Electronic Supplementary Material (ESI) for ChemComm.


# Supporting information

# Rhodium catalysed synthesis of seleno-ketals via carbene transfer reactions of diazoesters

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## General Information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Chemicals used in this manuscript were purchased from Sigma Aldrich, Alfa Aesar, Chempur, Fluorochem and Carl Roth. Solvents used in reactions were p.A. grade. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Macherey-Nagel silica gel aluminium plates with F-254 indicator, visualized by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.063 – 0.2 mm). Solvent mixtures are understood as volume/volume. 1 H-NMR, 19F-NMR and 13C-NMR were recorded on a Varian AV600/AV400 or an Agilent DD2 400 NMR spectrometer in $CDCl_3$. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated br (broadened singlet), s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet); coupling constants (J) are in Hertz (Hz). HRMS data were recorded on a ThermoFisher Scientific LTQ Orbitrap XL using ESI ionization or on a Finnigan MAT 95 using EI ionization at 70 eV. IR spectra were recorded on a Perkin Elmer-100 spectrometer and are reported in terms of frequency of absorption ($cm^{-1}$).

LEDs used in this manuscript were purchased from Conrad Electronics: High Power LED-Module, 3 W, 30 lm, 30 °, 470 nm, art.nr. 180745 – 62.

The following equipment was utilized: Syringe pump: Chemyx Inc. Model Fusion 710.

## Important Safety Note

Handling of diazo compounds should only be done in a well-ventilated fume cupboard using an additional blast shield. No incidents occurred handling of diazoalkanes during the preparation of this manuscript, yet the reader should be aware of carcinogenicity and explosiveness of the herein described diazo compounds. General safety precautions when working with diazomethane and its derivatives should be followed. Any reactions described in this manuscript should not be performed without strict risk assessment and proper safety precautions.

## Experimental Procedures

### General procedure for the synthesis of symmetrical diselenides (GP-I)[1]

To a stirred solution of selenium powder (2.0 Eq.) and aryl/aliphatic iodide (1.0 mmol, 1.0 Eq.) in dry DMSO (2.0 mL) was added CuO nanoparticles (0.1 Eq.) followed by KOH (2.0 Eq.) under argon atmosphere at 90 °C. The progress of the reaction was monitored by TLC. After the reaction was complete, the reaction mixture was allowed to cool to room temperature and it was then quenched with water and extracted with EtOAc. The combined organic layers were dried over anhydrous MgSO4. The solvent was removed under reduced pressure, and the residue was purified by flash chromatography on a silica gel column chromatography (n-hexane) to give the pure diselenides.

### General procedure for the synthesis of seleno-ketals (GP-II)

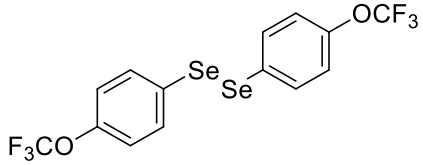
Diselenide (0.14 mmol, 0.7 Eq.) and $Rh_2(OAc)_4$ (0.5 mol-%) were suspended in 1.5 mL of water. Then the diazoalkane (0.2 mmol, 1 Eq.) and another 1 mL of water were added to the reaction mixture. The resultant reaction mixture was allowed to stir under open air at room temperature for 2 hours. The completion of the reaction was indicated by a color change of the reaction mixture to light green or yellowish green. The reaction mixture was extracted with diethyl ether and the crude product was purified by column chromatography on silica gel using n-pentane : diethyl ether (40 : 1 to 20 : 1) to afford the desired product.

| Diselenides | References |
|---|---|
| 1,2-di-p-tolyldiselane | *The spectral data is in accordance with the literature:* D. Singh, A. M. Deobald, L. R. S. Camargo, G. Tabarelli, O. E. D. Rodrigues, A. L. Braga, *Org. Lett.,* **2010**, *12*, 3288-3291. |
| 1,2-bis(4-fluorophenyl)diselane | *The spectral data is in accordance with the literature:* Z. Li, F. Ke, H. Deng, H. Xu, H. Xiang, X. Zhou*, Org. Biomol. Chem.,* **2013**, *11*, 2943–2946. |
| 1,2-bis(4-chlorophenyl)diselane | *The spectral data is in accordance with the literature:* M. S. Beigi, I. Yavarib, F. Sadeghizadeh, *RSC Adv.*, **2015**, *5*, 87564–87570 |
| 1,2-bis(4-methoxyphenyl)diselane | *The spectral data is in accordance with the literature:* D. Singh, A. M. Deobald, L. R. S. Camargo, G. Tabarelli, O. E. D. Rodrigues, A. L. Braga, *Org. Lett.,* **2010**, *12*, 3288-3291. |
| 1,2-bis(4-bromophenyl)diselane | *The spectral data is in accordance with the literature:* M. S. Beigi, I. Yavari, F, Sadeghizadeh, *RSC Adv*., **2015**, *5*, 87564–87570. |
| 1,2-di-m-tolyldiselane | *The spectral data is in accordance with the literature:* J. Li, C. Ma, D. Xing, W. Hu, *Org. Lett.*, **2019**, *21*, 2101-2105. |
| 1,2-bis(3-chlorophenyl)diselane | *The spectral data is in accordance with the literature:* F. H. Cui, J. Chen, S. X. Su, Y. l. Xu, H. S. Wang, Y. M Pan, *Adv.Synth.Catal.*, **2017**, *359*, 3950 –3961. |
| 1,2-bis(3-bromophenyl)diselane | *The spectral data is in accordance with the literature:* F. H. Cui, J. Chen, S. X. Su, Y. l. Xu, H. S. Wang, Y. M Pan, *Adv.Synth.Catal.*, **2017**, *359*, 3950 –3961. |
| 1,2-bis(3-(trifluoromethyl)phenyl)diselane | *The spectral data is in accordance with the literature:* M. Jiang, H. Yang, H. Fu, *Org. Lett.,* **2016**, *18*, 1968-1971. |
| 1,2-di-o-tolyldiselane | *The spectral data is in accordance with the literature:* D. Singh, A. M. Deobald, L. R. S. Camargo, G. Tabarelli, O. E. D. Rodrigues, A. L. Braga, *Org. Lett.,* **2010**, *12*, 3288-3291. |
| 1,2-bis(2-fluorophenyl)diselane | *The spectral data is in accordance with the literature:* L. Yu, J. Wang, T. Chen, Y. Wang, Q. Xu, *Appl. Organometal. Chem.*, **2014**, *28*, 652–656. |
| 1,2-bis(2-chlorophenyl)diselane | *The spectral data is in accordance with the literature:* D. Singh, A. M. Deobald, L. R. S. Camargo, G. Tabarelli, O. E. D. Rodrigues, A. L. Braga, *Org. Lett.,* **2010**, *12*, 3288-3291. |
| 1,2-di(thiophen-2-yl)diselane | *The spectral data is in accordance with the literature:* M. S. Beigi, I. Yavari, F, Sadeghizadeh, *RSC Adv*., **2015**, *5*, 87564–87570. |
| 1,2-di(pyridin-2-yl)diselane | *The spectral data is in accordance with the literature:* K.K. Bhasin, J. Singh, *J. Organomet. Chem.*, **2002**, *658*, 71-76 |
| 1,2-diheptyldiselane | *The spectral data is in accordance with the literature:* D. Singh, A. M. Deobald, L. R. S. Camargo, G. Tabarelli, O. E. D. Rodrigues, A. L. Braga, *Org. Lett.,* **2010**, *12*, 3288-3291. |

## Characterization data

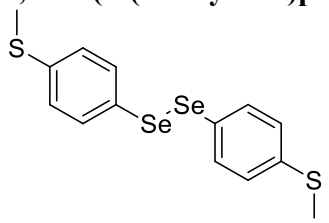
## diselenides

### 1,2-bis(4-(trifluoromethoxy)phenyl)diselane




The titled compound was synthesized according to the general procedure-I. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as yellow oil with 72% yield.
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.61 (d, *J* = 8.8 Hz, 1H), 7.13 (d, *J* = 8.7 Hz, 2H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 149.1, 134.4, 133.2, 128.9, 128.6, 121.9, 121.6, 120.3 (q, *J* = 261.2 Hz) ppm.
**$^{19}$F NMR** (376 MHz, Chloroform-*d*): δ = -57.9 ppm.
**IR (KBr):** 1892, 1761, 1582, 1485, 1397, 1252, 1205, 1159, 1064, 1013, 921, 836, 804, 719, 656 $cm^{-1}$.

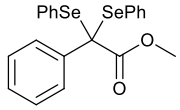
### 1,2-bis(4-(methylthio)phenyl)diselane




The titled compound was synthesized according to the general procedure-I. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as yellow foam with 81% yield.
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.49 (d, *J* = 8.3 Hz, 4H), 7.12 (d, *J* = 8.4 Hz, 4H), 2.48 (s, 6H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 139.2, 133.0, 127.1, 126.9, 15.6 ppm.
**HRMS (ESI):** Mass found: 428.87534, calculated mass for $C_{14}H_{14}NaS_2Se_2^+$: 428.87596.
**IR (KBr):** 3459, 3029, 2920, 2851, 2652, 2327, 2111, 2002, 1881, 1740, 1623, 1566, 1466, 1423, 1379, 1323, 1216, 1088, 1003, 959, 830, 795, 709 $cm^{-1}$.

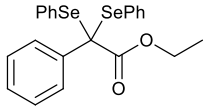
## Seleno-ketals

### Methyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5a)



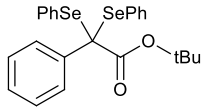

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as yellow gel (95%, 88 mg).
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.40 – 7.35 (m, 4H), 7.30 (m, 2H), 7.20 – 7.05 (m, 9H), 3.64 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.2, 137.9, 137.1, 129.3, 129.1, 128.6, 128.4, 127.4, 127.3, 59.8, 52.9 ppm.
**HRMS (ESI):** Mass found: 484.95297, calculated mass for $C_{21}H_{18}NaO_2Se_2^+$: 484.95294.
**IR (KBr):** 3416, 3056, 2946, 2842, 2643, 2324, 2190, 2162, 2097, 1957, 1881, 1808, 1713, 1576, 1473, 1436, 1291, 1232, 1067, 1012, 917, 858, 773 $cm^{-1}$.

## Ethyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5b)



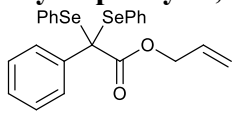

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (88%, 84 mg).
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.39 – 7.34 (m, 4H), 7.32 – 7.23 (m, 6H), 7.21 – 7.13 (m, 2H), 7.10 – 7.04 (m, 3H), 4.11 (q, *J* = 7.1 Hz, 2H), 1.10 (t, *J* = 7.1 Hz, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.8, 137.9, 136.9, 129.6, 129.0, 128.7, 128.4, 127.4, 127.2, 62.3, 59.7, 13.7 ppm.
**HRMS (ESI):** Mass found:498.96854, calculated mass for $C_{22}H_{20}NaO_2Se_2^+$: 498.96859.
**IR (KBr):** 3855, 3421, 3150, 3057, 2981, 2930, 2679, 2325, 2212, 1995, 1802, 1715, 1576, 1474, 1439, 1366, 1299, 1095, 1067, 968, 918, 851, 738 $cm^{-1}$.

## tert-butyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5c)



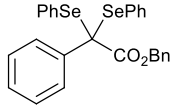

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (79%, 80 mg).
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.31 (dt, *J* = 8.3, 1.2 Hz, 4H), 7.27 – 7.21 (m, 2H), 7.19 – 7.09 (m,6H), 7.08 – 7.04 (m, 3H), 1.33 (s, 9H) ppm.
**$^{13}$C NMR** (101 MHz, Chloroform-*d*): δ = 169.9, 137.9, 136.3, 130.4, 129.0, 128.6, 128.3, 127.2, 127.1, 83.3, 27.6 ppm.
**HRMS (ESI):** Mass found: 526.99963, calculated mass for $C_{24}H_{24}NaO_2Se_2^+$: 526.99989.
**IR (KBr):** 3418, 2978, 2335, 2121, 1880, 1806, 1710, 1578, 1475, 1444, 1368, 1392, 1330, 1247, 1212, 1148, 981, 841, 737, 689 $cm^{-1}$.

## allyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5d)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (93%, 91 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.42 – 7.34 (m, 4H), 7.32 – 7.24 (m, 2H), 7.20 – 7.11 (m, 6H), 7.10 – 7.03 (m, 3H), 5.75 (ddt, *J* = 17.2, 10.4, 5.7 Hz, 1H), 5.34 – 5.12 (m, 2H), 4.55 (dt, *J* = 5.8, 1.4 Hz, 2H) ppm.
**$^{13}$C NMR** (101 MHz, Chloroform-*d*): δ = 170.5, 137.8, 137.0, 131.3, 129.5, 129.1, 128.8, 128.5, 127.4, 127.3, 118.9, 66.8, 59.6 ppm.
**HRMS (ESI):** Mass found:510.96857, calculated mass for $C_{23}H_{20}NaO_2Se_2^+$: 510.96859.
**IR (KBr):** 3548, 3058, 2940, 2726, 2332, 2097, 1954, 1992, 1880, 1730, 1688, 1575, 1474, 1439, 1366, 1297, 1269, 1193, 1065, 988, 935, 735, 687 $cm^{-1}$.

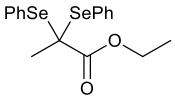
## Benzyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5e)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column
chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (82%, 88 mg).
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.33 – 7.20 (m, 11H), 7.16 – 6.99 (m, 9H), 5.11 (s, 2H) ppm.

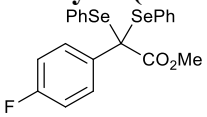
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.6, 137.7, 136.9, 135.0, 129.4, 129.0, 128.8, 128.4, 128.3, 128.2, 127.3, 127.3, 67.8, 59.4 ppm.
**HRMS (ESI):** Mass found: 560.98397, calculated mass for $C_{27}H_{22}NaO_2Se_2^+$: 560.98424.
**IR (KBr):** 3448, 3058, 2951, 2671, 2333, 2104, 1991, 1950, 1878, 1807, 1721, 1577, 1474, 1443, 1373, 1297, 1176, 1068, 996, 907, 838, 736, 689 $cm^{-1}$.

## Ethyl 2,2-bis(phenylselanyl)propanoate (5f)



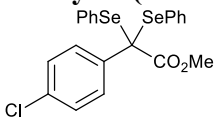

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 80:1) as colorless oil (51%, 42 mg).
**$^{1}$H NMR** (600 MHz, Chloroform-d): δ = 7.72 – 7.67 (m, 4H), 7.45 – 7.41 (m, 2H), 7.38 – 3.32 (m, 4H), 4.16 (q, J = 7.2 Hz, 2H), 1.66 (s, 3H), 1.24 (t, J = 7.1 Hz, 3H) ppm.
**$^{1}$3C NMR** (151 MHz, Chloroform-d): δ = 171.9, 137.5, 129.4, 128.8, 128.7, 62.0, 47.0, 26.3, 13.9 ppm.
**HRMS (ESI)**: Mass found: 436.95273, calculated mass for $C_{17}H_{18}NaO_2Se_2^+$: 436.95294.
**IR (KBr)**: 3840, 3488, 3057, 2982, 2664, 2324, 2092, 1993, 1936, 1804, 1721, 1575, 1473, 1438, 1371, 1323, 1298, 1239, 1140, 1099, 1019, 906, 859, 736, 699 $cm^{-1}$.

## Methyl 2-(4-fluorophenyl)-2,2-bis(phenylselanyl)acetate (5g)



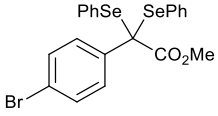

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (93%, 89 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.40 – 7.27 (m, 6H), 7.23 – 7.08 (m, 6H), 6.81 – 6.70 (m, 2H), 3.67 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.1, 161.6 (d, *J* = 248.3 Hz), 137.0, 133.7 (d, *J* = 3.0 Hz), 130.7 (d, *J* = 8.2 Hz), 129.3, 128.6, 114.2 (d, *J* = 21.7 Hz), 58.3, 53.06 ppm.
**$^{19}$F NMR** (564 MHz, Chloroform-*d*): δ = -114.4 (tt, *J* = 8.5, 5.2 Hz) ppm.
**HRMS (ESI):** Mass found: 502.94348, calculated mass for $C_{21}H_{17}FNaO_2Se_2^+$: 502.94352.
**IR (KBr):** 3863, 3421, 3057, 2993, 2943, 2697, 2099, 1885, 1805, 1716, 1595, 1501, 1472, 1437, 1332, 1297, 1229, 1160, 1104, 1066, 1016, 922, 841, 689 $cm^{-1}$.

## Methyl 2-(4-chlorophenyl)-2,2-bis(phenylselanyl)acetate (5h)



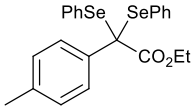

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (96%, 95 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.42 – 7.28 (m, 6H), 7.23 – 7.15 (m, 4H), 7.13 – 6.99 (m, 4H), 3.66 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.9, 137.0, 137.0, 136.4, 133.1, 130.2, 129.3, 129.3, 129.1, 128.6, 127.4, 58.3, 53.0 ppm.
**HRMS (ESI):** Mass found: 518.91339, calculated mass for $C_{21}H_{17}ClNaO_2Se_2^+$: 518.91397.
**IR (KBr):** 3883, 3444, 3148, 3057, 2949, 2842, 2670, 2329, 2242, 2175, 2108, 2024, 1993, 1882, 1804, 1721, 1580, 1483, 1435, 1399, 1306, 1273, 1230, 1091, 912, 832, 784, 739, 690 $cm^{-1}$.

## Methyl 2-(4-bromophenyl)-2,2-bis(phenylselanyl)acetate (5i)



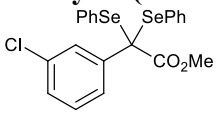
The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (88%, 95 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.40 – 7.27 (m, 6H), 7.24 – 7.15 (m, 6H), 7.06 – 6.98 (m, 2H), 3.66 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.8, 137.0, 132.3, 131.4, 130.5, 130.4, 129.3, 129.1, 128.6, 121.3, 58.3, 53.0 ppm.
**HRMS (ESI):** Mass found: 562.86267, calculated mass for $C_{21}H_{17}BrNaO_2Se_2^+$: 562.86346.
**IR (KBr):** 3430, 3057, 2949, 2842, 2650, 2324, 2098, 1984, 1885, 1803, 1720, 1579, 1480, 1435, 1394, 1303, 1273, 1229, 1176, 1071, 1005, 911, 828, 782, 737 cm$^{-1}$.

## Ethyl 2,2-bis(phenylselanyl)-2-(p-tolyl)acetate (5j)



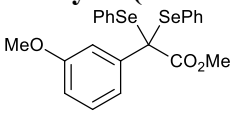

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (90%, 88 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.41 – 7.34 (m, 4H), 7.32 – 7.24 (m, 2H), 7.20 – 7.12 (m, 4H), 7.07 – 7.00 (m, 2H), 6.92 – 6.84 (m, 2H), 4.10 (q, *J* = 7.1 Hz, 2H), 2.26 (s, 3H), 1.09 (t, *J* = 7.1 Hz, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.9, 137.1, 136.8, 135.0, 129.7, 128.9, 128.6, 128.3, 128.0, 62.3, 59.7, 20.9, 13.7 ppm.
**HRMS (ESI):** Mass found: 512.98334, calculated mass for $C_{23}H_{22}NaO_2Se_2^+$: 512.98424.
**IR (KBr):** 3872, 3452, 3054, 2982, 2926, 2666, 2330, 2217, 2107, 1995, 1873, 1800, 1725, 1682, 1605, 1574, 1510, 1472, 1438, 1368, 1300, 1265, 1203, 1173, 1096, 1054, 824, 734 cm$^{-1}$.

## Methyl 2-(3-chlorophenyl)-2,2-bis(phenylselanyl)acetate (5k)



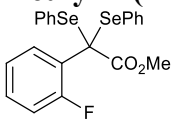

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (92%, 91 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.41 – 7.14 (m, 10H), 7.13 – 6.96 (m, 4H), 3.66 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.7, 139.7, 137.1, 133.3, 129.5, 129.2, 129.0, 128.6, 128.6, 127.3, 126.9, 58.2, 53.1 ppm.
**HRMS (ESI):** Mass found: 518.91241, calculated mass for $C_{21}H_{17}ClNaO_2Se_2^+$: 518.91397.
**IR (KBr):** 3879, 3665, 3446, 3150, 2998, 2853, 2670, 2494, 2326, 2232, 2179, 2112, 1997, 1931, 1881, 1802, 1721, 1694, 1569, 1472, 1432, 1307, 1274, 1235, 1183, 1067, 1018, 903, 827, 788 cm$^{-1}$.

## Methyl 2-(3-methoxyphenyl)-2,2-bis(phenylselanyl)acetate (5l)



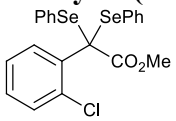

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (87%, 86 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.46 – 7.37 (m, 4H), 7.35 – 7.27 (m, 2H), 7.19 (m, 4H), 7.09 – 6.99 (m, 1H), 6.76 (m, 1H), 6.69 – 6.59 (m, 2H), 3.65 (s, 3H), 3.58 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.1, 158.6, 139.2, 137.1, 129.4, 129.2, 128.5, 128.4, 121.0, 114.3, 113.5, 59.7, 55.1, 52.9 ppm.
**HRMS (ESI):** Mass found: 514.96356, calculated mass for $C_{22}H_{20}NaO_3Se_2^+$: 514.96351.
**IR (KBr):** 3431, 3069, 2999, 2944, 2837, 2327, 2113, 1944, 1874, 1874, 1798, 1723, 1688, 1602, 1575, 1474, 1432, 1318, 1293, 1214, 1054, 1015, 919, 881, 761, 683 cm$^{-1}$.

**Methyl 2-(2-fluorophenyl)-2,2-bis(phenylselanyl)acetate (5m)**



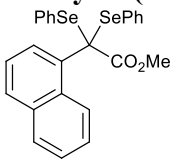

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (87%, 83 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.43 – 7.36 (m, 4H), 7.33 – 7.26 (m, 2H), 7.21 – 7.06 (m, 6H), 6.99 – 6.90 (m, 1H), 6.76 – 6.65 (m, 1H), 3.60 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.5, 159.2 (d, *J* = 249.7 Hz), 137.2, 131.1, 129.7 (d, *J* = 8.8 Hz), 129.3, 129.1, 128.5, 125.8 (d, *J* = 11.1 Hz), 122.7 (d, *J* = 3.6 Hz), 115.5 (d, *J* = 22.2 Hz), 54.7, 52.9 ppm.
**$^{19}$F NMR** (282 MHz, Chloroform-*d*): δ = -107.5 ppm.
**HRMS (ESI):** Mass found: 502.94333, calculated mass for $C_{21}H_{17}FNaO_2Se_2^+$: 502.94352.
**IR (KBr):** 3867, 3371, 3057, 2951, 2845, 2666, 2330, 2200, 2089, 1999, 1942, 1871, 1803, 1735, 1690, 1610, 1575, 1476, 1437, 1321, 1258, 1205, 1155, 1103, 1063, 1005, 908, 834, 737, 686 cm$^{-1}$.

**Methyl 2-(2-chlorophenyl)-2,2-bis(phenylselanyl)acetate (5n)**



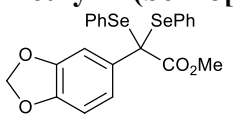

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1) as colorless oil (78%, 77 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.38 (ddd, *J* = 8.3, 4.1, 1.6 Hz, 5H), 7.33 – 7.23 (m, 3H), 7.20 – 7.11 (m, 4H), 7.09 – 7.02 (m, 1H), 6.81 (ddd, *J* = 8.1, 7.4, 1.3 Hz, 1H), 3.57 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.4, 137.0, 134.2, 133.7, 132.7, 130.7, 129.2, 129.1, 128.4, 125.5, 59.0, 52.8 ppm.
**HRMS (ESI):** Mass found: 518.91382, calculated mass for $C_{21}H_{17}ClNaO_2Se_2^+$: 518.91397.
**IR (KBr):** 3856, 3426, 3059, 2945, 2846, 2670, 2325, 2097, 2018, 1807, 1720, 1574, 1463, 1434, 1326, 1302, 1270, 1226, 1128, 1058, 1015, 920, 855, 771, 738, 657 cm$^{-1}$.

**Methyl 2-(naphthalen-1-yl)-2,2-bis(phenylselanyl)acetate (5o)**




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (83%, 85 mg).
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 8.02 (d, *J* = 8.6 Hz, 1H), 7.84 – 7.77 (m, 1H), 7.67 – 7.58 (m, 2H), 7.55 – 7.40 (m, 2H), 7.30 – 7.15 (m, 6H), 7.11 – 6.98 (m, 5H), 3.49 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 172.2, 136.8, 134.1, 131.4, 130.4, 129.7, 129.4, 129.0, 128.9, 128.3, 125.6, 125.4, 124.9, 123.9, 114.9, 52.8 ppm.
**HRMS (ESI):** Mass found: 534.96832, calculated mass for $C_{25}H_{20}NaO_2Se_2^+$: 534.96859.
**IR (KBr):** 3454, 3054, 2949, 2844, 2654, 2330, 2089, 1882, 1804, 1713, 1677, 1573, 1509, 1473, 1435, 1301, 1216, 1169, 1084, 1018, 1018, 974, 913, 872, 777, 689 cm$^{-1}$.

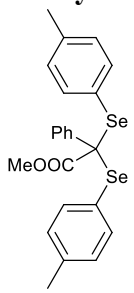
**Methyl 2-(benzo[d][1,3]dioxol-5-yl)-2,2-bis(phenylselanyl)acetate (5p)**




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (71%, 72 mg).

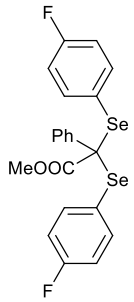
**$^1$H NMR** (400 MHz, Chloroform-*d*): δ = 7.40 (dt, *J* = 7.6, 1.1 Hz, 4H), 7.34 – 7.28 (m, 2H), 7.23 – 7.17 (m, 4H), 6.72 (d, *J* = 2.0 Hz, 1H), 6.64 (dd, *J* = 8.2, 2.0 Hz, 1H), 6.46 (d, *J* = 8.3 Hz, 1H), 5.88 (s, 2H), 3.64 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.2, 146.9, 146.7, 137.0, 131.6, 129.5, 129.2, 128.5, 123.0, 109.4, 106.8, 101.1, 53.0 ppm.
**HRMS (ESI):** Mass found: 528.94244, calculated mass for $C_{22}H_{18}NaO_4Se_2^+$: 528.94277.
**IR (KBr):** 3057, 2950, 2897, 2778, 2326, 2194, 2088, 1988, 1953, 1875, 1720, 1676, 1605, 1575, 1483, 1437, 1359, 1307, 1231, 1133, 1100, 1035, 890, 811, 777, 737, 689 cm$^{-1}$.

## Methyl 2-phenyl-2,2-bis(p-tolylselanyl)acetate (5q)



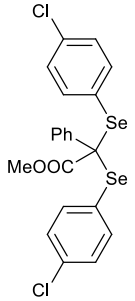

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (90%, 88 mg).
**$^1$H NMR** (400 MHz, Chloroform-*d*): δ = 7.28 – 7.23 (m, 4H), 7.17 – 7.05 (m, 5H), 6.98 (d, *J* = 7.8 Hz, 4H), 3.65 (s, 3H), 2.30 (s, 6H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.2, 139.4, 138.2, 137.2, 129.3, 128.7, 127.4, 127.2, 125.7, 59.7, 52.9, 21.3 ppm.
**HRMS (ESI):** Mass found: 512.98425, calculated mass for $C_{23}H_{22}NaO_2Se_2^+$: 512.98424.
**IR (KBr):** 3857, 3654, 3363, 3163, 3022, 2919, 2859, 2664, 2328, 2202, 1993, 1949, 1897, 1794, 1736, 1689, 1592, 1485, 1445, 1390, 1304, 1205, 1173, 1110, 799 cm$^{-1}$.

## Methyl 2,2-bis((4-fluorophenyl)selanyl)-2-phenylacetate (5r)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (86%, 86 mg).
**$^1$H NMR** (300 MHz, Chloroform-*d*): δ = 7.41 – 7.30 (m, 4H), 7.17 – 7.04 (m, 5H), 6.93 – 6.82 (m, 4H), 3.67 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.0, 163.6 (d, *J* = 250.2 Hz), 139.3 (d, *J* = 8.3 Hz), 137.6, 128.5, 127.6 (d, *J* = 5.2 Hz), 124.0, 115.8 (d, *J* = 21.6 Hz), 53.0 ppm.
**$^{19}$F NMR** (564 MHz, Chloroform-*d*): δ = -111.1 (tt, *J* = 8.6, 5.6 Hz) ppm.
**HRMS (ESI):** Mass found: 520.93459, calculated mass for $C_{21}H_{16}F_2NaO_2Se_2^+$: 520.93410.
**IR (KBr):** 3899, 3168, 3062, 2953, 2848, 2662, 2331, 2114, 1817, 1735, 1689, 1582, 1482, 1442, 1394, 1319, 1221, 1160, 1005, 822, 746 cm$^{-1}$.

**Methyl 2,2-bis((4-chlorophenyl)selanyl)-2-phenylacetate (5s)**




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (88%, 93 mg).

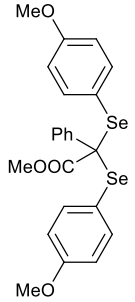
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.30 – 7.24 (m, 4H), 7.17 – 7.07 (m, 9H), 3.68 (s, 3H) ppm.

**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.0, 138.3, 137.4, 135.9, 128.7, 128.5, 127.7, 127.7, 127.3, 60.0, 53.1 PPM.

**HRMS (ESI):** Mass found: 552.87459, calculated mass for $C_{21}H_{16}Cl_2NaO_2Se_2^+$: 552.87500.

**IR (KBr):** 3445, 3059, 2949, 2848, 2654, 2327, 2179, 2085, 2025, 1994, 1898, 1721, 1692, 1565, 1470, 1440, 1384, 1320, 1292, 1231, 1175, 1087, 1007, 918, 863, 724, 692 ppm.

**Methyl 2,2-bis((4-methoxyphenyl)selanyl)-2-phenylacetate (5t)**




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (82%, 87 mg).

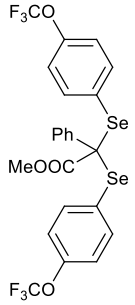
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.35 – 7.21 (m, 1H), 7.13 – 7.06 (m, 1H), 6.82 – 6.63 (m, 1H), 3.77 (s, 1H), 3.66 (s, 1H) ppm.

**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.2, 160.6, 139.0, 138.2, 128.6, 128.6, 128.5, 127.4, 127.2, 126.5, 119.9, 114.1, 60.1, 55.1, 52.9 ppm.

