Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

Synthesis of chiral N^{β}-protected amino diselenides from the corresponding amino alkyl iodides using $NaBH_2Se_3$ as a selenating reagent and their conversion to seleninic acids

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General: All solvents were freshly distilled before use. Amino acids were used as received from Sigma-Aldrich Company. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AMX 400 MHz and 100 MHz respectively, with $CDCl_3$ or DMSO-d_6 as an internal standard. Mass spectra were recorded using high resolution mass spectrometer (HRMS) Q-T of mass spectrometer. All the reactions were monitored using TLCs with precoated silica gel plates purchased from Merck. Column Chromatography was performed with Merck silica gel (100–200) at normal atmospheric pressure. Chiral HPLC analysis of isomers was carried out by Agilent 1100 series having G1311A VWD at λ = 254 nm, Phenominex made Lux, pore size-5μ, Cellusole-1, diameter x length = 250 x 4.60 mm.

Experimental section

General procedure for the synthesis of N^{β}-protected amino diselenides 2, 5

To a solution of sodium borohydride ($NaBH_4$, 10 mmol) in dry THF, black selenium powder (30 mmol) was added at 0 ^{o}C under N_2 atmosphere. The consumption of selenium powder in less than 10 min lead to heterogeneous reddish suspension which indicated the formation of $NaBH_2Se_3$. To the resulting $NaBH_2Se_3$ suspension, a THF solution containing N^{α}-protected amino alkyl iodide **1** or **4** (10 mmol) was added drop wise at 0 ^{o}C and the stirring was continued for another 20 min. After completion of the reaction (as monitored by TLC), the reaction mixture was filtered through celite, washed with THF and the solvent was removed under reduced pressure. The crude mass was diluted with EtOAc, washed with water (2 x 20 mL), brine (2 x 20 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude residue **2** or **5** was purified by column chromatography on silica gel with EtOAc/hexane (2:8).

General procedure for the synthesis of N^{β}-protected amino seleninic acids 6

To a solution of N^{β}-protected amino diselenide **2** (10 mmol) in DCM at 0 ^{o}C, 35% aqueous H_2O_2 (64 mmol) was added drop wise under vigorous stirring till the completion of reaction (monitored by TLC). The change in color from yellow to colorless indicated the complete consumption of diselenide. After 30 min, the desired product seleninic acid **6** was formed as white precipitate, which was filtered off and recrystalized using ethanol as a solvent. All seleninic acids were isolated as stable solids.

Characterization data of the synthesized compounds 2, 5, 6

Boc-Val-CH_2-Se-)$_2$, **2a:** Yield: 94%; Grey solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.91 (d, J = 6.8 Hz, 3H), 0.93 (d, J = 6.8 Hz, 3H), 1.44 (s, 9H), 1.88-1.92 (m, 1H), 3.12-3.17 (m, 2H), 3.63-3.65 (m, 1H), 4.80 (bd, J = 9.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.17, 20.00, 28.90, 31.56, 35.06, 56.82, 79.63, 156.21; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 281.02; HRMS, m/z: Calcd for $C_{20}H_{40}N_2O_4Se_2Na$ $(M + Na)^+$: 555.1216; found: 555.1213.

Boc-Leu-CH_2-Se-)$_2$, **2b:** Yield: 91%; Grey solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.93 (d, J = 8.8 Hz, 6H), 1.43 (s, 9H), 1.63-1.74 (m, 3H), 2.96-3.02 (m, 2H), 3.83-3.98 (m, 1H), 4.98 (bd, J = 9.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.54, 25.43, 28.91, 37.81, 43.51, 49.64, 79.59, 155.85; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 281.35; HRMS, m/z: Calcd for $C_{22}H_{44}N_2O_4Se_2Na$ $(M + Na)^+$: 583.1529; found: 583.1299.

Boc-Phe-CH_2-Se-)$_2$, 2c: Yield: 95%; Grey solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.41 (s, 9H), 2.90-3.03 (m, 2H), 3.16-3.26 (m, 2H), 4.07-4.09 (m, 1H), 5.03 (bd, *J* = 9.0 Hz, 1H), 7.16-7.29 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.38, 34.92, 39.50, 52.14, 79.38, 128.48, 128.52, 129.38, 137.44, 155.18; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 280.00; ESI-MS, m/z: Calcd for $C_{28}H_{40}N_2O_4Se_2Na$ $(M + Na)^+$: 651; found: 651.

Boc-Phg-CH_2-Se-)$_2$, 2d: Yield: 93%; Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.42 (s, 9H), 3.31-3.38 (m, 2H), 4.90-4.95 (m, 1H), 5.25 (brs, 1H), 7.26-7.39 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.84, 30.14, 55.78, 80.23, 126.88, 128.11, 129.16, 141.56, 155.59; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 301.91; ESI-MS, m/z: Calcd for $C_{26}H_{36}N_2O_4Se_2Na$ $(M + Na)^+$: 623; found: 623.

Cbz-Ala-CH_2-Se-)$_2$, 2e: Yield: 91%; Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (d, *J* = 6.8 Hz, 3H), 2.99-3.04 (m, 2H), 3.97-4.00 (m, 1H), 5.06 (s, 2H), 5.15 (brs, 1H), 7.26-7.33 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.67, 36.66, 46.64, 65.63, 127.12, 127.47, 127.50, 135.46, 154.60; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 288.53; ESI-MS, m/z: Calcd for $C_{22}H_{28}N_2O_4Se_2Na$ $(M + Na)^+$: 567.0277; found: 567.0271.

Cbz-Val-CH_2-Se-)$_2$, 2f: Yield: 92%; Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.84 (d, *J* = 8.0 Hz, 3H), 0.86 (d, *J* = 8.0 Hz, 3H), 1.80-1.85 (m, 1H), 3.00-3.09 (m, 2H), 3.63-3.70 (m, 1H), 4.99 (s, 2H), 5.05 (bd, *J* = 12.4 Hz, 1H), 7.18-7.25 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.83, 19.94, 31.87, 34.85, 57.75, 67.14, 128.53, 128.57, 128.94, 137.04, 156.82; ^{77}Se NMR (76

MHz, $CDCl_3$): δ 277.13; HRMS, m/z: Calcd for $C_{26}H_{36}N_2O_4Se_2Na$ $(M + Na)^+$: 623.0903; found: 623.0914.

Cbz-Phe-CH_2-Se-)$_2$, 2g: Yield: 94; Red solid; ^1H NMR (400 MHz, $CDCl_3$): δ 2.89 (d, *J* = 5.6 Hz, 2H), 3.01 (d, *J* = 7.2 Hz, 1H), 3.13 (d, *J* = 10.8 Hz, 1H), 4.12-4.21 (m, 1H), 5.03 (s, 2H), 5.16 (brs, 1H), 7.15-7.32 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 34.94, 39.68, 52.79, 66.63, 126.69, 128.07, 128.45, 128.49, 128.59, 129.62, 136.48, 137.22, 155.75; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 286.77; ESI-MS, m/z: Calcd for $C_{34}H_{37}N_2O_4Se_2$ $(M + H)^+$: 697.1084; found: 697.1094.

Fmoc-Phe-CH_2-Se-)$_2$, 2h: Yield: 92%; Golden yellow solid; ^1H NMR (400 MHz, $CDCl_3$): δ 2.90 (d, *J* = 6.4 Hz, 2H), 3.05-3.14 (m, 2H), 4.13-4.18 (m, 1H), 4.28 (t, *J* = 8.0 Hz, 1H), 4.37 (d, *J* = 8.0 Hz, 2H), 5.10 (brs, 1H, NH), 7.15-7.73 (m, 13H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 34.58, 39.76, 47.21, 52.87, 69.06, 119.92, 124.99, 126.70, 127.00, 127.63, 128.58, 129.36, 137.15, 141.29, 143.86, 155.71; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 286.36; ESI-MS, m/z: Calcd for $C_{48}H_{45}N_2O_4Se_2$ $(M + H)^+$: 873; found: 873.

Fmoc-Ile-CH_2-Se-)$_2$, 2i: Yield: 94%; Golden yellow solid; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85-0.92 (m, 6H), 1.06-1.13 (m, 2H), 1.41-1.51 (m, 1H), 3.11 (d, *J* = 8.4 Hz, 1H), 3.17 (d, *J* = 3.6 Hz, 1H), 3.79-3.86 (m, 1H), 4.18 (t, *J* = 6.0 Hz, 1H), 4.36 (d, *J* = 6.0 Hz, 2H), 5.00 (bd, *J* = 9.2 Hz, 1H), 7.26-7.75 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 12.06, 15.82, 25.52, 34.04, 38.92, 47.75, 56.94, 67.00, 125.52, 125.57, 127.47, 128.09, 141.76, 144.41, 156.67; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 299.31; ESI-MS, m/z: Calcd for $C_{42}H_{48}N_2O_4Se_2Na$ $(M + Na)^+$: 827; found: 827.

Fmoc-Leu-CH_2-Se-)$_2$, 2j: Yield: 93%; Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.91 (d, *J* = 6.8 Hz, 6H), 1.42 (t, *J* = 6.8 Hz, 2H), 1.62-1.67 (m, 1H), 3.08 (d, *J* = 5.2 Hz, 1H), 3.18 (d, *J* = 5.2 Hz, 1H), 3.94 (t, *J* = 6.0 Hz, 1H), 4.17 (d, *J* = 6.0 Hz, 2H), 4.34-4.41 (m, 1H), 5.09 (bd, *J* = 8.4 Hz, 1H), 7.26-7.75 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.60, 25.48, 37.34, 43.77, 47.76, 50.23, 66.89, 120.43, 125.54, 127.48, 128.12, 141.78, 144.40, 156.44; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 296.92; ESI-MS, m/z: Calcd for $C_{42}H_{49}N_2O_4Se_2$ $(M + H)^+$: 805; found: 805.

Boc-L-Sec-OMe, **5a:** Yield: 89%; Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.37 (s, 9H), 3.33-3.40 (m, 2H), 3.69 (s, 3H), 4.55-4.58 (m, 1H), 5.39 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.90, 35.06, 52.17, 56.82, 79.63, 156.21, 172.81; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 371.11; ESI-MS, m/z: Calcd for $C_{18}H_{32}N_2O_8Se_2Na$ $(M + Na)^+$: 587; found: 587.

Cbz-L-Sec-OMe, **5b:** Yield: 85%; Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.27-3.33 (m, 2H), 3.73 (s, 3H), 4.62-4.68 (m, 1H), 5.01 (s, 2H), 5.42 (s, 1H), 7.22-7.27 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 37.79, 52.25, 53.18, 66.97, 127.05, 128.52, 129.19, 135.95, 156.79, 172.20; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 373.26; ESI-MS, m/z: Calcd for $C_{24}H_{28}N_2O_8Se_2Na$ $(M + Na)^+$: 655; found: 655.

Fmoc-L-Sec-OMe, **5c:** Yield: 87%; Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.28-3.35 (m, 2H), 3.66 (s, 3H), 4.17 (t, *J* = 6.0 Hz, 1H), 4.35 (d, *J* = 6.0 Hz, 2H), 4.54-4.59 (m, 1H), 5.02 (brs, 1H), 7.25-7.74 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 34.18, 47.20, 52.23, 54.80, 67.29,

120.14, 125.17, 127.29, 127.90, 141.39, 143.69, 156.55, 171.67; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 374.12; ESI-MS, m/z: Calcd for $C_{38}H_{36}N_2O_8Se_2Na$ $(M + Na)^+$: 831; found: 831.

Boc-Val-CH_2-SeO_2H, **6a:** Yield: 90%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, *J* = 4.4 Hz, 6H), 1.43 (s, 9H), 1.87-1.92 (m, 1H), 3.33-3.37 (m, 2H), 3.83-3.89 (m, 1H), 4.86 (bd, *J* = 3.6 Hz, 1H), 8.14 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.32, 28.77, 32.99, 52.00, 62.15, 80.77, 156.59; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 1213.18; ESI-MS (in methanol solution), m/z: Calcd for $C_{11}H_{23}NO_4SeNa$ (as seleninate methyl ester + $Na)^+$: 336; found: 336.

Boc-Leu-CH_2-SeO_2H, **6b:** Yield: 90%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.92 (d, *J* = 4.4 Hz, 6H), 1.42 (s, 9H), 1.63-1.78 (m, 3H), 3.23 (d, *J* = 9.6 Hz, 1H), 3.36 (d, *J* = 11.6 Hz, 1H), 4.04-4.11 (m, 1H), 4.90 (brs, 1H), 7.38 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.21, 25.33, 28.82, 44.59, 45.62, 64.81, 80.98, 156.63; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 1214.61; ESI-MS, m/z: Calcd for $C_{12}H_{25}NO_4SeNa$ (as seleninate methyl ester + $Na)^+$: 350; found: 350.

Boc-Phe-CH_2-SeO_2H, **6c:** Yield: 91%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.39 (s, 9H), 2.86-2.98 (m, 2H), 3.26-3.34 (m, 2H), 4.23-4.31 (m, 1H), 5.08 (brs, 1H), 7.18-7.30 (m, 5H), 8.87 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.80, 41.45, 48.38, 62.72, 80.97, 127.59, 129.33, 129.86, 136.87, 156.40; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 1213.63; ESI-MS (in methanol solution), m/z: Calcd for $C_{15}H_{23}NO_4SeNa$ (as seleninate methyl ester + $Na)^+$: 384; found: 384.

Boc-Phg-CH_2-SeO_2H, **6d:** Yield: 89%; White solid; ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.40 (s, 9H), 3.31-3.35 (m, 2H), 4.30 (t, J = 11.6 Hz, 1H), 4.95 (brs, 1H), 7.25-7.37 (m, 5H), 7.74 (brs, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 29.05, 50.66, 63.74, 79.29, 126.99, 128.21, 129.50, 143.01, 155.54; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 1215.35; ESI-MS (in methanol solution), m/z: Calcd for $C_{14}H_{21}NO_4SeNa$ (as seleninate methyl ester + Na)$^+$: 370; found: 370.

Cbz-Ala-CH_2-SeO_2H, **6e:** Yield: 92%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.28 (d, J = 5.8 Hz, 3H), 2.98 (d, J = 5.2 Hz, 1H), 3.03 (d, J = 5.2 Hz, 1H), 4.09-4.17 (m, 1H), 5.15 (s, 2H), 6.21 (brs, 1H), 7.21-7.27 (m, 5H), 8.02 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.83, 48.44, 61.37, 67.18, 127.05, 127.87, 128.70, 136.17, 156.67; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 1218.31; ESI-MS, m/z: Calcd for $C_{11}H_{15}NO_4Se$: 305; found: 304 (M^-).

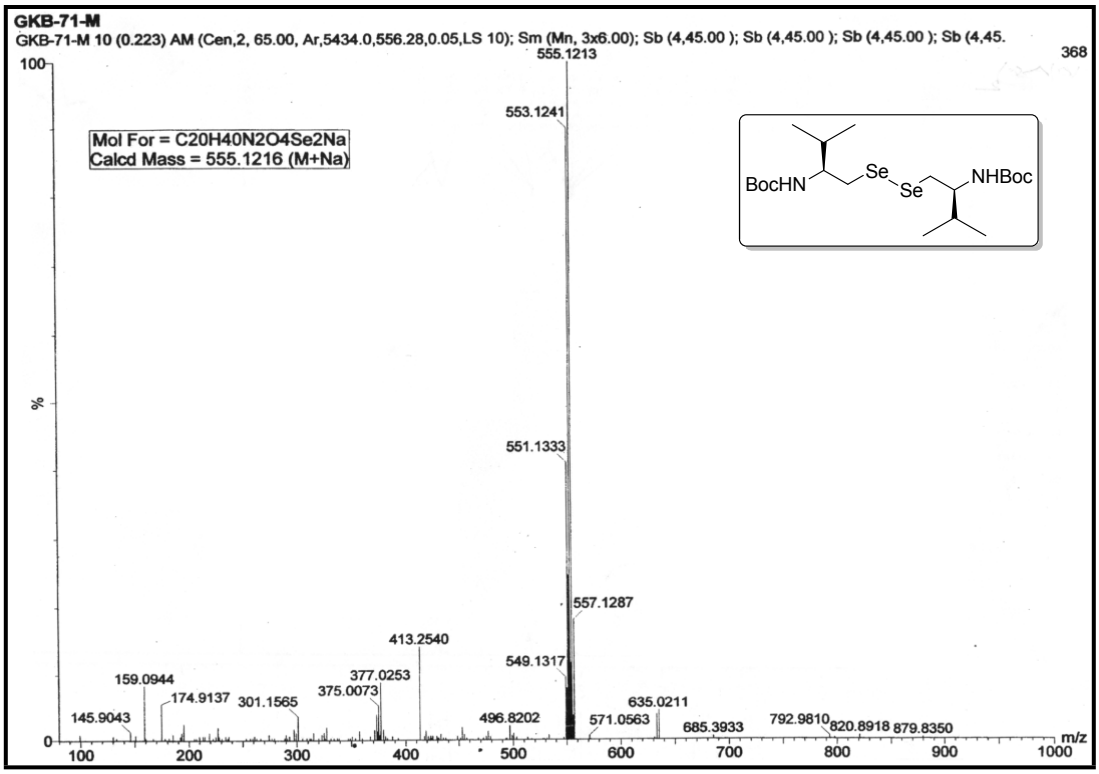
Cbz-Val-CH_2-SeO_2H, **6f:** Yield: 90%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.95 (d, J = 6.4 Hz, 6H), 1.88-1.93 (m, 1H), 3.18-3.31 (m, 2H), 3.93-3.98 (m, 1H), 5.09 (s, 2H), 5.32 (bd, J = 8.0 Hz, 1H), 6.50 (s, 1H), 7.26-7.36 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.17, 32.68, 52.23, 61.03, 67.19, 128.26, 128.51, 128.58, 136.17, 156.65; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 1214.04; ESI-MS (in methanol solution), m/z: Calcd for $C_{14}H_{21}NO_4SeNa$ (as seleninate methyl ester + Na)$^+$: 370; found: 370.

Cbz-Phe-CH_2-SeO_2H, **6g:** Yield: 92%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.88-2.98 (m, 2H), 3.26 (d, J = 5.6 Hz, 2H), 4.32-4.39 (m, 1H), 5.05 (s, 2H), 5.33 (brs, 1H), 7.14-7.35 (m, 10H), 8.37 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 41.03, 48.40, 61.33, 67.14, 127.24, 128.17, 128.27, 128.55, 128.92, 129.32, 136.13, 137.03, 156.29; ^{77}Se NMR (76 MHz, $CDCl_3$): δ

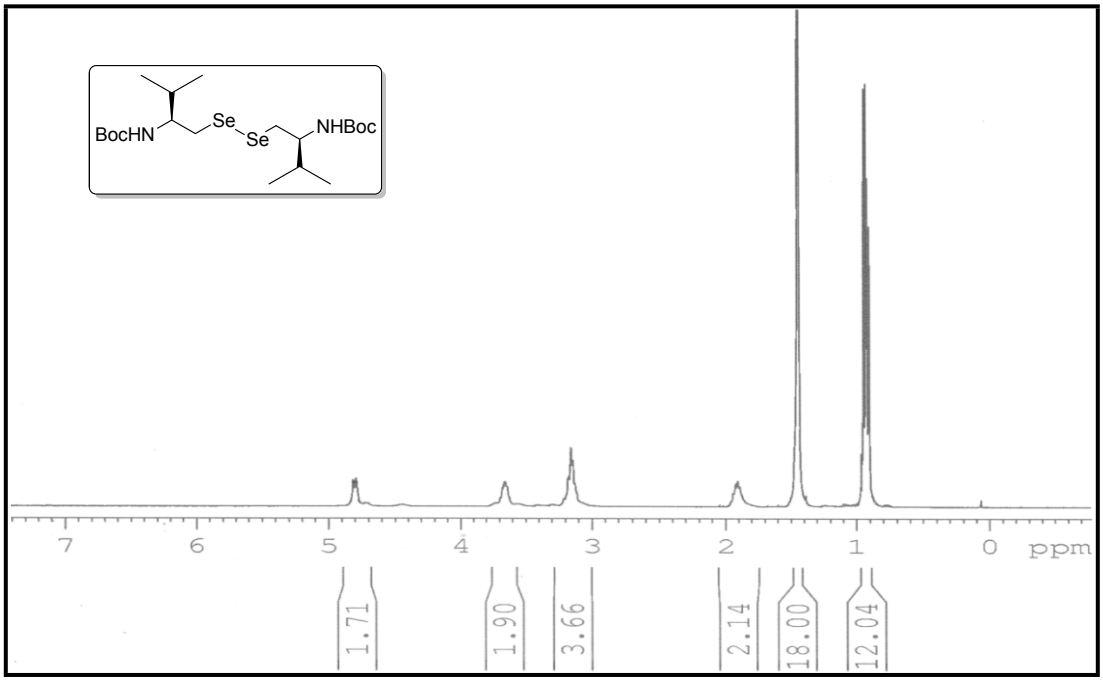
1216.43; ESI-MS (in methanol solution), m/z: Calcd for $C_{18}H_{22}NO_4Se$ (as seleninate methyl ester + H)$^+$: 396; found: 396.

Fmoc-Phe-CH_2-SeO_2H, **6h:** Yield: 90%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.91-2.99 (m, 2H), 3.23-3.27 (m, 2H), 3.99-4.03 (m, 1H), 4.13 (t, *J* = 6.0 Hz, 1H), 4.25 (d, *J* = 6.0 Hz, 2H), 5.31 (s, 1H), 7.18-7.70 (m, 13H), 8.49 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 39.81, 47.19, 52.92, 62.58, 69.06, 119.93, 124.85, 126.72, 127.02, 127.65, 128.58, 129.36, 137.16, 141.31, 143.86, 155.72; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 1216.13; ESI-MS (in methanol solution), m/z: Calcd for $C_{25}H_{26}NO_4Se$ (as seleninate methyl ester + H)$^+$: 484; found: 484.

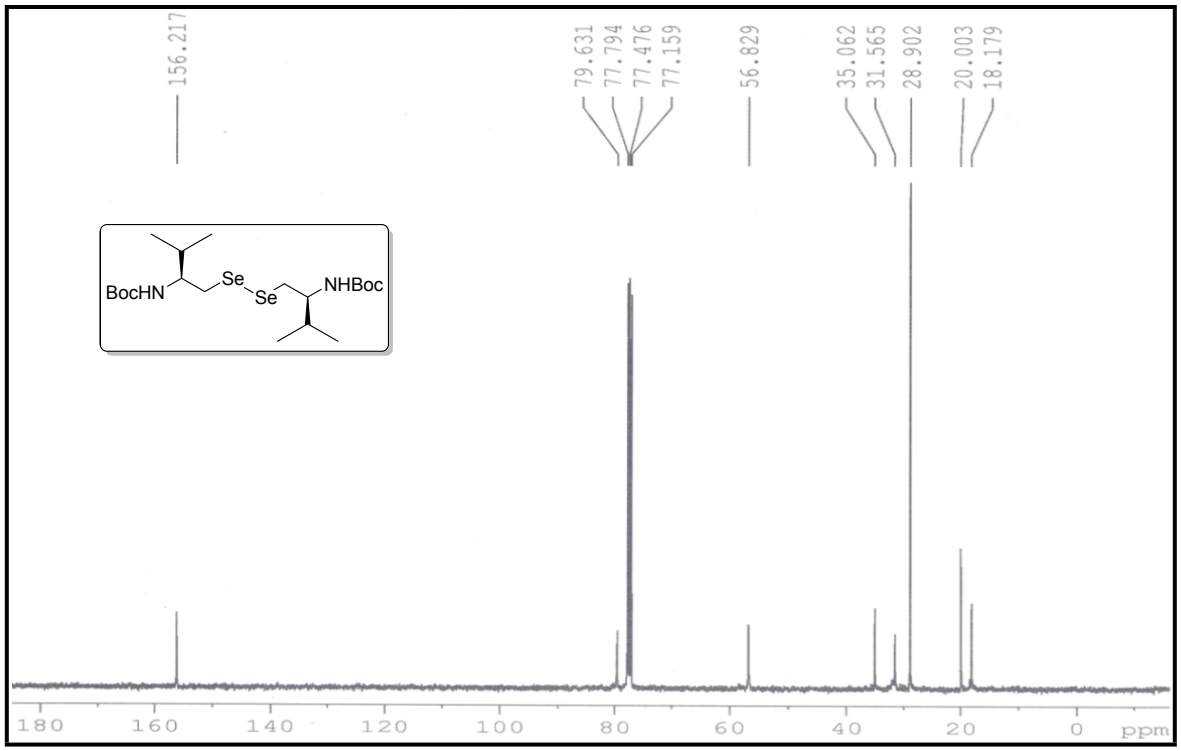
Fmoc-Ile-CH_2-SeO_2H, **6i:** Yield: 91%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.82-0.86 (m, 6H), 1.18-1.23 (m, 2H), 2.02-2.08 (m, 1H), 3.09 (d, *J* = 5.4 Hz, 1H), 3.16 (d, *J* = 5.4 Hz, 1H), 3.99-4.03 (m, 1H), 4.18 (t, *J* = 6.0 Hz, 1H), 4.29 (d, *J* = 6.0 Hz, 2H), 5.31 (s, 1H), 7.16-7.74 (m, 8H), 8.47 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 12.06, 18.24, 25.35, 43.65, 45.73, 62.07, 47.17, 68.35, 126.69, 127.18, 127.74, 128.66, 141.33, 143.91, 156.17; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 1217.81; ESI-MS (in methanol solution), m/z: Calcd for $C_{22}H_{27}NO_4SeNa$ (as seleninate methyl ester + Na)$^+$: 472; found: 472.



