
Supporting Information

One-pot synthesis of functionalized β -amino sulfides/ β -amino selenides *via* ring opening of cyclic sulfamidates.

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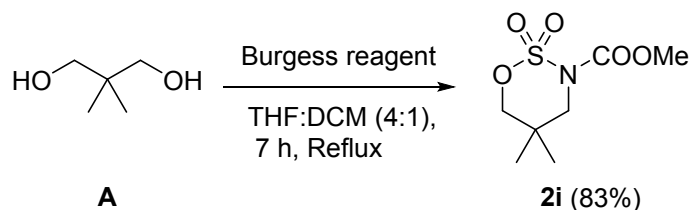
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General Information:

All the reactions were performed in oven dry apparatus and were stirred magnetically. Melting point values reported are uncorrected. Infrared spectra were recorded using an FTIR instrument and the frequencies are reported in wavenumber (cm^{-1}) and intensities of the peak are denoted as s (strong), w (weak), m (medium), and broad (br). ^1H and ^{13}C spectra were recorded at 400 MHz and 100 MHz NMR instruments respectively. Chemical shifts are reported in parts per million downfield from the internal reference, tetramethylsilane (TMS). Multiplicity is indicated using the following abbreviations: s (singlet), d (doublet), dd (double doublet) t (triplet), m (multiplet), bs (broad singlet). Mass spectra were recorded on Q-TOF electro-spray instrument. References for the compound reported previously are indicated against each of them along with the characterization data.

Experimental Data:

Synthetic procedure of compound 2i



2,2-Dimethylpropane-1,3-diol **A** (52.1 mg, 0.5 mmol) was dissolved in THF/ CH_2Cl_2 (4:1, 5 mL) and the Burgess reagent (297.9 mg, 1.25 mmol) was added at 25 °C in a single portion. The resultant solution was immediately warmed to reflux (using a preheated oil bath) and stirred for 7 h. Upon completion, the reaction contents were cooled to 25 °C, poured into saturated aqueous NH_4Cl (25 mL), and extracted with CH_2Cl_2 (3×25 mL). The combined organic layers were then washed with water (50 mL), dried over (anhydrous Na_2SO_4), and concentrated. The crude product was purified by flash column chromatography (silica gel) and eluted with 20% ethyl acetate-petroleum ether solvent system to give the desired product in high purity.

General procedure for the synthesis of functionalized β -amino sulfides/selenides

To a well-stirred solution of sulfamidate (0.2 mmol, 1 equiv) in DMF (2 mL), the disulfide/diselenide (0.1 mmol, 0.5 equiv) was added, followed by the addition of rongalite (0.6 mmol, 3.0 equiv) and K_2CO_3 (0.3 mmol, 1.5 equiv) respectively. The mixture was stirred

for 1–3 h at room temperature. The reaction was monitored by TLC. After complete consumption of the starting material, 2N HCl (3 mL) was added and the stirring was continued for further 12 h at room temperature. Finally the reaction mixture was neutralised by addition of ammonium hydroxide solution and extracted with DCM (5 mL $\times$ 3). The combined organic extract was washed with brine, dried over anhydrous sodium sulfate and concentrated under vacuum. The crude product was purified by silica gel (230–400 mesh) column chromatography.

Characterization of New Compounds:

Compound 2i

Yield 83% (92.7 mg); Foam solid; FTIR (Neat): 3736 (br), 2966 (m), 1748 (s, C=O), 1734 (s), 1540 (m), 1396 (m), 1286 (s), 1177 (s), 971 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 4.30 (s, 2H, OCH_2), 3.89 (s, 3H, OCH_3), 3.79 (s, 2H, NCH_2), 1.13 (s, 6H, $\text{C}(\text{CH}_3)_2$); ^{13}C NMR (100 MHz, CDCl_3): δ 152.7 (C=O), 82.4 (CH_2O), 57.1 (CH_2N), 54.7 (OCH_3), 31.6 ($\text{C}(\text{CH}_3)_2$), 22.0 (2CH_3); HRMS m/z : Calcd for $\text{C}_7\text{H}_{13}\text{NO}_5\text{SNa}$ ($\text{M}+\text{Na}$) $^+$: 246.0412; found: 246.0413.

Compound 4a

Yield 86% (46.6 mg); Pale yellow liquid; $[\alpha]_{\text{D}}^{22} = -13.7$ (c 2, CHCl_3); FTIR (Neat): 3338 (br, NH), 3061 (w), 3027 (w), 2961 (m), 2929 (m), 2874 (w), 1585 (w), 1458 (m), 1089 (w), 738 (s), 698 (s) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.34–7.14 (m, 10H, H-Ar), 3.79–3.71 (m, 2H, CH_2Ph), 3.11 (dd, $J = 13.1, 5.1$ Hz, 1H, CH_2S), 2.97 (dd, $J = 13.1, 6.8$ Hz, 1H, CH_2S), 2.74–2.68 (m, 1H, CH), 1.72 (bs, 1H, NH), 1.62–1.55 (m, 2H, CH_2CH_3), 0.91 (t, $J = 7.5$ Hz, 3H, CH_2CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 140.5, 136.6, 129.5, 128.9, 128.4, 128.1, 126.9, 126.0 (Ar-C), 57.0 (CH-N), 51.0 (CH_2Ph), 38.3 (C-S), 26.3 (CH_2CH_3), 9.9 (CH_2CH_3); HRMS m/z : Calcd for $\text{C}_{17}\text{H}_{21}\text{NSNa}$ ($\text{M}+\text{Na}$) $^+$: 294.1292; found: 294.1289.

