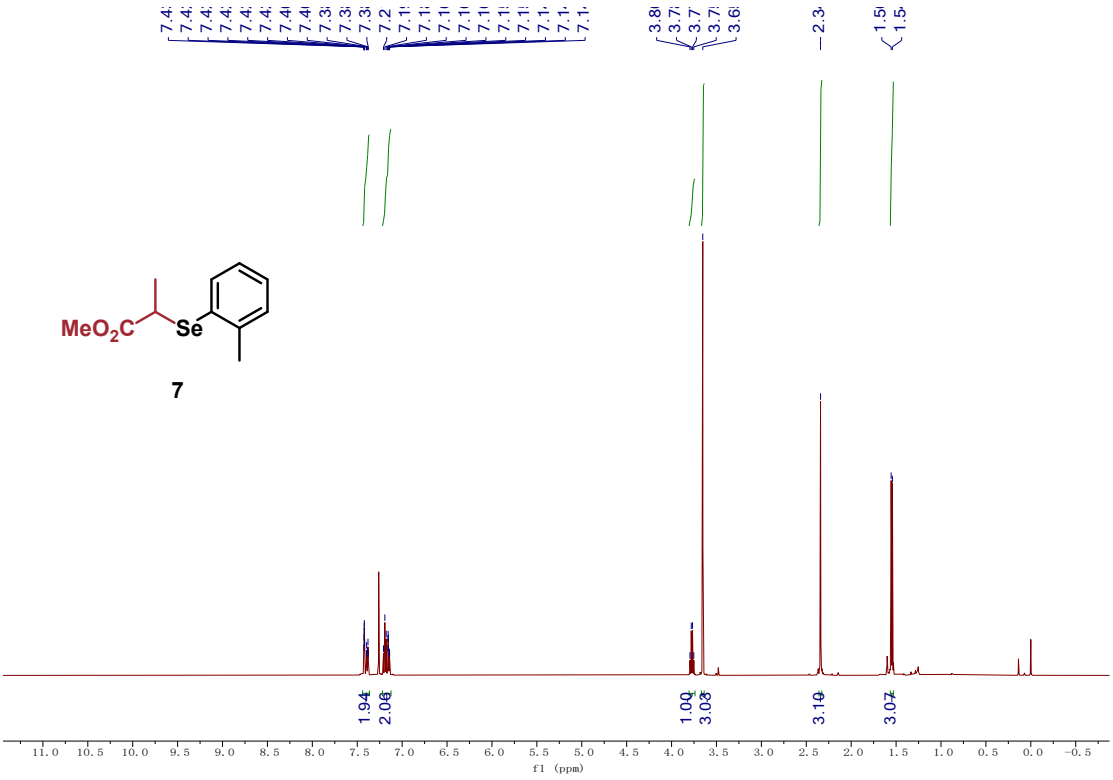
**$^{1}$H NMR of 6 (500 MHz, $CDCl_3$)**



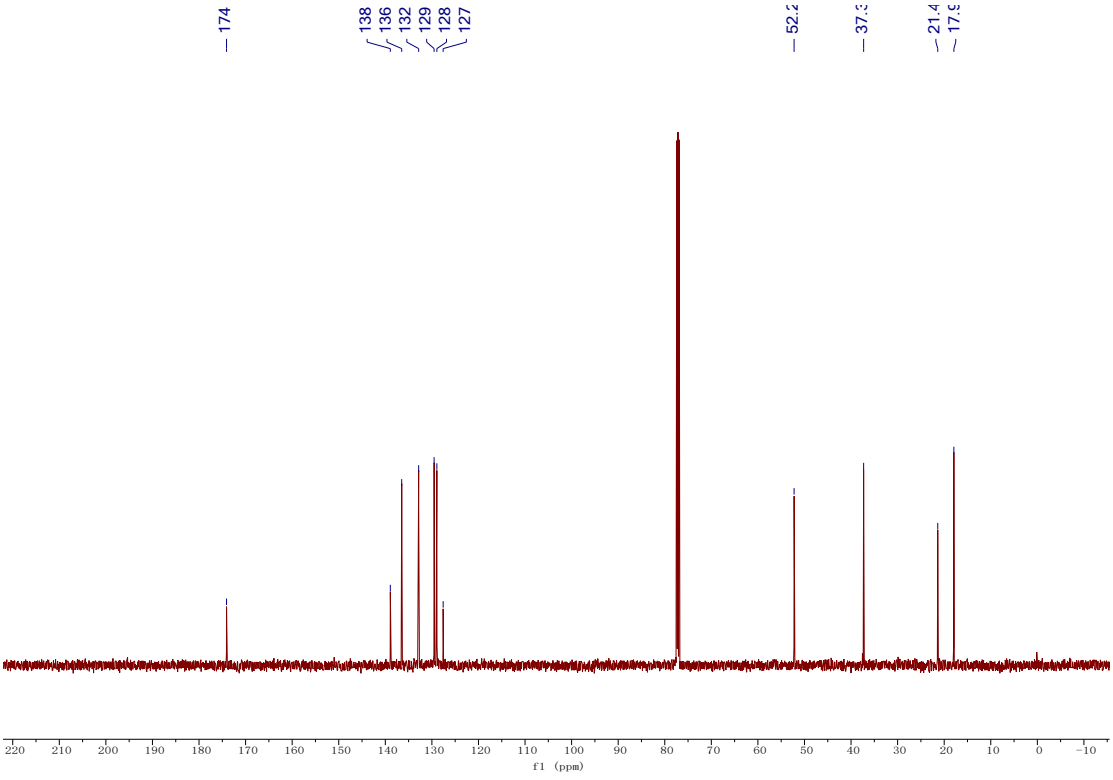

**$^{13}$C NMR of 6 (126 MHz, $CDCl_3$)**



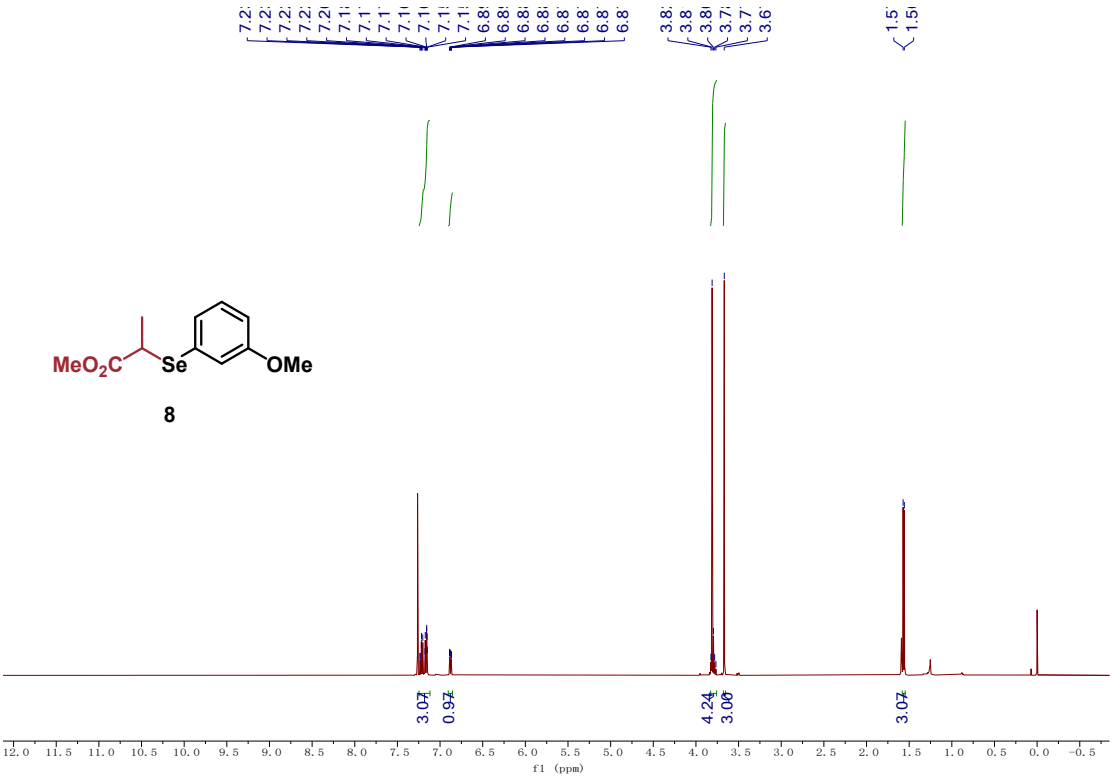
**$^{1}$H NMR of 7 (500 MHz, $CDCl_3$)**



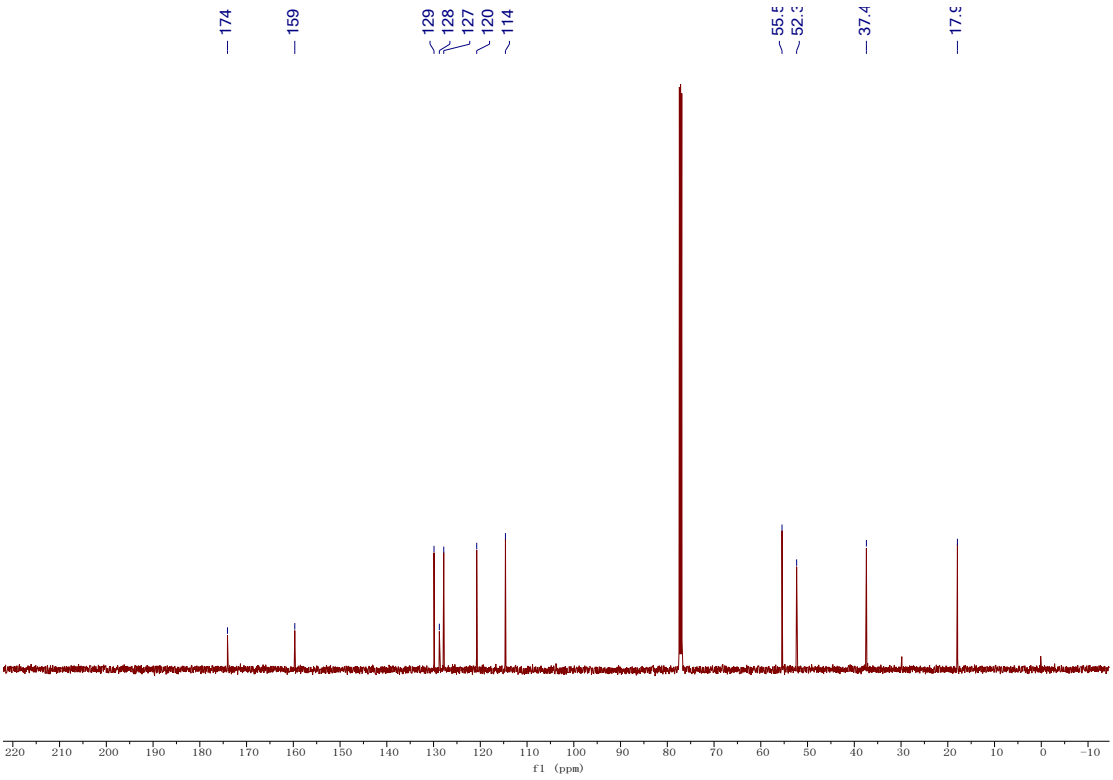

**$^{13}$C NMR of 7 (126 MHz, $CDCl_3$)**



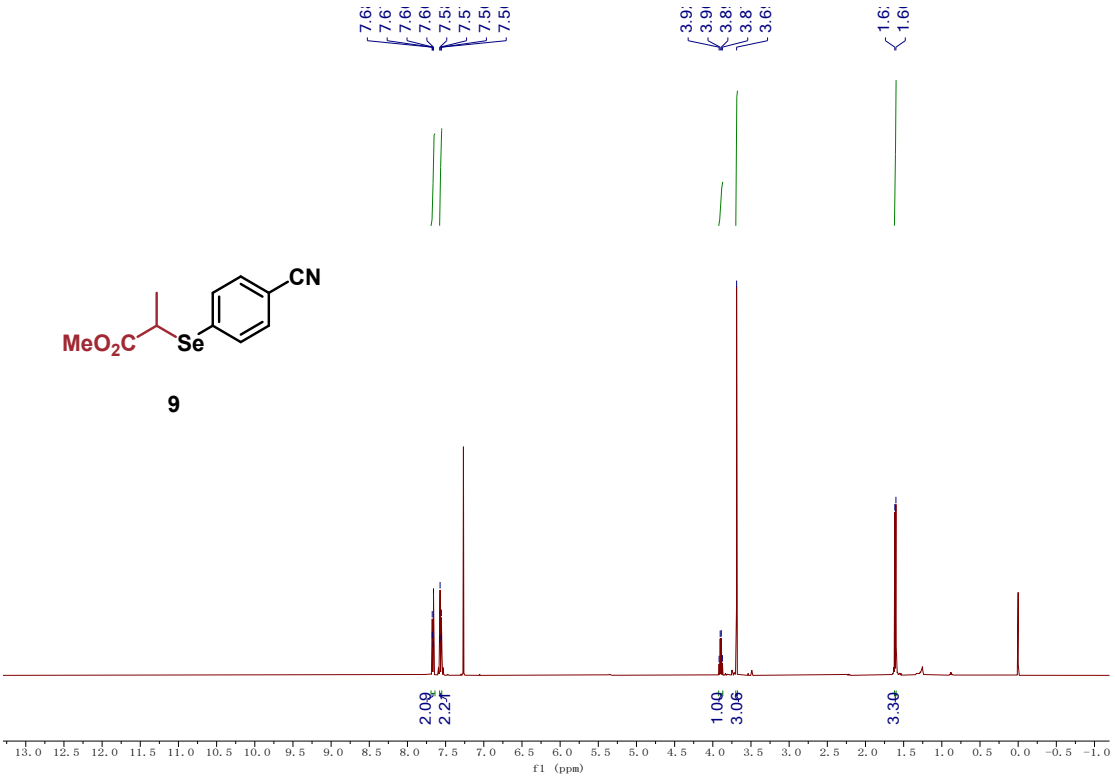
**$^{1}$H NMR of 8 (500 MHz, $CDCl_3$)**



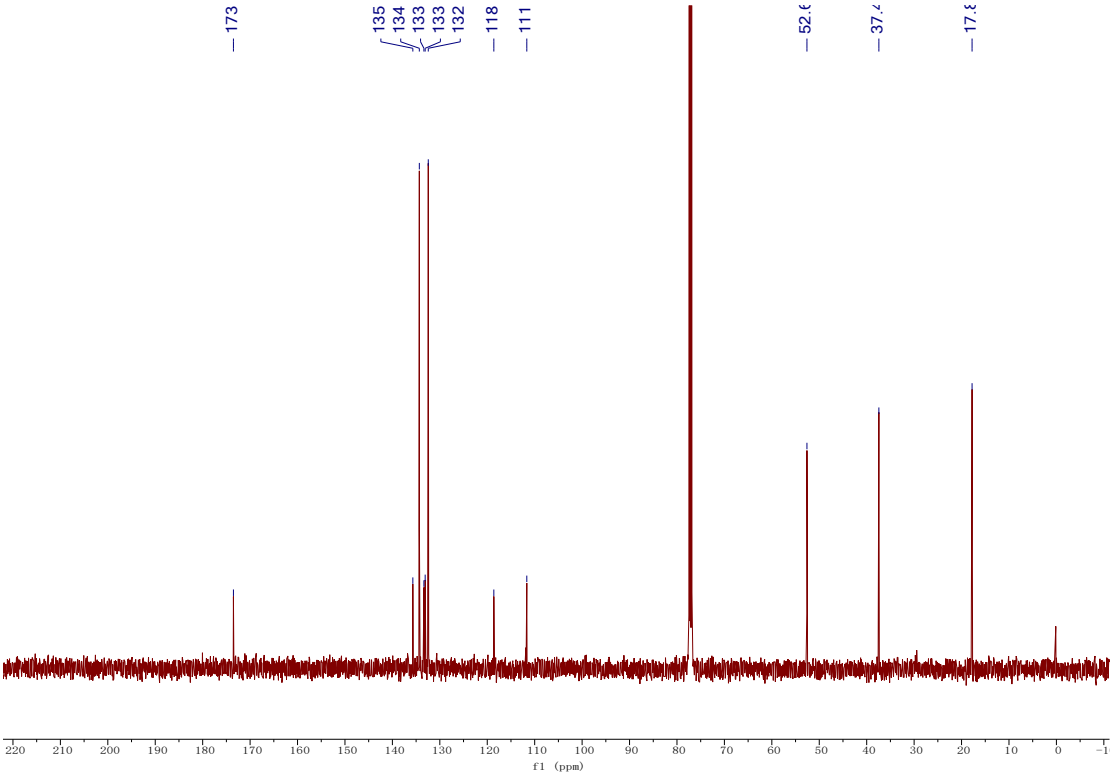

**$^{13}$C NMR of 8 (126 MHz, $CDCl_3$)**



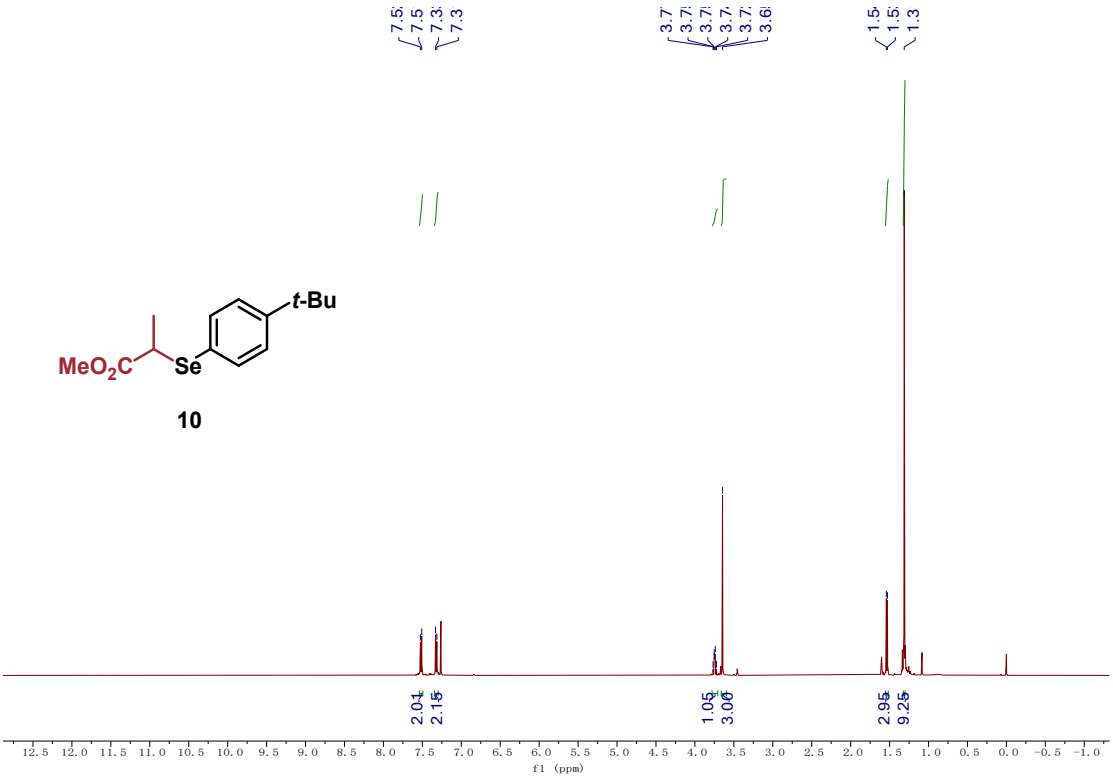
**$^{1}$H NMR of 9 (500 MHz, $CDCl_3$)**




