An Electrochemical Selenofunctionalization of Unactivated Alkenes: Access to *β*-Hydroxy-Selenides

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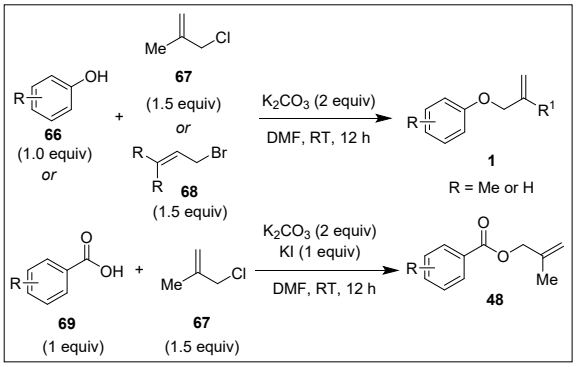
Supporting Information

Experimental:

General methods:

IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. ^{1}H NMR spectra were recorded on Bruker Advance 400 (400 MHz) and 600 (600 MHz) spectrometers at 295 K in $CDCl_3$; chemical shifts (δ ppm) and coupling constants (Hz) are reported in standard fashion concerning either internal standard tetramethylsilane (TMS) (δH = 0.00 ppm) or $CDCl_3$ (δH = 7.26 ppm). $^{13}C\{^{1}H\}$ NMR spectra were recorded on Bruker Advance 400 (100 MHz) and 600 (150 MHz) spectrometers at RT in $CDCl_3$. Chemical shifts (δ ppm) are reported relative to $CDCl_3$ [δ = 77.16 ppm (central line of the triplet)] and DMSO-d_6 [δ = 39.52 ppm (central line of the septate)]. In the $^{13}C\{^{1}H\}$ NMR, the nature of carbons (C, CH, CH_2, and CH_3) was determined by recording the DEPT-135 spectra and is given in parentheses and noted as s = singlet (for C), d = doublet (for CH), t = triplet (for CH_2) and q = quartet (for CH_3). In the ^{1}H-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublet, m = multiplet and br. s = broad singlet. The assignment of signals was confirmed by ^{1}H, $^{13}C\{^{1}H\}$ CPD, and DEPT spectra. High-resolution mass spectra (HRMS) were recorded on an Agilent 6538 UHD Q-TOF electron spray ionization (ESI) mode and atmospheric pressure chemical ionization (APCI) modes. Melting points are recorded using Tempo and Mettler FP1 melting point apparatus in capillary tubes and are uncorrected. Electrolysis reactions were conducted using ElectraSyn 2.0 Package supply purchased from IKA Instruments. Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Solvents were distilled before use; petroleum ether the boiling range of 60−80 °C was used. Cyclic voltammetry (CV) analysis was performed on Autolab PGSTAT 302 N, using a platinum disk electrode as working electrode, a platinum wire electrode as counter electrode and Ag/AgCl electrode as a reference electrode. Cyclic voltammogram was recorded at 50 mV/s scan rate. Phenols, 3-chloro-2-methylprop-1-ene, allyl bromide, CH_3CN, DCM, $LiClO_4$ and solvents were purchased from Sigma-Aldrich/Avra/BLD/TCI/local sources and used as received. Acme's silica gel (60–120 mesh) was used for column chromatography (approximately 20 g per one gram of crude material).

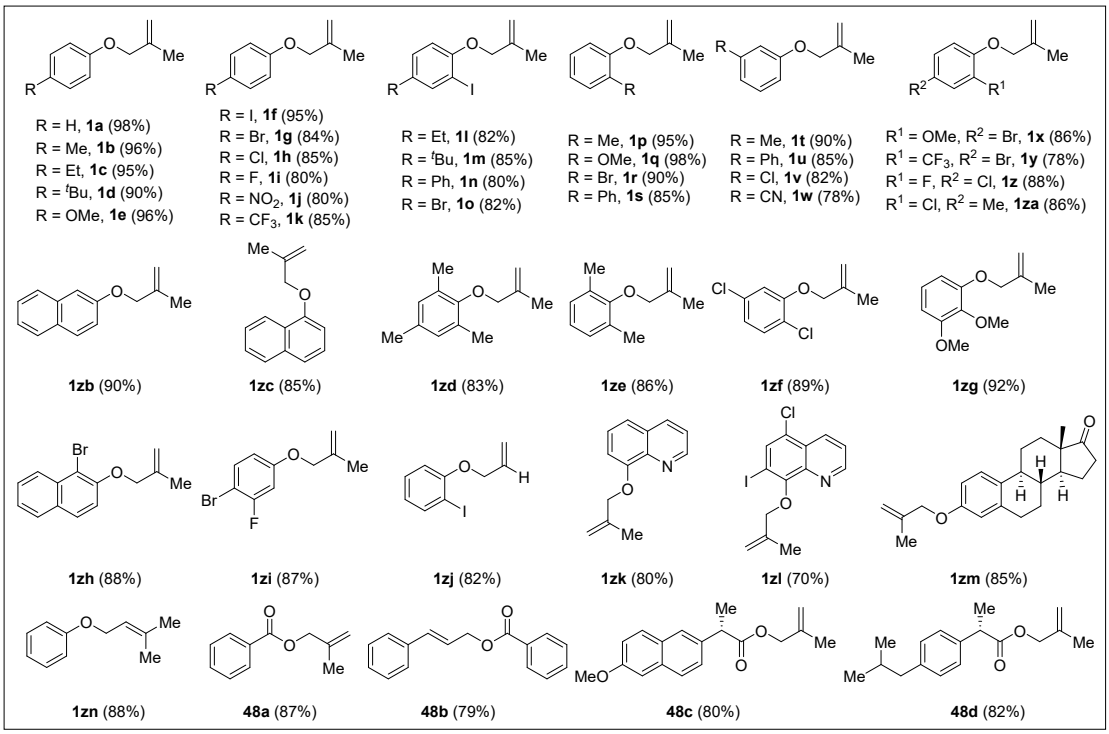
General Procedure - 1 (GP-1) for the Preparation of Allyl Ethers/Esters (1/48):



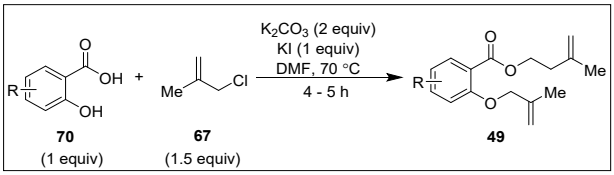
The reaction was carried out in a 25 mL oven-dried round bottom flask equipped with magnetic stir bar and charged with phenols **66** (1.0 equiv), 3-chloro-2-methylprop-1-ene **67** / allyl bromides **68** (1.5 equiv), K_2CO_3 (2.0 equiv), and DMF (8 mL). The resulting reaction mixture was stirred at rt for 12 h. The reaction progress was monitored by TLC. After completion of the reaction, it was worked up with DCM (3 × 10 mL) and the combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 100:00–95:05) as the eluent furnished the desired product **1** (70 to 98%), as yellow liquids.

The reaction was carried out in a 25 mL oven-dried round bottom flask equipped with magnetic stir bar and charged with acids **69** (1.0 equiv), 3-chloro-2-methylprop-1-ene **67**, K_2CO_3 (2.0 equiv), KI (1 equiv), and DMF (8 mL). The resulting reaction mixture was stirred at rt for 12 h. The reaction progress was monitored by TLC. After completion of the reaction, it was worked up with DCM (3 × 10 mL) and the combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 100:00–95:05) as the eluent furnished the desired product **48** (80 to 87%), as yellow liquids.

Table-S1. The following starting material ((2-methylallyl)oxy)benzenes **1** / 2-methylallyl benzoates **48** are prepared by using the literature.[1–3]

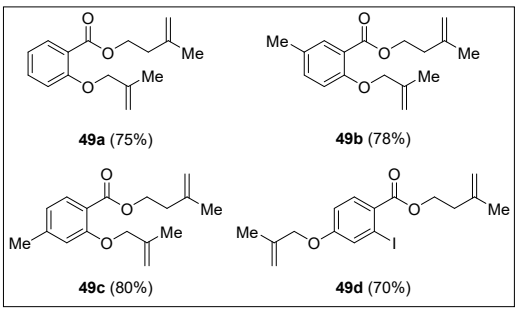


General Procedure - 2 (GP-2) for the Preparation of 3-Methylbut-3-en-1-yl 2-((2-methylallyl)oxy)benzoates 49:

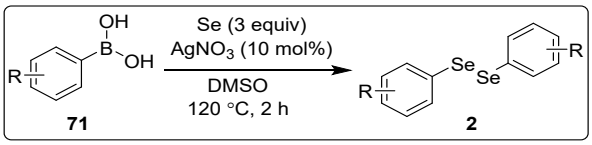


The reaction was carried out in a 25 mL oven-dried round bottom flask equipped with magnetic stir bar and charged with hydroxy acids **70** (1.0 equiv), 3-chloro-2-methylprop-1-ene **67** (1.5 equiv), K_2CO_3 (2.0 equiv), KI (1 equiv), and DMF (8 mL). The resulting reaction mixture was stirred at rt to 70 °C for 4-5 h. The reaction progress was monitored by TLC. After completion of the reaction, it was worked up with DCM (3 × 10 mL) and the combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 100:00–95:05) as the eluent furnished the desired product **49** (80 to 90%), as yellow liquids.

Table-S2. The following starting material 3-methylbut-3-en-1-yl 2-((2-methylallyl)oxy)benzoates **49** are prepared by using the literature.[4]

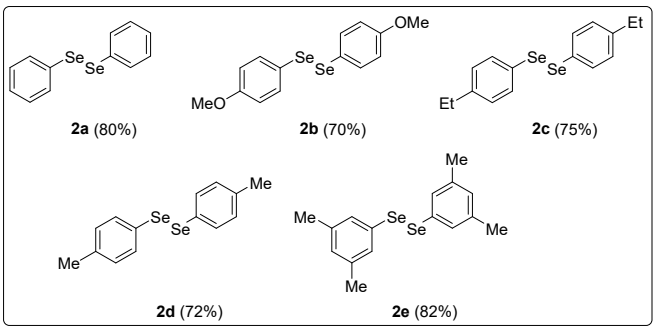


General Procedure - 3 (GP-3) for the Preparation of Diaryl Diselenides 2.



The reaction was carried out in a 25 mL oven-dried round bottom flask equipped with magnetic stir bar and charged with arylboronic acid **71** (1 equiv), selenium (3 equiv), $AgNO_3$ (10 mol%), and DMSO (2.0 mL). The mixture was stirred in a heating mantle preheated to 120 °C for 2 h. The reaction progress was monitored by TLC. After cooling to room temperature, the reaction mixture was diluted with H_2O (10 mL) and extracted with EtOAc (3×10 mL). The combined organic phase was washed with water and brine (30mL), dried over anhydrous Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 100:00–95:05) as the eluent furnished the desired product **2** (70 to 85%), as yellow liquids or yellow solids (Table S3).

Table-S3. The following starting material diaryl diselenide **2a-2e** are prepared by using the literature.[5]



General Procedure - 4 (GP-4) for the Preparation of *β*-hydroxy selenides 3-47:

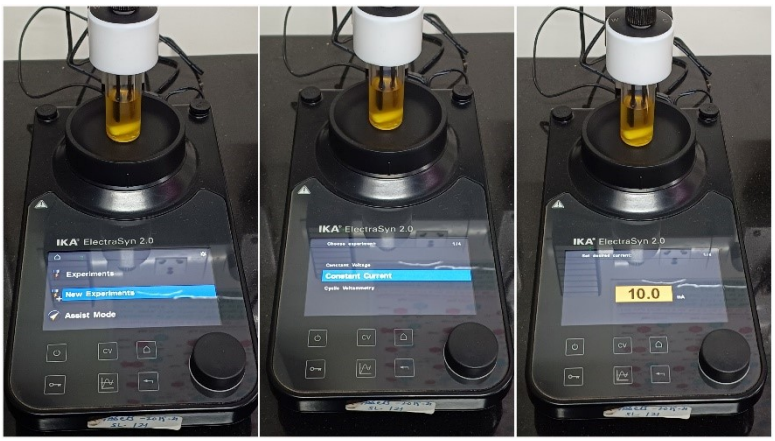
To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added allylphenyl ether **1** (0.37 mmol, 1 equiv), diphenyl diselenide **2** (0.29 mmol, 0.8 equiv), and $LiClO_4$ (32

mg, 0.05 M) in 6 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 2 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the desired product *β*-hydroxy selenides i.e., (2-methyl-4-aryl-1-(phenylselanyl)butan-2-ols) **3-47** (70% to 98% yields).

General Procedure - 5 (GP-5) for the Preparation of *β*-hydroxy selenides 50-58:

To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added 2-methylallyl benzoate **48** / 2-methylallyl alkyl-2-((2-methylallyl)oxy)benzoate **49** (0.37 mmol, 1 equiv), diphenyl diselenide **2** (0.29 mmol, 0.8 equiv), and $LiClO_4$ (32 mg, 0.05 M) in 6 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 2 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the corresponding desired *β*-hydroxy selenides **50-58** (75% to 89%).

Photographic guide for electrochemical reaction:



Left: Select New “Experiment” **Middle:** Select “Constant Current” **Right:** Select “10 mA”

Left: Reference electrode chose “No” **Middle:** Select “Time” **Right:** Select “2 h”

Left: Select “0.37 mmol” **Middle:** Alternate the polarity Chose “No” **Right:** Select “Start the experiment”

Cyclic Voltammograms:

The cyclic voltammetry (CV) studies were carried out to further investigate the reaction mechanism, and below Figure 1S shows the cyclic voltammetry (CV) curves with 0.1 M $LiClO_4$ solution in CH_3CN as a background. The voltammogram was obtained at a scan rate of 50 mV/s with Pt wire as a counter electrode, Ag/AgCl as a reference electrode which is submerged in saturated aqueous KCl solution, and Pt disk electrode as a working electrode. Within the scanning window (0 to 2.5 V). (1) An obvious there is no peak for background (Black curve). (2) The CV of ((2-methylallyl)oxy)benzene **1a** displayed oxidation peak at 1.75 V *vs* Ag/AgCl (Red curve). (3) When testing diphenyl diselenide **2a**, an oxidation peak was seemed at 1.40 V *vs* Ag/AgCl (Blue curve). (4) The mixture of ((2-methylallyl)oxy)benzene

1a and diphenyl diselenide **2a** showed two oxidation signals at 1.84 V and 1.33 V *vs* Ag/AgCl (Pink curve), respectively.

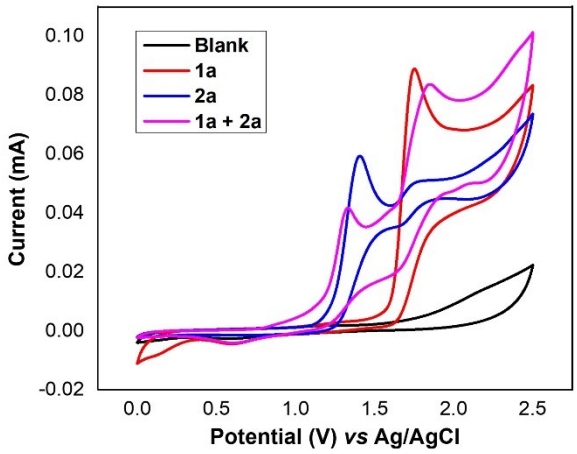
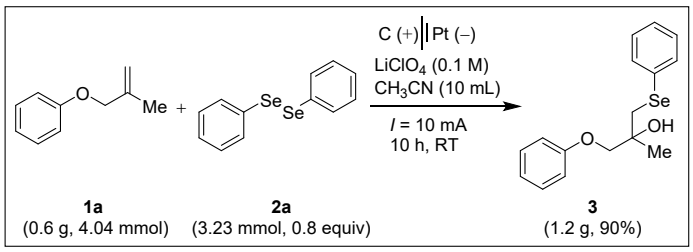


Figure S1. Cyclic voltammograms of reactants and their mixtures in 0.1 M $LiClO_4$ solution in CH_3CN at room temperature: 1) Background; 2) ((2-methylallyl)oxy)benzene **1a** (0.01 M); 3) diphenyl diselenide **2a** (0.01 M); 4) ((2-methylallyl)oxy)benzene **1a** (0.01 M) + diphenyl diselenide **2a** (0.01 M); The voltammogram was obtained at a scan rate of 50 mV/s with Pt wire as a counter electrode, Ag/AgCl as a reference electrode, and Pt disk electrode as a working electrode.

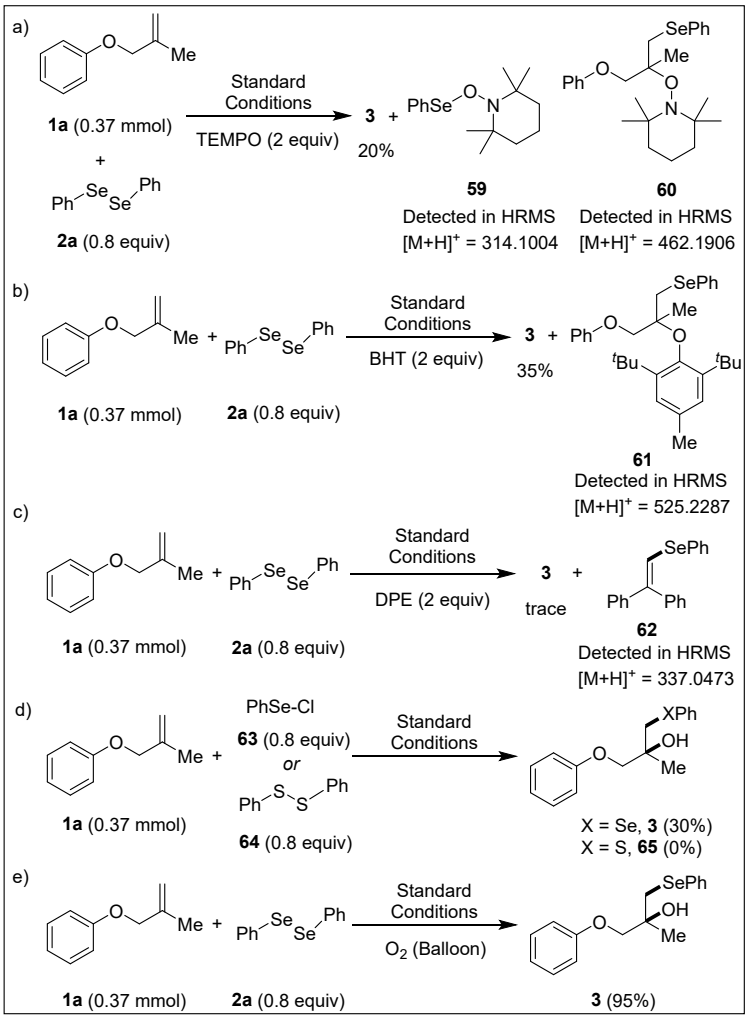
Scheme S1: Scale up reaction.



To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added ((2-methylallyl)oxy)benzene **1a** (600 mg, 4.04 mmol), diphenyl diselenide **2a** (3.23 mmol, 0.8 equiv), and $LiClO_4$ (32 mg, 0.05 M) in 10 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 10 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (5 mL) and the mixture was poured into aqueous NH_4Cl solution (50 mL) and extracted with DCM (5 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica

gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the desired product 2-methyl-1-phenoxy-3-(phenylselanyl)propan-2-ol **3** (1.2 g, 90%), as yellow oil.

Scheme S2: Control Experiments



a) To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added allylphenyl ether **1a** (55 mg, 0.37 mmol), diphenyl diselenide **2a** (0.29 mmol, 0.8 equiv), TEMPO (116 mg, 2 equiv), and $LiClO_4$ (32 mg, 0.05 M) in 6 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 2 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure. The crude mixture was subjected to mass

spectrometric analysis to confirm the formation of 2,2,6,6-tetramethyl-1-((phenylselanyl)oxy)piperidine **59** and 2,2,6,6-tetramethyl-1-((2-methyl-1-phenoxy-3-(phenylselanyl)propan-2-yl)oxy)piperidine **60**. HRMS-ESI (m/z) for **59**: calcd for $C_{15}H_{24}NOSe^+$ $[M + H]^+$ 314.1018; found 314.1004. HRMS-ESI (m/z) for **60**: calcd for $C_{25}H_{36}NO_2Se^+$ $[M + H]^+$ 462.1906; found 462.1906.

b) To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added allylphenyl ether **1a** (55 mg, 0.37 mmol), diphenyl diselenide **2a** (0.29 mmol, 0.8 equiv), BHT (163 mg, 2 equiv), and $LiClO_4$ (32 mg, 0.05 M) in 6 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 2 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure. The crude mixture was subjected to mass spectrometric analysis to confirm the formation of (2-(2,6-di-*tert*-butyl-4-methylphenoxy)-2-methyl-3-phenoxypropyl)(phenyl)selane **61**. HRMS-ESI (m/z): calcd for $C_{31}H_{41}O_2Se^+$ $[M + H]^+$ 525.2266; found 525.2287.

c) To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added allylphenyl ether **1a** (55 mg, 0.37 mmol), diphenyl diselenide **2a** (0.29 mmol, 0.8 equiv), DPE (133 mg, 2 equiv), and $LiClO_4$ (32 mg, 0.05 M) in 6 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 2 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure. The crude mixture was subjected to mass spectrometric analysis to confirm the formation of (2,2-diphenylvinyl)(phenyl)selane **62**. HRMS-ESI (m/z): calcd for $C_{20}H_{17}Se^+$ $[M + H]^+$ 337.0490; found 337.0473.

d) To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added allylphenyl ether **1a** (55 mg, 0.37 mmol), PhSeCl **63** (0.29 mmol, 0.8 equiv) or diphenyl disulfide **64** (0.29 mmol, 0.8 equiv), and $LiClO_4$ (32 mg, 0.05 M) in 6 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 2 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the desired product **3** (30%) and **65** (0%), of yield respectively.

e) To an oven dried ElectraSyn vial (10 mL) with magnetic stirring bar, were added allylphenyl ether **1a** (55 mg, 0.37 mmol), diphenyl diselenide **2a** (0.29 mmol, 0.8 equiv), and $LiClO_4$ (32 mg, 0.05 M) in 6 mL CH_3CN. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum) were inserted into the mixture and kept O_2 Balloon. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 2 h. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the desired product **3** in 95% of yield.

Karl Fischer Titration:

F-Factor of water equivalent = M (mass of distilled water) / A (Volume of KFR)

F-Factor of water equivalent = 25 mg/5.05 mL

F-Factor of water equivalent = **4.95 mg/mL**

4.716 g (6 mL of CH_3CN) of sample consumed a titre of 13.5 mL with a factor of 4.95. So,

13.5 × 4.95 = **66.8 mg** (~ 10 equiv with respect to the substrate **1** taken) of water present in the sample of 4.716 g (4716 mg/ 6 mL of CH_3CN).

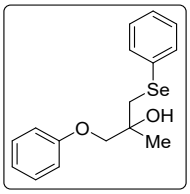
% of water content = B (mL of KFR) × F (Factor) / V (Volume of sample) × D (density of sample) × 10

Or

% of water content = B (mL of KFR) × F (Factor) / M (weight of grams of sample) × 10

% of water content = 13.5 × 4.95 / 6 × 0.786 × 10 = 66.15 / 47.16 = **1.40%**

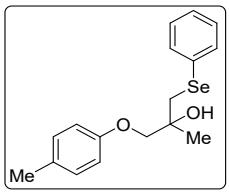
Characterization of compounds:



2-Methyl-1-phenoxy-3-(phenylselanyl)propan-2-ol (3):

GP4 was carried out with ((2-methylallyl)oxy)benzene **1a** (55 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3** (113 mg, 95%), as a yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.90, R_f (**2a**) = 0.95, R_f (**3**) = 0.20, UV detection].

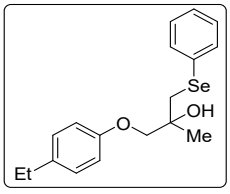
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3438, 3056, 2927, 1591, 1484, 1383, 1236, 1169, 1045, 838, 746, 686 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.55 – 7.49 (m, 2H), 7.28 – 7.21 (m, 2H), 7.20 – 7.13 (m, 3H), 6.97 – 6.91 (m, 1H), 6.83 – 6.78 (m, 2H), 3.89 (d, J = 8.9 Hz, 1H), 3.82 (d, J = 8.9 Hz, 1H), 3.36 (d, J = 12.7 Hz, 1H), 3.21 (d, J = 12.7 Hz, 1H), 2.76 (br. s, 1H), 1.40 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 158.4, 132.7 (2 × Ar–CH), 130.6, 129.5 (2 × Ar–CH), 129.2 (2 × Ar–CH), 127.1, 121.2, 114.6 (2 × Ar–CH), 73.5, 72.1, 38.8, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{18}KO_2Se^+$ $[M+K]^+$: 361.0104, found: 361.0108.



2-Methyl-1-(phenylselanyl)-3-(*p*-tolyloxy)propan-2-ol (4):

GP4 was carried out with 1-methyl-4-((2-methylallyl)oxy)benzene **1b** (60 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **4** (121 mg, 98%), as a yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1b**) = 0.90, R_f (**2a**) = 0.95, R_f (**4**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3433, 2927, 1595, 1509, 1239, 1044, 817, 749 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.59 – 7.50 (m, 2H), 7.25 – 7.14 (m, 3H), 7.06 (d, J = 8.6 Hz, 2H), 6.74 (d, J = 8.5 Hz, 2H), 3.90 (d, J = 8.9 Hz, 1H), 3.82 (d, J = 8.9 Hz, 1H), 3.38 (d, J = 12.7 Hz, 1H), 3.22 (d, J = 12.7 Hz, 1H), 2.77 (s, 1H), 2.29 (br. s, 3H), 1.41 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.4, 132.7 (2 × Ar–CH), 130.7, 130.5, 129.9 (2 × Ar–CH), 129.2 (2 × Ar–CH), 127.1, 114.5 (2 × Ar–CH), 73.8, 72.1, 38.9, 24.5, 20.6 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{20}KO_2Se^+$ $[M+K]^+$: 375.0260, found: 375.0264.

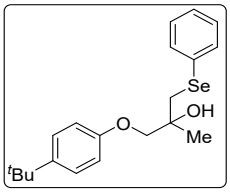


1-(4-Ethylphenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (5):

GP4 was carried out with 1-ethyl-4-((2-methylallyl)oxy)benzene **1c** (65 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **5** (118 mg, 92%), as a yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1c**) = 0.90, R_f (**2a**) = 0.95, R_f (**5**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3435, 3051, 2958, 1596, 1508, 1456, 1236, 1043, 828, 741, 685 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.59 – 7.52 (m, 2H), 7.23 – 7.17 (m, 3H), 7.13 – 7.07 (m, 2H), 6.81 – 6.75 (m, 2H), 3.92 (d, J = 8.8 Hz, 1H), 3.84 (d, J = 8.8 Hz, 1H), 3.39 (d, J = 12.7 Hz, 1H), 3.24 (d, J = 12.7 Hz, 1H), 2.83 (br. s, 1H), 2.61 (q, J = 7.6 Hz, 2H), 1.43 (s,

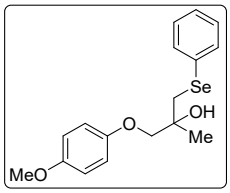
3H), 1.24 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.5, 137.0, 132.6 (2 × Ar–CH), 130.7, 129.2 (2 × Ar–CH), 128.8 (2 × Ar–CH), 127.1, 114.5 (2 × Ar–CH), 73.7, 72.1, 38.8, 28.1, 24.4, 16.0 ppm. **HRMS:** *m/z* calcd for $C_{18}H_{22}KO_2Se^+$ $[M+K]^+$: 389.0417, found: 389.0423.



1-(4-(*Tert*-butyl)phenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (6):

GP4 was carried out with 1-(*tert*-butyl)-4-((2-methylallyl)oxy)benzene **1d** (75 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **6** (111 mg, 80%), as a yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1d**) = 0.90, R_f (**2a**) = 0.95, R_f (**6**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3437, 3055, 2955, 1598, 1509, 1461, 1240, 1180, 1038, 825, 742 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.50 – 7.38 (m, 2H), 7.21 – 7.15 (m, 2H), 7.14 – 7.02 (m, 3H), 6.71 – 6.64 (m, 2H), 3.81 (d, J = 8.9 Hz, 1H), 3.73 (d, J = 8.9 Hz, 1H), 3.28 (d, J = 12.7 Hz, 1H), 3.12 (d, J = 12.7 Hz, 1H), 2.68 (br. S, 1H), 1.31 (s, 3H), 1.21 (s, 9H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.2, 143.9, 132.7 (2 × Ar–CH), 130.7, 129.2 (2 × Ar–CH), 127.1, 126.3 (2 × Ar–CH), 114.1 (2 × Ar–CH), 73.6, 72.1, 38.9, 34.2, 31.6 (3 × CH_3), 24.5 ppm. **HRMS:** *m/z* calcd for $C_{20}H_{26}NaO_2Se^+$ $[M+Na]^+$: 401.0990, found: 401.1009.

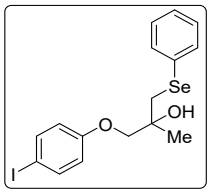


1-(4-Methoxyphenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (7):

GP4 was carried out with 1-methoxy-4-((2-methylallyl)oxy)benzene **1e** (66 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **7** (125 mg, 96%), as a yellow liquid [TLC

control (petroleum ether/ethyl acetate 95:05), R_f (**1e**) = 0.80, R_f (**2a**) = 0.95, R_f (**7**) = 0.10, UV detection].

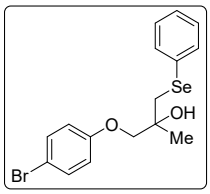
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3455, 3057, 2929, 1581, 1505, 1455, 1225, 1014, 826, 747 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.57 – 7.51 (m, 2H), 7.23 – 7.15 (m, 3H), 6.84 – 6.74 (m, 4H), 3.88 (d, J = 8.9 Hz, 1H), 3.80 (d, J = 8.9 Hz, 1H), 3.77 (s, 3H), 3.37 (d, J = 12.7 Hz, 1H), 3.22 (d, J = 12.7 Hz, 1H), 2.82 (br. s, 1H), 1.41 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 154.1, 152.6, 132.6 (2 × Ar–CH), 130.7, 129.2 (2 × Ar–CH), 127.1, 115.6 (2 × Ar–CH), 114.6 (2 × Ar–CH), 74.4, 72.1, 55.8, 38.8, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{20}NaO_3Se^+$ $[M+Na]^+$: 375.0470, found: 375.0484.



1-(4-Iodophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (8):

GP4 was carried out with 1-iodo-4-((2-methylallyl)oxy)benzene **1f** (101 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **8** (138 mg, 84%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1f**) = 0.90, R_f (**2a**) = 0.95, R_f (**8**) = 0.20, UV detection].

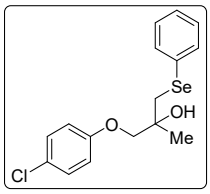
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3429, 3060, 2927, 1579, 1475, 1235, 1036, 816, 744 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.54 – 7.47 (m, 4H), 7.21 – 7.11 (m, 3H), 6.59 – 6.52 (m, 2H), 3.84 (d, J = 8.9 Hz, 1H), 3.78 (d, J = 8.9 Hz, 1H), 3.35 (d, J = 12.8 Hz, 1H), 3.20 (d, J = 12.8 Hz, 1H), 2.79 (br. s, 1H), 1.41 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 158.2, 138.2 (2 × Ar–CH), 132.7 (2 × Ar–CH), 130.4, 129.2 (2 × Ar–CH), 127.2, 116.9 (2 × Ar–CH), 83.3, 73.5, 72.0, 38.8, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{17}INaO_2Se^+$ $[M+Na]^+$: 470.9331, found: 470.9336.



1-(4-Bromophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (9):

GP4 was carried out with 1-bromo-4-((2-methylallyl)oxy)benzene **1g** (84 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **9** (115 mg, 78%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1g**) = 0.90, R_f (**2a**) = 0.95, R_f (**9**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3439, 3061, 2929, 1583, 1481, 1239, 1039, 821, 747 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 7.45 – 7.38 (m, 2H), 7.24 – 7.19 (m, 2H), 7.11 – 7.01 (m, 3H), 6.58 – 6.52 (m, 2H), 3.73 (d, J = 8.8 Hz, 1H), 3.67 (d, J = 8.8 Hz, 1H), 3.24 (d, J = 12.8 Hz, 1H), 3.09 (d, J = 12.8 Hz, 1H), 2.70 (br. s, 1H), 1.30 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 157.4, 132.7 (2 × Ar–CH), 132.2 (2 × Ar–CH), 130.3, 129.2 (2 × Ar–CH), 127.2, 116.3 (2 × Ar–CH), 113.3, 73.6, 71.9, 38.7, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{17}Br^{79}KO_2Se^+$ $[M+K]^+$: 438.9209, found: 438.9215; $C_{16}H_{17}Br^{81}KO_2Se^+$ $[M+K]^+$: 440.9188, found: 440.9208.

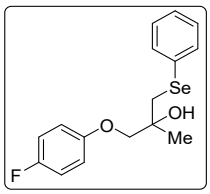


1-(4-Chlorophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (10):

GP4 was carried out with 1-chloro-4-((2-methylallyl)oxy)benzene **1h** (67 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **10** (104 mg, 80%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1h**) = 0.90, R_f (**2a**) = 0.95, R_f (**10**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3442, 3060, 2980, 1586, 1486, 1243, 1039, 910, 824, 751 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.55 – 7.47 (m, 2H), 7.22 – 7.11 (m, 5H), 6.73 – 6.66 (m, 2H), 3.84 (d, J = 8.8 Hz, 1H), 3.77 (d, J = 8.8 Hz, 1H), 3.34 (d, J = 12.8 Hz, 1H), 3.19

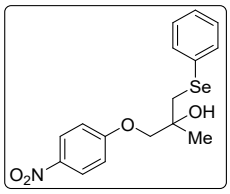
(d, J = 12.8 Hz, 1H), 2.73 (br. s, 1H), 1.39 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.9, 132.8 (2 × Ar–CH), 130.4, 129.3 (2 × Ar–CH), 129.2 (2 × Ar–CH), 127.2, 126.1, 115.9 (2 × Ar–CH), 73.8, 72.0, 38.8, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{17}ClO_2Se^+$ $[M^+]^+$: 356.0077, found: 356.0121.



1-(4-Fluorophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (11):

GP4 was carried out with 1-fluoro-4-((2-methylallyl)oxy)benzene **1i** (61 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **11** (102 mg, 82%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1i**) = 0.90, R_f (**2a**) = 0.95, R_f (**11**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3455, 3062, 2927, 1581, 1499, 1202, 1036, 910, 823, 743 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.60 – 7.46 (m, 2H), 7.24 – 7.11 (m, 3H), 6.98 – 6.88 (m, 2H), 6.77 – 6.69 (m, 2H), 3.86 (d, J = 8.8 Hz, 1H), 3.79 (d, J = 8.8 Hz, 1H), 3.37 (d, J = 12.8 Hz, 1H), 3.22 (d, J = 12.8 Hz, 1H), 2.83 (br. s, 1H), 1.42 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 157.5 (d, $J_{C\text{-}F}$ = 238.6 Hz) 154.5 (d, $J_{C\text{-}F}$ = 1.7 Hz), 132.7 (2 × Ar–CH), 130.5, 129.2 (2 × Ar–CH), 127.1, 115.8 (d, $J_{C\text{-}F}$ = 23.0 Hz), 115.6 (d, $J_{C\text{-}F}$ = 8.0 Hz), 74.1, 72.1, 38.7, 24.4 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –123.46 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{17}FNaO_2Se^+$ $[M+Na]^+$: 363.0270, found: 363.0260.

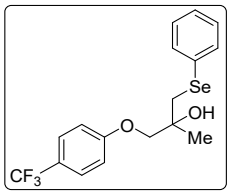


2-Methyl-1-(4-nitrophenoxy)-3-(phenylselanyl)propan-2-ol (12):

GP4 was carried out with 1-((2-methylallyl)oxy)-4-nitrobenzene **1j** (71 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **12** (110 mg, 82%), as a yellow liquid [TLC

control (petroleum ether/ethyl acetate 95:05), R_f (**1j**) = 0.85, R_f (**2a**) = 0.95, R_f (**12**) = 0.15, UV detection].

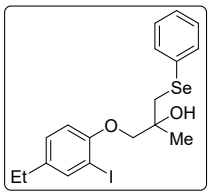
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3458, 3070, 2931, 1593, 1505, 1335, 1259, 1117, 1111, 850, 750 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.17 – 8.06 (m, 2H), 7.54 – 7.46 (m, 2H), 7.21 – 6.99 (m, 3H), 6.85 – 6.75 (m, 2H), 3.93 (d, J = 8.9 Hz, 1H), 3.88 (d, J = 8.9 Hz, 1H), 3.36 (d, J = 13.0 Hz, 1H), 3.20 (d, J = 13.0 Hz, 1H), 2.68 (br. s, 1H), 1.43 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 163.2, 141.8, 132.9 (2 × Ar–CH), 130.0, 129.3 (2 × Ar–CH), 127.4, 125.8 (2 × Ar–CH), 114.5 (2 × Ar–CH), 73.9, 71.9, 38.7, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{18}NO_4Se^+$ $[M+H]^+$: 368.0396, found: 368.0405.



2-Methyl-1-(phenylselanyl)-3-(4-(trifluoromethyl)phenoxy)propan-2-ol (13):

GP4 was carried out with 1-((2-methylallyl)oxy)-4-(trifluoromethyl)benzene **1k** (80 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **13** (114 mg, 79%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1k**) = 0.90, R_f (**2a**) = 0.95, R_f (**13**) = 0.20, UV detection].

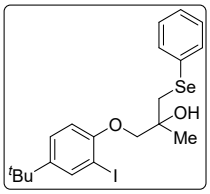
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3437, 3064, 2932, 1729, 1611, 1519, 1461, 1322, 1252, 1113, 1038, 835, 745 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.54 – 7.47 (m, 4H), 7.19 – 7.09 (m, 3H), 6.83 (d, J = 8.6 Hz, 2H), 3.91 (d, J = 8.9 Hz, 1H), 3.85 (d, J = 8.9 Hz, 1H), 3.37 (d, J = 12.9 Hz, 1H), 3.21 (d, J = 12.9 Hz, 1H), 2.75 (br. s, 1H), 1.43 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 160.7, 132.8 (2 × Ar–CH), 130.2, 129.2 (2 × Ar–CH), 127.3, 126.9 (q, J = 3.8 Hz), 124.5 (q, J = 265.6 Hz), 123.3 (q, J = 38.4 Hz), 114.5 (2 × Ar–CH), 73.5, 72.0, 38.8, 24.5 ppm. **^{19}F NMR** (376 MHz, $CDCl_3$) δ = –61.38 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{17}F_3KO_2Se^+$ $[M+K]^+$: 428.9977, found: 428.9991.



1-(4-Ethyl-2-iodophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (14):

GP4 was carried out with 4-ethyl-2-iodo-1-((2-methylallyl)oxy)benzene **1l** (112 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **14** (144 mg, 82%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1l**) = 0.90, R_f (**2a**) = 0.95, R_f (**14**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3443, 3053, 2959, 1583, 1477, 1389, 1259, 1045, 810, 748 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.60 – 7.50 (m, 3H), 7.19 – 7.07 (m, 3H), 7.04 (d, J = 8.2 Hz, 1H), 6.51 (d, J = 8.3 Hz, 1H), 3.91 (d, J = 8.6 Hz, 1H), 3.79 (d, J = 8.6 Hz, 1H), 3.46 (d, J = 12.9 Hz, 1H), 3.32 (d, J = 12.9 Hz, 1H), 2.78 (br. s, 1H), 2.55 (q, J = 7.6 Hz, 2H), 1.46 (s, 3H), 1.20 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 154.5, 139.0, 138.4, 132.6 (2 × Ar–CH), 130.1, 129.1 (2 × Ar–CH), 128.7, 126.9, 111.8, 86.5, 74.2, 72.3, 38.3, 27.6, 24.4, 15.8 ppm. **HRMS:** *m/z* calcd for $C_{18}H_{25}INO_2Se^+$ $[M+NH_4]^+$: 494.0090, found: 494.0111.

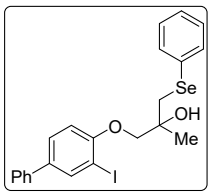


1-(4-(*Tert*-butyl)-2-iodophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (15):

GP4 was carried out with 4-(*tert*-butyl)-2-iodo-1-((2-methylallyl)oxy)benzene **1m** (122 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **15** (147 mg, 79%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1m**) = 0.90, R_f (**2a**) = 0.95, R_f (**15**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3441, 3057, 2955, 1582, 1477, 1259, 1044, 816, 742 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.63 (d, J = 2.3 Hz, 1H), 7.47 – 7.41 (m, 2H), 7.15 – 7.10 (m, 1H), 7.06 – 6.95 (m, 3H), 6.42 (d, J = 8.6 Hz, 1H), 3.81 (d, J = 8.6 Hz, 1H), 3.70 (d,

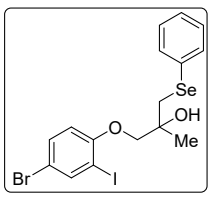
J = 8.6 Hz, 1H), 3.36 (d, J = 12.9 Hz, 1H), 3.23 (d, J = 12.9 Hz, 1H), 2.72 (br. s, 1H), 1.37 (s, 3H), 1.19 (s, 9H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 154.2, 145.9, 136.1, 132.5 (2 × Ar–CH), 130.1, 129.0 (2 × Ar–CH), 126.9, 126.2, 111.4, 86.5, 74.1, 72.2, 38.3, 34.0, 31.4, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{20}H_{29}INO_2Se^+$ $[M+NH_4]^+$: 522.0403, found: 522.0435.



1-((3-Iodo-[1,1'-biphenyl]-4-yl)oxy)-2-methyl-3-(phenylselanyl)propan-2-ol (16):

GP4 was carried out with 3-iodo-4-((2-methylallyl)oxy)-1,1'-biphenyl **1n** (129 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **16** (150 mg, 78%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1n**) = 0.90, R_f (**2a**) = 0.95, R_f (**16**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3442, 3053, 2928, 1588, 1468, 1383, 1270, 1046, 750, 691 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.98 (d, J = 2.2 Hz, 1H), 7.59 – 7.51 (m, 4H), 7.44 (ddd, J = 7.7, 4.4, 2.1 Hz, 3H), 7.38 – 7.32 (m, 1H), 7.18 – 7.04 (m, 3H), 6.62 (d, J = 8.5 Hz, 1H), 3.96 (d, J = 8.6 Hz, 1H), 3.86 (d, J = 8.6 Hz, 1H), 3.50 (d, J = 13.0 Hz, 1H), 3.35 (d, J = 13.0 Hz, 1H), 2.88 (br. s, 1H), 1.51 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 155.9, 139.3, 137.7, 136.2, 132.7 (2 × Ar–CH), 130.02, 130.01, 129.2 (2 × Ar–CH), 128.9 (2 × Ar–CH), 128.1, 127.3, 127.0, 126.8 (2 × Ar–CH), 111.9, 87.1, 74.2, 72.3, 38.4, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{25}INO_2Se^+$ $[M+NH_4]^+$: 542.0090, found: 542.0105.

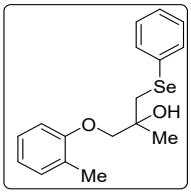


1-(4-Bromo-2-iodophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (17):

GP4 was carried out with 4-bromo-2-iodo-1-((2-methylallyl)oxy)benzene **1o** (131 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **17** (156 mg, 80%), as a yellow liquid [TLC

control (petroleum ether/ethyl acetate 95:05), R_f (**1o**) = 0.90, R_f (**2a**) = 0.95, R_f (**17**) = 0.20, UV detection].

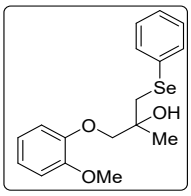
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3445, 3063, 2926, 1571, 1461, 1267, 1040, 747, 684 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.81 (d, J = 2.4 Hz, 1H), 7.54 – 7.49 (m, 2H), 7.29 (dd, J = 8.7, 2.4 Hz, 1H), 7.13 – 7.06 (m, 3H), 6.39 (d, J = 8.7 Hz, 1H), 3.84 (d, J = 8.5 Hz, 1H), 3.75 (d, J = 8.5 Hz, 1H), 3.44 (d, J = 13.0 Hz, 1H), 3.28 (d, J = 13.0 Hz, 1H), 2.76 (br. s, 1H), 1.46 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 155.8, 140.9, 132.8 (2 × Ar–CH), 132.2, 129.8, 129.2 (2 × Ar–CH), 127.2, 113.8, 112.9, 87.3, 74.2, 72.2, 38.4, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{16}Br^{79}IKO_2Se^+$ $[M+K]^+$: 564.8175, found: 564.8194; $C_{16}H_{16}Br^{81}IKO_2Se^+$ $[M+K]^+$: 566.8155, found: 566.8184.



2-Methyl-1-(phenylselanyl)-3-(o-tolyloxy)propan-2-ol (18):

GP4 was carried out with 1-methyl-2-((2-methylallyl)oxy)benzene **1p** (60 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **18** (110 mg, 89%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1p**) = 0.90, R_f (**2a**) = 0.95, R_f (**18**) = 0.20, UV detection].

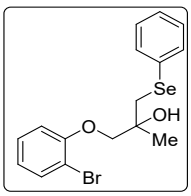
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3444, 3055, 2828, 1589, 1480, 1242, 1120, 1044, 747, 686 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.60 – 7.53 (m, 2H), 7.21 – 7.17 (m, 3H), 7.16 – 7.11 (m, 2H), 6.96 – 6.82 (m, 1H), 6.71 (d, J = 8.3 Hz, 1H), 3.93 (d, J = 8.8 Hz, 1H), 3.87 (d, J = 8.8 Hz, 1H), 3.44 (d, J = 12.8 Hz, 1H), 3.27 (d, J = 12.8 Hz, 1H), 2.78 (br. s, 1H), 2.24 (s, 3H), 1.47 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.3, 132.7 (2 × Ar–CH), 130.7, 130.5, 129.2 (2 × Ar–CH), 127.1, 126.9, 126.5, 120.8, 110.9, 73.4, 72.2, 39.0, 24.6, 16.4 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{20}KO_2Se^+$ $[M+K]^+$: 375.0260, found: 375.0269.



1-(2-Methoxyphenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (19):

GP4 was carried out with 1-methoxy-2-((2-methylallyl)oxy)benzene **1q** (66 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **19** (114 mg, 88%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1q**) = 0.80, R_f (**2a**) = 0.95, R_f (**19**) = 0.10, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3456, 3060, 2932, 1588, 1502, 1454, 1249, 1122, 1030, 746, 684 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.58 – 7.52 (m, 2H), 7.22 – 7.16 (m, 3H), 6.96 (ddd, J = 8.1, 7.3, 1.6 Hz, 1H), 6.90 – 6.85 (m, 2H), 6.81 (dd, J = 7.8, 1.6 Hz, 1H), 4.02 (d, J = 9.1 Hz, 1H), 3.85 (d, J = 9.1 Hz, 1H), 3.83 (s, 3H), 3.39 (d, J = 12.6 Hz, 1H), 3.34 (br. s, 1H), 3.29 (d, J = 12.6 Hz, 1H), 1.41 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 149.9, 148.3, 132.5 (2 × Ar–CH), 130.9, 129.1 (2 × Ar–CH), 126.9, 122.2, 121.0, 115.4, 112.1, 75.8, 72.4, 55.9, 38.2, 24.2 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{20}NaO_3Se^+$ $[M+Na]^+$: 375.0470, found: 375.0482.