**HRMS (ESI):** Mass found: 544.97360, calculated mass for $C_{23}H_{22}NaO_4Se_2^+$: 544.97407.

**IR (KBr):** 3500, 3060, 3003, 2948, 2838, 2663, 2537, 2336, 1994, 1719, 1585, 1487, 1442, 1402, 1287, 1242, 1173, 1101, 1071, 1023, 921, 823, 695 cm$^{-1}$.

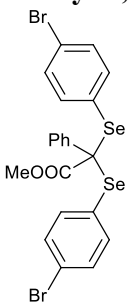
**Methyl 2-phenyl-2,2-bis((4-(trifluoromethoxy)phenyl)selanyl)acetate (5u)**




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (74%, 93 mg).

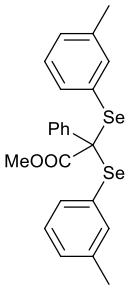
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.40 – 7.37 (m, 2H), 7.36 – 7.34(m, 2H), 7.13 – 7.06 (m, 5H), 7.05 – 6.97 (m, 4H), 3.66 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.9, 150.1, 138.5, 137.1, 128.5, 127.7 (d, *J* = 12.9 Hz), 127.4, 121.1, 120.8, 119.4, 60.1, 53.1 ppm.
**$^{19}$F NMR** (564 MHz, Chloroform-*d*): δ = -57.8 ppm.
**HRMS (ESI):** Mass found: 652.91772, calculated mass for $C_{23}H_{16}F_6NaO_4Se_2^+$: 652.91754.
**IR (KBr):** 3065, 2955, 2848, 2326, 2159, 1818, 1738, 1690, 1584, 1485, 1445, 1397, 1253, 1206, 1008, 839, 804, 691, 656 cm$^{-1}$.

## Methyl 2,2-bis((4-bromophenyl)selanyl)-2-phenylacetate (5v)



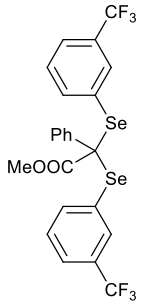

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (88%, 109 mg).
**$^{1}$H NMR** (600 MHz, Chloroform-*d*): δ = 7.32 – 7.28 (m, 4H), 7.21 – 7.18 (m, 4H), 7.15 – 7.09 (m, 5H), 3.68 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.0, 138.4, 137.3, 131.7, 128.5, 127.9, 127.8, 127.7, 124.3, 59.8, 53.1 ppm.
**HRMS (ESI):** Mass found: 640.77432, calculated mass for $C_{21}H_{16}Br_2NaO_2Se_2^+$: 640.77397.
**IR (KBr):** 3777, 3369, 3069, 2924, 2854, 2287, 2118, 1992, 1902, 1738, 1689, 1595, 1553, 1460, 1375, 1206, 1060, 1000, 809, 747, 701 cm$^{-1}$.

## Methyl 2-phenyl-2,2-bis(m-tolylselanyl)acetate (5w)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (92%, 90 mg).
**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.25 – 7.19 (m, 2H), 7.17 – 7.03 (m, 11H), 3.65 (s, 3H), 2.22 (s, 6H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.3, 138.1, 138.0, 137.7, 134.1, 129.9, 129.0, 128.8, 128.2, 127.3, 127.2, 52.8, 21.1 ppm.
**HRMS (ESI):** Mass found: 512.98421, calculated mass for $C_{23}H_{22}NaO_2Se_2^+$: 512.98424.
**IR (KBr):** 3855, 3648, 3358, 3052, 2920, 2671, 2325, 2110, 1991, 1922, 1737, 1689, 1591, 1446, 1319, 1273, 1205, 1171, 1001, 903, 873, 808, 772, 685 cm$^{-1}$.

## Methyl 2-phenyl-2,2-bis((3-(trifluoromethyl)phenyl)selanyl)acetate (5x)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (77%, 92 mg).

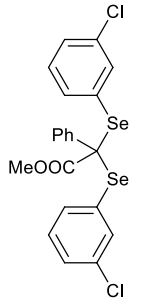
**$^1$H NMR** (400 MHz, Chloroform-*d*): δ = 7.61 (d, *J* = 7.7 Hz, 2H), 7.56 (d, *J* = 7.9 Hz, 2H), 7.51 (s, 2H), 7.33 (t, *J* = 7.8 Hz, 2H), 7.16 – 7.00 (m, 5H), 3.69 (s, 3H) ppm.

**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.9, 140.14 , 136.82 , 134.95 , 133.70 (d, *J* = 4.1 Hz), 130.83 (q, *J* = 32.5 Hz), 130.02 (d, *J* = 13.0 Hz), 128.87 , 128.35 , 128.11 , 127.84 , 126.00 (d, *J* = 3.9 Hz), 53.24 .

**HRMS (ESI):** Mass found: 620.92776, calculated mass for $C_{23}H_{16}F_6NaO_2Se_2^+$: 620.92771.

**IR (KBr):** 3064, 2953, 2849, 2647, 2330, 2199, 1903, 1726, 1693, 1580, 1418, 1317, 1235, 1167, 1125, 1066, 1004, 897, 794, 691 cm$^{-1}$.

## Methyl 2,2-bis((3-chlorophenyl)selanyl)-2-phenylacetate (5y)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane: diethyl ether – 40:1 → 20:1) as colourless oil (84%, 89 mg).

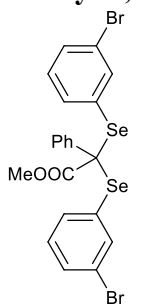
**$^1$H NMR** (400 MHz, Chloroform-*d*): δ = 7.30 – 7.23 (m, 6H), 7.17 – 7.07 (m, 7H), 3.70 (s, 3H) ppm.

**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.0, 137.1, 136.5, 134.9, 133.9, 130.5, 129.5, 129.3, 128.5, 127.9, 127.7, 60.3, 53.2 ppm.

**HRMS (ESI):** Mass found: 552.87396, calculated mass for $C_{21}H_{16}Cl_2NaO_2Se_2^+$: 552.87500.

**IR (KBr):** 3134, 3059, 2950, 2850, 2661, 2327, 2112, 1981, 1871, 1730, 1690, 1595, 1565, 1454, 1397, 1318, 1290, 1205, 1173, 1100, 1068, 876, 823, 776, 678 cm$^{-1}$.

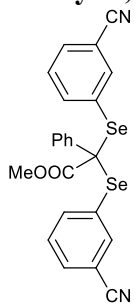
## Methyl 2,2-bis((3-bromophenyl)selanyl)-2-phenylacetate (5z)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (72%, 89 mg).

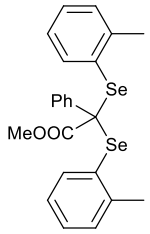
**$^1$H NMR** (600 MHz, Chloroform-*d*): δ = 7.46 – 7.40 (m, 3H), 7.35 – 7.30 (m, 2H), 7.18 – 7.03 (m, 8H), 3.71 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.0, 139.3, 137.1, 135.4, 132.2, 130.8, 129.8, 129.3, 128.5, 128.0, 127.7, 121.9, 60.5, 53.2 ppm.
**HRMS (ESI):** Mass found: 640.77391, calculated mass for C21H16Br2NaO2Se2+: 640.77397.
**IR (KBr):** 3854, 3464, 3358, 3051, 2950, 2848, 2665, 2331, 2114, 1990, 1922, 1864, 1808, 1737, 1688, 1594, 1558, 1452, 1393, 1204, 1056, 1001, 871, 812, 772, 674 cm$^{-1}$.

## Methyl 2,2-bis((3-cyanophenyl)selanyl)-2-phenylacetate (5aa)



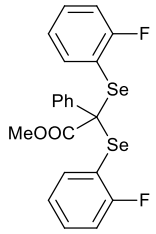

The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (46%, 47 mg).
**$^1$H NMR** (300 MHz, Chloroform-*d*): δ = 7.63 – 7.57 (m, 4H), 7.56 – 7.52 (m, 2H), 7.35 – 7.27 (m, 2H), 7.21 – 7.10 (m, 3H), 7.08 – 7.02 (m, 2H), 3.74 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.7, 140.9, 139.8, 136.4, 132.6, 130.5, 129.1, 128.4, 128.4, 128.0, 117.8, 112.8, 60.5, 53.5 ppm.
**HRMS (ESI):** Mass found: 534.94305, calculated mass for $C_{23}H_{16}N_2NaO_2Se_2^+$: 534.94344.
**IR (KBr):** 3857, 3418, 3062, 2950, 2924, 2854, 2685, 2480, 2327, 2181, 2229, 2092, 1992, 1955, 1917, 1806, 1720, 1560, 1463, 1440, 1401, 1235, 1095, 1006, 903, 794, 724, 683 cm$^{-1}$.

## Methyl 2-phenyl-2,2-bis(o-tolylselanyl)acetate (5ab)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (94%, 92 mg).
**$^1$H NMR** (300 MHz, Chloroform-*d*): δ = 7.48 – 7.42 (m, 2H), 7.22 – 6.91 (m, 11H), 3.55 (s, 3H), 2.23 (s, 6H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.8, 142.9, 137.9, 137.8, 130.8, 129.7, 129.3, 128.7, 127.3, 127.2, 125.8, 114.9, 59.2, 52.8, 22.8 ppm.
**HRMS (ESI):** Mass found: 512.98412, calculated mass for C23H22NaO2Se2+: 512.98424.
**IR (KBr):** 3856, 3128, 3057, 3004, 2949, 2847, 2662, 2327, 2179, 2113, 1990, 1802, 1730, 1691, 1589, 1454, 1377, 1322, 1270, 1204, 1172, 1120, 1051, 1027, 1004, 914, 800, 745, 698 cm$^{-1}$.

## Methyl 2,2-bis((2-fluorophenyl)selanyl)-2-phenylacetate (5ac)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (71%, 71 mg).

**$^{1}$H NMR** (300 MHz, Chloroform-*d*): δ = 7.41 – 7.34 (m, 2H), 7.33 – 7.16 (m, 4H), 7.10 – 7.01 (m, 3H), 7.01 – 6.88 (m, 4H), 3.76 (s, 3H) ppm.

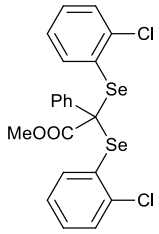
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.1, 163.1 (d, *J* = 246.1 Hz), 139.2, 136.7, 131.8 (d, *J* = 8.1 Hz), 128.7, 127.6, 127.4, 124.1 (d, *J* = 3.7 Hz), 116.4 (d, *J* = 22.1 Hz), 115.2 (d, *J* = 24.6 Hz), 53.3 ppm.

**$^{19}$F NMR** (282 MHz, Chloroform-*d*): δ = -99.77 ppm.

**HRMS (ESI):** Mass found: 520.93399, calculated mass for $C_{21}H_{16}F_2NaO_2Se_2^+$: 520.93410.

**IR (KBr):** 3885, 3661, 3456, 3066, 2951, 2850, 2665, 2329, 2098, 2025, 1989, 1951, 1911, 1734, 1689, 1579, 1494, 1464, 1257, 1210, 1172, 1113, 1049, 1003, 936, 911, 854 cm$^{-1}$.

## Methyl 2,2-bis((2-chlorophenyl)selanyl)-2-phenylacetate (5ad)




The titled compound was synthesized according to the general procedure-II. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (73%, 77 mg).

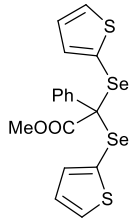
**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.33 – 7.23 (m, 6H), 7.17 – 7.01 (m, 5H), 6.99 – 6.92 (m, 2H), 3.79 (s, 3H) ppm.

**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 170.9, 138.1, 137.2, 135.3, 130.9, 129.7, 129.4, 129.2, 127.8, 127.5, 126.6, 53.6 ppm.

**HRMS (ESI):** Mass found:552.87500, calculated mass for $C_{21}H_{16}Cl_2NaO_2Se_2^+$: 552.87500.

**IR (KBr):** 3447, 3058, 2949, 2924, 2852, 2666, 2329, 2182, 2112, 1990, 1954, 1801, 1723, 1691, 1569, 1493, 1321, 1234, 1202, 1175, 1101, 1015, 918, 857, 813 cm$^{-1}$.

## Methyl 2-phenyl-2,2-bis(thiophen-2-ylselanyl)acetate (5ae)



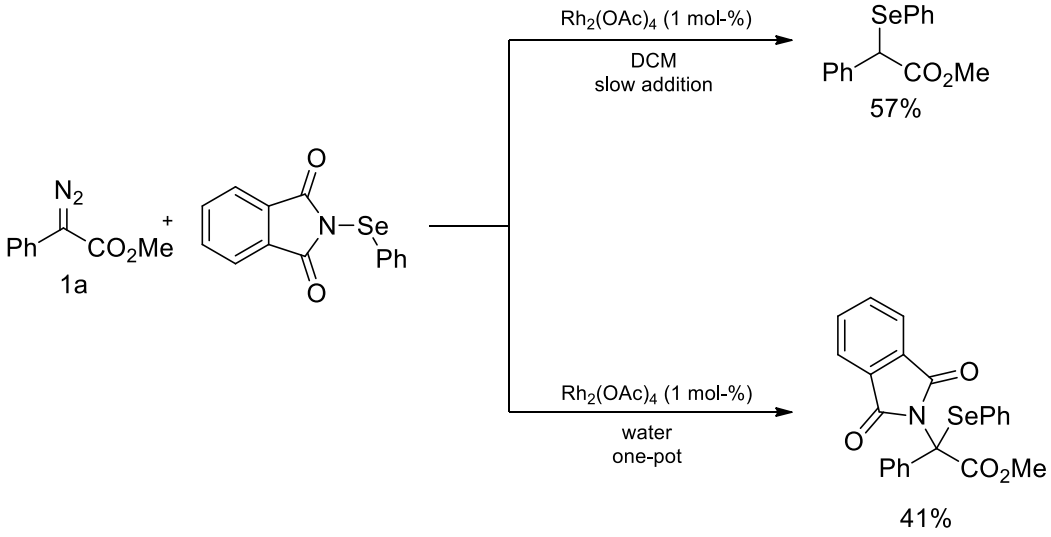

The titled compound was synthesized according to the general procedure-ii. And was obtained after silica gel column chromatography (pentane:diethyl ether – 40:1 → 20:1) as colorless oil (77%, 73 mg).

**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.39 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.15 (dq, *J* = 5.7, 3.4 Hz, 3H), 7.04 (dd, *J* = 3.6, 1.2 Hz, 1H), 6.92 (dd, *J* = 5.3, 3.6 Hz, 1H), 3.75 (s, 2H) ppm.

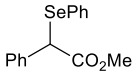
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.1, 138.7, 137.0, 133.1, 128.4, 128.4, 127.7, 127.7, 123.6, 77.1, 76.9, 76.7, 63.1, 53.2 ppm.

**HRMS (ESI):** Mass found: 496.86536, calculated mass for $C_{17}H_{14}NaO_2S_2Se_2^+$: 496.86579.
**IR (KBr):** 3814, 3465, 3097, 2949, 2923, 2853, 2662, 2329, 2109, 1990, 1954, 1792, 1733, 1686, 1594, 1493, 1393, 1323, 1235, 1208, 1173, 1076, 1003, 963, 909, 843, 701 $cm^{-1}$.

## Reactions of N-seleno phthalmide:




## Methyl 2-phenyl-2-(phenylselanyl)acetate (9)



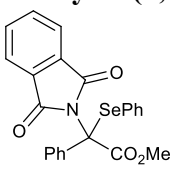

N-seleno phthalmide **8** (0.2 mmol, 1 Eq.) and $Rh_2(OAc)_4$ (1 mol-%) were dissolved in 1.0 mL DCM. Then the diazoalkane (0.2 mmol, 1 Eq.) was dissolved in 1 mL of DCM and added to the reaction mixture over 2h via syringe pump. The resultant reaction mixture was allowed to stir at room temperature another 1 hour. The completion of the reaction was indicated by a colour change of the reaction mixture to light green. The crude product was purified by column chromatography on silica gel using n-pentane : diethyl ether (40 : 1 to 20 : 1) to afford the product **9** as colorless oil (57%, 35 mg).

**$^1$H NMR** (400 MHz, Chloroform-*d*): δ = 7.50 – 7.45 (m, 2H), 7.42 – 7.37 (m, 2H), 7.35 – 7.26 (m, 4H), 7.25 – 7.21 (m, 1H), 4.90 (s, 1H), 3.65 (s, 3H) ppm.

**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 171.5, 136.1, 135.6, 128.9, 128.7, 128.6, 128.4, 127.9, 52.4, 48.1 ppm.

The spectral data is in accordance with the literature: M. Tiecco, L. Testaferri, A. Temperini, L. Bagnoli, F. Marini, C. Santi, Synlett. **2001**, *5*, 706–708.

## Methyl 2-(1,3-dioxoisoindolin-2-yl)-2-phenyl-2-(phenylselanyl)acetate (10)



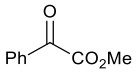

N-seleno phthalmide **8** (0.2 mmol, 1 Eq.) and $Rh_2(OAc)_4$ (1 mol-%) were dissolved in 1.0 mL DCM. Then the diazoalkane (0.2 mmol, 1 Eq.) and another 1 mL of DCM were added to the reaction mixture. The resultant reaction mixture was allowed to stir under open air at room temperature for 2 – 3 hours. The completion of the reaction was indicated by a colour change of the reaction mixture to light green. The crude product was purified by column chromatography on silica gel using n-pentane : diethyl ether (40 : 1 to 20 : 1) to afford the product **10** as colorless oil (41%, 37 mg).

**$^1$H NMR** (400 MHz, Chloroform-*d*): δ = 7.37 (dd, *J* = 7.9, 1.3 Hz, 3H), 7.33 – 7.26 (m, 2H), 7.21 – 7.03 (m, 9H), 3.64 (s, 3H) ppm.

**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 174.2, 171.2, 137.9, 137.0, 129.3, 129.1, 128.6, 128.4, 127.4, 127.3, 77.1, 76.9, 76.7, 59.8, 52.9 ppm.

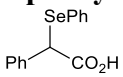
**HRMS (ESI):** Mass found: 474.02133, calculated mass for $C_{23}H_{17}NNaO_4Se^+$: 474.02150.
**IR (KBr):** 3425, 3057, 2949, 2846, 2648, 2333, 2084, 2007, 1959, 1878, 1816, 1719, 1577, 1474, 1436, 1323, 1230, 1066, 1010, 914, 856, 773, 736, 688 $cm^{-1}$.

## Methyl 2-oxo-2-phenylacetate (11)



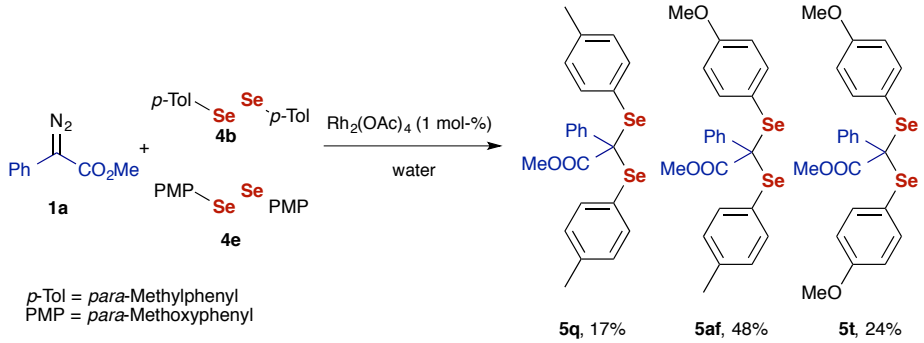
A solution of seleno-ketal **5a** (0.5 mmol, 1 Eq.) in chloroform (5 mL) was treated dropwise with $H_2O_2$ (4 mmol, 8 Eq.) at 0 °C. The reaction was stirred at the same temperature for 2 h. The solution was washed twice with water, dried and concentrated. The crude product was purified by silica gel column chromatography using n-pentane : diethyl ether (40 : 1 - 20 : 1). and isolated as a colorless oil (63%, 52 mg).
**$^1$H NMR** (400 MHz, Chloroform-*d*): δ 8.05 – 7.97 (m, 2H), 7.69 – 7.63 (m, 1H), 7.57 – 7.47 (m, 1H), 3.98 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 186.0, 164.0, 134.9, 132.4, 130.0, 128.8, 52.7 ppm.
The spectral data is in accordance with the literature: T. Huang, T. Chen, L. Han, *J. Org. Chem.* **2018**, *83*, 2959−2965.

## 2-phenyl-2-(phenylselanyl)acetic acid (12)



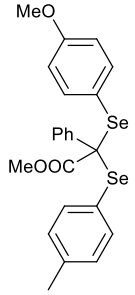
To a stirred solution of seleno ketal **5a** (0.5 mmol, 1 Eq.) in methanol (5 mL) and water (0.5 mL) was added NaOH (5 mmol, 10 Eq.) under open air at RT. The reaction mixture was stirred for 4h and completion of the reaction was monitored by TLC. Methanol was evaporated in reduced pressure and then the crude mixture was acidified by 1N HCl until pH 6. Finally aqueous mixture was extracted with ethyl acetate and purified by silica gel column chromatography (hexane : ethyl acetate – 1:2) as light yellow solid (51%, 74mg).
**$^1$H NMR** (400 MHz, Chloroform-*d*): δ = 10.70 (br s, 1H), 7.53 – 7.46 (m, 2H), 7.43 – 7.37 (m, 2H), 7.34 – 7.17 (m, 6H), 4.85 (s, 3H) ppm.
**$^{13}$C NMR** (151 MHz, Chloroform-*d*): δ = 176.9, 135.6, 129.1, 128.8, 128.7, 128.5, 128.1, 47.8 ppm.
The spectral data is in accordance with the literature: M. Tiecco, L. Testaferri, A. Temperini, L. Bagnoli, F. Marini, C. Santi, Synlett. **2001**, *5*, 706–708.

## Control Experiments:




1,2-di-p-tolyldiselane (0.14 mmol, 0.35 equiv), 1,2-bis(4-methoxyphenyl)diselane (0.14 mmol, 0.35 equiv) and $Rh_2(OAc)_4$ (0.5 mol-%) were suspended in 3 mL of water. Then the diazoalkane (0.4 mmol, 1 Eq.) and another 1.5 mL of water were added to the reaction mixture. The resultant reaction mixture was allowed to stir under open air at room temperature for 3 hours. The completion of the reaction was indicated by a color change of the reaction mixture to yellowish green. The reaction mixture was extracted with diethyl ether and the crude product was purified by column chromatography on silica gel using n-pentane : diethyl ether (40 : 1 to 20 : 1) to afford the three different cross coupling products **5p** -17% (33 mg), **5af**- 48% (97 mg), **5s**- 24% (50mg) respectively.

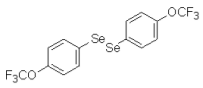
**Methyl 2-((4-methoxyphenyl)selanyl)-2-phenyl-2-(p-tolylselanyl)acetate (5af)**

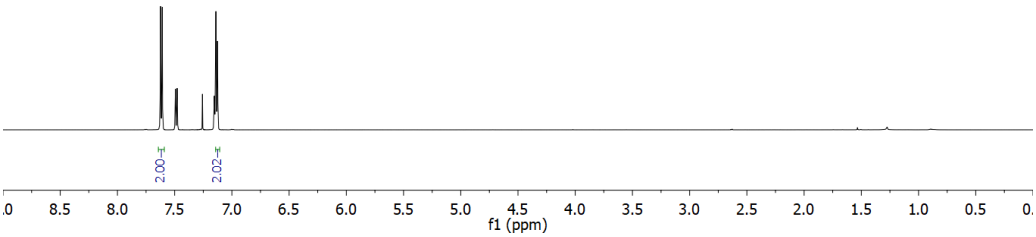



**$^{1}$H NMR** (400 MHz, Chloroform-*d*): δ = 7.31 – 7.23 (m, 4H), 7.16 – 7.03 (m, 5H), 7.02 – 6.96 (m, 2H), 6.73 – 6.65 (m, 2H), 3.77 (s, 3H), 3.66 (s, 3H), 2.30 (s, 3H) ppm.
**$^{13}$C NMR** (101 MHz, Chloroform-*d*): δ = 171.2, 160.6, 139.3, 139.1, 138.2, 137.1, 129.3, 128.6, 127.4, 127.2, 125.8, 119.8, 114.0, 77.3, 77.0, 76.6, 55.1, 52.9, 21.3 ppm.
**HRMS (ESI):** Mass found: 528.97876, calculated mass for $C_{23}H_{22}NaO_3Se_2^+$: 528.97916.
**IR (KBr):** 3015, 2948, 2840, 2336, 2193, 2084, 1893, 1798, 1719, 1585, 1487, 1442, 1400, 1288, 1239, 1175, 1102, 1073, 1016, 913, 693 $cm^{-1}$.

## 1,2-bis(4-(trifluoromethoxy)phenyl)diselane

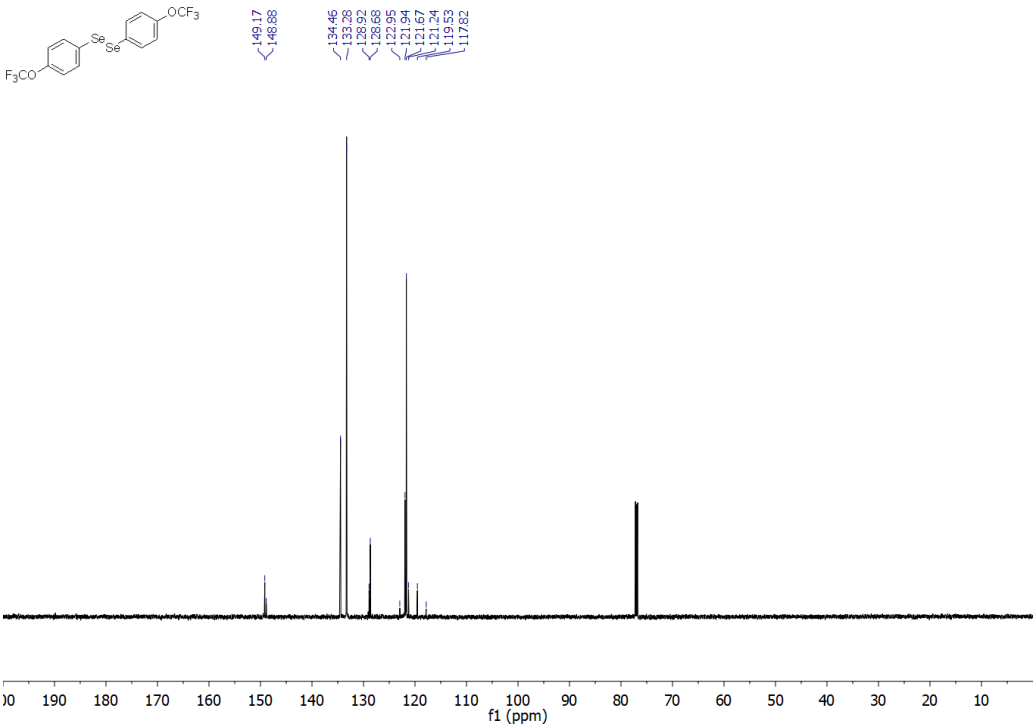
$^{1}$H NMR (400 MHz, Chloroform-*d*):



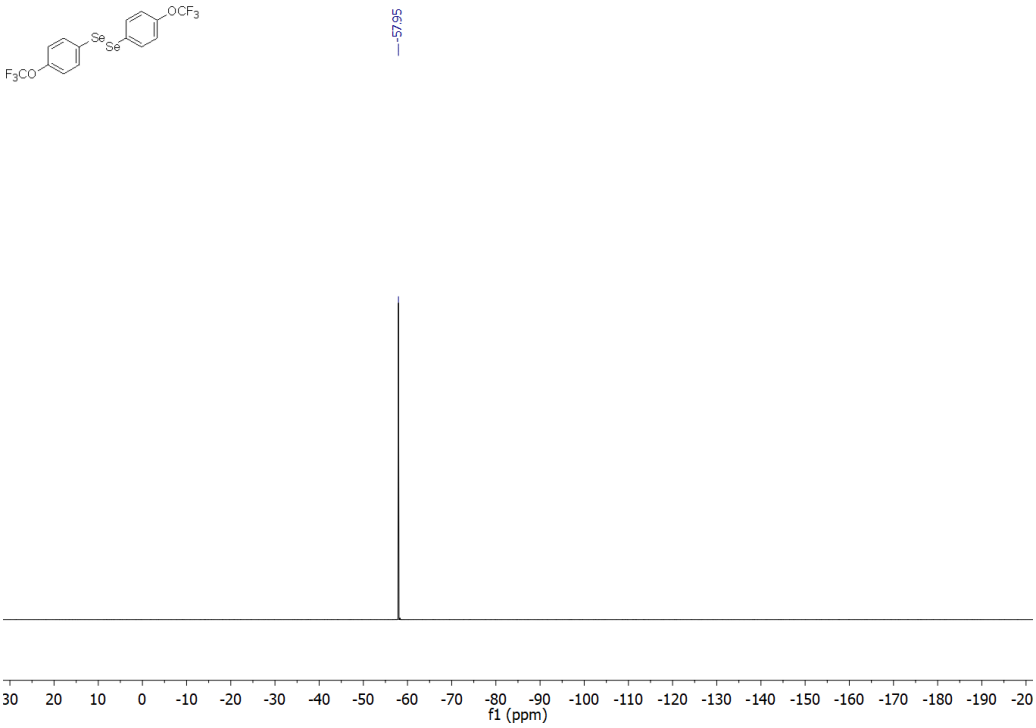




$^{13}$C NMR (151 MHz, Chloroform-*d*):



