Supplementary Material (ESI) for Organic and Biomolecular Chemistry

Electronic Supplementary Information

Synthesis, characterization and structures of 2-(3,5-dimethylpyrazol-1-yl)ethylseleno derivatives and their probable glutathione peroxidase (GPx) like activity

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1. Synthesis and characterization data of compounds 4 - 14

dmpz$CH_2CH_2SeCH_2CH_2COOH$ (4)

Colorless crystals (0.8 g, 59 %), m.p. 132-133 ^oC. Anal. Calcd. for $C_{10}H_{16}N_2O_2Se_1$: Calcd.: C, 43.64; H, 5.86; N, 10.18 %. Found: C, 43.78; H, 5.93; N, 9.67 %. IR (neat) 3585 (υ OH), 1710 (υ CO). NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.21, 2.24 (each 3H, s, -C*H*$_3$), 2.75-2.91 (6H, m, -C*H*$_2$), 4.28 (2H, t, *J* = 8.0 Hz, -NC*H*$_2$), 5.80 (1H, s, H-4 dmpz), 8.10 (br, O*H*); $^{13}C\{^1H\}$ NMR: δ 10.9, 12.7 (each s, *Me*), 17.7 ($^1J_{C\text{-}Se}$ = 62.0 Hz, Se*C*H$_2$); 22.2 ($^1J_{C\text{-}Se}$ = 68.0 Hz, Se*C*H$_2$); 35.9 (*C*H$_2$COOH), 49.0 (N*C*H$_2$), 105.4, (*C*-4 dmpz), 139.3, 147.6 (each s, *C*-3, 5 dmpz), 175.1 (CO); $^{77}Se\{^1H\}$ NMR: δ 166.9 ppm. MS (IT) *m*/*z*: 299 $[M + Na]^+$, 277 $[M + H]^+$, 203 $[dmpzCH_2CH_2Se]^+$; 152 $[SeCH_2CH_2COOH]^+$.

dmpz$CH_2CH_2SeCH_2CH_2CH_2COOH$ (5)

Colorless solid (0.042 g, 60 %), m.p. 69-70 ^oC. IR (neat) 3080 (υ OH), 1710 (υ CO); NMR spectral data ($CDCl_3$): ^1H NMR: δ 1.96 (m, $SeCH_2CH_2CH_2CO$), 2.20, 2.25 (each 3H, s, -C*H*$_3$), 2.43 (2H, t, *J* = 7.2 Hz, $SeCH_2CH_2CH_2CO$), 2.53 (2H, t, *J* = 7.3 Hz, $SeCH_2CH_2CH_2CO$), 2.90 (2H, t, *J* = 7.3 Hz, NCH_2CH_2Se), 4.24 (2H, t, *J* = 7.5 Hz, NC*H*$_2$), 5.79 (1H, s, H-4 dmpz), 6.59 (br, O*H*); $^{13}C\{^1H\}$ NMR: δ 11.2, 13.2 (each s, *Me*), 22.8 ($^1J_{C\text{-}Se}$ = 67.0 Hz), 23.2 ($^1J_{C\text{-}Se}$ = 63.0 Hz) (each Se*C*H$_2$), 25.9 (CH_2CH_2CO), 34.2 (*C*H$_2$CO), 49.2 (N*C*H$_2$), 105.4 (*C*-4 dmpz), 139.4, 147.9 (each s, *C*-3, 5 dmpz), 176.7 (CO); $^{77}Se\{^1H\}$ NMR: δ 148.9 ppm. MS (IT) *m*/*z*: 313 $[M + Na]^+$, 289 $[M + H]^+$, 203 $[dmpzCH_2CH_2Se]^+$, 166 $[SeCH_2CH_2CH_2COOH]^+$.

dmpz$CH_2CH_2SeCH_2COOCH_2CH_3$ (6)

Pale yellow liquid (0.1 g, 84 %). IR (neat): 1730 (υ CO); NMR spectral data ($CDCl_3$): ^1H NMR: δ 1.23 (3H, t, *J* = 7.0 Hz, -$COOCH_2CH_3$), 2.16, 2.22 (each 3H, s, -C*H*$_3$); 3.03 (Se*C*H$_2$CO), 3.07 (t, *J* = 7.0 Hz, Se*C*H$_2$), 4.12 (2H, q, *J* = 7.0 Hz, -$COOCH_2CH_3$), 4.22 (2H, t, *J* = 7.0 Hz, NC*H*$_2$), 5.74 (1H, s, H-4 dmpz); $^{13}C\{^1H\}$ NMR: δ 11.0, 13.5 (each s, *Me*), 14.1 (CH_2Me), 22.5 ($^1J_{C\text{-}Se}$ = 69.0 Hz), 24.7 ($^1J_{C\text{-}Se}$ = 67.0 Hz) (each Se*C*H$_2$), 48.6 (N*C*H$_2$), 61.3 ($COOCH_2$), 105.1 (*C*-4 dmpz), 138.9, 147.8 (each s, *C*-3, 5 dmpz), 171.4 (CO); $^{77}Se\{^1H\}$ NMR δ: 202.4 ppm. MS (IT) *m*/*z*: 291 $[M + H]^+$, 261 $[M - Et]^+$, 203 $[dmpzCH_2CH_2Se]^+$.

dmpz$CH_2CH_2SeCH_2CH_2COOCH_2CH_3$ (7)

Pale yellow liquid (0.095 g, 86%). IR (neat): 1730 (υ CO); NMR spectral data ($CDCl_3$): ^1H NMR: δ 1.25 (3H, t, *J* = 7.1 Hz, -$COOCH_2CH_3$), 2.20, 2.26 (each 3H, s, -C*H*$_3$), 2.64 (4H, s, Se*C*H$_2$*C*H$_2$COO), 2.96 (2H, t, *J* = 7.2 Hz, NC*H*$_2$), 4.13 (2H, q, *J* = 7.2 Hz, -$COOCH_2CH_3$), 4.20 (2H, t, *J* = 7.2 Hz, NCH_2CH_2Se), 5.78 (1H, s, H-

4 dmpz); $^{13}C\{^1H\}$ NMR: δ 11.0, 13.3 (each s, *Me*), 14.0 ($COOCH_2Me$), 17.6 ($^1J_{C\text{-}Se}$ = 63.0 Hz, $SeCH_2CH_2CO$), 23.3 ($^1J_{C\text{-}Se}$ = 67.0 Hz, $SeCH_2CH_2NH_2$), 35.5 (CH_2CO), 49.0 (NCH_2), 60.5 (OCH_2), 104.9 (*C*-4 dmpz), 138.9, 147.7 (C-3, 5 dmpz), 172.0 (CO); $^{77}Se\{^1H\}$ NMR: δ 171.1 ppm. MS (IT) *m/z*: 327 $[M + Na]^+$, 305 $[M + H]^+$, 275 $[M - Et]^+$, 207 [M-dmpz]$^+$, 179 $[SeCH_2CH_2COOEt]^+$.

dmpz$CH_2CH_2SeCH_2CH_2CH_2COOCH_2CH_3$ (8)

Pale yellow liquid (0.58 g, 91%). IR (neat): 1730 (υ CO); NMR spectral data ($CDCl_3$): ^1H NMR: δ 1.22 (3H, t, *J* = 7.0, -$COOCH_2CH_3$), 1.89 (m,$SeCH_2CH_2$), 2.17, 2.23 (6H, each s, -CH_3), 2.34 (2H, t, *J* = 7.0 Hz, -$SeCH_2$), 2.45 (2H, t, *J* = 7.3 Hz, -CH_2CO), 2.89 (2H, t, *J* = 7.2 Hz, CH_2Se-), 4.08 (2H, qr, *J* = 7.1 Hz, -$COOCH_2CH_3$), 4.16 (2H, t, *J* = 7.3 Hz, NCH_2), 5.75 (1H, s, H-4 dmpz); $^{13}C\{^1H\}$ NMR: δ 11.2, 13.5 (each s, *Me*), 14.2 ($COCH_2Me$), 23.1 ($^1J_{C\text{-}Se}$ = 67.0), 23.2 ($^1J_{C\text{-}Se}$ = 62.0 Hz) (each $SeCH_2$), 25.6 (-CH_2-), 34.0 (CH_2CO), 49.3 (NCH_2), 60.4 ($COCH_2$), 105.0 (C-4 dmpz), 138.9, 147.6 (C-3, 5 dmpz), 172.9 (CO); $^{77}Se\{^1H\}$ NMR: δ 147.0 ppm. MS (IT) *m/z*: 341 $[M + Na]^+$, 319 $[M + H]^+$, 289 $[M - Et]^+$, 221 [M-dmpz]$^+$, 193 $[SeCH_2CH_2CH_2COOEt]^+$.

dmpz$CH_2CH_2SeCH_2Ph$ (9)

Pale yellow liquid (0.55 g, 76 %). Anal. Calcd. for $C_{14}H_{18}N_2Se$: Calcd.: C, 57.34; H, 6.19; N, 9.55 %. Found: C, 57.11; H, 6.21; N, 9.31 %. NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.18, 2.21 (each 3H, s, -CH_3), 2.87 (2 H, t, *J* = 7.1 Hz, -CH_2Se), 3.59 (2H, s, $J_{Se\text{-}H}$ = 6.3 Hz, -$SeCH_2Ph$), 4.12 (2H, t, *J* 7.1, -CH_2N), 5.77 (1H, s, H-4 dmpz), 7.19-7.30 (5 H, m, *Ph-H*); $^{13}C\{^1H\}$ NMR: δ 10.9, 13.3 (each s, *Me*), 23.1 ($^1J_{C\text{-}Se}$ = 69.0 Hz, $SeCH_2$), 26.9 ($^1J_{C\text{-}Se}$ = 59.0 Hz, $SeCH_2Ph$), 48.9 (NCH_2), 104.7 (C-4 dmpz), 126.6, 128.3, 128.6, 138.8 (Ph); 138.9, 147.3 (each s, C-3, 5 dmpz); $^{77}Se\{^1H\}$ NMR: δ 244.6 ppm. MS (IT) *m/z*: 317 $[M + Na]^+$, 295 $[M + H]^+$, 267, 197.

dmpz$CH_2CH_2SeCH_2CH{=}CH_2$ (10)

Pale yellow liquid (0.12 g, 67 %). NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.20, 2.25 (each 3H, s, -CH_3), 2.87 (2H, t, *J* = 7.2 Hz, -CH_2Se); 3.02 (2H, d, *J* = 4.5 Hz, -$CH_2CH{=}CH_2$), 4.18 (2H, t, *J* = 7.2 Hz, -CH_2N), 4.98-5.05 (2H, m, -$CH_2CH{=}CH_2$), 5.77 (1H, s, H-4 dmpz); 5.84 (1H, m, -$CH_2CH{=}CH_2$); $^{13}C\{^1H\}$ NMR: δ 11.1, 13.4 (each s, *Me*), 22.4 ($^1J_{C\text{-}Se}$ = 69.0 Hz, $SeCH_2$), 26.0 ($^1J_{C\text{-}Se}$ = 57.0 Hz, $SeCH_2{=}CH$), 49.0 (NCH_2), 105.0 (*C*-4 dmpz), 116.6 ($CH_2{=}$), 134.6 (=*C*H), 139.0, 147.5 (each s, C-3, 5 dmpz); $^{77}Se\{^1H\}$ NMR: δ 182 ppm. MS (IT) *m/z*: 267 $[M + Na]^+$, 245 $[M + H]^+$, 203 $[dmpzCH_2CH_2Se]^+$.

dmpz$CH_2CH_2SeCH_2CH_2NH_2$ (11)

Yellow liquid (0.146 g, 24 %). IR (neat): 3350 (υ NH_2). NMR spectral data ($CDCl_3$): ^{1}H NMR: δ 2.12, 2.18 (each 3H, s, -CH_3), 2.66 (2H, t, *J* = 6.6 Hz, SeC$H_2$$CH_2NH_2$), 2.89 ((2H, t, *J* = 7.4 Hz, SeC$H_2$$CH_2$dmpz), 3.00 ((2H, t, *J* = 6.6 Hz, $SeCH_2$C$H_2$$NH_2$), 4.17 (2H, t, *J* = 7.4 Hz, -$SeCH_2$CH_2dmpz), 4.68 (2H, s, br, -NH_2), 5.71 (1H, s, H-4 dmpz); ^{13}C{^{1}H} NMR: δ 11.0, 13.2 (each s, *Me*), 23.0 ($^1J_{C\text{-}Se}$ = 66.0 Hz) 24.4 ($^1J_{C\text{-}Se}$ = 62.0 Hz) (each Se*C*H_2), 40.9 (*C*H_2NH_2), 48.8 (N*C*H_2), 104.8 (*C*-4 dmpz), 139.0, 147.4 (each s, *C*-3, 5 dmpz); ^{77}Se{^{1}H} NMR: δ 118.9 ppm. MS (IT) *m*/*z*: 288 $[M + K]^+$, 248 $[M + H]^+$, 203 [dmpz$CH_2CH_2Se]^+$.

dmpz$CH_2CH_2SeCH_2CH_2NH_2$. 2HCl (12)

To a stirred methanol solution (25 ml) of dmpz$CH_2CH_2SeCH_2CH_2NH_2$ (0.14 g, 0.56 mmol), concentrated HCl was added till solution became acidic. The reaction mixture was stirred at ~ 45-50 ^{o}C for 2 h and then allowed to cool to room temperature. The solvent was evaporated *in vacuo* to yield a hygroscopic colorless solid (0.096 g, 60 %), m.p. 163-164 °C. IR: 3260 (υ NH_2). NMR spectral data (D_2O): ^{1}H NMR: δ 2.18, 2.23 (each 3H, s, -CH_3), 2.59 (2H, t, *J* = 7.0 Hz, -CH_2Se), 2.87 (2H, t, *J* = 6.7 Hz, -C$H_2$$CH_2NH_2$), 3.05 (2H, t, *J* = 6.6 Hz, -CH_2C$H_2$$NH_2$), 4.36 (2H, t, *J* = 6.9 Hz, -CH_2N), 6.15 (1H, s, Ar-*H*); ^{13}C{^{1}H} NMR: δ 10.1, 10.3 (each s, *Me*), 19.6 ($^1J_{C\text{-}Se}$ = 67.0 Hz), 21.6 ($^1J_{C\text{-}Se}$ = 68.0 Hz) (each Se*C*H_2), 39.2 (*C*H_2NH_2), 48.1 (N*C*H_2), 107.5 (*C*-4 dmpz), 145.7, 146.3 (each s, C-3, 5 dmpz); ^{77}Se{^{1}H} NMR: δ 119.7 ppm. MS (IT) *m*/*z*: 319 $[M]^+$, 291, 248 [MH-2HCl], 203 [dmpz$CH_2CH_2Se]^+$.

dmpz$CH_2CH_2SeC_5H_4N$ (13)

To an ice cold ethanolic solution of di-2-pyridyl diselenide (py_2Se_2) (0.6 g, 1.91 mmol), sodium borohydride (0.16 g, 4.22 mmol) was added with stirring. The yellow solution faded to colorless, the reaction mixture was allowed to warm to room temperature. To this dmpzCH_2CH_2Br (0.775 g, 3.82 mmol) was added and stirred further for 2 h. The solvent was evaporated *in vacuo* and the residue was dissolved in water and extracted with diethyl ether (3 x 50 ml) which on evaporation *in vacuo* afforded a yellow oil which was purified by column chromatography with ethyl acetate-hexane mixture (30:70) to give a pale yellow oil (0.7 g. 66 %). Anal. Calcd. for $C_{12}H_{15}N_3Se$: Calcd.: C, 51.43; H, 5.40; N, 14.99 %. Found: C, 50.83; H, 5.38; N, 14.39 %. NMR spectral data ($CDCl_3$): ^{1}H NMR: δ 2.20, 2.24 (each 3H, s, -CH_3), 3.48 (2H, t, *J* = 7.5 Hz, -SeCH_2), 4.33 (2H, t, *J* = 7.5 Hz, -NCH_2), 5.74 (1H, s, H-4 dmpz), 7.00 (1H, t, *J* = 6.0 Hz, C_5H_4N), 7.29 (1H, d, *J* = 7.8 Hz, C_5H_4N), 7.41 (1H, td, *J* = 8.6 Hz, C_5H_4N), 8.43 (1H, d, *J* = 4.2 Hz, C_5H_4N); ^{13}C{^{1}H} NMR: δ 10.9, 13.4 (s, *Me*), 24.8 ($^1J_{C\text{-}Se}$ = 65.0 Hz, *C*H_2Se), 48.5 (N*C*H_2), 104.7 (*C*-4 dmpz), 120.4,