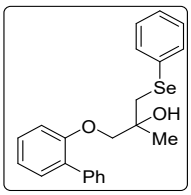


1-(2-Bromophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (20):

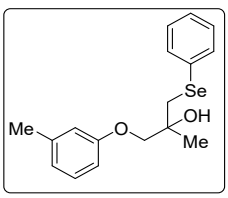
GP4 was carried out with 1-bromo-2-((2-methylallyl)oxy)benzene **1r** (84 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **20** (120 mg, 81%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1r**) = 0.90, R_f (**2a**) = 0.95, R_f (**20**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3433, 3061, 2977, 1581, 1468, 1384, 1259, 1123, 1039, 746, 681 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.57 – 7.46 (m, 3H), 7.21 – 7.05 (m, 4H), 6.82 (td, J = 7.7, 1.4 Hz, 1H), 6.66 (dd, J = 8.2, 1.3 Hz, 1H), 3.92 (d, J = 8.6 Hz, 1H), 3.82 (d, J =

8.6 Hz, 1H), 3.44 (d, J = 12.9 Hz, 1H), 3.30 (d, J = 12.9 Hz, 1H), 2.83 (br. s, 1H), 1.46 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 154.5, 133.2, 132.6 (2 × Ar–CH), 130.1, 129.2 (2 × Ar–CH), 128.5, 127.0, 122.3, 113.2, 112.3, 74.0, 72.3, 38.3, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{17}BrKO_2Se^+$ $[M+K]^+$: 438.9209, found: 438.9230.



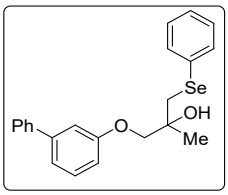
1-([1,1'-Biphenyl]-2-yloxy)-2-methyl-3-(phenylselanyl)propan-2-ol (21):
GP4 was carried out with 2-((2-methylallyl)oxy)-1,1'-biphenyl **1s** (83 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **21** (116 mg, 79%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1s**) = 0.90, R_f (**2a**) = 0.95, R_f (**21**) = 0.20, UV detection].
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3439, 3054, 2928, 1586, 1472, 1241, 1121, 1041, 841, 748 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.48 – 7.40 (m, 4H), 7.37 (t, J = 7.5 Hz, 2H), 7.30 (ddd, J = 7.0, 6.4, 3.5 Hz, 2H), 7.25 (dd, J = 8.3, 2.1 Hz, 1H), 7.18 – 7.12 (m, 3H), 7.03 (t, J = 7.4 Hz, 1H), 6.83 (d, J = 8.2 Hz, 1H), 3.93 (d, J = 8.8 Hz, 1H), 3.79 (d, J = 8.8 Hz, 1H), 3.14 (d, J = 12.7 Hz, 1H), 3.02 (d, J = 12.7 Hz, 1H), 2.43 (br. s, 1H), 1.27 (s, 3H)ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 155.2, 138.4, 132.6 (2 × Ar–CH), 131.3, 130.8, 130.5, 129.6 (2 × Ar–CH), 129.2 (2 × Ar–CH), 128.8, 128.1 (2 × Ar–CH), 127.1, 127.08, 121.5, 112.7, 74.2, 72.3, 38.4, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{22}NaO_2Se^+$ $[M+Na]^+$: 421.0677, found: 421.0699.



2-Methyl-1-(phenylselanyl)-3-(*m*-tolyloxy)propan-2-ol (22):
GP4 was carried out with 1-methyl-3-((2-methylallyl)oxy)benzene **1t** (60 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **22** (103 mg, 83%), as a yellow liquid [TLC

control (petroleum ether/ethyl acetate 95:05), R_f (**1t**) = 0.90, R_f (**2a**) = 0.95, R_f (**22**) = 0.20, UV detection].

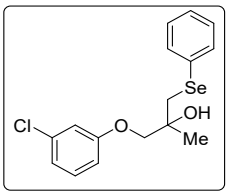
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3437, 3051, 2923, 1591, 1464, 1381, 1262, 1158, 1051, 749, 685 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.55 (ddd, J = 5.2, 4.0, 2.1 Hz, 2H), 7.23 – 7.17 (m, 3H), 7.17 – 7.11 (m, 1H), 6.78 (dd, J = 7.8, 0.6 Hz, 1H), 6.68 – 6.60 (m, 2H), 3.91 (d, J = 8.9 Hz, 1H), 3.83 (d, J = 8.9 Hz, 1H), 3.38 (d, J = 12.7 Hz, 1H), 3.22 (d, J = 12.7 Hz, 1H), 2.75 (br. s, 1H), 2.32 (s, 3H), 1.42 (s, 3H). ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 158.4, 139.6, 132.7 (2 × Ar–CH), 130.7, 129.3, 129.2 (2 × Ar–CH), 127.1, 122.1, 115.5, 111.5, 73.5, 72.1, 38.9, 24.5, 21.6 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{20}KO_2Se^+$ $[M+K]^+$: 375.0260, found: 375.0263.



1-([1,1'-Biphenyl]-3-yloxy)-2-methyl-3-(phenylselanyl)propan-2-ol (23):

GP4 was carried out with 3-((2-methylallyl)oxy)-1,1'-biphenyl **1u** (83 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **23** (117 mg, 80%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1u**) = 0.90, R_f (**2a**) = 0.95, R_f (**23**) = 0.20, UV detection].

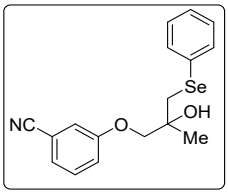
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3424, 3054, 2926, 1587, 1472, 1424, 1286, 1205, 1051, 752, 694 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.60 – 7.53 (m, 4H), 7.45 (t, J = 7.5 Hz, 2H), 7.39 – 7.29 (m, 2H), 7.23 – 7.10 (m, 4H), 7.04 – 7.00 (m, 1H), 6.81 (dd, J = 8.0, 2.3 Hz, 1H), 3.97 (d, J = 8.8 Hz, 1H), 3.89 (d, J = 8.8 Hz, 1H), 3.41 (d, J = 12.8 Hz, 1H), 3.25 (d, J = 12.8 Hz, 1H), 2.76 (br. s, 1H), 1.45 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 158.8, 142.8, 141.0, 132.8 (2 × Ar–CH), 130.6, 129.8, 129.3 (2 × Ar–CH), 128.9 (2 × Ar–CH), 127.6, 127.3 (2 × Ar–CH), 127.2, 120.2, 113.6, 113.4, 73.6, 72.2, 38.9, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{22}KO_2Se^+$ $[M+K]^+$: 437.0417, found: 437.0430.



1-(3-Chlorophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (24):

GP4 was carried out with 1-chloro-3-((2-methylallyl)oxy)benzene **1v** (67 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **24** (107 mg, 82%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1v**) = 0.90, R_f (**2a**) = 0.95, R_f (**24**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3434, 3063, 2929, 1585, 1467, 1231, 1040, 886, 749, 680 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.58 – 7.48 (m, 2H), 7.22 – 7.11 (m, 4H), 6.96 – 6.90 (m, 1H), 6.77 (t, J = 2.2 Hz, 1H), 6.68 (ddd, J = 8.3, 2.4, 0.6 Hz, 1H), 3.86 (d, J = 8.8 Hz, 1H), 3.79 (d, J = 8.8 Hz, 1H), 3.36 (d, J = 12.8 Hz, 1H), 3.20 (d, J = 12.8 Hz, 1H), 2.78 (br. s, 1H), 1.42 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 158.9, 134.8, 132.8 (2 × Ar–CH), 130.3, 130.2, 129.2 (2 × Ar–CH), 127.2, 121.3, 115.0, 112.9, 73.6, 72.0, 38.8, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{17}ClNaO_2Se^+$ $[M+Na]^+$: 378.9975, found: 378.9977.

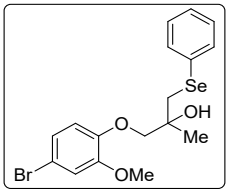


3-(2-Hydroxy-2-methyl-3-(phenylselanyl)propoxy)benzonitrile (25):

GP4 was carried out with 3-((2-methylallyl)oxy)benzonitrile **1w** (64 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **25** (101 mg, 79%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1w**) = 0.85, R_f (**2a**) = 0.95, R_f (**25**) = 0.15, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3467, 3065, 2978, 1586, 1475, 1438, 1266, 1148, 750, 682 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.53 – 7.47 (m, 2H), 7.29 (t, J = 8.0 Hz, 1H), 7.20 (d, J = 7.6 Hz, 1H), 7.17 – 7.09 (m, 3H), 6.99 (ddd, J = 8.4, 2.6, 0.9 Hz, 1H), 6.95 – 6.92 (m, 1H), 3.85 (d, J = 8.8 Hz, 1H), 3.79 (d, J = 8.8 Hz, 1H), 3.33 (d, J = 12.9 Hz, 1H), 3.19 (d, J = 12.9 Hz, 1H), 2.71 (br. s, 1H), 1.42 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 158.2,

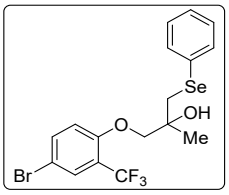
132.9 (2 × Ar–CH), 130.3, 130.1, 129.2 (2 × Ar–CH), 127.3, 124.8, 119.6, 118.6, 117.5, 113.0, 73.5, 71.9, 38.7, 24.4, 14.2 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{17}KNO_2Se^+$ $[M+K]^+$: 386.0056, found: 386.0066.



1-(4-Bromo-2-methoxyphenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (26):

GP4 was carried out with 4-bromo-2-methoxy-1-((2-methylallyl)oxy)benzene **1x** (95 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **26** (135 mg, 85%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1x**) = 0.80, R_f (**2a**) = 0.95, R_f (**26**) = 0.10, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3461, 3063, 2929, 1583, 1494, 1230, 1129, 1024, 912, 743 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.57 – 7.47 (m, 2H), 7.22 – 7.12 (m, 3H), 6.99 – 6.91 (m, 2H), 6.60 (d, J = 9.0 Hz, 1H), 3.93 (d, J = 9.1 Hz, 1H), 3.80 (s, 3H), 3.79 (d, J = 9.1 Hz, 1H), 3.35 (d, J = 12.7 Hz, 1H), 3.25 (d, J = 12.7 Hz, 1H), 3.14 (br. s, 1H), 1.39 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 150.5, 147.4, 132.6 (2 × Ar–CH), 130.6, 129.1 (2 × Ar–CH), 126.9, 123.5, 115.9, 115.3, 113.9, 75.5, 72.3, 56.1, 38.2, 24.2 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{19}Br^{79}NaO_3Se^+$ $[M+Na]^+$: 452.9575, found: 452.9593; $C_{17}H_{19}Br^{81}NaO_3Se^+$ $[M+Na]^+$: 454.9555, found: 454.9578.

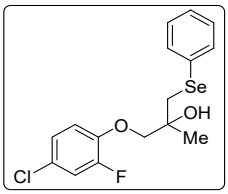


1-(4-Bromo-2-(trifluoromethyl)phenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (27):

GP4 was carried out with 4-bromo-1-((2-methylallyl)oxy)-2-(trifluoromethyl)benzene **1y** (109 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **27** (129 mg, 75%), as a yellow

liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1y**) = 0.90, R_f (**2a**) = 0.95, R_f (**27**) = 0.20, UV detection].

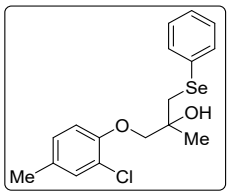
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3447, 3065, 2934, 1594, 1483, 1408, 1259, 1123, 1039, 812, 741, 679 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.64 (d, J = 2.4 Hz, 1H), 7.54 – 7.44 (m, 3H), 7.18 – 7.04 (m, 3H), 6.59 (d, J = 8.8 Hz, 1H), 3.86 (d, J = 8.5 Hz, 1H), 3.81 (d, J = 8.5 Hz, 1H), 3.37 (d, J = 13.1 Hz, 1H), 3.20 (d, J = 13.1 Hz, 1H), 2.70 (br. s, 1H), 1.43 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 155.0, 136.0, 132.7 (2 × Ar–CH), 129.9 (q, J = 5.3 Hz), 129.7, 129.2 (2 × Ar–CH), 127.2, 122.9 (q, J = 272.7 Hz), 120.2 (q, J = 31.1 Hz), 114.2, 112.4, 73.6, 72.0, 38.1, 24.3 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –62.44 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{16}Br^{79}F_3NaO_2Se^+$ $[M+Na]^+$: 490.9343, found: 490.9325; $C_{17}H_{16}Br^{81}F_3NaO_2Se^+$ $[M+Na]^+$: 492.9323, found: 492.9311.



1-(4-Chloro-2-fluorophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (28):

GP4 was carried out with 4-chloro-2-fluoro-1-((2-methylallyl)oxy)benzene **1z** (74 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **28** (99 mg, 72%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1z**) = 0.90, R_f (**2a**) = 0.95, R_f (**28**) = 0.20, UV detection].

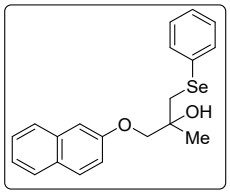
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3405, 3063, 2929, 1584, 1500, 1268, 1205, 1128, 1029, 902, 747 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.55 – 7.48 (m, 2H), 7.19 – 7.10 (m, 3H), 7.05 (dd, J = 10.7, 2.5 Hz, 1H), 6.95 (ddd, J = 8.8, 2.4, 1.7 Hz, H), 6.67 (t, J = 8.8 Hz, 1H), 3.88 (d, J = 8.8 Hz, 1H), 3.81 (d, J = 8.8 Hz, 1H), 3.36 (d, J = 12.9 Hz, 1H), 3.22 (d, J = 12.9 Hz, 1H), 2.83 (br. s, 1H), 1.42 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 152.3 (d, J = 250.2 Hz), 145.4 (d, J = 10.6 Hz), 132.7 (2 × Ar–CH), 130.2, 129.2 (2 × Ar–CH), 127.2, 125.9 (d, J = 9.0 Hz), 124.2 (d, J = 3.5 Hz), 117.0 (d, J = 21.2 Hz), 115.6 (d, J = 1.3 Hz), 74.7, 72.1, 38.4, 24.4, 24.1 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –131.17 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{16}ClFO_2Se^+$ $[M^+]^+$: 373.9983, found: 373.9977.



1-(2-Chloro-4-methylphenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (29):

GP4 was carried out with 2-chloro-4-methyl-1-((2-methylallyl)oxy)benzene **1za** (73 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **29** (110 mg, 80%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1za**) = 0.90, R_f (**2a**) = 0.95, R_f (**29**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3389, 3062, 2929, 1603, 1490, 1386, 1244, 1130, 1045, 750, 687 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.56 – 7.49 (m, 2H), 7.22 – 7.12 (m, 3H), 7.06 (d, J = 8.5 Hz, 1H), 6.78 (d, J = 2.6 Hz, 1H), 6.62 (dd, J = 8.4, 2.6 Hz, 1H), 3.84 (d, J = 8.9 Hz, 1H), 3.77 (d, J = 8.9 Hz, 1H), 3.35 (d, J = 12.8 Hz, 1H), 3.20 (d, J = 12.8 Hz, 1H), 2.82 (br. s, 1H), 2.28 (s, 3H), 1.40 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 157.1, 134.6, 132.8 (2 × Ar–CH), 131.2, 130.5, 129.2 (2 × Ar–CH), 128.5, 127.2, 115.3, 113.2, 73.8, 72.1, 38.8, 24.5, 19.2 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{19}ClO_2Se^+$ $[M^+]^+$: 370.0233, found: 370.0241.

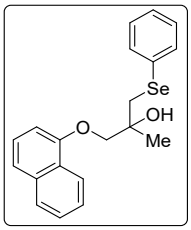


2-Methyl-1-(naphthalen-2-yloxy)-3-(phenylselanyl)propan-2-ol (30):

GP4 was carried out with 2-((2-methylallyl)oxy)naphthalene **1zb** (73 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **30** (117 mg, 86%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zb**) = 0.90, R_f (**2a**) = 0.95, R_f (**30**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3440, 3055, 2975, 1713, 1614, 1459, 1259, 1211, 1037, 831, 741, 684 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.78 (d, J = 8.2 Hz, 1H), 7.72 (t, J = 9.5 Hz, 2H), 7.61 – 7.53 (m, 2H), 7.46 (ddd, J = 8.2, 6.9, 1.3 Hz, 1H), 7.37 (ddd, J = 8.1, 6.9, 1.2 Hz, 1H), 7.21 – 7.09 (m, 4H), 7.02 (d, J = 2.4 Hz, 1H), 4.04 (d, J = 8.9 Hz, 1H), 3.97 (d, J =

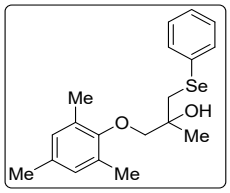
8.9 Hz, 1H), 3.44 (d, J = 12.8 Hz, 1H), 3.28 (d, J = 12.8 Hz, 1H), 2.84 (br. s, 1H), 1.49 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.3, 134.5, 132.8 (2 × Ar–CH), 130.6, 129.4, 129.2 (2 × Ar–CH, 1 × Ar–C), 127.7, 127.2, 126.9, 126.5, 123.9, 118.7, 107.0, 73.6, 72.1, 38.9, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{20}H_{20}KO_2Se^+$ $[M+K]^+$: 411.0260, found: 411.0270.



2-Methyl-1-(naphthalen-1-yloxy)-3-(phenylselanyl)propan-2-ol (31):

GP4 was carried out with 1-((2-methylallyl)oxy)naphthalene **1zc** (73 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **31** (116 mg, 85%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zc**) = 0.90, R_f (**2a**) = 0.95, R_f (**31**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3440, 3055, 2983, 1582, 1459, 1395, 1266, 1100, 1014, 755 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.20 (d, J = 7.4 Hz, 1H), 7.84 – 7.79 (m, 1H), 7.57 – 7.46 (m, 4H), 7.44 (d, J = 8.3 Hz, 1H), 7.32 (t, J = 7.9 Hz, 1H), 7.12 – 6.99 (m, 3H), 6.67 (d, J = 7.6 Hz, 1H), 4.07 (d, J = 8.9 Hz, 1H), 4.02 (d, J = 8.9 Hz, 1H), 3.54 (d, J = 12.9 Hz, 1H), 3.35 (d, J = 12.9 Hz, 1H), 2.81 (br. s, 1H), 1.55 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 153.9, 134.5, 132.7 (2 × Ar–CH), 130.3, 129.2 (2 × Ar–CH), 127.7, 127.1, 126.5, 125.9, 125.5, 125.4, 121.8, 120.8, 104.9, 73.6, 72.2, 39.3, 24.9 ppm. **HRMS:** *m/z* calcd for $C_{20}H_{20}KO_2Se^+$ $[M+K]^+$: 411.0260, found: 411.0268.

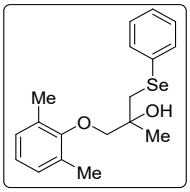


1-(Mesityloxy)-2-methyl-3-(phenylselanyl)propan-2-ol (32):

GP4 was carried out with 1,3,5-trimethyl-2-((2-methylallyl)oxy)benzene **1zd** (71 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum

ether/ethyl acetate, 95:05) furnished the product **32** (118 mg, 87%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zd**) = 0.90, R_f (**2a**) = 0.95, R_f (**32**) = 0.20, UV detection].

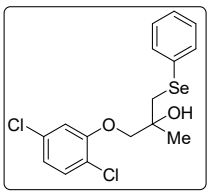
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3456, 2924, 1579, 1472, 1379, 1211, 1143, 1025, 850, 740 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.62 – 7.50 (m, 2H), 7.30 – 7.18 (m, 3H), 6.81 (s, 2H), 3.77 (d, J = 8.9 Hz, 1H), 3.64 (d, J = 8.9 Hz, 1H), 3.44 (d, J = 12.4 Hz, 1H), 3.27 (d, J = 12.4 Hz, 1H), 2.85 (br. s, 1H), 2.23 (s, 9H), 1.42 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 152.5, 133.6, 132.6 (2 × Ar–CH), 131.2, 130.5 (2 × Ar–C), 129.7 (2 × Ar–CH), 129.3 (2 × Ar–CH), 127.1, 77.3, 72.4, 38.9, 24.4, 20.8, 16.4 ppm. **HRMS:** *m/z* calcd for $C_{19}H_{24}KO_2Se^+$ $[M+]^+$: 403.0573, found: 403.0581.



1-(2,6-Dimethylphenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (33):

GP4 was carried out with 1,3-dimethyl-2-((2-methylallyl)oxy)benzene **1ze** (65 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **33** (113 mg, 88%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1ze**) = 0.90, R_f (**2a**) = 0.95, R_f (**33**) = 0.20, UV detection].

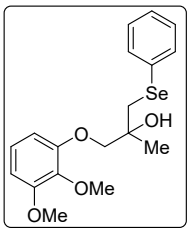
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3453, 2926, 1580, 1468, 1266, 1198, 1092, 1025, 751, 687 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.56 (ddd, J = 8.8, 4.8, 2.4 Hz, 2H), 7.28 – 7.19 (m, 3H), 7.00 (d, J = 7.5 Hz, 2H), 6.92 (dd, J = 8.3, 6.5 Hz, 1H), 3.80 (d, J = 8.9 Hz, 1H), 3.67 (d, J = 8.9 Hz, 1H), 3.45 (d, J = 12.4 Hz, 1H), 3.28 (d, J = 12.4 Hz, 1H), 2.86 (br. s, 1H), 2.27 (s, 6H), 1.43 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 154.7, 132.6 (2 × Ar–CH), 131.1, 130.9 (2 × Ar–C), 129.3 (2 × Ar–CH), 129.1 (2 × Ar–CH), 127.1, 124.3, 72.4, 38.9, 24.4, 16.5 ppm. **HRMS:** *m/z* calcd for $C_{18}H_{22}NaO_2Se^+$ $[M+Na]^+$: 373.0677, found: 373.0692.



1-(2,5-Dichlorophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (34):

GP4 was carried out with 1,4-dichloro-2-((2-methylallyl)oxy)benzene **1zf** (80 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **34** (123 mg, 86%), as a yellow solid, mp = 62–64 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zf**) = 0.90, R_f (**2a**) = 0.95, R_f (**34**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3437, 3064, 2932, 1579, 1468, 1393, 1256, 1033, 900, 811, 738, 683 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.57 – 7.48 (m, 2H), 7.21 (d, J = 8.4 Hz, 1H), 7.15 – 7.03 (m, 3H), 6.85 (dd, J = 8.4, 2.3 Hz, 1H), 6.60 (d, J = 2.3 Hz, 1H), 3.84 (d, J = 8.6 Hz, 1H), 3.76 (d, J = 8.6 Hz, 1H), 3.40 (d, J = 13.1 Hz, 1H), 3.24 (d, J = 13.1 Hz, 1H), 2.81 (br. s, 1H), 1.45 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 153.9, 133.0, 132.8 (2 × Ar–CH), 130.6, 129.8, 129.2 (2 × Ar–CH), 127.2, 121.6, 121.3, 113.8, 73.9, 72.2, 38.3, 24.3 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{16}Cl_2KO_2Se^+$ $[M+K]^+$: 428.9324, found: 428.9338.

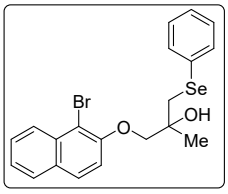


1-(2,3-Dimethoxyphenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (35):

GP4 was carried out with 1,2-dimethoxy-3-((2-methylallyl)oxy)benzene **1zg** (77 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the product **35** (132 mg, 94%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**1zg**) = 0.50, R_f (**2a**) = 0.95, R_f (**35**) = 0.10, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3420, 2934, 1592, 1471, 1376, 1250, 1176, 1101, 914, 742 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.61 – 7.52 (m, 2H), 7.25 – 7.15 (m, 3H), 6.95 (t, J = 8.4 Hz, 1H), 6.60 (dd, J = 8.4, 1.0 Hz, 1H), 6.48 (dd, J = 8.4, 1.1 Hz, 1H), 4.00 (d, J = 9.1

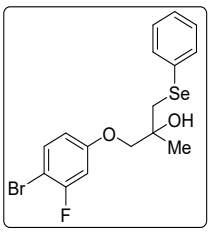
Hz, 1H), 3.88 (d, J = 9.1 Hz, 1H), 3.87(s, 3H), 3.82 (s, 3H), 3.41 (d, J = 12.6 Hz, 1H), 3.28 (d, J = 12.6 Hz, 1H), 3.11 (br. s, 1H), 1.43 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 153.5, 152.4, 138.5, 132.4 (2 × Ar–CH), 130.5, 128.9 (2 × Ar–CH), 126.8, 123.6, 107.3, 105.7, 75.1, 71.9, 60.8, 55.9, 38.3, 24.1 ppm. **HRMS:** *m/z* calcd for $C_{18}H_{26}NO_4Se^+$ $[M+NH_4]^+$: 400.1022, found: 400.1047.



1-((1-Bromonaphthalen-2-yl)oxy)-2-methyl-3-(phenylselanyl)propan-2-ol (36):

GP4 was carried out with 1-bromo-2-((2-methylallyl)oxy)naphthalene **1zh** (102 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **36** (131 mg, 79%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zh**) = 0.90, R_f (**2a**) = 0.95, R_f (**36**) = 0.20, UV detection].

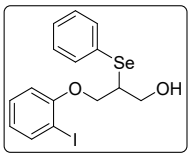
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3622, 3162, 3002, 2623, 1631, 1435, 1383, 1039, 918, 750 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.20 (dd, J = 8.5, 0.6 Hz, 1H), 7.77 (d, J = 8.1 Hz, 1H), 7.71 (d, J = 8.9 Hz, 1H), 7.61 – 7.57 (m, 1H), 7.56 – 7.52 (m, 2H), 7.41 (ddd, J = 8.0, 6.9, 1.1 Hz, 1H), 7.12 – 7.04 (m, 2H), 7.04 – 6.97 (m, 2H), 4.10 (d, J = 8.6 Hz, 1H), 3.98 (d, J = 8.6 Hz, 1H), 3.50 (d, J = 12.9 Hz, 1H), 3.36 (d, J = 12.9 Hz, 1H), 2.98 (br. s, 1H), 1.51 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 152.5, 133.0, 132.6 (2 × Ar–CH), 130.2, 130.1, 129.1 (2 × Ar–CH), 128.9, 128.1, 127.8, 127.0, 126.2, 124.6, 114.8, 109.5, 75.1, 72.5, 38.3, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{20}H_{19}Br^{79}KO_2Se^+$ $[M+K]^+$: 488.9365, found: 488.9402; $C_{20}H_{19}Br^{81}KO_2Se^+$ $[M+K]^+$: 490.9345, found 490.9394.



1-(4-Bromo-3-fluorophenoxy)-2-methyl-3-(phenylselanyl)propan-2-ol (37):

GP4 was carried out with 1-bromo-2-fluoro-4-((2-methylallyl)oxy)benzene **1zi** (91 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **37** (121 mg, 78%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zi**) = 0.90, R_f (**2a**) = 0.95, R_f (**37**) = 0.20, UV detection].

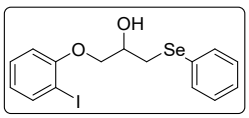
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3435, 3064, 2930, 1592, 1477, 1284, 1161, 1029, 744, 685 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.56 – 7.45 (m, 2H), 7.35 (dd, J = 8.7, 8.1 Hz, 1H), 7.21 – 7.09 (m, 3H), 6.54 (dd, J = 10.3, 2.8 Hz, 1H), 6.48 (ddd, J = 8.9, 2.8, 1.0 Hz, 1H), 3.82 (d, J = 8.8 Hz, 1H), 3.76 (d, J = 8.8 Hz, 1H), 3.33 (d, J = 12.9 Hz, 1H), 3.18 (d, J = 12.9 Hz, 1H), 2.73 (br. s, 1H), 1.41 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 159.4 (d, J = 247.1 Hz), 158.8 (d, J = 9.8 Hz), 133.3 (d, J = 1.7 Hz), 132.9 (2 × Ar–CH), 130.2, 129.3 (2 × Ar–CH), 127.3, 111.8 (d, J = 3.1 Hz), 103.6 (d, J = 25.9 Hz), 99.8 (d, J = 21.4 Hz), 73.9, 71.9, 38.8, 24.5 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –105.21 ppm. **HRMS:** *m/z* calcd for $C_{16}H_{16}Br^{79}FO_2Se^+$ $[M^+]^+$: 417.9477, found: 417.9487; $C_{16}H_{16}Br^{81}FO_2Se^+$ $[M^+]^+$: 419.9457, found 419.9471.



3-(2-Iodophenoxy)-2-(phenylselanyl)propan-1-ol (38):

GP4 was carried out with 1-(allyloxy)-2-iodobenzene **1zj** (96 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **38** (64 mg, 40%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zj**) = 0.90, R_f (**2a**) = 0.95, R_f (**38**) = 0.10, UV detection].

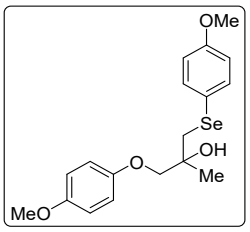
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3395, 3060, 2926, 1578, 1464, 1381, 1263, 1008, 829, 750, 689 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.70 (dd, J = 7.8, 1.5 Hz, 1H), 7.58 (dd, J = 7.3, 1.6 Hz, 2H), 7.30 – 7.15 (m, 4H), 6.73 – 6.62 (m, 2H), 4.35 (dd, J = 9.5, 4.3 Hz, 1H), 4.16 (dd, J = 9.0, 9.0 Hz, 1H), 4.08 – 3.94 (m, 2H), 3.69 – 3.59 (m, 1H), 2.46 (br. s, 1H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.8, 139.6, 135.2 (2 × Ar–CH), 129.6, 129.5 (2 × Ar–CH), 128.3, 127.6, 123.2, 112.2, 86.6, 70.0, 63.6, 46.1 ppm. **HRMS:** *m/z* calcd for $C_{15}H_{15}INaO_2Se^+$ $[M+Na]^+$: 456.9174, found: 456.9177.



1-(2-Iodophenoxy)-3-(phenylselanyl)propan-2-ol (38'):

GP4 was carried out with 1-(allyloxy)-2-iodobenzene **1zj** (96 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **38'** (48 mg, 30%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zj**) = 0.90, R_f (**2a**) = 0.95, R_f (**38'**) = 0.15, UV detection].

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.75 (dd, J = 7.6, 1.4 Hz, 1H), 7.56 (dd, J = 7.4, 2.0 Hz, 2H), 7.31 – 7.19 (m, 4H), 6.73 (dd, J = 7.9, 1.3 Hz, 2H), 4.19 – 4.04 (m, 1H), 4.12 – 4.03 (m, 2H), 3.35 (dd, J = 13.0, 6.0 Hz, 1H), 3.22 (dd, J = 13.0, 6.6 Hz, 1H), 1.60 (s, 1H). **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 156.7, 139.4, 132.8 (2 × Ar–CH), 129.7, 129.4 (2 × Ar–CH), 129.3, 127.4, 123.2, 112.4, 86.8, 71.4, 69.3, 31.7.

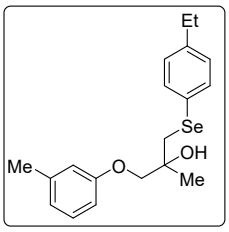


1-(4-Methoxyphenoxy)-3-((4-methoxyphenyl)selanyl)-2-methylpropan-2-ol (39):

GP4 was carried out with 1-methoxy-4-((2-methylallyl)oxy)benzene **1e** (66 mg, 0.37 mmol), 1,2-bis(4-methoxyphenyl)diselane **2b** (110 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the product **39** (113 mg, 80%), as a yellow

liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1e**) = 0.80, R_f (**2b**) = 0.80, R_f (**39**) = 0.10, UV detection].

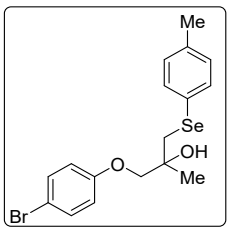
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3465, 2930, 2836, 1587, 1499, 1456, 1226, 1035, 820, 748 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.50 – 7.44 (m, 2H), 6.81 – 6.76 (m, 2H), 6.74 – 6.69 (m, 4H), 3.83 (d, J = 8.8 Hz, 1H), 3.75 (d, J = 8.8 Hz, 1H), 3.76 (s, 3H), 3.72 (s, 3H), 3.28 (d, J = 12.7 Hz, 1H), 3.13 (d, J = 12.7 Hz, 1H), 2.78 (br. s, 1H), 1.38 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 159.3, 154.1, 152.6, 135.3 (2 × Ar–CH), 120.4, 115.5 (2 × Ar–CH), 114.9 (2 × Ar–CH), 114.6 (2 × Ar–CH), 74.1, 72.2, 55.8, 55.2, 39.9, 24.4 ppm. **HRMS:** *m/z* calcd for $C_{18}H_{22}NaO_4Se^+$ $[M+Na]^+$: 405.0576, found: 405.0586.



1-((4-Ethylphenyl)selanyl)-2-methyl-3-(*m*-tolyloxy)propan-2-ol (42):

GP4 was carried out with 1-methyl-3-((2-methylallyl)oxy)benzene **1t** (60 mg, 0.37 mmol), 1,2-bis(4-ethylphenyl)diselane **2c** (109 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **42** (113 mg, 84%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1t**) = 0.90, R_f (**2c**) = 0.95, R_f (**42**) = 0.20, UV detection].

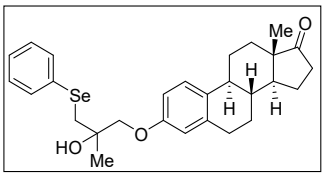
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.51 – 7.44 (m, 2H), 7.15 (t, J = 7.8 Hz, 1H), 7.04 (d, J = 8.2 Hz, 2H), 6.78 (d, J = 7.5 Hz, 1H), 6.69 – 6.59 (m, 2H), 3.89 (d, J = 8.9 Hz, 1H), 3.82 (d, J = 8.9 Hz, 1H), 3.37 (d, J = 12.7 Hz, 1H), 3.20 (d, J = 12.7 Hz, 1H), 2.80 (br. s, 1H), 2.58 (q, J = 7.6 Hz, 2H), 2.33 (s, 3H), 1.43 (s, 3H), 1.20 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 158.4, 143.5, 139.5, 133.2 (2 × Ar–CH), 129.2, 128.8 (2 × Ar–CH), 127.0, 121.9, 115.4, 111.5, 73.5, 72.1, 39.2, 28.5, 24.4, 21.6, 15.5 ppm. **HRMS:** *m/z* calcd for $C_{19}H_{24}NaO_2Se^+$ $[M+Na]^+$: 387.0834, found: 387.0830.



1-(4-Bromophenoxy)-2-methyl-3-(p-tolylselanyl)propan-2-ol (43):

GP4 was carried out with 1-bromo-4-((2-methylallyl)oxy)benzene **1g** (84 mg, 0.37 mmol), 1,2-di-*p*-tolyldiselane **2d** (101 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **43** (122 mg, 80%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1g**) = 0.90, R_f (**2d**) = 0.95, R_f (**43**) = 0.20, UV detection].

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 – 7.37 (m, 2H), 7.34 – 7.28 (m, 2H), 6.94 (d, J = 8.0 Hz, 2H), 6.65 – 6.58 (m, 2H), 3.80 (d, J = 8.8 Hz, 1H), 3.73 (d, J = 8.8 Hz, 1H), 3.31 (d, J = 12.9 Hz, 1H), 3.15 (d, J = 12.9 Hz, 1H), 2.73 (br. s, 1H), 2.23 (s, 3H), 1.40 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 157.4, 137.4, 133.2 (2 × Ar–CH), 132.1 (2 × Ar–CH), 130.0 (2 × Ar–CH), 126.3, 116.3 (2 × Ar–CH), 113.2, 73.4, 72.1, 39.1, 24.4, 21.1 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{19}Br^{79}KO_2Se^+$ $[M+K]^+$: 452.9365, found 452.9381. $C_{17}H_{19}Br^{81}KO_2Se^+$ $[M+K]^+$: 454.9345, found: 454.9368.

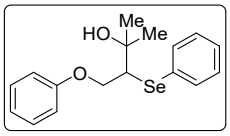


(8*R*,9*S*,13*S*,14*S*)-3-(2-Hydroxy-2-methyl-3-(phenylselanyl)propoxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (44):

GP4 was carried out with (8*R*,9*S*,13*S*,14*S*)-13-methyl-3-((2-methylallyl)oxy)-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one **1zm** (120 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the product **44** (156 mg, 85%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**1zm**) = 0.60, R_f (**2a**) = 0.95, R_f (**44**) = 0.20, UV detection].

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.44 (d, J = 4.3 Hz, 2H), 7.22 – 6.95 (m, 4H), 6.65 – 6.41 (m, 2H), 3.80 (dd, J = 8.7, 2.3 Hz, 1H), 3.74 (dd, J = 8.5, 2.0, 1H), 3.26 (d, J = 12.6 Hz, 1H), 3.12

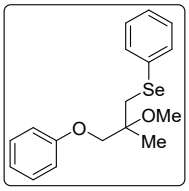
(d, J = 12.6 Hz, 1H), 2.77 (s, 2H), 2.40 (dd, J = 18.8, 8.5 Hz, 1H), 2.28 (d, J = 8.3 Hz, 1H), 2.24 – 1.66 (m, 6H), 1.59 – 1.34 (m, 6H), 1.31 (s, 3H), 0.81 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 221.0, 156.4, 137.7, 132.6 (2 × Ar–CH), 132.5, 130.8, 129.1 (2 × Ar–CH), 126.9, 126.3, 114.6, 112.2, 73.7, 72.0, 50.3, 48.0, 43.9, 38.7, 38.3, 35.9, 31.6, 29.6, 26.5, 25.9, 24.4, 21.6, 13.8 ppm. **HRMS:** *m/z* calcd for $C_{28}H_{34}KO_3Se^+$ $[M+K]^+$: 537.1305, found: 537.1330.



2-Methyl-4-phenoxy-3-(phenylselanyl)butan-2-ol (45):

GP4 was carried out with ((3-methylbut-2-en-1-yl)oxy)benzene **1zn** (60 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **45** (109 mg, 88%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zn**) = 0.90, R_f (**2a**) = 0.95, R_f (**45**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3435, 2973, 1736, 1590, 1485, 1229, 746, 689 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 7.62 (dd, J = 7.7, 1.8 Hz, 2H), 7.29 – 7.22 (m, 5H), 6.96 – 6.92 (m, 1H), 6.82 (dd, J = 8.7, 0.9 Hz, 2H), 4.36 (dd, J = 6.0, 2.1 Hz, 2H), 3.49 (dd, J = 6.5, 5.3 Hz, 1H), 2.99 (s, 1H), 1.44 (s, 3H), 1.42 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 158.0, 134.7 (2 × Ar–CH), 129.6 (2 × Ar–CH), 129.4 (2 × Ar–CH), 127.9, 121.5, 115.5, 114.7 (2 × Ar–CH), 73.1, 69.7, 57.9, 28.8, 27.7 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{20}NaO_2Se^+$ $[M+Na]^+$: 359.0521, found 359.0517.

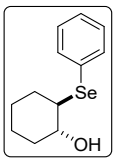


(2-Methoxy-2-methyl-3-phenoxypropyl)(phenyl)selane (46):

GP4 was carried out with ((2-methylallyl)oxy)benzene **1a** (55 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **46** (112 mg, 90%), as a yellow liquid [TLC control

(petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.90, R_f (**2a**) = 0.95, R_f (**46**) = 0.30, UV detection].

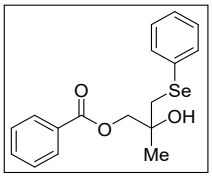
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2931, 1591, 1237, 1081, 747, 689 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 7.51 (dd, J = 7.2, 2.3 Hz, 2H), 7.28 – 7.23 (m, 2H), 7.21 – 7.14 (m, 3H), 6.95 – 6.92 (m, 1H), 6.86 (dd, J = 8.7, 0.9 Hz, 2H), 4.07 (d, J = 9.2 Hz, 1H), 3.90 (d, J = 9.2 Hz, 1H), 3.35 (d, J = 12.4 Hz, 1H), 3.30 (d, J = 12.4 Hz, 1H), 3.299 (s, 3H), 1.41 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 158.7, 132.9 (2 × Ar–CH), 130.9, 129.5 (2 × Ar–CH), 129.1 (2 × Ar–CH), 126.9, 121.0, 114.7 (2 × Ar–CH), 76.7, 71.2, 50.3, 35.1, 20.7 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{19}OSe^+$ [(M+H) + (-H_2O)]: 319.0595, found 319.0609.



(1*R*,2*R*)-2-(Phenylselanyl)cyclohexan-1-ol (47):[6]

GP4 was carried out with cyclohexene **1zo** (31 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **47** (89 mg, 94%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1zo**) = 0.90, R_f (**2a**) = 0.95, R_f (**47**) = 0.20, UV detection].

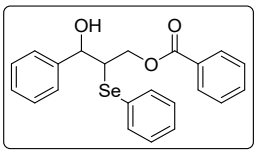
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.62 – 7.57 (m, 2H), 7.35 – 7.24 (m, 3H), 3.37 – 3.26 (m, 1H), 3.00 (s, 1H), 2.90 (ddd, J = 12.3, 10.0, 4.0 Hz, 1H), 2.20 – 2.10 (m, 2H), 1.72 (ddd, J = 6.2, 5.1, 2.9 Hz, 1H), 1.65 – 1.57 (m, 1H), 1.41 (ddd, J = 15.8, 12.5, 3.6 Hz, 1H), 1.34 – 1.18 (m, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 136.2 (2 × Ar–CH), 129.1 (2 × Ar–CH), 128.2, 126.7, 72.3, 53.5, 33.9, 33.4, 26.9, 24.5 ppm.



2-Hydroxy-2-methyl-3-(phenylselanyl)propyl benzoate (50):

GP4 was carried out with 2-methylallyl benzoate **48a** (65 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **50** (98 mg, 76%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**48a**) = 0.70, R_f (**2a**) = 0.95, R_f (**50**) = 0.20, UV detection].

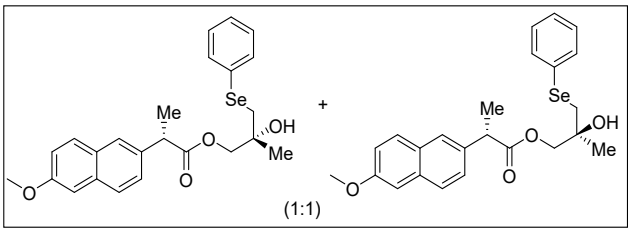
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3469, 3062, 2972, 1712, 1589, 1268, 1109, 1023, 813, 706 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.01 – 7.95 (m, 2H), 7.60 – 7.54 (m, 3H), 7.46 – 7.41 (m, 2H), 7.23 – 7.17 (m, 3H), 4.31 (d, *J* = 11.2 Hz, 1H), 4.27 (d, *J* = 11.2 Hz, 1H), 3.34 (d, *J* = 12.9 Hz, 1H), 3.18 (d, *J* = 12.9 Hz, 1H), 2.66 (br. s, 1H), 1.41 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 166.3, 133.3, 133.0 (2 × Ar–CH), 130.5, 129.8 (2 × Ar–CH), 129.4 (2 × Ar–CH), 128.5 (2 × Ar–CH), 127.4, 113.1, 71.8, 70.3, 39.5, 24.8 ppm. **HRMS:** *m/z* calcd for $C_{17}H_{18}NaO_3Se^+$ $[M+Na]^+$: 373.0313, found: 373.0322.



3-Hydroxy-3-phenyl-2-(phenylselanyl)propyl benzoate (51):

GP4 was carried out with cinnamyl benzoate **48b** (88 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **51** (128 mg, 84%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**48b**) = 0.60, R_f (**2a**) = 0.95, R_f (**51**) = 0.15, UV detection].

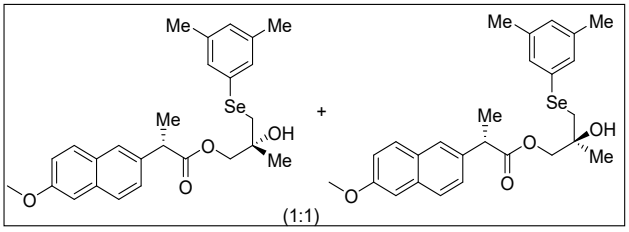
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3466, 3061, 1708, 1447, 1264, 1108, 749, 701 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.89 (dd, *J* = 8.2, 1.2 Hz, 2H), 7.56 – 7.50 (m, 3H), 7.38 (dd, *J* = 10.8, 4.7 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.31 – 7.20 (m, 6H), 5.05 (d, *J* = 4.4 Hz, 1H), 4.71 (dd, *J* = 11.8, 5.7 Hz, 1H), 4.47 (dd, *J* = 11.8, 6.6 Hz, 1H), 3.86 (dd, *J* = 11.8, 5.6 Hz, 1H), 3.06 (s, 1H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 166.4, 140.8, 134.9 (2 × Ar–CH), 133.2, 129.8 (2 × Ar–CH), 129.4 (2 × Ar–CH), 128.5 (2 × Ar–CH), 128.45 (2 × Ar–C), 128.43 (2 × Ar–CH), 128.1, 128.04, 126.3 (2 × Ar–CH), 73.72, 64.30, 52.11ppm. **HRMS:** *m/z* calcd for $C_{22}H_{20}KO_3Se^+$ $[(M+K)]^+$: 451.0209, found 451.0206.



(*R*)-2-Hydroxy-2-methyl-3-(phenylselanyl)propyl (*S*)-2-(6-methoxynaphthalen-2-yl)propanoate (52) and (*S*)-2-hydroxy-2-methyl-3-(phenylselanyl)propyl (*S*)-2-(6-methoxynaphthalen-2-yl)propanoate (52'):

GP4 was carried out with 2-methylallyl (*S*)-2-(6-methoxynaphthalen-2-yl)propanoate **48c** (105 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the products [**52 + 52'**] (150 mg, 89%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**48c**) = 0.60, R_f (**2a**) = 0.95, R_f (**52 + 52'**) = 0.10, UV detection].

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.70 (dd, J = 8.7, 2.3 Hz, 2H), 7.66 (s, 1H), 7.45 (d, J = 7.5 Hz, 2H), 7.38 (d, J = 8.4 Hz, 1H), 7.25 – 7.14 (m, 4H), 7.12 (s, 1H), 4.16 – 3.98 (m, 2H), 3.91 (s, 3H), 3.85 (q, J = 7.1 Hz, 1H), 3.05 (t, J = 12.4 Hz, 1H), 2.93 (dd, J = 12.7, 3.8 Hz, 1H), 2.40 (br. s, 1H), 1.58 (d, J = 7.2 Hz, 3H), 1.18 (d, J = 8.4 Hz, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 174.31, 174.29, 157.7, 135.41, 135.37, 133.8, 132.74, 132.71, 130.48, 130.46, 129.3, 129.2 (2 × Ar–CH), 128.9, 127.30, 127.28, 127.22, 127.20, 126.2, 126.04, 126.03, 119.16, 119.15, 105.7, 71.67, 71.60, 69.96, 69.92, 55.3, 45.4, 38.8, 24.4, 24.3, 18.3 ppm. **HRMS:** *m/z* calcd for $C_{24}H_{26}KO_4Se^+$ $[M+K]^+$: 497.0628, found: 497.0642.