Compound 4b

Yield 93% (53.0 mg); Oily liquid; $[\alpha]_{\text{D}}^{22} = -48.8$ (c 1, CHCl_3); FTIR (Neat): 3586 (m), 2957 (m), 2922 (s), 2853 (m), 1458 (w), 1090 (w), 1018 (s), 802 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.29–7.22 (m, 7H, H-Ar), 7.06 (d, $J = 7.9$ Hz, 2H, H-Ar), 3.77–3.69 (m, 2H, CH_2Ph), 3.06 (dd, $J = 13.1, 5.0$ Hz, 1H, CH_2S), 2.92 (dd, $J = 13.1, 6.9$ Hz, 1H, CH_2S), 2.69–2.63 (m, 1H, CH), 2.30 (s, 3H, CH_3Ar), 1.76 (bs, 1H, NH), 1.60–1.53 (m, 2H, CH_2CH_3), 0.90

(t, $J = 7.4$ Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 140.5, 136.2, 132.7, 130.4, 129.7, 128.4, 128.2, 126.9 (Ar-C), 57.1 (CH-N), 51.0 (CH₂Ph), 39.1 (C-S), 26.2 (CH₂CH₃), 21.0 (CH₃-Ar), 9.9 (CH₂CH₃); HRMS m/z : Calcd for C₁₈H₂₃NSH (M+H)⁺: 286.1629; found: 286.1628.

Compound 4c

Yield 95% (57.2 mg); Yellow gummy liquid; $[\alpha]_D^{22} = -14.9$ (c 1, CHCl₃); FTIR (Neat): 3336 (br, NH), 2960 (w), 1684 (m), 1522 (s), 1508 (s), 1245 (m), 1033 (m), 828 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.30–7.21 (m, 7H, H-Ar), 6.80 (d, $J = 8.7$ Hz, 2H, H-Ar), 3.77 (s, 3H, OMe), 3.76–3.67 (m, 2H, CH₂Ph), 3.00 (dd, $J = 13.2, 4.8$ Hz, 1H, CH₂S), 2.85 (dd, $J = 13.2, 7.1$ Hz, 1H, CH₂S), 2.64–2.58 (m, 1H, CH), 1.87 (bs, 1H, NH), 1.59–1.52 (m, 2H, CH₂CH₃), 0.88 (t, $J = 7.4$ Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 158.9, 140.5, 133.3, 128.4, 128.2, 126.9, 126.5, 114.6 (Ar-C), 56.9 (CH-N), 55.3 (OMe), 51.0 (CH₂Ph), 40.4 (C-S), 26.1 (CH₂CH₃), 9.8 (CH₂CH₃); HRMS m/z : Calcd for C₁₈H₂₃NOSH (M+H)⁺: 302.1579; found: 302.1572.

Compound 4d

Yield 90% (54.9 mg); Gummy liquid; $[\alpha]_D^{22} = -6.9$ (c 1, CHCl₃); FTIR (Neat): 3422 (br, NH), 2959 (m), 2924 (s), 2852 (m), 1656 (w), 1475 (m), 1095 (m), 1012 (w), 814 (w) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.33–7.22 (m, 9H, H-Ar), 3.80–3.72 (m, 2H, CH₂Ph), 3.07 (dd, $J = 13.1, 5.1$ Hz, 1H, CH₂S), 2.94 (dd, $J = 13.1, 6.8$ Hz, 1H, CH₂S), 2.71–2.65 (m, 1H, CH), 1.77 (bs, 1H, NH), 1.63–1.55 (m, 2H, CH₂CH₃), 0.91 (t, $J = 7.5$ Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 140.3, 135.2, 132.1, 131.0, 129.1, 128.5, 128.2, 127.1 (Ar-C), 56.9 (CH-N), 51.1 (CH₂Ph), 38.7 (C-S), 26.2 (CH₂CH₃), 9.9 (CH₂CH₃); HRMS m/z : Calcd for C₁₇H₂₀ClNSH (M+H)⁺: 306.1083; found: 306.1081.

Compound 4e

Yield 93% (64.9 mg); Yellow oily liquid; $[\alpha]_D^{22} = -21.1$ (c 1, CHCl₃); FTIR (Neat): 3415 (br, NH), 2961 (m), 2926 (m), 1594 (w), 1449 (s), 1428 (m), 1020 (s), 742 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.54 (d, $J = 7.9$ Hz, 1H, H-Ar), 7.31–7.00 (m, 8H, H-Ar), 3.79 (s, 2H, CH₂Ph), 3.11 (dd, $J = 12.7, 5.3$ Hz, 1H, CH₂S), 3.00 (dd, $J = 12.7, 6.6$ Hz, 1H, CH₂S), 2.80–2.74 (m, 1H, CH), 1.82 (bs, 1H, NH), 1.67–1.60 (m, 2H, CH₂CH₃), 0.94 (t, $J = 7.4$ Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 140.3, 138.0, 133.1, 129.0, 128.5, 128.2, 127.8,

127.03, 126.8, 124.3 (Ar-C), 56.8 (CH-N), 51.1 (CH₂Ph), 37.7 (C-S), 26.5 (CH₂CH₃), 10.0 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₇H₂₀BrNSH (M+H)⁺: 350.0578; found: 350.0577.