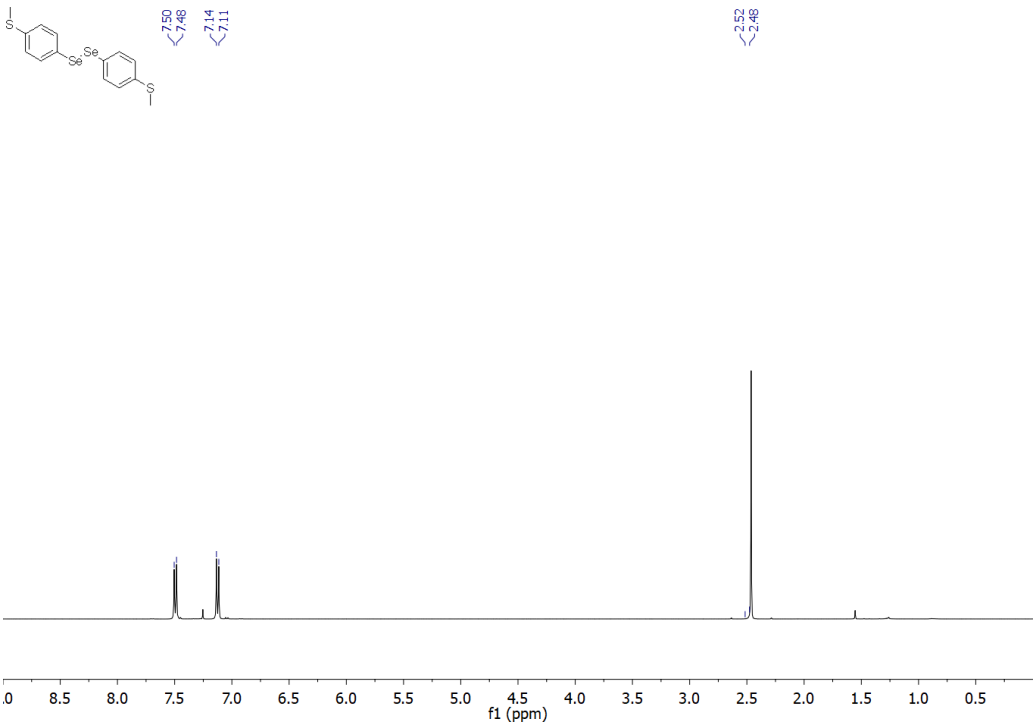



$^{19}$F NMR (376 MHz, Chloroform-*d*):

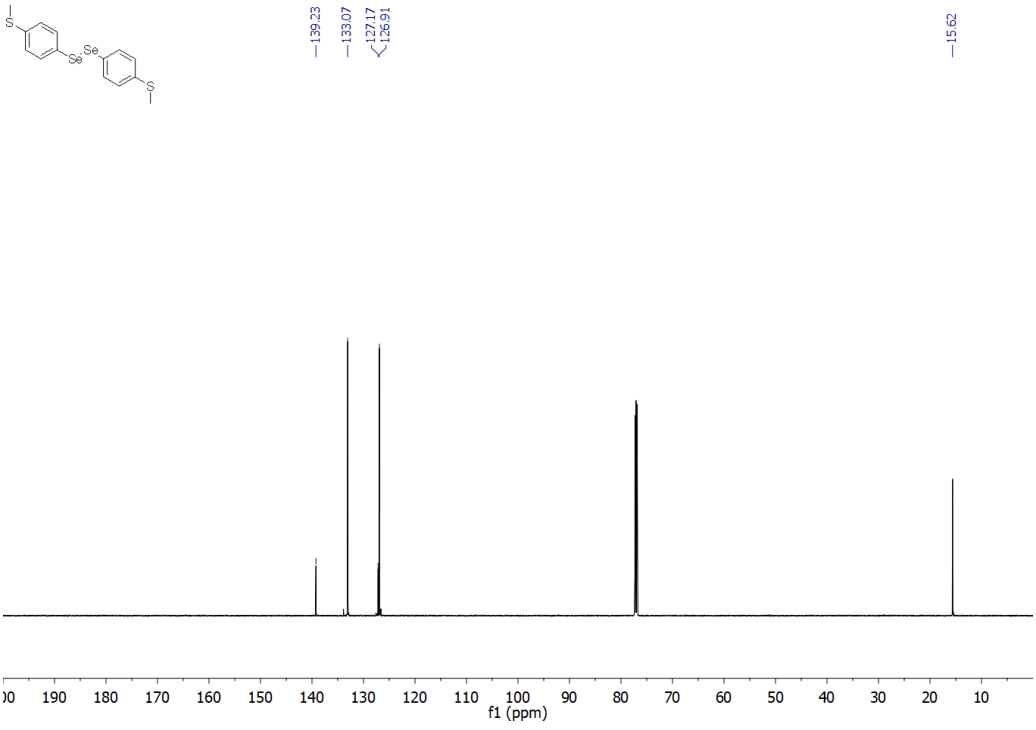


## 1,2-bis(4-(methylthio)phenyl)diselane

$^{1}$H NMR (400 MHz, Chloroform-*d*):

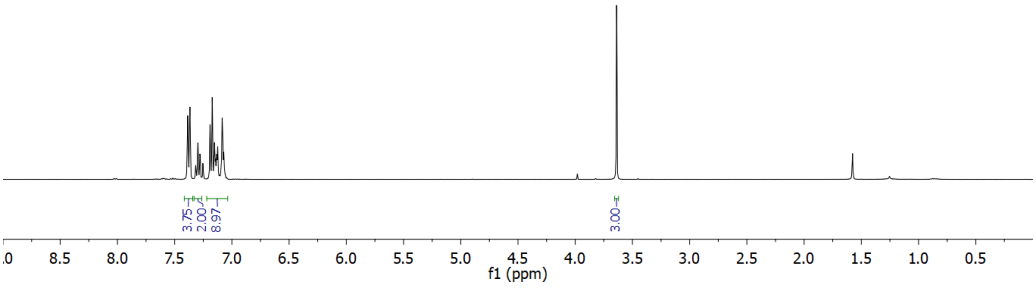



$^{13}$C NMR (151 MHz, Chloroform-*d*):



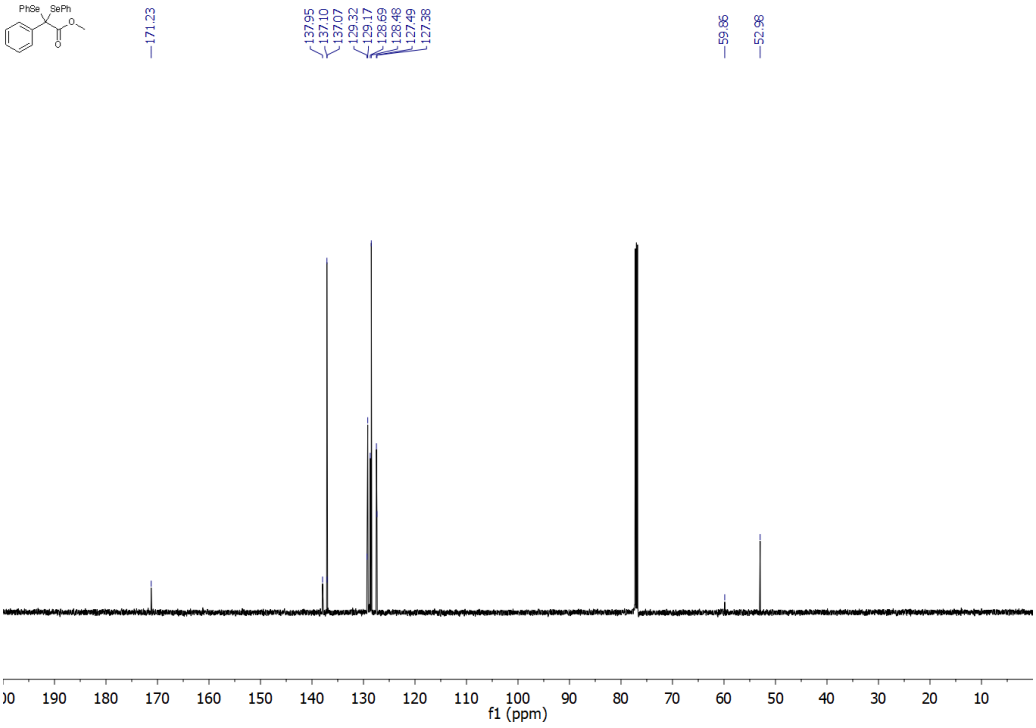
## Methyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5a)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

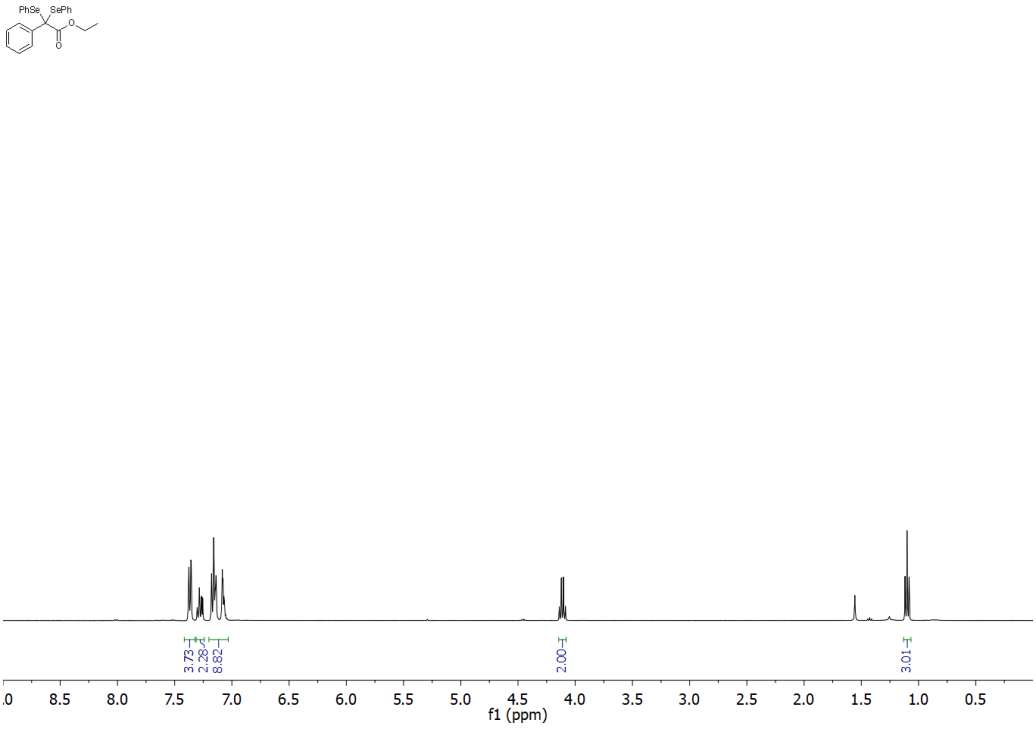

$^{13}$C NMR (151 MHz, Chloroform-*d*):



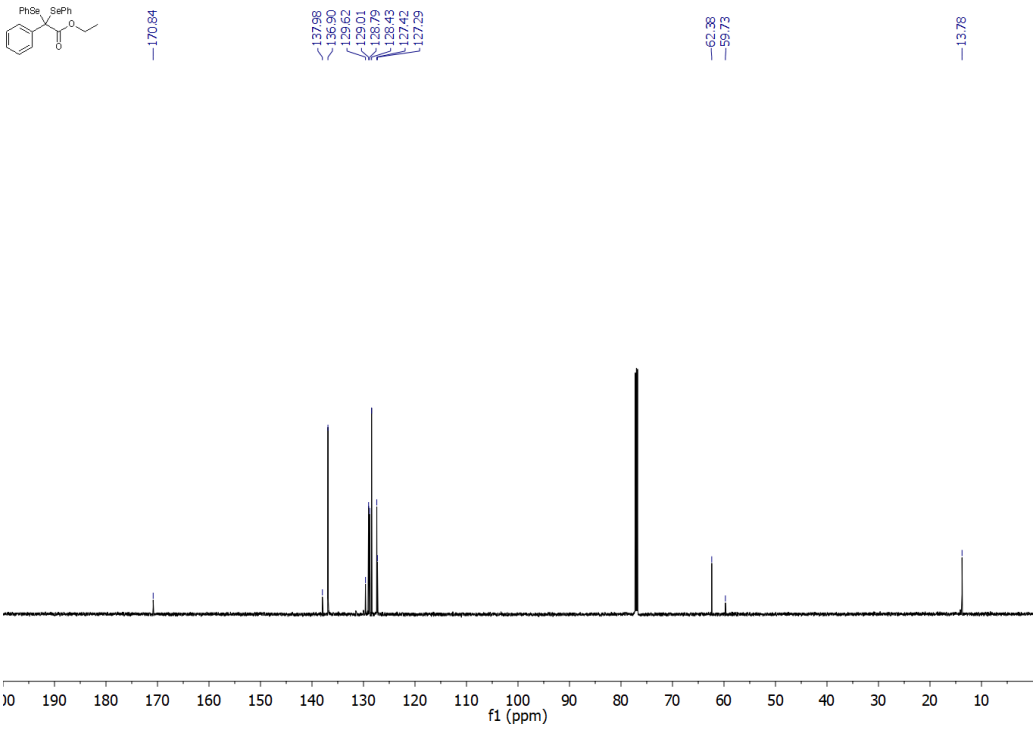



## Ethyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5b)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

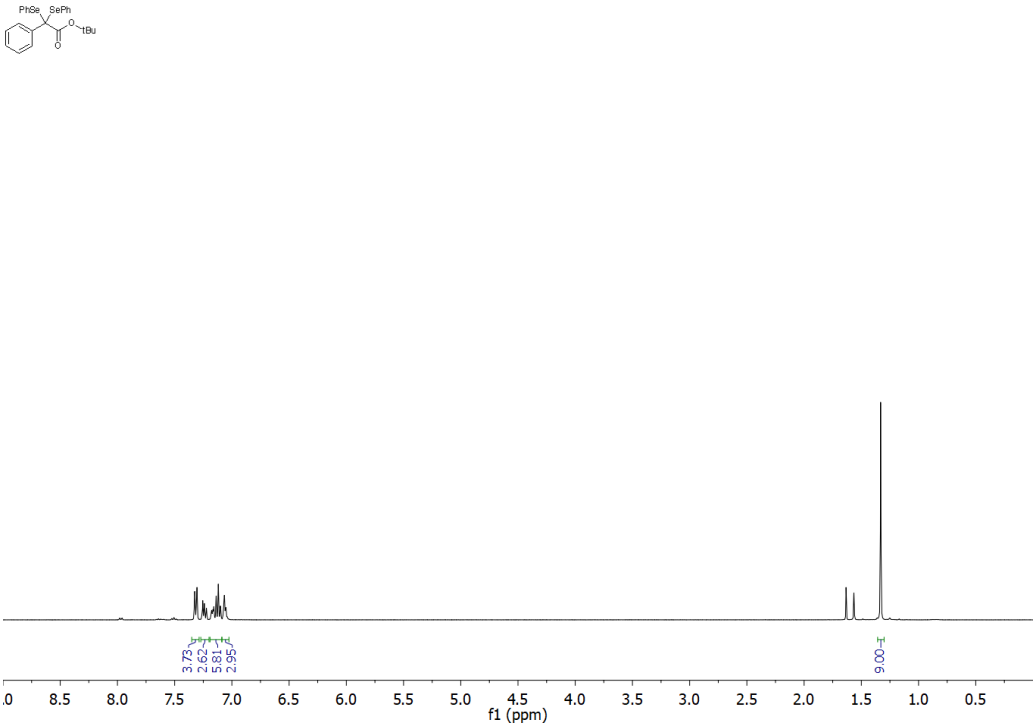



$^{13}$C NMR (151 MHz, Chloroform-*d*):

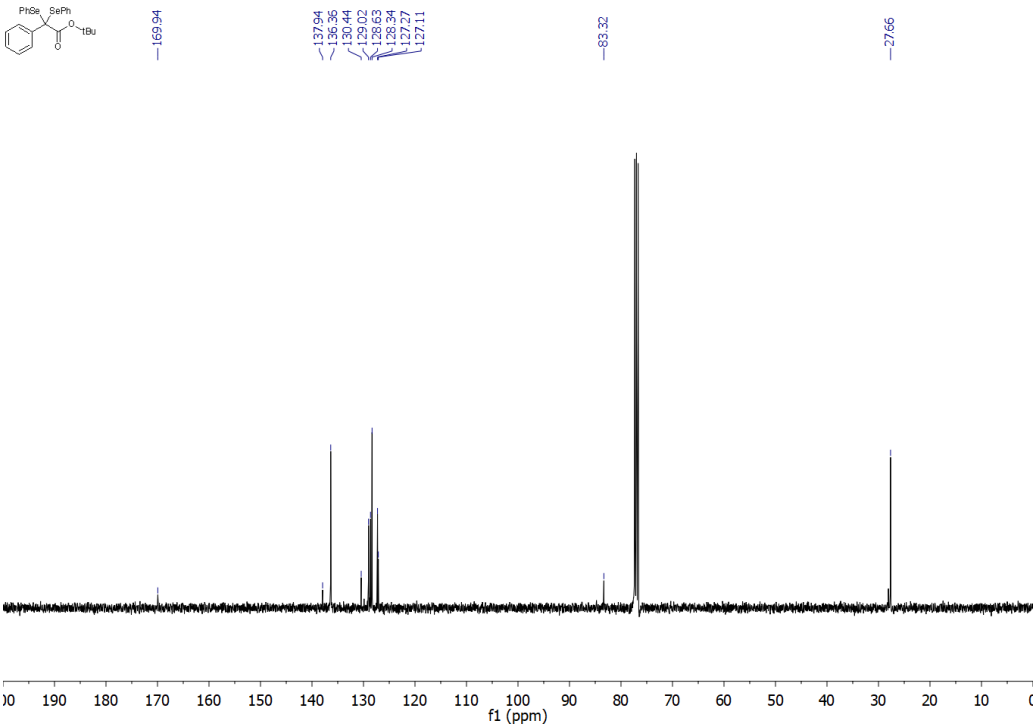


## tert-butyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5c)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

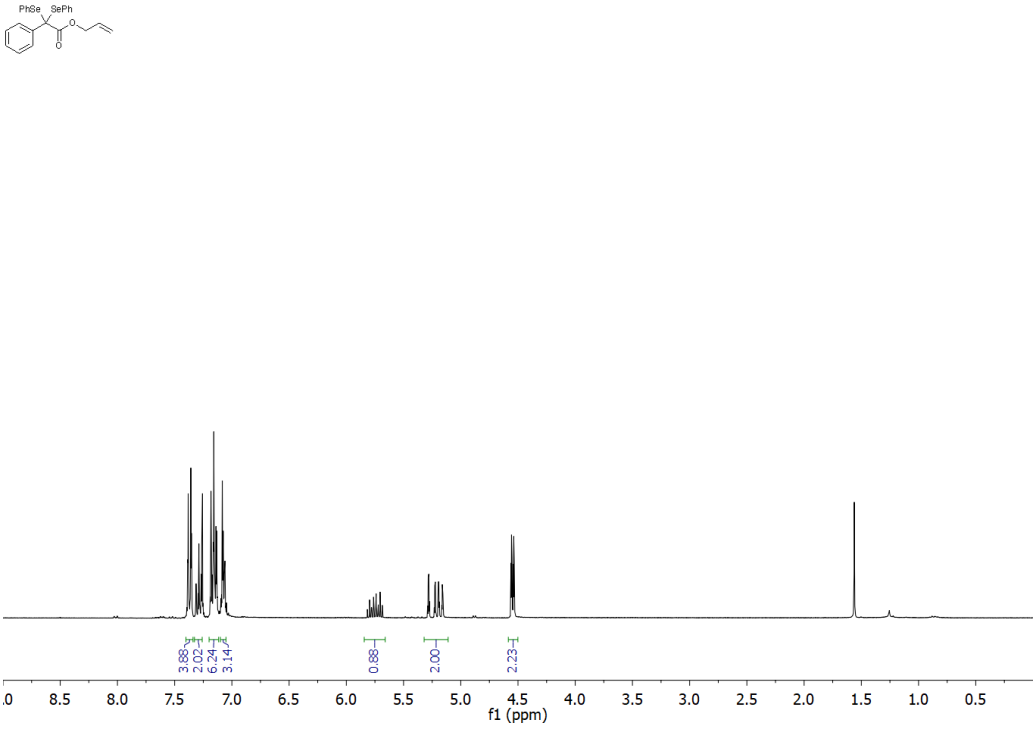



$^{13}$C NMR (101 MHz, Chloroform-*d*):

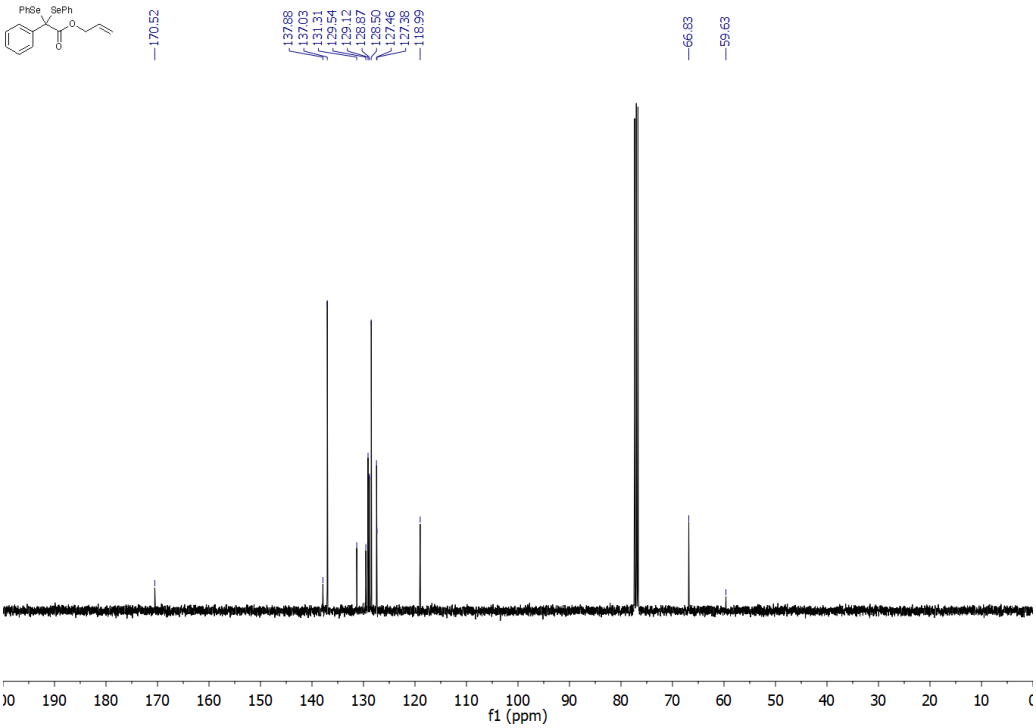


## allyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5d)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

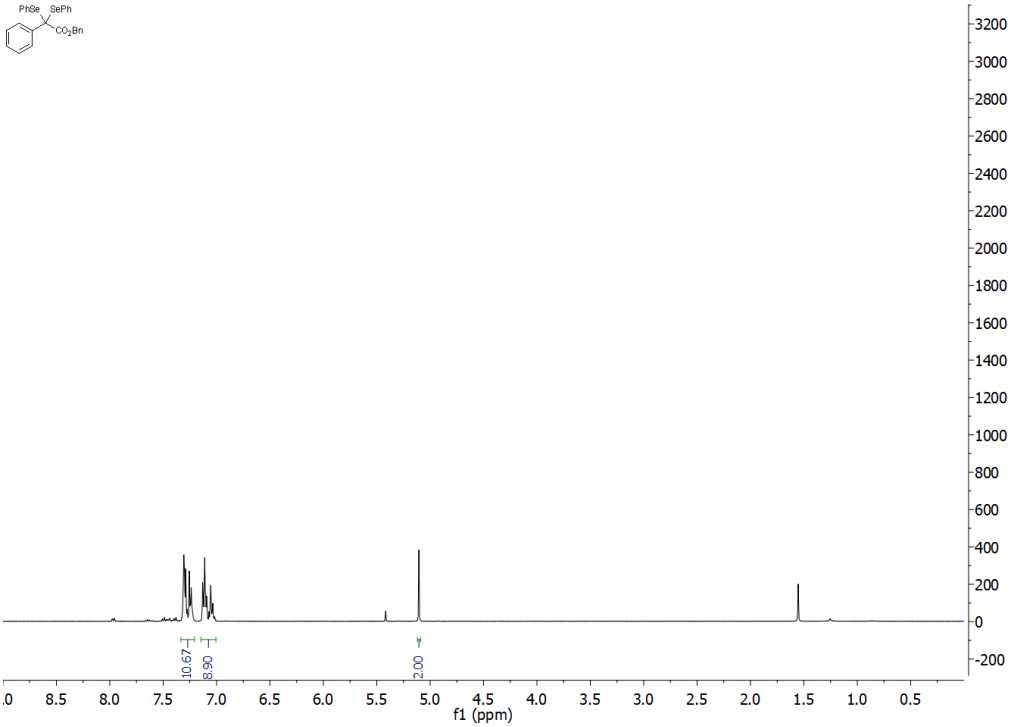



$^{13}$C NMR (101 MHz, Chloroform-*d*):

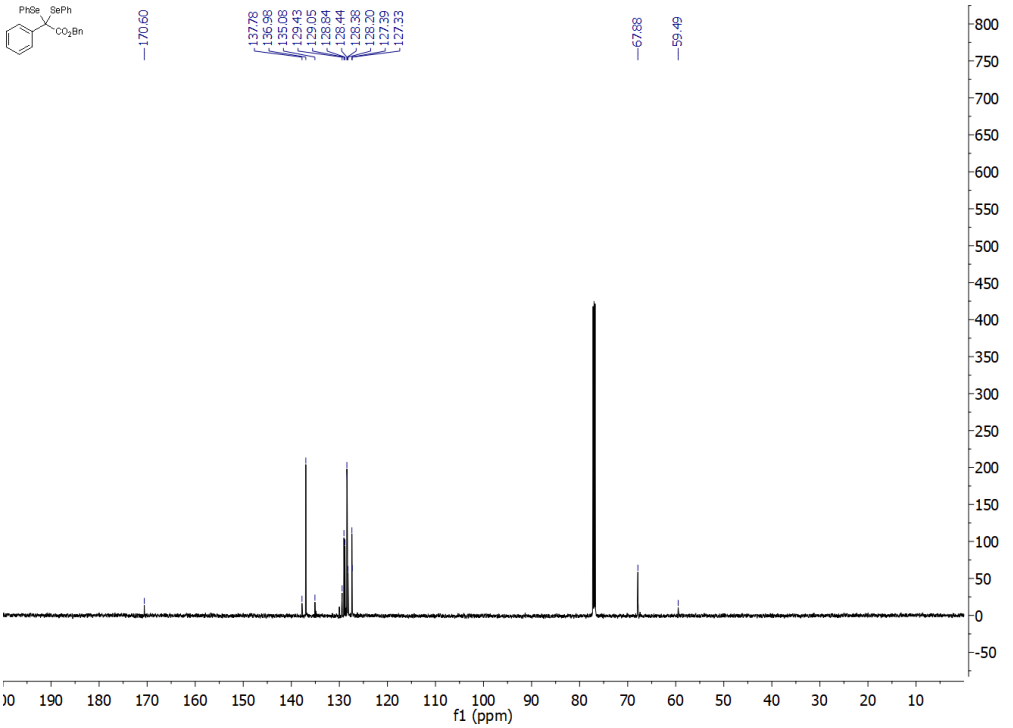


## Benzyl 2-phenyl-2,2-bis(phenylselanyl)acetate (5e)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

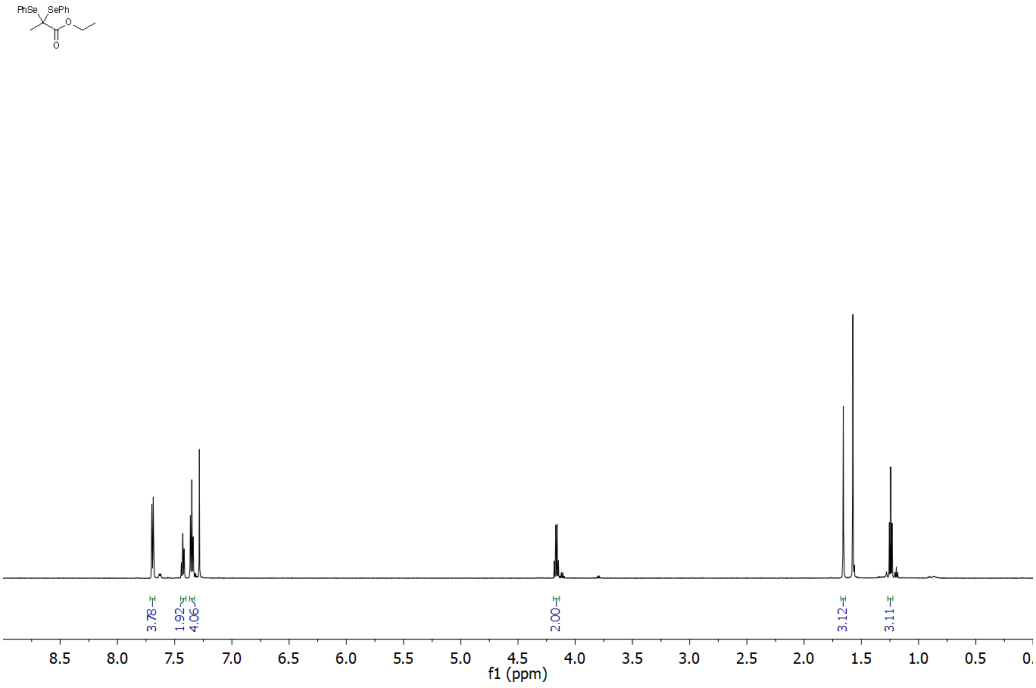



$^{13}$C NMR (151 MHz, Chloroform-*d*):

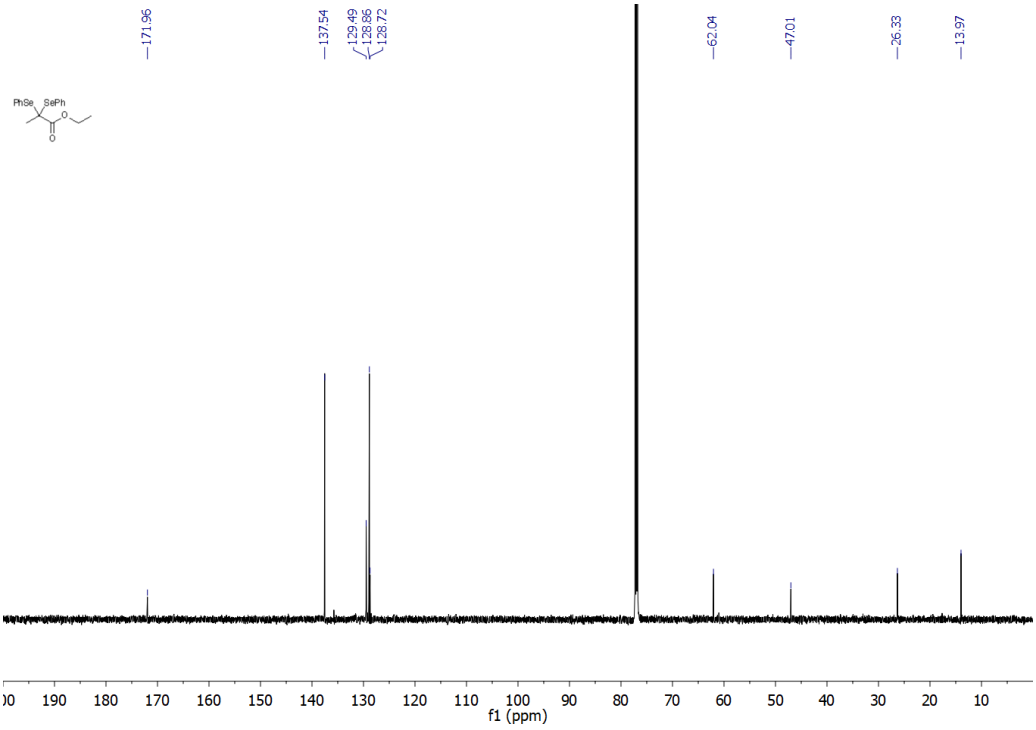


## Ethyl 2,2-bis(phenylselanyl)propanoate (5f)

$^{1}$H NMR (600 MHz, Chloroform-*d*):