125.4 ($^1J_{C\text{-}Se}$ = 30.0 Hz), 135.8, 139.1, 147.6 (*C*-3, 5 dmpz), 150.0, 154.2; $^{77}Se\{^1H\}$ NMR: δ 321.7 ppm. MS (IT) *m*/*z*: 304 $[M + Na]^+$, 282 $[M + H]^+$, 254, 186 $[pySeCH_2CH_2]^+$.

dmpz$CH_2CH_2SeCH_2C_6H_4$(*p*-OTs) (14)

By using compound $ClCH_2C_6H_4$(*o*-OTs) the title compound dmpz$CH_2CH_2SeCH_2$Ph(*o*-OTs) (**14**) was prepared as a yellow liquid (0.658 g, 61 %). Anal. Calcd. for $C_{21}H_{24}N_2O_3SSe$: Calcd.: C, 54.42; H, 5.22; N, 6.04 %. Found: C, 53.93; H, 5.34; N, 5.66 %.NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.18 (6H, s, -C*H*$_3$), 2.41 (3H, s, OTs-C*H*$_3$), 2.84 (2H, t, *J* = 7.0 Hz, -C*H*$_2$Se), 3.50 (2H, s, $J_{Se\text{-}H}$ = 6.3 Hz, C*H*$_2$-Ph), 4.10 (2H, t, *J* = 7.2 Hz, -C*H*$_2$N), 5.74 (1H, s, Ar-*H*), 6.95 (1H, d, *J* = 8.1 Hz, Ar-*H*), 7.08-7.18 (2H, m, Ar-*H*), 7.30 (3H, d, *J* = 7.8 Hz, Ar-*H*), 7.73 (2H, d, *J* = 8.1 Hz, Ar-*H*); $^{13}C\{^1H\}$ NMR: δ 10.9, 13.3 (each s, *Me*), 20.5 ($^1J_{C\text{-}Se}$ = 62.0 Hz), 21.5, 23.5 ($^1J_{C\text{-}Se}$ = 69.0 Hz), 48.7, 104.8, 122.1, 127.1, 127.9, 128.1, 129.7, 131.1, 132.6, 132.9, 139.0, 145.5, 147.3, 147.4; $^{77}Se\{^1H\}$ NMR: δ 240.6 ppm. MS (IT) *m*/*z*: 465 $[M + H]^+$, 369 [M - dmpz]$^+$, 203 $[dmpzCH_2CH_2Se]^+$.

2. Synthesis of dmpzCH_2CH_2Br (a)

To a well stirred mixture of crushed NaOH (2.08 g, 52.0 mmol) and tetra-*n*-butyl ammonium bromide (TBAB) (0.167 g, 0.52 mmol) in a dichloromethane (50 ml) at 0-5 ^oC for 1 h, 3, 5-dimethylpyrazole (0.5 g, 5.2 mmol) and 1, 2-dibromoethane (1.57 ml, 3.41 g, 18.22 mmol) were added. The reaction mixture was stirred for 30 h at room temperature and the progress of the reaction was monitored by TLC. The reaction mixture was filtered and the filtrate was evaporated *in vacuo* to give a pale yellow oil which was purified by column chromatography using ethyl acetate-hexane mixture (30:70) as an eluent. The solvents were evaporated on a Rotavap to give a pale yellow liquid (0.7 g, 66 %). NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.19, 2.25 (each 3H, s, -CH_3), 3.66 (2H, t, J = 6.6 Hz, -CH_2Br), 4.29 (2H, t, J = 6.6 Hz, -NCH_2CH_2Br), 5.77 (1H, s, H-4 dmpz).

1. Synthesis of tosylated precursors

Tosylation of salicylaldehyde [2-(4-OTs)C_6H_4CHO] (b)

Salicylaldehyde (5.0 g, 40.94 mmol) was dissolved in pyridine (50 ml) and the solution was cooled at 0 ^oC. Tosyl chloride (4-Me$C_6H_4SO_2Cl$) (7.8 g, 40.94 mmol) was added and stirred overnight to get a white precipitate. To this reaction mixture, distilled water (50 ml) was added and extracted with diethyl ether. The organic extract was washed with dilute HCl followed by dilute NaOH. The solvent was evaporated *in vacuo* to yield a white crystalline solid (10.7 g, 94 %). NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.45 (3H, s, -CH_3), 7.20 (1H, d, J = 8.1 Hz, Ar-*H*), 7.33 (2H, d, J = 8.1 Hz, Ar-*H*), 7.39 (1H, t, J = 7.6 Hz, Ar-*H*), 7.58 (1H, td, ^1J = 7.1 Hz, ^2J = 1.5 Hz, Ar-*H*), 7.63 (2H, d, J = 8.4 Hz, Ar-*H*), 7.86 (1H, dd, ^1J = 7.8 Hz, ^2J = 1.8 Hz, Ar-*H*), 9.99 (1H, s, -C*H*O).

Synthesis of [2-(4-OTs)$C_6H_4CH_2OH$] (c)

To a stirred methanol solution (200 ml) of **b** (43.0 g, 155.62 mmol), sodium borohydride (7.1 g, 187.7 mmol) was added slowly with constant stirring at 0 ^oC. The progress of reaction was monitored by TLC and after completion of reaction (~ 4 h) methanol was evaporated *in vacuo*. The residue was then dissolved in water and extracted with ethyl acetate (3 x 150 ml). The crude product obtained on evaporation of ethyl acetate *in vacuo* was purified on column chromatography with ethyl acetate and hexane mixture (20:80) to yield **c** as a colorless oil (37.6 g, 87 %). NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.47 (3H, s, -CH_3), 4.58 (2H, s, ArCH_2), 6.86

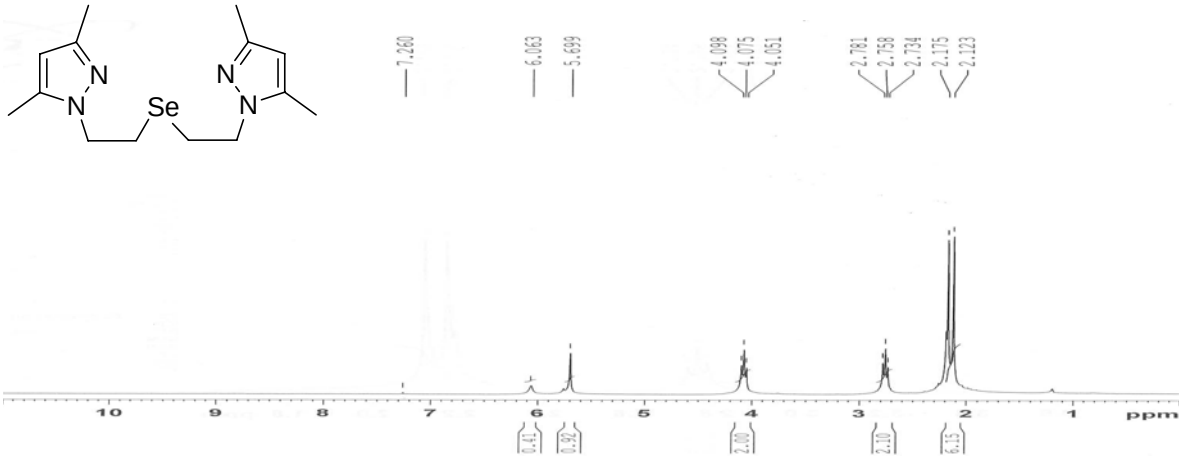
(1H, d, *J* = 8.1 Hz, Ar-*H*), 7.20 (1H, t, *J* = 7.0 Hz, Ar-*H*), 7.28 (1H, t, *J* = 7.2 Hz, Ar-*H*), 7.35 (2H, d, *J* = 8.1 Hz, Ar-*H*), 7.51 (1H, d, *J* = 7.5 Hz, Ar-*H*), 7.7 (1H, d, *J* = 8.4 Hz, Ar-*H*).

Synthesis of [2-(4-OTs)$C_6H_4CH_2Cl$] (d)

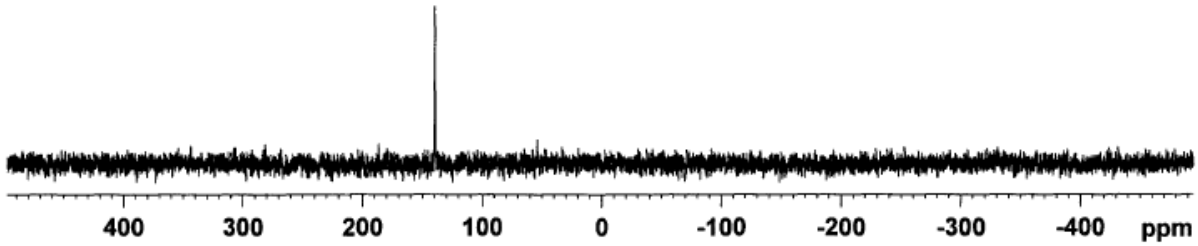
A 500 ml two necked round bottom flask was charged with 70 ml $SOCl_2$ and immersed in a tub containing ice-salt mixture. To this, a pyridine (40 ml) solution of **c** (43.0 g, 154.5 mmol) was added drop wise through a dropping funnel with vigorous stirring. *(Caution: The reaction is highly exothermic, gives SO_2 and HCl fumes. Also pyridine is known to hazardous for health. Hence the reaction must be carried out in a well ventilated fume hood).* The temperature was maintained below 0 ^oC till complete addition of **c** (~ 2 h). The reaction mixture was brought to room temperature. It was then stirred further for 5 h and poured in a chilled (-5 to 10 ^oC) 40 % HCl very slowly with constant stirring. The reaction mixture was then extracted with ethyl acetate (3 x 100 ml) and on evaporation of organic solvent *in vacuo* gave a pinkish-white pure crystalline solid **d** was obtained (42.5 g, 93 %). M.p. 52 ^oC. NMR spectral data ($CDCl_3$): ^1H NMR: δ 2.45 (3H, s, -CH_3), 4.47 (2H, s, ArCH_2), 7.06 (1H, m, Ar-*H*), 7.26 (2H, m, Ar-*H*), 7.43 (2H, d, *J* = 8.1 Hz, Ar-*H*), 7.46 (1H, m, Ar-*H*), 7.76 (2H, d, *J* = 8.4 Hz, Ar-*H*); $^{13}C\{^1H\}$ NMR: δ 21.8, 40.3, 122.6, 127.5, 128.5, 129.8, 130.1, 131.0, 131.1, 132.5, 145.9, 147.3.

3. ^{1}H NMR, ^{77}Se{^{1}H} NMR and LC-MS of the 2-(3, 5-dimethylpyrazol-1-yl) ethylseleno derivatives

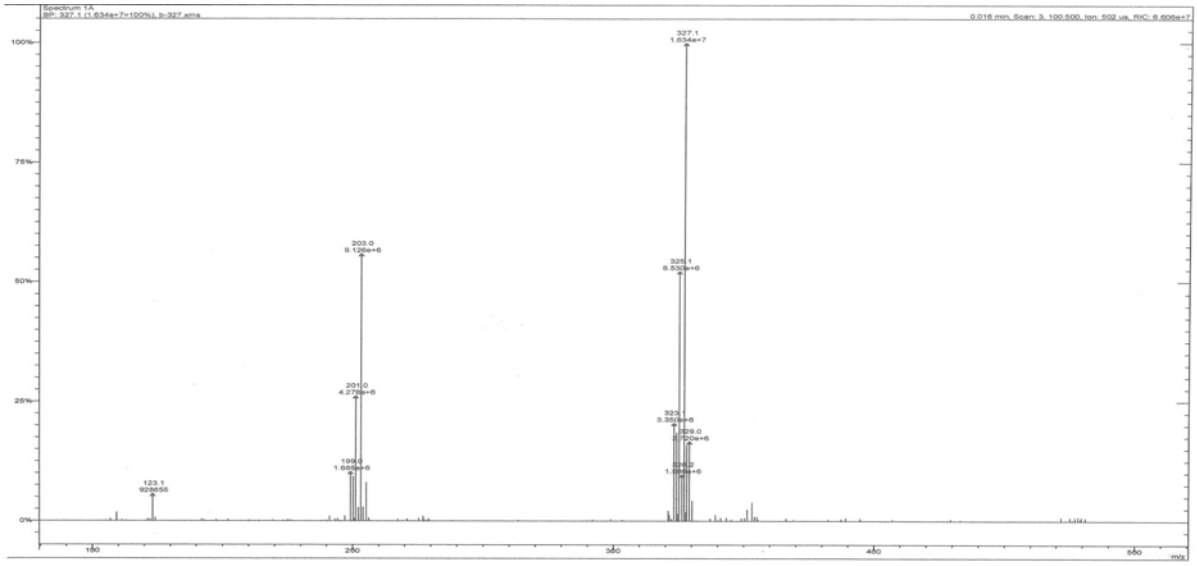
$(dmpzCH_2CH_2)_2Se$ (1):