**$^{13}$C NMR of 9 (126 MHz, $CDCl_3$)**

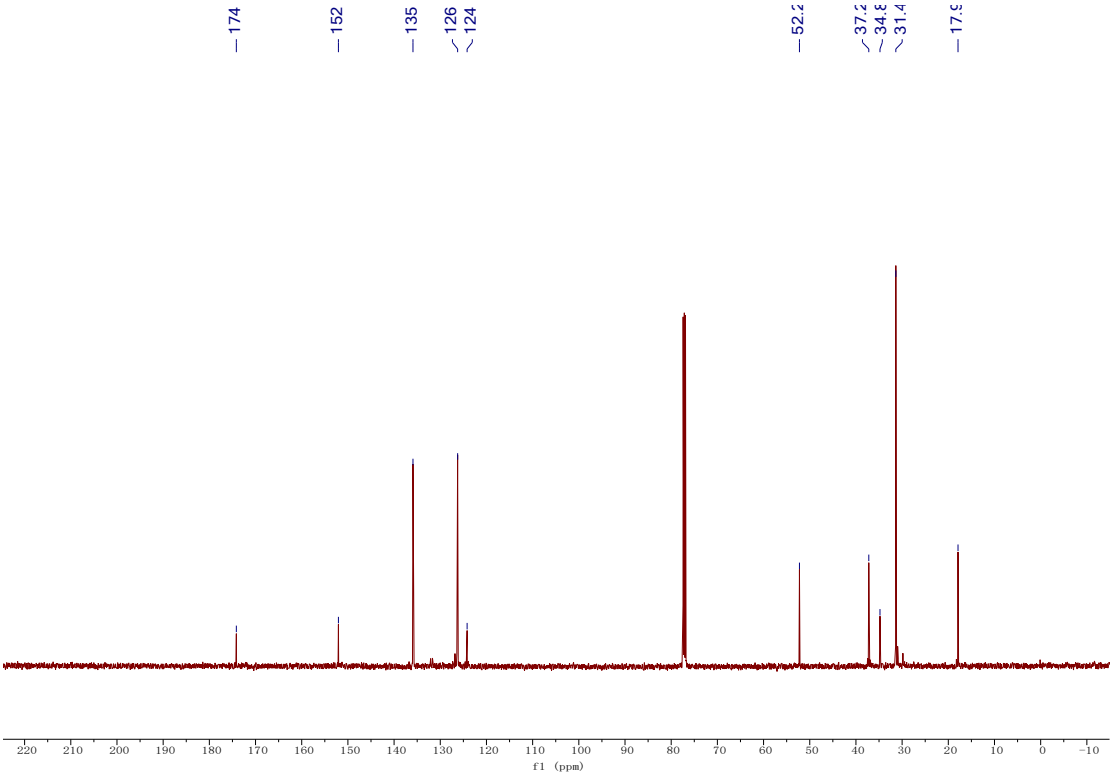


**$^{1}$H NMR of 10 (500 MHz, $CDCl_3$)**

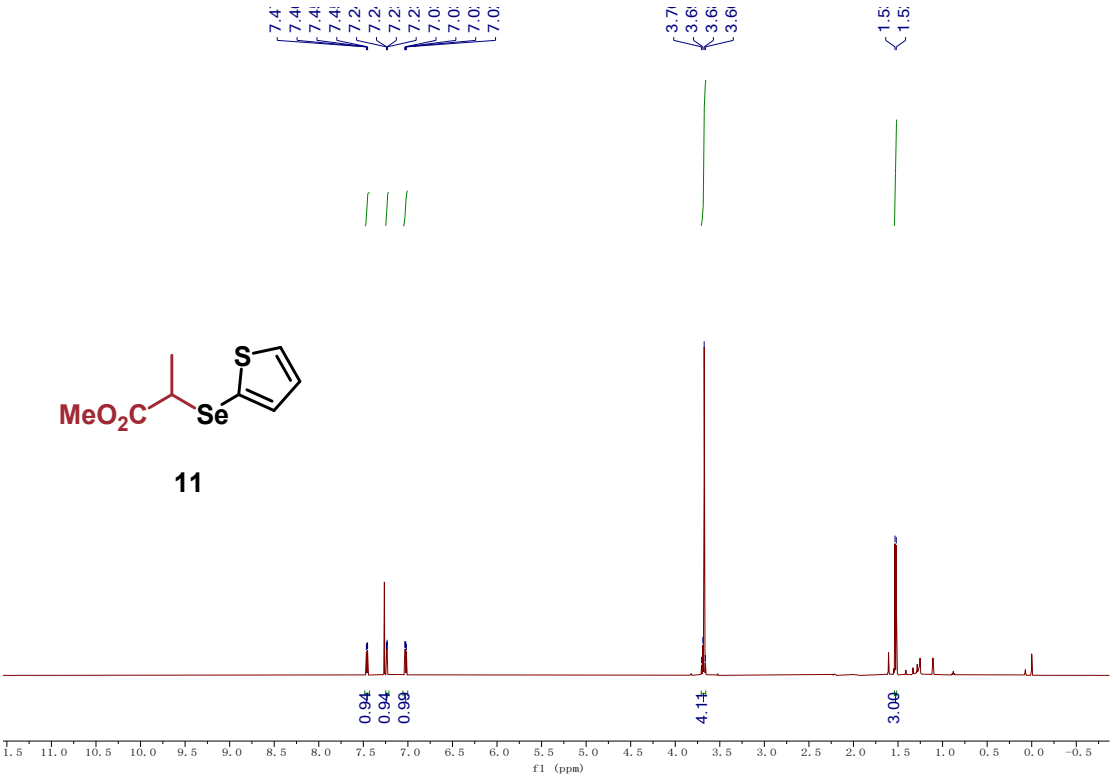



10

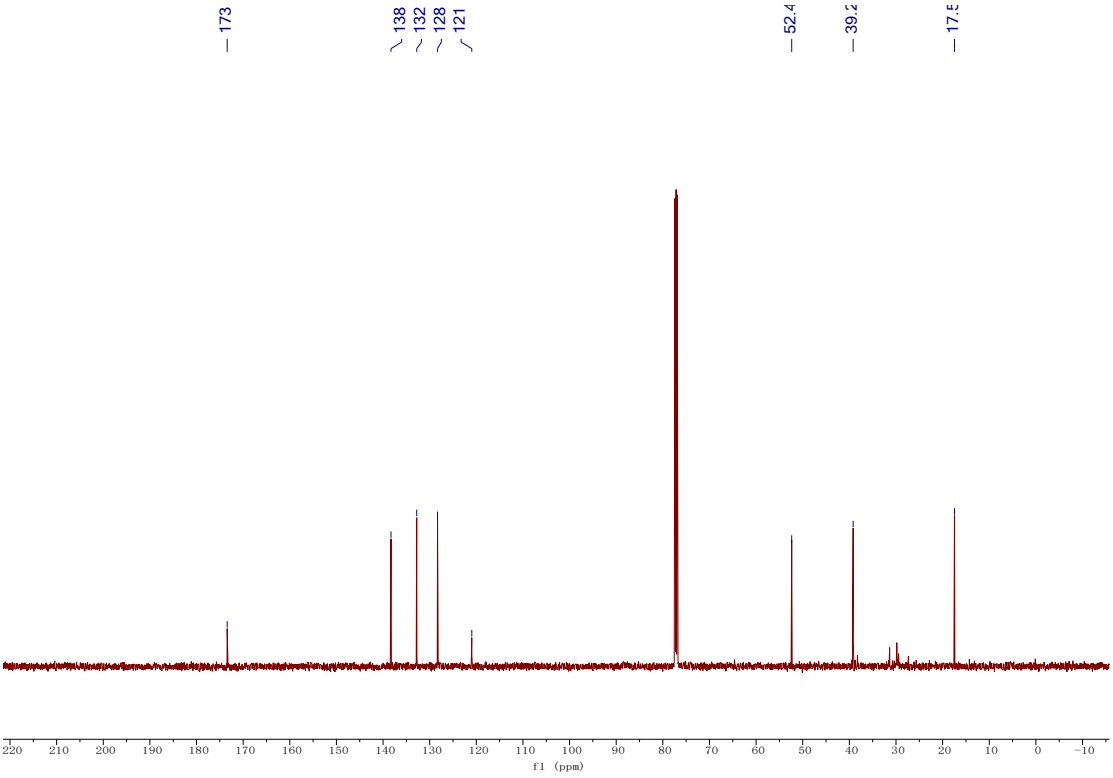
**$^{13}$C NMR of 10 (126 MHz, $CDCl_3$)**



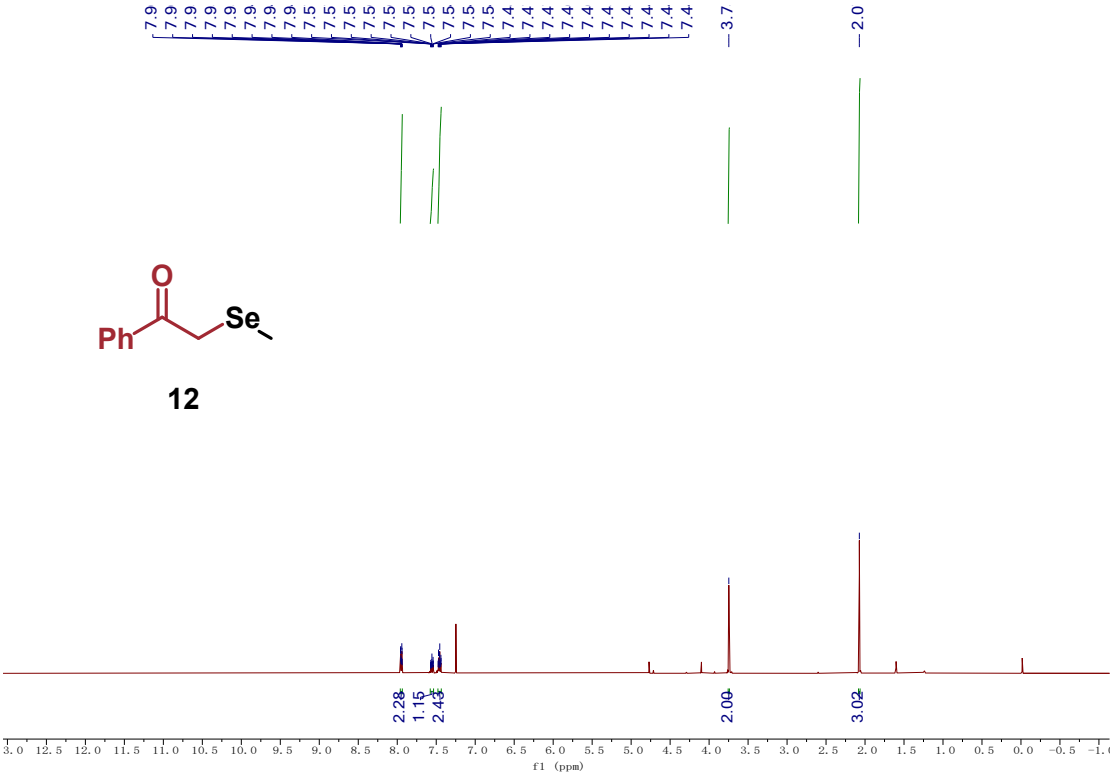
**$^{1}$H NMR of 11 (500 MHz, $CDCl_3$)**




