

Supporting Information

Visible-light-induced oxidative coupling of vinylarenes with diselenides leading to α -aryl and α -alkyl selenomethylketones

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1. General information

Solvents and reagents

Reagents were used as received without further purification unless otherwise indicated. Solvents were dried and distilled prior to use. Petroleum ether used had a boiling point range of 60–90°C. Diselenides were prepared from the corresponding iodides with elemental selenium according to Braga's report.¹

Chromatography

Chromatographic purification of products was performed as flash column chromatography on silica gel (200–300 meshes). Thin-layer chromatography (TLC) was carried out on silica plates (TLC Silica GF₂₅₄). Visualization of the compounds was accomplished by projecting UV-light onto the developed plates.

NMR spectra

NMR spectra were recorded on a Bruker Avance- III HD (¹H NMR: 400 MHz, ¹³C NMR: 100 MHz) spectrometer. Chemical shifts are referenced to residual solvent signals (CDCl₃: 7.26 ppm and 77.16 ppm for ¹H NMR and ¹³C NMR respectively) and reported in parts per million (ppm) relative to tetramethylsilane (TMS). Spin–spin coupling constants (*J*) were given in Hz. Multiplicities of NMR signals are abbreviated as follows: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet.

Mass spectra

High resolution mass spectrometry (HRMS) analyses were carried out on a Thermo Fisher Q Exactive Mass Spectrometer.

Melting points

Melting points were determined on glass slides using a WRX-4 digital display microscopic melting point apparatus and were presented uncorrected.

Optical rotations

Optical rotations were determined using a WZZ-2S polarimeter with a sodium lamp (D-line, $\lambda = 589$ nm). $[\alpha]_{\text{temp}}^{\text{D}}$ -Values are reported at a certain temperature (temp) and all concentrations are given in g/100 mL.

2. Light sources, glassware and setup for irradiations

Light sources

All photoreactions were performed using a 24 W energy-saving household CFL bulb (cool daylight, 6500 K) by Opplé. Please refer to the website (https://detail.tmall.com/item.htm?id=36296589905&spm=a1z09.2.0.0.289f2e8dLvLPLS&_u=nkj7u3r52c7) for more detail. The emission spectrum of such a lamp was recorded and is presented in Figure S5a.

Glassware

All reactions were performed in 10 mL vials made of Synthware. For detailed technical information, the reader is directed to the homepage of Synthware: <http://www.xinweier.com/>.

Setup for irradiations

All photoreactions were performed with the 24W energy-saving CFL-bulb introduced above. The reaction vessel was placed approximately 1 cm from the light source. A typical reaction setup is shown in Figure S1. A fan placed next to the photoreactor was used to keep the temperature constant during the reaction time (about 24 °C).

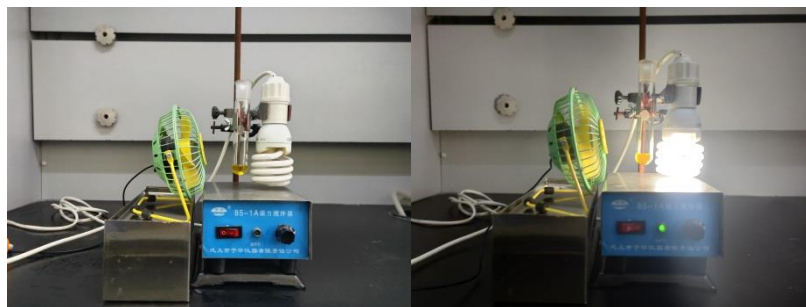


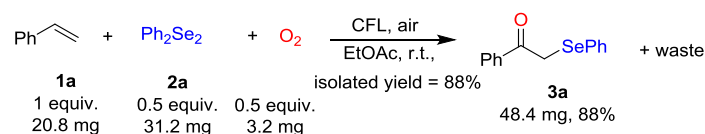
Figure S1: Setup for irradiations with a 24 W CFL

3. Green chemistry metrics

(1) E-Factor analysis

According to its original definition (*Green Chem.* **2007**, 9, 1273), the Sheldon E factor value (mass of waste/mass of product) takes into account only the mass of waste generated in a process, and its calculation is performed by simply dividing the sum of the molecular weight of all substances produced by molecular weight of the desired products, with reference to the stoichiometric equation. Thus, the amount of silica gel and the mass of solvent used for work-up and chromatography are usually not included in the calculation. We have followed this trend in our own calculation.

This work:



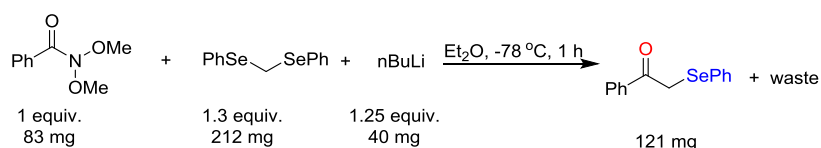
Total amount of reactants: 20.8+ 31.2+3.2 = 55.2mg

Amount of final product: 48.4 mg

Amount of waste: 55.2–48.4 = 6.8mg

E-Factor = Amount of waste/Amount of product =6.8 /48.4 = 0.14

Pace's work (*Org. Lett.* **2018** 2685-2688):



Total amount of reactants: 83+212+40 =335 mg

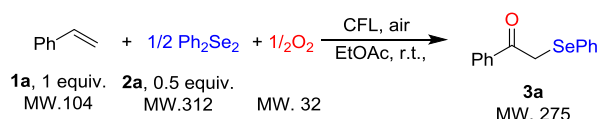
Amount of final product: 121 mg

Amount of waste: 335–121 = 214 mg

E-Factor = Amount of waste/Amount of product =214 /121 = 1.77

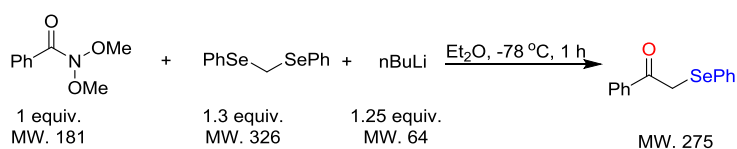
(2) Atom economy (AE):

This work:



$$\begin{aligned} \text{AE} &= [\text{MW of product}] \div \sum(\text{MW of stoichiometric reactants}) \times 100\% \\ &= [(275)/(104+1/2*312+1/2*32)] \times 100\% \\ &= 100\% \end{aligned}$$

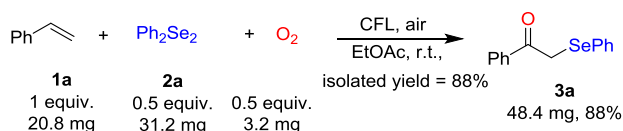
Pace's work (*Org. Lett.* **2018** 2685-2688):



$$\begin{aligned} \text{AE} &= [\text{MW of product}] \div \sum(\text{MW of stoichiometric reactants}) \times 100\% \\ &= [(275)/(181+1.3*326+1.25*64)] \times 100\% \\ &= 40\% \end{aligned}$$

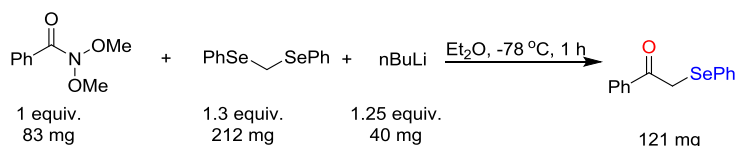
(3) Process mass intensity (PMI):

This work:



$$\begin{aligned} \text{PMI} &= \sum(\text{mass of stoichiometric reactants})/[\text{mass of product}] \\ &= (20.8+31.2+3.2)/48.4 \\ &= 1.14 \end{aligned}$$

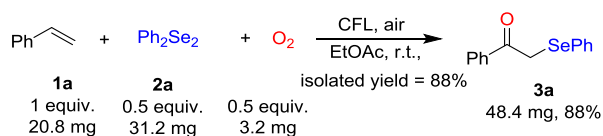
Pace's work (*Org. Lett.* **2018** 2685-2688):



$$\begin{aligned} \text{PMI} &= \sum(\text{mass of stoichiometric reactants})/[\text{mass of product}] \\ &= (83+212+40)/121 \\ &= 2.77 \end{aligned}$$

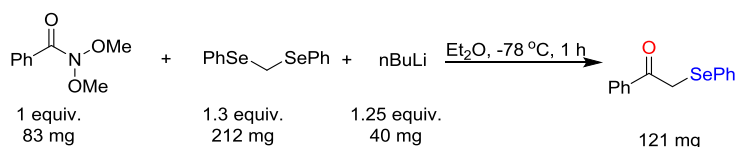
(4) Reaction mass efficiency (RME):

This work:



$$\begin{aligned}
 \text{RME} &= [\text{mass of product} / \sum (\text{mass of stoichiometric reactants})] \times 100\% \\
 &= [48.4 / (20.8 + 31.2 + 3.2)] \times 100\% \\
 &= 88\%
 \end{aligned}$$

Pace's work (*Org. Lett.* **2018** 2685-2688):

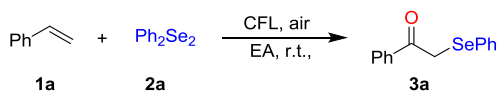


$$\begin{aligned}
 \text{RME} &= [\text{mass of product} / \sum (\text{mass of stoichiometric reactants})] \times 100\% \\
 &= [121 / (83 + 212 + 40)] \times 100\% \\
 &= 36\%
 \end{aligned}$$

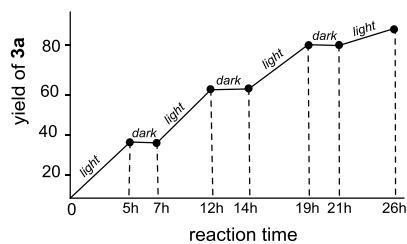
4. Mechanistic studies

1) Light-dark cycle experiments.

The reaction was performed under air atmosphere using 0.2 mmol of styrene, 0.1 mmol Ph₂Se₂ and 2.0 mL ethyl acetate. The reaction was alternately irradiated with a 24 W fluorescent household bulb and kept in the dark for 2 hour intervals. Aliquots were taken at the start and after each interval, the solvent was removed with a rotary evaporator and diluted with CDCl₃ and subjected to ¹H NMR measurements. The reaction yield was determined by ¹H NMR spectroscopy using 1,3,5-trimethoxybenzene as internal standard. The changes in yield observed during the dark phases fall within the margin of error and are thus negligible (Scheme S1).

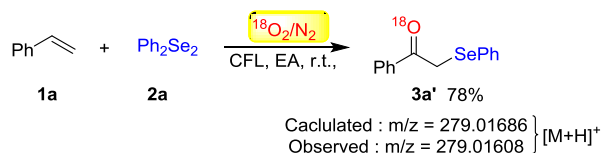


Conditions	Time (h)	NMR Yield of 3a
Light	5	37
Dark	7	35
Light	12	71
Dark	14	72
Light	19	84
Dark	21	85
Light	26	93



Scheme S1. Light-dark cycle experiments

2) ^{18}O labeling experiments



The styrene **1a** (0.2 mmol) and diphenyl diselenide **2a** (0.1 mmol) in ethyl acetate (2 mL) were added to a Schlenk tube equipped with a magnetic stir bar. A balloon filled with $^{18}\text{O}_2/\text{N}_2$ was connected to the Schlenk tube through the side arm and purged one time. The mixture was stirred for 20 h under visible-light irradiation. Then, the solvent of reaction mixture was evaporated under reduced pressure to an oily residue, which was separated on a silica gel column with petroleum ether and ethyl acetate as eluent to afford the desired products. Isolated yield: 78%.

The detection of HRMS for **3a'** $\text{C}_{14}\text{H}_{12}^{18}\text{OSe}$ ($\text{M}+\text{H}$) $^+$: calculated 279.01686, found 279.01608 suggests that the carbonyl oxygen atom of the α -selenoketone originates from dioxygen. The HRMS spectra of **3a'** was pasted here for information (Figure S2).

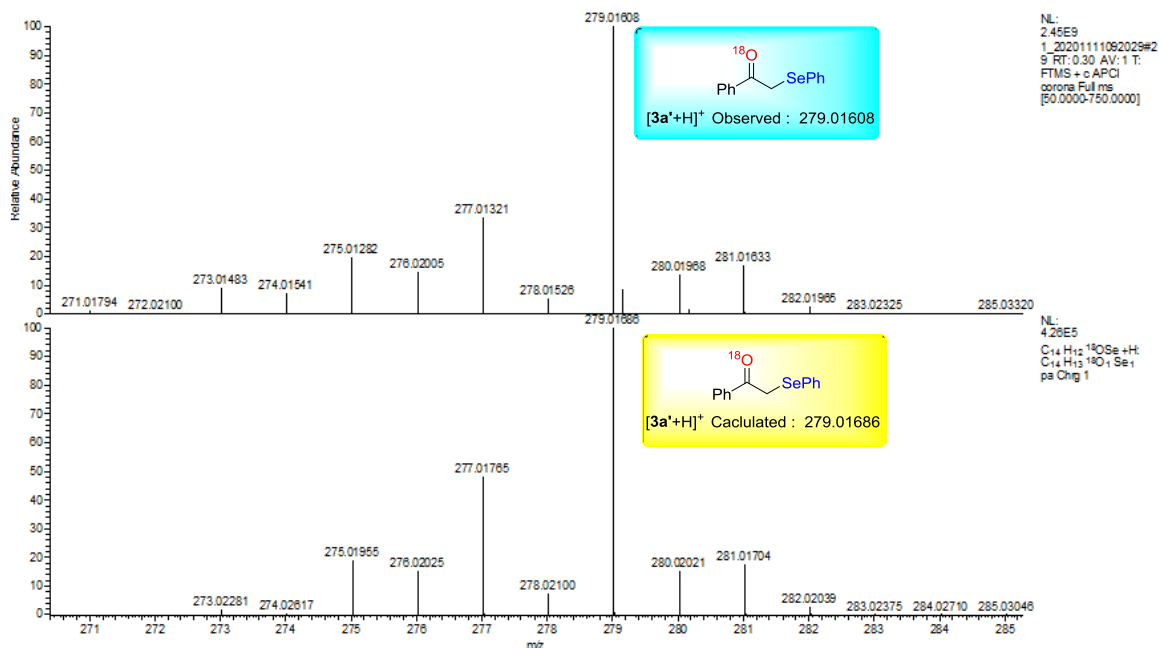
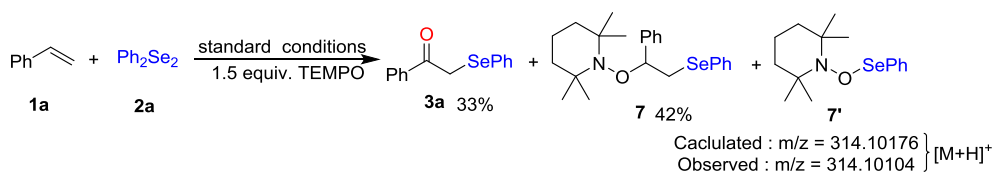


Figure S2 HRMS spectra of **3a'**

3) TEMPO trapping experiments



To a 10 mL vessel with magnetic stir bar were added 0.2 mmol of styrene, 0.1 mmol Ph_2Se_2 , 0.3 mmol of TEMPO and 2 mL of EtOAc. The reaction mixture was stirred in air and irradiated with a 24W household compact fluorescent lamp from a distance of 1 cm. After 20 hours, the reaction mixture was diluted with dichloromethane and the crude material was examined by ESI-MS. A trace amount of TEMPO adducts **7'** was detected on ESI-MS implying the generation of selenium radical **A** during the reaction (Figure S3). Additionally, 42% isolated yield of **7** was obtained, suggesting that the benzyl radical species **B** is the intermediate of this reaction. The NMR spectra of **7** was pasted here for information (Figure S4).

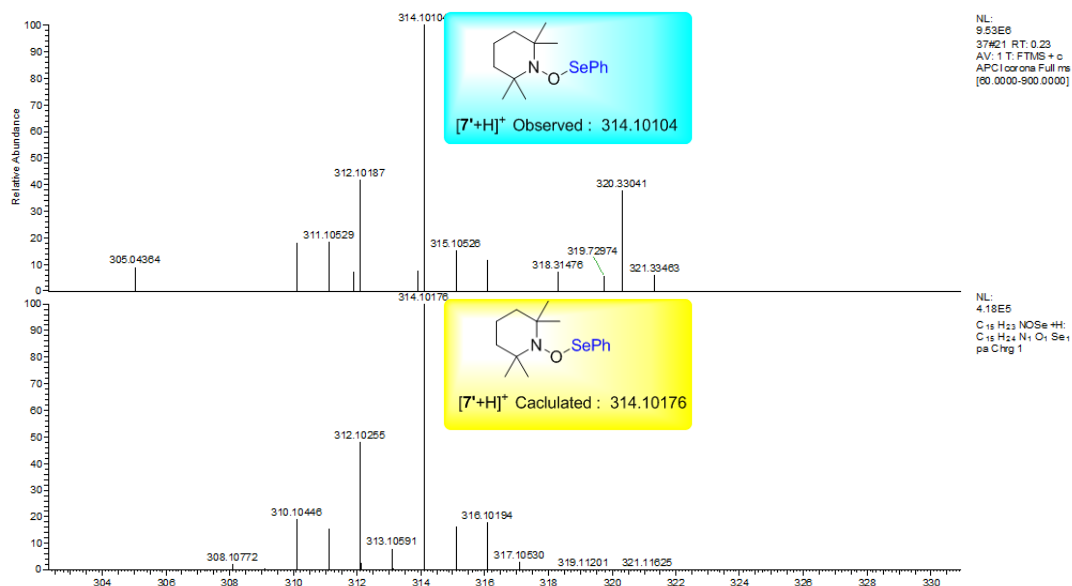
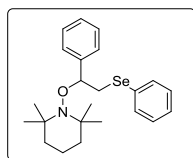


Figure S3 HRMS spectra of **7'**



2,2,6,6-Tetramethyl-1-(1-phenyl-2-(phenylselenanyl)ethoxy)piperidine (7). The crude product was purified by silica gel chromatography (petroleum ether/ethyl acetate = 90/1) to give compound **7** (35 mg, 42%) as an oil. ^1H NMR (400 MHz, CDCl_3) δ/ppm =7.31 – 7.14 (m, 7H), 7.13 – 7.07 (m, 3H), 4.80 (dd, $J = 9.8, 4.1$ Hz, 1H), 3.57 (dd, $J = 11.5, 4.1$ Hz, 1H), 3.22 (dd, $J = 11.5, 9.8$ Hz, 1H), 1.62 – 1.03 (m, 9H), 1.1 (s, 3H), 0.93 (s, 3H), 0.57 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ/ppm = 140.8, 131.6, 129.7, 127.8, 126.9, 126.7, 126.6, 125.6, 84.8, 58.9, 39.3, 33.1, 19.3, 16.1. Spectral data are in good agreement with literature values.²

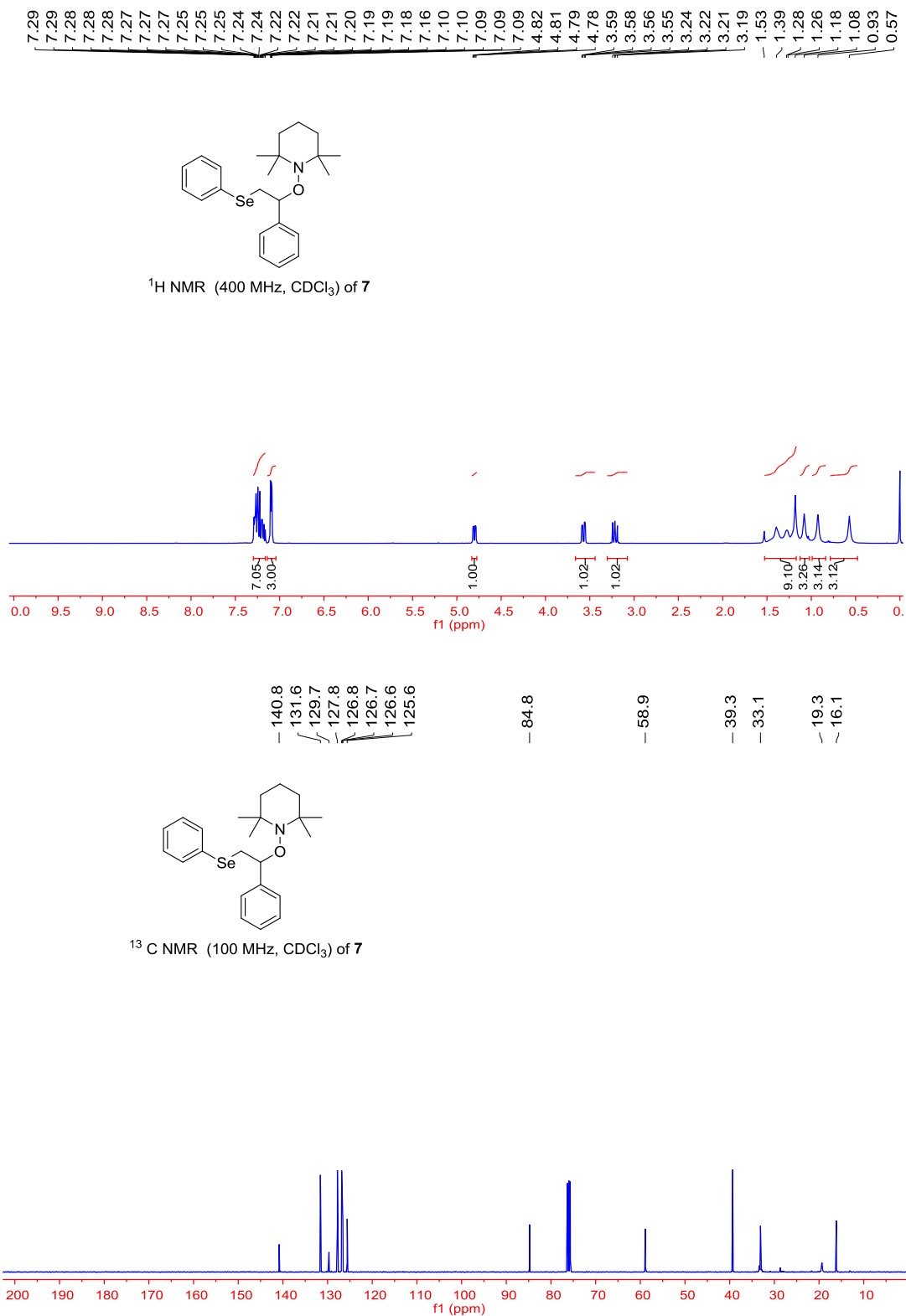


Figure S4 NMR spectra of 7

4) UV-Vis absorption and emission spectra

UV-visible absorption spectra were collected on a UV1800PC (Jinghua, China). The samples were prepared 1.80×10^{-4} mol/L in EtOAc. The spectra obtained were listed as follow (Figure S5a). Figure S5a reveals that absorption maxima of diphenyl diselenide **2a** are located around 330 nm, with corresponding absorption band

extending from visible light region, while styrene **1a** has no absorb to UV-visible light. Additionally, UV-visible absorption spectra of 1:1 molar ratio double-component samples in EtOAc were also collected and no obvious changes absorption was observed. This UV-Vis indicates that the involvement of any photoactive intermediates (EDA complexes) seems very unlikely.

All reactions were performed using 24 W energy-saving household CFL bulb by Oppl (see section II of this SI), the emission spectrum of which is shown in Figure S5b. The emission spectrum of blue and green lamp was also collected and is provided below (Figure S5c and S5d). These results corroborate the use of commercial white and blue lamp as suitable for the preparation of selenomethyl ketones, while the emission spectrum of green light source and the absorption spectrum of the substrate do not exactly match.

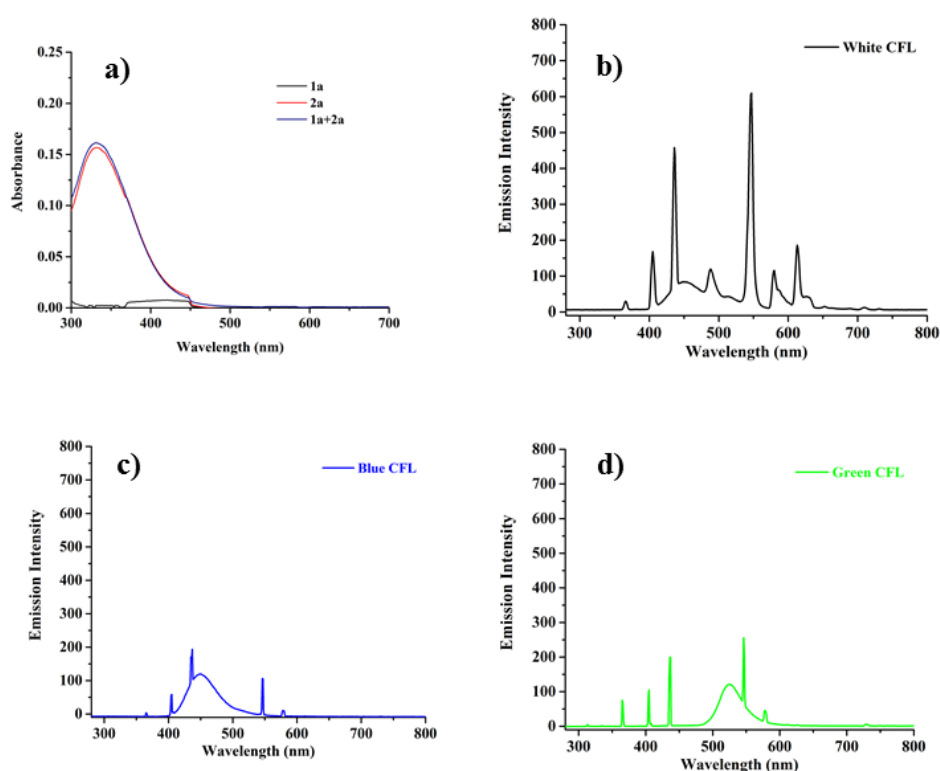
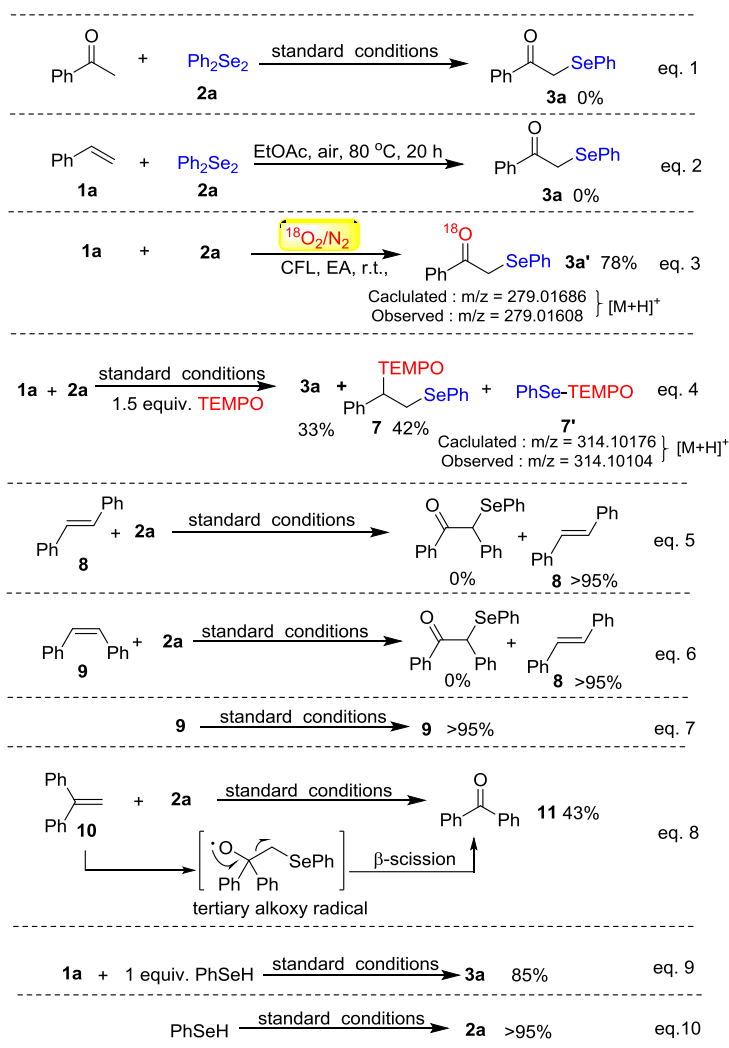


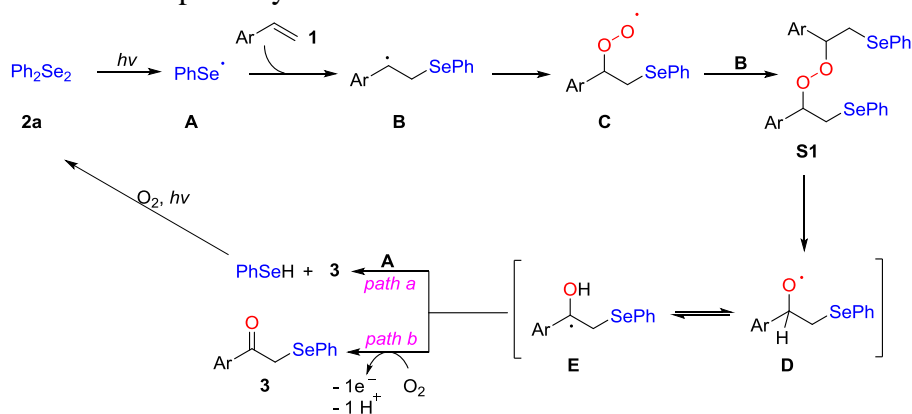
Figure S5. a) UV-visible absorption spectra of samples; b) Emission spectra of CFL lamps used; c) Emission spectra of blue lamps used; d) Emission spectra of green lamps used.

5) Experiments for mechanistic studies



Scheme S2. Control experiments

6) Tentative reaction pathway

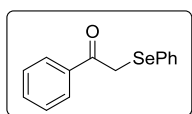


The formation of ketones **3** is described in detail as above. Analogue of intermediates **C**, **S1**, **D** and **E** are involved as previously reported literatures (see ref. 36).

5. General procedure for the synthesis of α -selenomethyl ketones

The reaction was carried out in an open air system. To a 10 mL vessel with magnetic stir bar were added 0.2 mmol vinylarene, 0.1 mmol diselenide and 2 mL of EtOAc. The reaction mixture was stirred and irradiated with a 24W household compact fluorescent lamp from a distance of 1 cm. The reaction was monitored by TLC. After completion of reaction (*ca.* 20 h), the solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent.

6. Characterization data

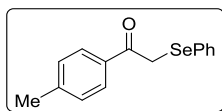


1-Phenyl-2-(phenylselenanyl)ethan-1-one (3a). Compound **3a** was prepared according to the general procedure and isolated as an oil (48 mg, 88% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ /ppm= 7.83 – 7.78 (m, 2H), 7.51 – 7.43 (m, 3H), 7.36 (t, $J = 7.7$ Hz, 2H), 7.23 – 7.16 (m, 3H), 4.10 (s, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ /ppm= 193.9, 134.4, 133.0, 132.2, 128.2, 128.0, 127.7, 127.6, 127.1, 31.7.

Spectral data are in good agreement with literature values.³



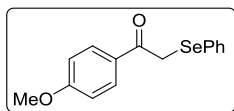
2-(Phenylselenanyl)-1-(*p*-tolyl)ethan-1-one (3b). Compound **3b** was prepared according to the general procedure and isolated as a yellow solid (52 mg, 90% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

mp = 34-35 °C.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ /ppm= 7.71 (d, $J = 8.2$ Hz, 2H), 7.50 – 7.43 (m, 2H), 7.24 – 7.13 (m, 5H), 4.08 (s, 2H), 2.33 (s, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ /ppm= 193.7, 143.2, 132.9, 131.9, 128.3, 128.2, 128.1, 127.8, 127.0, 31.7, 20.7.

Spectral data are in good agreement with literature values.³



1-(4-Methoxyphenyl)-2-(phenylselenanyl)ethan-1-one (3c). Compound **3c** was prepared according to the general procedure and isolated as a white solid (53 mg, 86% yield) after flash chromatography (petroleum ether/ethyl acetate = 15/1).

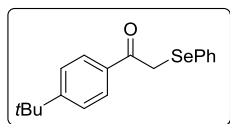
mp = 63-65 °C.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ /ppm= 7.79 (d, $J = 8.9$ Hz, 2H), 7.49 – 7.45 (m, 2H),

7.23 – 7.18 (m, 3H), 6.83 (d, $J = 8.9$ Hz, 2H), 4.07 (s, 2H), 3.79 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) $\delta/\text{ppm} = 192.7, 162.6, 132.8, 130.0, 128.2, 128.2, 127.4, 126.9, 112.7, 54.5, 31.6$.

Spectral data are in good agreement with literature values.³

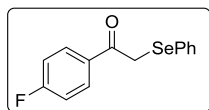


1-(4-(*tert*-Butyl)phenyl)-2-(phenylselanyl)ethan-1-one (3d). Compound **3d** was prepared according to the general procedure and isolated as an oil (61 mg, 92% yield) after flash chromatography (petroleum ether/ethyl acetate = 20/1).

^1H NMR (400 MHz, CDCl_3) $\delta/\text{ppm} = 7.75$ (d, $J = 8.5$ Hz, 2H), 7.49 – 7.44 (m, 2H), 7.37 (d, $J = 8.5$ Hz, 2H), 7.23 – 7.17 (m, 3H), 4.09 (s, 2H), 1.26 (s, 9H).

^{13}C NMR (100 MHz, CDCl_3) $\delta/\text{ppm} = 193.7, 156.1, 132.9, 131.8, 128.2, 127.6, 126.9, 124.6, 34.1, 31.7, 30.0$.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{18}\text{H}_{21}\text{OSe}$ 333.0752; Found 333.0758.



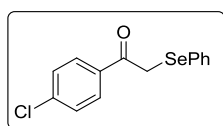
1-(4-Fluorophenyl)-2-(phenylselanyl)ethan-1-one (3e). Compound **3e** was prepared according to the general procedure and isolated as an oil (49 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

^1H NMR (400 MHz, CDCl_3) $\delta/\text{ppm} = 7.84 - 7.77$ (m, 2H), 7.47 – 7.42 (m, 2H), 7.24 – 7.16 (m, 3H), 7.04 – 6.97 (m, 2H), 4.06 (s, 2H).

^{13}C NMR (100 MHz, CDCl_3) $\delta/\text{ppm} = 192.4, 164.76$ (d, $J = 255.3$ Hz), 133.1, 130.8 (d, $J = 3.1$ Hz), 130.4 (d, $J = 9.4$ Hz), 128.3, 127.8, 127.2, 114.7 (d, $J = 21.9$ Hz), 31.5.

^{19}F NMR (376 MHz, CDCl_3) $\delta/\text{ppm} = -104.7$.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{14}\text{H}_{12}\text{FOSe}$ 295.0032; Found 295.0032.

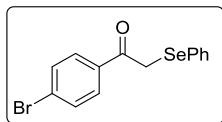


1-(4-Chlorophenyl)-2-(phenylselanyl)ethan-1-one (3f). Compound **3f** was prepared according to the general procedure and isolated as an oil (49 mg, 79% yield) after flash chromatography (petroleum ether/ethyl acetate = 60/1).

^1H NMR (400 MHz, CDCl_3) $\delta/\text{ppm} = 7.72$ (d, $J = 8.6$ Hz, 2H), 7.46 – 7.41 (m, 2H), 7.75 (d, $J = 8.6$ Hz, 2H), 7.24 – 7.17 (m, 3H), 4.05 (s, 2H).

^{13}C NMR (100 MHz, CDCl_3) $\delta/\text{ppm} = 192.7, 138.7, 133.1, 132.7, 129.1, 128.3, 127.9, 127.7, 127.3, 31.4$.

Spectral data are in good agreement with literature values.³



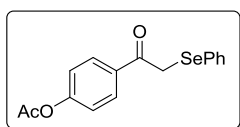
1-(4-Bromophenyl)-2-(phenylselanyl)ethan-1-one (3g). Compound **3g** was prepared according to the general procedure and isolated as yellow solid (57 mg, 80% yield) after flash chromatography (petroleum ether/ethyl acetate = 60/1).

mp = 41-42 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.72 (d, *J* = 8.6 Hz, 2H), 7.48 (d, *J* = 8.6 Hz, 2H), 7.46 – 7.41 (m, 2H), 7.24 – 7.17 (m, 3H), 4.04 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 192.9, 133.1, 133.1, 130.9, 129.2, 128.3, 127.6, 127.4, 127.3, 31.4.

Spectral data are in good agreement with literature values.⁴



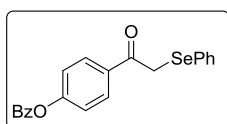
4-(2-(Phenylselanyl)acetyl)phenyl acetate (3h). Compound **3h** was prepared according to the general procedure and isolated as a white solid (57 mg, 85% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1).

mp = 56-59 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.83(d, *J* = 8.8 Hz, 2H), 7.48 – 7.41 (m, 2H), 7.23 – 7.16 (m, 3H), 7.08 (d, *J* = 8.8 Hz, 2H), 4.07 (s, 2H), 2.24 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=192.7, 167.8, 153.4, 133.0, 131.9, 129.3, 128.3, 127.8, 127.2, 120.8, 31.5, 20.1.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₆H₁₅O₃Se 335.0181; Found 335.0180.