^1H NMR ($CDCl_3$) of $(dmpzCH_2CH_2)_2Se$

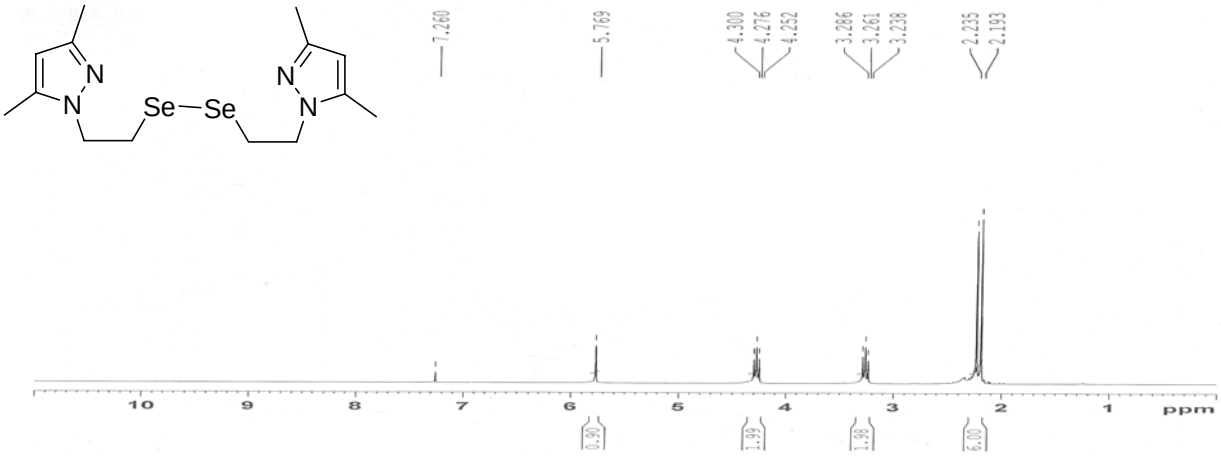


$^{77}Se\{^1H\}$ NMR in $CDCl_3$ for $(dmpzCH_2CH_2)_2Se$: δ 139.6 ppm

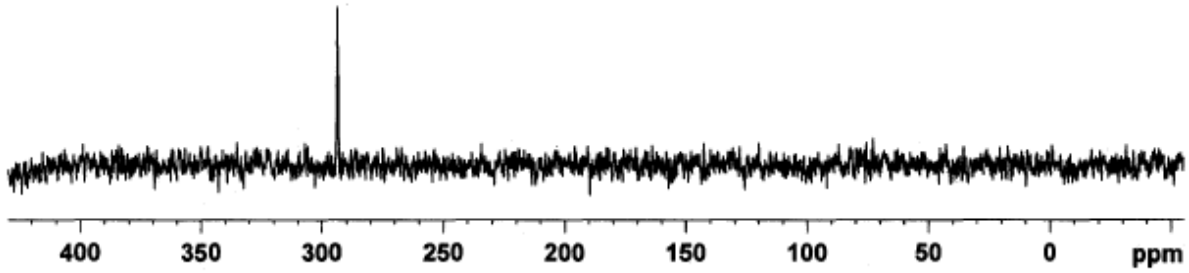


LC-MS of $(dmpzCH_2CH_2)_2Se$

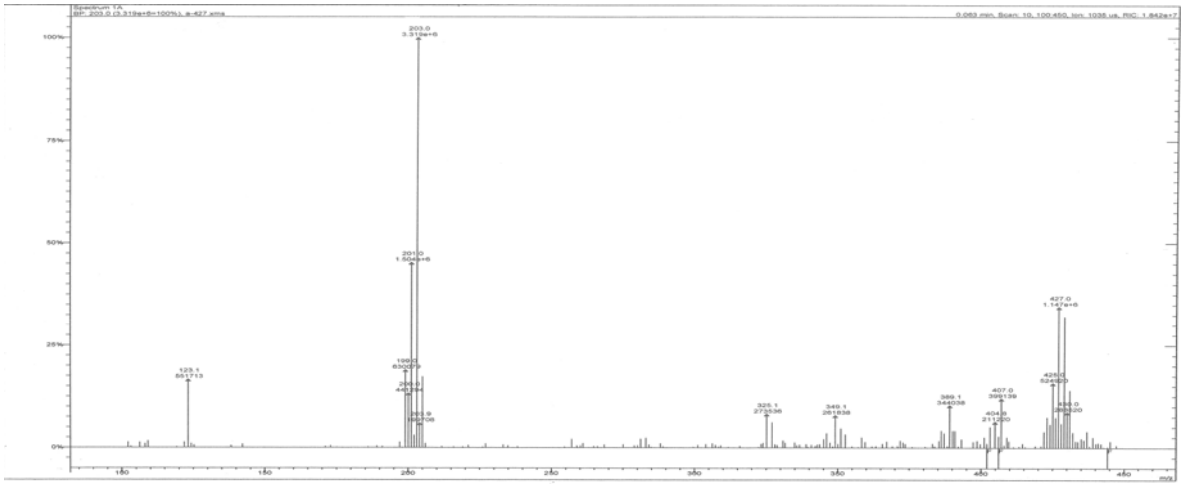
$(dmpzCH_2CH_2Se)_2$ (2):



^1H NMR ($CDCl_3$) of $(dmpzCH_2CH_2Se)_2$



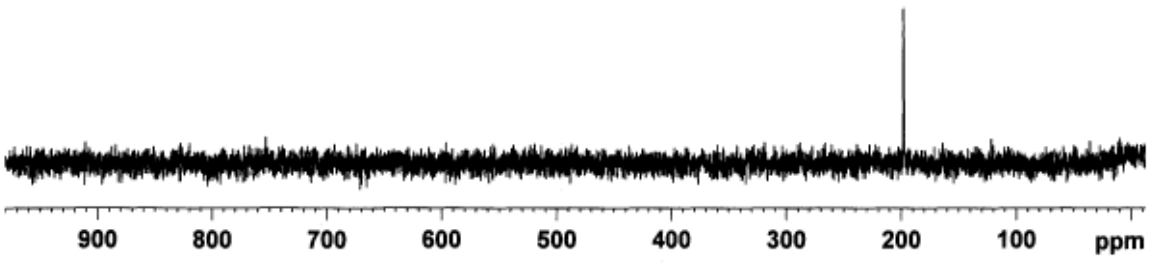
$^{77}Se\{^1H\}$ NMR in $CDCl_3$ for $(dmpzCH_2CH_2Se)_2$: δ 293.5 ppm



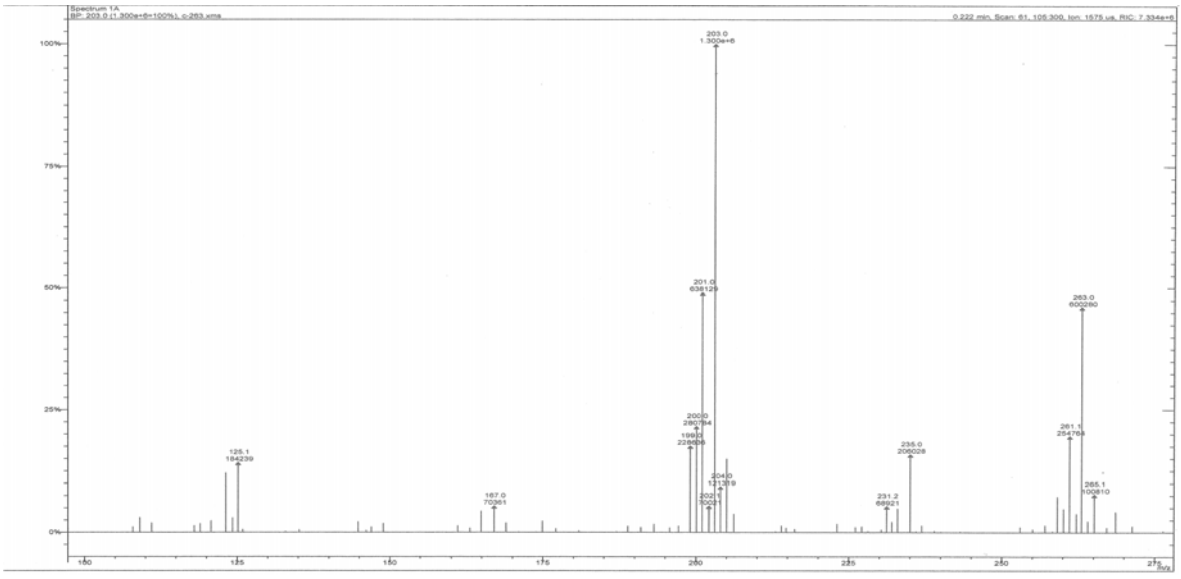
LC-MS of $(dmpzCH_2CH_2Se)_2$

dmpzCH$_2$CH$_2$SeCH$_2$COOH (3):

^{1}H NMR ($CDCl_3$) of dmpzCH$_2$CH$_2$SeCH$_2$COOH

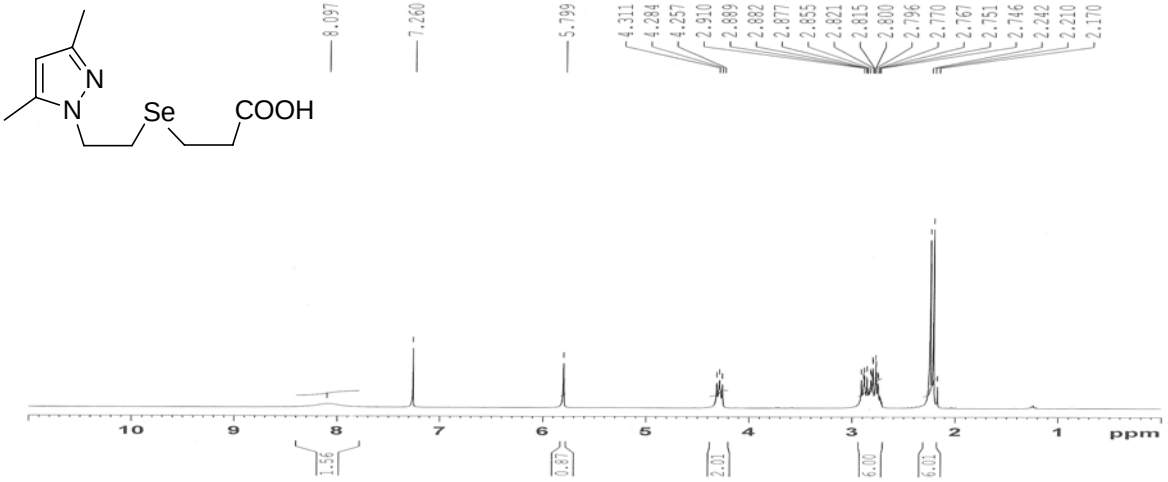


^{77}Se{^{1}H} NMR in $CDCl_3$ for dmpzCH$_2$CH$_2$SeCH$_2$COOH: δ 198.0 ppm

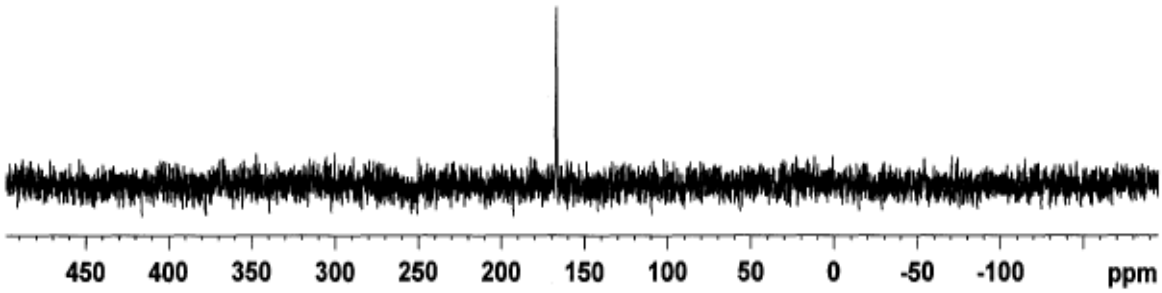


LC-MS of dmpzCH$_2$CH$_2$SeCH$_2$COOH

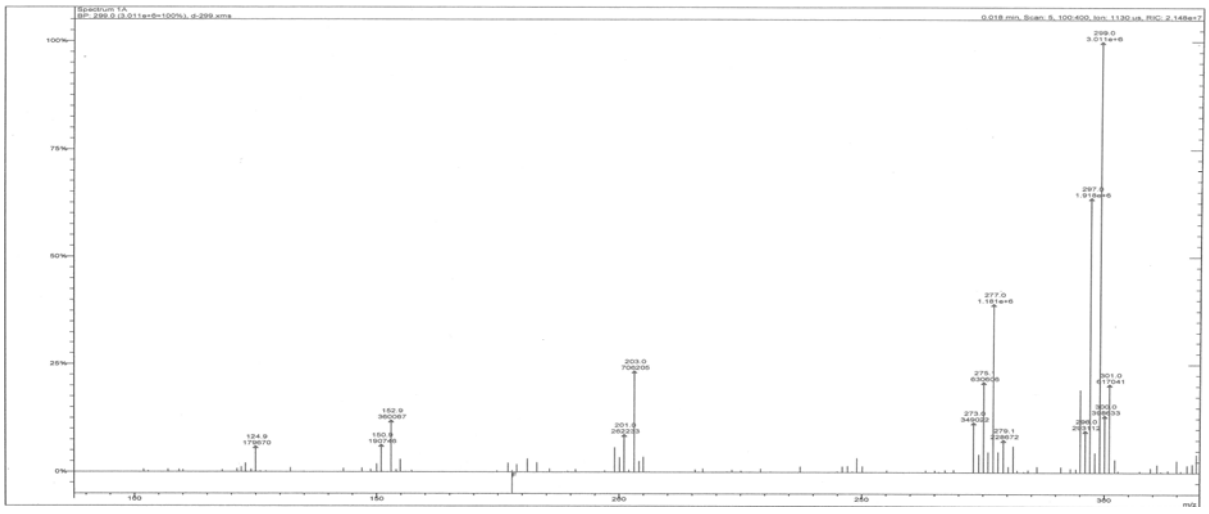
dmpz$CH_2CH_2SeCH_2CH_2COOH$ (4):



^{1}H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2CH_2COOH$

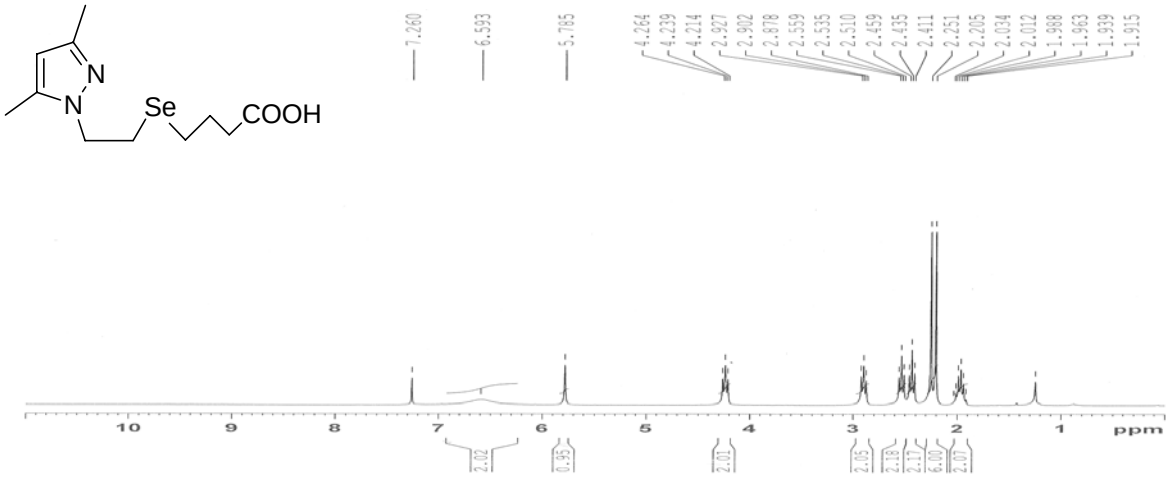


$^{77}Se\{^{1}H\}$ NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2CH_2COOH$: δ 166.9 ppm

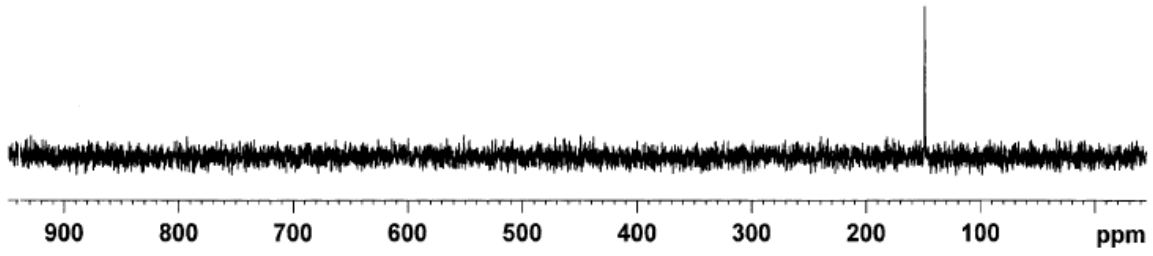


LC-MS of dmpz$CH_2CH_2SeCH_2CH_2COOH$

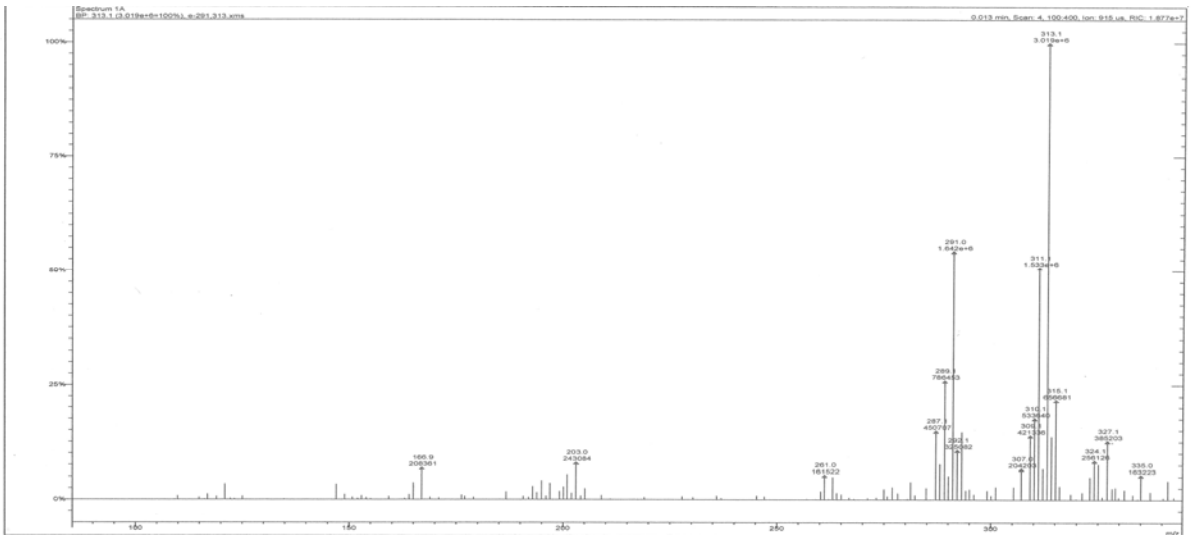
dmpz$CH_2CH_2SeCH_2CH_2CH_2COOH$ (5):



