Electronic Supplementary Information (ESI)

‘One-pot’ synthesis and redox evaluations of chiral chalcogenocysteinol and β-bis-chalcogenoamine derivatives from L-Serine Methyl Ester

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1. General Information

Chemistry: Hydrogen nuclear magnetic resonance ($^1$H NMR) spectra were obtained on a Bruker DPX-600 MHz, DPX-400 MHz or DPX-200 MHz spectrometer. Spectra were recorded in CDCl$_3$ solutions. Chemical shifts are reported in parts per million, referenced to the peak of TMS. Data are reported as follows: chemical shift (d), multiplicity (br = broad, s = singlet, d = doublet, dd = double doublet, t = triplet, m = multiplet), and coupling constant (J) in hertz and integrated intensity. Carbon-13 nuclear magnetic resonance ($^{13}$C NMR) spectra were obtained at 50 MHz or 100 MHz. Spectra were recorded in CDCl$_3$ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl$_3$. Selenium-77 nuclear magnetic resonance ($^{77}$Se NMR) spectra were obtained at 114 MHz. Spectra were recorded in CDCl$_3$ solutions and were used as internal standard diphenyldiselenide. Chemical shifts are reported in parts per million, referenced to the internal standard peak at 463 ppm. High-resolution mass spectra were obtained on a XEVO G2 Q-TOF spectrometer. Thin layer chromatography (TLC) was performed using Merck Silica Gel GF254, 0.25 mm. For visualization, TLC plates were placed under ultraviolet light or stained with either iodine vapor or acidic vanillin. Anhydrous solvents were obtained as follows: THF was distilled from sodium and benzophenone. Triethylamine was distilled from CaH$_2$. All other solvents were used as purchased. The corresponding Mesyl N-Boc protected Methyl Ester 1a was previously prepared and characterized.

Cyclic voltammetric measurements were performed using an AutoLab galvanostat/potentiostat Eco Chemie PGSTAT 302N. In all electrochemical analyses, a three-electrode system was used and consisted of a glassy carbon working electrode, a platinum wire auxiliary electrode and a platinum pseudo-reference electrode (ferrocene was used as internal standard; Fc/Fc+ couple in acetonitrile; E$_{1/2}$ = 0.497 V). All electrochemical experiments were carried out under aerobic conditions at room temperature using dry CH$_3$CN solution of compounds containing 0.1 mol/L tetrabutylammonium hexafluorophosphate (TBAPF$_6$) as the supporting electrode.

2. General procedure for the synthesis β-chalcogenocysteinols 2a-f

Under an argon atmosphere, NaBH$_4$ (0.056 g, 1.5 mmol) was added to a solution of diorganyl dichalcogenide (0.159 g, 0.5 mmol) in THF (3.3 mL) at room temperature. Ethanol (1.1 mL) was added dropwise and the mixture was stirred for 10 min. After this time, a solution of Mesyl N-Boc protected Methyl Ester 1 (1 mmol) in THF (3.0 mL) was added, and the resulting mixture was stirred at room temperature for 12 h. After that time, an additional portion of NaBH$_4$ (0.0945 g, 2.5 mmol) and more ethanol (1.1 mL) were added to the reaction mixture, which was left under stirring for another 12 h. The reaction was quenched with 10 mL of an NH$_4$Cl solution, and the aqueous layer was extracted with CH$_2$Cl$_2$ (3.0 × 20 mL). The combined organic extracts were dried over MgSO$_4$, filtered, and evaporated to dryness. The crude products were purified in a silica gel column for chromatographic purification, using hexane–ethylacetate (30:70) as the eluent, furnishing the pure chiral chalcogenocysteinols 2 a-f.
(R)-2-tert-Butyl carbamoyl-1-hydroxy-3-(phenylselanyl)propane (2a). Physical state: white solid. Melting point: 46-47°C. Yield: 69%. NMR $^1$H (600 MHz, CDCl$_3$): δ = 7.54 (d, $J = 6.0$ Hz, 2H), 7.26 – 7.25 (m, 3H), 5.06 (s, 1H), 3.87-3.78 (m, 1H), 3.75 (dd, $J = 6.0$ Hz, $J' = 12.0$ Hz, 1H), 3.66(dd, $J' = 6.0$ Hz, $J'' = 12.0$ Hz, 1H), 3.16-3.04 (m, 2H), 1.42 (s, 9H) ppm. NMR $^{13}$C (151 MHz, CDCl$_3$): δ = 155.8, 132.9, 129.6, 129.2, 127.2, 79.8, 64.2, 52.2, 29.4, 28.3 ppm. NMR $^{77}$Se (114 MHz, CDCl$_3$): δ = 251.845 ppm. HRMS-ESI: m/z calcd for C$_{14}$H$_{21}$NO$_3$Se [M + Na$^+$] 354.0579, found: 354.0593.