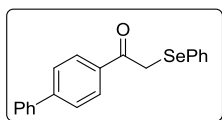
4-(2-(Phenylselanyl)acetyl)phenyl benzoate (3i). Compound **3i** was prepared according to the general procedure and isolated as a yellow solid (60 mg, 76% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1).

mp = 86-88 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 8.13 (d, *J* = 7.8 Hz, 2H), 7.88 (d, *J* = 8.6 Hz, 2H), 7.58 (dd, *J* = 10.6, 4.3 Hz, 1H), 7.50 – 7.42 (m, 4H), 7.24 – 7.18 (m, 5H), 4.10 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 192.7, 163.5, 153.7, 133.1, 132.9, 132.0, 129.4, 129.2, 128.3, 128.0, 127.9, 127.7, 127.2, 120.9, 31.6.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₂₁H₁₇O₃Se 397.0337; Found 397.0345.



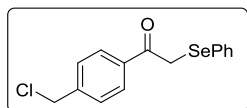
1-([1,1'-Biphenyl]-4-yl)-2-(phenylselanyl)ethan-1-one (3j). Compound **3j** was prepared according to the general procedure and isolated as a white solid (64 mg, 91% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

mp = 105-107 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.88 – 7.83 (m, 2H), 7.58 – 7.51 (m, 4H), 7.49 – 7.44 (m, 2H), 7.42 – 7.35 (m, 2H), 7.34 – 7.29 (m, 1H), 7.22 – 7.15 (m, 3H), 4.11 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=193.5, 144.9, 138.7, 133.1, 133.0, 132.9, 128.3, 128.2, 127.9, 127.3, 127.1, 126.23, 126.20, 31.7.

Spectral data are in good agreement with literature values.⁵

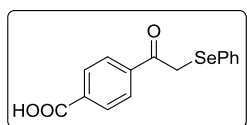


1-(4-(Chloromethyl)phenyl)-2-(phenylselanyl)ethan-1-one (3k). Compound **3k** was prepared according to the general procedure and isolated as an oil (54 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 20/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.79 (d, *J* = 8.4 Hz, 2H), 7.45 (dd, *J* = 7.7, 1.8 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.27 – 7.13 (m, 3H), 4.53 (s, 2H), 4.08 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 193.2, 141.5, 134.2, 133.1, 128.3, 128.1, 127.8, 127.7, 127.2, 44.2, 31.6.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₄ClOSe 324.9893; Found 324.9891.



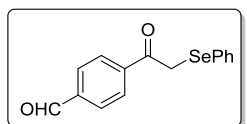
4-(2-(Phenylselanyl)acetyl)benzoic acid (3l). Compound **3l** was prepared according to the general procedure and isolated as a yellow solid (52 mg, 81% yield) after flash chromatography (petroleum ether/ethyl acetate = 1/1).

mp = 126-128 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 8.09 (d, *J* = 8.5 Hz, 2H), 7.87 (d, *J* = 8.5 Hz, 2H), 7.44 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.30 – 7.09 (m, 3H), 4.11(s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=193.1, 169.7, 138.4, 133.3, 131.9, 129.4, 128.3, 127.7, 127.4, 127.3, 31.6.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₂O₃Se 321.00244; Found 321.00192



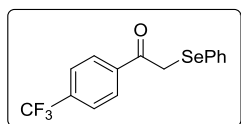
4-(2-(Phenylselanyl)acetyl)benzaldehyde (3m). Compound **3m** was prepared according to the general procedure and isolated as a yellow solid (45 mg, 74% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1).

mp = 47-49 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 10.01 (s, 1H), 7.91 (d, *J* = 8.5 Hz, 2H), 7.85 (d, *J* = 8.6 Hz, 2H), 7.43 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.27 – 7.13 (m, 3H), 4.09(s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=193.0, 190.5, 138.8, 137.9, 133.2, 128.7, 128.3, 128.2, 127.4, 127.3, 31.6.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₂O₂Se 305.00753; Found 306.00696.



2-(Phenylselanyl)-1-(4-(trifluoromethyl)phenyl)ethan-1-one (3n). Compound **3n** was prepared according to the general procedure and isolated as a yellow solid (59 mg, 86% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

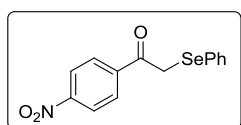
mp = 44-46 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm=7.86 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 8.2 Hz, 2H), 7.46 – 7.33 (m, 2H), 7.27 – 7.10 (m, 3H), 4.06 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=192.7, 137.1, 133.3, 128.3, 128.0, 127.4, 127.3, 124.6 (q, *J*_{C-F} = 3.7 Hz), 123.8, 121.1, 31.4.

¹⁹F NMR (376 MHz, CDCl₃) δ/ppm= -63.1.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₁F₃OSe 345.00000; Found 344.99936



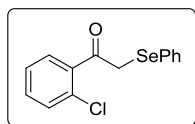
1-(4-Nitrophenyl)-2-(phenylselanyl)ethan-1-one (3o). Compound **3o** was prepared according to the general procedure and isolated as a red solid (53 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1).

mp = 72-74 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm=8.16 (d, *J* = 8.7 Hz, 2H), 7.90 (d, *J* = 8.7 Hz, 2H), 7.45 – 7.35 (m, 2H), 7.34 – 7.14 (m, 3H), 4.07 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=191.9, 149.2, 139.0, 133.4, 128.6, 128.4, 127.6, 127.0, 122.7, 31.5.

Spectral data are in good agreement with literature values.³



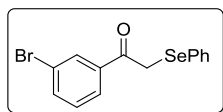
1-(2-Chlorophenyl)-2-(phenylselanyl)ethan-1-one (3p). Compound **3p** was prepared according to the general procedure and isolated as an oil (43 mg, 70% yield) after flash chromatography (petroleum ether/ethyl acetate = 100/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.41 – 7.32 (m, 3H), 7.30 – 7.24 (m, 2H), 7.21 – 7.14 (m, 4H), 4.14 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 197.3, 137.7, 133.7, 132.0, 131.1, 130.4, 130.3,

129.2, 128.6, 128.0, 126.8, 36.4.

Spectral data are in good agreement with literature values.⁵



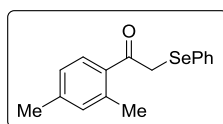
1-(3-Bromophenyl)-2-(phenylselanyl)ethan-1-one (3q). Compound **3q** was prepared according to the general procedure and isolated as a yellow solid (55 mg, 77% yield) after flash chromatography (petroleum ether/ethyl acetate = 50/1).

mp = 36-37 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.88 (t, *J* = 1.8 Hz, 1H), 7.71 – 7.67 (m, 1H), 7.61 – 7.56 (m, 1H), 7.47 – 7.42 (m, 2H), 7.25 – 7.17 (m, 4H), 4.03 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=192.4, 136.1, 135.0, 133.3, 130.7, 129.1, 128.3, 127.5, 127.4, 126.2, 121.9, 31.4.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₄H₁₂BrOSe 354.9231; Found 354.9234.

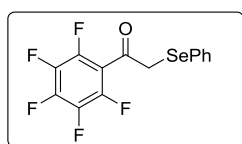


1-(2,4-Dimethylphenyl)-2-(phenylselanyl)ethan-1-one (3r). Compound **3r** was prepared according to the general procedure and isolated as an oil (51 mg, 84% yield) after flash chromatography (petroleum ether/ethyl acetate = 90/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm=7.45 – 7.39 (m, 2H), 7.36 (d, *J* = 7.9 Hz, 1H), 7.23 – 7.09 (m, 3H), 6.96 (d, *J* = 1.7 Hz, 1H), 6.90 (dd, *J* = 7.9, 1.7 Hz, 1H), 4.05 (s, 2H), 2.34 (s, 3H), 2.25 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=196.7, 141.2, 138.6, 132.5, 132.2, 131.9, 128.19, 128.12, 128.08, 126.7, 125.1, 34.5, 20.42, 20.36.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₆H₁₆OSe 305.04391; Found 305.04330.



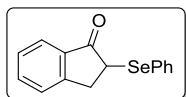
1-(Perfluorophenyl)-2-(phenylselanyl)ethan-1-one (3s). Compound **3s** was prepared according to the general procedure and isolated as an oil (46 mg, 63% yield) after flash chromatography (petroleum ether/ethyl acetate = 20/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.52 – 7.47 (m, 2H), 7.41 – 7.23 (m, 3H), 3.99 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 144.7 (m), 143.0 (m), 142. (m), 140.5 (m), 137.7 (m), 135.1 (m), 133.2, 128.3, 127.7, 126.3, 36.8.

¹⁹F NMR (376 MHz, CDCl₃) δ/ppm=-139.7, -149.2, -160.2.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₄H₈F₅OSe 366.9655; Found 366.9667.

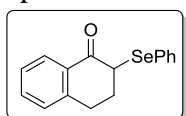


2-(Phenylselanyl)-2,3-dihydro-1H-inden-1-one (3t). Compound **3t** was prepared according to the general procedure and isolated as an oil (38 mg, 66% yield) after flash chromatography (petroleum ether/ethyl acetate = 50/1).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ/ppm = 7.70 (d, J = 7.7 Hz, 1H), 7.54 – 7.48 (m, 3H), 7.31 – 7.24 (m, 2H), 7.20 – 7.15 (m, 3H), 4.14 (dd, J = 7.6, 2.7 Hz, 1H), 3.56 (dd, J = 18.0, 7.6 Hz, 1H), 3.09 (dd, J = 18.0, 2.6 Hz, 1H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ/ppm = 202.2, 151.2, 134.5, 134.3, 134.0, 128.0, 127.4, 126.7, 126.5, 125.2, 123.4, 42.3, 34.1.

Spectral data are in good agreement with literature values.⁶

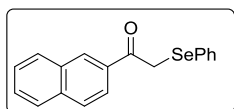


2-(Phenylselanyl)-3,4-dihydronaphthalen-1(2H)-one (3u). Compound **3u** was prepared according to the general procedure and isolated as an oil (42 mg, 70% yield) after flash chromatography (petroleum ether/ethyl acetate = 50/1).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ/ppm = 7.99 (dd, J = 7.8, 1.1 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.44 – 7.37 (m, 1H), 7.28 – 7.21 (m, 4H), 7.16 (d, J = 7.7 Hz, 1H), 4.19 (t, J = 4.5 Hz, 1H), 3.22 – 3.10 (m, 1H), 2.81 (dt, J = 17.1, 4.5 Hz, 1H), 2.50 – 2.39 (m, 1H), 2.34 – 2.24 (m, 1H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ/ppm = 192.7, 141.8, 134.4, 132.5, 130.5, 130.2, 128.1, 127.6, 127.3, 127.1, 126.7, 126.6, 125.9, 47.6, 28.3, 26.0.

Spectral data are in good agreement with literature values.⁷



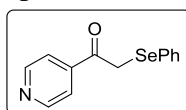
1-(Naphthalen-2-yl)-2-(phenylselanyl)ethan-1-one (3v). Compound **3v** was prepared according to the general procedure and isolated as a yellow solid (54 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

mp = 66-68 °C.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ/ppm = 8.20 (s, 1H), 7.89 (dd, J = 8.6, 1.8 Hz, 1H), 7.82 – 7.72 (m, 3H), 7.55 – 7.42 (m, 4H), 7.24 – 7.15 (m, 3H), 4.21 (s, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ/ppm = 194.0, 134.6, 133.2, 131.7, 131.3, 129.5, 128.6, 128.2, 128.1, 127.6, 127.5, 127.2, 126.7, 125.7, 123.3, 31.8.

Spectral data are in good agreement with literature values.³

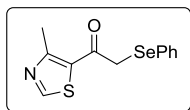


2-(Phenylselanyl)-1-(pyridin-4-yl)ethan-1-one (3w). Compound **3w** was prepared according to the general procedure and isolated as an oil (31 mg, 56% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 8.67 (d, *J* = 6.2 Hz, 2H), 7.53 (d, *J* = 6.2 Hz, 2H), 7.46 – 7.34 (m, 2H), 7.31 – 7.13 (m, 3H), 4.02 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 193.8, 150.8, 141.5, 134.4, 129.4, 128.6, 128.1, 121.6, 32.2.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₃H₁₂N₁OSe 278.0079; Found 278.0081.



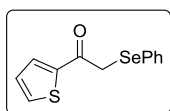
1-(4-Methylthiazol-5-yl)-2-(phenylselanyl)ethan-1-one (3x). Compound **3x** was prepared according to the general procedure and isolated as a yellow solid (51 mg, 86% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

mp = 78-80 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 8.76 (s, 1H), 7.50 – 7.41 (m, 2H), 7.25 – 7.17 (m, 3H), 3.92 (s, 2H), 2.66 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 133.2, 130.5, 128.3, 128.2, 127.4, 127.2, 126.7, 35.7, 17.2.

HRMS (ESI /Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₂H₁₂N₁OSe 297.9799; Found 297.9800.

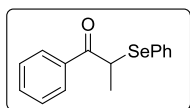


2-(Phenylselanyl)-1-(thiophen-2-yl)ethan-1-one (3y). Compound **3y** was prepared according to the general procedure and isolated as an oil (47 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 15/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.56 (dd, *J* = 4.9, 0.9 Hz, 1H), 7.51 – 7.47 (m, 2H), 7.47 – 7.44 (m, 1H), 7.23 – 7.18 (m, 3H), 7.01 – 6.97 (m, 1H), 3.99 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 187.3, 141.7, 133.2, 133.1, 131.7, 128.2, 128.1, 127.2, 127.1, 32.1.

Spectral data are in good agreement with literature values.⁵

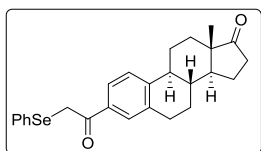


1-Phenyl-2-(phenylselanyl)propan-1-one (3z). Compound **3z** was prepared according to the general procedure and isolated as an oil (35 mg, 61% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.88 – 7.77 (m, 2H), 7.54 – 7.43 (m, 1H), 7.42 – 7.31 (m, 4H), 7.30 – 7.25 (m, 1H), 7.23 – 7.15 (m, 2H), 4.62 (q, *J* = 6.8 Hz, 1H), 1.57 (d, *J* = 6.8 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 195.3, 135.6, 134.8, 131.8, 128.0, 127.9, 127.5, 127.4, 125.8, 38.7, 16.2.

Spectral data are in good agreement with literature values.⁸



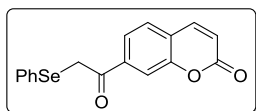
(8R,9S,13S,14S)-13-Methyl-3-(2-(phenylselanyl)acetyl)-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (3aa). Compound **3aa** was prepared according to the general procedure and isolated as an oil (69 mg, 77% yield) after flash chromatography (petroleum ether/ethyl acetate = 6/1).

$[\alpha]_{25}^D = +167.1$, $c=0.023$ g/100 mL, CHCl_3 .

$^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta/\text{ppm} = 7.58$ (d, $J = 8.2$ Hz, 1H), 7.52 (s, 1H), 7.49 – 7.45 (m, 2H), 7.28 (d, $J = 8.2$ Hz, 1H), 7.24 – 7.17 (m, 3H), 4.07 (s, 2H), 2.89 – 2.81 (m, 2H), 2.50 – 2.33 (m, 2H), 2.32 – 2.21 (m, 1H), 2.02 – 1.88 (m, 3H), 1.63 – 1.34 (m, 7H), 0.85 (s, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta/\text{ppm} = 220.1, 193.9, 144.7, 136.0, 132.9, 132.0, 128.4, 128.3, 128.2, 127.0, 125.1, 124.6, 49.5, 46.9, 43.7, 36.8, 34.8, 31.7, 30.5, 28.3, 25.2, 24.5, 20.6, 12.8$.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{26}\text{H}_{29}\text{O}_2\text{Se}$ 453.1327; Found 453.1326.



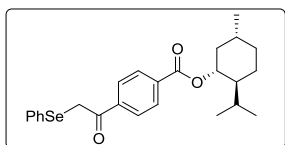
7-(2-(Phenylselanyl)acetyl)-2H-chromen-2-one (3ab). Compound **3ab** was prepared according to the general procedure and isolated as a yellow solid (59 mg, 86% yield) after flash chromatography (petroleum ether/ethyl acetate = 4/1).

mp = 90-92 °C.

$^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta/\text{ppm} = 7.71 - 7.63$ (m, 3H), 7.50 – 7.40 (m, 3H), 7.24 – 7.16 (m, 3H), 6.45 (d, $J = 9.6$ Hz, 1H), 4.08 (s, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta/\text{ppm} = 192.2, 159.0, 152.7, 141.4, 136.9, 133.2, 128.4, 127.5, 127.3, 127.1, 123.2, 121.2, 117.9, 116.1, 31.5$.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{17}\text{H}_{13}\text{O}_3\text{Se}$ 345.0024; Found 345.0026.



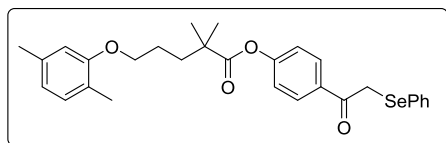
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-(2-(phenylselanyl)acetyl)benzoate (3ac). Compound **3ac** was prepared according to the general procedure and isolated as an oil (82 mg, 90% yield) after flash chromatography (petroleum ether/ethyl acetate = 45/1).

$[\alpha]_{25}^D = -30.2$, $c=0.046$ g/100 mL, CHCl_3 .

$^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta/\text{ppm} = 8.00$ (d, $J = 8.6$ Hz, 1H), 7.83 (d, $J = 8.6$ Hz, 1H), 7.47 – 7.39 (m, 2H), 7.24 – 7.16 (m, 3H), 4.88 (td, $J = 10.9, 4.4$ Hz, 1H), 4.08 (s, 2H), 2.09 – 2.01 (m, 1H), 1.91 – 1.81 (m, 1H), 1.70 – 1.62 (m, 2H), 1.54 – 1.43 (m, 2H), 1.19 (d, $J = 11.9$ Hz, 1H), 1.10 – 1.00 (m, 2H), 0.86 (d, $J = 3.7$ Hz, 3H), 0.85 (d, $J = 4.2$ Hz, 3H), 0.72 (d, $J = 7.0$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm =193.2, 164.1, 137.5, 133.6, 133.1, 128.7, 128.3, 127.6, 127.5, 127.3, 74.5, 46.2, 39.9, 33.2, 31.7, 30.4, 25.5, 22.6, 21.0, 19.7, 15.5.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{25}\text{H}_{31}\text{O}_3\text{Se}$ 459.1433; Found 459.1428.



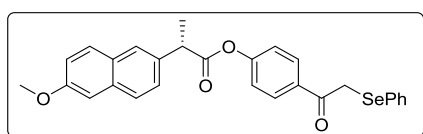
4-(2-(Phenylselanyl)acetyl)phenyl

5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (3ad). Compound **3ad** was prepared according to the general procedure and isolated as an oil (89 mg, 85% yield) after flash chromatography (petroleum ether/ethyl acetate = 20/1).

^1H NMR (400 MHz, CDCl_3) δ/ppm = 7.84 – 7.76 (m, 2H), 7.48 – 7.40 (m, 2H), 7.22 – 7.15 (m, 3H), 7.04 – 6.98 (m, 2H), 6.92 (d, J = 7.4 Hz, 1H), 6.59 (d, J = 7.4 Hz, 1H), 6.54 (s, 1H), 4.06 (s, 2H), 3.90 (t, J = 5.2 Hz, 2H), 2.22 (s, 3H), 2.09 (s, 3H), 1.80 (s, 4H), 1.30 (s, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm =192.7, 174.7, 155.8, 153.9, 135.5, 133.0, 131.8, 129.3, 129.3, 128.3, 127.9, 127.1, 122.5, 120.7, 119.8, 110.9, 66.6, 41.6, 36.1, 31.6, 24.2, 24.1, 20.4, 14.8.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{29}\text{H}_{33}\text{O}_4\text{Se}$ 525.1539; Found 525.1542.



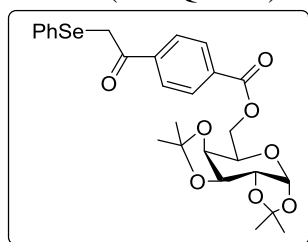
4-(2-(Phenylselanyl)acetyl)phenyl (S)-2-(6-methoxynaphthalen-2-yl)propanoate (3ae). Compound **3ae** was prepared according to the general procedure and isolated as an oil (83 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1).

$[\alpha]_{25}^{\text{D}}$ = + 91.5, c =0.033 g/100 mL, CHCl_3 .

^1H NMR (400 MHz, CDCl_3) δ/ppm = 7.76 (d, J = 8.7 Hz, 2H), 7.71 – 7.64 (m, 3H), 7.45 – 7.38 (m, 3H), 7.21 – 7.15 (m, 3H), 7.11 – 7.04 (m, 2H), 7.00 – 6.94 (m, 2H), 4.07 – 4.00 (m, 3H), 3.84 (s, 3H), 1.62 (d, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm =192.7, 171.5, 156.8, 153.6, 133.7, 133.0, 132.9, 131.9, 129.2, 128.3, 128.2, 127.9, 127.8, 127.1, 126.5, 125.2, 124.9, 120.6, 118.2, 104.6, 54.3, 44.6, 31.5, 17.4.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{28}\text{H}_{25}\text{O}_4\text{Se}$ 505.0913; Found 505.0921.



((3aR,5R,5aS,8aS,8bR)-2,2,7,7-Tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)methyl 4-(2-(phenylselanyl)acetyl)benzoate (3af). Compound

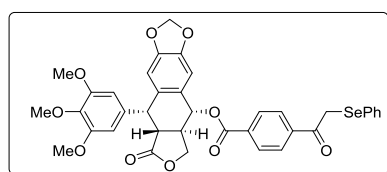
3af was prepared according to the general procedure and isolated as an oil (86 mg, 77% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1).

$[\alpha]_{25}^D = -41.0$, $c=0.13$ g/100 mL, CHCl_3 .

$^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta/\text{ppm} = 8.01$ (d, $J = 8.5$ Hz, 2H), 7.82 (d, $J = 8.5$ Hz, 2H), 7.46 – 7.40 (m, 2H), 7.25 – 7.16 (m, 3H), 5.50 (d, $J = 5.0$ Hz, 1H), 4.59 (dd, $J = 7.9, 2.5$ Hz, 1H), 4.48 (dd, $J = 11.6, 4.7$ Hz, 1H), 4.39 (dd, $J = 11.6, 7.7$ Hz, 1H), 4.29 (dd, $J = 5.0, 2.5$ Hz, 1H), 4.26 (dd, $J = 7.9, 1.8$ Hz, 1H), 4.14 – 4.10 (m, 1H), 4.09 (s, 2H), 1.45 (s, 3H), 1.41 (s, 3H), 1.29 (s, 3H), 1.27 (s, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta/\text{ppm} = 193.2, 164.5, 137.8, 133.2, 132.8, 128.9, 128.3, 127.6, 127.5, 127.3, 108.8, 107.8, 95.3, 70.1, 69.7, 69.5, 65.1, 63.4, 31.6, 25.0, 25.0, 23.9, 23.5$.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{27}\text{H}_{31}\text{O}_8\text{Se}$ 563.1179; Found 563.1182.



(5*R*,5*aR*,8*aR*,9*R*)-8-Oxo-9-(3,4,5-trimethoxyphenyl)-5,5*a*,6,8,8*a*,9-hexahydrofuro[3',4':6,7]naphtho[2,3-*d*][1,3]dioxol-5-yl 4-(2-(phenylselanyl)acetyl)benzoate (3ag).

Compound **3ag** was prepared according to the general procedure and isolated as a white solid (91 mg, 64% yield) after flash chromatography (petroleum ether/ethyl acetate = 4/1).

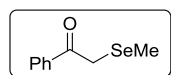
$[\alpha]_{25}^D = -38.1$, $c=0.10$ g/100 mL, CHCl_3 .

$\text{mp} = 84\text{--}86$ °C.

$^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta/\text{ppm} = 8.02$ (d, $J = 8.4$ Hz, 2H), 7.87 (d, $J = 8.4$ Hz, 2H), 7.47 – 7.41 (m, 2H), 7.27 – 7.17 (m, 3H), 6.78 (s, 1H), 6.53 (s, 1H), 5.93 (d, $J = 8.3$ Hz, 2H), 4.59 (d, $J = 3.4$ Hz, 1H), 4.38 (dd, $J = 9.1, 6.2$ Hz, 1H), 4.26 (t, $J = 9.7$ Hz, 1H), 4.12 – 4.05 (m, 2H), 3.72 (d, $J = 11.0$ Hz, 9H), 1.97 (s, 1H), 1.22 – 1.16 (m, 4H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta/\text{ppm} = 192.9, 172.6, 164.9, 151.7, 147.3, 146.8, 138.4, 136.2, 133.7, 133.2, 131.9, 131.6, 128.9, 128.4, 127.8, 127.4, 127.0, 108.9, 107.1, 106.0, 100.7, 73.8, 70.4, 59.7, 55.1, 44.6, 42.7, 37.8, 31.6, 28.7$.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{37}\text{H}_{33}\text{O}_{10}\text{Se}$ 717.1233; Found 717.1237.

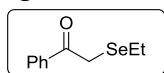


2-(Methylselanyl)-1-phenylethan-1-one (4a). Compound **4a** was prepared according to the general procedure and isolated as an oil (30 mg, 70% yield) after flash chromatography (petroleum ether/ethyl acetate = 50/1).

$^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta/\text{ppm} = 7.93$ – 7.87 (m, 2H), 7.50 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.6$ Hz, 2H), 3.69 (s, 2H), 2.02 (s, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta/\text{ppm} = 194.5, 135.1, 133.2, 128.7, 128.7, 27.9, 5.9$.

Spectral data are in good agreement with literature values.⁵

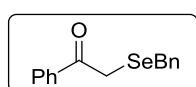


2-(Ethylselanyl)-1-phenylethan-1-one (4b). Compound **4b** was prepared according to the general procedure and isolated as an oil (30 mg, 66% yield) after flash chromatography (petroleum ether/ethyl acetate = 70/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm=8.03 – 7.69 (m, 2H), 7.55 – 7.44 (m, 1H), 7.39 (dd, *J* = 8.3, 7.0 Hz, 2H), 3.72 (s, 2H), 2.60 (q, *J* = 7.5 Hz, 2H), 1.34 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=194.0, 134.2, 132.1, 127.7, 127.6, 25.2, 18.2, 14.1.

Spectral data are in good agreement with literature values.⁴



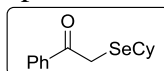
2-(Benzylselanyl)-1-phenylethan-1-one (4c). Compound **4c** was prepared according to the general procedure and isolated as a yellow solid (36 mg, 63% yield) after flash chromatography (petroleum ether/ethyl acetate = 40/1).

mp = 82-84 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.89 – 7.82 (m, 2H), 7.53 – 7.47 (m, 1H), 7.42 – 7.36 (m, 2H), 7.31 – 7.27 (m, 2H), 7.26 – 7.21 (m, 2H), 7.18 – 7.15 (m, 1H), 3.80 (s, 2H), 3.64 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 194.0, 137.2, 134.3, 132.2, 128.2, 127.7, 127.6, 127.5, 126.0, 27.6, 25.5.

Spectral data are in good agreement with literature values.⁹

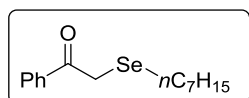


2-(Cyclohexylselanyl)-1-phenylethan-1-one (4d). Compound **4d** was prepared according to the general procedure and isolated as an oil (32 mg, 57% yield) after flash chromatography (petroleum ether/ethyl acetate = 120/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm=7.94 – 7.80 (m, 2H), 7.57 – 7.45 (m, 1H), 7.41 – 7.37 (m, 2H), 3.74 (s, 2H), 3.00 (tt, *J* = 10.6, 3.7 Hz, 1H), 2.06 – 1.88 (m, 2H), 1.68 – 1.63 (m, 2H), 1.48 – 1.37 (m, 2H), 1.36 – 1.21 (m, 4H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 194.6, 134.3, 132.1, 127.7, 127.6, 39.8, 32.9, 28.7, 28.6, 25.7, 24.8, 24.7.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₄H₁₈OSe 283.05956; Found 283.05908.

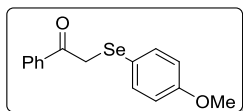


2-(Heptylselanyl)-1-phenylethan-1-one (4e). Compound **4e** was prepared according to the general procedure and isolated as an oil (36 mg, 60% yield) after flash chromatography (petroleum ether/ethyl acetate = 100/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm=7.96 – 7.82 (m, 2H), 7.56 – 7.44 (m, 1H), 7.41 – 7.37 (m, 2H), 3.70 (s, 2H), 2.60 (t, *J* = 7.5 Hz, 2H), 1.71 – 1.48 (m, 3H), 1.33 – 1.13 (m, 7H), 0.80 (t, *J* = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=194.0, 134.2, 132.1, 127.7, 127.6, 30.7, 28.8, 28.7, 27.7, 25.5, 24.8, 21.6, 13.1.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₂₂OSe : 299.09086; Found : 299.09042.

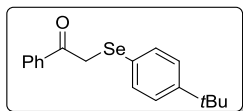


2-((4-Methoxyphenyl)selanyl)-1-phenylethan-1-one (4f). Compound **4f** was prepared according to the general procedure and isolated as an oil (40 mg, 65% yield) after flash chromatography (petroleum ether/ethyl acetate = 25/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.79 (d, *J* = 8.7 Hz, 2H), 7.56 – 7.41 (m, 1H), 7.38 – 7.28 (m, 4H), 6.73 (d, *J* = 8.7 Hz, 2H), 3.99 (s, 2H), 3.72 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 194.0, 159.1, 135.9, 134.5, 132.1, 127.7, 127.5, 117.8, 113.9, 54.6, 32.3.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₅O₂Se 307.0232; Found 307.0242.

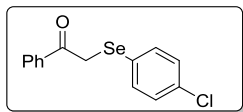


2-((4-*tert*-Butyl)phenyl)selanyl)-1-phenylethan-1-one (4g). Compound **4g** was prepared according to the general procedure and isolated as an oil (48 mg, 72% yield) after flash chromatography (petroleum ether/ethyl acetate = 50/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm=7.78 (d, *J* = 8.0 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.41 – 7.31 (m, 4H), 7.21 (d, *J* = 8.0 Hz, 2H), 4.07 (s, 2H), 1.23 (s, 9H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 194.2, 150.4, 134.5, 133.1, 132.2, 127.7, 127.5, 125.3, 124.4, 33.6, 31.8, 30.2.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₈H₂₁OSe 333.0752; Found 333.0761.



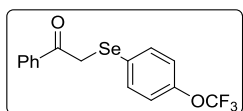
2-((4-Chlorophenyl)selanyl)-1-phenylethan-1-one (4h). Compound **4h** was prepared according to the general procedure and isolated as a white solid (51 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 65/1).

mp = 52-54 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm=7.80 (d, *J* = 8.0 Hz, 2H), 7.53 – 7.47 (m, 1H), 7.40 – 7.33 (m, 4H), 7.16 (d, *J* = 8.0 Hz, 2H), 4.08 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=193.6, 134.4, 134.3, 133.5, 132.4, 128.4, 127.6, 125.9, 31.8.

Spectral data are in good agreement with literature values.¹⁰



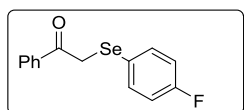
1-Phenyl-2-((4-(trifluoromethoxy)phenyl)selanyl)ethan-1-one (4i). Compound **4i** was prepared according to the general procedure and isolated as an oil (50 mg, 69% yield) after flash chromatography (petroleum ether/ethyl acetate = 15/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.79 (d, *J* = 8.3 Hz, 2H), 7.51 – 7.44 (m, 3H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.04 (d, *J* = 8.3 Hz, 2H), 4.10 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=193.7, 134.7, 134.3, 132.4, 127.7, 127.6, 126.0, 120.6, 120.4 (q, *J* = 249.8 Hz), 31.8.

¹⁹F NMR (376 MHz, CDCl₃) δ/ppm=-57.84.

HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₂F₃O₂Se 360.9949; Found 360.9954.



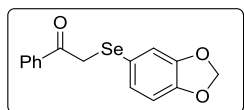
2-((4-Fluorophenyl)selanyl)-1-phenylethan-1-one (4j). Compound **4j** was prepared according to the general procedure and isolated as an oil (37 mg, 63% yield) after flash chromatography (petroleum ether/ethyl acetate = 40/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.90 – 7.66 (m, 2H), 7.52 – 7.46 (m, 1H), 7.45 – 7.40 (m, 2H), 7.36 (dd, *J* = 8.4, 7.1 Hz, 2H), 6.89 (t, *J* = 8.7 Hz, 2H), 4.04 (s, 1H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=193.8, 161.97 (d, *J* = 248.6 Hz), 135.8 (d, *J* = 8.1 Hz), 134.3, 132.3, 127.6 (d, *J* = 3.1 Hz), 122.2, 121.1, 115.4 (d, *J* = 21.6 Hz), 32.0.

¹⁹F NMR (376 MHz, CDCl₃) δ/ppm=-112.7.

Spectral data are in good agreement with literature values.¹¹

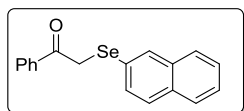


2-(Benzo[*d*][1,3]dioxol-5-ylselanyl)-1-phenylethan-1-one (4k). Compound **4k** was prepared according to the general procedure and isolated as an oil (40 mg, 63% yield) after flash chromatography (petroleum ether/ethyl acetate = 30/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.80 (d, *J* = 7.1 Hz, 2H), 7.55 – 7.44 (m, 1H), 7.37 (dd, *J* = 8.4, 7.1 Hz, 2H), 7.07 – 6.73 (m, 2H), 6.64 (d, *J* = 8.5 Hz, 1H), 5.90 (s, 2H), 4.01 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 193.9, 147.3, 147.0, 134.4, 132.2, 128.4, 127.7, 127.6, 118.7, 114.4, 108.1, 100.3, 32.5.

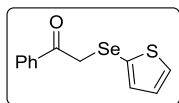
HRMS (ESI/Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₃O₃Se 321.0024; Found 321.0032.



2-(Naphthalen-2-ylselanyl)-1-phenylethan-1-one (4l). Compound **4l** was prepared according to the general procedure and isolated as an oil (49 mg, 75% yield) after flash chromatography (petroleum ether/ethyl acetate = 50/1).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 8.29 (dt, *J* = 7.6, 1.0 Hz, 1H), 7.83 – 7.66 (m, 5H), 7.52 – 7.38 (m, 3H), 7.33 – 7.23 (m, 3H), 4.10 (s, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm = 195.2, 135.5, 134.9, 134.4, 134.1, 133.2, 129.8, 128.8, 128.7, 128.5, 128.2, 127.7, 127.1, 126.4, 125.8, 32.7.
Spectral data are in good agreement with literature values.¹²

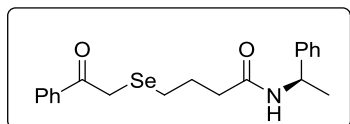


1-Phenyl-2-(thiophen-2-ylselanyl)ethan-1-one (4m). Compound **4m** was prepared according to the general procedure and isolated as an oil (45 mg, 80% yield) after flash chromatography (petroleum ether/ethyl acetate = 20/1).

^1H NMR (400 MHz, CDCl_3) δ/ppm = 7.78 (dd, J = 8.3, 1.1 Hz, 2H), 7.52 – 7.46 (m, 1H), 7.40 – 7.31 (m, 3H), 7.08 (dd, J = 3.5, 1.1 Hz, 1H), 6.88 (dd, J = 5.3, 3.5 Hz, 1H), 4.05 (s, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm = 193.7, 136.2, 134.4, 132.3, 131.1, 127.7, 127.6, 127.2, 121.2, 34.8.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{12}\text{H}_{11}\text{OSSe}$ 282.9690; Found 282.9688.



(R)-4-((2-Oxo-2-phenylethyl)selanyl)-N-(1-phenylethyl)butanamide (4n).

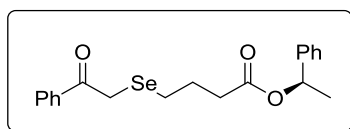
Compound **4n** was prepared according to the general procedure and isolated as an oil (40mg, 52% yield) after flash chromatography (petroleum ether/ethyl acetate = 2/1).

$[\alpha]_D^{25}$ = + 31.6, $c=0.038$ g/100 mL, CHCl_3 .

^1H NMR (400 MHz, CDCl_3) δ/ppm = 7.88 – 7.82 (m, 2H), 7.54 – 7.45 (m, 1H), 7.39 (dd, J = 8.4, 7.0 Hz, 2H), 7.30 – 7.16 (m, 5H), 5.88 (d, J = 8.0 Hz, 1H), 5.10 – 5.02 (m, 1H), 3.69 (s, 2H), 2.62 (td, J = 7.2, 1.5 Hz, 2H), 2.20 (t, J = 7.3 Hz, 2H), 2.04 – 1.90 (m, 2H), 1.42 (d, J = 7.0 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm = 194.2, 170.0, 142.2, 134.1, 132.3, 127.68, 127.66, 127.64, 126.3, 125.2, 47.7, 35.1, 25.6, 24.6, 24.2, 20.8.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for : $\text{C}_{20}\text{H}_{23}\text{NO}_2\text{Se}$ 390.09668; Found 390.09631.