^{1}H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2CH_2CH_2COOH$

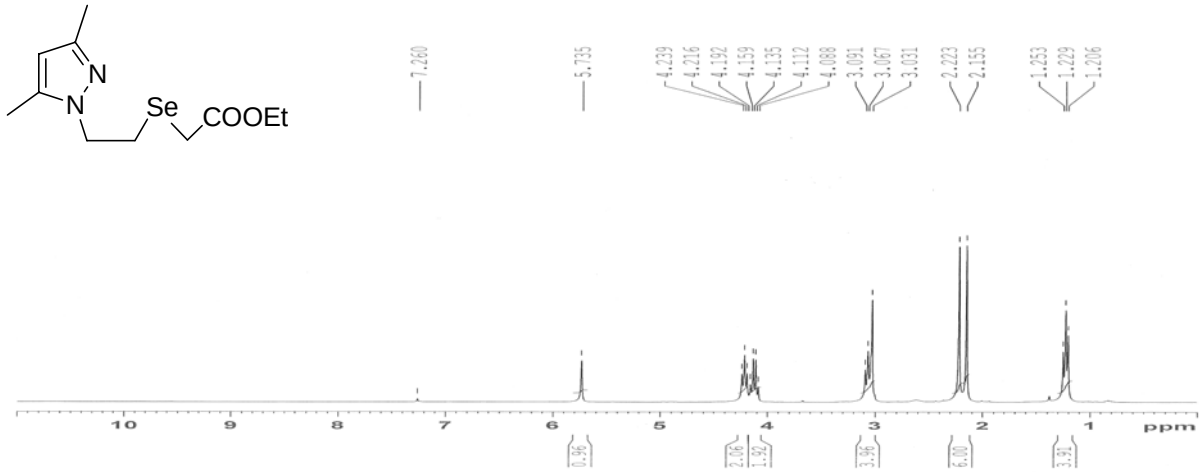


$^{77}Se\{^{1}H\}$ NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2CH_2CH_2COOH$: δ 148.8 ppm

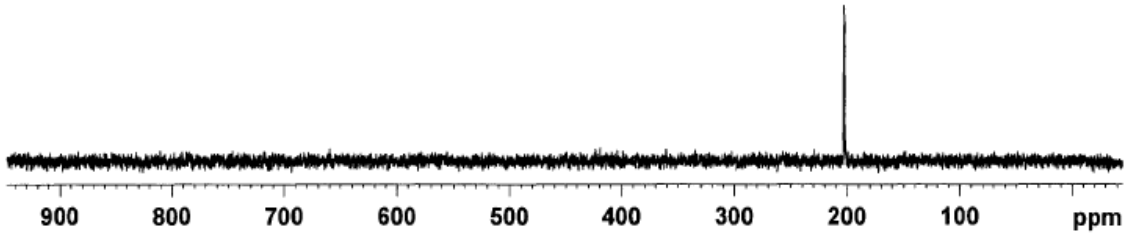


LC-MS of dmpz$CH_2CH_2SeCH_2CH_2CH_2COOH$

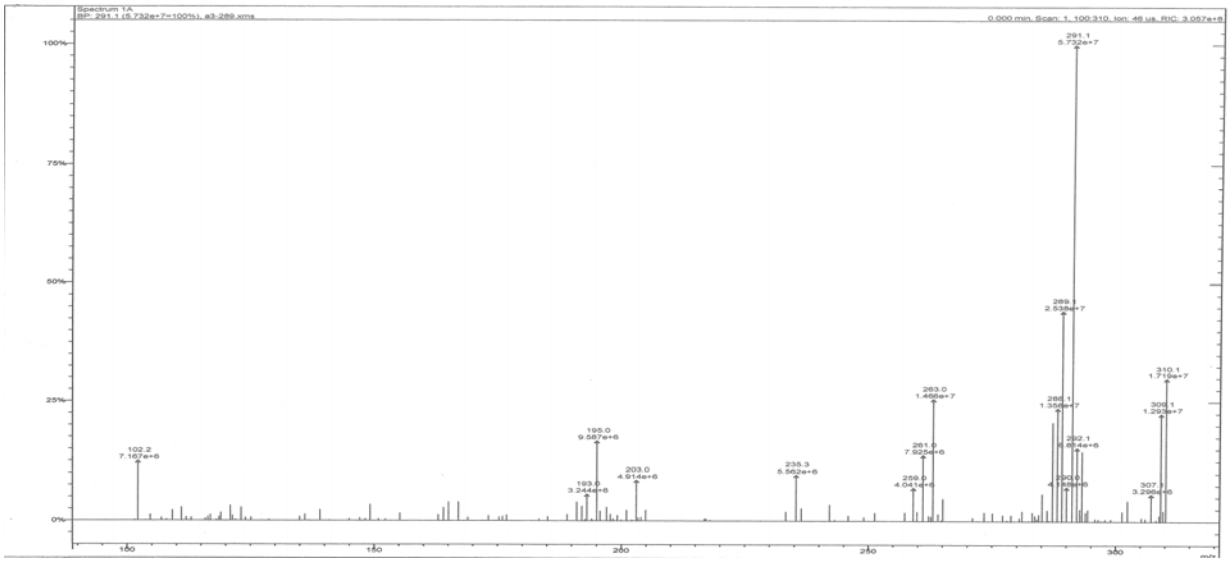
dmpz$CH_2CH_2SeCH_2COOCH_2CH_3$ (6):



^{1}H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2COOC_2H_5$

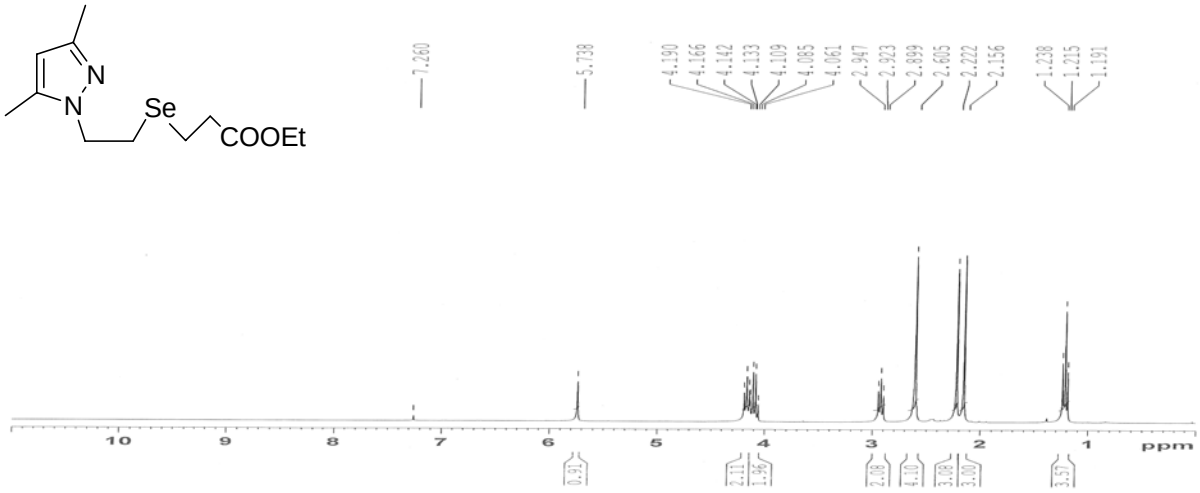


^{77}Se{^{1}H} NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2COOC_2H_5$: δ 202.4 ppm

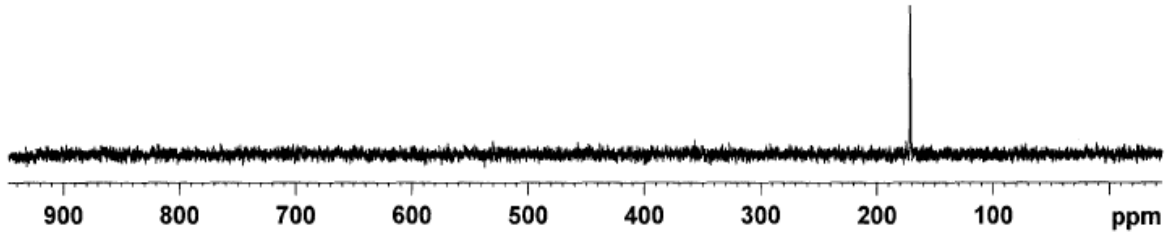


LC-MS of dmpz$CH_2CH_2SeCH_2COOC_2H_5$

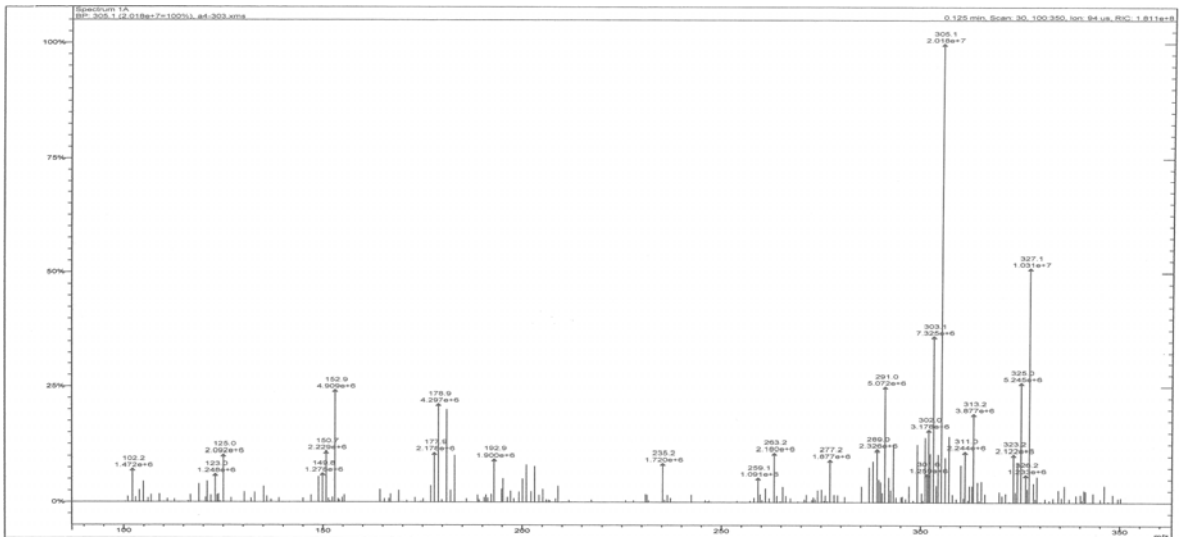
dmpz$CH_2CH_2SeCH_2CH_2COOCH_2CH_3$ (7):



^{1}H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2CH_2COOC_2H_5$

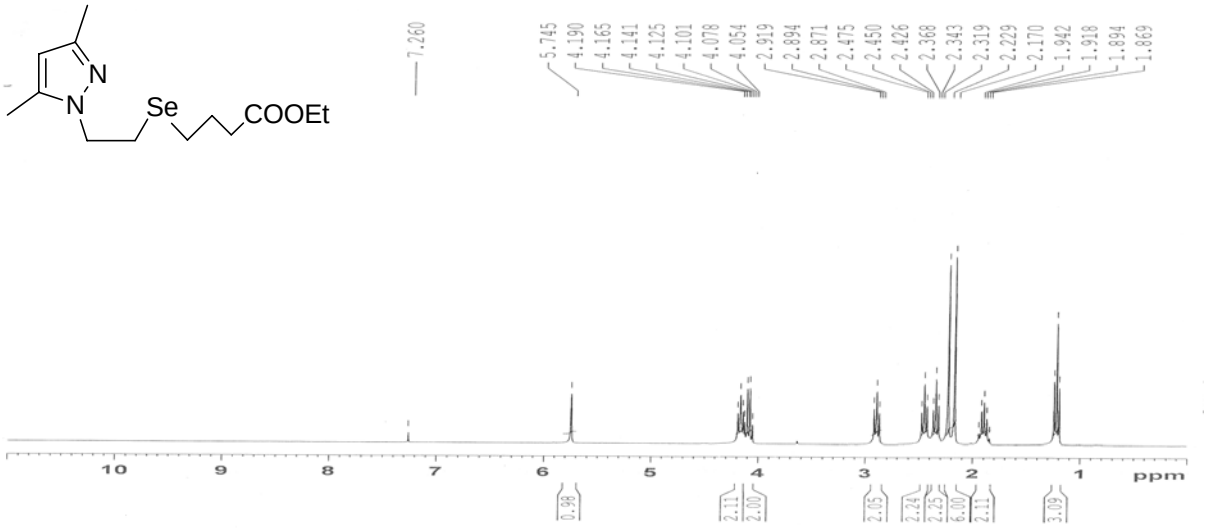


$^{77}Se\{^{1}H\}$ NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2CH_2COOC_2H_5$: δ 171.1 ppm

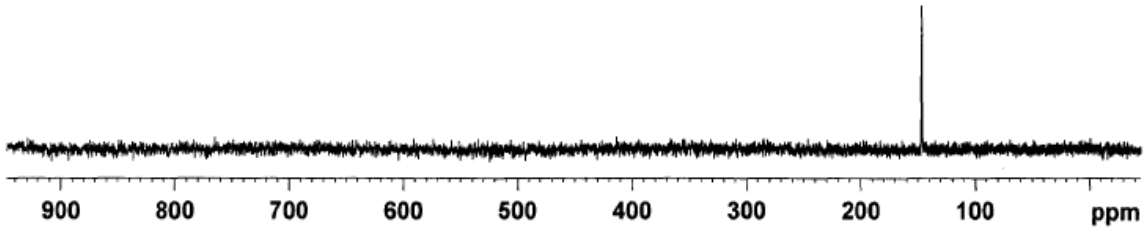


LC-MS of dmpz$CH_2CH_2SeCH_2CH_2COOC_2H_5$

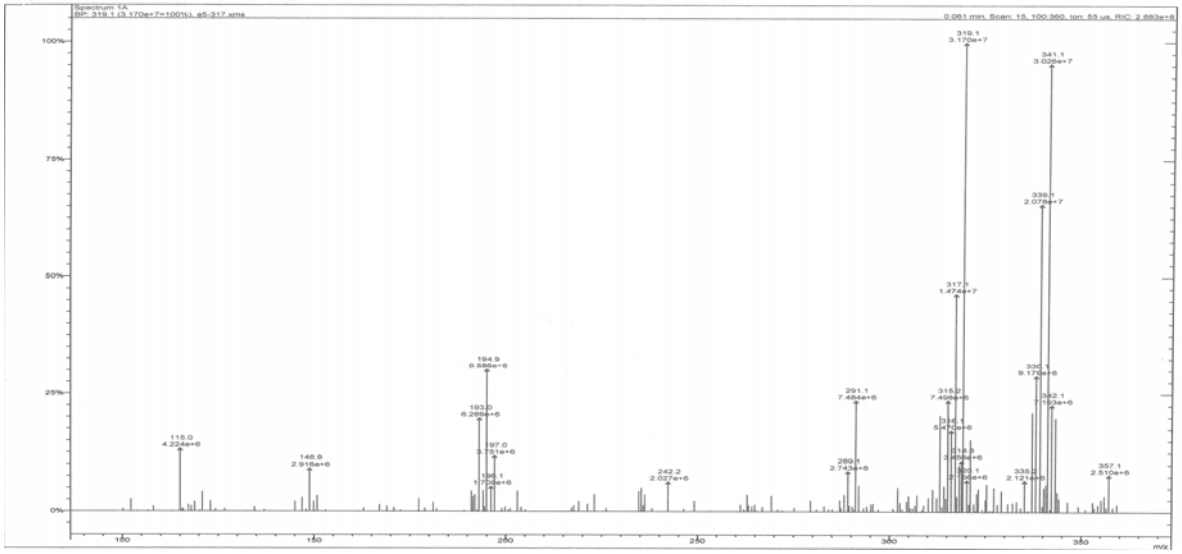
dmpz$CH_2CH_2SeCH_2CH_2CH_2COOCH_2CH_3$ (8):