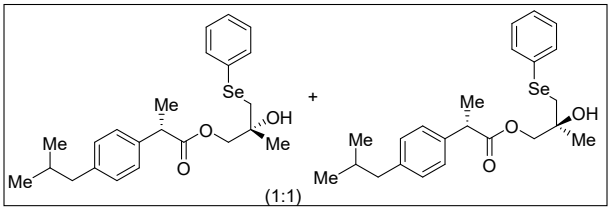


(*R*)-3-((3,5-Dimethylphenyl)selanyl)-2-hydroxy-2-methylpropyl (*S*)-2-(6-methoxynaphthalen-2-yl)propanoate (53) and (*S*)-3-((3,5-dimethylphenyl)selanyl)-2-hydroxy-2-methylpropyl (*S*)-2-(6-methoxynaphthalen-2-yl)propanoate (53'):

GP4 was carried out with 2-methylallyl (*S*)-2-(6-methoxynaphthalen-2-yl)propanoate **48c** (105 mg, 0.37 mmol), 1,2-bis(3,5-dimethylphenyl)diselane **2e** (109 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the products [**53 + 53'**] (156 mg, 84%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**48c**) = 0.60, R_f (**2e**) = 0.95, R_f (**53 + 53'**) = 0.10, UV detection].

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.70 (dd, J = 8.7, 2.4 Hz, 2H), 7.66 (s, 1H), 7.39 (d, J = 8.4 Hz, 1H), 7.18 – 7.10 (m, 4H), 6.87 (s, 1H), 4.16 – 4.01 (m, 2H), 3.91 (s, 3H), 3.85 (q, J = 7.1 Hz, 1H), 3.07 (dd, J = 17.1, 12.7 Hz, 1H), 2.94 (dd, J = 12.7, 4.3 Hz, 1H), 2.47 (br. s, 1H), 2.28

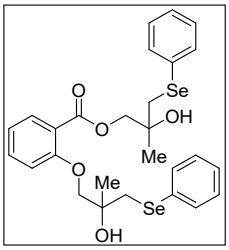
(s, 6H), 1.59 (d, J = 7.2 Hz, 3H), 1.18 (d, J = 8.9 Hz, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 174.3, 157.7, 138.8, 135.44, 135.39, 133.8, 130.5, 130.4, 130.1, 130.0, 129.3, 129.2, 129.1, 128.9, 127.29, 127.26, 126.22, 126.20, 126.0, 119.14, 119.12, 105.6, 71.7, 71.6, 70.06, 70.00, 55.3, 45.4, 38.8, 24.4, 24.3, 21.2, 18.3 ppm. **HRMS:** *m/z* calcd for $C_{26}H_{30}NaO_4Se^+$ $[M+Na]^+$: 509.1202, found: 509.1199.



(*R*)-2-Hydroxy-2-methyl-3-(phenylselanyl)propyl (*S*)-2-(4-isobutylphenyl)propanoate (54) and (*S*)-2-hydroxy-2-methyl-3-(phenylselanyl)propyl (*S*)-2-(4-isobutylphenyl)propanoate (54'):

GP4 was carried out with 2-methylallyl 2-(4-isobutylphenyl)propanoate **48d** (96 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL), for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the products [**54 + 54'**] (139 mg, 87%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**48d**) = 0.60, R_f (**2a**) = 0.95, R_f (**54 + 54'**) = 0.20, UV detection].

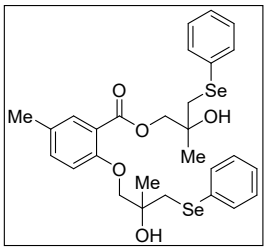
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 – 7.36 (m, 2H), 7.14 (ddd, J = 4.0, 2.8, 1.3 Hz, 3H), 7.08 (d, J = 8.0 Hz, 2H), 6.99 (dd, J = 8.1, 2.9 Hz, 2H), 4.06 – 3.83 (m, 2H), 3.58 (q, J = 7.2 Hz, 1H), 2.95 (t, J = 12.7 Hz, 1H), 2.82 (dd, J = 12.8, 4.3 Hz, 1H), 2.35 (d, J = 7.2 Hz, 3H), 1.81 – 1.67 (m, 1H), 1.39 (d, J = 7.2 Hz, 3H), 1.07 (d, J = 11.1 Hz, 3H), 0.79 (d, J = 6.6 Hz, 6H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 174.33, 174.31, 140.8, 140.7, 137.6, 137.5, 132.80, 132.78, 130.5, 129.45, 129.43, 129.2, 127.2, 71.7, 71.6, 69.8, 69.7, 45.1, 45.0, 38.8, 30.2, 24.3, 24.2, 22.4, 18.1 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{30}NaO_3Se^+$ $[M+Na]^+$: 457.1252, found: 457.1250.



2-Hydroxy-2-methyl-3-(phenylselanyl)propyl 2-(2-hydroxy-2-methyl-3-(phenylselanyl)propoxy)benzoate (55):

GP5 was carried out with 2-methylallyl 2-((2-methylallyl)oxy)benzoate **49a** (91 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the product **55** (170 mg, 82%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**49a**) = 0.70, R_f (**2a**) = 0.95, R_f (**55**) = 0.10, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3387, 1653, 1450, 1372, 1254, 995, 824, 757, 693 cm^{-1}. **^{1}H NMR** (400 MHz, DMSO-d_6) δ = 7.71 – 7.64 (m, 1H), 7.51 – 7.41 (m, 5H), 7.22 – 7.10 (m, 6H), 7.05 (d, J = 8.3 Hz, 1H), 7.02 – 6.95 (m, 1H), 5.23 (br. s, 1H), 5.12 (br. s, 1H), 4.20 (d, J = 10.9 Hz, 1H), 4.16 (d, J = 10.9 Hz, 1H), 3.99 (d, J = 9.0 Hz, 1H), 3.89 (d, J = 9.0 Hz, 1H), 3.30 (s, 2H), 3.18 (s, 2H), 1.34 (s, 3H), 1.27 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, DMSO-d_6) δ = 164.8, 157.9, 133.9, 131.5 (2 × Ar–CH), 131.3, 131.2, 131.2, 131.0 (2 × Ar–CH), 129.2 (2 × Ar–CH), 129.0 (2 × Ar–CH), 126.5, 126.1, 120.2, 119.5, 113.5, 74.3, 71.1, 70.7, 69.9, 37.8, 37.6, 24.9, 24.6 ppm. **HRMS:** *m/z* calcd for $C_{27}H_{30}NaO_5Se_2^+$ $[M+Na]^+$: 617.0316, found: 617.0327.

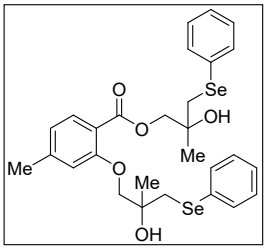


2-Hydroxy-2-methyl-3-(phenylselanyl)propyl 2-(2-hydroxy-2-methyl-3-(phenylselanyl)propoxy)-5-methylbenzoate (56):

GP5 was carried out with 2-methylallyl 5-methyl-2-((2-methylallyl)oxy)benzoate **49b** (96 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the product **56** (170 mg, 80%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**49b**) = 0.70, R_f (**2a**) = 0.95, R_f (**56**) = 0.10, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3409, 1656, 1492, 1257, 1199, 1005, 822, 754, 688 cm^{-1}. **^{1}H NMR** (400 MHz, DMSO-d_6) δ = 7.52 – 7.40 (m, 5H), 7.29 (dd, J = 8.5, 2.0 Hz, 1H), 7.27 – 7.08 (m, 6H), 6.95 (d, J = 8.5 Hz, 1H), 5.21 (br. s, 1H), 5.09 (br. s, 1H), 4.19 (d, J = 10.9 Hz, 1H), 4.15 (d, J = 10.9 Hz, 1H), 3.95 (d, J = 9.0 Hz, 1H), 3.86 (d, J = 9.0 Hz, 1H), 3.28 (s, 2H),

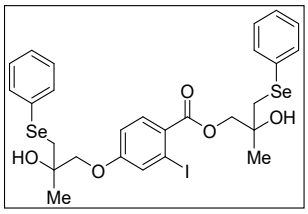
3.18 (s, 2H), 2.23 (s, 3H), 1.33 (s, 3H), 1.27 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, DMSO-d_6) δ = 164.9, 155.9, 134.2, 131.5 (2 × Ar–CH), 131.3, 131.3, 131.1, 131.0 (2 × Ar–CH), 129.2 (2 × Ar–CH), 129.1, 129.0 (2 × Ar–CH), 126.5, 126.1, 119.4, 113.7, 74.6, 71.1, 70.7, 69.9, 37.8, 37.6, 24.9, 24.6, 19.9 ppm. **HRMS:** *m/z* calcd for $C_{28}H_{32}NaO_5Se_2^+$ $[M+Na]^+$: 631.0472, found: 631.0488.



2-Hydroxy-2-methyl-3-(phenylselanyl)propyl 2-(2-hydroxy-2-methyl-3-(phenylselanyl)propoxy)-4-methylbenzoate (57):

GP5 was carried out with 2-methylallyl 4-methyl-2-((2-methylallyl)oxy)benzoate **49c** (96 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the product **57** (180 mg, 85%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**49c**) = 0.80, R_f (**2a**) = 0.95, R_f (**57**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3415, 1657, 1457, 1366, 1267, 1006, 823, 756, 692 cm^{-1}. **^{1}H NMR** (400 MHz, DMSO-d_6) δ = 7.61 (d, J = 7.9 Hz, 1H), 7.51 – 7.42 (m, 4H), 7.24 – 7.07 (m, 6H), 6.86 (s, 1H), 6.79 (d, J = 7.9 Hz, 1H), 5.24 (br. s, 1H), 5.13 (br. s, 1H), 4.18 (d, J = 10.9 Hz, 1H), 4.14 (d, J = 10.9 Hz, 1H), 3.98 (d, J = 9.0 Hz, 1H), 3.88 (d, J = 9.0 Hz, 1H), 3.31 (s, 2H), 3.18 (s, 2H), 2.30 (s, 3H), 1.35 (s, 3H), 1.28 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, DMSO-d_6) δ = 164.6, 158.3, 144.6, 131.4 (2 × Ar–CH), 131.3, 131.2, 131.1 (2 × Ar–CH), 129.1 (2 × Ar–CH), 129.0 (2 × Ar–CH), 126.4, 126.1, 120.9, 116.4 (2 × Ar–C), 114.1, 74.3, 71.2, 70.7, 69.8, 37.8, 37.6, 24.9, 24.6, 21.4 ppm. **HRMS:** *m/z* calcd for $C_{28}H_{32}NaO_5Se_2^+$ $[M+Na]^+$: 631.0472, found: 631.0482.

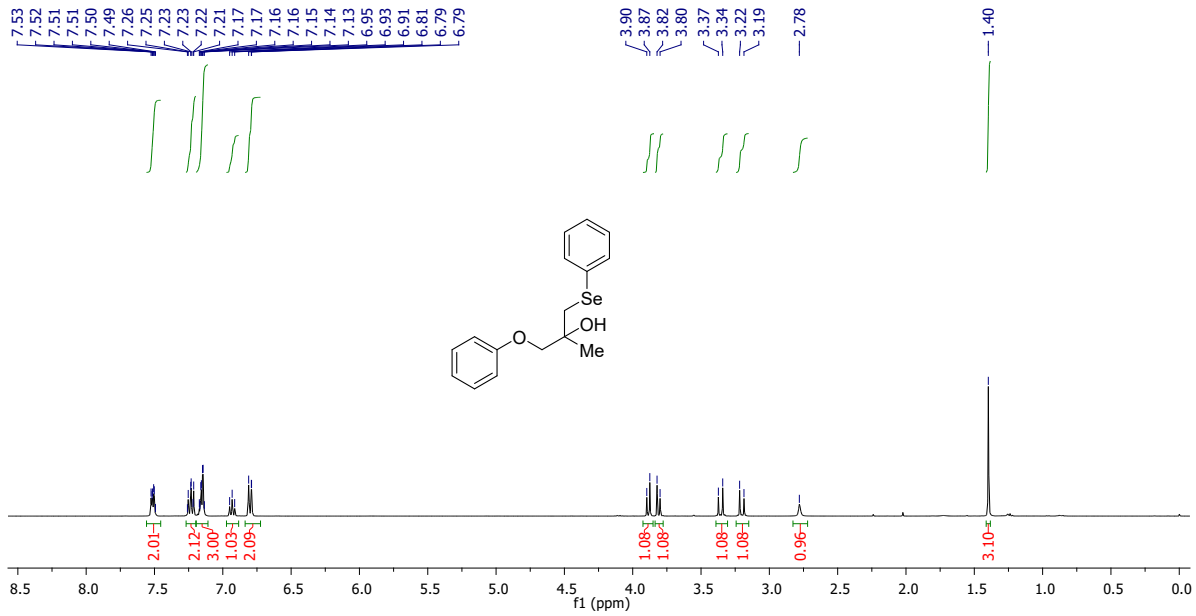


2-Hydroxy-2-methyl-3-(phenylselanyl)propyl 4-(2-hydroxy-2-methyl-3-(phenylselanyl)propoxy)-2-iodobenzoate (58):

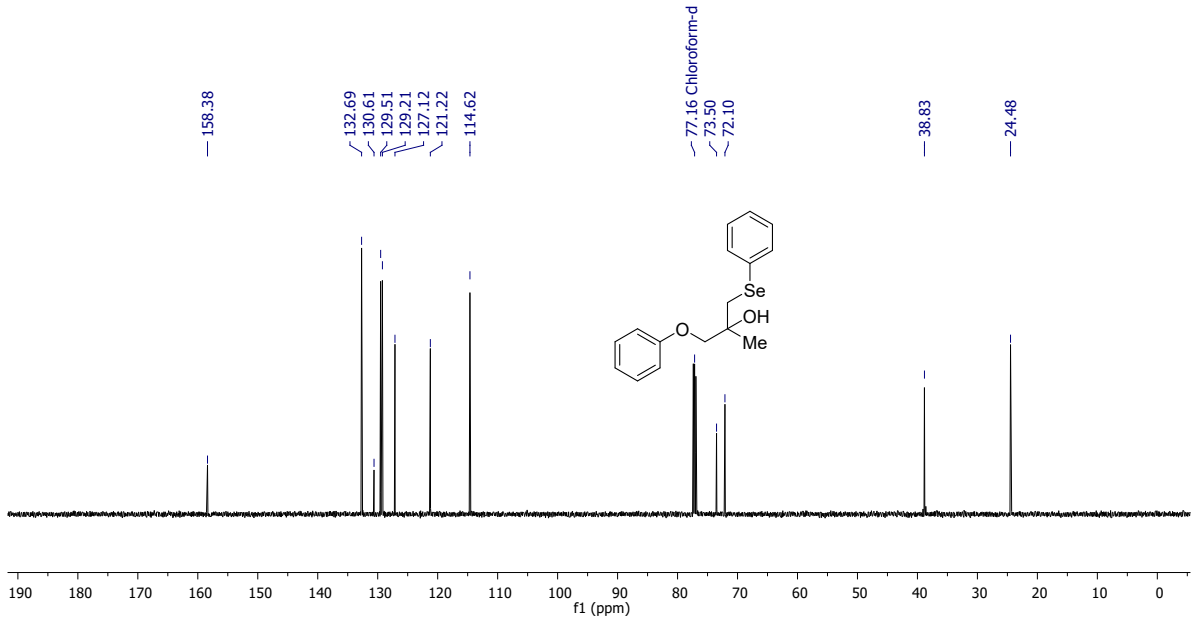
GP5 was carried out with 2-methylallyl 2-iodo-4-((2-methylallyl)oxy)benzoate **49d** (138 mg, 0.37 mmol), diphenyl diselenide **2a** (92 mg, 0.29 mmol), and $LiClO_4$ (32 mg, 0.05 M) in CH_3CN (6 mL) for 2 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10) furnished the product **58** (192 mg, 75%), as a yellow liquid [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**49d**) = 0.80, R_f (**2a**) = 0.95, R_f (**58**) = 0.30, UV detection]. **IR:** (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3446, 3062, 2926, 1712, 1578, 1465, 1405, 1282, 1231, 1111, 1027, 749, 686 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.78 (d, J = 8.1 Hz, 1H), 7.58 – 7.49 (m, 4H), 7.28 – 7.24 (m, 1H), 7.23 – 7.19 (m, 3H), 7.13 (d, J = 1.8 Hz, 1H), 7.10 – 6.98 (m, 3H), 4.30 (d, J = 11.3 Hz, 1H), 4.27 (d, J = 11.3 Hz, 1H), 3.94 (d, J = 8.6 Hz, 1H), 3.85 (d, J = 8.6 Hz, 1H), 3.45 (d, J = 13.0 Hz, 1H), 3.32 (d, J = 6.1 Hz, 1H), 3.29 (d, J = 6.1 Hz, 1H), 3.16 (d, J = 12.9 Hz, 1H), 2.52 (br. s, 1H), 2.07 (br. s, 1H), 1.47 (s, 3H), 1.40 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 165.7, 156.6, 139.3, 133.0 (2 × Ar–CH), 132.9 (2 × Ar–CH), 131.2, 130.4, 129.9, 129.4 (2 × Ar–CH), 129.2 (2 × Ar–CH), 127.5, 127.1, 123.6, 112.2, 93.6, 74.1, 72.3, 71.7, 70.7, 39.7, 38.5, 24.8, 24.5 ppm. **HRMS:** *m/z* calcd for $C_{27}H_{29}IKO_5Se_2^+$ $[M+K]^+$: 758.9022, found: 758.9047.

NMR Spectra:

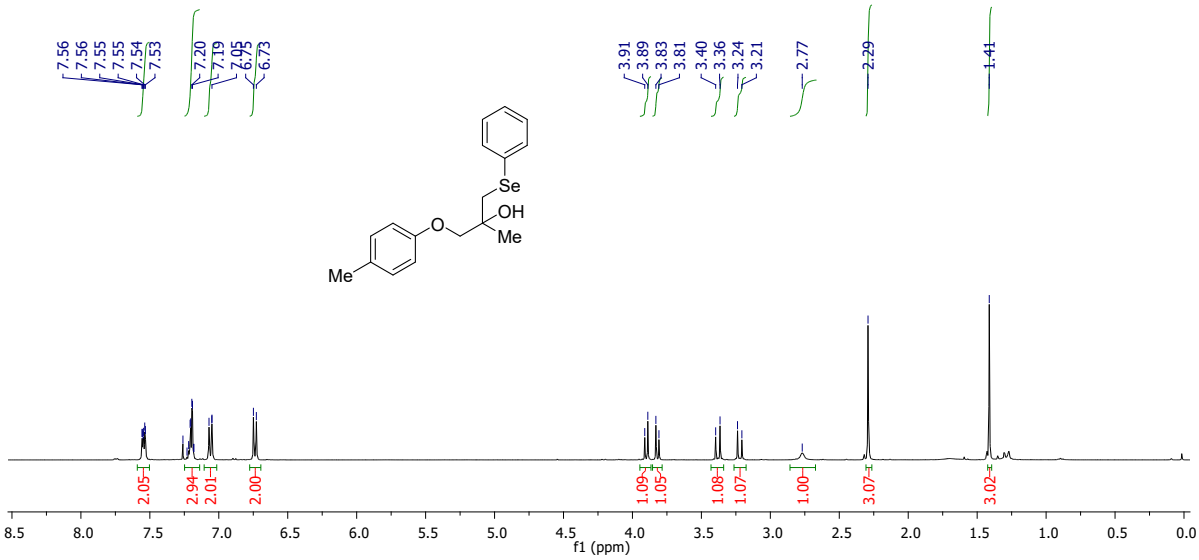
^{1}H-NMR (400 MHz) spectrum of **3** in $CDCl_3$



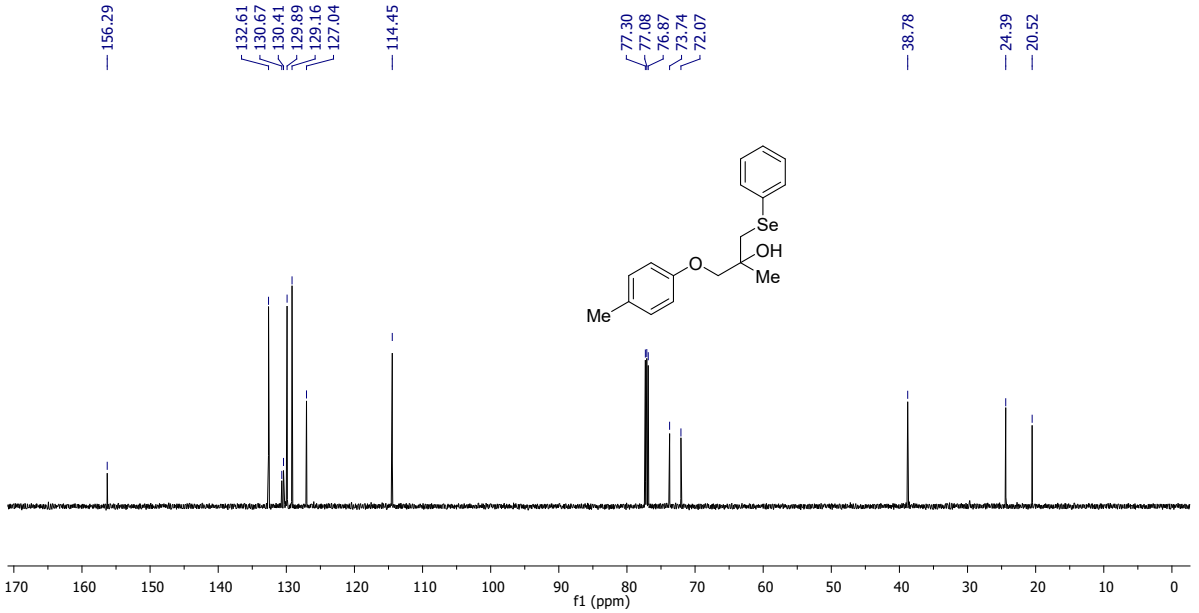
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3** in $CDCl_3$



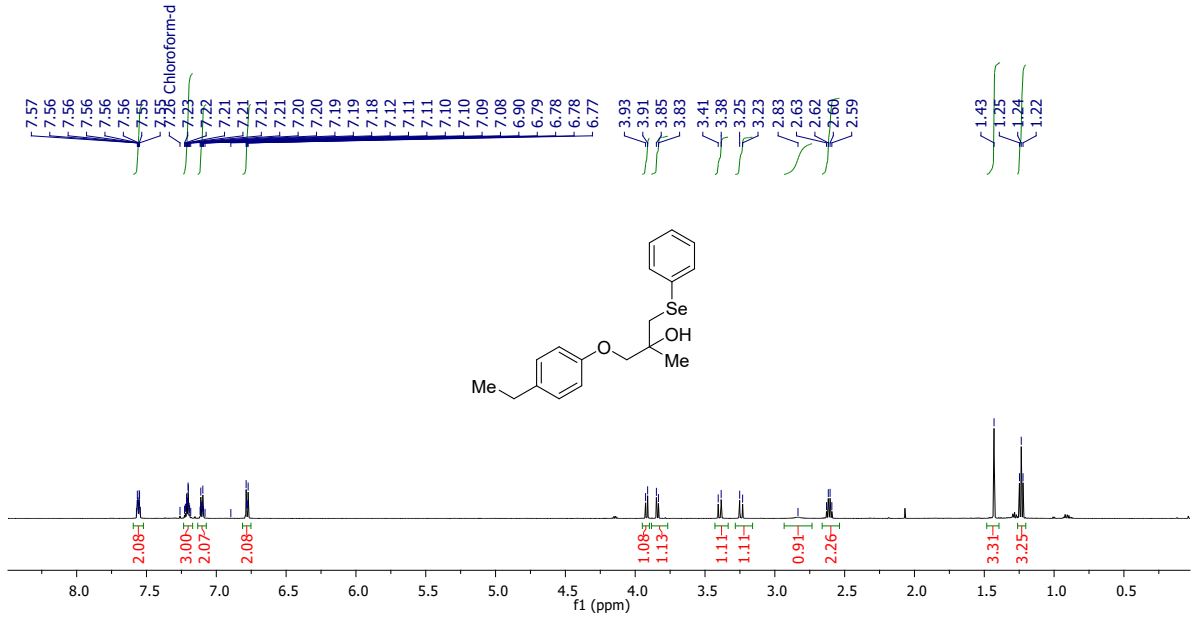
^{1}H-NMR (400 MHz) spectrum of **4** in $CDCl_3$



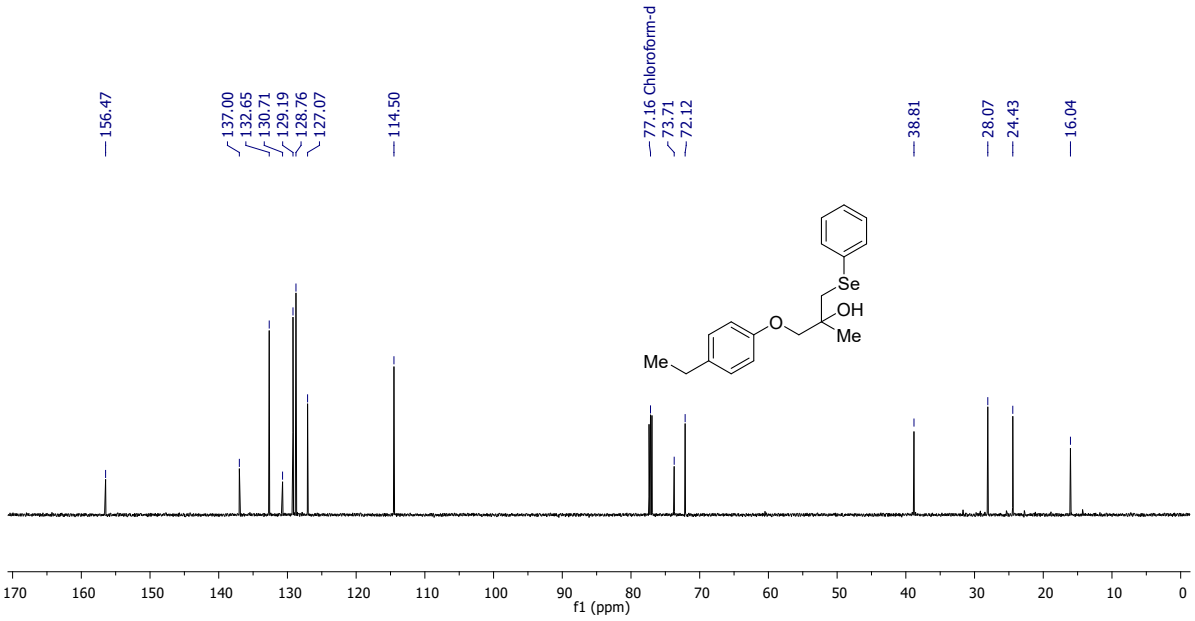
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **4** in $CDCl_3$



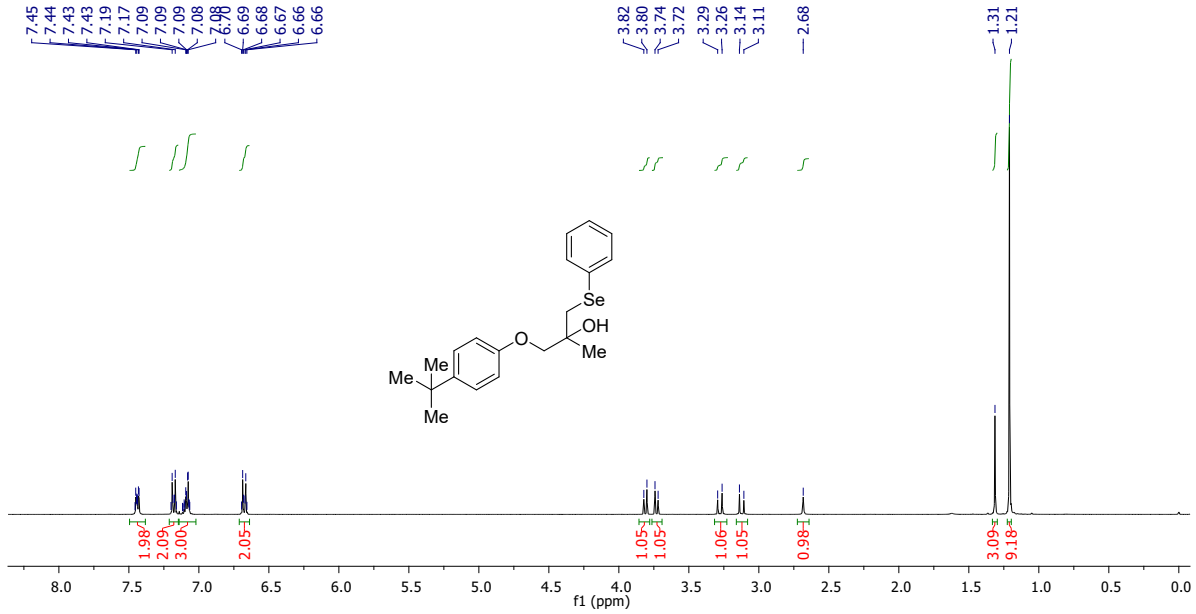
^{1}H-NMR (400 MHz) spectrum of **5** in $CDCl_3$



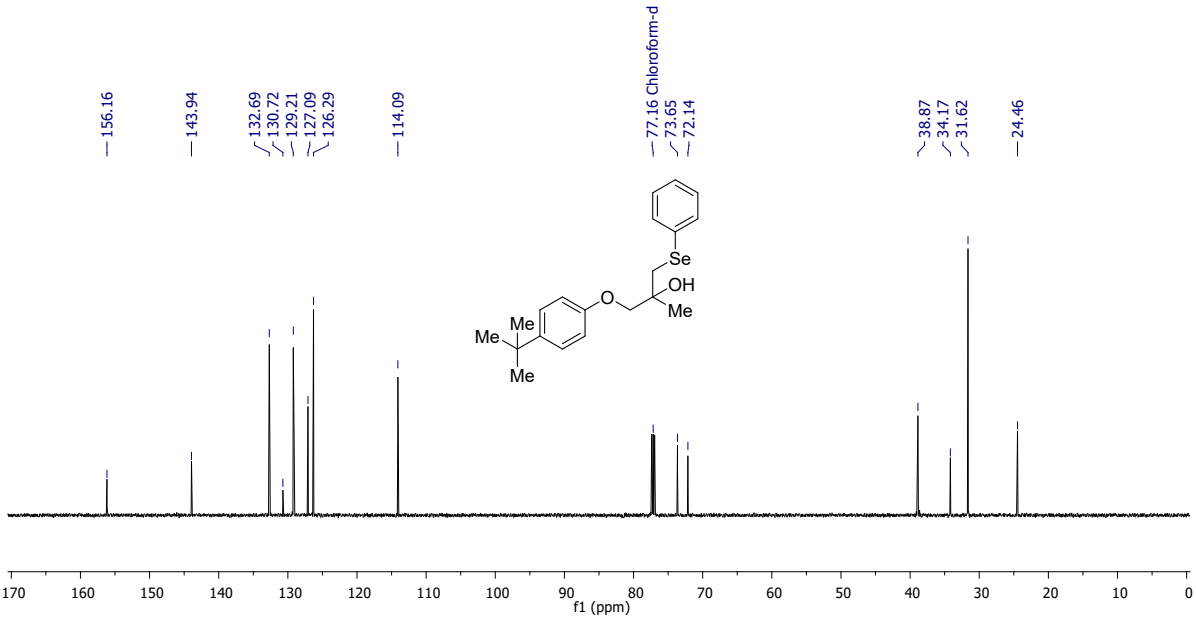
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **5** in $CDCl_3$



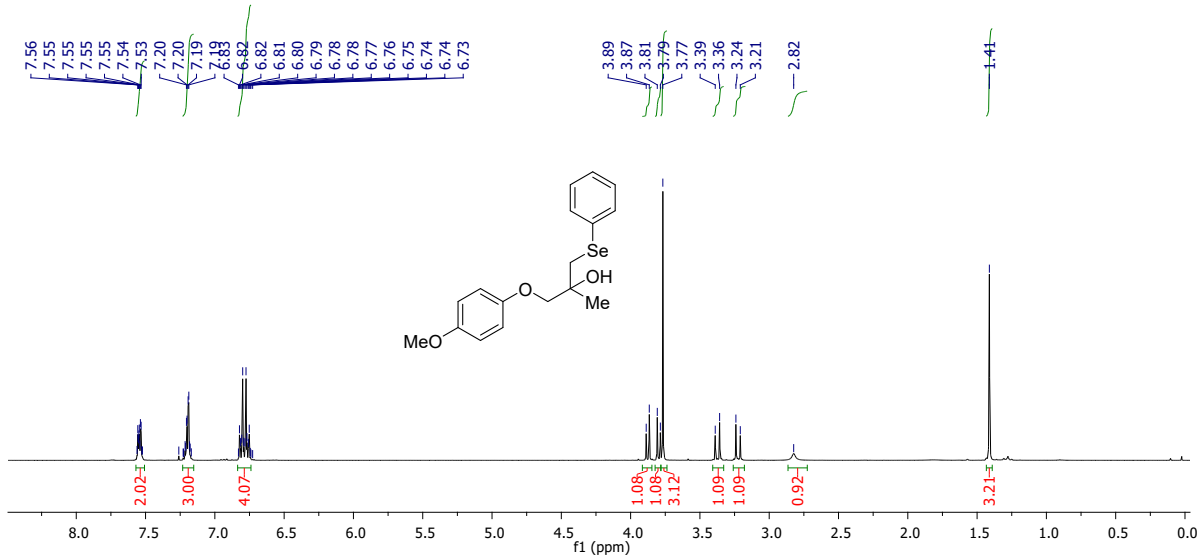
^{1}H-NMR (400 MHz) spectrum of **6** in $CDCl_3$



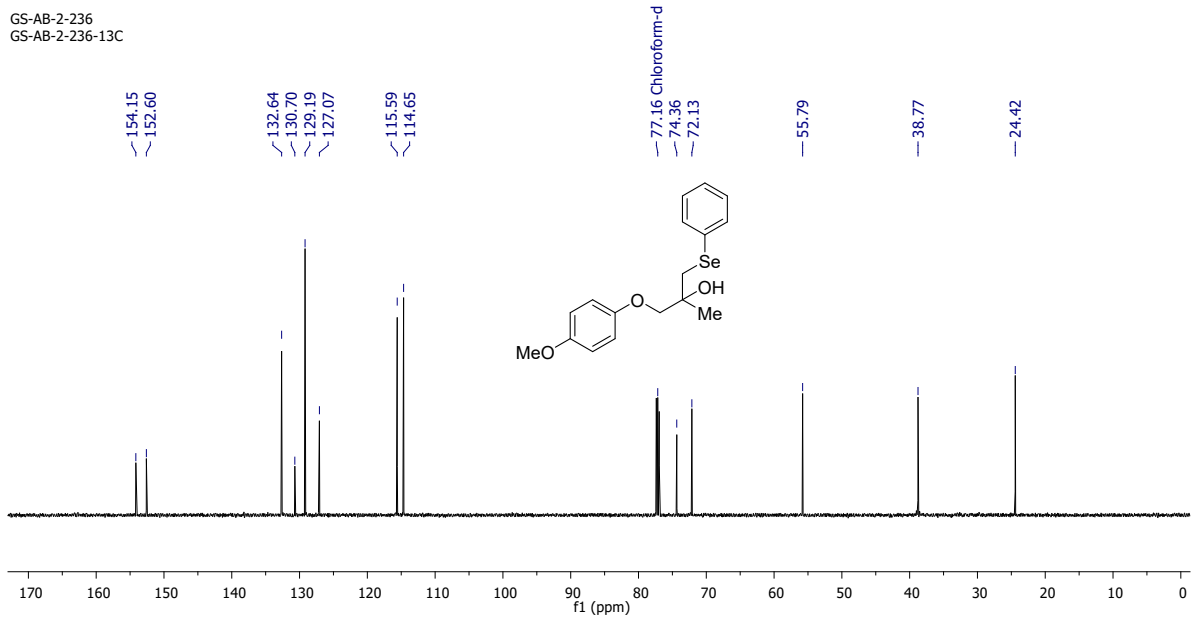
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **6** in $CDCl_3$



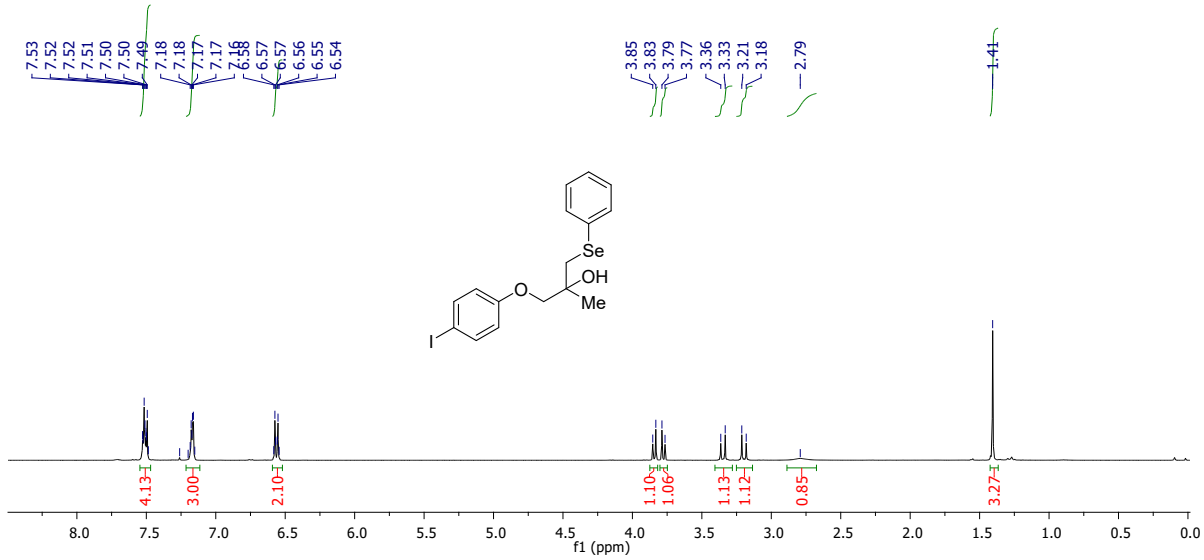
^{1}H-NMR (400 MHz) spectrum of **7** in $CDCl_3$



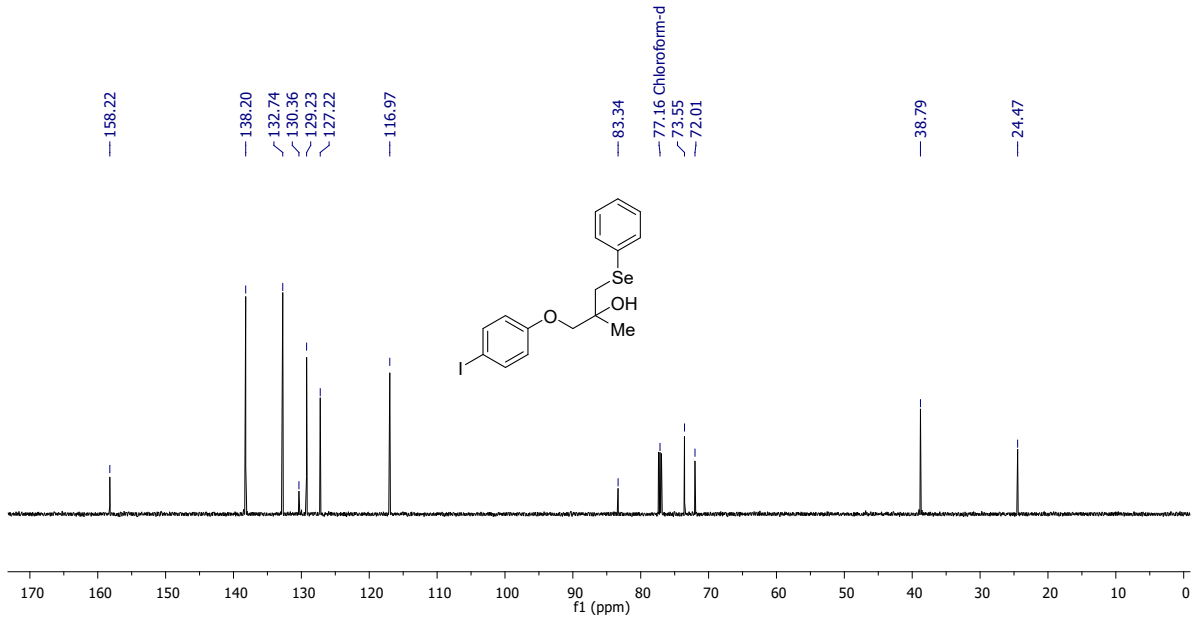
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **7** in $CDCl_3$



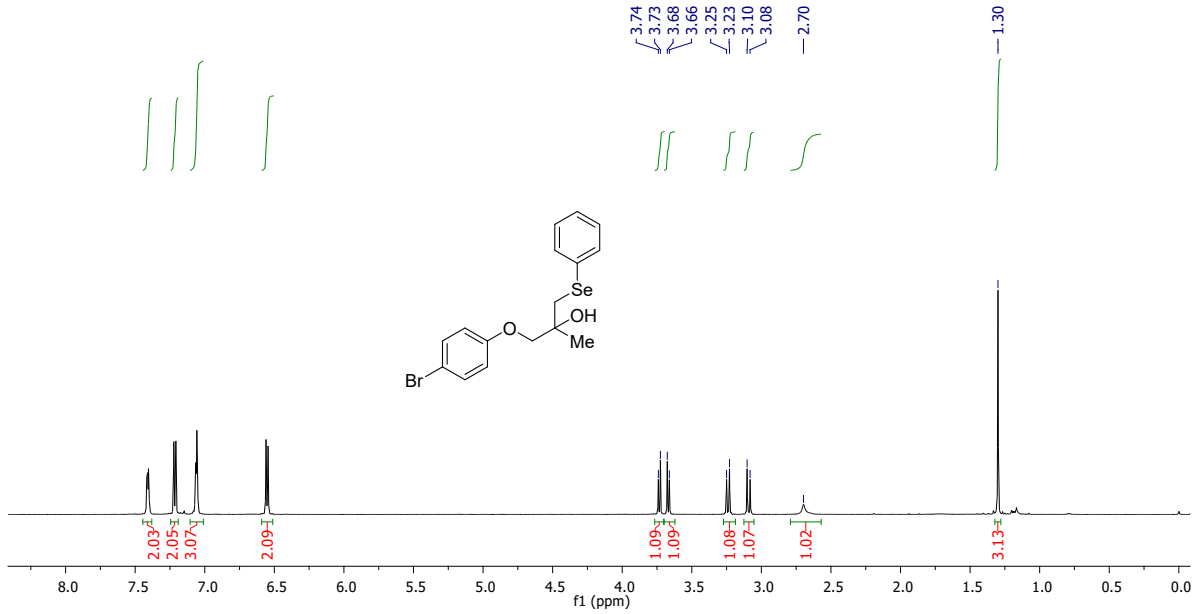
^{1}H-NMR (400 MHz) spectrum of **8** in $CDCl_3$



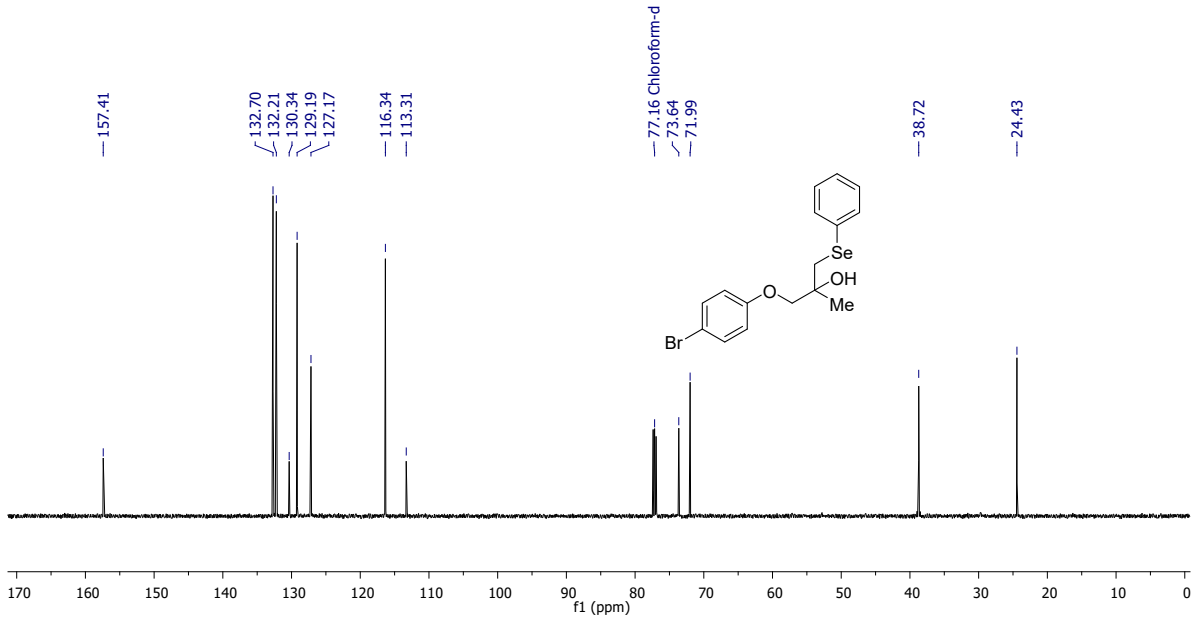
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **8** in $CDCl_3$



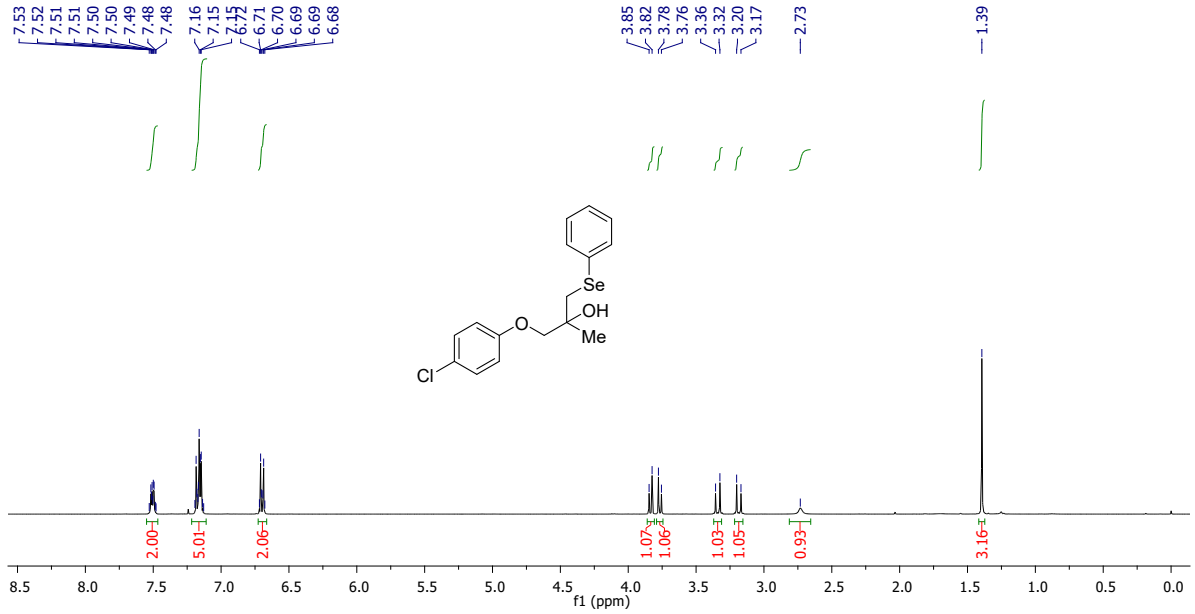
^{1}H-NMR (600 MHz) spectrum of **9** in $CDCl_3$