HRMS of Boc-Val-CH_2-Se-)$_2$, 2a



^{1}H NMR of Boc-Val-CH_2-Se-)$_2$, 2a



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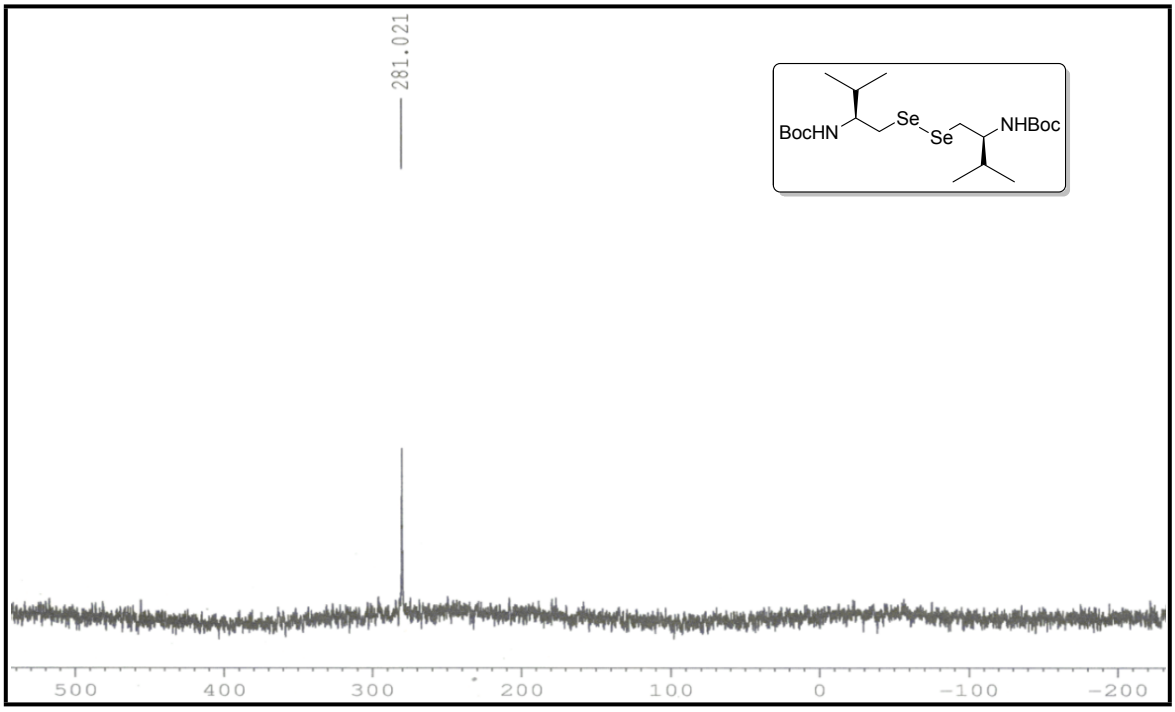

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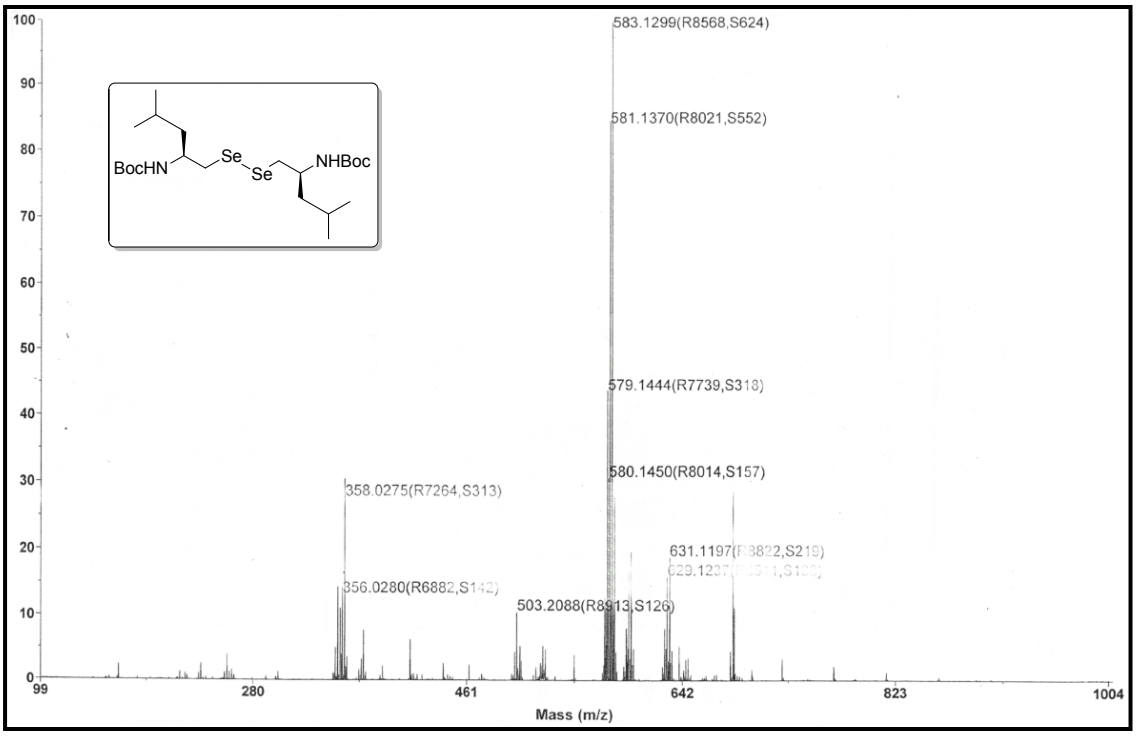

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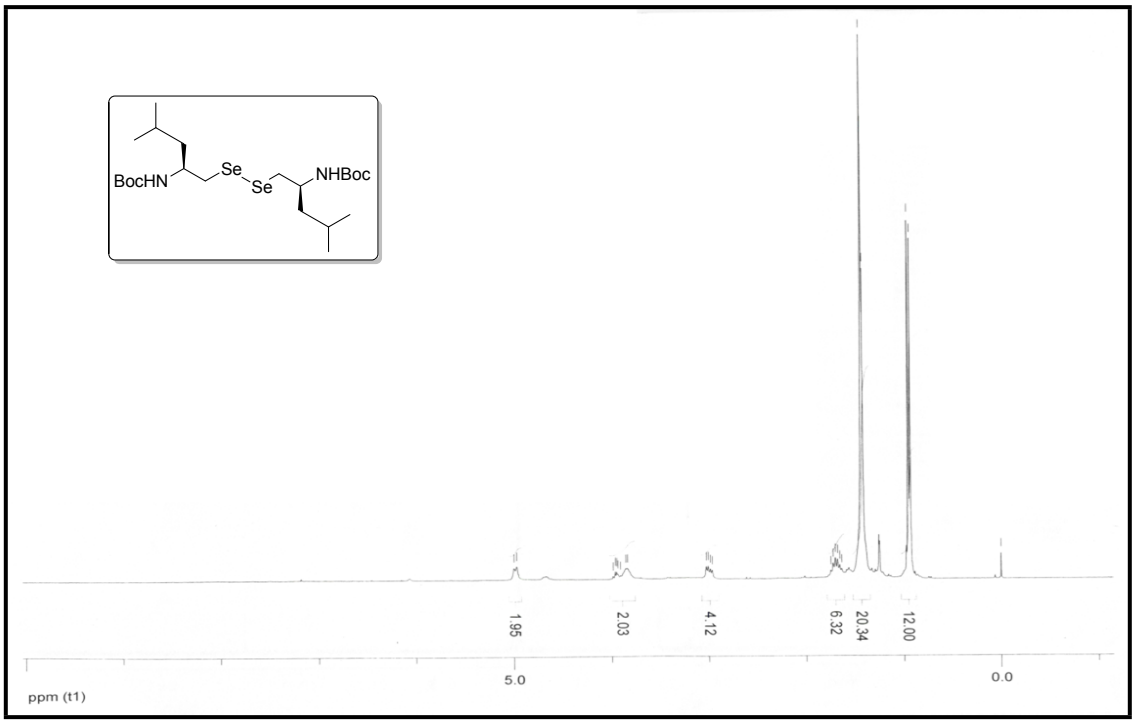

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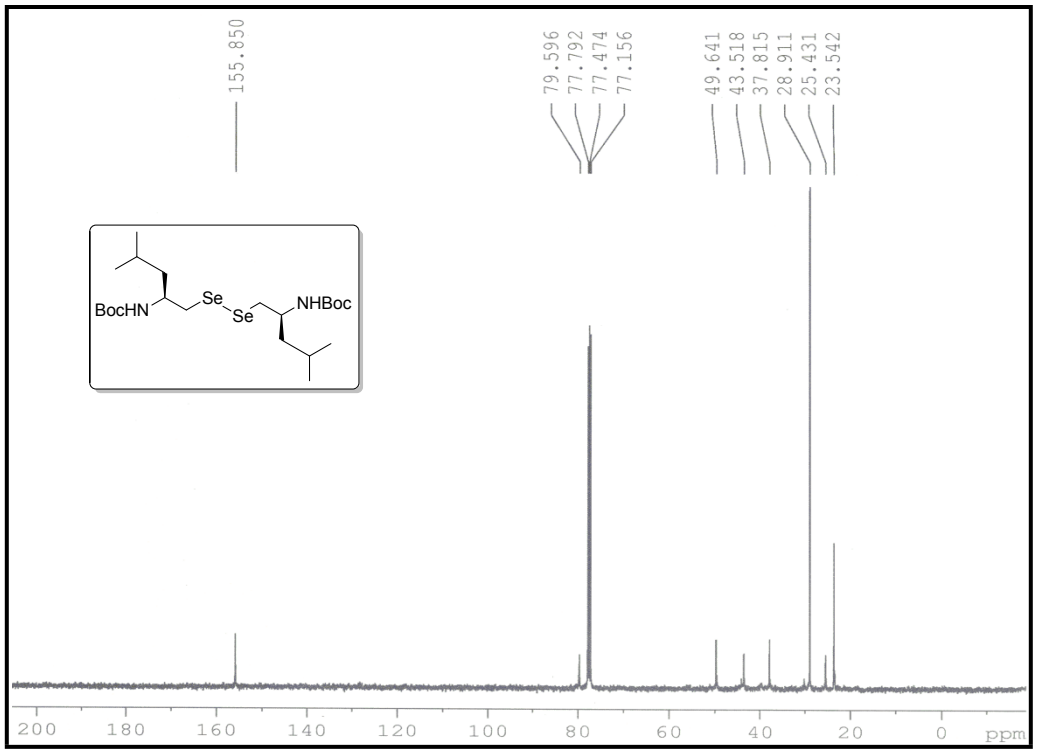

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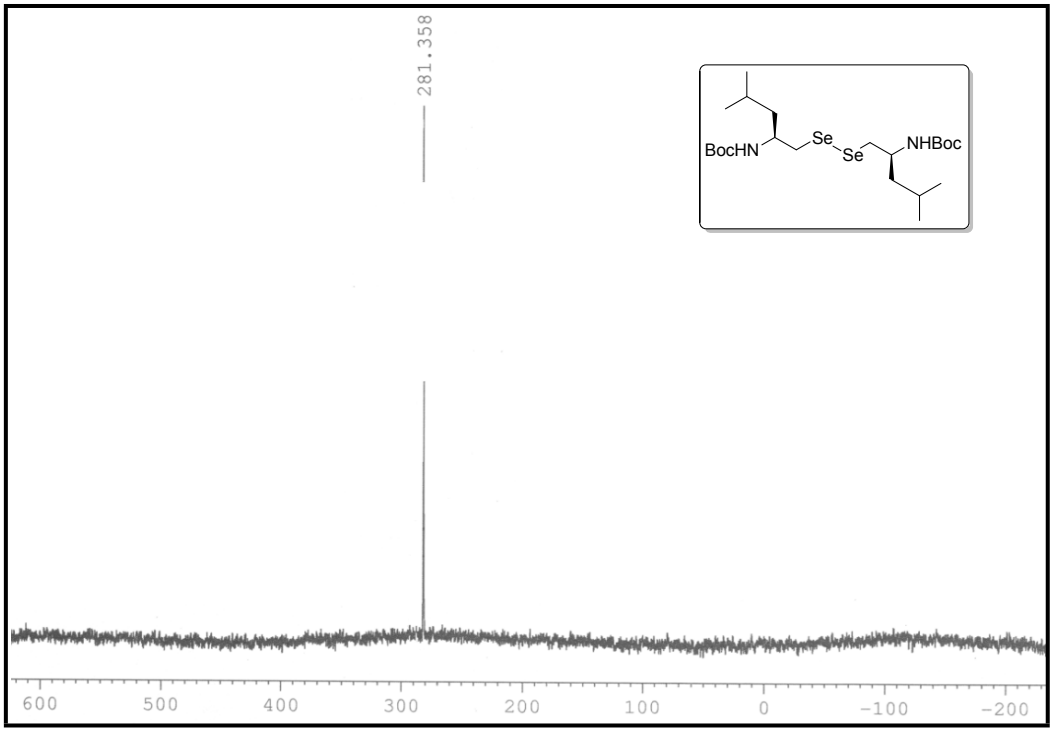
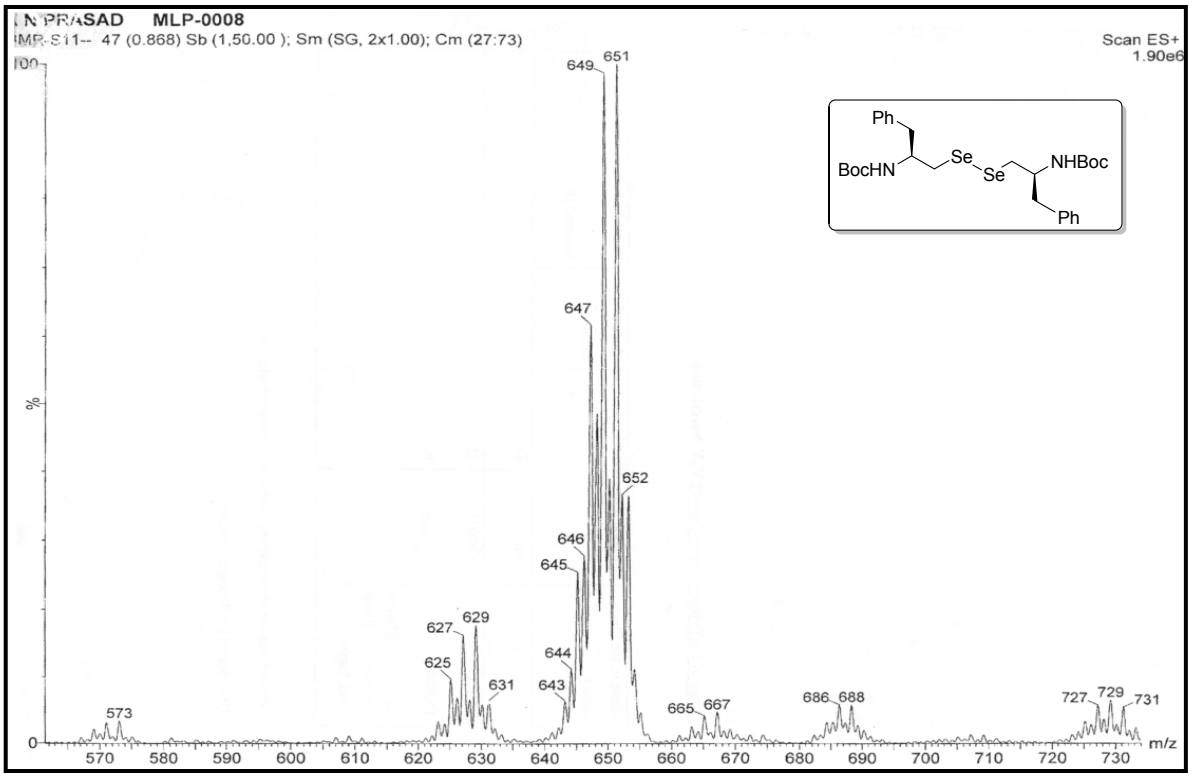
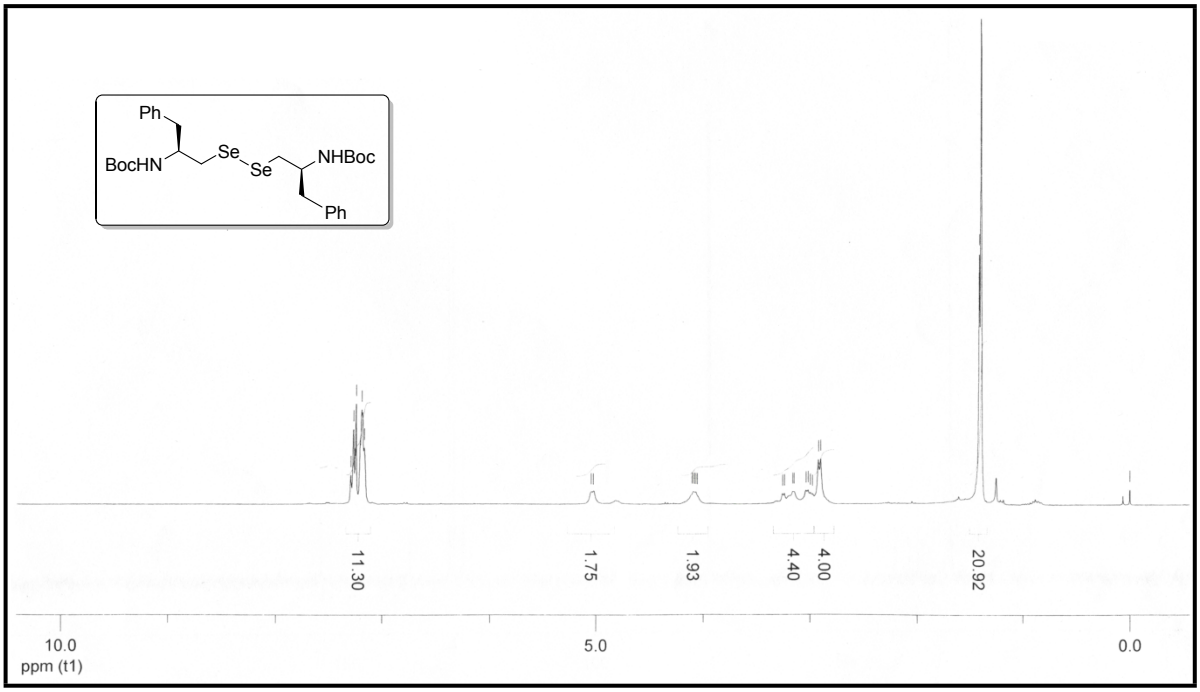


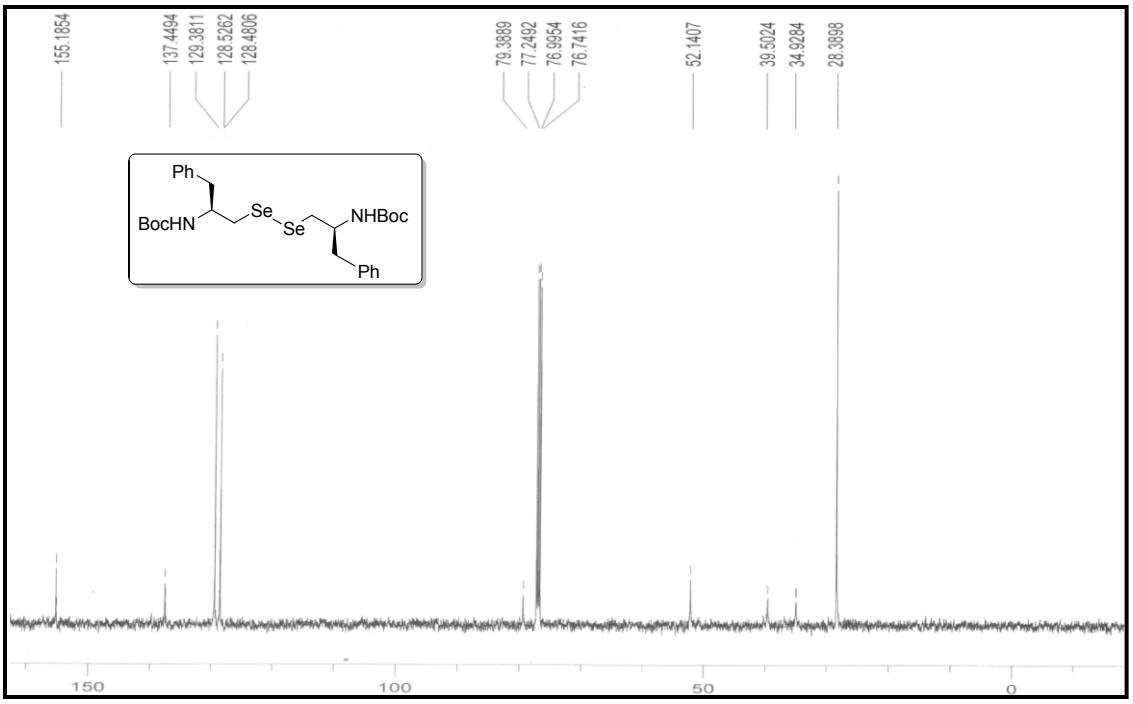
^{77}Se NMR of Boc-Leu-CH_2-Se-)$_2$, 2b



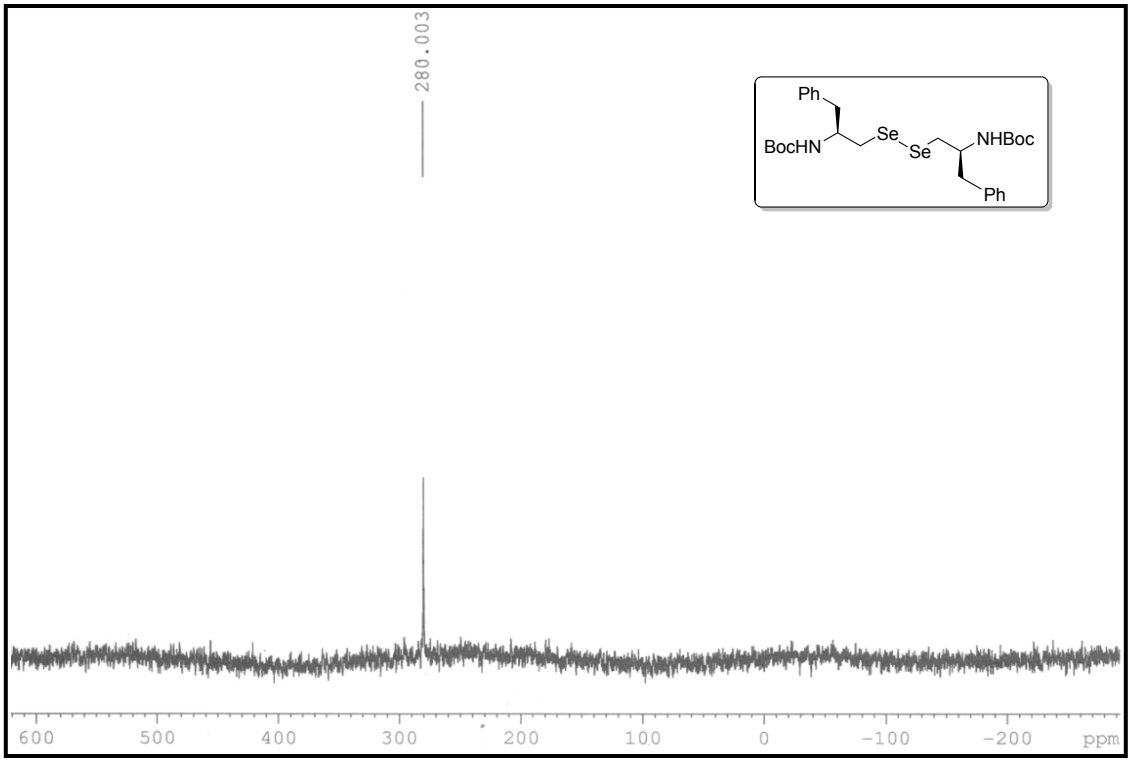
ESI-MS of Boc-Phe-CH_2-Se-)$_2$, 2c



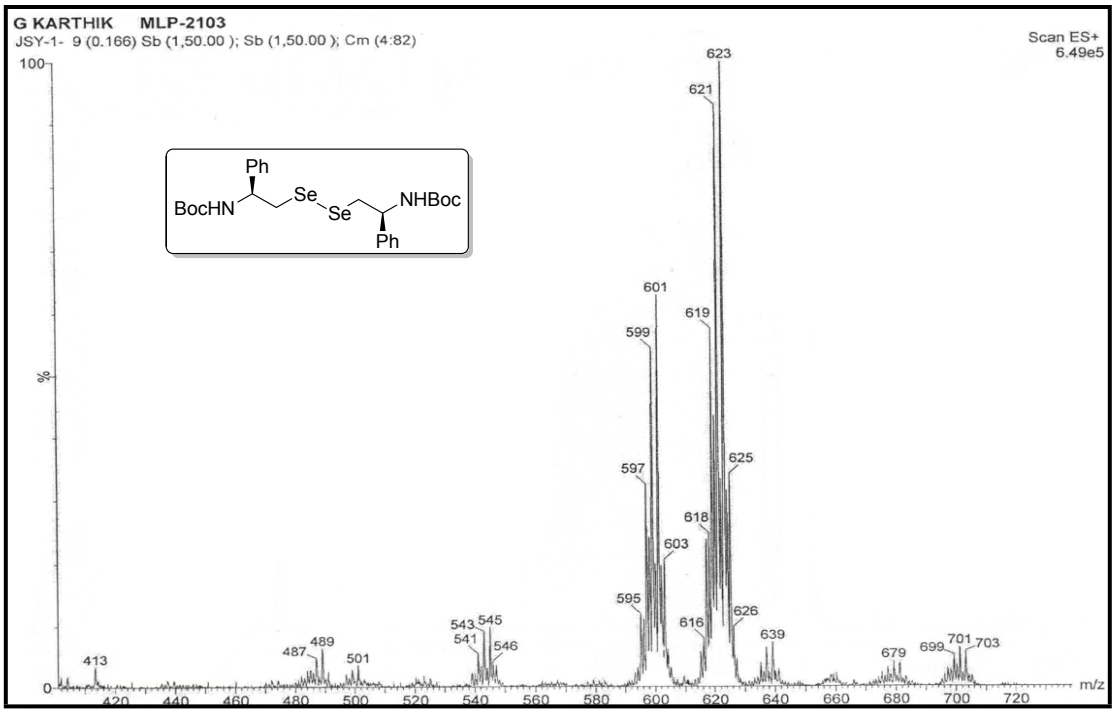
^{1}H NMR of Boc-Phe-CH_2-Se-)$_2$, 2c



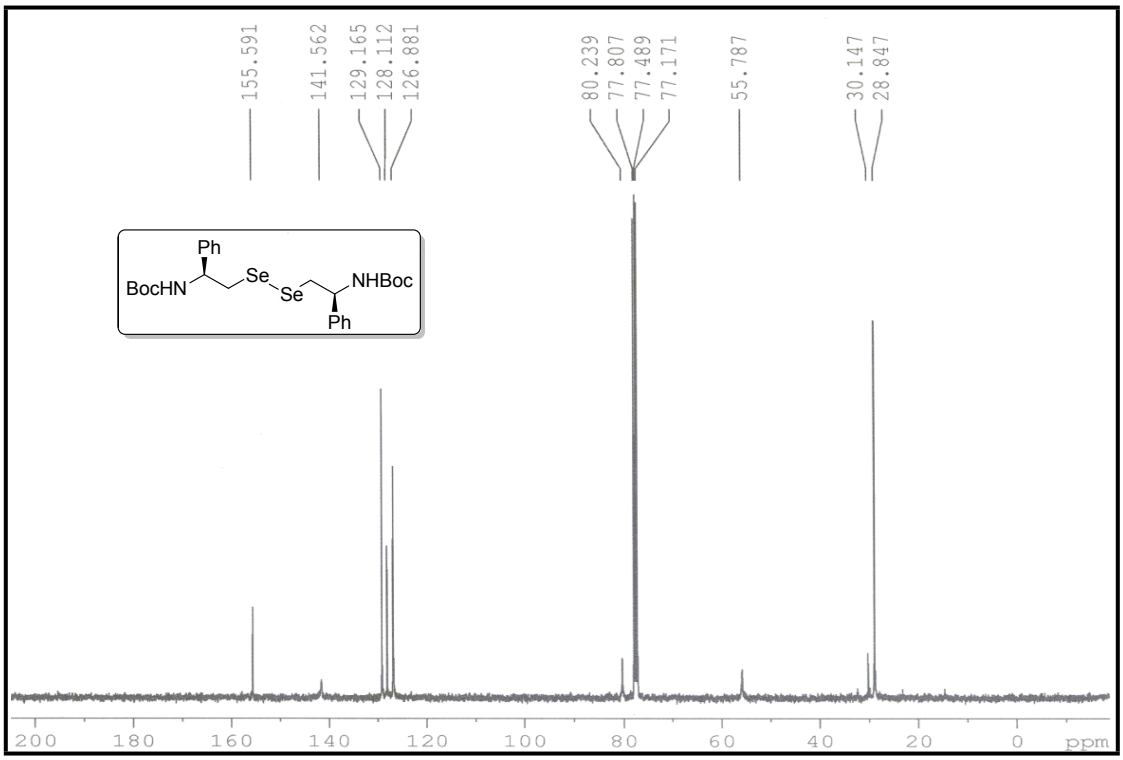
^{13}C NMR of Boc-Phe-CH$_2$-Se-)$_2$, 2c



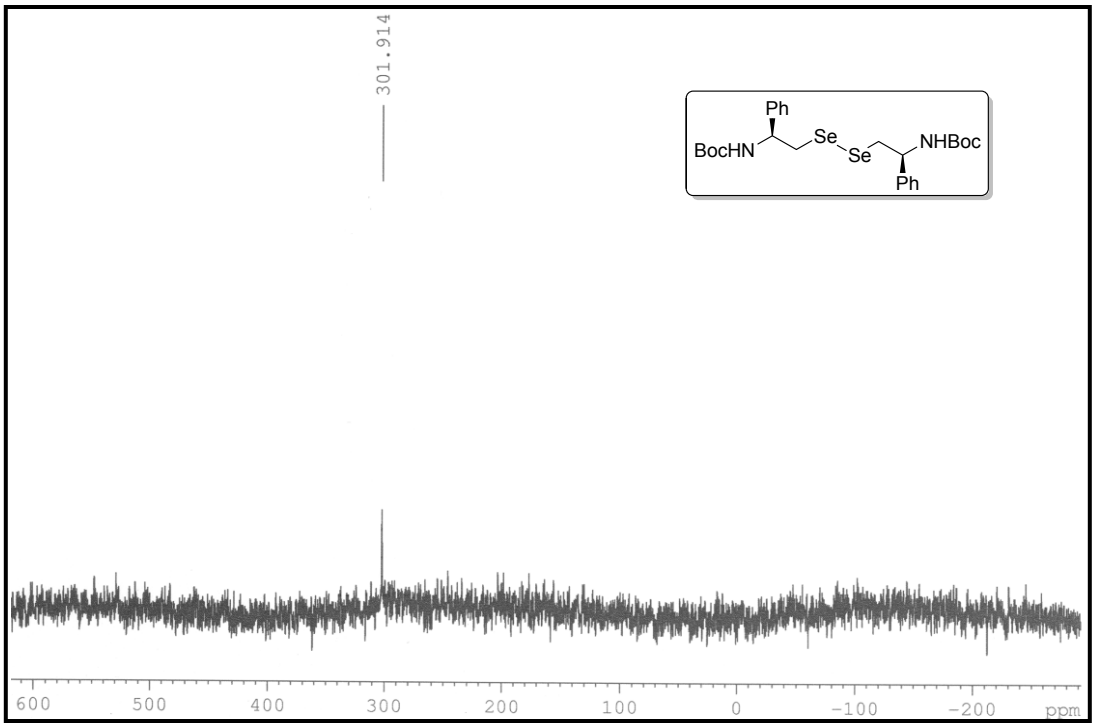
^{77}Se NMR of Boc-Phe-CH$_2$-Se-)$_2$, 2c