^{1}H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2CH_2CH_2COOC_2H_5$

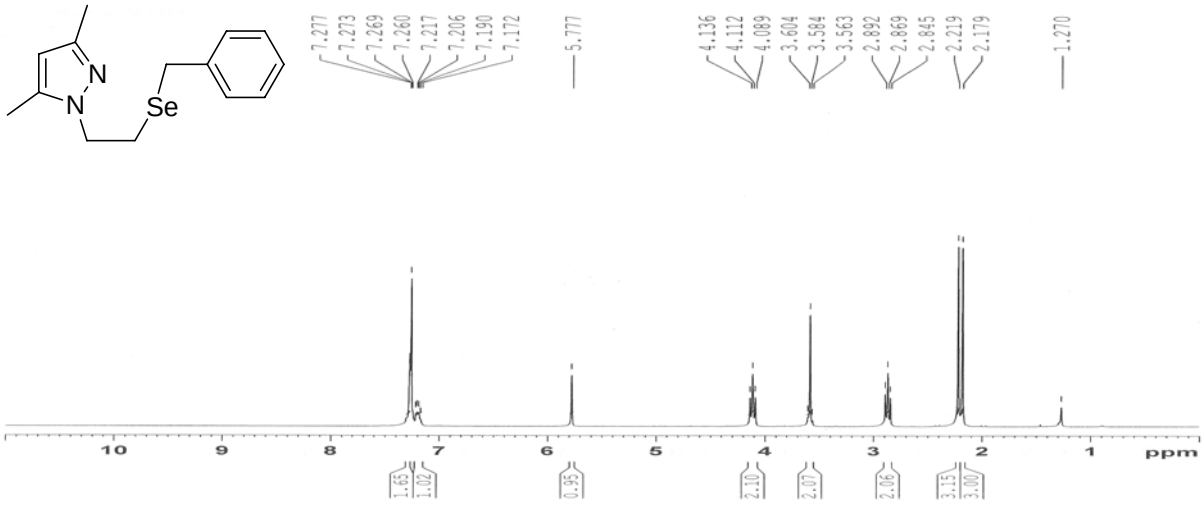


$^{77}Se\{^{1}H\}$ NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2CH_2CH_2COOC_2H_5$: δ 147.0 ppm

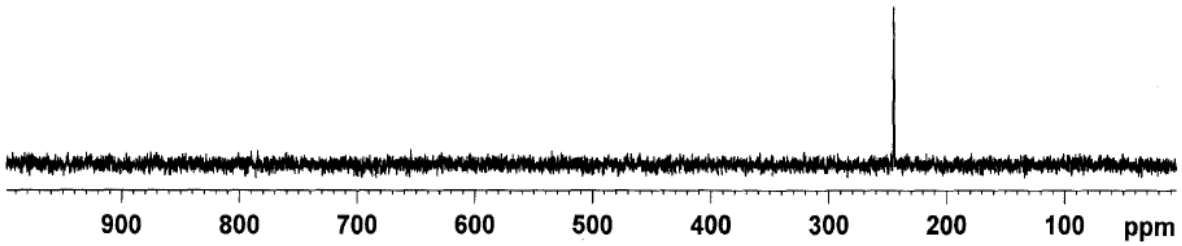


LC-MS of dmpz$CH_2CH_2SeCH_2CH_2CH_2COOC_2H_5$

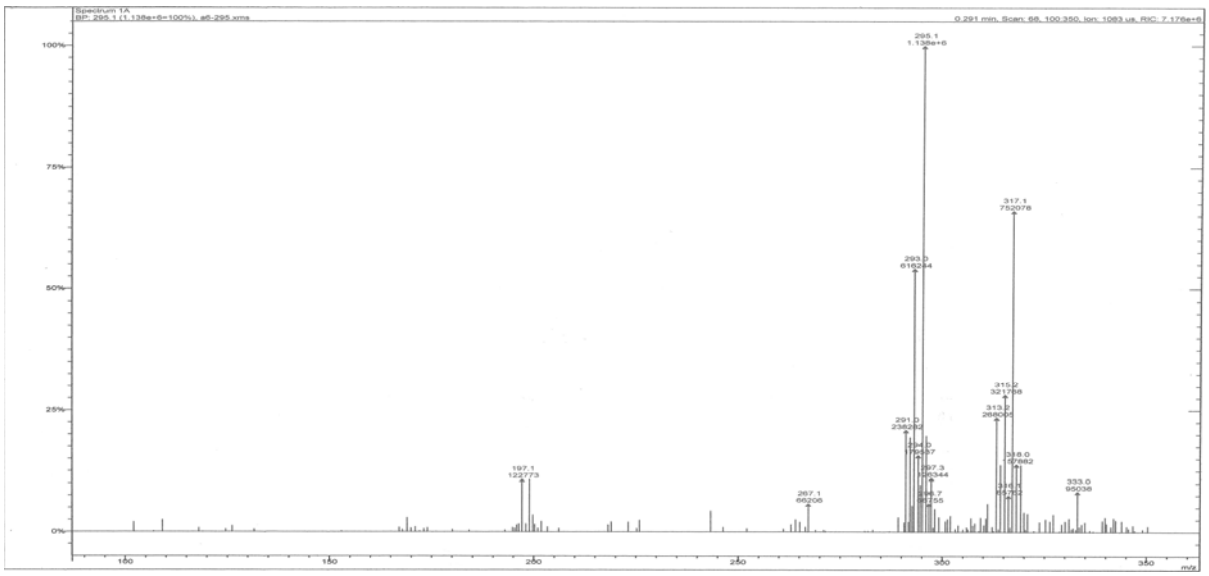
dmpz$CH_2CH_2SeCH_2Ph$ (9):



^1H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2Ph$

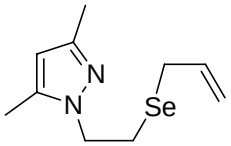


$^{77}Se\{^1H\}$ NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2Ph$: δ 244.6 ppm

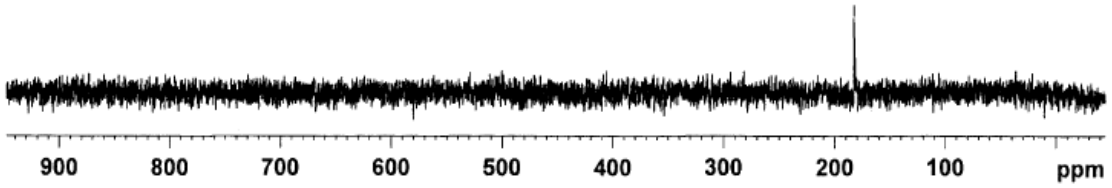


LC-MS of dmpz$CH_2CH_2SeCH_2Ph$

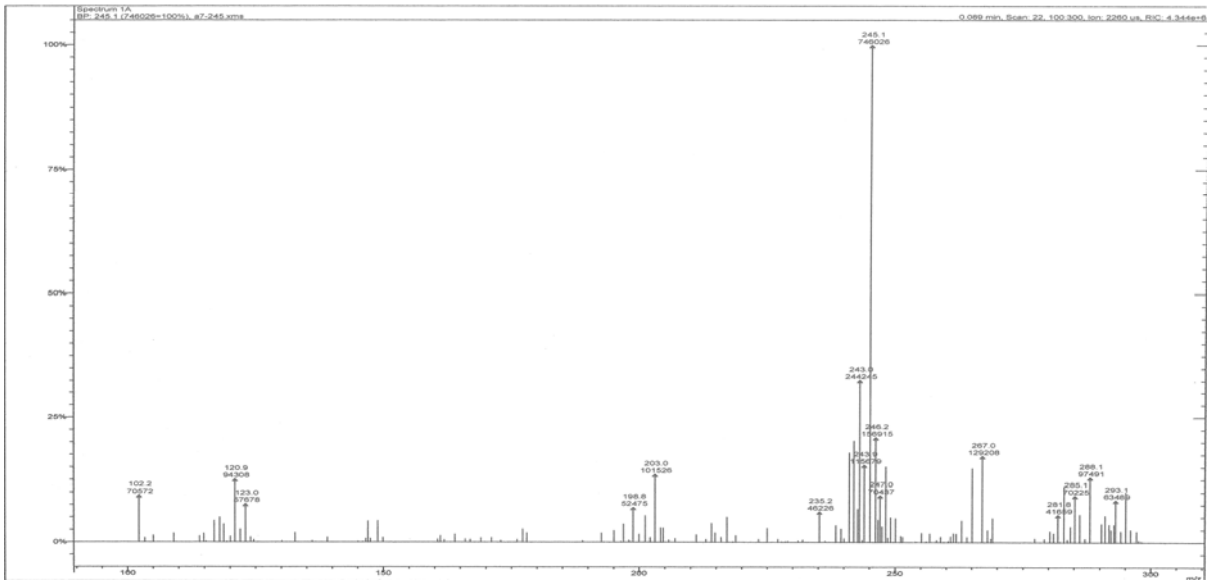
dmpz$CH_2CH_2SeCH_2CH=CH_2$ (**10**):



^{1}H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2CH=CH_2$

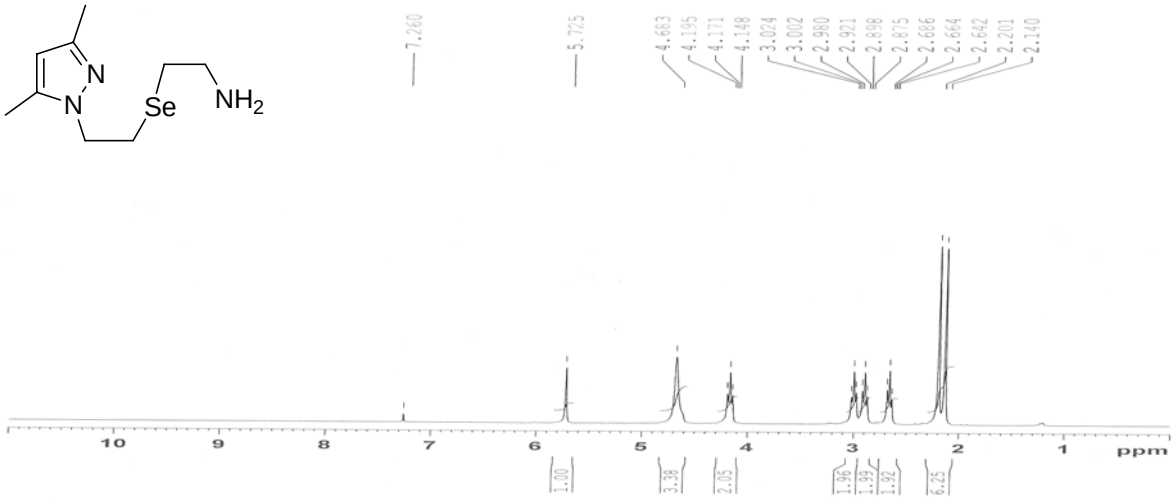


^{77}Se{^{1}H} NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2CH=CH_2$: δ 182.0 ppm

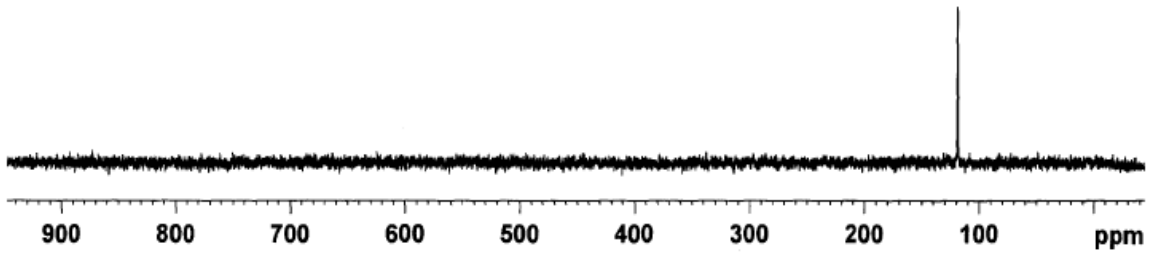


LC-MS of dmpz$CH_2CH_2SeCH_2CH=CH_2$

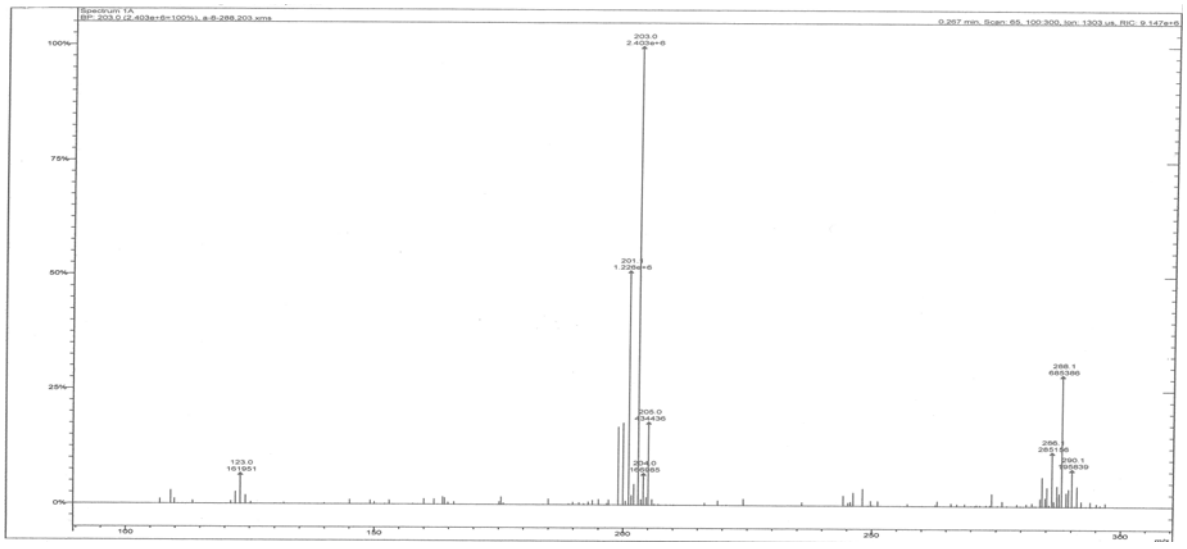
dmpz$CH_2CH_2SeCH_2CH_2NH_2$ (11):