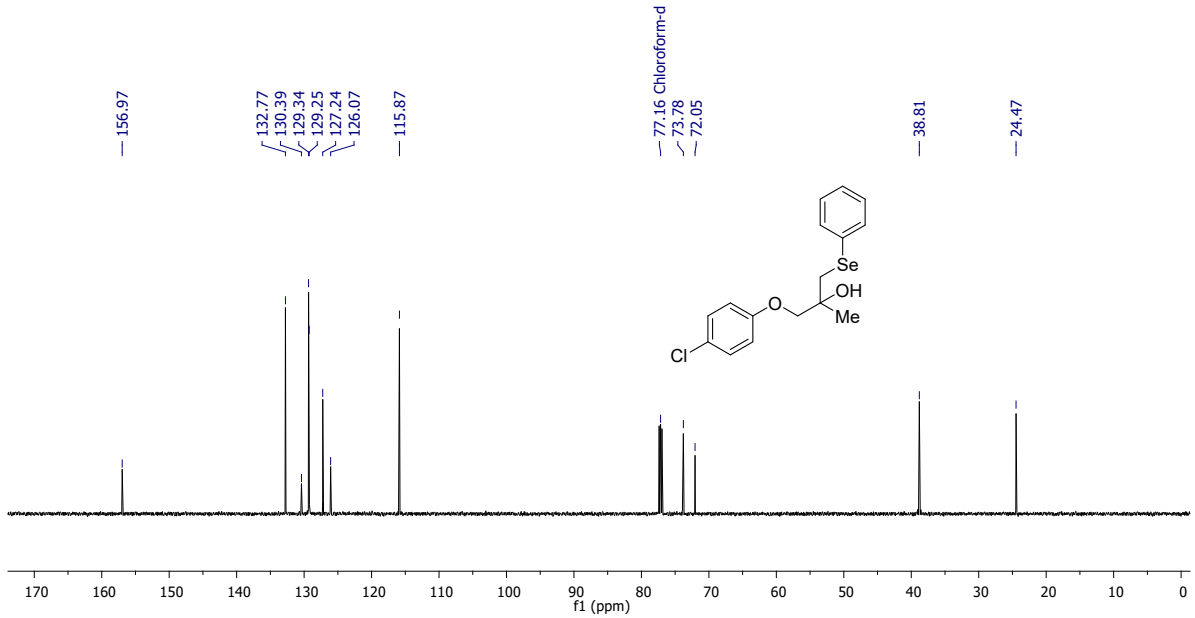
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **9** in $CDCl_3$



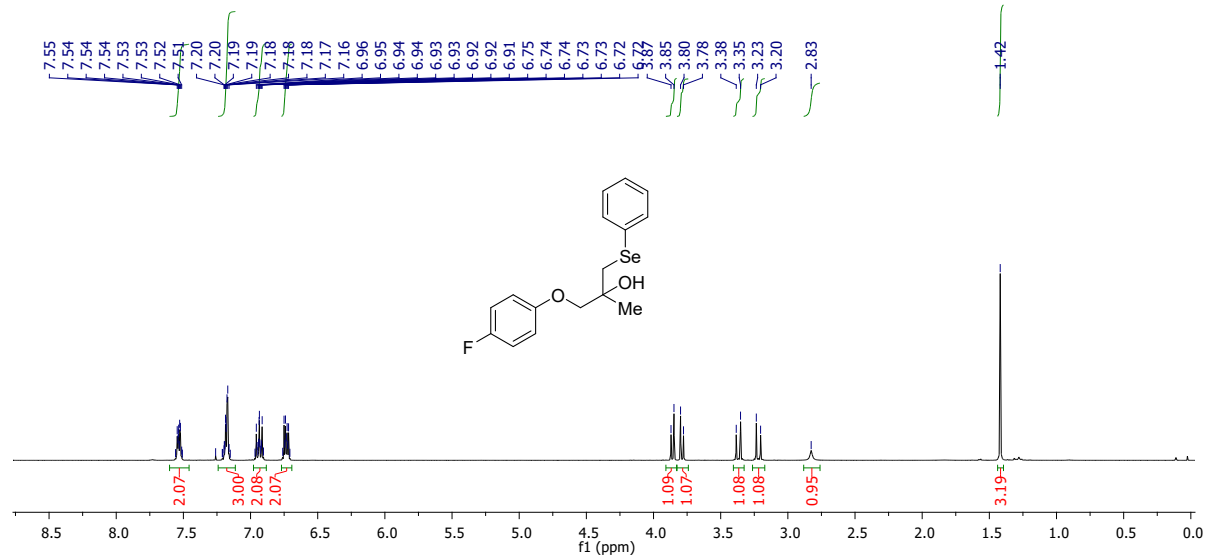
^{1}H-NMR (400 MHz) spectrum of **10** in $CDCl_3$



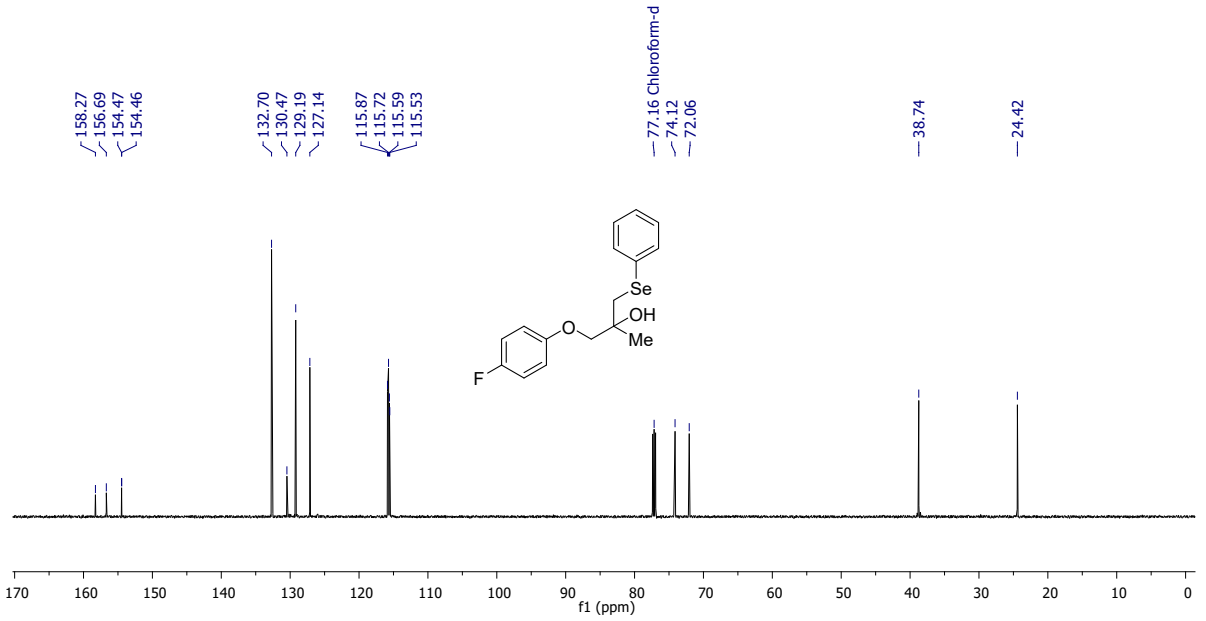
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **10** in $CDCl_3$



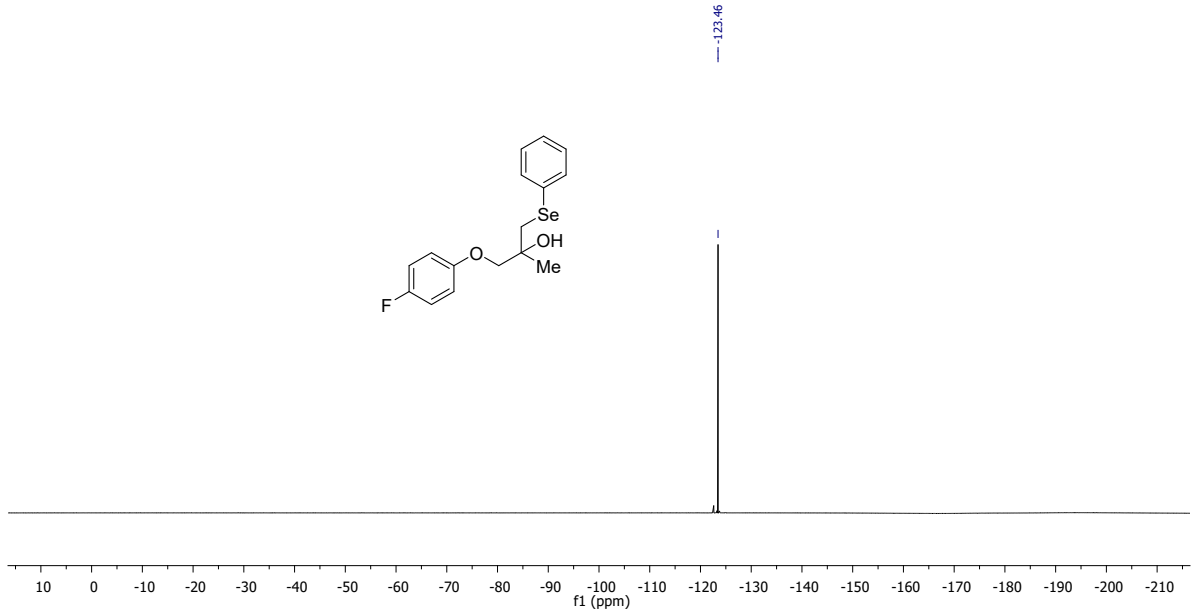
^{1}H-NMR (400 MHz) spectrum of **11** in $CDCl_3$



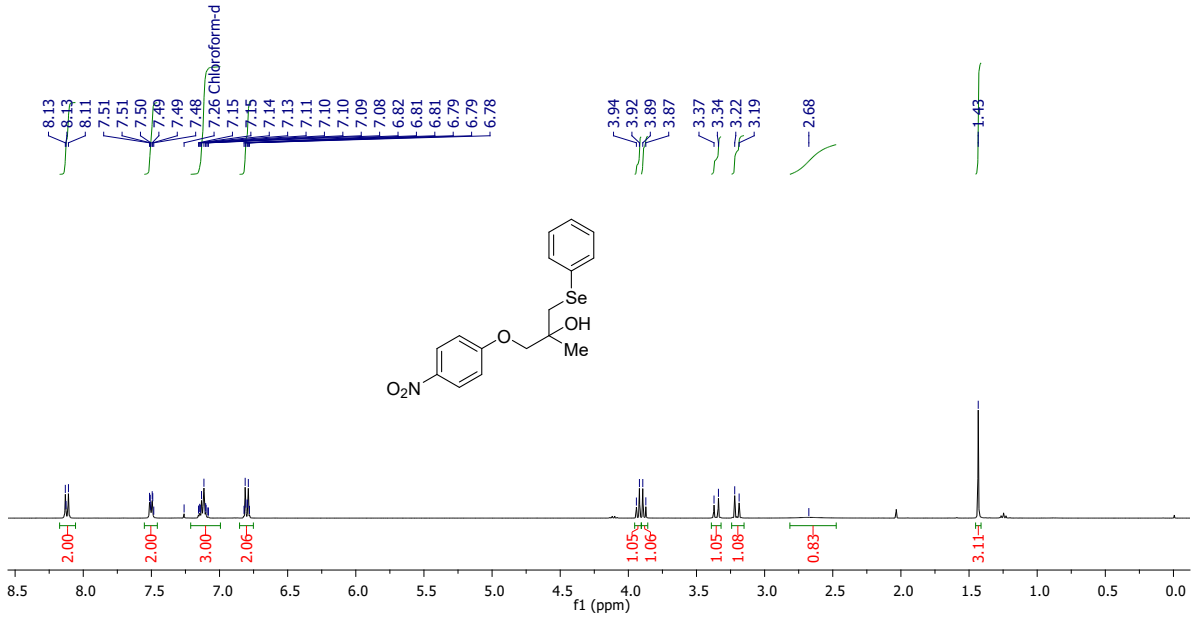
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **11** in $CDCl_3$



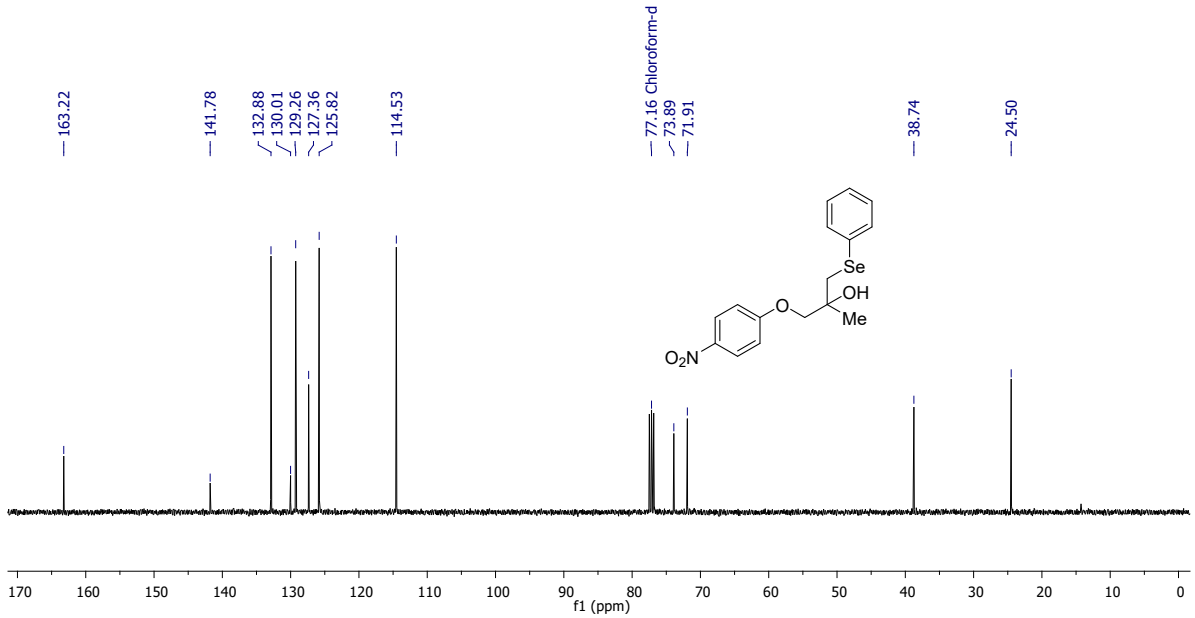
^{19}F NMR (565 MHz) spectrum of **11** in $CDCl_3$



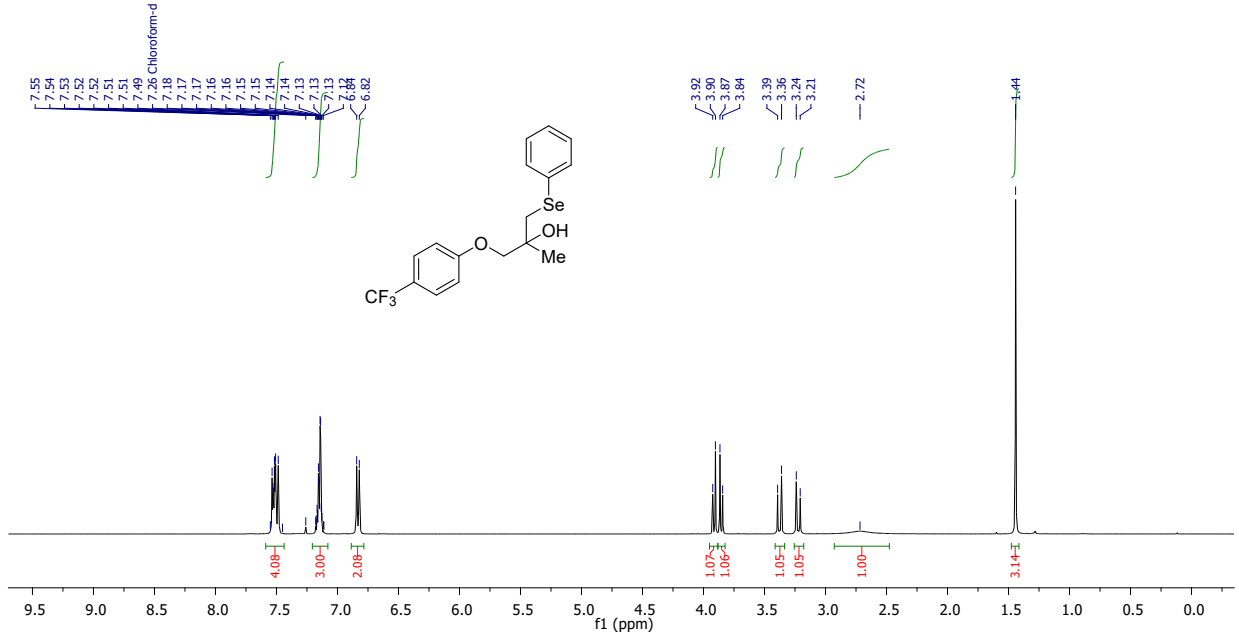
^{1}H-NMR (400 MHz) spectrum of **12** in $CDCl_3$



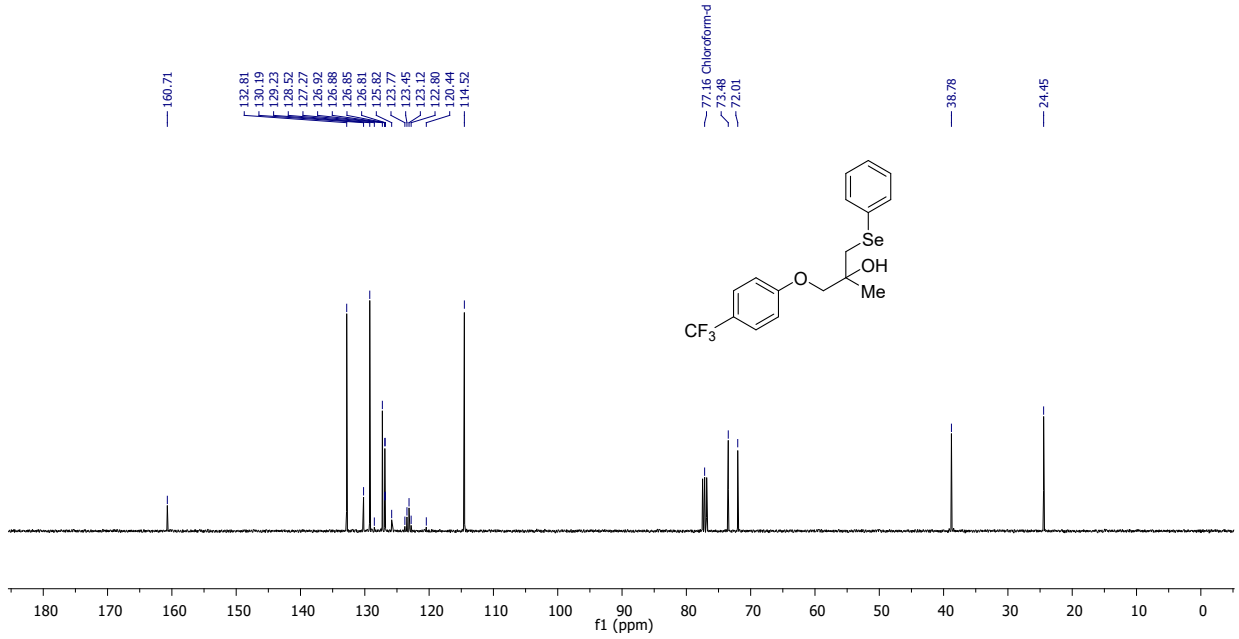
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **12** in $CDCl_3$



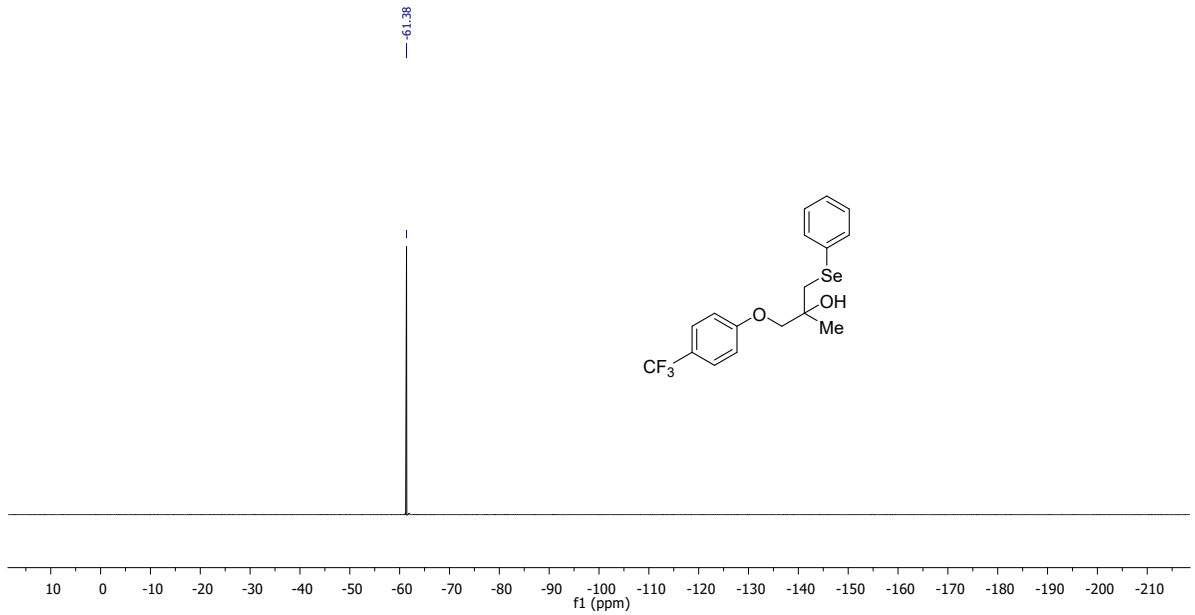
^{1}H-NMR (400 MHz) spectrum of **13** in $CDCl_3$



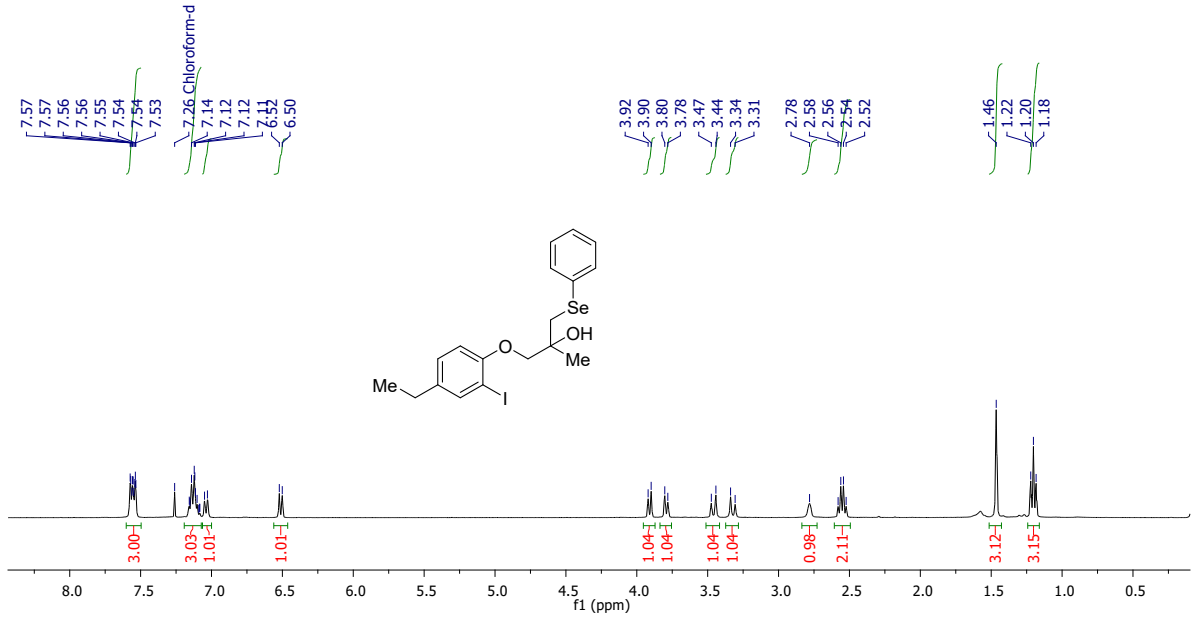
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **13** in $CDCl_3$



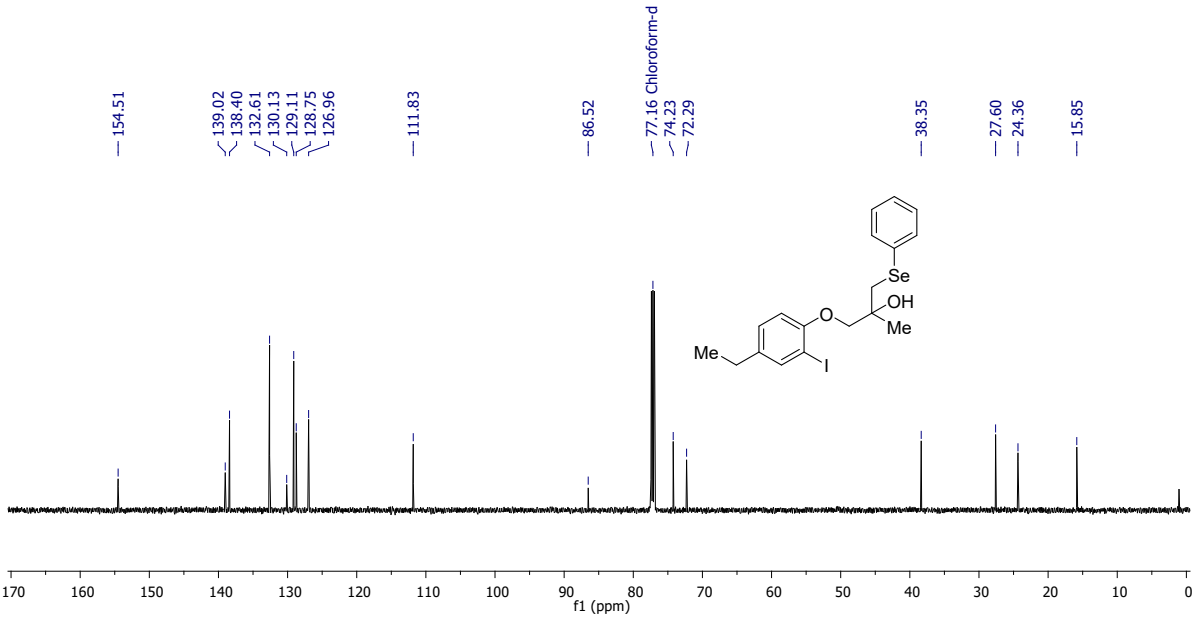
^{19}F NMR (376 MHz) spectrum of **13** in $CDCl_3$



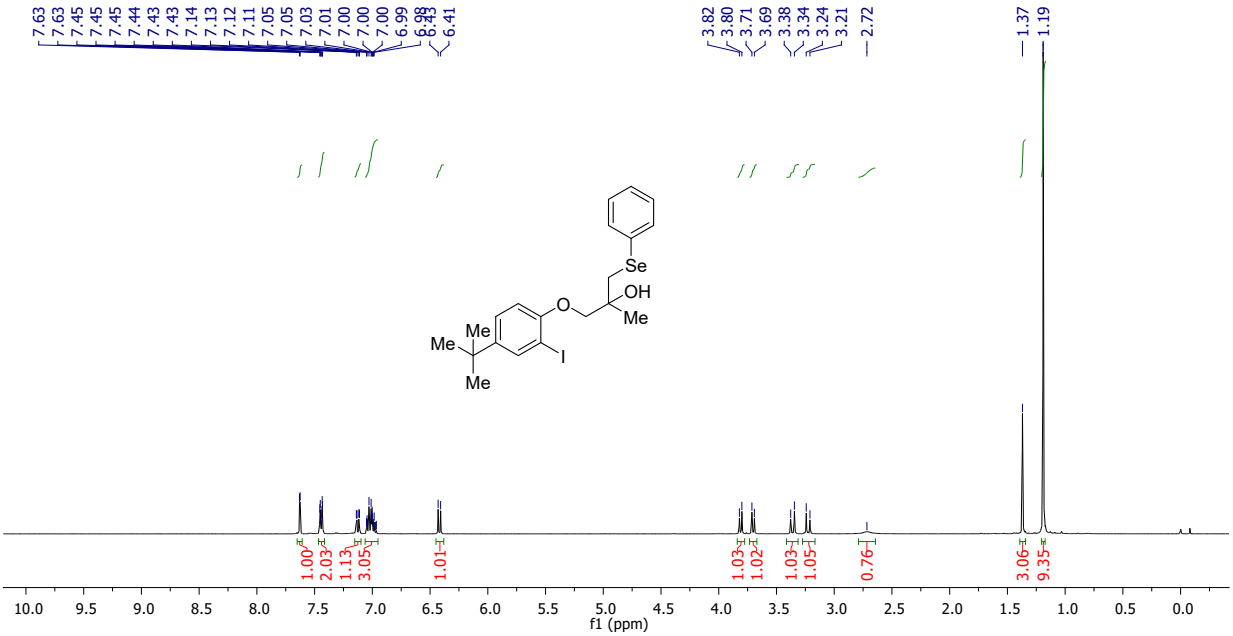
^{1}H-NMR (400 MHz) spectrum of **14** in $CDCl_3$



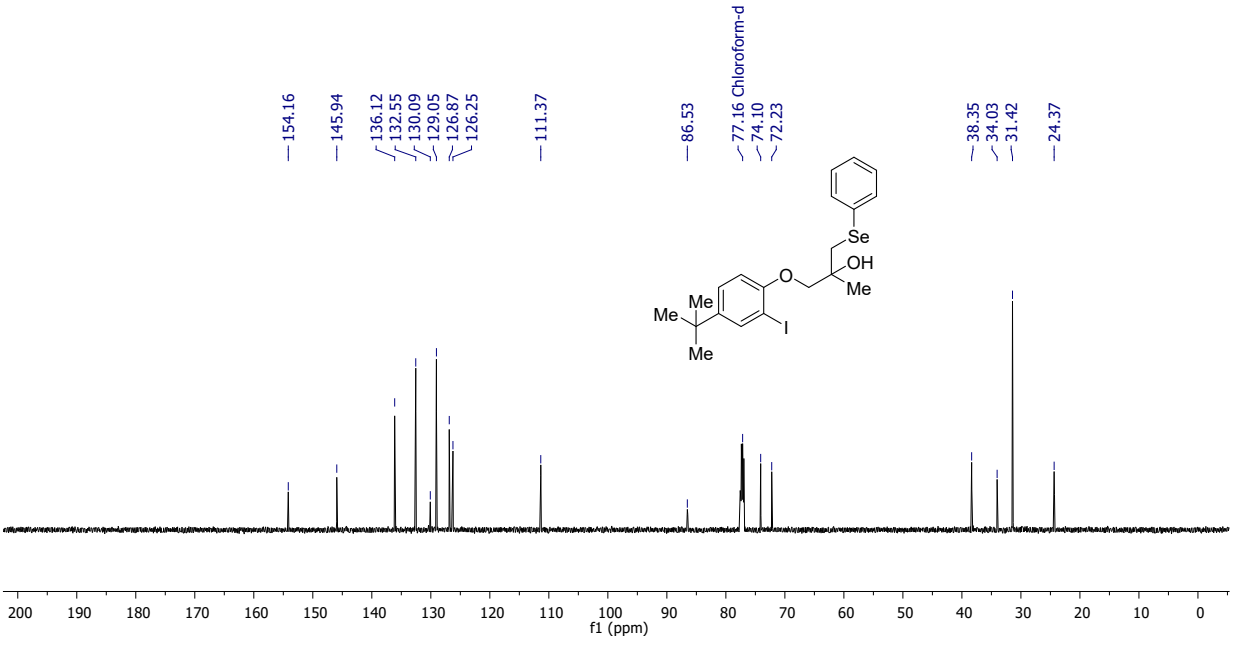
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **14** in $CDCl_3$



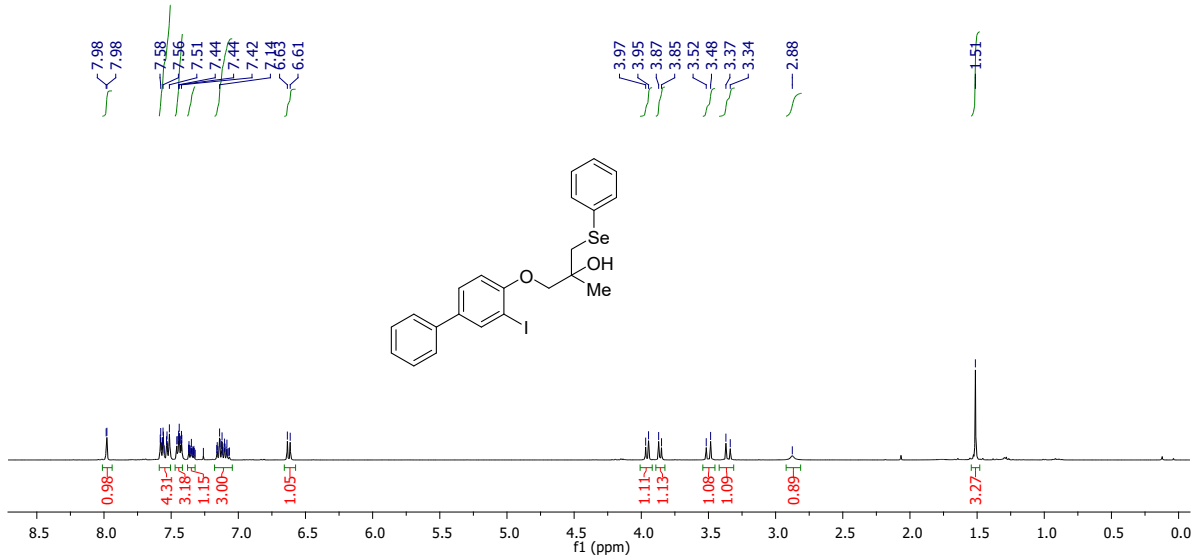
^{1}H-NMR (400 MHz) spectrum of **15** in $CDCl_3$



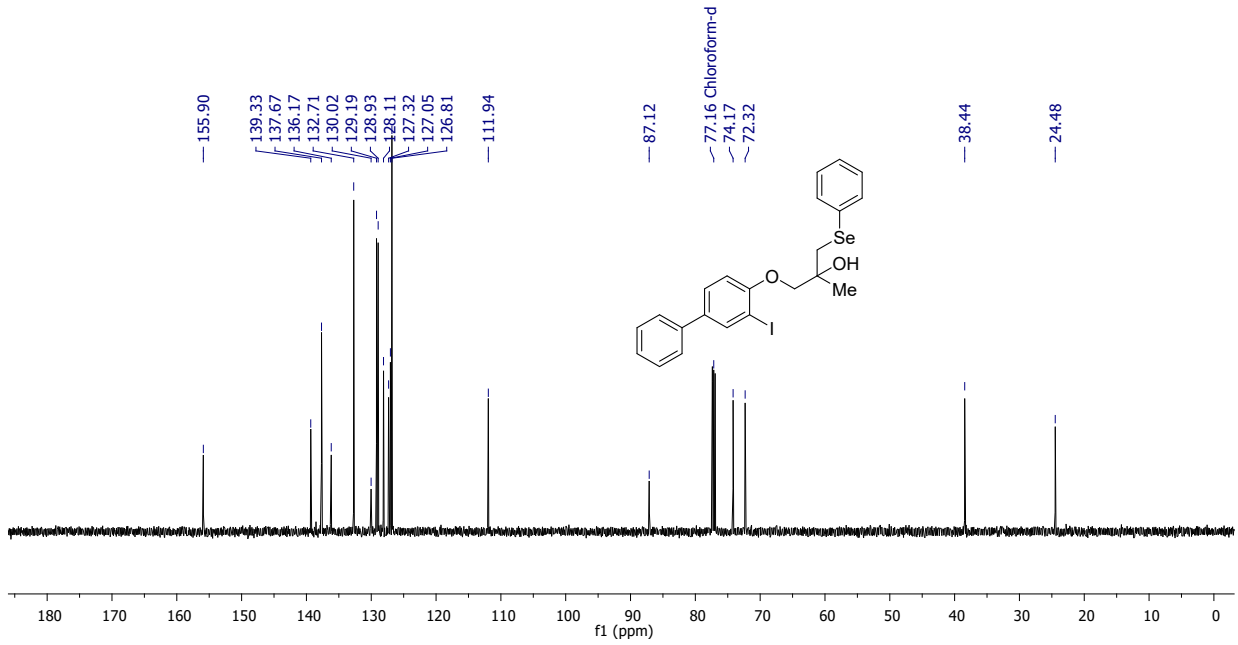
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **15** in $CDCl_3$



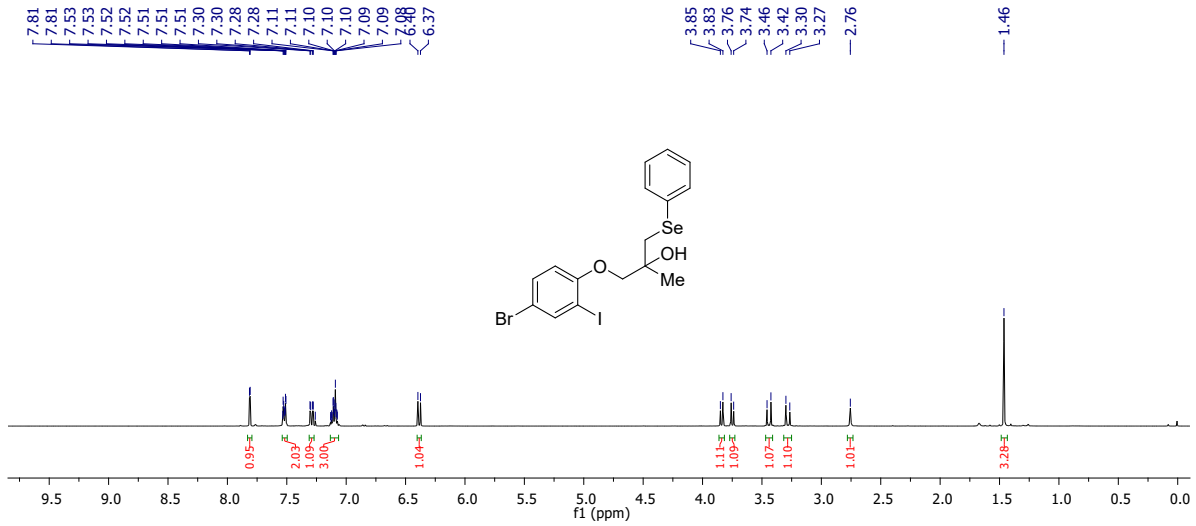
^{1}H-NMR (400 MHz) spectrum of **16** in $CDCl_3$



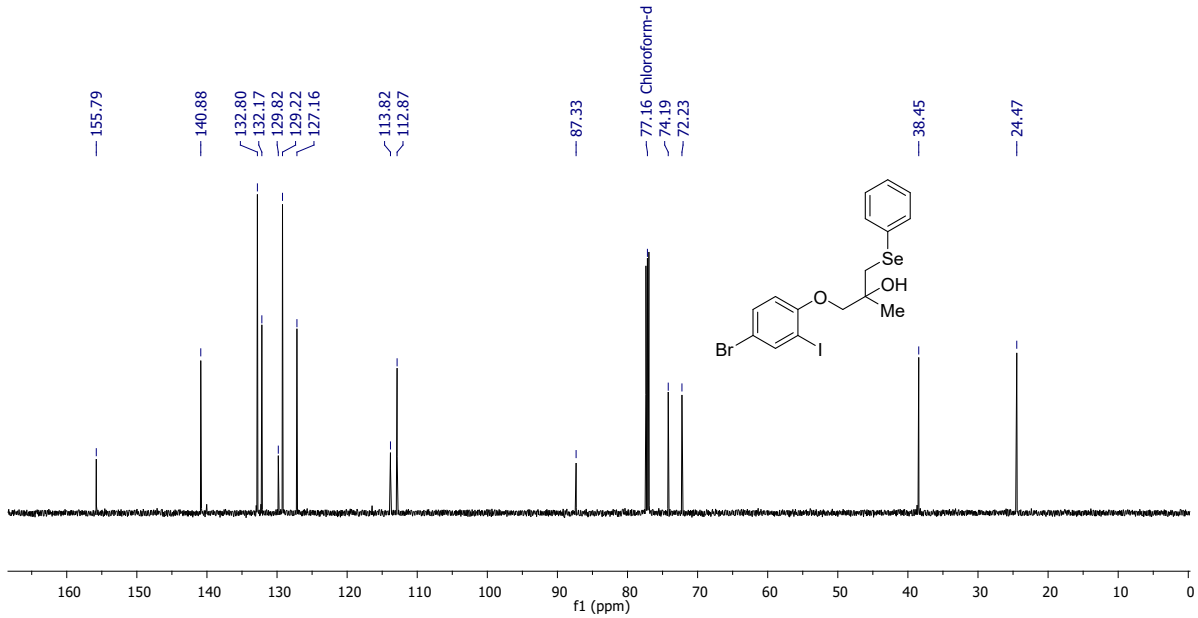
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **16** in $CDCl_3$



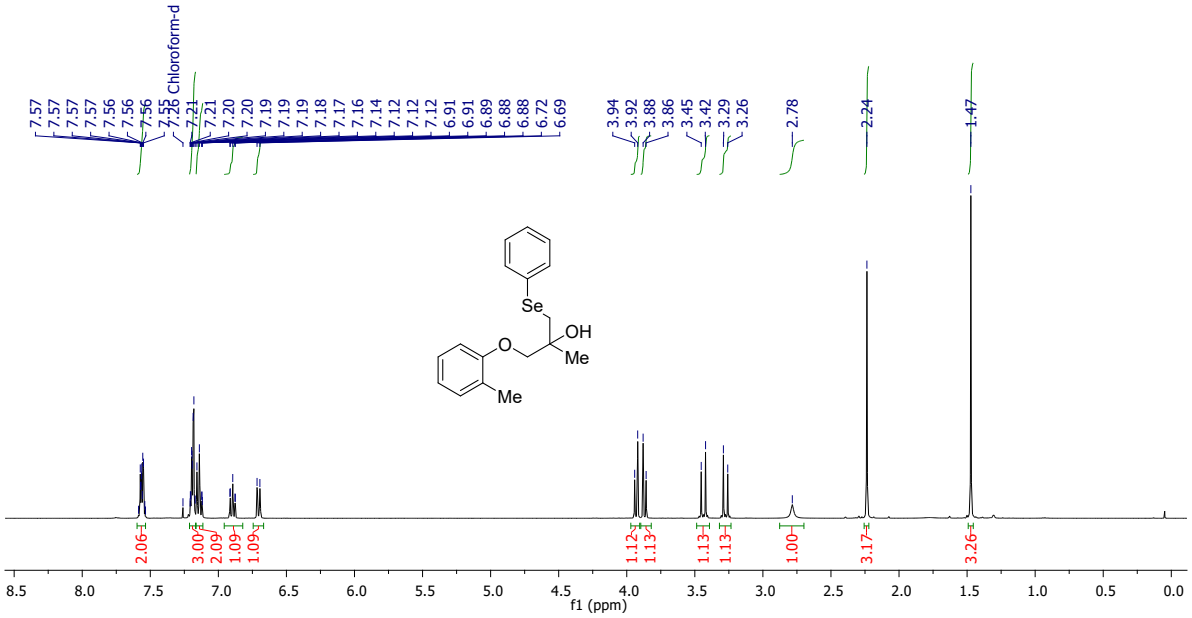
^{1}H-NMR (400 MHz) spectrum of **17** in $CDCl_3$



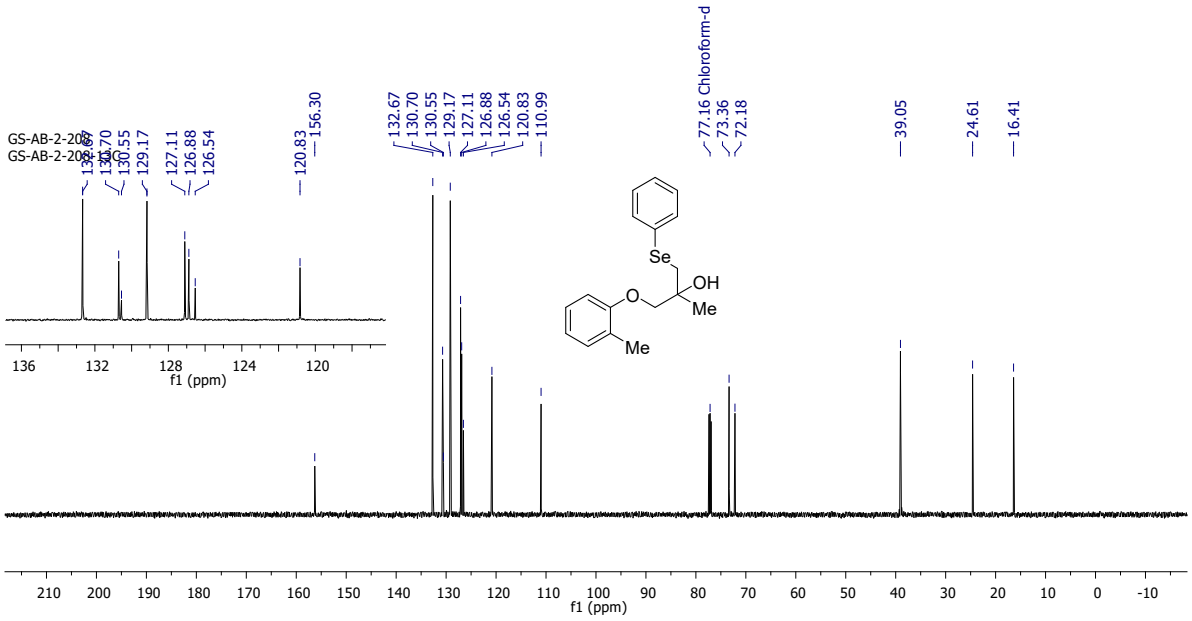
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **17** in $CDCl_3$



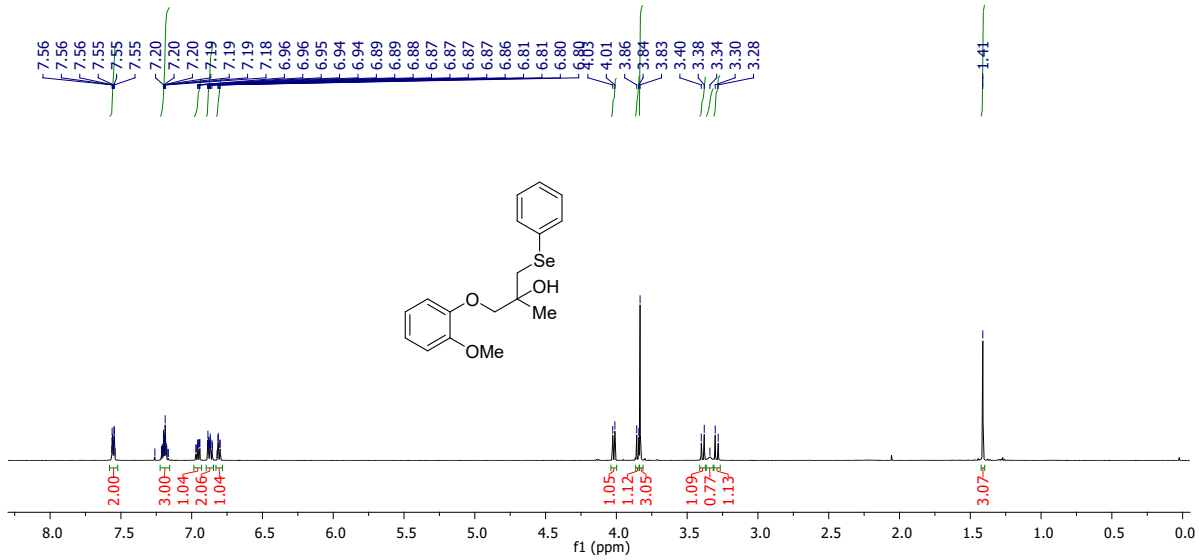
^{1}H-NMR (400 MHz) spectrum of **18** in $CDCl_3$