(R)-2-tert-Butyl carbamoyl-1-hydroxy-3-(4-tolylselanyl)propane (2b). Physical state: red oil. Yield: 50%. NMR$^1$H (400 MHz, CDCl$_3$): δ = 7.43 (d, $J = 12.0$ Hz, 2H), 7.07 (d, $J = 12.0$ Hz, 2H), 5.14 (s, 1H), 3.85-3.76 (m, 1H), 3.71(dd, $J' = 4.0$ Hz, $J'' = 8.0$ Hz, 1H), 3.63 (dd, $J' = 4.0$, $J'' = 8$ Hz, 1H), 3.12-2.98 (m, 2H), 2.3 (s, 3H), 1.41 (s, 9H) ppm. NMR$^{13}$C (100 MHz, CDCl$_3$): δ = 155.6, 137.2, 133.2, 130.0, 125.9, 79.6, 64.0, 52.3, 29.8, 28.3, 21.1 ppm. NMR$^{77}$Se (114 MHz, CDCl$_3$): δ = 244.49 ppm. HRMS-ESI: m/z calcd for C$_{15}$H$_{23}$NO$_3$Se [M + Na$^+$] 368.0735, found: 368.0716.

(R)-2-tert-Butyl carbamoyl-1-hydroxy-3-(2-methoxyphenylselanyl)propane (2c). Physical state: red oil. Yield: 35 %. NMR$^1$H (400 MHz, CDCl$_3$): δ = 7.47 (d, $J = 4$ Hz, 1H), 7.22 – 7.19 (m, 1H), 6.90 – 6.81 (m, 2H), 5.41 (s, 1H), 3.85 (s, 3H), 3.75 (dd, $J' = 4$ Hz, $J'' = 8$ Hz, 1H), 3.70-3.60 (m, 2H), 3.18 – 3.03 (m, 2H), 1.41 (s, 9H) ppm. NMR$^{13}$C (100 MHz, CDCl$_3$): δ = 157.7, 155.7, 132.2, 128.1, 121.4, 118.6, 110.4, 79.4, 63.7, 55.9, 51.8, 28.2, 27.0 ppm. NMR$^{77}$Se (114 MHz, CDCl$_3$): δ = 183.32 ppm. HRMS-ESI: m/z calcd for C$_{15}$H$_{23}$NO$_3$Se [M + Na$^+$] 384.0685; found: 384.0723.

(R)-2-tert-Butyl carbamoyl-1-(2-chlorophenylselanyl)-3-hydroxypropane (2d). Physical state: yellow oil. Yield: 42%. NMR$^1$H (400 MHz, CDCl$_3$): δ = 7.55-7.34 (m, 2H), 7.30-7.24 (m, 2H), 5.38 (s, 1H), 3.88-3.77 (m, 1H), 3.76 (dd, $J' = 8.0$ Hz, $J'' = 12.0$ Hz, 1H), 3.64 (dd, $J' = 8.0$ Hz, $J'' = 12.0$ Hz, 1H), 3.19-3.08 (m, 2H), 1.41 (s, 9H) ppm. NMR$^{13}$C (100 MHz, CDCl$_3$): δ = 155.6, 134.7, 131.0, 129.2, 127.1, 79.5, 66.2, 48.1, 31.3, 28.4 ppm. NMR$^{77}$Se (114 MHz, CDCl$_3$): δ = 194.89 ppm. HRMS-ESI: m/z calcd for C$_{14}$H$_{20}$ClNO$_3$Se [M + Na$^+$] 388.0189, found: 388.0199.

