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Visible-Light-Driven PCy_3-promoted deselenization of 1,2-diselenides

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1. General information

All reagents and solvents were purchased from TCI, Sigma-Aldrich, Alfa Aesar, Acros and Energy Chemical. All commercial reagents were used as supplied unless otherwise stated. Organic solutions were concentrated by rotary evaporation below 45 °C. All reactions were monitored by TLC, GC-MS. Analytical thin-layer chromatography was performed using Merck Kieselgel 60 F254 0.20 mm precoated glass-backed silica gel plates. Visualization of the chromatogram was performed by UV absorbance (λ_{max} = 254 nm) and/or by staining with aqueous potassium permanganate. Flash column chromatography was performed using silica gel (EM 60 F254 300 - 400 mesh) with the appropriate solvent system.

The photoreactor used in this research was bought from Taobao (www.taobao.com) (Fig. S1: blue LED, 50W, 450 nm).

Fig. S1 The photoreactor

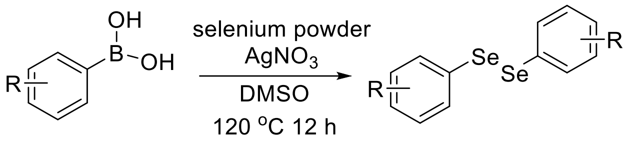
Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Bruker DPX 400 (400 MHz) or Avance 500 (500 MHz) spectrometer. Chemical shifts (δ) are recorded in parts per million (ppm) and are quoted to the nearest 0.01 ppm relative to the residual solvent protons ($CDCl_3$ = 7.26 ppm). Coupling constants (J) are quoted in Hertz (Hz), and data reported as follows: Chemical shift (multiplicity, coupling constant, number of protons). Coupling constants were reported to the nearest 0.1 Hz and multiplicity reported according to the following: s =singlet, d = doublet, t = triplet, q = quartet, qui = quintet m = multiplet, br = broad, with associated combinations e.g. dd = doublet of doublets

Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker AVANCE 500 (125 MHz) spectrometer. Chemical shifts (δ) are recorded in parts per million (ppm) and are quoted to the nearest 0.1 ppm relative to the residual solvent protons ($CDCl_3$ = 77.2 ppm). High-resolution mass spectra were recorded on a micrOTOF-Q II 10410 mass spectrometer.

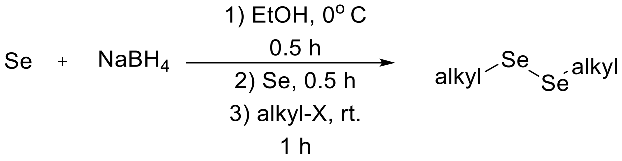
Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification.

2. Materials and methods

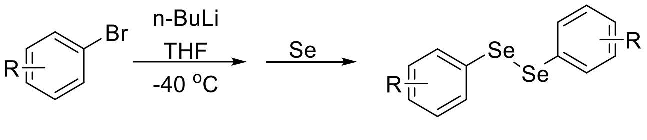
Preparation of starting materials



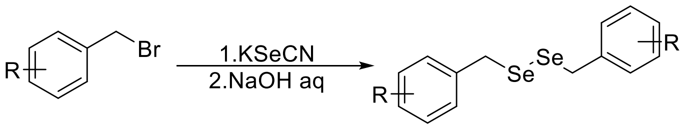
Phenylboric acid (5 mmol), selenium powder (15 mmol), and silver nitrate (5 mmol) were added to DMSO solution (15 mL) and the reaction mixture was stirred in air at 130 °C for 12 hours. Then the reaction mixture was cooled to ambient temperature, and evaporated under reduced pressure. The residue was purified by column chromatography to give the desired diselenides [1].



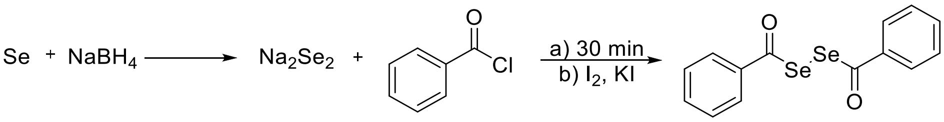
Selenium (2 mmol) was added to a stirred solution of sodium borohydride (5 mmol) in ethanol (5 mL) at 0 °C. Stirring was continued for 30 min at this temperature. Then, selenium (2 mmol) was added to the reaction mixture and stirred for 30 min at 0 °C. Finally, iodides (7 mmol) was added over a period of 5 min. After stirring for a further hour at room temperature, the reaction mixture was extracted with hexane and washed with water, dried over Na_2SO_4, and concentrated in vacuo. [2].



n-Butyl lithium (8 mmol) was added to the THF (15 mL) solution of bromobenzene (2 mmol) at - 40 °C. Stirring was continued for 2 hours at this temperature. Then, selenium (8 mmol) was added to the reaction mixture and stirred for 2 hours at - 40 °C. After stirring for a further hour at room temperature, water was added to the reaction. the reaction was concentrated, and the residue was purified by silica gel column chromatography separation to give the desired diselenides. [3].

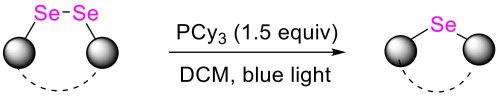


To a solution of bromide (1 mmol) in ethanol (5 mL) was added potassium selenocyanate (1.2 mmol). After being stirred for 15 min, the reaction mixture was added with an aqueous solution of sodium hydroxide (3 M 0.8 mL). After 1 h, the mixture was added with water and then extracted with Et_2O. The organic layer was washed with brine, dried over anhydrous magnesium sulfate. The residue was purified by silica gel column chromatography separation to give the desired diselenides. [4].



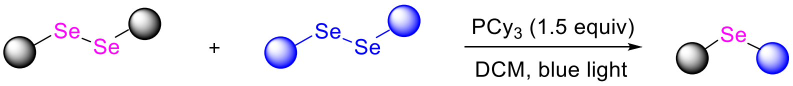
Selenium (2 mmol) was added to a stirred solution of sodium borohydride (2.4 mmol) in ethanol (3 mL) at 0 °C. Stirring was continued for 30 min at this temperature. Then, benzoyl chloride (2 mmol) was added to the reaction mixture and stirred for 30 min at 0 °C. Stirring was continued for 30 min at this temperature. Finally, a solution of iodine (1 mmol) and potassium iodide (0.4 mmol) was added to the reaction mixture and stirred for 30 min at 0 °C. After reaction finished, the organic phase was washed with 1 % $NaHCO_3$ solution. The reaction mixture was then concentrated, and purified by silica gel column chromatography separation to give the desired diselenides [5].

General procedure A for deselenizative reaction of diselenides



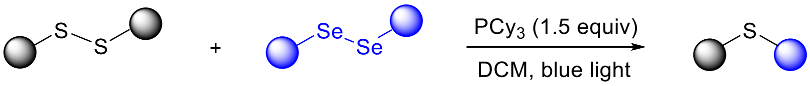
Diselenide (0.2 mmol) and PCy_3 (0.3 mmol) were placed in a 10.0 mL quarz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (2.0 mL) was added. The quarz test tube was sealed and placed approximately 3 cm from a 50W blue LED (λ=450 nm). This mixture was then stirred and irradiated for 2 h at room temperature. The reaction mixture was evaporated and purified directly by column chromatography to afford the product.

General procedure B for deselenizative cross-coupling reaction of two different diselenides



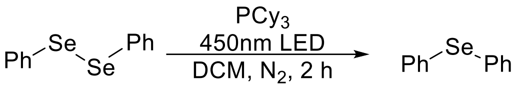
Diselenide (0.1 mmol), another diselenide (0.1 mmol) and PCy_3 (0.3 mmol) were placed in a 10.0 mL quarz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (2.0 mL) was added. The quarz test tube was sealed and placed approximately 3 cm from a 50W blue LED (λ=450 nm). This mixture was then stirred and irradiated for 2 h at room temperature. The reaction mixture was evaporated and purified directly by column chromatography to afford the product.

General procedure C for deselenizative cross-coupling reaction between a diselenide and a disulfide



Diselenide (0.1 mmol), disuifide (0.1 mmol) and PCy_3 (0.3 mmol) were placed in a 10.0 mL quarz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (2.0 mL) was added. The quarz test tube was sealed and placed approximately 3 cm from a 50W blue LED (λ=450 nm). This mixture was then stirred and irradiated for 2 h at room temperature. The reaction mixture was evaporated and purified directly by column chromatography to afford the product.

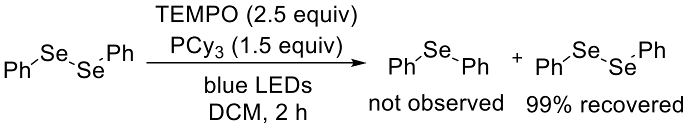
3. Gram-Scale synthesis



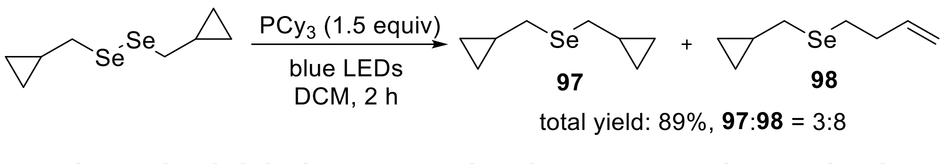
Diphenyl diselenide (5 mmol) and PCy_3 (15 mmol) were placed in a 30 mL quarz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (10 mL) was added. The quarz test tube was sealed and placed approximately 3 cm from a 50W blue LED (λ = 450 nm). This mixture was then stirred and irradiated for 2 h at room temperature. The reaction mixture was evaporated and purified directly by column chromatography to afford the product (90%, 1.05 g).

4. Mechanistic studies

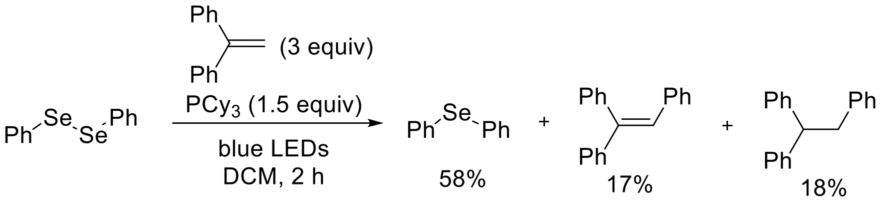
4.1 Radical inhibition experiment



Diphenyl diselenide (0.2 mmol), PCy_3 (0.3 mmol) and TEMPO (0.5 mmol) were placed in a 10 mL quarz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (2 mL) was added. The quarz test tube was sealed and placed approximately 3 cm from a 50 W blue LED (λ=450 nm). This mixture was then stirred and irradiated for 2 h at room temperature. No target product generation was detected by GC-MS.



1,2-Bis(cyclopropylmethyl)diselane (0.2 mmol) and PCy_3 (0.3 mmol) were placed in a 10.0 mL quarz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (2 mL) was added. The quarz test tube was sealed and placed approximately 3 cm from a 50W blue LED (λ=450 nm). The reaction mixture was detected by GC-MS. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.89 – 5.76 (m, 1H), 5.10 – 4.99 (m, 2H), 2.71 – 2.64 (m, 2H), 2.60 – 2.50 (m, 3H), 2.46 – 2.39 (m, 2H), 1.24 (s, 3H), 1.11 – 0.99 (m, 1.5H), 0.59 (d, *J* = 7.6 Hz, 6H), 0.20 (d, *J* = 4.5 Hz, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 137.6, 115.5, 35.0, 29.3, 29.7, 22.6, 12.2, 12.1, 6.64, 6.59.



Diphenyl diselenide (0.2 mmol), PCy_3 (0.3 mmol) and 1,1-diphenylethylene (0.6 mmol) were placed in a 10 mL quarz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (2 mL) was added. The quarz test tube was sealed and placed approximately 3 cm from a 50W blue LED (λ=450 nm). This mixture was then stirred and irradiated for 2 h at room temperature. The reaction mixture was detected by GC-MS.

4.2 Ultraviolet absorption and fluorescence quenching experiments

UV/Vis absorption spectra were recorded on a SHIMADZU UV-2600 spectrophotometer. 1*10^{-3} mol/L^{-1} PhSeSePh and PCy_3 solutions using DCM as the solvent were prepared. The samples were measured in Surui® fluorescence quartz cuvettes (chamber volume = 3.500 mL, H × W × D = 48 mm × 12.5 mm × 12.5 mm, path length = 10 mm).

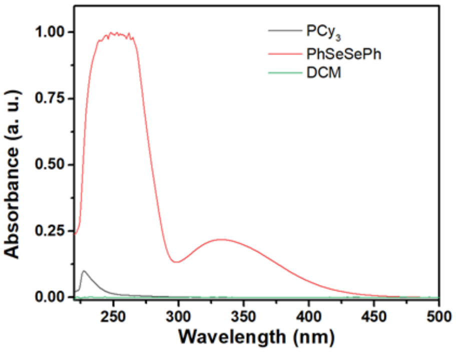


Fig. S2 UV-Vis absorption spectrum

The fluorescence spectrum is recorded on the burker F-7000. Solution with PhSeSePh and different concentrations PCy_3 were prepared. The samples were measured in Surui® fluorescence quartz cuvettes (chamber volume = 3.500 mL, H × W × D = 48 mm × 12.5 mm × 12.5 mm, path length = 10 mm). The following parameters were set: data interval = 0.5 nm, scan-speed = 500 nm/min, excitation wavelength λex = 330 nm.

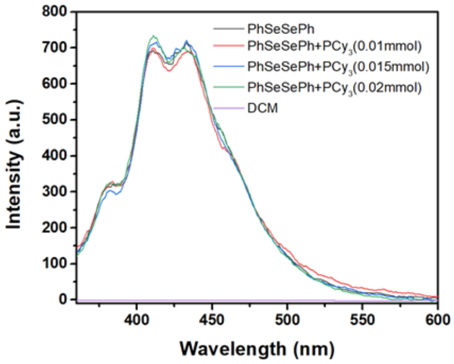


Fig. S3 Fluorescence spectrum

4.3 Light on/off experiment

To study the necessity of continuous irradiation with visible light for the progress of the reaction, the reaction proceeding was monitored by GC-MS using 1,2-diphenyldiselane as an internal standard before and after light irradiation and dark periods.

The control experiments shown below with successive intervals of irradiation and dark periods did result in interruption of the reaction progress in the absence of light, demonstrating that light is a necessary component for the reaction after triggering the reaction in the first part.

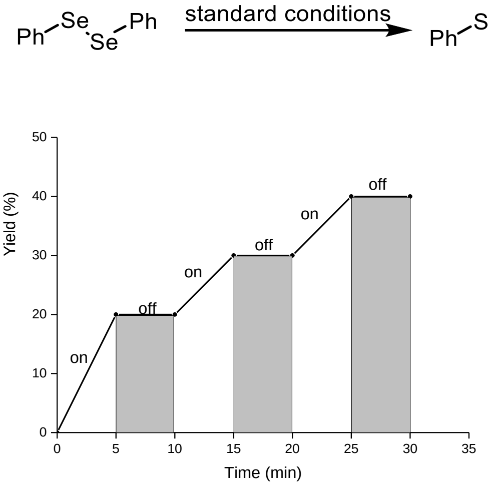


Fig. S4 Light on-off experiment.

5. Computational details

The density functional theory (DFT) method was used to optimize geometries of XXX. The bond dissociation energy (BDE)[6]was calculated based on the homolytic reaction. The geometries of all radicals were also optimized. Moreover, the natural bond orbital (NBO)[7] analysis was performed to obtain the atomic charge of all radicals. All the calculations were carried out at the level of M06-2X[8]/6-311+g* within Gaussian 09 package.

PCy_3

Zero-point correction= 0.487906 (Hartree/Particle)

Thermal correction to Energy= 0.507526

Thermal correction to Enthalpy= 0.508470

Thermal correction to Gibbs Free Energy= 0.440402

Sum of electronic and zero-point Energies= -1046.461974

Sum of electronic and thermal Energies= -1046.442354

Sum of electronic and thermal Enthalpies= -1046.441410

Sum of electronic and thermal Free Energies= -1046.509478

$PhCH_2SeSeCH_2Ph$

Zero-point correction= 0.241117 (Hartree/Particle)

Thermal correction to Energy= 0.256464

Thermal correction to Enthalpy= 0.257408

Thermal correction to Gibbs Free Energy= 0.194927

Sum of electronic and zero-point Energies= -5344.692057

Sum of electronic and thermal Energies= -5344.676710

Sum of electronic and thermal Enthalpies= -5344.675766

Sum of electronic and thermal Free Energies= -5344.738246

$PhCH_2SeCH_2Ph$

Zero-point correction= 0.240076 (Hartree/Particle)

Thermal correction to Energy= 0.253663

Thermal correction to Enthalpy= 0.254607

Thermal correction to Gibbs Free Energy= 0.195731

Sum of electronic and zero-point Energies= -2943.114040

Sum of electronic and thermal Energies= -2943.100453

Sum of electronic and thermal Enthalpies= -2943.099509

Sum of electronic and thermal Free Energies= -2943.158385

$PhCH_2SePCy_3$

Zero-point correction= 0.609498 (Hartree/Particle)

Thermal correction to Energy= 0.637741

Thermal correction to Enthalpy= 0.638685

Thermal correction to Gibbs Free Energy= 0.549698

Sum of electronic and zero-point Energies= -3718.789620

Sum of electronic and thermal Energies= -3718.761377

Sum of electronic and thermal Enthalpies= -3718.760433

Sum of electronic and thermal Free Energies= -3718.849420

PhSeSePh

Zero-point correction= 0.182876 (Hartree/Particle)

Thermal correction to Energy= 0.195967

Thermal correction to Enthalpy= 0.196912

Thermal correction to Gibbs Free Energy= 0.138515

Sum of electronic and zero-point Energies= -5266.134974

Sum of electronic and thermal Energies= -5266.121882

Sum of electronic and thermal Enthalpies= -5266.120938

Sum of electronic and thermal Free Energies= -5266.179334

PhSeCH_2Ph

Zero-point correction= 0.211001 (Hartree/Particle)

Thermal correction to Energy= 0.222602

Thermal correction to Enthalpy= 0.223546

Thermal correction to Gibbs Free Energy= 0.169537

Sum of electronic and zero-point Energies= -2903.836546

Sum of electronic and thermal Energies= -2903.824946

Sum of electronic and thermal Enthalpies= -2903.824002

Sum of electronic and thermal Free Energies= -2903.878011

PhSePCy$_3$

Zero-point correction= 0.580742 (Hartree/Particle)

Thermal correction to Energy= 0.607899

Thermal correction to Enthalpy= 0.608844

Thermal correction to Gibbs Free Energy= 0.521173

Sum of electronic and zero-point Energies= -3679.513080

Sum of electronic and thermal Energies= -3679.485923

Sum of electronic and thermal Enthalpies= -3679.484979

Sum of electronic and thermal Free Energies= -3679.572649

PhSePh

Zero-point correction= 0.182636 (Hartree/Particle)

Thermal correction to Energy= 0.193718

Thermal correction to Enthalpy= 0.194663

Thermal correction to Gibbs Free Energy= 0.142211

Sum of electronic and zero-point Energies= -2864.561040

Sum of electronic and thermal Energies= -2864.549957

Sum of electronic and thermal Enthalpies= -2864.549013

Sum of electronic and thermal Free Energies= -2864.601465

SePCy$_3$

Zero-point correction= 0.490319 (Hartree/Particle)

Thermal correction to Energy= 0.510946

Thermal correction to Enthalpy= 0.511890

Thermal correction to Gibbs Free Energy= 0.441102

Sum of electronic and zero-point Energies= -3448.061467

Sum of electronic and thermal Energies= -3448.040840

Sum of electronic and thermal Enthalpies= -3448.039896

Sum of electronic and thermal Free Energies= -3448.110684

PhCH$_2$Se$^•$ radical

Zero-point correction= 0.119555 (Hartree/Particle)

Thermal correction to Energy= 0.126578

Thermal correction to Enthalpy= 0.127522

Thermal correction to Gibbs Free Energy= 0.085495

Sum of electronic and zero-point Energies= -2672.302625

Sum of electronic and thermal Energies= -2672.295603

Sum of electronic and thermal Enthalpies= -2672.294659

Sum of electronic and thermal Free Energies= -2672.336686

$PhCH_2^\bullet$ radical

Zero-point correction= 0.115176 (Hartree/Particle)

Thermal correction to Energy= 0.120857

Thermal correction to Enthalpy= 0.121801

Thermal correction to Gibbs Free Energy= 0.085500

Sum of electronic and zero-point Energies= -270.727485

Sum of electronic and thermal Energies= -270.721803

Sum of electronic and thermal Enthalpies= -270.720859

Sum of electronic and thermal Free Energies= -270.757160

$PhSe^\bullet$ radical

Zero-point correction= 0.090785 (Hartree/Particle)

Thermal correction to Energy= 0.096537

Thermal correction to Enthalpy= 0.097482

Thermal correction to Gibbs Free Energy= 0.059137

Sum of electronic and zero-point Energies= -2633.024554

Sum of electronic and thermal Energies= -2633.018801

Sum of electronic and thermal Enthalpies= -2633.017857

Sum of electronic and thermal Free Energies= -2633.056201

$Ph^\bullet$ radical

Zero-point correction= 0.088129 (Hartree/Particle)

Thermal correction to Energy= 0.092472

Thermal correction to Enthalpy= 0.093416

Thermal correction to Gibbs Free Energy= 0.060104

Sum of electronic and zero-point Energies= -231.417821

Sum of electronic and thermal Energies= -231.413478

Sum of electronic and thermal Enthalpies= -231.412534

Sum of electronic and thermal Free Energies= -231.445846

$PhCO^{\bullet}$ radical

```
Zero-point correction=                           0.098259 (Hartree/Particle)
 Thermal correction to Energy=                    0.104568
 Thermal correction to Enthalpy=                  0.105512
 Thermal correction to Gibbs Free Energy=        0.067031
 Sum of electronic and zero-point Energies=          -344.759101
 Sum of electronic and thermal Energies=              -344.752792
 Sum of electronic and thermal Enthalpies=            -344.751848
 Sum of electronic and thermal Free Energies=         -344.790329
```

$^{\bullet}SPCy_3$ radical

```
Zero-point correction=                           0.491215 (Hartree/Particle)
 Thermal correction to Energy=                    0.512146
 Thermal correction to Enthalpy=                  0.513090
 Thermal correction to Gibbs Free Energy=        0.442099
 Sum of electronic and zero-point Energies=          -1444.689471
 Sum of electronic and thermal Energies=              -1444.668540
 Sum of electronic and thermal Enthalpies=            -1444.667596
 Sum of electronic and thermal Free Energies=         -1444.738587
```

$PhCOSePCy_3$

```
Zero-point correction=                           0.591243 (Hartree/Particle)
 Thermal correction to Energy=                    0.620088
 Thermal correction to Enthalpy=                  0.621032
 Thermal correction to Gibbs Free Energy=        0.530292
 Sum of electronic and zero-point Energies=          -3792.830300
 Sum of electronic and thermal Energies=              -3792.801455
 Sum of electronic and thermal Enthalpies=            -3792.800511
 Sum of electronic and thermal Free Energies=         -3792.891251
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$PhCOSPCy_3$

```
Zero-point correction=                           0.591486 (Hartree/Particle)
 Thermal correction to Energy=                    0.620095
 Thermal correction to Enthalpy=                  0.621040
 Thermal correction to Gibbs Free Energy=        0.530568
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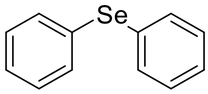
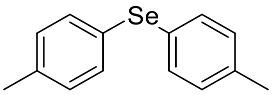
6. Characterization of products in details

Diphenylselane (1)



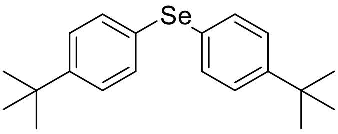
1, obtained by the **General method A**, is a colorless liquid (44.3 mg, 95%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 – 7.35 (m, 4H), 7.34 – 7.17 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 132.9, 131.1, 129.3, 127.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_{11}Se$: 235.0020; found: 235.0027.

di-*p*-tolylselane (2)



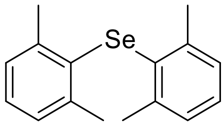
2, obtained by the **General method A**, is a succinite liquid (35 mg, 67%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.9 Hz, 4H), 7.08 (d, *J* = 7.6 Hz, 4H), 2.33 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.1, 132.9, 130.1, 127.7, 21.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}Se$: 263.0333; found: 263.0339 .

bis(4-(tert-butyl)phenyl)selane (3)



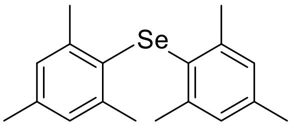
3, obtained by the **General method A**, is a succinite solid (53.9 mg, 78%), melting point: 96-97°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.5 Hz, 4H), 7.29 (d, *J* = 8.5 Hz, 4H), 1.30 (s, 18H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.3, 132.7, 127.6, 126.3, 34.5, 31.2; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{20}H_{27}Se$: 347.1272; found: 347.1260.

bis(2,6-dimethylphenyl)selane (4)



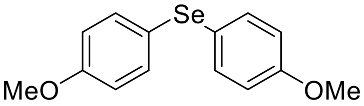
4, obtained by the **General method A**, is a succinite solid (53.2 mg, 92%), melting point: 50 – 51°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.10 – 7.02 (m, 2H), 7.00 (d, J = 7.5 Hz, 4H), 2.27 (s, 12H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.4, 132.9, 128.0, 127.3, 23.6; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{16}H_{19}Se$: 291.0646; found: 291.0652.

Dimesitylselane (5)



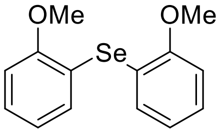
5, obtained by the **General method A**, is a succinite solid (60.9 mg, 96%), melting point: 93 – 94°C; **^{1}H NMR** δ 6.82 (s, 4H), 2.23 (s, 12H), 2.22 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.3, 136.9, 129.4, 128.9, 23.5, 20.8; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{18}H_{23}Se$: 319.0959; found: 319.0965.

bis(4-methoxyphenyl)selane (6)



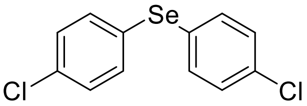
6, obtained by the **General method A**, is a succinite solid (35.8 mg, 61%), melting point: 47-48°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.4 Hz, 4H), 6.81 (d, *J* = 8.5 Hz, 4H), 3.78 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.2, 134.6, 122.1, 115.0, 55.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}O_2Se$: 295.0232; found: 295.0240.

bis(2-methoxyphenyl)selane (7)



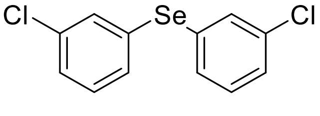
7, obtained by the **General method A**, is a succinite solid (35.2 mg, 60%), melting point: 48 – 49°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.35 – 7.05 (m, 4H), 7.05 – 6.71 (m, 4H), 3.86 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 158.1, 133.7, 128.7, 121.5, 118.8, 110.6, 55.9; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}O_2Se$: 295.0232; found: 295.0241.

bis(4-chlorophenyl)selane (8)



8, obtained by the **General method A**, is a succinite solid (44.1 mg, 73%), melting point: 94 – 95°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.4 Hz, 4H), 7.24 (d, *J* = 8.5 Hz, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 134.3, 133.9, 129.6, 129.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_9Cl_2Se$: 302.9241; found: 302.9243.

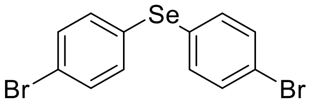
bis(3-chlorophenyl)selane (9)



9, obtained by the **General method A**, is a succinite solid (35.2 mg, 60%), melting point: 94 – 95°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.45 (s, 2H), 7.37 – 7.18 (m, 6H); **^{13}C**

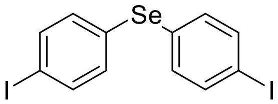
NMR (125 MHz, $CDCl_3$) δ 135.1, 132.7, 132.0, 131.1, 130.5, 128.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_9Cl_2Se$: 302.9241; found: 302.9248.

bis(4-bromophenyl)selane (10)



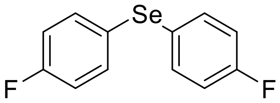
10, obtained by the **General method A**, is a succinite solid (57.9 mg, 74%), melting point: 114 – 115°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47-7.35 (m, 4H), 7.30 (d, *J* = 8.4 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 134.5, 133.3, 132.5, 132.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_9Br_2Se$: 390.8231; found: 390.8238.

bis(4-iodophenyl)selane (11)



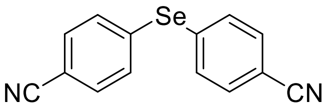
11, obtained by the **General method A**, is a succinite solid (51.4 mg, 53%), melting point: 140– 141°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 4H), 7.16 (d, *J* = 8.0 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.4, 134.7, 130.5, 93.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_9I_2Se$: 484.7797; found: 484.7791.

bis(4-fluorophenyl)selane (12)



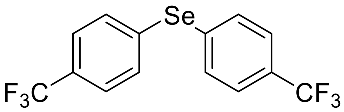
12, obtained by the **General method A**, is a succinite liquid (44.7 mg, 83%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 8.7, 5.4 Hz, 4H), 6.98 (t, *J* = 8.7 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.5 (d, *J* = 247.6 Hz), 134.9 (d, *J* = 7.9 Hz), 125.7 (d, *J* = 3.4 Hz), 116.6 (d, *J* = 21.6 Hz); **^{19}F NMR** (471 MHz, CDCl3) δ -113.10; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_9F_2Se$: 270.9832; found: 270.9836.

4,4'-selenodibenzonitrile (13)



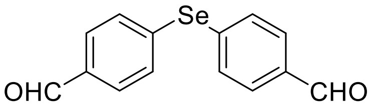
13, obtained by the **General method A**, is a succinite solid (28.3 mg, 50%), melting point: 103 – 105°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.2 Hz, 4H), 7.55 (d, *J* = 8.0 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.3, 132.7, 130.5, 118.2, 111.4; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_9N_2Se$: 284.9925; found: 284.9931.

Bis (4-(trifluoromethyl) phenyl) selane (14)



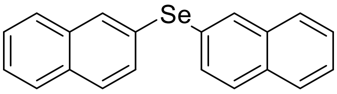
14, obtained by the **General method A**, is a succinite liquid (44.3 mg, 60%).**^{1}H NMR** (500 MHz, CDCl$_3$) δ 7.70 (d, *J* = 7.3 Hz, 6H), 7.52 (d, *J* = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 134.9, 130.8, 126.1 (q, *J* = 3.6 Hz), 123.9 (d, *J* = 272.1 Hz); **^{19}F NMR** (471 MHz, CDCl$_3$) δ -62.68; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{14}H_9F_6Se$: 370.9768; found: 370.9764.

4,4'-selenodibenzaldehyde (15)



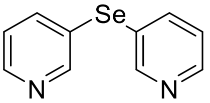
15, obtained by the **General method A**, is a succinite solid (28.3 mg, 62%), melting point: 73 – 76°C; **^{1}H NMR** (500 MHz, CDCl$_3$) δ 9.99 (s, 2H), 7.80 (d, *J* = 8.1 Hz, 4H), 7.61 (d, *J* = 8.1 Hz, 4H). **^{13}C NMR** (125 MHz, CDCl$_3$) δ 191.3, 138.8, 135.5, 133.0, 130.5; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{14}H_{11}O_2Se$: 290.9919; found: 290.9912.

di(naphthalen-2-yl) selane (16)



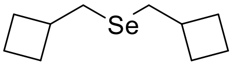
16, obtained by the **General method A**, is a succinite solid (45.3 mg, 68%), melting point: 138 – 139°C; **^{1}H NMR** (400 MHz, CDCl$_3$) δ 8.00 (s, 2H), 7.81-7.66 (m 7H), 7.58 – 7.39 (m, 6H); **^{13}C NMR** (100 MHz, CDCl$_3$) δ 134.0, 132.4, 131.9, 130.4, 128.8, 128.5, 127.8, 127.4, 126.5, 126.2; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{20}H_{15}Se$: 335.0333; found: 335.0309.

Di (pyridin-3-yl) selane (17)



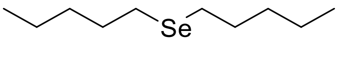
17, obtained by the **General method A**, is a succinite solid (22 mg, 47%).**^{1}H NMR** (500 MHz, CDCl$_3$) δ 8.83 – 8.61 (m, 2H), 8.53 – 8.42 (m, 2H), 7.93 – 7.62 (m, 2H), 7.22 – 7.13 (m, 2H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 152.4, 149.2, 140.1, 127.7, 124.2; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{10}H_9N_2Se$: 236.9925; found: 236.9934.

Bis (cyclobutylmethyl) selane (18)



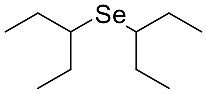
18, obtained by the **General method A**, is a colorless liquid (33.5 mg, 70%).**^{1}H NMR** (400 MHz, CDCl$_3$) δ 2.87 – 2.56 (m, 4H), 2.56 – 2.31 (m, 2H), 2.10 (s, 4H), 1.81 (s, 4H), 1.73 – 1.53 (m, 4H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 36.4, 30.8, 28.9, 17.5; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{10}H_{19}Se$: 219.0646; found: 219.0650.

Dipentylselane (19)



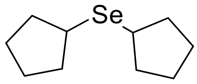
19, obtained by the **General method A**, is a colorless liquid (38 mg, 86%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.54 (t, *J* = 7.5 Hz, 4H), 1.78 – 1.52 (m, 4H), 1.44 – 1.23 (m,8H), 0.89 (t, *J* = 7.0 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 32.2, 30.4, 23.9, 22.2, 14.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{10}H_{23}Se$: 223.0959; found: 223.0963.

Di (pentan-3-yl) selane (20)



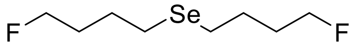
20, obtained by the **General method A**, is a colorless liquid (23.9 mg, 54%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.68 (p, *J* = 6.4 Hz, 2H), 1.67 (td, *J* = 13.7, 7.2 Hz, 8H), 0.98 (t, *J* = 7.3 Hz, 12H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ45.0, 28.5, 12.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{10}H_{23}Se$: 223.0959; found: 223.0963.

Dicyclopentylselane (21)



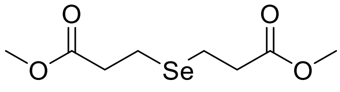
21, obtained by the **General method A**, is a colorless liquid (26.1 mg, 60%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.25 (p, *J* = 7.0 Hz, 2H), 2.55 – 1.93 (m, 4H), 1.80-1.69 (m, 4H), 1.74-1.55 (m, 8H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ37.5, 34.8, 25.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{10}H_{19}Se$: 219.0646; found: 219.0650.

Bis (4-fluorobutyl) selane (22)



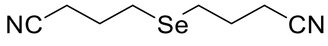
22, obtained by the **General method A**, is a colorless liquid (36.7 mg, 80%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.51 (d, *J* = 5.6 Hz, 2H), 4.40 (d, *J* = 0.9 Hz, 2H), 2.59 (s, 4H), 1.91 – 1.70 (m, 8H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 83.5 (d, *J* = 164.9 Hz), 30.5 (d, *J* = 19.7 Hz), 26.3 (d, *J* = 4.7 Hz), 23.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_8H_{17}F_2Se$: 231.0458; found: 231.0464.

dimethyl 3,3'-selenodipropionate (23)



23, obtained by the **General method A**, is a succinite liquid (45.1 mg, 89%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.69 (d, *J* = 12.8 Hz, 6H), 2.84 – 2.68 (m, 8H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.5, 51.7, 35.4, 17.8; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_8H_{15}O_4Se$: 255.0130; found: 255.0138.

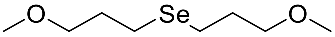
4,4'-selenodibutanenitrile (24)



24, obtained by the **General method A**, is a succinite liquid (37.9 mg, 88%).**^{1}H NMR**

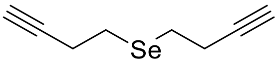
(400 MHz, $CDCl_3$) δ 2.68 (t, *J* = 7.1 Hz, 4H), 2.51 (t, *J* = 7.0 Hz, 4H), 1.99 (p, *J* = 7.0 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 118.9, 25.8, 22.1, 17.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_8H_{13}N_2Se$: 217.0238; found: 217.0232.

Bis (3-methoxypropyl) selane (25)



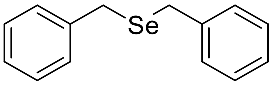
25, obtained by the **General method A**, is a succinite liquid (38.3 mg, 85%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.44 (t, *J* = 6.2 Hz, 4H), 3.33 (s, 6H), 2.62 (t, *J* = 7.3 Hz, 4H), 1.98 – 1.85 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ72.0, 58.6, 30.5, 20.4; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_8H_{19}O_2Se$: 227.0545; found: 227.0550.

Di (but-3-yn-1-yl) selane (26)



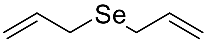
26, obtained by the **General method A**, is a succinite liquid (35.2 mg, 95%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.77 (t, *J* = 7.4 Hz, 4H), 2.66 – 2.52 (m, *J* = 7.1, 2.3 Hz, 4H), 2.04 (t, *J* = 2.6 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 83.1, 69.4, 21.9, 20.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_8H_{11}Se$: 187.0020; found: 187.0016.

Dibenzylselane (27)



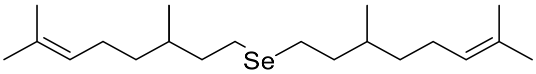
27, obtained by the **General method A**, is a succinite liquid (38.7 mg, 74%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.59 – 6.92 (m, 10H), 3.82 (s, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 139.0, 129.0, 128.4, 127.1, 32.6; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}Se$: 263.0333; found: 263.0339.

Diallylselane (28)



28, obtained by the **General method A**, is a colorless liquid (27.7 mg, 86%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.88 (td, *J* = 17.5, 7.6 Hz, 2H), 5.19 – 4.82 (m, 4H), 3.16 (d, *J* = 6.2 Hz, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 135.0, 116.4, 25.4; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_6H_{11}Se$: 163.0020; found: 163.0011.

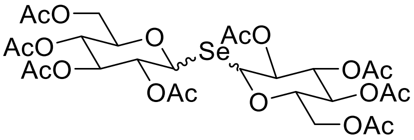
Bis (3,7-dimethyloct-6-en-1-yl) selane (29)



29, obtained by the **General method A**, is a colorless liquid (55.8 mg, 78%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.09 (t, *J* = 6.5 Hz, 2H), 3.02 – 2.84 (m, 4H), 2.06 – 1.92 (m, 4H), 1.80 – 1.71 (m, 2H), 1.68 (s, 6H), 1.61 (s, 6H), 1.59 – 1.48 (m, 4H), 1.40 – 1.32 (m, 2H), 1.21 – 1.12 (m, 2H), 0.91 (d, *J* = 5.9 Hz, 6H);**^{13}C NMR** (100 MHz, $CDCl_3$) δ 131.2, 124.7, 37.8, 36.7, 32.9, 25.7, 25.4, 21.4, 19.2, 17.6;

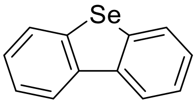
HRMS (ESI) m/z $[M+H]^+$ Calcd. for $C_{20}H_{39}Se$: 359.2211; found: 359.2190.

(2S,3S,4R,5S)-2-(acetoxymethyl)-6-(((3R,4S,5R,6R)-3,4,5-triacetoxy-6-(acetoxymethyl) tetrahydro-2H-pyran-2-yl)selanyl)tetrahydro-2H-pyran-3,4,5-triyl triacetate (30)



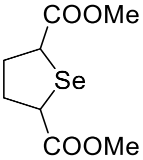
30, obtained by the **General method A**, is a succinite liquid (99.4 mg, 67%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.46 (s, 2H), 5.38 – 5.27 (m, 2H), 5.14 – 4.86 (m, 3H), 4.47 (s, 1H), 4.26 – 3.90 (m, 6H), 2.24 – 1.87 (m, 24H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 170.3, 170.1, 167.0, 169.5, 80.8, 75.4, 71.4, 69.2, 67.1, 60.8, 20.7, 20.5, 20.41, 20.35; **HRMS** (ESI) m/z $[M+Na]^+$ Calcd. for $C_{28}H_{38}NaO_{18}Se$: 765.1116; found: 765.1235.

Dibenzo [*b,d*] selenophene (31)



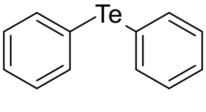
31, obtained by the **General method A**, is a succinite solid (30.1 mg, 65%), melting point: 73 – 74°C;**^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.14 (d, J = 7.8 Hz, 2H), 7.90 (d, J = 7.9 Hz, 2H), 7.47 (t, J = 7.5 Hz, 2H), 7.40 (t, J = 7.5 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.3, 138.2, 126.8, 126.0, 124.8, 122.8; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_9Se$: 232.9864; found: 232.9869.

dimethyl tetrahydroselenophene-2,5-dicarboxylate (32)



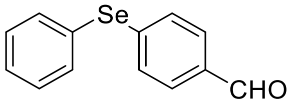
32, obtained by the **General method A**, is a succinite liquid (32.1 mg, 64%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.16 (t, J = 5.5 Hz, 2H), 3.67 (s, 6H), 2.60 (td, J = 8.1, 3.5 Hz, 2H), 2.18 – 2.08 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.3, 52.3, 42.1, 34.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_8H_{13}O_4Se$: 252.9974; found: 252.9980.

Diphenyltellane(33)



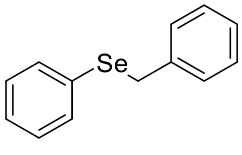
33, obtained by the **General method A**, is a succinite liquid (46.8 mg, 83%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.04 – 7.63 (m, 1H), 7.40 – 6.95 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 137.58, 129.26, 128.05, 107.92; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_{11}Te$: 284.9917; found: 284.9927.

4-(phenylselanyl)benzaldehyde (34)



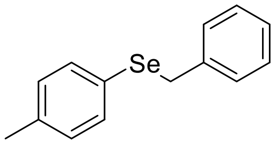
34, obtained by the **General method B**, is a succinite liquid (46.8 mg, 30%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.69 (d, *J* = 8.1 Hz, 2H), 7.62 (d, *J* = 7.0 Hz, 2H), 7.50 – 7.29 (m, 5H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 191.4, 142.8, 135.5, 134.4, 130.1, 129.9, 128.9, 127.9; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{11}OSe$: 262.9970; found: 262.9976.

Benzyl (phenyl) selane (35)



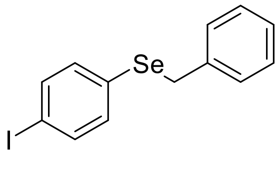
35, obtained by the **General method B**, is a succinite liquid (40 mg, 81%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 (s, 2H), 7.21 (dd, *J* = 13.6, 6.8 Hz, 8H), 4.09 (d, *J* = 7.9 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.6, 133.5, 130.4, 128.9, 128.8, 128.4, 127.3, 126.8, 32.2; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{13}N_2Se$: 249.0177; found: 249.0182.

Benzyl (*p*-tolyl) selane (36)



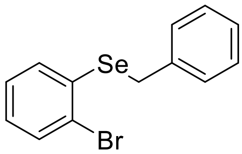
36, obtained by the **General method B**, is a succinite liquid (37.1 mg, 71%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.0 Hz, 2H), 7.27 – 7.09 (m, *J* = 18.0, 7.3 Hz, 5H), 7.04 (d, *J* = 7.7 Hz, 2H), 4.05 (s, 2H), 2.31 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 138.9, 137.4, 134.0, 129.8, 128.8, 128.4, 126.7, 126.5, 32.5, 21.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}Se$: 263.0333; found: 263.0338.

Benzyl (4-iodophenyl) selane (37)



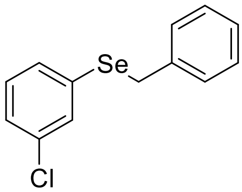
37, obtained by the **General method B**, is a succinite solid (56 mg, 75%), melting point: 80 – 81℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 (s, 2H), 7.36 – 6.97 (m, *J* = 30.1, 11.1 Hz, 7H), 4.07 (d, *J* = 8.9 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 138.2, 137.9, 135.2, 130.1, 128.8, 128.2, 127.0, 93.0, 32.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}ISe$: 374.9143; found: 374.9149.

Benzyl (2-bromophenyl) selane (38)



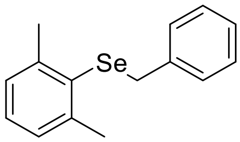
38, obtained by the **General method B**, is a succinite solid (58.7 mg, 90%), melting point: 68 – 70°C; **^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.52 (d, *J* = 7.9 Hz, 1H), 7.38 – 7.12 (m, 7H), 7.12 – 6.94 (m, *J* = 7.6 Hz, 1H), 4.17 (s, 2H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 136.9, 134.4, 132.8, 131.6, 129.0, 128.6, 127.74, 127.69, 127.1, 125.6, 31.4; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{13}H_{12}BrSe$: 326.9282; found: 326.9295.

Benzyl (3-chlorophenyl) selane (39)



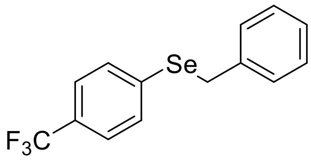
39, obtained by the **General method B**, is a succinite solid (34.4 mg, 61%), melting point: 55 – 56°C; **^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.41 (s, 1H), 7.35 – 7.04 (m, 8H), 4.12 (s, 2H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 138.0, 134.5, 132.9, 132.0, 131.3, 129.9, 128.9, 128.5, 127.4, 127.1, 32.2; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{13}H_{12}ClSe$: 282.9787; found: 282.9794.

Benzyl (2,6-dimethylphenyl) selane (40)



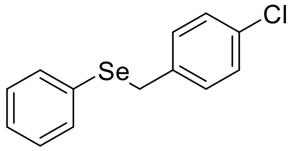
40, obtained by the **General method B**, is a succinite liquid (42.4 mg, 77%).**^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.21 – 6.82 (m, *J* = 25.1, 21.6, 5.9 Hz, 8H), 3.82 (s, 2H), 2.40 (s, 6H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 143.7, 139.4, 131.1, 128.54, 128.52, 128.2, 127.4, 126.6, 31.4, 24.4; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{15}H_{17}Se$: 277.0490; found: 277.0498.

Benzyl (4-(trifluoromethyl) phenyl) selane (41)



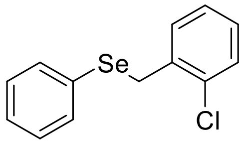
41, obtained by the **General method B**, is a succinite liquid (38.5 mg, 61%).**^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.60 – 7.36 (m, 4H), 7.36 – 6.66 (m, 5H), 4.17 (s, 2H); **^{13}C NMR** (100 MHz, CDCl$_3$) δ 137.6, 132.4, 128.9, 128.6, 127.2, 125.6 (q, *J* = 3.8 Hz), 124.1 (d, *J* = 272.0 Hz), 31.8; **^{19}F NMR** (471 MHz, CDCl$_3$) δ -62.57; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{14}H_{12}F_3Se$: 317.0051; found: 317.0047.

(4-chlorobenzyl)(phenyl)selane (42)



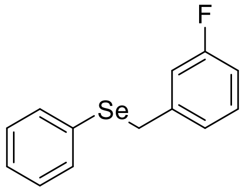
42, obtained by the **General method B**, is a succinite solid (43.4 mg, 77%), melting point: 55 – 56°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 – 7.33 (m, 2H), 7.27 – 7.15 (m, 5H), 7.13 – 7.05 (m, 2H), 4.03 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 137.3, 133.9, 132.5, 130.1, 129.8, 129.0, 128.5, 127.6, 31.4; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}ClSe$: 282.9787; found: 282.9794.

(2-chlorobenzyl)(phenyl)selane (43)



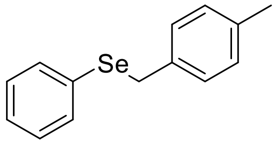
43, obtained by the **General method B**, is a succinite solid (36.1 mg, 64%), melting point: 55 – 56°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 – 7.43 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.28 – 7.19 (m, 3H), 7.17 – 6.96 (m, 3H), 4.17 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.6, 134.4, 133.8, 130.6, 129.8, 129.7, 128.9, 128.2, 127.6, 126.6, 30.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}ClSe$: 282.9787; found: 282.9794.

(3-fluorobenzyl)(phenyl)selane (44)



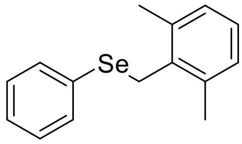
44, obtained by the **General method B**, is a succinite liquid (26.5 mg, 50%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 – 7.34 (m, 2H), 7.29 – 7.06 (m, 4H), 7.02 – 6.76 (m, 3H), 4.05 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.7 (d, *J* = 245.9 Hz), 141.3 (d, *J* = 7.5 Hz), 133.8, 129.8 (d, *J* = 8.0 Hz), 129.0, 127.6,124.5 (d, *J* = 2.8 Hz), 115.7 (d, *J* = 21.7 Hz), 113.7 (d, *J* = 21.1 Hz), 31.6 (d, *J* = 1.7 Hz).;**^{19}F NMR** (471 MHz, $CDCl_3$) δ -113.12;**HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}FSe$: 267.0083; found: 267.0090.

(4-methylbenzyl)(phenyl)selane (45)



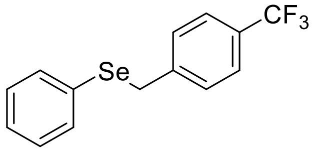
45, obtained by the **General method B**, is a succinite liquid (37.6 mg, 72%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 – 7.32 (m, 2H), 7.32 – 7.16 (m, 3H), 7.07 (dd, *J* = 22.1, 8.0 Hz, 4H), 4.08 (s, 2H), 2.30 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.5, 135.4, 133.3, 130.7, 129.1, 128.9, 128.7, 127.1, 32.0, 21.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}Se$: 263.0333; found: 263.0338.

(2,6-dimethylbenzyl)(phenyl)selane (46)



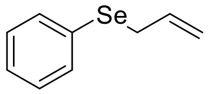
46, obtained by the **General method B**, is a succinite liquid (30.8 mg, 56%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 – 7.45 (m, 2H), 7.34 – 7.21 (m, 3H), 7.15 – 6.86 (m, 3H), 4.18 (s, 2H), 2.34 (s, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 137.0, 134.2, 133.6, 130.9, 129.0, 128.2, 127.2, 126.9, 77.3, 19.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{15}H_{17}Se$: 277.0490; found: 277.0498.

phenyl(4-(trifluoromethyl)benzyl)selane (47)



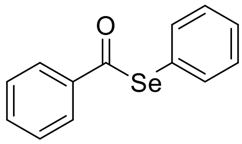
47, obtained by the **General method B**, is a succinite white solid (46.7 mg, 74%), melting point: 64 – 65°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 – 7.36 (m, 4H), 7.28 – 7.20 (m, 5H), 4.08 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.8, 135.3, 130.4, 129.4 (d, *J* = 32.4 Hz), 129.1, 129.0, 126.9, 125.4 (q, *J* = 3.7 Hz), 124.1 (d, *J* = 272.0 Hz). 38.8; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -62.43; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{12}F_3Se$: 317.0051; found: 317.0057.

allyl(phenyl)selane (48)



48, obtained by the **General method B**, is a succinite liquid (30.8 mg, 78%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.56 – 7.42 (m, 2H), 7.25 (d, *J* = 4.8 Hz, 3H), 6.05 – 5.79 (m, 1H), 5.07 – 4.85 (m, 2H), 3.52 (d, *J* = 7.4 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 134.4, 133.3, 128.9, 127.1, 116.8, 30.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_9H_{11}Se$: 199.0020; found: 199.0023.

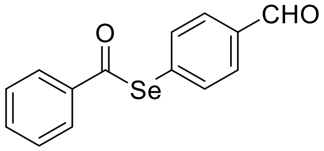
Se-phenyl benzoselenoate (49)



49, obtained by the **General method B**, is a succinite liquid (38.1 mg, 73%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.6 Hz, 2H), 7.66 – 7.57 (m, 3H), 7.56 – 7.34 (m, 5H).

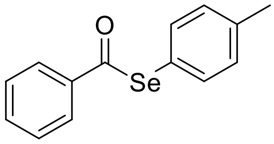
^{13}C NMR (125 MHz, $CDCl_3$) δ 193.4, 138.6, 136.4, 133.9, 129.4, 129.1, 129.0, 127.4, 125.9; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{11}OSe$: 262.9970; found: 262.9975.

Se-(4-formylphenyl) benzoselenoate (50)



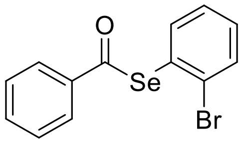
50, obtained by the **General method B**, is a white solid (38.2 mg, 66%), melting point: 73 – 74°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 10.06 (s, 1H), 8.00 – 7.87 (m, 4H), 7.79 (d, *J* = 8.1 Hz, 2H), 7.73 – 7.61 (m, 1H), 7.59 – 7.45 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 191.8, 191.7, 138.1, 136.5, 136.3, 134.3, 133.9, 130.0, 129.1, 127.4; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{11}O_2Se$: 290.9919; found: 290.9925.

Se-(*p*-tolyl) benzoselenoate (51)



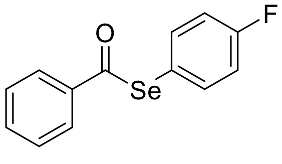
51, obtained by the **General method B**, is a white solid (47.9 mg, 87%), melting point: 72 – 73°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.10 – 7.76 (m, 2H), 7.74 – 7.55 (m, 1H), 7.53 – 7.34 (m, 4H), 7.38 – 7.09 (m, 2H), 2.39 (d, *J* = 3.1 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 193.8, 139.2, 138.6, 136.3, 133.8, 130. 2, 128.9, 127.3, 122.1, 21.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{13}OSe$: 277.0126; found: 277.0133.

Se-(2-bromophenyl) benzoselenoate (52)



52, obtained by the **General method B**, is a succinite solid (50.3 mg, 74%), melting point: 91 – 92°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.4 Hz, 2H), 7.82 – 7.68 (m, 2H), 7.67 – 7.57 (m, 1H), 7.55 – 7.43 (m, 2H), 7.40 – 7.31 (m, 1H), 7.31 – 7.26 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 191.6, 138.7, 138.3, 134.0, 133.3, 130.8, 130.1, 129.2, 129.0, 127.9, 127.4; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{10}BrOSe$: 340.9075; found: 340.9084.

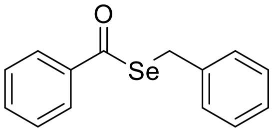
Se-(4-fluorophenyl) benzoselenoate (53)



53, obtained by the **General method B**, is a succinite liquid (44.7 mg, 80%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.5 Hz, 2H), 7.68 – 7.46 (m, 5H), 7.18 – 7.06 (m, 2H);

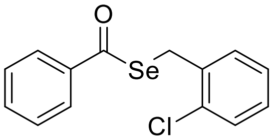
^{13}C NMR (125 MHz, $CDCl_3$) δ 193.2, 163.4 (d, *J* = 249.3 Hz), 138.3 (d, *J* = 8.3 Hz), 138.3, 134.0, 129.0, 127.3, 120.5 (d, *J* = 3.4 Hz), 116.7 (d, *J* = 21.7 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ -111.86; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{10}FOSe$: 280.9875; found: 280.9870.

Se-benzyl benzoselenoate (54)



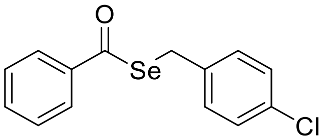
54, obtained by the **General method B**, is a succinite liquid (50 mg, 89%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.5 Hz, 2H), 7.70 – 7.52 (m, 1H), 7.48 – 7.10 (m, 7H), 4.34 (s, 2H);**^{3}C NMR** (125 MHz, $CDCl_3$) δ 194.5, 139.0, 138.9, 133.7, 129.1, 128.9, 128. 7, 127.3, 127.1, 29.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{13}OSe$: 277.0126; found: 277.0133.

Se-(2-chlorobenzyl) benzoselenoate (55)



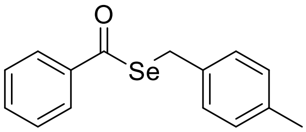
55, obtained by the **General method B**, is a white solid (46.4 mg, 75%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (d, 2H), 7.65 – 7.50 (m, 2H), 7.49 – 7.41 (m, 2H), 7.40 – 7.34 (m, 1H), 7.24 – 7.11 (m, 2H), 4.44 (s, 2H); **^{13}C NMR** (125MHz, $CDCl_3$) δ 194.3, 138.7, 137.1, 134.1, 133.7, 131.1, 129.5, 128.8, 128.5, 127.2, 127.0, 26.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{12}ClOSe$: 310.9736; found: 310.9744.

Se-(4-chlorobenzyl) benzoselenoate (56)



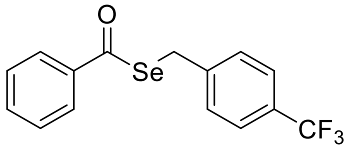
56, obtained by the **General method B**, is a white solid (52.6 mg, 85%), melting point: 84 – 85°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.5 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.27 (dd, *J* = 25.6, 8.3 Hz, 4H), 4.28 (s, 2H).; **^{13}C NMR** (125 MHz, $CDCl_3$) δ 194.1, 138.6, 137.7, 133.8, 132.7, 130.3, 128.8, 128.7, 127.2, 28.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{12}ClOSe$: 310.9736; found: 310.9744.

Se-(4-methylbenzyl) benzoselenoate (57)



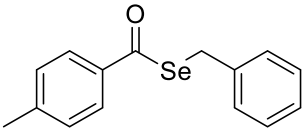
57, obtained by the **General method B**, is a white solid (45.7 mg, 79%), melting point: 77 – 79°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.69 – 7.48 (m, 1H), 7.48 – 7.36 (m, 2H), 7.34 – 7.18 (m, 2H), 7.09 (d, *J* = 7.7 Hz, 2H), 4.32 (s, 2H), 2.31 (s, 3H);**^{13}C NMR** (125 MHz, $CDCl_3$) δ 194.6, 138.9, 136.7, 135.9, 133.6, 129.3, 128.9, 128.8, 127.2, 28.9, 21.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{15}H_{15}OSe$: 291.0283; found: 291.0289.

Se-(4-(trifluoromethyl)benzyl) benzoselenoate (58)



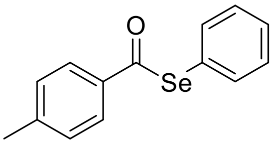
58, obtained by the **General method B**, is a white solid (52.2 mg, 76%), melting point: 96 – 97°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 – 7.83 (m, 2H), 7.64 – 7.57 (m, 1H), 7.55 (d, *J* = 8.2 Hz, 2H), 7.51 – 7.42 (m, 4H), 4.36 (s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 193.8, 143.5, 138.5, 133.9, 129.3, 129.2 (d, *J* = 32.5 Hz), 128.9, 127.3, 125.5 (q, *J* = 3.7 Hz), 124.1 (d, *J* = 272.0 Hz), 28.1; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -62.48; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{15}H_{12}F_3OSe$: 345.0000; found: 345.0009.

Se-benzyl 4-methylbenzoselenoate (59)



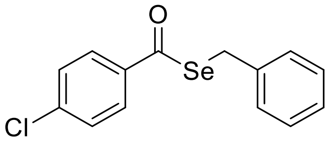
59, obtained by the **General method B**, is a white solid (38.8 mg, 67%), melting point: 77 – 78°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.63 – 7.05 (m, 7H), 4.40 (s, 2H), 2.45 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 193.9, 144.6, 139.1, 136.3, 129.4, 129.0, 128.6, 127.3, 126.9, 21.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{15}H_{15}OSe$: 291.0283; found: 291.0288.

Se-phenyl 4-methylbenzoselenoate (60)



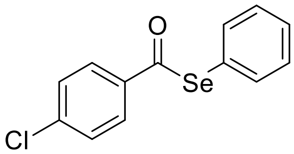
60, obtained by the **General method B**, is a white solid (34.1 mg, 62%), melting point: 72 – 73°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 3.4 Hz, 2H), 7.46 (d, *J* = 3.2 Hz, 3H), 7.29 (d, *J* = 7.9 Hz, 2H), 2.44 (s, 3H);**^{13}C NMR** (125 MHz, $CDCl_3$) δ 189.6, 144.5, 135.1, 134.1, 129.4, 129.2, 127.5, 21.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{13}OSe$: 277.0126; found: 277.0133.

Se-benzyl 4-chlorobenzoselenoate (61)



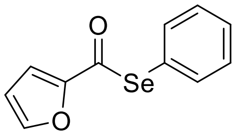
61, obtained by the **General method B**, is a succinite solid (34.7 mg, 56%), melting point: 84 – 85°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.5 Hz, 2H), 7.41 (d, *J* = 8.5 Hz, 2H), 7.36 (d, *J* = 7.3 Hz, 2H), 7.32 – 7.26 (m, 2H), 7.25 – 7.15 (m, 1H), 4.34 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 193.3, 140.1, 138.7, 137.2, 129.1, 129.0, 128.6, 128.5, 127.1, 29.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{12}ClOSe$: 310.9736; found: 310.9731.

Se-phenyl 4-chlorobenzoselenoate (62)



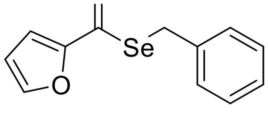
62, obtained by the **General method B**, is a white solid (36.7 mg, 62%), melting point: 81 – 82°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 5.4 Hz, 2H), 7.58 – 7.30 (m, 5H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 192.2, 140.3, 136.9, 136.2, 129.4, 129.2, 129.2, 128.6, 125.5; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{10}ClOSe$: 296.9580; found: 296.9586.

Se-phenyl furan-2-carboselenoate (63)



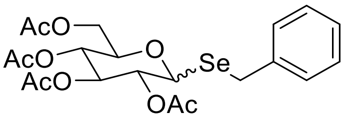
63, obtained by the **General method B**, is a white solid (42.7 mg, 80%), melting point: 51 – 52°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.59 (d, *J* = 7.4 Hz, 2H), 7.42 (d, *J* = 6.5 Hz, 3H), 7.22 (d, *J* = 3.4 Hz, 1H), 6.62 – 6.56 (m, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 180.8, 151.8, 146.6, 136.3, 129.3, 129.1, 124.8, 115.2, 112.8; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{11}H_9O_2Se$: 252.9762; found: 252.9768.

Se-benzyl furan-2-carboselenoate (64)



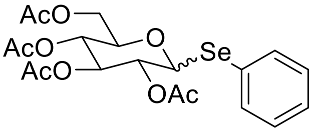
64, obtained by the **General method B**, is a white solid (42.4 mg, 80%), melting point: 53 – 54°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.59 (s, 1H), 7.35 (d, J = 7.4 Hz, 2H), 7.32 – 7.27 (m, 2H), 7.24 – 7.16 (m, 2H), 6.55 (d, J = 1.9 Hz, 1H), 4.32 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 181.8, 152.2, 146.4, 138.9, 129.0, 128.6, 127.0, 114.9, 112.6, 27.9; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_{11}OSe$: 266.9919; found: 266.9927.

(2R,3R,4S,5R)-2-(acetoxymethyl)-6-(benzylselanyl)tetrahydro-2H-pyran-3,4,5-triyl triacetate (65)



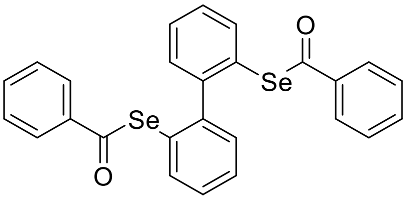
65, obtained by the **General method B**, is a succinite liquid (35.1 mg, 35%).**^{1}H NMR** (500 MHz, CDCl$_3$) δ 7.39 – 7.20 (m, 5H), 5.43 (s, 1H), 5.38 – 5.30 (m, 1H), 4.96 (d, *J* = 9.4 Hz, 1H), 4.52 (d, *J* = 10.1 Hz, 1H), 4.20 – 4.09 (m, 2H), 4.05 (d, *J* = 11.5 Hz, 1H), 3.93 (d, *J* = 11.5 Hz, 1H), 3.82 (s, 1H), 2.16 (s, 3H), 2.12 – 1.95 (m, 9H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 170.3, 170.2, 170.0, 169.7, 137.9, 129.0, 128.5, 127.0, 77.4, 75.50, 71.6, 67.8, 67.3, 61.5, 26.7, 20.8, 20.7, 20.5; **HRMS** (ESI) m/z [M+Na]$^+$ Calcd. for $C_{21}H_{26}NaO_9Se$: 525.0634; found: 525.0663.

(2R,3R,4S,5R)-2-(acetoxymethyl)-6-(phenylselanyl)tetrahydro-2H-pyran-3,4,5-triyl triacetate (66)



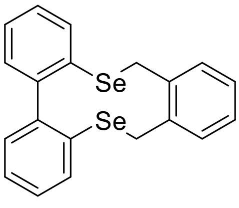
66, obtained by the **General method B**, is a succinite liquid (22.4 mg, 23%).**^{1}H NMR** (500 MHz, CDCl$_3$) δ 7.55 (s, 2H), 7.28 (d, *J* = 7.1 Hz, 3H), 6.28 (s, 1H), 5.50 (s, 1H), 5.32 – 5.20 (m, 2H), 4.69 (s, 1H), 4.05 (d, *J* = 16.7 Hz, 2H), 2.13 (d, *J* = 15.4 Hz, 6H), 2.00 (d, *J* = 22.5 Hz, 6H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 170.3, 170.1, 170.0, 169.9, 134.4, 129.1, 128.0, 127.4, 83.4, 68.8, 68.6, 68.4, 67.6, 61.6, 20.9, 20.61, 20.57; **HRMS** (ESI) m/z [M+Na]$^+$ Calcd. for $C_{20}H_{24}NaO_9Se$: 511.0478; found: 511.0495.

Se,Se'-([1,1'-biphenyl]-2,2'-diyl) dibenzoselenoate (67)



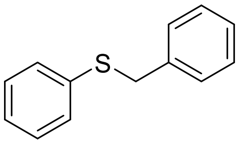
67, obtained by the **General method B**, is a white solid (62.4 mg, 60%), melting point: 154 – 156°C; **^{1}H NMR** (500 MHz, CDCl$_3$) δ 7.78 – 7.67 (m, 6H), 7.56 – 7.51 (m, 2H), 7.43 – 7.32 (m, 10H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 193.0, 146.7, 138.5, 137.4, 133.6, 130.5, 129.0, 128.7, 128.4, 127.3, 126.9; **HRMS** (ESI) m/z [M+H]$^+$ Calcd. for $C_{26}H_{19}O_2Se$: 522.9710; found: 522.9731.

6,11-dihydrotribenzo[*b,d,h*][1,6]diselenecine (68)



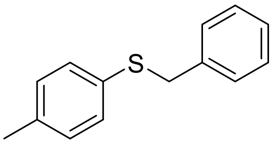
68, obtained by the **General method B**, is a white solid (19.5 mg, 47%), melting point: 118 – 120°C; ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 6.8 Hz, 2H), 7.61 (d, *J* = 6.9 Hz, 2H), 7.39 – 7.30 (m, 2H), 7.26 – 7.20 (m, 4H), 7.19 – 7.11 (m, 2H), 4.31 (s, 4H). ^{13}**C NMR** (125 MHz, $CDCl_3$) δ 141.9, 141.6, 131.8, 131.1, 129.5, 128.8, 127.5, 126.4, 125.9, 29.8; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{20}H_{17}OSe$: 416.9655; found: 416.9681.

benzyl(phenyl)sulfane (69)



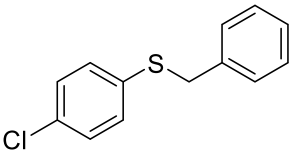
69, obtained by the **General method C**, is a colorless liquid (32.4 mg, 81%).^{1}**H NMR** (500 MHz, $CDCl_3$) δ 7.35 – 7.20 (m, 9H), 7.20 – 7.13 (m, 1H), 4.11 (d, *J* = 2.3 Hz, 2H); ^{13}**C NMR** (125 MHz, $CDCl_3$) δ 137.4, 136.3, 129.8, 128.8, 128.5, 127.1, 126.3, 39.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{13}S$: 201.0732; found: 201.0728.

Benzyl (*p*-tolyl) sulfane (70)



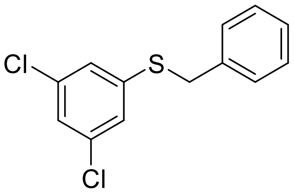
70, obtained by the **General method C**, is a colorless liquid (35.6 mg, 83%).^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.35 – 7.11 (m, 7H), 7.05 (d, *J* = 7.7 Hz, 2H), 4.06 (s, 2H), 2.29 (s, 3H). ^{13}**C NMR** (100 MHz, $CDCl_3$) δ 137.8, 136.6, 132.5, 130.7, 129.7, 128.9, 128.5, 127.1, 39.8, 21.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}S$: 215.0889; found: 215.0893.

Benzyl (4-chlorophenyl) sulfane (71)



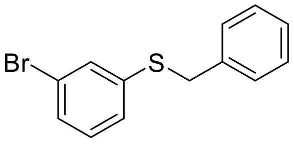
71, obtained by the **General method C**, is a colorless liquid (42.3 mg, 90%).^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.28 – 7.22 (m, 5H), 7.20 (s, 4H), 4.07 (s, 2H); ^{13}**C NMR** (100 MHz, $CDCl_3$) δ 137.1, 134.6, 132.4, 131.3, 128.9, 128.8, 128.5, 127.3, 39.2; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}ClS$: 235.0343; found: 235.0348.

Benzyl (3,5-dichlorophenyl) sulfane (72)



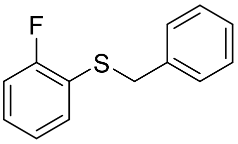
72, obtained by the **General method C**, is a white solid (30.1 mg, 56%), melting point:60 – 61℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 (s, 5H), 7.12 (s, 3H), 4.11 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 140.3, 136.0, 135.0, 128.8, 128.7, 127.6, 126.6, 126.0, 38.2; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{11}Cl_2S$: 268.9953; found: 268.9960.

Benzyl (3-bromophenyl) sulfane (73)



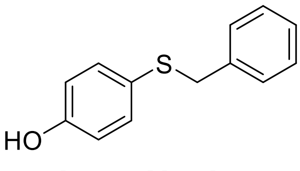
73, obtained by the **General method C**, is a succinite solid (45.6 mg, 87%), melting point: 64 – 65℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.34 – 7.20 (m, 6H), 7.18 (d, *J* = 7.9 Hz, 1H), 7.14 – 7.03 (m, 1H), 4.10 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 138.8, 136.7, 131.8, 130.1, 129.2, 128.8, 128.6, 127.8, 127.4, 122.6, 38.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}BrS$: 278.9838; found: 278.9827.

Benzyl (2-fluorophenyl) sulfane (74)



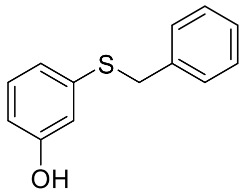
74, obtained by the **General method C**, is a colorless liquid (38.9 mg, 89%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.36 – 7.11 (m, 7H), 7.11 – 6.93 (m, 2H), 4.09 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 161.7 (d, *J* = 245.5 Hz), 137.2, 133.0, 128.82, 128.76, 128.4, 127.2, 124.3 (d, *J* = 3.7 Hz), 122.7 (d, *J* = 17.7 Hz), 115.6 (d, *J* = 22.5 Hz), 38.37; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -128.34; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}FS$: 219.0638; found: 219.0642.

4-(benzylthio) phenol (76)



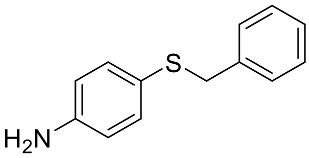
76, obtained by the **General method C**, is a white solid (38.9 mg, 90%), melting point: 111 – 112℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 – 7.02 (m, 7H), 6.70 (d, *J* = 7.3 Hz, 2H), 5.19 (s, 1H), 3.97 (s, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.2, 138.0, 134.3, 128.9, 128.3, 127.0, 126.1, 115.9, 41.2; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{13}OS$: 217.0682; found: 217.0690.