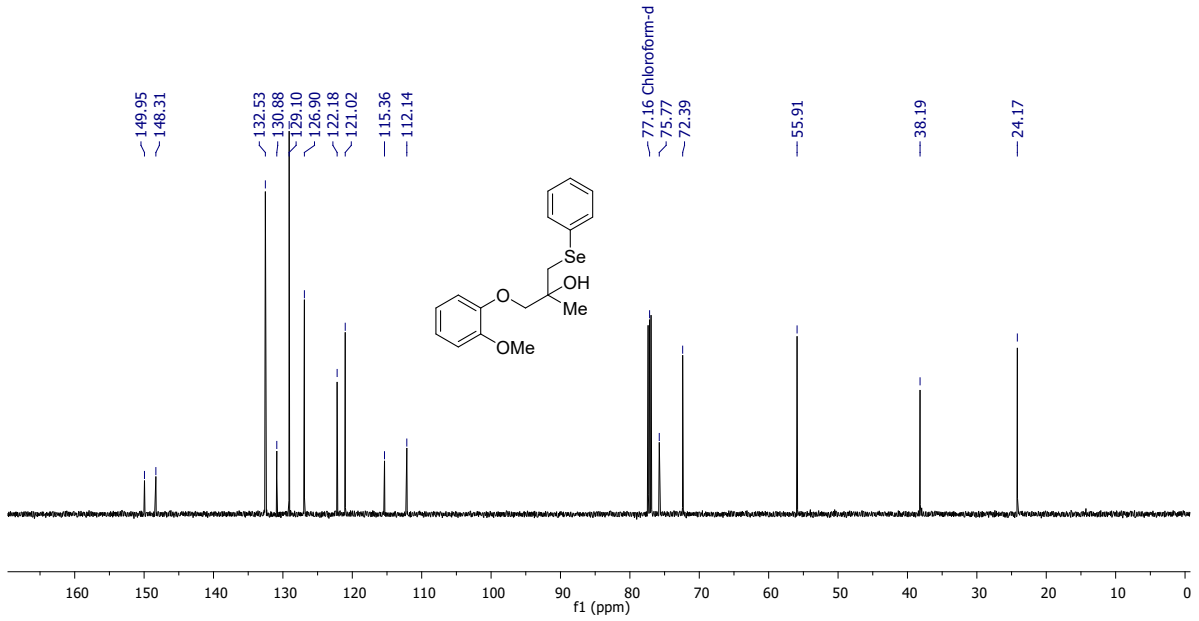
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **18** in $CDCl_3$



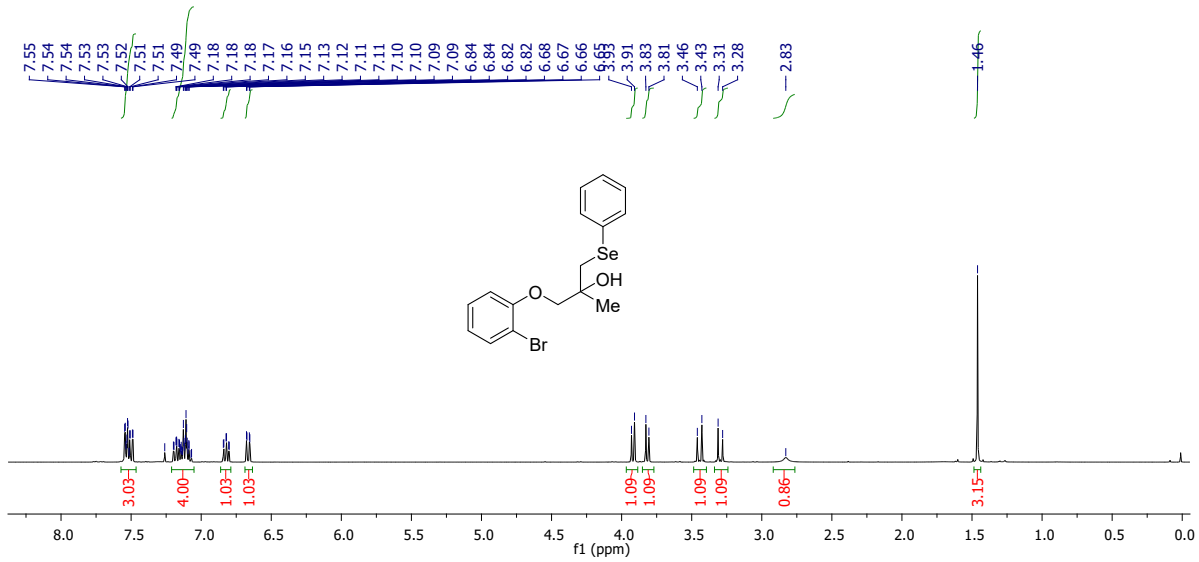
^{1}H-NMR (400 MHz) spectrum of **19** in $CDCl_3$



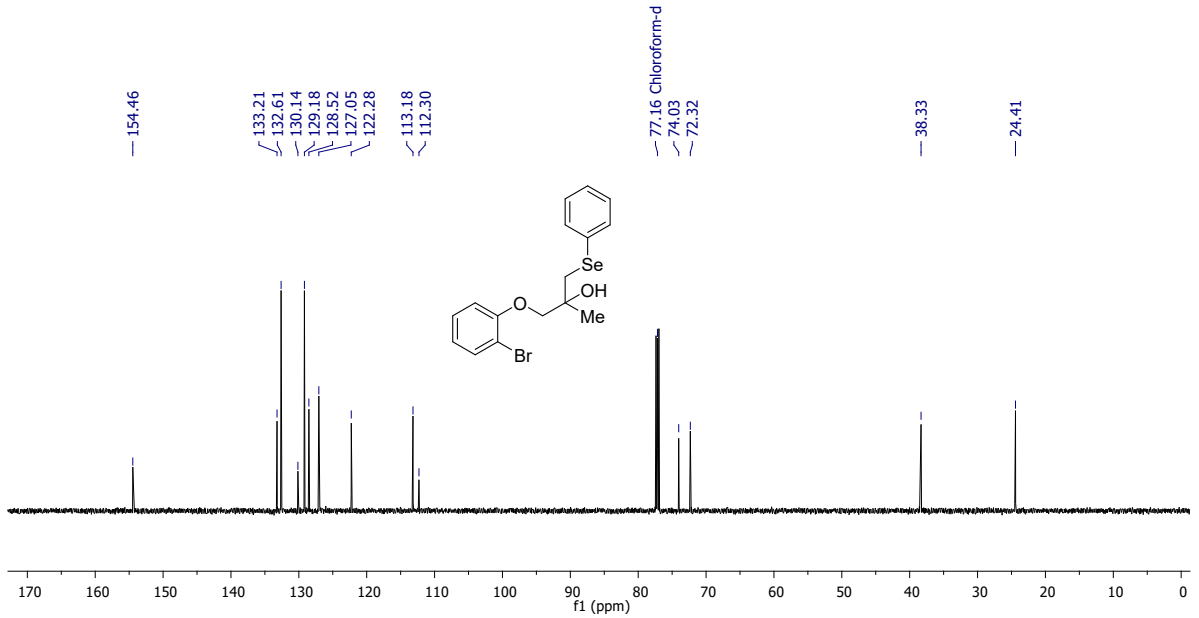
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **19** in $CDCl_3$



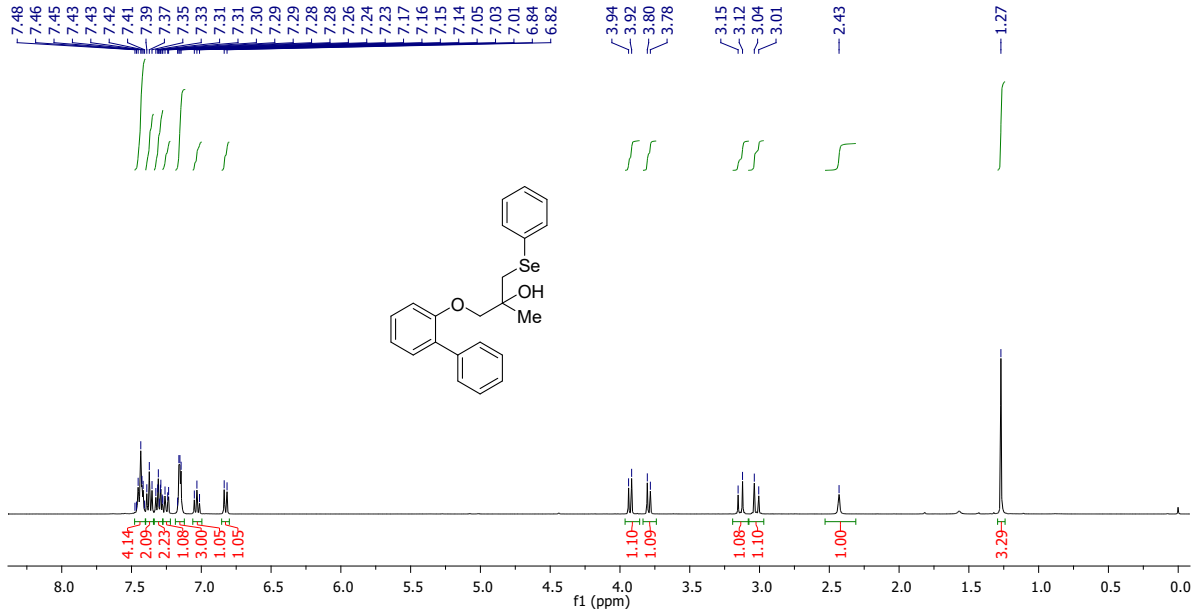
^{1}H-NMR (400 MHz) spectrum of **20** in $CDCl_3$



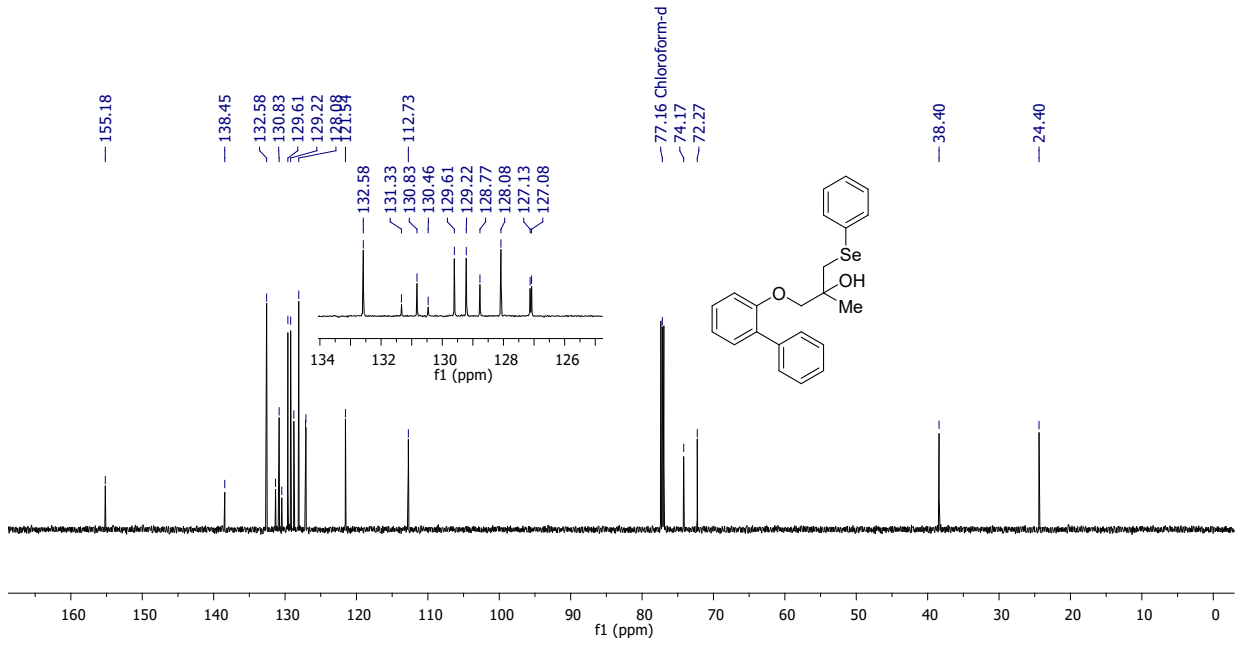
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **20** in $CDCl_3$



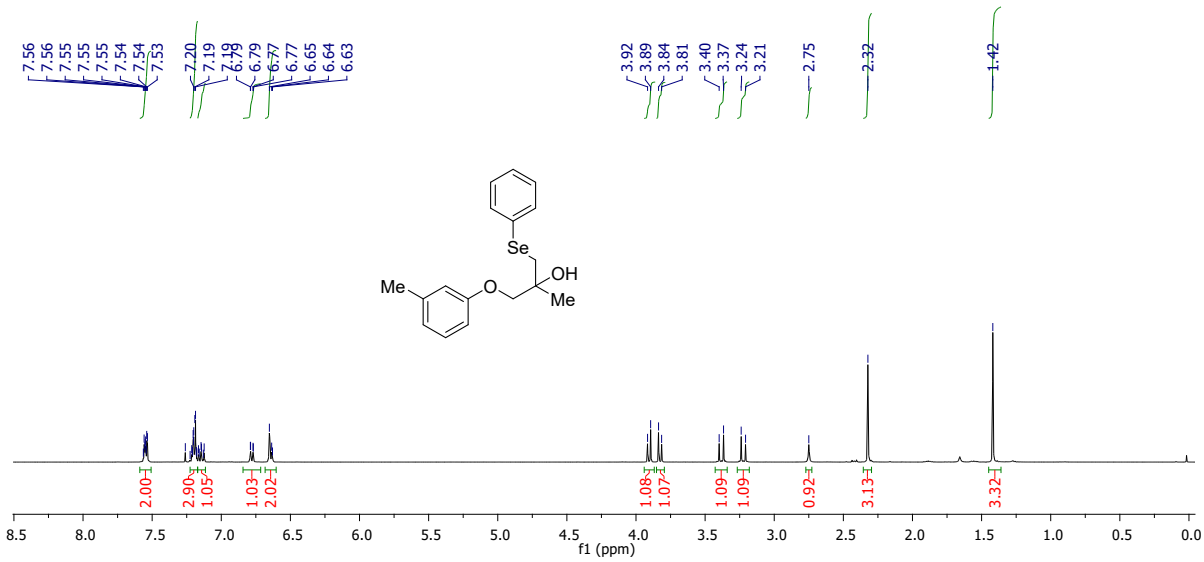
^{1}H-NMR (400 MHz) spectrum of **21** in $CDCl_3$



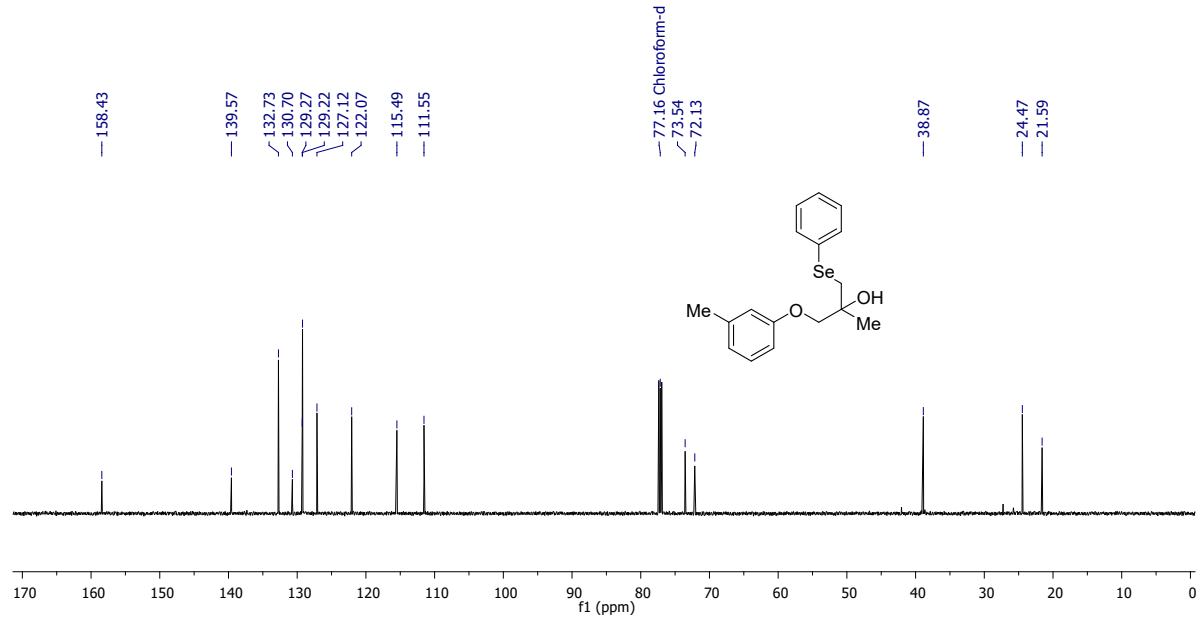
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **21** in $CDCl_3$



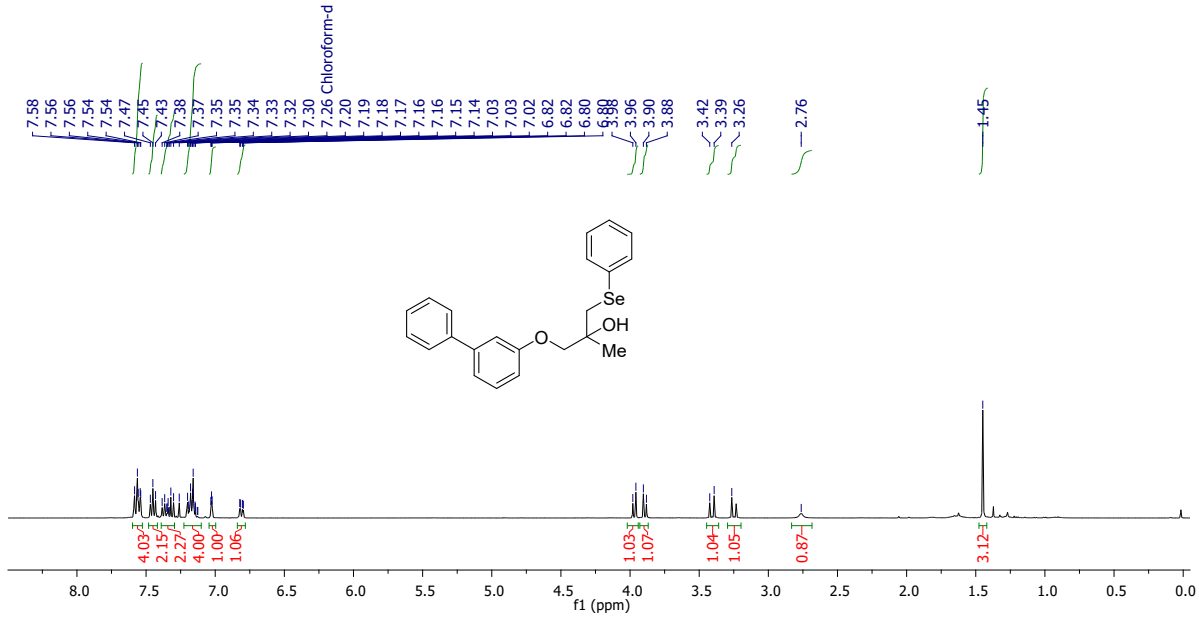
^{1}H-NMR (400 MHz) spectrum of **22** in $CDCl_3$



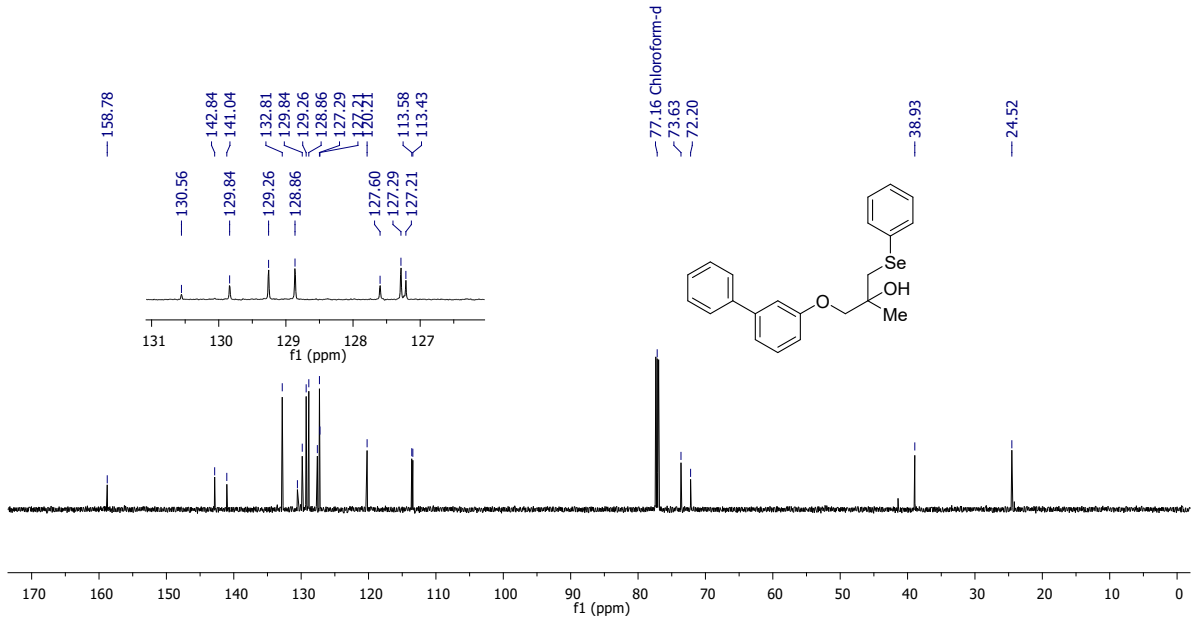
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **22** in $CDCl_3$



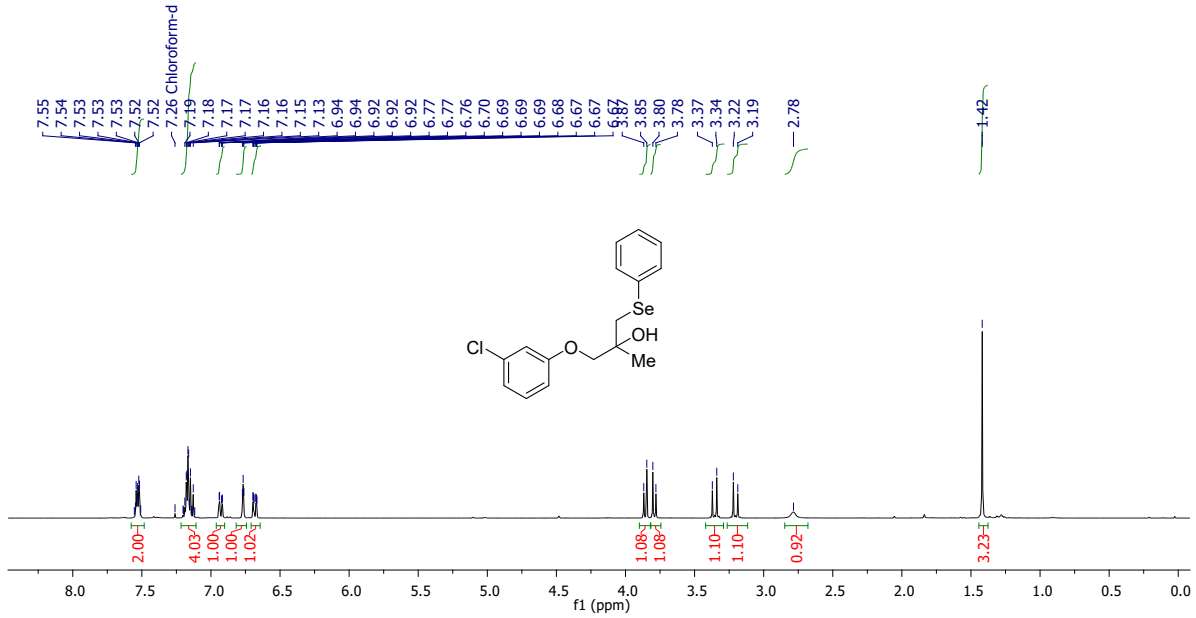
^{1}H-NMR (400 MHz) spectrum of **23** in $CDCl_3$



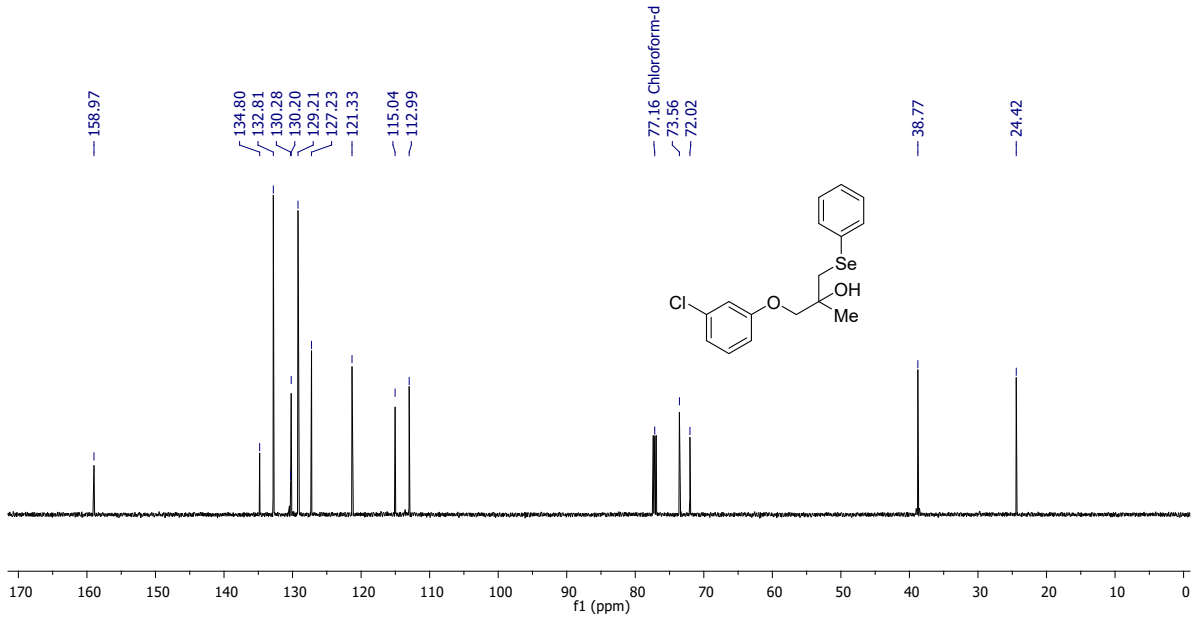
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **23** in $CDCl_3$



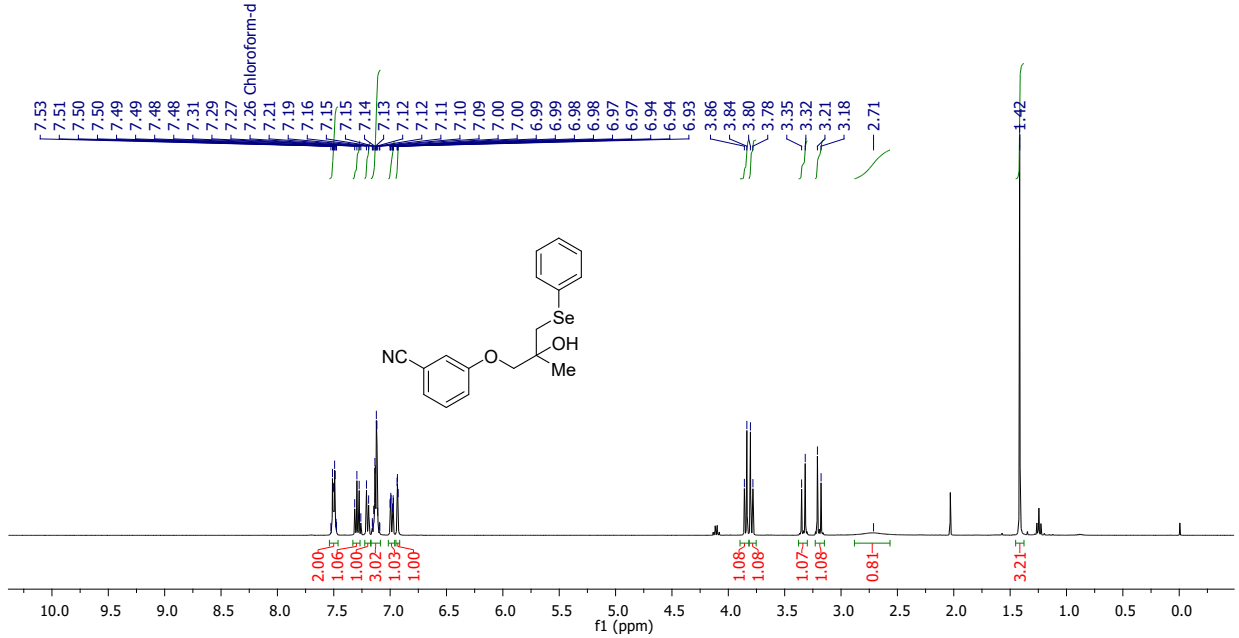
^{1}H-NMR (400 MHz) spectrum of **24** in $CDCl_3$



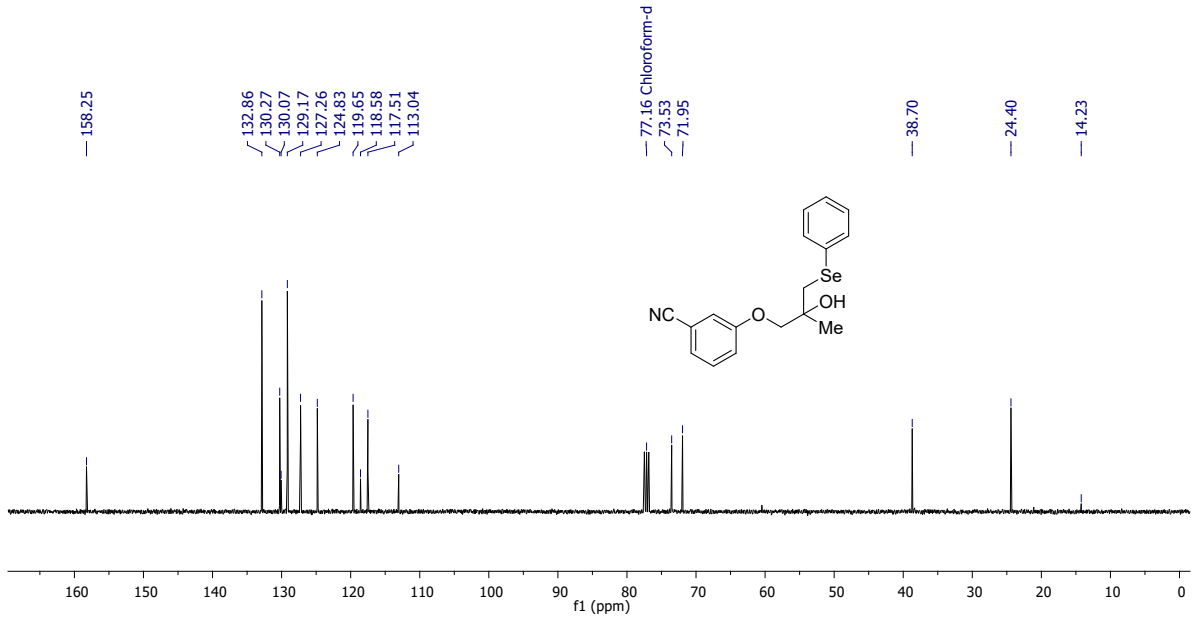
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **24** in $CDCl_3$



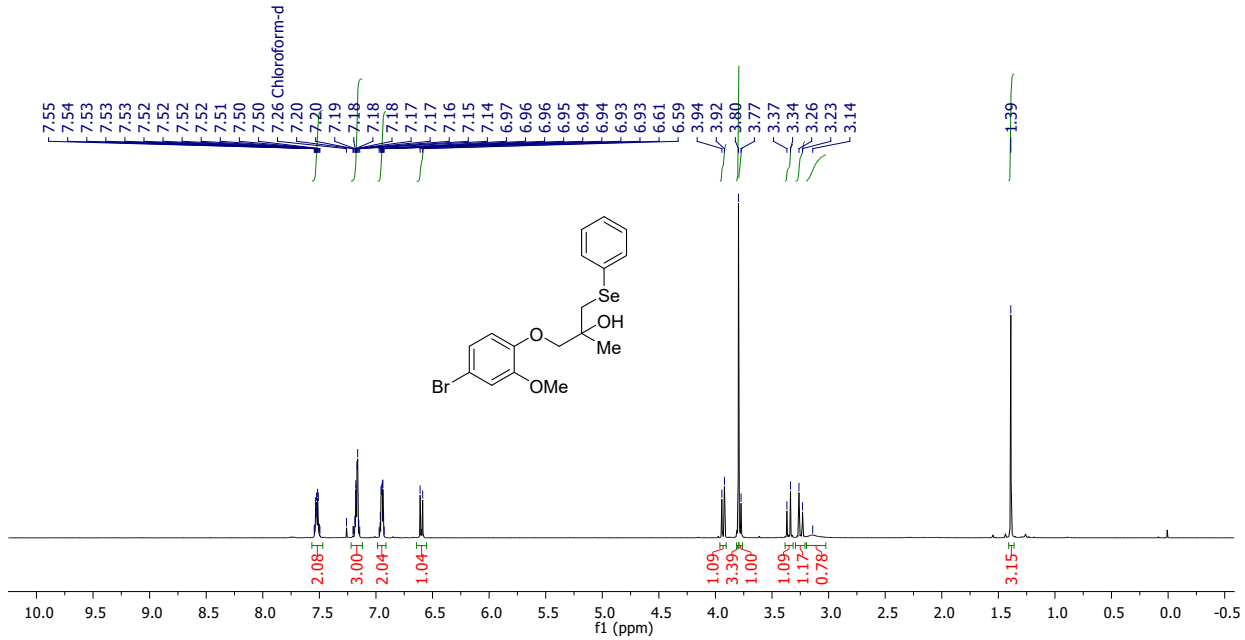
^{1}H-NMR (400 MHz) spectrum of **25** in $CDCl_3$



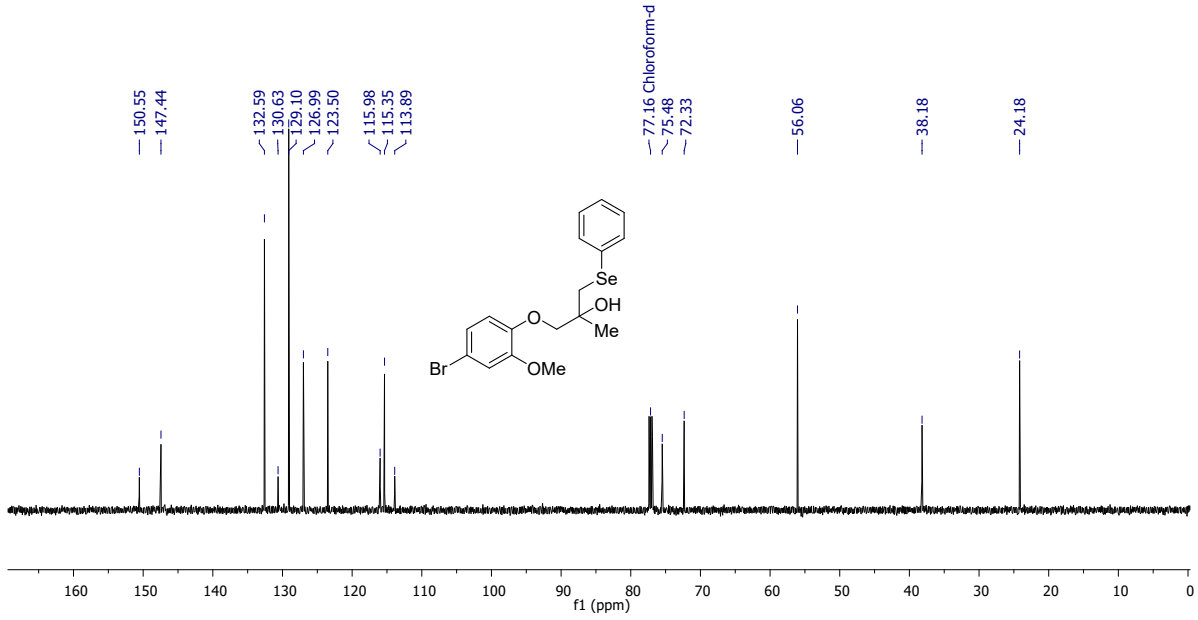
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **25** in $CDCl_3$



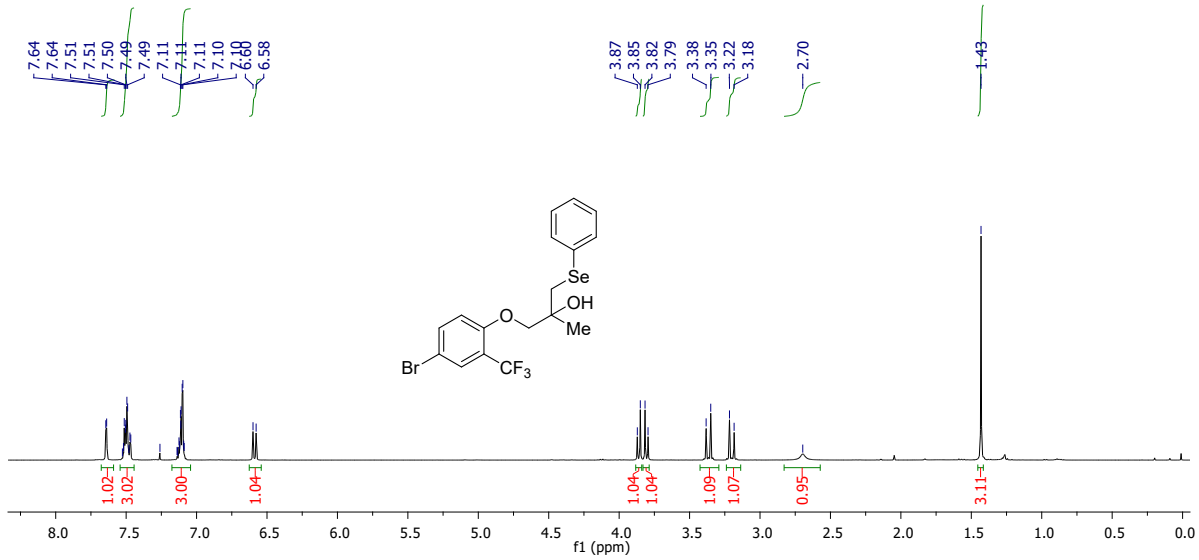
^{1}H-NMR (400 MHz) spectrum of **26** in $CDCl_3$



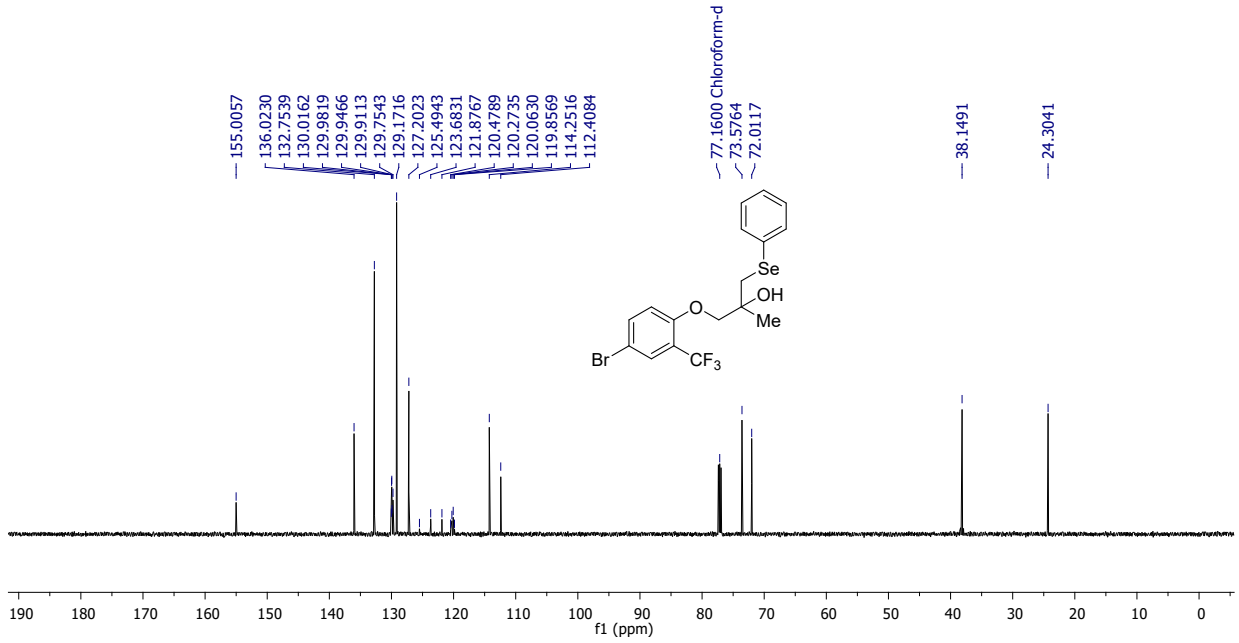
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **26** in $CDCl_3$



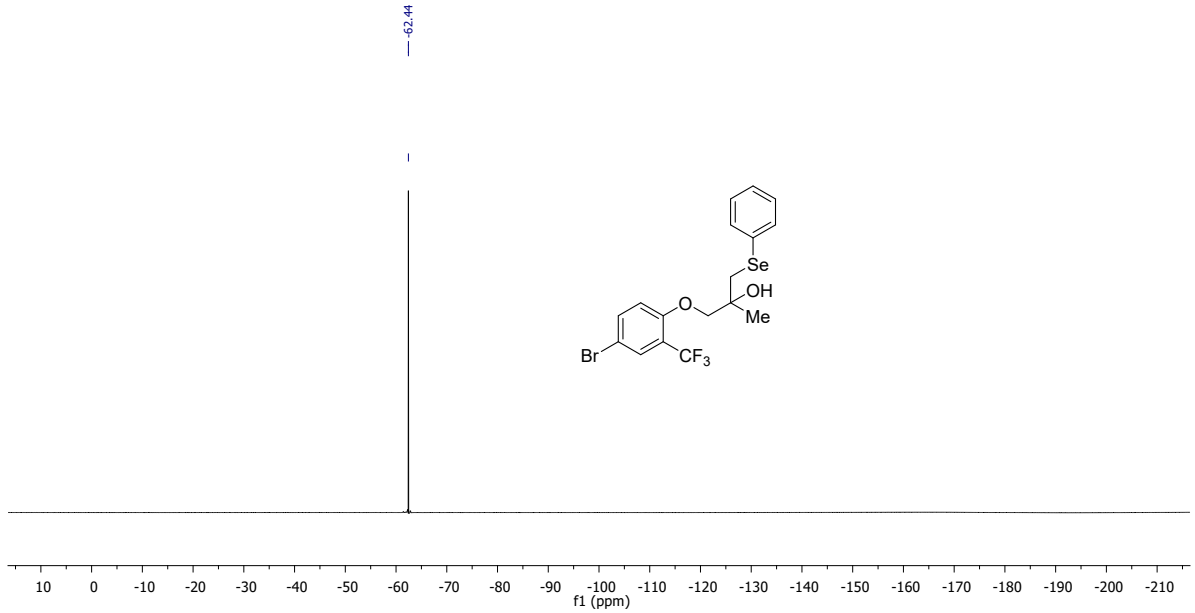
^{1}H-NMR (400 MHz) spectrum of **27** in $CDCl_3$



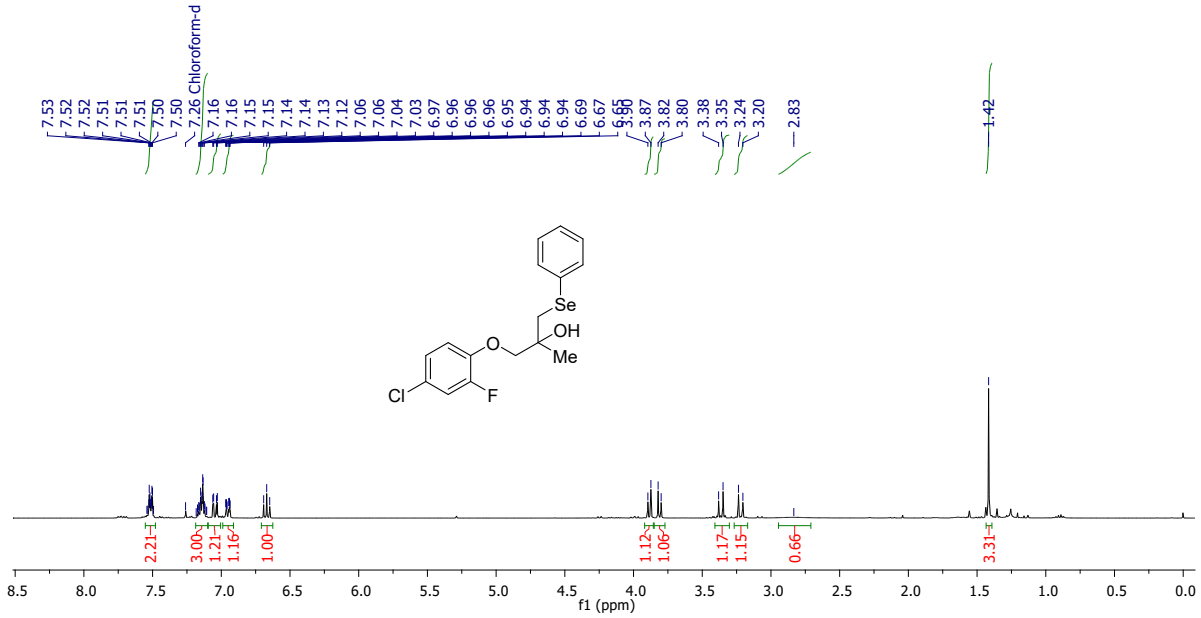
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **27** in $CDCl_3$



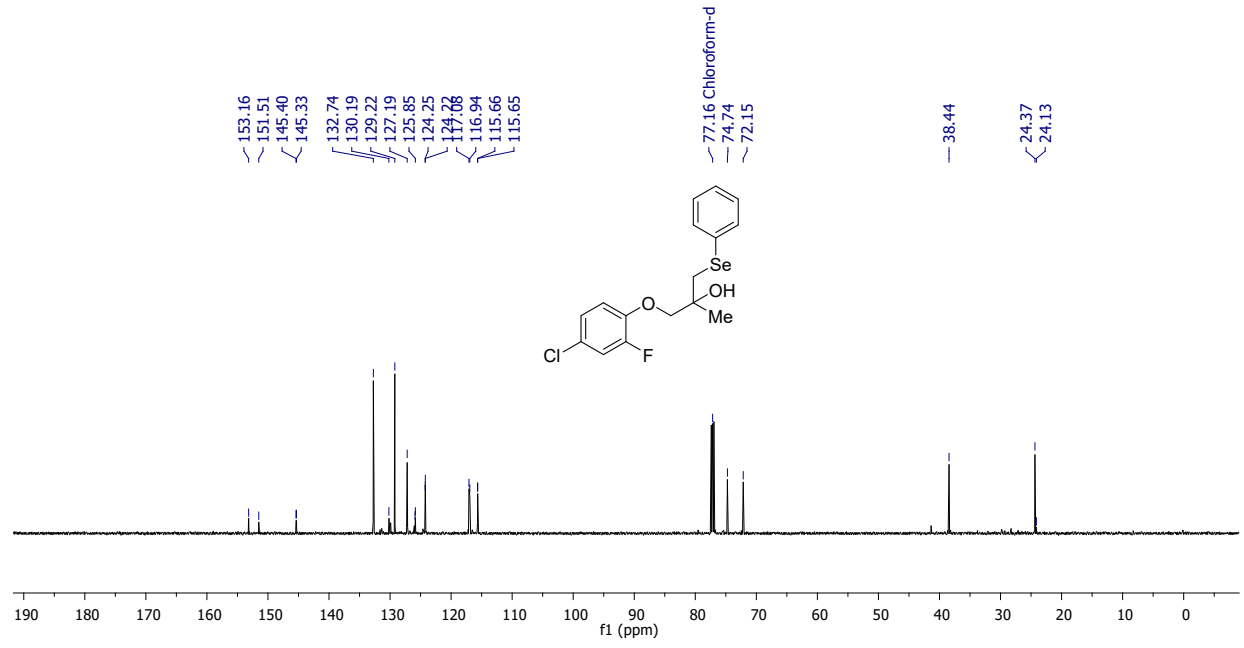
^{19}F NMR (565 MHz) spectrum of **27** in $CDCl_3$



