Supporting Information

$B(C_6F_5)_3$ -Catalyzed One-Pot Selenation of Phosphonates and Indoles

Ying Chen, Xin Li, Dongfang Wu, Ruijie Tao, Yong Ling and Gong-Qing Liu*

Organoselenium Synthesis and Function Laboratory, School of Pharmacy, Nantong University, Nantong 226019, People's Republic of China.

E-mail: gqliu@ntu.edu.cn

Content

1. General information	S2
2. Optimization of the reaction conditions	S2
3. Mechanistic studies	S3
1) IR spectroscopy studies	S3
2) NMR studies	S4
3) HCN generation detection	S4
4. General procedure for the synthesis of phosphoroselenoates	S6
5. General procedure for the synthesis of 3-selenylindoles	S18
6. References	S23
7. Copies of NMR spectra	S24

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.

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 GF_{254}). Visualization of the compounds was accomplished by projecting UV-light onto the developed plates.

Instrumentations

- a) 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.
- b) High resolution mass spectrometry (HRMS) analyses were carried out on a Thermo Fisher Q Exactive Mass Spectrometer.
- c) Melting points were determined on glass slides using a WRX-4 digital display microscopic melting point apparatus and were presented uncorrected.

2. Optimization of the reaction conditions

Table S1. Solvent screening ^a

entry	solvent	isolated yield (%)
1	CH ₂ Cl ₂	85
2	CHCl ₃	72
3	DCE	68
4	DMF	30
5	DMSO	23
6	EtOAc	43
7	THF	23
8	acetone	28
9	CH ₃ CN	37
10	Toluene	65

^a Reaction conditions: **1a** (0.25 mmol, 1.0 equiv.), **2a** (0.25 mmol, 1.0 equiv.), $B(C_6F_5)_3$ (0.0125 mmol, 0.05 equiv.), solvent (2 mL), r.t., N_2 , 5 h.

Table S2. Reaction times and amount of $B(C_6F_5)_3$ screening ^a

entry	X (mol%)	T (h)	isolated yield (%)
1	5	5	85
2	2	5	39
3	2	10	70
4	0	5	32
5	0	10	47

^a Reaction conditions: **1a** (0.25 mmol, 1.0 equiv.), **2a** (0.25 mmol, 1.0 equiv.), $B(C_6F_5)_3$, CH_2Cl_2 (2 mL), r.t., N_2 .

3. Mechanistic studies

1) IR spectroscopy studies

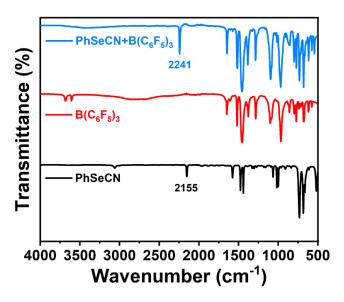


Figure S1. IR spectra of PhSeCN (black), $B(C_6F_5)_3$ (red) and a mixture of PhSeCN and $B(C_6F_5)_3$ (blue).

2) NMR studies

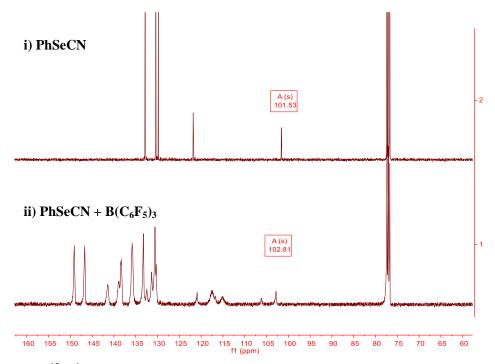


Figure S2. $^{13}C\{^{1}H\}$ NMR spectra in CDCl₃: i) PhSeCN, ii) A mixture of PhSeCN and B(C₆F₅)₃ (1:1).

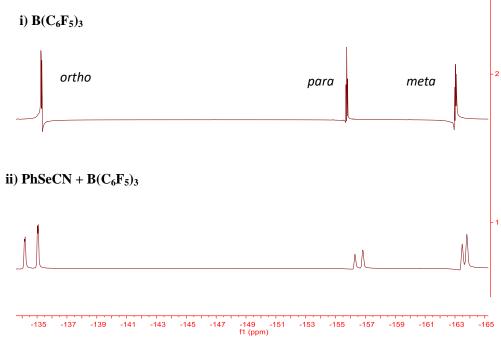
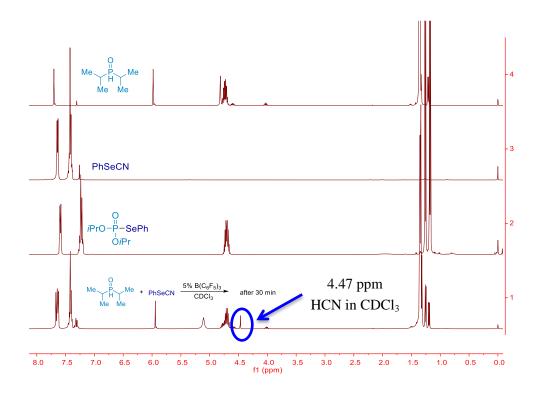


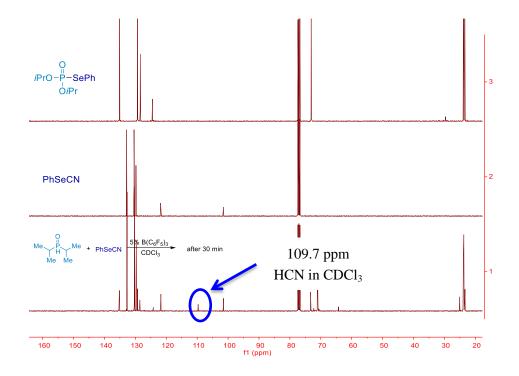
Figure S3: 19 F NMR spectra in CDCl₃: i) B(C₆F₅)₃, ii) A mixture of PhSeCN and B(C₆F₅)₃ (1:1).

3) HCN generation detection

$$\frac{O}{IPrO-P-H} + PhSeCN \xrightarrow{5 \text{ mol}\% B(C_6F_5)_3} \frac{O}{CH_2Cl_2, r.t., 30 \text{ min}}$$
 $\frac{O}{IPrO-P-SePh} + HCN = \frac{O}{IPrO-P-SePh} + \frac{O}{IPRO-P-SePh}$

Considering reaction pathway (Scheme 8 in the manuscript), the resulting hydrogen cyanide (HCN) would be produced during the reaction. To confirm this assumption, additional NMR studies were conducted. After running the reaction in an NMR tube at room temperature for 30 minutes, NMR spectroscopy is recorded. As expected, the generation of HCN was clearly detected by NMR.





4. General procedure for the synthesis of phosphoroselenoates

To a 20 mL round-bottom flask were added phosphonate (0.25 mmol), PhSeCN (0.25 mmol), $B(C_6F_5)_3$ (0.0125 mmol) and CH_2Cl_2 (2 mL) under N_2 atmosphere. This reaction mixture was allowed to stir for 5 h at room temperature. Then, 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.

O,O-Dimethyl *Se*-phenyl phosphoroselenoate (3a). Compound 3a was prepared according to the general procedure and isolated as an oil (56 mg, 85% yield) after flash chromatography (petroleum ether/ethyl acetate = 4/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.56 (d, J = 6.7 Hz, 2H), 7.44 – 6.97 (m, 3H), 3.73 (s, 3H), 3.69 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ /ppm = 134.6 (d, J_{C-P} = 4.7 Hz), 128.6 (d, J_{C-P} = 2.2 Hz), 127.9 (d, J_{C-P} = 2.7 Hz), 122.2 (d, J_{C-P} = 8.5 Hz), 53.0 (d, J_{C-P} = 5.7 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm =21.8.

Spectral data are in good agreement with literature values.¹

$$\begin{array}{c} \text{Me} \\ \text{O} \\ \text{O-P-Se} \\ \text{Me} \\ \text{O} \\ \text{Me} \end{array}$$

O,O-Diisopropyl *Se*-phenyl phosphoroselenoate (3b). Compound 3b was prepared according to the general procedure and isolated as an oil (66 mg, 82% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.59 (d, J = 8.1 Hz, 2H), 7.33 – 7.17 (m, 3H), 4.89 – 4.57 (m, 2H), 1.26 (d, J = 6.2 Hz, 6H), 1.18 (d, J = 6.2 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃) δ /ppm = 134.1 (d, J_{C-P} = 5.0 Hz), 128.3 (d, J_{C-P} = 1.9 Hz), 127.5 (d, J_{C-P} = 2.5 Hz), 123.5 (d, J_{C-P} = 8.3 Hz), 72.1 (d, J_{C-P} = 6.6 Hz), 22.9 (d, J_{C-P} = 3.8 Hz), 22.5 (d, J_{C-P} = 5.9 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 14.7.

Spectral data are in good agreement with literature values.¹

*O,O-Di-tert-*butyl *Se-*phenyl phosphoroselenoate (3c). Compound 3c was prepared according to the general procedure and isolated as an oil (70 mg, 80% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.62 (d, J = 8.0 Hz, 2H), 7.46 – 7.02 (m, 3H), 1.39 (s, 18H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 134.7 (d, $J_{C-P} = 4.7$ Hz), 128.0 (d, $J_{C-P} = 2.2$ Hz), 127.2 (d, $J_{C-P} = 2.7$ Hz), 124.9 (d, $J_{C-P} = 8.9$ Hz), 84.4 (d, $J_{C-P} = 10.4$ Hz), 29.2 (d, $J_{C-P} = 4.4$ Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm =5.0.

Spectral data are in good agreement with literature values.²

O,O-Dibutyl *Se*-phenyl phosphoroselenoate (3d). Compound 3d was prepared according to the general procedure and isolated as an oil (79 mg, 91% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.57 (d, J = 8.4 Hz, 2H), 7.40 – 7.07 (m, 3H), 4.31 – 3.78 (m, 4H), 1.55 (dq, J = 8.2, 6.6 Hz, 4H), 1.38 – 1.21 (m, 4H), 0.82 (t, J = 7.4 Hz, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 134.5 (d, $J_{C-P} = 4.7$ Hz), 128.4 (d, $J_{C-P} = 2.1$ Hz), 127.7 (d, $J_{C-P} = 2.5$ Hz), 122.8 (d, $J_{C-P} = 8.3$ Hz), 66.5 (d, $J_{C-P} = 6.5$ Hz), 31.0 (d, $J_{C-P} = 7.3$ Hz), 17.7, 12.5.

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 18.0.

Spectral data are in good agreement with literature values.¹

O,O-Dibenzyl *Se*-phenyl phosphoroselenoate (3e). Compound 3e was prepared according to the general procedure and isolated as an oil (85 mg, 81% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.48 (d, J = 8.1 Hz, 2H), 7.30 – 7.21 (m, 7H), 7.21 – 7.11 (m, 6H), 5.07 – 4.98 (m, 4H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 134.8 (d, J_{C-P} = 4.7 Hz), 134.2 (d, J_{C-P} = 7.8 Hz), 128.5 (d, J_{C-P} = 2.2 Hz), 127.9 (d, J_{C-P} = 2.7 Hz), 127.52, 127.50, 127.0, 122.2 (d, J_{C-P} = 8.6 Hz), 68.1 (d, J_{C-P} = 6.1 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 18.5.

Spectral data are in good agreement with literature values.¹

O,O,Se-Triphenyl phosphoroselenoate (3f). Compound 3f was prepared according to the general procedure and isolated as an oil (75 mg, 77% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.55 – 7.37 (m, 2H), 7.33 – 7.14 (m, 7H), 7.15 – 7.07 (m, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 149.2 (d, J_{C-P} = 8.6 Hz), 135.2 (d, J_{C-P} = 4.8 Hz), 128.7 (d, J_{C-P} = 1.3 Hz), 128.5 (d, J_{C-P} = 2.5 Hz), 128.3 (d, J_{C-P} = 2.9 Hz), 124.6 (d, J_{C-P} = 1.6 Hz), 121.6 (d, J_{C-P} = 9.1 Hz), 119.6 (d, J_{C-P} = 5.1 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 9.5.

Spectral data are in good agreement with literature values.¹

6-(Phenylselanyl)dibenzo[c,e][1,2]oxaphosphinine 6-oxide (3g). Compound 3g was prepared according to the general procedure and isolated as an oil (70 mg, 75% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.80 (ddd J = 14.9, 7.7 Hz, 1H), 7.69 (dd, J = 8.1, 6.5 Hz, 1H), 7.60 – 7.49 (m, 2H), 7.39 (tdd, J = 7.5, 3.6, 1.0 Hz, 1H), 7.31 – 7.20 (m, 1H), 7.17 (dq, J = 8.2, 2.1 Hz, 2H), 7.12 – 7.02 (m, 3H), 6.92 (t, J = 7.6 Hz, 2H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 149.3 (d, J_{C-P} = 9.8 Hz), 135.9 (d, J_{C-P} = 3.7 Hz), 134.9 (d, J_{C-P} = 7.5 Hz), 132.7 (d, J_{C-P} = 2.7 Hz), 129.5 (t, J_{C-P} = 5.5 Hz), 128.0 (dd, J_{C-P} = 5.2, 2.7 Hz), 127.4 (d, J_{C-P} = 15.0 Hz), 125.6, 124.4, 123.9, 123.9, 123.7,

122.2 (d, J = 11.2 Hz), 120.9 (d, $J_{C-P} = 7.1$ Hz), 120.8 (d, $J_{C-P} = 11.7$ Hz), 119.0 (d, $J_{C-P} = 7.0$ Hz).

³¹**P NMR** (162 MHz, CDCl₃) $\delta/ppm = 31.4$.

Spectral data are in good agreement with literature values.³

Se-Phenyl diphenylphosphinoselenoate (**3h**). Compound **3h** was prepared according to the general procedure and isolated as an oil (70 mg, 78% yield) after flash chromatography (petroleum ether/ethyl acetate = 4/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.87 – 7.64 (m, 4H), 7.51 – 7.26 (m, 8H), 7.16 (t, J = 8.0 Hz, 1H), 7.07 (t, J = 7.5 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 136.3 (d, J_{C-P} = 3.3 Hz), 133.9, 133.0, 132.3 (d, J_{C-P} = 3.3 Hz), 131.4 (d, J_{C-P} = 10.6 Hz), 129.3 (d, J_{C-P} = 1.7 Hz), 128.5 (d, J_{C-P} = 13.2 Hz), 123.7 (d, J_{C-P} = 15.7 Hz)..

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 40.0.

Spectral data are in good agreement with literature values.¹

Se-Phenyl di(naphthalen-2-yl)phosphinoselenoate (**3i**). Compound **3i** was prepared according to the general procedure and isolated as a solid (83 mg, 73% yield) after flash chromatography (petroleum ether/ethyl acetate = 2/1). Mp= 230 – 231 °C.

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 8.39 – 8.31 (m, 2H), 7.90 – 7.73 (m, 6H), 7.53 – 7.43 (m, 7H), 7.20 – 7.01 (m, 4H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 136.4 (d, J = 3.3 Hz), 134.8 (d, J = 2.7 Hz), 133.6 (d, J = 9.6 Hz), 132.4 (d, J = 14.4 Hz), 131.5, 130.9 (d, J = 5.8 Hz), 129.3 (d, J = 1.7 Hz), 129.2, 128.5, 128.4, 127.8, 127.7, 127.1, 126.1 (d, J = 12.0 Hz).

³¹**P NMR** (162 MHz, CDCl₃) $\delta/ppm = 40.0$.

Spectral data are in good agreement with literature values.⁴

Se-Phenyl bis(3,5-dimethylphenyl)phosphinoselenoate (3j). Compound 3j was prepared according to the general procedure and isolated as a solid (74 mg, 72% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1). Mp= 262 - 263 °C.

¹**H NMR** (400 MHz, CDCl₃) $\delta/ppm = 7.56 - 7.47$ (m, 1H), 7.42 (d, J = 8.3 Hz, 2H),

7.38 - 7.27 (m, 4H), 7.22 - 7.14 (m, 2H), 7.09 (dd, J = 8.3, 6.7 Hz, 1H), 7.02 (s, 1H), 2.23 (s, 12H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 138.2 (d, J_{C-P} = 13.9 Hz), 136.4 (d, J_{C-P} = 3.2 Hz), 134.0 (d, J_{C-P} = 3.3 Hz), 133.2 (d, J_{C-P} = 96.0 Hz), 131.5, 129.2 (d, J_{C-P} = 2.3 Hz), 128.9 (d, J_{C-P} = 10.5 Hz), 124.2 (d, J_{C-P} = 5.6 Hz), 21.3.

³¹**P NMR** (162 MHz, CDCl₃) $\delta/ppm = 41.5$.

Spectral data are in good agreement with literature values.³

O-Ethyl *Se*-phenyl phenylphosphonoselenoate (3k). Compound 3k was prepared according to the general procedure and isolated as an oil (65 mg, 80% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.58 - 7.47 (m, 2H), 7.44 - 7.40 (m, 1H), 7.32 - 7.25 (m, 4H), 7.23 - 7.18 (m, 2H), 7.11 (t, J = 7.6 Hz, 1H), 4.39 - 4.18 (m, 2H), 1.34 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ /ppm = 136.5 (d, J_{C-P} = 3.6 Hz), 132.8 (d, J_{C-P} = 138.0 Hz), 132.5 (d, J_{C-P} = 3.3 Hz), 131.1 (d, J_{C-P} = 11.0 Hz), 129.4 (d, J_{C-P} = 2.2 Hz), 128.8 (d, J_{C-P} = 2.5 Hz), 128.2 (d, J_{C-P} = 14.9 Hz), 124.2 (d, J_{C-P} = 6.7 Hz), 62.6 (d, J_{C-P} = 7.1 Hz), 16.2 (d, J_{C-P} = 7.1 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 36.7

Spectral data are in good agreement with literature values.⁴

The corresponding aniline (0.25 mmol) was dissolved in ethanol and 50% HBF $_4$ (0.25 mmol). The resulting solution was cooled to 0 °C, and 90% solution of tertbutylnitrite (0.25 mmol) was then added dropwise. The mixture was allowed to warm to room temperature with stirring for 1 h. The precipitates were collected by filtration and washed with diethyl ether three times.