Compound 4f

Yield 87% (55.0 mg); Yellow liquid; [α]_D²² = -11.5 (*c* 1, CHCl₃); FTIR (Neat): 3434 (br, NH), 1648 (m), 1522 (w), 1034 (w) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.08 (d, *J* = 8.9 Hz, 2H, H-Ar), 7.31–7.23 (m, 7H, H-Ar), 3.85–3.78 (m, 2H, CH₂Ph), 3.18 (dd, *J* = 12.7, 5.4 Hz, 1H, CH₂S), 3.09 (dd, *J* = 12.7, 6.3 Hz, 1H, CH₂S), 2.86–2.80 (m, 1H, CH), 1.68–1.61 (m, 3H, NH & CH₂), 0.97 (t, *J* = 7.4 Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 147.8, 145.0, 140.1, 128.4, 128.1, 127.1, 126.5, 123.9 (Ar-C), 56.6 (CH-N), 51.0 (CH₂Ph), 36.6 (C-S), 26.4 (CH₂CH₃), 9.9 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₇H₂₀N₂O₂SH (M+H)⁺: 317.1324; found: 317.1327.

Compound 4g

Yield 85% (53.7 mg); Yellow liquid; [α]_D²² = +5.9 (*c* 1, CHCl₃); FTIR (Neat): 3394 (br, NH), 2961 (m), 2928 (m), 1516 (s), 1456 (w), 1338 (s), 1305 (m), 1106 (w), 733 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.15 (d, *J* = 8.2 Hz, 1H, H-Ar), 7.49 (t, *J* = 7.6 Hz, 1H, H-Ar), 7.39 (d, *J* = 8.2 Hz, 1H, H-Ar), 7.33–7.20 (m, 6H, H-Ar), 3.82 (s, 2H, CH₂Ph), 3.09 (dd, *J* = 12.3, 5.6 Hz, 1H, CH₂S), 3.03 (dd, *J* = 12.2, 6.2 Hz, 1H, CH₂S), 2.89–2.83 (m, 1H, CH), 1.71–1.63 (m, 3H, NH & CH₂), 0.97 (t, *J* = 8.2 Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 146.5, 140.2, 137.6, 133.3, 128.4, 128.1, 127.1, 127.0, 126.0, 124.5 (Ar-C), 56.3 (CH-N), 51.0 (CH₂Ph), 36.9 (C-S), 26.5 (CH₂CH₃), 9.9 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₇H₂₀N₂O₂SH (M+H)⁺: 317.1324; found: 317.1326.

Compound 4h

Yield 89% (50.9 mg); Yellow liquid; [α]_D²² = -37.3 (*c* 1, CHCl₃); FTIR (Neat): 3367 (br, NH), 2924 (m), 1648 (m), 1521 (m), 1273 (w), 1019 (s), 824 (w), 749 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.33–7.17 (m, 7H, H-Ar), 6.60–6.58 (m, 2H, H-Ar), 3.77–3.67 (m, 3H, NH & CH₂Ph), 2.97 (dd, *J* = 13.3, 4.7 Hz, 1H, CH₂S), 2.80 (dd, *J* = 13.1, 7.4 Hz, 1H, CH₂S), 2.63–2.57 (m, 1H, CH), 1.76 (bs, 2H, NH), 1.61–1.50 (m, 2H, CH₂CH₃), 0.87 (t, *J* = 7.4 Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 146.0, 140.6, 134.1, 128.5, 128.3, 127.0, 123.5, 115.7 (Ar-C), 57.1 (CH-N), 51.1 (CH₂Ph), 41.0 (C-S), 26.1 (CH₂CH₃), 9.9 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₇H₂₂N₂SH (M+H)⁺: 287.1582; found: 287.1588.

Compound 4i

Yield 89% (51.1 mg); Yellow liquid; $[\alpha]_{\text{D}}^{22} = -39.0$ (*c* 1, CHCl₃); FTIR (Neat): 3402 (br, NH), 2960 (m), 2923 (s), 2853 (m), 1582 (s), 1456 (m), 1258 (w), 101 (w), 890 (w) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.29–7.23 (m, 5H, H-Ar), 7.06 (t, *J* = 7.9 Hz, 1H, H-Ar), 6.81 (d, *J* = 7.6 Hz, 1H, H-Ar), 6.70 (bs, 1H, H-Ar), 6.57 (d, *J* = 7.8 Hz, 1H, H-Ar), 3.97 (bs, 2H, OH & NH), 3.82–3.71 (m, 2H, CH₂Ph), 3.11 (dd, *J* = 13.1, 4.5 Hz, 1H, CH₂S), 2.92 (dd, *J* = 13.1, 7.3 Hz, 1H, CH₂S), 2.74–2.68 (m, 1H, CH), 1.70–1.53 (m, 2H, CH₂CH₃), 0.90 (t, *J* = 7.3 Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 156.7, 139.5, 137.3, 130.0, 128.6, 128.4, 127.3, 121.3, 116.6, 113.9 (Ar-C), 57.1 (CH-N), 50.8 (CH₂Ph), 37.9 (C-S), 25.9 (CH₂CH₃), 10.1 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₇H₂₁ONSH (M+H)⁺: 288.1422; found: 288.1423.