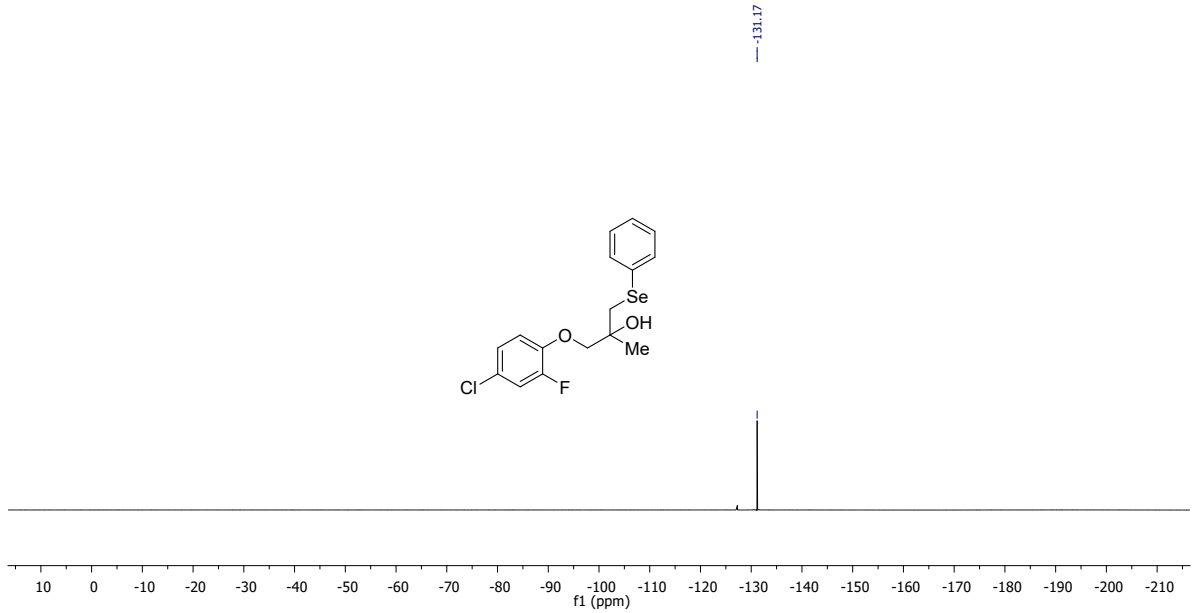
^{1}H-NMR (400 MHz) spectrum of **28** in $CDCl_3$



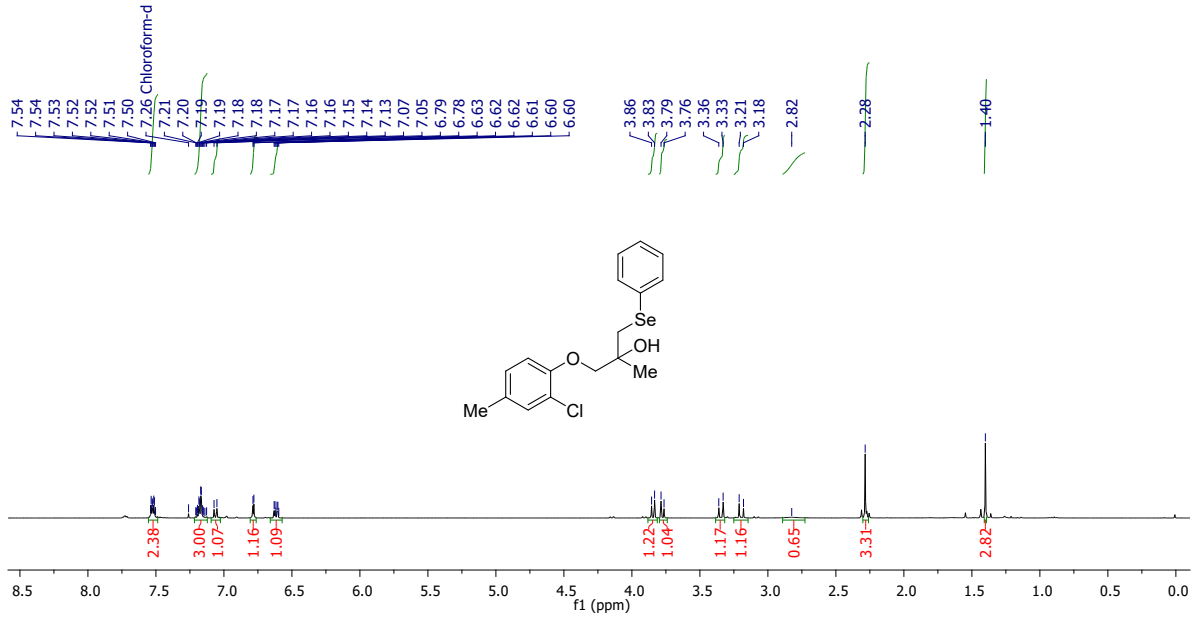
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **28** in $CDCl_3$



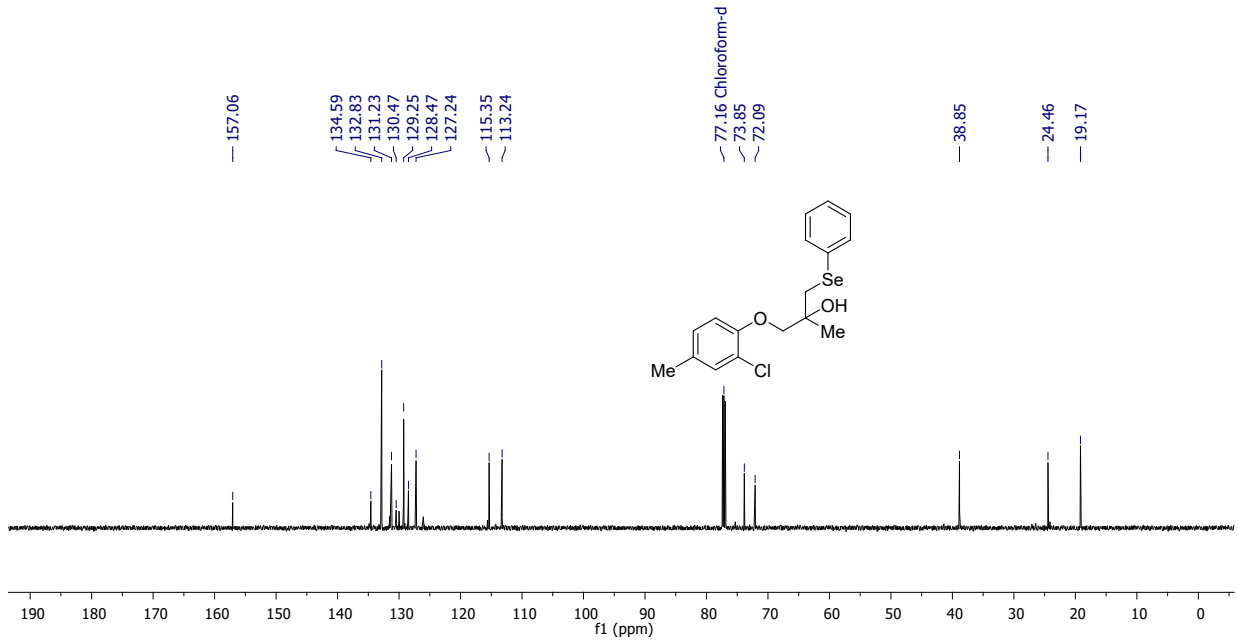
^{19}F NMR (565 MHz) spectrum of **28** in $CDCl_3$



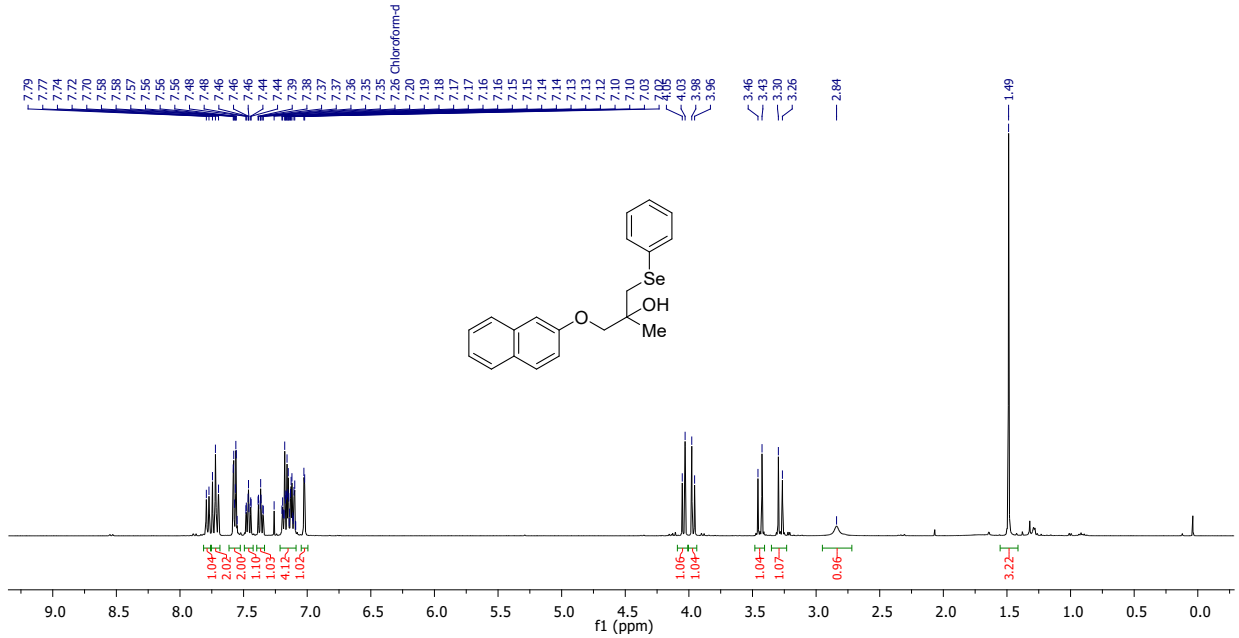
^{1}H-NMR (400 MHz) spectrum of **29** in $CDCl_3$



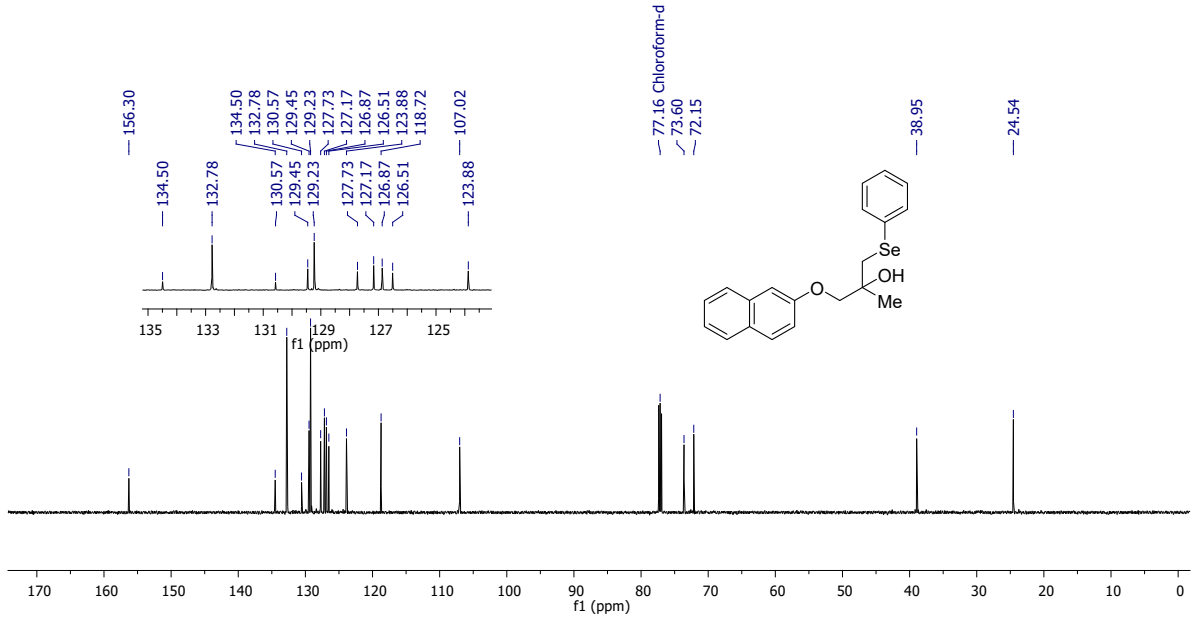
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **29** in $CDCl_3$



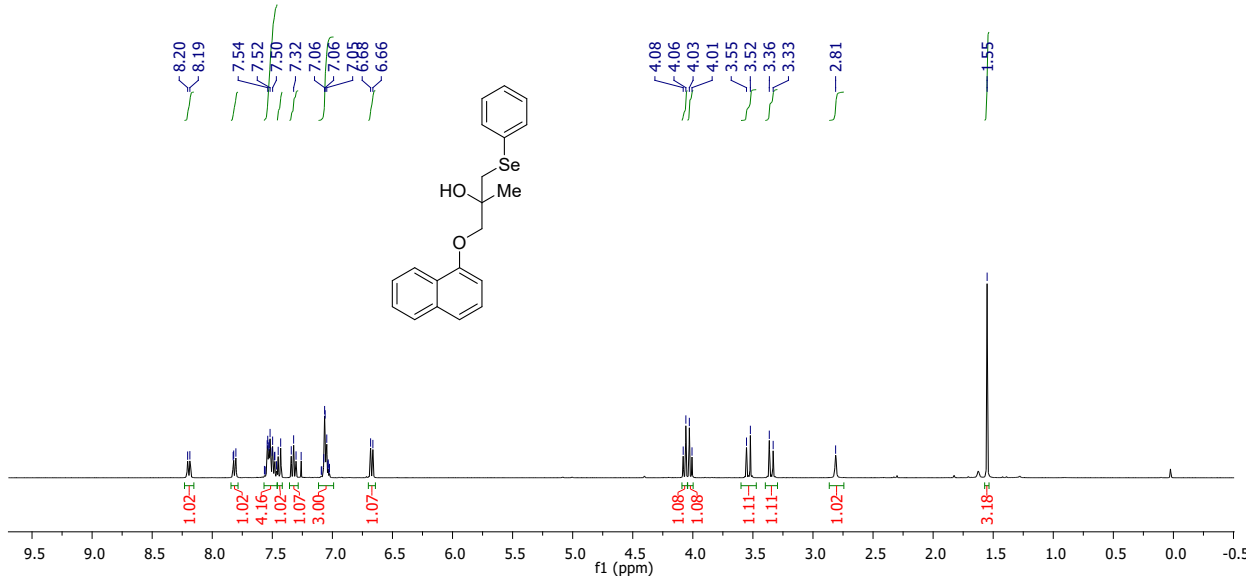
^{1}H-NMR (400 MHz) spectrum of **30** in $CDCl_3$



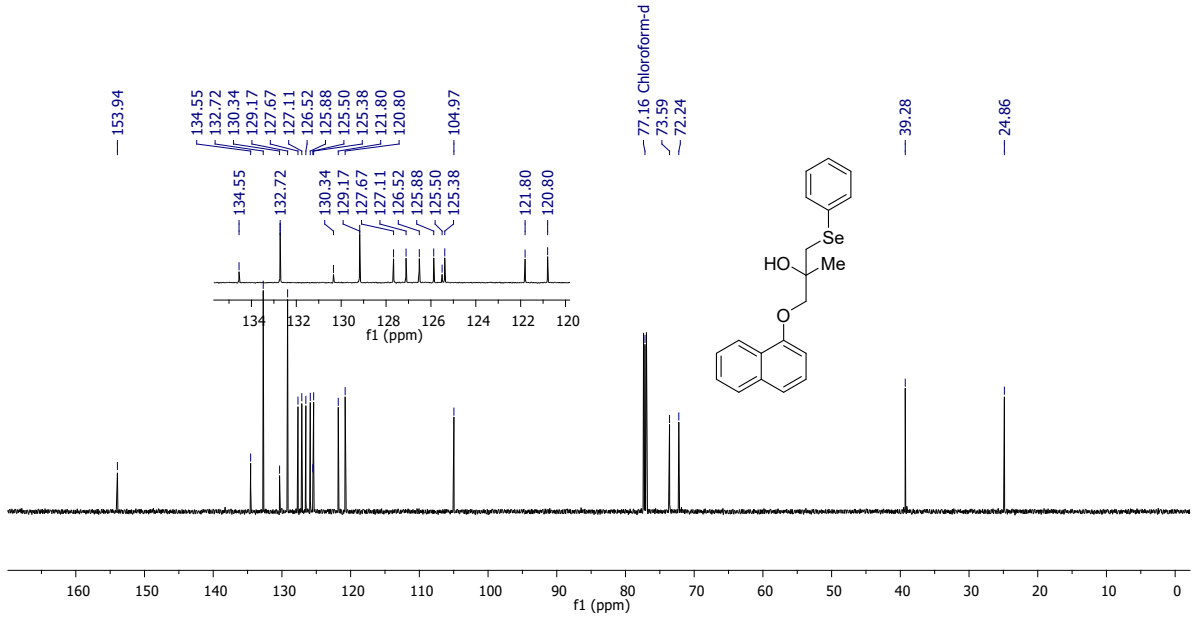
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **30** in $CDCl_3$



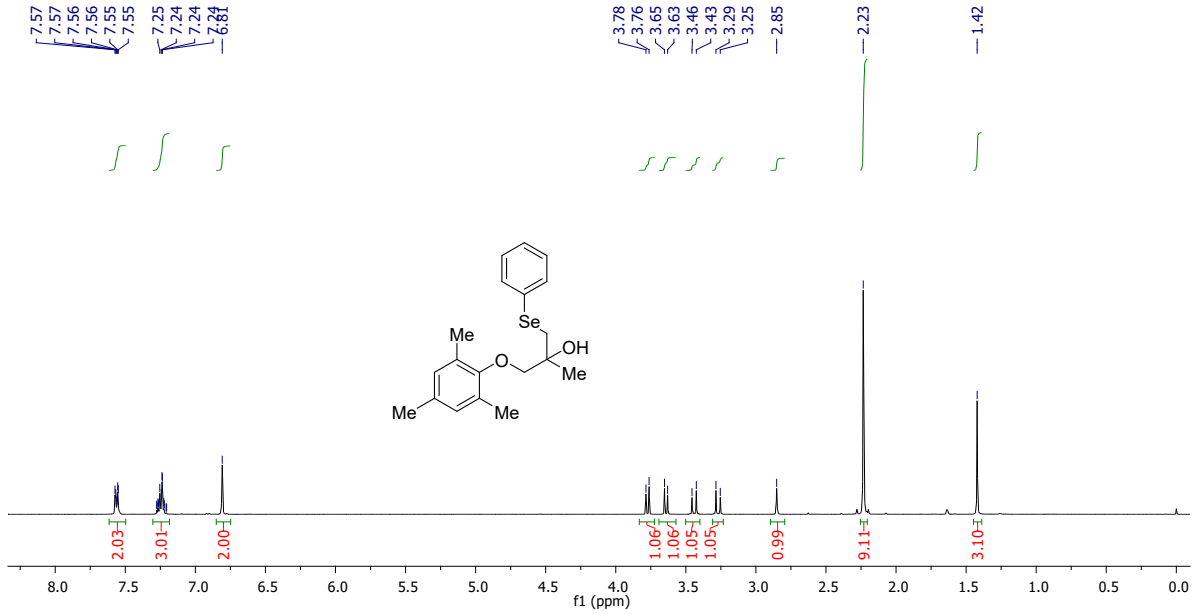
^{1}H-NMR (400 MHz) spectrum of **31** in $CDCl_3$



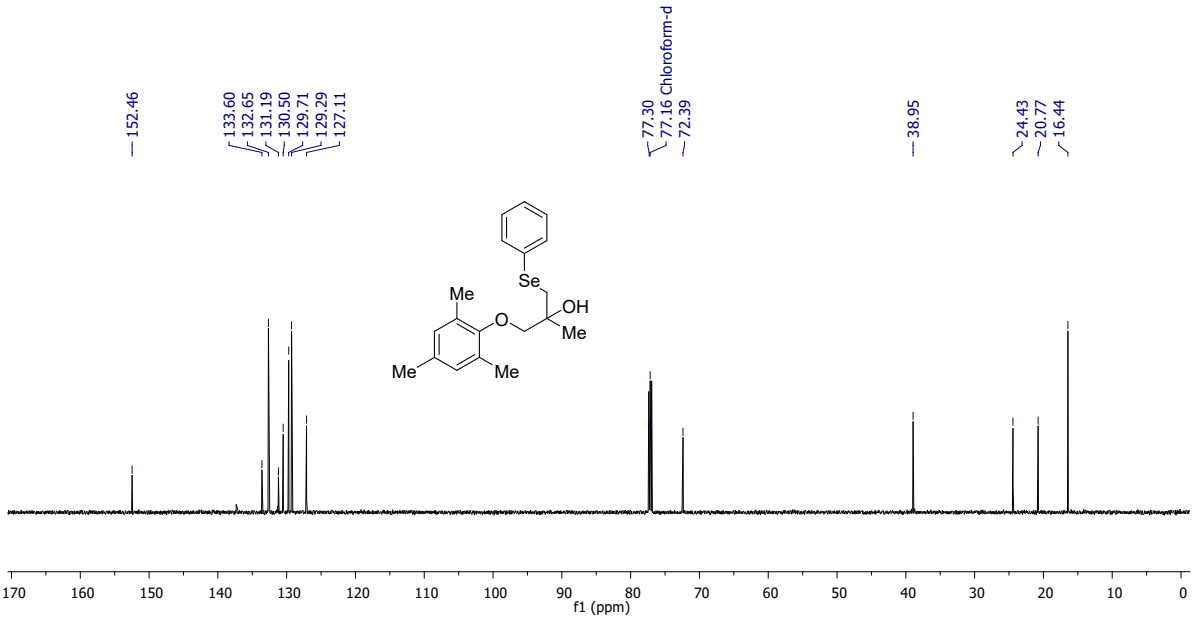
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **31** in $CDCl_3$



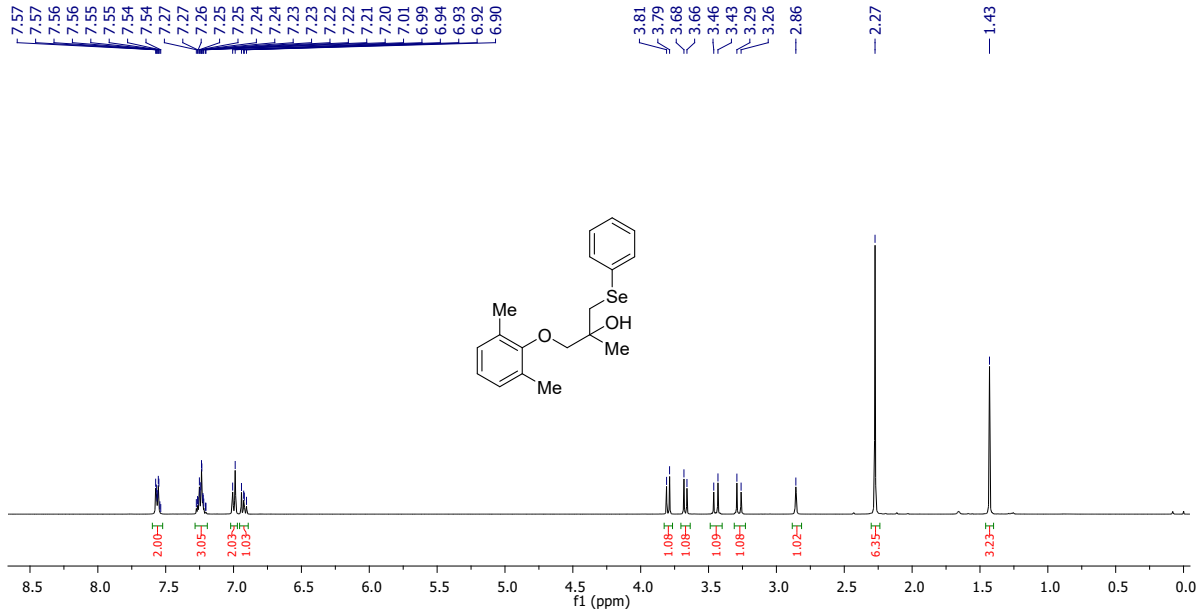
^{1}H-NMR (400 MHz) spectrum of **32** in $CDCl_3$



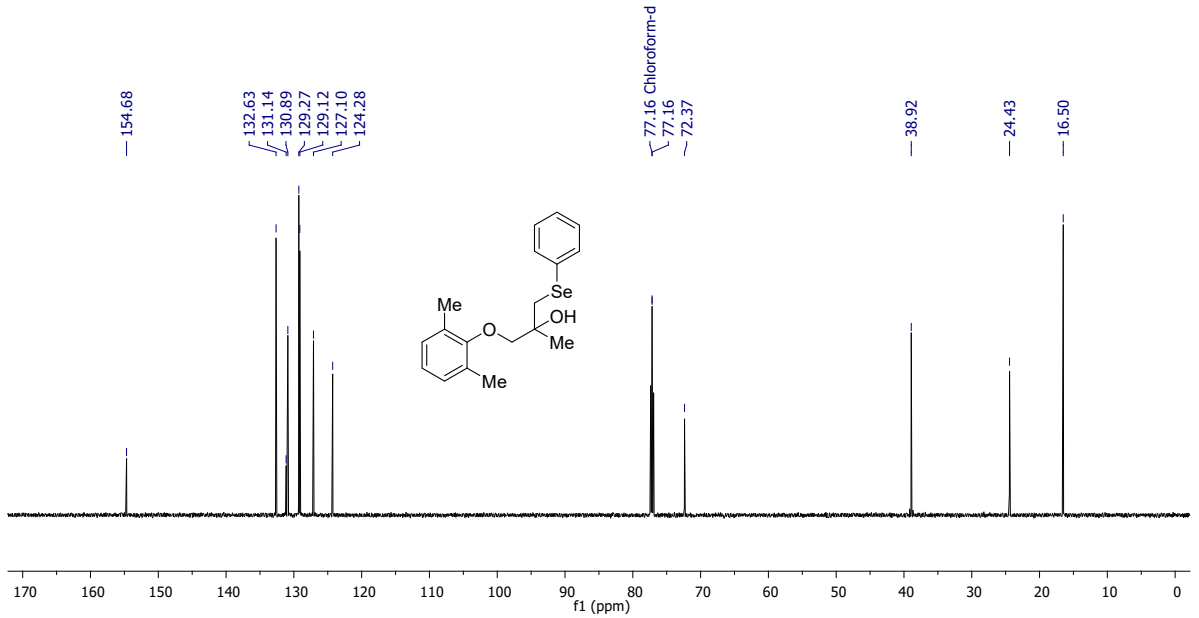
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **32** in $CDCl_3$



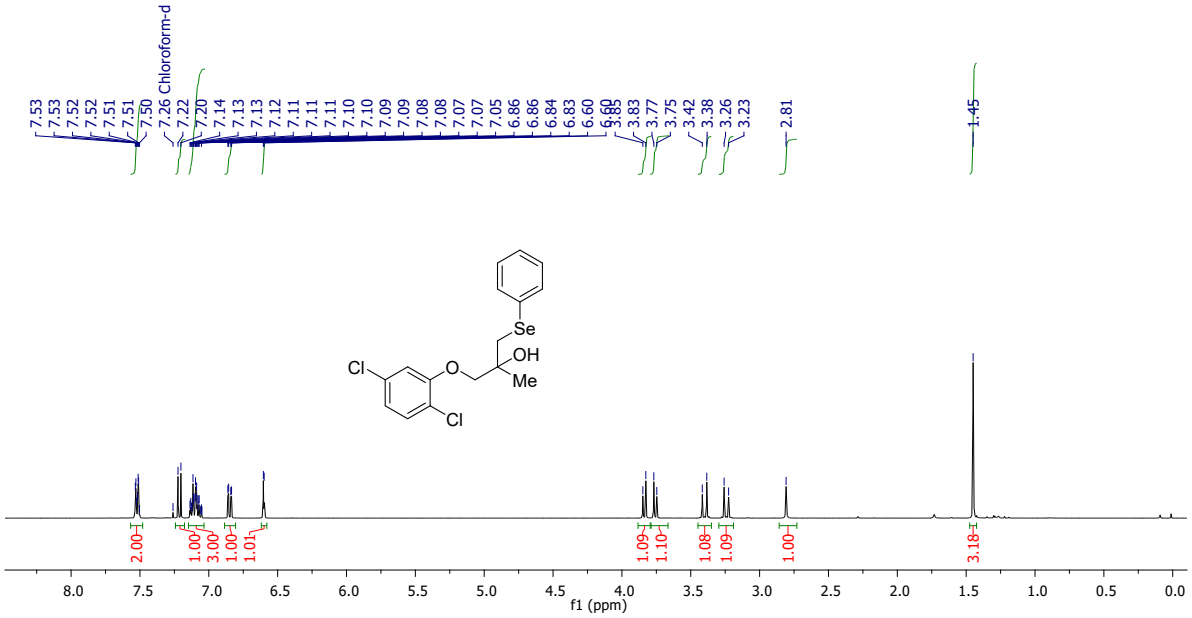
^{1}H-NMR (400 MHz) spectrum of **33** in $CDCl_3$



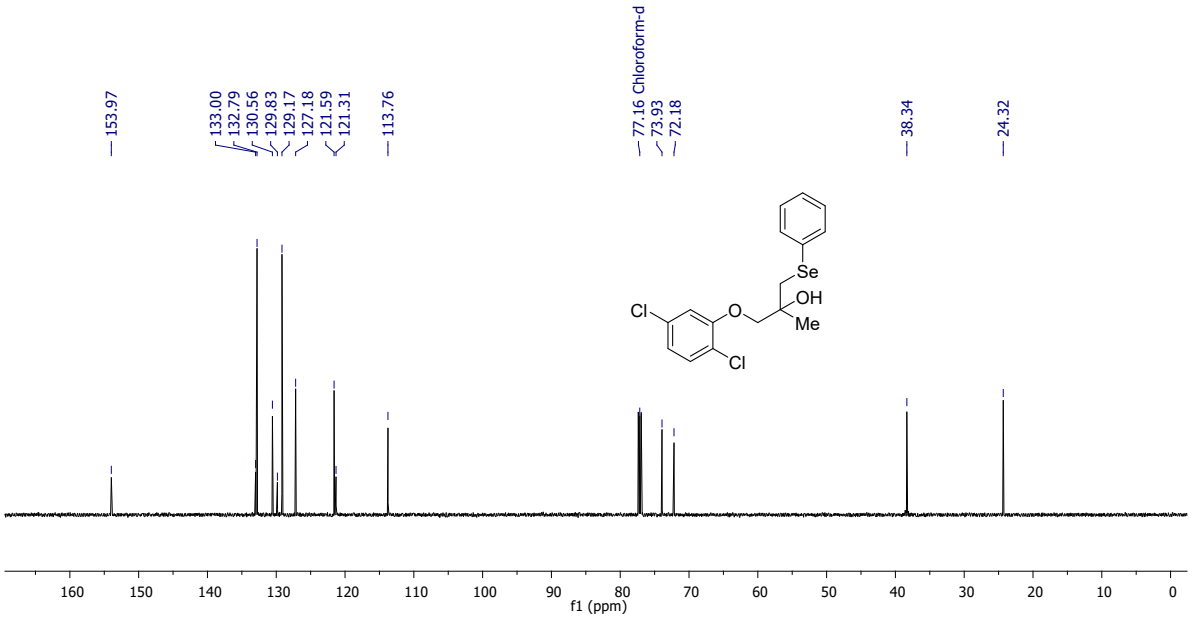
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **33** in $CDCl_3$



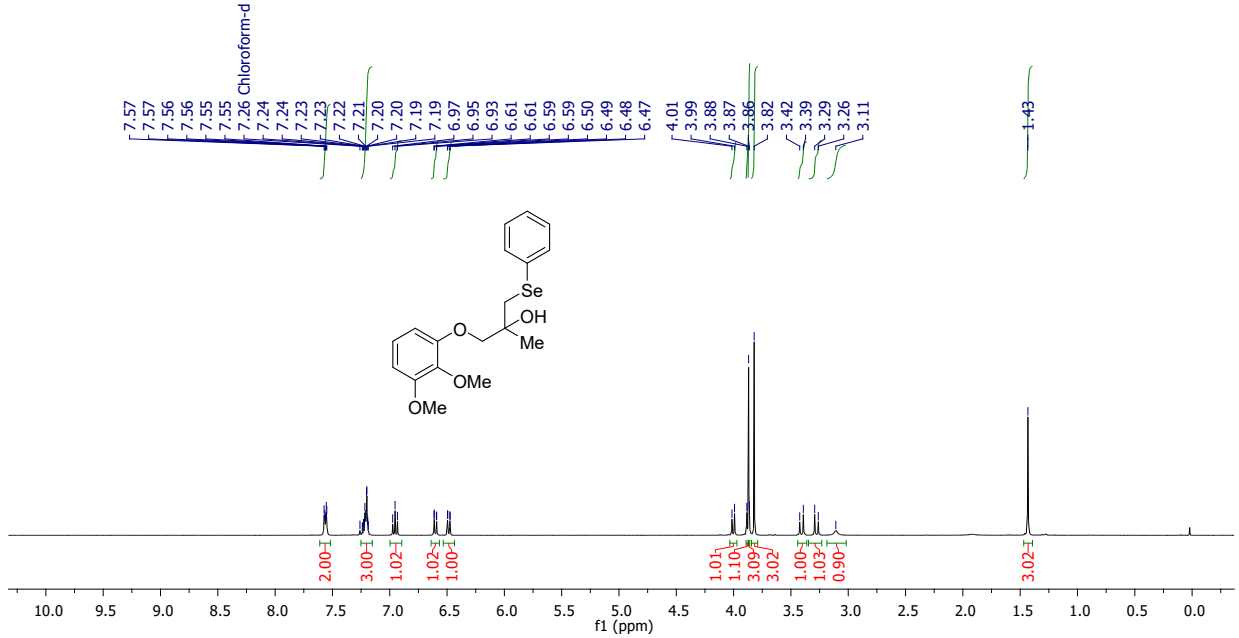
^{1}H-NMR (400 MHz) spectrum of **34** in $CDCl_3$



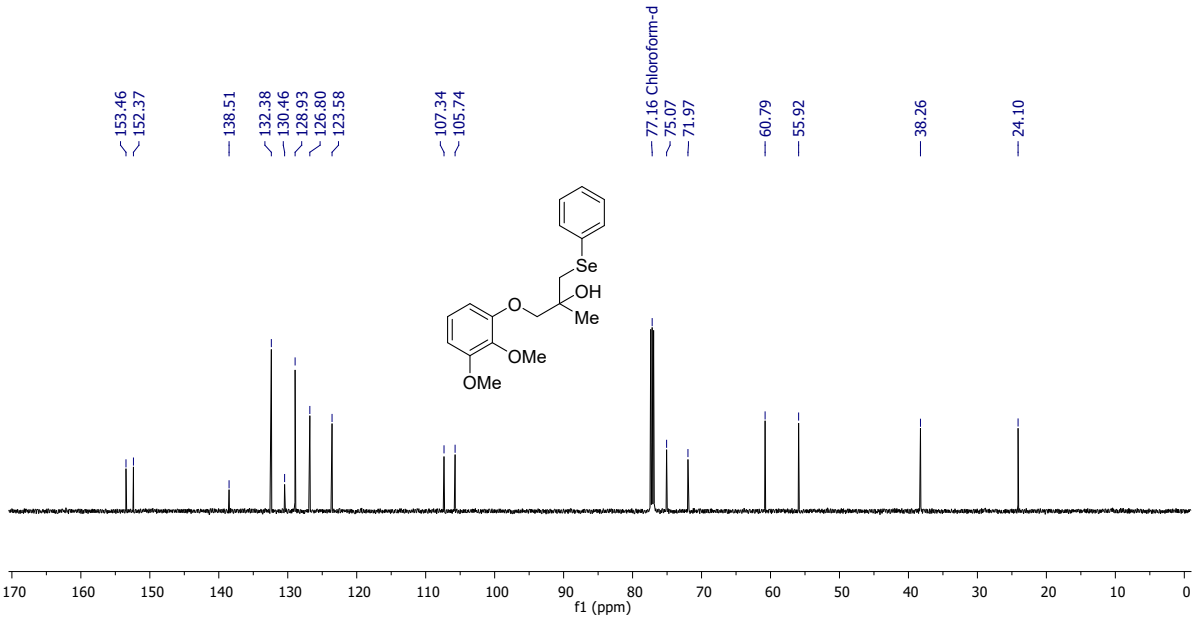
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **34** in $CDCl_3$



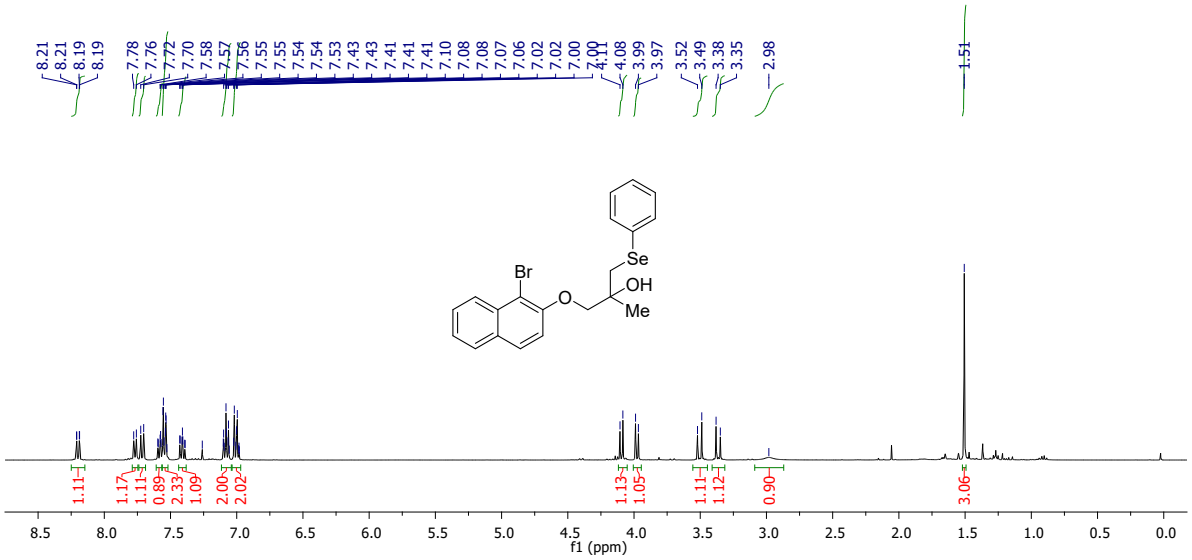
^{1}H-NMR (400 MHz) spectrum of **35** in $CDCl_3$



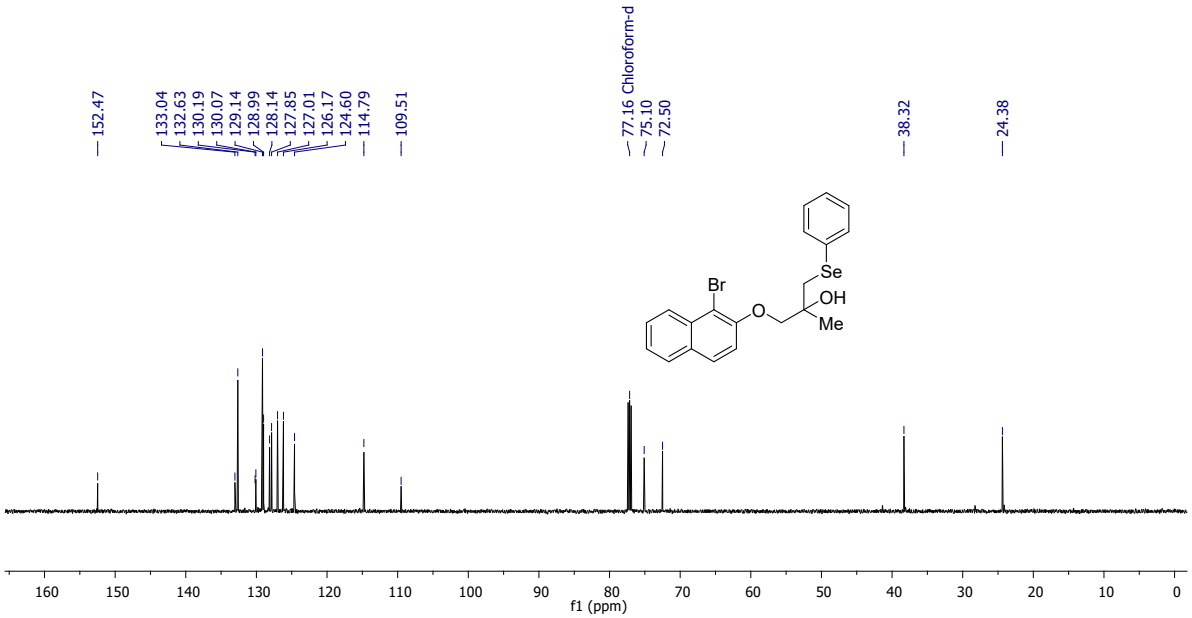
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **35** in $CDCl_3$



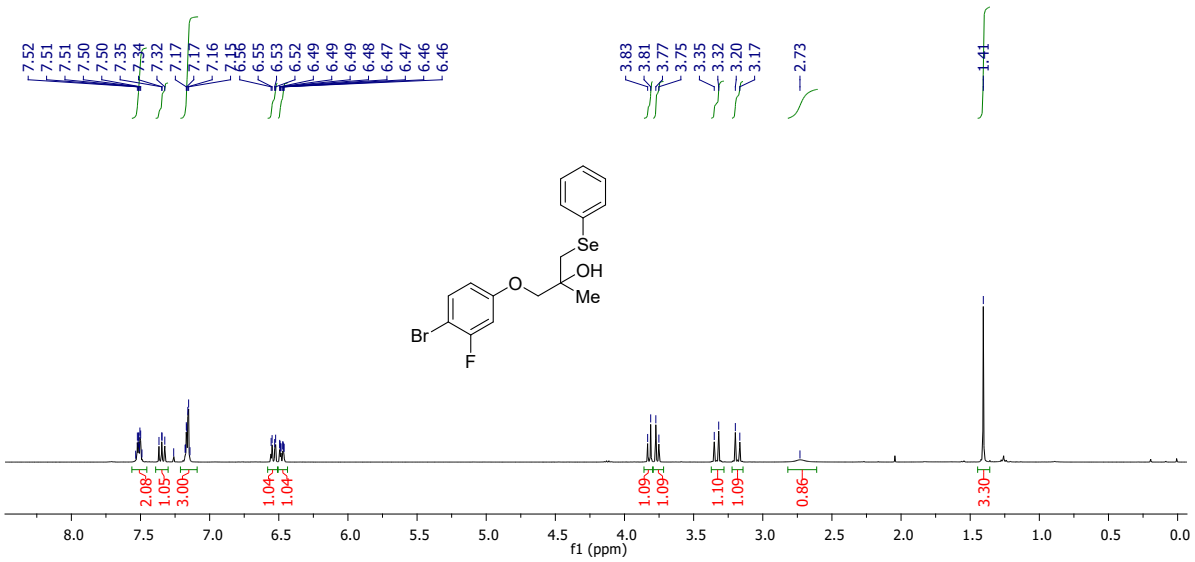
^{1}H-NMR (400 MHz) spectrum of **36** in $CDCl_3$



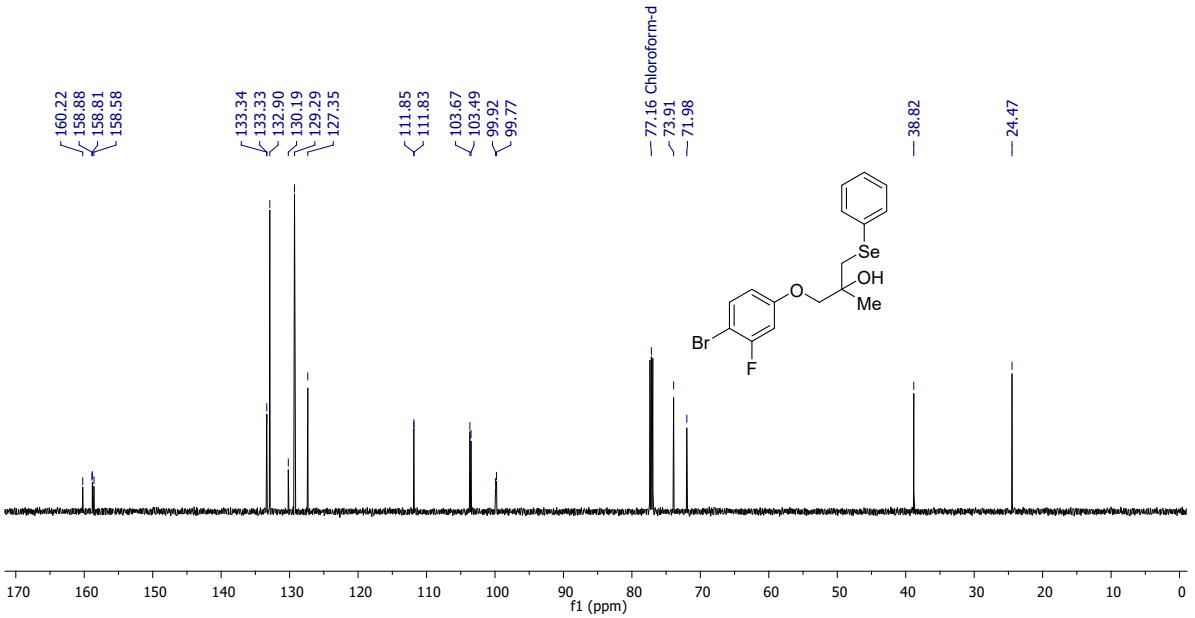
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **36** in $CDCl_3$