(R)-2-tert-Butyl carbamoyl-1-hydroxy-3-(phenylthio)propane (2e). Physical state: white solid. Melting point: 49-50°C. Yield: 69%. NMR$^1$H (400 MHz, CDCl$_3$): δ = 7.40 (d, $J = 8.0$ Hz, 2H), 7.31 – 7.27 (m, 2H), 7.21 – 7.17 (m, 1H), 5.16 (s, 1H), 3.85-3.73 (m, 2H), 3.67 (dd, $J' = 4.0$ Hz, $J'' = 8.0$ Hz, 1H), 3.21 – 3.04 (m, 2H), 1.44 (s, 9H) ppm. NMR$^{13}$C (100 MHz, CDCl$_3$): δ = 155.9, 135.6, 129.5, 128.9, 126.3, 79.8, 63.3, 51.8, 35.0, 28.3 ppm. HRMS-ESI: m/z calcd for C$_{14}$H$_{21}$NO$_3$S [M + Na$^+$] 306.1134, found: 306.1130.

(R)-2-tert-Butyl carbamoyl-1-hydroxy-3-(phenyltellanyl)propane (2f). Physical state: red oil. Yield: 56%. NMR$^1$H (400 MHz, CDCl$_3$): δ = 7.76 (d, $J = 8.0$ Hz, 2H), 7.31-7.26 (m, 1H), 7.22-7.16 (m, 2H), 5.09 (s, 1H), 3.86-3.73 (m, 1H), 3.72-3.60 (m, 2H), 3.19-3.05 (m, 2H), 1.43 (s, 9H) ppm. NMR$^{13}$C (100 MHz, CDCl$_3$): δ = 155.7, 138.4, 129.3, 127.8, 111.7, 79.7, 65.3, 52.9, 28.3, 11.5 ppm. HRMS-ESI: m/z calcd for C$_{14}$H$_{21}$NO$_3$Te [M + Na$^+$] 404.0476, found: 404.0501.

Under an argon atmosphere, NaBH₄ (0.045 g, 1.2 mmol) was added to a solution of diorganodichalcogenide (0.054g, 0.25 mmol) in THF (3.3 mL) at room temperature. Ethanol (1.1 mL) was added dropwise and the mixture was stirred for 10 min. After this time, a solution of Mesylβ-organochalcogenamine 3 in THF (3.0 mL) was added, and the resulting mixture was stirred at 80 °C for 2 h. The reaction was quenched with 10 mL of an NH₄Cl solution, and the aqueous layer was extracted with CH₂Cl₂ (3.0 × 20 mL). The combined organic extracts were dried over MgSO₄, filtered, and evaporated to dryness. The crude products were purified in a silica gel column for chromatographic purification, using hexane–ethyl acetate (5:95) as the eluent.

(R) 2-tert-Butylcarbamoyl-1-(phenylselanyl)-3-(phenylthio) propane (5a). Physical state: white solid. Melting point: 46-47°C. Yield: 61%. NMR ¹H (600 MHz, CDCl₃): 7.50 (d, J = 6 Hz, 2H), 7.34 (d, J = 6 Hz, 2H), 7.27-7.23 (m, 5H), 7.19-7.16 (m, 1H), 4.90 (s, 1H), 4.04-3.94 (m, 1H), 3.29-3.18 (m, 2H), 3.13 (dd, J' = 6.0 Hz, J = 12.0 Hz, 1H), 3.09 (dd, J' = 6.0 Hz, J = 12.0 Hz, 1H), 1.39 (s, 9H) ppm. NMR ¹³C (151 MHz, CDCl₃): 154.8, 132.8, 129.6, 129.1, 127.1, 126.4, 79.5, 50.0, 38.0, 31.9, 28.2 ppm. NMR ⁷⁷Se (114 MHz, CDCl₃): δ = 248.55 ppm. HRMS-ESI: m/z calcd for C₂₀H₂₅NO₃SSe [M + Na⁺]: 446.0663, found: 446.0665