The resulting aryl diazonium salt and KSeCN (0.375 mmol, 1.5 equiv.) were dissolved in CH₃CN, and the mixture was stirred at 0 °C for 0.5 h under open air conditions. Subsequently, the reaction mixture was poured into water and extracted with dichloromethane three times. The combined organic layers were dried over anhydrous sodium sulfate, and the solvent was removed under reduced pressure to afford the aryl selenocyanates.

Next, the obtained selenocyanates, dimethyl phosphonate (0.25 mmol), $B(C_6F_5)_3$ (0.0125 mmol) were dissolved in CH_2Cl_2 under N_2 atmosphere. The mixture was stirred at room temperature for 5 h. Purification by column chromatography (petroleum ether/ethyl acetate) afforded the desired product **4**.

Se-(4-Fluorophenyl) O,O-dimethyl phosphoroselenoate (4a). Compound 4a was prepared according to the general procedure and isolated as an oil (51 mg, 72% yield) after flash chromatography (petroleum ether/ethyl acetate = 2/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.54 (dd, J = 8.8, 5.2 Hz, 2H), 6.98 – 6.93 (m, 2H), 3.74 (s, 3H), 3.71 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 162.3 (dd, J_{C-F} = 249.7, J_{C-P} = 3.0 Hz), 136.7 (dd, J_{C-P} = 8.3, J_{C-P} =4.4 Hz), 116.8 (dd, J_{C-F} = 8.7, J_{C-P} = 3.5 Hz), 115.9 (dd, J_{C-F} = 21.9, J_{C-P} = 2.3 Hz), 53.1 (d, J_{C-P} = 5.9 Hz).

¹⁹**F NMR** (376 MHz, CDCl₃) δ/ppm = -111.7 (d, J_{P-F} = 4.8 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 21.5 (d, J_{P-F} = 4.8 Hz)

Spectral data are in good agreement with literature values.⁵

Se-(4-Chlorophenyl) O,O-dimethyl phosphoroselenoate (4b). Compound 4b was prepared according to the general procedure and isolated as an oil (58 mg, 77% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.50 (d, J = 8.5 Hz, 2H), 7.22 (d, J = 8.5 Hz, 2H), 3.74 (s, 3H), 3.71 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 135.9 (d, J_{C-P} = 4.6 Hz), 134.5 (d, J_{C-P} = 3.1 Hz), 128.8 (d, J_{C-P} = 2.2 Hz), 120.3 (d, J_{C-P} = 8.8 Hz), 53.1 (d, J_{C-P} = 5.9 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 21.2

Spectral data are in good agreement with literature values.⁵

Se-(4-Bromophenyl) O,O-dimethyl phosphoroselenoate (**4c**). Compound **4c** was prepared according to the general procedure and isolated as a solid (69 mg, 80% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1). Mp= 96 - 97 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.44 (d, J = 8.5 Hz, 2H), 7.38 (d, J = 8.5 Hz, 2H), 3.74 (d, J = 13.2 Hz, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 137.1 (d, $J_{C-P} = 4.5$ Hz), 132.8 (d, $J_{C-P} = 2.1$ Hz), 123.8 (d, $J_{C-P} = 3.3$ Hz), 122.1 (d, $J_{C-P} = 8.6$ Hz), 54.2 (d, $J_{C-P} = 5.8$ Hz).

³¹**P NMR** (162 MHz, CDCl₃) $\delta/ppm = 21.1$.

Spectral data are in good agreement with literature values.⁴

Se-(4-(tert-Butyl)phenyl) O,O-dimethyl phosphoroselenoate (**4d**). Compound **4d** was prepared according to the general procedure and isolated as an oil (67 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 1/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.47 (dd, J = 8.5 Hz, 2H), 7.26 (d, J = 8.5 Hz, 2H), 3.75 (s, 3H), 3.71 (s, 3H), 1.23 (s, 9H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 151.2 (d, J_{C-P} = 2.9 Hz), 134.3 (d, J_{C-P} = 4.4 Hz), 125.8 (d, J_{C-P} = 2.3 Hz), 118.5 (d, J_{C-P} = 8.6 Hz), 52.9 (d, J_{C-P} = 5.6 Hz), 30.2.

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 22.3.

Spectral data are in good agreement with literature values.²

Se-(4-Methoxyphenyl) O,O-dimethyl phosphoroselenoate (4e). Compound 4e was prepared according to the general procedure and isolated as an oil (48 mg, 65% yield) after flash chromatography (petroleum ether/ethyl acetate = 2/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.46 (d, J = 8.8 Hz, 2H), 6.78 (d, J = 8.8 Hz, 2H), 3.72 (s, 3H), 3.71 (s, 3H), 3.70 (s, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 159.4 (d, J_{C-P} = 2.8 Hz), 136.3 (d, J_{C-P} = 4.2 Hz), 114.3 (d, J_{C-P} = 2.4 Hz), 112.0 (d, J_{C-P} = 8.7 Hz), 54.3, 52.9 (d, J_{C-P} = 5.7 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ/ppm = 22.2

Spectral data are in good agreement with literature values.⁵

$$\begin{array}{c} O \\ \text{MeO-P-Se-} \\ O \text{Me} \end{array} \longrightarrow \begin{array}{c} O \\ \text{OCF}_3 \end{array}$$

O,O-Dimethyl *Se*-(4-(trifluoromethoxy)phenyl) phosphoroselenoate (4f). Compound 4f was prepared according to the general procedure and isolated as an oil (61 mg, 70% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.60 (dd, J = 8.8 Hz, 2H), 7.10 (d, J = 8.8 Hz, 2H), 3.75 (s, 3H), 3.72 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 151.32 – 145.90 (m), 136.1 (d, $J_{C-P} = 4.6 \text{ Hz}$), 120.9 (d, $J_{C-P} = 2.1 \text{ Hz}$), 120.4 (d, $J_{C-P} = 8.6 \text{ Hz}$), 119.3 (q, $J_{C-F} = 256.0 \text{ Hz}$), 53.1 (d, $J_{C-P} = 5.9 \text{ Hz}$).

¹⁹**F NMR** (376 MHz, CDCl3) δ /ppm = -57.9.

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 21.0.

HRMS: $m/z [M + H]^+$ calcd for $C_9H_{11}F_3O_4PSe$, 350.9507; found, 350.9517.

Se-(2,4-Dichlorophenyl) O,O-dimethyl phosphoroselenoate (4g). Compound 4g was prepared according to the general procedure and isolated as a solid (58 mg, 70% yield) after flash chromatography (petroleum ether/ethyl acetate = 8/1). Mp= 104 - 105 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.66 (dd, J = 8.4, 1.9 Hz, 1H), 7.41 (dd, J = 2.3, 0.8 Hz, 1H), 7.14 (dd, J = 8.5, 2.3 Hz, 1H), 3.77 (d, J = 13.3 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃) δ /ppm = 138.7 (d, J = 6.3 Hz), 138.2 (d, J_{C-P} = 3.5 Hz), 136.0 (d, J_{C-P} = 2.7 Hz), 129.9 (d, J_{C-P} = 2.1 Hz), 128.1 (d, J_{C-P} = 2.0 Hz), 122.8 (d, J_{C-P} = 8.3 Hz), 54.3 (d, J_{C-P} = 5.6 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 20.3.

HRMS: m/z $[M + H]^+$ calcd for $C_8H_{10}Cl_2O_3PSe$, 334.8904; found, 334.8995.

O,O-Dimethyl *Se*-(naphthalen-2-yl) phosphoroselenoate (4h). Compound 4h was prepared according to the general procedure and isolated as an oil (50 mg, 64% yield) after flash chromatography (petroleum ether/ethyl acetate = 2/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.38 (d, J = 8.4 Hz, 1H), 8.04 – 7.84 (m, 1H), 7.86 – 7.80 (m, 1H), 7.79 – 7.74 (m, 1H), 7.53 (ddd, J = 8.4, 6.8, 1.4 Hz, 1H), 7.45 (ddd, J = 8.1, 6.9, 1.2 Hz, 1H), 7.34 (t, J = 7.7 Hz, 1H), 3.66 (s, 3H), 3.63 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 135.5 (d, J_{C-P} = 4.9 Hz), 133.9 (d, J_{C-P} = 3.3 Hz), 133.2 (d, J_{C-P} = 2.2 Hz), 129.4 (d, J_{C-P} = 3.2 Hz), 127.7, 126.9, 126.2, 125.5, 124.9 (d, J_{C-P} = 3.2 Hz), 121.6 (d, J_{C-P} = 9.4 Hz), 53.1 (d, J_{C-P} = 6.0 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ/ppm = 21.2.

Spectral data are in good agreement with literature values.¹

Se-(Benzo[d][1,3]dioxol-5-yl) O,O-dimethyl phosphoroselenoate (4i). Compound 4i was prepared according to the general procedure and isolated as an oil (48 mg, 62% yield) after flash chromatography (petroleum ether/ethyl acetate = 2/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.21 – 7.02 (m, 2H), 6.77 (d, J = 8.0 Hz, 1H), 5.99 (s, 2H), 3.82 (s, 3H), 3.79 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ /ppm = 148.8 (d, J_{C-P} = 3.0 Hz), 148.4 (d, J_{C-P} = 2.5 Hz), 130.1 (d, J_{C-P} = 5.3 Hz), 113.9 (d, J_{C-P} = 9.0 Hz), 109.4 (d, J_{C-P} = 2.7 Hz), 101.9, 54.0 (d, J_{C-P} = 5.7 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm =22.2.

Spectral data are in good agreement with literature values.²

Se-(2-Methoxy-4-nitrophenyl) *O,O-***dimethyl phosphoroselenoate** (**4j**). Compound **4j** was prepared according to the general procedure and isolated as a solid (58 mg, 68% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1). Mp= 75 – 76 °C.

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.83 (dd, J = 8.5, 1.5 Hz, 1H), 7.72 (dd, J = 8.6, 2.3 Hz, 1H), 7.68 – 7.60 (m, 1H), 3.93 (s, 3H), 3.77 (d, J = 13.4 Hz, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 158.4 (d, J = 5.1 Hz), 148.9 (d, J = 1.9 Hz), 135.1 (d, J = 4.2 Hz), 123.2 (d, J = 7.4 Hz), 116.5 (d, J = 1.5 Hz), 105.4 (d, J = 1.4 Hz), 56.6, 54.3 (d, J = 5.5 Hz).

³¹**P NMR** (162 MHz, CDCl₃) $\delta/ppm = 20.2$.

HRMS: $m/z [M + H]^+$ calcd for $C_9H_{13}NO_6PSe$, 341.9640; found, 341.9636.

$$\begin{array}{c|c} \text{Alkyl-Br} & \text{CH}_3\text{CN} \\ + \\ \text{KSeCN} & 30 \text{ min, r.t} \\ \end{array} \begin{array}{c} \text{Alkyl-SeCN} \\ \text{without purification} \\ \end{array} \begin{array}{c} 5 \text{ mol% B(C}_6\text{F}_5)_3 \\ \text{dimethyl phosphonate} \\ \text{CH}_2\text{Cl}_2, \text{ r.t., 5 h} \\ \text{OMe} \\ \end{array} \begin{array}{c} \text{OMe} \\ \text{OMe} \\ \end{array}$$

The reaction mixture of alkyl bromides (0.25 mmol) and KSeCN (0.375 mmol, 1.5 equiv.) was stirred in anhydrous CH_3CN at room temperature for 0.5 h. Subsequently, the reaction mixture was poured into water and extracted with dichloromethane three times. The combined organic layers were dried over anhydrous sodium sulfate, and the solvent was removed under reduced pressure to afford the alkyl selenocyanates. Next, the obtained selenocyanates, dimethyl phosphonate (0.25 mmol), $B(C_6F_5)_3$ (0.0125 mmol) were dissolved in CH_2Cl_2 under N_2 atmosphere. The mixture was stirred at room temperature for 5 h. Purification by column chromatography (petroleum ether/ethyl acetate) afforded the desired product 5.

Se-Benzyl O,O-dimethyl phosphoroselenoate (5a). Compound 5a was prepared according to the general procedure and isolated as an oil (50 mg, 72% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.30 – 7.15 (m, 5H), 4.00 (d, J = 12.9 Hz, 2H), 3.61 (d, J = 13.3 Hz, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 138.3 (d, $J_{C-P} = 4.5$ Hz), 129.0, 128.7, 127.5, 53.55 (d, $J_{C-P} = 5.3$ Hz), 29.4 (d, $J_{C-P} = 4.6$ Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 24.6.

Spectral data are in good agreement with literature values.⁶

Se-(4-Fluorobenzyl) O,O-dimethyl phosphoroselenoate (**5b**). Compound **5b** was prepared according to the general procedure and isolated as an oil (60 mg, 81% yield)

after flash chromatography (petroleum ether/ethyl acetate = 4/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.26 (dd, J = 8.6, 5.4 Hz, 2H), 6.97 – 6.85 (m, 2H), 3.98 (d, J = 13.3 Hz, 2H), 3.61 (d, J = 13.3 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 162.0 (d, J_{C-F} = 246.7 Hz), 134.95 – 133.69 (m), 130.6 (d, J_{C-F} = 8.3 Hz), 115.6 (d, J_{C-F} = 21.6 Hz), 53.6 (d, J_{C-P} = 5.4 Hz), 28.6 (d, J_{C-P} = 4.7 Hz).

¹⁹**F NMR** (376 MHz, CDCl₃) δ /ppm = 114.5.

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 24.3.

HRMS: m/z $[M + H]^+$ calcd for $C_9H_{13}FO_3PSe$, 298.9746; found, 298.9752.

Se-(4-Chlorobenzyl) O,O-dimethyl phosphoroselenoate (**5c**). Compound **5c** was prepared according to the general procedure and isolated as an oil (67 mg, 85% yield) after flash chromatography (petroleum ether/ethyl acetate = 4/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.23 (d, J = 8.8 Hz, 2H), 7.20 (d, J = 8.8 Hz, 2H), 3.97 (d, J = 13.4 Hz, 2H), 3.62 (d, J = 13.3 Hz, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 137.0 (d, $J_{C-P} = 4.2$ Hz), 133.3, 130.3, 128.8, 53.6 (d, $J_{C-P} = 5.5$ Hz), 28.6 (d, $J_{C-P} = 4.6$ Hz).

³¹**P NMR** (162 MHz, CDCl₃) $\delta/ppm = 24.1$.

HRMS: $m/z [M + H]^+$ calcd for $C_9H_{13}ClO_3PSe$, 314.9451; found, 314.9441.

O,O-Dimethyl *Se*-(4-(trifluoromethyl)benzyl) phosphoroselenoate (5d). Compound 5d was prepared according to the general procedure and isolated as an oil (68 mg, 78% yield) after flash chromatography (petroleum ether/ethyl acetate = 4/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.49 (d, J = 8.2 Hz, 2H), 7.41 (d, J = 8.1 Hz, 2H), 4.02 (d, J = 13.8 Hz, 2H), 3.61 (d, J = 13.3 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 144.14 – 141.47 (m), 129.4 (q, J_{C-P} = 44.0 Hz), 129.3, 125.6 (q, J = 3.8 Hz), 121.3 (q, J_{C-F} = 274.0 Hz), 53.6 (d, J = 5.6 Hz), 28.4 (d, J = 4.7 Hz).

¹⁹**F NMR** (376 MHz, CDCl₃) δ /ppm = -62.6.

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 23.7.

HRMS: $m/z [M + H]^+$ calcd for $C_{10}H_{13}F_3O_3PSe$, 348.9714; found, 348.9750.

Se-(4-Cyanobenzyl) O,O-dimethyl phosphoroselenoate (5e). Compound 5e was prepared according to the general procedure and isolated as a solid (50 mg, 66% yield)

after flash chromatography (petroleum ether/ethyl acetate = 3/1). Mp= 62 - 63 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.54 (d, J = 8.0 Hz, 2H), 7.42 (d, J = 8.0 Hz, 2H), 4.01 (d, J = 14.2 Hz, 2H), 3.61 (d, J = 13.3 Hz, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 144.3 (d, J_{C-P} = 3.7 Hz), 132.4, 129.7, 118.6, 111.1, 53.7 (d, J_{C-P} = 5.6 Hz), 28.5 (d, J_{C-P} = 4.7 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 23.3.

HRMS: $m/z [M + H]^+$ calcd for $C_{10}H_{13}NO_3PSe$, 305.9793; found, 305.9779.

Se-(3,5-Dimethoxybenzyl) O,O-dimethyl phosphoroselenoate (**5f**). Compound **5f** was prepared according to the general procedure and isolated as an oil (53 mg, 63% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 6.43 (d, J = 2.3 Hz, 2H), 6.27 (t, J = 2.3 Hz, 1H), 3.94 (d, J = 12.6 Hz, 2H), 3.71 (s, 6H), 3.65 (d, J = 13.3 Hz, 6H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 160.9, 140.4 (d, J_{C-P} = 4.9 Hz), 106.8, 99.6, 55.4, 53.6 (d, J_{C-P} = 5.3 Hz), 29.6 (d, J_{C-P} = 4.6 Hz).

³¹**P NMR** (162 MHz, CDCl₃) $\delta/ppm = 24.7$.

HRMS: $m/z [M + H]^+$ calcd for $C_{11}H_{18}O_5PSe$, 341.0052; found, 341.0039.