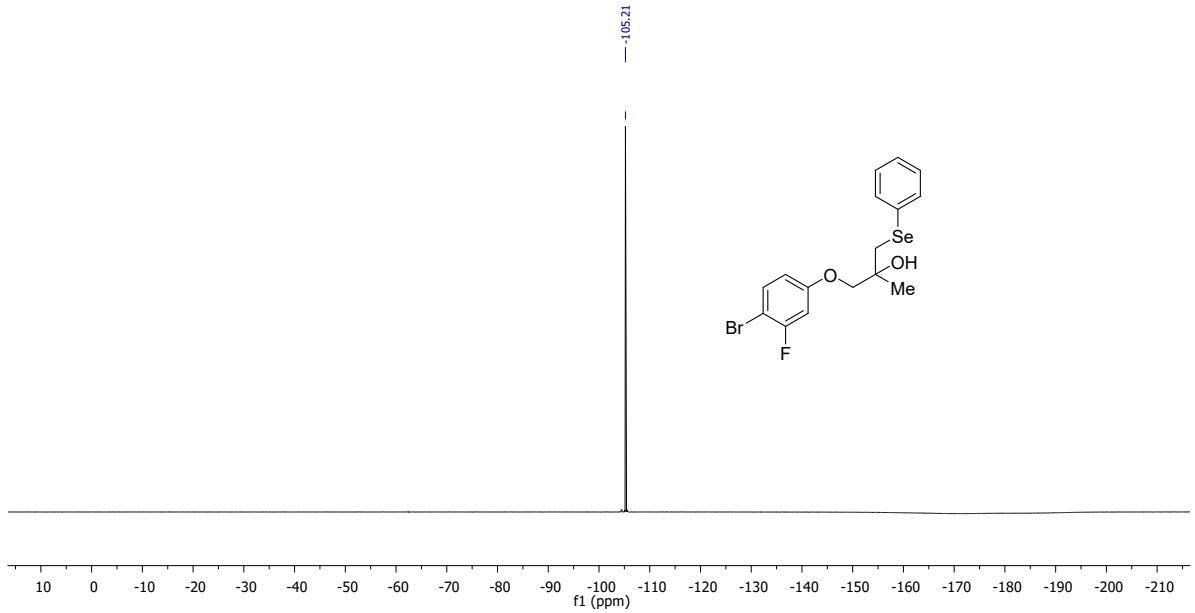
^{1}H-NMR (400 MHz) spectrum of **37** in $CDCl_3$



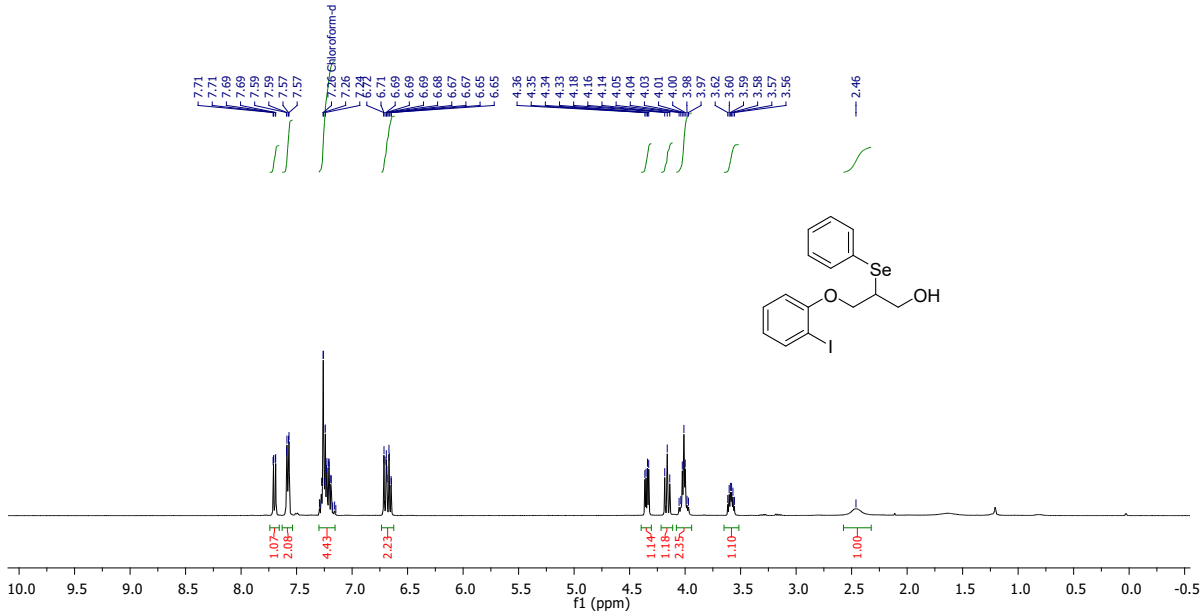
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **37** in $CDCl_3$



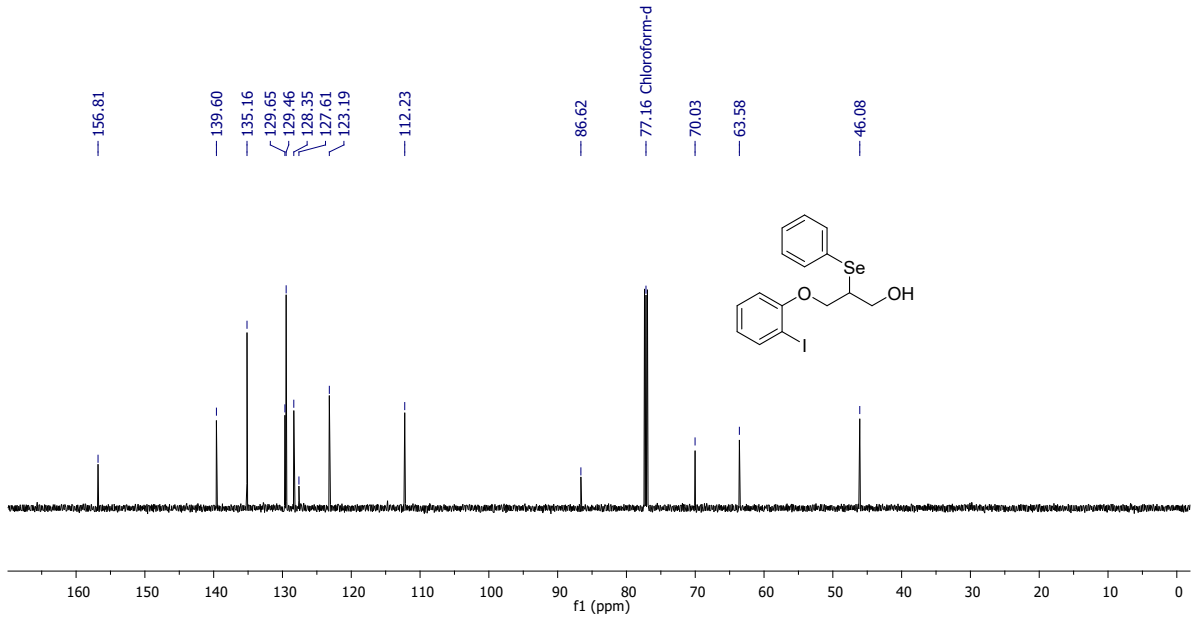
^{19}F NMR (565 MHz) spectrum of **37** in $CDCl_3$



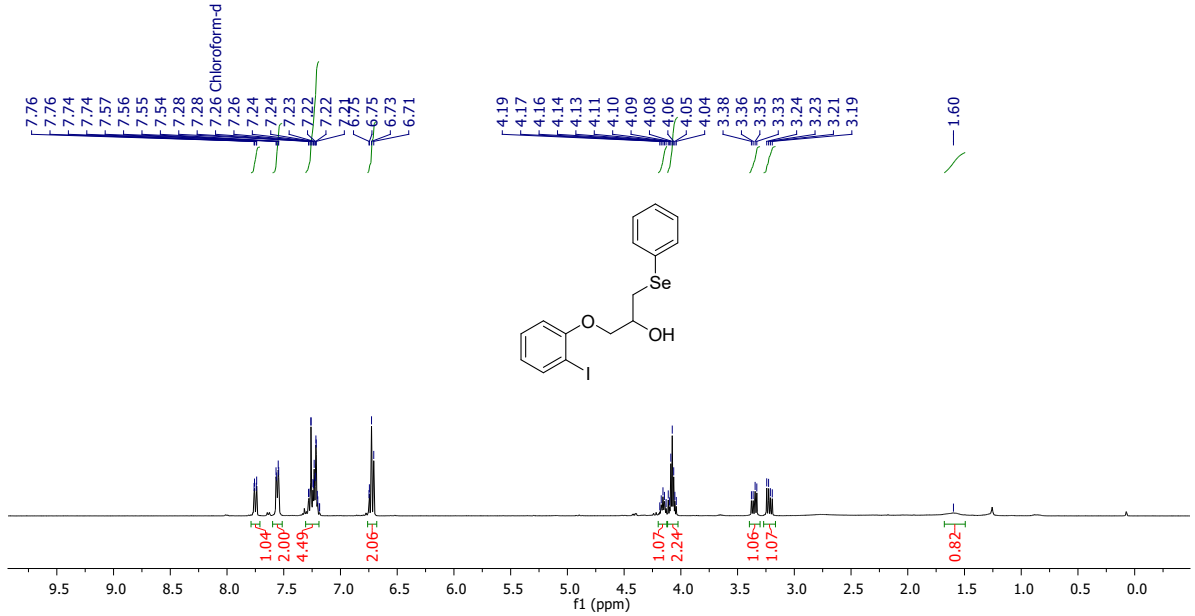
^{1}H-NMR (400 MHz) spectrum of **38** in $CDCl_3$



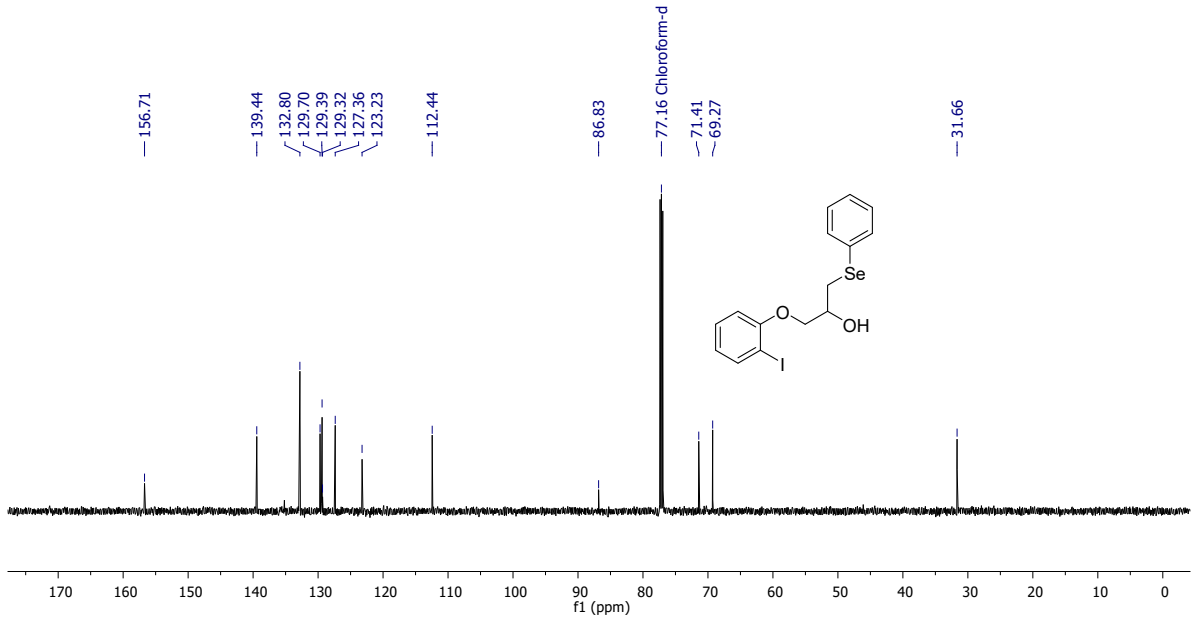
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **38** in $CDCl_3$



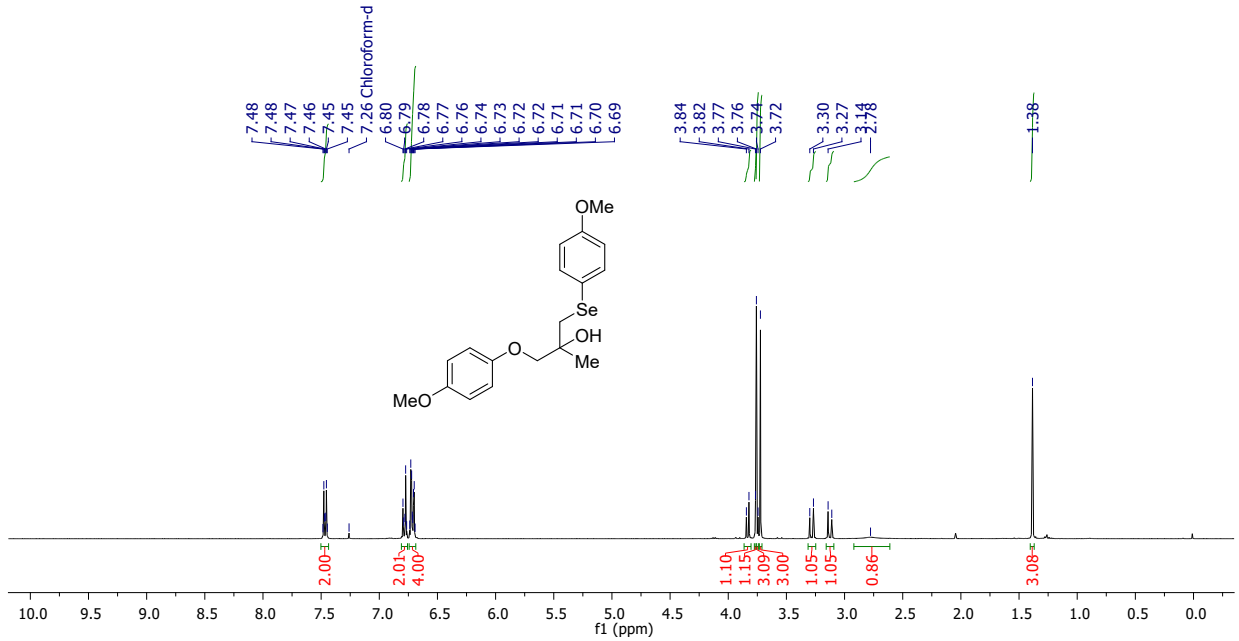
^{1}H-NMR (400 MHz) spectrum of **38'** in $CDCl_3$



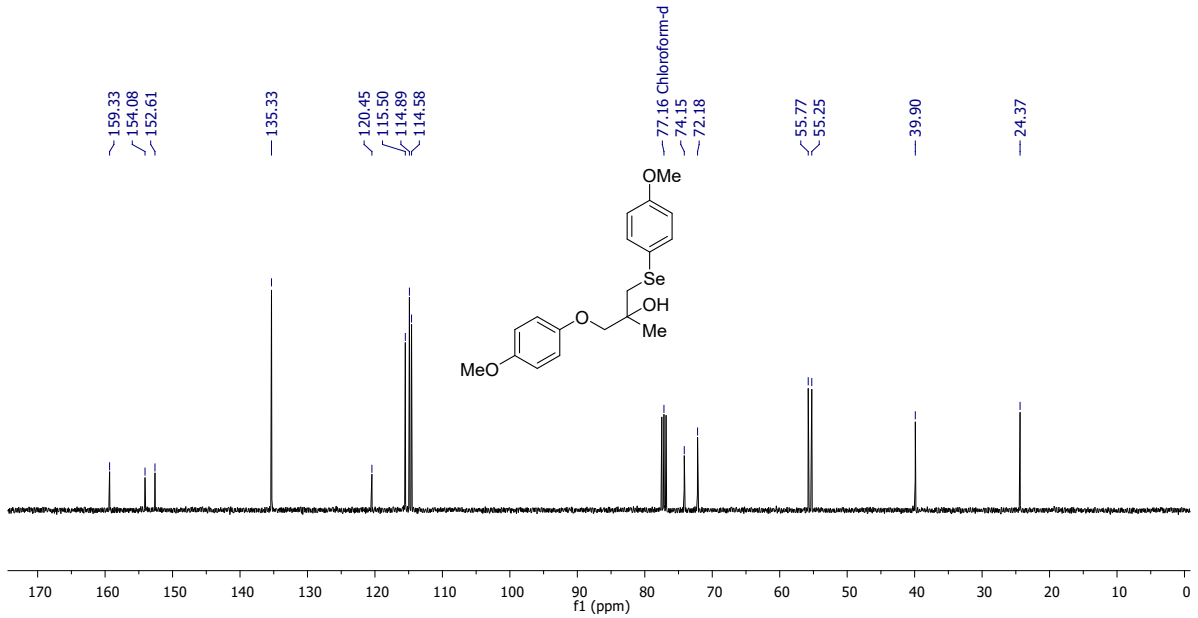
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **38'** in $CDCl_3$



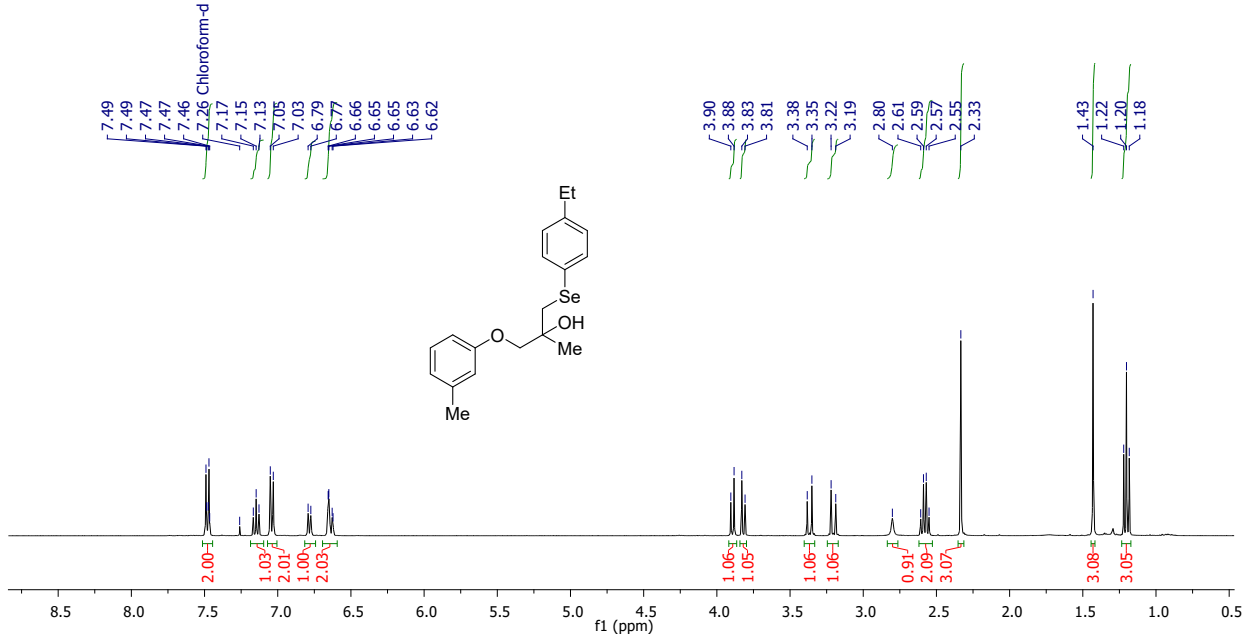
^{1}H-NMR (400 MHz) spectrum of **39** in $CDCl_3$



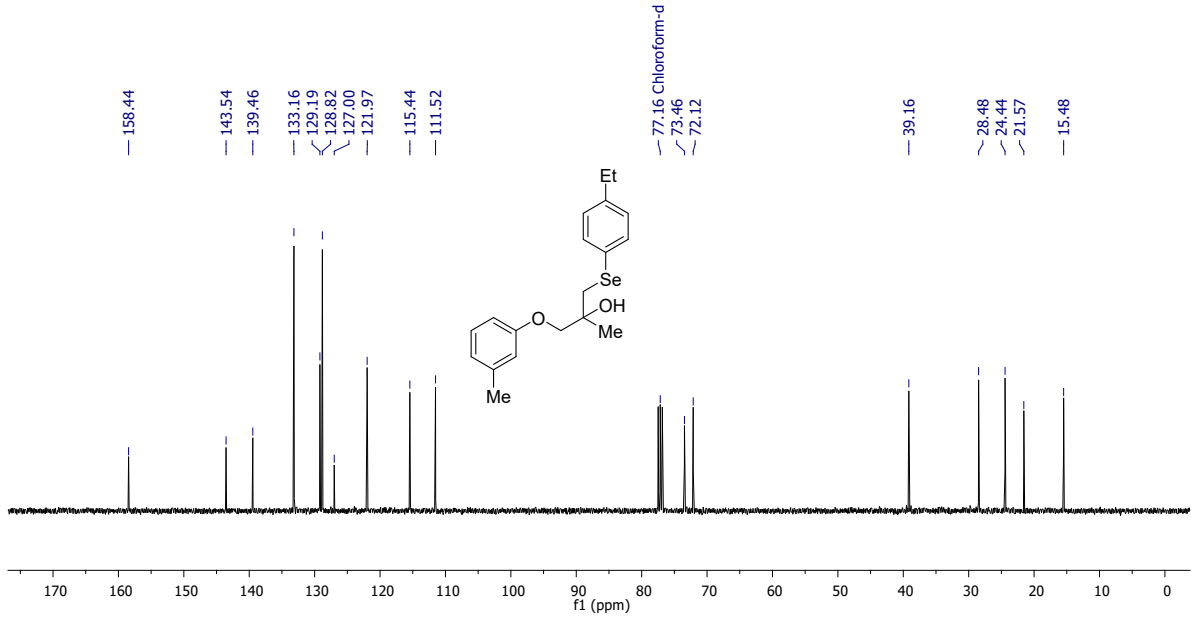
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **39** in $CDCl_3$



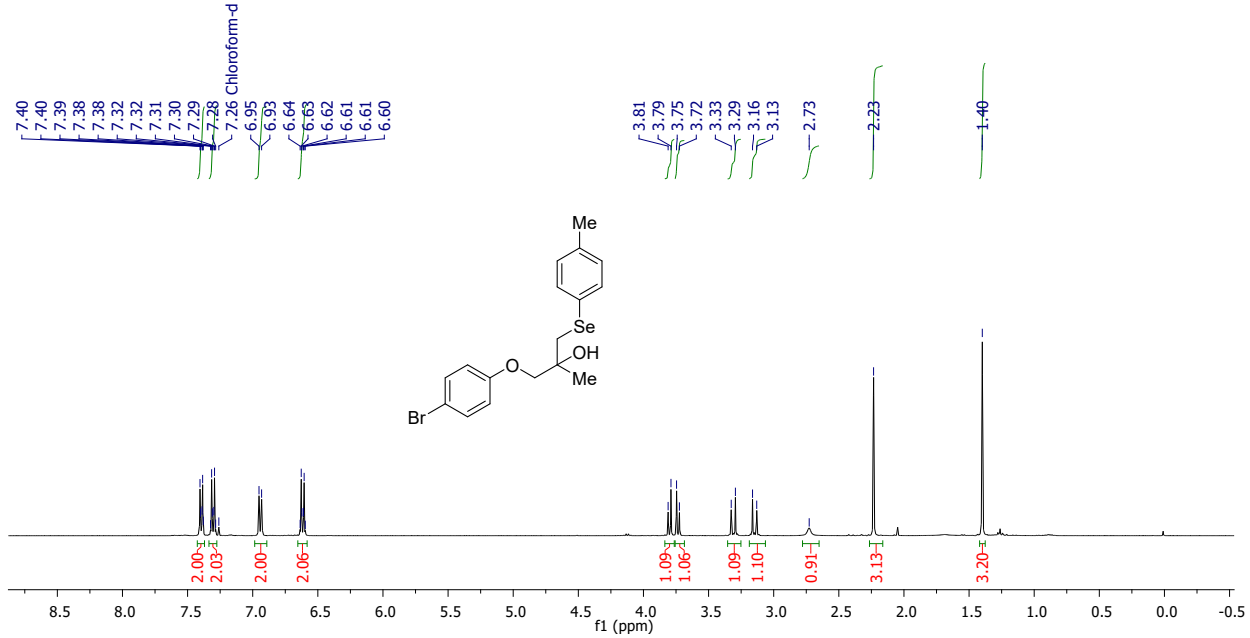
^{1}H-NMR (400 MHz) spectrum of **42** in $CDCl_3$



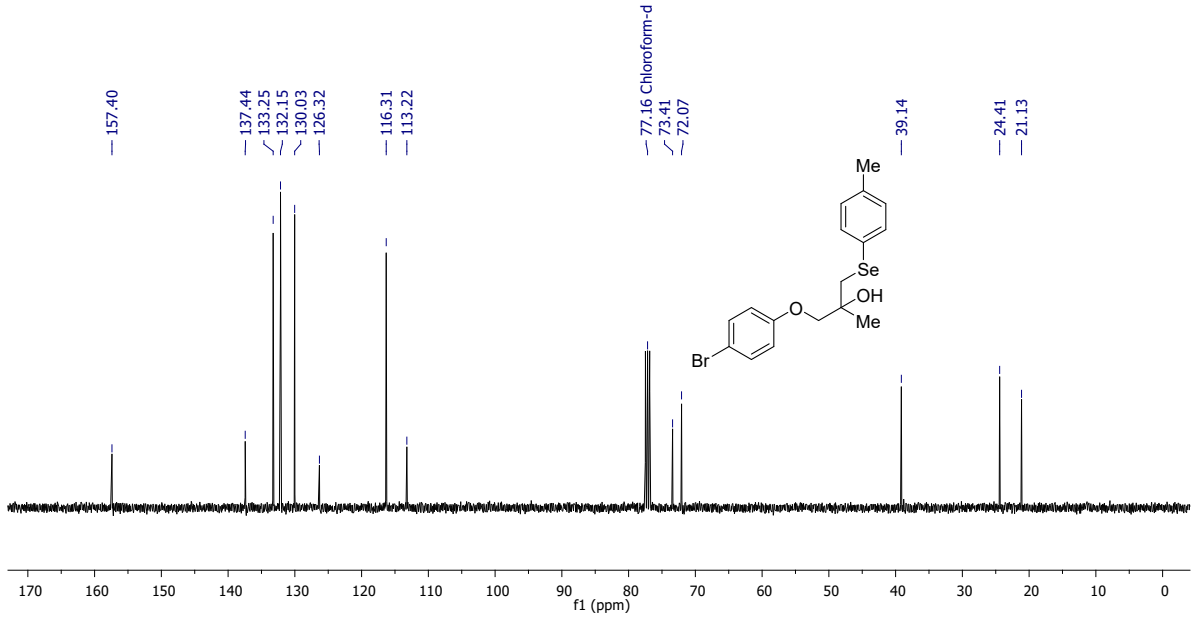
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **42** in $CDCl_3$



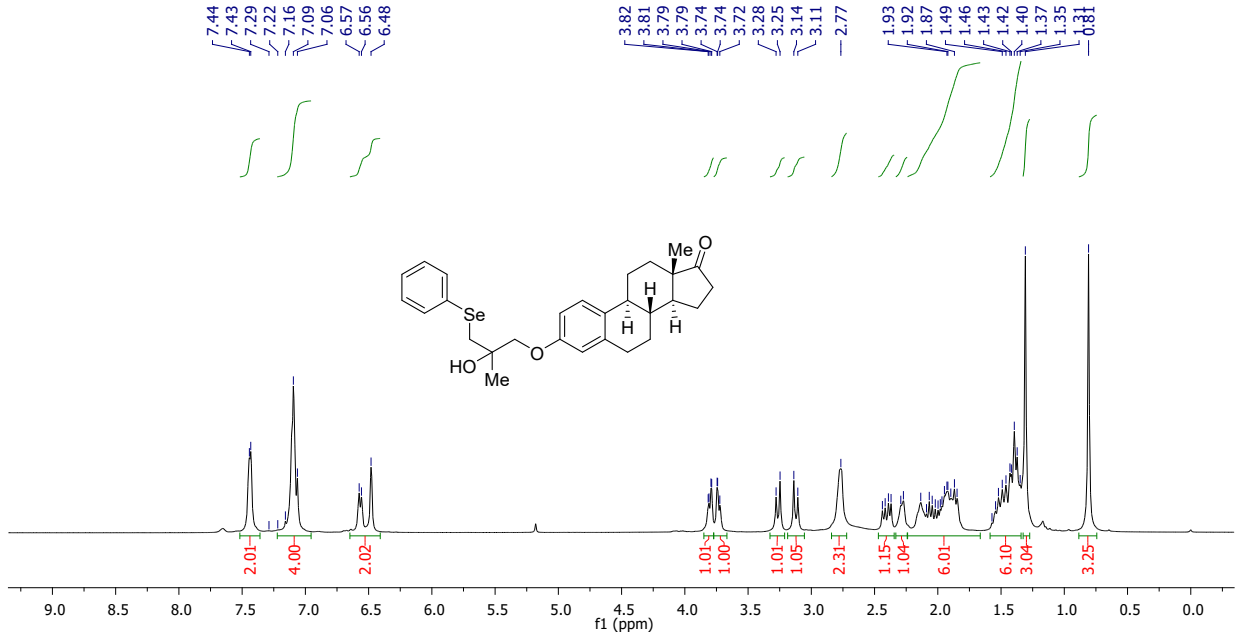
^{1}H-NMR (400 MHz) spectrum of **43** in $CDCl_3$



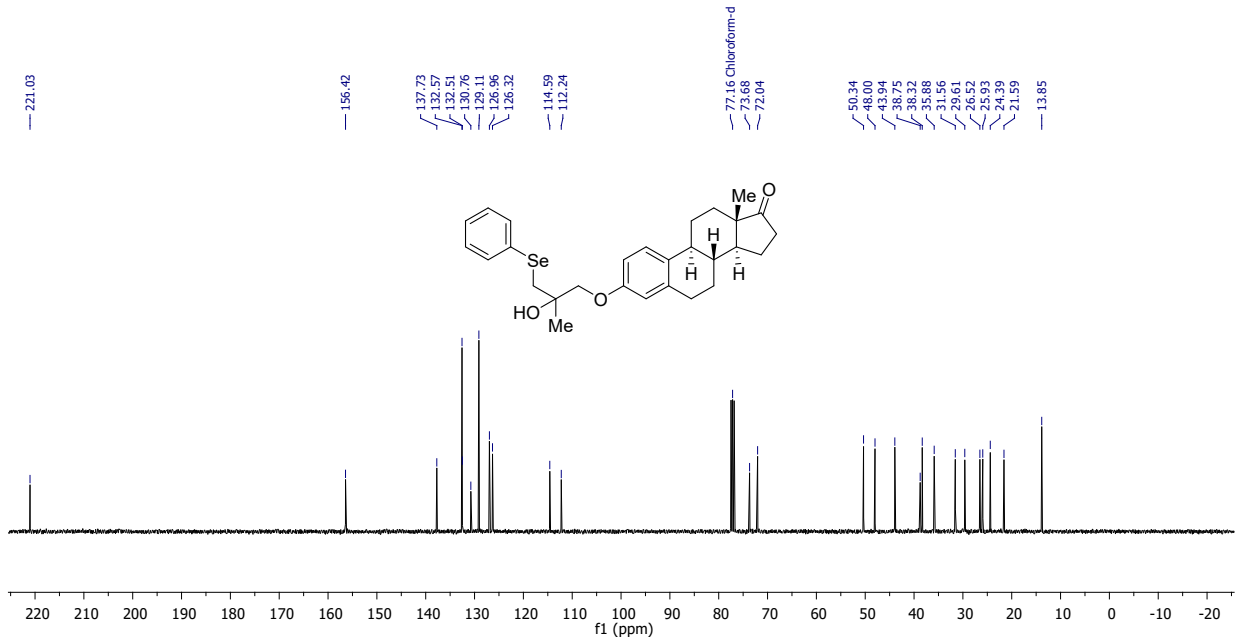
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **43** in $CDCl_3$



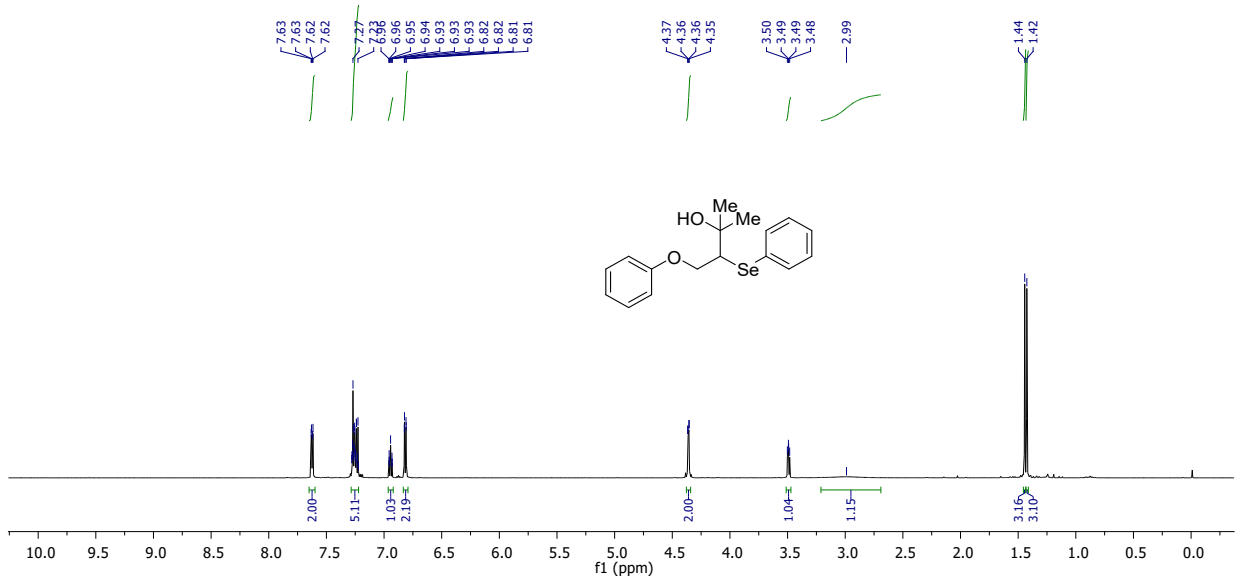
^{1}H-NMR (400 MHz) spectrum of **44** in $CDCl_3$



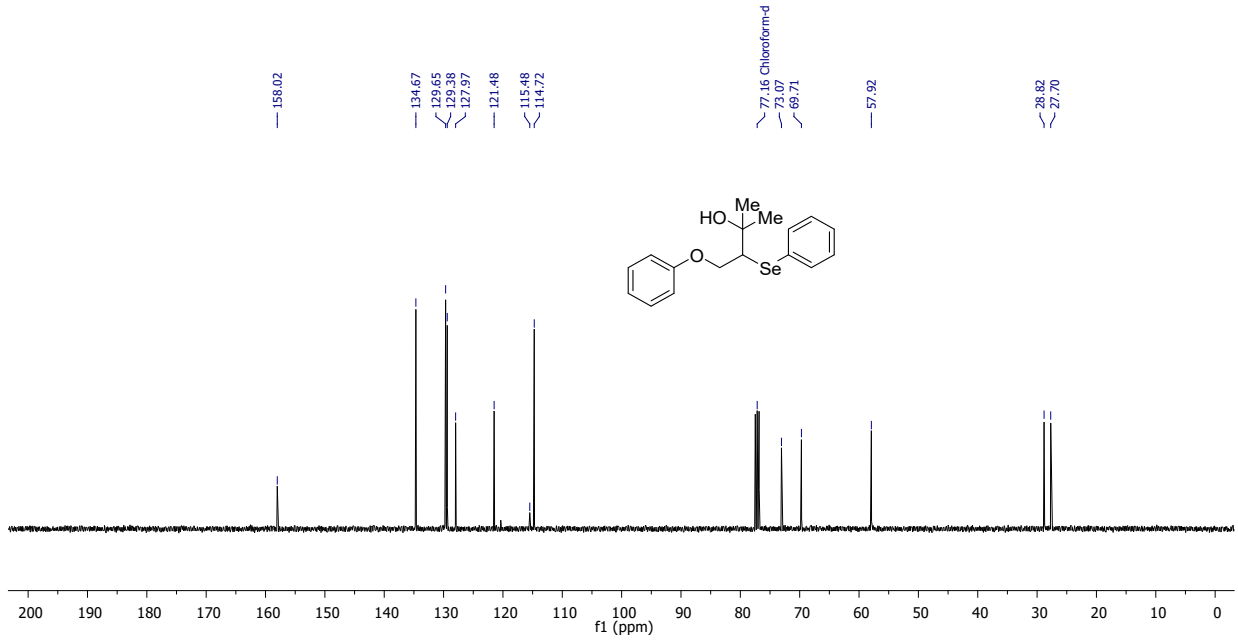
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **44** in $CDCl_3$



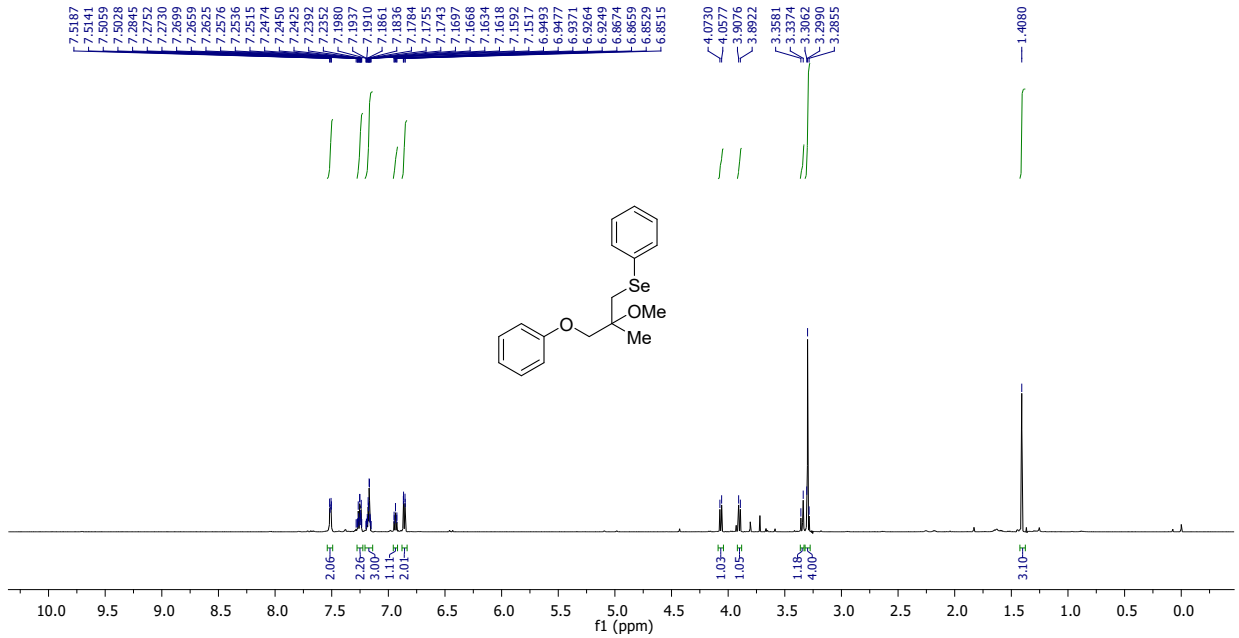
^{1}H-NMR (600 MHz) spectrum of **45** in $CDCl_3$



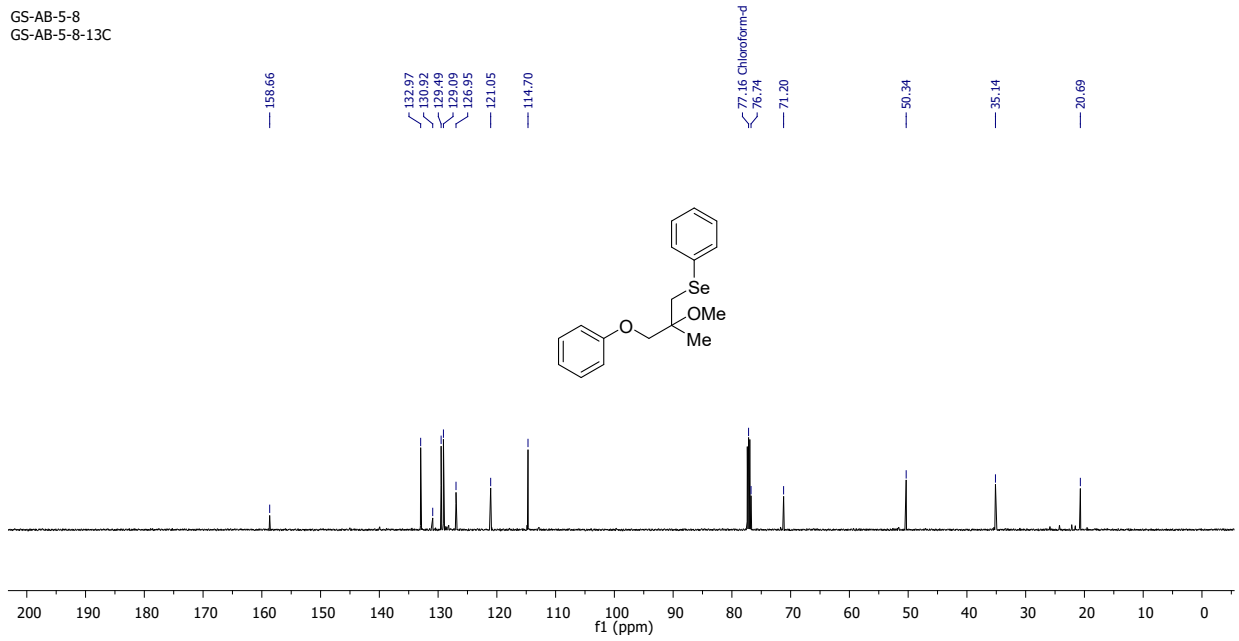
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **45** in $CDCl_3$



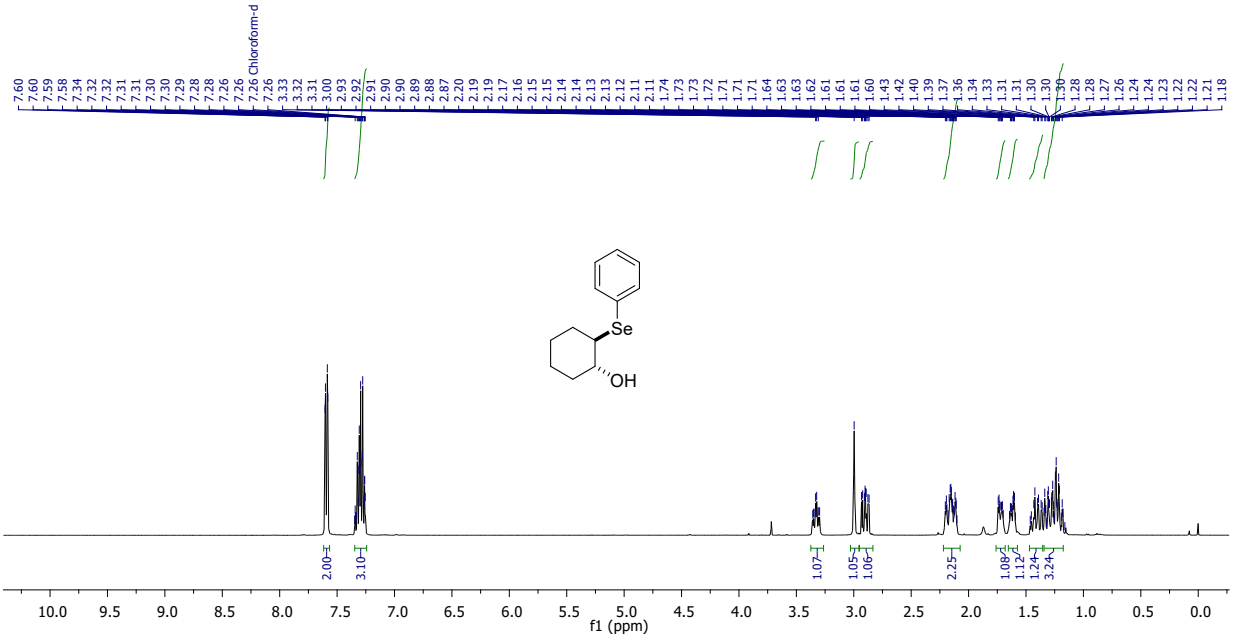
^{1}H-NMR (600 MHz) spectrum of **46** in $CDCl_3$



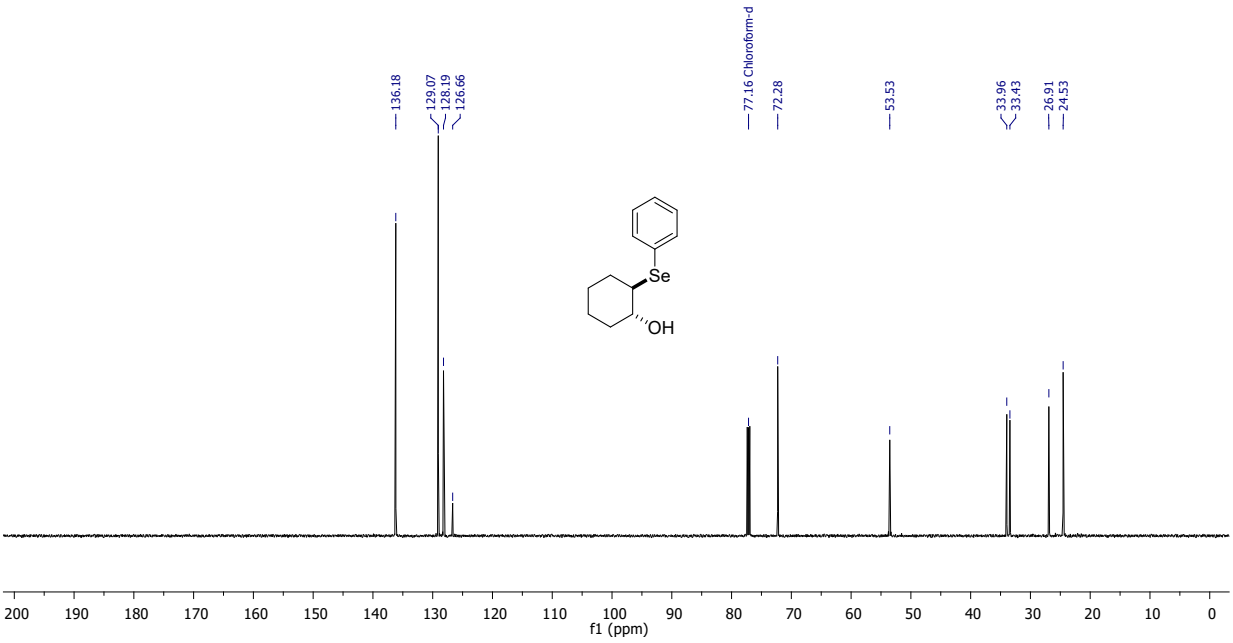
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **46** in $CDCl_3$