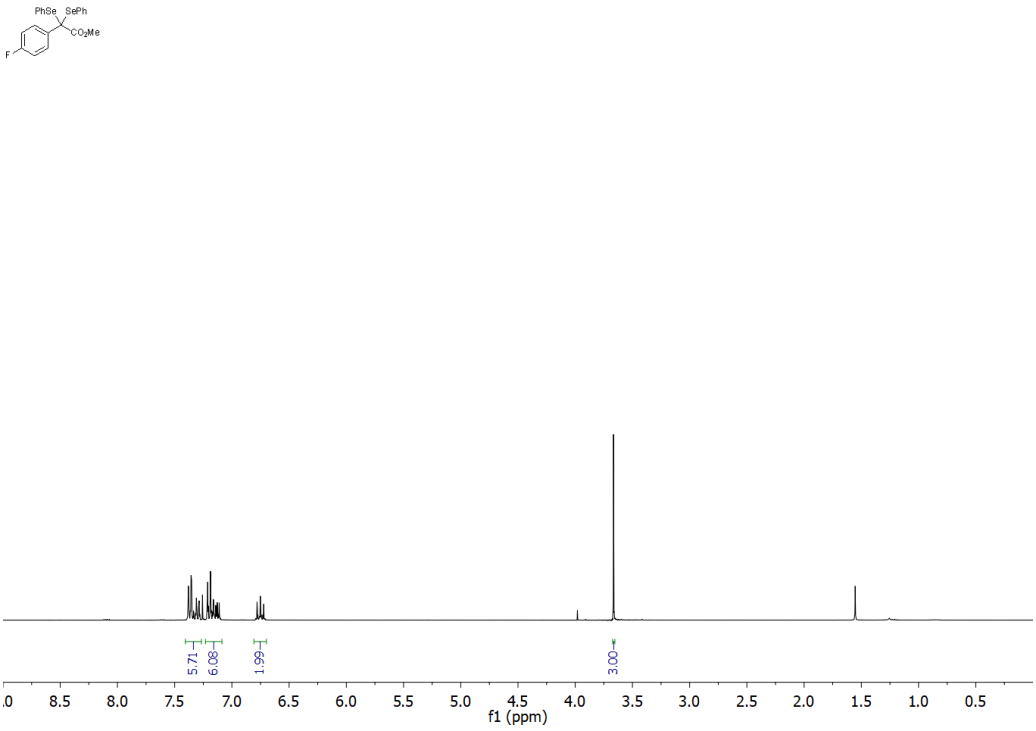



$^{13}$C NMR (151 MHz, Chloroform-*d*):

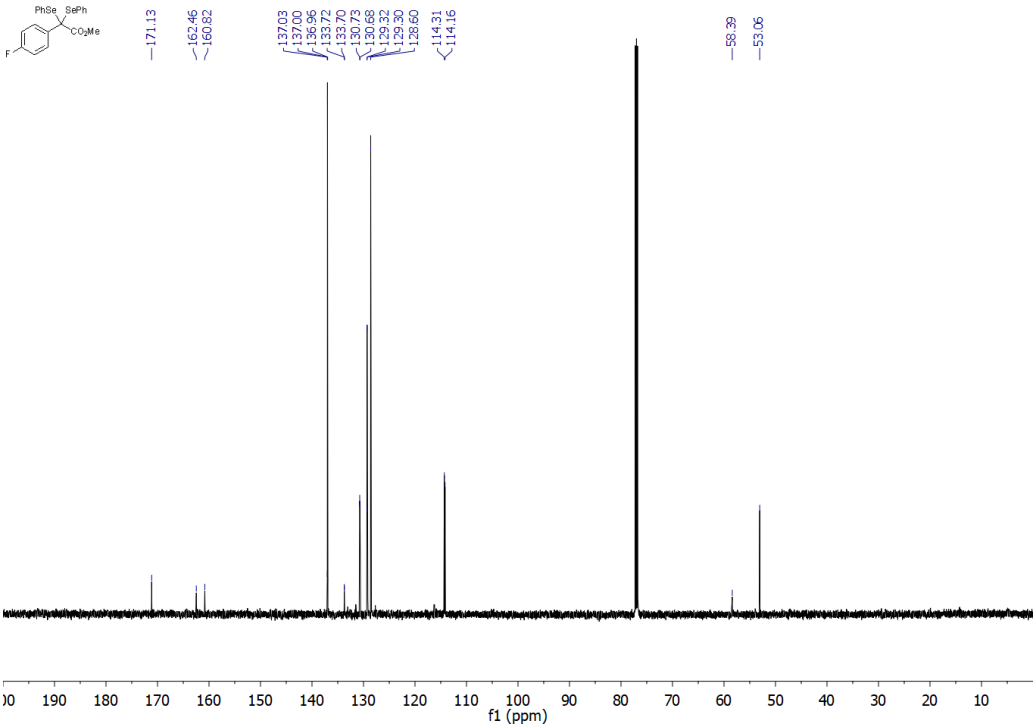


## Methyl 2-(4-fluorophenyl)-2,2-bis(phenylselanyl)acetate (5g)

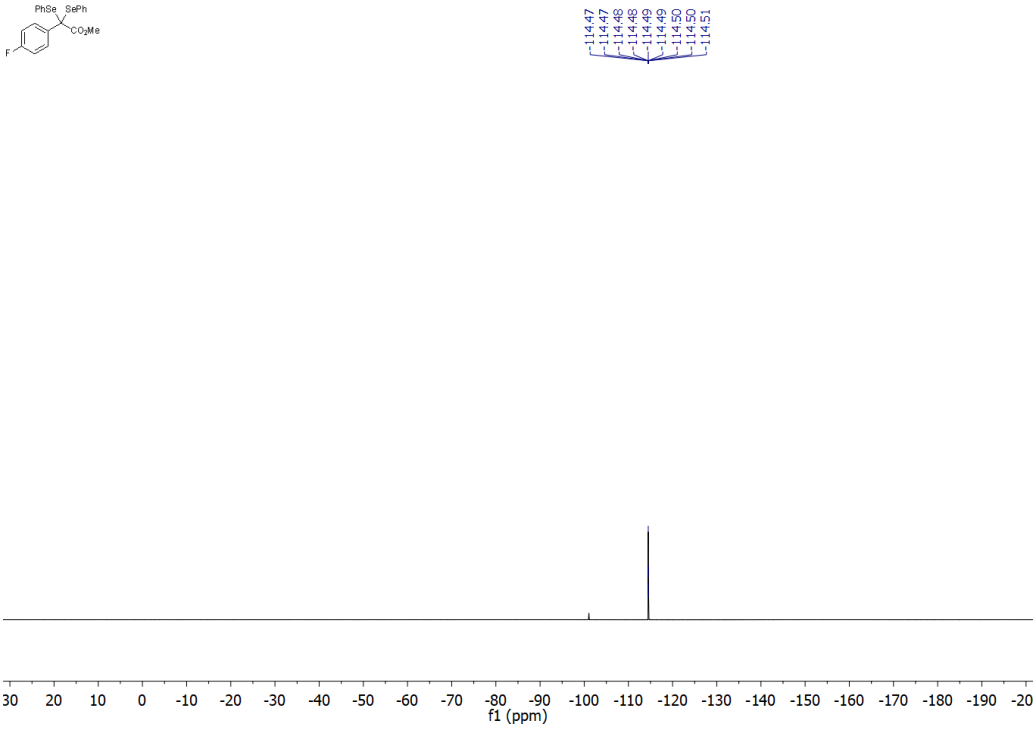
$^{1}$H NMR (300 MHz, Chloroform-*d*):




$^{13}$C NMR (151 MHz, Chloroform-*d*):

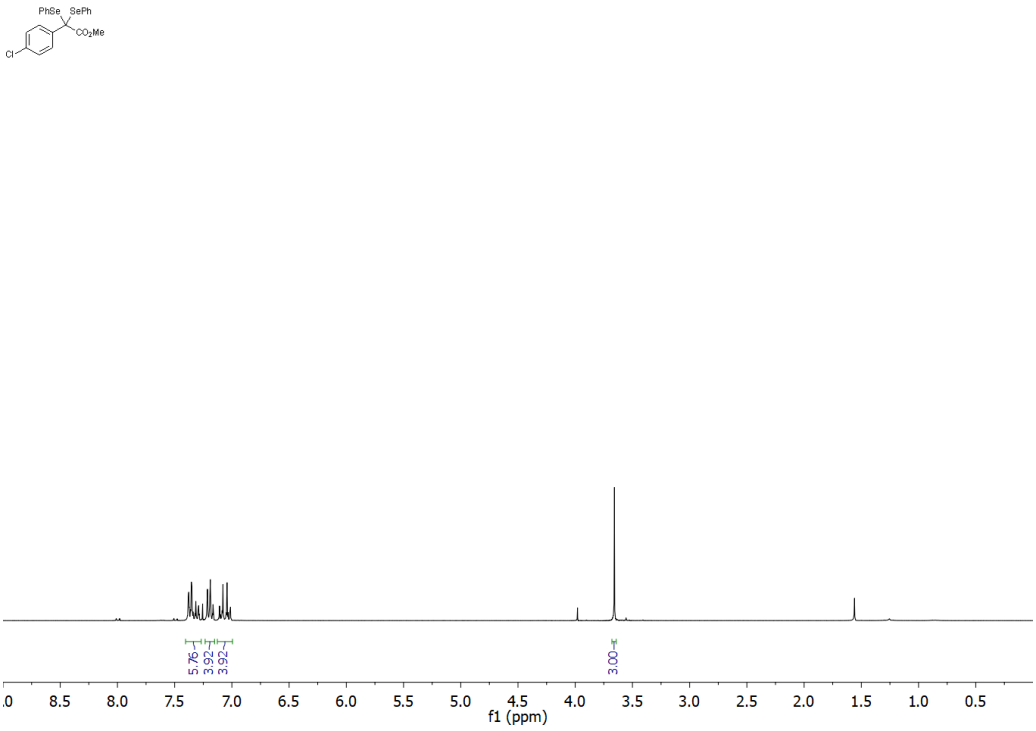


$^{19}$F NMR (564 MHz, Chloroform-*d*):

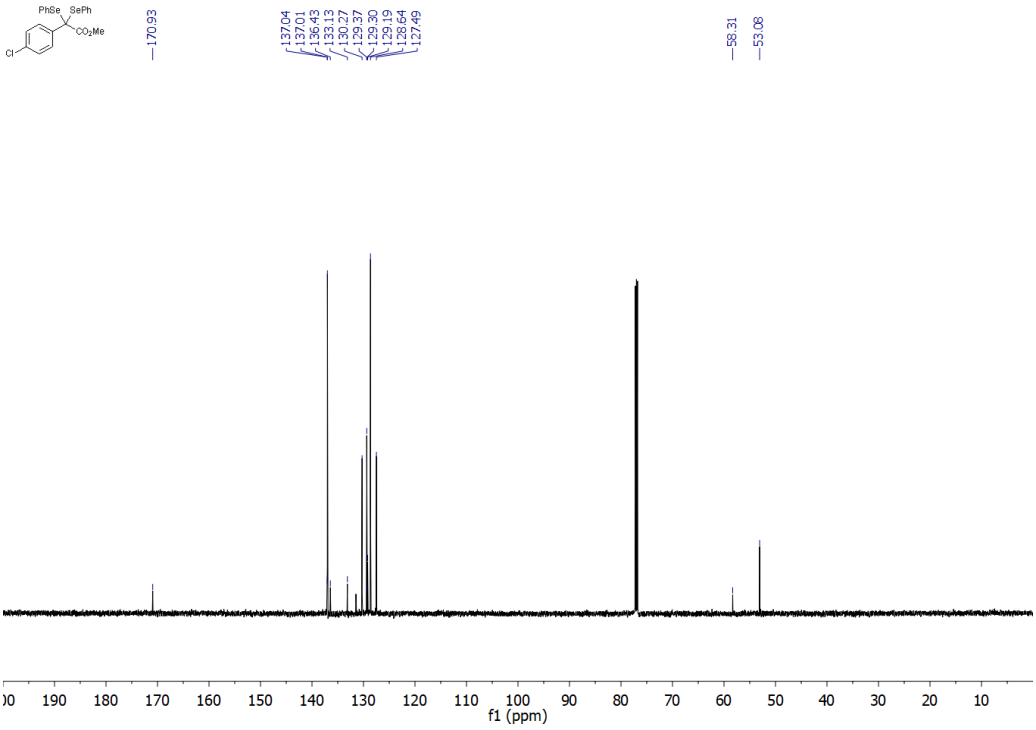


## Methyl 2-(4-chlorophenyl)-2,2-bis(phenylselanyl)acetate (5h)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

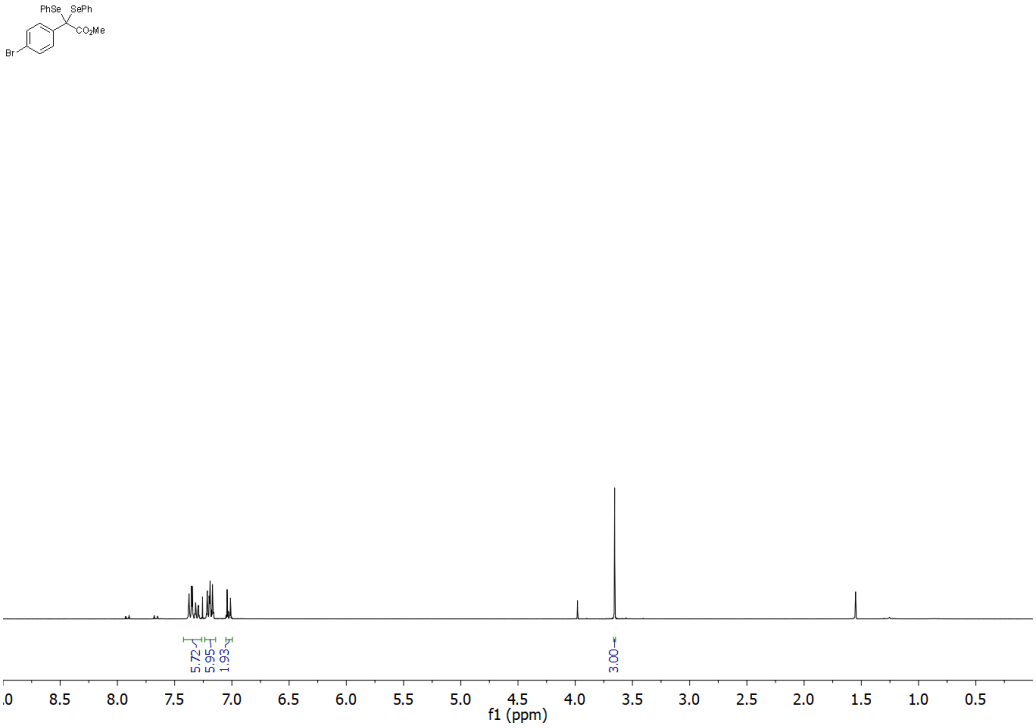



$^{13}$C NMR (151 MHz, Chloroform-*d*):

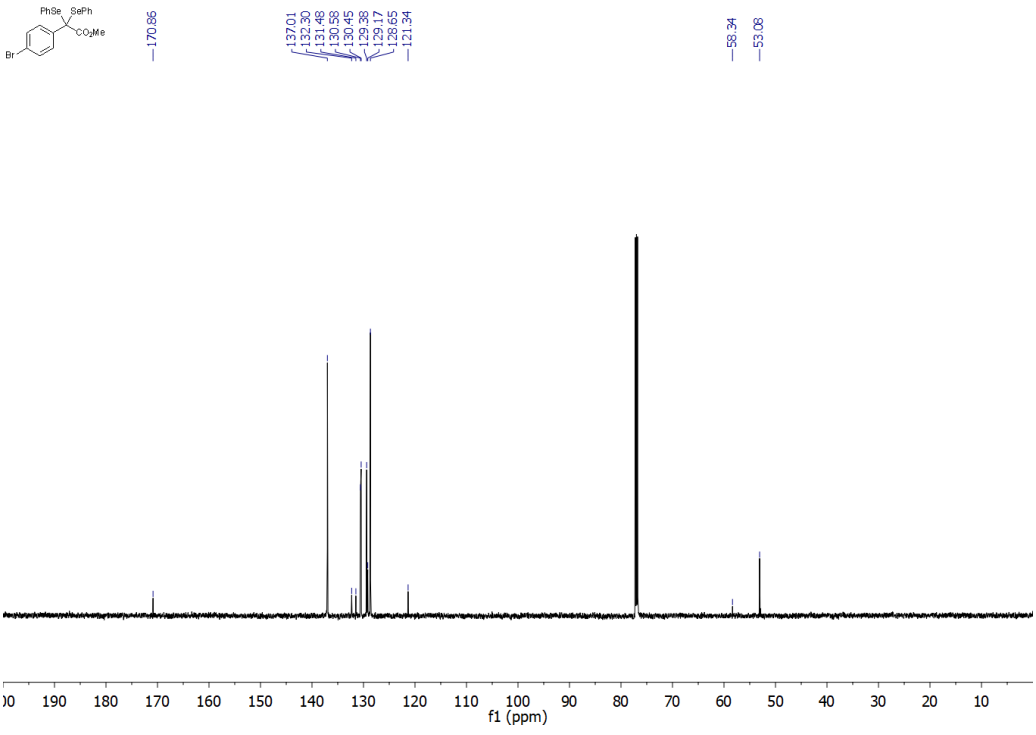


## Methyl 2-(4-bromophenyl)-2,2-bis(phenylselanyl)acetate (5i)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

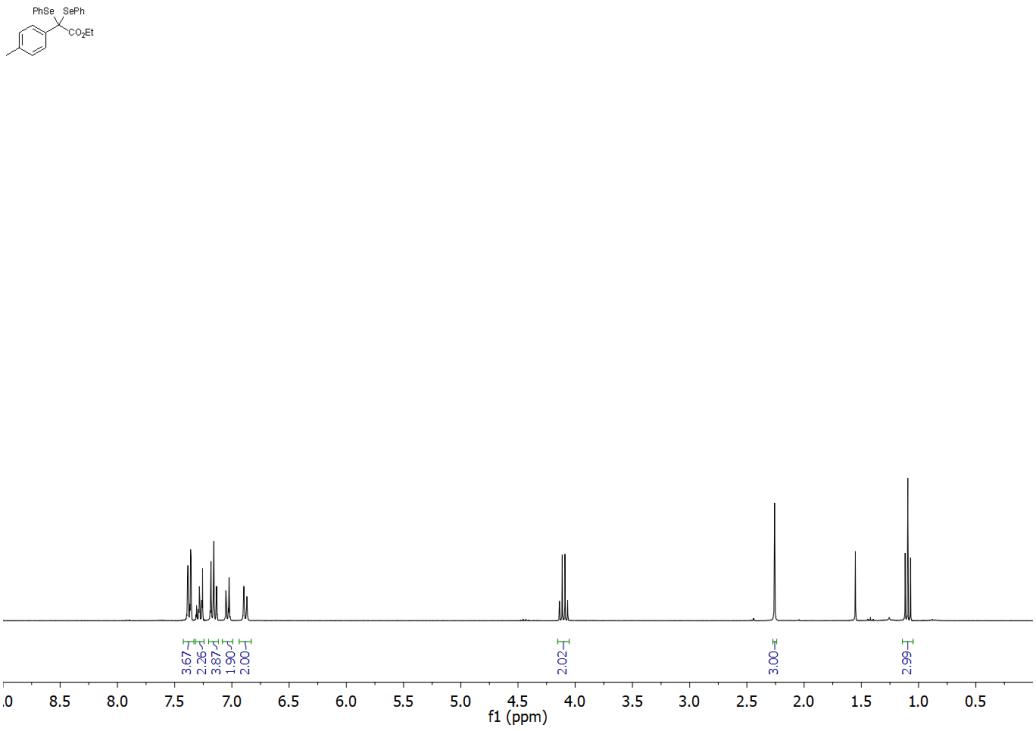



$^{13}$C NMR (151 MHz, Chloroform-*d*):

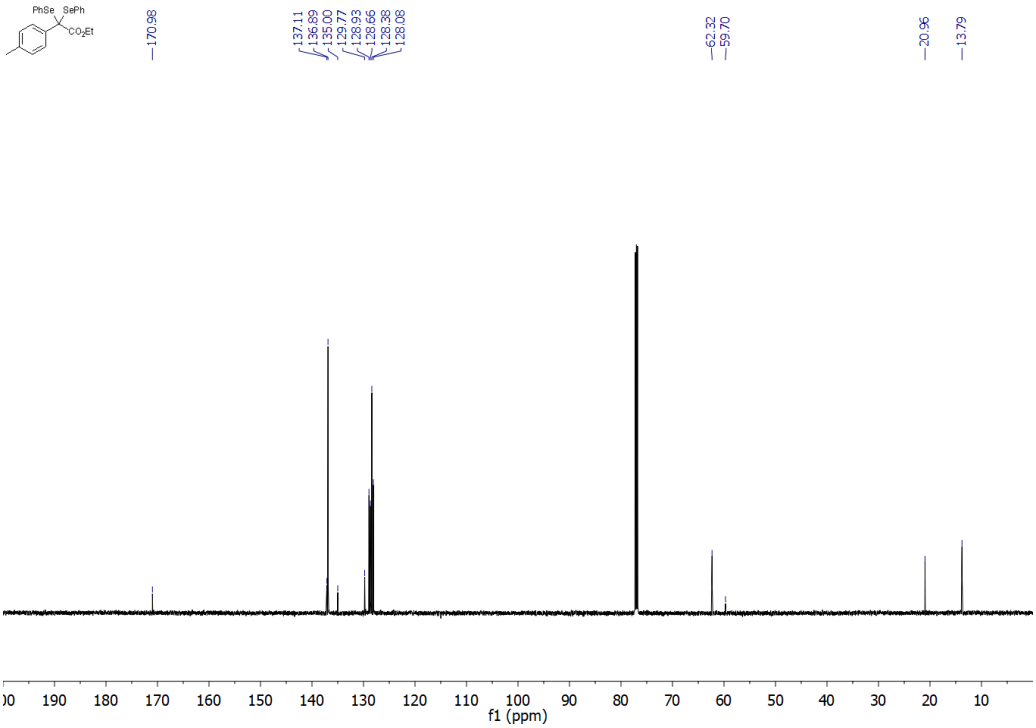


## Ethyl 2,2-bis(phenylselanyl)-2-(p-tolyl)acetate (5j)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

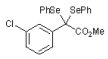


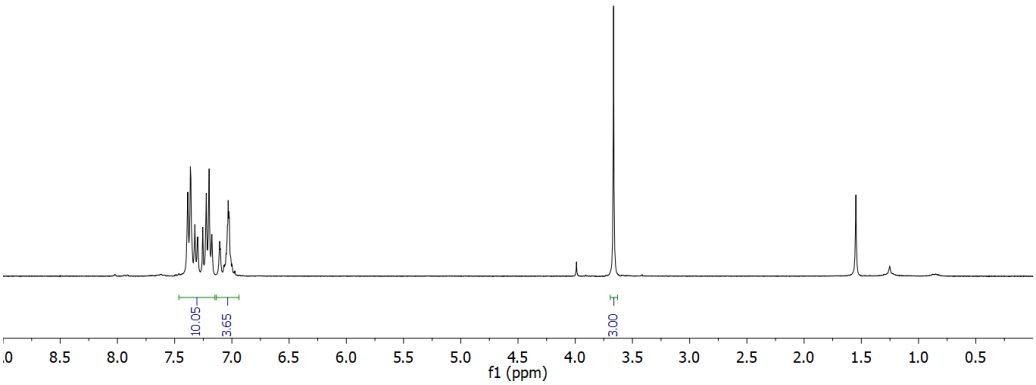

$^{13}$C NMR (151 MHz, Chloroform-*d*):



## Methyl 2-(3-chlorophenyl)-2,2-bis(phenylselanyl)acetate (5k)

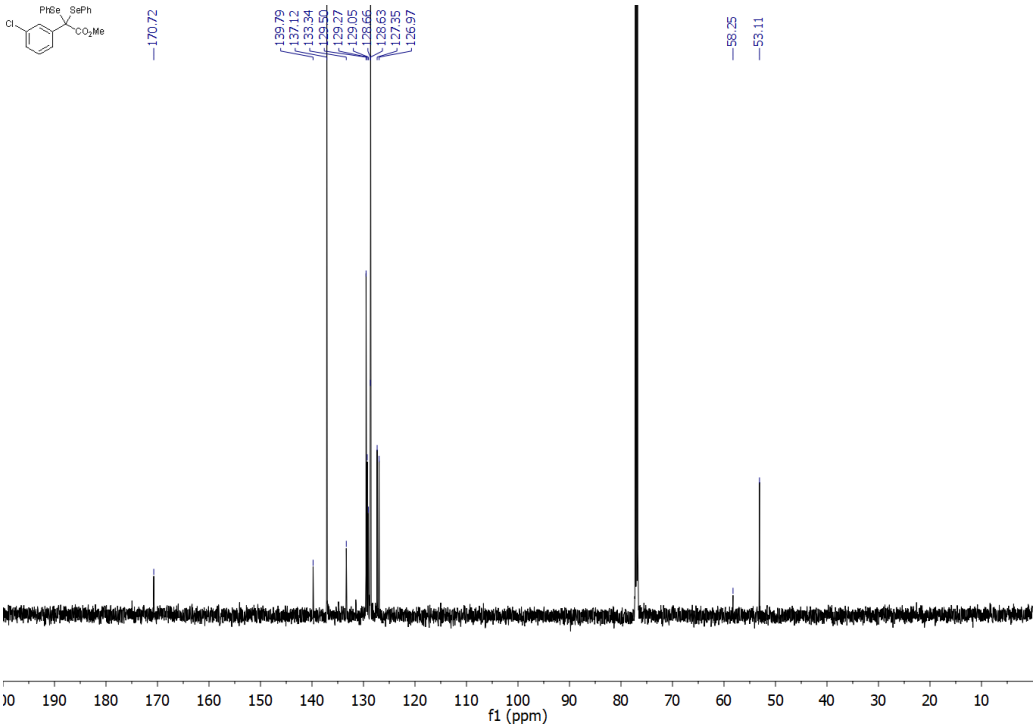
$^{1}$H NMR (300 MHz, Chloroform-*d*):



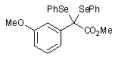

$^{13}$C NMR (151 MHz, Chloroform-*d*):

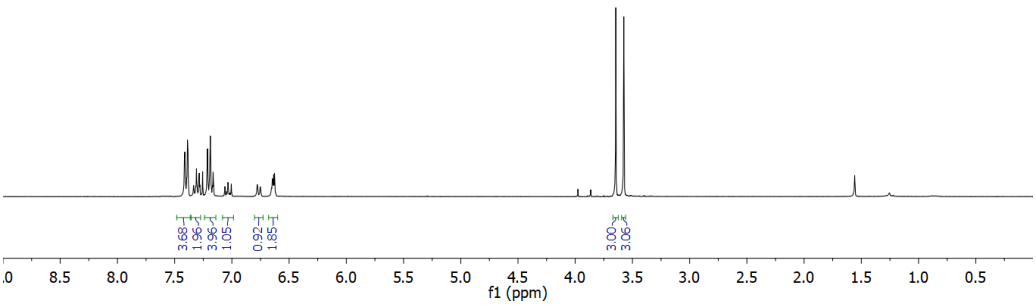


## Methyl 2-(3-methoxyphenyl)-2,2-bis(phenylselanyl)acetate (5l)

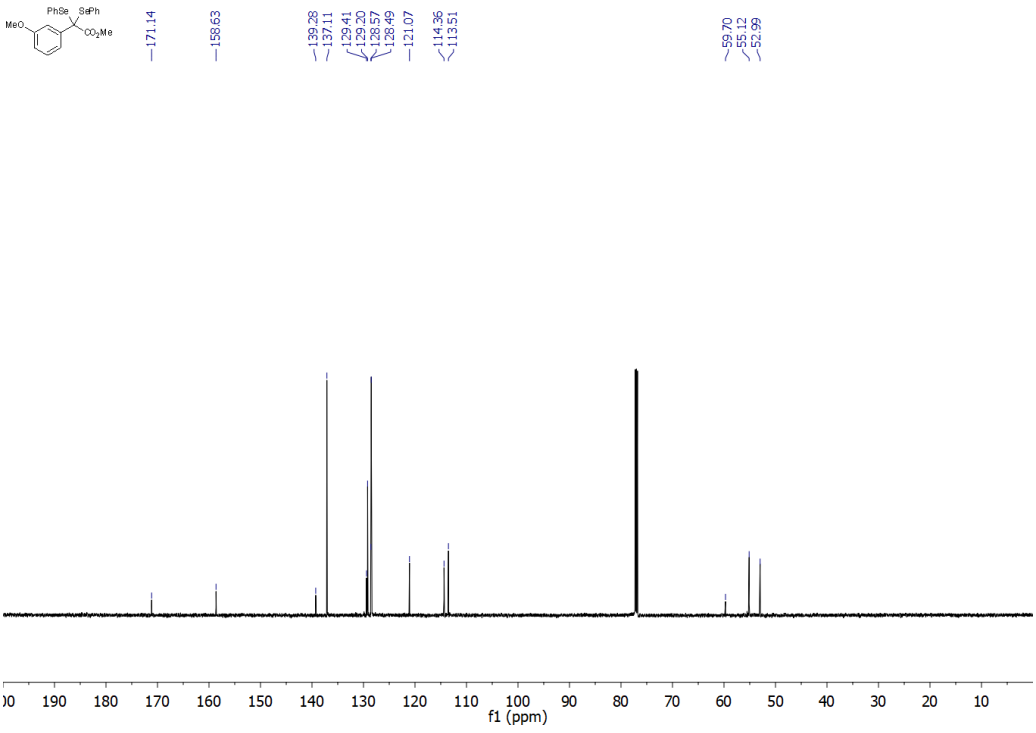
$^{1}$H NMR (300 MHz, Chloroform-*d*):