O,O-Dimethyl *Se*-(1-phenylethyl) phosphoroselenoate (5g). Compound 5g was prepared according to the general procedure and isolated as an oil (45 mg, 61% yield) after flash chromatography (petroleum ether/ethyl acetate = 4/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 7.35 - 7.30 (m, 2H), 7.25 (t, J = 7.6 Hz, 2H), 7.22 - 7.14 (m, 1H), 4.83 - 4.45 (m, 1H), 3.57 (dd, J = 13.3, 3.2 Hz, 6H), 1.84 (d, J = 7.2 Hz, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 143.6 (d, J_{C-P} = 5.4 Hz), 128.6, 127.6, 127.1, 53.5 (dd, J_{C-P} = 5.4, 3.4 Hz), 42.9 (d, J_{C-P} = 4.3 Hz), 24.3 (d, J_{C-P} = 5.9 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 24.6.

HRMS: $m/z [M + H]^+$ calcd for $C_{10}H_{16}O_3PSe$, 294.9997; found, 294.9985.

Ethyl 2-((Dimethoxyphosphoryl)selanyl)-2-phenylacetate (5h). Compound 5h was prepared according to the general procedure and isolated as an oil (57 mg, 65% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 7.47 – 7.39 (m, 2H), 7.31 – 7.19 (m, 3H), 4.96

(d, J = 10.0 Hz, 1H), 4.28 - 4.00 (m, 2H), 3.56 (dd, J = 13.4, 5.2 Hz, 6H), 1.19 (t, J = 7.2 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 170.2 (d, $J_{C-P} = 6.2$ Hz), 136.6 (d, $J_{C-P} = 3.9$ Hz), 128.8, 128.6, 128.5, 62.4, 53.7 (t, $J_{C-P} = 4.7$ Hz), 46.9 (d, $J_{C-P} = 3.8$ Hz), 14.0. ³¹P NMR (162 MHz, CDCl₃) δ/ppm = 23.1.

HRMS: m/z $[M + H]^+$ calcd for $C_{12}H_{18}O_5PSe$, 353.0052; found, 353.0044.

Se-Heptyl O,O-dimethyl phosphoroselenoate (5i). Compound 5i was prepared according to the general procedure and isolated as an oil (42 mg, 58% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 3.73 (s, 3H), 3.70 (s, 3H), 2.80 (dt, J = 14.1, 7.4 Hz, 2H), 1.75 – 1.58 (m, 2H), 1.40 – 1.12 (m, 8H), 0.81 (t, J = 6.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 52.5 (d, J_{C-P} = 5.6 Hz), 30.6, 30.2 (d, J_{C-P} = 4.5 Hz), 28.5, 27.6, 25.6 (d, J_{C-P} = 4.6 Hz), 21.5, 13.0.

³¹**P NMR** (162 MHz, CDCl₃) δ/ppm = 25.4.

HRMS: $m/z [M + H]^+$ calcd for $C_9H_{22}O_3PSe$, 289.0466; found, 289.0479.

Se-Allyl O,O-dimethyl phosphoroselenoate (**5j**). Compound **5j** was prepared according to the general procedure and isolated as an oil (31 mg, 55% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 5.91 (ddt, J = 17.1, 9.3, 7.6 Hz, 1H), 5.18 (d, J = 16.9 Hz, 1H), 5.02 (d, J = 9.9 Hz, 1H), 3.74 (d, J = 1.2 Hz, 6H), 3.43 (dd, J = 13.7, 7.6 Hz, 2H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 134.3 (d, J_{C-P} = 4.3 Hz), 118.1, 53.6 (d, J_{C-P} = 5.5 Hz), 28.2 (d, J_{C-P} = 4.5 Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 24.6.

HRMS: $m/z [M + H]^+$ calcd for $C_5H_{12}O_3PSe$, 230.9684; found, 230.9655.

O,O-Dimethyl Se-(pent-4-en-1-yl) phosphoroselenoate (5k). Compound 5k was prepared according to the general procedure and isolated as an oil (39 mg, 61% yield) after flash chromatography (petroleum ether/ethyl acetate = 1/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 5.70 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H), 5.05 – 4.64 (m, 2H), 3.72 (d, J = 13.2 Hz, 6H), 2.93 – 2.71 (m, 2H), 2.18 – 2.01 (m, 2H), 1.86 – 1.76 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 137.0, 115.8, 53.6 (d, $J_{C-P} = 5.6$ Hz), 33.5, 30.3 (d, $J_{C-P} = 4.3$ Hz), 25.8 (d, $J_{C-P} = 4.7$ Hz).

³¹**P NMR** (162 MHz, CDCl₃) δ /ppm = 25.1.

HRMS: $m/z [M + H]^+$ calcd for $C_7H_{16}O_3PSe$, 258.9997; found, 258.9991.

5. General procedure for the synthesis of 3-selenylindoles

The alkyl selenocyanates were prepared using the aforementioned method. Indole (0.25 mmol), alkyl selenocyanates (0.25 mmol), $B(C_6F_5)_3$ (0.0025 mmol) were dissolved in CH_2Cl_2 under N_2 atmosphere. This reaction mixture was allowed to stir for 5 h at room temperature. Upon completion, the solvent was removed in vacuum and the residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the corresponding product $\bf 6$.

Ethyl 2-((1*H*-indol-3-yl)selanyl)-2-phenylacetate (6a). Compound 6a was prepared according to the general procedure and isolated as an oil (55 mg, 61% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.45 (s, 1H), 7.53 (d, J = 7.6 Hz, 1H), 7.32 – 7.27 (m, 2H), 7.24 (d, J = 7.8 Hz, 1H), 7.16 – 7.05 (m, 5H), 7.01 (d, J = 2.6 Hz, 1H), 4.66 (s, 1H), 3.92 (p, J = 7.1 Hz, 2H), 0.97 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 171.8, 136.8, 136.1, 132.0, 130.3, 128.7, 128.5, 128.4, 127.7, 122.6, 120.7, 120.1, 111.4, 98.9, 61.5, 47.7, 13.9.

HRMS: $m/z [M + H]^+$ calcd for $C_{18}H_{18}NO_2Se$, 360.0497; found, 360.0490.

3-((1*H***-Indol-3-yl)selanyl)dihydrofuran-2(3***H***)-one (6b). Compound 6b was prepared according to the general procedure and isolated as an oil (41 mg, 58% yield) after flash chromatography (petroleum ether/ethyl acetate = 3/1).**

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.62 (s, 1H), 7.76 – 7.63 (m, 1H), 7.45 – 7.34 (m, 2H), 7.21 – 7.13 (m, 2H), 4.08 (td, J = 8.7, 3.8 Hz, 1H), 3.89 (td, J = 8.6, 7.2 Hz, 1H), 3.67 (dd, J = 8.7, 4.0 Hz, 1H), 2.60 – 2.52 (m, 1H), 2.24 – 2.12 (m, 1H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 177.0, 136.2, 132.5, 130.4, 123.0, 121.1, 119.7, 111.6, 95.9, 67.0, 35.7, 30.5, 29.7.

HRMS: $m/z [M + H]^+$ calcd for $C_{12}H_{12}NO_2Se$, 282.0028; found, 282.0033.

2-((1*H***-Indol-3-yl)selanyl)-***N***,***N***-dimethylacetamide (6c). Compound 6c was prepared according to the general procedure and isolated as an oil (40 mg, 57% yield) after flash chromatography (petroleum ether/ethyl acetate = 1/1).**

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 9.12 (s, 1H), 7.70 – 7.53 (m, 1H), 7.30 – 7.22 (m, 1H), 7.21 – 7.18 (m, 1H), 7.15 – 7.06 (m, 2H), 3.42 (s, 2H), 2.79 (s, 3H), 2.78 (s, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 170.5, 136.3, 131.6, 130.1, 122.5, 120.4, 119.7, 111.7, 97.0, 38.2, 35.8, 28.2.

HRMS: m/z $[M + H]^+$ calcd for $C_{12}H_{15}N_2OSe$, 283.0344; found, 283.0338.

3-((1*H***-Indol-3-yl)selanyl)butan-2-one (6d)**. Compound **6d** was prepared according to the general procedure and isolated as a solid (42 mg, 63% yield) after flash chromatography (petroleum ether/ethyl acetate = 7/1). Mp= 89 - 90 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.71 (s, 1H), 7.61 – 7.57 (m, 1H), 7.24 – 7.15 (m, 1H), 7.13 – 7.05 (m, 3H), 3.59 (q, J = 6.9 Hz, 1H), 2.28 (s, 3H), 1.35 (d, J = 6.9 Hz, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 205.4, 136.2, 131.9, 130.6, 122.7, 120.7, 119.8, 111.6, 96.0, 45.8, 27.6, 16.4.

HRMS: $m/z [M + H]^+$ calcd for $C_{12}H_{14}NOSe$, 268.0235; found, 268.0219.

3-((Trichloromethyl)selanyl)-1*H***-indole** (**6e**). Compound **6e** was prepared according to the general procedure and isolated as a solid (43 mg, 55% yield) after flash chromatography (petroleum ether/ethyl acetate = 8/1). Mp= 102 - 103 °C.

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 8.77 (s, 1H), 7.74 – 7.55 (m, 1H), 7.35 – 7.22 (m, 2H), 7.21 – 7.11 (m, 2H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 136.1, 132.1, 128.7, 123.7, 121.8, 119.4, 112.1, 102.4, 89.0.

HRMS: $m/z [M + H]^+$ calcd for $C_9H_7Cl_3NSe$, 313.8804; found, 313.8816.

2-((1*H***-Indol-3-yl)selanyl)acetonitrile (6f)**. Compound **6f** was prepared according to the general procedure and isolated as an oil (31 mg, 53% yield) after flash chromatography (petroleum ether/ethyl acetate = 5/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.55 (s, 1H), 7.67 (dd, J = 8.2, 1.0 Hz, 1H), 7.49 (d, J = 2.6 Hz, 1H), 7.44 – 7.31 (m, 1H), 7.27 – 7.08 (m, 2H), 3.11 (s, 2H). ¹³**C NMR** (100 MHz, CDCl₃) δ/ppm = 136.3, 132.1, 129.5, 123.2, 121.1, 119.6, 118.2,

111.7, 96.6, 8.2.

HRMS: $m/z [M + H]^+$ calcd for $C_{10}H_9N_2Se$, 236.9925; found, 236.9916.

2-(3-((1*H***-Indol-3-yl)selanyl)propyl)isoindoline-1,3-dione (6g)**. Compound **6g** was prepared according to the general procedure and isolated as a solid (58 mg, 61% yield) after flash chromatography (petroleum ether/ethyl acetate = 8/1). Mp= 95 - 96 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.42 (s, 1H), 7.73 – 7.69 (m, 2H), 7.64 – 7.57 (m, 3H), 7.39 – 7.24 (m, 2H), 7.18 – 7.03 (m, 2H), 3.70 (t, J = 7.1 Hz, 2H), 2.58 (t, J = 7.2 Hz, 2H), 1.85 (p, J = 7.2 Hz, 2H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 168.5, 136.3, 134.0, 132.0, 131.0, 130.2, 123.2, 122.6, 120.5, 120.1, 111.3, 97.7, 37.8, 29.0, 24.9.

HRMS: m/z $[M + H]^+$ calcd for $C_{19}H_{17}N_2O_2Se$, 385.0450; found, 385.0444.

$$\text{Ar-NH}_2 = \underbrace{ \begin{array}{c} 1.50\% \text{ HBF}_4 \text{ (1.0 equiv)} \\ \text{ $IBUONO \text{ (1.0 equiv)}} \\ \text{EtOH, 0 °C to r.t., 1 h} \\ 2. \text{ KSeCN (1.5 equiv.)} \\ \text{CH}_3\text{CN, 30 min, 0 °C} \quad \textit{in situ-generation} \end{array} } \underbrace{ \begin{array}{c} 1\text{H-indole} \\ 1 \text{ mol}\% \text{ B(C}_6\text{F}_5)_3 \\ \text{CH}_2\text{Cl}_2, \text{r.t., 5 h} \\ \text{N} \\ \text{N} \\ \text{T} \\ \text{N} \\ \text{N} \\ \text{T} \\ \text{N} \\ \text{T} \\ \text{T$$

The aryl selenocyanates were prepared using the aforementioned method. Indole (0.25 mmol), aryl selenocyanates (0.25 mmol), $B(C_6F_5)_3$ (0.0025 mmol) were dissolved in CH_2Cl_2 under N_2 atmosphere. This reaction mixture was allowed to stir for 5 h at room temperature. Upon completion, the solvent was removed in vacuum and the residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the corresponding product 7.

3-(p-Tolylselanyl)-1H-indole (7a). Compound 7a was prepared according to the general procedure and isolated as a solid (52 mg, 73% yield) after flash chromatography (petroleum ether/ethyl acetate = 15/1). Mp= 103 - 104 °C.

¹**H NMR** (400 MHz, CDCl₃) $\delta/ppm = 8.27$ (s, 1H), 7.63 (d, J = 7.9 Hz, 1H), 7.43 –

7.33 (m, 2H), 7.23 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.20 – 7.06 (m, 3H), 6.93 (d, J = 7.9 Hz, 2H), 2.21 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 136.4, 135.6, 131.1, 130.0, 129.9, 129.1, 129.1, 122.9, 120.8, 120.4, 111.4, 98.5, 21.0.

Spectral data are in good agreement with literature values.¹

3-((4-Methoxyphenyl)selanyl)-1*H***-indole** (**7b**). Compound **7b** was prepared according to the general procedure and isolated as a solid (57 mg, 76% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1). Mp= 84 - 85 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.19 (s, 1H), 7.56 (dd, J = 7.9, 1.2 Hz, 1H), 7.28 – 7.20 (m, 2H), 7.18 – 7.10 (m, 3H), 7.10 – 7.06 (m, 1H), 6.59 (d, J = 8.8 Hz, 2H), 3.59 (s, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 158.3, 136.4, 131.3, 130.8, 129.9, 123.5, 122.9, 120.8, 120.3, 114.8, 111.5, 99.3, 55.3.

Spectral data are in good agreement with literature values.¹

3-((4-Fluorophenyl)selanyl)-1*H***-indole (7c)**. Compound **7c** was prepared according to the general procedure and isolated as a solid (58 mg, 80% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1). Mp= 125 - 126 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.30 (s, 1H), 7.53 (d, J = 7.9 Hz, 1H), 7.42 – 7.27 (m, 2H), 7.23 – 7.06 (m, 4H), 6.74 (t, J = 8.7 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ /ppm = δ 161.6 (d, J_{C-F} = 244.6 Hz), 136.4, 131.1, 130.75 (d, J_{C-F} = 7.6 Hz), 130.0, 128.0 (d, J_{C-F} = 3.2 Hz), 123.1, 121.0, 120.2, 116.1 (d, J_{C-F} = 21.6 Hz), 111.5, 98.6.

¹⁹**F NMR** (376 MHz, CDCl₃) δ /ppm = -117.2.

Spectral data are in good agreement with literature values.⁷

3-((4-Chlorophenyl)selanyl)-1*H***-indole (7d)**. Compound **7d** was prepared according to the general procedure and isolated as a solid (64 mg, 83% yield) after flash chromatography (petroleum ether/ethyl acetate = 10/1). Mp= 148 - 149 °C.

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 8.34 (s, 1H), 7.51 (d, J = 8.0 Hz, 1H), 7.39 –

7.29 (m, 2H), 7.22 – 7.13 (m, 1H), 7.12 – 7.01 (m, 3H), 6.99 (d, J = 8.6 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ /ppm = 136.4, 132.1, 131.6, 131.3, 130.0, 129.7, 129.1, 123.1, 121.1, 120.2, 111.5, 97.8.

Spectral data are in good agreement with literature values.⁷

3-((4-(Methylthio)phenyl)selanyl)-1*H***-indole** (**7e**). Compound **7e** was prepared according to the general procedure and isolated as a solid (57 mg, 71% yield) after flash chromatography (petroleum ether/ethyl acetate = 8/1). Mp= 108 - 109 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.37 (s, 1H), 7.54 (d, J = 7.9 Hz, 1H), 7.43 – 7.25 (m, 2H), 7.24 – 7.12 (m, 1H), 7.13 – 7.04 (m, 3H), 6.95 (d, J = 8.3 Hz, 2H), 2.31 (s, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 136.4, 135.7, 131.1, 130.3, 129.8, 129.5, 127.6, 123.0, 120.9, 120.3, 111.4, 98.2, 16.2.

HRMS: $m/z [M + H]^+$ calcd for $C_{15}H_{14}NSSe$, 320.0007; found, 320.0024.

3-((2,4-Dichlorophenyl)selanyl)-1*H***-indole** (**7f**). Compound **7f** was prepared according to the general procedure and isolated as an oil (64 mg, 75% yield) after flash chromatography (petroleum ether/ethyl acetate = 8/1).

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = 8.57 (s, 1H), 7.56 (d, J = 8.0 Hz, 1H), 7.52 – 7.42 (m, 2H), 7.36 – 7.25 (m, 2H), 7.19 (t, J = 7.6 Hz, 1H), 6.83 (d, J = 8.5 Hz, 1H), 6.60 (d, J = 8.0 Hz, 1H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 136.6, 133.1, 132.4, 132.1, 131.5, 129.7, 129.6, 128.9, 127.4, 123.3, 121.3, 120.2, 111.6, 96.5.

HRMS: $m/z [M + H]^+$ calcd for $C_{14}H_{10}Cl_2NSe$, 341.9350; found, 341.9353.

3-((2-Bromo-4,6-dimethylphenyl)selanyl)-1*H***-indole** (**7g**). Compound **7g** was prepared according to the general procedure and isolated as a solid (73 mg, 77% yield) after flash chromatography (petroleum ether/ethyl acetate = 12/1). Mp= 135 – 136 °C. 1 **H NMR** (400 MHz, CDCl₃) δ /ppm = 8.11 (s, 1H), 7.60 (d, J = 7.7 Hz, 1H), 7.31 – 7.14 (m, 3H), 7.11 – 7.01 (m, 2H), 6.78 (s, 1H), 2.45 (s, 3H), 2.09 (s, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ /ppm = 144.7, 139.8, 136.0, 131.4, 130.3, 130.0, 129.8, 129.8, 122.6, 120.5, 120.5, 111.3, 100.4, 25.5, 20.7.