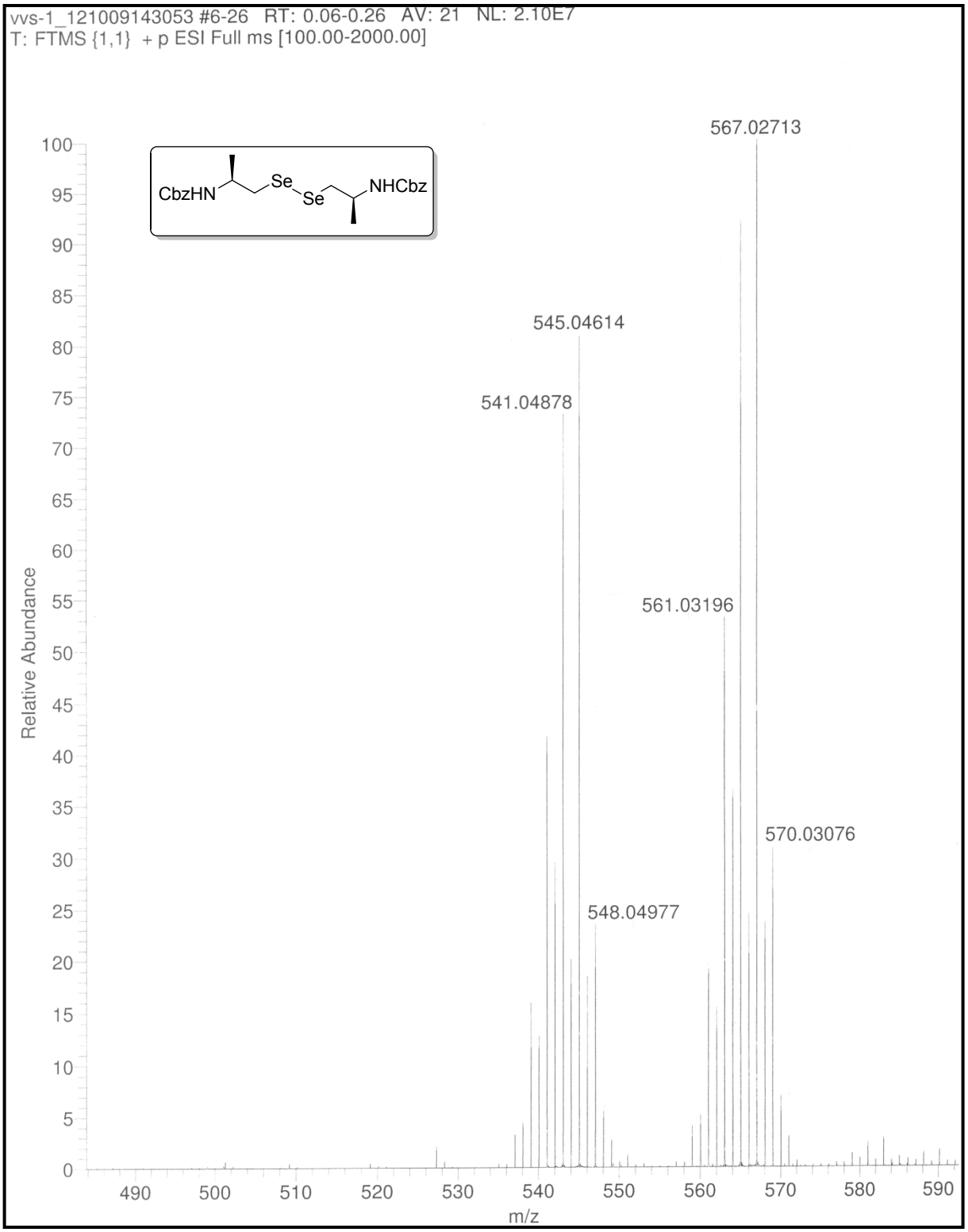
ESI-MS of Boc-Phg-CH_2-Se-)$_2$, 2d



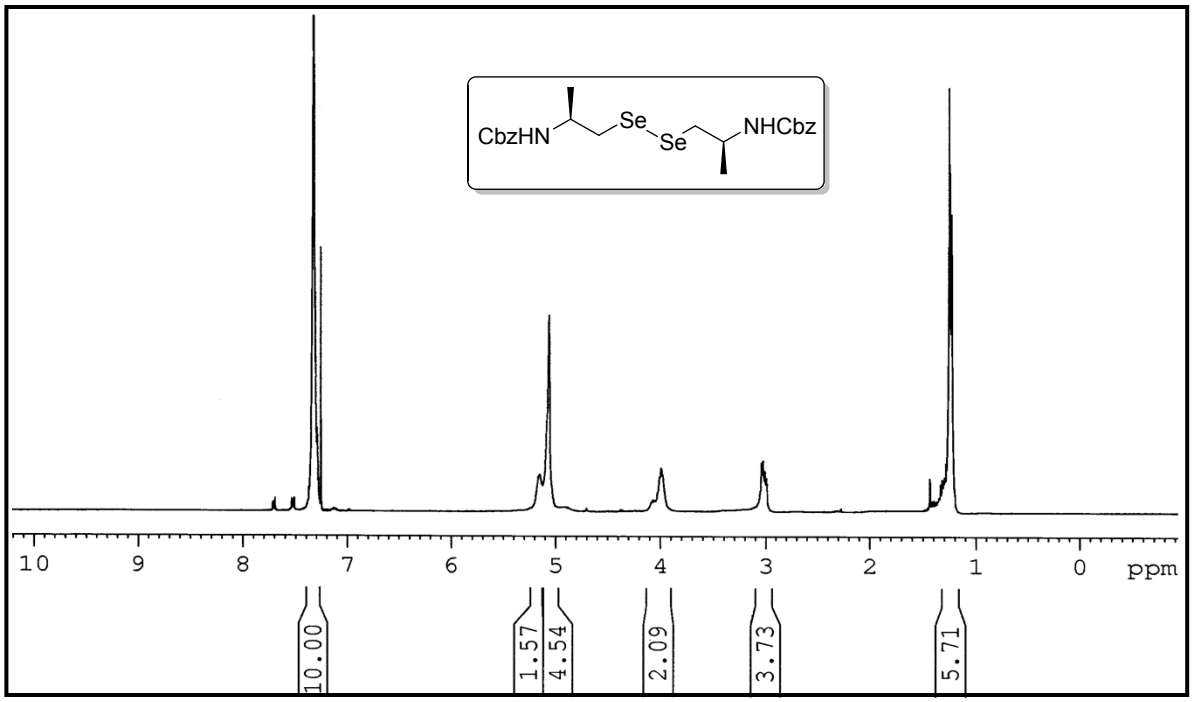
^{13}C NMR of Boc-Phg-CH_2-Se-)$_2$, 2d



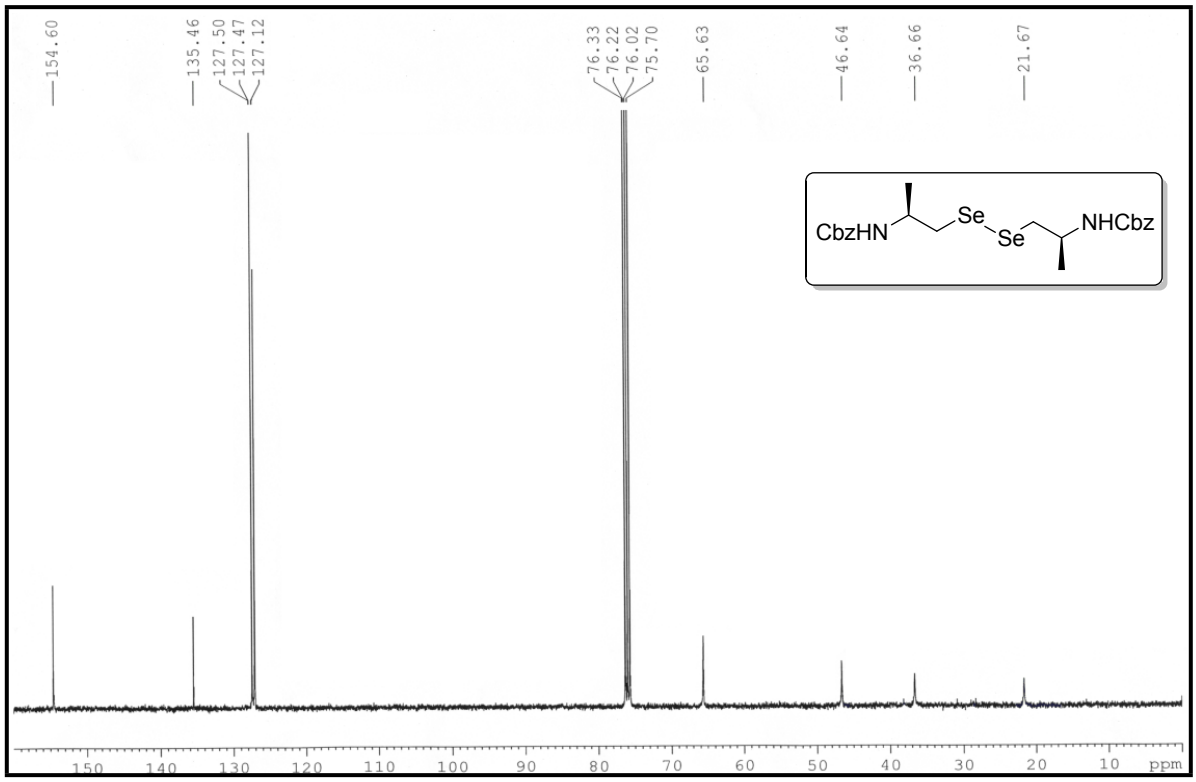
^{77}Se NMR of Boc-Phg-CH_2-Se-)$_2$, 2d



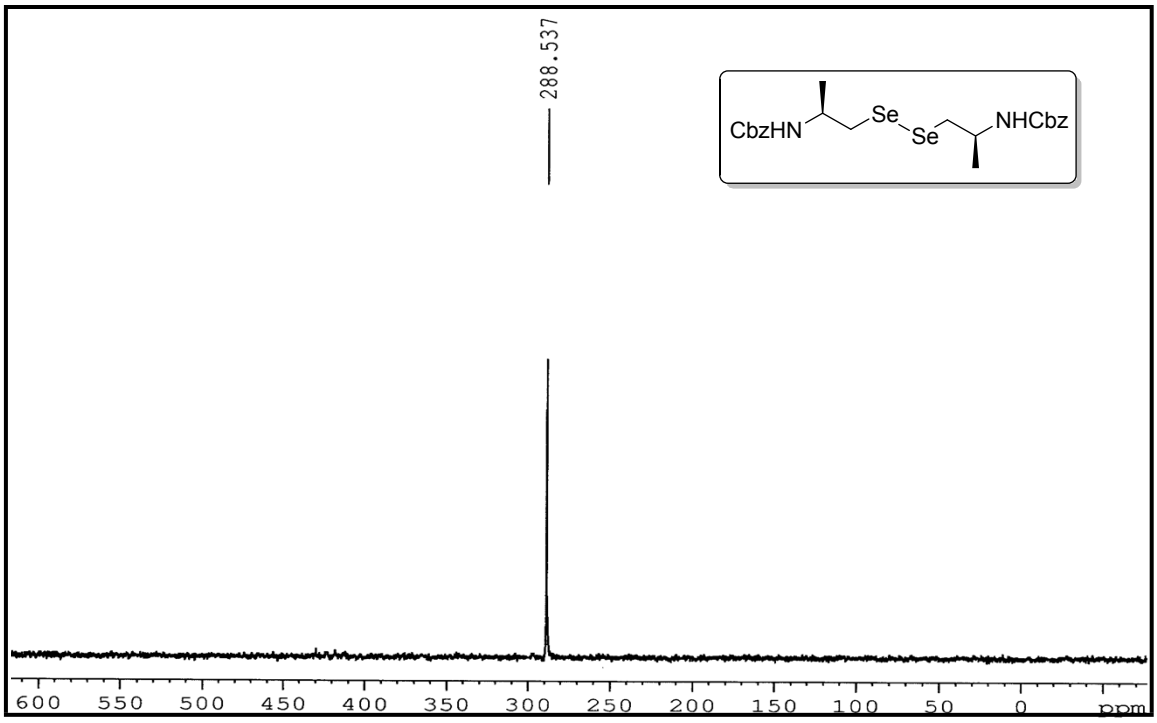
ESI-MS of Cbz-Ala-CH_2-Se-)$_2$, 2e



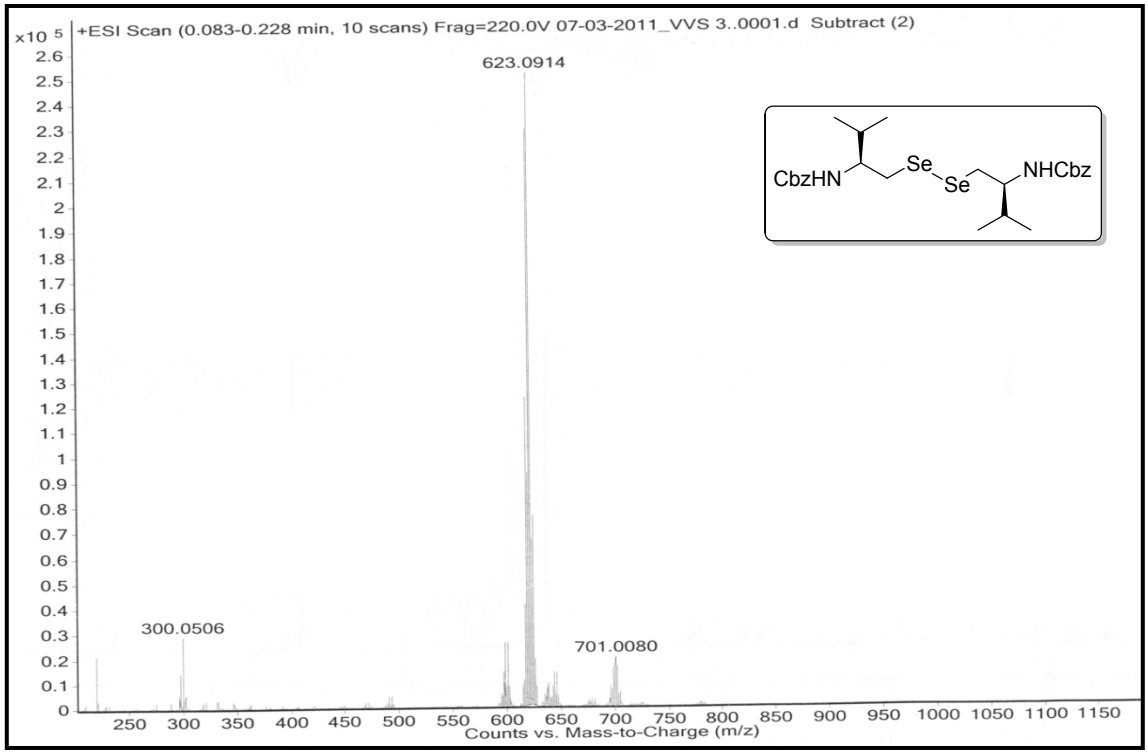
^{1}H NMR of Cbz-Ala-CH_2-Se-)$_2$, 2e



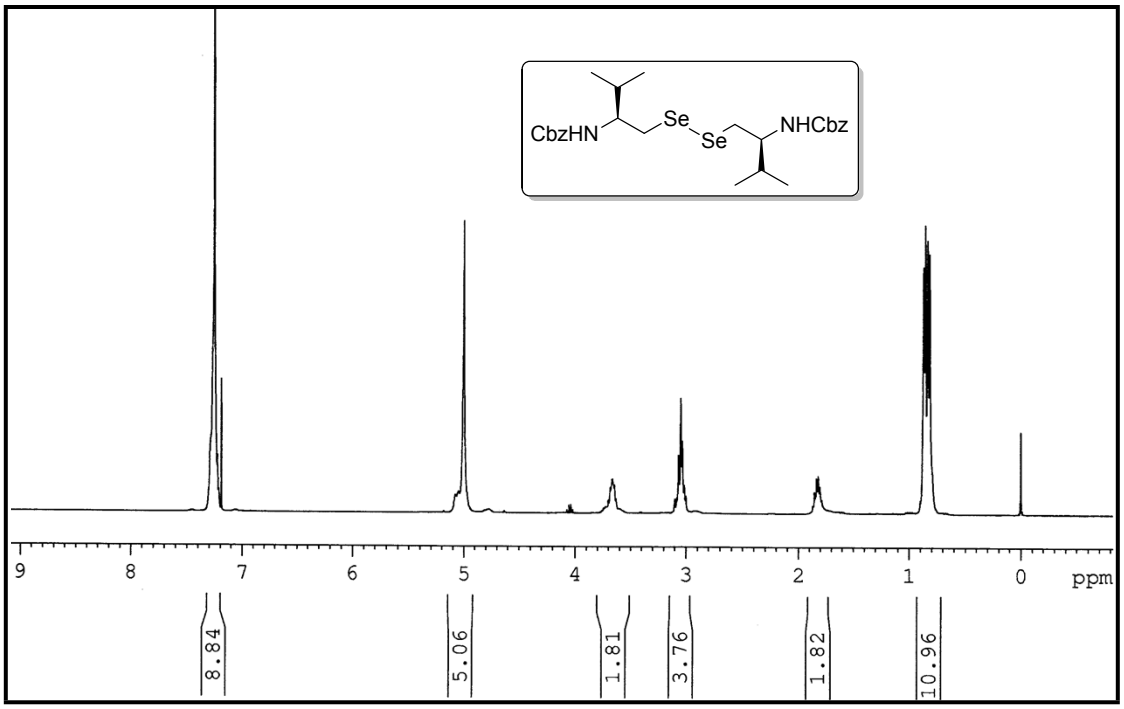
^{13}C NMR of Cbz-Ala-CH_2-Se-)$_2$, 2e



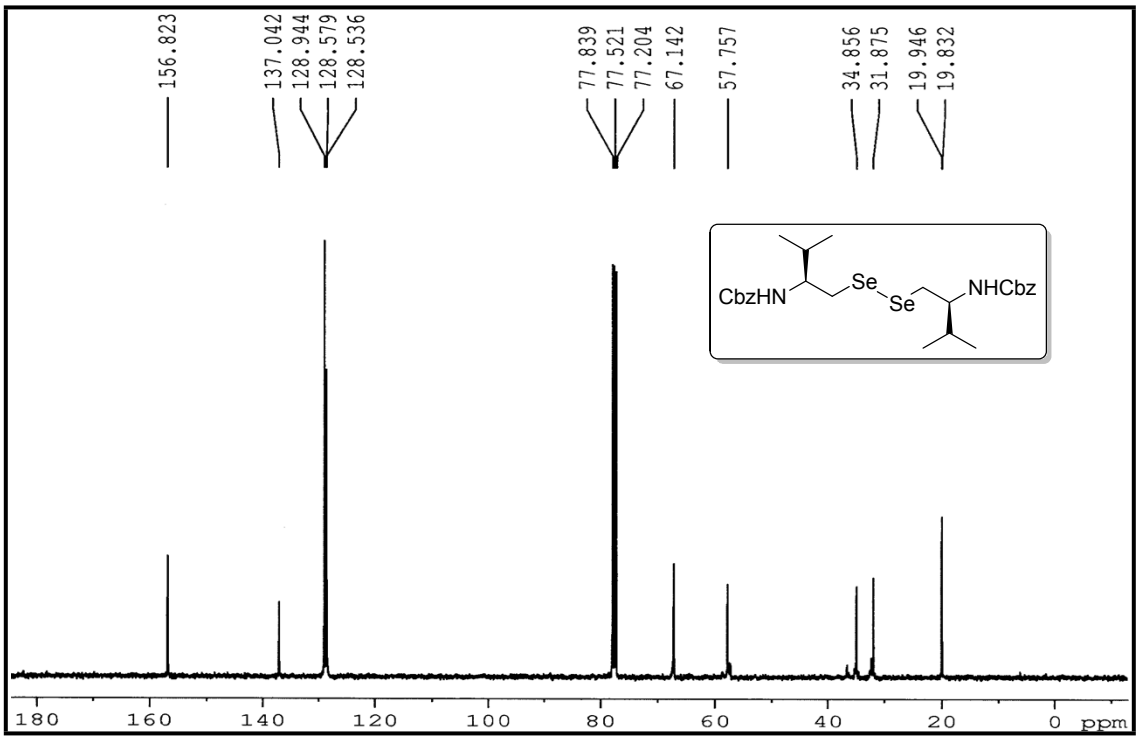
^{77}Se NMR of Cbz-Ala-CH_2-Se-)$_2$, 2e



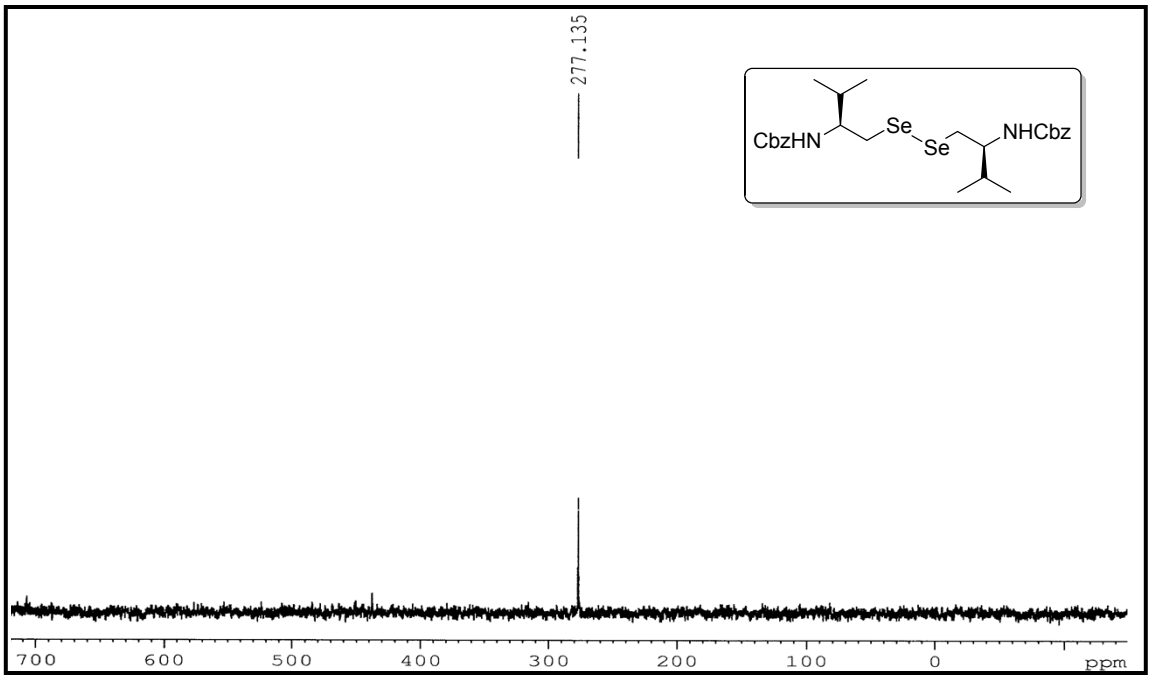
HRMS of Cbz-Val-CH_2-Se-)$_2$, 2f



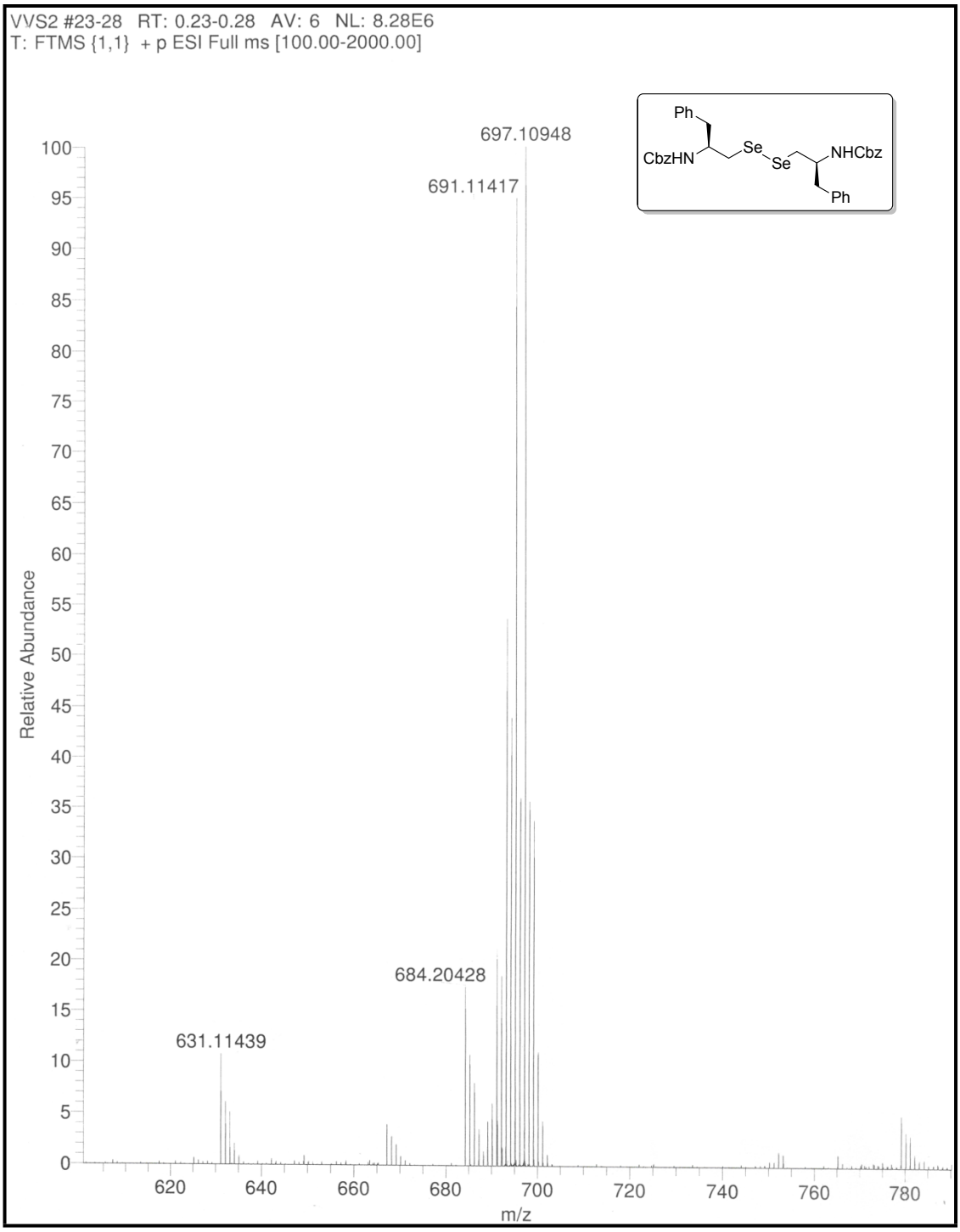
^{1}H NMR of Cbz-Val-CH_2-Se-)$_2$, 2f



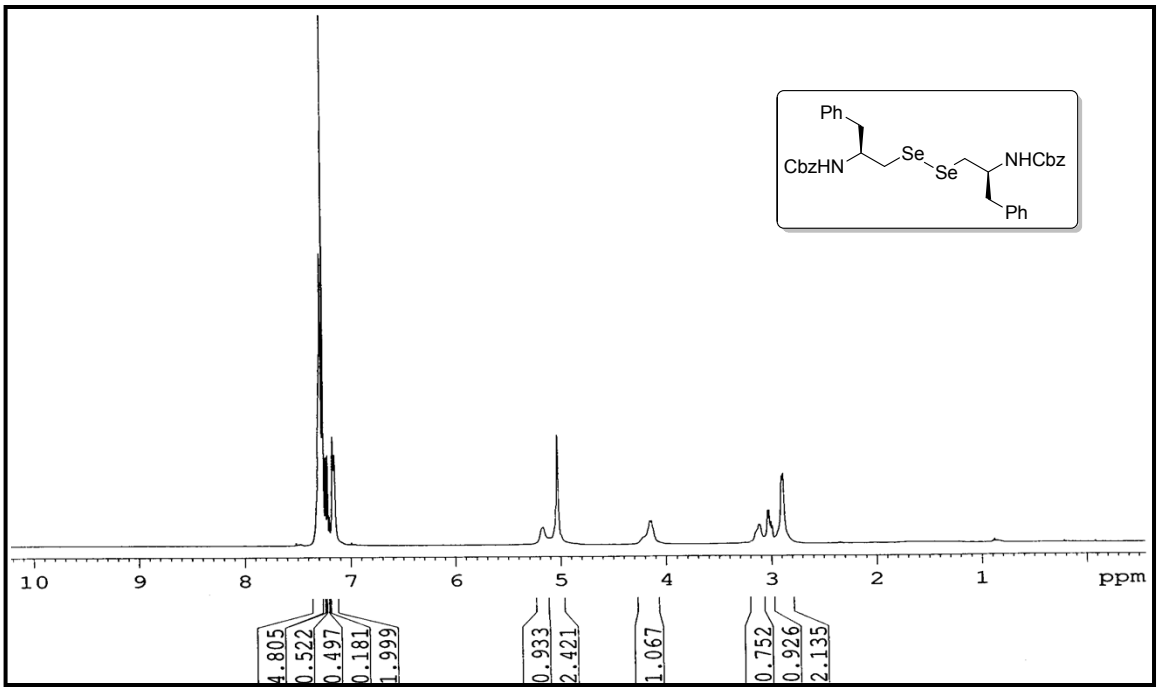
^{13}C NMR of Cbz-Val-CH_2-Se-)$_2$, 2f



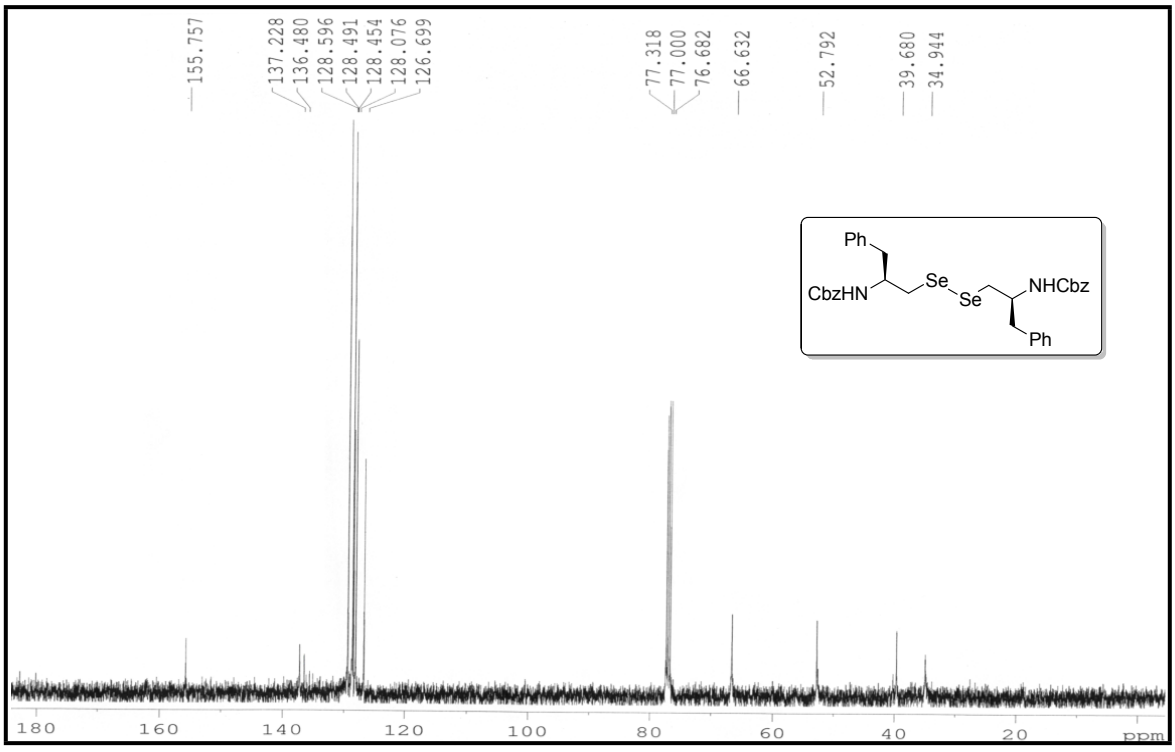
^{77}Se NMR of Cbz-Val-CH_2-Se-)$_2$, 2f