**$^{13}$C NMR of 11 (126 MHz, $CDCl_3$)**

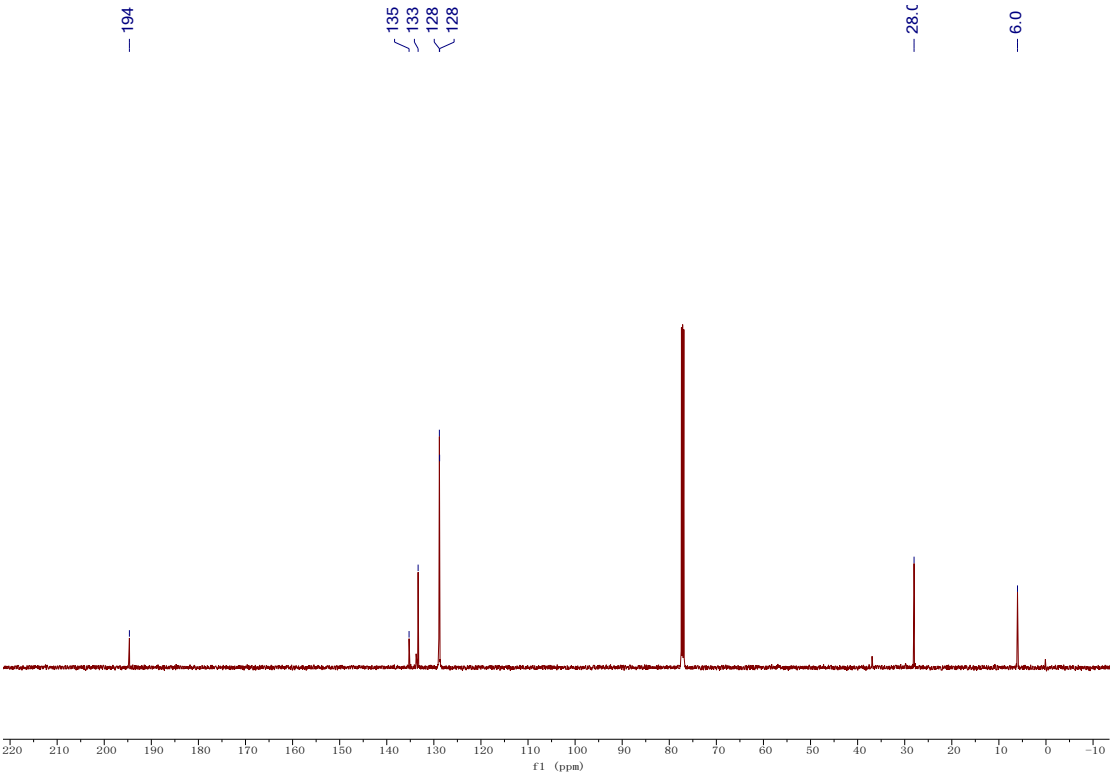


**$^{1}$H NMR of 12 (500 MHz, $CDCl_3$)**

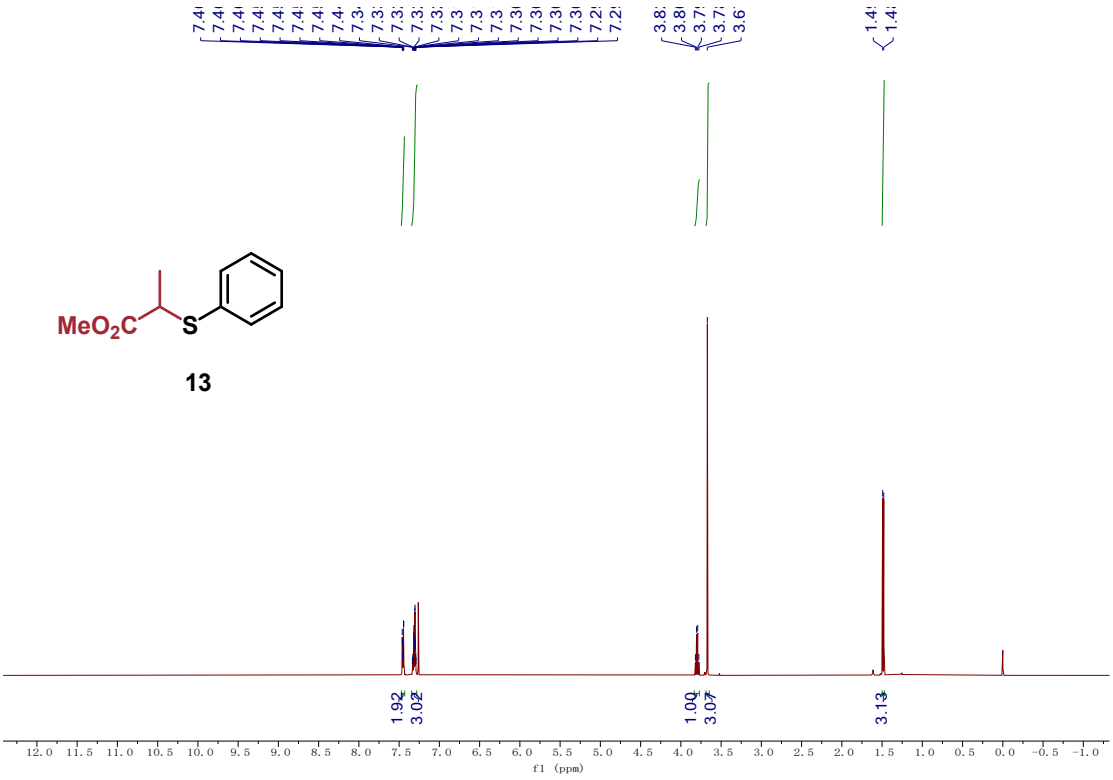



12

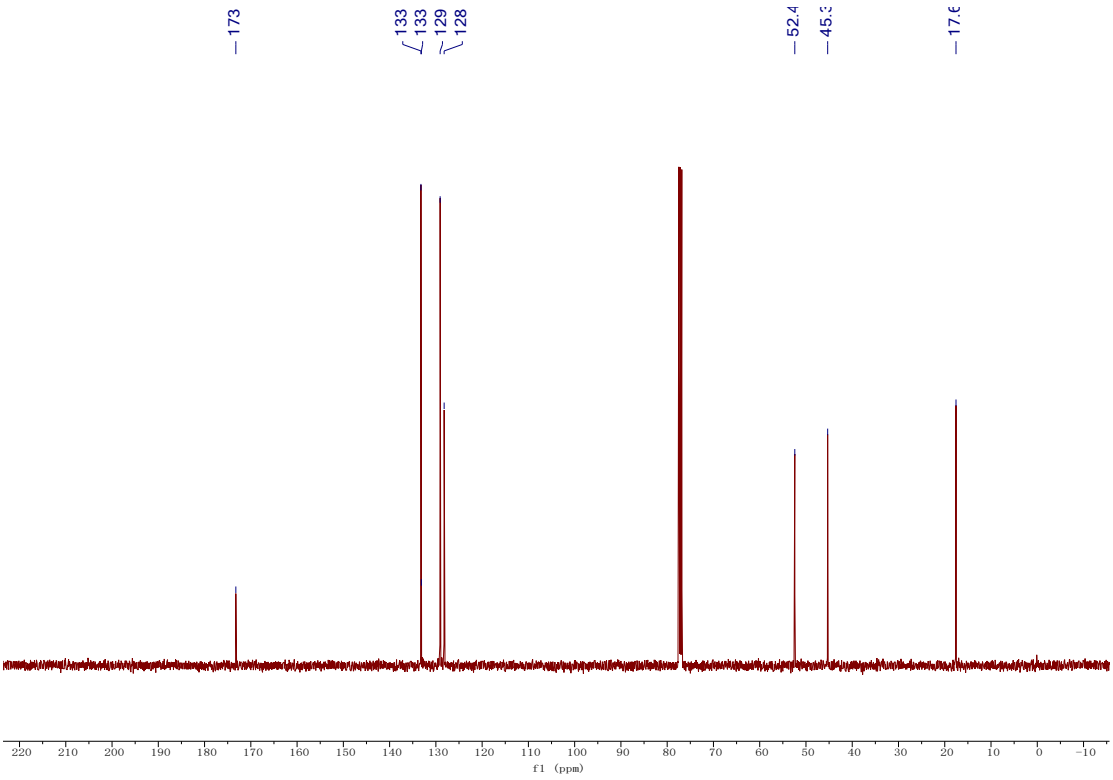
**$^{13}$C NMR of 12 (126 MHz, $CDCl_3$)**



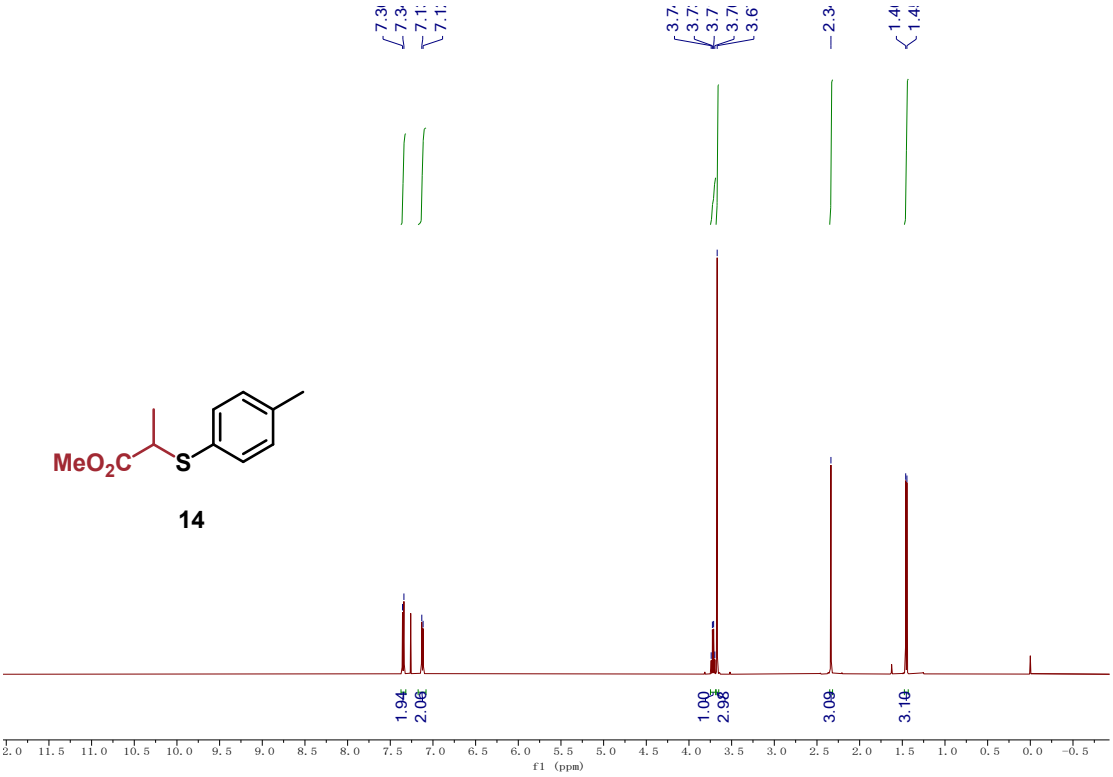
**$^{1}$H NMR of 13 (500 MHz, $CDCl_3$)**



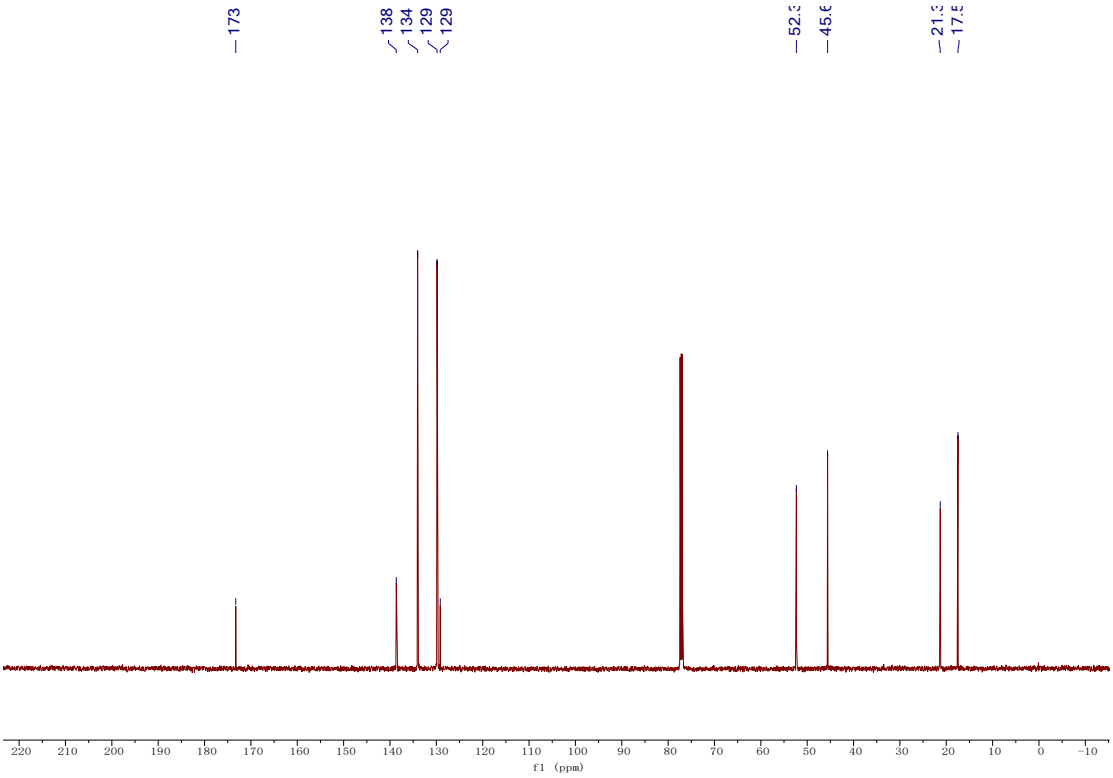

**$^{13}$C NMR of 13 (101 MHz, $CDCl_3$)**



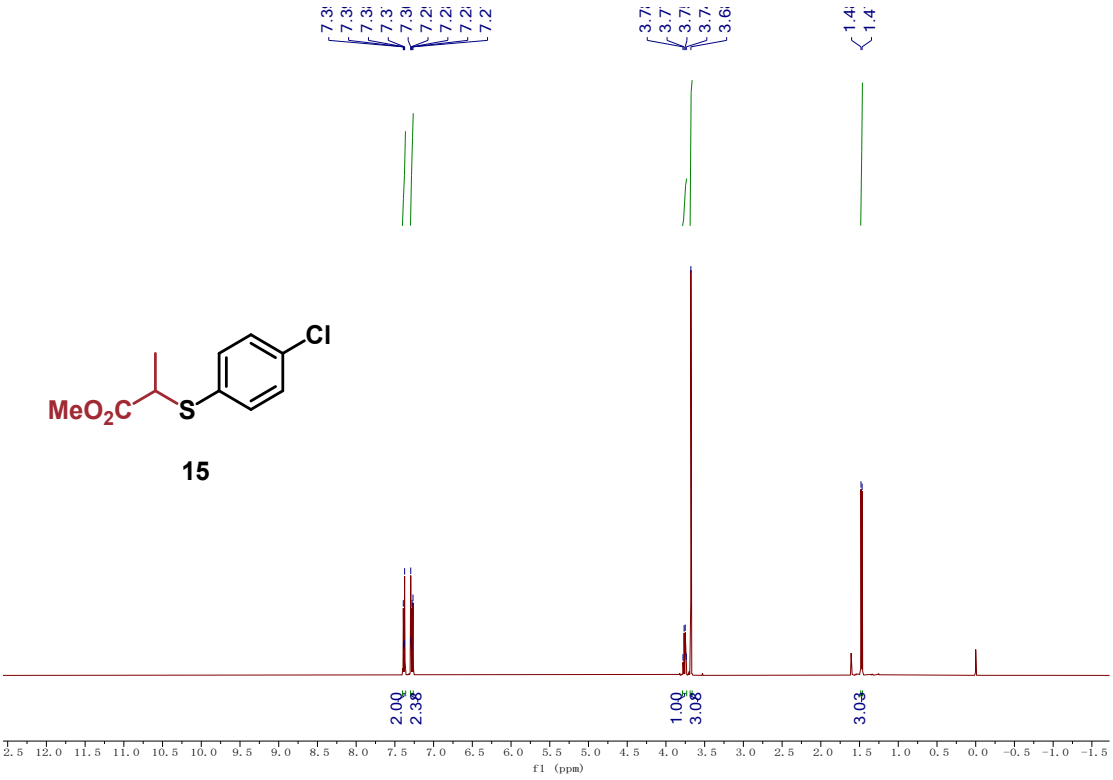
**$^{1}$H NMR of 14 (500 MHz, $CDCl_3$)**



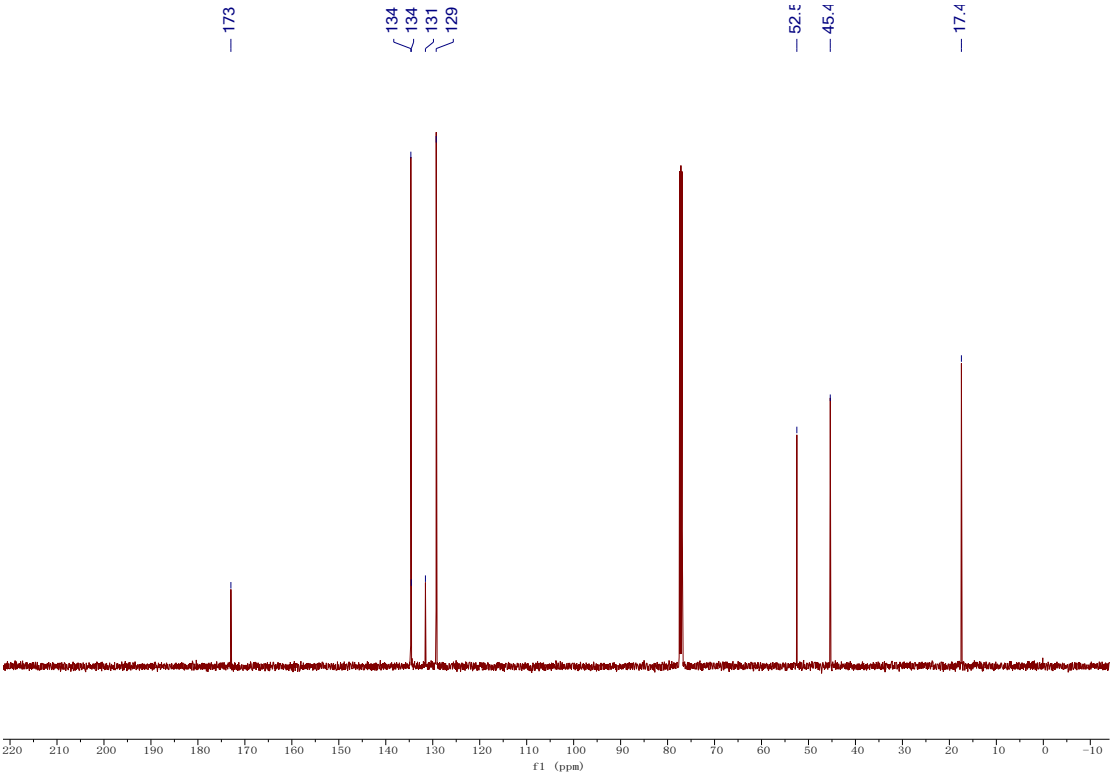

**$^{13}$C NMR of 14 (126 MHz, $CDCl_3$)**



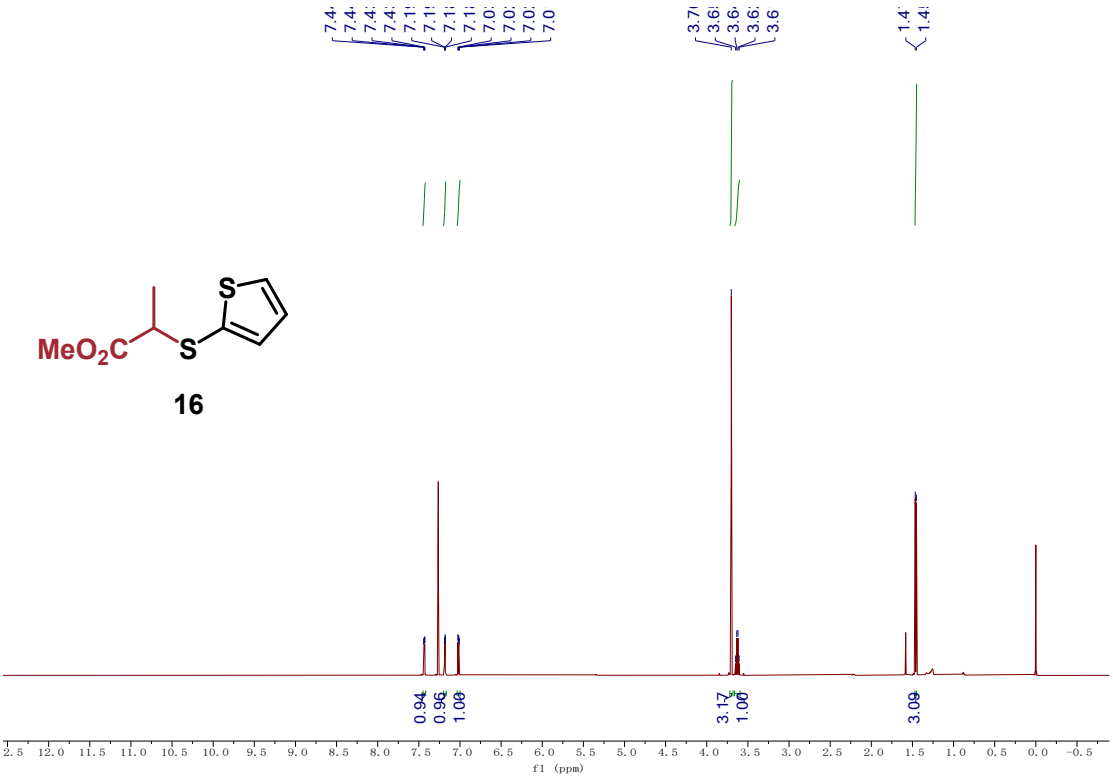
**$^{1}$H NMR of 15 (500 MHz, $CDCl_3$)**