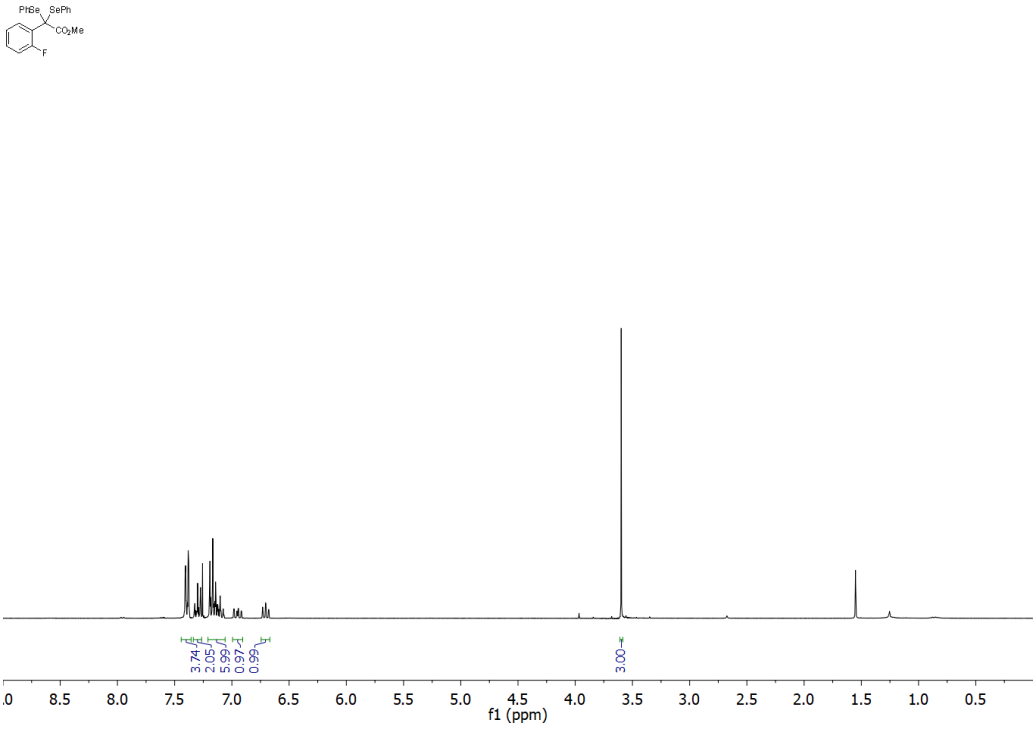

$^{13}$C NMR (151 MHz, Chloroform-*d*):



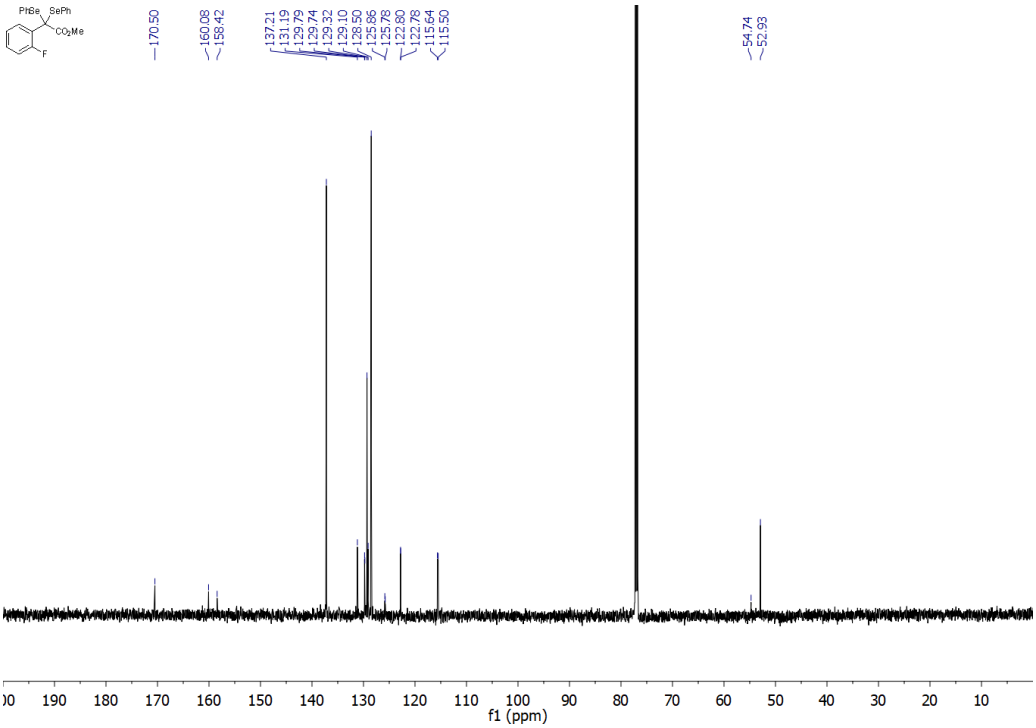



## Methyl 2-(2-fluorophenyl)-2,2-bis(phenylselanyl)acetate (5m)

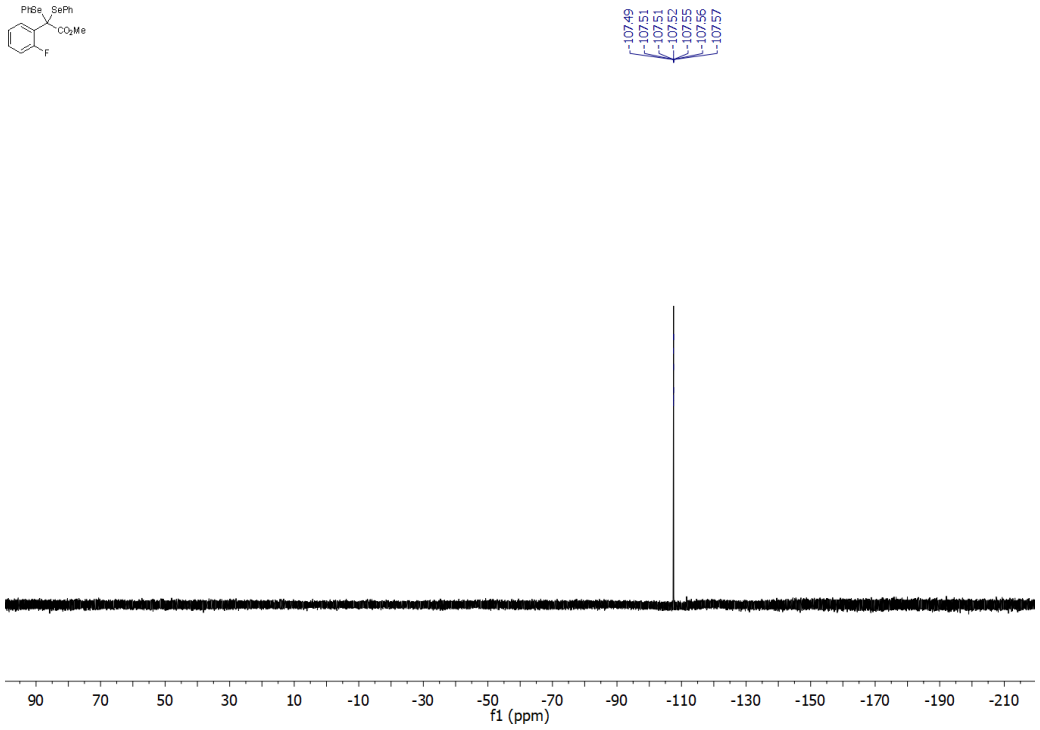
$^{1}$H NMR (300 MHz, Chloroform-*d*):




$^{13}$C NMR (151 MHz, Chloroform-*d*):

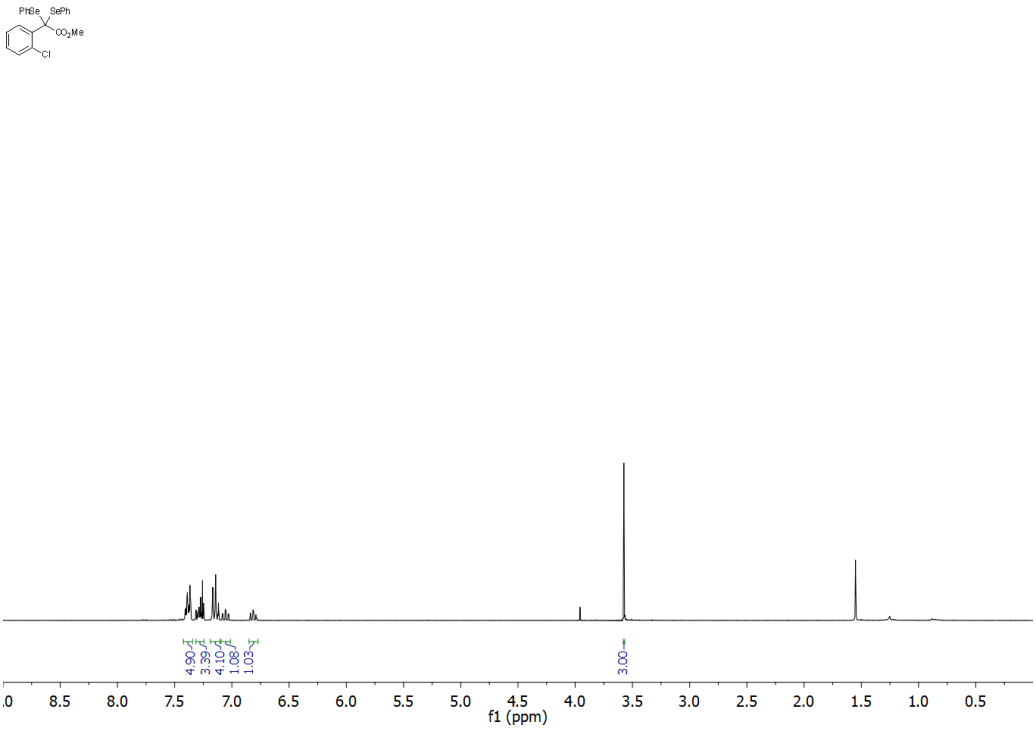


$^{19}$F NMR (282 MHz, Chloroform-*d*):

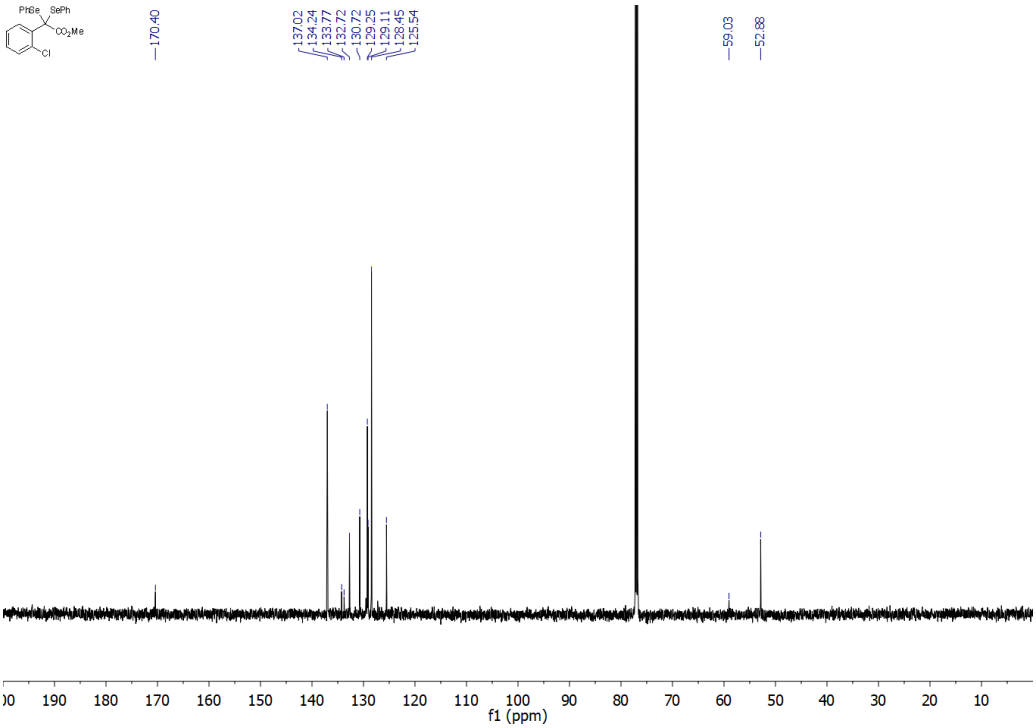


## Methyl 2-(2-chlorophenyl)-2,2-bis(phenylselanyl)acetate (5n)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

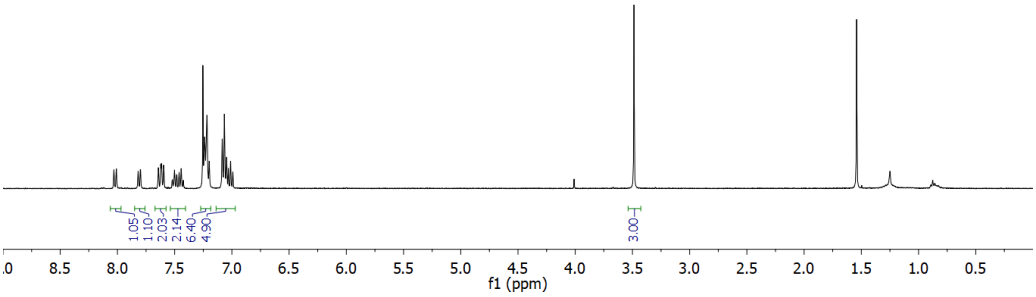



$^{13}$C NMR (151 MHz, Chloroform-*d*):



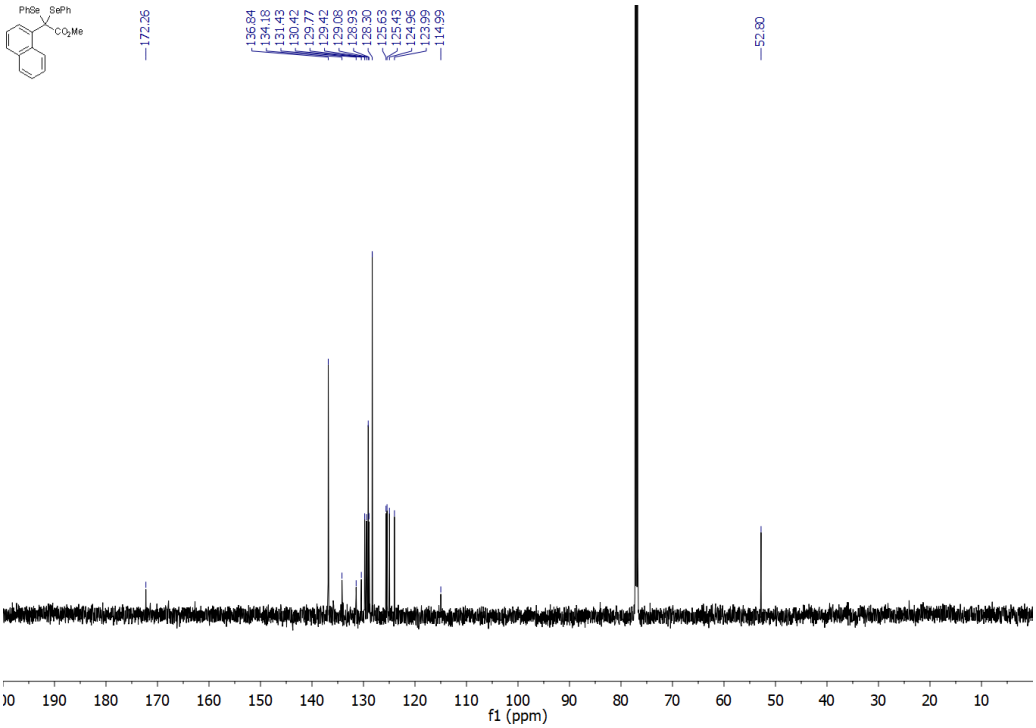
## Methyl 2-(naphthalen-1-yl)-2,2-bis(phenylselanyl)acetate (5o)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

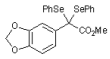

$^{13}$C NMR (151 MHz, Chloroform-*d*):

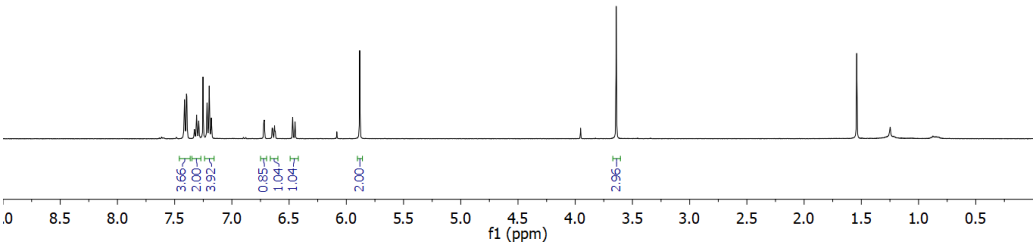


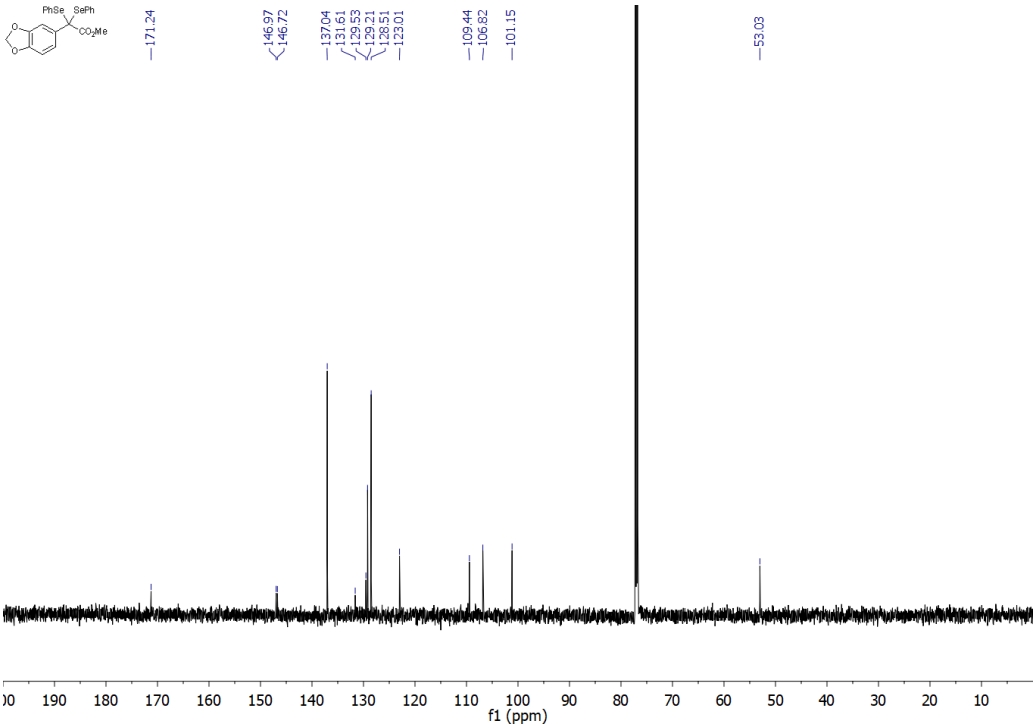
## Methyl 2-(benzo[d][1,3]dioxol-5-yl)-2,2-bis(phenylselanyl)acetate (5p)

$^{1}$H NMR (400 MHz, Chloroform-*d*):



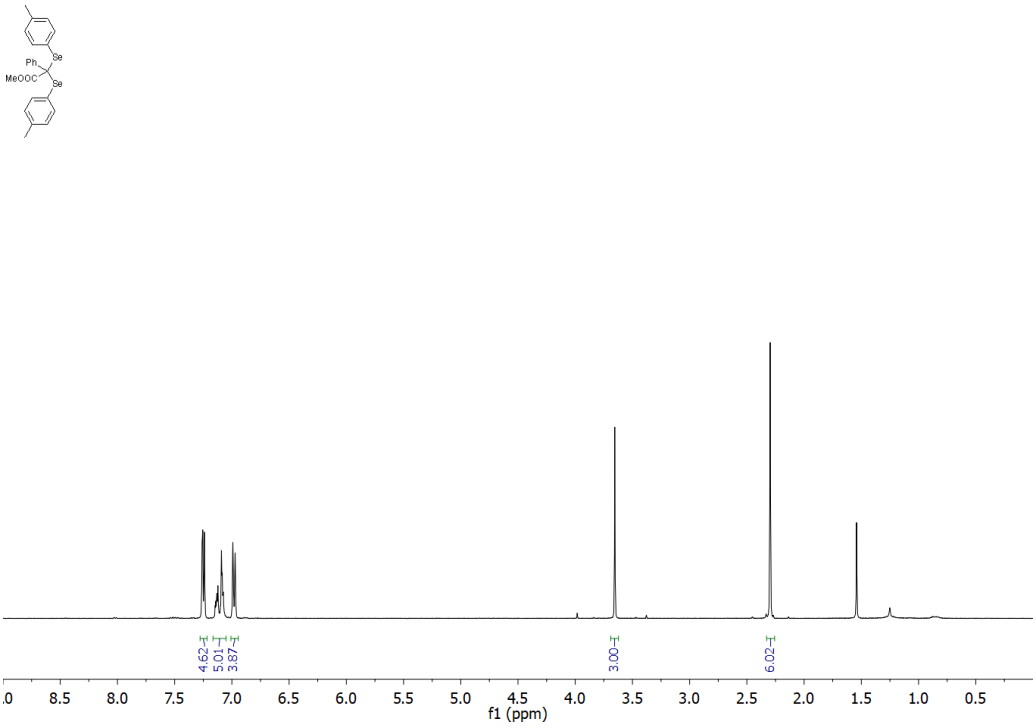




$^{13}$C NMR (151 MHz, Chloroform-*d*):

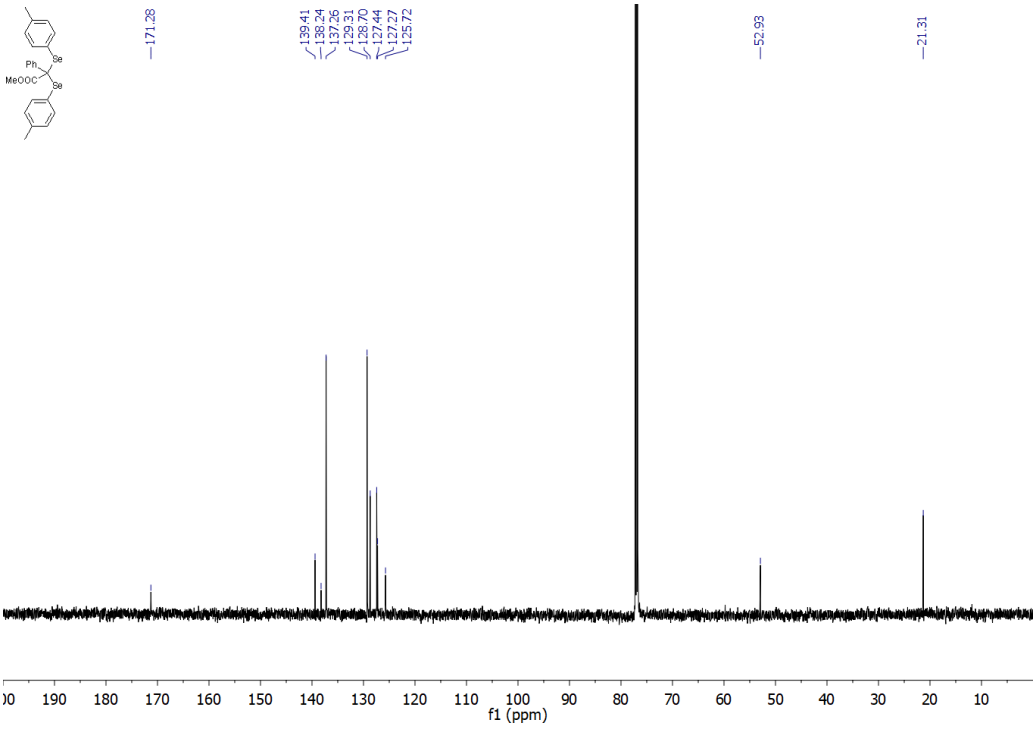


## Methyl 2-phenyl-2,2-bis(p-tolylselanyl)acetate (5q)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

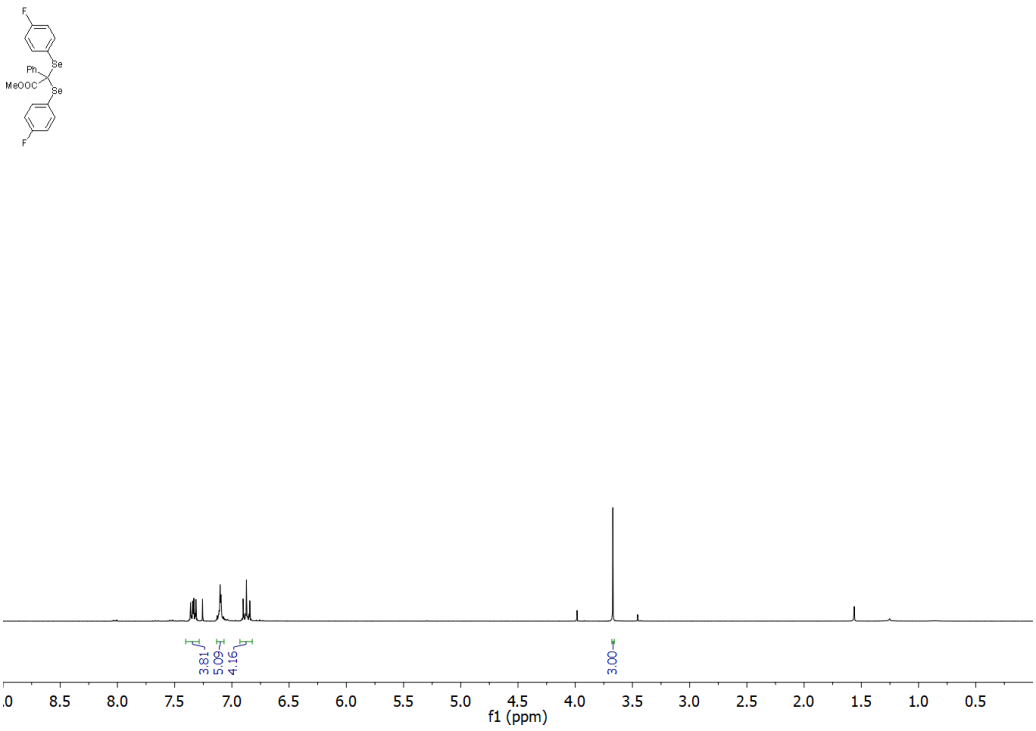



$^{13}$C NMR (151 MHz, Chloroform-*d*):

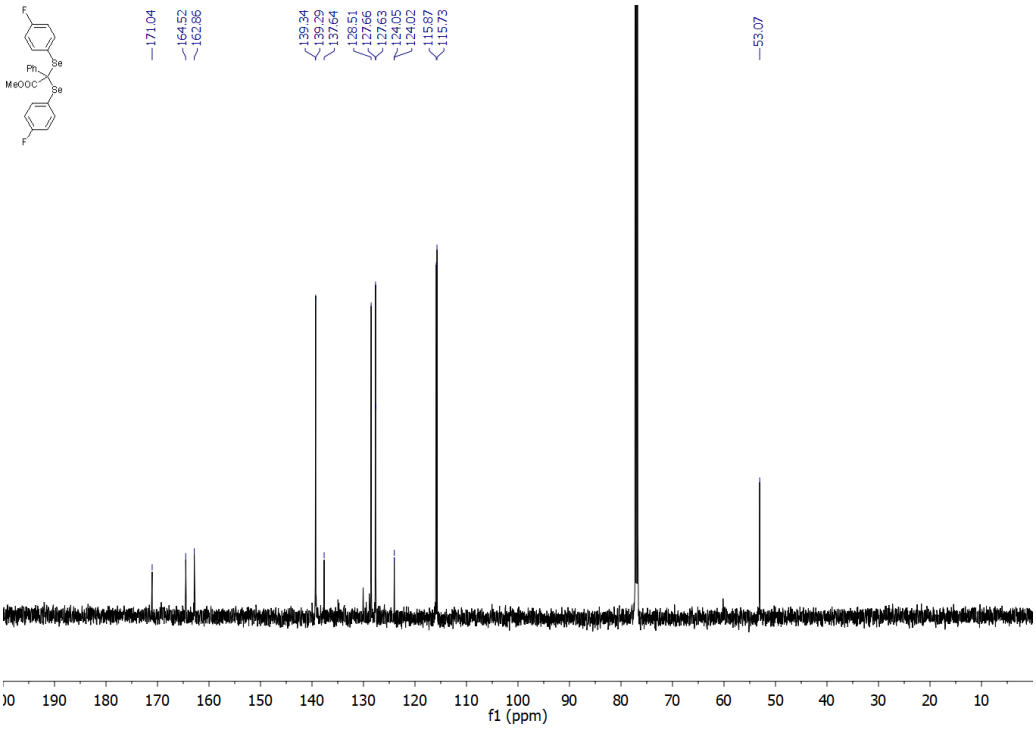


## Methyl 2,2-bis((4-fluorophenyl)selanyl)-2-phenylacetate (5r)

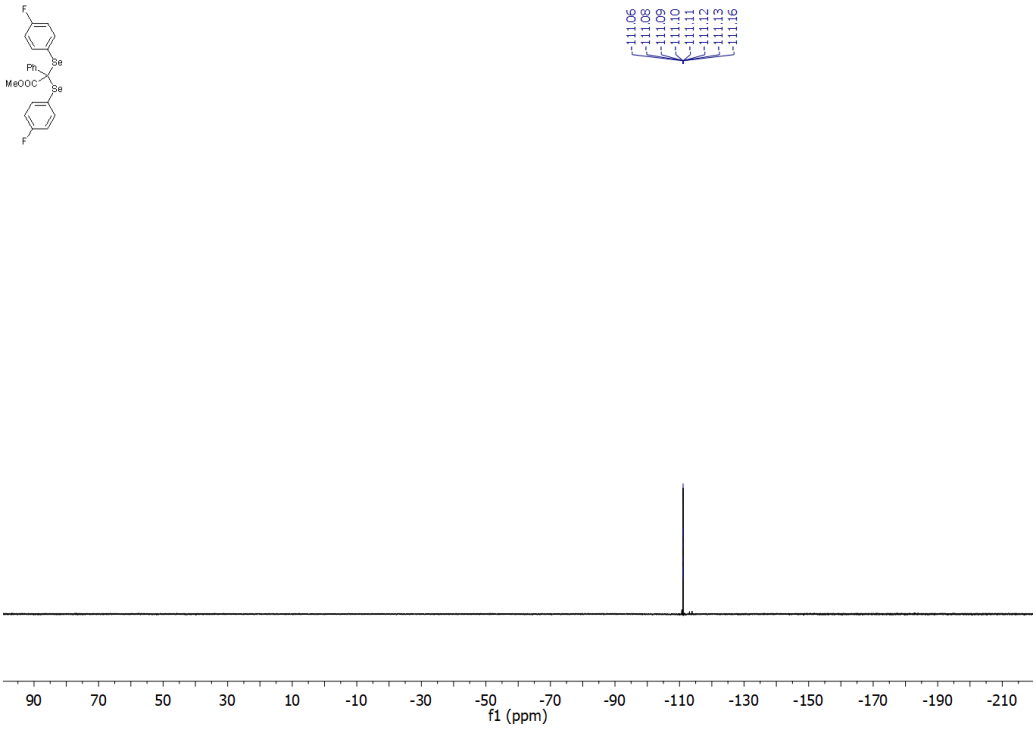
$^{1}$H NMR (300 MHz, Chloroform-*d*):