3-(benzylthio) phenol (77)



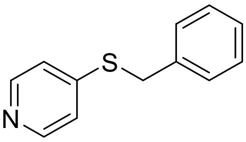
76, obtained by the **General method C**, is a white solid (35.9 mg, 83%), melting point: 111 – 113°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 – 7.16 (m, 5H), 7.16 – 7.01 (m, *J* = 7.9 Hz, 1H), 6.90 – 6.69 (m, 2H), 6.62 (d, *J* = 8.1 Hz, 1H), 4.08 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.7, 138.0, 137.2, 129.9, 128.8, 128.5, 127.2, 121.6, 116.0, 113.4, 38.5; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{13}OS$: 217.0682; found: 217.0690.

4-(benzylthio) aniline (78)



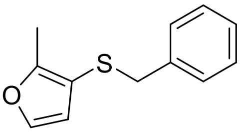
78, obtained by the **General method C**, is a white solid (40 mg, 93%), melting point: 217 – 220°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.31 – 7.13 (m, 5H), 7.12 (d, *J* = 8.2 Hz, 2H), 6.54 (d, *J* = 8.3 Hz, 2H), 3.92 (s, 2H), 3.53 (s, 2H);**^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.2, 138.4, 134.7, 128.9, 128.2, 126.8, 122.9, 115.47, 41.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{14}NS$: 216.0841; found: 216.0850.

4-(benzylthio)pyridine (79)



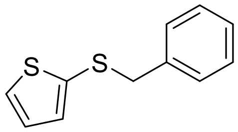
79, obtained by the **General method C**, is a white solid (38.2 mg, 95%), melting point: 161 – 163°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.38 (d, *J* = 4.4 Hz, 2H), 7.43 – 7.26 (m, 5H), 7.12 (d, *J* = 6.0 Hz, 2H), 4.21 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 149.14, 149.10, 135.5, 128.9, 128.7, 127.7, 120.8, 35.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_{12}NS$: 202.0685; found: 202.0691.

3-(benzylthio)-2-methylfuran (80)



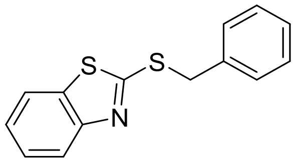
80, obtained by the **General method C**, is a colorless liquid (29 mg, 71%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.23 (d, *J* = 5.9 Hz, 4H), 7.09 (d, *J* = 6.7 Hz, 2H), 6.21 (s, 1H), 3.73 (s, 2H), 1.95 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 156.2, 140.4, 138.4, 128.9, 128.2, 126.9, 115.2, 109.2, 40.7, 11.2; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{12}H_{13}OS$: 205.0682; found: 205.0679.

2-(benzylthio)thiophene (81)



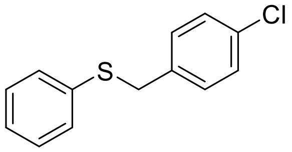
82, obtained by the **General method C**, is a colorless liquid (39.2 mg, 95%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36 – 7.17 (m, 4H), 7.15 (d, J = 6.4 Hz, 2H), 7.08 – 6.65 (m, 2H), 3.95 (s, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 137.6, 134.3, 133.5, 129.7, 128.9, 128.3, 127.4, 127.2, 43.8; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{11}H_{11}S_2$: 207.0297; found: 207.0300.

2-(benzylthio)benzo[*d*]thiazole (82)



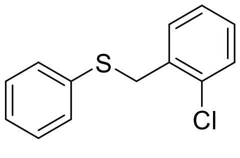
82, obtained by the **General method C**, is a white solid (37.6 mg, 73%), melting point: 40 – 41°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.1 Hz, 1H), 7.75 (d, J = 8.0 Hz, 1H), 7.53 – 7.37 (m, 3H), 7.37 – 6.95 (m, 4H), 4.62 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 166.4, 153.1, 136.1, 135.3, 129.1, 128.7, 127.7, 126.0, 124.3, 121.5, 121.0, 37.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{12}NS_2$: 258.0406; found: 258.0415.

(4-chlorobenzyl) (phenyl) sulfane (83)



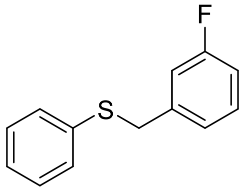
84, obtained by the **General method C**, is a succinite liquid (46.5 mg, 99%)white solid (46.5 mg, 99%), melting point: 77 – 78°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45 – 6.96 (m, 9H), 4.04 (s, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.1, 135.6, 132.9, 130.2, 130.1, 128.9, 128.6, 126.6, 38.5; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}ClS$: 235.0343; found: 235.0348.

(2-chlorobenzyl)(phenyl)sulfane (84)



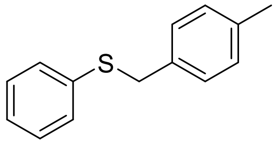
84, obtained by the **General method C**, is a white solid (46.5 mg, 87%), melting point: 77 – 78°C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 – 7.27 (m, 3H), 7.27 – 7.06 (m, 6H), 4.20 (s, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 135.7, 135.2, 134.0, 130.7, 129.6, 128.8, 128.5, 126.7, 37.0; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}ClS$: 235.0343; found: 235.0348.

(3-fluorobenzyl)(phenyl)sulfane (85)



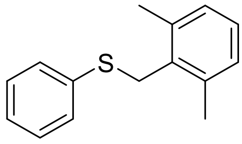
85, obtained by the **General method C**, is a succinite liquid (33.6 mg, 77%).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48 – 7.14 (m, 6H), 7.10 – 6.83 (m, 3H), 4.07 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.8 (d, *J* = 246.1 Hz), 140.1 (d, *J* = 7.4 Hz), 140.1, 135.7, 130.1, 129.9 (d, *J* = 8.3 Hz), 128.9, 126.6, 124.4 (d, *J* = 2.8 Hz), 115.7 (d, *J* = 21.8 Hz), 114.1 (d, *J* = 21.1 Hz). 38.7; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -113.12; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}FS$: 219.0638; found: 219.0642.

(4-methylbenzyl)(phenyl)sulfane (86)



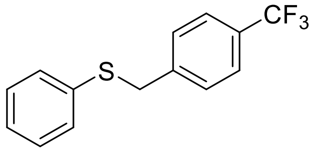
86, obtained by the **General method C**, is a pink solid (39.9 mg, 93%), melting point: 46 – 47℃; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.1 Hz, 2H), 7.27 – 7.22 (m, 2H), 7.22 – 7.13 (m, 3H), 7.08 (d, *J* = 7.4 Hz, 2H), 4.08 (s, 2H), 2.31 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.8, 136.6, 134.3, 129.6, 129.2, 128.8, 128.7, 126.1, 38.6, 21.1; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{15}S$: 215.0889; found: 215.0893.

(2,6-dimethylbenzyl)(phenyl)sulfane (87)



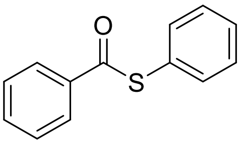
87, obtained by the **General method C**, is a white solid (42.5 mg, 93%), melting point: 50 – 51℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.5 Hz, 2H), 7.33 – 7.23 (m, 2H), 7.23 – 7.11 (m, 1H), 7.11 – 6.76 (m, 3H), 4.15 (s, 2H), 2.37 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 137.3, 137.3, 133.0, 129.9, 128.8, 128.2, 127.3, 126.3, 33.9, 19.6; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{15}H_{17}S$: 229.1045; found: 229.1050.

phenyl(4-(trifluoromethyl) benzyl) sulfane (88)



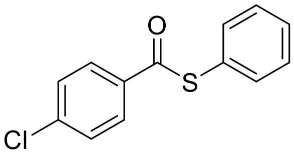
88, obtained by the **General method C**, is a succinite liquid (47.8 mg, 89%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44 (dd, *J* = 63.0, 8.0 Hz, 4H), 7.31 – 7.15 (m, 4H), 4.12 (s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.8, 135.3, 130.4, 129.4 (d, *J* = 32.4 Hz), 129.05, 128.96, 126.9, 125.4 (q, *J* = 3.7 Hz), 124.1 (d, *J* = 272.0 Hz), 38.8;**^{19}F NMR** (471 MHz, $CDCl_3$) δ -62.46; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{12}F_3S$: 269.0606; found: 269.0615.

S-phenyl benzothioate (89)



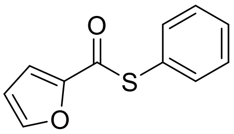
89, obtained by the **General method C**, is a white solid (38.6 mg, 89%), melting point: 56 – 57℃; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.8 Hz, 2H), 7.68 – 7.58 (m, 1H), 7.54 – 7.40 (m, 7H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 189.6, 136.4, 136.3, 136.0, 133.9, 129.5, 128.9, 127.5, 125.9; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{11}OS$: 215.0525; found: 215.0529.

S-phenyl 4-chlorobenzothioate (90)



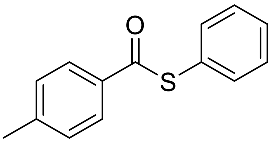
90, obtained by the **General method C**, is a white solid (40.8 mg, 82%), melting point: 81 – 82℃; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.77 – 7.35 (m, 7H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 189.0, 140.0, 135.0, 134.9, 129.6, 129.3, 129.0, 128.8, 126.9; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{10}OS$: 249.0135; found: 249.0141.

S-phenyl furan-2-carbothioate (91)



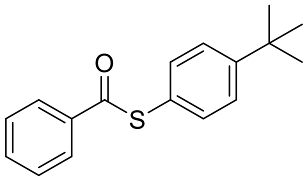
91, obtained by the **General method C**, is a colorless liquid (34.3 mg, 84%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.62 (s, 1H), 7.50 (d, *J* = 2.9 Hz, 2H), 7.48 – 7.43 (m, 3H), 7.26 (d, *J* = 3.5 Hz, 1H), 6.59 – 6.55 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 178.6, 150.3, 146.4, 135.1, 129.6, 129.2, 126.2, 116.2, 112.4; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{11}H_9O_2S$: 205.0318; found: 205.0312.

S-phenyl 4-methylbenzothioate (92)



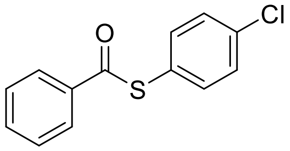
92, obtained by the **General method C**, is a white solid (32.9 mg, 72%), melting point: 64 – 65°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.1 Hz, 2H), 7.61 (d, *J* = 4.6 Hz, 2H), 7.44 (d, *J* = 1.4 Hz, 3H), 7.29 (d, *J* = 7.9 Hz, 2H), 2.42 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 192.7, 144.9, 136.3, 136.0, 129.6, 129.3, 128.9, 127.4, 125.9, 21.7; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{14}H_{13}OS$: 229.0682; found: 229.0688.

S-(4-(*tert*-butyl) phenyl) benzothioate (93)



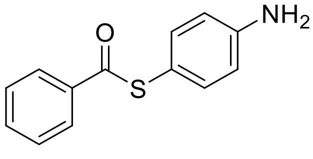
93, obtained by the **General method C**, is a hite solid (51.9 mg, 96%), melting point: 79 – 81°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.6 Hz, 2H), 7.71 – 7.51 (m, 1H), 7.51 – 7.33 (m, 6H), 1.34 (s, 9H);**^{13}C NMR** (125 MHz, $CDCl_3$) δ 190.5, 152.7, 136.9, 134.7, 133.5, 128.7, 127.4, 126.4, 123.8 34.8, 31.2; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{17}H_{19}OS$: 271.1151; found: 271.1160.

S-(4-chlorophenyl) benzothioate (94)



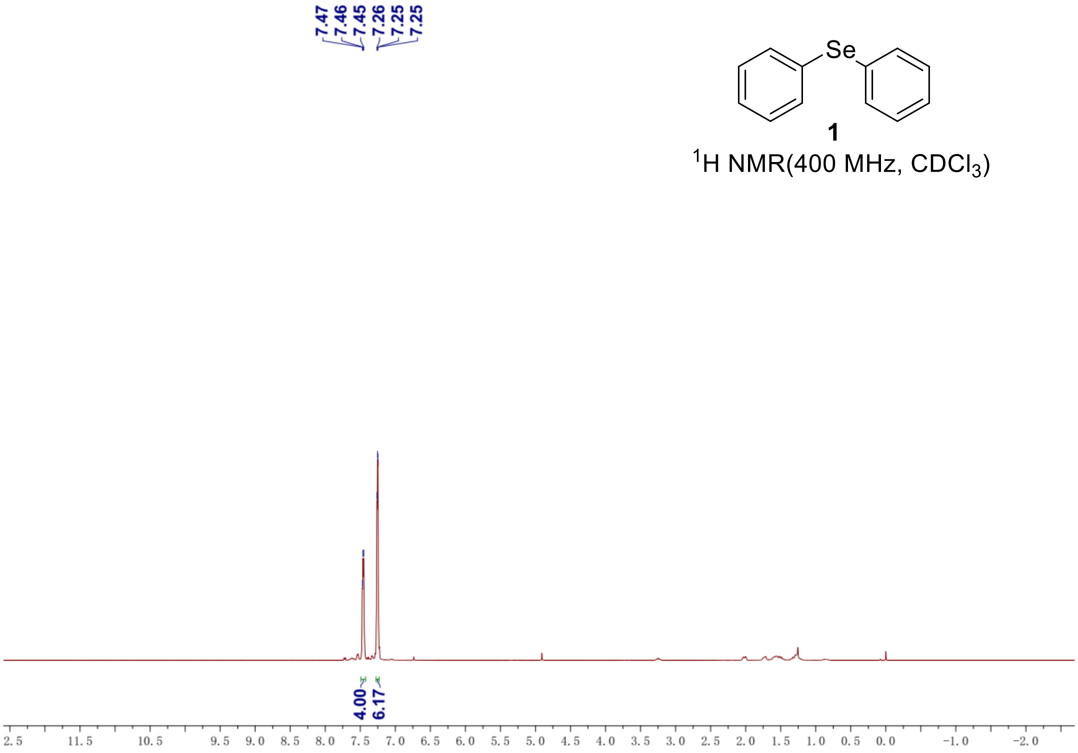
94, obtained by the **General method C**, is a white solid (49.2 mg, 99%), melting point: 72 – 74°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.7 Hz, 2H), 7.71 – 7.58 (m, 1H), 7.58 – 7.36 (m, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 190.1, 136.6, 135.1, 133.6, 129.5, 129.2, 128.7, 127.4, 127.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{10}OS$: 249.0135; found: 249.0141.

S-(4-aminophenyl) benzothioate (95)



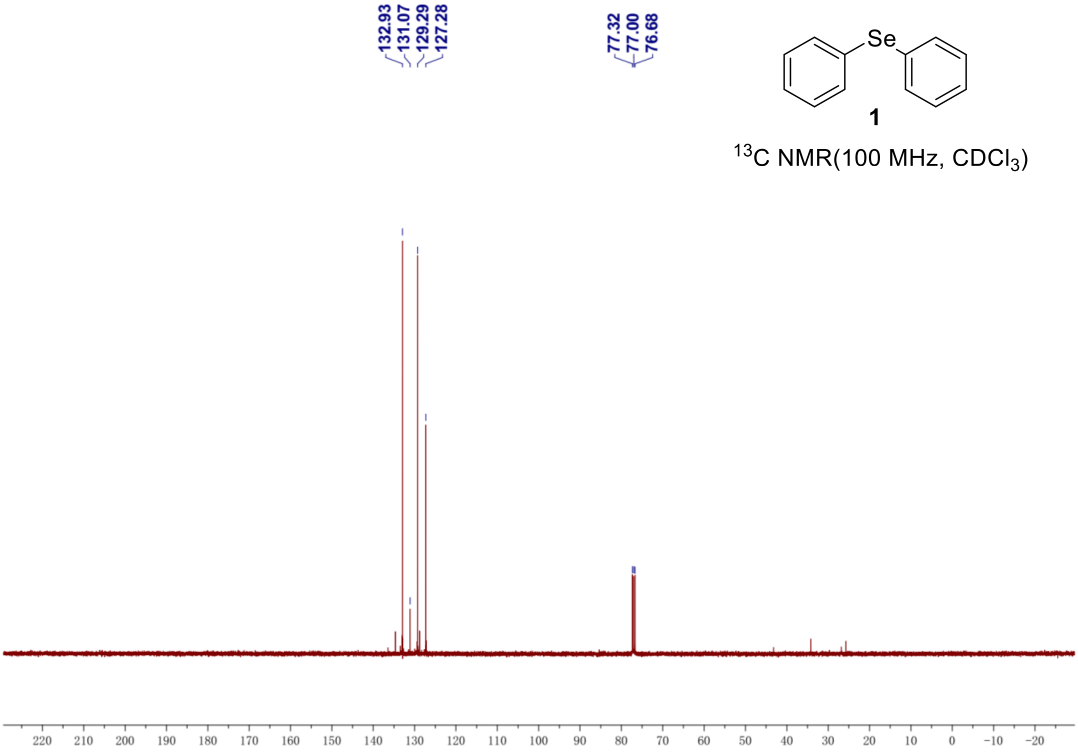
95, obtained by the **General method C**, is a white solid (27.5 mg, 60%), melting point: 113 – 115°C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.02 (d, *J* = 6.9 Hz, 2H), 7.58 (s, 1H), 7.47 (d, *J* = 7.1 Hz, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 6.72 (d, *J* = 8.0 Hz, 2H), 3.77 (s, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 191.8, 148.0, 136.8, 136.4, 133.4, 128.6, 127.4, 115.6, 114.3; **HRMS** (ESI) m/z $[M+H]^+$ Calcd. for $C_{13}H_{12}NOS$: 230.0634; found: 230.0639.

7. ^{1} H, ^{13} C and ^{19} F NMR spectra of products



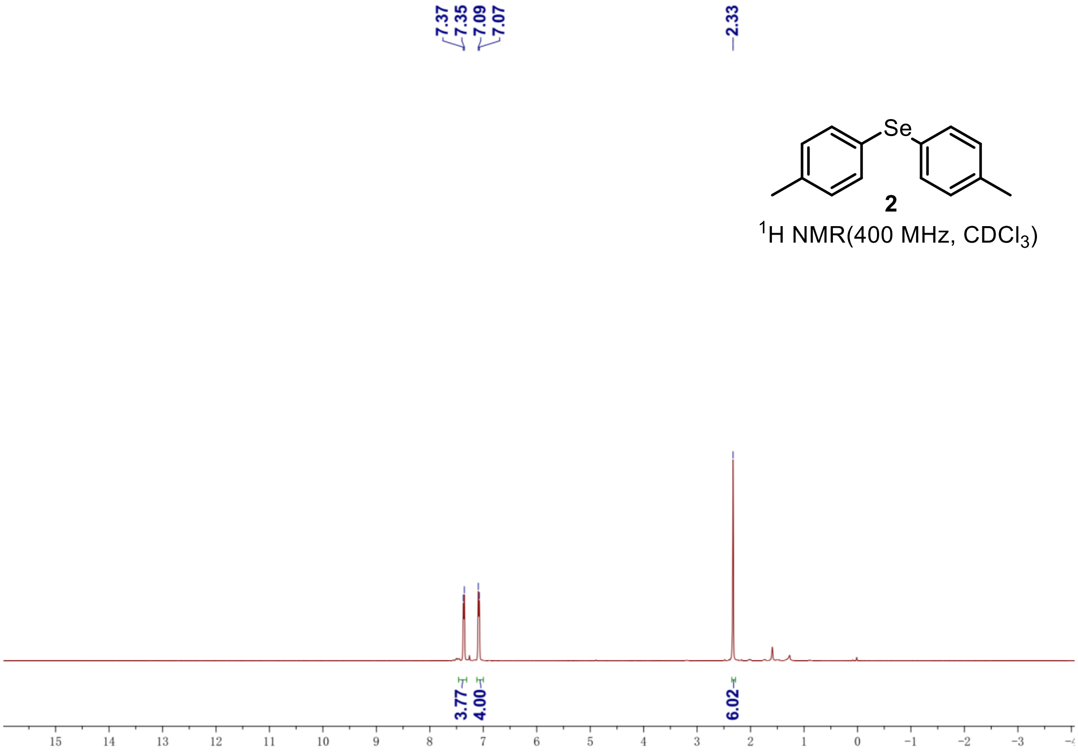
1

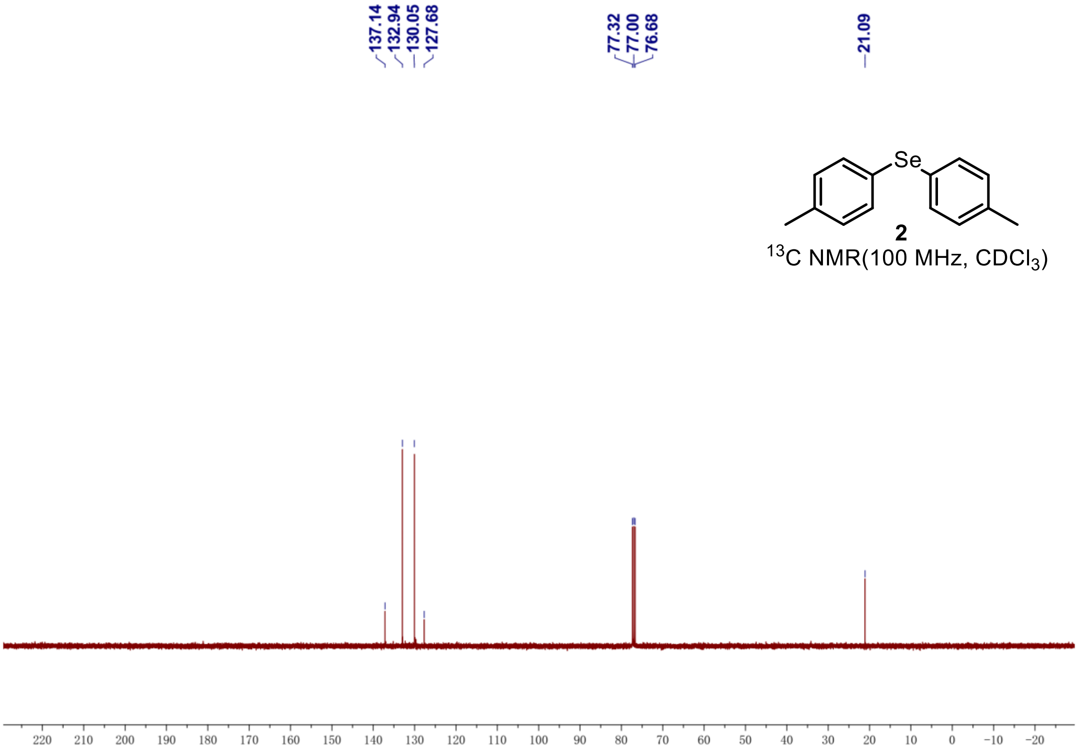
^{1}H NMR(400 MHz, CDCl$_3$)

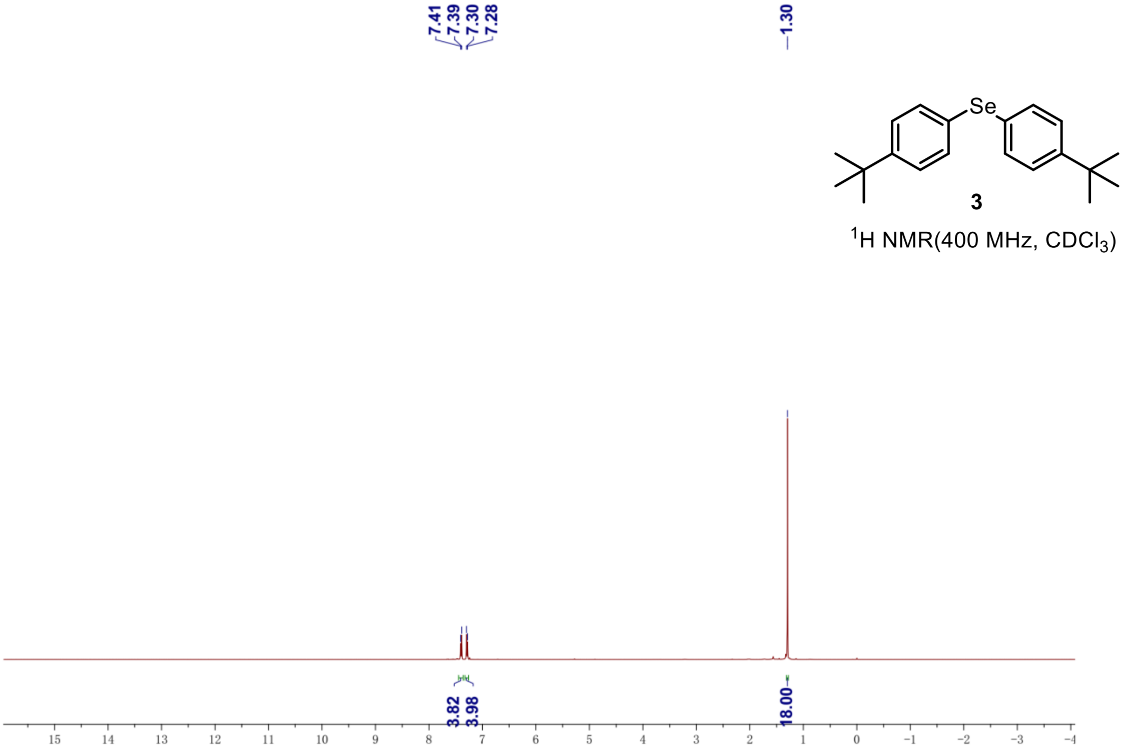


1

^{13}C NMR(100 MHz, CDCl$_3$)

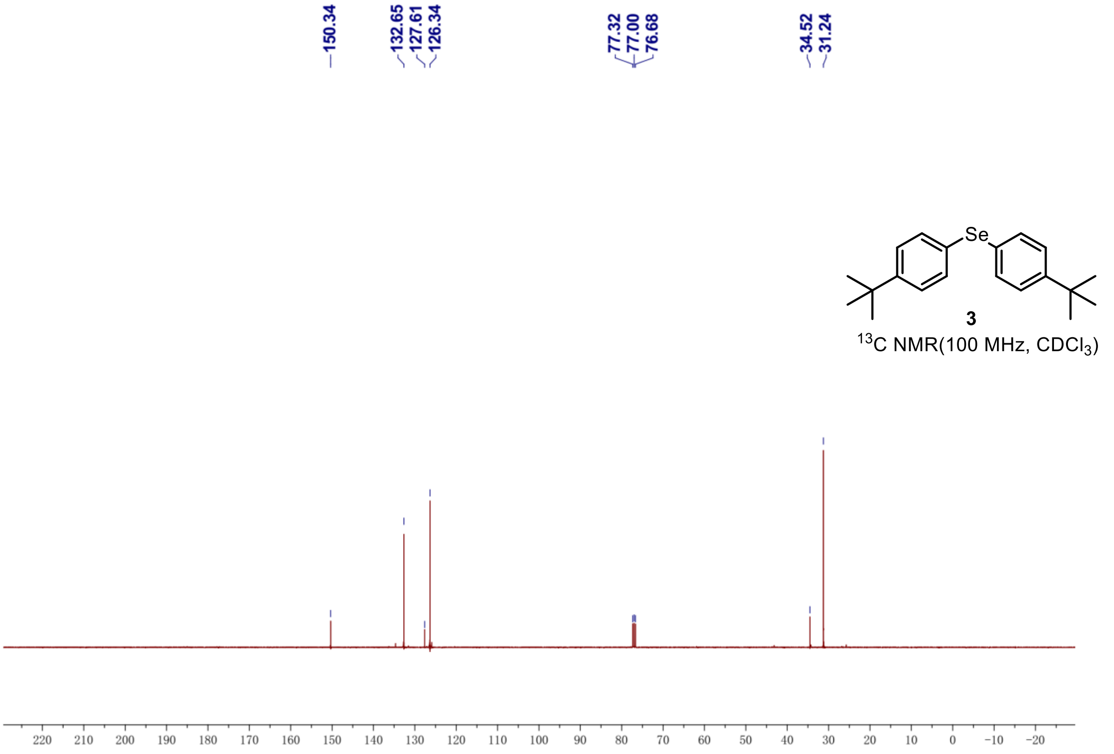

7.37
7.35
7.09
7.07
2.33
Se
2
^{1}H NMR(400 MHz, CDCl$_3$)
3.77
4.00
6.02
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3


137.14
132.94
130.05
127.68
77.32
77.00
76.68
21.09
Se
2
^{13}C NMR(100 MHz, CDCl$_3$)
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
−20



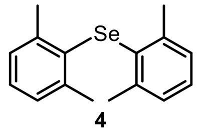
3

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



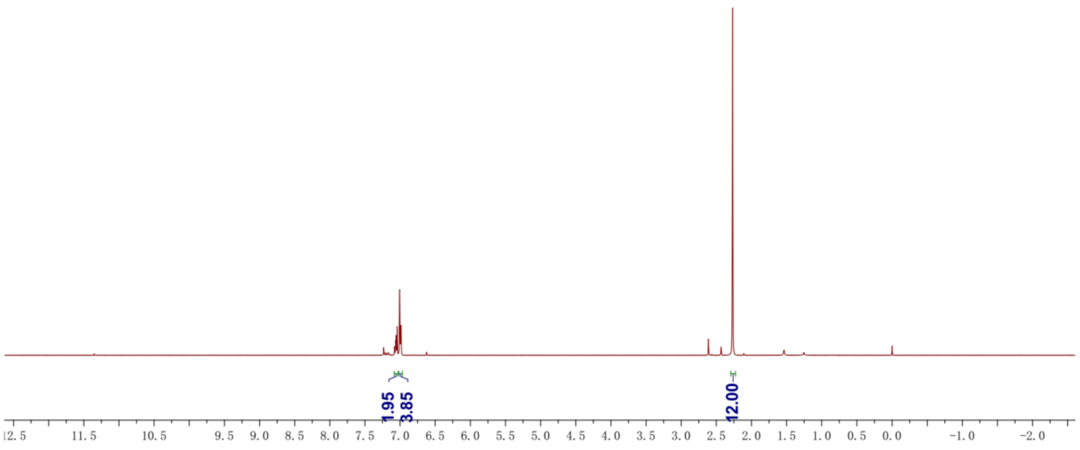
3

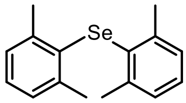
^{13}C NMR(100 MHz, $CDCl_3$)



4

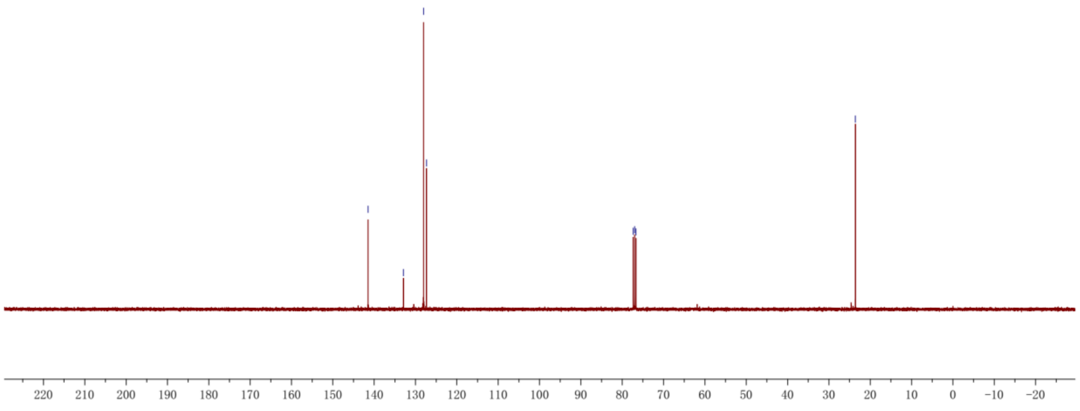
^1H NMR(400 MHz, $CDCl_3$)

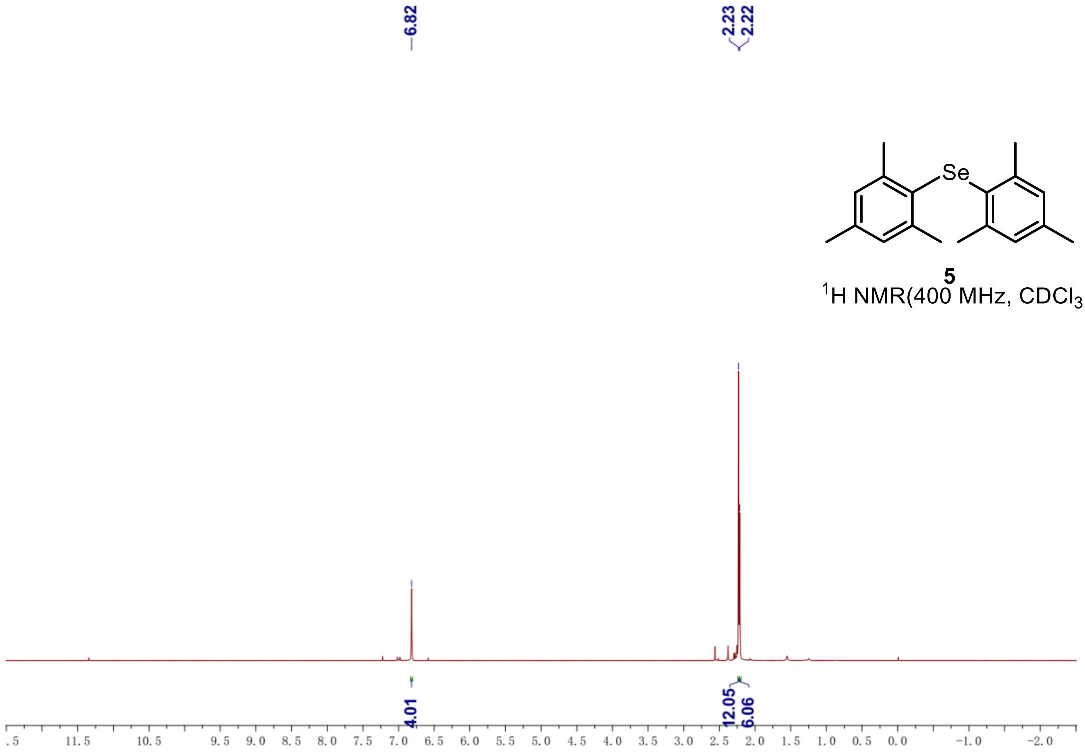




4

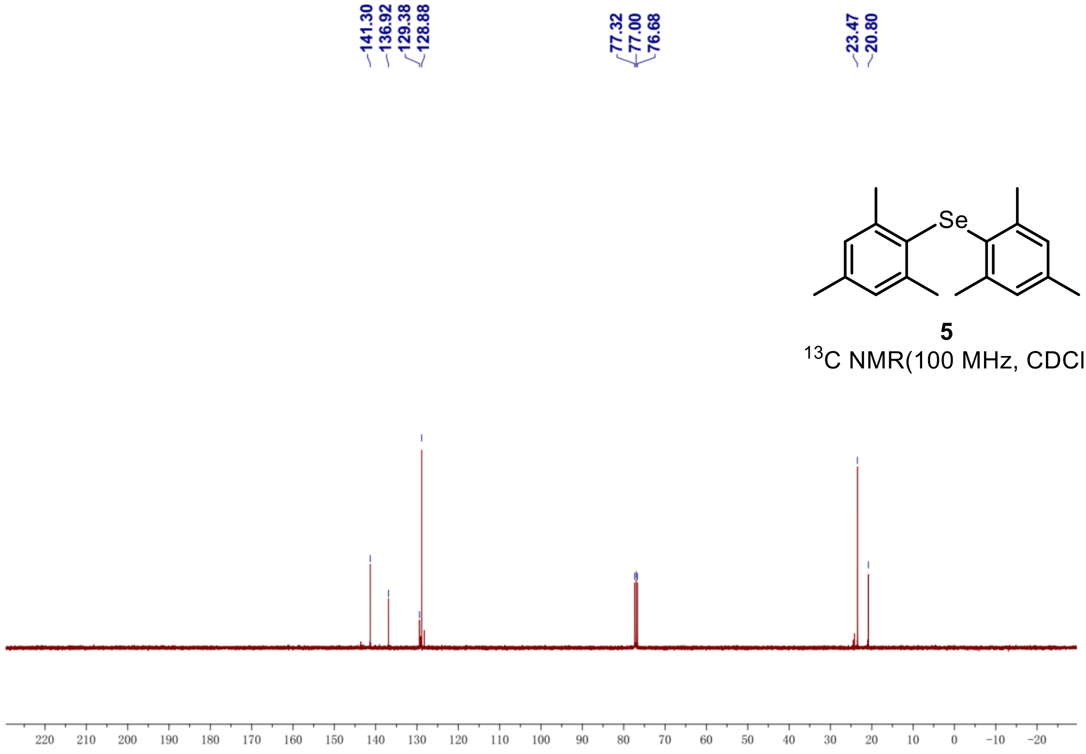
^{13}C NMR(100 MHz, $CDCl_3$)





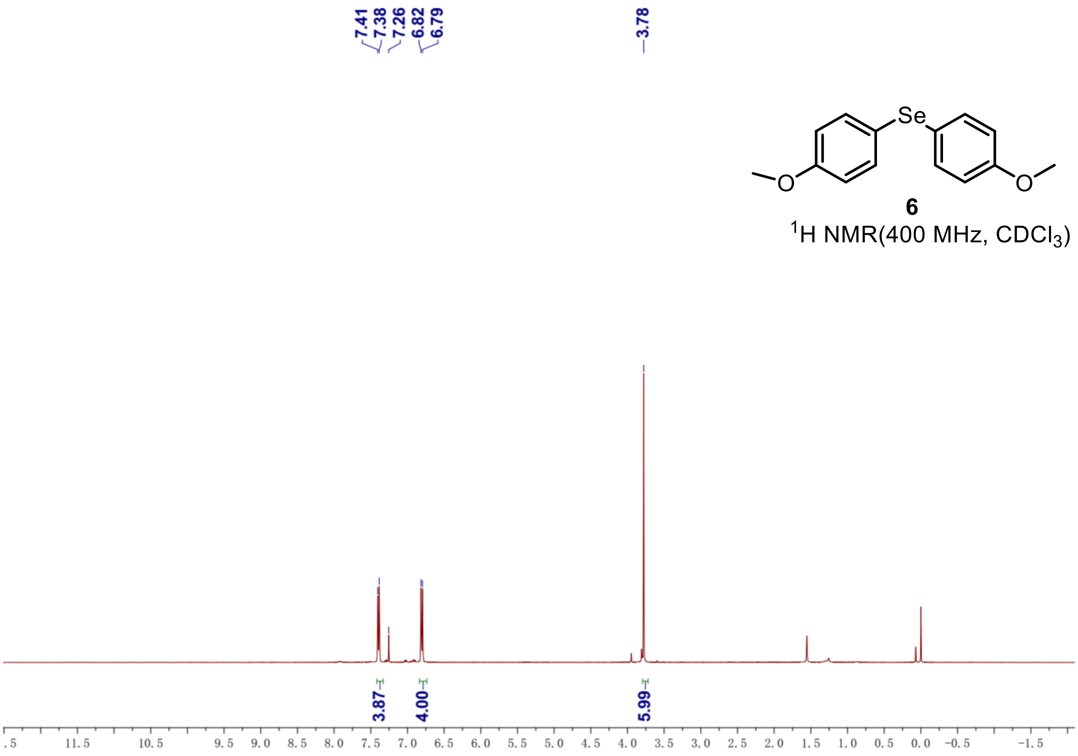
5

^{1}H NMR(400 MHz, CDCl$_3$)



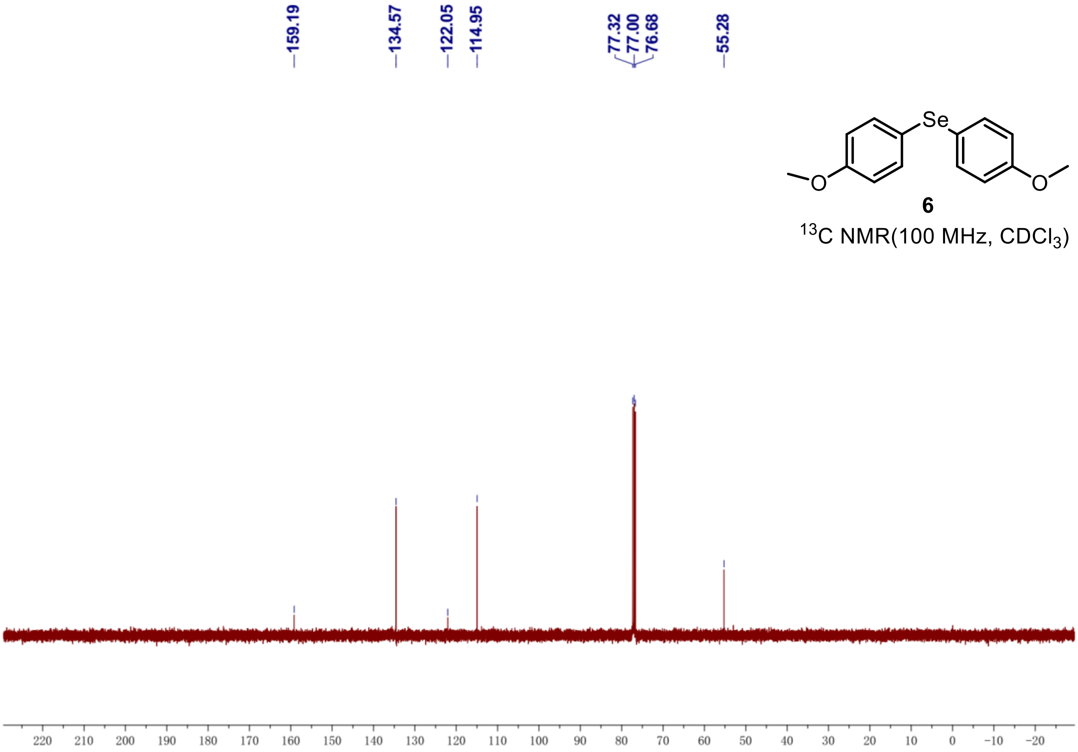
5

^{13}C NMR(100 MHz, CDCl$_3$)



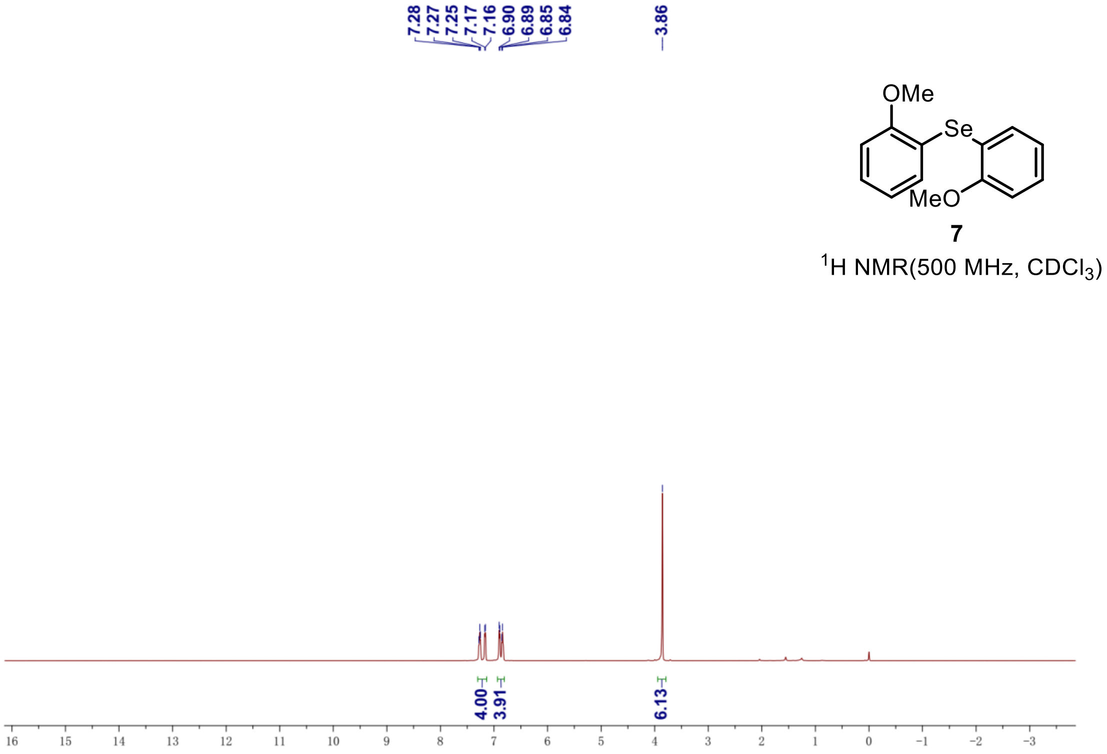
6

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



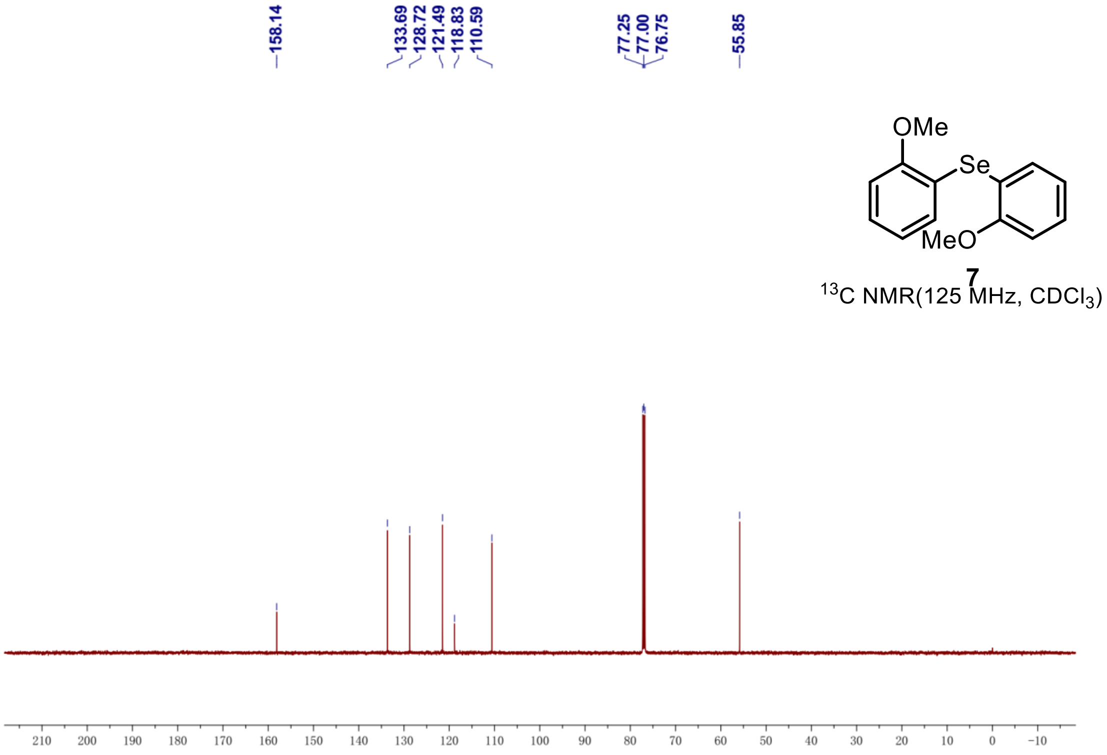
6

^{13}C NMR(100 MHz, $CDCl_3$)



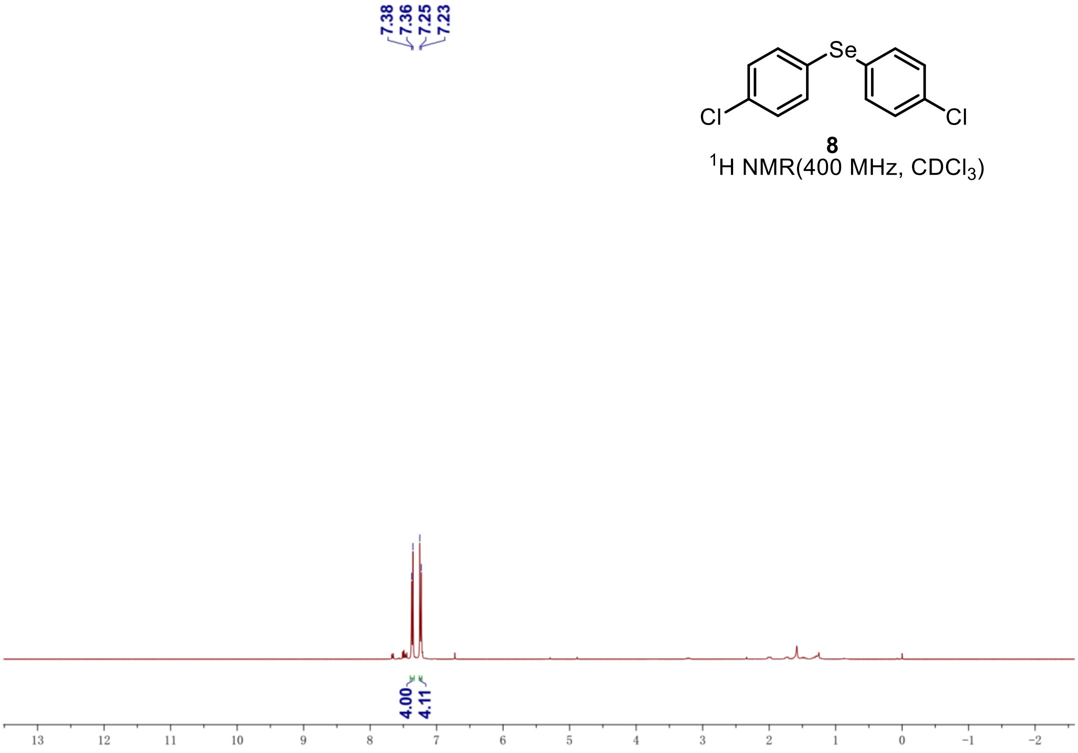
7

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



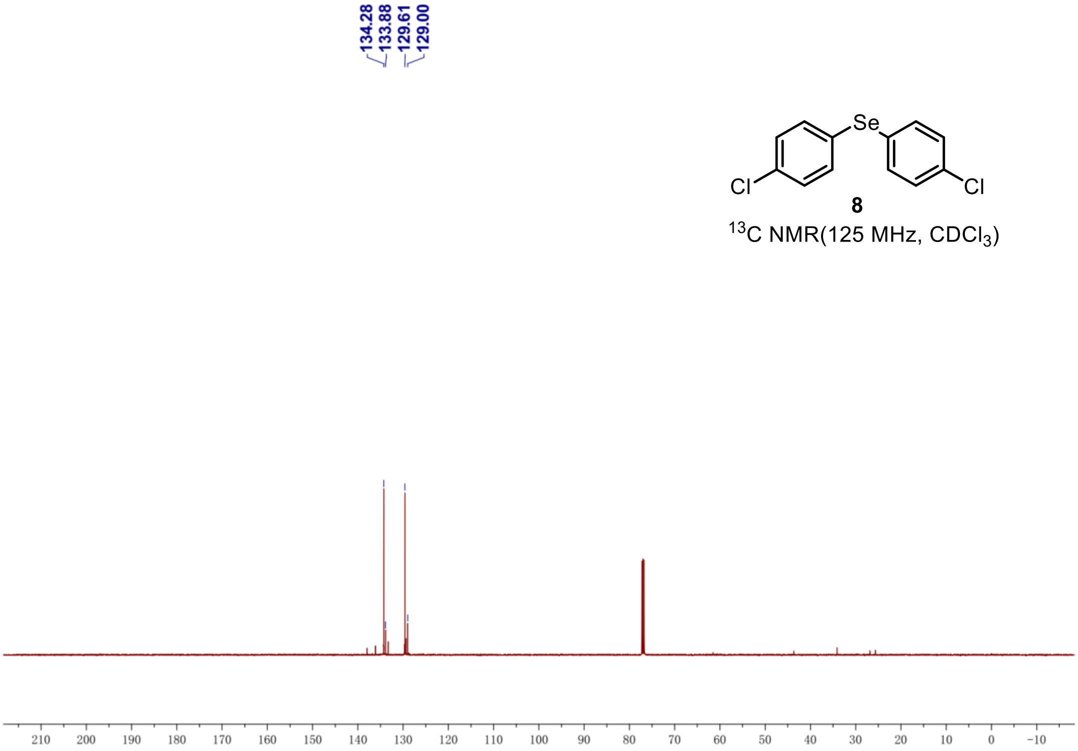
7

^{13}C NMR(125 MHz, $CDCl_3$)



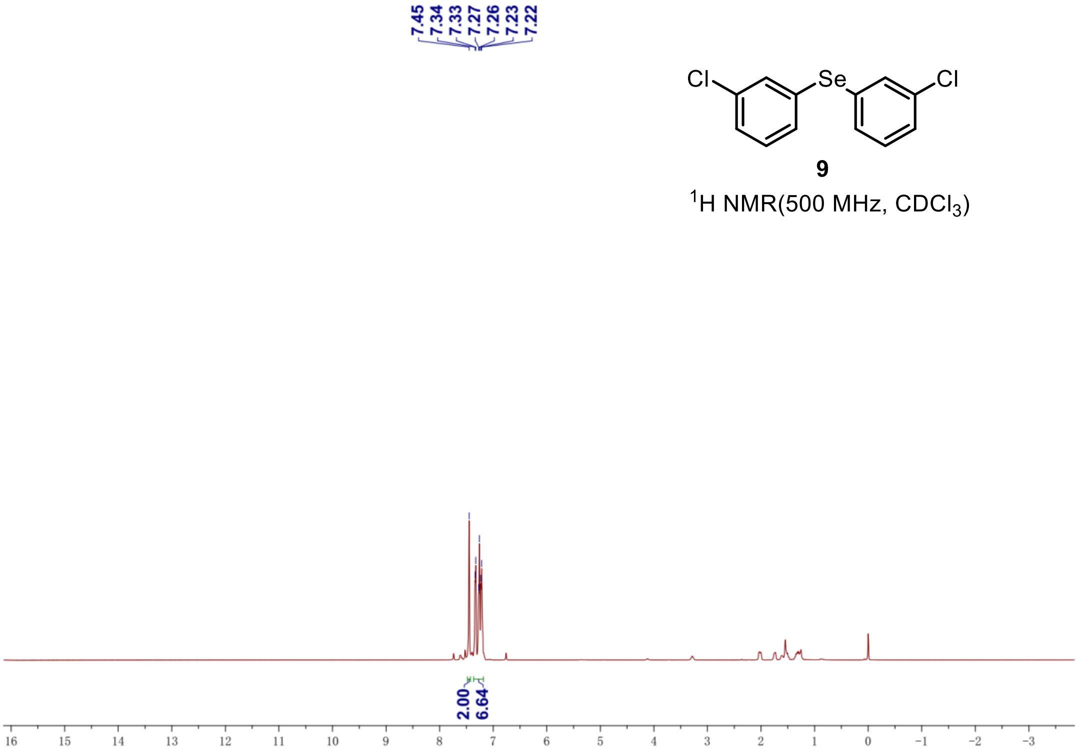
8

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



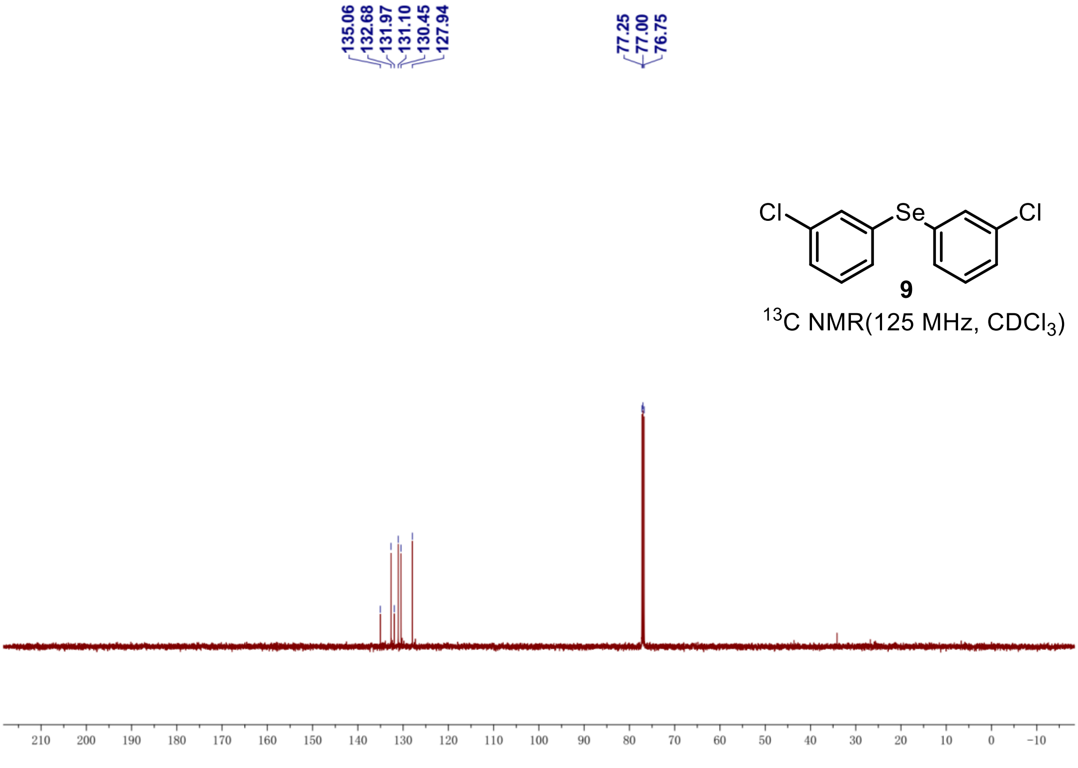
8

^{13}C NMR(125 MHz, $CDCl_3$)



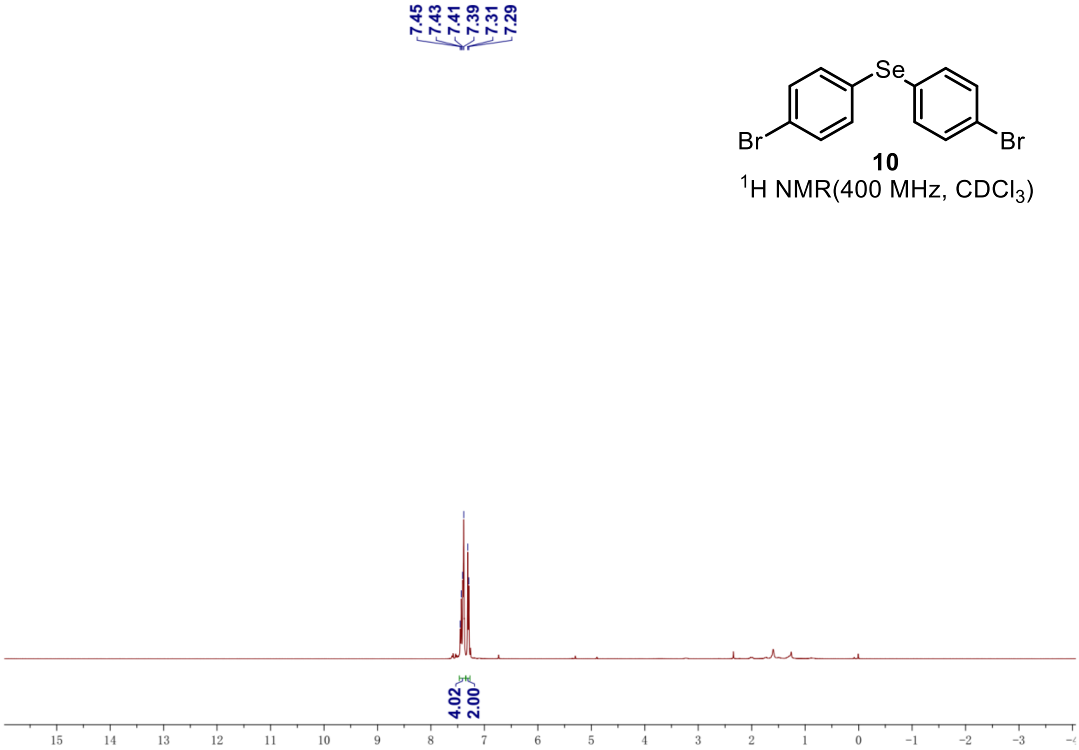
9

^{1}H NMR(500 MHz, CDCl$_3$)



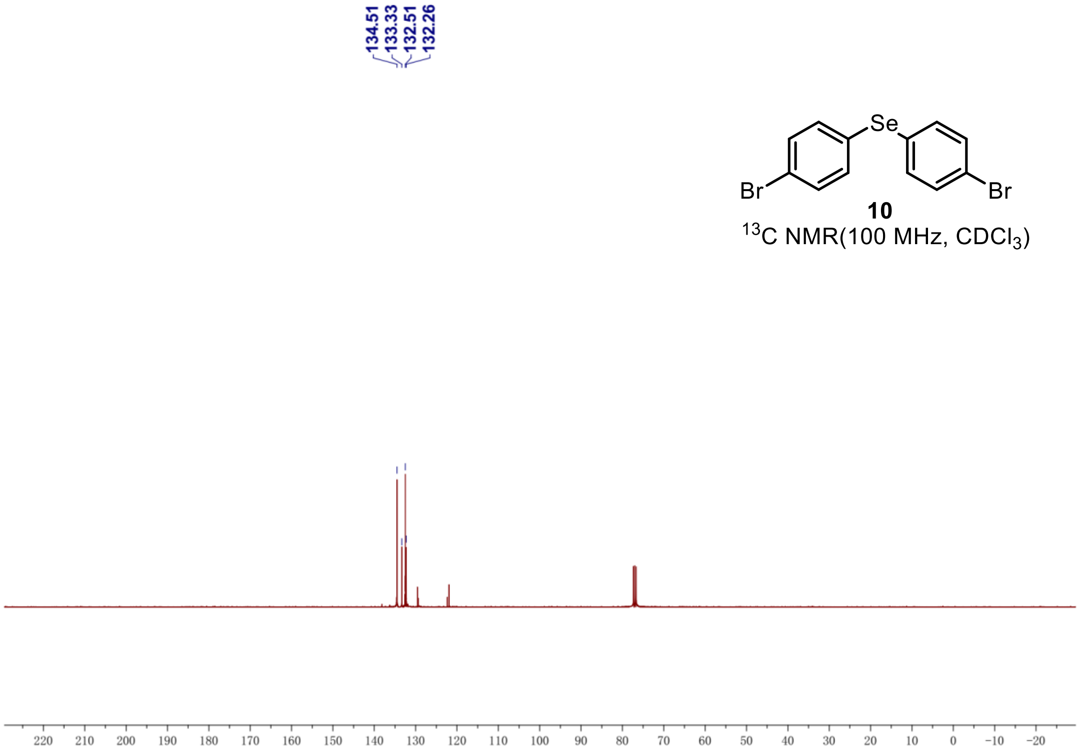
9

^{13}C NMR(125 MHz, CDCl$_3$)



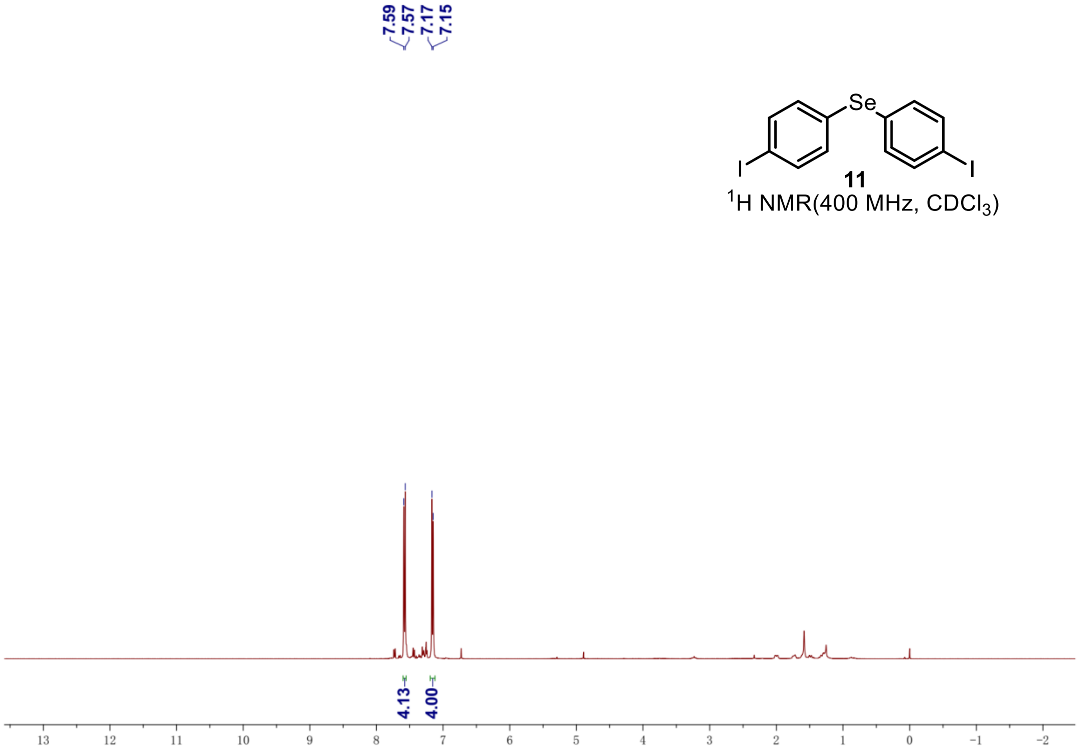
10

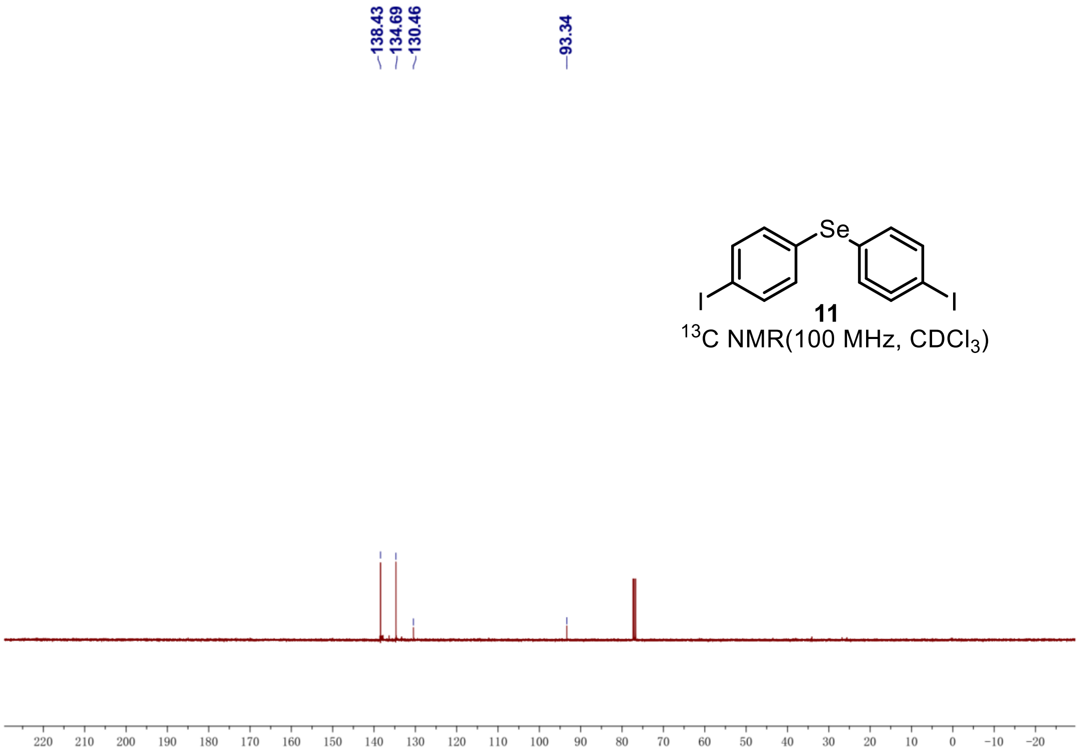
^{1}H NMR(400 MHz, CDCl$_3$)

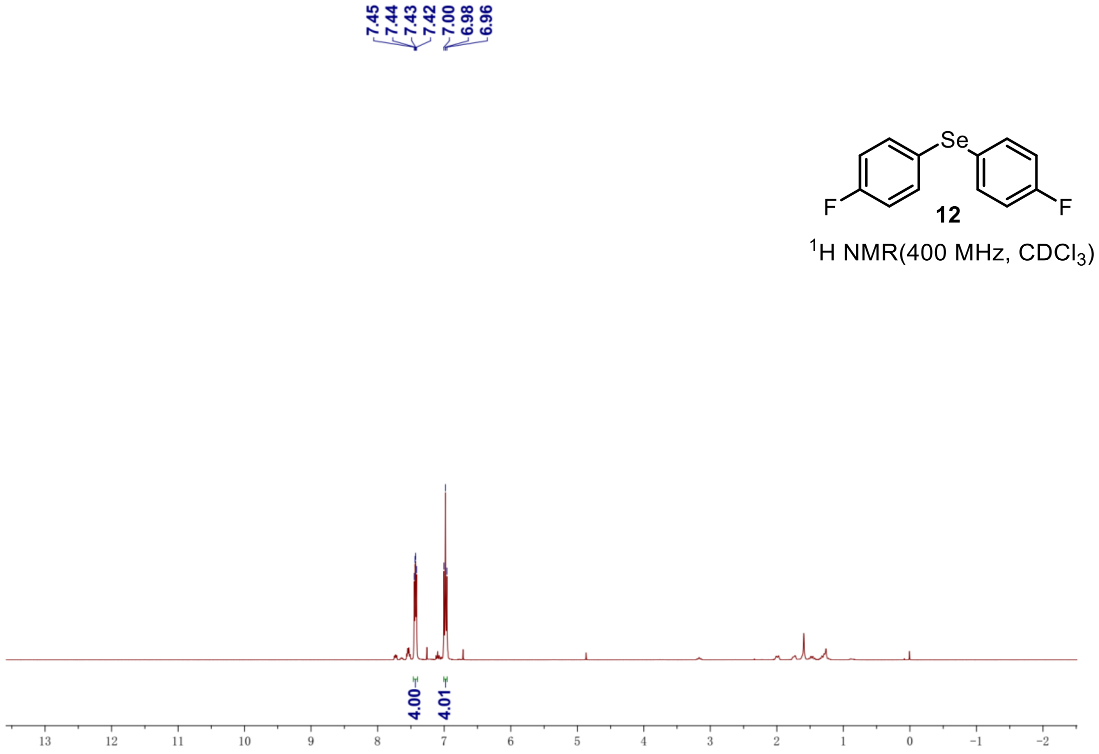


10

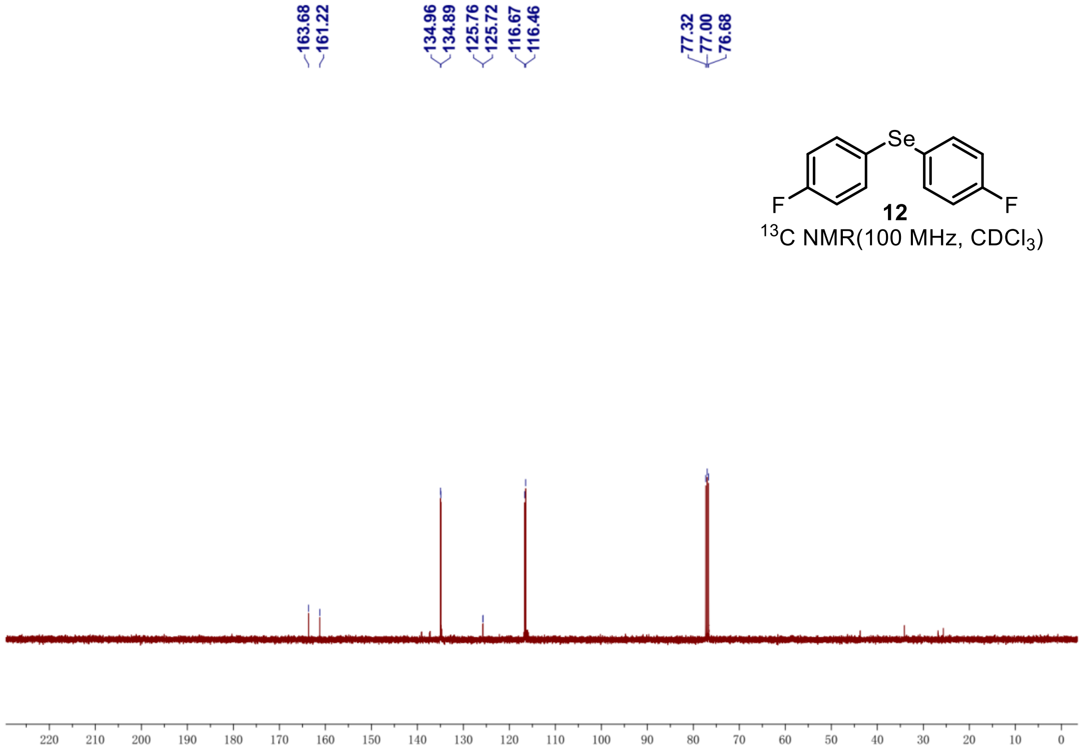
^{13}C NMR(100 MHz, CDCl$_3$)