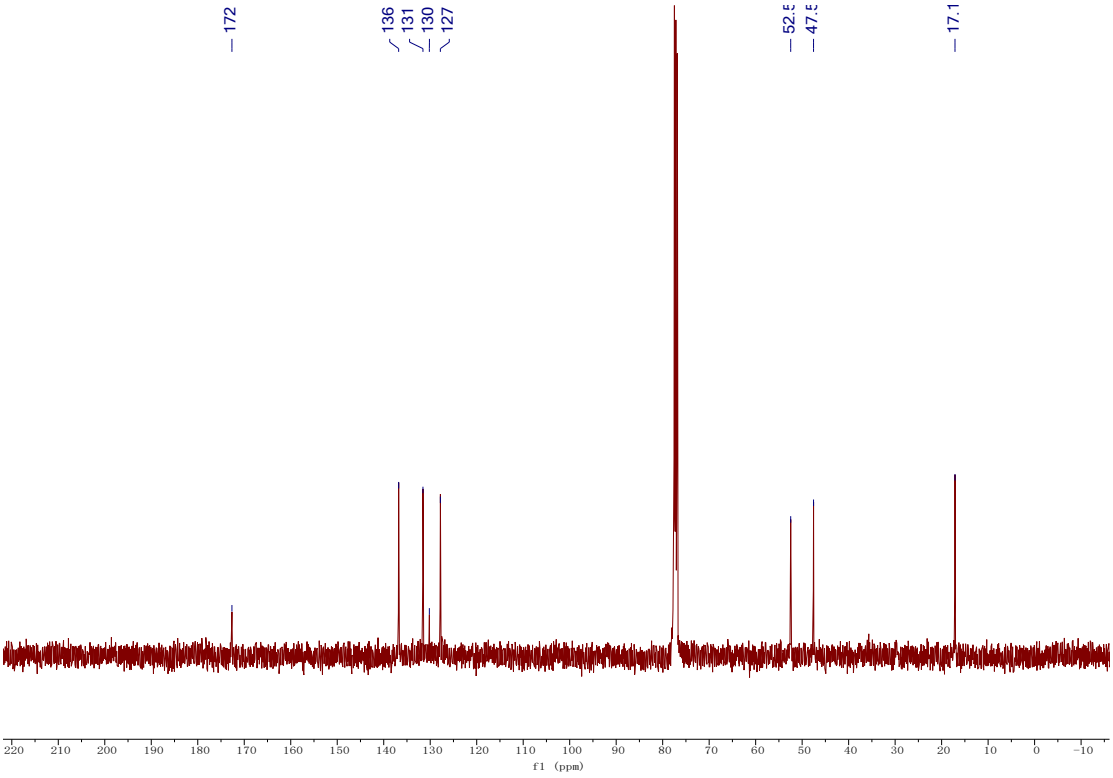

**$^{13}$C NMR of 15 (126 MHz, $CDCl_3$)**



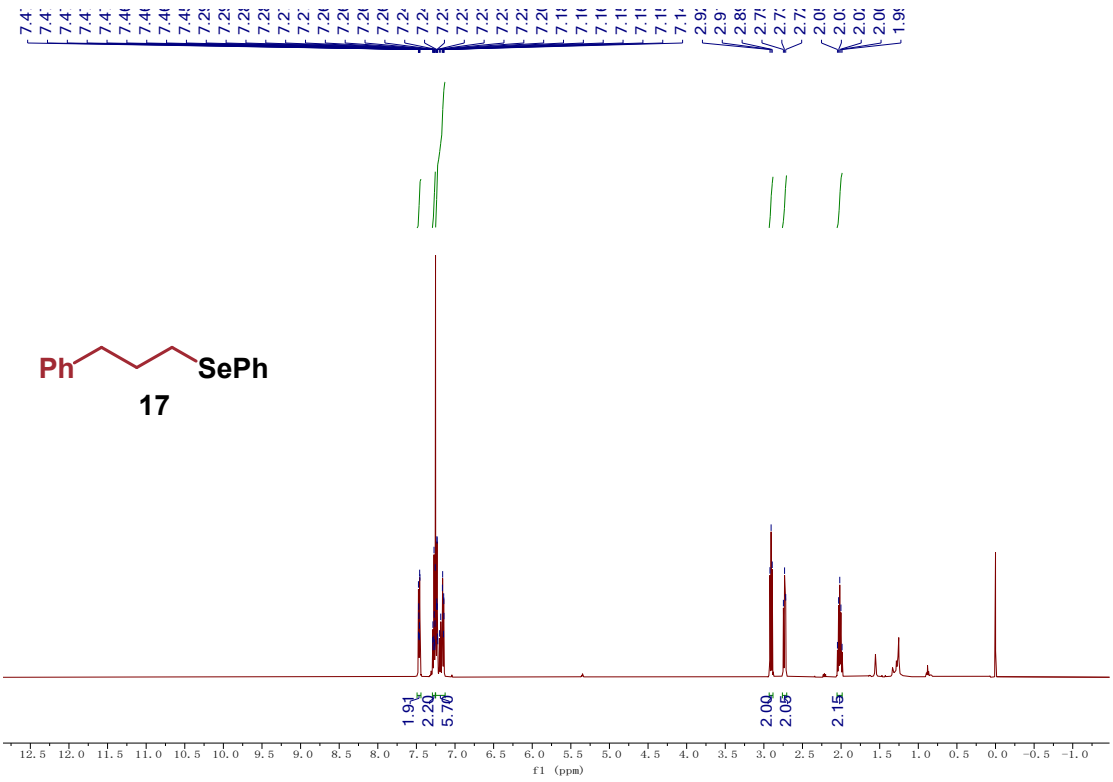
**$^{1}$H NMR of 16 (500 MHz, $CDCl_3$)**



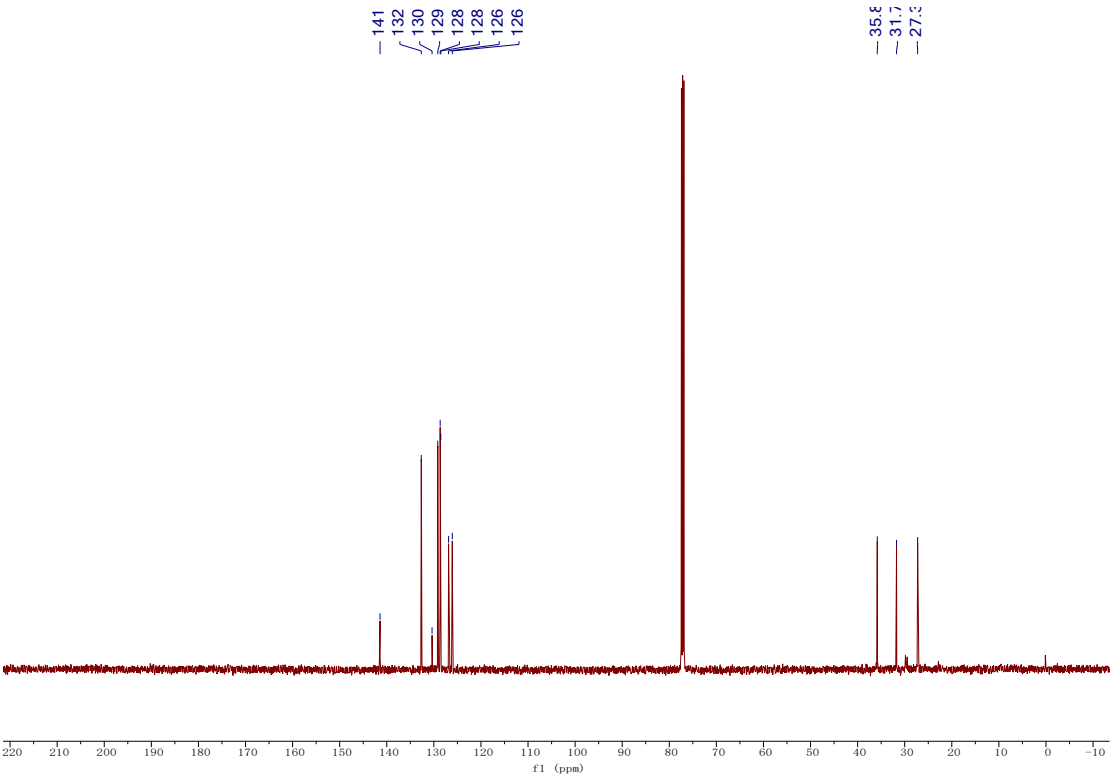

**$^{13}$C NMR of 16 (101 MHz, $CDCl_3$)**



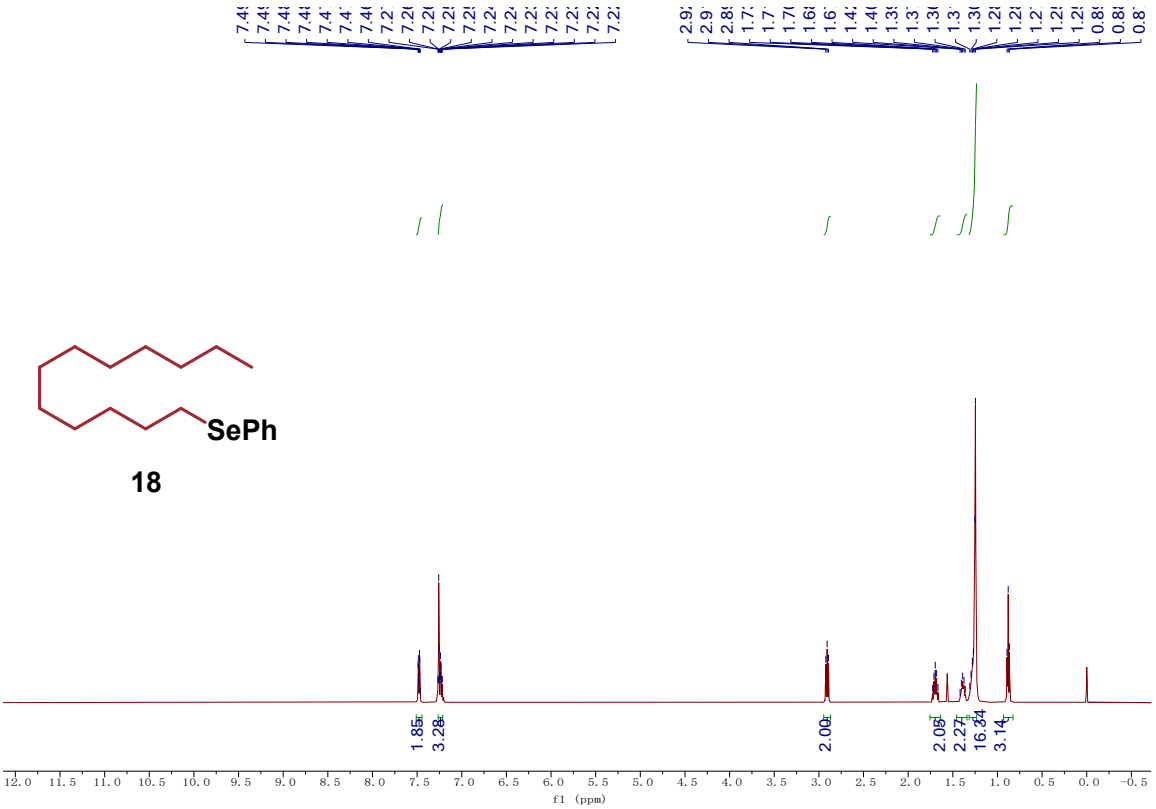
**$^{1}$H NMR of 17 (500 MHz, $CDCl_3$)**



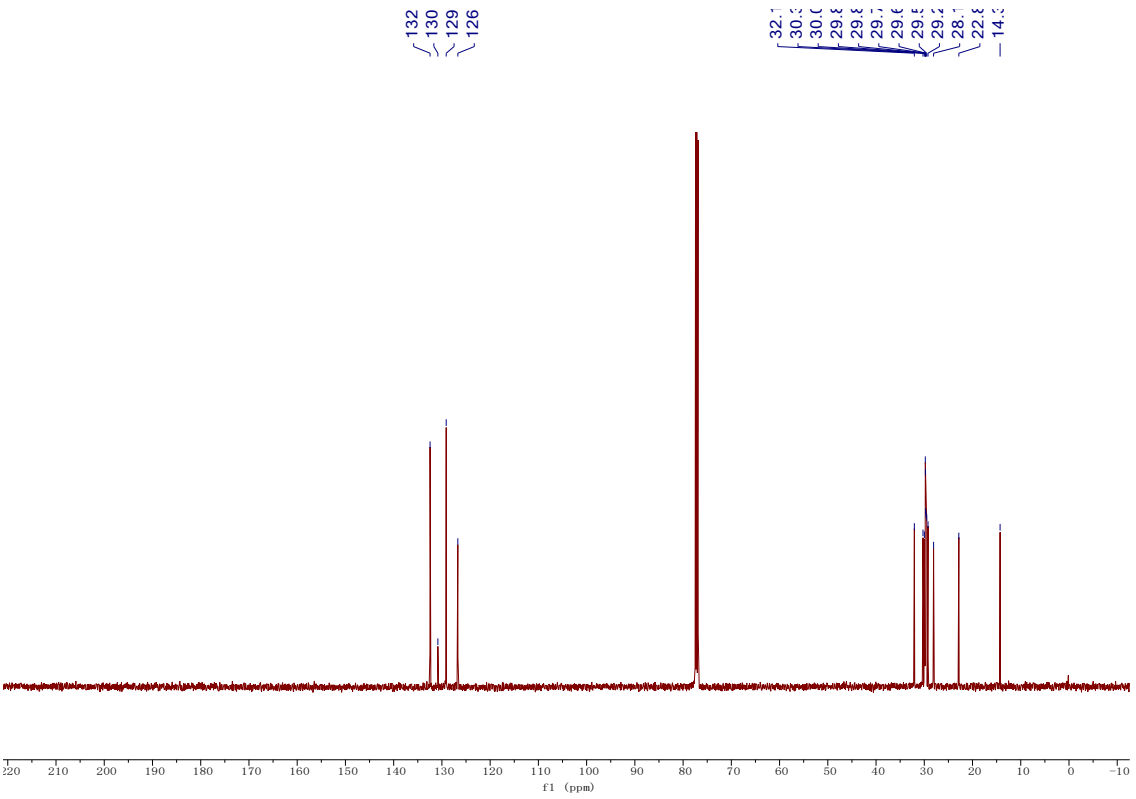

**$^{13}$C NMR of 17 (126 MHz, $CDCl_3$)**



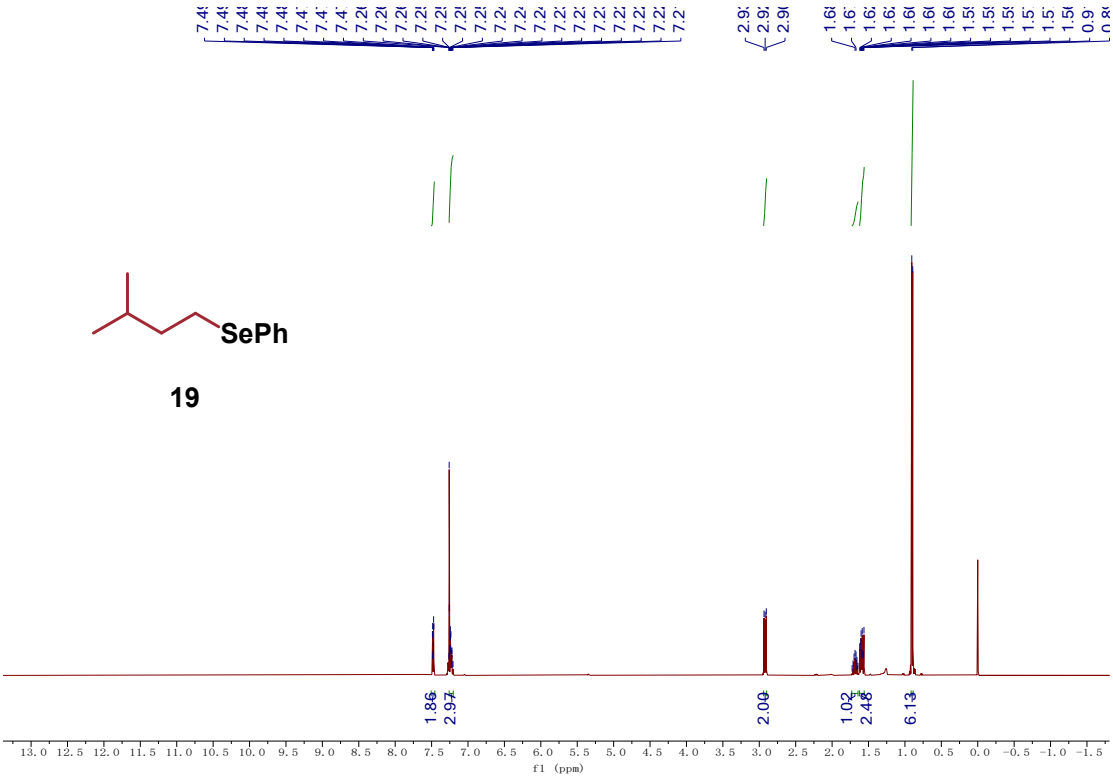
**$^{1}$H NMR of 18 (500 MHz, $CDCl_3$)**