(R)-1-Phenylethyl 4-((2-oxo-2-phenylethyl)selanyl)butanoate (4o). Compound **4o** was prepared according to the general procedure and isolated as an oil (44mg, 57% yield) after flash chromatography (petroleum ether/ethyl acetate = 20/1).

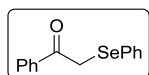
$[\alpha]_D^{25}$ = + 34.0, $c=0.11$ g/100 mL, CHCl_3 .

^1H NMR (400 MHz, CDCl_3) δ/ppm = 7.90 – 7.83 (m, 2H), 7.57 – 7.42 (m, 1H), 7.38 (dd, J = 8.4, 7.0 Hz, 2H), 7.30 – 7.17 (m, 5H), 5.81 (q, J = 6.6 Hz, 1H), 3.68 (s, 2H), 2.60 (t, J = 7.3 Hz, 2H), 2.36 (td, J = 7.3, 1.9 Hz, 2H), 2.06 – 1.82 (m, 2H), 1.45 (d, J

= 6.6 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm = 193.9, 171.0, 140.6, 134.1, 132.2, 127.7, 127.6, 127.5, 126.8, 125.0, 71.4, 33.2, 25.4, 23.9, 23.6, 21.2.

HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for : $\text{C}_{20}\text{H}_{22}\text{O}_3\text{Se}$ 391.08069; Found 391.08035.

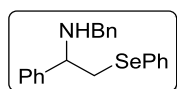


1-Phenyl-2-(phenylselanyl)ethan-1-ol (5). To ketone **3a** (55 mg, 0.2 mmol) in MeOH (10 mL) at room temperature was added NaBH_4 (15 mg, 0.4 mmol), and the mixture was stirred overnight. The resulting mixture was treated with water (10 mL), 1 M NaOH (10 mL) and then was extracted with EtOAc (3×20 mL). The combined organic layer was dried over Na_2SO_4 and concentrated. The resulting residue was chromatographed (petroleum ether/ethyl acetate = 5/1) to give alcohol **5** (51 mg, 93%) as an oil.

^1H NMR (400 MHz, CDCl_3) δ/ppm = 7.48 – 7.45 (m, 2H), 7.24 – 7.13 (m, 4H), 7.26 – 7.24 (m, 4H), 4.66 (dd, J = 9.4, 3.7 Hz, 1H), 3.22 (dd, J = 12.8, 3.7 Hz, 1H), 3.06 (dd, J = 12.8, 9.4 Hz, 1H), 2.75 (brs, 1H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm = 141.4, 132.1, 128.2, 128.1, 127.5, 126.9, 126.4, 124.8, 71.2, 37.4.

Spectral data are in good agreement with literature values.¹³



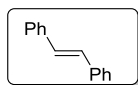
N-Benzyl-1-phenyl-2-(phenylselanyl)ethan-1-amine (6). Benzylamine (86 mg, 0.8 mmol) was added to ketone **3a** (55 mg, 0.2 mmol) in ether (10 ml) at 0 °C under argon. A solution of titanium tetrachloride (28 mg, 0.15 mmol) in heptane (2 ml) was then slowly introduced. The mixture was stirred for 30 min at 0 °C and then for 3 h at room temperature. The titanium salts were filtered and rinsed with ether. The resulting mixture was concentrated under reduced pressure with a rotary evaporator to give imine, which was used in the subsequent step without further purification.

To the resulting imine in ethanol (10 ml) at -78 °C under argon, were added successively sodium cyanoborohydride (12 mg, 0.2 mmol) and acetic acid (12 mg, 0.2 mmol). The reaction mixture was stirred for 1 h at -78 °C and quenched with water (10 ml). The resulting mixture was extracted with EtOAc (3×20 mL), and the combined extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by silica gel chromatography (petroleum ether/ethyl acetate = 30/1) to give compound **6** (53 mg, 73%) as an oil.

^1H NMR (400 MHz, CDCl_3) δ/ppm = 7.50–7.42 (m, 2H), 7.40–7.32 (m, 13H), 3.79 (dd, J = 9.7, 4.4 Hz, 1H), 3.69 (d, J = 13.3 Hz, 1H), 3.46 (d, J = 13.3 Hz, 1H), 3.21 (dd, J = 4.4, 12.3 Hz, 1H), 3.10 (dd, J = 9.7, 12.3 Hz, 1H), 2.35 (brs, 1H).

^{13}C NMR (100 MHz, CDCl_3) δ/ppm = 144.1, 141.3, 134.4, 130.7, 130.1, 129.7, 129.6, 129.2, 128.6, 128.2, 128.1, 128.0, 62.0, 52.5, 38.1.

Spectral data are in good agreement with literature values.¹⁴

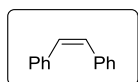


(E)-1,2-Diphenylethene (8).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.55 – 7.40 (m, 4H), 7.28 (t, *J* = 7.7 Hz, 4H), 7.23 – 7.10 (m, 2H), 7.04 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 136.3, 127.6, 126.6, 125.5.

Spectral data are in good agreement with literature values.¹⁵

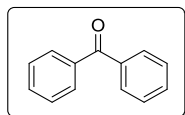


(Z)-1,2-Diphenylethene (9).

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.35 – 6.88 (m, 10H), 6.52 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm=136.2, 129.2, 127.8, 127.2, 126.0.

Spectral data are in good agreement with literature values.¹⁶



Benzophenone (11). To a 10 mL vessel with magnetic stir bar were added 0.2 mmol 1,1-diphenylethylene, 0.1 mmol Ph₂Se₂ and 2 mL of EtOAc. The reaction mixture was stirred and irradiated with a 24W household compact fluorescent lamp from a distance of 1 cm. After 20 h, the solvent was removed with a rotary evaporator. The crude product was purified by silica gel chromatography (petroleum ether/ethyl acetate = 10/1) to give compound **11** (16 mg, 43%) as an oil.

¹H NMR (400 MHz, CDCl₃) δ/ppm= 7.85 – 7.77 (m, 4H), 7.63 – 7.54 (m, 2H), 7.52 – 7.41 (m, 4H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm= 196.6, 196.7, 137.6, 132.5, 130.1, 128.3.

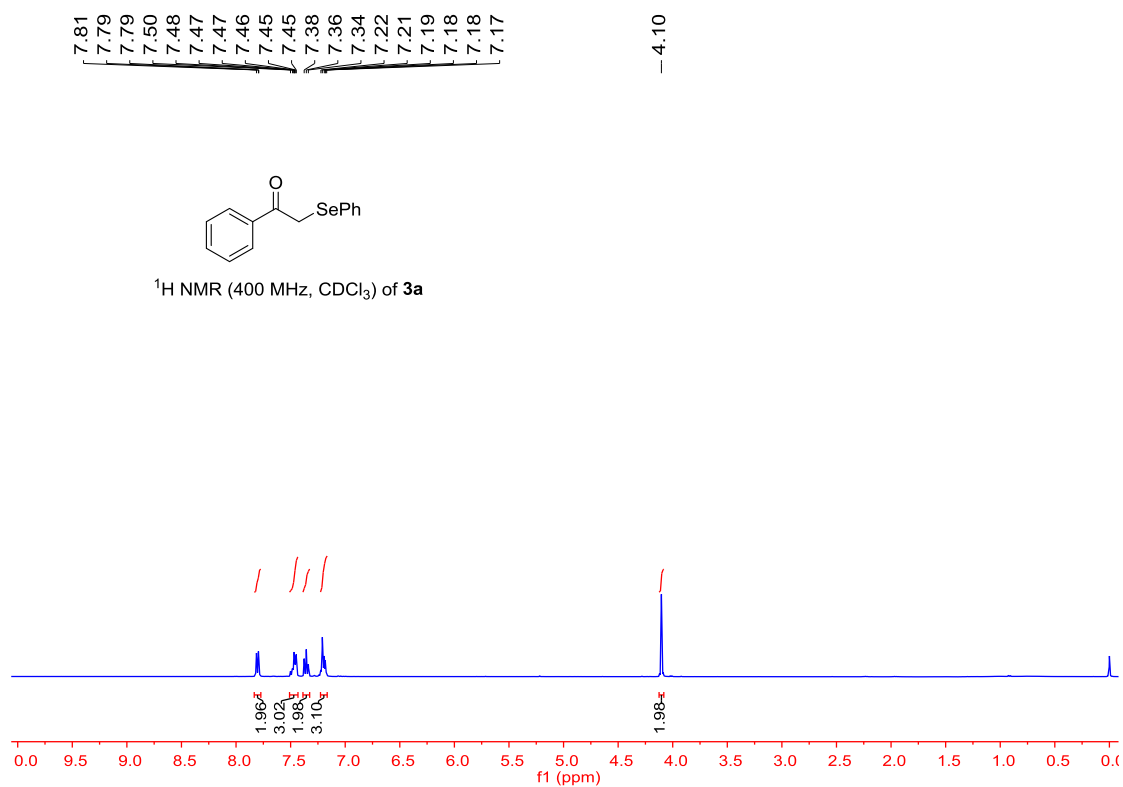
Spectral data are in good agreement with literature values.¹⁷

7. References

- (1) Singh, D.; Deobald, A. M.; Camargo, L. R. S.; Tabarelli, G.; Rodrigues, O. E. D.; Braga, A. L. *Org. Lett.* **2010**, *12*, 3288.
- (2) Nishiyama, Y.; Kawamatsu, H.; Funato, S.; Tokunaga, K.; Sonoda, N. *J. Org. Chem.* **2003**, *68*, 3599.
- (3) Bao, W. L.; Zhang, Y. M. *Synlett* **1996**, 1187.
- (4) Senatore, R.; Castoldi, L.; Ielo, L.; Holzer, W.; Pace, V. *Org. Lett.* **2018**, *20*, 2685.
- (5) Cao, Y.; Liu, J.; Liu, F.; Jiang, L.; Yi, W. *Org. Chem. Front.* **2019**, *6*, 825.
- (6) Torii, S.; Uneyama, K.; Handa, K. *Tetrahedron Lett.* **1980**, *21*, 1863.
- (7) Nishiyama, Y.; Koguma, Y.; Tanaka, T.; Umeda, R. *Molecules* **2009**, *14*, 3367.
- (8) Balasubramaniam, S.; Aidhen, I. S. *Synlett* **2011**, 1533.

- (9) Ouyang, Y.; Guo, C.; Qiu, R.; Li, N.; Chen, J.; Xu, X. *Youji Huaxue* **2015**, *35*, 731.
 (10) Beletskaya, I.; Sigeev, A.; Peregodov, A.; Petrovskii, P. *Chem. Lett.* **2005**, *34*, 1348.
 (11) Pitombo, L. R. M. *Chem. Ber.* **1959**, *92*, 745.
 (12) Amri, N.; Wirth, T. *Synthesis* **2020**, *52*, 1751.
 (13) Miniejew, C.; Outurquin, F.; Pannecoucke, X. *Tetrahedron* **2005**, *61*, 447.
 (14) Huang, B.; Li, Y.; Yang, C.; Xia, W. *Green Chem.* **2020**, *22*, 2804.
 (15) Ekebergh, A.; Begon, R.; Kann, N. *J. Org. Chem.* **2020**, *85*, 2966.
 (16) Xu, J.; Liu, N.; Lv, H.; He, C.; Liu, Z.; Shen, X.; Cheng, F.; Fan, B. *Green Chem.* **2020**, *22*, 2739.
 (17) Ma, S.; Zhou, T.; Li, G.; Szostak, M. *Adv. Synth. Catal.* **2020**, *362*, 1887.

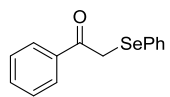
8. Copies of NMR spectra



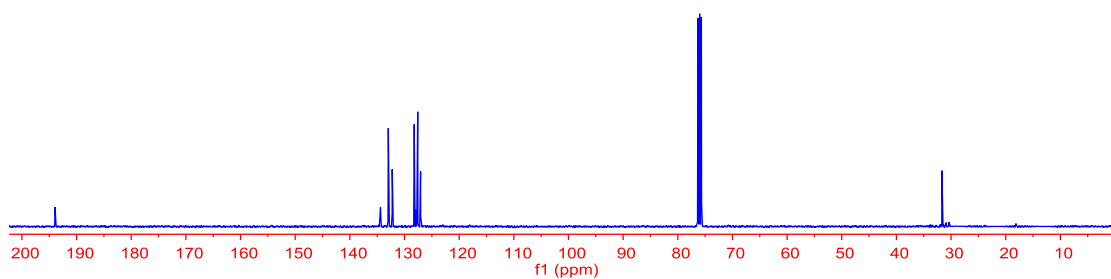
-193.9

134.4
133.0
132.2
128.2
128.0
127.7
127.6
127.1

-31.7



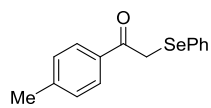
^{13}C NMR (100 MHz, CDCl_3) of **3a**



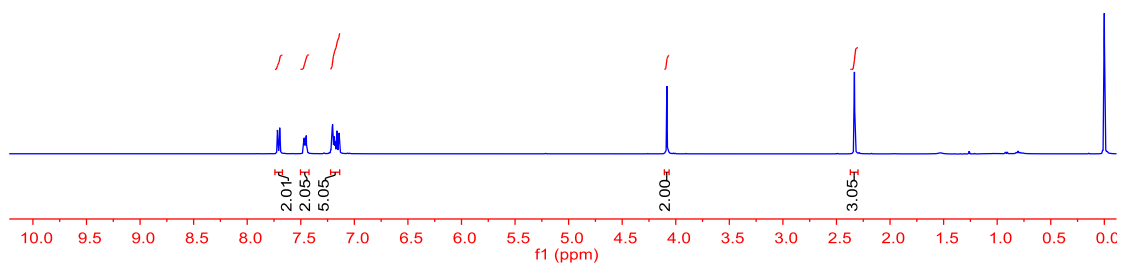
7.72
7.70
7.47
7.47
7.46
7.46
7.45
7.45
7.21
7.21
7.20
7.19
7.18
7.16
7.14

-4.08

-2.33



^1H NMR (400 MHz, CDCl_3) of **3b**

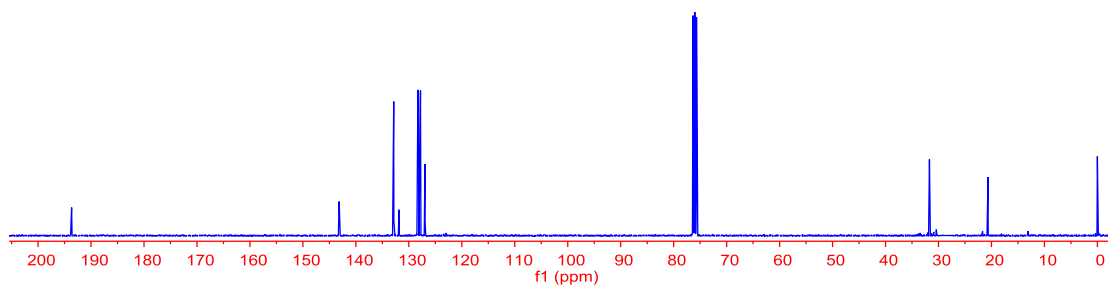
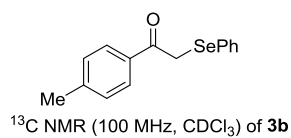


— 193.7

— 143.2
— 132.9
— 131.9
— 128.3
— 128.2
— 128.2
— 127.8
— 127.0

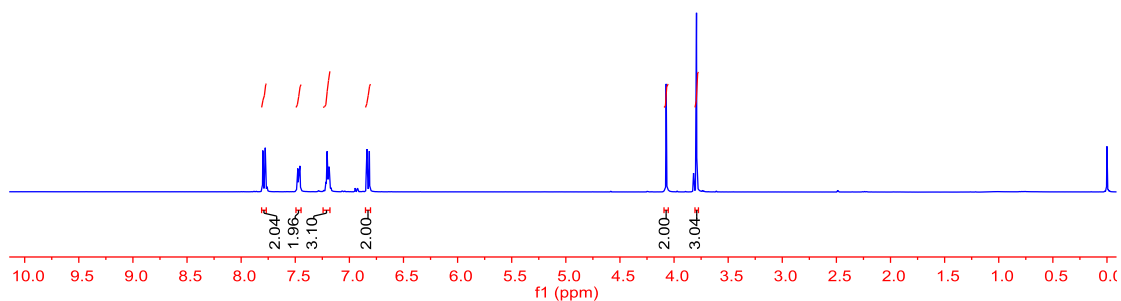
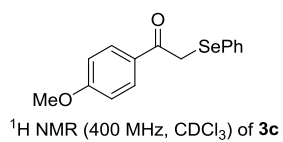
— 31.7

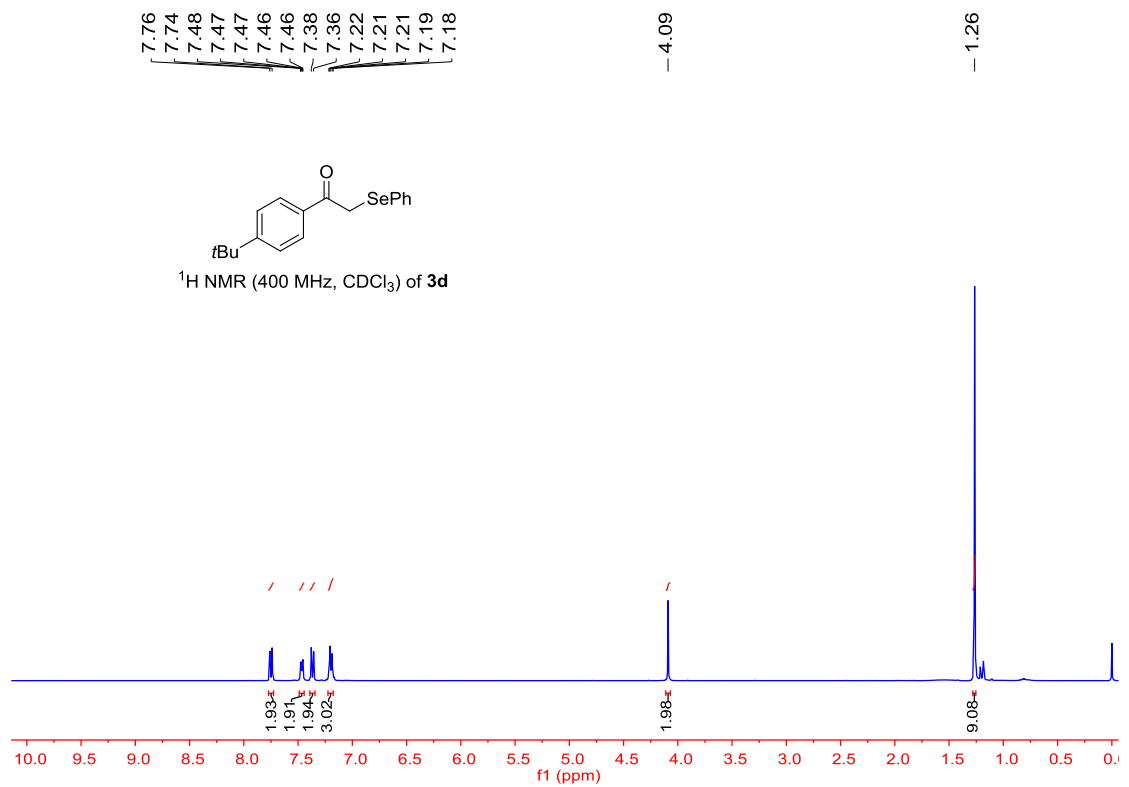
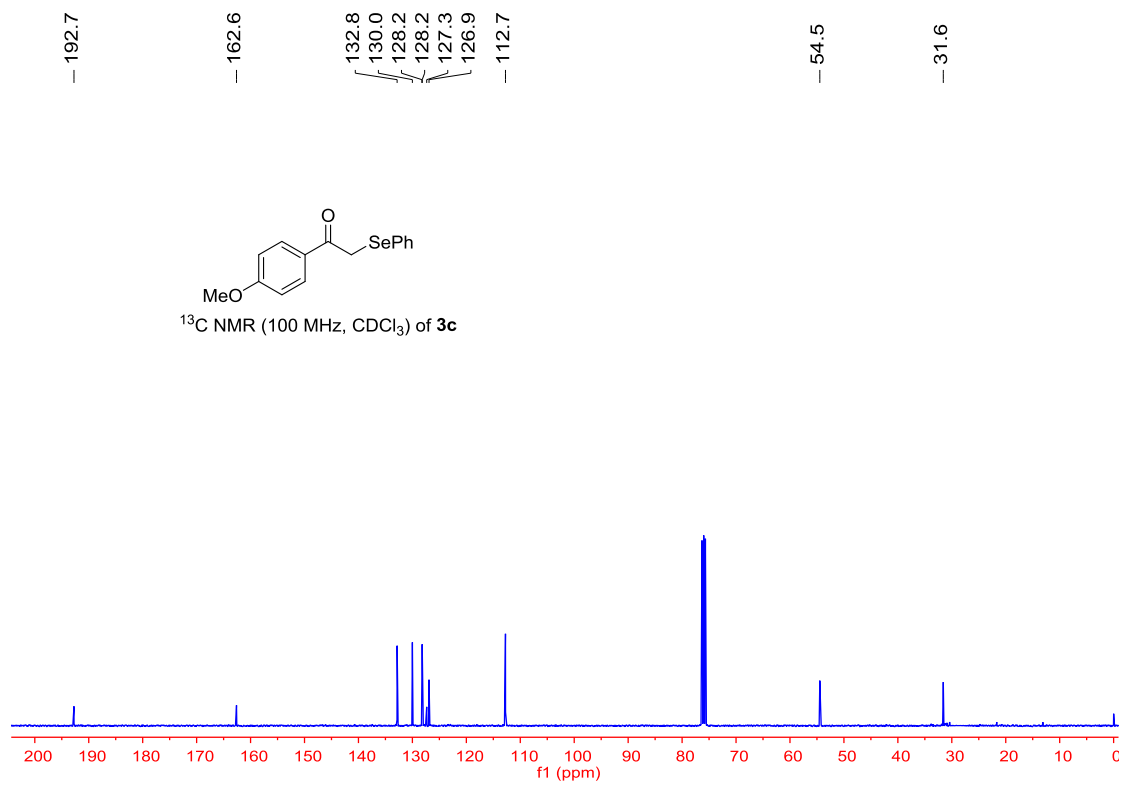
— 20.7

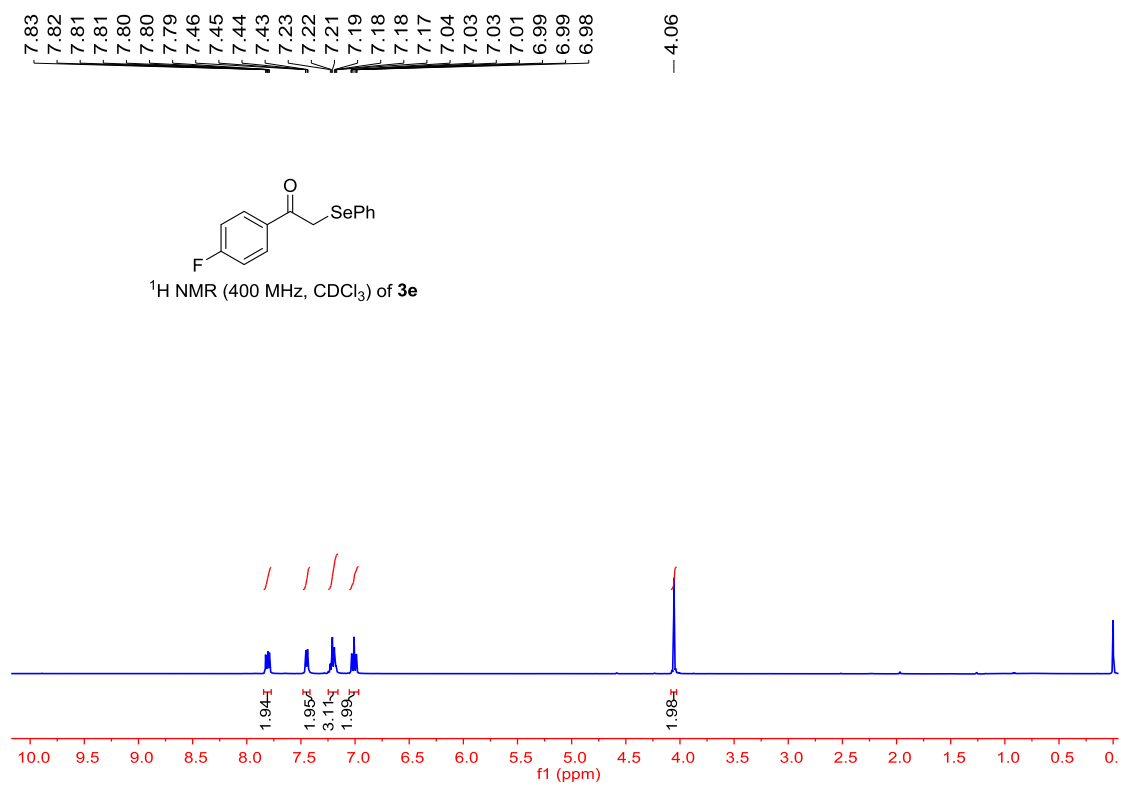
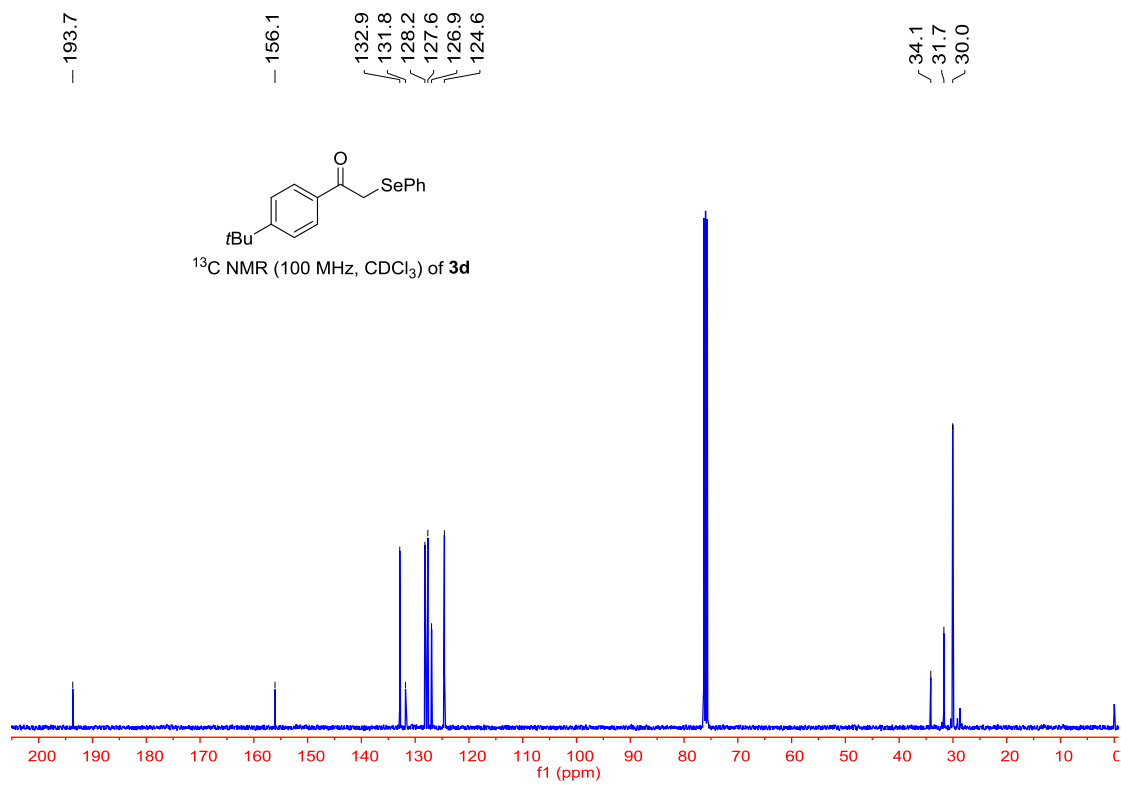


7.80
7.78
7.78
7.48
7.47
7.47
7.47
7.46
7.46
7.22
7.21
7.21
7.20
7.19
7.19
7.18
7.18
6.84
6.82

— 4.07
— 3.79





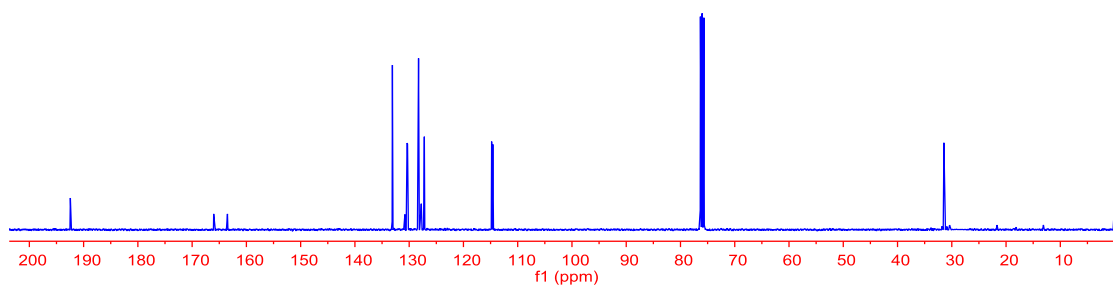
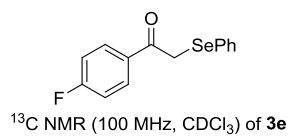


— 192.4

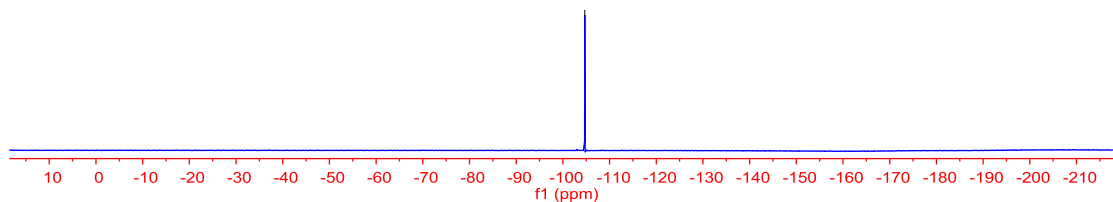
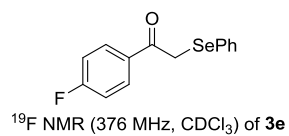
~ 166.0
~ 163.5

133.1
130.8
130.8
130.4
130.3
128.3
127.8
114.8
114.6

— 31.5

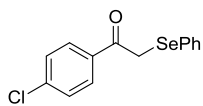


— -104.7

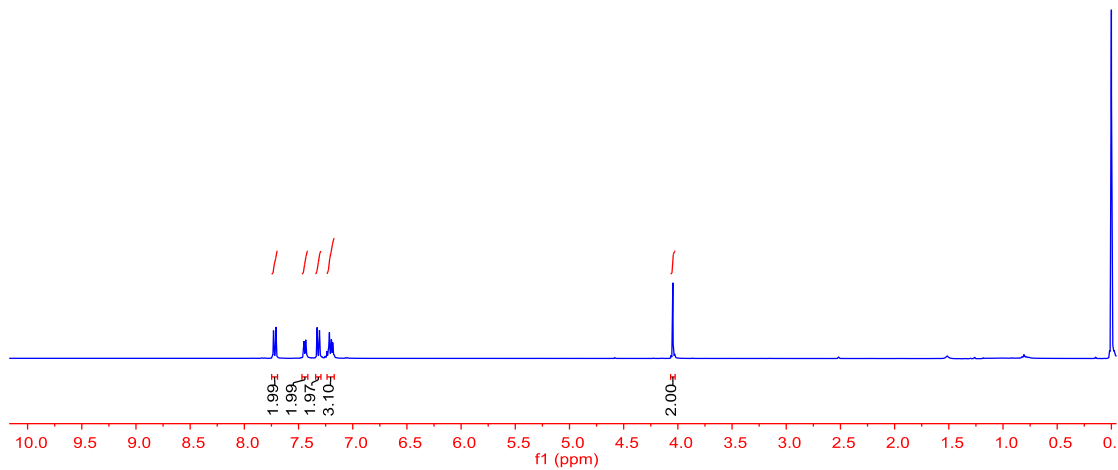


7.73
7.71
7.45
7.45
7.44
7.43
7.33
7.31
7.24
7.23
7.22
7.22
7.20
7.18
7.17

— 4.05



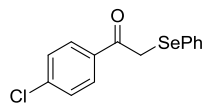
$^1\text{H NMR}$ (400 MHz, CDCl_3) of **3f**



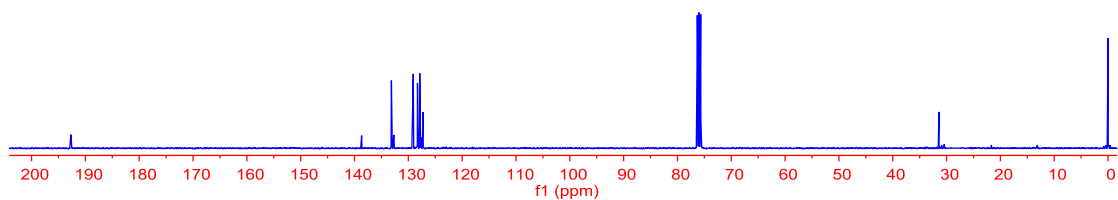
— 192.7

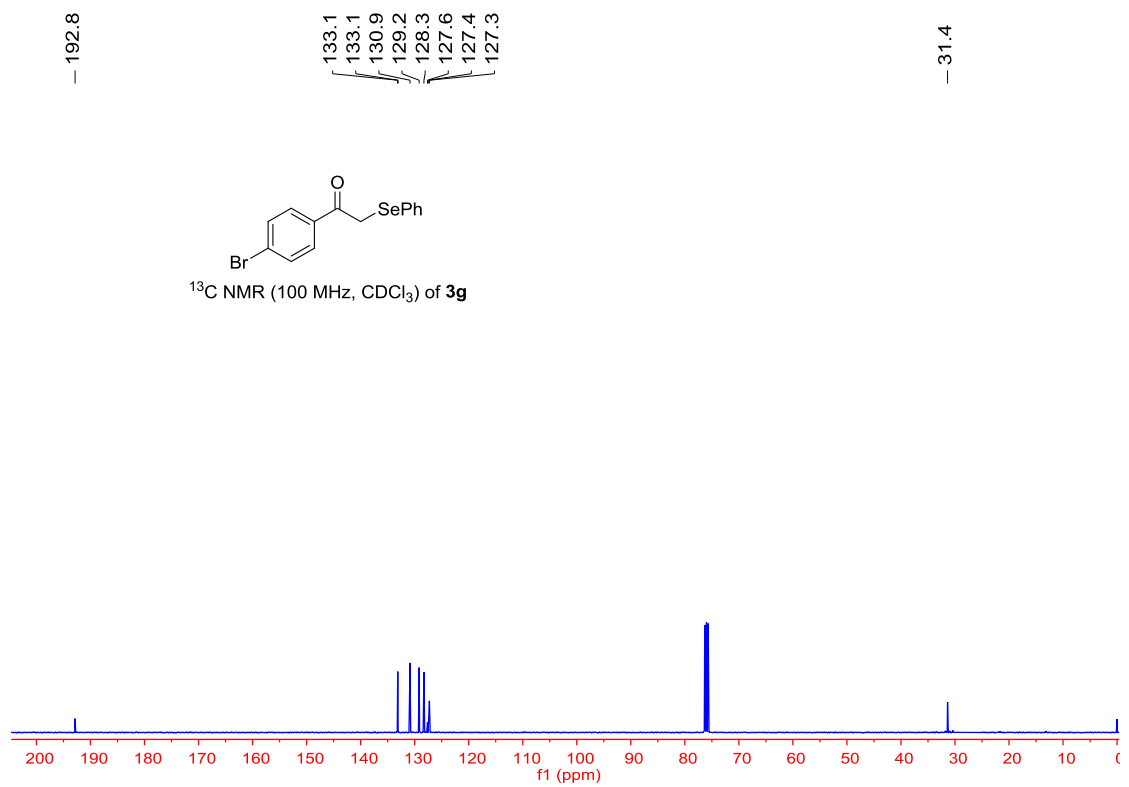
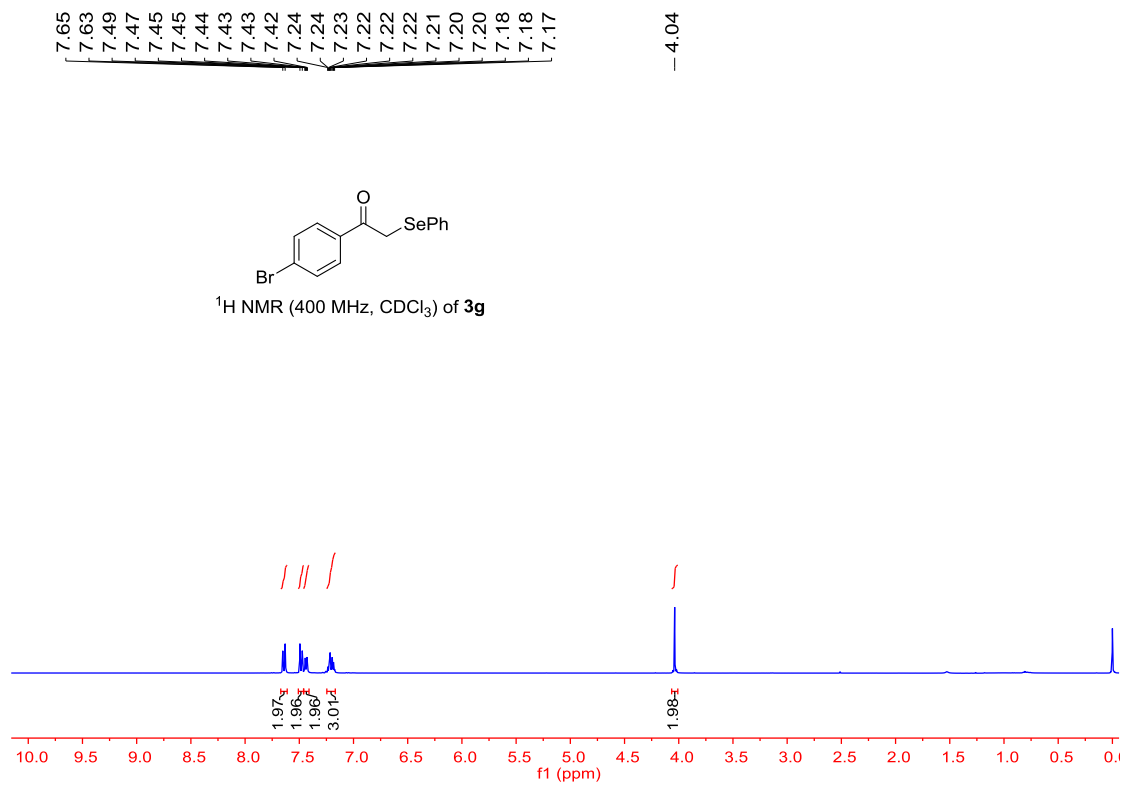
138.7
133.1
132.7
129.1
128.3
127.9
127.7
127.3