7.59
7.57
7.17
7.15
Se
I
I
11
^{1}H NMR(400 MHz, CDCl$_3$)
4.13
4.00
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2

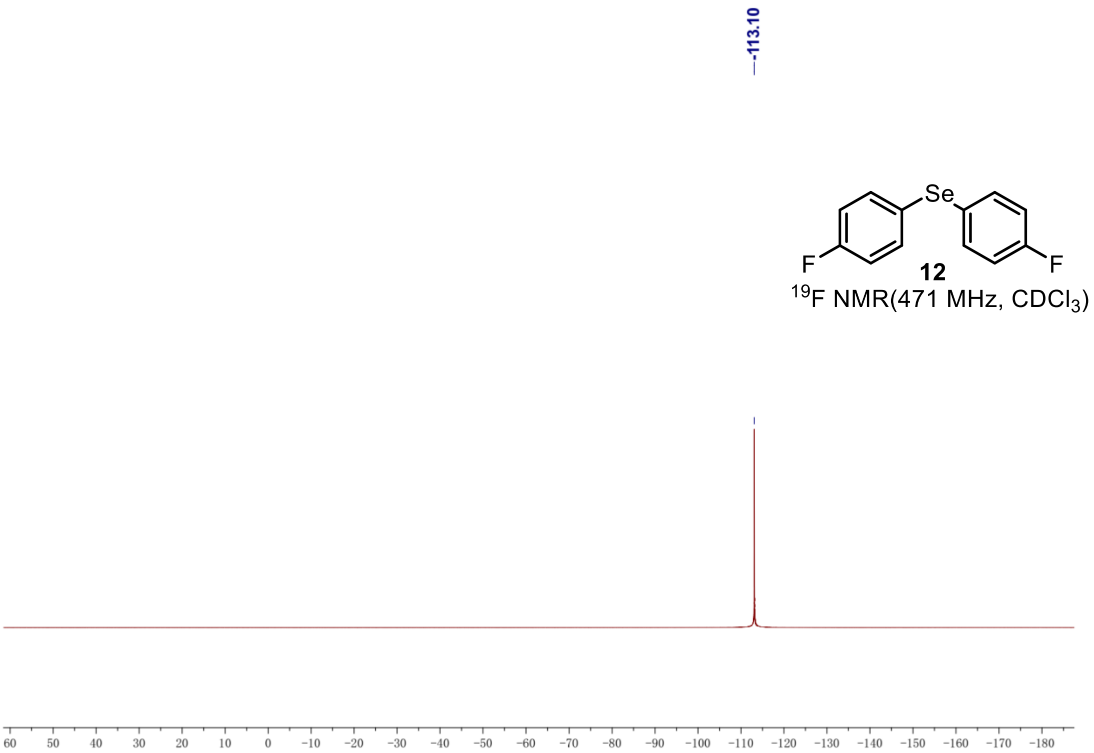

138.43
134.69
130.46
93.34
Se
I
I
11
^{13}C NMR(100 MHz, CDCl$_3$)
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
−20



7.45
7.44
7.43
7.42
7.00
6.98
6.96
Se
F
F
12
^{1}H NMR(400 MHz, CDCl$_3$)
4.00
4.01
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2

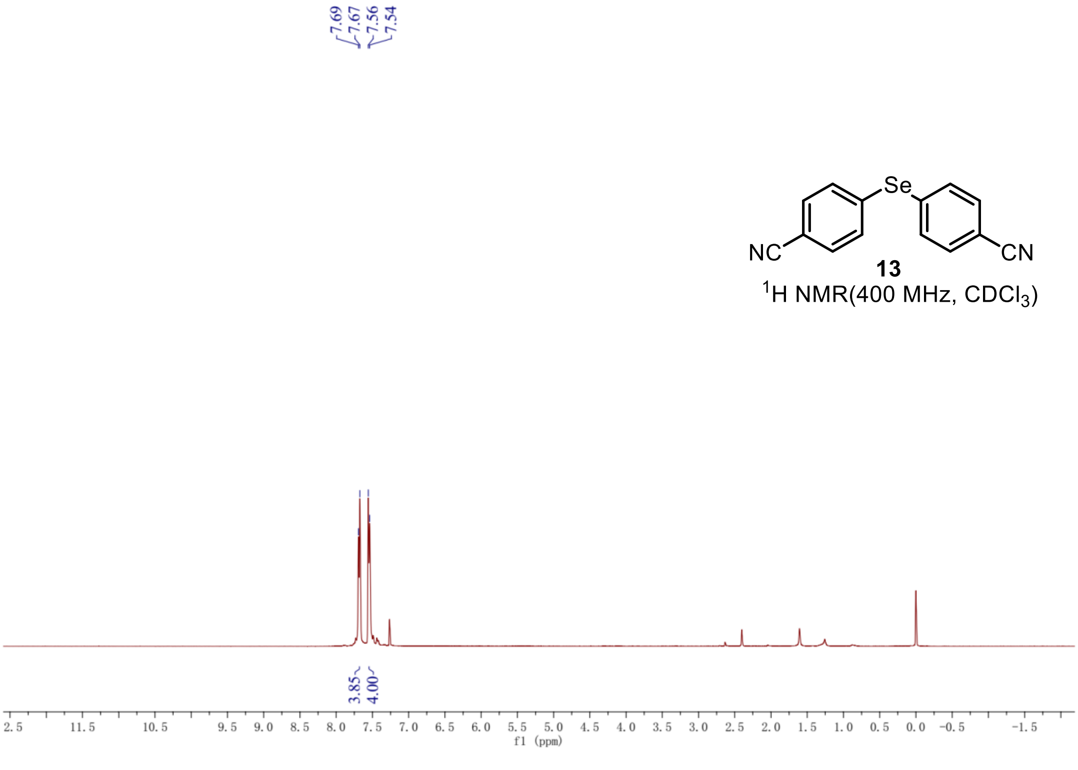


163.68
161.22
134.96
134.89
125.76
125.72
116.67
116.46
77.32
77.00
76.68
Se
F
F
12
^{13}C NMR(100 MHz, CDCl$_3$)
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



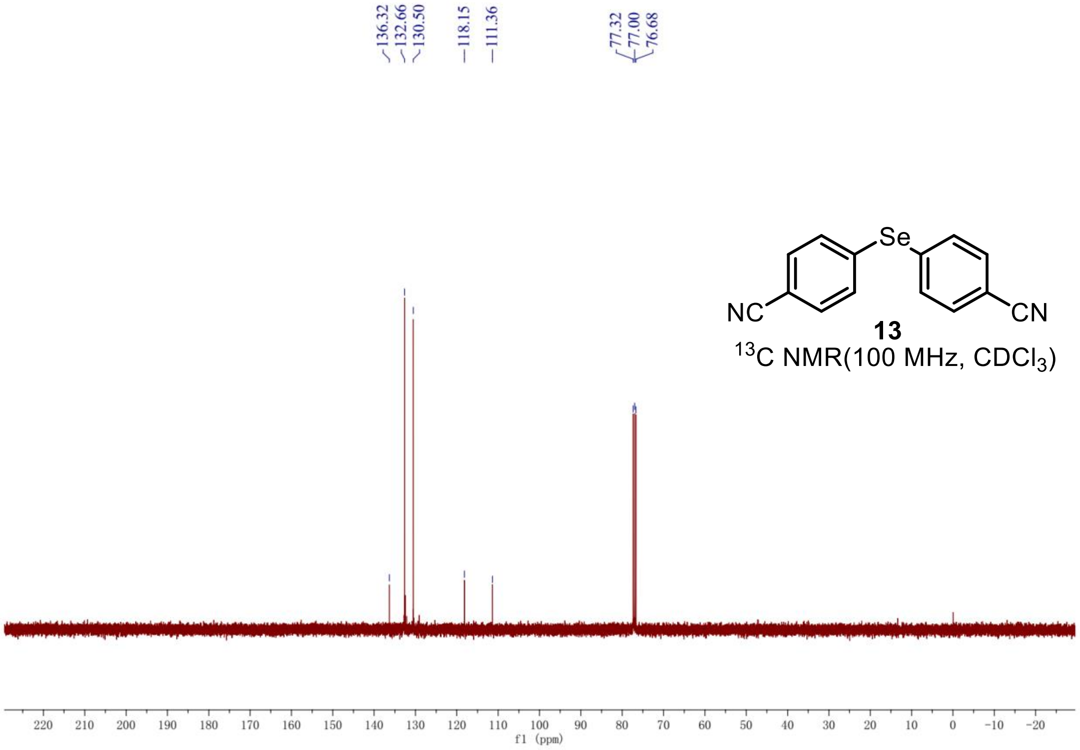
12

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



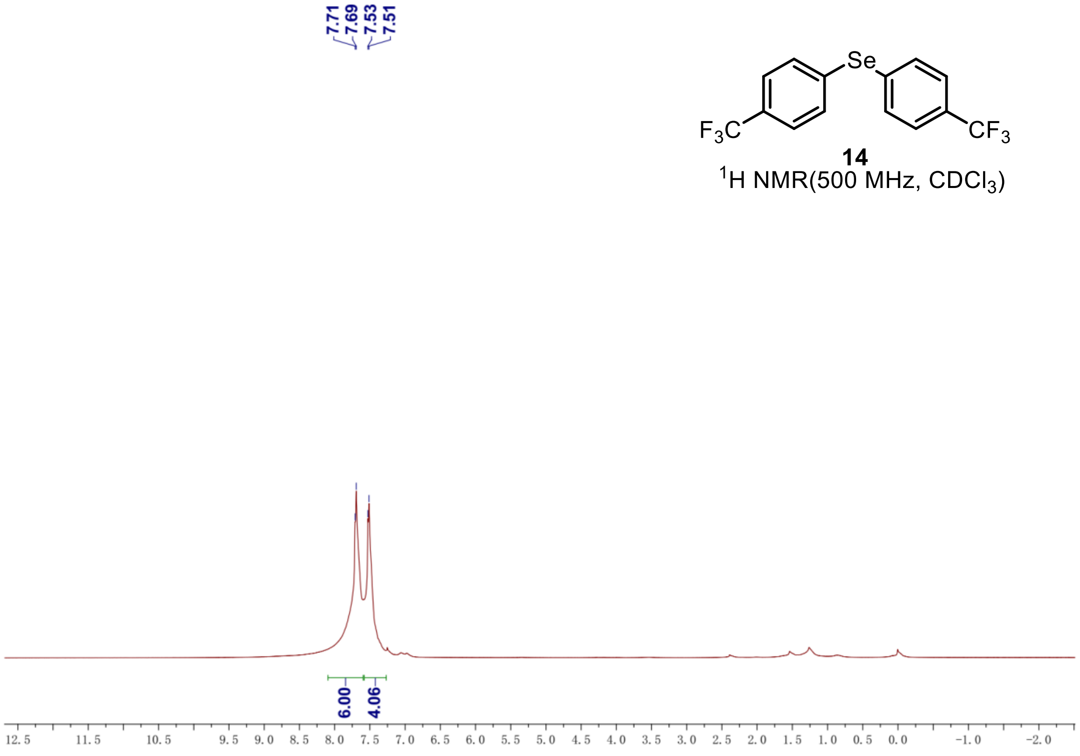
13

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



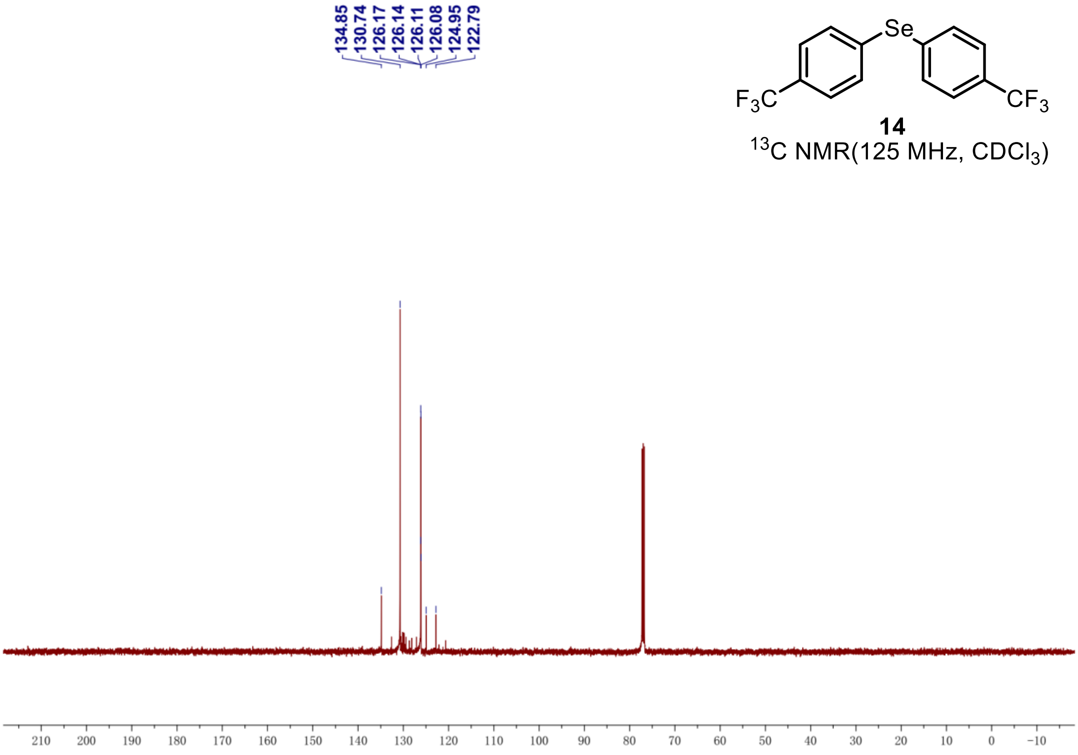
13

^{13}C NMR(100 MHz, $CDCl_3$)



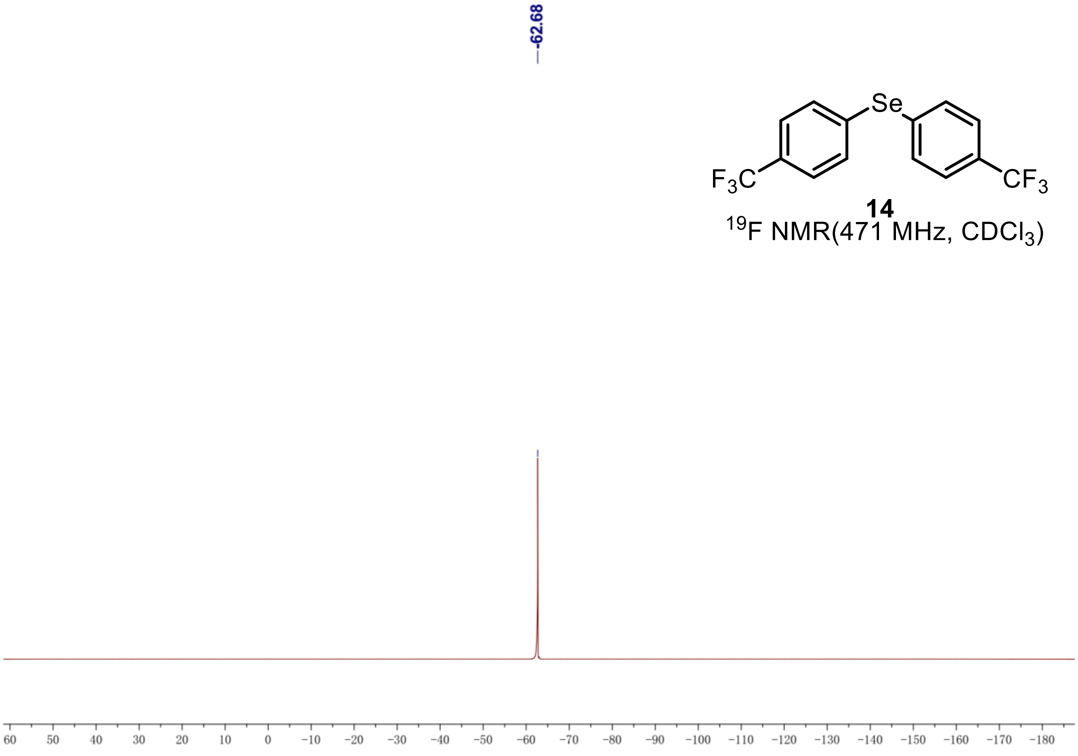
14

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



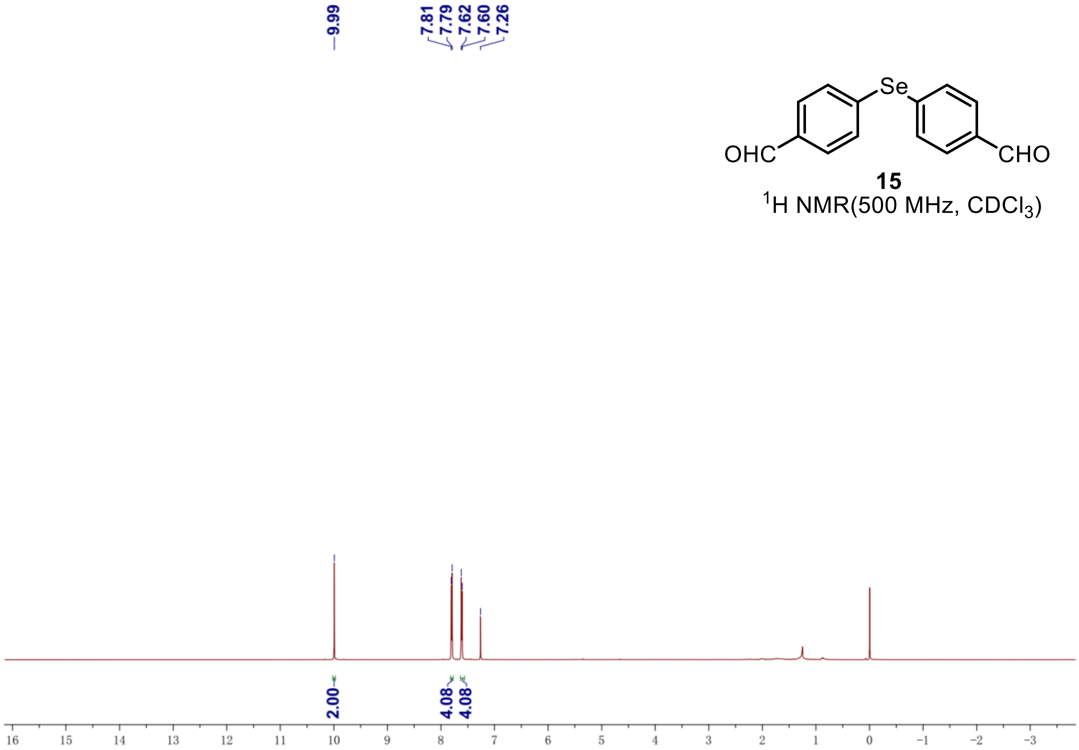
14

^{13}C NMR(125 MHz, CDCl$_3$)



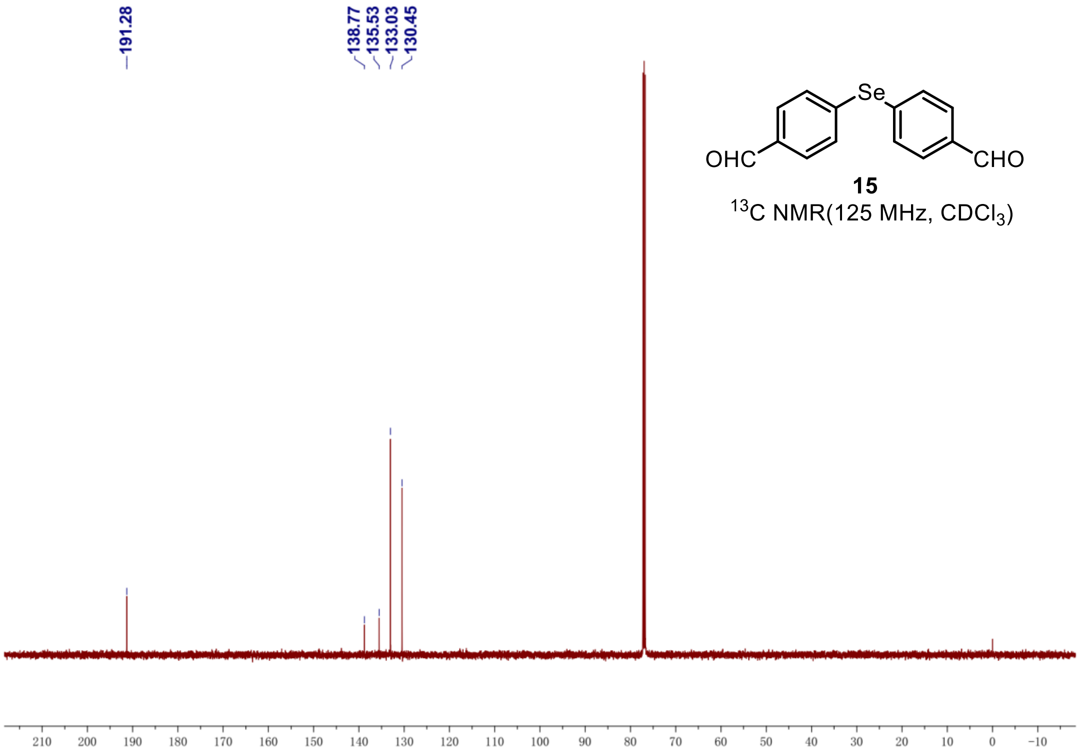
14

^{19}F NMR(471 MHz, CDCl$_3$)



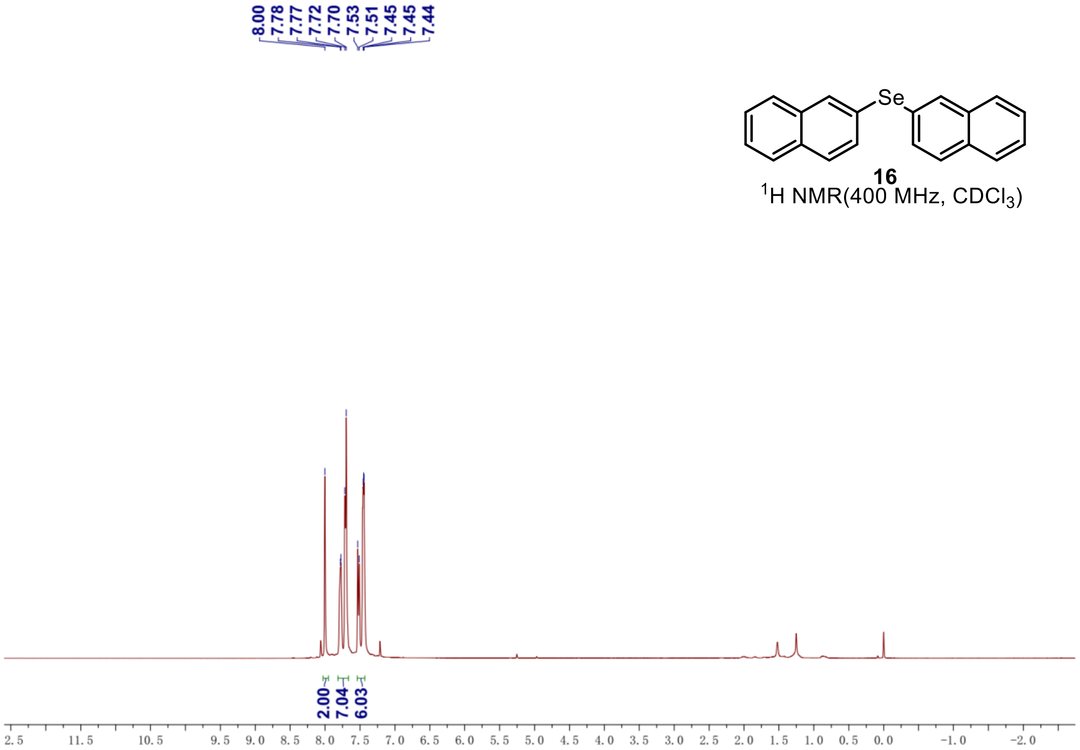
15

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



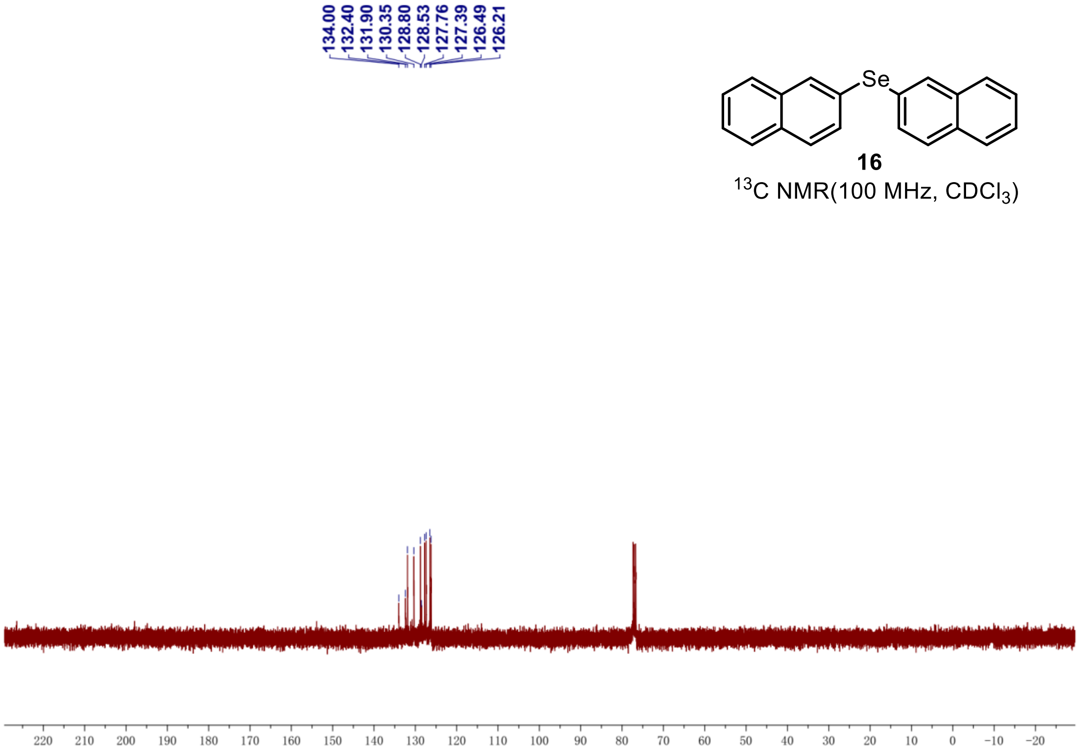
15

^{13}C NMR(125 MHz, $CDCl_3$)



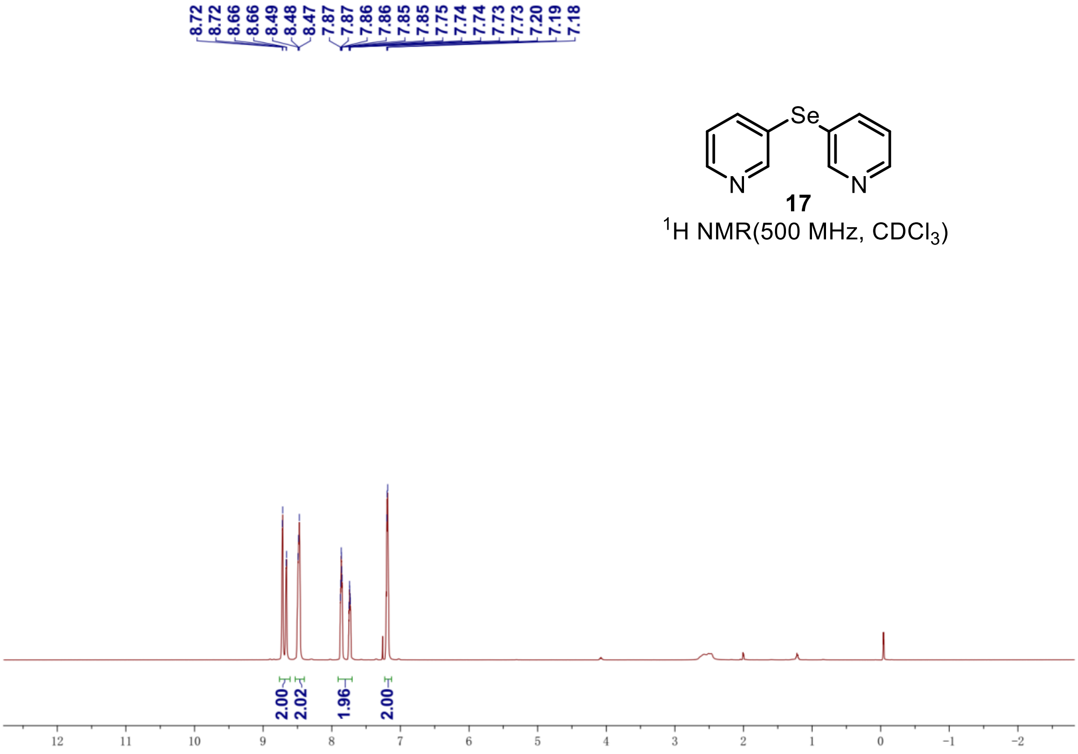
16

^{1}H NMR(400 MHz, CDCl$_3$)



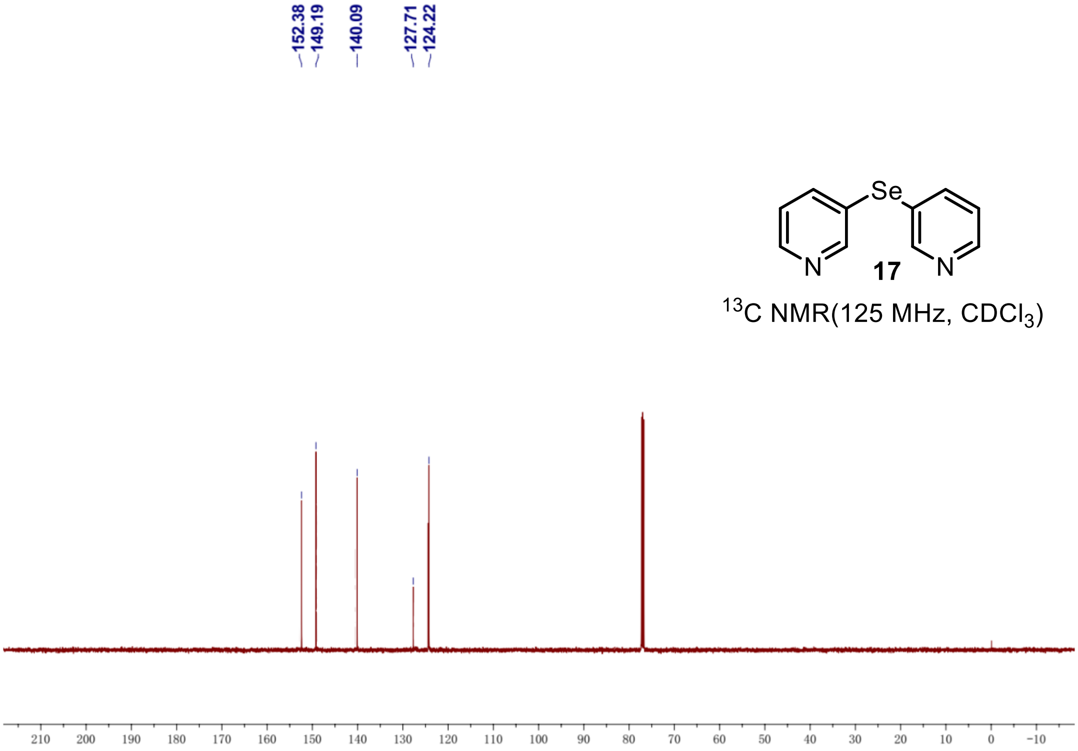
16

^{13}C NMR(100 MHz, CDCl$_3$)



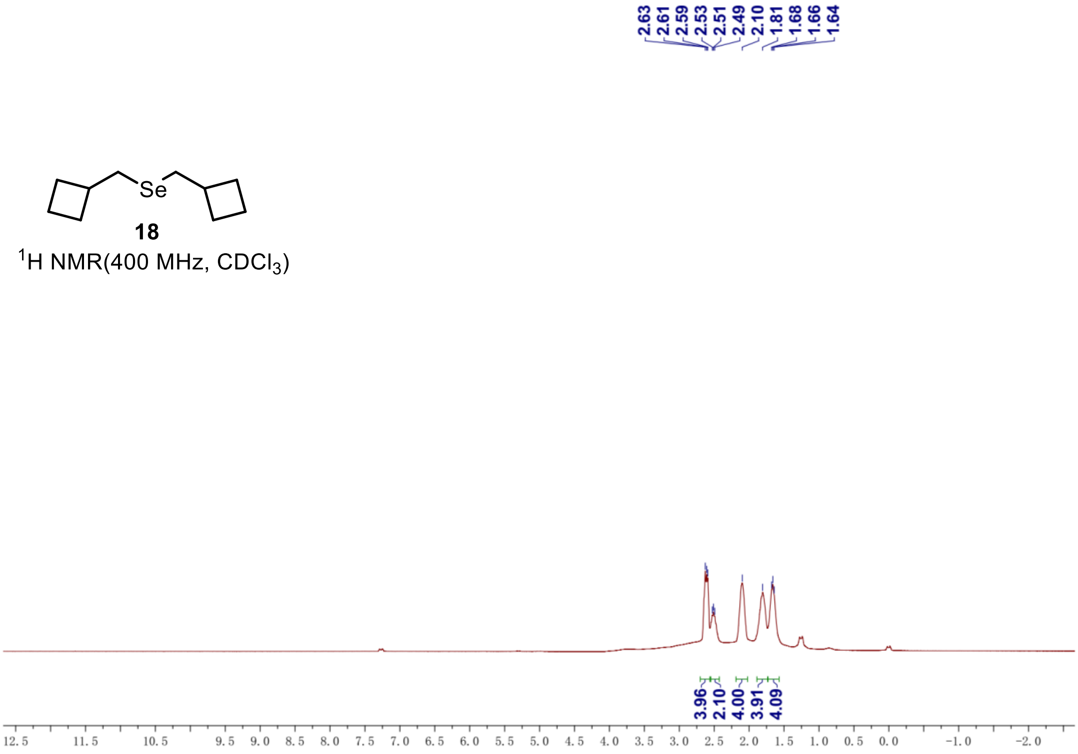
17

^{1}H NMR(500 MHz, CDCl$_3$)



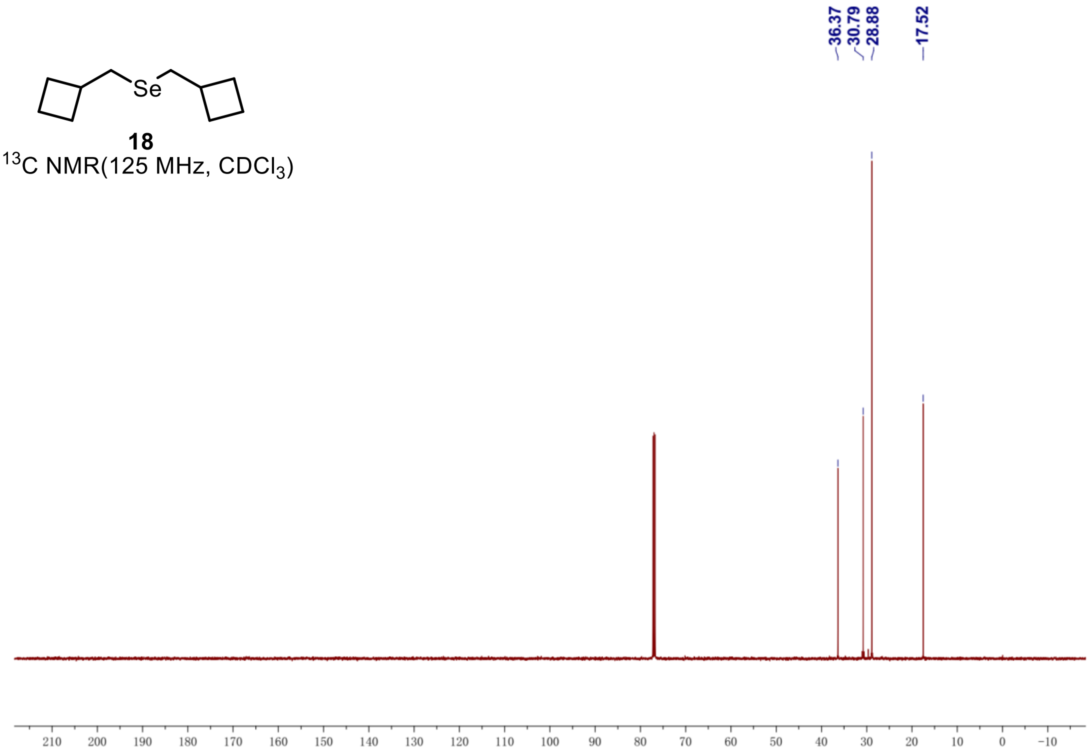
17

^{13}C NMR(125 MHz, CDCl$_3$)



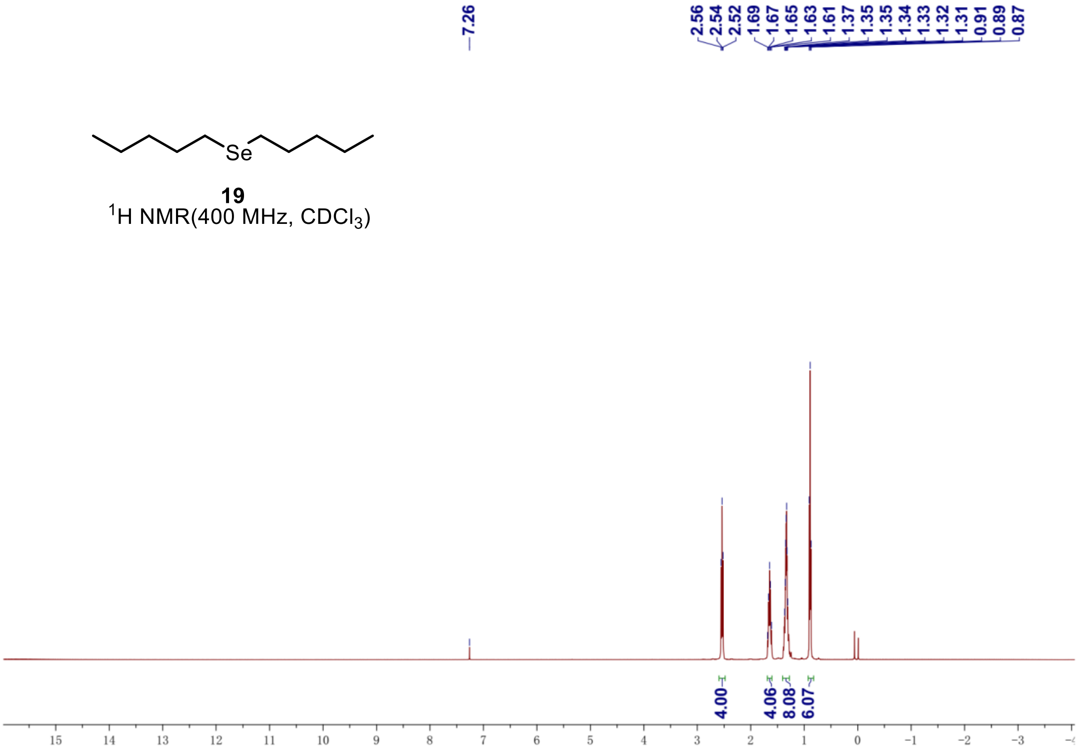
18

^{1}H NMR(400 MHz, CDCl$_3$)



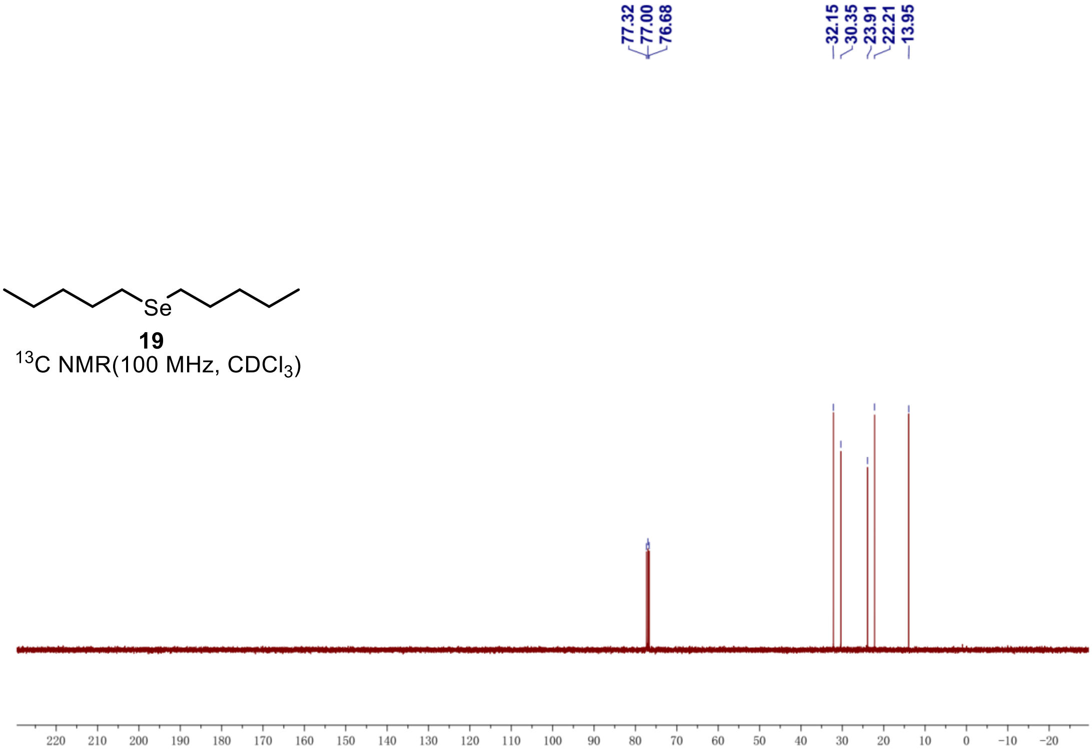
18

^{13}C NMR(125 MHz, CDCl$_3$)



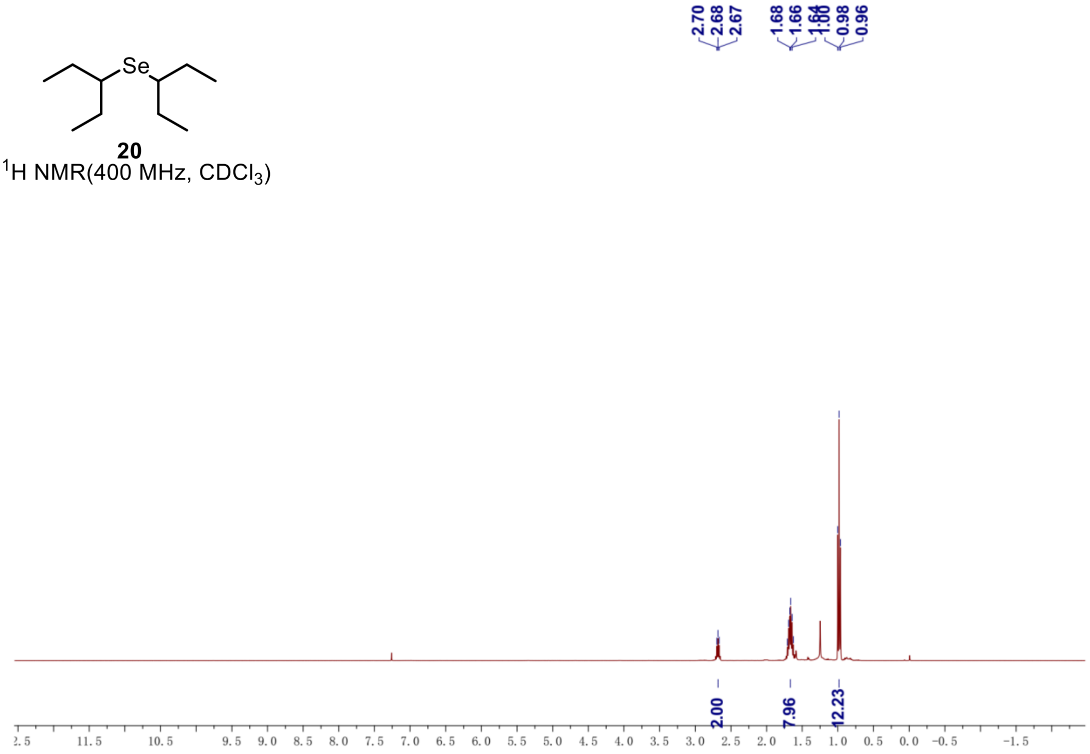
19

^{1}H NMR(400 MHz, CDCl$_3$)



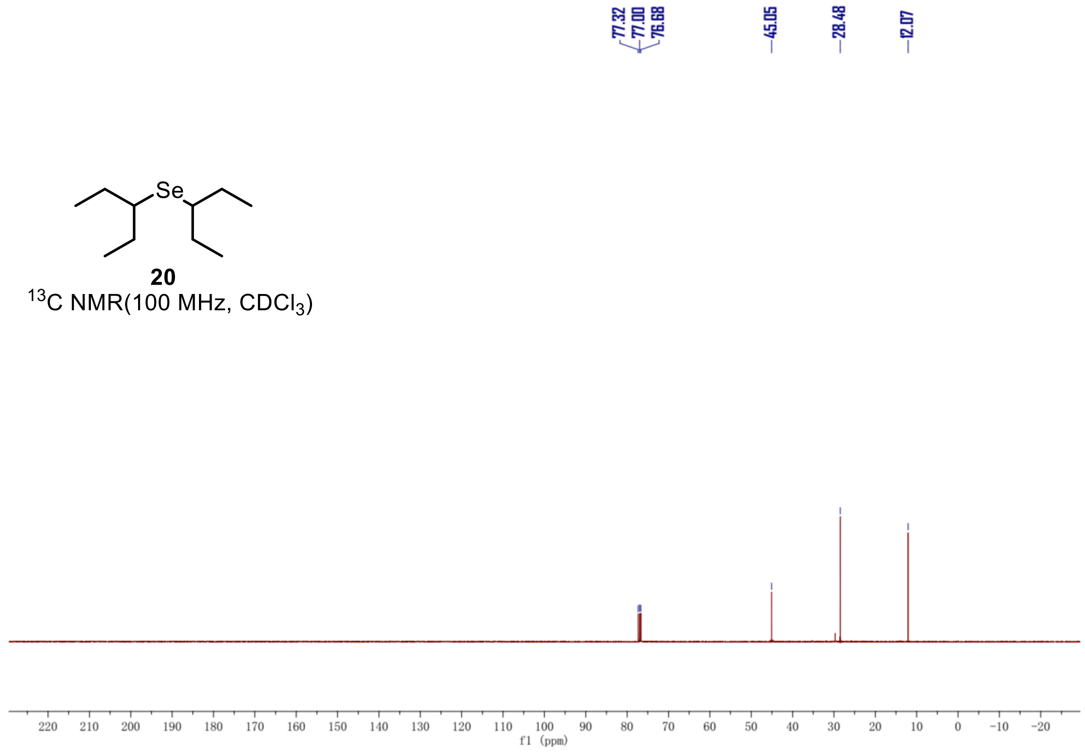
19

^{13}C NMR(100 MHz, CDCl$_3$)



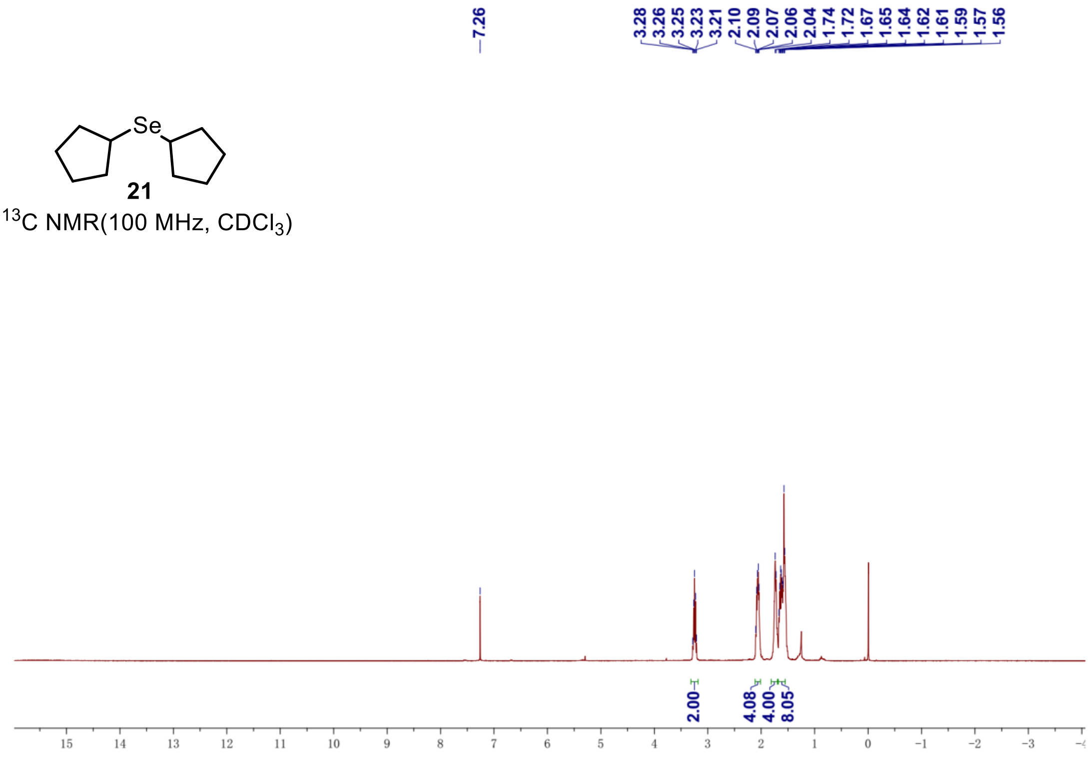
20

^{1}H NMR(400 MHz, CDCl$_3$)



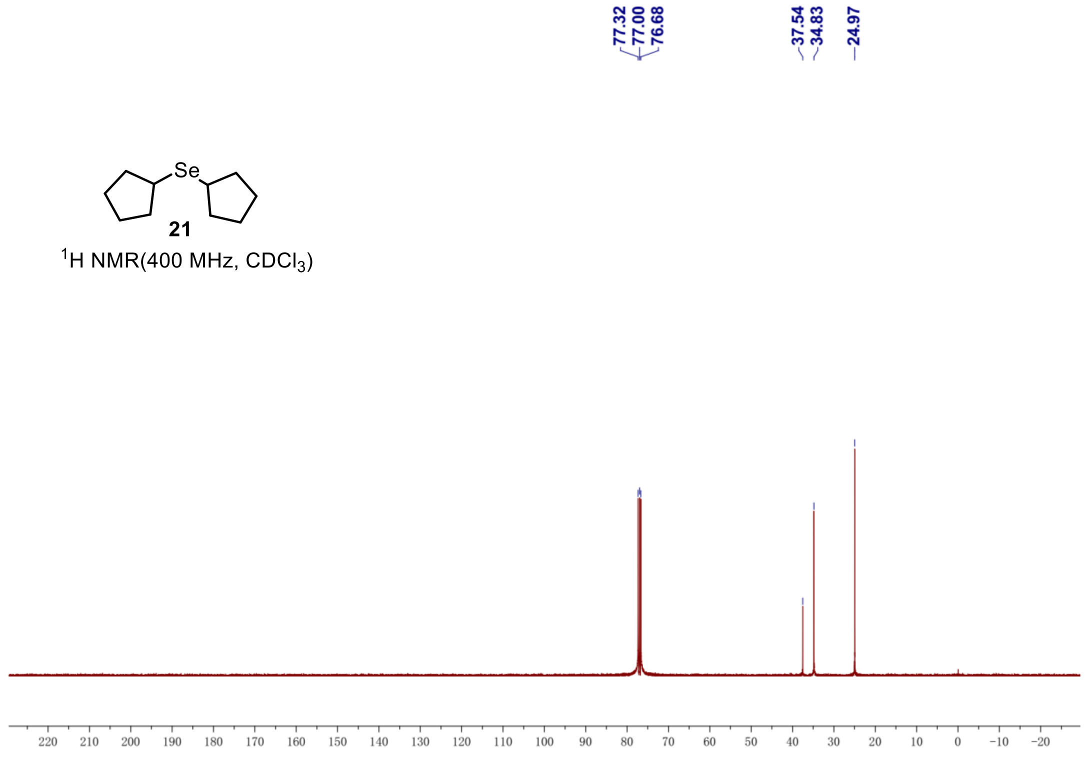
20

^{13}C NMR(100 MHz, CDCl$_3$)



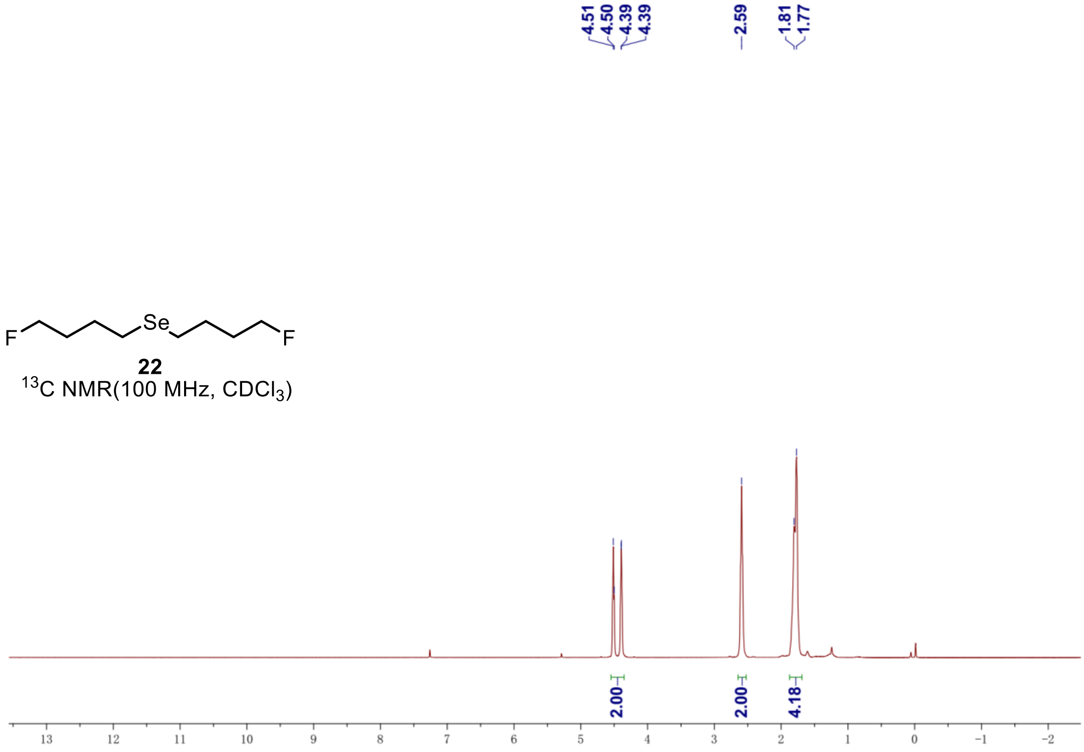
21

^{13}C NMR(100 MHz, CDCl$_3$)



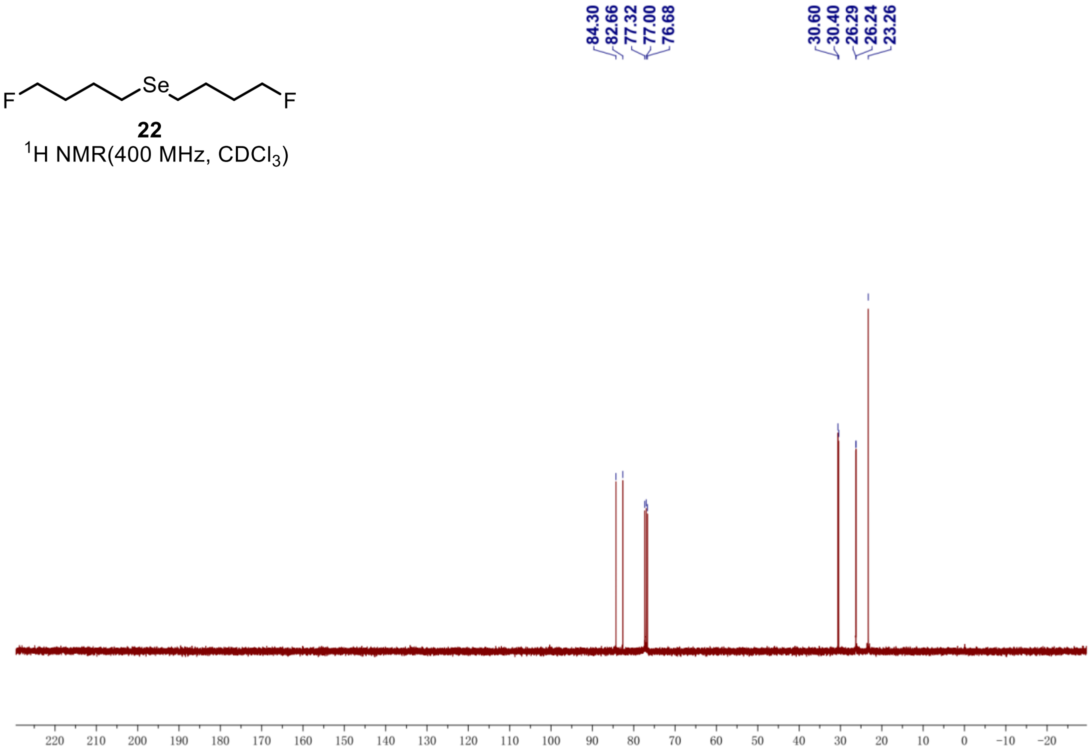
21

^{1}H NMR(400 MHz, CDCl$_3$)



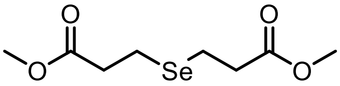
22

^{13}C NMR(100 MHz, CDCl$_3$)



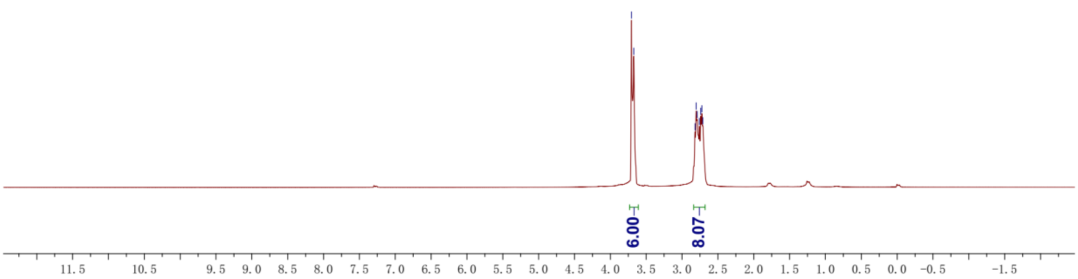
22

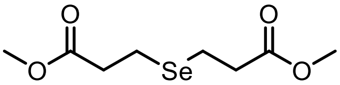
^{1}H NMR(400 MHz, CDCl$_3$)



23

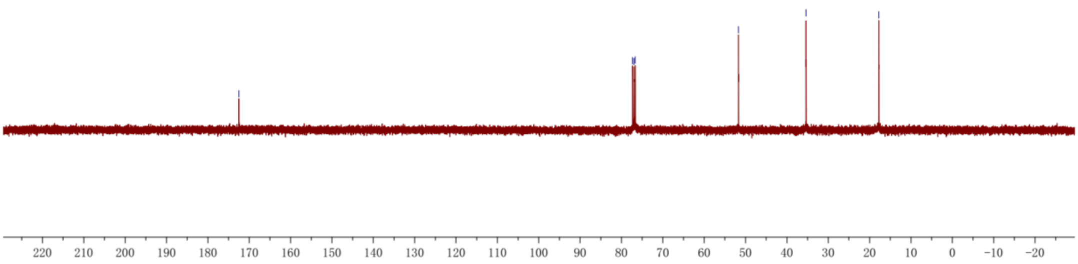
^{13}C NMR(100 MHz, $CDCl_3$)

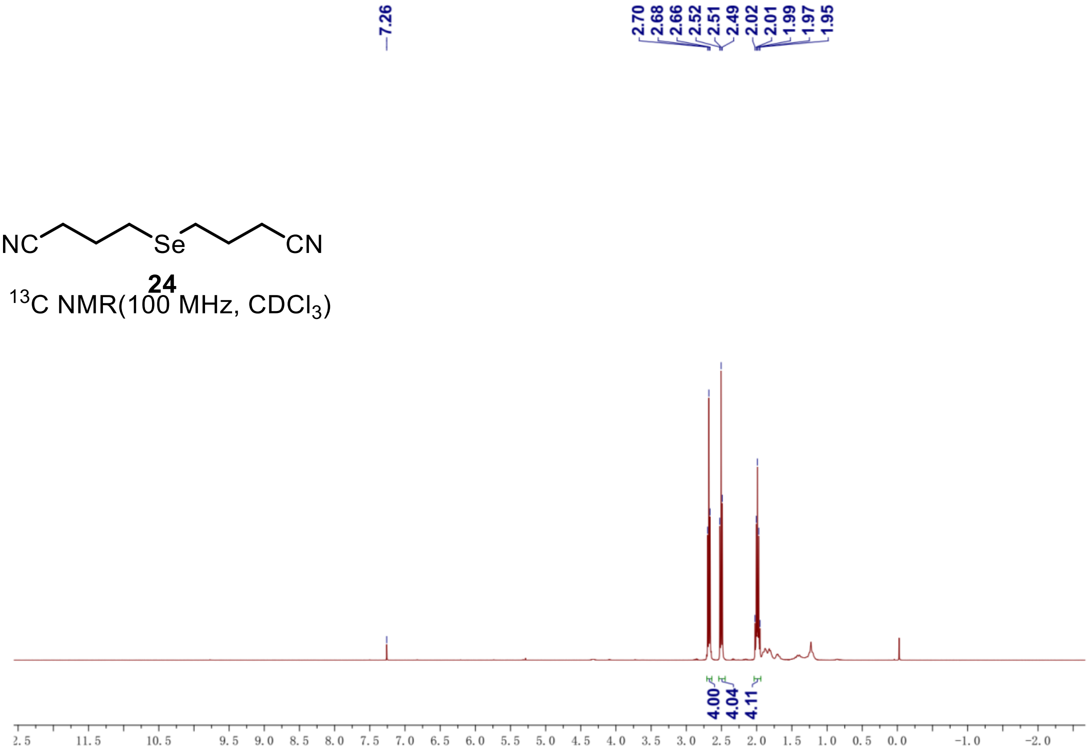




23

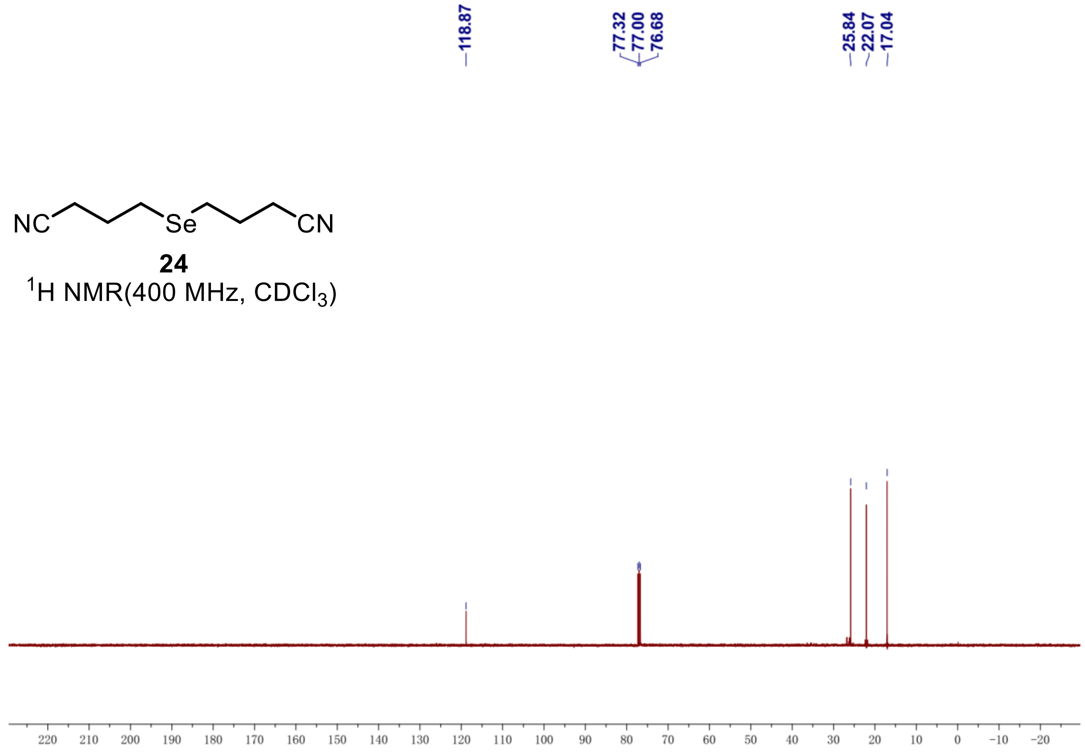
^{1}H NMR(400 MHz, $CDCl_3$)





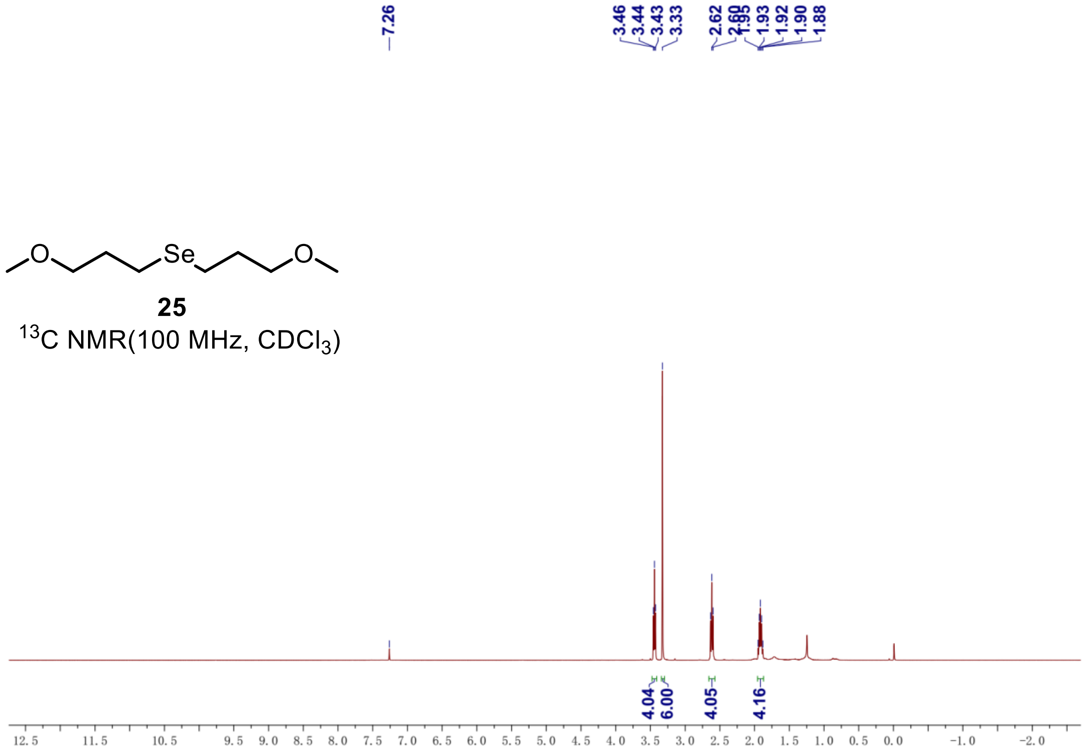
24

^{13}C NMR(100 MHz, CDCl$_3$)



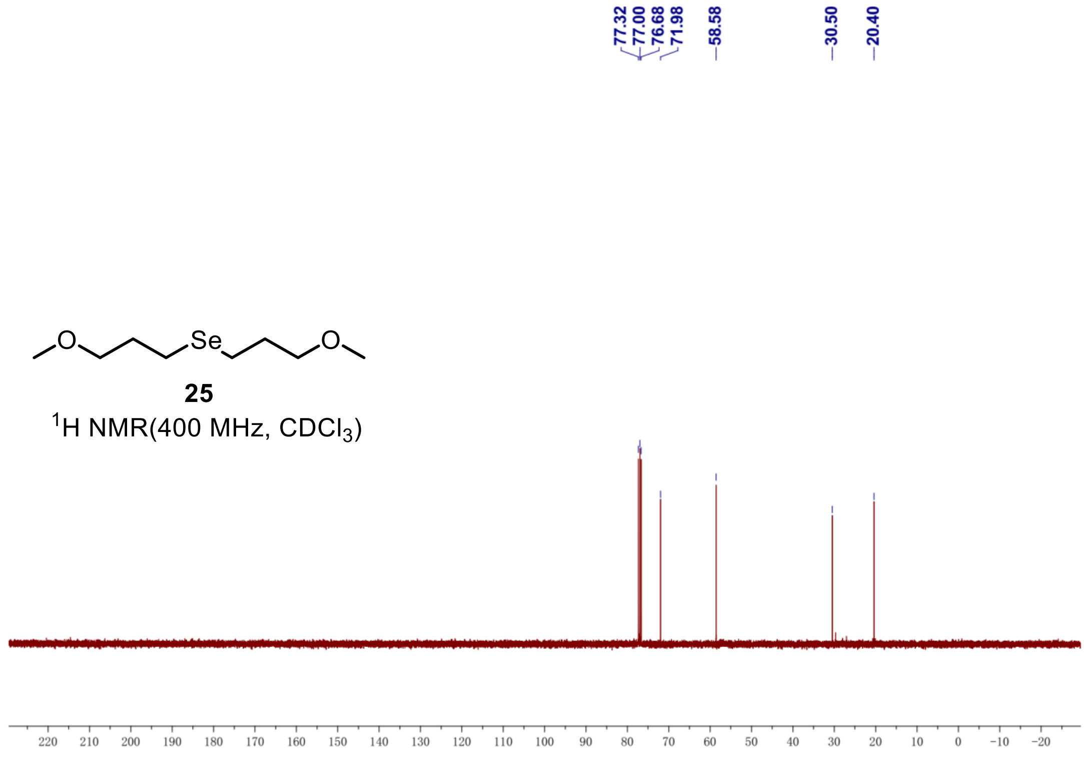
24

^{1}H NMR(400 MHz, CDCl$_3$)



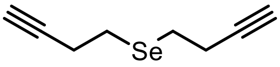
25

^{13}C NMR(100 MHz, CDCl$_3$)



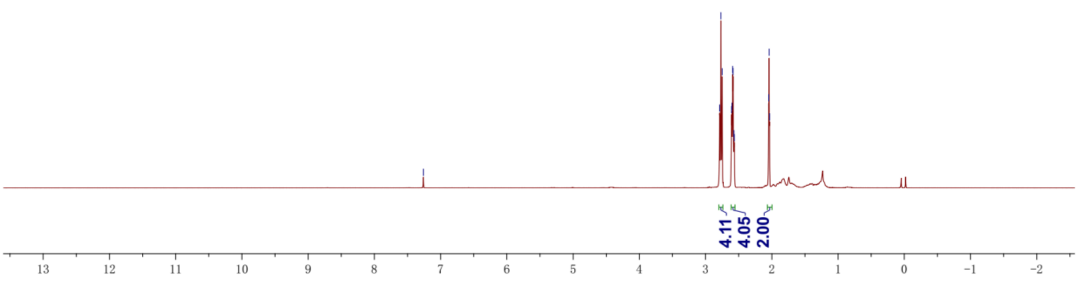
25

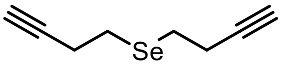
^{1}H NMR(400 MHz, CDCl$_3$)