2-tert-Butyl carboxamoyl-1,3-bis(phenylselanyl) propane (5b). Physical state: white solid. Melting point: 46-47°C. Yield: 83%. NMR ¹H (400 MHz, CDCl₃): 7.53-7.44 (m, 5H), 7.25-7.19 (m, 5H), 4.83 (s, 1H), 4.04-3.96 (m, 1H), 3.25-3.15 (m, 2H), 3.09 (dd, J = 4.0 Hz, J = 8.0 Hz, 2H), 1.38 (s, 9H) ppm. NMR ¹³C (100 MHz, CDCl₃): 154.8, 132.8, 129.6, 129.1, 127.1, 126.9, 79.6, 50.5, 32.7, 28.2 ppm. NMR ⁷⁷Se (114 MHz, CDCl₃): δ = 257.30 ppm, 250.06 ppm, 242.66 ppm. HRMS-ESI: m/z calcd for C₂₀H₂₅NO₃SSe [M + Na⁺]: 494.0108, found: 494.0108.

(S)-2-tert-Butyl carbamoyl-1-(phenylselanyl)-3-(phenyltellanyl) propane (5c). Physical state: colorless oil. Yield: 40%. NMR ¹H (400 MHz, CDCl₃): 7.74 (d, J = 4 Hz, 2H), 7.60-7.48 (m, 5H), 7.22 – 7.19 (m, 3H), 4.83 (s, 1H), 3.99-3.89 (m, 1H), 3.30 – 3.19 (m, 2H), 3.13 (dd, J = 8.0 Hz, J = 16.0 Hz, 2H), 1.44 (s, 9H) ppm. NMR ¹³C (100 MHz, CDCl₃): 155.0, 138.6, 133.0, 132.6, 130.85, 129.3, 129.2, 129.1, 127.8, 127.2, 126.9, 79.3, 51.1, 35.5, 28.3, 19.1 ppm. HRMS-ESI: m/z calcd for C₂₀H₂₅NO₃SSe [M + Na⁺]: 544.0005, found: 544.0023.

(R)-2-tert-Butyl carbamoyl-1-(phenylthio)-3-(4-tolyldiseleno) propane (5d). Physical state: red oil. Yield: 47%. NMR ¹H (400 MHz, CDCl₃): 7.43-7.37 (m, 3H), 7.34 (d, J = 8.0 Hz, 2H), 7.19-7.15 (m, 2H), 7.05 (d, J = 8.0 Hz, 2H), 4.79 (s, 1H), 4.01 – 3.83 (m, 1H), 3.26-3.13 (m, 2H), 3.12- 3.07 (m, 2H), 2.31 (s, 3H), 1.42 (s, 9H) ppm. NMR ¹³C (100 MHz, CDCl₃): 154.7, 138.6, 132.9, 132.6, 130.9, 129.3, 129.2, 129.1, 127.8, 127.20, 126.9, 79.3, 51.1, 35.6, 34.5, 28.3, 19.1 ppm. HRMS-ESI: m/z calcd for C₂₁H₂₇NO₂SSe [M + Na⁺]: 460.0820 found: 460.0848.