Compound 4j

Yield 85% (56.1 mg); Yellow liquid; $[\alpha]_{\text{D}}^{22} = +0.12$ (*c* 1, CHCl₃); FTIR (Neat): 2960 (m), 2924 (m), 2854 (m), 1518 (s), 1455 (m), 1331 (m), 1291 (w), 1109 (w) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.95 (s, 1H, H-Ar), 7.31–7.22 (m, 7H, H-Ar), 3.81 (s, 2H, CH₂Ph), 3.08 (dd, *J* = 12.3, 5.5 Hz, 1H, CH₂S), 3.00 (dd, *J* = 12.3, 6.3 Hz, 1H, CH₂S), 2.86–2.80 (m, 1H, CH), 2.38 (s, 3H, CH₃-Ar), 1.93 (bs, 1H, NH), 1.71–1.60 (m, 2H, CH₂CH₃), 0.96 (t, *J* = 7.4 Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 146.8, 140.2, 135.3, 134.4, 133.7, 128.5, 128.2, 127.5, 127.1, 126.2 (Ar-C), 56.5 (CH-N), 51.1 (CH₂Ph), 37.1 (C-S), 26.5 (CH₂CH₃), 20.5 (CH₃-Ar), 9.9 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₈H₂₂N₂O₂SH (M+H)⁺: 331.1480; found: 331.1482.

Compound 4k

Yield 82% (44.6 mg); Yellow gummy liquid; $[\alpha]_{\text{D}}^{22} = -10.0$ (*c* 1, CHCl₃); FTIR (Neat): 3422 (br, NH), 2926 (s), 2101 (w), 1667 (br), 1265 (m), 1164 (w), 1122 (w), 1020 (m), 749 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.39 (d, *J* = 4.6 Hz, 1H, H-Pyr), 7.43 (t, *J* = 7.7 Hz, 1H), 7.32–6.93 (m, 7H, H-Ar & H-Pyr), 3.86–3.78 (m, 2H, CH₂Ph), 3.42 (dd, *J* = 13.4, 5.2 Hz, 1H, CH₂S), 3.35 (dd, *J* = 13.4, 5.9 Hz, 1H, CH₂S), 2.84–2.79 (m, 1H, CH), 1.81 (bs, 1H, NH), 1.63–1.56 (m, 2H, CH₂CH₃), 0.96 (t, *J* = 7.4 Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 159.0, 149.3, 140.7, 135.8, 128.3, 128.1, 126.8, 122.5, 119.3 (Ar-C & Pyr-C), 57.7 (CH-N), 51.0 (CH₂Ph), 33.8 (C-S), 26.6 (CH₂CH₃), 10.2 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₆H₂₀N₂SH (M+H)⁺: 273.1425; found: 273.1423.

Compound 4l

Yield 95% (53.6 mg); Yellow gummy liquid; $[\alpha]_{\text{D}}^{22} = -19.8$ (*c* 1, CHCl₃); FTIR (Neat): 3337 (br, NH), 2959 (m), 2925 (s), 1654 (br), 1491 (m), 1374 (s), 1254 (s), 1144 (m), 984 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.36–7.21 (m, 5H, H-Ar), 3.90–3.80 (m, 2H, CH₂Ph), 3.55 (s, 3H, N(CH₃)₂), 3.53 (d, *J* = 5.6 Hz, 2H, CH₂S), 3.38 (s, 3H, N(CH₃)₂), 2.91–2.85 (m, 1H, CH), 1.64–1.52 (m, 3H, NH & CH₂), 0.96 (t, *J* = 7.4 Hz, 3H, CH₂CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 197.5 (C=S), 140.6, 128.3, 128.2, 126.8 (Ar-C), 57.4 (CH-N), 51.0 (CH₂Ph), 45.5, 42.0, 41.5, 26.8 (CH₂CH₃), 10.2 (CH₂CH₃); HRMS *m/z*: Calcd for C₁₄H₂₂N₂S₂H (M+H)⁺: 283.1303; found: 283.1306.

Compound 4n

Yield 95% (54.2 mg); Colorless oily liquid; $[\alpha]_{\text{D}}^{25} = +84.6$ (*c* 1, CHCl₃); FTIR (Neat): 3421 (br, NH), 2957 (m), 2928 (m), 1656 (br), 1458 (w), 1265 (w), 1165 (w), 1025 (w), 738 (s), 699 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.33–7.15 (m, 10H, H-Ar), 3.75 (s, 2H, CH₂Ph), 3.12 (dd, *J* = 13.0, 4.5 Hz, 1H, CH₂S), 2.91 (dd, *J* = 13.0, 7.7 Hz, 1H, CH₂S), 2.59–2.55 (m, 1H, CH), 2.04–1.92 (m, 1H, CH), 1.63 (bs, 1H, NH), 0.93 (d, *J* = 2.8 Hz, 3H, CH₃), 0.92 (d, *J* = 2.8 Hz, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 140.8, 136.8, 129.6, 128.9, 128.4, 128.3, 126.9, 126.1 (Ar-C), 61.1 (CH-N), 51.8 (CH₂Ph), 36.0 (C-S), 29.9 (CH(CH₃)₂), 18.6 (CH₃), 18.1 (CH₃); HRMS *m/z*: Calcd for C₁₈H₂₃NSH (M+H)⁺: 286.1629; found: 286.1625.

Compound 4o

Yield 94% (56.3 mg); Yellow oily liquid; $[\alpha]_{\text{D}}^{22} = +17.3$ (*c* 1, CHCl₃); FTIR (Neat): 3416 (br, NH), 2954 (s), 2923 (s), 2867 (m), 1644 (br), 1456 (m), 1018 (s), 737 (s), 691 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.35–7.16 (m, 10H, H-Ar), 3.79–3.70 (m, 2H, CH₂Ph), 3.09 (dd, *J* = 13.0, 5.2 Hz, 1H, CH₂S), 3.00 (dd, *J* = 13.0, 6.1 Hz, 1H, CH₂S), 2.84–2.78 (m, 1H, CH), 1.73–1.63 (m, 2H), 1.48–1.35 (m, 2H), 0.84 (d, *J* = 6.7 Hz, 3H, CH₃), 0.82 (d, *J* = 6.6 Hz, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 140.5, 136.7, 129.9, 129.0, 128.5, 128.3, 127.0, 126.2 (Ar-C), 53.9 (CH-N), 50.9 (CH₂Ph), 43.5 (CH₂), 39.3 (C-S), 25.0 (CH(CH₃)₂), 23.0 (CH₃), 22.8 (CH₃); HRMS *m/z*: Calcd for C₁₉H₂₅NSH (M+H)⁺: 300.1786; found: 300.1788.