26

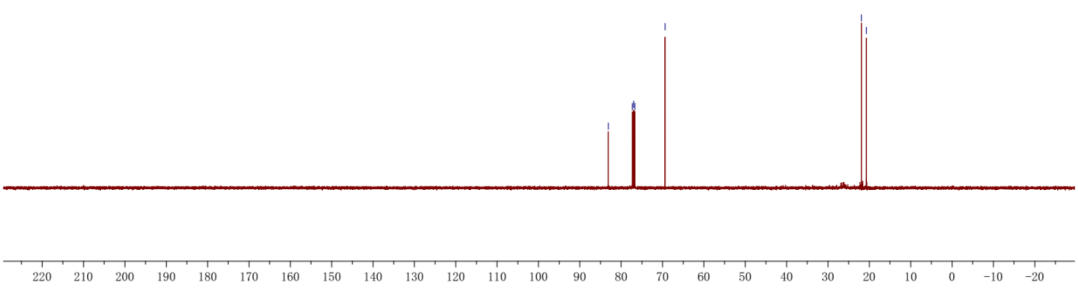
^{13}C NMR(100 MHz, CDCl$_3$)

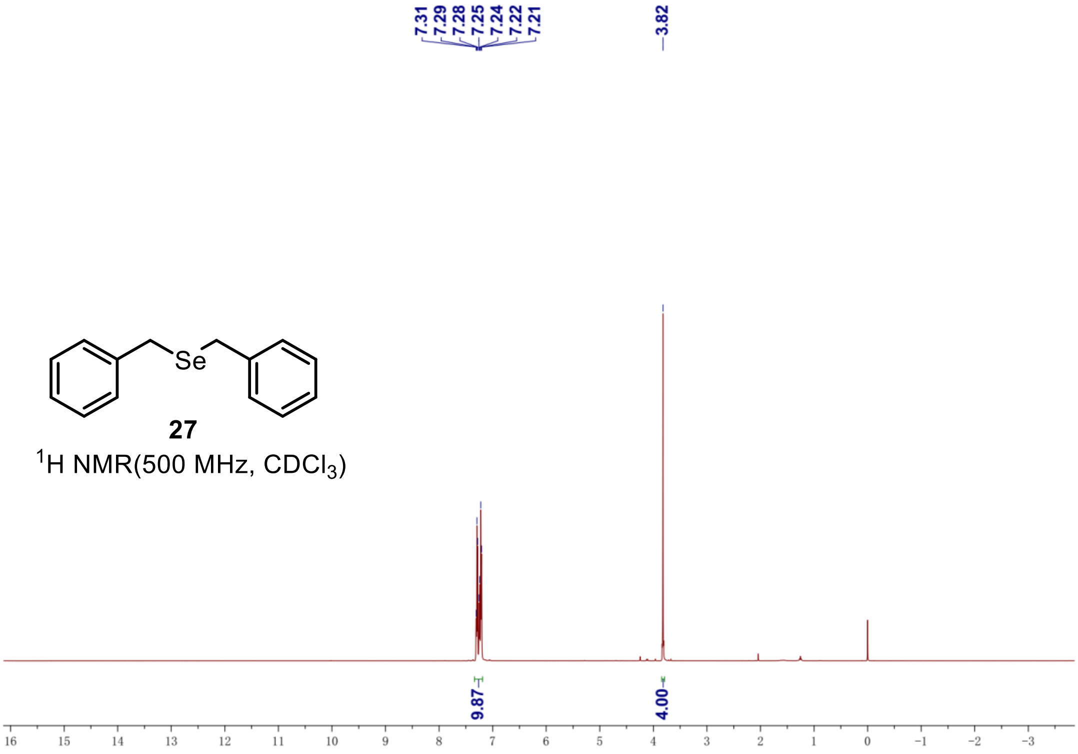




26

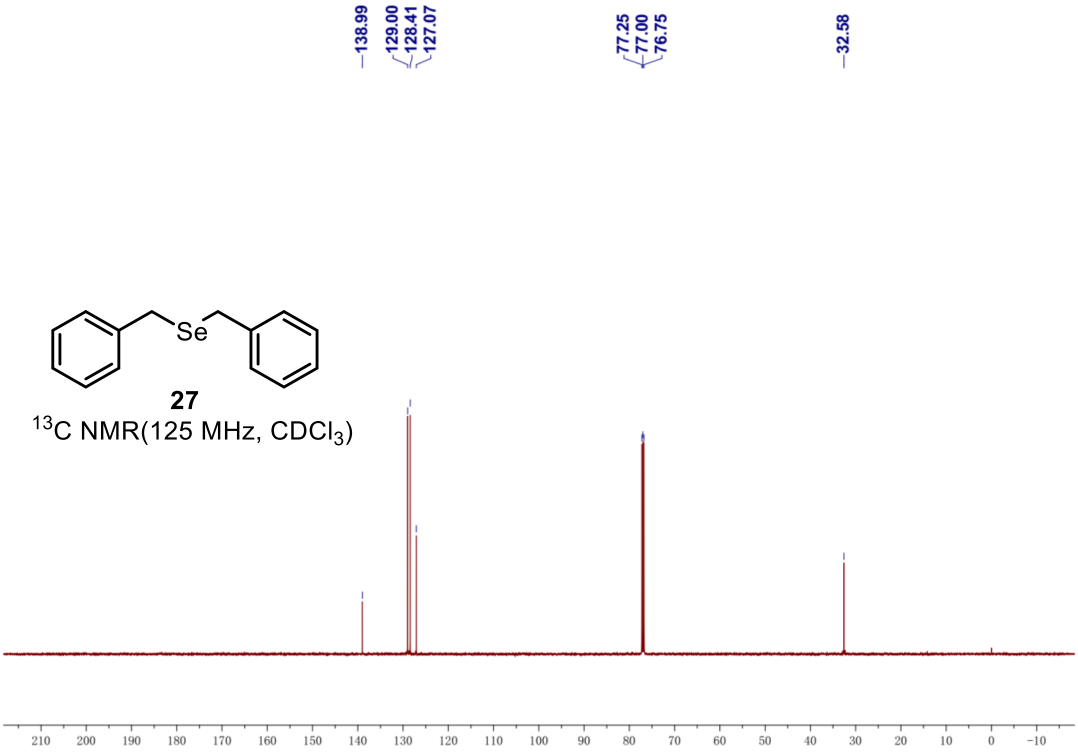
^{1}H NMR(400 MHz, CDCl$_3$)





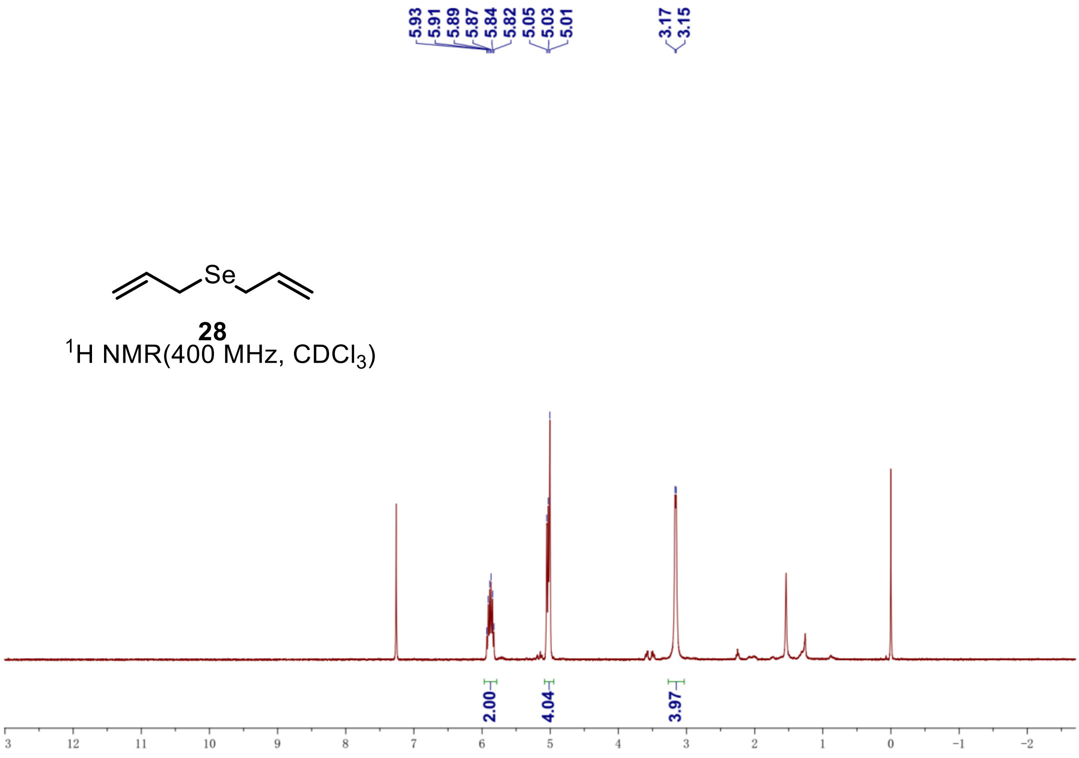
27

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

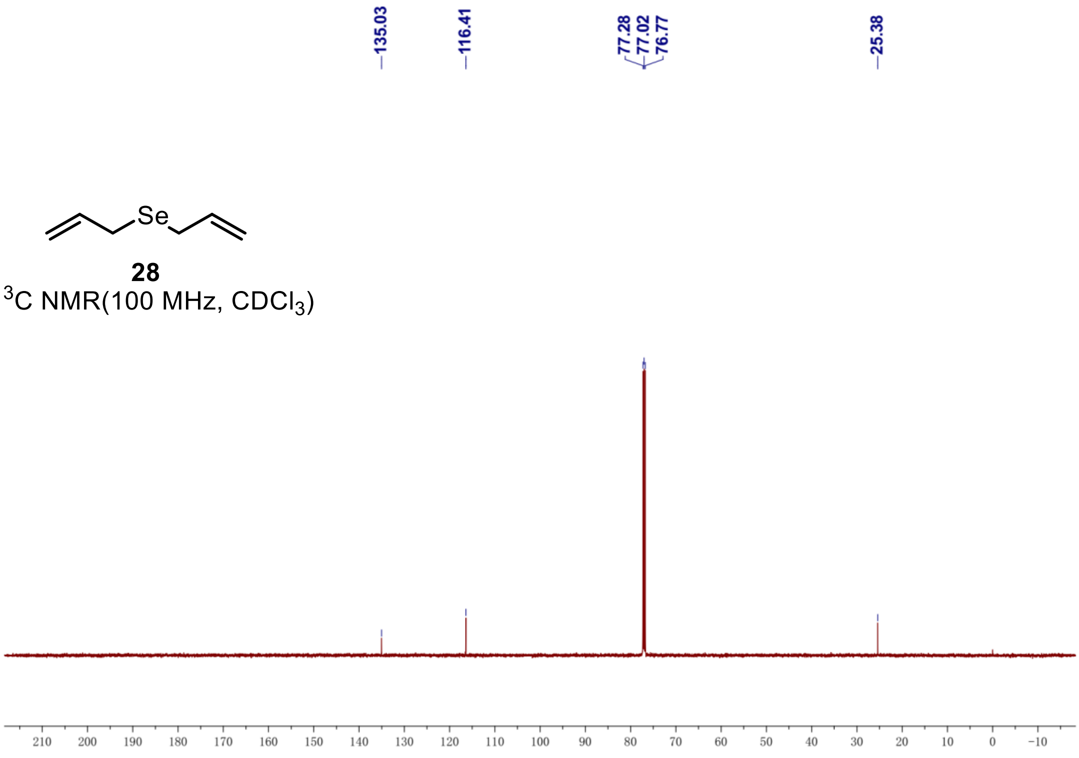


27

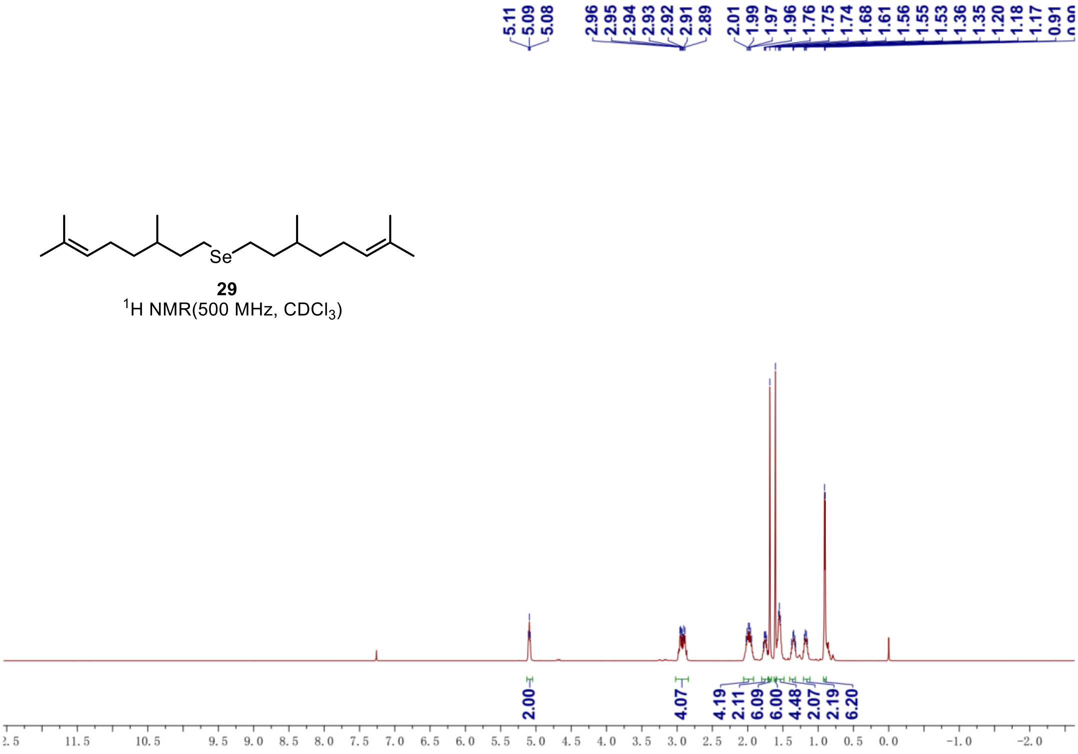
^{13}C NMR(125 MHz, $CDCl_3$)



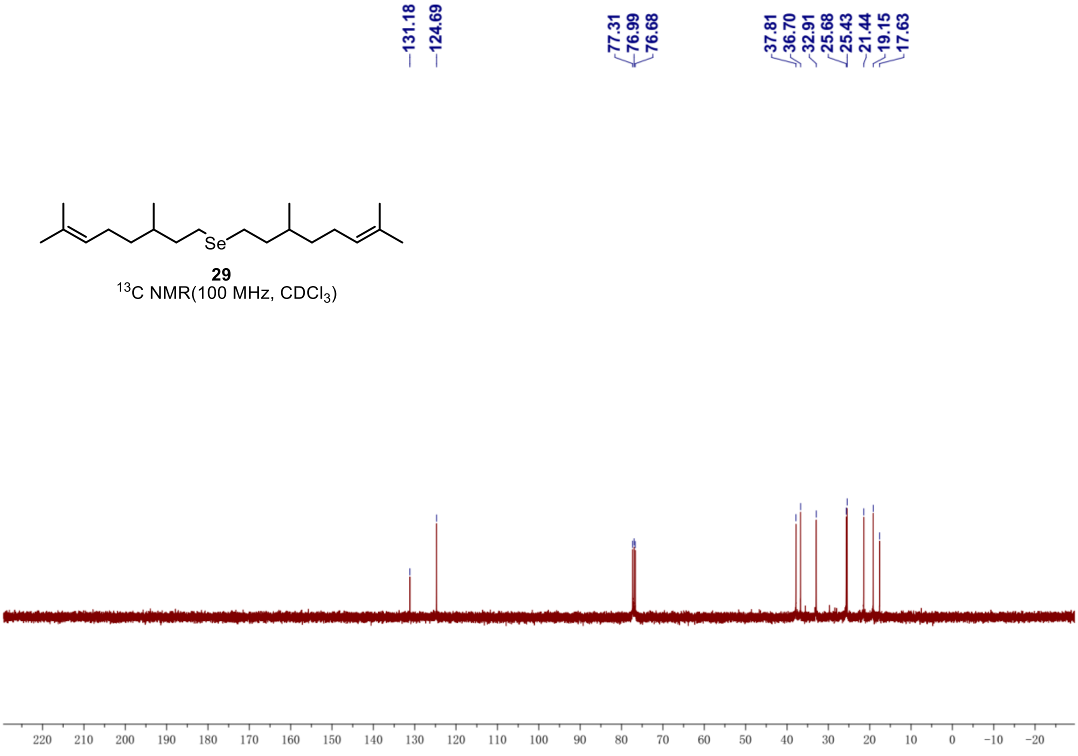
28
^{1}H NMR(400 MHz, CDCl$_3$)



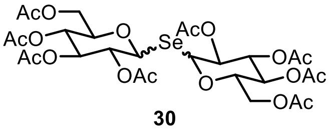
28
^{13}C NMR(100 MHz, CDCl$_3$)



29
^{1}H NMR(500 MHz, CDCl$_3$)

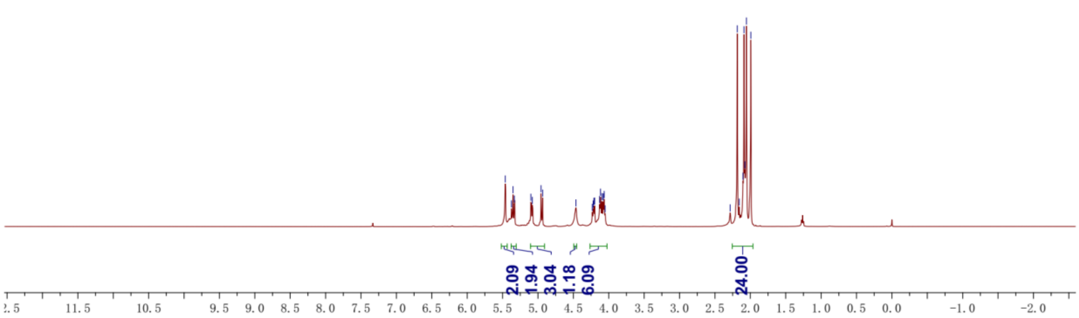


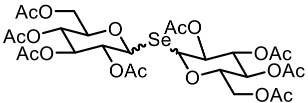
29
^{13}C NMR(100 MHz, CDCl$_3$)



30

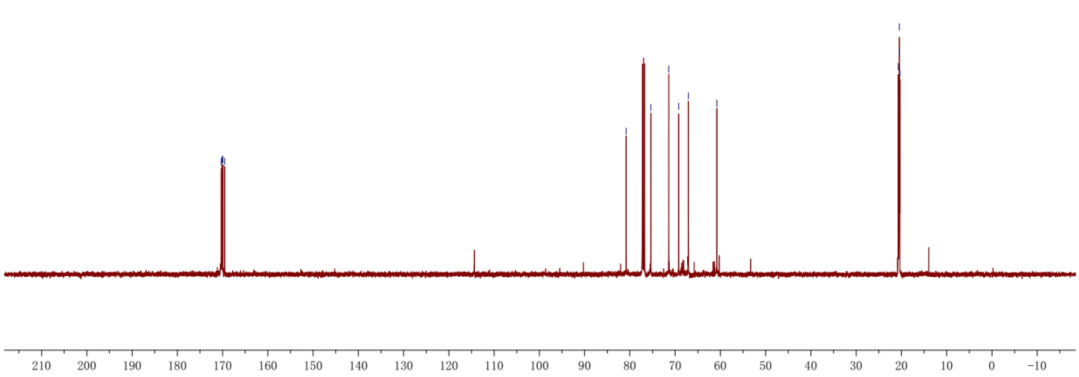
^{1}H NMR(500 MHz, CDCl$_3$)

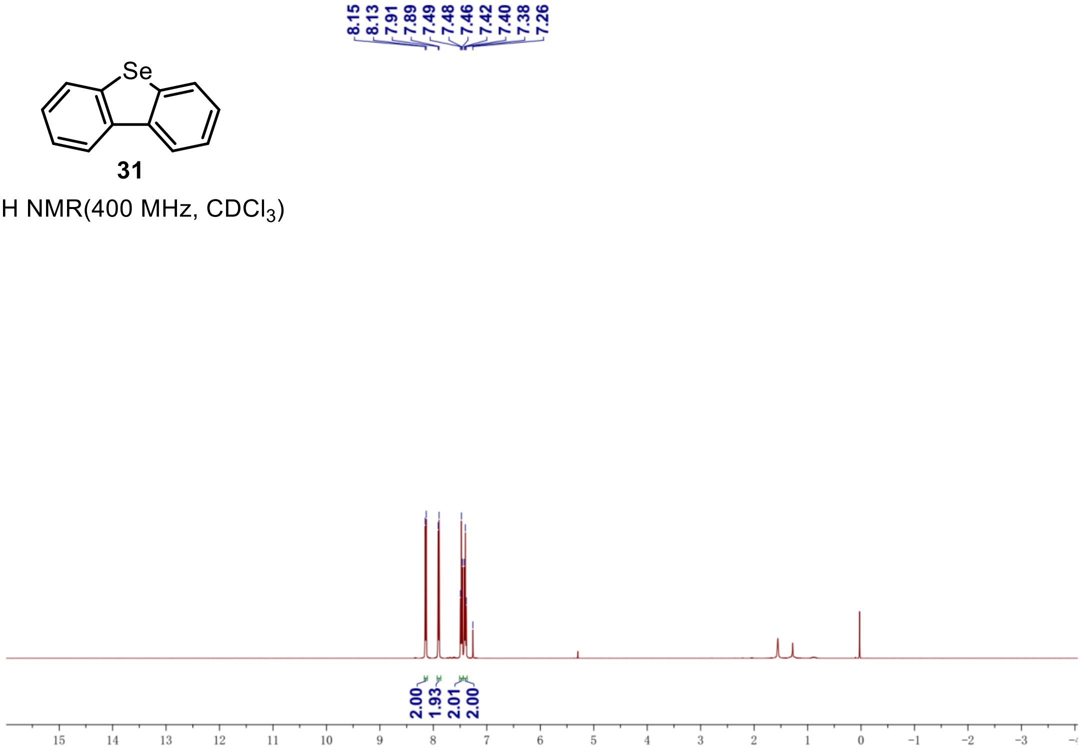




30

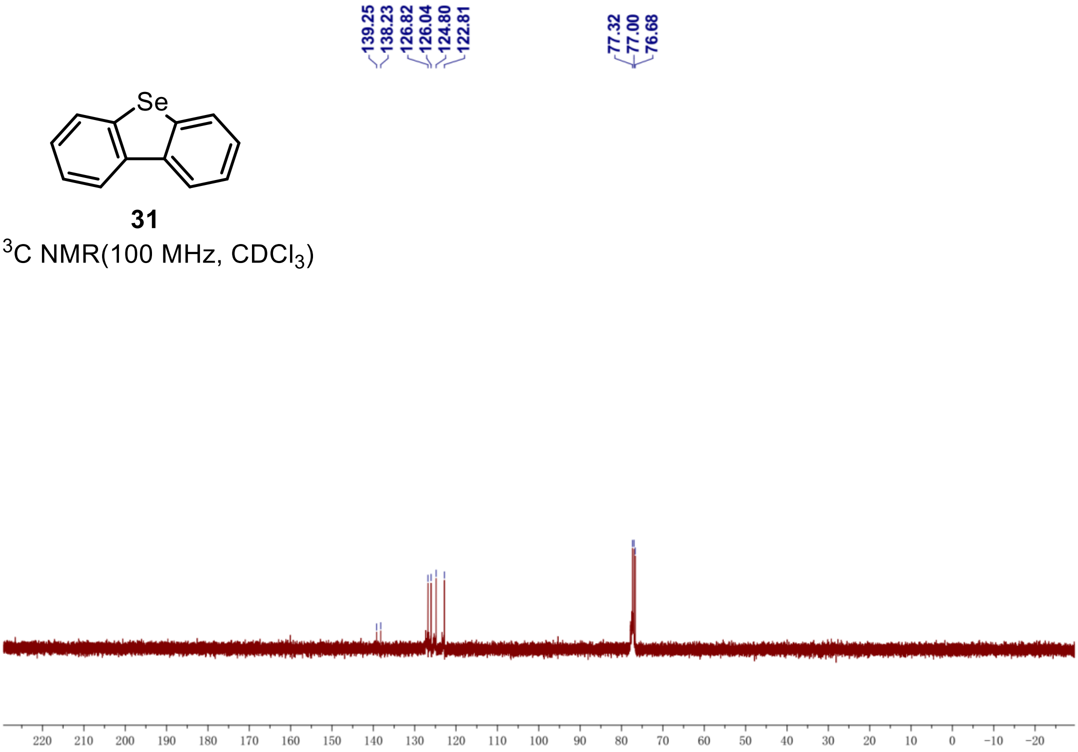
^{13}C NMR(125 MHz, CDCl$_3$)





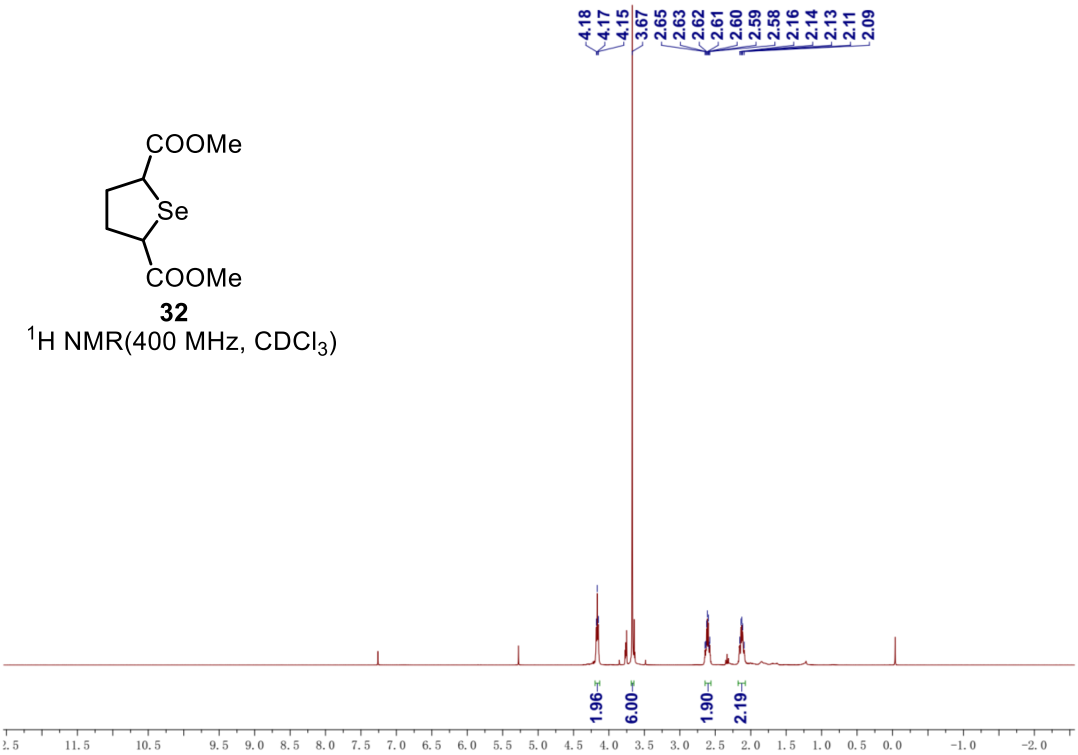
31

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



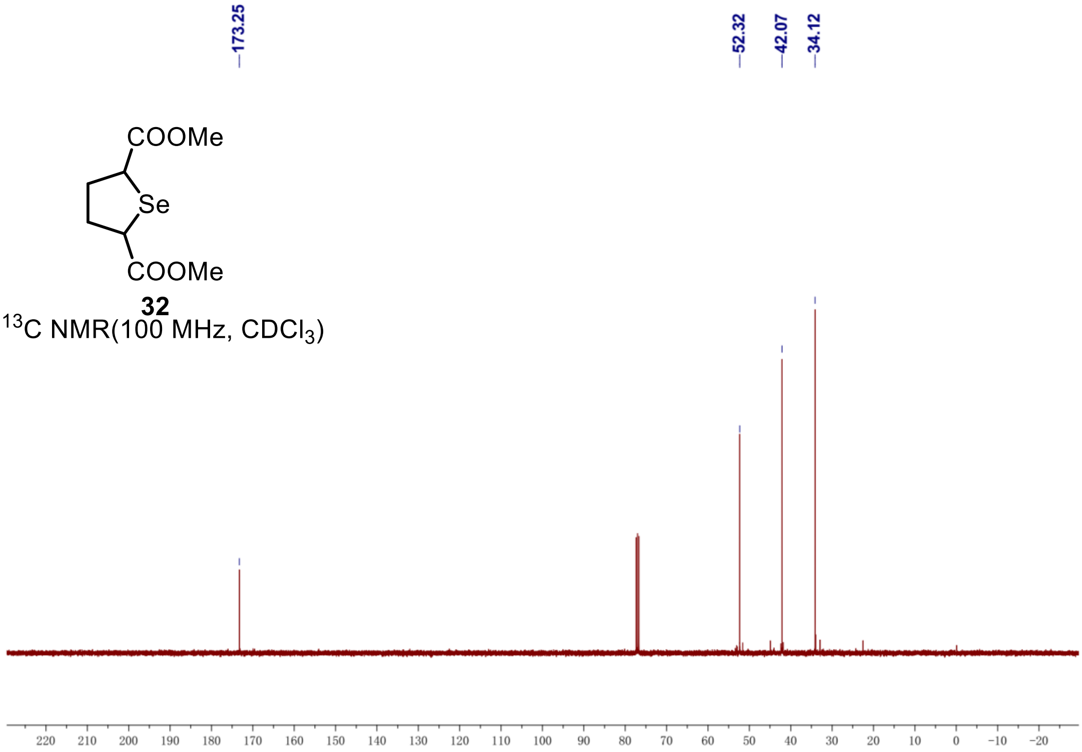
31

^{13}C NMR(100 MHz, $CDCl_3$)



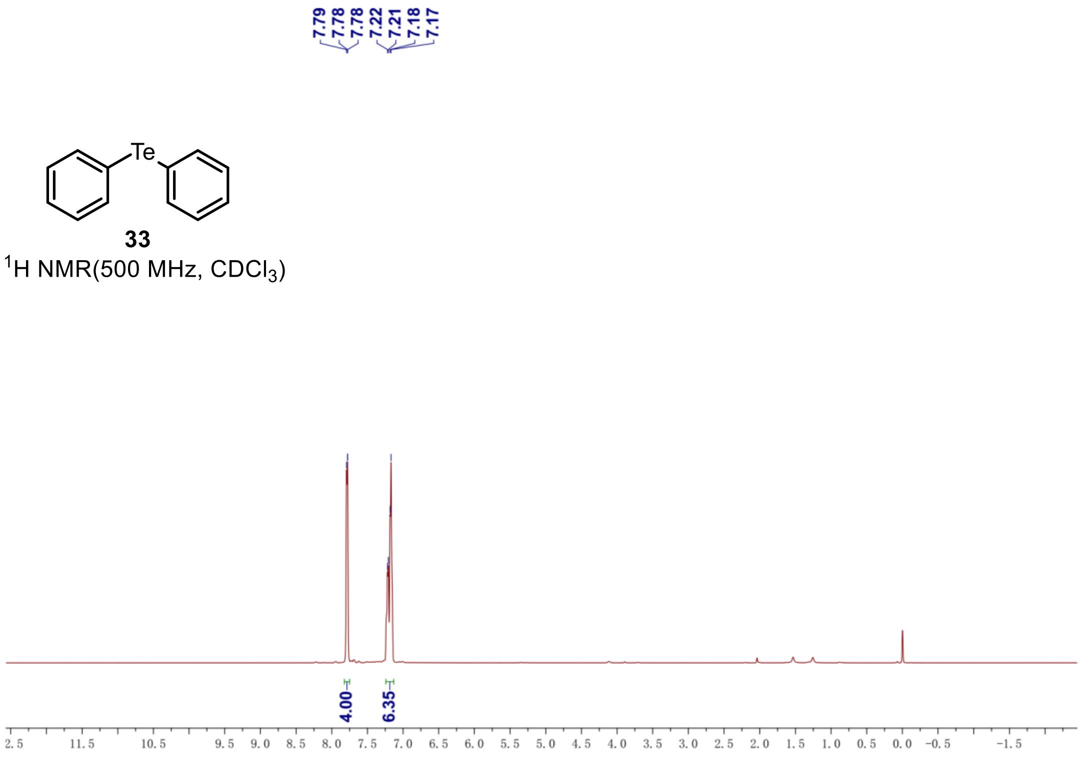
32

^1H NMR(400 MHz, $CDCl_3$)



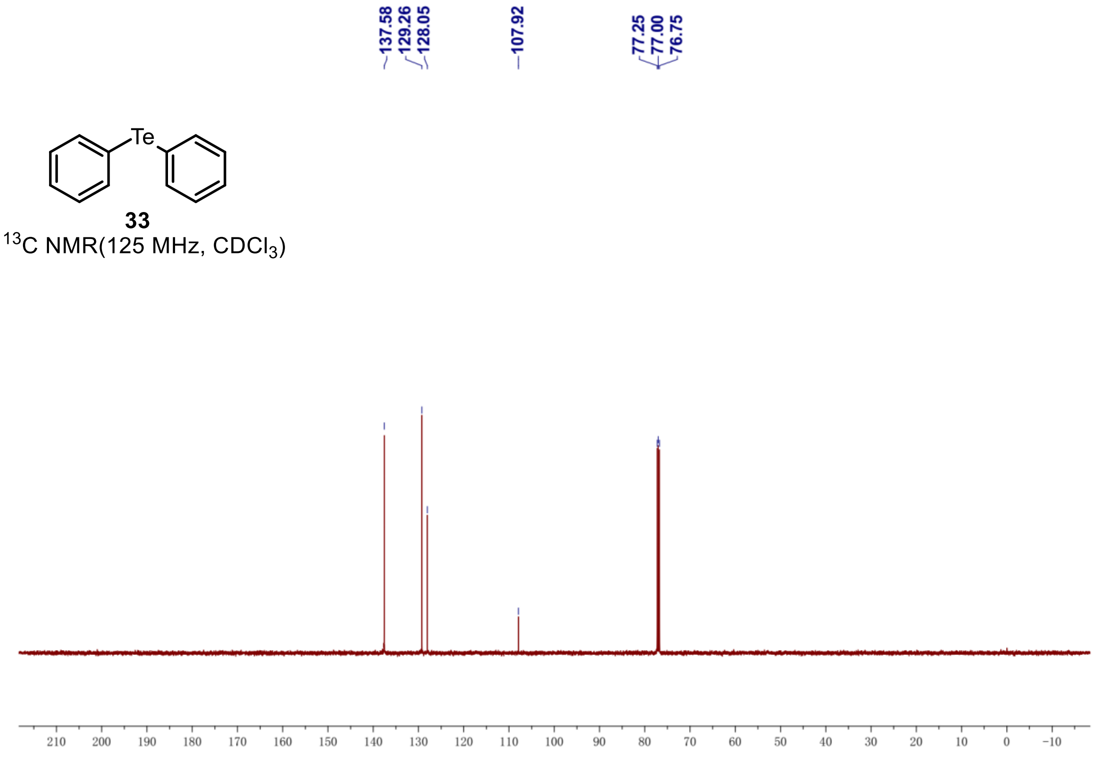
32

^{13}C NMR(100 MHz, $CDCl_3$)



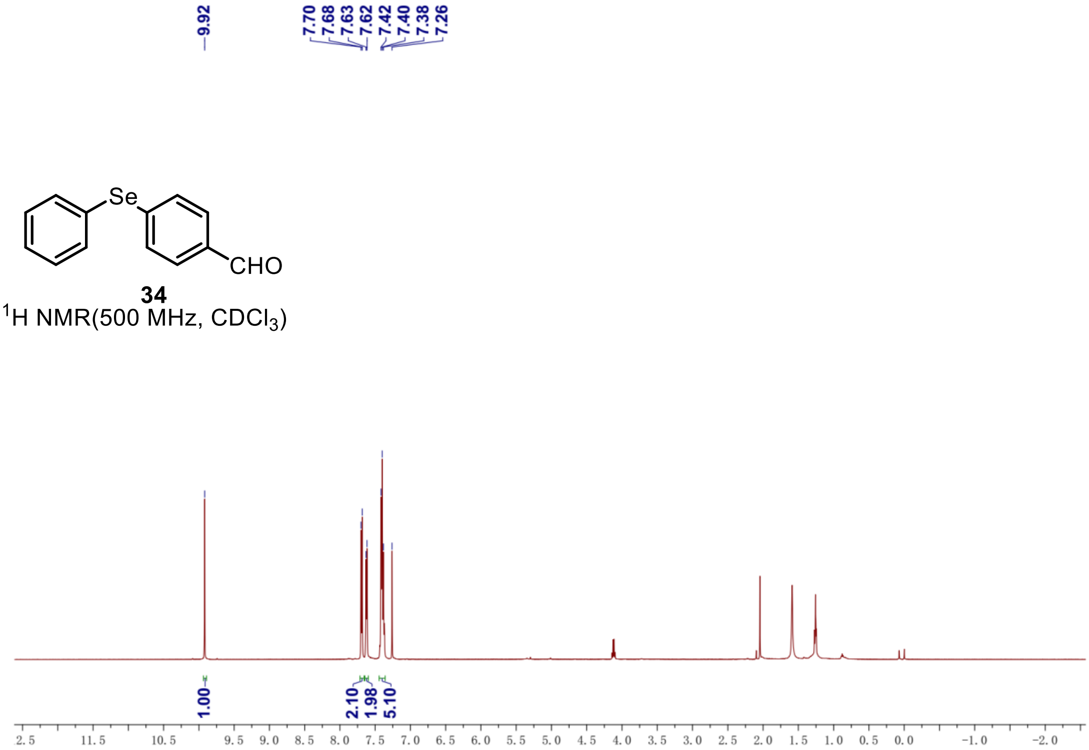
33

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



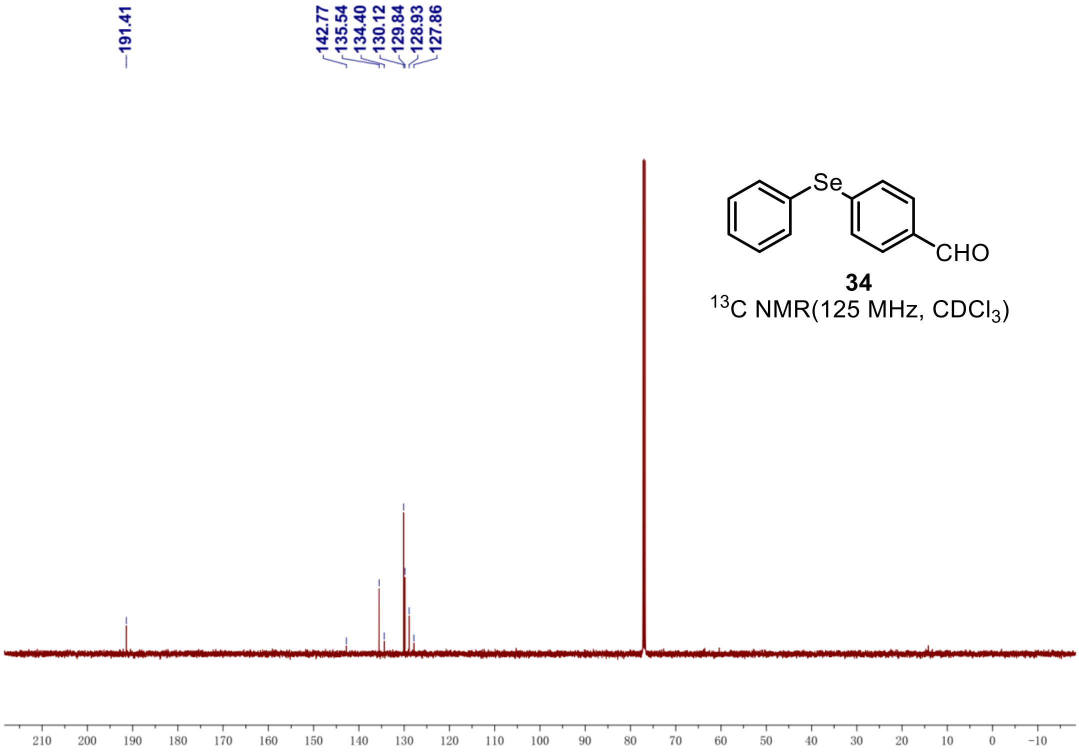
33

^{13}C NMR(125 MHz, $CDCl_3$)



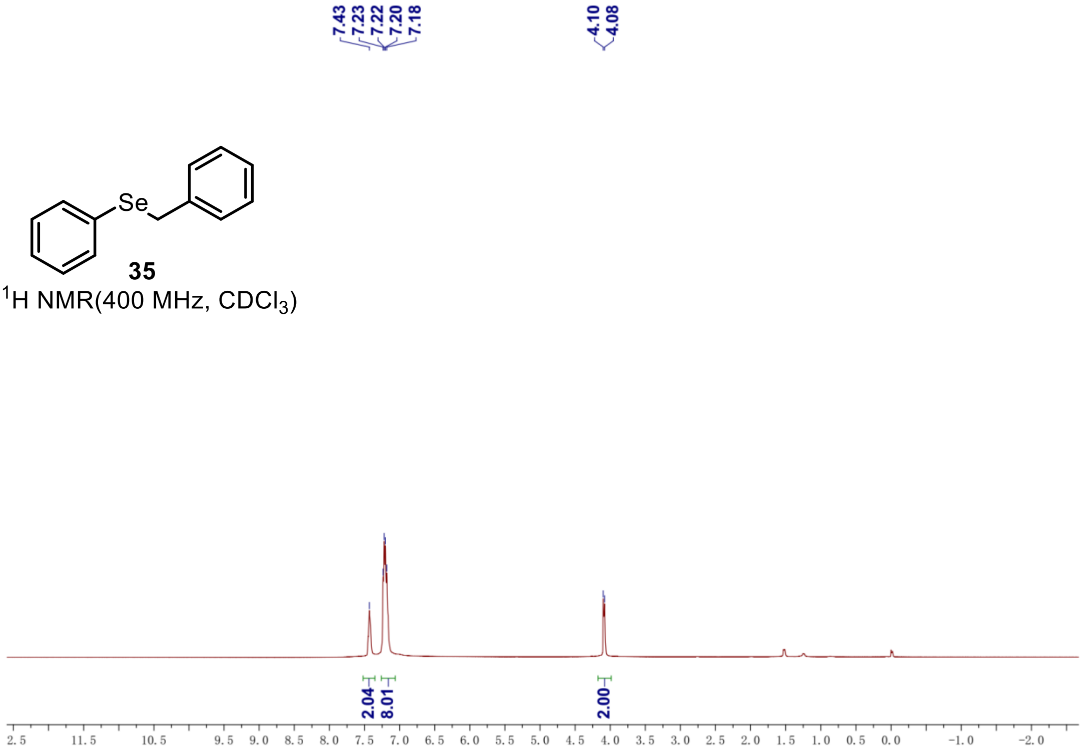
34

^{1}H NMR(500 MHz, CDCl$_3$)



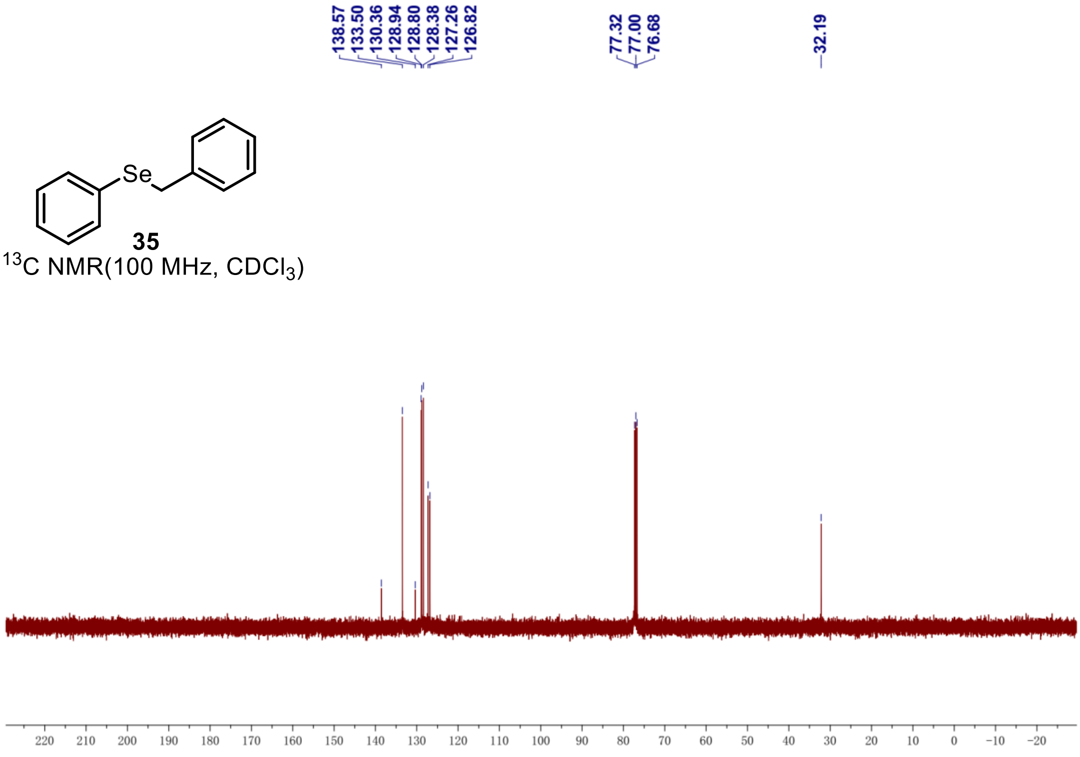
34

^{13}C NMR(125 MHz, CDCl$_3$)



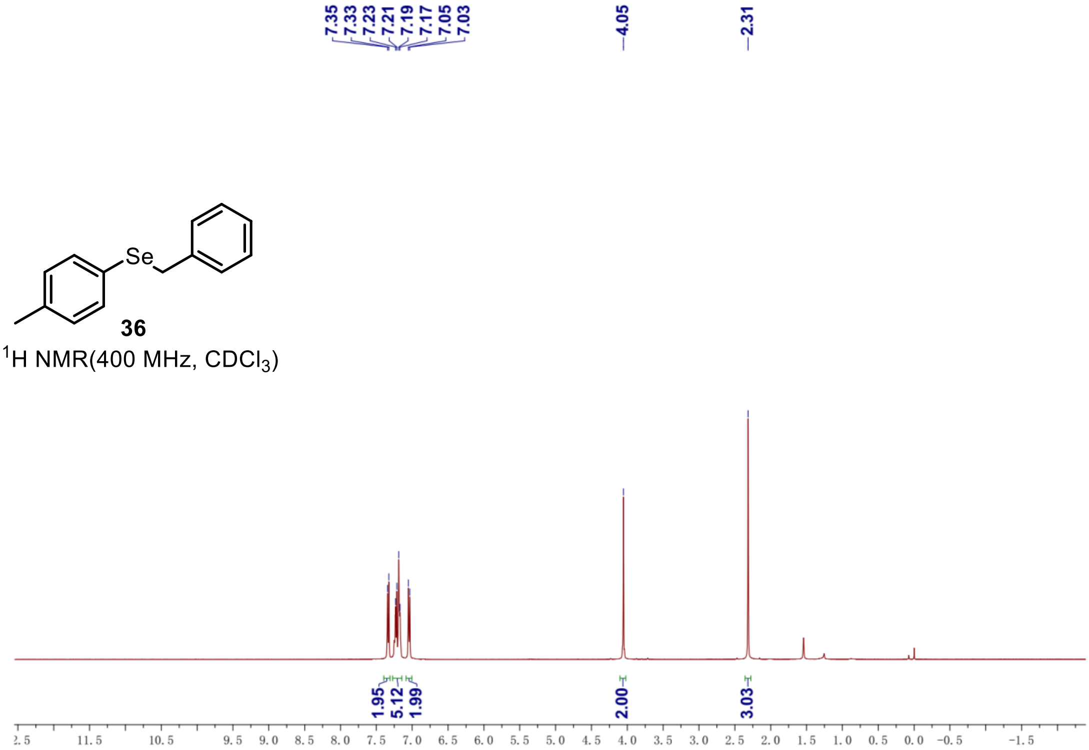
35

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



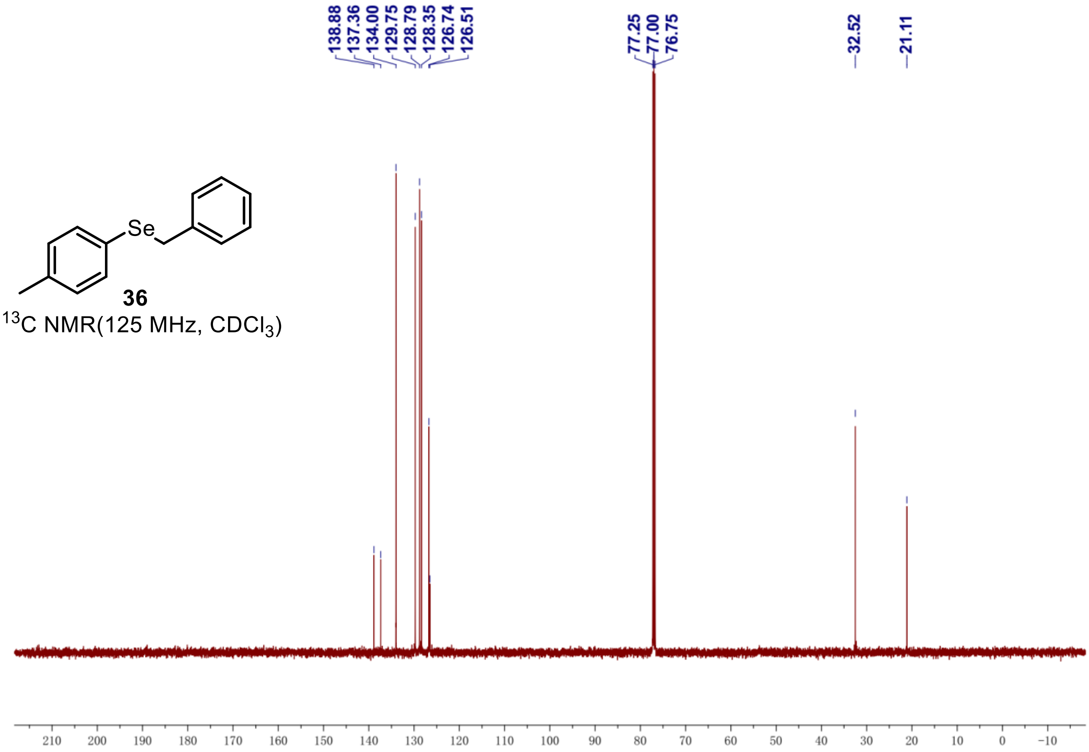
35

^{13}C NMR(100 MHz, $CDCl_3$)



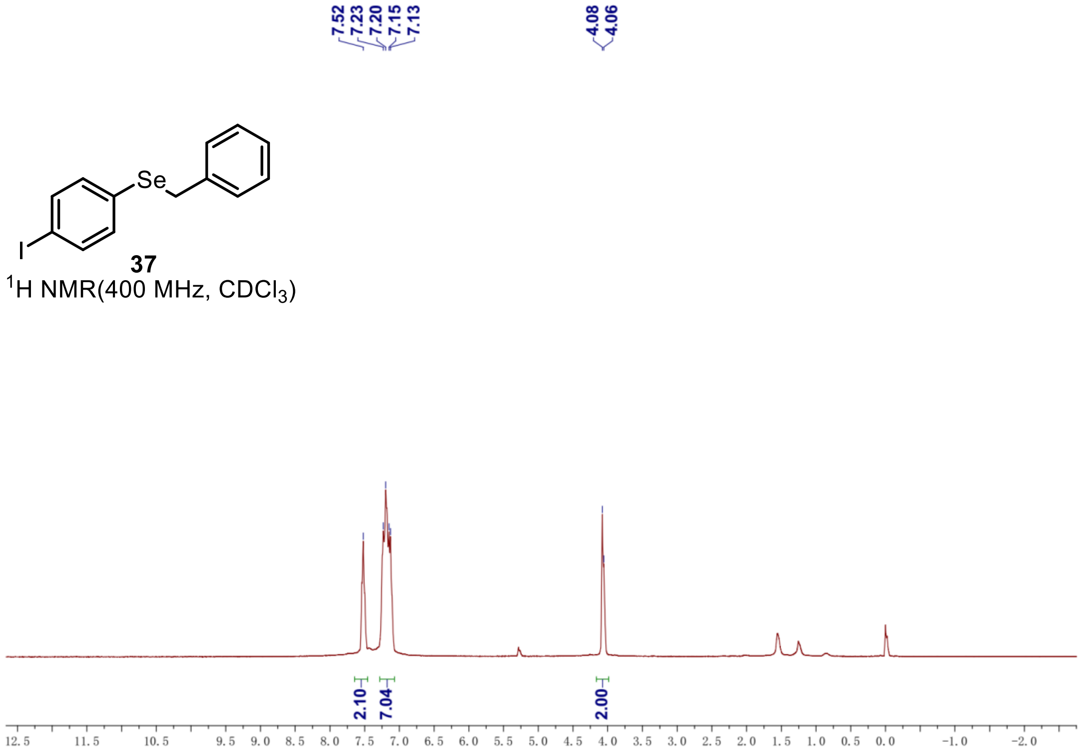
36

^{1}H NMR(400 MHz, CDCl$_3$)



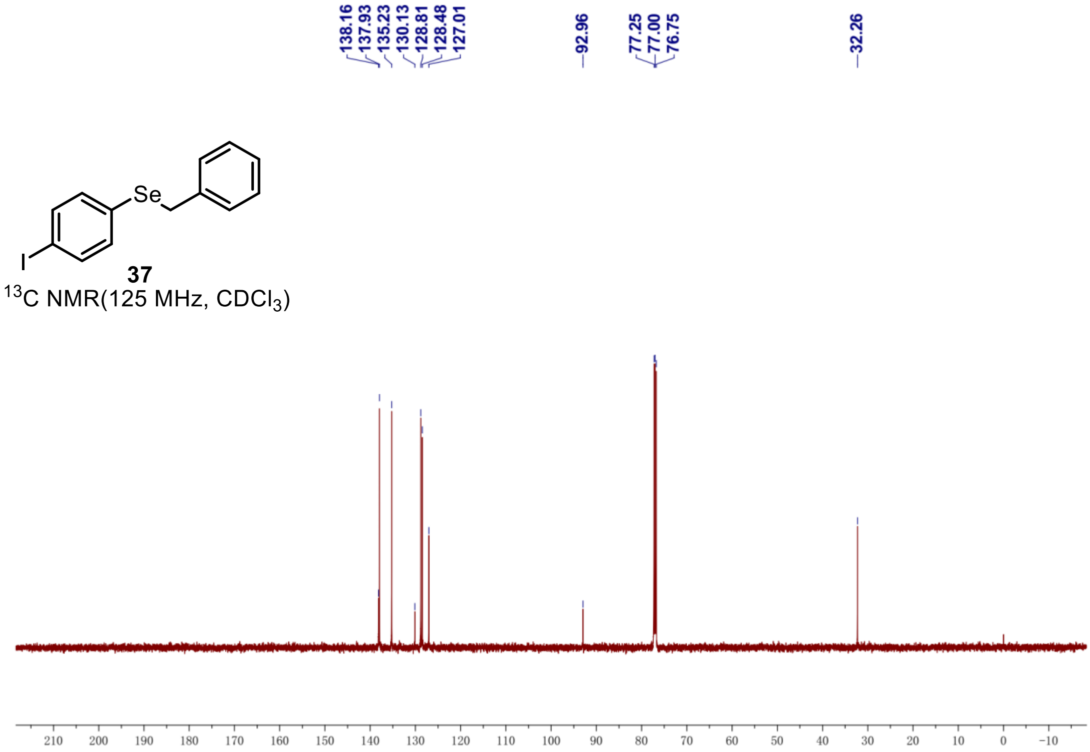
36

^{13}C NMR(125 MHz, CDCl$_3$)



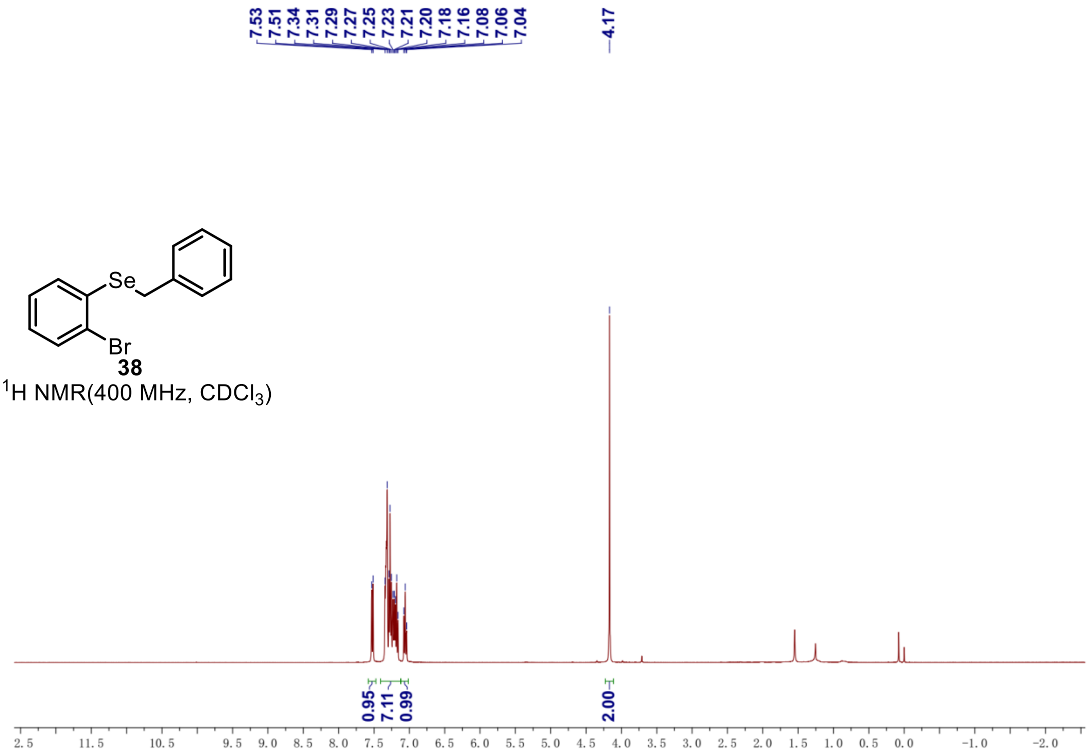
37

^{1}H NMR(400 MHz, CDCl$_3$)



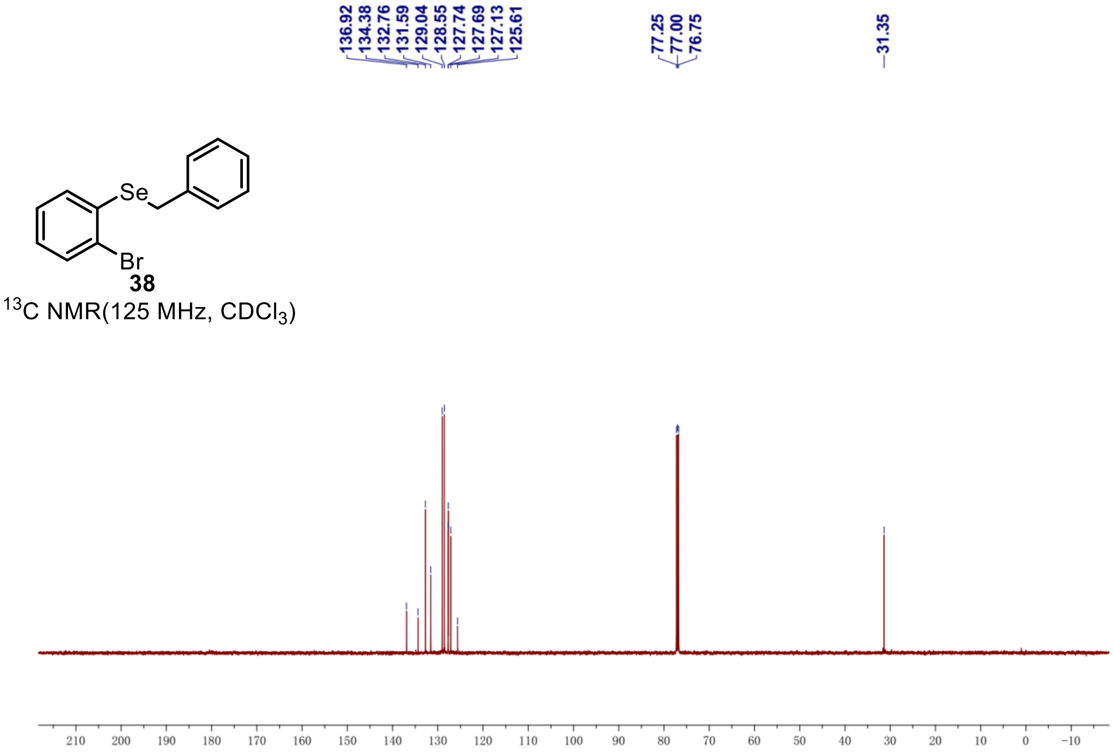
37

^{13}C NMR(125 MHz, CDCl$_3$)



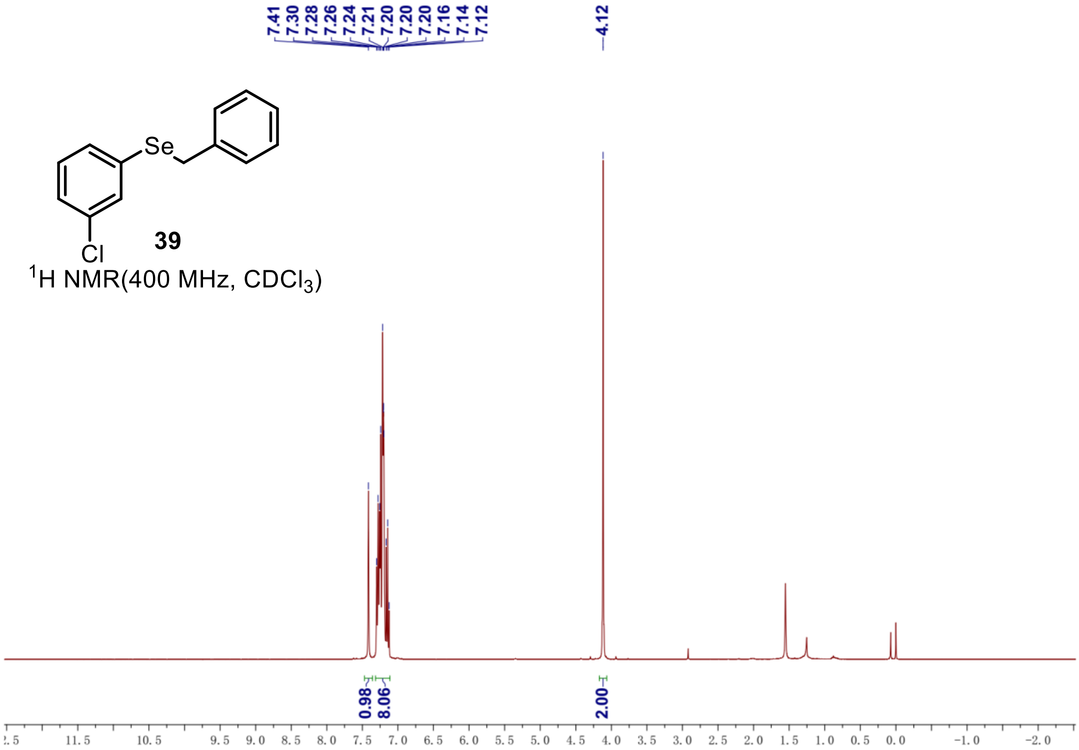
38

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



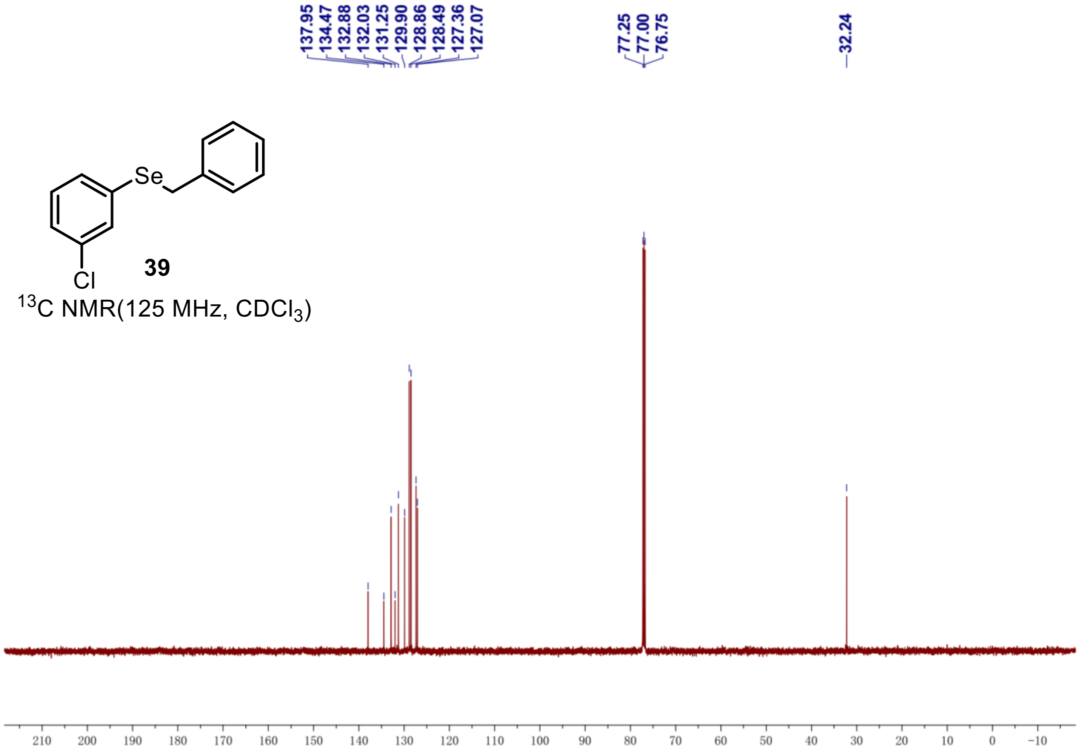
38

^{13}C NMR(125 MHz, $CDCl_3$)



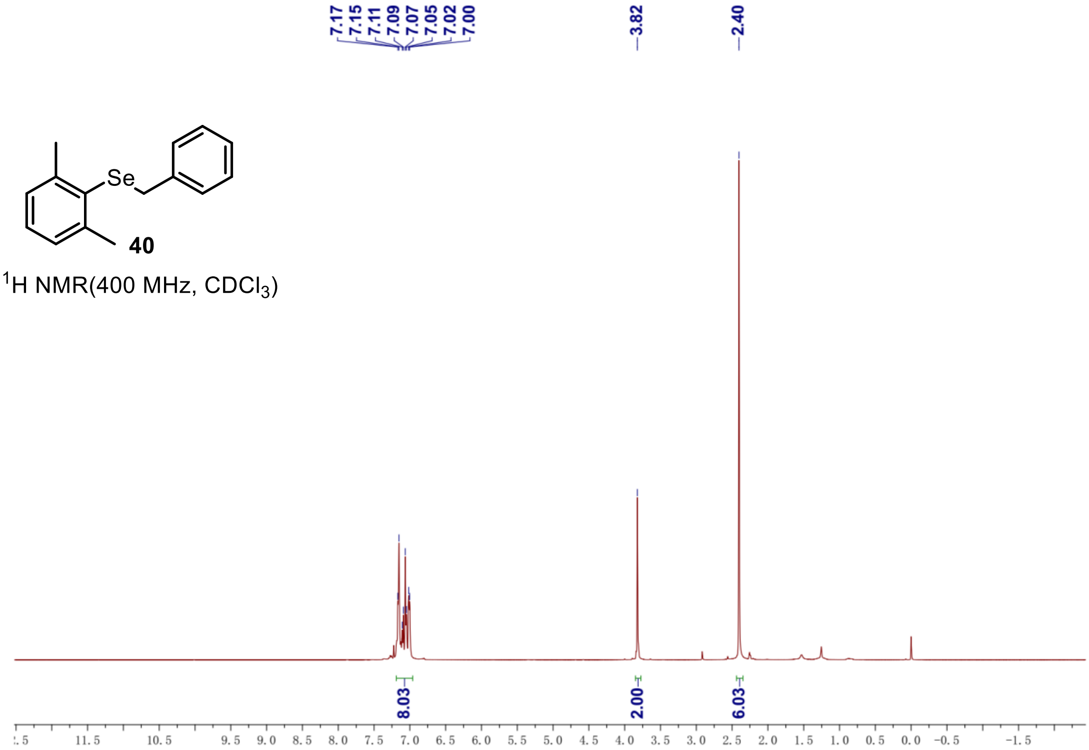
39

^{1}H NMR(400 MHz, CDCl$_3$)



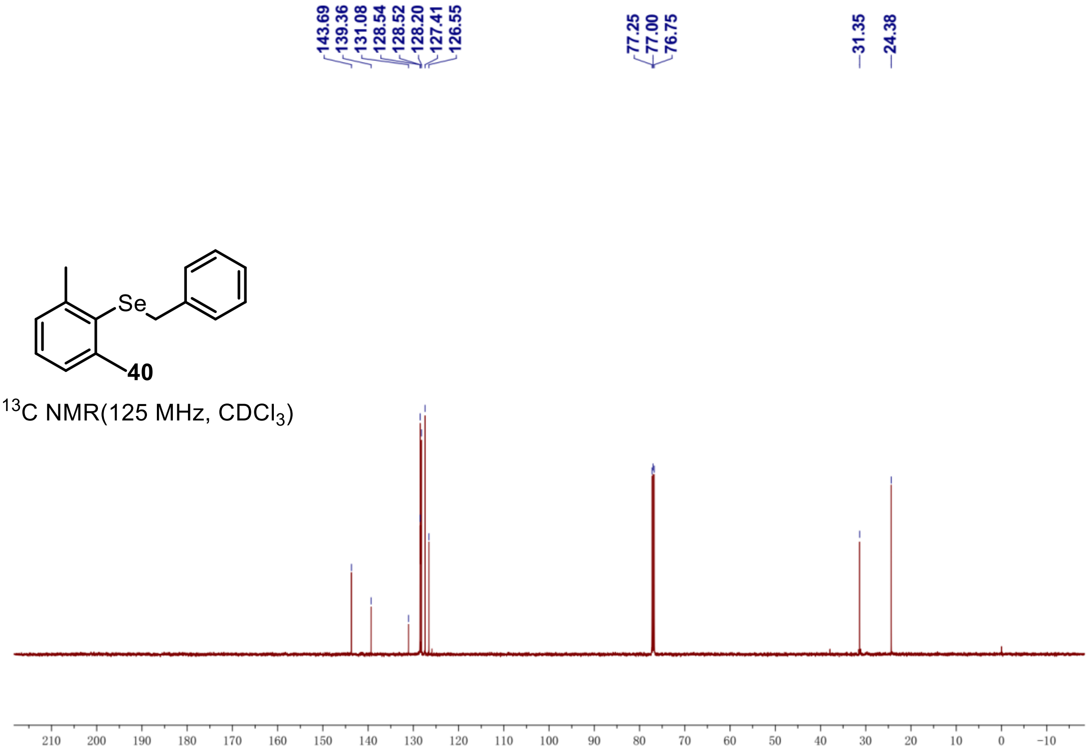
39

^{13}C NMR(125 MHz, CDCl$_3$)