(R)-2-tert-Butyl carbamoyl-1-(phenylselanyl)-3-(4-tolyldiseleno) propane (5e). Physical state: yellow oil. Yield: 45%. NMR ¹H (400 MHz, CDCl₃): 7.40 (d, J = 8.0 Hz,
(S)- 2-tert-Butyl carbamoyl-1-(phenyltellanyl)-3-(4-tolylselanyl)propane (5f). Physical state: yellow oil. Yield: 50%. NMR $^1$H (400 MHz, CDCl$_3$): 7.72 (d, $J$ = 8.0 Hz, 2H), 7.39 (d, $J$ = 8.0 Hz, 2H), 7.29-7.24 (m, 1H), 7.21-7.15 (m, 2H), 7.06 (d, $J$ = 8.0 Hz, 2H), 4.77 (s, 1H), 4.03-3.83 (m, 1H), 3.25-3.00 (m, 4H), 2.31 (s, 3H), 1.38 (s, 9H) ppm. NMR $^{13}$C (100 MHz, CDCl$_3$): 154.8, 137.3, 133.4, 132.9, 130.0, 129.1, 127.1, 79.7, 51.1, 35.5, 33.1, 28.3, 21.0 ppm. HRMS $^{77}$Se (114 MHz, CDCl$_3$): $\delta$ = 248.63 ppm, 241.13 ppm. HRMS-ESI: m/z calcd for C$_{21}$H$_{27}$NO$_2$Se$_2$ [M + Na$^+$]: 508.0264, found: 508.0280.

(R)- 2-tert-Butyl carbamoyl 1-(2-methoxyphenyrselenyl)-3-(phenylthio)propane (5g). Physical state: yellow oil. Yield: 44%. NMR $^1$H (600 MHz, CDCl$_3$): 7.46 (d, $J$ = 6.0 Hz, 1H), 7.35 (d, $J$ = 6.0 Hz, 2H), 7.30 – 7.23 (m, 4H), 7.19 – 7.16 (m, 1H), 6.88-6.83 (m, 2H), 5.09 (s, 1H), 4.05-3.96 (m, 1H), 3.88 (s, 3H), 3.34-3.23 (m, 2H), 3.12 (dd, $J$' = 6.0Hz, $J$'' = 12.0 Hz, 1H), 3.11-3.06 (m, 1H), 1.40 (s, 9H) ppm. NMR $^{13}$C (151 MHz, CDCl$_3$): 158.1, 154.9, 153.7, 133.2, 129.4, 129.5, 129.0, 128.6, 126.4, 126.3, 121.5, 110.6, 79.3, 56.1, 49.80, 38.2, 29.9, 28.1 ppm. HRMS-ESI: m/z calcd for C$_{21}$H$_{27}$NO$_3$SeTe [M + Na$^+$]: 476.0769, found: 476.0774.

(R)- 2-tert-Butyl carbamoyl 1-(2-methoxyphenyrselenyl)-3-(phenylselenyl)propane (5h). Physical state: red oil. Yield: 43%. NMR $^1$H (400 MHz, CDCl$_3$): 7.57 – 7.50 (m, 2H), 7.47 (d, $J$ = 4.0 Hz, 1H), 7.28 – 7.23 (m, 4H), 6.92 – 6.85 (m, 2H), 5.08 (s, 1H), 4.12-4.05 (m, 1H), 3.90 (s, 3H), 3.37-3.19 (m, 2H), 3.12 (dd, $J$' = 8.0 Hz, $J$'' = 16.0 Hz, 1H), 3.08 (dd, $J$' = 8.0Hz, $J$'' = 16.0 Hz, 1H), 1.41 (s, 9H) ppm. NMR $^{13}$C (100 MHz, CDCl$_3$): 158.0, 154.8, 133.1, 132.6, 129.7, 129.7, 129.1, 128.5, 127.0, 121.5, 118.4, 110.5, 79.5, 55.7, 50.1, 35.6, 32.5, 28.2 ppm. HRMS $^{77}$Se (114 MHz, CDCl$_3$): $\delta$ = 250.21 ppm, 180.07 ppm. HRMS-ESI: m/z calcd for C$_{21}$H$_{27}$NO$_3$Se$_2$ [M + Na$^+$]: 524.0214, found: 524.0214.