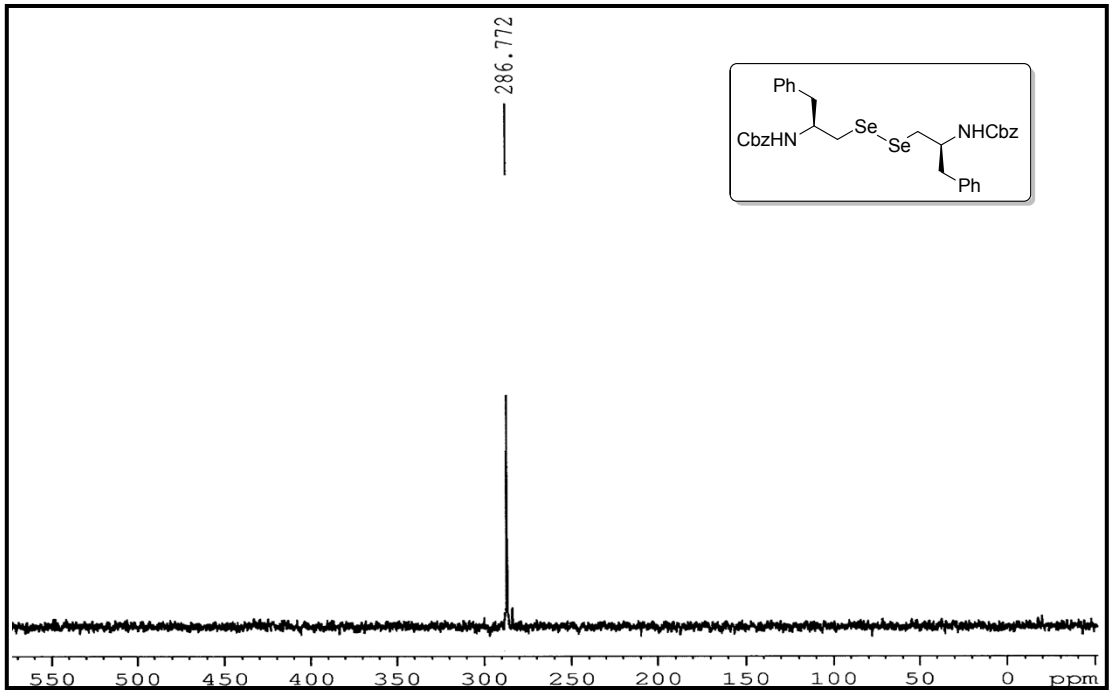
ESI-MS of Cbz-Phe-CH_2-Se-)$_2$, 2g



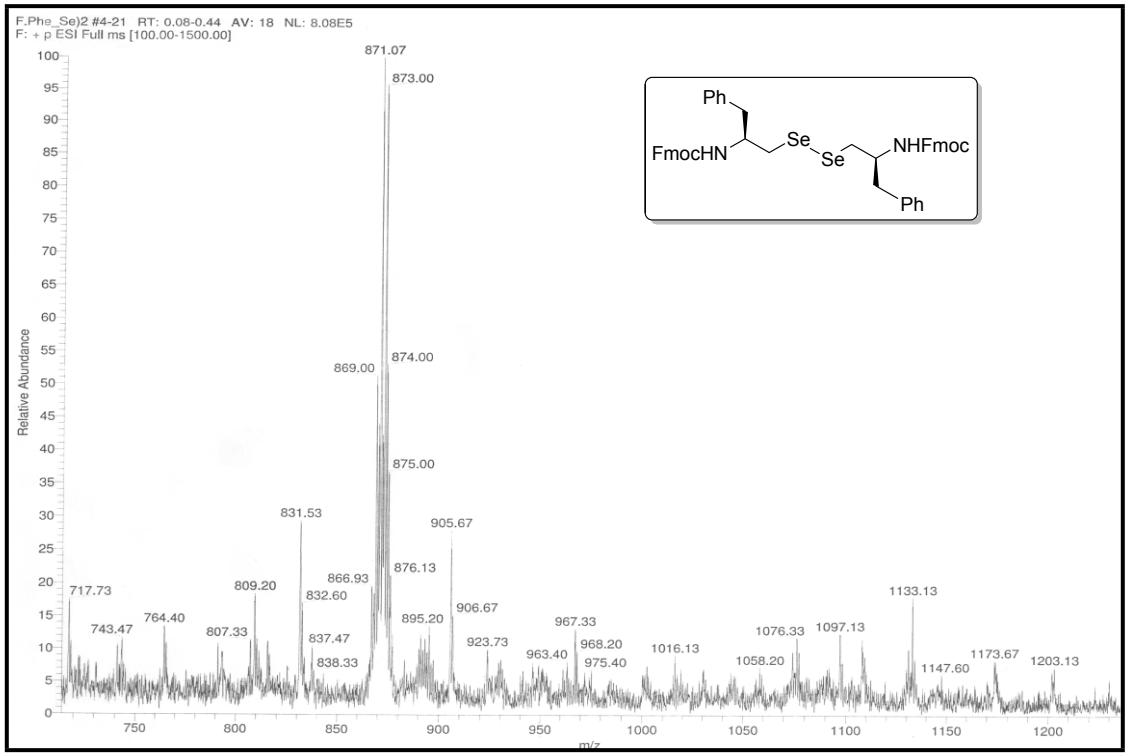
^{1}H NMR of Cbz-Phe-CH_2-Se-)$_2$, 2g



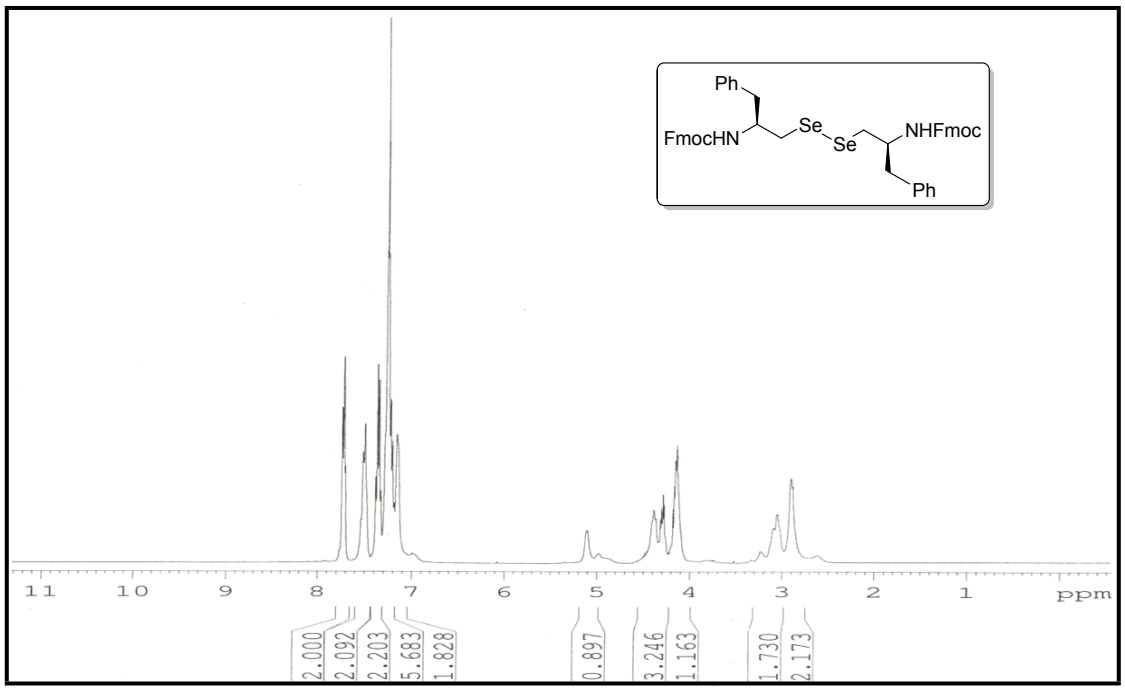
^{13}C NMR of Cbz-Phe-CH_2-Se-)$_2$, 2g



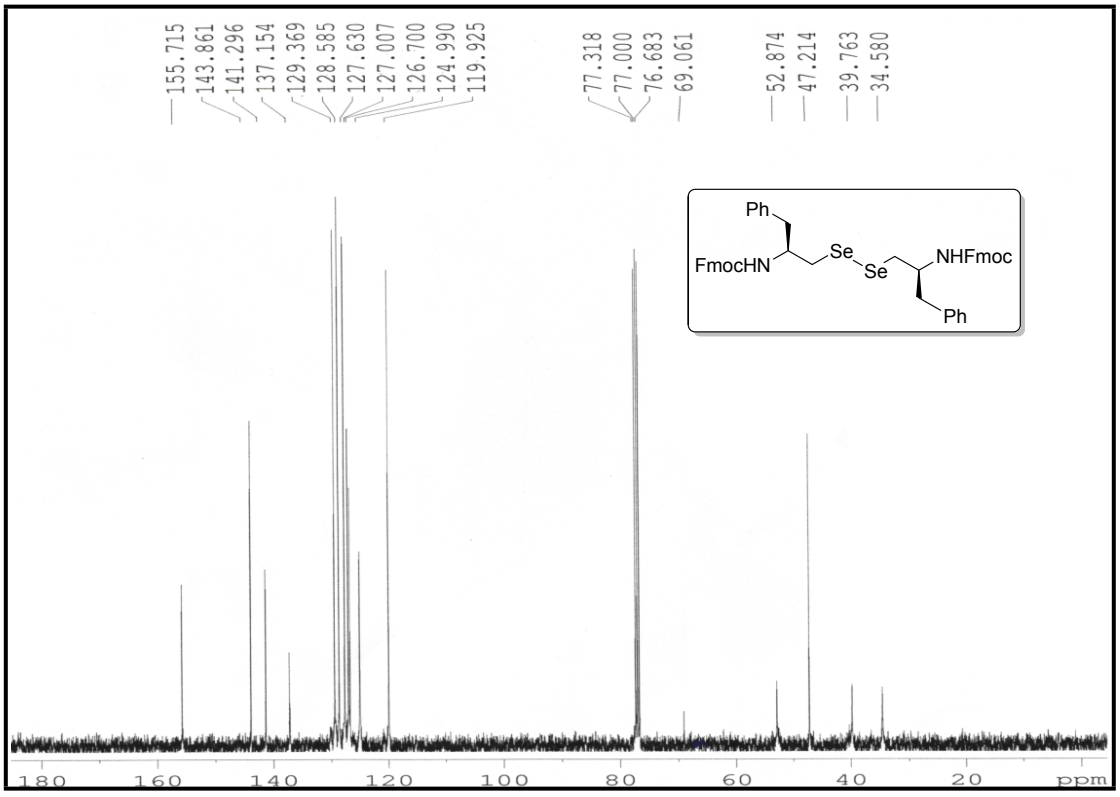
^{77}Se NMR of Cbz-Phe-CH_2-Se-)$_2$, 2g



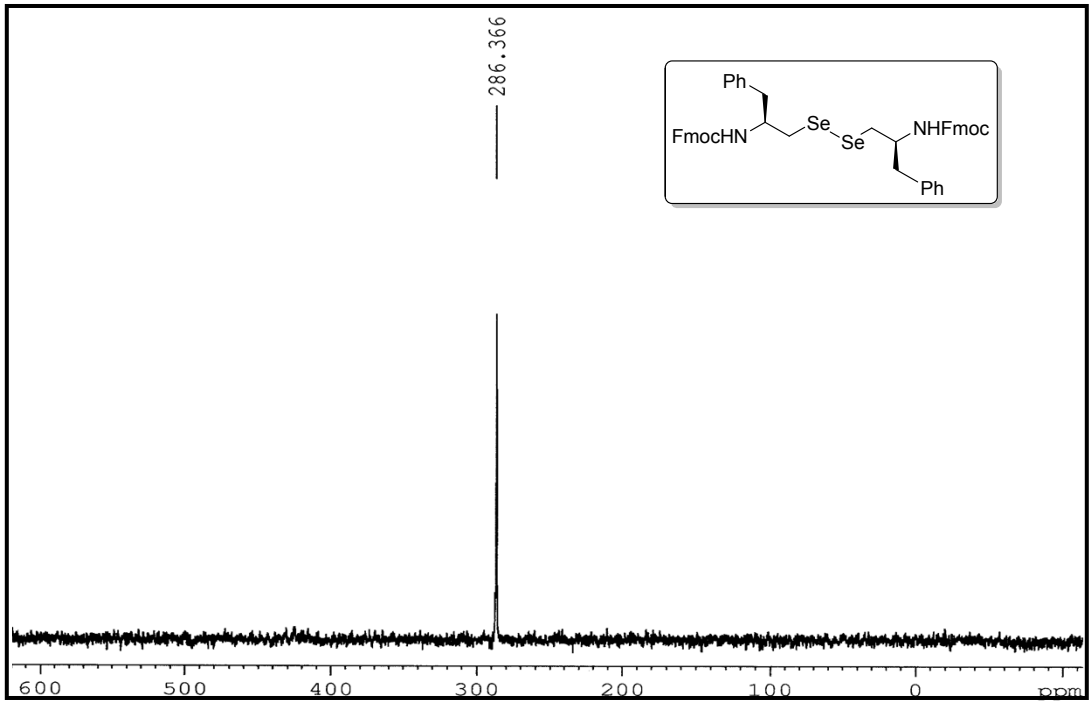
ESI-MS of Fmoc-Phe-CH_2-Se-)$_2$, 2h



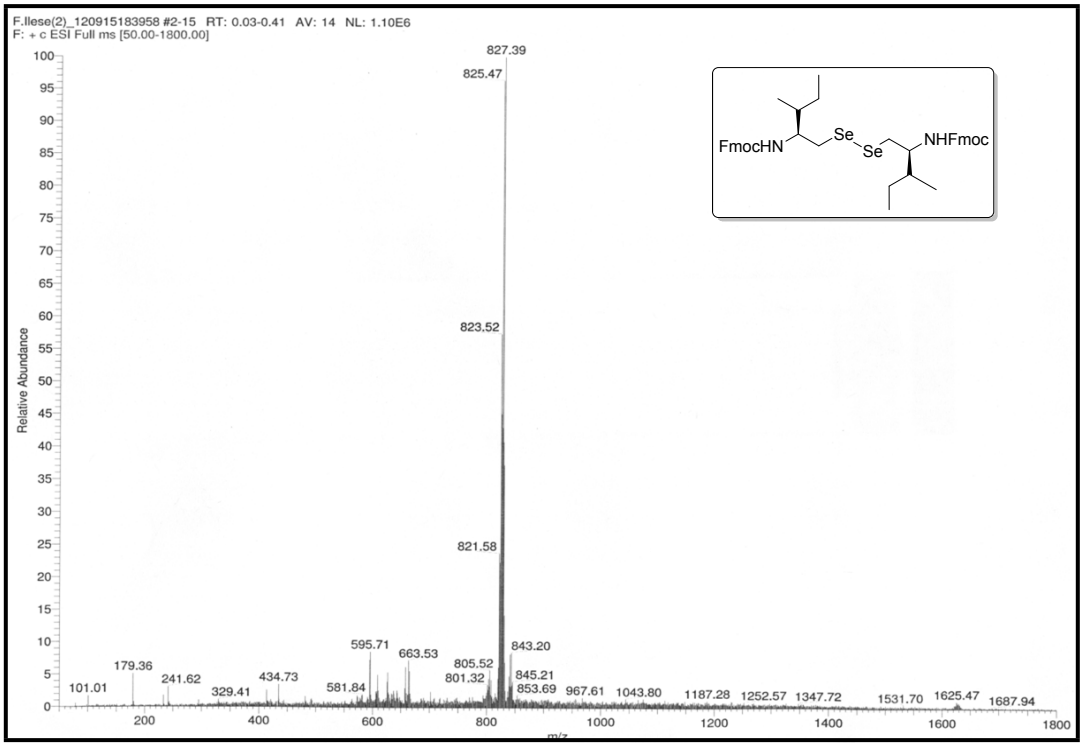
^{1}H NMR of Fmoc-Phe-CH_2-Se-)$_2$, 2h



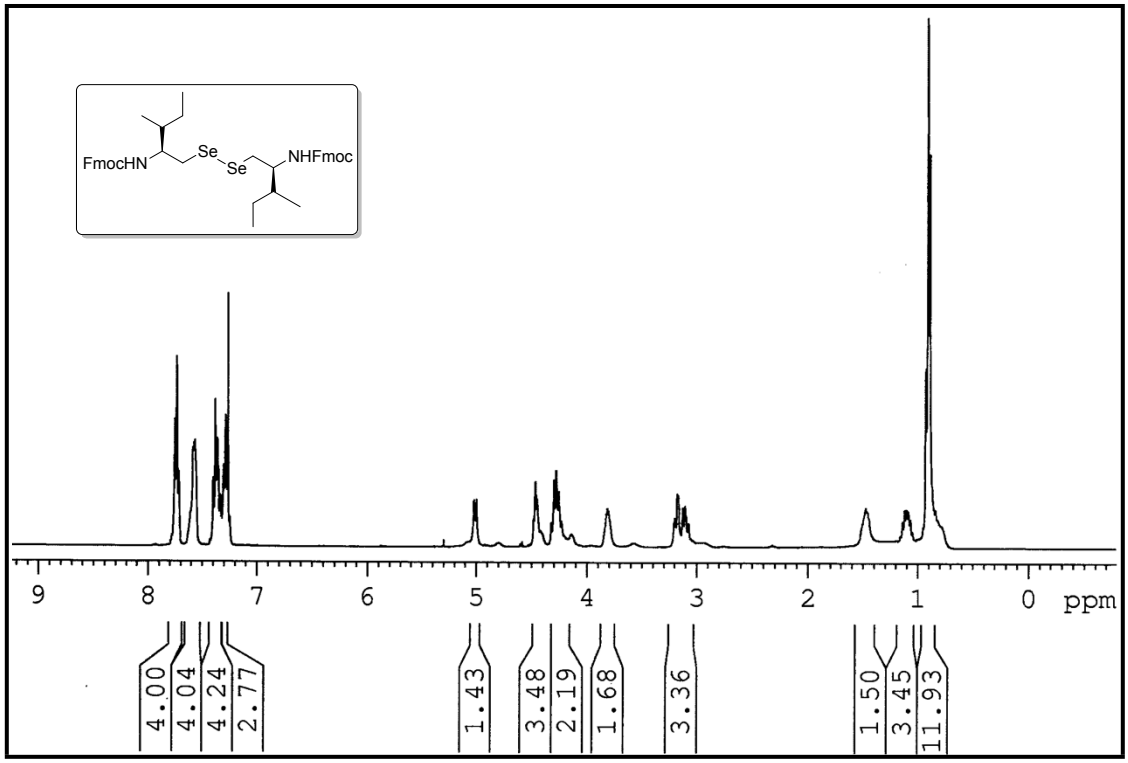
^{13}C NMR of Fmoc-Phe-CH_2-Se-)$_2$, 2h



^{77}Se NMR of Fmoc-Phe-CH_2-Se-)$_2$, 2h



ESI-MS of Fmoc-Ile-CH_2-Se-)$_2$, 2i



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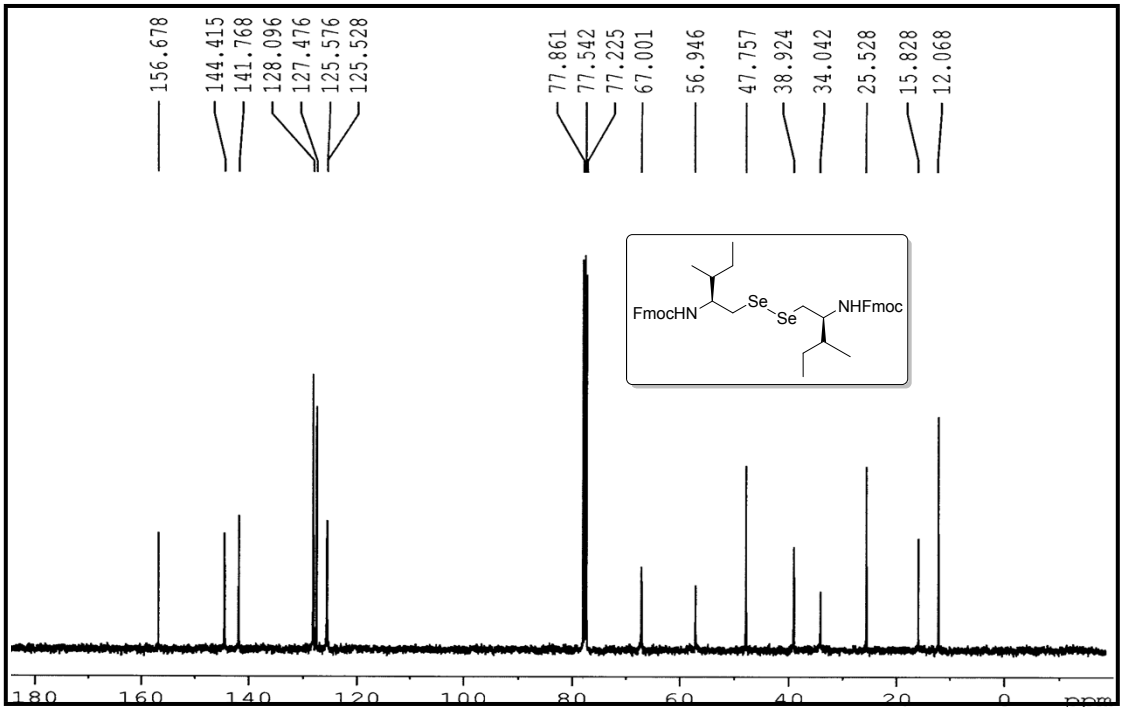
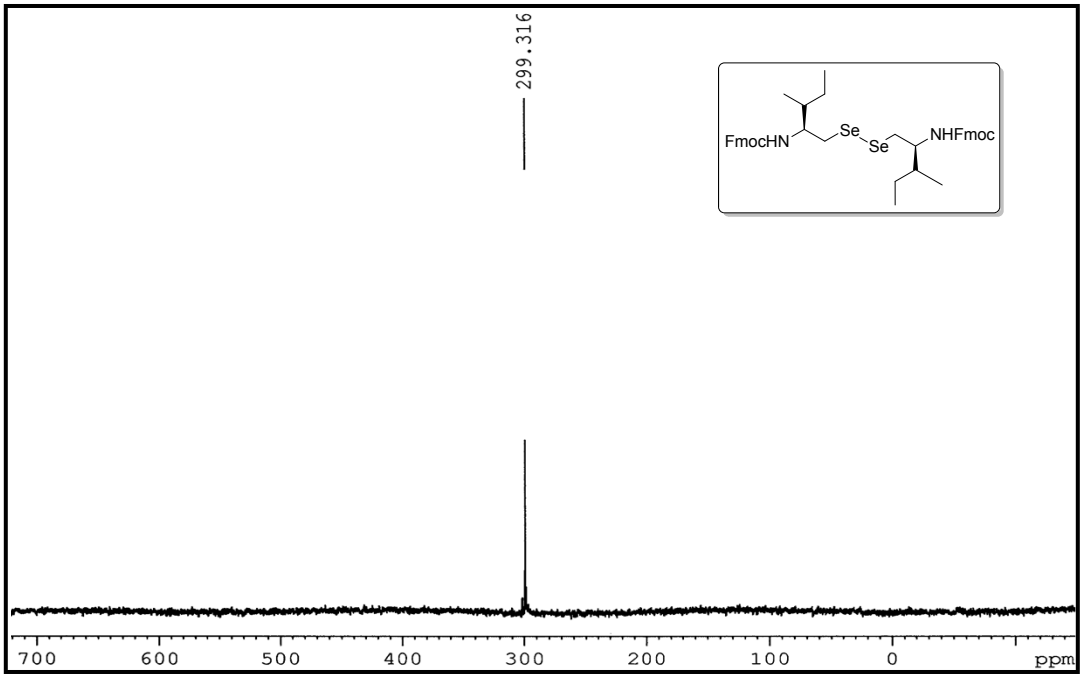
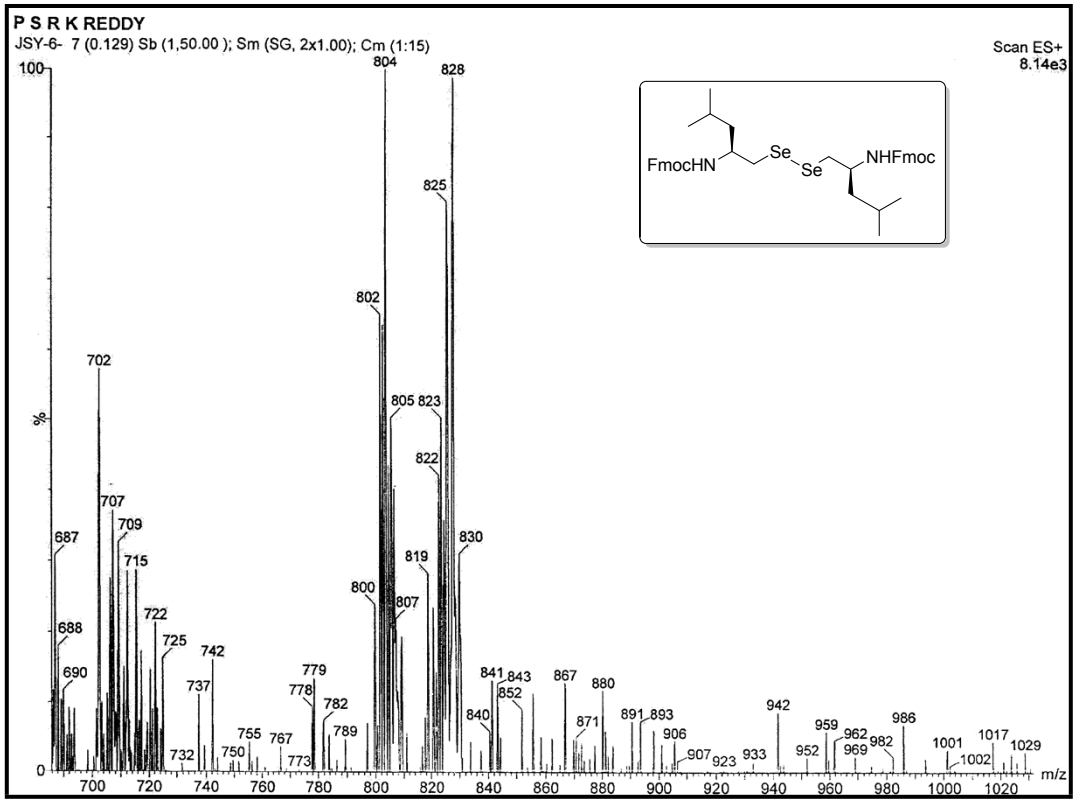