HRMS: $m/z [M + H]^+$ calcd for $C_{16}H_{15}BrNSe$, 379.9548; found, 379.9540.

3-(Mesitylselanyl)-1*H***-indole** (**7h**). Compound **7h** was prepared according to the general procedure and isolated as a solid (61 mg, 78% yield) after flash chromatography (petroleum ether/ethyl acetate = 8/1). Mp= 132 - 133 °C.

¹**H NMR** (400 MHz, CDCl₃) δ /ppm = 8.05 (s, 1H), 7.45 (d, J = 7.8 Hz, 1H), 7.22 (d, J = 8.2 Hz, 1H), 7.12 – 6.98 (m, 3H), 6.79 (s, 2H), 2.48 (s, 6H), 2.14 (s, 3H).

¹³C **NMR** (100 MHz, CDCl₃) δ/ppm = 142.5, 137.9, 136.1, 129.6, 128.7, 128.7, 128.1, 122.4, 120.2, 120.2, 111.2, 101.0, 24.5, 20.9.

Spectral data are in good agreement with literature values.⁷

3-((3,4,5-Trimethoxyphenyl)selanyl)-1*H***-indole** (**7i**). Compound **7i** was prepared according to the general procedure and isolated as a solid (60 mg, 66% yield) after flash chromatography (petroleum ether/ethyl acetate = 8/1). Mp= 129 - 130 °C.

¹**H NMR** (400 MHz, CDCl₃) δ/ppm = δ 8.71 (s, 1H), 7.66 (d, J = 7.8 Hz, 1H), 7.47 (d, J = 2.5 Hz, 1H), 7.41 (d, J = 7.7 Hz, 1H), 7.29 – 7.21 (m, 1H), 7.17 (td, J = 7.5, 6.9, 1.2 Hz, 1H), 6.51 (d, J = 1.1 Hz, 2H), 3.77 (d, J = 1.2 Hz, 3H), 3.64 (d, J = 1.2 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃) δ/ppm = 153.4 136.4, 136.2, 131.2, 129.8, 128.2, 122.9, 122.4, 120.8, 120.2, 106.2, 98.4, 60.9, 56.0.

Spectral data are in good agreement with literature values.⁸

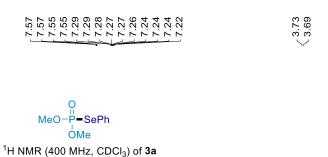
6. References

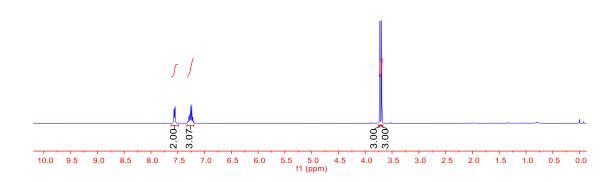
- 1. S. K. Bhunia, P. Das and R. Jana, *Org. Biomol. Chem.*, 2018, **16**, 9243.
- 2. Y.-Q. Zhang, C.-F. Zhou and G.-Q. Liu, Chin. J. Org. Chem., 2022, 42, 218.
- 3. S. Guo, S. Li, Z. Zhang, W. Yan and H. Cai, *Tetrahedron Lett.*, 2020, **61**, 151566
- 4. B. Yang, X.-Y. Zhang, H.-Q. Yue, W.-Z. Li, M. Li, L. Lu, Z.-Q. Wu, J. Li, K. Sun and S.-D. Yang, *Adv. Synth. Catal.*, 2022, **364**, 3528.
- 5. D. H. Mailahn, L. E. B. Iarocz, P. C. Nobre, G. Perin, A. Sinott, A. P. Pesarico, P. T. Birmann, L. Savegnago and M. S. Silva, *Eur. J. Med. Chem.*, 2021, **213**,

113052.

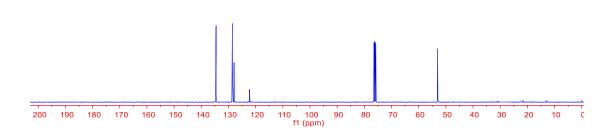
- 6. S. Mitra, S. Mukherjee, S. K. Sen and A. Hajra, *Bioorg. Med. Chem. Lett.*, 2014, **24**, 2198.
- 7. G. T. Quadros, S. P. de Medeiros, C. A. de Oliveira, M. W. Rambo, L. Abenante, E. J. Lenardao and F. Penteado, *Asian J. Org. Chem.*, 2023, **12**, e202300517.
- 8. Z. Wen, J. Xu, Z. Wang, H. Qi, Q. Xu, Z. Bai, Q. Zhang, K. Bao, Y. Wu and W. Zhang, *Eur. J. Med. Chem.*, 2015, **90**, 184.

7. Copies of NMR spectra



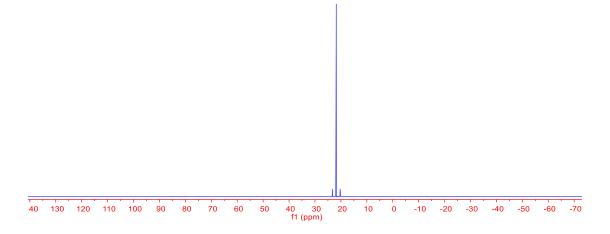




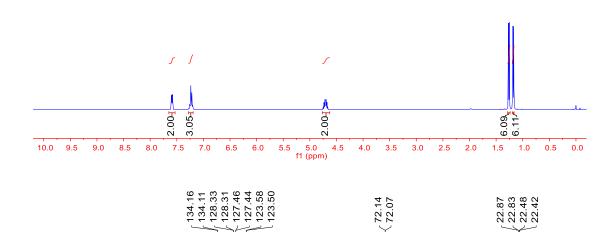


-21.80

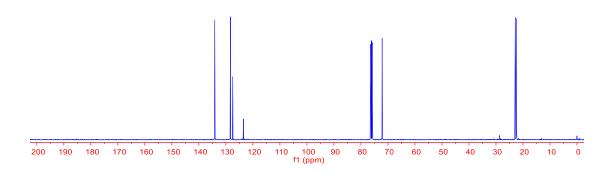
 31 P NMR (162 MHz, CDCl $_{3}$) of ${\bf 3a}$



 1 H NMR (400 MHz, CDCl₃) of **3b**



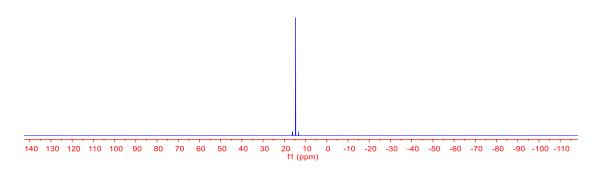
 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of 3b



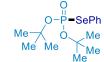




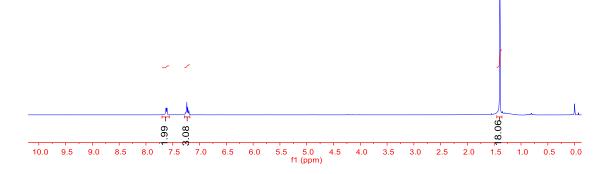
 $^{31}\text{P NMR}$ (162 MHz, CDCl $_3$) of $\mathbf{3b}$





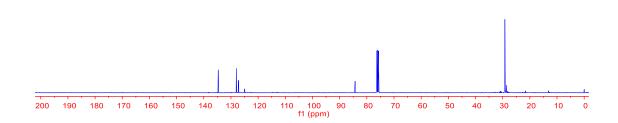


¹H NMR (400 MHz, CDCl₃) of **3c**



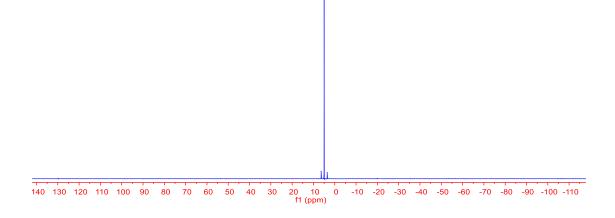


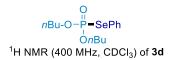
 13 C $\{^{1}$ H $\}$ NMR (100 MHz, CDCl $_{3}$) of **3c**

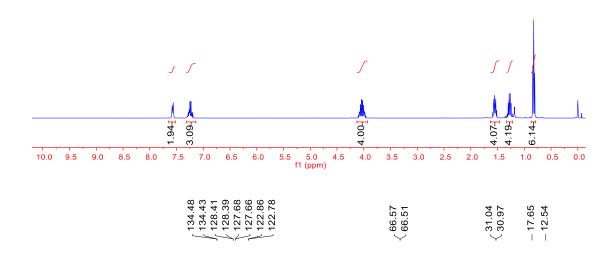


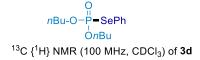
- 4.99

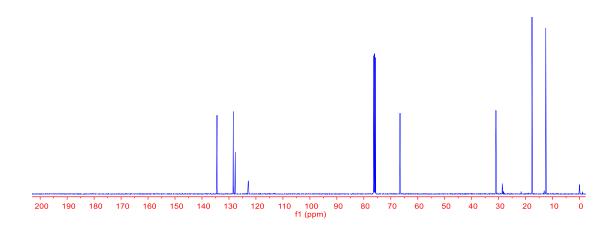
 $^{31}\text{P NMR}$ (162 MHz, CDCl₃) of **3c**

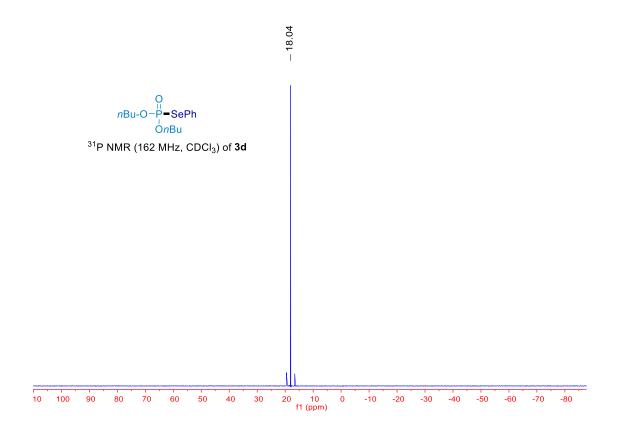




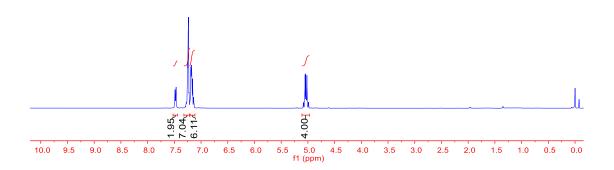






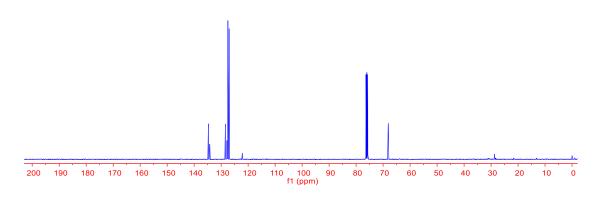


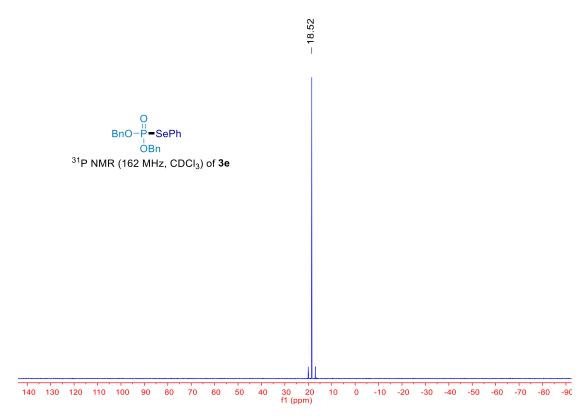






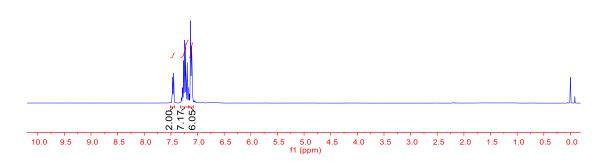
 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCI}_3)$ of 3e





PhO-P=SePh

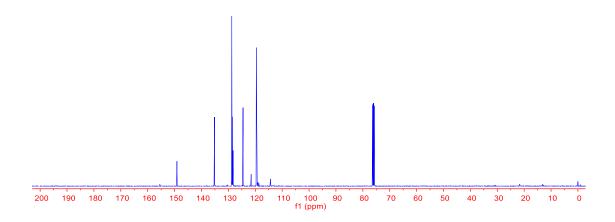
 1 H NMR (400 MHz, CDCl₃) of **3f**



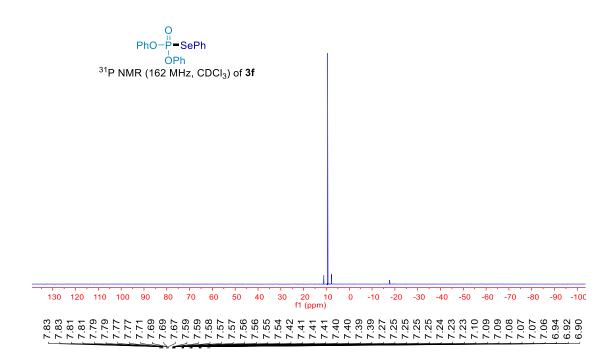
149.22 149.13 135.21 135.21 128.73 128.55 128.55 128.56 124.55 124.55 124.55 114.61 119.61

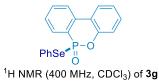
PhO-P-SePh OPh

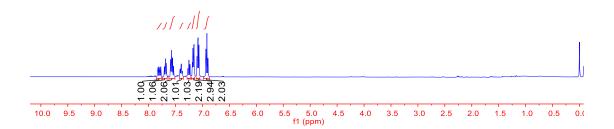
 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of 3f





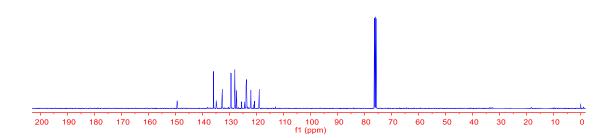






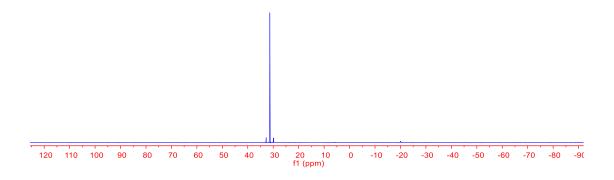


 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of $\boldsymbol{3g}$

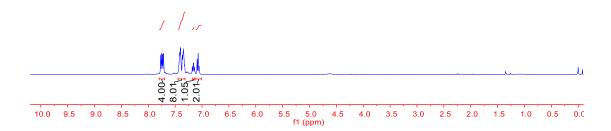


-31.34

 $^{31}\text{P NMR}$ (162 MHz, $\text{CDCI}_3)$ of $\boldsymbol{3g}$

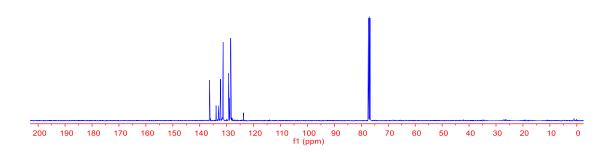


¹H NMR (400 MHz, CDCl₃) of **3h**

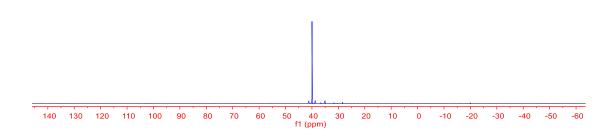




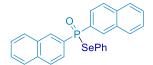
 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of $\boldsymbol{3h}$