$^{13}$C NMR (151 MHz, Chloroform-*d*):

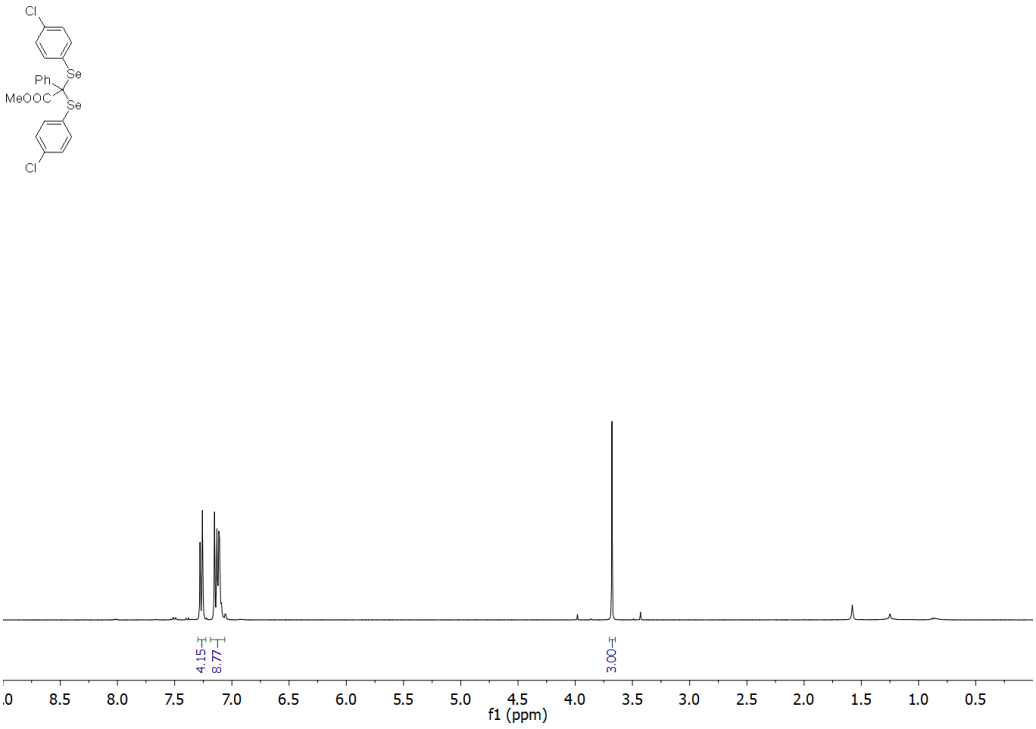


$^{19}$F NMR (564 MHz, Chloroform-*d*):

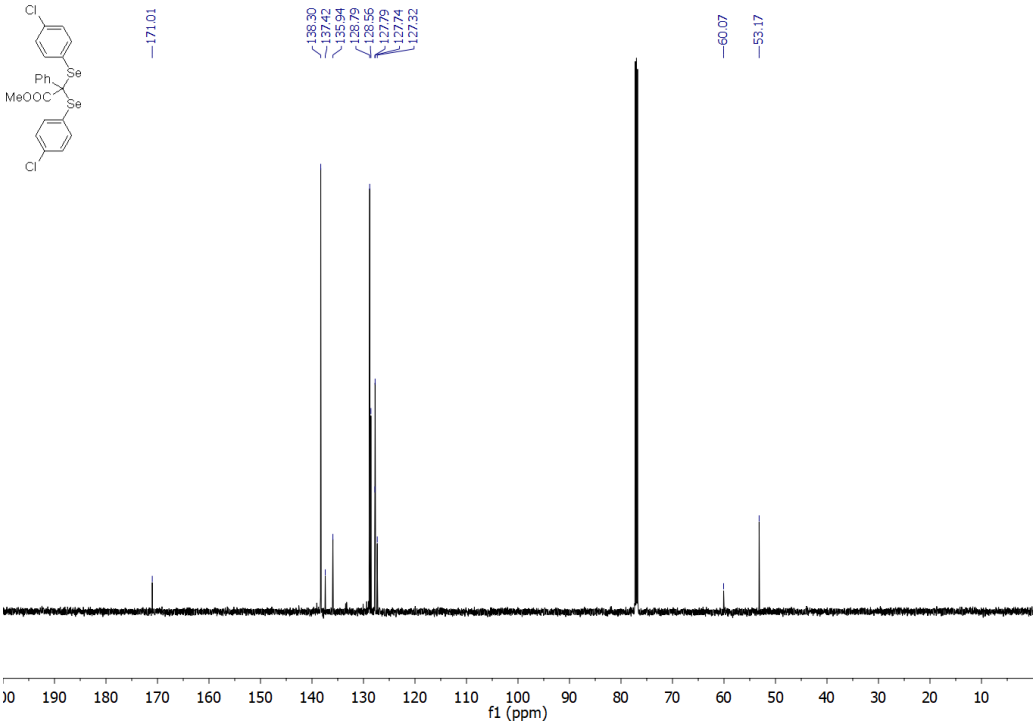


## Methyl 2,2-bis((4-chlorophenyl)selanyl)-2-phenylacetate (5s)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

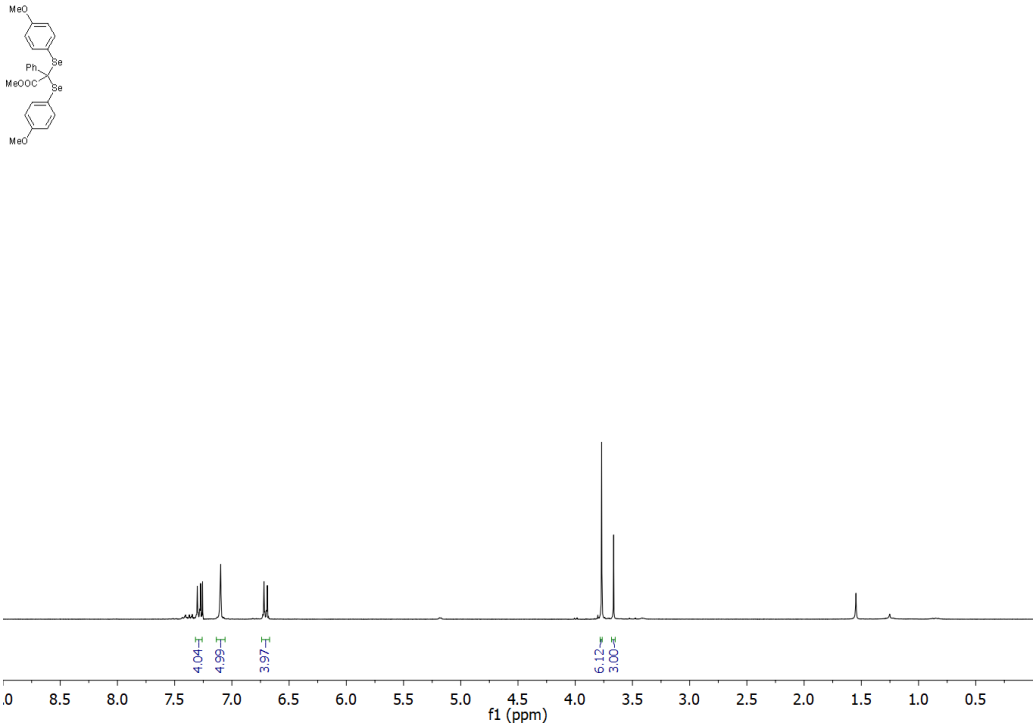



$^{13}$C NMR (151 MHz, Chloroform-*d*):

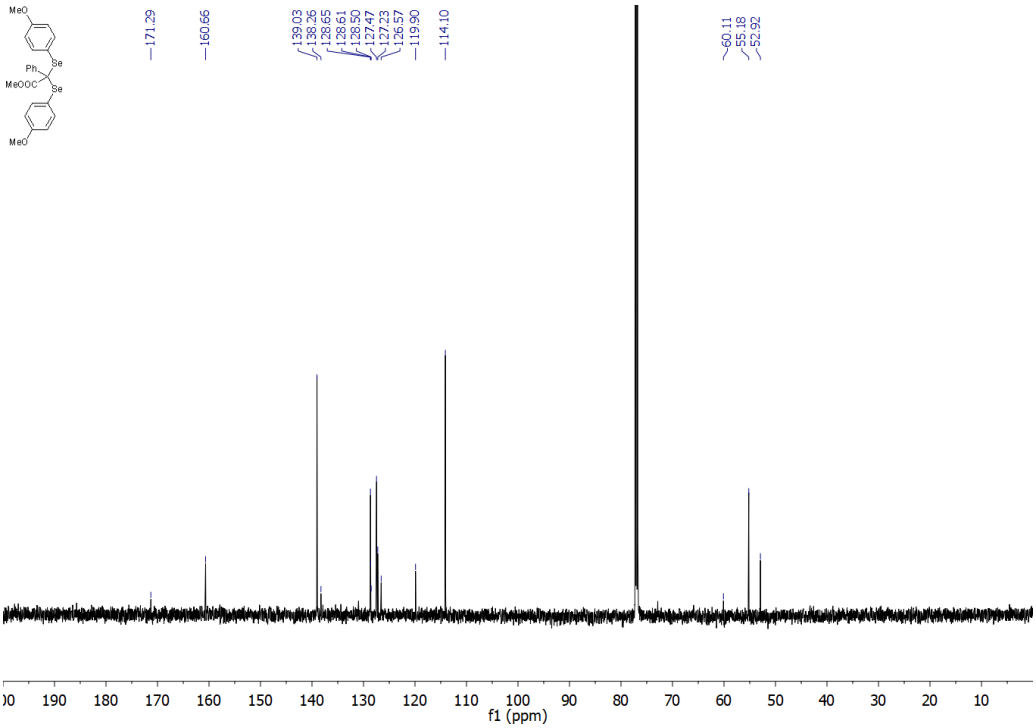


## Methyl 2,2-bis((4-methoxyphenyl)selanyl)-2-phenylacetate (5t)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

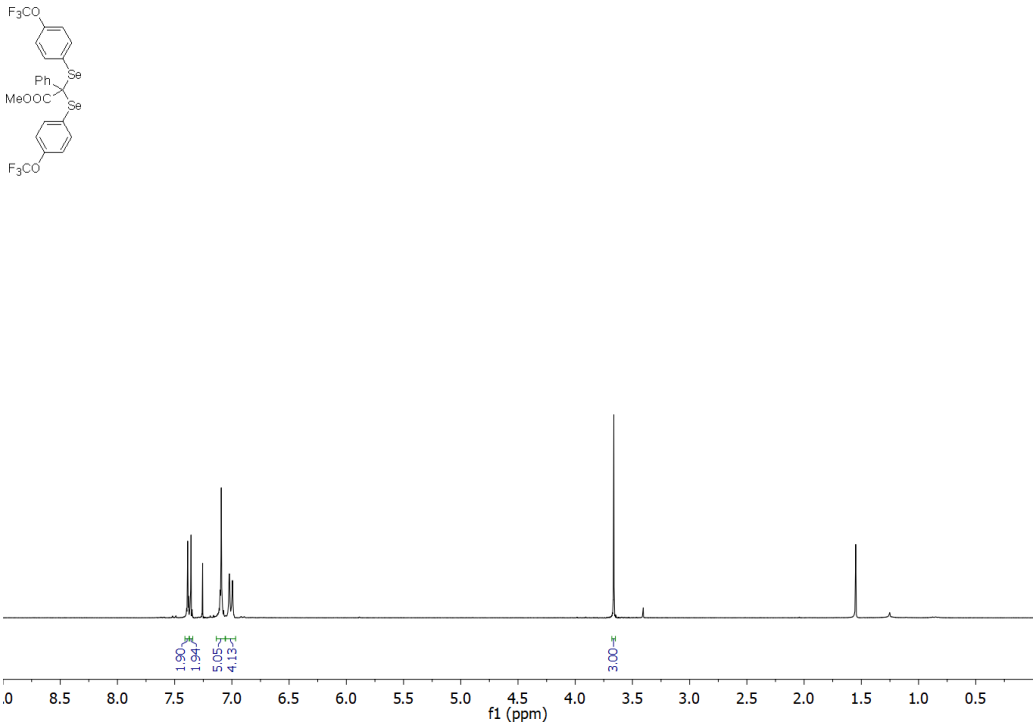



$^{13}$C NMR (151 MHz, Chloroform-*d*):

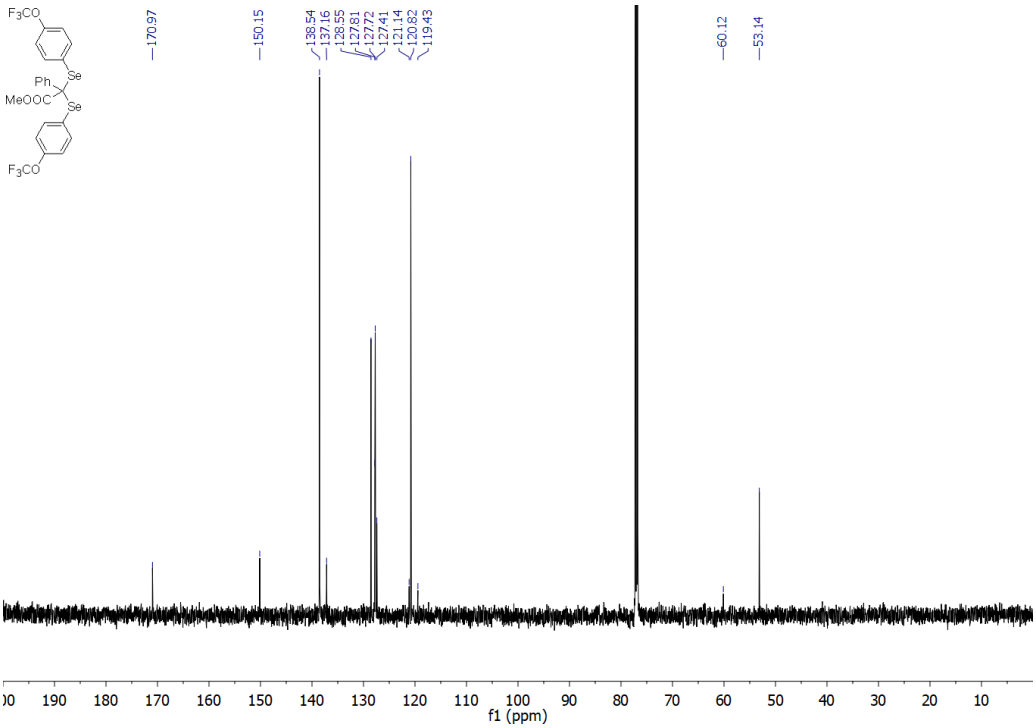


**Methyl 2-phenyl-2,2-bis((4-(trifluoromethoxy)phenyl)selanyl)acetate (5u)**

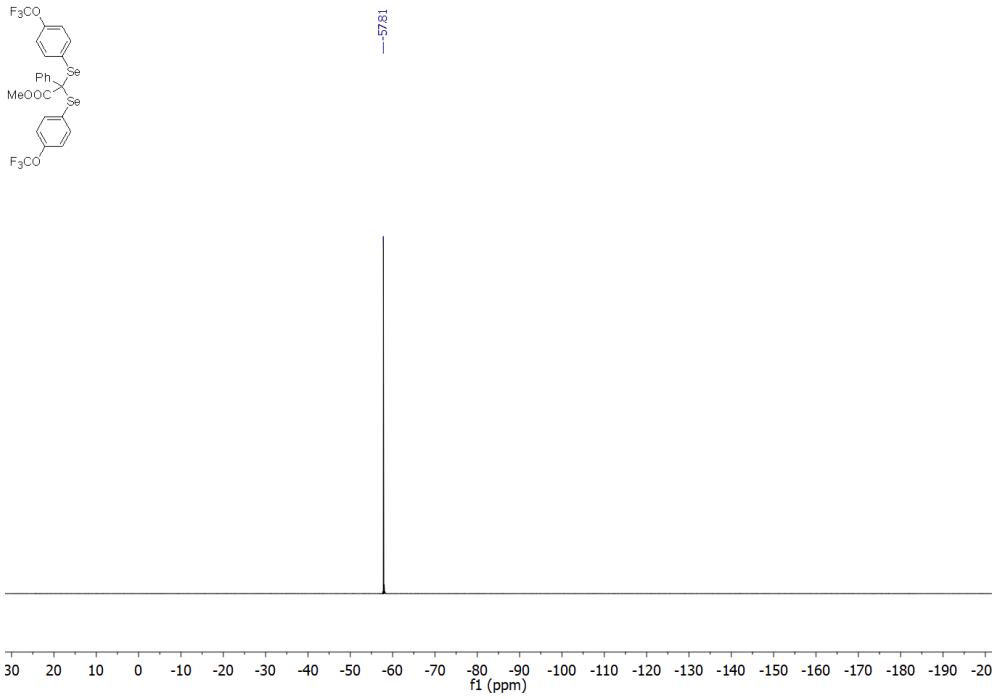
$^{1}$H NMR (300 MHz, Chloroform-*d*):




$^{13}$C NMR (151 MHz, Chloroform-*d*):

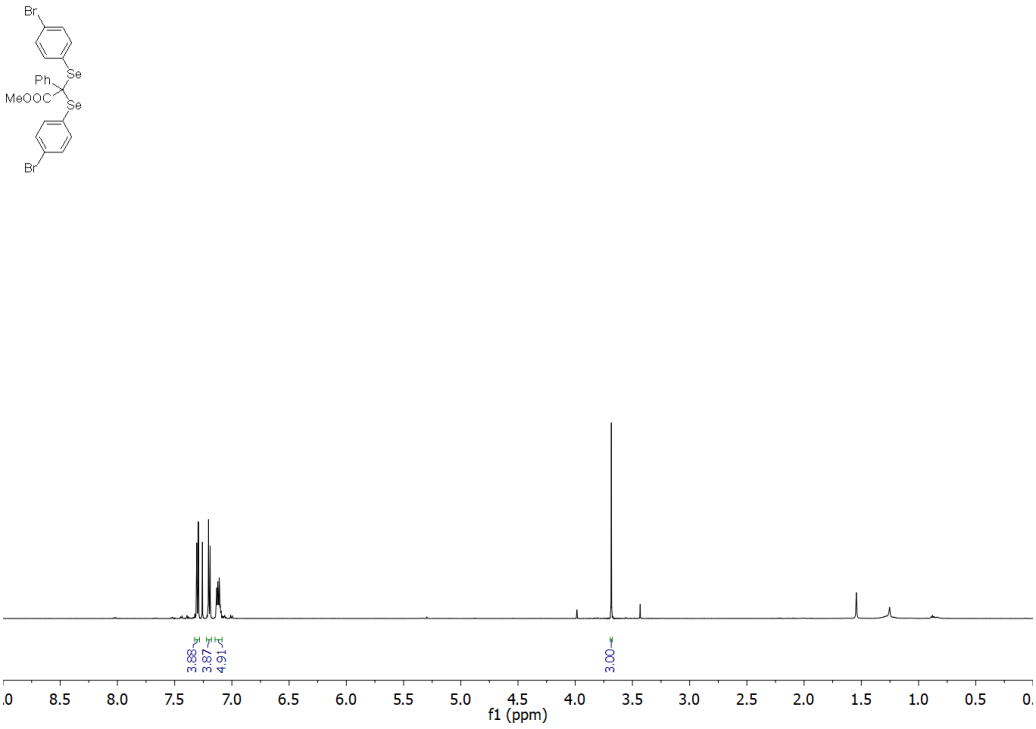


$^{19}$F NMR (564 MHz, Chloroform-*d*):

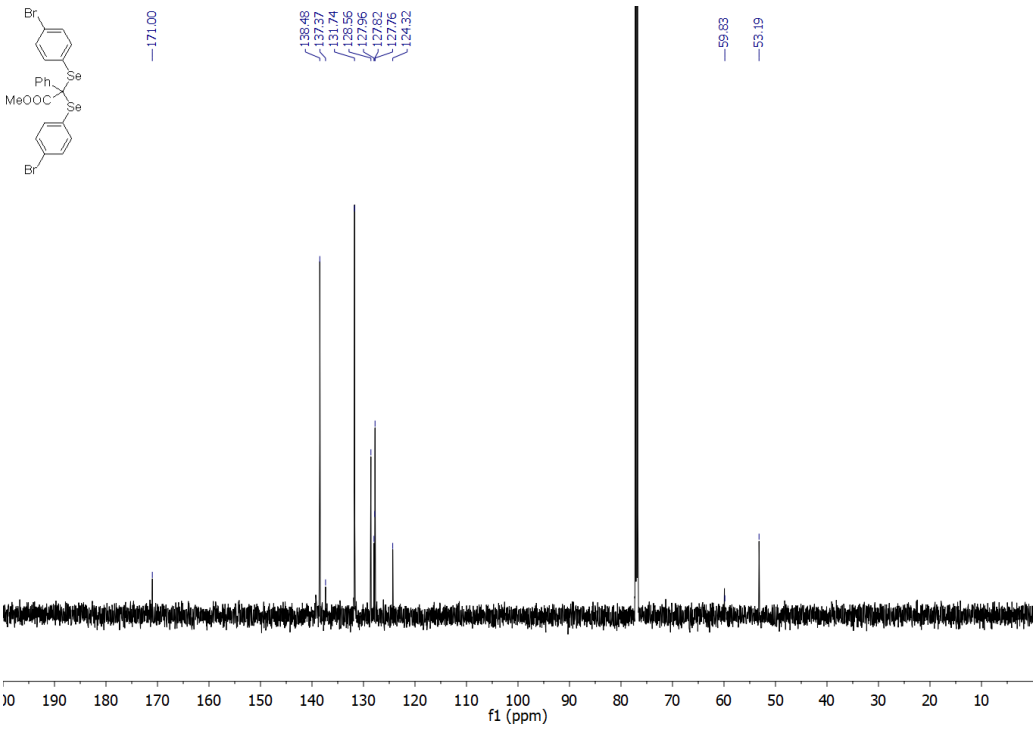


## Methyl 2,2-bis((4-bromophenyl)selanyl)-2-phenylacetate (5v)

$^{1}$H NMR (600 MHz, Chloroform-*d*):

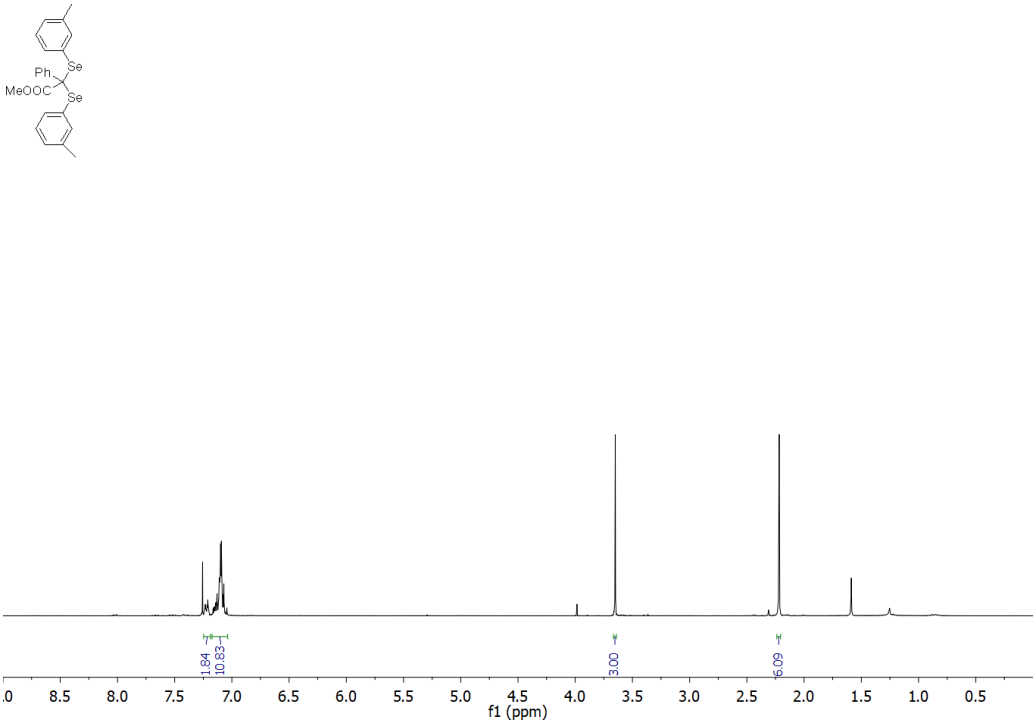



$^{13}$C NMR (151 MHz, Chloroform-*d*):

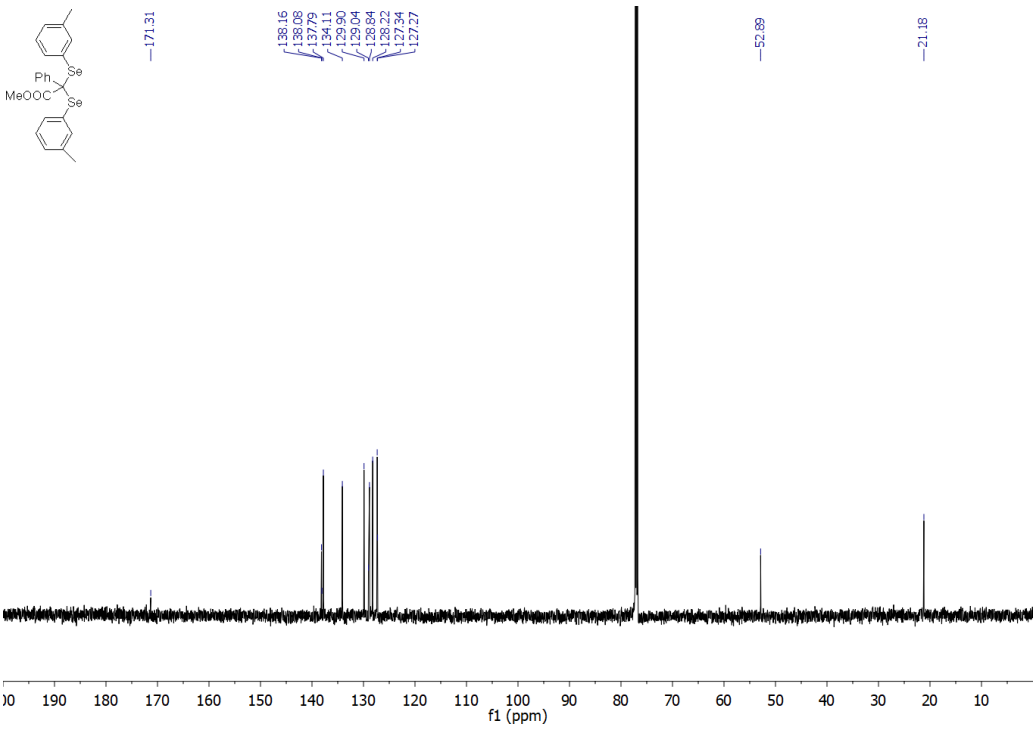


## Methyl 2-phenyl-2,2-bis(m-tolylselanyl)acetate (5w)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

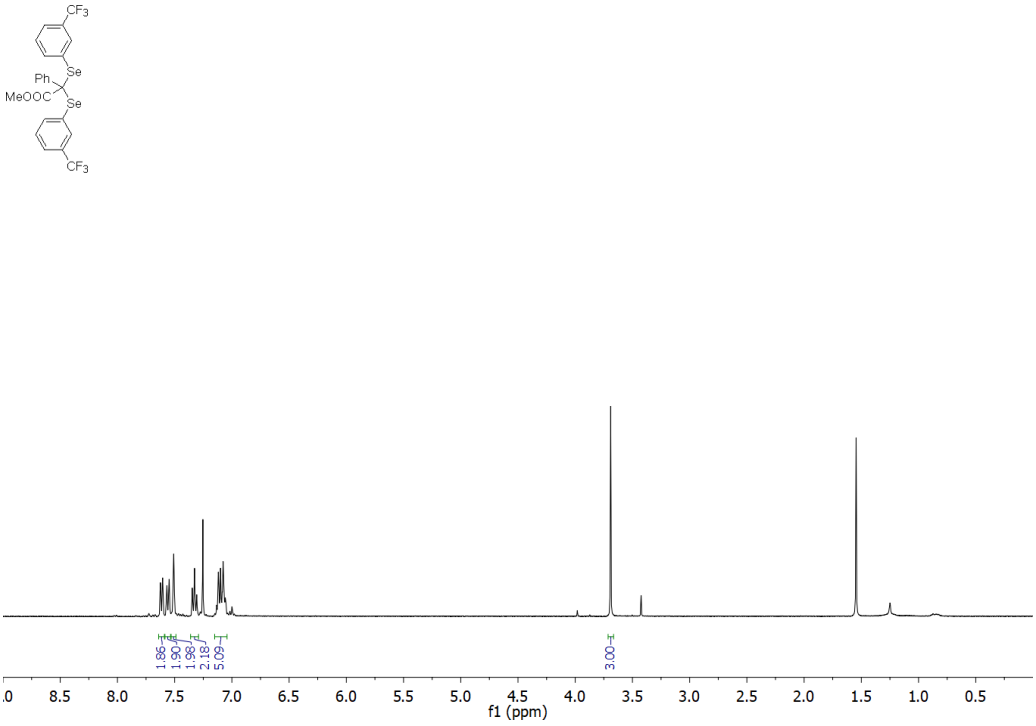



$^{13}$C NMR (151 MHz, Chloroform-*d*):