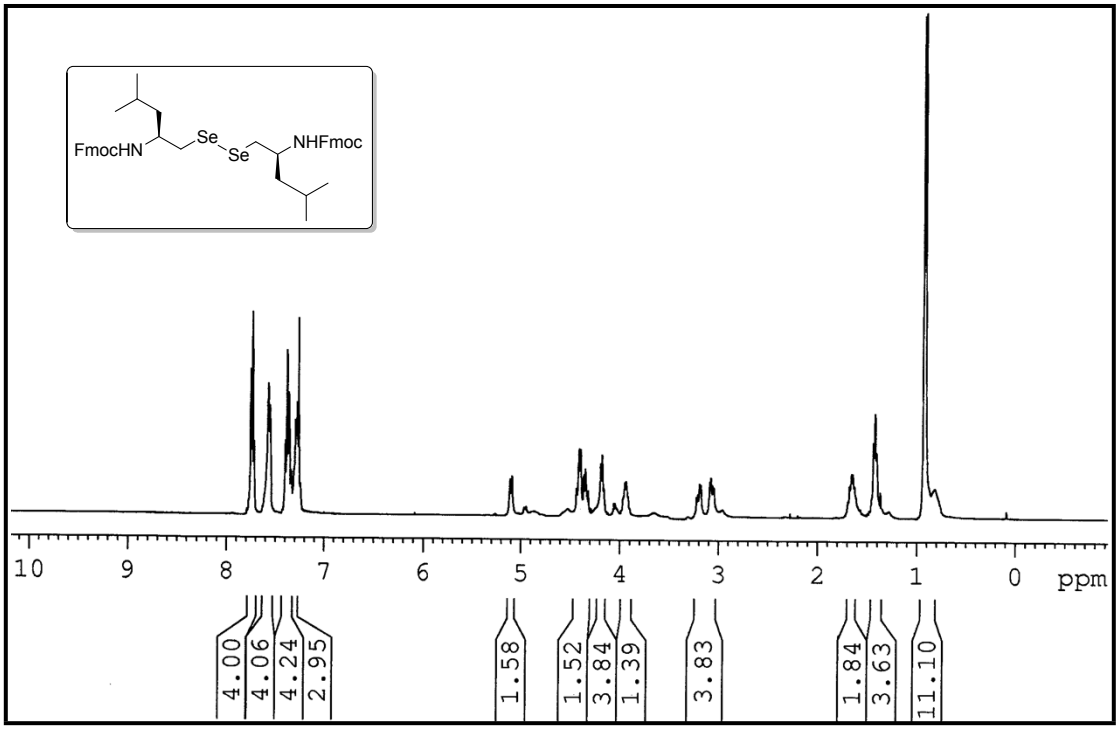
^{13}C NMR of Fmoc-Ile-CH_2-Se-)$_2$, 2i



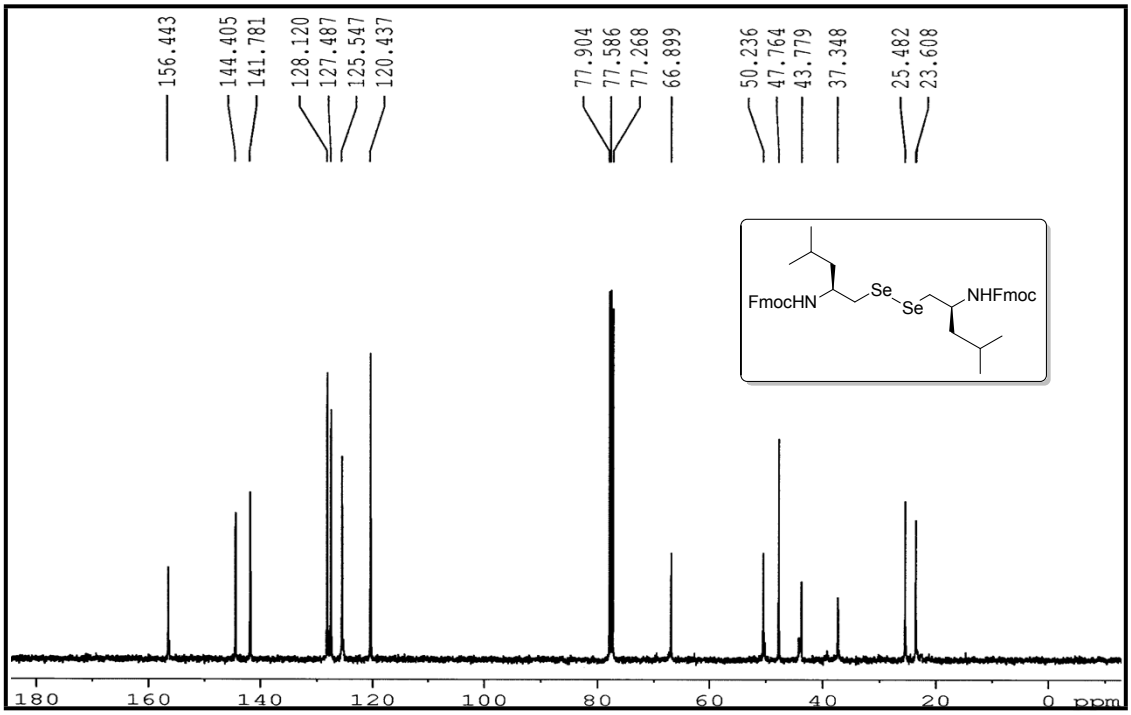
^{77}Se NMR of Fmoc-Ile-CH_2-Se-)$_2$, 2i



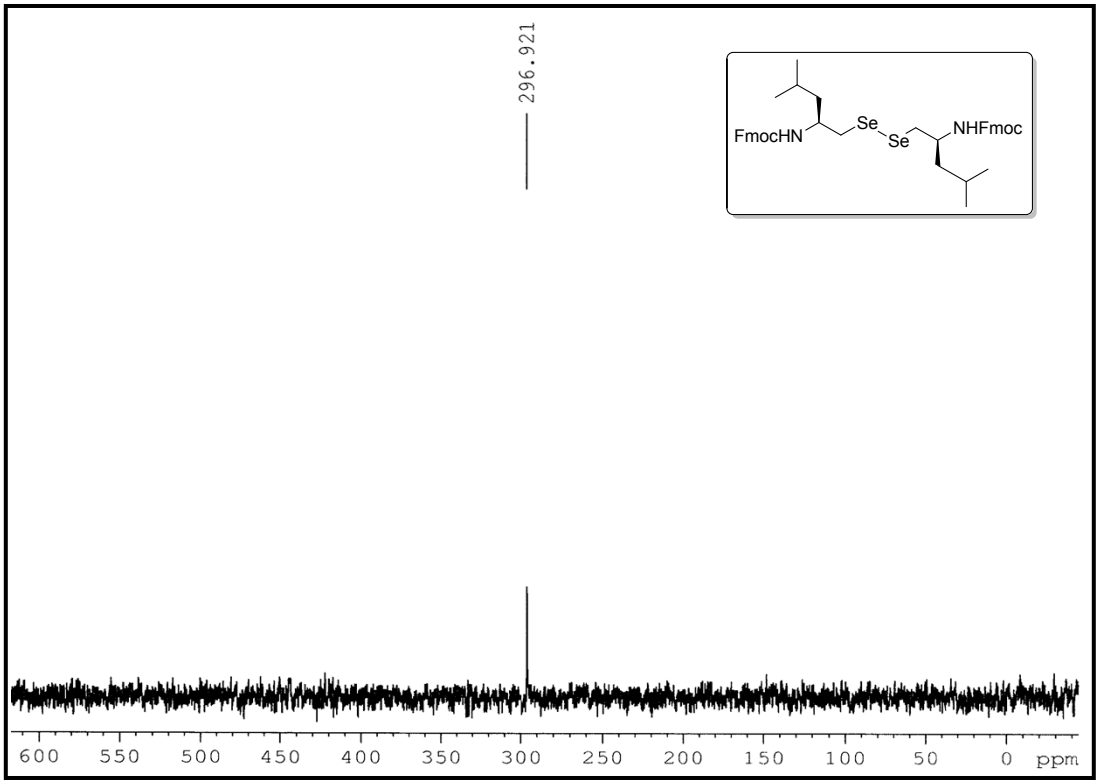
ESI-MS of Fmoc-Leu-CH_2-Se-)$_2$, 2j



^{1}H NMR of Fmoc-Leu-CH_2-Se-)$_2$, 2j



^{13}C NMR of Fmoc-Leu-CH_2-Se-)$_2$, 2j



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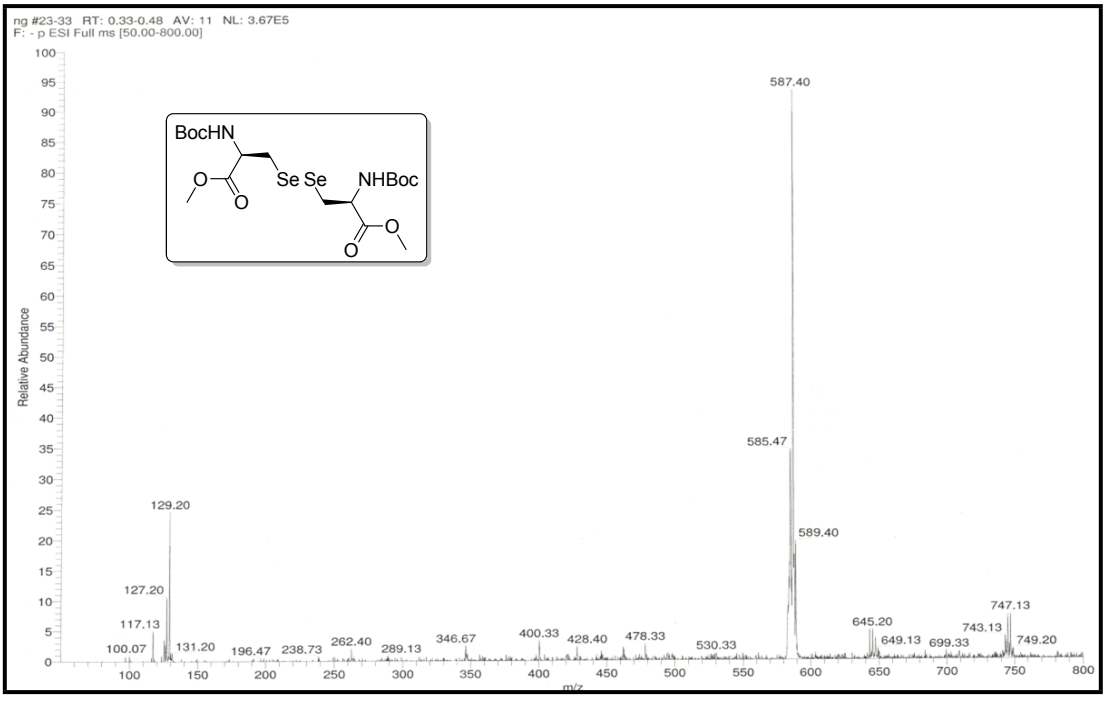
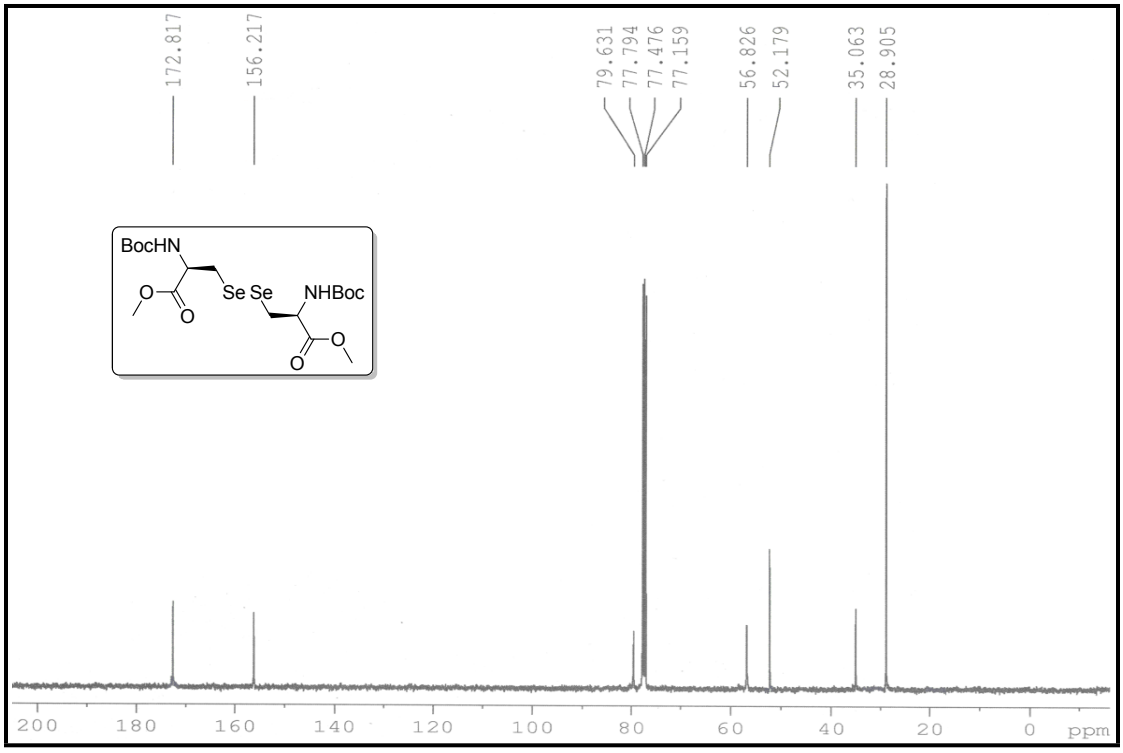
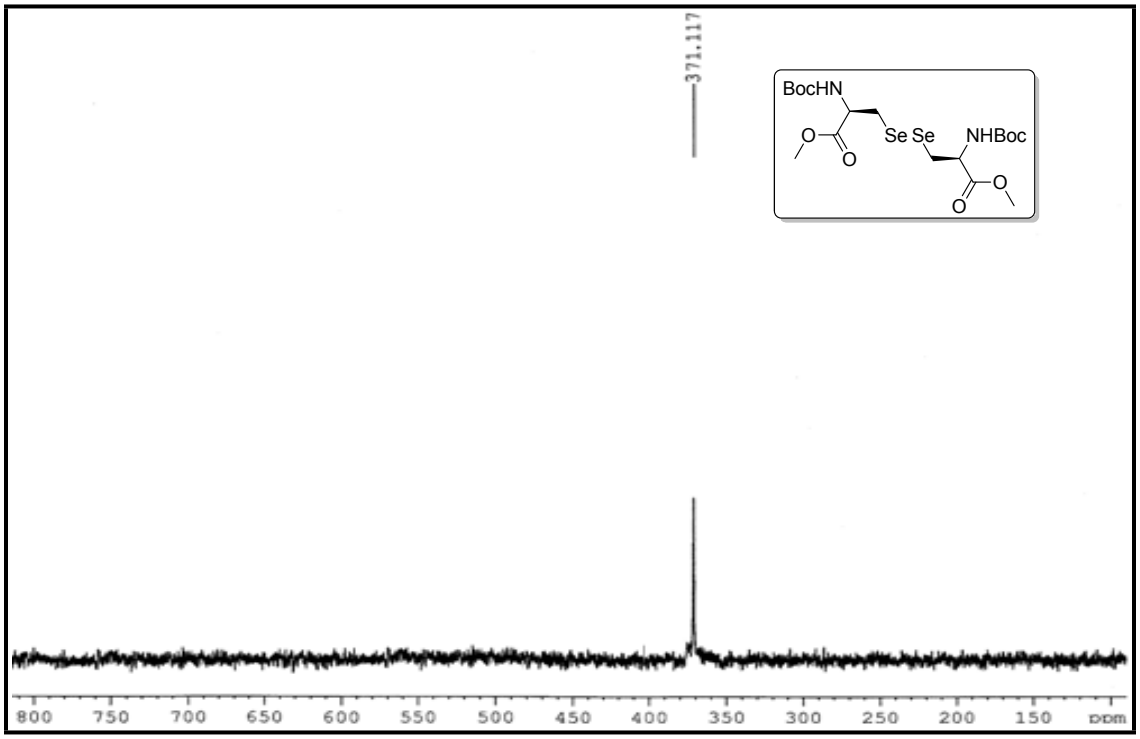