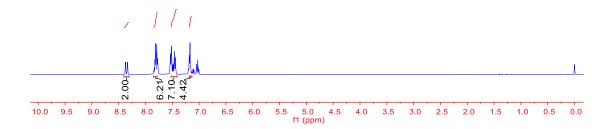
 31 P NMR (162 MHz, CDCl₃) of **3h**

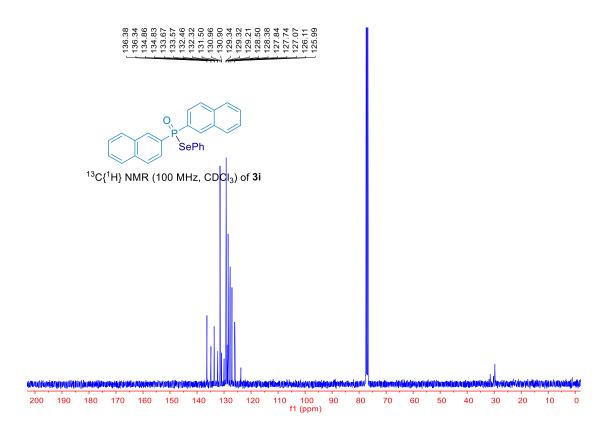


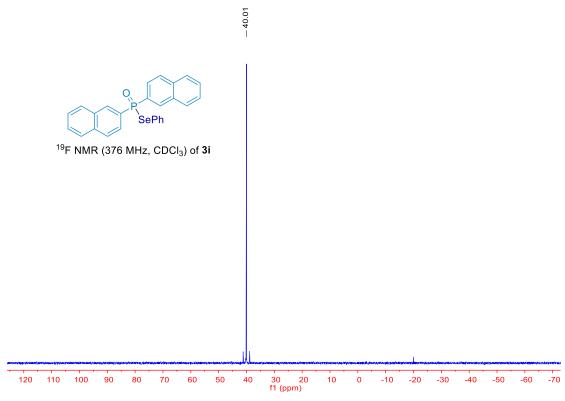
8.37 8.83 7.88 7.88 7.88 7.88 7.79 7.70

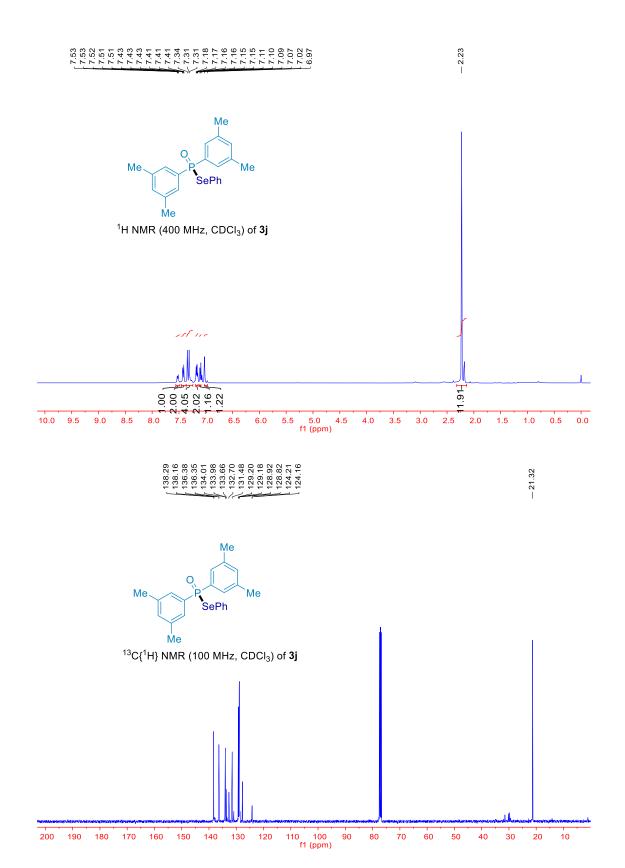


 1 H NMR (400 MHz, CDCl $_{3}$) of 3i

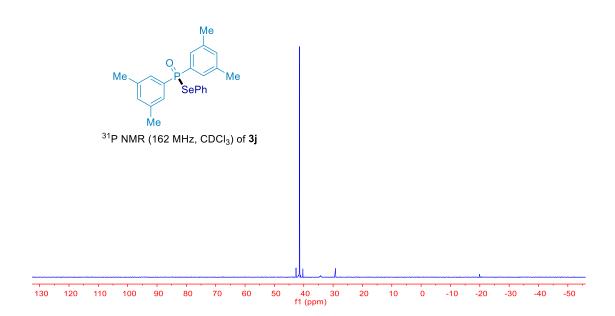






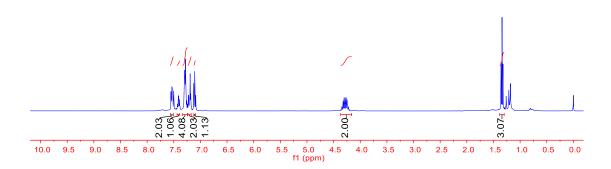


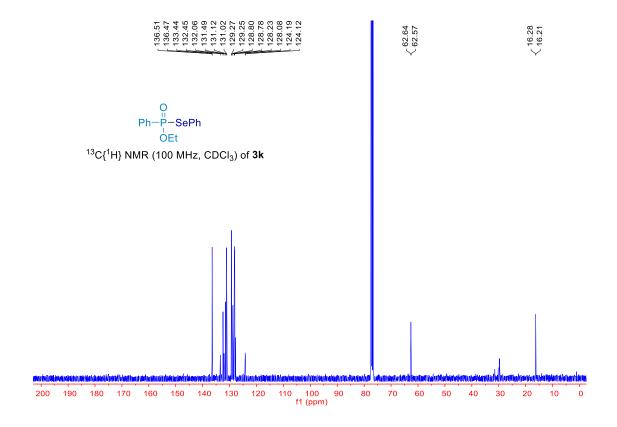


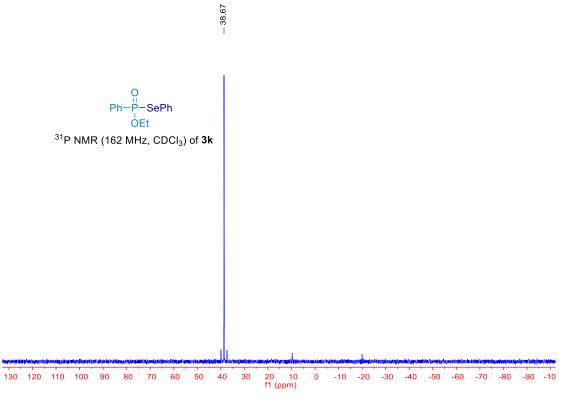


 6
 6
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7
 7

 1 H NMR (400 MHz, CDCl₃) of 3k

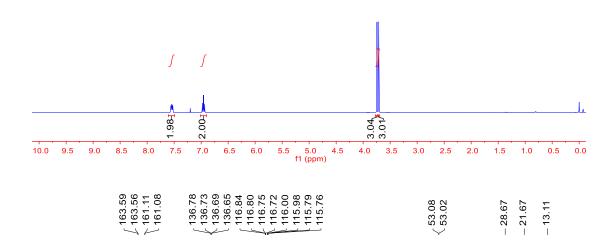




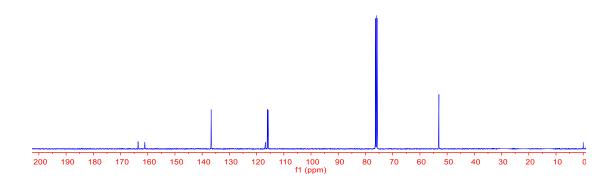


3.74

¹H NMR (400 MHz, CDCl₃) of **4a**

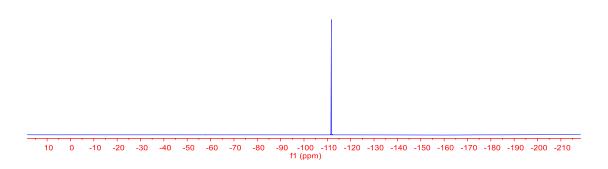


 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of 4a



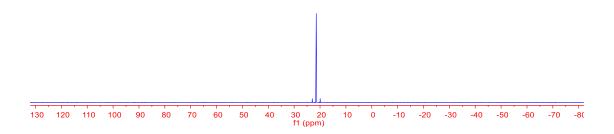


 19 F NMR (376 MHz, CDCl $_3$) of **4a**



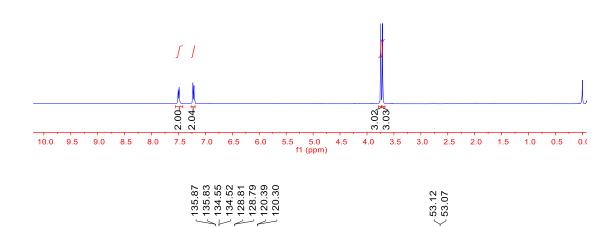
21.49

 31 P NMR (162 MHz, CDCl₃) of **4a**

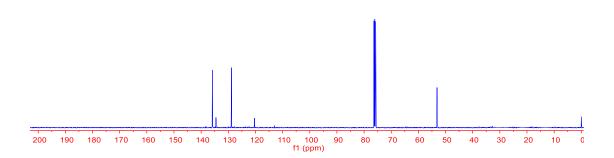




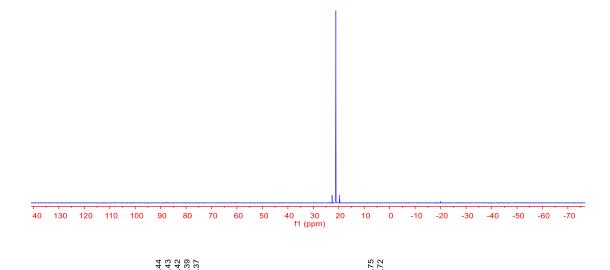
 1 H NMR (400 MHz, CDCl $_{3}$) of **4b**



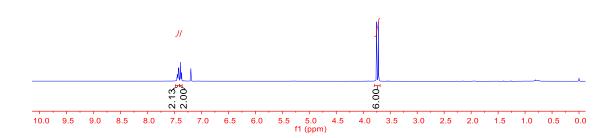
 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of 4b

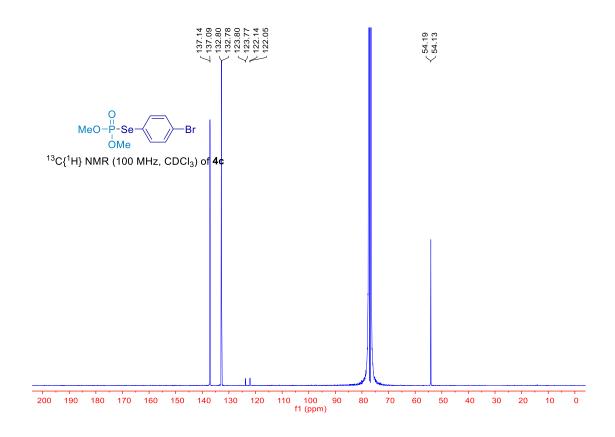


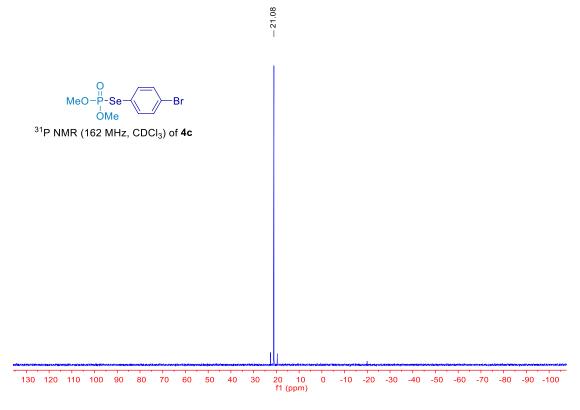




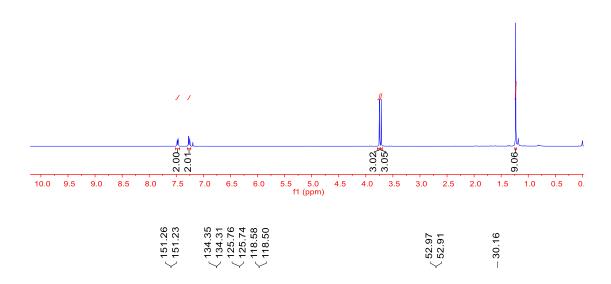
¹H NMR (400 MHz, CDCl₃) of **4c**



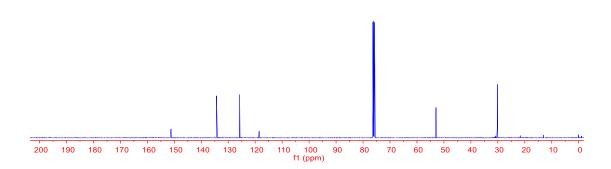




¹H NMR (400 MHz, CDCl₃) of **4d**

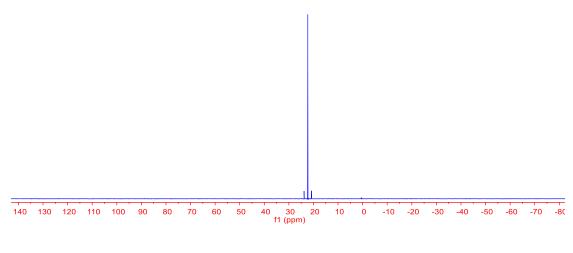


 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of 4d





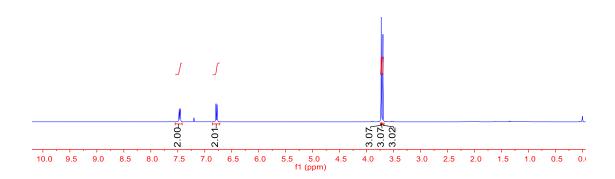
 $^{31}\text{P NMR}$ (162 MHz, CDCl $_3$) of **4d**

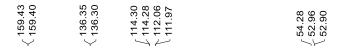


7.48 7.47 7.46 7.45 6.79

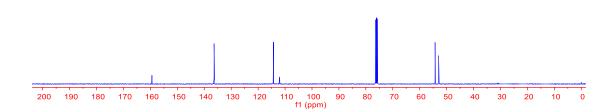
3.73

¹H NMR (400 MHz, CDCl₃) of **4e**



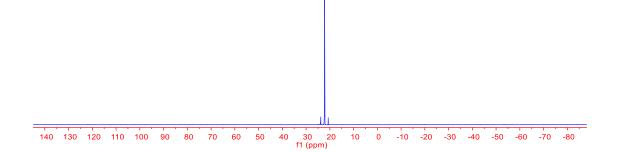


 13 C $\{^{1}$ H $\}$ NMR (100 MHz, CDCl $_{3}$) of **4e**



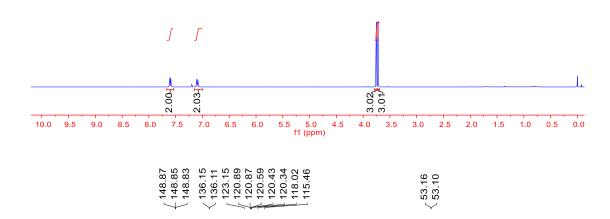
-22.23

³¹P NMR (162 MHz, CDCl₃) of **4e**

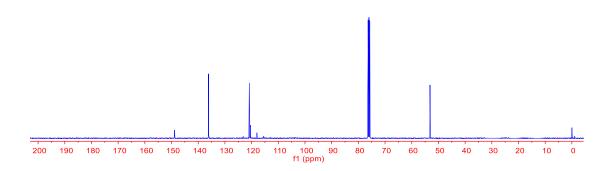




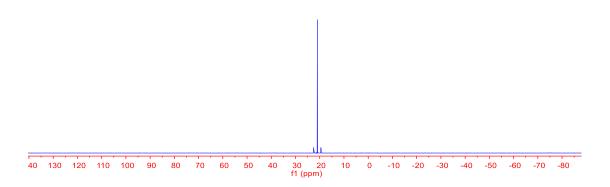
 1 H NMR (400 MHz, CDCl₃) of **4f**



 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCI}_3)$ of 4f

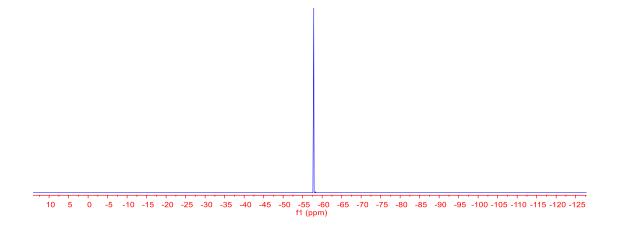


 $^{31}\text{P NMR}$ (162 MHz, CDCl₃) of **4f**



--57.85

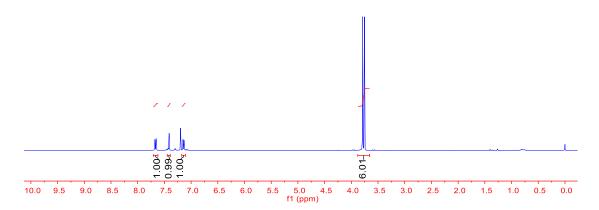
 19 F NMR (376 MHz, CDCl₃) of **4f**

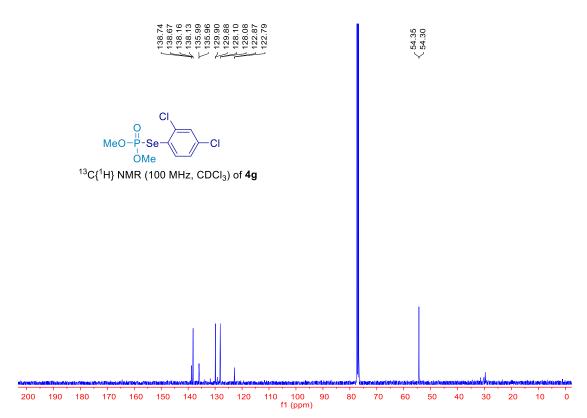


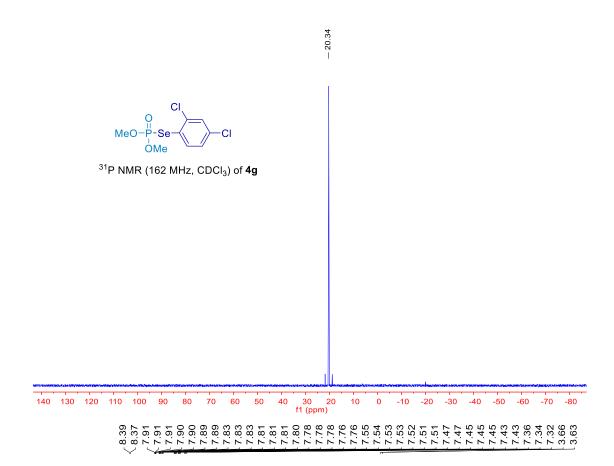


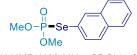
3.79

¹H NMR (400 MHz, CDCl₃) of **4g**

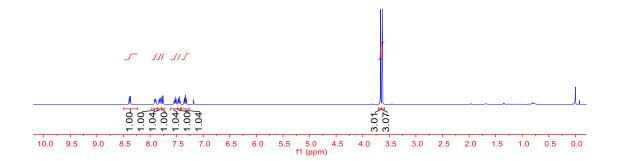


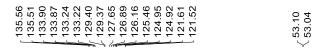




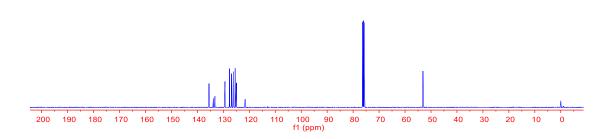


 1 H NMR (400 MHz, CDCl $_{3}$) of **4h**

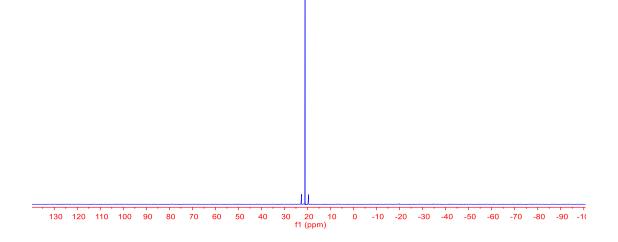


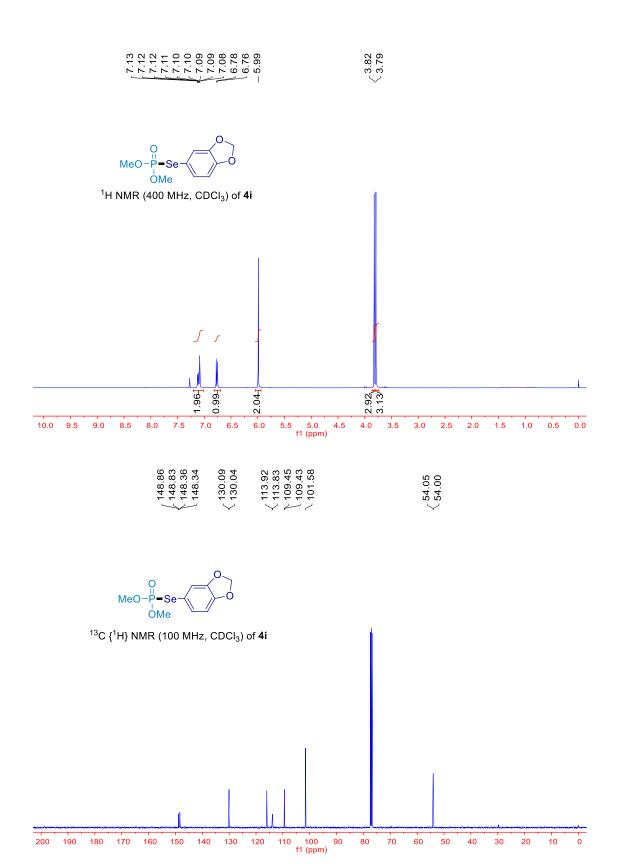


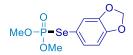
 $^{13}\text{C}\ \{^1\text{H}\}\ \text{NMR}\ (100\ \text{MHz},\ \text{CDCl}_3)$ of 4h