(S)-2-tert-Butyl carbamoyl- 1-(2-methoxyphenyrselenyl)-3-(phenyltellanyl)propane (5i). Physical state: red oil. Yield: 50%. NMR $^1$H (200 MHz, CDCl$_3$): 7.72 (d, $J$ = 8.0 Hz, 2H), 7.42 (d, $J$ = 8.0 Hz, 1H), 7.29 – 7.13 (m, 4H), 6.91 – 6.82 (m, 2H), 5.02 (s, 1H), 4.10-3.96 (m, 1H), 3.88 (s, 3H), 3.33 – 2.96 (m, 4H), 1.38 (s, 9H) ppm. NMR $^{13}$C (50 MHz, CDCl$_3$): 158.0, 154.8, 133.1, 132.7, 129.8, 129.1, 127.0, 121.50, 118.4, 111.8, 110.5, 79.2, 55.8, 50.3, 32.5, 28.0, 17.3 ppm. HRMS-ESI: m/z calcd for C$_{21}$H$_{27}$NO$_3$Se$_2$ [M + Na$^+$]: 574.0111, found: 574.0133.

(R)-2-tert-Butyl carbamoyl-1-(2-chlorophenylselenyl)-3-(phenylthio)propane (5j). Physical state: colorless oil. Yield: 44%. NMR $^1$H (200 MHz, CDCl$_3$): $\delta$ = 7.32 – 7.18 (m, 9H), 4.90 (s, 1H), 4.10-3.95 (m, 1H), 3.34-3.07 (m, 4H), 1.40 (s, 9H) ppm. NMR $^{13}$C (50 MHz, CDCl$_3$): 154.9, 135.4, 131.9, 131.3, 130.4, 129.8, 129.6, 129.1, 128.7, 127.8, 127.4, 126.5, 79.7, 50.0, 38.2, 30.5, 28.3 ppm. HRMS-ESI: m/z calcd for C$_{20}$H$_{24}$ClNO$_2$Se$_2$ [M + Na$^+$]: 480.0274, found: 480.0249.

(R)- 2-tert-Butyl carbamoyl 1-(2-chlorophenylselenyl)-3-(phenylselenyl)propane (5k). Physical state: yellow oil. Yield: 47%. NMR $^1$H (200 MHz, CDCl$_3$): 7.58 – 7.37 (m,
(S)-2-tert-Butyl carbamoyl-1-(2-chlorophenylselanyl)-3-(phenyltellanyl)propane (5l). Physical state: colorless oil. Yield: 70%. NMR $^{13}$C (50 MHz, CDCl$_3$): 154.8, 138.6, 132.9, 132.0, 129.6, 129.2, 127.9, 127.8, 127.3, 126.9, 126.5, 79.6, 50.3, 37.5, 31.3, 28.3 ppm. HRMS: m/z calcd for C$_{20}$H$_{24}$ClNO$_2$Se$_2$ [M + Na$^+$]: 527.9718 found: 527.9725.

2-tert-Butyl carbamoyl -1,3-bis(phenylthio)propane (5m). Physical state: white solid. Yield: 45%. NMR $^1$H (400 MHz, CDCl$_3$): 7.73 (d, $J$ = 8.0 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.28 – 7.20 (m, 5H), 7.20 – 7.12 (m, 1H), 5.11 – 4.66 (m, 1H), 4.07 – 3.85 (m, 1H), 3.27 – 3.16 (m, 2H), 3.16 – 3.05 (m, 2H), 1.39 (s, 9H) ppm. NMR $^{13}$C (100 MHz, CDCl$_3$): 154.8, 135.5, 134.7, 132.8, 129.7, 129.1, 128.9, 127.1, 126.3, 99.9, 79.6, 50.2, 37.9, 31.8, 28.2 ppm. HRMS-ESI: m/z calcd for C$_{20}$H$_{25}$NO$_2$S$_2$ [M + Na$^+$]: 496.0560 found: 496.0174.

(S)- 2-tert-Butyl carbamoyl 1-(phenyltellanyl)-3-(phenylthio)propane (5n) Physical state: yellow oil. Yield: 68%. NMR $^1$H (400 MHz, CDCl$_3$): 7.73 (d, $J$ = 8.0 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.28 – 7.20 (m, 5H), 7.20 – 7.12 (m, 1H), 5.11 – 4.66 (m, 1H), 4.07 – 3.85 (m, 1H), 3.27 – 3.16 (m, 2H), 3.16 – 3.05 (m, 2H), 1.39 (s, 9H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$): 154.8, 135.5, 134.7, 132.8, 129.7, 129.1, 128.9, 127.1, 126.3, 99.9, 79.6, 50.2, 37.9, 31.8, 28.2 ppm. HRMS-ESI: m/z calcd for C$_{20}$H$_{25}$NO$_2$S$_2$Te [M + Na$^+$]: 496.0560 found: 496.0174.