Compound 4p

Yield 92% (61.3 mg); Pale yellow liquid; $[\alpha]_{\text{D}}^{22} = +12.5$ (*c* 1, CHCl₃); FTIR (Neat): 3412 (br, NH), 3026 (m), 2921 (m), 1584 (m), 1454 (m), 1090 (m), 1026 (m), 738 (s), 699 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.29–7.12 (m, 15H, H-Ar), 3.77 (s, 2H, NCH₂Ph), 3.06–2.86 (m,

5H), 1.88 (bs, 1H, NH); ^{13}C NMR (100 MHz, CDCl_3): δ 140.1, 138.6, 136.4, 129.4, 129.4, 128.9, 128.5, 128.4, 128.1, 127.0, 126.4, 126.0 (Ar-C), 57.4 (CH-N), 51.3 (NCH_2Ph), 40.1, 38.0; HRMS m/z : Calcd for $\text{C}_{22}\text{H}_{23}\text{NSH}$ ($\text{M}+\text{H}$) $^+$: 334.1629; found: 334.1628.

Compound 4q

Yield 82% (49.4 mg); Yellow oily liquid; $[\alpha]_{\text{D}}^{24} = -0.5$ (c 1, CHCl_3); FTIR (Neat): 3412(br, NH), 2922(w), 1636 (m), 1408 (w), 1017 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.36–7.18 (m, 10H, H-Ar), 3.84 (d, $J = 13.3$ Hz, 1H, CH_2Ph), 3.68 (d, $J = 13.1$ Hz, 1H, CH_2Ph), 3.62 (s, 3H, OCH_3), 3.50 (t, $J = 6.2$ Hz, 1H), 3.24–3.22 (m, 2H), 1.98 (bs, 1H, NH); ^{13}C NMR (100 MHz, CDCl_3): δ 173.8 (C=O), 139.4, 135.2, 130.5, 129.0, 128.5, 128.3, 127.2, 126.7 (Ar-C), 59.7 (CH-N), 52.0, 51.9, 37.5 (C-S); HRMS m/z : Calcd for $\text{C}_{17}\text{H}_{19}\text{NO}_2\text{SH}$ ($\text{M}+\text{H}$) $^+$: 302.1215; found: 302.1209.

Compound 4r

Yield 80% (43.4 mg); Yellow oily liquid; FTIR (Neat): 3060 (br), 2965 (s), 2924 (s), 2852 (m), 1739 (w), 1584 (m), 1479 (s), 1439 (m), 1380 (m), 1257 (w), 1184 (m), 1018 (s), 809 (w) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.42–7.14 (m, 10H, H-Ar), 3.65 (s, 2H, CH_2Ph), 3.16 (s, 2H, CH_2S), 1.63 (bs, 1H, NH), 1.24 (s, 6H, 2 CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 140.9, 137.7, 129.5, 128.9, 128.4, 128.3, 126.9, 125.9 (Ar-C), 53.9 (C-N), 46.9 (CH_2Ph), 45.7 (C-S), 27.2 (2 CH_3); HRMS m/z : Calcd for $\text{C}_{17}\text{H}_{21}\text{NSH}$ ($\text{M}+\text{H}$) $^+$: 272.1473; found: 272.1471.

Compound 4s

Yield 82% (54.3 mg); Dark brown gummy liquid; $[\alpha]_{\text{D}}^{22} = +38.7$ (c 1, CHCl_3); FTIR (Neat): 3061 (m), 3028 (m), 2925 (m), 2851 (m), 1584 (m), 1479 (m), 1457 (m), 1091 (w), 745 (s), 698 (s) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.46–7.17 (m, 14H, H-Ar), 4.29 (d, $J = 5.2$ Hz, 1H, CH-N), 3.93–3.79 (m, 3H), 3.49 (dd, $J = 16.4, 7.5$ Hz, 1H, CH_2), 2.91 (dd, $J = 16.4, 7.4$ Hz, 1H, CH_2), 1.72 (bs, 1H, NH); ^{13}C NMR (100 MHz, CDCl_3): δ 143.2, 141.2, 140.5, 135.5, 131.6, 129.1, 128.5, 128.2, 128.0, 127.0, 127.0, 126.9, 124.8, 124.7 (Ar-C), 68.6 (CH-N), 51.8 (CH_2Ph), 50.7 (CH-S), 38.5 (CH_2); HRMS m/z : Calcd for $\text{C}_{22}\text{H}_{21}\text{NSH}$ ($\text{M}+\text{H}$) $^+$: 332.1473; found: 332.1485.