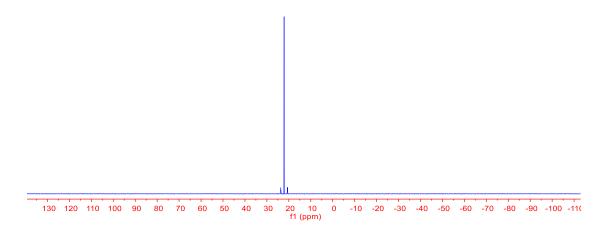
-21.05

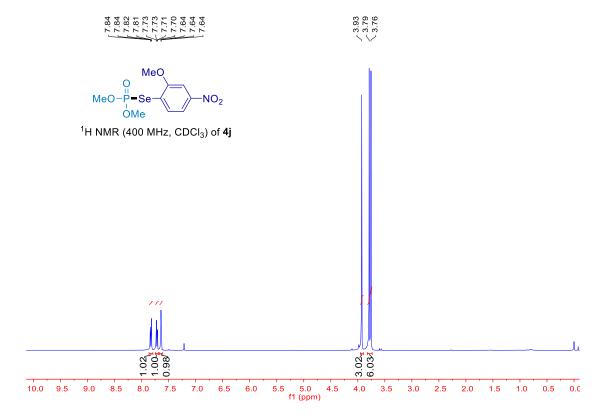


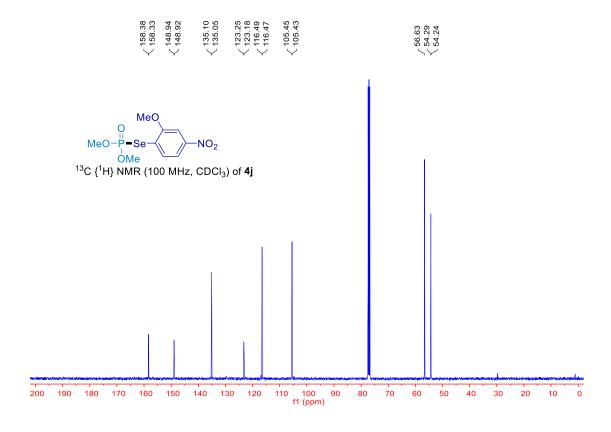


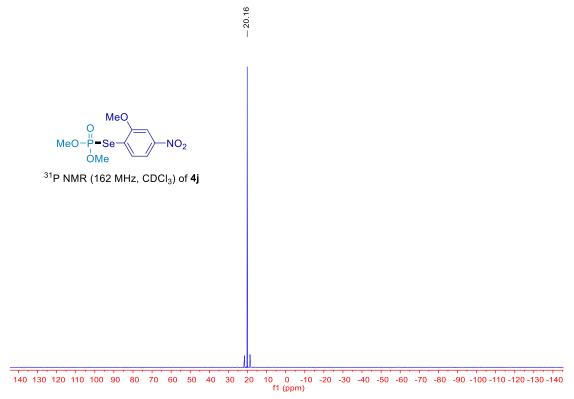


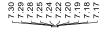
 31 P NMR (162 MHz, CDCl $_{3}$) of ${f 4i}$





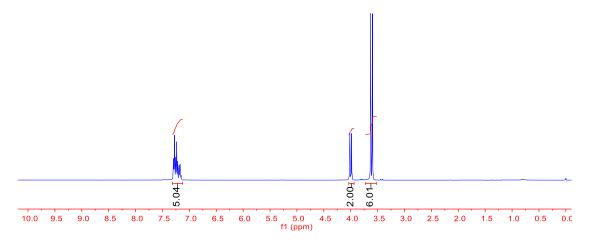






4.02 3.98 3.63 3.60

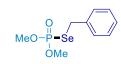
¹H NMR (400 MHz, CDCl₃) of **5a**



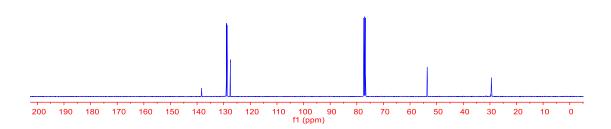
\[
 \text{138.28} \]
 \[
 \text{138.23} \]
 \[
 \text{128.97} \]
 \[
 \text{128.69} \]
 \[
 \text{128.69} \]
 \[
 \text{137.46}
 \]

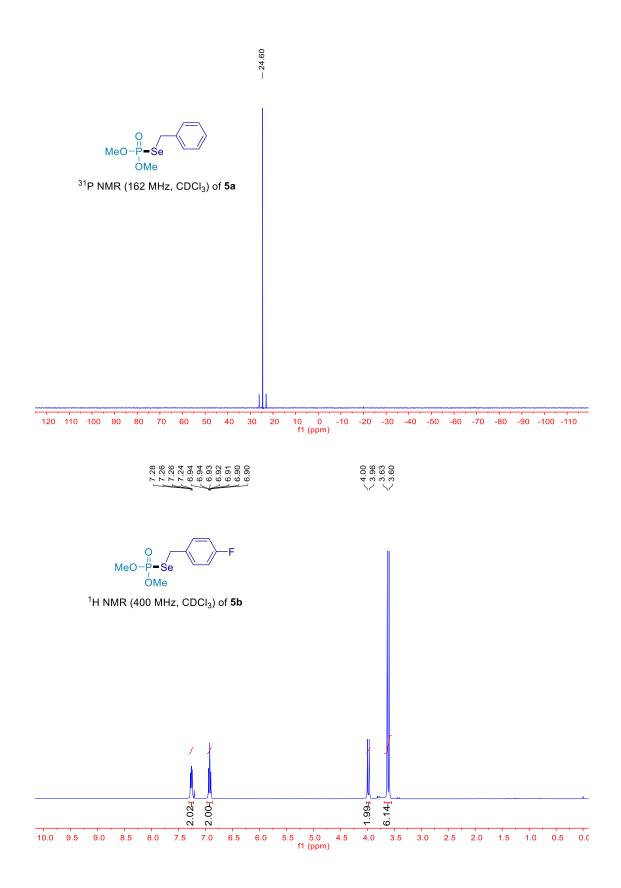
 $\langle 53.57 \\ 53.51$

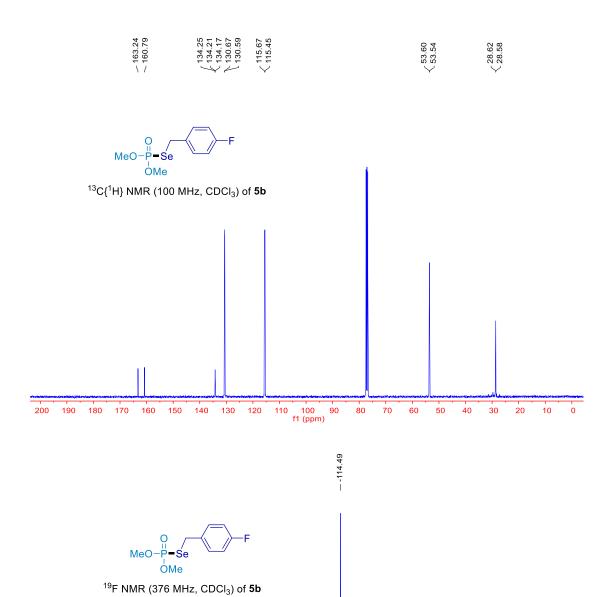
< 29.46 < 29.42

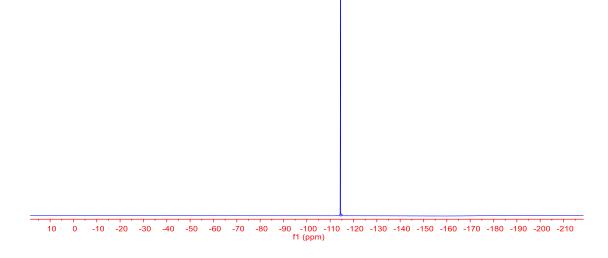


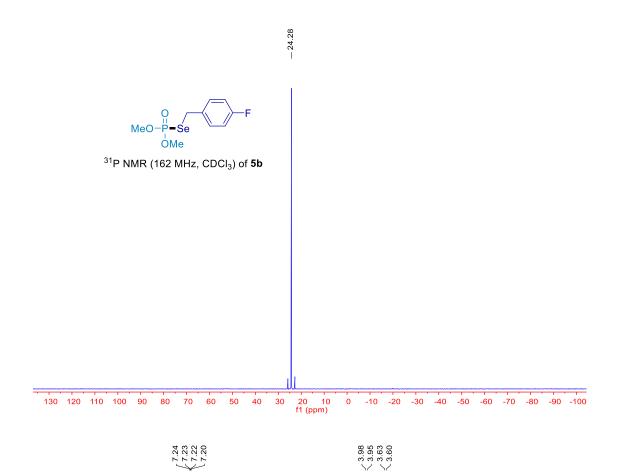
 $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl3) of 5a

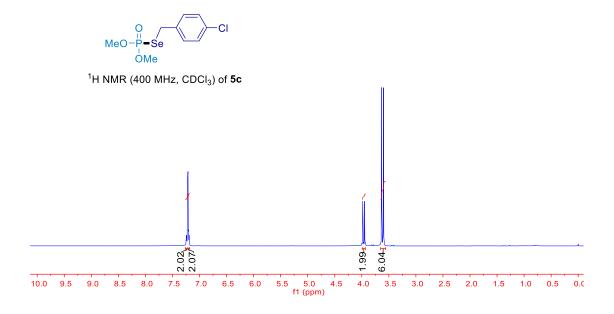


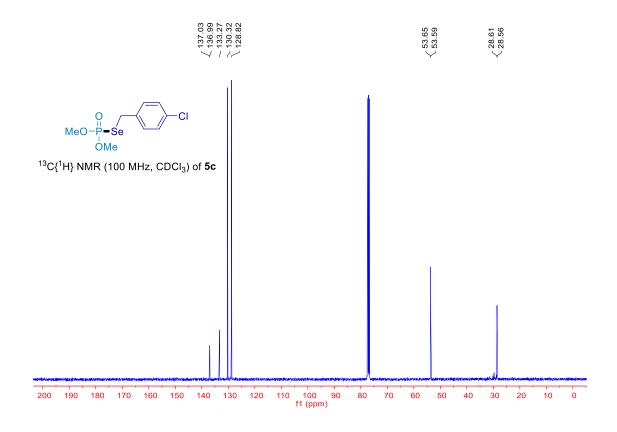


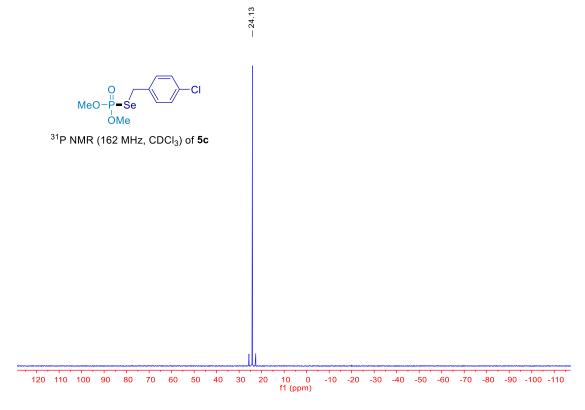


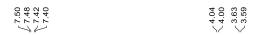




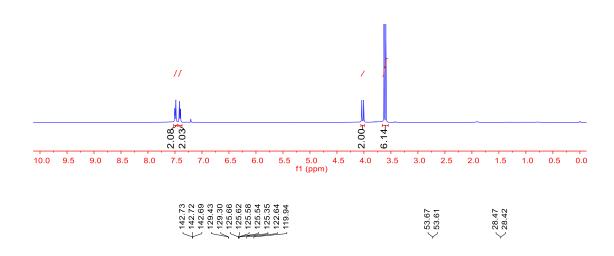


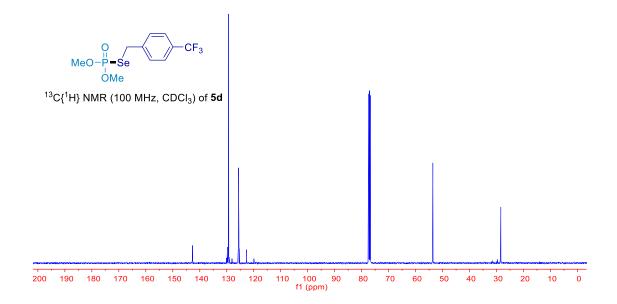


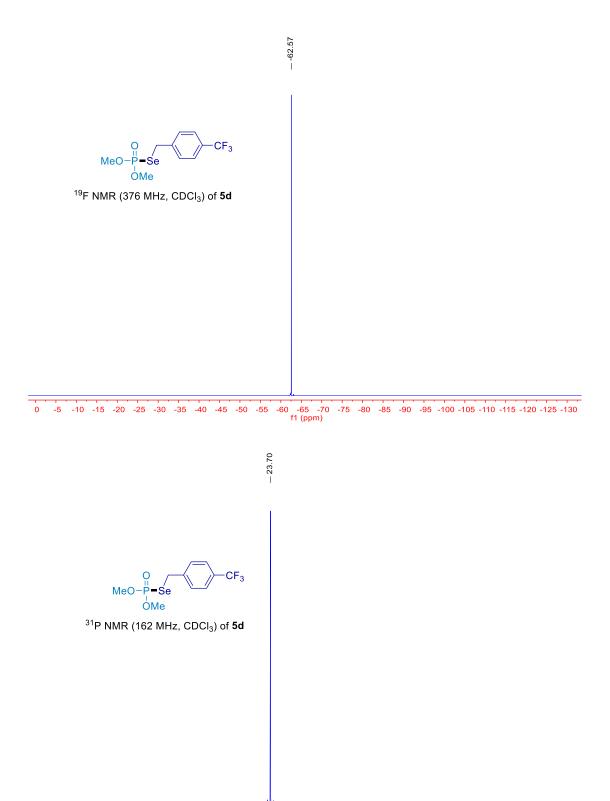




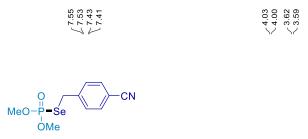
 1 H NMR (400 MHz, CDCl $_{3}$) of **5d**