40

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



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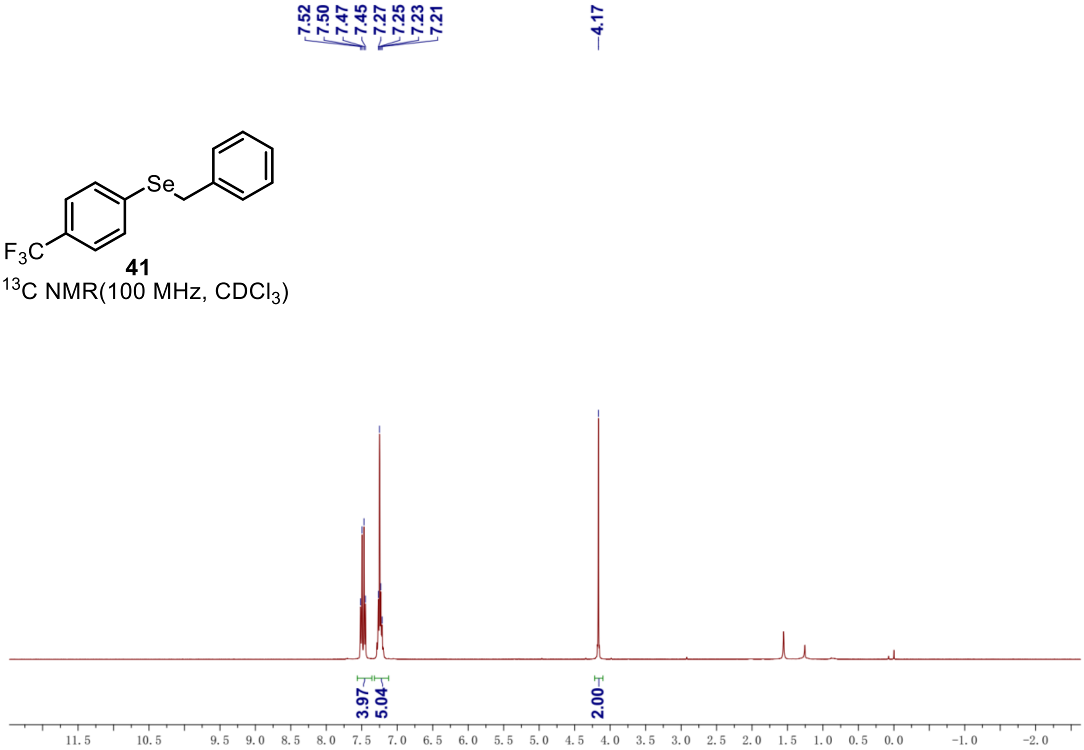
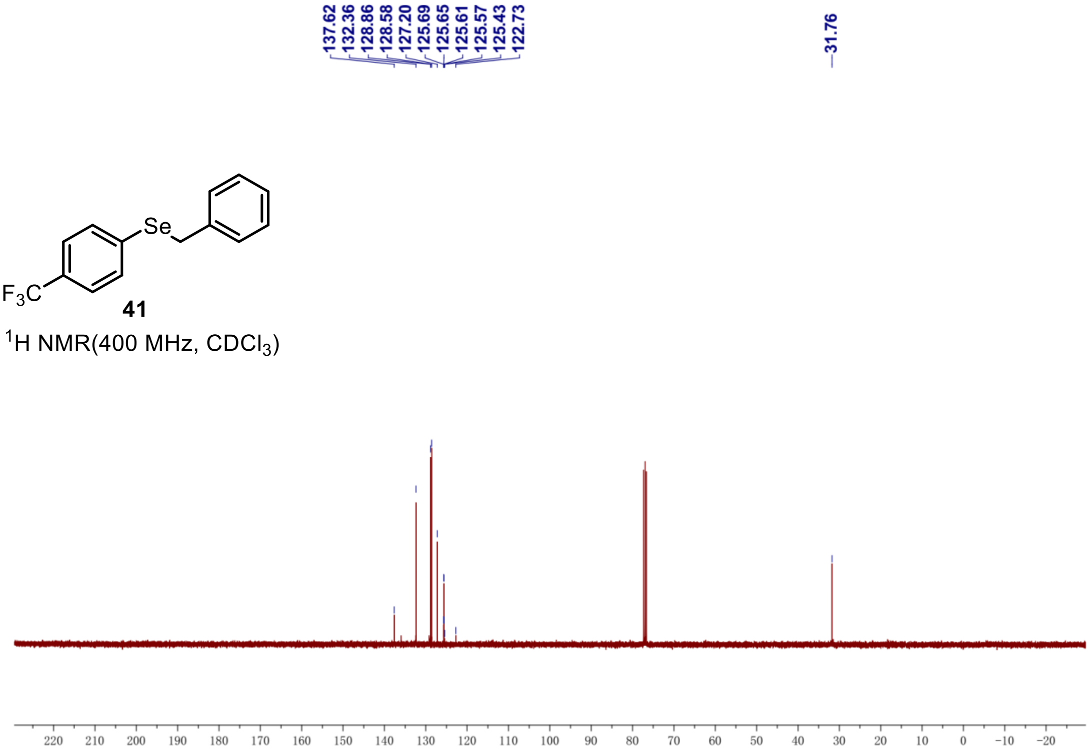


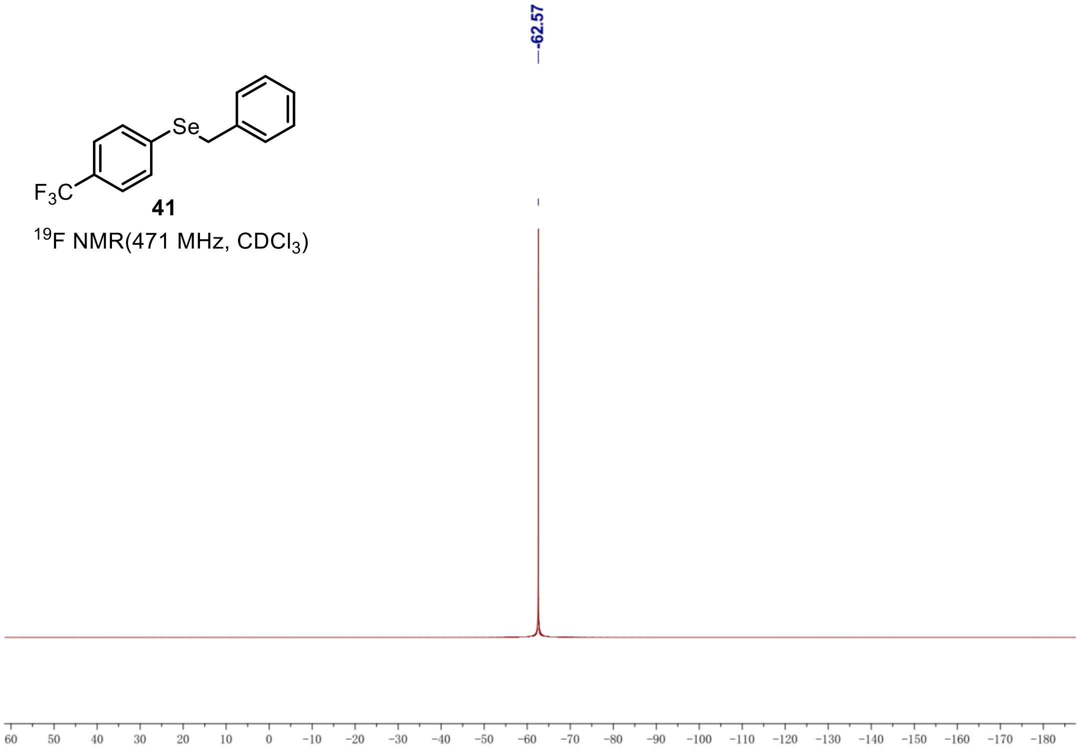
41

^{13}C NMR(100 MHz, $CDCl_3$)



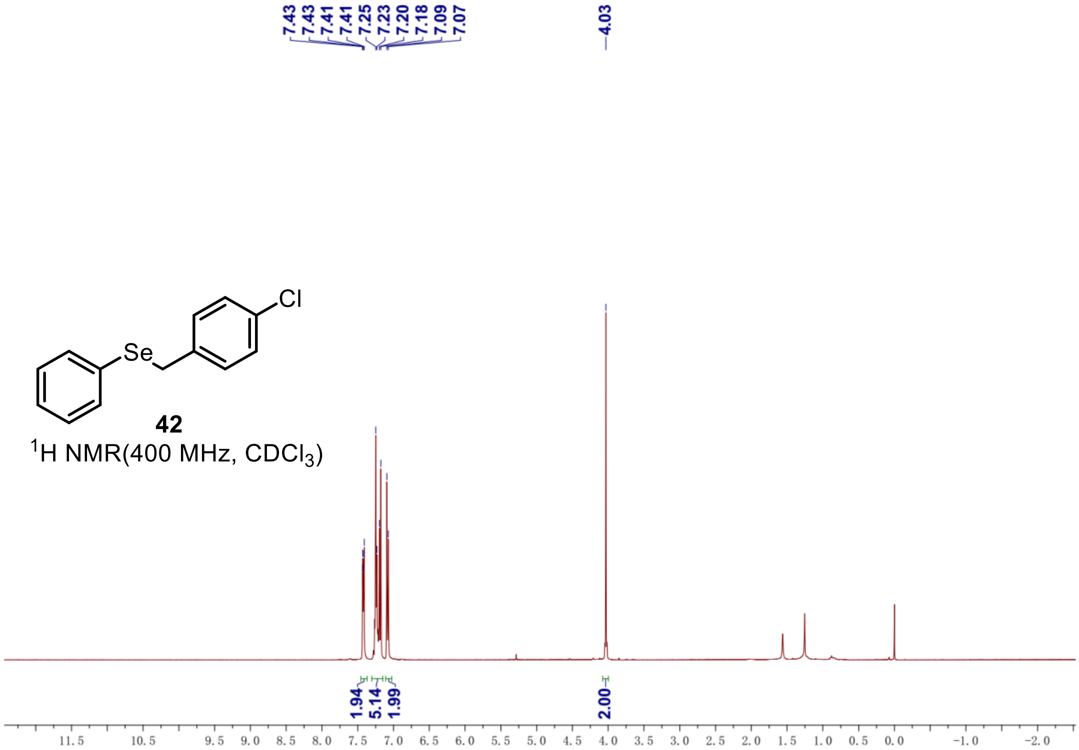
41

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



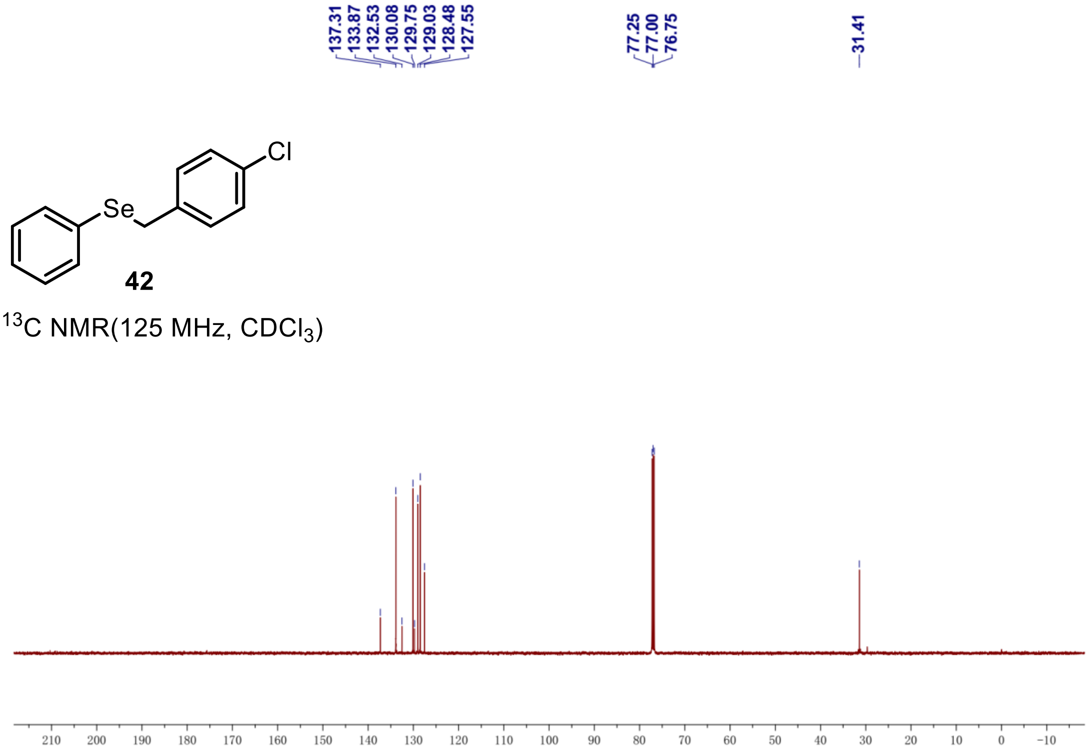
41

^{19}F NMR(471 MHz, CDCl$_3$)

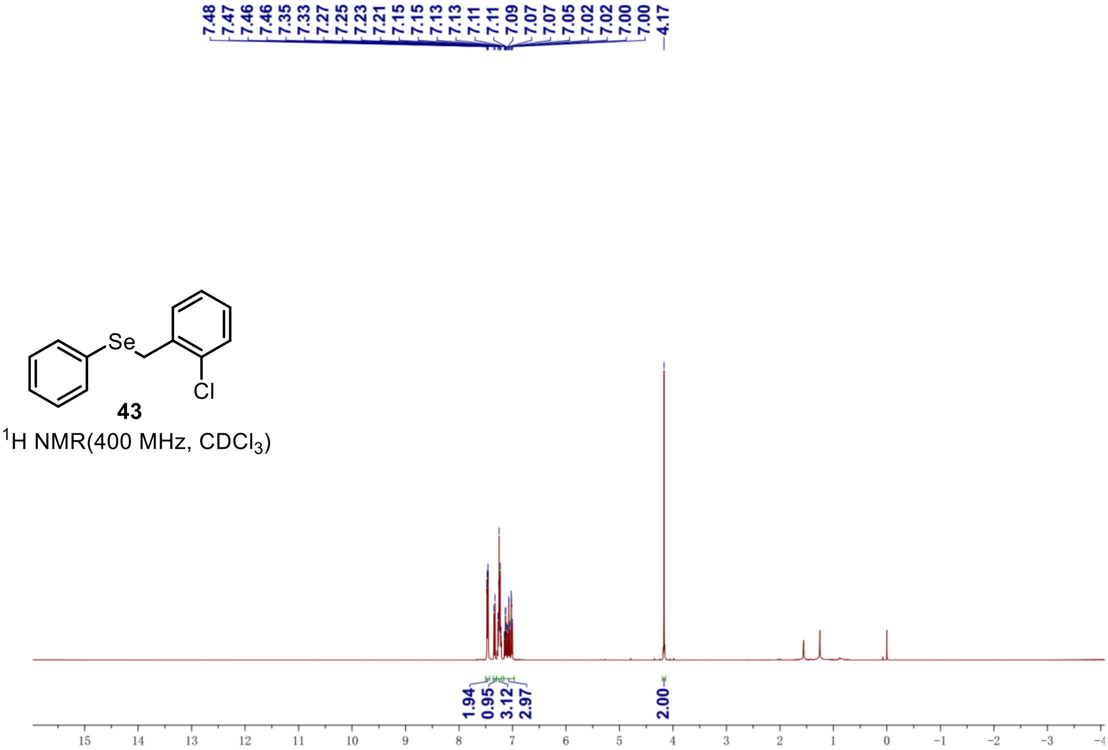


42

^{1}H NMR(400 MHz, CDCl$_3$)


42

^{13}C NMR(125 MHz, CDCl$_3$)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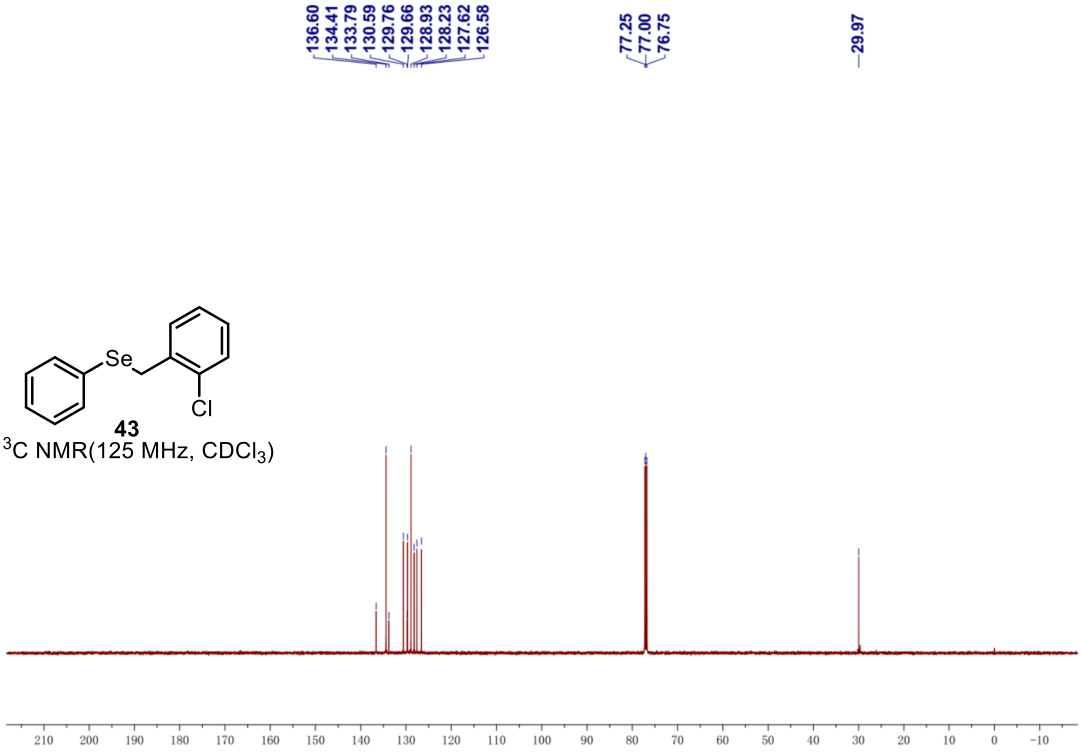
^{1}H NMR(400 MHz, CDCl$_3$)



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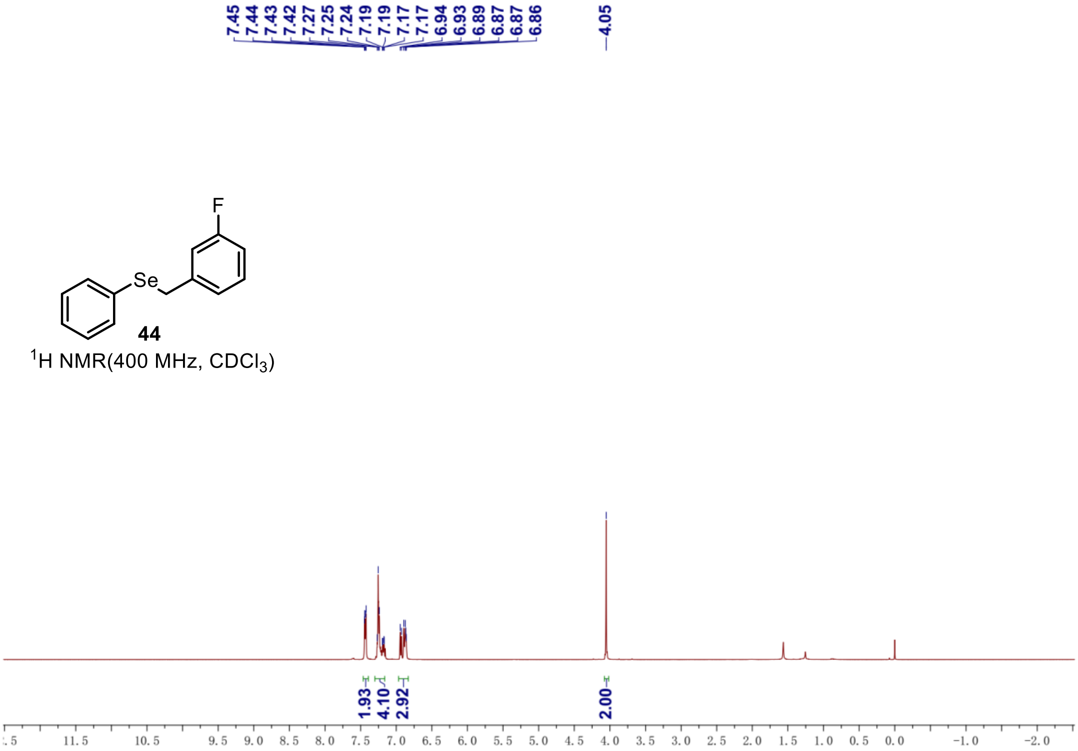
^{13}C NMR(125 MHz, CDCl$_3$)



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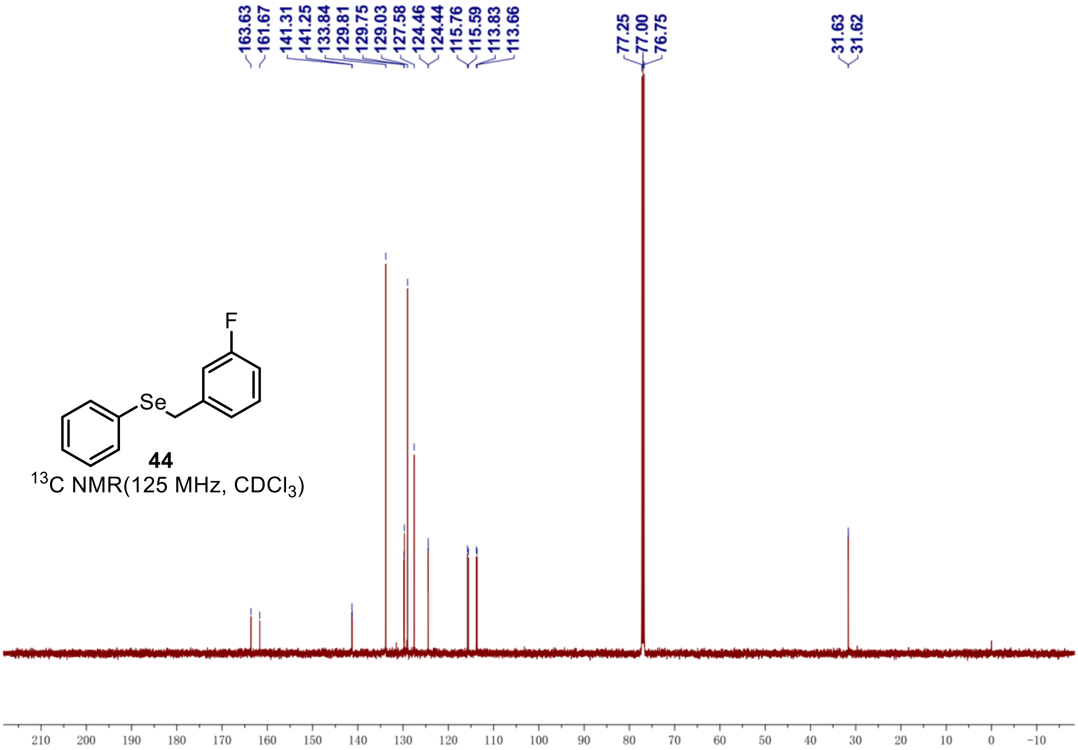

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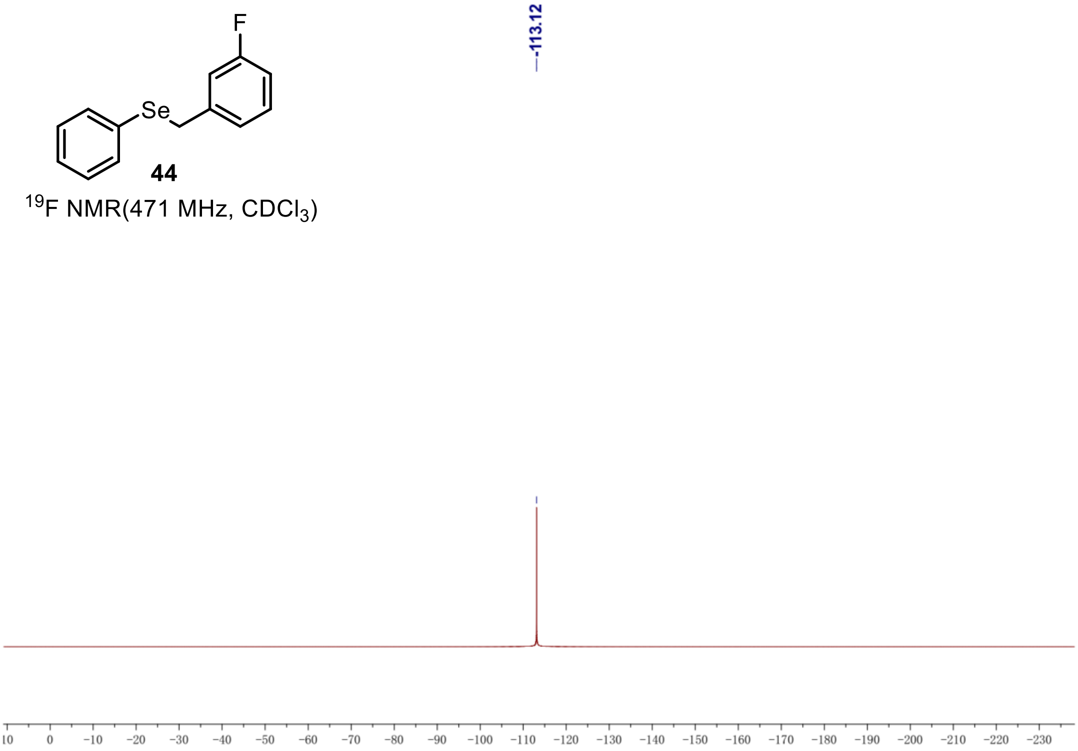
^{13}C NMR(125 MHz, $CDCl_3$)



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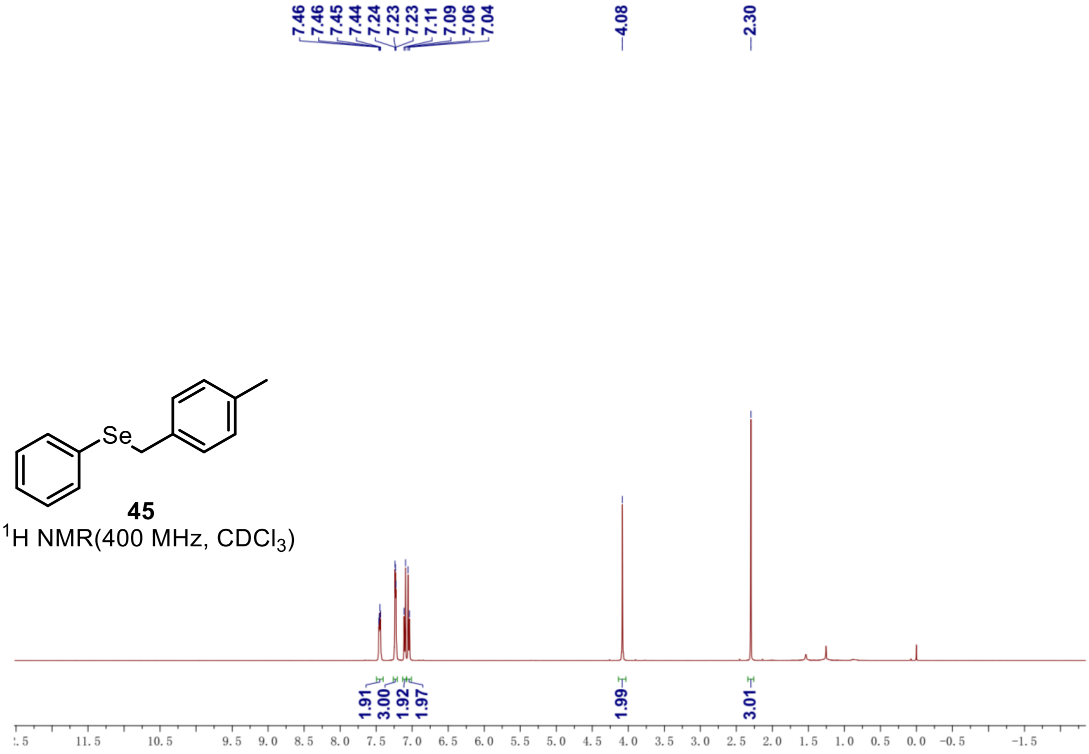
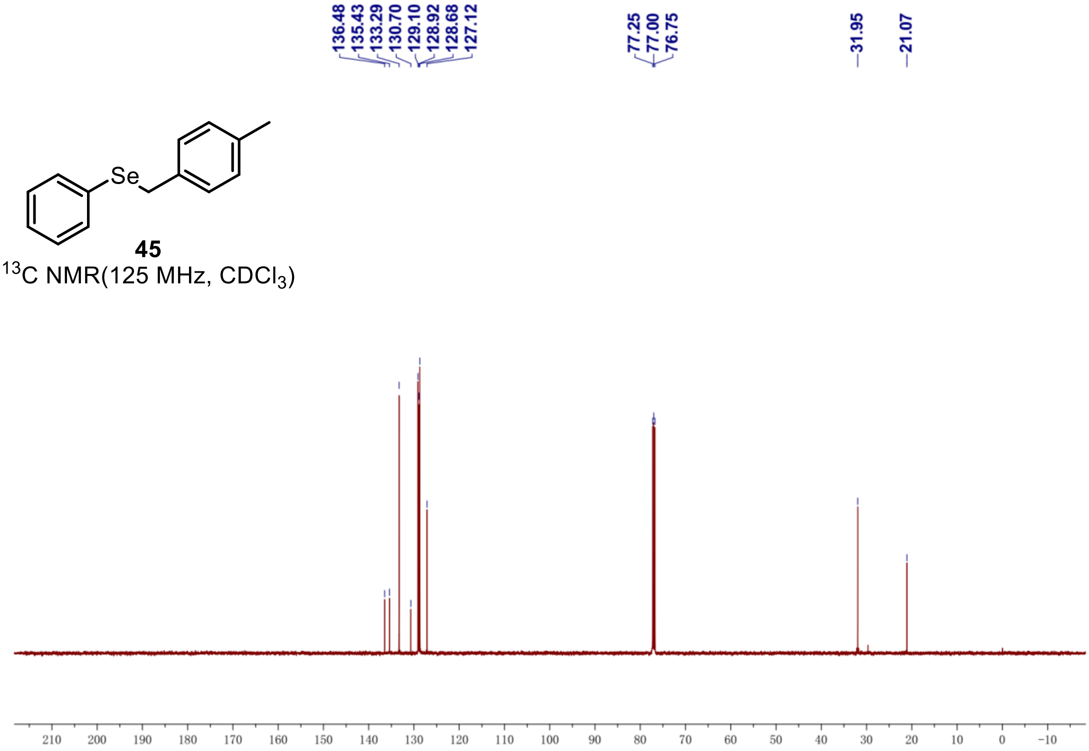


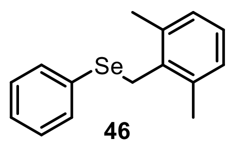
45

^{1}H NMR(400 MHz, CDCl$_3$)



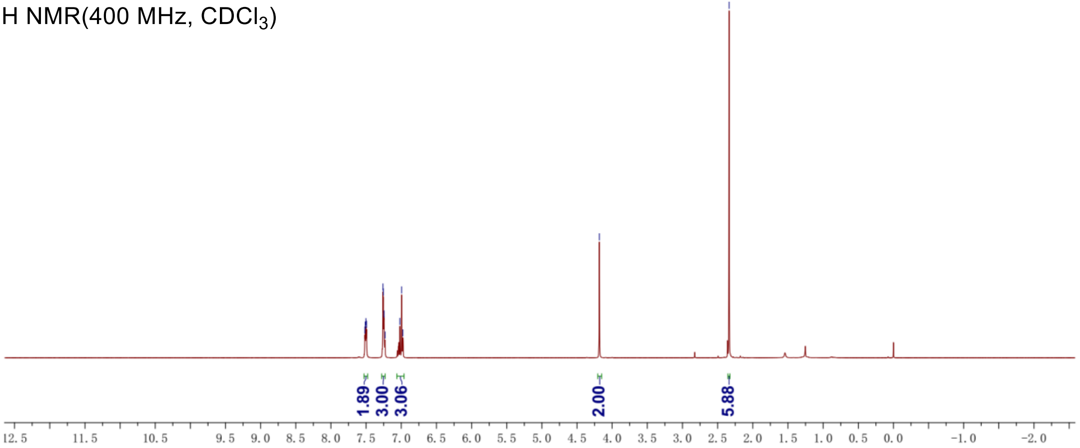
45

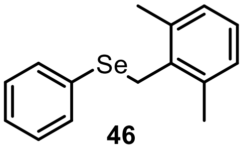
^{13}C NMR(125 MHz, CDCl$_3$)



46

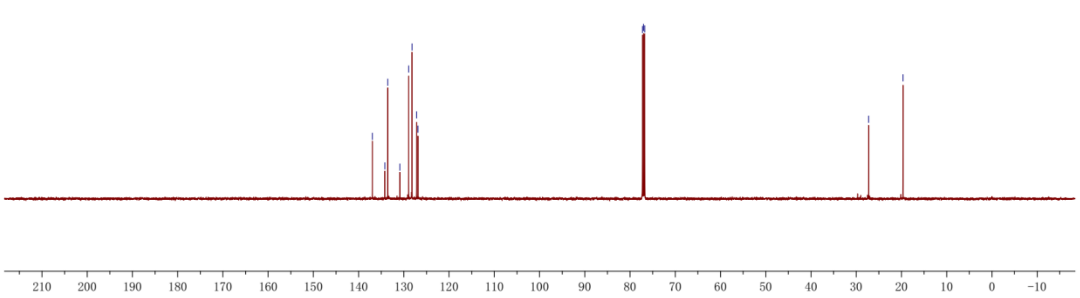
^{1}H NMR(400 MHz, $CDCl_3$)

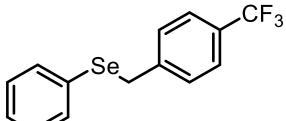




46

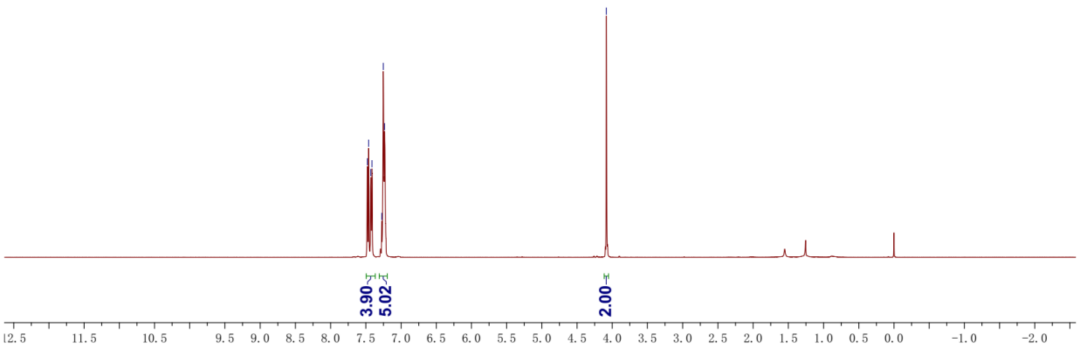
^{13}C NMR(125 MHz, $CDCl_3$)

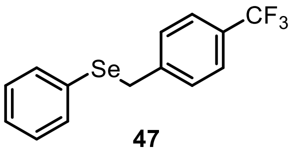




47

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





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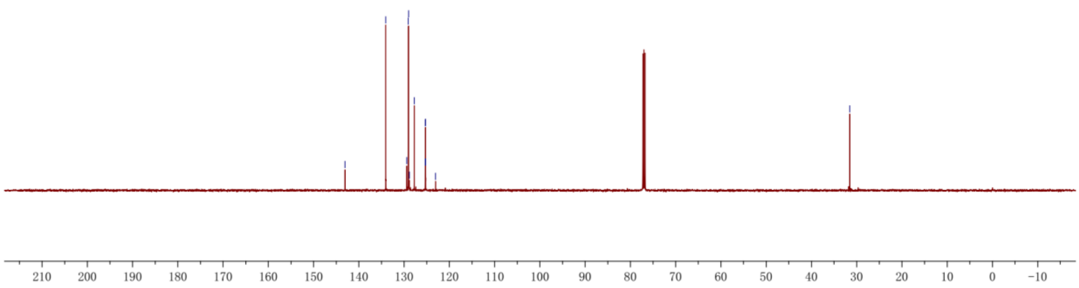
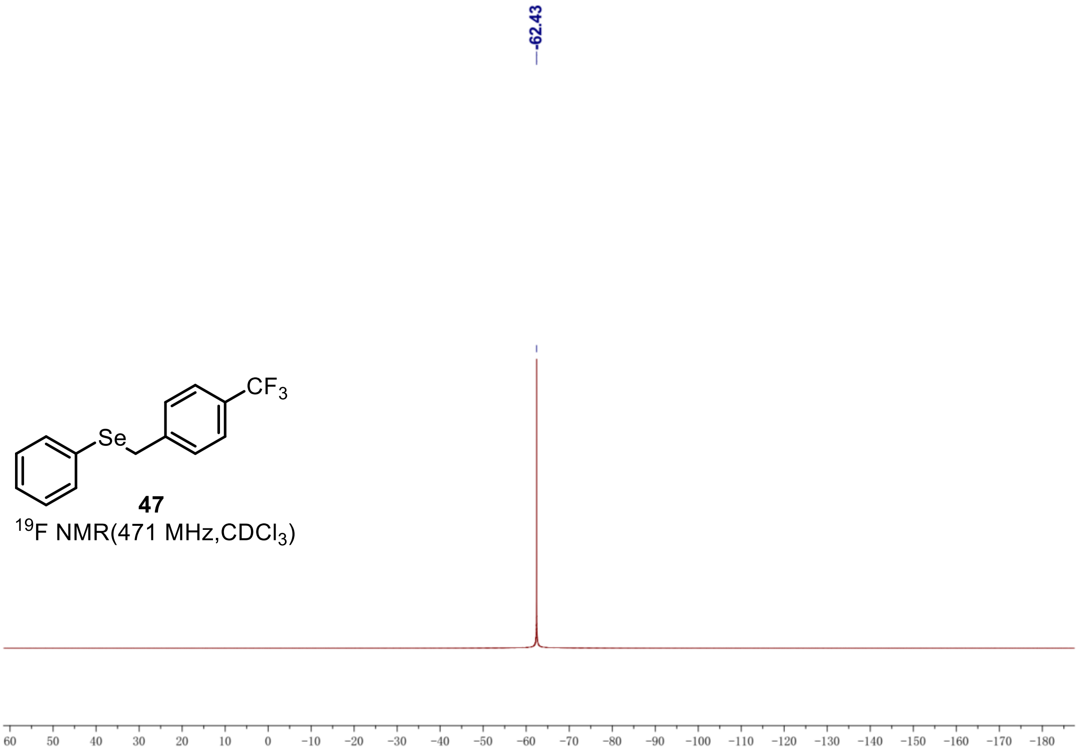
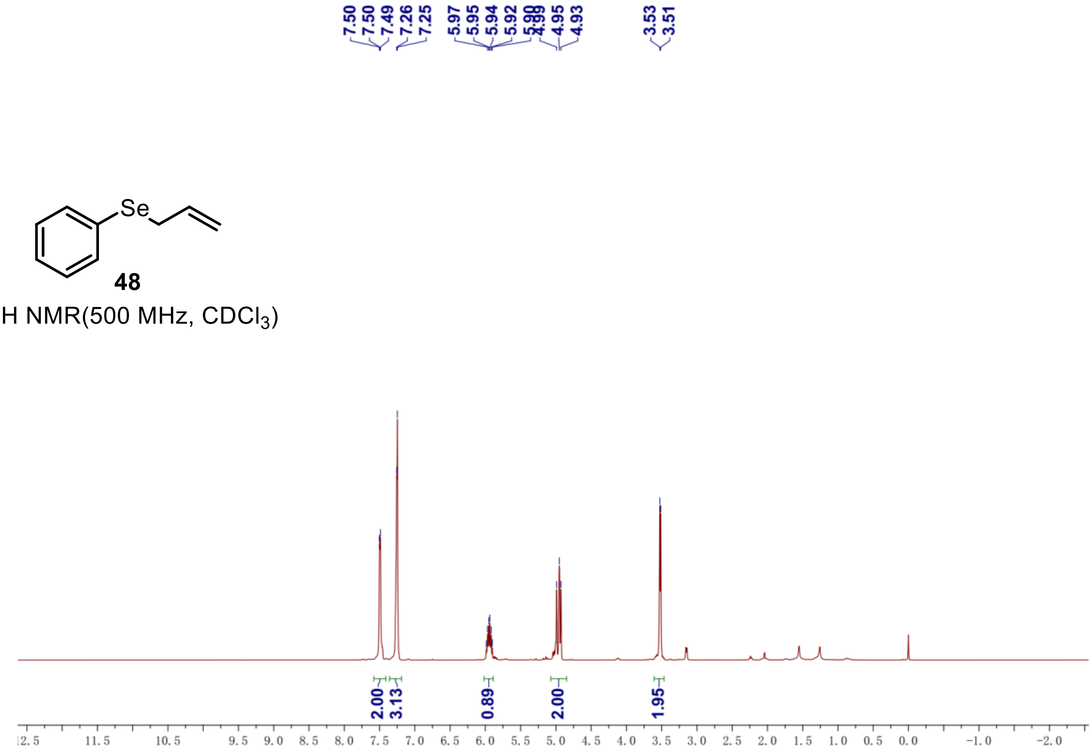




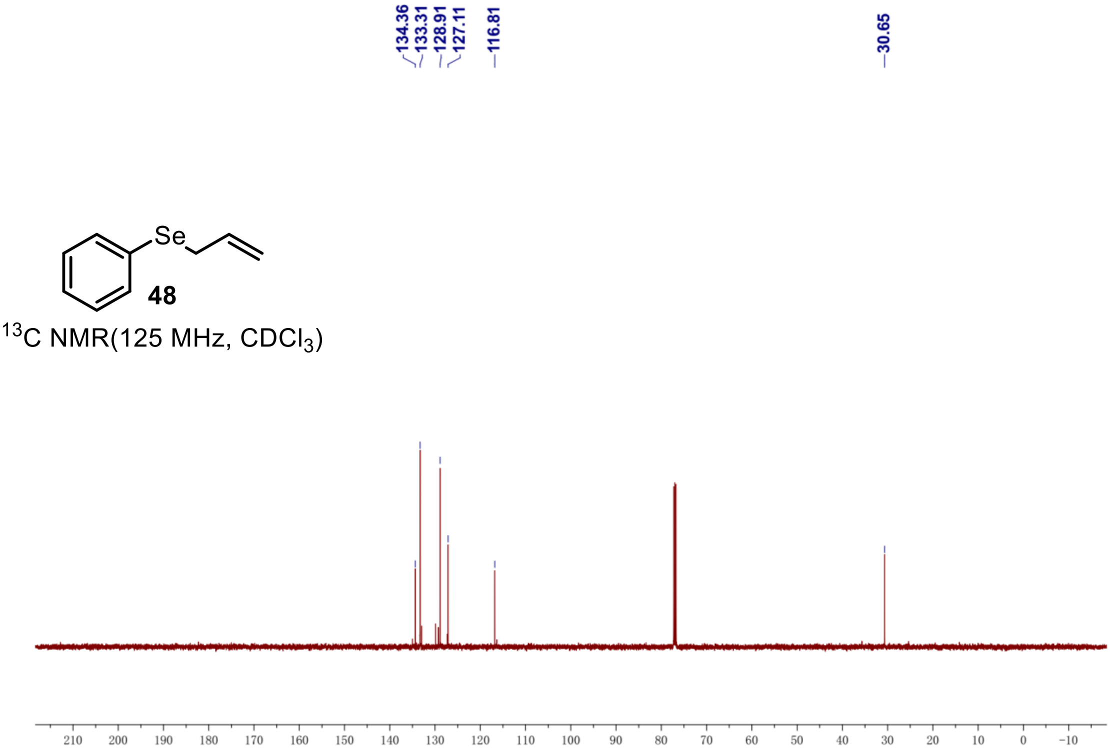
47

^{19}F NMR(471 MHz,CDCl$_3$)



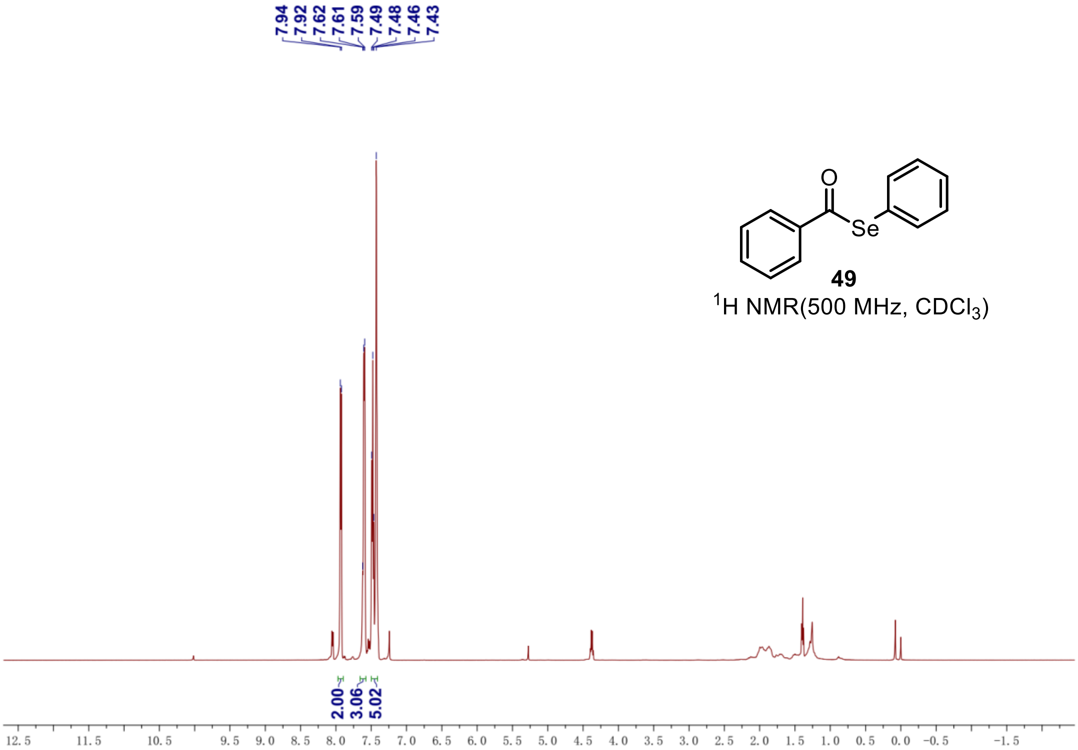
48

^{1}H NMR(500 MHz, CDCl$_3$)



48

^{13}C NMR(125 MHz, CDCl$_3$)



49

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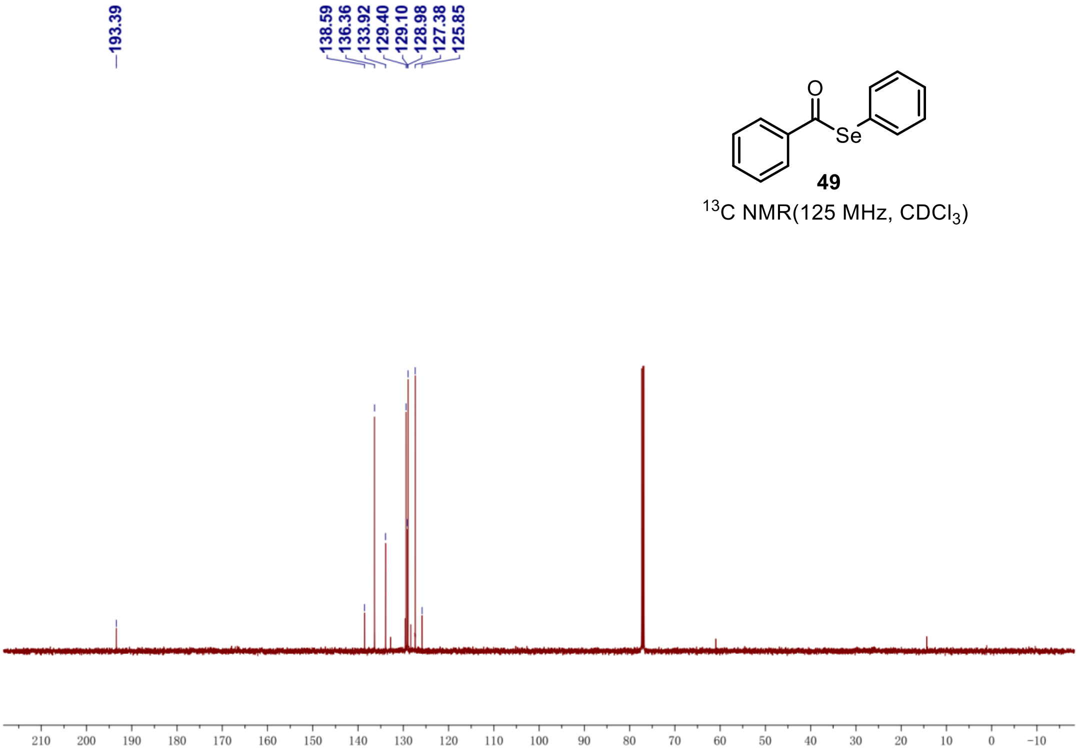
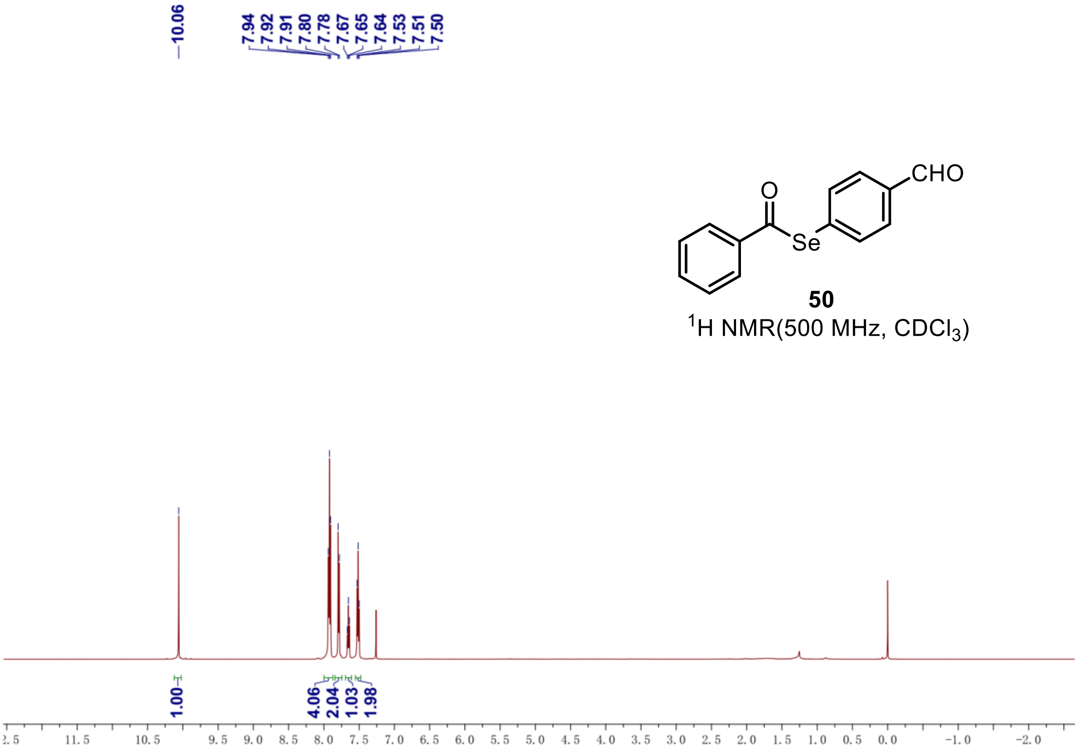


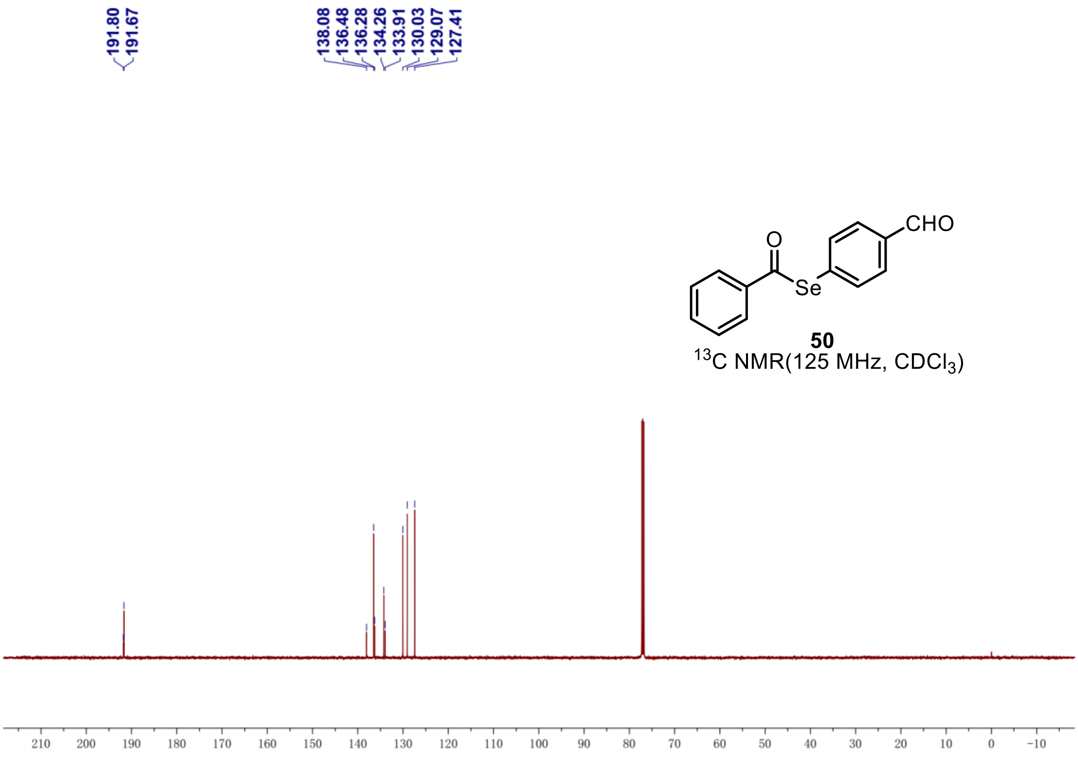
49

^{13}C NMR(125 MHz, CDCl$_3$)



50

^{1}H NMR(500 MHz, CDCl$_3$)



50

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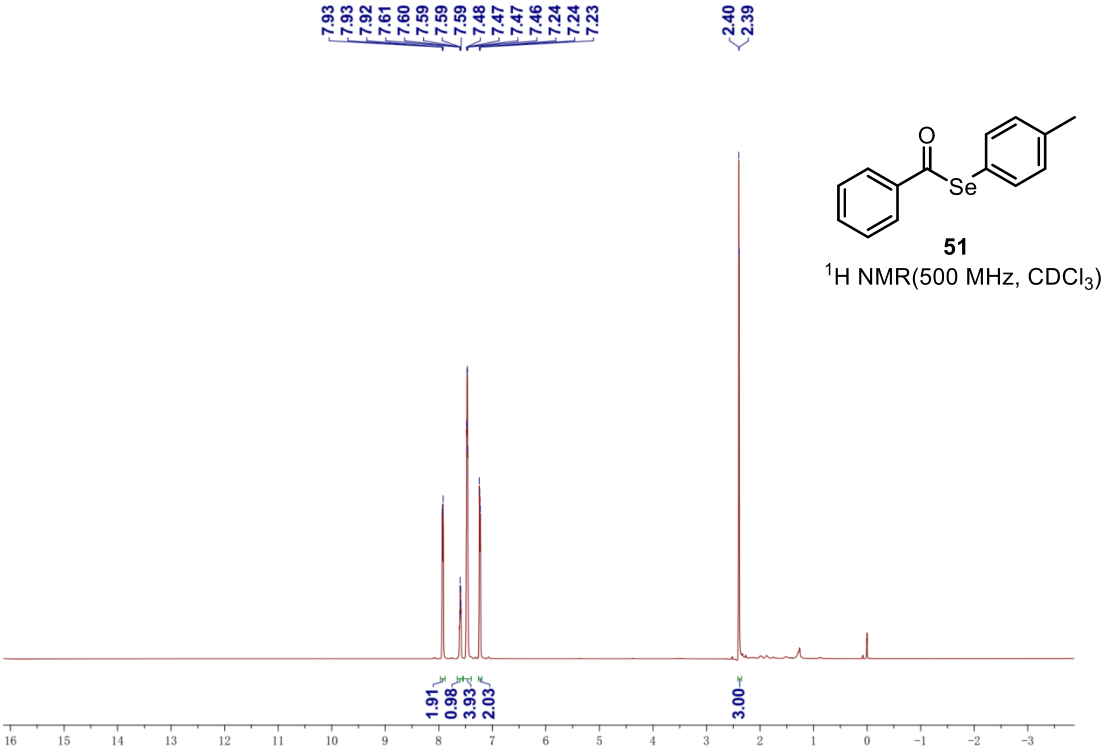

51

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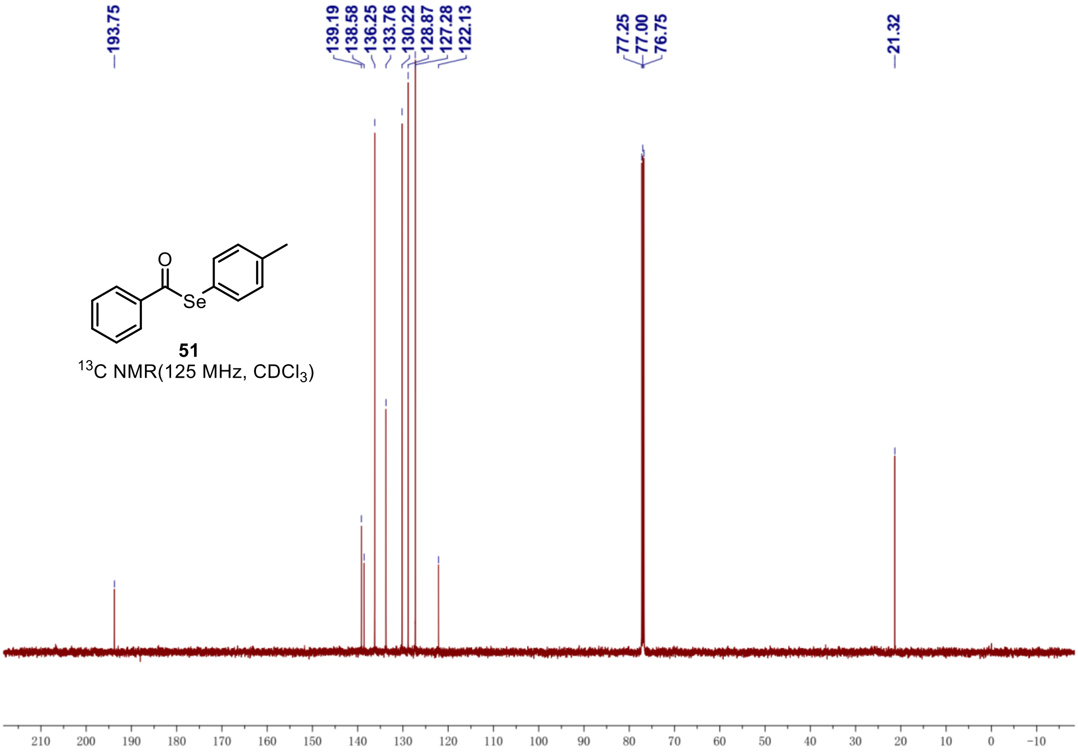

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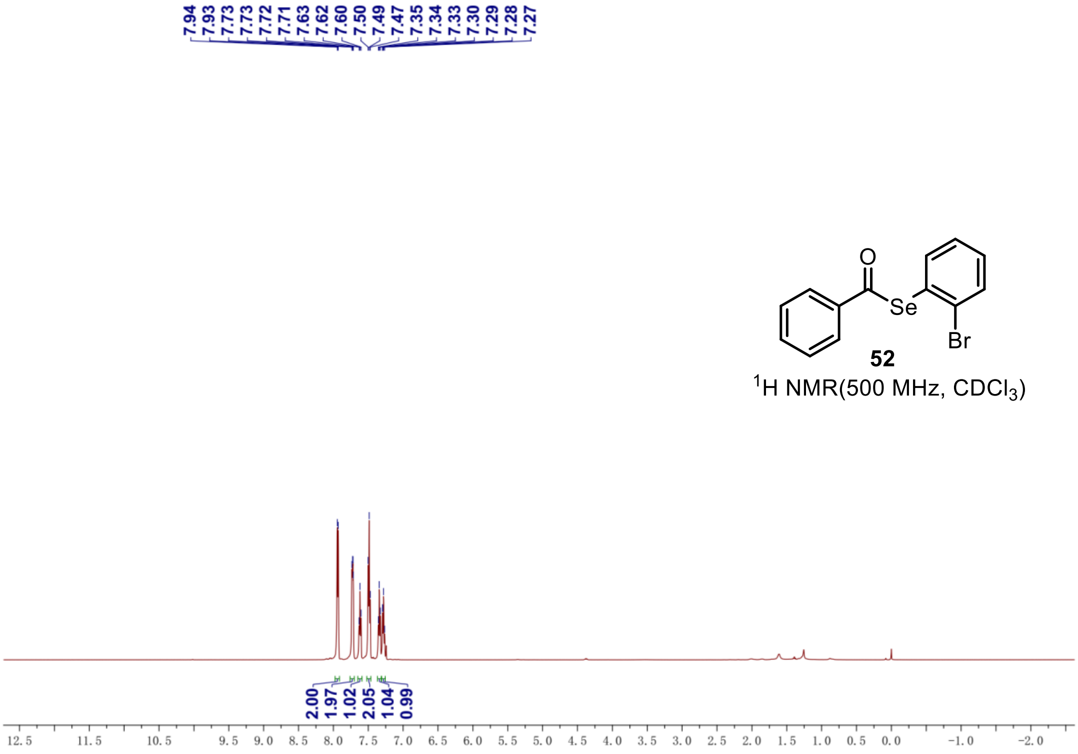

52

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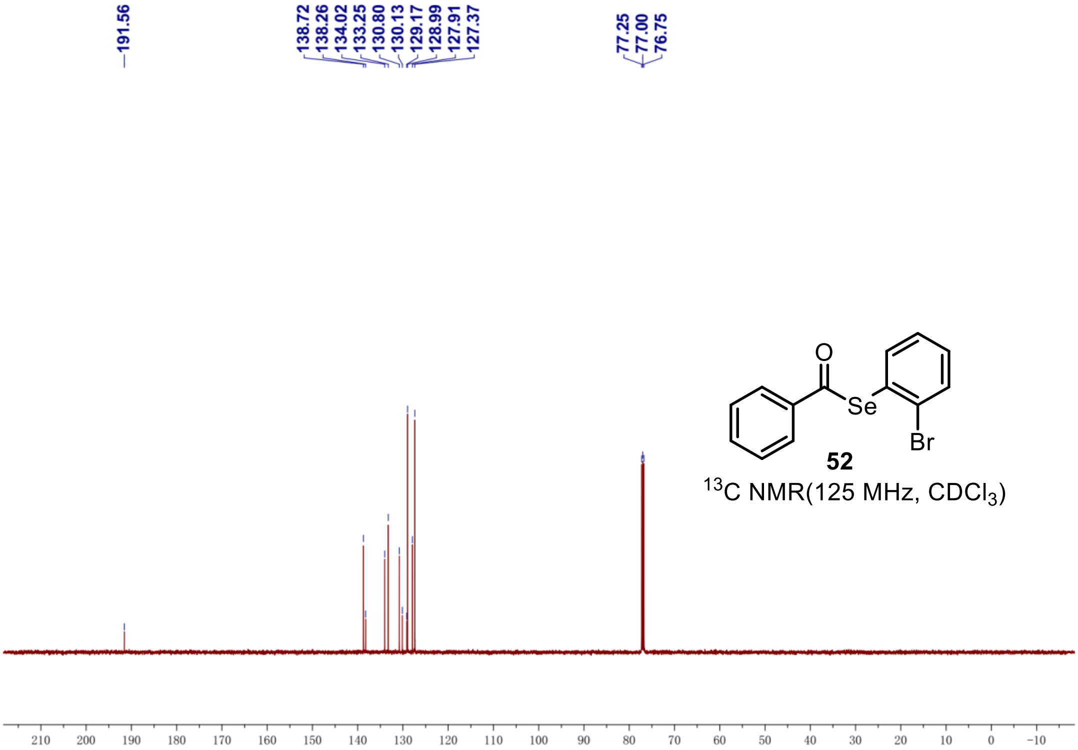

52

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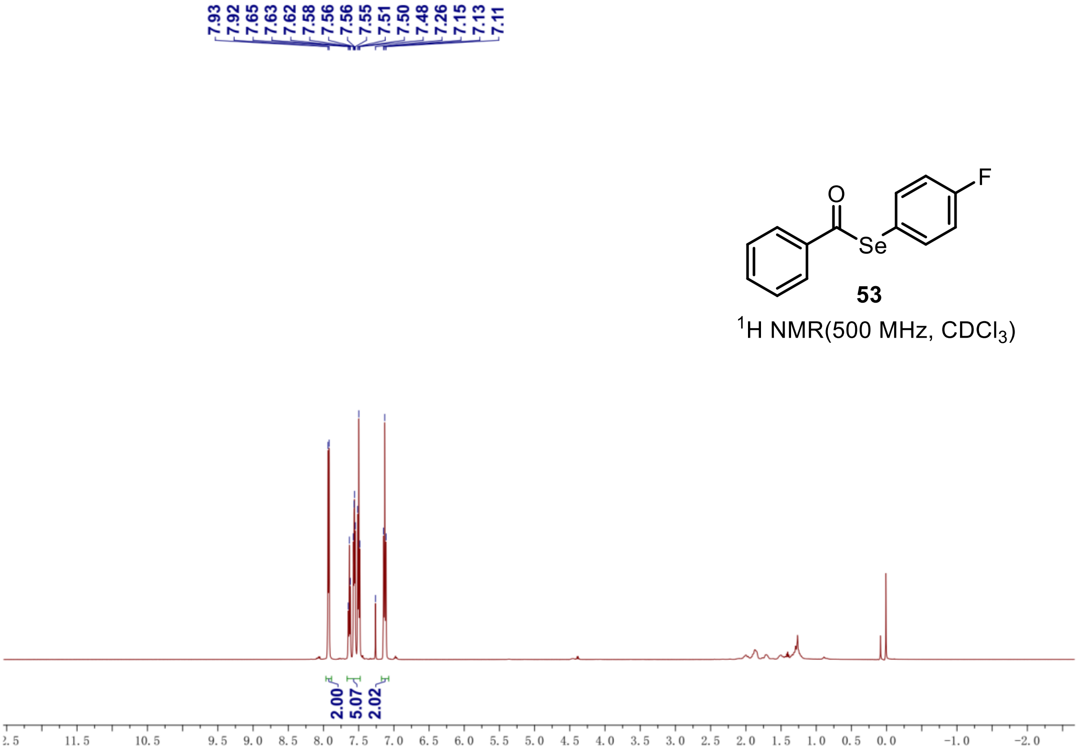

53

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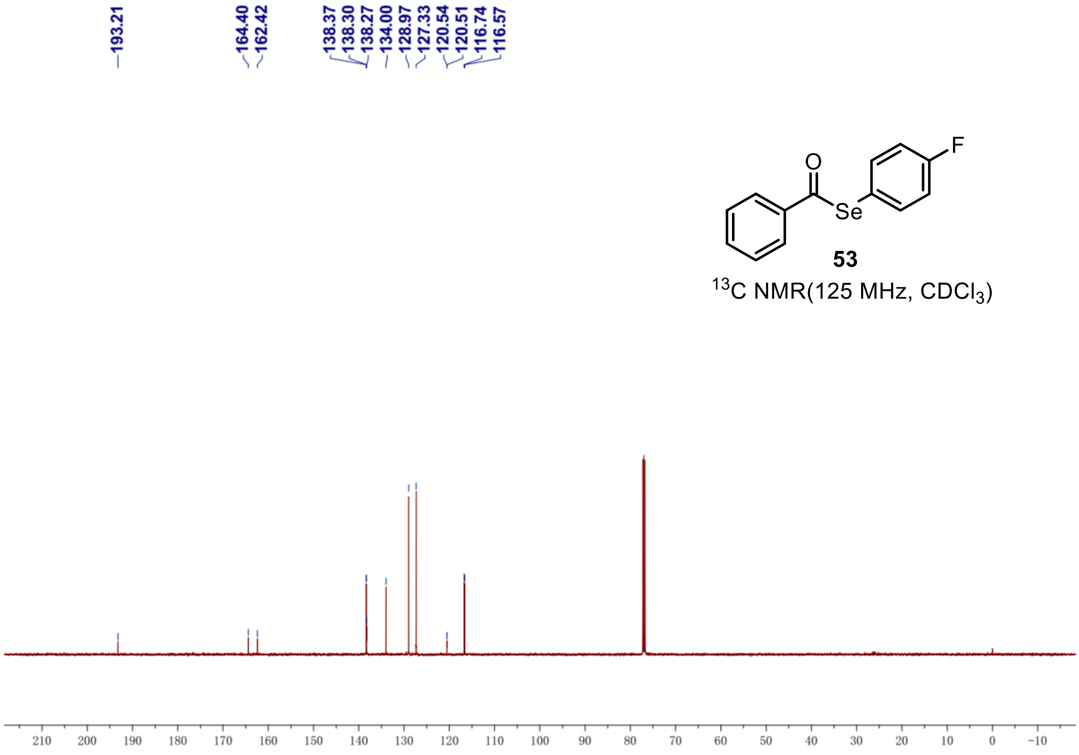
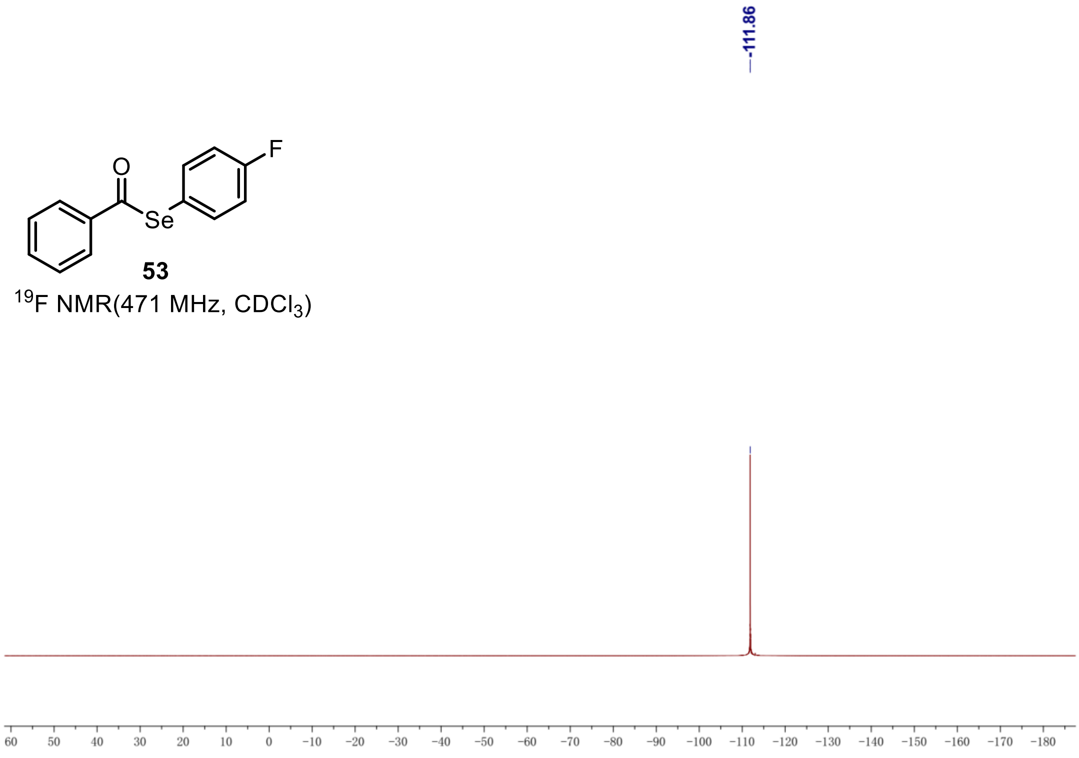