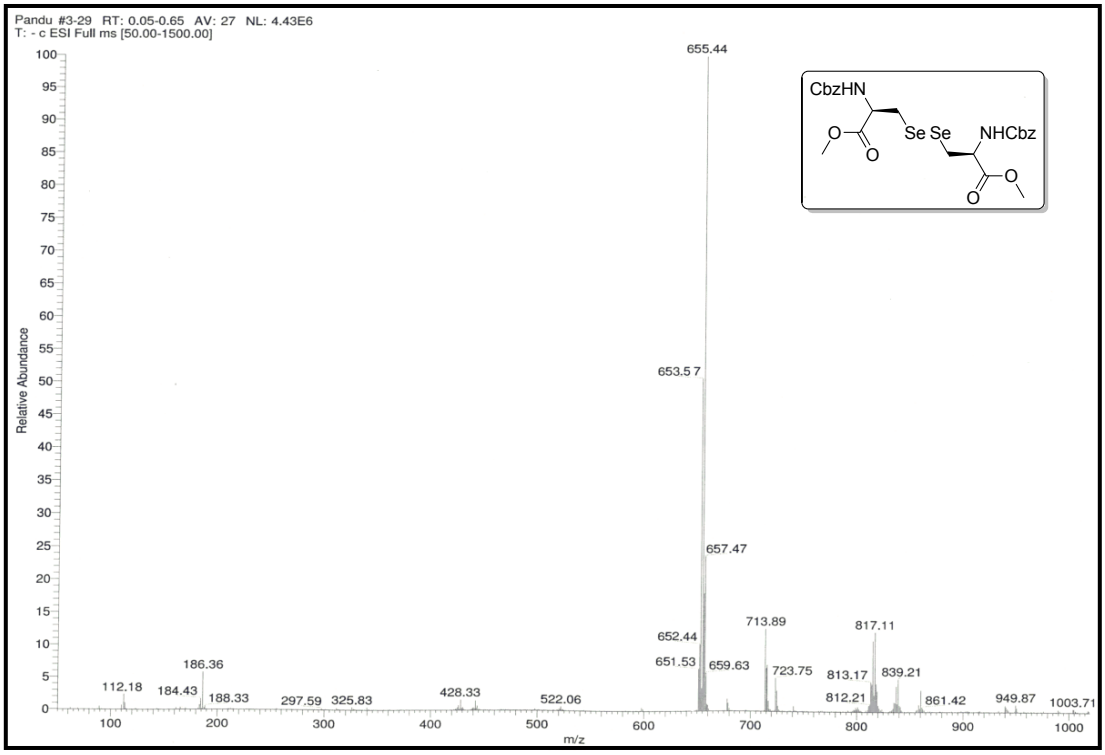
ESI-MS of Boc-L-Sec-OMe, 5a



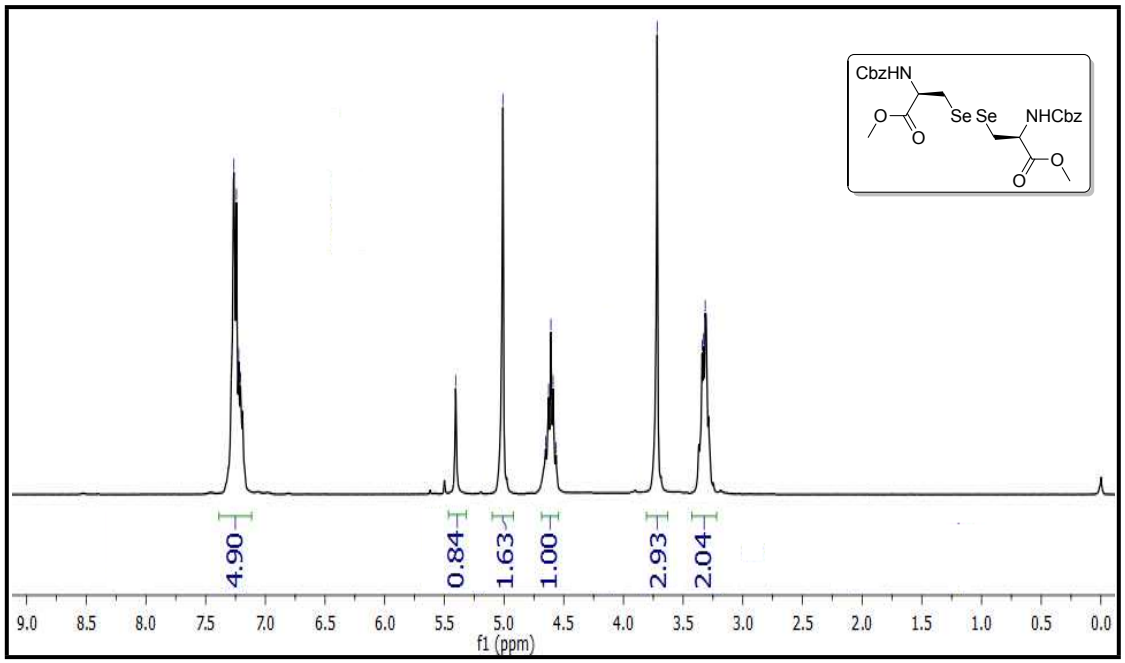
^{13}C NMR of Boc-L-Sec-OMe, 5a



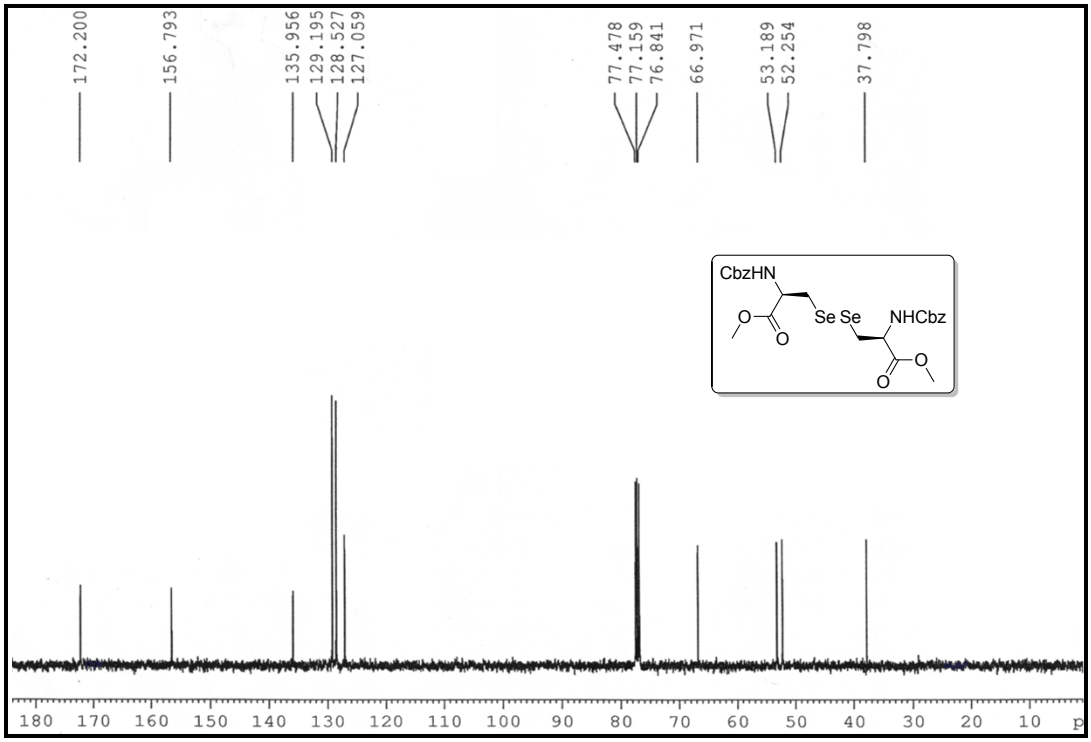
^{77}Se NMR of Boc-L-Sec-OMe, 5a



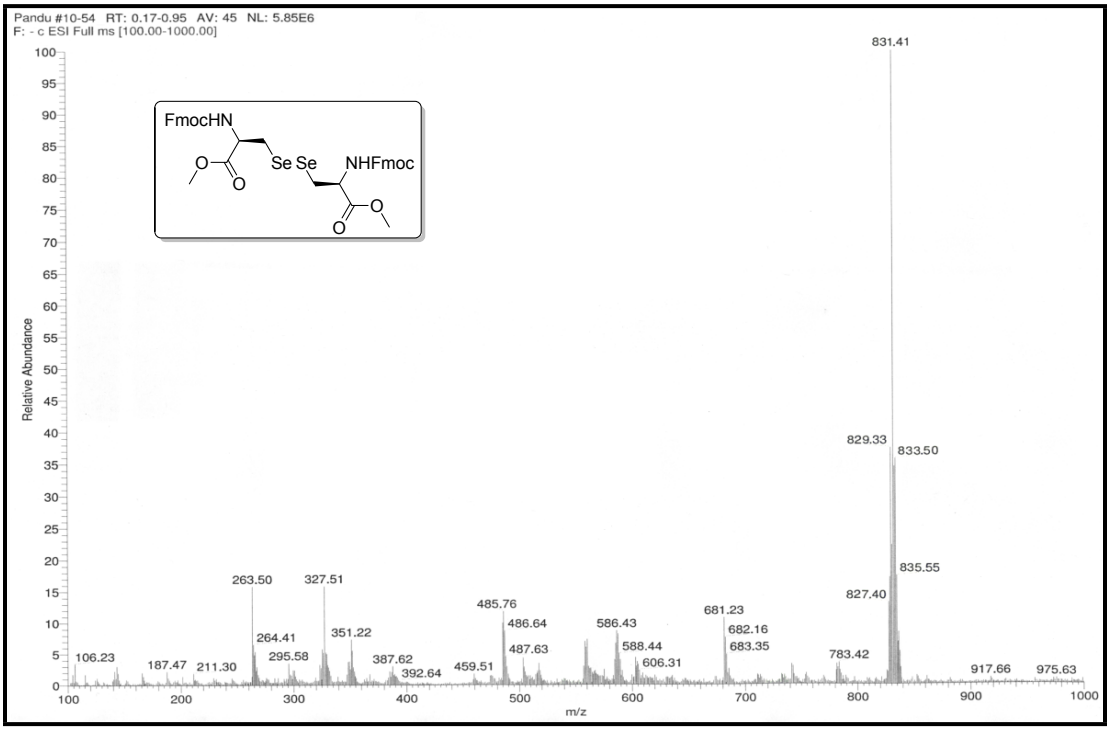
ESI-MS of Cbz-L-Sec-OMe, 5b



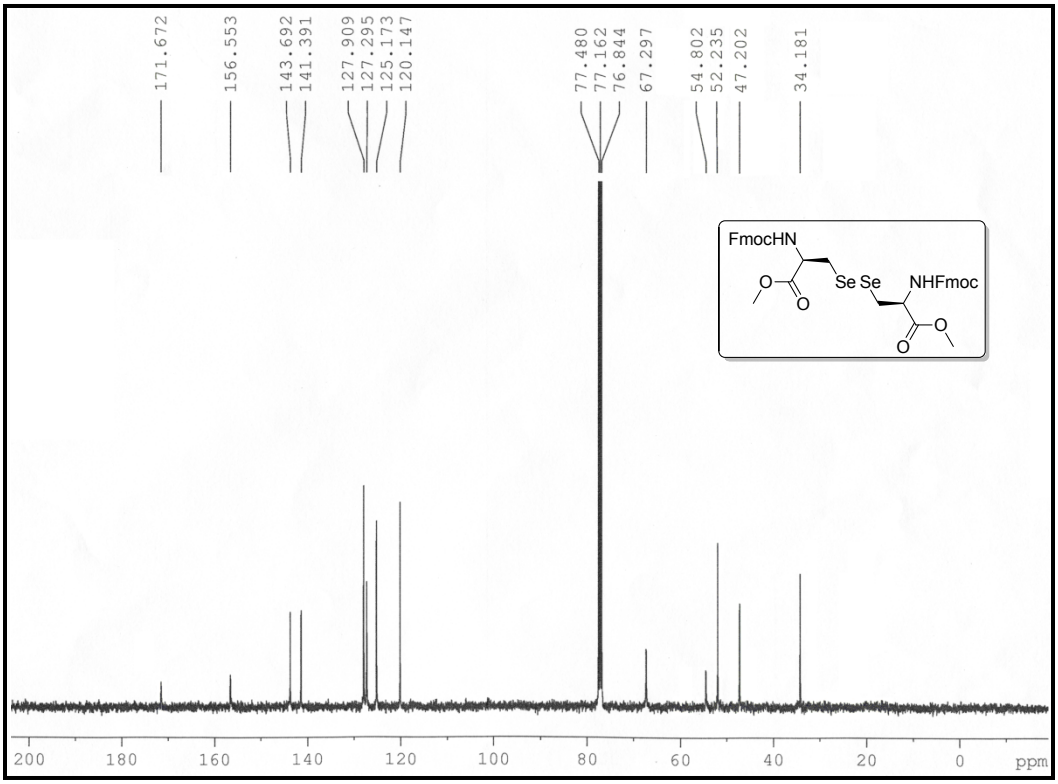
^{1}H NMR of Cbz-L-Sec-OMe, 5b



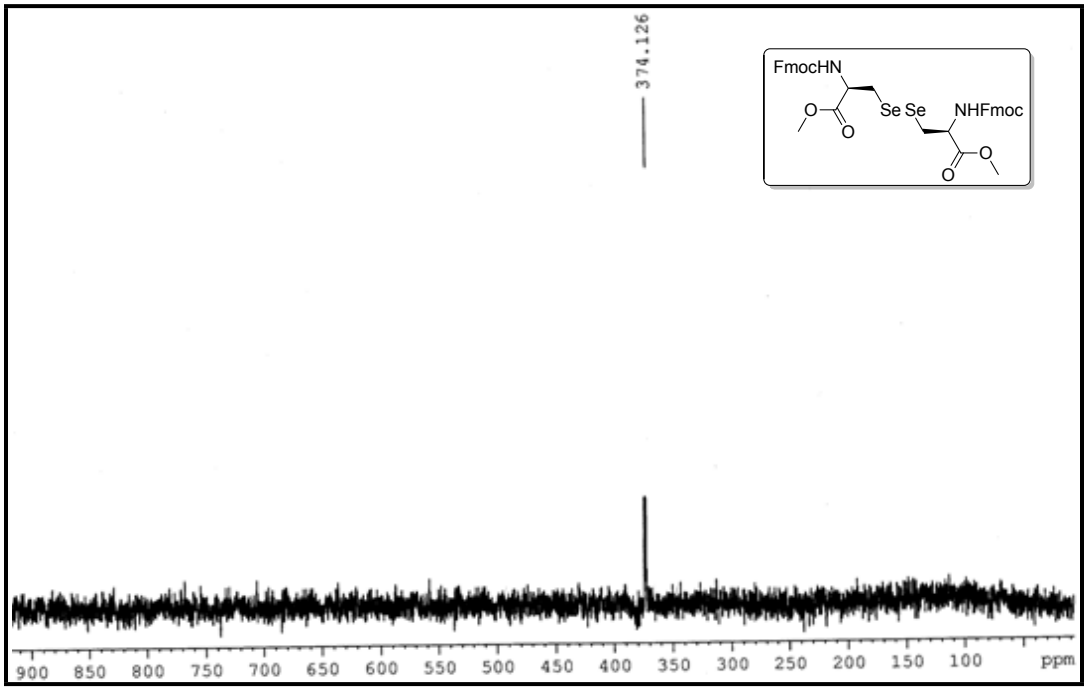
^{13}C NMR of Cbz-L-Sec-OMe, 5b



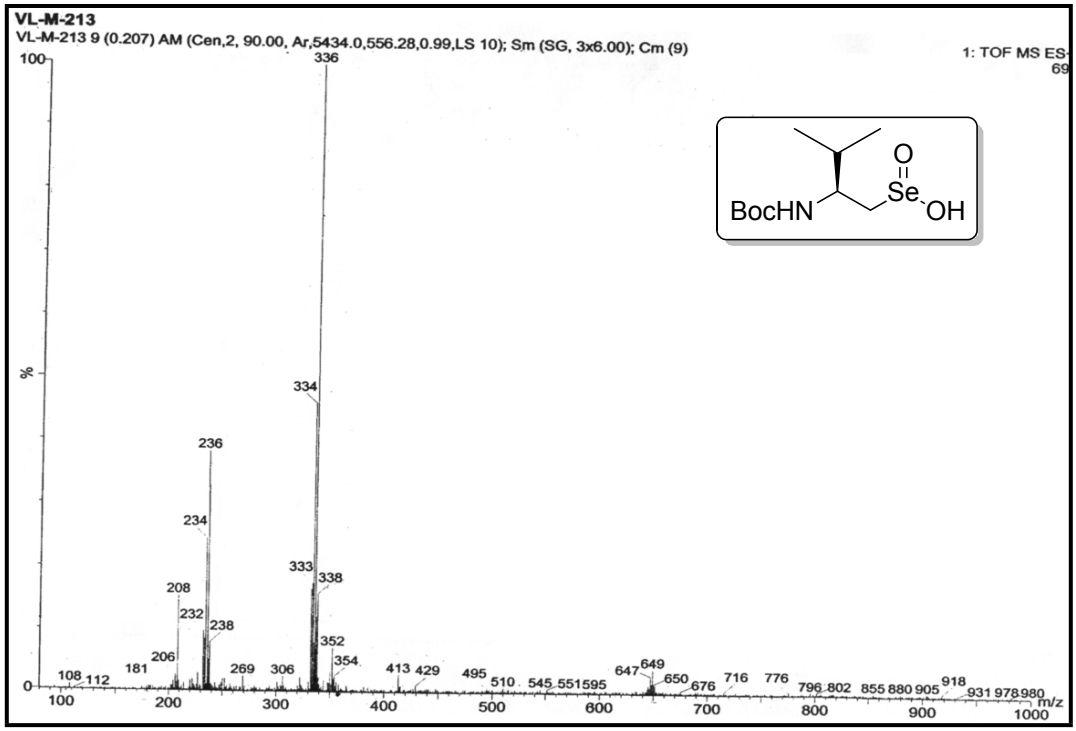
ESI-MS of Fmoc-L-Sec-OMe, 5c



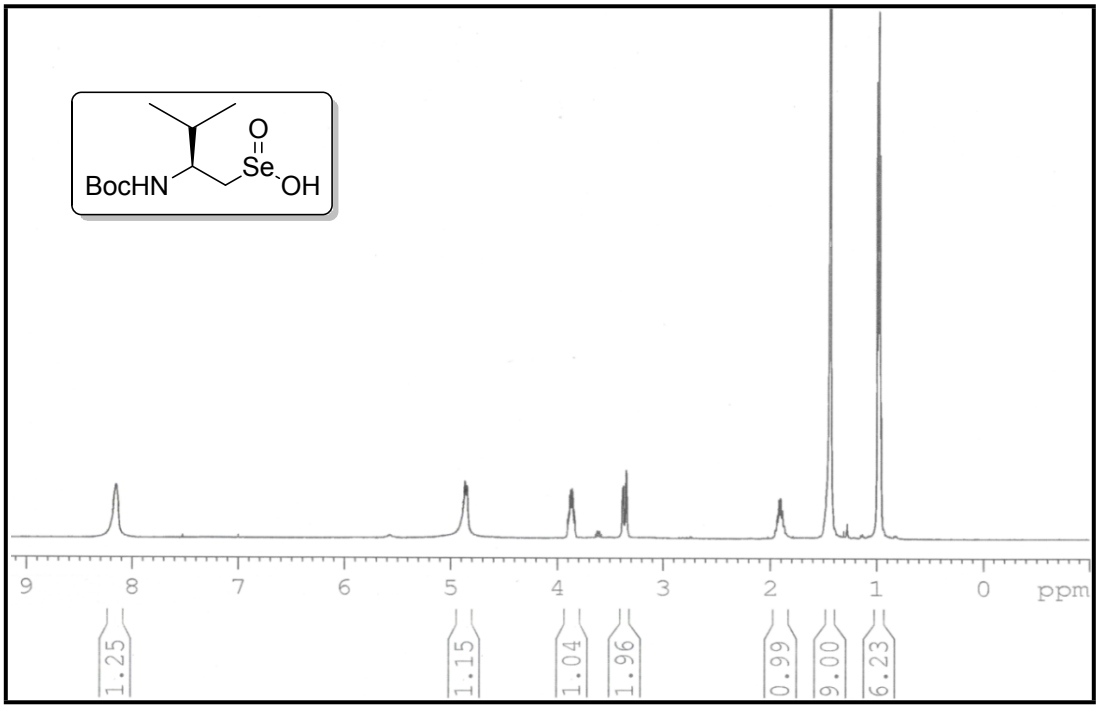
^{13}C NMR of Fmoc-L-Sec-OMe, 5c



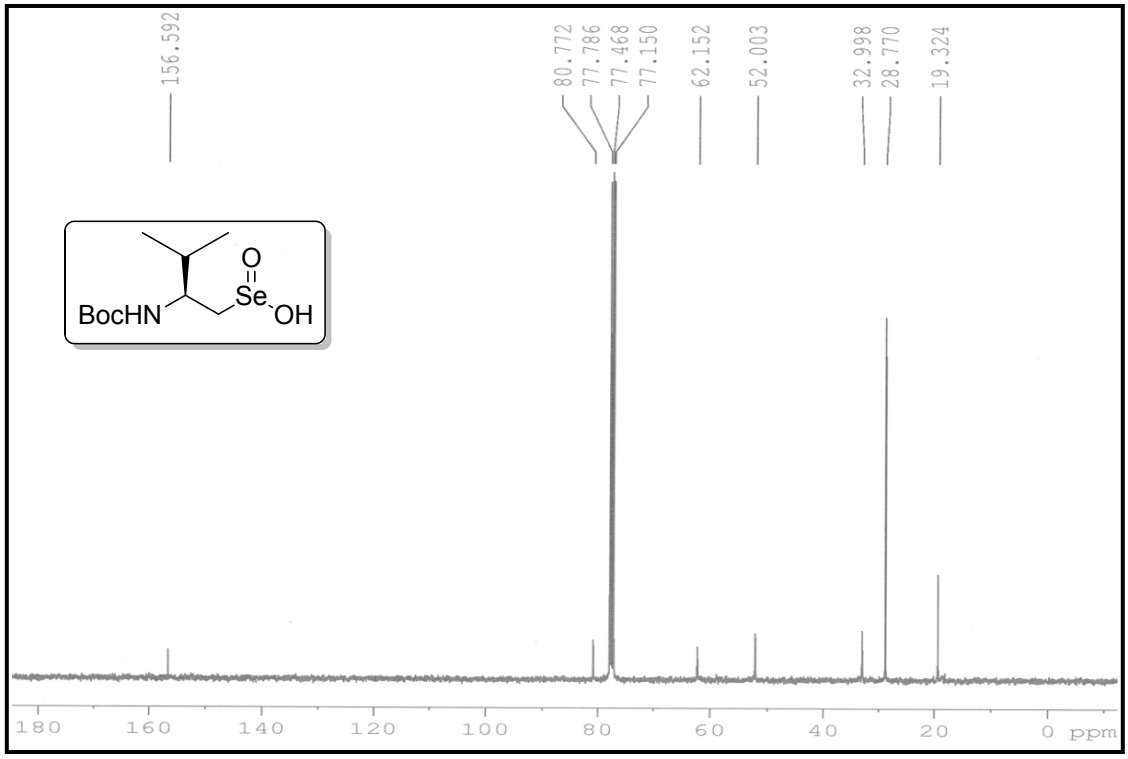
^{77}Se NMR of Fmoc-L-Sec-OMe, 5c



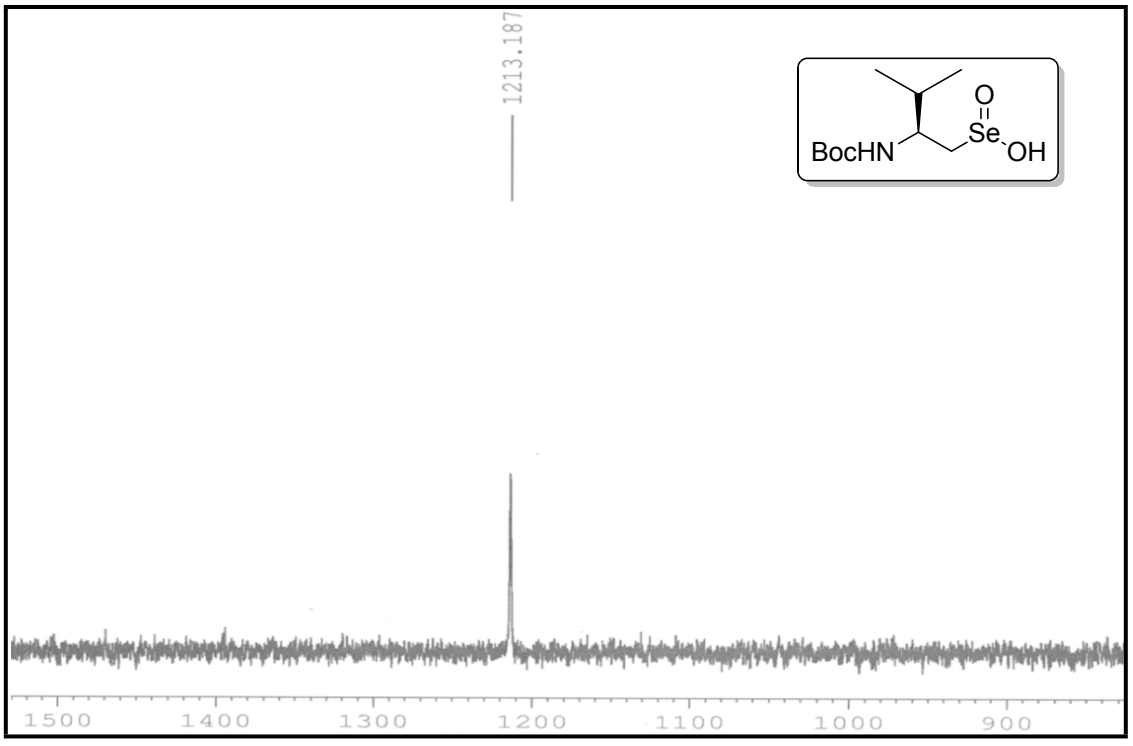
ESI-MS of Boc-Val-CH_2-SeO_2H, 6a



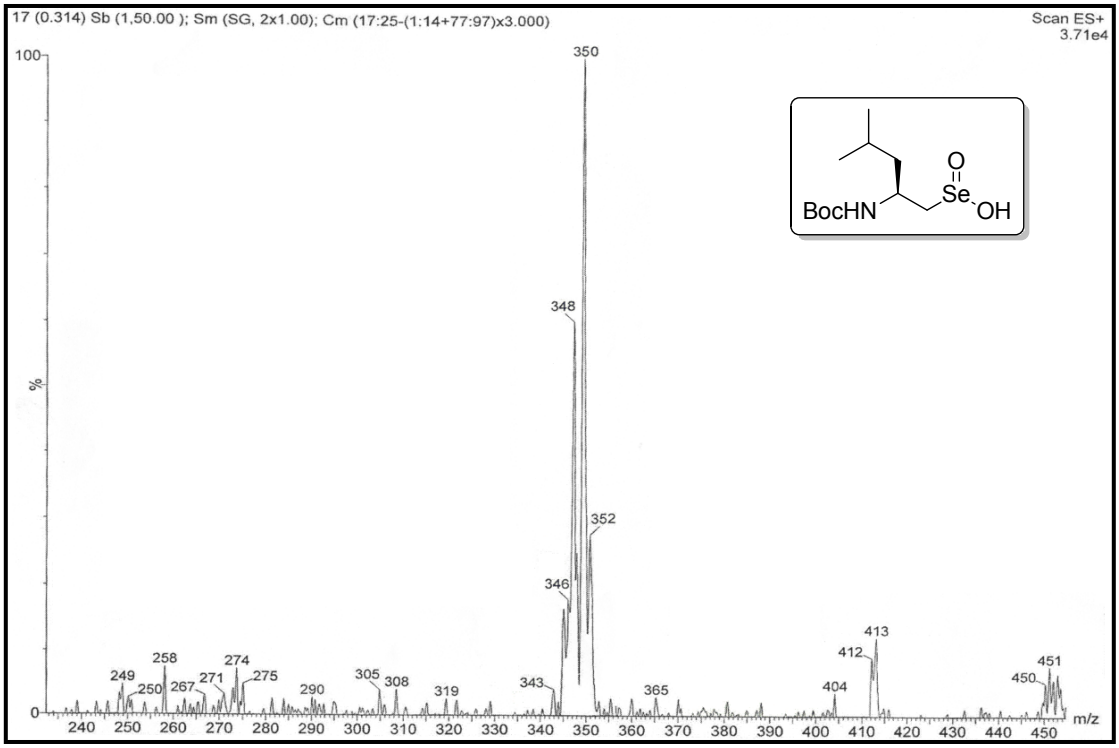
^{1}H NMR of Boc-Val-CH_2-SeO_2H, 6a



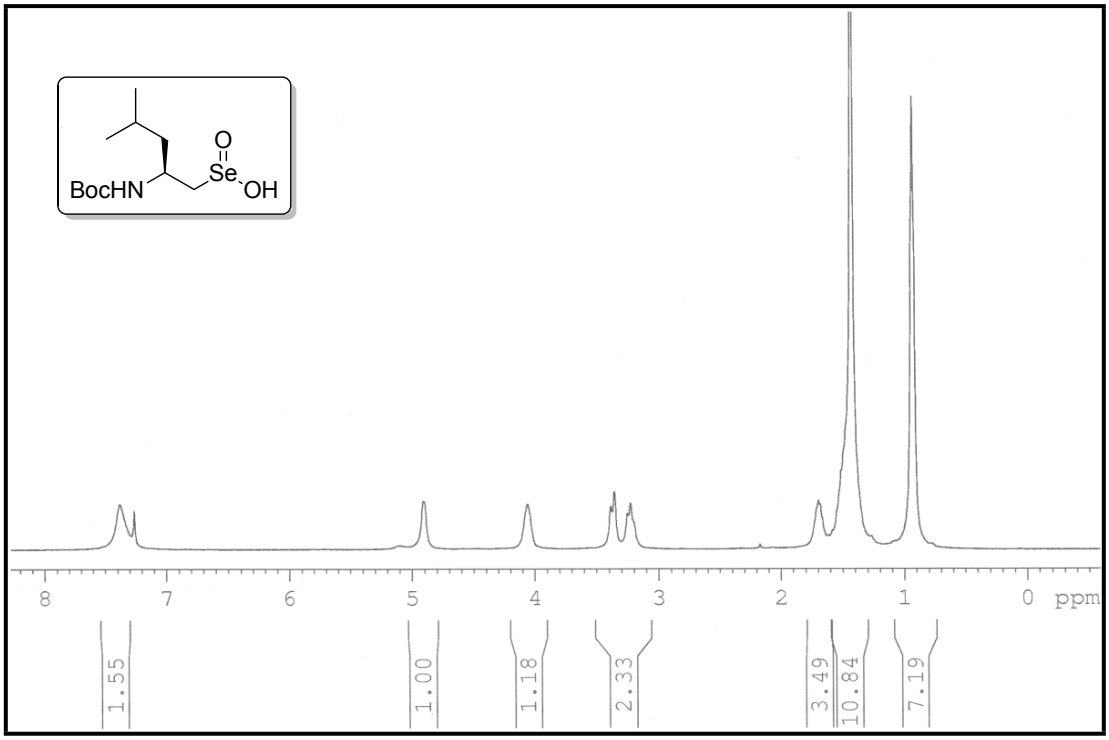
^{13}C NMR of Boc-Val-CH_2-SeO_2H, 6a



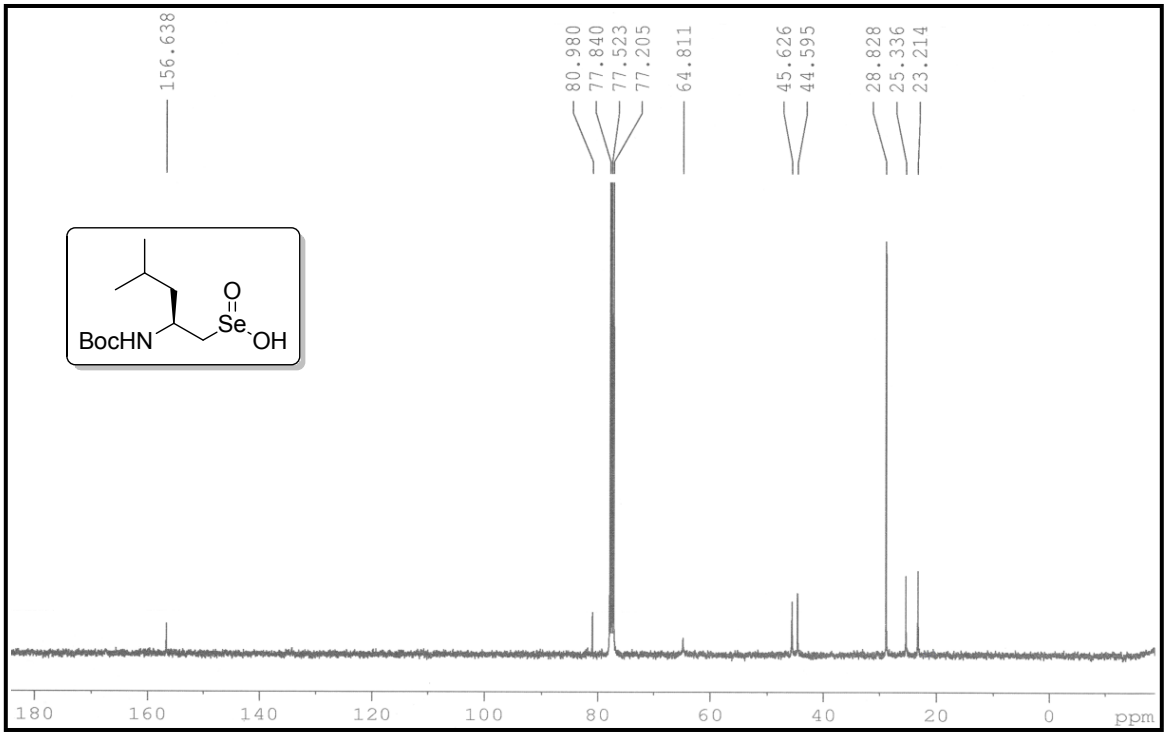
^{77}Se NMR of Boc-Val-CH_2-SeO_2H, 6a



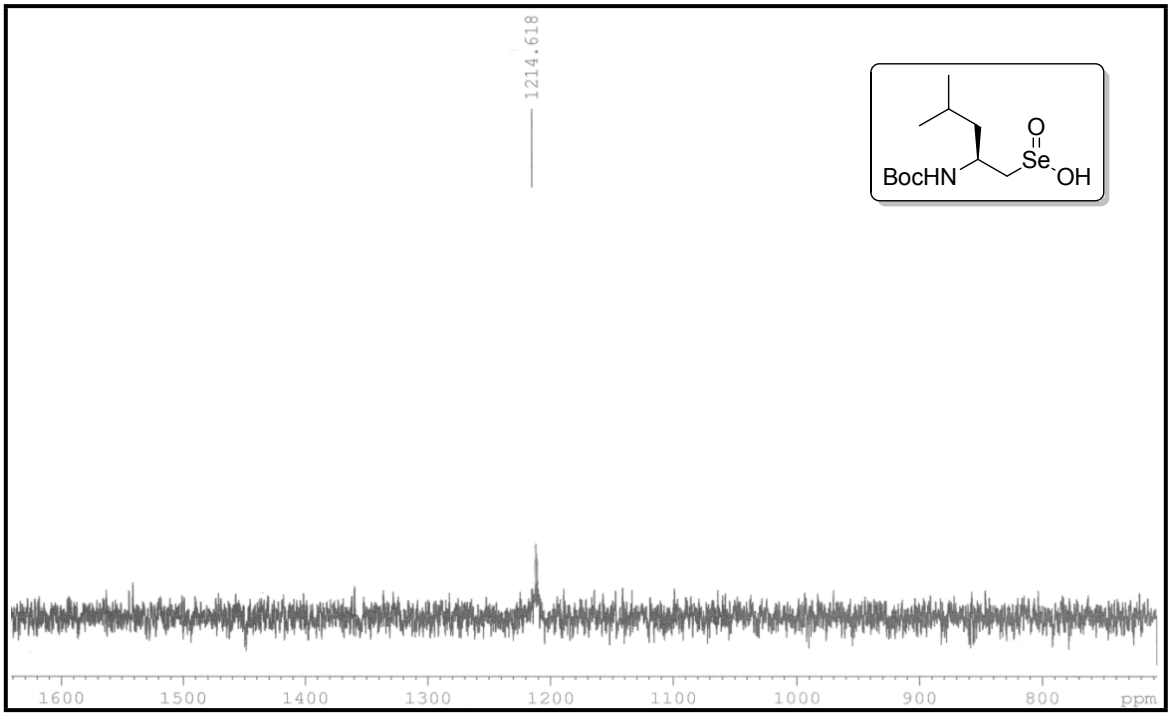
ESI-MS of Boc-Leu-CH_2-SeO_2H, 6b



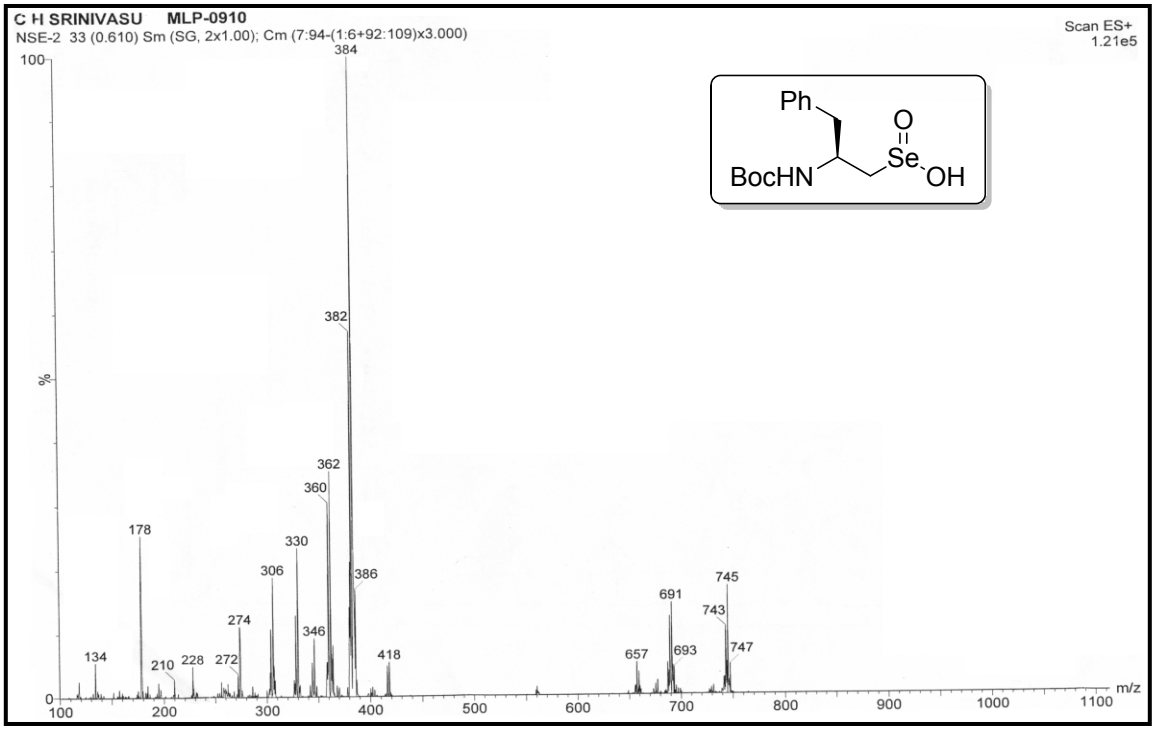
^{1}H NMR of Boc-Leu-CH_2-SeO_2H, 6b



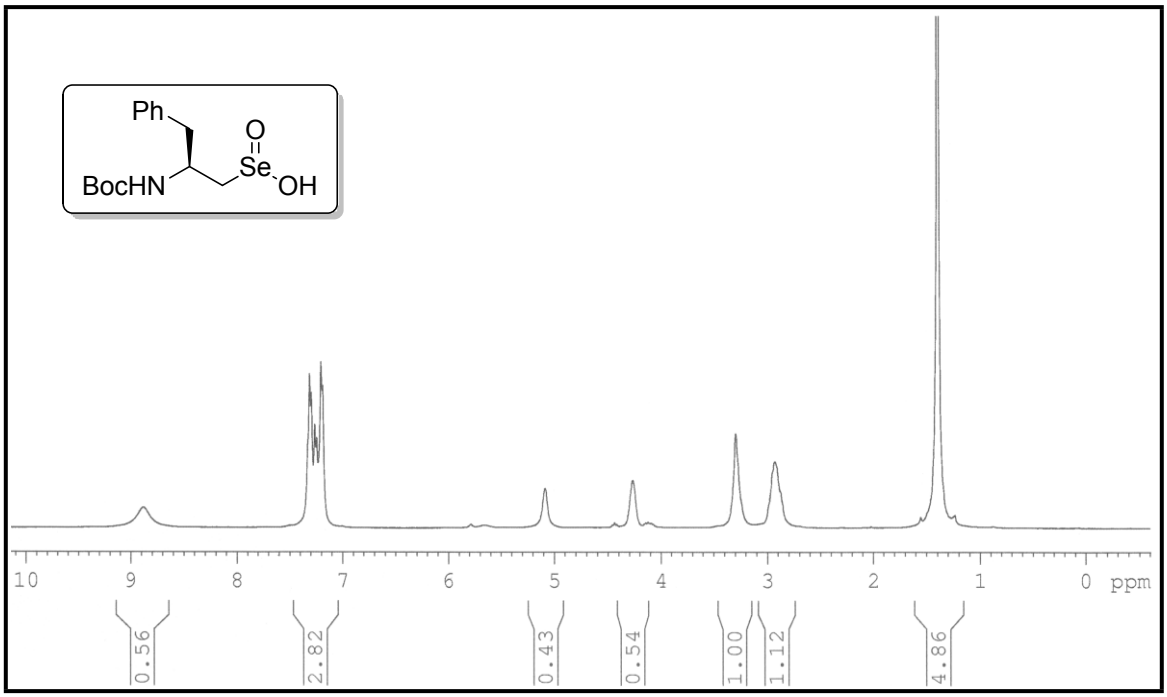