— 31.4



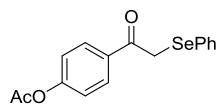
$^1\text{H NMR}$ (400 MHz, CDCl_3) of **3f**



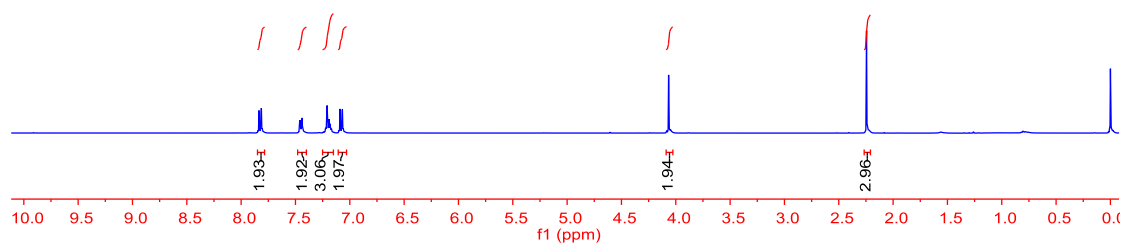


7.84
7.81
7.46
7.46
7.45
7.45
7.44
7.44
7.23
7.22
7.22
7.21
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7.20
7.20
7.19
7.19
7.18
7.18
7.17
7.09
7.07

— 4.07



¹H NMR (400 MHz, CDCl₃) of **3h**



— 192.7

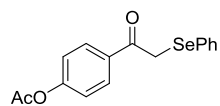
— 167.8

— 153.4

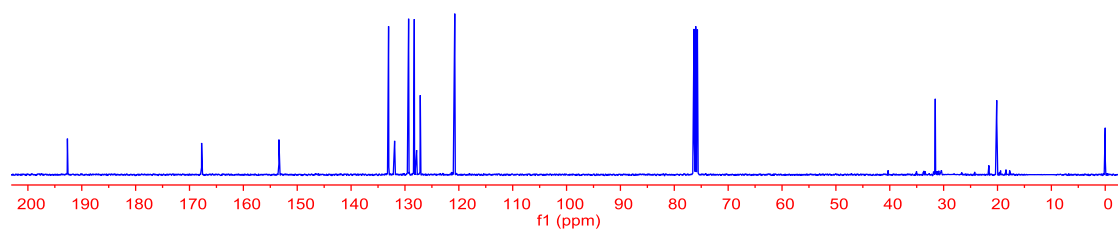
133.0
131.9
129.3
128.3
127.8
127.2
120.8

— 31.5

— 20.1

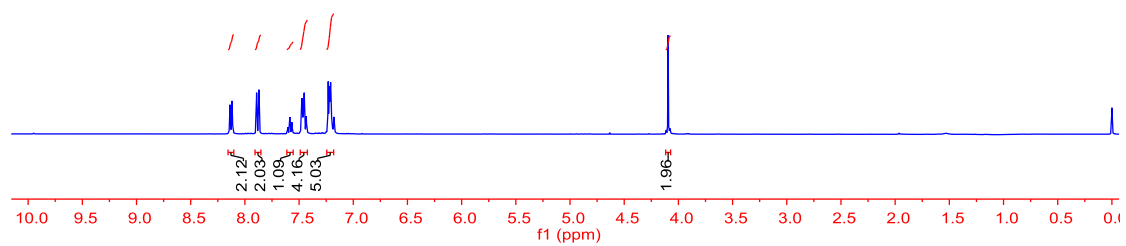


¹³C NMR (100 MHz, CDCl₃) of **3h**



8.14
8.12
7.89
7.87
7.86
7.60
7.59
7.57
7.57
7.48
7.48
7.47
7.46
7.46
7.45
7.43
7.43
7.23
7.22
7.21
7.21
7.20
7.19

— 4.10



— 192.7

— 163.5

— 153.7

133.1

132.9

132.0

129.4

129.2

128.3

128.0

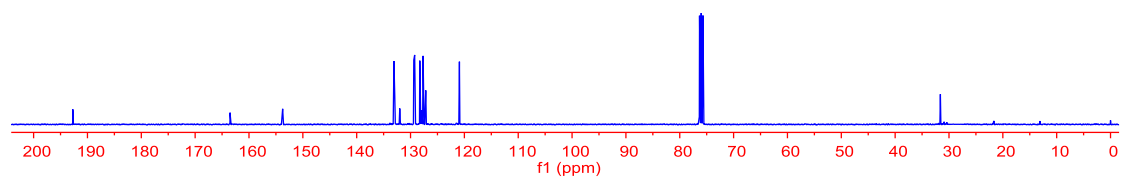
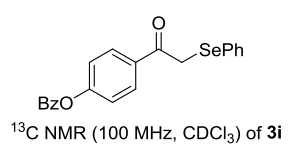
127.9

127.7

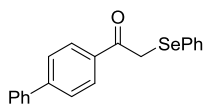
127.2

120.9

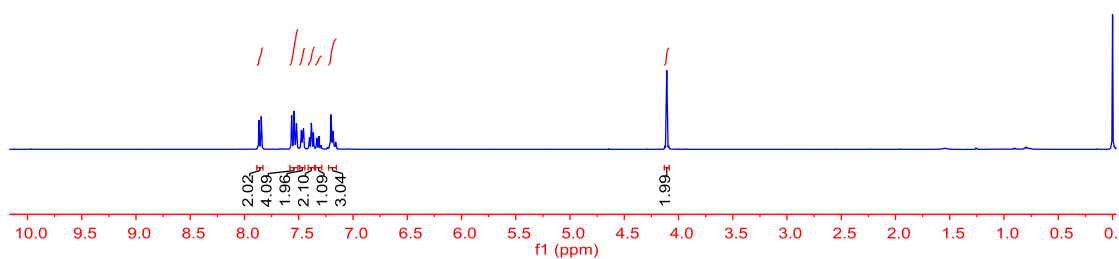
— 31.6



7.87
7.86
7.85
7.85
7.57
7.56
7.55
7.54
7.53
7.52
7.48
7.48
7.47
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7.46
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7.33
7.33
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7.20
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7.17
7.16
4.11



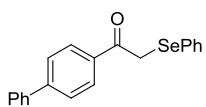
¹H NMR (400 MHz, CDCl₃) of 3j



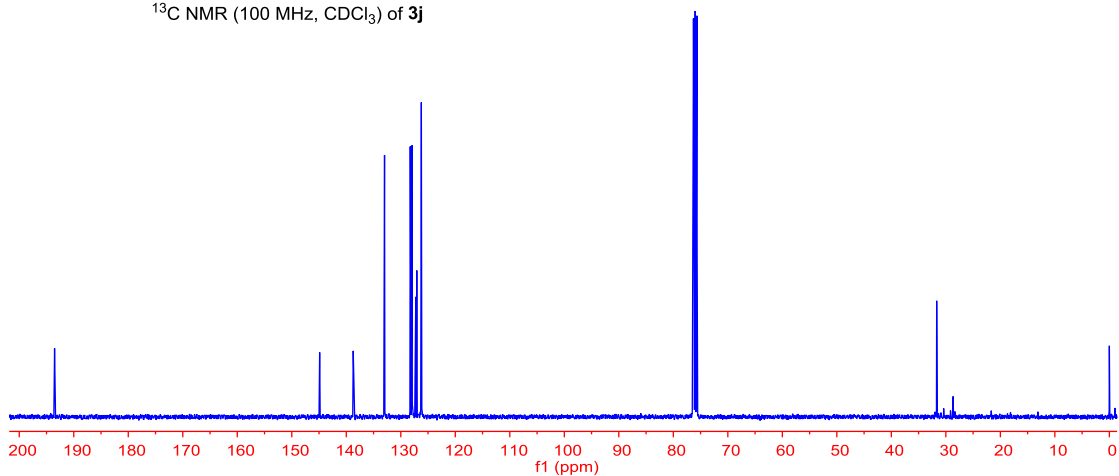
-193.5

144.9
138.7
133.0
133.0
132.9
128.3
128.2
128.0
127.9
127.3
127.1
126.2
126.2

-31.7

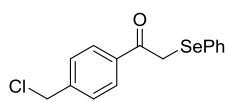


¹³C NMR (100 MHz, CDCl₃) of 3j

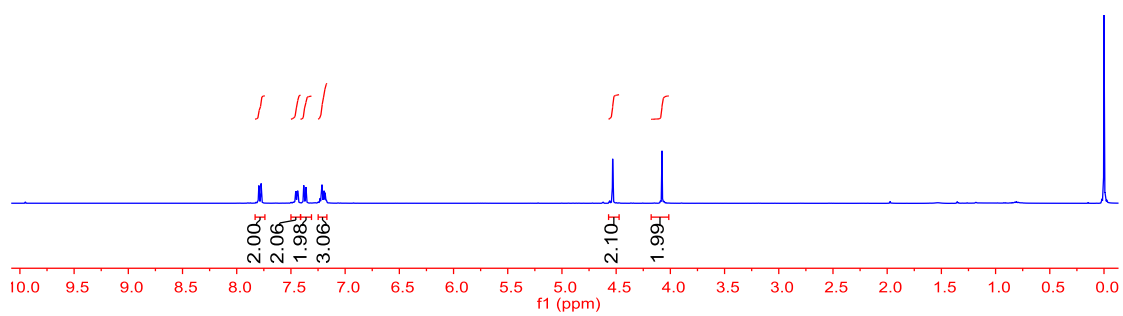


7.80
7.78
7.46
7.46
7.44
7.44
7.38
7.36
7.23
7.23
7.22
7.22
7.21
7.21
7.21
7.21
7.20
7.20
7.19
7.19
7.19
7.18
7.18
7.17

— 4.53
— 4.08



^1H NMR (400 MHz, CDCl_3) of **3k**

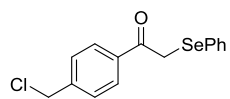


— 193.2

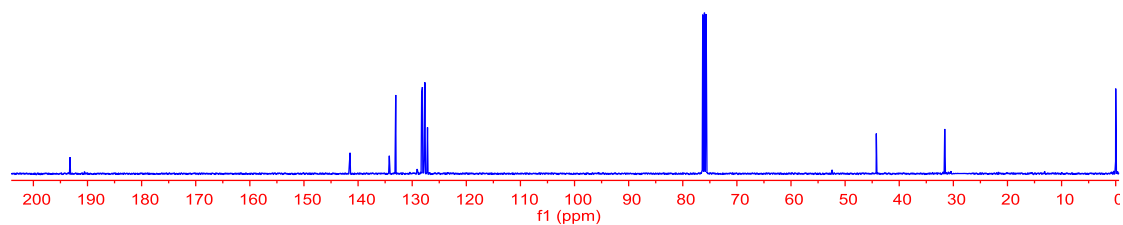
141.5
134.2
133.0
128.3
128.1
128.1
127.8
127.7
127.2

— 44.2

— 31.6

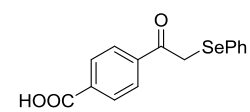


^{13}C NMR (100 MHz, CDCl_3) of **3k**

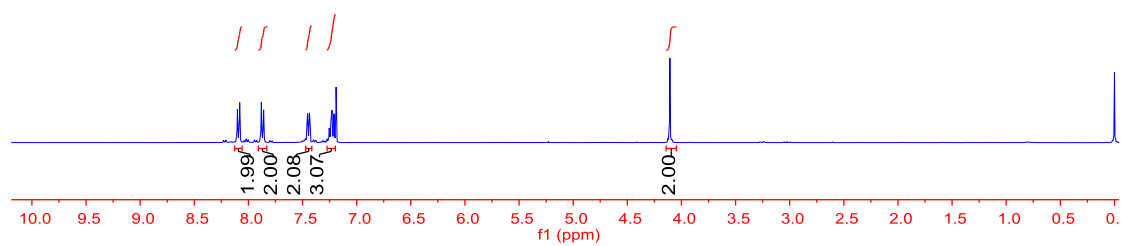


8.10
7.88
7.86
7.46
7.45
7.44
7.43
7.25
7.24
7.24
7.23
7.23
7.22
7.21
7.19

- 4.11



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

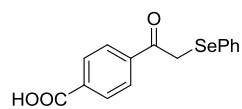


- 193.1

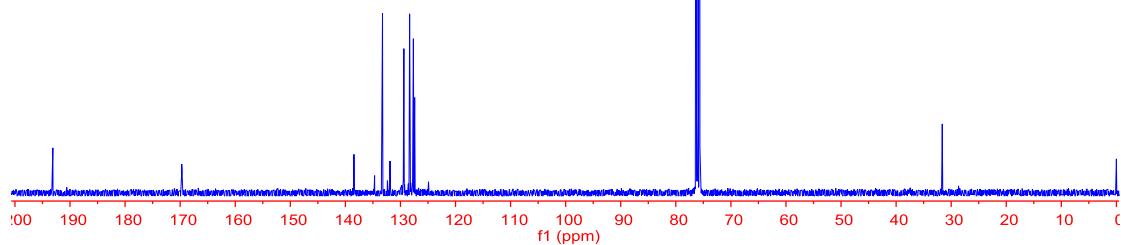
- 169.7

138.4
133.3
131.9
129.4
128.3
127.7
127.4
127.4

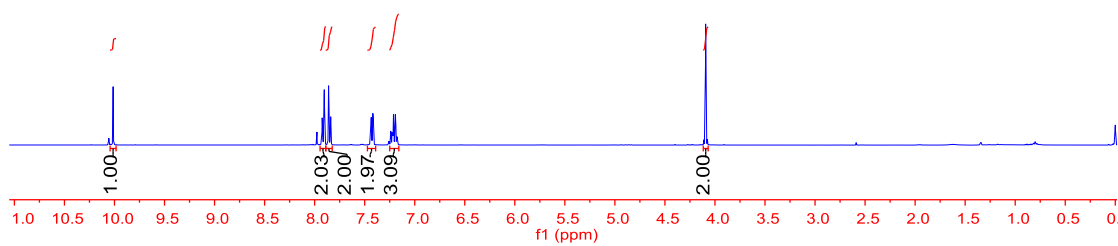
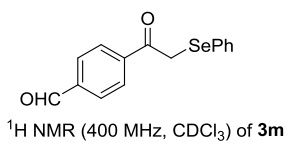
- 31.6



$^{13}\text{C NMR}$ (100 MHz, CDCl_3) of **3I**



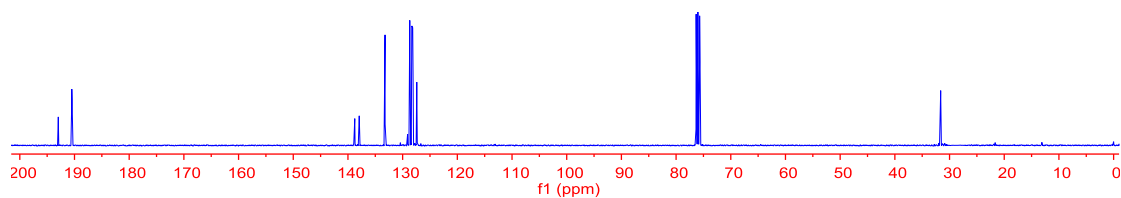
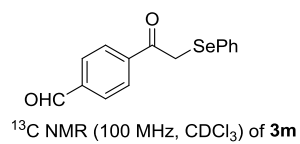
10.01
7.92
7.91
7.90
7.86
7.84
7.44
7.43
7.42
7.41
7.24
7.23
7.22
7.21
7.21
7.21
7.20
7.19
7.19
7.18
7.17
4.09



193.0
190.5

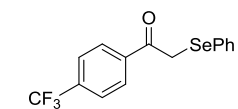
138.8
137.9
133.2
128.7
128.3
128.2
127.4
127.3

31.6

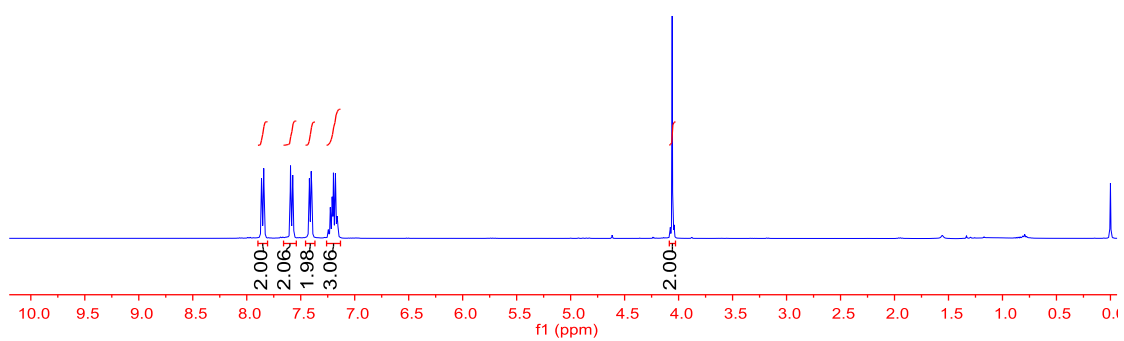


7.87
7.86
7.84
7.59
7.57
7.42
7.42
7.42
7.41
7.40
7.40
7.23
7.23
7.22
7.21
7.21
7.20
7.20
7.19
7.18
7.18
7.17
7.16
7.16

4.06



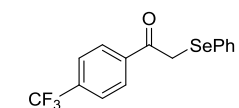
^1H NMR (400 MHz, CDCl_3) of **3n**



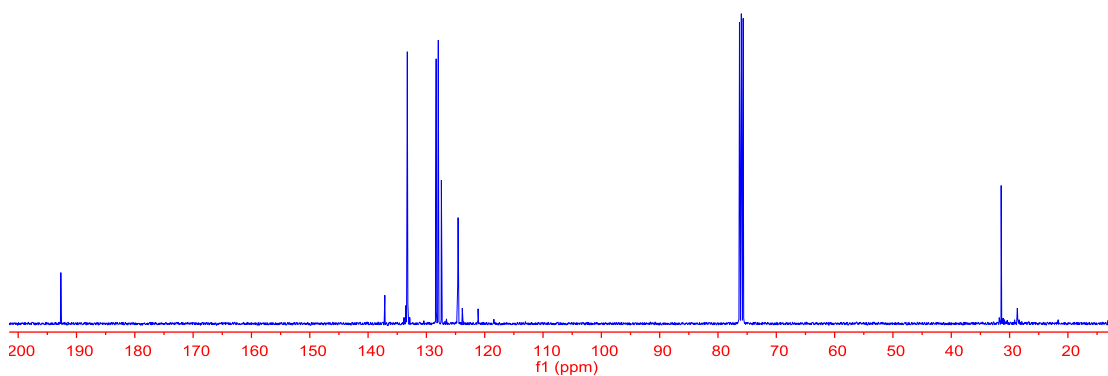
192.7

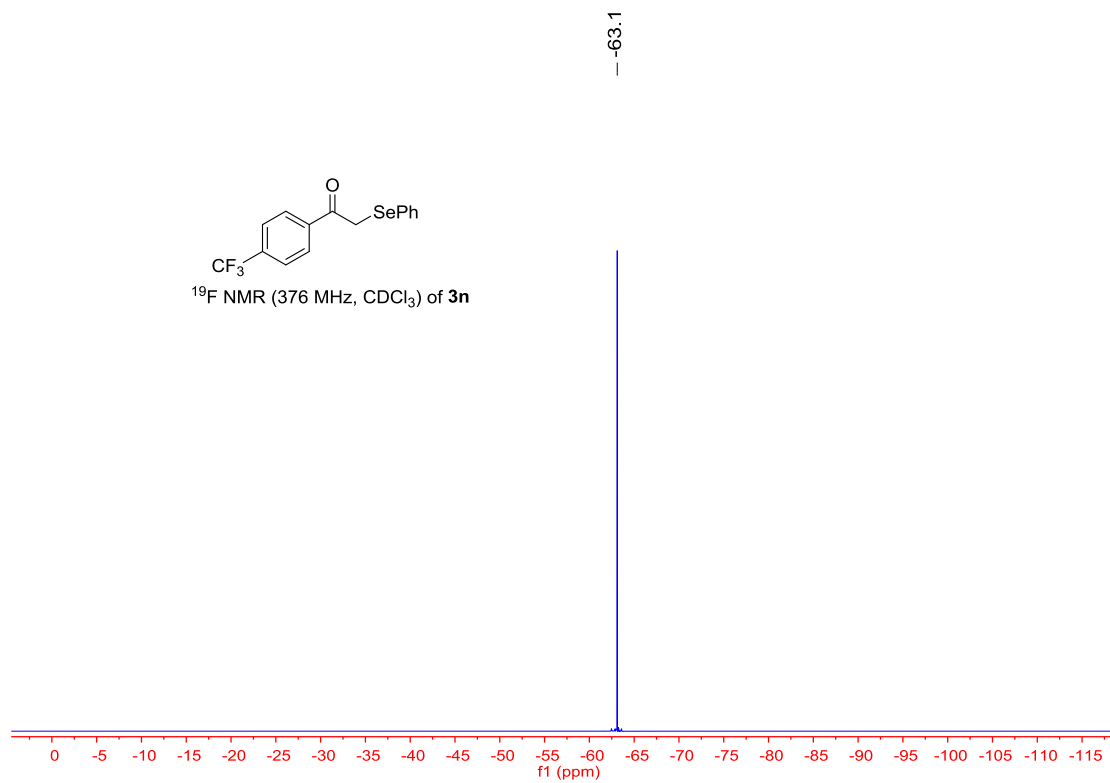
137.1
133.3
128.3
128.0
127.4
127.3
124.6
124.6
124.5
123.8
121.1

31.4

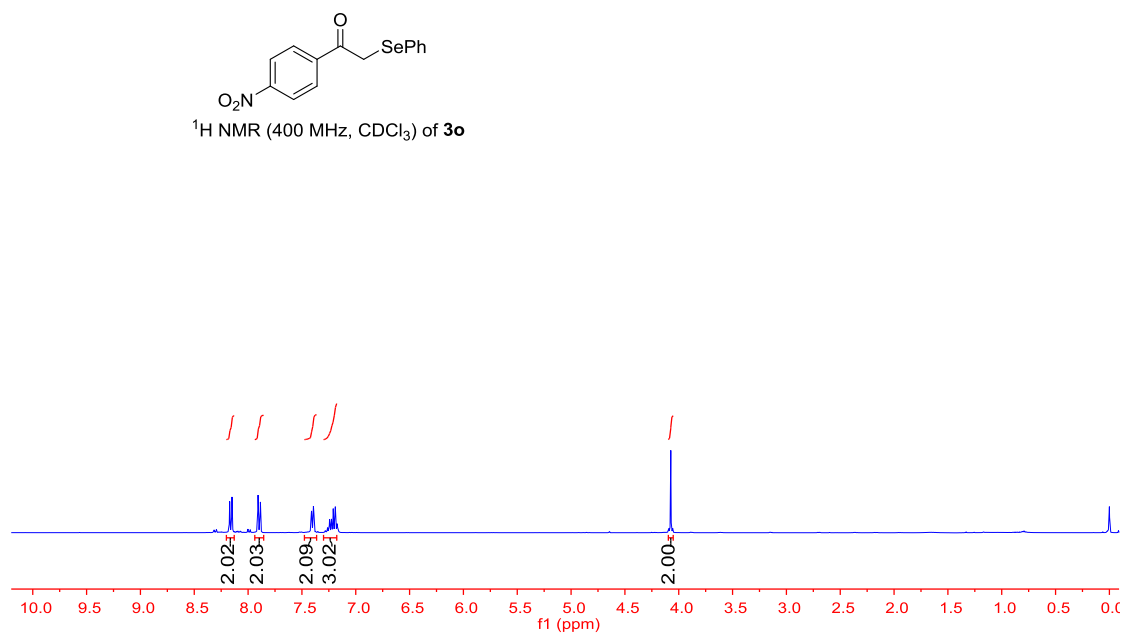


^{13}C NMR (100 MHz, CDCl_3) of **3n**





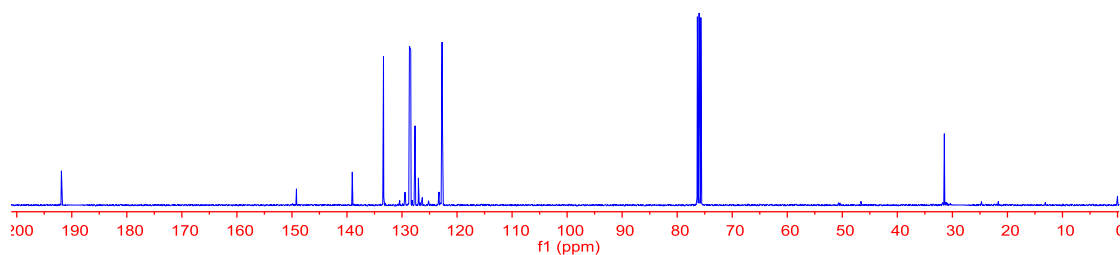
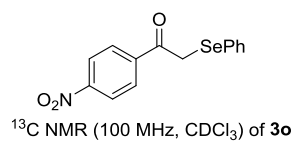
8.17
8.15
7.91
7.89
7.41
7.41
7.39
7.39
7.28
7.27
7.26
7.26
7.25
7.24
7.24
7.23
7.23
7.22
7.21
7.20
7.19
7.19
7.18
7.18
7.17
7.17
4.07



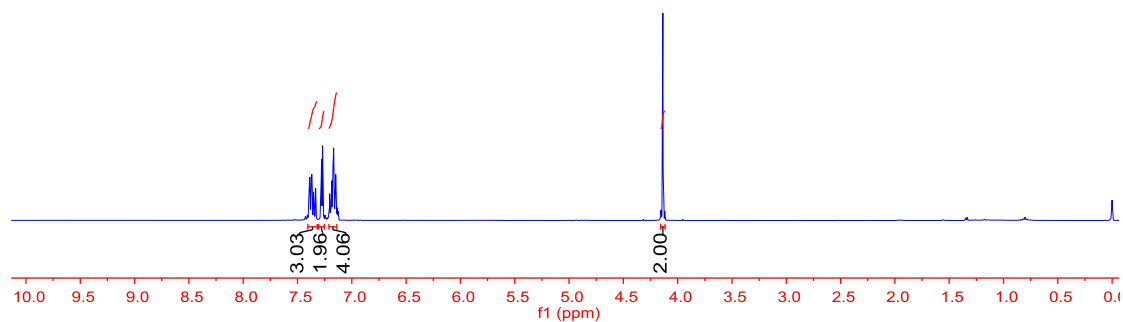
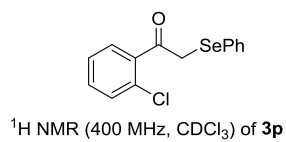
— 191.9

— 149.2
— 139.0
— 133.4
— 128.6
— 128.4
— 127.6
— 127.0
— 122.7

— 31.5



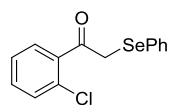
7.39
7.39
7.37
7.37
7.35
7.33
7.28
7.27
7.27
7.27
7.20
7.19
7.19
7.18
7.18
7.17
7.17
7.16
7.16
7.15
7.15
7.15
— 4.14



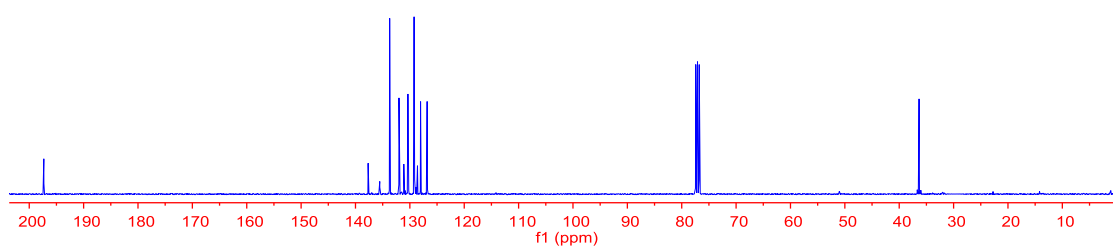
— 197.3

137.7
133.7
132.0
131.1
130.4
130.3
129.2
128.6
128.0
126.8

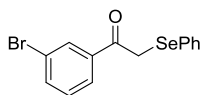
— 36.4



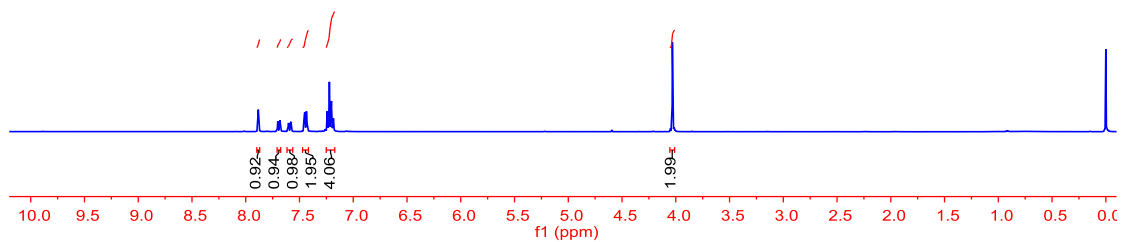
¹³C NMR (100 MHz, CDCl₃) of **3p**



7.89
7.88
7.88
7.70
7.70
7.69
7.68
7.68
7.60
7.60
7.60
7.58
7.58
7.58
7.45
7.45
7.44
7.44
7.43
7.24
7.24
7.23
7.23
7.22
7.22
7.21
7.20
7.19
7.18
4.03



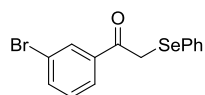
¹H NMR (400 MHz, CDCl₃) of **3q**



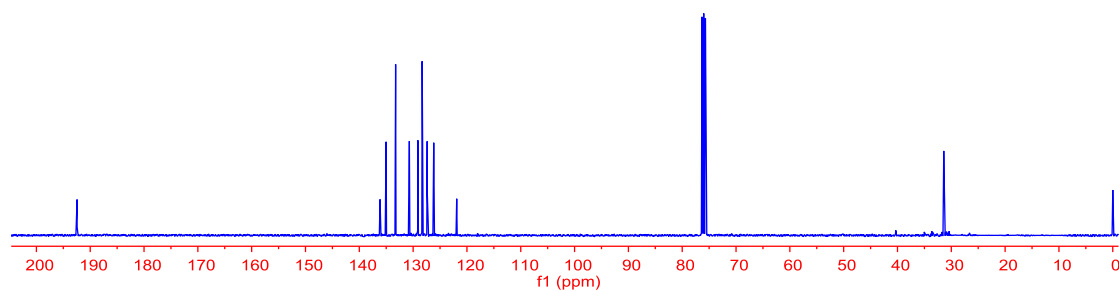
— 192.4

136.1
135.0
133.3
130.7
129.1
128.3
127.5
127.4
126.2
121.9

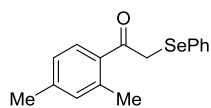
— 31.4



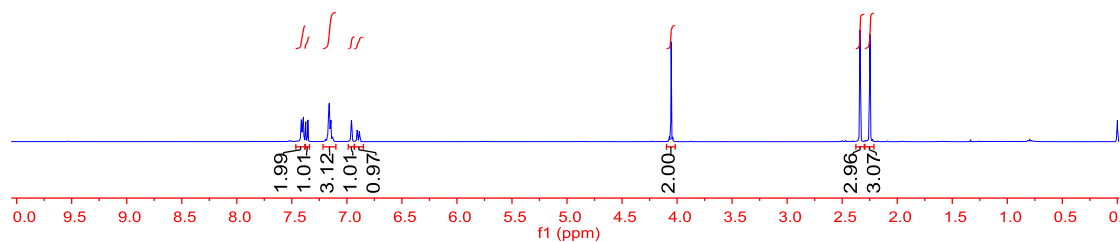
¹³C NMR (100 MHz, CDCl₃) of **3q**



7.42
7.41
7.41
7.41
7.40
7.39
7.37
7.35
7.20
7.19
7.19
7.18
7.18
7.17
7.16
7.16
7.16
7.16
7.15
7.15
7.14
7.14
7.13
7.13
7.12
6.96
6.96
6.91
6.90
6.89
6.88
4.05
2.34
2.25



¹H NMR (400 MHz, CDCl₃) of **3r**

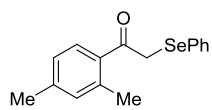


— 196.7

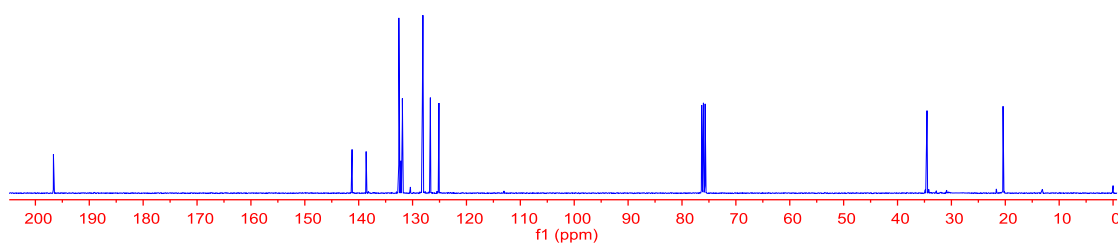
141.2
138.6
132.5
132.2
131.9
128.2
128.1
128.1
126.7
125.1