^{1}H NMR ($CDCl_3$) of dmpz$CH_2CH_2SeCH_2CH_2NH_2$

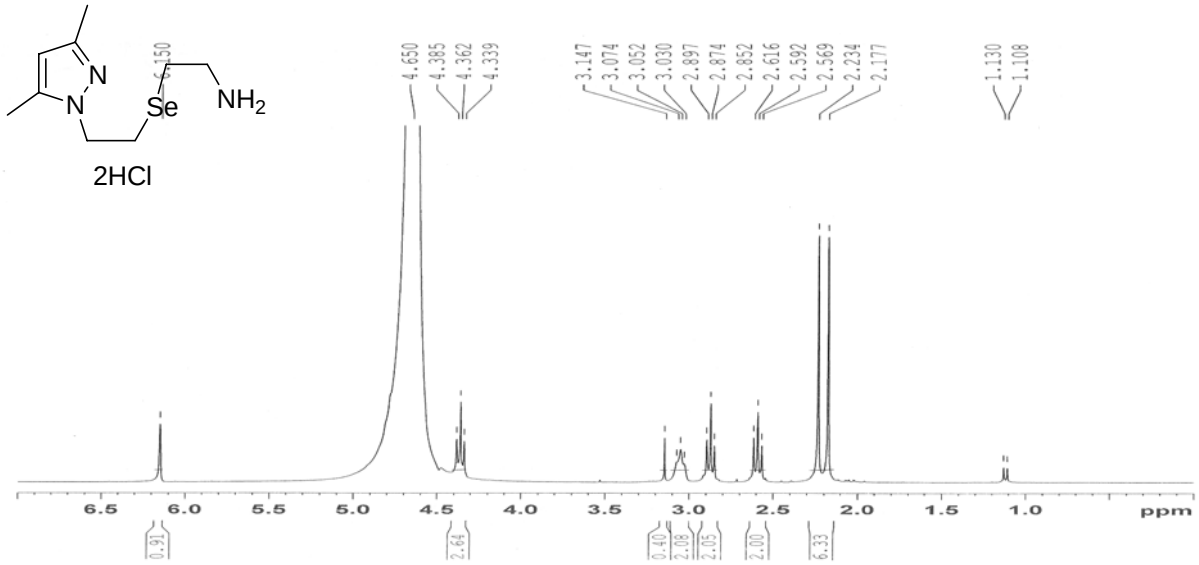


^{77}Se{^{1}H} NMR in $CDCl_3$ for dmpz$CH_2CH_2SeCH_2CH_2NH_2$: δ 118.9 ppm

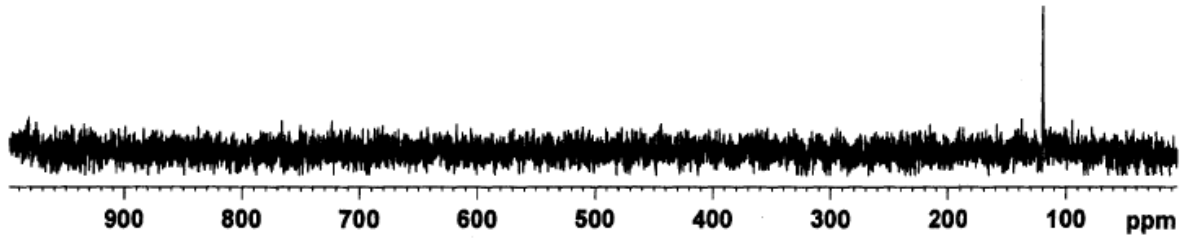


LC-MS of dmpz$CH_2CH_2SeCH_2CH_2NH_2$

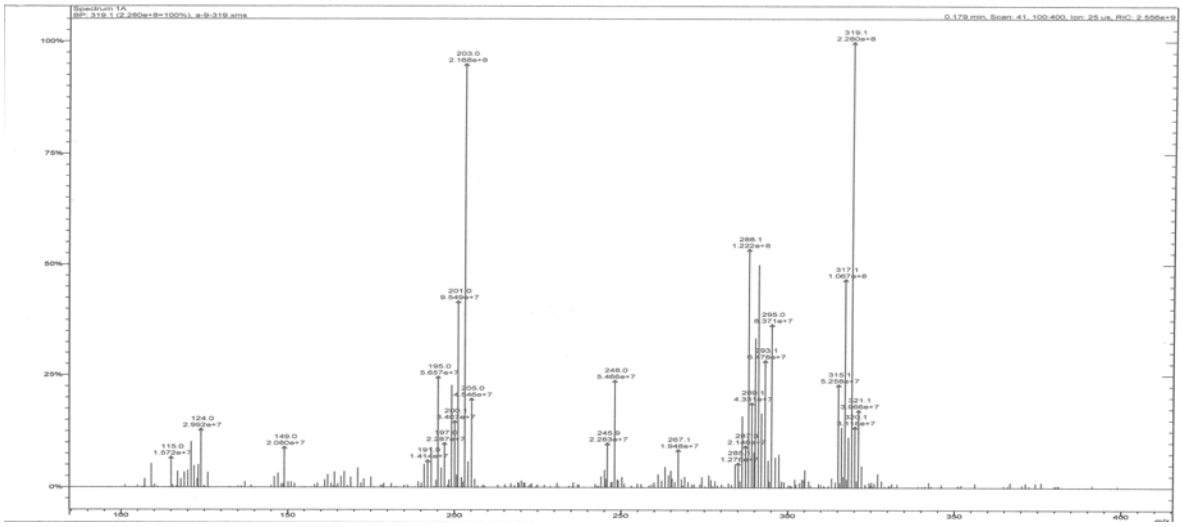
dmpz$CH_2CH_2SeCH_2CH_2NH_2$. 2HCl (12):



^{1}H NMR (D_2O) of dmpz$CH_2CH_2SeCH_2CH_2NH_2$. 2HCl

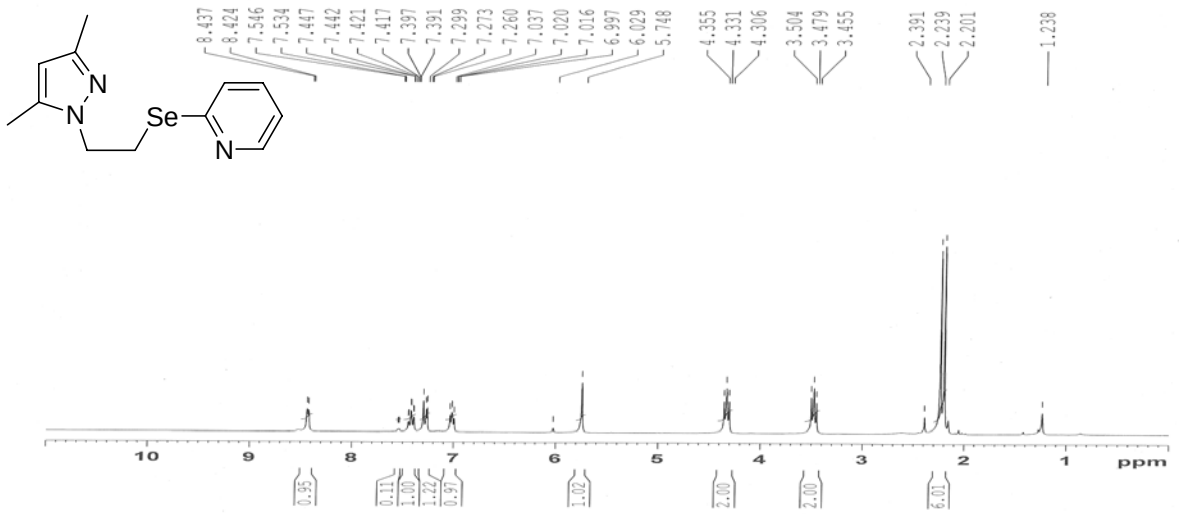


$^{77}Se\{^{1}H\}$ NMR in D_2O for dmpz$CH_2CH_2SeCH_2CH_2NH_2$ .2HCl: δ 119.7 ppm

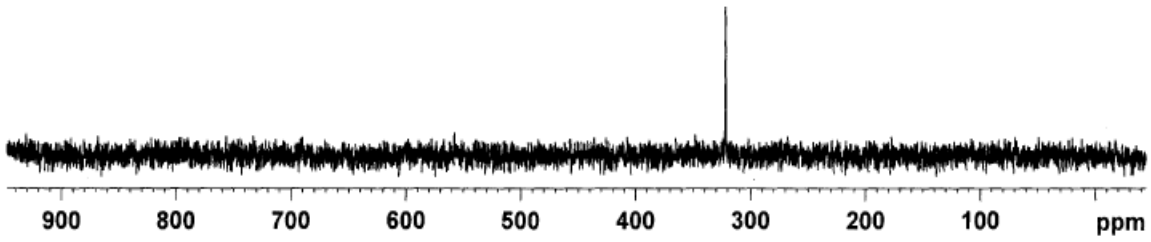


LC-MS of dmpz$CH_2CH_2SeCH_2CH_2NH_2$. 2HCl

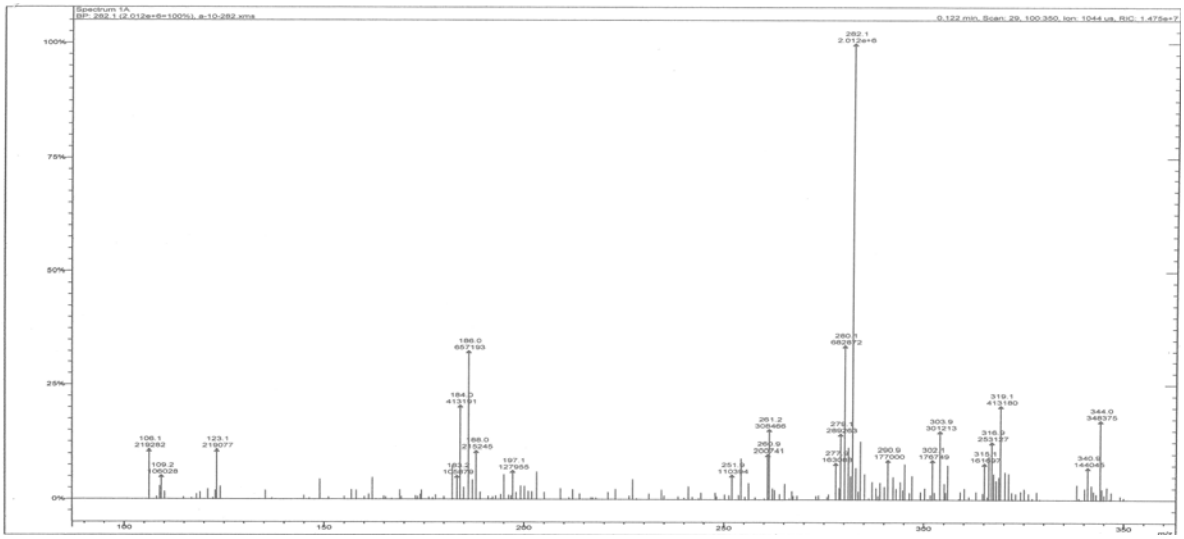
dmpzCH$_2$CH$_2$SeC$_5$H$_4$N (13):



^{1}H NMR (CDCl$_3$) of dmpzCH$_2$CH$_2$SeC$_5$H$_4$N

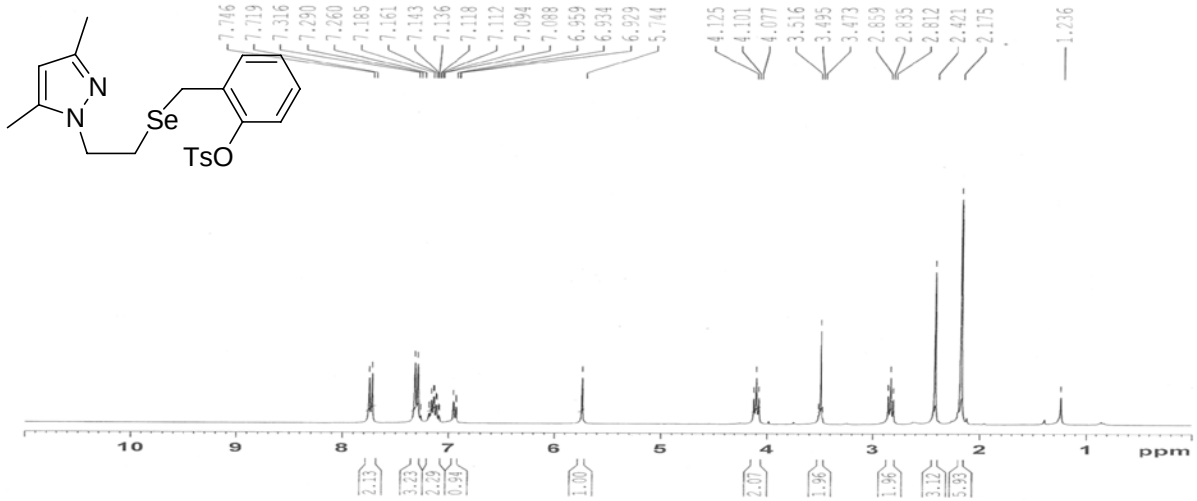


^{77}Se{^{1}H} NMR spectrum in CDCl$_3$ for dmpzCH$_2$CH$_2$SeC$_5$H$_4$N in CDCl$_3$: δ 321.6 ppm

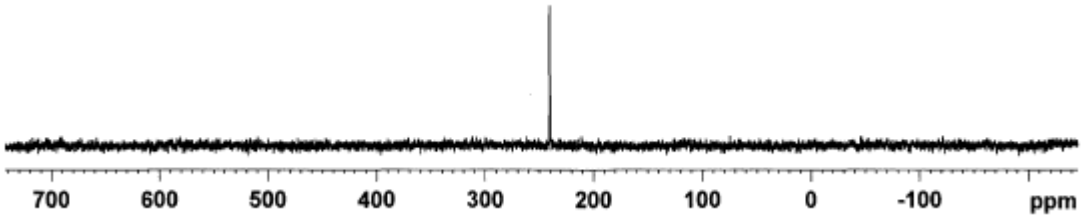


LC-MS of dmpzCH$_2$CH$_2$SeC$_5$H$_4$N

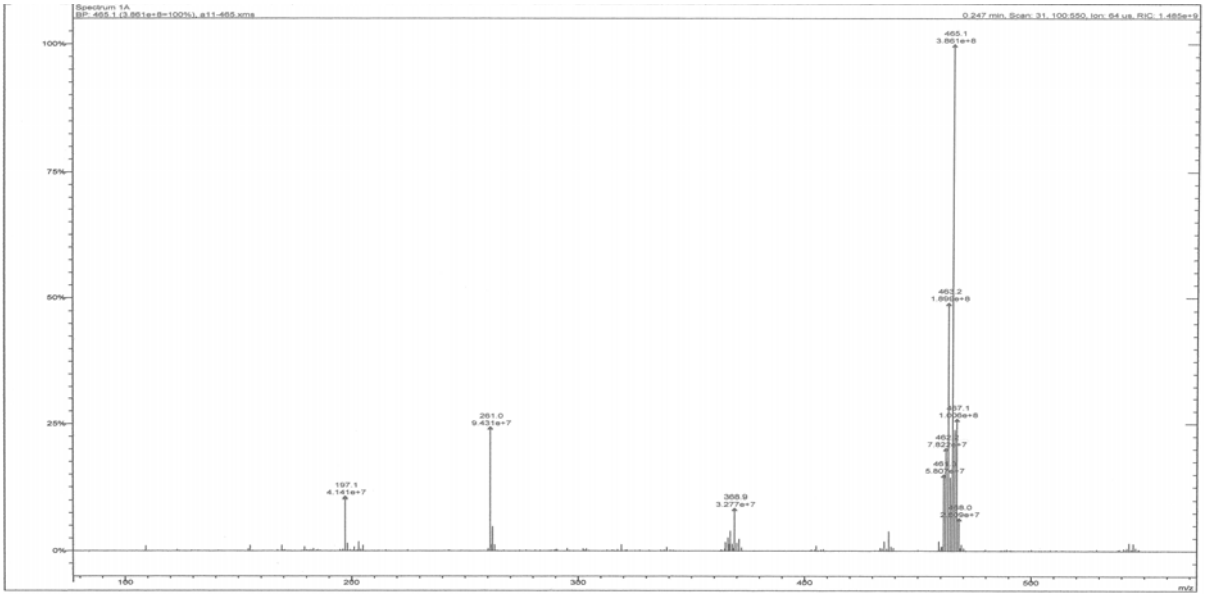
dmpzCH$_2$CH$_2$SeCH$_2$C$_6$H$_4$(*p*-OTs) (14):



^{1}H NMR ($CDCl_3$) of dmpzCH$_2$CH$_2$Se CH$_2$C$_6$H$_4$(*p*-OTs)