^{13}C NMR of Boc-Leu-CH_2-SeO_2H, 6b



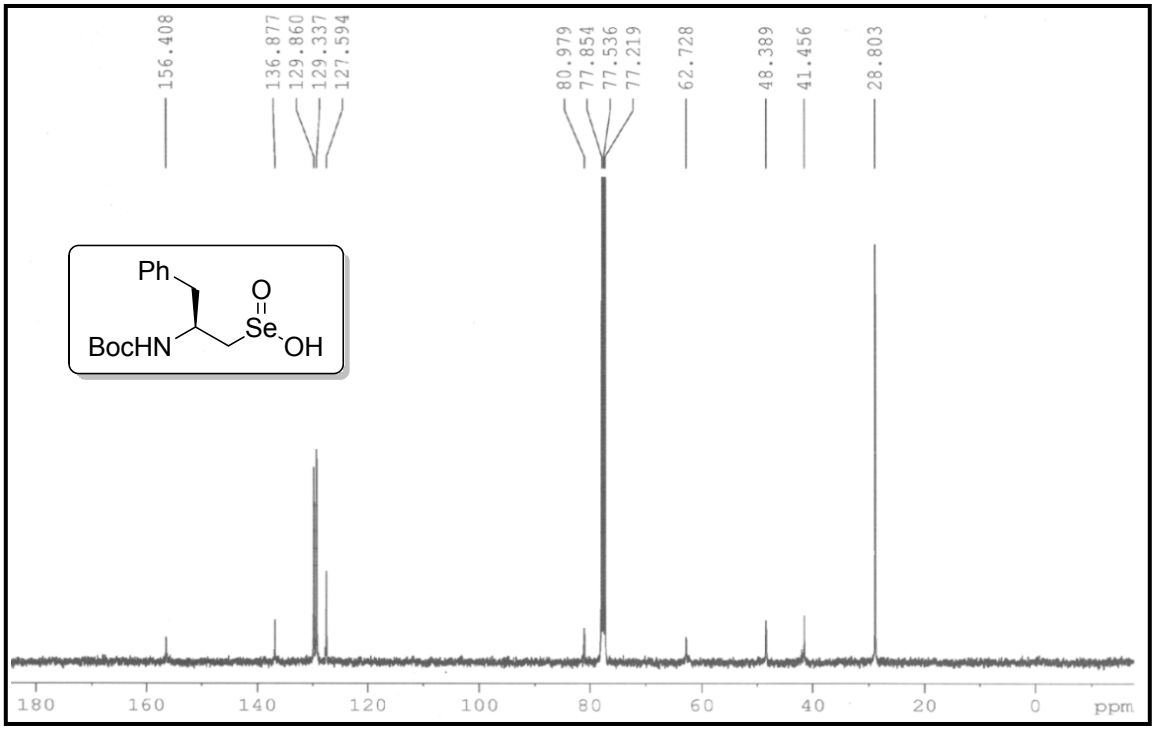
^{77}Se NMR of Boc-Leu-CH_2-SeO_2H, 6b



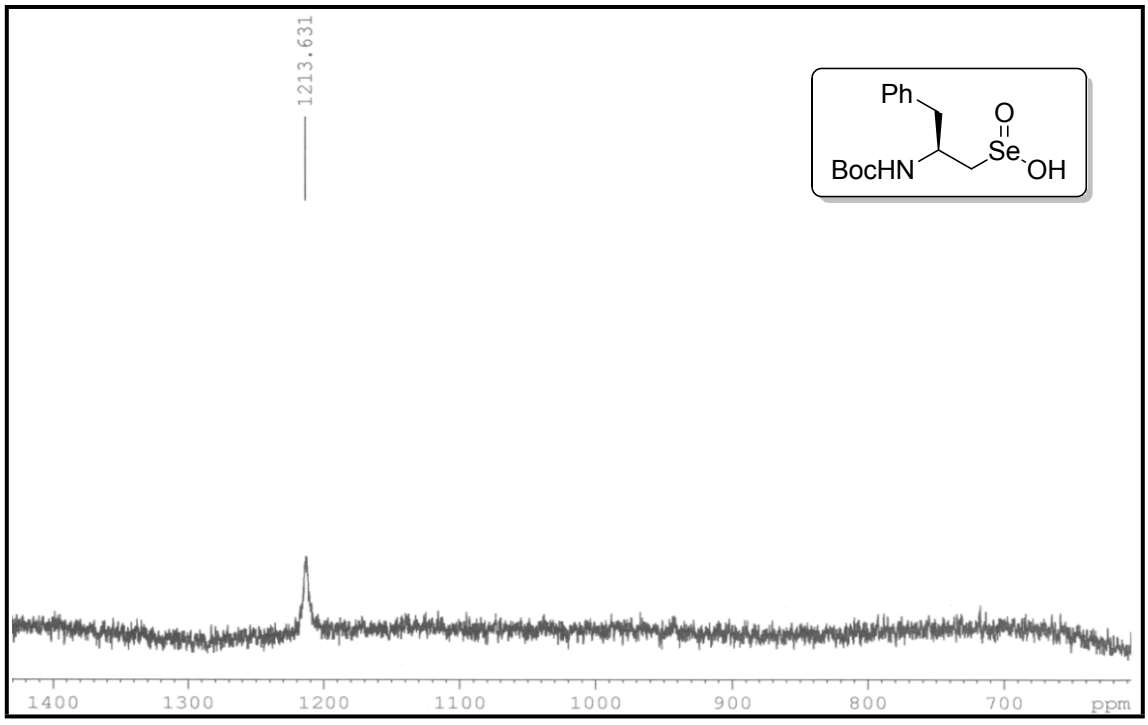
ESI-MS of Boc-Phe-CH_2-SeO_2H, 6c



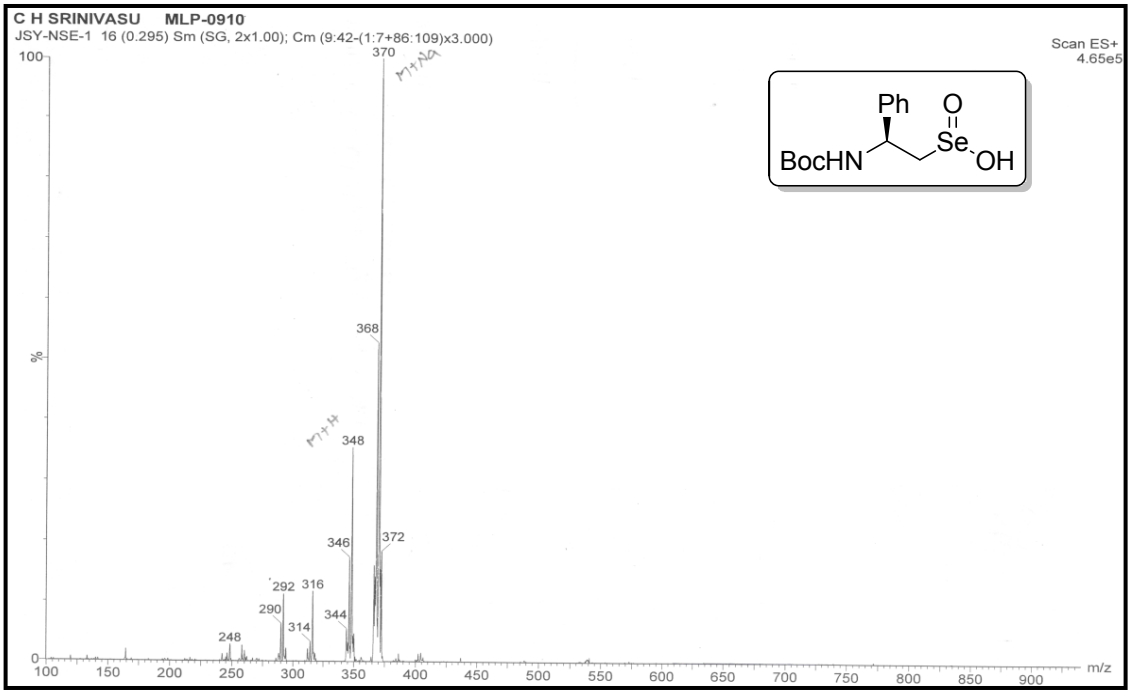
^{1}H NMR of Boc-Phe-CH_2-SeO_2H, 6c



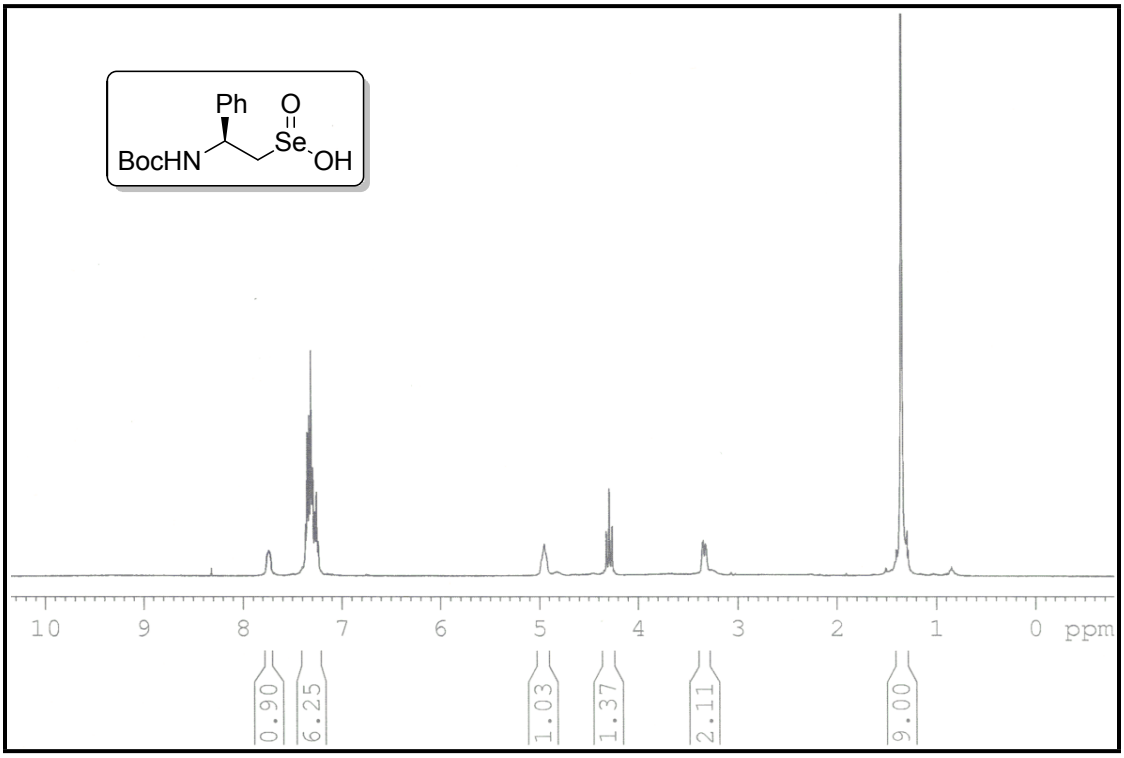
^{13}C NMR of Boc-Phe-CH_2-SeO_2H, 6c



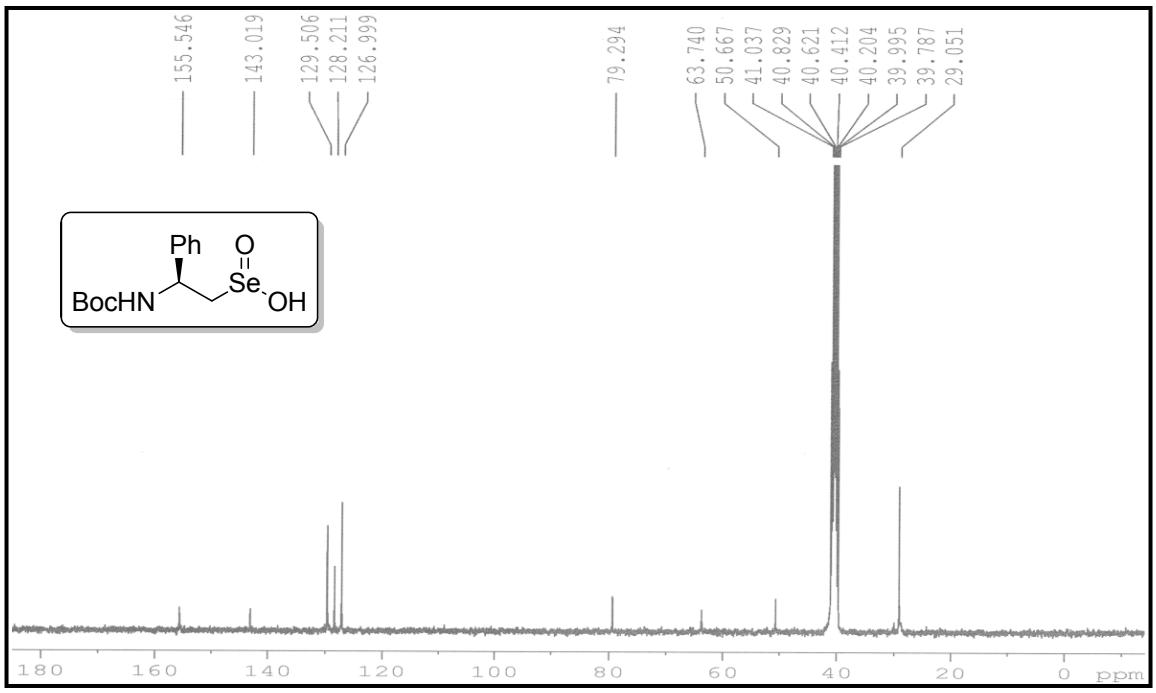
^{77}Se NMR of Boc-Phe-CH_2-SeO_2H, 6c



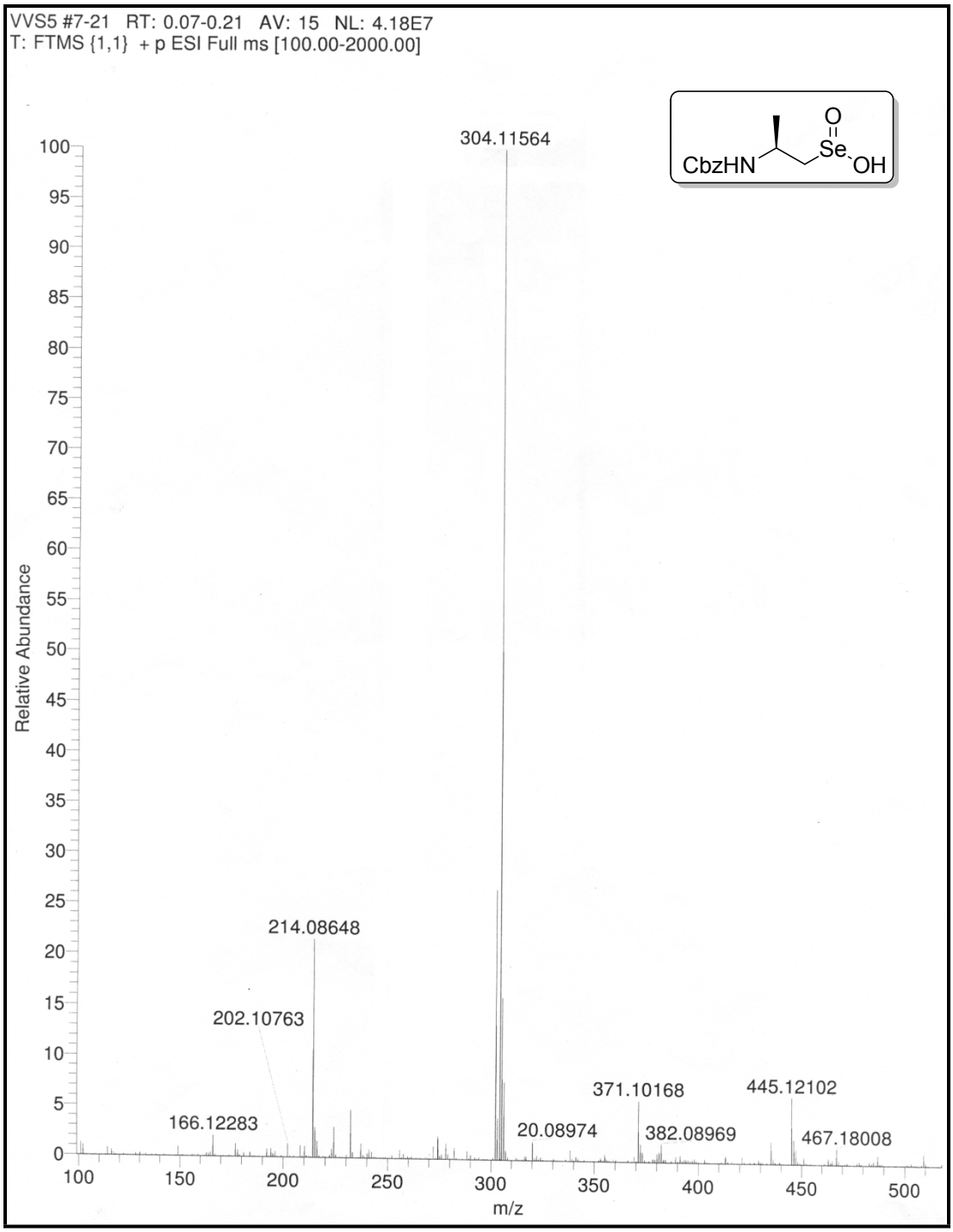
ESI-MS of Boc-Phg-CH_2-SeO_2H, 6d



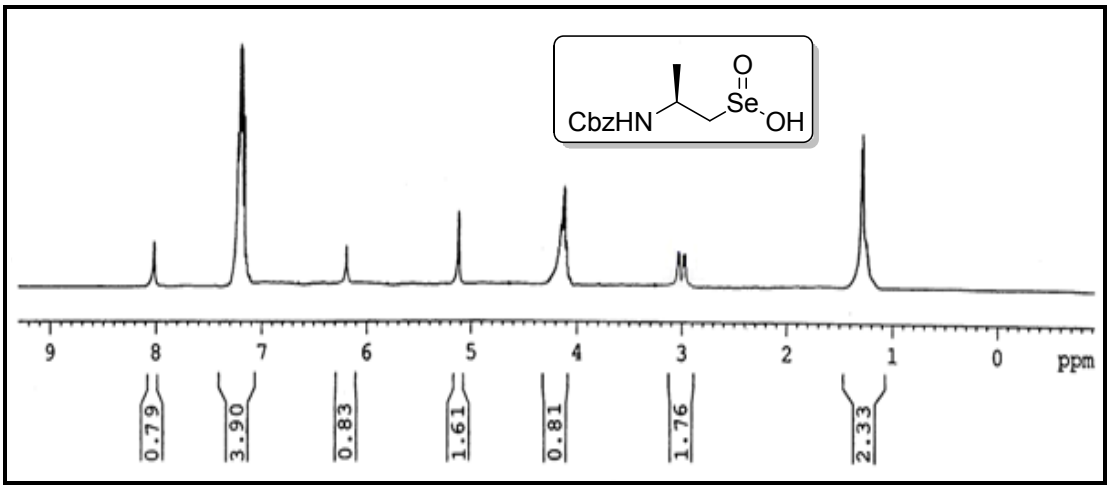
^{1}H NMR of Boc-Phg-CH_2-SeO_2H, 6d



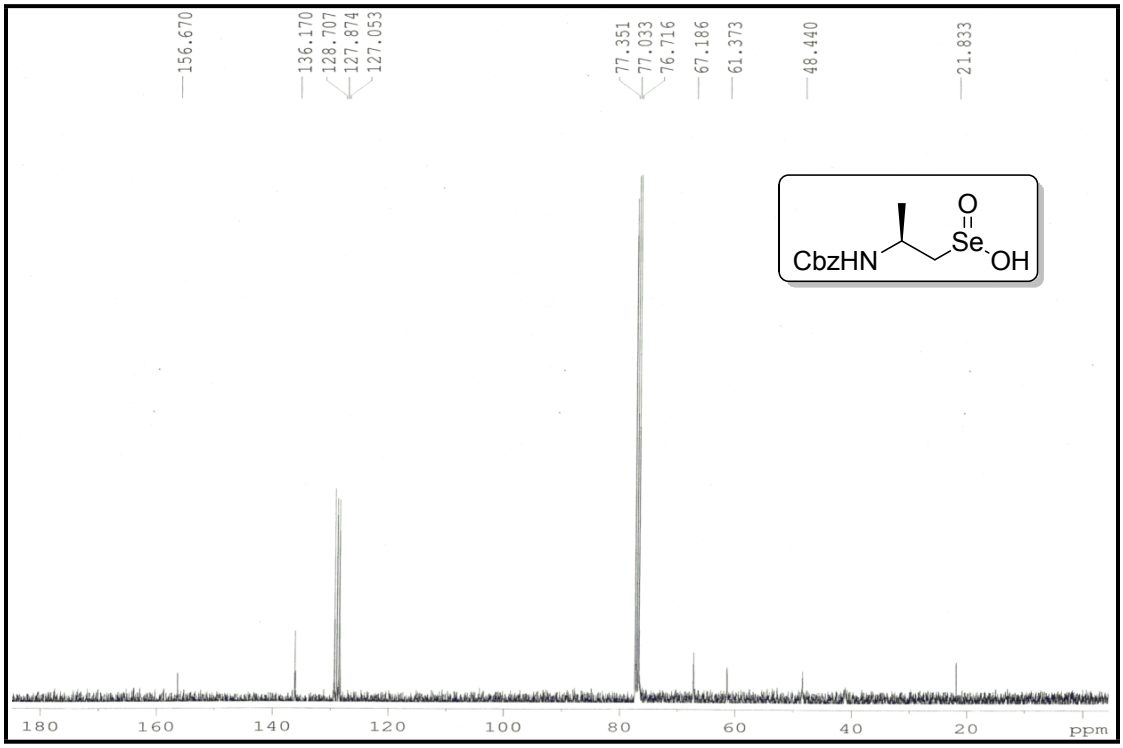
^{13}C NMR of Boc-Phg-CH_2-SeO_2H, 6d



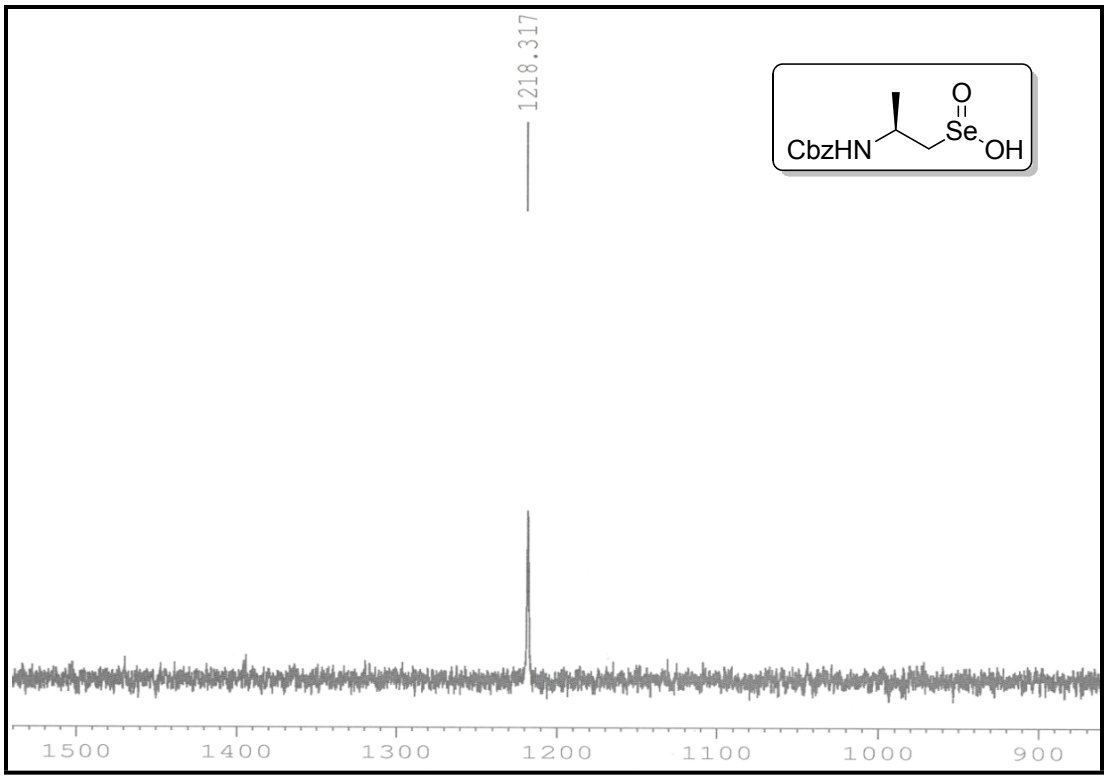
ESI-MS of Cbz-Ala-CH_2-SeO_2H, 6e



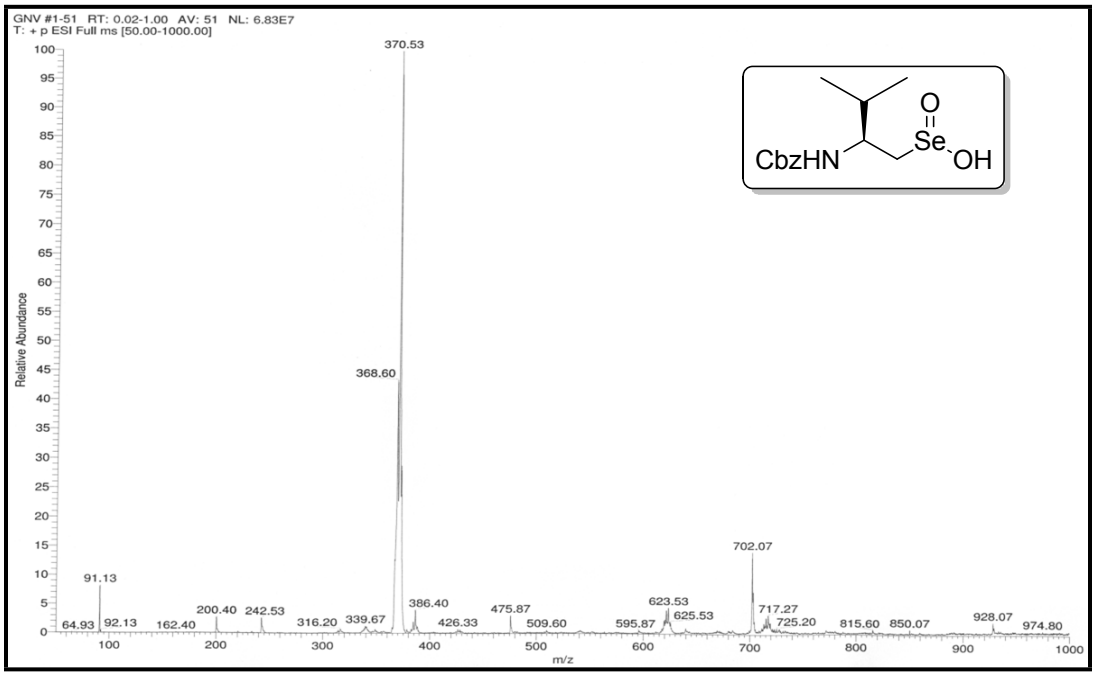
^{1}H NMR of Cbz-Ala-CH_2-SeO_2H, 6e



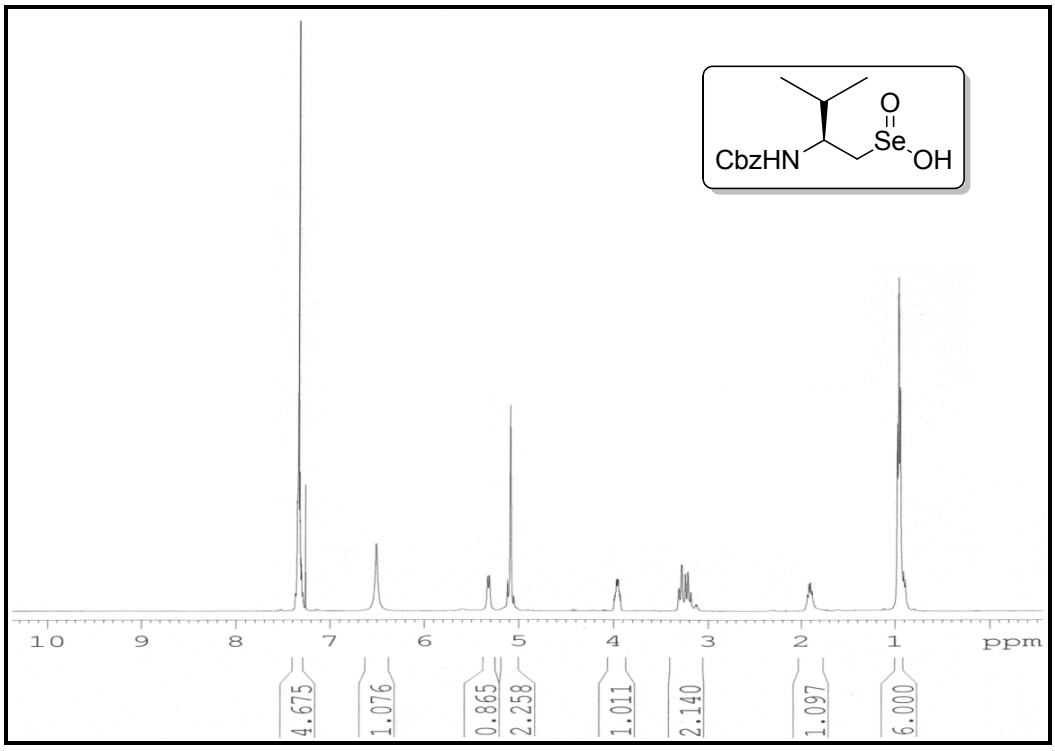
^{13}C NMR of Cbz-Ala-CH_2-SeO_2H, 6e



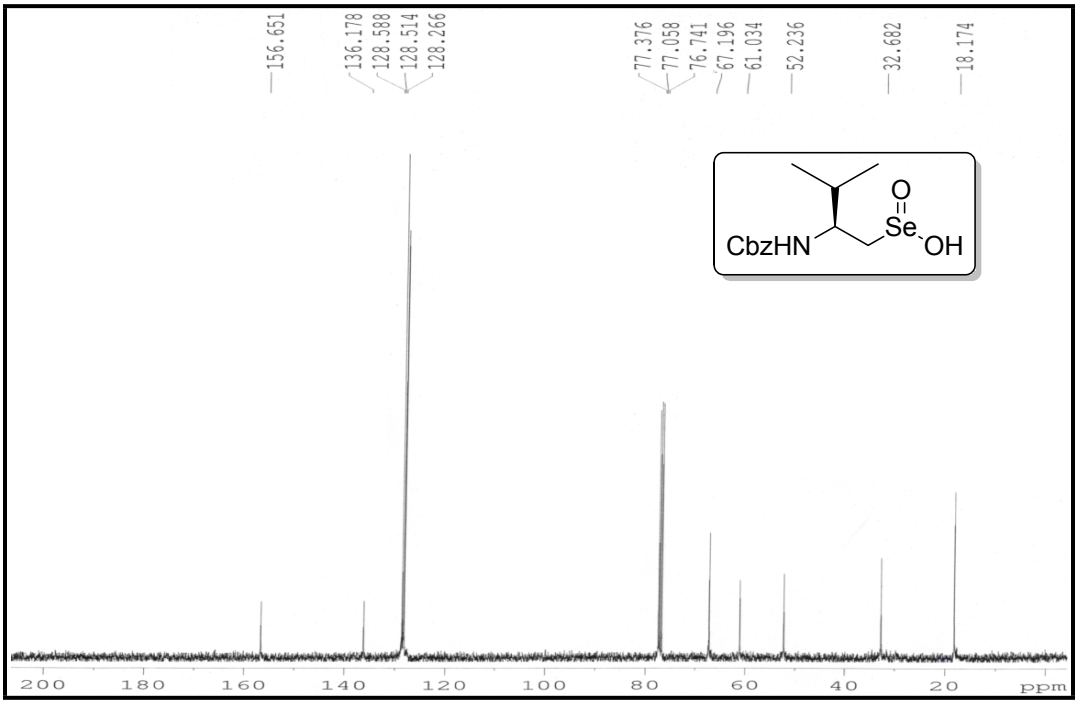
^{77}Se NMR of Cbz-Ala-CH_2-SeO_2H, 6e



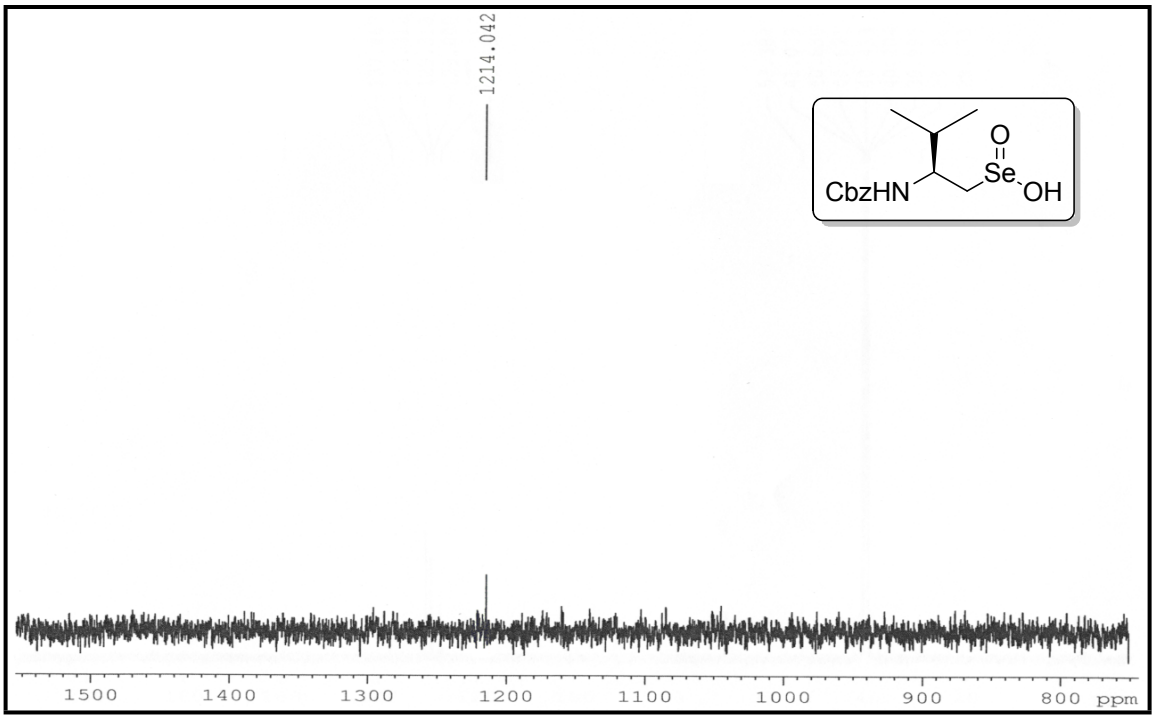
ESI-MS of Cbz-Val-CH_2-SeO_2H, 6f



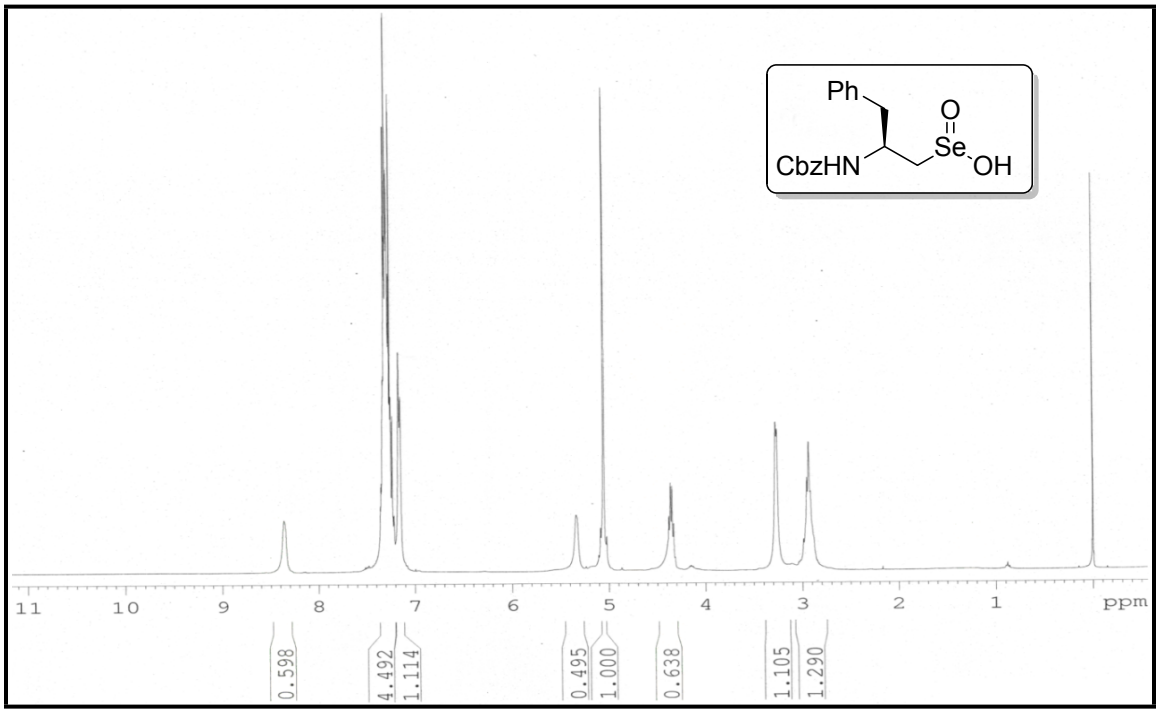
^{1}H NMR of Cbz-Val-CH_2-SeO_2H, 6f



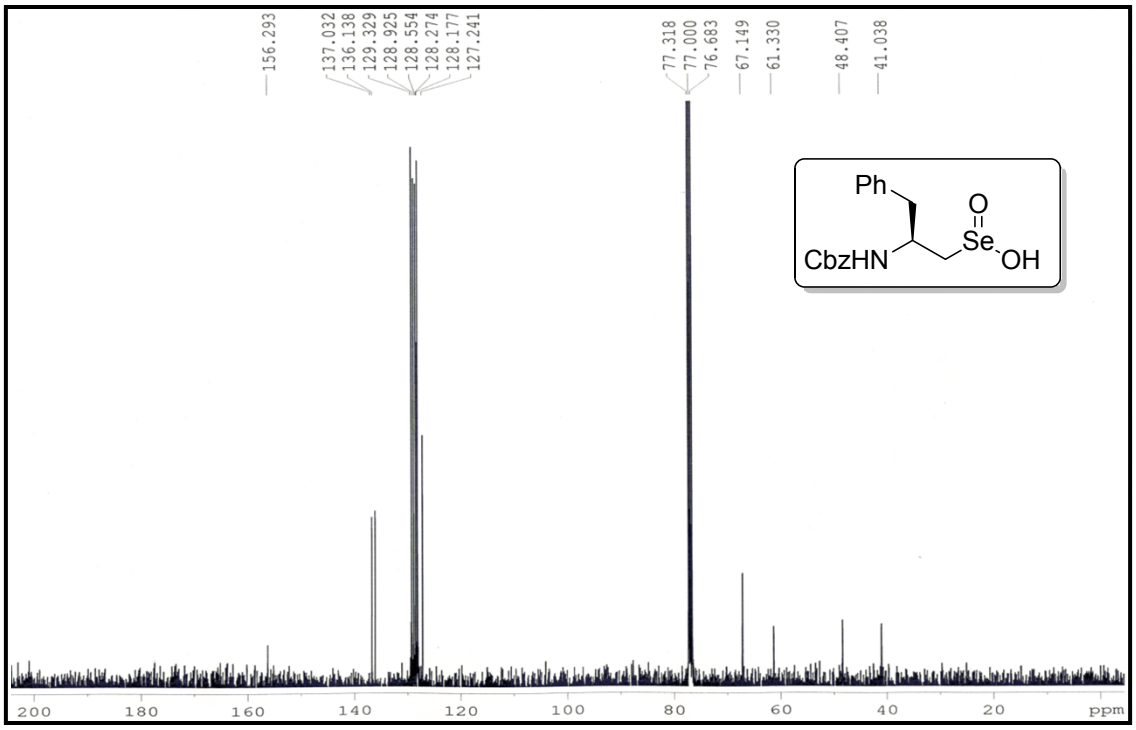
^{13}C NMR of Cbz-Val-CH_2-SeO_2H, 6f