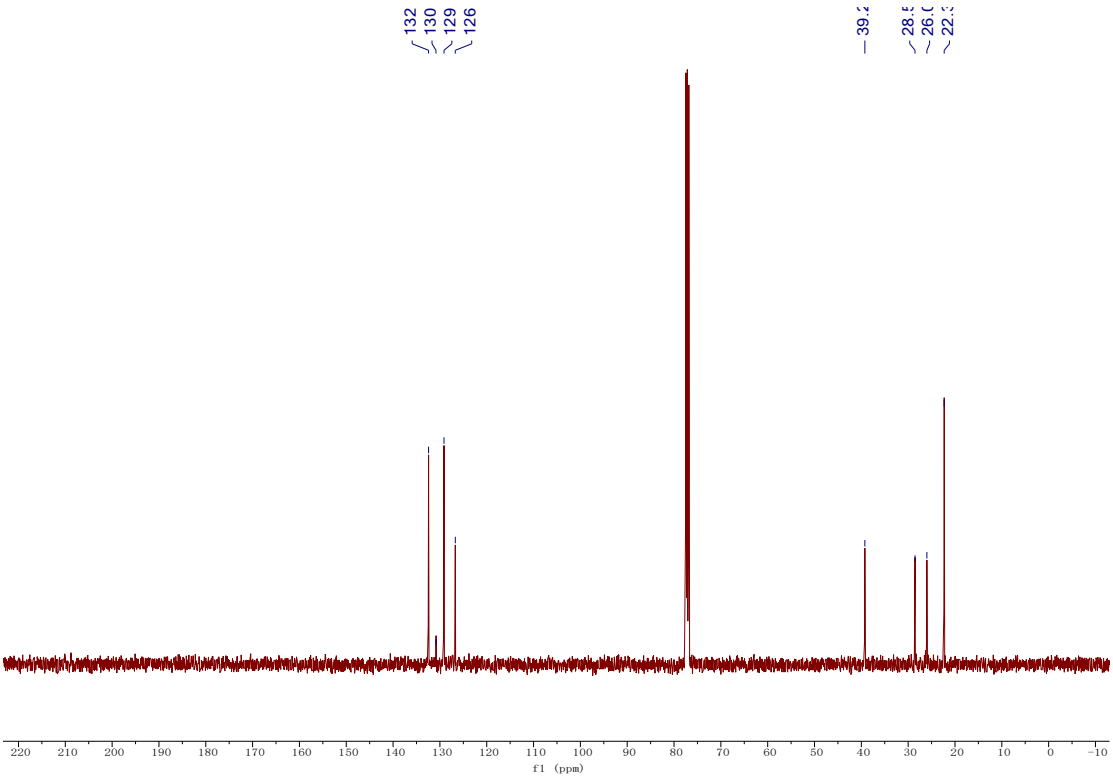

**$^{13}$C NMR of 18 (126 MHz, $CDCl_3$)**



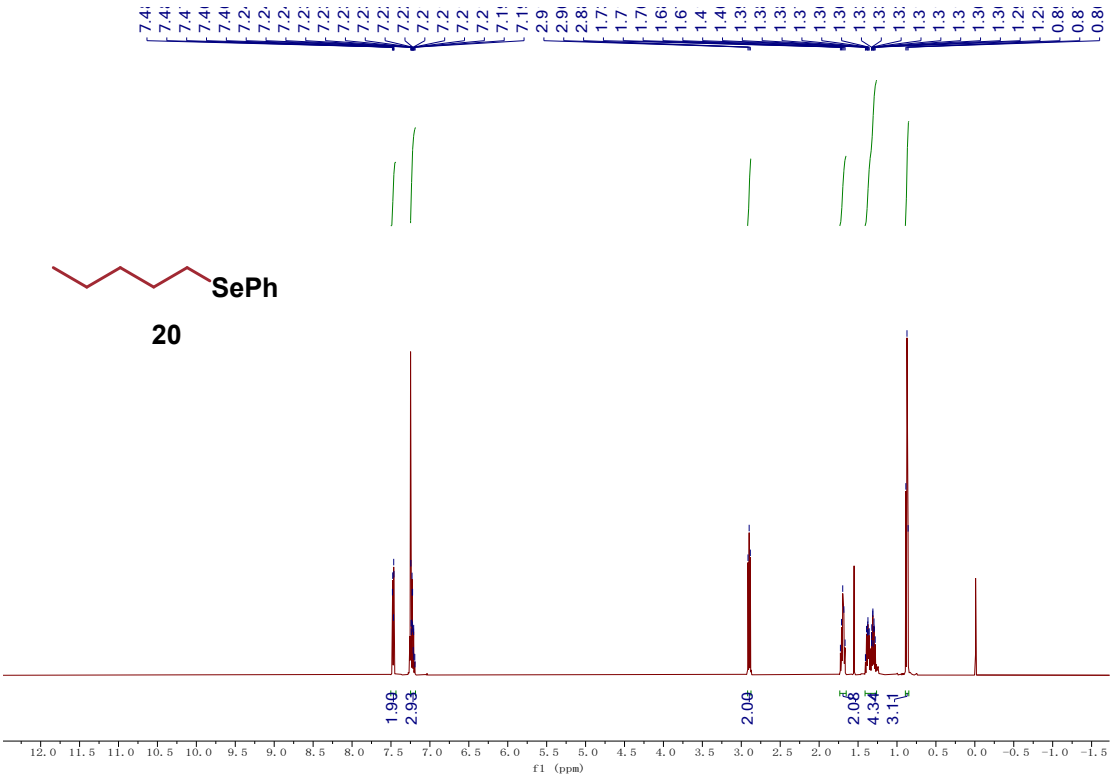
**$^{1}$H NMR of 19 (500 MHz, $CDCl_3$)**



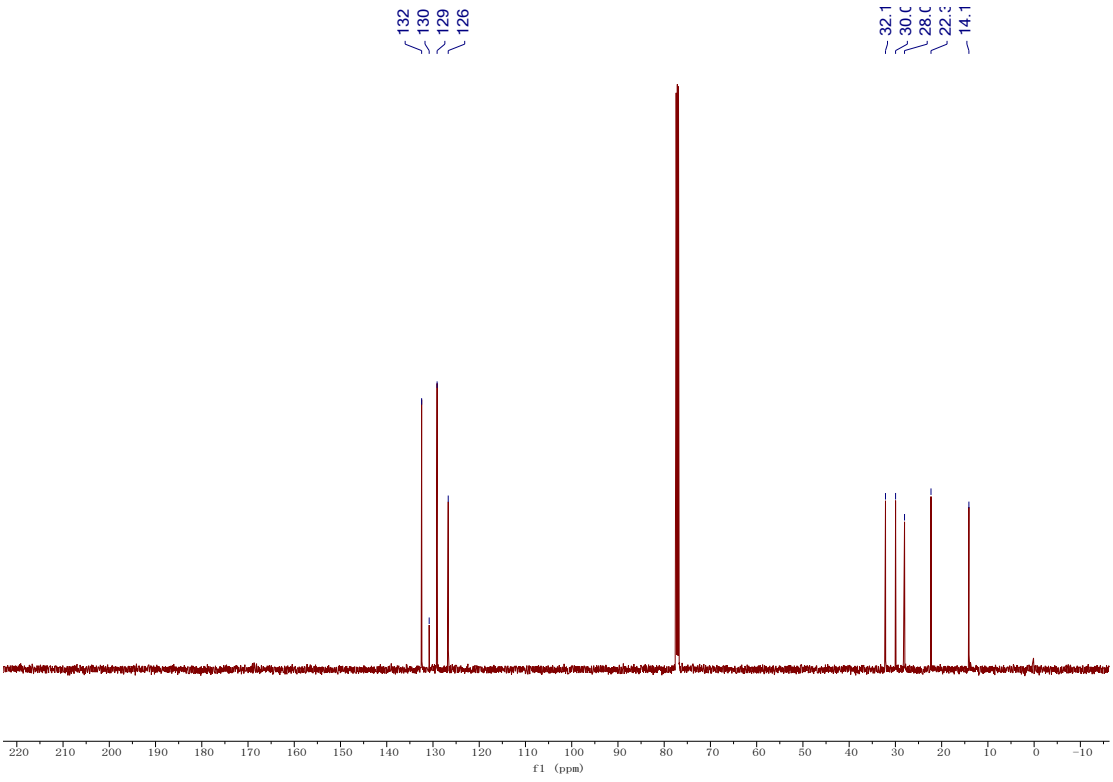

**$^{13}$C NMR of 19 (101 MHz, $CDCl_3$)**



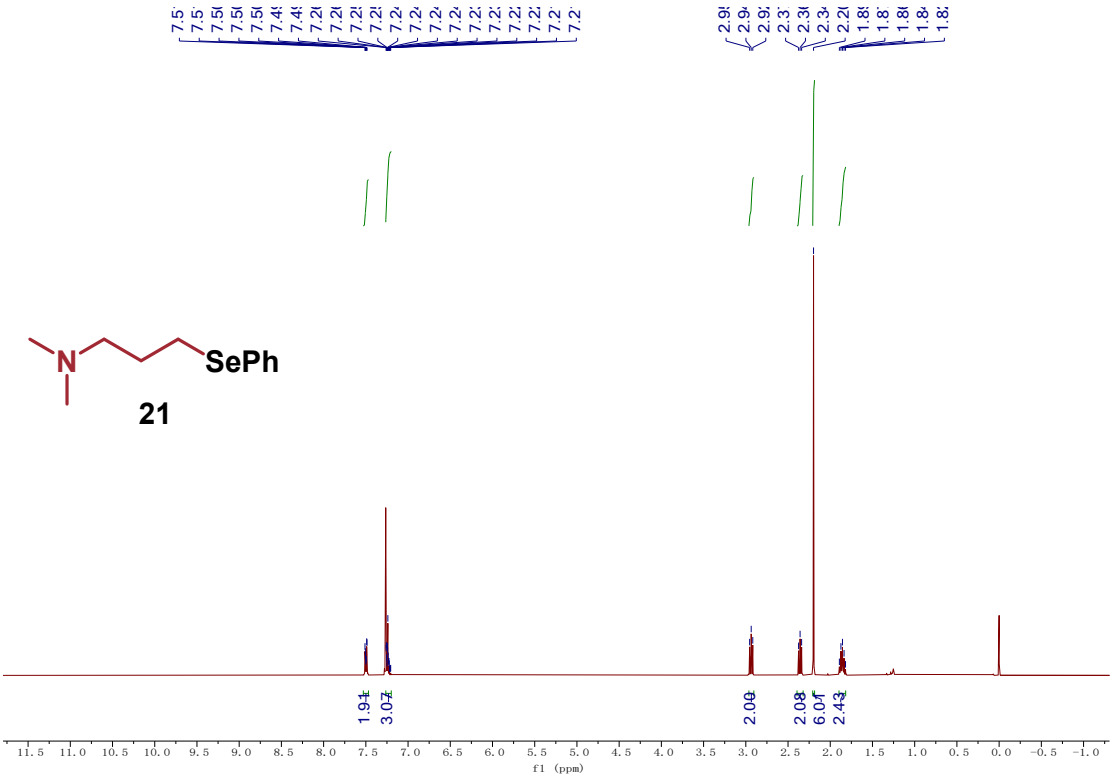
**$^{1}$H NMR of 20 (500 MHz, $CDCl_3$)**



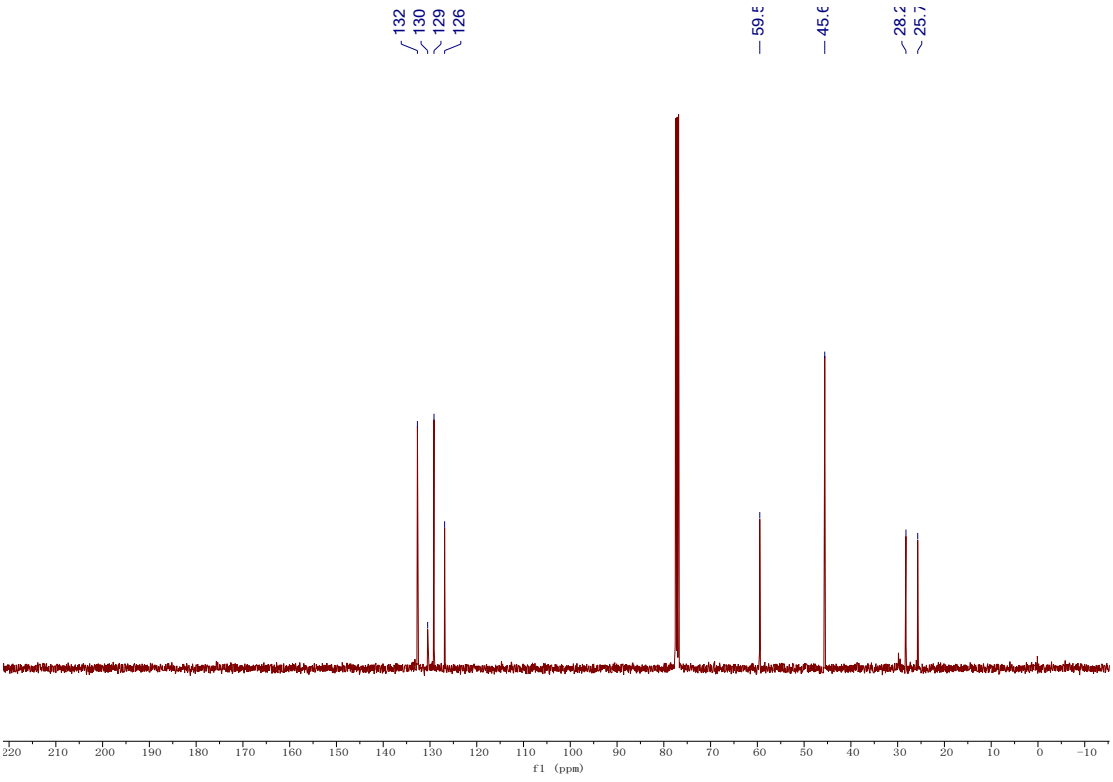

**$^{13}$C NMR of 20 (126 MHz, $CDCl_3$)**



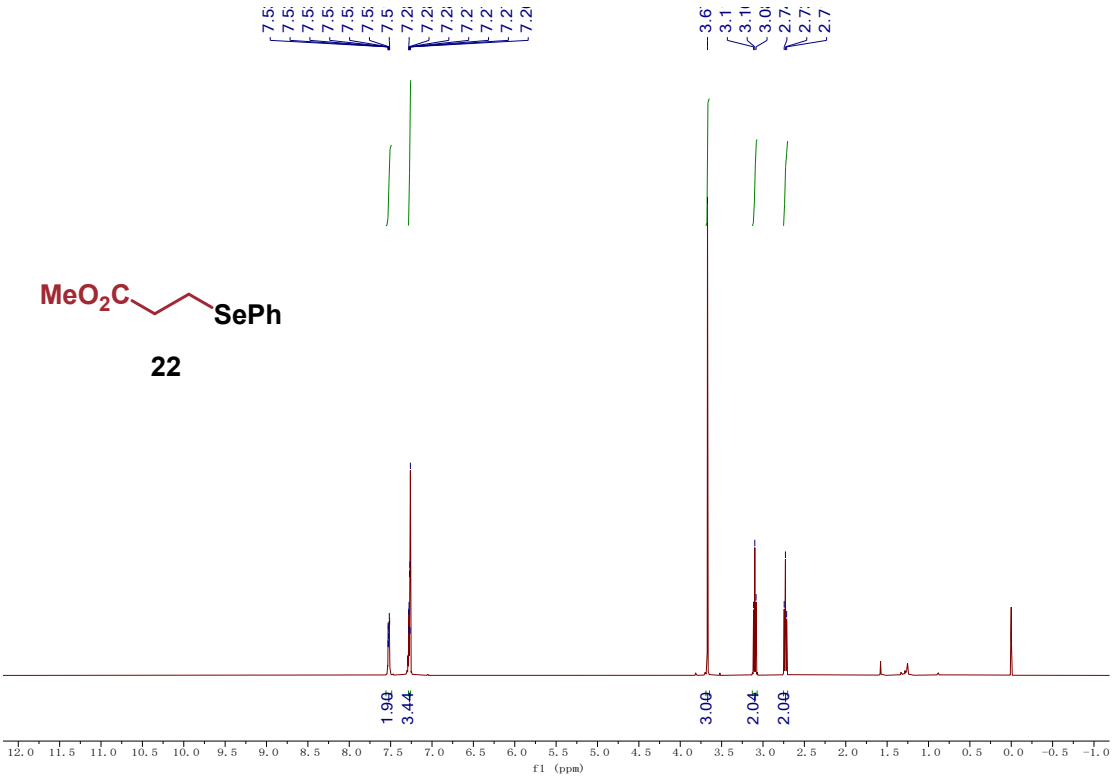
**$^{1}$H NMR of 21 (400 MHz, $CDCl_3$)**