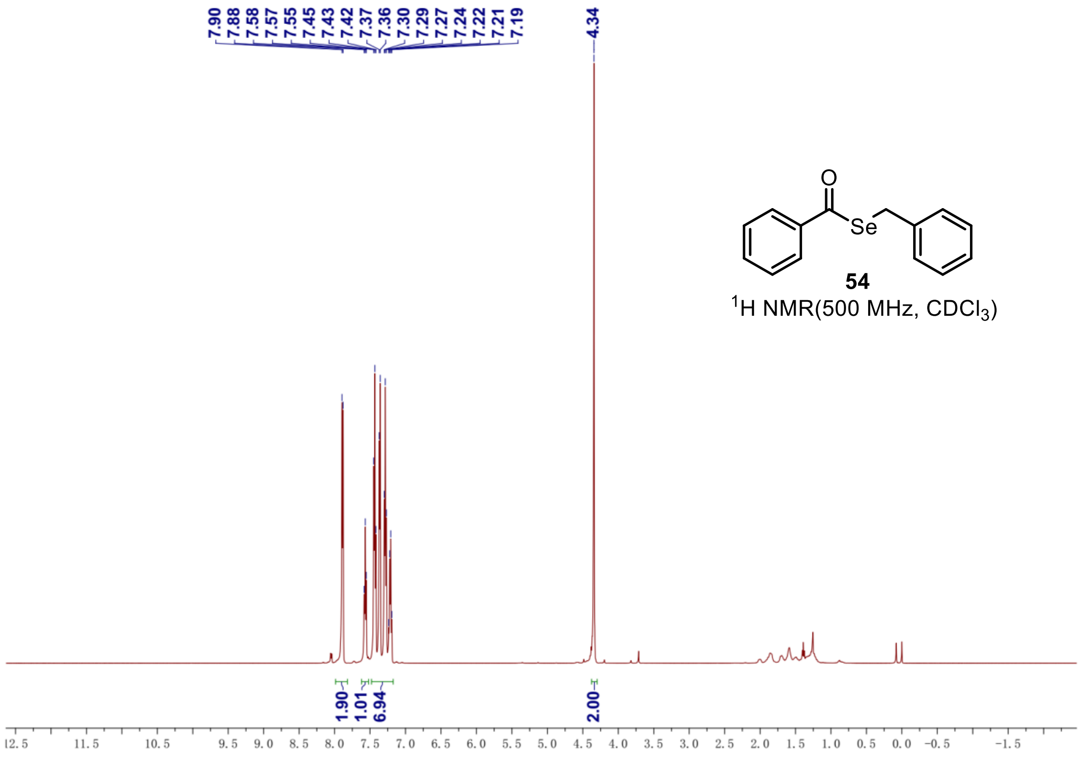
53

^{13}C NMR(125 MHz, CDCl$_3$)



53

^{19}F NMR(471 MHz, CDCl$_3$)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7.57
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7.45
7.43
7.42
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7.30
7.29
7.27
7.24
7.22
7.21
7.19
4.34
O
Se
54
^{1}H NMR(500 MHz, CDCl$_3$)
1.90
1.01
6.94
2.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
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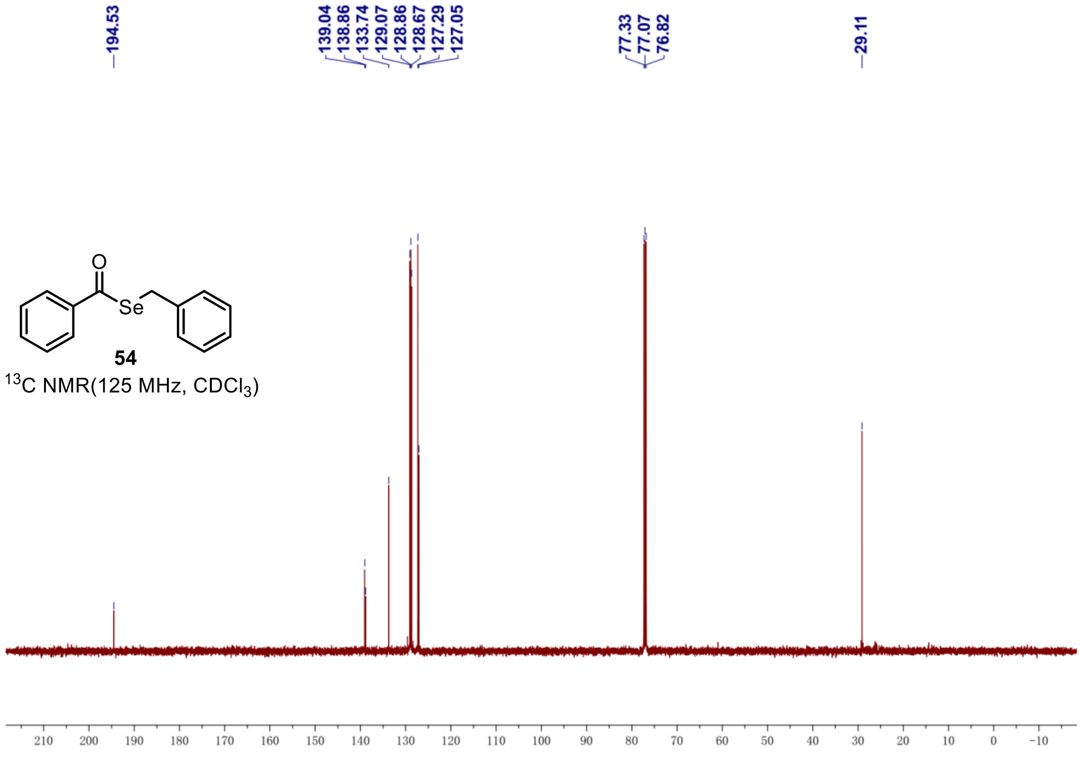
2.0
1.5
1.0
0.5
0.0
-0.5
-1.5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133.74
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128.86
128.67
127.29
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77.33
77.07
76.82
29.11
O
Se
54
^{13}C NMR(125 MHz, CDCl$_3$)
210
200
190
180
170
160
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140
130
120
110
100
90
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30
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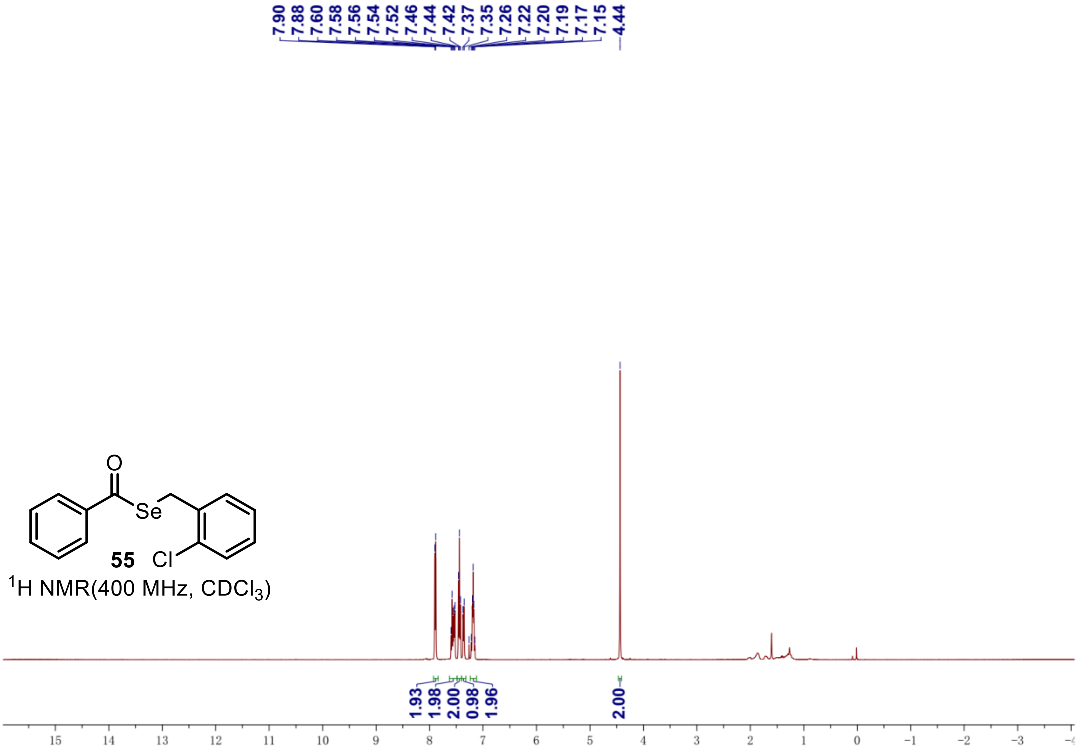
-10



55

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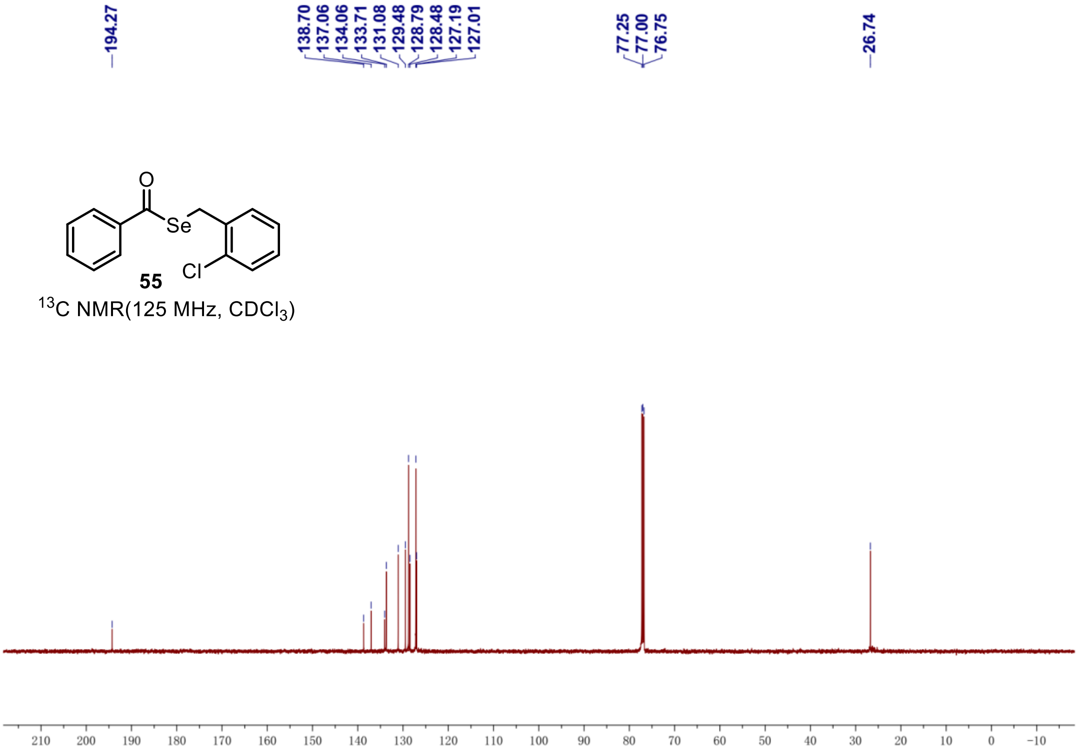
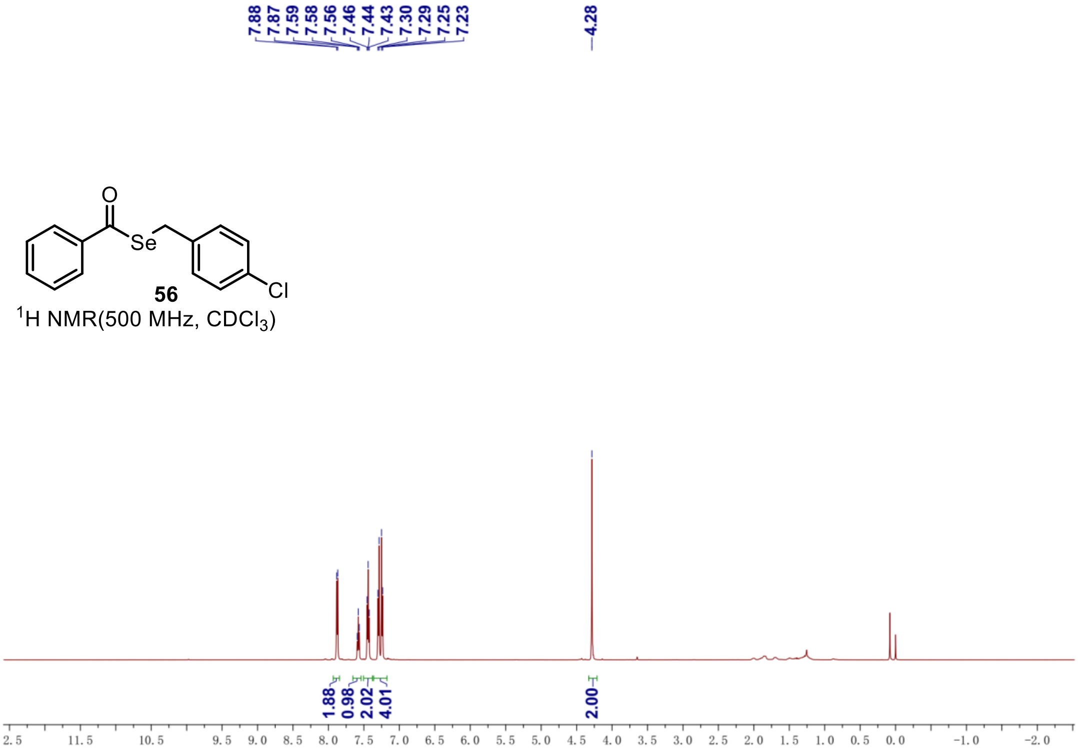


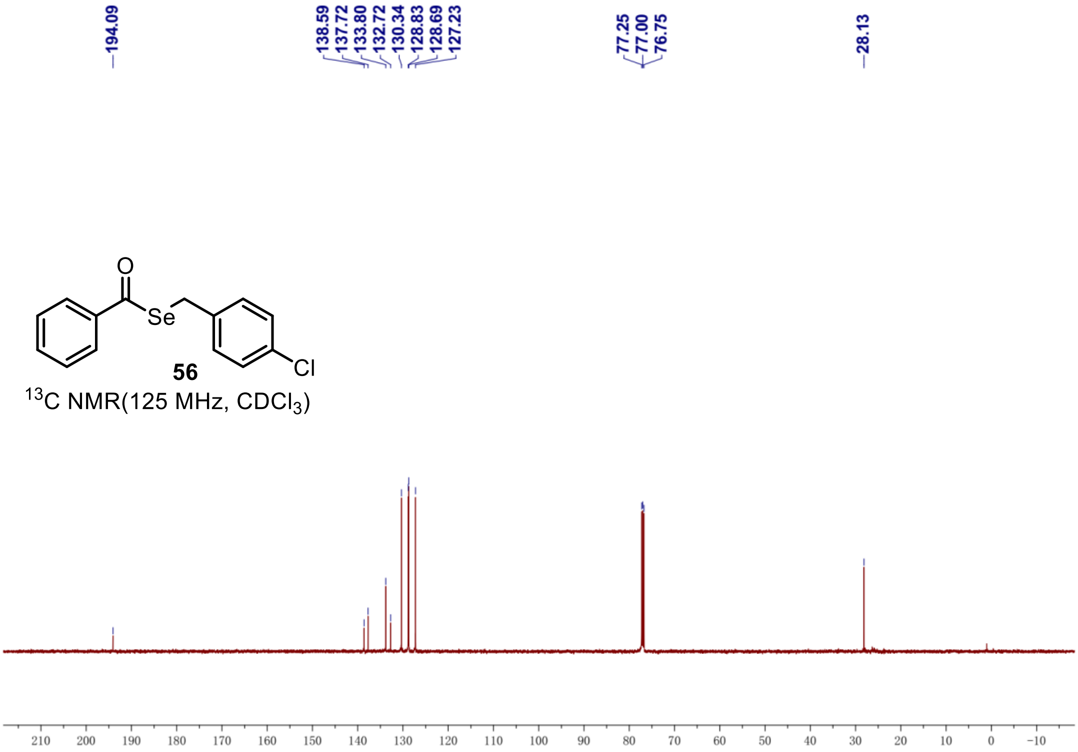
55

^{13}C NMR(125 MHz, CDCl$_3$)



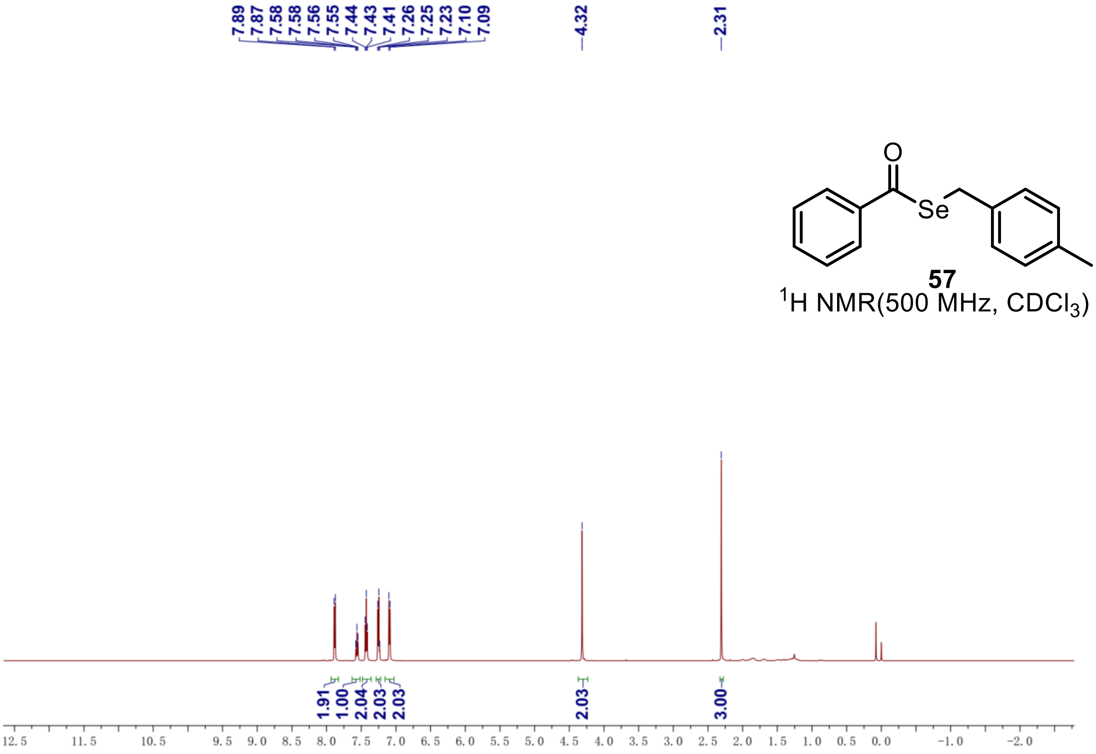
56

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



56

^{13}C NMR(125 MHz, $CDCl_3$)



57

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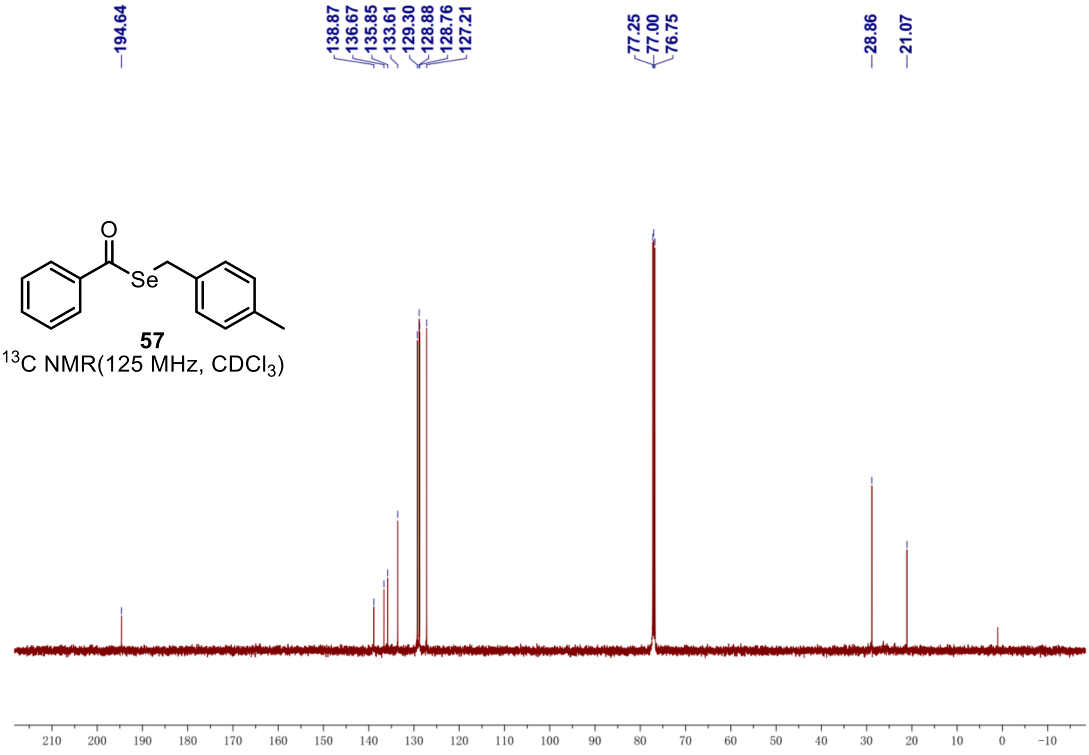

57

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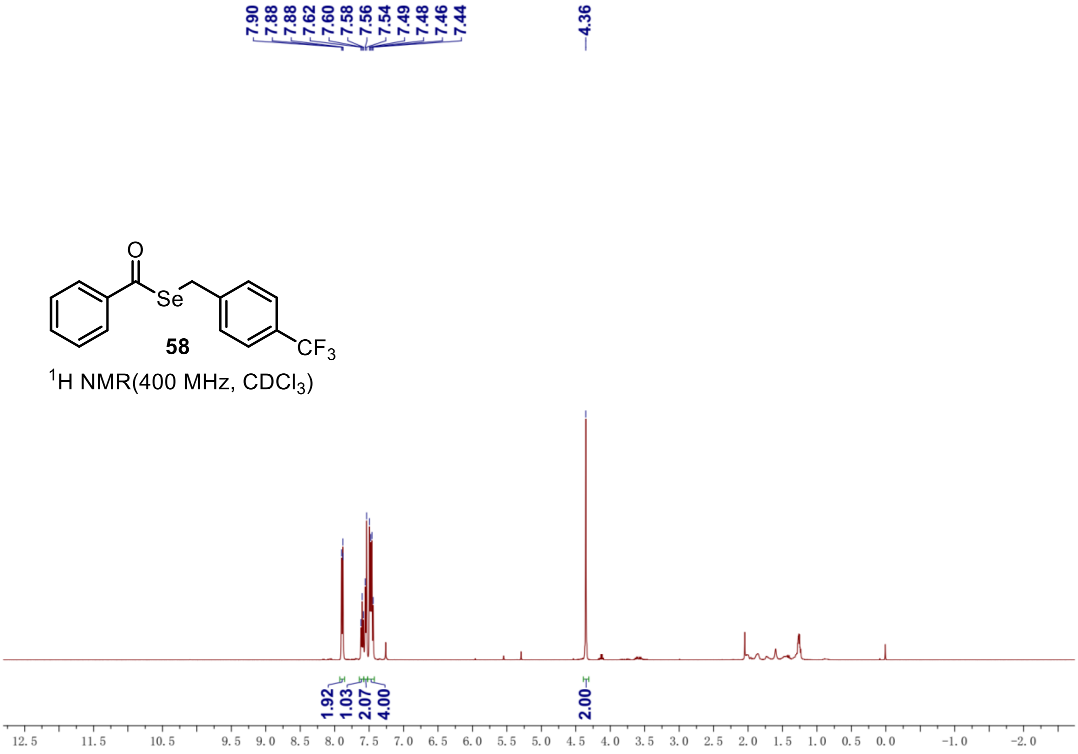

58

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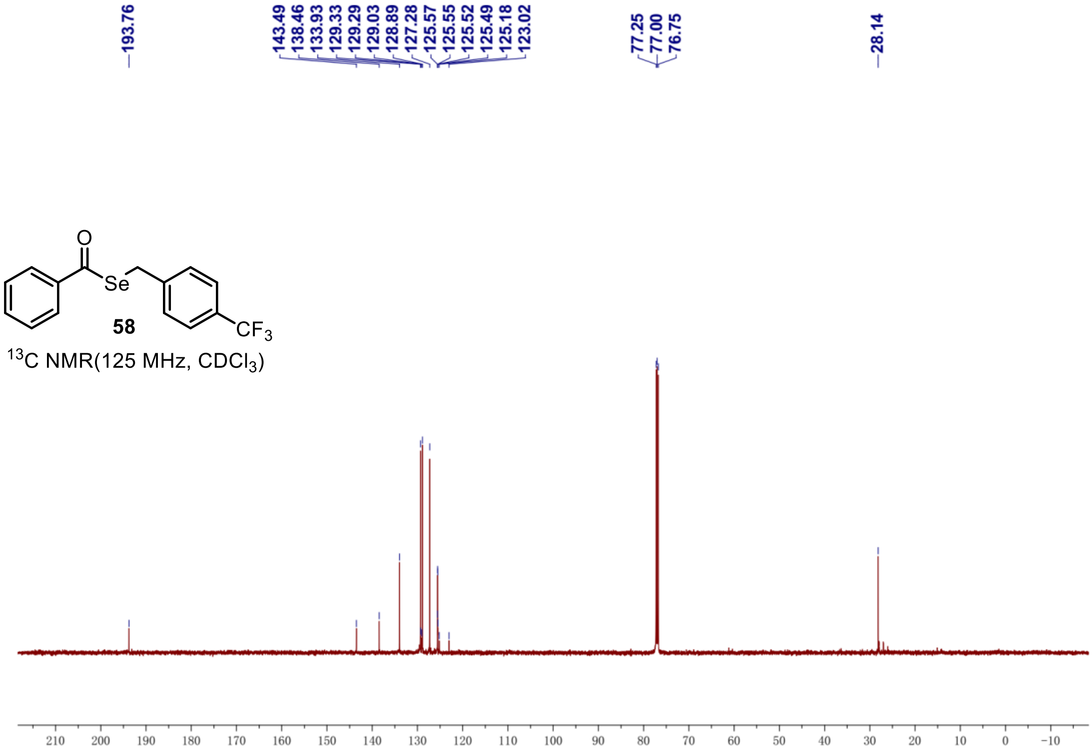

58

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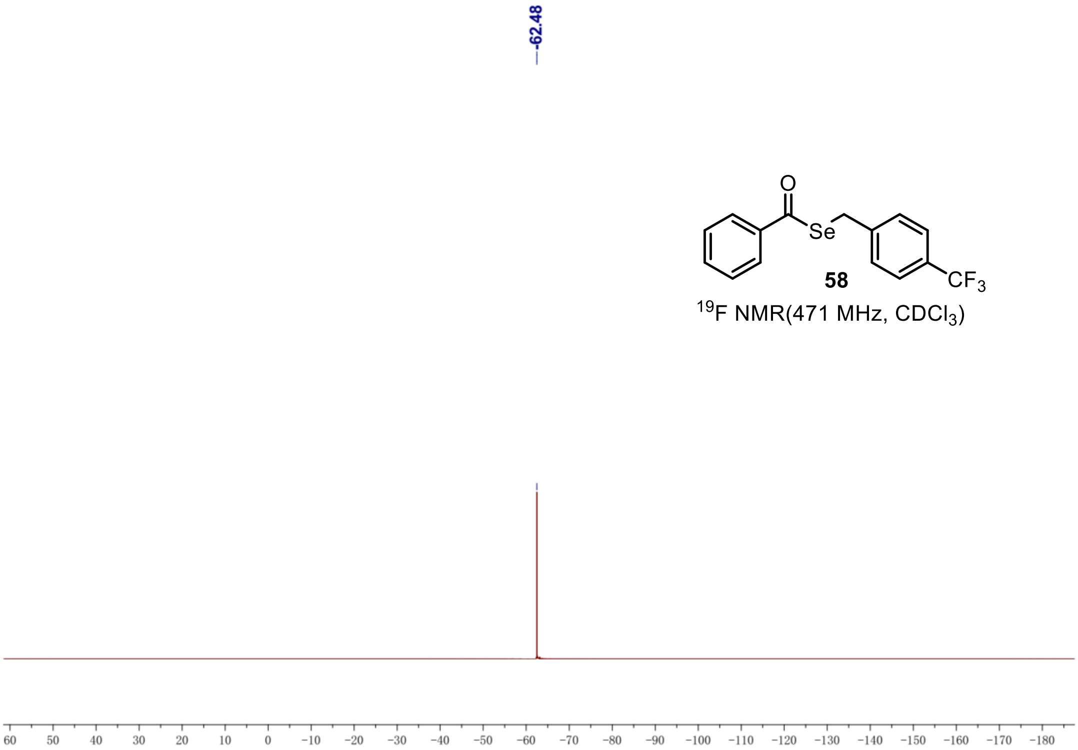
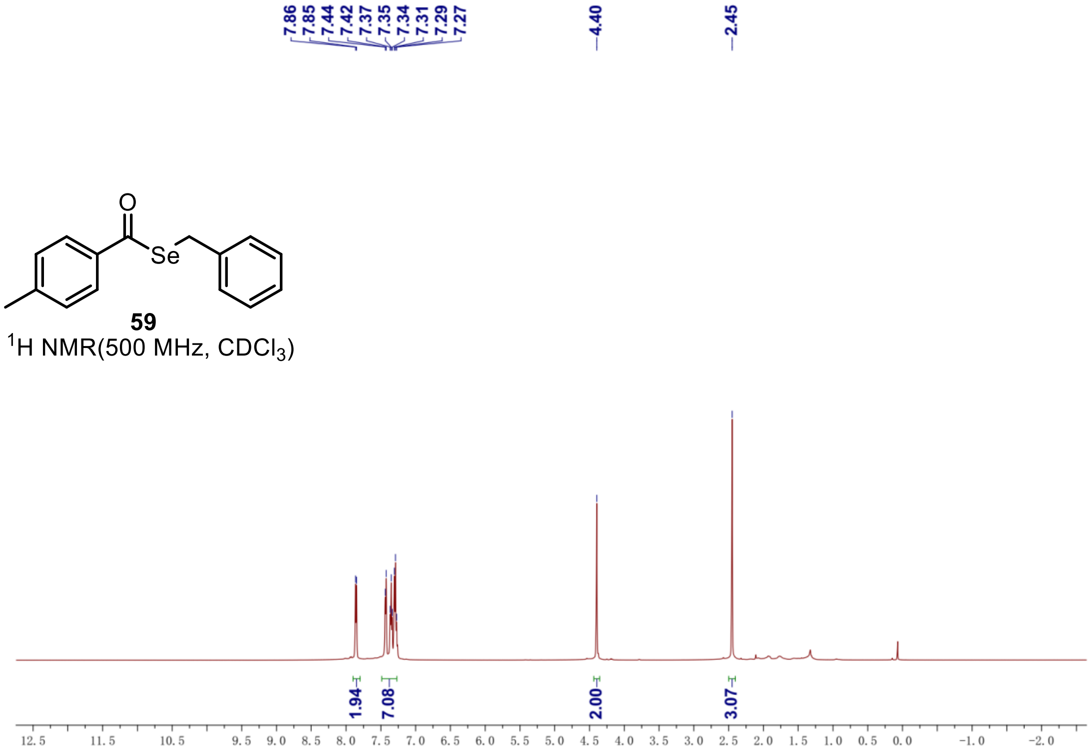


58

^{19}F NMR(471 MHz, CDCl$_3$)



59

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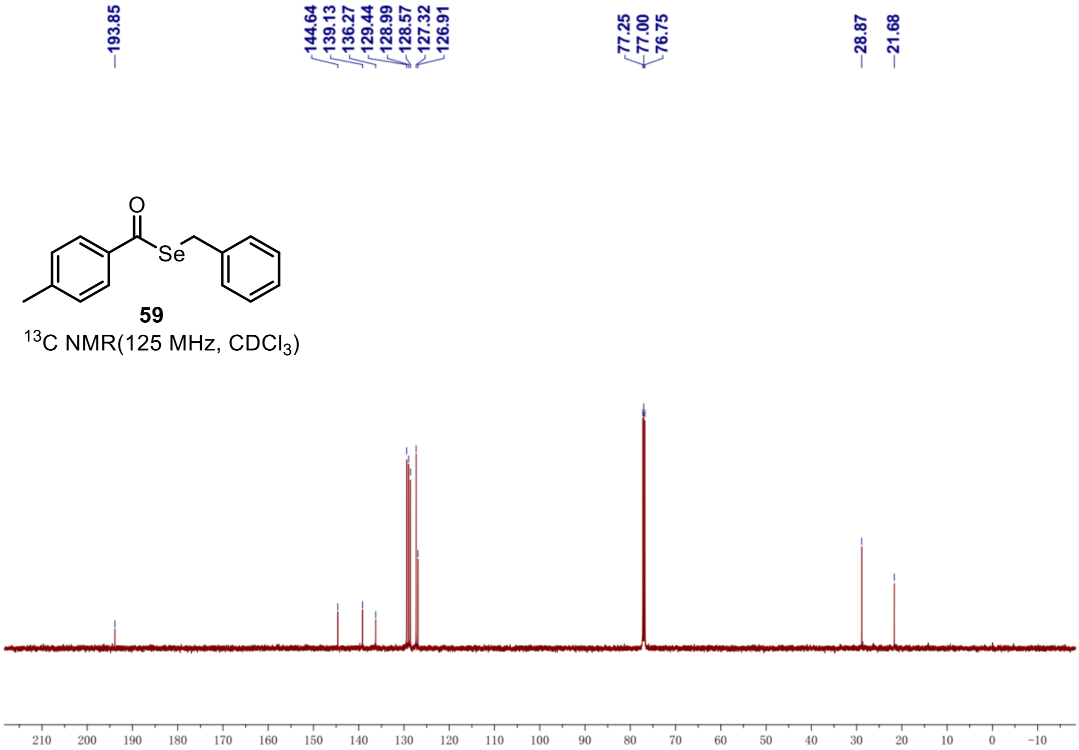
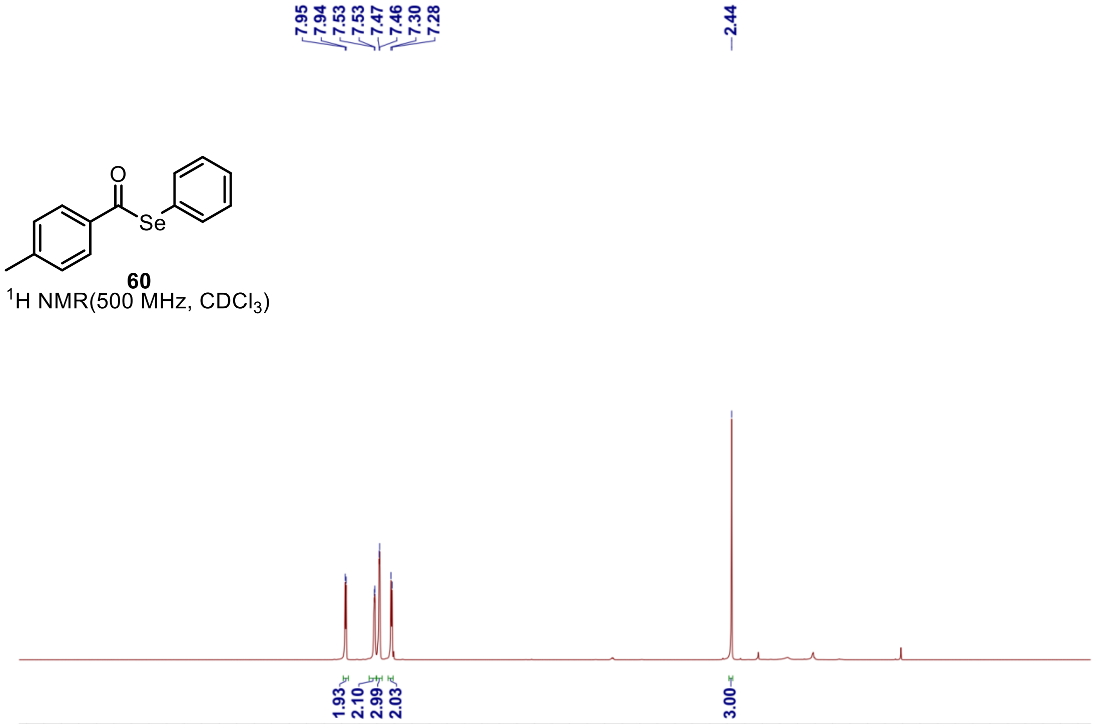


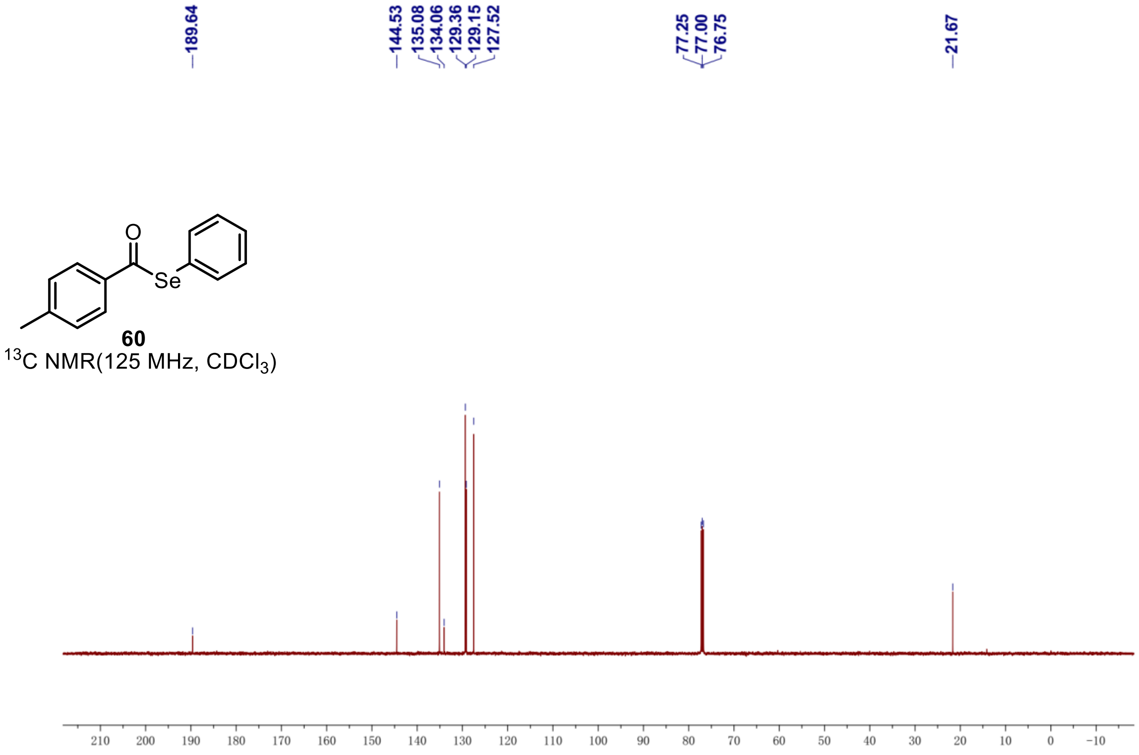
59

^{13}C NMR(125 MHz, CDCl$_3$)



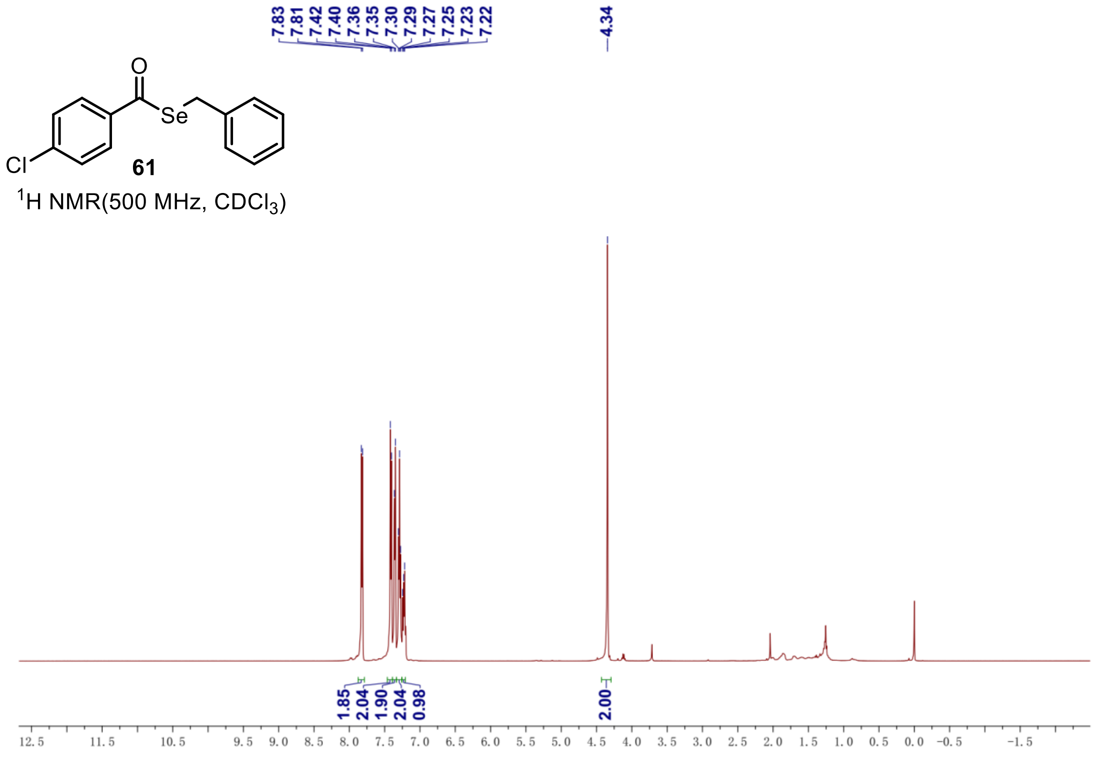
60

^{1}H NMR(500 MHz, CDCl$_3$)



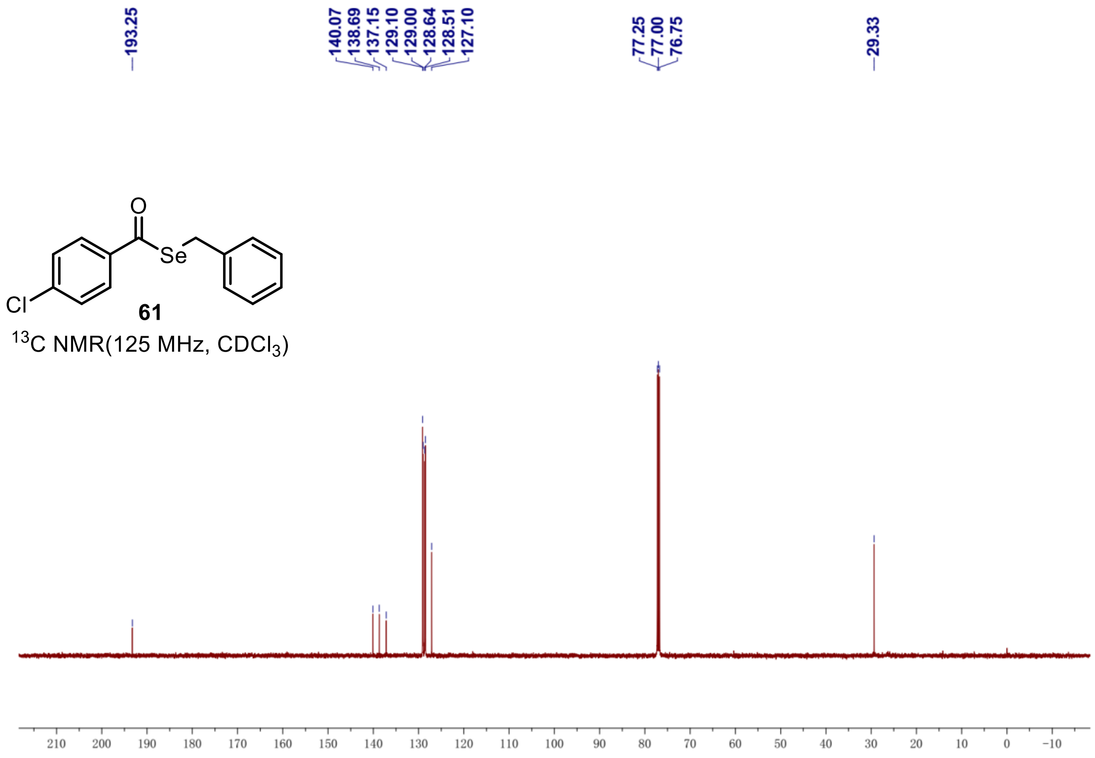
60

^{13}C NMR(125 MHz, $CDCl_3$)



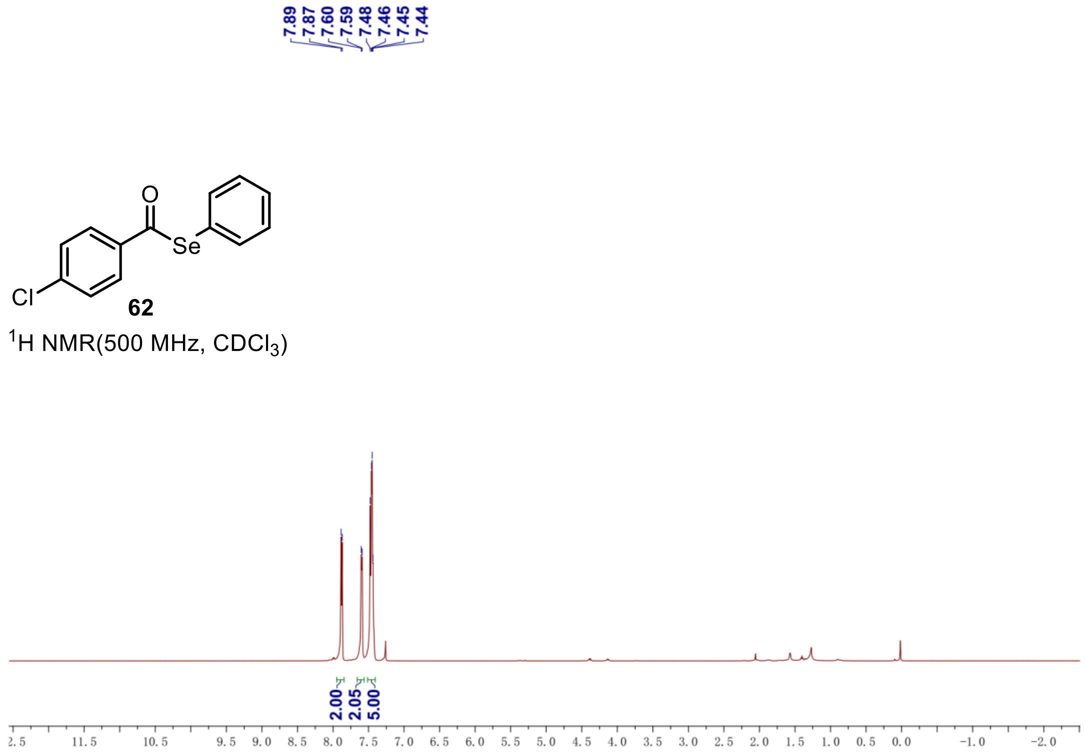
61

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



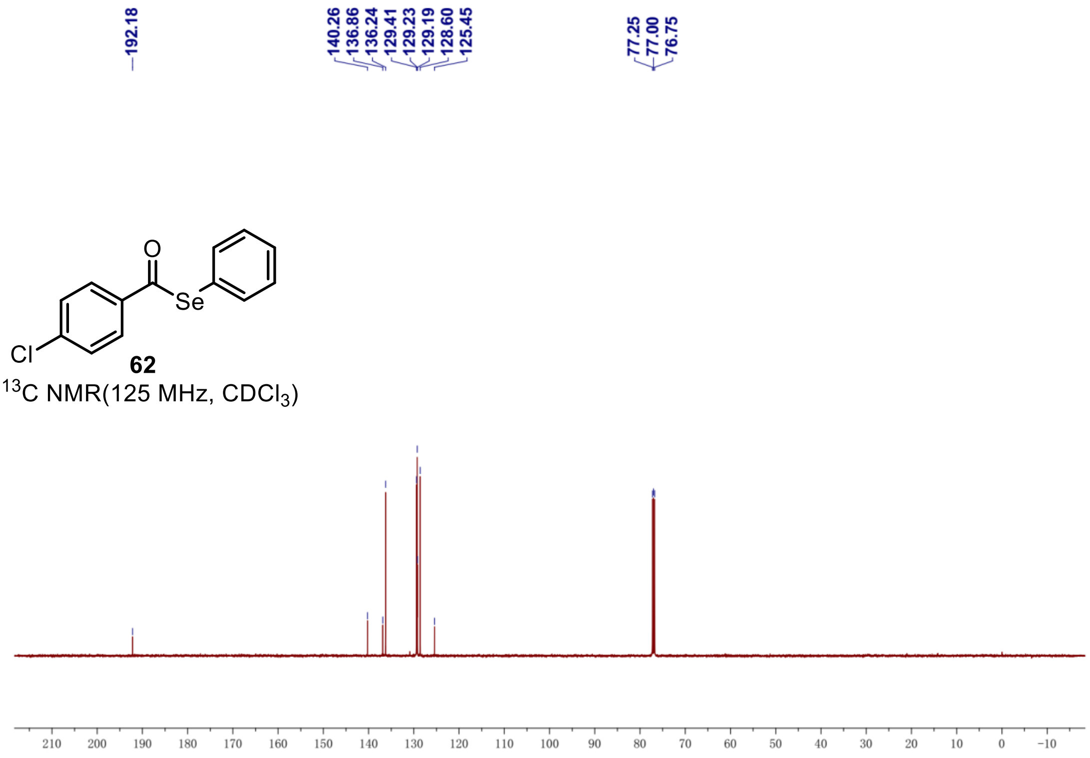
61

^{13}C NMR(125 MHz, CDCl$_3$)



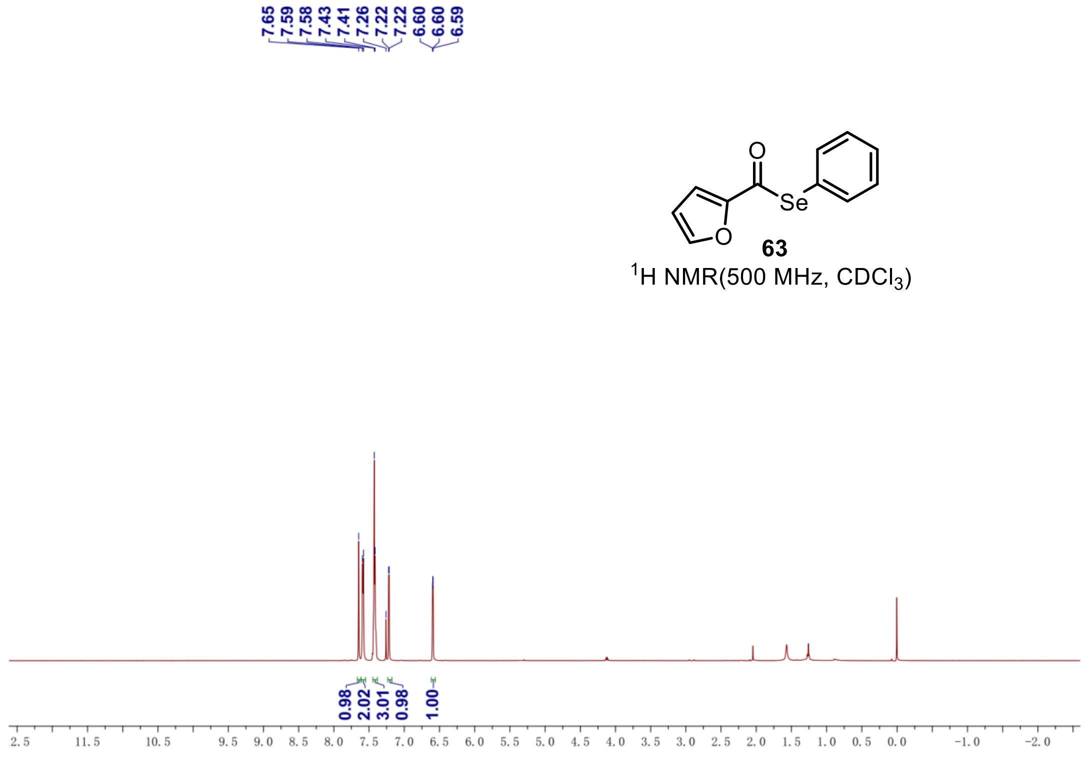
62

^{1}H NMR(500 MHz, CDCl$_3$)



62

^{13}C NMR(125 MHz, CDCl$_3$)



63

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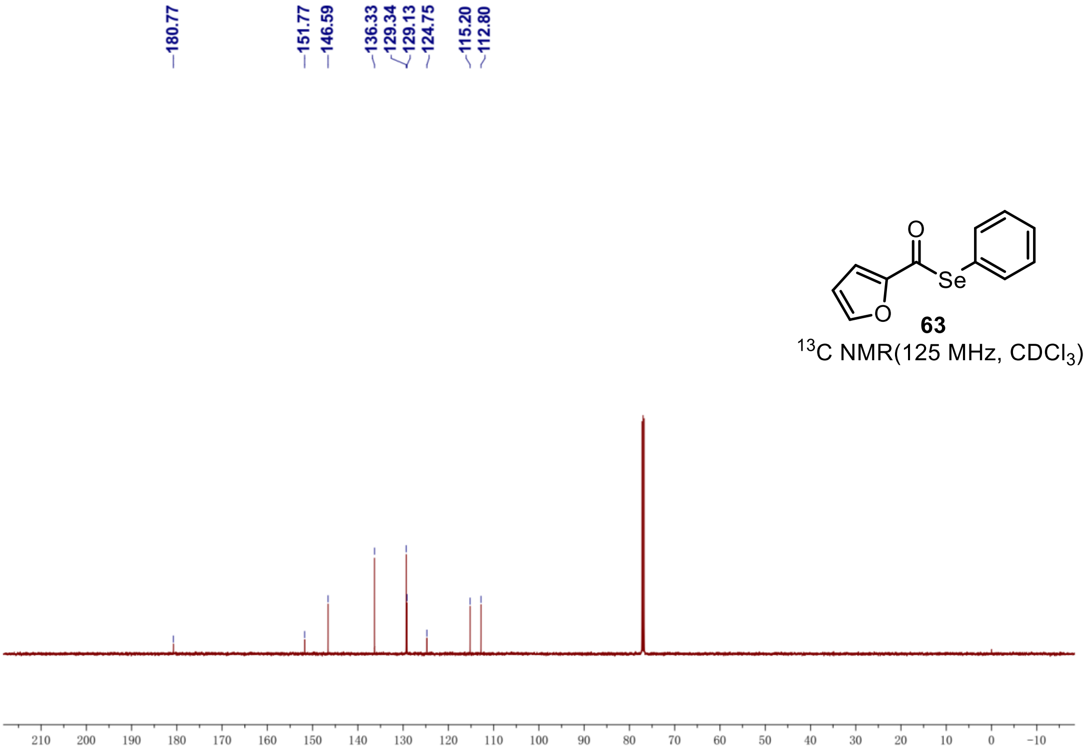

63

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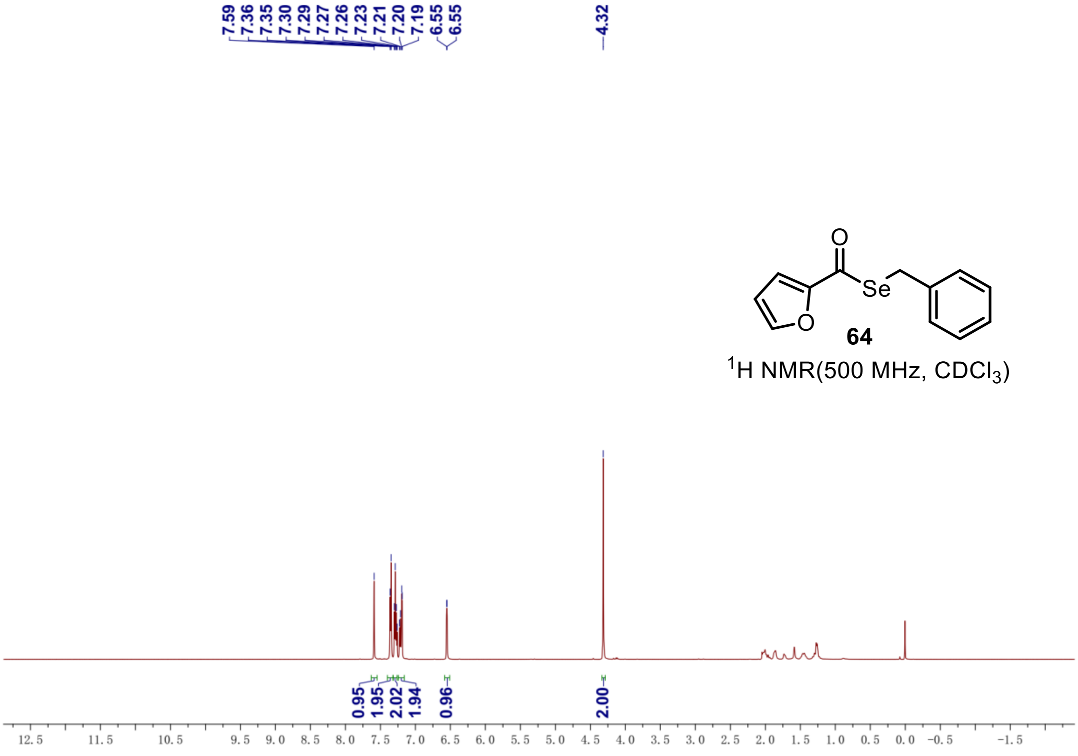

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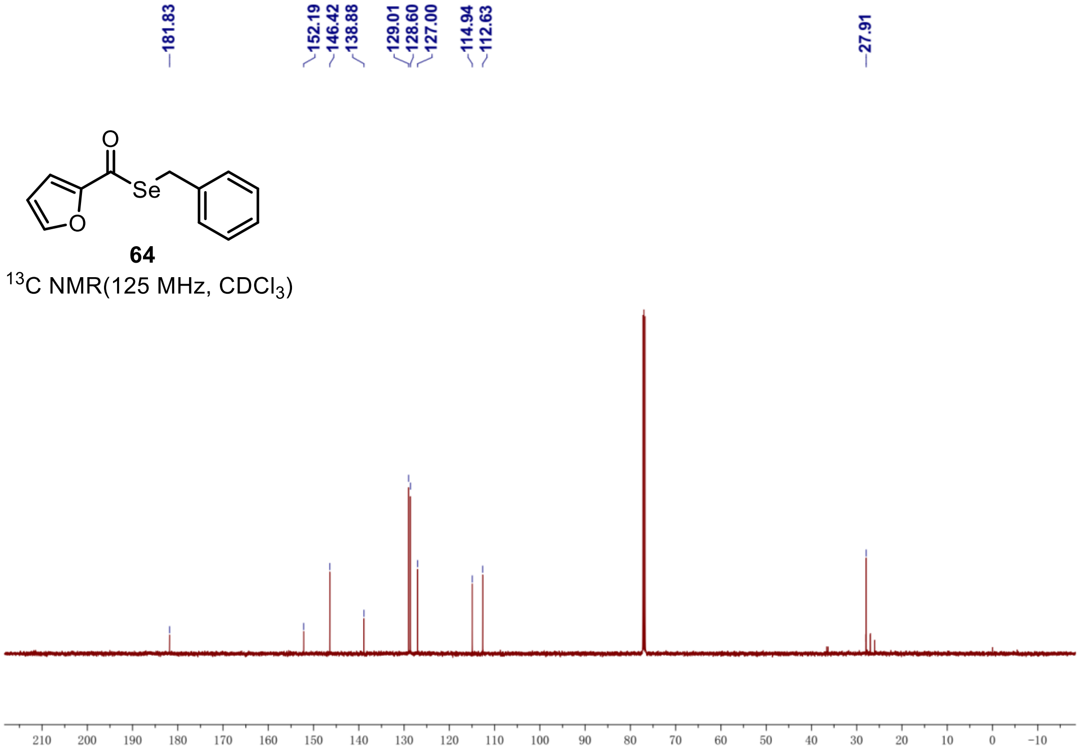

64

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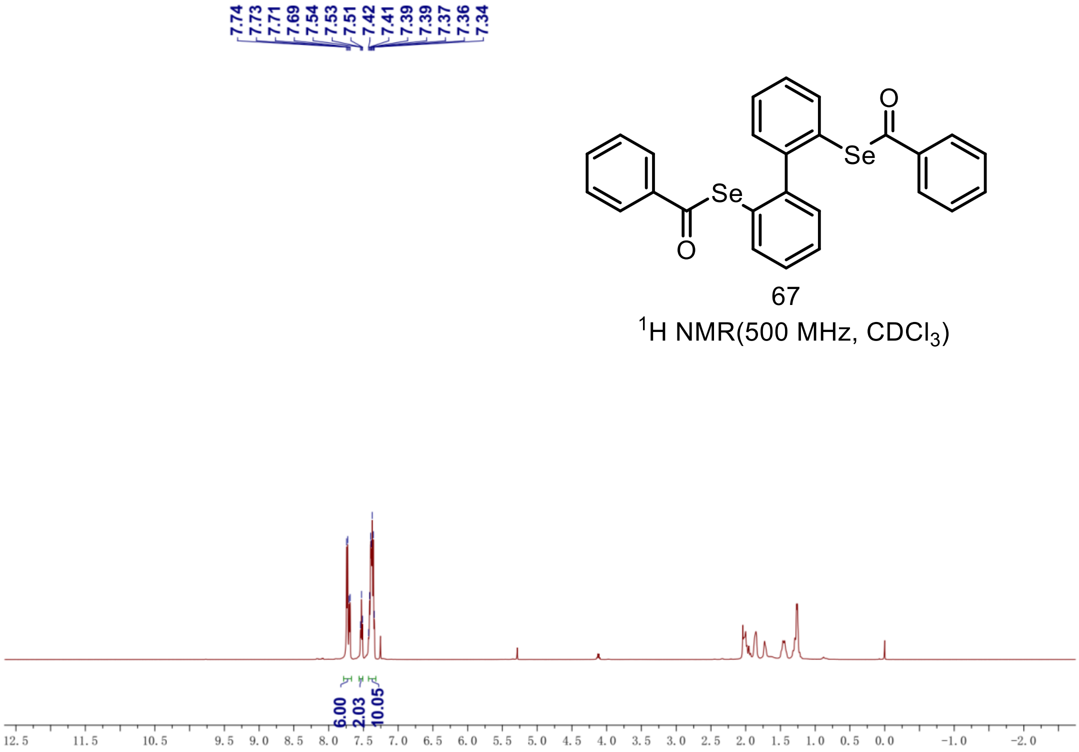
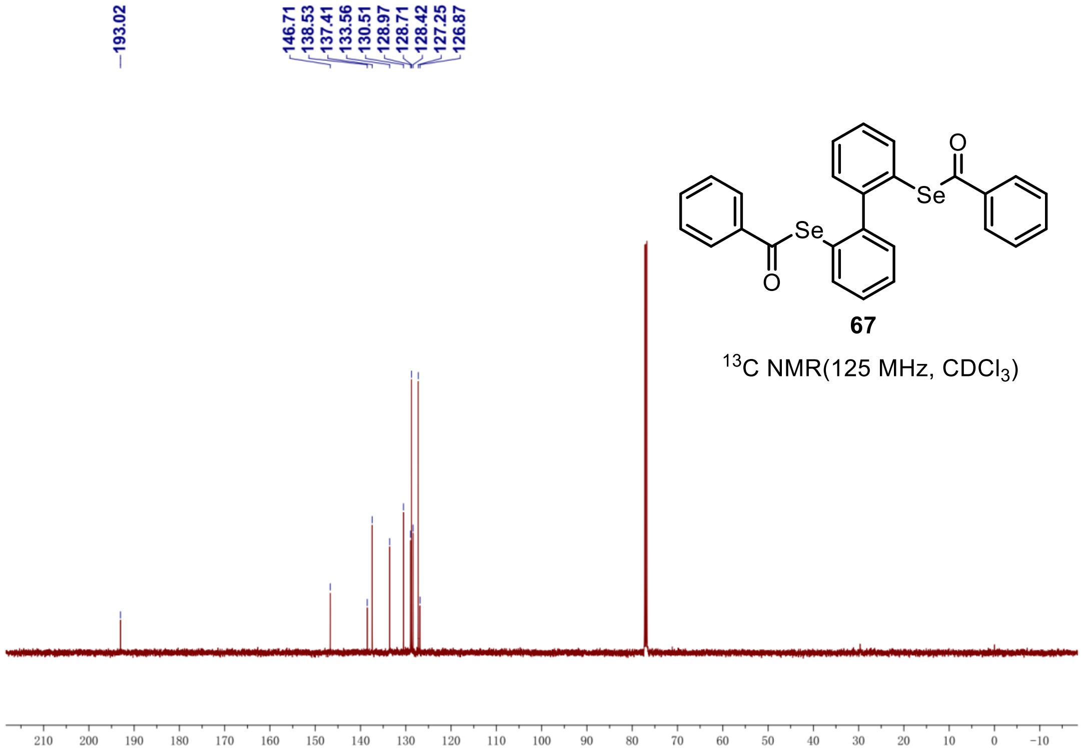


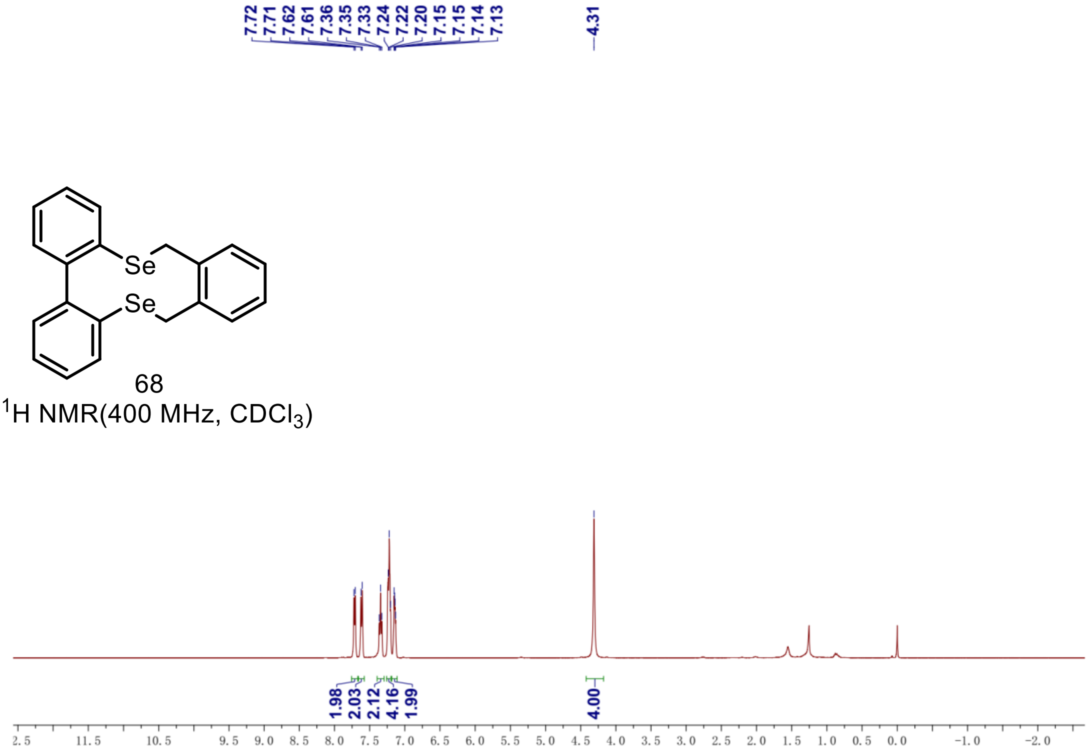
67

^{1}H NMR(500 MHz, CDCl$_3$)



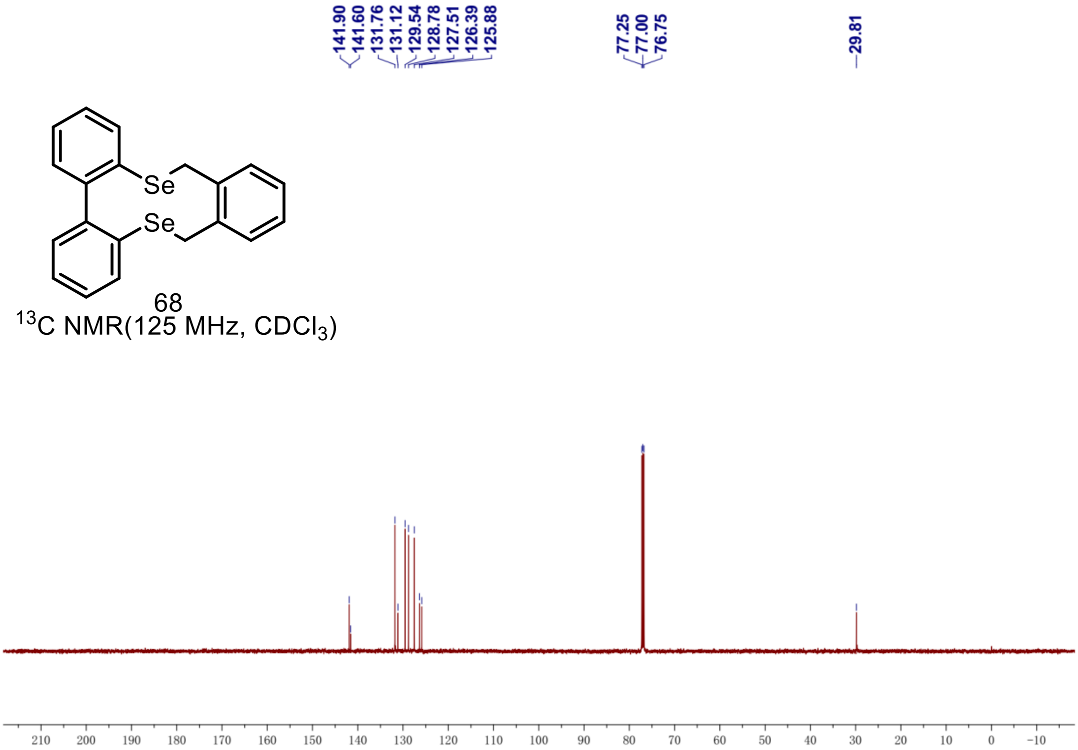
67

^{13}C NMR(125 MHz, CDCl$_3$)