Compound 4t

Yield 84% (53.0 mg); Colorless gummy liquid; $[\alpha]_{\text{D}}^{22} = -12.5$ (c 1, CHCl_3); FTIR (Neat): 3332 (br, NH), 2924 (m), 1693 (s, C=O), 1607 (m), 1441 (w), 1233 (m), 1087 (s), 1016 (s),

738 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.38–7.17 (m, 10H, H-Ar), 5.07 (s, 2H, CH_2Ph), 4.83–4.82 (m, 1H), 3.81–3.80 (m, 1H), 3.16–3.05 (m, 2H), 1.74–1.48 (m, 2H, CH_2CH_3), 0.91 (t, $J = 7.4$ Hz, 3H, CH_2CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 156.0 (C=O), 136.5, 136.2, 129.5, 129.1, 128.6, 128.2, 128.1, 126.3 (Ar-C), 66.7 (CH_2Ph), 52.3 (CH-N), 38.7 ($\text{CH}_2\text{-S}$), 26.7 (CH_2CH_3), 10.4 (CH_2CH_3); HRMS m/z : Calcd for $\text{C}_{18}\text{H}_{21}\text{NO}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$: 338.1191; found: 338.1195.

Compound 4u

Yield 85% (43.0 mg); Colorless gummy liquid; FTIR (Neat): 3344 (br, NH), 2961 (m), 2926 (m), 1718 (s), 1708 (s), 1527 (m), 1466 (w), 1249 (m), 738 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.36 (d, $J = 7.6$ Hz, 2H, H-Ar), 7.29–7.15 (m, 3H, H-Ar), 4.76 (bs, 1H, NH), 3.65 (s, 3H, OCH_3), 3.15 (d, $J = 6.6$ Hz, 2H, CH_2N), 2.90 (s, 2H, CH_2S), 1.01 (s, 6H, 2 CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 157.5 (C=O), 137.5, 129.4, 129.0, 126.1 (Ar-C), 52.2 (OCH_3), 50.0 (CH-N), 44.8 ($\text{CH}_2\text{-S}$), 36.6 ($\text{C}(\text{CH}_3)_2$), 24.9 (2 CH_3); HRMS m/z : Calcd for $\text{C}_{13}\text{H}_{19}\text{NO}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$: 276.1034; found: 276.1034.

Compound 5a

Yield 90% (57.3 mg); Pale yellow oily liquid; $[\alpha]_{\text{D}}^{22} = -31.7$ (c 1, CHCl_3); FTIR (Neat): 3394 (br, NH), 2959 (m), 2925 (s), 2853 (m), 1457 (m), 735 (s), 692 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.50–7.22 (m, 10H, H-Ar), 3.74 (s, 2H, CH_2Ph), 3.16 (dd, $J = 12.2, 5.0$ Hz, 1H, CH_2Se), 3.00 (dd, $J = 12.2, 6.5$ Hz, 1H, CH_2Se), 2.74–2.68 (m, 1H, CH-N), 1.69 (bs, 1H, NH), 1.62–1.54 (m, 2H, CH_2CH_3), 0.90 (t, $J = 7.4$ Hz, 3H, CH_2CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 140.5, 132.9, 130.6, 129.1, 128.4, 128.2, 127.0, 126.9 (Ar-C), 57.7 (CH-N), 51.1 (CH_2Ph), 33.7 (CH_2Se), 26.9 (CH_2CH_3), 10.1 (CH_2CH_3); ^{77}Se NMR (100 MHz, CDCl_3): δ 241.2; HRMS m/z : Calcd for $\text{C}_{17}\text{H}_{21}\text{NSeNa}$ ($\text{M}+\text{Na}$) $^+$: 342.0737; found: 342.0736.

Compound 5b

Yield 92% (61.1 mg); Yellow oily liquid; $[\alpha]_{\text{D}}^{22} = +89.2$ (c 1, CHCl_3); FTIR (Neat): 3306 (br, NH), 3027 (w), 2956 (s), 2926 (s), 1458 (m), 1022 (m), 735 (s) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.48–7.21 (m, 10H, H-Ar), 3.73 (s, 2H, CH_2Ph), 3.13 (dd, $J = 12.1, 4.6$ Hz, 1H, CH_2Se), 2.96 (dd, $J = 12.1, 7.6$ Hz, 1H, CH_2Se), 2.59–2.55 (m, 1H, CH), 2.01–1.90 (m, 1H, CH), 1.61 (bs, 1H, NH), 0.90 (d, $J = 6.9$ Hz, 6H, 2 CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 140.7, 132.8, 130.7, 129.0, 128.3, 128.2, 126.9, 126.8, (Ar-C), 61.8 (CH-N), 51.6 (CH_2Ph),

31.2 (C-Se), 30.2 (CH(CH₃)₂), 18.7 (CH₃), 18.1 (CH₃); ⁷⁷Se NMR (100 MHz, CDCl₃): δ 247.1; HRMS *m/z*: Calcd for C₁₈H₂₃NSeH (M+H)⁺: 334.1074; found: 334.1076.

Compound 5c

Yield 91% (63.0 mg); Yellow oily liquid; [α]_D²² = +14.2 (*c* 1, CHCl₃); FTIR (Neat): 3436 (br), 2955 (w), 2088 (w), 1645 (m), 1458 (w), 1018 (w) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.50–7.22 (m, 10H, H-Ar), 3.76–3.67 (m, 2H, CH₂Ph), 3.14 (dd, *J* = 12.1, 5.1 Hz, 1H, CH₂Se), 3.01 (dd, *J* = 12.1, 5.8 Hz, 1H, CH₂Se), 2.84–2.79 (m, 1H, CH), 1.70–1.60 (m, 2H), 1.47–1.32 (m, 2H), 0.83 (d, *J* = 6.8 Hz, 3H, CH₃), 0.81 (d, *J* = 6.8 Hz, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ 140.5, 133.1, 130.7, 129.1, 128.4, 128.3, 127.0, 126.9 (Ar-C), 54.3 (CH-N), 50.9 (CH₂Ph), 44.1 (CH₂), 34.6 (CH₂-Se), 25.0 (CH(CH₃)₂), 22.9 (CH₃), 22.8 (CH₃); ⁷⁷Se NMR (100 MHz, CDCl₃): δ 239.7; HRMS *m/z*: Calcd for C₁₉H₂₅NSeH (M+H)⁺: 348.1230; found: 348.1229.