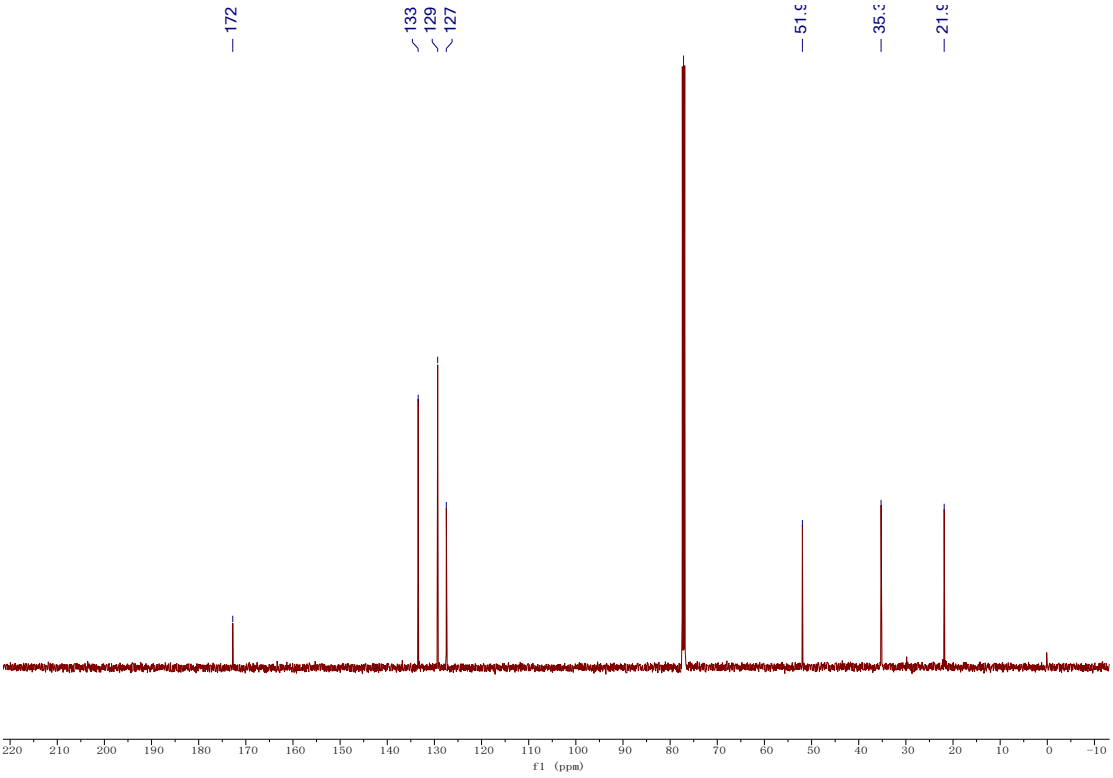

**$^{13}$C NMR of 21 (101 MHz, $CDCl_3$)**



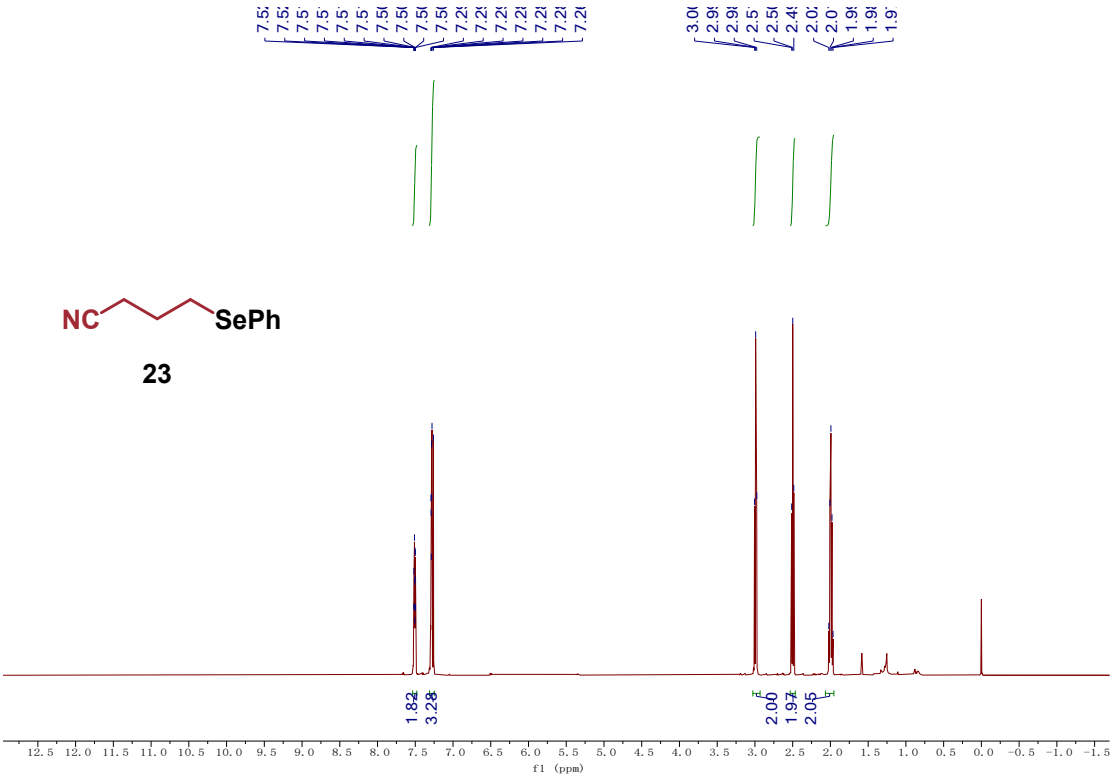
**$^{1}$H NMR of 22 (500 MHz, $CDCl_3$)**



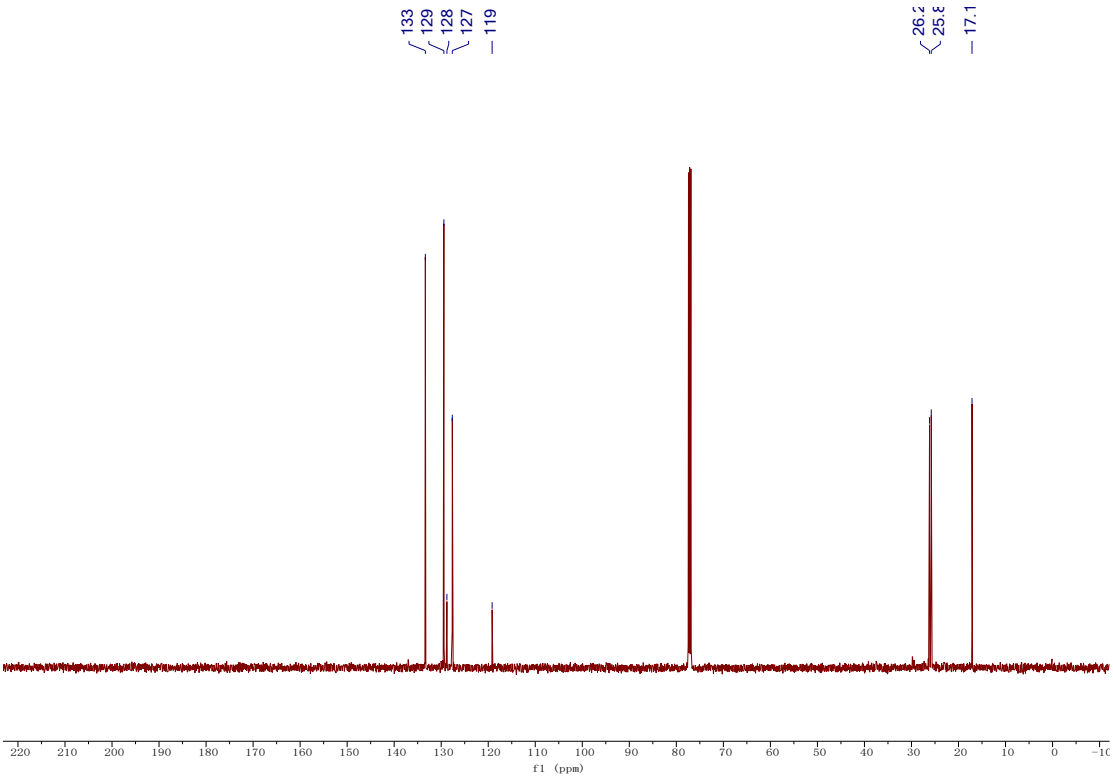

**$^{13}$C NMR of 22 (126 MHz, $CDCl_3$)**



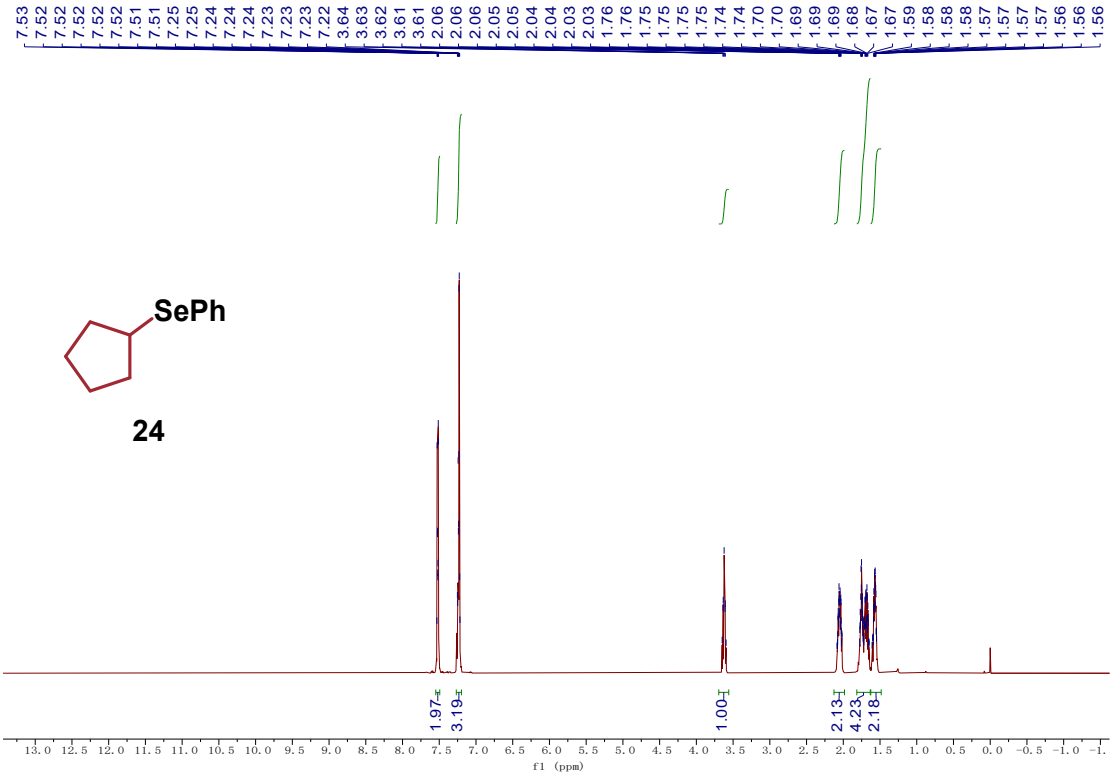
**$^{1}$H NMR of 23 (500 MHz, $CDCl_3$)**



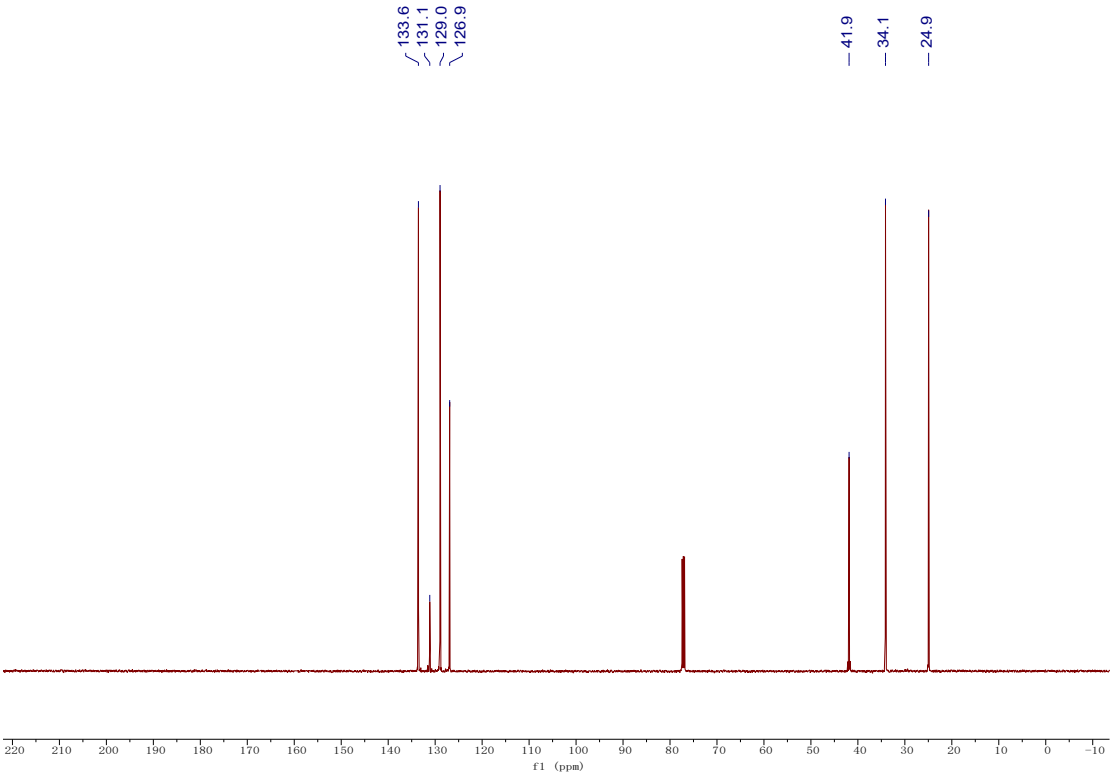

**$^{13}$C NMR of 23 (126 MHz, $CDCl_3$)**



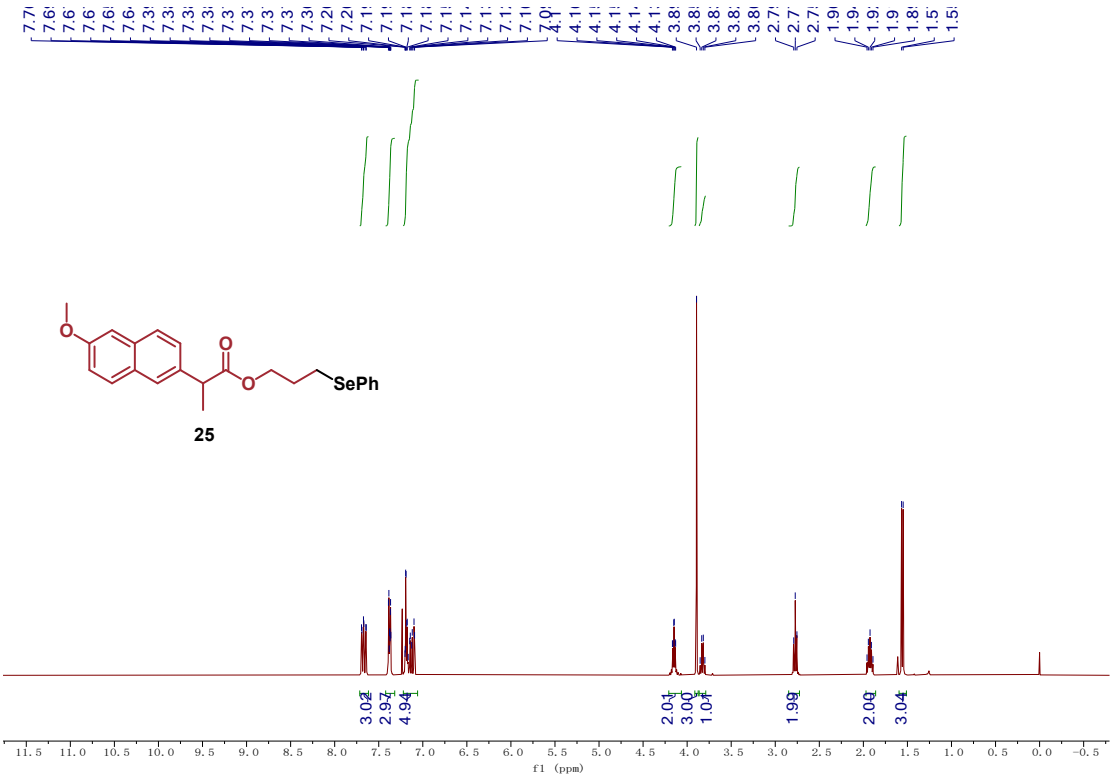
**$^{1}$H NMR of 24 (500 MHz, $CDCl_3$)**