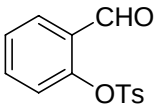


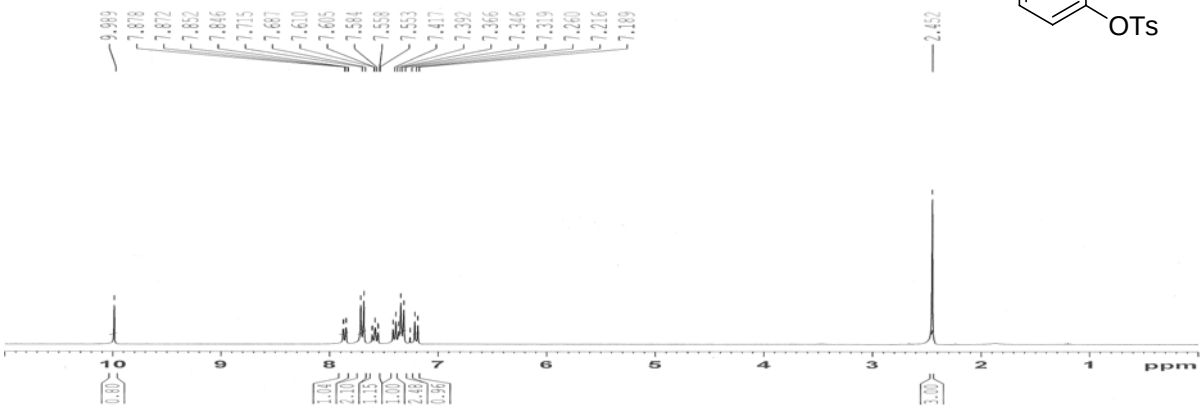
^{77}Se{^{1}H} NMR in $CDCl_3$ for dmpzCH$_2$CH$_2$SeCH$_2$C$_6$H$_4$(*p*-OTs): δ 240.6 ppm



LC-MS of dmpzCH$_2$CH$_2$SeCH$_2$C$_6$H$_4$(*p*-OTs)

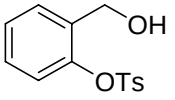
C_6H_4(*p*-OTs)CHO:





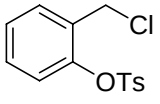
^{1}H NMR ($CDCl_3$) of C_6H_4(*p*-OTs)CHO

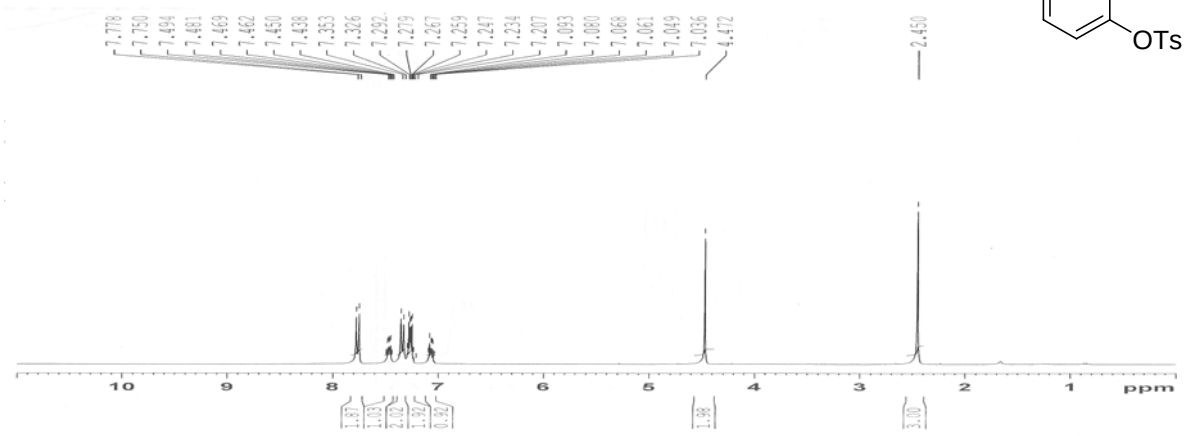
C_6H_4(*p*-OTs)CH_2OH:



^{1}H NMR ($CDCl_3$) of C_6H_4(*p*-OTs)CH_2OH

C_6H_4(*p*-OTs)CH_2Cl





^{1}H NMR ($CDCl_3$) of C_6H_4(*p*-OTs)CH_2Cl

4. Kinetic analysis data

Catalytic properties of 2-(3,5-dimethylpyrazol-1-yl)ethylseleno derivatives as the probable mimics of Glutathione Peroxidase (GPx)

Reaction

Initially H_2O_2 (aqueous solution) was standardized by titration with standardized $KMnO_4$ and the concentration of H_2O_2 was found 47.6 % (w/w).

A typical reaction mixture is obtained as given in equation below:

DTT^{red} (23.1 mg, 0.15 mmol) + 2-(3,5-dimethylpyrazol-1-yl)ethylseleno derivative as catalyst (0.015 mmol) + H_2O_2 (9.15 µl from 47.6 % aqueous solution, 0.15 mmol)

Amounts of 2-(3,5-dimethylpyrazol-1-yl)ethylseleno derivatives used as catalyst for reaction are as given below

Compound	**No.**	**Amount of catalyst (mg) (mmol)**
$(dmpzCH_2CH_2)_2Se$	(**1**)	4.9 mg (0.015 mmol)
$(dmpzCH_2CH_2Se)_2$	(**2**)	6.0 mg (0.015 mmol)
$dmpzCH_2SeCH_2COOH$	(**3**)	3.9 mg (0.015 mmol)
$dmpzCH_2CH_2SeCH_2CH_2COOH$	(**4**)	4.1 mg (0.015 mmol)
$dmpzCH_2CH_2SeCH_2CH_2CH_2COOH$	(**5**)	4.3 mg (0.015 mmol)
$dmpzCH_2CH_2SeCH_2CH_2NH_2$	(**11**)	3.7 mg (0.015 mmol)
		1.8 mg (0.0073 mmol)
		0.9 mg (0.00365 mmol)
		0.4 mg (0.0016 mmol)
$dmpzCH_2CH_2SeCH_2CH_2NH_2.2HCl$	(**12**)	4.2 mg (0.015 mmol)
$dmpzCH_2CH_2SePy$	(**13**)	4.2 mg (0.015 mmol)

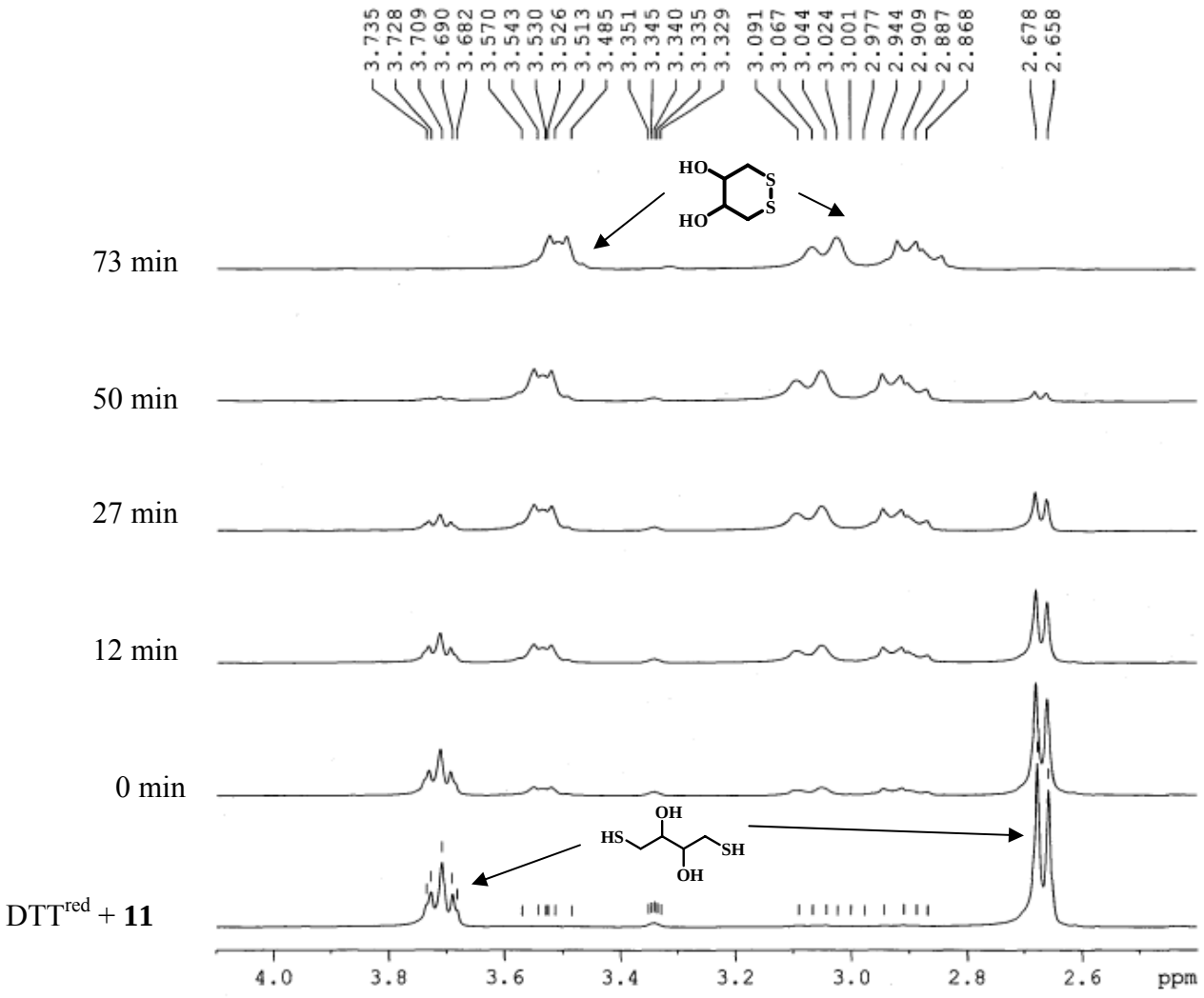
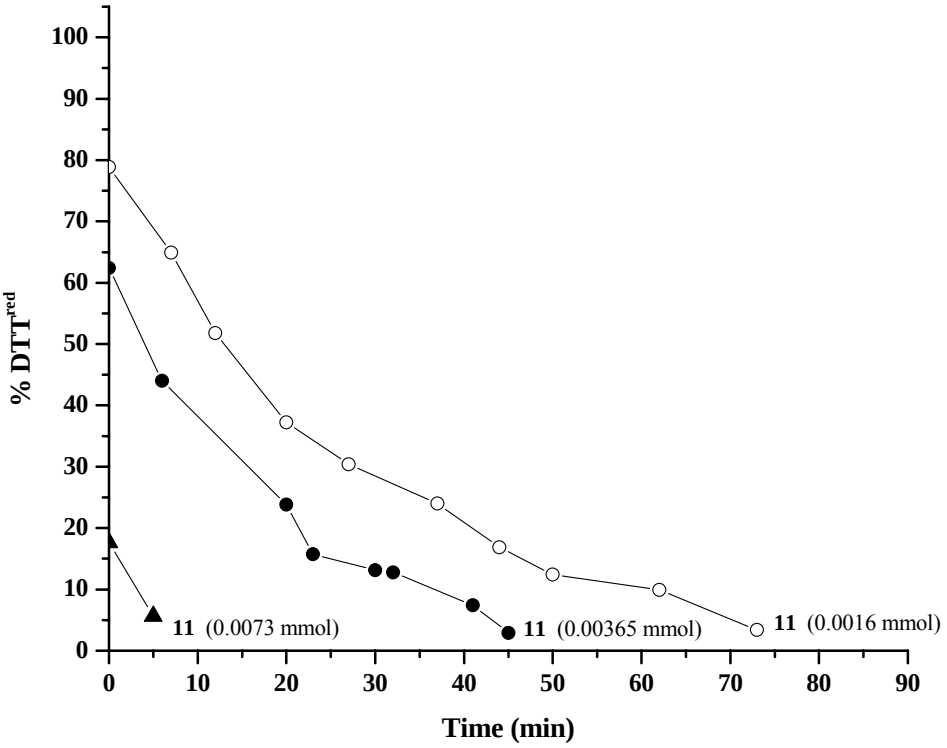


Figure 1. A series of ^{1}H NMR spectra obtained during the oxidation of DTT^{red} (0.15 mmol) with H_2O_2 (0.15 mmol) in the presence of a catalytic amount of **11** (0.0016 mmol) in CD_3OD (0.5 ml) at 25ºC.

Effect of concentration of dmpz$CH_2CH_2SeCH_2CH_2NH_2$ (11)

A typical reaction mixture is obtained as given in equation below:

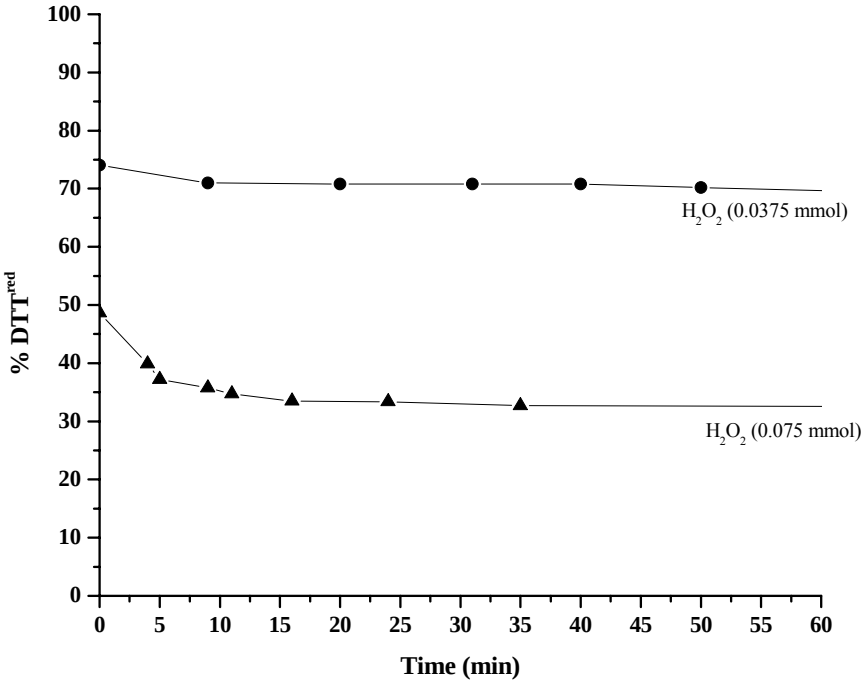
DTT^{red} (23.1 mg, 0.15 mmol) + **11** (x mmol) + H_2O_2 (9.15 µl from 47.6 % aqueous solution, 0.15 mmol)



Effect of concentration of H_2O_2

A typical reaction mixture is obtained as given in equation below:

DTT^{red} (23.1 mg, 0.15 mmol) + $dmpzCH_2CH_2SeCH_2CH_2NH_2$ (3.7 mg, 0.015 mmol) + H_2O_2 (x mmol)