Compound 5d

Yield 89% (67.7 mg); Colorless oily liquid; [α]_D²² = +7.3 (*c* 1, CHCl₃); FTIR (Neat): 3402 (br), 3026 (w), 2924 (w), 1578 (w), 1477 (m), 1454 (m), 1073 (w), 1022 (w), 736 (s), 699 (s) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.41–7.10 (m, 15H, H-Ar), 3.79–3.71 (m, 2H, NCH₂Ph), 3.11–2.81 (m, 5H), 1.80 (bs, 1H, NH); ¹³C NMR (100 MHz, CDCl₃): δ 140.1, 138.7, 132.6, 130.5, 129.4, 129.1, 128.5, 128.4, 128.1, 127.0, 126.8, 126.4 (Ar-C), 57.9 (CH-N), 51.2 (NCH₂Ph), 40.7 (CH-N), 33.2 (C-Se); ⁷⁷Se NMR (100 MHz, CDCl₃): δ 241.7; HRMS *m/z*: Calcd for C₂₂H₂₃NSeH (M+H)⁺: 382.1074; found: 382.1075.

Compound 5e

Yield 80% (55.7 mg); Yellow gummy liquid; [α]_D²² = –3.7 (*c* 1, CHCl₃); FTIR (Neat): 3412 (br), 2924 (m), 2854 (w), 1633 (w), 1446 (w), 1082 (w), 1018 (m) cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.51–7.23 (m, 10H, H-Ar), 3.85 (d, *J* = 13.1 Hz, 1H, CH₂Ph), 3.70 (d, *J* = 13.1 Hz, 1H, CH₂Ph), 3.60–3.55 (m, 4H), 3.21 (d, *J* = 6.2 Hz, 2H), 3.03 (bs, 1H, NH); ¹³C NMR (100 MHz, CDCl₃): δ 173.7 (C=O), 139.2, 133.4, 129.6, 129.2, 128.5, 128.4, 127.4, 127.3 (Ar-C), 60.1 (CH-N), 52.0, 51.9, 31.0 (C-Se); ⁷⁷Se NMR (100 MHz, CDCl₃): δ 265.3; HRMS *m/z*: Calcd for C₁₇H₁₉NO₂SeH (M+H)⁺: 350.0659; found: 350.0656.

Compound 5f

Yield 79% (50.3 mg); Yellow gummy liquid; FTIR (Neat): 3060 (w), 2965 (s), 2927 (s), 2853 (m), 1579 (w), 1476 (m), 1226 (w), 1155 (w), 1022 (m), 735 (s), 692 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.54–7.22 (m, 10H, H-Ar), 3.64 (s, 2H, CH_2Ph), 3.20 (s, 2H, CH_2Se), 1.55 (bs, 1H, NH), 1.25 (s, 6H, 2CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 140.9, 132.7, 131.4, 129.1, 128.4, 128.3, 126.9, 126.7 (Ar-C), 53.7 (C-N), 46.9 (CH_2Ph), 41.9 (C-Se), 27.5 (2CH_3); ^{77}Se NMR (100 MHz, CDCl_3): δ 232.3; HRMS m/z : Calcd for $\text{C}_{17}\text{H}_{21}\text{NSeH}$ ($\text{M}+\text{H}$) $^+$: 320.0917; found: 320.0918.

Compound 5g

Yield 81% (61.3 mg); Dark brown gummy liquid; $[\alpha]_{\text{D}}^{22} = +38.7$ (c 1, CHCl_3); FTIR (Neat): 3585 (m), 2922 (w), 1742 (w), 1380 (w), 1308(w), 1193 (w), 1018 (s) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.59–7.16 (m, 14H, Ar-H), 4.34 (d, $J = 5.2$ Hz, 1H, CH-N), 3.95–3.90 (m, 1H), 3.84–3.76 (m, 2H), 3.50 (dd, $J = 16.5, 7.6$ Hz, 1H, CH_2), 2.98 (dd, $J = 16.5, 6.1$ Hz, 1H, CH_2), 1.63 (bs, 1H, NH); ^{13}C NMR (100 MHz, CDCl_3): δ 143.5, 141.7, 140.6, 134.8, 129.4, 129.2, 128.5, 128.2, 128.0, 127.7, 127.0, 127.0, 124.7, 124.6 (Ar-C), 69.1 (CH-N), 50.7 (CH_2Ph), 46.5 (C-Se), 38.7 (CH_2); ^{77}Se NMR (100 MHz, CDCl_3): δ 371.6; HRMS m/z : Calcd for $\text{C}_{22}\text{H}_{21}\text{NSeH}$ ($\text{M}+\text{H}$) $^+$: 380.0917; found: 380.0925.