(S)- 2-tert-Butyl carbamoyl 1-(phenylselanyl)-3-(phenylthio)propane (5o). Physical state: colorless oil. Yield: 70%. $^1$H NMR (400 MHz, CDCl$_3$): 7.51 – 7.48 (m, 2H), 7.33 – 7.32 (m, 2H), 7.28 – 7.20 (m, 5H), 7.20 – 7.12 (m, 1H), 5.11 – 4.66 (m, 1H), 4.07 – 3.85 (m, 1H), 3.27 – 3.16 (m, 2H), 3.16 – 3.05 (m, 2H), 1.39 (s, 9H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$): 154.8, 135.5, 134.7, 132.8, 129.7, 129.1, 128.9, 127.1, 126.3, 99.9, 79.6, 50.2, 37.9, 31.8, 28.2 ppm. HRMS-ESI: m/z calcd for C$_{20}$H$_{25}$NO$_2$S$^2$ [M + Na$^+$]: 446.0663 found: 446.0691.

(S)-2-tert-Butyl carbamoyl-1-(2-chlorophenylselanyl)-3-(phenylselanyl)propane (5p). Physical state: colorless oil. Yield: 45%. $^1$H NMR (400 MHz, CDCl$_3$): 7.58 – 7.46 (m, 2H), 7.48 – 7.38 (m, 1H), 7.37 – 7.28 (m, 1H), 7.26 – 7.16 (m, 3H), 7.16 – 7.06 (m, 2H), 4.88-4.86 (m, 1H), 4.07 – 3.95 (m, 1H), 3.31 – 3.17 (m, 2H), 3.13 (dd, $J$' = 12Hz, $J$'' = 8Hz, 2H), 1.39 (s, 9H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$): 154.8, 132.9, 131.9, 130.4, 129.5, 129.2, 127.7, 127.3, 127.2, 79.5, 50.2, 37.9, 31.42, 28.3 ppm. HRMS-ESI: m/z calcd for C$_{20}$H$_{24}$ClNO$_2$Se$_2$ [M + Na$^+$]: 527.9718 found: 527.9747.
4. Compounds Description: NMR

Figure 1. $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 2a in CDCl$_3$ at 600 MHz, 151 MHz
and 114 MHz respectively.