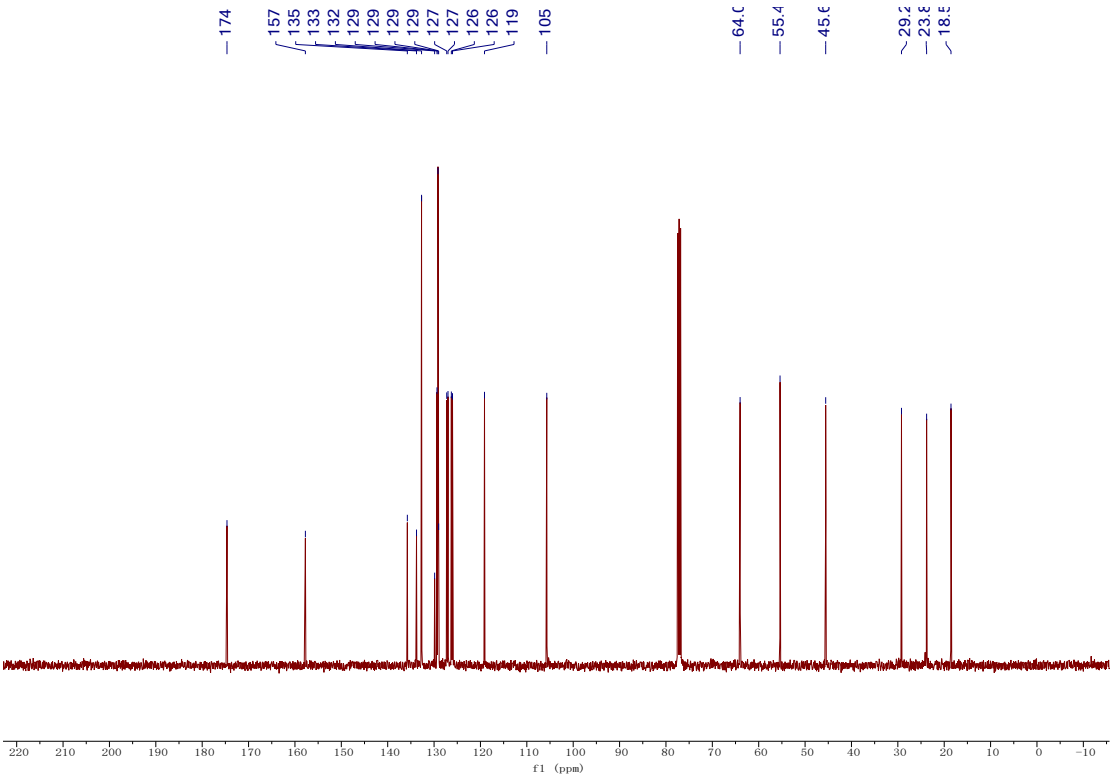

**$^{13}$C NMR of 24 (126 MHz, $CDCl_3$)**



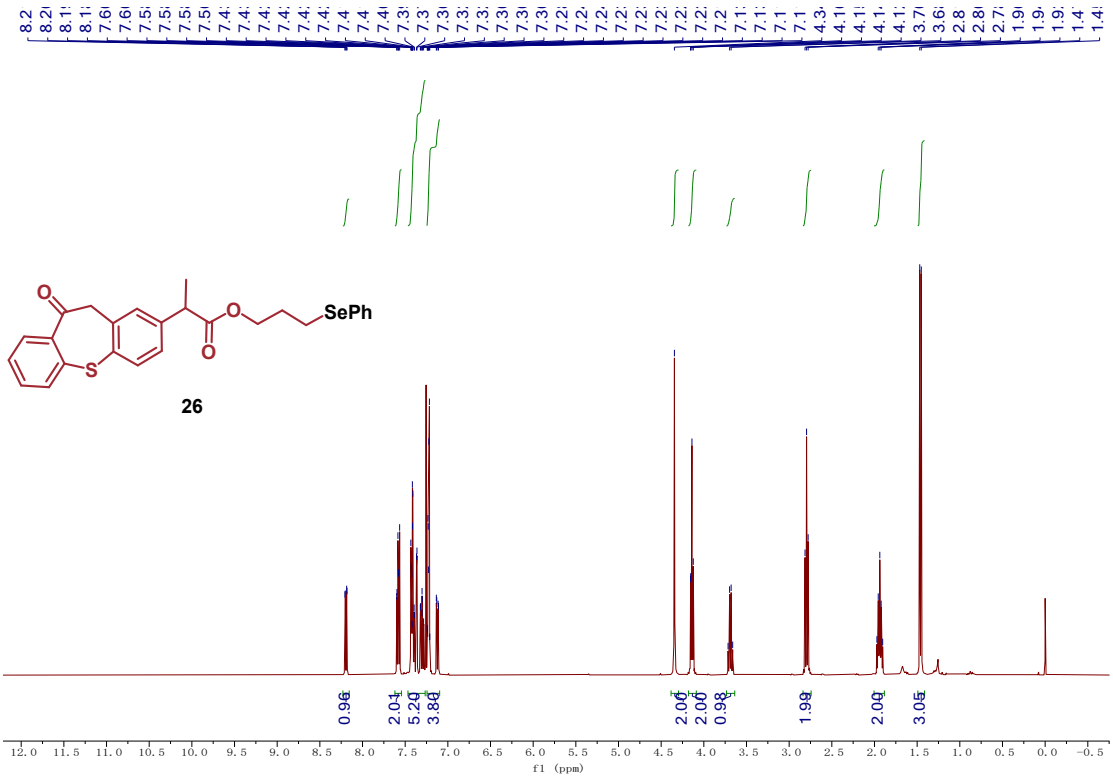
**$^{1}$H NMR of 25 (400 MHz, $CDCl_3$)**



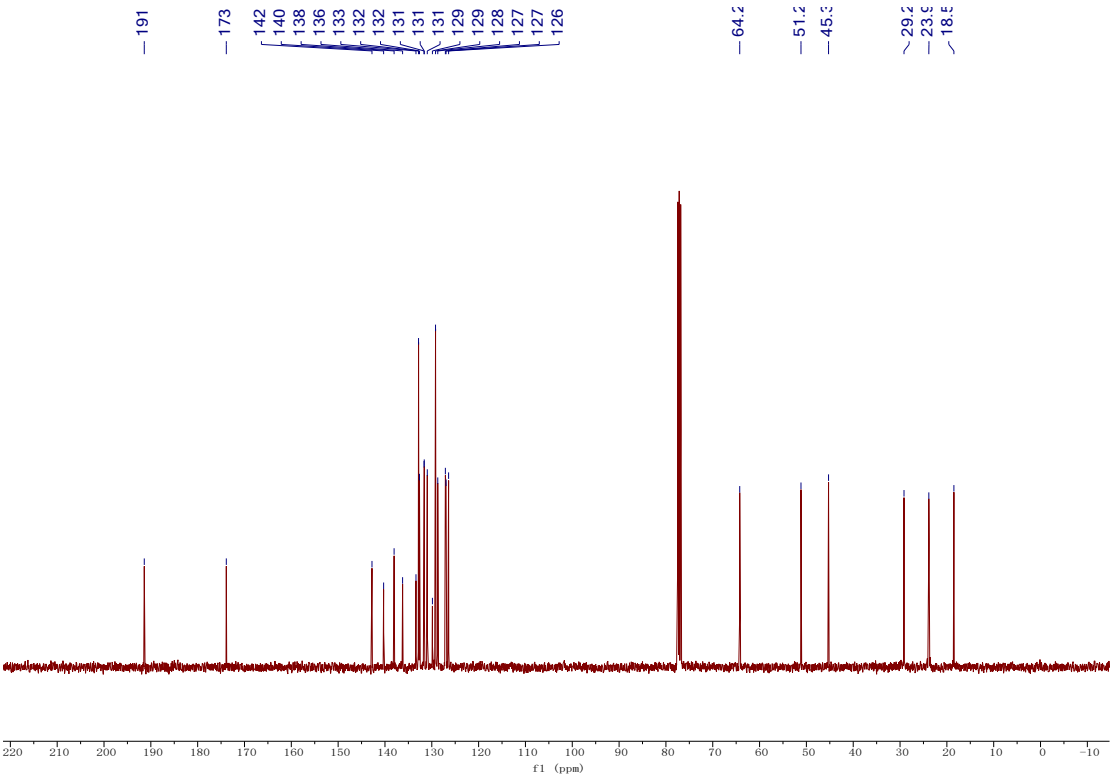

**$^{13}$C NMR of 25 (101 MHz, $CDCl_3$)**



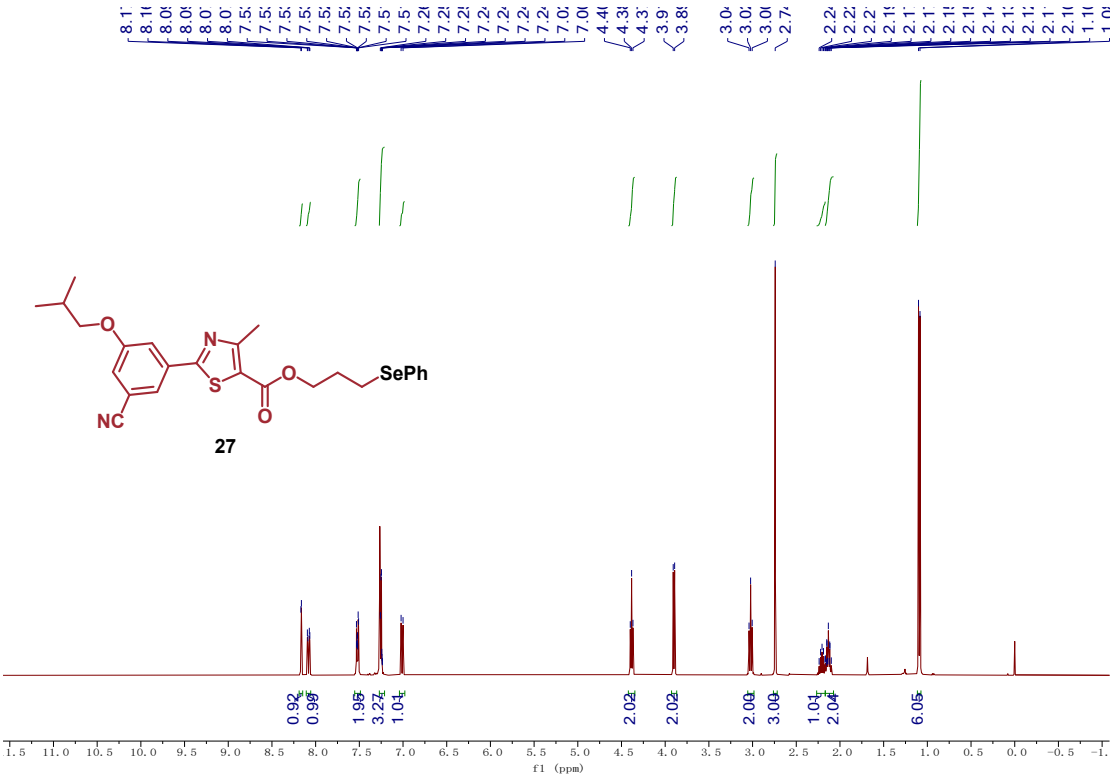
**$^{1}$H NMR of 26 (400 MHz, $CDCl_3$)**



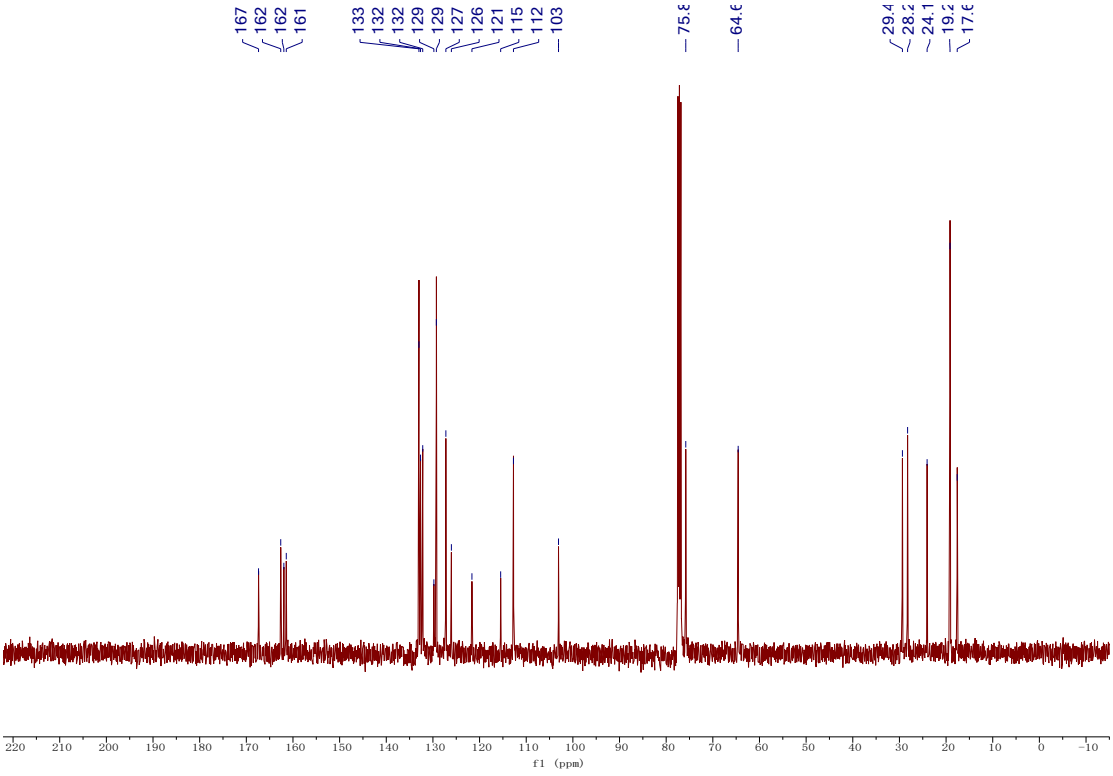

**$^{13}$C NMR of 26 (101 MHz, $CDCl_3$)**



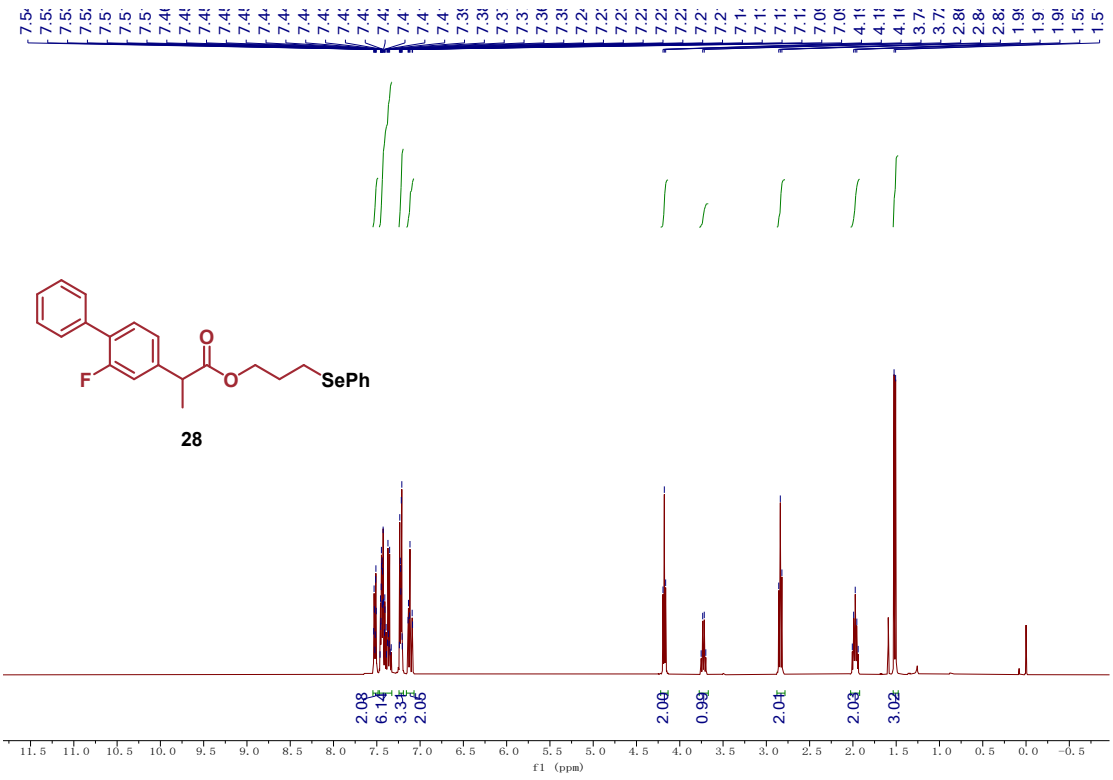
**$^{1}$H NMR of 27 (400 MHz, $CDCl_3$)**