— 34.5

20.4
20.4

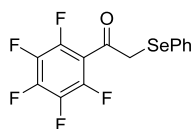


¹³C NMR (100 MHz, CDCl₃) of **3r**

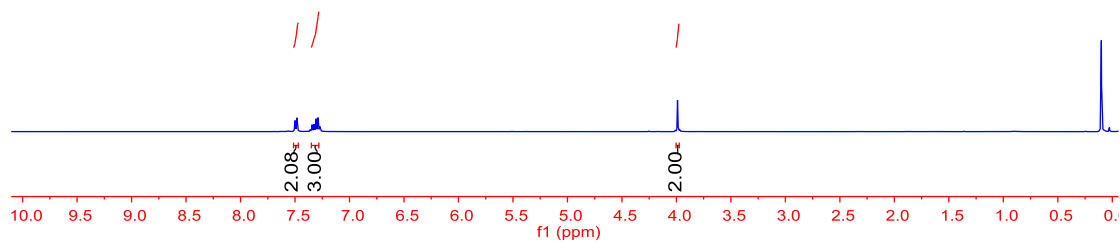


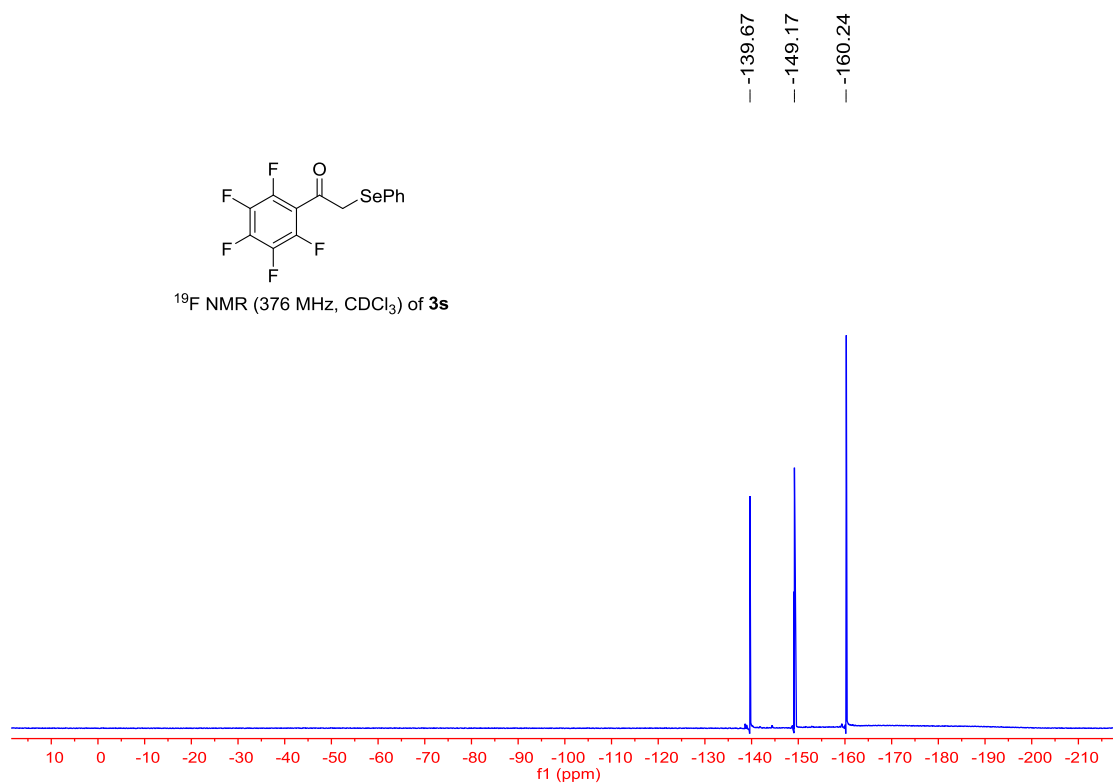
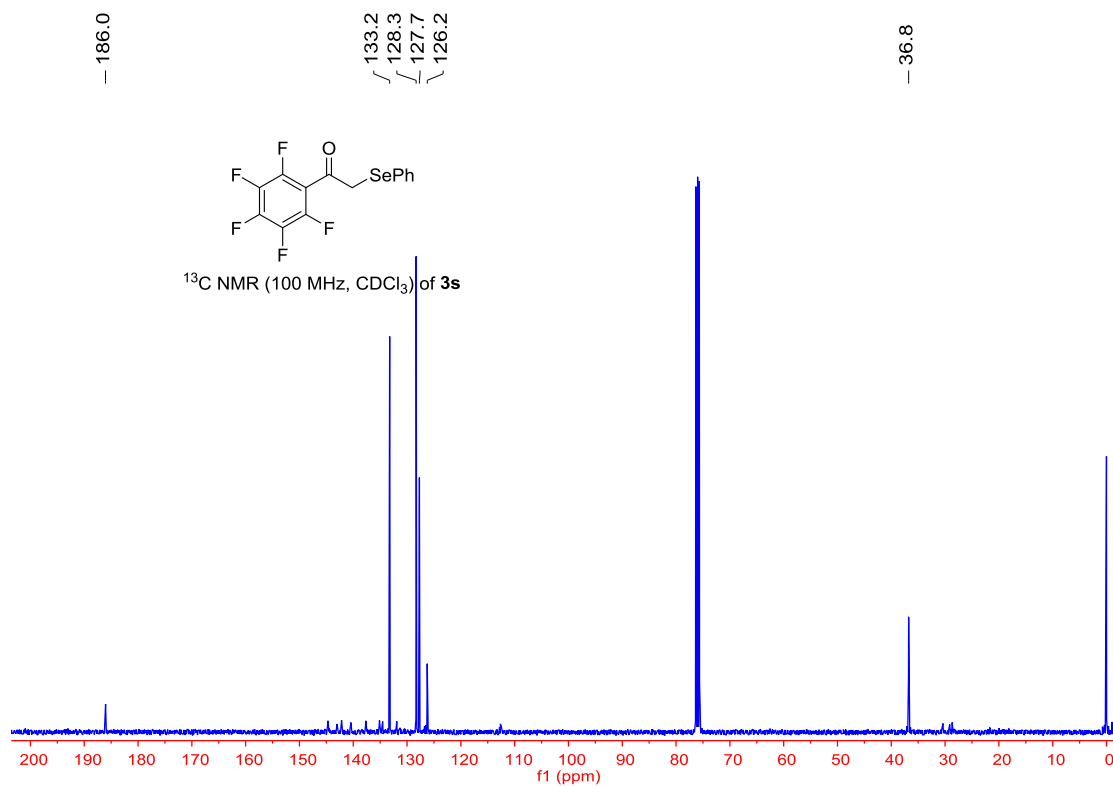
7.50
7.50
7.50
7.48
7.48
7.34
7.34
7.33
7.33
7.32
7.31
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.27
7.27

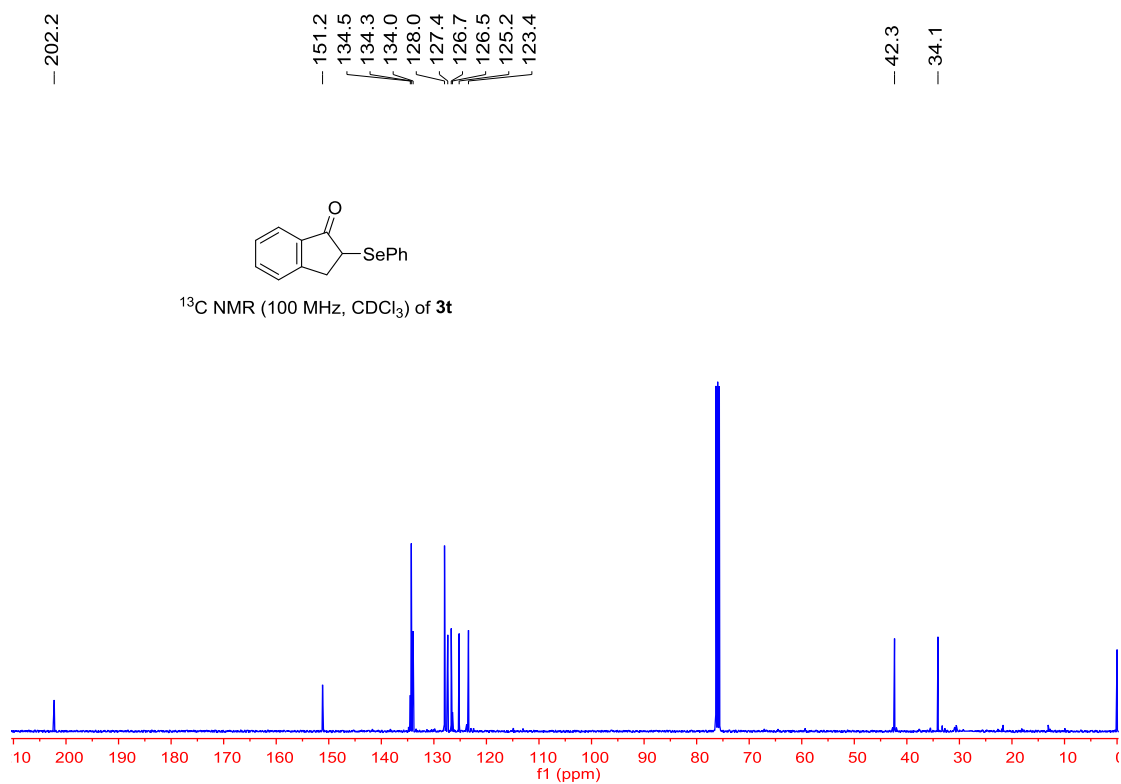
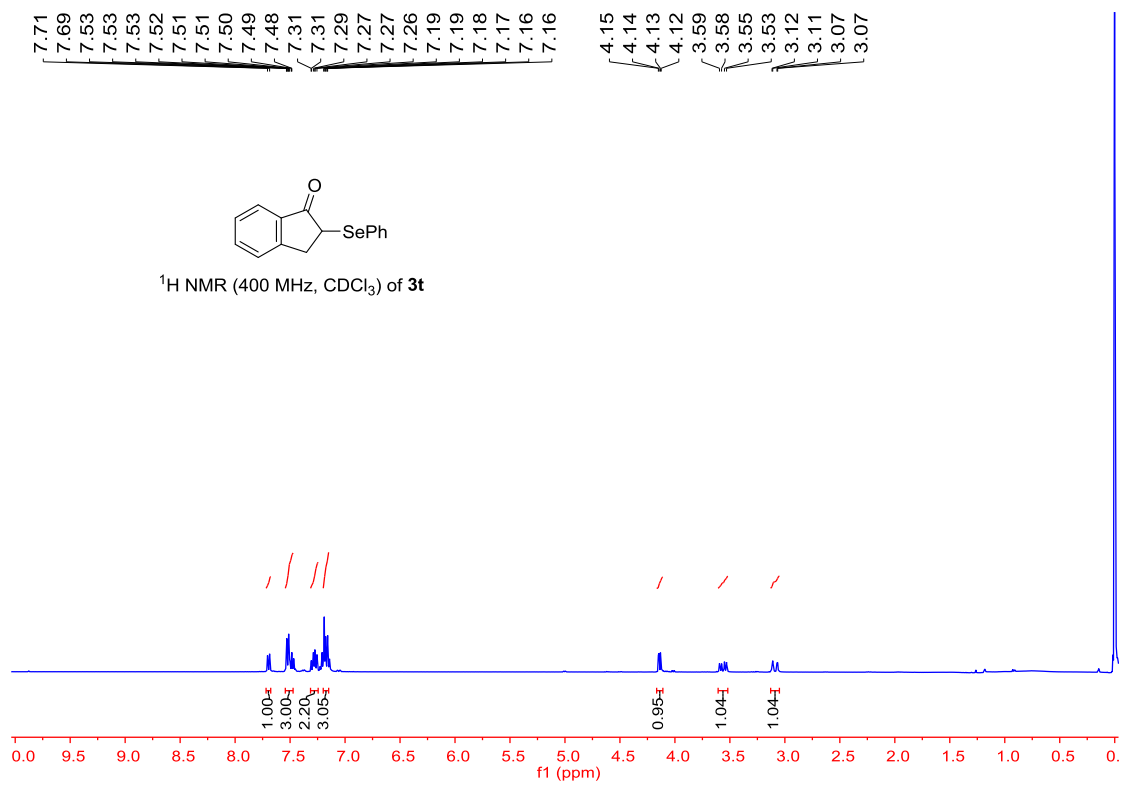
— 3.99

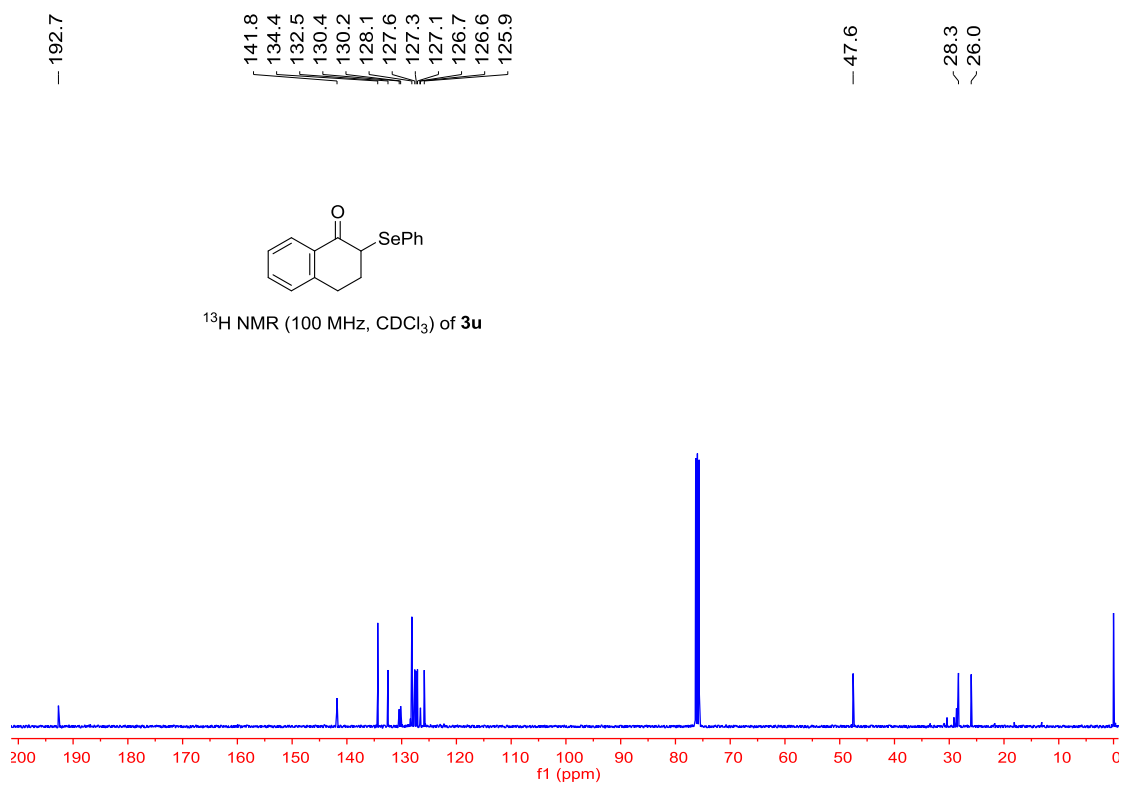
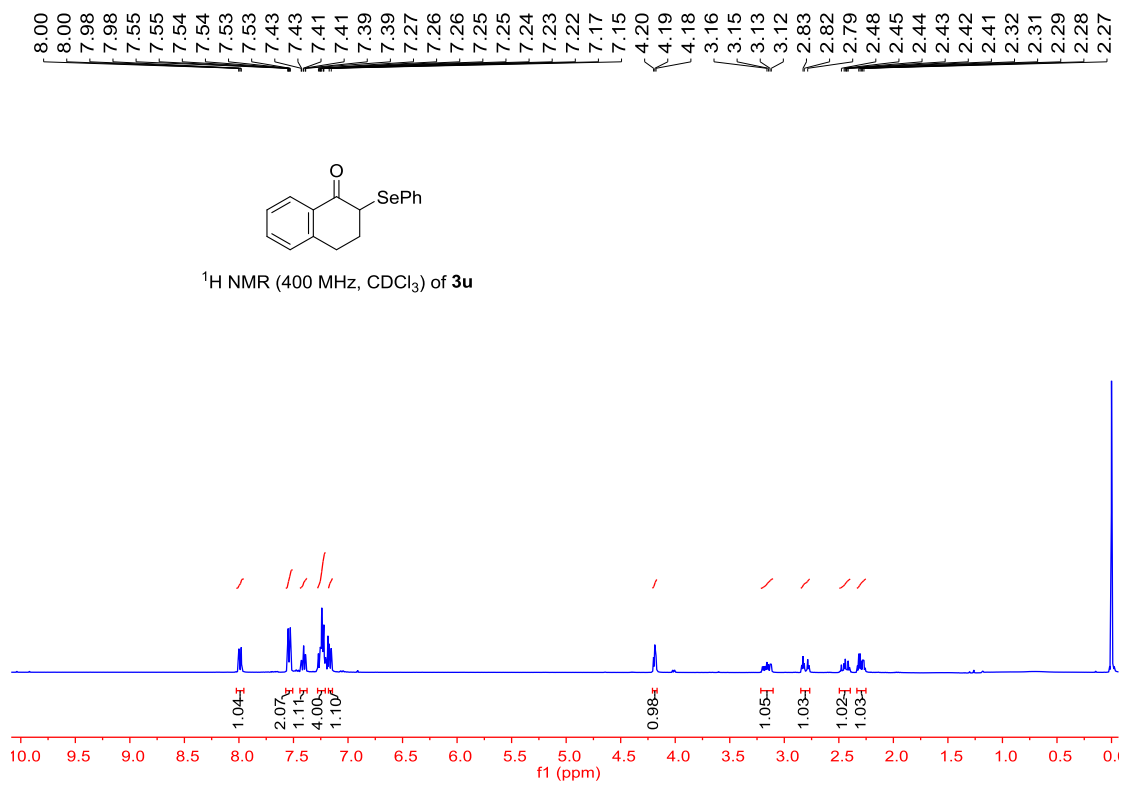


¹H NMR (400 MHz, CDCl₃) of **3s**

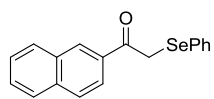




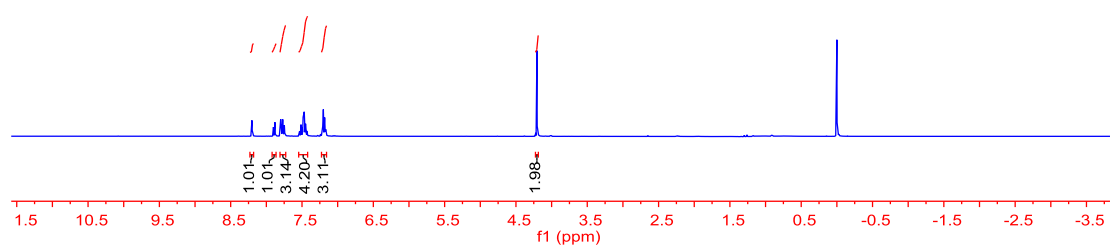




8.20
7.90
7.90
7.88
7.88
7.80
7.79
7.78
7.77
7.75
7.75
7.53
7.53
7.52
7.51
7.51
7.50
7.49
7.49
7.48
7.48
7.47
7.46
7.45
7.45
7.45
7.43
7.43
7.22
7.22
7.21
7.21
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.17
7.16
4.21



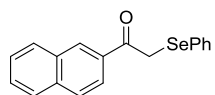
$^1\text{H NMR}$ (400 MHz, CDCl_3) of **3v**



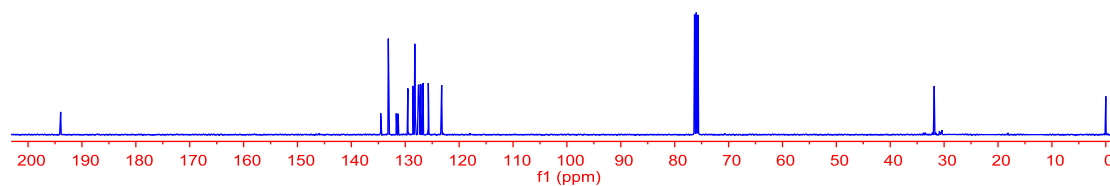
— 194.0

134.6
133.1
131.7
131.3
129.5
128.6
128.2
128.1
127.6
127.4
127.1
126.7
125.7
123.2

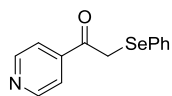
— 31.8



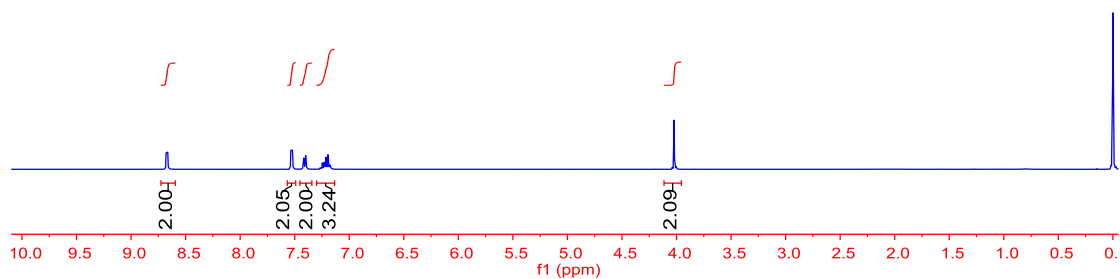
$^{13}\text{C NMR}$ (100 MHz, CDCl_3) of **3v**



8.68
8.66
7.54
7.52
7.42
7.42
7.41
7.41
7.40
7.40
7.26
7.25
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.18
7.18
7.18
7.17
4.02



^1H NMR (400 MHz, CDCl_3) of **3w**



— 193.8

— 150.8

— 141.5

— 134.4

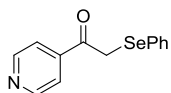
— 129.4

— 128.6

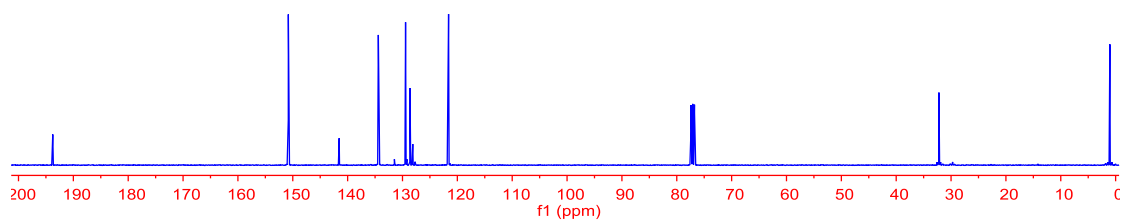
— 128.1

— 121.6

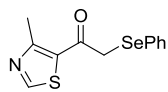
— 32.2



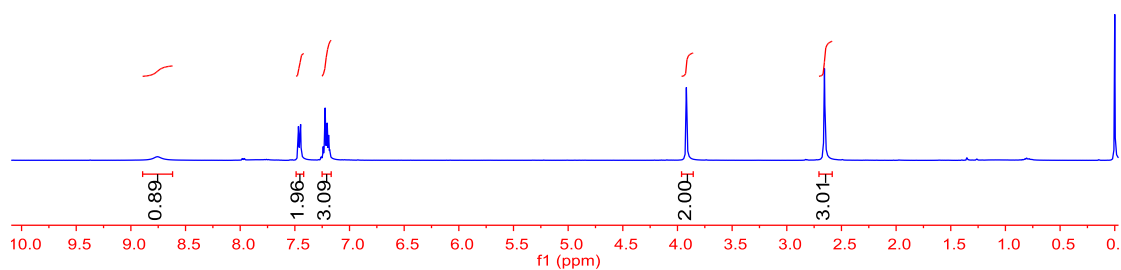
^{13}C NMR (100 MHz, CDCl_3) of **3w**



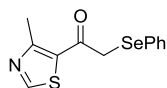
- 8.76
 7.47
 7.47
 7.45
 7.45
 7.24
 7.23
 7.22
 7.20
 7.19
 7.18
 - 3.92
 - 2.66



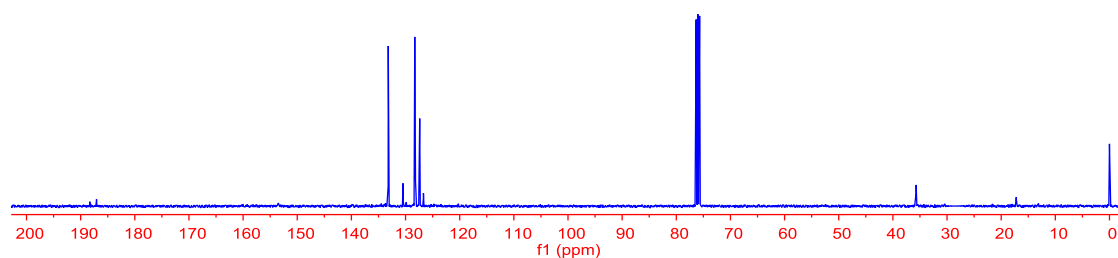
^1H NMR (400 MHz, CDCl_3) of **3x**

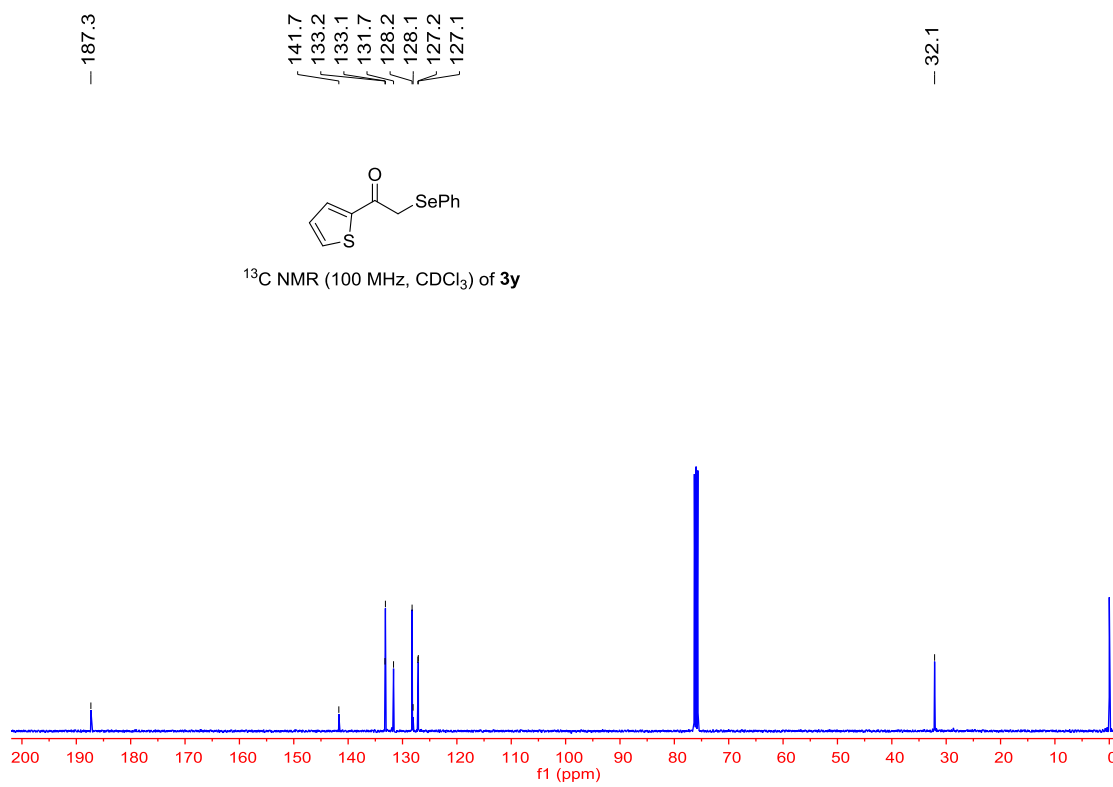
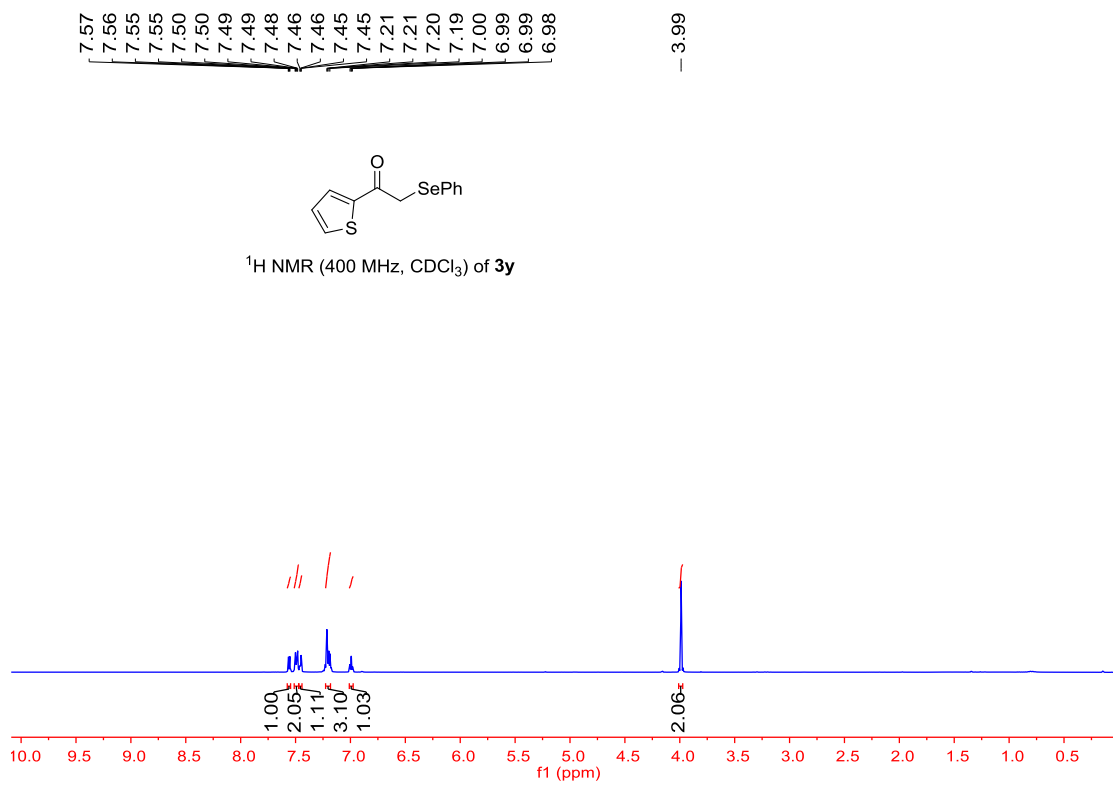


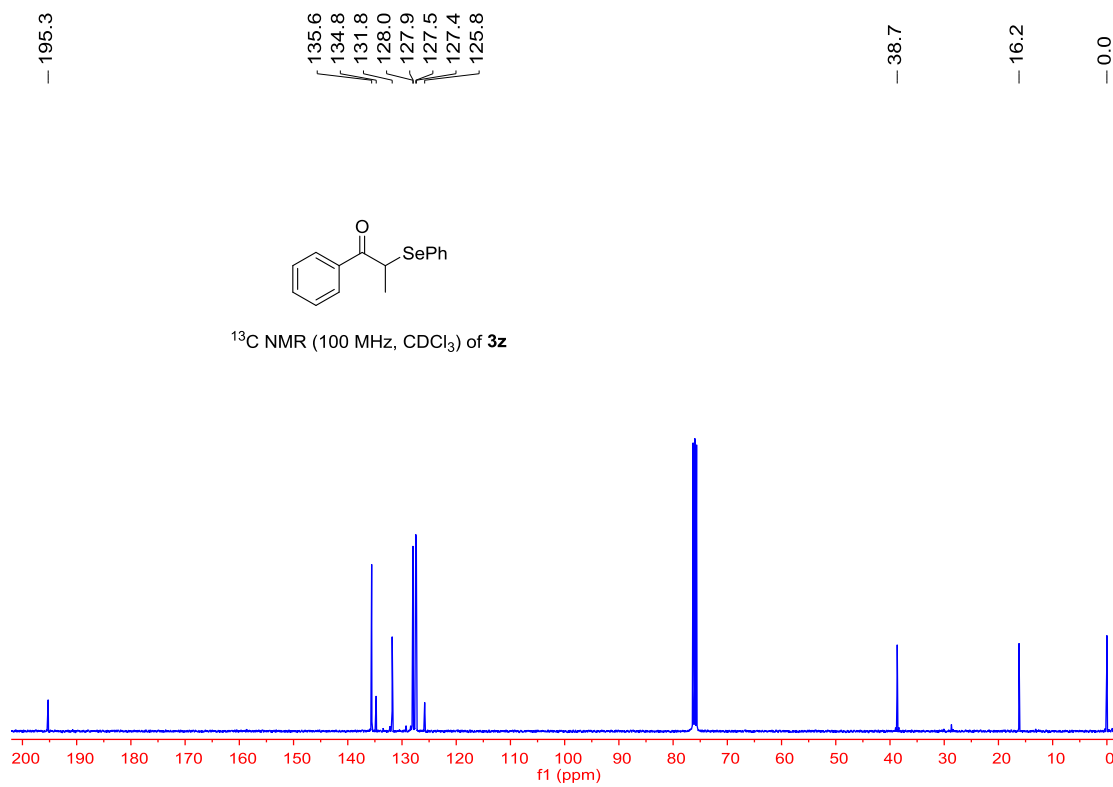
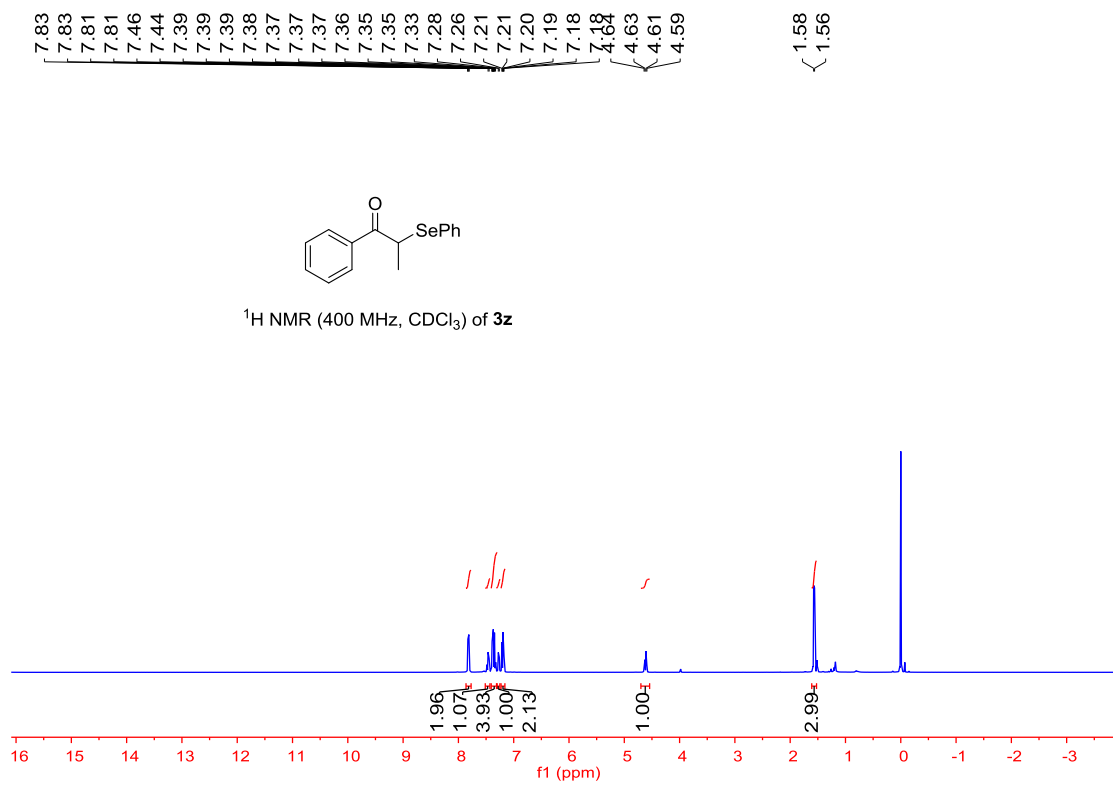
- 187.1
 133.2
 130.5
 128.3
 128.2
 127.4
 127.2
 126.7
 - 35.7
 - 17.2

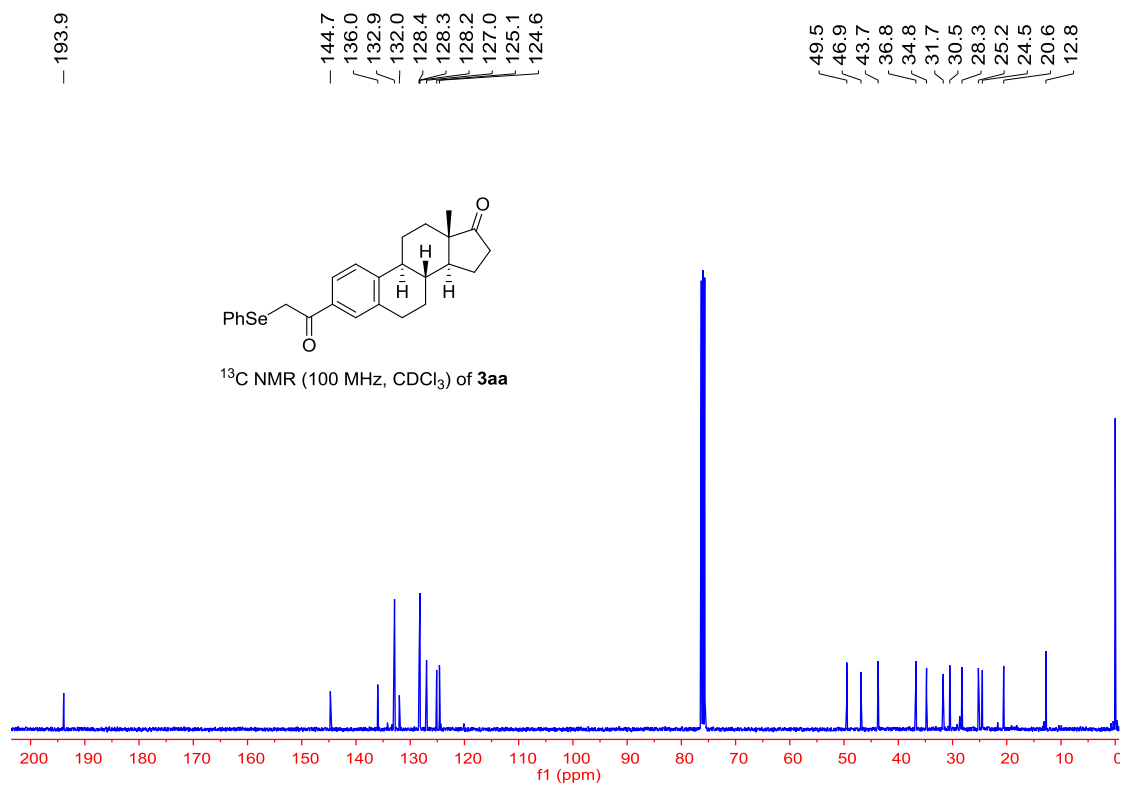
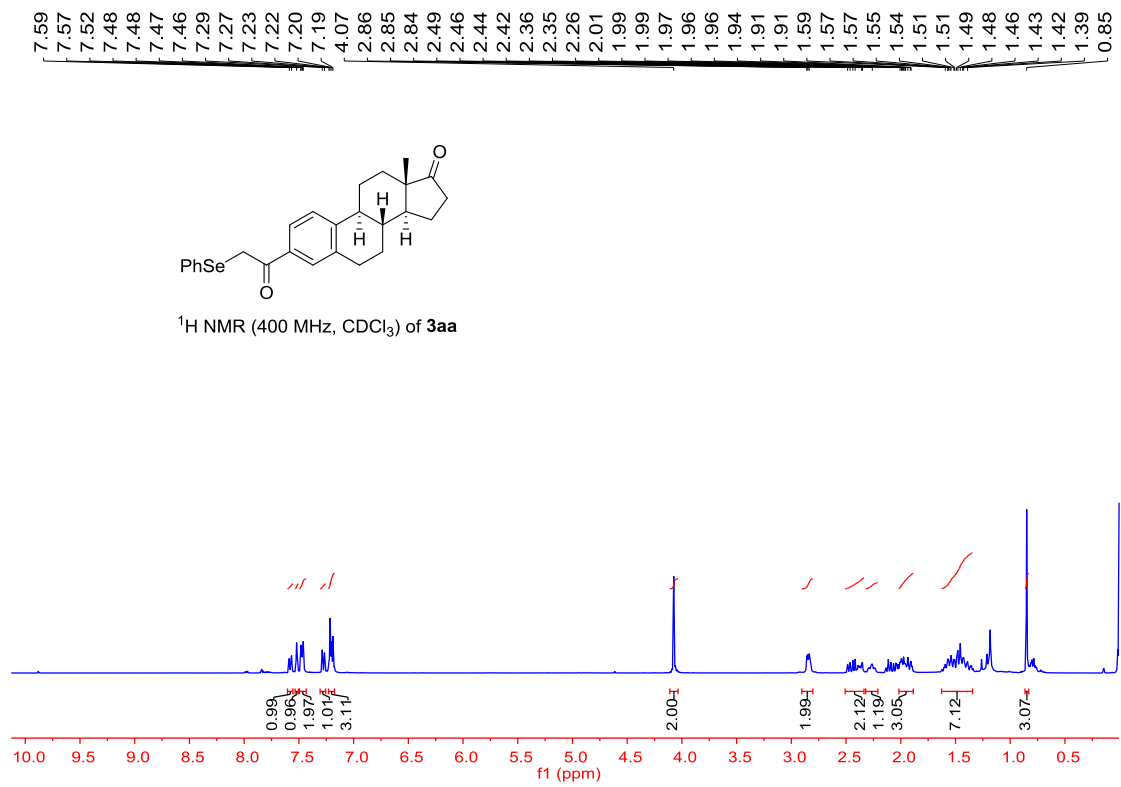