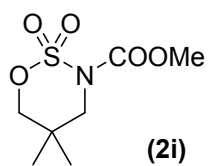
Compound 5h

Yield 83% (60.1 mg); Yellow oily liquid; $[\alpha]_{\text{D}}^{22} = -9.5$ (c 1, CHCl_3); FTIR (Neat): 3322 (br), 2965 (m), 2934 (m), 1704 (s), 1694 (s), 1517 (m), 1276 (m), 1236 (s), 1073 (m), 736 (s) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.52–7.22 (m, 10H, H-Ar), 5.09–5.02 (m, 2H, CH_2Ph), 4.83 (d, $J = 6.8$ Hz, 1H), 3.83–3.81 (bs, 1H), 3.11 (bs, 2H), 1.68–1.47 (m, 2H, CH_2CH_3), 0.89 (t, $J = 7.4$ Hz, 3H, CH_2CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 155.9 (C=O), 136.6, 132.8, 130.2, 129.2, 128.6, 128.1, 128.1, 127.1 (Ar-C), 66.7 (CH_2Ph), 52.7 (CH-N), 33.7 (C-Se), 27.6 (CH_2CH_3), 10.4 (CH_2CH_3); ^{77}Se NMR (100 MHz, CDCl_3): δ 240.6; HRMS m/z : Calcd for $\text{C}_{18}\text{H}_{21}\text{NSeO}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 386.0635; found: 386.0640.

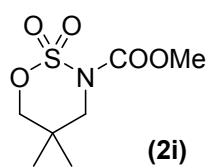
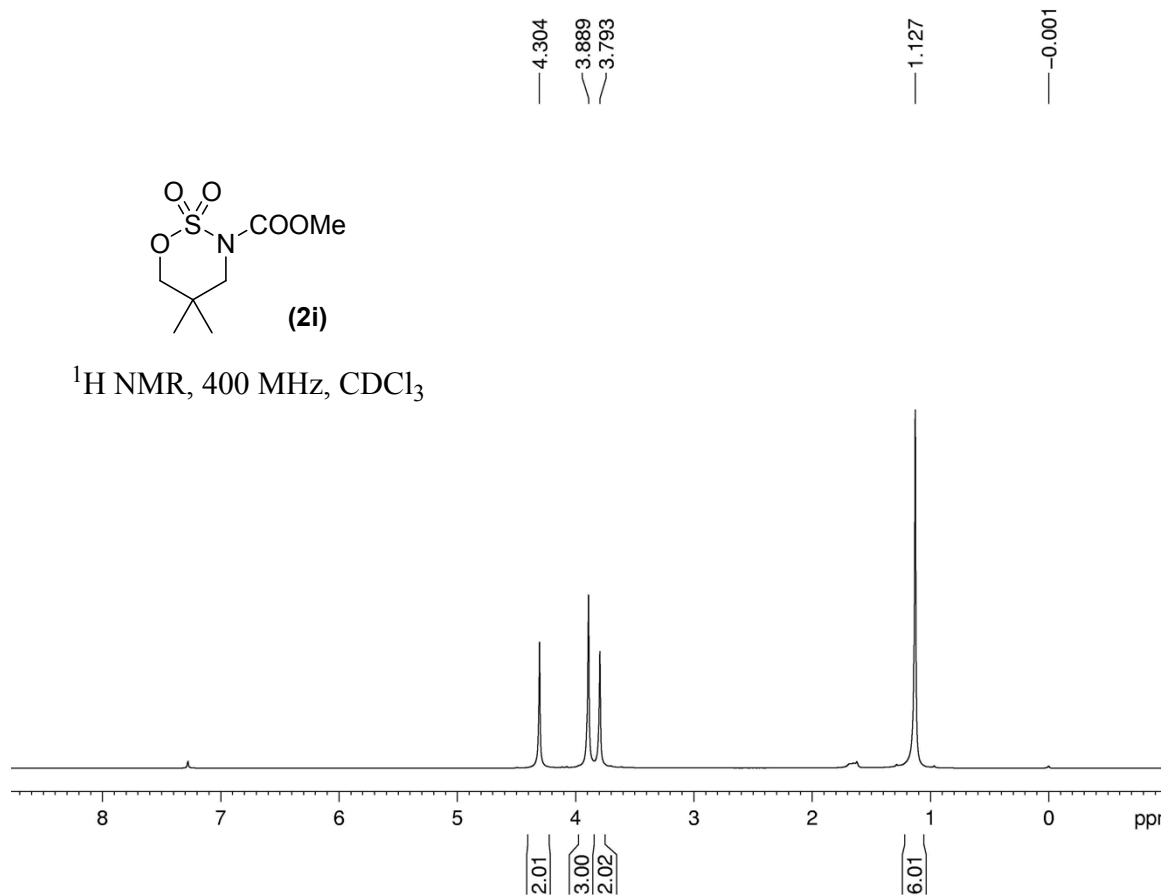
Compound 5i

Yield 81% (48.6 mg); Colorless oily liquid; FTIR (Neat): 3344 (br, NH), 2960 (m), 1707 (s), 1526 (m), 1477 (w), 1248 (m), 1023 (w), 737 (m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.54–7.24 (m, 5H, H-Ar), 4.66 (bs, 1H, NH), 3.64 (s, 3H, OCH_3), 3.13 (d, $J = 6.6$ Hz, 2H, CH_2N), 2.91 (s, 2H, CH_2Se), 1.00 (s, 6H, 2CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ 157.4 (C=O), 132.9, 131.2, 129.2, 127.0 (Ar-C), 52.2 (OCH_3), 50.4 (CH-N), 40.4 ($\text{CH}_2\text{-Se}$), 36.6

(C(CH₃)₂), 25.3 (2CH₃); ⁷⁷Se NMR (100 MHz, CDCl₃): δ 234.9; HRMS *m/z*: Calcd for C₁₃H₁₉NO₂SeNa (M+Na)⁺: 324.0479; found: 324.0473.



^1H NMR, 400 MHz, CDCl_3



^{13}C NMR, 100 MHz, CDCl_3