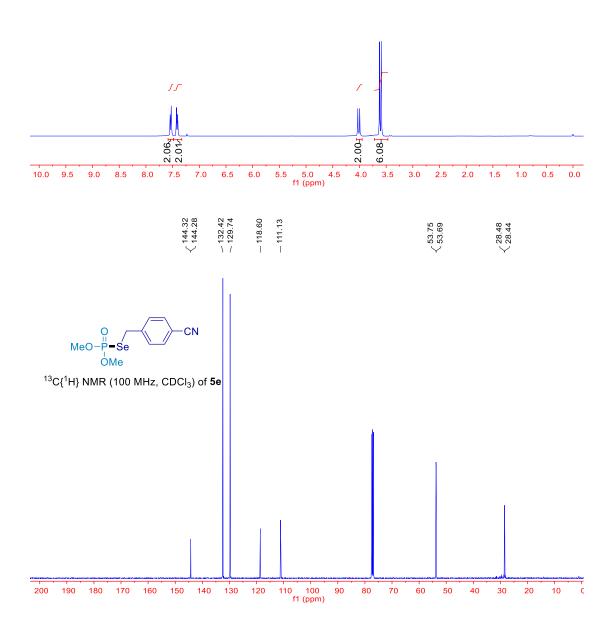


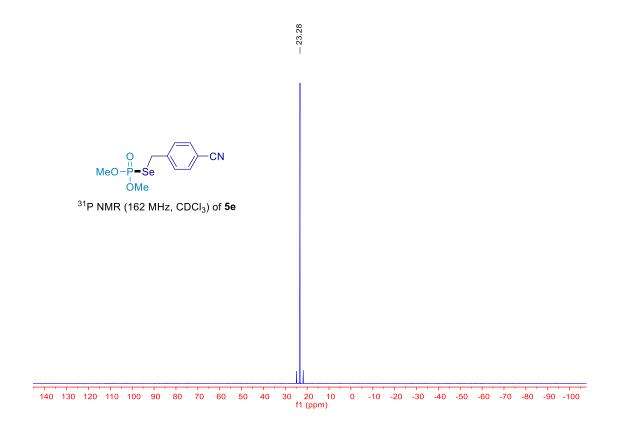


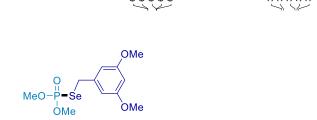
40 30 20 10 0 f1 (ppm) -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120



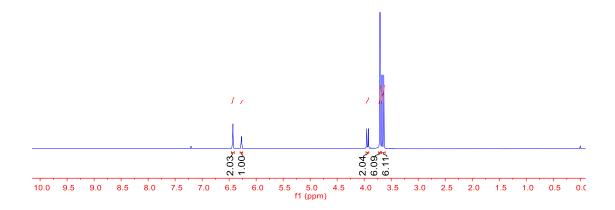


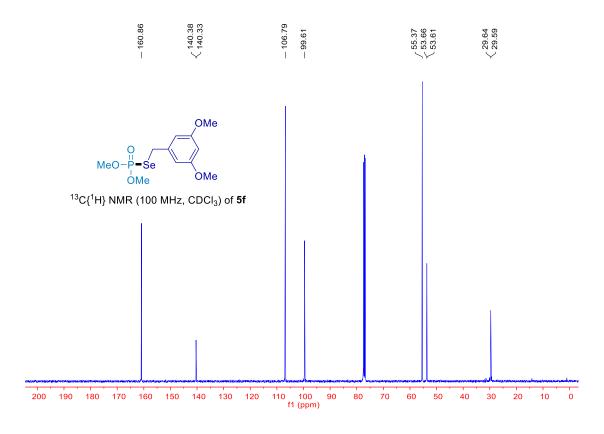


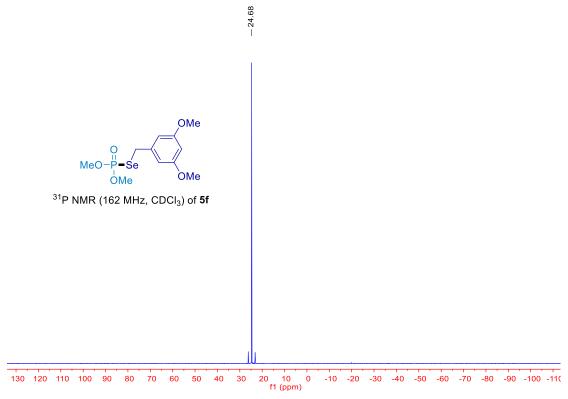


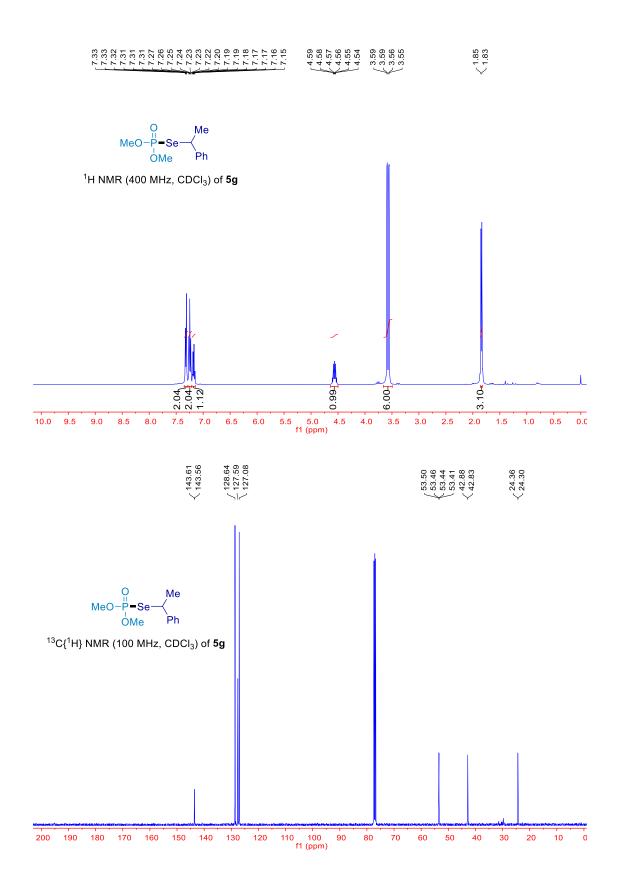


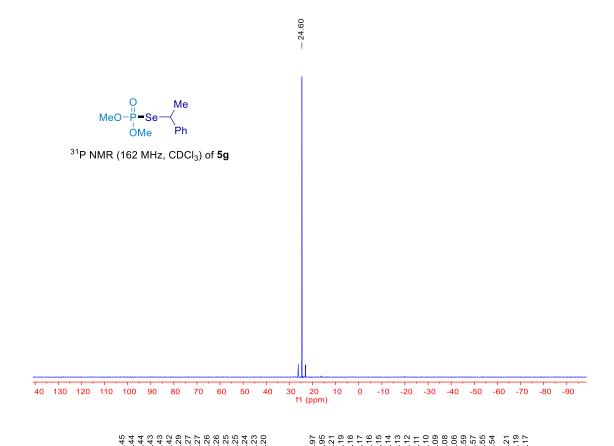
 1 H NMR (400 MHz, CDCl₃) of **5f**

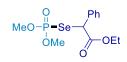




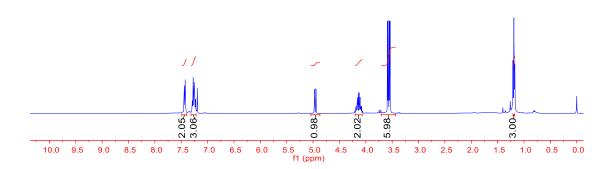


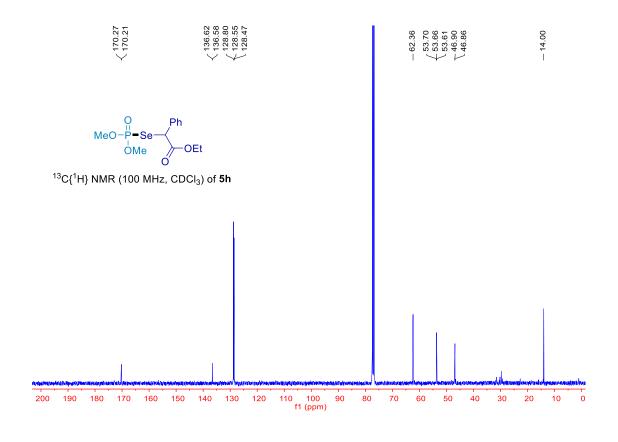


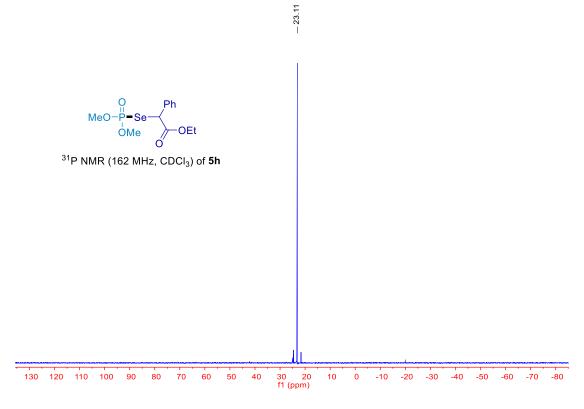




 1 H NMR (400 MHz, CDCl₃) of **5h**

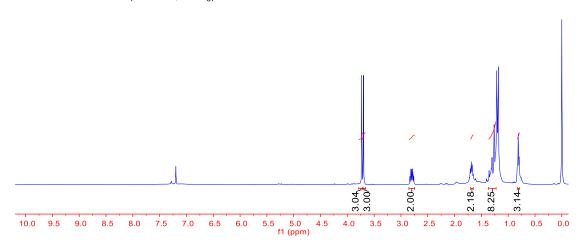


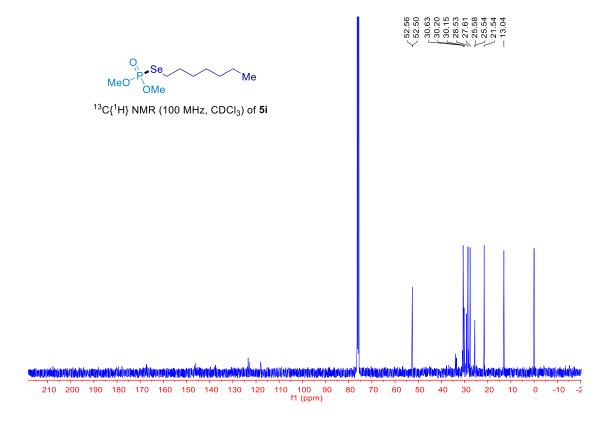


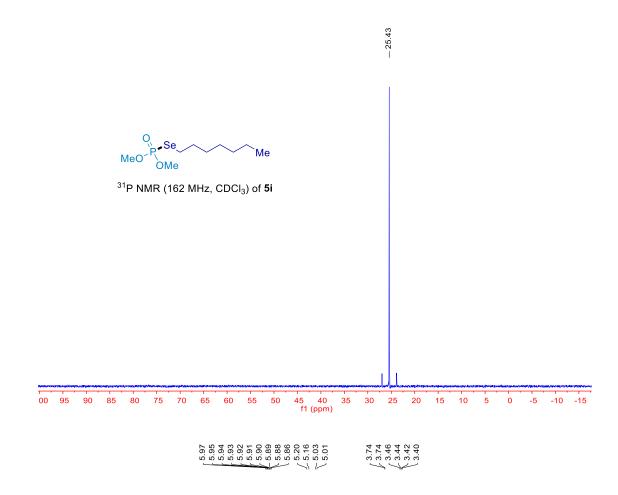


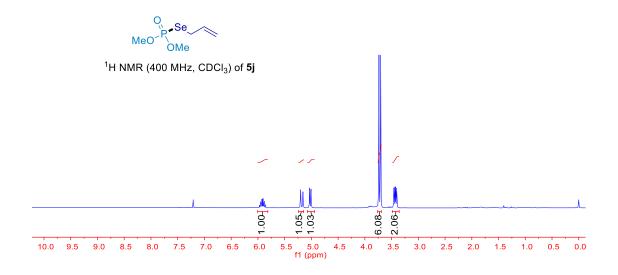


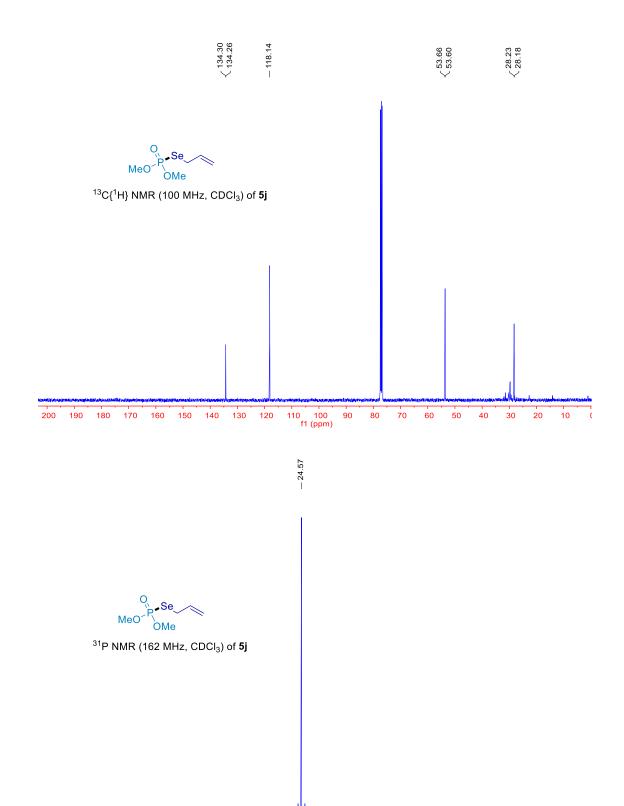
 1 H NMR (400 MHz, CDCl $_{3}$) of **5i**





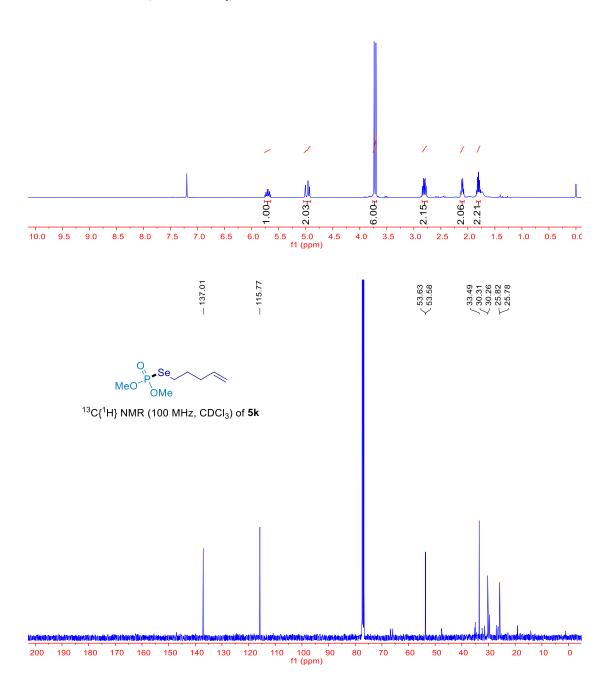




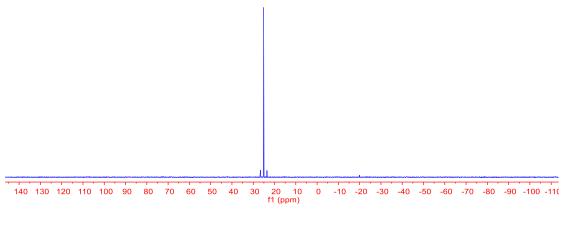


140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 f1 (ppm)