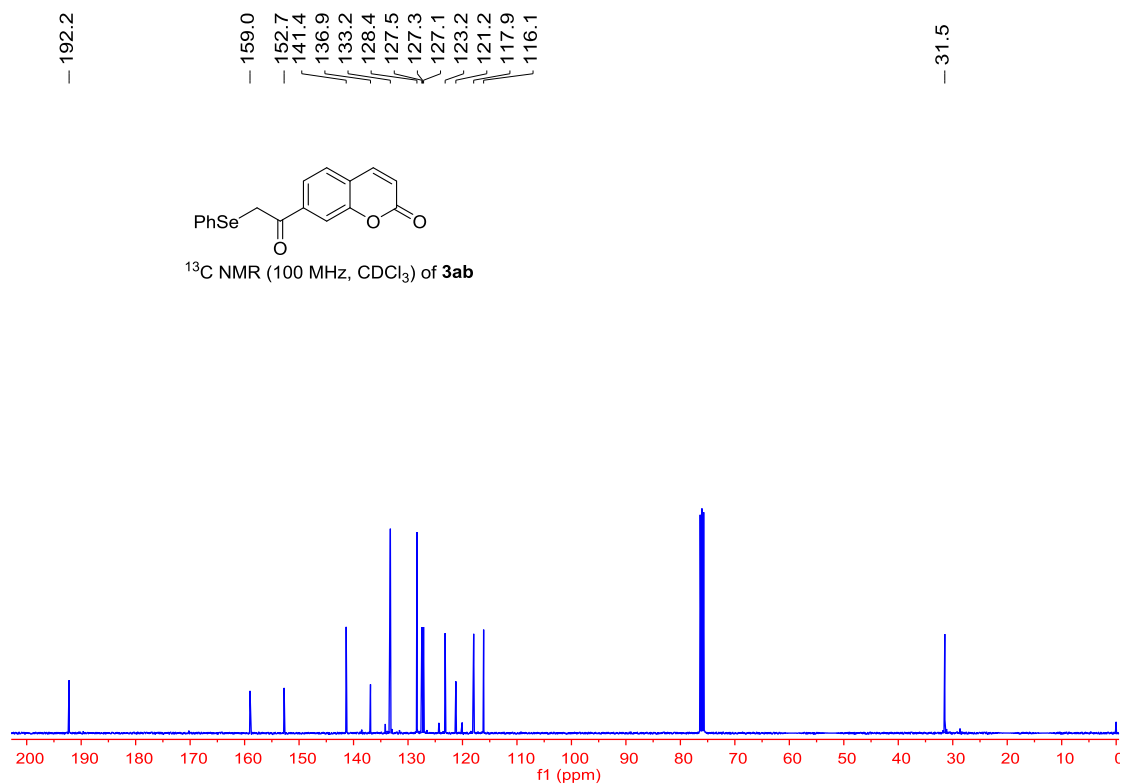
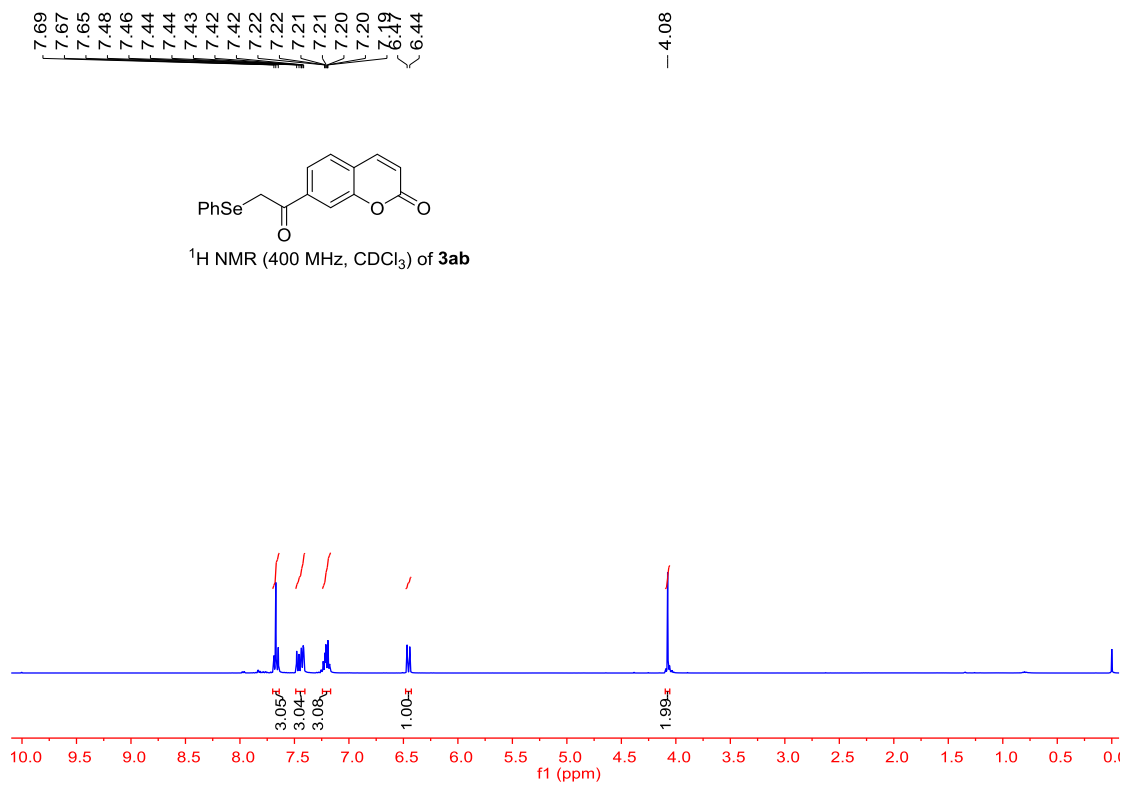
^{13}C NMR (100 MHz, CDCl_3) of **3x**

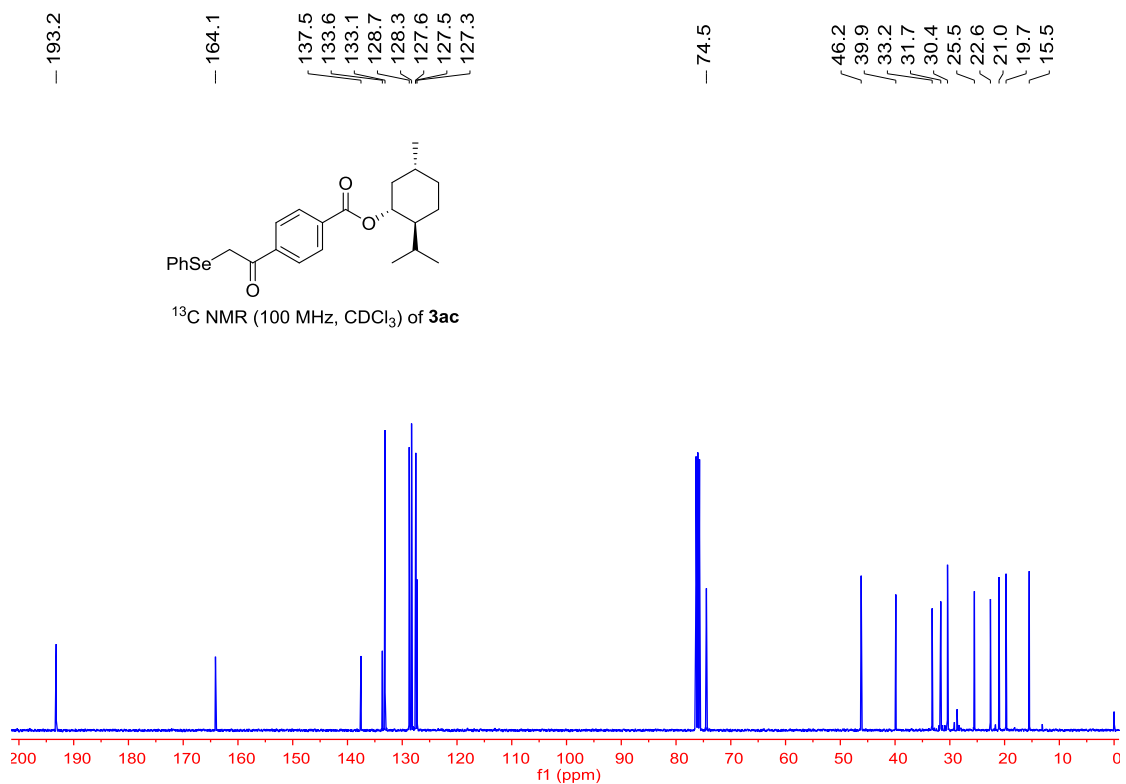
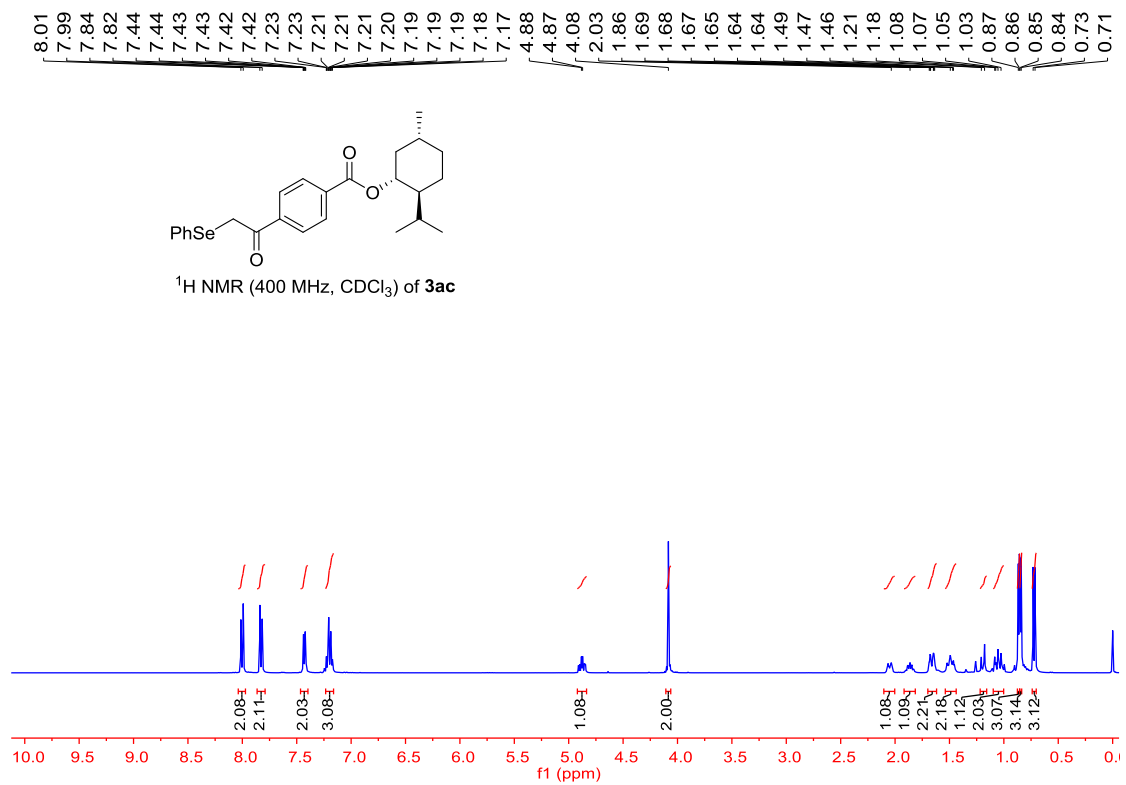








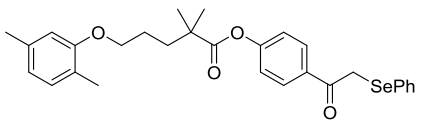




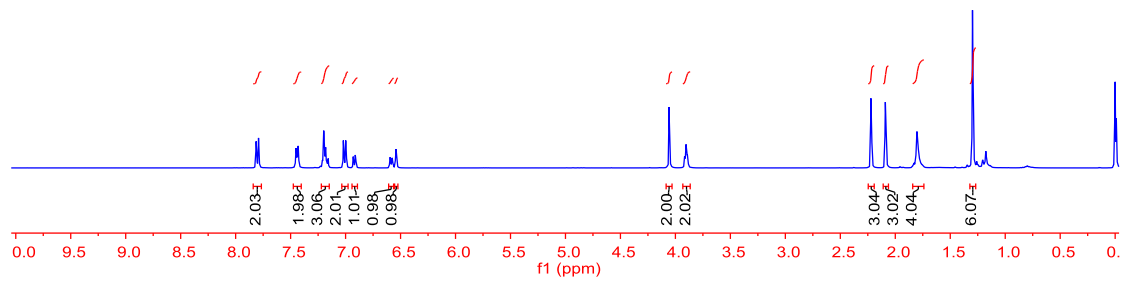
7.81
7.81
7.79
7.79
7.45
7.45
7.44
7.44
7.44
7.43
7.20
7.18
7.16
7.02
7.02
7.00
6.99
6.93
6.91
6.59
6.58
6.54

4.06
3.92
3.90
3.89

2.22
2.09
1.80
1.30



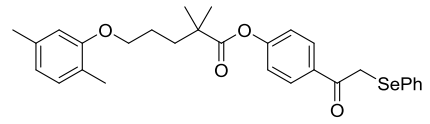
¹H NMR (400 MHz, CDCl₃) of 3ad



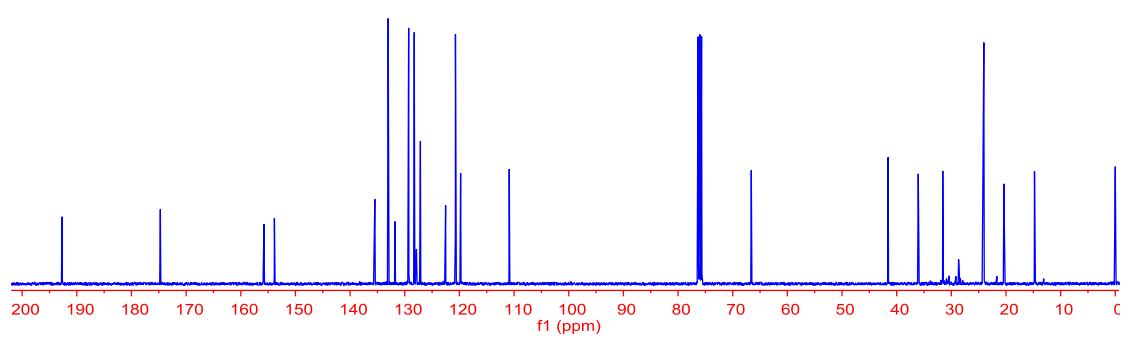
192.7
174.7
155.8
153.9
135.5
133.0
131.8
129.3
129.3
128.3
127.9
127.1
122.5
120.7
119.8
110.9

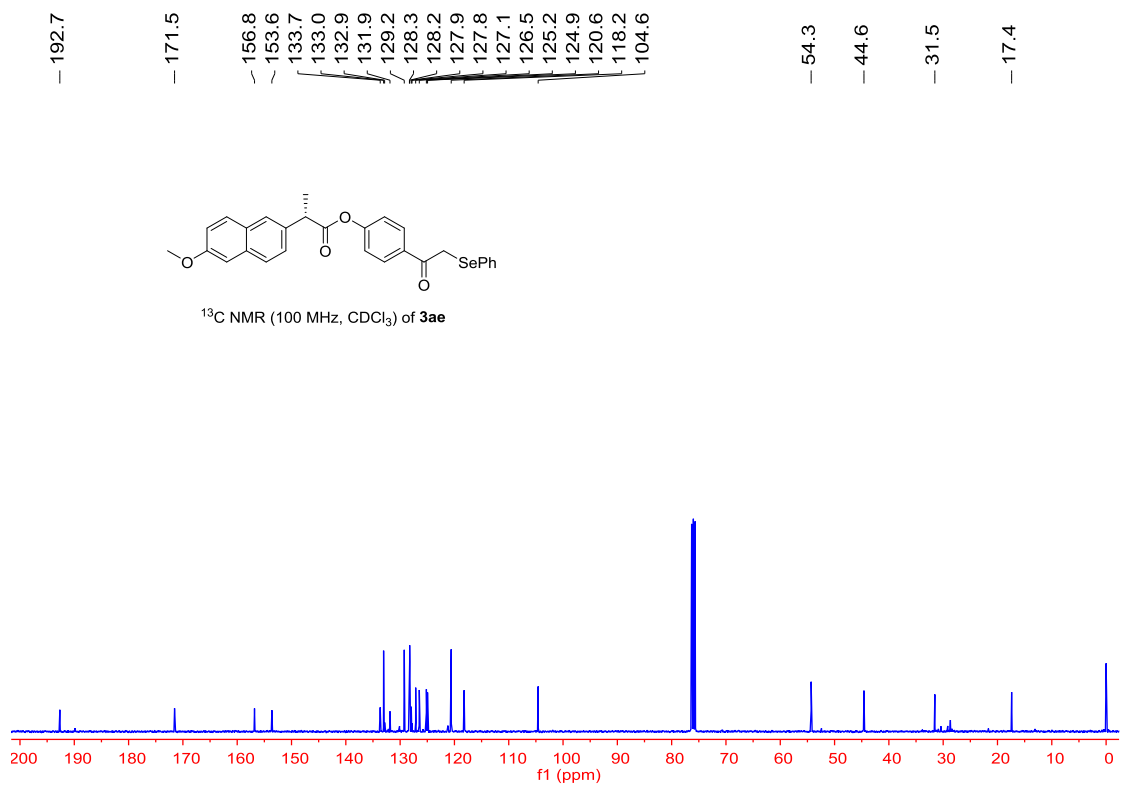
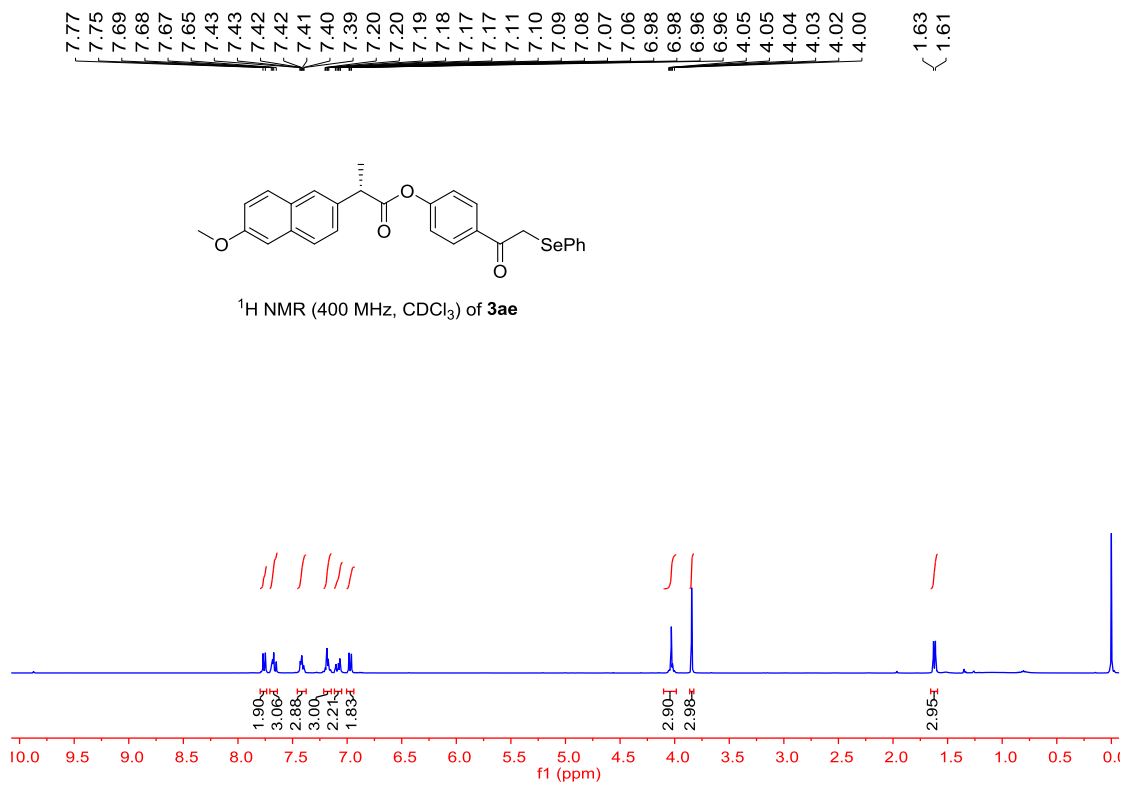
66.6

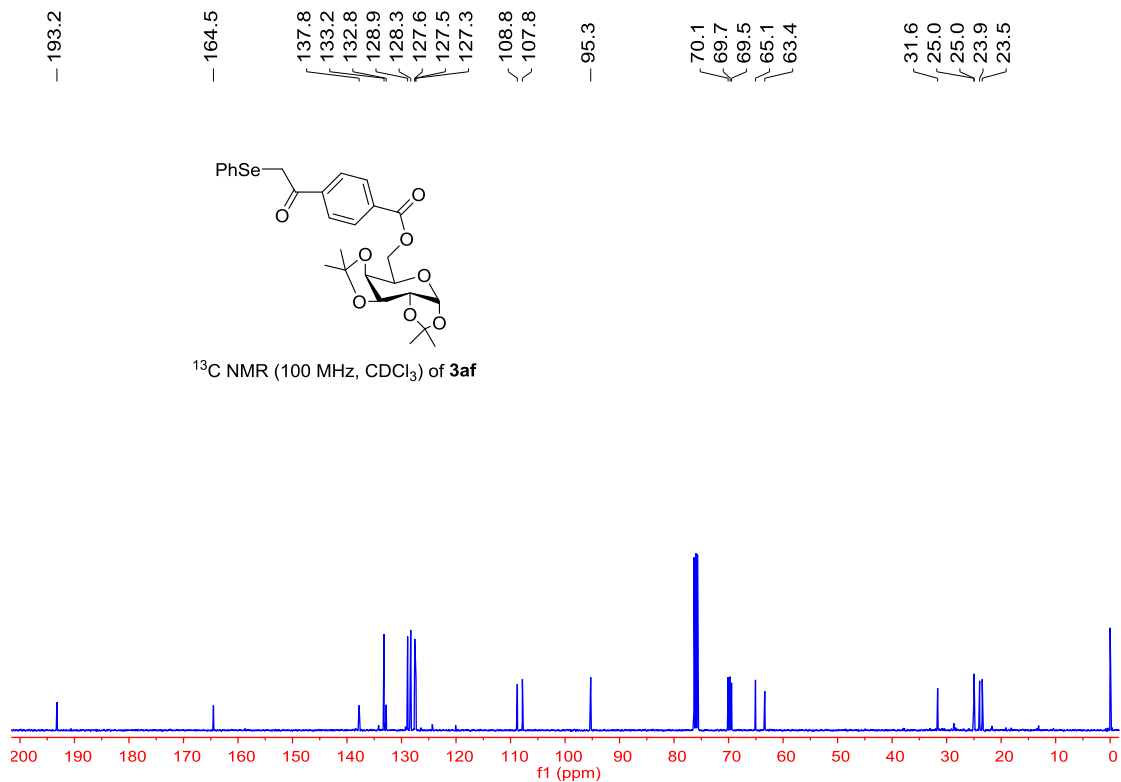
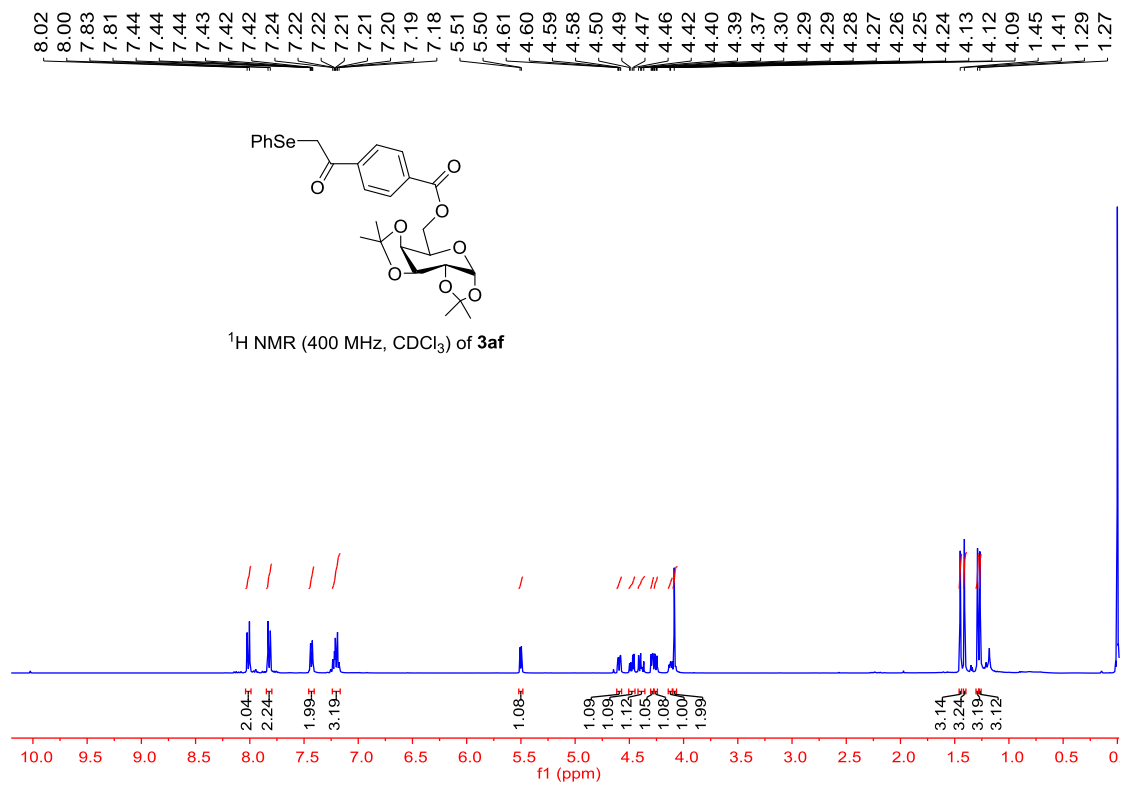
41.6
36.1
31.6
24.2
24.1
20.4
14.8



¹³C NMR (100 MHz, CDCl₃) of 3ad

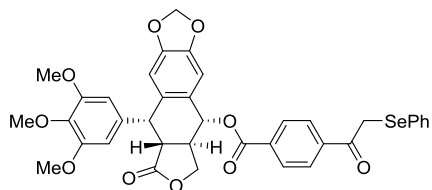




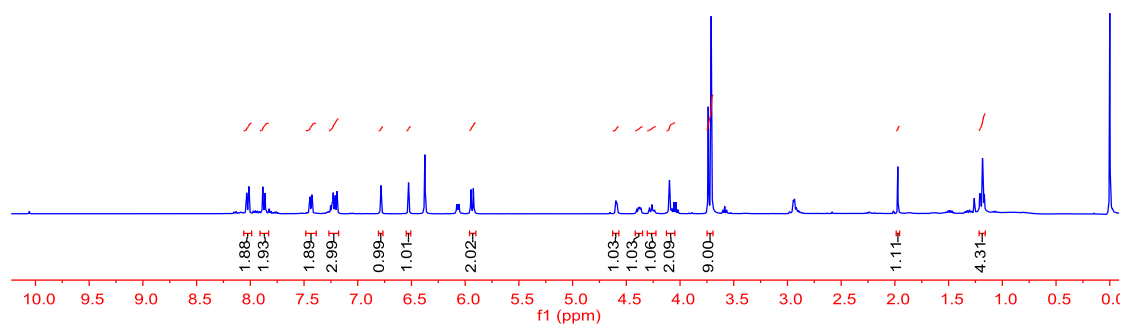


8.03
8.01
7.88
7.86
7.44
7.43
7.42
7.25
7.24
7.23
7.21
7.19
6.78
6.53
5.94
5.92
4.60
4.59
4.40
4.39
4.38
4.36
4.28
4.26
4.24
4.10
4.07
4.06
3.74
3.71

- 1.97
1.21
1.20
1.18
1.17



¹H NMR (400 MHz, CDCl₃) of **3ag**



- 192.9

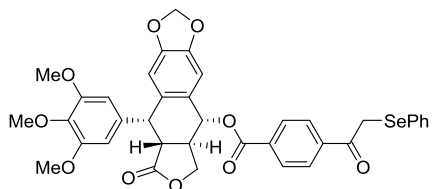
172.6
164.9
151.7
147.3
146.8
138.4
136.2
133.7
133.2
131.9
131.6
128.9
128.4
127.8
127.4
127.0

108.9
107.1
106.0
100.7

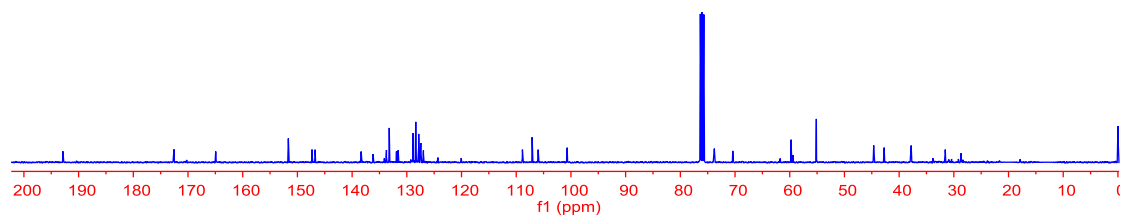
- 73.8
- 70.4

- 59.7
- 55.1

- 44.6
- 42.7
- 37.8
- 31.6
- 28.7



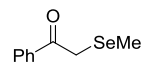
¹³C NMR (100 MHz, CDCl₃) of **3ag**



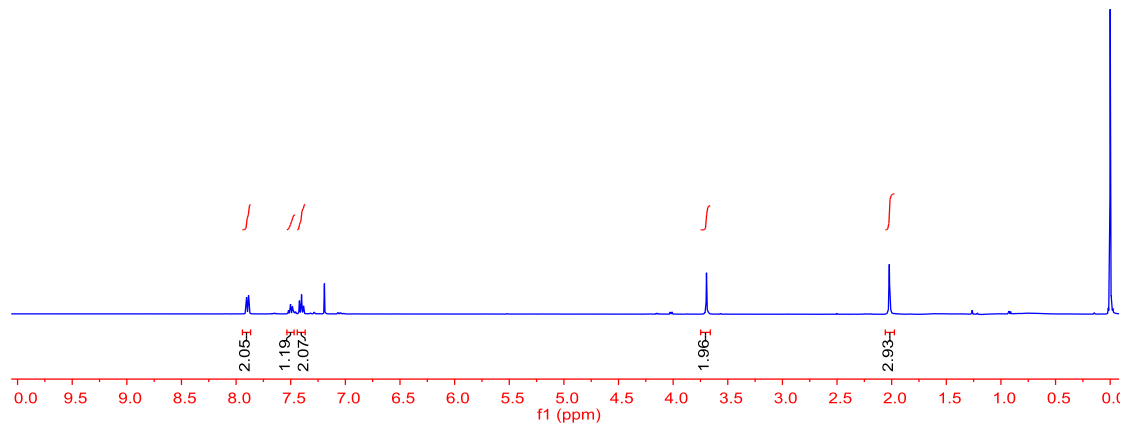
7.90
7.89
7.88
7.52
7.50
7.48
7.42
7.40
7.38