**Figure 2.** $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 2b in CDCl$_3$ at 400 MHz, 100 MHz and 114 MHz respectively.
Figure 3. $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 2c in CDCl$_3$ at 400 MHz, 100 MHz and 114 MHz respectively.
Figure 4. $^{1}H$, $^{13}C$ and $^{77}Se$ NMR of the compound 2d in CDCl$_3$ at 400 MHz, 100 MHz and 114 MHz respectively
Figure 5. $^1$H and $^{13}$C NMR of the compound 2e in CDCl$_3$ at 400 MHz and 100 MHz respectively
Figure 6. $^1$H and $^{13}$C NMR of the compound 2f in CDCl$_3$ at 400 MHz and 100 MHz respectively.
Figure 7. $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 5a in CDCl$_3$ at 600 MHz, 151 MHz and 114 MHz respectively.
Figure 8. $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 5b in CDCl$_3$ at 400 MHz, 100 MHz and 114 MHz respectively.
Figure 9. $^1$H and $^{13}$C of the compound 5c in CDCl$_3$ at 400 MHz and 100 MHz respectively
Figure 10. $^1$H and $^{13}$C of the compound 5d in CDCl$_3$ at 400 MHz and 100 MHz respectively.
**Figure 11.** $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 5e in CDCl$_3$ at 400 MHz, 100 MHz and 114 MHz respectively
Figure 12. $^1$H and $^{13}$C of the compound 5f in CDCl$_3$ at 400 MHz and 100 MHz respectively.
Figure 13. $^1$H and $^{13}$C of the compound 5g in CDCl$_3$ at 400 MHz and 100 MHz respectively.
Figure 14. $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 5h in CDCl$_3$ at 400 MHz, 100 MHz and 114 MHz respectively.
Figure 15. $^{1}$H and $^{13}$C of the compound 5i in CDCl$_3$ at 400 MHz and 100 MHz respectively
Figure 16. $^1$H and $^{13}$C of the compound 5j in CDCl$_3$ at 400 MHz and 100 MHz respectively.
Figure 17. $^1$H, $^{13}$C and $^{77}$Se NMR of the compound 5k in CDCl$_3$ at 400 MHz, 100 MHz and 114 MHz respectively.
Figure 18. $^1$H and $^{13}$C of the compound 5l in CDCl$_3$ at 400 MHz and 100 MHz respectively.
Figure 19. $^1$H and $^{13}$C of the compound 5m in CDCl$_3$ at 400 MHz and 100 MHz respectively
Figure 20.$^1$H and $^{13}$C of the compound 5n in CDCl$_3$ at 400 MHz and 100 MHz respectively.
Figure 21. $^1$H and $^{13}$C of the compound 5o in CDCl$_3$ at 400 MHz and 100 MHz respectively.
Figure 22. $^1$H and $^{13}$C of the compound 5p in CDCl$_3$ at 400 MHz and 100 MHz respectively
5. Cyclic voltammograms

Figure 23. Cyclic voltammogram of compound 2a measured in dry acetonitrile solution, using 0.1 M TBAPF$_6$ as supporting electrolyte, at scan rate 50-200 mV/s, respectively.

Figure 24. Cyclic voltammogram of compound 2e measured in dry acetonitrile solution, using 0.1 M TBAPF$_6$ as supporting electrolyte, at scan rate 50-200 mV/s, respectively.
Figure 25. Cyclic voltammogram of compound 5a measured in dry acetonitrile solution, using 0.1 M TBAPF₆ as supporting electrolyte, at scan rate 50-200 mV/s, respectively.

Figure 26. Cyclic voltammogram of compound 5b measured in dry acetonitrile solution, using 0.1 M TBAPF₆ as supporting electrolyte, at scan rate 50-200 mV/s, respectively.
Figure 27. Cyclic voltammogram of compound 5c measured in dry acetonitrile solution, using 0.1 M TBAPF₆ as supporting electrolyte, at scan rate 50-200 mV/s, respectively.

Figure 28. Cyclic voltammogram of compound 5h measured in dry acetonitrile solution, using 0.1 M TBAPF₆ as supporting electrolyte, at scan rate 50-200 mV/s, respectively.
Figure 29. Cyclic voltammogram of compound 5k measured in dry acetonitrile solution, using 0.1 M TBAPF₆ as supporting electrolyte, at scan rate 50-200 mV/s, respectively.
6. High resolution mass spectra

Figure 30. HRMS spectrum of 2a
Figure 31. HRMS spectrum of 2b

Figure 32. HRMS spectrum of 2c
Figure 33. HRMS spectrum of 2d
Figure 34. HRMS spectrum of 2e
Figure 35. HRMS spectrum of 2f
Figure 36. HRMS spectrum of 5a
Figure 37. HRMS spectrum of 5b
Figure 38. HRMS spectrum of 5c
Figure 39. HRMS spectrum of 5d
Figure 40. HRMS spectrum of 5e
Figure 41. HRMS spectrum of 5f
Figure 42. HRMS spectrum of 5g
Figure 43. HRMS spectrum of 5h
Figure 44. HRMS spectrum of 5i
Figure 45. HRMS spectrum of 5j
Figure 46. HRMS spectrum of 5k
Figure 47. HRMS spectrum of 5l
Figure 48. HRMS spectrum of 5m
Figure 49. HRMS spectrum of 5n
Figure 50. HRMS spectrum of 5o
Figure 51. HRMS spectrum of 5p