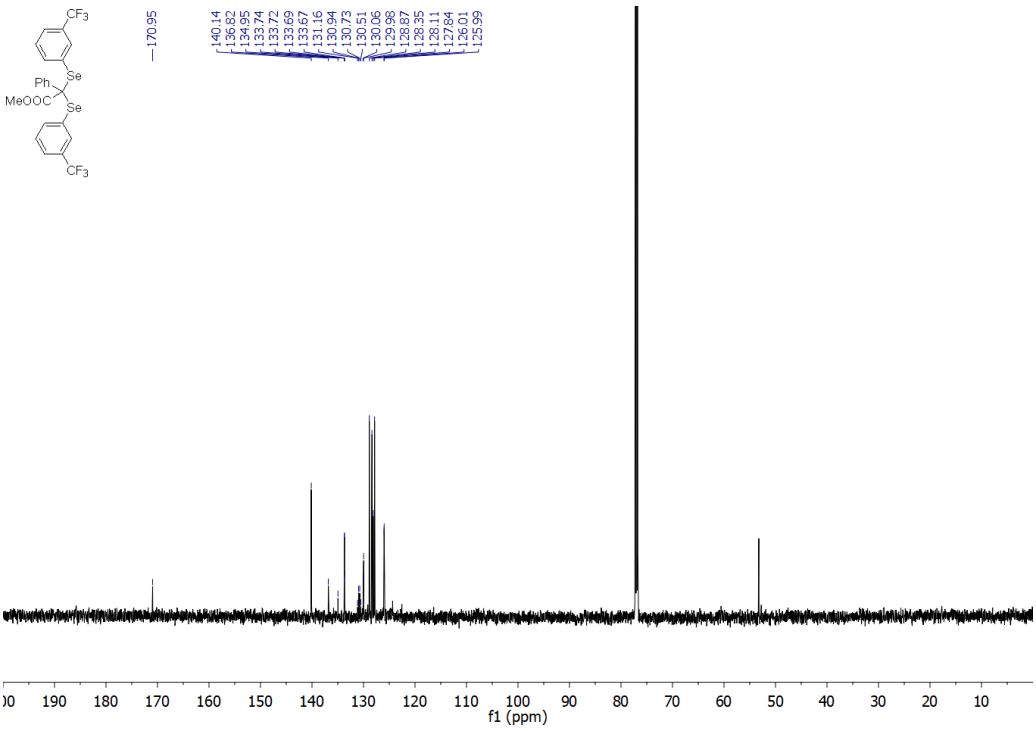


## Methyl 2-phenyl-2,2-bis((3-(trifluoromethyl)phenyl)selanyl)acetate (5x)

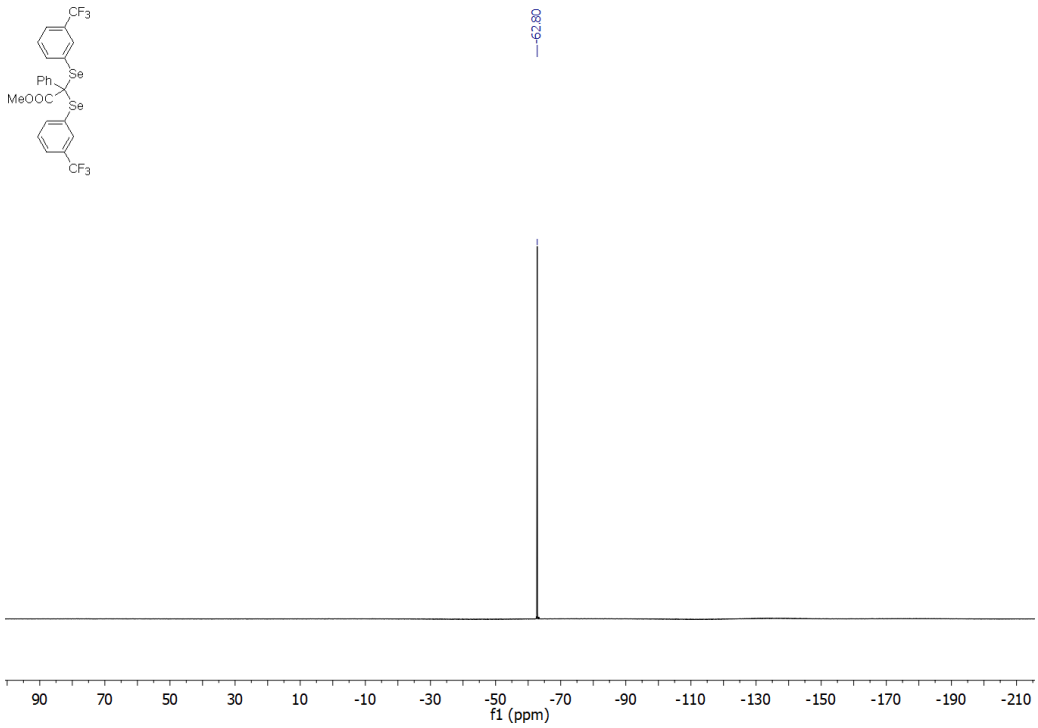
$^{1}$H NMR (400 MHz, Chloroform-*d*):




**$^{13}$C NMR** (151 MHz, Chloroform-*d*):

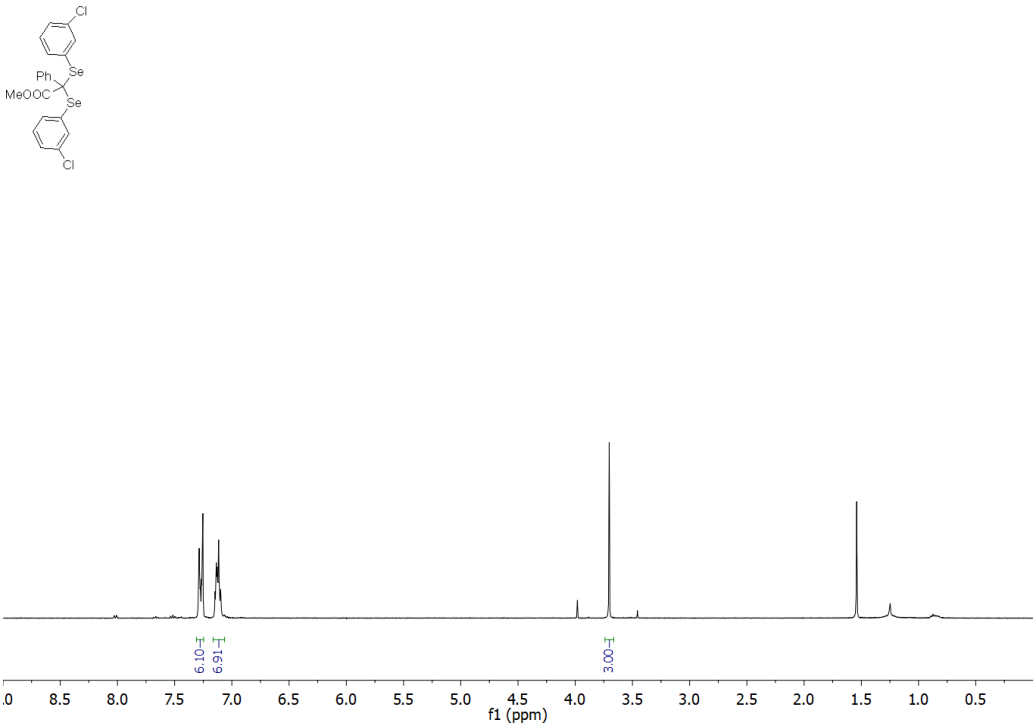


$^{19}$F NMR (564 MHz, Chloroform-*d*):

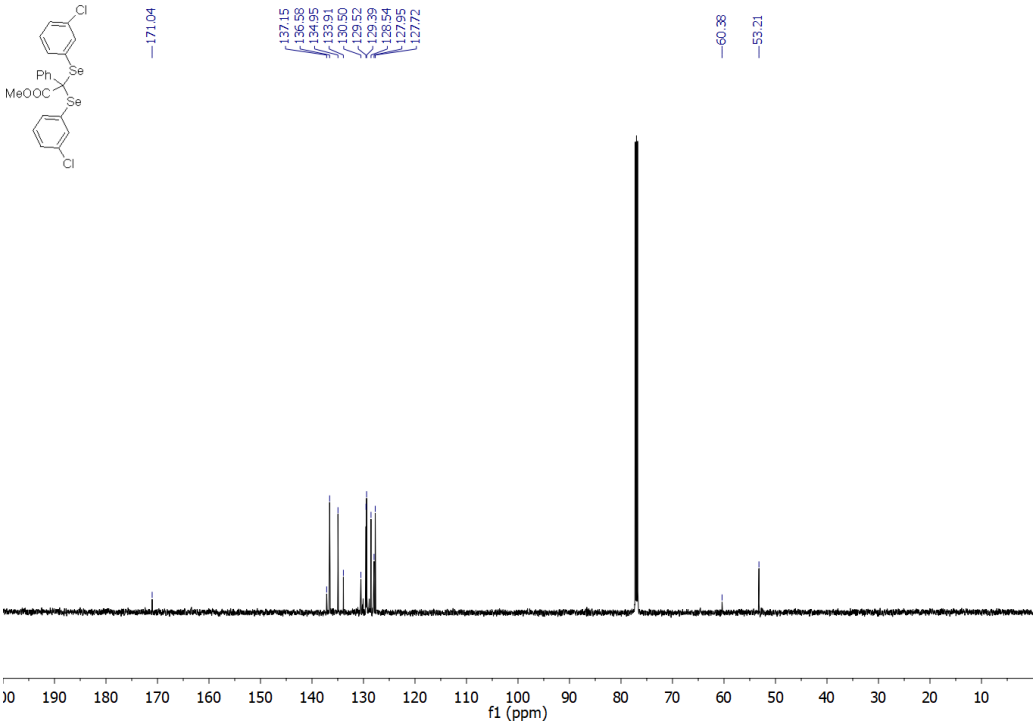


## Methyl 2,2-bis((3-chlorophenyl)selanyl)-2-phenylacetate (5y)

$^{1}$H NMR (600 MHz, Chloroform-*d*):

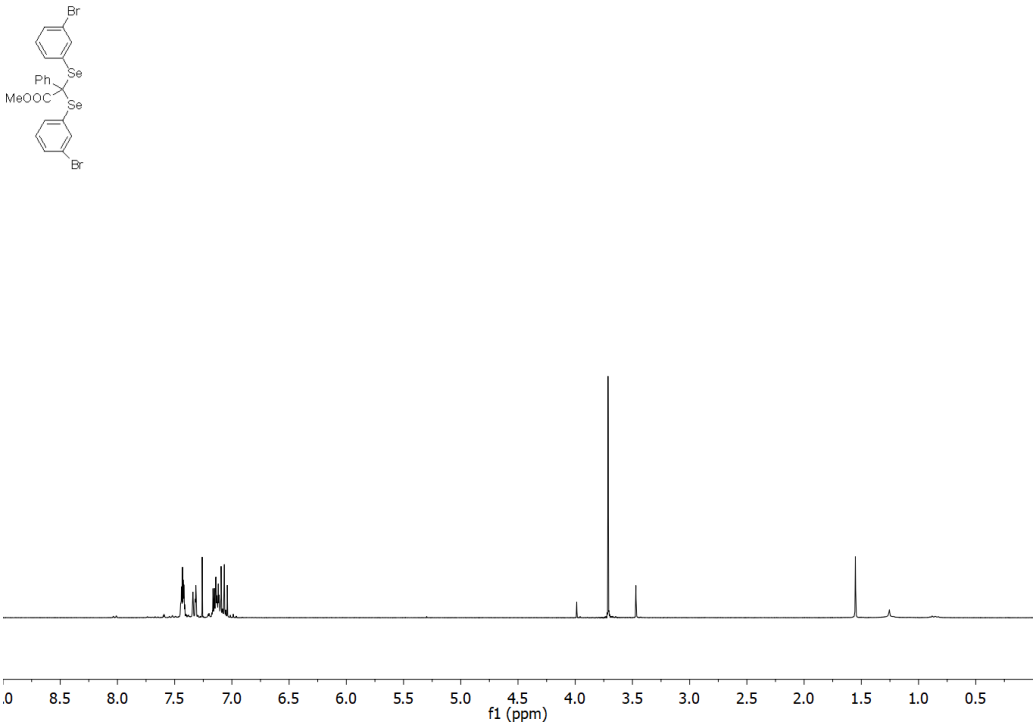



$^{13}$C NMR (151 MHz, Chloroform-*d*):

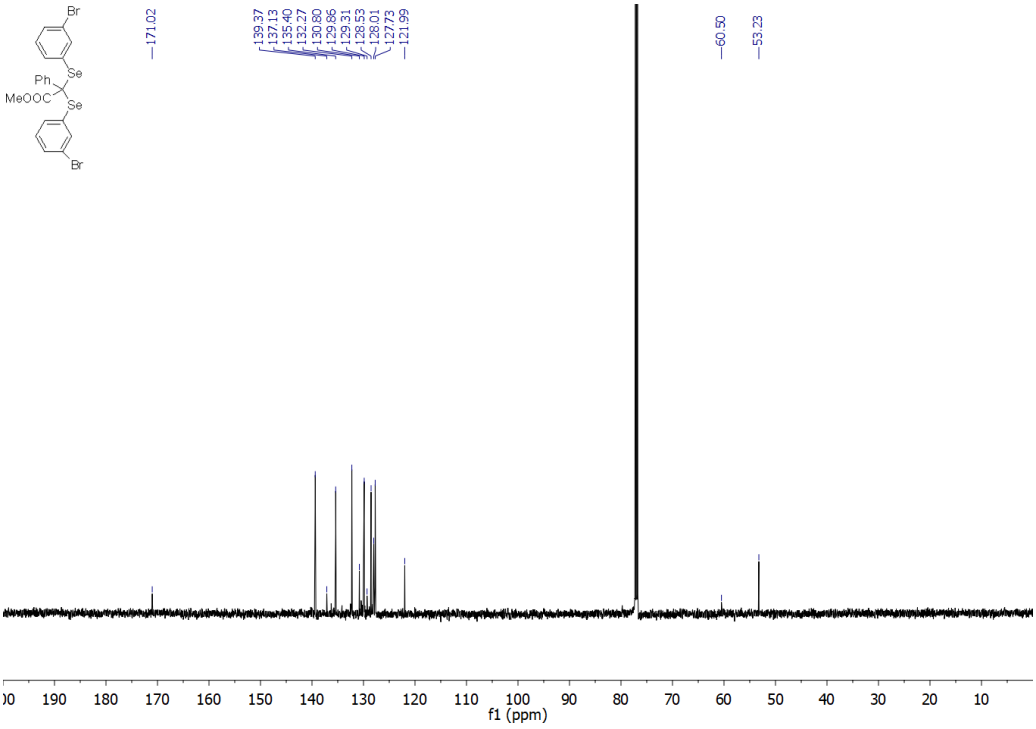


## Methyl 2,2-bis((3-bromophenyl)selanyl)-2-phenylacetate (5z)

$^{1}$H NMR (600 MHz, Chloroform-*d*):

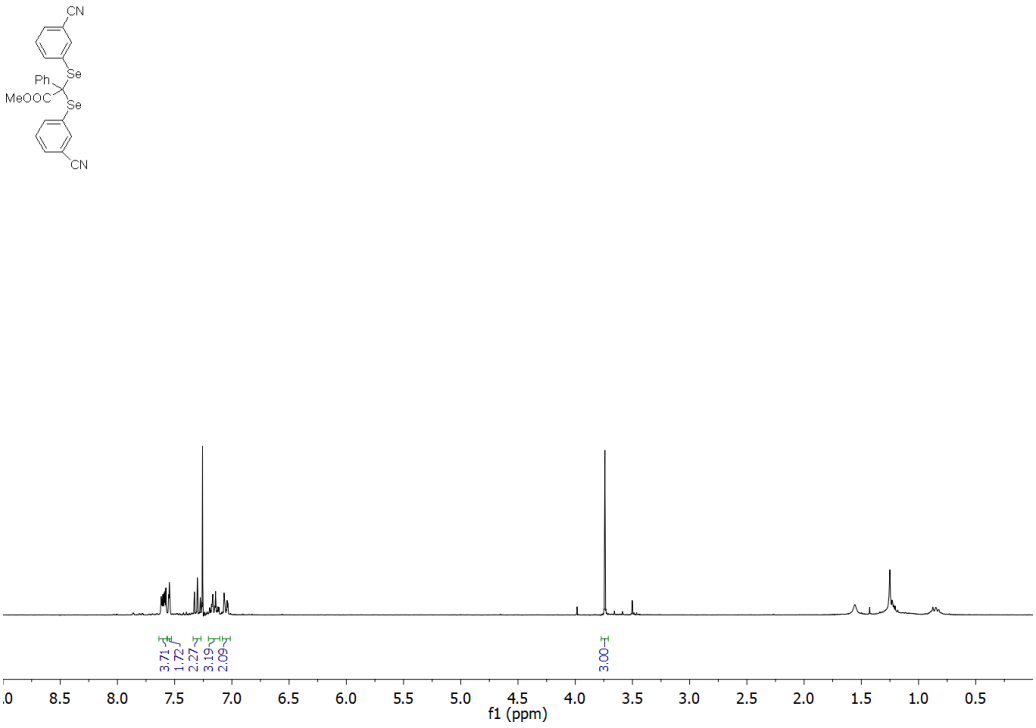



$^{13}$C NMR (151 MHz, Chloroform-*d*):

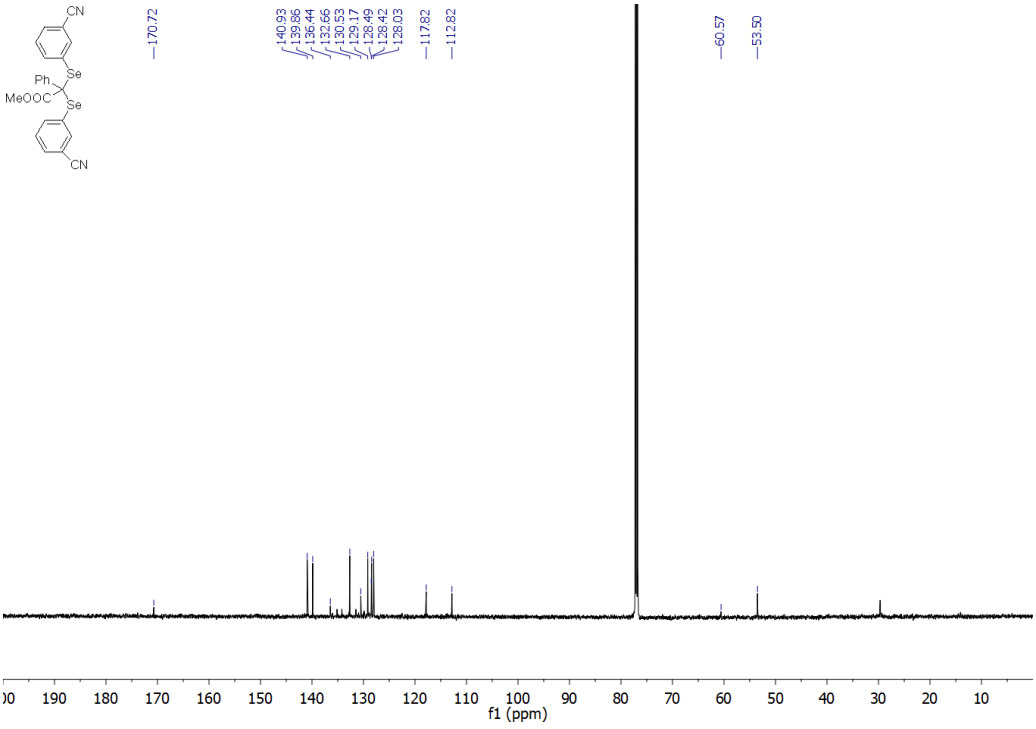


## Methyl 2,2-bis((3-cyanophenyl)selanyl)-2-phenylacetate (5aa)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

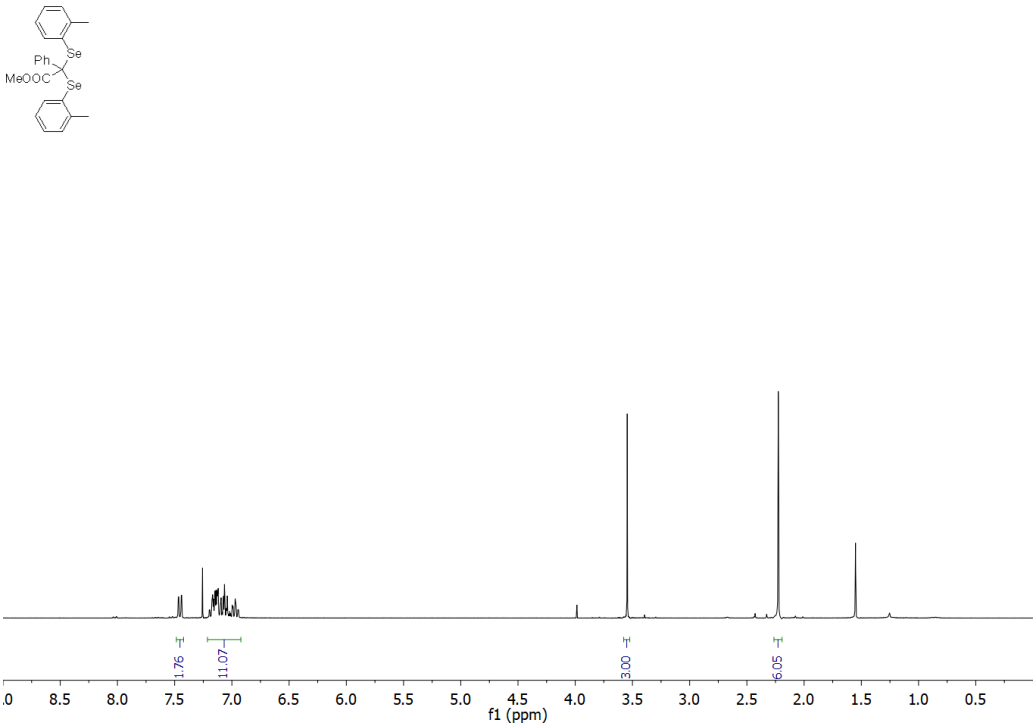



$^{13}$C NMR (151 MHz, Chloroform-*d*):

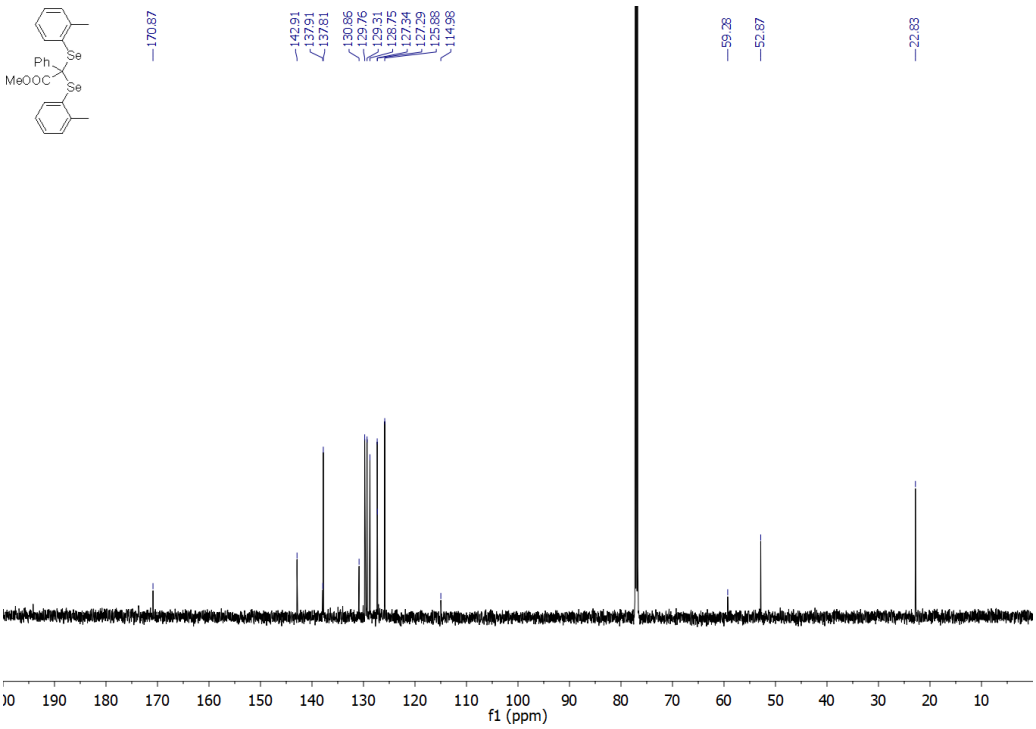


## Methyl 2-phenyl-2,2-bis(o-tolylselanyl)acetate (5ab)

$^{1}$H NMR (300 MHz, Chloroform-*d*):

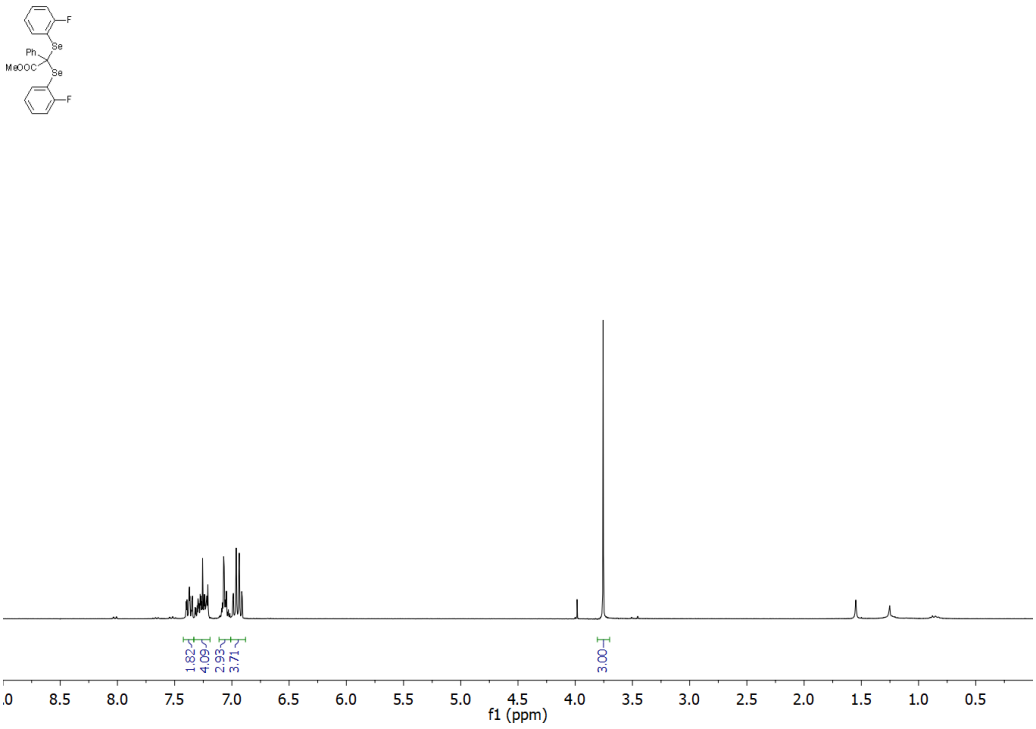



$^{13}$C NMR (151 MHz, Chloroform-*d*):

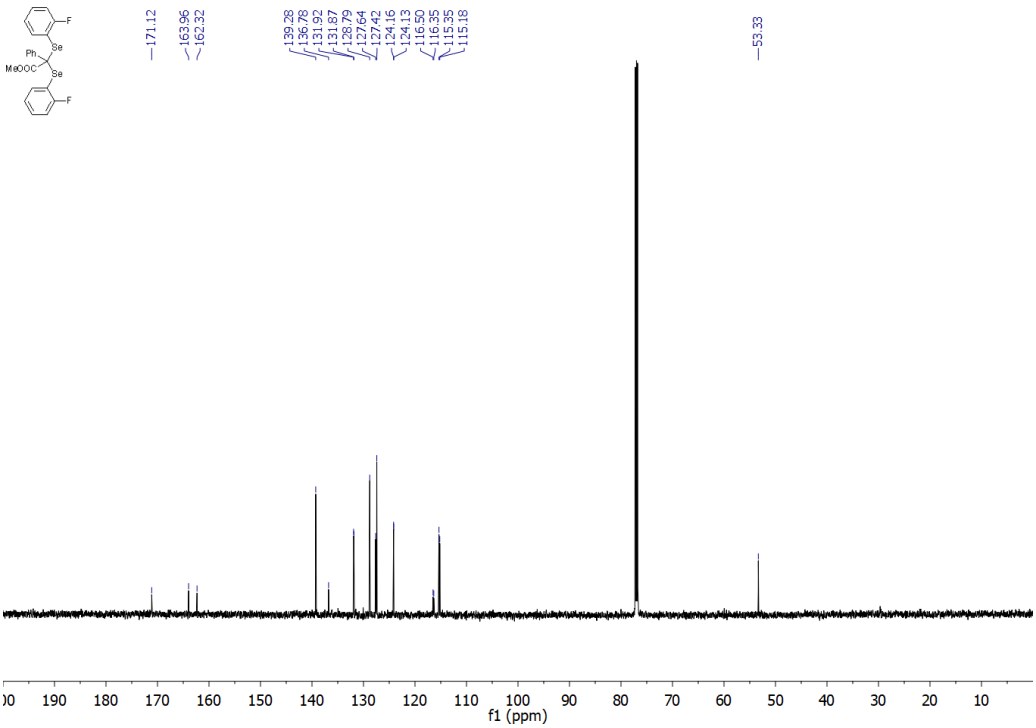


## Methyl 2,2-bis((2-fluorophenyl)selanyl)-2-phenylacetate (5ac)

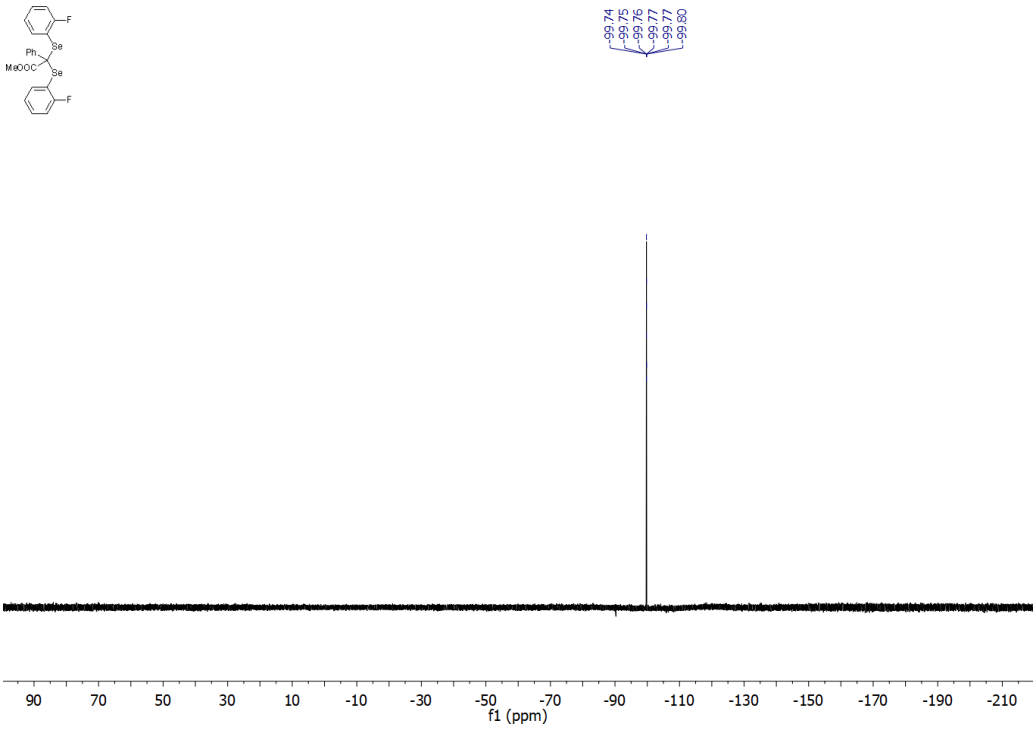
$^{1}$H NMR (300 MHz, Chloroform-*d*):