— 3.69

— 2.02



¹H NMR (400 MHz, CDCl₃) of **4a**

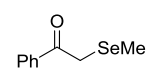


— 194.5

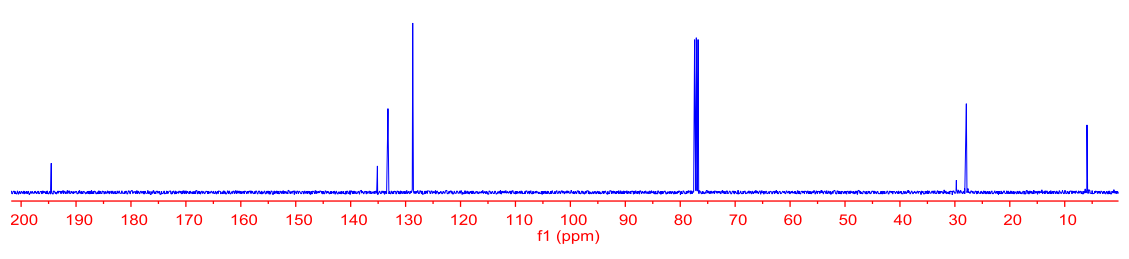
135.1
133.2
128.7
128.7

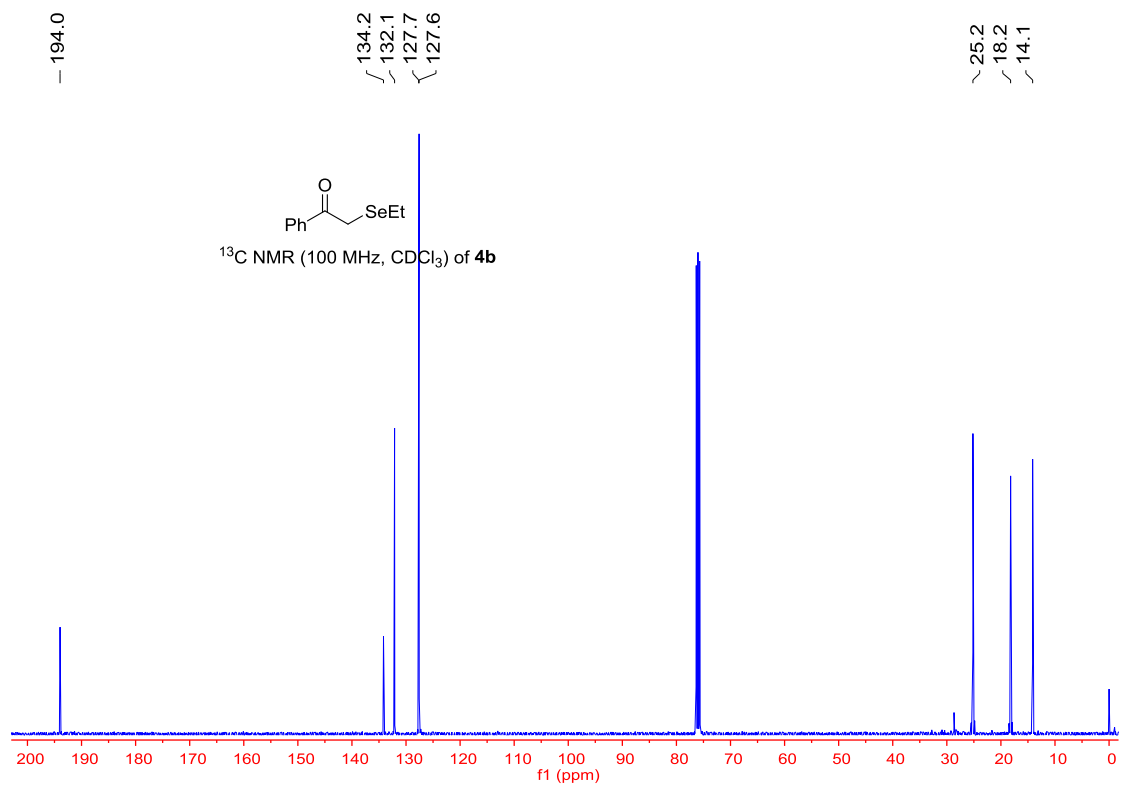
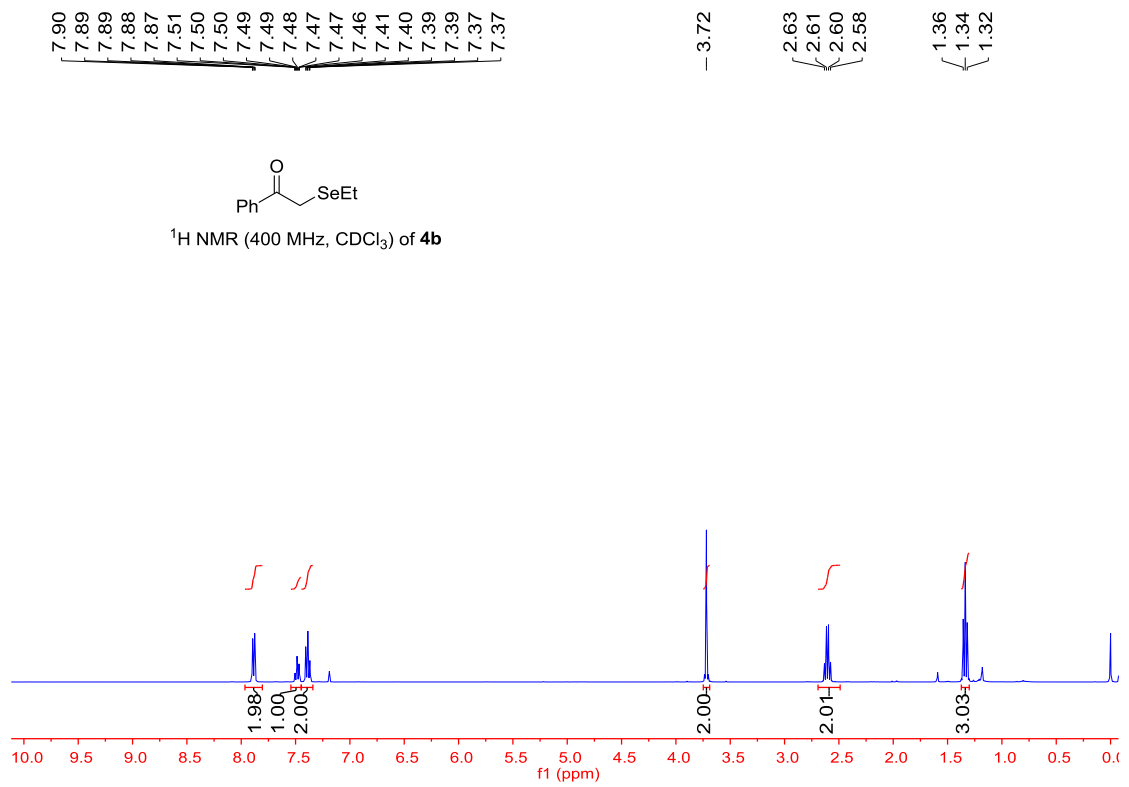
— 27.9

— 5.9

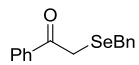


¹³C NMR (100 MHz, CDCl₃) of **4a**

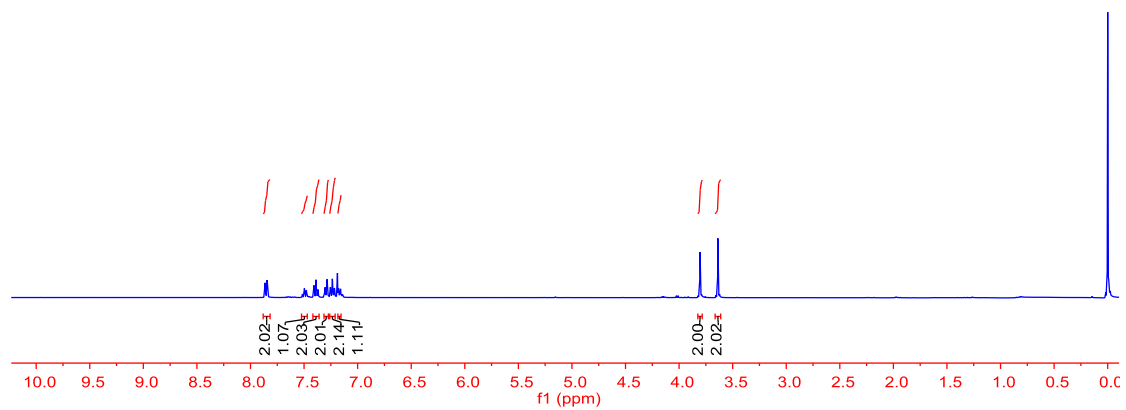




7.86
7.85
7.85
7.84
7.52
7.50
7.50
7.49
7.48
7.48
7.41
7.40
7.39
7.37
7.37
7.31
7.30
7.29
7.25
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7.23
7.22
7.18
7.18
7.17
7.17
7.16
3.80
3.64



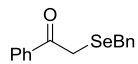
¹H NMR (400 MHz, CDCl₃) of **4c**



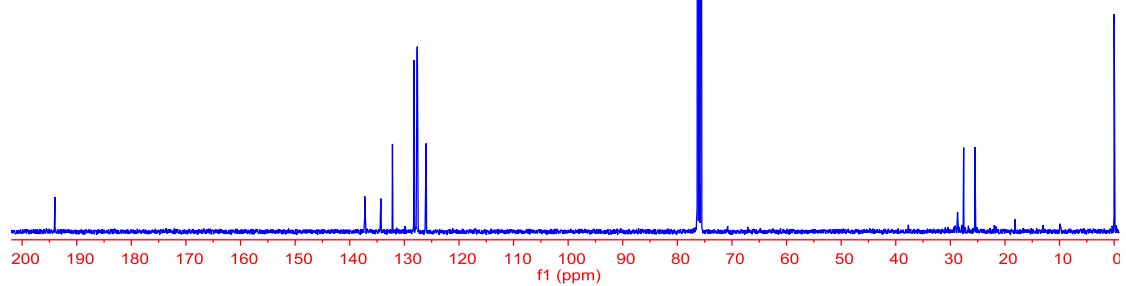
194.0

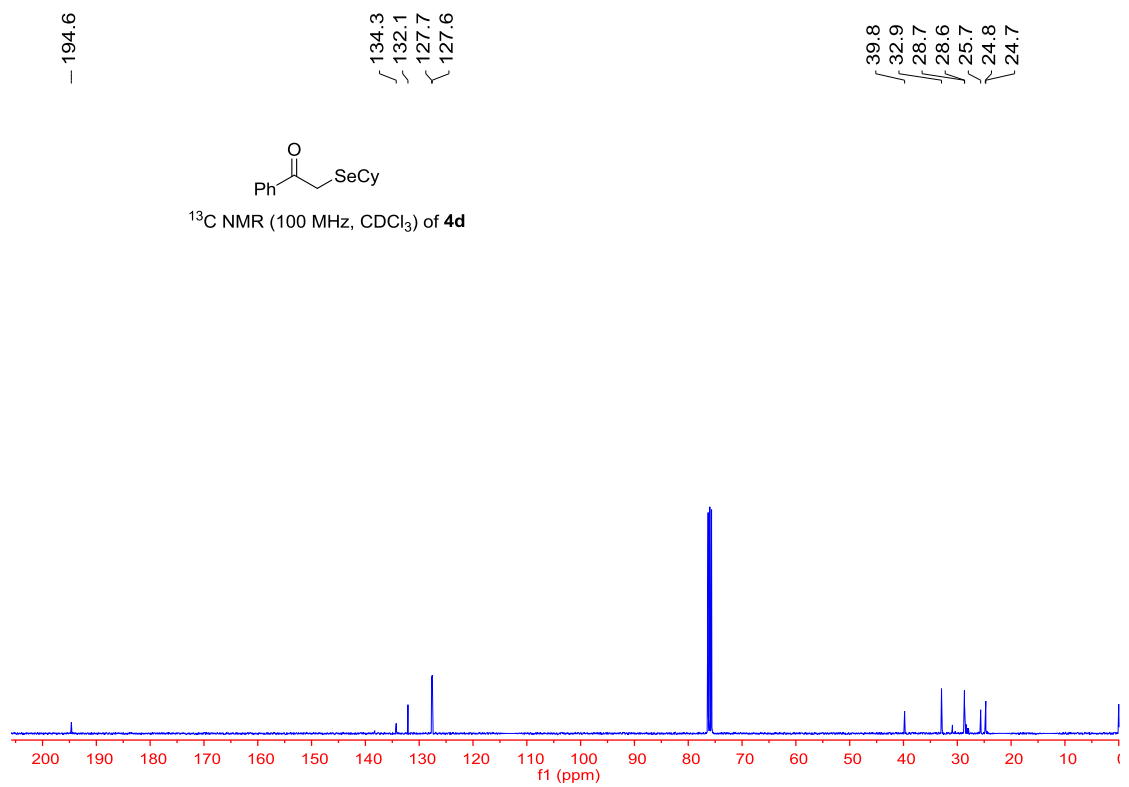
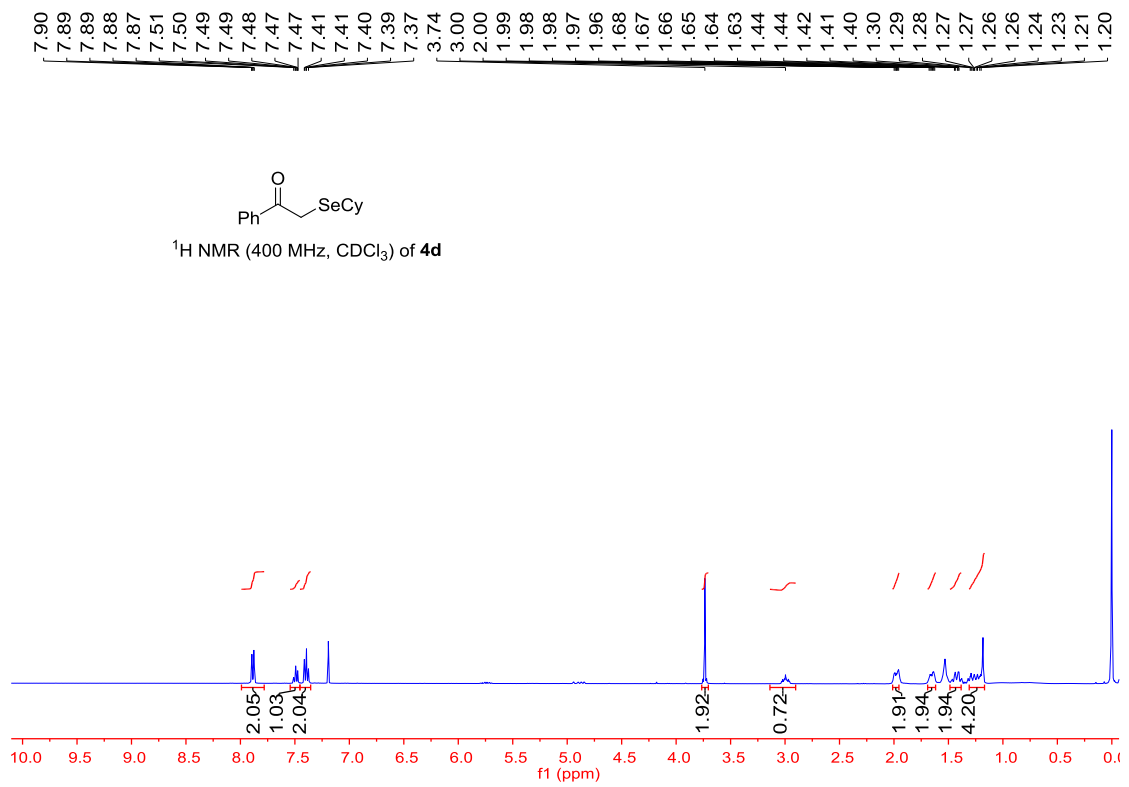
137.2
134.3
132.2
128.2
127.7
127.6
127.5
126.0

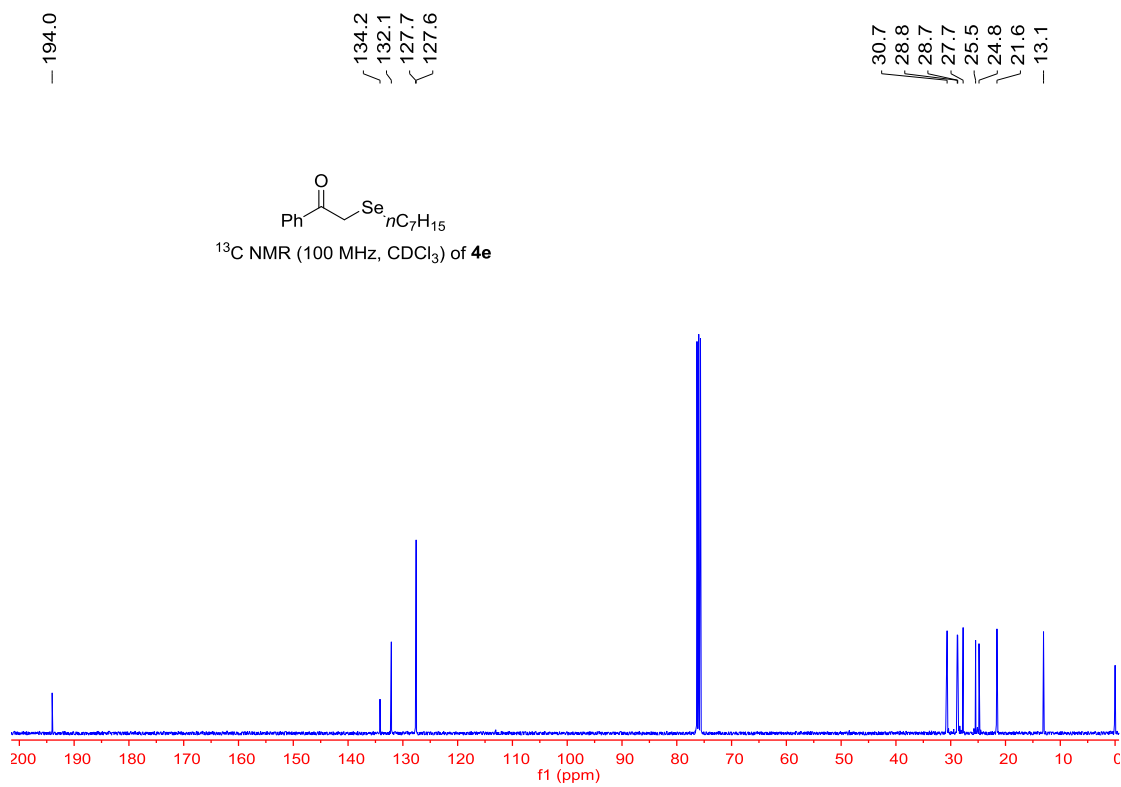
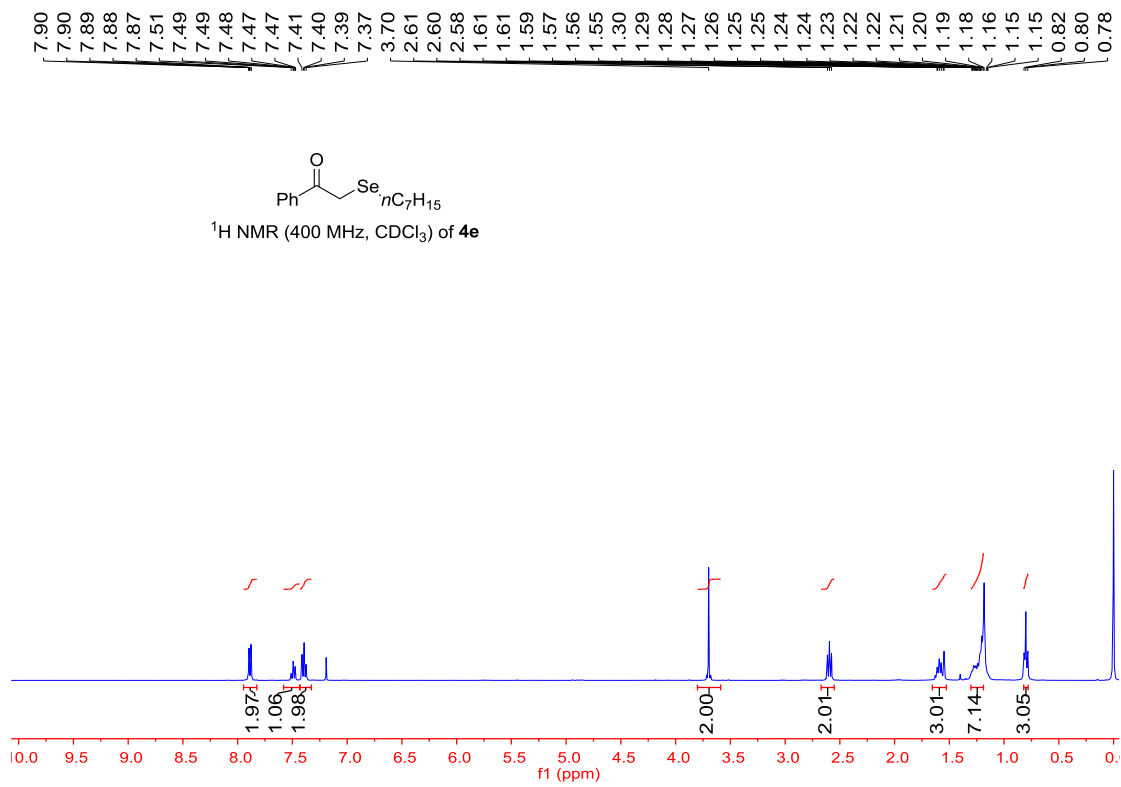
27.6
25.5



¹³C NMR (100 MHz, CDCl₃) of **4c**

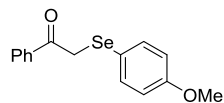




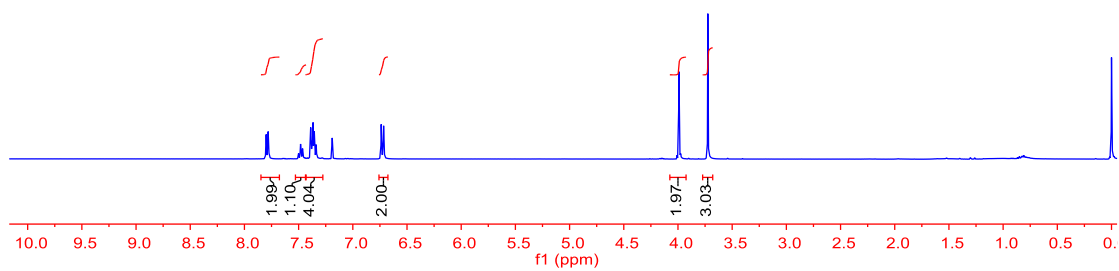


7.80
7.80
7.78
7.78
7.50
7.48
7.48
7.47
7.46
7.46
7.38
7.37
7.37
7.36
7.34
6.74
6.72

3.99
3.72



¹H NMR (400 MHz, CDCl₃) of **4f**



194.0

159.1

135.9

134.5

132.1

127.7

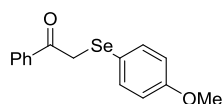
127.5

117.7

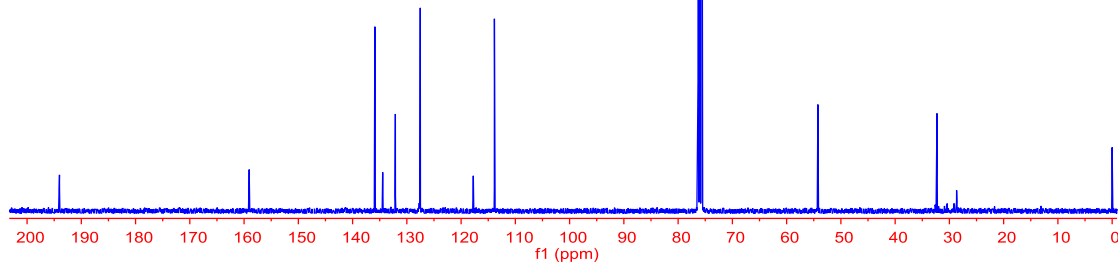
113.9

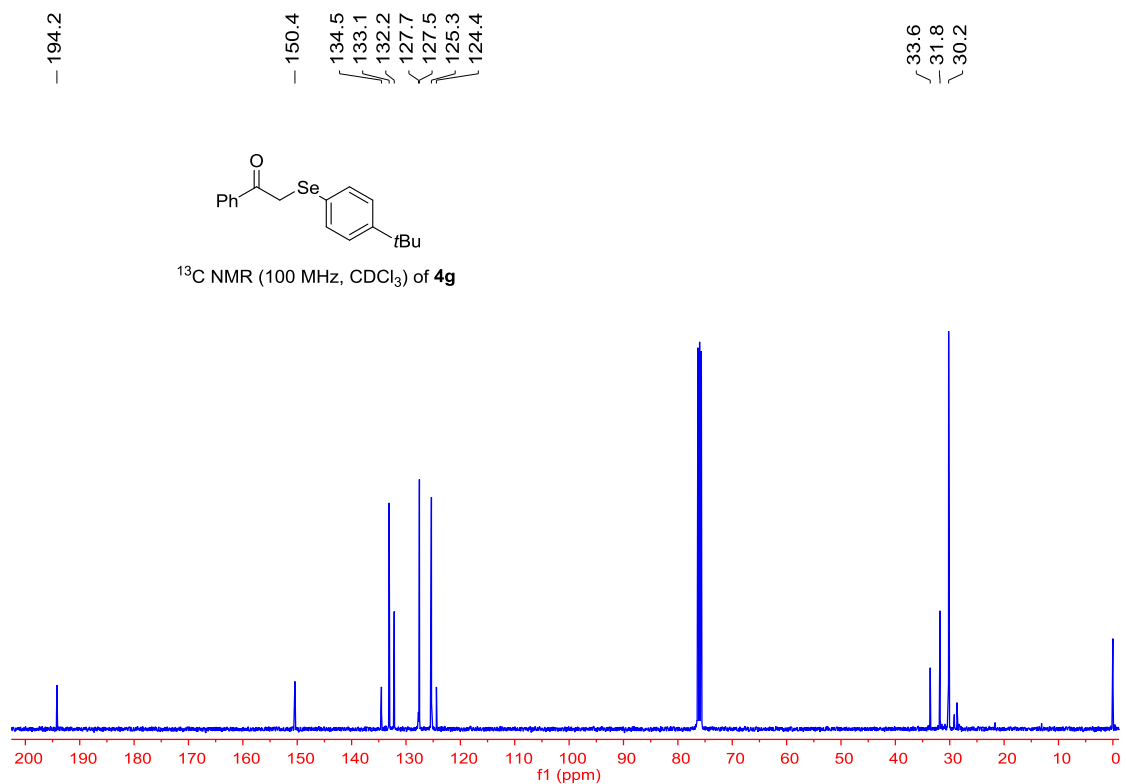
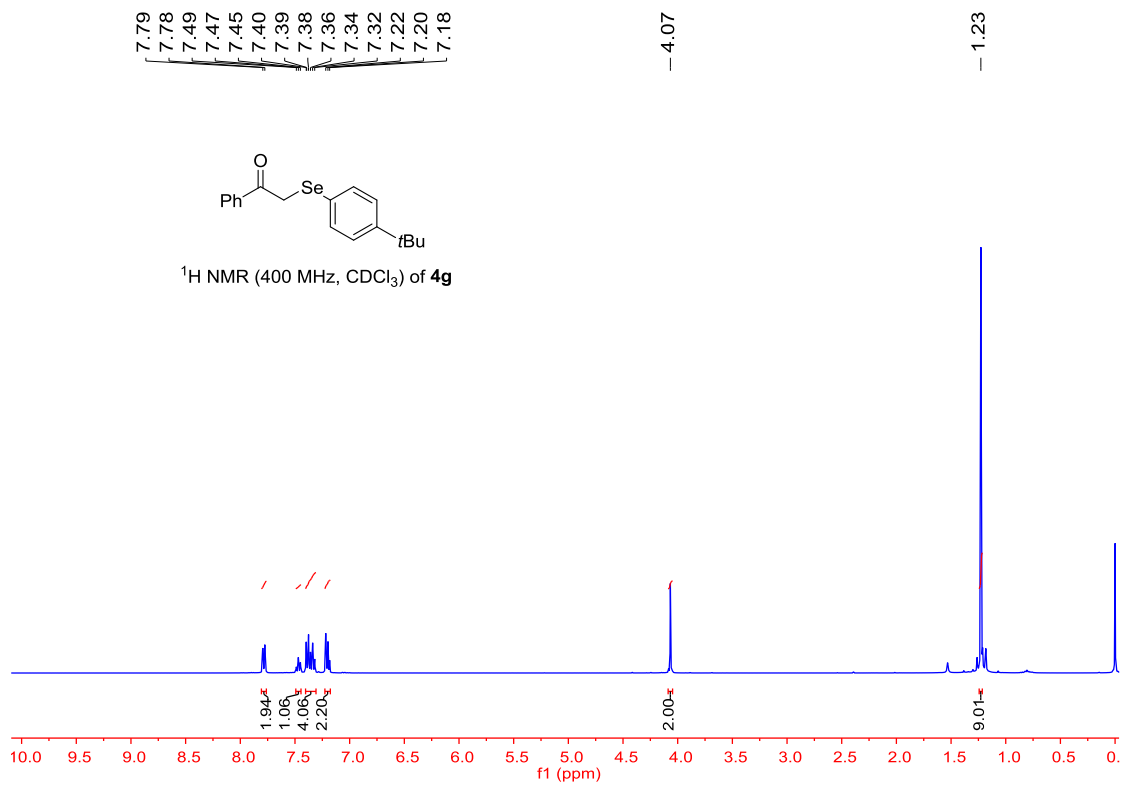
54.2

32.3



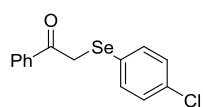
¹³C NMR (100 MHz, CDCl₃) of **4f**



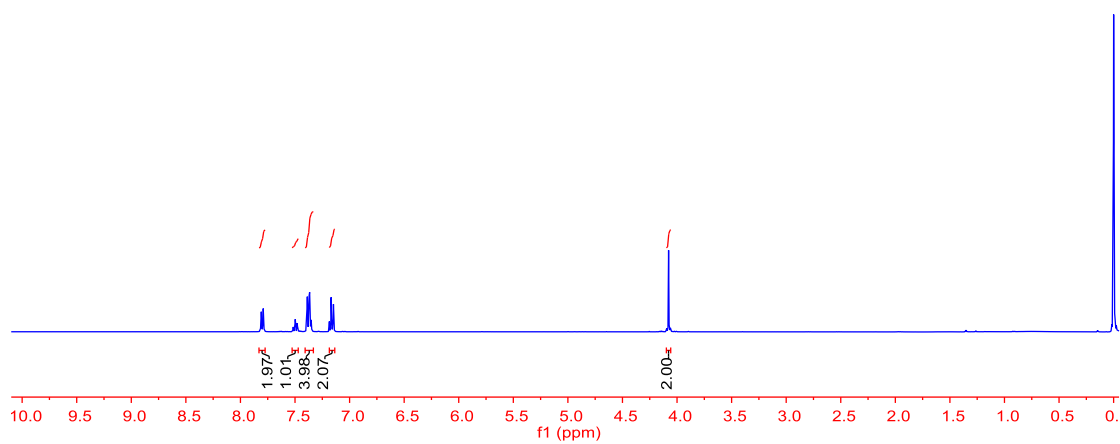


7.81
7.79
7.52
7.51
7.50
7.48
7.48
7.39
7.38
7.37
7.37
7.36
7.36
7.35
7.17
7.15

— 4.08



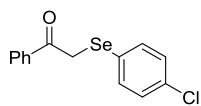
^1H NMR (400 MHz, CDCl_3) of **4h**



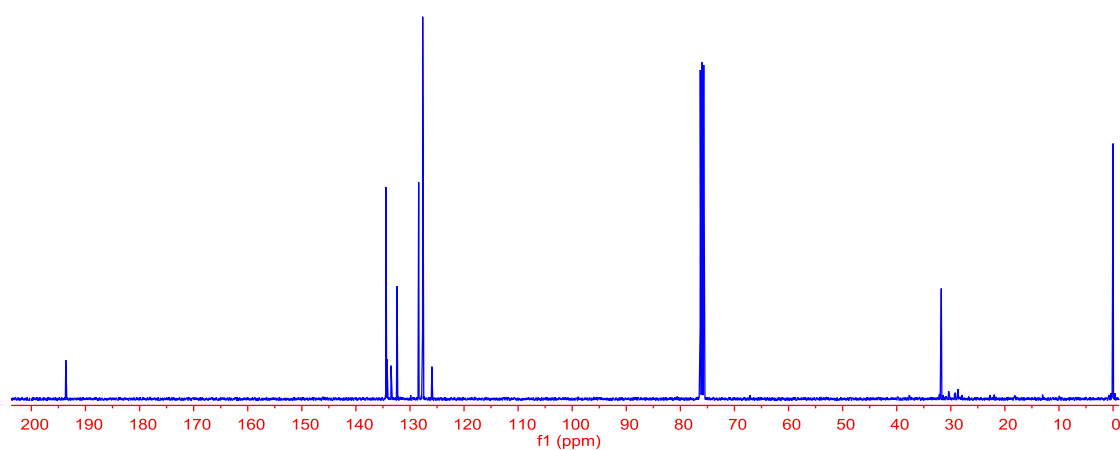
— 193.6

134.4
134.3
133.5
132.4
128.4
127.6
125.9

— 31.8

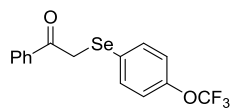


^{13}C NMR (100 MHz, CDCl_3) of **4h**

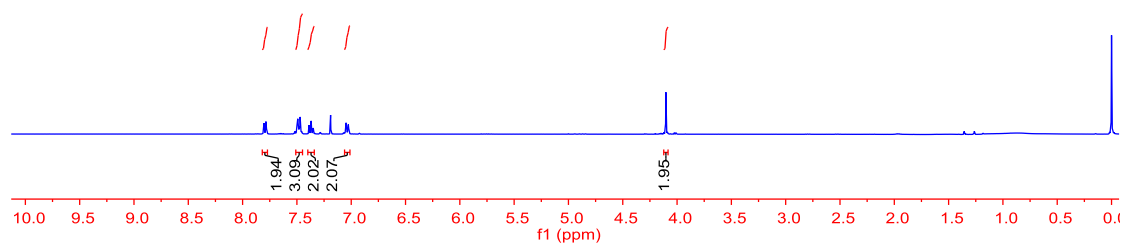


7.80
7.78
7.50
7.49
7.49
7.48
7.48
7.47
7.46
7.45
7.39
7.37
7.35
7.05
7.03

- 4.10



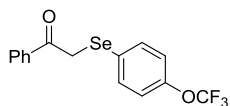
¹H NMR (400 MHz, CDCl₃) of **4i**



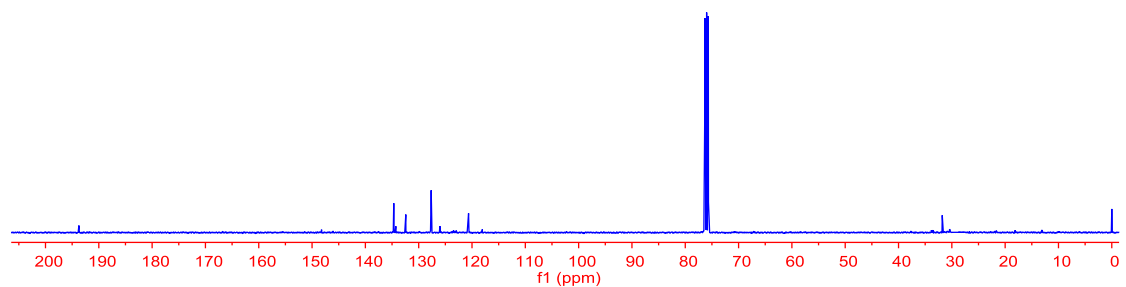
- 193.7

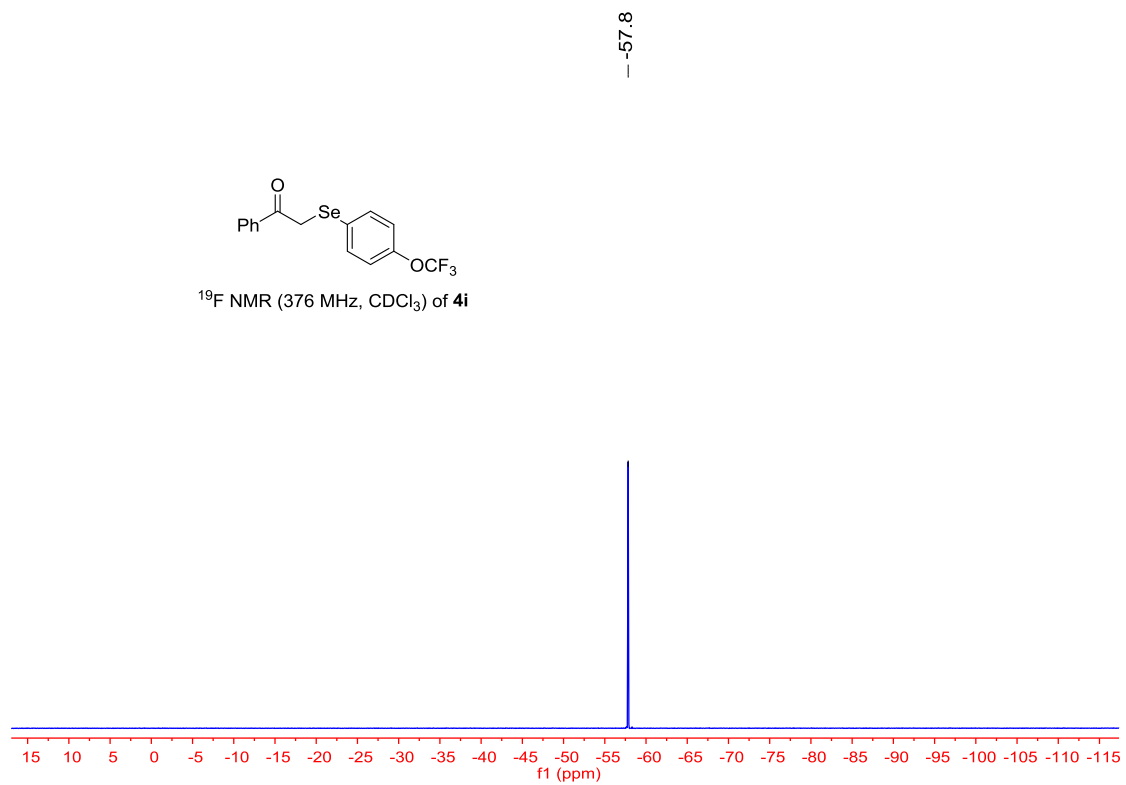
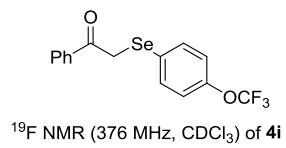
134.7
134.3
132.4
127.7
127.6
126.0
126.0
123.4
123.0
120.6

- 31.8

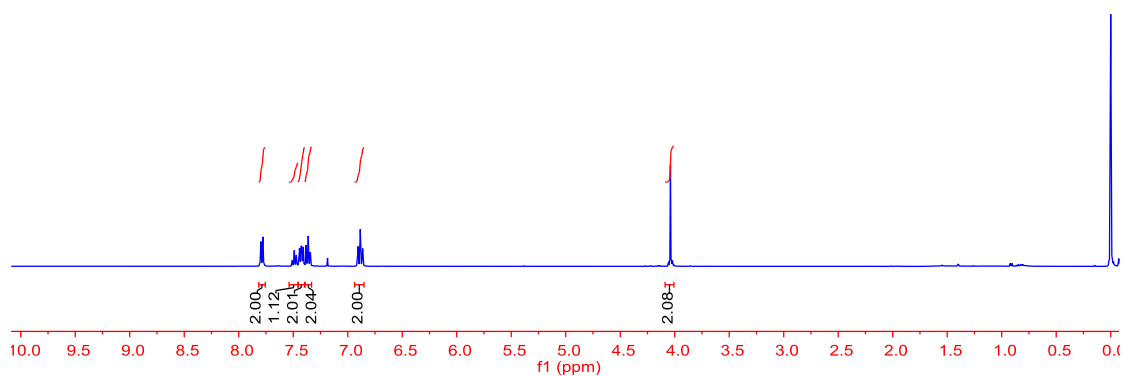
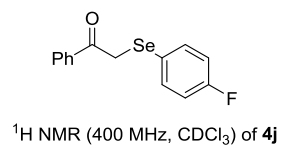