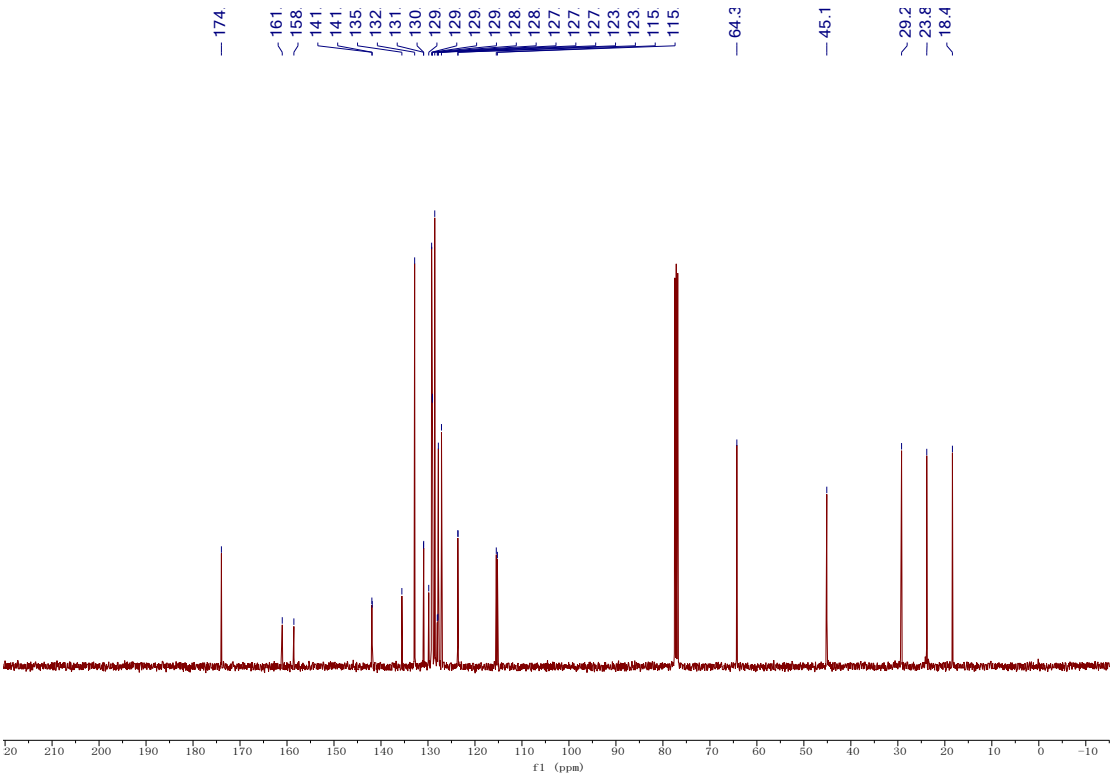

**$^{13}$C NMR of 27 (101 MHz, $CDCl_3$)**



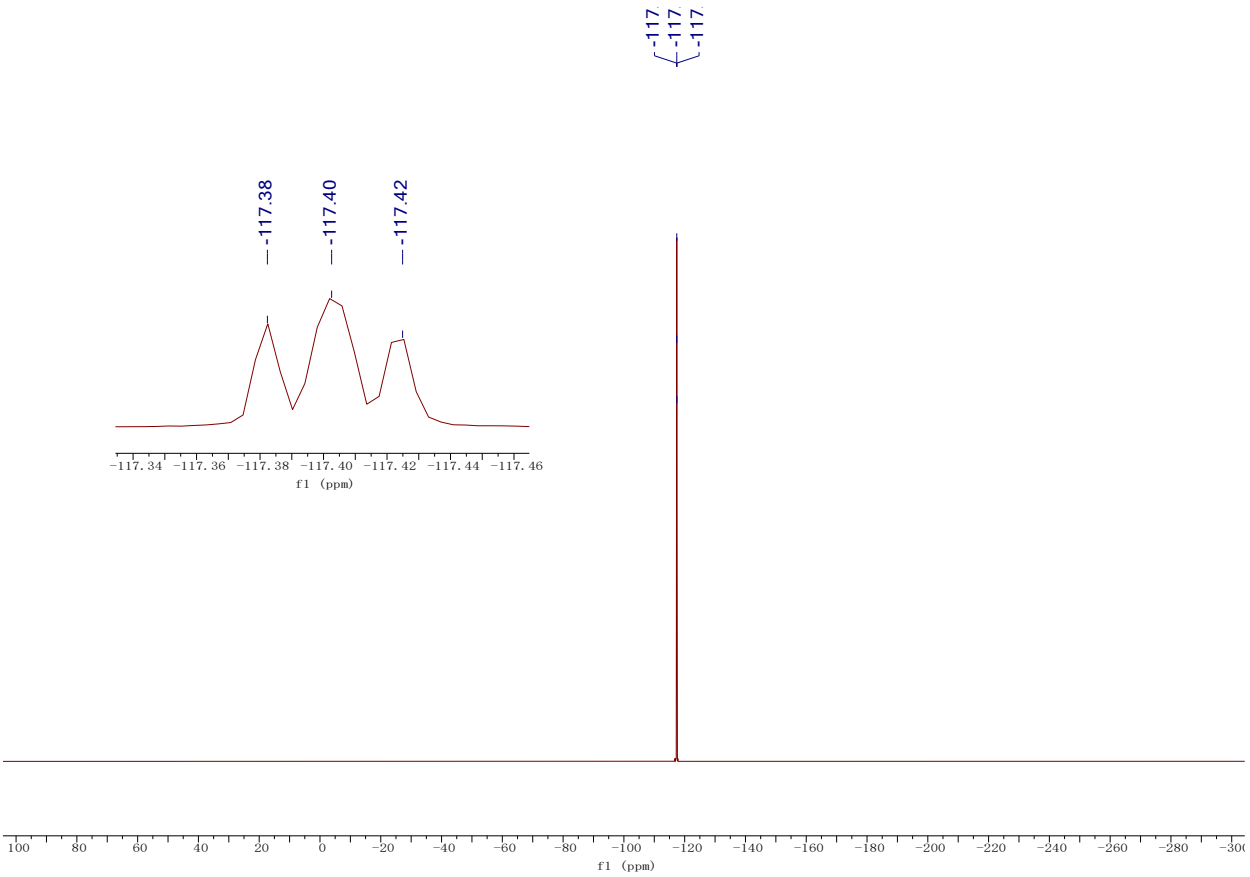
**$^{1}$H NMR of 28 (400 MHz, $CDCl_3$)**



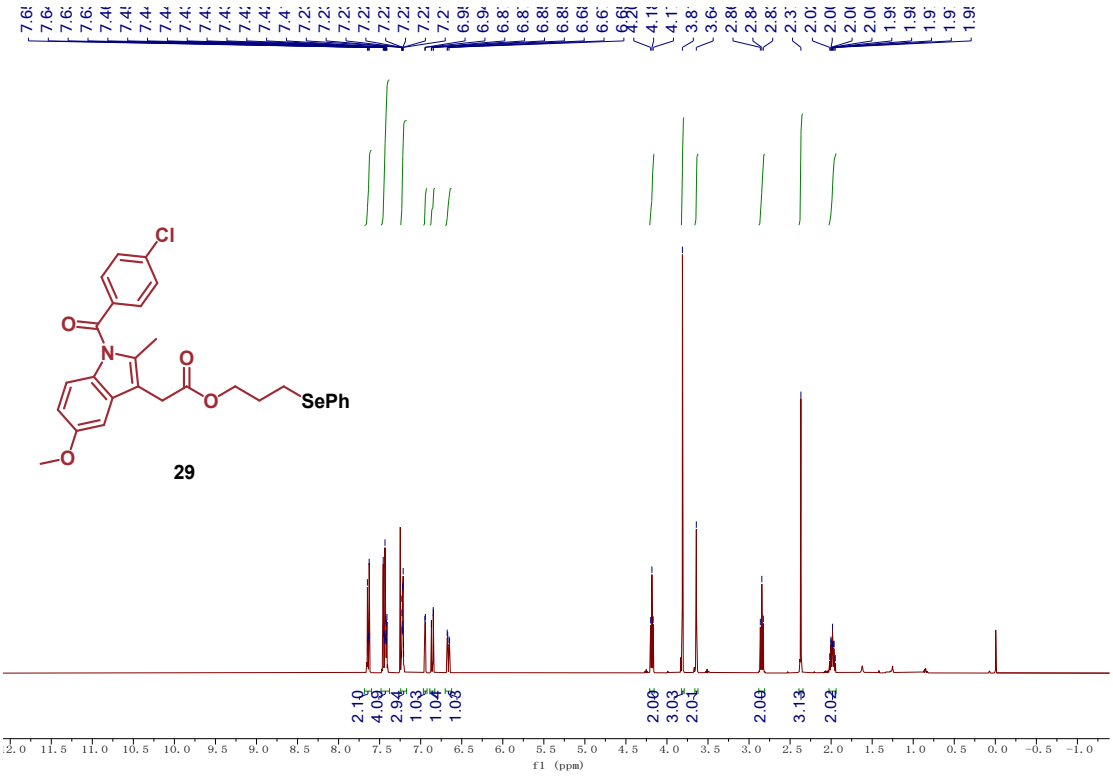

**$^{13}$C NMR of 28 (101 MHz, $CDCl_3$)**



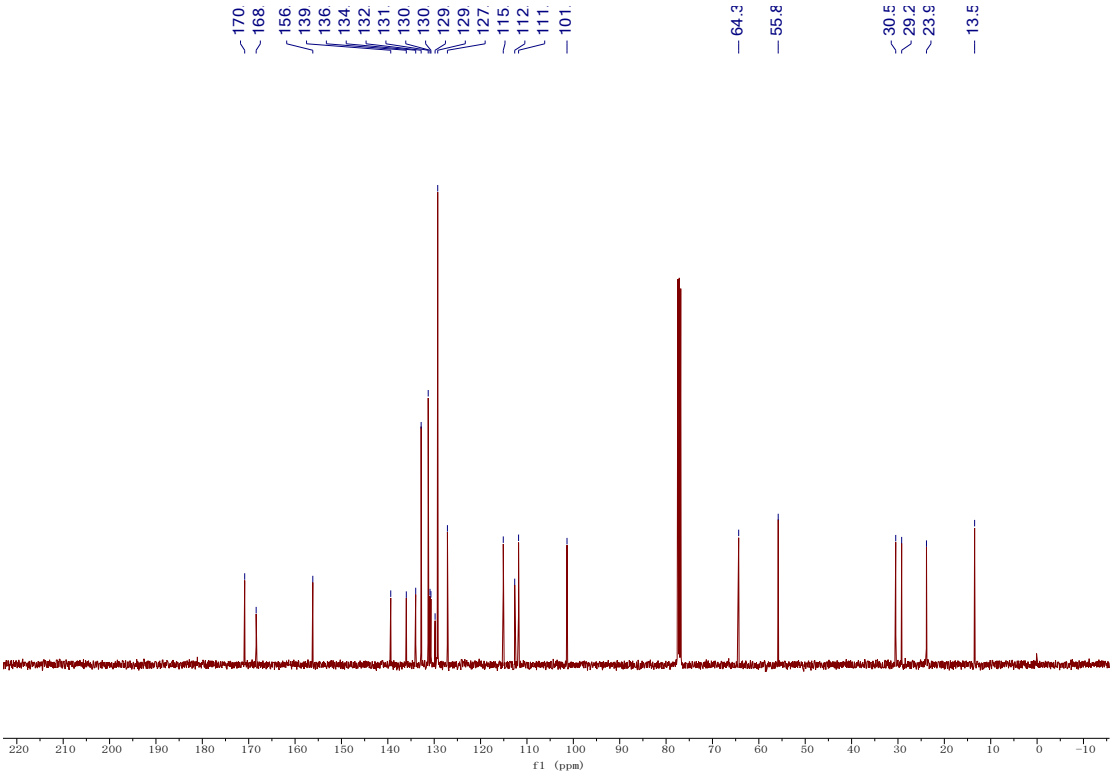

**$^{19}$F NMR of 28 (471 MHz, $CDCl_3$)**


-117.38
-117.40
-117.42
-117.34 -117.36 -117.38 -117.40 -117.42 -117.44 -117.46
f1 (ppm)
100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
f1 (ppm)

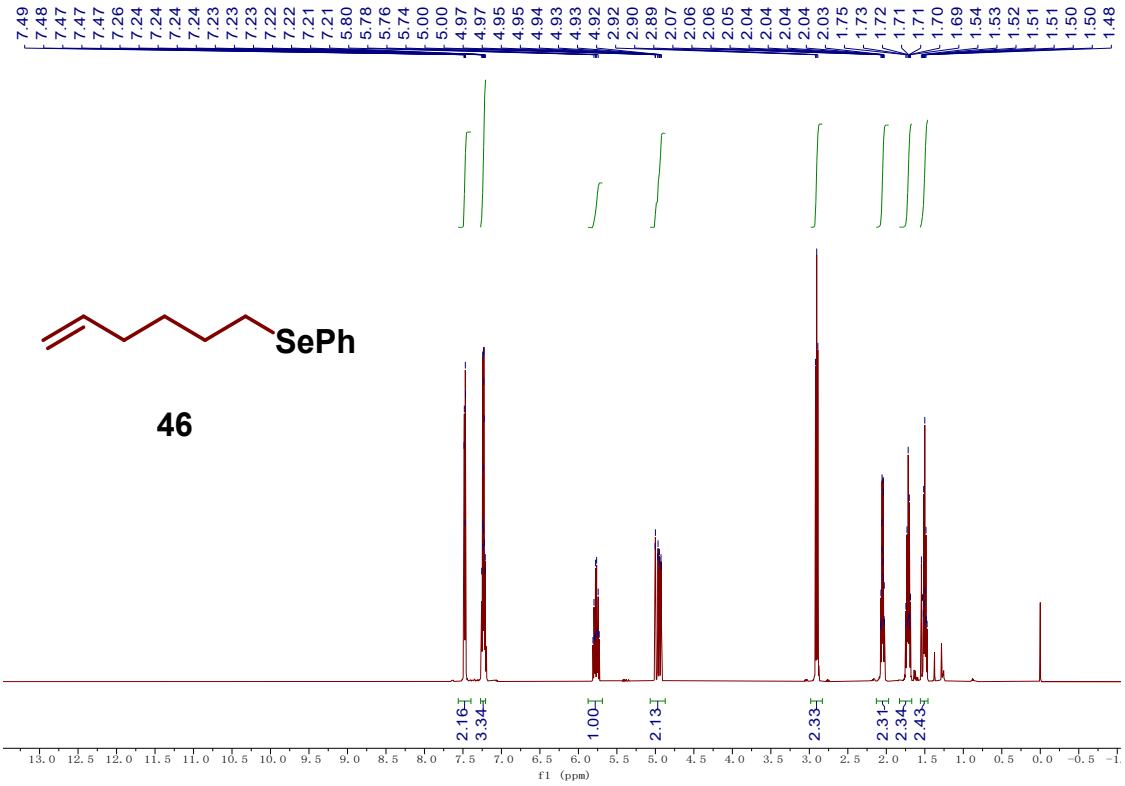
**$^{1}$H NMR of 29 (400 MHz, $CDCl_3$)**



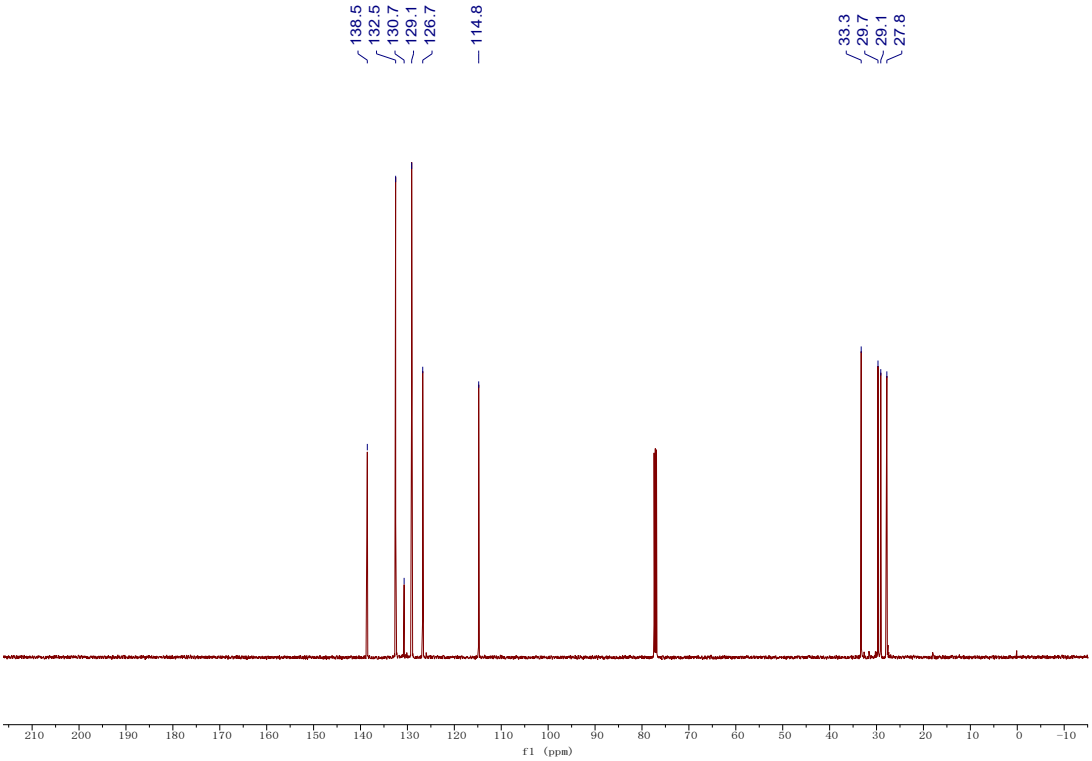

**$^{13}$C NMR of 29 (101 MHz, $CDCl_3$)**



**$^{1}$H NMR of 31 (500 MHz, $CDCl_3$)**




**$^{13}$C NMR of 31 (126 MHz, $CDCl_3$)**



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