$^{13}$C NMR (151 MHz, Chloroform-*d*):

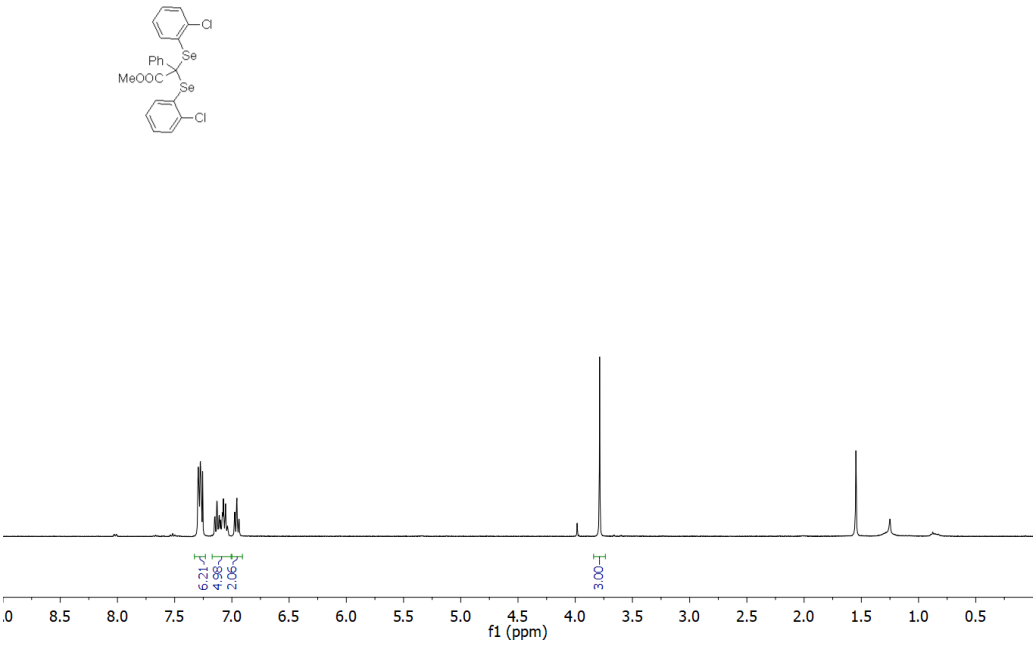


$^{19}$F NMR (282 MHz, Chloroform-*d*):

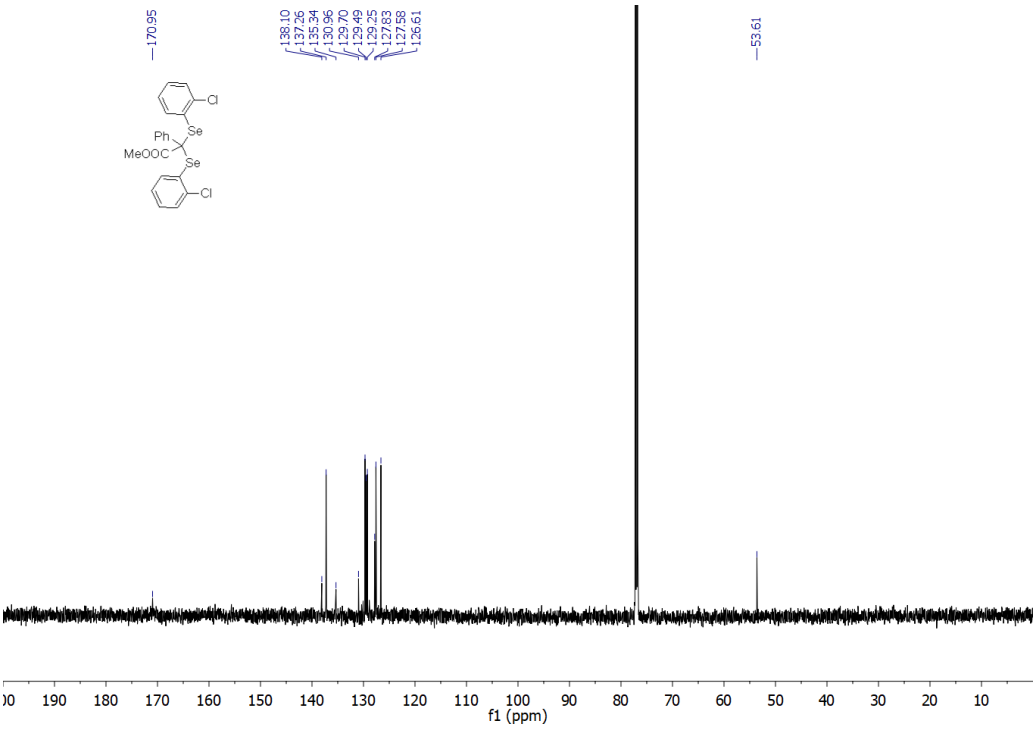


## Methyl 2,2-bis((2-chlorophenyl)selanyl)-2-phenylacetate (5ad)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

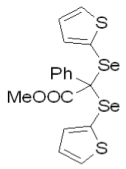


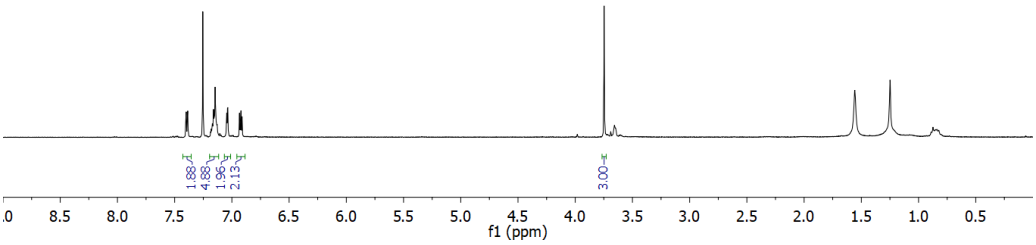

$^{13}$C NMR (151 MHz, Chloroform-*d*):



## Methyl 2-phenyl-2,2-bis(thiophen-2-ylselanyl)acetate (5ae)

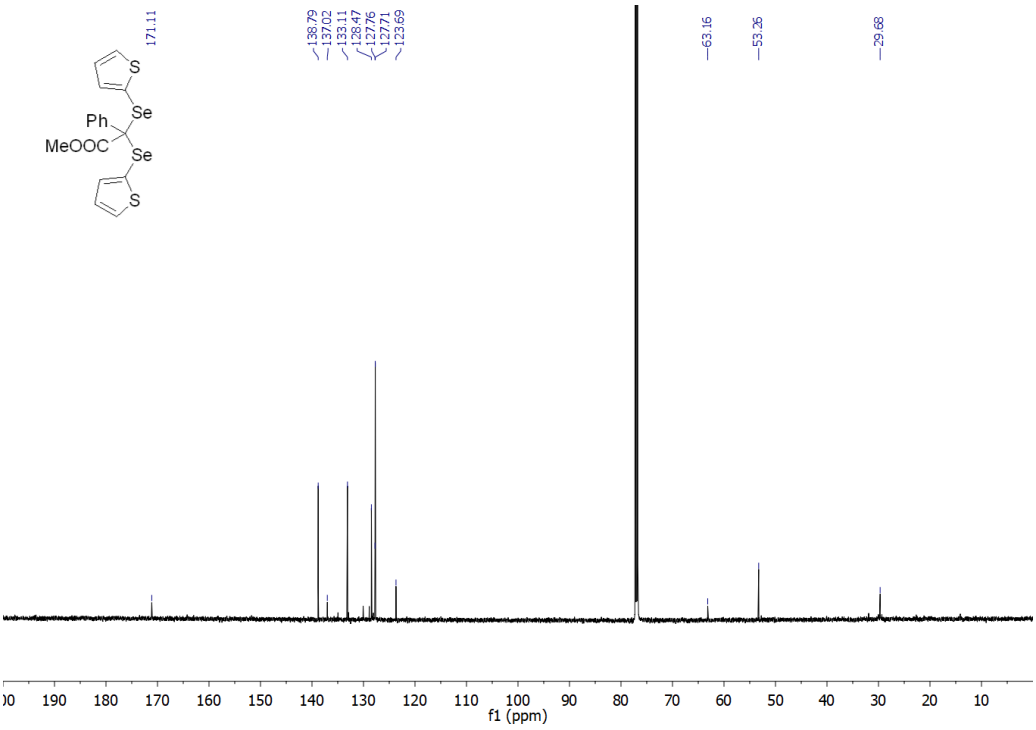
$^{1}$H NMR (400 MHz, Chloroform-*d*):



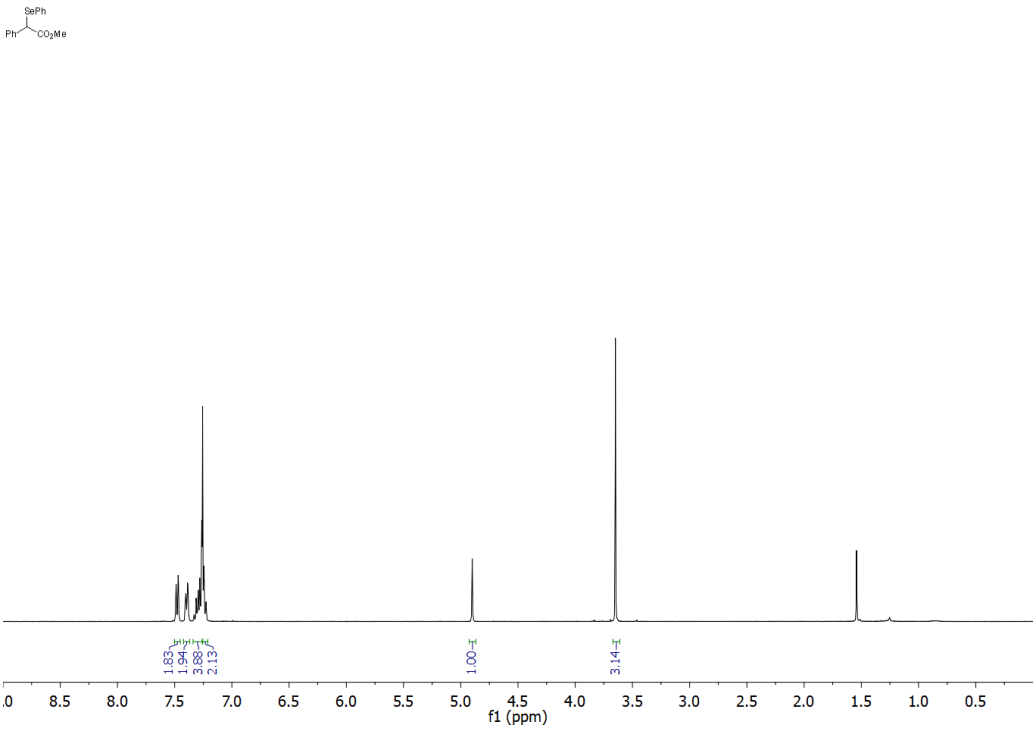

$^{13}$C NMR (151 MHz, Chloroform-*d*):



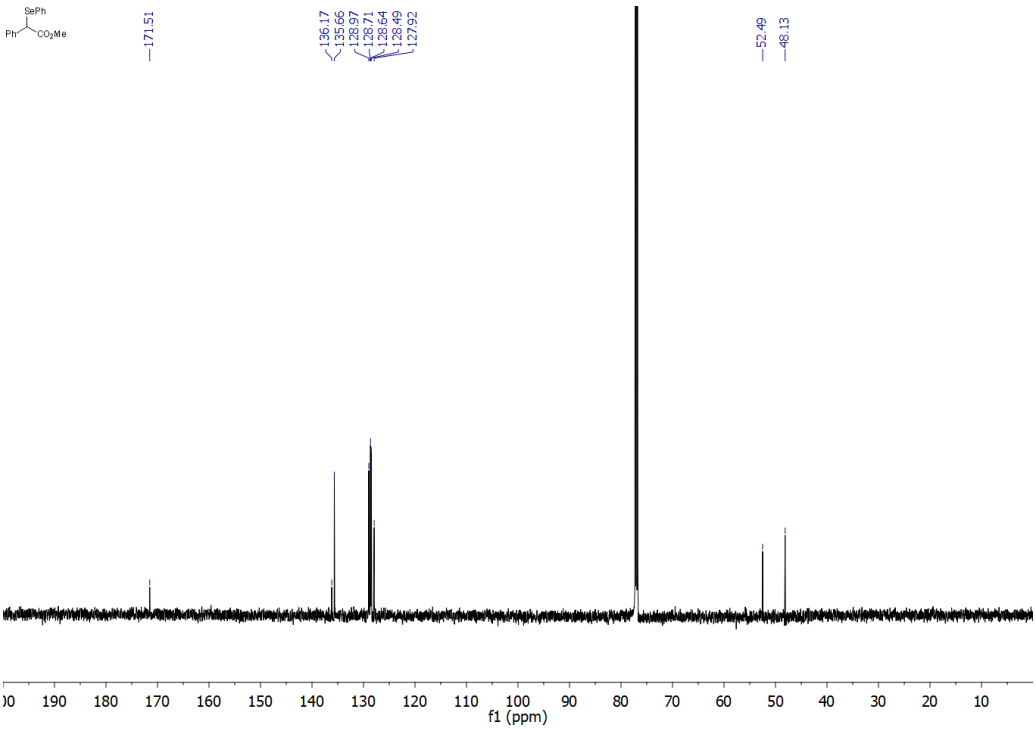



## Methyl 2-phenyl-2-(phenylselanyl)acetate (9)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

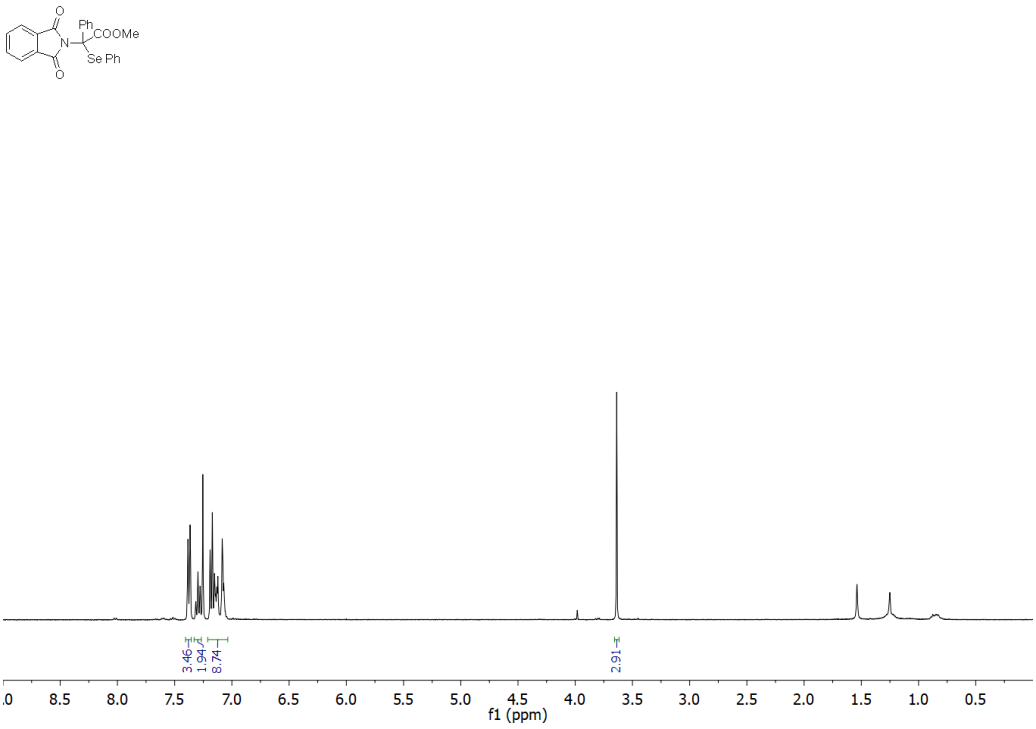



$^{13}$C NMR (151 MHz, Chloroform-*d*):

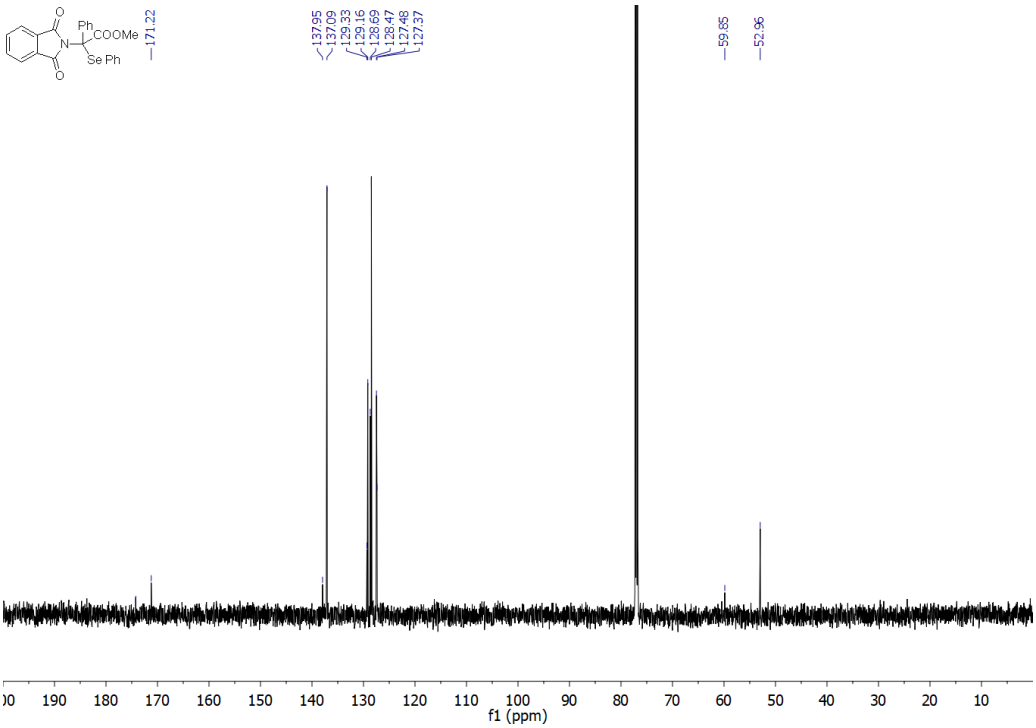


## Methyl 2-(1,3-dioxoisoindolin-2-yl)-2-phenyl-2-(phenylselanyl)acetate (10)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

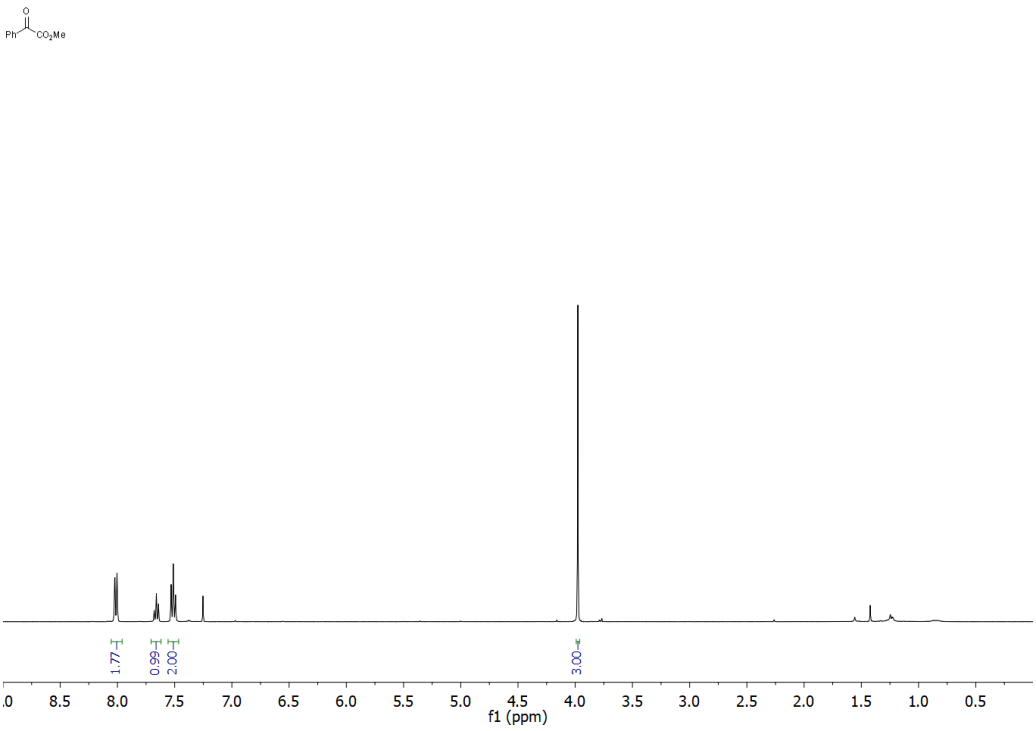



**$^{13}$C NMR** (151 MHz, Chloroform-*d*):

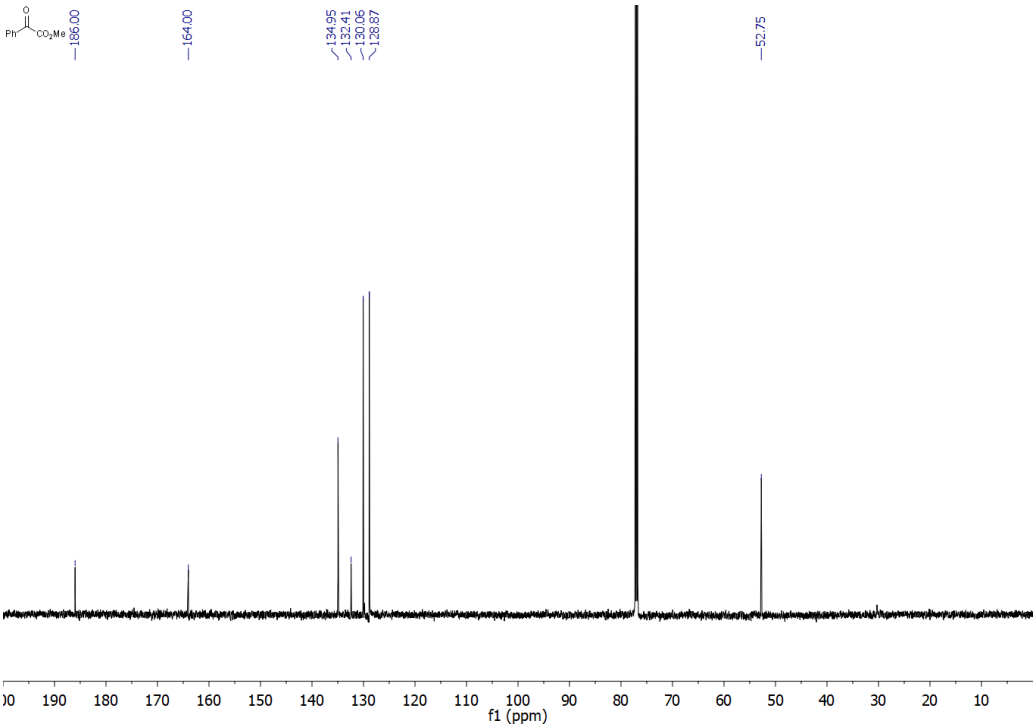


## Methyl 2-oxo-2-phenylacetate (11)

$^{1}$H NMR (400 MHz, Chloroform-*d*):

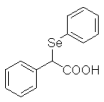


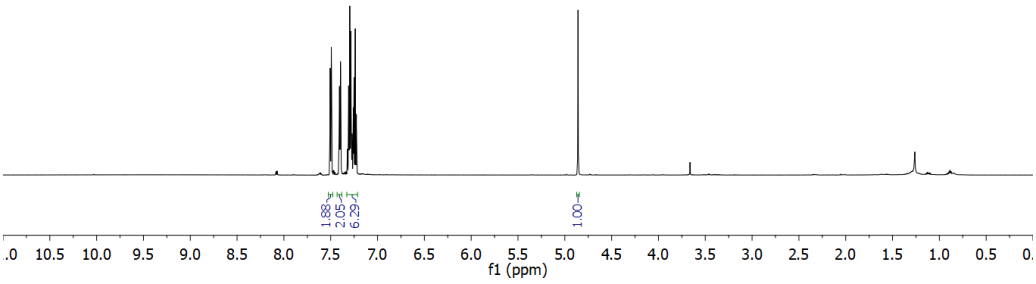

$^{13}$C NMR (151 MHz, Chloroform-*d*):



## 2-phenyl-2-(phenylselanyl)acetic acid (12)

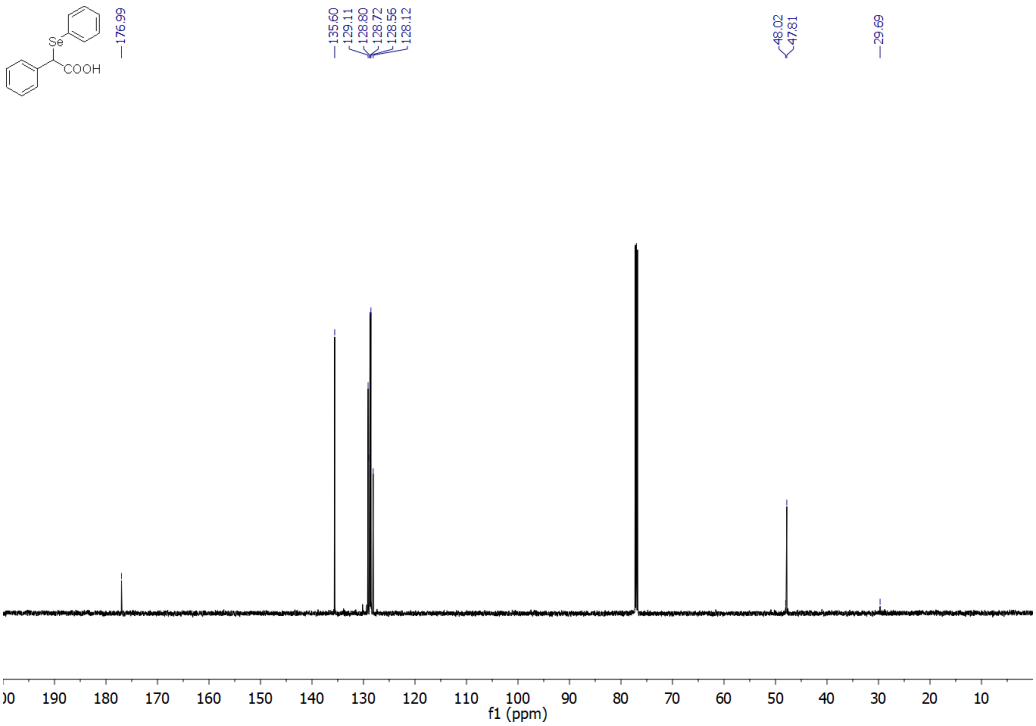
$^{1}$H NMR (400 MHz, Chloroform-*d*):



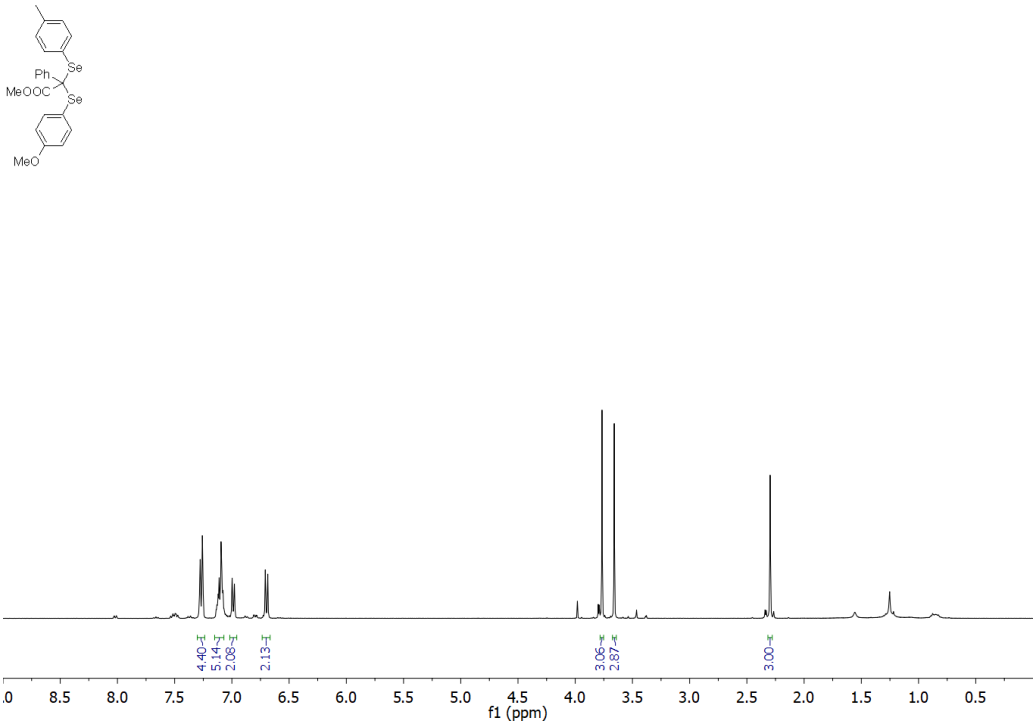

$^{13}$C NMR (151 MHz, Chloroform-*d*):



**Methyl 2-((4-methoxyphenyl)selanyl)-2-phenyl-2-(p-tolylselanyl)acetate (5af)**

$^{1}$H NMR (400 MHz, Chloroform-*d*):




$^{13}$C NMR (101 MHz, Chloroform-*d*):