¹³C NMR (100 MHz, CDCl₃) of **4i**





7.80
 7.80
 7.80
 7.79
 7.79
 7.78
 7.78
 7.77
 7.51
 7.51
 7.49
 7.49
 7.49
 7.47
 7.47
 7.47
 7.44
 7.43
 7.43
 7.42
 7.41
 7.38
 7.37
 7.36
 7.35
 7.34
 6.91
 6.89
 6.86
 4.04

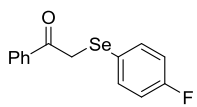


— 193.8

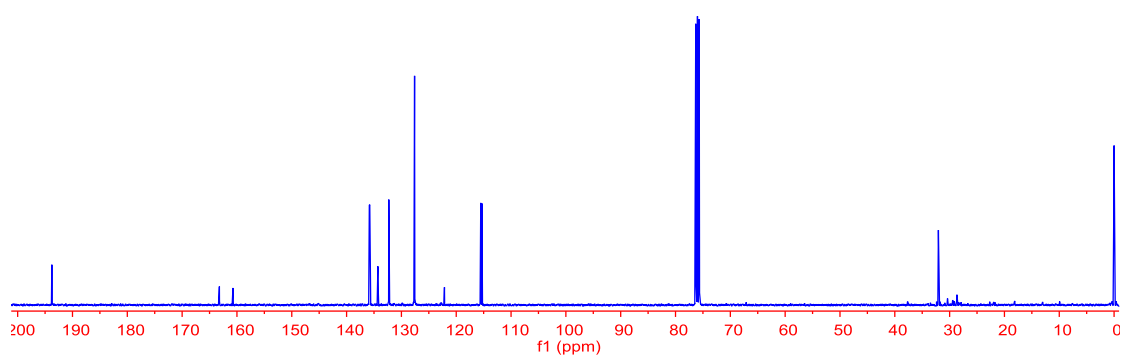
~ 163.2
~ 160.7

135.9
135.8
134.3
132.3
127.6
122.2
122.1
115.5
115.3

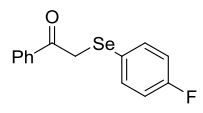
— 32.0



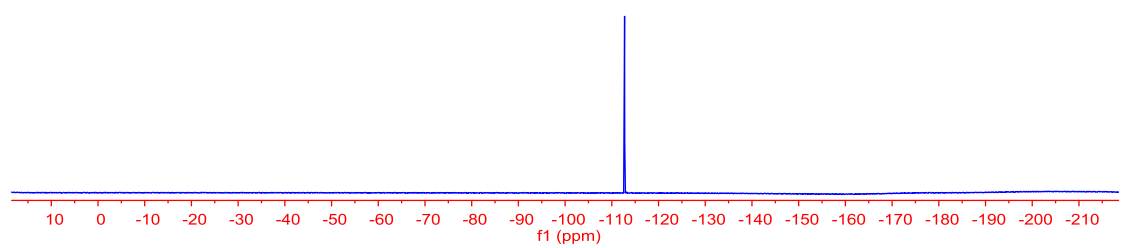
¹³C NMR (100 MHz, CDCl₃) of **4j**

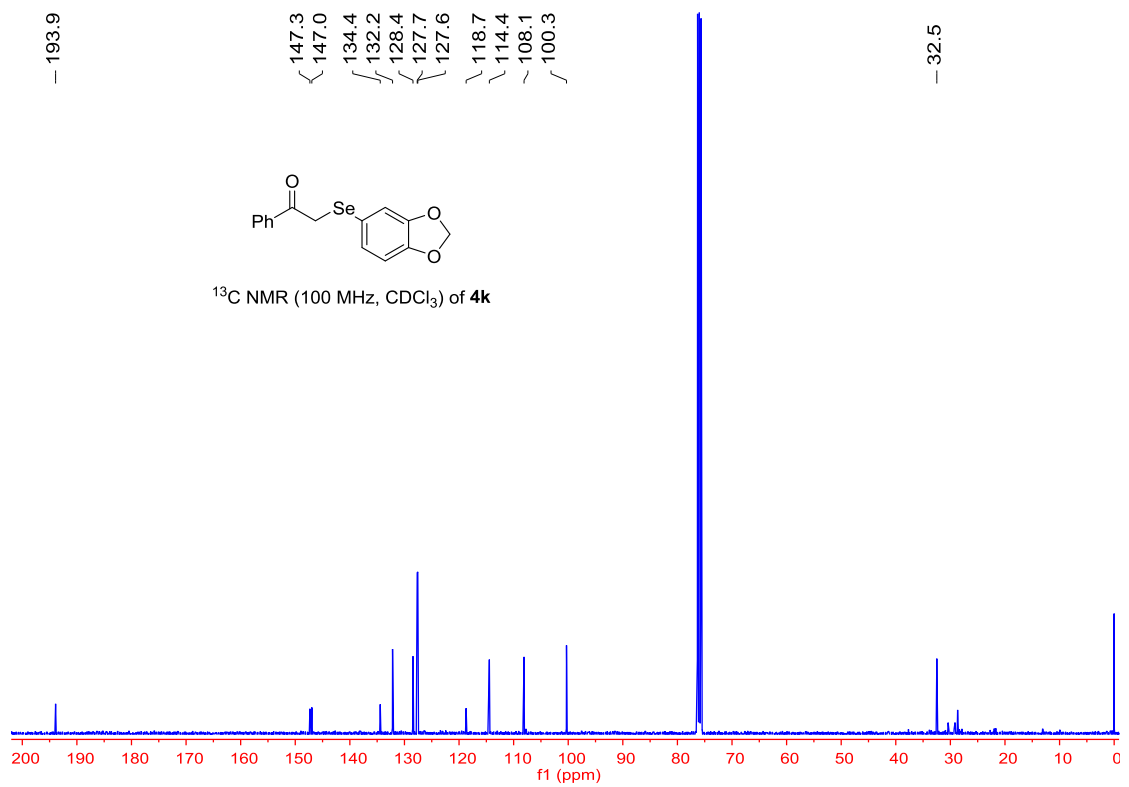
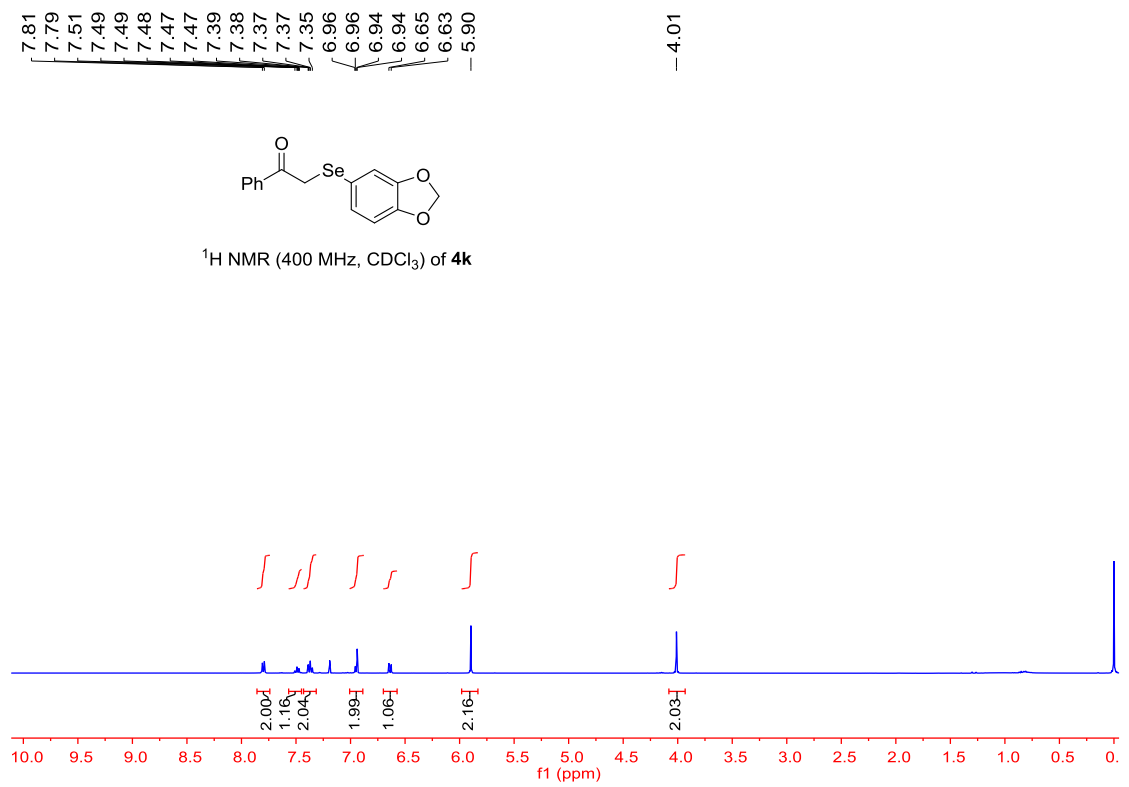


— -112.7

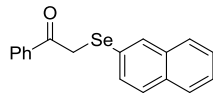


¹⁹F NMR (376 MHz, CDCl₃) of **4j**

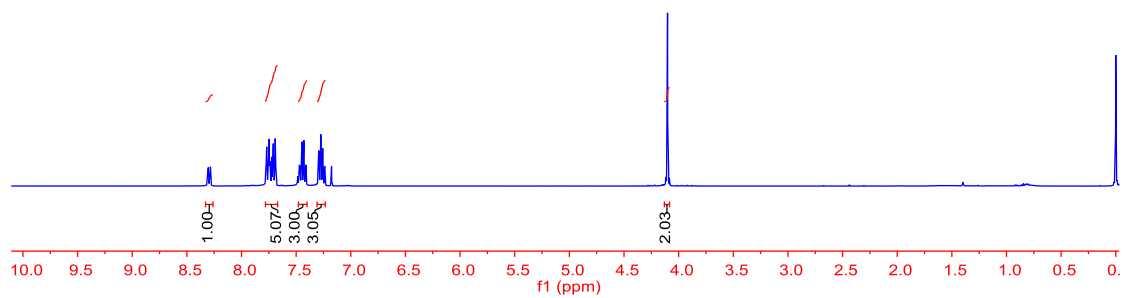




8.31
8.30
8.30
8.29
8.28
8.28
7.77
7.77
7.77
7.75
7.75
7.74
7.74
7.72
7.72
7.71
7.71
7.70
7.69
7.69
7.48
7.47
7.47
7.46
7.45
7.45
7.44
7.43
7.43
7.42
7.42
7.41
7.41
7.41
7.29
7.29
7.28
7.27
7.26
7.26
7.25
7.24
7.24
4.10



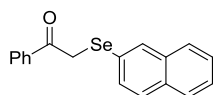
¹H NMR (400 MHz, CDCl₃) of 4I



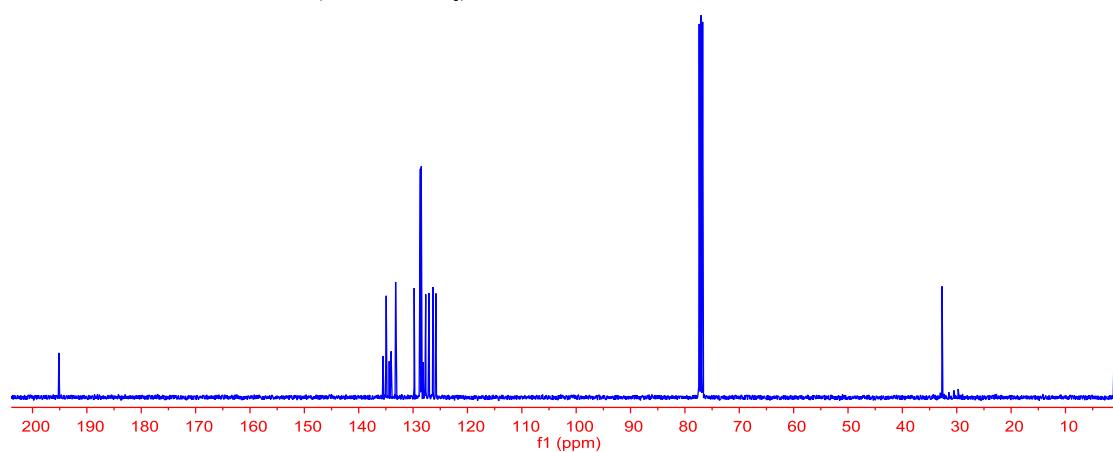
— 195.1

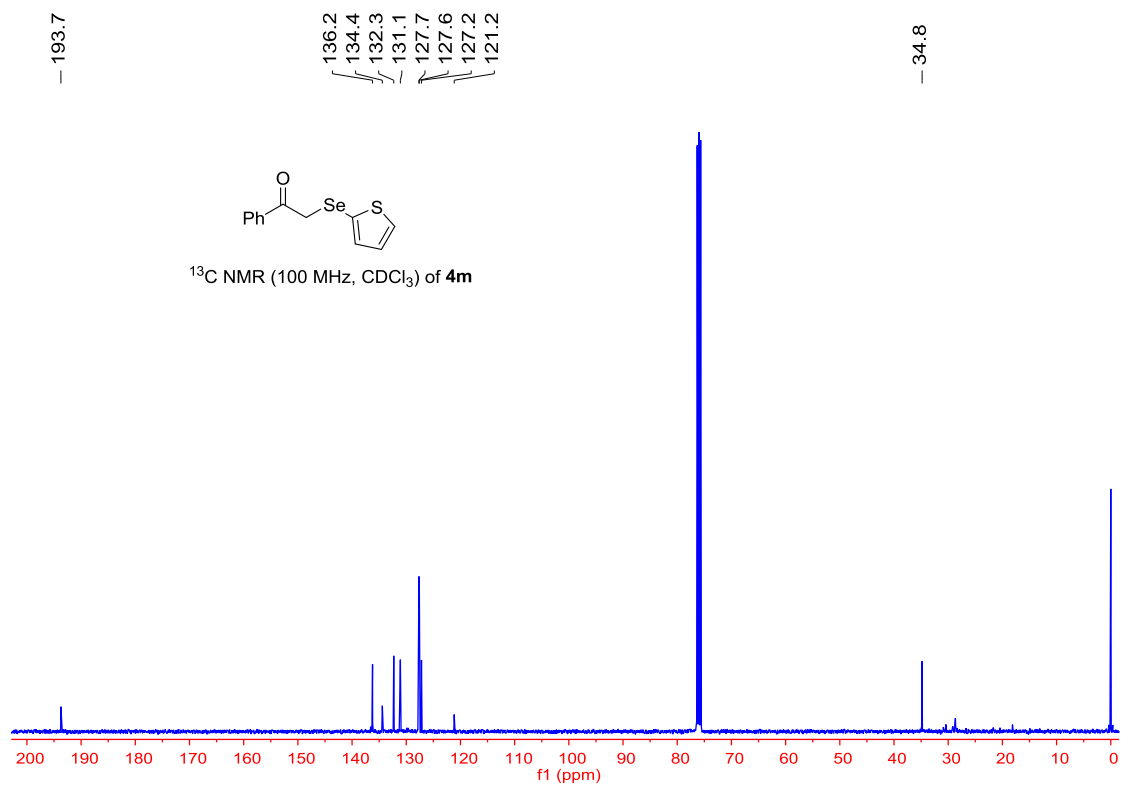
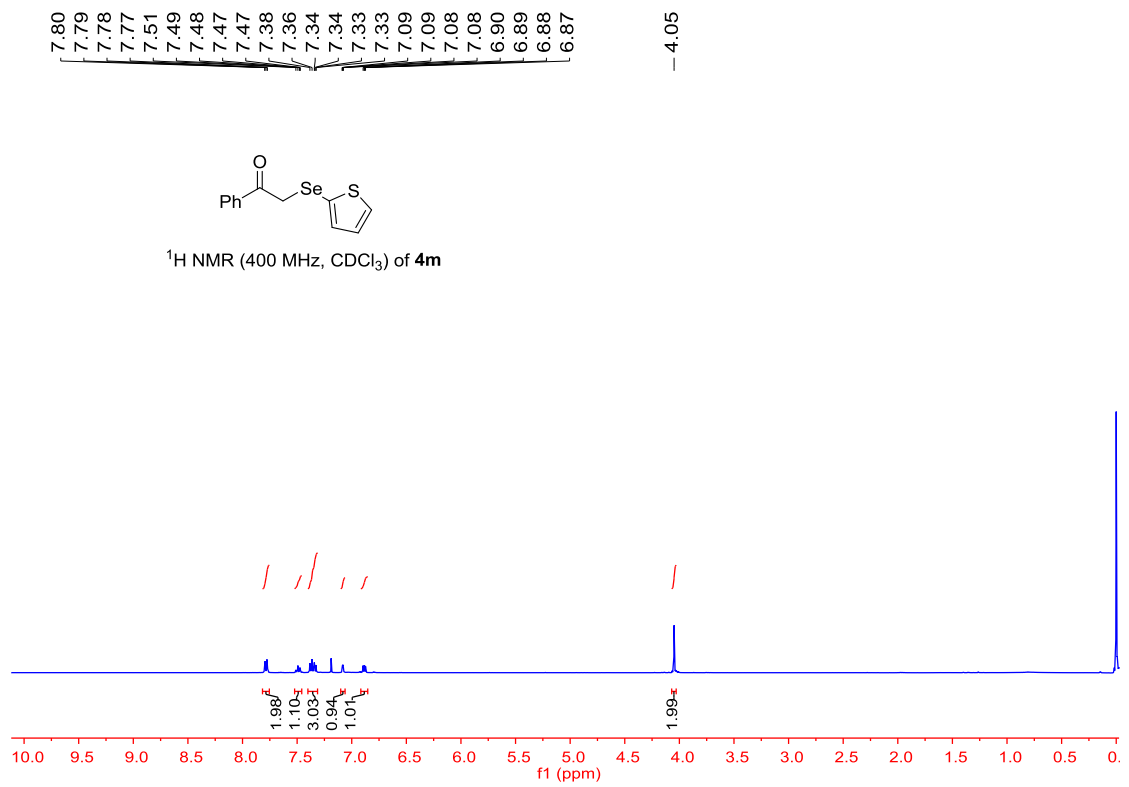
135.5
134.9
134.4
134.0
133.2
129.8
128.8
128.7
128.5
128.2
127.7
127.1
126.3
125.8

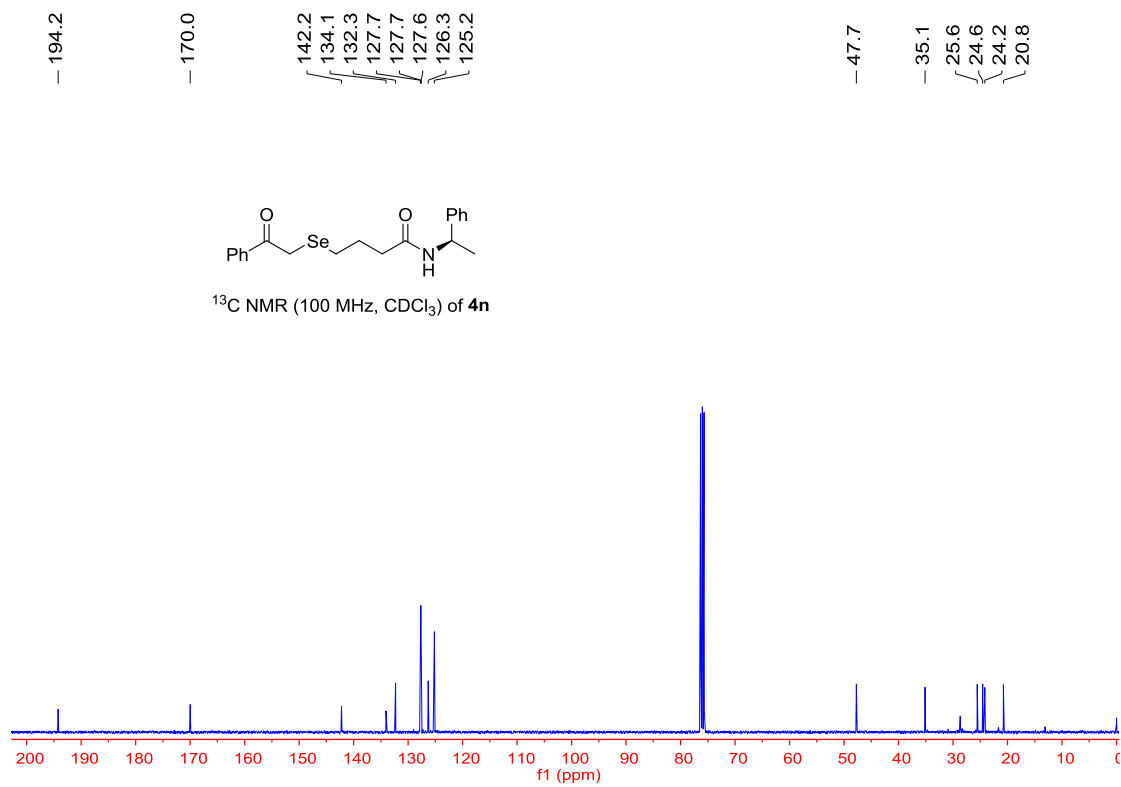
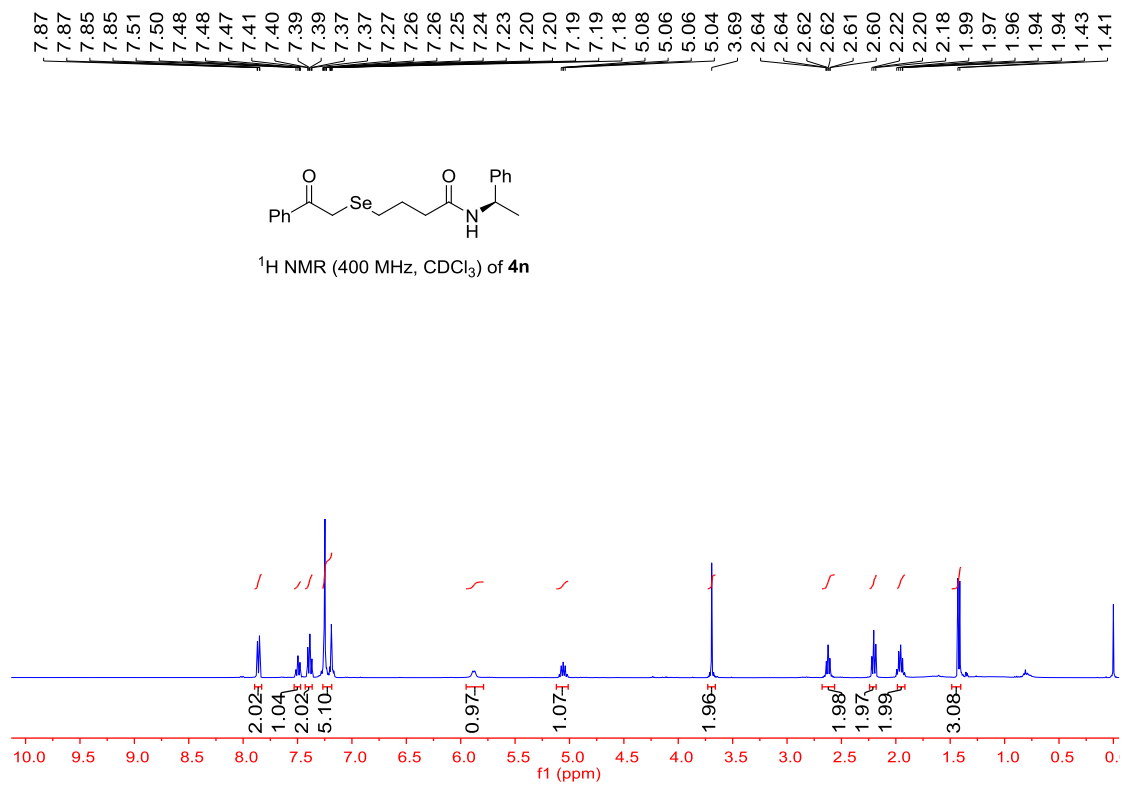
— 32.7



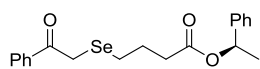
¹³C NMR (100 MHz, CDCl₃) of 4I



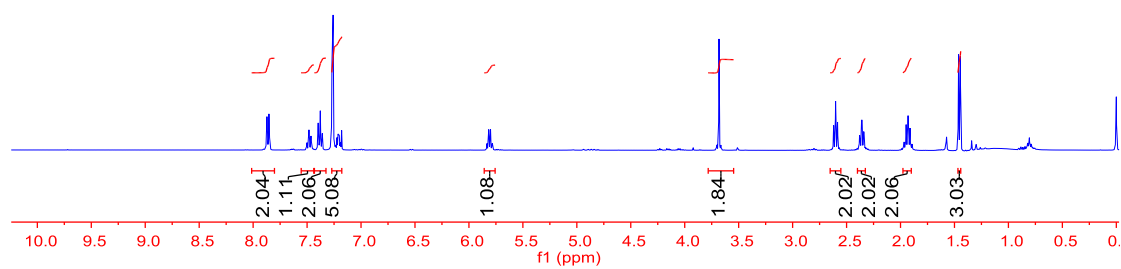




7.87
7.87
7.85
7.85
7.50
7.48
7.48
7.47
7.46
7.46
7.40
7.39
7.38
7.38
7.36
7.36
7.27
7.26
7.22
7.21
7.21
7.19
7.18
7.18
5.82
3.88
2.62
2.60
2.58
2.38
2.37
2.36
2.36
2.34
2.34
1.95
1.93
1.91
1.46
1.45



^1H NMR (400 MHz, CDCl_3) of **4o**



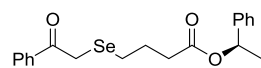
- 193.9

- 171.0

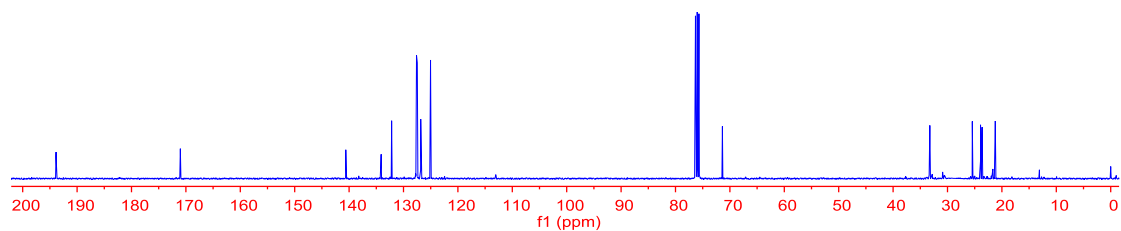
140.6
134.1
132.2
127.7
127.6
127.5
126.8
125.0

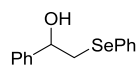
- 71.4

33.2
25.4
23.9
23.6
21.2

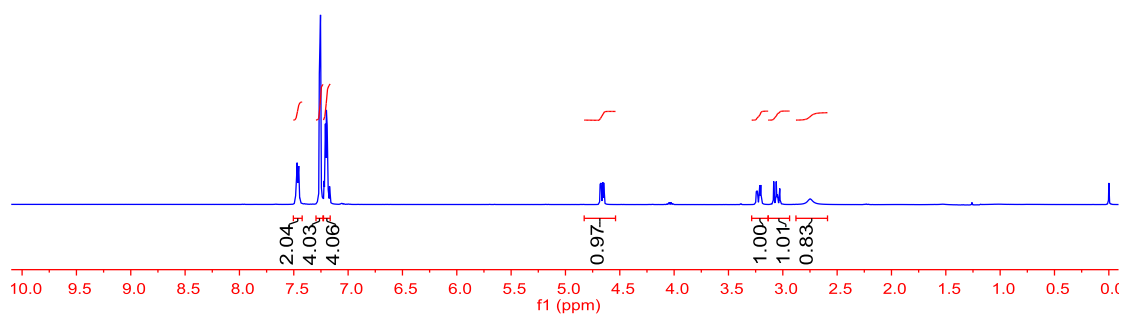


^{13}C NMR (100 MHz, CDCl_3) of **4o**





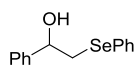
¹H NMR (400 MHz, CDCl₃) of **5**



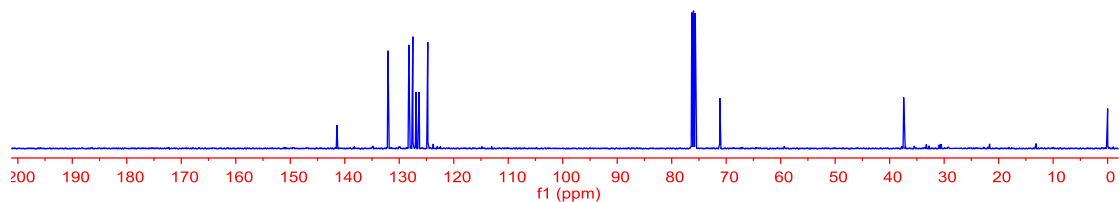
141.4
132.1
128.2
128.1
127.5
126.9
126.4
124.8

71.2

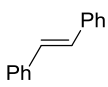
37.4



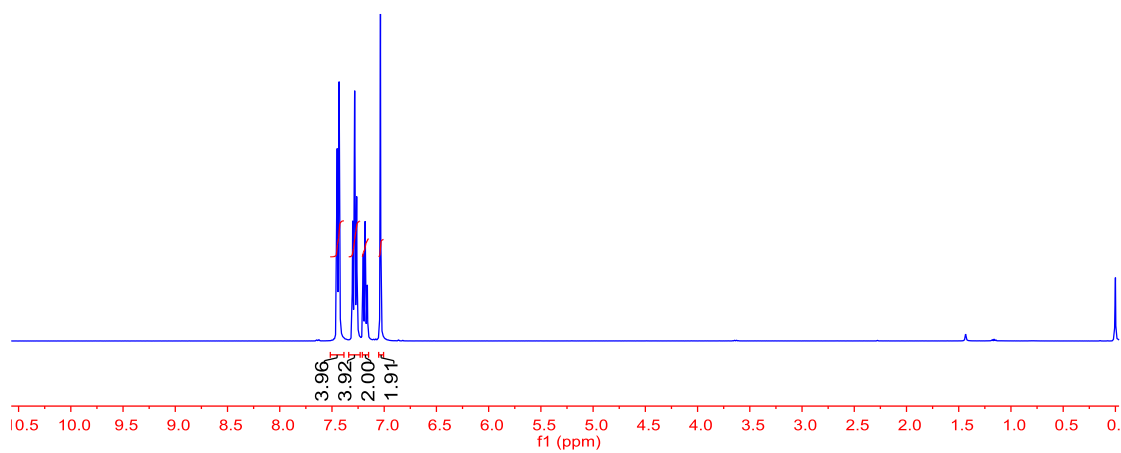
¹³C NMR (100 MHz, CDCl₃) of **5**



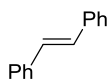
7.45
7.45
7.43
7.30
7.30
7.28
7.27
7.26
7.20
7.20
7.20
7.19
7.18
7.18
7.17
7.16
7.04



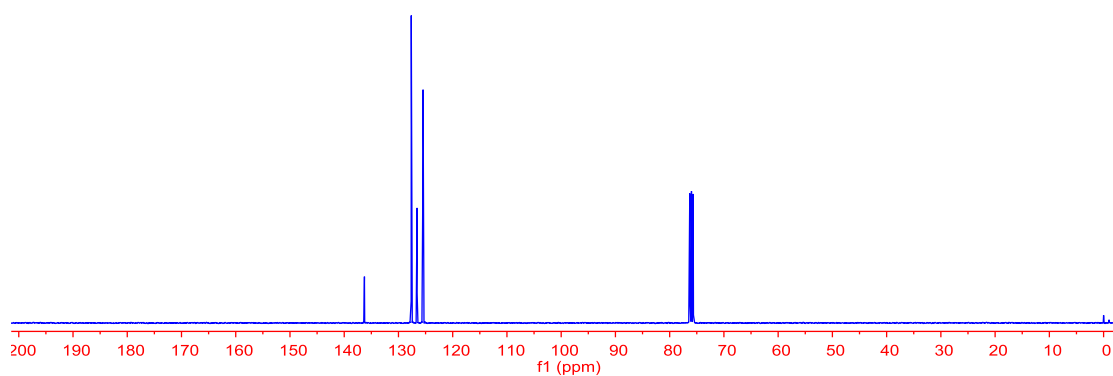
^1H NMR (400 MHz, CDCl_3) of **8**



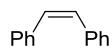
136.3
127.6
126.6
125.5



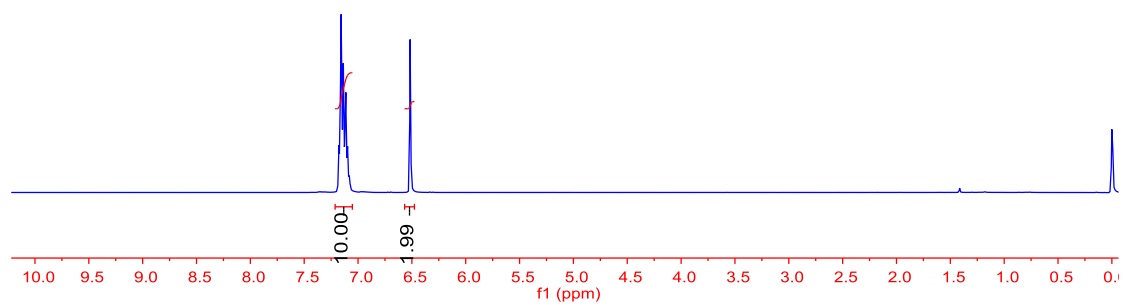
^{13}C NMR (100 MHz, CDCl_3) of **8**



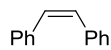
7.18
7.17
7.16
7.15
7.14
7.13
7.12
7.11
7.11
7.10
7.10
6.99



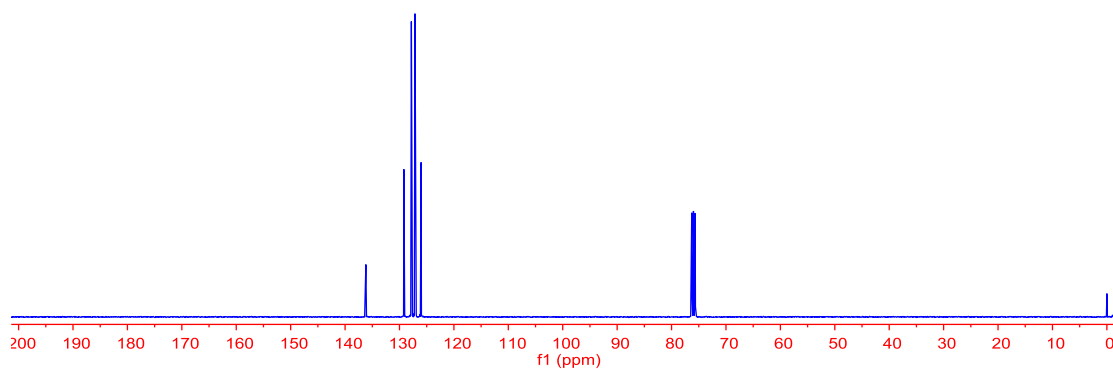
¹H NMR (100 MHz, CDCl₃) of **9**



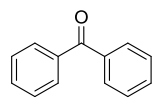
136.2
129.2
127.8
127.2
126.0



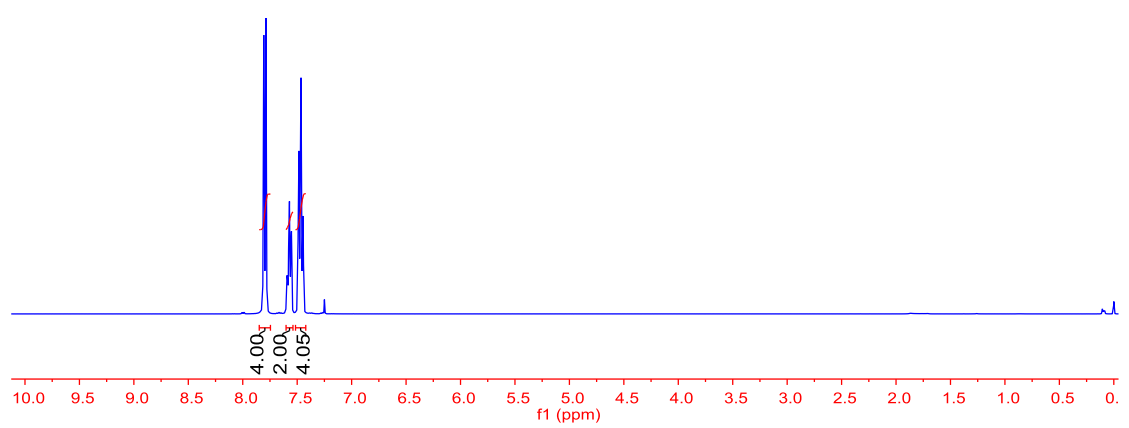
¹³C NMR (100 MHz, CDCl₃) of **9**



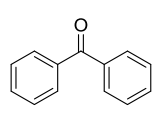
7.81
7.79
7.79
7.60
7.59
7.59
7.58
7.57
7.56
7.56
7.55
7.55
7.49
7.48
7.47
7.47
7.46
7.45
7.45



¹H NMR (400 MHz, CDCl₃) of 11



196.8
196.7
137.6
132.4
130.1
128.3



¹³C NMR (400 MHz, CDCl₃) of 11