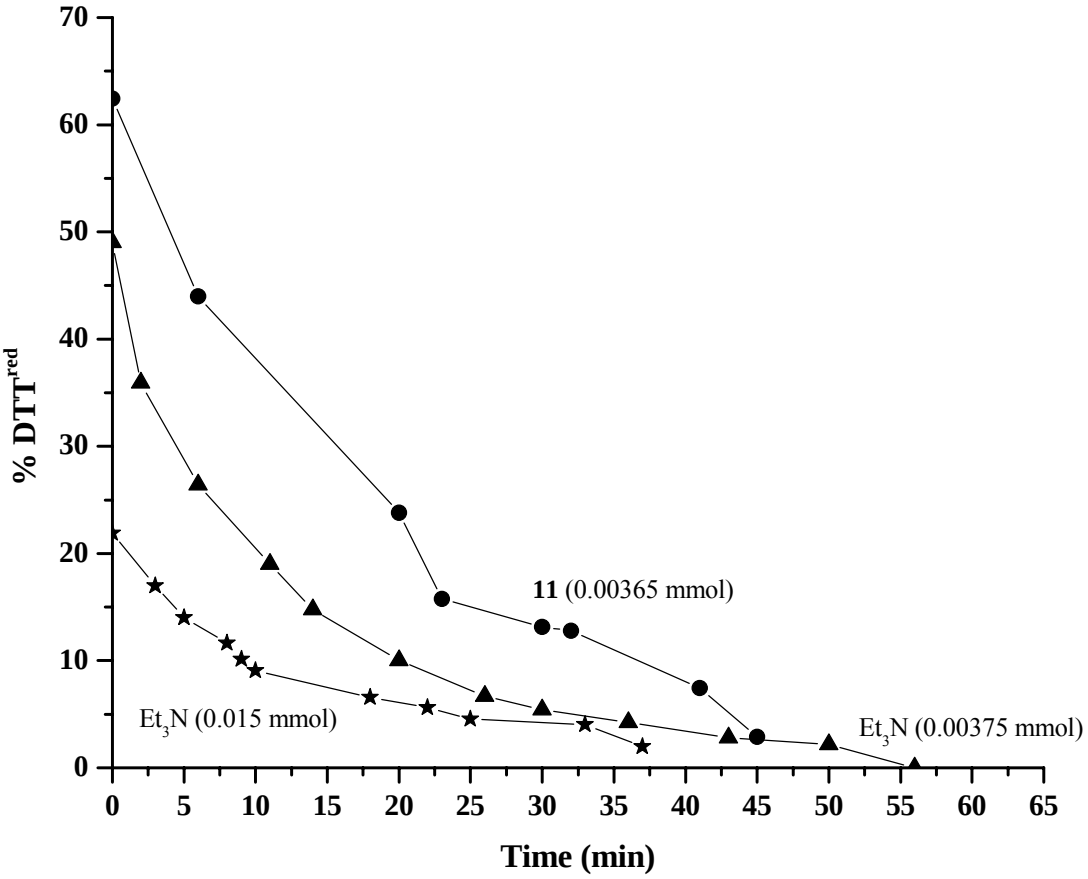


Effect of basic condition

A typical reaction mixture is obtained as given in equation below:

(1) DTT^{red} (23.1 mg, 0.15 mmol) + $dmpzCH_2CH_2SeCH_2CH_2NH_2$ (**11**) (0.9 mg, 0.00365 mmol) + H_2O_2 (9.15 µl from 47.6 % aqueous solution, 0.15 mmol) and

(2) DTT^{red} (23.1 mg, 0.15 mmol) + Et_3N (x mmol) + H_2O_2 (9.15 µl from 47.6 % aqueous solution, 0.15 mmol)



Crystal Information for **dmpz$CH_2CH_2SeCH_2COOH$ (3)**

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 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and
is
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R-
 factors based on ALL data will be even larger.
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H6C H 0.6458 -0.1876 0.9474 0.097 Uiso 1 1 calc R . .
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H8B H 0.7354 0.2429 0.8152 0.065 Uiso 1 1 calc R . .
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C6 0.078(7) 0.075(6) 0.040(4) 0.010(5) -0.010(5) -0.010(5)
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H6A C6 H6B 109.5 . . ?
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H6B C6 H6C 109.5 . . ?
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O1 C9 C8 122.7(9) . . ?
O2 C9 C8 112.9(9) . . ?
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Crystal Information for **dmpz$CH_2CH_2SeCH_2CH_2COOH$ (4)**

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_diffrn_radiation_monochromator  graphite
_diffrn_measurement_device_type      'Rigaku AFC7S'
_diffrn_measurement_method            \w-2\q
_diffrn_detector_area_resol_mean ?
_diffrn_standards_number              3
_diffrn_standards_interval_count      150
_diffrn_standards_decay_%             -0.23
_diffrn_reflns_number            3241
_diffrn_reflns_av_R_equivalents  0.0199
_diffrn_reflns_av_sigmaI/netI    0.0675
_diffrn_reflns_limit_h_min       -9
_diffrn_reflns_limit_h_max       17
_diffrn_reflns_limit_k_min       0
_diffrn_reflns_limit_k_max       14
_diffrn_reflns_limit_l_min       -10
_diffrn_reflns_limit_l_max       10
_diffrn_reflns_theta_min         2.93
_diffrn_reflns_theta_max         27.51
_reflns_number_total             2708
_reflns_number_gt                1595
_reflns_threshold_expression     >2sigma(I)

_computing_data_collection           'WinAFC'
```

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_computing_cell_refinement            'WinAFC'
_computing_data_reduction             'CrystalStructure'
_computing_structure_solution         'SIR 2004'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     'Ortep 3 for windows'
_computing_publication_material   'WinGX 1.80.01'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and
is
 not relevant to the choice of reflections for refinement.  R-factors
based
 on F^2^ are statistically about twice as large as those based on F, and
R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0249P)^2^+1.4004P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      SHELXL
_refine_ls_extinction_coef        0.0026(6)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          2708
_refine_ls_number_parameters      140
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.1006
_refine_ls_R_factor_gt            0.0372
_refine_ls_wR_factor_ref          0.0977
_refine_ls_wR_factor_gt           0.0775
_refine_ls_goodness_of_fit_ref    1.000
_refine_ls_restrained_S_all       1.000
_refine_ls_shift/su_max           0.000
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 _atom_site_adp_type
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 _atom_site_symmetry_multiplicity
 _atom_site_calc_flag
 _atom_site_refinement_flags
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Se1 Se 0.03797(3) 0.85299(4) 0.11515(6) 0.04673(17) Uani 1 1 d . . .
O1 O 0.1407(3) 0.4479(3) 0.1374(5) 0.0732(11) Uani 1 1 d . . .
O2 O 0.2768(2) 0.4969(3) 0.0002(5) 0.0628(10) Uani 1 1 d . . .
H2 H 0.2852 0.4241 -0.0003 0.094 Uiso 1 1 calc R . .
N1 N -0.2796(2) 0.8407(3) 0.0357(4) 0.0380(8) Uani 1 1 d . . .
N2 N -0.3285(2) 0.7342(3) 0.0472(4) 0.0378(8) Uani 1 1 d . . .
C6 C -0.3042(3) 1.0603(4) 0.0728(7) 0.0595(13) Uani 1 1 d . . .
H6A H -0.2464 1.0705 0.1479 0.089 Uiso 1 1 calc R . .
H6B H -0.3579 1.1123 0.1058 0.089 Uiso 1 1 calc R . .
H6C H -0.2859 1.0796 -0.0391 0.089 Uiso 1 1 calc R . .
C8 C 0.0609(3) 0.6803(4) 0.1197(6) 0.0496(12) Uani 1 1 d . . .
H8A H 0.0509 0.6498 0.2315 0.060 Uiso 1 1 calc R . .
H8B H 0.0128 0.6410 0.0432 0.060 Uiso 1 1 calc R . .
C4 C -0.4301(3) 0.8844(4) 0.1180(6) 0.0433(10) Uani 1 1 d . . .
H4 H -0.4871 0.9253 0.1523 0.052 Uiso 1 1 calc R . .
C3 C -0.3392(3) 0.9338(4) 0.0788(5) 0.0396(10) Uani 1 1 d . . .
C5 C -0.4212(3) 0.7616(4) 0.0968(5) 0.0369(9) Uani 1 1 d . . .
C9 C 0.1678(3) 0.6540(4) 0.0689(6) 0.0456(10) Uani 1 1 d . . .
H9A H 0.1768 0.6843 -0.0434 0.055 Uiso 1 1 calc R . .
H9B H 0.2152 0.6957 0.1440 0.055 Uiso 1 1 calc R . .
C2 C -0.1721(3) 0.8432(4) -0.0012(5) 0.0398(9) Uani 1 1 d . . .
H2A H -0.1588 0.9115 -0.0720 0.048 Uiso 1 1 calc R . .
H2B H -0.1551 0.7713 -0.0622 0.048 Uiso 1 1 calc R . .
C1 C -0.1063(3) 0.8509(4) 0.1559(5) 0.0456(10) Uani 1 1 d . . .
H1A H -0.1208 0.7831 0.2269 0.055 Uiso 1 1 calc R . .
H1B H -0.1236 0.9231 0.2160 0.055 Uiso 1 1 calc R . .
C10 C 0.1916(3) 0.5222(4) 0.0727(6) 0.0433(10) Uani 1 1 d . . .
C7 C -0.4984(3) 0.6652(4) 0.1163(6) 0.0523(12) Uani 1 1 d . . .
H7A H -0.4848 0.6005 0.0414 0.078 Uiso 1 1 calc R . .
H7B H -0.5648 0.6968 0.0904 0.078 Uiso 1 1 calc R . .
H7C H -0.4953 0.6365 0.2295 0.078 Uiso 1 1 calc R . .

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 _atom_site_aniso_U_33
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 _atom_site_aniso_U_12
Se1 0.0340(2) 0.0354(2) 0.0709(3) -0.0010(3) 0.00317(18) 0.0018(2)
O1 0.071(2) 0.0385(17) 0.113(3) 0.004(2) 0.048(2) -0.0012(17)
O2 0.0512(19) 0.0359(16) 0.104(3) 0.0097(18) 0.0321(18) 0.0111(15)
N1 0.0304(16) 0.0315(17) 0.053(2) -0.0025(17) 0.0070(14) 0.0008(14)
N2 0.0337(18) 0.0301(16) 0.050(2) -0.0020(16) 0.0052(15) -0.0005(14)
C6 0.051(3) 0.032(2) 0.095(4) -0.006(3) -0.005(3) 0.006(2)
C8 0.046(3) 0.032(2) 0.071(3) -0.001(2) 0.010(2) 0.0055(18)
C4 0.033(2) 0.044(2) 0.054(3) -0.005(2) 0.0067(19) 0.0089(17)
```

```
C3 0.038(2) 0.034(2) 0.047(3) -0.0045(19) 0.0002(19) 0.0037(18)
C5 0.034(2) 0.039(2) 0.038(2) 0.0011(19) 0.0040(18) 0.0001(17)
C9 0.044(2) 0.036(2) 0.058(3) -0.001(2) 0.008(2) 0.001(2)
C2 0.034(2) 0.037(2) 0.049(2) -0.001(2) 0.0108(18) 0.0041(18)
C1 0.036(2) 0.049(2) 0.052(3) -0.002(2) 0.0103(18) 0.002(2)
C10 0.039(2) 0.037(2) 0.054(3) -0.003(2) 0.006(2) -0.0004(19)
C7 0.043(2) 0.054(3) 0.061(3) 0.005(2) 0.008(2) -0.007(2)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
;

loop_
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Se1 C1 1.944(4) . ?
Se1 C8 1.952(4) . ?
O1 C10 1.197(5) . ?
O2 C10 1.315(5) . ?
O2 H2 0.8200 . ?
N1 C3 1.356(5) . ?
N1 N2 1.358(4) . ?
N1 C2 1.461(4) . ?
N2 C5 1.336(5) . ?
C6 C3 1.487(6) . ?
C6 H6A 0.9600 . ?
C6 H6B 0.9600 . ?
C6 H6C 0.9600 . ?
C8 C9 1.512(5) . ?
C8 H8A 0.9700 . ?
C8 H8B 0.9700 . ?
C4 C3 1.368(5) . ?
C4 C5 1.387(6) . ?
C4 H4 0.9300 . ?
C5 C7 1.495(5) . ?
C9 C10 1.506(6) . ?
C9 H9A 0.9700 . ?
C9 H9B 0.9700 . ?
C2 C1 1.503(6) . ?
C2 H2A 0.9700 . ?
C2 H2B 0.9700 . ?
C1 H1A 0.9700 . ?
C1 H1B 0.9700 . ?
C7 H7A 0.9600 . ?
```

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C7 H7B 0.9600 . ?
C7 H7C 0.9600 . ?

loop_
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C1 Se1 C8 97.90(18) . . ?
C10 O2 H2 109.5 . . ?
C3 N1 N2 111.9(3) . . ?
C3 N1 C2 127.9(3) . . ?
N2 N1 C2 119.8(3) . . ?
C5 N2 N1 105.2(3) . . ?
C3 C6 H6A 109.5 . . ?
C3 C6 H6B 109.5 . . ?
H6A C6 H6B 109.5 . . ?
C3 C6 H6C 109.5 . . ?
H6A C6 H6C 109.5 . . ?
H6B C6 H6C 109.5 . . ?
C9 C8 Se1 109.4(3) . . ?
C9 C8 H8A 109.8 . . ?
Se1 C8 H8A 109.8 . . ?
C9 C8 H8B 109.8 . . ?
Se1 C8 H8B 109.8 . . ?
H8A C8 H8B 108.2 . . ?
C3 C4 C5 107.0(4) . . ?
C3 C4 H4 126.5 . . ?
C5 C4 H4 126.5 . . ?
N1 C3 C4 105.7(3) . . ?
N1 C3 C6 122.5(4) . . ?
C4 C3 C6 131.8(4) . . ?
N2 C5 C4 110.2(4) . . ?
N2 C5 C7 120.1(4) . . ?
C4 C5 C7 129.6(4) . . ?
C10 C9 C8 112.4(3) . . ?
C10 C9 H9A 109.1 . . ?
C8 C9 H9A 109.1 . . ?
C10 C9 H9B 109.1 . . ?
C8 C9 H9B 109.1 . . ?
H9A C9 H9B 107.9 . . ?
N1 C2 C1 111.6(3) . . ?
N1 C2 H2A 109.3 . . ?
C1 C2 H2A 109.3 . . ?
N1 C2 H2B 109.3 . . ?
C1 C2 H2B 109.3 . . ?
H2A C2 H2B 108.0 . . ?
C2 C1 Se1 113.6(3) . . ?
C2 C1 H1A 108.8 . . ?
Se1 C1 H1A 108.8 . . ?
C2 C1 H1B 108.8 . . ?
```

```
Se1 C1 H1B 108.8 . . ?
H1A C1 H1B 107.7 . . ?
O1 C10 O2 123.0(4) . . ?
O1 C10 C9 124.5(4) . . ?
O2 C10 C9 112.5(4) . . ?
C5 C7 H7A 109.5 . . ?
C5 C7 H7B 109.5 . . ?
H7A C7 H7B 109.5 . . ?
C5 C7 H7C 109.5 . . ?
H7A C7 H7C 109.5 . . ?
H7B C7 H7C 109.5 . . ?

loop_
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 _geom_torsion_atom_site_label_4
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 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
C3 N1 N2 C5 0.8(4) . . . . ?
C2 N1 N2 C5 174.4(3) . . . . ?
C1 Se1 C8 C9 172.9(3) . . . . ?
N2 N1 C3 C4 -0.6(5) . . . . ?
C2 N1 C3 C4 -173.5(4) . . . . ?
N2 N1 C3 C6 -178.5(4) . . . . ?
C2 N1 C3 C6 8.5(7) . . . . ?
C5 C4 C3 N1 0.2(5) . . . . ?
C5 C4 C3 C6 177.8(5) . . . . ?
N1 N2 C5 C4 -0.6(5) . . . . ?
N1 N2 C5 C7 177.5(4) . . . . ?
C3 C4 C5 N2 0.3(5) . . . . ?
C3 C4 C5 C7 -177.6(4) . . . . ?
Se1 C8 C9 C10 178.9(3) . . . . ?
C3 N1 C2 C1 77.4(5) . . . . ?
N2 N1 C2 C1 -95.1(4) . . . . ?
N1 C2 C1 Se1 179.4(3) . . . . ?
C8 Se1 C1 C2 -89.2(3) . . . . ?
C8 C9 C10 O1 -13.3(7) . . . . ?
C8 C9 C10 O2 168.4(4) . . . . ?

_diffrn_measured_fraction_theta_max    0.999
_diffrn_reflns_theta_full              27.51
_diffrn_measured_fraction_theta_full   0.999
_refine_diff_density_max    0.436
_refine_diff_density_min   -0.415
_refine_diff_density_rms    0.087
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