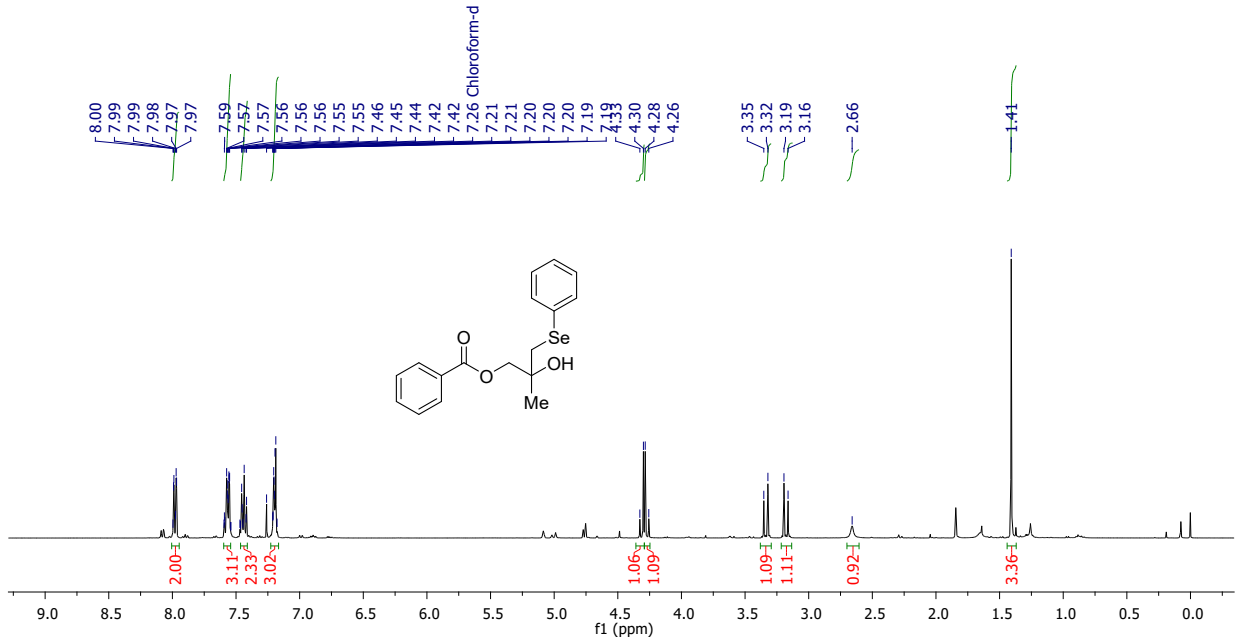
^{1}H-NMR (400 MHz) spectrum of **47** in $CDCl_3$



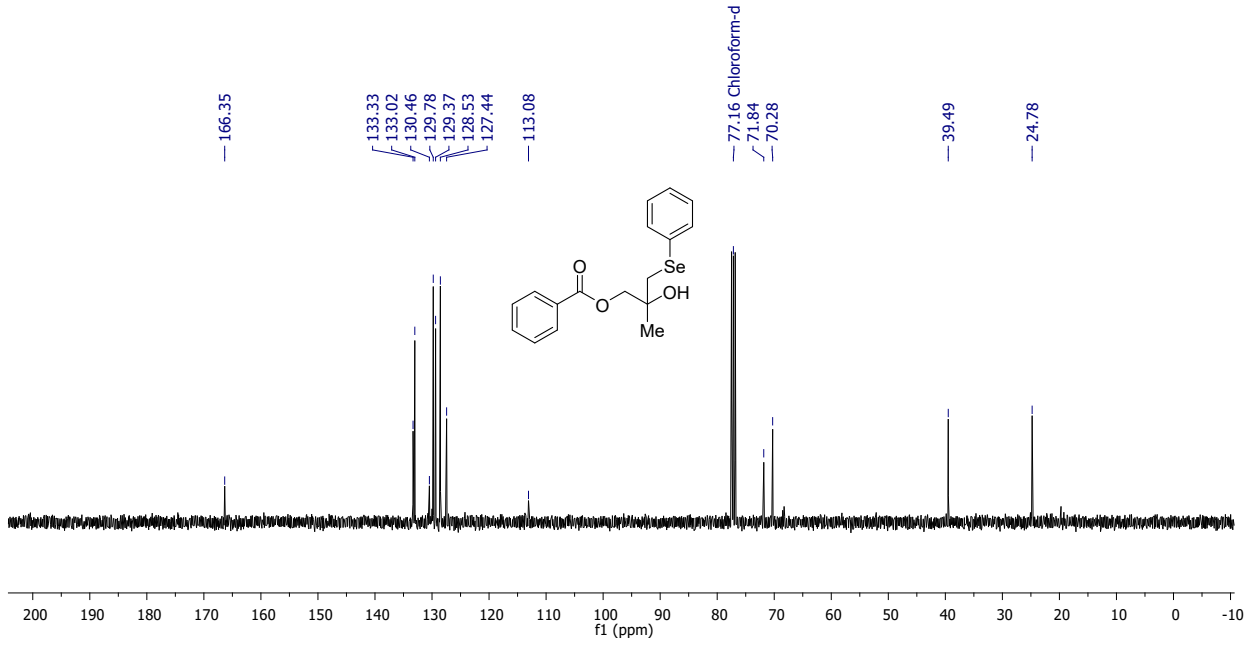
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **47** in $CDCl_3$



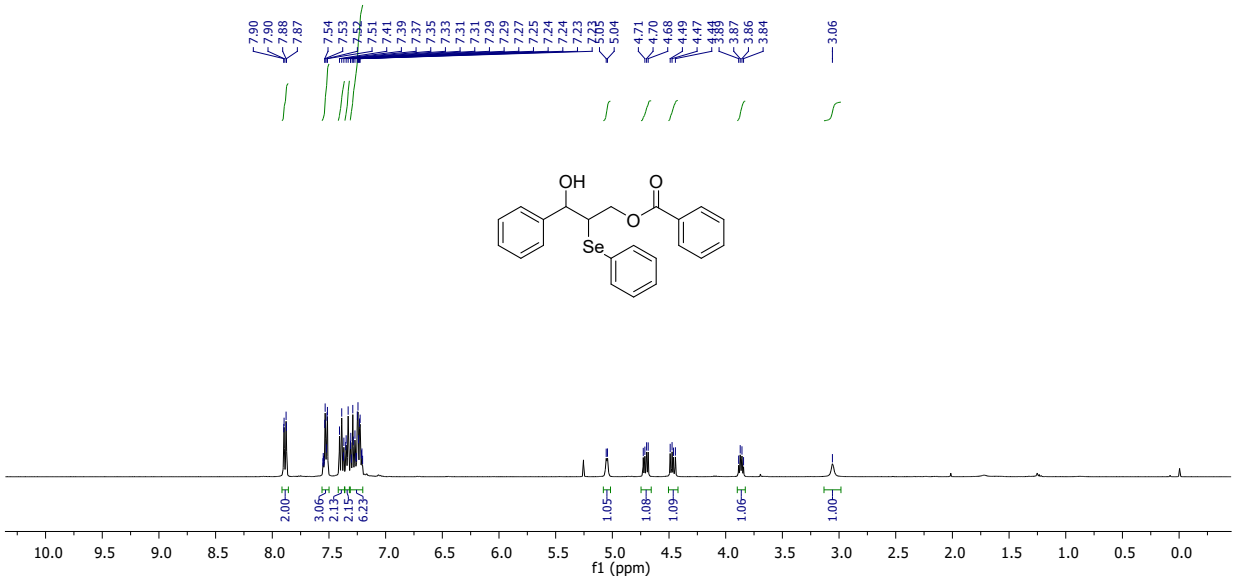
^{1}H-NMR (400 MHz) spectrum of **50** in $CDCl_3$



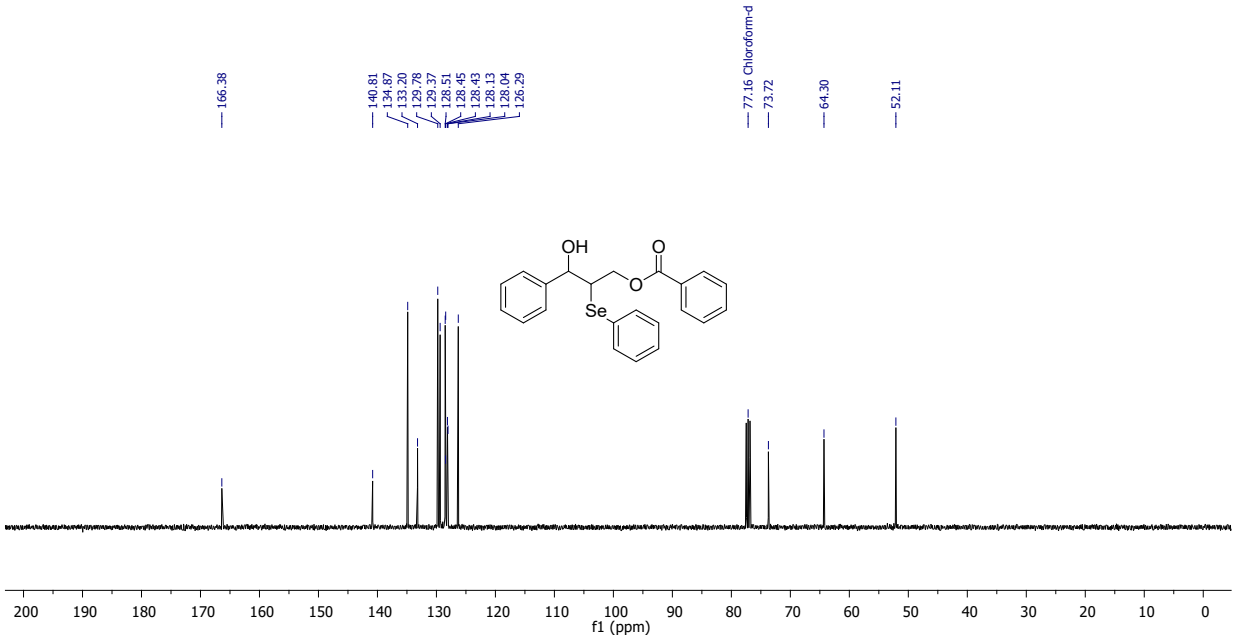
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **50** in $CDCl_3$



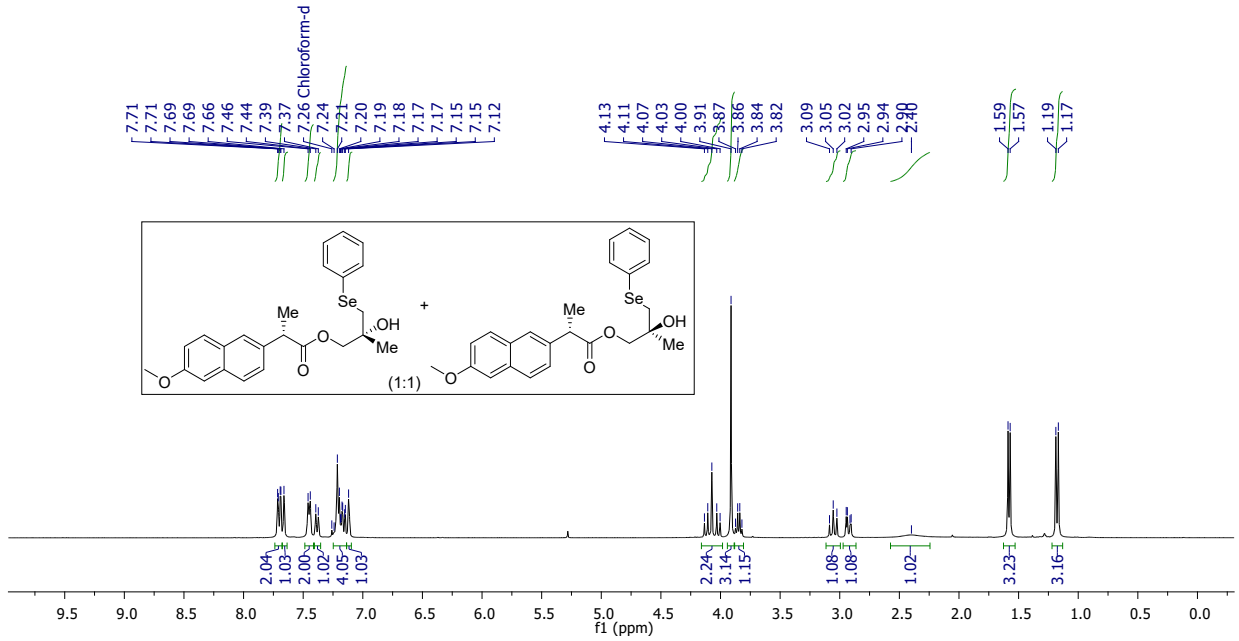
^{1}H-NMR (400 MHz) spectrum of **51** in $CDCl_3$



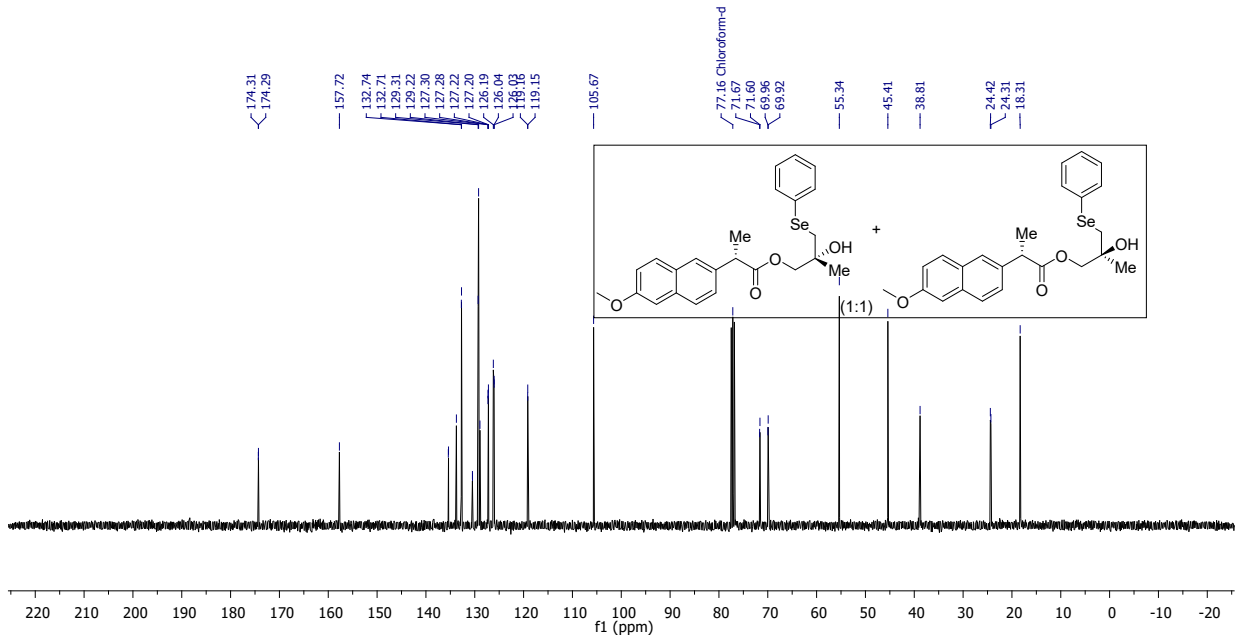
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **51** in $CDCl_3$



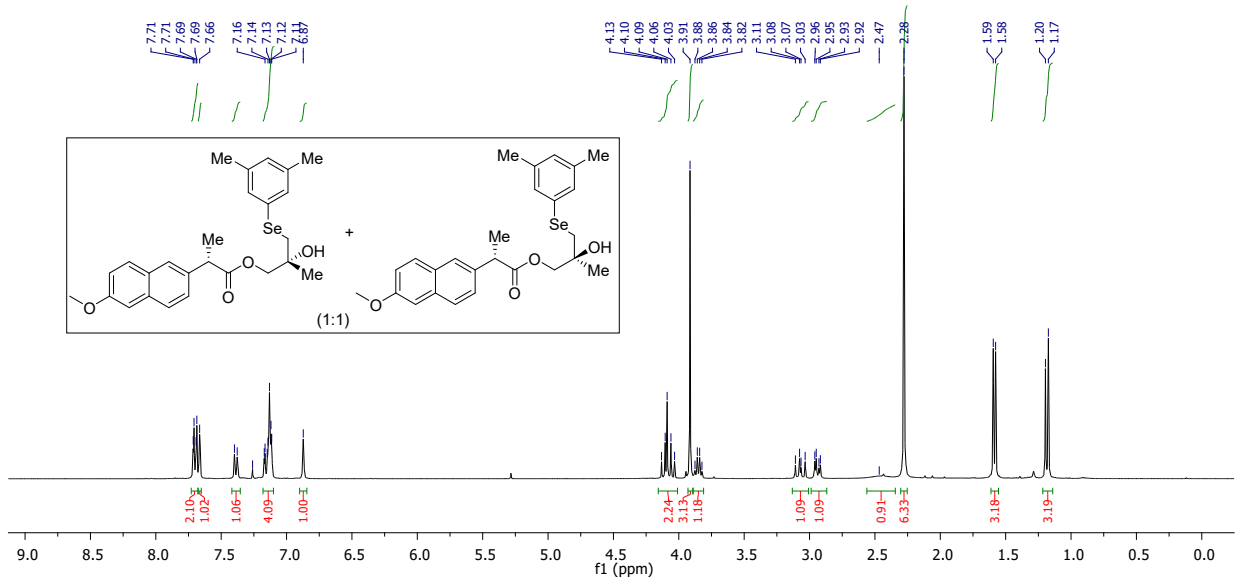
^{1}H-NMR (400 MHz) spectrum of (**52 + 52'**) in $CDCl_3$



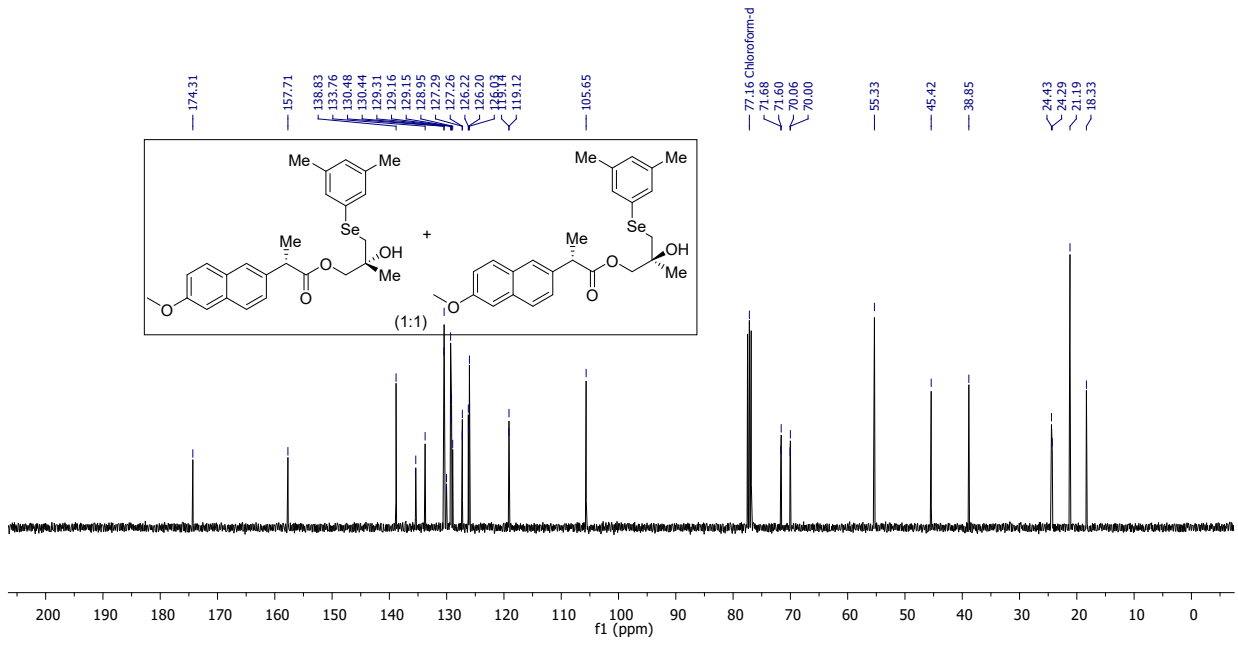
^{13}C{^{1}H}-NMR (101 MHz) spectrum of (**52 + 52'**) in $CDCl_3$



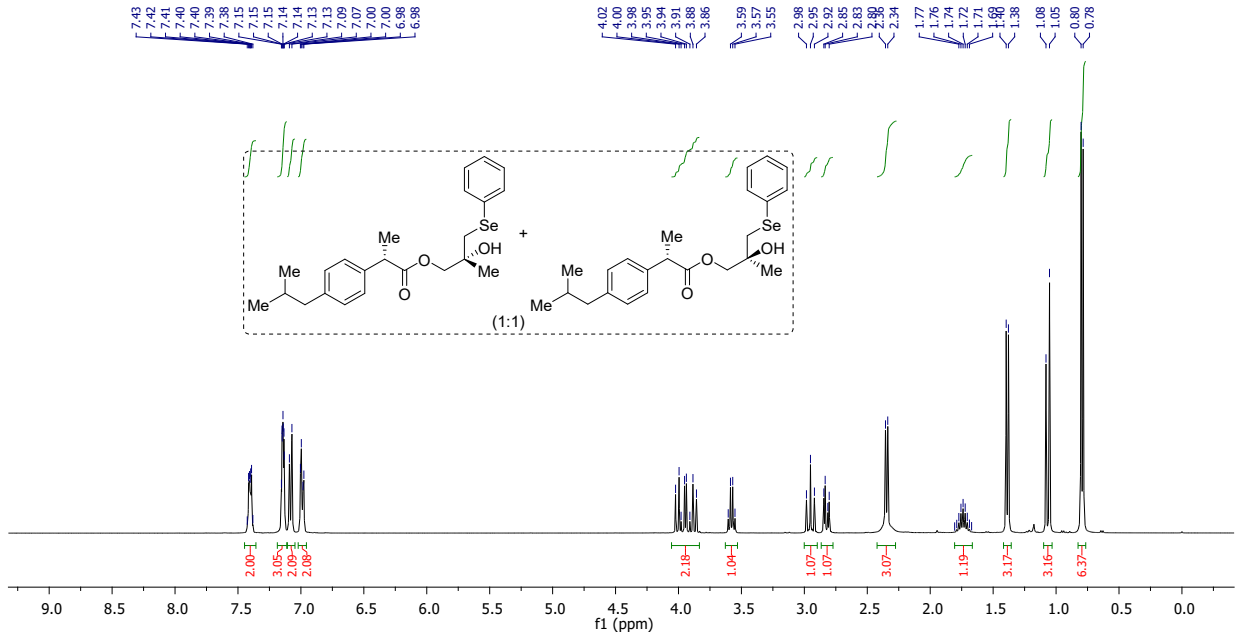
^{1}H-NMR (400 MHz) spectrum of (**53** + **53'**) in $CDCl_3$



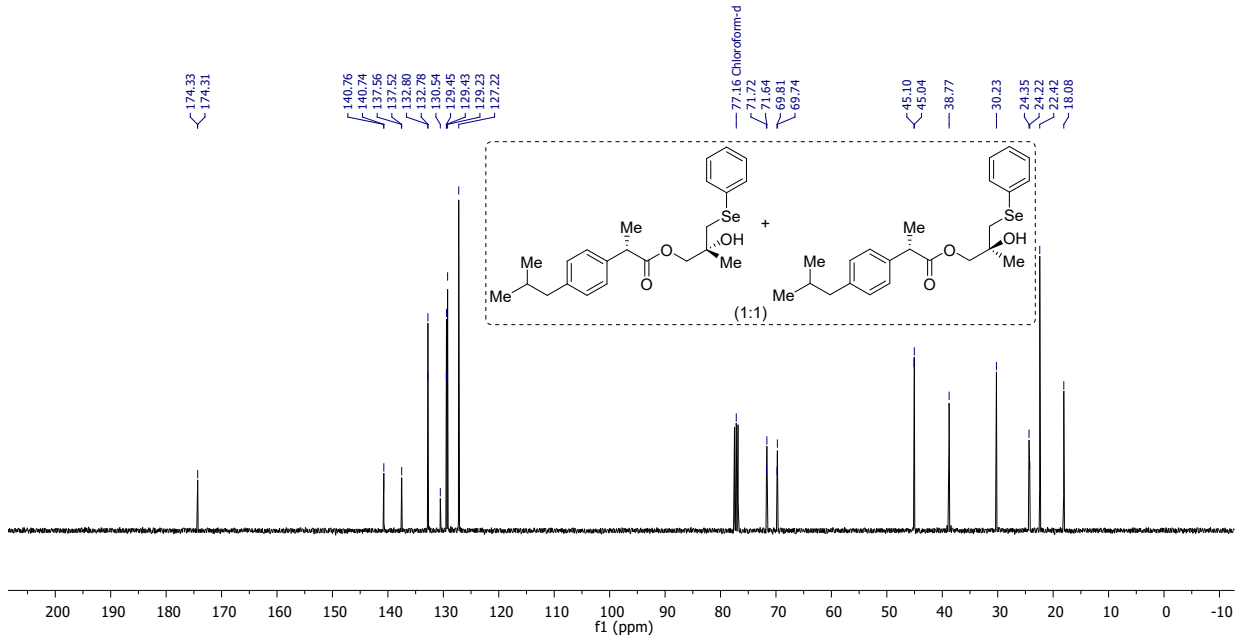
^{13}C{^{1}H}-NMR (101 MHz) spectrum of (**53** + **53'**) in $CDCl_3$



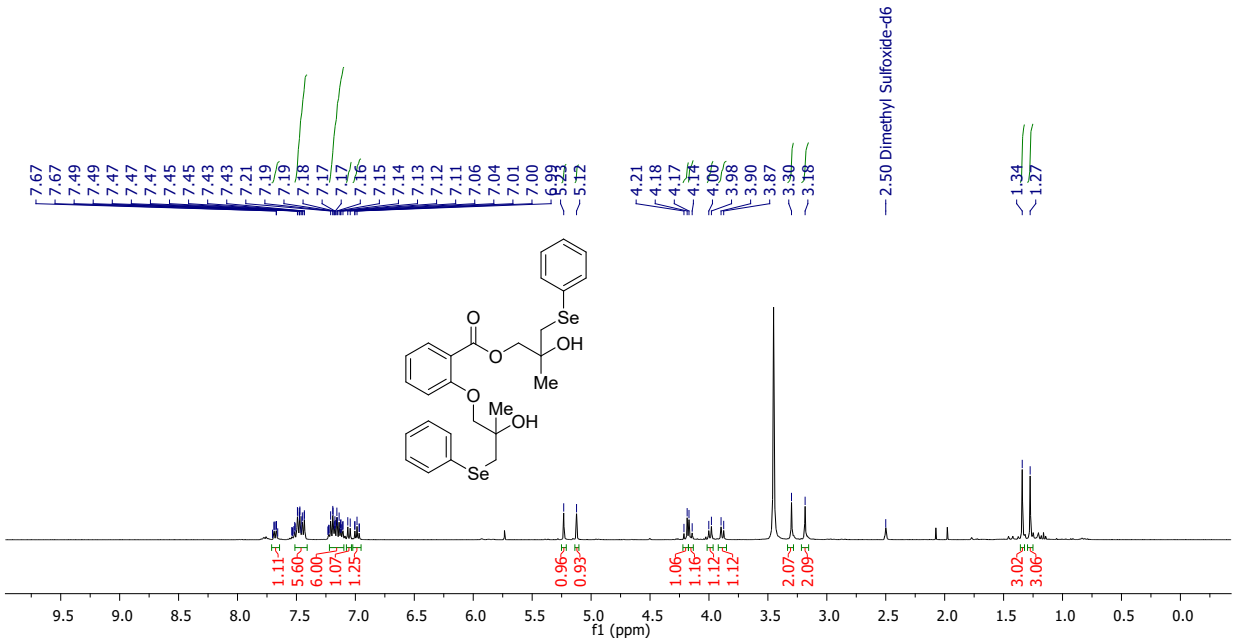
^{1}H-NMR (400 MHz) spectrum of (**54 + 54'**) in $CDCl_3$



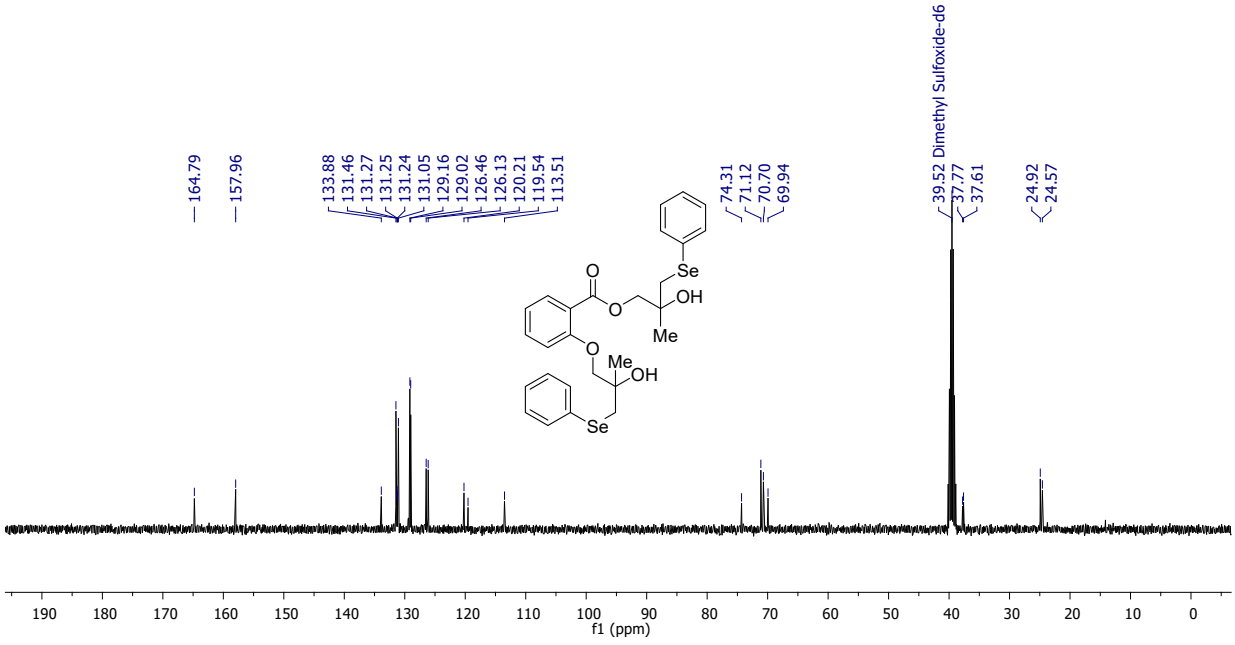
^{13}C{^{1}H}-NMR (101 MHz) spectrum of (**54 + 54'**) in $CDCl_3$



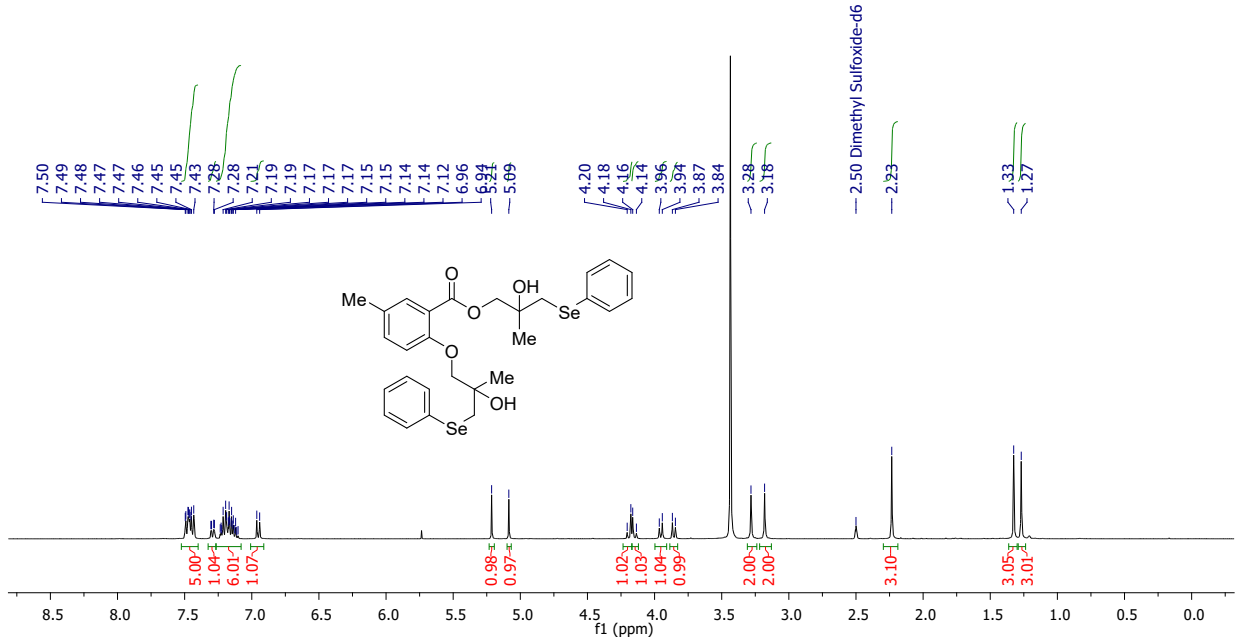
^{1}H-NMR (400 MHz) spectrum of **55** in DMSO-d_6



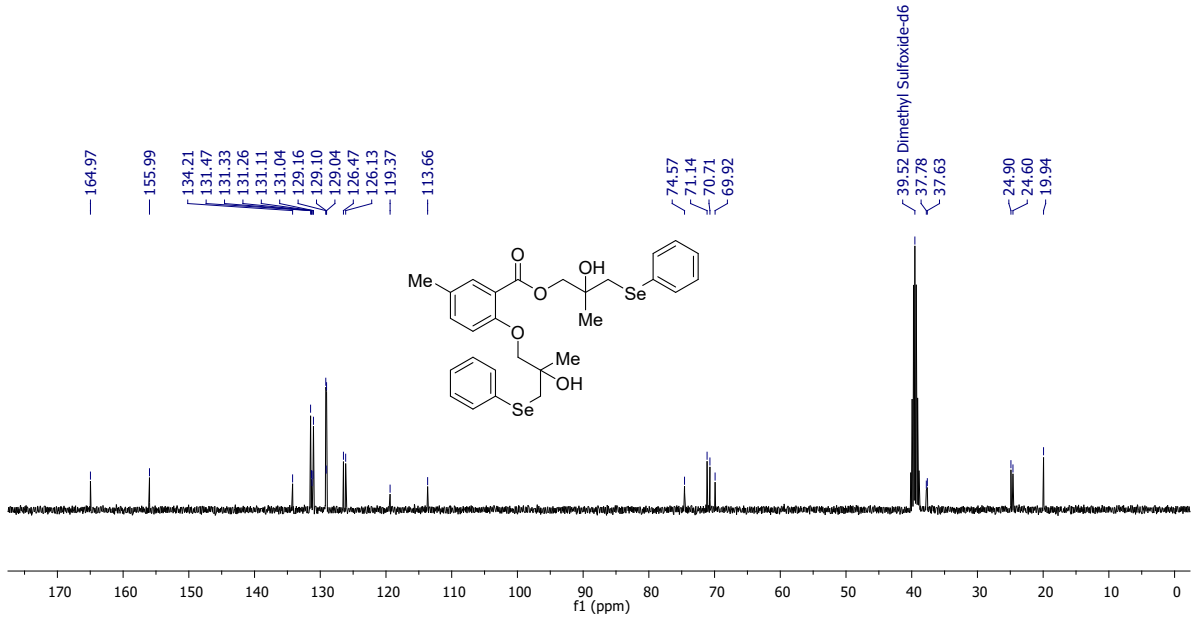
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **55** in DMSO-d_6



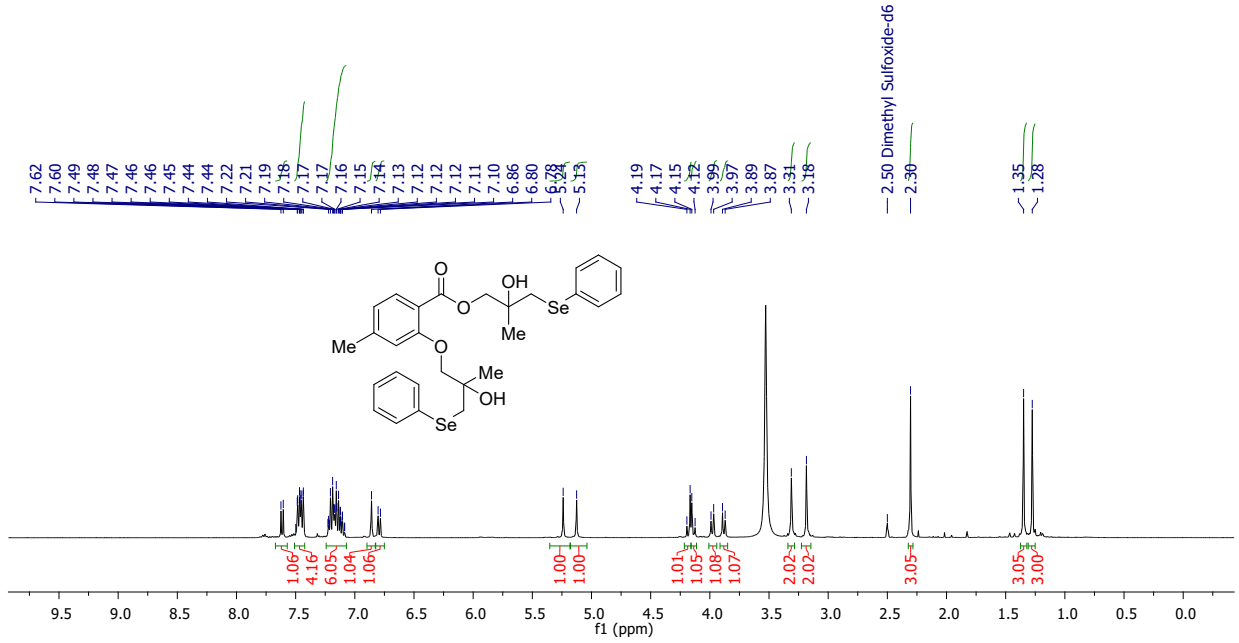
^{1}H-NMR (400 MHz) spectrum of **56** in DMSO-d_6



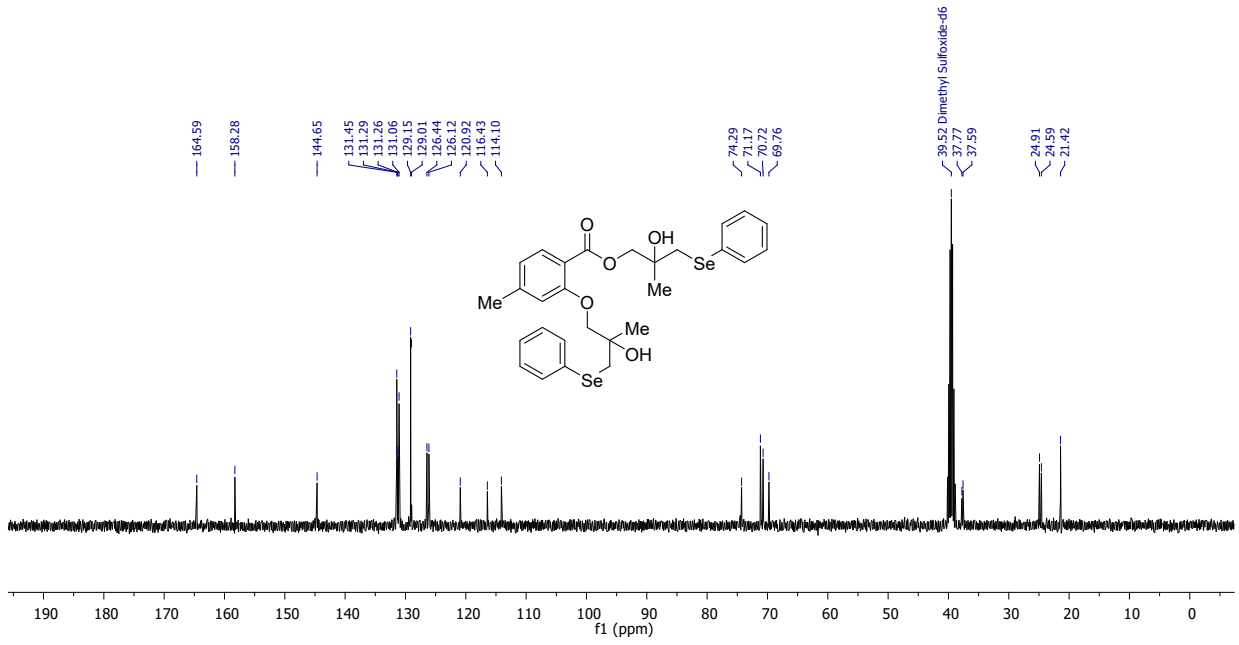
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **56** in DMSO-d_6



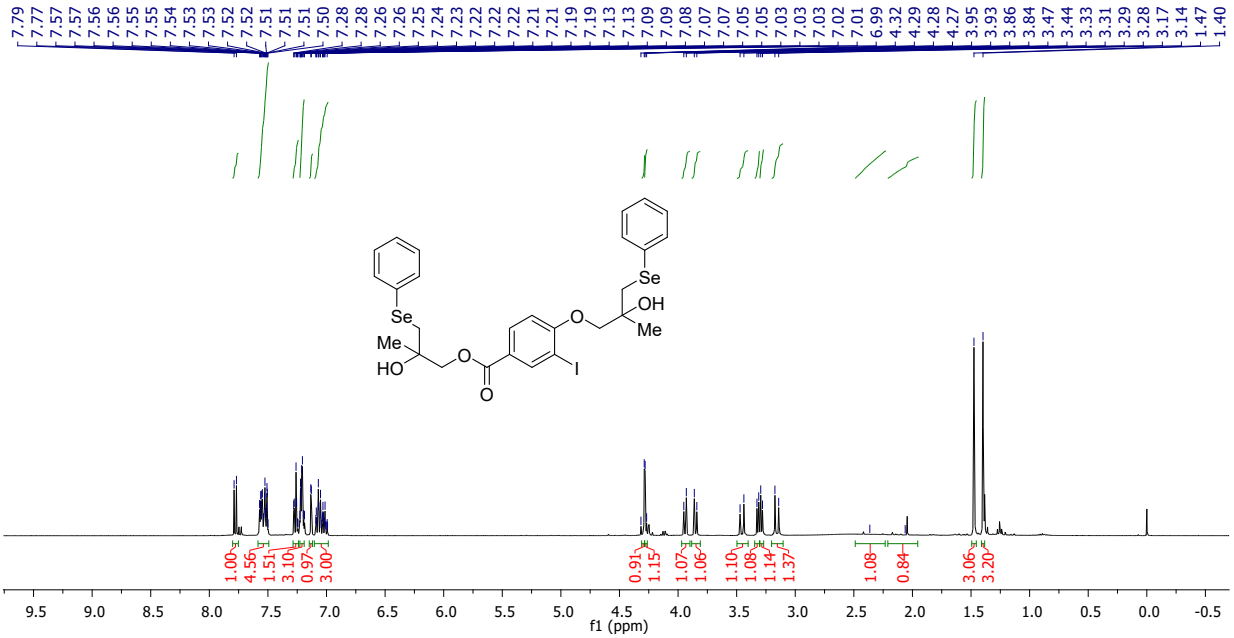
^{1}H-NMR (400 MHz) spectrum of **57** in DMSO-d_6



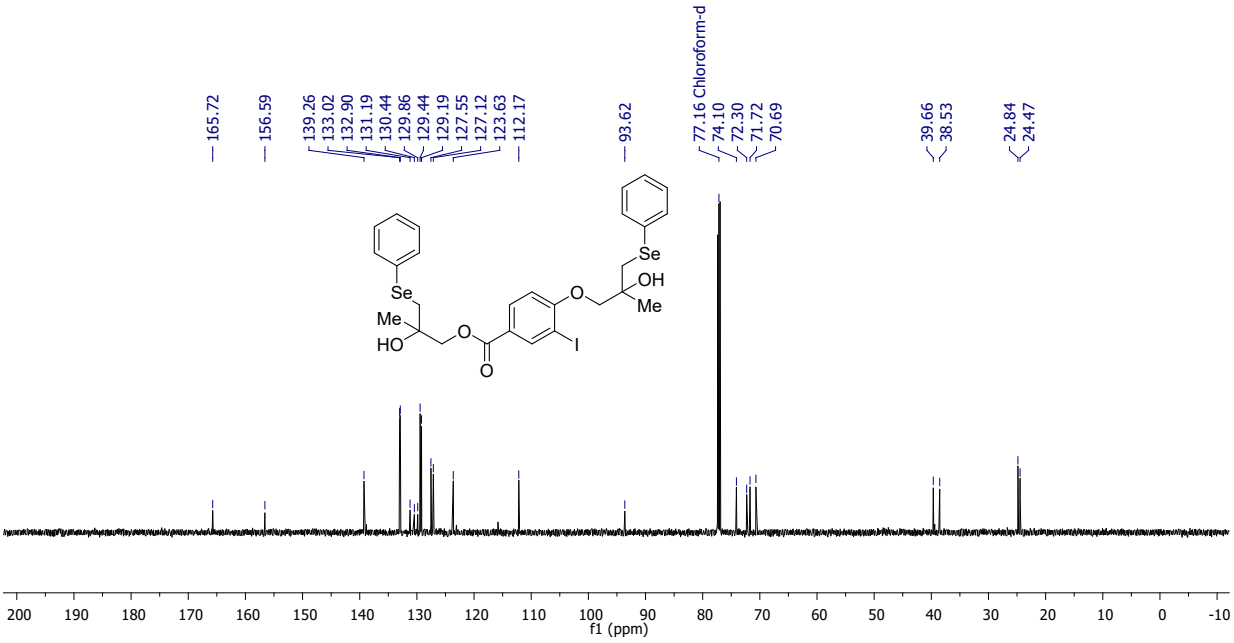
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **57** in DMSO-d_6



^{1}H-NMR (400 MHz) spectrum of **58** in $CDCl_3$



^{13}C{^{1}H}-NMR (151 MHz) spectrum of **58** in $CDCl_3$



Crystal structure data:

X-Ray crystal structure of compound **34**: Crystal of compound **34** were obtained by dissolving the product in CH_2Cl_2/CH_3CN/MeOH (1:1:1) and allowing the solvent to slowly evaporate at room temperature. The crystal structure information for this compound has been deposited at the Cambridge Crystallographic Data Centre. **CCDC No.** 2314641 contains the crystal structure information of this compound and can be obtained free of charge *via* http://www.ccdc.cam.ac.uk

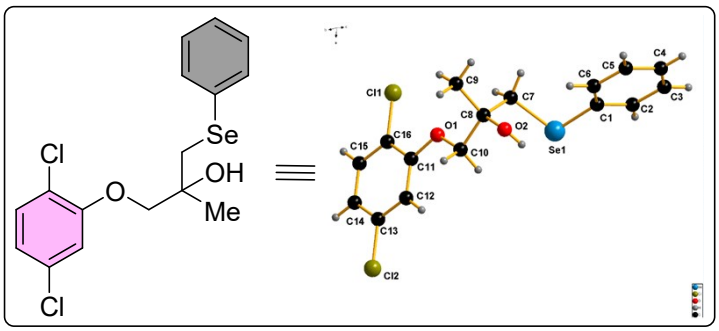


Figure S2: X-ray structure of the product **34** with the ellipsoids drawn at the 50% probability level (**CCDC**-2314641).

Table 4S: Crystal data and structure refinement for **34** (**CCDC**-2314641)

Table 1 Crystal data and structure refinement for mo_GS_AB_2_266_0ma.	
Identification code	**34**
Empirical formula	$C_{16}H_{16}Cl_2O_2Se$
Formula weight	390.15
Temperature/K	298
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	9.4200(18)
b/Å	25.033(5)
c/Å	7.5118(14)
α/°	90
β/°	111.342(6)
γ/°	90
Volume/Å^3	1649.9(6)
Z	4
ρ_{calc}g/cm^3	1.571
μ/mm^{-1}	2.600
F(000)	784.0
Crystal size/mm^3	0.22 × 0.137 × 0.02
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.642 to 54.328
Index ranges	$-12 \leq h \leq 12$, $-31 \leq k \leq 32$, $-9 \leq l \leq 9$

Reflections collected	25233
Independent reflections	3649 [R_{int} = 0.0759, R_{sigma} = 0.0489]
Data/restraints/parameters	3649/0/192
Goodness-of-fit on F^2	1.027
Final R indexes [I>=2σ (I)]	R_1 = 0.0396, wR_2 = 0.0681
Final R indexes [all data]	R_1 = 0.0775, wR_2 = 0.0794
Largest diff. peak/hole / e $Å^{-3}$	0.31/-0.45

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