¹H NMR (400 MHz, CDCl₃) of **5k**

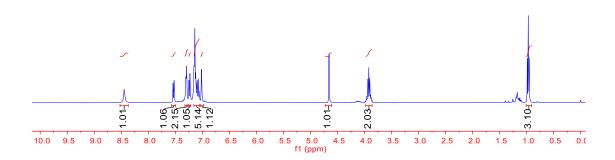


 31 P NMR (162 MHz, CDCl₃) of **5k**

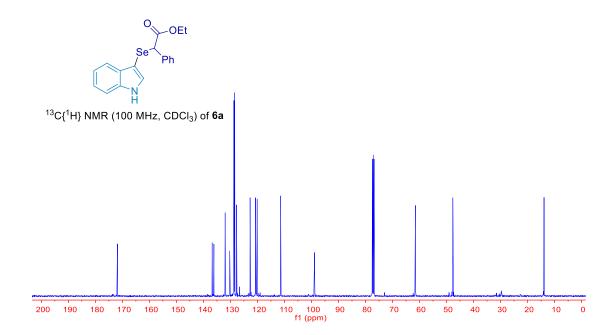




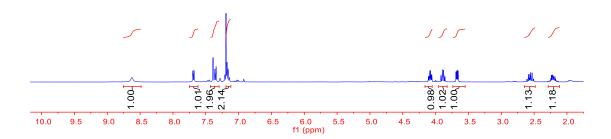
¹H NMR (400 MHz, CDCl₃) of **6a**

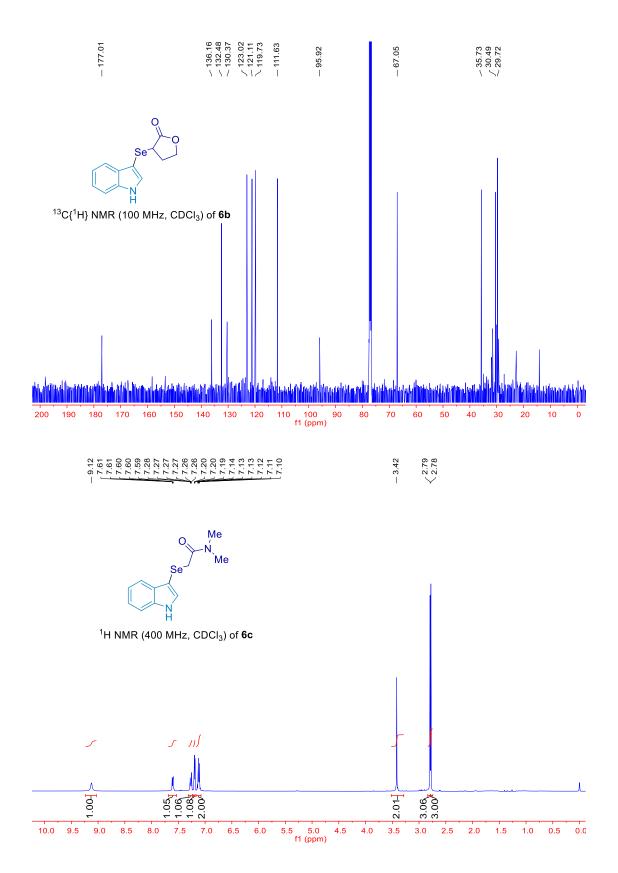


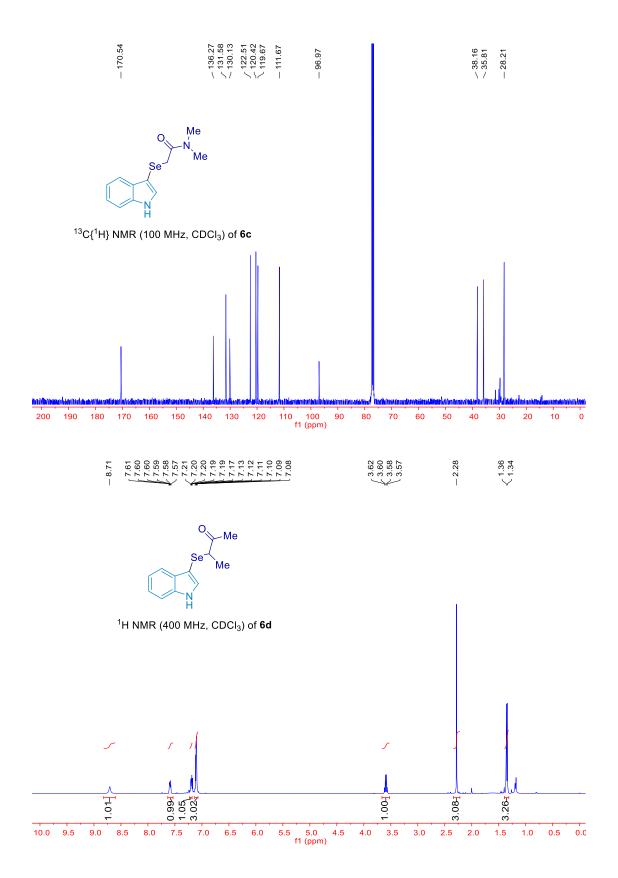


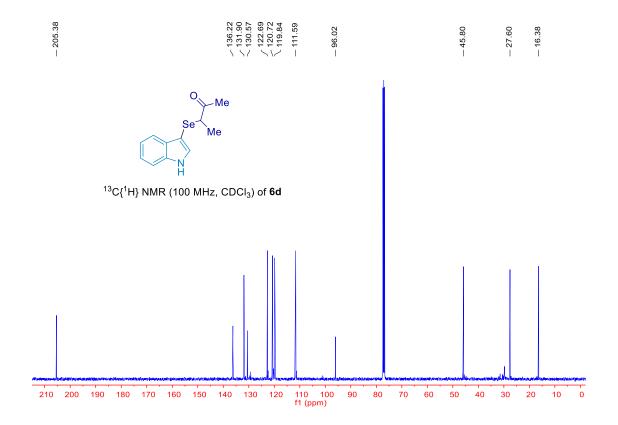


 1 H NMR (400 MHz, CDCl $_{3}$) of **6b**

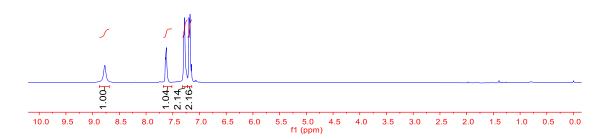


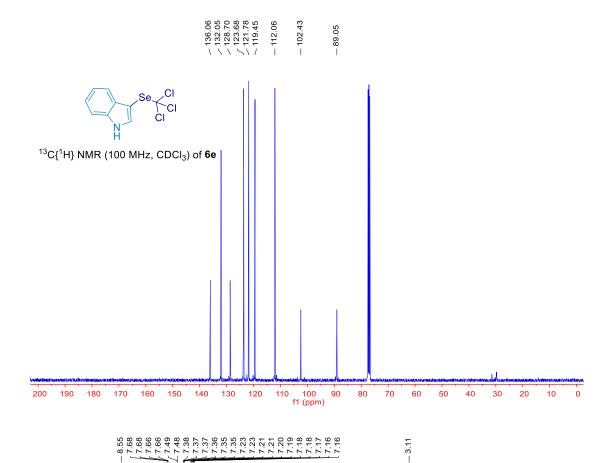


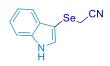




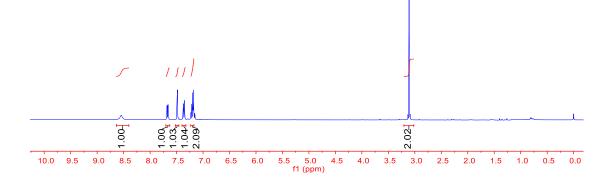
¹H NMR (400 MHz, CDCl₃) of **6e**

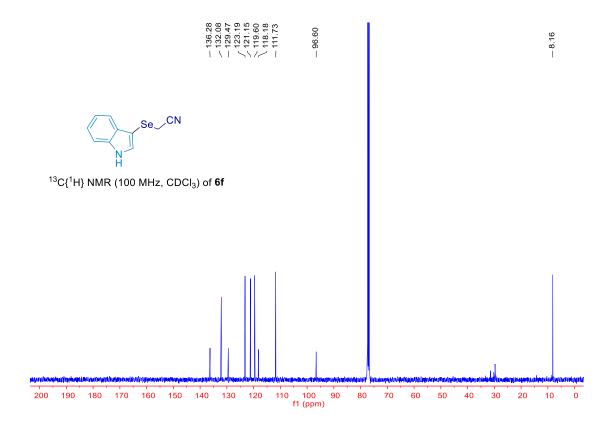


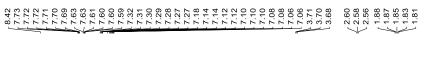




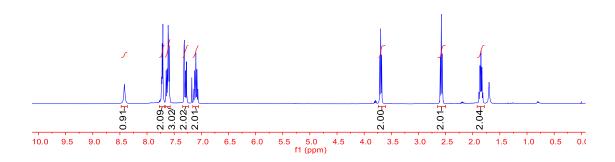
¹H NMR (400 MHz, CDCl₃) of **6f**

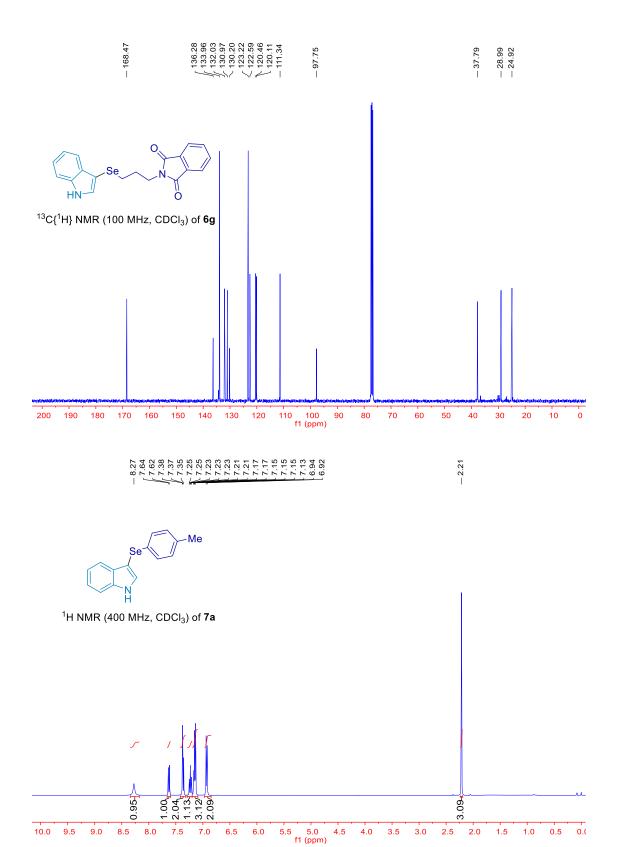


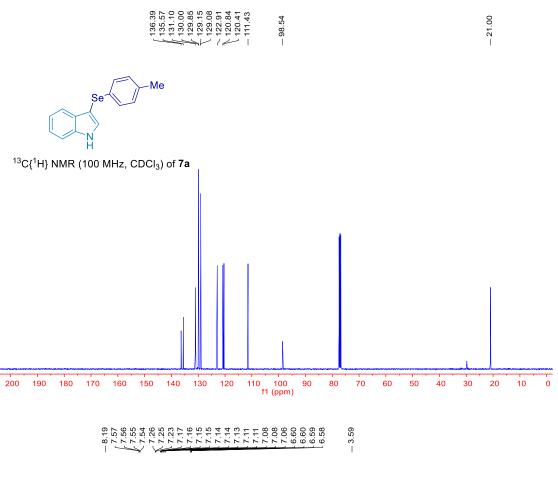


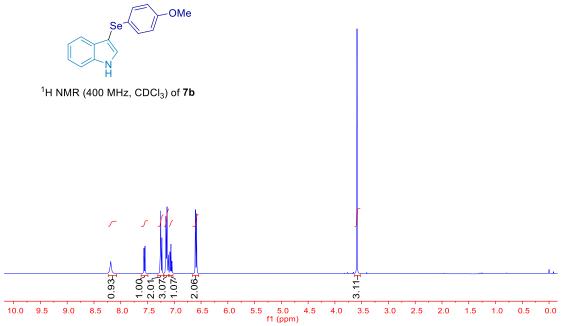


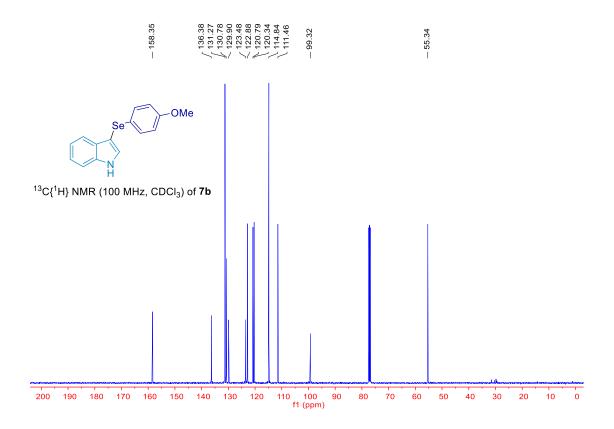
 1 H NMR (400 MHz, CDCl $_{3}$) of $\mathbf{6g}$





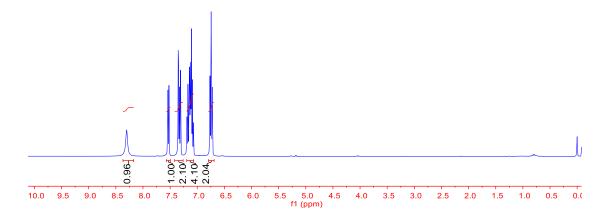




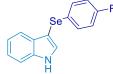


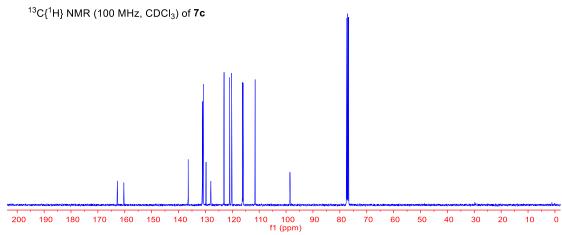


 1 H NMR (400 MHz, CDCl $_{3}$) of **7c**



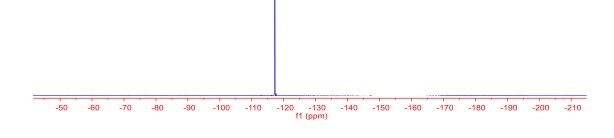




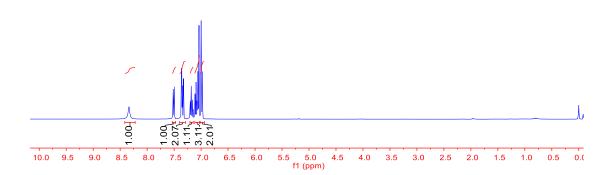




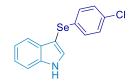
 ^{19}F NMR (376 MHz, CDCl $_3$) of 7c



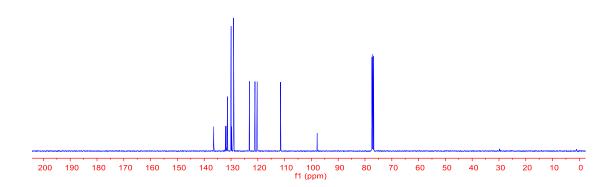
 1 H NMR (400 MHz, CDCl $_{3}$) of **7d**



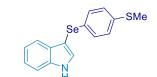
136.43 132.09 131.35 129.05 129.05 123.13 121.05 120.22 -111.53



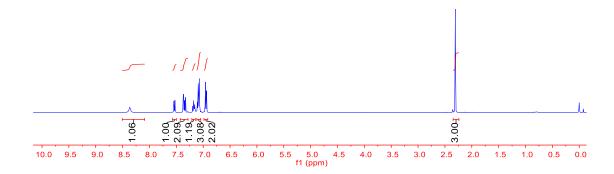
 $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl3) of 7d

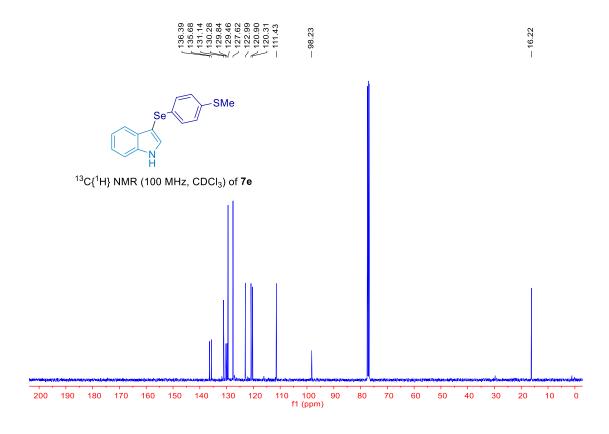






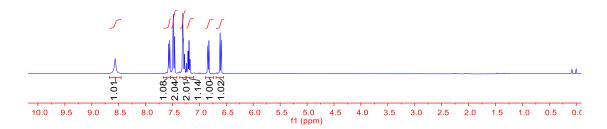
¹H NMR (400 MHz, CDCl₃) of **7e**

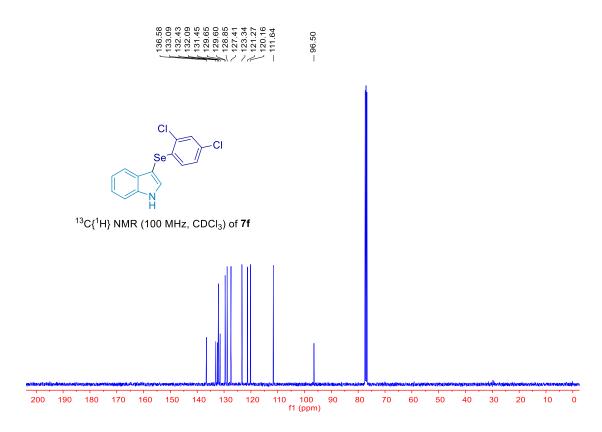


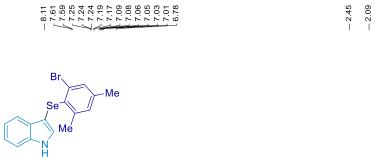


6.57 7.57 7.48 7.48 7.48 7.48 7.32 7.33 7.33 7.33 7.33 7.33 7.33 7.33 7.33 7.34

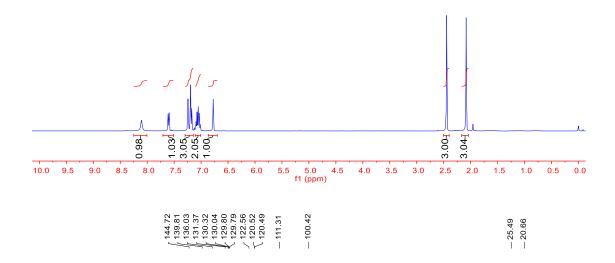
¹H NMR (400 MHz, CDCl₃) of **7f**

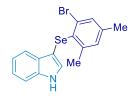




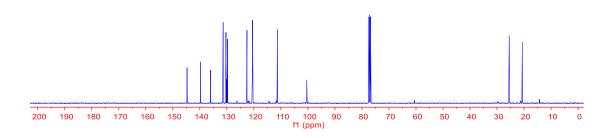


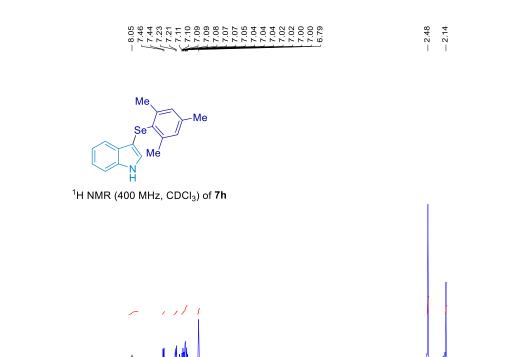
 1 H NMR (400 MHz, CDCl $_{3}$) of **7g**

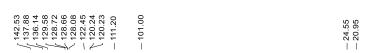




 $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl₃) of 7g







3.5

5.5 5.0 4.5 f1 (ppm)

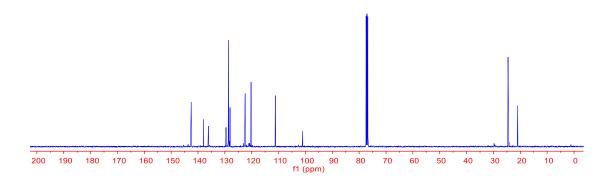
6.0

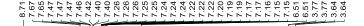
6.14<u>-</u> 3.04<u>-</u>

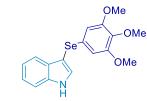
10.0 9.5

9.0 8.5

 $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl3) of 7h







¹H NMR (400 MHz, CDCl₃) of **7i**