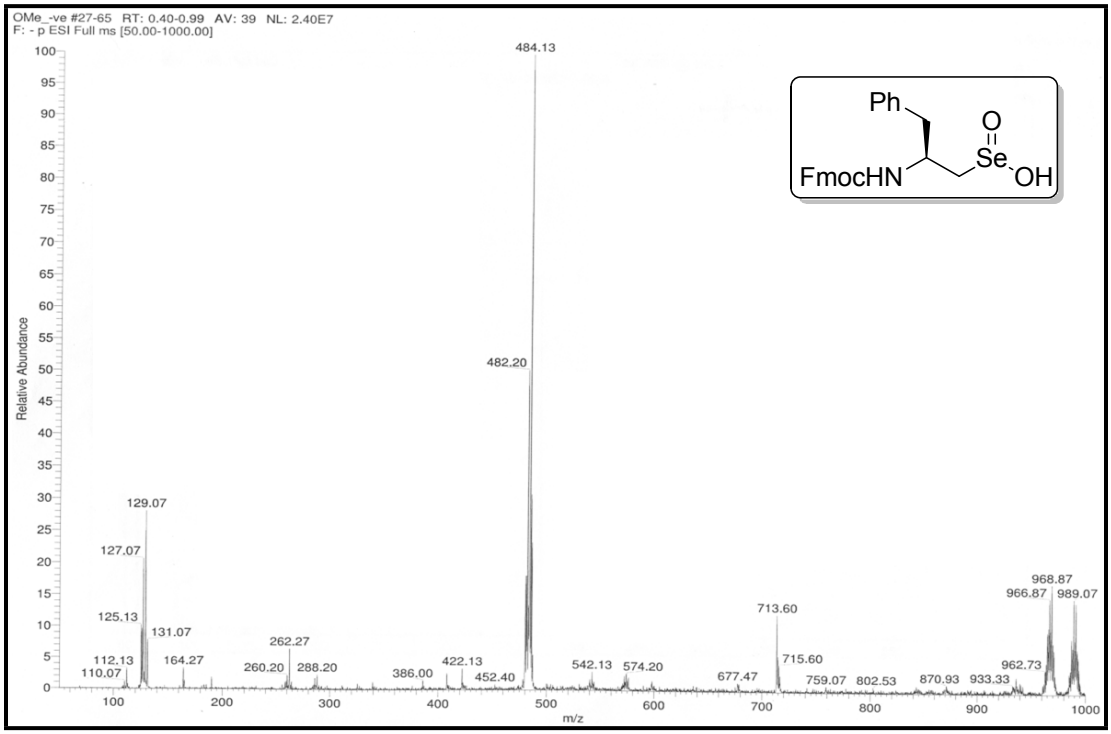
^{77}Se NMR of Cbz-Val-CH_2-SeO_2H, 6f



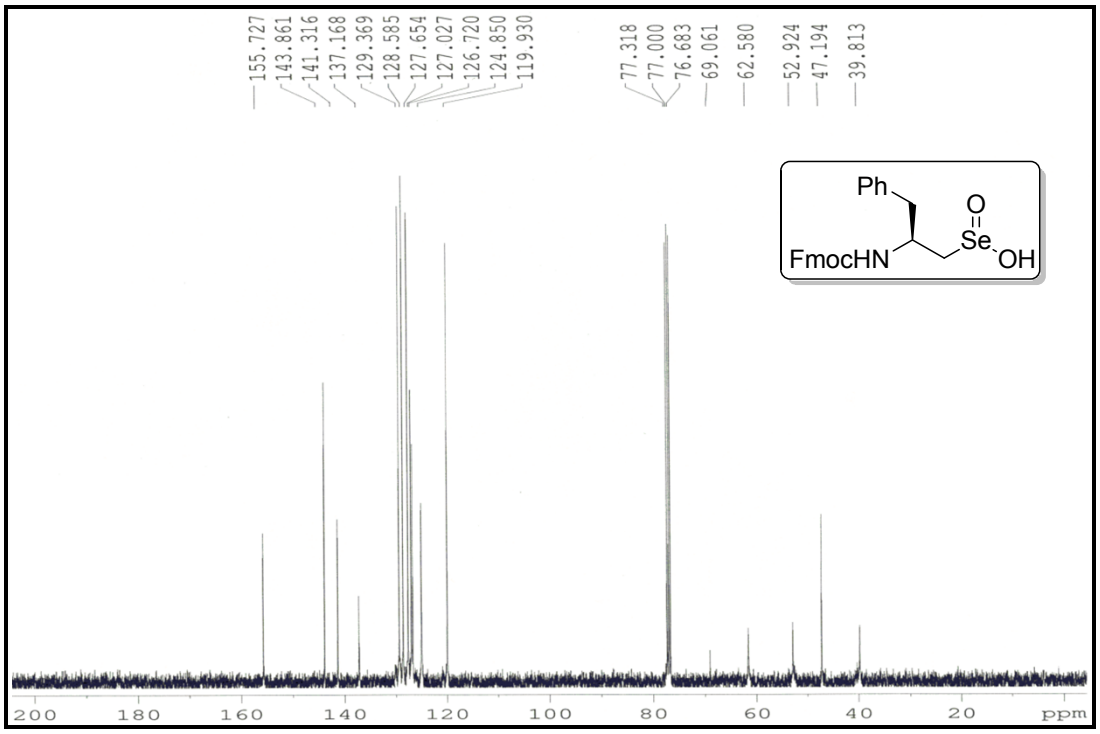
^{1}H NMR of Cbz-Phe-CH_2-SeO_2H, 6g



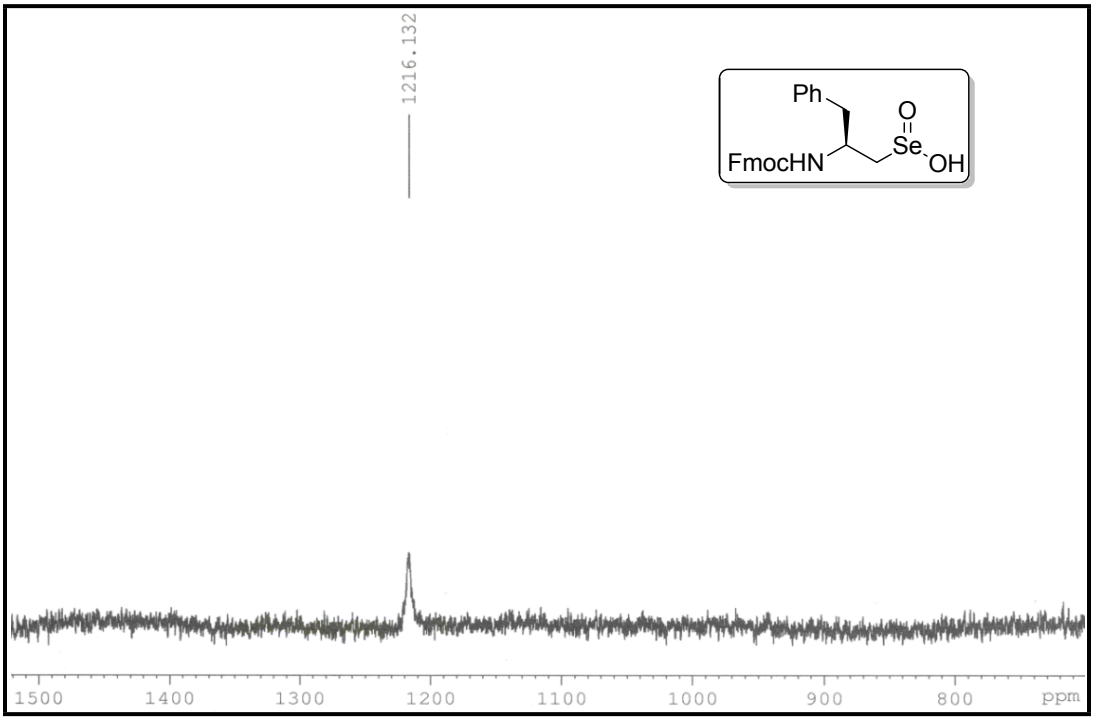
^{13}C NMR of Cbz-Phe-CH_2-SeO_2H, 6g



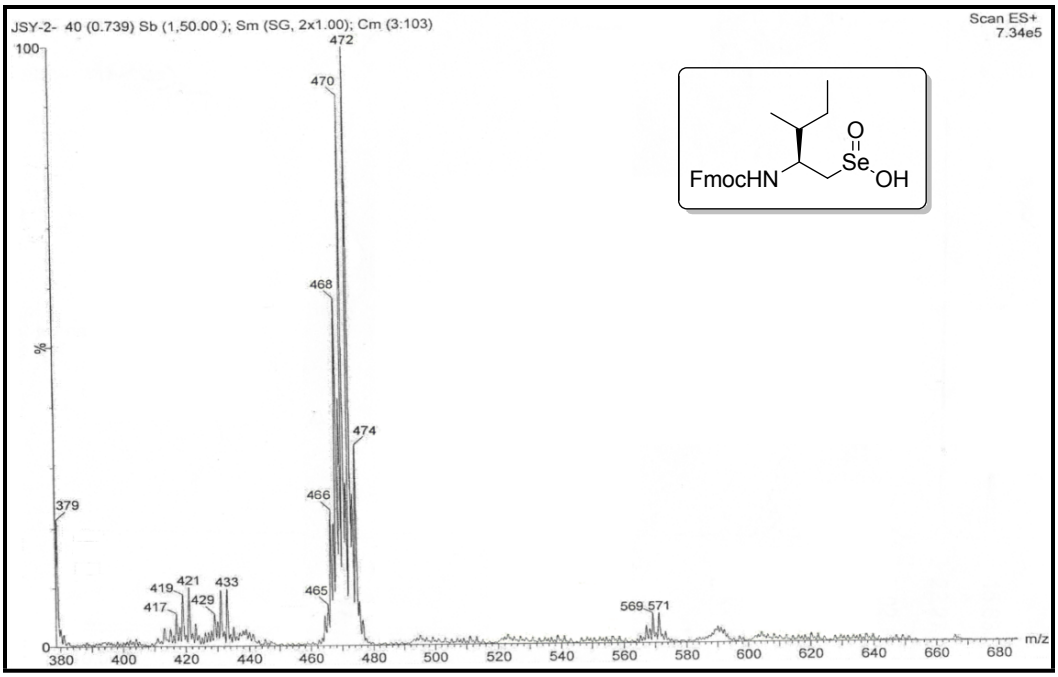
ESI-MS of Fmoc-Phe-CH_2-SeO_2H, 6h



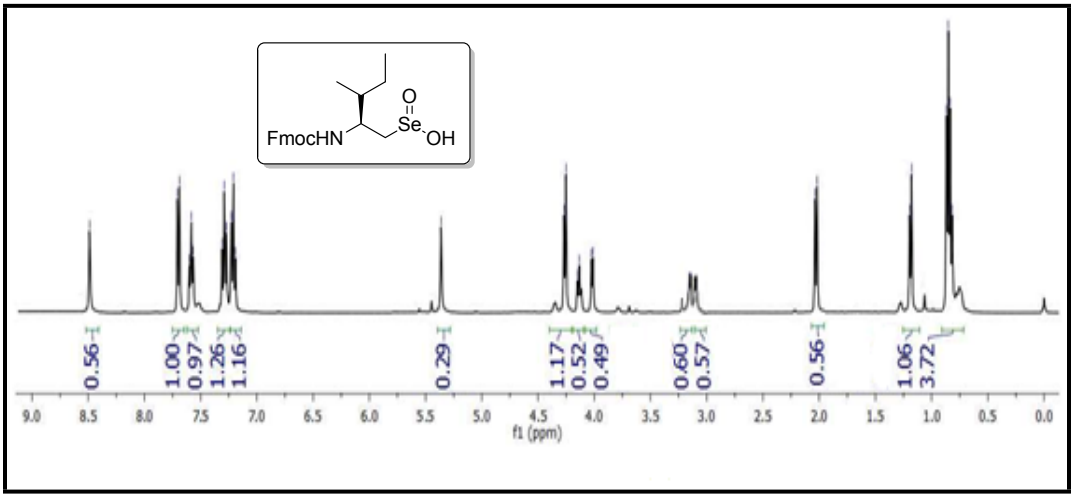
^{13}C NMR of Fmoc-Phe-CH_2-SeO_2H, 6h



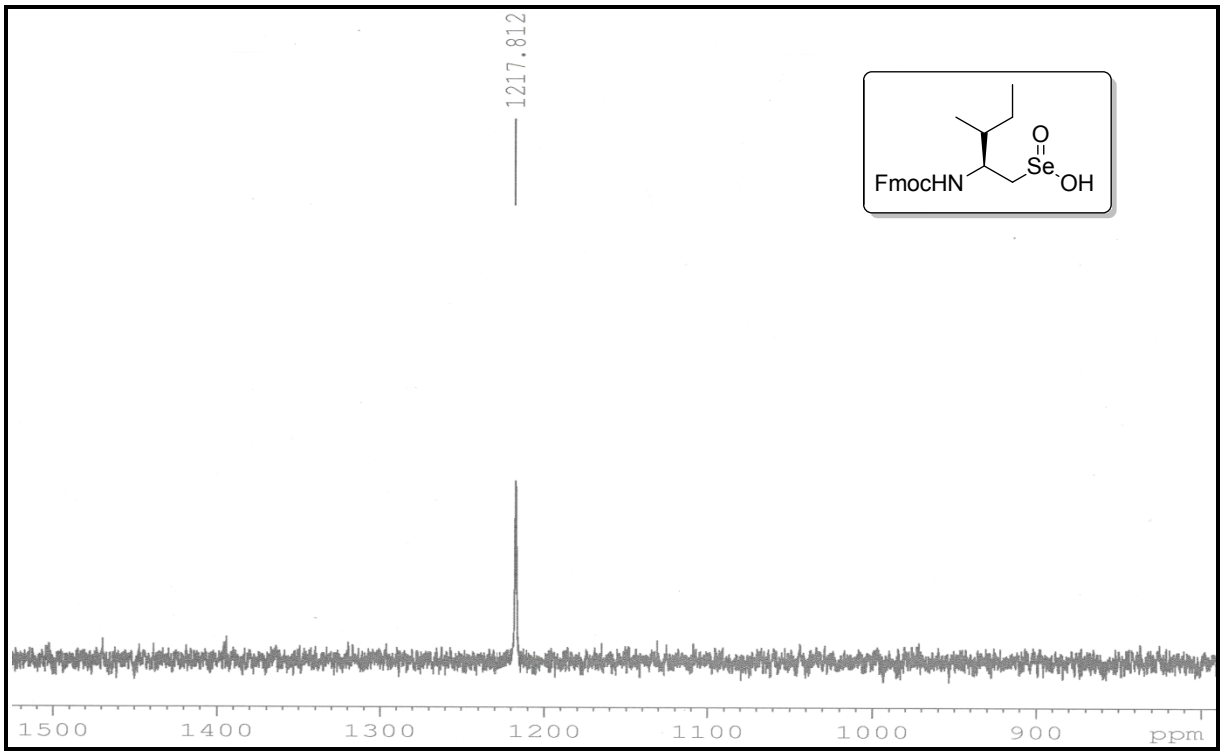
^{77}Se NMR of Fmoc-Phe-CH_2-SeO_2H, 6h



ESI-MS of Fmoc-Ile-CH_2-SeO_2H, 6i

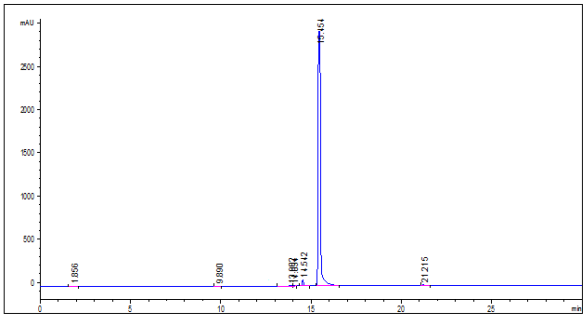


^{1}H NMR of Fmoc-Ile-CH_2-SeO_2H, 6i

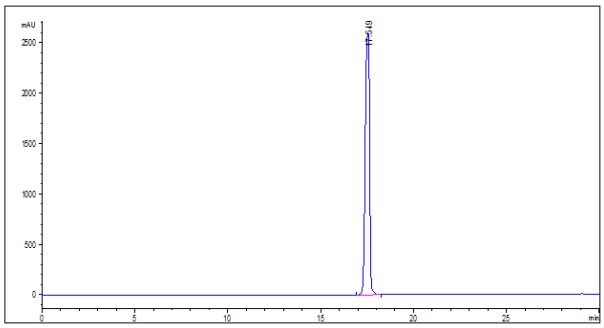


^{77}Se NMR of Fmoc-Ile-CH_2-SeO_2H, 6i

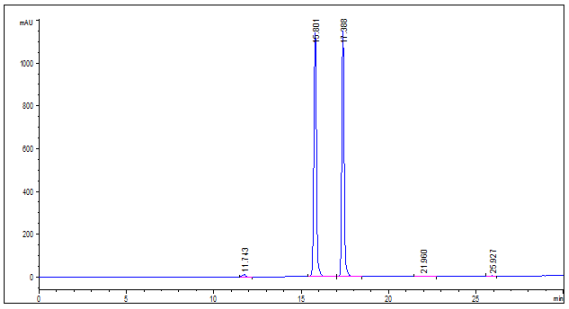
Chiral HPLC chromatograms



2h, Fmoc-L-Phe-CH_2-Se)$_2$-



2h*, Fmoc-D-Phe-CH_2-Se)$_2$-



2h + **2h*** mixture, Fmoc-L, D-Phe-CH_2-Se)$_2$-

Figure 1 Chiral HPLC of 2h, 2h* and mixture of 2h and 2h*

Chiral HPLC particulars:

Agilent 1100 series having G1311A VWD at λ = 254 nm.

Column Type: Phenominex made Lux, pore size-5µ, Cellusole-1.

Column Dimensions: diameter x length = 250 x 4.60 mm.

Flow rate: 1.0 mL/min, 30 min.

Method: n-hexane/isopropanol (70:30).