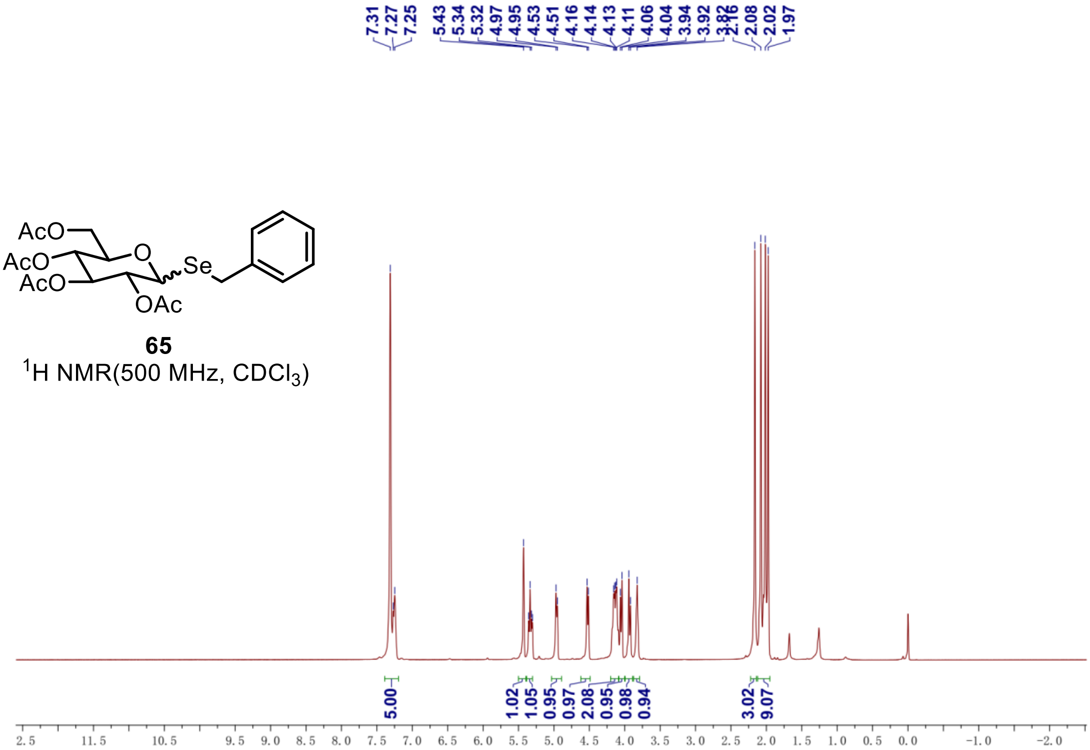
68

^{1}H NMR(400 MHz, CDCl$_3$)



68

^{13}C NMR(125 MHz, CDCl$_3$)



65

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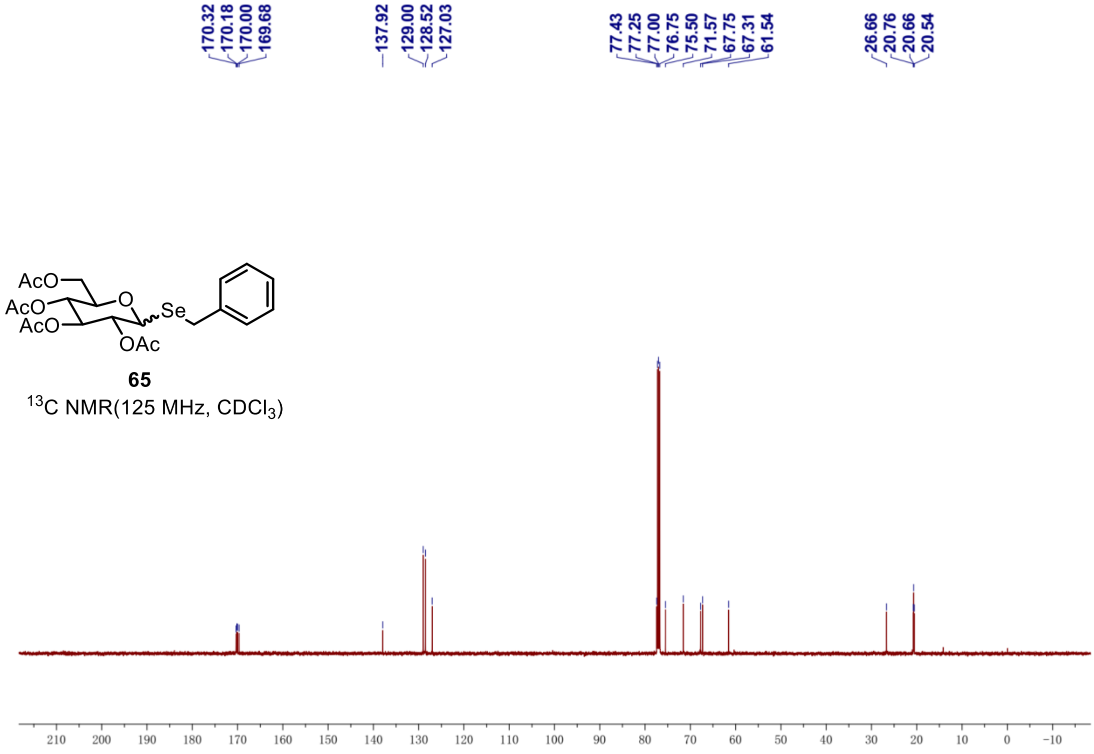

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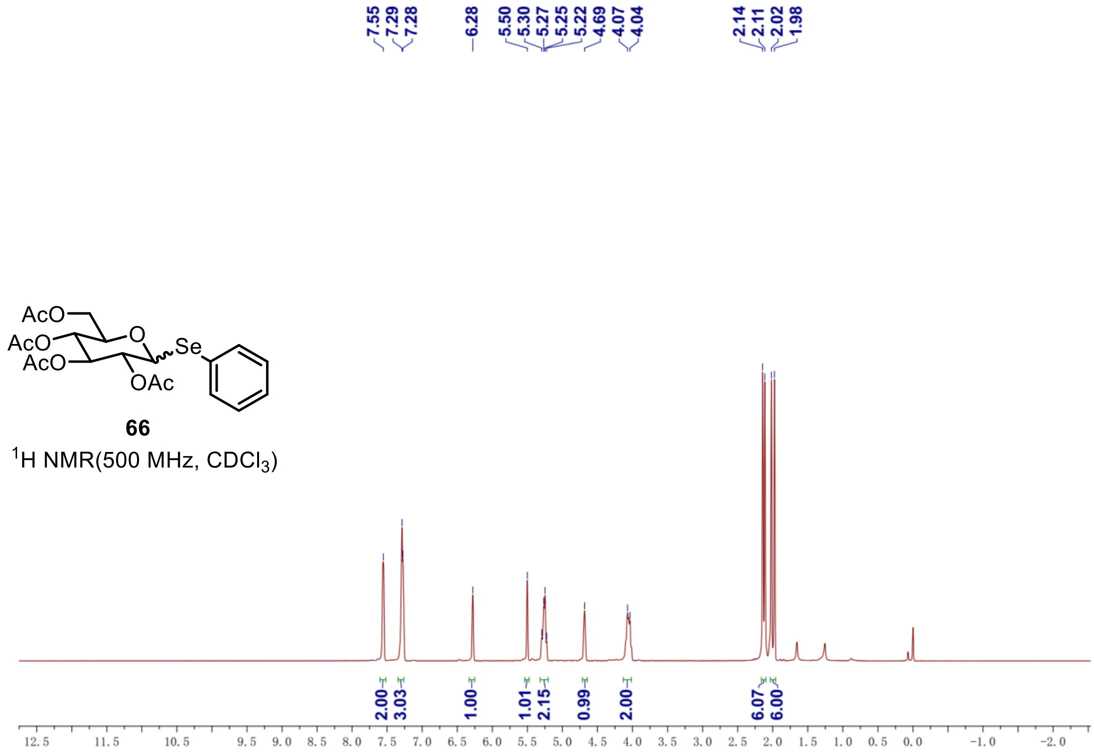

66

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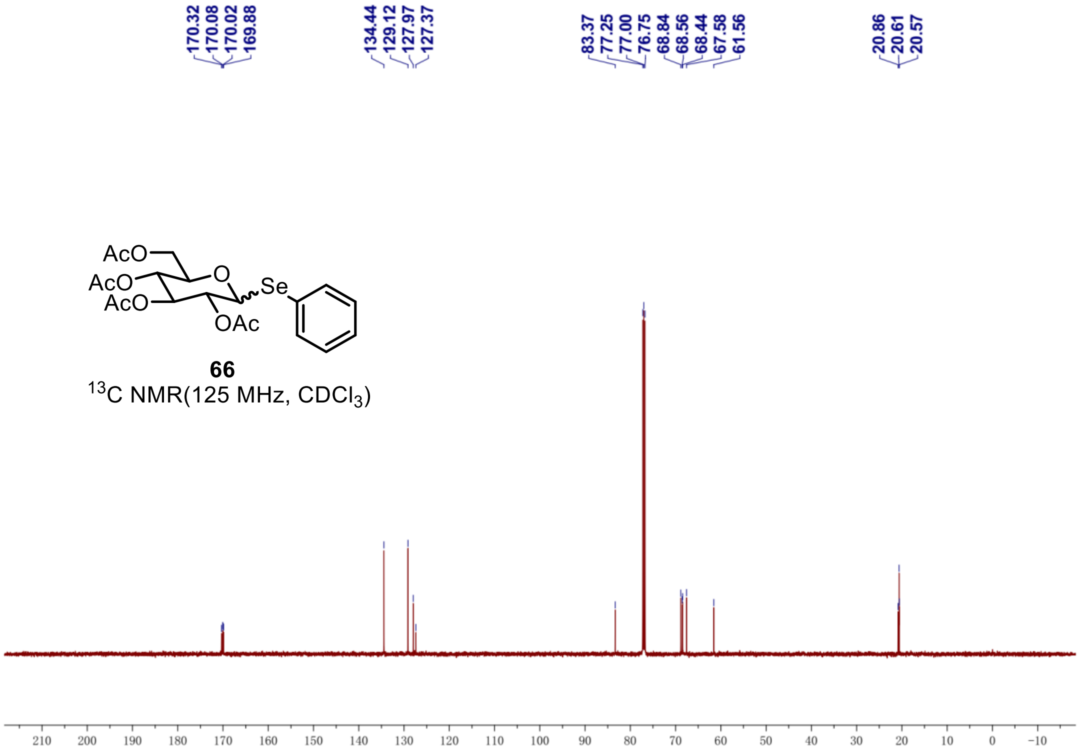

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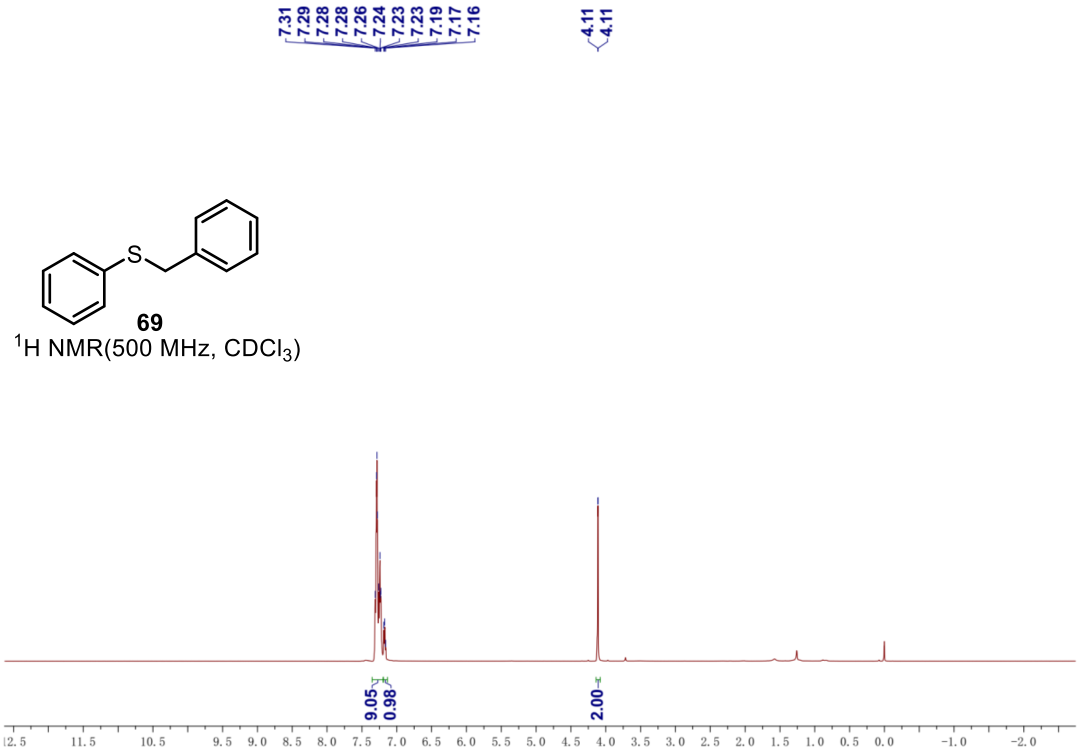

69

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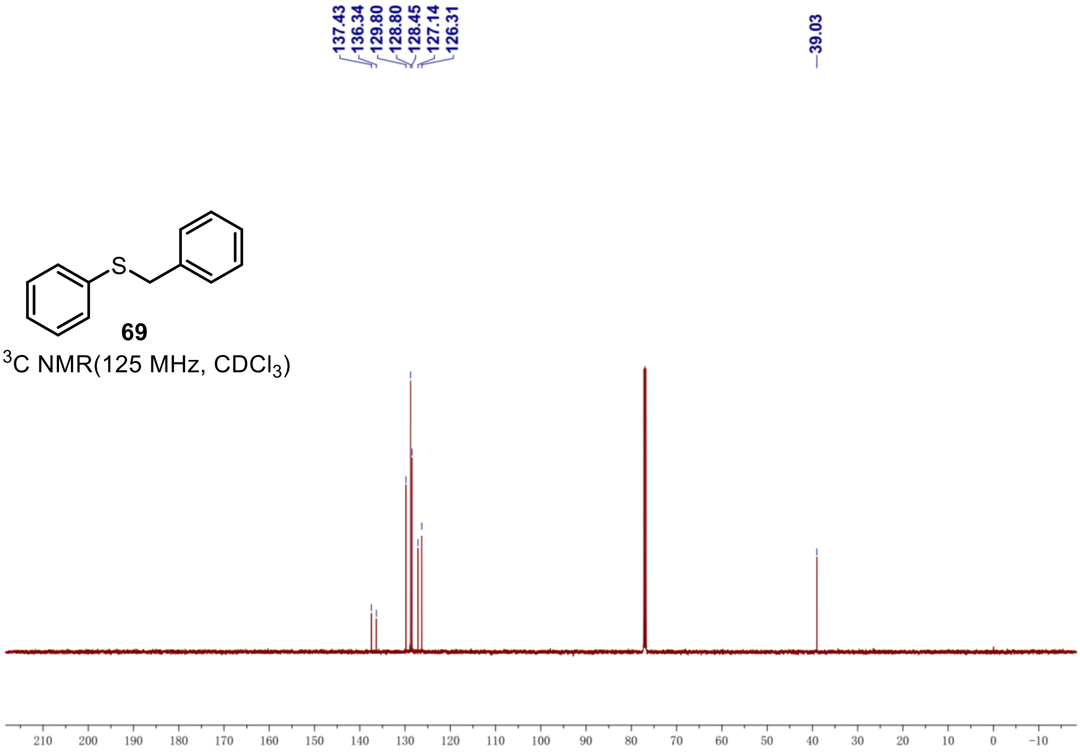
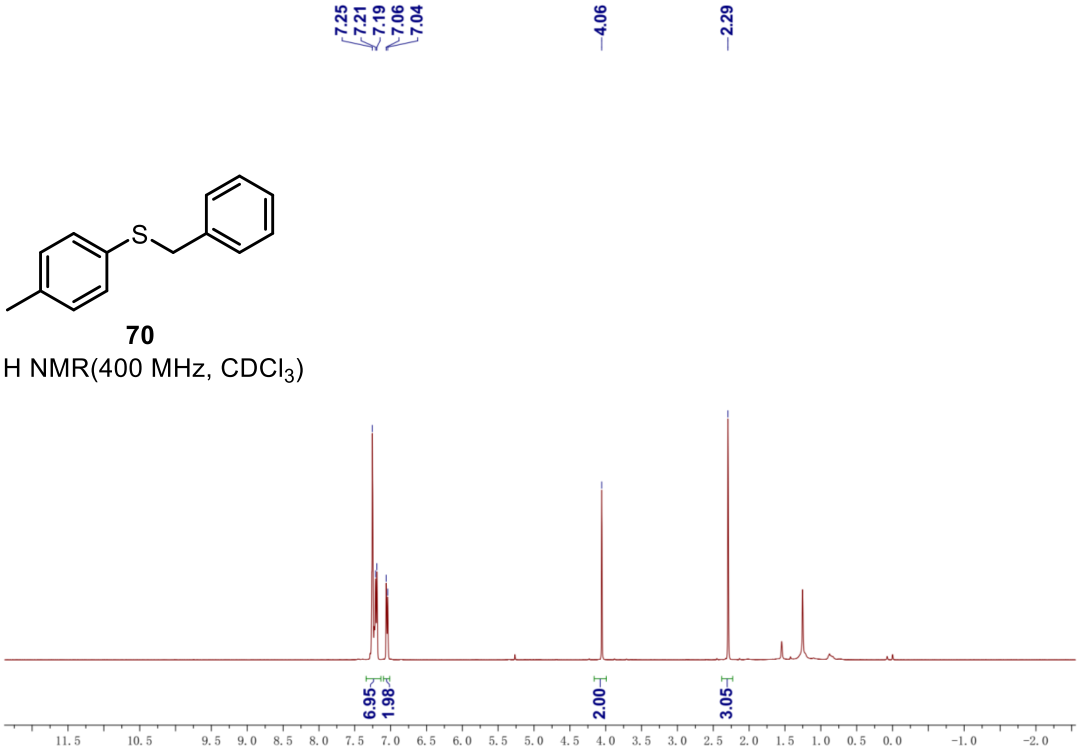


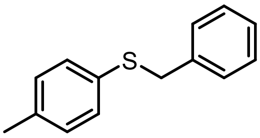
69

^{13}C NMR(125 MHz, CDCl$_3$)



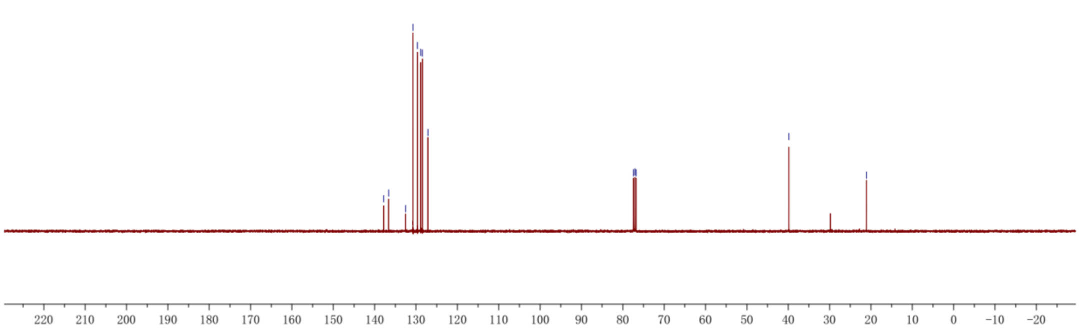
70

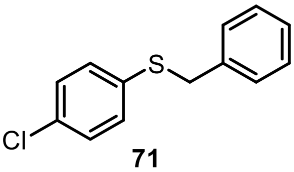
^{1}H NMR(400 MHz, CDCl$_3$)



70

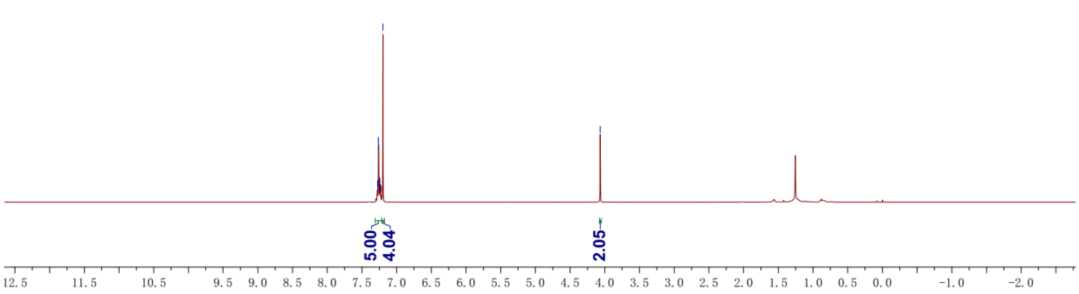
^{13}C NMR(100 MHz, CDCl$_3$)

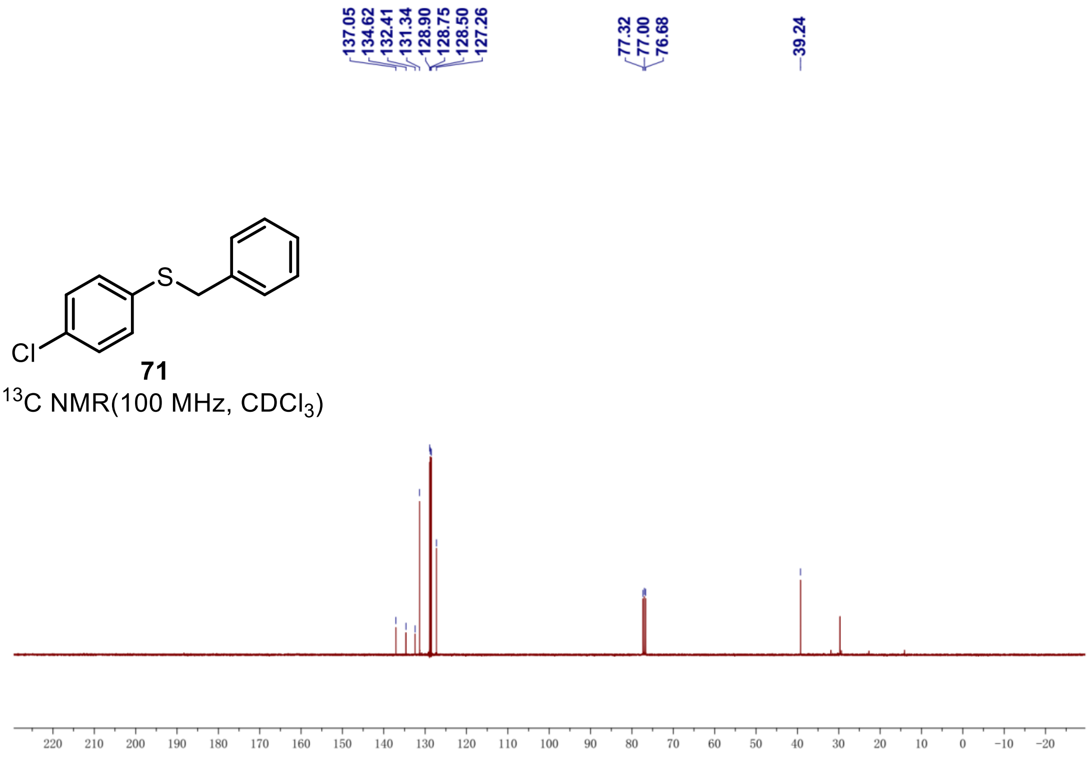




71

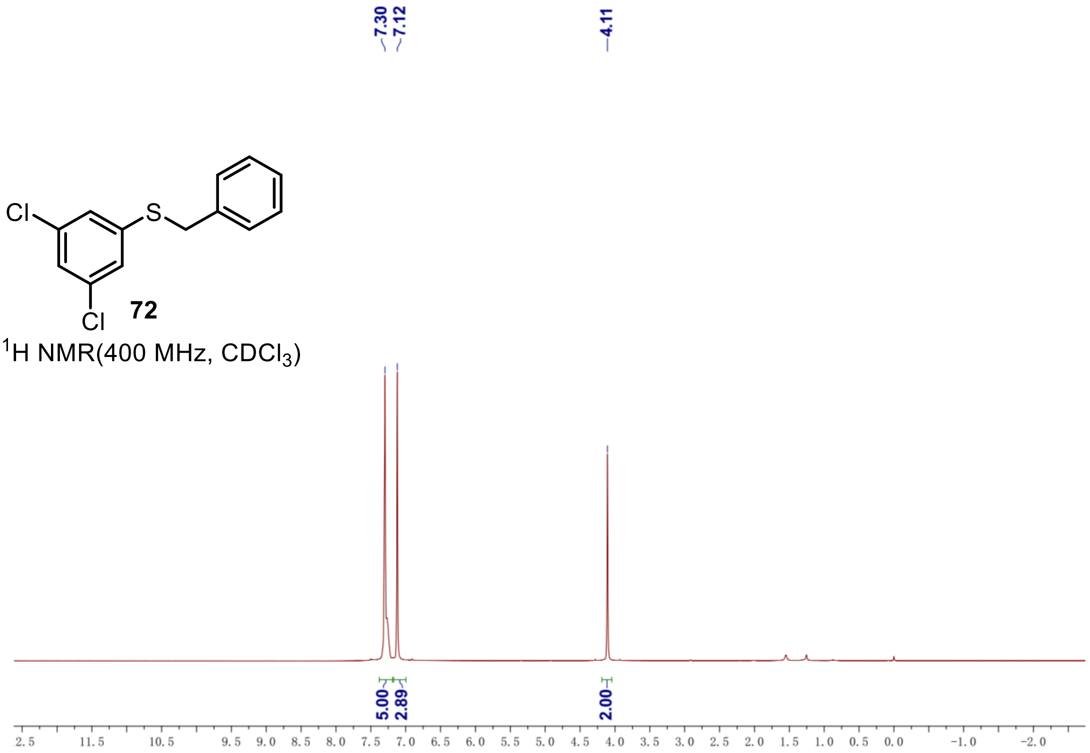
^{1}H NMR(400 MHz, CDCl$_3$)





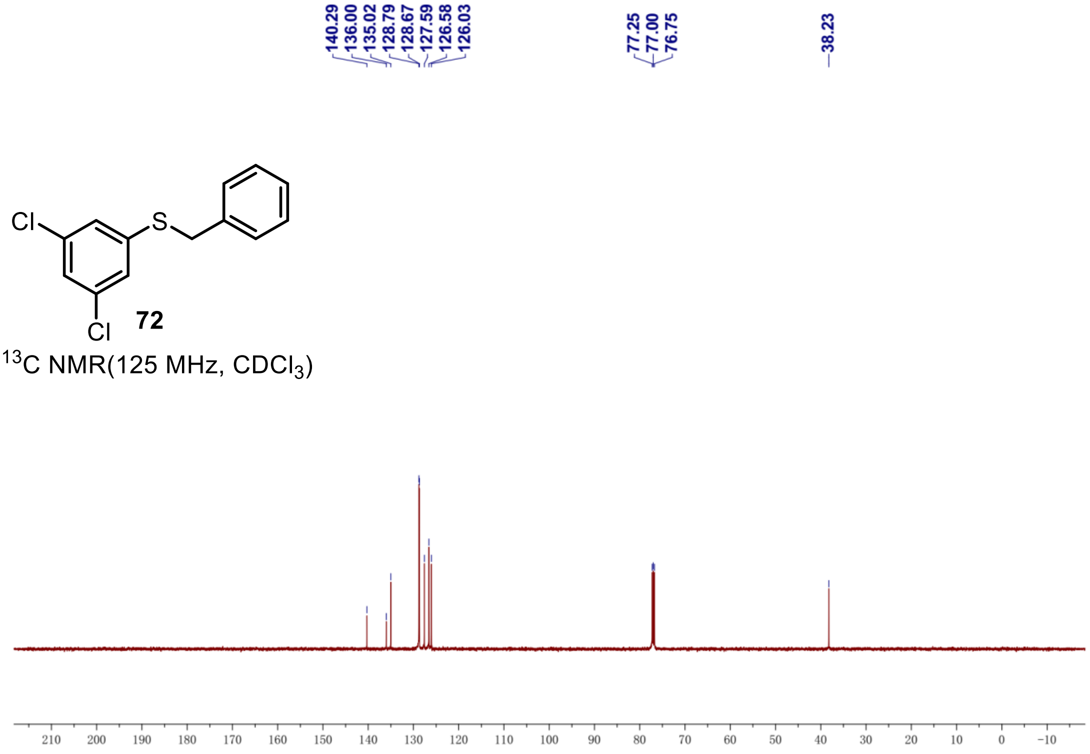
71

^{13}C NMR(100 MHz, CDCl$_3$)



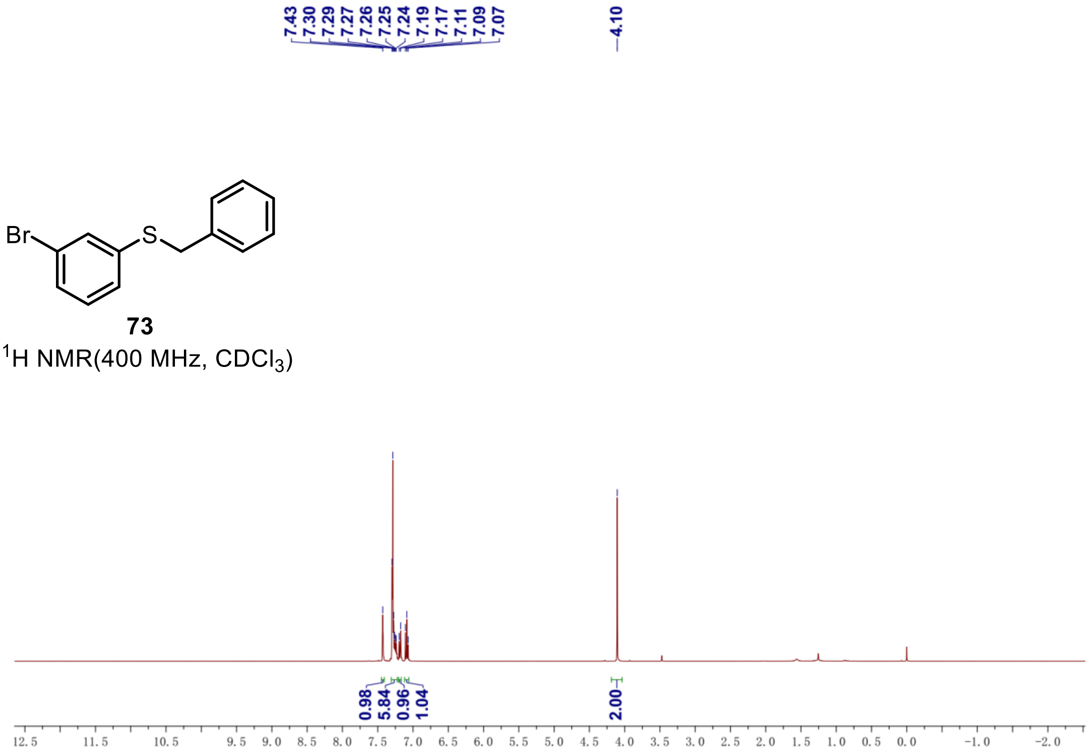
72

^{1}H NMR(400 MHz, CDCl$_3$)



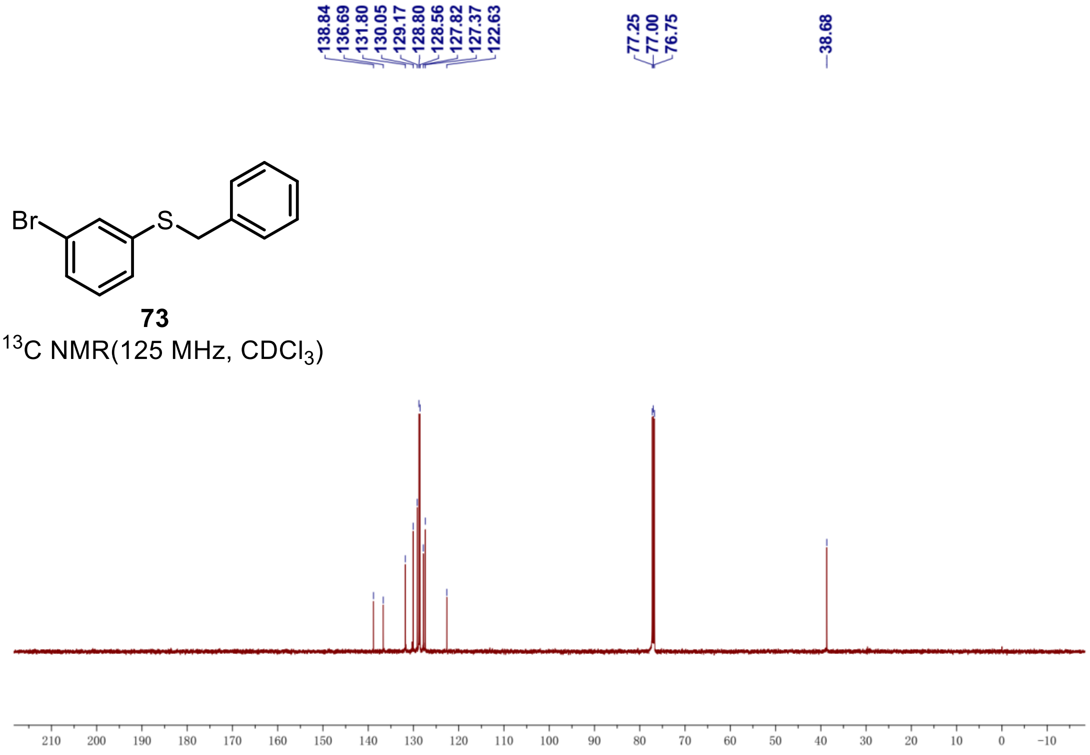
72

^{13}C NMR(125 MHz, CDCl$_3$)



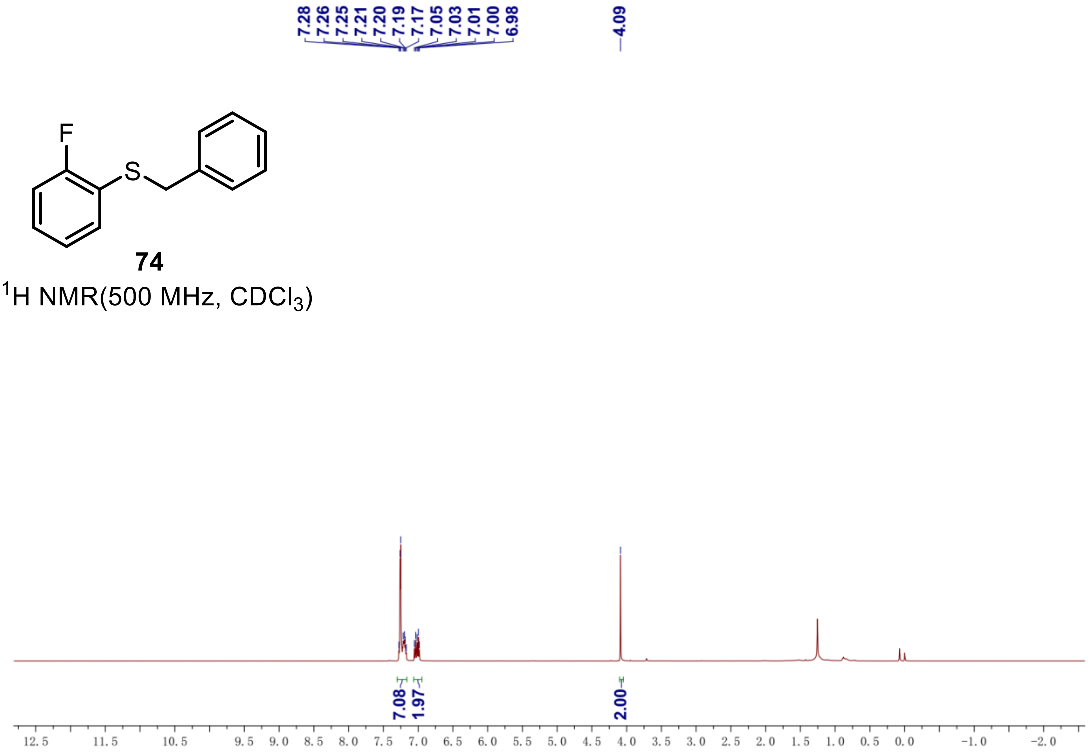
73

^{1}H NMR(400 MHz, CDCl$_3$)



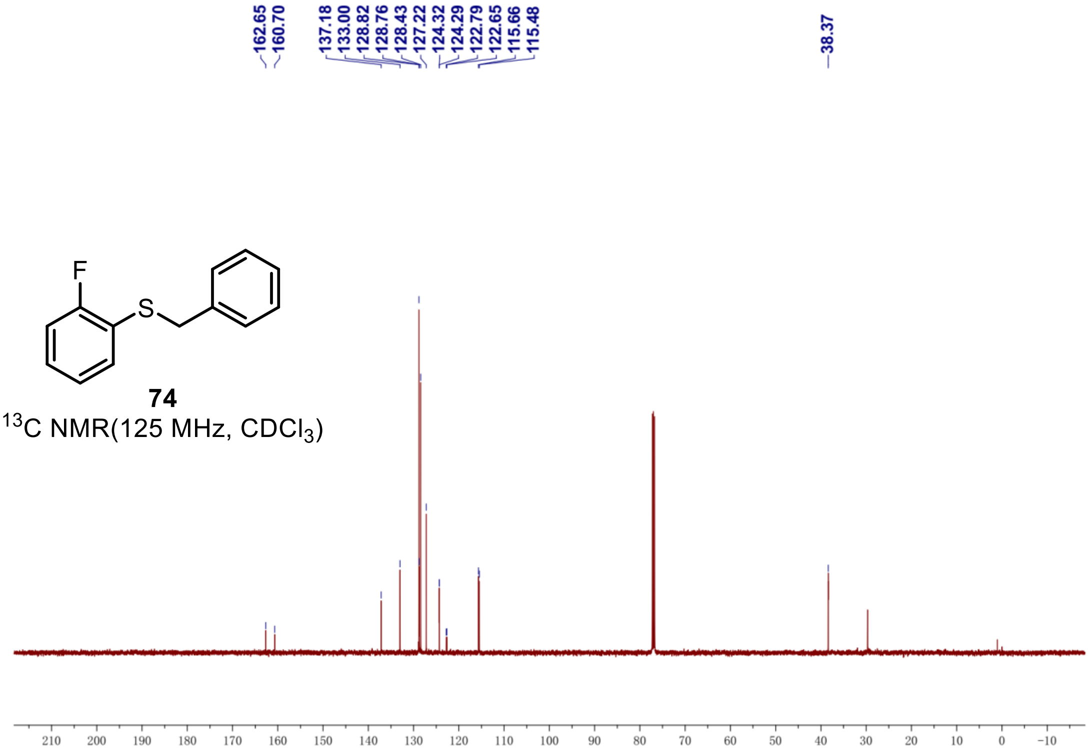
73

^{13}C NMR(125 MHz, CDCl$_3$)



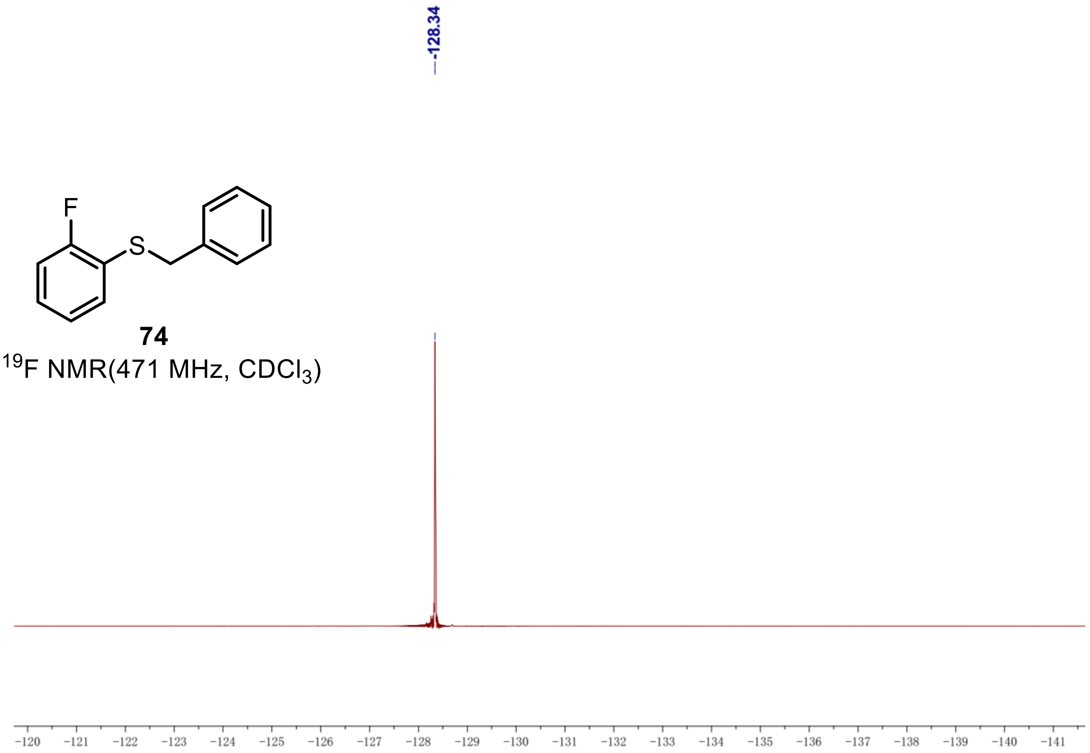
74

^{1}H NMR(500 MHz, CDCl$_3$)



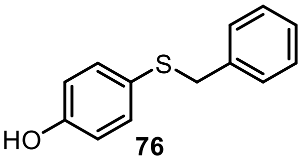
74

^{13}C NMR(125 MHz, $CDCl_3$)



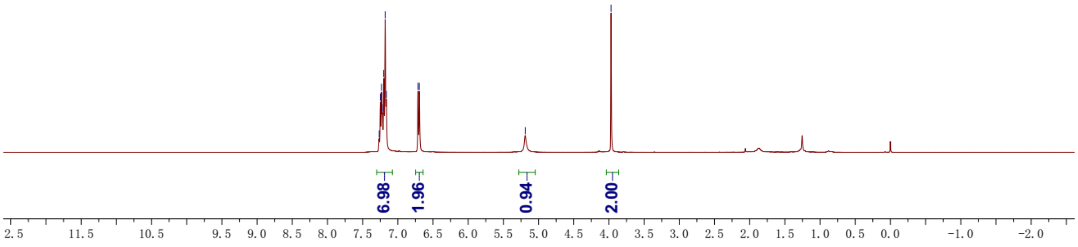
74

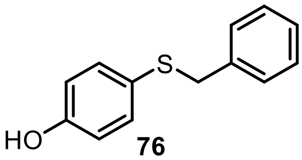
^{19}F NMR(471 MHz, $CDCl_3$)



76

^{1}H NMR(400 MHz, CDCl$_3$)





76

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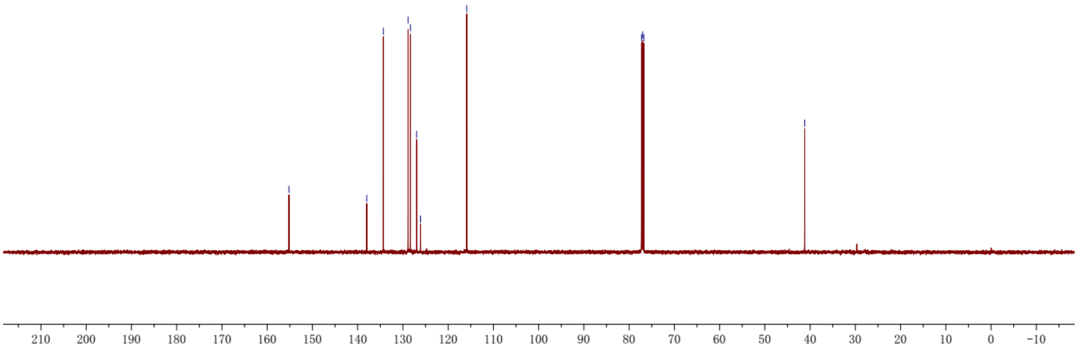
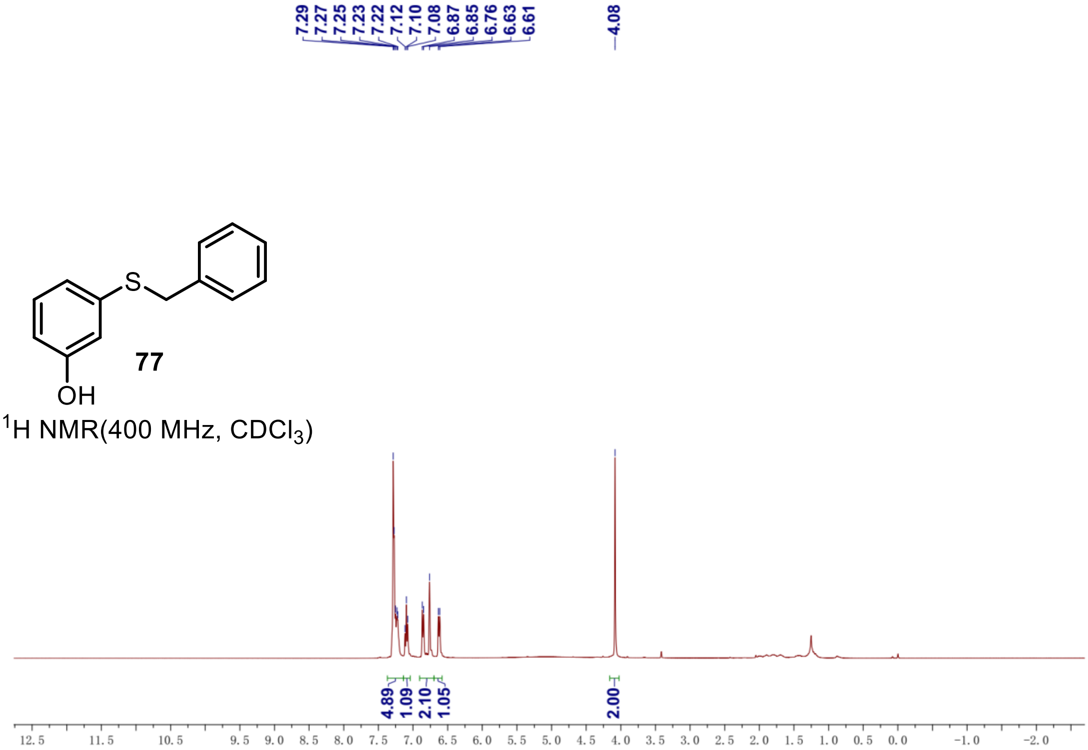
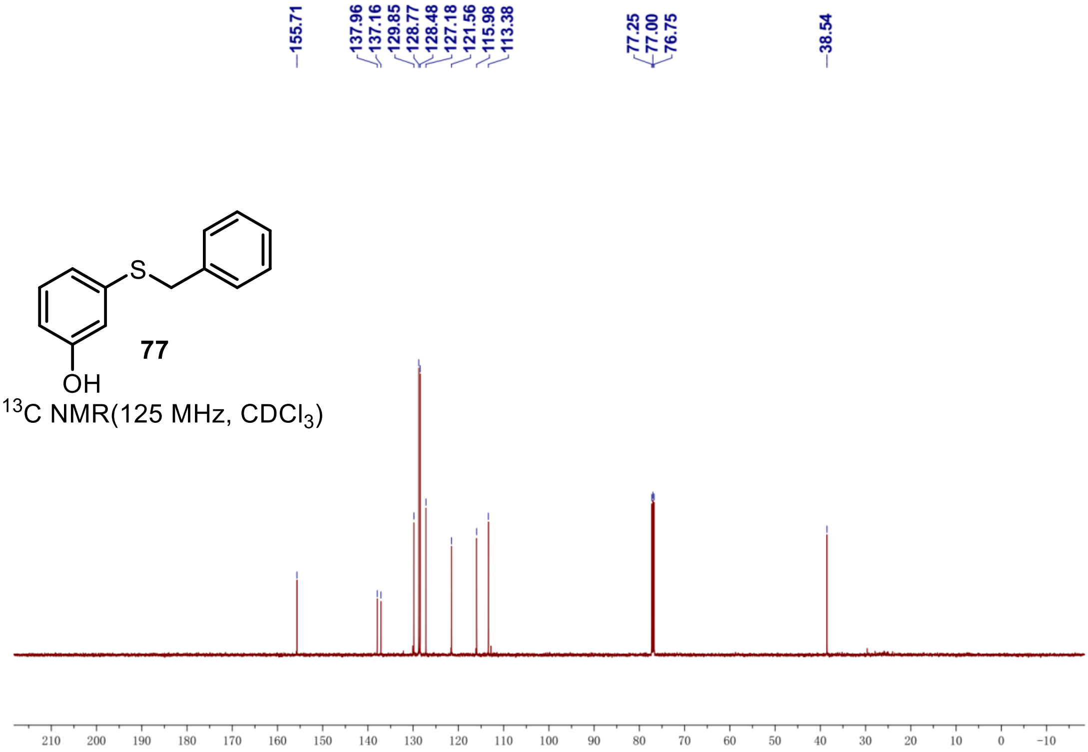




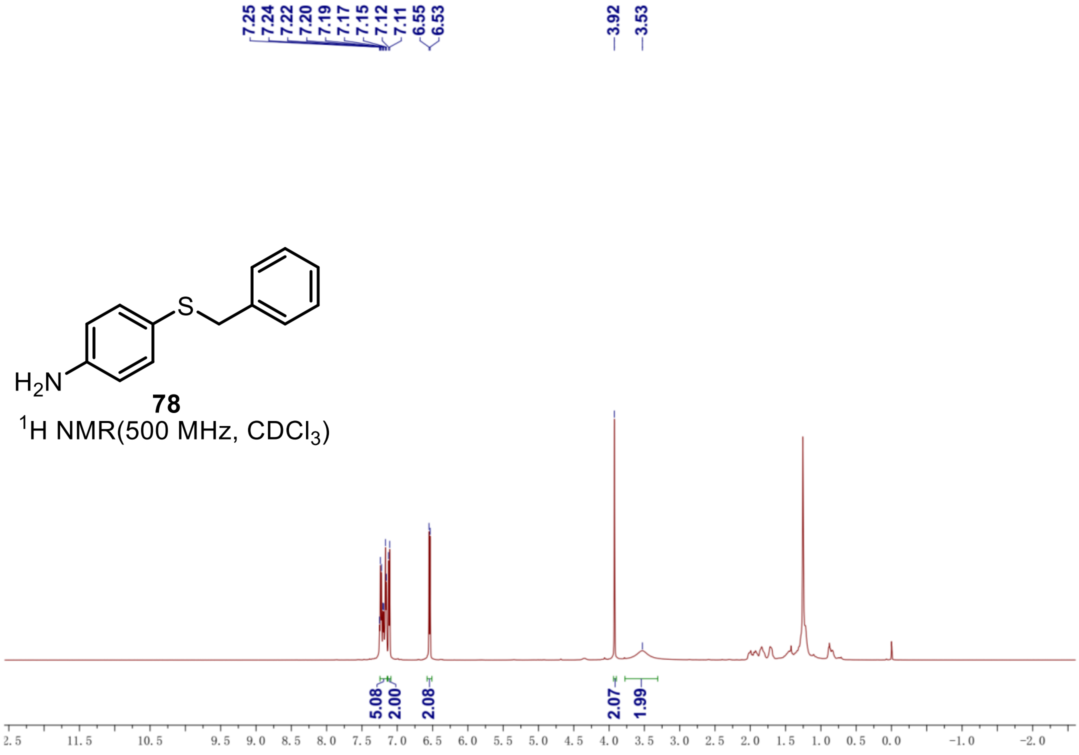
77

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



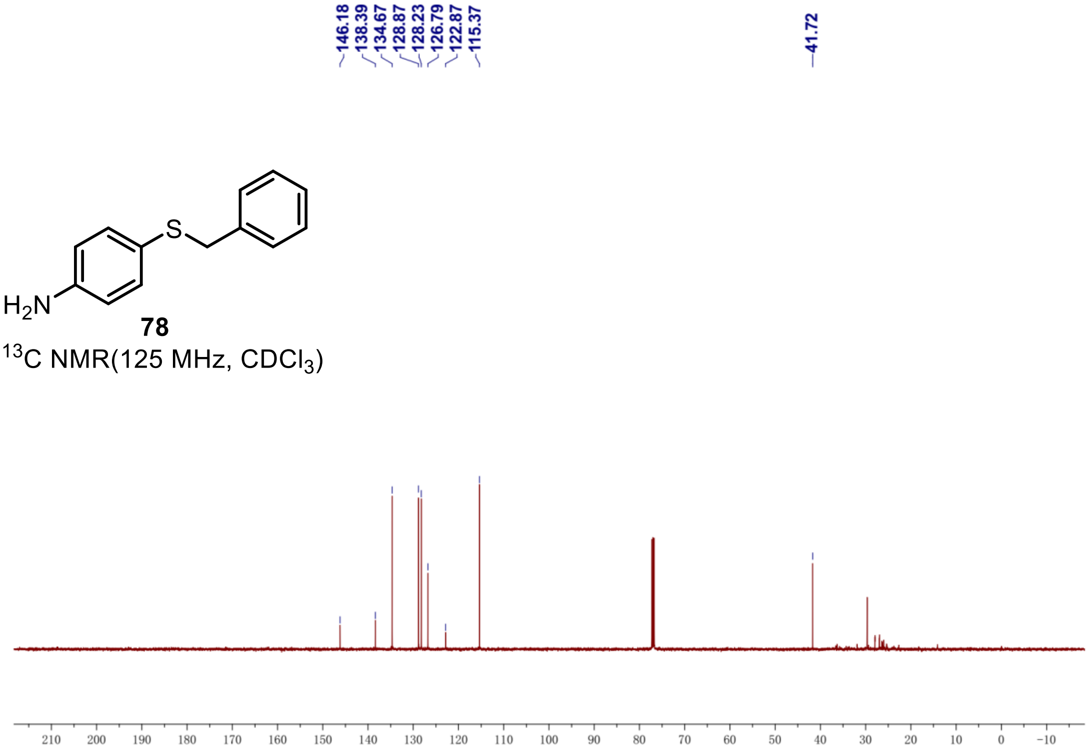
77

^{13}C NMR(125 MHz, $CDCl_3$)



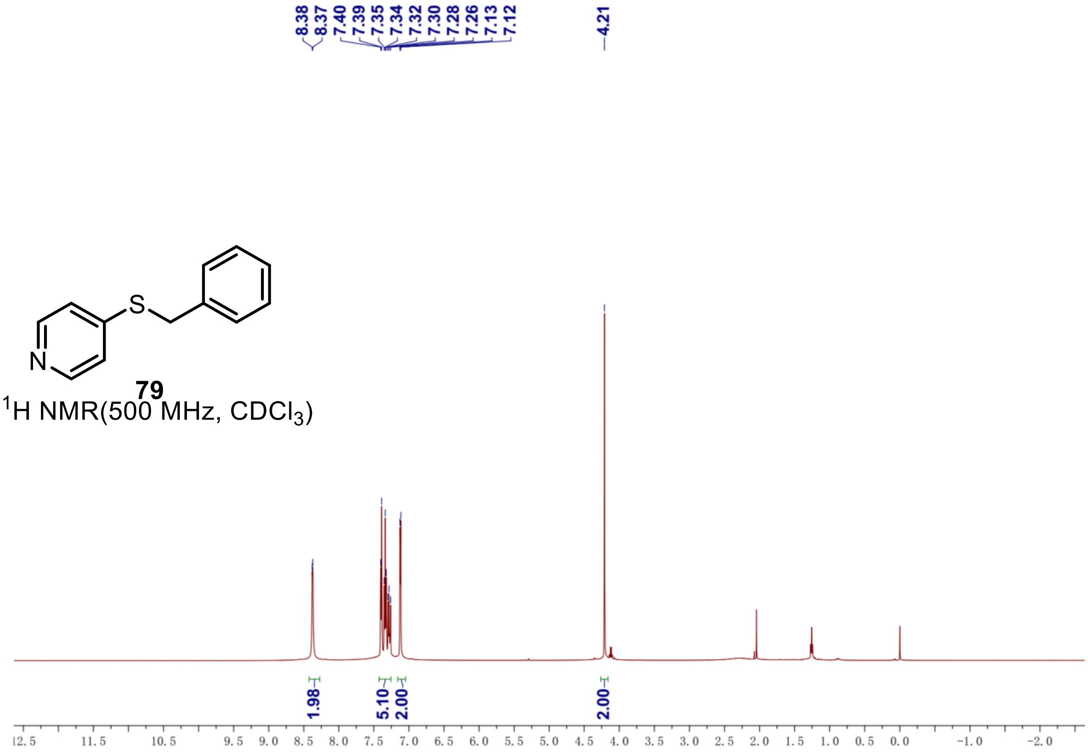
78

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



78

^{13}C NMR(125 MHz, $CDCl_3$)



79

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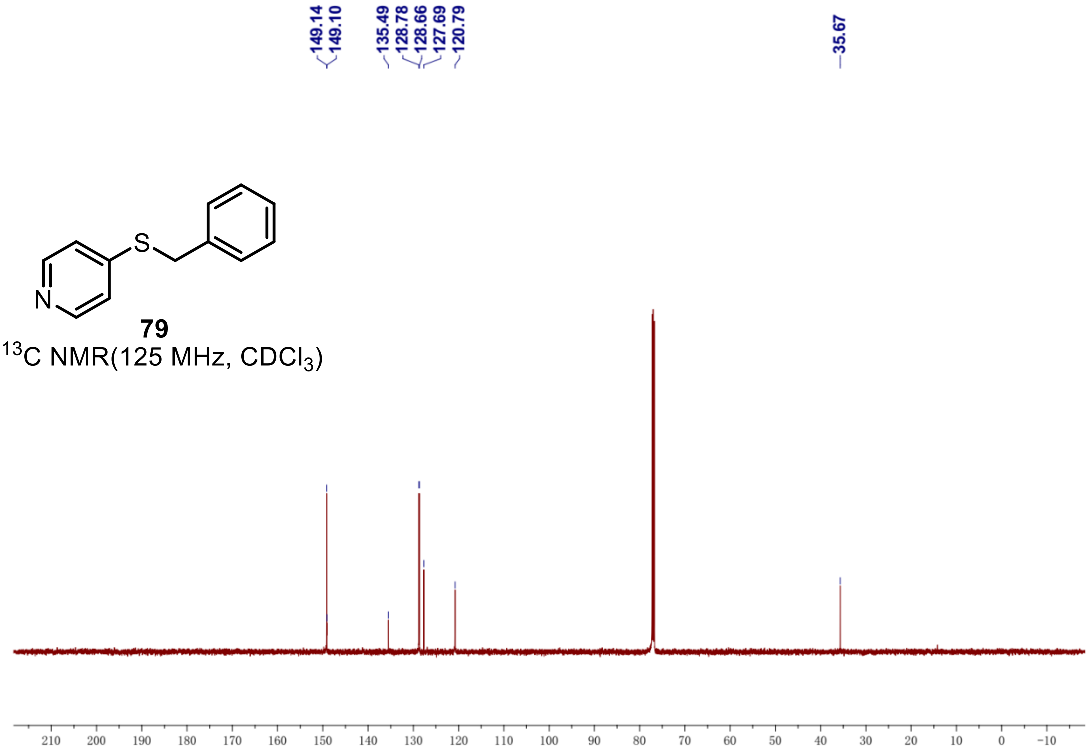

79

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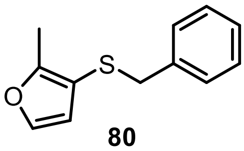
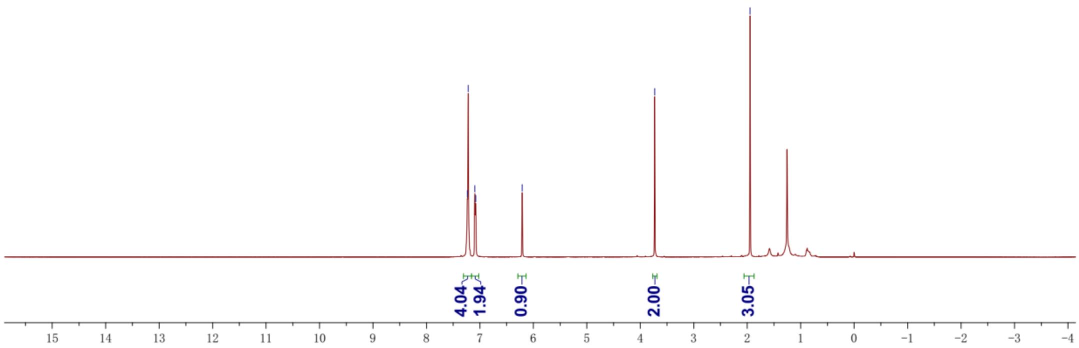
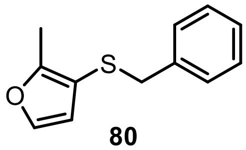


80

^{1}H NMR(400 MHz, CDCl$_3$)





80

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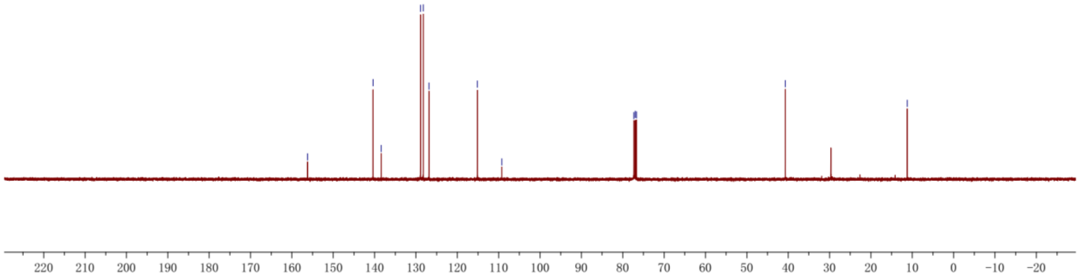

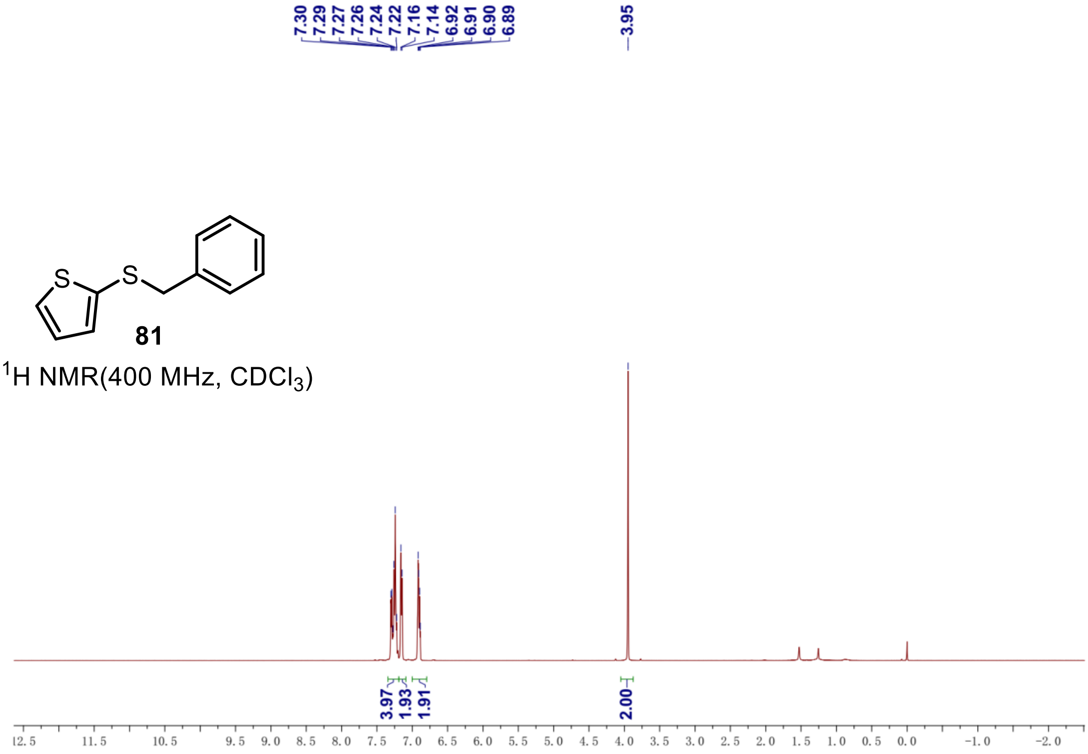

81

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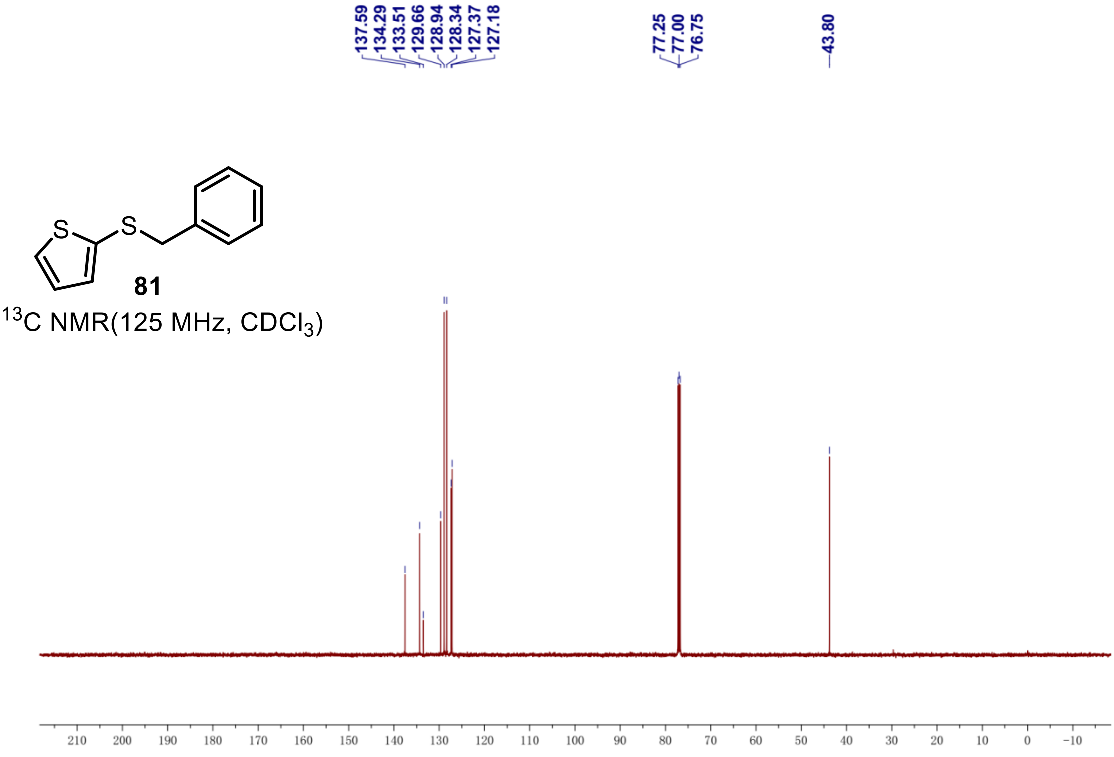

81

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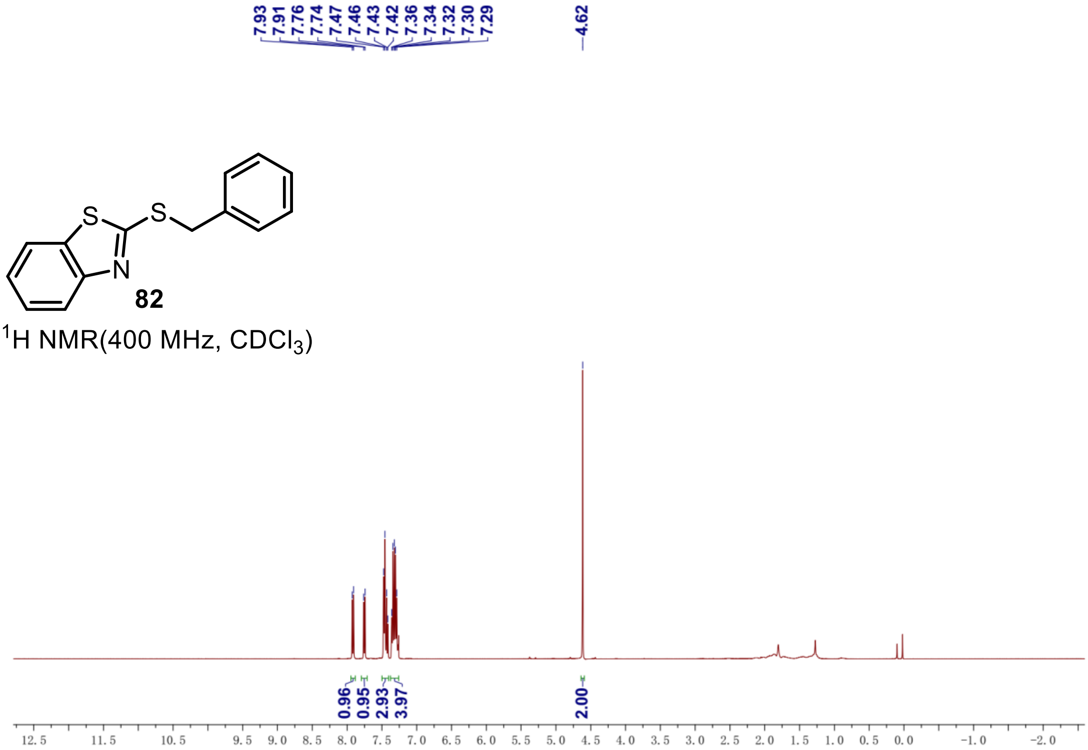

82

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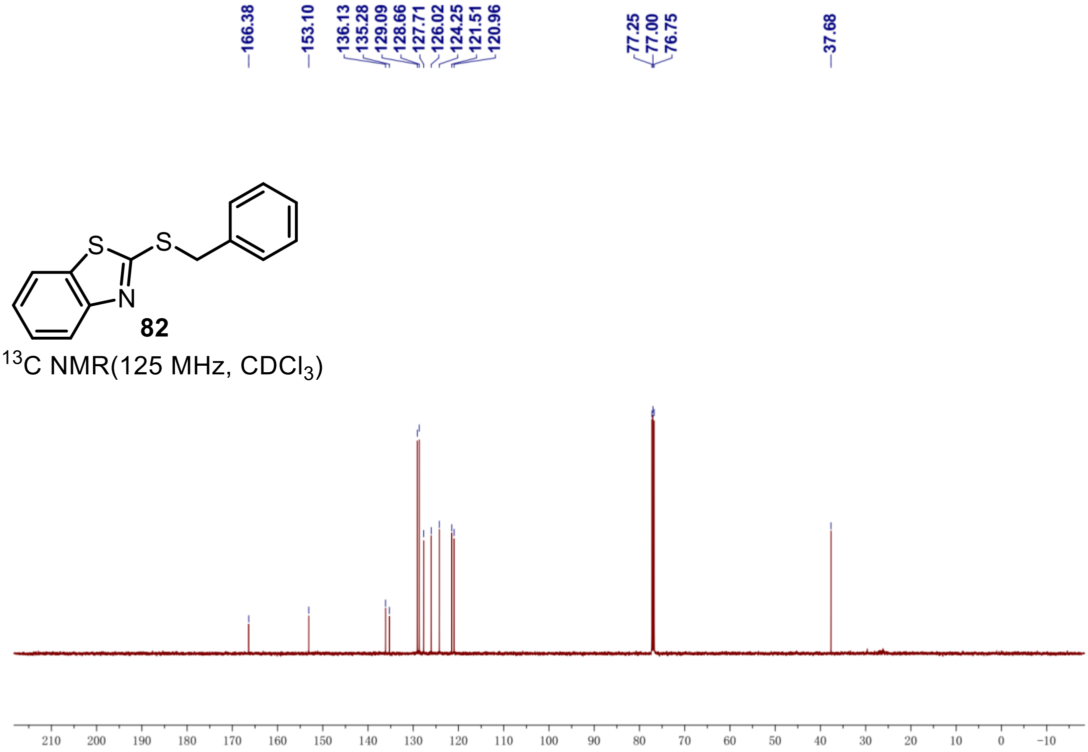

82

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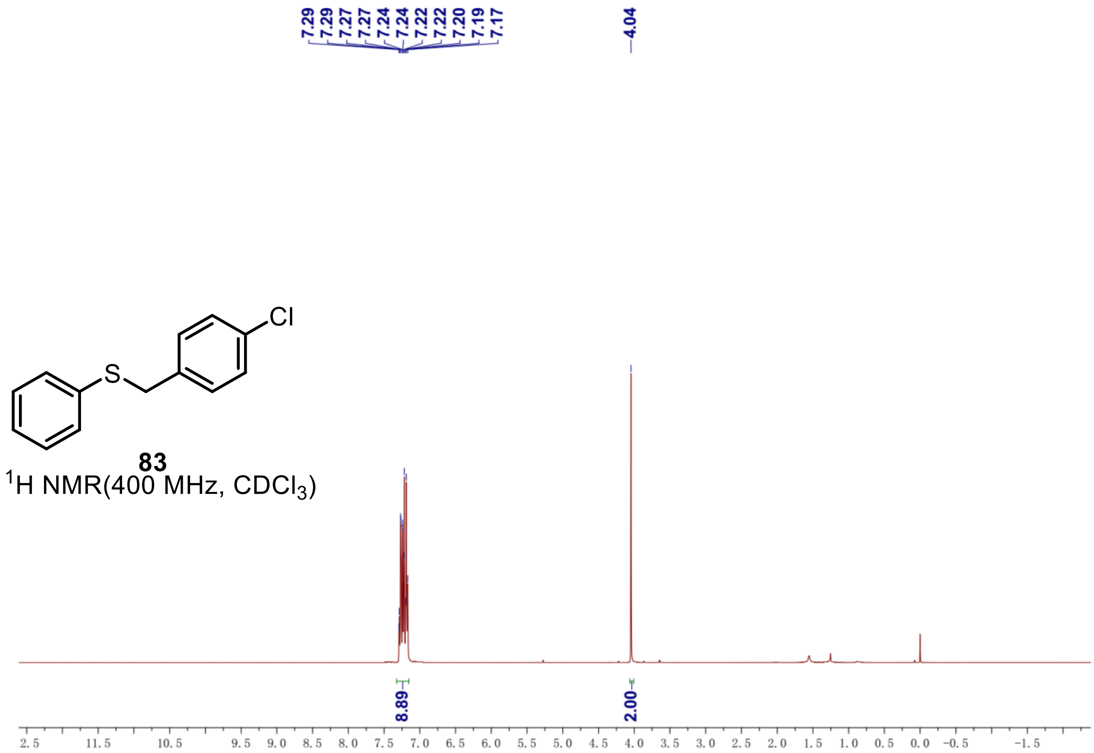

83

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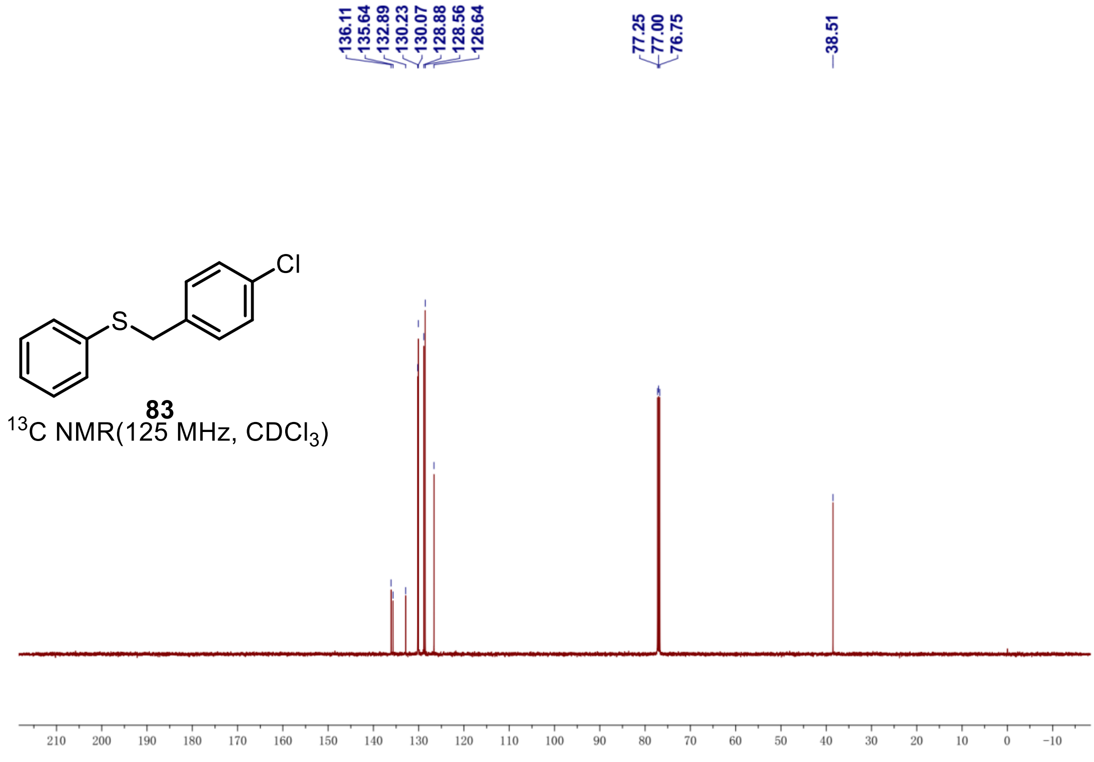

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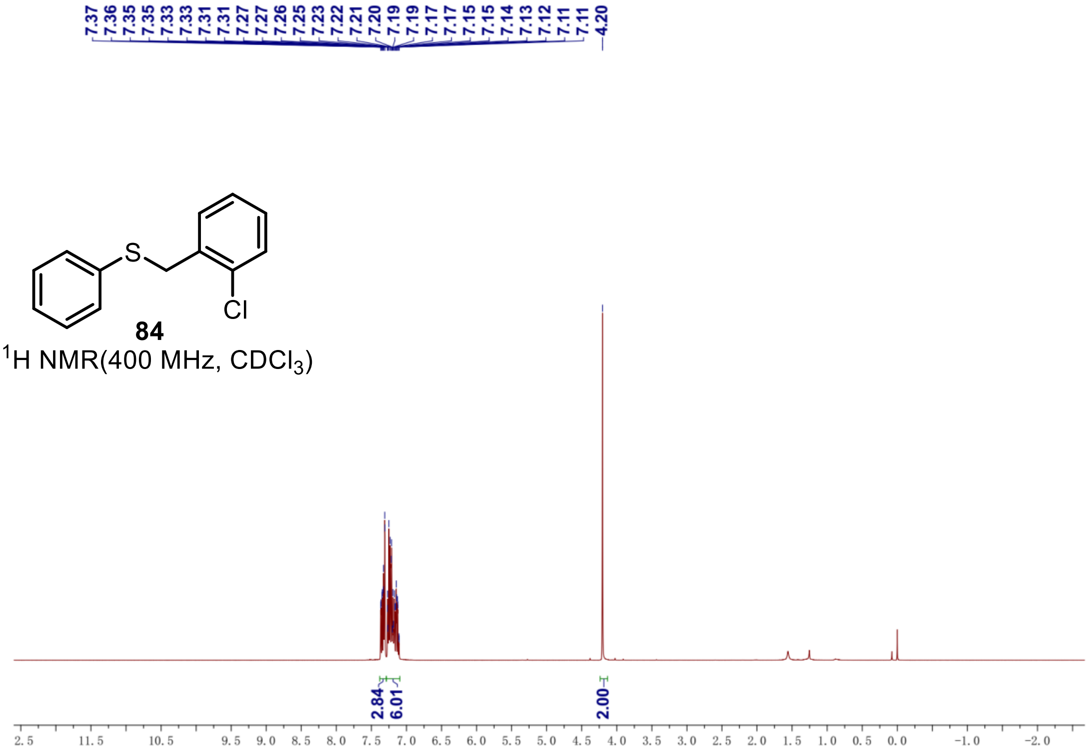

84

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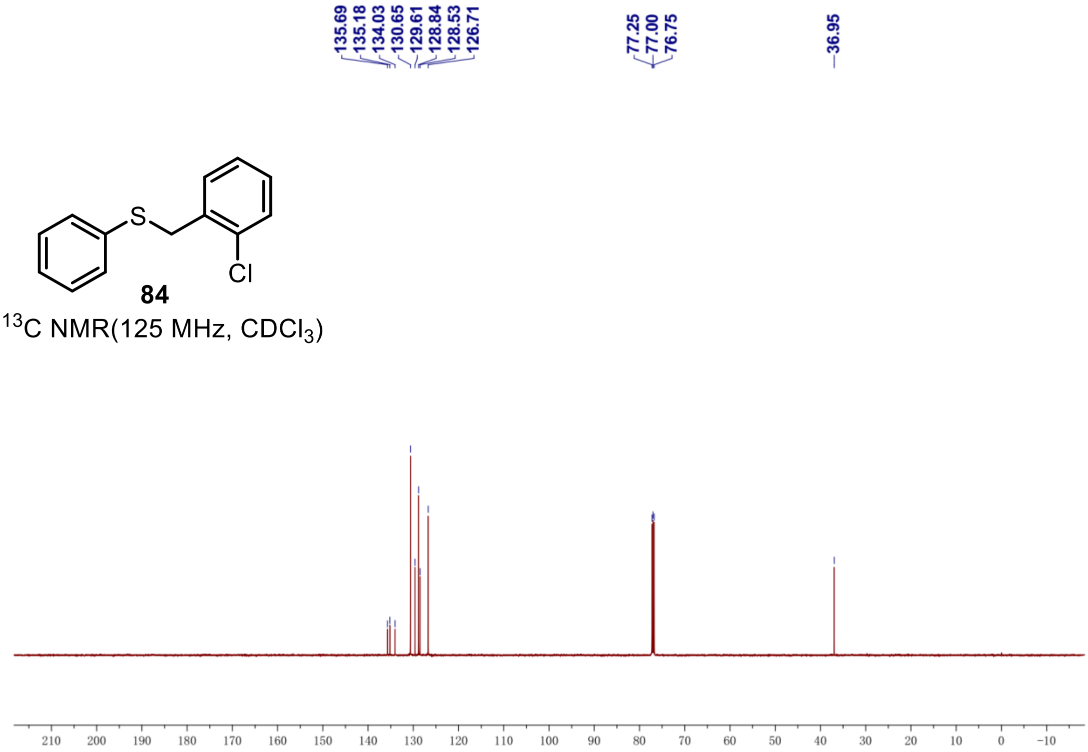

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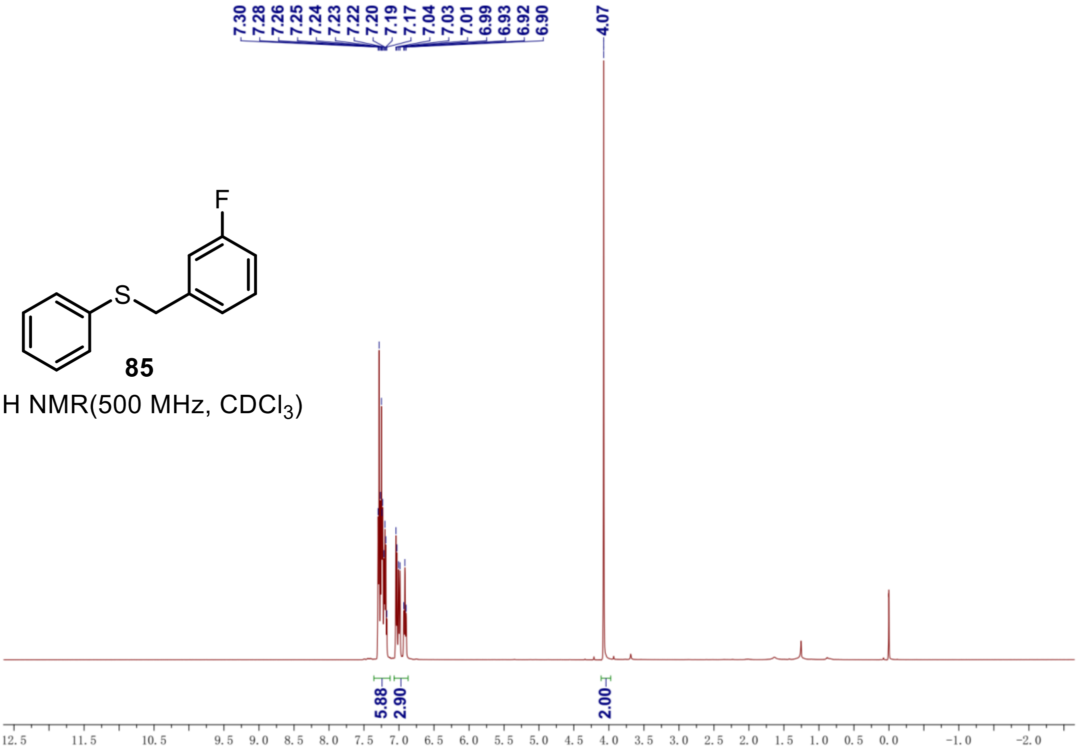

85

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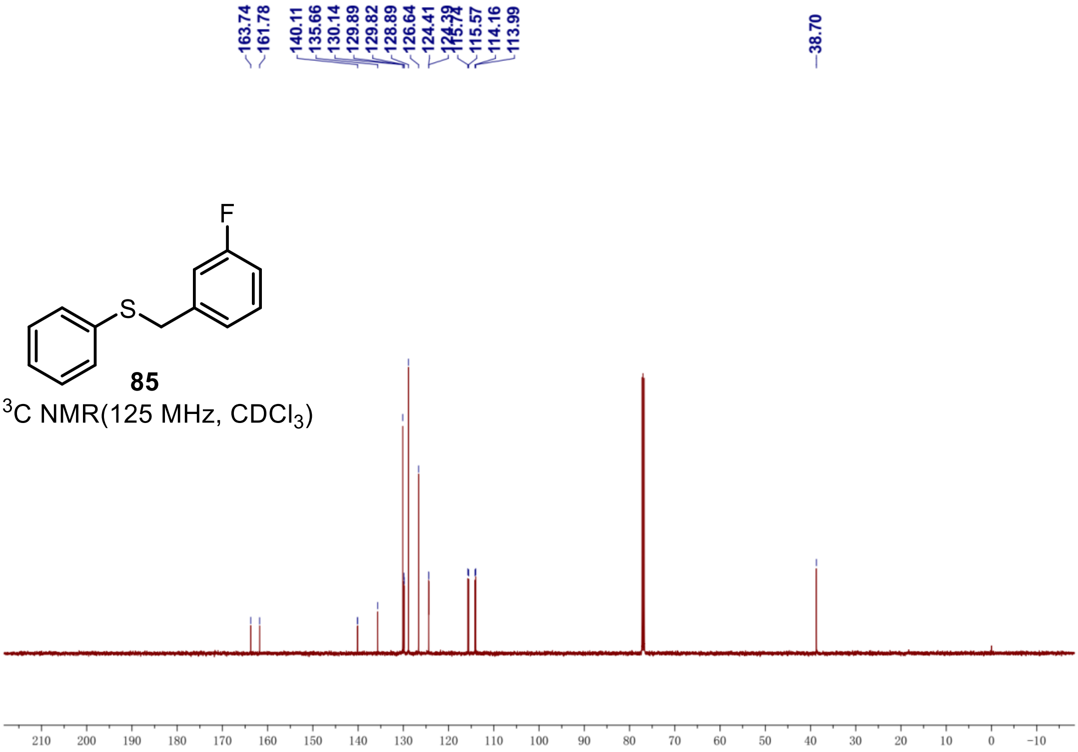

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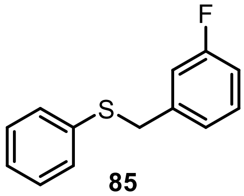
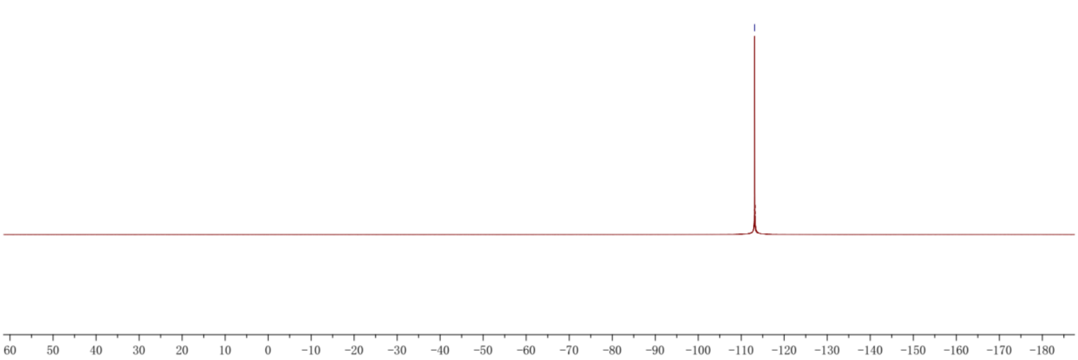
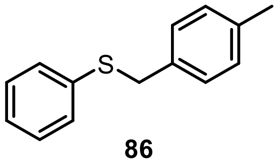


85

^{19}F NMR(471 MHz, CDCl$_3$)





86

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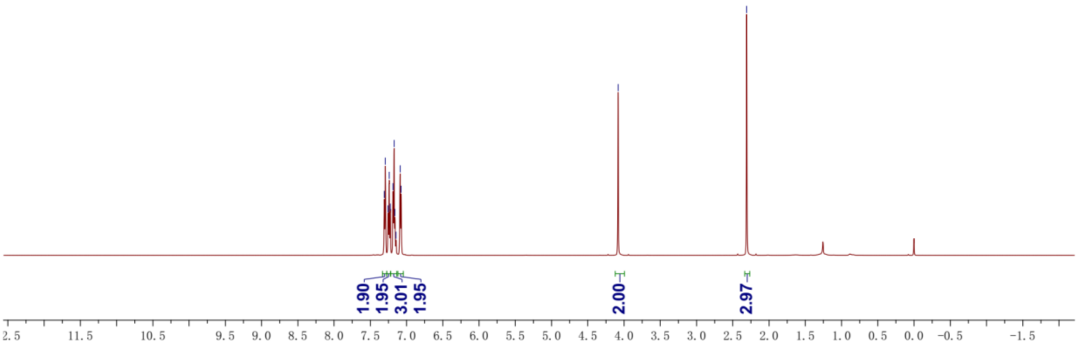

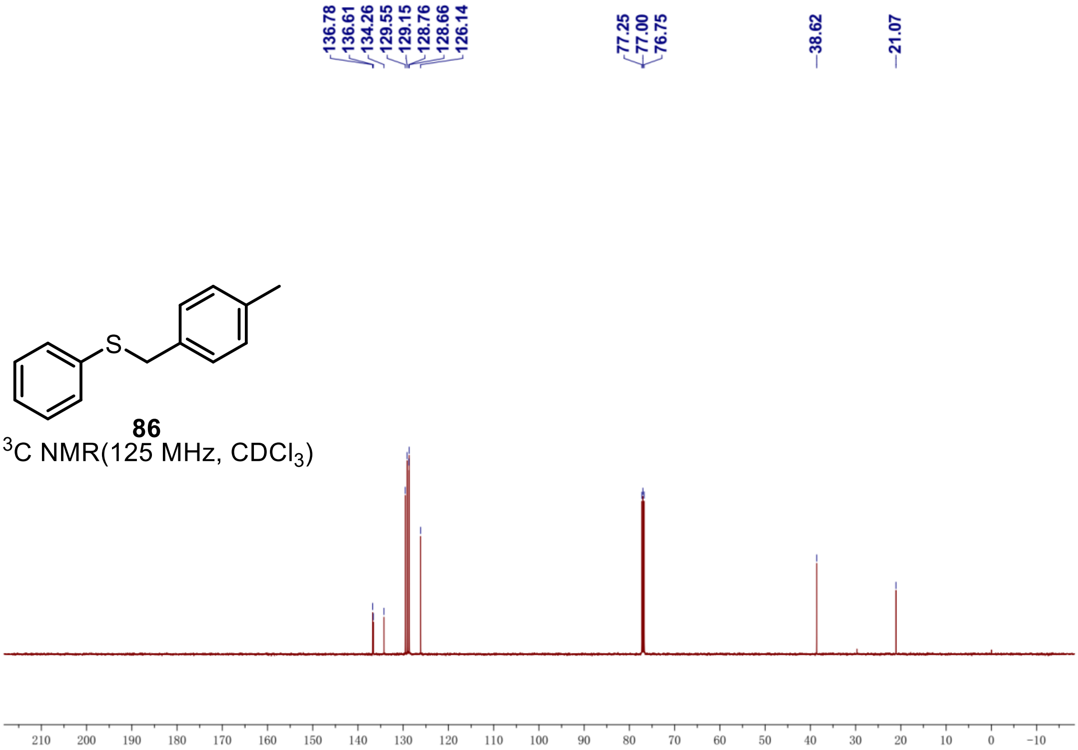

86

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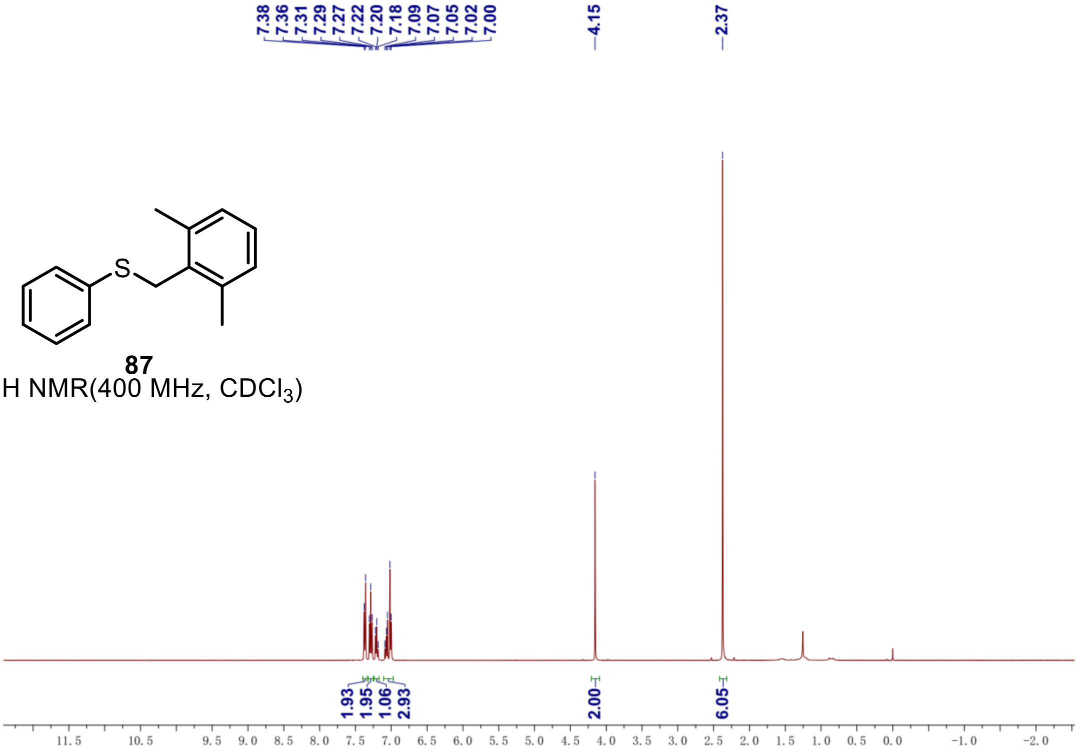

87

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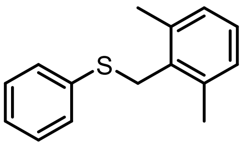

87

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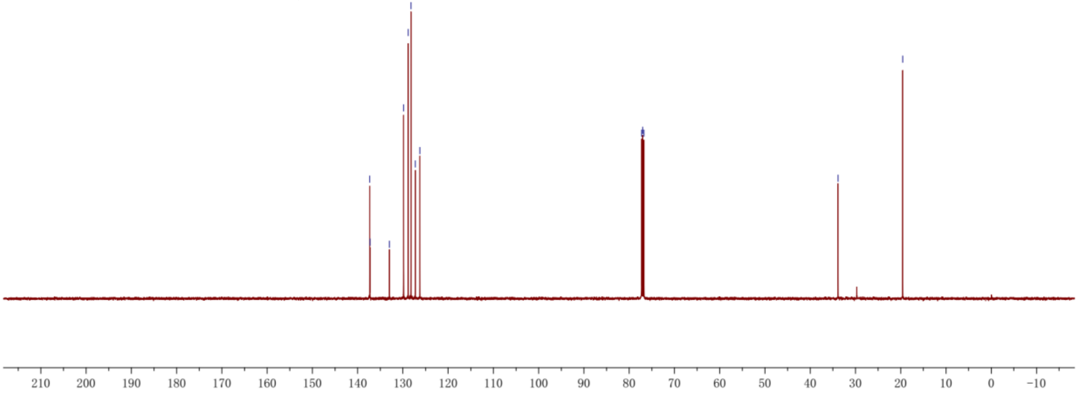
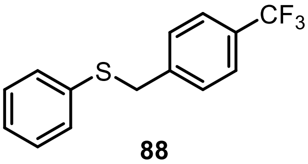
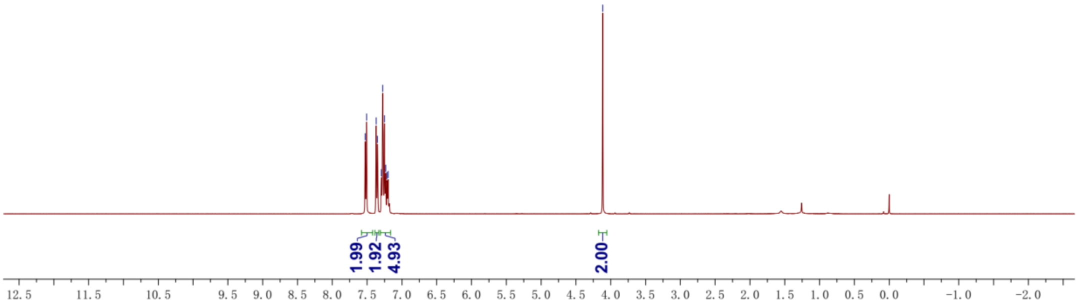
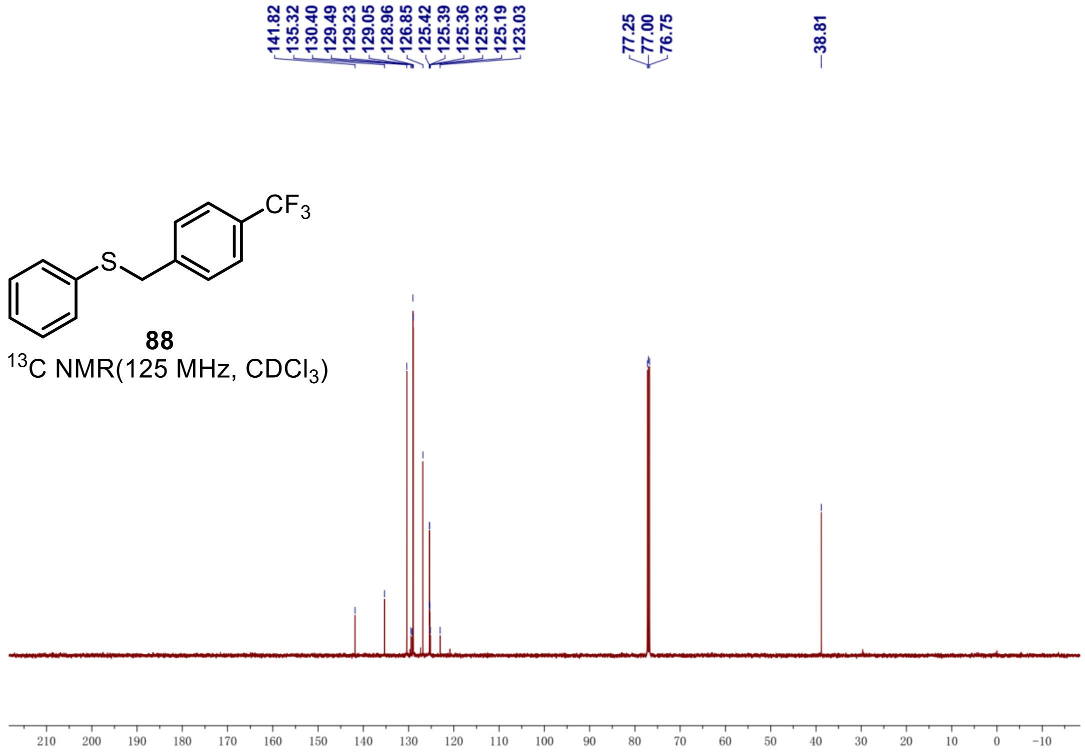




88

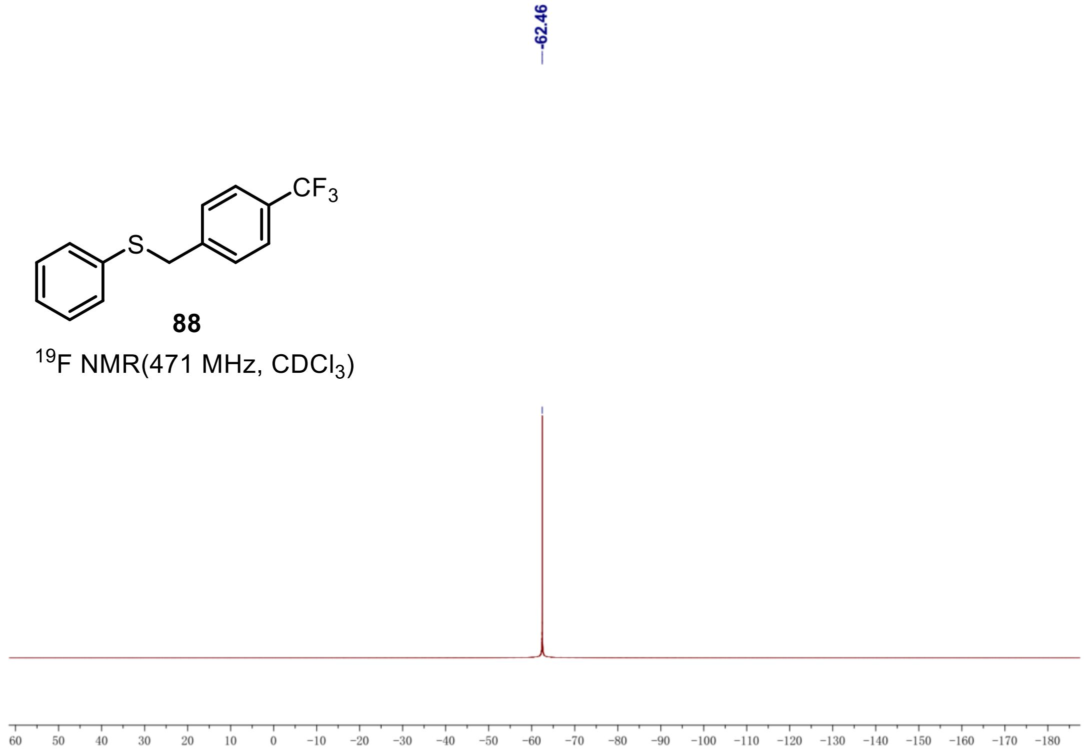
^{1}H NMR(400 MHz, CDCl$_3$)





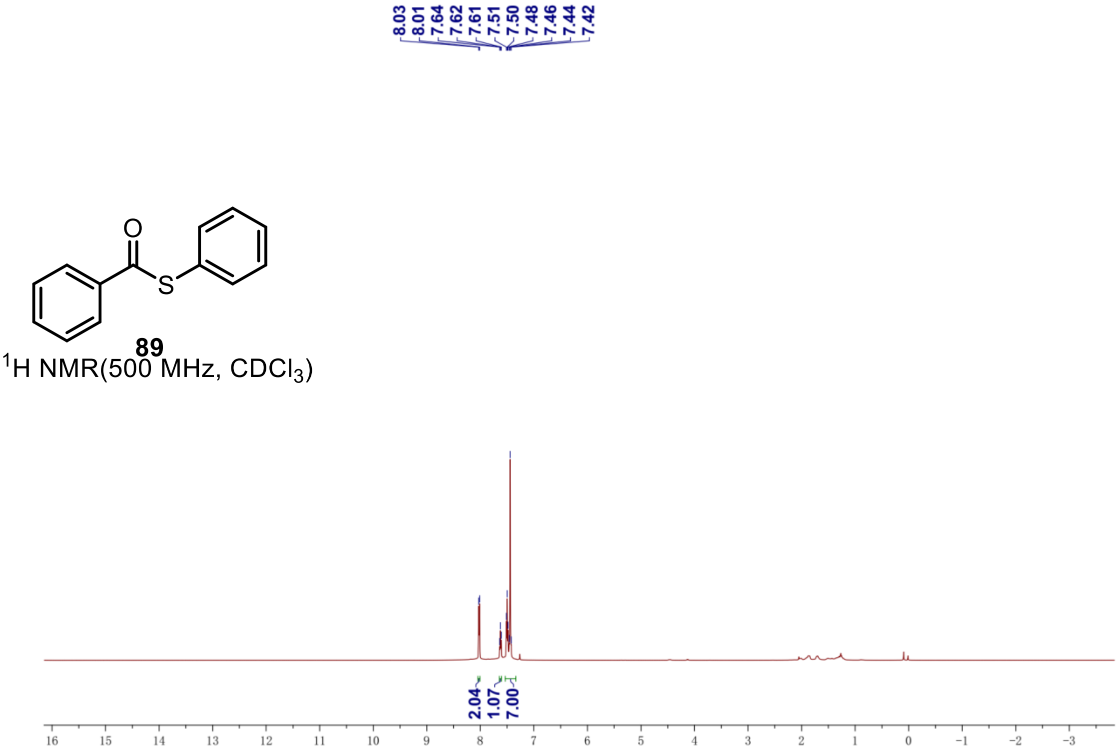
88

^{13}C NMR(125 MHz, CDCl$_3$)



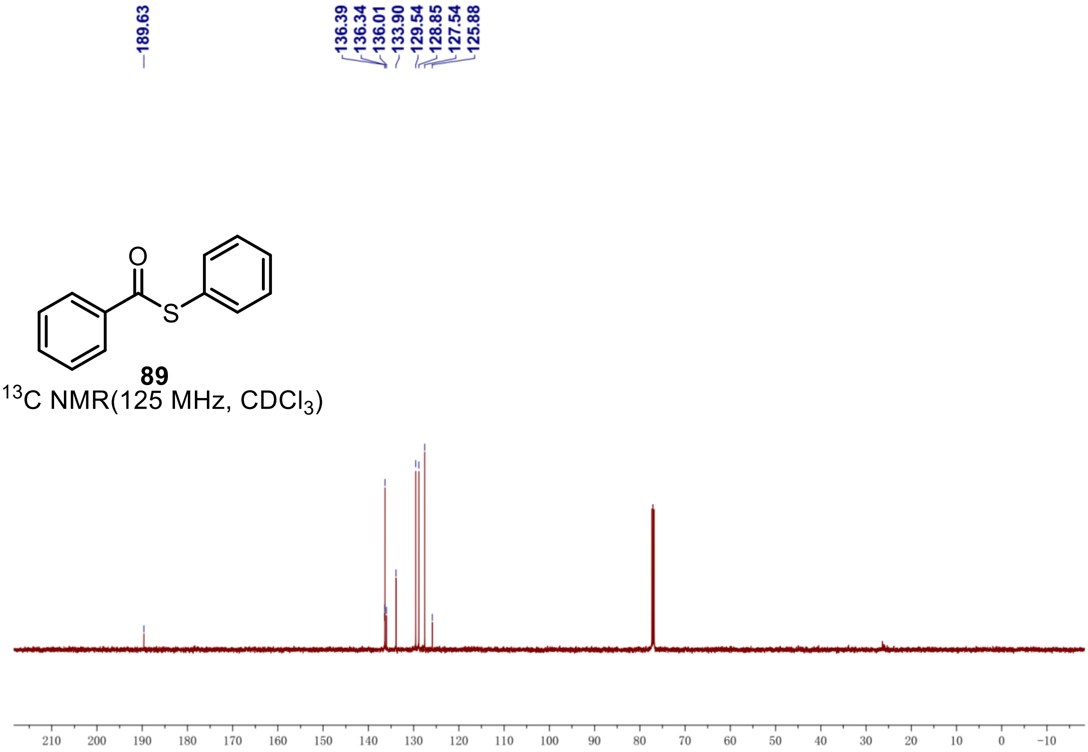
88

^{19}F NMR(471 MHz, CDCl$_3$)



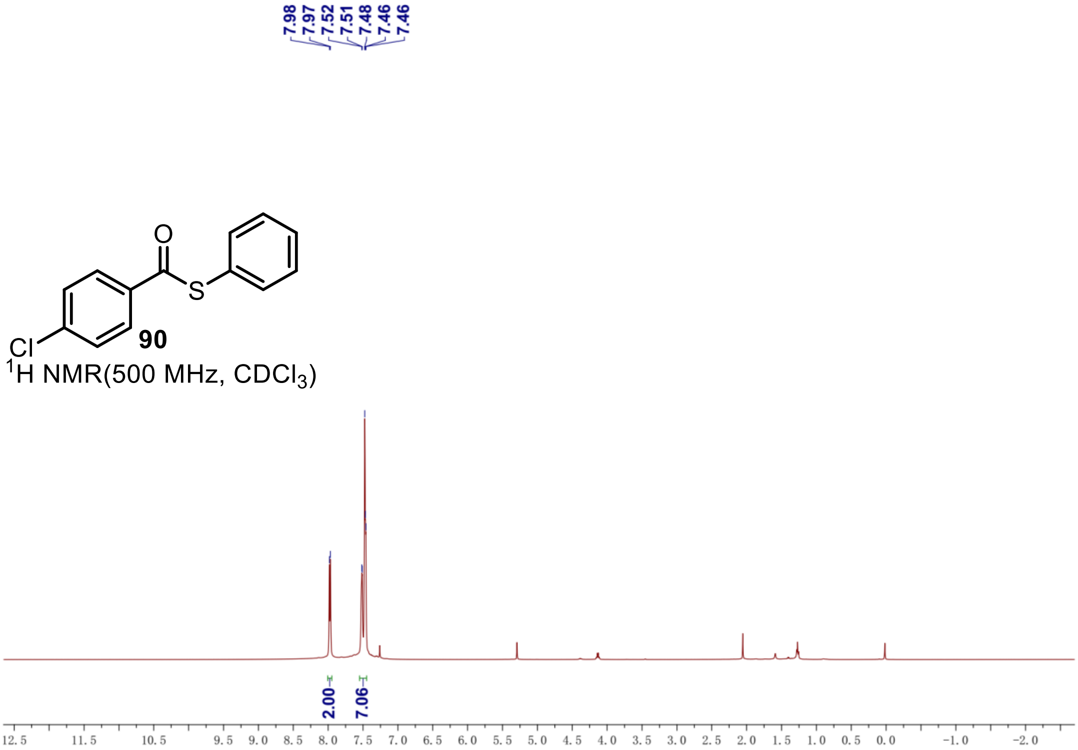
89

^{1}H NMR(500 MHz, CDCl$_3$)



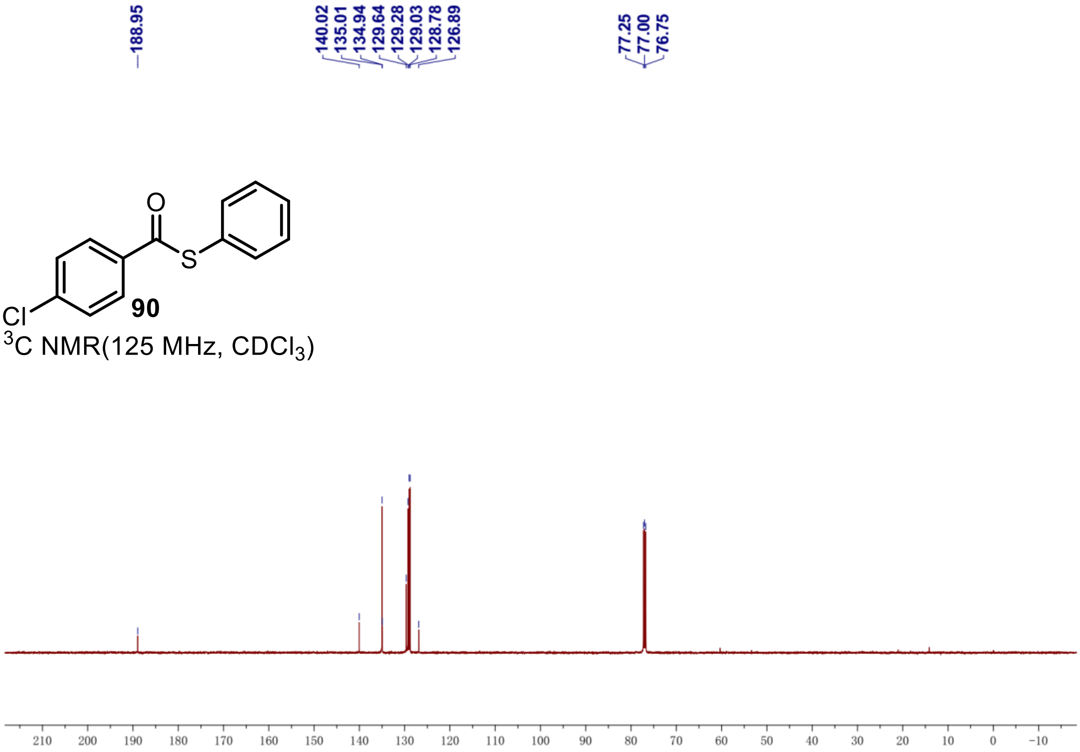
89

^{13}C NMR(125 MHz, CDCl$_3$)



90

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



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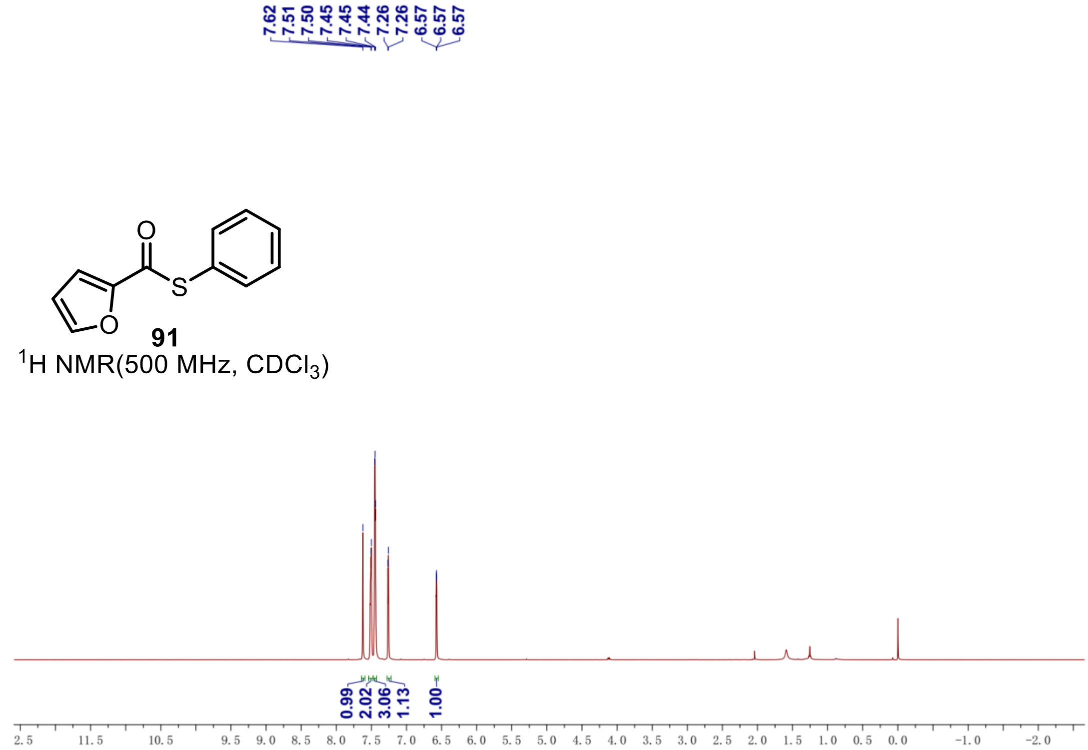
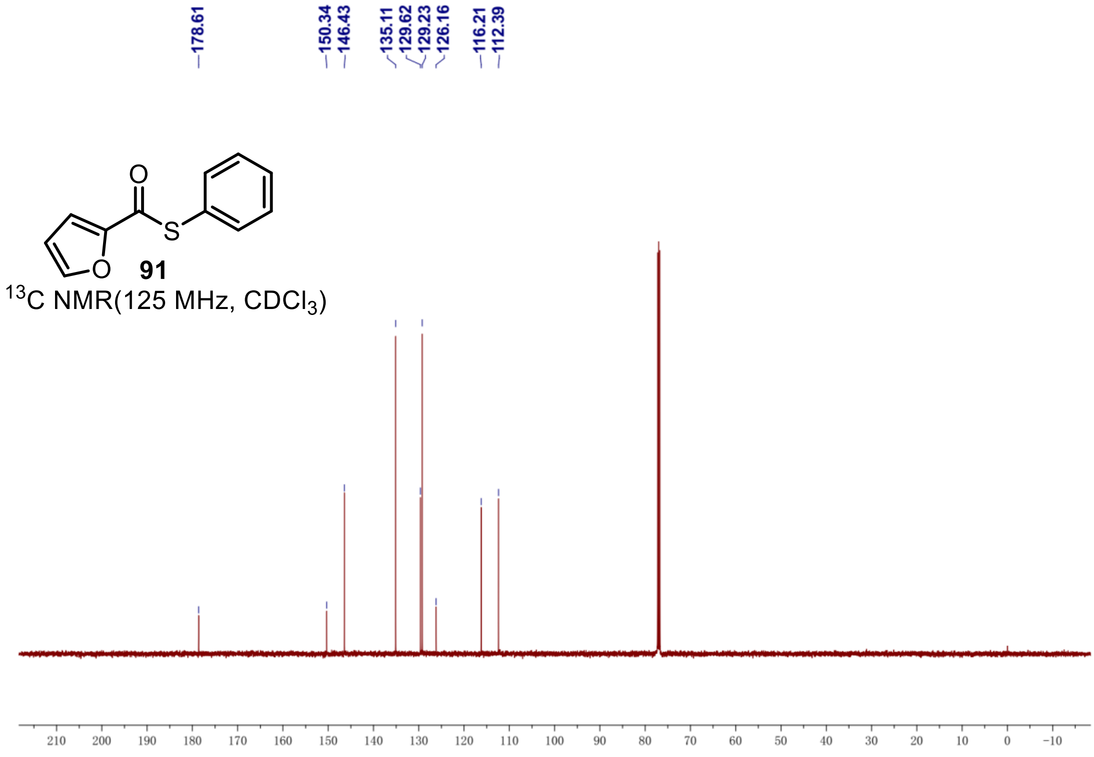


91

^{1}H NMR(500 MHz, CDCl$_3$)



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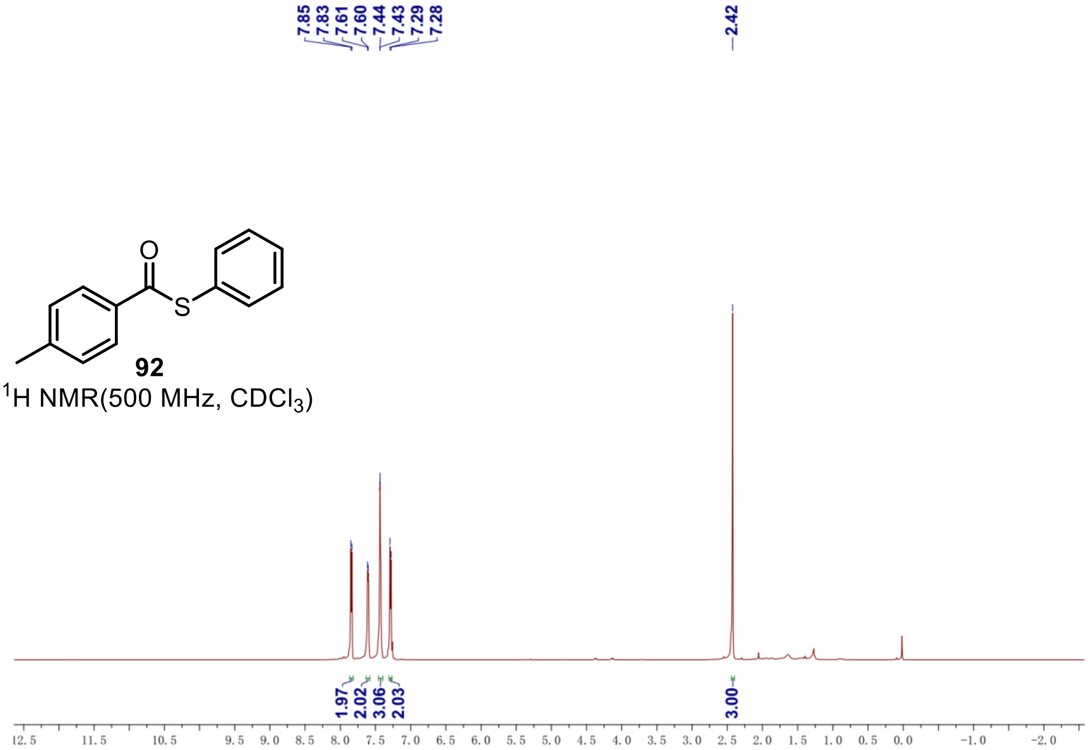
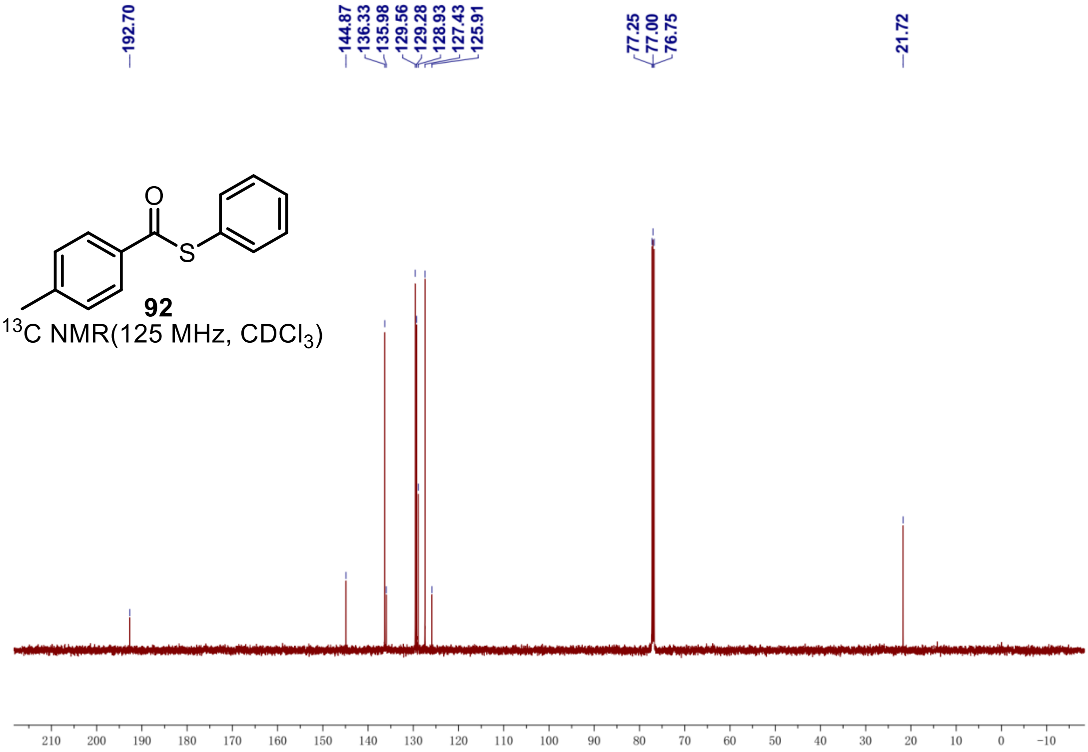

92
^{1}H NMR(500 MHz, $CDCl_3$)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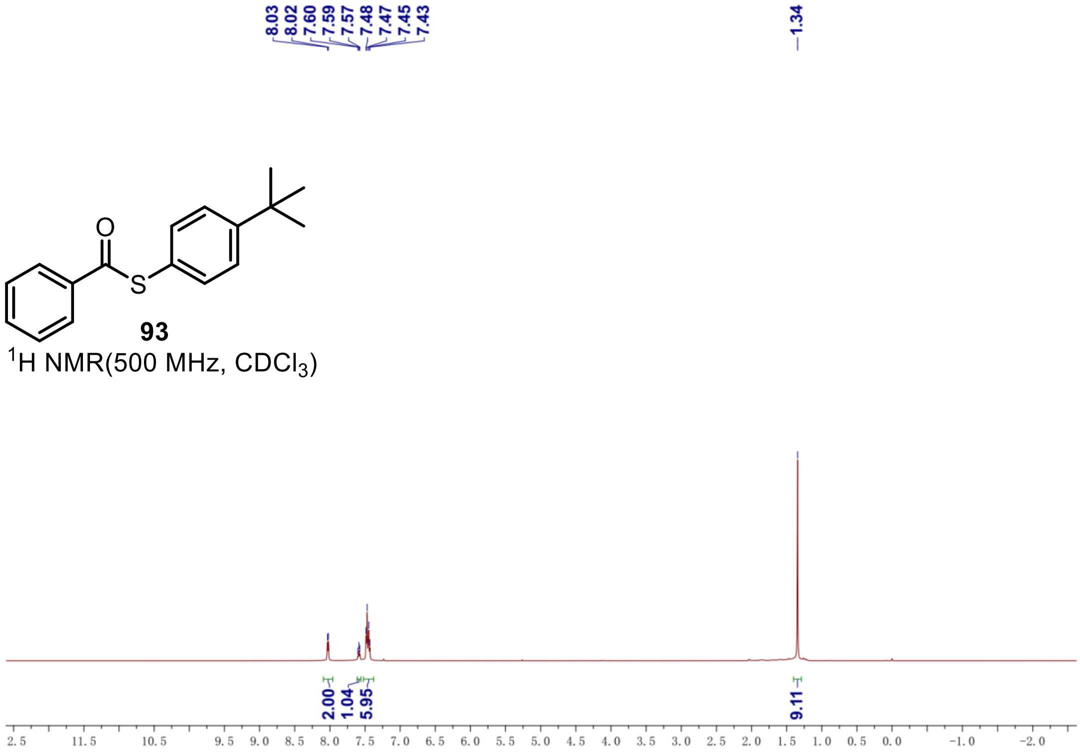
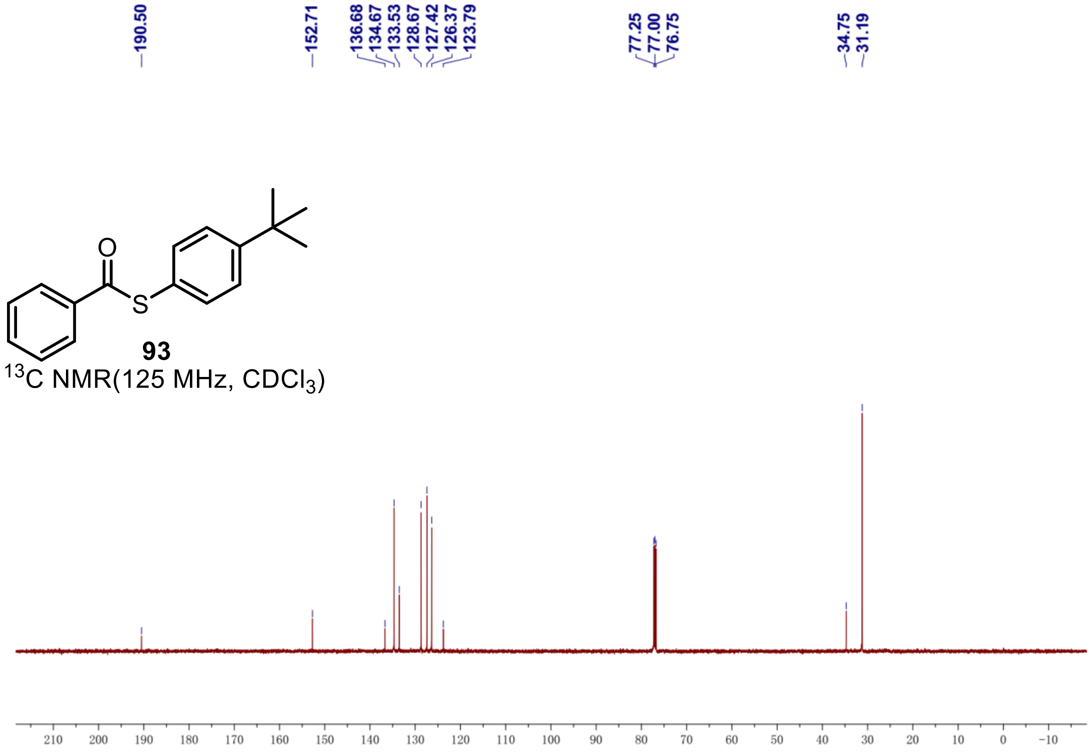


93

^{1}H NMR(500 MHz, CDCl$_3$)



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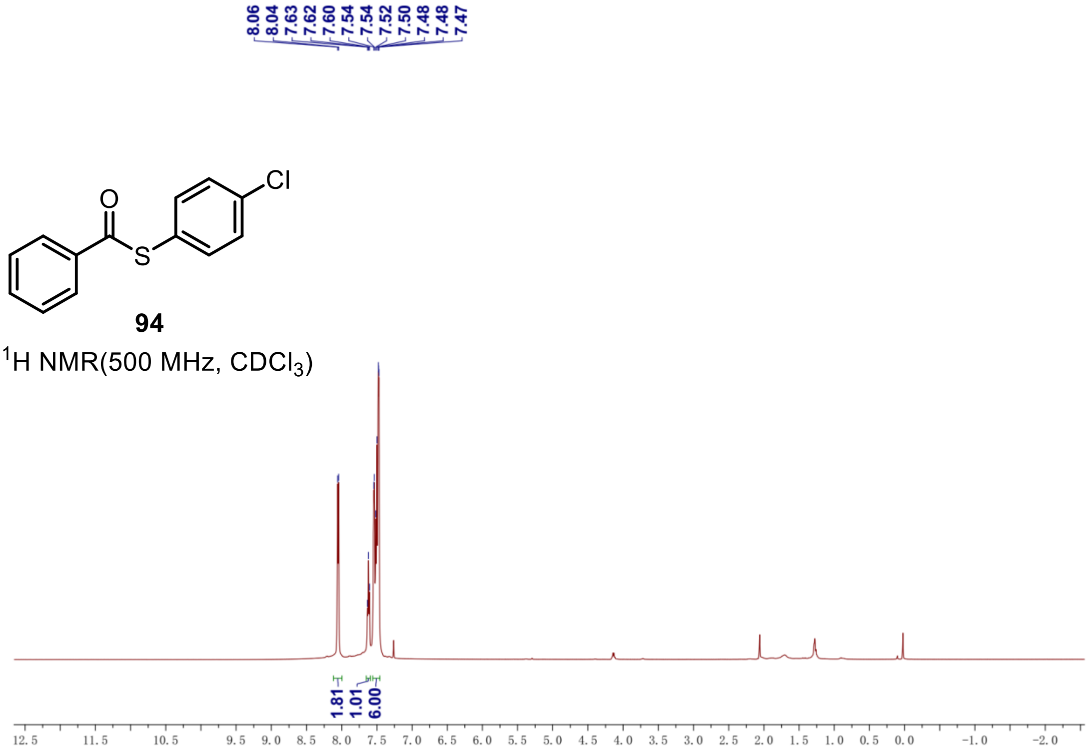

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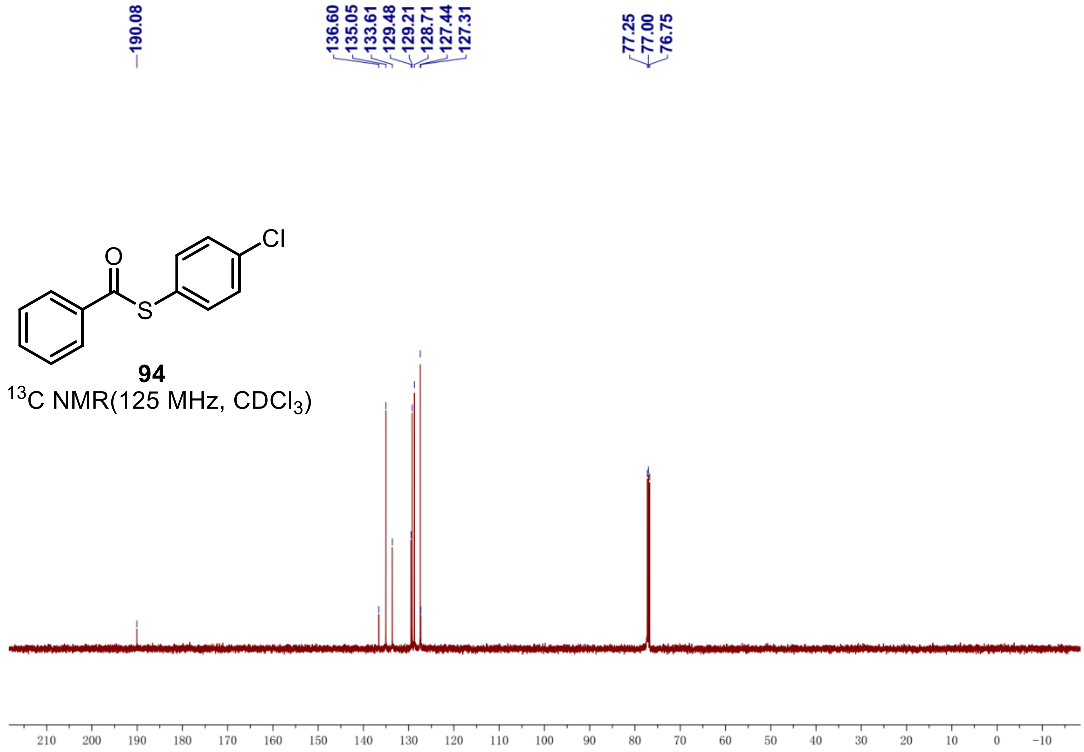
^{1}H NMR(500 MHz, $CDCl_3$)



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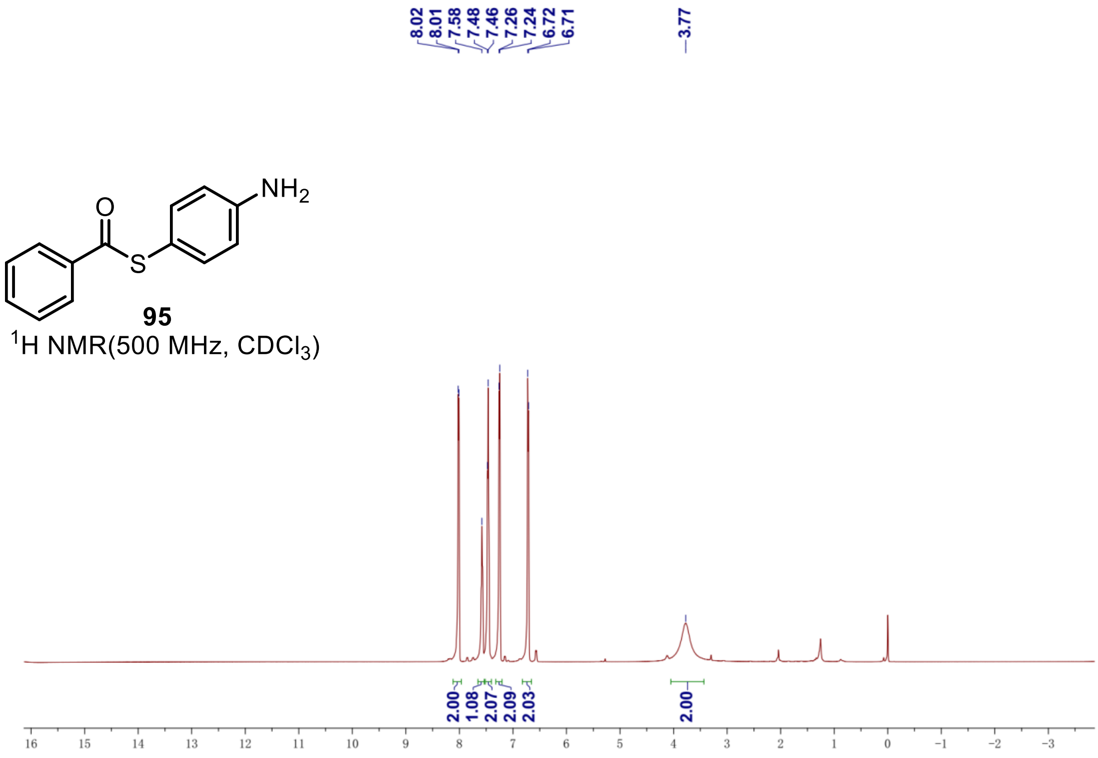
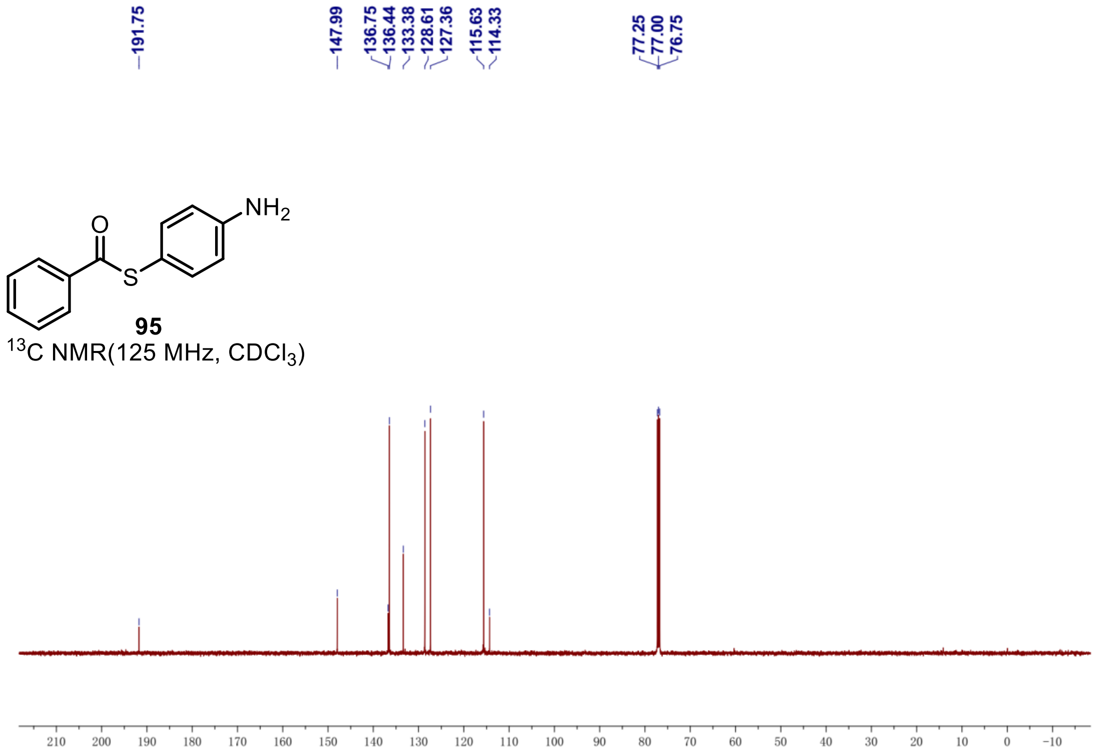
^{13}C NMR(125 MHz, $CDCl_3$)



95

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



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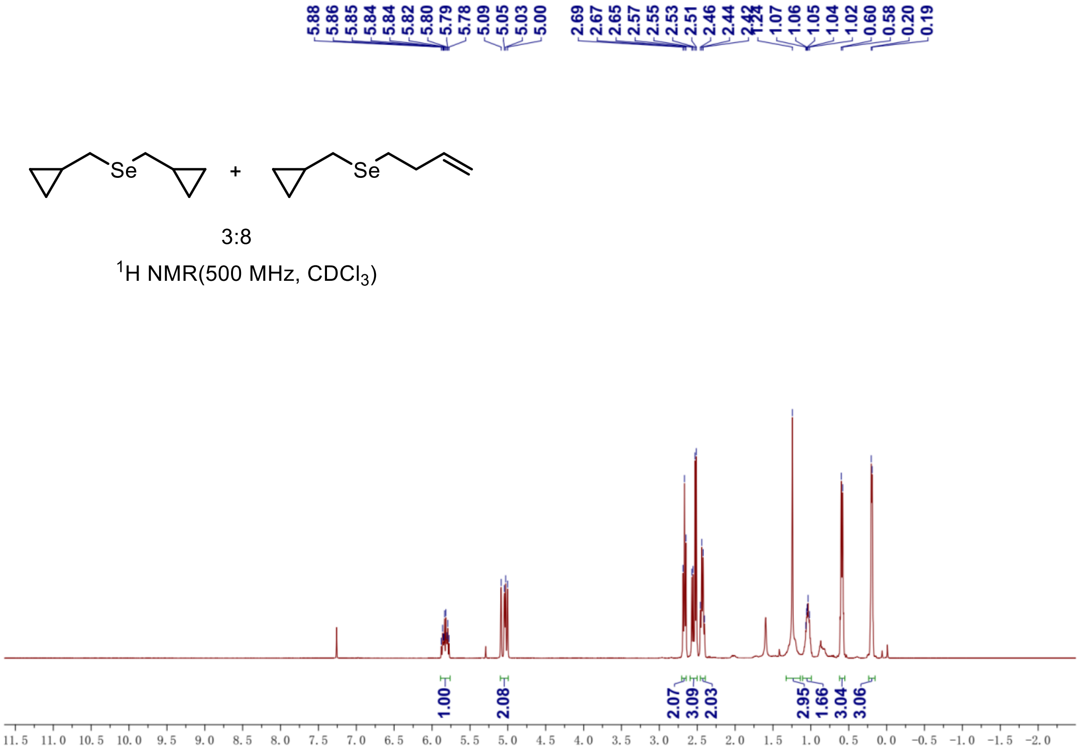
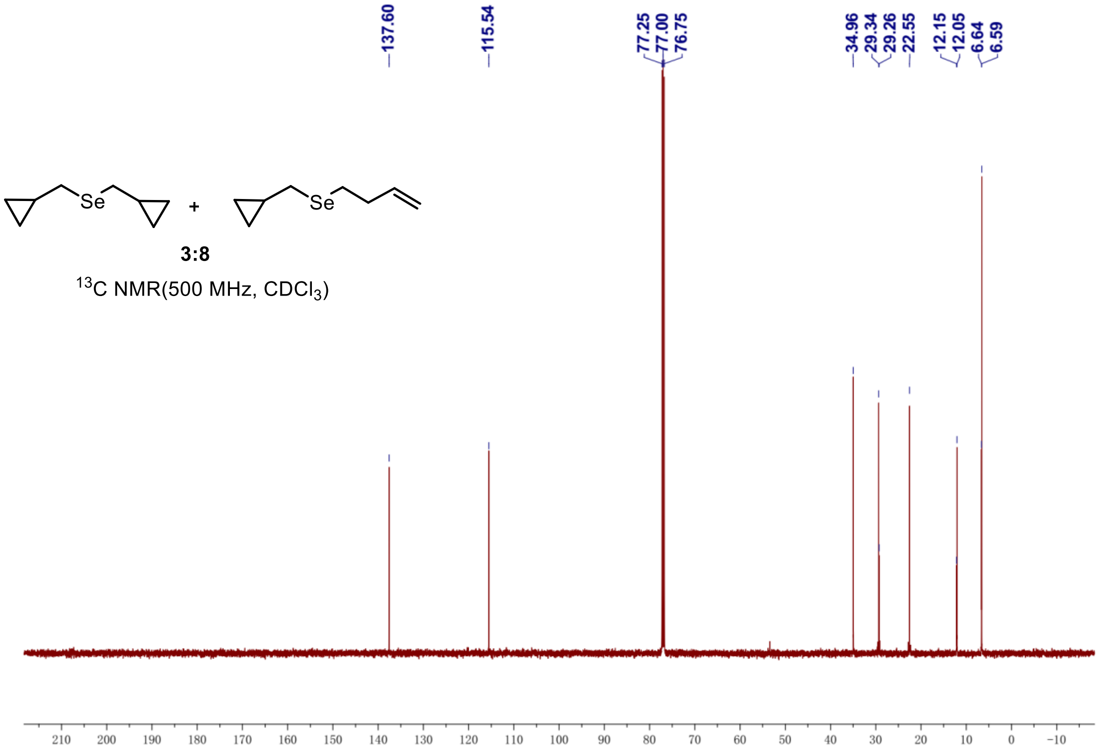


3:8

^{1}H NMR(500 MHz, CDCl$_3$)



3:8

^{13}C NMR(500 MHz, CDCl$_3$)

8. Reference

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